Amtrak Penn Station New York

Remedial Investigation Work Plan

January 23, 2018

National Railroad Passenger Corporation

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List of Abbreviations

AOI	Area of Interest
CDI	Chronic Daily Intake
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain of Custody
CSM	Conceptual Site Model
DQO	Data Quality Objectives
DQI	Data Quality Indicators
DUSR	Data Usability Summary Report
EPC	Exposure Point Concentration
HHRA	Human Health Risk Assessment
LIRR	Long Island Rail Road
LTS	Long-Term Stewardship
mg/kg	milligram per kilogram
msl	mean sea level
MTA	Metropolitan Transportation Authority
NRCS	Natural Resources Conservation Service
NYSDEC	New York State Department of Environmental Conservation
ORNL-RAIS	Oak Ridge National Laboratory Risk Assessment Information System
OSWER	Office of Solid Waste and Emergency Response
PCBs	Polychlorinated Biphenyls
PSNY	Penn Station New York
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance / Quality Control
RAGS	Risk Assessment Guidance for Superfund
RSL	Regional Screening Levels
RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SOGR	State of Good Repair
SOP	Standard Operating Procedure
TAL	Target Analyte List
TCL+30	Target Compound List plus a 30-compound library search
TSCA	Toxic Substances Control Act
USEPA	United States Environmental Protection Agency
USDA	United States Department of Agriculture
USGS	United States Geological Survey

Glossary of Terms

Air: Indoor air within PSNY.

Area of Interest (AOI): Platform/Track level at PSNY designated into six (6) physical areas to facilitate implementation of the RI. Encompasses approximately 28 acres, including 11 platforms (over 4 miles in length), 21 tracks (over 14 miles in length), and 4 yard areas.

Aroclors: Trade name for various mixtures of chlorinated biphenyl congeners manufactured by Monsanto Chemical Company which were sold in the United States.

Askarel: A mixture of polychlorinated biphenyls, formerly used as an electrical insulator in transformers and capacitors.

Ballast Fines: Fine particles encountered within ballast stone.

Ballast Stone: One to four-inch diameter, sub-angular stone placed under/around rail track and ties to stabilize the rail structure.

Beyond the Concrete Track Structure: Areas outside the designated concrete track structure (AOI-1) in AOI 2 through 6. This area includes East and West running track as well as A, C, D and E yards.

Center of The Track Gauge: Centerline directly between two rails.

Chain of Custody (COC): Document recording transfer of sample custody from field technician to lab technician.

Chronic Daily Intake (CDI): The daily dose of a chemical averaged over time expressed as milligram of chemical per kilogram body weight of the receptor per day.

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA): Federal law commonly known as the “Superfund”. Federal law that provided broad Federal authority to respond directly to releases or threatened releases of hazardous substances that may endanger public health or the environment.

Conceptual Site Model: A written and/or illustrative representation of the physical, chemical and biological processes that control the transport, migration and actual/potential impacts of contamination.

Concrete: Concrete surfaces within PSNY including the concrete track structure, platforms, and supporting columns.

Concrete Track Structure: The track structure that is stabilized with concrete and not ballast stone. Wood ties are cemented within concrete.

Concrete Trough: A channel constructed of concrete present in the center of the concrete track structure. Troughs are graded so that liquids will travel towards drains.

Data Quality Objectives (DQO): DQOs define the purpose of data collection, clarify what the data should represent to inform the decisions to be made, and specify the performance requirements for the quality of information to be obtained from the data.

Data Quality Indicators (DQI): Indicators of acceptable data quality such as clean field blanks, high % laboratory matrix spikes, and acceptable DUSR reports.

Data Usability Summary Report (DUSR): Report summarizing Stantec’s data quality validation.

Decontaminated: Any material or object that has been cleaned to accepted standard.

DER-10: *Technical Guidance for Site Investigation and Remediation* administered by the New York State Department of Environmental Conservation's Division of Environmental Remediation. Issued May 3, 2010.

Diesel Fuel Wash: Applying diesel fuel to the concrete track structure in order to remove remaining "tar-like" material.

Dust: Any air borne particulate that is deposited on a surface.

Exposure Point Concentration (EPC): The concentration of a chemical of concern that could be contacted by a receptor over the exposure period. The EPC can be estimated using sampling data and/or chemical transport and environmental fate models.

Human Health Risk Assessment (HHRA): The process to estimate the nature and probability of adverse health effects in humans who may be exposed to chemicals in contaminated environmental media, now or in the future.

Long-Term Stewardship (LTS): Actions needed where remediation is ongoing or is completed to protect human health and the environment.

Mean sea level (msl): The global annual average level of the oceans.

Milligram per kilogram (mg/kg): Unit of measurement describing concentration of a substance in a solid medium (e.g. per million of a chemical in soil).

Natural Resources Conservation Service (NRCS): An agency of the USDA that provides technical assistance to farmers and other private landowners and managers.

New York State Department of Environmental Conservation (NYSDEC): The State of New York government agency designed to protect the environment and public health.

Oak Ridge National Laboratory Risk Assessment Information System (ORNL-RAIS): Contains an online calculator tool and database used for risk assessment purposes.

Office of Solid Waste and Emergency Response (OSWER): Former name of the Office of Land and Emergency Management (OLEM). Provides policy, guidance, and direction for the USEPA's emergency response and waste programs.

Oil Dri®: Loose granular absorbent material applied to a concrete track structure after a diesel and surfactant wash to quickly dry up remaining liquid.

Passenger Envelope: The public region of PSNY consisting of the Upper and Lower Concourses, the Passenger Platforms, and the passageways (stairs, escalators, and elevators) connecting these three levels. Describes physical boundaries and the receptors present within those boundaries.

Platform: Standing area adjacent to the track. Allows passengers to enter and exit trains at appropriate heights.

Polychlorinated Biphenyls (PCBs): Chemical substance that is limited to the biphenyl molecule that has been chlorinated to varying degrees or any combinations of substances which contain such substance (40 CFR 761.3).

Quality Assurance Project Plan (QAPP): A document that presents in specific terms the policies, organization, objectives, functional activities, and specific quality assurance/quality control activities involved with the acquisition of environmental information designed to achieve the data quality goals or objectives of a specific project or operation.

Quality Assurance / Quality Control (QA/QC): The total integrated program for assuring the reliability of monitoring and measurement data, which includes a system for integrating the quality planning, quality assessment, and quality improvement efforts to meet data end-use requirements.

Rail Interval: The vertical interval above the tie.

Railroad Zero/Railroad 0: Also referred to as milepost “zero”. Located on a concrete beam between signal 115W and 520E above Track 11.

Regional Screening Levels (RSL): USEPA generic risk-based concentrations for chemical constituents in soil, air, and tap water. RSLs are derived from standardized equations combining exposure information assumptions with USEPA toxicity data.

Remedial Investigation (RI): A process to determine the nature and extent of a discharge of a contaminant at a site or a discharge of a contaminant that has migrated or is migrating from the site, and may include data collected, site characterization, sampling, monitoring, and the gathering of any other sufficient and relevant information necessary to determine the necessity for remedial action.

Restricted Areas: Locations within PSNY where the public is not permitted and access is limited to railroad employees or other authorized individuals.

Risk Assessment Guidance for Superfund (RAGS): A complete description of USEPA's risk assessment methodology and definitions of the four components of risk assessment.

Sediment: Fine-grained, particulate material, often compacted, typically present on the concrete track structure.

Sensitivity: The ability to achieve the project-required reporting limits.

State Of Good Repair (SOGR): Projects which include repair and maintenance of infrastructure within PSNY to keep the station in good operating condition.

Surfactant: Compounds that lower the surface tension between a liquid and a solid allowing the solid to become mobile and be removed with the liquid.

“Tar-like” Material: A hard compacted fine-grained material that is present on the concrete track structure beneath sediment.

Target Analyte List (TAL): Target analyte list includes metals and cyanide designated for analysis by the USEPA. Analysis required for site characterization by the NYSDEC (DER-10).

Target Compound List plus 30 (TCL+30): Target compound list includes Volatile Organic Compounds (VOCs), Semi-Volatile Organic Compounds (SVOCs), Polychlorinated Biphenyls (PCBs), pesticides, and herbicides with a 30-compound library search. Analysis required for site characterization by the NYSDEC (DER-10).

Tie Interval: The sampling interval from the top of the railroad tie to the bottom of the railroad tie.

Toxic Substances Control Act (TSCA): Federal regulation passed in 1976 and administered by the USEPA to govern the use and safety of chemicals.

Track Gauge: The area between two corresponding rails on the railroad track structure.

United States Department of Agriculture (USDA): The U.S. federal executive department responsible for developing and executing federal laws related to farming, agriculture, forestry, and food.

United States Environmental Protection Agency (USEPA): The U.S. federal executive department responsible for developing and executing federal laws related to the protection of human health and the environment.

United States Geological Survey (USGS): A bureau within the U.S. Department of Interior responsible for classification of the public lands, and examination of the geological structure, mineral resources, and products of the national domain.

Wood Tie: Wood blocks used to support the rail. A component of the track structure.

1 Introduction

This Remedial Investigation Work Plan (RIWP) has been developed to describe the activities to be undertaken to complete a Remedial Investigation (RI) at Amtrak's Penn Station, New York City, New York (PSNY). Penn Station is a multi-level structure situated below the street level as further described in Section 1.1. Remedial measures are required on the Platform/Track Level, located approximately 50 feet below the street level due to the discovery of polychlorinated biphenyls (PCBs) in fine-grained heavily compacted material (henceforth referred to as "sediment" in this document) located on the concrete track structures adjacent to rail platforms in 2016. The detected concentrations of PCBs are regulated by the Toxic Substances Control Act (TSCA). Given the unique location, size, type and nature of operations at PSNY, the cleanup will be completed following a TSCA risk-based option per 40 CFR§ 761.61 (c). A risk-based application will be submitted to the United States Environmental Protection Agency (USEPA) under a separate cover.

This RIWP describes activities voluntarily completed by Amtrak as of December 1, 2017, proposes additional characterization efforts and sets forth a plan to evaluate risk to potential receptors. The RI will be used to develop a remedial action approach that will be protective of human health and the environment. Mott MacDonald prepared this RIWP on behalf of Amtrak with contributions from Stantec Consulting Services, Inc (Stantec), and TRC Environmental (TRC); collectively the environmental consulting team.

1.1 Site Description

PSNY is located in the Manhattan Borough of New York City and the Track Level encompasses over 28 acres extending from 7th Avenue to 10th Avenue and from West 31st Street to West 33rd Street. A site location map is provided as Figure 1. PSNY is located below Madison Square Garden and Two Penn Plaza and is comprised of three main levels which includes:

- Upper Concourse
- Lower Concourse, and
- Platform/Track Level.

All levels of PSNY are situated below street level as shown in Figures 2a through 2c. The Upper Concourse is generally an open area occupied by both Amtrak and New Jersey Transit which is divided into two sections defined by business operator. Both Amtrak and New Jersey Transit utilize respective areas for rail business operations including; customer waiting, ticketing and customer services with Amtrak's operations below Madison Square Garden and New Jersey Transit's operations below Two Penn Plaza. The Amtrak waiting area, ticketing, and customer service is located below Madison Square Garden. New Jersey Transit provides similar service under Two Penn Plaza. Additionally, The Upper Concourse includes vendor areas and access to the Lower Concourse and Track Level via stairs, escalators, elevators, and extends from 7th Avenue to 8th Avenue and from 31st Street to 33rd Street. The Lower Concourse consists of corridors and the Metropolitan Transportation Authority (MTA) police and Long Island Rail Road (LIRR) ticketing, waiting areas, additional vendor services and customer service. This level provides access to the Platforms/Track Level via stairs, escalators, and elevators. This level extends from 7th Avenue to 8th Avenue and from 31st Street to 34th Street. The Platform/Track Level consists of platforms and tracks, providing access for passengers to board trains. Additionally, the Platform/Track Level contains electrical substations and other areas occupied by Amtrak, LIRR and New Jersey Transit workers (i.e. break room, locker room, offices etc.) and train yards beyond the platform area used for storage and equipment transfers. This level extends from 7th Avenue to 10th Avenue and from 31st Street to 33rd Street.

Currently, there are eleven (11) platforms, twenty-one (21) tracks and four (4) train yards (A, C, D, and E). The platforms and tracks generally extend in an east-west orientation with the 11 platforms combining for

over 4 miles in length, and the 21 tracks combining for over 14 miles in length. The face of the seven tunnels that service the station is used to define the facility limits and is excluded from the workplan.

BOP SE LLC (commonly referred to as Brookfield) owns a portion of the project area that Amtrak uses and operates at PSNY. Currently, BOP SE LLC is in the process of constructing the Manhattan West Southeast Tower, which requires excavation in E yard and in track areas. Remediation of PCBs in this location will be completed by BOP SE LLC. following an EPA-approved Self-Implementing Cleanup Plan. The remediation details are described in a Self Implementing Cleanup Plan dated November 6, 2017 prepared by AKRF. The excavation area is shown on Figure 2c and are excluded from this project.

1.2 Site Operational History

PSNY is a historic railroad station in New York City, named for the Pennsylvania Railroad (PRR), its builder and original tenant. The development of the station and the adjoining North River Tunnels and East River Tunnels began in 1901 and was completed after nine years of construction, in 1910. On November 27, 1910, PSNY was opened to the public.

The original PSNY head house and train shed were considered a masterpiece of the “Beaux-Arts” style and one of the architectural jewels of New York City. The original Station was a vast structure that occupied two whole city blocks. The boundaries surrounding the structure were 31st and 34th Streets, between 7th and 8th Avenues. The original Pennsylvania Station building was 784 by 430 feet, covering an area of 8 acres. It was one of the first rail terminals to separate arriving from departing passengers on two concourses. Over 500 buildings were initially cleared for its construction. The building boasted an ornate exterior, arcade, waiting room, concourse, and carriage-ways.

In the late 1960’s and early 1970’s, railroads in the northeastern portion of the United States were in financial crisis. To prevent a total collapse of the freight and passenger rail service in that area of the country, Congress created Conrail and Amtrak; Conrail assumed operation of the freight rail (and some commuter rail) services in the area, and Amtrak assumed all intercity passenger rail operations.

Both Amtrak and Conrail were given ownership or control of various portions of the bankrupt assets by Congress. In 1976, ownership of most of the Northeast Corridor rail line (NEC) was transferred to Amtrak, including stations and the support facilities necessary to operate Amtrak services. These support facilities included various maintenance of way facilities, as well as facilities used to maintain rolling stock. Penn Station was one of the larger and more unique facilities transferred to Amtrak during this time.

Amtrak owns PSNY, or has designated easements for railroad operations, but it is subject to numerous restrictions, including heavy restrictions on Amtrak’s ability to sell or lease the subterranean operating track structure of the property, or to itself use the property for anything other than railroad purposes.

Rail services at PSNY are provided by Amtrak (intercity rail), New Jersey Transit (commuter), and LIRR (commuter). New Jersey Transit exclusively uses Tracks 1 through 4, Amtrak and New Jersey Transit share Tracks 5 through 14, and LIRR uses Tracks 15 through 21. PSNY operates 24 hours a day, 365 days a year, and is the busiest passenger transportation facility in North America with each of the station's 21 tracks in use every 2 minutes on average during weekdays. It serves more than 430,000 passengers with over 1,300 train movements per weekday. Track access to conduct any environmental activities associated with this work plan are complicated by the complexity of PSNY, its heavy volume of traffic, extensive utility network, as well as Federal Railroad Safety rules. Access to sampling locations requires Amtrak contractor safety training, watchmen protection, and track outages that need to be coordinated to minimize impact to rail operations. Further, the nature of the site also limits the type of equipment which can effectively be deployed and used within the station.

1.3 Areas of Interest

The primary focus of the RI is the Platform/Track Level, based on the findings of evaluations conducted to date. However, investigation will occur at other levels of PSNY as outlined in this RIWP. The station was designated into six (6) Areas of Interest (AOI)s to facilitate implementation of the physical aspects of the

RI and associated technical reporting. These AOIs were designated based on current and prior operations, known conditions and accessibility, and are presented on Figure 2c.

- AOI-1 corresponds to the concrete track structure and adjacent platforms. The concrete track structure includes wood ties and a concrete trough in the center of the track gauge;
- AOI-2 corresponds to A, D, and E yards located west of AOI-1;
- AOI-3 corresponds to C yard, located west of AOI-6;
- AOI-4 corresponds to track areas east of AOI-1 and AOI-6;
- AOI-5 corresponds to track areas west of AOI-1 and AOI-6 that are not included in AOI-2 and AOI-3;
- AOI-6 corresponds to Tracks 19, 20, and 21 and adjacent platforms. These tracks differ from others in that they are typical ballast stone and not concrete track construction.

1.4 Objectives of Remedial Investigation

The primary goals and objectives of the RI are to:

- Identify representative groups of people present in different locations at PSNY who may be exposed to PCBs and other potential contaminants of concern.
- Evaluate the presence of PCBs to assess remedial actions for the protection of human health and the environment.
- Assess PCBs both within the underground portions of PSNY and potential migration of PCBs within the Passenger Envelope of PSNY. Receptors will include railroad workers, the general public and others, as identified.
- Information developed in the RI will be utilized to develop compliance with applicable regulations (i.e. 40 CFR 761.61)
- Develop realistic and feasible remedies that may be required based on the potential risks posed to human health and the environment; and the implementation and long-term stewardship of selected remedial actions.

The information gathered during the RI will be evaluated and summarized in an Application for a Risk-Based Disposal Approval and Notification and Certification of Disposal of PCB remediation waste in accordance with 40 CFR 761.61(c).

2 Environmental Setting

2.1 Topography and Surface Conditions

Review of the United States Geological Survey (USGS) Topographic Map dated 2016 (see Figure 1) indicates that the topographic elevation of the ground surface (at street level) at PSNY ranges from approximately 30 to 40 feet above mean sea level (msl), and is primarily level with a gentle slope to the west. The Hudson River is located approximately 2,000 feet west of PSNY, and the East River is located approximately 6,000 feet east of PSNY. As previously mentioned in Section 1.0, the majority of the RI will be completed at the Platform/Track level, which is approximately 50 feet below the sidewalk level (approximately 10 to 20 feet below msl).

2.2 Subsurface and Geologic Conditions

According to the United States Department of Agriculture (USDA) Natural Resources Conservation Service (NRCS) soil mapping tool, PSNY is underlain by soils that are classified as urban land, till substratum with zero to three present slopes. The soil is described as urban fill, which may contain a mix of native and non-native materials. The USDA NRCS mapping tool can be found at https://websoilsurvey.sc.egov.usda.gov/App/HomePage.htm.

According to the USGS Bedrock Geology Map of New York (Lower Hudson Sheet) dated 2008, bedrock geology at PSNY consists of the undivided Manhattan Formation. The unit age is Middle Ordovician, and is comprised of pelitic schists (Manhattan Schist), gneiss, and amphibolite. Pelitic schists is a schistose metamorphic rock derived by metamorphism of an argillaceous or a fine-grained aluminous sediment. Gneiss is a foliated rock formed by regional metamorphism, in which bands or lenticules of granular minerals alternate with bands or lenticules in which minerals having flaky or elongate prismatic habits predominate. Generally, less than half of the minerals show preferred orientation. Amphibolite is a crystalloblastic rock consisting mainly of amphibole and plagioclase with little or no quartz.

During initial site characterization in 2016 through November 2017, test holes were excavated at various locations east and west of the concrete track structure as well as in the yard areas. Bedrock was encountered at depths ranging from immediately below the railroad tie to approximately 2 feet below the railroad tie. Most of the soil at PSNY was removed during construction of this station structure such that only ballast stone and ballast fines are above bedrock.

3 Site Characterization

PCBs were detected in sediment located on the concrete track structure of Track 1, adjacent to Platform 1 in early 2016, as a result of routine environmental work associated with an engineering maintenance project. PCBs were used widely as insulating fluids in electrical equipment, as well as in hydraulic systems, surface coatings, flame retardants and other applications. PCBs have been used as coolants and lubricants in transformers, capacitors, and other electrical equipment. Rail locomotives and self-propelled rail cars contained transformers. Some dielectric fluids contained PCBs. The transformers were typically located on the undercarriage of the railcars. As such, sampling described in the RIWP is biased to locations in the track gauge. The chemical properties of PCBs such as nonflammability, chemical and thermal stability, and miscibility with organic compounds, lead to the wide usage in various applications. PCBs have relatively low water solubility and low vapor pressures that allow them to partition between water and the atmosphere. PCBs tend to partition to the more organic components of the environment. For that reason, PCBs adsorb onto organic matter in soils and sediments.

PCBs were manufactured in the United States from 1929 to 1977 and are regulated by TSCA. Currently, LIRR, New Jersey Transit and Amtrak no longer use electrical equipment that contains PCBs. PCBs appear as colorless to light yellow oily liquids or waxy solids. Many commercial PCB mixtures are known in the United States by the trade name Aroclor. PCB mixtures used in transformer fluids were commonly termed “askarel mixtures”. Askarel-insulated transformers were utilized in both commuter and long-distance passenger rail service.

To fully characterize the extent of PCBs in the various media throughout PSNY, Amtrak initiated a station-wide sampling approach considering potential receptors as well as the type, duration and frequency of work tasks performed by Amtrak personnel. Sampling and characterization of environmental media was initiated (and continues as of the preparation of this RIWP) for sediment on the PSNY track structures including “tar-like” material discovered below the sediment, ballast fines and ballast stone located beyond the concrete track structures, concrete components of the concrete track structures, dust that has accumulated on the various surfaces throughout the station, and station level air. Access to sampling locations require Amtrak contractor safety training, watchmen protection, and track outages that need to be coordinated to minimize impact to rail operations. Sections 3.1 through 3.7 summarize the findings of sampling completed to date (as of December 1, 2017) for each media type at PSNY.

Sample collection and equipment is further described below. Please note the sampling equipment was decontaminated as described in section 6.4. Samples of solid media were analyzed for PCBs using standard methods; USEPA Method 3540C (soxhlet extraction) and analyzed via USEPA Method 8082 by a New York State certified laboratory. Air samples were analyzed by USEPA Method TO-10A by a New York State certified laboratory.

3.1 Sediment and “Tar-Like” Material

Following the 2016 detection of PCBs in sediment that accumulated on the concrete track structure at Track 1, Amtrak retained Mott MacDonald to collect sediment samples from concrete track structures within PSNY. Samples of the sediment were collected and submitted for PCB analysis from Tracks 1 through 18 in 2016 (sampling of accumulated sediment activities continue as of the preparation of this RIWP). The majority of Tracks 19 through 21 do not contain concrete track structures (except for an eight-foot section of concrete track structure at Track 21) and thus, no samples were collected in the initial sampling program. Samples were collected at a frequency of one sample per 100 linear feet of concrete track structure, where sediment was present within the track gauge. Additional sediment samples were collected adjacent to drainage structures, when encountered. Also, as part of a state of good repair (SOGR) project proposed for Track 10, samples were collected at a higher frequency (one sample per 30 linear feet) to evaluate conditions for worker protection. Each sample of sediment was biased toward areas which exhibited visual evidence of impacts within the track gauge. PCBs were detected at concentrations ranging from 4.32 milligrams per kilogram (mg/kg) to 80,100 mg/kg (sum of Aroclors

detected). The sediment analytical results are provided on the attached Figures 3a (Odd Tracks) and 3b (Even Tracks).

Following the removal of sediment, a “tar-like” material was encountered on the concrete track structure. Samples of the “tar-like” material were collected from Tracks 2, 3, 4, 5, and 7 and submitted for analysis. The samples were collected from the center of the track gauge biased to areas where the potential for impacts were believed to be the greatest. PCBs were detected at concentrations ranging from 12.9 mg/kg to 39,300 mg/kg (sum of Aroclors detected). The “tar-like” material sampling locations and the analytical results are presented on Figure 4.

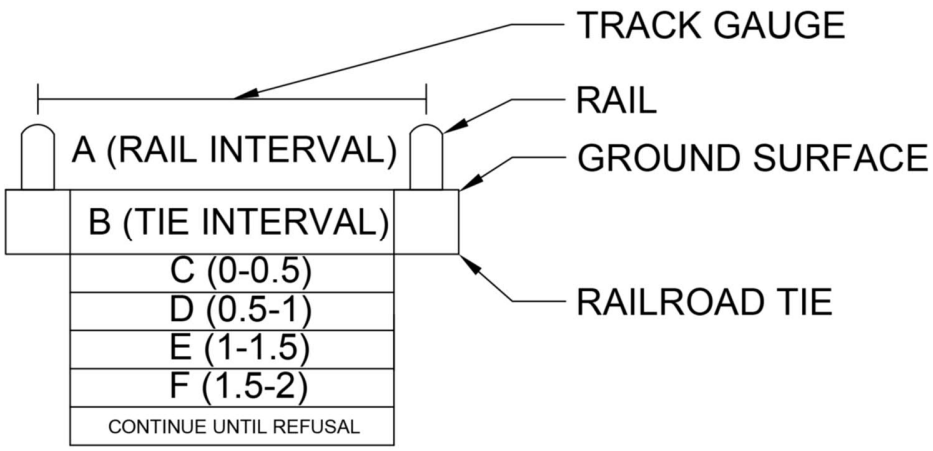
Following the confirmation of the presence of PCBs in the sediment and “tar-like” material, Amtrak retained Clean Harbors Inc. Environmental Services (Clean Harbors) to begin removing the material from the concrete track structures where visibly present (Section 4.1). Photographs of “tar-like” material are included below:

3.2 Ballast Fines

Beyond the concrete track structures (outside of AOI-1), the track structures are generally comprised of ballast fines and ballast stone. The percentage of ballast fines compared to ballast stone varies along the track structure. The ballast fines are a fine-grained material that is comingled with and/or present below ballast stone. Samples of the ballast fines have been collected from various depths inside the center of the track gauge (ballast fines sampling continues as of the preparation of this RIWP). Samples were collected east and west of the concrete track structure as part of characterization for planned SOGR projects.

Samples were collected via hand-dug test holes at a rate of one location per 25 linear feet within the track gauge. The test holes were excavated using a shovel, post hole digger or equivalent hand tool. At each 25-foot interval, material encountered within three vertical zones (the rail interval, the tie interval, and 0-0.5 feet below the tie interval) were sampled. For clarification purposes, the “rail interval” corresponds to, and will be defined as, the vertical interval above the wooden tie. The “tie interval” corresponds to the vertical interval from the top of the wooden tie to the bottom of the tie. The sampling intervals are illustrated in the *Sampling Interval Diagram* provided in Appendix A and shown below. If a sufficient amount of ballast fines were present at the target depth interval, a sample was collected for analysis. If ballast fines were not present, no sample was collected for analysis at that depth interval. In addition, at every 150 linear feet of track, samples were collected and analyzed for PCBs at every six-inch depth interval below the bottom of the tie until refusal was encountered. Analytical results from ballast fines sampling indicate concentrations of PCBs ranging from 0.134 mg/kg to 97.1 mg/kg (sum of Aroclors detected). The ballast fines sample locations and the analytical results are provided on Figures 5a through 5h.

Sampling Interval Diagram:



Ballast fines samples were also collected in areas of SOGR planned projects. Specifically, samples were collected in the vicinity of the following areas:

- Switch 73 & 83 (Figure 6b)
- 10th Ave Switch (Figure 6c)
- 9th Ave Switch (Figure 6d)
- Switch 93 & 105 (Figure 6e)
- Switch 619, 621, & 635 (Figure 6f)
- Switch 49 (Figure 6g)

The analytical results for ballast fines samples collected at SOGR projects are provided on the attached Figures 6a through 6f. A photograph depicting an example of ballast fines is included below:

3.3 Ballast Stone

Railroad ballast stone is present on the surface of the track structure at AOIs 2 through 6. Surface ballast stone samples were generally collected at a rate of one per 150 linear feet of track in conjunction with the

ballast fines sampling described in Section 3.2 (sampling of ballast stone continues as of the preparation of this RIWP). The ballast stone samples were collected from the first sampling interval encountered. Analytical results from ballast stone sampling indicated the concentration of PCBs ranged from not detected to 1.43 mg/kg (sum of Aroclors detected). The ballast stone sampling locations collected in the vicinity of Tracks 9, 12 and 18 and associated analytical results are shown on Figures 5e, 6f, and 5h, respectively. A photograph depicting an example of ballast stone is included below:

3.4 Concrete

Amtrak retained Clean Harbors to remove sediment and "tar-like" material and to wash the concrete track structure. The washing process is described in Section 4.1. After the concrete track structure has been washed and passed a visual inspection, concrete samples were collected from the center of the trough in the track gauge to document any residual concentrations of PCBs.

Concrete samples were collected at a rate of one sample per 100 linear feet of track structure. If a drain was encountered in the track structure, an additional concrete sample was collected adjacent to the drain and submitted for analysis (concrete sampling continues as of the preparation of this RIWP). The analytical results from concrete sampling indicated concentration of PCBs ranging from not detected to 6,828 mg/kg. The removal of sediment and "tar-like" material, and track washing is on-going. The concrete "after final washing" analytical results (as of December 1, 2017) are presented on Figures 7a and 7b. A photograph of concrete (after final washing) is included below:

3.5 Dust

As part of routine cleaning and maintenance, Amtrak periodically conducts industrial cleaning to remove residual material from areas reachable by the general public (i.e. floors, walls and handrails). However, bulk dust has accumulated on various surfaces outside of the reach of the general public, including, but not limited to, the tops of overhead light fixtures, conduits, pipes, brackets, walls and signs. Limited sampling has been completed at platforms and substations as described in sections 3.5.1 and 3.5.2 below (dust sampling continues as of the preparation of this RIWP). A photograph of dust on top of a light fixture is included below:

3.5.1 Platforms

Bulk dust samples were collected from the top of light fixtures on platforms adjacent to Tracks 4, 6, and 12. PCBs were detected at concentrations ranging from 2.28 mg/kg to 32.9 mg/kg (sum of Aroclors detected). The dust sample locations and analytical results are presented on Figure 8.

3.5.2 Substations

Bulk dust samples have been collected from surfaces in Substations E01, 2C, and 43. The samples were collected from various surfaces (i.e., floors, walls, and on equipment) where visible dust was present (sampling at additional substations continues as of the preparation of this RIWP). Analytical results indicated that PCB concentrations ranged from 1.4 mg/kg to 33.3 mg/kg. The substation locations are presented on Figures 2c and 4. The substation dust sample locations and the analytical results are presented on Figures 9a through 9c.

3.6 Air

As part of the site characterization, Amtrak requested that air sampling for PCBs be conducted in various areas and locations in PSNY. Air sampling was performed to evaluate potential exposures to railroad employees and the general public. The types of air sampling and evaluation performed to date are described below.

Air sampling was conducted by TRC in accordance with USEPA Method TO-10A - *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*. The air samples were collected at a flow rate of approximately five (5) liters per minute, for approximately 300 to 400 minutes, and a total volume of around 1,800 to 2,000 liters for each sample. The sampling trains were calibrated at the beginning and end of the sampling period using a primary calibrator (i.e., mini-Buck Calibrator Model M-5, or equivalent). Sampling was performed at the approximate breathing height of an adult. No quartz-fiber pre-filter was utilized during sampling, so the sample analysis included both the particulate and vapor phases for PCBs. A minimum of one (1) field blank was included with each sample set for quality assurance purposes. The samples were analyzed by Con-Test Analytical Laboratories (Con-Test), located in East Longmeadow, Massachusetts, an independent third-party industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033).

3.6.1 Flagmen/Watchmen Exposure Sampling

On May 6, 2016, two (2) area air samples were collected for PCBs in the vicinity of the Amtrak Flagmen/Watchmen working within the station while Clean Harbors was removing sediment and “tar-like” material on Track 10. The sampling pumps were stationed at the east and west end of Platform 5, adjacent to where the flagmen were located. Analytical results for the samples ranged from 33 to 66 nanograms per cubic meter of air (ng/m^3). (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.2 A, D and E Yard Areas

On May 24, 2016, three (3) area air samples were collected for PCBs in the vicinity of typical Amtrak track employee work areas within or around the A, D and E train yards (AOI-2) at PSNY. The sampling pumps were stationed at the north side of Track 6E and south side of Track 1A at the track level and on the diagonal platform near the Amtrak offices. Analytical results for the samples ranged from less than 22 to 40 ng/m^3, with an average concentration of 30.0 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.3 Track Walking Simulation Studies

On June 21, July 12, and July 19, 2016, personal air sampling on Clean Harbors personnel was conducted as they walked back and forth on sections of the concrete track structure where sediment and “tar-like” material had not been removed as part of track washing procedures, to be representative of Amtrak personnel walking on the track. Six (6) air samples were collected: two (2) samples on June 21, 2016, two (2) samples on July 12, 2016 and two (2) samples on July 19, 2016. During each of the three (3) simulation studies, one (1) sampling pump was stationed in the center of Platform 1 to measure the ambient levels of PCBs within the station. In addition to the Platform 1 sample, one (1) sampling pump was placed on a Clean Harbors employee while he walked back and forth along the concrete track

structure on either Track 4 or Track 7 for approximately 30 minutes. At the conclusion of the walking exercise, the sampler was then placed in the center of Platform 1 and left running for the remainder of the work shift. Analytical results for the samples ranged from 90 to 138 ng/m^3, with an average of 118.7 ng/m^3. By comparison, the ambient concentrations measured on Platform 1 ranged from 68 to 91 ng/m^3, with an average concentration of 75.3 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.4 Ballast Digging Simulation Studies

On August 5, August 24, and October 5, 2016, personal air sampling of Clean Harbors personnel was conducted as track ballast was excavated between the track gauges in PSNY to be representative of Amtrak personnel doing excavation work. Six (6) air samples were collected: two (2) samples on August 5, 2016, two (2) samples on August 24, 2016, and two (2) samples on October 5, 2016. During each of the three (3) simulation studies, one (1) sampling pump was stationed approximately 50 feet away from the digging location to measure the ambient levels of PCBs within the station. In addition to the area sample, one (1) sampling pump was placed on a Clean Harbors employee while excavating ballast for approximately 60 minutes. At the conclusion of the ballast digging study, the sampler was then placed adjacent to the area sample and left running for the remainder of the work shift. Analytical results for the samples were all less than 28 ng/m^3, while the ambient concentrations measured in A and E Yards were all less than 30 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.5 31st Street Elevated Substation

On January 12, 2017, air sampling was conducted within the 31st Street elevated substation, located above Tracks 1E and 2E in E yard, to determine ambient concentrations during normal daytime operations. Two (2) area air samples were collected. Analytical results for the samples were all less than 27 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.6 7th Avenue Elevated Substation

On February 3, 2017, air sampling was conducted within the 7th Avenue elevated substation, located above the east end of Tracks 1, 2, 3 and 4, to determine ambient concentrations during normal daytime operations. Two (2) area air samples were collected. Analytical results for the samples were all less than 27 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 for additional details in Appendix B.)

3.6.7 Track Level Sampling

On May 17, August 31, November 29, 2016, and February 23, 2017, air sampling was conducted at Platform/Track Level, on the passenger platforms, to determine ambient concentrations during normal daytime operations. During each event, samples were collected between approximately 9:30am and 4:30pm, and five (5) samples were collected from the following locations:

- East end of Platform 1, adjacent to the JOPD shack
- East and West ends of Platform 4
- East and West ends of Platform 9

Analytical results for the samples ranged from less than 21 to 184 ng/m^3, with an overall average concentration of 63.05 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.8 Lower Concourse Sampling

On the evenings of October 18 and November 30, 2016, air sampling was conducted in the East, Central and Exit Concourses on the Lower Concourse of PSNY, where the LIRR operates, to determine ambient concentrations at the Concourse Level. Sampling was conducted between approximately 10:30pm and 5:30am. During each event, a total of nine (9) air samples were collected in the following locations:

- East Concourse – by entrance to Tracks 15/16 and Tracks 20/21,
- Central Concourse – by entrance to Tracks 15/16 and Tracks 18/19, and
- Exit Concourse – by entrance to Tracks 3/4, Tracks 7/8, Tracks 11/12, Tracks 15/16, and Tracks 18/19.

Analytical results for the lower level area air samples collected on October 18, 2016 ranged from less than 24 to 100 ng/m^3, while results for the samples collected on November 30, 2016 ranged from less than 21 to 100 ng/m^3. The overall average air concentration for the two events was 36.28 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 for additional details in Appendix B.)

3.6.9 Upper Concourse Sampling

On the evening of January 24-25, 2017, air sampling was conducted on the Upper Concourse of PSNY, where Amtrak and New Jersey Transit operate, to determine ambient concentrations at the Concourse Level. Six (6) samples were collected between approximately 10:00pm and 4:00am. Samples were collected from the following locations:

- Amtrak waiting area – near the entrances to platforms 3, 5 and 8, and
- New Jersey Transit waiting area – near the entrances to platforms 1, 4 and 5.

Analytical results for the samples ranged from less than 22 to 66 ng/m^3, with an average concentration of 31.17 ng/m^3. All of the samples collected in the Amtrak waiting area were less than 22 ng/m^3, while the samples collected in the New Jersey Transit waiting area ranged from 26 to 66 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

3.6.10 Station-Wide Sampling

On the evening of June 21-22, 2017, air sampling was conducted on the Track Level and the Lower, and Upper Concourses of PSNY, to determine ambient concentrations at the Concourse Level. Samples were collected between approximately 10:00pm and 5:00am. 23 air samples were collected from the following locations:

Platform/Track Level sampling:

- Track level sample location 01 - East side of Platform 2, near stairs
- Track level sample location 02 - West side of Platform 2, near stairs
- Track level sample location 03 - East side of Platform 4, near stairs
- Track level sample location 04 - West side of Platform 4, near stairs
- Track level sample location 05 - East side of Platform 7, near stairs
- Track level sample location 06 - West side of Platform 7, near stairs
- Track level sample location 07 - East side of Platform 9, near stairs
- Track level sample location 08 - West side of Platform 9, near stairs

Lower Concourse air sampling:

Exit Concourse

- Lower level sample location 01 - By entrance to Tracks 3/4
- Lower level sample location 02 - By entrance to Tracks 7/8

- Lower level sample location 03 - By entrance to Tracks 11/12
- Lower level sample location 04 - By entrance to Tracks 15/16
- Lower level sample location 05 - By entrance to Tracks 18/19

Central Concourse

- Lower level sample location 06 - By entrance to Tracks 15/16
- Lower level sample location 07 - By entrance to Tracks 18/19

Main Gate Area

- Lower level sample location 08 - By Tracks 15/16
- Lower level sample location 09 - By Tracks 20/21

Upper Concourse air sampling:

Amtrak Concourse

- Upper level sample location 01 - By entrance to Tracks 3/4
- Upper level sample location 02 - By entrance to Tracks 7/8
- Upper level sample location 03 - By entrance to Tracks 11/12

New Jersey Transit Concourse

- Upper level sample location 04 - By entrance to Tracks 1/2
- Upper level sample location 05 - By entrance to Tracks 5/6
- Upper level sample location 06 - By entrance to Tracks 9/10

Analytical results for the Track Level samples ranged from less than 21 to 227 ng/m^3, with an average concentration of 104.0 ng/m^3. Results for the Lower Concourse samples ranged from less than 19 to 61 ng/m^3, with an average concentration of 34.67ng/m^3. Results for the Upper Concourse samples ranged from less than 19 to 49 ng/m^3, with an average concentration of 29.0 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated September 19, 2017 in Appendix B for additional details.)

On the evening of September 26-27, 2017, air sampling was conducted on the Track Level and the Lower, and Upper Concourses of PSNY, to determine ambient concentrations at the Concourse Level. Samples were collected between approximately 11:00pm and 6:30am. 26 air samples were collected from the following locations:

Platform/Track Level sampling:

- Track level sample location 01 - East side of Platform 2, near stairs
- Track level sample location 02 - West side of Platform 2, near stairs
- Track level sample location 03 - East side of Platform 4, near stairs
- Track level sample location 04 - West side of Platform 4, near stairs
- Track level sample location 05 - East side of Platform 6, near stairs
- Track level sample location 06 - West side of Platform 6, near stairs
- Track level sample location 07 - East side of Platform 8, near stairs
- Track level sample location 08 - West side of Platform 8, near stairs
- Track level sample location 09 - East side of Platform 10, near stairs
- Track level sample location 10 - West side of Platform 10, near stairs

Lower Concourse air sampling:

Exit Concourse

- Lower level sample location 01 - By entrance to Tracks 3/4
- Lower level sample location 02 - By entrance to Tracks 7/8
- Lower level sample location 03 - By entrance to Tracks 11/12

- Lower level sample location 04 - By entrance to Tracks 15/16
- Lower level sample location 05 - By entrance to Tracks 18/19

Central Concourse

- Lower level sample location 06 - By entrance to Tracks 15/16
- Lower level sample location 07 - By entrance to Tracks 18/19

Main Gate Area

- Lower level sample location 08 - By Tracks 11/12
- Lower level sample location 09 - By Tracks 15/16
- Lower level sample location 10 - By Tracks 18/19

Upper Concourse air sampling:

Amtrak Concourse

- Upper level sample location 01 - By entrance to Tracks 7/8
- Upper level sample location 02 - By entrance to Tracks 11/12
- Upper level sample location 03 - By entrance to Tracks 15/16

New Jersey Transit Concourse

- Upper level sample location 04 - By entrance to Tracks 3/4
- Upper level sample location 05 - By entrance to Tracks 7/8

Analytical results for the Track Level samples ranged from less than 22 to 107 ng/m^3, with an average concentration of 42.1 ng/m^3. Results for the Lower Concourse samples ranged from less than 20 to 46 ng/m^3, with an average concentration of 25.7ng/m^3. Results for the Upper Concourse samples ranged from less than 21 to 24 ng/m^3, with an average concentration of 22.2 ng/m^3. (Refer to the TRC report *Summary of PCB Air Sampling Events* dated October 16, 2017 in Appendix B for additional details.)

3.7 Drains and Sumps

Drains are present in the center of the track gauge within the concrete track structure (AOI-1), however drains exist within other areas of the track level. These drains flow to three (3) sumps located below the Platform/Track level at PSNY. The sumps ultimately discharge to the New York City sanitary sewer system. To date, no drains have been sampled. A photograph of sediment in drains is included below:

Clean Harbors collected samples of sludge material from the 7th Avenue and 9th Avenue sumps in conjunction with clean-out and maintenance activities. PCBs were detected at concentrations of 9.5 mg/kg and 32.5 mg/kg (sum of Aroclors) in the sludge material at the 7th and 9th Avenue Sumps, respectively. It should be noted that these sumps also accept wastewater from the station's Upper and Lower Concourse levels.

4 Contaminant Removal and Track Cleaning

Following the detection of PCBs at PSNY in early 2016, Amtrak initiated removal activities to address PCB contaminated media at the Platform/Track Level of PSNY. The removal efforts currently underway are related to the removal of sediment and “tar-like” material from the concrete track structures and dust at platforms and substations. Particulate air concentrations are monitored during removal and track cleaning operations as described in Section 4.4.

In addition, Amtrak undertakes various maintenance activities and construction projects to keep the station in a SOGR. The SOGR projects include the removal of ballast stone and ballast fines associated with track or communication projects. As feasible, Amtrak has integrated removal tasks into planned SOGR projects to take advantage of track access and expedite the removal of sediment and “tar-like” material. All waste generated from any removal operation has been disposed of as a TSCA waste. The removal operations performed to date are summarized below.

4.1 Sediment, “Tar-Like” Material Removal and Track Cleaning

Beginning in April 2016, Amtrak retained Clean Harbors to remove the sediment and “tar-like” material from the concrete track structures in AOI-1. Loose debris is removed from the track structure, placed into containers, and then transported to the sub-basement where the waste is temporarily stored (less than 90 days) before being disposed offsite as a TSCA waste.

Following debris removal, sediment removal commences by first spraying water onto the work area with a garden hose to wet the concrete track structure, minimizing dust created by the work, followed by scraping, sweeping, and vacuuming the sediment into containers. The contaminated sediment is then transported to the sub-basement where the waste is temporarily stored (less than 90 days) before being disposed offsite as a TSCA waste.

Once the sediment has been removed, the “tar-like” material removal is initiated using electric-powered chisels. The removal process is continued with use of hand scrapers and wire brushes. The resultant debris is shoveled into containers and transported in containers to the sub-basement where the waste is temporarily stored (less than 90 days) before being disposed offsite as a TSCA waste. Remaining debris is vacuumed using shop vacuums with HEPA filters. The filters are periodically replaced. Used filters and debris are disposed of as a TSCA waste. The removal is considered complete after a visual inspection determines no sediment and/or “tar-like” material remains on the track structure. As of December 1, 2017, approximately 82% of the concrete track structure has undergone sediment and “tar-like” material removal.

In some areas, residual “tar-like” material remains after the scraping/chisel/power brush operations. Residual ‘tar-like” material is removed by a washing process. The washing process is initiated by spraying down the track structure with diesel fuel. The diesel fuel is allowed to soak on the track structure for a short period of time (typically 10-15 minutes), followed by additional removal activities using electric power drills with a wire wheel brush as an attachment.

After the application of diesel fuel and scrubbing, the area is sprayed with a surfactant and additional brushing is performed to remove residual sediment and “tar-like” material. Lastly, Oil Dri ® is applied to the area to absorb any residual diesel fuel and surfactant. The Oil Dri ® is manually rubbed into the area and, following its application, is removed with a shop vac. The debris from the shop vac is placed into containers and handled in the same way as all other waste generated during the process. The washing process is considered complete when no sediment or “tar-like” material is observed on the track structure via visual inspection. All waste generated is disposed of as a TSCA waste. Washing of the concrete track structure is ongoing (as of the preparation of this RIWP) and coincides with the sediment and “tar-like” material removal. As of December 1, 2017, approximately 76% of the track structure has been washed and met the visual inspection standard.

4.2 Bulk Dust Removal

Dust has accumulated on various surfaces throughout PSNY including, but not limited to, the tops of overhead light fixtures, conduits, pipes, brackets, walls, signs, and other surfaces. Clean Harbors initiated dust removal efforts at substations and on platform lighting in conjunction with the "tar-like" material and sediment removal and concrete track structure cleaning operations. Where visually present, dust is removed from surfaces that may be safely reached while standing on a four (4) foot ladder. The dust is removed by a "slow hand wiping" procedure using dampened cloths until no visible dust remains. The dust, and disposable cleaning materials are placed into gaylord bags and transferred to the sub-basement where the waste is temporarily stored (<90 days) before being disposed offsite as a TSCA waste.

4.2.1 Platforms

Dust removal has been completed on facility features such as the tops of overhead light fixtures, conduits, pipes, brackets, walls, signs, and other surfaces on portions of the platforms associated with Tracks, 4, 9, 10, 11, and 16. Dust has been partially removed from the facility features adjacent to Tracks 1, 2, 3, 5, 6, 7, 8, 12, 13, 14, 15, 17, 18, 19, 20, and 21. As of December 1, 2017, approximately 58% of dust from reachable features has been removed from the platforms.

4.2.2 Substations

Clean Harbors removed dust from surfaces that could safely be worked on within Substations E01, 2C, and 43 subsequent to bulk dust sample collection. Surfaces included transformers, equipment, floors, utility conduit and various other surfaces. See Figures 9a through 9c for analytical results.

4.3 Waste Disposal

All waste generated by the project has been disposed of as TSCA hazardous waste. Clean Harbors conducts and manages the waste disposal procedures. Waste from the operations is transferred into an approved container, labeled, and stored onsite at PSNY in the sub-basement for less than 90 days until transported to a disposal facility. Mott MacDonald assists Amtrak with administrative elements associated with hazardous waste disposal for the project. Amtrak and Clean Harbors provide Mott MacDonald with the hazardous waste manifests and Certificates of Disposal. A hazardous waste generation and disposal tracking table is generated and submitted monthly to Amtrak. Hazardous waste disposal documentation is kept on file at PSNY.

4.4 Air Monitoring

Real-time monitoring is performed to monitor and document airborne particulate levels leaving the work zone during track cleaning, dust removal, and SOGR projects. Mott MacDonald's *Air Monitoring Plan* for PSNY is included as Appendix C.

5 Risk Assessment

5.1 Relevant Guidance and Policies

As previously stated, PSNY has been an operating passenger train station since the beginning of the 20th Century and will continue in this capacity into the foreseeable future. It is a critical component of Northeast Corridor passenger rail service through NYC. Rail and passenger platform areas will not be re-developed for residences, daycare, health facilities, or other "unrestricted uses". In fact, access to many areas of PSNY other than the Upper and Lower Concourses, and the Passenger Platform section of the Platform/Track Level is highly restricted to only Amtrak employees and authorized contractors, while other areas are limited to passengers boarding and departing from trains as will be discussed further in this RIWP.

Although the RI described in this RIWP has many of the elements of a conventional site investigation, PSNY is not a conventional open-air site and the exposure media of interest are focused on the operational aspects of the train operations and function of the station for passenger transit and supporting facilities. Traditional environmental media (soil, groundwater, surface water, and outdoor ambient air) as envisioned by the methods of EPA Risk Assessment Guidance for Superfund (RAGS) are not present within PSNY as it is entirely underground. Although the Upper and Lower Concourses are within the general category of indoor spaces, the Passenger Platforms, and non-public Restricted Areas of PSNY are physically distinct and have unique characteristics.

Regulations, policies, and guidance that may apply include, but are not limited to, NYSDEC 6NYCRR Part 375 and associated guidance; TSCA (1976), section 761.61; and Risk Assessment Guidance for Superfund (RAGS), Volume I: Parts A, B, E, and F.

Section 761.61 of TSCA provides three options for managing PCB contaminated sites. Section 761.61 (a) is self-implementing; Section 761.61 (b) is performance-based; and Section 761.61 (c) provides for risk-based remediation. Section 761.61 (a) of TSCA prescribes cleanup levels for high and low occupancy areas based on a pre-determined number of hours per week a person would be assumed to have contact with the PCBs on the site. These somewhat arbitrary designations may or may not coincide with real-world activity patterns, particularly on complex sites such as PSNY. The limitations of applying the self-implementing procedures of 761.61(a) are acknowledged in the regulations. Under 761.61(a) the regulation states: "The procedure may be less practical for larger or environmentally diverse sites. For these other sites, the self-implementing procedure still applies, but a USEPA Regional Administrator may authorize more practical procedures through paragraph (c) of this section". Although some areas of the Site may be considered "low occupancy" because people are seldom present, the concepts and definitions of high and low occupancy, as well as the prescribed cleanup levels as defined by Section 761.61 (a) of TSCA, are not relevant to a risk-based approach as described in Section 761.61 (c) of TSCA.

TSCA 761.61(c) provides an option for risk-based cleanup at complex sites where the prescriptive self-implementing options are not appropriate. If an entity wishes to use a risk-based approach for PCB remediation, rather than the options in 761.61(a) and (b), the entity must submit an application for approval to the USEPA Regional Administrator. The application to the USEPA Regional Administrator, requesting approval for a risk-based cleanup, must demonstrate "a commensurate level of protection for human health and the environment" as provided by the self-implementing levels (implied). TSCA does not define protection for human health and the environment.

Although TSCA 761.61(c) provides for risk-based cleanup, the regulations do not prescribe the approach, primarily because TSCA pre-dates the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the resulting promulgation of guidance documents and directives. The USEPA PCB Site Revitalization Guidance under TSCA (November 2005) defaults to Office of Solid Waste and Emergency Response (OSWER) risk assessment guidance for Superfund. Therefore, risk assessment guidance developed under CERCLA (USEPA, 1990 and 2005) is applicable to TSCA 761.61(c).

It is important to understand that PSNY plays a critical role in passenger rail service on the Northeast Corridor. There are no uncertainties about how this property will be used in the future. Amtrak will control PSNY into the future and has plans to continuously occupy the property for passenger transit and railroad operations.

5.2 Conceptual Site Model

The Conceptual Site Model (CSM) for the Human Health Risk Assessment (HHRA) is an analysis and representation of the physical pathways by which chemicals of potential concern move from source(s) to locations where people (receptors) may come into direct physical contact with the media containing those chemicals through inhalation, ingestion, and skin contact (dermal contact or absorption through skin). The CSM is a tool to organize data gathering prior to initiating an investigation and to communicate the findings. The CSM for PSNY is a "living analysis" and will be refined as additional data are collected and the investigation proceeds.

The evaluation of potential risks from exposure to contaminants in the environment hinges on the concept of completed pathways of exposure. Exposure pathways describe the ways by which a chemical (or physical or biological) agent can move from a source to a receptor (USEPA, 1989). Exposure means direct physical contact between the receptor (person) and the environmental media (e.g. soil, groundwater, or air) that contains the chemical(s). Four elements must be present for an exposure pathway to be complete:

1. A source of the contaminant and a mechanism by which the chemical(s) is released into the environment;
2. Mechanisms by which the chemical or the environmental media containing the chemical(s) can be transported to locations where receptors are located;
3. A point or points of contact where the receptors and the environmental media containing the chemical(s) are both present at the same time; and
4. A route or routes of exposure by which the receptor gets the environmental media containing the chemical(s) into their body (e.g. ingestion, inhalation, dermal contact with absorption through the skin).

If any of these elements is absent, the pathway is not complete and the person does not take the chemical into their body. Even if all the requirements are satisfied for a complete pathway of exposure, the amount of the chemical (the dose) that gets into the person's body must be sufficient to cause an adverse effect or increase the chances of an adverse effect occurring above levels of concern.

Pathways and routes of exposure are depicted on the CSM for each of the receptors proposed to be evaluated in the HHRA. Pathways are identified as complete, potentially complete but insignificant, or incomplete. As used in the CSM, "Not Applicable" indicates that the receptor is not physically present.

Complete Pathways of Exposure: There is reasonable evidence to suggest that the receptor comes into direct physical contact with the media containing the chemical(s) and internalizes a dose. Complete pathways of exposure will be evaluated quantitatively in the HHRA.

Potentially Complete but Insignificant Pathways of Exposure: Reasonable evidence suggests that if direct contact with the media containing the chemical(s) does occur, the contact is very limited in frequency and/or intensity, and is unlikely to contribute significantly to the receptor's internalized dose and subsequent risk. Potentially Complete but Insignificant Pathways of Exposure will be evaluated qualitatively.

Incomplete Pathways of Exposure: Either the chemical(s) do not occur in the pathway (medium) and/or the receptor is not in direct contact with the medium. Incomplete pathways of exposure will not be further evaluated.

The overall Conceptual Model for PSNY separates the station into two main regions; the Passenger Envelope and all other Restricted Areas of the station.

The Passenger Envelope is defined by both physical boundaries and the receptors who are present within those boundaries. The Passenger Envelope is the public region of PSNY consisting of the Upper and Lower Concourses, the Passenger Platforms, and the passageways (stairs, escalators, and elevators) connecting these three levels. A *Screening Level Evaluation of the Passenger Envelope* has been conducted by Stantec and is included as Appendix D to this RIWP. Table 1 presents the *Conceptual Site Model for the Passenger Envelope.*

The HHRA described in this section will address potential exposures to receptors in the Restricted Areas of PSNY. For this project, Restricted Areas are those locations within PSNY where the public is not allowed and access is permitted only for railroad workers and other authorized individuals. The Restricted Areas include, but may not be limited to the concrete tracks, stone ballast track areas to the east and west of the concrete tracks, staging areas for equipment and out of service trains, electrical substations, and limited access locations on the Platforms (e.g. employee offices, locker rooms, and break rooms). Table 2 presents the *Conceptual Site Model for the Restricted Areas of PSNY.*

5.3 Selection of Chemicals of Potential Concern

PCBs are the chemicals of concern for PSNY; however, other chemicals of potential concern (e.g., heavy metals, PAHs, etc.) may be identified from analysis of select samples collected during the RI. As described in Section 6.5.5 and in Table 3 *Sample Analytical Methods and Frequency*, select samples will be analyzed for Target Compound List plus a 30-compound library search (TCL+30) and Target Analyte List (TAL) analyses.

The maximum detected concentrations of chemicals measured in media sampled during the RI will be compared to the USEPA Regional Screening Levels (RSLs) for soils on industrial properties and NYSDEC Restricted Use Soil Cleanup Objectives (SCO) (Table 375-6.8(b)). Chemicals detected at concentrations equal to or higher than the lower of the two screening levels (RSL or Restricted Use SCO) will be identified as chemicals of potential concern and quantitatively evaluated in the HHRA for the Restricted Areas of PSNY.

Unless the findings of the RI indicate otherwise, PCBs are the chemical of concern for the Passenger Envelope.

5.4 Exposure Assessment

Exposure is defined by the USEPA risk assessment guidelines as the contact of a receptor with a chemical or physical agent (USEPA, 1989 and 1992) or the media containing that agent. The goal of the exposure assessment is to identify potential exposure pathways and to estimate the intake of chemicals received by potentially exposed populations (National Research Council 1983; USEPA, 1992). Exposure assessment involves estimating human exposures from multiple routes, for example, ingestion, dermal contact, and inhalation (USEPA, 1989 and 1992) using a combination of direct measurements and mathematical models.

The exposure assessment is a detailed evaluation of the receptors and pathways of exposure depicted in the CSM.

5.4.1 Receptors and Pathways of Exposure for the Passenger Envelope

A *Screening Level Evaluation* (Appendix D) for receptors in the Passenger Envelope was conducted based on PCB concentrations measured in air samples collected from the Passenger Platforms, the Lower Concourse, and the Upper Concourse. Samples of accumulated dust collected from on top of light fixtures on the Passenger Platforms and analyzed for PCBs were used to evaluate potential direct contact with accessible surfaces. The receptors evaluated for the Passenger Envelope are employees of Amtrak, New Jersey Transit, LIRR and vendors who work on the Upper and Lower Concourses; railroad workers with job duties on the Platforms; housekeepers who clean on the Upper and Lower Concourses and on the Platforms; police and station security personnel; casual visitors to PSNY; and train passengers. These receptors are a representative cross-section of the potential receptors for this area.

Concentrations of PCBs measured in air on the Upper and Lower Concourses and the Passenger Platforms; and concentrations of PCBs in dust collected from the top of light fixtures on the Platforms were evaluated. The Passenger Envelope *Screening Level Evaluation* prepared by Stantec is attached as Appendix D. It is Amtrak's intent to conduct quarterly synoptic air monitoring for PCBs on the Upper and Lower Concourses and Passenger Platforms while the RI is in process. New data from each sampling event will be assessed in the context of the screening level evaluation.

5.4.2 Receptors and Pathways of Exposure for Restricted Areas

The receptors that will be evaluated in the HHRA for the non-public Restricted Areas of PSNY represent classes of Amtrak employees who have regular job duties outside of the public areas of the Passenger Envelope. Contractors may also be present and authorized to work on specific projects in Restricted Areas of PSNY. Exposures to contractors will be represented by the classes of Amtrak employees.

The media containing chemicals of concern in the Restricted Areas are particulates (dusts) and vapors in air, (PCBs) embedded in concrete tracks and associated structures, dust on structural surfaces and equipment, sediments, and ballast fines and ballast stone in the tracks. Amtrak employees are assumed to have direct contact with chemicals of concern through inhalation of vapors and particulate (dusts) in air; skin contact with dusts on surfaces, ballast fines, ballast stone, and sediments, with subsequent absorption of lipophilic chemicals such as PCBs through the dermis; and incidental ingestion of chemicals of concern on surfaces through hand to mouth transfer.

Table 4: Receptors and Pathways of Exposure for Restricted Areas

Amtrak Employee Classification	Activities/Job Duties	Potential Pathways & Routes of Exposure	Estimated Exposure Frequency
Transportation Workers, Conductors, Locomotive Engineers	Brief inspection of locomotives and railcars; other brief tasks at track level. May walk across tracks and stone ballast when entering or departing trains at beginning and end of shift.	• Inhalation • Skin contact with outside of railcars, handrails & concrete surfaces • Hand to mouth transfer of dust	0.5 hours/day, 250 days/year
Mechanical Department	Inspect and repair train equipment.	• Inhalation • Skin contact with train equipment, handrails, tools, concrete surfaces • Hand to mouth transfer of dust	1 hours/day, 250 days/year
Division Engineering (1)	Track inspection; communication & signals; catenary & 3rd rail maintenance; walking on ballast.	• Inhalation • Skin contact with handrails, concrete surfaces, tools & equipment, sediments & ballast fines, ballast stone • Hand to mouth transfer of dust	8 hours/day, 250 days/year

Amtrak Employee Classification	Activities/Job Duties	Potential Pathways & Routes of Exposure	Estimated Exposure Frequency
Division Engineering (2)	Track repairs; wood tie replacement; breaking concrete near platforms; intrusive repairs & replacements; communications & signal repairs.	• Inhalation • Skin contact with handrails, concrete surfaces, tools & equipment, sediments & ballast fines, ballast stone • Hand to mouth transfer of dust	8 hours/day, 20 days/year
Production Engineering	Major construction projects; rebuild track; replace switches; walking on, or digging in ballast; catenary & 3rd rail construction.	• Inhalation • Skin contact with ballast, fines & sediments, concrete surfaces, tools and equipment, sediments and ballast fines, ballast stone • Hand to mouth transfer of dust	12 hours/day, 20 days/year

It should be noted that even though workers are expected to be present in these areas, an individual may not have actual physical contact with surfaces or solid media, and therefore the assumptions about frequency and duration of exposure are conservative, and more likely to overestimate rather than underestimate exposure.

5.4.3 Exposure Point Concentrations

The Exposure Point Concentration (EPC) is the concentration of a chemical of potential concern that could be contacted by a receptor over the exposure period. The EPC can be estimated using sampling data and/or chemical transport and environmental fate models. It is anticipated that EPCs will be derived from empirical data for all media evaluated during the RI, including air. For the PSNY Restricted Area HHRA, EPCs will be derived from validated laboratory analytical results using the most recent version of USEPA (2015) ProUCL statistical software available at the time the HHRA is conducted. The current version of ProUCL (version 5.1.00) was published in 2015 (USEPA 2015). Time trends in air concentrations of PCBs, if any, will be considered when deriving EPCs for inhalation.

5.4.4 Quantification of Exposure

USEPA risk assessment algorithms will be used to estimate receptor daily intake of chemicals of potential concern from each complete pathway of exposure (USEPA, 1989; 2011; 2014; and 2017). Chronic daily intake (CDI) is derived from the EPC for each chemical of potential concern and is expressed in units of milligrams of chemical per kilogram of body weight per day (mg/kg-day). Soil in the traditional sense is not expected to be a medium of exposure for receptors in PSNY, although soil may be present in some areas. Settled dust on surfaces, ballast fines and sediments in the track areas (collectively “solids”) will be evaluated during Risk Assessment. Alternative methods will be considered to evaluate exposures to concrete chips and ballast stone. The concentrations of PCBs directly measured in air will be used to evaluate inhalation exposures.

5.4.5 Incidental Ingestions of Solids

Chronic daily intake of chemicals of potential concern from incidental ingestion of solids as settled dusts, fines and soils will be estimated using the following equation developed for ingestion of soils (USEPA, 1989):

$$CDI_{ca} \text{ or } CDI_{nc} = \frac{(C_{solids} \times IR \times CF \times EF \times ED)}{(BW \times AT)}$$

Where:

CDI_{ca} = Lifetime average daily dose for carcinogenic effects, averaged over a lifetime of 70 years, in mg/kg-day;

CDI_{nc} = Average daily dose for non-carcinogenic effects, averaged over the exposure duration, in mg/kg-day;

C_{solids} = Concentrations of chemical of potential concern in solids, in mg/kg

IR = Particle ingestion rate (mg/day);

CF = Conversion factor, 1E-06 kilograms per milligram (kg/mg);

EF = Exposure frequency, in days/year;

ED = Exposure duration, in years;

BW = Average body weight, in kilograms (kg); and,

AT = Averaging time, in days; equals 70 years x 365 days/year for carcinogenic effects and ED x 365 days/year for non-carcinogenic effects.

5.4.6 Dermal Contact with Solids

The CDI from dermal contact with chemicals of concern in solids and subsequent absorption into the bloodstream will be estimated using the following equation for dermal exposure to soils (USEPA, 2004):

$$CDI_{ca} \text{ or } CDI_{nc} = \frac{(C_{solids} \times CF \times SA \times SAF \times ABS_d \times EF \times ED)}{(BW \times AT)}$$

Where:

CDI_{ca} = Chronic daily intake (dose) for carcinogenic effects, averaged over a lifetime of 70 years, in mg/kg-day;

CDI_{nc} = Average daily dose for non-carcinogenic effects, averaged over the exposure duration, in mg/kg-day;

C_{solids} = Concentration of chemical of potential concern in solids, in mg/kg;

CF = Conversion factor, 1E-06 kg/mg;

SA = Skin surface area for soil contact, in square centimeters per day (cm^2/day);

SAF = Soil adherence factor, in milligrams per square centimeter (mg/cm^2);

ABS_d = Chemical-specific dermal absorption factor (unitless);

EF = Exposure frequency, in days/year;

ED = Exposure duration, in years;

BW = Average body weight, in kilograms (kg); and,

AT = Averaging time, in days, equals 70 years x 365 days/year for carcinogenic effects and ED x 365 days/year for non-carcinogenic effects.

5.4.7 Inhalation of Chemicals of Concern in Air

The following equation will be used to calculate the Exposure Concentration (EC) of chemicals of potential concern in air (USEPA, 2009):

$$EC = \frac{CA \times ET \times EF \times ED}{AT}$$

Where:

EC = Exposure concentration in air (mg/m^3);

CA = Concentration of chemical of potential concern in air (mg/m^3);

ET = Exposure time (hours/day);

EF = Exposure frequency (days/year);

ED = Exposure duration (years); and

AT_c = Averaging time for cancer (lifetime in years x 365 days/year x 24 hours/day)

AT_{nc} = Averaging time for non-cancer effects (ET x EF x ED)

5.5 Approach to Characterizing Risk

Cancer risk and non-cancer hazard from exposure to chemicals of potential concern in air and other media in PSNY will be estimated using standard equations in USEPA RAGS (USEPA, 1989; 2004 and 2009).

5.5.1 Cancer Risk

Cancer risk from incidental ingestion and dermal absorption of chemicals will be estimated using the following formula.

$Cancer\ Risk_{(ingestion)} = CDI_{ca} \times SF$

$Cancer\ Risk_{(dermal)} = CDI_{ca} \times (SF/GIABS)$

Where:

CDI_{ca} = Lifetime average daily dose, averaged over a lifetime of 70 years, in mg/kg day

SF = Cancer Slope factor, in (mg/kg day)-1

GIABS = Gastrointestinal Absorption Factor

For multiple chemical or mixture exposures, the total Cancer Risk for a given receptor is conservatively estimated by summing the Risks for all chemicals of potential concern for each exposure route, using the following simple additive equation (USEPA, 1989):

$$\text{Multiple Substance Risk} = \sum_{i=1}^{N} \text{Risk}_i$$

Where:

Multiple Substance Risk =Total CR from multiple substances, unitless probability

$Risk_i$ = CR for the i^{th} chemical (a total of N).

Cancer risk from inhalation of chemicals of potential concern in air is calculated by multiplying the Exposure Concentration in air (EC) by the Inhalation Unit Risk (IUR).

Cancer Risk = EC x IUR

The inhalation risk from multiple chemicals of potential concern is assumed to be additive for each receptor.

5.5.2 Non-Cancer Hazard

Potential non-cancer hazard from exposure to a chemical of potential concern in environmental media is expressed as the Hazard Quotient (HQ) and is calculated as a simple ratio using the following equation (USEPA, 1989).

$$HQ = \frac{CDI_{nc}}{RfD}$$

Where:

CDI_{nc} = Chronic Daily Intake for non-cancer health effects, in mg/kg day;

RfD = Chronic or sub-chronic reference dose, in mg/kg day.

Non-cancer hazard from exposure to multiple substances is quantified as a hazard index (HI), which is the sum of all possible chemical-specific HQs (USEPA, 1989):

$$\text{Hazard Index} = \frac{ADD_1}{RfD_1} + \frac{ADD_2}{RfD_2} + \ldots \frac{ADD_i}{RfDi}$$

For any given receptor, the total HI from all exposure pathways is assumed to be additive, as indicated in the following equation (USEPA 1989):

$$\text{Total HI} = HI_{pathway1} + HI_{pathway2} + \ldots HI_{pathwayi}$$

Non-cancer hazard from inhalation of chemicals of potential concern in air is calculated by dividing the Exposure Concentration in air by the Reference Concentration.

HQ = EC ÷ RfC

While the cancer risk assessment methodology does not generally distinguish between the types of cancers associated with individual chemicals, the methodology for evaluating non-cancer hazard provides the option for separating hazard estimates by chemicals according to the target organ where the critical effect (basis for the RfD or RfC) is produced. For example, hazard can be evaluated separately for chemicals that produce a critical effect on the liver vs. the immune system or kidneys.

5.6 Risk Based Remedial Goals

Risk-based remedial goal concentrations of chemicals of potential concern may be derived for representative receptors within PSNY using the Oak Ridge National Laboratory Risk Assessment Information System (ORNL-RAIS) on-line calculator tool. The on-line calculator has embedded USEPA default values, some of which can be modified to better match receptor characteristics. Risk based remedial goals will be evaluated in the RI (USEPA, 1991).

5.7 Uncertainties and Mitigating Factors Relevant to PSNY

The approach to the HHRA for the Restricted Areas of PSNY is based on USEPA methodology and is inherently conservative - meaning that exposures and subsequent cancer risks and non-cancer hazards are more likely to be overestimated rather than underestimated. Uncertainties associated with the variable values used in standard equations to estimate internal dose, extrapolation of toxicity factors derived from laboratory animal studies to humans, and representativeness of the sampling scheme and analytical data are common elements in most, if not all, risk assessments for contaminated sites. Some sources of uncertainty specific to evaluating risks for receptors within PSNY include, but are not limited to the following:

- PSNY is not a conventional open-air site and the exposure media of interest are focused on the operational aspects of the train operations and function of the station for passenger transit and supporting facilities. Traditional environmental media (soil, groundwater, surface water, and outdoor ambient air) as envisioned by the methods in EPA RAGS are not present within PSNY. PSNY is entirely underground. Although the Upper and Lower Concourses are within the general category of indoor spaces, the Passenger Platforms, and non-public Restricted Areas of PSNY are physically distinct and have unique characteristics.

- Samples of settled dust, ballast fines, ballast stone, sediments and concrete will be evaluated in Risk Assessment. This is a source of uncertainty because the evaluation of chemicals of potential concern in these solid media is likely to be different from the model assumptions used in RAGS methodology. For example, ballast stone and concrete are not analogous to soil as RAGS methodology assumes, and evaluation of exposure from contact with these solid materials is expected to be potential uncertainty consideration.

- The HHRA considers the activity patterns of workers within PSNY based on employee job descriptions provided by Amtrak. This reduces the uncertainty for frequency of exposure used to estimate exposure for individual categories of Amtrak employees.

6 Remedial Investigation Approach

The RI is being undertaken to further investigate the extent of impacts present in media at PSNY. The primary focus of the RI will be to determine the extent of PCBs in media at PSNY. Other target compounds will be evaluated as described in this RIWP to confirm PCBs are the primary COC at PSNY. The information gathered will be used to evaluate and develop a remedial strategy that is protective of human health and the environment. Please Note: sampling activities are ongoing concurrent with the preparation of this RIWP, however, the descriptions provided below are presented as future sampling activities. This section summarizes the additional proposed sampling to evaluate various media present at PSNY. The media to be sampled include ballast fines, ballast stone, concrete, soil, sediment, dust and air. To ensure that quality assurance and DQOs are met, the RI will be completed in accordance with the site-specific QAPP (Appendix E).

6.1 Physical Characterization Activities

Samples will be collected during periods of planned track outages, periods of availability, and when access to the various AOIs can be coordinated with appropriate worker protection. Such access will be limited in extent and restricted by worker safety and logistics. PSNY is the busiest passenger train station in the country, with each of the station's 21 tracks in use every 2 minutes on weekdays and rail activity occurring 24 hours a day, 365 days a year. Consequently, there will be logistical challenges with scheduling and implementing sampling events. As a result, all samples will not be collected in a single mobilization, rather sampling will occur as access becomes available. The information collected will be summarized in an RI Report for PSNY.

Sampling on the Track Level, Platforms and other areas of PSNY will be completed with Amtrak protection. Sampling on the Track Level will occur only when the tracks have been taken out of service in accordance with Amtrak procedures. Sampling personnel will be accompanied by Amtrak personnel at all times during sampling. All field personnel will have completed Amtrak safety training and have a valid safety card. Sampling personnel will don appropriate PPE as stated in Amtrak's approved Site-Specific Safety Work Plans. The procedures for sample collection and handling is summarized by media below.

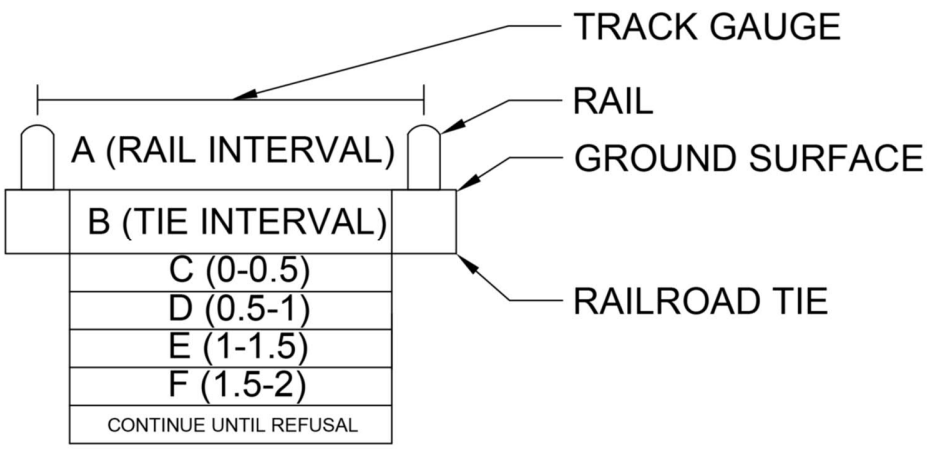
A *Sampling and Analysis Plan (SAP) at Areas of Interest 1 through 6* was prepared in a document dated July 21, 2017. The SAP is fully incorporated into this RIWP and, as such, information is consolidated negating the need for a stand-alone SAP document.

6.1.1 Ballast Fines

In areas outside of the footprint of the concrete track structure (AOI-1), the track structure is generally comprised of ballast fines and ballast stone. This includes areas in AOIs 2 through 6. Ballast fines are a fine-grained material that is comingled with the ballast stone. The percentage of ballast fines compared to ballast stone varies along the track structure and at times, ballast fines are not present.

Samples of the ballast fines will be collected from inside the center of the track gauge; the likely location of PCB deposits from leaking transformers. Samples will be collected via test holes installed by manual methods at a rate of one per 25 linear feet. The proposed sampling locations are presented on Figure 10. At 25-foot intervals, material encountered within three vertical zones (rail interval, tie interval, and 0-0.5 feet below the tie) most frequently encountered by Amtrak employees will be inspected for ballast fines. For clarification purposes, the “rail interval” corresponds to, and will be defined as the vertical interval above the tie. The “tie interval” corresponds to the vertical interval from the top of the tie to the bottom of the tie. A sample will also be collected at the interval of 0-0.5 feet below the tie. See the sampling interval diagram for a visual depiction of proposed sampling depth intervals below and included as Appendix A.

Sampling Interval Diagram:



The material at each sample depth will be inspected for the presence of ballast fines. If a sufficient amount of ballast fines is present at the target depth interval, a sample will be collected for analysis and transferred directly into laboratory provided containers and stored on ice in a cooler. If ballast fines are not present, no ballast fines sample will be collected for analysis. Sample collection times, depths and locations will be recorded in a project dedicated field book.

In addition, at every 150 feet of linear track, samples will be collected at every six-inch depth interval below the bottom of the tie until refusal is encountered. Based on previous sampling, it is assumed that refusal will be encountered at approximately 0 to 3 feet below the bottom of the tie.

The test holes will be excavated first to a pre-determined depth (or refusal if encountered) and then sampled from sidewall starting with the deepest interval upwards. The test holes will be excavated using a shovel, post hole digger and/or equivalent hand tools. The hand tools will be decontaminated prior to, and in between each sampling location. Decontamination procedures are described in Section 6.4 and in the QAPP included in Appendix E. Samples will be retrieved from respective intervals using dedicated pre-cleaned stainless steel scoopulas or trowels. A scoopula or trowel will be used to dress the area prior to sample collection. A separate scoopula will be used to transfer material directly into laboratory provided containers to be stored on ice in a cooler. Scoopulas used to dress the area will not be used to transfer sample material. The sampling will be completed in general accordance with TSCA 761.61 subpart N. Analysis will be performed on ballast fines samples as described in Section 6.5.5 and Table 3. All sampled locations with analytical results will be mapped. Proposed ballast fines sample locations are presented on Figure 10.

6.1.2 Ballast Stone

Railroad ballast stone is present in the track structure at AOIs 2 through 6. One ballast stone sample will be collected from the surface at every 6th sampling location (every 150 feet of linear track) and submitted for analysis. Ballast stone samples will be co-located with ballast fines samples and collected according to the sampling procedure outlined in 6.1.1. The samples will be collected from the center of the of the track gauge. Sample collection times and locations will be recorded in a project dedicated field book. Care will be taken to avoid the collection of ballast fines with ballast stone samples. The sample will be crushed by the laboratory prior to analysis. Analysis will be performed on ballast stone samples as described in Section 6.5.5 and Table 3. All sampled locations with analytical results will be mapped. Proposed ballast stone sample locations are presented on Figure 10.

6.1.3 Concrete

Amtrak is currently having the sediment and “tar-like” material removed from the concrete track structure in AOI-1. After the sediment and “tar-like” material has successfully been removed, the tracks are washed with diesel fuel followed by a surfactant. Oil-Dri® is then applied to complete the wash process. After the tracks have been washed, concrete samples will be collected from the concrete track structure to document the remaining concentrations of PCBs. The proposed concrete sampling locations are shown on Figure 11.

Concrete samples will be collected at a rate of one per 100 linear feet of track structure. The samples will be collected from the center of the trough in the middle of the track gauge where the greatest potential for PCBs to be present exists. Since the sediment and “tar-like” material is present along most of the length of the concrete track structure, the 100 linear feet sampling scheme is considered adequate to document the remaining concentration of PCBs. If a drain is encountered in the track structure, an additional concrete sample will be collected adjacent to the drain. Samples collected around drains would document any variations in the distribution of impacts compared to samples collected away from drains.

Concrete samples will be collected from the surface using a battery powered pneumatic impact hammer and chisel bit to remove an area of concrete approximately 4 by 4 inches and to a depth of approximately 1/4 inch in thickness. Chisel bits will be decontaminated prior to use according to the method described in Section 6.4 and in the QAPP included as Appendix E. The resulting sample will be transferred into laboratory provided containers using a dedicated pre-cleaned stainless steel scoopula and/or trowels and stored on ice in a cooler. Sample collection times and locations will be recorded in a project dedicated field book. The sample will be crushed by the laboratory prior to analysis as described in the QAPP (Appendix E). Analysis will be performed on concrete samples as described in Section 6.5.5 and Table 3. All sampled locations along with results will be mapped.

6.1.4 Soil

Soil is present in a few limited areas adjacent to Track 1 (on the south side) and West of Platforms 1 and 2. Soil adjacent to tracks will be sampled from the A, B and C intervals (Appendix A) according to the procedure and frequency outlined in Section 6.1.1 (Ballast Fines Sample Collection). Samples will also be collected from deeper intervals every 150 linear feet until refusal is encountered. Soil encountered in yard areas outside the track gauge will be sampled from the A, B and C intervals (Appendix A) according to the procedure outlined in 6.1.1 at a rate of one per 900 square feet of area. Samples will also be collected in yard areas outside the track gauge from deeper intervals until refusal is encountered at every other location. The proposed soil sampling locations are shown on Figure 12. Sample collection times and locations will be recorded in a project dedicated field book. Analysis will be performed on soil samples as described in Section 6.5.5. Please note if soil is encountered in additional areas during the RI, the proposed sampling scheme will be followed and sampled locations along with analytical results will be mapped.

6.1.5 Sediment in Drains

Drains are present in the center of the concrete track structure within AOI-1 as well as at other areas of the Track Level. The drains are connected to sumps and discharge to the New York City sanitary sewer system. Drains may contain sediment or fine-grained materials. As part of the RI, each drain will be inspected by removing the cover. If 12 or more inches of sediment is present, two (2) samples will be collected. One (1) sample will be collected from the top 6 inches and one (1) from the bottom 6 inches of sediment. If less than 12 inches of sediment is present within a manhole, one (1) sample will be collected from the bottom six inches of sediment. Sediment will be retrieved from the drain manholes and placed on plastic sheeting using hand tools such as post hole diggers, shovels or hand augers; whichever is more efficient. A dedicated, pre-cleaned stainless steel trowel or scoopula will be utilized to transfer the sample directly into laboratory provided containers and stored on ice in a cooler. The depth, location and collection times of the samples will be recorded in the field book. Analysis will be performed on sediment samples collected from drains as described in Section 6.5.5 and Table 3. Any material removed from the drains that is not sampled will be disposed of as a TSCA waste. The location of the drains and analytical results will be mapped.

6.1.6 Dust

Bulk dust samples will be collected at proposed locations (Figure 13) where a sufficient mass (Section 6.5.5) is present for analysis. At each sample location, the area will be visually inspected to identify a surface with a sufficient mass of dust. Depending on the location and area of the station, surfaces may include; top of overhead light fixtures, de-energized 3rd rail cover, columns, wires or conduit and other equipment. Each bulk dust sample will be collected using dedicated, pre-cleaned stainless steel scoopulas. The dust will be transferred directly to laboratory provided containers. Containers will be placed on a scale in the field, tared and filled with bulk dust during sample collection until the required minimum mass has been collected. Once a sufficient mass of bulk dust has been collected, the containers will be sealed, labeled and stored on ice in a cooler. The location and collection times of the samples will be recorded in the field book. Analysis will be performed on bulk dust samples as described in Section 6.5.5 and Table 3. Sampled locations and analytical results will be mapped.

6.1.7 Air

Indoor air samples will be collected at the Upper Concourse, Lower Concourse, and Platform/Track Levels of PSNY. Ten (10) Platform Level sample locations have been designated, which include the east and west sides of Platforms 2, 4, 6, 8 and 10 by stairs leading up to the Upper and Lower Concourses. Ten (10) Lower Concourse sample locations have been designated, which include five (5) exit Concourse (by entrances to Tracks 3/4, 7/8, 11/12, 15/16 and 18/19), two (2) Central Concourse (by entrances to Tracks 15/16, and 18/19), and three (3) Main Gate Areas (by tracks 11/12, 15/16, and 18/19). Five (5) Upper Level sample locations have been designated, which include three (3) Amtrak Concourse (by entrance to tracks 7/8, 11/12, and 15/16), and two (2) New Jersey Transit Concourse (by entrance to tacks 3/4 and 7/8). The air sample locations are presented on figures included in Appendix B. Quarterly events are planned for the Platform/Track Level and the Lower and Upper Concourse areas. The proposed air sample locations at the Track level are presented on Figure 13. Air samples will be collected according to the QAPP (Appendix E) and TRC's *PCB in Air Sampling Plan – Penn Station* dated September 5, 2017 (Appendix B).

6.2 Mapping

Sample locations will be recorded by field personnel relative to fixed points located on the Platforms. The fixed reference points are benchmarks located on the Platforms relative to Railroad 0. Amtrak has indicated that milepost "zero" at PSNY is located on a concrete beam between signal 115W and 520E above Track 11. Amtrak uses measurements east and west of the milepost "zero". The "zero" location for each platform has been marked in the field. The sampling locations will be measured from these established "zero" locations. East of point "zero" will be denoted with an "E" and west of point "zero" will be denoted with a "W". For example, a location 100 feet west of point "zero" on a track will be referred to as "W100".

Benchmarks were established on the east and west ends of each Platform. The locations were physically marked by a nail driven into the concrete on the platforms. The benchmarks establish points with a fixed location. As samples are collected, the locations and surface features (as deemed necessary) will be measured using the established benchmarks as reference. Additional benchmark locations are planned and will be mapped. For further information regarding the mapping and Railroad 0, see Mott MacDonald's Survey Plan dated March 3, 2017 included in the QAPP as Appendix E. The benchmark locations are presented on Figure 14.

6.3 Data Quality Objectives

Data Quality Objectives (DQOs) define the purpose of data collection, clarify what the data should represent to inform the decisions to be made, and specify the performance requirements for the quality of the information to be obtained from the data (USEPA 2006). DQOs are qualitative and quantitative statements developed to ensure that data collected are of known and appropriate quality for the purposes for which they are intended. If the sampling programs change, the DQOs may need to be modified or supplemented.

PCBs were initially detected in sediment located on the concrete track structures adjacent to platforms at PSNY. The results of sample analysis will be used to evaluate the site conditions for future risk assessment, feasibility, and compliance with the TSCA regulations. The sampling program objectives is to determine the nature and extent of PCB contamination.

The data quality indicators (DQI) precision, accuracy, representativeness, completeness, comparability, and sensitivity will be used to evaluate DQOs. The DQOs ensure the collection of the appropriate quantity of samples based on both the technical regulatory requirements and the need to develop a reasonable conceptual model. As appropriate the DQOs were developed in accordance with the USEPA Guidance on Systematic Planning using the Data Quality Objectives Process (USEPA QA/G-4, February 2006). A QAPP which further describes the DQOs is included as Appendix E.

6.4 Decontamination Procedures

Equipment decontamination is needed to prevent the potential or likelihood of cross-contamination during sample collection. Field equipment falls into two categories consumable and reusable. Consumable field equipment is dedicated equipment that is immediately disposed of once it is used. Consumable equipment includes trowels, scoopulas and gloves used in sample collection for this project. The consumable equipment is exclusively used at a sample location/depth and then disposed of as a TSCA hazardous waste. Consumable equipment will not be reused.

Reusable equipment consisting of hand tools, post hole digger shovels and other equipment that is reused at multiple sampling locations. Equipment that is reused will be decontaminated between sampling locations using Simple Green®. The Simple Green® is applied to the equipment and cleaned using a dedicated brush. The equipment is then rinsed with de-ionized water. All liquids used for decontamination will be collected and disposed of as TSCA hazardous waste. This method is the most feasible option as Amtrak's safety policy does not permit the use or storage of any flammable solvents or volatile organic compounds in PSNY. Decontamination procedures are further described in the QAPP which is included in Appendix E.

6.5 Sampling and Analysis Procedures

All samples will be collected according to the procedures specified in Section 6.1 and the QAPP (Appendix E).

6.5.1 Sampling Nomenclature

Samples of solid material (e.g. ballast fines, and ballast stone) collected in the track areas will be identified/numbered according to the following convention:

- AOI#-Track#-Sample Location-Interval #-Media
    - AOI# – Refers to Area of Interest 1-6
    - Track# – Refers to track number or designation
    - Sample Location – Direction followed by distance in feet from Railroad 0. E indicates to the East and W indicates to the West.
    - Interval#
        - A – rail interval
        - B – tie interval
        - C – 0–0.5 feet below the tie
        - D and so on every 0.5–foot interval until refusal
        - CS – Concrete sample from surface
        - CD – Concrete sample from below surface (note depth)
    - Media
        - A – Air

- BF – Ballast Fines
- BS – Ballast Stone
- C – Concrete
- CD – Concrete adjacent to Drain
- D – Dust
- S – Soil
- SD – Sediment within Drain

As an example, a sample collected from AOI 5, on Track 10, 555 feet west of Railroad 0, at the tie interval, and of ballast stone, would be identified according to the following convention:

- AOI5–Track10–W555–B–BS

Field duplicate samples of solid material collected in the track areas will be identified by the inclusion of "DUP" after the track number. A duplicate of the above example collected on November 30, 2017 would be identified according to the following convention:

- AOI5–Track10–DUP–113017–B–BS

Field blank samples (aqueous) will be identified/numbered according to the following convention:

- AOI#–Track#–FB–date collected

As an example, a field blank collected during AOI 5, Track 10 sampling activities occurring on November 30, 2017 will be identified according to the following convention:

- AOI5–Track10–FB–113017

Samples of air or solid material (e.g. dust) collected from non-track areas will be identified/numbered according to the following convention:

- Location – Area#–Sample #
    - Location – Upper or Lower Concourse, or reference closest Track or Platform (include Railroad 0)
    - Area# – Specific area where sample was collected (e.g. Break Room)
    - Sample# – Numeric number

As an example, a sample collected from the Lower Concourse in the Amtrak Break Room would be identified according to the following convention:

- Lower Concourse-Amtrak Break Room–1

Field duplicate samples for air collected in the track areas will be identified by adding a D after the numeric number.

6.5.2 Sample Labeling

All samples of air, ballast fines, ballast stone, concrete, dust, soil, and sediment in drains will be labeled using laboratory provided labels following the designated sampling nomenclature described in the preceding section.

Sample locations will be recorded by field personnel relative to fixed points located on the platforms. The fixed reference points will be benchmarks located on the platforms relative to Railroad 0. Sample locations in the Upper and Lower Concourse Levels will be recorded by their locations relative to fixed features.

6.5.3 Sample Custody

Each sample will be identified using a sample label marked on the container in permanent marker containing the following information:

- Project name and number;
- Sample naming conventions are described in Section 6.5.1;
- Analysis;
- Preservative;
- Date;
- Time; and,
- Sampler's name / initials.

The objective of the chain of custody (COC) procedure is to document the history of each sample and its handling. Custody records trace a sample from its collection through all transfers of custody until it is transferred to the laboratory. Samples are considered to be in custody if they are within sight of the individual responsible for their security or locked in a secure location. Each person who takes possession of the samples is responsible for sample integrity and safekeeping. COC procedures are provided below:

- The COC form is completed at the time of sample collection. The sample identification number, sampling location, depth, date, time, and analysis requested are recorded on the form;
- The sampling team will check the sample numbers on the individual jars against the COC form; and,
- Field samplers are responsible for the care and custody of the samples collected until the samples are transferred to another party or locked in a secure location.

All samples will be accompanied by a completed COC form. An example of COC forms are included in the QAPP provided as Appendix E. The sample identification numbers, date collected, matrix, and requested analyses will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the form. This record documents transfer of custody of samples from the sampler to another person, to an off-site laboratory, or to/from a secure storage area. The original COC form will accompany the shipment, and remaining copies will be retained by the sampler and returned to the Project Manager or project file.

6.5.4 Sample Handling

Most of the sampling occurs during the overnight hours of 9pm to 5am. Sample handling is critical as the samples cannot be provided directly to the laboratory. The following describes the procedures for sample handling until the samples are in the custody of the analytical laboratory.

- The field sampler will transfer the samples directly into laboratory-provided containers, label each sample, and immediately place the samples in a cooler with ice. The sample time, location, and identification will be recorded in sampler's field notes and COC form.
- The samples, will then be transported in the cooler with ice by the field sampler. The sample(s) will then either remain in the cooler on ice or placed in a refrigerator in a locked and secured room. The samples will be kept at an appropriate temperature of 4 ± 2ºC. The completed COC will remain with the samples at all times.
- When the samples are ready to be relinquished to the analytical laboratory courier, the samples will be removed from the refrigerator and placed on ice in a cooler or if the samples are already in

the cooler the samples will be given to the analytical laboratory courier. The analytical laboratory will assume custody of the samples when given to the courier. It should be noted that the cooler will have a custody seal on it before it is relinquished to the laboratory. Air samples collected by TRC will be placed in a cooler with ice packs and shipped via overnight courier to Con-Test.

6.5.5 Sample Analyses and Methods, Laboratories, and Quality Assurance / Quality Control

All samples of ballast fines, ballast stone, concrete, dust, soil, and sediment from drains will be analyzed by Alpha Analytical Inc. (Alpha), a New York state certified laboratory (License #11627) located in Westborough, Massachusetts. Samples will be analyzed as listed on Table 3. All air samples will be analyzed by Con-Test Analytical Laboratories, located in East Longmeadow, Massachusetts, an independent third-party industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033) for PCBs. Analytical and extraction methods, sample container volumes, sample preservations and holding times are summarized in the QAPP included as Appendix E. Analyses to be performed, frequency and minimum quantities for each media are described in the attached Table 3 *Sample Analytical Methods and Frequency*.

6.5.6 Field Duplicate and Field Blank Sample Collection

Field Duplicate and Field Blank (equipment rinsate) samples will be collected during the field investigation at the following frequency. It is anticipated that each sampling event will take place over more than one sequential days.

- Field Blank and Field Duplicate samples will be collected at a rate of one sample per 20 samples collected (ballast fines, ballast stone, concrete, soil, sediment, and bulk dust samples).
- One Field Duplicate sample (air) will be collected during each air sampling event.

Quality assurance/Quality Control measures including internal project assessment and oversight will be implemented as described in the QAPP. Each sampling period/event will last one or more sequential days.

6.5.7 Data Validation

All laboratory analytical reports will be provided in New York Category B deliverable format as described in the QAPP. The Data Validation process flow chart and the Data Validation Plan is discussed further and provided in the QAPP (Appendix E).

6.6 Data Management

Deliverable documents generated for the project will be maintained in the Mott MacDonald files and electronic documents will be saved to the Mott MacDonald SharePoint internet website. Pertinent documents generated by the project team including Mott MacDonald, Stantec, TRC, and Clean Harbors will also be saved on the Mott MacDonald SharePoint site. The documentation will be kept on file until the project is completed. At that time Amtrak will take custody of the files.

6.7 Regulatory Interaction and Reporting

Amtrak provides monthly updates to the USEPA (with copies to the NYSDEC) regarding environmental compliance activities completed at PSNY. Monthly summary memos will continue to be prepared throughout the duration of the RI to inform USEPA of the ongoing progress.

6.8 Schedule

PSNY operates 24 hours a day, 365 days a year, and is the busiest passenger transportation facility in North America with each of the station's 21 tracks in use every 2 minutes on weekdays. It serves more than 430,000 passengers and more than 1,300 train movements per weekday and operates 300% over original design capacity. Passengers rely and depend on the infrastructure for daily commuting and to get

them where they need to go. Taking just a single track out of service is disruptive and impacts many stakeholders.

The implementation of the RI will be restricted by Amtrak's ability to coordinate and schedule track outages given the 24-hour operation of the facility. In addition, site constraints including limited work hours, restricted access, and subterranean conditions make implementation of the RI problematic and delay productivity. The magnitude of the RI includes the collection of over 8,000 samples. In consideration of these conditions implementation of the RI will take longer than normal. The estimated RI schedule is provided below. Please note sampling is currently underway and monthly updates are provided to EPA but due to the constraints of working at PSNY the RI will begin when the removal of sediment and "tar-like" material followed by track washing is completed in approximately December 2018. The RI information will be included in an Application for Risk Based Disposal and Approval and Notification and Certification of disposal of PCB remediation waste consistent with 40 CFR 761.61(c).

PSNY RI Schedule

Activity	Start	End
Remedial Investigation	Ongoing as track maintenance, cleaning and access allows.	December 2023
Risk Assessment	Data reviewed periodically.	January 2025
Remedial Investigation Report*	Data reviewed periodically.	July 2025

* The RIR information will be included in an application for Risk Based Disposal and Approval and Notification and Certification of disposal of PCB remediation waste consistent with 40 CFR 761.61(c). The application will be submitted to the USEPA.

References:

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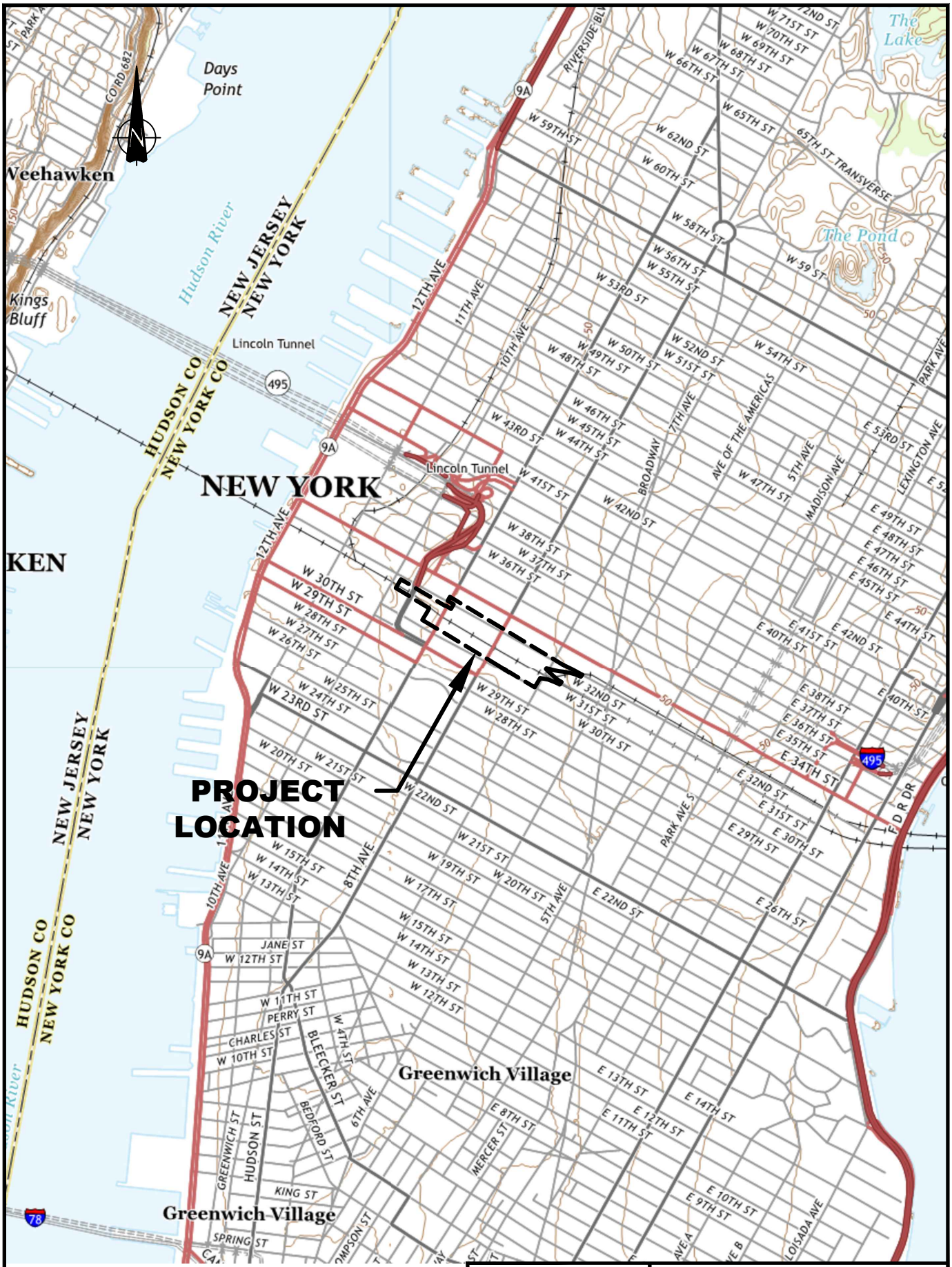
Figures



SOURCE:
Weehawken, NJ-NY; Jersey City, NJ-NY; Brooklyn, NY; and Central Park, NY-NJ 2016 Quadrangles
USGS Topographic Quadrangles 7.5 Minute Series

2000 0 1000 2000 4000

MOTT MACDONALD

111 Wood Avenue South
Iselin, New Jersey 08830

AMTRAK
PENNSYLVANIA STATION, NEW YORK

SITE LOCATION MAP
FIGURE 1

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

PLOT DATE: 01/19/2018 02:44 PM
SAVE DATE: 01/19/2018 02:43 PM

34th Street

To Port Authority Bus Terminal, 8th Ave & 41st St (6 blocks)

Down to Subway

ONE PENN PLAZA ENTRANCE

Down to Lower Level

One Penn Plaza

Down to LIRR

EXIT

M7 M20

33rd Street (one way)

Madison Square Garden

Taxis

Hudson News

Auntie Anne's

Down to LIRR

Up to Street

T.G.I. Friday's

Häagen Dazs Nathan's Pizza Hut KFC Taco Bell Tim Horton

Hudson Book Sellers

Zaro's Bread Basket

Moe's Southwest Grill

Planet Smoothie

Soup Stop

Hudson News

Dunkin Donuts

Kabooz's

Don Pepi Pizza

Krispy Kreme Donuts

PNC ATM

Tiecoon

Wireless To Go

Drago Shoe Repair

Primo Cappuccino

Hudson News Dunkin Donuts

Duane Reade Drugs

Down to LIRR & Subway

Up to Street & Madison Square Garden

Up to Street

Two Penn Plaza

8th Avenue (one way)

7th Avenue (one way)

To PATH 6th Ave & 32nd St (one block)

AMTRAK CONCOURSE

AMTRAK WAITING ROOM

Amtrak Customer Service

Amtrak Ticketing

United Airlines Ticketing

Club Acela

T15/16

T13/14

T11/12

T9/10

T7/8

T5/6

T3/4

T1/2

Down to Lower Level

Down to Lower Level & Tracks 13/14

Up to Street & Down to Lower Level

T11/12

Up to Street & Madison Square Garden

Wells Fargo ATM

Auntie Anne's

Elegance

New York New York

GNC

Hudson News Dunkin Donuts

Chick Pea

Primo Cappuccino

Don Pepi Deli

Penn Sushi

Baggage Check

Up from Tracks 1, 2, 3, 4

Pennsylvania Plaza

The Petal Pusher

Perfumania

Coco Moko Cafe

Down to Mezzanine

Auntie Anne's

Zaro's Bread Basket

Chop' N Toss

NJ TRANSIT Ticketing

NJ TRANSIT Customer Service

NJ TRANSIT MEZZANINE

NJ TRANSIT 7TH AVE CONCOURSE

31st Street (one way)

N E S W

M20

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RIWP\DWG\PENN STATION SITE DESCRIPTION.DWG

		Revision
		Date

Professional Engineer - N.J. Lic. No.

Date

Designed	
Drawn	JLM
Checked	
Approved	
Date	1/22/2018

MM MOTT MACDONALD

Certificate No. 24GA28016600

111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK

PENNSYLVANIA STATION, NEW YORK

SITE DESCRIPTION

UPPER CONCOURSE MAP

FIGURE 2a

Job	No.	
372088	B/O	Total

PLOT DATE: 01/19/2018 02:44 PM
SAVE DATE: 01/19/2018 02:44 PM

34ST STREET (ABOVE)

31ST STREET (ABOVE)

8TH AVENUE (ABOVE)

7TH AVENUE (ABOVE)

LONG ISLAND RAILROAD

HILTON CORRIDOR

WEST END CONCOURSE

EXIT CONCOURSE

CENTRAL CONCOURSE

MEZZANINE

MTA Police

Long Island Rail Road Station Management

To One Penn Plaza & Street

K-Mart

Up To One Penn Plaza & Street

Duane Reade Drugs

Hudson News

Subway Entrance A C E

Subway Entrance 1 2 3

Subway Entrance C E

HSBC ATM

Subway

tarti D-lite

Planet Smoothie Rosa's Pizza

Starbucks

GNC

Carvel Cinnabon

Moe's Southwest Grill

Rose Pasta & Pizza

Le Bon Cafe

Soleman Shoe Repair

Penn Books

Central Market

Starbucks

Planet Smoothie tasti D-lite

Nathan's Pizza Hut Colombo Tim Horton

TGIF

Caruso Pizza Häagen Dazs

Penn Station Bookstore

Knot Just Pretzels KFC Riese Restaurant

Up to Street

Chickpea Red Mango

Fresh Flowers

Bank of America ATM

LIRR Customer Service

Jamba Juice

Carlton Cards

Auntie Anne's

au bon pain

Hot & Crusty

The Petal Pusher

McDonalds

LIRR Ticketing

Tracks Raw Bar & Grill

Penn Wine & Spirits

Books

Cookie Cafe

Don Pepi Express

Up to Upper Level

Up to Upper Level/Street

Platform to Track 17

Platform to Tracks 15 & 16

Platform to Tracks 13 & 14

Platform to Tracks 11 & 12

Platform to Tracks 9 & 10

Platform to Tracks 7 & 8

Platform to Tracks 5 & 6

Platform to Tracks 3 & 4

Platform to Tracks 1 & 2

Platform to Tracks 18 & 19

TRACK 1 … TRACK 21

To New Jersey

To Long Island

To Boston

To Queens (A C)

To Brooklyn (A C)

To World Trade Center (E)

To South Ferry (1)

To Brooklyn (2 3)

To Broadw (A C)

To Chec (E)

To The Bro (1 2)

To Harle (3)

N

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RWP\DWG\PENN STATION SITE DESCRIPTION.DWG

Revision	Date

Professional Engineer - N.J. Lic. No. | Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
SITE DESCRIPTION
LOWER CONCOURSE MAP
FIGURE 2b

Job	No.	
372088	B/O	Total

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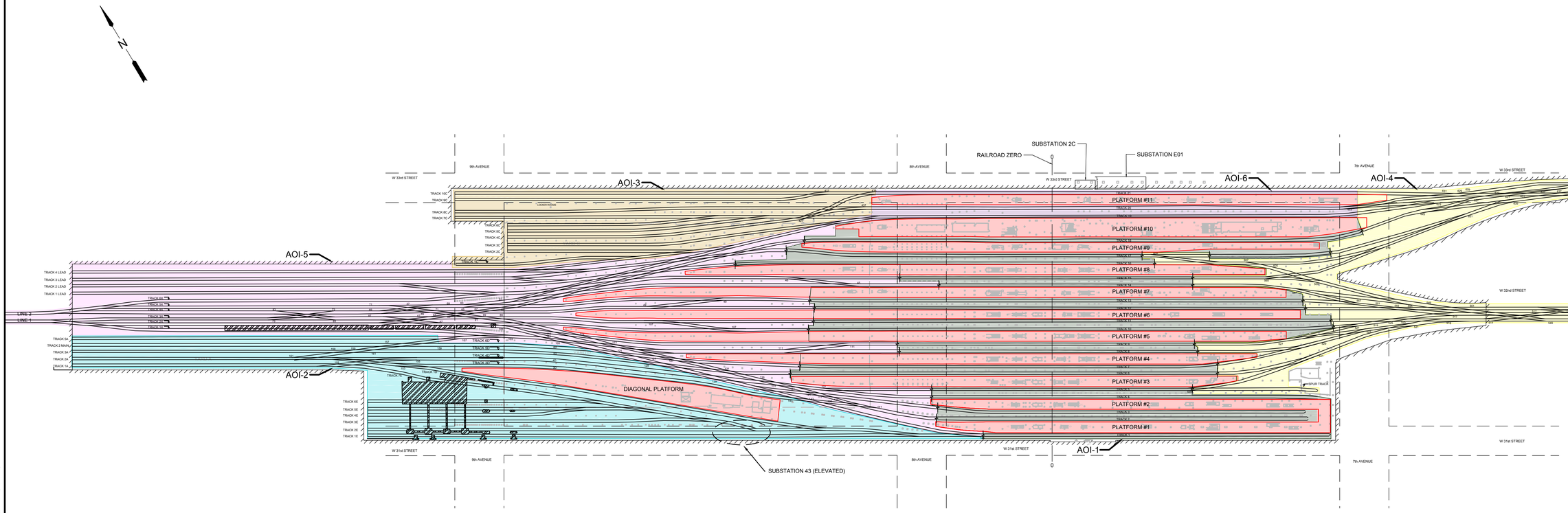

NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. RAILROAD ZERO DENOTES A REFERENCE POINT AT PENNSYLVANIA STATION, NEW YORK. SAMPLE LOCATIONS WILL BE REFERENCED FROM RAILROAD ZERO.

3. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

4. THE PROPOSED EXCAVATION AREA IS EXCLUDED FROM THE PROJECT. THIS AREA WILL BE REMEDIATED BY BOP SE L.L.C. (COMMONLY REFEREED TO AS BROOK FIELD) AS DESCRIBED IN A SELF-IMPLEMENTING CLEANUP PLAN PROPOSED BY ARKF DATED 11/6/17.

LEGEND:

- 89 — SWITCH NUMBER
- BALLAST/CONCRETE TRANSITION
- WALLS
- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM
- PROPOSED EXCAVATION AREA (BOP SE LLC, OTHERWISE KNOWN AS "BROOK FIELD" EXCLUDED FROM RIWP)

0 200 400

Revision	Date

Professional Engineer - N.J. Lic. No. _____ Date

Designed	
Drawn	JLM
Checked	
Approved	
Date	1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
AREAS OF INTEREST MAP
AIR MONITORING PLAN
FIGURE 2c

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\MULTIPLE_AOIS\FULL_STATION\DWG\AOI-1-2-3-4-5-6_AREAS-OF-INTEREST-MAP-EXCAVATION.DWG

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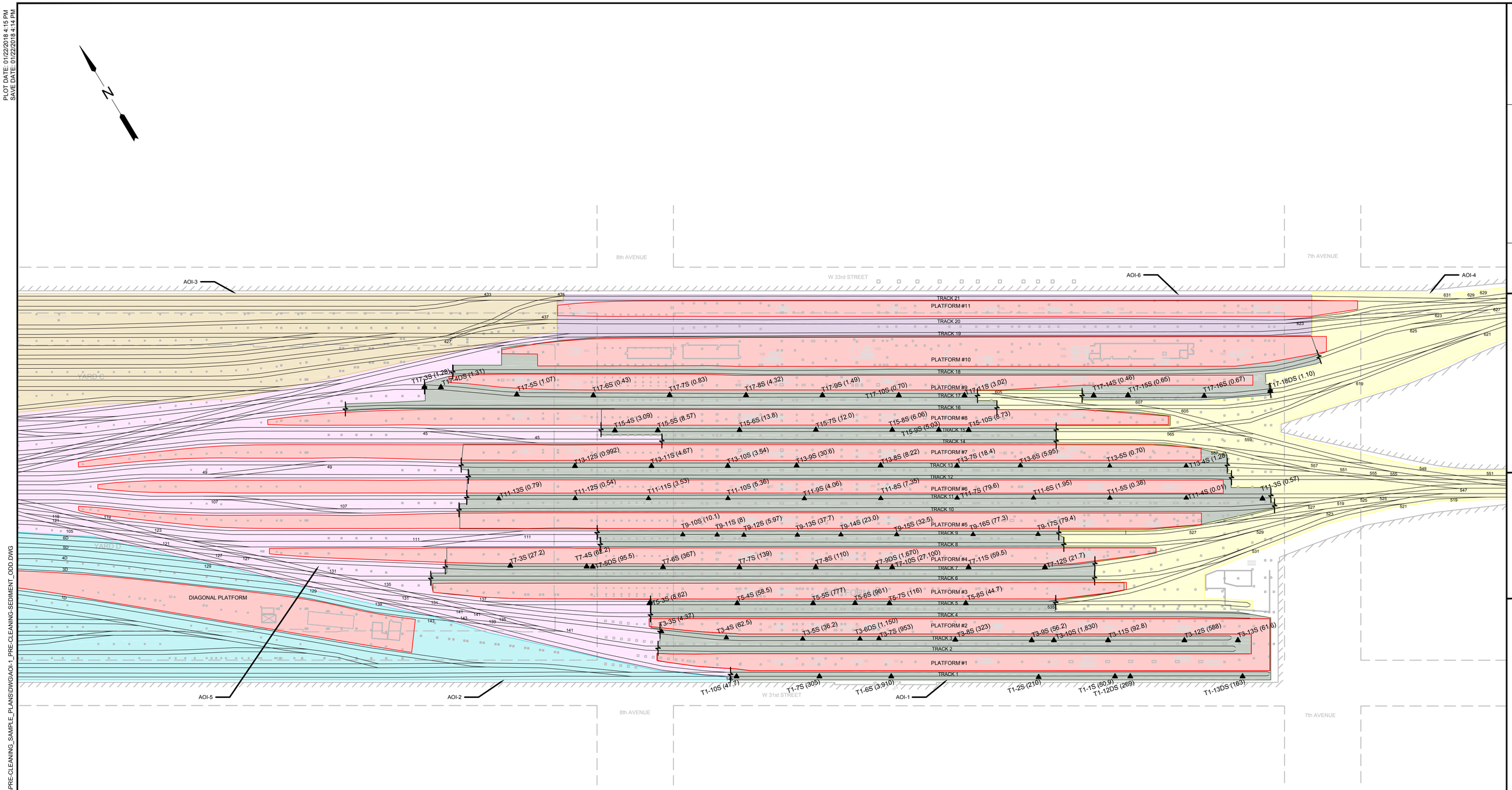
SAVE DATE: 01/22/2018 4:14 PM



NOTES:
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2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

3. THE ANALYTICAL DATA HAS NOT UNDERGONE VALIDATION.

4. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM
- * SAMPLES WERE REANALYZED
- ▲ T1-1S: PRE CLEANING SEDIMENT SAMPLE LOCATION AT AOI-1; T1-12DS: PRE CLEANING SEDIMENT SAMPLE LOCATED ADJACENT TO DRAIN AT AOI-1
- (2.73) DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)
- (10.6/12) INDICATES DUPLICATE SAMPLE OBTAINED
- ND NOT DETECTED ABOVE THE LABORATORY'S REPORTING LIMIT

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
SEDIMENT ANALYTICAL RESULTS
ODD TRACKS
FIGURE 3a

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_1_TRACKS-1-19\PRE-CLEANING_SAMPLE_PLANS\DWG\AOI-1_PRE-CLEANING-SEDIMENT_ODD.DWG

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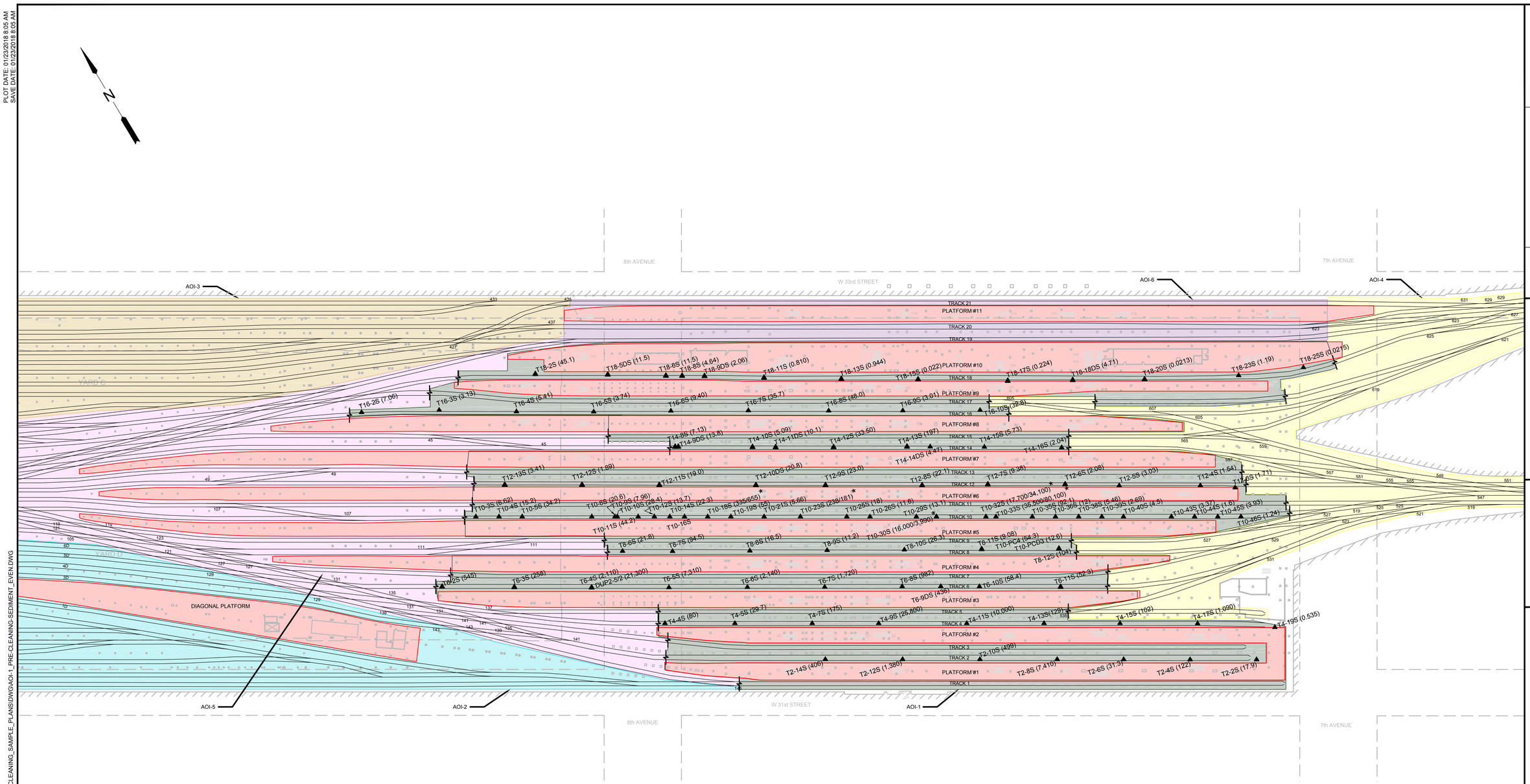

NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

3. THE ANALYTICAL DATA HAS NOT UNDERGONE VALIDATION.

4. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
	LIMITS OF PLATFORM
*	SAMPLES WERE REANALYZED
▲	T2-2S: PRE CLEANING SEDIMENT SAMPLE LOCATION AT AOI-1 T12-10DS: PRE CLEANING SEDIMENT SAMPLE LOCATED ADJACENT TO DRAIN AT AOI-1
(1.06)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)
(10.6/12)	INDICATES DUPLICATE SAMPLE OBTAINED
ND	NOT DETECTED ABOVE THE LABORATORY'S REPORTING LIMIT

0 120 240

Revision	Date

Professional Engineer - N.J. Lic. No.

Date 1/22/2018
Approved
Checked
Drawn JLM
Designed

MOTT MACDONALD
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
SEDIMENT ANALYTICAL RESULTS
EVEN TRACKS
FIGURE 3b

Job 372088
No.
B/O
Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_1_TRACKS-1-19\PRE-CLEANING_SAMPLE_PLANS\DWG\AOI-1_PRE-CLEANING-SEDIMENT_EVEN.DWG

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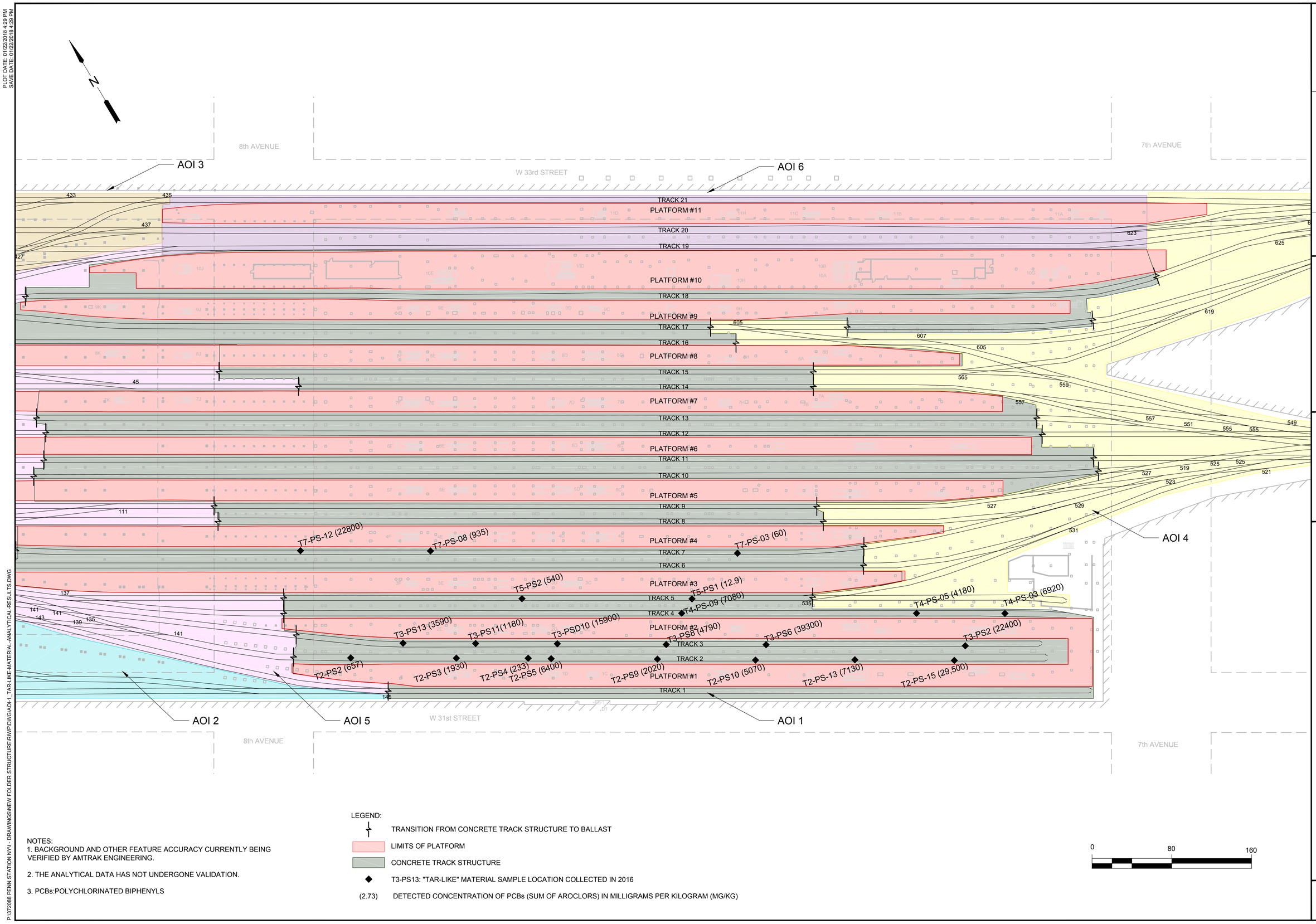

NOTES:
1. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

2. THE ANALYTICAL DATA HAS NOT UNDERGONE VALIDATION.

3. PCBs:POLYCHLORINATED BIPHENYLS

LEGEND:

- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM
- CONCRETE TRACK STRUCTURE
- T3-PS13: "TAR-LIKE" MATERIAL SAMPLE LOCATION COLLECTED IN 2016
- (2.73) DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)

Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Designed | Drawn: JLM | Checked | Approved | Date: 1/22/2018

MOTT MACDONALD
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Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
"TAR-LIKE" MATERIAL ANALYTICAL RESULTS
FIGURE 4

Job: 372088 | No. | B/O | Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RIWP\DWG\AOI-I_TAR-LIKE-MATERIAL-ANALYTICAL-RESULTS.DWG

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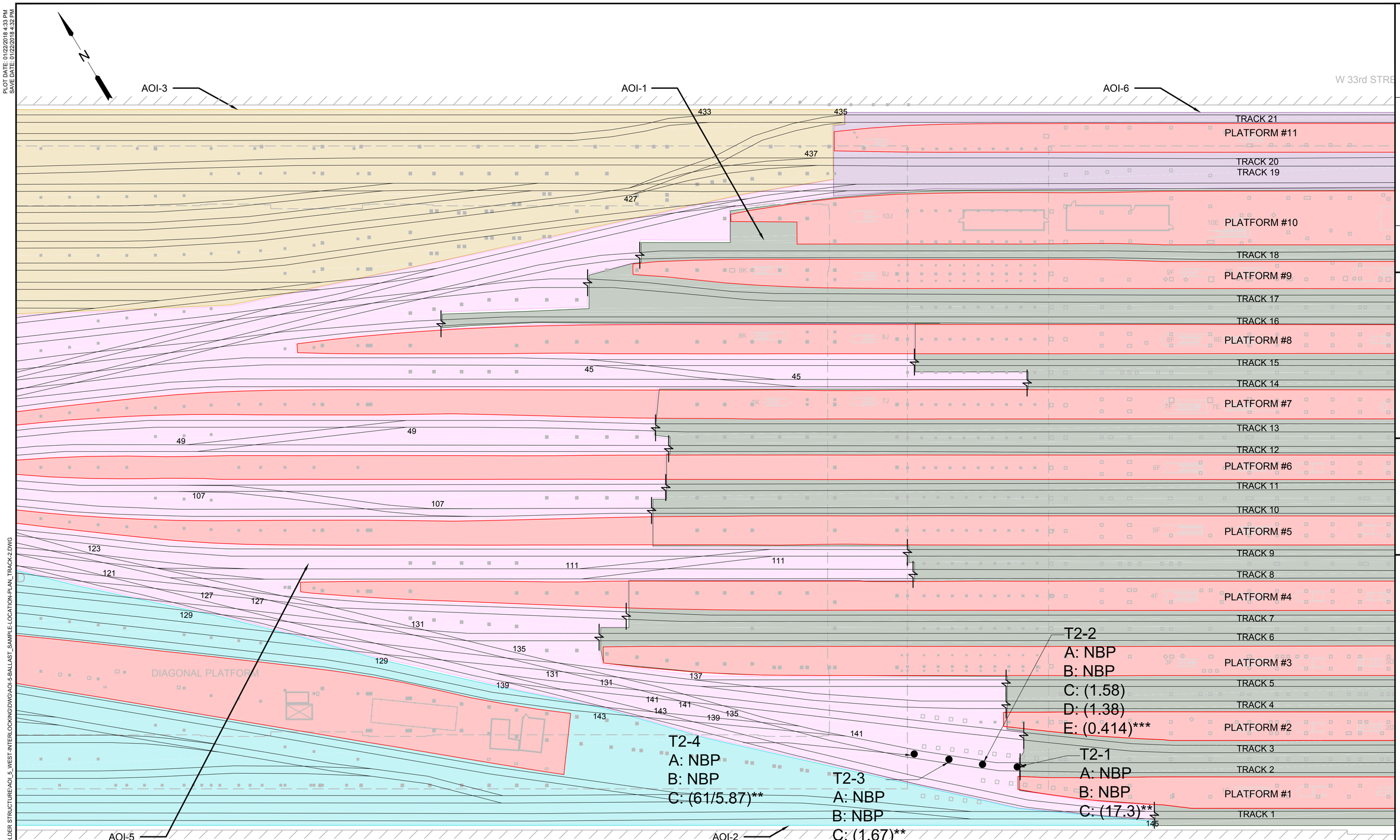

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2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

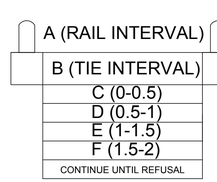
5. PCBs: POLYCHLORINATED BIPHENYLS

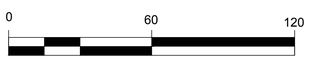
LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM





Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 5
FIGURE 5a - TRACK 2

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_5_WEST-INTERLOCKING\DWG\AOI-5-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-2.DWG

PLOT DATE: 01/22/2018 4:34 PM
SAVE DATE: 01/19/2018 3:07 PM

N

AOI-3

AOI-1

AOI-6

W 33rd STRE

TRACK 21
PLATFORM #11
TRACK 20
TRACK 19
PLATFORM #10
TRACK 18
PLATFORM #9
TRACK 17
TRACK 16
PLATFORM #8
TRACK 15
TRACK 14
PLATFORM #7
TRACK 13
TRACK 12
PLATFORM #6
TRACK 11
TRACK 10
PLATFORM #5
TRACK 9
TRACK 8
PLATFORM #4
TRACK 7
TRACK 6
PLATFORM #3
TRACK 5
TRACK 4
PLATFORM #2
TRACK 3
TRACK 2
PLATFORM #1
TRACK 1

433 435 437 427 45 45 49 49 107 107 123 121 127 127 129 131 135 129 139 131 131 137 141 141 143 143 139 135 141 111 111 145

DIAGONAL PLATFORM

T3-3
A: NBP
B: BO
C: (1.37)**

T3-2
A: NBP
B: BO
C: (10.7)**

T3-4
A: NBP
B: BO
C: (1.64)**

T3-1
A: NBP
B: (2.29/2.4)
C: (7.98)
D: (11.8)***

AOI-5

AOI-2

NOTES:
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3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM

A (RAIL INTERVAL)
B (TIE INTERVAL)
C (0-0.5)
D (0.5-1)
E (1-1.5)
F (1.5-2)
CONTINUE UNTIL REFUSAL

RAIL

RAILROAD TIE

0 60 120

Revision | Date

Professional Engineer - N.J. Lic. No.

Date

Designed | Drawn JLM | Checked | Approved | Date 1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 5
FIGURE 5b - TRACK 3

Job 372088 | No. | B/O | Total

P:\372088 PENN STATION NY\1 - DRAWINGS\NEW FOLDER STRUCTURE\AOI_5_WEST-INTERLOCKING\DWG\AOI-5-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-3.DWG

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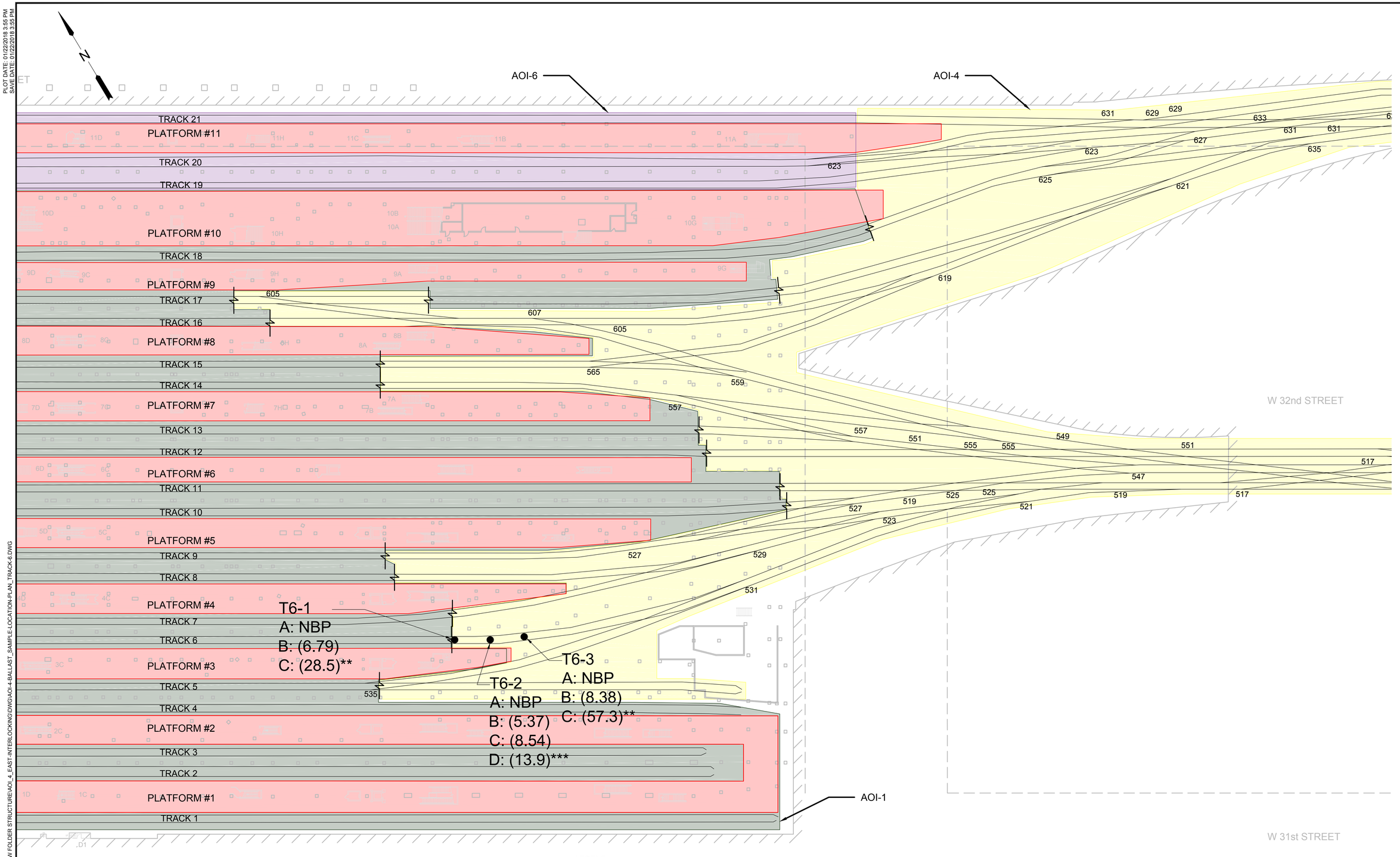

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4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

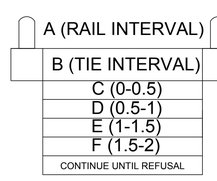
5. PCBs: POLYCHLORINATED BIPHENYLS

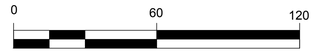
LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM





Revision		
Date		
Professional Engineer - N.J. Lic. No.	Date	
Designed		
Drawn	JLM	
Checked		
Approved		
Date	1/22/2018	

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Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 4
FIGURE 5c - TRACK 6

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_4_EAST-INTERLOCKING\DGW\GAOI-4-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-6.DWG

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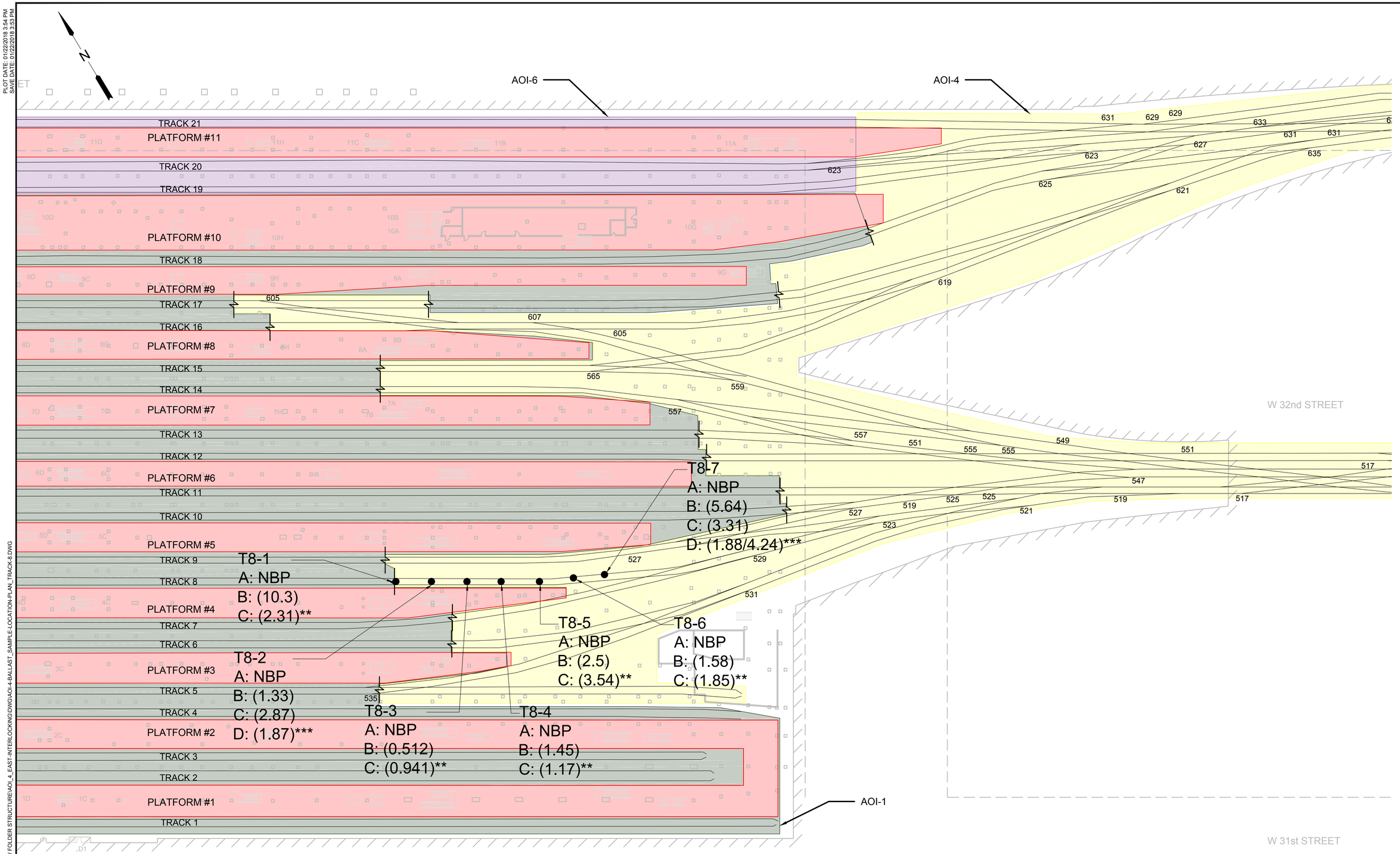

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5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

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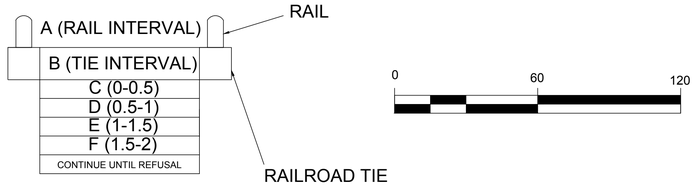
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM



Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

Revision

Date

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111 Wood Avenue South
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AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 4
FIGURE 5d - TRACK 8

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NYI - DRAWINGS\NEW FOLDER STRUCTURE\AOI_4_EAST - INTERLOCKING\DWG\AOI-4-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-8.DWG

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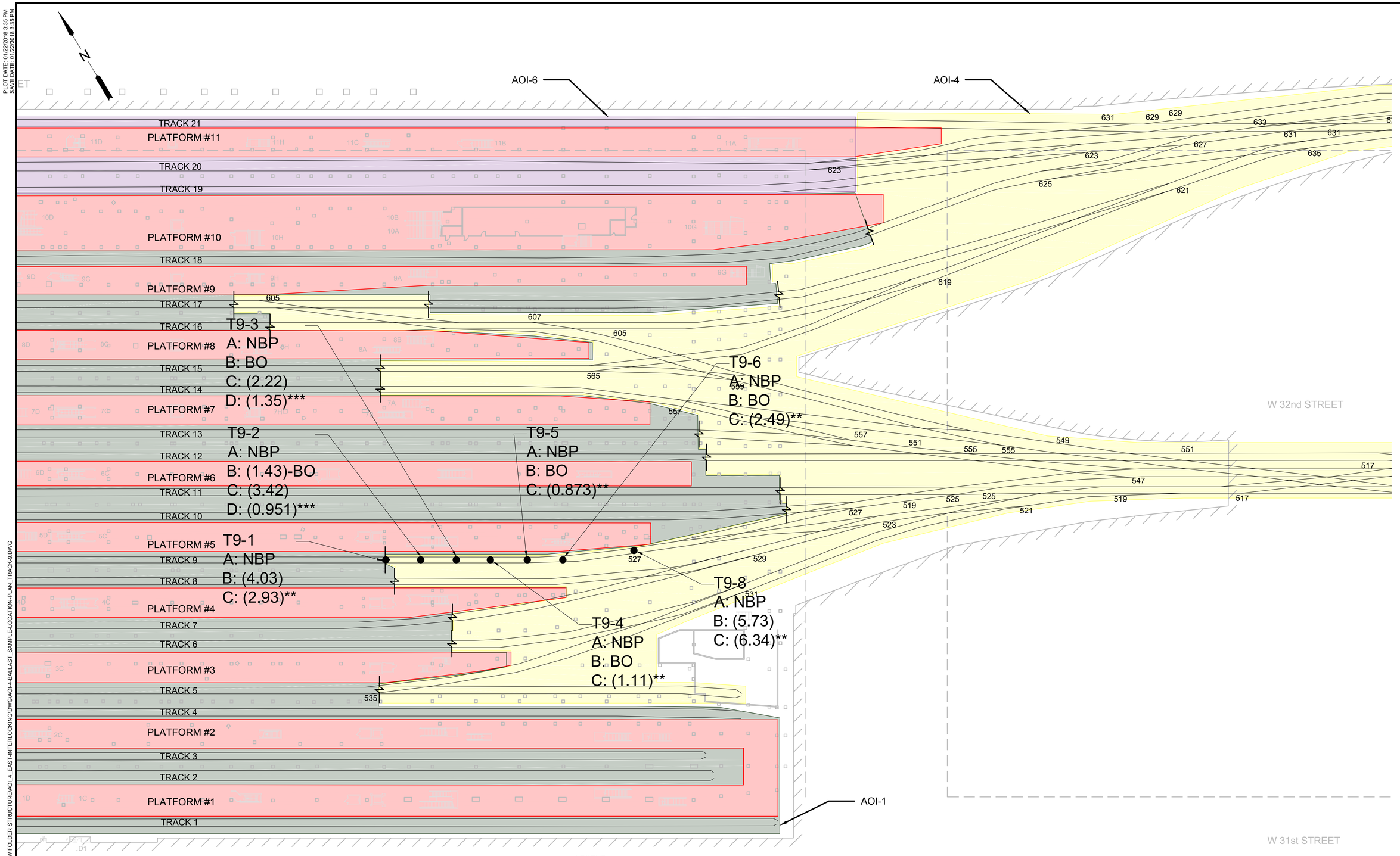

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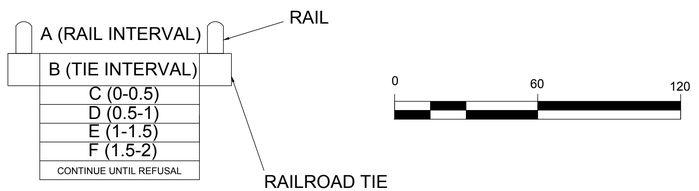
5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT
	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
	LIMITS OF PLATFORM



Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

Revision / Date

M MOTT MACDONALD
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 4
FIGURE 5e - TRACK 9

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NYI\ - DRAWINGS\NEW FOLDER STRUCTURE\AOI_4_EAST-INTERLOCKING\DWG\AOI-4-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-9.DWG

PLOT DATE: 01/22/2018 4:35 PM
SAVE DATE: 01/19/2018 3:06 PM

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N

AOI-3

AOI-1

AOI-6

AOI-5

AOI-2

LOCKER ROOMS

YARD C

YARD D

DIAGONAL PLATFORM

TRACK 21
PLATFORM #11
TRACK 20
PLATFORM #10
TRACK 18
PLATFORM #9
TRACK 17
TRACK 16
PLATFORM #8
TRACK 15
TRACK 14
PLATFORM #7
TRACK 13
TRACK 12
PLATFORM #6
TRACK 11
TRACK 10
PLATFORM #5
TRACK 9
TRACK 8
PLATFORM #4
TRACK 7
TRACK 6
PLATFORM #3
TRACK 5
TRACK 4
PLATFORM #2
TRACK 3
TRACK 2
PLATFORM #1
TRACK 1

T13-6
A: NBP
B: BO
C: (1.75)**

T13-4 *

T13-2 *

T13-5 *

T13-1
A: NBP
B: BO
C: (2.68)**

T13-3
A: NBP
B: BO
C: (3.05)
D: (3.85)**

NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM

A (RAIL INTERVAL)
B (TIE INTERVAL)
C (0-0.5)
D (0.5-1)
E (1.5-2)
F (1.5-2)
CONTINUE UNTIL REFUSAL

RAIL

RAILROAD TIE

0 60 120

Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Designed | Drawn JLM | Checked | Approved | Date 1/22/2018

MOTT MACDONALD
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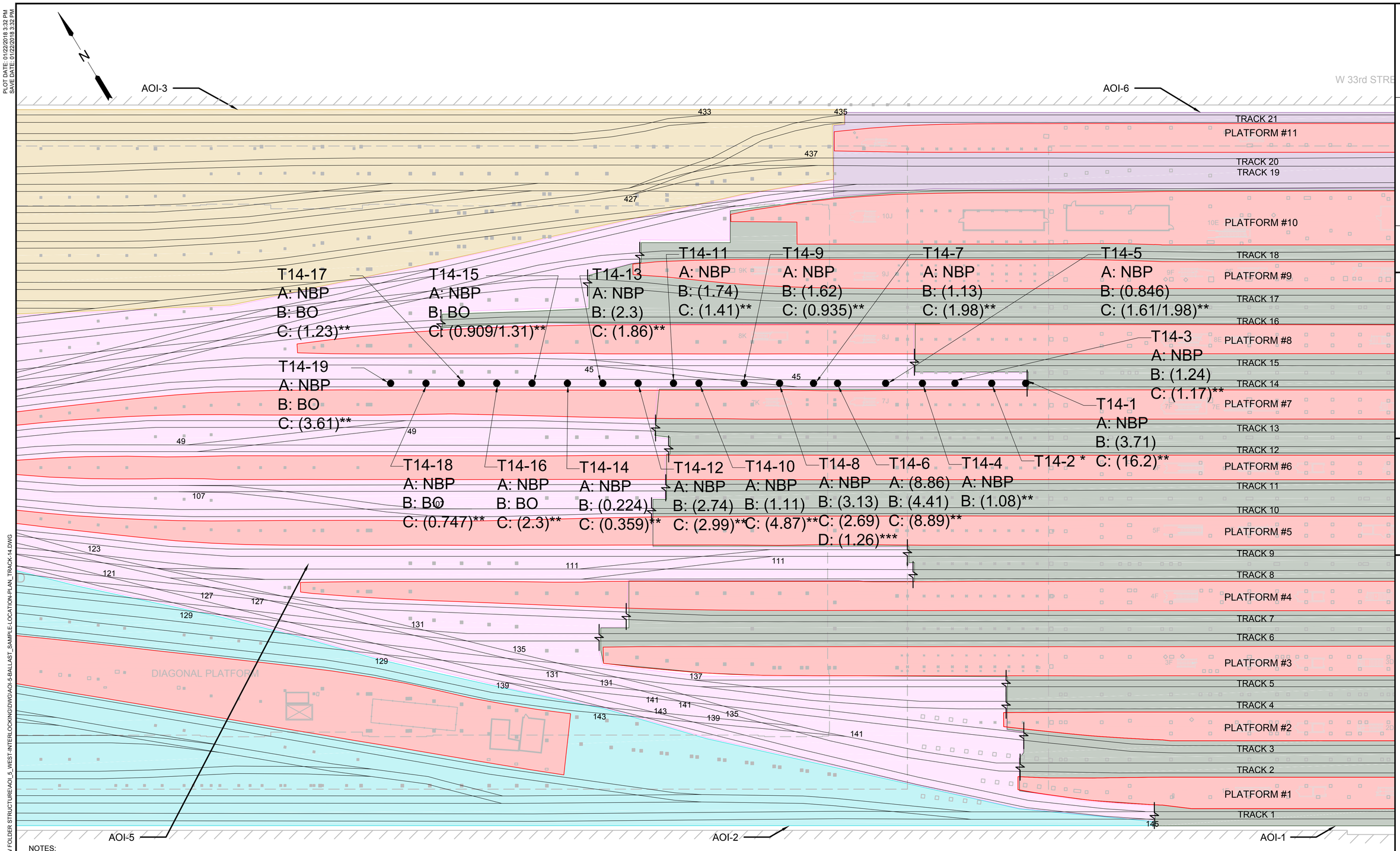
AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 5
FIGURE 5f - TRACK 13

Job 372088 | No. | B/O | Total

PLOT DATE: 01/22/2018 3:32 PM
SAVE DATE: 01/22/2018 3:32 PM



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

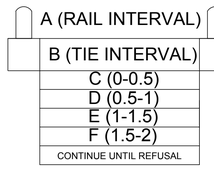
5. PCBs: POLYCHLORINATED BIPHENYLS

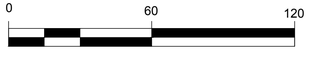
LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES

TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

LIMITS OF PLATFORM





P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_5_WEST-INTERLOCKING\DWG\AOI-5-BALLAST_SAMPLE-LOCATION-PLAN_TRACK-14.DWG

Revision | Date

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Designed | Drawn: JLM | Checked | Approved | Date: 1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 5
FIGURE 5g - TRACK 14

Job: 372088 | No. | B/O | Total

PLOT DATE: 01/22/2018 3:30 PM
SAVE DATE: 01/22/2018 3:30 PM

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N

AOI-6

AOI-4

AOI-1

TRACK 21
PLATFORM #11
TRACK 20
TRACK 19
PLATFORM #10
TRACK 18
PLATFORM #9
TRACK 17
TRACK 16
PLATFORM #8
TRACK 15
TRACK 14
PLATFORM #7
TRACK 13
TRACK 12
PLATFORM #6
TRACK 11
TRACK 10
PLATFORM #5
TRACK 9
TRACK 8
PLATFORM #4
TRACK 7
TRACK 6
PLATFORM #3
TRACK 5
TRACK 4
PLATFORM #2
TRACK 3
TRACK 2
PLATFORM #1
TRACK 1

T18-1
A: NBP
B: (0.597)-BO
C: (0.67)
D: ND
E: (0.148)***

T18-2
A: NBP
B: BO
C: (0.264/0.258)**

T18-3
A: NBP
B: BO
C: (0.619)**

W 32nd STREET

W 31st STREET

NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. SAMPLING INTERVAL IS A THROUGH C EVERY 25 FEET. EVERY 150 FEET SAMPLING INTERVAL IS INTERVAL A THROUGH REFUSAL.

5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

●	T6-1: BALLASTED AREA SAMPLE LOCATION/PCB RESULT IN PARENTHESES
*	NO SAMPLE TAKEN; BALLAST ONLY PRESENT
**	SAMPLE LOCATION TERMINATED
***	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
(10.9)	DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
(1.88/4.24)	DUPLICATE SAMPLE OBTAINED
NBP	NO BALLAST PRESENT
BO	BALLAST ONLY WITH NO FINES
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT
	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
	LIMITS OF PLATFORM

A (RAIL INTERVAL)
B (TIE INTERVAL)
C (0-0.5)
D (0.5-1)
E (1-1.5)
F (1.5-2)
CONTINUE UNTIL REFUSAL

RAIL

RAILROAD TIE

0 60 120

Revision | Date

Professional Engineer - N.J. Lic. No.
FOR REVIEW PURPOSES ONLY
Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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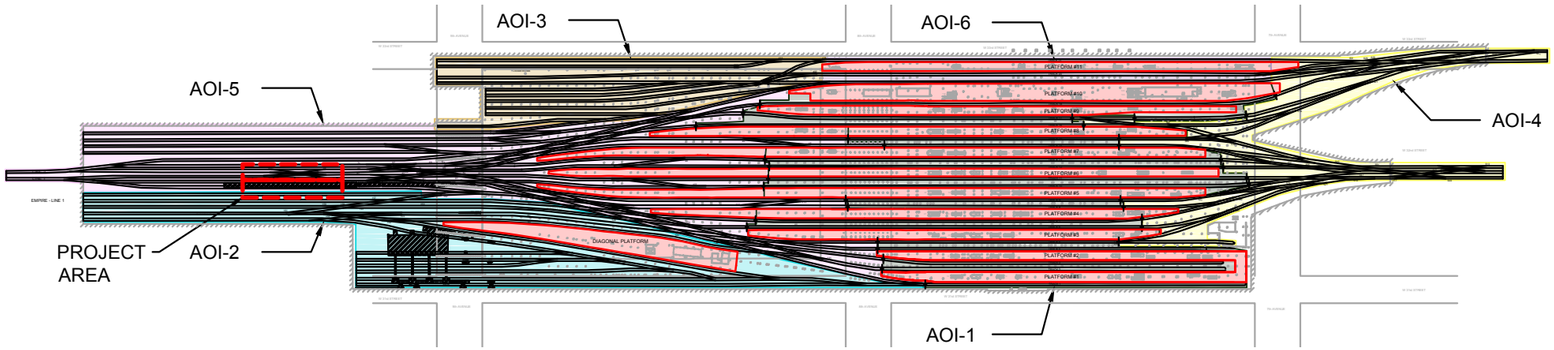
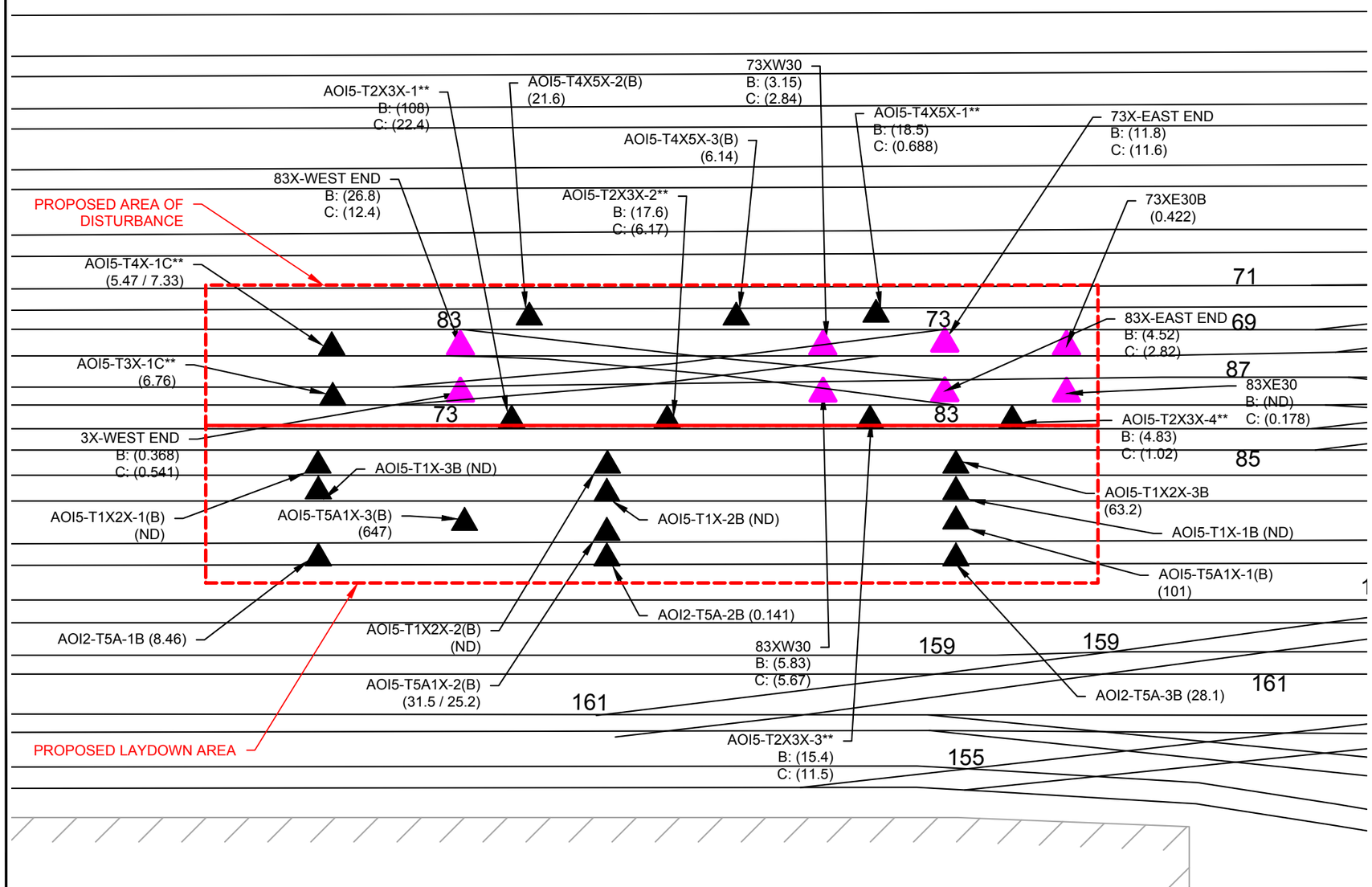
AMTRAK
PENNSYLVANIA STATION, NEW YORK
BALLASTED AREAS ANALYTICAL RESULTS
AREA OF INTEREST 4
FIGURE 5h - TRACK 18

Job	No.	
372088	B/O	Total



AREA OF INTEREST LOCATION PLAN



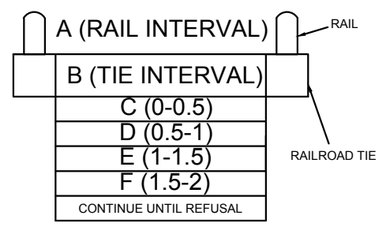
LEGEND:

- SAMPLE LOCATION (COLLECTED MAY 2016)
- SAMPLE LOCATION (COLLECTED AUGUST 2016)
- (2.08) DETECTED CONCENTRATION OF TOTAL POLYCHLORINATED BIPHENYLS (PCBs) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
- (1.32/1.55) DUPLICATE SAMPLE COLLECTED
- 73 SWITCH NUMBER
- * SAMPLE LOCATION TERMINATED
- ** REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NJ PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. SAMPLES OUTSIDE THE GAUGE WERE COLLECTED AT 6 INCH INTERVALS FROM THE GROUND SURFACE.
4. SAMPLES WITHIN THE GAUGE WERE COLLECTED FROM THE FIRST DEPTH INTERVAL WHERE BALLAST FINES WERE PRESENT.
5. UNDERGROUND UTILITIES WERE ENCOUNTERED AT AOI5-T4X5X-2(B) AND AOI5-T4X5X-3(B). THEREFORE, NO SAMPLES WERE COLLECTED BELOW THE B INTERVAL.
6. PCBs EQUAL SUM OF AROCLORS

0 30 60



Revision	Date

Professional Engineer - N.J. Lic. No. | Date 1/22/2018

Approved | Checked | Drawn JLM | Designed

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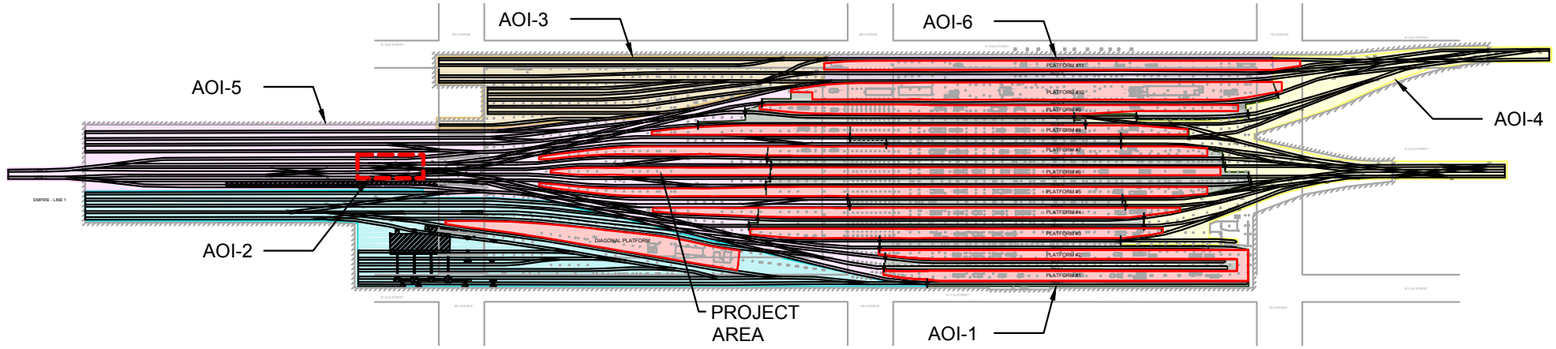
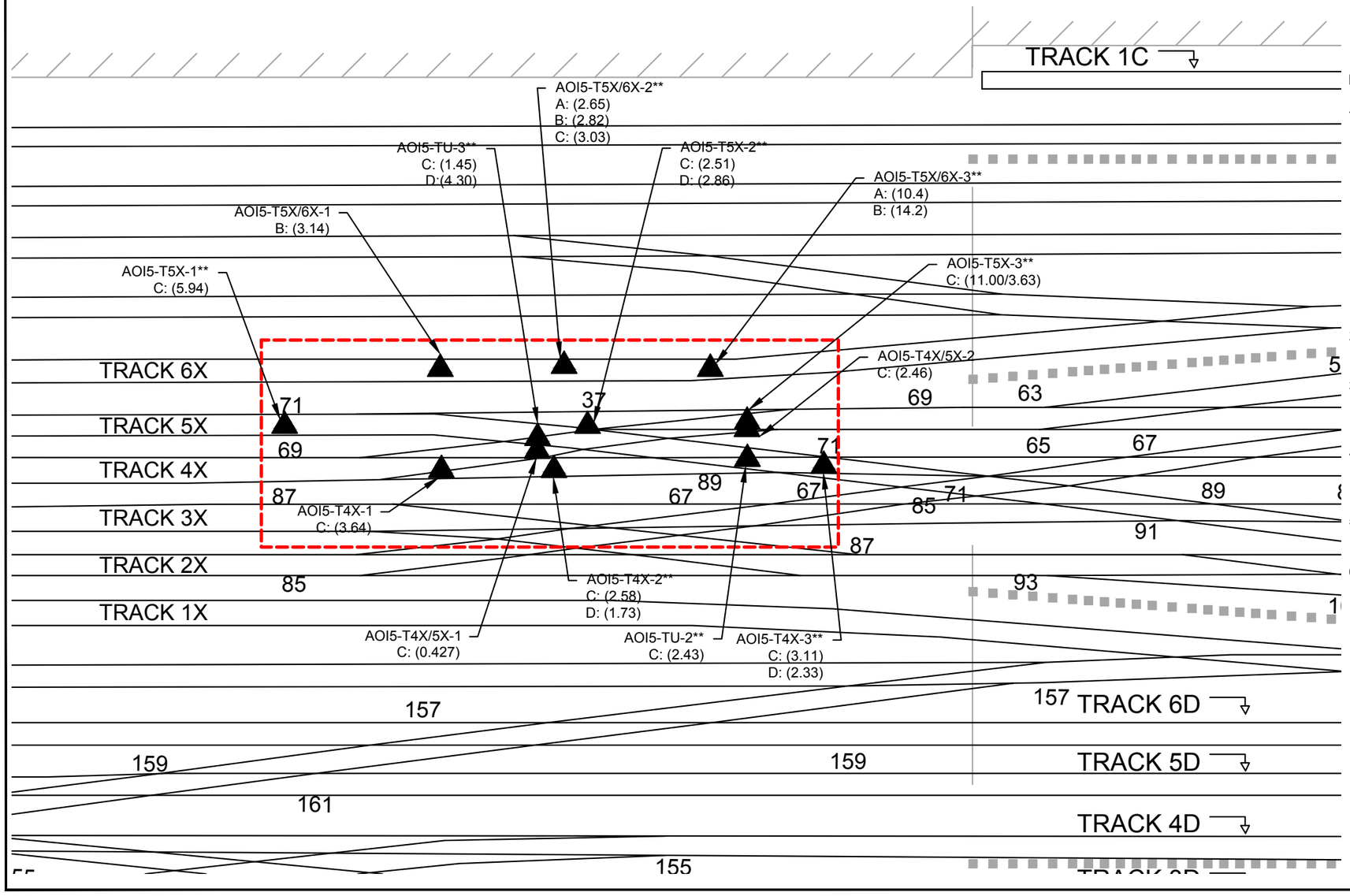
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
SWITCH 73 & 83
FIGURE 6a

Job 372088 | No. | B/O | Total



AREA OF INTEREST LOCATION PLAN



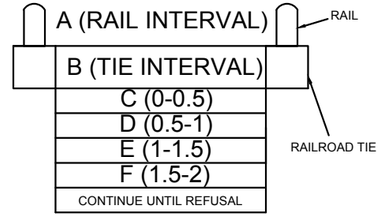
LEGEND:

▲	SAMPLE LOCATION
(2.08)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
(1.32/1.55)	DUPLICATE SAMPLE COLLECTED
69	SWITCH NUMBER
*	SAMPLE LOCATION TERMINATED
**	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
⌇	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NJ PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. SAMPLES WERE COLLECTED INSIDE THE GAUGE FROM THE FIRST INTERVAL WHERE BALLAST FINES WERE PRESENT AT INTERVALS A, B, C. (WHERE POSSIBLE).
4. DEEPER SAMPLES WERE COLLECTED AT THE "D" SAMPLING INTERVAL AT AOI5-TU-3, AOI5-T4X-2, AOI5-T4X-3, AND AOI5-T5X-2.
5. SAMPLES WERE COLLECTED OUTSIDE THE GAUGE AT INTERVALS A, B, AND C (WHERE POSSIBLE).
6. PCBs: POLYCHLORINATED BIPHENYLS

0 30 60



Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Date 1/22/2018 | Approved | Checked | Drawn JLM | Designed

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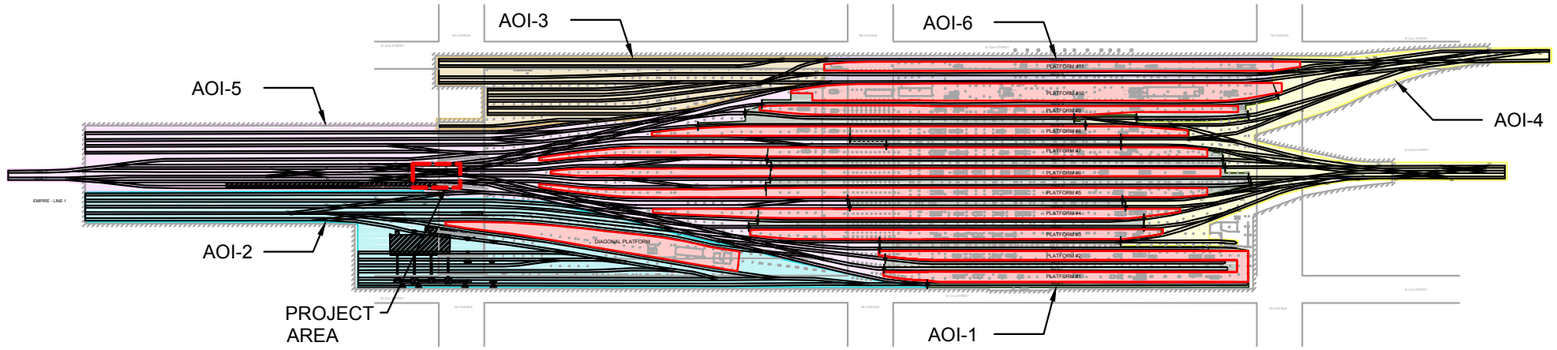
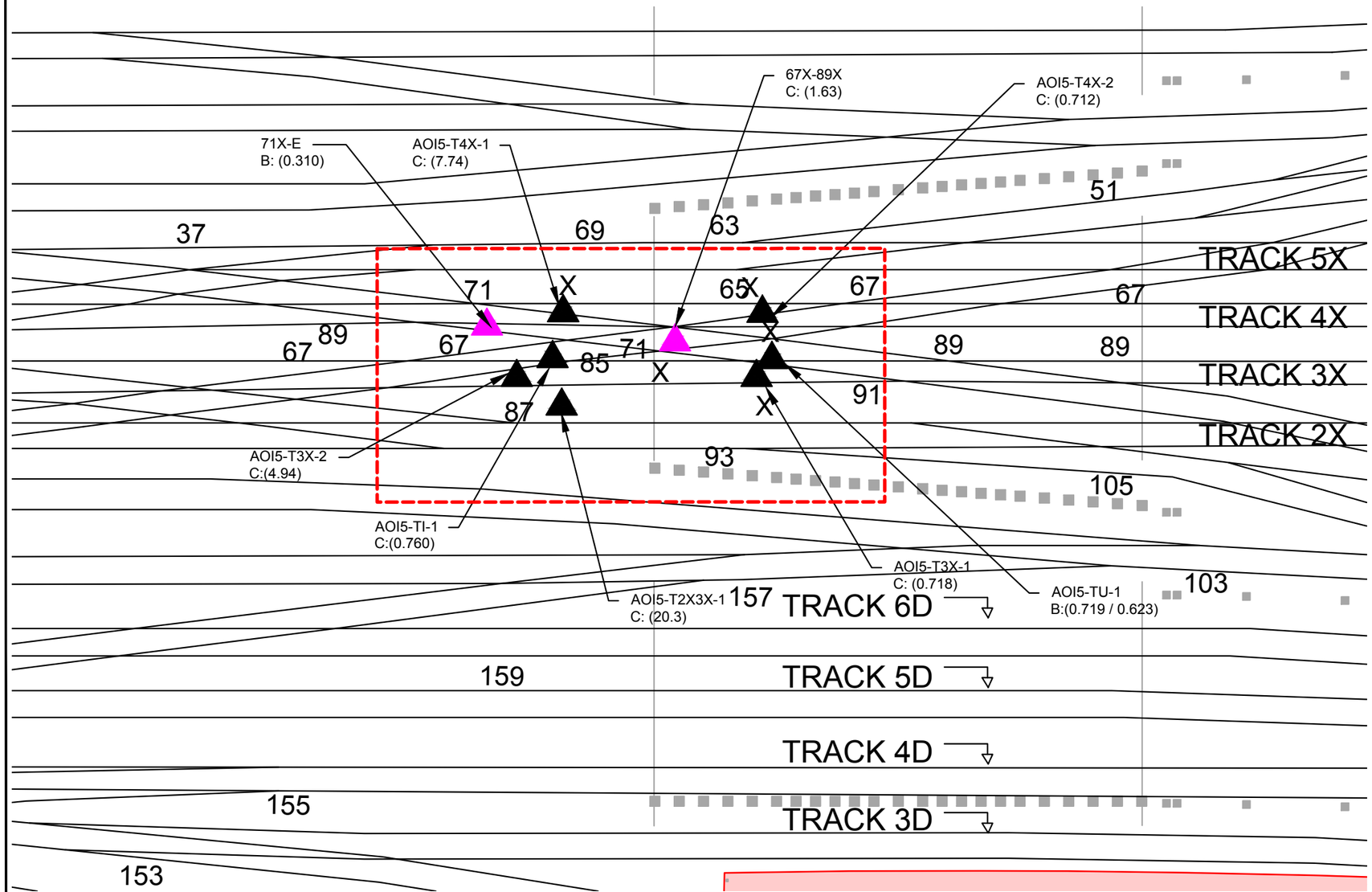
AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
10TH AVE SWITCH
FIGURE 6b

Job 372088 | No. | B/O | Total

PLOT DATE: 01/22/2018 03:28 PM
SAVE DATE: 01/22/2018 03:28 PM



AREA OF INTEREST LOCATION PLAN



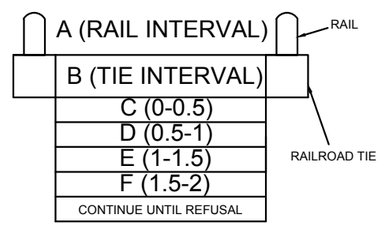
LEGEND:

▲	SAMPLE LOCATION (COLLECTED JUNE 15, 2016)
▲	SAMPLE LOCATION (COLLECTED OCTOBER 20, 2016)
X	ATTEMPTED SAMPLE LOCATION, NO BALLAST FINES PRESENT FOR SAMPLE ANALYSIS
(2.08)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
(1.32/1.55)	DUPLICATE SAMPLE COLLECTED
71	SWITCH NUMBER
*	SAMPLE LOCATION TERMINATED
**	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
⚡	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. SAMPLES WERE COLLECTED FROM THE FIRST INTERVAL WHERE BALLAST FINES WERE PRESENT AND REFUSAL WAS ENCOUNTERED BELOW THE DEEPEST SAMPLING INTERVAL.
4. THE DISTANCE MEASURED BETWEEN THE EAST END OF SWITCH 71 AND THE WEST END OF SWITCH 65 IS APPROXIMATELY 48 FEET.
5. PCBs: POLYCHLORINATED BIPHENYLS

0 25 50



Revision	
Date	
Date	1/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

Professional Engineer - N.J. Lic. No.

Date

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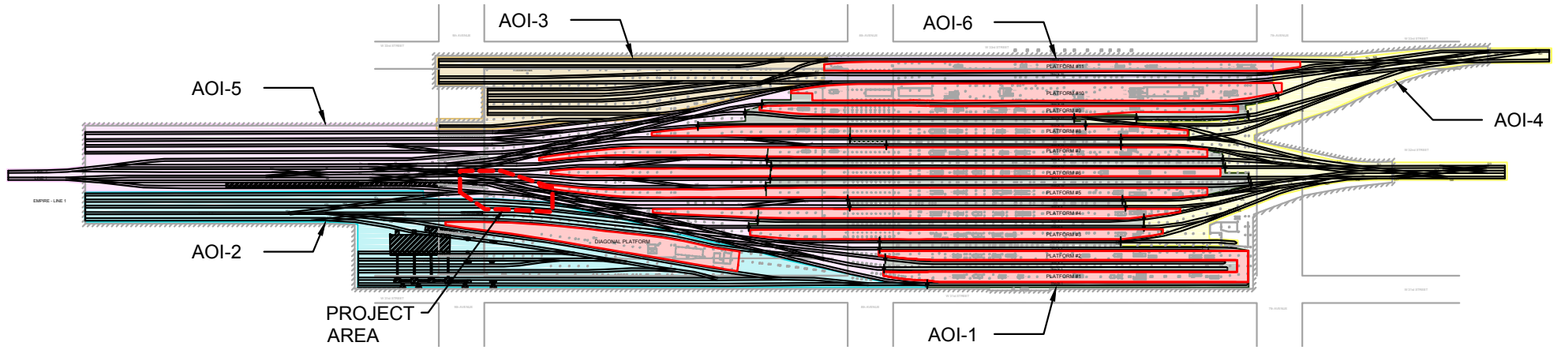
AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
9TH AVE SWITCH
FIGURE 6c

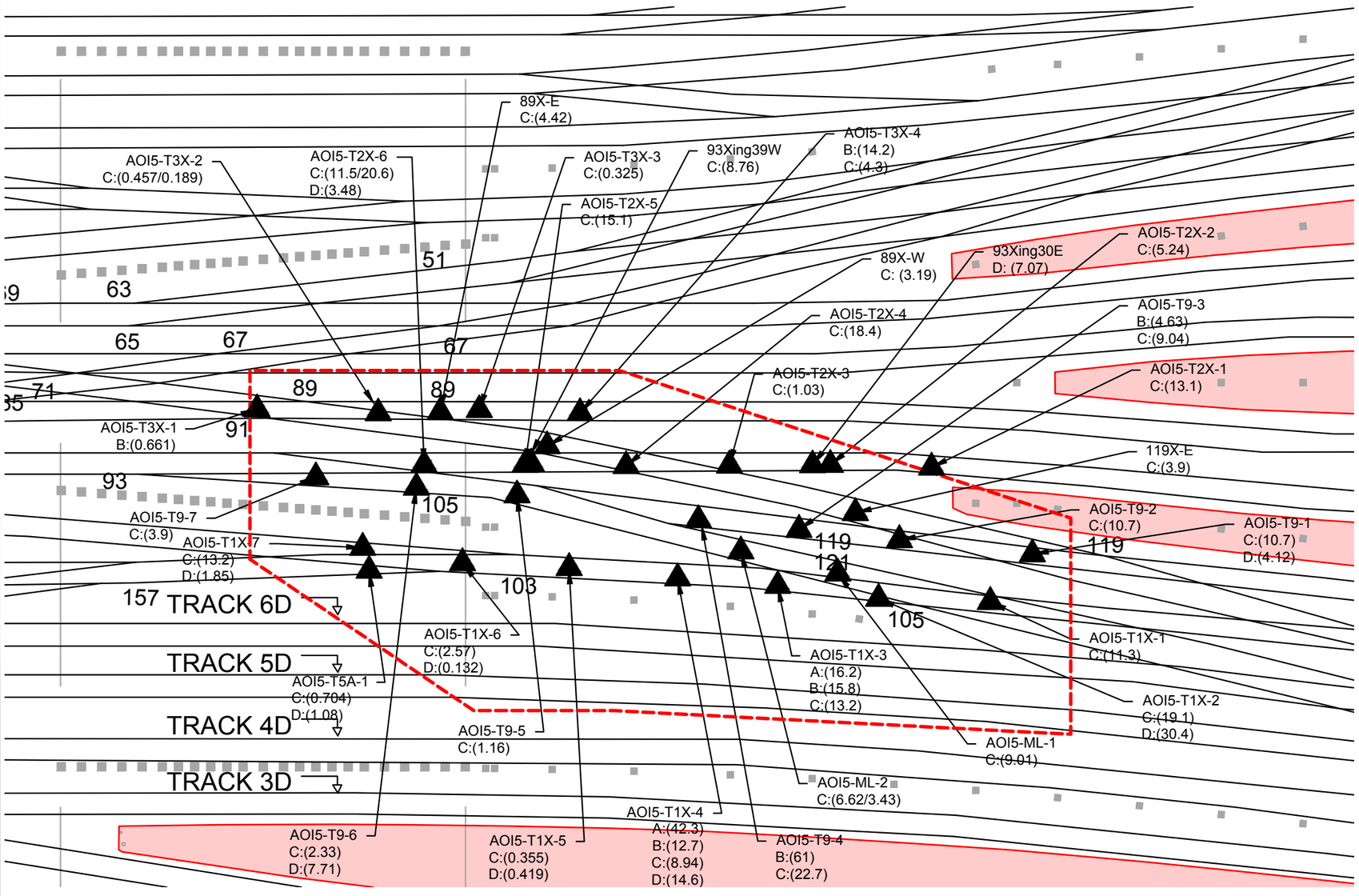
Job	No.
372088	B/O / Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURES\SWITCH REPLACEMENT PROJECTS\PREVIOUS PRIORITY PROJECTS\DWG\AOI-5_9TH-AVE-SWITCH.DWG

PLOT DATE: 01/22/2018 03:22 PM
SAVE DATE: 01/22/2018 03:21 PM



AREA OF INTEREST LOCATION PLAN



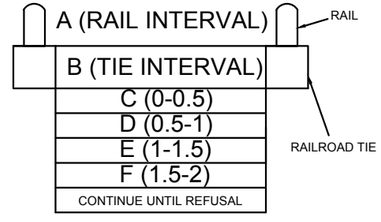
LEGEND:

▲	SAMPLE LOCATION
(2.08)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
(1.32/1.55)	DUPLICATE SAMPLE COLLECTED
89	SWITCH NUMBER
*	SAMPLE LOCATION TERMINATED
**	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NJ PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. SAMPLES WITHIN THE GAUGE WERE COLLECTED FROM THE FIRST DEPTH INTERVAL WHERE BALLAST FINES WERE PRESENT.
4. REFUSAL ENCOUNTERED BELOW THE DEEPEST SAMPLING INTERVAL.
5. SAMPLES WERE COLLECTED BETWEEN JUNE AND SEPTEMBER 2016.
6. PCBs: POLYCHLORINATED BIPHENYLS

0 30 60



Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Date 1/22/2018 | Approved | Checked | Drawn JLM | Designed

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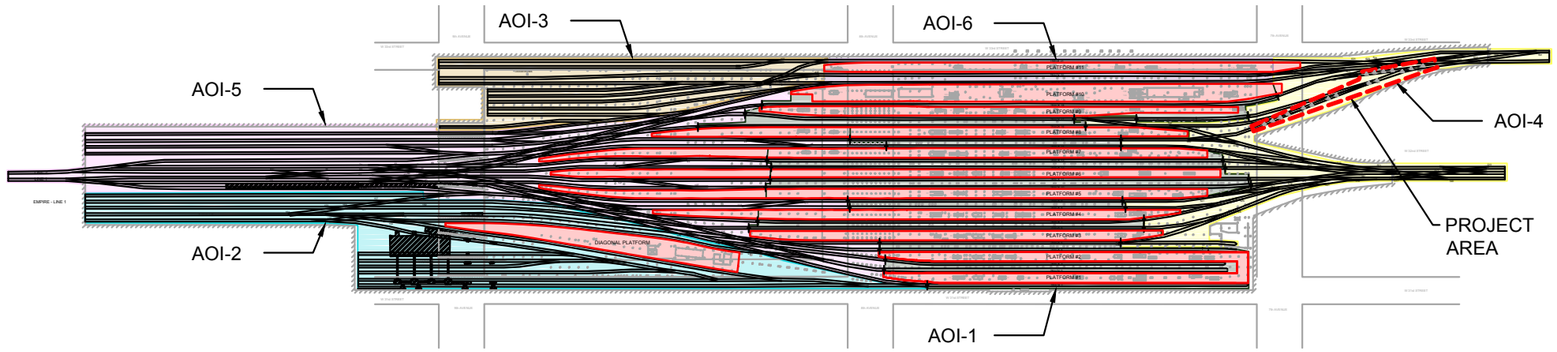
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
SWITCH 93 & 105
FIGURE 6d

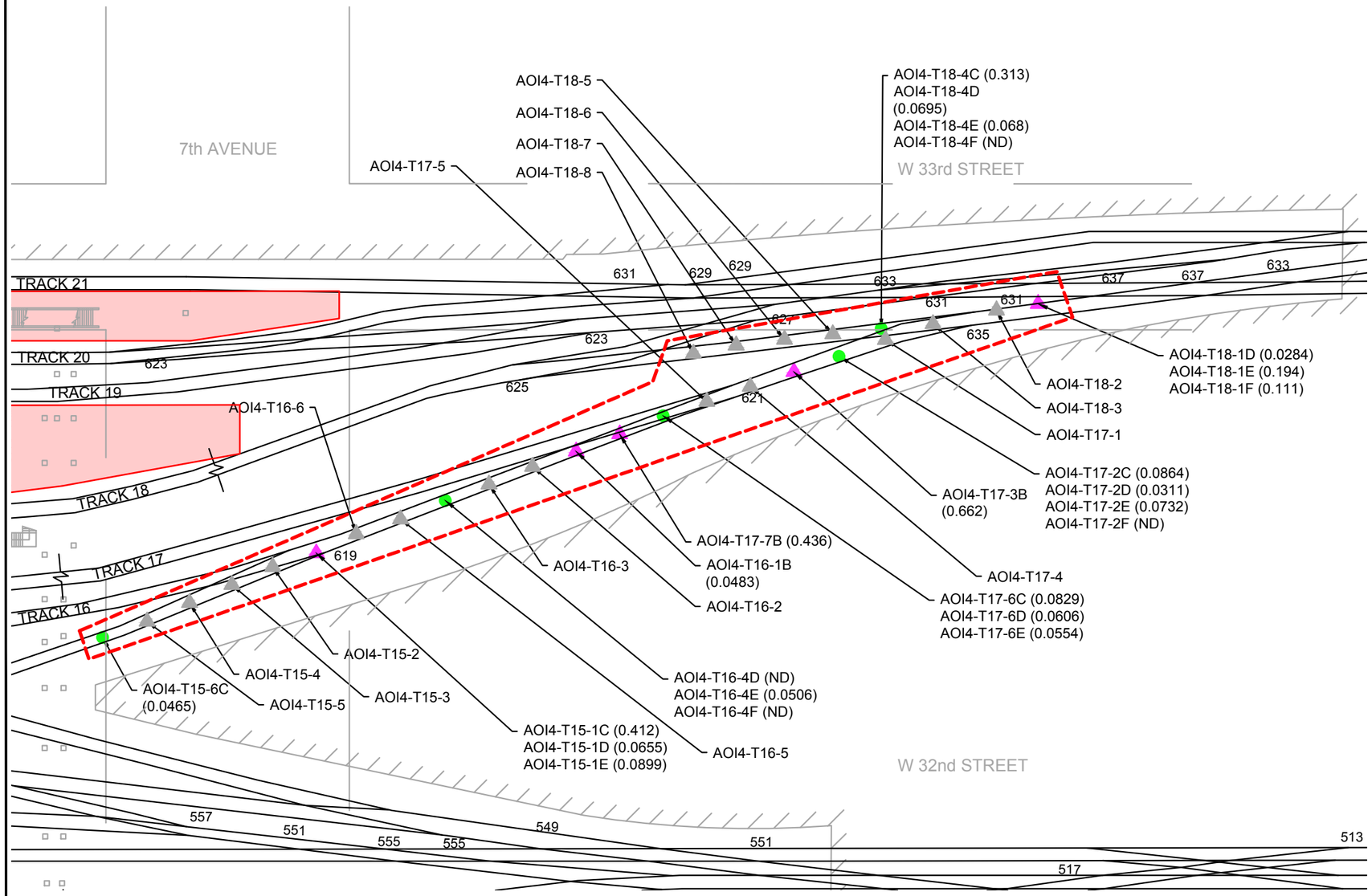
Job 372088 | No. | B/O | Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\SWITCH REPLACEMENT PROJECTS\PREVIOUS PRIORITY PROJECTS\DWG\AOI-5_SWITCH93&105.DWG

PLOT DATE: 01/22/2018 04:20 PM
SAVE DATE: 01/22/2018 04:19 PM



AREA OF INTEREST LOCATION PLAN



LEGEND:

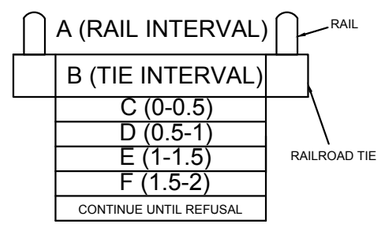
▲	SAMPLE LOCATION (SEE NOTE 3)
●	SAMPLE LOCATION (SEE NOTES 4 & 5)
▲	SAMPLE LOCATION (SEE NOTE 6)
(2.08)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
(1.32/1.55)	DUPLICATE SAMPLE COLLECTED
619	SWITCH NUMBER
*	SAMPLE LOCATION TERMINATED
**	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
⚡	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

N

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. SAMPLES WERE COLLECTED AT INTERVALS A & B (WHERE POSSIBLE).
4. SAMPLES WERE COLLECTED AT INTERVALS A, B, C, D, E, & F (WHERE POSSIBLE).
5. SAMPLE LOCATIONS WERE HAND DUG TO TOP OF BEDROCK RANGING FROM APPROXIMATELY 1 TO 2' BELOW THE BOTTOM OF TIE.
6. DUE TO THE PRESENCE OF BALLAST AT THIS LOCATION NO SAMPLES WERE COLLECTED FOR ANALYSIS.
7. THE SAMPLES WERE COLLECTED ON 8/6/2016.
8. PCBs: POLYCHLORINATED BIPHENYLS

0 50 100



Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Date	Approved	Checked	Drawn	Designed
1/22/2018			JLM	

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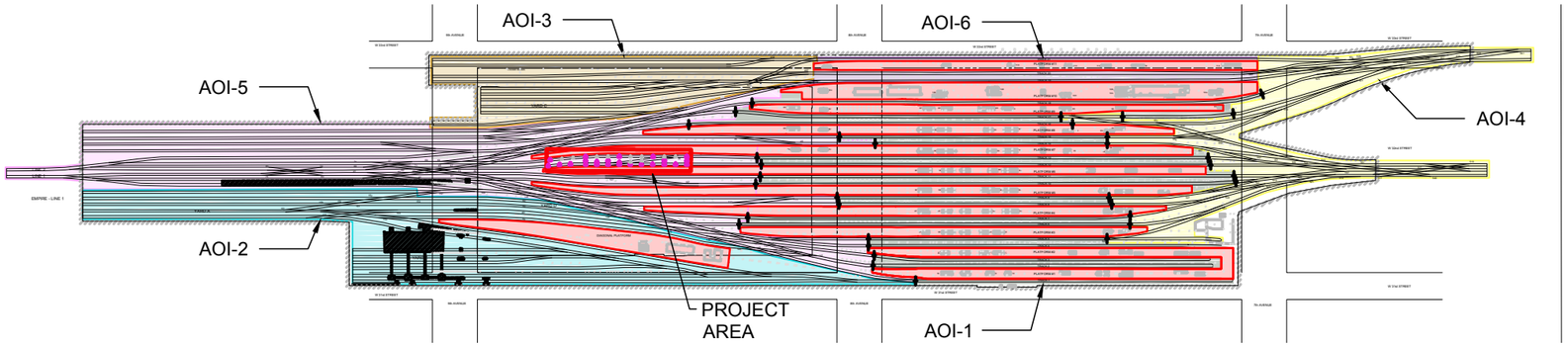
AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
SWITCH 619, 621, & 635
FIGURE 6e

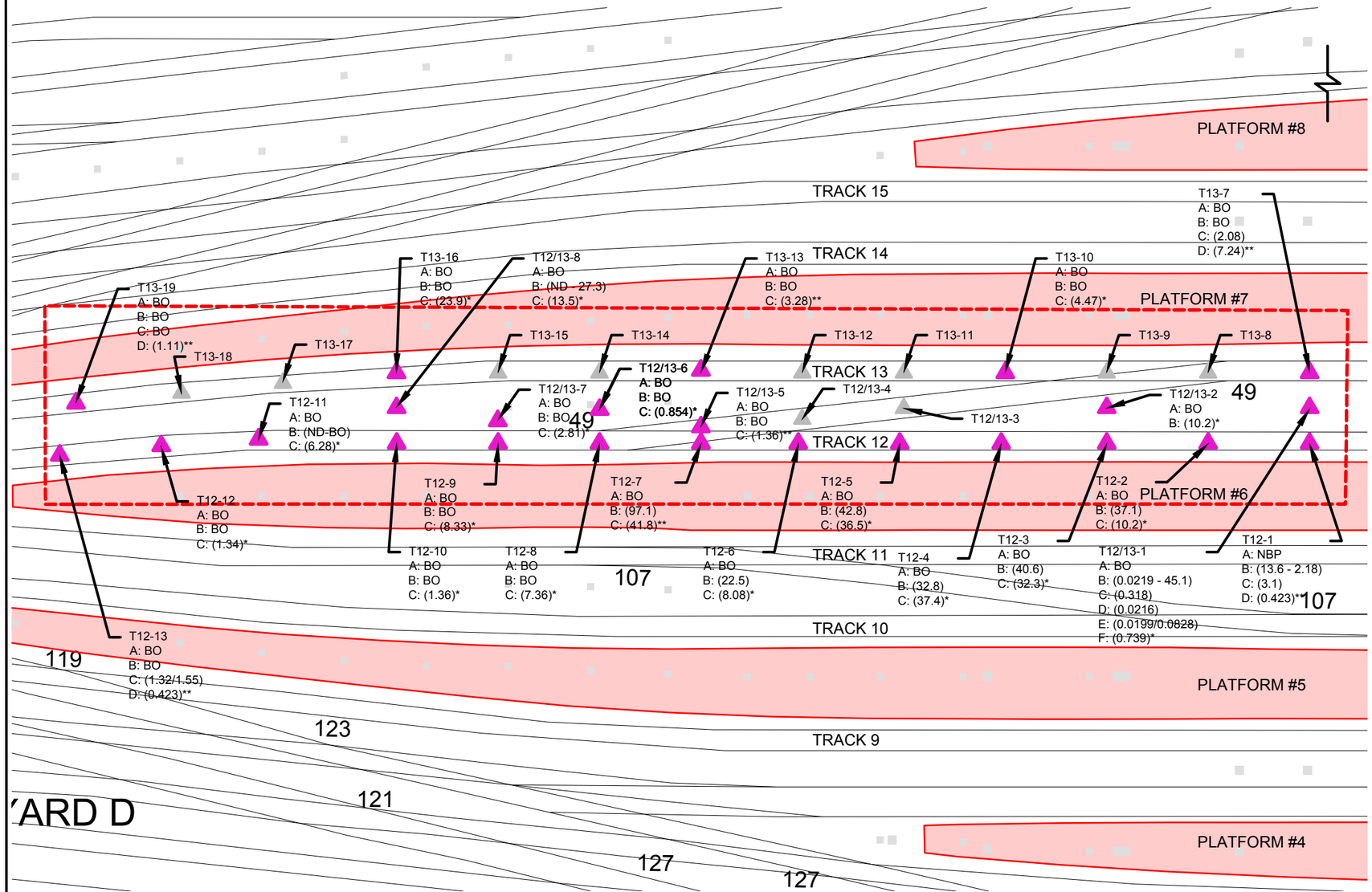
Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\1 - DRAWINGS\NEW FOLDER STRUCTURES\SWITCH REPLACEMENT PROJECTS\PREVIOUS PRIORITY PROJECTS\DWG\AOI-4_SWITCH619&621&635.DWG

PLOT DATE: 01/19/2018 03:50 PM
SAVE DATE: 01/19/2018 03:50 PM



AREA OF INTEREST LOCATION PLAN



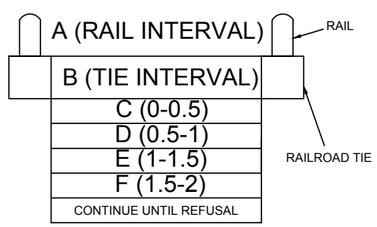
LEGEND:

▲	SAMPLE LOCATION
▲	NO SAMPLE COLLECTED AS NO BALLAST FINES WERE ENCOUNTERED
(2.08)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG)
(1.32/1.55)	DUPLICATE SAMPLE COLLECTED
49	SWITCH NUMBER
BO	BALLAST STONE ONLY WITH NO FINES ENCOUNTERED
NBP	NO BALLAST PRESENT
ND	NOT DETECTED
(ND-27.3)	BALLAST STONE SAMPLE RESULT - BALLAST FINES RESULT
*	SAMPLE LOCATION TERMINATED
**	REFUSAL ENCOUNTERED BELOW SAMPLE DEPTH
⚡	TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.
2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.
3. THE DATA PRESENTED HAS UNDERGONE VALIDATION.
4. PCBs: POLYCHLORINATED BIPHENYLS

0 25 50



Revision	
Date	
Professional Engineer - N.J. Lic. No.	Date
Date	1/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

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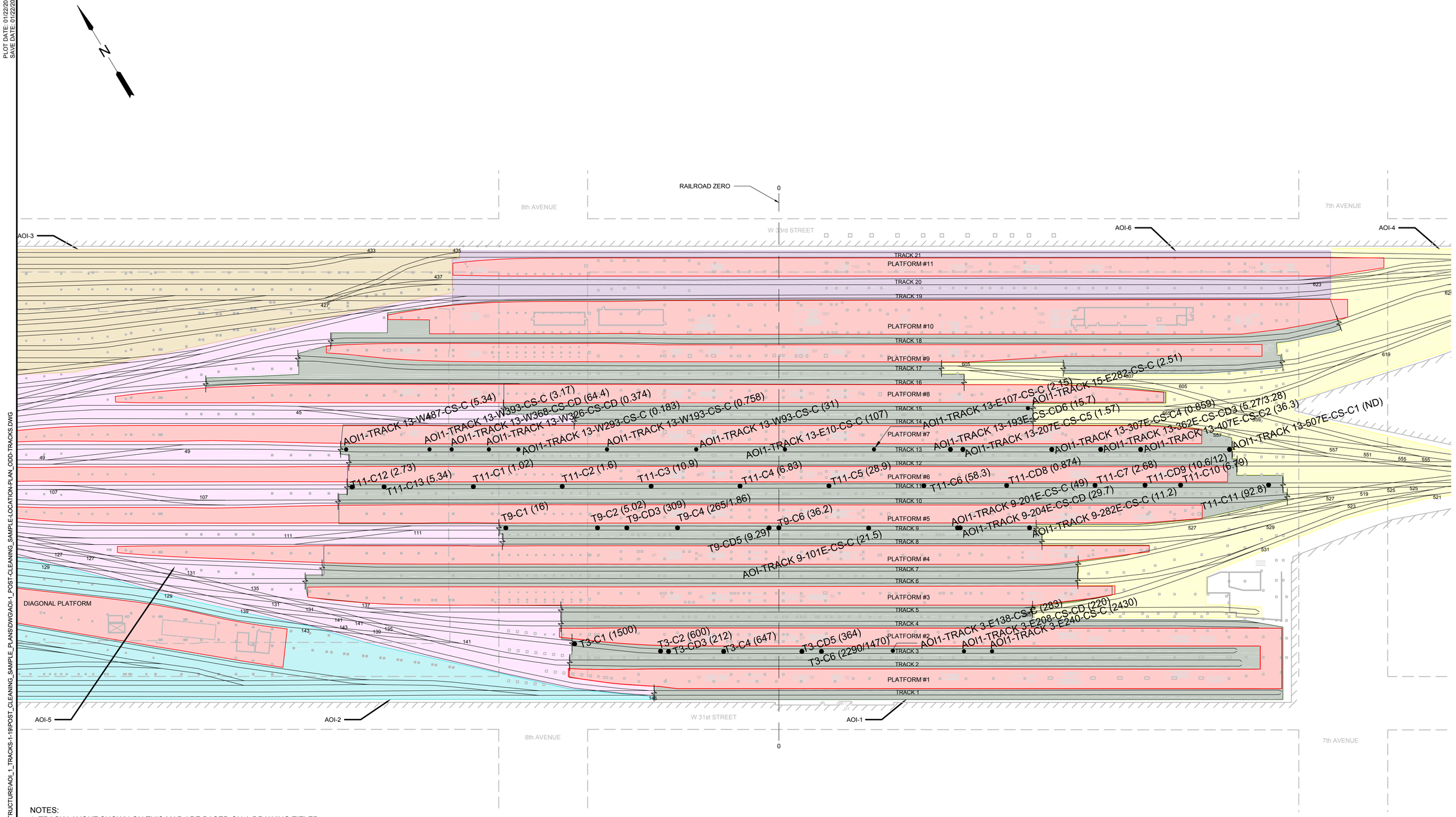
AMTRAK
PENNSYLVANIA STATION, NEW YORK
ANALYTICAL RESULTS AT SOGR PROJECTS
SWITCH 49
FIGURE 6f

Job	No.	
372088	B/O	Total

\\MIL-DATA.HMMG.CC\PROJECTS-I\NE\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\SWITCH REPLACEMENT PROJECTS\AOI-5_49\AOI-5_SWITCH-49_-SAMPLE-LOCATION-PLAN.DWG

PLOT DATE: 01/22/2018 4:27 PM
SAVE DATE: 01/22/2018 4:27 PM

P:\372088 PENN STATION NYI - DRAWINGS\NEW FOLDER STRUCTURE\AOI_1_TRACKS-1-19\POST_CLEANING_SAMPLE_PLANS\DWG\AOI-1_POST-CLEANING_SAMPLE-LOCATION-PLAN_ODD-TRACKS.DWG



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

- ● T3-C2: CONCRETE SAMPLE LOCATION (AFTER FINAL WASHING)/PCB RESULT IN PARENTHESES
- T9-CD3: POST CLEANING AFTER FINAL WASHING CONCRETE SAMPLE LOCATION ADJACENT TO DRAIN
- (2.73) DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
- (10.6/12) INDICATES DUPLICATE SAMPLE OBTAINED
- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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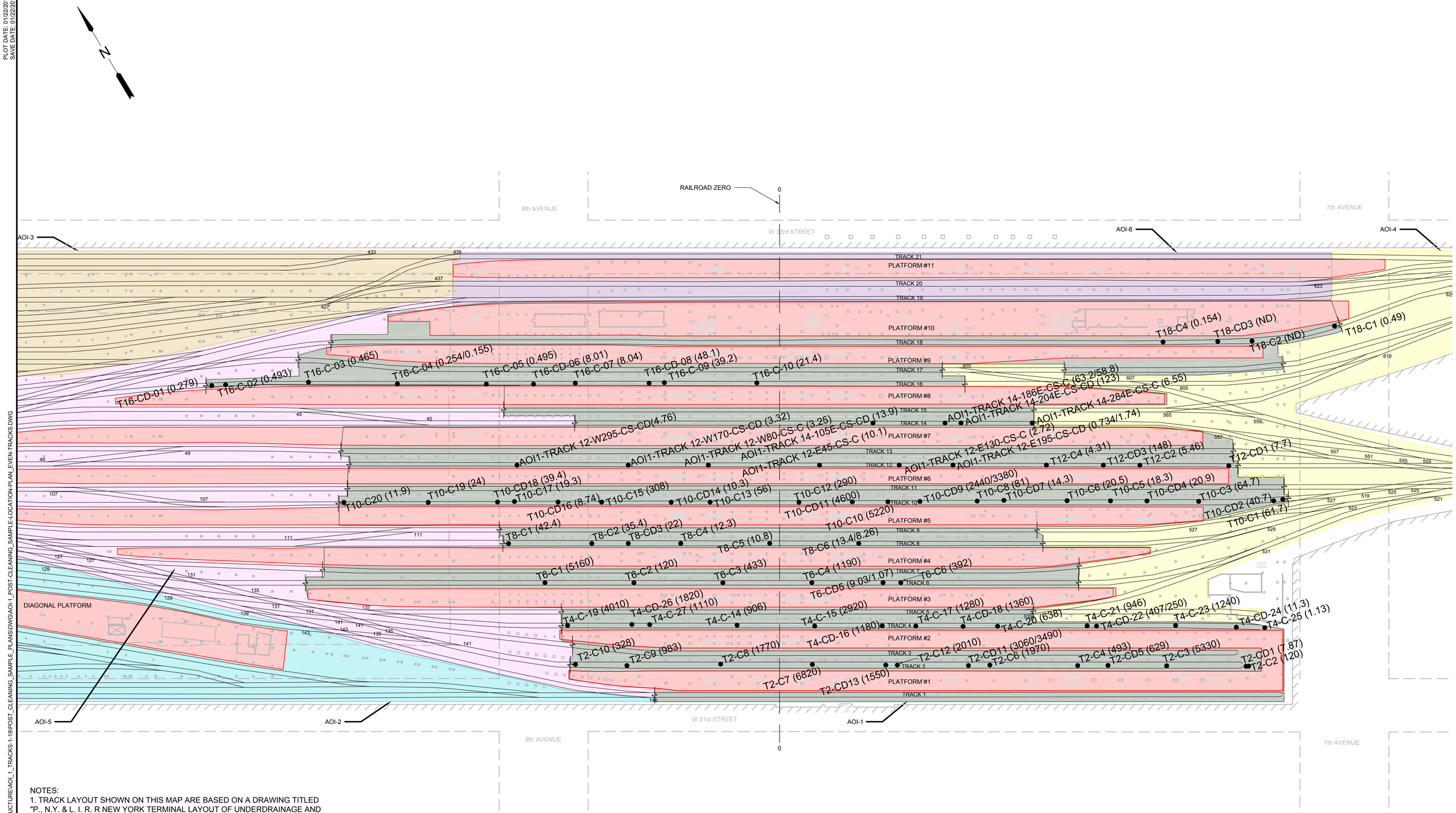
AMTRAK
PENNSYLVANIA STATION, NEW YORK
CONCRETE ANALYTICAL RESULTS (AFTER FINAL WASHING)
ODD TRACKS
FIGURE 7a

Job	No.	
372088	B/O	Total

PLOT DATE: 01/22/2018 4:05 PM
SAVE DATE: 01/22/2018 4:05 PM

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_1_TRACKS-1-19\POST_CLEANING_SAMPLE_PLANS\DWG\AOI-1_POST-CLEANING_SAMPLE-LOCATION-PLAN_EVEN-TRACKS.DWG



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.

3. RESULTS OF PCBs (SUM OF AROCLORS) ARE REPORTED IN MILLIGRAMS PER KILOGRAM (MG/KG).

4. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

5. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

- ● CONCRETE SAMPLE LOCATION (AFTER FINAL WASHING)/PCB RESULT IN PARENTHESES
  T4-CD26: POST CLEANING AFTER FINAL WASHING CONCRETE SAMPLE LOCATION ADJACENT TO DRAIN
- (10.9) DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS)
- (2440/3380) INDICATES DUPLICATE SAMPLE OBTAINED
- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	J.L.M.			1/22/2018

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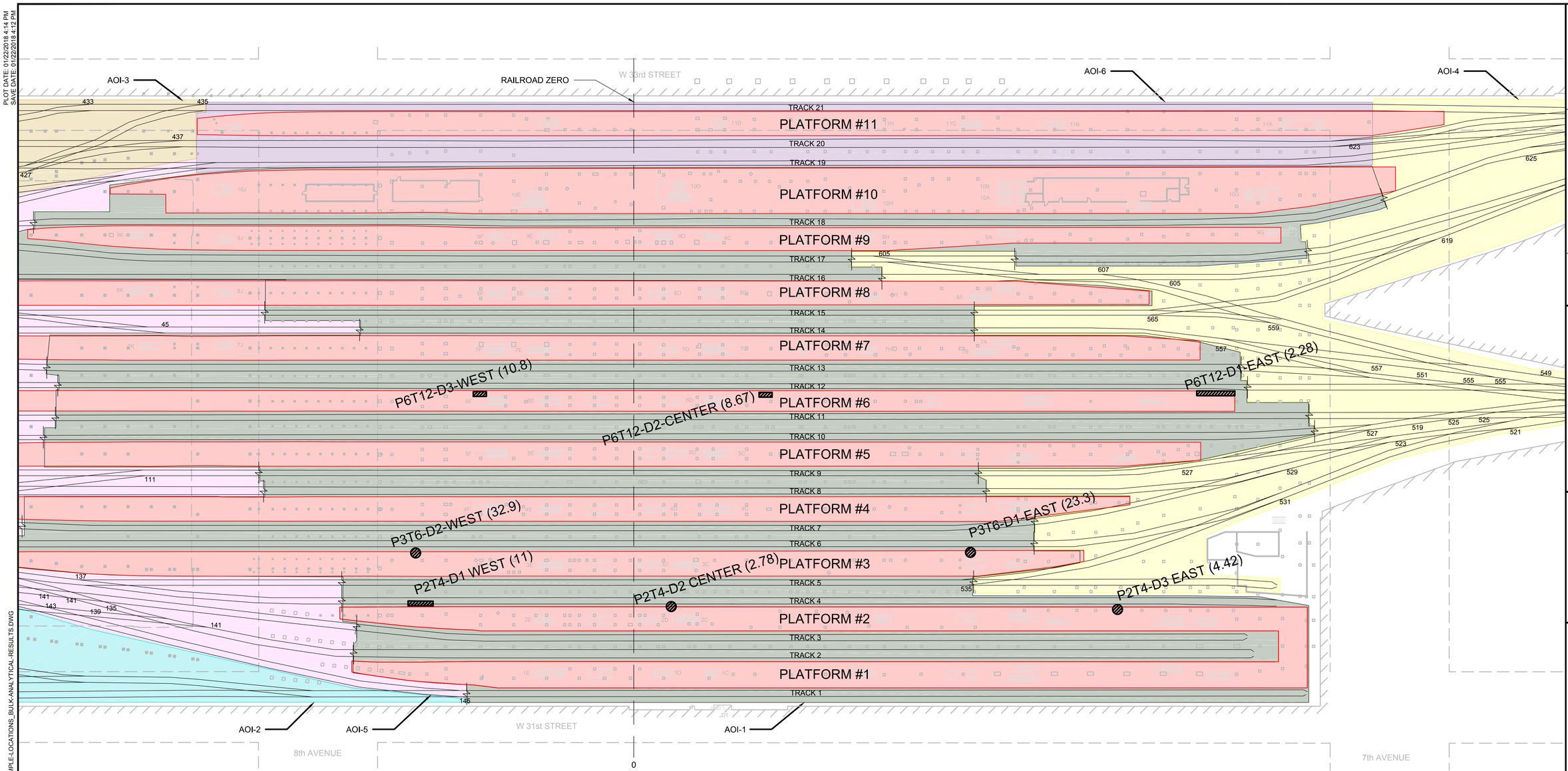
AMTRAK
PENNSYLVANIA STATION, NEW YORK
CONCRETE ANALYTICAL RESULTS (AFTER FINAL WASHING)
EVEN TRACKS
FIGURE 7b

Job	No.
372088	
	B/O / Total

PLOT DATE: 01/22/2018 4:14 PM
SAVE DATE: 01/22/2018 4:12 PM



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

3. ALL DUST SAMPLES WERE COLLECTED FROM THE TOP SURFACE OF LIGHT FIXTURES BETWEEN AUGUST 9 AND AUGUST 31, 2016.

4. A DUST SAMPLE WAS NOT COLLECTED FROM THE CENTER OR WEST END OF TRACK 6 BECAUSE NO SIGNIFICANT AMOUNT OF DUST WAS PRESENT.

5. A DUST SAMPLE WAS NOT COLLECTED FROM THE WEST END OF TRACK 12 BECAUSE NO SIGNIFICANT AMOUNT OF DUST WAS PRESENT.

6. PCBs: POLYCHLORINATED BIPHENYLS

LEGEND:

- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM
- P3T6-D2 WEST: DUST SAMPLE COLLECTED FROM A SINGLE LIGHT FIXTURE (2016)
- P2T4-D1 WEST: DUST SAMPLE COLLECTED FROM A SPAN OF LIGHT FIXTURES (2016)
- (4.42) DETECTED CONCENTRATION OF PCBs (SUM OF AROCLORS) IN MG/KG

N

0 80 160

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

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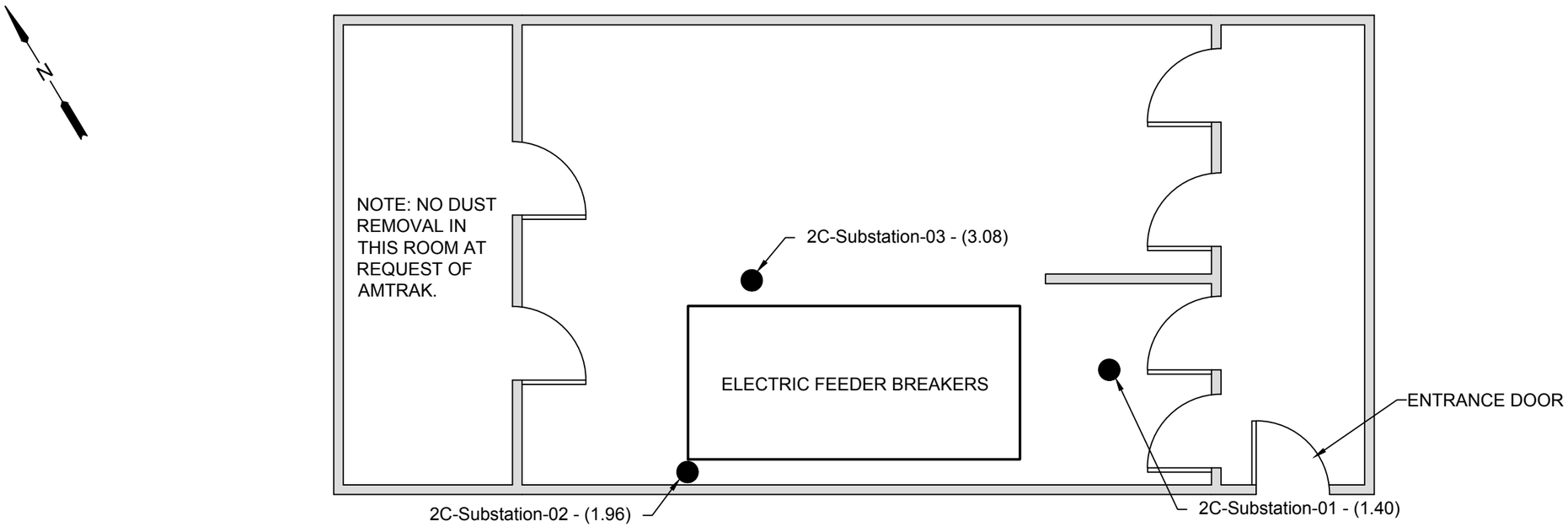
AMTRAK
PENNSYLVANIA STATION, NEW YORK
BULK DUST ANALYTICAL RESULTS
FIGURE 8

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RIWP\DWG\AOI-1-2-3-4-5-6_DUST-SAMPLE-LOCATIONS_BULK-ANALYTICAL-RESULTS.DWG



LEGEND:

●	DUST SAMPLE LOCATION
2C-Substation-01	SAMPLE NUMBER
(1.96)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)
	DOOR

NOTES:

1. DUST SAMPLE LOCATIONS ARE APPROXIMATE
2. THE DATA PRESENTED HAS UNDERGONE VALIDATION.
3. PCBs: POLYCHLORINATED BIPHENYLS

0 5 10

Revision	Date

Professional Engineer - N.J. Lic. No. _____ Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

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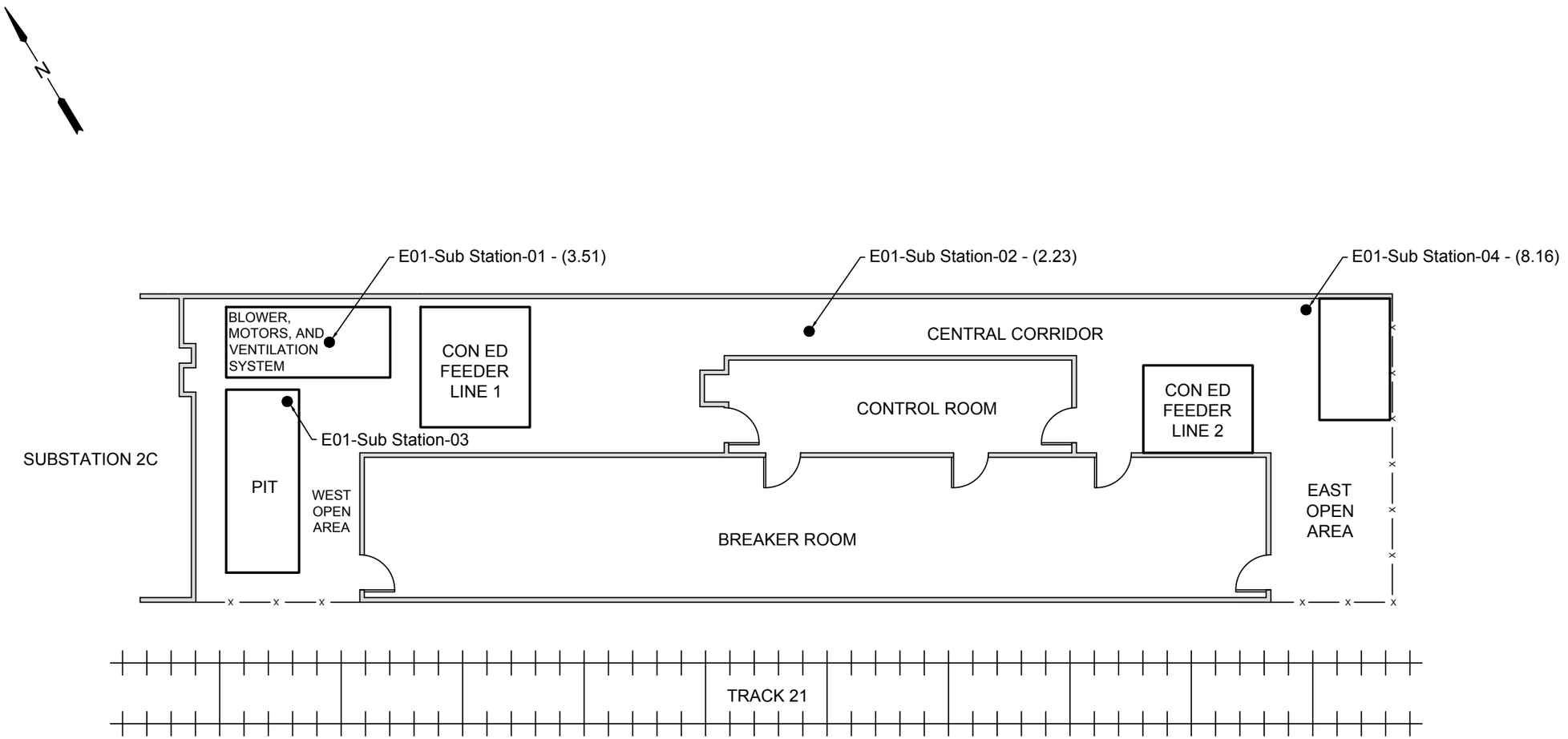
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
SUBSTATION ANALYTICAL RESULTS
2C SUBSTATION
FIGURE 9a

Job	No.	
372088	B/O	Total



LEGEND:

●	DUST SAMPLE LOCATION
E01-Sub Station-01	SAMPLE NUMBER
(3.51)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)
—x—	CHAIN LINK FENCE
	DOOR

NOTES:

1. DUST SAMPLE LOCATIONS ARE APPROXIMATE.
2. THE E01 SUBSTATION IS CURRENTLY USED AND OCCUPIED BY LONG ISLAND RAILROAD.
3. A SAMPLE WAS COLLECTED AT LOCATION E01-SUB STATION-03 BUT A SUFFICIENT AMOUNT OF VOLUME WAS NOT PRESENT TO COMPLETE THE ANALYSIS.
4. THE DATA PRESENTED HAS UNDERGONE VALIDATION.
5. PCBs: POLYCHLORINATED BIPHENYLS

0 10 20

Revision	Date

Professional Engineer - N.J. Lic. No. Date

Date	1/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

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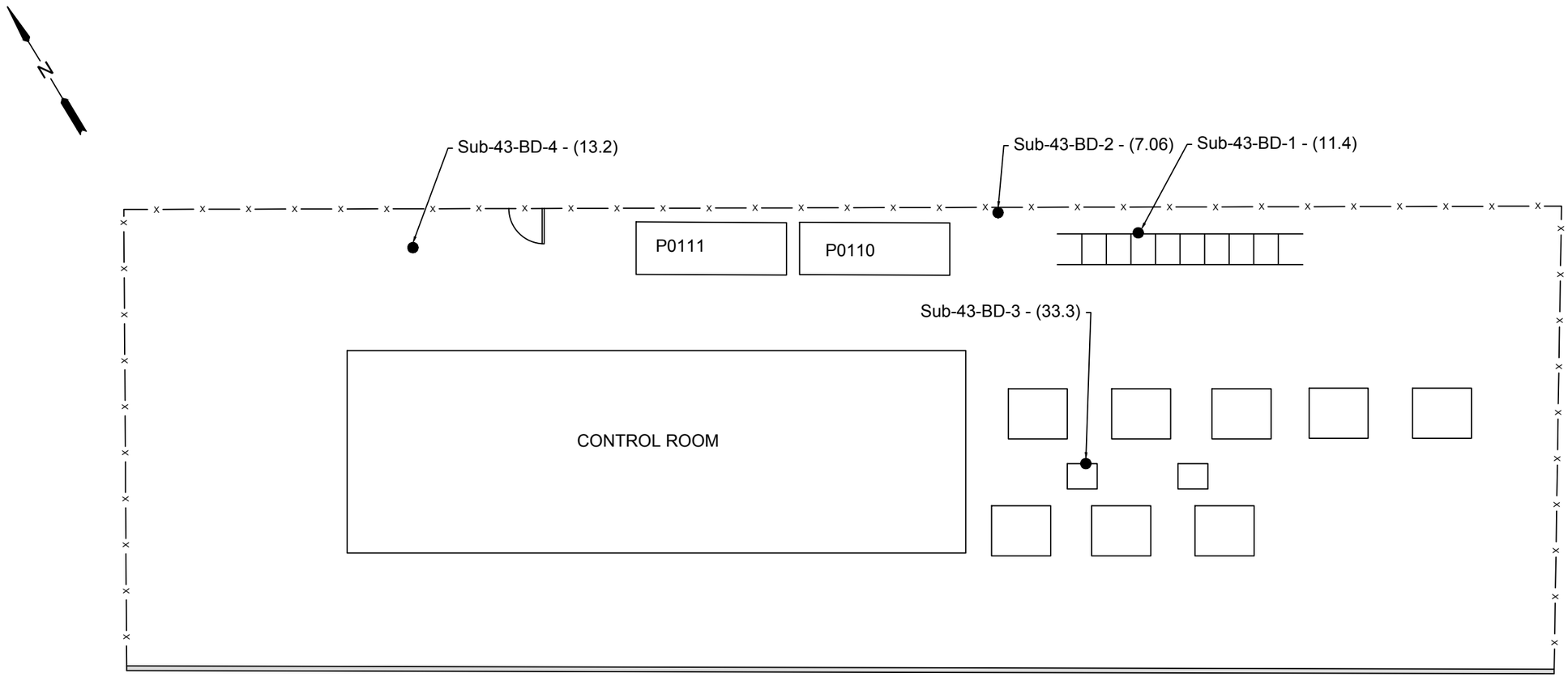
AMTRAK
PENNSYLVANIA STATION, NEW YORK
SUBSTATION ANALYTICAL RESULTS
E01 SUBSTATION

FIGURE 9b

Job	No.	
372088	B/O	Total



LEGEND:

●	DUST SAMPLE LOCATION
43-Sub-BD-1	SAMPLE NUMBER
(11.4)	DETECTED CONCENTRATION OF TOTAL PCBs (SUM OF AROCLORS) IN MILLIGRAMS PER KILOGRAM (MG/KG)
—x—	CHAIN LINK FENCE
	DOOR
	LADDER HUNG ON WALL
	APPROXIMATE LOCATION OF TRANSFORMER/SWITCH

NOTES:

1. DUST SAMPLE LOCATIONS ARE APPROXIMATE.
2. DRAWING IS NOT TO SCALE.
3. PCBs: POLYCHLORINATED BIPHENYLS

Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Date 1/22/2018
Approved
Checked
Drawn JLM
Designed

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111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
SUBSTATION ANALYTICAL RESULTS
SUBSTATION 43

FIGURE 9c

Job 372088 | No. | B/O | Total

AREA OF INTEREST (AOI) LOCATION PLAN
NOT TO SCALE

- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM

NOTES:

1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NJ PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. THE SAMPLING LOCATIONS ARE APPROXIMATE; ACTUAL LOCATIONS WILL BE DETERMINED BASED ON FIELD OBSERVATIONS.

3. RAILROAD ZERO DENOTES A REFERENCE POINT AT PENNSYLVANIA STATION, NEW YORK. SAMPLE LOCATIONS WILL BE REFERENCED FROM RAILROAD ZERO.

4. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

5. THE PROPOSED EXCAVATION AREA IS EXCLUDED FROM THE PROJECT. THIS AREA WILL BE REMEDIATED AS DESCRIBED IN A SELF-IMPLEMENTING CLEANUP PLAN PROPOSED BY ARKF AND DATED 11/6/17. NO SAMPLES WILL BE TAKEN IN EXCAVATION AREAS.

LEGEND:

- PROPOSED TEST PIT LOCATION TO 0.5 FEET BELOW THE RAILROAD TIE
- PROPOSED TEST PIT LOCATION TO BE COMPLETED UNTIL REFUSAL IS ENCOUNTERED. SURFACE BALLAST SAMPLE WILL ALSO BE COLLECTED.
- 89 SWITCH NUMBER
- BALLAST/CONCRETE TRANSITION
- WALLS
- PROPOSED EXCAVATION AREA (BOP SE LLC, OTHERWISE KNOWN AS "BROOK FIELD" EXCLUDED FROM RIWP)

0 50 100

P:\372088 PENN STATION NYI - DRAWINGS\NEW FOLDER STRUCTURE\MULTIPLE_AOIS\F

AMTRAK
PENNSYLVANIA STATION, NEW YORK
PROPOSED BALLAST STONE AND BALLAST FINES SAMPLE LOCATION MAP
FIGURE 10

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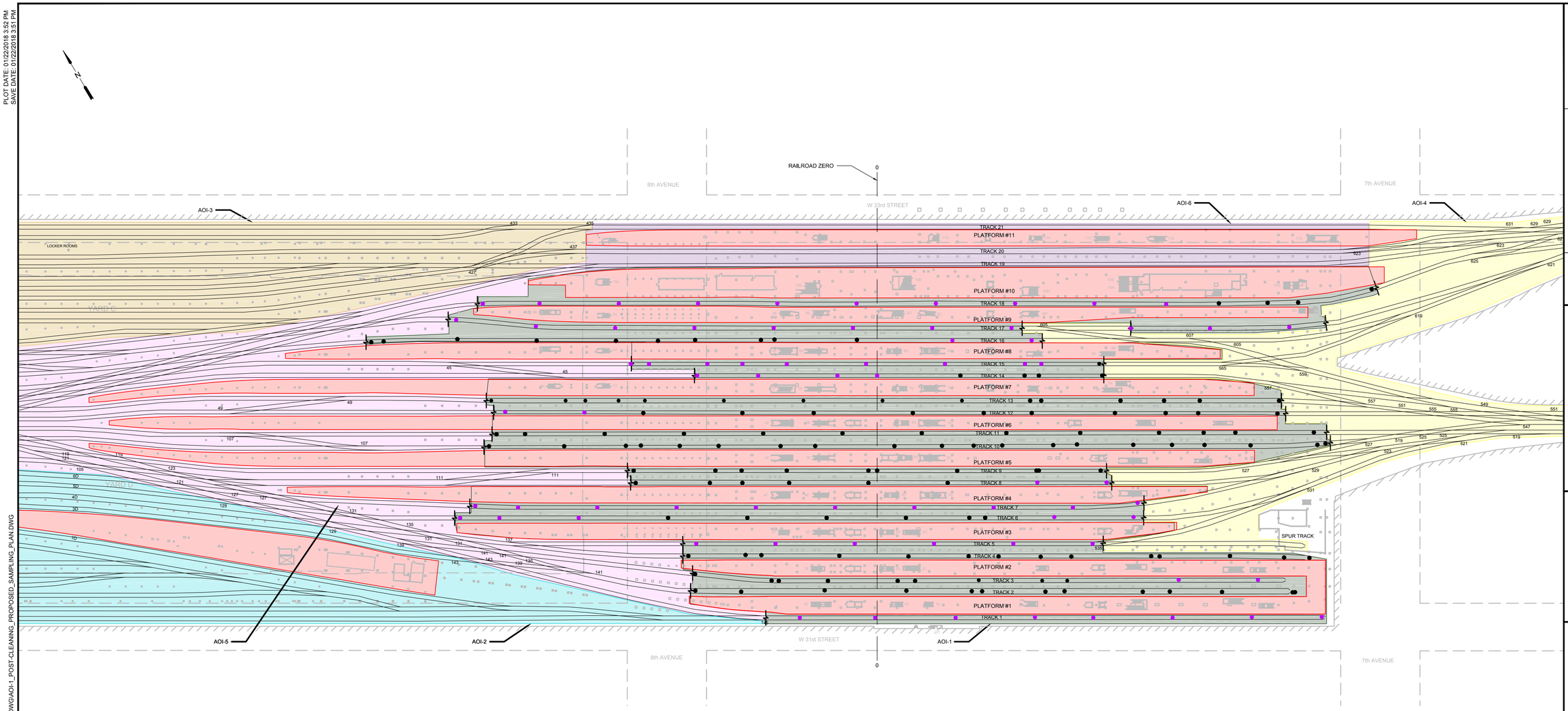
Professional Engineer - N.J. Lic. No. ______ Date ______

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

Job	No.	
372088	B/O	Total

PLOT DATE: 01/22/2018 3:52 PM
SAVE DATE: 01/22/2018 3:51 PM

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\AOI_1_TRACKS-1-19\POST_CLEANING_SAMPLE_PLANS\DWG\AOI-1_POST-CLEANING_PROPOSED_SAMPLING_PLAN.DWG



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

3. THE SAMPLING LOCATIONS ARE APPROXIMATE; ACTUAL LOCATIONS WILL BE DETERMINED BASED ON FIELD OBSERVATIONS.

4. THE CONCRETE ADJACENT TO THE DRAINS WILL ALSO BE SAMPLED AS ENCOUNTERED.

5. CONCRETE SAMPLES COLLECTED ON MAP ARE AS OF 12/1/2017.

LEGEND:

- CONCRETE SAMPLE COLLECTED (AFTER FINAL WASHING)
- PROPOSED CONCRETE SAMPLE LOCATION
- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM

0 120 240

Revision	Date

Professional Engineer - N.J. Lic. No. Date

Date	1/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

MOTT MACDONALD
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
PROPOSED CONCRETE SAMPLING LOCATIONS AFTER FINAL WASHING
FIGURE 11

Job	No.	
372088	B/O	Total

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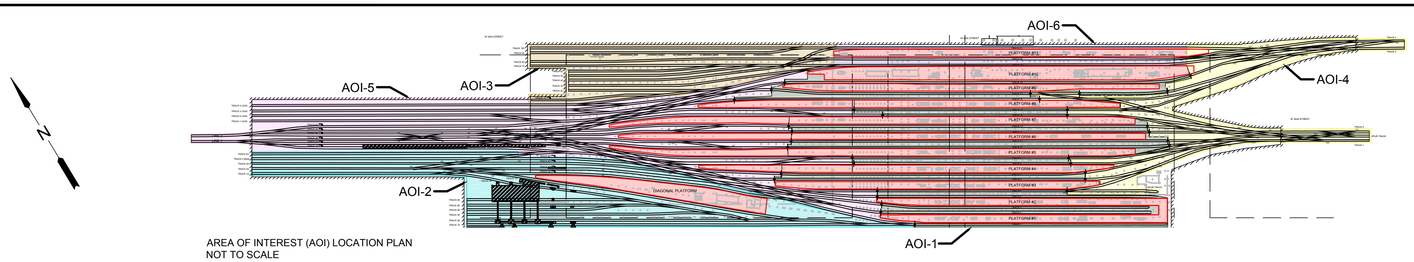
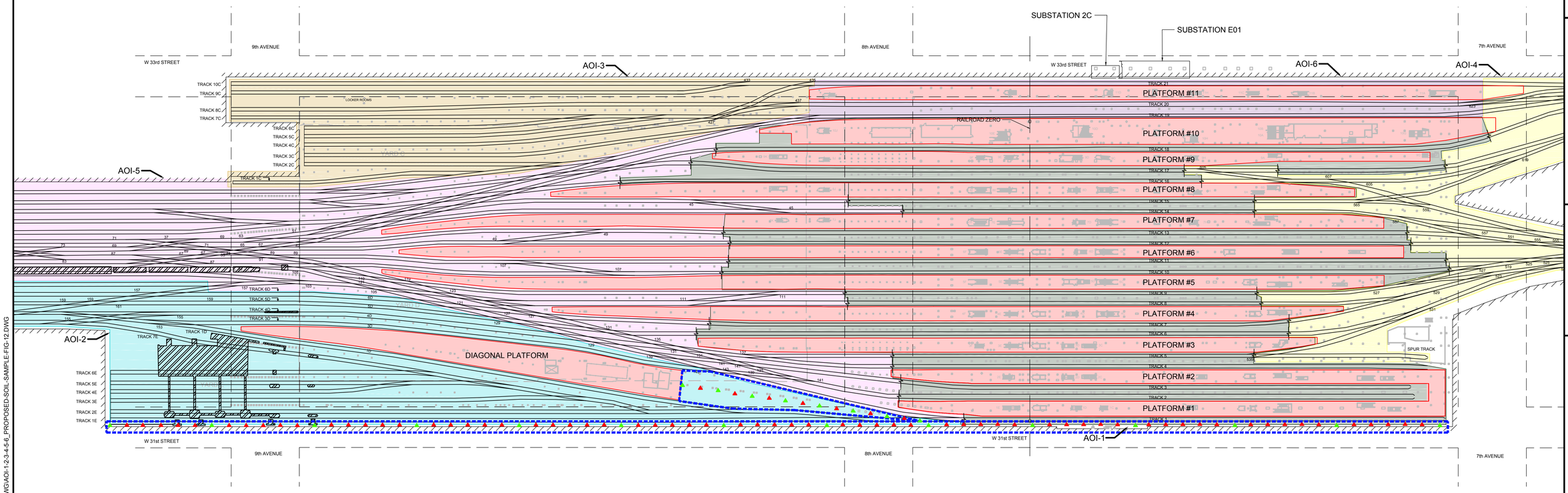
SAVE DATE: 01/22/2018 3:42 PM



AREA OF INTEREST (AOI) LOCATION PLAN
NOT TO SCALE

- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. RAILROAD ZERO DENOTES A REFERENCE POINT AT PENNSYLVANIA STATION, NEW YORK. SAMPLE LOCATIONS WILL BE REFERENCED FROM RAILROAD ZERO.

3. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

4. THE PROPOSED EXCAVATION AREA IS EXCLUDED FROM THE PROJECT. THIS AREA WILL BE REMEDIATED BY BOP SE LLC (COMMONLY REFERRED TO AS BROOK FIELD) AS DESCRIBED IN A SELF-IMPLEMENTING CLEANUP PLAN PROPOSED BY ARKF DATED 11/6/17

LEGEND:

- 89 SWITCH NUMBER
- BALLAST/CONCRETE TRANSITION
- WALLS
- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM
- PROPOSED EXCAVATION AREA (BOP SE LLC, OTHERWISE KNOWN AS "BROOK FIELD" EXCLUDED FROM RIWP)
- AREAS WHERE SOIL MAY BE PRESENT
- PROPOSED SOIL SAMPLE LOCATION TO 0.5 FEET BELOW THE RAILROAD TIE INTERVAL
- PROPOSED SOIL SAMPLE LOCATION TO REFUSAL

0 70 140

Revision	Date

Professional Engineer - N.J. Lic. No.

Date 1/22/2018
Approved
Checked
Drawn JLM
Designed

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
PROPOSED SOIL SAMPLE LOCATIONS
FIGURE 12

Job 372088 | No. | B/O | Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\MULTIPLE_AOIS\FULL_STATION\DWG\AOI-1-2-3-4-5-6_PROPOSED-SOIL-SAMPLE-FIG-12.DWG

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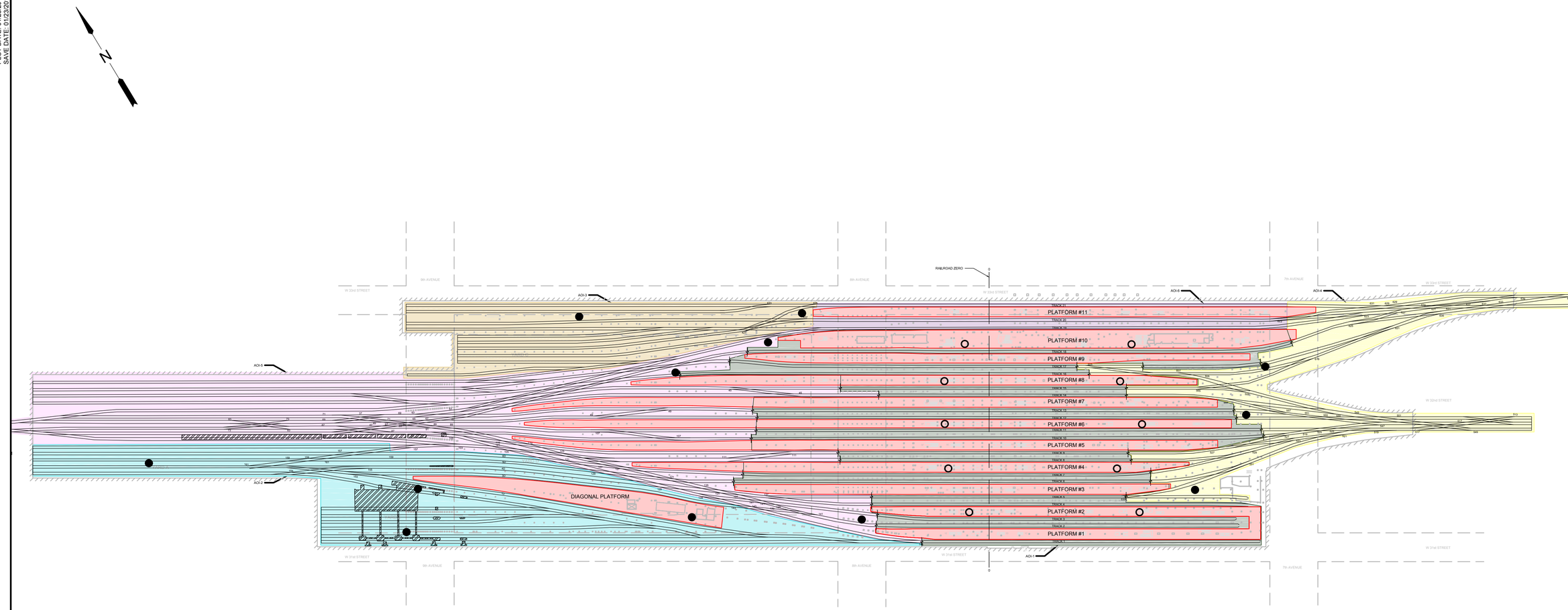

NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

3. BULK DUST SAMPLES WILL BE COLLECTED FROM SURFACES WHERE A SUFFICIENT AMOUNT OF DUST IS PRESENT FOR ANALYSIS.

4. THE PROPOSED EXCAVATION AREA IS EXCLUDED FROM THE PROJECT. THIS AREA WILL BE REMEDIATED BY BOP SE L.L.C. (COMMONLY REFERRED TO AS BROOK FIELD) AS DESCRIBED IN A SELF-IMPLEMENTING CLEANUP PLAN PROPOSED BY ARKF DATED 11/6/2017.

LEGEND:

- TRANSITION FROM CONCRETE TRACK STRUCTURE TO BALLAST
- LIMITS OF PLATFORM
- BULK DUST SAMPLE LOCATION AT AOI-2 THROUGH AOI-6
- BULK AIR SAMPLE LOCATION AT PLATFORM LEVEL
- PROPOSED EXCAVATION AREA (BOP SE LLC, OTHERWISE KNOWN AS "BROOK FIELD" EXCLUDED FROM RIWP)

0 200 400

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Designed	Drawn	Checked	Approved	Date
	JLM			1/22/2018

M M MOTT MACDONALD
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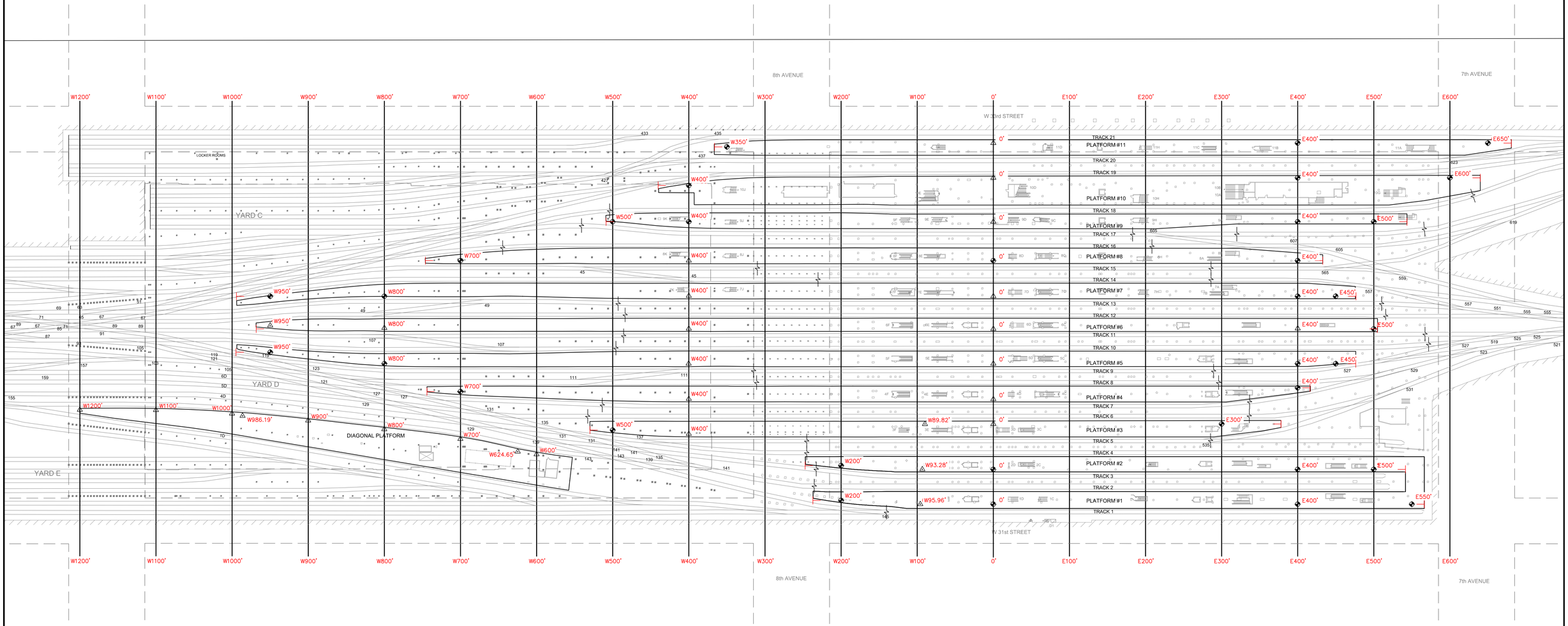
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
PROPOSED BULK DUST AND AIR SAMPLING LOCATIONS AT PLATFORM / TRACK LEVEL
FIGURE 13

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RIWP\DWG\AOI-1-2-3-4-5-6_DUST-SAMPLE-LOCATIONS.DWG



TRACK AND PLATFORM LAYOUT AS SHOWN ON THIS PLAN IS SHOWN FOR REFERENCE ONLY. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

PLATFORM	WEST END STATION	EAST END STATION
11	W 366.3	E 680.6
10	W 440.6	E 639.8
9	W 508.8	E 543.5
8	W 746.2	E 432.8
7	W 994.5	E 476.5
6	W 968.5	E 504.5
5	W 995.1	E 476.2
4	W 744.2	E 416.1
3	W 530.0	E 378.0
2	W 247.3	E 541.5
1	W 237.1	E 566.3
DIAGONAL	W 1200.2	E 552.7

NOTE: END OF PLATFORMS MEASURED FROM STATION 0'

LEGEND

△ SET NAIL IN CONCRETE
MEASURED STATION AND COORDINATES

◆ SET NAIL IN CONCRETE
MEASURED STATION ONLY

⊣ MEASURED END OF PLATFORM

N

Cadd: P:\372088 Penn Station NY\I - Drawings\Master Base Drawing\Platform Station Markout Plan.dwg

Scale 1"=50' | Job 372088 | Book – | File PENN STATION
B/O – | No. – | Page –
Total –

AMTRAK

PENNSYLVANIA STATION, NEW YORK
BENCHMARK LOCATIONS

FIGURE 14

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Tel: 908.730.6000 Fax: 973.267.2890

Designed	Drawn	Checked	Approved	Date
–	JLM			1/22/18

Date	Revision

Tables

Table 1

Conceptual Site Model for the Passenger Envelope

Table 1 – Conceptual Site Model for the Passenger Envelope

Sources of Potential Exposure from PCBs	Potential Exposure Routes	Passengers	House Keeping	Amtrak / NJ Transit Customer Service	Amtrak Porters	Police/Security	LIRR Customer Service	Visitors	Vendors	Railroad Worker
Settled Dust on Accessible Surfaces on Passenger Platforms	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**N/A**	**Complete**	**Complete**	**N/A**	**N/A**	**N/A**	**Complete**
Settled Dust on Accessable Surfaces Lower & Upper Concourses	Dermal Contact & Incidental Ingestion	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**	**Incomplete/ Not Significant**
Air in Platform Area	Inhalation	**Complete**	**Complete**	**N/A**	**Complete**	**Complete**	**N/A**	**N/A**	**N/A**	**Complete**
Air in Lower Level Concourse	Inhalation	**Complete**	**Complete**	**N/A**	**N/A**	**Complete**	**Complete**	**Complete**	**Complete**	**Incomplete/ Not Significant**
Air in Upper Level of Concourse	Inhalation	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**	**N/A**	**Complete**	**Complete**	**Incomplete/ Not Significant**

Notes:

Complete: Exposure pathway is complete or potentially complete.

Incomplete/Not Significant: Exposure pathway is incomplete, or is potentially complete but insignificant.

N/A: Exposure pathway is not applicable because the receptior is not frequently present in this area of the Passenger Envelope.

Revised: January 23, 2018

Table 2

Conceptual Site Model for the Restricted Areas of PSNY

Table 2 – Conceptual Site Model for the Restricted Areas of PSNY

Sources of Potential Exposure from COCs	Potential Exposure Routes	Engineering (1)	Engineering (2)	Production Engineering (major construction)	Mechanical Department	Transportation Workers, Train Engineers/ Conductors
Concrete surfaces of Concrete Track Structure (Track Level)*	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Dust / Surfaces and Equipment present on Concrete Track Structure (Track Level)	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Dust / Surfaces and Equipment Outside of Platform Area and Concrete Track Structure	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Dust / Surfaces and Equipment present at Platform Area	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Air in Track Level	Inhalation	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Air in Platform Area	Inhalation	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**
Sediments, Fines, and Ballast Stone present at track level beyond concrete track structure	Dermal Contact & Incidental Ingestion	**Complete**	**Complete**	**Complete**	**Complete**	**Complete**

Notes:

COCs: Contaminants of Concern include but may not be limited to PCBs.

Complete: Exposure pathway is complete or potentially complete.

Incomplete/Not Significant: Exposure pathway is incomplete or is potentially complete but insignificant.

***** After sediment and "tar-like" material removal.

Revised: January 23, 2018

Table 3

Sample Analytical Methods and Frequency

Table 3
Sample Analytical Methods and Frequency
Penn Station, New York

Media	Analysis Methods	Frequency	Minimum Quantity
Ballast Fines	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of deep sample locations	220 grams (32 ounces)
Ballast Stone	PCBs	All sample locations	45 grams (8 ounces)
Concrete	PCBs	All sample locations	45 grams (8 ounces)
Soil	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of deep sample locations	220 grams (32 ounces)
Sediment in Drains	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of sediment in drains sample locations	220 grams (32 ounces)
Bulk Dust	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of bulk dust sample locations	220 grams (32 ounces)
Air	PCBs	All sample locations	1500 – 2000 liters

Abbreviations:
TCL +30: Target Compound List plus a 30-compound library search
TAL: Target Analyte List

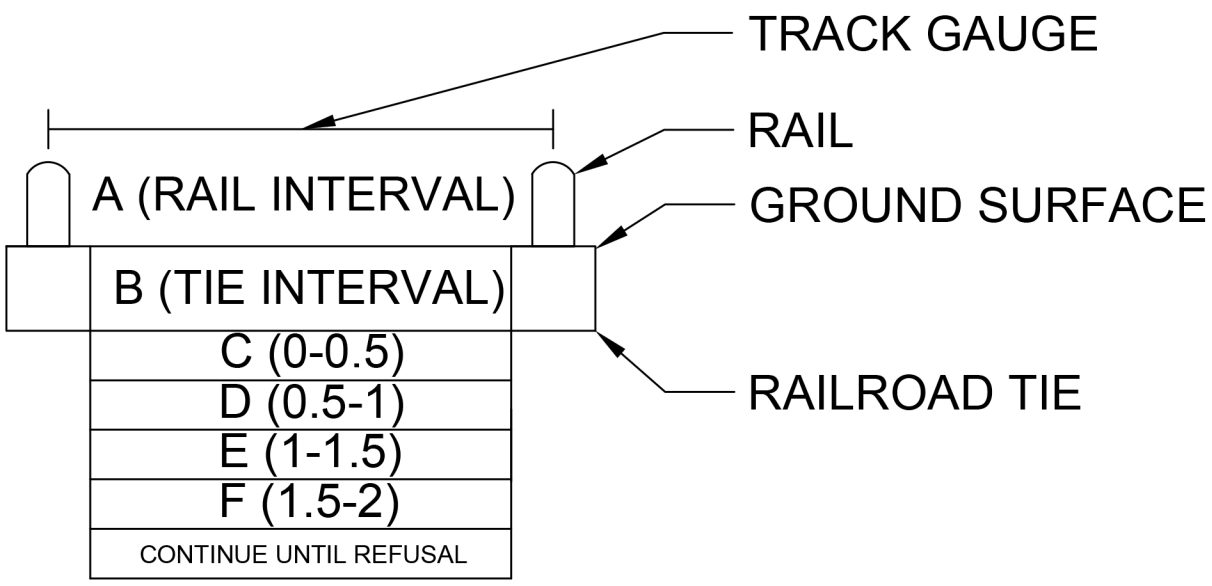
Appendices

Appendix A

Sampling Interval Diagram

Appendix A

Sampling Interval Diagram
Penn Station, New York



Appendix B

Air Sampling Plans and Reports Prepared by TRC

Appendix B Contents

1 Summary of PCB Air Sampling Events prepared by TRC
dated September 19, 2017

2 Report of PCB September 2017 Synoptic Air Sampling Event Performed at Penn Station prepared by TRC
dated October 16, 2017

3 PCB in Air Sampling Plan - Penn Station prepared by TRC
dated September 5, 2017

1430 Broadway
10th Floor
New York, NY 10018

212.221.7822 PHONE
212.221.7840 FAX

www.TRCsolutions.com

September 19, 2017

Mr. Craig Caldwell
National Railroad Passenger Corporation
30th Street Station
Philadelphia, PA 19104

Re: Summary of PCB Air Sampling Events
Performed at Penn Station
New York, New York
TRC Project # 258098

Dear Mr. Caldwell:

Following the discovery of polychlorinated biphenyls (PCBs) in accumulated sediment on the concrete body tracks adjacent to the Penn Station New York (Penn Station) platforms, Amtrak requested that TRC Environmental Corporation (TRC) perform air sampling in various areas and locations in Penn Station. This work is being performed as part of an ongoing evaluation of potential exposures to employees, passengers, and the general public. All air sampling was performed by TRC Industrial Hygienists under the supervision of Mr. John P. Springston, CIH, CSP, FAIHA, TRC's Industrial Hygiene Program Manager.

AIR MONITORING

Air sampling and analysis was conducted in accordance with USEPA Method TO-10A - *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*. The air samples were collected at a flow rate of approximately five (5) liters per minute, for approximately 300 to 400 minutes, and a total volume of around 1,800 to 2,000 liters for each sample. The sampling trains were calibrated at the beginning and end of the sampling period using a primary calibrator. Sampling was performed at the approximate breathing height of an adult. No quartz-fiber pre-filter was utilized during sampling, so the sample analysis included both the particulate and vapor phases for PCBs. Analysis of the samples was performed by Con-Test Analytical Laboratories, located in East Longmeadow, Massachusetts, an independent third-party industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033).

Flagmen/Watchmen Exposure Sampling

On May 6, 2016, TRC performed area air sampling for PCBs in the vicinity of the Amtrak Flagmen/Watchmen working within the station while Clean Harbors Environmental Services (Clean Harbors) was conducting PCB clean-up activities on Track 10. A total of two (2) samples were collected. The sampling pumps were stationed at the east and west end of Platform 5, adjacent to

where the flag men were located. Analytical results for the area air samples collected ranged from 33 to 66 nanograms per cubic meter of air (ng/m^3). Please refer to TRC Report of PCB in Air Sampling – Flagmen/Watchmen Exposure, dated June 7, 2016, for additional details.

A, D and E Yard Areas

On May 24, 2016, TRC performed area air sampling for PCBs in the vicinity of where the Amtrak track employees may be working within or around the A, D and E train yards at Penn Station. A total of three (3) samples were collected. The sampling pumps were stationed at the north side of tracks 1E, 2E, and 3E platform, near the Amtrak offices; the west end of the E Yard, between E ladder and track 6E; and the middle of A Yard, south of platform 1A. Analytical results for the area air samples collected ranged from less than 22 to 40 ng/m^3, with an average concentration of 30.0 ng/m^3. Please refer to TRC Report of PCB in Air Sampling – A, D and E Yard Areas, dated June 9, 2016, for additional details.

Track Walking Simulation Studies

On June 21, July 12, and July 19, 2016, TRC performed area and personal air sampling on Clean Harbors personnel as they walked back and forth on the uncleaned concrete track pad in Penn Station. A total six (6) air samples were collected: two (2) samples on June 21, 2016, two (2) samples on July 12, 2016 and two (2) samples on July 19, 2016. During each of the three (3) simulation studies, one (1) sampling pump was stationed in the center of Platform 1 to measure the background levels of PCBs within the station. In addition to the Platform 1 sample, one (1) sampling pump was placed on a Clean Harbors employee while he walked back and forth along the concrete pad on either Track 4 or Track 7 for approximately 30 minutes. At the conclusion of the walking exercise, the sampler was then placed in the center of Platform 1 and left running for the remainder of the work shift. Analytical results for the PCB personal air samples ranged from 90 to 138 ng/m^3, with an average of 118.7 ng/m^3. By comparison, the background concentrations measured on Platform 1 ranged from 68 to 91 ng/m^3, with an average concentration of 75.3 ng/m^3. Please refer to TRC Report of Track Walking Simulation Study, dated August 26, 2016, for additional details.

Ballast Digging Simulation Studies

On August 5, August 24, and October 5, 2016, TRC performed area and personal air sampling on Clean Harbors personnel while they dug up old ballast between the track gauges in Penn Station. A total six (6) air samples were collected: two (2) samples on August 5, 2016, two (2) samples on August 24, 2016, and two (2) samples on October 5, 2016. During each of the three (3) simulation studies, one (1) sampling pump was stationed approximately 50 feet away from the digging location to measure the background levels of PCBs within the station. In addition to the area sample, one (1) sampling pump was placed on a Clean Harbors employee while he dug up ballast for approximately 60 minutes. At the conclusion of the ballast digging exercise, the sampler was then placed adjacent to the area sample and left running for the remainder of the work shift. Analytical results for the PCB personal air samples were all less than 28 ng/m^3, while the background concentrations measured in A and E Yards were all less than 30 ng/m^3.

31st Street Elevated Substation

On January 12, 2017, TRC performed area air sampling within the 31st Street elevated substation, located above tracks 1E and 2E in E yard, to determine background concentrations during normal daytime operations. A total of two (2) area air samples were collected. Analytical results for the area air samples collected were all less than 27 ng/m^3. Please refer to TRC Report of PCB in Air Sampling – Elevated Substation, dated February 1, 2017, for additional details.

7th Avenue Elevated Substation

On February 3, 2017, TRC performed area air sampling within the 7th Avenue elevated substation, located above the east end of tracks 1, 2, 3 and 4, to determine background concentrations during normal daytime operations. A total of two (2) area air samples were collected. Analytical results for the area air samples collected were all less than 27 ng/m^3. Please refer to TRC Report of PCB in Air Sampling – 7th Avenue Elevated Substation, dated February 14, 2017, for additional details.

Track Level Sampling

On May 17, August 31, November 29, 2016, and February 23, 2017, TRC performed area air sampling at track level, on the passenger platforms, to determine background concentrations during normal daytime operations. During each event, sampling was conducted between approximately 9:30am and 4:30pm and the sampling pumps were stationed at the east and west ends of Platforms 4 and 9, and on the east end of Platform 1, adjacent to the JOPD shack. Analytical results for the area air samples collected ranged from less than 21 to 184 ng/m^3, with an overall average concentration of 63.05 ng/m^3. Please refer to TRC Reports of PCB in Air Sampling – Platform/Pedestrian Exposure, dated June 7, 2016, October 24, 2016, January 10, 2017, and March 9, 2017, for additional details.

Lower Level Sampling

On the evenings of October 18 and November 30, 2016, TRC performed area air sampling in the East, Central and Exit Concourses on the lower level of Penn Station, where the Long Island Rail Road (LIRR) operates. Sampling was conducted between approximately 10:30pm and 5:30am. During each event, a total of nine (9) air samples were collected in the following locations:

- East Concourse – by entrance to tracks 15/16 and tracks 20/21,
- Central Concourse – by entrance to tracks 15/16 and tracks 18/19, and
- Exit Concourse – by entrance to tracks 3/4, tracks 7/8, tracks 11/12, tracks 15/16, and tracks 18/19.

Analytical results for the lower level area air samples collected on October 18, 2016 ranged from less than 24 to 100 ng/m^3, while results for the samples collected on November 30, 2016 ranged from less than 21 to 100 ng/m^3. The overall average air concentration for the two events was 36.28 ng/m^3. Please refer to TRC Report of Lower Level PCB Air Sampling, dated January 10, 2017, for additional details.

Upper Level Sampling

On the evening of January 24-25, 2017, TRC performed area air sampling on the upper level of Penn Station, where Amtrak and New Jersey Transit (NJT) operate. Samples were collected between approximately 10:00pm and 4:00am. Samples were collected from the following locations:

- Amtrak waiting area – near the entrances to platforms 3, 5 and 8, and
- New Jersey Transit waiting area – near the entrances to platforms 1, 4 and 5.

Analytical results for the area air samples ranged from less than 22 to 66 ng/m^3, with an average concentration of 31.17 ng/m^3. All of the air samples collected in the Amtrak waiting area were less than 22 ng/m^3, while the samples collected in the New Jersey Transit waiting area ranged from 26 to 66 ng/m^3. Please refer to TRC Report of PCB Air Sampling – Upper Concourse Level, dated February 2, 2017, for additional details.

Station-Wide Sampling

On the evening of June 21-22, 2017, TRC performed concurrent area air sampling on the track, lower, and upper levels of Penn Station. Samples were collected between approximately 10:00pm and 5:00am. A total of 23 air samples were collected from the following locations:

Track level sampling:

- Track level sample location 01 - East side of Platform 2, near stairs
- Track level sample location 02 - West side of Platform 2, near stairs
- Track level sample location 03 - East side of Platform 4, near stairs
- Track level sample location 04 - West side of Platform 4, near stairs
- Track level sample location 05 - East side of Platform 7, near stairs
- Track level sample location 06 - West side of Platform 7, near stairs
- Track level sample location 07 - East side of Platform 9, near stairs
- Track level sample location 08 - West side of Platform 9, near stairs

Lower level air sampling:

Exit Concourse

- Lower level sample location 01 - By entrance to tracks 3/4
- Lower level sample location 02 - By entrance to tracks 7/8
- Lower level sample location 03 - By entrance to tracks 11/12
- Lower level sample location 04 - By entrance to tracks 15/16
- Lower level sample location 05 - By entrance to tracks 18/19

Central Concourse

- Lower level sample location 06 - By entrance to tracks 15/16
- Lower level sample location 07 - By entrance to tracks 18/19

Main Gate Area

- Lower level sample location 08 - By tracks 15/16
- Lower level sample location 09 - By tracks 20/21

Upper level air sampling:

Amtrak Concourse

- Upper level sample location 01 - By entrance to tracks 3/4
- Upper level sample location 02 - By entrance to tracks 7/8
- Upper level sample location 03 - By entrance to tracks 11/12

New Jersey Transit Concourse

- Upper level sample location 04 - By entrance to tracks 1/2
- Upper level sample location 05 - By entrance to tracks 5/6
- Upper level sample location 06 - By entrance to tracks 9/10

Analytical results for the track level samples ranged from less than 21 to 227 ng/m^3, with an average concentration of 104.0 ng/m^3. Results for the lower level samples ranged from less than 19 to 61 ng/m^3, with an average concentration of 34.67 ng/m^3. Results for the upper level samples ranged from less than 19 to 49 ng/m^3, with an average concentration of 29.0 ng/m^3. Please refer to TRC Report of PCB Synoptic Air Sampling Event, dated July 13, 2017, for additional details.

CONDITIONS AND LIMITATIONS

Air sampling results are limited in that they represent airborne levels at the time of sampling. Changes in operating procedures, ventilation, temperature, occupancy, equipment, contaminant sources, and other conditions may cause variations in air sampling results.

This report has been prepared for TRC's client. The results and opinions set forth by TRC are based on the information in our possession as of the date of this report. TRC appreciates the opportunity to provide you with these industrial hygiene consulting services. If you have any questions or comments, please contact us at (212) 221-7822. We look forward to working with you on future endeavors.

Sincerely,

TRC ENVIRONMENTAL CORP.

John P. Springston, CIH, CSP, FAIHA
Industrial Hygiene Program Manager

Kara Sweeney Parker
Project Manager

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10th Floor
New York, NY 10018

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212.221.7840 FAX

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October 16, 2017

Mr. Craig Caldwell
National Railroad Passenger Corporation
30th Street Station
Philadelphia, PA 19104

Re: Report of PCB September 2017 Synoptic Air Sampling Event
Performed at Penn Station
New York, New York
TRC Project # 258098

Dear Mr. Caldwell:

Following the discovery of polychlorinated biphenyls (PCBs) in accumulated sediment on the concrete body tracks adjacent to the Penn Station New York platforms, Amtrak requested that TRC Environmental Corporation (TRC) perform air sampling on the track level, lower concourse, and upper concourse levels of Penn Station. This work was performed as part of an ongoing evaluation of potential exposures to employees, passengers, and the general public. Area air sampling was performed on the evening of September 26-27, 2017. Air sampling was performed by TRC Industrial Hygienists under the supervision of Mr. John P. Springston, CIH, CSP, FAIHA, TRC's Industrial Hygiene Program Manager.

AIR MONITORING

Air sampling and analysis was conducted in accordance with USEPA Method TO-10A - *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*. A total of 26 air samples, including one (1) duplicate sample, were collected. The sampling pumps were stationed in the following locations:

Track level air sampling:

- Track level sample location 01 - East side of Platform 2, near stairs;
- Track level sample location 02 - West side of Platform 2, near stairs;
- Track level sample location 03 - East side of Platform 4, near stairs;
- Track level sample location 04 - West side of Platform 4, near stairs;
- Track level sample location 05 - East side of Platform 6, near stairs;
- Track level sample location 06 - West side of Platform 6, near stairs;
- Track level sample location 07 - East side of Platform 8, near stairs;
- Track level sample location 08 - West side of Platform 8, near stairs;

- Track level sample location 09 - West side of Platform 10, near stairs, and
- Track level sample location 10 - West side of Platform 10, near stairs.

Lower level air sampling:

Exit Concourse

- Lower level sample location 01 - By entrance to tracks 3/4;
- Lower level sample location 02 - By entrance to tracks 7/8;
- Lower level sample location 03 - By entrance to tracks 11/12;
- Lower level sample location 04 - By entrance to tracks 15/16, and
- Lower level sample location 05 - By entrance to tracks 18/19.

Central Concourse

- Lower level sample location 06 - By entrance to tracks 15/16, and
- Lower level sample location 07 - By entrance to tracks 18/19.

Main Gate Area

- Lower level sample location 08 - By tracks 11/12;
- Lower level sample location 09 - By tracks 15/16, and
- Lower level sample location 10 - By tracks 18/19.

Mezzanine and Upper level air sampling:

Amtrak Concourse

- Upper level sample location 01 - By entrance to tracks 7/8;
- Upper level sample location 02 - By entrance to tracks 11/12, and
- Upper level sample location 03 - By entrance to tracks 15/16.

New Jersey Transit Concourse

- Upper level sample location 04 - By entrance to tracks 3/4, and
- Upper level sample location 05 - By entrance to tracks 7/8.

The air samples were collected at a flow rate of approximately five (5) liters per minute for approximately 370 minutes and a total volume of around 1,900 liters for each sample. The sampling trains were calibrated at the beginning and end of the sampling period using a primary calibrator. Sampling was performed at the approximate breathing height of the passengers standing in the station. Samples were collected between approximately 11:00pm and 6:30am. No quartz-fiber pre-filter was utilized, so sample analysis included both particulate and vapor phases. Analysis of the samples was performed by Con-Test Analytical Laboratories, located in East Longmeadow, Massachusetts, an independent third-party industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033).

TEMPERATURE AND RELATIVE HUMIDITY MONITORING

Temperature and relative humidity readings were recorded at select locations throughout the sampling period using Dickson TP425 data loggers. Readings were collected every three (3) minutes. Following is a summary of the readings collected on each level. Please refer to Attachment 1 for the complete logged information and summary chart.

Table 1 – Temperature and Relative Humidity Readings Summary

Location	Temperature (ºF)			Relative Humidity (%)		
	Min	Max	Avg	Min	Max	Avg
Track Level	77.2	84.7	82.4	47.1	64.5	53.7
Lower Concourse	77.5	83.4	81.1	49.8	62.7	54.4
Upper Concourse	75.6	79.2	78.5	45.2	63.7	50.2

PRESSURE MEASUREMENTS

Pressure readings at each sampling location were measured and recorded throughout the sampling period using a Testo 511 absolute pressure meter. Measurements were taken at the sampling start time, mid-shift, and at the sampling end time for a total of three (3) readings at each location. Pressure readings were found to be similar between the track level, lower level, and upper level. Please refer to Attachment 2 for the complete data table.

TRAIN TRAFFIC OBSERVATIONS

Train activity at each platform was monitored by TRC Industrial Hygienists during the sampling event. A copy of the observation sheet is located in Attachment 3.

RESULTS

Analytical results for the area air samples collected on September 26-27, 2017 ranged from less than 20 to 107 nanograms per cubic meter of air (ng/m^3). The results for the air samples collected during this round, as well as prior rounds of samplings, for each level are summarized in Tables 1 through 3 below. Figures 1 and 2, on the following pages, are representative photographs of the area air sampling that was conducted. Please refer to Attachment 4 for the sample location diagram.

Table 2 – Track Level PCB Area Air Sample Results

Sample location	PCB Concentration (ng/m^3)					
	5/17/16	8/31/16	11/29/16	2/23/17	6/21/17	9/26/17
Platform 1 – west end	92	184	122	98	-	-
Platform 2 – west side	-	-	-	-	122	93
Platform 2 – east side	-	-	-	-	227	107
Platform 4 – west side	29	81	25	<19	106	<22

Sample location	PCB Concentration (ng/m^3)					
	5/17/16	8/31/16	11/29/16	2/23/17	6/21/17	9/26/17
Platform 4 – east side	114	151	45	67	102	58
Platform 6 – west side	-	-	-	-	-	<22
Platform 6 – east side	-	-	-	-	-	31
Platform 7 – west side	-	-	-	-	80	-
Platform 7 – east side	-	-	-	-	152	-
Platform 8 – west side	-	-	-	-	-	<22
Platform 8 – east side	-	-	-	-	-	<22
Platform 9 – west side	<24	28	<21	<19	22	-
Platform 9 – east side	61	41	<21	<19	<21	-
Platform 10 –west side	-	-	-	-	-	<22
Platform 10 – east side	-	-	-	-	-	<22

Table 3 – Lower Concourse Level PCB Area Air Sample Results

Sample location	PCB Concentration (ng/m^3)			
	10/18/16	11/30/16	6/21/17	9/26/17
Main Gate Area – entrance to tracks 20/21	<24	<21	45	-
Main Gate Area – entrance to tracks 18/19	-	-	-	<21
Main Gate Area – entrance to tracks 15/16	36	26	61	30
Main Gate Area – entrance to tracks 11/12	-	-	-	25
Central Concourse – entrance to tracks 18/19	<25	<21	20	<20
Central Concourse – entrance to tracks 15/16	30	<21	23	<21
Exit Concourse – entrance to tracks 18/19	<25	<21	<19	<20
Exit Concourse – entrance to tracks 15/16	<24	<21	<19	<22
Exit Concourse – entrance to tracks 11/12	37	21	29	22
Exit Concourse – entrance to tracks 7/8	100	91	50	30
Exit Concourse – entrance to tracks 3/4	64	45	46	46

Table 4 – Mezzanine and Upper Concourse Level PCB Area Air Sample Results

Sample location	PCB Concentration (ng/m^3)		
	1/24/2017	6/21/2017	9/26/2017
Amtrak waiting area – entrance to tracks 3/4	<22	49	-
Amtrak waiting area – entrance to tracks 7/8	<22	22	21
Amtrak waiting area – entrance to tracks 11/12	<22	<19	<21
Amtrak waiting area – entrance to tracks 15/16	-	-	<21
NJT waiting area – entrance to tracks 1/2	29	44	-
NJT waiting area – entrance to tracks 3/4	-	-	24
NJT waiting area – entrance to tracks 5/6	26	22	-
NJT waiting area – entrance to tracks 7/8	-	-	24
NJT waiting area – entrance to tracks 9/10	66	18	-

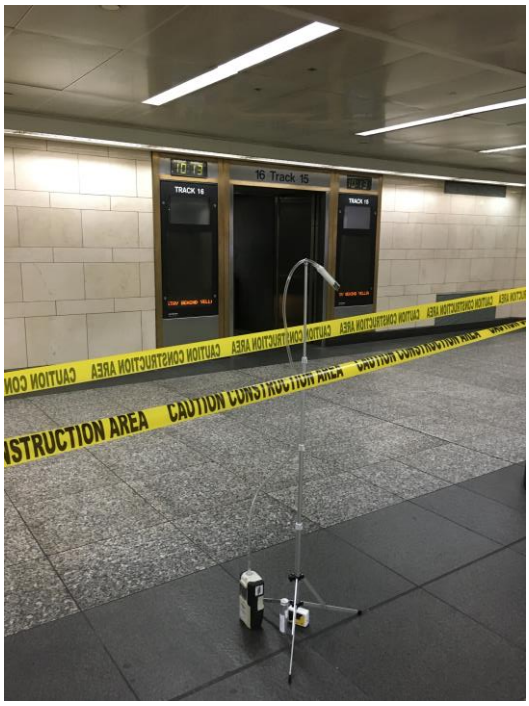

Figure 1 – Lower Central Concourse, by tracks 15/16

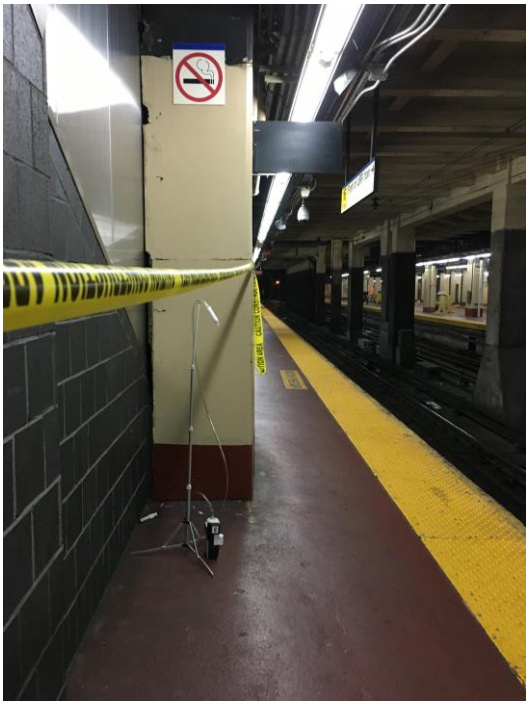

Figure 2 – Track level, west side of Platform 8, near stairs

DISCUSSION

In general, airborne PCB concentrations at track level were similar to, and slightly lower than, those measured in the previous five (5) rounds of sampling, conducted between May 2016 and June 2017. The overall average PCB concentrations at track level during this round was 42.1 ng/m^3. In comparison, average PCB concentrations for the previous rounds ranged between 44.4 and 104 ng/m^3.

Airborne PCB concentrations on the lower concourse level were similar to, and slightly lower than, those measured in October 2016, November 2016, and June 2017. The average concentration for this round was 25.7 ng/m^3. In comparison, the average PCB concentrations measured in the prior three (3) rounds ranged between 30 and 40.6 ng/m^3.

Airborne PCB concentrations on the upper level were also similar to, and slightly lower than, those previously measured in January and June of 2017. The average concentration for this round was 22.2 ng/m^3. In comparison, the average PCB concentrations measured in the prior two (2) rounds were 31.2 and 29.0 ng/m^3.

Results were below the current USEPA guideline values for evaluating PCBs in school indoor air, which were issued in July 2015.[1] According to the EPA, the calculated indoor air levels were revised to reflect more recent data on dietary exposure. The EPA has established updated exposure levels for evaluating school indoor air for age groups ranging from age 1 to age 19+ and have provided applicable exposure levels rounded to the nearest hundred ng/m^3. The exposure levels for evaluating school indoor air are as follows:

Age (in years)			
6 - <12 Elementary School	**12 - <15 Middle School**	**15 - <19 High School**	**19+ Adult**
300 ng/m^3	500 ng/m^3	600 ng/m^3	500 ng/m^3

As per the EPA, these updated exposure levels were derived to serve as health protective values intended for evaluation purposes. According to the EPA, these levels "…should not be interpreted nor applied as "bright line" or "not-to-exceed" criteria, but may be used to guide thoughtful evaluation of indoor air quality in schools." The EPA has also revised the terminology describing these criteria from 'Recommended Public Health Levels for PCBs in Indoor School Air' to 'Exposure Levels for Evaluation of PCBs in Indoor School Air' because "…the Agency believes the revised terminology better reflects the intended purpose of these levels."

Results were also well below the New York State Department of Health (NYSDOH) PCBs air guideline value of 1,000 ng/m^3 for residential settings where people may be involuntarily exposed to

[1] USEPA. 2015. Exposure Levels for Evaluation of Polychlorinated Biphenyls (PCBs) in Indoor School Air. Available online at https://www.epa.gov/pcbs/exposure-levels-evaluation-polychlorinated-biphenyls-pcbs-indoor-school-air

chemicals from soil vapor intrusion.[2] While the USEPA and NYSDOH guideline values are not directly applicable to the workplace or the general public, they are provided within this report for comparison purposes. These levels are more protective of the general population, especially children, than are occupational limits.

CONDITIONS AND LIMITATIONS

Air sampling results are limited in that they represent airborne levels at the time of sampling. Changes in operating procedures, ventilation, temperature, occupancy, equipment, contaminant sources, and other conditions may cause variations in air sampling results.

This report has been prepared for TRC's client. The results and opinions set forth by TRC are based on the information in our possession as of the date of this report. TRC appreciates the opportunity to provide you with these industrial hygiene consulting services. If you have any questions or comments, please contact us at (212) 221-7822. We look forward to working with you on future endeavors.

Sincerely,

TRC ENVIRONMENTAL CORP.

John P. Springston, CIH, CSP, FAIHA
Industrial Hygiene Program Manager

Kara Sweeney Parker
Project Manager

Attachments

[2] NYSDOH, 2006. Guidance for Evaluating Soil Vapor Intrusion in the State of New York. Troy, NY: Bureau of Environmental Exposure Investigation.

ATTACHMENT 1

TEMPERATURE AND RELATIVE HUMIDITY READINGS

TRC

Amtrak IH Air Sampling

Temperature and Relative Humidity Summary

Date: 09/26/2017

Location	Tracks	Temperature (°F)			Relative Humidity (%)			Dickson ID#
		Min	Max	AVG	Min	Max	AVG	
Upper Concourse Level								
Upper Level 02	11/12	75.6	79.2	78.5	47.9	63.9	50.7	TRC-10
Upper Level 05	7/8 (NJT)	77.1	80.0	79.0	42.4	63.4	49.7	TRC-23
AVERAGE		**75.6**	**79.2**	**78.5**	**45.2**	**63.7**	**50.2**	
Lower Concourse Level								
Lower Level 02	7/8	77.6	81.6	79.6	53.9	62.5	58.2	TRC-21
Lower Level 04	15/16	78.1	85.5	81.8	47.1	65.3	49.5	TRC-12
Lower Level 06	15/16	76.7	80.7	78.2	54.5	59.6	57.3	TRC-16
Lower Level 09	15/16	80.0	85.8	84.7	43.5	63.3	52.7	TRC-20
AVERAGE		**77.5**	**83.4**	**81.1**	**49.8**	**62.7**	**54.4**	
Track Level								
Track Level 01	3/4	76.8	83.4	82.2	46.6	59.5	49.9	TRC-07
Track Level 04	7/8	76.9	81.3	80.6	53.0	59.7	56.5	TRC-15
Track Level 05	11/12	71.8	86.5	80.4	45.2	75.1	55.5	TRC-08
Track Level 08	15/16	77.3	87.7	84.9	47.9	69.7	54.4	TRC-22
Track Level 09	18/19	83.3	84.5	83.9	48.8	58.6	52.2	TRC-04
AVERAGE		**77.2**	**84.7**	**82.4**	**47.1**	**64.5**	**53.7**	

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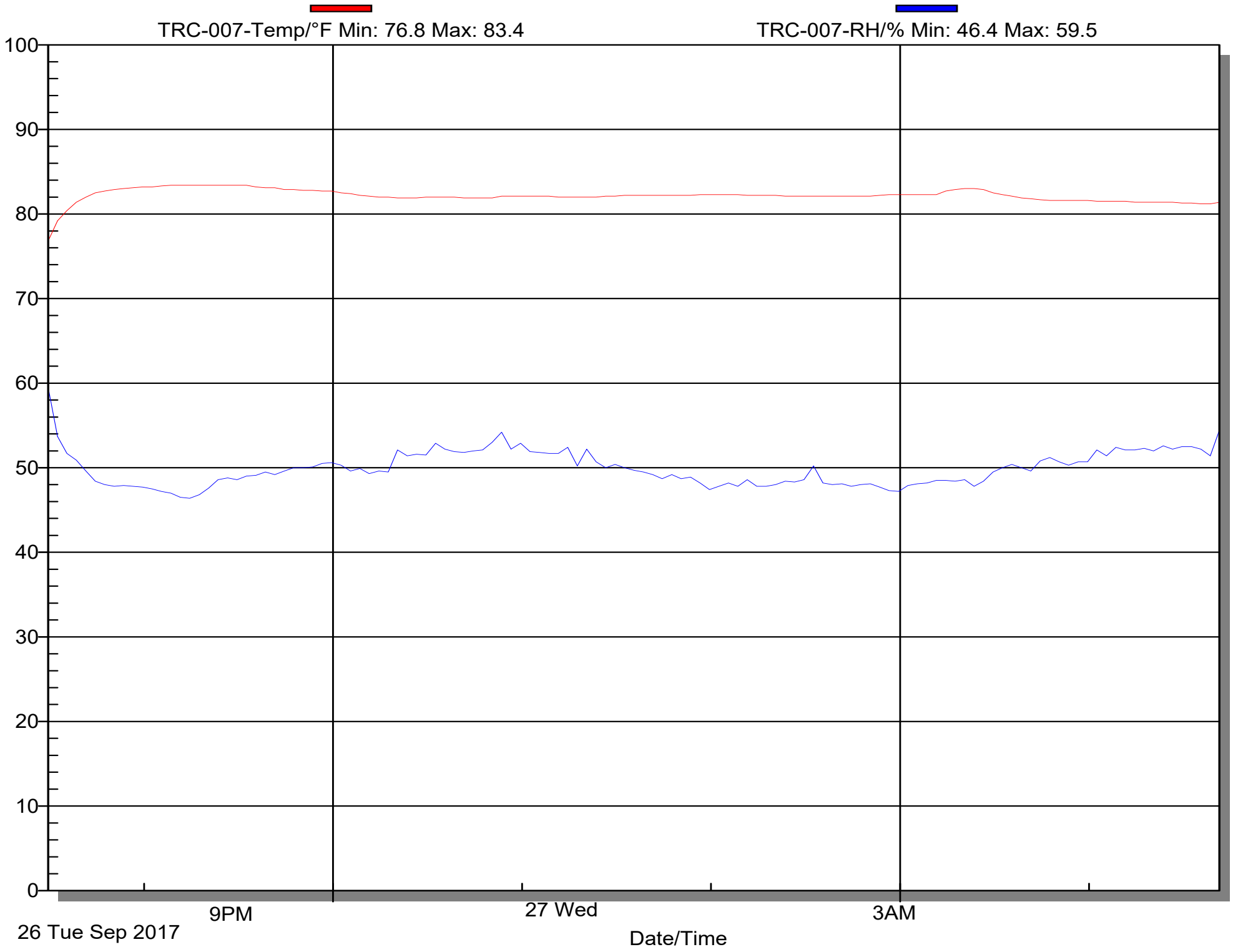
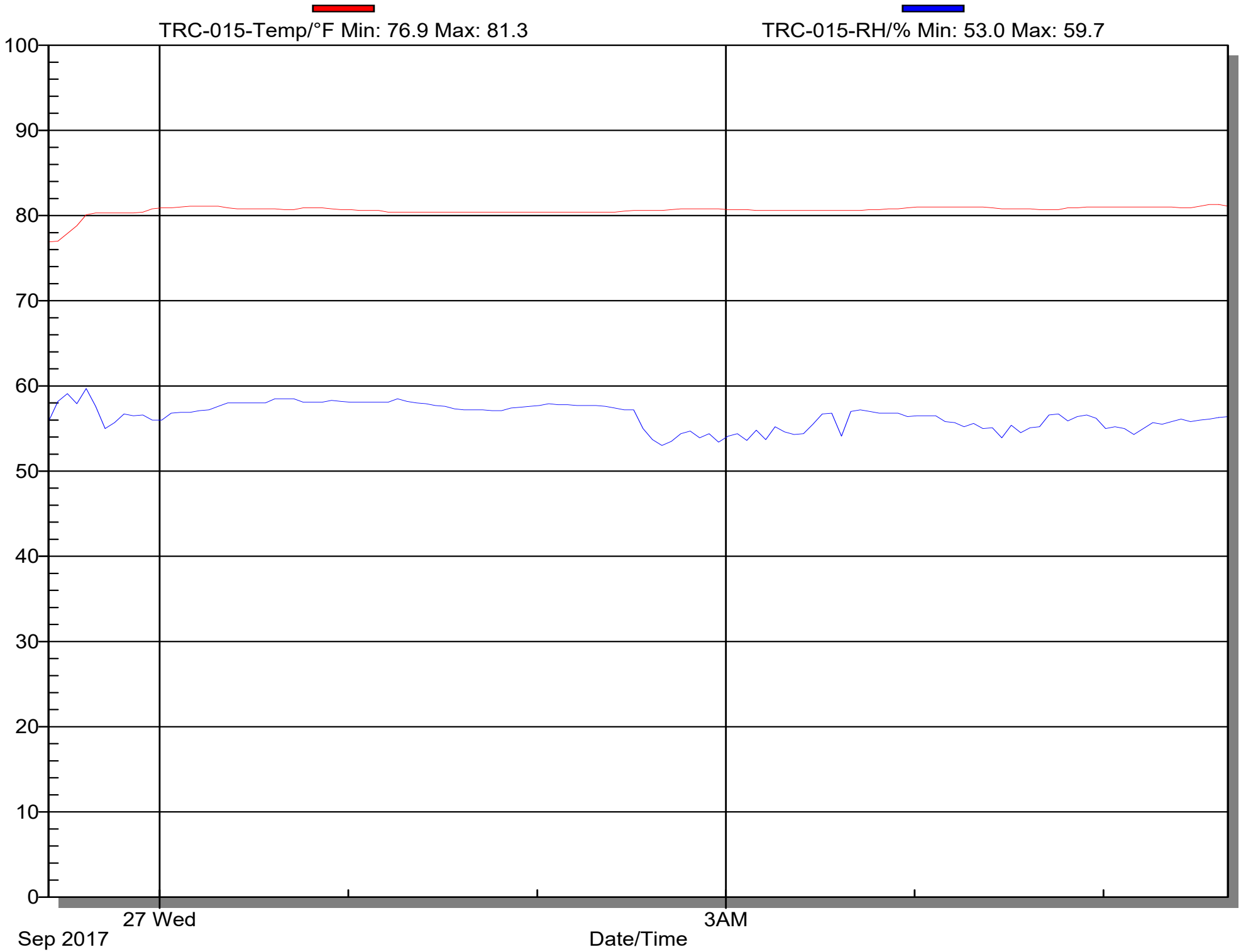
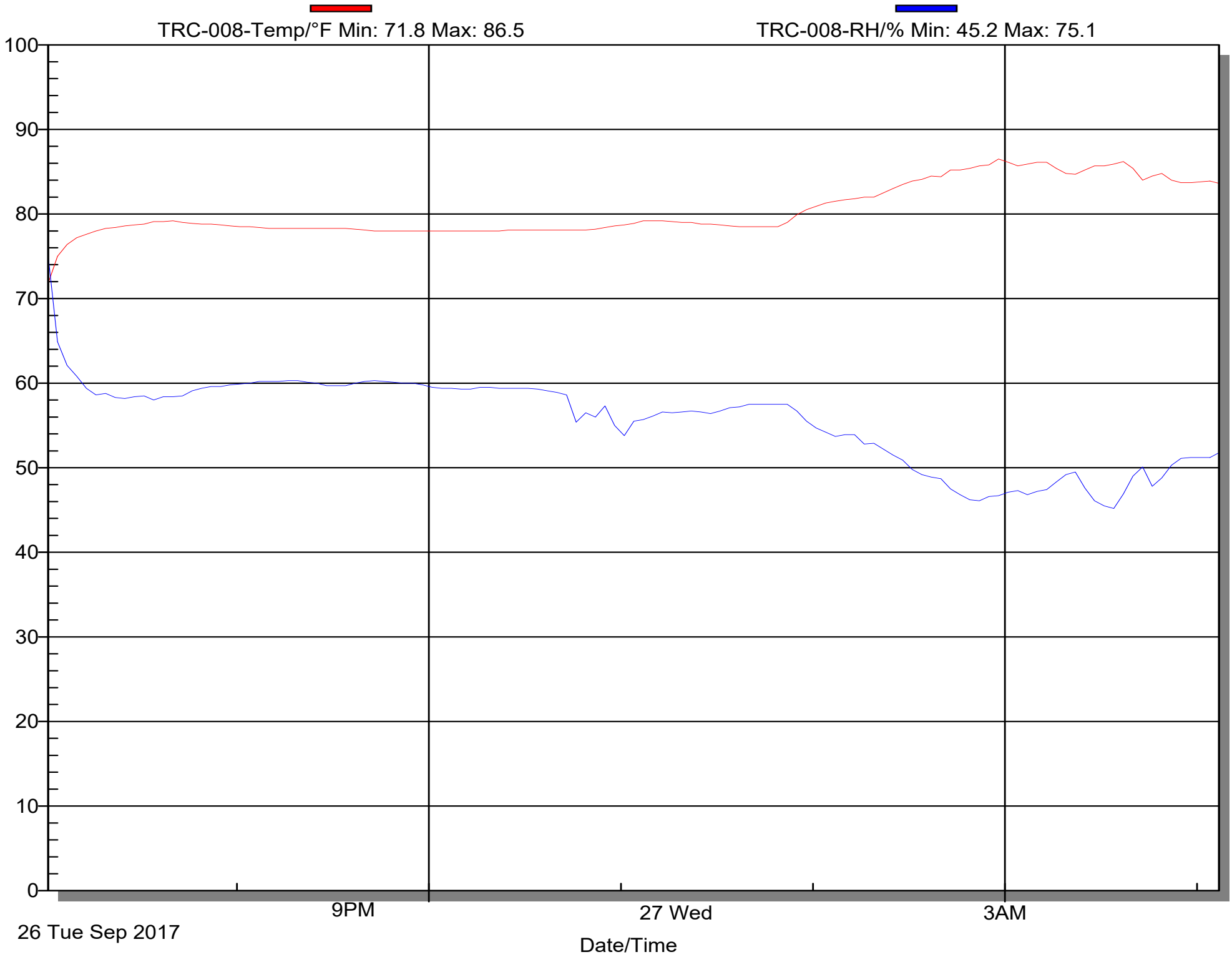
TRC-07 Track Level 01



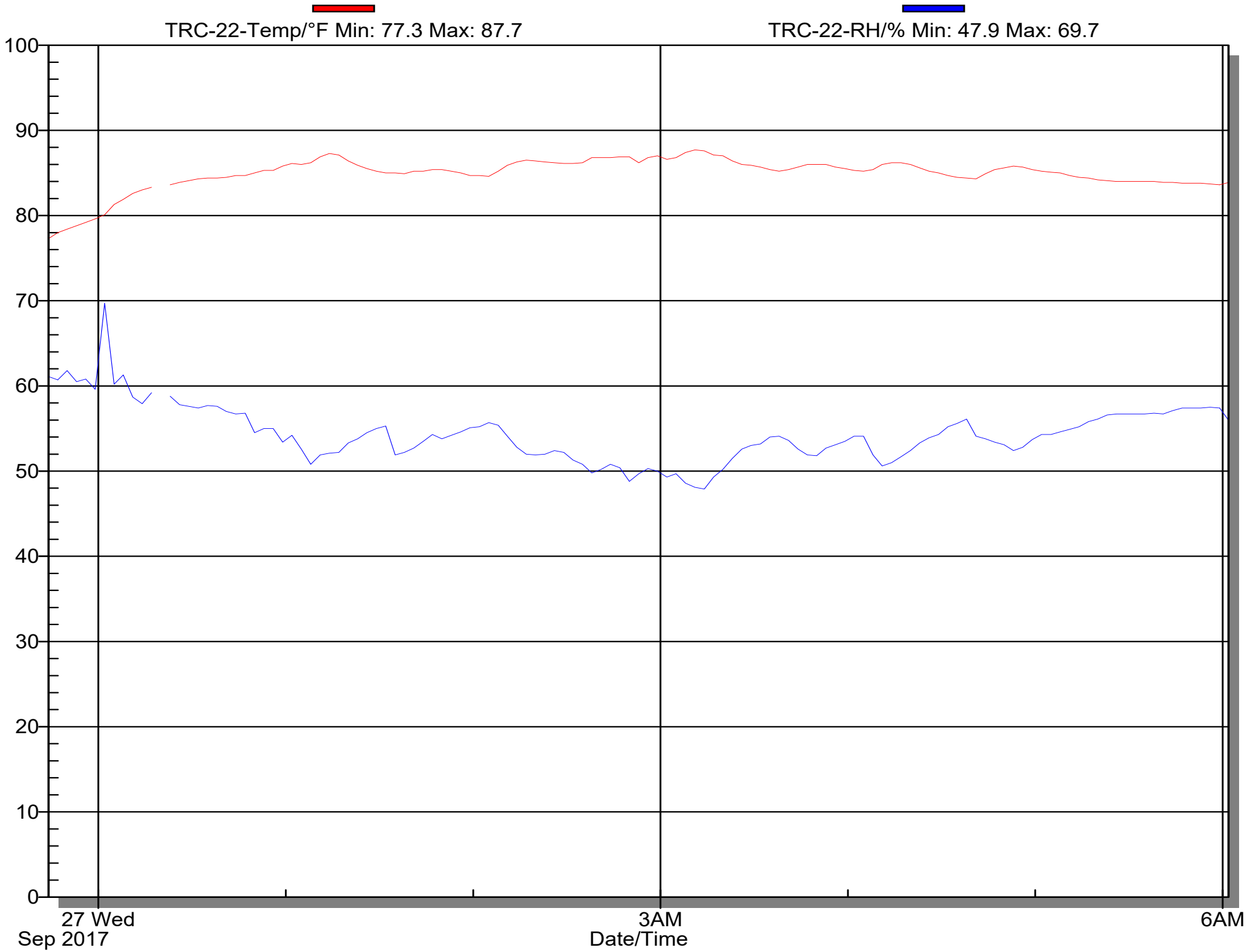
TRC-15 Track Level 04



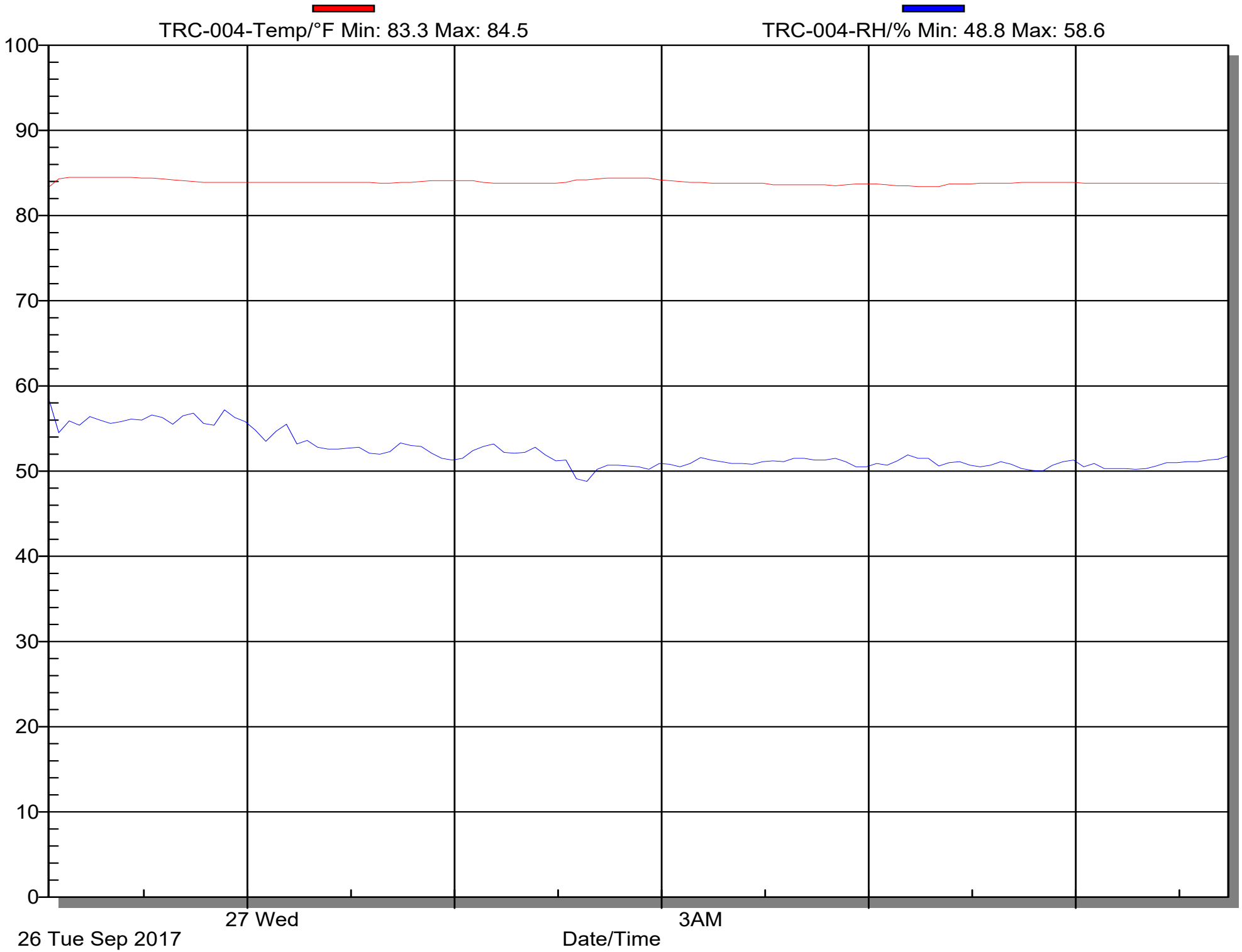
TRC-08 Track Level 05



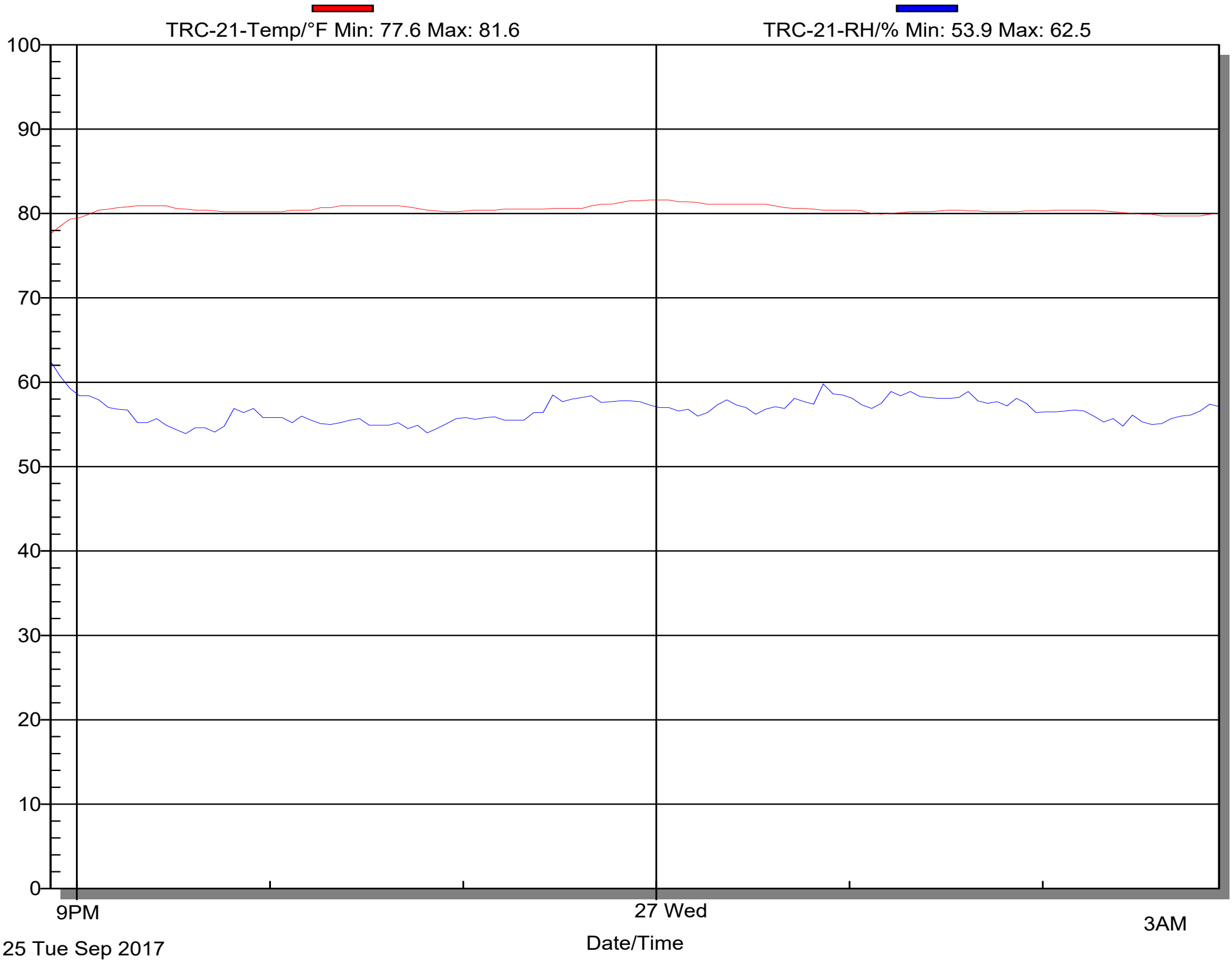
TRC-22 Track Level 08



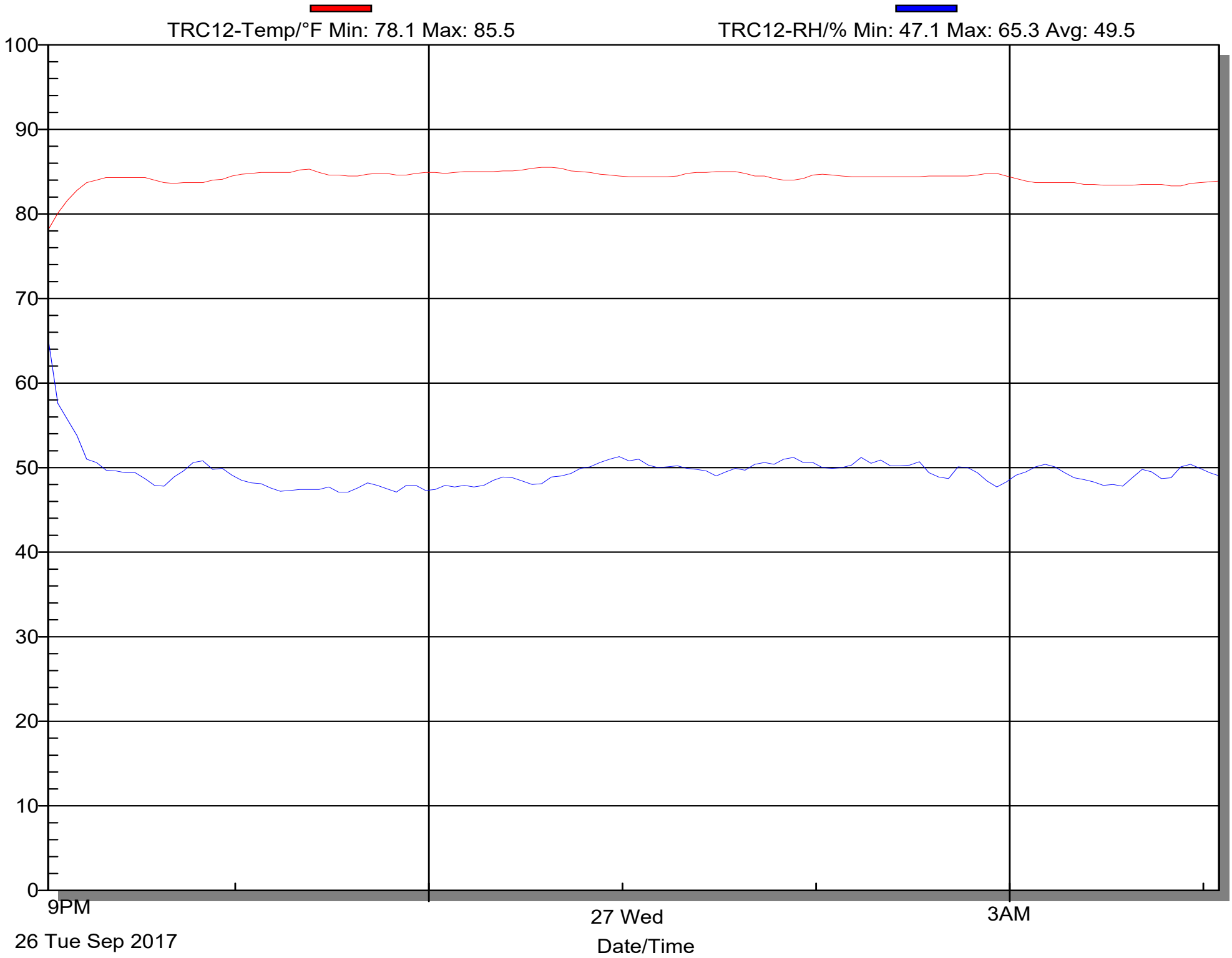
TRC-04 Track Level 09



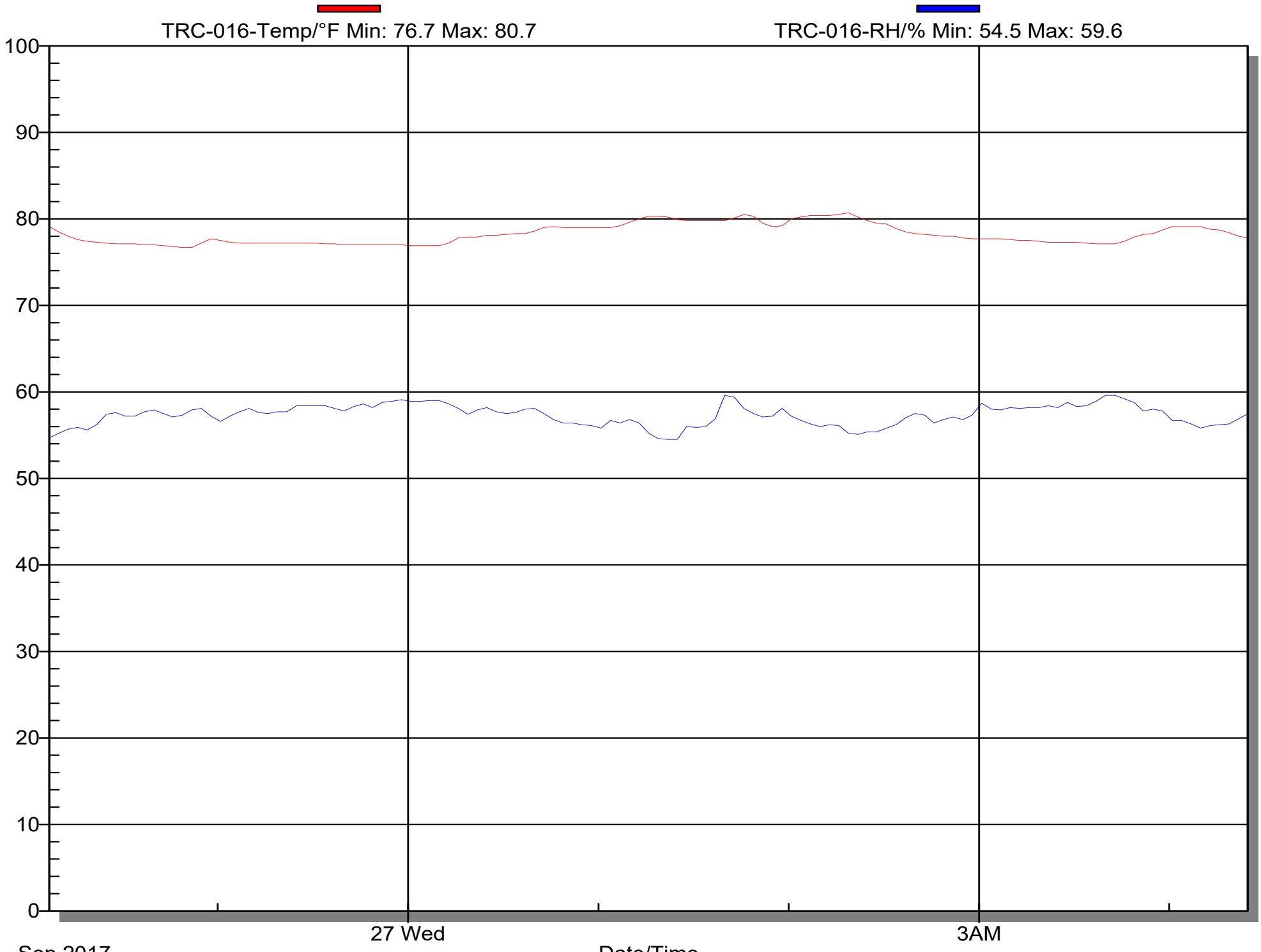
TRC 21 - Lower Level 02



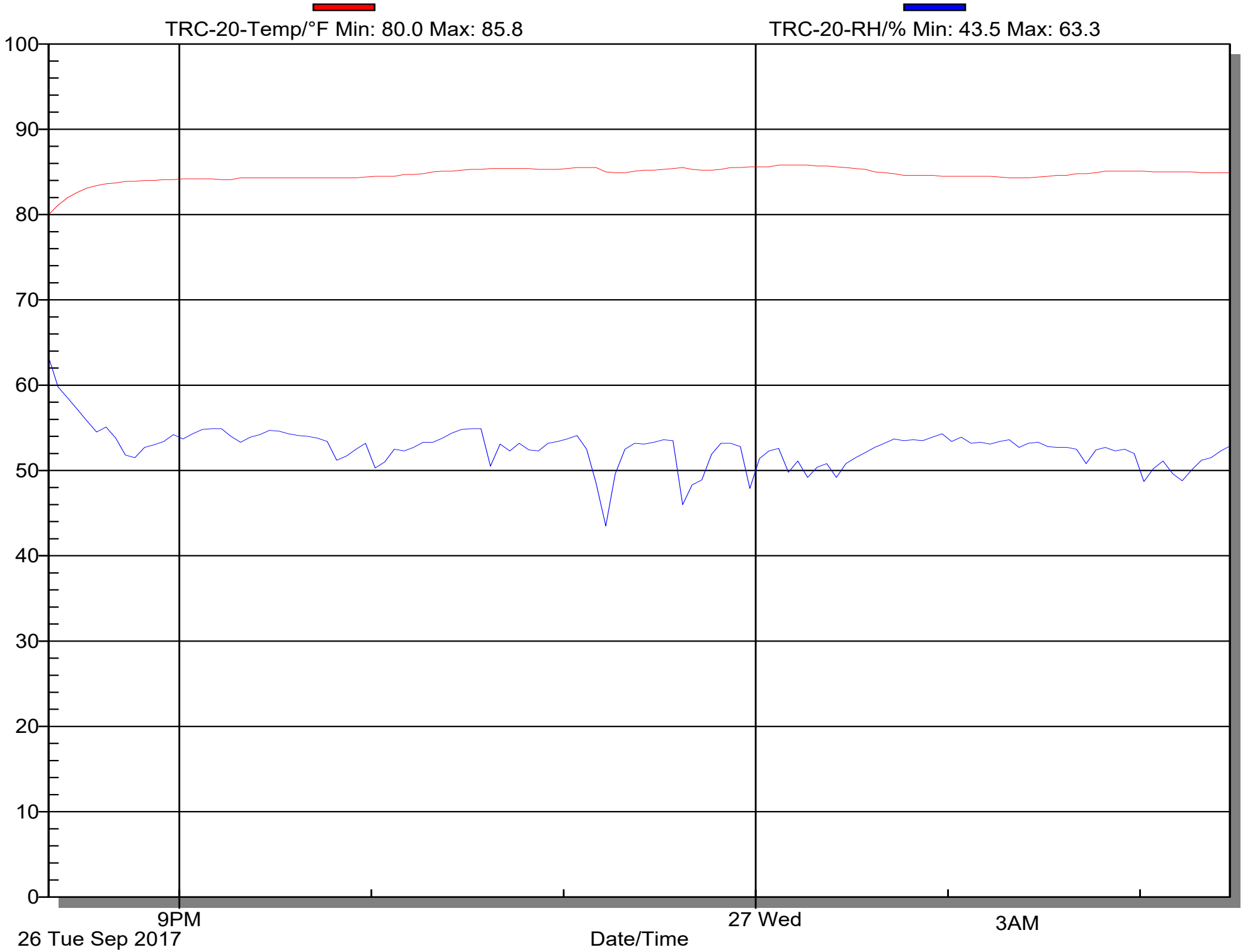
TRC-012 Lower Level 04



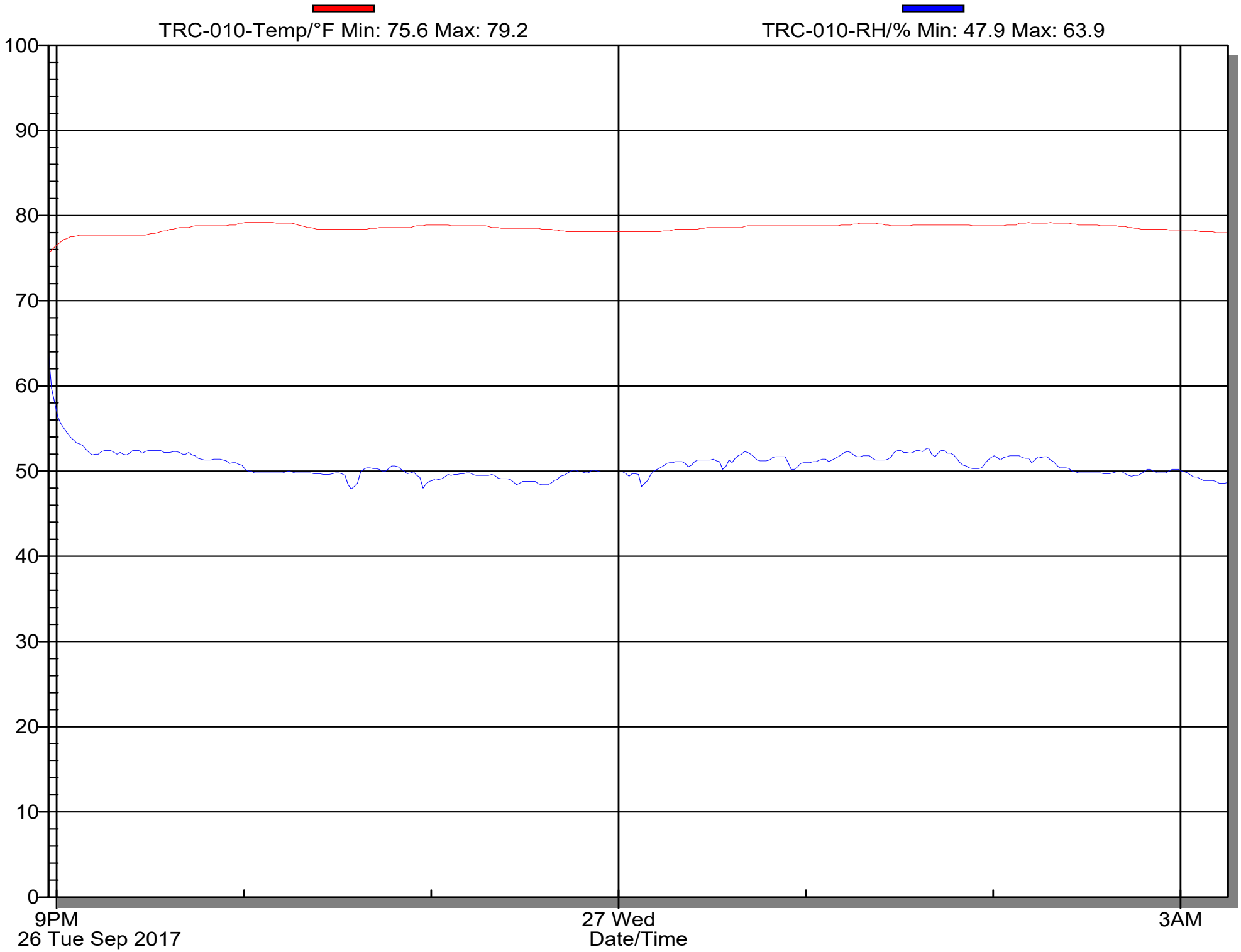
TRC-16 Lower Level 06



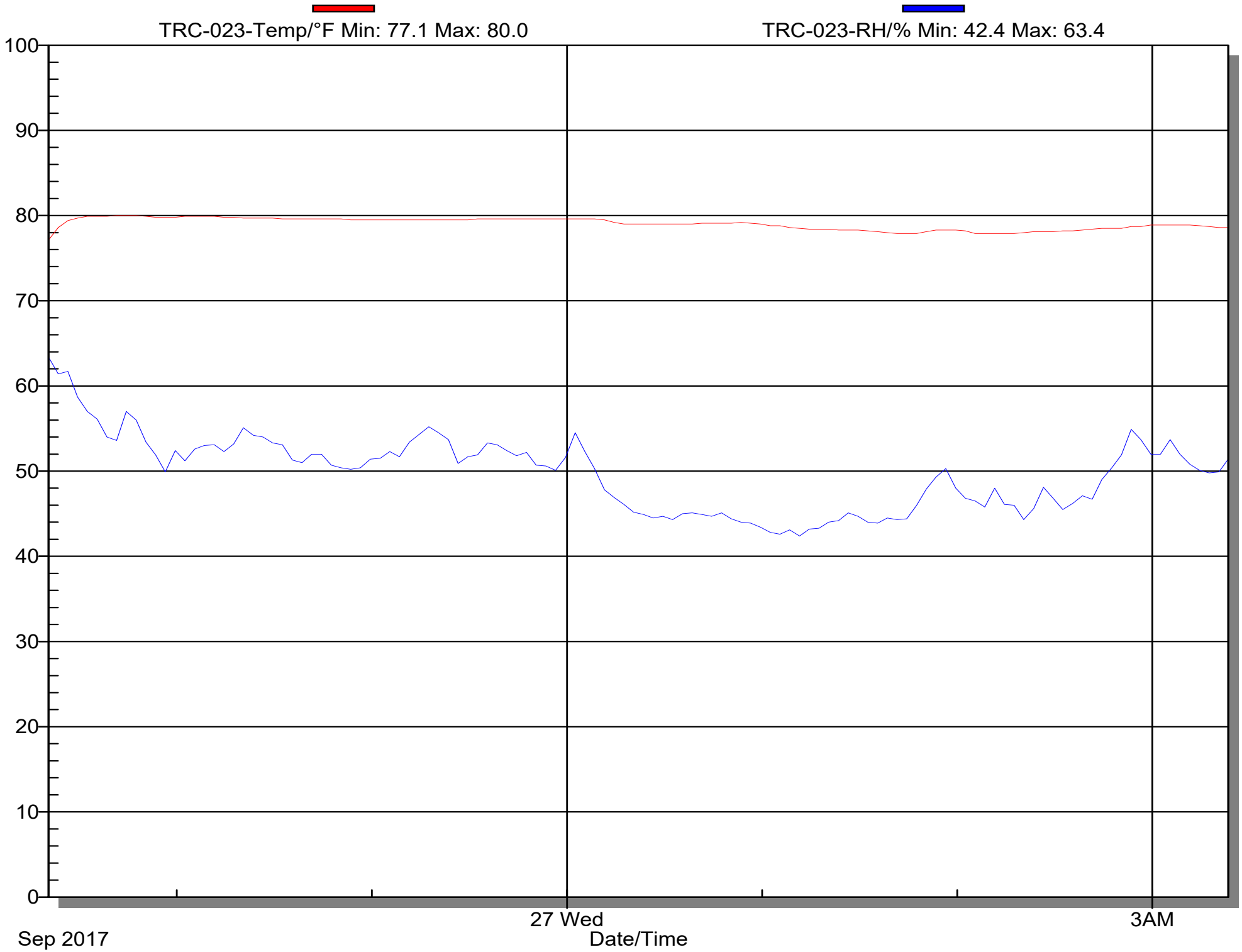
TRC-20 Lower Level 09



TRC-10 Upper Level 02



TRC-23 Upper Level 05



ATTACHMENT 2

PRESSURE MEASUREMENTS

TRC

Amtrak IH Air Sampling

Pressure Log

Instrument Used: Absolute Pressure Meter 'Testo 511' SN 39113448/607

Date: 09/26/2017

Location		Tracks	Pressure Reading		
			START	MIDDLE	END
Upper Concourse Level					
	Upper Level 01	7/8	1015.1	1013.8	1013.5
	Upper Level 02	11/12	1015	1013.7	1013.5
	Upper Level 03	15/16	1014.9	1013.8	1013.4
	Upper Level 04	3/4 (NJT)	1015.1	1013.6	1013.5
	Upper Level 05	7/8 (NJT)	1015.2	1013.8	1013.6
	AVERAGE		**1015.1**	**1013.7**	**1013.5**
Lower Concourse Level					
	Lower Level 01	3/4	1015.4	1013.8	1013.5
	Lower Level 02	7/8	1015.4	1013.8	1013.5
	Lower Level 03	11/12	1015.3	1013.8	1013.5
	Lower Level 04	15/16	1015.4	1013.8	1013.4
	Lower Level 05	18/19	1015.3	1013.8	1013.1
	Lower Level 06	15/16	1015.5	1013.8	1013.2
	Lower Level 07	18/19	1015.6	1013.8	1013.2
	Lower Level 08	11/12	1015.6	1013.9	1013.2
	Lower Level 09	15/16	1015.5	1013.9	1013.3
	Lower Level 10	18/19	1015.4	1013.9	1013.2
	AVERAGE		**1015.4**	**1013.8**	**1013.3**
Track Level					
	Track Level 01	3/4	1015.6	1014.3	1013.9
	Track Level 02	3/4	1015.5	1014.4	1013.8
	Track Level 03	7/8	1015.4	1014.4	1013.7
	Track Level 04	7/8	1015.4	1014.3	1013.7
	Track Level 05	11/12	1015.2	1014.3	1013.5
	Track Level 06	11/12	1015.1	1014.4	1013.5
	Track Level 07	15/16	1014.9	1014.4	1013.4
	Track Level 08	15/16	1014.8	1014.4	1013.5
	Track Level 09	18/19	1014.8	1014.3	1013.6
	Track Level 10	18/19	1014.9	1014.2	1013.6
	AVERAGE		**1015.2**	**1014.3**	**1013.6**

IH Signature: Sara M Cahill _____

ATTACHMENT 3

TRAIN ACTIVITY OBSERVATIONS

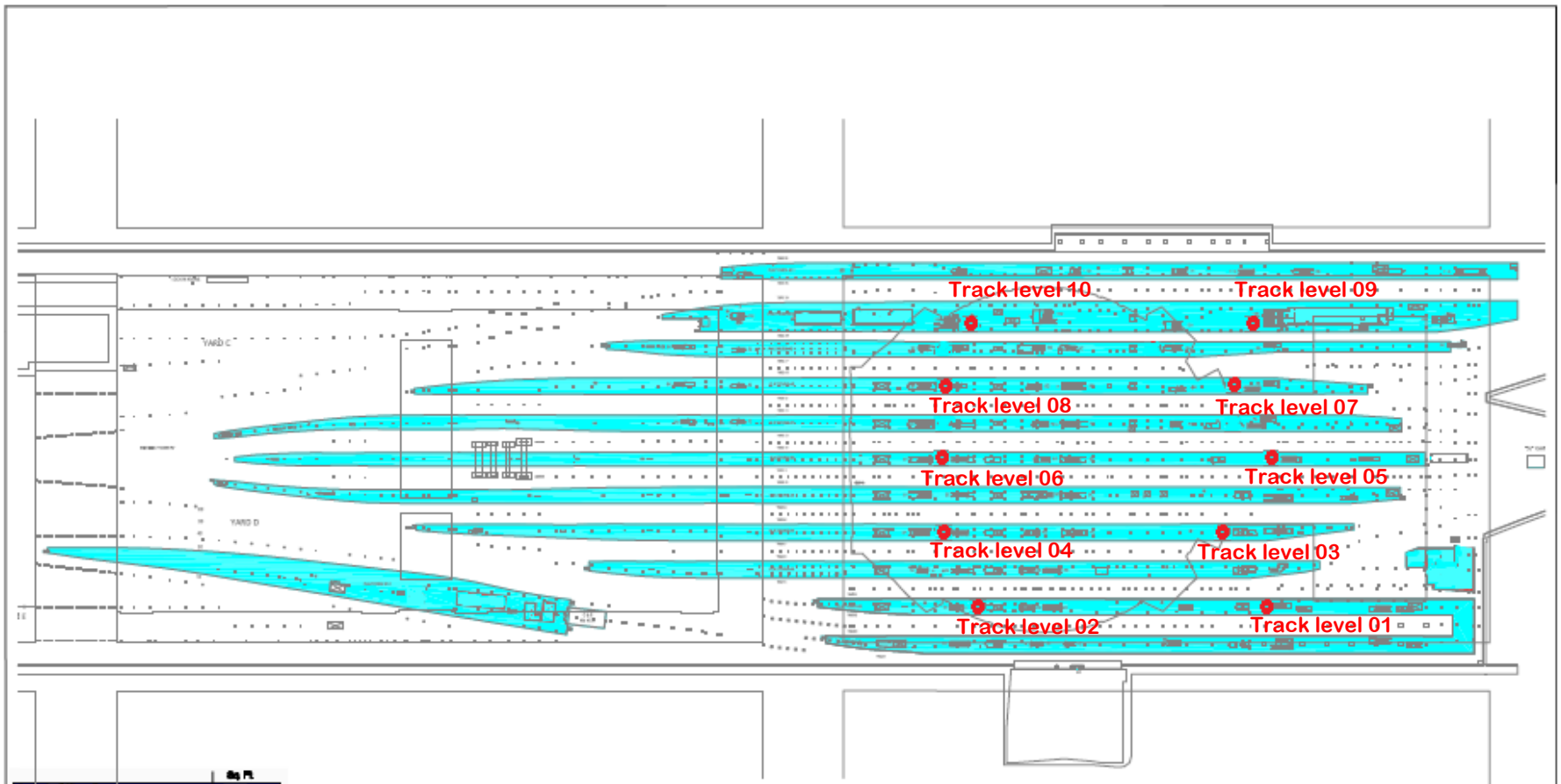
TRC
AMTRAK IH AIR SAMPLING
TRAIN TRAFFIC LOG

Date: 09/26/2017 – 09/27/2017

	Train Traffic		
Track #	**Arrival Time**	**Departure Time**	**Direction (E/W)**
9	Unknown	2213	Westbound
10	Unknown	2213	Westbound
9	2235	2305	Westbound
10	2258	Remained Stationed	Westbound
1	2215	2240	Westbound
2	2250	Remained Stationed	Eastbound
1	2300	Remained Stationed	Westbound
18	2229	2237	Eastbound
17	2253	2325	Eastbound
18	2240	2251	Eastbound
18	2256	2310	Eastbound
18	2314	2318	Eastbound
12	Unknown	2340	Eastbound
20	0000	0002	Westbound
3	2315	0007	Westbound
16	2329	2342	Eastbound
15	2333	2349	Eastbound
16	2346	2352	Westbound
15	2351	0025	Eastbound
16	0008	0016	Eastbound
19	0025	0030	Westbound
5	0219	0241	Eastbound
7	Unknown	0325	Westbound
7	0355	Remained Stationed	Westbound

ATTACHMENT 4

SAMPLE LOCATION DIAGRAMS



Amtrak®

Office of Chief Engineer
STRUCTURES
National Railroad Passenger Corporation
30th Street Station, Philadelphia, Pennsylvania 19104

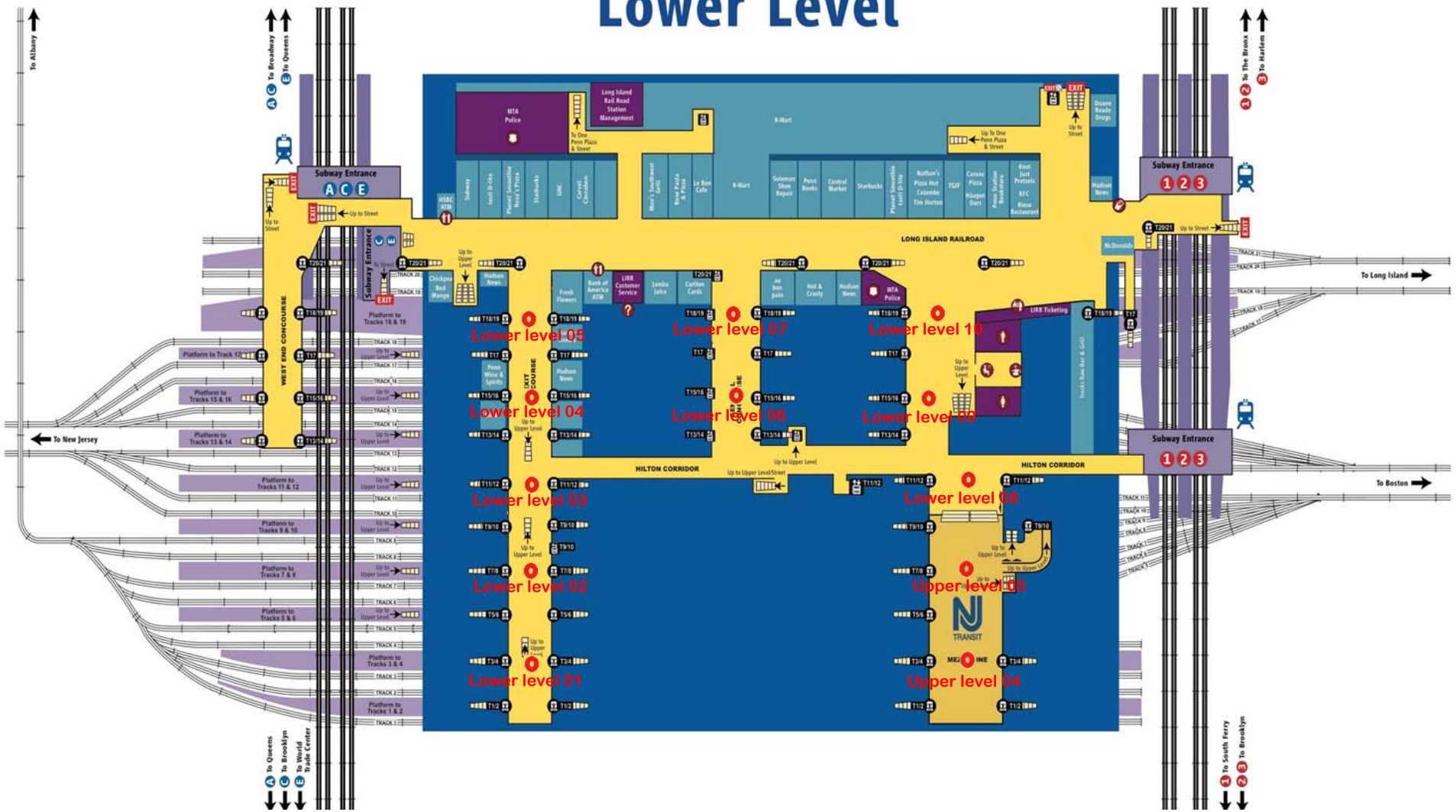
PROJECT NORTH

KEY PLAN

NEW YORK CITY, NY
PENN STATION NEW YORK
SQUARE FOOT ANALYSIS
PLATFORM LEVEL

A-4

Lower Level


To Albany
To Broadway
To Queens
To The Bronx
To Harlem
Subway Entrance
A C E
Subway Entrance
1 2 3
LONG ISLAND RAILROAD
To Long Island
To New Jersey
To Boston
WEST END CONCOURSE
HILTON CORRIDOR
HILTON CORRIDOR
Lower level 05
Lower level 07
Lower level 10
Lower level 04
Lower level 06
Lower level 09
Lower level 03
Lower level 08
Lower level 02
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Lower level 01
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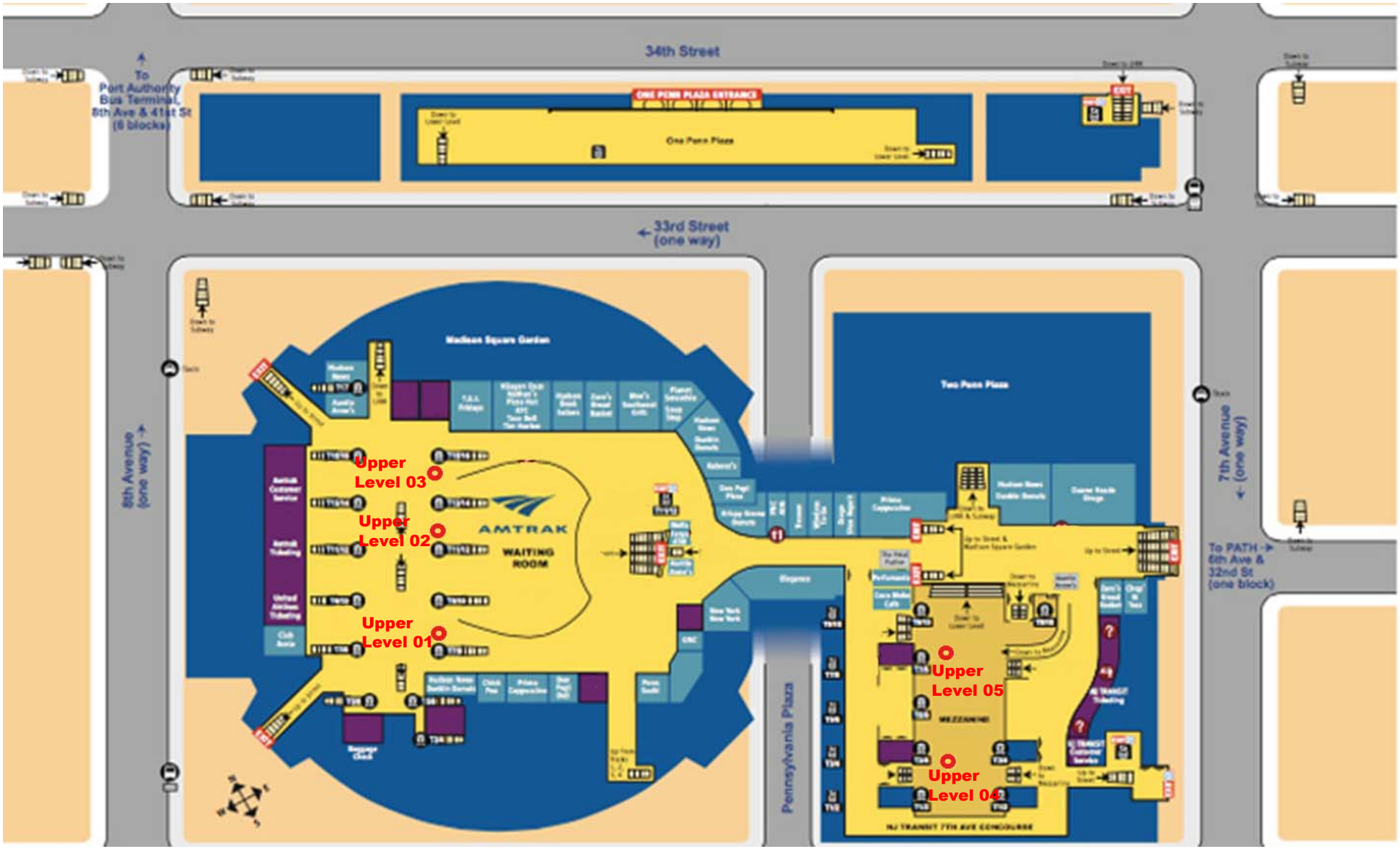
To Queens
To Brooklyn
To World Trade Center
To South Ferry
To Brooklyn


34th Street
To Port Authority Bus Terminal 8th Ave & 41st St (8 blocks)
ONE PENN PLAZA ENTRANCE
One Penn Plaza
EXIT
33rd Street (one way)
8th Avenue (one way)
7th Avenue (one way)
To PATH 6th Ave & 32nd St (one block)
Madison Square Garden
Two Penn Plaza
Upper Level 03
Upper Level 02
Upper Level 01
AMTRAK
WAITING ROOM
Pennsylvania Plaza
Upper Level 05
Upper Level 04
NJ TRANSIT 7TH AVE CONCOURSE

1430 Broadway
10th Floor
New York, NY 10018

212.221.7822 PHONE
212.221.7840 FAX

www.TRCsolutions.com

September 5, 2017

Mr. Richard Mohlenhoff, PE
Director, Environmental Projects
National Railroad Passenger Corporation
400 West 31st Street, 4th floor
New York, NY 10001

Re: PCB in Air Sampling Plan – Penn Station
New York, New York
TRC Project # 258098

Dear Mr. Mohlenhoff:

Following the discovery of polychlorinated biphenyls (PCBs) in accumulated sediment on the concrete body tracks adjacent to the Penn Station New York platforms, Amtrak requested that TRC Environmental Corporation (TRC) perform PCB air sampling within the station to determine background concentrations. To date, several rounds of air sampling have been conducted at various times on the track level, and on the upper and lower concourses. This work has been performed as part of an ongoing evaluation of potential exposures to employees, passengers, and the general public. At this time, Amtrak has requested that TRC develop a plan to perform air sampling on all three levels at the same time.

AIR SAMPLING PLAN

Air sampling will be conducted throughout Penn Station to determine potential PCB inhalation exposure levels to passengers, employees, and the general public. Sampling will be performed at track level, on the lower concourse level, which primarily services riders of the Long Island Rail Road, and on the upper concourse level, which primarily services riders of Amtrak and New Jersey Transit. Please see Attachment A for a copy of the proposed sample location drawings. The sampling is tentatively scheduled to be performed in late September of 2017.

Sampling and analysis for polychlorinated biphenyls (PCBs) will be conducted in accordance with EPA TO-10A method. Samples will be collected by drawing a measured volume of air through a sorbent cartridge containing polyurethane foam (PUF), using a low volume vacuum pump, at a flow rate of approximately 5 liters per minute (lpm). The sampling trains will be

calibrated at the beginning and end of the sampling period using a primary calibrator. No quartz-fiber pre-filter will be utilized, so sample analysis will include both particulate and vapor phases. Sampling will be performed at a height of around five (5) feet. Samples will be collected between approximately 10:00pm and 5:00am. A field blank sample will be included for quality assurance purposes. Analysis of the samples will be performed by Con-Test Analytical Laboratories, located in East Longmeadow, Massachusetts, an independent third-party industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033).

Track level sampling:

Sampling pumps will be stationed at the following ten (10) locations:

- Track level 01 - East side of Platform 2, near stairs;
- Track level 02 - West side of Platform 2, near stairs;
- Track level 03 - East side of Platform 4, near stairs;
- Track level 04 - West side of Platform 4, near stairs;
- Track level 05 - East side of Platform 6, near stairs;
- Track level 06 - West side of Platform 6, near stairs;
- Track level 07 - East side of Platform 8, near stairs;
- Track level 08 - West side of Platform 8, near stairs;
- Track level 07 - East side of Platform 10, near stairs, and
- Track level 08 - West side of Platform 10, near stairs.

Lower level air sampling:

Sampling pumps will be stationed at the following nine (9) locations:

Exit Concourse

- Lower level 01 - By entrance to tracks 3/4
- Lower level 02 - By entrance to tracks 7/8
- Lower level 03 - By entrance to tracks 11/12
- Lower level 04 - By entrance to tracks 15/16
- Lower level 05 - By entrance to tracks 18/19

Central Concourse

- Lower level 06 - By entrance to tracks 15/16
- Lower level 07 - By entrance to tracks 18/19

Main Gate Area

- Lower level 08 - By tracks 11/12
- Lower level 09 - By tracks 15/16
- Lower level 10 - By tracks 18/19

Mezzanine and Upper level air sampling:

Sampling pumps will be stationed at the following locations:

Amtrak Concourse

- Upper level 01 - By entrance to tracks 7/8
- Upper level 02 - By entrance to tracks 11/12
- Upper level 03 - By entrance to tracks 15/16

New Jersey Transit Concourse

- Upper level 04 - By entrance to tracks 3/4
- Upper level 05 - By entrance to tracks 7/8

One (1) duplicate sample will be collected at one of the track level locations, on either platform 2 or platform 4, for quality control purposes. Additionally, temperature and relative humidity will be collected and data logged at select locations on each level, and samplers will make note of any train traffic during the sampling period.

TRC appreciates the opportunity to provide you with these continuing industrial hygiene testing and consulting services. If you have any questions or comments, please contact us at (212) 221-7822.

Sincerely,

TRC ENVIRONMENTAL CORP.

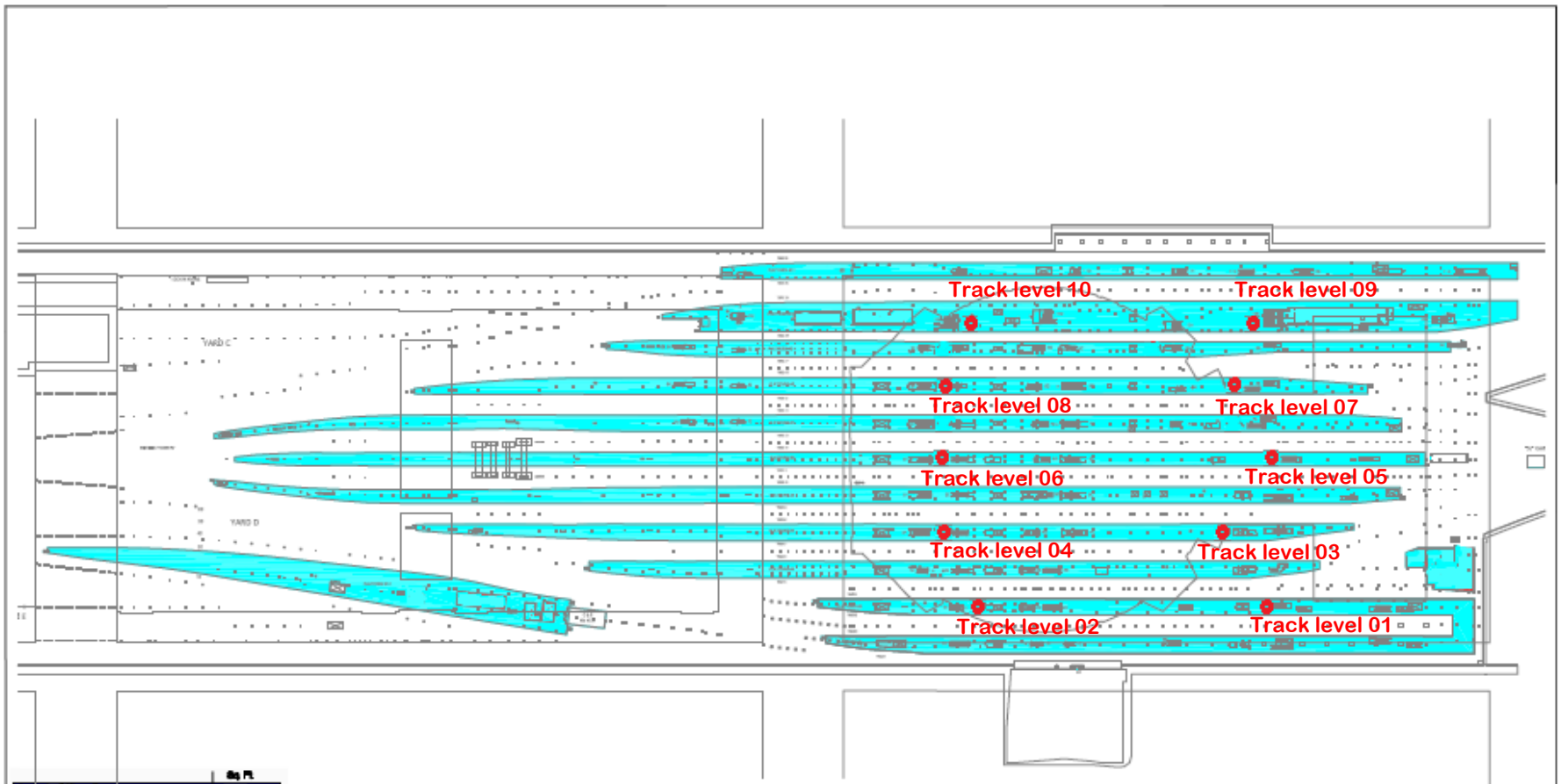
John P. Springston, CIH, CSP, FAIHA
Industrial Hygiene Program Manager

Kara Sweeney Parker
Project Manager

Attachment A – Sample location drawings

ATTACHMENT A

SAMPLE LOCATION DRAWINGS



Amtrak®

Office of Chief Engineer
STRUCTURES
National Railroad Passenger Corporation
30th Street Station, Philadelphia, Pennsylvania 19104

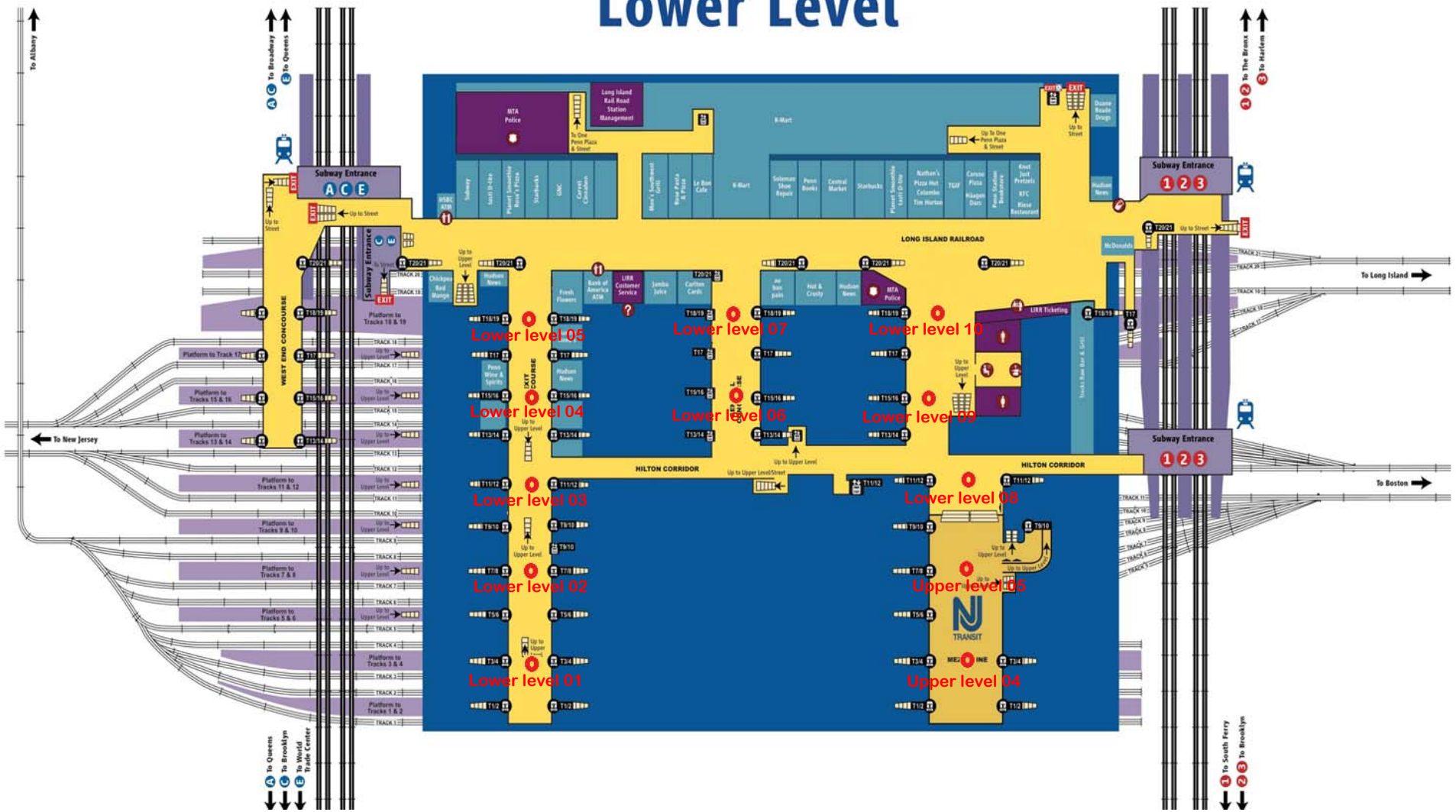
PROJECT NORTH

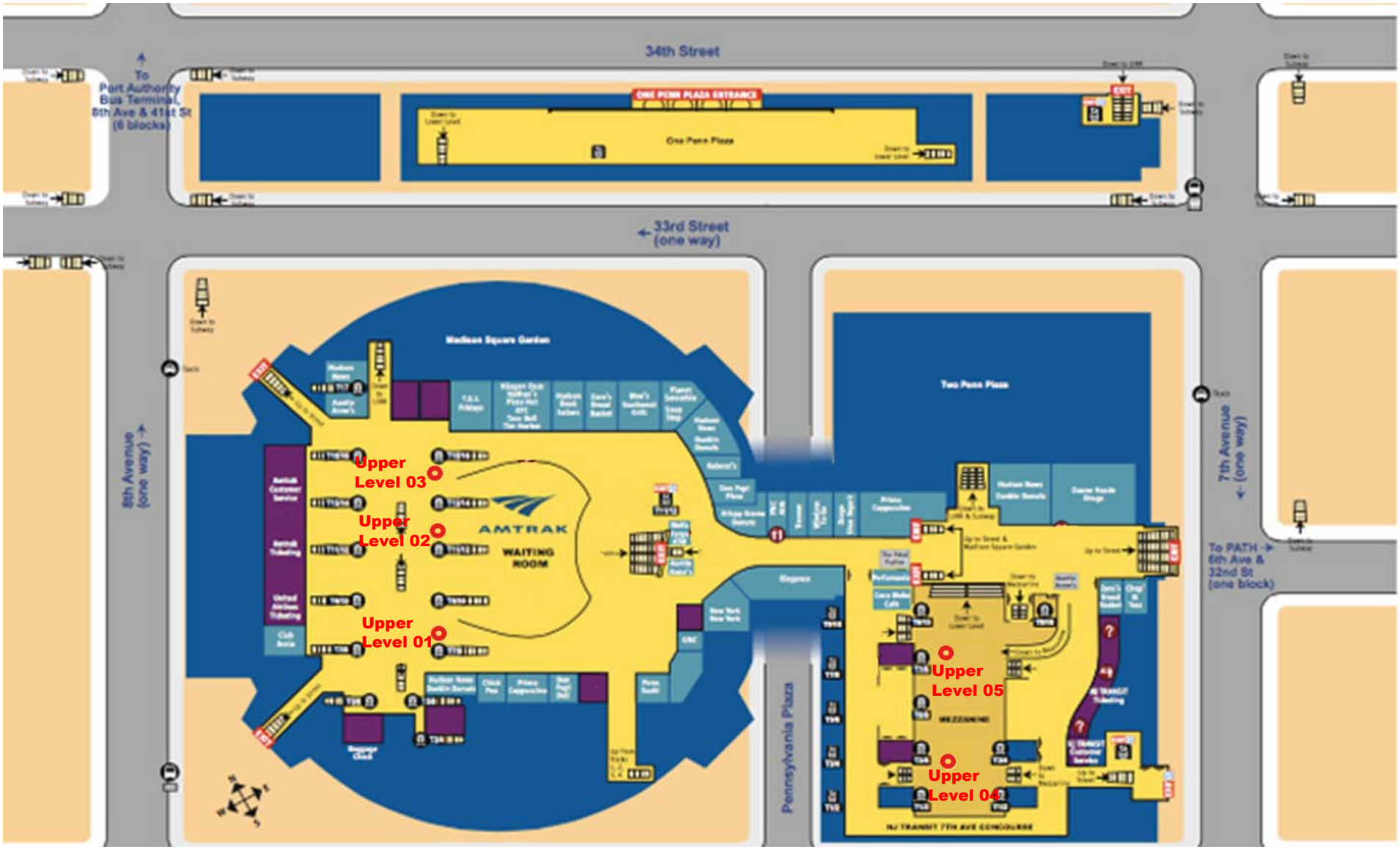
KEY PLAN

NEW YORK CITY, NY
PENN STATION NEW YORK
SQUARE FOOT ANALYSIS
PLATFORM LEVEL

A-4

Lower Level


To Albany
To Broadway
To Queens
To The Bronx
To Harlem
Subway Entrance
A C E
Subway Entrance
1 2 3
LONG ISLAND RAILROAD
To Long Island
To New Jersey
To Boston
WEST END CONCOURSE
HILTON CORRIDOR
HILTON CORRIDOR
Lower level 05
Lower level 07
Lower level 10
Lower level 04
Lower level 06
Lower level 09
Lower level 03
Lower level 08
Lower level 02
Upper level 05
Lower level 01
Upper level 04
To Queens
To Brooklyn
To World Trade Center
To South Ferry
To Brooklyn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Madison Square Garden
Two Penn Plaza
Upper Level 03
Upper Level 02
Upper Level 01
AMTRAK
WAITING ROOM
Pennsylvania Plaza
Upper Level 05
Upper Level 04
NJ TRANSIT 7TH AVE CONCOURSE

Appendix C

Air Monitoring Plan prepared by Mott MacDonald

MOTT MACDONALD

Air Monitoring Plan

Penn Station, New York

January 23, 2018

Mott MacDonald
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United States of America

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Air Monitoring Plan

Penn Station, New York

January 23, 2018

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2 Air Monitoring 3
2.1 Procedure 3

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Figure 1 – Areas of Interest

1 INTRODUCTION

Mott MacDonald has prepared this Air Monitoring Plan (Plan), on behalf of the National Railroad Passenger Corporation (Amtrak), to monitor dust at Penn Station New York (PSNY). This dust may be generated at the track level during removal of concrete, wood block ties, and sediment/"tar-like" material from the track structure. Dust may also be generated during the removal of dust from light fixtures, and other overhead conduits and appurtenances on platforms at PSNY. The purpose of this Plan is to minimize fugitive dust generation and conduct air monitoring to document any impacts outside of the work area.

The platform/track level at Penn Station encompasses approximately 28 acres and includes 11 platforms totaling over four miles in length, 21 tracks totaling over 14 miles in length, and four yard areas. The overall Station has been designated into six Areas of Interests (AOIs) as presented on Figure 1.

- AOI-1 corresponds to the concrete track structure and adjacent platforms. The concrete track structures include wood block ties and have a concrete trough in the center of the gauge.
- AOI-2 corresponds to yard areas A, D, and E that are located west of AOI-1.
- AOI-3 corresponds to C yard located west of AOI-6.
- AOI-4 corresponds to track areas east of AOI-1 and AOI-6.
- AOI-5 corresponds to track areas west of AOI-1 and AOI-6 that are not included in AOI-2 and AOI-3.
- AOI-6 corresponds to Tracks 19, 20, and 21 and adjacent platforms. These are typical ballast stone and not concrete track construction.

This plan primary focus is for AOI-1 and the platforms but can be adapted for use throughout any of the listed AOIs.

1.1 Background Information

PCBs have been detected in sediment located on the concrete track structure adjacent to the platforms; the sediment has accumulated over time on the concrete track structure. Samples of the sediment were collected and submitted for PCB analysis from Tracks 1 through 18 in 2016. Tracks 19 through 21 are typical ballast stone and tie construction and do not contain concrete track structures. PCBs were detected in sediment samples. Following the removal of the sediment, a "tar-like" material was encountered on the track structure. PCBs were detected in the "tar-like" material. Following the removal of the sediment and "tar-like" material and cleaning

of the track structure, concrete chip samples are collected for analysis to document the concentrations remaining in the concrete.

Dust has accumulated on various surfaces including, but not limited to, the tops of overhead light fixtures, conduits, pipes, brackets, walls, signs, and other surfaces. PCBs were detected in dust samples.

2 Air Monitoring

Airborne particulate matter (PM) consists of many different substances suspended in air in the form of particles (solids or liquid droplets) that vary widely in size. Inhalation hazards are caused if the intake of these particles includes intake of vapors and/or contaminated dust. Particles less than 10 micrometers in diameter (PM-10), which include both respirable fine (less than 2.5 micrometers) and coarse (less than 10 micrometers) dust particles, pose the greatest potential health concern because they can pass through the nose and throat and enter the lungs.

During the performance of planned work activities, PM in the form of potentially PCB- affected dust may be generated. The greatest potential for the generation of affected dust is during the excavation and handling of impacted media. The purpose of the air monitoring is to document the PM that is leaving the work area.

2.1 Procedure

Air monitoring will be completed using a minimum of two Thermo Electron Corporation personal Data Ram model (PDR-1000) portable dust meters. An additional backup dust meter will be present in the event of equipment malfunction. The meters will be calibrated in the field and placed at temporary monitoring stations approximately five feet from the work area. PM air monitoring will determine if fugitive dust particles are present in the ambient air during active removal activities. The locations of the monitoring stations will be determined daily based on the work being performed and potential receptors.

The dust monitors provide real-time monitoring of PM less than 10 micrometers in size (PM-10) and integrated over a period of 15 minutes for comparison to the airborne PM action levels. The dust monitors are equipped with an audible alarm to indicate exceedance of the action level. The threshold action level established is 0.15 milligrams per cubic meter (mg/m^3) for a 15-minute average. The established action level was taken from the New York State Department of Health (NYSDOH) Community Air Monitoring Plan in the New York State Department of Environmental Conservation (NYSDEC) document titled DER-10 Technical Guidance for Site Investigation and Remediation dated May 2010. The air monitoring and dust control measures will consist of the following:

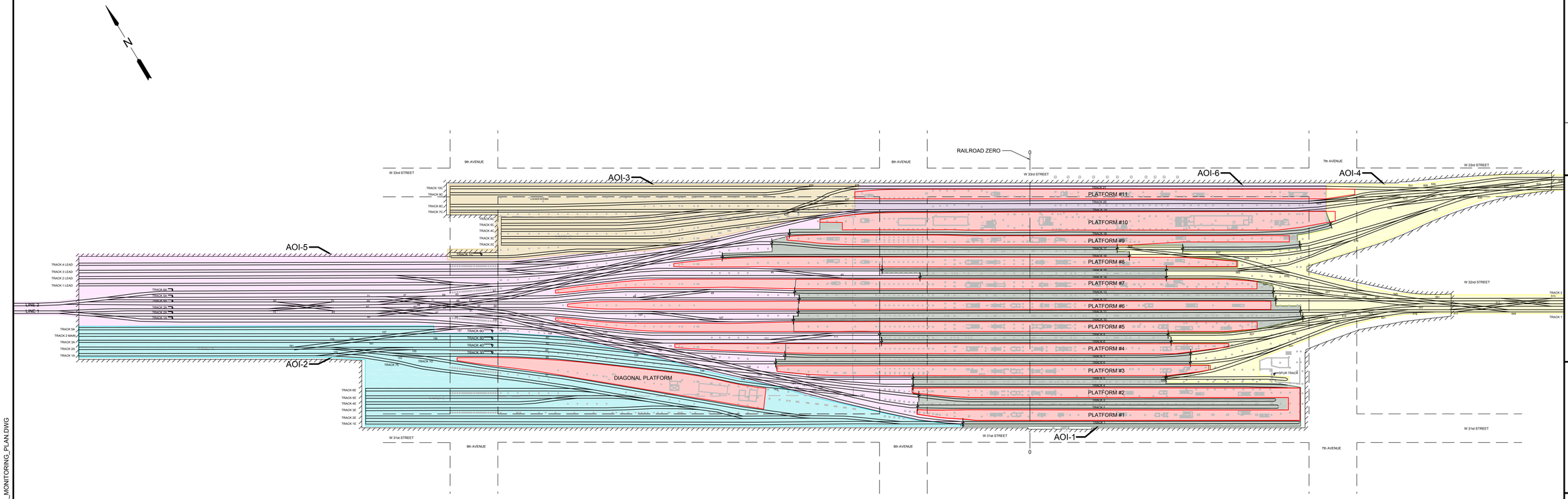
- PM concentrations will be monitored continuously at monitoring stations approximately five feet from activities being performed on track structures or platform level. In addition, the presence of dust will be visually assessed during all work activities. The dust monitors shall automatically log instantaneous readings every 60 seconds. In

addition, readings will be manually recorded at a rate of one per 30 minutes of work and reported in Amtrak's daily log.

If PM levels in air leaving the work zone are detected in excess of 0.150 mg/m^3, it should be determined if the excess concentrations are attributable to background conditions not related to work activities (e.g. work being conducted by others). If background contributing conditions are confirmed, work may proceed. If the working site PM concentrations are more than the action level, dust suppression measures (e.g. misting with water) should be implemented. Water will be applied either by small hand held sprayers, sprinklers, or hose nozzles. Should the action level of 0.150 mg/m^3 continue to be exceeded after a 15-minute period, or visible dust persists, work must stop until additional corrective actions can be implemented.

Figures

PLOT DATE: 01/22/2018 11:45 AM
SAVE DATE: 01/22/2018 11:45 AM



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. RAILROAD ZERO DENOTES A REFERENCE POINT AT PENNSYLVANIA STATION, NEW YORK. SAMPLE LOCATIONS WILL BE REFERENCED FROM RAILROAD ZERO.

3. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

LEGEND:

- 89 — SWITCH NUMBER
- BALLAST/CONCRETE TRANSITION
- WALLS
- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM

0 200 400

No.	Revision	Date

Professional Engineer - N.J. Lic. No.
FOR REVIEW PURPOSES ONLY
Date

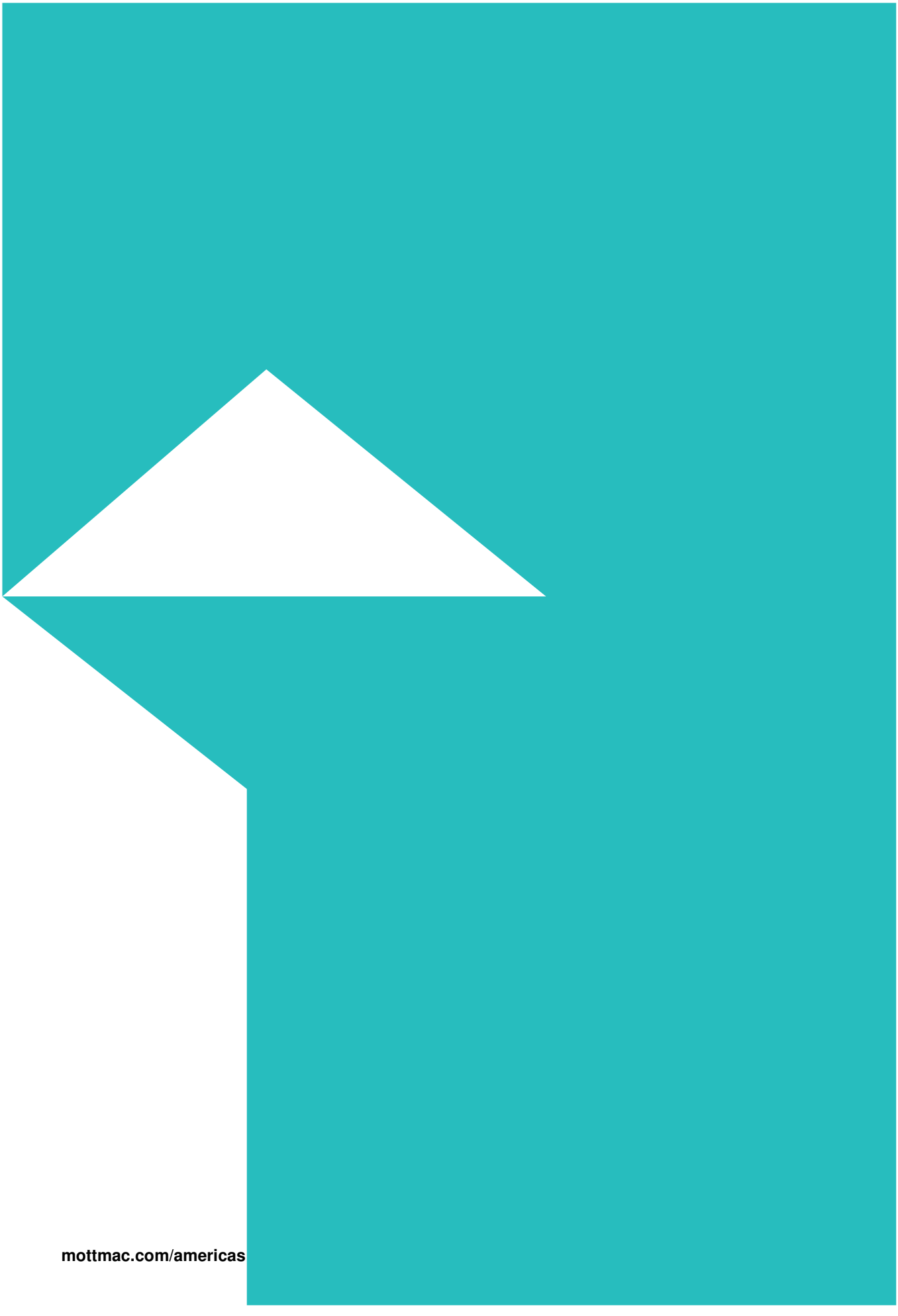
Date	1/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

MOTT MACDONALD
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
AREAS OF INTEREST MAP
AIR MONITORING PLAN
FIGURE 1

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\1 - DRAWINGS\NEW FOLDER STRUCTURE\MULTIPLE_AOIS\FULL_STATION\DWG\AOI-1-2-3-4-5-6_AREAS-OF-INTEREST-MAP-AIR_MONITORING_PLAN.DWG



mottmac.com/americas

Appendix D

Screening Level Evaluation for the Passenger Envelope, Penn Station, New York prepared by Stantec dated January 23, 2018

Screening Level Evaluation for the Passenger Envelope, Penn Station, New York

Penn Station, New York City

Stantec

Prepared for:
Amtrak

Prepared by:
Stantec Consulting Services, Inc.

January 23, 2018

Sign-off Sheet

This document entitled Screening Level Evaluation for the Passenger Envelope, Penn Station, New York was prepared by Stantec Consulting Services Inc. ("Stantec") for the account of Amtrak (the "Client"). Any reliance on this document by any third party is strictly prohibited. The material in it reflects Stantec's professional judgment in light of the scope, schedule and other limitations stated in the document and in the contract between Stantec and the Client. The opinions in the document are based on conditions and information existing at the time the document was published and do not take into account any subsequent changes. In preparing the document, Stantec did not verify information supplied to it by others. Any use which a third party makes of this document is the responsibility of such third party. Such third party agrees that Stantec shall not be responsible for costs or damages of any kind, if any, suffered by it or any other third party as a result of decisions made or actions taken based on this document.

Prepared by Deborah L Gray
(signature)

Deborah L. Gray, Ph.D., DABT

Reviewed by
(signature)

Christopher La Londe, MPH-VPH

Approved by
(signature)

Frank Aceto, PG #464

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Executive Summary

Early in 2016, polychlorinated biphenyl compounds (PCBs) were detected in sediment on the Concrete Track structures in Pennsylvania (Penn) Station, New York City (PSNY). Amtrak quickly responded to this discovery by collecting samples of sediment on the Concrete Tracks, concrete chips, and accumulated bulk dust, as well as air samples. At the same time, Amtrak initiated a program to remove the sediment and clean the Concrete Track structures and other areas at PSNY. Cleaning activities are ongoing as of December 2017.

A Remedial Investigation (RI) of PSNY has been initiated, but is anticipated to take 5 years or more to complete given the difficulties of working in the busiest passenger transportation facility in North America. Amtrak considered it prudent to address concerns about potential exposures to PCBs for members of the public and people who work in the Station concurrent with implementing the RI. This Screening Level Evaluation of the Passenger Envelope of PSNY was based on an understanding of receptor activities and currently available empirical data documenting concentrations of PCBs in air and in samples of settled dust. The quantity and quality of analytical data were considered sufficient to support this Screening Level Evaluation in advance of completing the RI.

PSNY is not an "open-air site" with traditional environmental media (soil, groundwater, surface water, and ambient air) as envisioned by the Comprehensive Environmental Response, Compensation, and Liabilities Act (CERCLA). Therefore, guidance documents designed specifically for CERCLA sites may not be appropriate for PSNY. However, this Screening Level Evaluation used conservative methods consistent with USEPA Risk Assessment Guidance for Superfund (RAGS).

PSNY is a complex multi-layered structure that is entirely below ground. Madison Square Garden and a high-rise office building (Two Penn Plaza) sit overtop of PSNY. The entrances to PSNY are from street level, and then descending to the Station. There are three main levels in PSNY; the Upper Concourse, the Lower Concourse, and the Platform/Track Level. The levels are connected by passageways consisting of stairs, escalators, and elevators.

The Passenger Envelope is defined by physical boundaries and the groups of people (receptors) who are present within those boundaries; specifically, those areas of PSNY where members of the public are present coincident with passenger train usage. The Passenger Envelope includes the publicly accessible portions of the Upper and Lower Concourses, the Passenger Platforms, and the passageways (stairs, elevators, escalators) connecting these areas. Members of the public include train passengers, non-railroad workers, and other visitors to PSNY. Railroad workers who have duties in the Concourses (e.g. customer service employees) or Platforms, but do not have duties in other non-public areas of PSNY were addressed in this Screening Level Evaluation.

In contrast to the Passenger Envelope, the Restricted Areas of PSNY are only accessible to railroad workers or other authorized personnel. Restricted Areas include the tracks where trains are actively moving; areas where out of service trains and track maintenance equipment are staged; electrical substations and catenary wires; utility conduits; track switches; and employee locker rooms/break rooms/offices. On the Platform, the edge of the concrete Platforms marks the physical boundary of the Passenger Envelope. There are also specific sections of the Platforms where access is restricted to only railroad workers or other authorized personnel. For example, railroad workers may report to locker rooms/offices on the Platforms, but perform most of their workday activities in Restricted Areas. These classes of railroad workers will be evaluated in the Risk Assessment portion of the RI.

Concentrations of PCBs measured in air in the three areas of the PSNY Passenger Envelope have been well characterized by multiple rounds of sampling conducted from April 2016 through September 2017(TRC reports). Sample results for bulk dust were used as a surrogate for potential contact with settled dust on surfaces on the Passenger Platforms.

Screening level concentrations for PCBs in air and dust (corresponding to an incremental lifetime cancer risk of 1 in 100,000 or 1E-05) were derived using the Oak Ridge National Laboratory Risk Assessment Information System (ORNL-RAIS) on-line calculator with USEPA default variable values adjusted to align with specific receptor activities within the three areas of the Passenger Envelope. Concentrations of PCBs measured in air on the Concourses and Passenger Platforms, and in samples of bulk dust from light fixtures on the Passenger Platforms were compared to receptor-specific screening levels.

The conclusions of this Screening Level Evaluation for the Passenger Envelope of PSNY are summarized below.

- The mean concentrations of PCBs measured in air on the Concourses and Passenger Platforms were lower than receptor-specific screening levels for all representative groups of individuals who are frequently present within the Passenger Envelope of PSNY.
- The mean concentration of PCBs measured in samples of bulk dust collected from on top of light fixtures was lower than receptor-specific screening levels for hand contact with surfaces and incidental ingestion of dusts on the Passenger Platforms for all representative groups of individuals who are frequently present within this area of the Passenger Envelope.

Amtrak intends to continue periodic synoptic air monitoring. The results of each air monitoring event will be compared to the assumptions established in this Screening Level Evaluation.

Abbreviations

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CSM	Conceptual Site Model
LIRR	Long Island Railroad
NJ Transit	New Jersey Transit
ORNL-RAIS	Oak Ridge National Laboratory Risk Assessment Information System
PCBs	Polychlorinated Biphenyl Compounds
PSNY	Penn Station, New York
RAGS	Risk Assessment Guidance for Superfund
RfD	Reference Dose
RI	Remedial Investigation
RIWP	Remedial Investigation Work Plan
TSCA	Toxic Substances Control Act
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

Early in 2016, polychlorinated biphenyl compounds (PCBs) were detected in sediment on the Concrete Track structures in Pennsylvania (Penn) Station, New York City (PSNY). Amtrak quickly responded to this discovery by collecting sediment, concrete chip, and accumulated bulk dust and air samples. At the same time, Amtrak initiated a program to remove the sediment and clean the Concrete Track structures and other areas at PSNY. Cleaning activities are ongoing as of December 2017.

Amtrak's actions to remove PCB-contaminated sediments from the Concrete Track structures, the findings of initial sampling, and the proposed strategy for determining the extent of PCB contamination within PSNY are described in detail in the Remedial Investigation Work Plan (RIWP) to which this report is an Appendix.

1.1 PURPOSE

The purpose of this Screening Level Evaluation is to determine if the concentrations of PCBs detected in air and dust in public areas of PSNY are safe for people who are frequently present in the Station. The public areas of PSNY comprise the Passenger Envelope. This Screening Level Evaluation is a conservative assessment of air monitoring conducted from April 2016 through September 2017 and findings from bulk dust samples. PSNY is a highly visible public venue, and this Screening Level Evaluation reflects Amtrak's commitment to the safety of the public who use the Station and the people who work there.

Amtrak intends to continue conducting periodic air sampling for PCBs in the Passenger Envelope with the implementation of the RIWP and any potential remedial measures. The results of future air sampling (and any other sampling that may be relevant) will be incorporated into the Screening Level Evaluation to determine if future findings are consistent with the data on which the evaluation described in this report is based.

1.2 HISTORY OF PENN STATION

PSNY was constructed by the former Pennsylvania Railroad between 1901 and 1910 and has been an operating railroad station for over 100 years. The Station has been owned and operated by Amtrak since the 1970s. PSNY is a critical component of passenger rail service in the Northeast Corridor and will continue to fill that role into the future. Note that Figures showing the layout of Penn Station and photographs (Appendix A) from publicly available sources are included as attachments to this report.

The structure has been modified numerous times over the past century and is currently undergoing renovations. Track level at PSNY occupies over four full City blocks (28 acres) between 7th Avenue and 9th Avenue, and West 31st Street to West 33rd Street in Manhattan (Appendix A, Photo #1).

SCREENING LEVEL EVALUATION FOR THE PASSENGER ENVELOPE, PENN STATION, NEW YORK

Introduction
January 23, 2018

The entire Station is below the street elevation and consists of three levels; the Upper Concourse, the Lower Concourse, and the Platform/Track Level. As of November 2017, PSNY has seven (7) tunnels through which trains arrive and depart, twenty-one (21) tracks with a combined length of 14 miles, and eleven (11) Passenger Platforms that service the tracks with a combined length of 4 miles. There are also four (4) train yards and associated infrastructure within PSNY.

Amtrak, New Jersey Transit (NJ Transit), and the Long Island Railroad (LIRR) operate passenger trains through PSNY. NJ Transit trains use Tracks 1 through 4, Amtrak and NJ Transit both use Tracks 5 through 14, and LIRR uses Tracks 15 through 21. PSNY operates 24-hours a day year-round and is the busiest passenger transportation facility in North America, serving more than 430,000 passengers every day.

2.0 EXPOSURE ASSESSMENT

2.1 DEFINITION OF THE PASSENGER ENVELOPE

PSNY is not an "open-air site" with traditional environmental media (soil, groundwater, surface water, and ambient air) as envisioned by the Comprehensive Environmental Response, Compensation, and Liabilities Act (CERCLA). Therefore, guidance documents designed specifically for CERCLA sites may not be appropriate for PSNY. However, this Screening Level Evaluation used conservative methods consistent with USEPA Risk Assessment Guidance for Superfund (RAGS).

PSNY is a complex multi-layered structure that is entirely below ground. Madison Square Garden and a high-rise office building (Two Penn Plaza) sit overtop of PSNY. The entrances to PSNY are from street level, and then descending to the Station (Appendix A, Photo #2). The Station is below street elevation and consists of three main levels; the Upper Concourse, the Lower Concourse, and the Platform/Track Level. The levels are connected by passageways consisting of stairs, escalators, and elevators.

The Passenger Envelope is defined by physical boundaries and the groups of people (receptors) who are present within those boundaries; specifically, those areas of PSNY where members of the public are present coincident with passenger train usage. The Passenger Envelope includes the publicly accessible portions of the Upper and Lower Concourses, the Passenger Platforms, and the passageways (stairs, elevators, escalators) connecting these areas. Members of the public include train passengers, non-railroad workers, and other visitors to PSNY. Railroad workers who have duties in the Concourses (e.g. customer service employees) or Passenger Platforms, but do not have duties in other non-public areas of PSNY were addressed in this Screening Level Evaluation.

In contrast to the Passenger Envelope, the Restricted Areas of PSNY are only accessible to railroad workers or other authorized personnel. Restricted Areas include the tracks where trains are actively moving; areas where out of service trains and track maintenance equipment is staged; electrical substations and catenary wires; utility conduits; track switches; and employee locker rooms/break rooms/offices. On the Platform, the edge of the concrete Platforms marks the physical boundary of the Passenger Envelope. There are also specific sections of the Platforms where access is restricted to railroad workers or other authorized personnel. For example, railroad workers may report to locker rooms/offices on the Platforms, but perform most of their workday activities in Restricted Areas. These classes of railroad workers will be evaluated in the Risk Assessment portion of the RI.

2.1.1 Upper Concourse

Amtrak and NJ Transit ticketing and customer services are located on the Upper Concourse. There is a passenger waiting area, merchandise shops, and restaurants (Appendix A, Photos #3, 4). The Upper Concourse is connected to the Lower Concourse and Passenger Platforms/Track Area by stairways, elevators (Appendix A, Photo #13), and escalators (Photo #1 Below and in Appendix A as Photo #12) directly to the Passenger Platforms/Track Areas that service Amtrak and NJ Transit trains. Figure 1 is a schematic of the Upper Level Concourse (NJ Transit 2017).

Photo 1. Escalators (Photo Credit: WSJ/Associated Press)

2.1.2 Lower Concourse

LIRR ticketing and customer service is located on the Lower Concourse (Appendix A, Photos #5, #6) along with merchandise shops, and restaurants. Stairways and elevators connect the Lower Concourse with the Passenger Platform/Track Level that services LIRR trains (Appendix A, Photo #11, #12). Figure 2 is a schematic of the Lower Level Concourse (NJ Transit 2017).

2.1.3 Passenger Platforms

The Passenger Platforms are on Track Level of the Passenger Envelope. There are eleven Platforms serving 21 tracks. Trains arrive and depart through large portals on the east and west ends of the Station (Appendix A, Photos #7, 8, 9, 10). Except for the public portions of the Passenger Platforms, access to other areas on the Platform/Track Level is restricted to Amtrak, NJ Transit, LIRR workers and other authorized personnel. Figure 3 is a schematic of the Track Level and Passenger Platforms (Amtrak 2017).

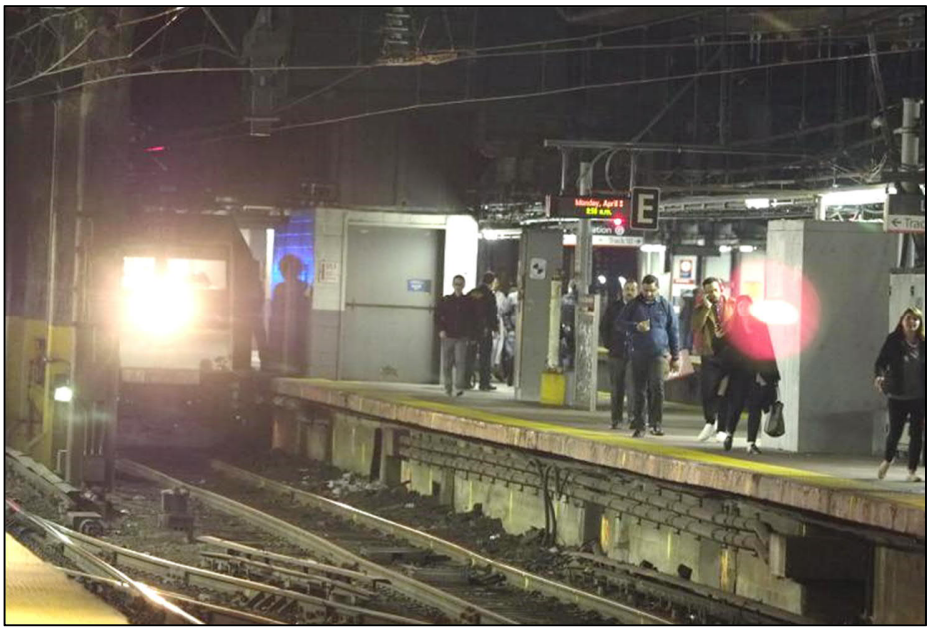

Photo 2. Platform Level. (Photo Credit: Anthony Delmundo/nydailynews.com)

The physical surroundings of the Passenger Platforms and track areas are distinctly different from the finished interior environment of the Concourses. Although the space is open with visibility of tracks and train movements, the outside environment is not visible from the Passenger Platforms. Surfaces on the Platforms are concrete with tile and painted finishes on some of the walls. Concrete, bedrock, structural steel, concrete track structures, utilities, and catenary wires are prominent features of the vast subterranean space, where trains arrive and depart. The Passenger Platforms are illuminated by fluorescent lights suspended from the ceiling.

2.2 DATASETS

Two sources of information were used to evaluate potential receptor exposures to PCBs in the Passenger Envelope; air monitoring results and analytical results from bulk dust samples.

2.2.1 Air Monitoring Results for PCBs

Beginning in April 2016, TRC Environmental (on behalf of Amtrak) has collected air samples from representative locations on the Passenger Platforms; and Upper and Lower Concourses. In June and September 2017, air monitoring was conducted within the same 24-hour period on the Platforms and Concourses (synoptic sampling). Air samples were collected and analyzed consistent with USEPA Method TO-10A. Analytical results are presented as the sum of the Aroclor concentrations detected in each sample (both vapor and particulate phase PCBs). Air sampling plans and reports prepared by TRC are provided in Appendix B to the RIWP. Tables presenting the individual air monitoring results for portions of the Platform/Track Level (Table 1), the Lower Concourse (Table 2), and the Upper Concourse (Table 3) are attached to this Screening Level Evaluation.

Text Table 1 presents statistical summaries of the concentrations (sum of Aroclors detected) in air samples collected from the Passenger Platforms, Lower Concourse, and Upper Concourse from April 2016 through September 2017. Laboratory analytical reports for the air sampling conducted within the Passenger Envelope are provided with the TRC reports.

Text Table 1. Summary of PCB Concentrations in Air for the Passenger Envelope, PSNY

Areas	Frequency of Detection	Min	Max	KM Mean	KM SD
Passenger Platforms	33/47	22	227	63	49
Lower Concourse	21/37	20	100	32	20
Upper Concourse	11/17	18	66	27	13

1. All concentrations in ng/m^3
2. Air samples collected by TRC from April 2016 through September 2017
3. KM Kaplan Meier statistics better represent the center and spread of statistical distributions with datasets that include non-detect values at the laboratory reporting limits (RL); calculated with USEPA ProUCL Version 5.1.00. SD is the Standard Deviation around the KM mean.
4. Statistics rounded to nearest whole number.
5. Frequency of Detection is the number of samples with PCB concentrations above the laboratory RL divided by the total number of samples in each area

2.2.2 Bulk Dust Sample Results for PCBs

Samples of accumulated dust were collected from the tops of light fixtures on Passenger Platforms 2, 3, and 6 and analyzed for PCBs. Concentrations of individual Aroclors were added and reported as the concentration (sum of Aroclors detected) in each sample as shown in Text Table 2.

Text Table 2. PCBs in Dust from Light Fixtures on Passenger Platforms (2016)

Location	PCB mg/kg
P6T12-D3-West	10.80
P6T12-D1-East	2.28
P6T12-D2-Center	8.67
P3T6-D2-West	32.90
P3T6-D1-East	23.30
P2T4-D1-West	11.00
P2T4-D2-Center	2.78
P2T4-D3-East	4.42
Kaplan Meier Mean	12.02
Kaplan Meier SD	10.79

1. Kaplan Meier statistics calculated with USEPA ProUCL Version 5.1.00

2.3 RECEPTORS

Hundreds of thousands of people are present in PSNY every day, and can be represented by one of the following groups. Railroad workers who may be present on the Platforms while reporting to locker rooms/offices, but who perform most of their daily activities in non-public Restricted Areas will be evaluated in the risk assessment portion of the RI.

- Amtrak & NJ Transit Ticketing and Customer Service Agents: These individuals spend most of their day working at the ticket counter or other nearby locations in the Upper Concourse. They rarely go to the Passenger Platforms during the work day.
- LIRR Ticketing and Customer Service Agents: These individuals spend most of their day working at the ticket counter or other nearby locations in the Lower Concourse. They rarely go to the Passenger Platforms during the work day.
- Vendors and Shop Workers: There are shops and restaurants on both the Upper and Lower Concourses (Appendix A, Photo #16). Shop workers rarely go to the Passenger Platforms during the work day.
- Porters (Amtrak Red Caps): These individuals assist Amtrak passengers and move luggage from the Amtrak customer service area on the Upper Concourse to the Passenger Platforms. The Porters may walk briefly through the Lower Concourse, but it is assumed that their daily activities are evenly split between the Amtrak customer service area on the Upper Concourse and the Platforms where Amtrak trains arrive and depart.
- Railroad Worker: These individuals report to an enclosed office, or other areas on non-public Sections of the Passenger Platforms that service trains. This category of worker spends all of his/her day performing railroad duties which may include: cleaning the train coaches while trains are parked; performing inspections; completing paperwork; or performing other duties on the Passenger Platforms.
- Housekeeping: These individuals move through the Concourses and Passenger Platforms cleaning surfaces, emptying trash cans, etc. It is assumed that these individuals divide their work day between the two Concourses with slightly less time cleaning on the Passenger Platforms.
- Police: Police and security personnel move through the Concourses and Passenger Platforms. It is assumed that police and security divide their work day between the two Concourses with slightly less time spent patrolling the Passenger Platforms.
- Casual Visitors: This category of receptors includes people who occasionally visit the shops and restaurants, but are not train-riding passengers, and are not employees of the businesses in PSNY. Homeless individuals who seek shelter in PSNY are also in this category. Casual visitors rarely go to the Passenger Platforms.
- Passengers: This receptor category includes: individuals who commute in and out of PSNY every work day (five days a week); individuals who commute through PSNY two times a week (e.g. people who enter NYC on Monday and depart on Friday); and people who come through PSNY less frequently, including both children and adults (Appendix A, Photo #15). Note that some receptors who work in PSNY may also arrive and depart by train.

The estimated maximum number of hours per day, days per year, and years that a representative receptor might be present in the Upper Concourse, Lower Concourse, and Passenger Platforms is indicated in Text Table 3.

Text Table 3. Representative Receptors

Receptors	Exposure Hours/Day by Location			Days Per Week	Days Per Year	Years
	Upper Concourse	Lower Concourse	Platforms			
Amtrak/NJ Transit Customer Service Ticketing Agents	8	--	--	5	250	25
LIRR Customer Service Ticketing Agents	--	8	--	5	250	25
Vendors & Shop Workers	8	--	--	5	250	25
Vendors & shop Workers	--	8	--	5	250	25
Red Caps (Porters)	4	--	4	5	250	25
Railroad Worker	--	--	8	5	250	25
Housekeeping	3	3	2	5	250	25
Police and Transit Security	3	3	2	5	250	25
Casual Visitors (Not Passengers)	2	2	--	1	50	25
Passengers	0.5	0.5	0.5	5	250	25

2.4 PATHWAYS AND ROUTES OF EXPOSURE

The Conceptual Site Model (CSM) is an analysis and representation of the physical pathways by which chemicals of potential concern, PCBs in this case, move from sources to locations where

receptors (people) may come into direct contact with the environmental media containing the chemicals, and the routes of exposure (e.g. inhalation, ingestion, dermal absorption) by which the chemicals are taken into the receptor's body. The CSM for the Passenger Envelope of PSNY is a "living analysis" and may be modified if indicated by the findings of future investigations. This Screening Level Evaluation of the Passenger Envelope of PSNY is based on the current understanding of the CSM. A simplified representation of the CSM for the Passenger Envelope is shown in Text Table 4.

Text Table 4. Conceptual Site Model for the Passenger Envelope, PSNY

Receptors	Exposure Areas and Pathways of Exposure					
	Upper Concourse		Lower Concourse		Passenger Platforms	
	Air (1)	Surface Dust (2)	Air	Surface Dust	Air	Surface Dust
Amtrak & NJ Transit Customer Service and Ticket Agents	C	IC/NS	NA	NA	NA	NA
LIRR Customer Service / Ticket Agents	NA	NA	C	IC/NS	NA	NA
Vendors	C	IC/NS	C	IC/NS	NA	NA
Amtrak Porters	C	IC/NS	NA	NA	C	C
Railroad Worker	IC/NS	IC/NS	IC/NS	IC/NS	C	C
Police / Security	C	IC/NS	C	IC/NS	C	C
Housekeeping	C	IC/NS	C	IC/NS	C	C
Passengers	C	IC/NS	C	IC/NS	C	C
Casual Visitors	C	IC/NS	C	IC/NS	NA	NA

1. Air – Inhalation of air
2. Surface Dust – Hand contact with settled dust on surfaces resulting in incidental ingestion.
3. C – Exposure pathway is complete
4. IC/NS – Exposure pathway is incomplete or is not significant
5. NA – Not Applicable. The receptor is not frequently present in this area.

The receptors described in Section 2.3 may be exposed to PCBs in air throughout the Passenger Envelope and in settled dust on accessible surfaces on the Passenger Platforms. The rationale for designating pathways of exposure as complete or incomplete is discussed below.

2.4.1 Inhalation of PCBs in Air

PCBs have been measured in air samples from all three levels of the Passenger Envelope (Text Table 1). Inhalation of PCBs in air is a completed pathway of exposure for all groups of individuals who work in PSNY, or visit PSNY.

2.4.2 Dermal Contact and Incidental Ingestion of PCBs from Surfaces

High contact surfaces (e.g. ticket machines, ATMs, benches, railings) in the Upper and Lower Concourses are cleaned regularly. Walls, floors, and other structural surfaces in the Upper and Lower Concourses are constructed from stone, glazed tiles, metal, or are covered with smooth non-porous coatings. Easily accessible high-contact surfaces on the Passenger Platforms and passageways are cleaned regularly. All doors, escalators, handrails are cleaned nightly on each platform. Platforms 1 thru 7 are on a one week schedule; and one is cleaned every night in order by track number. Platforms 8 thru 11 are on an every four-day schedule; and one is cleaned every night in order by track number.

Easily reachable surfaces on the Passenger Platforms are also cleaned. However, the Passenger Platforms are within the larger open environment of the Track Level (where the public is not allowed). Surfaces on the Platforms may be influenced by the movement of air that originates from the tracks.

Given the physical differences between the two Concourses as compared to the Platform/Track Level, and the proximity of the Platforms to the concrete track structures, it is much more likely that contact with PCB-containing dust could be a complete pathway of exposure to receptors on the Platforms as compared to the Concourses. Hand contact with dust on surfaces may also occur in the passageways (stairs, escalators, and elevators) connecting the Passenger Platforms with the Concourses. Exposure to PCB-containing dust on surfaces in the Upper and Lower Concourses is unlikely to be significant, if it occurs at all.

The Screening Level Evaluation for the Passenger Envelope, PSNY used conservative USEPA default assumptions for soil to estimate incidental ingestion from hand to mouth transfer of settled dust containing PCBs on accessible surfaces on the Passenger Platforms. These are relatively straight-forward calculations that typically yield conservative estimates of chronic daily intake (dose).

3.0 SCREENING LEVEL EVALUATION OF EXPOSURE

This Screening Level Evaluation was based on empirical data for the concentrations of PCBs measured in air and settled dust, direct observations of receptor activities in the Passenger Envelope, and information about worker duties and movements provided by Amtrak. All receptors considered in the Screening Level Evaluation are assumed to have potential long-term (chronic) exposures to PCBs within the Passenger Envelope of PSNY.

Screening levels are typically concentrations of individual chemicals, PCBs in this case, in environmental media (soil/dust and air) that are mathematically derived to correspond to a pre-determined cancer risk (1 in 100,000 for this evaluation) and/or a non-cancer hazard quotient of 1.0. For this evaluation, screening levels were based on cancer risk for PCBs. The algorithms used to derive screening level concentrations incorporate variable values that represent receptor behaviors and characteristics (e.g. frequency and duration of exposure, amount of soil/dust ingested per day, hours per day for inhalation, body weight, and averaging time); and toxicity factors (e.g. oral cancer slope factor-CSF, inhalation unit risk-IUR, oral Reference Dose-RfD, and Reference Concentration-RfC) that characterize the dose-response relationship of the chemical for cancer and non-cancer health effects.

Screening levels are concentrations that an individual could be exposed to every day for many years (chronic exposure) and not experience a hypothetical cancer risk higher than that used to derive the screening level (e.g. 1 in 100,000 for this evaluation of PSNY) or a non-cancer hazard for systemic health effects higher than the reference dose. Concentrations of chemicals (PCBs) in environmental media from a site that are higher than risk-based screening levels do not necessarily mean that there are unacceptable risks to receptors, but may suggest that additional investigation is warranted. On the other hand, if the concentrations of a chemical (PCBs) measured in air or soil/dust are lower than screening levels, then it may be concluded that potential exposures are not likely to be of concern to human health.

The screening level concentrations for PCBs in air and dust developed for this evaluation of receptors within the Passenger Envelope of PSNY are consistent with USEPA RAGS methodology and the methods described in the User's Guide accompanying the November EPA Regional Screening Levels (RSL) Tables (USEPA, 2017). As discussed in the following sections, the generic default variable values embedded in the Oak Ridge National Laboratory-Risk Assessment Information System (ORNL-RAIS) on-line calculator were modified to align with the behavior patterns of the representative receptors known to be present within the Passenger Envelope of PSNY. This on-line calculator is maintained by ORNL on behalf of USEPA and is a frequently-used tool for the initial screening of sample data for environmental site assessments.

3.1 EXPOSURE TO PCBS IN AIR

The concentrations of PCBs detected in air in the Upper and Lower Concourses, and on the Passenger Platforms were compared to receptor-specific screening level concentrations derived using the ORNL-RAIS on-line calculator.

3.1.1 Screening Levels Derived Using the ORNL-RAIS Calculator

The ORNL-RAIS calculator was used to derive concentrations (sum of Aroclors detected) in air corresponding to a 1 in 100,000 incremental lifetime cancer risk (1E-05) for receptors breathing the air for 8 hours/day, 4 hours/day, or 2 hours/day, 250 days/year (5 days/week for 50 weeks), for 25 years. The exposure frequency (250 days per year) and exposure duration (25 years) are USEPA default values for evaluating "indoor" workers. Screening levels were calculated such that hours of exposure per day align with the receptor activities described in Section 2.3. Documentation from the ORNL-RAIS calculator is provided in Appendix B. The screening level concentrations of nanograms (sum of Aroclors detected) per cubic meter of air (ng/m^3) for these three exposure times are:

- 8 hours/day 215 ng/m^3
- 4 hours/day 430 ng/m^3
- 2 hours/day 858 ng/m^3

An individual receptor could spend 8 hours/day in one area (e.g. Upper Concourse, or Lower Concourse, or the Passenger Platforms) and experience an estimated incremental lifetime cancer risk of 1E-05 from exposure to an average concentration of 215 ng/m^3 PCBs in air in that area. The following categories of receptors spend all or most of their 8-hour workday in the same area.

- Amtrak and NJ Transit customer service and ticket agents; vendors and shop workers on the Upper Concourse
- LIRR customer service and ticket agents; vendors and shop workers on the Lower Concourse
- Vendors and shop workers on both Concourses
- Railroad Workers on the Passenger Platforms

Other categories of receptors who work in PSNY have duties on multiple levels of the Passenger Envelope. Each receptor's daily exposure to PCBs in air is a function of the amount of time he/she spends in a location(s) and the average concentration of PCBs in that location. Thus, the person may be exposed to varying concentrations of PCBs throughout the day. The following categories of receptors have daily duties on the Concourses and Passenger Platforms:

- Amtrak porters (Red Caps) - have activities on the Upper Concourse and the Passenger Platforms, and are assumed to spend 4 hours of an 8-hour day in each area.
- Police and Station Security, and Housekeeping - have activities on both Concourses and the Passenger Platforms. These two receptors are assumed to be present 3 hours per day on the Upper Concourse, 3 hours per day on the Lower Concourse, and 2 hours per day on the Passenger Platforms.
- And finally, train passengers and casual visitors to PSNY are not present in the Station for 8 hours every day. A casual visitor may come to PSNY to eat, shop, or sight-see, but is unlikely to spend more than 2 hours per day on one or both Concourses. Daily commuters may be present on all levels of PSNY while waiting for trains and disembarking from trains. It is conservatively estimated that a commuter may spend 1.5 to 2.0 hours per day in PSNY, with the least amount of time spent on the Passenger Platforms.

3.1.2 Comparison of Detected Concentrations of PCBs in Air to Screening Levels

Individuals move about the areas within PSNY, and the concentrations (sum of Aroclors detected) (volatile and particulate phases) measured in air are variable over time and location. The mean PCB concentration measured in air on each of the three levels was used to represent exposures over time and over the large areas within the Passenger Envelope of PSNY.

Upper Concourse: The KM mean (26.75 ng/m^3) concentration of PCBs measured in air samples from the Upper Concourse was lower than the screening level of 215 ng/m^3 for receptors who spend their 8-hour work day in this area of PSNY.

Lower Concourse: The KM mean (32.1 ng/m^3) concentration of PCBs measured in air samples from the Lower Concourse was lower than the screening level of 215 ng/m^3 for receptors who spend their 8-hour work day in this area of PSNY.

Passenger Platforms: The KM mean concentration (64 ng/m^3) of PCBs measured in air on the Passenger Platforms was lower than the screening levels for Railroad Workers who spend 8-hours per day in this area (215ng/m^3); Amtrak Porters who spend 4-hours per day in this area (430 ng/m^3), and for other receptors (Train Passengers, Police and Station Security, and Housekeeping employee) who spend 2 hours per day or less in this area (858 ng/m^3).

3.2 EXPOSURE TO PCBS IN SETTLED DUST ON PASSENGER PLATFORMS

3.2.1 Screening Levels Derived Using the ORNL-RAIS Calculator

Screening level concentrations for PCBs in settled dust on accessible surfaces on the Passenger Platforms were derived using the ORNL-RAIS on-line calculator for a generic indoor worker contact with soil (Appendix B). The default algorithms in the calculator assume that the indoor worker ingests 50 mg of soil every day, 250 days/year, for 25 years.

The calculator generates individual screening levels for incidental ingestion and inhalation, as well as an overall screening level which considers both routes of exposure Since the concentrations (sum of Aroclors detected) in air (volatile and particulate phases) in the Passenger Envelope of PSNY have been well characterized by empirical sample results, the screening level concentrations for incidental ingestion of soil were used to evaluate contact with settled dust.

Surfaces within the Passenger Envelope are cleaned regularly, reducing the likelihood that receptors have actual contact with settled dust in accessible areas. There is no visible dust on easily accessible high-contact surfaces. This Screening Level Evaluation is conservative and more likely overestimates, rather than underestimates actual conditions on accessible surfaces within the Passenger Envelope. Furthermore, the analytical results for concentrations of PCBs in dust came from samples of bulk dust that had accumulated on top of light fixtures over many years. The tops of the light fixtures on the Passenger Platforms are not accessible for casual hand contact, and are likely to overestimate potential exposures.

The opportunity for contact with settled dust on accessible surfaces on the Passenger Platforms is a function of how much time the receptor spends on the Platforms, and his/her activities while on the Platforms. Screening levels for incidental ingestion of PCBs in settled dust were calculated for categories of receptors who are present on the Passenger Platforms and adjusted by the number of hours per day the receptor is assumed to be on the Platform. The USEPA default soil ingestion rate for a standard 8-hour "indoor worker" (ORNL-RAIS on-line calculator) is 50 mg of soil (dust) per day. The Railroad Worker who conducts all of his/her daily activities on the Passenger Platforms most closely resembles the indoor worker envisioned by USEPA's default assumptions. For those receptors who are present on the Passenger Platforms less than 8-hours per day, the amount of dust ingested from hand contact with surfaces on the Platforms was adjusted to align with the number of hours each receptor is present in this area. A receptor who spends 4 hours per day on the Platforms (Amtrak Porters) is assumed to ingest 25 mg of dust/day; and a receptor who spends 2 hours per day on the Platforms (Housekeepers, Police and Station Security) is assumed to ingest 12.5 mg of dust/day. Passengers spend much less time on the Platforms waiting for trains, but may consume food items while waiting. Passengers are assumed to ingest 10 mg of dust/day through hand to mouth transfer. Screening level concentrations for incidental ingestion of PCBs in settled dust (mg PCB/kg dust) from surfaces on the Passenger Platforms are shown in Text Table 5 below.

Text Table 5. Screening Levels for PCBs in Settled Dust

Receptor	Screening Level PCBs (mg/kg) in Dust
Railroad Worker	33
Amtrak Porters	65
Housekeeping & Security	131
Passengers	164

3.2.2 Comparison of Concentrations of PCBs in Bulk Dust to Screening Levels

This Screening Level Evaluation uses concentrations of PCBs detected in bulk dust collected from on top of the light fixtures on the Passenger Platforms (prior to cleaning the fixtures) as a conservative surrogate for potential exposures. The dust had accumulated on the light fixtures over many years before samples were collected in 2016. Even though accessible surfaces on the Passenger Platforms are cleaned regularly, this Screening Level Evaluation conservatively assumes that settled dust is present on all surfaces on the Platforms, and that incidental ingestion of PCBs in dust is a complete pathway of exposure.

As stated previously, the conservative screening level for the Railroad Worker was calculated using the USEPA default assumptions predicated on this individual consistently ingesting 50 mg of dust (soil) containing an average concentration of 33 mg PCB/kg every day, 250 days per year for 25 years. The higher screening levels for Amtrak Porters, Housekeeping and Security, and Passengers are commensurate with the shorter amounts of time these individuals spend on the Passenger Platforms.

The mean concentration of PCBs measured in the bulk dust accumulated on the light fixtures (12.02 mg/kg) is more likely to overestimate rather than underestimate PCB concentrations in settled dust on readily accessible surfaces on the Passenger Platforms as discussed above

4.0 DISCUSSION

As of December 2017, a Remedial Investigation (RI) is in process for PSNY, however, it may be 5 years or more before the RI is completed due to the complexities of working in Penn Station. Amtrak considered it prudent to address concerns about potential exposures to PCBs for members of the public and people who work in the Station concurrently with implementing the RI. This Screening Level Evaluation of the Passenger Envelope of PSNY was based on an understanding of receptor activities and currently available empirical data documenting concentrations of PCBs in air and in samples of settled dust. The quantity and quality of analytical data were considered sufficient to support this Screening Level Evaluation in advance of completing the RI.

4.1 SOURCES OF UNCERTAINTY

Although this Screening Level Evaluation is not a formal risk assessment, the assumptions are consistent with USEPA RAGS guidance which states that a discussion of uncertainties should be included in each evaluation. Risk assessment brings together multiple sources of information to evaluate how people may come into direct physical contact with environmental media containing chemicals of potential concern (PCBs in the case of PSNY). Risk assessment uses mathematical algorithms to quantitatively estimate how much of a chemical in a medium such as soil (dust) or air a person gets into his/her body per unit time (daily dose), and the type of adverse effect the chemical might cause. However, it is not possible to know the true value or even the range of variability for the numerical factors that are used in equations to estimate dose and potential cancer risk and/or non-cancer hazard for a receptor. Thus, there are uncertainties associated with the imperfect knowledge of the true value (and/or variability) of the individual variables in the risk assessment equations.

In risk assessment, uncertainty refers to the limitations that prevent us from knowing the true exposure status of an individual receptor, and the true implications of exposure if it occurs at all. Risk assessment methods frequently compensate by using assumptions and variable values that are more likely to overestimate rather than underestimate exposure. There are many generic sources of uncertainty that are common to almost all risk assessments, such as; extrapolation of toxicity studies on laboratory animals to humans, the assumption that the concentration of a chemical in an environmental medium (e.g. PCBs in soil or dust) that a person may physically contact is represented by sample data and is consistent over time, and that the receptor gets the medium and the chemical(s) into his/her body as frequently and for as long (months, years) as the variable values in the algorithms imply.

Some of the major sources of uncertainty which are considered in this Screening Level Evaluation of receptors in the Passenger Envelope of PSNY are briefly discussed below.

Receptors: Receptor behavior patterns and activities are determinants of potential exposure. All receptors identified in this evaluation are assumed to be present in one or more areas of the

Passenger Envelope every day for 250 days per year for 25 years. It is very unlikely that any individual is consistently present in PSNY every work day for 25 years. These assumptions about the frequency and length of time a receptor is present in one or more of the three areas of the Passenger Envelope are extremely conservative and more likely to overestimate rather than underestimate potential exposures.

Although other individuals, such as children may be present in PSNY while traveling or site-seeing, they are not there frequently or for extended periods of time. Potential exposures to PCBs within the Passenger Envelope for receptors who are not frequently present are expected to be less than the chronic, long-term exposures described in this Screening Level Evaluation.

Air Monitoring Data: The air monitoring results collected from April 2016 through September 2017 are an empirical measurement of potential exposures to airborne PCBs within the Passenger Envelope. The concentrations (sum of Aroclors detected) measured in air have been variable over time and monitoring location. The inhalation pathway is well represented by the data and does not rely on modeled concentrations. The results of subsequent air monitoring will be reviewed within the context established by this Screening Level Evaluation.

Bulk Dust Data: Samples of bulk dust from on top of light fixtures on the Passenger Platforms were used to evaluate potential hand contact (and subsequent ingestion) with settled dust containing PCBs on accessible surfaces on the Passenger Platforms. It should be noted that the locations where the bulk dust samples were collected are not accessible to receptors evaluated for the Passenger Envelope, and are likely to over-estimate potential contact with accessible surface areas on the Platforms that are cleaned regularly. This approach was conservative, and is more likely to over-estimate rather than under-estimate potential exposures to PCBs in settled surface dust for the following reasons:

- Dust from on top of the light fixtures represented years of accumulation prior to recent sampling;
- Light fixtures on the Passenger Platform are being cleaned, and some may be changed in the future;
- The tops of the light fixtures, and other surfaces where dust has accumulated such as rafters, utility conduits, etc. are not accessible to receptors on the Passenger Platforms; and
- Accessible surfaces on the Passenger Platforms and other areas are cleaned regularly.

4.2 ALTERNATIVE SCREENING LEVELS FOR PCBS IN INDOOR AIR

When PCBs were initially detected in air samples from PSNY, the results were compared to USEPA Exposure Levels for Evaluation of Polychlorinated Biphenyls (PCBs) in Indoor School Air (USEPA 2012; 2017). Much of the information documenting the presence of PCBs in building materials and indoor air that inspired USEPA to develop these screening levels was based on sampling

conducted inside New York City Schools. Consequently, members of the public and regulators are familiar with the exposure levels for schools. As shown in the Text Table 6, levels for evaluating PCBs in indoor school air range from 100 ng/m^3 for very young children (daycare age) who are in the school environment for 2 years, up to 600 ng/m^3 for high school students who are in the school environment for 4 years. The KM mean concentrations of PCBs measured in air in the Upper Concourse (27 ng/m^3), the Lower Concourse (32 ng/m^3) and the Passenger Platforms (63 ng/m^3) are all lower than the USEPA level for evaluating exposures to daycare age children.

USEPA developed the exposure levels for evaluating PCBs in indoor school air using a method which considers other non-school sources of daily exposure to PCBs. The method accounts for the time children and young adult students spend inside a school building. Text Table 6 below shows the age ranges of the students, the hours per day, days per year, and number of years each age range is assumed to be present in the school building.

The concentration of PCBs in air inside the school adds to other sources of exposure such that the total daily exposure for each receptor equals the non-cancer reference dose (RfD) of 20 ng PCBs/kg body weight-day (RfD for Aroclor 1254).

The USEPA exposure levels for evaluating PCBs in indoor school air differ from the screening levels derived for this evaluation of receptors in the Passenger Envelope of PSNY with respect to the health outcome (cancer vs. non-cancer health effect) and duration of exposure (chronic vs. sub-chronic). Chronic exposures take place over a period of 7 or more years and are considered long-term exposures. Sub-chronic exposures are 6 years or less.

Text Table 6. USEPA Exposure Levels for Evaluating PCBs in Indoor School Air (2017)

Receptor Age	Days/Year	Hours/Day	Years	Exposure Level (ng/m^3)
1 to 3 Years	185	8	2	100
3 to 6 Years	180	6.5	3	200
6 to 12 Years	180	6.5	6	300
12 to 15 Years	180	6.5	3	500
15 to 19 Years	180	6.5	4	600
19+ Years	180	8	3	500

In risk assessments of workers, such as the receptors evaluated for the Passenger Envelope of PSNY, it is commonly assumed that exposures are chronic and occur over an employment period of 25 years. For receptors with chronic exposures, screening levels for PCBs (or other chemical carcinogens) are based on incremental lifetime cancer risk. The screening levels for PCBs in air in the Passenger Envelope of PSNY derived using the ORNL-RAIS on-line calculator are consistent with USEPA risk assessment methodology for evaluating chronic long-term exposures.

Discussion
January 23, 2018

The USEPA levels for evaluating exposures to PCBs in indoor school are directly relevant to the shorter time periods each age group is assumed to be present in a school environment (daycare through pre-school/kindergarten; elementary school; middle school; high school; and post-high school). PCB levels based on non-cancer health effects for the short-term sub-chronic exposures of school age children are conservative and protective of these young receptors.

5.0 SUMMARY AND CONCLUSIONS

5.1 SUMMARY

Concentrations of PCBs measured in air samples from the Passenger Platforms, Upper Concourse, and Lower Concourse of PSNY were compared to conservative screening level concentrations derived to align with the frequency with which representative categories of receptors are expected to be present in these three areas.

- The KM mean concentrations of PCBs measured in air on the Upper and Lower Concourses were lower than the screening level of 215 ng/m^3, which corresponds to an estimated 1 in 100,000 (1E-05) incremental lifetime risk of cancer for a receptor who is exposed for 8 hours a day, 250 days per year for 25 years.
- The KM mean concentration of PCBs measured in air on the Passenger Platforms was lower than the screening levels for a receptor who is present on the Platforms 8 hours per day (215 ng/m^3), a receptor who is present on the Platforms 4 hours per day (430 ng/m^3), and a receptor who is present on the Platforms 2 hours per day (858 ng/m^3).

- No receptor evaluated for the Passenger Envelope of PSNY has a daily exposure to PCBs in air that even approaches the screening level for an 8-hour day.
- The mean concentration of PCBs measured in samples of bulk dust from on top light fixtures (prior to cleaning the fixtures) on the Passenger Platforms was compared to conservative screening levels for incidental ingestion from hand to mouth transfer of settled dust on accessible surfaces. The screening level concentrations for PCBs in settled dust were derived to align with the amount of time representative categories of receptors were estimated to be present on the Passenger Platforms.
    - The mean concentration of PCBs measured in bulk dust (12.02 mg/kg) was lower than the screening level for receptors who are present on the Passenger Platforms 8 hours per day (33 mg/kg), 4 hours per day (65 mg/kg), 2 hours per day (131 mg/kg), and for passengers (164 mg/kg).

5.2 CONCLUSIONS

Concentrations of PCBs measured in air in the three areas of the PSNY Passenger Envelope have been well characterized by multiple rounds of sampling conducted from April 2016 through September 2017. The quality and quantity of air sampling results are sufficient for this Screening Level Evaluation of potential exposures to receptors who are frequently present in the Concourses and Passenger Platforms. Sample results for bulk dust collected from on top of light fixtures were used as a surrogate for potential contact with settled dust on surfaces on the Passenger Platforms.

Summary and Conclusions
January 23, 2018

The conclusions of this Screening Level Evaluation for the Passenger Envelope of PSNY are summarized below.

- The mean concentrations of PCBs detected in air on the Concourses and Passenger Platforms were lower than receptor-specific screening levels for all representative groups of individuals who are frequently present in the Passenger Envelope of PSNY.
- The mean concentration of PCBs detected in samples of bulk dust was lower than receptor-specific screening levels for hand contact with surfaces and incidental ingestion of dusts on the Passenger Platforms for all representative groups of individuals who are frequently present in this area of the Passenger Envelope.

Amtrak intends to continue periodic synoptic air monitoring. The results of each air monitoring event will be compared to the assumptions established in this Screening Level Evaluation.

6.0 REFERENCES

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FIGURES

FIGURE 1 - NJ Transit and Amtrak Schematic of the Upper Level Concourse

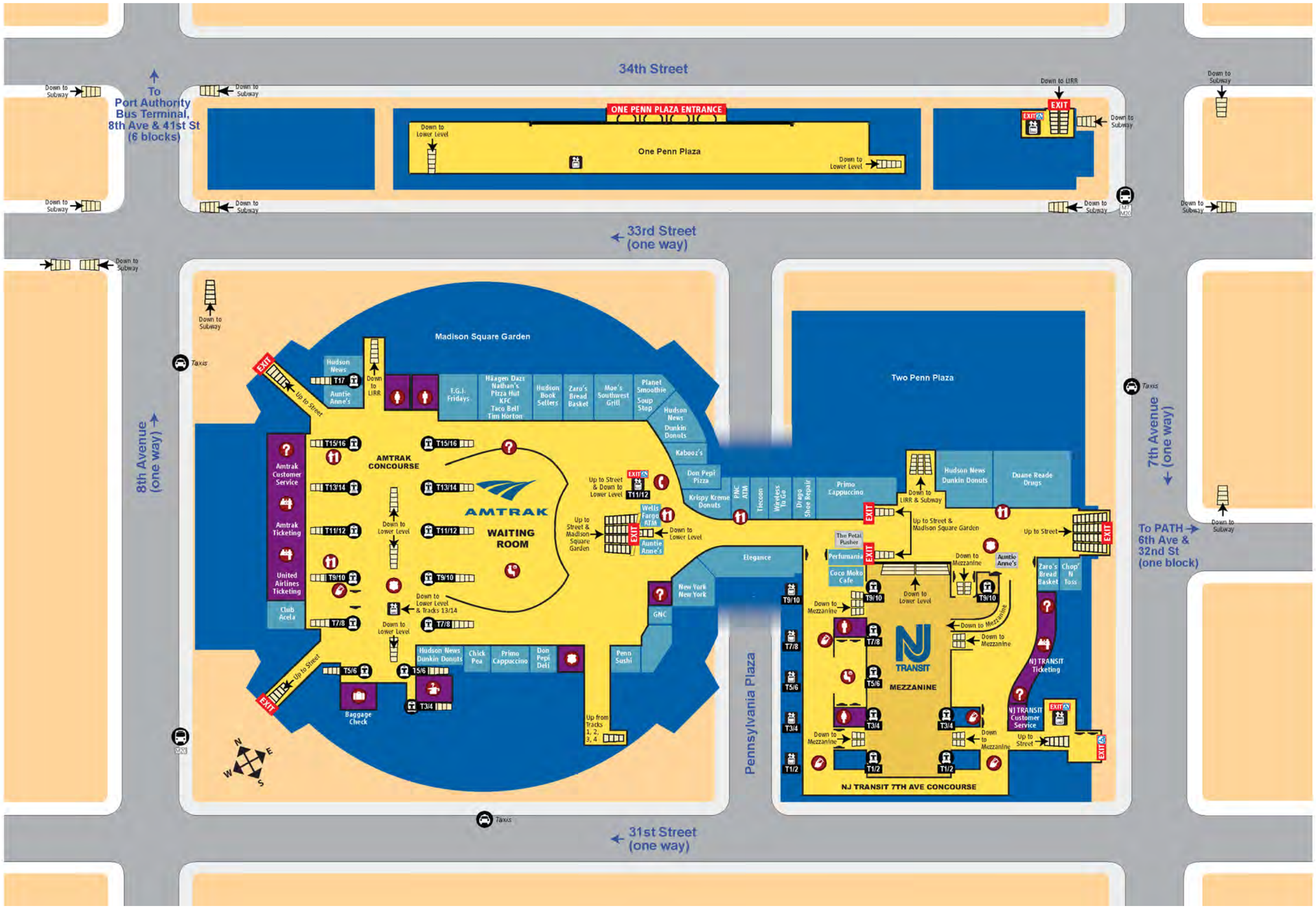


FIGURE 2 - NJ Transit and Amtrak Schematic of the Lower Level Concourse

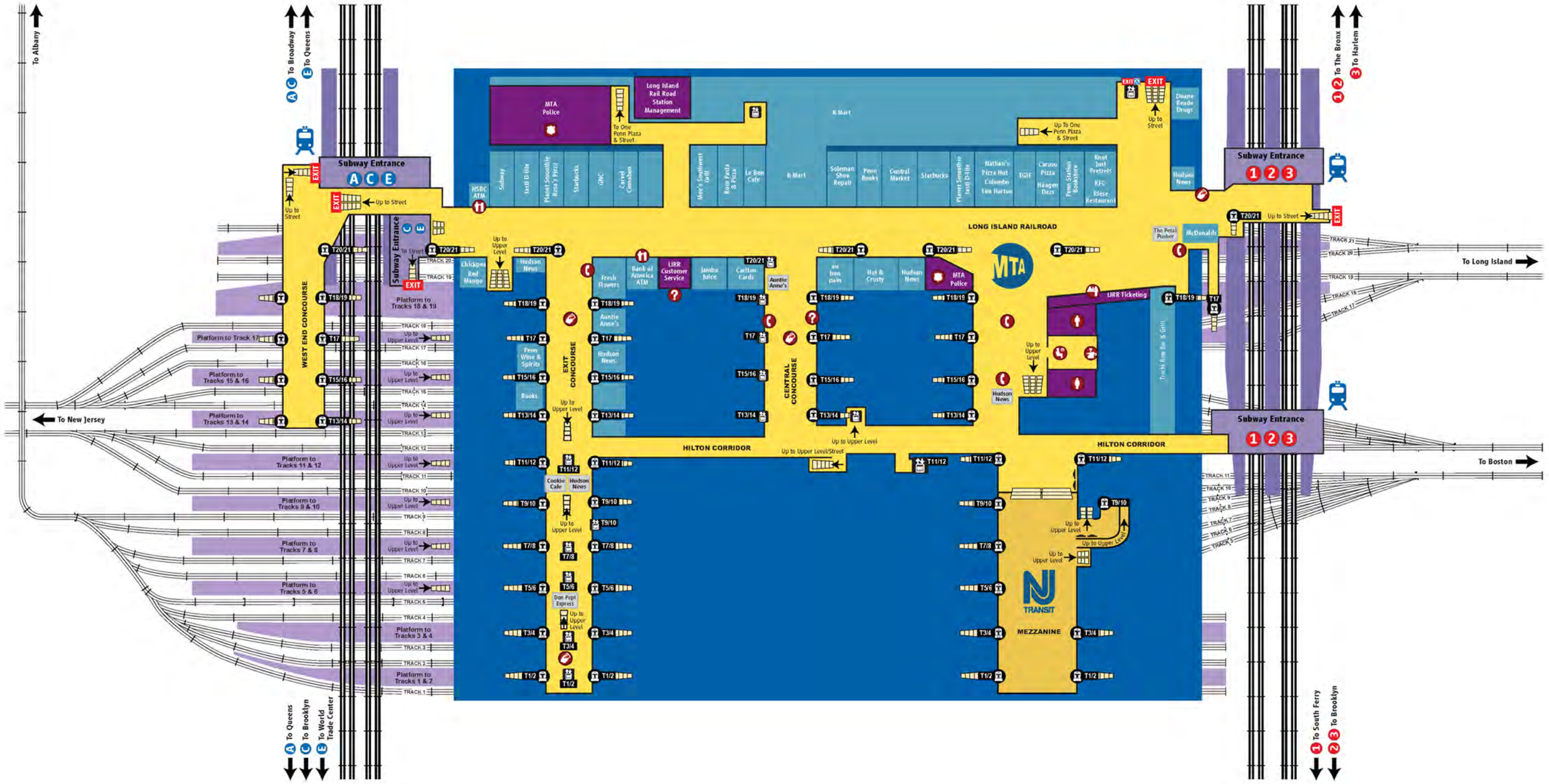
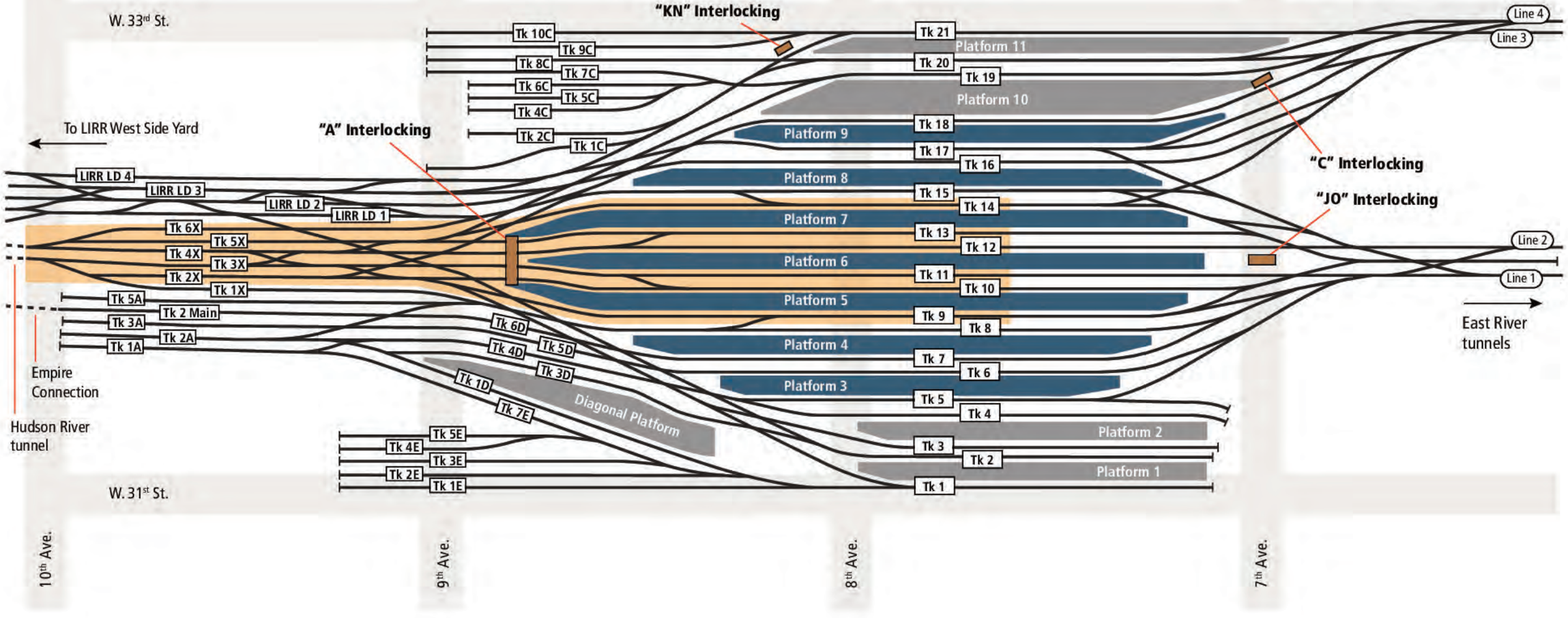


FIGURE 3 - Amtrak Penn Station Track System Schematic



TABLES

Table 1: Concentrations of PCBs Detected in Air on the Passenger Platforms										
Location	**4/21/2016**	**5/10/2016**	**5/17/2016**	**6/22/2016**	**7/13/2016**	**7/20/2016**	**9/1/2016**	**11/29/2016**	**6/22/2017**	**9/27/2017**
Platform										
P1-E	37	--	--	--	--	--	--	--	--	--
P1-W	<30.3	--	--	--	--	--	--	--	--	--
P5-E	54	--	--	--	--	--	--	--	--	--
P5-W	27	--	--	--	--	--	--	--	--	--
04-West WA-E5/Near Stair F5 - West Side of Work Area	--	33	--	--	--	--	--	--	--	--
06-East WA- Rear Decon/Near Decon - East Side of Work Area	--	66	--	--	--	--	--	--	--	--
Platform 1 - West End	--	--	92	--	--	--	184	122	--	--
Platform 2 - West Side	--	--	--	--	--	--	--	--	122	94
Platform 2 - East Side	--	--	--	--	--	--	--	--	227	107
Platform 4 - East End	--	--	114	--	--	--	151	45	106	<21
Platform 4 - West End	--	--	29	--	--	--	81	25	102	58
Platform 6 - West Side	--	--	--	--	--	--	--	--	--	<21
Platform 6 - East Side	--	--	--	--	--	--	--	--	--	31
Platform 7 - West Side	--	--	--	--	--	--	--	--	80	--
Platform 7 - East Side	--	--	--	--	--	--	--	--	152	--
Platform 8 - West Side	--	--	--	--	--	--	--	--	--	<21
Platform 8 - East Side	--	--	--	--	--	--	--	--	--	<21
Platform 9 - West End	--	--	<24	--	--	--	28	<21	22	--
Platform 9 - East End	--	--	61	--	--	--	41	<21	<21	--
Platform 10 - West End	--	--	--	--	--	--	--	--	--	<21
Platform 10 - East End	--	--	--	--	--	--	--	--	--	<21
03-Platform 1	--	--	--	87	--	--	--	--	--	--
09-Platform	--	--	--	--	71	--	--	--	--	--
11-Platform 1	--	--	--	--	--	68	--	--	--	--

"--" Data not available
"<" Reporting limit

Concentrations reported as nanograms per cubic meter (ng/m^3)

Table 2 – Concentrations of PCBs Detected in Air on the Lower Concourse				
Location	**10/19/2016**	**12/01/2017**	**06/22/2017**	**09/27/2017**
East Concourse – entrance to tracks 20/21	<24	<21	45	--
East Concourse – entrance to tracks 18/19	--	--	--	<21
East Concourse – entrance to tracks 15/16	36	26	61	30
East Concourse – entrance to tracks 11/12	--	--	--	25
Central Concourse – entrance to tracks 18/19	<25	<21	20	<21
Central Concourse – entrance to tracks 15/16	30	<21	23	<21
Exit Concourse – entrance to tracks 18/19	<25	<21	<19	<21
Exit Concourse – entrance to tracks 15/16	<24	<21	<19	<21
Exit Concourse – entrance to tracks 11/12	37	21	29	22
Exit Concourse – entrance to tracks 7/8	100	91	50	30
Exit Concourse – entrance to tracks 3/4	64	45	46	46
"--" Data not available "<" Reporting limit	Concentrations reported as nanograms per cubic meter (ng/m^3)			

Table 3 – Concentrations of PCBs Detected in Air on the Upper Concourse			
Sample location	**1/25/2017**	**6/22/2017**	**9/27/2017**
Amtrak waiting area – entrance to tracks 3/4	<22	49	--
Amtrak waiting area – entrance to tracks 7/8	<22	22	21
Amtrak waiting area – entrance to tracks 11/12	<22	<19	<21
Amtrak waiting area – entrance to tracks 15/16	--	--	<21
NJT waiting area – entrance to tracks 1/2	29	44	--
NJT waiting area – entrance to tracks 3/4	--		24
NJT waiting area – entrance to tracks 5/6	26	22	--
NJT waiting area – entrance to tracks 7/8	--	--	24
NJT waiting area – entrance to tracks 9/10	66	18	--
"--" Data not available "<" Reporting limit	Concentrations reported as nanograms per cubic meter (ng/m^3)		

APPENDIX A - PHOTOGRAPHS OF PSNY FROM PUBLICLY AVAILABLE SOURCES

Photo #1 Aerial View of Penn Station. (Photo credit: Associated Press).

Photo #2 Penn Station Entrance (Photo credit: Seagall/nydailynews.com).

Photo #3 Upper Concourse Level. (Photo credit: Amtrak).

Photo #4 Upper Concourse Level - Amtrak Waiting Area (Photo credit: Jeenah Moon/NY Times).

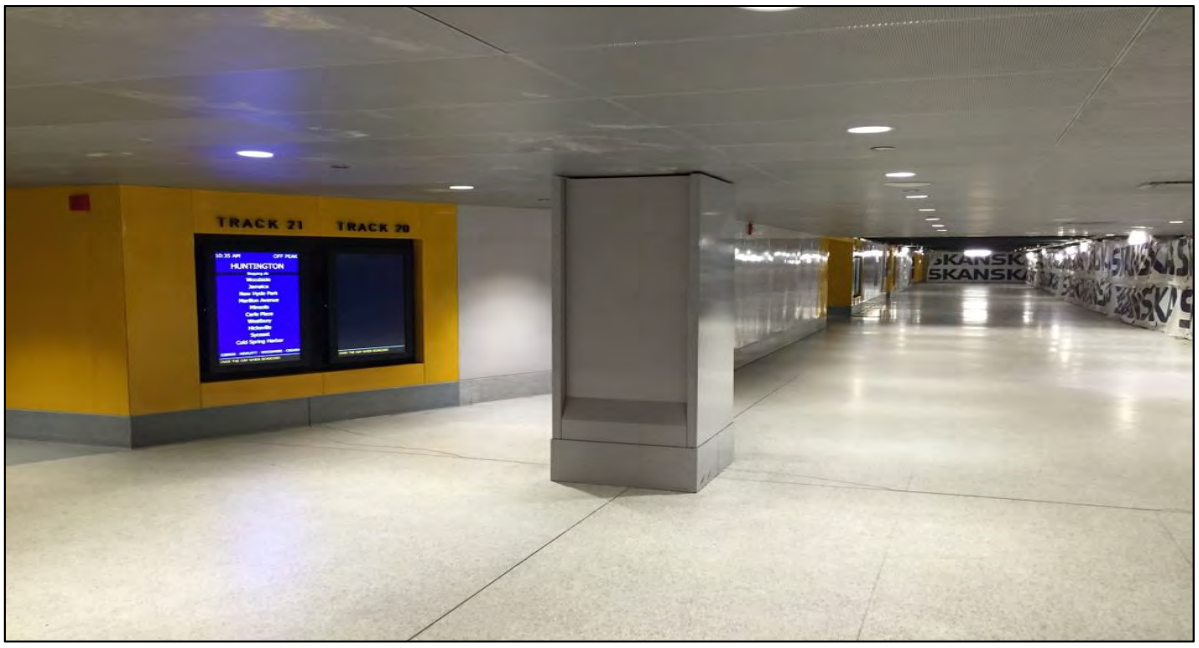


Photo #5 Lower Concourse Level. (Photo credit: Larry Higgs/nj.com)

Photo #6 Lower Concourse Level and Platform Level. (Photo credit: Larry Hags/nj.com)

Photo #7 Platform Level Track Level. (Anthony Delmundo/nydailynews.com)

Photo #8 Platform Level with Renovated Track #10. (Photo credit: Amtrak)

Photo #9 Platform Level. (Photo credit: Amtrak)

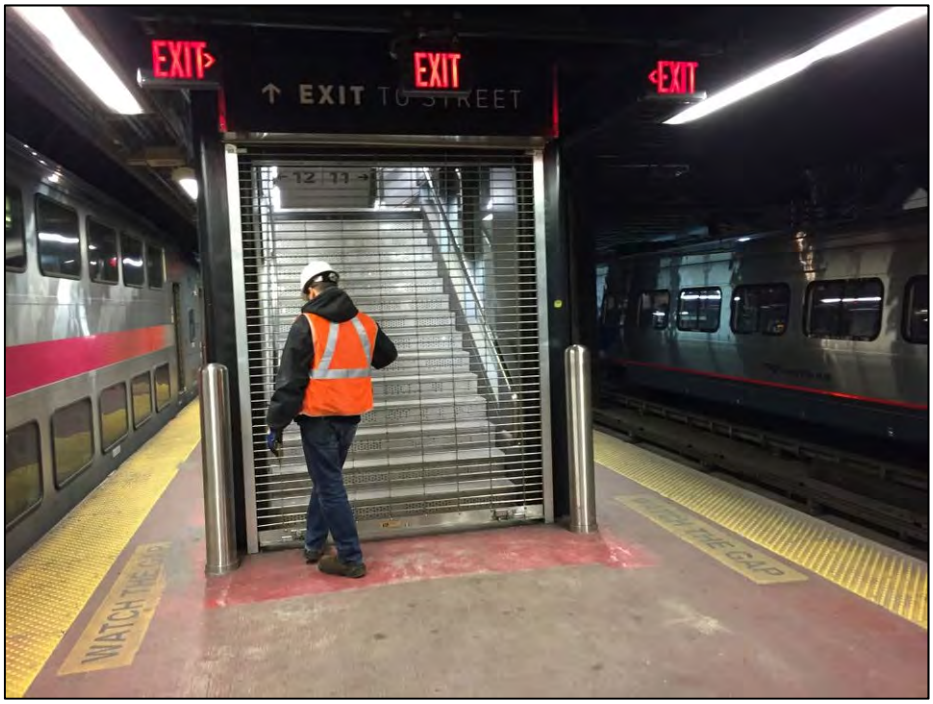


Photo #10 Platform Level. (Photo credit: Larry Higgs/nj.com)

Photo #11 Original Stairway. (Photo credit: Municipal Art Society of New York)

Photo #12 Escalator. (Photo credit: WSJ/Associated Press)

Photo #13 Elevator. (Photo credit: Karsten Moran/NY Times)

Photo #14 Passengers Waiting at Amtrak Concourse. (Photo credit: Mary Altaffer/Associated Press)

Photo #15 Vendors at Upper Level. (Photo credit: Connie Ma/animalnewyork.com)

APPENDIX B - DERIVATION OF SCREENING LEVELS FOR EXPOSURES TO PCBS IN AIR AND INGESTED DUST

Stantec

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for air PCBs: 8 Hours Exposure Time

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{inAir} (averaging time)	365
EF_{inAir} (exposure frequency) d/yr	250
ED_{inAir} (exposure duration) years	25
ET_{inAir} (exposure time) hours	8
LT (lifetime) yr	70

Output generated 27OCT2017:08:31:01

Site-Specific

Indoor Worker PRG for Ambient Air

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Carcinogenic PRG TR=1.0E-5 (ug/m^3)	Noncarcinogenic PRG HI=1 (ug/m^3)
Polychlorinated Biphenyls (high risk)	5.71E-04	I	-		2.15E-01	-

Output generated 27OCT2017:08:31:01

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for air PCBs: 4 Hours Exposure Time

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{inAir} (averaging time)	365
EF_{inAir} (exposure frequency) d/yr	250
ED_{inAir} (exposure duration) years	25
ET_{inAir} (exposure time) hours	4
LT (lifetime) yr	70

Output generated 29NOV2017:12:14:30

Site-Specific

Indoor Worker PRG for Ambient Air

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Carcinogenic PRG TR=1.0E-5 (ug/m^3)	Noncarcinogenic PRG HI=1 (ug/m^3)
Polychlorinated Biphenyls (high risk)	5.71E-04	I	-		4.29E-01	-

Output generated 29NOV2017:12:14:30

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for air PCBs: 2 Hours Exposure Time

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{inAir} (averaging time)	365
EF_{inAir} (exposure frequency) d/yr	250
ED_{inAir} (exposure duration) years	25
ET_{inAir} (exposure time) hours	2
LT (lifetime) yr	70

Output generated 17NOV2017:15:43:43

Site-Specific

Indoor Worker PRG for Ambient Air

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Carcinogenic PRG TR=1.0E-5 (ug/m^3)	Noncarcinogenic PRG HI=1 (ug/m^3)
Polychlorinated Biphenyls (high risk)	5.71E-04	I	-		8.58E-01	-

Output generated 17NOV2017:15:43:43

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for PCBs Dust Ingestion: 25 mg/day

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{iw} (averaging time)	365
EF_{iw} (exposure frequency) d/yr	250
ED_{iw} (exposure duration) yr	25
ET_{iw} (exposure time) hr	4
LT (lifetime) yr	70
BW_{iw} (body weight)	80
IR_{iw} (soil ingestion rate) mg/day	25
$City_{PEF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{wp} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	93.77
PEF (particulate emission factor) m ^{3}/kg	1359344438
A (PEF Dispersion Constant)	16.2302
B (PEF Dispersion Constant)	18.7762
C (PEF Dispersion Constant)	216.108
V (fraction of vegetative cover) unitless	0.5
U_m (mean annual wind speed) m/s	4.69
U_t (equivalent threshold value)	11.32
F(x) (function dependant on U_m/U_t) unitless	0.194
$City_{VF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	68.18
foc (fraction organic carbon in soil) g/g	0.006
p_b (dry soil bulk density) g/cm ^{3}	1.5
p_s (soil particle density) g/cm ^{3}	2.65
n (total soil porosity) L_{pore}/L_{soil}	0.43396
$_a$ (air-filled soil porosity) L_{air}/L_{soil}	0.28396
$_w$ (water-filled soil porosity) L_{water}/L_{soil}	0.15
T (exposure interval) s	819936000
A (VF Dispersion Constant)	11.911

Output generated 25SEP2017:08:30:39

Site-Specific
Indoor Worker Equation Inputs for Soil

Variable	Value
B (VF Dispersion Constant)	18.4385
C (VF Dispersion Constant)	209.7845
City$_{VF\ mass\text{-}loading}$ (Climate Zone) Selection	Default
VF_{ml} (volitization factor - mass-limit) m^3/kg	.
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m^2-s per kg/m^3	68.18
A_c (acres)	.5
T (exposure interval) yr	26
d_c (depth of source) m	.
p_b (dry soil bulk density) g/cm^3	1.5
A (VF Dispersion Constant - Mass Limit)	11.911
B (VF Dispersion Constant - Mass Limit)	18.4385
C (VF Dispersion Constant - Mass Limit)	209.7845

Output generated 25SEP2017:08:30:39

Site-Specific

Indoor Worker PRG for Soil

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Chronic RfD (mg/kg-day)	Chronic RfD Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Ingestion SF $(mg/kg\text{-}day)^{-1}$	SFO Ref	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Volatilization Factor (m^3/kg)	Particulate Emission Factor (m^3/kg)
Polychlorinated Biphenyls (high risk)	-		-		2.00E+00	I	5.71E-04	I	5.32E+05	1.36E+09

Soil Saturation Concentration (mg/kg)	Solubility (mg/L)	RBA	Ingestion PRG TR=1.0E-5 (mg/kg)	Inhalation PRG TR=1.0E-5 (mg/kg)	Carcinogenic PRG TR=1.0E-5 (mg/kg)	Ingestion PRG HQ=1 (mg/kg)	Inhalation PRG HQ=1 (mg/kg)	Noncarcinogenic PRG HI=1 (mg/kg)
-	0.7	1	6.54E+01	2.28E+02	5.08E+01	-	-	-

Output generated 25SEP2017:08:30:39

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for PCBs Dust Ingestion: 12.5 mg/day

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{iw} (averaging time)	365
EF_{iw} (exposure frequency) d/yr	250
ED_{iw} (exposure duration) yr	25
ET_{iw} (exposure time) hr	2
LT (lifetime) yr	70
BW_{iw} (body weight)	80
IR_{iw} (soil ingestion rate) mg/day	12.5
$City_{PEF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{wp} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	93.77
PEF (particulate emission factor) m ^{3}/kg	1359344438
A (PEF Dispersion Constant)	16.2302
B (PEF Dispersion Constant)	18.7762
C (PEF Dispersion Constant)	216.108
V (fraction of vegetative cover) unitless	0.5
U_m (mean annual wind speed) m/s	4.69
U_t (equivalent threshold value)	11.32
F(x) (function dependent on U_m/U_t) unitless	0.194
$City_{VF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	68.18
foc (fraction organic carbon in soil) g/g	0.006
p_b (dry soil bulk density) g/cm ^{3}	1.5
p_s (soil particle density) g/cm ^{3}	2.65
n (total soil porosity) L_{pore}/L_{soil}	0.43396
$_a$ (air-filled soil porosity) L_{air}/L_{soil}	0.28396
$_w$ (water-filled soil porosity) L_{water}/L_{soil}	0.15
T (exposure interval) s	819936000
A (VF Dispersion Constant)	11.911

Output generated 29NOV2017:14:21:10

Site-Specific
Indoor Worker Equation Inputs for Soil

Variable	Value
B (VF Dispersion Constant)	18.4385
C (VF Dispersion Constant)	209.7845
City$_{VF\ mass\text{-}loading}$ (Climate Zone) Selection	Default
VF_{ml} (volitization factor - mass-limit) m^3/kg	.
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m^2-s per kg/m^3	68.18
A_c (acres)	.5
T (exposure interval) yr	26
d_c (depth of source) m	.
p_b (dry soil bulk density) g/cm^3	1.5
A (VF Dispersion Constant - Mass Limit)	11.911
B (VF Dispersion Constant - Mass Limit)	18.4385
C (VF Dispersion Constant - Mass Limit)	209.7845

Output generated 29NOV2017:14:21:10

Site-Specific

Indoor Worker PRG for Soil

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Subchronic RfD (mg/kg-day)	Subchronic RfD Ref	Subchronic RfC (mg/m^3)	Subchronic RfC Ref	Ingestion SF $(mg/kg\text{-}day)^{-1}$	SFO Ref	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Volatilization Factor (m^3/kg)
Polychlorinated Biphenyls (high risk)	-		-		2.00E+00	I	5.71E-04	I	5.32E+05

Particulate Emission Factor (m^3/kg)	Soil Saturation Concentration (mg/kg)	Solubility (mg/L)	RBA	Ingestion PRG TR=1.0E-5 (mg/kg)	Inhalation PRG TR=1.0E-5 (mg/kg)	Carcinogenic PRG TR=1.0E-5 (mg/kg)	Ingestion PRG HQ=1 (mg/kg)	Inhalation PRG HQ=1 (mg/kg)	Noncarcinogenic PRG HI=1 (mg/kg)
1.36E+09	-	0.7	1	1.31E+02	4.56E+02	1.02E+02	-	-	-

Output generated 29NOV2017:14:21:10

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for PCBs Dust Ingestion: 10 mg/day

Variable	Value
TR (target cancer risk) unitless	1.0E-5
THQ (target hazard quotient) unitless	1
AT_{ia} (averaging time)	365
EF_{ia} (exposure frequency) d/yr	250
ED_{ia} (exposure duration) yr	25
ET_{ia} (exposure time) hr	1.5
LT (lifetime) yr	70
BW_{ia} (body weight)	80
IR_{ia} (soil ingestion rate) mg/day	10
$City_{PEF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{wp} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	93.77
PEF (particulate emission factor) m ^{3}/kg	1359344438
A (PEF Dispersion Constant)	16.2302
B (PEF Dispersion Constant)	18.7762
C (PEF Dispersion Constant)	216.108
V (fraction of vegetative cover) unitless	0.5
U_m (mean annual wind speed) m/s	4.69
U_t (equivalent threshold value)	11.32
F(x) (function dependant on U_m/U_t) unitless	0.194
$City_{VF}$ (Climate Zone) Selection	Default
A_c (acres)	.5
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) g/m ^{2}-s per kg/m ^{3}	68.18
foc (fraction organic carbon in soil) g/g	0.006
p_b (dry soil bulk density) g/cm ^{3}	1.5
p_s (soil particle density) g/cm ^{3}	2.65
n (total soil porosity) L_{pore}/L_{soil}	0.43396
$_a$ (air-filled soil porosity) L_{air}/L_{soil}	0.28396
$_w$ (water-filled soil porosity) L_{water}/L_{soil}	0.15
T (exposure interval) s	819936000
A (VF Dispersion Constant)	11.911

Output generated 24SEP2017:17:03:27

Site-Specific
Indoor Worker Equation Inputs for Soil

Variable	Value
B (VF Dispersion Constant)	18.4385
C (VF Dispersion Constant)	209.7845
City$_{VF\ mass\text{-}loading}$ (Climate Zone) Selection	Default
VF_{ml} (volitization factor - mass-limit) m^3/kg	.
Q/C_{vol} (inverse of the ratio of the geometric mean air concentration to the emission flux at the center of a square source) $g/m^2\text{-}s\ per\ kg/m^3$	68.18
A_c (acres)	.5
T (exposure interval) yr	26
d_c (depth of source) m	.
p_b (dry soil bulk density) g/cm^3	1.5
A (VF Dispersion Constant - Mass Limit)	11.911
B (VF Dispersion Constant - Mass Limit)	18.4385
C (VF Dispersion Constant - Mass Limit)	209.7845

Output generated 24SEP2017:17:03:27

Site-Specific

Indoor Worker PRG for Soil

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Chronic RfD (mg/kg-day)	Chronic RfD Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Ingestion SF $(mg/kg\text{-}day)^{-1}$	SFO Ref	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Volatilization Factor (m^3/kg)	Particulate Emission Factor (m^3/kg)
Polychlorinated Biphenyls (high risk)	-		-		2.00E+00	I	5.71E-04	I	5.32E+05	1.36E+09

Soil Saturation Concentration (mg/kg)	Solubility (mg/L)	RBA	Ingestion PRG TR=1.0E-5 (mg/kg)	Inhalation PRG TR=1.0E-5 (mg/kg)	Carcinogenic PRG TR=1.0E-5 (mg/kg)	Ingestion PRG HQ=1 (mg/kg)	Inhalation PRG HQ=1 (mg/kg)	Noncarcinogenic PRG HI=1 (mg/kg)
-	0.7	1	1.64E+02	6.08E+02	1.29E+02	-	-	-

Output generated 24SEP2017:17:03:27

Screening Level Evaluation for The Passenger Envelope, PSNY

Screening level for PCBs Dust Ingestion: 50 mg/day

Variable	Value
EW_{iw} (weeks worked - indoor worker) weeks/year	
DW_{iw} (days worked - indoor worker) days/week	
THQ (target hazard quotient) unitless	1
AT_{iw} (averaging time - indoor worker)	365
EF_{iw} (exposure frequency - indoor worker) day/year	250
ED_{iw} (exposure duration - indoor worker) year	25
ET_{iw} (exposure time - indoor worker) hour	8
LT (lifetime) year	70
BW_{iw} (body weight - indoor worker)	80
IR_{iw} (soil ingestion rate - indoor worker) mg/day	50
TR (target cancer risk) unitless	1.0E-5
$City_{PEF}$ (Climate Zone) Selection	Default
A_c (PEF acres)	0.5
Q/C_{wind} (g/m^2-s per kg/m^3)	93.77
PEF (particulate emission factor) m^3/kg	1359344438
A (PEF Dispersion Constant)	16.2302
B (PEF Dispersion Constant)	18.7762
C (PEF Dispersion Constant)	216.108
V (fraction of vegetative cover) unitless	0.5
U_m (mean annual wind speed) m/s	4.69
U_t (equivalent threshold value)	11.32
F(x) (function dependent on U_m/U_t) unitless	0.194
$City_{VF}$ (Climate Zone) Selection	Default
A_c (VF acres)	0.5
Q/C_{vol} (g/m^2-s per kg/m^3)	68.18
foc (fraction organic carbon in soil) g/g	0.006
p_b (dry soil bulk density) g/cm^3	1.5
p_s (soil particle density) g/cm^3	2.65
n (total soil porosity) L_{pore}/L_{soil}	0.43396
$_a$ (air-filled soil porosity) L_{air}/L_{soil}	0.28396
$_w$ (water-filled soil porosity) L_{water}/L_{soil}	0.15

Output generated 15DEC2017:08:35:18

Site-Specific
Indoor Worker Equation Inputs for Soil

Variable	Value
T (exposure interval) s	819936000
A (VF Dispersion Constant)	11.911
B (VF Dispersion Constant)	18.4385
C (VF Dispersion Constant)	209.7845
$City_{VF\ mass\text{-}loading}$ (Climate Zone) Selection	Default
VF_{ml} (volitization factor - mass-limit) m^3/kg	.
Q/C_{vol} (g/m^2-s per kg/m^3)	68.18
A_e (VF mass-limit acres)	0.5
T (exposure interval) yr	26
d_e (depth of source) m	.
p_b (dry soil bulk density) g/cm^3	1.5
A (VF Dispersion Constant - Mass Limit)	11.911
B (VF Dispersion Constant - Mass Limit)	18.4385
C (VF Dispersion Constant - Mass Limit)	209.7845
T_w (groundwater temperature) Celsius	

Output generated 15DEC2017:08:35:18

Site-Specific

Indoor Worker PRG for Soil

ca=Cancer, nc=Noncancer, ca* (Where nc SL < 100 x ca SL),
ca** (Where nc SL < 10 x ca SL), max=SL exceeds ceiling limit (see User's Guide), sat=SL exceeds csat,
Smax=Soil SL exceeds ceiling limit and has been substituted with the max value (see User's Guide),
Ssat=Soil inhalation SL exceeds csat and has been substituted with the csat

Chemical	Chronic RfD (mg/kg-day)	Chronic RfD Ref	Chronic RfC (mg/m^3)	Chronic RfC Ref	Ingestion SF $(mg/kg\text{-}day)^{-1}$	SFO Ref	Inhalation Unit Risk $(ug/m^3)^{-1}$	IUR Ref	Volatilization Factor (m^3/kg)	Particulate Emission Factor (m^3/kg)
Polychlorinated Biphenyls (high risk)	-		-		2.00E+00	I	5.71E-04	I	5.32E+05	1.36E+09

Soil Saturation Concentration (mg/kg)	Solubility (mg/L)	RBA	Ingestion PRG TR=1.0E-5 (mg/kg)	Inhalation PRG TR=1.0E-5 (mg/kg)	Carcinogenic PRG TR=1.0E-5 (mg/kg)	Ingestion PRG HQ=1 (mg/kg)	Inhalation PRG HQ=1 (mg/kg)	Noncarcinogenic PRG HI=1 (mg/kg)
-	0.7	1	3.27E+01	1.14E+02	2.54E+01	-	-	-

Output generated 15DEC2017:08:35:18

Appendix E

Quality Assurance Project Plan (QAPP)

MOTT MACDONALD

Appendix E - Quality Assurance Project Plan

Pennsylvania Station, New York

January 23, 2018

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Appendix E - Quality Assurance Project Plan

Pennsylvania Station, New York

January 23, 2018

QAPP Approval Record

Name	Title	Company	Date	Signature
Robert Trepp Jr.	Project Manager	Mott MacDonald	1/23/18	Robert Trepp Jr
Jennifer Kohlsaat	Quality Control Coordinator	Mott MacDonald	1/23/18	Jennifer Kohlsaat
Kevin Koch	Quality Assurance Officer	Mott MacDonald	1-23-18	Kevin E. Koch

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Issue And Revision Record

Revision	Date	Originator	Checker	Approver	Description
1.0	7/21/2017	Kevin Soldo	Robert Trepp Jr.	Kevin Koch	1st Report Submission
2.0	1/23/2018	Kevin Soldo	Robert Trepp Jr.	Kevin Koch	2nd Report Submission

1 INTRODUCTION

Amtrak is currently developing a Remedial Investigation (RI) for Pennsylvania Station, New York (PSNY) to investigate polychlorinated biphenyls (PCBs) and other potential contaminants of concern in designated Areas of Interest (AOI) at the Platform/Track Level, as well as the Upper and Lower Concourses as defined below. This Quality Assurance Project Plan (QAPP) presents the organization, objectives, functional activities, and specific quality assurance and quality control (QA/QC) activities associated with the investigative activities at PSNY. The QAPP is intended to be a companion document that will be used in conjunction with the Remedial Investigation Work Plan (RIWP) or other task-specific work plans that are developed as the project proceeds.

This site-specific QAPP confirms that specific sampling activities are planned and executed in a manner consistent with providing reproducible and reliable analytical results. This QAPP has been prepared in general accordance with the United States Environmental Protection Agency (USEPA) *Requirements for QA Project Plans* (QA-R5) dated March 2001 and provides information regarding the following:

- Project scope of work and work tasks;
- Data quality objectives (DQO) and data quality indicators (DQI);
- Project organization and responsibility;
- Sample collection and field data acquisition;
- Sample handling and chain-of-custody (COC);
- Sample analysis; and
- Data review, verification and validation.

PSNY is located in the Manhattan Borough of New York City and encompasses over 28 acres at the Platform/Track Level extending from 7th Avenue to 10th Avenue and from West 31st Street to West 33rd Street. A site location map is provided as Figure 1. PSNY is located below Madison Square Garden and Two Penn Plaza and is comprised of the following three main levels:

- Upper Concourse;
- Lower Concourse; and
- Platform/Track Level.

All levels are situated below street level as shown in Figures 2 through 4. The Upper Concourse is a primarily open area divided into two sections. Under Madison Square Garden is the Amtrak waiting area, ticketing, and customer service. These services are provided for New Jersey

Transit under Two Penn Plaza. This level additionally includes vendor areas and access to the Lower Concourse and Platform/Track Level via stairs, escalators, and elevators. This level extends from 7th Avenue to 8th Avenue and from 31st Street to 33rd Street. The Lower Concourse consists of corridors and the Metropolitan Transportation Authority (MTA) police and Long Island Rail Road (LIRR) ticketing (as well as waiting areas and additional vendor services) and customer service. This level provides access to the Platform/Track level via stairs, escalators, and elevators. This level extends from 7th Avenue to 8th Avenue and from 31st Street to 34th Street. The Platform/Track Level consists of platforms and tracks, providing access for passengers to board trains. Additionally, the Platform/Track Level also contains electrical substations, and other areas occupied by Amtrak, LIRR & New Jersey Transit workers (i.e. break room, locker room, offices etc.). This level extends from 7th Avenue to 10th Avenue and from 31st Street to 33rd Street.

Currently, there are eleven (11) platforms, twenty-one (21) tracks and four (4) train yards (A, C, D, and E). The 11 platforms combined are over 4 miles in length, and the 21 tracks combined are over 14 miles in length in an east to west direction. The face of the seven tunnels that service the station are used to define the facility limits and are excluded from the workplan.

BOP SE LLC (commonly referred to as Brookfield) owns a portion of the project area that Amtrak uses and operates. Currently BOP SE LLC is in the process of constructing the Manhattan West Southeast Tower Project which requires excavation in E yard and in track areas. Remediation of PCBs will be completed by BOP SE LLC. following a USEPA-approved Self-Implementing Cleanup Plan. The remediation details are described in a Self Implementing Cleanup Plan dated November 6, 2017 prepared by AKRF, Inc. The excavation areas are shown on Figure 4 and are excluded from this project.

This QAPP focuses on the sampling of ballast fines, ballast stone, concrete, dust, soil, air, as well as sediment in drains. The samples will be collected during periods of track outages, availability, and when access to the various AOIs can be coordinated safely with active rail operations. Such access may be limited in extent and restricted by work safety and logistics. PSNY is the busiest passenger train station in the country, with each of the station's 21 tracks in use every 2 minutes on weekdays. Consequently, there will be significant logistical challenges with scheduling and implementing sampling events. As a result, samples will not be collected in a single mobilization, rather as access becomes available. The information collected will be used to develop a RI Report for PSNY.

The primary focus of the RI is the Platform/Track Level based on the findings of evaluations conducted to date. However, investigation will occur at other levels of PSNY as outlined in the RIWP or other task-specific workplans that may be developed as the work proceeds. The station was designated into six (6) AOIs to facilitate implementation of the physical aspects of

the RI and associated technical reporting. These AOIs were designated based on current and historical operations, known conditions and accessibility, and are presented on Figure 2.

- AOI-1 corresponds to the concrete track structure and adjacent platforms. The concrete track structure includes wood ties and a concrete trough in the center of the track gauge;
- AOI-2 corresponds to A, D, and E yards located west of AOI-1;
- AOI-3 corresponds to C yard, located west of AOI-6;
- AOI-4 corresponds to track areas east of AOI-1 and AOI-6;
- AOI-5 corresponds to track areas west of AOI-1 and AOI-6 that are not included in AOI-2 and AOI-3;
- AOI-6 corresponds to Tracks 19, 20, and 21 and adjacent platforms. These tracks differ from others in that they are typical ballast stone and not concrete track construction.

2 PROJECT MANAGEMENT

2.1 Distribution List

This QAPP is being distributed to:

- Richard Mohlenhoff, Amtrak
- Craig Caldwell, Amtrak
- Nathaniel Peterson, Amtrak
- Robert Trepp Jr., Mott MacDonald
- Kevin Koch, Mott MacDonald
- Jennifer Kohlsaat, Mott MacDonald
- Kevin Herrighty, Mott MacDonald
- Marlon MacPherson, Mott MacDonald
- Eric Kolakowski, Mott MacDonald
- Jack Springston, TRC
- Frank Aceto, Stantec
- Terry Kalaghan, Stantec
- Deb Gray, Stantec
- Larry Pedersen, Clean Harbors
- Gina Hall, Alpha Analytical, Inc.
- Meghan Kelley, Con-Test Laboratory
- James S. Haklar, Ph.D., United States Environmental Protection Agency

2.2 Project Organization and Responsibility

The following table represents the organized structure for this project:

Name	Company	Phone	Responsibility
Richard Mohlenhoff	Amtrak	212-630-7249	Responsible Party Representative
Craig Caldwell	Amtrak	215-280-7908	Responsible Party Representative
Nathaniel Peterson	Amtrak	518-925-9690	Responsible Party Representative
Robert Trepp Jr.	Mott MacDonald	973-912-3347	Project Manager
Kevin Koch	Mott MacDonald	973-912-2490	Quality Assurance Officer
Jennifer Kohlsaat	Mott MacDonald	973-912-2475	Quality Control Coordinator

Name	Company	Phone	Responsibility
Kevin Herrighty	Mott MacDonald	973-912-2480	Health and Safety Coordinator
Marlon MacPherson	Mott MacDonald	973 912 3486	Data Manager
Eric Kolakowski	Mott MacDonald	973-912-2432	Field Oversight Coordinator
Jack Springston	TRC	212-221-7822	Air Testing Program Manager
Frank Aceto	Stantec	610-840-2556	Technical Advisor
Terry Kalaghan	Stantec	610-840-2542	Data Validation Program Manager
Deb Gray	Stantec	614-643-4362	Risk Assessment Program Manager
Larry Pederson	Clean Harbors, Inc. Environmental Services	732-589-4727	Field Service Project Manager
Gina Hall	Alpha Analytical, Inc.	508-898-9220	Analytical Laboratory Project Manager
Meghan Kelley	Con-Test Analytical Laboratory	413-525-2332	Analytical Laboratory Project Manager
James S. Haklar, Ph.D.	United States Environmental Protection Agency	732-906-6817	Regulatory Oversight

2.3 Background Information

PCBs have been detected in fine-grained heavily compacted material (referred to as sediment for this project) located on the concrete track structures adjacent to the platforms; the sediment has accumulated over time on the concrete track structure. Samples of the sediment were collected and submitted for PCB analysis from Tracks 1 through 18 in 2016. The majority of Tracks 19 through 21 do not contain concrete track structures (except for an eight-foot section of concrete track structure at Track 21) and thus, no samples were collected in the initial sampling program. PCBs were detected at concentrations ranging from 4.32 milligram per kilogram (mg/kg) to 80,100 mg/kg (sum of Aroclors detected) in sediment samples (USEPA

Method 8082). Following the removal of sediment, a “tar-like” material was encountered on the track structure. PCBs were detected in the samples from the “tar-like” material at concentrations ranging from 12.9 mg/kg to 39,300 mg/kg. Amtrak retained Clean Harbors, Inc. Environmental Services (Clean Harbors) to remove the sediment and “tar-like” material from the track structure for offsite disposal as a Toxic Substances Control Act (TSCA) regulated waste. Following the removal of the sediment and “tar-like” material and subsequent cleaning of the track structure, concrete chip samples were collected for analysis to document the concentrations remaining in the concrete. PCBs were detected in the concrete at concentrations ranging from not detected above the laboratory reporting limit, to 6,820 mg/kg.

Amtrak has developed an overall RI approach to delineate PCBs at PSNY. The sampling approach takes into consideration potential receptors as well as the type, duration and frequency of work tasks by Amtrak personnel. Sampling and characterization of environmental media is needed for the following:

- Ballast fines (ballast that has weathered or become worn that consist of a fine-grain material);
- Ballast stone (angular granite stones located between rail ties);
- Concrete (concrete that is present on the track structure);
- Dust (dust that has accumulated in certain track level and platform areas of PSNY);
- Soil (soil present adjacent to the track structure and other areas of PSNY);
- Air (air in the vicinity of platforms and other areas of PSNY); and
- Sediment (sediment that is present in the drainage system and remaining on the concrete track structure)

Samples are proposed to be collected according to the procedures outlined in Section 2.4 below.

2.4 Project Description

The objective of the RI is to determine the extent of impacts in different media (ballast fines, ballast stone, concrete, dust, soil, air and sediment in drains) at PSNY for the purpose of completing a RI. The primary focus of the RI will be to determine the extent of PCBs in media at PSNY. Other target compounds will be evaluated as described in the RIWP to confirm PCBs are the primary contaminant of concern at PSNY. Sampling on the Platform/Track Level and other areas of PSNY will be completed with Amtrak protection. Sampling on tracks will occur during periods of planned track outage, periods of availability, and when access to the various AOIs can be coordinated with appropriate worker protection. Sampling personnel will be accompanied by Amtrak personnel at all times during sampling. All field personnel will have completed Amtrak safety training and have a valid safety card. Sampling personnel will don

appropriate Personal Protection Equipment (PPE) as stated in Amtrak's approved Site-Specific Safety Work Plans. The sampling will generally be completed during the evening between the hours of 9PM and 5AM. Sampling also may be conducted during daytime hours, if field tasks can be performed without disruption or impact to rail operations.

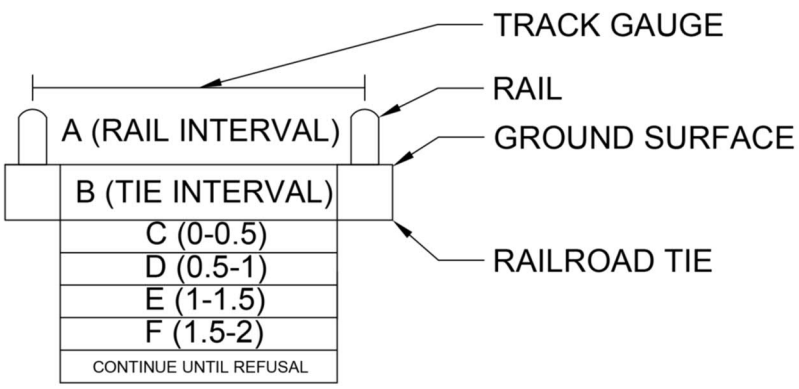
The proposed sampling and analysis for the RI is described in the RIWP dated January 23, 2018. To assist readers and users of this QAPP, information related to the collection of samples for analysis is repeated below. Standard Operating Procedures (SOPs) describing the detailed procedures for sample collection and analysis are included in Appendix A.

2.4.1 Ballast Fines Sample Collection

In areas outside of the footprint of the concrete track structure (AOI-1), the track structure is generally comprised of ballast fines and ballast stone. This includes areas in AOIs 2 through 6. Ballast fines are a fine-grained material that is comingled with the ballast stone. The percentage of ballast fines compared to ballast stone varies along the track structure and at times, ballast fines are not present.

Samples of the ballast fines will be collected from inside the center of the track gauge where the greatest potential for PCBs to be present exists. Samples will be collected via test holes installed by manual methods at a rate of one per 25 linear feet. At 25-foot intervals, material encountered within three vertical zones (rail interval, tie interval, and 0-0.5 feet below the tie) most frequently encountered by Amtrak employees will be inspected for ballast fines. For clarification purposes, the "rail interval" corresponds to, and will be defined as the vertical interval above the tie. The "tie interval" corresponds to the vertical interval from the top of the tie to the bottom of the tie. A sample will also be collected at the interval of 0-0.5 feet below the tie. See the sampling interval diagram for a visual depiction of proposed sampling depth intervals below and included as Appendix B:

Sampling Interval Diagram:



The material at each sample depth will be inspected for the presence of ballast fines to collect a sample for analysis. If a sufficient amount of ballast fines are present at the target depth interval, a sample will be collected for analysis and transferred directly into laboratory provided containers and stored on ice in a cooler. If ballast fines are not present, no ballast fines sample will be collected for analysis. Sample collection times, depths and locations will be recorded in a project dedicated field book.

In addition, at every 150 feet of linear track, samples will be collected at every six-inch depth interval below the bottom of the tie until refusal is encountered. Based on previous sampling, it is assumed that refusal will be encountered at approximately 0 to 3 feet below the bottom of the tie.

The test holes will be excavated first to a pre-determined depth (or refusal, if encountered) and then sampled from the sidewall starting with the deepest interval upwards. The test holes will be excavated using a shovel, post hole digger and/or equivalent hand tools. The hand tools will be decontaminated prior to, and in between each sampling location. Decontamination procedures are described in Section 2.5 and in a SOP included in Appendix A. Samples will be retrieved from respective intervals using dedicated pre-cleaned stainless steel scoopulas or trowels. A scoopula or trowel will be used to dress the area prior to sample collection. A separate scoopula will be used to transfer material directly into laboratory provided containers and stored on ice in a cooler. Scoopulas used to dress the area will not be used to transfer sample material. The sampling will be completed in general accordance with TSCA 761.61 subpart N. Analysis will be performed on ballast fines samples as described in Section 3.3. All sampled locations and analytical results will be mapped.

2.4.2 Ballast Stone Sample Collection

Railroad ballast stone is present in the track structure at AOIs 2 through 6. One ballast stone sample will be collected from the surface at every 6th sampling location (every 150 feet of linear track) and submitted for analysis. Ballast stone samples will be co-located with ballast fines samples and collected according to the sampling procedure outlined in Section 2.4.1. The samples will be collected from the center of the track gauge where the greatest potential for PCBs to be present exists. Sample collection times and locations will be recorded in a project dedicated field book. Care will be taken to avoid the collection of ballast fines with ballast stone samples. The sample will be crushed by the laboratory prior to analysis. Analysis will be performed on ballast stone samples as described in Section 3.3. All sampled locations and analytical results will be mapped.

2.4.3 Concrete Sample Collection

Amtrak is currently having the sediment and “tar-like” material removed from the concrete track structure in AOI-1. After the sediment and “tar-like” material have successfully been removed, the tracks are washed with diesel fuel followed by a surfactant. Oil-Dri® is then applied to complete the wash process. After the tracks have been washed, concrete samples will be collected from the concrete track structure to document the remaining concentrations of PCBs.

Concrete samples will be collected at a rate of one per 100 linear feet of track structure. The samples will be collected from the center of the trough in the middle of the track gauge where the greatest potential for PCBs to be present exists. Since the sediment and “tar-like” material are present along the majority of the concrete track structure, the 100 linear feet sampling scheme is considered to be adequate to document the remaining concentration of PCBs. If a drain is encountered in the track structure, an additional concrete sample will be collected adjacent to the drain. The sampling around drains would document any variations in the distribution of impacts compared to samples collected away from drains.

Concrete samples will be collected from the surface using a battery powered pneumatic impact hammer and chisel bit to remove an area of concrete approximately 4 by 4 inches and to a depth of approximately 1/4 inch in thickness. Chisel bits will be decontaminated prior to use according to the method described in Section 2.5 and in the SOP included as Appendix A. The resulting sample will be transferred into laboratory provided containers using a dedicated pre-cleaned stainless steel scoopula and/or trowels and stored on ice in a cooler. Sample collection times and locations will be recorded in a project dedicated field book. The sample will be crushed by the laboratory prior to analysis. Analysis will be performed on concrete samples as described in Section 3.3. All sampled locations and analytical results will be mapped.

2.4.4 Soil Sample Collection

Soil is present in a few limited areas adjacent to Track 1 (on the south side) and West of Platforms 1 and 2. Soil adjacent to tracks will be sampled from the A, B and C intervals (Appendix B) according to the procedure and frequency outlined in Section 2.4.1 (Ballast Fines Sample Collection). Samples will also be collected from deeper intervals every 150 linear feet until refusal is encountered. Soil encountered in yard areas outside the track gauge will be sampled from the A, B and C intervals (Appendix B) according to the procedure outlined in Section 2.4.1 at a rate of one per 900 square feet of area. Samples will also be collected in yard areas outside the track gauge from deeper intervals until refusal is encountered at every other location. The proposed soil sampling locations are further described in the January 23, 2018 RIWP. Sample collection times and locations will be recorded in a project dedicated field book. Analysis will be performed on soil samples as described in Section 3.3. Please note if

soil is encountered in additional areas during the RI, the proposed sampling scheme will be followed. All sampled locations and analytical results will be mapped.

2.4.5 Sediment in Drains Sample Collection

Drains are present in the center of the concrete track structure within AOI-1 as well as at other areas of the Track Level. The drains are connected to sumps and discharge to the New York City sanitary sewer system. Drains may contain sediment or fine-grained materials. As part of the RI, each drain will be inspected by removing the cover. If 12 or more inches of sediment is present, two (2) samples will be collected. One (1) sample will be collected from the top 6 inches and one (1) from the bottom 6 inches of sediment. If less than 12 inches of sediment is present within a manhole, one (1) sample will be collected from the bottom six inches of sediment. Sediment will be retrieved from the drain manholes and placed on plastic sheeting using hand tools such as post hole diggers, shovels or hand augers; whichever if more efficient. All equipment used to retrieve sediment will be decontaminated between drains as described in Section 2.5 and in the SOP included as Appendix A. A dedicated, pre-cleaned stainless steel trowel or scoopula will be utilized to transfer the sample directly into laboratory provided containers and stored on ice in a cooler. The depth, location and collection times of the samples will be recorded in the field book. Analysis will be performed on sediment samples collected from drains as described in Section 3.3. Any material removed from the drains that is not sampled will be disposed of as a TSCA waste. All sampled locations and analytical results will be mapped.

2.4.6 Dust Sample Collection

Bulk dust samples will be collected at proposed locations where a sufficient mass (Section 3.3) is present to collect a sample for analysis. At each sample location, the area will be inspected for a surface with a sufficient mass of dust to be sampled based on visual inspection. Depending on the location and area of the station surfaces may include; top of overhead light fixtures, 3rd rail cover, columns, wires or conduit and other equipment. Each bulk dust sample will be collected using dedicated, pre-cleaned stainless steel scoopulas. The dust will be transferred directly to laboratory provided containers. Containers will be placed on a scale in the field, tared and filled with bulk dust during sample collection until the required minimum mass has been collected. Once a sufficient mass of bulk dust has been collected, the containers will be sealed, labeled and stored on ice in a cooler. The location and collection times of the samples will be recorded in the field book. Analysis will be performed on bulk dust samples as described in Section 3.3. Additional detail on the proposed dust sampling locations and the rationale is provided in the RIWP dated January 23, 2018. The number and location of bulk dust samples to be collected will be determined during the RI. All sampled locations and analytical results will be mapped.

2.4.7 Air Sample Collection

Air sampling will be conducted throughout PSNY to determine potential PCB inhalation exposure levels to passengers, employees, and the general public. Sampling will be performed at Track Level, on the Lower Concourse Level, which primarily services riders of the LIRR, and on the Upper Concourse Level, which primarily services riders of Amtrak and New Jersey Transit. Air sampling will be completed as needed to complete the RI.

Analysis will be performed on air samples as described in Section 3.3. Samples will be collected by drawing a measured volume of air through a sorbent cartridge containing polyurethane foam (PUF), using a low volume vacuum pump, at a flow rate of approximately 5 liters per minute (lpm) for approximately 300 to 400 minutes and a total air volume of around 1500 to 2000 liters. The sampling trains will be calibrated at the beginning and end of the sampling period using a primary calibrator. No quartz-fiber pre-filter will be utilized, so sample analysis will include both particulate and vapor phases. Sampling will be performed at a height of approximately 5 feet. Samples will be collected between approximately 10 PM and 5 AM due to rail operations. Samples were previously collected during regular daytime hours and there is no significant difference in average PCB air concentrations compared with samples collected at night. Samples are collected at night to minimize disruptions to rail operations and to accommodate the significant logistical constraints of working at PSNY.

Platform/Track Level Sampling:

Sampling pumps will be stationed at the following ten (10) locations:

- Track Level 01 - East side of Platform 2, near stairs;
- Track Level 02 - West side of Platform 2, near stairs;
- Track Level 03 - East side of Platform 4, near stairs;
- Track Level 04 - West side of Platform 4, near stairs;
- Track Level 05 - East side of Platform 6, near stairs;
- Track Level 06 - West side of Platform 6, near stairs;
- Track Level 07 – East side of Platform 8, near stairs;
- Track Level 08 – West side of Platform 8, near stairs;
- Track Level 07 – East side of Platform 10, near stairs; and
- Track Level 08 – West side of Platform 10, near stairs;

Lower Concourse Level Air Sampling:

Sampling pumps will be stationed at the following ten (10) locations:

Exit Concourse

- Lower Level 01 - By entrance to Tracks 3/4;
- Lower Level 02 - By entrance to Tracks 7/8;
- Lower Level 03 - By entrance to Tracks 11/12;
- Lower Level 04 - By entrance to Tracks 15/16; and
- Lower Level 05 - By entrance to Tracks 18/19.

Central Concourse

- Lower Level 06 - By entrance to Tracks 15/16;
- Lower Level 07 - By entrance to Tracks 18/19;

Main Gate Area

- Lower Level 08 - By Tracks 11/12;
- Lower Level 09 - By Tracks 15/16; and
- Lower Level 10 - By Tracks 18/19.

Upper Concourse Level Air Sampling:

Sampling pumps will be stationed at the following five (5) locations:

Amtrak Concourse

- Upper Level 01 - By entrance to Tracks 7/8;
- Upper Level 02 - By entrance to Tracks 11/12; and
- Upper Level 03 - By entrance to Tracks 15/16.

New Jersey Transit Concourse

- Upper Level 04 - By entrance to Tracks 3/4; and
- Upper Level 05 - By entrance to Tracks 7/8.

Air sampling is further described in the *PCB in Air Sampling Plan* prepared by TRC dated September 5, 2017 which is included in Appendix B of the January 23, 2018 RIWP.

2.5 Decontamination Procedures

Equipment decontamination is needed to prevent the potential or likelihood of cross-contamination during sample collection. Field equipment falls into two categories consumable and reusable. Consumable field equipment is dedicated equipment that is immediately disposed of once it is used. Consumable equipment includes trowels, scoopulas and gloves used in sample collection for this project. The consumable equipment is exclusively used at a

sample location/depth and then disposed of as a TSCA hazardous waste. Consumable equipment will not be reused.

Reusable equipment consisting of hand tools, post hole digger shovels and other equipment that will be reused at multiple sampling locations. Equipment that is reused will be decontaminated between sampling locations and depths using Simple Green®. The Simple Green® is applied to the equipment and cleaned using a dedicated brush. The equipment is then rinsed with de-ionized water. All liquids used for decontamination will be collected and disposed of as TSCA hazardous waste. This method is the most feasible option as Amtrak's safety policy does not permit the use or storage of any solvents or volatile organic compounds in PSNY. The SOP which further describes the decontamination procedure is included in Appendix A.

2.6 Quality Objectives and Criteria for Measurement Data

DQOs are qualitative and quantitative statements developed to ensure that data collected are of known and appropriate quality for the purposes for which they are intended. The following DQOs have been defined for the project. If the sampling programs change, the DQOs may need to be modified or supplemented.

PCBs have been detected in sediment, "tar-like" material and concrete located on the track structures adjacent to platforms at PSNY. The results of sample analysis will be used to evaluate the site conditions for future risk assessment, feasibility, and compliance with the TSCA regulations. The sampling program objectives is to determine the nature and extent of PCB contamination.

The following DQIs will be used to evaluate DQOs: precision, accuracy, representativeness, completeness, comparability, and sensitivity.

2.7 Special Training Requirements / Certifications

2.7.1 Laboratory Certification

All analytical laboratories providing services will be certified by the New York State Department of Health.

All ballast fines, ballast stone, concrete, dust, soil, and sediment in drains samples will be labeled and logged on a COC form, stored on ice and transported under COC procedures to be analyzed by Alpha Analytical Inc. (Alpha), a New York state certified laboratory (License # 11627), located in Westborough, Massachusetts.

All air samples will be labeled and logged on a COC form, and transported under COC procedures to be analyzed by Con-Test® Analytical Laboratory (Con-Test), located in East

Longmeadow, Massachusetts, an industrial hygiene laboratory accredited by the American Industrial Hygiene Association (Lab ID 100033). Sample Handling is further described in Section 3.2.2.

2.7.2 Special Training

All aspects of the project will be completed by environmental professionals trained to complete their required duties.

Training requirements for laboratory analysis are listed in the Laboratory Quality Assurance Manuals (Appendix C).

Training requirements for field personnel are listed below:

- 40-hour OSHA HAZWOPER training (40 CFR 1910.120).
- 8-hour HAZWOPER refresher training (annually after completing the 40-hour OSHA HAZWOPER training).
- Amtrak Contractor Safety Training and all personnel must have a valid certification card in their possession at all times while on-site.

Additionally, field personnel will have an understanding of the project/task scope of work and quality assurance data needs.

2.8 Documents and Records

Project files and records will be stored at Mott MacDonald's Iselin, New Jersey office and retained as required by applicable record retention policy. Electronic versions of the project documents will be made available on the project SharePoint internet website. The documentation will be kept on file for at least 25 years after the project is completed. At that time Amtrak will take custody of the files or they will be archived at Mott MacDonald's long-term storage facility.

2.8.1 Quality Assurance Project Plan

QAPP revision, distribution and disposition will be the responsibility of the Project Manager. Updates to the QAPP will be forwarded to the individuals on the distribution list.

2.8.2 Sample Designation Documentation

Samples will be collected according to the procedures specified in Section 2.4 and the respective SOPs included as Appendix A. Samples of solid material (e.g. ballast fines, ballast stone, concrete, and sediment in drains) collected in the Track areas will be identified/numbered according to the following convention.

- AOI#-Track#-Sample Location-Interval #-Media
    - AOI# – Refers to Area of Interest 1-6
    - Track# – Refers to track number or designation
    - Sample Location – Direction followed by distance in feet from Railroad 0. E indicates to the East and W indicates to the West.
    - Interval#
        - A – rail interval
        - B – tie interval
        - C – 0–0.5 feet below the tie
        - D and so on every 0.5–foot interval until refusal
        - CS – Concrete sample from surface
        - CD – Concrete sample from below surface (note depth)
    - Media
        - A – Air
        - BF – Ballast Fines
        - BS – Ballast Stone
        - C – Concrete
        - CD – Concrete adjacent to Drain
        - D – Dust
        - S – Soil
        - SD – Sediment within Drains

As an example, a sample collected from AOI 5, on Track 10, 555 feet west of Railroad 0, at the tie interval, and of ballast stone, would be identified according to the following convention:

- AOI5–Track10–W555–B–BS

Field duplicate samples collected in the track areas will be identified by the inclusion of “DUP” after the track number. A duplicate of the above example collected on November 30, 2017 would be identified according to the following convention:

- AOI5–Track10-DUP–113017–B–BS

Field blank samples (aqueous) will be identified/numbered according to the following convention:

- AOI#–Track#–FB–date collected

As an example, a field blank collected during AOI 5, Track 10 sampling activities occurring on November 30, 2017 will be identified according to the following convention:

- AOI5–Track10–FB–113017

Samples of air or solid material (e.g. bulk dust) collected from non-track areas will be identified/numbered according to the following convention.

- Location – Area#–Sample #
    - Location – Upper or Lower Concourse, or reference closest Track or Platform (include railroad 0)
    - Area# – Specific area where sample was collected (e.g. Break Room)
    - Sample# – Numeric number

As an example, a sample collected from the Lower Concourse Level in the Amtrak Break Room would be identified according to the following convention:

- Lower Concourse Level-Amtrak Break Room–1

Field duplicate samples for air collected in the track areas will be identified by adding a D after the numeric number.

2.8.3 Field Notebooks

Field notebooks will provide the means of recording data collection activities performed. Notebooks will be assigned to field personnel and will be the responsibility of the individual person until field activities are concluded, at which time the field notebook will be returned to the project file or Project Manager.

Field notebooks will be bound with numbered pages. The project name and number will be clearly noted on the first page of the field notebook. Any pertinent information regarding the site and the sampling procedures will be documented. Entries made in these notebooks must note the date and time. Information recorded in these notebooks will include:

- Name of the individual making the entry;
- Date and time of arrival and departure at the site;
- Location of the samples taken;
- The method of collection;
- Numbers of samples taken;
- Date and time of collection;
- Sample identification number(s) including duplicate cross-reference information;
- Any field instrument calibration performed and/or instrument readings; and
- Unusual conditions.

In addition, the names of visitors to the site, subcontractors, field sampling or investigation team personnel and the purpose of the visit will also be recorded in the field team supervisor notebook.

2.8.4 Sample Location Documentation

Sample locations will be recorded by field personnel relative to fixed points located on the platforms. The fixed reference points will be benchmarks located on the platforms relative to Railroad 0. See Figure 5 for benchmark locations. Please note as the project proceeds, additional reference points may be established for locations not near platforms. These fixed reference points will be surveyed and used for identifying sample locations.

2.8.5 Laboratory Data Report Packages

Category B data deliverables (NYDEC, 2010) will be provided by the analytical laboratories for all analyses. The New York Category B is a full deliverable that generally meets USEPA requirements and New York State Analytical Services Protocol. The full deliverable format includes; sample delivery group narrative, contract lab sample information sheets, New York State Department of Environmental Conservation (NYDEC) data package summary forms, COC forms, and test analyses results including tentatively identified compounds for analysis of Volatile Organic Compounds (VOC) and Semi-Volatile Organic Compounds (SVOC). In addition, QA/QC information and documentation is also included consisting of; calibration standards, surrogate recoveries, blank results, spike recoveries, duplicate results, confirmation (lab check/QC) samples, internal standard area and retention time summary, chromatograms, raw data files, and other specific information as described in DER-10 and most current NYDEC Analytical Services Protocol (ASP).

2.8.6 Project Reports

Deliverable documents generated for the project will be maintained in the Mott MacDonald files and electronic documents will be saved to the Mott MacDonald SharePoint site. Pertinent documents generated by the project team including Mott MacDonald, Stantec, TRC, and Clean Harbors will also be saved on the Mott MacDonald SharePoint internet website. The documentation will be kept on file until the project is completed. At that time, Amtrak will take custody of the files.

3 Data Generation and Acquisition

3.1 Sampling Process Design and Method

Sampling will be conducted according to the procedures specified in the respective SOPs included as Appendix A. An overview of the Track area sampling process design is presented in Section 2.4.

3.2 Sample Handling and Custody

3.2.1 Sample Custody

Each sample will be identified using a sample label marked on the container in permanent marker containing the following information:

- Project name and number;
- Sample naming conventions are described in Section 2.8.2;
- Analysis;
- Preservative;
- Date;
- Time; and,
- Sampler's name / initials.

The objective of the COC procedure is to document the history of each sample and its handling. Custody records trace a sample from its collection through all transfers of custody until it is transferred to the laboratory. Samples are considered to be in custody if they are within sight of the individual responsible for their security or locked in a secure location. Each person who takes possession of the samples is responsible for sample integrity and safekeeping. COC procedures are provided below:

- The COC form is completed at the time of sample collection. The sample identification number, sampling location, depth, date, time, and analysis requested are recorded on the form;
- The sampling team will check the sample numbers on the individual jars against the COC form; and,
- Field samplers are responsible for the care and custody of the samples collected until the samples are transferred to another party.

All samples will be accompanied by a completed COC form. An example of COC forms is included in Appendix D. The sample identification numbers, date collected, matrix, and requested analyses will be listed on the COC form. When transferring the possession of

samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the form. This record documents transfer of custody of samples from the sampler to another person, to an off-site laboratory, or to/from a secure storage area. The original COC form will accompany the shipment, and remaining copies will be retained by the sampler and returned to the Project Manager or project file.

3.2.2 Sample Handling

The majority of sampling occurs during the overnight hours of 9 pm to 5 am. Sample handling is critical as the samples cannot be provided directly to the laboratory. The following describes the procedures for sample handling until the samples are in the custody of the analytical laboratory.

- The field sampler will transfer the samples directly into laboratory-provided containers, label each sample, and immediately place the samples in a cooler with ice. The sample time, location, and identification will be recorded in sampler's field notes and COC form.
- The samples, will then be transported in the cooler with ice by the field sampler. The sample(s) will then either remain in the cooler on ice or placed in a refrigerator in a locked and secured room. The samples will be kept at an appropriate temperature of 4 ± 2º Celcius. The completed COC will remain with the samples at all times.
- When the samples are ready to be relinquished to the analytical laboratory, the samples will be removed from the refrigerator and placed on ice in a cooler or if the samples are already in the cooler the samples will be given to the analytical laboratory. The analytical laboratory will assume custody of the samples. It should be noted that the cooler will have a custody seal on it before it is relinquished to the laboratory. Air samples collected by TRC will be placed in a cooler with ice packs and shipped via overnight courier to Con-Test.

3.3 Analytical Methods

All ballast fines, ballast stone, concrete, dust, soil, and sediment in drains samples will be analyzed by Alpha. All air samples will be analyzed by Con-Test. Analyses to be performed, frequency and minimum quantities for each media are described in the attached Table 1 *Sample Analytical Methods and Frequency*. Analytical and extraction methods, sample container volumes, sample preservations and holding times are summarized in the attached Table 2 *Sample Preservation and Holding Times*.

New York Category B data deliverables will be provided by Alpha. The analyses will be performed according to the protocols and standards described in the laboratory quality assurance manuals included in Appendix C.

3.4 Quality Control

The purpose of sample quality assurance is to document the identity of the sample and its handling from collection until delivery to the laboratory, at which point the laboratory's internal quality assurance procedures are implemented. All materials such as field and laboratory notebooks and logbooks, field and laboratory data records, correspondence, reports and COC records will be clearly labeled in accordance with Mott MacDonald's internal filing system.

Field quality control samples will consist of field duplicate samples and field blanks. Field duplicate samples (excluding air samples) will be collected at an approximate rate of one duplicate per 20 samples. The sample material will be homogenized and then divided into two glass sample jars for laboratory analysis. The second sample will be the field duplicate sample.

Field blank samples will be collected at a rate of one blank per 20 samples to assess decontamination of reusable equipment. A volume of deionized water will be poured over the equipment and collected in the appropriate sample container for laboratory analysis.

For air sampling, field quality control samples will consist of one (1) duplicate sample and two (2) field blanks with each set of samples collected per sampling period/event. Each sampling period/event will last one or more sequential days.

Field blanks will be analyzed according to the methods described in the attached Table 2 *Sample Preservation and Holding Times.*

3.5 Instrument/Equipment Testing, Inspection, Maintenance, Calibration, and Frequency

Air sampling pumps will be calibrated at the beginning and end of the sampling period using a Bios Defender 510 primary calibrator, or equivalent. Primary calibrators will be factory calibrated on a yearly basis.

Laboratory instruments will be inspected, tested, calibrated, and maintained according to the procedures specified in the Laboratory Quality Assurance Manuals included in Appendix C.

3.6 Inspection/Acceptance Requirements for Supplies and Consumables

All containers and consumables used will be dedicated and provided in sealed containers prior to use. The containers and consumables will be inspected prior to use and damaged materials will not be used.

3.7 Data Management

Data will be managed by using the Data Validation Tracking Sheet included in Stantec's Data Validation Plan dated November 30, 2017 (Appendix E). A blank copy of the tracking sheet is

included in Appendix E. Data will be archived on Mott MacDonald's project SharePoint internet website.

4 Internal Project Assessment and Oversight

A set of measures known as Internal Quality Controls (IQCs) will be implemented to ensure that DQOs are being met. These systematic measures will be implemented as internal controls to prevent issues associated with field sampling and analysis and reliability of data. Issues with quality control will be documented and corrective actions initiated to address the issues. The overall goal of the IQCs are to identify and correct errors related to the field sampling and laboratory analysis as well as maintain quality assurance. The IQC to be implemented are described below:

4.1 Internal Quality Controls

Field Staff Training: All field sampling staff will be trained by the Field Oversight Coordinator prior to staff beginning new field activities. The training will be in accordance with all relevant SOPs. Staff will also receive annual refresher training.

Laboratory Analysis Controls: Alpha will perform internal audits of laboratory processes according to their SOPs. Documentation of internal Alpha audits will be obtained and stored with the project files.

Field Staff Audits: On-site audits will be performed quarterly by the Quality Assurance Officer, Project Manager, or an assigned field inspector. This will ensure field sampling is performed following SOPs and that DQOs are met.

Sample Documentation Audits: Quality control measures for field sample collection, COC procedures and handling will be periodically checked by the project manager or designated personnel. Quality controls are further described in Section 3.4. The sample documentation audits will be completed on a quarterly basis by the Quality Assurance Coordinator, Project Manager, or designated personnel.

Data Quality Audits: Data Validation will be performed to check and verify DQOs are met. In addition, the Project Manager, Quality Assurance Officer, or designated personnel will review Data Usability Summary Reports to evaluate if the DQOs are met. Any corrective actions identified will be flagged and addressed.

SOP Review: PSNY SOPs will be reviewed by the Quality Assurance Officer or designated personnel on an annual basis or more frequently to determine if revisions are needed. Revised SOPs will be identified by their revision number and date indicated on the cover sheet.

Audits and reviews will be completed as described above. The status of the audits and reviews will be tracked and documented on a Project Assessment Tracking Form. An example form is included in Appendix F.

4.2 Corrective Actions

Corrective actions needed as determined based on audits and reviews will be implemented as necessary and reported on a Corrective Actions Form. A copy of the Corrective Action Form is included in Appendix F. Examples of corrective actions include: additional staff training, additional IQC measures and revisions to SOPs. Field activities may be rescheduled if an issue is identified that might prevent DQOs from being met. If data quality does not meet the project's specifications, data may be rejected and resampling may occur. Corrective action documentation will be saved in the project file as described in Section 2.8.

5 Data Validation and Usability

5.1 Data Review, Validation, and Verification Requirements

Laboratory data will be validated as described in the Data Validation Plan (Stantec, 2017). The Data Validation Plan is provided in Appendix E.

Mott MacDonald will review the laboratory conformance/non-conformance summary upon receipt from the laboratory. Analytical data and the summary will be reviewed and a Data Screening Checklist Form (Appendix E) will be completed as described in Stantec (2017). The following items are included as part of the checklist:

- Verify that samples were received by the laboratory and that the condition upon receipt was documented (temperature, preservation, container breakage, etc.).
- Verify that the requested analysis is performed and the dates of analysis were provided.
- Verify that the requested results were reported along with the original laboratory data qualifiers.
- Verify that the requested reporting limits were included for all samples and results at or below the reporting limits were clearly identified.
- Verify conformance with contract requirements (laboratory licensure, pricing, turnaround time, etc.).
- Other screening activities as determined necessary for preliminary use of the non-validated data.

5.2 Validation and Verification Methods

Data validation and verification will be conducted following the generalized process flow illustrated on Table 3. Data validation will generally conform to the guidelines set forth in National Functional Guidelines for Organic Superfund Methods Data Review (USEPA, 2016) and National Functional Guidelines for Inorganic Superfund Methods Data Review (USEPA, 2016b) and DER-10 / Technical Guidance for Site Investigation and Remediation (NYDEC, 2010). Data validation will be completed as summarized in the Data Validation Plan and Standard Operating Procedure (Stantec, 2017).

5.3 Usability/Reconciliation with Data Quality Objectives

Data collected will be validated in terms of DQIs described in the following sections. Data usability will be presented in the Data Usability Summary Report (DUSR) and reconciled with the project objectives. Analytical acceptance limits are summarized on Table 4.

5.3.1 Data Quality Indicators

Data collected will be validated in terms of the DQIs: precision, accuracy, representativeness, completeness, comparability, and sensitivity. The fundamental quality assurance objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve acceptance of the analytical results and thereby meet the project objectives. Taken together, the quality assurance objectives provide both quantitative and qualitative assessments of data collection and analytical procedures at the field and laboratory levels.

5.3.1.1 Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Precision is usually stated in terms of relative percent difference (RPD) or percent relative standard deviation (%RSD). Measurement of precision is dependent upon sampling technique and analytical method. Field duplicate and laboratory duplicate samples will be used to measure precision for project samples. Both sampling and analysis will be as consistent as possible. For a pair of measurements, RPD will be used in this project. For a series of measurements, %RSD will be used. The total precision of a series of measurements can be related by the additive nature of the variances. Equations for RPD and %RSD are presented below:

The RPD and %RSD will be calculated using the following equations:

$$RPD = \frac{|S-D|}{(S+D)\div 2} \times 100$$

Where,

S = First sample value (eg. Matrix Spike [MS] value)

D = Second sample value (eg. Matrix Spike Duplicate [MSD] value)

%RSD = S/x x 100%; and:

$$S = \frac{\sqrt{\sum_{I=1}^{n}(xi - x)2 \div n - 1)}}{X}$$

Where:

S = standard deviation

X_i = each observed value

X = the arithmetic mean of all observed values

n = total number of values

5.3.1.2 Accuracy

Accuracy measures the bias in a measurement system that may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are:

- Laboratory error,
- Sampling inconsistency,
- Field and/or laboratory contamination
- Matrix interference, and
- Preservation.

Field blanks, MS quality control samples, and Laboratory Control Spikes (LCSs) will be used to measure accuracy for project samples. Accuracy is calculated using the following equation:

$$\%R = (SSR - SR) \div SA \times 100$$

Where:

%R = percent recovery

SSR = spike sample result

SR = sample result

SA = amount of spike added to sample

5.3.1.3 Completeness

Completeness is defined as the percentage of data that are considered to be valid to achieve the objectives of the investigation compared to the total amount of data. Deficiencies in the data may be due to sampling techniques, poor accuracy, precision, or laboratory error. While the deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. An evaluation of completeness necessarily involves an evaluation of the impact of missing data on the ability of the project to achieve its goals. The goal for completeness is 90%. The equation used for completeness is presented below:

$$C = D \div (P \times n) \times 100$$

Where:

C = percent complete

D = number of confident quantifications

P = number of analytical parameters per sample requested for analysis

n = number of samples requested for analysis

5.3.1.4 Representativeness

An important goal of the field investigations is to collect data that are representative of conditions at the site. Samples that are considered representative are properly collected to accurately characterize the nature and extent of contamination at a general sampling location. Since the true conditions (e.g., chemical concentrations), are not known in an absolute sense, they cannot be compared to the measured values in a quantitative fashion. As such, representativeness will be measured by using standardized collection methods (e.g., sampling, handling, preserving and decontamination) and laboratory analytical methods.

5.3.1.5 Comparability

Comparability describes the extent to which valid comparisons between measurements taken at different locations and different times can be made. Like representativeness, comparability can only be ensured in a qualitative fashion. Consistency in sampling methods, measurement devices, calibration practices, and reporting limits and units will help to ensure comparability. Deviations from protocols will be noted in field records and used for data validation.

5.3.1.6 Sensitivity

Sensitivity is defined as the ability to achieve the project-required reporting limits as defined in Table 5.

5.3.2 Data Usability Summary Report

A DUSR will be prepared for each laboratory data package upon completion of the data validation process and assessment of the DQIs. The DUSR will generally conform to the guidelines set forth in NYDEC, 2010. The DUSR will include a brief summary of the sample and analytical parameters, data deficiencies, analytical protocol deviations, and quality control problems that result in qualification of the data. Actions will be taken in accordance with the Data Validation Plan and Standard Operating Procedure (Stantec, 2017). Data that is rejected will be considered not useable.

References

NYDEC, 2010, Technical Guidance for Site Investigation and Remediation (DER-10) dated May 2010.

Stantec, 2017, Data Validation Plan and Standard Operating Procedure, Amtrak Penn Station, November 30, 2017.

USEPA, 1999, Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, January 1999.

USEPA, 2001, Guidance for Quality Assurance Project Plans, EPA QA/G-5, March 2001.

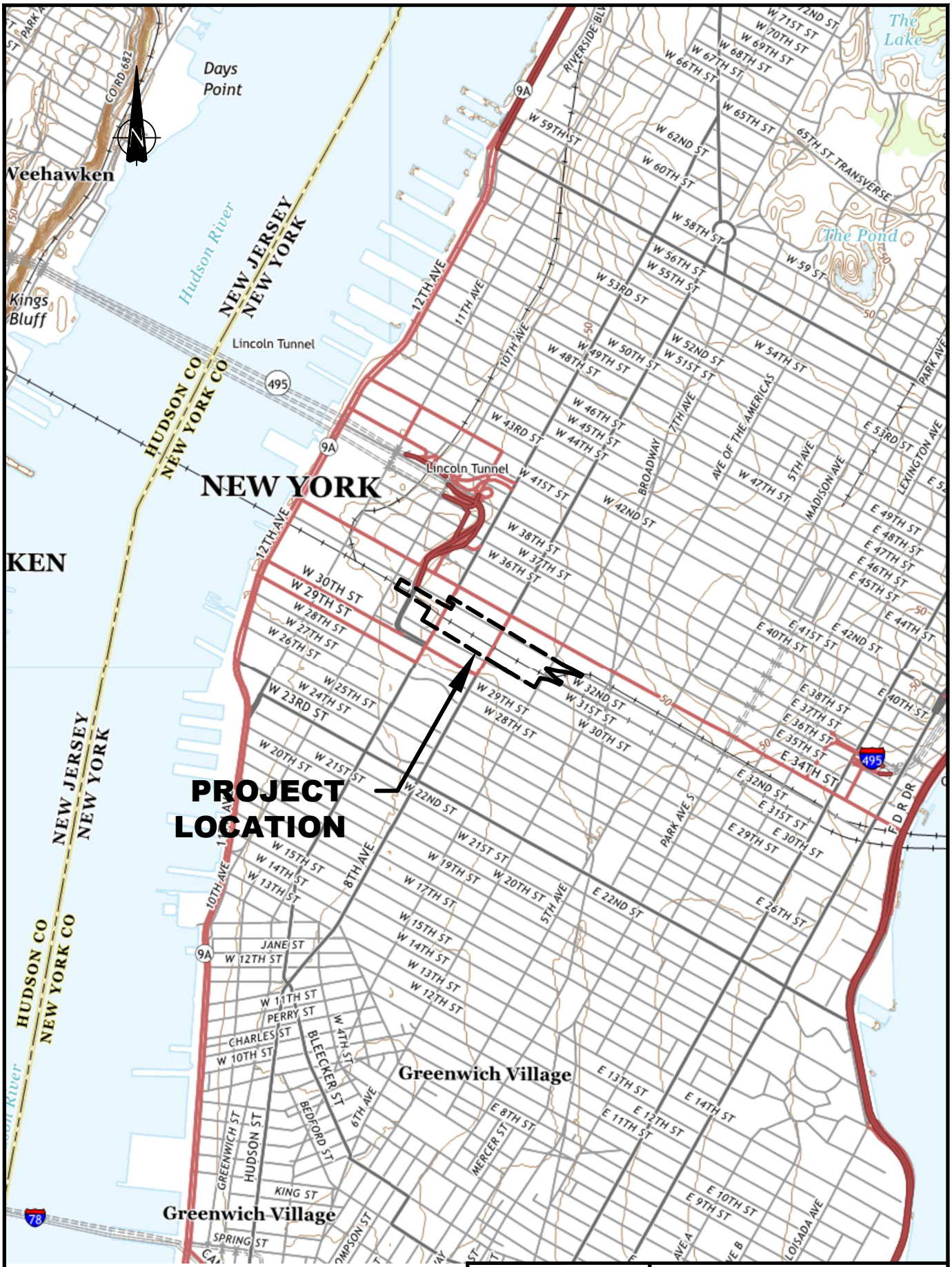
USEPA, 2015, SW846 Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Compendium, Third Edition, Update V, August 13, 2015.

USEPA, 2016, National Functional Guidelines for Organic Superfund Methods Data Review, EPA-540-R- 2016-002, September 2016.

USEPA, 2016b, National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R- 2016-001, September 2016.

mottmac.com/americas

Figures



SOURCE:
Weehawken, NJ-NY; Jersey City, NJ-NY; Brooklyn, NY; and Central Park, NY-NJ 2016 Quadrangles
USGS Topographic Quadrangles 7.5 Minute Series

2000 0 1000 2000 4000

MOTT MACDONALD

111 Wood Avenue South
Iselin, New Jersey 08830

AMTRAK
PENNSYLVANIA STATION, NEW YORK

SITE LOCATION MAP
FIGURE 1

Designed	Drawn	Checked	Approved	Date
	ACC			12/14/2017

PLOT DATE: 01/23/2018 01:11 PM
SAVE DATE: 01/23/2018 01:10 PM

34th Street

To Port Authority Bus Terminal, 8th Ave & 41st St (6 blocks)

Down to Subway

ONE PENN PLAZA ENTRANCE

Down to Lower Level

One Penn Plaza

Down to LIRR

EXIT

M7 M20

33rd Street (one way)

Madison Square Garden

Taxis

Hudson News

Auntie Anne's

Down to LIRR

T.G.I. Friday's

Häagen Dazs Nathan's Pizza Hut KFC Taco Bell Tim Horton

Hudson Book Sellers

Zaro's Bread Basket

Moe's Southwest Grill

Planet Smoothie

Soup Stop

Hudson News

Dunkin Donuts

Kabooz's

Don Pepi Pizza

Krispy Kreme Donuts

PNC ATM

Tiecoon

Wireless To Go

Drago Shoe Repair

Primo Cappuccino

Hudson News Dunkin Donuts

Duane Reade Drugs

Up to Street

Amtrak Customer Service

Amtrak Ticketing

United Airlines Ticketing

Club Acela

AMTRAK CONCOURSE

AMTRAK WAITING ROOM

Up to Street & Down to Lower Level

Up to Street & Madison Square Garden

Wells Fargo ATM

Down to Lower Level

Down to Lower Level & Tracks 13/14

Elegance

New York New York

GNC

Hudson News Dunkin Donuts

Chick Pea

Primo Cappuccino

Don Pepi Deli

Penn Sushi

Baggage Check

Up from Tracks 1, 2, 3, 4

Pennsylvania Plaza

Two Penn Plaza

The Petal Pusher

Perfumania

Coco Moko Cafe

Down to Mezzanine

Auntie Anne's

Zaro's Bread Basket

Chop' N Toss

NJ TRANSIT Ticketing

NJ TRANSIT MEZZANINE

NJ TRANSIT Customer Service

NJ TRANSIT 7TH AVE CONCOURSE

8th Avenue (one way)

7th Avenue (one way)

To PATH 6th Ave & 32nd St (one block)

31st Street (one way)

T15/16 T13/14 T11/12 T9/10 T7/8 T5/6 T3/4 T1/2

M20

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\RIWP\DWG\PENN STATION SITE DESCRIPTION.DWG

Revision | Date

Professional Engineer - N.J. Lic. No. | Date

Designed | Drawn JLM | Checked | Approved | Date 1/22/2018

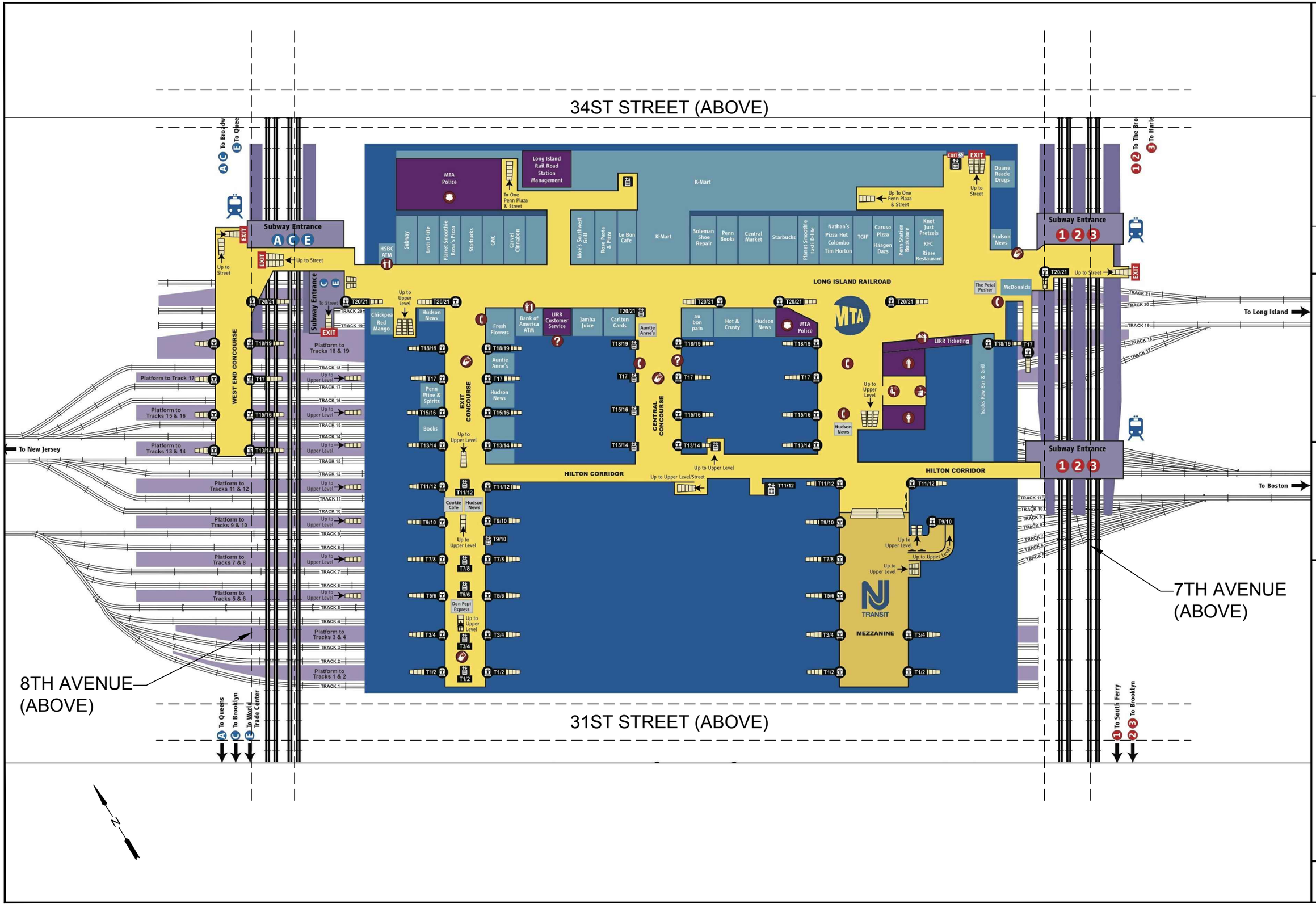
MM MOTT MACDONALD
Certificate No. 24GA28016600
111 Wood Avenue South
Iselin, New Jersey 08830-4112

AMTRAK
PENNSYLVANIA STATION, NEW YORK
SITE DESCRIPTION
UPPER CONCOURSE MAP
FIGURE 2

Job 372088 | No. | B/O | Total

PLOT DATE: 01/23/2018 01:10 PM
SAVE DATE: 01/19/2018 02:45 PM

34ST STREET (ABOVE)



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Revision	Date

Professional Engineer - N.J. Lic. No. | Date

	Date
Designed	
Drawn	JLM
Checked	
Approved	
	1/22/2018

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AMTRAK
PENNSYLVANIA STATION, NEW YORK
SITE DESCRIPTION
LOWER CONCOURSE MAP
FIGURE 3

Job	No.	
372088	B/O	Total

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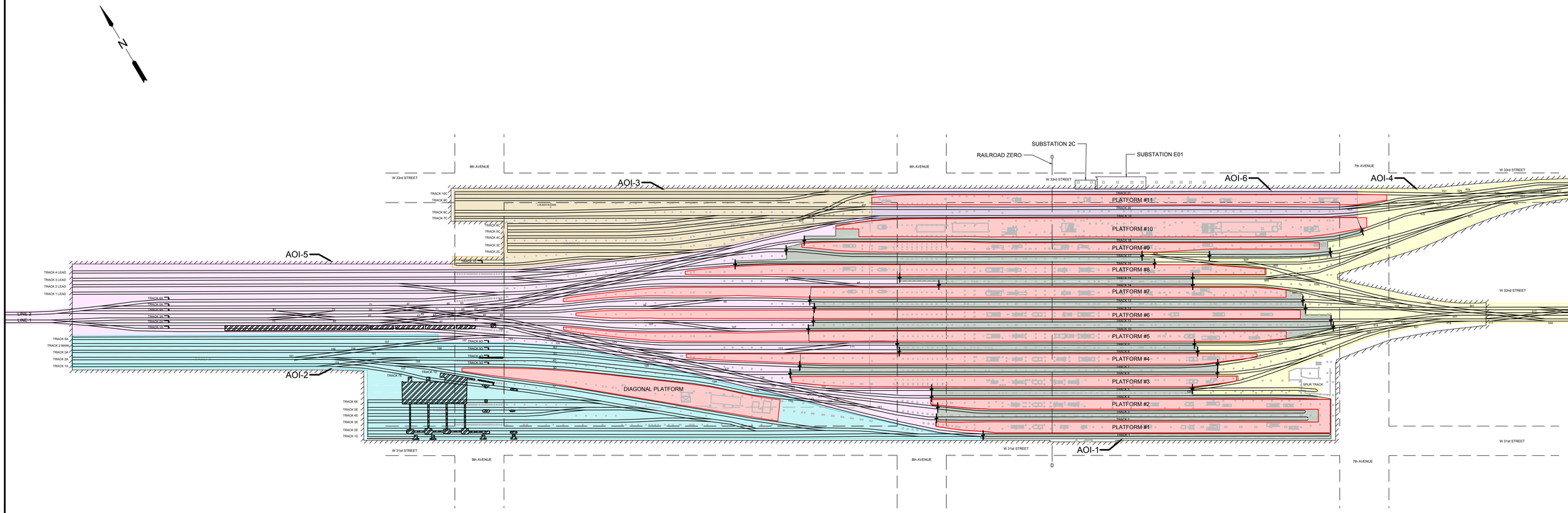
SAVE DATE: 01/23/2018 9:44 AM



NOTES:
1. TRACK LAYOUT SHOWN ON THIS MAP ARE BASED ON A DRAWING TITLED "P., N.Y. & L. I. R. R NEW YORK TERMINAL LAYOUT OF UNDERDRAINAGE AND SUBSTRUCTURES" DATED NOVEMBER 14, 1905. PLATFORM STRUCTURES, STAIRCASES, ESCALATORS, ELEVATORS, COLUMNS, AND STREETS ARE BASED ON A DRAWING TITLE "NEW YORK CITY, NY PENN STATION NEW YORK SQUARE FOOT ANALYSIS - PLATFORM LEVEL" DATED JANUARY 31, 2007. THESE HAVE BEEN MODIFIED WHERE APPROPRIATE BASED ON FIELD MEASUREMENTS. THE ORIENTATION OF TRACKS AND ALL STRUCTURES ARE FOR REFERENCE AND MAY NOT REFLECT ACTUAL CONDITIONS.

2. RAILROAD ZERO DENOTES A REFERENCE POINT AT PENNSYLVANIA STATION, NEW YORK. SAMPLE LOCATIONS WILL BE REFERENCED FROM RAILROAD ZERO.

3. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

4. THE PROPOSED EXCAVATION AREA IS EXCLUDED FROM THE PROJECT. THIS AREA WILL BE REMEDIATED BY BOP SE L.L.C. (COMMONLY REFERRED TO AS BROOK FIELD) AS DESCRIBED IN AN A SELF-IMPLEMENTING CLEANUP PLAN PROPOSED BY ARKF DATED 11/6/2017.

LEGEND:

- 89 — SWITCH NUMBER
- BALLAST/CONCRETE TRANSITION
- WALLS
- AOI 1: CONCRETE TRACK STRUCTURES
- AOI 2: A, D, AND E YARDS
- AOI 3: C YARD
- AOI 4: RUNNING TRACKS EAST OF CONCRETE TRACK STRUCTURES
- AOI 5: RUNNING TRACKS WEST OF CONCRETE TRACK STRUCTURES
- AOI 6: TRACKS 19, 20, AND 21 AT PLATFORM AREAS
- LIMITS OF PLATFORM
- EXCAVATION AREA (BOP SE LLC, OTHERWISE KNOWN AS "BROOK FIELD" EXEMPT FROM RIWP)

0 200 400

Revision	Date

Professional Engineer - N.J. Lic. No.

Date

Date	01/22/2018
Approved	
Checked	
Drawn	JLM
Designed	

MOTT MACDONALD
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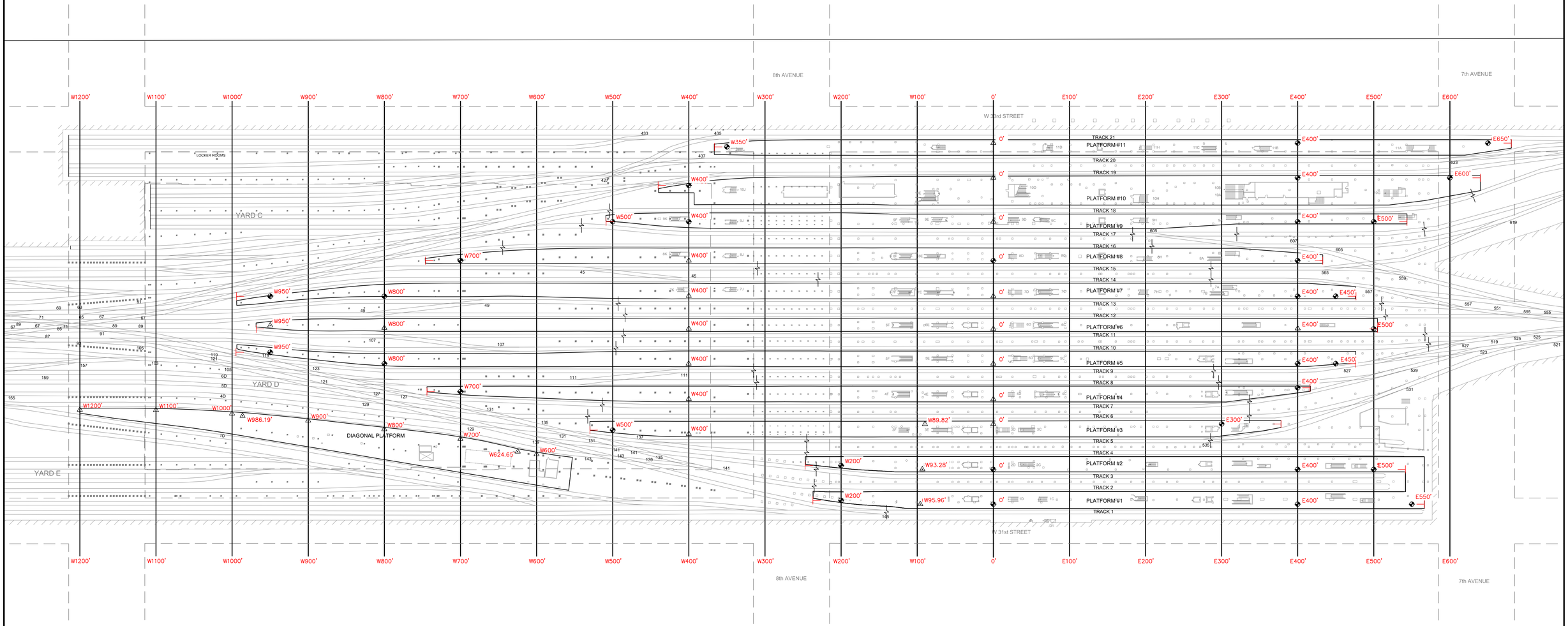
AMTRAK
PENNSYLVANIA STATION, NEW YORK
AREAS OF INTEREST/PLATFORM TRACK LEVEL MAP
FIGURE 4

Job	No.	
372088	B/O	Total

P:\372088 PENN STATION NY\I - DRAWINGS\NEW FOLDER STRUCTURE\MULTIPLE_AOIS\FULL_STATION\DWG\AOI-1-2-3-4-5-6_AREAS-OF-INTEREST-MAP.DWG



TRACK AND PLATFORM LAYOUT AS SHOWN ON THIS PLAN IS SHOWN FOR REFERENCE ONLY. BACKGROUND AND OTHER FEATURE ACCURACY CURRENTLY BEING VERIFIED BY AMTRAK ENGINEERING.

PLATFORM	WEST END STATION	EAST END STATION
11	W 366.3	E 680.6
10	W 440.6	E 639.8
9	W 508.8	E 543.5
8	W 746.2	E 432.8
7	W 994.5	E 476.5
6	W 968.5	E 504.5
5	W 995.1	E 476.2
4	W 744.2	E 416.1
3	W 530.0	E 378.0
2	W 247.3	E 541.5
1	W 237.1	E 566.3
DIAGONAL	W 1200.2	E 552.7

NOTE: END OF PLATFORMS MEASURED FROM STATION 0'

LEGEND

△ SET NAIL IN CONCRETE
MEASURED STATION AND COORDINATES

◆ SET NAIL IN CONCRETE
MEASURED STATION ONLY

⊣ MEASURED END OF PLATFORM

N

Cadd: P:\372088 Penn Station NY\I - Drawings\Master Base Drawing\Platform Station Markout Plan.dwg

Scale 1"=50' | Job 372088 | Book – | File PENN STATION
B/O – | No. – | Page –
Total –

AMTRAK

PENNSYLVANIA STATION, NEW YORK
BENCHMARK LOCATIONS

FIGURE 5

MOTT MACDONALD
Certificate No. 24GA28016600
412 Mount Kemble Avenue
Suite G22
Morristown, New Jersey 07960
Tel: 908.730.6000 Fax: 973.267.2890

Designed	Drawn	Checked	Approved	Date
–	JLM			1/22/18

Date	Revision

Tables

Table 1
Sample Analytical Methods and Frequency
Penn Station, New York

Media	Analysis Methods	Frequency	Minimum Quantity
Ballast Fines	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of deep sample locations	220 grams (32 ounces)
Ballast Stone	PCBs	All sample locations	45 grams (8 ounces)
Concrete	PCBs	All sample locations	45 grams (8 ounces)
Soil	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of deep sample locations	220 grams (32 ounces)
Sediment in Drains	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of sediment in drains sample locations	220 grams (32 ounces)
Bulk Dust	PCBs	All sample locations	45 grams (8 ounces)
	TCL+30 & TAL	20% of bulk dust sample locations	220 grams (32 ounces)
Air	PCBs	All sample locations	1500 – 2000 liters

Abbreviations:
TCL +30: Target Compound List plus a 30-compound library search
TAL: Target Analyte List

Table 2
Sample Preservation and Holding Times
Penn Station, New York

Analytical Parameter	Matrix	EPA Analytical Method	EPA Extraction Method	Sample Container / Volume	Sample Preservation	Holding Time From Collection
Metals	Soil, ballast fines/stone, or bulk dust	6010C/7471A	3050B (7471A Mercury)	Glass 60mL/2oz jar	Cool to 4°C (±2°C)	180 days (28 days mercury)
Total Metals	Water	6010C/7470A	3005A (7470A Mercury)	Plastic 500mL jar	HNO_3	180 days (28 days mercury)
VOCs	Soil, ballast fines/stone, or bulk dust	8260C	5035A	Three glass 40 mL vials	Field extraction - One 40 mL vial with MeOH & Two 40 mL vials with reagent water Cool to 4°C (±2°C)	14 days for MeOH vials (do not require freezing) 7 days for unpreserved vials
				Encore Sampler	Cool to 4°C (±2°C) if analyzed within 48 hours; > 48 hours cool to 0°C	Encore sampler must be frozen or analyzed in 48 hours; once frozen 14 day hold time.
VOCs	Water	8260C	5030C	Three glass 40mL vials	HCl	14 days
SVOCs	Soil, ballast fines/stone, or bulk dust	8270D	3540C (Soxhlet for concretes / ballast/solids) 3546 (soils)	Glass 250mL/8oz jar with PTFE lined lid	Cool to 4°C (±2°C)	14 days until extraction and 40 days after extraction
SVOCs	Water	8270D	3510C	Two 1-Liter amber jars with PTFE lined lid	Cool to 4°C (±2°C)	7 days until extraction and 40 days after extraction
PCBs (Aroclor)	Soil, ballast fines/stone, or bulk dust	8082A	3540C (Soxhlet)	250mL/8 oz. glass jar with PTFE lined lid	Cool to 4°C (±2°C)	14 days until extraction and 40 days after extraction

Table 2
Sample Preservation and Holding Times
Penn Station, New York

Analytical Parameter	Matrix	EPA Analytical Method	EPA Extraction Method	Sample Container / Volume	Sample Preservation	Holding Time From Collection
PCBs (Aroclor)	Water	8082A	3510C	Two 1-Liter amber jars with PTFE lined lid	Cool to 4°C (±2°C)	7 days until extraction and 40 days after extraction
PCBs (Aroclor)	Air	TO-10A	3540C (Soxhlet)	Sorbent polyurethane foam cartridge	Cool to 4°C (±2°C)	7 days
Pesticides	Soil, ballast fines or bulk dust	8081B	3546 (Ballast Fines, bulk dust or soil)	Glass jar 250mL/8oz with PTFE lined lid	Cool to 4°C (±2°C)	14 days until extraction and 40 days after extraction
Pesticides	Water	8081B	3510C	Two amber 500mL jar with PTFE lined lid	Cool to 4°C (±2°C)	7 days until extraction and 40 days after extraction
Cyanide	Soil, ballast fines/stone, or bulk dust	9010C/9012B	9010C/9012B	Glass 250mL/8oz jar	Cool to 4°C (±2°C)	14 days
Cyanide	Water	9010C/9012B	9010C/9012B	Plastic 250mL/8oz jar	NaOH	14 days

Notes:
C: Celsius
EPA: United States Environmental Protection Agency
HCl: Hydrochloric Acid
HNO_3: Nitric Acid
MeOH: Methanol
mL: Milliliter
NaOH: Sodium Hydroxide
oz: Ounce
PCBs: Polychlorinated Biphenyls
PTFE: Polytetrafluorethylene (Teflon)
SVOCs: Semi-Volatile Organic Compounds
VOC: Volatile Organic Compounds

Table 3
Data Validation and Reporting Process
Penn Station, New York

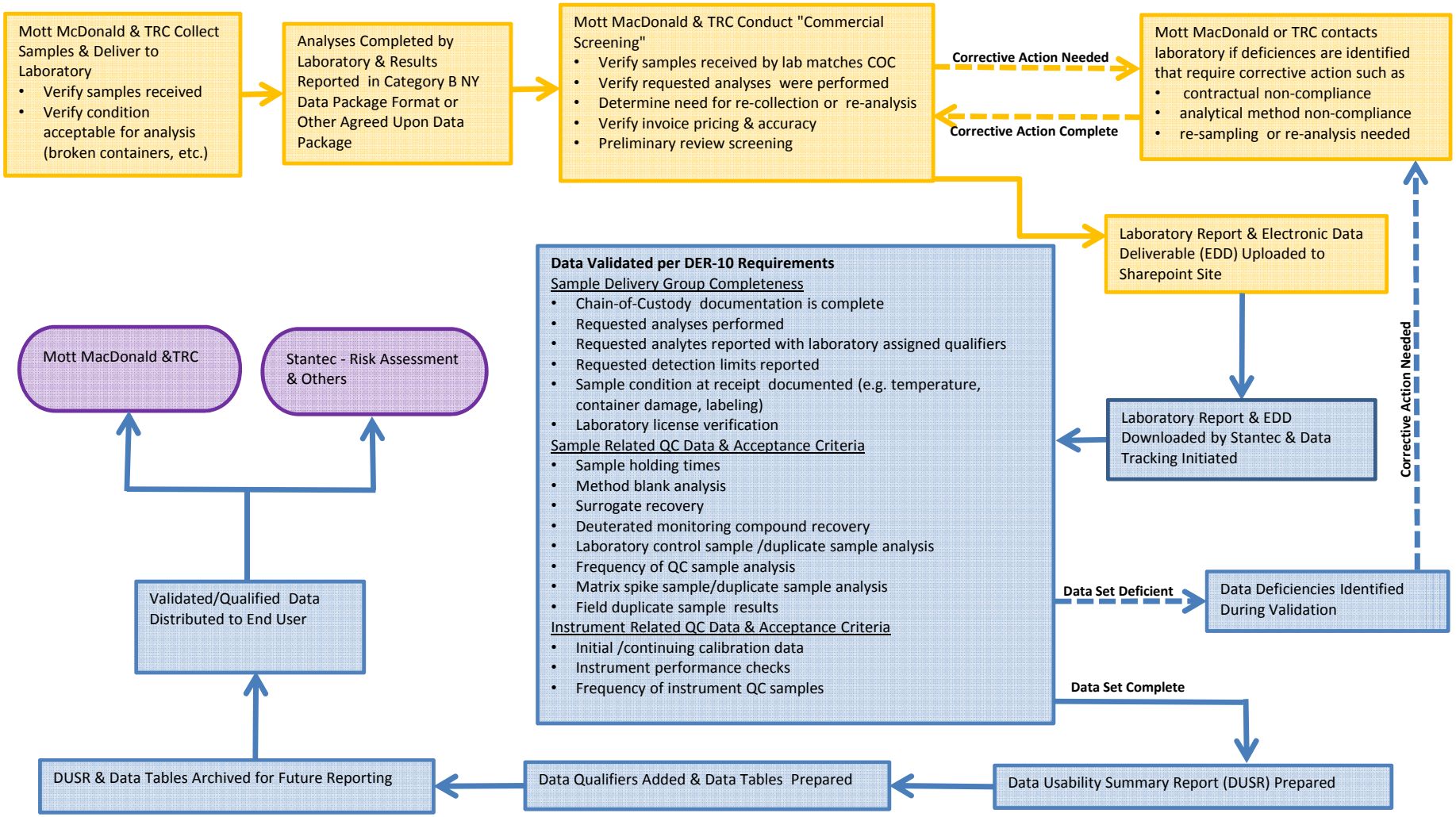


Table 4.1

Data Quality Indicator Acceptance Limits

Aroclor Polychlorinated Biphenyls

EPA Methods 8082A and TO-10A

QA/QC Parameter	EPA Method 8082A	EPA Method 8082A	EPA Method TO-10A
Matrix	Aqueous	Non-Aqueous	Air
Initial Calibration %RSD	20%	20%	20%
Continuing Calibration %D	±25% opening ±50%closing	±25% opening ±50% closing	15%
Laboratory Control/Duplicate %R	±50%	±50%	±25%
Laboratory Control/Duplicate RPD	RPD 20%	RPD 20%	RPD 15%
Matrix Spike/Spike Duplicate %R	Aroclor 1016 29-135% Aroclor 1260 29-135%	Aroclor 1016 29-135% Aroclor 1260 29-135%	Not Applicable
Matrix Spike/Spike Duplicate RPD	Aroclor 1016 RPD 15% Aroclor 1260 RPD 20%	Aroclor 1016 RPD 15% Aroclor 1260 RPD 20%	Not Applicable
Surrogate %R	30-150%	30-150%	60-120%
Difference between Columns %D	25%	25%	25%
Internal Standards %R	50-150%	50-150%	50-150%
Field Duplicate RPD	30%	50%	25%
Method Blank	< reporting limit		
Notes			
%D	Percent difference		
%R	Percent recovery		
%RSD	Percent relative standard deviation		
EPA	United States Environmental Protection Agency		
QA/QC	Quality assurance/quality control		
RPD	Relative percent difference		

Table 4.2

Data Quality Indicator Acceptance Limits

Organochlorine Pesticides

EPA Method 8081B

QA/QC Parameter	EPA Method 8081B	EPA Method 8081B
Matrix	Aqueous	Non-Aqueous
Initial Calibration %RSD	20% (25% for alpha-BHC and delta-BHC; 30% for Toxaphene)	20% (25% for alpha-BHC and delta-BHC; 30% for Toxaphene)
Continuing Calibration %D	±25%	±25%
Laboratory Control/Duplicate %R	gamma-BHC (Lindane) 50 -120% Heptachlor epoxide 50 -150% Dieldrin 30 -130% Endrin 50 -120% 4,4'-DDE 50 -150% Endosulfan sulfate 50 -120% Trans-Chlordane 50 -130%	gamma-BHC (Lindane) 50 -120% Heptachlor epoxide 50 -150% Dieldrin 30 -130% Endrin 50 -120% 4,4'-DDE 50 -150% Endosulfan sulfate 50 -120% Trans-Chlordane 50 -130%
Matrix Spike/Spike Duplicate %R	gamma-BHC (Lindane) 56 -123% Heptachlor 40 -131% Aldrin 40 -120% Dieldrin 52 -126% Endrin 56 -121% 4,4'-DDT 38 -127%	gamma-BHC (Lindane) 46 -127% Heptachlor 35 -130% Aldrin 34 -132% Dieldrin 31 -134% Endrin 42 -139% 4,4'-DDT 23 -134%
Matrix Spike/Spike Duplicate RPD	gamma-BHC (Lindane) 15% Heptachlor 20% Aldrin 22% Dieldrin 18% Endrin 21% 4,4'-DDT 27%	gamma-BHC (Lindane) 50% Heptachlor 31% Aldrin 43% Dieldrin 38% Endrin 45% 4,4'-DDT 50%
Surrogate %R	30-150%	30-150%
Difference between Columns %D	25%	25%
Blanks	< reporting limit	
Field Duplicate RPD	30%	50%
Notes		
%D	Percent difference	
%R	Percent recovery	
%RSD	Percent relative standard deviation	
EPA	United States Environmental Protection Agency	
QA/QC	Quality assurance/quality control	
RPD	Relative percent difference	

Table 4.3

Data Quality Indicator Acceptance Limits

Volatile Organic Compounds

EPA Method 8260C

QA/QC Parameter	EPA Method 8260C		
Matrix	Aqueous, Non-Aqueous		
Initial Calibration	Analyte See Table 16 of the National Functional Guidelines for Organic Methods Data Review, 2016	Max %RSD By analyte 20% - 40%	Min RRF By analyte 0.010 – 0.600
Continuing Calibration %D	Analyte See Table 16 of the National Functional Guidelines for Organic Methods Data Review, 2016	Opening Max %D By analyte ±20% - ±40%	Closing Max %D By analyte ±25% - ±50%
Matrix Spike/Spike Duplicate %R	Aqueous 1,1-Dichloroethene 61 – 145% Trichloroethene 71 – 120% Benzene 76 – 127% Toluene 76 – 125% Chlorobenzene 75 – 130%	Non-Aqueous 1,1-Dichloroethene 59 – 172% Trichloroethene 62 – 137% Benzene 66 – 142% Toluene 59 – 139% Chlorobenzene 60 – 133%	
Matrix Spike/Spike Duplicate RPD	Aqueous 1,1-Dichloroethene 14% Trichloroethene 14% Benzene 11% Toluene 13% Chlorobenzene 13%	Non-Aqueous 1,1-Dichloroethene 22% Trichloroethene 24% Benzene 21% Toluene 21% Chlorobenzene 21%	
Surrogate %R	Aqueous 1,2-Dichloroethane-d4 70 – 125% Toluene-d8 80 - 120% 4-Bromofluorobenzene 70 - 130% Dibromofluoromethane 70 - 130%	Non-Aqueous 1,2-Dichloroethane-d4 70 - 130% Toluene-d8 30 - 130% 4-Bromofluorobenzene 70 - 130% Dibromofluoromethane 70 - 130%	
Field Duplicate RPD	Aqueous 30%	Non-Aqueous 50%	
Internal Standards %R	Area Response 50-200%		
Method Blank	< reporting limit		
Notes			
%D	Percent difference		
%R	Percent recovery		
%RSD	Percent relative standard deviation		
EPA	United States Environmental Protection Agency		
QA/QC	Quality assurance/quality control		
RRF	Relative Response Factor		
RPD	Relative percent difference		

Table 4.4

Data Quality Indicator Acceptance Limits

Metals

EPA Methods 6010C and 7470A/7471A

QA/QC Parameter	EPA Method 6010C	EPA Method 6010C	EPA Method 7470A	EPA Method 7471A
Matrix	Aqueous	Non-Aqueous	Aqueous	Non-Aqueous
Initial Calibration	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL
Initial Calibration Verification %R	90 - 110%	90 - 110%	85 - 115%	85 - 115%
Continuing Calibration Verification %R	90 - 110%	90 - 110%	85 - 115%	85 - 115%
Interference Check Sample %R	80 - 120%	80 - 120%	--	--
Laboratory Control Sample %R	70 - 130% 50 - 150% Antimony and Silver	70 - 130% 50 - 150% Antimony and Silver	--	--
Laboratory Duplicate RPD	20% for results ≥ 5x QL; QL for results < 5x QL	35% for results ≥ 5x QL; QL for results < 5x QL	20% for results ≥ 5x QL; QL for results < 5x QL	35% for results ≥ 5x QL; QL for results < 5x QL
Matrix Spike %R	75 - 125%	75 - 125%	75 - 125%	75 - 125%
Serial Dilution %D	10%	15%	--	--
Blanks	< reporting limit			
Field Duplicate RPD	30%	50%	30%	50%
Notes				
%D	Percent difference			
%R	Percent recovery			
QL	Quantitation limit			
EPA	United States Environmental Protection Agency			
QA/QC	Quality assurance/quality control			
r^2	Correlation coefficient			
RPD	Relative percent difference			

Table 4.5

Data Quality Indicator Acceptance Limits

Cyanide

EPA Methods 9010C and 9012B

QA/QC Parameter	EPA Method 9012B	EPA Method 9012B	EPA Method 9010C	EPA Method 9010C
Matrix	Aqueous	Non-Aqueous	Aqueous	Non-Aqueous
Initial Calibration	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL	$r^2 \geq 0.995$ ± 30 %D y-intercept < QL
Initial Calibration Verification %R	85-115%	85-115%	85-115%	85-115%
Continuing Calibration Verification %R	85-115%	85-115%	85-115%	85-115%
Laboratory Duplicate RPD	20% for results ≥ 5x QL; QL for results < 5x QL	35% for results ≥ 5x QL; QL for results < 5x QL	20% for results ≥ 5x QL; QL for results < 5x QL	35% for results ≥ 5x QL; QL for results < 5x QL
Matrix Spike %R	75 - 125%	75 - 125%	75 - 125%	75 - 125%
Blanks	< reporting limit			
Field Duplicate RPD	30%	50%	30%	50%
Notes				
%D	Percent difference			
%R	Percent recovery			
QL	Quantitation limit			
EPA	United States Environmental Protection Agency			
QA/QC	Quality assurance/quality control			
r^2	Correlation coefficient			
RPD	Relative percent difference			

Table 4.6

Data Quality Indicator Acceptance Limits

Semi-Volatile Organic Compounds

EPA Method 8270D

QA/QC Parameter	EPA Method 8270D		
Matrix	Aqueous, Non-Aqueous		
Initial Calibration	Analyte See Table 30 of the National Functional Guidelines for Organic Methods Data Review, 2016	Max %RSD By analyte 20% - 50%	Min RRF By analyte 0.010 – 0.400
Continuing Calibration %D	Analyte See Table 30 of the National Functional Guidelines for Organic Methods Data Review, 2016	Opening Max %D By analyte 20% - 50%	Closing Max %D By analyte 25% - 50%
Matrix Spike/Spike Duplicate %R	Aqueous Phenol 12 – 110% 2-Chlorophenol 27 – 123% N-Nitroso-di-n-propylamine 41 – 116% 4-Chloro-3-methylphenol 23 – 97% Acenaphthene 46 – 118% 4-Nitrophenol 10 - 80 % 2,4-Dinitrotoluene 24 - 96 Pentachlorophenol 9 - 103 Pyrene 26 - 127	Non-Aqueous Phenol 26 - 90 2-Chlorophenol 25 - 102 N-Nitroso-di-n-propylamine 41 - 126 4-Chloro-3-methylphenol 26 - 103 Acenaphthene 31 - 137 4-Nitrophenol 11 - 114 2,4-Dinitrotoluene 28 - 89 Pentachlorophenol 17 - 109 Pyrene 35 - 142	
Matrix Spike/Spike Duplicate RPD	Aqueous Phenol 0 - 42 2-Chlorophenol 0 - 40 N-Nitroso-di-n-propylamine 0 - 38 4-Chloro-3-methylphenol 0 - 42 Acenaphthene 0 - 31 4-Nitrophenol 0 - 50 2,4-Dinitrotoluene 0 - 38 Pentachlorophenol 0 - 50 Pyrene 0 - 31	Non-Aqueous Phenol 0 - 35 2-Chlorophenol 0 - 50 N-Nitroso-di-n-propylamine 0 - 38 4-Chloro-3-methylphenol 0 - 33 Acenaphthene 0 - 19 4-Nitrophenol 0 - 50 2,4-Dinitrotoluene 0 - 47 Pentachlorophenol 0 - 47 Pyrene 0 - 36	
Surrogate %R	Aqueous 2-Fluorophenol 21-120 Phenol-d6 10 - 120 Nitrobenzene-d5 23 - 120 2-Fluorobiphenyl 15 – 120 2,4,6-Tribromophenol 10 - 120 4-Terphenyl-d14 41-149	Non-Aqueous 2-Fluorophenol 25-120 Phenol-d6 10 - 120 Nitrobenzene-d5 23 - 120 2-Fluorobiphenyl 30 – 120 2,4,6-Tribromophenol 10 - 136 4-Terphenyl-d14 18 - 120	
Field Duplicate RPD	Aqueous 30%	Non-Aqueous 50%	
Method Blank	< reporting limit		
Internal Standards %R	Area Response 50-200%		
Notes			
%D	Percent difference		
%R	Percent recovery		

Table 4.6

Data Quality Indicator Acceptance Limits

Semi-Volatile Organic Compounds

EPA Method 8270D

%RSD	Percent relative standard deviation
RRf	Relative Response Factor
RPD	Relative percent difference

Table 5.1

Laboratory Reporting Limits

Aroclor PCBs

EPA Method 8082A and TO-10A

Analyte	**CAS Number**	**Laboratory Reporting Limits**		
		EPA Method 8082A[(1)]		EPA Method TO-10A[(2)]
		Aqueous (µg/L)	Non-aqueous (µg/kg)	Air (µg/m^3)
Aroclor 1016	12674-11-2	0.083	33.5	0.022
Aroclor 1221	11104-28-2	0.083	33.5	0.022
Aroclor 1232	11141-16-5	0.083	33.5	0.022
Aroclor 1242	53469-21-9	0.083	33.5	0.022
Aroclor 1248	12672-29-6	0.083	33.5	0.022
Aroclor 1254	11097-69-1	0.083	33.5	0.022
Aroclor 1260	11096-82-5	0.083	33.5	0.022
Aroclor 1262	37324-23-5	0.083	33.5	0.022
Aroclor 1268	11100-14-4	0.083	33.5	0.022
Notes				
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference			
(2)	Con-Test Analytical Laboratory - minimum laboratory reporting limit without dilution or matrix interference			
µg/L	Micrograms per liter			
µg/kg	Micrograms per kilogram			
µg/m^3	Micrograms per cubic meter			

Table 5.2

Laboratory Reporting Limits

Pesticides

EPA Method 8081B

Analyte	CAS Number	Laboratory Reporting Limits	
		EPA Method 8081B[(1)]	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
Delta-BHC	319-86-8	0.02	7.992
Lindane	58-89-9	0.02	3.33
Alpha-BHC	319-84-6	0.02	3.33
Beta-BHC	319-85-7	0.02	7.992
Heptachlor	76-44-8	0.02	3.996
Aldrin	309-00-2	0.02	7.992
Heptachlor epoxide	1024-57-3	0.02	14.985
Endrin	72-20-8	0.04	3.33
Endrin aldehyde	7421-93-4	0.04	9.99
Endrin ketone	53494-70-5	0.04	7.992
Dieldrin	60-57-1	0.04	4.995
4,4'-DDE	72-55-9	0.04	7.992
4,4'-DDD	72-54-8	0.04	7.992
4,4'-DDT	50-29-3	0.04	14.985
Endosulfan I	959-98-8	0.02	7.992
Endosulfan II	33213-65-9	0.04	7.992
Endosulfan sulfate	1031-07-8	0.04	3.33
Methoxychlor	72-43-5	0.2	14.985
Toxaphene	8001-35-2	0.2	149.85
cis-Chlordane	5103-71-9	0.02	9.99
trans-Chlordane	5103-74-2	0.02	9.99
Chlordane	57-74-9	0.2	64.935
Notes			
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference		
µg/L	Micrograms per liter		
µg/kg	Micrograms per kilogram		

Table 5.3

Laboratory Reporting Limits

Volatile Organic Compounds

EPA Method 8260C

Analyte	CAS Number	Laboratory Reporting Limits	
		EPA Method 8260C [(1)]	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	2.5	1
1,1,1-Trichloroethane	71-55-6	2.5	1
1,1,2,2-Tetrachloroethane	79-34-5	0.5	1
1,1,2-Trichloroethane	79-00-5	1.5	1.5
1,1-Dichloroethane	75-34-3	2.5	1.5
1,1-Dichloroethene	75-35-4	0.5	1
1,1-Dichloropropene	563-58-6	2.5	5
1,2,3-Trichlorobenzene	87-61-6	2.5	5
1,2,3-Trichloropropane	96-18-4	2.5	10
1,2,4,5-Tetramethylbenzene	95-93-2	2	4
1,2,4-Trichlorobenzene	120-82-1	2.5	5
1,2,4-Trimethylbenzene	95-63-6	2.5	5
1,2-Dibromo-3-chloropropane	96-12-8	2.5	5
1,2-Dibromoethane	106-93-4	2	4
1,2-Dichlorobenzene	95-50-1	2.5	5
1,2-Dichloroethane	107-06-2	0.5	1
1,2-Dichloropropane	78-87-5	1	3.5
1,3,5-Trimethylbenzene	108-67-8	2.5	5
1,3-Dichlorobenzene	541-73-1	2.5	5
1,3-Dichloropropane	142-28-9	2.5	5
1,4-Dichlorobenzene	106-46-7	2.5	5
1,4-Diethylbenzene	105-05-5	2	4
1,4-Dioxane	123-91-1	250	40
2,2-Dichloropropane	594-20-7	2.5	5
2-Butanone	78-93-3	5	10
2-Hexanone	591-78-6	5	10
4-Ethyltoluene	622-96-8	2	4
4-Methyl-2-pentanone	108-10-1	5	10
Acetone	67-64-1	5	10
Acrylonitrile	107-13-1	5	10
Benzene	71-43-2	0.5	1
Bromobenzene	108-86-1	2.5	5
Bromochloromethane	74-97-5	2.5	5
Bromodichloromethane	75-27-4	0.5	1
Bromoform	75-25-2	2	4
Bromomethane	74-83-9	2.5	2
Carbon disulfide	75-15-0	5	10

Analyte	CAS Number	Laboratory Reporting Limits	
		EPA Method 8260C [(1)]	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
Carbon tetrachloride	56-23-5	0.5	1
Chlorobenzene	108-90-7	2.5	1
Chloroethane	75-00-3	2.5	2
Chloroform	67-66-3	2.5	1.5
Chloromethane	74-87-3	2.5	5
cis-1,2-Dichloroethene	156-59-2	2.5	1
cis-1,3-Dichloropropene	10061-01-5	0.5	1
Dibromochloromethane	124-48-1	0.5	1
Dibromomethane	74-95-3	5	10
Dichlorodifluoromethane	75-71-8	5	10
Ethyl ether	60-29-7	2.5	5
Ethylbenzene	100-41-4	2.5	1
Hexachlorobutadiene	87-68-3	2.5	5
Isopropylbenzene	98-82-8	2.5	1
Methyl tert butyl ether	1634-04-4	2.5	2
Methylene chloride	75-09-2	2.5	10
Naphthalene	91-20-3	2.5	5
n-Butylbenzene	104-51-8	2.5	1
n-Propylbenzene	103-65-1	2.5	1
o-Chlorotoluene	95-49-8	2.5	5
o-Xylene	95-47-6	2.5	2
p/m-Xylene	179601-23-1	2.5	2
p-Chlorotoluene	106-43-4	2.5	5
p-Isopropyltoluene	99-87-6	2.5	1
sec-Butylbenzene	135-98-8	2.5	1
Styrene	100-42-5	2.5	2
tert-Butylbenzene	98-06-6	2.5	5
Tetrachloroethene	127-18-4	0.5	1
Toluene	108-88-3	2.5	1.5
trans-1,2-Dichloroethene	156-60-5	2.5	1.5
trans-1,3-Dichloropropene	10061-02-6	0.5	1
trans-1,4-Dichloro-2-butene	110-57-6	2.5	5
Trichloroethene	79-01-6	0.5	1
Trichlorofluoromethane	75-69-4	2.5	5
Vinyl acetate	108-05-4	5	10
Vinyl chloride	75-01-4	1	2
Notes			
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference		
µg/L	Micrograms per liter		
µg/kg	Micrograms per kilogram		

Table 5.4

Laboratory Reporting Limits

Semi-Volatile Organic Compounds

EPA Method 8270D

Analyte	CAS Number	Laboratory Reporting Limits	
		EPA Method 8270D[(1)]	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
1,2,4-Trichlorobenzene	120-82-1	5	166.5
1,2-Dichlorobenzene	95-50-1	2	166.5
1,3-Dichlorobenzene	541-73-1	2	166.5
1,4-Dichlorobenzene	106-46-7	2	166.5
2,4,5-Trichlorophenol	95-95-4	5	166.5
2,4,6-Trichlorophenol	88-06-2	5	99.9
2,4-Dichlorophenol	120-83-2	5	149.85
2,4-Dimethylphenol	105-67-9	5	166.5
2,4-Dinitrophenol	51-28-5	20	799.2
2,4-Dinitrotoluene	121-14-2	5	166.5
2,6-Dinitrotoluene	606-20-2	5	166.5
2-Chloronaphthalene	91-58-7	2	166.5
2-Chlorophenol	95-57-8	2	166.5
2-Methylnaphthalene	91-57-6	2	199.8
2-Methylphenol	95-48-7	5	166.5
2-Nitroaniline	88-74-4	5	166.5
2-Nitrophenol	88-75-5	10	359.64
3,3'-Dichlorobenzidine	91-94-1	5	166.5
3-Methylphenol/4-Methylphenol	106-44-5	5	239.76
3-Nitroaniline	99-09-2	5	166.5
4,6-Dinitro-o-cresol	534-52-1	10	432.9
4-Bromophenyl phenyl ether	101-55-3	2	166.5
4-Chloroaniline	106-47-8	5	166.5
4-Chlorophenyl phenyl ether	7005-72-3	2	166.5
4-Nitroaniline	100-01-6	5	166.5
4-Nitrophenol	100-02-7	10	233.1
Acenaphthene	83-32-9	2	133.2
Acenaphthylene	208-96-8	2	133.2
Acetophenone	98-86-2	5	166.5
Anthracene	120-12-7	2	99.9
Benzo(a)anthracene	56-55-3	2	99.9
Benzo(a)pyrene	50-32-8	2	133.2
Benzo(b)fluoranthene	205-99-2	2	99.9
Benzo(ghi)perylene	191-24-2	2	133.2
Benzo(k)fluoranthene	207-08-9	2	99.9
Benzoic Acid	65-85-0	50	539.46
Benzyl Alcohol	100-51-6	2	166.5

Analyte	CAS Number	Laboratory Reporting Limits	
		EPA Method 8270D(1)	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
Biphenyl	92-52-4	2	379.62
Bis(2-chloroethoxy)methane	111-91-1	5	179.82
Bis(2-chloroethyl)ether	111-44-4	2	149.85
Bis(2-chloroisopropyl)ether	108-60-1	2	199.8
Bis(2-Ethylhexyl)phthalate	117-81-7	3	166.5
Butyl benzyl phthalate	85-68-7	5	166.5
Carbazole	86-74-8	2	166.5
Chrysene	218-01-9	2	99.9
Dibenzo(a,h)anthracene	53-70-3	2	99.9
Dibenzofuran	132-64-9	2	166.5
Diethyl phthalate	84-66-2	5	166.5
Dimethyl phthalate	131-11-3	5	166.5
Di-n-butylphthalate	84-74-2	5	166.5
Di-n-octylphthalate	117-84-0	5	166.5
Fluoranthene	206-44-0	2	99.9
Fluorene	86-73-7	2	166.5
Hexachlorobenzene	118-74-1	2	99.9
Hexachlorobutadiene	87-68-3	2	166.5
Hexachlorocyclopentadiene	77-47-4	20	476.19
Hexachloroethane	67-72-1	2	133.2
Indeno(1,2,3-cd)Pyrene	193-39-5	2	133.2
Isophorone	78-59-1	5	149.85
Naphthalene	91-20-3	2	166.5
Nitrobenzene	98-95-3	2	149.85
n-Nitrosodi-n-propylamine	621-64-7	5	166.5
P-Chloro-M-Cresol	59-50-7	2	166.5
Pentachlorophenol	87-86-5	10	133.2
Phenanthrene	85-01-8	2	99.9
Phenol	108-95-2	5	166.5
Pyrene	129-00-0	2	99.9
Notes			
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference		
µg/L	Micrograms per liter		
µg/kg	Micrograms per kilogram		

Table 5.5

Laboratory Reporting Limits

Metals

EPA Methods 6010A and 7471A

Analyte	**CAS Number**	**Laboratory Reporting Limits**	
		EPA Methods 6010A/7470A[(1)]	**EPA Methods 6010C/7471A[(1)]**
		Aqueous (µg/L)	**Non-aqueous (µg/kg)**
Aluminum	7429-90-5	10	4,000
Antimony	7440-36-0	4	2,000
Arsenic	7440-38-2	0.5	400
Barium	7440-39-3	0.5	400
Beryllium	7440-41-7	0.5	200
Cadmium	7440-43-9	0.2	400
Calcium	7440-70-2	100	4,000
Chromium (Total)	7440-47-3	1	400
Cobalt	7440-48-4	0.5	800
Copper	7440-50-8	1	400
Iron	7439-89-6	50	2,000
Lead	7439-92-1	1	2,000
Magnesium	7439-95-4	70	4,000
Manganese	7439-96-5	1	400
Mercury	7439-97-6	0.2	80
Nickel	7440-02-0	2	1,000
Potassium	7440-09-7	100	100,000
Selenium	7782-49-2	5	800
Silver	7440-22-4	0.4	400
Sodium	7440-23-5	100	80,000
Thallium	7440-28-0	0.5	800
Vanadium	7440-62-2	5	400
Zinc	7440-66-6	10	2,000
Notes			
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference		
µg/L	Micrograms per liter		
µg/kg	Micrograms per kilogram		

Table 5.6

Laboratory Reporting Limits

Cyanide

EPA Methods 9101C and 9012B

Analyte	**CAS Number**	**Laboratory Reporting Limits**	
		EPA Method 9010C/9012B[(1)]	
		Aqueous (µg/L)	Non-aqueous (µg/kg)
Cyanide, Total	57-12-5	0.083	172
Notes			
(1)	Alpha Analytical - minimum laboratory reporting limit without dilution or matrix interference		
µg/L	Micrograms per liter		
µg/kg	Micrograms per kilogram		

Appendices

Appendix A

Sampling and Analysis Standard Operating Procedures

Mott MacDonald Standard Operating Procedures

Standard Operating Procedures Table of Contents

1. Mott MacDonald Standard Operating Procedures

a. Decontamination of Field/Sampling Equipment

b. Sample Collection

2. Alpha Analytical Standard Operating Procedures

a. Soxhlet Extraction (Method 3540C)

b. Total Solids in Solid and Semisolid Samples (Percent Solids)

c. Particle Size Reduction Work Instruction

d. PCBs By Capillary Column Gas Chromatography

e. Total and Amendable Cyanide

f. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

g. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

h. Buchi Concentration

i. Acid Digestion of Solid Samples for Metals Analysis

j. Mercury Determination in Solids by Cold Vapor Atomic Absorption Technique (CVAA)

k. Organochlorine Pesticides

l. Inductively Coupled Plasma – Atomic Emission Spectrometry

m. Microwave Assisted Acid Digestion for Metals by 3015A/3051A for Determination by ICP

n. Microwave Extraction (EPA 3546)

3. TRC Standard Operation Procedures

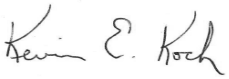
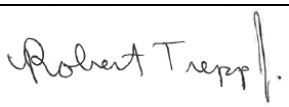
a. PCB Indoor Air Sampling

4. Contest Standard Operation Procedures

a. Compendium Method TO-4A/TO-10A by Soxhlet Extraction (Method 3540C)

MOTT MACDONALD

Standard Operating Procedure
Decontamination of Field/Sampling Equipment
Penn Station, New York
Version 1

	NAME	TITLE	SIGNATURE	DATE
Author	**Francis Pandolfo**	**Environmental Scientist**		**11/28/2017**
Reviewer	**Kevin Koch, PE, LSRP**	**Quality Assurance Coordinator**		**11/28/2017**
Authoriser	**Robert Trepp Jr., LSRP**	**Project Manager**		**11/28/2017**

Effective Date:	**12/13/2017**
Review Date:	**12/13/2017**

1. PURPOSE

- Equipment decontamination is required to prevent the potential or likelihood of cross-contamination or bias during sample collection and field operations.

2. INTRODUCTION

- Decontamination refers to the cleaning and sanitizing of field and pre-cleaning of sampling equipment.
- Field/sampling equipment falls into 2 categories;
    - Reusable equipment
        - Reusable equipment consists of; hand tools, post hole diggers, shovels, buckets, etc.
    - Consumable equipment
        - Consumable equipment consists of; scoopulas, trowels, laboratory sample bottles, latex gloves, etc.
- The following procedure serves as a guide in performing the decontamination of field/sampling equipment.

3. SCOPE

- Consumable equipment is decontaminated prior to sampling events to ensure it is clean.
- Consumable equipment is designated for use at each sample location and is immediately disposed of as TSCA hazardous waste once it is used.
    - Consumable equipment will not be reused.
    - Consumable equipment will be disposed of as TSCA waste after use.
- Reusable equipment is designated for use at multiple sample locations and requires decontamination between each use.
- Decontamination procedures must be implemented;
    - Before and after sampling events, and;
    - Between and after sample locations.

4. RESPONSIBILITIES

- Decontamination is the responsibility of all field personal.
- Equipment should not be used if it is unclear if it was decontaminated.
- Be sure all equipment and decontamination materials are accounted for and available for the sampling event.

5. LIST OF EQUIPMENT

- 5-gallon plastic buckets
- Scrub brushes
- Simple Green ®
- Plastic sheeting
- DI water

6. SPECIFIC PROCEDURE

Equipment that is reused will be deconned between sampling locations as described below;

- A decontamination station will be established in proximity to the sample location.
- The decontamination station will consist of plastic sheeting laid on the ground with two 5 -Gallon buckets;
    - The first bucket, or wash bucket, is filled with a dilution of 10:1 Simple Green ® and DI water solution for cleaning.
    - The second bucket, or rinse bucket, is filled with only DI water for rinsing.
- The equipment is placed into the wash bucket and the Simple Green ® dilution is applied using a dedicated scrub brush with firm plastic bristles.
- The equipment is then transferred to the rinse bucket and rinsed to remove any remnant surfactant.
- If the equipment passes a visual inspection after the first wash it is considered clean and ready for use.
- All liquids used for decontamination will be collected into drums and disposed of as a TSCA hazardous waste.

7. SAFETY

- Simple Green ® is a commercially available and environmentally friendly, non-toxic, and biodegradable cleaner. It is the most feasible option due as Amtrak's safety policy does not permit the storage or use of solvents or volatile organic compounds in PSNY. Please refer to the attached SDS for Simple Green ®.
- Required PPE;
    - Latex Gloves
    - Rubber boot covers
    - Safety Glasses
    - Tyvek Suit

8. DEFINITIONS

- PSNY: Penn Station New York
- SOP: Standard Operating Procedure
- Deconned: Decontaminated
- TSCA: Toxic Substances Control Act
- DI Water: Deionized water
- SDS: Safety Data Sheet
- PPE: Personal Protective Equipment

9. CHANGE HISTORY

SOP Number	Effective Date	Significant Changes	Previous SOP Number
Draft Version	**11/29/2017**	**Draft Original SOP**	**Draft**
Version 1	**12/13/2017**	**Incorporated Review Comments**	**Version 1**

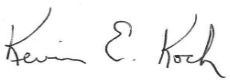
MOTT MACDONALD

Standard Operating Procedure

Sample Collection

Penn Station, New York

Version 1

	NAME	TITLE	SIGNATURE	DATE
Author	**Francis Pandolfo**	**Environmental Scientist**		**11/28/2017**
Reviewer	**Kevin Koch, PE, LSRP**	**Quality Assurance Coordinator**		**11/28/2017**
Authoriser	**Robert Trepp Jr., LSRP**	**Project Manager**		**11/28/2017**

Effective Date:	**12/13/2017**
Review Date:	**12/13/2017**

1. PURPOSE

- Consistent sampling procedures are necessary to further investigate the extent of impacts present in media at PSNY.

2. INTRODUCTION

- Sample collection occurs across multiple medias and locations throughout PSNY.
- Samples can fall into the following medias;
    - o Ballast Fines
    - o Ballast Stone
    - o Concrete
    - o Soil
    - o Sediment in Drains
    - o Dust
    - o Air
- The following procedure serves as a guide in performing sampling operations within PSNY.

3. SCOPE

- Samples are to be collected at designated locations in PSNY.
- Samples will only be collected if the required amount of material is present at the sample locations.
- Samples will be collected based on track outages, foul time and protection availability.

4. RESPONSIBILITIES

- Sampling equipment will be dedicated or decontaminated (see Decontamination of Field/Sampling Equipment SOP).
- Be sure all equipment and sampling materials are accounted for and available for the sampling event.
- Samples should be immediately placed on ice in a cooler after collection.
- Be sure measurements of the track structure and sample location are accurate during sampling and corelated to Railroad 0.
- Chain of custodies and sample labels should be written correctly and clearly.
- Sample coolers must have signed custody seal on them.

5. LIST OF EQUIPMENT

- Laboratory glassware
- Stainless-steel scoopulas
- Stainless-steel trowels
- Latex gloves
- Cooler with ice
- Plastic sheeting
- Sample labels
- Chain of custody

- Cooler custody seal
- Decontamination materials (See Decontamination SOP)

6. SPECIFIC PROCEDURE

- **6.1 Ballast Fines**
    - Samples of Ballast Fines will be collect from inside the center of the track gauge.
    - Samples will be collected from test holes excavated to a depth of 0.5 feet below the railroad tie at a rate of one per 25 linear feet of track structure.
    - At every 150 linear feet of track structure, test holes will be excavated until refusal is reached. Samples will be collected at six-inch depth intervals from the bottom of the test hole to the bottom of the rail tie.
    - The sample will be collected using a shovel, post hole digger and other equivalent hand tools.
    - The hand tools used to excavate the test pit will be decontaminated prior to, and in between each sample location (see decontamination SOP).
    - The sample analysis and volume is provided in the QAPP dated January 23, 2018.
    - The test pits will be excavated first and then sampled from the deepest interval upwards.
    - Samples will be collected using dedicated stainless-steel scoopulas/trowels. One scoopula/trowel will be used to dress the area prior to sampling and another clean scoopula/trowel will be used to transfer the sample material directly into laboratory provided glass ware. Care will be given to avoid collecting ballast stone when sampling.
- **6.2 Ballast Stone**
    - Samples of ballast stone will be collected at a rate of one per every 150 linear feet of track structure from the surface at the first interval encountered.
    - Samples will be collected using dedicated stainless-steel scoopulas and trowels. Care will be given to avoid collecting ballast fines when sampling.
    - The sample analysis and volume is provided in the QAPP dated January 23, 2018.
- **6.3 Concrete**

- Samples of concrete will be collected from the center of the trough in the middle of the track gauge at a rate of one per every 100 linear feet of concrete track structure.
- An additional concrete sample will be collected adjacent to drains if a drain is encountered.
- Concrete samples are collected from the surface of the track structure using a pneumatic impact hammer and chisel bit to remove an area of concrete approximately 4” by 4” and to a depth of approximately 1/4” in thickness.
- The chisel bits are decontaminated between sample locations (see decontamination SOP).
- The area shall be cleared of dust and other debris prior to start of sampling.
- Samples will be collected using dedicated stainless-steel scoopulas/trowels. The sample material will be placed into laboratory provided glassware.
- The sample analysis and volume is provided in the QAPP dated January 23, 2018.

- **6.4 SOIL**
  - Samples of soil (not consisting of ballast or ballast fines or other debris) will be collected at a rate of one per every 150 linear feet of track structure where present between the station wall and one per every 400 square feet in yard areas.
  - The test holes will be excavated by hand until the predetermined depth is reached. The test holes will be excavated using a shovel, post hole digger, and other equivalent hand tools.
  - Hand tools used to excavate the test pit will be decontaminated prior to, and in between each sample location (see decontamination SOP).
  - The sample analysis and volume is provided in the QAPP dated January 23, 2018.
  - Samples will be collected using dedicated stainless-steel scoopulas and trowels samples will be collected from every six-inch depth interval from the bottom of the tie to the bottom of the test pit. One scoopula/trowel will be used to dress the area prior to sampling and another clean scoopula/trowel will be used to transfer the sample material directly into laboratory provided glass ware. Samples will be collected from the deepest interval upwards.
- **6.5 SEDIMENT IN DRAINS**
  - When drains are encountered Mott MacDonald will inspect the structure for viable amounts of sediment to be sampled.

- If 12 or more inches of sediment is present within the drain, two samples will be collected.
  - The first sample will be collected from the top six of sediment.
  - The second sample will be collected from the bottom six inches of sediment.
- If less than 12 inches of sediment is present within the drain only one sample will be collected from the bottom six inches of sediment.
- Samples will be retrieved from the drain structure using hand tools such as post hole diggers and/or hand augers and placed on dedicated, clean plastic sheeting.
- All reusable equipment used to retrieve samples will be decontaminated between sample locations (see decontamination SOP).
- Samples will be collected using dedicated stainless-steel scoopulas or trowels. One scoopula/trowel will be used to sample each discrete six inch intervalt of sediment on the plastic sheeting and transfer the sample material directly into laboratory provided glass ware.

- **6.6 DUST**
  - Bulk dust samples will be collected from surfaces in various areas across PSNY.
  - At each sample location, the area will be inspected for a surface with a sufficient mass of accumulated dust to be sampled.
  - A representative sample location will be selected based on visual inspection and may include;
    - 3rd rail covers
    - Ground surface
    - Walls
    - Conduit
    - Light fixtures
    - Signs
    - Wires
    - Columns
    - Electrical equipment
    - Other surfaces as available
  - Samples will be collected using dedicated stainless-steel scoopulas and trowels and transferred into laboratory provided glassware.
  - A scale (tared weight of sample container subtracted) will be used to ensure that at least 45 grams of dust is collected which is required by the lab per sample.
  - The sample analysis and volume is provided in the QAPP dated January 23, 2018.

- **6.7 AIR**
    - See the PCB Indoor Air Sampling SOP prepared by TRC.

7. SAFETY

- Required PPE;
    - Latex Gloves
    - Safety Glasses
    - Hard Hat
    - Steel Toed Boots
    - Tyvek Suit
    - Hearing protection
    - Respirator
    - Rubber overboots
    - Safety Vest
    - Amtrak Safety Cards

8. ABBREVIATIONS

- PSNY: Penn Station New York
- USEPA: United States Environmental Protection Agency
- PCB: Polychlorinated biphenyl
- SOP: Standard Operating Procedure
- TCL: Target Compound List
- TAL: Target Analyte List
- PPE: Personal Protective Equipment
- QAPP: Quality Assurance Project Plan prepared by Mott MacDonald dated January 23, 2018.

9. CHANGE HISTORY

SOP Number	Effective Date	Significant Changes	Previous SOP Number
Draft Version	**11/28/2017**	**Draft Original SOP**	**Draft**
Version 1	**12/13/2017**	**Incorporated Review Comments**	**Version 1**

Safety Data Sheet: **Simple Green® All-Purpose Cleaner** ★

Version No. 13000-14B Issue Date: September 13, 2014 Supersedes Date: January 7, 2014 *OSHA HCS-2012 / GHS*

Section 1: IDENTIFICATION

Product Name: Simple Green® All-Purpose Cleaner
Additional Names:

Manufacturer's Part Number: **Please refer to Section 16*

Recommended Use: Cleaner & Degreaser for water tolerant surfaces.
Restrictions on Use: Do not use on non-rinsable surfaces.

Company: Sunshine Makers, Inc.
15922 Pacific Coast Highway
Huntington Beach, CA 92649 USA

Telephone: 800-228-0709 ● 562-795-6000 *Mon – Fri, 8am – 5pm PST*
Fax: 562-592-3830
Email: info@simplegreen.com

Emergency Phone: Chem-Tel 24-Hour Emergency Service: 800-255-3924

Section 2: HAZARDS IDENTIFICATION

This product is not classified as hazardous under 2012 OSHA Hazard Communication Standards (29 CFR 1910.1200).

OSHA HCS 2012
Label Elements
Signal Word: None **Hazard Symbol(s)/Pictogram(s):** None required

Hazard Statements: None
Precautionary Statements: None
Hazards Not Otherwise Classified (HNOC): None
Other Information: None Known

Section 3: COMPOSITION/INFORMATION ON INGREDIENTS

Ingredient	CAS Number	Percent Range
Water	7732-18-5	> 84.8%*
Ethoxylated Alcohol	68439-46-3	< 5%*
Sodium Citrate	68-04-2	< 5%*
Tetrasodium *N,N*-bis(carboxymethyl)-L-glutamate	51981-21-6	< 1%*
Sodium Carbonate	497-19-8	< 1%*
Citric Acid	77-92-9	< 1%*
Isothiazolinone mixture	55965-84-9	< 0.2%*
Fragrance	Proprietary Mixture	< 1%*
Colorant	Proprietary Mixture	< 1%*

**specific percentages of composition are being withheld as a trade secret*

Section 4: FIRST-AID MEASURES

Inhalation: Not expected to cause respiratory irritation. If adverse effect occurs, move to fresh air.
Skin Contact: Not expected to cause skin irritation. If adverse effect occurs, rinse skin with water.
Eye Contact: Not expected to cause eye irritation. If adverse effect occurs, flush eyes with water.
Ingestion: May cause upset stomach. Drink plenty of water to dilute. See section 11.

Most Important Symptoms/Effects, Acute and Delayed: None known.

Indication of Immediate Medical Attention and Special Treatment Needed, if necessary: Treat symptomatically

Section 5: FIRE-FIGHTING MEASURES

Suitable & Unsuitable Extinguishing Media: Use Dry chemical, CO2, water spray or "alcohol" foam. Avoid high volume jet water.
Specific Hazards Arising from Chemical: In event of fire, fire created carbon oxides may be formed.
Special Protective Actions for Fire-Fighters: Wear positive pressure self-contained breathing apparatus; Wear full protective clothing.

See section 16 for NFPA rating.

Section 6: ACCIDENTAL RELEASE MEASURES

Personal Precautions, Protective Equipment and Emergency Procedures: *For non-emergency and emergency personnel:* See section 8 – personal protection. Avoid eye contact. Safety goggles suggested.

Environmental Precautions: Do not allow into open waterways and ground water systems.

Methods and Materials for Containment and Clean Up: Dike or soak up with inert absorbent material. See section 13 for disposal considerations.

Section 7: HANDLING AND STORAGE

Precautions for Safe Handling: Ensure adequate ventilation. Keep out of reach of children. Keep away from heat, sparks, open flame and direct sunlight. Do not pierce any part of the container. Do not mix or contaminate with any other chemical. Do not eat, drink or smoke while using this product.

Conditions for Safe Storage including Incompatibilities: Keep container tightly closed. Keep in cool dry area. Avoid prolonged exposure to sunlight. Do not store at temperatures above 109°F (42.7°C). If separation occurs, mix the product for reconstitution.

Section 8: EXPOSURE CONTROLS / PERSONAL PROTECTION

Exposure Limit Values: No components listed with TWA or STEL values under OSHA or ACGIH.

Appropriate Engineering Controls: Showers, eyewash stations, ventilation systems

Individual Protection Measures / Personal Protective Equipment (PPE)
Eye Contact: Use protective glasses or safety goggles if splashing or spray-back is likely.
Respiratory: Use in well ventilated areas or local exhaust ventilations when cleaning small spaces.
Skin Contact: Use protective gloves (any material) when used for prolonged periods or dermally sensitive.
General Hygiene Considerations: Wash thoroughly after handling and before eating or drinking.

Section 9: PHYSICAL AND CHEMICAL PROPERTIES

Appearance: Green Liquid
Odor: Added sassafras odor
Odor Threshold: Not determined
pH ASTM D-1293**:** 8.5 – 9.5
Freezing Point ASTM D-1177**:** 0-3.33°C (32-38°F)
Boiling Point & Range ASTM D-1120**:** 101°C (213.8°F)
Flash Point ASTM D-93**:** > 212°F
Evaporation Rate ASTM D-1901**:** ½ Butyl Acetate @ 25°C
Flammability (solid, gas): Not applicable
Upper/Lower Flammability or Explosive Limits: Not applicable
Vapor Pressure ASTM D-323**:** 0.60 PSI @77°F, 2.05 PSI @100°F
Vapor Density: Not determined

Partition Coefficient: n-octanol/water: Not determined
Autoignition Temperature: Non-flammable
Decomposition Temperature: 109°F
Viscosity: Like water
Specific Gravity ASTM D-891**:** 1.01 – 1.03
VOCs: ***Water & fragrance exemption in calculation*

SCAQMD 304-91 / EPA 24:	0 g/L	0 lb/gal	0%
CARB Method 310**:	2.5 g/L	0.021 lb/gal	0.25%
SCAQMD Method 313:	Not tested		

VOC Composite Partial Pressure: Not determined
Relative Density ASTM D-4017**:** 8.34 – 8.42 lb/gal
Solubility: 100% in water

Section 10: STABILITY AND REACTIVITY

Reactivity: Non-reactive.
Chemical Stability: Stable under normal conditions 70°F (21°C) and 14.7 psig (760 mmHg).
Possibility of Hazardous Reactions: None known.
Conditions to Avoid: Excessive heat or cold.
Incompatible Materials: Do not mix with oxidizers, acids, bathroom cleaners, or disinfecting agents.
Hazardous Decomposition Products: Normal products of combustion - CO, CO2.

Section 11: TOXICOLOGICAL INFORMATION

Likely Routes of Exposure:
Inhalation - Overexposure may cause headache.
Skin Contact - Not expected to cause irritation, repeated contact may cause dry skin.
Eye Contact - Not expected to cause irritation.
Ingestion - May cause upset stomach.

Symptoms related to the physical, chemical and toxicological characteristics: no symptoms expected under typical use conditions.
Delayed and immediate effects and or chronic effects from short term exposure: no symptoms expected under typical use conditions.
Delayed and immediate effects and or chronic effects from long term exposure: headache, dry skin, or skin irritation may occur.
Interactive effects: Not known.

Numerical Measures of Toxicity

Acute Toxicity:
Oral LD_{50} (rat) > 5 g/kg body weight
Dermal LD_{50} (rabbit) > 5 g/kg body weight
Calculated via OSHA HCS 2012 / Globally Harmonized System of Classification and Labelling of Chemicals

Skin Corrosion/Irritation: Non-irritant per Dermal Irritection® assay modeling. No animal testing performed.
Eye Damage/Irritation: Minimal irritant per Ocular Irritection® assay modeling. No animal testing performed.
Germ Cell Mutagenicity: Mixture does not classify under this category.
Carcinogenicity: Mixture does not classify under this category.
Reproductive Toxicity: Mixture does not classify under this category.
STOT-Single Exposure: Mixture does not classify under this category.
STOT-Repeated Exposure: Mixture does not classify under this category.
Aspiration Hazard: Mixture does not classify under this category.

Section 12: ECOLOGICAL INFORMATION

Ecotoxicity: Volume of ingredients used does not trigger toxicity classifications under the Globally Harmonized System of Classification and Labelling of Chemicals.
Aquatic: Aquatic Toxicity - Low, based on OECD 201, 202, 203 + Microtox: EC_{50} & IC_{50} ≥100 mg/L. Volume of ingredients used does not trigger toxicity classifications under the Globally Harmonized System of Classification and Labelling of Chemicals.
Terrestrial: Not tested on finished formulation.

Persistence and Degradability: Readily Biodegradable per OCED 301D, Closed Bottle Test
Bioaccumulative Potential: No data available.
Mobility in Soil: No data available.
Other Adverse Effects: No data available.

Section 13: DISPOSAL CONSIDERATIONS

Unused or Used Liquid: May be considered hazardous in your area depending on usage and tonnage of disposal – check with local, regional, and or national regulations for appropriate methods of disposal.

Empty Containers: May be offered for recycling.

Never dispose of used degreasing rinsates into lakes, streams, and open bodies of water or storm drains.

Section 14: TRANSPORT INFORMATION

U.N. Number: Not applicable
U.N. Proper Shipping Name: Cleaning Compound, Liquid NOI
Transport Hazard Class(es): Not applicable
NMFC Number: 48580-3
Packing Group: Not applicable
Class: 55
Environmental Hazards: Marine Pollutant - NO
Transport in Bulk (according to Annex II of MARPOL 73/78 and IBC Code): Unknown.
Special precautions which user needs to be aware of/comply with, in connection with transport or conveyance either within or outside their premises: None known.

U.S. (DOT) / Canadian TDG: Not Regulated for shipping.
ICAO/ IATA: Not classified as Hazardous
IMO / IDMG: Not classified as Hazardous
ADR/RID: Not classified as Hazardous

Section 15: REGULATORY INFORMATION

All components are listed on: TSCA and DSL Inventory.

SARA Title III: Sections 311/312 Hazard Categories – Not applicable.
Sections 313 Superfunds Amendments and Reauthorizations Act of 1986 – Not applicable.
Sections 302 – Not applicable.

Clean Air Act (CAA): Not applicable
Clean Water Act (CWA): Not applicable

State Right To Know Lists: No ingredients listed
California Proposition 65: No ingredients listed
Texas ESL:

Ethoxylated Alcohol	68439-46-3	60 µg/m^3 long term	600 µg/m^3 short term
Sodium Citrate	68-04-2	5 µg/m^3 long term	50 µg/m^3 short term
Sodium Carbonate	497-19-8	5 µg/m^3 long term	50 µg/m^3 short term
Citric Acid	77-92-9	10 µg/m^3 long term	100 µg/m^3 short term

Section 16: OTHER INFORMATION

Size	UPC	Size	UPC
2 oz. Pump	043318130366	1 Gallon w/ Dilution Bottle	043318000669
2 oz. Pump	043318131035	1 Gallon	043318000799
4 oz. Pump	043318130014	1 Gallon w/ Dilution Bottle	043318001383
16 oz. Trigger	043318130021	1 Gallon w/ Dilution Bottle	043318002021
22 oz. Trigger	043318130229	1 Gallon	043318130052
24 oz. Trigger, 12 per case	043318000034	1 Gallon w/ Dilution Bottle, 112 per case	043318480140
24 oz. Trigger	043318000300	1 Gallon w/ Dilution Bottle, 4 per case	043318480416
24 oz. Trigger	043318130137	1 Gallon w/ Dilution Bottle, 24 per case	043318480492
32 oz. Trigger	043318000652	1 Gallon w/ laundry	043318002052
32 oz. Trigger	043318130335	1 Gallon w/ towel	043318001222
67.6 oz	043318000393	140 oz.	043318001390
67.6 oz.	043318130144	140 oz., 168 per case	043318561405
1 Gallon w/ Dilution Bottle	043318000539	140 oz. w/ Dilution Bottle	043318001468
1 Gallon w/ Dilution Bottle	043318000645		

USA items listed only. Not all items listed. USA items may not be valid for international sale.

Section 16: OTHER INFORMATION - continued

NFPA:

Health – None
Flammability – Non-flammable
Stability – Stable
Special - None

Acronyms

NTP	National Toxicology Program	IARC	International Agency for Research on Cancer
OSHA	Occupational Safety and Health Administration	CPSC	Consumer Product Safety Commission
TSCA	Toxic Substances Control Act	DSL	Domestic Substances List

Prepared / Revised By: Sunshine Makers, Inc., Regulatory Department.
This SDS has been revised in the following sections: Revised SDS layout

DISCLAIMER: The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

Alpha Anlytical Standard Operating Procedures

Soxhlet Extraction

References: **EPA Method 3540C** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is applicable to solids, soils, and sludges.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the extraction of semivolatile organic compounds from solids such as soils, sludge's, and wastes. The Soxhlet extraction procedure ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the extraction of a variety of semivolatile organic compounds, which are then be analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Weighed samples, 2-30 grams, are prepared for extraction by mixing the sample with anhydrous sodium sulfate until the sample is free flowing. The sample is then spiked with the appropriate surrogate and LCS spike, placed in a Soxhlet extractor and extracted for 16-24hours using the appropriate solvent (Table 1). The extract is allowed to cool prior to proceeding with additional extract preparation steps.

Any water is removed from the sample extract by filtering through a powder funnel containing approximately 20g of anhydrous sodium sulfate. The extract is then concentrated using an S-EVAP bath solvent recovery system and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to analytical method SOPs for Reporting Limit information.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas) must be

scrupulously cleaned, following the cleaning SOP and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples should be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

7. Equipment and Supplies

7.1 Soxhlet Extractor: For large soxhlets (8270, DRO, EPH, PEST, etc.) add 200mL of the extraction solvent to a 250mL flat bottom flask and boiling stones. Attach a 45/50 soxhlet to the flat bottom flask and place a plug of glasswool into the soxhlet. For all PCB products, add 100mL of the extraction solvent to a 150mL flat bottom Erlenmeyer flask, and add boiling stones to the flask. Attach a 45/50 reduced volume soxhlet to the Erlenmeyer flask and place a plug of glasswool into the soxhlet. For either size soxhlet, wet the glasswool with extraction solvent. Using a spatula, cover the siphon tube with a plug of glass wool. For all pesticide samples use filter paper in place of glass wool. Add enough sodium sulfate to Soxhlet to keep glass wool or filter paper in place (typically 5-10g).

7.1.1 250mL Flat Bottom Flask

7.1.2 45/50 Soxhlet Extractor

7.1.3 150mL Flat Bottom Erlenmeyer Flask

7.1.4 45/50 Reduced Volume Soxhlet

7.2 Top Loading Balance: Capable of weighing to 0.01g.

7.3 Heating Plate: Rheostat controlled.

7.4 Whatman filter paper: use for filtering pesticides/8081. (Whatman no.1 or equivalent)

7.5 Syringes: 1mL, 250µL, 25ul, for measuring surrogates and Spikes

7.6 Disposable Borosilicate Transfer Pipets.

7.7 Spatulas: Stainless steel.

7.8 Beakers: 250mL stainless steel.

7.9 Aluminum weighing dishes: VWR Cat #25433-089.

7.10 Mortar and Pestle: Capable of reducing particle size to <1mm.

7.11 Kuderna-Danish (KD) apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic Kek clip. Add the Macro Synder column to the Evaporation Flask. The evaporation flask is attached directly to the Concentrator Tube using the Plastic Clip.

7.11.1 Concentrator tube: 25mL, graduated. A ground-glass stopper is used to prevent evaporation of extracts.

7.11.2 Evaporation flask: 500mL.

7.11.3 Snyder column: Three-ball macro.

7.11.4 Plastic Kek clips.

7.12 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.13 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.14 Boiling Chips: Solvent-extracted, approximately 10/40 mesh (silicon carbide). PTFE boiling stones D1069103

7.15 Brady labeling system: Thermal label generator.

7.16 Sodium Sulfate glass filtering funnels. Add a plug of glass wool to the base of the 104mm stainless steel funnel. Add approximately 20grams of baked sodium sulfate.

7.17 Glass wool: SUPELCO, silane treated.

7.18 N-EVAP: Organomation; utilized for micro blow down.

7.19 Gauze Pad: 2''x2'' 12 ply, Used as a blank matrix for wipe samples.

7.20 Multi-Position Stirring Plates.

7.21 Magnetic Stirring Bars.

7.22 250mL Erlenmeyer flask.

7.23 Solvent pump dispenser: Dispensette Organic 100ml

8. Reagents and Standards

Reagent grade inorganic chemicals are used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades are used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a stainless steel beaker. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent.

8.3 Methylene Chloride: Pesticide quality or equivalent. No expiration date listed.

8.4 Hexane: Pesticide quality or equivalent. No expiration date listed.

8.5 Acetone: Pesticide quality or equivalent. No expiration date listed.

8.6 1:1 Acetone/Methylene Chloride: Using a 2000mL Graduated Cylinder add 2000mL of Acetone and 2000mL of Methylene Chloride to a 4 Liter amber bottle and record preparation.

8.7 1:1 Acetone/Hexane: Using a 2000mL Graduated Cylinder, add 2000mL of Acetone and 2000mL of Hexane into a 4 Liter amber bottle and record preparation.

8.8 Nitrogen Gas: Reagent grade, used to purge and pressurize the extraction cell and as the blow-down gas in the Turbovap II auto-concentrator units and the N-EVAP.

8.9 Spiking Solutions: The various surrogate and LCS/MS spiking solutions used in the extraction steps are listed in WI/14826 Soxhlet Extraction Guide. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.10 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 16 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. The specific QC requirements of the analytical methods are listed in WI/14826 Soxhlet Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for most methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions and volumes are listed in Table 2.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD are carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/14826 Soxhlet Extraction Guide.

9.8 Method Sequence

See Section 10.

10. Procedure

All soil soxhlet extractions follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the ELN.

10.1 Sample Preparation and Extraction

10.1.1 Samples are batched into the ELN to create the Work Group. See Work Instruction 2421, Labeling and Generating Work Groups and Batches. Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Labels are printed and placed on the cap of the soil container.

10.1.2 All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

10.1.3 Prepare the hotplate station for soxhlet extraction. Turn on the hotplate to the calibrated mark. Rinse all condensers with approximately 20mL of 1:1 DCM/Acetone solution. Turn on the chiller serving the soxhlets being extracted. Chiller temperature should be pre-set at 10C.

10.1.4 During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the analyst must contact login or the project manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

10.1.5 When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

10.1.6 The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or wipe) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

10.1.7 Align soxhlet extractors (See Section 7.1) containing the extraction solvent (See Table 1) in the hood adjacent to the top loading balance. See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Weigh 2-30 grams of sample (depending on matrix) into a beaker or weighing dish and record in the ELN. Add approximately 15g anhydrous sodium sulfate to the beaker and sample. Mix with a spatula until a free flowing. For samples that contain higher amounts of water or organic matter, larger amounts of sodium sulfate may be required. Certain samples may require grinding with a mortar and pestle to achieve a free flowing consistency. Individually transfer the sample into the soxhlet extractor from the beaker or weighing dish. Weighing dishes are used for all PCB (8082) samples. Add the appropriate surrogate and spiking solution. (Refer to WI/14826 Soxhlet Extraction Guide). Sodium Sulfate is used for the QC substrate for all methods except for wipes. A Gauze Pad is used as a QC substrate for wipes.

10.1.8 For all wipe (matrix 7) samples, the entire sample is extracted. Transfer the entire wipe into the Soxhlet Set-up (See Section 7.1).

10.1.9 See Work Instruction WI/2421 for labeling and extraction instructions.

10.1.10 After weighing all samples, attach the soxhlet set-up to the condensers. Record the time of extraction in the “Extraction Date” column of the ELN as well as stop time on the whiteboard above the hotplate. Record the hotplate ID in the “Extraction Unit ID” column of the ELN for each sample.

10.1.11 Extract the sample for 16 – 24 hours making sure to achieve four to six cycles/hour. See WI/2421 for labeling details.

10.1.12 Record the time that the samples are taken off of the hotplate in the “Stop Date/Time” column of the ELN. Allow the extract to cool after the extraction is complete.

10.1.13 After the extract has cooled, drain the solvent from the extractor into the bottom flask. Rinse the Soxhlet with solvent if any residual sample is present and drain into the bottom flask. The extracted samples are disposed of in the waste barrels.

10.1.14 Once the samples have cooled to room temperature, filter through a sodium sulfate funnel and into a labelled KD setup or Buchi vessel. The sodium sulfate funnel contains glass wool and approximately 20g of sodium sulfate. **NOTE:** Due to contamination issues, pesticide samples require filter paper instead of glass wool.

10.1.15 For ETPH Analysis, the sample extract is filtered through a funnel packed with glass wool and approximately 20 grams of sodium sulfate, collecting the filtrate using into a 250mL Erlenmeyer flask. Add 3 grams of Deactivated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes @650rpm. Filter the extract through a funnel plugged with a filter paper, collecting the filtrate in a Bucchi vessel for concentration, see section 10.2.3

10.1.16 Proceed to sample concentration. Note all DRO, ETPH, and 8270 products are concentrated using the Bucchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique

10.2.1.1 Assemble a KD apparatus (Section 7.11) for each sample and QC by attaching the 25 mL concentrator tube to the bottom of the 500 mL KD flask. Use a blue kek clip to help insure that they do not separate. Place a boiling stone in the bottom of the concentrator tube. Assemble a sodium sulfate funnel and place on

top of the flask. See WI 10995, Solvent rinsing/filtering guide. Drain the sample through this sodium sulfate funnel. After the sodium sulfate funnel is done draining remove and discard it. Alternatively, the sample may be filtered directly into a Buchi vessels for DRO and ETPH samples (See Section 10.2.3).

10.2.1.2 Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a 75^{o}C hot water bath(SEVAP) so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.3 If a Hexane exchange is required (see Table 1), when the sample volume reaches 5 to 15mL, remove the condenser from the Synder Column and add 20 mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow the sample to concentrate to 15-20mL and exchange with another 20mL of hexane. Allow the sample to boil until the intensity decreases (little to no chatter in the Snyder column). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to approximately 15mL.

10.2.1.4 Remove the KD apparatus from the water bath. Remove Kek clip. Wipe the joint of the flask and concentration tube with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool for 5 minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI/2421).

10.2.1.5 Place the Concentrator tube on the N-EVAP. Using a disposable pipet direct nitrogen over the sample. The N-EVAP is set at 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they reach the appropriate final volume (see S-Evap/N-Evap Concentration Standard Procedure WI#18528 for listing of appropriate volumes.

10.2.1.6 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI/3827, Extract Vialing Procedure, WI/2426, GC Extract Vialing Procedure and WI/2423, GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Bucchi

The Buchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Buchi will recover >95% of solvent emissions. Refer to Alpha SOP/12838 for Buchi concentration set-up and procedure.

10.3 Instrument Maintenance

10.3.1 Refrigeration Re-circulator

The Refrigeration Re-circulator should be checked periodically to insure that it is running correctly and that the level of reagent water is constant with manufactures recommendation.

10.3.2 Analytical Balance

10.3.2.1 All balances are checked daily and calibrated/serviced every six months by an instrument service company. All service records are kept on file.

10.3.2.2 Keep balances clean. Brush off any sample spills immediately.

11. Data Evaluation, Calculations and Reporting

Not Applicable.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Holding time exceedence and improper preservation are noted on the nonconformance report form.

12.2 When analysis of samples indicates possible extraction problems, such as poor surrogate recoveries, poor LCS/MS/MSD recoveries, or suspected contamination in blanks or samples, re-extractions are required. Depending on the particular failure, the re-extraction may be of a specific sample or the entire extraction batch.

12.3 The analyst that determines the need for re-extraction must fill out a sample re-extract request form. This form notes the reason for the re-extraction request along with any special requirements, and the date and time that the re-extract is needed. Re-extraction request forms are maintained on file to help track the cause for re-extractions, and to be used as a tool in improving systems to minimize the need for re-extractions.

12.4 Depending on the results of the re-extraction, the first, second, or both sets of results may be reported to the client, along with a narrative report detailing the problems encountered and the resolution.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

Form 02-50 Sample Cleanup and Vialing Guide

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/2423 GC Mass Spec Extract Vialing Procedure

WI/2426 GC Extract Vialing Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

Form 02-58 Sample Extraction Guide

WI/14826 Soxhlet Extraction Guide

SOP/12838 Buchi concentration

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

LIMS Product Code	Extraction Solvent	Exchange Solvent Required	Typical Final Volume	Appropriate Cleanup Technique
8082	1:1 Hexane/Acetone	hexane	5-10 mL	Sulfuric acid
8081	1:1 DCM/Acetone	hexane	10 mL	Florisil
8270	DCM	---	1 mL	---
8270 CNCRT	DCM	---	1mL	---
TPH *	DCM	---	1 mL	---
MA EPH	DCM	hexane	1 mL	Silica gel Fractionation
NJ EPH	DCM	hexane	1 mL	Silica gel Fractionation
EPH-TPH**	DCM	hexane	1mL	---
ETPH	DCM	---	1 mL	Silica gel

***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

****EPH-TPH includes the following LIMS Products: NJEPH-TPH-CAT1, NJEPH-TPH-CAT2**

Total Solids in Solid and Semisolid Samples

(Percent Solids)

Reference Method: **SM 2540 G,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Soils, solids and sludges.

Definitions: See Alpha Analytical Quality Manual

This method is applicable to the determination of total solids in such solid and semisolid samples as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by completing an initial demonstration of capability

2. Summary of Method

A homogenized aliquot of sample is weighed in a tared dish and set in a 103º - 105ºC oven until dry. The sample and dish are cooled and re-weighed, thus the percent of solids in the original sample can be calculated.

2.1 Method Modifications from Reference

Aluminum pans are used instead of porcelain dishes. However, if the sample is corrosive, then the porcelain dishes are used.

3. Reporting Limits

The Reported Detection Limit is 0.1%.

4. Interferences

4.1 Humidity: Humidity in the laboratory may cause samples to pick up moisture. When not being weighed, samples should be kept tightly capped or in a dessicator.

4.2 Large rocks / debris: Large rocks or debris may cause false high results and therefore should not be included in the sample aliquot.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. Personal protective equipment is to be worn at all times within the laboratory areas. At a minimum, a labcoat, gloves and safety glasses are worn.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic containers with minimal headspace. Containers are covered immediately to minimize the loss of sample moisture.

6.2 Sample Preservation

Samples are refrigerated at 4 ºC.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are kept refrigerated at 4 ºC until the time of analysis.

For samples received, marked and commented as **Foreign Soils** reference SOP 2296 Treatment of Foreign Soils.

For samples received, marked and commented as **Containing or May contain Asbestos** reference WI 2535 Asbestos Handling Procedures.

7. Equipment and Supplies

7.1 Analytical Balance: Capable of weighing to 0.01g

7.2 Aluminum Weighing Dishes or Pans

7.3 Porcelain Evaporation Dishes

7.4 Dessicator: With a color-indicator dessicant.

7.5 Drying Oven: Capable of maintaining 103 – 105 ºC.

7.6 Oven Trays

7.7 Computer: with connection to LIMS and the Analytical Balance (Sect. 7.1)

8. Reagents and Standards

None.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate is analyzed per batch of 20 samples or less. Duplicate determinations must agree within 20%. If this criterion is not met, the sample and it's duplicate are reanalyzed.

If sample, used for batch duplicate, is non-homogeneous, then data may be reported with a narrative.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

Prepare evaporation dishes, if necessary.

Generate a LIMS Batch

Open the appropriate Excel Spreadsheet

Record the Tare weights

Homogenize sample

Record the Gross weights

Dry samples in the oven 2+ hours (samples logged with product ME-TS-2540 must be dried overnight.

Cool samples in the dessicator

Record the Net Weight (1)

Dry samples again for 1+ hours unless samples were originally dried overnight.

Note: if samples are dried overnight, then one weight is used; drying overnight should be noted on excel format. This statement can be applied to all samples, except samples with state of origin ME.

Cool samples in the dessicator

Record the Net Weight (2)

Save to LIMS

Note: Samples with state of origin ME must be logged using product ME-TS-2540; two weights are required to prove constant weight, all samples must be dried overnight.

10. Procedure

10.1 Equipment Set-up

10.1.1 LIMS Knowledge: Prior to utilizing this SOP, the analyst must first be familiar with the operation of the Laboratory Information Management System (LIMS) and the generation of a sample batch or workgroup.

10.1.2 Porcelain Dish Preparation: Porcelain evaporation dishes are used only if a sample is corrosive to aluminum. To prepare the porcelain dishes, bake them in the 103 – 105 ºC drying oven for a minimum of 1 hour before placing them in the dessicator. Cool in the dessicator for a minimum of one hour.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Generating a LIMS Batch

Utilizing the computer (Sect. 7.7), generate a LIMS batch of samples and assign a Workgroup (WG) number to the batch. When generating the batch, choose a sample that will be duplicated. Print out a copy of the LIMS batchsheet.

10.3.2 Using the Excel Spreadsheet

10.3.2.1 Ensure that the Balance Software Wedge is open. Do this by clicking on the "Balance" icon on the Desktop, and then minimize the window that appears. This will open the lines of communication between the balance and the computer.

10.3.2.2 To open the WetChem Excel sheets, click on the the "Shortcut to Wetchem" icon on the Desktop.

10.3.2.3 Open the sheet entitled "TS_S.xlt.

10.3.2.4 Input the following information into the appropriate spaces on the Excel sheet: The WG number (as assigned in Section 10.3.1), the Chemist's initials, the date and the time.

10.3.2.5 Click on the "Get Samples" button on the Excel Sheet.

10.3.2.6 The Samples assigned to the WG number will be uploaded onto the Excel Sheet. However, verify that the Samples uploaded are the same samples that were printed on the LIMS batchsheet in Section 10.3.1.

10.3.3 Write the sample ID's on the weighing dishes (either aluminum or prepared porcelain dishes).

10.3.4 Taking the Tare Weight

10.3.4.1 On the Excel Sheet, click on the cell for the "Tare" weight for the appropriate sample.

10.3.4.2 Weigh the corresponding empty dish on a tared balance. When the weight is stable, push the "Print" button on the balance. This will transfer the weight of the empty dish into the Excel Sheet.

10.3.4.3 Repeat Sections 10.3.4.1 and 10.3.4.2 until the weight of all of the empty dishes has been recorded on the Excel Sheet.

10.3.5 Taking the Gross Weight

10.3.5.1 Homogenize the sample by mixing with a spatula or spoon.

10.3.5.2 Remove a 5 – 10g aliquot of soil sample or 20 – 25g of a sludge sample and place it in the weighing dish.

10.3.5.3 Click on the cell for the "Gross Weight" corresponding to the sample to be weighed.

10.3.5.4 Zero the balance. Weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the Excel Sheet.

10.3.5.5 Place the dish onto an oven tray.

10.3.5.6 Repeat Sections 10.3.5.1 through 10.3.5.5 until all of the samples have been weighed.

10.3.5.7 Once the samples have all been weighed and the weights recorded in the Excel Sheet, click on "File" then on "Save As". Type in the WG number from the LIMS batchsheet (generated in Section 10.3.1) as the filename. Then click "OK". The batch has been saved under the WG number in the "My Documents" folder.

10.3.6 Drying the Samples : Phase I

10.3.6.1 Place the oven tray in the 103 – 105 ºC drying oven.

10.3.6.2 After a minimum of two hours (samples with state of origin ME must be dried overnight), if samples appear dry, move the oven tray of dried samples to a dessicator. Allow to cool completely.

10.3.7 Taking the First Net Weight

10.3.7.1 Open the "My Documents" folder by clicking on the icon on the Desktop.

10.3.7.2 Select the WG number of the batch you are going to weigh. This will open the Excel Sheet. Verify that the correct batch sheet has been opened.

10.3.7.3 Click on the cell for the "Net Weight(1)" corresponding to the sample to be weighed.

10.3.7.4 Zero the balance. Weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the Excel Sheet.

10.3.7.5 Repeat Sections 10.3.7.3 and10.3.7.4 until the Net Weight (1) for all samples has been recorded.

10.3.7.6 Click on the "Save" button to save the weights.

10.3.7.7 If samples have been dried overnight, type "Dried Overnight" in the comments field and proceed to Section 10.3.10.

10.3.8 Drying the samples : Phase II

10.3.8.1 If the Net Weight of the samples is taken on the same day as the Gross Weight, the tray of samples must be placed back in the 103 – 105 ºC drying oven for a minimum of one hour.

10.3.8.2 After drying, move the oven tray of samples to a dessicator. Allow to cool completely.

10.3.9 Taking the Second Net Weight

10.3.9.1 Open the "My Documents" folder by clicking on the icon on the Desktop.

10.3.9.2 Click on the WG number of the batch you are going to weigh. This will open the Excel Sheet. Verify that the correct batch sheet has been opened.

10.3.9.3 Click on the cell for the "Net Weight(2)" corresponding to the sample to be weighed.

10.3.9.4 Zero the balance. Weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the Excel Sheet.

10.3.9.5 Repeat Sections 10.3.9.3 and 10.3.9.4 until the Net Weight (2) for all samples has been recorded.

10.3.9.6 Click on the "Save" button to save the weights.

10.3.9.7 If Net Weight (1) and Net Weight (2) are within 4% or 50mg, the Excel Spreadsheet will display the word "Acceptable" in the far right column next to the appropriate sample. If "Acceptable" is displayed for all samples in the batch, proceed to Section 10.3.10.

10.3.9.8 If Net Weight (1) and Net Weight (2) are <u>not</u> within 4% or 50mg, repeat Sections 10.3.8 and 10.3.9 for those samples. This will allow the chemist to record a Net Weight (3), (4) or (5), until the word "Acceptable" is displayed in the far right column next to the appropriate sample.

If the samples have been dried <u>≥</u> 24 hours, and the weights are still not within 4% or 50mg, consult the Department Supervisor as to how to proceed.

10.3.10 Saving the Batch

10.3.10.1 Click on the "Save" button to save the weights in the Excel sheet.

10.3.10.2 Click on the "Save to LIMS" button on the spreadsheet.

10.4 Continuing Calibration

Not applicable.

10.5 Preventative Maintenance

The temperature of the laboratory ovens is recorded constantly on a circular chart recorder. The chart recorder and the laboratory ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

Analytical balances are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file. The calibration of the balances is verified on a daily basis and records are kept in a Logbook.

11. Data Evaluation, Calculations and Reporting

The Excel Spreadsheet is programmed to calculate the Percent Solids results. This is the formula that is used for calculation:

$$\% \text{ Total Solids} = \frac{(A - B)}{(C - B)} \times 100$$

Where: A = Final Net Weight (weight of dried residue + dish, g)
B = Tare weight (weight of dish, g)
C = Initial Gross Weight (weight of wet sample + dish, g)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Improper preservation is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial **(IDC)**

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Unless the containers are labeled as hazardous material (i.e. low flashpoint, ignitable, containing asbestos or high levels of toxic materials), the dried samples are disposed of into the trash.

If sample containers are labeled as hazardous, refer to the Chemical Hygiene Plan for waste handling and disposal instructions.

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

WI 2535 Asbestos Handling Procedures

SOP 2296 Treatment of Foreign Soils

16. Attachments

None.

Particle Size Reduction Work Instruction

Summary: Particle size reduction is required when a sample does not meet the size requirements defined by an SOP or reference method. In the Organic Extractions Laboratory, particle size reduction is most commonly needed for TCLP/SPLP leaching, Microwave Extraction, Waste Dilutions/Screen extraction, and Soxhlet Extraction. The most common matrix that requires particle size reduction are solids (such as concrete, rocks, wood, caulking, etc.).

Required Materials: Hammer, aluminum weigh dish (tall), lab mat, cardboard.

Procedure:

1) Place the cardboard on a hard, durable surface such as a cement floor or stainless steel counter top. Crushing samples on a lab bench is discouraged due to the lack of durability of the surface. If the samples must be crushed in a hood due to asbestos concerns, place an extra layer of cardboard on the surface to protect it as much as possible.
2) Place the lab mat over the cardboard. This will create a clean area to process the sample and make for easy cleanup once the sample is crushed.
3) Place the sample into an aluminum weigh dish. Place another weigh dish on top of the sample. Place the weigh dishes on the cardboard/lab mat.
4) Use the hammer to crush the contents of the aluminum dish. As soon as the sample is believed to be the correct size or either of the aluminum dishes forms a hole, stop hitting the sample with the hammer.
5) Remove any particles that meet the size requirement, replace both of the aluminum dishes, and continue to crush the sample until the required volume is achieved.
6) The lab mat should be replaced between every new sample.
7) The cardboard should be replaced every 2-3 samples to reduce the chance of damaging the surface of the counter, floor or lab bench.

PCBs

By Capillary Column Gas Chromatography

Reference Methods: Method 8082A SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Quality Control Requirements and Performance Standards for Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Action under the Massachusetts Contingency Plan (MCP), Revision No.1, July 1, 2010.

State of Connecticut, Department of Environmental Protection, RRCP, Version 2.0, July 2006.

1. Scope and Application

Method 8082A is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid and liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).
Matrices: Extracts from solid and liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.
This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume or weight of sample (approximately 1L for liquids, 15g to 30g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique. See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), or other appropriate technique. Solid samples may also be extracted with hexane:acetone (1:1) using Method 3546 (microwave). See extraction SOP for details.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique. See extraction SOP for details.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

Internal standard calibration is used for all analysts. The internal standard used is 1-bromo-2-nitrobenzene.

3. Reporting Limits

The reporting limits for this method as outlined is as follows:

- Aqueous samples: 0.25 ug/L / Aroclor (based on a 1L extraction)
- Soil Samples: 33.3 ug/kg / Aroclor (based on a 15g extraction)
- Solid of Difficult Matrices (i.e Caulking, Concrete, etc. are logged using the Alpha Low Level 8082 products): based on a 15g extraction
    - Aroclors 1016, 1221, 1232, 1242, 1254: 20 ug/kg
    - Aroclors 1248, 1260: 13.3 ug/kg
    - Aroclors 1262, 1268: 6.67 ug/kg

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the helium (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.

4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2^{o} C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2^{o} C.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL, for standards preparation

7.5 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent

7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used,

provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.
NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2° C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.4 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.

8.5 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampulated calibration standards are obtain from **Ultra**:

- Aroclor 1016, Cat. #PP-282, at 100ug/ml
- Aroclor 1260, Cat. #PP-361, at 100ug/ml
- Aroclor 1262, Cat. #PP-371, at 100ug/ml
- Aroclor 1268, Cat. #PP-382, at 100ug/ml
- Aroclor 1221, Cat. #PP-292, at 100ug/ml
- Aroclor 1232, Cat. #PP-302, at 100ug/ml
- Aroclor 1242, Cat. #PP-312, at 100ug/ml
- Aroclor 1248, Cat. #PP-342, at 100ug/ml
- Aroclor 1254, Cat. #PP-351, at 100ug/ml

8.6 Second Source Standards: (CCAL) Continuing Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Continuing Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampulated standards are obtain from **Accustandard**:

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml

- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml
- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting of 500ul of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500ul of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 5ug/ml and DCB at 10ug/ml.
- **CCAL Surrogates Stock:** is prepared by diluting of 1ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1ml of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 10ug/ml and DCB at 20ug/ml.
- **Extraction Surrogates Stock:** is prepared by diluting of 10ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000ml of Acetone to achieve concentration of TCMX and DCB at 2ug/ml.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solution is prepared by diluting of 6.25ml of Arochlor 1016/1260 (Restek, Cat. #32039) to 500ml of Acetone to achieve concentration of Arochlor 1016/1260 at 12.5ug/ml.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water for water samples or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) pair is extracted with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. For Aroclor analysis, the LCS/LCSD are spiked with a mixture of Aroclor 1016 and 1260. The recovery acceptance criteria are listed in Table 2. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike

Upon client request, a matrix spike and matrix spike duplicate pair is extracted and analyzed with each batch of 20 or less samples. The MS/MSD pair is extracted and analyzed for standard PCB analysis. The recovery acceptance criteria are listed in Table 2. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be narrated for inclusion in the client report.

9.6 Laboratory Duplicate

Upon client request, a Laboratory Duplicate is extracted and analyzed with each batch of 20 or less samples. The relative percent difference (RPD) acceptance criteria are listed in Table 2. If the RPD criteria are not met, the failure may be attributed to matrix effect and must be narrated for inclusion in the client report.

9.7 Surrogates

All extracted samples and associated QC are spiked with Extraction Surrogates Stock to achieve concentration of TCMX and DCB at 0.5ug/ml. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 2. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level to identify the standard lot number)

1. Prime
2. Blank
3. Standard Level 1
4. Standard Level 2
5. Standard Level 3

6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1. 1016/1260 Continuing Calibration Standard **(identified with the standard lot number)**
2. Extraction Blank
3. Laboratory Control Sample
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples up to 16
7. Repeat 1 – 6 as needed.

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below, but may be altered as long as method performance criteria are met.

Temperature1: 120 ºC	Injector temerature: 250ºC
Time1: 0 minutes	Injector mode: Pulsed Split
Ramp1: 45ºC/minute	1.4:1 split, 0.20 min pulse
Temperature2: 200ºC	Injector Flow: 5.7 ml/min split flow
Time2: 0 minutes	Detector temperature: 350ºC
Ramp2: 15ºC/minute	Carrier gas: Helium
Temperature3: 230ºC	Carrier flow: 20ml/min
Time3: 0 minutes	Carrier mode: Constant flow
Ramp3: 30ºC/minute	Makeup gas: Argon/methane (P5)
Final temperature 330ºC	Total detector flow: 55ml/min
Final time: 2 minutes	Injection Volume: 1 µL

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 **Calibration Factor:** Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

10.2.6 **Initial Calibration Criteria**

If the **RSD for an analyte is < 20%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of linearity as long as the correlation coefficient is **≥0.995**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.7 **Initial Calibration Verification**

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20%** (**15% for CT RCP**) when compared to the mean response factor from the initial calibration.

10.2.8 Retention Time Window

10.2.8.1 The retention time window used for the identification of target analytes is ± 0.07 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.8.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more then one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. Minimum of 3 peaks must be chosen for each Aroclor. In these instances, the Analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty injections**. A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method. For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition (Method 8082A 11.6.2)

10.4.2 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20% difference** when compared to the initial calibration curve (**± 15% for CT RCP**). The Target data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual. A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples.

10.5 Preventive Maintenance

10.5.1 **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.5.2 **Other Maintenance:** ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375°C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 8082A, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of five calibration points** based on average response factors. The %RSD must meet the criteria of ≤20% for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:
C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Soil/sediment samples

$$\text{Concentration } (\mu g/Kg, \text{ dry weight}) = \frac{C \times DF \times Vf \times 1000}{W \text{ (gm)}} \div \%S$$

where:
C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.1.3 Reporting Results

11.1.3.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.3.2 Reporting Results for PCBs in Caulk Samples

If in the screen sample Aroclor concentration as calculated above is **> 20000ppm**, the Client is contacted by a Customer Service Representative and these results are sent to the LIMS and reported to the Client.

If the sample concentration as calculated above for any Aroclor is **< 20000ppm**, the sample is sent for re-extraction by Method 3540C (Alpha SOP/1954).

11.1.3.3 Summation Rules

"TOTAL" concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of "reportable" Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column "A" data, unless all individuals are reported from "B" column. "Total" is calculated based on the associated "Report List". See Work Instruction #14335 for details.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

.

13. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: STANDARD SOLUTIONS

Table 2: QC ACCEPTANCE CRITERIA

TABLE 1

STANDARD SOLUTIONS

STANDARD SOLUTIONS	Stock solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)	Spike Solution (ug/mL)	LCS Solution (ug/mL)
PCB									
Aroclor 1016/1260	100	0.1	0.5	1	2.5	5	10	12.5	12.5
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10		
Internal Standard									
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25		
Surrogates:									
Tetrachloro-m-xylene	2.0	0.64	0.02	0.04	0.08	0.16	0.32	2	2
Decachlorobiphenyl	2.0	1.28	0.04	0.08	0.16	0.32	0.64	2	2

TABLE 2

QC ACCEPTANCE CRITERIA

	Aqueous , Soils	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%
Decachlorobiphenyl	30%	150%

	Aqueous, Soils % Recovery		Duplicate and/or MSD	
MS/MSD and LCS	**Lower Control Limit**	**Upper Control Limit**	**Aqueous RPD**	**Soil RPD**
Aroclor 1016, 1260	40%	140%	30%	50%

Soxhlet Extraction

References: **EPA Method 3540C** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is applicable to solids, soils, and sludges.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the extraction of semivolatile organic compounds from solids such as soils, sludge's, and wastes. The Soxhlet extraction procedure ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the extraction of a variety of semivolatile organic compounds, which are then be analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Weighed samples, 2-30 grams, are prepared for extraction by mixing the sample with anhydrous sodium sulfate until the sample is free flowing. The sample is then spiked with the appropriate surrogate and LCS spike, placed in a Soxhlet extractor and extracted for 16-24hours using the appropriate solvent (Table 1). The extract is allowed to cool prior to proceeding with additional extract preparation steps.

Any water is removed from the sample extract by filtering through a powder funnel containing approximately 20g of anhydrous sodium sulfate. The extract is then concentrated using an S-EVAP bath solvent recovery system and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to analytical method SOPs for Reporting Limit information.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas) must be

scrupulously cleaned, following the cleaning SOP and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples should be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

7. Equipment and Supplies

7.1 Soxhlet Extractor: For large soxhlets (8270, DRO, EPH, PEST, etc.) add 200mL of the extraction solvent to a 250mL flat bottom flask and boiling stones. Attach a 45/50 soxhlet to the flat bottom flask and place a plug of glasswool into the soxhlet. For all PCB products, add 100mL of the extraction solvent to a 150mL flat bottom Erlenmeyer flask, and add boiling stones to the flask. Attach a 45/50 reduced volume soxhlet to the Erlenmeyer flask and place a plug of glasswool into the soxhlet. For either size soxhlet, wet the glasswool with extraction solvent. Using a spatula, cover the siphon tube with a plug of glass wool. For all pesticide samples use filter paper in place of glass wool. Add enough sodium sulfate to Soxhlet to keep glass wool or filter paper in place (typically 5-10g).

7.1.1 250mL Flat Bottom Flask

7.1.2 45/50 Soxhlet Extractor

7.1.3 150mL Flat Bottom Erlenmeyer Flask

7.1.4 45/50 Reduced Volume Soxhlet

7.2 Top Loading Balance: Capable of weighing to 0.01g.

7.3 Heating Plate: Rheostat controlled.

7.4 Whatman filter paper: use for filtering pesticides/8081. (Whatman no.1 or equivalent)

7.5 Syringes: 1mL, 250µL, 25ul, for measuring surrogates and Spikes

7.6 Disposable Borosilicate Transfer Pipets.

7.7 Spatulas: Stainless steel.

7.8 Beakers: 250mL stainless steel.

7.9 Aluminum weighing dishes: VWR Cat #25433-089.

7.10 Mortar and Pestle: Capable of reducing particle size to <1mm.

7.11 Kuderna-Danish (KD) apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic Kek clip. Add the Macro Synder column to the Evaporation Flask. The evaporation flask is attached directly to the Concentrator Tube using the Plastic Clip.

7.11.1 Concentrator tube: 25mL, graduated. A ground-glass stopper is used to prevent evaporation of extracts.

7.11.2 Evaporation flask: 500mL.

7.11.3 Snyder column: Three-ball macro.

7.11.4 Plastic Kek clips.

7.12 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.13 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.14 Boiling Chips: Solvent-extracted, approximately 10/40 mesh (silicon carbide). PTFE boiling stones D1069103

7.15 Brady labeling system: Thermal label generator.

7.16 Sodium Sulfate glass filtering funnels. Add a plug of glass wool to the base of the 104mm stainless steel funnel. Add approximately 20grams of baked sodium sulfate.

7.17 Glass wool: SUPELCO, silane treated.

7.18 N-EVAP: Organomation; utilized for micro blow down.

7.19 Gauze Pad: 2"x2" 12 ply, Used as a blank matrix for wipe samples.

7.20 Multi-Position Stirring Plates.

7.21 Magnetic Stirring Bars.

7.22 250mL Erlenmeyer flask.

7.23 Solvent pump dispenser: Dispensette Organic 100ml

8. Reagents and Standards

Reagent grade inorganic chemicals are used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades are used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a stainless steel beaker. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent.

8.3 Methylene Chloride: Pesticide quality or equivalent. No expiration date listed.

8.4 Hexane: Pesticide quality or equivalent. No expiration date listed.

8.5 Acetone: Pesticide quality or equivalent. No expiration date listed.

8.6 1:1 Acetone/Methylene Chloride: Using a 2000mL Graduated Cylinder add 2000mL of Acetone and 2000mL of Methylene Chloride to a 4 Liter amber bottle and record preparation.

8.7 1:1 Acetone/Hexane: Using a 2000mL Graduated Cylinder, add 2000mL of Acetone and 2000mL of Hexane into a 4 Liter amber bottle and record preparation.

8.8 Nitrogen Gas: Reagent grade, used to purge and pressurize the extraction cell and as the blow-down gas in the Turbovap II auto-concentrator units and the N-EVAP.

8.9 Spiking Solutions: The various surrogate and LCS/MS spiking solutions used in the extraction steps are listed in WI/14826 Soxhlet Extraction Guide. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.10 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 16 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. The specific QC requirements of the analytical methods are listed in WI/14826 Soxhlet Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for most methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions and volumes are listed in Table 2.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD are carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/14826 Soxhlet Extraction Guide.

9.8 Method Sequence

See Section 10.

10. Procedure

All soil soxhlet extractions follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the ELN.

10.1 Sample Preparation and Extraction

10.1.1 Samples are batched into the ELN to create the Work Group. See Work Instruction 2421, Labeling and Generating Work Groups and Batches. Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Labels are printed and placed on the cap of the soil container.

10.1.2 All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

10.1.3 Prepare the hotplate station for soxhlet extraction. Turn on the hotplate to the calibrated mark. Rinse all condensers with approximately 20mL of 1:1 DCM/Acetone solution. Turn on the chiller serving the soxhlets being extracted. Chiller temperature should be pre-set at 10C.

10.1.4 During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the analyst must contact login or the project manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

10.1.5 When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

10.1.6 The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or wipe) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

10.1.7 Align soxhlet extractors (See Section 7.1) containing the extraction solvent (See Table 1) in the hood adjacent to the top loading balance. See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Weigh 2-30 grams of sample (depending on matrix) into a beaker or weighing dish and record in the ELN. Add approximately 15g anhydrous sodium sulfate to the beaker and sample. Mix with a spatula until a free flowing. For samples that contain higher amounts of water or organic matter, larger amounts of sodium sulfate may be required. Certain samples may require grinding with a mortar and pestle to achieve a free flowing consistency. Individually transfer the sample into the soxhlet extractor from the beaker or weighing dish. Weighing dishes are used for all PCB (8082) samples. Add the appropriate surrogate and spiking solution. (Refer to WI/14826 Soxhlet Extraction Guide). Sodium Sulfate is used for the QC substrate for all methods except for wipes. A Gauze Pad is used as a QC substrate for wipes.

10.1.8 For all wipe (matrix 7) samples, the entire sample is extracted. Transfer the entire wipe into the Soxhlet Set-up (See Section 7.1).

10.1.9 See Work Instruction WI/2421 for labeling and extraction instructions.

10.1.10 After weighing all samples, attach the soxhlet set-up to the condensers. Record the time of extraction in the “Extraction Date” column of the ELN as well as stop time on the whiteboard above the hotplate. Record the hotplate ID in the “Extraction Unit ID” column of the ELN for each sample.

10.1.11 Extract the sample for 16 – 24 hours making sure to achieve four to six cycles/hour. See WI/2421 for labeling details.

10.1.12 Record the time that the samples are taken off of the hotplate in the “Stop Date/Time” column of the ELN. Allow the extract to cool after the extraction is complete.

10.1.13 After the extract has cooled, drain the solvent from the extractor into the bottom flask. Rinse the Soxhlet with solvent if any residual sample is present and drain into the bottom flask. The extracted samples are disposed of in the waste barrels.

10.1.14 Once the samples have cooled to room temperature, filter through a sodium sulfate funnel and into a labelled KD setup or Buchi vessel. The sodium sulfate funnel contains glass wool and approximately 20g of sodium sulfate. **NOTE:** Due to contamination issues, pesticide samples require filter paper instead of glass wool.

10.1.15 For ETPH Analysis, the sample extract is filtered through a funnel packed with glass wool and approximately 20 grams of sodium sulfate, collecting the filtrate using into a 250mL Erlenmeyer flask. Add 3 grams of Deactivated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes @650rpm. Filter the extract through a funnel plugged with a filter paper, collecting the filtrate in a Bucchi vessel for concentration, see section 10.2.3

10.1.16 Proceed to sample concentration. Note all DRO, ETPH, and 8270 products are concentrated using the Bucchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique

10.2.1.1 Assemble a KD apparatus (Section 7.11) for each sample and QC by attaching the 25 mL concentrator tube to the bottom of the 500 mL KD flask. Use a blue kek clip to help insure that they do not separate. Place a boiling stone in the bottom of the concentrator tube. Assemble a sodium sulfate funnel and place on

top of the flask. See WI 10995, Solvent rinsing/filtering guide. Drain the sample through this sodium sulfate funnel. After the sodium sulfate funnel is done draining remove and discard it. Alternatively, the sample may be filtered directly into a Buchi vessels for DRO and ETPH samples (See Section 10.2.3).

10.2.1.2 Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a 75°C hot water bath(SEVAP) so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.3 If a Hexane exchange is required (see Table 1), when the sample volume reaches 5 to 15mL, remove the condenser from the Synder Column and add 20 mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow the sample to concentrate to 15-20mL and exchange with another 20mL of hexane. Allow the sample to boil until the intensity decreases (little to no chatter in the Snyder column). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to approximately 15mL.

10.2.1.4 Remove the KD apparatus from the water bath. Remove Kek clip. Wipe the joint of the flask and concentration tube with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool for 5 minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI/2421).

10.2.1.5 Place the Concentrator tube on the N-EVAP. Using a disposable pipet direct nitrogen over the sample. The N-EVAP is set at 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they reach the appropriate final volume (see S-Evap/N-Evap Concentration Standard Procedure WI#18528 for listing of appropriate volumes.

10.2.1.6 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI/3827, Extract Vialing Procedure, WI/2426, GC Extract Vialing Procedure and WI/2423, GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Bucchi

The Buchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Buchi will recover >95% of solvent emissions. Refer to Alpha SOP/12838 for Buchi concentration set-up and procedure.

10.3 Instrument Maintenance

10.3.1 Refrigeration Re-circulator

The Refrigeration Re-circulator should be checked periodically to insure that it is running correctly and that the level of reagent water is constant with manufactures recommendation.

10.3.2 Analytical Balance

10.3.2.1 All balances are checked daily and calibrated/serviced every six months by an instrument service company. All service records are kept on file.

10.3.2.2 Keep balances clean. Brush off any sample spills immediately.

11. Data Evaluation, Calculations and Reporting

Not Applicable.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Holding time exceedence and improper preservation are noted on the nonconformance report form.

12.2 When analysis of samples indicates possible extraction problems, such as poor surrogate recoveries, poor LCS/MS/MSD recoveries, or suspected contamination in blanks or samples, re-extractions are required. Depending on the particular failure, the re-extraction may be of a specific sample or the entire extraction batch.

12.3 The analyst that determines the need for re-extraction must fill out a sample re-extract request form. This form notes the reason for the re-extraction request along with any special requirements, and the date and time that the re-extract is needed. Re-extraction request forms are maintained on file to help track the cause for re-extractions, and to be used as a tool in improving systems to minimize the need for re-extractions.

12.4 Depending on the results of the re-extraction, the first, second, or both sets of results may be reported to the client, along with a narrative report detailing the problems encountered and the resolution.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

Form 02-50 Sample Cleanup and Vialing Guide

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/2423 GC Mass Spec Extract Vialing Procedure

WI/2426 GC Extract Vialing Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

Form 02-58 Sample Extraction Guide

WI/14826 Soxhlet Extraction Guide

SOP/12838 Buchi concentration

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

LIMS Product Code	Extraction Solvent	Exchange Solvent Required	Typical Final Volume	Appropriate Cleanup Technique
8082	1:1 Hexane/Acetone	hexane	5-10 mL	Sulfuric acid
8081	1:1 DCM/Acetone	hexane	10 mL	Florisil
8270	DCM	---	1 mL	---
8270 CNCRT	DCM	---	1mL	---
TPH *	DCM	---	1 mL	---
MA EPH	DCM	hexane	1 mL	Silica gel Fractionation
NJ EPH	DCM	hexane	1 mL	Silica gel Fractionation
EPH-TPH**	DCM	hexane	1mL	---
ETPH	DCM	---	1 mL	Silica gel

***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

****EPH-TPH includes the following LIMS Products: NJEPH-TPH-CAT1, NJEPH-TPH-CAT2**

Total and Amenable Cyanide

References: **Method 9010C / 9012B,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2 and Revision 3 2004

SM 4500CN-CEG. Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-204-00-1-A, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1994.

Method 9014 (Modified). SW-846, Test Methods for Evaluating Solid Waste: Physical / Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: This method is applicable to waters, liquids, solids, soils and sludges.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The following SOP is a reflux-distillation procedure used to extract soluble cyanide salts and many insoluble cyanide complexes from wastes and leachates. It is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Cyanide, in the form of hydrocyanic acid (HCN) is purged from the sample and captured into an alkaline scrubber solution. The concentration of cyanide in the scrubber solution is then determined by flow injection analysis on a Lachat Analyzer.

This method was designed to address the problem of "trace" analyses (<1000ppm). The method may also be used for "minor" (1000ppm – 10,000ppm) and "major" (>10,000ppm) analyses by adapting the appropriate sample dilution. However, the amount of sodium hydroxide in the standards and the sample analyzed must be the same.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the distillation unit and/or the Lachat Instrument, and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide concentration in the absorbing solution is then determined colorimetrically by Lachat flow injection analysis.

2.1 Method Modifications from Reference

The sample size used is 50mL. The Midi distillation unit has demonstrated the ability to achieve the same RDL using 50mL instead of 500mL sample volume. Refer to EPA Method 335.4.

Modification for Method 9014: An automated determination of cyanide using the Lachat instrument is used instead of manual spectrophotometric determination.

Modification for amenable cyanide: Analysis is not prepped under amber light.

3. Detection Limits

The Reported Detection Limit for aqueous samples is 0.005mg/L; soil and solid samples is 1mg/Kg.

4. Interferences

4.1 Instrumental

None.

4.2 Parameters

4.2.1 Interferences are eliminated or reduced by using the distillation procedure. However, chlorine and sulfide are interferences. Refer to Section 9.1.

4.2.2 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10mg/L and in conjunction with certain organic compounds.

4.2.3 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10mg/L were not found to interfere.

4.2.4 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. Add anti-foaming agent to the sample during the distillation procedure (Section 9.2).

4.2.5 Carbonates and aldehydes are possible interferences

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following chemicals have the potential to be highly toxic or hazardous. For detailed explanations consult the MSDS:

- Cyanide
- Sulfuric acid
- Pyridine
- Chloramine-T

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

Oxidizing agents such as chlorine decompose most cyanides. Testing for chlorine must be done in the field prior to sample preservation.

6.2 Sample Preservation

Prior to preservation, samples must be tested for chlorine (Section 6.1).

Aqueous samples are preserved with 50% sodium hydroxide in the field to a pH $\geq$ 12 at the time of collection.

Samples and distillates are stored in the refrigerator at 4 ±2 °C.

6.3 Sample Handling

When properly preserved, cyanide samples are stored for up to 14 days prior to sample preparation steps.

Distillates must be analyzed within 14 days of distillation. Samples must be analyzed within 14 days of receipt.
Note: for MCP-TCN samples must be analyzed within 24h after distillation.

7. Equipment and Supplies

7.1 Cyanide Midi Distillation Unit: Lab Crest, BGL or comparable midi distillation unit. With reaction vessels, collection vessels, cold fingers and impingers.

7.2 pH paper: Range 1-14

7.3 Lead Acetate Paper

7.4 Vacuum source

7.5 50mL centrifuge tubes: New, plastic, with caps.

7.6 KI starch paper: Residual Chlorine sensitivity

7.7 Class A volumetric flasks: 25, 50, 100, 500 and 1000mL

7.8 Graduated cylinders: 50mL glass or plastic

7.9 Eppendorf pipettor or pipets: 0.5, 1, 2, and 5mL

7.10 Lachat 8000 Flow Analyzer: Including Quick Chem software, autosampler, pump and accessories.

7.11 Balance: Capable of weighing to 0.0001gram

7.12 Beakers: 100mL

7.13 Chiller

7.14 Stir plate

7.15 Stir bars

8. Standards and Reagents

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.1 Standards and Reagents for Distillation

8.1.1 Reagent Water: All references to water in this method refer to Deionized Water (DI) from Alpha's water treatment system.

8.1.2 Ascorbic Acid, $C_6H_8O_6$: Powder. Store at room temperature. Expires upon manufacturer's specified date.

8.1.3 Sodium hydroxide solution (1N), NaOH: In a 1L volumetric flask, dissolve 40g of NaOH. Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.1.4 Sulfamic acid (0.4N), H_2NSO_3H: In a 1L volumetric flask, dissolve 40g H_2NSO_3H. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.5 Sulfuric acid (1:1), H_2SO_4: To a 1L volumetric flask, add 500mL DI water. Slowly and carefully add 500mL of concentrated H_2SO_4. Store at room temperature. Expires one month from date of preparation.

8.1.6 Magnesium chloride solution (2.5M), $MgCl_2 \cdot 6H_2O$: In a 1L volumetric flask, dissolve 510g of $MgCl_2 \cdot 6H_2O$. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.7 LCS, 1000ppm cyanide stock solution: Commercially available standard with a certificate of analysis and from a different source than the Lachat calibration standards. Purchased from Ricca, Catalog # 2543-32. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.8 LCS 10ppm cyanide working solution: Pipet 1mL of 1000ppm cyanide stock solution(Section 8.1.7) into a 100mL volumetric flask. Add 10mL of 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare each day of use.

8.1.9 1000ppm Stock Spiking Solution: 1000ppm cyanide standard available commercially with a certificate of analysis. This is from a different source than the LCS (8.1.7). Purchased from LabChem Inc., Catalog # LC13545. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.10 10ppm Working Cyanide Spiking Solution: Pipet 1mL of the 1000ppm Stock Spiking Solution (Section 8.1.9) into a 100mL volumetric flask. Add 10mL 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare fresh each day of use.

8.1.11 pH 4 Acetate Buffer solution: In a 500mL volumetric flask, dissolve 410g of sodium acetate trihydrate. Bring to volume with DI water. Adjust to pH of 4.5 with acetic acid (Section 8.1.13). Store at room temperature. Expires 6 months from date of preparation.

8.1.12 Lead Carbonate Powder, [Pb (CO3)]

8.1.13 LCS 0.5 ppm Cyanide Working Solution: Pipet 5mL of the 10ppm Working Cyanide Spiking Solution (Section 8.1.10) into a 100mL volumetric flask . Add 1mL of 10N NaOH (Section 8.1.16). Bring to volume with DI water. Prepare each day of use.

8.1.14 Concentrated Acetic Acid: Store at room temperature. Expires upon manufacturer's specified date.

8.1.15 Ottawa Sand

8.1.16 Sodium hydroxide solution (10N), NaOH: In a 1L volumetric flask, dissolve 400g of NaOH. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.17 Total Cyanide SRM: ERA catalog # 541. Store in room temperature. Expires upon manufacturer's specified date.

8.1.18 Calcium Hypochlorite Solution: Dissolve 5g Ca(OCl)2 in 100mL Deionized water. Store in an amber colored bottle in the dark. Expires monthly.

8.2 Standards and Reagents for Lachat Analysis

8.2.1 Helium gas: To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat part number 50100). Bubble He vigorously through the solution for one minute.

8.2.2 Reagent 1. Carrier, 0.1N Sodium Hydroxide: In a 1L plastic container add 10mL of 10N NaOH (Section 8.1.16). Bring to 1L volume with DI. Store at room temperature. Prepare fresh bi-weekly.

8.2.3 Reagent 2. Acetate Buffer, 2.68M: In a 1L volumetric flask, dissolve 163g spdium acetate trihydrate (acetic acid, sodium salt trihydrate, $CH_3CO_2NA•H_2O$) in approximately 800mL of water. Add 40mL of acetic acid to solution. Dilute to the mark and invert to mix. Store at room temperature. Prepare fresh monthly.

8.2.4 Reagent 3. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500mL DI. Prepare fresh daily.

8.2.5 Reagent 4. Pyridine-Barbituric Acid Reagent: Under a fume hood, place 15g barbituric acid in a 1L beaker and add 100mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75mL pyridine (C_5H_5N) while stirring and mix until the barbituric acid dissolves. Add the 15mL concentrated hydrochloric acid (12M HCl) and mix. Store at room temperature. Prepare fresh weekly.

8.2.6 0.5ppm Calibration standard: Pipet 5mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.7 0.2ppm Calibration standard: Pipet 2mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.8 0.1ppm Calibration standard: Pipet 1mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use. This calibration standard is also used as the Continuing Calibration Verification sample.

8.2.9 0.04ppm Calibration standard: Pipet 5mL of the 0.2ppm calibration standard (Section 8.2.7) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.10 0.02ppm Calibration standard: Pipet 1mL of the 0.5ppm calibration standard (Section 8.2.6) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.11 0.01ppm Calibration standard: Pipet 10mL of the 0.02ppm calibration standard (Section 8.2.10) and 10mL of 0.1N NaOH into a container and mix. Prepare each day of use.

8.2.12 0.004ppm Calibration standard: Pipet 5mL of 0.04ppm calibration standard (Section 8.2.9) into a 50mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.13 0.1ppm ICV standard: Pipet 1mL of the 10ppm LCS cyanide working solution (8.1.8) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

9. Procedure

9.1 Screening for Chlorine and Sulfide Interference

9.1.1 Chlorine Interference

Oxidizing agents, such as chlorine, decompose most cyanides. Test by placing a drop of sample on a strip of potassium iodide (KI) - starch paper previously moistened with acetate buffer solution, pH 4. If positive indication is noted, then treat an aliquot of sample with Ascorbic Acid (Section 8.1.2). Repeat this test until the KI paper is negative. Immediately inform the Department Supervisor of this interference.

Manganese dioxide, nitrosyl chloride, etc., if present also may cause discoloration of the test paper.

9.1.2 Sulfide Interference
Oxidized products of sulfide convert CN- to SCN- rapidly, especially at high pH. Test for S^{-2} by placing a drop of sample on lead acetate test paper previously moistened with acetic acid buffer solution, pH 4 (Section 8.1.11). Darkening of the paper indicates presence of S^{-2}. Add powdered lead carbonate [Pb (CO3)] in 1g increments to the whole sample volume. Re-test with acetate paper. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Record in the sample prep logbook the amount of lead carbonate added to the sample.

9.2 Distillation

9.2.1 Add 50mL of shaken liquid sample, or 1gram of a well-homogenized solid sample and 50mL of DI, to the 50mL reaction vessel.

9.2.2 For the Liquid High LCS, fill one 50mL reaction vessel with 50mL DI. For the soil High LCS, add 1g Ottawa Sand (Section 8.1.15) and 50mL of DI. After the system has been charged with air, add 1mL of 10ppm LCS cyanide working solution (8.1.8) to the closed system. (Final concentration equals 0.2mg/L.)

For the Liquid Low LCS, fill one 50mL reaction vessel with 50mL of DI. For a soil Low LCS, add 0.2-0.3 g of SRM (sec 8.1.17) and 50mL of DI. Record exact SRM weight.For liquid samples: After the system has been charged with air, add 0.5mL of 10ppm LCS cyanide working solution (Section 8.1.8) to the closed system. (Final concentration equals 0.1mg/L.) **Don't add liquid Cyanide Standard for soil samples!** Final LCS soil concentration will change based on SRM lot
Samples for Method 9010C/9012B: Prepare a LCS Duplicate along with the LCSs described above.

9.2.3 For the method blank for liquid samples, fill one 50mL reaction vessel with 50mL of DI. For the method blank for soil samples, fill a 50mL reaction vessel with 1g of Ottawa Sand (Section 8.1.15) and 50mL DI.

9.2.4 For the matrix spike, fill a 50mL reaction vessel with 50mL of sample that has been chosen to be spiked. For soil samples, use 1g of soil and add 50mL of DI water. After the system has been charged with air, add 1mL of 10ppm working cyanide spiking solution (8.1.10) to the closed system.

Samples for Method 9010C/9012B: Prepare a Matrix Spike Duplicate (MSD) in the same manner as the MS, as described above.

9.2.5 For the duplicate, fill a 50mL reaction vessel with a duplicate aliquot of 50mL, or 1g soil and 50mL DI water of a sample that has been chosen to be duplicated.

9.2.6 Into the receiver or scrubber tube add 5mL of a 1N NaOH solution and add 40mL of DI water.

9.2.7 Arrange tubes in the distillation unit noting in the logbook which sample is in which glassware. The glassware is numbered and consistently placed in the same position in the distillation unit.

9.2.8 Assemble the unit completely. Turn on the pump. There must be gas bubbling in each tube. Check to make sure all connections are tight and bubbles are flowing at an equal rate in each sample tube. If not, adjust flow rate with the knobs in front of each receiver tube and/or check lines to ensure they are not obstructed.

9.2.9 Add 5mL of 0.4N sulfamic acid (8.1.4) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left of the vessel wall.

9.2.10 Add 5mL of 1:1 H_2SO_4 (8.1.5) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left on the vessel wall. Turn on the heat. Samples are to come to a boil on all of the midi-still units.

9.2.11 After 2 minutes of heating, add 2mL of 2.5M $MgCl_2$ Solution (8.1.6) to each sample tube, followed by a rinse with DI. If foaming occurs, an additional 2mL of $MgCl_2$ Solution may be added. If foaming continues, stop the distillation for that sample and reduce the sample size by 2 – 5x (as determined by the severity of the foaming). Contact the Inorganics Supervisor for guidance.

9.2.12 Turn on the chiller.

9.2.13 Set the timer-dial on the distillation unit to “110”.

9.2.14 After 110 minutes the unit will shut off; leave the chiller running for an additional 30 minutes while the tubes cool down.

9.2.15 Pour contents of the scrubber tube into a new, labeled, centrifuge tube (Section 7.5). Carefully rinse the scrubber tube with DI water and add rinseate to the centrifuge tube to bring to 50mL volume. Cap and refrigerate for later analysis by the Lachat Instrument.

9.3 Initial Calibration of Lachat Instrument

9.3.1 Allow 15 minutes for heating unit to warm up to 60 °C.

9.3.2 Prepare a series of 7 calibration standards (Sections 8.2.6 – 8.2.12) and a 0.1N NaOH blank. Alternatively, calibration standards may be prepared by auto-diluting a 0.5ppm calibration standard (Section 8.2.6). Perform this function per the Lachat manufacturer’s instructions for the Quick Chem 8000.

9.3.3 Set up manifold as shown in Table 1.

9.3.4 Input data system parameters as shown in Table 2.

9.3.5 Place standards and blank in the autosampler, per the manufacturer’s instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

9.3.6 Inject the standards, per the manufacturer’s instructions.

9.3.7 Prepare a standard curve by plotting instrument response against standard concentration values. A calibration curve is fitted to the calibration solution concentration/response data using the computer. The calibration coefficient of the curve must be greater than or equal to 0.995 before sample analysis can begin.

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

9.4 Standardization (Continuing Calibration Verification)

9.4.1 After the calibration has been established, it must be verified by the analysis of an Initial Calibration Verification Standard (ICV) (Section 8.2.13). The ICV of 0.1ppm must be made from a different source than the calibration standards. If the measurements exceed ±10% of 0.1ppm, the analysis is terminated. See Section 10.6 for Corrective Actions.

9.4.2 A Blank and a Continuing Calibration Verification (CCV) sample (Section 8.2.8) are analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the CCV value of 0.1ppm and the blank result must be less than the reporting limit of 0.005 mg/L. See Section 10.6 for CCV Corrective Actions and Section 10.2 for Blank corrective actions.

9.5 Lachat Analysis

9.5.1 Following initial calibration and standardization, (Section 9.3 and 9.4), place the samples in the autosampler, per the manufacturer's instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

9.5.2 Inject the samples, per the manufacturer's instructions.

9.5.3 The data system calculates sample concentration using the regression equation. Results are mg/L for Aqueous samples and mg/Kg for soil and solid samples.

9.5.4 If sample concentrations are greater than the highest calibration standard, the distilled sample is diluted with 0.1N sodium hydroxide (NaOH) diluent (Section 8.2.2), and reanalyzed. When the automated diluter is used, 0.1N NaOH is also used. **Do not dilute distilled samples or standards with DI water.**

9.6 Preventative Maintenance

Preventative maintenance is recorded in the instrument maintenance logbook and is performed on the Lachat instrument as follows:

Daily:

1) Clean the autosampler
2) Clean the surfaces on the auto-dilutor
3) Prime the dilutor with fresh DI water
4) Clean the pump surfaces
5) Clean the detector with DI and dry with Kim-Wipes
6) Clean the instrument surfaces with DI, wipe clean with a paper towel

Bi-weekly:

1) Clean the injection ports with DI. Take apart the injection valve and inspect it for corrosion. Make sure that the valve connectors are tight and the o-rings are not worn. If the O-rings look worn replace with new ones.
2) Perform a scan disk and disk de-fragmentation on the computer.

Monthly:

1) Using DI water, clean the unions and the tees that are associated with the manifold.
2) Delete Temporary files on the computer, and clear the hard drive of all unnecessary files.

Every 6 months:

1) Replace the o-rings in the injection valve.
2) Replace the o-rings in the manifold
3) Back up the files on the computer. Delete backed files on the hard drive.

9.7 Calculations

9.7.1 The Lachat data system calculates sample concentration using the regression equation.

9.7.2 Report only those values that fall between the lowest and the highest calibration standards.

9.7.3 Report results in mg CN/L for liquids and in mg CN/kg for soils.

9.8 Amenable Cyanide Prep

9.8.1 Add 25mL, or 1g and 25mL of DI (8.1.1), to a 100mL beaker with a stir bar.

9.8.2 Add 1mL of 10ppm LCS cyanide working solution (8.1.8) to beaker.

9.8.3 Prep one sample in duplicate.

9.8.4 Add 1-2mL of calcium hypochlorite solution (8.1.18) to all samples and QC under the hood.

9.8.5 Check for the presence of chlorine by placing a drop of the sample on a KI-starch paper (7.6). It should turn blue if there is sufficient chlorine.

9.8.6 Check samples every 15 minutes for one hour for the presence of chlorine and add more calcium hypochlorite if needed.

9.8.7 After one hour of digesting, add ascorbic acid (8.1.2) until KI strip no longer turns blue.

9.8.8 Bring sample volume up to 50mL and distill following steps 9.2.6 thru 9.2.15.

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

Refer to Alpha SOP/ 1734 and 1739 for DOC information.

10.2 Blank

A minimum of one method blank is distilled and analyzed per batch of 20 or less samples. The Method Blank is utilized to determine if contamination or any memory effects are occurring. (Section 9.2.1) The blank result must be less than the reporting limit of 0.005 mg/L for liquids and 1 mg/kg for soils. If the blank result is outside of acceptance criteria, it is injected another time. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable blank analysis must be reanalyzed.

10.3 Laboratory Control Samples (LCS) / Laboratory Control Sample Duplicate (LCSD)

Distill and analyze two LCSs per batch of 20 samples. A Low LCS is analyzed at 0.1mg/L and a high LCS is analyzed at 0.2mg/L. (Section 9.2.2)

LCS measurements for Method SM 4500 CN-CE must be within ±10%. For Method 9010C/9012B, the LCS measurements must be within ± 15% for liquids and +/- 20% for soils.

Samples for Method 9010C/9012B: LCSDs are distilled and analyzed along with the LCSs, as described above. The RPD between LCS and LCSD must be ≤ 20% for liquids and ≤35% for soils.
For soil samples: LCS and LCSD recovery must be within vendor specified acceptance criteria (it will be different for different lots of SRM)

If any LCS fails acceptance criteria for either % Recovery or RPD, analysis is terminated and samples are redigested and analyzed.

10.4 Matrix Spike

Distill and analyze one spike per batch of 20 samples. For Method 9010C/9012B distill and analyze one spike per batch of 10 samples.
For Method 9010C/9012B, the % Recovery must be within ±20% for liquids and ±35% for solids. For SM 4500CN-CE, the % Recovery must be within ±10%. (Section 9.2.4).

Samples for Method 9010C/9012B: A Matrix Spike Duplicate (MSD) is distilled and analyzed along with the MS, as described above. The RPD between MS and MCSD must be ≤ 20% for liquids and ≤35% for soils.

10.5 Duplicates

Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. (Section 9.2.5)

The RPD must be 20% or less for liquids and 35% or less for soils and solids. See Section 12 for Corrective Action if these criteria are not met.

10.6 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The Initial Calibration Verification Standard (ICV) (Section 8.2.13) is analyzed immediately following the calibration to verify the curve. If the measurements exceeds ±10% of 0.1ppm, the analysis is terminated and recalibration must occur. An acceptable result for the ICV must be obtained prior to any sample analysis.

The Continuing Calibration Verification Standard (CCV) (Section 8.2.8) is analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the of 0.1ppm. If the CCV is not within acceptance criteria, the standard is injected again. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable calibration verification must be reanalyzed.

10.7 Control Limits

Refer to SOP/ 1734.

10.8 Analytical Sequence

The analytical sequence is:

- Screening of samples for chlorine and sulfide
- Prep of amenable cyanide, if needed
- Distillation:
    - Samples
    - LCS Low
    - LCS High
    - Blank
    - Matrix Spike
    - Duplicate
- Analysis:
    - Calibration and Standardization of Lachat Instrument
    - CCV
    - CCB
    - ICV
    - ICB
    - 10 samples
    - CCV
    - CCB
    - 10 samples
    - CCV

- CCB
- Calculation of sample cyanide concentration

11. Method Performance

Refer to SOP/ 1732 for MDL/LOD/LOQ information. Refer to SOP/ 1734 and 1739 for DOC information.

12. Corrective Actions

Holding time exceedence and improper preservation are noted on the nonconformance report form. The analyst narrates the nonconformance when the project is turned in for review. The narration must state what the nonconformance was and any corrective action taken.

Perform routine preventative maintenance according to Section 9.6. Record all maintenance in the instrument logbook. Notify the Department Manager if the instrument problems are not routine in nature. The Department Manager determines whether the problem can be corrected with in-house technical staff or if the instrument vendor should be contacted to schedule service. All service calls are documented in the Instrument logbook, and a copy of the service report is given to the Department Manager.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. If any part of batch quality control does not meet acceptance criteria, the Department Manager is notified. If enough sample remains and holding time has not expired, then the batch is redistilled and reanalyzed. If there is not sufficient sample remaining to allow redistillation, then that analysis is repeated and both sets of data are reported with the nonconformance narrated on the final report.

If either the ICV, ICB, Method Blank, LCS, LCSD, CCV, or CCB recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected. The CCV/CCB standard is remade and reanalyzed. If the standard passes, all samples analyzed since the previous passing standard are reanalyzed. The raw data is noted and all data associated with the failing standard must have one line drawn through the data, indicating its unusability.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all samples analyzed since the previous passing CCV/CCB are reanalyzed.

If following reanalysis of the LCS, it is found to still be outside acceptance criteria, the entire sample batch must be redistilled and reanalyzed. If the %RPD between the LCS/LCSD fails after reinjection, then the entire sample batch must be redistilled and reanalyzed.

If the Method Blank fails it is re-poured and reinjected. If failure continues, the associated sample data is evaluated as follows: Sample results below the detection limit may be reported with a narrative included. If samples have positive results, and the results are greater than 10x the concentration found in the method blank, the data may be reported with a narrative included. Any positive samples with results less than 10x the concentration found in the method blank must be redistilled and reanalyzed.

If the Matrix Spike recovery does not meet acceptance criteria, and the LCS recovery is acceptable, matrix interference may be assumed. The associated data may be reported with a narrative included.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are redistilled and reanalyzed within holding time.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

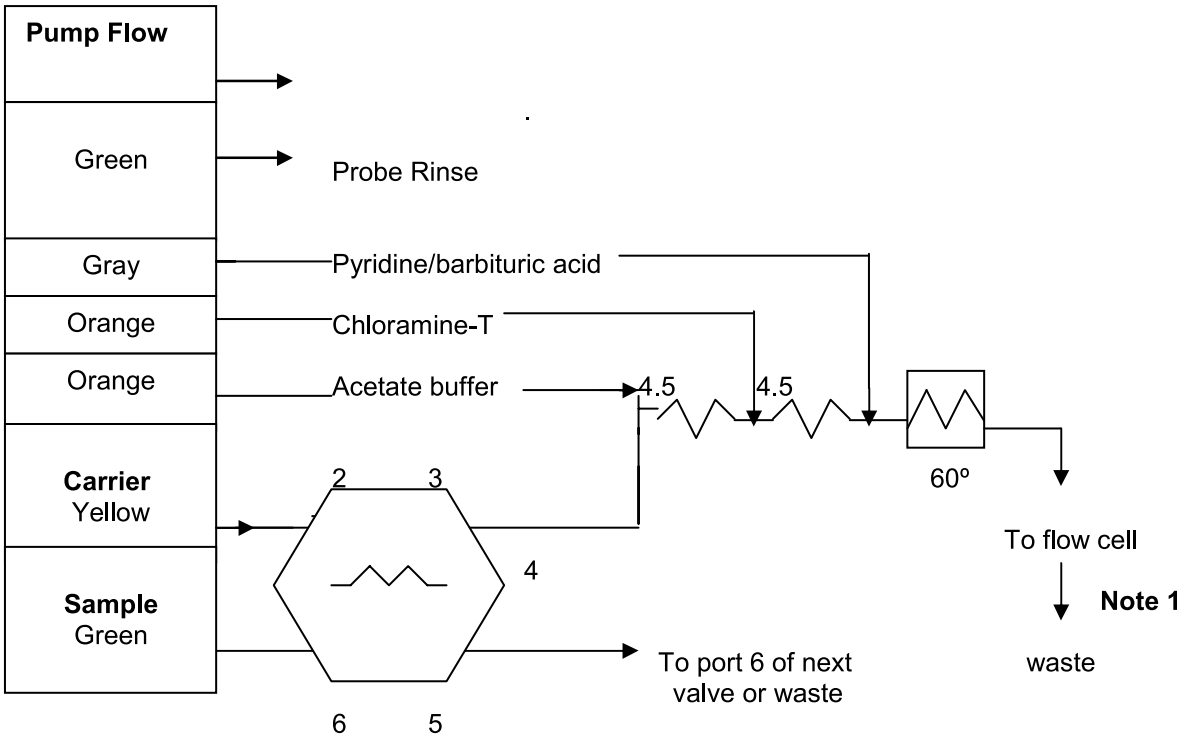
See Chemical Hygiene Plan for waste handling and disposal.

15. Attachments

TABLE 1: Cyanide Manifold Diagram

TABLE 2: Data System Parameters for QC 8000

TABLE 1
Cyanide Manifold Diagram



Sample Loop = 150cm x 0.8mm i.d. Interference Filter = 570nm
QC8000 Sample loop = 150cm x 0.8mm i.d.

CARRIER is 0.1 N sodium hydroxide solution.

All manifold tubing is 0.8mm (0.030 in) i.d. This is 5.2µL/cm.

4.5 is 70.0cm of tubing on a 4.5cm coil support

APPARATUS: An injection valve, flow cell, a 10mm path length flow cell, and a colorimetric detector module are required.

The box  shows 650cm of tubing wrapped around the heater block at the specified temperature.

Note 1: 2 meter back pressure loop, 0.52mm i.d.

TABLE 2
Data system Parameters for QC 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample Throughput: 80 samples/hour, 45 s/sample
Pump Speed: 35
Cycle Period: 45

Analyte Data:

Concentration Units: mg CN^-/L
Peak Base Width: 39 s
% Width Tolerance: 100
Threshold: 25000
Inject to Peak Start: 42 s
Chemistry: Direct

Calibration Data:

Levels	1	2	3	4	5	6	7	8
Concentration ug/50mL	25	10	5	2	1	0.5	0.2	0

Calibration Fit Type: 1st Order Polynomial
Calibration Rep. Handling: Replace
Weighting Method: 1/X
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:

Min. Probe in Wash Period: 14 s
Probe in Sample Period: 20 s

Valve Timing:

Load Time: 0.0 s
Load Period: 20 s
Inject Period: 25 s

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, 2006.

Method 5035A, Closed System Purge &Trap and Extraction for Volatile Organics in Soil and Waste Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, Draft, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December, 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May, 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The following compounds may be determined by this method:

8260C LIST OF ANALYTES		
Dichlorodifluoromethane	Carbon tetrachloride	Isopropylbenzene
Chloromethane	1,2-Dichloroethane	1,4-Dichloro-2-butane
Vinyl chloride	Benzene	1,1,2,2-Tetrachloroethane
Chloroethane	Trichloroethene	Trans-1,4-dichloro-2-butene
Bromomethane	1,2-Dichloropropane	1,2,3-Trichloropropane
Trichlorofluoromethane	Bromodichloromethane	n-Propylbenzene
Ethyl ether	Dibromomethane	Bromobenzene
Acetone	4-Methyl-2-pentanone	2-Chlorotoluene
1,1-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
Carbon disulfide	Toluene	4-Chlorotoluene
Methylene chloride	Trans-1,3-dichloropropene	Tert-butylbenzene
Acrylonitrile	Ethyl-methacrylate	1,2,4-Trimethylbenzene
Methyl-tert-butyl ether	1,1,2-Trichloroethane	Sec-butylbenzene
Trans-1,2-dichloroethene	2-Hexanone	p-Isopropyltoluene
1,1-Dichloroethane	1,3-Dichloropropane	1,3-Dichlorobenzene
Vinyl acetate	Tetrachloroethene	1,4-Dichlorobenzene
2-Butanone	Chlorodibromomethane	n-Butylbenzene
2,2-Dichloropropane	1,2-Dibromoethane	1,2-Dichlorobenzene
Cis-1,2-dichloroethene	Chlorobenzene	1,2-Dibromo-3-chloropropane
Chloroform	1,1,1,2-Tetrachloroethane	1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl benzene	Hexachlorobutadiene
Tetrahydrofuran	p/m Xylene	Naphthalene
1,1,1-Trichloroethane	o Xylene	1,2,3-Trichlorobenzene

8260C LIST OF ANALYTES (continued)		
1,1-Dichloropropene	Styrene	Bromoform
Acrolein	2-Chloroethylvinyl ether	Ethanol
Cyclohexanone	Ethyl acetate	1,3,5-Trichlorobenzene
Iodomethane	Methyl methacrylate	Tert-amyl methyl ether
Di-isopropyl ether	n-Butanol	1,4-Dioxane
Ethyl Tert-Butyl Ether	Pentachloroethane	Isopropyl Alcohol (IPA)
Hexane		

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of

the analysis. Running laboratory reagent blanks as described in Section 10.3 and 9.1 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally all affected samples are rerun for confirmation.. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab, for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below, but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures. The Lab will analyze the high level sample first, followed by the low level sample if the results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol, and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.1.2.2.2 and 11.1.2.2.3)

6.1.2.2 Low Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedence, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples requiring aromatic analysis must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples and High Level Soils): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (Varian Archon/8100, Tekmar Solatek, EST Centurion) coupled to the desorber (concentrator) (Tekmar Velocity or EST Encon).

7.1.1 Purge gas = Helium, analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the helium purge gas is introduced at the bottom of the water column as finely divided bubbles

7.1.3 The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260^{o}C. The trap is not heated above manufacturer's specifications

7.2. Purge and Trap System (For Low Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) coupled to the desorber (concentrator) (Varian Archon/8100, Tekmar Solatek, EST Centurion with EST Encon, Tekmar Velocity, or equivalents).

7.2.1. Purge gas = Helium, analytical grade (99.999%).

7.2.2. The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the helium purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3. The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.

7.2.4. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, or equivalent.
7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, or equivalent

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition, and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

Thruput Target 4.12 software or Enviroquant E.02.02 (or equivalent) is used for data processing, and allows searching of any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / Enviroquant data system.

7.4 Wiretrol or Microsyringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.1g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf Pipets

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in the beginning of this SOP. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 initial calibration standard. This standard is made from a second source than the Initial Calibration Standards.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Blank samples must be matrix specific, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed; TCLP samples need a TCLP blank.

Analyze a matrix-specific blank each day prior to sample analysis to demonstrate that interferences from the analytical system are under control. The blank must contain the internal standards and surrogates.

Analyze the reagent water blank from the same source of water used for preparing the standards, QC samples and making sample dilutions. The method blank must not contain any target analytes at or above the compound reporting limits.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

A LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.4.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run anytime after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium, 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass scan range:	35 – 260 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	1 minute
Desorb preheat:	245°C
Desorb temp:	255°C
Desorb time:	1 minute
Bake temp:	270°C
Bake time:	10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level of the either at or below the reporting limit. The calibration is performed using instrument conditions listed in Section 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards, and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the ThruPut Target / Enviroquant software.

10.2.1.1 **Low Level/High Level Soil Curve on Archon or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto Archon Autosampler.

10.2.1.2 **Aqueous/High Level Soil Curve on Solatek or Centurion:** To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a microsyringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 40mL VOA vial to the top. Load the vial onto the Autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP Enviroquant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the Target / Enviroquant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the Target / Enviroquant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 **Initial Calibration Criteria:** The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed.

10.2.3.1 **Relative Standard Deviation Criteria:** If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to

quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if **r** ≥ 0.990, weighted linear with a weighting factor of 1/SD2 and r > 0.990, or quadratic fit if $\mathbf{r^2}$ ≥ 0.995. A minimum of six points is required and the low point of the calibration must be re-quantitated and recover within 70-130% to be deemed acceptable. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.2 Minimum Response Factors: Table 1 lists the suggested minimum response factors for the most common analytes. Each calibration level must be evaluated against the specified criteria. Analytes that fall below the criteria, but are greater than or equal to 0.05, are narrated for inclusion on the final report. There are certain very poor purgers (1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds) that should meet a 0.001 response factor. If an analyte falls below 0.05 (or 0.001 for 1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds), then corrective action must be taken to resolve the problem before analysis can proceed.

10.2.4 Evaluation of Retention Times: The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 Initial Calibration Verification: After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes except for “difficult” analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 Analysis of Samples

Retrieve sample VOA vials from the sample bank refrigerator just prior to loading onto the purge and trap system. High level soil samples must be shaken for 1 – 2 minutes to extract the volatile components into the methanol. Let sample settle prior to taking methanol aliquot. Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the Archon or Centurion unit.

10.3.1.1 Low level soil samples: (Archon or Centurion)
Take the low level VOA vial and place directly into the rack of the Archon sampling unit. Surrogate and internal standards are added automatically by the Archon prior to sample purging.

10.3.1.2 Aqueous samples: (Solatek or Centurion)

Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The auto-sampler will then sample 10mL from the VOA vial.

10.3.1.3 High level soil samples: (Archon/Solatek/Centurion)

Shake for 2 minutes, ensuring the methanol has completely penetrated the soil in the vial.

10.3.1.3.1 Through liquid path

Load a maximum of 430µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.1.3.2 Through soil path

Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.2 Qualitative Analysis:

10.3.2.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.2.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The Target / Enviroquant data system is configured to make this check.

10.3.2.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.2.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.2.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

10.3.2.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.3.2.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.3.2.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as "tentative", and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3 Quantitative Analysis:

10.3.3.1 Quantitation of a target compound detected in a sample is performed automatically by the Target / Enviroquant data processing software, using the formulae found in Alpha's Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve.

If non-target compounds are to be reported, the quantitation is performed automatically by the Target / Enviroquant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 The continuing calibration standard must also be evaluated for the suggested minimum response factor criteria, as specified in section 10.2.3.2

10.4.6 Internal Standard Retention Time:

The retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.7 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.1.2 Data Calculations

11.1.2.1 Results of Aqueous Sample Analysis:

$$concentration\ (ug/L) = \frac{(Conc.)\ (Vp)\ (DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.1.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.1.2.2.1 Low-Level Samples:

$$concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (DF)}{(W)\ (\%S)}$$

11.1.2.2.2 High-Level Samples:

$$concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (5000)\ (DF)}{(W)\ (Ve)\ (\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.1.2.2.3 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\% \text{ moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

$$V_t = \frac{[\text{mL of solvent} + (\%\text{moisture} \times \text{g of sample})]}{100} \times 1000\text{mL/mL}$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$\text{Corrected concentration (mg/Kg)} = \frac{(\text{Conc.})\,(V_t + \text{methanol vol.})\,(Vp)\,(DF)}{(W)\,(Ve)\,(\%S)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: 8260 REPORTING LIMITS

TABLE 2: 8260 QC ACCEPTANCE CRITERIA

TABLE 3: BFB TUNING CRITERIA

TABLE 4: STANDARD SOLUTIONS

TABLE 5: 8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 6: 8260C Quantitation Ions

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)[1]	RDL (µg/KG)[2]
Acetone [3,4,5]	0.100	5.0	10	250
Acrolein [5]		5.0	25	1250
Acrylonitrile [3,4]		5.0	5	200
Benzene [3,4,5]	0.500	0.5	1	50
Bromobenzene [3,4]		2.5	5	250
Bromochloromethane [3,4,5]		2.5	5	250
Bromodichloromethane [3,4,5]	0.200	0.5	1	50
Bromoform [3,4,5]	0.100	2.0	4	200
Bromomethane [3,4,5]	0.100	1.0	2	100
2-Butanone [3,4,5]	0.100	5.0	10	500
n-Butyl benzene [3,4]		0.5	1	50
sec-Butyl benzene [3,4]		0.5	1	50
tert-Butyl benzene [3,4]		2.5	5	250
Carbon disulfide [3,4,5]	0.100	5.0	10	500
Carbon tetrachloride [3,4,5]	0.100	0.5	1	50
Chlorobenzene [3,4,5]		0.5	1	50
Chloroethane [3,4,5]	0.100	1.0	2	100
2-Chloroethylvinyl ether [3]		10.0	20	1000
Chloroform [3,4,5]	0.200	0.75	1.5	75
Chloromethane [3,4,5]	0.100	2.5	5	250
o-Chlorotoluene [3,4]		2.5	5	250
Cyclohexane [5]	0.100	10	20	1000
Cyclohexanone		10	20	1000
p-Chlorotoluene [3,4]		2.5	5	250
Dibromochloromethane [3,4,5]	0.100	0.5	1	50
1,2-Dibromo-3-chloropropane [3,4,5]	0.050	2.5	5	250
1,2-Dibromoethane [3,4,5]	0.100	2.0	5	250
Dibromomethane [3,4]		5.0	10	500
1,2-Dichlorobenzene [3,4,5]	0.400	2.5	5	250
1,3-Dichlorobenzene [3,4,5]	0.600	2.5	5	250
1,4-Dichlorobenzene [3,4,5]	0.500	2.5	5	250
1,4-Dichlorobutane [3,4]		5.0	10	500
trans-1,4-Dichloro-2-butene [3,4]		2.5	5	250
Dichlorodifluoromethane [3,4,5]		5.0	10	500
1,1-Dichloroethane [3,4,5]	0.200	0.75	1.5	75
1,2-Dichloroethane [3,4,5]	0.100	0.5	1	50
1,1-Dichloroethene [3,4,5]	0.100	0. 5	1	50
cis-1,2-Dichloroethene [3,4,5]	0.100	0.5	1	50
trans-1,2-Dichloroethene [3,4,5]	0.100	0.75	1.5	75

Table 1 (continued)

Standard Reported Detection Limits

US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)[1]	RDL (µg/KG) [2]
1,2-Dichloropropane [3,4,5]	0.100	1.75	3.5	175
1,3-Dichloropropane [3,4]		2.5	5	250
2,2-Dichloropropane [3,4]		2.5	5	250
1,1-Dichloropropene [3,4]		2.5	2.5	250
cis-1,3-Dichloropropene [3,4,5]	0.200	0.5	1	50
p-Diethylbenzene [4]		2.0	4	200
Diisopropyl Ether [6]		2.0	4	200
1,4-Dioxane [5] (non-SIM)		250	100	5000
trans-1,3-Dichloropropene [3,4,5]	0.200	0.5	1	50
Ethanol [7]		N/A	1000	50000
Ethyl acetate		10.0	20	1000
Ethylbenzene [3,4,5]	0.100	0.5	1	50
Ethyl ether [3,4]		2.5	5	250
4-Ethyltoluene [4]		2.0	4	200
Ethyl methacrylate [3,4]		5.0	10	500
Ethyl-Tert-Butyl-Ether [6]		2.0	4	200
Freon-113 [5]		10.0	20	1000
Hexachlorobutadiene [3,4]		0.5	5	250
Hexane		1.0	1.0	50
2-Hexanone [3,4,5]	0.100	5.0	10	500
Iodomethane		5.0		
Isopropyl Alcohol (IPA)		25		
Isopropylbenzene [3,4,5]	0.100	0.5	1	50
p-Isopropyltoluene [3,4]		0.5	1	50
Methyl Acetate [5]	0.100	20	20	1000
Methylene chloride [3,4,5]	0.100	3.0	10	500
Methyl Cyclohexane [5]	0.100	20	4	200
Methyl Methacrylate		1.0		
4-Methyl-2-pentanone [3,4,5]	0.100	5.0	10	500
Methyl-tert-butyl-ether [3,4,5]	0.100	1.0	2	100
Naphthalene [3,4]		2.5	5	250
n-Butanol [5]		100	200	10000
n-Propylbenzene [3,4]		0.5	1	50
Pentachloroethane		2.0	N/A	N/A
Styrene [3,4,5]	0.300	1.0	2	100
Tert-Butyl Alcohol [5]		30	100	5000
Tertiary-Amyl Methyl Ether [6]		2.0	4	200
1,1,1,2-Tetrachloroethane [3,4]		0.5	1	50

1,2,4,5-Tetramethylbenzene (4)		2.0	4	200
Analyte	**Recommended Minimum Response Factor**	**RDL (µg/L)**	**RDL(µg/KG)(1)**	**RDL(µg/KG)(2)**
1,1,2,2-Tetrachloroethane (3,4,5)	0.300	0.5	1	50
Tetrachloroethene (3,4,5)	0.200	0.5	1	50
Tetrahydrofuran (3)		10.0	20	1000
Toluene (3,4,5)	0.400	0.75	1	75
1,2,3-Trichlorobenzene (3,4,5)		2.5	5	250
1,2,4-Trichlorobenzene (3,4,5)	0.200	2.5	5	250
1,3,5-Trichlorobenzene (6)		2.0	5	250
1,1,1-Trichloroethane (3,4,5)	0.100	0.5	1	50
1,1,2-Trichloroethane (3,4,5)	0.100	0.75	1.5	75
Trichloroethene (3,4,5)	0.200	0.5	1	50
Trichlorofluoromethane (3,4,5)	0.100	2.5	5	250
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	5	250
1,3,5-Trimethylbenzene (3,4)		2.5	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.100	1.0	2	100
m/p-Xylenes (3,4,5)	0.100	1.0	2	100
o-Xylene (3,4,5)	0.300	1.0	2	100
1,4-Dioxane (5) SIM		3.0		
1,1,2,2-Tetrachloroethane SIM		0.1		

(1) Detection Limits are for Low-level Aqueous preserved samples.
(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.

Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for New York and New Jersey reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Acetone	2000	0.5	2	10	20	30	50	100	200
Acrolein	2000	0.5	2	10	20	30	50	100	200
Acrylonitrile	2000	0.5	2	10	20	30	50	100	200
Benzene	2000	0.5	2	10	20	30	50	100	200
Bromobenzene	2000	0.5	2	10	20	30	50	100	200
Bromochloromethane	2000	0.5	2	10	20	30	50	100	200
Bromodichloromethane	2000	0.5	2	10	20	30	50	100	200
Bromoform	2000	0.5	2	10	20	30	50	100	200
Bromomethane	2000	0.5	2	10	20	30	50	100	200
2-Butanone	2000	0.5	2	10	20	30	50	100	200
n-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
sec-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
tert-Butyl benzene	2000	0.5	2	10	20	30	50	100	200
Carbon disulfide	2000	0.5	2	10	20	30	50	100	200
Carbon tetrachloride	2000	0.5	2	10	20	30	50	100	200
Chlorobenzene	2000	0.5	2	10	20	30	50	100	200
Chloroethane	2000	0.5	2	10	20	30	50	100	200
2-Chloroethylvinyl Ether	2000	0.5	2	10	20	30	50	100	200
Chloroform	2000	0.5	2	10	20	30	50	100	200
Chloromethane	2000	0.5	2	10	20	30	50	100	200
o-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
p-Chlorotoluene	2000	0.5	2	10	20	30	50	100	200
Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Cyclohexanone	2000	0.5	2	10	20	30	50	100	200
Dibromochloromethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromo-3-chloropropane	2000	0.5	2	10	20	30	50	100	200
1,2-Dibromoethane	2000	0.5	2	10	20	30	50	100	200
Dibromomethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,4-Dichlorobutane	2000	0.5	2	10	20	30	50	100	200
trans-1,4-Dichloro-2-butene	2000	0.5	2	10	20	30	50	100	200
Dichlorodifluoromethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
cis-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
trans-1,2-Dichloroethene	2000	0.5	2	10	20	30	50	100	200
1,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,3-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
2,2-Dichloropropane	2000	0.5	2	10	20	30	50	100	200
1,1-Dichloropropene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
cis-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
trans-1,3-Dichloropropene	2000	0.5	2	10	20	30	50	100	200
p-Diethylbenzene	2000	0.5	2	10	20	30	50	100	200
Diisopropyl Ether	2000	0.5	2	10	20	30	50	100	200
1,4-Dioxane (non-SIM)	10000	100	400	1000	2000	3000	4000	5000	6000
Ethanol	10000	100	200	300	500	1000	2500	5000	N/A
Ethyl Acetate	2000	0.5	2	10	20	30	50	100	200
Ethylbenzene	2000	0.5	2	10	20	30	50	100	200
Ethyl ether	2000	0.5	2	10	20	30	50	100	200
Ethyl methacrylate	2000	0.5	2	10	20	30	50	100	200
Ethyl Tert-Butyl Ether	2000	0.5	2	10	20	30	50	100	200
4-Ethyltoluene	2000	0.5	2	10	20	30	50	100	200
Freon-113	2000	0.5	2	10	20	30	50	100	200
Halothane	2000	0.5	2	10	20	30	50	100	200
Hexachlorobutadiene	2000	0.5	2	10	20	30	50	100	200
2-Hexanone	2000	0.5	2	10	20	30	50	100	200
Hexane	2000	0.5	2	10	20	30	50	100	200
Iodomethane	2000	0.5	2	10	20	30	50	100	200
Isopropyl Alcohol (IPA)	10000	2.5	10	50	100	150	250	500	1000
Isopropylbenzene	2000	0.5	2	10	20	30	50	100	200
p-Isopropyltoluene	2000	0.5	2	10	20	30	50	100	200
Methyl Acetate	2000	0.5	2	10	20	30	50	100	200
Methylene Chloride	2000	0.5	2	10	20	30	50	100	200
Methyl Cyclohexane	2000	0.5	2	10	20	30	50	100	200
Methyl Methacrylate	2000	0.5	2	10	20	30	50	100	200
4-Methyl-2-pentanone	2000	0.5	2	10	20	30	50	100	200
Methyl-tert-butyl-ether	2000	0.5	2	10	20	30	50	100	200
Naphthalene	2000	0.5	2	10	20	30	50	100	200
n-Butanol	5000	2.5	10	50	100	150	250	500	N/A
n-Propylbenzene	2000	0.5	2	10	20	30	50	100	200
Pentachloroethane	1000	0.5	2	10	20	30	50	100	200
Styrene	4000	1	4	20	40	60	100	200	400
Tert-Butyl alcohol	10000	2.5	10	50	100	150	250	500	1000
Tertiary-Amyl Methyl Ether	2000	0.5	2	10	20	30	50	100	200
1,1,1,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane	2000	0.5	2	10	20	30	50	100	200
Tetrachloroethene	2000	0.5	2	10	20	30	50	100	200
Tetrahydrofuran	2000	0.5	2	10	20	30	50	100	200
1,2,4,5-Tetramethylbenzene	2000	0.5	2	10	20	30	50	100	200
Toluene	2000	0.5	2	10	20	30	50	100	200

1,2,3-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200

Table 4 (continued)

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Target Compound	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
1,2,4-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trichlorobenzene	2000	0.5	2	10	20	30	50	100	200
1,1,1-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
1,1,2-Trichloroethane	2000	0.5	2	10	20	30	50	100	200
Trichloroethene	2000	0.5	2	10	20	30	50	100	200
Trichlorofluoromethane	2000	0.5	2	10	20	30	50	100	200
1,2,3-Trichloropropane	2000	0.5	2	10	20	30	50	100	200
1,2,4-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
1,3,5-Trimethylbenzene	2000	0.5	2	10	20	30	50	100	200
Vinyl acetate	2000	0.5	2	10	20	30	50	100	200
Vinyl chloride	2000	0.5	2	10	20	30	50	100	200
m/p-Xylenes	4000	1	4	20	40	60	100	200	400
o-Xylene	4000	1	4	20	40	60	100	200	400
1,4-Dioxane (SIM)	100	0.5	2	10	20	30	50	100	200
1,1,2,2-Tetrachloroethane (SIM)		0.05	0.1	0.2	0.5	1.0	2.0	5.0	10.0

Target Compounds	Stock (µg/mL)	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Internal Standards									
Fluorobenzene	2500	10	10	10	10	10	10	10	10
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10
Surrogates									
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

TABLE 5

8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile		p-isopropyltoluene
Methyl Tert Butyl Ether		1,3-dichlorobenzene
Halothane		1,4-dichlorobenzene
Trans-1,2-dichloroethene		n-butylbenzene
Diisopropyl Ether		p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		1,3,5-Trichlorobenzene
1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		
1,1-dichloropropene		
Carbon Tetrachloride		
Tertiary-Amyl Methyl Ether		
1,2-dichloroethane-d4 (surr)		
1,2-dichloroethane		
Benzene		
Trichloroethene		
Methyl Cyclohexane		
1,2-dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
Cis-1,3-dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl Alcohol (IPA)		
Hexane		

TABLE 6
8260C Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-tetramethylbenzene	119
Trichloroethene	95	1,2-dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-trichlorobenzene	180
1,2-dichloropropane	63	1,2,4-trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

TABLE 6
8260C Quantitation Ions (continued)

Analyte	Quantiation Ion	Analyte	Quantiation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45
Hexane	57		

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
naphthalene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: **EPA 8270 D**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists “difficult” compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953.

4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2ºC.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 **Gas Chromatograph, Hewlett Packard 6890 (or equivalent):** An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 **Column:** Rxi-5Sil MS30m x 0.32mm ID, 0.25µm film thickness or column of similar configuration.

7.1.3 **Mass Spectrometer, Hewlett Packard 5973 (or equivalent):** Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 **Data System:** A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *MSD Chemstation/Enviroquant version E.02.02* is used for data reduction.

7.2 Syringe: 10 µL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer.

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Vendor	Standard	Catalog No.	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/ml
	605 Benzidines Mix	31030	2000ug/ml
	Benzoic Acid Mix	31879	2000ug/ml
	Acid Surrogate Mix	31025	2000ug/ml
	Acid Surrogate Mix	31087	10000ug/ml
	BN Surrogate Mix	31024	1000ug/ml
	B/N Surrogate Mix	31086	5000ug/ml
	Custom SV Standard	562843	2000ug/ml
	Custom ABN Addition Standard	567302	2000ug/ml
	Benzaldehyde Standard	33017	2000ug/ml
	Alpha-Terpineol Standard	33912	2000ug/ml
	8270 Benzidines Mix#2	31852	2000ug/ml
	1,4-Dioxane	557629	10,000ug/mL
Absolute	Atrazine	70023	1000ug/ml
	Aromatic Amines Mix	99410	2000ug/ml
AccuStandard	Parathion	M-622-19	1000ug/ml
	2,6-Dichlorophenol	M-8040-08	1000ug/ml
SPEX	n-Decane	S-1115	1000ug/ml
	n-Octadecane	S-2850	1000ug/ml
	2,6-Dichlorophenol	S-1415	1000ug/ml
	a-Terpineol	S-3356-AC	1000ug/ml
	Diesel Range Organics Mix	DRO-1000	1000ug/ml
	Custom SVOA	SVO-ALAMA-7-4	2000ug/ml
	1,4-Dioxane	S-1715	1000ug/mL
Ultra	Atrazine	EPA-1176A	1000ug/ml
	Parathion	SP-140-1	100ug/ml

8.1.1 ABN Mega Mix Standard, 200µg/mL

Use 5mL of each of the following:
- 605 Benzidines Mix
- Benzoic Acid Mix
- Acid Surrogate Mix

and use 10mL of each of the following:
- 8270 Mega Mix

and use 2mL of each of the following:
- B/N Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Standard, 200ug/mL

Use 5mL of each of the following:
- Custom SV Standard
- Custom ABN Addition Standard
- Benzaldehyde Standard

and use 10mL of each of the following:
- a-Terpineol Standard
- 2,6-Dichlorophenol

Bring up to 50mL volume with DCM.

8.1.3 ADP Standard, 200ug/ml

Use 5ml of:
- 8270 Benzidines Mix#2

and use 10mL of each of the following:
- Parathion
- Atrazine
- n-Decane
- n-Octadecane

Bring up to 50mL volume with DCM.

8.1.4 Calibration Stock Standards Preparation

A minimum of 5 calibration standards for each analyte are prepared.

Level	*Concentration (ug/mL)*
L1	1
L2	2
L3	3
L4	5
L5	10
L6	20
L7	50
L8	100
L9	150
L10	200

8.2 Internal Standard Solution

This is a premixed, certified solution from Supelco, 2000ng/mL in DCM, catalog #4-8902. Each 500µL of standards, blank and sample extracts are spiked with 10µL of Internal Standard Solution, resulting in a concentration of 40ng/µL.
The internal standards are: 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12}.

8.3 GC/MS Tuning Standard

A methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP) is used for checking the tune. The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

This working standard is prepared from a stock solution, purchased from Ultra Scientific, Catalog# GCM-150.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL DCM.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml each of Base-Neutrals Surrogate Mix #31086 and Acid Surrogate Mix #31087. Bring up to volume with Acetone. The final concentration is 50ug/ml for the Acid surrogates and 25ug/ml for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

8.5.1 Spike Solution Preparation

ABN SPK1:

In a 200ml volumetric flask add 8ml of 8270 Mega Mix #31850, 4ml of Benzoic Acid Mix#31879, Custom SV Standard#562843, Benzaldehyde Standard#33017, Custom SVOA#SVOA-ALAMA-7-4; Bring up to volume with Acetone. The final concentration is 40ug/ml.

ABN SPK2:

In a 200ml volumetric flask add 8ml of Atrazine#EPA-1176A and Parathion#M-622-19 and 4ml of 8270 Benzidine Mix#2 #31852; Bring up to volume with Acetone. The final concentration is 40ug/ml.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extracion could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with “B” flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based on in-house control limits. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on in-house control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
  (1) ABN 50 ppm
  (2) AP9 50 ppm
  (3) ADP 50 ppm
- Method Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC

Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be ≤ 20% for the mean response factor to be used for sample quantitation.

An alternate calculation fits may be performed provided that the minimum correlation coefficient ≥ 0.99 is met.

When linear regression model is used a minimum quantitation check of the lowest calibration point is performed. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.3 Equipment Operation and Sample Processing

GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500µL of sample extract to another vial, and add 10µL of the internal standard solution (Section 8.2).

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq$ 20%, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Alpha SOP/1732 DL/LOD/LOQ Generation

Alpha SOP/1739 IDC/DOC Generation

Alpha SOP/1729 Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria

Table 9: Difficult analytes

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-60% of mass 198
365	> 1% of mass 198
441	Present but less than 24% mass 442
442	> 50% of mass 198
443	15-24% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	Nitrobenzene-d_5	23-120%	23-120%
BN-8270D	2-Fluorobiphenyl	15-120%	30-120%
BN-8270D	p-Terphenyl-d_{14}	41-149%	18-120%
Acid-8270D	Phenol-d_6	10-120%	10-120%
Acid-8270D	2-Fluorophenol	21-120%	25-120%
Acid-8270D	2,4,6-Tribromophenol	10-120%	10-136%

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A

ACCEPTABLE AQUEOUS QC LIMITS

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene			70-130	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20
1,2-Dichlorobenzene	40-140	30	70-130	20
1,3-Dichlorobenzene	40-140	30	70-130	20
1,3-Dinitrobenzene	15-130	30		
1,4-Dichlorobenzene	36-97	30	70-130	20
1-Methylnaphthalene	41-103	30		
2,3,4,6-Tetrachlorophenol			70-130	20
2,4,5-Trichlorophenol	30-130	30	70-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20
2,4-Dichlorophenol	30-130	30	70-130	20
2,4-Dimethylphenol	30-130	30	70-130	20
2,4- Dimethylaniline	40-140	30	70-130	20
3,4- Dimethylaniline	40-140	30	70-130	20
2,3- Dimethylaniline	40-140	30	70-130	20
2,4,5-Dimethylaniline	40-140	30	70-130	20
4-Chlorotoluidine	40-140	30	70-130	20
2-Ethylaniline	40-140	30	70-130	20
O-toluidine	40-140	30	70-130	20
2-Napthylamine	40-140	30	70-130	20
2,4-Dinitrophenol	20-130	30	20-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20
2,6-Dinitrotoluene	40-140	30	70-130	20
2-Chloronaphthalene	40-140	30	70-130	20
2-Chlorophenol	27-123	30	70-130	20
2-Methylnaphthalene	40-140	30	70-130	20
2-Methylphenol	30-130	30	70-130	20
2-Nitroaniline	52-143	30	70-130	20
2-Nitrophenol	30-130	30	70-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20
3,3'-Dimethylbenzidine			20-160	20
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20
3-Nitroaniline	25-145	30	70-130	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20
4-Chloroaniline	40-140	30	20-160	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
4-Nitroaniline	51-143	30	70-130	20
4-Nitrophenol	10-80	30	20-160	20
Acenaphthene	37-111	30	70-130	20
Acenaphthylene	45-123	30	70-130	20
Acetophenone	39-129	30	70-130	20
Aniline	40-140	30	20-160	20
Anthracene	40-140	30	70-130	20
Atrazine			70-130	20
Azobenzene	40-140	30	70-130	20
Benzaldehyde			20-160	20
Benzidine	10-75	30	20-160	20
Benzo(a)anthracene	40-140	30	70-130	20
Benzo(a)pyrene	40-140	30	70-130	20
Benzo(b)fluoranthene	40-140	30	70-130	20
Benzo(ghi)perylene	40-140	30	70-130	20
Benzo(k)fluoranthene	40-140	30	70-130	20
Benzoic Acid	10-164	30	20-160	20
Benzyl Alcohol	26-116	30	20-160	20
Biphenyl	40-140	30	70-130	20
Bis(2-chloroethoxy)methane	40-140	30	70-130	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20
Butyl benzyl phthalate	40-140	30	70-130	20
Caprolactam			20-160	20
Carbazole	55-144	30	70-130	20
Chrysene	40-140	30	70-130	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20
Dibenzofuran	40-140	30	70-130	20
Diethyl phthalate	40-140	30	70-130	20
Dimethyl phthalate	40-140	30	70-130	20
Di-n-butylphthalate	40-140	30	70-130	20
Di-n-octylphthalate	40-140	30	70-130	20
Fluoranthene	40-140	30	70-130	20
Fluorene	40-140	30	70-130	20
Hexachlorobenzene	40-140	30	70-130	20
Hexachlorobutadiene	40-140	30	70-130	20
Hexachlorocyclopentadiene	40-140	30	20-160	20
Hexachloroethane	40-140	30	20-160	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20
Isophorone	40-140	30	70-130	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Naphthalene	40-140	30	70-130	20
Nitrobenzene	40-140	30	70-130	20
NitrosoDiPhenylAmine(NDPA)/Diphenylamine (DPA)	40-140	30	70-130	20
n-Nitrosodimethylamine	22-74	30	20-160	20
n-Nitrosodi-n-propylamine	29-132	30	70-130	20
P-Chloro-M-Cresol	23-97	30	70-130	20
Pentachlorophenol	9-103	30	20-160	20
Phenanthrene	40-140	30	70-130	20
Phenol	12-110	30	20-160	20
Pyrene	26-127	30	70-130	20
Pyridine	10-66	30		
2-Fluorophenol	*21-120*		*15-110*	
Phenol-d6	*10-120*		*15-110*	
Nitrobenzene-d5	*23-120*		*30-130*	
2-Fluorobiphenyl	*15-120*		*30-130*	
2,4,6-Tribromophenol	*10-120*		*15-110*	
4-Terphenyl-d14	*41-149*		*30-130*	

TABLE 3B

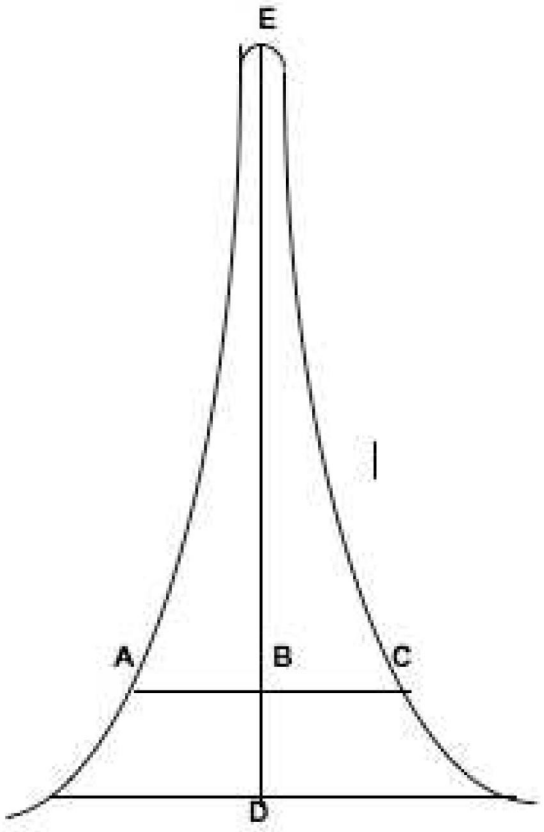
ACCEPTABLE SOIL QC LIMITS

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30
1,2-Dichlorobenzene	40-140	50	70-130	30
1,3-Dichlorobenzene	40-140	50	70-130	30
1,3-Dinitrobenzene	40-140	50		
1,4-Dichlorobenzene	28-104	50	70-130	30
1-Methylnaphthalene	26-130	50		
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30
2,4,5-Trichlorophenol	30-130	50	70-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30
2,4-Dichlorophenol	30-130	50	70-130	30
2,4-Dimethylphenol	30-130	50	70-130	30
2,4-Dinitrophenol	4-130	50	20-160	30
2,4-Dinitrotoluene	28-89	50	70-130	30
2,6-Dinitrotoluene	40-140	50	70-130	30
2-Chloroaniline	30-130	50		
2-Chloronaphthalene	40-140	50	70-130	30
2-Chlorophenol	25-102	50	70-130	30
2-Methylnaphthalene	40-140	50	70-130	30
2-Methylphenol	30-130.	50	70-130	30
2-Nitroaniline	47-134	50	70-130	30
2-Nitrophenol	30-130	50	70-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30
3,3'-Dimethylbenzidine	15-115	50		
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30
3-Nitroaniline	26-129	50	70-130	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30
4-Chloroaniline	40-140	50	20-160	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30
4-Nitroaniline	41-125	50	70-130	30
4-Nitrophenol	11-114	50	20-160	30
Acenaphthene	31-137	50	70-130	30
Acenaphthylene	40-140	50	70-130	30
Acetophenone	14-144	50	70-130	30
Aniline	40-140	50	20-160	30
Anthracene	40-140	50	70-130	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	Acceptance Criteria	Duplicate RPD	Acceptance Criteria	Duplicate RPD
Atrazine	40-140	50	70-130	30
Azobenzene	40-140	50	70-130	30
Benzaldehyde	40-140	50	20-160	30
Benzidine	10-66	50	20-160	30
Benzo(a)anthracene	40-140	50	70-130	30
Benzo(a)pyrene	40-140	50	70-130	30
Benzo(b)fluoranthene	40-140	50	70-130	30
Benzo(e)Pyrene	40-140	50		
Benzo(ghi)perylene	40-140	50	70-130	30
Benzo(k)fluoranthene	40-140	50	70-130	30
Benzoic Acid	10-110	50	20-160	30
Benzyl Alcohol	40-140	50	20-160	30
Biphenyl	54-104	50	70-130	30
Bis(2-chloroethoxy)methane	40-117	50	70-130	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30
Butyl benzyl phthalate	40-140	50	70-130	30
Caprolactam	15-130	50	20-160	30
Carbazole	54-128	50	70-130	30
Chrysene	40-140	50	70-130	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30
Dibenzofuran	40-140	50	70-130	30
Diethyl phthalate	40-140	50	70-130	30
Dimethyl phthalate	40-140	50	70-130	30
Di-n-butylphthalate	40-140	50	70-130	30
Di-n-octylphthalate	40-140	50	70-130	30
Diphenamid	40-140	50		
Fluoranthene	40-140	50	70-130	30
Fluorene	40-140	50	70-130	30
Hexachlorobenzene	40-140	50	70-130	30
Hexachlorobutadiene	40-140	50	70-130	30
Hexachlorocyclopentadiene	40-140	50	20-160	30
Hexachloroethane	40-140	50	20-160	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30
Isophorone	40-140	50	70-130	30
Naphthalene	40-140	50	70-130	30
Nitrobenzene	40-140	50	70-130	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30
n-Nitrosodimethylamine	22-100	50	20-160	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
n-Nitrosodi-n-propylamine	32-121	50	70-130	30
Parathion, ethyl	40-140	50	20-160	30
P-Chloro-M-Cresol	26-103	50	70-130	30
Pentachloronitrobenzene	42-153	50		
Pentachlorophenol	17-109	50	20-160	30
Phenanthrene	40-140	50	70-130	30
Phenol	26-90	50	20-160	30
Pyrene	35-142	50	70-130	30
Pyridine	10-93	50	20-160	30
Thionazin	40-140	50		
2-Fluorophenol	*25-120*		*30-130*	
Phenol-d6	*10-120*		*30-130*	
Nitrobenzene-d5	*23-120*		*30-130*	
2-Fluorobiphenyl	*30-120*		*30-130*	
2,4,6-Tribromophenol	*10-136*		*30-130*	
4-Terphenyl-d14	*18-120*		*30-130*	

TABLE 4 – Tailing Factor Calculation



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
3,3'-Dichlorobenzidine	252	254, 126
2,4-Dichlorophenol	162	164, 98
Diethyl phthalate	149	177, 150

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	212	211, 213
Dimethyl phthalate	163	194, 164
2,4-Dimethylphenol	107	121,122
Di-n-butyl phthalate	149	150, 104
Di-n-octyl phthalate	149	167, 43
4,6-Dinitro-2-methylphenol	198	51, 105
O-Toluidine	106	107, 77
2-Ethylaniline	106	121, 77
2,4-Dimethylaniline	121	120, 106
2,3-Dimethylaniline	106	121, 120
3,4- Dimethylaniline	121	120,106
2,4,5-Trimethylaniline	120	135, 134
4-Chlorotoluidine	106	141, 140
2-Napthylamine	143	115, 116
2,4-Dinitrophenol	184	107,91
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
Diphenamide	167	72, 165
1,4-Dioxane	88	58,43
Ethyl parathion	109	97, 291
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95, 138
1-Methylnaphthalene	115	141, 142
2-Methylnaphthalene	142	141
2-Methylphenol	108	107,90
3/4-Methylphenol	108	107,90
Naphthalene	128	129, 127
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	92,65
4-Nitroaniline	138	65, 108, 92, 80, 39
Nitrobenzene	77	123, 65

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2-Nitrophenol	139	109, 65
4-Nitrophenol	65	109, 139
n-Nitrosodimethylamine	74	42,44
n-Nitrosodi-n-butylamine	84	57, 41, 116, 158
n-Nitrosodi-n-propylamine	70	42, 101, 130
n-Nitrosodiphenylamine/Diphenylamine	169	168, 167
Pentachlorobenzene	250	252, 108, 248, 215, 254
Pentachloronitrobenzene	237	142, 214, 249, 295, 265
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Phenol	94	65, 66
Pyrene	202	200, 203
Pyridine	79	52
1,2,4,5-Tetrachlorobenzene	216	214, 179, 108, 143, 218
2,3,4,6-Tetrachlorophenol	232	131, 230, 166, 234, 168
m-Toluidine	106	107, 79
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	200,198
2,4,6-Trichlorophenol	196	198, 200
Acenaphthene-d_{10} (IS)	164	162, 160
Chrysene-d_{12} (IS)	240	120, 236
1,4-Dichlorobenzene-d_4 (IS)	152	150, 115
Naphthalene-d_8 (IS)	136	68
Perylene-d_{12} (IS)	264	260, 265
Phenanthrene-d_{10} (IS)	188	94, 80
2-Fluorobiphenyl (Surrogate)	172	171
2-Fluoroophenol (Surrogate)	112	64
Nitrobenzene-d_5 (Surrogate)	82	128, 54
Phenol-d_6 (Surrogate)	99	42, 71
Terphenyl-d_{14} (Surrogate)	244	122, 212
2,4,6-Tribromophenol (Surrogate)	330	62,141

TABLE 6

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34
Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
O-Toluidine	2	N/A
2-Ethylaniline	2	N/A
2,4-Dimethylaniline	2	N/A
2,3-Dimethylaniline	2	N/A
3,4-Dimetylaniline	2	N/A
2,4,5-Trimethylaniline	2	N/A
4-Chlorotoluidine	2	N/A
2-Napthylamine	2	N/A
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
1,4-Dioxane	5	166.67
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34
1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

* **Note**: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
O-Toluidine	2-Ethylaniline	2-Napthylamine	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2,4-Trichlorobenzene	2,4-Dimetthylaniline	2,3,4,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,2-Dichlorobenzene	3,4-Dimetthylaniline	2,3,5,6-Tetrachlorophenol	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	2,3-Dimetthylaniline	2,4,6-Tribromophenol, surr	Benzyl butyl phthalate	Benzo(b)fluoranthene	
1,4-Dichlorobenezne	2,4,5-Trimrthylaniline	2,4-Dinitrophenol	Carbazole	Benzo(k)fluoranthene	
2,4-Dichlorophenol	4-Chlorotoludine	2,4-Dinitrotoluene	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2,4-Dimethylphenol	1,2,4,5-Tetrachlorobenzene	3-Nitroaniline	Diphenamid	Chrysene	
2-Chloroaniline	1,2-Dichlorobenzene	4,6-Dinitro-2-methylphenol	Fluoranthene	Di-n-octylphthalate	
2-Chlorophenol	1,3-Dichlorobenzene	4-Bromophenyl-phenyl ether	n-Octadecane		
2-Fluorophenol, surr	1,4-Diclorobenzene	4-Chlorophenyl-phenyl ether	Parathion		
2-Methylphenol	1-chloror-2-nitrobenzene	4-Nitroaniline	Phenanthrene		
2-Nitrophenol	1-Methylnapthalene	4-Nitrophenol	Pyrene		
3-Methylphenol / 4-Methylphenol	2,4,5-Trichlorphenol	Acenaphthene	Terphenyl-d14, surr		
Acetophenone	2,4,6-Trichlorophenol	Atrazine			
Aniline	2,6-Dichlorophenol	Azobenzene			
Benzaldehyde	2,6-Dinitrotoluene	Dibenzofuran			
Benzyl Alcohol	2-Chloronaphthalene	Dichloran			
Bis(2-chloroethoxy)methane	2-Fluorobiphenyl, surr	Diethyl phthalate			
Bis(2-chloroethyl)ether	2-Methylnaphthalene	Fluorene			

Table 7 (cont.)
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
bis(2-Chloroisopropyl)ether	2-Nitroaniline	Hexachlorobenzene			
Hexachloroethane	3-Choloroaniline	NDPA/DPA			
Isophorone	4-Chloro-3-Methylphenol	Pentachloronitrobenzene			
m-Toluidine	4-Chloroaniline	Pentachlorophenol			
n-Decane	Acenaphthylene				
Nitrobenzene	a-Terpineol				
Nitrobenzene-d5, surr	Benzoic Acid				
N-Nitrosodimethylamine	Biphenyl				
N-Nitrosodi-n-propylamine	Caprolactam				
Phenol	Dimethyl Phthalate				
Phenol-d6, surr	Hexachlorobutadiene				
Pyridine 1,4-Dioxane	Hexachlorocyclopentadiene				
Phenol-d6, surr	Naphthalene				

Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

Table 8 (cont.)

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Buchi Concentration

Method Reference: This standard operating procedure (SOP) is a performance-based method. This SOP combined with the Buchi Concentration Work Instruction describes the procedure as developed by Alpha Analytical.

1. Scope and Application

Matrices: This method is applicable to aqueous, solids, soils, and sludge samples.

Definitions: Refer to Alpha Analytical Quality Manual.

The Buchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Sample extracts are filtered into Buchi glassware following extraction. The Buchi unit is prepared for use as outlined in the Buchi Concentration Work Instruction (See WI 16257). Samples are concentrated using vacuum, heat, and oscillation to reach final volume. The Buchi will recover >95% of solvent emissions. Once concentrated, proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

2.1 Method Modifications from Reference

None

3. Reporting Limits

Refer to analytical SOPs for Reporting Limit information.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to

be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Refer to sample extraction SOPs.

7. Equipment and Supplies

7.1 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels

8. Reagents and Standards

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's RO water treatment system.

8.2 Acetone: Pesticide quality. No expiration date listed.

8.3 Dichloromethane: Pesticide quality. No expiration date listed.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not Applicable.

9.2 Laboratory Control Sample (LCS)

Not Applicable.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable

9.5 Matrix Spike

Not Applicable

9.6 Laboratory Duplicate

Not Applicable

9.7 Method-specific Quality Control Samples

Not Applicable

9.8 Method Sequence

Refer to Section 10.3

9.9 Chiller temperature logbook

The chiller temperature must be recorded each day the instrument is used. This document is reported to regulatory organizations.

9.10 Solvent recovery measurements

Each Buchi unit must demonstrate solvent recovery greater or equal to 95%. This must be done weekly for the first month and then monthly thereafter. This information must be recorded in the solvent recovery spreadsheet for review by regulators.

10. Procedure

10.1 Equipment Set-up

10.1.1 Prepare the Buchi equipment for operation as outlined in the Buchi Concentration WI.

10.1.1.1 Set-up includes draining the solvent collection flasks into the appropriate hazardous waste container.

10.1.1.2 Check the chiller water level. If low, refill with reagent water to level marked on the chiller unit.

10.1.1.3 Turn “on” all components at least 20 minutes before use to allow temperatures to equilibrate to operational values. This includes the Base Unit, Chiller, Pump, Block and Controller. Push the “start” button on the block once and turn “off “the oscillation to start the block heating. It is important to start the chiller by pressing the start button. Just turning the unit on will not start the chiller.

10.1.1.4 The proper Buchi concentration program must also be selected on the controller as outlined in the WI.

10.2 Initial Calibration

Not Applicable

10.3 Equipment Operation and Sample Processing

10.3.1 Equipment operation and sample processing are outlined in the Buchi Concentration WI. Some items to note are described below.

10.3.1.1 Add 5mL of water into each block cell position to improve head conductivity between the heater block and extract.

10.3.1.2 It is helpful to set the rotation to 90 RPM before loading the samples to reduce the possibility of samples bumping / boiling.

10.3.1.3 All positions on the block must have a vessel in order to form a vacuum. Use empty vessels if necessary to fill the 12-position block.

10.3.1.4 Once all samples have been concentrated, the extracts can be vialed or moved to the next step (e.g. extract cleanup). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.4 Continuing Calibration

Not Applicable

10.5 Preventive Maintenance

10.5.1 Refrigeration Re-circulator: The Refrigeration Re-circulator should be checked once every shift (when in use) to insure that it is at the specified temperature, running correctly and that the level of water is constant with manufactures recommendation.

10.5.2 Buchi System: Contact factory representative with inquiries and maintenance issues.

10.5.2.1 Buchi Vacuum Cover: The Buchi unit vacuum cover requires cleaning in a fume hood using acetone.

10.5.3 See Buchi Cleaning Procedure (ID 23508) for proper cleaning instructions.

11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to appropriate analytical SOPs.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

MDL is not applicable to this concentration method. Refer to analytical SOPs.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

IDC/DOC is not applicable to this concentration method. Refer to analytical SOPs.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

WI 16257 Buchi Concentration Work Instruction

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

SOP ID 1728 Waste Management and Disposal SOP

SOP ID 2110 Semivolatile Organic Compounds by Gas Chromatography/Mass Spectromety (GC/MS)

SOP ID 2111 Semivolatile Organic Compounds by Gas Chromotography/Mass Spectrometry (GC/MS)

SOP ID 2125 TPH- Diesel Range Organics

SOP ID 2127 Extractable Total Petroleum Hydrocarbons (ETPH)

16. Attachments

Not Applicable

Acid Digestion of Solid Samples for Metals Analysis

Reference Method: **EPA 3050B,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1996.

1. Scope and Application

Matrices: Sediments, sludge, soils.
Definitions: See Alpha Laboratories Quality Manual Appendix A

This method provides two separate digestion procedures. Samples prepared by this method may be analyzed by ICP- AES or ICP-MS for all the listed metals, provided the detection limits are adequate for the analytical end use of the data.

Alternative determinative techniques may be used if they are scientifically valid and the QC criteria of the method, including those dealing with interferences, can be achieved. Other elements and matrices may be digested by this method if performance is demonstrated for the analytes of interest, in the matrices of interest, at the concentration levels of interest.

The recommended determinative techniques for each element are listed below:

ICP- AES		
Aluminum	Antimony	Barium
Beryllium	Cadmium	Calcium
Chromium	Cobalt	Copper
Iron	Lead	Magnesium
Manganese	Molybdenum	Nickel
Potassium	Silver	Sodium
Thallium	Vanadium	Zinc

ICP-MS	
Arsenic	Beryllium
Cobalt	Cadmium
Chromium	Iron
Lead	Molybdenum
Selenium	Thallium

This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become “environmentally available”. By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment. If total digestion is required, Method 3052 is preferable.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for

the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.
This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A representative sample from 1.25g to 5g is digested in a solution of Aqua Regia (3:1 HCl:HNO_3) and further additions nitric acid (HNO_3 conc.) and heated in a heat source.
Samples digested in the Hot Block: Nitric acid and Hydrochloric acid are added to the sample and reflux for 30 minutes. 5-10 mL of DI water is added to wash down the walls of the digestion vessel and 1 mL of concentrated Nitric acid is added. The samples are again heated in the heat source and allowed to reflux at 90-100°C for an additional 30 minutes.
After digestion, the extract is brought to a final volume of 50mL and allowed to settle or filtered if necessary.

2.1 Method Modifications from Reference

Digestates are prepared in the same manner for both ICP-AES and ICP-MS determinative methods.

Method section 7.5 Aqua Regia digestion with optional filtration performed when settling is not feasible based on sample composition (i.e.: high suspended solids, precipitates or floatables).

3. Reporting Limits

The Reporting Limit is determined by the amount of sample used for preparation. Therefore, a review of Client requirements for Reporting Limits is necessary prior to sample preparation. Also refer to the analytical SOP.

4. Interferences

None.

5. Health and Safety

Caution must be used when handling the following:

- cHCL
- cHNO_3
- Aqua Regia

These chemicals are all corrosives and can cause harm to skin and eyes. When using these corrosives, the analyst must wear a lab coat, gloves, and protective eye wear.

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard.

From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic jars

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are refrigerated at 4 ± 2 °C upon receipt, and are digested within 90 days of collection.

7. Equipment and Supplies

7.1 Polypropylene Digestion Vessel: 50mL volume, SCP Science

7.2 Reflux Cap, SCP Science

7.3 Weighing Tray

7.4 Spatula, Stainless Steel

7.5 Volumetric Flasks: 200mL volume, Class A

7.6 Whatman 40 or equivalent Filter Paper

7.7 Hot Block, thermostatically controlled, calibrated with correction factor application.

7.8 Balance: Capable of weighing to 0.001g.

7.9 Polypropylene Bottles: 250mL volume

8. Reagents and Standards

8.1 Hydrochloric Acid, concentrated (cHCl): 18M; store at room temperature.

8.2 Nitric Acid, concentrated ($cHNO_3$): 18M; store at room temperature.

8.3 Aqua Regia: Prepare a 3:1 solution of cHCl: $cHNO_3$. This solution is prepared fresh each day of use and discarded after use.

8.4 50% Hydrochloric Acid (HCl): 500mL cHCl diluted to 1 liter with DI water; store at room temperature.

8.5 10% Nitric Acid (HNO_3): 100mL $cHNO_3$ diluted to 1 liter with DI water; store at room temperature.

8.6 Reagent Water: Deionized water (DI) from Alpha's water treatment system.

8.7 1000ppm and 10,000ppm Single Element Stock Standards: All stock standards are commercially prepared and certified. All standards are in acidic aqueous solutions. Standards are stored at room temperature, and the vendors' expiration date is used.

8.8 10ppm Single Element Intermediate Standards: Intermediate standards are made from dilution 1:10 commercially prepared and certified stock standards (Section 8.9). All standards are in acidic aqueous solutions. Standards are stored at room temperature and the vendor's expiration date is used.

8.9 Multi-element Stock Standards: All stock standards are commercially prepared and certified. All standards are in acidic aqueous solution. Standards are stored at room temperature, and the vendors' expiration date is used.

8.9.1 **CLP ICP Standard #1:** Al, Ba at 2000 µg/mL; Fe at 1000 µg/mL; Co, Mn, Ni, V, Zn at 500 µg/mL; Cu at 250 µg/mL; Cr at 200 µg/mL; Be and Ag at 50 µg/mL.

CLP ICP Standard #3: As, Se, Tl at 2000 µg/mL; Pb at 500 µg/mL; Cd at 50 µg/mL.

Ag Spike Standard: 100 ug/mL prepared standard.

1000ppm and 10,000 ppm Standards of individual metals

8.10 Working Standards: The working standards are prepared from the stock standards in 10% nitric acid and then brought to a 500mL final volume. Standards are stored at room temperature, this solution expires 12 months after the date of preparation or the expiration of the parent solution, whichever is earliest.

8.10.1 IPS Working Standard: To a 500mL Class A volumetric flask, add 250mL of DI water the add 25mL of $cHNO_3$ (Section 8.2), cap and mix by inverting. Add 50mL of CLP ICP Standard #1 stock (Section 8.11.1), 25mL of 1000ppm Sb Standard (Section 8.9), 2.5mL of 1000ppm Cd Standard (Section 8.9) and dilute to a final volume of 500mL with DI water.

The resulting concentration of this solution is: 200 mg/L of Al and Ba; 100 mg/L of Fe; 50 mg/L of Co, Mn, Ni, V and Zn; 25 mg/L of Cu; 20 mg/L of Cr; 5 mg/L of Be and Ag ; 50 mg/L of Sb; and 5 mg/L of Cd; 10% $cHNO_3$.

8.10.2 FPS Working Standard: To a 500mL Class A volumetric flask, add 250mL of DI water the add 25mL of $cHNO_3$ (Section 8.2), cap and mix by inverting. Add 3mL of CLP ICP Standard #3 stock (Section 8.11.2), 25mL of 1000ppm Pb Standard (Section 8.9) and dilute to a final volume of 500mL with DI water.

The resulting concentration of this solution is: 12 mg/L of As, Se and Tl; 53 mg/L of Pb; and 0.3 mg/L of Cd; 10% $cHNO_3$.

8.10.3 MIX Working Standard: To a 500mL Class A volumetric flask, add 50mL of DI water the add 25mL of $cHNO_3$ (Section 8.2), cap and mix by inverting. Add 50mL each 1000ppm of the following from Section 8.9: ICP Boron Standard, ICP Mo Standard, ICP Sr Standard, ICP Ti Standard. Add 50mL each 10,000ppm of: ICP Ca Standard, ICP Mg Standard, ICP K Standard, ICP Na Standard and dilute to a final volume of 500mL with DI water.

The resulting concentration of this solution is: 100 mg/L of B, Mo, Sr and Ti; 1000 mg/L of Ca, Mg, K, and Na.

8.10.4 Silver Spike Standard: To a 100mL Class A volumetric flask, add 50mL of DI water the add 5mL of $cHNO_3$ (Section 8.2), cap and mix by inverting. Add 10 mL of 1000 ug/mL Ag standard and dilute to the final 100 mL volume mark. Transfer to an amber glass 200 mL bottle.
The resulting concentration of this solution is: 100 mg/L of Ag.

8.11 Standard Reference Material (SRM)

A standard reference material is used for all solids and soil digestions as the LCS. The SRM is purchased from a vendor (ERA) and evaluated by the vendor control limits (95%confidence limits).

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

PBS, Prep blank for soil: Digest one PBS per batch of 20 samples or less. The Method Blank is carried through the complete preparation procedure and contains the same volume of reagents as the sample solutions. The Method Blank is used to assess contamination from the laboratory environment.

9.2 Laboratory Control Sample (LCS)

LCSS, Laboratory Control Sample for soil: Digest one LCSS per batch of 20 samples or less.

For samples that are prepared in the 50mL Polypropylene digestion vessel and utilize between 0.3 and 0.4 grams of Standard Reference Material (SRM); Environmental Resource Associates, Cat 540, lot number D0xx-540.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Digest one MS per batch of 20 samples or less for MET-T products or upon client request.

For samples that are prepared in the 50mL Polypropylene digestion vessel and utilize between 1.25 to 5 grams of sample: To a second aliquot of the sample chosen for the MS, add 1.0mL of IPS (Section 8.9.2), FPS (Section 8.9.3), and MIX (Section 8.9.4) working stock standards and 0.25 mL of 100ppm Ag standard (Section 8.9.5). If the desired metal is not included in the spiking solution, then also add 50µL of 1000ppm desired metal stock standard (Section 8.9).

9.6 Laboratory Duplicate

One duplicate sample is digested per matrix batch of 20 or less for MET-T products or upon client request.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Mix the sample thoroughly to obtain a homogeneous and representative aliquot.
- Weigh the appropriate amount of sample, and QC samples.
- **A**dd 1 mL cHNO_3 and 3 mL cHCl to the each sample vial.
- Cover flasks with a reflux cap and heat in the digestion block at 90-100 °C for 30 minutes.
- Raise and rotate each sample vessel to the elevated position in the sample rack and allow to cool slightly.
- Using 5-10 mL DI water, rinse down the inner walls of the sample vessel.
- Add 1 mL of c$HNO_{3\ to}$ each vessel.
- Rotate and lower each sample vessel to the heating position and heat at 90-100 °C for 30 minutes.
- Remove from samples from the digestion block. Bring samples to a 50 mL final volume with DI water and let settle or filter if necessary.

10. Procedure

10.1 Equipment Set-up

10.1.1 Turn on the heat source (Section 7.7) to a temperature of 95 ºC ± 3 ºC.

10.1.2 Set up the electronic laboratory notebook completing all fields including the following information:

- Date, Chemist's initials, Method
- Job number, Metals analysis requested
- Sample weight
- Type of acid used and its Lot#, Final Volume
- Comments on color of sample, texture of sample

- MS / LCSS used
- Time ON and OFF the heat source

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Homogenization:

Homogenize the entire contents of the sample container to a consistent appearance to achieve a representative sample.

10.3.2 Duplicate: A second aliquot of the sample chosen to be duplicated.

10.3.3 Preparation Blank Solid (or Method Blank), (PBS):

The Method Blank is carried through the complete preparation procedure and contains the same volume of reagents as the sample solutions. The Method Blank is used to assess contamination from the laboratory environment.

10.3.4. Laboratory Control Sample Solid, (LCSS):

10.3.4.1: In the 50mL Polypropylene digestion vessel and utilize between 0.3 and 0.4 grams of SRM: Carry through entire process as a sample.

10.3.5 Matrix Spike, (MS):

10.3.5.1: To an aliquot of the sample designated for the MS, add 1.0mL each of IPS (Section 8.9.2), FPS (Section 8.9.3), and MIX (Section 8.9.4) working stock standards and 0.25 mL of 100ppm Ag standard (Section 8.9). If the desired metal is not included in the spiking solution, then also add 50µL of 1000ppm desired metal stock standard (Section 8.9).

10.3.6 Digestion Procedure:

Weigh a representative sample from 1.25g to 5g into a 50 mL digestion tube. Under a laboratory hood, slowly add 1 mL cHNO_3 (Section 8.2) followed immediately by the addition of 3 mL cHCl (Section 8.1) to each sample in the digestion vessel. Cover each vessel with a Reflux Cap.
NOTE: The acid combination used creates Aqua Regia, a powerful oxidizer which is a strong irritant; do not remove samples containing this concentrated form from the laboratory hood.
Heat the samples in the digestion vessel in the digestion block at 95 +/-3ºC, and reflux for 30 minutes without boiling. Allow the samples to cool slightly by elevating the vessels in the rack-locks, add 1mL of cHNO_3, and reflux for another 30 minutes.
Ensure the sample is covered by the acid at all times during heating. Record in the laboratory notebook the time samples are placed in the digestion block and the time samples are taken out of the digestion block. The samples are brought up to a final volume of 50 mL. Instrumental analysts are to allow the sample to settle before decanting a sample aliquot.

10.3.6.1 Filtration Procedure:

If the sample has high suspended solids, precipitates upon cooling or floatables the sample filtration is performed as follows:

- Filter the digestate through Whatman No. 41 filter paper (or equivalent) and collect filtrate in a 50-mL digestion tube. Wash the filter paper with no more than 5 mL of hot (~95 ºC) HCl, then with 20 mL of hot (~95 ºC) reagent water. Pre-heat the acid and reagent water in digestion tubes on the digestion heating block. Collect washings in the same 50-mL digestion vessel.
- Remove the filter and residue from the funnel, and place them back in the original digestion tube. Add 5 mL of conc. HCl, place the vessel back on the heating source, and heat at 95 ºC ± 3 ºC until the filter paper dissolves. Remove the vessel from the heating source and wash the cover and sides with reagent water. Filter the residue and collect the filtrate and combine with the first filtrate in the 50-mL digestion vessel. Allow filtrate to cool.
- Bring to a final volume of 50mL with DI water, cap and mix by inverting a minimum of 3 times. Deliver to Instrument room with all appropriate batch paperwork.

NOTE: High concentrations of metal salts with temperature-sensitive solubilities can result in the formation of precipitates upon cooling of primary and/or secondary filtrates. If precipitation occurs in the flask upon cooling, do not dilute to volume but add up to 10 mL of concentrated HCl to dissolve the precipitate. After precipitate is dissolved, dilute to volume with reagent water.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

The Hot Block temperature is calibrated on an annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

Refer to analytical SOPs.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances and improper preservation are noted on the batch sheet by the prep analyst and conveyed to the department supervisor or manager to include on nonconformance report.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the appropriate maintenance logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples; record any trends or unusual performance on a nonconformance action form.

If any QC parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified

and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP# 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP# 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

CHP#2124 Chemical Hygiene Plan
SOP #1732 MDL/LOD/LOQ Generation
SOP# 1739 IDC/DOC Generation
SOP# 1728 Waste Management and Disposal SOP

16. Attachments

None.

Mercury Determination in Solids by Cold Vapor Atomic Absorption Technique (CVAA)

References: USEPA, "Method 7471B Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)," in Test Methods for Evaluating Solid Waste, SW846, Revision 2, February 2007

1. Scope and Application

Matrices: This cold-vapor atomic absorption method is applicable to the determination of total mercury (organic and inorganic) in solid samples (soils, sediments and sludges).

Definitions: Refer to Alpha Analytical Quality Manual.

This digestion and analytical procedure measures total mercury (organic & inorganic) in soil, sediment and sludge samples. Mercury can accurately be determined in the range of 0.005 to 0.25 mg/Kg for solid samples that do not require dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the AA and in the interpretation of AA data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Mercury	7439-97-6

2. Summary of Method

A 0.5 to 1.5 gram aliquot of sample is digested in an acidic, oxidizing solution (consisting of Hydrochloric acid, nitric acid, and potassium permanganate) and heated for a total of 32 minutes on a hotplate at 95°C to convert all forms of mercury to inorganic Hg (II). Once the samples have been digested, they are ready for analysis by the cold-vapor atomic absorption technique (CVAA).

The CVAA technique is based on the absorption of radiation at 253.7-nm by mercury vapor. The addition of the hydroxylamine-hydrochloride solution to the digestate, reduces the excess potassium permanganate without reducing the mercury and transforms the oxidized mercury to the insoluble and non-volatile $HgCL_2$. Mercury is then reduced with stannous chloride from $HgCL_2$ to elemental mercury, [Hg (0)]. The elemental mercury is aerated as mercury vapor from the digestate in a closed system where the vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorbance is measured as a function of mercury concentration based on the peak height measured.

2.1 Method Modifications from Reference

Method 7471B specifies use of 300 mL BOD bottles for sample and standard preparation and a final volume of approximately 100 mL. This method has been modified for use of 50 mL plastic digestion tubes. Reagent volumes have been reduced proportionately.

3. Reporting Limits

The mercury solid RL is 0.005 mg/Kg.

4. Interferences

4.1 Potassium permanganate is added to eliminate potential interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from distilled water.

4.2 High concentrations of copper may cause interference; however copper concentrations as high as 10 mg/L have no effect on the recovery of spiked mercury samples.

4.3 Additional portions of potassium permanganate may need to be added until the purple color persists due to interference from chloride or organic matter. During the oxidation step, chlorides are converted to free chlorine which absorbs radiation at 253.7-nm. Care must be taken to ensure that free chlorine is absent before mercury is reduced to its elemental state and mercury vapor is swept into the cell. This may be accomplished by using additional portions of sodium chloride-hydroxylamine hydrochloride solution.

Note: Chloride interference is not as common in solid matrices as aqueous matrices.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. All digestion and analysis procedures must be conducted in a laboratory exhaust hood.

5.2 Care must be taken when handling all samples, digestates, and standards since they are preserved to a pH <2. In addition, the digestate solutions contain strong oxidizing reagents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 10.0 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

The samples must be refrigerated and maintained at 4°±2°C until digestion and analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All solid samples must be analyzed within 28 days from date of collection.

7. Equipment and Supplies

7.1 PSA 10.035 MILLENNIUM MERLIN MERCURY ANALYZER

7.1.1 Lamp: Mercury, low pressure

7.1.2 HP Laser Jet P2015dn or equivalent, compatible with AA and AF software

7.1.3 Millennium AAS Detector:

7.1.4 Pumps: Two variable speed and independently controlled peristaltic pumps to deliver reagent and sample solutions as well as remove waste.

7.1.5 Drying Tube: Gas liquid separator and dryer tube to remove mercury vapor from solution and prevent water vapor from entering the absorption cell.

7.2 Disposable Digestion tubes, 50mL

7.3 Electric Hot Plate- Adjustable and capable of maintaining a temperature of 90-95°C equipped with graphite carbon blocks that each have 36 positions to hold sample tubes. Glassware - Assorted Class A: volumetric flasks, beakers, graduated cylinders and pipettes of appropriate sizes for preparing reagents, standards, and measuring sample volumes.

7.4 Air Displacement pipettes: Digital pipettes capable of delivering volumes ranging from 0.1 to 5000 µL with an assortment of high quality disposable pipette tips.

7.5 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g

7.6 Top-loading balance: Capable of accurate measurement to the nearest 0.01 g

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted.

Stock standard solutions are stored in a cabinet, out of direct light

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Concentrated hydrochloric acid (HCl). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Concentrated nitric acid (HNO_3). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.4 Aqua Regia. Mix concentrated HCl and HNO_3 at a ratio of 3:1. *(Digestion reagent)*

8.5 Potassium Permanganate ($KMnO_4$), Fisher P279-500 or equivalent

8.6 5% Solution (w/v) of Potassium Permanganate Solution. Dissolve 50g of $KMnO_4$ in deionized water and dilute to 1L. *(Digestion reagent)*

8.7 Hydroxylamine Hydrochloride ($NH_2OH \bullet HCl$). Fisher H330-500, or equivalent. *(Analytical reagent)*

8.8 Stannous Chloride ($SnCl_2 \bullet 2H_2O$). Fisher T142-500, or equivalent. *(Analytical reagent)*

8.9 Stannous Chloride Reduction Solution (2%). Add 20 g of $SnCl_2 \bullet 2H_2O$ to 100mL of concentrated HCl and dilute to 1L with deionized water. Prepare fresh daily. *(Analytical reagent)*

8.10 HCl Rinse Solution (10%). Dilute 900mL of concentrated HCl to 9L with deionized water. *(Analytical reagent)*

8.11 Sodium Chloride (NaCl). Fisher S271-3, or equivalent. *(Analytical reagent, added to the Hydroxylamine Hydrochloride Solution below)*

8.12 Sodium Chloride-Hydroxylamine Hydrochloride Solution. Dissolve 120g of NaCl and of $NH_2OH \bullet HCl$ in deionized water and dilute to 1L. *(Analytical reagent)*

8.13 1000mg/L Mercury Stock Standard from two different sources. One source is used to prepare the calibration curve (Ultra Scientific ICP-080) and the other is for verification of the calibration curve (Inorganic ventures CGHG1-1). Use the vendor's expiration date. *(Standards preparation)*

8.14 Mercury Working Standards. Three mercury working standards are prepared from successive dilutions of the mercury stock standard and are used to prepare the calibration curve. Acidity of the working standards must be maintained at 1% HCl acid. Add 1mL of concentrated HCl to a 100mL volumetric flask. A 1.0mg/L working standard is prepared by adding 0.1mL of the 1000mg/L Ultra Scientific stock standard to a final volume of 100mL. This standard is diluted 1:10 to make a 0.1mg/L working standard and 1:100 to make a 0.01 mg/L working standard. The 0.1 and 0.01mg/L working standard is diluted prior to preparation of the calibration standards. The 1.0mg/L working standard is prepared monthly. See Section 10.0, for specific details regarding working standard preparations. *(Standards preparation)*

8.15 1.0mg/L LCS and Matrix Spike Solution. Prepare from the 1000 mg/L stock standard (8.13) by adding a 0.1mL aliquot to a final volume of 100mL. This solution is stable for 1 month. *(Spiking Solution)*

8.16 Solid Matrix LCS Standard: Purchased from Environmental Resource Associates (ERA), Catalog #540 Metals in Soil QC Standard, or equivalent.

8.17 Solid Matrix Blank Sample: Purchased from Environmental Resource Associates (ERA), Catalog #058 Blank Soil QC Sample, or equivalent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 A method blank must be analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.1.2 Mercury concentrations must not be detectable in the method blank at values greater than the reporting limit.

9.1.3 Corrective Action: Digestion of the method blank and all associated samples must be performed until the method blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the method blank are non-detect for mercury or if the concentration of mercury is greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified in the project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

9.2.1 The LCS is digested along with the samples. A LCS must be digested and analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits are 80%-120%. The solid LCS recovery limits are continuously monitored and documented in-house through control charts. The SOP *Control Limit Generation* (1734) provides details explaining how control charts are generated and used for quality control.

9.2.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-digest and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

9.3.1 Initial Calibration Curve

9.3.1.1 The correlation coefficient (r) of the initial calibration curve must be ≥0.995. The initial calibration curve consists of five standards and a blank.

9.3.1.2 Corrective Action: If the correlation coefficient does not exceed 0.995, individual standards or the entire curve may be re-analyzed until the correlation coefficient is in control. If the correlation coefficient is still not in control, the initial calibration curve must be re-prepared and re-analyzed until the correlation coefficient is acceptable.

9.3.2 Initial Calibration Verification (ICV) Check Standard

9.3.2.1 The initial calibration verification check standard must be from a second source or lot number to verify the accuracy of the standard curve. The concentration of the ICV is at approximately the mid-level of the calibration curve.

9.3.2.2 The acceptable recovery QC limits for the ICV is 90%-110%.

9.3.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate the instrument.

9.3.3 Initial Calibration Blank (ICB)

9.3.3.1 An ICB must be analyzed immediately following the ICV.

9.3.3.2 The ICB concentration must not be greater than the reporting limit.

9.3.3.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICB still exceeds the control limits, re-calibrate the instrument and re-analyze a fresh blank.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Continuing Calibration Verification (CCV) Check Standard

9.4.1.1 A CCV must be analyzed at a minimum of every 10 samples and at the close of an analytical sequence. The concentration of the CCV is at approximately the mid-level of the calibration curve. This standard monitors instrument performance throughout the duration of the analytical run.

9.4.1.2 The acceptable recovery QC limits for the CCV is 90%-110%.

9.4.1.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since the last acceptable CCV.

9.4.2 Continuing Calibration Blank (CCB)

9.4.2.1 A CCB must be analyzed immediately after every CCV.

9.4.2.2 The CCB concentration must not be greater than the reporting limit.

9.4.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh blank. All samples associated with the out of control CCB

must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Section Head if the samples associated with the out of control CCB are non-detect for mercury or if sample concentrations for the affected metals are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

9.5.1 A matrix spike must be performed once per 20 samples (5% frequency). When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS.

9.5.2 The acceptable recovery QC limits is 80%-120% for the solid MS/MSD. For MCP projects the recovery QC limits are 75%-125% for the solid MS/MSD. Calculate MS Recovery as follows:

$$\% \text{ Recovery} = \frac{MS - R1}{MS_{\text{True Value}}} \times 100$$

9.5.3 Calculate the %RPD as in 12.3.2 above when analyzing a MS/MSD pair. The acceptable %RPD is $\leq$ 20%. Both the solid recovery limits and %RPD are continuously monitored and documented in-house through control charts.

9.5.4 Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % recovery, or %RPD, still exceed the control limits and the LCS is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy or precision of the affected mercury results as evidenced by matrix spike %recovery or %RPD outside of the QC limits.

9.6 Laboratory Duplicate

9.6.1 Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

9.6.2 Acceptable relative percent difference (RPD) for duplicate analysis is $\leq$ 20 % for solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

The RPD limits are continuously monitored and documented in-house through control charts.

9.6.3 Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the affected mercury results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 Serial Dilution (SD)

9.7.1.1 Serial dilution of one sample per batch of 20, at a 1:5 dilution, is useful in the assessment of matrix effects, and must be analyzed as needed to assess a new or unusual matrix.

9.7.1.2 The acceptable agreement between the undiluted and diluted results is ± 10%.

9.7.1.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the precision between the results still exceeds the control limits, include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the affected mercury results as evidenced by the SD outside of the QC limits.

9.7.2 CRA Standard Check Sample

9.7.2.1 The CRA Check sample is typically analyzed as part of the initial calibration curve, however, project specifications may require this to be analyzed at the beginning of the analytical sequence after the ICV and ICB. This solution verifies the accuracy at the low end of the calibration curve.

9.7.2.2 Results for mercury in the CRA solution must be within 70-130% recovery criteria or within project specified acceptance limits.

9.7.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CRA solutions still exceed the control limits, re-calibrate and/or re-analyze a fresh CRA solution. All samples associated with the out of control mercury CRA must be re-analyzed.

***NOTE**: The CRA analysis is not preformed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve. The calibration curve must meet the criteria for linearity.*

9.8 Method Sequence

Initial calibration curve (STD0, STD1, etc., to STD5)
ICV
ICB
CRA (if required by the project)
Method Blank (The assigned LIMS batch name)
LCS (The assigned LIMS batch name)
Sample analysis (samples 1-5 including SD, MS and MD samples)
CCV
CCB
Sample analysis (6-15)
CCV
CCB

The CRA analysis is not preformed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve. The calibration curve must meet the criteria for linearity.

10. Procedure

10.1 Equipment Set-up

10.1.1 Digestion Procedure Using Block Digestion

10.1.1.1 Weigh a 0.5 to 1.5g aliquot of well-homogenized sample into the digestion tube. A nominal weight of 0.5-0.6g of each sample is generally sufficient; however an increased amount of wet sample may be used, and depends upon the required reporting limits for the project. See the Section Head or the Project Manager if there are any questions regarding the reporting limits required or the amount of sample to be used.

10.1.1.2 A method blank, LCS, matrix spike sample, and matrix duplicate sample must be digested with each analytical batch at the frequency listed in Section 9, Quality Control. In some instances, the client may request matrix spike duplicate analysis as an alternative to matrix duplicate analysis.

If required, and a blank solid material of a similar matrix type of high enough purity can be obtained to meet required reporting limits, a solid material may be used in the Method Blank preparation to matrix match QC samples and field samples.

If required, a solid LCS may be prepared to matrix match QC samples and field samples.

10.1.1.3 The calibration standards are digested along with the samples.

10.1.1.4 Add 5mL of ASTM Type II laboratory reagent ASTM Type II laboratory reagent and 5 mL Aqua Regia (3:1 HCL to HNO3) to sample and calibration tubes.

10.1.1.5 Spike Laboratory Control Sample and Matrix Spike sample with 0.125mL of the Hg 1 mg/L spiking solution (see 8.14).

10.1.1.6 Heat samples and calibration curve to 95°C +/- 3°C on heated Blocks for 2 minutes, remove from heat, let cool to room temperature, then add 15mL ASTM Type II laboratory reagent grade water.

10.1.1.7 Add 7.50mL of 5% Potassium Permanganate solution, wait 15 minutes.

10.1.1.8 If the purple color dissipates, add additional portions of $KMnO_4$ solution until the purple color persists. Note the additional amount of $KMnO_4$ on the preparation bench sheet. Heat for an additional 30 minutes on heated blocks.

10.1.1.9 Allow the samples and standards to cool. Add 3.0mL of NaCl-NH_2OH•HCl solution to each digestion tube to reduce the excess permanganate. Add deionized water to bring the final volume to 50mL.

10.1.1.10 The digestates are now ready for analysis. The batch must be transferred to the metals instrument room with the project folder, the preparation paperwork and the metals preparation checklist.

10.2 Initial Calibration

A series of five calibration standards are prepared by pipetting suitable volumes of standard solution into 50mL centrifuge tubes. The preparation date of these standards, the initials of the analyst, the lot number of the source material, stock concentrations, volumes used, final volumes, final concentrations, and manufacturer, and expiration date must be recorded in the *Mercury CVAF, Mercury CVAA and Amalgam Working Standards Preparation Logbook*. The

PSA MILLENIUM MERLIN is calibrated using a multi-point calibration curve consisting of a blank and six standards.

10.2.1 The working standards are prepared as follows:

10.2.1.1 Mercury Stock Solution (1000mg/L). See Section 8.13. Check expiration date to ensure the standard has not expired.

10.2.1.2 Mercury Working Standard (1.0mg/L). See Section 8.14. Check expiration date to ensure the standard has not expired.

10.2.1.3 Diluted Mercury Working Standard (0.1mg/L): Dilute 0.1mL of working standard (10.2.1.2) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.4 Diluted Mercury Working Standard (0.01mg/L): Dilute 0.1 mL of working standard (10.2.1.3) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.5 The calibration standards may be prepared in 50mL screw cap plastic digestion tubes. Each tube must contain approximately 25mL of deionized water, which is then spiked with the volume listed in the table below. Prepare a six-point mercury calibration curve daily as follows

Volume of the Working Standard (mL)	Concentration of Working Standard (mg/L)	Concentration of Calibration Standard (µg/L)
0	None	Blank
0.025	0.1	0.05
0.05	0.1	0.1
0.25	0.1	0.5
0.1	1	2
0.25	1	5

10.2.1.6 Digest all standards along with the samples as described in Section 10.1, Equipment Set-up.

10.2.2 The PSA MILLENIUM MERLIN computer will calculate a linear regression, correlation coefficient ("r"), and slope of standard curve. The correlation coefficient must be greater than $\geq$ 0.995 for linearity. The linear regression is computed on a multipoint calibration.

10.3 Equipment Operation and Sample Processing

10.3.1 Before turning the instrument on, unplug and power off on rear panel. Connect the millennium AAS detector, plug back in and power on the back panel. The pump windings must be inspected for wear, rotated and/or changed. Secure the pump windings to the pump. Turn the gas on to 52 P.S.I. Turn on the mercury analyzer and the computer. Allow the instrument to warm up for 30 to 60 minutes.

10.3.2 Place the rinse line in the 10% HCl rinse solution. The $SnCl_2$ line from the instrument must be immersed into the 2% stannous chloride reduction solution.

10.3.3 All standard and sample information is entered into a sample information, or instrument sequence file. The following information is entered: sample ID, dilution and units. A hard copy of the sequence file is printed, given a page number, and becomes part of the permanent instrument run log. After one month, or more if suitable, of mercury analyses, the sequence printouts are bound and given an internal log identification number.

10.3.4 Place the calibration standards and samples onto the autosampler rack in the appropriate positions according to the sample information sequence file.

10.3.5 Begin the run sequence with the initial calibration curve and evaluate it for QC acceptance. Evaluate the ICV, ICB, CRA, Method Blank and LCS for QC acceptance prior to the analysis of the samples. The typical analytical sequence is listed in Section 9.8.

10.3.6 The instrument detection limits (IDLs) may be performed if required by the regulatory program or the client.

10.3.7 Dilute and reanalyze any samples that exceed the linear calibration range for mercury. Report the mercury result from the dilution analysis.

10.3.8 The *Mercury Data Review Checklist* must accompany all acceptable mercury results for primary and secondary review.

10.4 Continuing Calibration

10.4.1 The same time must be elapsed between CCVs and CCBs as is allowed between samples. Analyze the continuing calibration verification (CCV) and the continuing calibration blank (CCB) after each 10 samples and at the end of the analytical sequence.

10.5 Preventive Maintenance

Pump windings should be inspected for wear on a daily basis and replaced when they appear flattened (approximately once per month depending on use). The gas-liquid separator should be cleaned periodically when it appears coated with a yellow film. Add 30% KOH solution and let set for approximately 30 minutes. Rinse with DI water. The dryer tube should be replaced approximately once per year. (Part number H003S001 PS Analytical)

11. Data Evaluation, Calculations and Reporting

11.1 The mercury results are calculated by the following equation:

Solid:

$$\text{Mercury result in mg/Kg} = \frac{C \times B}{A} \times DF \times 1000 \times \frac{100}{\%\ \text{solid}}$$

Where:

A = Initial sample weight in grams, typically 1 gram
B = Digestion final volume in mL, typically 50 mL
C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor

11.2 All mercury results must be reported to three significant figures.

11.3 The primary analyst does the batching and data upload into the LIMS system.

11.4 All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.5 A secondary review is performed on all.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

13. Method Performance

13.1 Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1797 Waste Management and Disposal SOP

16. Attachments

None.

Organochlorine Pesticides
By Capillary Column Gas Chromatography

Reference Method No.: **Method 8081B**

References: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

SW-846 Pesticides by 8081, State of Connecticut Department of Environmental Protection Reasonable Confidence Protocol (RCP), Version 1.0, July 2005

1. Scope and Application

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. This SOP details the analysis for these compounds using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts from solid and liquid matrices.

Definitions: See Alpha Analytical Quality Manual

Regulatory Parameter List: The compounds listed below are determined by this method:

Parameter	CAS
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Alpha-chlordane	5103-71-9
Gamma-chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Chlordane	12789-03-6
Alachlor	15972-60-8

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13), analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the QA Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

A measured volume or weight of sample (for liquids, 500 mls or 1L, for solids, 15g to 30g) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), Method 3546 (microwave extraction), or other appropriate technique.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

After cleanup, the extract is analyzed by injecting a 1µL sample into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our routine reporting limits.

4. Interferences

4.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the hydrogen (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 9.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the extraction SOPs. Store dry glassware in a clean environment.

4.4 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.5 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOPs minimize these possible interferences. Analyst experience is also crucial in making compound determinations.

4.6 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

4.7 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.7.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.7.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

4.7.3 Interferences from phthalate esters are minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.8 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is often seen in sediment and some soil samples.

4.9 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Coeluting chlorophenols are eliminated by using Method 3620B (florisil).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and BHCs. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in either two 500 ml amber glass jars or two 1L amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2^{o} C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2^{o} C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.1.1 Data System: A computer system is interfaced to the GC for collection of data. Data acquisition is performed using Agilent Technologies GC ChemStation (Rev. A.10.02 [1757]) and/or Perkin Elmer Corp. Turbochrom software (6.1.0.1:F04).

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

7.2.1 Column pair 1

30m x 0.32mm ID fused silica capillary column (RTX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (RTX-clpesticideII) 0.25µm film thickness.

7.2.2 Column pair 2

30m x 0.32mm ID fused silica capillary column (STX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (STX-clpesticideII) 0.25µm film thickness.

7.3 Volumetric Flasks: 10mL and 25mL, for the preparation of standards.

7.4 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.5 Disposable Borosilicate Pipets

7.6 Vials: 2 mL clear glass, crimp-top and screw-cap.

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 $\pm$ 2^{o}C in Teflon(R)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Methylene chloride: Pesticide quality or equivalent.

8.4 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.5 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is

opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 2.

8.6 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at 10 concentration levels for individual pesticides and 9 concentration levels for Chlordane/Toxaphene mix; concentrations for all levels are listed in Table 2. Below are suggested procedures for preparation of individual pesticides (Sec. 8.6.1) and Chlordane/Toxaphene (Sec. 8.6.2) initial calibration standards.

8.6.1 Preparation of individual pesticides initial calibration standards;

(syringes needed: *100µL [A], 500µL [B]*)
(solvent: *hexane)*
(glassware: *1x25mL, 10x10mL, volumetric flasks*)

STOCK #1: *2000µg/L; 25mL*
50µL[A] of Accustandard M-8081-SC (Pesticides mix; 1000µg/mL)
250µL[B] of Accustandard CLP-032-R (TCMX, Deca; 200µg/mL)
500µL[B] of Accustandard P-1025 (Alachlor; 100µg/mL)
500µL[B] of Accustandard APP-9-112 (Hexachlorobenzene; 100µg/mL)

STOCK #2 (=LEVEL 9)**:** *100µg/L; 10mL.*
500µL[B] of STOCK #1
…..

LEVEL 1, 0.5 µg/L: 50µL[A] of STOCK #2, 10mL
LEVEL 2, 1.0 µg/L: 100µL[A] of STOCK #2, 10mL
LEVEL 3, 2.0 µg/L: 200µL[B] of STOCK #2, 10mL
LEVEL 4, 3.0 µg/L: 300µL[B] of STOCK #2, 10mL
LEVEL 5, 4.0 µg/L: 400µL[B] of STOCK #2, 10mL
LEVEL 6, 5.0 µg/L: 500µL[B] of STOCK #2, 10mL
LEVEL 7, 10 µg/L: 50µL[A] of STOCK #1, 10mL
LEVEL 8, 50 µg/L: 250µL[B] of STOCK #1, 10mL
LEVEL 9, 100 µg/L: 500µL[B] of STOCK #1, 10mL
LEVEL 10, 200 µg/L: 1mL[B] of STOCK #1, 10mL

8.6.2 Preparation of Chlordane/Toxaphene initial calibration standards;

(syringes needed: *100µL [A], 500µL [B]*)
(solvent: *hexane)*
(glassware: *10x10mL, volumetric flasks*)

STOCK #1: *50/100µg/mL (Chlordane/Toxaphene);10mL*
100µL[A] of Ultra Scientific EPA-1086 (Chlordane Solution; 5000µg/mL)
1mL[B] of Restek 32005 (Toxaphene Standard; 1000µg/mL)

STOCK #2 (=LEVEL 7)**:** *1000/2000µg/L; 10mL.*

200µL[A] of STOCK #1

LEVEL 1, 5/10 µg/L: 50µL[A] of STOCK #2, 10mL
LEVEL 2, 10/20 µg/L: 100µL[A] of STOCK #2, 10mL
LEVEL 3, 50/100 µg/L: 500µL[B] of STOCK #2, 10mL
LEVEL 4, 100/200 µg/L: 1mL[B] of STOCK #2, 10mL
LEVEL 5, 250/500 µg/L: 50µL[A] of STOCK #1, 10mL
LEVEL 6, 500/1000 µg/L: 100µL[A] of STOCK #1, 10mL
LEVEL 7, 1000/2000 µg/L: 200µL[A] of STOCK #1, 10mL
LEVEL 8, 2500/5000 µg/L: 500µL[B] of STOCK #1, 10mL
LEVEL 9, 5000/10000 µg/L: 1mL[B] of STOCK #1, 10mL

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25 µg/mL. Standard solution expires 6 months after the date of preparation.

8.8 Surrogate Standards: Tetrachloro-m-xylene and decachlorobiphenyl are used as surrogates. They are added to the calibration standards at the concentrations listed in Table 2, and are spiked into all samples and QC samples prior to extraction. The spiking solution is prepared in acetone at the concentrations listed in Table 2. Standard expires 6 months after the date of preparation.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting the appropriate stock standards in acetone. The spiking solution concentrations are listed in Table 2. Spiking solution expires 6 months after the date of preparation.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.2.1 LCS Duplicate (LCSD)

A Laboratory Control Sample Duplicate is extracted with each analytical batch. The LCSD consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCSD is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria is listed in Table 3. If Recovery limits are outside acceptance criteria, the non-conformance is narrated.

9.2.2 Connecticut RCP LCS Requirements

For CT- Reasonable Confidence Protocol (RCP) 8081B samples only, the LCS recovery criteria is 40 – 140%.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

A matrix spike (MS)/matrix spike duplicate (MSD) is extracted and analyzed upon Client request for each batch of 20 or less samples. The spike compounds and levels are listed in Table 2. The recovery acceptance criteria are 30-150%. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

A duplicate sample is extracted and analyzed upon Client request for each batch of 20 or less samples. The % RPD criteria are listed in Table 3. If the %RPD is not met, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Initial calibration:

1. Instrument Blank (may be omitted to conserve tray space)
2. Degradation check standard
3. Std Level 1
4. Std Level 2

5. Std Level 3
6. Std Level 4
7. Std Level 5
8. Std Level 6
9. Std Level 7
10. Std Level 8
11. Std Level 9
12. Std Level 10 (not applicable to chlordane and toxaphene)
13. Initial Calibration Verification Standard (ICV)
14. Repeat std Levels 1 –9 for other mixes until curve is complete.

    [**NOTE:** If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.]

Typical Daily sequence:

1. Degradation Check Standard
2. Pesticide Continuing Calibration Standard
3. Chlordane/Toxaphene Continuing Calibration Standard
4. Extraction Blank
5. Laboratory Control Sample
6. Laboratory Control Sample Duplicate
7. Matrix Spike (Upon Client Request)
8. Duplicate/Matrix Spike Duplicate (Upon Client Request)
9. Samples (up to 20 may be analyzed)
10. Cal check
11. Repeat 1 – 9 (as needed)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Extraction

Water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510C). See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Soxhlet extraction (Method 3540C) or microwave extraction (Method 3546). See extraction SOP for details.

10.1.2 Extract Cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. See extraction SOPs for details.

10.1.3 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Typical GC conditions for the HP 6890 instruments are listed below, but may be altered as long as method performance criteria are met.

Temperature 1:	120 °C
Time 1:	0 minute
Ramp 1:	45 °C/minute
Temperature 2	200 °C
Time 2:	0 minutes
Ramp 2:	15 ° C/minute
Temperature 3:	230 °C
Time 3:	0 minutes
Injector temperature:	250 °C
Ramp 3:	30 °C/minute
Final Temperature:	330 °C
Final Time:	2.0 minutes

Injector mode:	Pulsed Split
	2:1 split, 0.75 min pulse
Injector Flow:	6.0 mL/min split flow
Detector temperature:	375 °C
Carrier gas:	Hydrogen
Carrier flow:	17 mL/min
Carrier mode:	Constant flow
Makeup gas:	Argon/methane (P5)
Total detector flow:	55 mL/min
Injection volume:	1 µL

10.1.4 DDT and Endrin Breakdown

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance must be completed if the breakdown in greater than 15% for either compound (See Section 10.5.1). Both analytical columns must pass DDT/Endrin breakdown criteria prior to sample analysis.

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.6 and the concentrations listed in Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.3. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Turbochrom or Agilent Chemstation data acquisition software.

10.2.2.1 Record the calibration standard, unique lab identifier code (lot), the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 Calibration Factors

Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Target data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria

If the RSD for an analyte is $\leq$ 20%, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the RSD for any analyte is > 20%, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of linearity as long as the correlation coefficient is $\geq$0.995. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.7 Retention Time Windows

10.2.7.1 The retention time windows used for the identification of target analytes are calculated using the procedure recommended in Method 8000 and were found to be ± 0.015 minutes.

10.2.7.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at sufficiently high concentration, GC/MS analysis is used for confirmation.

10.2.8 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. This standard must be prepared using a second source that is different than the source used for the initial calibration. The %D for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve.

10.3 Equipment Operation and Sample Processing

10.3.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 10.1.3).

10.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. Confirmation is positive when the %RPD is $\leq$ 40% from the results of the two columns. In particularly difficult matrices, confirmation by GC/MS may be advisable (see Section 10.3.11).

10.3.3 The concentration reported for an identified target analyte in an extract is calculated using the Target data processing software. The Target methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

10.3.3.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze.

10.3.4 Each sample analysis must begin with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

10.3.5 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.3.6 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte in the mid-concentration standard during the initial calibration as the mid-point of the window. The widths of the windows are defined in Section 10.2.7.

10.3.7 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.3.8 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic 'fingerprint' retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peaks(s) of the same retention time and shape generated using internal calibration procedures.

10.3.9 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.10 For secondary column analysis, a second dissimilar column is utilized to confirm positive pesticide results. The laboratory must report the higher of the two results unless obvious interference is present on one of the columns. All required QA/QC parameters (e.g. calibrations, LCSs, etc.) must be met on the second column as well.

10.3.11 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.3.11.1 Full-scan GC/MS will normally require a concentration of approximately 10ng/µL in the final extract for each single-component compound.

10.3.11.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

10.3.11.3 GC/MS may not be used for confirmation when concentrations are below the sensitivity of the instrument.

10.3.11.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis.

10.3.11.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.3.11.6 A QC reference sample containing the compound should also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

10.4 Continuing Calibration Verification

10.4.1 Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must be injected at intervals of not less than once every twenty samples (after every 4 - 8 samples is routine practice to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence.

10.4.1.1 The calibration factor (for external standard compounds) and response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve. The Target data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual.

10.4.1.2 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.1.3 If routine maintenance does not return the instrument performance to meet the QC requirements (Section 10) based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a CF or RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples. Any QC failures must be written up by the analyst on narrative sheets for inclusion with the sample data.

10.4.2 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows described in section 10.2.7. The center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.5.1 Other Maintenance

Additional maintenance may be required if system performance degrades.

10.5.1.1 GC injector ports are of critical concern, especially in the analysis of DDT and Endrin.

10.5.1.1.1 Injectors that are contaminated or chemically active can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE.

Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

When such breakdown is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the degradation does not improve, it may be necessary to replace the column(s).

Calculate percent breakdown as follows:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD+DDE)}}{\text{sum of all peak areas (DDT+DDE+DDD)}} \times 100$$

$$\% \text{ breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde+ketone)}}{\text{sum of all peak areas (Endrin+aldehyde+ketone)}} \times 100$$

10.5.1.2 ECD detectors may also become contaminated, requiring bake out at elevated temperatures, or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Single Component Pesticides

The single component pesticide compounds are calculated as described in Section 10.3.3, and reported in µg/L or µg/Kg units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews should be done by two separate individuals.

11.1.1 Quantitation of Multiple-Component Analytes

Quantitation is based on use of 3-5 of the major peaks present for chlordane and 4-6 of the major peaks present for toxaphene. Each of these peaks is individually calibrated with a 9 point calibration based on average response factors. The %RSD must meet the criteria of ≤20%. The 3 to 5 or 4 – 6 major peaks are calculated as described in Section 10.3.3. After individual calculation meets criteria, the average of the major 3-5 or 4 – 6 peaks is used to determine the final concentration.

11.1.1.1 Toxaphene

Toxaphene is quantitated by the internal standard method, using the 4 – 6 largest peaks found in the standard and averaging the resulting concentrations.

11.1.1.2 Chlordane

Chlordane is a mixture of at least 11 major components and 30 or more minor components. Trans- and cis-Chlordane (alpha and gamma, respectively), are the two major components of Chlordane. However, the exact percentage of each in the chlordane material is not completely defined, and is not consistent from batch to batch.

11.1.1.2.1 The GC pattern of a Chlordane residue may differ considerably from that of the chlordane standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents

from the Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

11.1.1.2.2 Whenever possible, when Chlordane residue does not resemble Chlordane, the analyst should quantitate the peaks of alpha-Chlordane, gamma-Chlordane, Heptachlor, and trans-Nonachlor separately against the appropriate reference materials, and report the individual residues.

11.1.1.2.3 When the GC pattern of the residue resembles that of Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the 3-5 major peaks

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax ID 1732. These studies performed by the laboratory are maintained on file for review

13.2 Demonstration of Capability Studies

Refer to Qualtrax ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Qualtrax ID 1732 MDL/LOD/LOQ Generation

Qualtrax ID1739 IDC/DOC Generation

Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: QC ACCEPTANCE CRITERIA

TABLE 1

REPORTING LIMITS*

	RL (Aqueous)	RL (Soil)
Pesticides		
Alpha-BHC	0.02 µg/L	3.33 µg/Kg
Gamma-BHC (Lindane)	0.02 µg/L	2.67 µg/Kg
Heptachlor	0.02 µg/L	4 µg/Kg
Endosulfan I	0.02 µg/L	10 µg/Kg
Dieldrin	0.04 µg/L	5 µg/Kg
Endrin	0.04 µg/L	3.33 µg/Kg
4, 4'-DDD	0.04 µg/L	10 µg/Kg
4, 4'-DDT	0.04 µg/L	15 µg/Kg
Methoxychlor	0.2 µg/L	15 µg/Kg
Aldrin	0.02 µg/L	10 µg/Kg
Beta-BHC	0.02 µg/L	10 µg/Kg
Delta-BHC	0.02 µg/L	10 µg/Kg
Heptachlor Epoxide	0.02 µg/L	15 µg/Kg
trans-Chlordane	0.02 µg/L	10 µg/Kg
cis-Chlordane	0.02 µg/L	10 µg/Kg
4, 4'- DDE	0.04 µg/L	15 µg/Kg
Endosulfan II	0.04 µg/L	15 µg/Kg
Endrin Aldehyde	0.04 µg/L	10 µg/Kg
Endosulfan Sulfate	0.04 µg/L	3.33 µg/Kg
Endrin Ketone	0.04 µg/L	10 µg/Kg
Chlordane	0.2 µg/L	65 µg/Kg
Toxaphene	0.2 µg/L	150 µg/Kg
Alachlor [+]	0.1 µg/L	10 µg/Kg

*** RL:** Typical laboratory reporting limit. Actual RLs may be higher depending on sample matrix. RLs are not adjusted for % Moisture.

[+] CT-RCP- only compound.

TABLE 2

STANDARD SOLUTIONS

	Stock 1 solution (µg/mL)	Stock 2 solution (µg/mL)	Level 10 (µg/L)	Level 9 (µg/L)	Level 8 (µg/L)	Level 7 (µg/L)	Level 6 (µg/L)	Level 5 (µg/L)	Level 4 (µg/L)	Level 3 (µg/L)	Level 2 (µg/L)	Level 1 (µg/L)	Std. Spike Solution (µg/mL)	Std. LCS Solution (µg/mL)
Hexachlorobenzene	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alpha-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Lindane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan I	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Dieldrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDD	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDT	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Methoxychlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Tetrachloro-m-Xylene *	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Decachloro-Biphenyl *	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Aldrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Beta-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Delta-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor Epoxide	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Trans-Chlordane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Cis-Chlordane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'- DDE	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan II	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Aldehyde	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan Sulfate	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Ketone	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alachlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Chlordane	50000	1000	N/A	5000	2500	1000	500	250	100	50	10	5		
Toxaphene	100000	2000	N/A	10000	5000	2000	1000	500	200	100	20	10		
Internal Standard														
1-Bromo-2-Nitrobenzene	5000	5000	25	25	25	25	25	25	25	25	25	25		

* - surrogates

TABLE 3

QC ACCEPTANCE CRITERIA**

	Aqueous		Soil	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%	30%	150%
Decachlorobiphenyl	30%	150%	30%	150%

Compound	**LCS % Recovery (Aqueous)**		**LCS % Recovery (Soil)**		**Duplicate and/or MSD %RPD (Aqueous and Soil)**	
	Lower Control Limit	**Upper Control Limit**	**Target Analyte Limits**	**Marginal Exceedence Limits**	**Target Analyte Limits**	**Marginal Exceedence Limits**
Target Analytes LCS/LCSD	30%	150%	30%	150%	30%	50%
Target Analytes MS/MSD	30%	150%	30%	150%	30%	50%

** **NOTE:** CT-RCP, NJ-8081, and MCP-801 % Recovery LCS/ LCSD Acceptance Criteria is 40-140%

Inductively Coupled Plasma - Atomic Emission Spectrometry

Reference Method No.: **Method 6010C** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, February 2007.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Digestates from all matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion unless chemical interferants are suspected. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to Metals Preparation SOPs for the appropriate digestion procedures.

Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths for the elements in clean aqueous matrices. Table 3 lists the Reported Detection Limits. The reported detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 9) is demonstrated.

Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is made by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

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2. Summary of Method

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 must also be recognized and appropriate corrections made; tests for their presence are described in Section 9.4.4. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness, according to SM 2340B.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to Table 3 for method Reporting Limits.

4. Interferences

4.1 Spectral

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for

routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans must be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

4.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

4.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that each instrument may exhibit somewhat different levels of interference. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Major known interferences are Fe, Al, Ca, Mg, V, Ni, Cu, and Cr. To minimize any of these interferences, every analyte is analyzed on each instrument at or near its linear range and corrected for these interferences. This is done on an annual basis, and data is kept on file.

4.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear must be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon elements that could contribute spectral interferences.

4.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

4.1.7 The primary wavelength for each analyte is based upon the instrument manufacturer's recommendations. An alternate wavelength is chosen if there is an indication of elevated background or overlap of another spectral wavelength. The wavelength for each analyte must be as free from interferences as possible.

4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change must be determined and corrected and the correction factor updated. The interference check solutions must be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

4.1.9 When interelement corrections are applied, their accuracy must be verified, daily, by analyzing spectral interference check solutions. If the correction factor or multivariate correction matrices tested on a daily basis (by running a check solution on each analytical run) are found to be within 20% criteria for 5 consecutive days, analysis may be extended to a weekly basis. Also, if the nature of the samples analyzed is such that they do not contain concentrations of the interfering elements greater than the reported detection limit, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices are verified and updated on an annual basis or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. The standard solution must be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

4.1.10 When interelement corrections are not used, verification of absence of interferences is required.

4.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits, files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

4.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is >20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, using a peristaltic pump, use of an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 10.3.4.1 will help determine if a physical interference is present.

4.3 Chemical

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Additionally, if filtered samples are found to have an organic or sulfur like odor they are processed by heating after the addition of the acids to matrix match. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample must be the same as a normal sample analysis period, followed by analysis of the rinse

blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit must be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

4.5 Other Interferences

4.5.1 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations must be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO3 to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO3 to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples to be analyzed for soluble metals, that have not been filtered, must be filtered and preserved within 24 hours of sample collection.

Preserved samples have a hold time of 6 months, and are stored at ambient temperature.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

- **Thermo Scientific ICAP Duo 6500 (Trace7)**

7.1.1 Computer-controlled emission spectrometer with background correction.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Optional mass flow controller for argon nebulizer gas supply.

7.1.4 Optional peristaltic pump.

7.1.5 Optional Autosampler.

7.1.6 Argon gas supply - high purity.

7.2 Volumetric flasks of suitable precision and accuracy.

7.3 Volumetric pipets of suitable precision and accuracy.

8. Standards and Reagents

Reagent semiconductor and/or trace grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (conc), HCl. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.3 Nitric acid (conc), HNO_3. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.5 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

8.6 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All stock standards are ordered through ISO and American Association for Lab Accreditation vendors. All standards are in aqueous solutions and are generally at concentrations of 1000ppm and 10,000ppm.

8.7 Mixed calibration standard solutions

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care must be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards must be prepared, as needed, with the realization that concentration can change on aging.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.
Additionally, sulfur standards are stand-alone single element standards and therefore are not to be combined in a mixed calibration standard solution.

8.8 Blanks

Two types of blanks are required for the analysis for samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing.

8.8.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations (see Sections 10.2 and 10.4). Refer to Section 10.4.1.2 for acceptance criteria and/or corrective actions.

8.8.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Refer to Section 9.1 for acceptance criteria and/or corrective actions.

8.9 The Initial Calibration Verification Standard (ICV) and the Continuing Calibration Verification Standard (CCV)

These ICV is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard. The CCV is prepared from the same source as the calibration standards and must be at a concentration near the mid-point of the calibration curve. At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV must be at a concentration near the mid-point of the calibration curve.

8.9.1 Low Level Initial Calibration Verification Standard (LLICV) and the Low Level Continuing Calibration Verification Standard (LLCCV)

These standards are actually a series of standards (typically 3) that are at or below the RL for the respective elements included in the calibration sequence. They are prepared from the same source as the calibration standards but at the laboratory's discretion may be from a second source from the calibration.

8.10 Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

8.11 CRI

The CRI is an ICP standard that is analyzed at a concentration of 2 - 5 times each element's RDL. The CRI must be recovered within 70-130% of its true value. If the CRI does not meet these criteria, it is remade and reanalyzed. If the CRI fails a second time, the analysis is terminated, the problem determined and corrected. The instrument is then recalibrated.

CRI solutions are made for each type of instrument.

8.11.1 CRI Stock Standard Solution, for the TJA Trace instruments

To a 500mL volumetric flask, add 200mL DI water and 50mL of 1:1 HNO_3. Add the following volumes of each certified 1000ppm stock standard:

Pb	0.9 mL	Ni	1.6 mL
Se	0.4 mL	Ag	0.4 mL
Sb	2.0 mL	Tl	0.4 mL
As	0.4 mL	V	2.0 mL
Ba	0.8 mL	Zn	0.8 mL
Be	0.2 mL	Al	8.0 mL
Cd	0.2 mL	Ca	8.0 mL
Co	2.0 mL	Mg	8.0 mL
Cr	0.4 mL	B	2.0 mL
Cu	1.0 mL	Sr	0.4 mL
Fe	4.0 mL	Ti	0.4 mL
Mn	0.6 mL	Sn	0.4 mL
Mo	2.0 mL		

And the following volumes of each certified 10000ppm stock standard:

K	10.0 mL

Na	10.0 mL
Si	2.0 mL
S	2.0 mL

Bring to volume of 500mL with DI water. This solution expires 12 months after the date of preparation.

8.11.1.1 CRI Working Standard Solution

To a 1L volumetric flask, add 25mL of CRI Stock Standard Solution (Section 8.11.1). Bring to volume with DI water. This solution will contain elements in the following concentrations:

Pb	0.045 ppm	Ag	0.02 ppm
Se	0.02 ppm	Tl	0.02 ppm
Sb	0.10 ppm	V	0.10 ppm
As	0.02 ppm	Zn	0.04 ppm
Ba	0.04 ppm	Al	0.40 ppm
Be	0.01 ppm	Ca	0.40 ppm
Cd	0.01 ppm	Mg	0.40 ppm
Co	0.10 ppm	B	0.10 ppm
Cr	0.02 ppm	Sr	0.02 ppm
Cu	0.05 ppm	Ti	0.02 ppm
Fe	0.20 ppm	Sn	0.02 ppm
Mn	0.03 ppm	K	5.0 ppm
Mo	0.10 ppm	Na	5.0 ppm
Ni	0.08 ppm	Si	1.0 ppm
S	1.0 ppm		

8.12 Reporting Limit (RL) Verification Standard (LLICV/LLCCV)

The RL standard consists of a series of standards that are analyzed after the initial calibration verification (LLICV) and at the end of each run (LLCCV). Optionally, the LLCCV may be run every 10 samples with the CCV, CCB pair to eliminate the need for excessive reruns when low level instrument stability is questioned. These standards are at or below the RL included in the multi-point calibration sequence. The acceptance criteria are 70-130% to establish the RL for each analyte. The following standards are analyzed.

0.005 mg/L	Ag, As, Be, Cd
0.010 mg/L	B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sn, Sr, Ti, Tl, V
0.050 mg/L	Al, Sb, Fe, Zn, Ca, Mg, K, Na
0.25 mg/L	S

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample.

The method blank results must be less than the reported detection limit (RDL) for all analytes of concern. If the results of the method blank exceed the RDL for any analyte, perform re-analysis of a new aliquot of the method blank.

If the results continue to exceed the RDL, proceed as follows:

If all of the samples for the analyte are non-detected, and the method blank is at or above the RDL, no action is required.

If one or more associated samples for that analyte have positive results at or above the RDL, those samples must be considered to be out of control, and are re-digested and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCSW/SRM per sample batch. A LCS/SRM sample is a spiked volume of reagent water that is brought through the entire preparation and analytical process. The LCSW must have a % Recovery of ± 20% within the actual value or within vendor control limits (95% confidence limits) for the solid SRM.

If the LCSW or SRM % Recovery is outside the acceptable limits as stated in Table 2, or outside any vendor control limits, the LCS is rerun once. If upon reanalysis the LCS is still out of control, the failed analytes are re-prepped and re-analyzed. Otherwise, a nonconformance report form is raised to document the exact problem and this form is then authorized by the QA/QC Director and/or the Laboratory Manager(s).

9.3 Initial Calibration Verification (ICV)

For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration. The results of the ICV are to agree within 10% of the expected value; if not, re-analyze once, if still failing terminate the analysis, correct the problem, and recalibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of two) integrations.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The batch sheet is noted. If the standard fails again, instrument maintenance must be performed and the CCV/CCB standard is reanalyzed. If the standard passes, then all samples run after the last passing CCV/CCB pair must be re-analyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data after the last passing CCV/CCB is reanalyzed.

9.5 Matrix Spike

Analyze matrix spike samples at a frequency of one per matrix batch. The matrix spike is the same solution as used for the LCS (Table 4). A matrix spike sample is a sample brought through the entire sample preparation and analytical process.

9.5.1 The percent recovery is to be calculated as follows:

$$\% \text{ Recovery} = \frac{MS - S}{C} \times 100$$

where:

MS = Matrix Spike value
S = Sample value.
C = Concentration of the Spiking solution.

9.5.2 If the Matrix Spike falls outside of the limits as stated in Table 2, or outside any historical documentation for analytes of interest a post analytical spike is performed for the failed analytes. The same sample from which the MS/MSD aliquots were prepared should be spiked with a post digestion spike at a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The acceptable % Recovery of the post analytical spike is 80-120%. A nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.5.3 If the Post Spike fails the dilution test should be performed. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, then a chemical or physical interference effect should be suspected.

9.6 Laboratory Duplicate

A duplicate sample is analyzed once per matrix batch. This sample is brought through the entire sample preparation and analytical process.

9.6.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

9.6.2 If the Duplicate falls outside of the limits as stated in Table 2, or outside any historical documentation and the concentrations of the failing analytes are less than 5x the RL or a matrix interference is found a nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.7 Method-specific Quality Control Samples

9.7.1 Interference Check Standards

A check solution is analyzed once daily. One solution (ICSA) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of <2X RL. The other check solution (ICSAB) is the same solution spiked with a known amount of each analyte. These solutions are analyzed at the beginning of the first analytical run of the day.

If the analytes of interest in the ICSAB solution falls outside the acceptable limits of 80 – 120% of the true value, the solutions may be rerun once. The high level interferences are not evaluated for recovery just as in the ICSA. If the problem persists take corrective action which may include re-evaluation of the inter-element correction values (IECs). The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the ICSA/ICSAB re-analyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department.

9.7.2 Reporting Limit (RL) Verification Standard (LLICV/LLCCV)

The RL standards are actually a series of standards that are analyzed at the beginning and at the end of each run. The lowest of the RL standards may be used to evaluate the sensitivity of reportable elements under method 6010C. This may be a low level client-specific analysis, or it may be the standard reporting limits for an aqueous sample or a soil/solid material. The standards must have a percent recovery of 70-130%. If an element fails the acceptance criteria to establish a specific RL, the RL standard may be re-analyzed. If the element failure continues, then either re-calibrate the instrument and rerun the affected samples or analyze the affected samples on another instrument with a passing RL verification standard for the element(s) of interest.

9.8 Method Sequence

- Calibration of instrument
- Initial Calibration Verification Standard
- Initial Calibration Blank
- LLICV
- Interference Check Solution A
- Interference Check Solution AB
- CRI
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- Samples
- LLCCV
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards.

10.1.2 Instrument Set-Up

Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Startup Procedures

For iCAP Duo 6500

- Turn on power to the chiller
- Click on ThermoSpec Icon; enter analyst initials in login screen
- Click on Plasma icon to start instrument
- Allow to warm up for 30 minutes
- Enter analytical workgroup number (obtained from LIMS) globally under the Instrument menu by selecting Tools, then Options, then Analyst.
- Click on the Sequence tab and enter the sequence by selecting New Autosampler Table, Add Sequence, Add # of spaces.
- Enter the sample locations and IDs
- Press Run Auto-Session button (►) in menu bar.

10.1.2.1 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user.

Operating conditions for axial plasma will vary from 1100 – 1500 watts forward power, 15-19 Liters/min argon coolant flow, 0.5 – 0.7 L/min argon nebulizer flow, 140 – 200 rpm pump rate and a default 1 minute preflush time and 10 second measurement time is recommended for all simultaneous instruments.

10.1.2.2 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

10.1.2.2.1 The Thermo ICP's typically use a Meinhard Nebulizer. The nebulizer flow for each instrument is 1.0 +/- 0.2 mL/min.

10.1.2.2.2 The 6500 Duo instruments automatically perform a wavelength check at start up without user interaction.

10.1.2.2.3 The instrument operating condition finally selected as being optimum must provide the lowest reliable instrument detection limits and method detection limits.

10.1.2.2.4 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and argon pressures must be reoptimized.

10.1.2.2.5 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± the RDL. The upper control limit is the analyte instrument detection limit. Once established, the entire routine is periodically verified annually. In between that time, IEC's are done on a need be basis per analyte. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine must be kept on file. Special cases where continual verification is required are described elsewhere.

10.1.2.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.1.2.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit.

10.1.2.3.2 Determination of limits using reagent water MDLs represent a best case situation and do not represent possible matrix effects of real world samples.

10.1.2.3.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non-consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate.

10.1.2.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum

for three, preferably five, different concentration standards across the range. One of these must be near the upper limit of the range. The ranges which may be used for the analysis of samples must be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made must be documented and kept on file. The upper range limit must be an observed signal no more than 10% below the level extrapolated from lower standards. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst must also be aware that if an interelement correction from an analyte above the linear range exists, a second analyte where the interelement correction has been applied may be inaccurately reported. New dynamic ranges must be determined whenever there is a significant change in instrument response. The linear dynamic range is checked on an annual basis. For those analytes that are known interferences, and are present at above the linear range, the analyst must ensure that the interelement correction has not been inaccurately applied.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves must be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.2.4 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2 Initial Calibration

Calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 8.7. Flush the system with the calibration blank (Section 8.8.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a calibration blank, RL standard and a high level standard. Calibration curve verification is accomplished through the analysis of the ICV, LLICV and CRI standards.

10.3 Equipment Operation and Sample Processing

10.3.1 For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration.

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of three) integrations.

If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICB, ICV, CRI, CCV or CCB must be reanalyzed. The analysis data for the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

10.3.2 Rinse the system with the calibration blank solution (Section 8.8.1) before the analysis of each sample. The suggested default rinse time is one minute. Each ICP instrument may establish a reduction in this rinse time through a suitable demonstration.

10.3.3 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established.

10.3.4 If less than acceptable accuracy and precision data are generated a series of tests are performed prior to reporting concentration data for analyte elements. At a minimum, these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests, as outlined in Sections 10.3.4.1 and 10.3.4.2, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.3.4.1 Post Digestion Spike Addition: If the matrix spike recoveries are unacceptable an analyte spike added to a portion of a prepared sample, or its dilution, must be run, recovery limits equal to 80% to 120% of the known spike value. The spike addition must produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a dilution test (10.3.4.2) should be performed. If both the MS/MSD and post spike fail then a matrix effect must be suspected.

10.3.4.2 Dilution Test: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 10% of the original determination. If not, a chemical or physical interference effect must be suspected.

10.3.5 **CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.4 Continuing Calibration

10.4.1 Check calibration with an ICV following the initial calibration (Section 8.9). Verify calibration with the Continuing Calibration Verification (CCV) Standard (Section 8.9) at the end of the initial calibration sequence (ICV, ICB, ICSA, ICSAB, CRI, project specific RDL standards), after every ten samples, and at the end of an analytical run. At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV must be at a concentration near the mid-point of the calibration curve. Use a calibration blank (Section 8.8.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

A CRI (Section 8.11) must be analyzed after the ICSAB. The concentration of the CRI is 2 – 5 times that of each element's RDL. The linearity of the instrument is confirmed on an annual basis by an LDR standard at +/-10% recovery.

10.4.1.1 The results of the ICV are to agree within 10% of the expected value, and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

10.4.1.2 The results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

10.4.1.3 The results of the CRI must be within 30% of the true value. If they are not, correct the problem and recalibrate the instrument. (Any element may be analyzed on a different ICP that has passed the CRI.)

10.4.2 Verify the interelement and background correction factors at the beginning of each analytical run. Do this by analyzing the ICSA/ICSAB (Section 8.10). Results must be within 80 – 120% of the true value for the analytes of interest in the ICSAB.

10.4.3 When low-level sensitivity is required, a check standard at the requested limit of quantitation is analyzed to confirm the reported detection limit (RDL). This is performed on a project-by-project basis.

10.5 Preventive Maintenance

Whenever instrument maintenance is performed, it is noted in the instrument's Maintenance Logbook.

10.5.1 Daily

Inspect the nebulizer pump tubing from the Autosampler to the Nebulizer. Replace if necessary.

10.5.2 Monthly or as needed

Remove the torch, "shot glass", nebulizer and spray chamber. Clean each with 10% Nitric Acid and rinse with tap water. Coat the inside of the spray chamber and shot glass with concentrated Sulfuric Acid and soak for one hour, then rinse well with DI water. Soak the torch and nebulizer in aqua regia overnight, then rinse with DI water.

10.5.3 Every 6 months

Preventive Maintenance is performed by the Vendor or in-house personnel as follows:

- check the cooling system
- flush/refill the chiller with distilled water and antibacterial conditioner
- clean the instrument to regain intensity
- clean/replace air filters.

11. Data Evaluation, Calculations and Reporting

11.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

11.2 Soil samples

Soil samples are calculated as follows:

$$\mathbf{A} = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$\mathbf{B}\ (\text{concentration in mg/Kg}) = \frac{\text{Concentration of analyte (mg/L)}}{\mathbf{A}}$$

11.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{\mathbf{B}}{\%\ \text{Solids}}$$

11.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

$$\text{Final concentration in mg/L} = \text{Concentration of analyte (mg/L)} \times \text{Dilution Factor}$$

11.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

11.4.1 Total Hardness

Total Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

11.4.2 Calcium Hardness

Calcium Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)]

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Also refer to Section 9 for Quality Control and acceptance criteria.

If the ICSA or ICSAB is outside of the 80 – 120% recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected. The CCV/CCB standard is remade and reanalyzed. If the standard passes, then the data that had failed up to the previous passing standard is reanalyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data that had failed up to the previous passing CCV/CCB is reanalyzed.

The procedure outline above is also conducted for a failing LCS or Method Blank.

If the Matrix Spike does not meet acceptance criteria, a Post Spike is performed. The recovery must be within 80-120% of the true value for aqueous samples and within 80-120% of the true value for soil samples. If these criteria are met, then the Matrix Spike data is reported, with the post spike narrated on the final report. If the post spike fails the acceptance criteria, the Department Manager is notified to determine what type of matrix interference is present, and whether a serial dilution must be performed.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are redistilled and reanalyzed within holding time.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

If the ICSA or the ICSAB are outside of the 80-120% window then the standard in question must be re-analyzed. If the standard failure continues, then check the IECs for the element(s) in question and re-calculate and recalibrate the instrument. The instrument is recalibrated, verified with the ICV/ICB and the ICSA/ICSAB are then re-analyzed. If the standard failure repeats, then a fresh standard is prepared and re-analyzed. If failure continues notify the Department Supervisor.

The RL standards must have a % Recovery of 70-130%. If an element fails the acceptance criteria, the RL standards may be re-analyzed if the element must be included in the analytical event. If the element failure continues, then either re-calibrate the instrument and rerun the affected samples or analyze the sample on another instrument.

If the CRI (low level check standard), is recovered outside of the 70-130% window, the standard may be re-analyzed if the element must be included in the analytical event. If the element failure continues, then either re-calibrate the instrument or analyze the sample on another instrument.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

TABLE 1: Element Wavelengths

TABLE 2: Precision and Accuracy Acceptance Criteria

TABLE 3: Reporting Limits

TABLE 1
ELEMENT WAVELENGTHS

Element	6500 Duo wavelength (nm)
Pb	220.3
Se	196.0
Sb	206.8
As	189.0
Ba	455.4
Be	313.0
Cd	214.4
Co	228.6
Cu	324.7
Cr	267.7
Fe	259.9
Mn	257.6
Mo	202.0
Ni	231.6
Ag	328.0
Tl	190.8
V	292.4
Zn	206.2
Al	396.1
Ca	315.8
Mg	279.0
B	208.9
Si	212.9
Sn	189.9
Sr	421.5
Ti	334.9
Bi	223.0
Na	589.5
K	766.4
S	180.7

TABLE 2
PRECISION AND ACCURACY ACCEPTANCE CRITERIA

	% Recovery LCS		Aqueous % Recovery MS		Soil % Recovery SRM		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	75	125	29	171	20	20
Antimony	80	120	75	125	4	196	20	20
Arsenic	80	120	75	125	81	119	20	20
Barium	80	120	75	125	83	118	20	20
Beryllium	80	120	75	125	83	117	20	20
Boron	80	120	75	125	70	129	20	20
Cadmium	80	120	75	125	82	117	20	20
Calcium	80	120	75	125	83	117	20	20
Chromium	80	120	75	125	80	119	20	20
Cobalt	80	120	75	125	83	117	20	20
Copper	80	120	75	125	83	117	20	20
Iron	80	120	75	125	51	150	20	20
Lead	80	120	75	125	80	120	20	20
Magnesium	80	120	75	125	74	126	20	20
Manganese	80	120	75	125	83	117	20	20
Molybdenum	80	120	75	125	81	119	20	20
Nickel	80	120	75	125	82	117	20	20
Potassium	80	120	75	125	74	126	20	20
Sulfur	80	120	75	125	NA	NA	20	20
Selenium	80	120	75	125	80	120	20	20
Silica (SiO_2)	80	120	75	125	NA	NA	20	20
Silver	80	120	75	125	66	134	20	20
Sodium	80	120	75	125	74	127	20	20
Strontium	80	120	75	125	80	120	20	20
Thallium	80	120	75	125	79	120	20	20
Tin	80	120	75	125	69	131	20	20
Titanium	80	120	75	125	82	118	20	20
Vanadium	80	120	75	125	79	121	20	20
Zinc	80	120	75	125	82	119	20	20

TABLE 3
REPORTING LIMITS

Element	Aqueous (mg/L)	Soil (mg/Kg)
ALUMINUM	0.10	4.0
ANTIMONY	0.05	2.0
ARSENIC	0.005	0.40
BARIUM	0.01	0.40
BERYLLIUM	0.005	0.20
BORON	0.03	1.2
CADMIUM	0.005	0.40
CALCIUM	0.10	4.0
CHROMIUM	0.01	0.40
COBALT	0.02	0.80
COPPER	0.01	0.40
IRON	0.05	2.0
LEAD	0.01	2.0
MAGNESIUM	0.10	4.0
MANGANESE	0.01	0.40
MOLYBDENUM	0.05	2.0
NICKEL	0.025	1.0
POTASSIUM	2.5	100
SULFUR	0.25	10
SELENIUM	0.01	0.80
SILICON	0.50	20
SILVER	0.007	0.40
SODIUM	2.0	80
STRONTIUM	0.01	2.0
THALLIUM	0.02	0.80
TIN	0.05	4.0
TITANIUM	0.01	0.40
VANADIUM	0.01	0.40
ZINC	0.05	2.0

TABLE 4
LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Microwave Assisted Acid Digestion for Metals by 3015A/3051A for Determination by ICP

Reference Method No.: **EPA 3015A, EPA 3051A**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Final Update IV, Revision 1, 2007

1. Scope and Application

Matrices: Aqueous, Solids and Oils.

Definitions: See Alpha Analytical Quality Manual Appendix A

This digestion procedure is a hot acid leach for determining available metals. Subsequently they are analyzed by inductively coupled argon plasma spectroscopy (ICP) for **Sulfur**.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Aqueous: A representative 50mL of sample is digested in 1.0 mL of cHNO_3 and 0.5mL cHCl in a fluorocarbon (PFA or TFM) digestion vessel for 20 minutes using microwave heating. After the digestion process, the sample is cooled and filtered (if necessary) and brought to a 50 mL final volume in a clean sample digestion tube prior to analysis. Mars6 program- “3015A”.

Solids and Oils: A representative 0.25-0.5 g of sample is digested in 1.5 mL of cHNO_3 and 0.5mL cHCl in a fluorocarbon (PFA or TFM) digestion vessel for 10 minutes using microwave heating. After the digestion process, the sample is cooled and filtered and brought to a 50 mL final volume in a clean sample digestion tube prior to analysis. Mars6 program- “3051A”.

2.1 Method Modifications from Reference

None

3. Reporting Limits

Refer to analytical method SOPs.

4. Interferences

4.1 Many samples that contain organics will result in higher vapor pressures which have the potential to cause venting from the vessels. Venting can result in either loss of analytes and/or sample, which must be avoided.

4.2 Other interferences which can cause inconsistent readings are soap, sediment, high pH, and precipitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

NOTE: Caution must be taken when using concentrated HCl or HNO_3. They are corrosives and can cause harm to skin and eyes. When using these corrosives, one should wear a lab coat, gloves, and protective eyewear.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass bottles.

6.2 Sample Preservation

Aqueous samples for total metals analysis are preserved with conc HNO_3 to a pH <2.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2 °C.

7. Equipment and Supplies

7.1 Microwave: CEM MARS 6 Xpress

7.1.1 The microwave unit provides programs with a minimum of 574W, which can be programmed to within ±10 watts of the required power. Typical units provide a nominal 600W to 1200W power. The MARS6 provides up to 1600 W. Microwave temperature is monitored and controlled. The microwave output for digestions is > 900 W of power.

7.1.2 The microwave unit cavity is corrosion-resistant and well ventilated.

7.1.3 All electronics are protected against corrosion for safe operation.

7.1.4 Fluorocarbon (PFA or TFM) Digestion Vessels: 100mL capacity.

7.1.5 A rotating turntable is employed to insure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3rpm.

7.2 Graduated cylinder: 50mL.

7.3 Volumetric Flasks: 50mL.

7.4 Glass Fiber filters: Acid cleaned, 0.7 um nominal pore size

7.5 Whatman 40 or equivalent Filter Paper

7.6 Digestion Tubes: 50mL, calibrated, with caps.

7.7 Analytical balance: calibrated to 0.001 g.

7.8 Pipets: 5.0, 1.0, 0.5 mL.

8. Reagents and Standards

8.1 1:1 Hydrochloric Acid (HCl): Store at room temperature under a hood.

8.2 10% Nitric Acid (HNO_3): Store at room temperature under a hood.

8.3 Concentrated Nitric Acid ($cHNO_3$): Store at room temperature under a hood. Expires according to manufacturer's specified date.

8.4 Concentrated (cHCl): Store at room temperature under a hood. Expires according to manufacturer's specified date.

8.5 DI Water

8.6 1000ppm Single Element Stock Standards: These are commercially prepared standards for various elements. Store at room temperature. Standards expire upon manufacturer's specified date.

8.7 Spiking Solutions

Store at room temperature. Standards expire upon manufacturer's specified date.

8.7.1 ICP Spike Standard #3: Purchased commercially prepared, with a certificate of analysis. Contains the following: 2000ppm Arsenic, 50ppm Cadmium, 500ppm Lead, 2000ppm Selenium, 2000ppm Thallium.

8.7.2 FPS: To a 500mL volumetric flask, add 200mL of DI water and 25mL of concentrated HNO_3. Add 3mL of the well-shaken, room temperature, ICP Spike Standard #3 (Section 8.7.1) and add 25mL of 1000ppm Pb standard. Bring to volume with DI water.

1mL of this solution per 100mL of sample volume will yield the following concentrations in the spiked sample: 0.12ppm Arsenic, 0.051ppm Cadmium, 0.12 Selenium, 0.12ppm Thallium, and 0.51ppm Lead.

8.7.3 ICP Spike Standard #1: Purchased commercially prepared, with a certificate of analysis. Contains the following: 2000ppm Aluminum, 2000ppm Barium, 50ppm Beryllium, 200ppm Chromium, 500ppm Cobalt, 250ppm Copper, 1000ppm Iron, 500ppm Manganese, 500ppm Nickel, 50ppm Silver, 500ppm Vanadium, 500ppm Zinc.

8.7.4 IPS: To a 500mL volumetric flask, add 100mL DI water and 25mL of tHNO3. Add 50.0mL of the well-shaken, room temperature, ICP Spike Standard #1 (Section 8.5.5), 25.0mL of 1000ppm Antimony standard and 2.5mL of 1000ppm Cadmium standard.

Bring to volume with DI water.

0.5mL of this solution per 50mL of sample volume will yield the following concentrations in the spiked sample: 2ppm Aluminum, 2ppm Barium, 0.05ppm Beryllium, 0.2ppm Chromium, 0.5ppm Cobalt, 0.25ppm Copper, 1.0ppm Iron, 0.5ppm Manganese, 0.5ppm Nickel, 0.05ppm Silver, 0.5ppm Vanadium, 0.5ppm Zinc.

8.7.5 Mixed Standard: To a 500mL volumetric flask add 50mL of DI water and 25mL of tHNO3. Add 50mL of each of the following stock standards: 1000ppm Boron, 10,000ppm Calcium, 10,000ppm Magnesium, 1000ppm Molybdenum, 10,000ppm Potassium, 1000ppm Strontium, 10,000ppm Sodium, 1000ppm Titanium, and 1000ppm Tin. Bring to volume with DI water.

0.5mL of this solution per 50 mL of sample volume will yield the following concentrations in the spike sample: 1.0ppm Boron, 10ppm Calcium, 10ppm Magnesium, 1.0ppm Molybdenum, 5ppm Potassium, 1.0ppm Strontium, 10ppm Sodium, 1.0ppmTitanium.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One Blank is digested per matrix batch of 20 samples or less.

9.2 Laboratory Control Sample (LCS)

One LCS is digested per matrix batch of 20 samples or less.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

One Spiked sample is digested per matrix batch of 20 samples or less.

9.6 Laboratory Duplicate

One Duplicate sample is digested per matrix batch of 20 samples or less.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Acid rinse microwave vessels.

- Shake the sample well and dispense the appropriate volume into a numbered vessel.
- To each sample and QC sample aliquot, add cHNO_3 and cHCl.
- Place vessels in a microwave carousel tray.
- Run the appropriate microwave program.
- Allow samples to cool.
- Acid rinse volumetric flasks and Teflon funnels.
- If necessary, filter the samples into 50 mL digestion tubes.
- Bring samples to the appropriate final volume with DI water.
- Transfer the samples in the digestion tubes with caps.

10. Procedure

10.1 Equipment Set-up

10.1.1 Turn on microwave.

10.1.2 Microwave Calibration

10.1.2.1 Microwave Power Verification

The power is controlled through the microwave's temperature feedback control. The unit is checked and calibrated periodically by the manufacturer during PM visits.

10.1.2.2 Microwave Program Verification

All Programs are verified for each Microwave unit on a Quarterly basis and documented in the Microwave Quarterly Calibration Logbook.

- Record the Microwave ID and Microwave Program in the Logbook.
- Record the applicable Program Parameters.
  - TCLP Program: 41% power of 1600 watts, 22 minutes
  - 3015A Program: 170 ± 5C, 20 minutes
  - 3051A Program: 175 ± 5C, 10 minutes
- Run the applicable program

10.1.2.3 Microwave RPM Verification

On an annual basis each microwave is verified to have an average RPM greater than or equal to 3. The unit is checked and calibrated periodically by the manufacturer during PM visits.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Digestion

10.3.1.1 Acid Rinse Fluorocarbon Digestion Vessel, as follows:

10.3.1.1.1 Rinse 1 time with DI water.

10.3.1.1.2 Rinse 1 time with 50% HCl.

10.3.1.1.3 Rinse 1 time with DI water.

10.3.1.1.4 Rinse 1 time with 10% HNO_3.

10.3.1.1.5 Rinse 3 times with DI water.

10.3.1.2 Acid rinse the graduated cylinders following Sections 10.3.1.1.3 through 10.3.1.1.5.

10.3.1.3 Digestion:

10.3.1.3.1 Shake aqueous samples well for Aqueous add 50 mL sample; and for Solids or Oils add between 0.25 and 0.5 g into a numbered vessel.

10.3.1.3.2 PBC: for Aqueous, and Solids/Oils add 50 mL DI water.

10.3.1.3.3 LCS: Use 5mL of PBC extract and add 45mL of DI water; for Aqueous, and Solids/Oils add 50 mL DI water.

10.3.1.3.4 Add the appropriate amount of desired metal spike solutions.

10.3.1.3.5 Matrix Spike (MS): for Aqueous add 50 mL sample; Solids/Oils add 0.25 to 0.5 g of sample.

10.3.1.3.6 Add the spiking solutions (Section 8.7). If the desired metal is not present in these standards, add the appropriate amount of desired metal standard stock 1000ppm solution. The appropriate amount will be determined by the Department Manager.

10.3.1.3.7 To each sample and QC sample aliquot, add the appropriate cHNO_3 and cHCl.

10.3.1.3.8 Proceed to Section 10.3.2.

10.3.2 Place vessels in a microwave carousel tray. Place the full carousel in the microwave and run the appropriate program.

10.3.2.1 Aqueous samples use the 3015A program.

10.3.2.2 Solids and Oils use the 3051A program.

10.3.3 When the microwave program ends, allow the samples to cool.

10.3.4 If any of the samples in the batch contain sediment, those samples along with the method blank and the LCS must be filtered. Place a folded No.40 filter into the appropriate digestion tube, and pre-wet with DI water prior to filtering samples. Filter samples into the 50mL digestion tubes.

10.3.5 Rinse each vessel 3 times with DI water and filter each rinsate into the digestion tube.

10.3.6 Bring samples up to the appropriate volume with DI water.

10.3.7 Transfer the sample in the labeled 50mL digestion tube capped.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

An annual PM is performed by the manufacturer, CEM.

11. Data Evaluation, Calculations and Reporting

Refer to analytical method SOPs.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to analytical method SOPs.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP #1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP# 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP #1728 for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan
SOP #1732 MDL/LOD/LOQ Generation
SOP# 1739 IDC/DOC Generation
SOP# 1728 Waste Management and Disposal

16. Attachments

None.

Microwave Extraction

Reference Methods: **EPA 3546**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, February 2007.

1. Scope and Application

Matrices: Soil, solid, clay, sediment, sludge, solid waste

Definitions: Refer to Alpha Analytical Quality Manual.

Method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 75°C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540C), using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of a variety of semivolatile organic compounds, some of which are: substituted phenols, PCBs, and PCDDs/PCDFs. The extracts are analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Microwave. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Samples are prepared for extraction following the LEAN one-piece flow methodology and loaded into the extraction vessel. The appropriate solvent (See WI/14825 Microwave Extraction Guide) is added to the vessel and sealed. The extraction vessel containing the sample and solvent is heated to the extraction temperature and extracted for 20- 40 minutes. The extraction mixture is allowed to cool. The vessel is opened and the contents are filtered.

The extract is then concentrated and (as needed) exchanged into a solvent compatible with the cleanup or determinative procedure being employed. Extracts are then vialed and transferred to the Analytical Department.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting Limit information may be found in the various determinative method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas, extraction vessels) must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs. If necessary, Florisil, Sulfuric Acid, Silica Gel and/or Sulfur cleanup procedures may be employed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool at room temperature or in the refrigerator before opening.

5.2 During the heating step, some solvent vapors may escape through the vessel liner/seal cover. Follow the manufacturer's directions regarding the vessel assembly and instrument setup to prevent release of solvent vapors to the laboratory atmosphere.

5.3 The instrument contains flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. Follow the manufacturer's directions regarding replacement of extraction vessel seals when frequent vapor leaks are detected.

5.4 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.5 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.6 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.7 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.8 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.9 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.10 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described the in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples must be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

7. Equipment and Supplies

7.1 Spatulas: Stainless steel.

7.2 Beakers: Stainless Steel 250mL.

7.3 Mortar and Pestle: Capable of reducing particle size to <1mm.

7.4 Kuderna-Danish (K-D) apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic Kek clip. Add the Macro Snyder column to the Evaporation Flask. The Micro Snyder Column is attached directly to the Concentrator Tube using the Plastic Kek Clip.

7.4.1 Concentrator tube: 25mL, graduated and calibrated. A ground-glass stopper is used to prevent evaporation of extracts.

7.4.2 Evaporation flask: 500mL. Attach to concentrator tube with plastic kek clips.

7.4.3 Snyder column: Three-ball macro.

7.4.4 Plastic Kek Clips.

7.5 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.6 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.7 Boiling Chips: Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

7.8 Syringe: 1.0mL, 250µL, 25uL, Various sizes for measuring surrogates and spikes.

7.9 Disposable Borosilicate Transfer Pipets.

7.10 Brady labeling system: Thermal label generator.

7.11 Sodium Sulfate glass filtering funnels. Add a plug of glass wool to the base of the 104mm glass funnel. Add approximately 20grams of baked sodium sulfate.

7.12 Glass wool: SUPELCO, silane treated.

7.13 Whatman Filter Paper: used for filtering all Pesticide/8081.(Whatman no.1 or equivalent)

7.14 Graduated Cylinder: 25 and 50mL. Class A.

7.15 N-EVAP: Organomation; Various sizes utilized for micro blowdown.

7.16 Aluminum weighing dishes: VWR Cat#25433-089

7.17 Solvent pump dispenser: Dispensette Organic 100ml

7.18 Analytical Balance: Capable of weighing to 0.01g

7.19 Multi-position Stirring Plates

7.20 Magnetic Stirring Bars

7.21 250mL Erlenmeyer Flask

7.22 Microwave Accelerated Reaction System (MARS): CEM Corporation. The temperature performance requirements necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.0°C and automatically adjusting the microwave field output power within 2 seconds of sensing. Temperature sensor is accurate to ± 2°C and adjustable microwave wattage to 1600W. Temperature feedback control provides the primary performance mechanism for the method.

7.22.1 Microwave extraction vessels: 75ml and 100mL (Plus). With plugs and caps. Capable of accommodating 1g to 30g samples. Vessels are transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure requirements (minimum conditions of 75°C and 200psi) necessary to perform this procedure. Follow the manufacturer's instructions regarding cleaning, handling, and sealing the vessels.

7.22.2 Kevlar sleeves.

7.22.3 Extraction Vessel Turntable: Used to hold and rotate the extraction vessels during extraction.

8. Reagents and Standards

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's RO water treatment system.

8.2 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a glass or stainless steel beaker. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent. Sodium sulfate is also used for filtering.

8.3 Ottwa Sand: VWR catalog #JT3382-5. Purified by baking at 400C for 4 hours in a shallow tray or stainless steel beaker.

8.4 Hexane: Pesticide quality.

8.5 Acetone: Pesticide quality.

8.6 Dichloromethane: Pesticide quality.

8.7 Nitrogen Gas: Reagent grade, used to purge and pressurize the extraction cell and as the concentration gas in the Turbovap II auto-concentrator units and the N-EVAP.

8.8 Spiking Solutions: Commonly used surrogate and LCS/MS spiking solutions used in the extraction steps are listed in WI/14825 Microwave Extraction Guide. Additionally, the WI/14825 Microwave Extraction Guide has a complete listing of all surrogate and LCS/MS spiking solutions. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.9 Extraction Solvents: This method has been validated using a 1:1 mixture of hexane and acetone, 1:1 mixture of methylene chloride and acetone, or 100% Methylene Chloride for matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water of the surface of the solid particles. The water immiscible solvent extracts organic compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems. When 100% Methylene Chloride is used, water is added as a catalyst to absorb microwave energy for method 8270.

8.10 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 16 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. Specific QC requirements of the analytical methods are listed in WI/14825 Microwave Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate or sand for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in

addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for all Massachusetts Contingency Plan (MCP) methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions are listed in WI/14825 Microwave Extraction Guide.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD is carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. Requirements for MS and MSD are listed in WI/14825 Microwave Extraction Guide.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. Requirements for Duplicates are listed in WI/14825 Microwave Extraction Guide.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/14825 Microwave Extraction Guide.

9.8 Method Sequence

Refer to Section 10.

10. Procedure

All soil microwave soil extracts follow the LEAN “one-piece flow”. All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note the matrix “type” along with any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the logbook.

10.1 Sample Preparation and Extraction

10.1.1 Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Immediately after scanning, the samples batched into the ELN to create the Work Group. Labels are printed and placed on the cap of the soil container. See Work Instruction 2421, Labeling and Generating Work Groups and Batches.

10.1.2 All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

10.1.3 During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the chemist must contact their supervisor or manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Any artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample and are not to be included. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. Record the sample matrix "type/description" in the comments section of the ELN using the Sample Matrix Description spreadsheet.

10.1.4 When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

10.1.5 The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or Ottawa Sand) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

10.1.6 **MARS Microwave System Operation:** Rinse the reaction vessels, caps, and plugs with 1:1 Acetone/DCM.

10.1.6.1 See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Typically 15-30 grams of the sample and 15grams of sodium sulfate is extracted. Transfer the sample into the reaction vessel from the beaker or weighing tray. Add the appropriate surrogate and spiking solution. (Refer to WI/14825 Microwave Extraction Guide). Sodium Sulfate is used for the QC substrate for all methods except 8270 and 8270 SIMTECH where Ottawa Sand is used for 8270 QC.

10.1.6.2 **For all Methods except 8270:** Add 35mL of 1:1 Hexane:Acetone, 1:1 DCM:Acetone or 100% DCM (or the appropriate extraction solvent) to each reaction vessel. (Refer to Table 1). The amount of solvent used in the extraction will depend on the sample matrix. Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.3 Place a plug and a cap on each reaction vessel.

10.1.6.4 Place all labeled vessels onto the carousal and place the carousal into the microwave.

10.1.6.5 On the front panel of the microwave, select "Use This Method" or UTM from the User Directory. NOTE: UTM-75 is used for the smaller 75mL vessels while UTM-Plus is for the larger 100mL "Plus" vessels.

The following parameters are loaded with "Use this Method" program:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 30 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.6 Remove each reaction vessel from the carousel for cooling.

10.1.6.7 Once samples have cooled to room temperature, twist the cap off the reaction vessel and filter through a sodium sulfate funnel and into a labeled KD Flask and concentrator tube or Buchi tube. The sodium sulfate funnel contains glass wool and approximately 15-30 grams of sodium sulfate. Due to contamination issues, pesticide samples require filter paper instead of the glass wool. Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.8 Proceed to sample concentration and cleanup step as required by the analytical method for the analyte(s) of concern. (Refer to Table 1).

10.1.6.9 **For 8270 and 8270 SIM TECH:** See Work Instruction WI/2421 for proper labeling procedures and one-piece flow operation. Transfer 30 grams of the sample (a requirement) into the reaction vessel from the beaker or weighing tray. Note: sodium sulfate is **not** mixed with the sample. For QC samples, Ottawa sand is used. Add the appropriate surrogate and spiking solution. (Refer to WI/14825 Microwave Extraction Guide). After adding the sample to the reaction vessel, using a 1.0mL syringe, add 1.0mL of DI water to the sample.

10.1.6.10 Add 40mL of 100% DCM to each reaction vessel. (Refer to Table 1). Additional solvent may be necessary in some cases depending on sample matrix. Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.11 Place a plug and a cap on each reaction vessel.

10.1.6.12 Place all labeled vessels onto the carousal.

10.1.6.13 On the front panel of the microwave, select "8270" from the User Directory. NOTE: 8270-75 is used for the smaller 75mL vessels while 8270-Plus is for the larger 100mL "Plus" vessels.

The following parameters are loaded with 8270 method:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 10 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.14 Once samples have cooled to room temperature, filter through a sodium sulfate funnel (using DCM only) and into labeled Bucchi vessels for DRO, ETPH, and all ABN samples (See Section 10.2.2). Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.15 ETPH Analysis: Filter the sample extract through a 20gram sodium sulfate funnel containing glasswool into a 250mL Erlenmeyer flask. Add 3 grams of Deactivated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes @ 650rpm. Filter the extract through a funnel containing filter paper and collect into a Bucchi vessel for concentration, see Section 10.2.2.

10.1.6.16 Proceed to sample concentration. Note all DRO, ETPH, and ABN products are concentrated using the Buchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique

10.2.1.1 Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a hot water bath(SEVAP), (heated to approximately 75^{o}C so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.2 If a Hexane exchange is required (see Table 1), when the sample volume reaches 5-15mL, remove the condenser from the Synder Column and add 20mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow sample to continue to concentrate to 15-20mL and exchange with another 20mL of hexane. Allow the sample to boil until the intensity decreases (little to no chatter in the Snyder column). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to 15mL.

10.2.1.3 Remove the KD apparatus from the water bath and remove the plastic Kek clip. Wipe the joint of the flask and the concentration tube with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool for 5minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI 2421).

10.2.1.4 Place the Concentrator tube on the N-EVAP. Using a disposable pipet, direct the nitrogen over the sample. The N-EVAP is set at 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they are reduced to the appropriate volume (see S-Evap/N-Evap Concentration Standard Process WI#18528 for listing of appropriate volumes).

10.2.1.5 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Bucchi

The Bucchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Bucchi will recover >95% of solvent emissions. Refer to Alpha SOP/12838 for Buchi concentration set-up and procedure.

10.3 Preventive Maintenance

10.3.1 Microwave System (MARS):

10.3.1.1 The instrument must be kept clean. Wipe the inside of microwave with soap and water and dry with a cloth as needed.

10.3.1.2 All microwave cells, caps and plugs are to be dish washed and rinsed with solvent prior to use. Additionally, the sleeves must be handled with care. If the edge of the sleeve is dented or chipped it will not hold pressure.

10.3.2 Analytical Balance

10.3.2.1 All balances are checked for accuracy daily and calibrated/serviced every six months by an instrument service company. All service records are kept on file.

10.3.2.2 Keep balances clean. Brush of any sample spills immediately.

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11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issued through the Qualtrax System.

12.2 If the KD Concentrator tube is allowed to run dry, the extract volume is spilled, etc. the sample must be re-extracted.

12.3 Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

12.4 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

Form 02-50 Sample Cleanup and Vialing Guide

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/2423 GC Mass Spec Extract Vialing Procedure

WI/2426 GC Extract Vialing Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

WI/14825 Microwave Extraction Guide

SOP/12838 Buchi Concentration

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

LIMS Product Code	Extraction Solvent	Exchange Solvent Required	Typical Final Volume	Appropriate Cleanup Technique
8082	1:1 Hexane/Acetone	hexane	5-10 mL	Sulfuric acid/ Sulfur
8081	1:1 DCM/Acetone	hexane	10 mL	Florisil
8270 SIM	DCM	---	1mL	---
8270/8270SIMTECH	DCM	---	1 mL	---
TPH *	DCM	---	1 mL	---
EPH	DCM	hexane	1 mL	Silica gel Fractionation
EPH-TPH**	DCM	hexane	1 mL	---
ETPH	DCM	---	1 mL	Silica gel

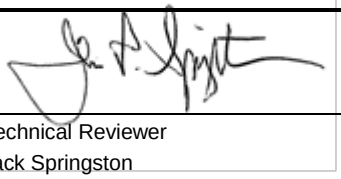
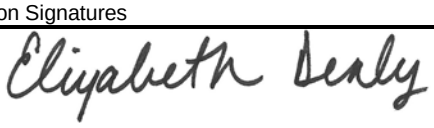
***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

****EPH-TPH includes the following LIMS Products: NJEPH-TPH-CAT1, NJEPH-TPH-CAT2**

TRC Standard Operating Procedures

Title: **PCB Indoor Air Sampling**	Procedure Number: **BSI 004**
	Revision Number: **0**
	Effective Date: **December 2016**

Authorization Signatures

			
Technical Reviewer Jack Springston	Date 12/9/16	BSI Practice Quality Coordinator Elizabeth Denly	Date 12/9/16

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ATTACHMENTS

Attachment A Example Field Sampling Data Sheet

Attachment B Example Chain-of-Custody Form

1.0 INTRODUCTION

1.1 Scope & Applicability

This Standard Operating Procedure (SOP) was prepared to provide guidance to TRC personnel in the logistics, collection techniques, and documentation requirements for collecting representative indoor air samples for polychlorinated biphenyl (PCB) analysis. These are standard (i.e., typically applicable) operating procedures that may be changed, as required, dependent upon site conditions, equipment limitations, or limitations imposed by the procedure. In addition, other local, state or federal regulatory requirements may be above and beyond the scope of this SOP and should be followed, if applicable. In all instances, the actual procedures used should be thoroughly documented and described in the field notes. The project-specific work plan (or equivalent) should be consulted to verify sampling requirements and details, as specified by the contractual agreement with the client.

This SOP is applicable to all Building Sciences and Industrial Hygiene (BSI) indoor air PCB sampling programs, unless otherwise noted in project documents.

1.2 Summary of Method

This SOP is based on EPA method TO-10A, *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*, January 1999.

The objective of indoor air sampling is to obtain a representative sample of air for laboratory analysis of constituents of interest (i.e., PCBs) at a given site. This objective requires that the sample be of sufficient quantity (i.e., volume) and quality for analysis by the selected analytical method. Indoor air samples for PCBs are typically collected using a low-volume sampling pump equipped with a glass cylinder sampling cartridge containing a polyurethane foam (PUF) plug sample medium for the collection of gas and particulate phase PCBs.

The PUF sampling medium is designed to trap airborne organic vapors. Air is drawn through the PUF sample medium with a low-volume pump. This feature is designed to permit the motor to operate at low sampling flow rates for extended periods without motor failure from overheating. The sampling cartridge is constructed of borosilicate glass, is filled with the PUF plug, and is connected to the sampling pump by way of flexible tubing.

1.3 Equipment

The following list of equipment may be utilized when conducting indoor air sampling for PCBs. Project-specific conditions or requirements may warrant the use of additional equipment and/or deletion of items from this list.

- Appropriate level of personal protective equipment (PPE) such as nitrile gloves and safety glasses, as set forth in a site-specific health and safety plan (HASP) or job safety analysis (JSA).
- Low-Volume, Continuous Flow Sampling Pump – SKC Leland Legacy Monitor and Sampling Pump (or equivalent): 2.5 to 15 liters per minute (lpm).

- Battery Pack – Rechargeable lithium-ion battery pack (or equivalent) compatible with the personal sampling pump.

- Sampling Cartridge – Constructed from a 20 millimeter (mm) inside diameter (I.D.) by 10 centimeter (cm) long borosilicate glass tube drawn down to a 7-mm outside diameter (O.D.) open connection (e.g., PUF/Glass Fiber Filter SKC Part No. 226-126 22 x 100 mm) attached to the pump by way of flexible tubing.

- Polyurethane Foam (PUF) Plugs – Cut into a cylinder, 22-mm (I.D.) and 7.6-cm long, fitted under slight compression inside the sampling cartridge. The PUF plug materials should be polyether type elastomer (density 0.0225 g/cm^3), not polyester. Plugs should be slightly larger in diameter than the internal diameter of the cartridge. Pre-cleaned PUF plugs and glass cartridges can typically be obtained from the analytical laboratory.

- Teflon® End Caps – End caps for the sample cartridge must fit tightly to provide an adequate seal to prevent pre- or post- sampling exposure to other potential sources of the target analytes.

- Flexible Tubing – Used to connect the cartridge assembly to the sampling pump (e.g., Tygon® tubing).

- Sample Cartridge Shipping Containers – The analytical laboratory should provide individually labeled containers that will be used to transport the sample cartridges to the field location and back to the laboratory for analysis. This may include aluminum foil wrapped around the cartridge and a glass jar large enough to hold the cartridge.

- Nitrile Gloves – For handling the cartridges.

- Kevlar Gloves – To prevent against cuts from possible glass breakage.

- Ice Chests – To hold samples at <4°C during shipment to the analytical laboratory.

- Gel Packs – To cool samples in ice chests in lieu of ice.

- Primary Flow Calibrator (Bios Defender 510-H, or equivalent).

- Rotameter.

- Temperature and Humidity Meter – Dickson TP425 Temperature and Humidity Logger, or equivalent. (optional).

- Barometric Pressure Meter – Extech Instrument, or equivalent (optional).

- Air Flow Velocity Meter – TSI 964 Straight Air Velocity Probe for use with Q-Trak Model 7565, or equivalent (optional).

1.4 Definitions

EPA – United States Environmental Protection Agency.

PCBs – Polychlorinated biphenyls. A group of 209 compounds in which chlorine atoms replace the hydrogen atoms in biphenyl molecules. PCBs were used in industry in electrical insulators and in the manufacture of plastics until 1979.

PCB Aroclor – Trade name for various mixtures of chlorinated biphenyl congeners manufactured by Monsanto Chemical Company which were sold in the United States. A PCB Aroclor may consist of over 100 different individual PCB congeners. There are nine common PCB Aroclor mixtures.

PCB Congener – Any single, unique well-defined chemical compound in the PCB category. The name of a congener specifies the total number of chlorine substitutes and the position of each chlorine. There are 209 separate PCB congeners based on the number and position of the chlorine atoms on the biphenyl ring structure.

PCB Homolog – A subcategory of PCB congeners having equal numbers of chlorine substituents. For example, tetrachlorobiphenyls are all PCB congeners with exactly 4 chlorine substituents that may be in any arrangement. There are ten different PCB homologs.

Soxhlet Extraction – A procedure for extracting nonvolatile and semivolatile organic compounds from solid matrices. Samples are extracted using an appropriate solvent in a Soxhlet extractor. The solvent is heated to reflux; a condenser cools the solvent vapor as it drips back down into the thimble holding the solid matrix. The thimble fills with warm solvent and the PCBs dissolve in the warm solvent. When the thimble is almost full with solvent, it is emptied by a siphon and the solvent returns to a distillation flask. This cycle is repeated for 16-24 hours at 4-6 cycles per hour. During each cycle, some of the PCBs dissolve in the solvent. After many cycles, the PCBs are concentrated in the distillation flask.

1.5 Health & Safety Considerations

TRC personnel will be on site when implementing this SOP. TRC personnel will use the appropriate level of PPE as required. The Project Manager or TRC's Safety Director can address any questions or safety concerns. Project-specific safety considerations should be documented in the project-specific work plan (or equivalent).

1.6 Cautions and Potential Problems

- Broken glass if the PUF sampling cartridge is dropped. Use of Kevlar gloves can protect against cuts from broken glass.

1.7 Personnel Qualifications

Since this SOP will be implemented at sites or in work areas that entail potential exposure to toxic chemicals or hazardous environments, all TRC personnel must be adequately trained. Project and client-specific training requirements for samplers and other personnel on site should be developed in project planning documents, such as the sampling plan or project-specific work plan. These requirements may include:

- Occupational Safety and Health Administration (OSHA) 40-hour Health and Safety Training for Hazardous Waste Operations and Emergency Response (HAZWOPER) workers and 8-hour annual HAZWOPER refresher training.
- OSHA 10-hour Construction Industry Outreach Training.

- Site-specific safety training.

2.0 PROCEDURES

2.1 Pre-Sampling Activities

1. Determine the required sampling volume, minimum/maximum flow rates, minimum/maximum sampling duration, and number and location of samples needed, dependent upon project objectives, sampling method limitations, and required PCB reporting limits. Consult with the Project Manager or the scope of work.

2. Field Measurement Equipment: If ancillary measurements are required, the associated equipment must be calibrated appropriately. Check with the Project Manager to determine if ancillary measurements are required.
    a. Temperature and Humidity: If erratic measurements are observed, consult the manufacturer for calibration requirements.
    b. Barometric Pressure: If erratic measurements are observed, consult the manufacturer for calibration requirements.
    c. Air Flow Velocity: Records of calibrations will be provided by the rental company and maintained in the project file.

2.2 Sampling Procedures

1. Flow Rate Calibration: Prior to use, the flow rate on the pump is calibrated by setting the flow rate to the respective set points using a suitable flow-measuring device. A primary flow calibrator (Bios Defender 510-H, or equivalent) can be used to calibrate field sampling pumps or a properly calibrated rotameter can be used. Calibration information (i.e., date, times, equipment make/model/serial number, and results of calibration) must be recorded in the field notes. The annual calibration of the primary flow calibrator is typically provided by the rental company and should be maintained in the project files.
    a. Don a new pair of nitrile gloves.
    b. Remove the aluminum foil, if present, from the pre-cleaned cartridge assembly (return the aluminum foil to the jar for later use).
    c. Remove the Teflon® end caps from the cartridge and return to the jar for later use.
    d. Attach the cartridge to the pump with flexible tubing (e.g., Tygon®). (NOTE: depending on the diameter of the tubing used, an adapter may be required to connect the cartridge and tubing.)
    e. Measure the flow rate of each personal sampling pump, with the PUF-installed sampling cartridge in line, prior to the onset of sampling.
    f. If using a battery-operated pump, allow the pump to run for 10 minutes prior to checking the flow rate.
    g. Connect the primary/secondary flow calibrator to the pump inlet.
    h. Adjust the flow rate to the desired sampling value.
    i. Observe the flow rate on the primary/secondary flow calibrator and verify it as constant for a minimum of 20 seconds.
    j. Record the flow rate on the Field Sampling Data Sheet. See Attachment A for an example Field Sampling Data Sheet.

2. Indoor Air Sampling for PCBs:
    a. Don a new pair of nitrile gloves.
    b. Position the sampling assembly with the intake pointed downward or in a horizontal position.
    c. Fasten the sampler to a location (e.g., on a tripod) with the sampling cartridge intake in the approximate breathing zone of the occupants.
    d. Begin air sampling by turning the power switch to the pump on.
    e. Activate the elapsed time meter and record the start time on the Field Sampling Data Sheet.
    f. At the conclusion of the sampling period, record the stop time on the Field Sampling Data Sheet and perform a post-sampling flow rate check.

3. Post-sampling Flow Rate Check:
    a. Measure the flow rate of each sampling pump, with the PUF-installed sampling cartridge in line.
    b. Connect the primary/secondary flow calibrator to the pump inlet.
    c. Observe the flow rate on the primary/secondary flow calibrator and verify it as constant for a minimum of 20 seconds.
    d. Record the flow rate on the Field Sampling Data Sheet.
    e. Measure and document flow rate drift between the initial and final flow rate measurements.
    f. Record the average flow rate for use in calculating the total sample volume.

4. Post-Sampling Activities:
    a. Remove the PUF-installed sampling cartridge from the pump and wrap it with its original packaging (e.g., aluminum foil).
    b. Replace the Teflon® end caps on the cartridge.
    c. Place the cartridge back into its original sealed and labeled container.
    d. Place the container in an ice chest under ice or gel ice packs (< 4°C).
    e. Complete the chain-of-custody (COC) form. See Attachment B for an example COC form.
    f. Transport the samples to the analytical laboratory for processing.

2.3 Ancillary Measurements

Field measurements may be required in association with the indoor air sampling for PCBs. Continuous temperature, barometric pressure, and/or humidity measurements may be required to be taken in each sampling location, using appropriate sensors and logging devices. Air flow velocity measurements may be required for supply and exhaust (or return) air vents in each room. It should be noted that sample volumes are many times corrected for temperature and barometric pressure, which may make these measurements necessary with each sampling event in each sampling location.

3.0 QUALITY ASSURANCE/QUALITY CONTROL

The following list is a summary of quality assurance/quality control (QA/QC) procedures that can be used to help ensure the accuracy and precision of the sampling method. The collection of specific field quality control samples will be specified in the project-specific planning documents.

3.1 Field Blanks

Field blanks consist of clean sample media. Field blanks accompany samples to the site and return to the laboratory in the same cooler or shipping container. Field blanks are used to ensure that there is no contamination as a result of the shipment/transportation/on-site storage activities. Typically, one field blank per day of sampling is collected for each analytical parameter. Field blanks should be handled the same as the regular samples, including removing from the sample container, removing and replacing the aluminum foil wrap (if present), and then placing back into the sample container. No air should be drawn through the field blank.

3.2 Cooler Temperature Blanks

Cooler temperature blanks consist of a sample container filled with non-preserved water (potable or distilled) and are included in all coolers which contain samples which require temperature preservation. The laboratory uses these temperature blanks to ensure that proper preservation of the samples has been maintained during sample shipment. The temperature of these blanks must be <4 ºC to demonstrate that proper preservation has been maintained. The laboratory records the results of the temperature blanks on the COC or sample login form immediately upon receipt of the samples at the laboratory, prior to inventory and refrigeration.

3.3 Field Duplicates

Field duplicates are collocated samples. Collocated samples are two samples collected next to each other in the same position at the same location, and at the same time. Collocated samples require two separate sample collections at the same location. Field duplicates are used to assess the sampling and analytical reproducibility. Field duplicates, when required, are typically collected at a frequency of one per day of sampling for each analytical parameter.

3.4 Field Surrogate Spikes

Media used for sampling can be pre-spiked with a field surrogate compound (e.g., 4,4'-Dibromobiphenyl) known not to exist in the environment and not to interfere with the quantitation of PCBs. Accuracy of field surrogate spikes is measured in terms of the percent recovery (%R) and, when utilized, is used to evaluate collection and extraction efficiency. The media are spiked in the laboratory, sent out to the field, and returned to the laboratory for analysis.

3.5 Field Spikes

Field spikes are clean PUF cartridges used for sampling, pre-spiked with a known concentration of PCBs and used to evaluate accuracy for the air matrix. The media are spiked in the laboratory, sent out to the field with all sample media, and returned to the laboratory for analysis. No air is drawn through the cartridges used for field spikes. The PUF cartridge used as a field spike remains in a sealed container and is not used during the sampling period. Field spikes, when utilized, are typically submitted at the same frequency as field blanks. Accuracy of field spikes is measured in terms of the %R.

3.6 Media Certification Checks

PUF media for all analyses are typically certified clean from the laboratories. The certifications are performed as batch checks and results are stored in the project files.

3.7 Breakthrough Checks

Breakthrough checks can be performed by utilizing two cartridges connected in series (front and back air cartridges) and performing separate analyses of each cartridge. When utilized, separate analyses of both PUF cartridges for PCBs can be performed for an assessment of analyte breakthrough. The percent breakthrough is calculated to determine the sampling efficiency. Breakthrough on the back half of the trap should be < 10% of the front half of the trap or < 100 nanograms (ng) per trap.

4.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

Information relevant to the indoor sampling location should be recorded during each sampling period (e.g., numbers of interior and exterior doors and windows, number of windows open and closed, type of ventilation system and its status during each sampling period, room temperature, and any other conditions that might affect PCB concentrations in air over time).

Calibration information (i.e., date, times, equipment make/model/serial number, and results of calibration) should also be recorded.

An example Field Sampling Data Sheet and an example COC form are presented in Attachments A and B, respectively.

5.0 REFERENCES

EPA TO-10A *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)*, January 1999.

US Green Building Council (USGBC) Leadership in Energy and Environmental Design (LEED) - Indoor Environmental Quality (EQ) – Indoor Air Quality.

6.0 SOP REVISION HISTORY

REVISION NUMBER	REVISION DATE	REASON FOR REVISION
0	DECEMBER 2016	Not applicable.

Attachment A

Example Field Sampling Data Sheet

Project Name:		Project Number:
Date:	TRC Field Representative(s):	

Calibrater Model/Serial Number:

Area ID:			Pump Serial Number:			
Sample ID:			Cartridge Serial Number:			
	Hour	Minute		Hour	Minute	Total Time:
Start Time:			Stop Time:			0
Start Calibrations:	1		End Calibrations:	1		Average Cal:
	2			2		0.000
	3			3		Total Volume:
Avg Start Calibration:		0.000	Avg End Calibration:		0.000	0.0

Temperature/Pressure readings:				
	Reading Time:	Temp:	Pressure:	Notes:
Start:				
Intermittent:				
Intermittent:				
Intermittent:				
End:				

Area ID:			Pump Serial Number:			
Sample ID:			Cartridge Serial Number:			
	Hour	Minute		Hour	Minute	Total Time:
Start Time:			Stop Time:			0
Start Calibrations:	1		End Calibrations:	1		Average Cal:
	2			2		0.000
	3			3		Total Volume:
Avg Start Calibration:		0.000	Avg End Calibration:		0.000	0.0

Temperature/Pressure readings:				
	Reading Time:	Temp:	Pressure:	Notes:
Start:				
Intermittent:				
Intermittent:				
Intermittent:				
End:				

Attachment B

Example Chain-of-Custody Form

TRC	TO-10A PCB AIR SAMPLE CHAIN OF CUSTODY FORM			
Client Name:	Project Name and Address:		Samples Collected By:	Page: 1 of 1
Date:	Weather Conditions:	Requested Turnaround Time:	Project Manager:	Project Number:

TO-10A PCB AIR SAMPLE INFORMATION

Air Sample ID No.	Cartridge Identification	Floor	Room/Area Description	Start Flow (L/min)	Stop Flow (L/min)	Start Time	Stop Time	Total Sampling Volume (L)	Ventilation System On	Analysis

Special Instruction to Laboratory:

CHAIN OF CUSTODY INFORMATION AND LABORATORY INFORMATION

Relinquished By:	Date	Time	Received By:	Date	Time	Method Of Submittal	
I. (Print):						Field	
(Sign):						Walk In	
II. (Print):						Fed-Ex	
(Sign):						Others	
TRC Comments:	Analysis Codes: (1) = 8082A; (2) = 680; (3) = 1668; (4) Soxhlet (3540) extraction		Lab Comments:			Analyzed By:	
						Print Name:	
						Sign:	

Contest Standard Operating Procedures

Compendium Method TO-4A/ TO-10A
By Soxhlet extraction (Method 3540C)

Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Polyurethane Foam (PUF) Sampling.

Approved:

Tod Kopyscinski
Laboratory Director

Katherine F. Allen
QA Officer

Revision Number: 13

NON-CONTROLLED COPY

Change Record

Revision	Date	Responsible Person	Description of Change
Original	05/05/2003	J. Beane/ S. Kocot	Original, including MCP Data Enhancement Criteria and NELAP format
1	01/13/2004	D.Damboragian	QC criteria updates, for AZ audit
2	11//19/2006	D.Damboragian	Instrument parameter changes
3	2/7/2007	D.Damboragian	Instrument and procedure revisions
4	3/26/2010	F. Derose	Updates made to sections: 1.0,5.0,6.0,7.0,9.0, & 10.0
5	03/16/2012	F. Derose	Updates to include Pesticides.
6	03/27/2012	F. Derose	Update to sections 12.2.3, 12.2.4, and 12.2.5 (frequency of 10 samples not 20).
7	08/15/2013	KFA	Updates from 2013 internal audit: Sec 1.0 (Reporting Limits updated), Sec 8.4(PUF lot check freq not 1 in 10 but per lot), Sec 8.6 (exp on bin for each lot), Sec 8.7 (take out aluminum foil rinsed with Hexane), Sec 9.1(ext'ed in 7 days), Sec 9.8a (Soxhlet cycle 18$\pm$2 hrs.), Sec 10.0 (addition of instruments), Sec 12.2.5 (take out 65-125%, use control chart limits), and Sec 12.0 (addition of RL ver.).
8	7/30/2014	KFA	Sec 12.2.1 (RSD criteria is $\leq$20% not $\leq$15%)
9	9/25/2014	KFA	Updates from annual internal audit: Section 6.23 (add new tables).
10	09/14/2015	KFA	Updates from annual Internal audit: Sec 2.0 (Delete ASE), Sec 5.0 (delete ASE and replace turbovap with Buchi), sec 9.0 (delete ASE procedure), added Buchi procedure, Sec's 10.1 and 10.2 (0.5um df not 0.32), Sec's 12.2.3 and 12.2.4(Changed 10 samples to 20 samples, Sec 12.2.3 (add breakdown criteria), and Sec 17.0 (delete ASE ref and replace turbovap with Buchi ref.).
11	08/31/2016	KFA	Updates from annual internal audit: sec 6.17 (addition of Cal points 5 and 10), Sec 10.1 (replace instrument 3 with 10 and add new column model number), and Sec 10.2 (new column model).
12	06/21/2017	DJD	Update from client inquiry: Section 12.2.3 (CCV criteria updated).
13	09/11/2017	KFA	Update from annual internal audit: Sec's 8.1.4 and 8.1.5 (new PUF lot check procedures), Sec 9.6 and 9.7 (new spiking levels), Sec 12.2.1 (ICV limits of 15% from 20%) and Sec 12.2.9 (Change in confirmation procedure)

Distribution/Training List

See Employee Training Record File for signed training statements for trained users.

1.0 SCOPE AND APPLICATION

Methods are TO-4A/ TO-10A to determine the concentrations of pesticides and polychlorinated biphenyls (PCBs) in air as aroclors extracted from polyurethane foam (PUF) sampling media. Open-tubular, capillary columns are employed with electron capture detectors (Micro-ECD). The target compounds are determined by dual-column analysis system.

Compound	Reporting Limit (µg)	Compound	Reporting Limit (µg)
Aroclor 1016	0.20	Gamma-BHC	0.03
Aroclor 1221	0.20	Beta-BHC	0.05
Aroclor 1232	0.20	Delta-BHC	0.05
Aroclor 1242	0.20	Heptachlor	0.05
Aroclor 1248	0.20	Aldrin	0.05
Aroclor 1254	0.20	Heptachlor Epoxide	0.05
Aroclor 1260	0.20	4,4-DDE	0.04
Aroclor 1262	0.20	Endosulfan-I	0.05
Aroclor 1268	0.20	Endosulfan-II	0.08
Hexachlorobenzene	0.05	4,4-DDT	0.04
Alpha-BHC	0.05	Endrin Aldehyde	0.08
Dieldrin	0.004	Endosulfan Sulfate	0.08
Endrin	0.08		
4,4-DDD	0.04		
Methoxychlor	0.5		
Endrin Ketone	0.08		

2.0 SUMMARY OF METHOD

The procedure is based on the adsorption of polychlorinated biphenyls (PCBs) and pesticides from ambient air on polyurethane foam (PUF) using a low (TO-10) or high (TO-4) volume sampler. Samples are collected using pre-cleaned, evacuated and dried cartridges (PUFs). Once the air sample is collected, the sorbent cartridge is put into a canister with a screw top (to contain the sample and prevent any influence from environmental factors), labeled and sent to the laboratory for analysis. It is highly recommended that the client sends a blank filter and sorbent cartridge with each sample order. Upon receipt, the sample is logged into the LIMS using information from the client Chain-of-Custody and delivered to the laboratory for analysis. TO-4A is sampled with a Quartz Fiber Filter and PUF. TO-10A is sampled with PUF only. At sample receipt, the sample type is documented and logged in for each individual test dependent upon sample volume and flow-rate.

For low volume PUFs (TO-10A), a sampling rate of 1-5ml/min for 4-24 hours is used.

For high volume PUFs (TO-4A), a sampling rate of ~8cfm for 4-24 hours is used.

TO-10 (Low Volume) cartridges and TO-4 (High Volume) cartridges with quartz filter are extracted using:

Method 3540 (Soxhlet Extraction) with 5% Diethylether in Hexane.
See Soxhlet SOP (Method 3540).

The extract is then concentrated prior to analysis.

3.0 INTERFERENCES

3.1 Interferences co-extracted from the samples will vary from matrix-to-matrix, possible sources of interference are listed below:

3.1.1 Contaminated solvents, reagents, or sample processing hardware.
3.1.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
3.1.3 Compounds extracted from the sample matrix to which the detector will respond.
3.1.4 Co-elution of target analytes.

3.2 Interferences by phthalate esters can pose a major problem in PCB determination. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. For this reason, avoid the use of plastics in the laboratory.

3.3 Cross-contamination of clean glassware can occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

3.4 Subtracting blank values from sample results is not permitted.

3.5 The PUF adsorbent is white and yellows upon exposure to light. The “yellowing” of PUF will not affect its ability to collect pesticides or PCBs.

3.6 Clean-up procedures include: Florisil Clean-up (Method 3630) See SOP, Sulfur Removal (Method 3660B), and Sulfuric Acid Clean-up (Method 3665A). Note: Sulfuric Acid Clean-up only performed on PCBs.

4.0 SAMPLE PRESERVATION/STORAGE/HOLDING TIME

All samples should be extracted within 7 days after collection and analyzed within 40 days of extraction. All samples should be stored at <4^{o}C or below until extracted. All analysis should be completed within 40 days of extraction.

5.0 EQUIPMENT & SUPPLIES

5.1 PUFs high volume, purchased from Tisch, P/N TE-1010 or equivalent.
5.2 Quartz Fiber Filters (TO-4A), SKC, P/N 225-1821 or equivalent. (These are baked at 400^{o}C for 5 hours.)
5.3 PUFs low volume, purchased from SKC, P/N 22692 or equivalent.
5.4 Storage canister and screw-cap (or aluminum foil)
5.5 Capillary column, gas chromatograph with splitless injector, auto injector, ECD and data system.
5.6 Volumetric glassware
5.7 Microsyringes
5.8 Vials, 2-mL crimp top and 4-mL screw cap
5.9 Water bath
5.10 Filter paper: Grade 413, 11cm
5.11 Glass funnels

5.12 White cotton gloves: for handling cartridges and filters
5.13 Concentrator tubes and a nitrogen evaporation apparatus with variable flow rates. Buchi Concentration Workstation or equivalent
5.14 Aluminum foil
5.15 Soxhlet Extractors. See Soxhlet SOP Method 3540.

6.0 REAGENTS & STANDARDS

6.1 Reagent Water: deionized water
6.2 Methylene Chloride: pesticide quality
6.3 Hexane: pesticide quality
6.4 Isooctane (2,2,4-Trimethyl Pentane): pesticide quality
6.5 Acetone: pesticide quality
6.6 Sodium Sulfate: ACS grade, granular, anhydrous, baked at 400°C for 4 hours
6.7 Glass Wool: pesticide quality
6.8 Nitrogen: Ultra High Purity
6.9 Diethyl Ether: Preserved with 2% ethanol.
6.10 Florisil: Pesticide grade.
6.12 Sulfuric Acid (concentrated): ACS grade
6.13 TetrabutylAmmonium hydrogen sulfite: sulfite reagent-TBA
6.14 Helium: Ultra High Purity
6.15 Sodium Thiosulfate: ACS grade, granular
6.16 Stock Standards: Purchased as certified solutions from Ultra Scientific("or equivalent"), 100 ug/mL in either methanol or hexane.

Aroclor - 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268

6.17 Working Stock, 1 ug/mL (1000 ppm): add 500 uL of PCB 1260 and PCB 1016 stock solution [Ultra Scientific ("or equivalent")100ug/mL] and 250 uL of Pesticide Surrogate Mix [Restek ("or equivalent") 200 ug/mL] to final volume of 50 mLs in isooctane. Prepared fresh for each new curve; expiration = 6 months. Expiration date to be written on vial.

Dilution	Working Stock (mLs)	Isooctane (mLs)	Final Conc. (ug/L)
1: 4	10	30	250
1: 5	8	32	200
1: 10	4	36	100
1: 20	2	38	50
1: 50	0.8 (=800ul)	39.2	20
1:100	0.4 (=400 µL)	39.6	10
1:200	0.2 (=200 µL)	39.8	5*

*In surrogate curve only

6.18 Spiking Stock, 10ug/mL: Add 0.5mL (500ul) of 100 ug/mL stock (of the appropriate Aroclor) to 4.5 mL (4500ul) of acetone. Spiking standards are obtained from a different source than calibration standards.

6.19 Surrogate Standard, 20 ug/L: Add 1.0mL of Restek or equivalent stock (200 ug/ml in methanol) to 9 mL of Acetone (pesticide grade).

6.20 Custom OP Pesticide Spike Mix: purchased from NSI Solutions or equivalent, custom number; Q-5120

6.21 Stock Standards: Purchased as certified solutions-Organochlorine pesticide mix AB#2 from Restek or equivalent, 8-80 ug/ml in 1:1 hexane:toluene.

6.22 Working Stock: add 125uL of Organochlorine Pesticide Mix (Restek#561687), add 25uL of Pesticide Surrogate mix at 200 ug/mL to 10 mLs of isooctane.

6.23 Pesticide Calibration Standards: Eight standard concentrations are used to create the calibration curve. See below:

Standard Concentration (ug/L)

	0.2	1	5	10	20	50	100	200
Tetrachloro-m-xylene			5	10	20	50	100	200
Hexachlorobenzene			5	10	20	50	100	200
Alpha-BHC			5	10	20	50	100	200
Gamma-BHC		1	5	10	20	50	100	200
Beta-BHC			5	10	20	50	100	200
Delta-BHC			5	10	20	50	100	200
Heptachlor			5	10	20	50	100	200
Aldrin		1	5	10	20	50	100	200
Alachlor			5	10	20	50	100	200
Heptachlor Epoxide			5	10	20	50	100	200
Gamma-Chlordane			5	10	20	50	100	200
Alpha-Chlordane			5	10	20	50	100	200
4,4-DDE		1	5	10	20	50	100	200
Endosulfan I			5	10	20	50	100	200
Dieldrin	0.2	1	5	10	20	50	100	200
Endrin			5	10	20	50	100	200
4,4-DDD		1	5	10	20	50	100	200
Endosulfan II			5	10	20	50	100	200
4,4-DDT		1	5	10	20	50	100	200
Endrin Aldehyde			5	10	20	50	100	200
Methoxychlor			5	10	20	50	100	200
Endosulfan Sulfate			5	10	20	50	100	200
Endrin Ketone			5	10	20	50	100	200
Decachlorobiphenyl			5	10	20	50	100	200

Pesticide Working Stock (1000ug/L): Add 625uL of Restek Custom OC Pesticides Standard (Cat# 565052) and 50uL of Restek Pesticide Surrogate Mix (Cat# 32457) to a 10mL volumetric flask. Bring to volume with isooctane.

Calibration Curve

Dilution	Working Stock (uL)	Isooctane (uL)	Final Concentration (ug/L)
5000X	2	9998	0.2
1000X	10	9990	1
200X	50	9950	5
100X	100	9900	10
50X	200	9800	20
20X	500	9500	50
10X	1000	9000	100
5X	2000	8000	200

7.0 SAFETY

See Material Safety Data Sheets (MSDSs) and Con-Test Chemical Hygiene Plan.

PCBs are absorbed through the skin. Exercise extreme caution when working with concentrated solutions or samples containing or suspected to contain PCBs. Nitrile gloves of sufficient thickness to be impervious to PCBs should be used.

If skin contamination occurs the liquid should be wiped off immediately and the skin washed with soap and water. Water alone is not sufficient. Contaminated clothing should be removed quickly and disposed of as recommended. Organic solvents should NOT be used to wash the skin.

8.0 SAMPLE MEDIA CLEANING PROCEDURES

PUFs are cleaned by Soxhlet extraction.

8.1 Soxhlet Cleaning procedure

8.1.1 PUFs are cleaned by Soxhlet for approximately 24 hours.

8.1.2 Use Acetone, and a clean 500mL Soxhlet stem and bulb for cleaning and flushing PUFs.

8.1.3 For cleaning PUFs, wrap PUF media in kim wipes and place them in the Soxhlet stem.
Low Volume PUF's- Wrap 10 PUFs in several kim wipes and place them in, you can fit 20-40 PUFs depending on how tight you roll the kim wipes.
High Volume PUFs- Wrap 1 PUF in several kim wipes and place them in soxhlet, you can fit 5-10 PUF's depending on how tight you roll the kim wipe.

PUF cartridges in stainless steel pan and dry in hood until no solvent odor is detected. PUFS are cleaned first with two rinses of 1:1 heaxane:acetone and then finally 1 cycle with acetone only.

8.1.4 Method EPA TO-10A:

One PUF cartridge is tested (extracted) by the Soxhlet from each batch and is certified before field use. Lot check is uploaded into Element as a PDF file and standard number/element number is allocated for the batch. If any media from the lot is not used within a month from the lot check, it must be re-certified.

8.1.5 Method EPA TO-4A:

At least one PUF cartridge assembly and one filter from each batch, or 10 percent of the batch, whichever is greater, should be tested and certified clean before the batch is considered for field use. If any media from the lot is not used within a month from the lot check, it must be re-certified.

Note: Also refer to Soxhlet Cleaning Procedure in SOP Soxhlet-3540C

8.2 In order for sample media to be used for sampling, the batch certified blanks must contain <10 ng/component in the plug or filter/plug combination for single component parameters and <100 ng/multi-component parameters.

8.3 Batch media certification for PUF and Filter/PUF combinations is good for 30 days from the time of clean-up. Expiration dates are marked on the bin for each lot. Certified clean cartridges do not need to be chilled when shipping to the field.

8.4 Place the clean PUF into a pre-cleaned (Hexane rinsed) glass sampling cartridge using white gloves and pre-cleaned forceps. Wrap the cartridge with aluminum foil and place in aluminum containers fitted with TFE fluorocarbon-lined caps. The foil wrapping may also be marked for identification.

9.0 PROCEDURE – EXTRACTION FOR TO-10A and TO-4A

9.1 Samples upon receipt are logged into the laboratory LIMS system by the sample management department. After, the sorbent cartridges (PUF) are labeled, they are stored in our walk-in refrigeration unit at less than 4 °C $\pm$ 2°C until extracted. All samples should be extracted within 7 days after collection.

9.2 Samples are extracted using Method 3540 – Soxhlet Extraction. See Method 3540 SOP for set-up procedures.

9.3 All glassware should be washed with a suitable detergent; rinsed with deionized water, acetone, and hexane. See Glassware Cleaning SOP.

9.4 The Extraction Solution (5% diethyl ether/hexane): Solution is prepared by mixing 1900 mL of hexane and 100 mL of diethyl ether.

9.5 Using precleaned cotton gloves, the glass PUF cartridges are removed from the sealed container, the PUF removed from the glass container and is placed into the pre-labeled

Soxhlet extractor using prerinsed forceps. Follow Method 3540 SOP for set-up and connection.

9.6 Prior to extraction, For EPA TO-10A add 200uL and for EPA TO-4A add 500uL of TCMX (Tetrachloro-m-xylene) and DCB (Decachlorobiphenyl) Surrogate standard at 2000ppb to each sample, blank, and laboratory fortified blank. The recovery of the laboratory surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc.

9.6.1 Surrogate Standard – For EPA TO-10A add 200uL at 2000ppb (TCMX/DCB), resulting in a final concentration of 200ug/L. (2.0 mL Extract Final Volume)

9.6.2 EPA TO-4A add 500uL at 2000ppb (TCMX/DCB), resulting in a final concentration of 200ug/L. (5.0 mL Extract Final Volume)

9.7 Prior to extraction, EPA TO-10A add 200uL and for EPA TO-4A add 500uL of 1016/1260 Matrix Spike Standard at 500ppb to each laboratory fortified blank and duplicate. The recovery of the laboratory surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc.

9.7a.1 EPA TO-10A - Matrix Spike Standard – 200uL at 500ppb (1016/1260), resulting in a final concentration of 50ug/L. (2.0 mL Extract Final Volume)

9.7b.1 EPA TO-4A - Matrix Spike Standard – 500uL at 500ppb (1016/1260), resulting in a final concentration of 50ug/L. (5.0 mL Extract Final Volume)

9.8 The water flow to the condenser towers of the Soxhlet extraction assembly should be checked and the heating unit turned on. As the samples boil, the Soxhlet extractors should be inspected to ensure that they are filling and siphoning properly (4 to 6 cycles/hour). Samples should cycle for a minimum of 18± 2 hours. At the end of the extraction process, the heating elements are turned off and the samples are cooled to room temperature.

9.9 Extract Concentration

9.9.1 Remove round bottom flask from Soxhlet Extraction Assembly and pour extract through pre-rinsed sodium sulfate into Buchi Concentration Tube.

9.10 Buchi Syncore Concentrator

9.10.1 First the vacuum pump, Syncore platform, and chiller must be turned on. To do so flip the green toggle buttons to the “on” position. Next push the green start button next to the screen on the vacuum pump to turn the chiller on.

Note: The Syncore platform should be set to 55°C.

Note: The chiller’s on/off status is indicated by the snowflake symbol on the vacuum pump screen. When the snowflake is back-lit with a black square, the chiller is on.

9.10.2 Check each individual cell on the Syncore platform so that it reflects the correct concentration glassware being used. If 1.0 ml concentration tubes are used then each cell should have a small orange O-ring at its lowest point. When using 3.0 ml concentration tubes remove the orange O-ring.

Note: Check each individual cell to see if DI water needs to be added to each cell so that the bottom black ring is submerged.

9.10.3 When placing live samples in the Syncore Concentrator, do so one at a time. Remove the sample ID sticker from the glassware before placing the sample in the Syncore Platform. Place the sticker on the sample position grid in order to indicate which sample is in which cell on the Syncore Concentrator.

9.10.4 All samples should have relatively the same volume. Add the appropriate solvent to samples when necessary in order to maintain uniform sample volume.

Note: Maximum sample volume should be below the top heating plate when the Syncore concentrator is running at full speed.

Note: Concentration time will vary based on the program selected and the type of solvent used.

Note: In order to select a desired program select "menu" on the screen attached to the vacuum pump. Scroll down to "programs" and hit the right arrow button. Continue to scroll down to "open" and use the black knob to select the desired program then hit OK.

Finally, check that the word "gradient" is present in the top left box of the vacuum pump screen. If it is not there go to "menu", "mode", then use the black knob to select "gradient and hit OK.

9.10.5 When starting the Syncore Concentrator place the vacuum plate on top of the concentration tubes and secure firmly using the hand screws. Double check that the correct program is selected for the samples being concentrated. Turn the large black RPM control knob on the Syncore platform to 250. Bring samples to the appropriate final volumes, after sample program on Syncore concentrator has finished.

9.10.6 After samples have been concentrated, empty the solvent collection vessels attached to the condenser. Do so as needed and at the end of each shift.

9.10.7 Samples are quantitatively transferred (with concentrator tube rinsing) to pre-labeled vials and brought up to a final volume of 2.0 mL, in hexane. Concentrated extracts are stored at 4 C $\pm$ 2°C until analyzed.

9.11 An acid clean-up is performed on all PCB samples (Method 3665a) and their corresponding method blanks. Never do an acid clean-up on a sample that is to be analyzed for pesticides. Pipet approximately 2 mLs of sample extract into a 4-mL screw cap vial that contains about 1 mL of sulfuric acid (transfer the remaining sample into a labeled 4-mL vial and place in the freezer). Cap the vial and shake for approximately 1 minute. Allow the layer to separate then pipet off about 1- mL of sample and transfer to a labeled crimp top vial.

9.12 Sulfur is removed by Method 3660B; Tetrabutylammonium (TBA) sulfite reagent. Prepare reagent by dissolving 3.39g Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. To remove impurities, extract this solution three times with 20

mL portions of hexane. Discard the hexane extracts, and add 25g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a PTFE-lined screw cap. This solution can be stored at room temperature for at least one month.

9.12.1 Transfer 1.0mL of sample extract to a 10mL clear vial with a PTFE-lined screw cap.

9.12.2 Add 1.0mL TBA sulfite reagent and 2mL 2-propanol, cap vial, and shake for 1 min. If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present.

9.12.3 Add 5mL organic free reagent water and shake for a least 1 min. Allow sample to stand for 5-10 min. Transfer the hexane layer (top) to a 1mL auto sampler vial.

9.13 A florisil sep-pack clean-up may also be needed. (See SOP Method 3620C)

9.13.1 Rinse the 5-mL syringe with hexane and then rinse the sep-pack cartridges by allowing hexane to drip through.

9.13.2 Add 5 mLs of the extract into the syringe and attach a rinsed cartridge.

9.13.3 Let 1.5 mLs drip through as a further rinse and then allow the next 3 mLs to drip into a labeled 4-mL vial. Discard the final 0.5 mLs.

9.13.4 Rinse the syringe again and repeat the procedure for any further samples.

9.14 With any clean-up procedure it is also necessary to perform the clean-up on the method blank and corresponding QC. Other clean-up procedures may be used.

10.0 PROCEDURE - INSTRUMENT ANALYSIS

10.1 Instrument Set-Up HP 7890 (ECD #5, #9, and #10)

Oven

Initial Temp: 125°C Maximum Temp: 330°C
Initial Time: 0.25 min Equilibration time: 0.50min
Ramps:

#	Rate	Final temp	Final time
1	30.00	200	0.00
2	20.00	260	0.00
3	45.00	320	2.00
4	0.0 (off)		

Post temp: 0°C
Post time: 0.00min
Run time: 9.08min

Front Inlet

Initial Temp: 210°C (On)
Pressure: 13.89 psi (On)
Purge flow: 12.5 mL/min
Purge time: 1.00 min
Total Flow: 25.0 mL/min
Gas type: Hydrogen

Column 1
Model Number: RTX-CLP PESTICIDES: 11139
340°C 30m x 0.32mmID x 0.5um df

Column 2
Model Number: RTX-CLP PESTICIDES II: 11324
340°C 30m x 0.32mmID x .25um df

Front Detector	**Back Detector**
Temperature: 320°C	Temperature: 320°C
Combined Flow: 60.0 mL/min	Combined Flow: 60.0 mL/min
Make-up Gas Type: Nitrogen	Make-up Gas Type: Nitrogen

10.2 Instrument Set-Up **HP 7890 (ECD#2 and #6)**

Column information: KIT-11191 Restek: 11139
Restek RTX-CLP , 30m x 0.32mmID x 0.5um
Restek RTX-CLP II #11324, 30m x 0.32mmID x 0.25um
STX 5 meter guard column #10027

Control Information:

Sample Inlet	:	GC
Injection Source	:	Manual
Oven		
Oven		On
Equilibration Time		0.1 min
Oven Program		

60 degrees C for 2.0 min
then 50°C/min to 200 degrees C for 0 min
then 20 °C/min to 260 degrees C for 0 min
then 45 °C/min to 320 degrees C for 5 min

Post Run Temperature 0 degrees C

Front Injector

Front Inlet SS		
Heater	On	30 °C
Pressure	On	7.2693 psi
Total Flow	On	53 mL/min
Septum Purge Flow	Off	
Mode	Pulsed Splitless	
Gas Saver	Off	
Purge flow to Split Vent	50 mL/min at 2 min	

10.3 Sample Analysis

Analyze samples by GC along with solvent blanks, calibration standards, method blanks (extracted blanks) and all appropriate quality control samples.

Samples are generally analyzed by an auto sampler. When an auto sampler stalls, lab analysts will restart the analysis using two blanks and an acceptable continuing calibration verification standard.

All Aroclor standards must be run for pattern recognition before running samples. Patterns are established before any sample analysis at the beginning of a sequence.

Aroclors are identified based on retention time and PCB pattern matching. All samples must be analyzed on a confirmation column. Retention times must fall within established windows on both columns.

11.0 CALCULATIONS

$$\text{Sample concentration, ug/m}^3 = \frac{\text{ug/L (from instrument) x (final prep volume, L)}}{\text{m}^3}$$

Flow = L/min
Volume (L) = flow x total minutes
m^3 = L/1000

The air volume is corrected to EPA standard temperature (25°C) and standard pressure (760 mm Hg) as follows:

$$V_s = V_a \left(\frac{P_b - P_w}{760\text{mmHg}}\right) \left(\frac{298K}{t_A}\right)$$

where: V_s = volume of air at standard conditions (25°C and 760 mm Hg), std. m^3.
V_a = total volume of air sampled, m^3.
P_b = average ambient barometric pressure, mm Hg.
P_w = vapor pressure of water at calibration temperature, mm Hg.
t_A = average ambient temperature, °C+273.

12.0 QUALITY CONTROL

12.1 Definitions

For definitions and explanations of quality control measures, refer to the Con-Test Analytical Quality Control Manual.

12.2 Quality Control Measures & Acceptance Criteria

12.2.1 Calibration Curve

PCB's

A 5-point calibration curve is used to calibrate the system for all Aroclors. The calibration factor variation over the working range must be ≤20% RSD or ("r" should be ≥0.995). Each curve must be verified with an independent standard (ICV) prior to sample analysis, (50ug/L). ICV must be within 15% of true value.

Each aroclor should be quantiated using a minimum of 5 peaks per isomer when 5 distinct peaks are available.

If a peak is not properly integrated by the data system, manual integration may be necessary. Manual integrations must comply with the Con-Test SOP on Chromatographic Integration Procedures. The integration of the peaks for the samples and quality control samples must be as consistent as possible with the integration used with the initial calibration. All manual integrations must be documented. In particular, the chromatogram must be expanded to clearly show the specific peak being integrated. A "before" integration is printed, showing the software integration, or lack thereof. An "after" integration showing the action taken by the analyst is printed.

The analyst initials and dates both records and they are included with the rest of the documentation for that data file.

The method file used by the data system for the initial calibration should be named with a unique identifier for initial calibration. The initial calibration method file includes a "C" as a suffix to denote that it is the original file. When the retention times change for target compounds due to daily instrument maintenance, the original file is copied to (saved as) a new file with the "C" suffix removed. The method file without the "C" suffix indicates that the retention times have been changed from the initial calibration. The original initial calibration method file with the "C" as a suffix must not be changed.

If the initial calibration does not meet the acceptability criteria, it may not be used for quantitative analyses. Investigate the cause of the problem and perform instrument maintenance if necessary. Then repeat the initial calibration.

Pesticide's

A 5-point calibration curve is used to calibrate the system for all Pesticides. The calibration factor variation over the working range must be ≤ 20% RSD or ("r" should be ≥ 0.995).
The Pesticide curve must be verified with an independent standard (ICV) prior to sample analysis. ICV must be within 15% of true value.

12.2.2 Retention Time Windows

Retention time windows will be determined by calculating the mean and standard deviation of the absolute retention times of the surrogate compounds and three (minimum) to five major peaks for each Aroclor.

For the Aroclors, each selected peak will be characteristic of that Aroclor, will be at least 25% of the height of the largest Aroclor peak, and will include at least one peak that is unique to that Aroclor. The retention times used for these calculations should be taken from three injections of the Aroclor standards made over a 72-hour period.

If the standard deviation of the retention times for any peak is 0.000, a default standard deviation of 0.01 minutes may be used. The width of the retention time window for each surrogate and Aroclor peak will be ±3 times the standard deviation of the mean absolute retention time established during the 72-hour period.

12.2.3 Continuing Calibration Check (Calibration Check Standard)

CCV's are analyzed at the beginning of each day and after every ten samples by the analysis of the midpoint standard; an RPD of 15% or less is acceptable for continuing use of the initial calibration curve.

The calibration check standard must meet the acceptance criteria before any samples are run. If the calibration check does not meet the acceptance criteria, corrective action is required including instrument maintenance. If the calibration check still fails the acceptance criteria, a new initial calibration is required.

12.2.4 Method Blank

Analyzed on each working day to demonstrate that no contamination is present. The method blank is matrix specific, and extracted with every batch or every 20 samples (whichever is more frequent). The target compounds and ranges must be ≤10ng for each single component analyte and <100ng for each multi-component analyte. (Analyzed from batch and not shipped to the field, to serve as a process blank.)

12.2.5 Laboratory Control Sample (LCS) / Quality Control Check Sample/ Laboratory Fortified Blank (LFB)

A matrix-specific LCS is analyzed every 10 samples. Prepared from a different stock than that of the calibration curve. The concentration should be between the low and mid-level standard, and must contain at least Aroclors 1016 and 1260. Percent recoveries must fall within control limits established from historical data.

12.2.6 Matrix Spikes/Matrix Spike Duplicates

The TO-4A/TO-10A matrix does not lend itself to the performance of matrix spike/matrix spike duplicates.

12.2.7 Surrogates

Surrogates are added to all blanks, standards, samples, and spikes. Analyze a minimum of two, one that elutes at the beginning of the run and one that elutes at the end of the run (TCMX and DCB). Percent recoveries must be 60-120%R in order to meet Method TO-4A/TO-10A criteria.

12.2.9 Confirmation

Confirm any hits on a second dissimilar column; report the higher of the two results, unless obvious interference is present on one of the columns, or there were QC outliers on one of the columns. RPD between primary and confirmatory column should be $\leq$40%. All QA/QC parameters (e.g. calibrations, LCSs, etc) must be met on the 2° column as well. For samples the higher of the two results is reported unless requested by client or obvious interference is present from one of the columns.

12.2.10 Solvent Blank –

Analyzed on each working day to demonstrate that no contamination is present in solvents used for extraction. One solvent process blank (all steps conducted but no filter/puff or puff included) is carried through the procedure and analyzed.

12.2.11 Field Blank-

During each sampling event, Client requested, one filter/puff or puff cartridge should be shipped to the field and returned, without drawing air through the sampler, serving as a field blank.

12.2.12 Field Spike –

During each sampling event, Client requested, one filter/puff or puff cartridge is spiked with a known amount of the standard of interest. The spike cartridge will remain in a sealed container and will not be used during the sampling event. The spike cartridge is extracted and analyzed with the other samples. This filed spike acts as a quality assurance check to determine matrix recoveries and to indicate sample degradation.

12.2.13 DDT and Endrin Breakdown

$$\% \text{ Breakdown of DDT} = \frac{\text{Sum of degradation peak areas (DDD + DDE}}{\text{Sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

$$\% \text{ Breakdown of Endrin} = \frac{\text{Sum of degradation of peak areas (aldehyde + ketone)}}{\text{Sum of all peak areas (Endrin + aldehyde + ketone)}} \times 100$$

Check for degradation problems by injecting a standard containing only 4,4-DDT and Endrin; look for the degradation products of 4,4-DDT (4,4-DDE, and 4,4-DDD) and Endrin (Endrin Ketone, and Endrin Aldehyde). See calculation above. Criteria = $\leq$ 15%.

12.2.14 Reporting Limit Verifications

12.2.14.1 Daily Reporting Limit Verification

A spiked standard at the reporting limit, which is analyzed daily at the beginning of the sequence. Percent recovery for the reporting limit verification is 50-150%.

12.2.14.2 Annual Reporting Limit Verification

PUF media that is spiked at the reporting limit and analyzed annually. Percent recovery for the reporting limit verification is 50-150%.

13.0 DATA PROCESSING

13.1 GC files

A file naming convention is used that allows differentiation between updates to the calibration file performed to adjust target retention times for the changes brought about by daily chromatographic maintenance (no file suffix) and updates due to analysis of a new calibration curve (addition of a “C” suffix to the file identifier)

13.2 Reporting Package

The reporting package that is delivered to clients will consist of the sample results, the surrogate recovery results and any matrix spikes, blanks , duplicates and lab fortified blanks that pertain to the clients samples, plus a case narrative.

13.3 Data Filing

13.3.1 Data to be filed in File Boxes

13.3.1.1 All raw data (chromatograms)

13.3.2 Data to be filed in Data Books

13.3.2.1 All types of spikes

13.3.3 Backup data filed on disc

14.0 CORRECTIVE ACTIONS/CONTINGENCIES OF HANDLING OUT-OF-CONTROL DATA

14.1 Refer to Con-Test Quality Assurance Manual.
14.2 Refer to Con-Test Corrective Actions SOP

15.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operation. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address waste generation. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards.

16.0 WASTE MANAGEMENT

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing the waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations.

Any sample containing PCBs over 2.0ppm are labeled and stored separately for disposal. Used standards are accumulated as a lab-pack and sent out to be disposed of properly by a waste management company.

17.0 REFERENCES

17.1 EPA, Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, SW-846, Rev.0, December 1996, Method 8000, 8082, and 3545.

17.2 Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD), second edition, January, 1999.

17.3 Compendium Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD), second edition, January, 1999.

17.4 Con-Test Analytical Chemical Hygiene Plan.

17.5 Con-Test Analytical Quality Assurance Manual.

17.6 HP 5890 Series II Gas Chromatograph Operating Manual, Edition 5, October 1991.

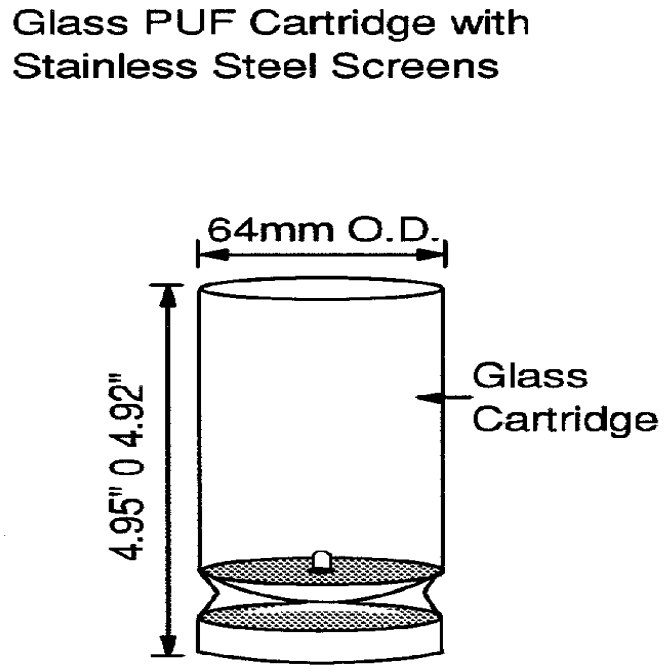
17.7 HP 6890 Series Gas Chromatograph Operating Manual, Edition 1, January 1990.

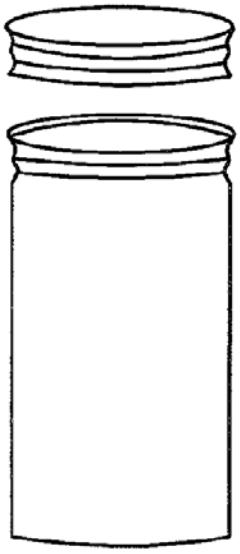
17.8 Syncore Application Guide for Buchi Turbo Vap Concentrator, Version A, January 2012.

17.9 Con-Test SOP on Procedures for Implementing Corrective Actions

17.10 Con-Test SOP on Chromatographic Integration Procedure

Figure 1. Glass PUFF Cartridge (TO-4), and Shipping Container





Aluminum Canister for Shipping
and Storage of the PUF Sampler

Figure 2. TO-10 Diagram

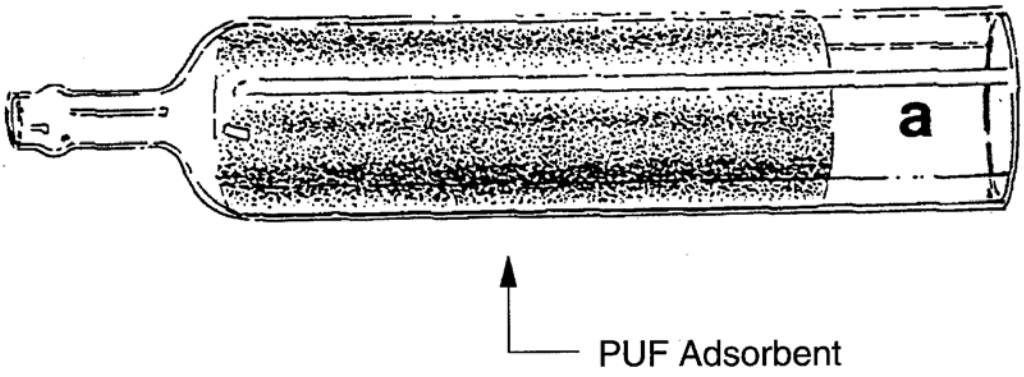


Figure 3. Method Specific Sampling Instructions

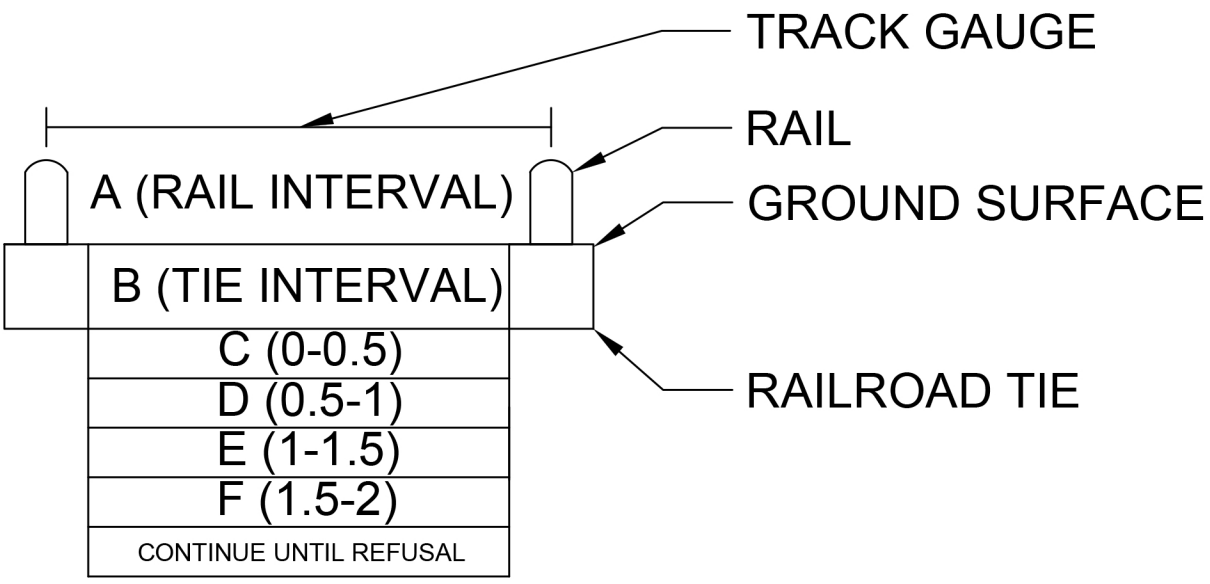
	TO-4A	TO-10A
MEDIA	PUFF CARTRIDGE WITH FILTER (QUARTZ FIBER)	PUFF CARTRIDGE (LOW VOLUME)
SAMPLING RATE AND INTERVAL	0.225 M3/MIN FOR UP TO 24HRS	1 TO 5 L/MIN FOR 4 TO 24 HOURS
SAMPLING VOLUME	UP TO 300 M3. DETERMINED BY PROJECT REPORTING LIMITS	DETERMINED BY PROJECT REPORTING LIMITS
SAMPLE HANDLING	HANDLE WITH ALUMINUM FOIL AND KEEP CHILLED AT 4C	HANDLE WITH ALUMINUM FOIL AND KEEP CHILLED AT 4C
MEDIA HOLD TIME / EXTRACTION HOLD TIME	30 DAYS FROM DATE OF MEDIA CERTIFICATION	30 DAYS FROM DATE OF MEDIA CERTIFICATION
EXTRACTION HOLD TIME	7 DAYS FROM SAMPLING TO EXTRACTION AT 4C	7 DAYS FROM SAMPLING TO EXTRACTION AT 4C

Appendix B

Sampling Interval Diagram

Appendix B

Sampling Interval Diagram
Penn Station, New York



Appendix C

Laboratory Quality Assurance Manuals

Quality Systems Manual

Alpha Analytical, Inc.

D/B/A

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1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our customers with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and electronic data deliverables must be available to the customer.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional contamination to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honesty and integrity with which each chemist, courier, login staff member, or office staff member performs their tasks. The customer or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical will vigorously pursue its mission into the next millennium.

Mark Woelfel

President

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3 Introduction

The Quality Systems Manual, referred to as Corporate Quality Systems Manual (CQSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, customers and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet customer needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as an uncontrolled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly to the QSM and SOPs.

All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) 2009 TNI standards, applicable EPA requirements, and applicable Department of Defense (DOD) Quality Systems Manual, standards.

Twenty-five (25) sections comprise the QSM. Related quality documentation including the listing of SOPs, forms, floor plan, equipment, personnel and laboratory qualifications are available. The QSM sections provide overview descriptions of objectives, policies, services and operations.

3.1 Scope

The QSM describes the requirements of the Laboratory to demonstrate competency in the operations for performing environmental tests for inorganic, organic, air and microbiological testing. The basis for the environmental tests is the methods found in documents published by the United States Environmental Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, DOD-QSM, and other procedures and techniques supplied by customers.

The QSM includes requirements and information for assessing competence and determining compliance by the laboratory to the quality system. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan the laboratory demonstrates achievement of the customer specified requirements through its documented processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and customers use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) TNI 2009 standards, applicable EPA requirements, regulations, guidance, and technical

standards and current DOD QSM standards. This Quality Systems Manual, laboratory Standard Operating Procedures (SOPs), and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by NELAC and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operations Director, Laboratory Technical Manager and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our customer's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accordance with NELAC and other regulatory programs, from a National Institute of Standards and Technology (NIST)-approved PT provider for the analytes established by EPA for water samples, and for other analytes and matrices. The specific analytes and matrices analyzed are based on the current scope of the laboratory services as documented in the laboratory SOPs and state certifications.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstrated according to the procedure described in SOP/ 1739 prior to use.

Alpha Analytical has developed a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program include: internal proficiency testing, electronic data audits and post-analysis data review by the QA Officer; a program to improve employee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequences. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form upon commencement of employment and each year following. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory Technical Manager to inform customers and suppliers of this policy when necessary.

> In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory Technical Manager to take appropriate action to prevent recurrence.

3.3 References

External reference documents are available electronically in the Qualtrax system for staff to access the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the electronic files in the Qualtrax system. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

3.4 Definitions

Appendix A lists the definitions as adopted by the laboratory. The definitions are from the 2009 TNI and DoD QSM standards.

4 Organization and Management

4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

Consulting firms,
Engineering firms,
Waste Management Companies,
Industrial sites,
Municipal agencies
Department of Defense projects.

4.2 Organization

The laboratory operates a quality system approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting customer specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise the integrity of the reporting of the final data.

The President is responsible for directing all areas of the company. The following job functions report to the President:

Operations Manager
Quality Assurance Officer
Customer Services Manager
Marketing / Business Development / Sales
Financial Services
Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

- Laboratory Technical Manager(s)
- Department Managers

The Laboratory Technical Manager(s) is(are) responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

The organics managers direct personnel in the organics extraction and instrumental laboratories.

The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.

The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff and reports directly to the President and has defined responsibility and authority for ensuring that the quality system is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Quality Systems Manual and reporting to the Laboratory Technical Manager and Senior Management the status of the quality program. The QAO oversees the Quality Systems Specialists and is responsible for oversight and/or review of quality control data and function independently from laboratory operations.

The Customer Services Manager is responsible for customer interactions, project coordination and laboratory personnel notification of project requirements.

The Marketing, Business Development and Sales personnel are responsible for increasing the volume of work from current customers and adding new customers to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Technical Manager, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The Controller is responsible for maintaining and reporting on the financial status of the company. The Controller directs financial personnel on proper accounting procedures and maintaining the list of approved suppliers and subcontractors. The Controller reports directly to the President.

The Human Resource Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak workloads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Technical Manager is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of NELAC and DoD QSM Accreditation, the Lead Laboratory Technical Manager is the Laboratory Technical Manager. The deputies for the Lead Technical Manager are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory Technical Manager meets the requirements specified in the Section 4.1.7.2 Volume 1, Module 2 of the 2009 TNI standards. If the Laboratory Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff member meeting the qualifications of Laboratory Technical Manager will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Manager's absence exceeds 35 consecutive calendar days.

4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of sample receipt. Standard turnaround for all other customers is ten business days from time of sample receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any special arrangements for rush or expedited turnaround time. The basis for data quality depends on customer, regulation and method performance criteria. Accuracy, precision, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for protecting customer confidentiality and proprietary rights. The review occurs during orientation and ethics training. It is the policy of the laboratory to release data to the customer authorized contact. Personnel assigned the duties of interacting with customers review project files and discuss data related only to the project. Personnel whose duties do not include routine customer contact must check with the customer service manager before discussing data with regulators or third parties

5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in order to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its customers with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and caliber in the field of analytical chemistry. This and state-of-the-art instrumentation and automation combine to insure data of known and documented quality.

5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distribution of the Quality Systems Manual. Management reviews and authorizes the manual. Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization of the Quality Systems Manual is documented electronically in Qualtrax. Updates of this manual occur at any time throughout the year. Document control procedures (SOP1729) apply to the distribution of the Quality Systems Manual. Controlled copies of the manual are maintained electronically within Qualtrax. Persons or organizations outside of Alpha

Analytical may receive uncontrolled copies. Copies are distinctly indicated "Uncontrolled Documents" within the footer of each page.

5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are conducted by qualified QA Specialists and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

- Verification that adequate written instructions are available for use;
- Analytical practices performed in the laboratory are consistent with SOPs;
- The quality control practices are applied during production;
- Corrective actions are applied as necessary;
- Deviations from approved protocols are occurring only with proper authorization and documentation;
- Reported data is correct and acceptable for reporting;
- SOPs, quality records, analytical records, electronic data files are maintained properly; and
- Personnel training files and records are satisfactory and current.

Before a scheduled internal audit, the assigned auditor reviews checklists, if used, and/or the SOP specific to the area. The checklist may be from an external source or prepared by the auditor. . After the audit, the auditor submits a summary or notes from the audit to the Laboratory Technical Manager or QAO as part of the audit report. The summary identifies discrepancies found during the audit. Technical personnel are responsible for the inspection and monitoring of in-process and final data. Personnel independent of those having direct responsibility for the work performed audit the quality system and processes.

Representatives sent by customers and government or accrediting agencies often perform external audits. These audits are most often announced inspections, but sometimes are not announced. The Quality Assurance Officer, Laboratory Technical Manager or assigned deputy, and/or appropriate Department Manager accompany the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the

nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Technical Manager on any outstanding audit findings.

5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by customers and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past performance of an external performance audit.

5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP 1736. The corrective action program at Alpha Analytical uses the Nonconformance workflow in Qualtrax to document and follow through the corrective action/preventative action process for three main areas: nonconformance's within the laboratory, customer complaints and failed PT studies. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Nonconformance reports are tracked for closure date and the type. Reports to management include the listing of open nonconformance reports and the frequency of the type of nonconformance occurring. A QA designee monitors the completeness of the forms, as well as verifies the actions are complete and acceptable.

Customers will be notified within 5 days of any question(s) regarding validity of results.

5.7 Managerial Review

The management review occurs at least once per year as part of the strategic planning process. Documentation of the management review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, closure status, audit progress and other quality assurance actions. Meetings include discussion and progress on quality system initiatives since the last meeting.

Prior to the meeting, an agenda is distributed to all personnel expected to be in attendance. The meeting is chaired by the President. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method.

Alpha Analytical uses quality control samples to evaluate the following:

1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
2. Adequate tests to define the variability and/or reproducibility of laboratory results;
3. Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
4. Measures to evaluate test performance, such as detection limits and quantitation limits or range of applicability such as linearity;
5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
6. Selection and use of reagents and standards of appropriate quality;
7. Measures to assure the selectivity of the test for its intended purpose;
8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance limits are used to determine the usability of the data. Control charts and/or calculated control limits monitor the long-term method performance by analyte, by instrument for water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional performance measures to ensure data comparability. Control limits are recalculated when trends are observed.

Where no reference method or regulatory criteria exist, the laboratory specifies the acceptance/rejection criteria in the SOP. The test SOP specifies the QC samples performed per batch of samples. The quality control samples are categorized into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Control Sample (LCS)
- Laboratory Control Sample Duplicate (LCSD)
- Matrix Spike (MS)
- Matrix Spike Duplicate (MSD)

Selection of samples for Duplicate, Matrix Spike (MS) & Matrix Spike Duplicate (MSD)

2. Duplicate samples
    a. Samples will be selected if identified and requested by customer
    b. If no samples are identified by the customer then random samples will be analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

3. Matrix Spike (MS) / Matrix Spike Duplicate (MSD) samples
    a. Samples will be selected if identified and requested by customer

b. If no samples are identified by the customer then random samples will be selected and analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

c. If MS/MSD is not required, LCS/LCSD may be substituted for precision and accuracy evaluation.

The frequency is dependent on the reference method and test protocol. The following is the default requirement for quality control checks in lieu of any other guidance. The frequency for each quality control sample is generally one (1) per every 20 samples.

5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. The analyst and reviewer sign and date the hardcopy of all manual integration. The analyst notes the rationale for performing the manual integration on the hardcopy printout and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \%R_{LCS}$$

where:
MV = Measured Value
TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%R_{MS}$$

where:
MV = Measured Value
TV = True Value
SV = Amount found in sample

Average ($\overline{X}$)

$$\sum_{i-1}^{n} X_i \Big/ n = \overline{X}$$

where:
$\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{\left(R_1 + R_2\right)/2} * 100 = \%RPD$$

where: R_1 = Larger of two observed values
R_2 = Smaller of two observed values

% Difference (%D)

$$\frac{X - \overline{X}}{\overline{X}} * 100 = \%D$$

where: $\overline{X}$ = Average of all values
X = Result of measurement

Standard Deviation of the sample (S_x)

$$\sqrt{\frac{\sum\left(X - \overline{X}\right)^2}{n-1}} = S_x$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Standard Deviation (%RSD)

$$\frac{S_x}{\overline{X}} * 100 = \%RSD$$

where: $\overline{X}$ = Average of all values
Sx = Standard Deviation (n - 1)

Range of Logs (for microbiological enumeration analysis)

10% of routine samples are analyzed in duplicate and the range of logs is determined.

MDL (See 40CFR Part 136 for details)

$$\left[\sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2 \Big/ n}{n-1}}\right] * t_{0.99} = MDL$$

where: *MDL* = The method detection limit
X = Result of each measurement
n = Number of values
t(n-1,1 = .99) = The students' T value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Reporting Limit (RL)

Lowest calibration standard or greater

Control Limits

Upper Control Limit: $\overline{X} + 3 * S_x = UCL$
Lower Control Limit: $\overline{X} - 3 * S_x = LCL$

Warning Limits

Upper Warning Limit: $\overline{X} + 2 * S_x = UWL$
Lower Warning Limit: $\overline{X} - 2 * S_x = UWL$

Method of Standard Additions (MSA): (See EPA 7000A for details)

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard analyte solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration Cx is calculated:

$$C_x = \frac{SB\ V_S\ C_s}{(SA - SB)\ V_X}$$

where SA and SB are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_s should be chosen so that SA is roughly twice SB on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

5.10 Document Control

The Document Control Procedure (SOP/1729) describes the process for controlled and uncontrolled documents. The use of the revision number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is present on each page of the document. A master list of documents includes the unique identification. Each controlled copy includes the revision number, published date and page number.

Full document control includes the status of each document: active, inactive or superseded/archived. Inactive documents are procedures not currently requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and any subsequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and approval. The history section of the document in Qualtrax includes a description of the nature of the document change.

Standard Operating Procedures (SOPs) are instructions for repetitive or standard operations performed by the laboratory. The SOP author is the person familiar with the topic. The standard format for writing SOPs is set-up as a template for administration and technical SOPs. Each SOP is peer reviewed, authorized by management, and QA before final publication and implementation. Authorized signatories for controlled documentation include one or more of the following personnel: Company President, Quality Assurance Officer, Laboratory Technical Manager, Department Manager, Department Team Leader. Personnel acknowledge approved documents as read, understood and agreed to through electronic attestation forms associated with each document as SOP Attestation Tests which reside in Qualtrax.

SOPs must receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOP must conform to applicable requirements of analytical methods and certification agencies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or add methods, the scope of the existing method is extended, or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information and a new revision number is assigned and published in Qualtrax.

The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, customer instructions, Laboratory Work Instructions, data sheets, check lists and forms.

5.11 Detection Limits

Detection Limits (DLs), previously referred to as Method Detection Limits (MDLs), are determined for all analytes as specified in the NELAC TNI 2009 standards and DoD QSM standards. DLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, Reporting Limits (RLs), are established. The RL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Laboratory reporting limits lie within the calibration range, at or above the RL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting limits are required below the lower level of the calibration curve, RL, or low-level check standard, method modifications are required. Refer to DL/LOD/LOQ SOP/1732. Note:."J" Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Detection Limit (DL) with a "J" qualifier as long as there is a LOD study on file.

5.12 LOD/LOQ Studies

A. LOD (Limit of Detection) Verification

1. LOD (Limit of Detection) verification is required annually for each target analyte in which test results are to be reported below the lowest calibration standard ("J" values) for each instrument, matrix and prep procedure.
    a. Quarterly LOD Verification is required for DOD projects unless option of analyzing an LOD with a DoD project batch is employed. In this case quarterly verification is not necessary.

2. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

3. The validity of the LOD shall be confirmed by ***qualitative*** identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests, and >1 up to 4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

4. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

B. LOQ (Limit of Quantitation) Verification

1. LOQ (Limit of Quantitation) verification is required annually for each target analyte that is not reported below the lowest calibration standard for each matrix and prep procedure. LOQ is not required if an annual LOD verification is performed. LOQ is required quarterly for all DoD projects unless option of analyzing an LOQ with a DoD project batch is employed. In this case quarterly verification is not necessary.

2. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria for accuracy.

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

The LOQ acceptance criteria are based on the established acceptance criteria for Laboratory Control Samples.

Refer to DL/LOD/LOQ SOP/1732

5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:

   a. Perform duplicate analyses on first 15 positive samples.

   b. Record duplicate analyses as D1 and D2 and calculate the logarithm of each result.

   c. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.

   d. Calculate the range (R) for each pair of transformed duplicates as the mean of these ranges.

6 Personnel

6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, customer specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

The H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During initial training, management provides access to documented operations procedures, observes personnel performance, and evaluates personnel proficiency. Management documents technical laboratory staff's proficiency initially and on a continuing basis through use of laboratory control samples and purchased proficiency evaluation standards.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and attestation process.

Either the Quality Assurance Officer, Operations Director and/or Technical Managers have the authority to stop work due to non-conformances and have the authority to resume work after it has been stopped.

6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Operations Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/1565 details the process for completing requirements and training to ensure personnel have adequate skills and competence for the job function.

A job description details the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of new employees and contract staff includes laboratory ethics and quality policies, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, report data values, that are not actual values obtained or improperly manipulated, or intentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section K of the Employee Handbook. Each employee must report personally or anonymously to the Laboratory Technical Manager, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics policy occurs annually with all personnel. The annual review includes annual renewals of the Ethics Agreement.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix F)
- Initial and annual ethics training
- Internal audits conducted annually
- Adherence to Manual Integration SOP/1731
- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director

- Anonymous reporting to HR Director - This is accomplished by writing a detailed description of the suspected ethics breach and submitting the information, anonymously, to the Human Resource Director.

- "No-fault" policy encouraging reporting of incidences without fear of retribution

- Electronic tracking and audit trails through LIMs and instruments enable where available.

6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as-needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial employment. Orientation training includes laboratory business practices, employment specifications, Ethics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the job function.

Managers ensure the training for new employees and review the continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of higher learning.

Training of new personnel to any job assignment takes place on-site according to the Training Program procedure. Laboratory personnel may perform their assigned methods/protocols without supervision only after documentation of acceptable proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SOPs. Health and Safety training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Chemical Hygiene Plan and Material Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to the laboratory operation. All training is documented via the Training Attestation Form, which is signed by all in attendance that they understood and will implement what was presented to them.

Training is an on-going opportunity to evaluate the laboratory operations. The updating of SOPs, Quality Systems Manual and other related information documents all changes to the quality system. Training is documented via the Training Attestation Form or in Qualtrax with training test records.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee's responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual's training record.

6.4 Records

The QA Department is responsible for maintaining training records. Certificates, demonstration of capability forms and other records of training are placed in the individual's training file.

Appropriate personnel are notified through email and/or Qualtrax or by the QA department when a revision is complete for the controlled version of a document. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet for each of the Westboro and Mansfield Facilities

The laboratory functional areas include:

Administration and offices
Sample receiving
Sample management
Air analysis (Mansfield Facility only)
Microbiological (Westboro Facility only)
General analytical chemistry
Metals sample preparation
Organic sample preparation
Metals analysis
Volatiles gas chromatography (GC)
Volatiles gas chromatography/mass spectrometry (GC/MS)
Volatiles air analysis (Mansfield Facility only)
Semivolatiles gas chromatography/mass spectrometry (GC/MS)
Semivolatiles gas chromatography (GC)
Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropriate cabinets and properly disposed of as required. All flammable solvents are stored in OSHA and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste areas houses the sample and chemical waste before pickup by a licensed waste hauler.

7.1 Environment

Lighting, noise, humidity, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analytical equipment. Air-handling systems minimize airborne contaminants that may jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature and recorded in the data logger system. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/1728 and Mansfield SOP/1797)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and uncluttered work areas. The job descriptions list specific housekeeping duties. Records, samples and waste materials are the common cause for clutter in the laboratory.

. Removal of administration and laboratory records to the record storage area occurs to reduce clutter and ensure traceability. The individual filling the laboratory record box, labels the box with a number, the contents, date and laboratory area. Authorized personnel assign and record into a permanent record the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Boxes are stored onsite and off-site for the record retention period identified in the NELAC and EPA regulations, whichever is more stringent.

Sample management personnel remove samples to the sample storage area after all data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. The archive sample storage area is not controlled or monitored. Based on customer specifications, samples are properly disposed or returned to the customer.

Waste materials, expired reagents, expired standards and materials are disposed of and not stored in the laboratory. Hazardous waste labeled accumulation containers in the laboratory collect designated waste streams for later bulk disposal. Laboratory personnel remove the less than five-gallon accumulation containers when full from the laboratory and place the containers in the bulk hazardous waste area. Refer to the Waste Management and Disposal SOPS for Westboro: SOP/1728 and Mansfield SOP/1797. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, customers, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offices have the emergency contact list for the laboratory.

8 Equipment and Reference Materials

8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for most major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

A brief summary of some common preventive maintenance procedures is provided in Appendix E. All instrument preventative and corrective maintenance is recorded in the maintenance logbook assigned to the equipment. After maintenance or repair, the instrument must successfully calibrate following the method SOP. Laboratory personnel must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and consumables for analytical equipment. Backup instrumentation for some analytical equipment is available on site for use in case of major equipment failure. The person discovering or suspecting an equipment maintenance problem or failure tags the equipment with 'out of service' tag. If routine maintenance measures do not eliminate the problem, the Laboratory Technical Manager or Operations Director is notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and traceable maintenance logbooks in which to document manufacturer's recommended maintenance procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetitive or on-going equipment problems other than normal maintenance requirements on nonconformance action forms. The nonconformance action form notifies management and the Quality Assurance Officer of a problem affecting the performance and data quality.

The laboratory groups some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

Equipment name;

Manufacturer's name, type identification, serial number or other unique identification;

Date received, date put into service, condition when received;

Current location;

Details of past maintenance and future schedule;

A history of any damage, malfunction, modification or repair;

Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing bromthymol blue (BTB) on each day of media preparation.

8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment location, serial number, model number, and purchase date. The unique identification number is attached to the piece of equipment.

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

8.3 Laboratory Water

Laboratory water is purified from central DI water systems and piped to all laboratory areas. In Westboro, the QA Department samples the laboratory grade water and submits the samples for analysis by the lab to document the water meets the drinking water certification criteria. The Laboratory Water Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/1738). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

TABLE 8-1

Parameter	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon (Westboro only)	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia Nitrogen (Westboro only)	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn (Westboro only)	Monthly (Required Annually)	< 0.05 mg/L
Total Metals (Westboro only)	Monthly (Required Annually)	< 0.1 mg/L
Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL

Parameter	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon (Westboro only)	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia Nitrogen (Westboro only)	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn (Westboro only)	Monthly (Required Annually)	< 0.05 mg/L
Total Metals (Westboro only)	Monthly (Required Annually)	< 0.1 mg/L
Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL
Water Quality Test (Biosuitability) (Westboro only)	Annually	0.8 – 3.0 ratio

8.4 Reference Materials

Reference materials include: Class 1 weights, NIST thermometers and reference standards. Logbooks record the reference materials used for calibration and verification. The Department Manager or QA Department maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area records the unique identifier on the reference standard certificate and the Department Manager maintains the certificate. The identifier allows traceability from the certificate to the analytical data.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

9.2 Traceability of Calibration

The program of calibration and/or verification and validation of equipment is such that measurements are traceable to national standards, where available. Calibration certificates indicate the traceability to national standards, provide the results, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. A body that provides traceability to a national standard calibrates reference standards. The laboratory maintains a permanent file of all such certifications.

9.3 Reference Standards and Materials

Alpha Analytical has a program for calibration and verification of reference standards. The results and program are recorded in the appropriate instrument logbook. Required in-service checks between calibrations and verifications are described in method SOPs and are recorded in the appropriate instrument logbook.

Calibration standards are maintained within the area of consumption. A logbook of use is maintained and use is limited strictly to method required calibrations. Each calibration standard is identified as to test method used, date received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are stated in applicable test method SOPs.

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and for no other purpose.

Standards and reagents are uniquely identified as outlined in Westboro SOP 1745 and Mansfield SOP 1816.

9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and reference method specifications. Before the start of each analytical sequence, initial calibration is verified by using a continuing calibration standard. Calibration verification or continuing calibration uses a standard from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, decafluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by analyzing a set of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each solution is analyzed once, unless the method or the customer requires multiple analyses. The relative response factor for each analyte is calculated for internal standard calibration. The calibration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within control limits.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent drift (%d) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percent differences or percent drift for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed until the continuing calibration analysis meets all criteria, or a new calibration is established before any samples are analyzed. No

samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.3 Cold Vapor Atomic Absorption Spectrophotometry (CVAA)

An initial calibration is performed daily with freshly prepared working standards that bracket the expected concentration range of the sample. A minimum of a three-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified every 10 samples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within ± 10% of the true value.

9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value for EPA Method 200.7 and 10% for SW846 6010 methods. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-certified every year. Records of thermometer calibrations are retained by the QA Department. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

NOTE: Electronic-based thermometers are calibrated on an annual basis and quarterly if associated with DoD projects. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to be applied and the expiration of the calibration check. Certificates are kept on file in the QA Department.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is

updated when a change in the thermometer is required due to breakage, damage or expired calibration.

9.5.7 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights, which bracket the weight to be measured. Additional calibration check procedures are performed on balances utilized in Microbiology laboratory. This additional procedure consists of a deflection test, which is performed to ensure that 100mg is detectable at a weight of 150 grams.

The balance logbook lists the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

9.5.8 Mechanical volumetric pipettes

Delivery volumes for the mechanical volumetric pipettes (i.e Eppendorf) are checked and recorded gravimetrically before use and on a quarterly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook. If used for DoD projects, then these pipettes are checked by comparing 10 measurements. For future DoD projects using QSM 5.0, these pipettes will be checked daily using 3 measurements.

Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range for the analysis are used for calibration. A second source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST certified. Standard buffer solutions are not retained or re-used. The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement to NIST.

9.5.11 Conductivity Meters

Three calibration standards of potassium chloride (KCL) solutions are analyzed annually on each instrument range. The calibration standards are used to verify instrument performance. The

acceptance criteria are defined in the test SOP. If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is used to calibrate each range of the instrument. A second standard is used to check the calibration each day the meter is used. The check standard is near the measurement range for the samples to be analyzed. The acceptance criterion is ± 20% of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceable is used to allow traceability. The check standard is performed at the end of the analysis run or at least after every 20 samples.

9.5.12 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60^{o}C for 24 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.

10 Test Methods and Standard Operating Procedures

10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or customer-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the customer.

The reference methods define the instrument operating conditions. In many of the reference methods, a range or general guidance on the operating conditions is defined. Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at the time of the analysis are documented in the appropriate laboratory or sequence log. A revision to the SOP takes place, when a day to day change in the operating condition improves performance for all matrices.

The laboratory SOPs include the operation of measurement equipment. The SOPs contain the - following information, as applicable:

- The equipment used in the procedure, including equipment type
- Equipment calibration and process for obtaining the measurement from the calibration
- The step by step instructions to perform the measurement
- Acceptance criteria for the calibrations
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- The basis used for the calibration standards such as traceability to NIST or EPA or demonstration of comparability
- Frequency at which the equipment will be calibrated, adjusted and checked
- The records maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in electronic form through Qualtrax. Each SOP clearly indicates the published date of the document and the revision number.

10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents in the Alpha Qualtrax system. Directories are available for each laboratory area and administrative area in appropriate subfolders. Each SOP includes or references where applicable:

1) identification of the test method and where applicable;
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application;
5) summary of method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies
10) reagents and standards
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measurements;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the customer and other recipients of the relevant reports.

The customer requests the reference method for sample analysis usually based on the regulatory program. The customer services staff may assist the customer with method selection when the customer specifies the regulatory program, but is unsure of the correct method required. The Laboratory Technical Manager or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on customer-defined method performance criteria. Customer services may recommend a procedure that meets the customer method performance criteria.

10.5 Method Validation/Initial Demonstration of Method Performance

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method.

10.6 Sample Aliquots

The aliquot sampling process from a submitted sample is part of a test method. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Personnel record observations made during aliquot sampling in the test method logbooks.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks. A second person recalculates all manual calculations. An independent qualified analyst also reviews the data. A Customer Services representative reviews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Customer. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Customer within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Customer Services Department of the sample number(s), Customer name, analysis and sample results (preliminary or confirmed). The Customer Services Department notifies the customer.

The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

a) The laboratory retains records of manufacturer’s statement of purity, of the origin, purity and traceability of all chemical and physical standards.
b) Original reagent containers are labeled with the date opened and the expiration date.
c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.

d) Where calibrations do not include the generation of a calibration curve, records show the calibration date and type of calibration standard used.

e) All prepared reagents and standards are uniquely identified and the contents are clearly identified with preparation date, concentration and preparer's initials. These procedures are outlined in Westboro SOP/1745 and Mansfield SOP/1816.

10.9 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

the software systems perform the required functions accurately,

the users understand how to use the system, and

auditors can assure themselves of the validity of the analytical data.

The computer systems used at Alpha Analytical are purchased. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Alpha Analytical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.

2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems.

Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/1562 and Computer Security SOP/1563. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Customers or Alpha's Couriers deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Customer service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are packed to minimize or prevent breakage. The containers are placed in plastic coolers or shipping packages and Chain-of Custody forms, seals (if requested) and labels enclosed. The bottle order is shipped by third party, picked up by the customer or customer representative or delivered by Alpha courier to the customer.

11.1 Sampling Supplies

11.1.1 Sample Containers

Sample containers provided by Alpha Analytical include labels, preservatives and a blank chain of custody form. Preservatives and containers are lot controlled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collection of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checks are performed by Microbiology staff and results recorded in Microbiology Sample Container Sterility Log.

11.1.2 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

11.1.3 Reagent Water

Alpha Analytical supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on customer specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/1738).

11.2 Sample Tracking

Alpha Analytical uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;

3. It is in one's physical possession and then locked up so that no one can tamper with it;

4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Information Management System (LIMS) to uniquely identify and track samples and analytical data throughout the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project if requested)
- Project name or description
- Analyses requested (per matrices received)
- Sample number (unique to this sample)
- Sample descriptions (customer ID, including number of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due
- Customer's name and address
- Notation of special handling instructions
- Additional comments or instruction for the laboratory
- Purchase order number(s), if applicable

Alpha Job Numbers (Process for assigning numbers)

Alpha Job Numbers are unique #'s automatically designated by our LIMS computer system for every individual customer project.

There are 3 parts to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

  For example…. L0904165 ---- Year 2009 and 4,165th job to be logged in this year.

  The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives its own bar-coded label. For example, L0904165-09A is the first container of the 9th sample listed on a customer's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The customer and customer services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediate notify customer services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. The problem is brought to the attention of a customer services manager who reports it to the customer. Plans for disposition of the affected samples or container are agreed upon with the customer, carried out, and recorded in the project records.

11.4 Sample Receipt Protocols

The sample management staff receives all samples. A unique job number is assigned to each shipment of samples received from a customer. The in-house records for the incoming job, including the internal Chain-of -Custody, are initiated with a Sample Delivery Group (SDG) form. The customer, and Alpha courier and/or the sample management personnel sign the sample custody form at the time of receipt at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, SDG form and the sample custody form are completed for external courier delivered samples.

The sample management staff examines the shipping containers, their contents, and accompanying customer documentation. Information about the sample identification, the location, date and time of collection, collector's name, preservation type, sample type, presence and condition of custody seals, the state of preservation of the samples and other required information is noted on the SDG form. Any discrepancies in documentation or problems with sample condition such as appropriate sample containers, thermal preservation variation, holding times and adequate sample volumes are noted and brought to the attention of the customer via the nonconformance action form. The Customer Services Manager provides clarification or further instruction to the sample management staff on the processing of the samples that are incomplete or missing required information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, customer sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and customer directives. Refrigerators are monitored and controlled to be within 4 ± 2°C. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Customer Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or customer specific requirements are recorded on nonconformance action forms and contact made with the customer by the Customer Services Manager or designee.

Sample storage facilities are located within the sample management area or in designated sample storage areas within the analytical departments. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure. Samples are returned to the sample storage area after the sample portion is removed for analysis. Extracts and digestates are tracked and follow the same internal custody operation. Extracts and digestates are removed to the waste disposal area after analysis for proper disposal.

Sample storage precautions are used to ensure that cross contamination does not occur during sample storage. Refrigerator storage blanks are monitored for volatile compounds as necessary. The storage blank information allows the assessment of potential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are recorded continuously in the data logger system. Corrective action is done as necessary when temperatures are not within the control criteria. In both the Westboro and Mansfield facilities, Automated Data loggers are linked to thermocouples in custody refrigerators and freezers in the Sample Storage areas as well as department standards/storage refrigerators and freezers. The Data logger is calibrated and certified by an outside vendor on a quarterly basis. Refrigerators and/or freezers not connected to the Data Logger system have temperatures measured with NIST traceable thermometers. Temperature records indicate the thermometer or sensor (Data logger) used for obtaining the measurement.

11.6 Sample Disposal

Samples are held for 21 calendar days after the report is released to the customer. Upon written customer request samples may be held longer in an uncontrolled area. Requests for controlled sample storage must be arranged at the time of contractual commitment. Air canister samples are held for 3 days after the report is released to the customer.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the customer, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to customers. Samples in storage are retained a minimum of 21 calendar days after reporting the results to the customer. Any samples requiring more than 21 calendar days are archived. Air canister samples requiring storage more than 3 business days require prior approval.

When a customer has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any customer specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the

control of this laboratory. Customers are informed that a commercial carrier will deliver their samples if required**.**

12 Records

Alpha Analytical has a record system that produces accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized customers, agencies or another regulator.

12.1 Record Keeping System and Design

The record keeping system allows reconstruction of laboratory processes that produced the analytical data of the sample.

a) The records include the names of personnel involved in sampling, preparation, calibration or testing.

b) Information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.

c) The record keeping system provides retrieval of working files and archived records for inspection and verification purposes.

d) Documentation entries are signed or initialed by responsible staff.

e) Generated data requiring operator logging on appropriate logsheets or logbooks are recorded directly and legibly in permanent ink

f) Entries in records are not obliterated by any method. Corrections to errors are made by one line marked through the error. The person making the correction signs and dates the correction.

g) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

12.2 Records Management and Storage

1. Records including calibration and test equipment, certificates and reports are safely stored, held secure and in confidence to the customer.
2. The laboratory maintains hardware and software necessary for reconstruction of data.
3. Records that are stored or generated by computers have hard copy or write-protected backup copies.
4. Alpha Analytical has established a record management system, for control of hard copy laboratory notebooks.
5. Access to archived information is carefully controlled and is limited to authorized personnel. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or transferred according to the customer's instructions. A plan will be developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

1. Analysts work sheets and data output records,
2. Reference to the specific method,
3. Calculation steps including definition of symbols to reduce observations to a reportable value,
4. Copies of all final reports
5. Archived SOPs,
6. Correspondence relating to laboratory activities for a specific project,
7. All nonconformance action reports, audits and audit responses,
8. Proficiency test results and raw data,
9. Data review and cross checking.

The basic information to tie together analysis and peripherals such as strip charts, printouts, computer files, analytical notebooks and run logs for Alpha Analytical includes:

1. Unique ID code for each Laboratory sample or QC sample;
2. Date of analysis;
3. Instrument identification and operating conditions;
4. SOP reference and version;
5. Calculations;
6. Analyst or operator's initials/signature.

In addition, Alpha Analytical maintains records of:

1. Personnel qualifications, experience and training
2. Initial and continuing demonstration of proficiency for each analyst
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records.

12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to records pertaining to:

a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes inter-laboratory transfers of samples, extracts and digestates.

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) Automated sample handling systems;

n) Records storage and retention; and

o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.

p) The COC records account for all time periods associated with the samples.

q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure.

r) Common carrier documents.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the customer and in accordance with relevant environmental regulations.

Customers specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Customers specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival.

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report format. The standard report format includes:

1. Title: “Certification of Analysis”
2. Name and address of the laboratory
3. Laboratory Job Number, page number and total number of pages included in the report.
4. Name and address of the customer
5. Alpha sample number, Customer identification, Sample location
6. Samples identified that do not meet the sample acceptance requirements for project.
7. Date of sample receipt, sample collection, analysis date and time, report date and analyst
8. Identification of data reported by subcontractors
9. Test name and EPA reference method number
10. Delivery method and sampling procedures when collected by lab personnel
11. Deviations or modifications that affect data quality
12. Statement that results relate only to the sample tested
13. Statement that report must be copied in full unless the laboratory provides written permission for partial copies
14. Glossary, References and limits of liability
15. Units of measure and reporting detection limit
16. Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
17. Signature, title and date of report
18. A “Certificate/Approval Program Summary” page is included at the end of the report that identifies analytes for which Alpha Analytical holds certification and for those analytes reported that it does not. This summary also includes the certification numbers for either NELAP certified states, State certifications (e.g. Massachusetts

laboratory certification identification number) and DoD certification identifications.

19. Alpha Analytical does not accept samples from private residents for drinking water analysis and therefore maximum contaminant levels are not necessary. If Alpha were to change its policy and report drinking water samples, MCLs would be included with the report.

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory's expense.

The laboratory notifies the customer in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature.

13.1 Data Qualifiers

The following data qualifiers are used in conjunction with analytical results depending on the definition, state or regulatory program and report type.

Note: "J" Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Method Detection Limit (DL) with a "J" qualifier as long as there is a LOD study on file. (See section 5.11)

Data Qualifier	Report Format	Qualifier Information	Regulatory Requirement
A	All DU	Spectra identified as "Aldol Condensation Product".	CT RCP, NC
B	All DU	The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte.	EPA Functional Guidelines 'MassDEP MCP, CT RCP, DoD, NJ-TO15/LL-TO15
C		Pesticide and Aroclor results identified have been confirmed by GC/MS	DE HSCA
C		Co-elution: target analyte co-elutes with a known lab standard (i.e. surrogates, internal standards, etc.) for co-extracted analyses.	Forensics
C		Co-eluting Congener	NJ

D	All DU	Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.	NJ-TO15/LL-TO15 - Air only EPA Functional Guidelines; EPA Region 2,5
E	All DU	Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.	EPA Region 2,5 CT RCP, NJ-TO15/LL-TO15
EMPC		Estimated maximum possible concentration. Indicates that a peak is detected but did not meet all the method required criteria.	NJ
G	All DU	The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.	In-house/Forensics.
G		A single quality control failure occurred during BOD analysis. Results should be used with caution	NC
H	All	The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.	NELAC
H		Sample result is estimated and biased high.	EPA Region 5
I	All DU	The lower value for the two columns has been reported due to obvious interference..	In-house.
J	DU	Estimated value. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs), DoD
J	DU-J	Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (DL). This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs), DoD

J	DU-JRDL	Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above one half the RL. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs), DoD
JN (NJ)	All DU	Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICD), where the identification is based on a mass spectral library search.	EPA Functional Guidelines 'NJ-TO15-LL
N	All DU	The matrix spike recovery exceeds the acceptance criteria. This flag is not applicable when the sample concentration is greater than 4x the spike added. (Metals only.)	ASP
ND	DU-J	Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for same-related analysis	In-house
ND	DU-JRDL	Not detected at one half the reporting limit (RL) for the sample.	In-house
P	All DU	The RPD between the results for the two columns exceeds the method-specified criteria.	MassDEP MCP, CT RCP
P		Elevated PQL due to matrix interference and/or sample dilution.	NC
Q	All DU	The quality control sample exceeds the associated acceptance criteria. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)	DoD
Q		Holding time exceeded. Does not meet NPDES requirements	NC
R	All DU	Analytical results are from sample re-analysis	DoD, Customer-specific
RE	All DU	Analytical results are from sample re-extraction.	DoD, Customer-specific

S		Not enough sample provided to prepare and/or analyze a method-required matrix spike (MS) and/or duplicate (MSD)	NC
U	NA	The analyte was analyzed for but not detected at or above the reporting limit.	EDD formats, DoD, NJDEP, NJ-TO15/LL-TO15, EPA Functional Guidelines

14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests.

The Purchasing SOP/1726 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The Purchasing SOP/1726 describes the details for receipt and inspection of purchased product.

The Purchasing SOP describes the process for raising, review and placement of purchase orders. It is company policy to purchase from third party certified suppliers and subcontractors wherever possible. Purchases must be from suppliers approved by the Laboratory. Laboratory or sampling subcontractors specified by the customer are noted as “Trial” on the purchase order. This identifies the subcontractor as a non-approved subcontractor.

The laboratory maintains list of approved vendors (Form 13-01) and subcontractors from whom it obtains support services or supplies required for tests.

14.1 Subcontracting Analytical Samples

Customers are advised, verbally and/or in writing, if any analyses will be subcontracted to another laboratory. Any testing covered under NELAC that requires subcontracting, will be subcontracted to another NELAC accredited laboratory for the tests to be performed. Any testing covered under the DOD QSM that requires subcontracting, will be subcontracted to another accredited DOD laboratory and must be project-specific approved from the DOD customer before analysis begins. These requirements for DOD projects pertain to both Westboro and Mansfield facilities. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties’ certifications or approvals. This document must indicate current approval for the subcontracted work. Any sample(s) needing special reports (*i.e.*, MCL exceedence) will be identified on the chain of custody when the laboratory subcontracts with another laboratory. Subcontractor Laboratory Certifications are located in Qualtrax under Customer Services folder

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. The quotation form lists the subcontractor in order to notify the customer of any subcontracted work. Customer notification of subcontracted work is in writing before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The person responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor.

15 Customer Relations

15.1 Customer Service

The majority of the customer services occur from personnel in the administration, sample receiving and sampling areas. Customer service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with customers must document and review customer specific project requirements. Call Tracker is used to document communications with customers (SOP/1723). Personnel must document customer interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and customer requests following the laboratory defined procedures.

15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Technical Manager address all capacity and capability issues. Where conflicts in workload arise, customer notification is immediate. The Project Communication Form (PCF) contains the documentation of all project information. Cooperation between laboratory and customer services staff allows direct communication and scheduling. Management arranges complex scheduling and coordination between departmental areas.

15.3 Complaint Processing

The laboratory staff documents all customers or other parties' complaints or concerns regarding the data quality or laboratory operations. The Nonconformance Report records complaints, correcting the concern, and resolving the concern with the customer or other party. The process uses the same form and process as the nonconformance action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiry and Complaint SOP/1722 is immediate to ensure the corrective action has effectively solved the concern.

16 Appendix A – Definitions/References

The following definitions are from Section 3.0 of the 2009 TNI Standard. The laboratory adopts these definitions for all work performed in the laboratory. In addition, there are clarifications to certain definitions according to the DoD QSM.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. (TNI)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (TNI)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (DoD; EPA QAD glossary)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation. (TNI)

Assessment (Clarification): The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria. (DoD)

Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (TNI)

Blanks include:

Equipment Blank: a sample of analyte-free media, which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

Instrument Blank: a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses, (TNI)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

Calibration: set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).

2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the Laboratory with a certificate of analysis or purity, or prepared by the Laboratory using support equipment that has been calibrated verified to meet specifications.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Method: A defined technical procedure for performing a calibration.

Calibration Standard: A substance or reference material used to calibrate an instrument. (TNI)

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols (TNI)

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq*., Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures (TNI)

Customer: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

Continuing calibration verification: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Quality Objectives (DQO):

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (TNI)

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: (previously referred to as Method Detection Limit –MDL) the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.

Detection Limit (DL) (Clarification): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%. (DoD)

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq*., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C 1251 et seq., Public Law 92-50086 Stat. 8.16, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field Measurement: The determination of physical, biological, or radiological properties, or chemical constituents; that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation. (TNI)

Finding: an assessment conclusion, referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. (TNI)

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

Holding Times: The maximum time that can elapse between two (2) specified activities. (TNI)

The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

Holding Times (DoD Clarification): The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (TNI)

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory: Body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (TNI).

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the customer. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory. (TNI)

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

Limit of Detection (Clarification): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD)

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence. (TNI)

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD)

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work. (ANSI/ASQ E4-2004)

Management System: System to establish policy and objectives and to achieve those objectives (ISO 9000)

Matrix: The substrate of a test sample. (TNI)

Matrix Spike (spiked sample, fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (TNI).

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (TNI).

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). (TNI)

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed. (TNI)

Method Detection Limit: (now referred to as Detection Limit) one way to establish a Detection Limit, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Method Detection Limit (MDL) (Clarification): The MDL is one way to establish a Detection Limit, not a Limit of Detection. (DoD)

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Mobile Laboratory: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel. (TNI)

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute. (NMI). (TNI)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which TNI is a part.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI).

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Procedure: A specified way to carry out an activity or a process. Procedures can be documented or not. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (TNI).

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is the type and quality needed and expected by the customer. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements or quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or

actual samples fortified by spiking intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users. (TNI)

Quality Manual Clarification: Alpha Analytical refers to Quality Manual as Corporate Quality Systems Manual (CQSM). (Alpha)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities. (TNI)

Quality System Matrix: These matrix definitions are to be used for purposes of batch and quality control requirements: (TNI)

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location. (TNI)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure. (TNI)**Second source calibration verification (ICV):** A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent. (TNI)

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (TNI)

Standard Method: a test method issued by an organization generally recognized as competent to do so.

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Technology: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques. (TNI)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Test Method: An adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e. the air, water and land) upon which human life depends. (US-EPA)

Validation: the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring

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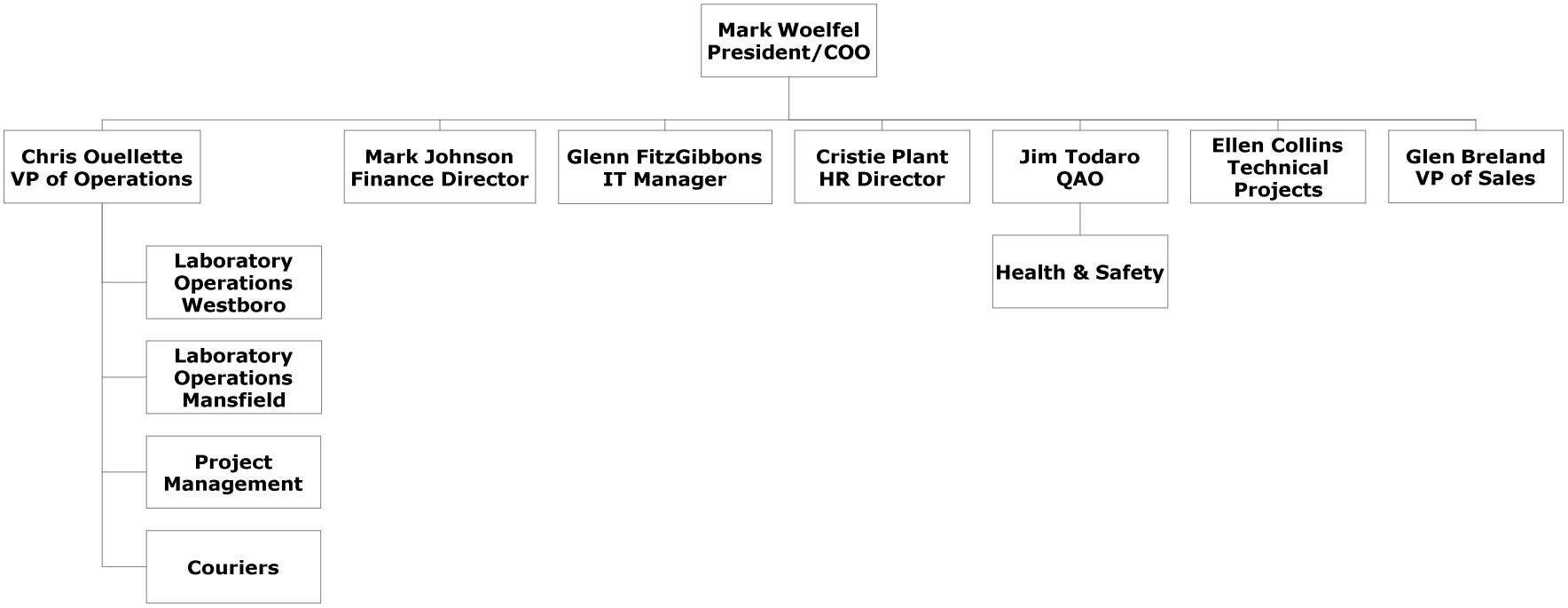
17 Appendix B – Organization Charts

The following charts provide an overview of the organizational structure of Alpha Analytical. The chart also identifies the key personnel responsible for the listed positions. For the various laboratory areas, the individual departmental supervisors are noted. For a listing of all current key personnel, please refer to Section 18, Appendix C.

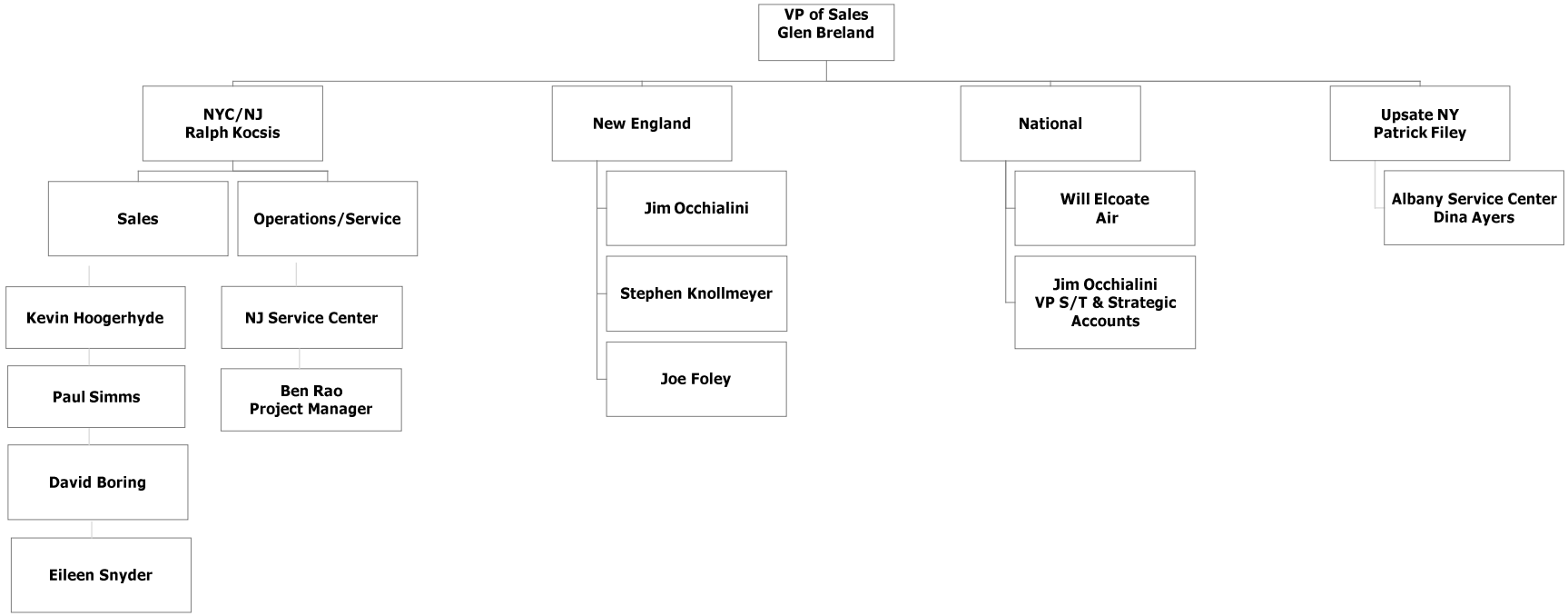
Updated 10/29/2013

Alpha Analytical
Company Organizational Chart



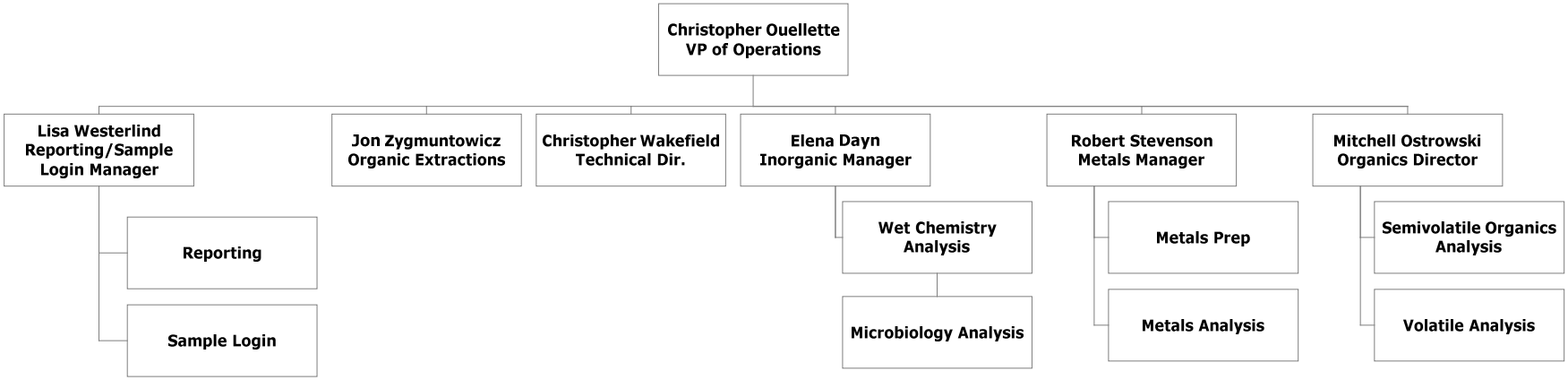
Updated 10/29/2013

2013 Sales Organization

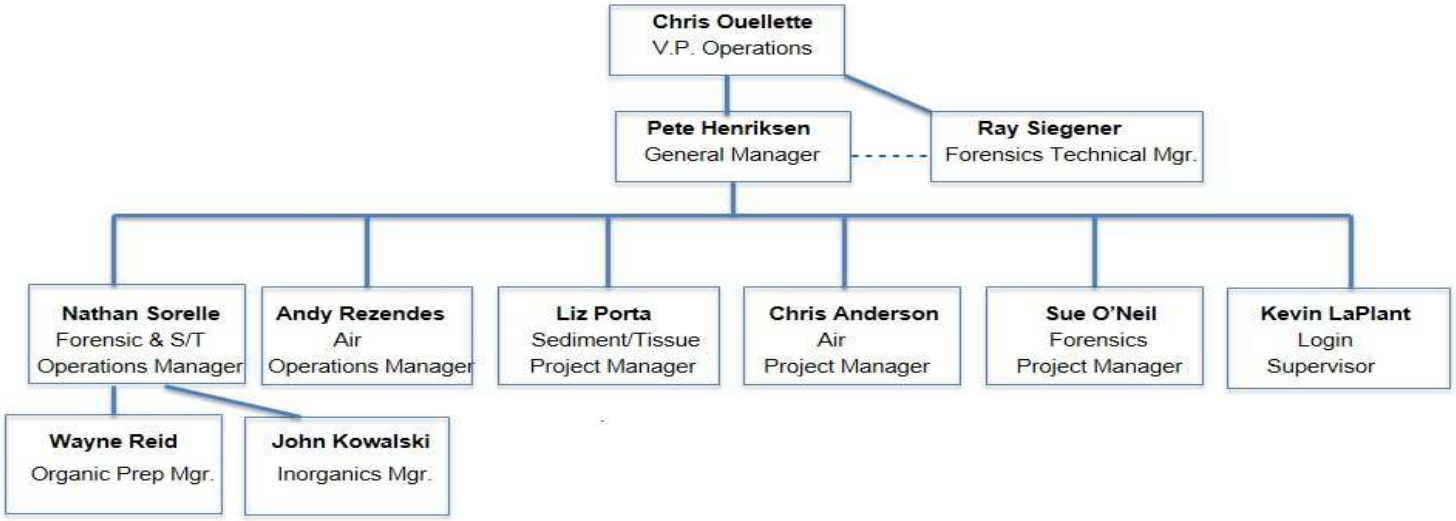


Updated 10/29/2013

Alpha Analytical
Laboratory Organizational Chart
WESTBOROUGH



Alpha Analytical
Laboratory Organization Chart
Mansfield



18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 7/25/2012.**

President / Sales Manager: Mark Woelfel

Director of Operations: Christopher Ouellette

Laboratory Technical Manager Westboro: Christopher Wakefield

Laboratory Technical Manager / Mansfield: Joseph Watkins

Laboratory Technical Manager- Air, Volatiles Manager, Mansfield: Andy Rezendes

Quality Assurance Officer: James C. Todaro

Quality Systems Specialists: Amy Rice, Rene Bennett, Jason Hebert

VP, Technical Projects: Ellen Collins

Human Resources Director: Cristie Plant

Vice Presidents, Technical Sales: Glen Breland, James Occhialini, Ralph Kocsis, Will Eloate, Pat Filey, Kevin Hoogerhyde

Technical Sales Reps: Paul Simms; Joe Foley; Steven Knollmeyer

Controller: Mark Johnson

A/P, Purchasing: Jennifer Walters

Credit & Collections Supervisor: Holly Palmer

Information Technology: Glenn Fitzgibbons

VP, Sales and Services: Glen Breland

Customer Services Manager, Westboro: Mary Davis

General Manager, Mansfield: Peter Henriksen

Inorganics Department Manager, Westboro: Elena Dayn

Metals Departmet Manager: Robert Stevenson

Organics Department Manager,Westboro: Mitch Ostrowski

Login Manager/ Reporting Manager Westboro Lisa Westerlind

Organic Extractions Supervisor, Westboro: John Zygmuntowicz

Forensic & S/T Operations Manager, Mansfield: Nathan Sorelle

Forensics Technical Manager, Mansfield: Ray Siegener

Equipment Maintenance: Chris Wakefield, Pat Sullivan, Greg Yogis

Environmental Health & Safety Coordinator: Jeanette Soucy

Courier Manager: Kevin Lento

Hazardous Materials Consultant: Triumvirate

19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals		
Equipment	**Frequency**	**Type of Calibration or Maintenance**
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights
COD Reactor	annually annually	complete operations check by service technician (external) reaction temperature verification
Conductivity Bridge	annually each use	verification of cell constant complete operations check by service technician (external) calibration verification
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check
DO Meter	annually each use	complete operations check by service technician (external) calibration against air as specified by manufacturer
Emergency/Safety Equipment	annually monthly	fire extinguishers and emergency exit lighting check eye washes, showers, fire blanket and first aid kits checked
Freezers	daily	temperature verification
Gas Chromatographs	as needed as needed beginning and end of batch and 10 to 20 samples as per method	injection port preparation; cleaning of detectors initial multi-point calibration continuing calibration verification (CCV) against initial calibration
ICP	Every other day Daily Annually Annually As needed	Change pump tubing Calibration, profile Complete operations check by service technician (external), Linear Dynamic Range determination Clean torch, clean nebulizer, clean spray chamber
Lachat analyzer	Daily As needed	Calibration, clean lines Change tubing, change O-rings
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or daily	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV
Mercury Analyzer	monthly each use	clean cell and change pump windings calibration using multi-point curve
Auto-pipettes	Monthly Annually	verification of accuracy verification of precision
Microwave	Quarterly Annually	power and temperature verification RPM verification
Ovens	annually daily	complete operations check by service technician (external) temperature verification
pH Meters	annually each use	complete operations check by service technician (external) calibration using certified buffers
Refrigerators (General Use)	daily	temperature verification
Refrigerators (Sample Management)	daily	temperature verification
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration
TCLP Rotator	annually	RPM verification
Thermometers (Mercury/Alcohol)	annually	calibration against NIST traceable thermometer (internal)
Thermometers (digital)	Quarterly	calibration against NIST traceable thermometer (external)
Thermometer (NIST Traceable)	annually	calibration and certification of conformance (external)
Turbidity meter	annually each use	cleaning & operations check by service technician (external) calibration using formazin
Weights (Class S-1)	annually	service/calibration and certification of conformance (external)

20 Appendix E – List of Analytical Methods

Certificate/Approval Program Summary

Last revised October 1, 2013 - Westboro Facility

The following list includes only those analytes/methods for which certification/approval is currently held. For a complete listing of analytes for the referenced methods, please contact your Alpha Customer Service Representative.

Connecticut Department of Public Health Certificate/Lab ID: PH-0574. ***NELAP Accredited Solid Waste/Soil.***

Drinking Water (Inorganic Parameters: Color, pH, Turbidity, Conductivity, Alkalinity, Chloride, Free Residual Chlorine, Fluoride, Calcium Hardness, Sulfate, Nitrate, Nitrite, Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Nickel, Selenium, Silver, Sodium, Thallium, Zinc, Total Dissolved Solids, Total Organic Carbon, Total Cyanide, Perchlorate. Organic Parameters: Volatile Organics 524.2, Total Trihalomethanes 524.2, 1,2-Dibromo-3-chloropropane (DBCP) 504.1, Ethylene Dibromide (EDB) 504.1, 1,4-Dioxane (Mod 8270). Microbiology Parameters: Total Coliform-MF mEndo (SM9222B), Total Coliform – Colilert (SM9223, Enumeration and P/A), E. Coli. – Colilert (SM9223, Enumeration and P/A), HPC – Pour Plate (SM9215B), Fecal Coliform – MF m-FC (SM9222D), Fecal Coliform-EC Medium (SM 9221E).

Wastewater/Non-Potable Water (Inorganic Parameters: Color, pH, Conductivity, Acidity, Alkalinity, Chloride, Total Residual Chlorine, Fluoride, Total Hardness, Silica, Sulfate, Sulfide, Ammonia, Kjeldahl Nitrogen, Nitrate, Nitrite, O-Phosphate, Total Phosphorus, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc, Total Residue (Solids), Total Dissolved Solids, Total Suspended Solids (non-filterable), BOD, CBOD, COD, TOC, Total Cyanide, Phenolics, Foaming Agents (MBAS), Bromide, Oil and Grease. Organic Parameters: PCBs, Organochlorine Pesticides, Technical Chlordane, Toxaphene, Acid Extractables (Phenols), Benzidines, Phthalate Esters, Nitrosamines, Nitroaromatics & Isophorone, Polynuclear Aromatic Hydrocarbons, Haloethers, Chlorinated Hydrocarbons, Volatile Organics, TPH (HEM/SGT), CT- Extractable Petroleum Hydrocarbons (ETPH), MA-EPH, MA-VPH. Microbiology Parameters: Total Coliform – MF mEndo (SM9222B), Total Coliform – MTF (SM9221B), E. Coli – Colilert (SM9223 Enumeration), HPC – Pour Plate (SM9215B), Fecal Coliform – MF m-FC (SM9222D), Fecal Coliform – A-1 Broth (SM9221E), Enterococcus - Enterolert.

Solid Waste/Soil (Inorganic Parameters: pH, Sulfide, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Tin, Vanadium, Zinc, Total Cyanide, Ignitability, Phenolics, Corrosivity, TCLP Leach (1311), SPLP Leach (1312 metals only), Reactivity. Organic Parameters: PCBs, PCBs in Oil, Organochlorine Pesticides, Technical Chlordane, Toxaphene, CT-Extractable Petroleum Hydrocarbons (ETPH), MA-EPH, MA-VPH, Dicamba, 2,4-D, 2,4,5-T, 2,4,5-TP(Silvex), Dalapon, Volatile Organics (SW 8260), Acid Extractables (Phenols) (SW 8270), Benzidines (SW 8270), Phthalates (SW 8270), Nitrosamines (SW 8270), Nitroaromatics & Cyclic Ketones (SW 8270), PAHs (SW 8270), Haloethers (SW 8270), Chlorinated Hydrocarbons (SW 8270).)

State of Illinois Certificate/Lab ID: 003155. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: SM2120B, 2320B, 2510B, 2540C, SM4500CN-CE, 4500F-C, 4500H-B, 4500NO3-F, 5310C, EPA 200.7, 200.8, 245.1, 300.0. Organic Parameters: EPA 504.1, 524.2.)

Wastewater/Non-Potable Water (Inorganic Parameters: SM2120B, 2310B, 2320B, 2340B, 2510B, 2540B,

2540C, 2540D, SM4500CL-E, 4500CN-E, 4500F-C, 4500H-B, 4500NH3-H, 4500NO2-B, 4500NO3-F, 4500P-E, 4500S-D, 4500SO3-B, 5210B, 5220D, 5310C, 5540C, EPA 120.1, 1664A, 200.7, 200.8, 245.1, 300.0, 350.1, 351.1, 353.2, 410.4, 420.1. Organic Parameters: EPA 608, 624, 625.)

Hazardous and Solid Waste (Inorganic Parameters: EPA 1010A, 1030, 1311, 1312, 6010C, 6020A, 7196A, 7470A, 7471B, 9012B, 9014, 9038, 9040C, 9045D, 9050A, 9065, 9251. Organic Parameters: 8011 (NPW only), 8015C, 8081B, 8082A, 8151A, 8260C, 8270D, 8315A, 8330.)

Maine Department of Human Services Certificate/Lab ID: 2009024.
Drinking Water (Inorganic Parameters: SM9215B, 9222D, 9223B, EPA 180.1, 353.2, SM2120B, 2130B, 2320B, 2510C, 2540C, 4500Cl-D, 4500CN-C, 4500CN-E, 4500F-C, 4500H+B, 4500NO3-F, 5310C, EPA 200.7, EPA 200.8, 245.1, EPA 300.0. Organic Parameters: 504.1, 524.2.)

Wastewater/Non-Potable Water (Inorganic Parameters: EPA 120.1, 1664A, 300.0, 350.1, 351.1, 353.2, 410.4, 420.1, 8315A, 9010C, SM2120B, 2310B, 2320B, 2510B, 2540B, 2540C, 2540D, 426C, 4500Cl-E, 4500CN-C, 4500CN-E, 4500F-B, 4500F-C, 4500H+B, 4500Norg-C, 4500NH3-B, 4500NH3-H, 4500NO2-B, 4500NO3-F, 4500P-B, 4500P-E, 4500S2-D, 4500SO3-B, 5540C, 5210B, 5220D, 5310C, 9010B, 9030B, 9040C, 7470A, 7196A, 2340B, EPA 200.7, 6010C, 200.8, 6020A, 245.1, 1311, 1312, 3005A, Enterolert, 9223B, 9222D. Organic Parameters: 608, 624, 625, 8011, 8081B, 8082A, 8330, 8151A, 8260C, 8270D, 3510C, 3630C, 5030B, ME-DRO, ME-GRO, MA-EPH, MA-VPH.)

Solid Waste/Soil (Inorganic Parameters: 9010B, 9012A, 9014, 9040B, 9045C, 6010C, 6020A, 7471B, 7196A, 9050A, 1010, 1030, 9065, 1311, 1312, 3005A, 3050B, 9038, 9251. Organic Parameters: ME-DRO, ME-GRO, MA-EPH, MA-VPH, 8260C, 8270D, 8330, 8151A, 8081B, 8082A, 3540C, 3546, 3580A, 3620C, 3630C, 5030B, 5035.)

Massachusetts Department of Environmental Protection Certificate/Lab ID: M-MA086.
*Drinking Water (*Inorganic Parameters: (EPA 200.8 for: Sb,As,Ba,Be,Cd,Cr,Cu,Pb,Ni,Se,Tl) (EPA 200.7 for: Ba,Be,Ca,Cd,Cr,Cu,Na,Ni) 245.1, (300.0 for: Nitrate-N, Fluoride, Sulfate); (EPA 353.2 for: Nitrate-N, Nitrite-N); (SM4500NO3-F for: Nitrate-N and Nitrite-N); 4500F-C, 4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, 2320B, SM2540C, SM4500H-B. Organic Parameters: (EPA 524.2 for: Trihalomethanes, Volatile Organics); (504.1 for: 1,2-Dibromoethane, 1,2-Dibromo-3-Chloropropane), EPA 332. Microbiology Parameters: SM9215B; ENZ. SUB. SM9223; ColilertQT SM9223B; MF-SM9222D.)

*Non-Potable Water (*Inorganic Parameters:, (EPA 200.8 for: Al,Sb,As,Be,Cd,Cr,Cu,Pb,Mn,Ni,Se,Ag,Tl,Zn); (EPA 200.7 for: Al,Sb,As,Be,Cd,Ca,Cr,Co,Cu,Fe,Pb,Mg,Mn,Mo,Ni,K,Se,Ag,Na,Sr,Ti,Tl,V,Zn); 245.1, SM4500H,B, EPA 120.1, SM2510B, 2540C, 2340B, 2320B, 4500CL-E, 4500F-BC, 426C, SM4500NH3-BH, (EPA 350.1 for: Ammonia-N), LACHAT 10-107-06-1-B for Ammonia-N, SM4500NO3-F, 353.2 for Nitrate-N, SM4500NH3-BC-NES, EPA 351.1, SM4500P-E, 4500P-B,E, 5220D, EPA 410.4, SM 5210B, 5310C, 4500CL-D, EPA 1664, SM14 510AC, EPA 420.1, SM4500-CN-CE, SM2540D.
Organic Parameters: (EPA 624 for Volatile Halocarbons, Volatile Aromatics),(608 for: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT,Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs-Water), (EPA 625 for SVOC Acid Extractables and SVOC Base/Neutral Extractables), 600/4-81-045-PCB-Oil. Microbiology Parameters: (ColilertQT SM9223B; Enterolert-QT: SM9222D-MF.)

New Hampshire Department of Environmental Services Certificate/Lab ID: 200307. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: SM 9222B, 9223B, 9215B, EPA 200.7, 200.8, 300.0, SM4500CN-E, 4500H+B, 4500NO3-F, 2320B, 2510B, 2540C, 4500F-C, 5310C, 2120B, EPA 332.0. Organic Parameters: 504.1, 524.2.)

Non-Potable Water (Inorganic Parameters: SM9222D, 9221B, 9222B, 9221E-EC, EPA 3005A, 200.7, 200.8, 245.1, SW-846 6010C, 6020A, 7196A, 7470A, SM3500-CR-D, EPA 120.1, 300.0, 350.1, 350.2, 351.1, 353.2, 410.4, 420.1, 426C, 1664A, SW-846 9010B, 9010C, 9030, 9040B, 9040C, SM2120B, 2310B, 2320B, 2340B, 2540B, 2540D, 4500H+B, 4500CL-E, 4500CN-E, 4500NH3-H, 4500NO3-F, 4500NO2-B, 4500P-E, 4500-S2-D, 4500SO3-B, 5210B, 5220D, 2510B, 2540C, 4500F-C, 5310C, 5540C, LACHAT 10-204-00-1-A, LACHAT 10-107-06-2-D, 3060A. Organic Parameters: SW-846 3510C, 3630C, 5030B, 8260C, 8270D, 8330, EPA 624, 625, 608, SW-846 8082A, 8081B, 8015C, 8151A, 8330, 8270D-SIM.)

Solid & Chemical Materials (Inorganic Parameters: SW-846 6010C, 6020A, 7196A, 7471B, 1010, 1010A, 1030, 9010C, 9012B, 9014, 9030B, 9040C, 9045C, 9045D, 9050, 9065, 9251, 1311, 1312, 3005A, 3050B, 3060A. Organic Parameters: SW-846 3540C, 3546, 3050B, 3580A, 3620D, 3630C, 5030B, 5035, 8260C, 8270D, 8270D-SIM, 8330, 8151A, 8015B, 8015C, 8082A, 8081B.)

New Hampshire Department of Environmental Services Certificate/Lab ID: 2064. ***NELAP Accredited.***
Drinking Water (Organic Parameters: **EPA 524.2:** Di-isopropyl ether (DIPE), Ethyl-t-butyl ether (ETBE), Tert-amyl methyl ether (TAME)).

Non-Potable Water (Organic Parameters: **EPA 8260C:** 1,3,5-Trichlorobenzene. **EPA 8015C(M):** TPH.)

Solid & Chemical Materials (Organic Parameters: **EPA 8260C:** 1,3,5-Trichlorobenzene.)

New Jersey Department of Environmental Protection Certificate/Lab ID: MA935. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: SM9222B, 9221E, 9223B, 9215B, 4500CN-CE, 4500NO3-F, 4500F-C, EPA 300.0, 200.7, 200.8, 245.1, 2540C, SM2120B, 2320B, 2510B, 5310C, SM4500H-B. Organic Parameters: EPA 332, 504.1, 524.2.)

Non-Potable Water (Inorganic Parameters: SM5210B, EPA 410.4, SM5220D, 4500Cl-E, EPA 300.0, SM2120B, 2340B, SM4500F-BC, EPA 200.7, 200.8, 351.1, LACHAT 10-107-06-2-D, EPA 353.2, SM4500NO3-F, 4500NO2-B, EPA 1664A, SM5310C, 4500-PE, EPA 420.1, SM4500P-B5+E, 2540B, 2540C, 2540D, EPA 120.1, SM2510B, 9222D, 9221B, 9221C, 9221E, 9222B, 9215B, 2310B, 2320B, 4500NH3-H, 4500-S D, 4500SO4-E, EPA 350.1, 350.2, SW-846 1312, 7470A, 5540C, SM4500H-B, 4500SO3-B, SM3500Cr-D, 4500CN-CE, EPA 245.1, SW-846 9040B, 9040C, 3005A, 3015, EPA 6010B, 6010C, 6020, 6020A, 7196A, 3060A, SW-846 9010C, 9030B. Organic Parameters: SW-846 8260B, 8260C, 8270C, 8270D, 8270C-SIM, 8270D-SIM, 3510C, EPA 608, 624, 625, SW-846 3630C, 5030B, 5030C, 8011, 8015C, 8081A, 8081B, 8082, 8082A, 8151A, 8330, 1,4-Dioxane by NJ Modified 8270, 8015B, NJ EPH.)

Solid & Chemical Materials (Inorganic Parameters: SW-846, 6010B, 6010C, 6020, 6020A, 7196A, 3060A, 9030B, 1010, 1010A, 1030, 1311, 1312, 3005A, 3050B, 7471A, 7471B, 9010C, 9012B, 9014, 9038, 9040B, 9040C, 9045C, 9045D, 9050A, 9065, 9251. Organic Parameters: SW-846 8015B, 8015C, 8081A, 8081B, 8082, 8082A, 8151A, 8330, 8260B, 8260C, 8270C, 8270D, 8270C-SIM, 8270D-SIM, 3540C, 3546, 3580A, 3620C, 3630C, 5030B, 5030C, 5035L, 5035H, NJ EPH.)

New York Department of Health Certificate/Lab ID: 11148. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: SM9223B, 9222B, 9215B, EPA 200.8, 200.7, 245.1, SM5310C, EPA 332.0, SM2320B, EPA 300.0, SM2120B, 4500CN-E, 4500F-C, 4500NO3-F, 2540C, SM 2510B. Organic Parameters: EPA 524.2, 504.1.)

Non-Potable Water (Inorganic Parameters: SM9221E, 9222D, 9221B, 9222B, 9215B, 5210B, 5310C, EPA 410.4, SM5220D, 2310B, 2320B, EPA 200.7, 300.0, SM4500CL-E, 4500F-C, SM15 426C, EPA 350.1, SM4500NH3-BH, EPA 351.1, LACHAT 10-107-06-2, EPA 353.2, SM4500-NO3-F, 4500-NO2-B, 4500P-E, 2340B, 2540C, 2540B, 2540D, EPA 200.8, EPA 6010C, 6020A, EPA 7196A, SM3500Cr-D,

EPA 245.1, 7470A, SM2120B, 4500CN-CE, EPA 1664A, EPA 420.1, SM14 510C, EPA 120.1, SM2510B, SM4500S-D, SM5540C, EPA 8315A, 3005A, 9010C, 9030B. Organic Parameters: EPA 624, 8260C, 8270D, 8270D-SIM, 625, 608, 8081B, 8151A, 8330A, 8082A, EPA 3510C, 5030B, 5030C, 8015C, 8011.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 1010A, 1030, EPA 6010C, 6020A, 7196A, 7471B, 8315A, 9012B, 9014, 9065, 9050A, 9038, 9251, EPA 1311, 1312, 3005A, 3050B, 9010C, 9030B, 9040C, 9045D. Organic Parameters: EPA 8260C, 8270D, 8270D-SIM, 8015C, 8081B, 8151A, 8330A, 8082A, 3540C, 3546, 3580A, 5035A-H, 5035A-L.)

North Carolina Department of the Environment and Natural Resources Certificate/Lab ID : 666. (Inorganic Parameters: SM2310B, 2320B, 4500Cl-E, 4500Cn-E, 9012B, 9014, Lachat 10-204-00-1-X, 1010A, 1030, 4500NO3-F, 353.2, 4500P-E, 4500SO4-E, 300.0, 4500S-D, 5310B, 5310C, 6010C, 6020A, 200.7, 200.8, 3500Cr-B, 7196A, 245.1, 7470A, 7471B, 1311,1312. Organic Parameters: 608, 8081B, 8082A, 624, 8260B, 625, 8270D, 8151A, 8015C, 504.1, MA-EPH, MA-VPH.)

Drinking Water Program Certificate/Lab ID: 25700. (Inorganic Parameters: Chloride EPA 300.0. Organic Parameters: 524.2)

Pennsylvania Department of Environmental Protection Certificate/Lab ID : 68-03671. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: 200.7, 200.8, 300.0, 332.0, 2120B, 2320B, 2510B, 2540C, 4500-CN-CE, 4500F-C, 4500H+-B, 4500NO3-F, 5310C. Organic Parameters: EPA 524.2, 504.1)

Non-Potable Water (Inorganic Parameters: EPA 120.1, 1312, 3005A,3015, 3060A, 200.7, 200.8, 410.4, 1664A, SM2540D, 5210B, 5220D, 4500-P,BE, 245.1, 300.0, 350.1, 350.2, 351.1, 353.2, 420.1, 6010C, 6020A, 7196A, 7470A, 9030B, 2120B, 2310B, 2320B, 2510B, 2540B, 2540C, 3500Cr-D, 426C, 4500CN-CE, 4500Cl-E, 4500F-B, 4500F-C, 4500H+-B, 4500NH3-H, 4500NO2-B, 4500NO3-F, 4500S-D, 4500SO3-B, 5310BCD, 5540C, 9010C, 9040C. Organic Parameters: EPA 3510C, 3630C, 5030B, 625, 624, 608, 8081B, 8082A, 8151A, 8260C, 8270D, 8270D-SIM, 8330, 8015C, NJ-EPH.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 350.1, 1010, 1030, 1311, 1312, 3005A, 3050B, 3060A, 6010C, 6020A, 7196A, 7471B, 9010C, 9012B, 9014, 9040B, 9045D, 9050A, 9065, SM 4500NH3-BH, 9030B, 9038, 9251. Organic Parameters: 3540C, 3546, 3580A, 3620C, 3630C, 5035, 8015C, 8081B, 8082A, 8151A, 8260C, 8270D, 8270D-SIM, 8330, NJ-EPH.)

Rhode Island Department of Health Certificate/Lab ID: LAO00065. ***NELAP Accredited via NJ-DEP.***
Refer to MA-DEP Certificate for Potable and Non-Potable Water.
Refer to NJ-DEP Certificate for Potable and Non-Potable Water.

Texas Commisson on Environmental Quality Certificate/Lab ID: T104704476. ***NELAP Accredited.***
Non-Potable Water (Inorganic Parameters: EPA 120.1, 1664, 200.7, 200.8, 245.1, 245.2, 300.0, 350.1, 351.1, 353.2, 410.4, 420.1, 6010, 6020, 7196, 7470, 9040, SM 2120B, 2310B, 2320B, 2510B, 2540B, 2540C, 2540D, 426C, 4500CL-E, 4500CN-E, 4500F-C, 4500H+B, 4500NH3-H, 4500NO2B, 4500P-E, 4500 $S2^{-}$D, 510C, 5210B, 5220D, 5310C, 5540C. Organic Parameters: EPA 608, 624, 625, 8081, 8082, 8151, 8260, 8270, 8330.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 1311, 1312, 9012, 9014, 9040, 9045, 9050, 9065.)

Virginia Division of Consolidated Laboratory Services Certificate/Lab ID: 460195. ***NELAP Accredited.***
Drinking Water (Inorganic Parameters: EPA 200.7, 200.8, 300.0, 2510B, 2120B, 2540C, 4500CN-CE, 245.1, 2320B, 4500F-C, 4500NO3-F, 4500H+B, 5310C. Organic Parameters: EPA 504.1, 524.2.)

Non-Potable Water (Inorganic Parameters: EPA 120.1, 1664A, 200.7, 200.8, 245.1, 300.0, 350.1, 351.1, 351.2, 3005A, 3015, 1312, 6010B, 6010C, 3060A, 353.2, 420.1, 2340B, 6020, 6020A, SM4500S-D, SM4500-CN-CE, Lachat 10-204-00-1-X, 7196A, 7470A, 2310B, 2320B, 2510B, 2540B, 2540C, 2540D, 3500Cr-D, 426C, 4500Cl-E, 4500F-B, 4500F-C, 4500NH3-H, 4500NO2-B, 4500NO3-F, 4500 SO3-B, 4500H-B, 4500PE, 510AC, 5210B, 5310B 5310C, 5540C, 9010Cm 9030B, 9040C. Organic Parameters: EPA 3510C, 3630C, 5030B, 8260B, 608, 624, 625, 8011, 8015C, 8081A, 8081B, 8082, 8082A, 8151A, 8260C, 8270C, 8270D, 8270C-SIM, 8270D-SIM, 8330,)

Solid & Hazardous Waste (Inorganic Parameters: EPA 1010A, 1030, 3060A, 3050B, 1311, 1312, 6010B, 6010C, 6020, , 7196A, 7471A, 7471B, 6020A, 9010C, 9012B, 9030B, 9014, 9038, 9040C, 9045D, 9251, 9050A, 9065. Organic Parameters: EPA 5030B, 5035, 3540C, 3546, 3550B, 3580A, 3620C, 3630C, 6020A, 8260B, 8260C, 8015B, 8015C, 8081A, 8081B, 8082, 8082A, 8151A, 8270C, 8270D, 8270C-SIM, 8270D-SIM, 8330.)

Department of Defense, L-A-B Certificate/Lab ID: L2217.
Drinking Water (Inorganic Parameters: SM 4500H-B. Organic Parameters: EPA 524.2, 504.1.)

Non-Potable Water (Inorganic Parameters: EPA 200.7, 200.8, 6010C, 6020A, 245.1, 7470A, 9040B, 9010B, 180.1, 300.0, 332.0, 6860, 351.1, 353.2, 9060, 1664A, SM 4500CN-E, 4500H-B, 4500Norg-C, 4500NO3-F, 5310C, 2130B, 2320B, 2340B, 2540C, 5540C, 3005A, 3015, 9056, 7196A, 3500-Cr-D. Organic Parameters: EPA 8015C, 8151A, 8260C, 8270D, 8270D-SIM, 8330A, 8082A, 8081B, 3510C, 5030B, MassDEP EPH, MassDEP VPH.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 200.7, 6010C, 6020A, 7471A, 6860, 1311, 1312, 3050B, 7196A, 9040B, 9045C, 9010C, 9012B, 9251, SM3500-CR-D, 4500CN-CE, 2540G, Organic Parameters: EPA 8015C, 8151A, 8260C, 8270D, 8270D-SIM, 8330A/B-prep, 8082A, 8081B, 3540C, 3546, 3580A, 5035A, MassDEP EPH, MassDEP VPH.)

The following analytes are not included in our current NELAP/TNI Scope of Accreditation:
EPA 524.2: Acetone, 2-Butanone (Methyl ethyl ketone (MEK)), Tert-butyl alcohol, 2-Hexanone, Tetrahydrofuran, 1,3,5-Trichlorobenzene, 4-Methyl-2-pentanone (MIBK), Carbon disulfide, Diethyl ether. **EPA 8260B:** 1,2,4,5-Tetramethylbenzene, 4-Ethyltoluene. **EPA 8260 Non-potable water matrix:** Iodomethane (methyl iodide), Methyl methacrylate. **EPA 8260 Soil matrix:** Tert-amyl methyl ether (TAME), Diisopropyl ether (DIPE), Azobenzene. **EPA 8330A:** PETN, Picric Acid, Nitroglycerine, 2,6-DANT, 2,4-DANT. **EPA 8270C:** Methyl naphthalene, Dimethyl naphthalene, Total Methylnapthalenes, Total Dimethylnaphthalenes, 1,4-Diphenylhydrazine. **EPA 625:** 4-Chloroaniline, 4-Methylphenol. Total Phosphorus in a soil matrix, TKN in a soil matrix, NO2 in a soil matrix, NO3 in a soil matrix. **EPA 9071:** Total Petroleum Hydrocarbons, Oil & Grease.

Certificate/Approval Program Summary

Last revised October 1, 2013 – Mansfield Facility

The following list includes only those analytes/methods for which certification/approval is currently held. For a complete listing of analytes for the referenced methods, please contact your Alpha Customer Service Representative.

Connecticut Department of Public Health Certificate/Lab ID: PH-0141.

Wastewater/Non-Potable Water (Inorganic Parameters: pH, Turbidity, Conductivity, Alkalinity, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Vanadium, Zinc, Total Residue (Solids), Total Suspended Solids (non-filterable). Organic Parameters: PCBs, Organochlorine Pesticides, Technical Chlordane, Toxaphene, Acid Extractables, Benzidines, Phthalate Esters, Nitrosamines, Nitroaromatics & Isophorone, PAHs, Haloethers, Chlorinated Hydrocarbons, Volatile Organics.)

Solid Waste/Soil (Inorganic Parameters: pH, Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Titanium, Vanadium, Zinc, Total Organic Carbon, Corrosivity, TCLP 1311, SPLP 1312. Organic Parameters: PCBs, Organochlorine Pesticides, Technical Chlordane, Toxaphene, Volatile Organics, Acid Extractables, Benzidines, Phthalates, Nitrosamines, Nitroaromatics & Cyclic Ketones, PAHs, Haloethers, Chlorinated Hydrocarbons.)

Florida Department of Health Certificate/Lab ID: E87814. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters: SM2320B, SM2540D, SM2540G.)

Solid & Chemical Materials (Inorganic Parameters: 6020, 7470, 7471, 9045. Organic Parameters: EPA 8260, 8270, 8082, 8081.)

Air & Emissions (EPA TO-15.)

Louisiana Department of Environmental Quality Certificate/Lab ID: 03090. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters: EPA 180.1, 245.7, 1631E, 3020A, 6020A, 7470A, 9040, 9050A, SM2320B, 2540D, 2540G, 4500H-B, Organic Parameters: EPA 3510C, 3580A, 3630C, 3640A, 3660B, 3665A, 5030B, 8015D, 3570, 8081B, 8082A, 8260B, 8270C, 8270D.)

Solid & Chemical Materials (Inorganic Parameters: EPA 1311, 3050B, 3051A, 3060A, 6020A, 7196A, 7470A, 7471B, 7474, 9040B, 9045C, 9060. Organic Parameters: EPA 3540C, 3570, 3580A, 3630C, 3640A, 3660, 3665A, 5035, 8015D, 8081B, 8082A, 8260B, 8270C, 8270D.)

Biological Tissue (Inorganic Parameters: EPA 6020A. Organic Parameters: EPA 3570, 3510C, 3610B, 3630C, 3640A, 8270C, 8270D.)

Air & Emissions (EPA TO-15.)

New Hampshire Department of Environmental Services Certificate/Lab ID: 2206. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters: EPA 180.1, 1631E, 6020A, 7470A, 9040B, 9050A, SM2540D, 2540G, 4500H+B, 2320B, 3020A, . Organic Parameters: EPA 3510C, 3630C, 3640A, 3660B, 8081B, 8082A, 8270C, 8270D, 8015D.)

Solid & Chemical Materials (Inorganic Parameters: SW-846 1311, 3050B, 3051A, 6020A, 7471B, 9040B, 9045C. Organic Parameters: SW-846 3540C, 3580A, 3630C, 3640A, 3660B, 3665A, 8270C, 8015D, 8082A, 8081B.)

New Jersey Department of Environmental Protection Certificate/Lab ID: MA015. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters: SW-846 1312, 3020A, SM2320B, SM2540D, 2540G, 4500H-B, EPA 180.1, 1631E, SW-846 7470A, 9040C, 6020A, 9050A. Organic Parameters: SW-846 3510C, 3580A, 3630C, 3640A, 3660B, 3665A, 8015D, 8081B, 8082A, 8270C, 8270D)

Solid & Chemical Materials (Inorganic Parameters: SW-846 1311, 1312, 3050B, 3051A, 6020A, 7471B, 7474, 9040B, 9040C, 9045C, 9045D, 9060, 9060A. Organic Parameters: SW-846 3540C, 3570, 3580A, 3630C, 3640A, 3660B, 3665A, 8081B, 8082A, 8270C, 8270D, 8015D.)

Atmospheric Organic Parameters (EPA 3C, TO-15, TO-10A, TO-13A-SIM.)

Biological Tissue (Inorganic Parameters: SW-846 6020A. Organic Parameters: SW-846 8270C, 8270D, 3510C, 3570, 3610C, 3630C, 3640A)

New York Department of Health Certificate/Lab ID: 11627. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters: SM2320B, SM2540D, 6020A, 1631E, 7470A, 9050A, EPA 180.1, 3020A. Organic Parameters: EPA 8270C, 8270D, 8081B, 8082A, 3510C.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 6020A, 7471B, 7474, 9040C, 9045D, 9060A. Organic Parameters: EPA 8270C, 8270D, 8081B, 8082A, 1311, 3050B, 3580A, 3570, 3051A.)

Air & Emissions (EPA TO-15, TO-10A.)

Pennsylvania Certificate/Lab ID: 68-02089 ***NELAP Accredited***

Non-Potable Water (Inorganic Parameters: 1312, 1631E, 180.1, 3020A, 6020A, 7470A, 9040B, 9050A, 2320B, 2540D, 2540G, SM4500H+-B. Organic Parameters: 3510C, 3580A, 3630C, 3640A, 3660B, 3665A, 8015D, 8081B, 8082A, 8270C, 8270D .)

Solid & Hazardous Waste (Inorganic Parameters: EPA 1311, 3051A, 6020A, 7471B, 7474 9040B, 9045C, 9060. Organic Parameters: EPA3050B, 3540C, 3570, 3580A, 3630C, 3640A, 3660B, 3665A, 8270C, 8270D, 8081B, 8015D, 8082A.)

Rhode Island Department of Health Certificate/Lab ID: LAO00299. ***NELAP Accredited via NJ-DEP.***

Refer to NJ-DEP Certificate for Non-Potable Water.

Texas Commission of Environmental Quality Certificate/Lab ID: T104704419-08-TX. ***NELAP Accredited.***

Solid & Chemical Materials (Inorganic Parameters: EPA 6020, 7470, 7471, 1311, 9040, 9045, 9060. Organic Parameters: EPA 8015, 8270, 8081, 8082.)

Air (Organic Parameters: EPA TO-15)

Virginia Division of Consolidated Laboratory Services Certificate/Lab ID:460194. ***NELAP Accredited.***

Non-Potable Water (Inorganic Parameters:EPA 3020A, 6020A, 245.7, 9040B. Organic Parameters: EPA 3510C, 3640A, 3660B, 3665A, 8270C, 8270D, 8082A, 8081B, 8015D.)

Solid & Chemical Materials (Inorganic Parameters: EPA 6020A,7470A,7471B,9040B,9045C,3050B,3051, 9060. Organic Parameters: EPA 3540C, 3580A, 3630C, 3640A, 3660B, 3665A, 3570, 8270C, 8270D, 8081B, 8082A, 8015D.)

Washington State Department of Ecology Certificate/Lab ID: C954. *Non-Potable Water* (Inorganic Parameters: SM2540D, 180.1, 1631E.)

Solid & Chemical Materials (Inorganic Parameters: EPA 6020, 7470, 7471, 7474, 9045C, 9050A, 9060. Organic Parameters: EPA 8081, 8082, 8015, 8270.)

U.S. Army Corps of Engineers

Department of Defense, L-A-B Certificate/Lab ID: L2217.01.

Non-Potable Water (Inorganic Parameters: EPA 6020A, SM4500H-B. Organic Parameters: 3020A, 3510C, 8270C, 8270D, 8270C-ALK-PAH, 8270D-ALK-PAH, 8082A, 8081B, 8015D-SHC, 8015D.)

Solid & Hazardous Waste (Inorganic Parameters: EPA 1311, 3050B, 6020A, 7471A, 9045C, 9060, SM 2540G, ASTM D422-63. Organic Parameters: EPA 3580A, 3570, 3540C, 8270C, 8270D, 8270C-ALK-PAH, 8270D-ALK-PAH 8082A, 8081B, 8015D-SHC, 8015D.

Air & Emissions (EPA TO-15.)

Analytes Not Accredited by NELAP
Certification is not available by NELAP for the following analytes: **8270C:** Biphenyl. **TO-15:** Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 2-Methylnaphthalene, 1-Methylnaphthalene.

21 Appendix F – Alpha Code of Ethics Agreement

Alpha Analytical, Inc.
Ethical Conduct and Data Integrity Agreement

A. ***Personal Pledge:*** I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report data, test results and conclusions that are accurate, precise and of the highest quality.

B. ***Protocol Pledges:*** I agree to adhere to the following protocols and principles of ethical conduct in fulfilling my work assignments at Alpha:

1. All work assigned to me will be performed using Standard Operating Procedures (SOPs) that are based on EPA approved methods or Alpha methods.

2. I will only report results or data that match the actual results observed or measured.

3. I will not intentionally nor improperly manipulate or falsify data in any manner, including both sample and QC data. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented in the appropriate laboratory notebooks and raw data and include my initials or signature and date.

4. I will not intentionally report dates and times of analyses that are not the actual dates and times the analyses were conducted.

5. I will not intentionally represent another individual's work as my own or represent my work as someone else's.

6. I will not make false statements to, or seek to otherwise deceive Alpha staff, leaders or customers. I will not, through acts of commission, omission, erasure or destruction, improperly report measurements, standards results, data, test results or conclusions.

C. ***Guardian Pledge:***

1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Technical Manager or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.

2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha's QA Officer, senior leadership or corporate officers, including the President of the company.

3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or improper. Also, I will not discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. ***Agreement Signature:*** I have read and fully understand all provisions of the *Alpha Analytical Ethical Conduct and Data Integrity Agreement*. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

_____
Employee Signature

_____
Printed Name

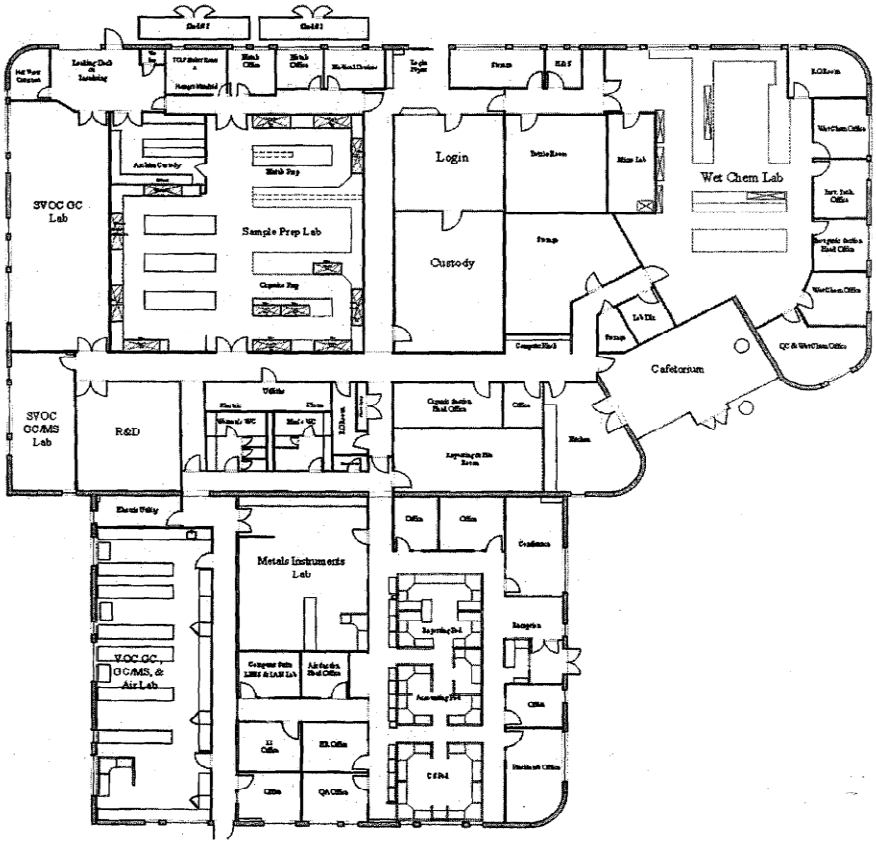
_____
Date

Review Requirements
The *Ethical Conduct and Data Integrity Agreement* must be signed at the time of hire (or within 2 weeks of a staff member's receipt of this policy). Furthermore, each staff member will be required to review and sign this agreement every year. Such signature is a condition of continued employment at Alpha. Failure to comply with these requirements will result in immediate discharge from Alpha employment. This agreement is not an employment contract and does not modify in any manner the company's *Employment-at-Will* Agreement.

22 Appendix G – Floor Plan Westboro Facility

Alpha Woods Hole Labs

FIGURE II
FLOOR PLAN



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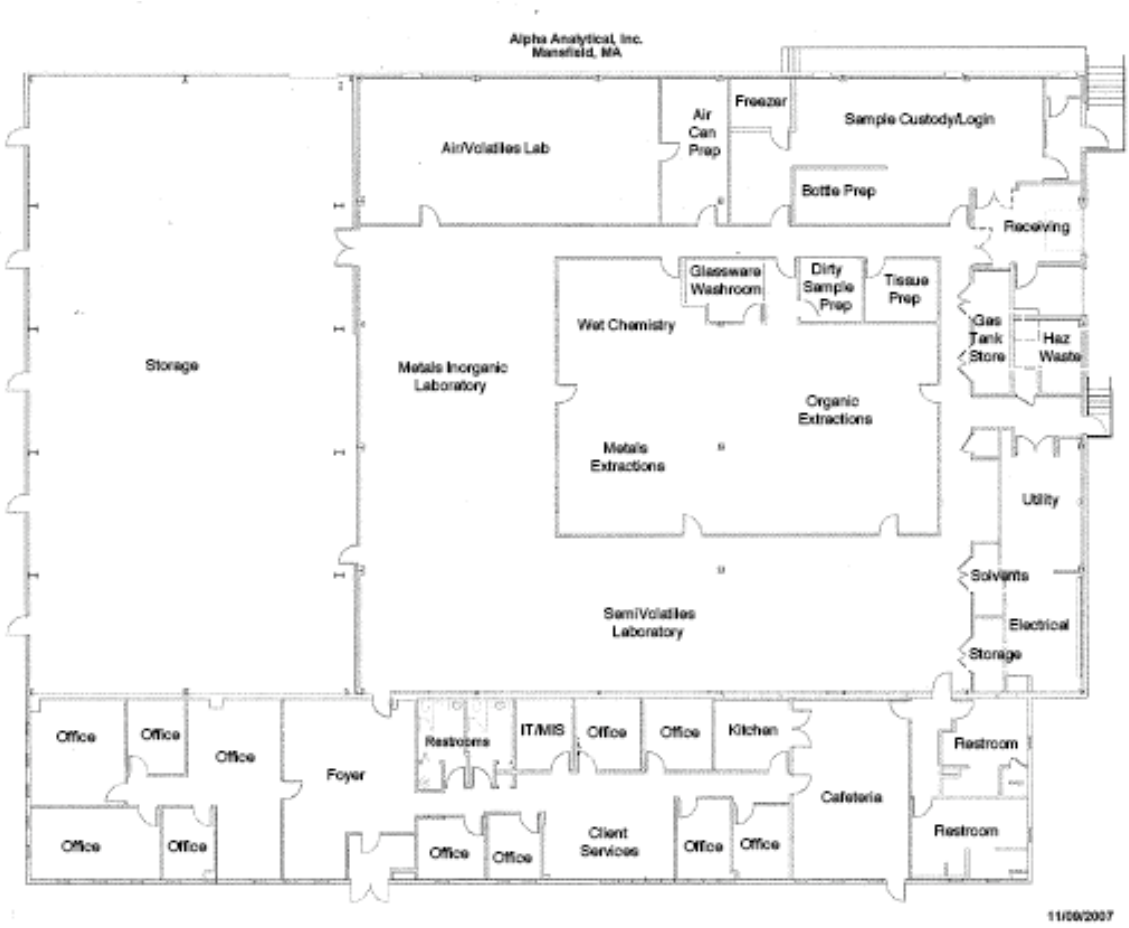
23 Appendix H– Floor Plan Mansfield Facility



24 Appendix I – Job Titles and Requirements

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Technical Manager (Director) Organic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 24 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be susbsituted for 1 year of experience.	Two (2) years with the analysis of organic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Inorganic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of inorganic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Microbiology Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in the Biological Sciences, including at least one course having microbiology as a major component. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of microbiological analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Quality Assurance Officer	BS/BA in Chemistry, Biology, Environmental or related Science	Two (2) years Environmental Laboratory Experience	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Knowledgeable in Federal, State and DOD Programs (NELAC, etc.) 3. Able to develop QA/QC policies and certification requirements 4. Able to develop training programs for quality procedures 5. Documented training and/or experience in QA and QA procedures 6. Knowledge of safe laboratory practices and emergency protocols

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Laboratory Coordinator	High School Diploma; Associates or BS/BA in Chemistry, Biology or Environmental or related Science preferred	1 year +	1. Knowledge of safe laboratory practices and emergency protocols 2. Proficient in all methods and SOP's within their department 3. Experience with and understanding of LIMS 4. Proven ability to meet TAT (turn around times)
Quality Systems Specialist	BS/BA Chemistry	2 years +	1. General knowledge of laboratory methods 2. Experience with and understanding of LIMS 3. Strong attention to detail 4. Strong oral/written communication and organizational skills 5. Knowledge of QA/QC policies and certification requirements
EH&S Coordinator	High School or Equivalent	2 years +	1. General knowledge of lab operations 2. Detailed knowledge of safe lab practices and emergency protocols 3. Hazardous Waste Management and RCRA Regulation Training 4. DOT Hazardous Materials Regulations Training 5. OSHA Compliance Training 6. Able to develop and deliver new hire and ongoing safety training programs
Lab Technician I	HS or Equivalent	0-1 years. 1+ years preferred.	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician II	HS or Equivalent	2-4 years	1. All skills of Lab Technician I 2. Trained in majority of technician skills relative to department
Lab Technician III	HS or Equivalent	5 years +	1. All skills of Lab Technician II 2. Experienced in training staff
Lab Technician/Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician/Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Chemist I 2. Trained in majority of department methods

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Lab Technician/Chemist III	BS/BA in Chemistry, Biology, Environmental or related Science	5 years +	1. All skills of Chemist II 2. Experienced in training staff
Analyst I	HS or Equivalent	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analyst II	HS or Equivalent	2-4 years	1. All skills of Analyst I 2. Experienced in machine operation, maintenance and troubleshooting
Analyst III	HS or Equivalent	5 years +	1. All skills of Analyst II 2. Experienced in data review and reporting 3. Experienced in training staff
Analytical Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	6 mos-1 year	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analytical Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Analytical Chemist I 2. Experienced in machine operation, maintenance and troubleshooting
Analytical Chemist III	BS/BA in Chemistry, Biology, or Environmental or related Science	5 years +	1. All skills of Analytical Chemist II 2. Experienced in data review and reporting 3. Experienced in training staff
Data Deliverable Specialist I	HS Diploma, BS/BA or Associates preferred	0-1 years	1. Introductory knowledge of laboratory methods 2.Able to follow direction and Standard Operating Procedures (SOP's) 3. Working knowledge of Adobe Acrobat, Microsoft Word, Excel 4. Good writing and typing skills
Data Deliverable Specialist II	HS Diploma, BS/BA or Associates preferred	2-4 years	1. All skills of Data Deliverable Specialist I 2. General knowledge of laboratory methods 3. Understanding of data review/ data reporting process 4. Experience with and understanding of LIMS and electronic data deliverables

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Data Deliverable Specialist III	HS Diploma, BS/BA or Associates preferred	5 years +	1. All skills of Data Deliverable Specialist II 2. Intermediate/advanced knowledge of laboratory methods 3. Able to perform report review 4. Experience with and understanding of LIMS and electronic data deliverables 5. Able to initiate re-work where necessary
Laboratory Intern	2 Semesters of Chemistry, Biology or Environmental Science	None; Lab work study experience preferred	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures

KEY

* Internal terms only. Full title would have "Environmental Laboratory" and specific department preceding it.

** Substitutions: Equivalent knowledge may be substituted for a degree in some instances.

*** Not meant to be an exhaustive list of skill requirements. For full list of skills consult the "Laboratory Skills" list. Actual Job Duties and Responsibilities can be found within job descriptions for each position.

25 Appendix J – Standard Operating Procedures

WESTBORO SOP #	Title
1723	Customer Service
1724	Quote/Contract Procedure
1725	Project Communication Form Generation
1727	Accounts Payable Invoice Processing
1728	Waste Management and Disposal
1730	Balance Calibration Check
1733	Thermometer Calibration
1735	Analytical Guidelines for Method Validation
1737	Inorganics Glassware Cleaning and Handling
1738	Water Quality Monitoring
1745	Reagent, Solvent and Standard Control
1747	Datalogger Operation
1948	Separatory Funnel Liquid-Liquid Extraction – EPA 3510C
1953	Organic Extraction Glassware Cleaning & Handling
1954	Soxhlet Extraction – EPA 3540C
1955	Sulfur Cleanup – EPA 3660A
1956	Oil and Waste Dilution – EPA 3580A
1959	Microwave Extraction – EPA 3546
1960	Sulfuric Acid Cleanup – EPA 3665A
1962	Florisil Cleanup
1963	Fractionation Cleanup
1964	Preparation of Samples for Chlorinated Herbicides
2022	Volatile Organic Compounds – EPA 624
2107	Volatile Organic Compounds – EPA 524.2
2108	Volatile Organic Compounds – EPA 8260C
2109	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM – EPA 8270D (modified)
2110	Semivolatile Organics by GC/MS – EPA 625

2111	Semivolatile Organics by GC/MS – EPA 8270D
2112	TCLP/SPLP Extraction - Volatile Organics SW-846 Method 1311/1312
2113	EDB & DBCP in Water by Microextraction & Gas Chromatography – EPA 504.1, 8011
2116	Organochlorine Pesticides by Capillary Column GC – EPA 8081B
2119	Extractable Petroleum Hydrocarbons – MADEP
2120	Volatile Petroleum Hydrocarbons – MADEP
2122	Organochlorine Pesticides & PCBs by Capillary Column GC – EPA 608
2123	Polychlorinated Biphenyls in Oil – EPA 600/4-81-045
2125	TPH-Diesel Range Organics, Maine 4.1.25, EPA 8015C (Modified)
2126	TPH- Gasoline Range Organics, Maine 4.2.17, EPA 8015C (Modified)
2127	CT-ETPH
2128	Herbicides by 8151A
2129	PCBs by Capillary Column Gas Chromatography - EPA 8082A
2131	New Jersey EPH Method
2132	Microwave Assisted Acid Digestion of Aqueous Samples & Extracts – EPA 3015
2133	TCLP Extraction Metals and Semi-Volatile Organics – SW-846 Method 1311
2134	Hot Block Digestion for Aqueous Samples EPA 3005A
2135	SPLP Extraction Inorganics and Semivolatile Organics, EPA 1312
2136	Hot Plate Digestion of Sediments, Sludges and Soils, EPA 3050B
2144	Metals by Inductively Coupled Plasma – EPA 6010C
2145	Mercury in Liquid Waste by Cold-Vapor Atomic Absorption – EPA 7470A
2146	Mercury in Soil or Solid Waste by Cold-Vapor AA – EPA 7471B
2149	Metals by Inductively Coupled Plasma – EPA 200.7
2153	Mercury in Water by Automated Cold-Vapor Atomic Absorption – EPA 245.1
2156	Metals by Inductively Coupled Plasma-Mass Spectrometry – EPA 6020A
2159	Metals by Inductively Coupled Plasma-Mass Spectrometry – EPA 200.8
2161	Fecal Coliform by Membrane Filtration – SM 9222D
2163	Fecal Coliform by Multiple Tube Fermentation – SM 9221E
2191	Heterotrophic Plate Count – SM 9215B
2192	Total Coliform/E.Coli – Presence/Absence (Colilert) – SM 9223B

2193	Total Coliform by Membrane Filtration – SM 9222B
2194	Total Coliform by Multiple Tube Fermentation – SM 9221B
2195	Chlorophyll A – SM 10200H
2196	E. Coli – Membrane Filtration
2197	Chlorophyll A – EPA 446
2198	Air Density Monitoring
2199	Inhibitory Residue Test
2200	Enterococcus – MF
2201	Total Coliform, E.Coli & Enterococcus by Quantification Methods (Quanti Tray)
2202	pH, Liquid Samples – EPA 9040C, SM 4500H^+-B
2203	pH, Soil & Waste Samples – EPA 9045D
2204	Hexavalent Chromium – EPA 7196A, SM 3500Cr-D
2205	Biological Oxygen Demand – SM 5210B
2206	Ammonia Nitrogen –SM 4500NH_3-BH, Lachat 10-107-06-1-A
2207	Total Kjeldahl Nitrogen – SM 4500N_{org}-C, Lachat 10-107-06-2-D
2208	Chemical Oxygen Demand – SM 5220D
2209	Oil & Grease by n-Hexane Extraction Method & Gravimetry – EPA 1664A
2210	Cyanide, Total – EPA 9010B, SM 4500CN-CE
2211	Phenol, Total - EPA 420.1, EPA 9065, SM 510AC
2212	Sulfate, Turbidimetric Method – EPA 9038, SM 426C, SM 4500SO_4-E
2213	Alkalinity, Titration Method –SM 2320B
2214	Determination of Inorganic Anions by Ion Chromatography – EPA 300.0
2215	Total Organic Carbon/Dissolved Organic Carbon – EPA 9060, SM 5310C
2216	Chloride – SM 4500Cl-E, EPA 9251
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen – EPA 353.2, SM 4500NO_3-F
2218	Total Solids (Dried @ 103-105^o) and TVS – SM 2540B, SM 2540E
2219	Total Dissolved Solids – SM 2540C
2220	Total Suspended Solids – SM 2540D
2221	Total Sulfide – SM 4500S2-AD, EPA 9030B
2222	MBAS, Anionic Surfactants – SM 5540C
2223	Fluoride, Electrode Method – SM 4500F-BC

2224	Turbidity, Nephelometric Method – EPA 180.1, SM 2130B
2225	Orthophosphate, Colorimetric Single Reagent Method – SM 4500P-E
2226	Total Phosphorous, Colorimetric Single Reagent Method – SM 4500P-E
2227	Flashpoint – EPA 1010
2228	Reactivity – EPA Chapter 7.3
2229	Total Solids (Dried @ 103-105^{o}) – SM 2540G
2230	Specific Conductance and Salinity
2231	True and Apparent Color, Visual Comparison Method
2232	Acidity, Titration Method
2233	Determination of Formaldehyde by HPLC, EPA 8315A
2234	Sulfite, Iodometric
2235	Ferrous Iron
2236	Residual Chlorine
2237	ORP
2238	Ignitability of Solids EPA 1030
2239	Physiologically Available Cyanide (PAC)
2240	Total Settleable Solids SM 2540 F
2241	Fixed and Volatile Solids in Solid and Semisolid Samples – SM 2540G
2242	Tannin & Lignin
2243	Nitrite - Manual Colorimetric Method
2244	Paint Filter Liquids Test
2245	Odor, Threshold Odor Test
2249	Dissolved Oxygen
2250	Nitroaromatics and Nitramines by HPLC, EPA 8330A
2251	Perchlorate by IC/MS/MS
2274	Data Validation Package Generation
3743	Free Cyanide
9177	Total Phenol - SEAL Method
9733	Oil & Grease and TPH in Soil
10807	Percent Organic Matter in Soil

MANSFIELD SOP #	Title
1753	Glassware Cleaning
1754	Balance Calibration
1755	Pipette Checks
1796	Sample Management - Forensics
1797	Haz Waste
1816	Reagent Solvent Std Control
2137	6020
2138	Hg 245.1, 7470A
2139	Hg soil 7471
2140	AVS SEM
2141	Hydride Generation
2142	Hg Liq 1631E
2143	Hg soil 7474
2148	Metals soil digest 3050
2150	Metals Microwave 3015 3015
2151	Metals Acid Digest 3010/3020
2152	Seawater Extr
2154	TCLP 1311
2155	8270C
2157	PAH by SIM
2158	8081
2160	8082 Aroclors/Cong GC
2162	Pest/PCB Aro/Cong GC/MS SIM
2164	1,4-Dioxane GC/MS SIM
2165	Sep Funnel 3510C
2166	Tissue Prep
2167	GPC
2168	Sulfur Cleanup 3660
2169	Sulfuric Acid Cleanup 3665

2170	Silica Gel Cleanup
2171	% Lipids
2172	MSE
2173	Soxhlet
2174	Soxhlet of PUFs
2175	% Solids
2176	Conductivity
2178	pH in Aqueous and Soil
2179	TSS
2180	Turbidity
2181	Alkalinity
2182	TOC soot-soil
2183	Particle Size
2184	Particlulates in Air PM-10
2185	Volatile Solids
2186	TO-15
2187	APH
2188	Air PIANO
2189	Dissolved Gases
2190	Can Cleaning
2246	TPH and SHC
2247	Alkylated PAH
2248	Organic Lead
2252	Fixed Gases new system
2253	TO-11A
2255	PIANO Volatiles
2256	Ethanol in Oil
2257	Whole Oil Analysis
2259	Density Determination
2260	Alumina Cleanup
2261	Shaker Table

2263	Gravimetric Determ
2264	Tissue Extr
2265	Organic Waste Dilution
2267	Newfields SGC
2268	Newfields DCM
4246	PAHs by SPME
6398	TO-17
6438	Mercury in Sorbent Tubes by CVAA
7900	Mercury 1631E Using Cetac-M-8000 Analyzer
9480	EPA-TO-12
9745	Formaldehyde - HPLC
10480	Nitroaromatics and Nitramines by HPLC
CORPORATE SOP #	**Title**
1559	Sample Receipt & Login
1560	Sample Custody and Tracking
1561	Bottle Order Preparation
1729	Document Control
1731	Manual Integration
1732	DL LOD LOQ
1734	Control Limit Generation
1736	Corrective and Preventative Actions
1739	Demonstration of Capability (DOC) Generation
1740	Internal Audit Procedure
1741	Data Review – Organics
1742	Calculating Measurement Uncertainty
1743	Annual Management Review
1744	Sample Compositing Procedure
1746	Nonconformance Planning/Procedures
1566	Report Generation and Approval

1567	Organics Data Deliverable Package Review
1722	Customer Inquiry and Complaint Procedures
1562	Computer System Backup/Control
1563	Computer and Network Security
1564	Software Validation and Control
1726	Purchasing Procedure
1565	Training Program

CON-TEST ANALYTICAL LABORATORY

QUALITY ASSURANCE MANUAL

39 Spruce Street
East Longmeadow, Massachusetts
(413) 525-2332

Tod Kopyscinski
Laboratory Technical Director

03/03/2017
Review/Approval Date

Katherine F. Allen
Quality Assurance Officer

03/03/2017
Review/Approval Date

Revision Number: 25

Controlled Copy Number: Non-Controlled Copy

Revision Record

Revision	Date	Responsible Person	Description of Change
1	1/1/2000	Sondra S. Kocot	Initial Version
2	7/16/2000	Sondra S. Kocot	Update App. A, delete distribution table and replace with distribution statement only; Update Equipment List: General Release
3	3/22/2002	Sondra S. Kocot	Update Method List, Equipment List, and Organizational Chart (Appendix A)
3A	11/21/2002	Sondra S. Kocot	Update Method List and Organizational Chart (Appendix A)
4	10/02/2003	Sondra S. Kocot	Update Organizational Chart (Appendix A); Statement for "Lab Ethics in Data Manipulation"; Statement for "Samples/Reports Involved in Litigation", change in storage-time for non-metal waters; update Equipment List
5	04/14/2004	Sondra S. Kocot	Update accreditation (section 3.3.4.1); add AZ office address (Intro. Section)
6	05/10/2004	Sondra S. Kocot	Updates for compliance with MA DEP Microbiology Audit and AZ Audit; additions affect primarily sections 4.0 & 7.0, with the addition of the Chem. Hygiene Plan as an Appendix, Org. chart also updated.
7	10/08/2004	Sondra S. Kocot	Updates include: Organizational chart, Equipment List, Metals Training, Uncertainty Statement (section 3)
8	02/21/2005	Sondra S. Kocot	Updates include: Organizational chart, EPA reference (200.7, 40 CFR Part 136 App C) added for non-potable ICP water samples
9	03/22/2005	Sondra S. Kocot	Edit MDL study paragraph to include discussion of outliers; update equipment list
10	07/19/2005	Sondra S. Kocot	Updates for compliance with June 2005 AIHA-LAP, LLC Audit: Organizational chart (App A), sections 3.2.1, 3.3.4.4, 9.2.4, and 13.0.
11	05/24/2007	Edward J. Denson/ Sondra Slesinski	Annual Updates
12	10/22/2007 , 12/05/2007	Edward J. Denson/ Sondra Slesinski	Updates per Oct 2007 AIHA-LAP, LLC audit and Nov 2007 client audit: See next page for detailed change record
13	07/14/2008	Katherine F. Delisle	Updates per recommendation of Massachusetts, to include new methods. See detailed change record.
14	03/25/2009	Katherine F. Delisle	Updates per changes in policy for Eppendorf's and MDLs. See detailed change record.
15	01/11/2010	Katherine F. Allen	Updates from July 2009 AIHA-LAP, LLC audit and MA June 2009 audit. See detailed change record.
16	06/23/2010	Katherine F. Allen	Updates from January 2010 NJ audit. App D was removed and made a controlled document #252. Updates to Sec's 3.2.1, 3.3.3.1, 4.1.2 (method blanks), and 4.2 (calibration). Section 4.2 (last 2 paragraphs deleted), and Section 11.0 (equipment list updated).
17	04/05/2011	Katherine F. Allen	Updates from Annual Review. See detailed change record.
18	10/07/2011	Katherine F. Allen	Updates from September 2011 AIHA-LAP, LLC audit. QA manual reworked.
19	08/20/2012	Katherine F. Allen	Updates from June 2012 NJ audit. See detailed change record.
20	10/15/2012	Katherine F. Allen	Updates from Sept 2012 NH audit. See detailed change record.
21	08/14/2013	Katherine F. Allen	Updates from June 2013 NY and MA Audits. See detailed change record.
22	09/03/2013	Katherine F. Allen	Updates from August AIHA-LAP, LLC audit. See detailed change record.

23	04/09/2015	Katherine F. Allen	Updates from annual review: See detailed change record.
24	10/20/2015	Katherine F. Allen	Updates from Sept 2015 AIHA-LAP, LLC audit. See detailed change record.
25	03/03/2017	Katherine F. Allen	Updates from annual SOP review: See detailed change record.

Revision 12/12a **Detailed Change Record**

Introduction	Reference to "industrial hygiene" as well as "environmental"
Section 1.0	Reference to "compliance with all accrediting authorities, including ISO/IEC 17025"
Section 1.3.4	Added reference to accrediting authorities
Section 2.0	For job descriptions of Technical Director, QA Officer, Supervisor, and Analyst, academic and experience qualifications were added.
Section 2.1	Commitment of management to the QA policy statement and for improvements in the management system.
Section 2.2.2.4	"Ensures compliance with all accrediting authorities and organizations (AIHA-LAP, LLC – ISO/IEC 17025, NELAP, and various states)"
Section 3.3.4	Added North Carolina certification
Section 3.3.4 (12/07 edit)	Added Florida certification
Section 3.3.4.2	Internal PT program for AIHA-LAP, LLC fields of testing not covered by AIHA proficiency studies
Section 3.3.4.3	Written pre-approval for subcontracting needed; Con-Test is not responsible for the work of subcontract lab's which the client specifies that we use
Section 4.2.1.2	AIHA-LAP, LLC IHLAP RL's verified per matrix in each batch
Section 4.4 (12/07 edit)	Added ICP-MS method 6020 maintenance
Section 4.11	Procurement policy added
Section 4.12	Include a statement that lots of IH media are tested to ensure no contamination, and that records of such tests are maintained by each department; also stated is that Con-Test supplies IH sampling media to the clients, who perform the sampling.
Section 6.2.2 (12/07 edit)	Use of Infra-Red gun is specified regarding sample temperature
Section 6.2.3	Assignment of laboratory numbers: "environmental" samples changed to "all" samples
Section 6.2.5	Sample storage: A locked storage area will be provided should the client require secure storage for samples which require special handling due to legal proceedings.
Section 9.0	Addition of "management review"
Sections 9.0, 9.3	Section for "Corrective Actions/Preventative Actions" was added
Section 9.2	Internal audit (per AIHA-LAP, LLC, ISO/IEC 17025 requirements) must be conducted annually
Section 10.0 (12/07 edit)	Add TOC to analytical method list
Section 11.0(12/07 edit)	Update equipment list
Section 12.0 (12/07 edit)	TCLP sampling for VOA & metals: the verbiage for preservation with acid was deleted.
Section 14.2	AIHA-LAP, LLC IHLAP/ELLAP trainees must have a training period of 20 business day's duration, prior to completing a DOC and working independently on client samples. This 20-day period must be clearly documented on the IDOC training form.
Appendix A	Updated Organizational Chart
Appendix B	Addition of ISO/IEC 17025:2005
Appendix C	Edited "Training/IDOC" form to include "authorization" date, and specified 20-business-day training duration for AIHA-LAP, LLC IHLAP/ELLAP

Revision 13 **Detailed Change Record**

Section 3.3.3.1	Include proper use of QC trends, including monitoring for presence of trends indicating that an analysis could be heading towards "out-of-control" situation.
Section 4.2	Edit to include calibration frequency of reference weights, reference thermometers, and analytical balances.

Section 4.4	Edit to include annual calibration of Conductivity meter and bi-annual calibration of Infra-Red thermometer gun.
Section 6.2.2	Edit to include bi-annual calibration of Infra-Red thermometer gun.
Section 8.5	New section, including a list of Standard Operating Procedures (SOP's). See Appendix E.
Section 10.0	Addition of ICP-MS methods, ICP method, and mercury method. Updating EPA reference to include EPA/600R-94-11, May 2004.
Section 11.0	Deleted Bausch & Lomb 601 spectrophotometer in equipment listing section.
Section 13.1	MCL exceedance policy
Appendix A	Updated Organizational chart
Appendix E	New appendix to include listing of Standard Operating Procedures (SOPs).

Revision 14 **Detailed Change Record**

Section 3.3.4.3	Sub-contracting lab policy addition
Section 4.2	Eppendorf calibration frequency
Section 4.3	Eppendorf calibration frequency
Section 4.4	Eppendorf calibration frequency
Section 4.10	MDL policy for frequency
Section 11.0	Equipment section updated to include new Mercury Instrument, Beckman Centrifuge, flashpoint apparatus, and ENCON evaporation system.
Appendix A	Updated Organizational chart
Appendix E	Updated Listing of SOP's

Revision 15 **Detailed Change Record**

Section 3.3.3.3.1	Addition of Non-Conforming work policy
Section 3.3.4	Inclusion of WA state certification for EPH and VPH
Section 6.2.3	Change in how laboratory numbers are assigned
Section 7.6	Change to Data Storage in respect to (new LIMS) Element
Section 15.1	Addition of communication to the subcontracting lab of special report requirements
Appendix A	Updated Organizational chart
Appendix C	Updated IDOC form
Appendix E	Updated Listing of SOP's

Revision 16 **Detailed Change Record**

Section 9.2.2	Internal method audit section added
Appendix D	Appendix D was removed and made a controlled document #252
Section 3.2	Section updated for typos and changes in verbiage
Section 3.3.3.1	Section updated for changes in verbiage
Section 4.1.2	Method blank section updated for changes in verbiage
Section 4.2	Calibration section updated for changes in verbiage. Last 2 paragraphs removed from SOP
Section 11.0	Equipment List updated
Appendix A	Updated Organizational chart
Section 10.0	Addition of Herbicide Method SW-846 8151A

Revision 17 **Detailed Change Record**

Section 2.2 and 2.4	Deputies were added in absence of the Technical Director and QA Officer
Section 4.2, 4.3, and 4.4	Eppendorf calibration frequency change
Section 6.2.2	Sample Acceptance Policy added
Section 10.0	21st edition of Standard Methods added to reference section. Method SW-846 6010B changed to SW-846 6010C. Flame and Furnace deleted. SW-846 8015B switched to SW-846 8015C and addition of SW-846 8270D and SW-846 8260C.

Appendix A	Updated Organizational chart
Appendix E	Updated Listing of SOP's

Revision 18 Detailed Change Record

QA Manual Retyped and Reformatted

Section 1.0	Objectives added
Section 2.2	Added Project Chemists are under Technical Director
Section 2.3	Deletion of Customer Services Manager under Administration Manager
Section 2.4	Used to be section 13.0: Addition of QA reports to management and monthly meeting with Technical Director.
Section 2.5	Addition of Laboratory Manager
Section 2.10	Used to be Appendix "A": Organization Chart and org chart updated
Section 3.2.1	Addition to Estimation of Uncertainty of Measurements and reference to the new SOP "Estimation of Uncertainty of Measurements"
Section 3.2.2.6	Addition of nonconforming work being immediately evaluated and "Customers notified and work is recalled when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its procedures or the agreed requirements of the customer".
Section 3.2.2.7	Addition of Corrective actions and root cause investigations will be immediately issued.
Section 3.3.3	Addition of Control limits calculated annually with at least 20 data points.
Section 3.3.3.1	Addition of Control Charts assessed monthly.
Section 3.3.3.3	Addition of "Root Cause" investigation.
Section 3.3.3.3.1	Addition of Customers notified and work is recalled when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its procedures or the agreed requirements of the customer, Corrective Action taken immediately, and deviations that result in nonconforming work shall be immediately evaluated.
Section 3.3.4.2	In-house AIHA-LAP, LLC PT's run twice annually instead of quarterly. Addition of a blank sample as well as 4 varying samples. Addition of unacceptable PT results immediately initiate a corrective action and a root cause investigation will begin. Addition of blind samples are made up and spiked by either the department Supervisor (Technical Manager) or the QA Officer.
Section 3.3.4.3	Included MCL exceedances must be reported by sub-lab within 24 hours.
Section 4.2	Addition of calibration certificates from external services must be accredited to ISO/IEC 17025:2005 by a recognized accrediting body. And Addition of Refer to Manufacturer's instructions for procedures on how to transport and store measuring equipment and reference standards. Addition of documented training for staff doing in house calibrations and verifications.
Section 4.2.1.1	Reporting Limits are not less than the lowest calibration standard.
Section 4.3	Addition of "For Equipment, Reference Standards, and Reference Materials is transported, stored, maintained, inspected, and cleaned according to manufacturer's instructions" and External Services for calibration of weights, NIST Thermometers, and Eppendorf's must be accredited to ISO/IEC 17025:2005 by a recognized accreditation body.
Section 4.3.1	New Section to include Equipment List which used to be Section 11.0. Equipment listing updated.
Section 4.10	MDL spiked reagent water changed to spiked media and addition of wipe material criteria.
Section 4.10.1	New Reporting Limit section
Section 4.13	Addition of "refer to manufacturer's instructions for the procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment, reference materials, and reference standards to ensure proper functioning and in order to prevent contamination or deterioration".
Section 4.14	Addition of level of acceptable contamination for lead wipe sampling defined and corrective action performed if above this level.
Section 4.16	Section renamed Review of Requests, Tenders, and Contracts. Additional detail provided along with reference to SOP Review of Requests, Tenders, and Contracts, Doc #290.
Section 6.2.2	Addition of samples checked by log-in staff
Section 7.6	Data storage procedural change

Section 8.0 and 8.1	Addition of "all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work".
Section 8.5	Used to be Appendix "E": SOP listing and SOP listing Updated
Section 9.0	Addition of review of overall objectives to the management review.
Section 9.3	Addition of an Outline for Corrective/Preventative Actions and reference to the CA/PA SOP.
Sections 9.3.1, 9.3.2, 9.3.3, and 9.3.4	Additional details provided on the corrective/preventative action program.
Section 10.0	Addition of statement, "Deviations from test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client" as well as will be noted on the final report.
Section 12.2	Addition to training section: "All personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work".
Section 12.6	Used to be Appendix "C": Demonstration of Capability
Section 13.1	Addition of, "Clients notified and work is recalled when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its procedures or the agreed requirements of the client" and "Deviations from test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client"
Section 13.3	Corrective actions initiated immediately if warranted from client inquiry and reference of Corrective action section 9.3 in QA Manual and CA/PA SOP.
Section 14.0	Used to be Appendix "B": References
Revision 19	**Detailed Change Record**
Section 2.10	Updated Organizational chart
Section 3.3.4.1	Addition of Virginia and Maine Certifications
Section 8.3	Addition of written explanations for rerun samples and standards as well as anything that might need explanation in the future.
Section 8.5	Updated SOP listing
Revision 20	**Detailed Change Record**
Section 2.10	Updated Organizational chart
Section 5.2	Formulas for automated computations are initially verified then locked.
Section 8.5	Updated SOP listing
Revision 21	**Detailed Change Record**
Capabilities	Rephrasing
Section 2.10	Updated Organization chart
Section 4.3.1	Updated Equipment Listing
Section 4.3 and 4.4	IR Temperature guns calibrated quarterly
Section 7.0 and 7.3	Addition of items included on reports
Section 8.5	Updated SOP listing
Section 9.0	Managerial reviews identify author and be paginated.
Revision 22	**Detailed Change Record**
Section 2.10	Updated Organizational Chart
Section 4.3.1	Updated Equipment listing
Section 8.5	Updated SOP listing
Section 9.2	Addition of "latest" AIHA-LAP, LLC and NELAC site assessor's checklist to be used

Revision 23	**Detailed Change Record**
Section 2.0	Chemical Hygiene Officer (safety officer) added
Section 2.10	organizational chart removed and note added referring to external document.
Section 2.2	Updated Lab Technical Director description
Section 3.3.4.2	More information provided on proficiency samples
Section 4.2	NIST thermometer and weights calibrated every year
Section 4.12	Note added that if all media is purchased then the table of tests are not needed.
Section 4.3.1	Updated Equipment Listing
Section 6.2.2	Infrared temperature gun verified quarterly
Section 7.6	Third paragraph removed from data storage section and added that Lead and Copper potable water records need to be kept for a period of 12 years.
Section 8.0	Added other documents to master list of controlled documents
Section 8.1	Added that SOP's and QAM are available to personnel on F: Drive.
Section 8.5	Revision and date of review of each SOP removed and note added stating, for current revision and date of review see master list of controlled documents maintained by the QA department and available upon request.

Revision 24	**Detailed Change Record**
Section 3.3.3.1	Addition of lead control limit requirement
Section 4.2	NIST long stem thermometers purchased annually and digital sent out for calibration annually.
Section 4.2.2	Second Source standard traceable to ISO 17025 and ISO Guide 34.
Section 4.2.3	Standard traceable to ISO 17025 and ISO Guide 34.
Section 4.3.1	Equipment update
Section 4.11	Additional details of procurement added
Section 7.7	Reference to Records Maintenance Matrix added.

Revision 25	**Detailed Change Record**
Section 3.2.2	Additional statements added regarding free from undue pressures
Section 3.3.4.1	Deletion of WA certification
Section 4.3.1	Updated Equipment listing
Section 7.1	Addition of significant figures
Section 7.6	Updated server name to be SQL2014PRI.
Section 8.0	Additional comments added as to what is found on each document and list of SOPs updated to include any new SOP. Updated SOP listing.
Section 9.3.4	Added an internal audit may be necessary
Section 10.0	Addition of methods: EPA 537, ISO 25101, SM 5310B; EPA 300.0, 6010D, 6020B, 7303, 5503 and 6009 and deletion of some methods: SM5310C, 7300, NIOSH 1501, 1003, 7600, 3500, 1550 and 5026
Section 11.0	Updated preservation section
Section 13.1	Expanded MCL section to include MA 310 CMR42.13 requirements

Distribution/Training List

See Employee Training Record File for signed training statements for trained user

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CON-TEST Analytical Laboratory

Location of Facility

Con-Test Analytical Laboratory, a full-service facility is located at 39 Spruce Street, East Longmeadow, Massachusetts 01028. The laboratory is easily accessible from both CT Interstate I-91 and I-90 (Massachusetts Turnpike).

Brief Company History

Con-Test celebrated its thirtieth year in 2014. Con-Test was started in 1984 as a consulting and engineering firm with laboratory services and in 1994 the company was sold. In 1996 the laboratory was bought back and became strictly a family owned, independent laboratory.

We are proud to have established a reputation based on ***quality, integrity, and reliability*** within the environmental field. Initially, laboratory testing was limited to industrial hygiene analysis mainly in support of in-house consulting services. But the laboratory rapidly expanded its capabilities to include numerous techniques in air analysis, classical (wet) chemistry, metals, and organics.

Con-Test is presently a privately owned, independent laboratory which provides environmental and industrial hygiene analytical services with AIHA-LAP, LLC IHLAP and AIHA-LAP, LLC ELLAP (Environmental Lead) accreditation. Continuing to update our accreditations and technology, we also attained the nationally recognized NELAP accreditation and certification. We are also individually certified in many areas and states by a diverse group of recognized organizations and we have consistently demonstrated proficiency in numerous analyses and matrices under established programs.

Capabilities

The laboratory has the capability for water, air, soil or solid matrices, and lead in soil, air, wipes and paint. The laboratory currently serves a diverse range of clients in an even broader range of analytical services. Analyses are performed to satisfy the following regulatory requirements and purposes:

- National Polluant Discharge Elimination System (NPDES)
- Industrial Pretreatment Program (IPP)
- Resource Conservation and Recovery Act (RCRA)
- EPA Requirements
- OSHA Compliance Requirements
- Code of Federal Regulations (CFR) Requirements
- Massachusetts Department of Environmental Protection (DEP)
- Safe Drinking Water Act (SDWA)
- Clean Water Act
- Massachusetts Water Resources Authority (MWRA)
- Hazardous Waste Characterization (SW-846)
- Groundwater Monitoring Programs

- Industrial Hygiene/Indoor Air Quality (AIHA-LAP, LLC)
- Microbiology
- Well Water Testing
- State Certifications (MA, CT, NY, VT, RI, NH, NJ, NC, ME, VA and FL)
- Connecticut RCP (Reasonable Confidence Protocols)
- Massachusetts MCP (Massachusetts Contingency Plan)

Con-Test Analytical Laboratory is an established laboratory, which realizes the need for remaining on the cutting edge of environmental/industrial hygiene technology. Automation of systems to the greatest extent possible is a primary objective of the laboratory. Current applications and systems are continually being expanded and updated whenever possible to achieve unrivaled quality and information turnaround. Con-Test believes that the use of state of the art instrumentation, including data management systems is imperative in maintaining needed efficiency and effectiveness of services. The laboratory is equipped with the latest instrumentation including Gas Chromatographs (GC), GC Mass Spectrometers (GC/MS), LC/MS/MS, Lachat Auto Analyzer, Inductively Coupled Plasma-Atomic Emission Spectrometers (ICP-AES), Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS), High Performance Liquid Chromatography (HPLC), and a Laboratory Information Management System (LIMS).

The laboratory is committed to providing analytical services of the highest quality achievable, offering a high level of client commitment, balancing response and prompt turnaround with quality and reliable analyses. The laboratory strives to maintain, and ultimately exceed, established quality standards when providing objective and cost effective services in today's competitive environmental/analytical marketplace. The laboratory's Quality Assurance program insures accuracy of data from testing methodologies to provide a high level of confidence in test results and is committed to continuous improvement.

1.0 Introduction, Objectives, and Quality Assurance Policy Statement

The objective of the Con-Test Quality Assurance Program is to assure the production of the highest quality of data and service possible, with commitment to compliance with all regulatory authorities and organizations, including ISO/IEC 17025. This manual outlines the quality control and quality assessment systems which are used to achieve Con-Test's Quality Assurance Goals. The QA program is management's tool to ensure commitment to quality and excellence. All personnel concerned with testing and calibration activities within the laboratory will familiarize themselves with the quality documentation and implement the policies and procedures in their work. All tests and calibrations shall always be carried out in accordance with stated methods and customers' requirements.

The Quality Assurance Program addresses all areas of Industrial Hygiene and Environmental chemistry.

1.1 Quality Control

Quality control consists of specific procedures or measures adapted to specific operating conditions. These procedures, which apply to every phase of business done at Con-Test Analytical Laboratory, provide a quality structure upon which each procedure is constructed. The purpose is to ensure quality of data and service to our clients.

1.2 Quality Assessment

Quality assessment involves the continuous evaluation of data and monitoring of analytical processes to ensure that quality control procedures are performing correctly.

1.3 Major Elements of the Quality Assurance Program

1.3.1 The use of appropriate methodologies by technically competent, well-trained personnel, using state of the art instrumentation and equipment.

1.3.2 Adherence to well defined standard operating procedures, with emphasis on sound laboratory techniques.

1.3.3 Monitoring of analytical methods to ensure that data user's needs for precision, accuracy, and sensitivity are met. Assessment of data by use of quality control samples including (but not limited to); blanks, independent laboratory control samples, duplicate samples, matrix spiked samples, and surrogate spiked samples.

1.3.4 Internal and external system and performance audits to monitor compliance with procedures and accrediting authorities (AIHA-LAP, LLC – ISO/IEC 17025, NELAP, and various states), and assess performance of analytical methods.

2.0 Laboratory Structure, Personnel, and Responsibility Organizational Structure (See External Document #318)

2.1 General Manager

The General Manager is immediately responsible for all functions pertaining to laboratory operations including overall financial monitoring and management (P&L), preparation of financial reports and statements, marketing, and overseeing issues concerning client relations and laboratory efficiency. Additional responsibilities include; laboratory personnel management including support and performance evaluation, cost analysis and pricing, and overall laboratory business coordination. The top management is committed to the quality assurance policy and objectives, while continually striving to improve the effectiveness of the management system.

2.2 Laboratory Technical Director

The laboratory Technical Director is responsible for overseeing all aspects of Laboratories Technical operations. The Technical Director provides scientific management, organization, direction, and support to both clients and laboratory personnel to ensure that the highest quality and appropriate product is delivered. The Technical Director ensures compliance with all accrediting authorities and organizations (AIHA-LAP, LLC – ISO/IEC 17025, NELAP, and various states). The daily duties for this position include managing the Quality Control. The Technical Director is also responsible for addressing client and lab personnel questions or concerns with methodology and data quality, and makes recommendations on technical issues. The Technical Director must certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented: all employees must have on file a Demonstration of Capability, and documentation that they have read and understood the QA Manual and appropriate SOP's. He/she must ensure that the training of each member of the technical staff is kept up-to-date (on-going). Other major duties include: coordinating with General Manager and Quality Assurance Officer on technical issues, final review and approval of analysis reports and maintenance of technical as well as program standards. The Technical Director shall possess a bachelor's degree in an applicable physical or biological science (with at least 24 college credits in chemistry and 4 college credits in microbiology), a minimum 3 years' relevant nonacademic analytical chemistry experience (a minimum of 2 years' experience must be in industrial hygiene/metals analyses within the laboratory's scope of accreditation; the remaining one year can be from other non-AIHA-LAP, LLC laboratory analytical procedures). The Technical Director must possess knowledge of IH chemistry calculations with respect to lead-in-air principles and calculations. Relevant academic experience may be substituted for work experience. A relevant master's degree shall be considered equivalent to one year of work experience. The Laboratory Manager has been named the deputy in the absence of the Technical Director.

2.3 Administrative Manager

The Administrative Manager is responsible for managing the Administrative Assistant staffing. This individual is also responsible for all aspects of the corporate accounting system such as payroll, accounts payable, accounts receivable, collections, as well as preparation of financial reports (P&L) and statements.

2.4 Quality Assurance Officer

It is the responsibility of the Quality Assurance Officer to maintain and administer all aspects of the laboratory's Quality Assurance plan therefore ensuring all QA goals are achieved. The Quality Assurance Officer assists the Technical Director in ensuring compliance with all accrediting authorities and organizations (AIHA-LAP, LLC – ISO/IEC 17025, NELAP, and various states). The QA officer addresses every analysis performed in

the lab, including documentation of procedures, formulation and use of control charts, addressing of client and regulatory agency quality concerns and audits, and validation of data. The QA Officer also develops and maintains QC procedures for all analytical areas and prepares quality control reports, monthly or when applicable, for presentation to the laboratory director for routine assessment of measurement systems for precision and accuracy. The QA Officer shall possess a bachelor's degree in an applicable basic or applied science and have at least one year of nonacademic analytical experience appropriate to the types of analyses performed by the laboratory; or in lieu of a bachelor's degree, four years of nonacademic analytical experience. The QA Officer shall have documented training in statistics. Training in quality control procedures is strongly encouraged. The Technical Director has been named deputy in the absence of the QA Officer.

2.4.1 The Quality Assurance Officer prepares Quality Assurance Reports for review by the laboratory management on a regular basis (monthly). The main structure of a report is based on summarization of one or more of the following categories: Quality Assurance Activities, Quality Control Performance, Corrective Actions, and QA review of data packages. Each month the Technical Director and the QA Officer meet and discuss the contents of the monthly report.

2.5 Laboratory Manager

It is the responsibility of the Laboratory Manager to oversee staff and production management and to meet the needs of clients on data completeness and delivery. The laboratory manager assists in laboratory renovations and design, improvements in the facility and work flow, standardization of lab processes and maintenance of leading edge technology, as well as final review and approval of analysis reports.

2.6 Section Heads/ Supervisors

It is the duty of each Section Head to perform and maintain proficiency in the analysis of specified areas and techniques, provide training, supervision, and direction to analysts, insure that work flows smoothly, that quality and turnaround standards are maintained and ultimately exceeded, ensure safety measures are being followed, and to assist development of projects, planning and quality control program in the specified area.

A Supervisor shall possess a bachelor's degree in chemistry, biology, or a closely related field, and have at least 30 college credits in chemistry (or 4 college credits in microbiology for a microbiology supervisor). A supervisor shall have a minimum 2 years' experience in chemical analysis (or a minimum one year experience in microbiology for a microbiology supervisor).

Per MA DEP regulations, the following requirements apply:

Inorganic chemistry I (includes AA Spectroscopy): the supervisor shall have a minimum of 2 years' laboratory experience in chemical analysis, including 6 months training or experience in the operation of an AA spectrophotometer.

Inorganic chemistry II (Includes ICP): the laboratory supervisor shall have a minimum of 2 years' laboratory experience in chemical analysis, including one year training or experience in ICP methods.

Organic chemistry I (includes GC): the laboratory supervisor shall have a minimum of 2 years' experience in chemical analysis, including 6 months training or experience in the operation of a GC.

Organic chemistry II (includes GC/MS): the laboratory supervisor shall have a minimum of 2 years' experience in chemical analysis, including 6 months training or experience in GC methods and one year training or experience in the operation of a GC/MS.

2.7 Chemical Hygiene Officer (Safety Officer)

The Chemical Hygiene Officer is responsible for the development and implementation of the Chemical Hygiene Plan for the laboratory.

- Responsible individuals will be designated for duties to insure compliance with safety, training and medical monitoring requirements of the plan
    - The laboratory supervisors are responsible for conducting regular hazard inspections using the Department Safety Check List (Document#316), either by themselves or a designated individual in the department. The completed checklist is forwarded to the Chemical Hygiene Officer at the end of each month. The Chemical Hygiene Officer addresses any deficiencies and retains the checklists in a binder.
- Ensuring laboratory personnel are using the proper personal protective equipment
- Evaluation of Hood Performance, Coordinate the operation, acquisition and maintenance or fume hoods, emergency safety showers, eyewashes and fire extinguishers
- Hazardous Chemical and Waste training
- Evaluating circumstances requiring pre-approval for work, i.e. dangerous samples or procedures using dangerous reagents
- Provisions for working with Particularly Hazardous Substances
- Enforcement of safety policies

- Provide technical expertise and administrative support to the laboratory community in the area of laboratory safety and health, and direct inquiries to appropriate resources
- Ensure that extremely hazardous substances are appropriately handled and stored and that specific standard operating procedures are developed and followed which instruct all personnel in the safe use of these substances
- Review specific operating procedures for the use, disposal, spill clean-up, and decontamination of extremely hazardous chemicals and substances
- Investigate all incident reports, chemical spills and near-misses to prevent repeat incidents
- Act as a liaison between the laboratory and management bringing unresolved and potentially serious health and safety problems to their attention
- Chemical Hygiene/Safety Committee containing representatives from all departments/areas of the company
- Ensuring all employees receive proper safety training
- Helps maintain SDS sheets

2.8 Individual Analysts

It is the responsibility of each analyst to be cognizant of always maintaining and ultimately exceeding quality standards during the generation of consistently reliable data of the highest achievable.

At Contest, it is the duty of each and every employee to help foster an attitude of continuous improvement in the laboratory with regard to decreasing turnaround in all areas, improving quality of results and providing excellent customer service to produce "delighted clients" who have no reason to go anywhere else.

Per AIHA-LAP, LLC policy, an analyst shall possess a bachelor's degree in chemistry or a related science. A technician is one who does not have a degree in chemistry or a related science. An AIHA-LAP, LLC analyst must complete in-house training per AIHA-LAP, LLC policies (see section 14.0 of this QA Manual).

Per MA DEP regulations, the following requirements apply:

Instrumentation analysts shall possess a high school diploma or equivalent and 8 college credits in chemistry for an instrumentation analyst; have a minimum of 6 months training or experience in the operation of the appropriate instrumentation except for GC/MS or ICP. One year of training or experience is required for the operation of GC/MS or ICP.

Non-instrumentation analyst shall possess a minimum of a high school diploma or equivalent; an analyst shall receive specialized training in the methods to be performed.

2.9 Statement of Confidence

Due to the inherent nature of work provided by Con-Test, employees are required to work with confidentiality. Information concerning analysis data and reports is considered confidential and will be released only to a client or their authorized representative.

Only authorized personnel have access to, and the responsibility for control and issuance of data, materials, and supplies.

2.10 Laboratory Security

Con-Test Analytical Laboratory is a secure laboratory. In order to assure our clients strictest confidentiality, Con-Test has several security measures, including a building security system and laboratory entry system restricting access to only authorized personnel. Unauthorized sample contact or data manipulation is therefore controlled.

2.11 Con-Test Analytical Laboratory organization chart

To view organizational chart, see External Document #318.

3.0 Quality Assurance Objectives

The purpose of Con-Test's Quality Assurance Plan is to ensure the production of quality, objective, and cost effective services to our clients. The laboratory operation offers a high level of client commitment, balancing response and prompt turnaround with quality and reliable analyses.

3.1 Quality Assurance Goals

3.1.1 Establish and maintain the quality management and assurance systems in the production of consistently reliable and accurate "quality data" of known precision and accuracy.

3.1.2 Monitor analytical methods to insure use of appropriate, EPA, State, or recognized agency endorsed or approved methodology insuring that client's need for precision, accuracy, and sensitivity are met or ultimately exceeded.

3.1.3 Insure the use of sound laboratory techniques and practices, by competent trained individuals.

3.1.4 Establish and maintain Standard Operating Procedures for all processes producing uniformity and definition.

3.1.5 Maintain systems for early identification of problems and defined procedures for quick resolution.

3.1.6 Promote a positive attitude toward improvement of total quality.

3.2 Measurement of Data

In the pursuit of the highest data quality achievable, Con-Test utilizes specific procedures applicable to defined situations in the tracking and evaluation of data and data systems.

3.2.1 Use of Quality Control Measures

Quality control measures are part of the daily laboratory routine from which data quality is assessed and controlled. These defined processes are built into each analysis or Standard Operating Procedure. Standard Operating Procedures address all aspects and processes performed in the lab and ensure correct definition and proper utilization through incorporation of method specific QC into applicable methodology. An overview of the entire process and its utilization is addressed in the following sections.

Most Quality Control data, which is obtained during sample analysis provides an indication of the "Quality" of sample data and therefore is provided in laboratory deliverable packages with the applicable sample data. It must be noted that not all reports will contain QC information. This does not mean that the same care and attention was not given to all samples but that regulations dictated that specific QC measures be analyzed on an alternate sample in the analytical batch. Other QC measures are not reported to clients because it does not provide supplemental information about the sample and is therefore not helpful.

The components of uncertainty are identified and estimated for all quantitative tests in the laboratory using the standard quality control procedures for determining precision and accuracy as outlined in section 4.0 of this manual. These include but are not limited to the use of standard reference material; laboratory fortified sampling media or blanks and their duplicates, and sample duplicates.

Primary components of uncertainty arise from: instrument calibration bias, instrument noise/drift, instrument response/line voltage transients, purity of reagents/variation of reagent addition, and analyst technique (including dilutions and subjective measurements).

Quality control results associated with samples are reported in the QC summary report that accompanies the sample results. Where necessary for the interpretation of the test results and when requested, the overall estimate of uncertainty is reported to the client.

See SOP "Estimation of Uncertainty of Measurements" for details on the procedure of estimating uncertainty as well as an example of how Con-Test calculates the estimation of uncertainty.

3.2.2 Laboratory Data Integrity and Ethics Policy

Con-Test Analytical Laboratory understands the importance of environmental testing data to nearly every significant public health and environmental management decision made and consequently has developed this policy to ensure that strict ethical standards are adhered to in the performance of analytical procedures and reporting of analytical results. Con-test will ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work. Con-Test Analytical Laboratory is committed to compliance with all applicable laws, regulations, and other requirements that are imposed upon the laboratory in the conduct of its business, and to practice the highest professional laboratory standards.

3.2.2.1 Principles and Program Components

1) The laboratory is ethically and morally obligated to provide data that is precise, accurate, and of known and documented quality.
2) The laboratory will self-police its operations in order to maintain data user confidence.
3) Data integrity training will be provided to all employees.
4) A quality assurance officer will be appointed to insure compliance with the ethics policy within the laboratory.
5) An enforcement policy through disciplinary action will be implemented.
6) A confidential mechanism will be implemented for anonymously reporting alleged misconduct that will require a full investigation.
7) Procedures are described for guidance on the recall of data if and when necessary.
8) Internal auditing and corrective action procedures are in place to detect integrity issues.
9) Internal data integrity investigations will be thoroughly documented.

3.2.2.2 Role of Quality Assurance Officer within Ethics Program

A Quality Assurance Officer (QAO) is appointed within the laboratory with direct access to the laboratory director and highest levels of management. Among other duties the Quality Assurance Officer will be responsible for compliance within the laboratory and adherence to the data integrity policy. The QA Officer also maintains an internal auditing system whereby all analytical methods are audited at least annually to detect and correct systematic errors, improper practices, and non-compliance. Internal audits are conducted randomly, on a pre-determined schedule, in response to external audit findings, based on client complaints, or anonymous allegations of misconduct. A master list of corrective actions is maintained and progress in the resolution of corrective actions is reported to management on a monthly basis.

3.2.2.3 Data Integrity and Ethics Training Program

Data Integrity Training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. The training will include training in the critical need for honesty and full disclosure in analytical reporting, acceptable and unacceptable scientific practices, including proper manual integration, calibration, and documentation procedures. The laboratory ethics policy will be discussed including the mission statement and consequences of non-compliance including possible enforcement and disciplinary actions. The initial data integrity training and annual refresher training shall have a signature attendance sheet that demonstrates all staff has participated and understand their obligations related to data integrity.

3.2.2.4 Enforcement Actions

Employees who violate the laboratory data integrity policy or knowingly bypass required quality control or quality assurance procedures will be disciplined consistent with the severity of circumstances surrounding the violation. Individuals who knowingly and intentionally falsify data or otherwise commit criminal acts will not be tolerated. Individuals who are discovered using improper practices including "peak juicing", "peak shaving", inappropriate and inconsistent manual integration, falsifying dates, inappropriate changes in the concentrations of standards, and fabricating data ("dry Lab"), after investigation, will be subject to disciplinary action up to and including immediate termination as specified in the employee personnel handbook.

3.2.2.5 Internal Investigations, Reporting, and Monitoring

While it is hoped that allegations of misconduct or violations of the laboratory ethics and data integrity policy will be brought to the attention of supervisors, senior management or the Quality Assurance Officer, issues may also be raised and reporting privately and anonymously to any of the same individuals without fear of reprisal. In the case that employees wish to anonymously report misconduct, a locked drop-box is provided. The QA Officer routinely checks the lock box for reports containing anonymous allegations. All allegations of misconduct will be investigated free from the influence of those being investigated. All investigations and resolutions to allegations of misconduct will be conducted privately and discreetly and must be reported to senior management.

3.2.2.6 Data Recall

In the normal course of business, periodically there will be some reports submitted to customers with erroneous data. There may be many possible causes for the erroneous data, including calculation errors, data entry errors, analytical problems that were not caught during data review, and deviations from standard operating procedures. Some erroneous data could be caused by misconduct or deceptive data recording practices by an individual within the laboratory.

Erroneous data (nonconforming work), once discovered, will immediately be evaluated and subject to the corrective action reporting procedures. When necessary, the customer is notified and work is recalled when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its procedures or the agreed requirements of the customer. Revised report forms will be completed for each report involved, and the client will be notified that changes will be made to the report. A revised report will be issued. A corrective action form will be completed and the error will be recorded in the corrective action database and investigated, unless the error was a simple typographical error that did not affect the data. Discovery of erroneous data might lead to an investigation of improper practice and disciplinary action.

3.2.2.7 Proper and Improper Practices

In the course of laboratory testing it is inevitable that some things will go wrong from time to time. Problems encountered in the laboratory should never be covered up. Improper practices can be perpetuated by inadequate training, ineffective internal assessments, and lack of independent QA review. Most improper practices are shortcuts, appearing to be done to save time and effort. In any case, a corrective action shall be issued immediately and a root cause investigation will be initiated.

Data should always be able to be reconstructed without having to talk to the analyst who performed the test and should stand by itself.

In the light of these principles, realities, attitudes, and the associated pressures, an extensive, although not all-inclusive list of proper and improper practices are presented below.

3.2.2.7.1 Proper Practices

1) Analytical results must be reported from actual analysis.
2) Record exceptions to and deviations from documented procedures.
3) Records must be complete to trace actual analysis and stand by themselves without discussion with analyst.
4) If calibration or QC is not within limits – consistently integrate peaks and perform corrective action of maintenance.
5) Only reject points from an MDL calculation using statistical evaluation or if a known error has occurred.
6) Document all calibration and QC data.
7) Adjust laboratory reporting limit and upper end of linearity based on current initial calibration.
8) Report and document problems and the need for corrective actions.
9) Report knowledge of unethical behavior to management.
10) Exceeded holding times must be reported to clients.
11) Document all manual integrations with before and after print-out, reason, name, and date.
12) Document all out-of-control events.
13) Retain non-compliant data or data for assays that did not work.
14) Document corrective actions and maintenance procedures.

3.2.2.7.2 Improper Practices

1) Fabrication of data or other information
2) Misrepresentation of QC sample results
3) Improper date/time setting or recording
4) Improper peak integration
5) Improper GC/MS tuning
6) Improper calibration and verification
7) Data file substitution or modification
8) Unwarranted sample dilution
9) Improper alteration of analytical conditions
10) Unwarranted manipulation of computer software
11) Concealment of a known problem malfunction issues

Any of these items shall have a corrective action issued, with a root cause investigation.

3.2.2.7.3 Avoid Non-Authentic Data – Intentional or accidental reporting of incorrect data

1) Wrong number of significant figures used in calculations and reports
2) Quality Control samples not analyzed or reported at proper frequency
3) Missing units, headers, and initials in the record

Any of these items shall have a corrective action issued, with a root cause investigation.

3.3 Specific Routine Procedures Used to Assess Data Precision and Accuracy

3.3.1 Precision: Assessment of Precision

Precision, as defined by the Environmental Protection Agency (SW-846), "is the measure of the degree of agreement among duplicate sample analyses without assumption of knowledge of its true value." At Con-Test, precision is estimated by means of duplicate analyses expressed as relative percent difference or range. Duplicate control limits vary from zero (no difference between duplicate samples) to the historical mean of the applicable accumulated set of duplicate measurements plus three standard deviation units.

Con-Test analyzes duplicates at a frequency of at least 5% or one per batch in order to construct data control charts, and sometimes more frequently if required or deemed necessary.

3.3.2 Accuracy: Assessment of Accuracy

Accuracy, as defined by the EPA, "is the closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy is the combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to the true value."

Bias is the deviation of the measured value from a known spiked amount due to matrix effects and other undeterminable sources.

By determining the recovery of a known amount of target analyte spiked into a sample (matrix spike) or medium blank, Con-Test monitors the accuracy of an analytical process. An indication of laboratory total

accuracy can be obtained after the accumulation of a significant number of observations.

Matrix spikes, as well as matrix spike duplicates are utilized for the assessment of accuracy and precision. Con-Test utilizes control limits for accuracy based on the historical mean percent recovery of the applicable accumulated population plus or minus three standard deviation units.

3.3.3 Assessment of Data Quality

Historical monitoring and evaluation of performance through the use of X bar and R control charts provides a reliable way of assessing quality of data. Through the compiling and plotting of historical data points duplicate and spike results) a historical data point spread or control chart (assuming a normal distribution) using the population is obtained. Through the use of statistics, specifically the calculation of the mean value of the population and the standard deviation (average difference from the mean value), control limits are calculated annually, using at least 20 data points. The purpose of control limits is to demonstrate that the method is performing in a state of statistical control.

3.3.3.1 Control Charts and Control Limits

Control charts provide a tool for distinguishing the pattern of indeterminate (random) variation from the determinate (assignable cause) variation.

The control chart is actually a graphical representation of quality control efficiency. The data from a series of analyses can be plotted with the vertical scale in units of the test result and the horizontal scale in units of time or sequence of analysis. The mean value of the population and standard deviation can be calculated and the spread can be established.

A minimum of twenty data points is normally required to determine chart limits. The determination of appropriate control limits or statistically acceptable deviations can be based on the capability of the procedure as known from past experience or can be arbitrarily set at a desired level (prescriptive limits). Commonly, the limits are set at three standard deviation units on each side of the mean.

If a procedure is "in control", the results will almost always fall within the established statistical control limits. The charts may also disclose trends and cycles from assignable causes.

Control charts are generated and assessed on a regular basis (monthly); in order to identify, explain, and correct any observed trends in a timely manner. A "trend" is defined as 7 consecutive points on either side of the mean. Trends indicate issues, which necessitate explanation. If trend continues a corrective action shall be needed. Charts are generated according to a schedule. They are assessed and trends are identified by the following criteria:

- Must span 7 or more analysis dates
- Must be outside the 90-110% recovery window
- If the chart is for RPD, and the chart trend is below the mean, then it is not designated as a trend
- The mean may be skewed, due to an extreme outlying point, causing a false trend
- If all data points for an analyte are always below 70% recovery, an investigation is warranted. (It may need to be classified as a difficult compound, or a corrective action may be issued)
- For "real time" trend analysis, only review the last quarter (3 months) of data: anything further back is too old for real-time viewing

Any trends that are identified are logged into a database, and assigned to the analyst, who will investigate the trend and write an explanation, and then forward it back to the QA department.

When evaluating control charts, the following general criteria are considered:

- 1 Measurement > Control Limit
  Analyze another
  Stop test if > Control Limit

- 2 of 3 successive point > Warning Limit
  Analyze another
  Stop test if > Warning Limit evaluate bias and correct

- 4 out of 5 points exceed 1 standard deviation or decreasing or increasing order on the same side of the central line.
  Analyze another
  Stop test if exceeds 1 standard deviation or same pattern and correct

- 7 Successive points on the same side of the central line.
  Stop test and correct

Although, in most cases, the laboratory monitors and establishes its own control limits, in order to meet method requirements, method specified control limits take precedence over those established in the laboratory. Data outside laboratory control limits but within method specified limits may be considered of sufficient accuracy to report.

For the AIHA, LAP-LLC lead program, laboratory determined statistical acceptance limits and frequencies must be at least as stringent as the interim limits of 80-120%.

3.3.3.2 Con-Test Classification System for Waters and Wastes

The laboratory has developed control charts and acceptance limits based on general matrix stability and characteristics. Most waters and wastes can be adequately categorized and evaluated under two major groupings; Potable & Non-Potable Water. Classification of Waters for the purpose of comparison and evaluation of data to establish control limits is based on the following table and comments.

Con-Test Classification of Waters and Wastes
Categorization of Potable and Non-Potable Water

Potable Water	Non-Potable Water
-Public Drinking Water	-Wastewater
-Well Water (other than monitoring)	-Effluent of Discharge Waters
-Water (depending on matrix characteristics)*	-Storm Water (matrix related)
-Bulk or Bottled Water	-Water (depending on matrix)*
-Other	-Monitoring Well Water
	-Leachate
	-Ground Water (other than effluent)
	-Recreational water (Pools, Beaches)
	-Streams, Lakes, Rivers
	-Other

*If the sample has been evaluated by the client as "WATER" it may be compared to either of the above categories according to the laboratories discretion (determined matrix characteristics).

Samples previously classified by Clients in one of the above categories may occasionally be laboratory re-classified and subsequently compared to matrix control limits other than the one which was specified by the client when deemed more applicable by the laboratory. This is based upon laboratory matrix characterization including; appearance, matrix consistency, and matrix components. If difficulties encountered in the analysis of a sample can reasonably be determined as matrix and not system related, a sample matrix may be compared to limits other than one listed on the chain of custody or categorized as Other than one of the above.

3.3.3.3 Out of Control Events:

An '" Out of control Event" is any event, which does not fall within established control limits. Con-Test laboratory takes immediate corrective action whenever quality control data is outside acceptance limits. Data is either not reported or reported as qualified data until the root cause of the problem is determined and corrected. Records are kept of all out of control events.

If the sample values do not meet the minimal acceptance criteria, a root cause investigation is conducted to determine, correct, and document the source or suspected cause of the variance. The root cause investigation continues until acceptance criteria are met or the data is flagged with an appropriate explanation of the variance. Attempting to accurately identify the root cause of the variance shall involve initiation of a formal corrective action.

The following steps are taken for these events:

The analyst/technician will attempt to determine why the analytical values exceed the control limits and correct the problem if identified. Calibration-related out of control events are documented on non-conformance forms. Out of control events detected during control chart review are logged into a database, and assigned to the analyst, who will investigate the

trend and write an explanation, and the forward it back to the QA department.

Additional actions include:

- Data Integrity Validation

  A check of data transcription from log books, calculations, method requirements, reviews of sample matrix data and other possible causes.

- Data Re-evaluation

  The analyst will re-analyze / possibly re-prepare both the quality control samples and samples a second time if the samples are such that significant degradation has not occurred or sufficient sample is available.

  Additional QC measures can be utilized to eliminate suspect sources of error. (I.e. Fortified Blanks can be prepared to run with the samples to eliminate suspicion of inaccuracy in spiking procedures and/or spiking equipment).

- Determination of Root Cause

  In the event a consistent bias is discovered in procedure, method, or the like, a formal corrective action is initiated to ensure that the problem is tracked to resolution.

3.3.3.3.1 Nonconforming Work

When necessary, the customer is notified and work is recalled when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. Deviations that result in nonconforming work shall be immediately evaluated. Correction is taken immediately, together with any decision about the acceptability of the nonconforming work.

Non-conforming QC samples such as infrequent LCS/MS/CCV failures are addressed through case

narrative notes with the analytical report and/or sample re-preparation and/or reanalysis when applicable.

Non-conforming QC problems will be addressed by the QA department immediately through corrective action. This will start a root cause investigation including routine data review, data validation, internal or external audits as follows: An evaluation of the significance and extent of the problem will be conducted by the QA department, with oversight by management staff including the laboratory manager and laboratory director. If the problem significantly affects previously reported data, the client is notified by the project chemist assigned to the particular client and a new report will be issues after the problem is corrected. If a significant problem is found that is not able to be addressed and corrected immediately, all affected work will be halted in the laboratory by the QA department and/or laboratory manager. Clients with affected samples will be notified by their project chemist, and work will be subcontracted to a qualified laboratory at the clients' request. Work will not be resumed on affected analyses until a root cause analysis of the technical aspects of the procedure is performed by laboratory management, previously reported results are reviewed for accuracy and method compliance, and the QA department has approved changes that will bring the method into full compliance. When all three of these conditions have been met, the laboratory director will again allow work to be accepted for these procedures.

3.3.3.4 Sample Matrix Interferences

Samples, which indicate the presence of interferences, are normally treated in one or more of the following ways in an attempt to eliminate the interference(s) and obtain a defensible, valid result.

- The sample is successively diluted and reanalyzed to eliminate interferences.
- Modification of sample matrix is used to remove interferences or to stabilize the analyte of interest.
- The sample is analyzed by method of Standard Additions.

- An applicable (approved) alternate method or wavelength which is not subject to the interference(s) is utilized.
- Various sample/extract clean-up procedures may be employed.

3.3.3.5 Non-method Performance Factors

The following are examples of non-method performance factors:

- Sample non-homogeneity
- Method applicability questionable due to sample matrix or other factors outside the control of the laboratory.

- Client did not submit samples according to method required or recommended procedure (i.e. field blank/media blank for background or contamination determination, etc.).

3.3.3.6 Data Conclusions

If upon reanalysis the data meets acceptance criteria and it can be reasonably assumed the original variance or bias in technique or procedure has since been eliminated or corrected, the analysis may continue and results are reported.

If the result continues to fall outside the established control limit range and the laboratory method performance factors for that analyte are shown to be in control, the variance is judged to be matrix related, not system related. The data user is informed that the result for that analyte is suspect due to matrix chemical or physical effects and analysis by an alternate method if possible should be considered.

Result data is flagged with the appropriate message on the analysis report if the interference could not be satisfactorily eliminated or response was marginal.

If analysis results are rejected or considered of questionable integrity they may not be utilized or plotted on the X bar and R charts.

3.3.4 Certification, Accreditation, & Regulatory Agencies

Con-Test Laboratory holds and maintains certification and accreditation from a number of different state, federal, local, and regulatory agencies encompassing all regulated services.

3.3.4.1 Certifications and Licenses

Original certificates are displayed in the log-in reception area. Copies are available on the network and in a binder in the QA department.

Con-Test holds certifications/accreditations and licenses with the following agencies:

- AIHA-LAP, LLC Accreditation # 100033
- AIHA-LAP, LLC Environmental Lead Laboratory Accreditation Program (ELLAP) (NLLAP recognized)
- Commonwealth of Massachusetts Chemical Analysis of Potable, Non-Potable, and Microbiological Analysis of Water Certificate of Approval, Lab ID #MA0100
- Connecticut State Approved Public Health Laboratory # PH-0567 – Potable Water, Wastewater, Sewage, and Soil
- ELAP/1° NELAP Accreditation, State of New York Environmental Laboratory Certification
  Lab ID #10899 – Solid & Hazardous Waste, Air and Emissions, Potable Water, and Non-Potable Water
- New Hampshire (State of), Department of Environmental Services
  Lab ID #2516 - 2° NELAP Accreditation - Drinking Water, Wastewater, and Solids
  1° NELAP Accreditation for EPH and VPH
- Rhode Island and Providence Plantations, Department of Health, Analytical Laboratory Certification (Certification # LA000112)

- Vermont Lead Regulatory Program, Vermont Department of Health – License # LL015036

- New Jersey, Department of Environmental Protection, Lab ID # MA007 - 1° and 2° NELAP
  Clean Air Program (CAP) – Atmospheric Organics, Atmospheric Inorganics (Non-Metals)

- North Carolina (State of), Department of the Environmental and Natural Resources
  Lab ID # 652 – Wastewater and Solids and Hazardous Waste

- Florida, Department of Health,
  Lab ID # E871027 - 2° NELAP Accreditation
  Air and Emissions

- State of Maine Certification Program
  Lab ID #MA00100
  Certificate #2011028
  Drinking Water, Wastewater, and Solids

- Commonwealth of Virginia Department of General Services Division of Consolidated Laboratory Services
  Lab ID #460217 - 2° NELAP Accreditation
  Certificate #1827
  Drinking Water, Wastewater, Solids, and Air

3.3.4.2 Participation in Proficiency Sample Programs

In the maintenance of certification and accreditation in the applicable areas, Con-Test participates in a wide range of environmental laboratory proficiency programs in which Con-Test's expertise is demonstrated through the analysis of proficiency samples. Proficiency samples are managed, analyzed, and reported in the same manner as real environmental samples. They utilize the same staff and methods as used for routine analysis of that analyte as well as procedures, equipment, facilities and frequency of analysis.

Those proficiencies which are regularly participated in include the following:

- New York State DOH Proficiency Studies (two potable and two non-potable rounds per year)
- DMR QA Studies (performed annually)
- AIHA-LAP, LLC Proficiency Analytical Testing (IHPAT) Program (four rounds per year)
- AIHA-LAP, LLC Environmental Lead Proficiency Analytical Testing (ELPAT) Program (four rounds per year)
- Commercial Vendor ("Environmental Resource Associates" (ERA)) WP, WS, AE, and Soil studies

An Internal QC program is run for AIHA-LAP, LLC Fields of Testing not covered by the AIHA-LAP, LLC proficiency studies (total/respirable dust, Hg in Air, TO-11, TO-15, and TO-10A). Twice annually, the laboratory shall prepare a minimum of 4 independently prepared blind samples at varying levels, as well as a blank, with the resulting data treated as it would be in a round robin program. These blind samples are made up and spiked by either that department supervisor (technical manager), or by the QA Officer.

Acceptance criteria for results are from the laboratory-generated control limits (which have been established by the control charting program, and are generated and reviewed annually).

The QA Officer and Technical Director carefully review results of Proficiency tests when available. Any unacceptable result will immediately initiate a corrective action, and a "root cause" investigation will begin. The original runs and paperwork are reviewed with the analyst to determine possible root causes. Corrective actions, including additional maintenance of equipment, quality control sample analysis, or modifications to

SOPs are implemented when necessary. A make-up proficiency sample is ordered and analyzed and licensing authorities are notified in a report, if appropriate, of corrective actions.

3.3.4.3 Use of External Laboratories

When samples are received for an analysis which is not performed by the laboratory, a qualified outside laboratory is found to perform the analysis. Only outside laboratories that have demonstrated proficiency in the analysis requested are selected. Laboratories are deemed proficient if they are:

1) Accredited by AIHA-LAP, LLC
2) Certified by a state or recognized "Quality" agency
3) Accredited under NELAP for any part of the testing covered under NELAP.

The laboratory shall advise the client in writing of its intention to subcontract any portion of the testing to another party and the written approval from the client will be retained. The laboratory shall retain records demonstrating that these two requirements have been met.

Only laboratories following approved and standard methods will be used for outside work. When work is placed with a NELAP laboratory, the final report cover sheet will indicate the laboratory's NELAP id.

For AIHA-LAP, LLC and NELAP analyses, written pre-approval from the client is required. This may include "blanket" approval for any current or future projects.

Con-Test Analytical Laboratory is responsible to the client for subcontractors' work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used.

Communication to the subcontracting laboratory of any special report requirements, like immediately notifying Con-Test of MCL drinking water exceedances is facilitated by the Chain of Custody. The following is stamped on all subcontracting chain of custodies: "Subcontracted lab must notify Con-Test Analytical Lab of any MCL exceedance within 24 hours of obtaining valid data".

Con-Test Analytical Laboratory requires each of our subcontracting laboratories to provide current copies of all the certificates they hold for each state they are certified in. In addition, Con-Test Analytical reserves the right to audit any subcontracting laboratory that we send large volumes of business. A qualified representative from Con-Test Analytical will perform this on-site inspection.

3.3.4.4 Non-Routine Industrial Hygiene Samples

New analytical procedures for the laboratory and or non-routine samples require special attention. Validation of method by a three-step process is required. This includes the determination of single-operator precision and bias, analysis of independently prepared unknown samples, and determination of method ruggedness.

Method development includes determination of recovery and stability of analyte on the medium, precision and accuracy of analytical measurement. Clients are informed of the non-routine, non-regulatory nature of these special tests. Incompletely developed, qualitative tests are reported as "estimated" or "semi-quantitative" with appropriate notes or qualifiers.

4.0 Internal Quality Control Checks and Frequency

Con-Test employs a wide range of quality control checks adapted to specific situations and methodology in the assessment of data quality thus ensuring production of data of known precision and bias. Record generation for quality control begins when the samples arrive in the laboratory, continues through analysis and evaluation and ends with the plotting of quality control results on X bar and R charts. Each project is unique and therefore in each work plan, the numbers and types of blanks, references, duplicates, and spiked samples (etc.) will vary. Minimum frequencies are specified below.

Due to the inherent variability and substantial number of distinct methodologies and applicable Quality Control measures only a generalization of QC measures and frequency by major department is offered below.

4.1 Blanks

For all analytical determinations, blank analyses are performed as a routine procedure when samples are analyzed. Blank determinations are analysis specific and are subjected to the same preparation methodology as regular samples. Blank analysis determines when background peaks or materials are sufficiently low (or absent) to permit the analysis of samples to proceed.

If satisfactory blanks are not obtained in these steps, additional steps are taken to determine cause and to eliminate the source of contamination.

At Con-Test one or more of the following types of blanks is analyzed individually or multiply throughout an analysis run.

4.1.1 Reagent Blanks

A reagent blank consists of laboratory pure water and any reagents added to the sample during analysis or straight solvent.

Reagent blanks are run for use in monitoring baseline correction and are inserted at regular intervals during large batches of samples to check for carryover contamination and/or instrument baseline drift.

4.1.2 Method Blanks

A method blank must be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination or background resulting from the analytical process.

At least one method blank is analyzed for each applicable analysis batch. One method blank is analyzed per batch of twenty samples or less. Results from method blanks are not subtracted from corresponding sample results but are reported along with samples for evaluation by the data user.

4.1.3 Trip Blanks

Trip blanks are analyzed for determination of contamination attributable to shipping and handling procedures. This type of blank is especially useful in documenting contamination of volatile organics samples. Trip blanks are analyzed when applicable.

4.1.4 Holding Blanks

Holding blanks are kept in the volatile organics refrigerator and analyzed periodically to determine contamination from sources also being held for analysis in the refrigerator. Holding blanks are analyzed every two weeks or when possible contamination is suspected.

4.2 Calibration

Verification and/or validation of equipment, such as balances, thermometers, and spectrophotometers, shall be performed with National Institute of Standards and Technology (NIST) traceable standards. Calibration certificates must indicate NIST Traceability along with the measurement results and the associated uncertainty and/or a statement of compliance with an identified metrological specification, such as tolerance. External services used for calibration of weights, NIST thermometers and

Eppendorf's must be accredited to ISO/IEC 17025:2005 by a recognized accreditation body. Reference standards, such as Class S weights and NIST traceable thermometers, are used for calibration only and shall be calibrated by an organization that can provide traceability to NIST and be accredited to ISO/IEC 17025:2005 by a recognized accreditation body. NIST Digital thermometer is sent out annually for calibration. Long stem NIST thermometers are purchased annually. Reference weights and reference thermometers are re-calibrated every year. Analytical balances must be checked each day of use with a minimum of two ASTM Class 1 weights, in ranges appropriate to the laboratory's weighing needs. Measurements produced in the laboratory are based upon comparison to analyzed standards. The reference standard results are utilized to generate calibration curves, which are then used in the quantification of sample results. Eppendorf pipettes are calibrated annually by an outside vendor, and on a weekly basis they are verified by the analyst to ensure that they remain within specifications. Laboratory staff performing in-house calibrations and verifications shall have received documented training.

Refer to manufacturer's instructions for procedures on how to transport and store measuring equipment and reference standards.

4.2.1 Instrument Calibration

All instruments are calibrated using standard solutions of known concentrations. The standards are either purchased certified standards or carefully prepared by the laboratory. Major analytical equipment calibrated with standard materials includes: gas chromatographs, GC/MS, IC, HPLC, ICP, ICP/MS, Lachat Auto Ion-Analyzer, UV-VIS spectrophotometer, and analytical balances.

4.2.1.1 Initial Calibration

Initial calibration of any analytical instrument is instrument, as well as methodology, dependent. Calibration normally consists of use of several levels of a reference standard and a blank.

Generally, instrument standard calibration (and therefore sample quantification) in the Organics department is based on calibration curves comprised of 3-5 standards of known concentration; for the Metals department, 2-5 standards; and for Wet Chemistry, 3-5 standards. All the above are excluding the calibration blank, if required. The minimum number of standards used is often dictated by the SOP or method.

Sufficient raw data are retained to reconstruct the calibration used to calculate the sample result. Calibration standards include a

concentration at or below the regulatory/decision level but above the laboratory's detection limit. Reporting limits are not less than the lowest calibration standard.

For AIHA-LAP, LLC samples a RL verification is analyzed with each batch of samples. This is a standard spiked at the reporting limit. Annually a matrix matched reporting limit (RL) verification needs to be analyzed.

Results of samples must be within the calibration range (bracketed by standards) or the results must be flagged as having less certainty, unless reported to the MDL and qualified with a "J" flag at the request of the client. Results over calibration for will not be reported unless requested by the client.

Note: Due to CT RCP requirements to report two dilutions, clients requesting to follow CT RCP protocols will be requesting to report data over the calibration with "E" qualifiers if applicable.

If calibration parameters are outside of method specified performance criteria, data will be flagged as estimated or not reported until a valid calibration is obtained.

4.2.2 Calibration Validation through use of Laboratory Control Samples (LCS's) and/or Reference Materials

All calibrations must be verified by a second source of material which is independent from the calibration standards. They consist of either a laboratory control matrix spiked with analytes representative of the target analytes (LCS) or certified material (Reference) or ICV standard.

All calibrations must be validated by this second source material prior to sample analysis. Reference materials are traceable to NIST, ISO/IEC 17025 and ISO Guide 34 when available.

4.2.3 Calibration Check Samples

Calibration Check Standards are utilized to determine the stability of calibration of an instrument between periodic re-calibrations, or for assessment of linearity agreement between subsequent calibration standards and corresponding curves.

The Organics department analyzes one or more check standards consisting of all required compounds when validating use of a previously calculated calibration curve. With longer analysis runs throughout the laboratory these samples are

run periodically to verify continuous instruments calibration stability and ensure consistent performance of the method.

Where traceability to the SI is not technically possible or reasonable, the laboratory shall use certified reference materials provided by a competent supplier (refer to ISO/IEC 17025 4.6.4), or use specified methods and/or consensus standards that are clearly described and agreed to by all parties concerned. A competent supplier is an NMI or an accredited reference material producer (RMP) that conforms with ISO guide 34 in combination with ISO/IEC 17025, or ILAC Guidelines for the Competence of Reference Material Producers, ILAC G12. Conformance is demonstrated through accreditation by an ILAC recognized signatory.

4.3 Laboratory Instruments/Equipment, Maintenance Logs, and Reference Standards and Materials

The laboratory shall be furnished with all items of equipment (including reference standards and materials) required for the correct performance of tests for which accreditation is sought. In those cases, where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this standard are met.

Equipment, Reference Materials, and Reference Standards are transported, stored, maintained, inspected, and cleaned according to the manufacturer's instructions. Any defective item of equipment is clearly marked and taken out of service until it has been shown to perform satisfactorily.
Each item of equipment, reference standard, or reference material is labeled to show its calibration status. As a mechanism for tracking instrument performance, logbooks are provided for each instrument (GC, GC/MS, LC/MS/MS, ICP, ICP/MS, HPLC, IC, Lachat Auto Analyzer, UV/VIS Spectrophotometers, microscopes, balances, TOC analyzer, and incubators). Equipment, reference materials, and reference standard records include:

1) Name of item of equipment or reference model
2) Manufacturer, identification, model number, serial number
3) Date of installation and dates of service
4) Current location
5) Condition when received
6) Copy of manufacturer's instructions or manuals
7) Dates and results of calibrations/verifications and date of next calibration/verification
8) Details of maintenance carried out to date, and planned for the future
9) History of any damage, malfunction, modification, or repair

The laboratory supervisors are responsible for this data and periodically examine all these books. Any significant change in a critical parameter triggers further examination and possible instrument service.

These books along with preventative maintenance act as an operational tool for minimizing instrument down time, and maintaining them in optimum condition.

Support equipment is calibrated/ verified annually using NIST traceable references over the range of use. Balances, ovens, refrigerators, freezers, incubators, and water baths are checked with NIST traceable references and recorded. The accuracy of all thermometers, are verified annually by comparison with a certified thermometer. IR temperature guns and dial thermometers must be calibrated quarterly. Additional monitoring as prescribed by the test method SOP is recorded. Eppendorf pipettes are calibrated on an annual basis by an outside vendor, and verified weekly by the analyst to ensure that it remains in specifications. External services used for calibration of weights, certified thermometers and Eppendorf's must be accredited to ISO/IEC 17025:2005 by a recognized accreditation body.

The sterilization temperature and cycle times of each autoclave run for biological tests are recorded by use of appropriate chemical or biological sterilization indicators. A maximum-temperature registering thermometer is used with each autoclave run, to ensure that the sterilization temperature of each cycle is reached. Spore suspensions are used weekly to verify the autoclave operation. The autoclave timer is checked quarterly by a stopwatch and recorded. Autoclave tape is only used as an indicator that each batch has been exposed to the sterilization process.

Refrigerator and freezer temperatures are recorded twice daily, with acceptance criteria of 4°C $\pm$ 2°C for refrigerators and less than 0° C for freezers. Incubator temperatures are recorded twice daily (with readings separated by at least 4 hours), with acceptance ranges of 35.0°C $\pm$ 0.5°C (for total coliform and HPC), 44.5°C $\pm$ 0.2°C (for fecal coliform), and 20.0°C $\pm$ 1°C (for BOD).

4.3.1 Equipment List

The following is a list of commonly utilized major analytical equipment. Please note this is not a complete listing.

Equipment	Number	Make and Model
Gas Chromatographs	18	Agilent/Hewlett Packard – 5890/6890/7890 3-PID-FID 3-FID-FID 1-FID 9-ECD-ECD 1-TCD-FID 1-FPD

GC/MS	16	Agilent/Hewlett Packard – MSD 5970/5972/5973/5975 7-Purge and Trap – EST 3-Direct Inject 1-Direct Inject with Cryo 4-Air Entech Auto samplers 1-Air PE Auto Sampler
LC/MS/MS	1	Agilent 6400 Series Triple Quadrupole LC/MS system
Concentration Workstations	9	3-N EVAP 6-Buchi Syncore Turbovaps
TCLP Extractors	4	80 station capacity
Sonic dismembrator	1	Fisher Model 500
Microwave Extractor/Digester	2	MARS Xpress

Equipment	Number	Make and Model
Mercury Analyzer System	1	Perkin Elmer FIMS 100
Ion Chromatograph	1	Dionex ICS 2000
ICP	2	Perkin Elmer – Optima 4300 Dual View Simultaneous
ICP-MS	2	Perkin Elmer ELAN 9000 Agilent7800/7900 ICP-MS
UHPLC	1	Dionex Ultimate 3000
Digestion Block	2	SPC Science Digi-Prep MS
Automated Analyzer	1	Lachat Quikchem 8000 FIA+
Spectrophotometer	3	ThermoSpectronic Genesys 20
Kjeldahl Apparatus	1	FOSS Tecator Digester
NH3/TKN Distillation Unit	1	FOSS Kjeltec8100 Distillation Unit

Flashpoint Apparatus	2	1-PetroTest PM4, closed cup 1-Koehler closed cup
Microscopes	3	Olympus (BH2)
pH/ Ion Meter	3	1-Orion EA 920 1-ThermoElectron Orion 420A+ 1-Accumet AB15
Conductivity Meter	1	YSI Model 35
Dissolved Oxygen Meter	1	YSI Model 58
TOC analyzer	1	Teledynde Lotix TOC Combustion Unit
BOD auto analyzer	1	Skalar Model 21088903-01 BOD analyzer
Turbidimeter	2	1-VWR Model 46210-200 1-Intertek WTW Turb 550
Beckman Centrifuge	1	Beckman Model J6-HC
ENCON Evaporator System	1	Model DE4-B
Soxhlet Extractors	225	Soxhlet glassware and heating mantles
Analytical Balances	17	

4.4 Annual Preventative Maintenance

In order to maintain instruments in optimum working condition, minimize instrument down time and delayed results, Con-Test maintains service contracts with regularly scheduled preventative maintenance guidelines. Service contracts and preventative maintenance schedules are standard for sophisticated and vital equipment such as GC's, GC/MS, HPLC, IC, ICP, ICP-MS, UV-VIS Spectrophotometer, Lachat Auto-Analyzer, and the TOC analyzer. In the event of unavoidable down time Con-Test has alternate methods of analysis for most analytes.

Common routine maintenance activities should be performed according to the following schedule.

Instrument	Method	Activity	Frequency
GC/MS VOA-Purge and Trap	624 8260	Check Gas	Daily
		System Bake	Daily
		Replace Septa	As Needed
		Clean/Replace Liner	As Needed
		Change Column	As Needed
		Change Ferrules	As Needed
		Replace Trap	As Needed
		Change Vacuum Pump Oil	Twice Per Year
GC/MS Semi-VOA	625 8270	Check Gas	Daily
		System Bake	Daily
		Replace Septa	Daily
		Replace Glass Wool	Daily
		Clean/Replace Liner	As Needed
		Change Column	As Needed
		Change Ferrules	As Needed
		Change Vacuum Pump Oil	Twice Per Year
GC	608, 8081, 8082 602, 8015, 8100	Check Gas	Daily
		Replace Septa	Daily
		Replace Glass Wool	As Needed
		Clean/Replace Liner	As Needed
		Change Column	As Needed
		Change Ferrules	As Needed
Eppendorf Pipettes		Calibration/Verification	Annual by vendor Weekly by Analyst

Instrument	Method	Activity	Frequency
Infrared Thermometer Gun		Calibration/Verification	Quarterly
ICP	6010 200.7	Check/Change Pump Tubing	Daily
		Change Capillary Tubing	As Needed
		Clean Nebulizer	Monthly/ As Needed
		Clean Spray Change	Monthly/ As Needed
		Clean Torch	As Needed

ICP-MS	6020 200.8	Check/Change Pump Tubing Change Torch Change Injector Clean Gem Cone Tips Clean Scott Spray Chamber	Daily As Needed As Needed As Needed As Needed
Mercury Cold Vapor	7470 7471 245.2 NIOSH 6009	Clean Cell Clean Windows Change Tubing	Monthly Monthly As Needed
Lachat Auto Analyzer Nitrate	SM 4500 NO3-F Lachat 10-107-04-1-A	Rinse/Flush System Change Tubing Clean Optics/Pump	Daily As Needed As Needed
pH/Ion Meter & Electrodes	SM 4500 H-B SW-846 9040	Rinse/Clean Electrodes	Daily
Conductivity Meter & Bridge	SM 2510B	Rinse/Clean Bridge Replatinize Bridge Calibration	Daily As Needed Annual
UV Lamp (Microbiology)	SM 9223	Clean with Ethanol with cloth	As Needed

4.5 Surrogate Additions

Surrogates are organic compounds added to a sample, which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not formally found in environmental samples.

Surrogate additions are regularly utilized in the organics section of the laboratory. Organic compounds are added to a sample just before processing so that the overall efficiency of a method can be determined. Surrogate spikes and their recovery are used to create control charts for the organics section. If the surrogate recovery is outside of the control limits, the data is considered questionable and the sample is re-analyzed to confirm possible matrix interference, spiking procedure problems, or reported as estimated data.

4.6 Duplicate Analysis

Laboratory duplicates (smallest number of replicates) are two sample aliquots split in the laboratory from the same container and analyzed independently under identical conditions. From the analysis of duplicates a measure of precision or repeatability associated with the laboratory procedure can be obtained. The comparison of results for duplicate samples to what has been previously achieved provides assurance that the methodology is performing within establishing limits of precision. A large number of data points are usually needed to calculate control limits representative of analyzed data.

It is standard practice in the Wet Chemistry and Metals laboratories to prepare and analyze one duplicate for each 10-20 samples or one per batch analyzed. Samples selected for duplicate analysis are at random on this basis. The number of duplicate samples performed may be more frequent if dictated by the method, SOP, or statement of work.

4.7 Sample Matrix Spikes (MS)

A matrix spike is an aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A sample matrix spike is usually utilized to provide a means of assessing accuracy for the method used on a specific sample matrix. The recovery of the spiked analyte is expressed as a percent of the amount of target analyte added to the sample. The purpose of this procedure is to evaluate the consistent deviation of measured values from the true value as a result of systematic errors and to determine if sample matrix composition has any effect on analyte recovery. This procedure facilitates the identification of possible method interfering substances (matrix bias) which may be present in the spiked sample so that appropriate action can be taken. This enables the laboratory to ensure methodology is performing with established limits of accuracy.

It is standard practice in the laboratory to prepare and analyze one matrix spike for each 10-20 samples or one per batch analyzed. The number of spiked samples performed can be more frequent if dictated by the method, SOP, or statement of work.

4.8 Sample Matrix Spike Duplicates (MSD)

Matrix spike duplicates, as defined in SW-846: "Intra-laboratory split samples spiked with identical concentrations of target analyte, which undergo the same processes". Matrix spikes and matrix spike duplicates are routinely analyzed periodically in the accumulation of precision and accuracy data or when conditions exist where matrix applicability is questionable or matrix interferences are suspected.

4.9 Laboratory Fortified Blanks (LFB) and LFB Duplicates

It is standard practice in the laboratory to prepare and analyze one duplicate for each 10-20 samples or one per analytical batch. LFB's reflect achieved accuracy under ideal conditions for a methodology and analysis procedure but do not provide an indication of system or method accuracy with respect to bias associated with a given sample matrix. Laboratory Fortified Blanks may be also known as Blank Spikes.

4.10 Method Detection Limits (MDL)

The MDL has been determined by the laboratory and documented for each analyte where spiking solutions are available. MDL's can be determined by the procedure presented in 40 CFR Part 136 Appendix B Revision 1.11 and must be determined at least for all drinking water and wastewater certified analyses, including Lead. All sample processing steps of the analytical method, are included in the determination of MDL. The standard deviation of the analysis of at least seven portions of spiked media is calculated. For wipe samples, the MDL shall be determined using wipe materials meeting ASTM E1792, "Standard Specification for Wipe Sampling Materials for Lead in Surface Dust", or with wipe materials meeting specifications issued by EPA (reference EPA publication," Interpretive Guidance for the Federal Program TSCA Sections 402/403", March 14, 2002 and/or subsequent EPA published guidance). The spiked media is at an estimated concentration between the actual MDL and 10X the actual MDL. The MDL is the product of 3.14 times the calculated standard deviation for 7 replicates. Under ideal conditions, the MDL should be about one-fifth the practical and routinely achievable detection level that can be reported with relatively good certainty that any reported value is reliable.

All data points produced in a MDL study must be used in the calculation, unless: 1) a point is a statistical outlier (outside 99% confidence limits), 2) a point (or a whole run, if a multiple point run, as with organics or metals analyses) is eliminated if suspect (e.g. incomplete analysis due to a leak or spill). Reasons for elimination must be documented.

MDL studies are conducted over a three-day time period. If the method has an extraction procedure to it, then it must be extracted over a three-day time period, and then analyzed over a three-day time period. MDL studies are run annually for all drinking water methods and all lead analyses. The wet chemistry department and the metals departments run MDL studies annually. The organics department, if it is not a drinking water method, will run a one-time MDL study and repeat if conditions change, receive new instrumentation or new methodology occurs.

The vendors that are selected to use are for various reasons, including some of the following reasons:
1- Pricing (Some offer Discounts)
2- Availability of Product
3- Good History

4.11 Chemical Control Program

The control of incoming reagents and chemicals is accomplished through the use of a Reagent/Standard Log. The logbook contains date received, receiver, laboratory lot number, reagent description, and amount. When packages are received, the analyst checks the packing slip against the material in the package. Discrepancies are reported to the Administrative Supervisor.

If the material is accepted, the packing slip is signed and the container labeled with the date of receipt and when applicable, the expiration date.

Traceability of standards and standard materials are also documented. Tracking of standards (stock, intermediate or working) is accomplished by the use of a Standard Log or Element. Certificates of analysis for purchased standards and information on laboratory manufactured standards are also kept in either the Standard Log or Element.

The laboratory maintains reagent grade type, deionized water, using a Nano-pure water system. This water is available for use in reagents and standards as well as in analysis determinations.

The quality of the reagent water is tested routinely. It must meet the following criteria for microbiology testing (as defined in Table 9020I of SM 9000):
NOTE: If all media is purchased the following tests do not need to be conducted as the reagent water is not utilized.

TEST	MONITORING FREQUENCY	LIMIT
Chemical Tests:		
Conductivity	Continuously, or with each use	>0.5 mega ohms resistance, or <2 umhos/cm at 25°C
pH	With each use	5.5 - 7.5
Total Organic Carbon	Monthly	<1.0 mg/L
Heavy Metals, single (Cd, Cr, Cu, Ni, Pb, and Zn)	Annually (or more frequently if a problem arises)	<0.05 mg/L
Heavy Metals, totals	Annually (or more frequently if a problem arises)	≤ 0.1 mg/L
Ammonia/Organic Nitrogen	Monthly	< 0.1 mg/L
Total Residual Chlorine	Monthly, or with each use	<DL
Bacteriological Tests:		
Heterotrophic Plate Count (SM 9215)	Monthly	<500 CFU/mL (Per 310 CMR 42.08(5)(c)(12d)

Microbiology Media must have both positive and negative cultures analyzed for each new low number, to determine performance compared to a previously acceptable lot. "Quanti-Cult" kits (Idexx/Remel) are used, and contain the following organisms:

Escherichia coli	(T. coliform + and E. coli +)
Klebsiella pneumonia	(T. coliform + and E. coli -)
Pseudomonas aerugonosa	(T. coliform – and E. coli -)

Industrial Hygiene sampling media lots (filters, wipes, tubes) need to be tested prior to analysis, to ensure there is no contamination. Results of such testing must be maintained in each department.

Industrial Hygiene sampling media are supplied to the client, who is responsible for collecting samples.

4.12 Laboratory Environment

Calibration and testing occur only within the laboratory, designed, built and maintained as laboratory space. All spaces are temperature and humidity controlled.

Electronic balances are located away from drafts and doorways and mounted on marble slabs in areas where their use would be affected by vibrations. Biological work areas are sterilized between uses. Neighboring test areas of incompatible activities are effectively separated. Specific work areas are defined and access is controlled. (Only authorized laboratory personnel and escorted visitors may enter the work area). Housekeeping is a major concern for the laboratory. Each employee is required to act in a manner that promotes neatness and cleanliness in following Good Laboratory Practices. All work areas are to be free of clutter and possible contaminants. The importance of maintaining clean work areas cannot be overemphasized. Smoking is prohibited inside the building. Work areas include entries to the laboratory, sample receipt, sample storage, laboratory analysis, chemical and waste storage, and data handling and storage.

All equipment, reference standards, and reference materials required for the accredited tests are available in the laboratory. Records are maintained for all equipment, reference standards, and reference measurement materials, and services used by the laboratory. Reference materials traceable to national standards of measurement or to national standard reference materials are stored away from heavy use areas or major equipment that may affect the proper operation of the materials. Refer to manufacturer's instructions for the procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment, reference materials, and reference standards to ensure proper functioning and in order to prevent contamination or

deterioration. Certificates of Traceability are available for the reference thermometer and the Class S weights. The reference materials and standards are used only for calibration to maintain the validity of performance.

4.13 Control of Contamination

Lead Wipe Sampling: Housekeeping shall be adequate to prevent contamination of samples (Dust, paint, etc.). Work areas have non-porous coatings to eliminate the possibility of prolonged counter contamination. In order to determine surface levels of lead in the Metals laboratory and therefore avoid possible contamination of samples, wipe sampling is performed on a regular basis. If any sample displays contamination (defined as a detected result at the reporting limit), clean area, re-sample, and re-test the contaminated site. Refer to SOP Internal Wipe Sampling, Document Number 32.

4.14 Total Quality Management (TQM)

Quality and turnaround ultimately determine the satisfaction of customers. Con-Test is not merely striving to meet client expectations but to constantly exceed them. Total Quality Management is an effective strategy for success by involving the entire resources of the organization. By tracking quality, educating and empowering employees, quality concerns can be addressed quickly and efficiently, while providing an opportunity for tomorrow's leaders to come forward.

Each project represents a problem to solve or an opportunity for improvement. The key to a quality improvement project is that the problem is scheduled for investigation and resolution. Quality Improvement follows the Define, Measure, Analyze, Implement, and Control Model.

4.15 Review of Requests, Tenders, and Contracts

Contract review is a primary function and integral part of the quality system at Con-Test. All contracts are reviewed and accepted only if the requirements are clearly understood, and the company has the capability and capacity to fulfill client expectations. Communication is maintained with the client from the time a request is processed through commencement of work. This includes informing the client of any deviation from the contract and obtaining approval to beginning testing. If a contract needs to be amended after work has commenced, it will be communicated to the client and the contract will be amended with a hand-filled in correction.

All new work is initiated by the Laboratory Management, delegating responsibilities for new work according to available resources. The staff meets prior to initiation of new work in order to determine if appropriate facilities and resources are available. The plan for any new testing shall be reviewed and approved by the Laboratory Director before commencing such work. After agreement is reached, facilities and resources are organized to efficiently perform the work. For any new testing requirements, the designated employee shall write a standard operating procedure based upon the

appropriate reference method and demonstrate capability to perform those tests prior to reporting results. The SOP(s) shall be under document control and Demonstration of Capability Statement(s) must be on file. See SOP Review of Requests, Tenders, and Contracts, Document Number 29.

5.0 Information Management (Network Design/LIMS)

For more efficient tracking of samples, processing of analysis data, and document control, Con-Test currently employs a Laboratory Information Management System or LIMS as well as a PC network. These systems provide authorized personnel with fingertip access to analysis information as well as user access to valuable organizational programs and applications.

By utilizing a media in which quality of data is easily controlled, the speed, efficiency and accuracy with which laboratory data is delivered is maximized.

5.1 Con-Test LIMS Definition:

The Con-Test LIMS is a file server PC Network based database that contains all information related to an analytical job that is received at the laboratory. All functions, including log-in, data transfer from instruments, billing, report generation, quality control and archive maintenance are handled by the system. The major benefits of the system are rapid report generation, standardization of report format, and minimization of human error due to inaccurate calculations, data transcriptions and misspellings.

Client specific information regarding fees, invoice history, and address are maintained in tables in the relational database. After an analysis passes quality control inspection, if all tests ordered at log-in for the job have been entered into the database the reports will be generated automatically by the LIMS. Reports are standardized in that long lists of compounds do not need to be reentered via a word processor with each analysis. Standard report elements including method references and limits of detection are maintained in files or tables that the report generator accesses. Invoice, Certificate of Analysis, Data Tabulations, and Quality Control Summary are generated at the same time and routinely are mailed together.

Data that is generated by computerized analytical instruments including Gas Chromatographs (GC), Gas Chromatograph/Mass Spectrometers (GC/MS), Inductively Coupled Plasma – Mass Spectroscopy (ICP/MS), and Inductively Coupled Plasma (ICP), is automatically transferred from the instrument into the database where the data is reviewed and edited by the analysts, only if necessary (ex. wrong file transferred). Any calculations that are required to determine the final analytical result are performed by the database and reviewed by the analyst. Transcription errors between instrument and report as well as calculation errors are virtually eliminated by this process.

All data relevant to an analytical job is maintained active on the file server for at least one year. After this time data is archived onto tapes from which it can be restored as needed. Hard copies of data including chain-of-custody documentation are maintained for 10 years off-site at a records maintenance facility.

5.2 Verification of Formulas and Automated Computations

Extensive program validation is performed before the use of any automated system. Automated computations and systems are programmed and thoroughly reviewed by professionals and not utilized until the system has undergone and satisfactorily completed an extensive validation process in order to ensure accurate generation of data. Formulas for automated computations are verified initially and then locked so they cannot be changed.

6.0 Sample Control and Management

6.1 Laboratory Couriers (Transportation of Samples)

Con-Test provides sample pick up and laboratory transportation service for regular clients with certain geographical and sample size limitations. This service is only available upon approval by Con-Test sample control personnel.

At the time of pick up, complete and proper documentation (chain of custody forms) must be signed and turned over to Con-Test Couriers.

6.2 Laboratory Sample Custody

6.2.1 Chain of Custody

Chain-of-custody documentation must be maintained for each transfer of sample. All individuals who handle samples will be required to sign and date paperwork.

6.2.2 Sample Receipt and Inspection

The laboratory receives samples by mail, courier pick-up, and by personal delivery. When samples arrive at the laboratory, the laboratory courier or client relinquishes custody of samples to the sample custodian with proper documentation of the transfer recorded on the chain of custody form.

Chain of custody documentation is required with all samples. The samples are then removed from the shipping or transportation containers and visually inspected for damage such as leakage, breakage, or contamination by one of the log-in staff.

The samples received are then compared with accompanying custody and analysis specification forms to make sure that the paperwork agrees with the labels on each sample container. The pH is taken on applicable samples and noted on the sample receipt checklist. Clients are encouraged to include a temperature blank in the cooler when samples are transported to the laboratory. Sample coolers that are carried by the laboratory couriers will contain a bottle of water that is used to monitor the temperature of the cooler. In all cases when a temperature blank is present, the temperature is recorded on the chain of custody and sample receipt form. If a temperature blank is not in the cooler, an Infra-Red thermometer gun is used to record the sample temperature. The accuracy of the Infra-Red thermometer gun must be verified quarterly. In cases where the temperature is not actually measured for any reason, a comment is put on the chain of custody form as to whether the samples were cold or at ambient temperature when received. Sample receipt form is documented with the procedure used for temperature measurement.

If samples are damaged or do not agree with the paperwork, then the Project Chemist is notified at once, and the appropriate action, listed below, is taken immediately to remedy the situation.

- Samples that are damaged upon receipt at the laboratory are immediately reported to the client so that a decision can be made by the client to void that particular sample or replace it.
- Incomplete sampling information on sampling sheets is brought to the attention of the client.
- Missing samples or missing paperwork is also brought to the attention of the appropriate person.
- Clients are advised of missed holding times and improper containers, temperatures, preservatives or the like.

In any case, clients shall be immediately notified of deficiencies or deviations for possible resolution. Decisions or comments made by the laboratory or client are documented on the chain of custody for future reference.

See below for Sample Acceptance Policy

Sample Acceptance Policy

Con-Test Analytical Laboratories' Sample Acceptance Policy is based on the requirements outlined in the NELAC standard. Samples not meeting the acceptance criteria will not be accepted by the laboratory or will be qualified on the final report. This policy will be available to clients along with sampling instructions, on-line on our company website www.contestlabs.com, and in our Quality Manual.

All samples submitted to Con-Test Analytical Laboratories must:

1) Be accompanied by a chain of custody with proper, full and complete documentation, including sample identification, location, state sample was collected in, date and time of collection, the collector's name, type of preservation (if any), type of sample (matrix), any special comments concerning the sample, tests requested, and desired turn-around time. It is the client's responsibility to communicate specific methods or required detection limits.

2) Be labeled appropriately with a unique sample identification written with indelible ink on water resistant labels. If the laboratory cannot determine identity of a sample, it will be rejected and the client will be contacted for further instructions or re-sampling.

3) Be in an appropriate sample container. If the container is inappropriate, the client will be contacted for further instructions or re-sampling. If analysis is possible, the final report will be qualified. Samples must be appropriately sealed to prevent leakage or cross-contamination.

4) Samples should be shipped in a manner to preserve the sample's safety, quality, and integrity. It is the client's responsibility to ship samples to the lab at the appropriate temperature for sample preservation. Sample temperature will be monitored upon receipt.

5) Adhere to specified holding times. If samples are received past the holding time or will expire before the analysis can commence, the client will be notified and asked how to proceed. If the samples are analyzed, they will be qualified in the final report.

6) Contain adequate sample volume to perform the necessary testing. If sufficient volume is not present, the client will be contacted for further instruction or re-sampling.

If samples show signs of damage, contamination or inadequate preservation, the client will be contacted. If analysis is performed, the final report will be qualified. If analysis can't be performed the client will be notified for further instructions or re-sampling.

6.2.3 Assignment of Laboratory Numbers

Each sample that meets the minimum acceptance requirements for receipt by the laboratory is assigned a unique identification number.

Laboratory sample numbers begin with the last two digits of the year in which the sample was logged in. Then, follows a letter which corresponds to the month the sample was received. "A" = January, "B" = February, "C" = March, etc....

The following four-digit number specifies the work order number, followed by the 2-digit individual sample identification (15A0000-00) which is assigned in ascending order depending on the day and time of receipt.

6.2.4 Internal Sample Tracking & Analysis Scheduling

After assigning individual laboratory identification number the sample custodian records the appropriate information on the chain of custody. The sample custodian then enters the information for each sample into the Laboratory Information Management System (LIMS).

This includes but not limited to; requested analysis, sample ID, log-in date and time, submitter ID, laboratory due date and priority, date sampled, sample matrix, container, preservative, date received, receiver, and other appropriate laboratory identifications.

Upon completion of data entry, the LIMS generates the appropriate forms which are utilized to initiate and track the samples through the laboratory process. The original chain of custody record is scanned into LIMS so it may be viewed by analysts to get required information. The original chain of custody is then attached to a cover sheet and forwarded to a project manager for review.

A work order summary is generated by the LIMS for each work order to ensure tests have been logged in correctly. The work order summary is compared to the chain of custody for each sample.

Samples are then transferred into the appropriate laboratory section, preserved if necessary, and moved into one of the sample storage area refrigerators (pending analysis). Samples remain there until the analysis is to be performed. The requested analysis is then scheduled to be performed by the appropriate analyst or supervisor noting the holding time of the samples.

6.2.5 Log Out and Storage of Samples

Samples are stored in defined, secure areas at all times. Samples are removed from pending analysis storage to pending disposal storage when samples have been analyzed and applicable reports completed and issued.

Aqueous samples are stored for a minimum of one month, soil samples for a minimum of two months (all metals matrices for two months) before characterization and disposal or can be returned to clientele upon request. Clients may request longer retention times.

A locked storage area will be provided should the client require secure storage for samples which require special handling due to legal proceedings.

6.2.6 Disposal of Samples and Wastes

Appropriate samples and wastes are characterized and disposed of according to the appropriate Federal, State, & local regulations. Whenever possible, non-hazardous waste is recycled. Hazardous wastes are disposed of through Licensed Hazardous Waste Firms.

7.0 Data: Generation, Verification & Approval, Reports, Reduction & Storage

7.1 Data Generation

Upon notification of the analysis from sample queries, the analyst or technician responsible for the analysis or preparation collects the sample(s) from cold storage and using standard operating procedures, completes the preparation and subsequent analysis under specified, controlled conditions (including the appropriate QA/QC measures). Before the analysis of any sample, it is the responsibility of the project chemist to verify that all information was correctly recorded into LIMS and matches the chain of custody.

Errors which are detected are brought to the attention of sample custodians and corrected before analysis begins. Upon completion of the analytical run, the analyst or technician makes the appropriate calculations, verifies quality control data, completes bench forms, and any other accompanying paperwork, and organizes the data (logs all information into the specified permanently bound data book or creates a data printout package).

From this point, the analyst/technician enters into the LIMS and batches or selects the samples which were analyzed in their analytical run. The LIMS assigns the group a unique batch identification number which is used for efficient tracking.

Raw data can be entered into the LIMS in two ways. LIMS has the capability of accepting raw data directly downloaded from analytical equipment or excel spreadsheets or if the data is not downloaded, it can be entered manually. Once the raw data is entered, the analyst/technician must enter his/her initials and date of analysis along with any factors associated with the sample, which must be taken into account for calculations to achieve the desired sample result or concentration. This may include sample volume or weight, final volume of preparation, dilution factor, concentration factor, air volume, square feet or the like.

Upon entering all required data, the LIMS performs the needed calculations. The analyst/technician checks the LIMS calculated values for the samples against his/her calculated values to ensure there have been no errors (transcription, calculation etc....). The analyst/technician then saves the data which was previously entered into memory and exits the system. All data is entered into the LIMS. The data and paperwork is then submitted to quality control for approval. When raw data is being evaluated, at least three significant figures is used. Data reported to client gets reported to two significant figures.

7.2 Data verification and Approval

The Quality Control Department is responsible for verifying all data entries before it is released to clients. The initial demonstration of capability (IDOC) represents the validation of the analytical method. After the generation and reduction of data by the analyst/technician, analysis documentation, chromatograms, printouts, and any and all other pertinent data acquired are submitted for Quality Control Review. Data reviewers are specified and trained for each analytical procedure.

Data verification includes examination of calibrations, spike recoveries and sample duplicates against benchmark limits as well as checking for transcription errors and spot-checking for calculation errors. Instrument printouts are also examined and transcriptions verified.

If at any time the data submitted by an analyst/technician does not meet specified quality requirements or is considered questionable, the data is rejected and returned to the analyst/technician for review and reanalysis, if necessary. The new data must be then approved in the same manner. Quality control personnel also verify that appropriate data flags, comments, and narratives are added when needed.

Once these verifications have been made, the data is QC approved in the LIMS and automatically is moved into the report generation phase of the LIMS.

7.3 Report Generation and Management Review of Reports

The Laboratory Information Management System (LIMS) automatically organizes data once it has been approved into several predefined formats dependent on several factors including; the sample type, parameters, and number of samples. Laboratory deliverable packages have been designed to include all required information as well as additional valuable details on the total quality of information. Report formats are easily interpretable because they are provided in a form which is clear and concise. Data is presented in a means which does not require knowledge of statistics or major data manipulations or conversions in order to be easily utilized by data users.

Final reports are first thoroughly reviewed and then signed by designated personnel before release to clients. If samples or reports are involved in litigation, it is the policy of the laboratory to follow the advice and direction of the court regarding records that are subpoenaed or samples that are impounded.

Final test reports contain the following information: The first page contains Con-test's address, fax and phone number, client name, client address, project location, client job number, project number, laboratory work order number, signature of project manager, and report date. The next section contains PO number (if applicable), summary of analyses found in report along with client sample ID, laboratory ID # and matrix, case narrative summary, signature of person signing off the report with the following statements that include: "The results of analyses reported only relate to samples submitted to the Con-test Analytical Laboratory for testing" and "I certify that the analyses listed above, unless specifically listed as subcontracted, if any, were performed under my direction according to the approved methodologies listed in this document, and based upon my inquiry of those individuals immediately responsible for obtaining the information, the material contained in this report is, to the best of my knowledge and belief, accurate and complete". Then the results for each test are given, which include results, RL, MDL (if applicable), units, dilution factor, any data flags, method, date prepared, date/time analyzed, analyst, project location, date received, field sample #, sample ID #, sample matrix and date sampled. The next section of the report contains sample extraction data. This includes for each test method that is applicable, the lab ID, batch number, initial volume, final volume, and date prepared. Next is the Quality Control Section which includes for each batch for each test method the results for the Blank, LCS, LCS Dup, sample duplicates, matrix spikes, and any other reported QC that is applicable to the analysis being performed. This section is followed by the Flag/Qualifier summary section which gives the definition of each data flag used in the final test report. The next section is the Certifications summary which states for each compound/analyte found in the report, what states we are certified for that particular method. Then we have a listing of all certifications/accreditations we hold and when each expires along with the certification number. Lastly, the chain of custody and sample receiving checklist are included. Each report is paginated along with work order ID and final test report date and time, so that each page is easily identifiable.

7.4 Procedures and Format for Reporting Data to State, Local, and/or Federal Officials

Deliverables can include complete state and federal regulatory compliance forms, if required. All data reported is organized in a standard laboratory format unless otherwise requested, specified, or required by client and/or by a governing agency such as the United States Environmental Protection Agency or State Department of Environmental Protection. Con-Test's general analysis format includes all information required by Laboratory certifying agencies.

In certain situations, {such as reporting results for Agency Proficiencies or under the Safe Drinking Water Act (SDWA)} special forms are required for the reporting of data. The format dictated by the applicable forms is completed by the Laboratory and submitted to the appropriate individual or organization. Records of the results in the required formats are archived as normal formatted data.

7.5 Data Reduction

The following equations are commonly utilized in the reduction of analysis data:

Precision Chart Limit Calculations:

a) Calculate R

$\bar{R} = R/n$ where: n = total number of R values

b) Determine the Standard Deviation (S_R) for R

$$S_R = \frac{(R - \bar{R})^2}{n-1} \text{ for } n < 25$$

c) Calculate the Upper Control Limit for R

$UCL_R = \bar{R} + 3\,(S_R)$

d) Calculate the Upper Warning Limit for R

$UWL_R = \bar{R} + 2\,(S_R)$

Accuracy Chart Limits Calculations:

a) Calculate X

$X = X/n$ where: n = Total number of X values

b) Determine the Standard Deviation (S_X) for X

$$S_X = \frac{X - Xi)^2}{n-1} \quad \text{for } n < 25$$

use n in place of n-1 for n >/= 25

c) Calculate the Upper Control Limit for X

$UCL_X = X + 3\ (S_X)$

d) Calculate the Upper Warning Limit for X

$UWL_X = X + 2\ (S_X)$

e) Calculate the Lower Warning Limit for X

$LWL_X = X - 2\ (S_X)$

f) Calculate the Lower Warning Limit for X

$LCL_X = X - 3\ (S_X)$

Percent Recovery Calculation:

The following equation is used to compute percent recovery (%R). The value of %R is then compared to the laboratory established control limits to determine bias and associated interferences.

$\%R = (x1 - x2)/x3 \times 100$

Where:
x1 = measured value for spiked sample
x2 = measured value for un-spiked sample
x3 = known value of the spike in the sample

Relative Percent Difference Calculation:

The following equation is used to calculate Relative Percent Difference (RPD):

RPD = [V1 – V2]/ V3 x 100

Where: [V1 – V2] = Absolute difference between the two values
V3 = Average of the two values

Range: Based on Recovery

Recovery Value for X_1 = $\frac{X_{1R}}{X_{1S}}$

Recovery Value for X_2 = $\frac{X_{2R}}{X_{2S}}$

Range, R = [$X_1 - X_2$] [Absolute Value]

Where: X_{1R} = mg reported
X_{1S} = mg spiked on media
X_{2R} = mg reported
X_{2S} = mg spiked on media

Mean Recovery Value, X = $\frac{X_1 + X_2}{2}$

External Standard Calibration

The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF). The CF can also be calculated using the concentration of the standard rather that the mass in the denominator of the equation.

CF = Peak Area (or Height) of the Compound in the Standard
Mass of the Compound Injected

Internal Standard Calibration

For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows:

$$RF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

Where:

A_S = Peak Area (or height) of the analyte or surrogate
A_{IS} = Peak Area (or height) of the internal standard
C_S = Concentration of the analyte or surrogate, in ug/L
C_{IS} = Concentration of the internal standard, in ug/L

Note that in the equation above, RF is unit less, i.e., the units from the two area terms and the two concentration terms cancel out. Therefore, units other than ug/L may be used for the concentrations of the analyte, surrogate, and internal standard, provided that both C_S and C_{IS} are expressed in the same units. The mass of the analyte and internal standard may also be used in calculating the RF value.

Linear Calibration Using Response Factors

To evaluate the linearity of the initial calibration, calculate the mean CF (external standard calibration) or RF (internal standard calibration), the standard deviation (SD), and the RSD as follows:

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

$$RSD = \frac{SD}{CF} \times 100 \qquad RSD = \frac{SD}{RF} \times 100$$

Where n is the number of calibration standards and RSD is expressed as a percentage (%). If the RSD of the calibration or response factors is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

Linear Calibration Using Least Squares Regression

The regression will produce the slope and intercept terms for a linear equation in the form:

$y = ax + b$

Where:

y = instrument response (peak area or height
a = slope of the line (also called the coefficient of x)
x = concentration of the calibration standard
b = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99.

In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = \frac{(y - b)}{a}$$

When a weighted linear least squares regression is employed, the regression equation becomes:

$$y = \frac{1}{SD^2}(ax + b)$$

Which may be rearranged to solve for x, the concentration. Using internal standard quantitation, the regression equation is rearranged as shown below:

$$\frac{A_S C_{IS}}{A_{IS}} = aC_S + b$$

Where:

A_S = Area (or height) of the peak for the target analyte in the sample
A_{IS} = Area (or height) of the peak for the internal standard
C_S = Concentration of the target analyte in the calibration standard
C_{IS}= Concentration of the internal standard
a = Slope of the line (also called the coefficient of C_S)
b = the intercept

In calculating sample concentrations by the internal standard method, the regression equation is rearranged to solve for the concentration of the target analyte (C_S), as shown below.

$$C_S = \frac{\frac{A_S C_{IS}}{A_{IS}} - b}{a}$$

Non-linear Calibration

In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed.
NOTE: It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

Thus, non-linear calibration should not be employed for methods or instruments previously shown to exhibit linear calibration for the analytes of interest.

When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if

the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Whereas SW-846 methods employ five standards for a linear (first order) calibration model, a quadratic (second order) model requires six standards, and a third order polynomial requires seven standards.

Most curve fitting programs will use some form of least squares minimization to adjust the coefficients of the polynomial (a, b, c, and d, above) to obtain the polynomial that best fits the data.

The "goodness of fit" of the polynomial equation is evaluated by calculating the weighted coefficient of the determination (COD).

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \frac{(n-1)}{(n-p)} \sum_{i-1}^{n} (y_{obs} - y_I)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

Where:

y_{obs} = Observed response (area) for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration

Y_I = Calculated (or predicted) response at each concentration from the initial calibration(s)

n = Total number of calibration points (i.e., 6 for a quadratic model; 7 for a third order model)

p = Number of adjustable parameters in the polynomial equation (i.e., 3 for a third order; 2 for a second order polynomial)

Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99.

7.6 Data Storage

Con-Test Analytical Laboratory takes a layered approach to ensure the preservation of our hard copy and electronic laboratory records. Hard copy data includes log books, data and analysis books, instrument printouts and raw data. These records are kept in paper form at the laboratory until archiving is required. Throughout the year, hard copy data is transferred to an off-site storage facility. This permanent storage is organized and accessible through coded file boxes.

In most cases, an instrument's raw data has also been electronically stored into our LIMS (Laboratory Information Management System) database. This data is organized and accessible through our database server (SQL2014PRI). Final reports (including chain of custody documents) are also stored on this server. SQL2014PRI, along with our domain controller, benefit from a nightly backup routine (Monday - Friday). This practice is facilitated through the use of tape media. Backup and restoration procedures are guided with the use of current IT standard operating procedures.

At the close of each month, electronic data is preserved onto CD/DVD-ROM (Read Only Memory). By request, preserved CD/DVD-ROM data can be restored for client inquiry or quality control purposes. In all cases, the above-mentioned records will be retained for a period of ten years. The only exception to this rule is Lead and Copper potable water records needs to be stored for a period of 12 years. The hardcopy record is kept for 10 years and the electronic copy is kept for at least 12 years. Clients will be notified prior to facility closing, and records will be transferred according to their instruction.

7.7 Quality Record Storage

Quality records, which consist of internal audits, corrective actions/preventative actions, proficiency testing results, certificates of accreditation, Standard Method books, AIHA-LAP, LLC modules, the TNI standard, other miscellaneous methods, and calibration records for thermometers, balances, weights, spectrophotometers, Eppendorf pipettes, and conductivity meter are kept in the Quality Assurance office. They are stored in file cabinets and book shelves, which the Quality Assurance Officer maintains. Other Quality records, such as individual Initial Demonstrations of Capabilities, personnel training files, and controlled documents are stored in an auxiliary room, in locked file cabinets. These records can only be accessed by the QA Officer and Technical Director.

In all cases, the above-mentioned records will be retained for a period of at least ten years. Clients will be notified prior to facility closing in the event the laboratory will no longer be conducting business, and records will be transferred according to the client's instruction. Refer to Con-Test Analytical Laboratory's "records matrix", controlled document #387, for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records.

8.0 Document Control

All Con-Test Laboratory documentation is carefully controlled by the Lab Director and Quality Assurance Officer. This includes the Con-Test Laboratory Quality Assurance Manual, standard methods, and standard operating procedures. The responsibility for maintaining and approving documents falls directly upon the Lab Director and Quality Assurance Officer. Under the authority of top management, it is required that all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work.

After final approval of documentation by the Laboratory Director, the documents are placed in the controlled document program, distributed, and made readily available, to those individuals and/or companies whom those changes affect directly or indirectly. Clients receive "Non-Controlled" copies. All documents are password protected and are "Read-Only". All documents are uniquely identified, including a controlled document number, date, revision number, page numbering, the total number of pages, and the issuing authorities.

The Controlled Document Program is described in detail in the "Controlled Document" SOP.

To ensure utilization and proper representation of the Laboratory Quality Assurance Program, the QA Manual is reviewed, updated as needed, and then approved by the management annually. Interim additions or revisions may be affixed as the occasion arises. Also, included on the master list of controlled documents are all logbooks, manuals, checklists, instructions, chains of custodies, and guidance documents.

8.1 Availability to Laboratory Personnel

The Con-Test Laboratory Quality Assurance Manual, Standard Methods and Procedures are in the controlled document program and are available to all personnel on the F: Drive/Administration/QC1/non-controlled documents in pdf format Folder. Under the authority of top management, it is required that all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work.

8.2 Client Availability

Con-Test documents are updated and revised on a regular basis to reflect current procedure and policy. Those changes or revisions are readily available to clients upon request and/or in periodic client updating.

8.3 Corrections to Documents and Data

Log books, forms, data sheets, and chains of custody are formal laboratory records and need to be treated as such. Records shall be made in indelible ink, black is preferred. There are to be no omissions in the data. Erasures, "white-outs", removal of pages, and scribbling over are not acceptable ways of correcting errors.

Corrections should be kept to a minimum by exercising caution when transcribing data. Unfortunately, errors cannot be avoided completely and when they occur, they should be corrected according to the following procedures:

- Draw a single line through the incorrect entry, insert the correct entry into the closest space available and initial and date the correction.
- Groups of related errors on a single page should have one line through the entries and should be initialed and dated with a short comment on reason of deletion of data.

In order to establish a clear audit trail and to avoid any uncertainty about how and why specific procedures were followed in the laboratory, when a run is repeated or something occurs that is not routine, a note explaining this must be made on the cover sheet or bench sheet.

8.4 SOP Revision, Adoption of New Procedures & Departure from Existing Procedures

Standard Operating Procedures are reviewed and updated to reflect current methodology and procedure on at least an annual basis. SOP's are typically laboratory derivations of approved methodology. (Ex. Standard Methods, SW-846, EPA, NIOSH, and ASTM methods) Laboratory methods are continuously monitored for durability and credible agency endorsement.

Periodically methods may be reevaluated with support or approval revoked or other methods could be deemed acceptable alternatives.

Methodology is to reflect current needs and whenever possible it should be approved by a reputable organization. New methods or procedures may be adopted as necessary. Interim procedures may be appended to documents on approval by laboratory management.

Departures from documented procedures must be approved by management and/or Quality Control Department, depending on the nature of the departure. Documentation of variance from procedure should be on data and in the final report to the client, if the data is affected by the variance. The chain-of-custody form and laboratory bench sheets may also need to contain the documentation in some cases.

8.5 Complete Listing of Standard Operating Procedures

Note: for current revision and dates of review see master list of controlled documents, maintained by the QA department and available upon request.

Wetchemistry

SOP	Document #	SOP Title
SOP Autoclave	88	Autoclave Procedure
SOP ALK	2	Total Alkalinity
SOP NH3NESS	3	Ammonia
SOP MBAS	56	Anionic Surfactants as MBAS
SOP Balance-CAL	6	Balance Calibration
SOP BOD	47	Biological Oxygen Demand (BOD)
SOP COD	55	Chemical Oxygen Demand (COD)
SOP Chloride	58	Chloride
SOP TRC/FRC	43	Chlorine, Total and Free Residual
SOP Color	18	Color
SOP COND	44	Conductivity
SOP Cyanide	59	Cyanide
SOP DO	98	Dissolved Oxygen
SOP Dust	37	Dust, Total & Respirable
SOP Felron	92	Ferrous Iron
SOP FOG1664	93	Method 1664B
SOP Flashpoint	60	Flashpoint, Pensky-Martins Closed Cup Method
SOP Fluoride	17	Fluoride
SOP Glassware Wetchem	71	Glassware Washing: Wet Chemistry Dept.
SOP HARD	63	Total Hardness
SOP Ignitibility	103	Ignitibility

SOP Cr+6	70	Hexavalent Chromium
SOP MICRO	96	Microbiological Analysis of Water
SOP NO3NO2	4	Nitrate/Nitrite – Nitrogen
SOP NO2	42	Nitrite – Manual Method
SOP NO2Lachat	388	Nitrite by Lachat SM4500 NO3-F and Lachat 10-107-04-1-A
SOP ODOR	91	Odor
SOP Paint Filter	126	Paint Filter by Method 9095B
SOP pH	64	pH
SOP Phenol	65	Total Phenolics
SOP Phos	10	Phosphate, Total & Ortho
SOP RXT	87	Reactivity
SOP SOLPER	7	Solids – Percent Solids (Total Solids in Solid and Semisolid Samples)
SOP SOLVOL&FIX	115	Solids – Volatile Solids – Fixed Solids
SOP SOLPER VOL/FIX	8	Solids – Percent Volatile Solids/Fixed Solids (Fixed & Volatile Solids in Solid & Semisolid Samples)
SOP SOLSETT	24	Solids – Settleable Solids
SOP TDS	23	Solids – Total Dissolved Solids
SOP TS	21	Solids – Total Solids
SOP TSS	5	Solids – Total Suspended Solids
SOP Sulfate	66	Sulfate
SOP Sulfide	67	Sulfide
SOP TKN	68	Total Kjeldahl Nitrogen (TKN)
SOP TURB	69	Turbidity
SOP TOC3510B	99B	Total Organic Carbon – Method SM 5310B
SOP TOC Solid	376	Total Organic Carbon in Solid Samples by SW-846 9060A and Lloyd Kahn
SOP ORP	273	Oxidation Reduction Potential (ORP)
SOP Persulfate	275	Persulfate Anion (Groundwater)
SOP Pipet	11	Pipette Washing Protocol

Metals Department

SOP	Document #	SOP Title
SOP DIGIPREP	57	Digiprep Jr. Digestion Apparatus
SOP Glassware Metals	28	Washing Glassware Standard Procedure
SOP TurbMet	16	Metals Turbidity Screening Determination
SOP ICP200.7	22	ICP (Inductively Coupled Plasma Optical Emission Spectroscopy, 200.7, Potables and Wastewaters)
SOP ICP6010	72	ICP (Inductively Coupled Plasma Optical Emission Spectroscopy, 6010C, Non-Potables and Solids)
SOP AirsMetals	40	Metals in Air
SOP 3050B	29	Acid Digestion of Solid Materials (Soils, Sediments, Solids, Sludge/Wipes/Lead in Paint)
SOP 3051 MetalsMicro	135	Method 3051A: Microwave Assisted Digestion of Soils, Sediments, Sludge's, and Oils
SOP INT Wipe	32	Internal Wipe Sampling
SOP Hg	27	Mercury (Cold Vapor Technique) EPA 245.1, SW-846 7470A/7471B
SOP Hg in Air	131	Mercury in Air – Method NIOSH 6009
SOP MetalsWaters	39	Preliminary Treatment for Water Matrix Metals
SOP 200.8	112	ICP-MS EPA 200.8
SOP 6020A	113	ICP-MS SW-846 6020A
SOP Dissolved Metals Prep	394	Dissolved Metals Prep
SOP MetalsAirFilter	247	Determination of Metals in Suspended Particulate Matter (40 CFR App G) Air Filter
SOP IC300.0	73	Determination of Inorganic Anions by IC (Method EPA 300.0)

Organics/Air Lab

SOP	Document #	SOP Title
SOP RSK-175	140	Sample Prep and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibrium Technique
SOP Glassware/Ext	97	SOP for Washing Organics/Extractions Glassware
SOP CIP	86	Chromatographic Integration Procedures
SOP 504.1	75	EPA 504.1: 1,2-Dibromoethane (EDB) & 1,2-Dibromo-3-Chloropropane (DBCP)
SOP 524.2	34	Volatile Organics by GC/MS (Method EPA 524.2)
SOP 602	116	Volatile Organics by GC (Method EPA 602)
SOP 608	33	Organochlorine Pesticides & PCB's (EPA 608)
SOP 624	35	Volatile Organics by GC/MS (Method EPA 624)
SOP 625	20	Semi-Volatile Organics (Method EPA 625)
SOP PM-10PM2.5PEM	250	Determination of Particulate Matter as PM-10, PM-2.5, and IP-10A – Determination of Fine Particulate Matter in Indoor Air Using Size Specific Impaction
SOP PCB OIL	26	PCB's in Oil (Method EPA 600/4-81-045)
SOP 8082	51	Polychlorinated Biphenyls (PCB's) by GC Method SW-846 8082A
SOP 8081	53	Organochlorine Pesticides by GC Method SW-846 8081B
SOP 8260	50	Volatile Organics by GC/MS (Method SW-846 8260 B/C)
SOP 8270	20	Analytical Analysis of Semi-Volatile Organics (Method SW-846 8270D)
SOP Method3C	80	EPA Method 3C – Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources
SOP TO13A	77	Compendium Method TO-13A – Determination of Polycyclic Aromatic Hydrocarbons (PAHs) In Ambient Air Using GC/MS
SOP TO14A	46	Compendium Method TO-14 – Determination of Volatile Organic Compounds (VOC's) in Air Collected in Specially Prepared Canisters and Analyzed by GC/MS
SOP TO15	45	Compendium Method TO-15 – Determination

		of Volatile Organic Compounds (VOC's) in Air Collected in Specially Prepared Canisters and Analyzed by GC/MS
SOP TO17	49	Compendium Method TO-17 – Determination of Volatile Organic Compounds (VOC's) in Air Using Active Sampling onto Sorbent Tubes and Analyzed by GC/MS
SOP APH	110	Air-Phase Petroleum Hydrocarbons by GC/MS Method MADEP APH
SOP 3510CWaterExt	403	Water Extraction Procedure Method SW-846 3510C
SOP 3546 Microwave	100	Method 3546 Microwave Extraction Procedure
SOP 8015_8100	25	Total Petroleum Hydrocarbons(GC/FID)Methods SW-846 8100M/8015C/D
SOP Deter of Form in Air	228	Determination of Formaldehyde Air Collected in Specially prepared Canisters and Analyzed by GC/MS
SOP EPH	102	Analytical Analysis of Extractable Petroleum Hydrocarbons MA EPH by GC/FID
SOP GRO	105	Gasoline Range Organics (GRO) EPA 8015C/D
SOP VPH	101	Volatile Petroleum Hydrocarbons (VPH) Mass DEP VPH Method
SOP TCLP Ext	89	Extraction Procedure for Toxicity Characteristic Leaching Procedure
SOP PCB NIOSH 5503	109	Polychlorinated Biphenyls (PCB's) in Air NIOSH 5503
SOP Can Cleaning	111	Can Cleaning Procedure
SOP CTETPH	54	CT ETPH: Analysis of Extractable Petroleum Hydrocarbons (ETPH) using Methylene Chloride, GC/FID
SOP 8151A	255	Chlorinated Herbicides by Gas Chromatography Using Methylation Derivatization SW-846 8151A
SOP Florisil 3620C	251	Method 3620C Florisil Cleanup of Pesticides
SOP Copper Cleanup	395	Removal of Sulfur via Activated Copper SW-846 3660B
SOP TVOC	120	Determination of Total Volatile Organic Compounds (TVOC) in air Collected in specially Prepared canisters and analyzed by GC/MS
SOP 3540CSOXHLET	127	Method 3540C – Soxhlet Extraction Procedure For Polychlorinated Biphenyls (PCB's)
SOP SPLPmod	138	Extraction Procedure for Synthetic Precipitation Leaching Procedure Modified ZHE Procedure for 8260 Analysis
SOP SPLP	137	Extraction Procedure for Synthetic Precipitation Leaching Procedure Method 1311
SOP PCB Homolog	285	SOP for the Determination of PCB Homologues In Air by GC/MS by EPA Method 680

SOP Congener	289	Determination of PCB Congeners by GC/MS
SOP 6200	270	Volatile Organics by GC/MS – Method SM (20th) 6200B
SOP Air Order Completion	279	Summa Canister Air Order Completion
SOP Complaint Logbook Air	280	Complaint Logbook – Air Department
SOP Flow Controller Log Air	281	Flow Controller Logbook – Air Department
SOP Summa and Flow Controller Evaluation	282	Summa Canister and Flow Controller Evaluation Air Department
SOP TO-4A/TO-10A	311	Compendium Method TO-4A/TO-10A by ASE Extraction (Method 3545) and Soxhlet extraction (Method 3540C)
SOP TO-11A	295	Determination of Formaldehyde and Other Carbonyl Compounds in Ambient Air Using Adsorbent Cartridge Followed by HPLC (Compendium Method TO-11A)
SOP ALC/GLY	122	Alcohols/Glycols by GC/FID Method SW846 8015C/D
SOP 1,4-Dioxane8270	314	Low Level 1,4-Dioxane by8270 GC/MS (SIM) Using Large Volume Injection/Programmable Temperature Vaporization Inlet
SOP 1,4-Dioxane8260	121	1,4-Dioxane in water by Heat Purge & Trap, GC/MS (Method 8260B/C Sim)
SOP 8315A	301	Method 8315A: Determination of Formaldehyde & Carbonyl Compounds by High Performance Liquid Chromatography (HPLC) for Liquid & Solid Samples
SOP Airlab TICs	81	Tentatively Identified Compounds in Air
SOP Solvent Distillation	353	Solvent Distillation
SOP Ext Lot Check	396	Extraction Glassware Lot Check
SOP Compositing Samples	347	Compositing Samples
SOP PFAA	434	Determination of Selected Perfluorinated Alkyl Acids)PFAAs) in Drinking Water by Solid Phase Extraction & Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS) Drinking Water method EPA 537.1 and ISO 25101
SOP Buchi Solvent Recovery	382	Buchi Solvent Recovery Testing

General

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SOP Bottle Prep	291	Bottle Prep
SOP Controlled Documents	83	Procedure for Maintaining Controlled Documents

SOP Courier	242	Courier
SOP Chem Receipt	114	Chemical/Reagent Purchase, Receipt, and Storage
SOP CorrActionPrevActions	84	Procedure for Implementing Corrective Actions/Preventative Actions
SOP Haz Waste	108a	Sample Handling, Disposal and Hazardous Waste Handling Process
SOP Water Evap Room	119	Water Evaporation System Operation
SOP Chemical Hygiene Plan	252	Chemical Hygiene Plan
SOP HowToRetrieveFindAnd ReduceElectronicFilesInElement	246	How to Retrieve, Find, and Reduce Electronic Files in Element
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SOP Employee Hiring/ Job Posting	232	Employee Hiring/Job Posting SOP
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9.0 Internal Performance, Systems Audits, Management Review and Corrective/Preventative Actions

Performance and Systems Audits are a valuable tool in evaluating procedures and identifying current and potential problems thus allowing for immediate corrective/preventative actions and "root cause" analyses to begin.

Performance and Systems audits are an important part of monitoring laboratory adherence to established policy and procedure. Various internal performance and systems audits are conducted routinely throughout the year.

Management Review – The overall objectives shall be established, and shall be reviewed during management review. The quality policy statement is issued under the authority of top management. The following overall objectives are found throughout the Quality Assurance Manual, including Section 1.0:

- The laboratory management's commitment to good professional practice and to the quality of testing and calibration in servicing clients
- Con-Test's standard of service
- The purpose of the management system related to quality
- The requirement, that all personnel concerned with testing and calibration activities within the laboratory familiarize them-selves with the quality documentation and implement the policies and procedures in their work.
- The laboratory management's commitment to comply with the ISO: IEC 17025:2005 standard and to continually improve the effectiveness of the management system.

Annually the laboratory's top management shall conduct a review of the laboratory's management system and testing/calibration activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The elements of the review include:

1) The overall objectives as discussed above and in section 1.0
2) The suitability of policies and procedures
3) Reports from managerial and supervisory personnel, including QA Officer's monthly reports.
4) Outcome of recent internal audits
5) Corrective and Preventative actions
6) Assessments by external bodies (audits by MA, AIHA-LAP, LLC, NELAC, client audits)
7) Results of inter-laboratory comparisons or proficiency tests (PT results)
8) Changes in the volume and type of work
9) Customer feedback (client surveys)
10) Complaints
11) Recommendations for improvement
12) Other relevant factors, e.g. QA/QC activities, resources, and staff training

Results from the management review feed into the laboratory planning system and include the goals, objectives and action plans for the coming year. Findings from the management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are carried out immediately and within appropriate time frame. Managerial reviews must include identification and signature of the author as well as be paginated.

Corrective Actions/Preventative Actions are instituted immediately when nonconforming work or departures from policies and procedures in the management system or technical operations have been identified.

9.1 Performance Audits (Internal)

Internal Performance Audits are designed to measure the consistency, efficiency, and proficiency of the laboratory in obtaining the known true value of prepared samples, submitted as analyst blinds, for one or various tests. The Quality Assurance Officer administers performance audits and results are presented to the Laboratory Director for review and possible corrective actions.

9.2 System Audits

System audits are intensive laboratory system inspections evaluating laboratory adherence to approved procedure. These inspections are performed either by internal or external laboratory (regulatory agency) personnel at regular scheduled intervals.

Annually, the laboratory must conduct an internal audit which is compliant with AIHA-LAP, LLC requirements: the purpose of this audit is to verify that laboratory operations continue to comply with the requirements of ISO/IEC 17025:2005 and the AIHA-LAP, LLC program requirements. The latest AIHA-LAP, LLC site assessor's checklist and latest NELAC assessor's checklist shall be used for this internal systems audit, to ensure that all elements are evaluated.

9.2.1 Internal Quality and Management Systems Audit

The Quality Assurance Officer, Laboratory Manager, Laboratory Technical Director or other trained staff may perform internal Quality and Management Systems Audits. All discrepancies and deviations are immediately documented for review and subsequent correction by internal administration and personnel through the corrective action program. It is the intention of laboratory management to perform internal Quality and Management Systems Audits annually.

9.2.2 Internal Method Audits

The Quality Assurance Officer, Laboratory Manager, Laboratory Technical Director or other trained staff may perform internal method audits. All discrepancies and deviations are immediately documented for review and subsequent correction by internal administration and personnel through the corrective action program. It is the intention of laboratory management to audit each method annually. These internal method audits ensure that methods are being followed, SOPs are up to date, each method gets data validated, and also

incorporate data integrity checks. These audits consist of both quality control and quality assurance review.

9.2.3 External Audits

In the maintenance of certification and accreditation, external laboratory agencies require system audits by agency personnel to be performed. Upon issuance of a system audit report by said agencies to the Quality Assurance Officer, the laboratory shall be required to correct cited audit deficiencies. Continued certification and accreditation normally is based upon fulfillment of audit corrective actions to cited deficiencies.

9.2.4 Audit Findings and Corrective Action

Upon completion and issuance of audit reports to the Quality Assurance Officer, audit deficiencies and findings are codified per major laboratory area. The findings are then presented to the Laboratory Director for review, evaluation, and formulation of corrective action implementation strategies.

9.3 Corrective actions/Preventative Actions

Outline for Corrective/Preventative Actions:

1) Discovery/identification
2) Determination of root cause(s) via root cause analysis
3) Identify potential corrective action(s)
4) Choose according to the magnitude and risk of the problem
5) Implement the corrective action(s)
6) Monitor

For more details regarding the Corrective Action/Preventative Action Program, refer to SOP Corrective Actions/Preventative Actions, document number 84.

9.3.1 A problem with the management system or with technical operations may be identified through a variety of activities, including nonconforming work, internal/external audits, QA & management reviews, customer feedback/inquiries, and from staff observations. This is the discovery/identification step of the corrective/preventative action process.

9.3.2 The procedure for corrective action shall start with an investigation to determine the root cause(s) of the problem. Each investigation (root cause analysis) is different based upon the type of nonconformance, complexity of the problem, and range of impact. The points to take in to consideration when doing a root cause analysis are: personnel, samples, methods, controls and data.

For details on each point, refer to SOP Corrective Actions/Preventative Actions, document number 84.

9.3.3 The laboratory documentation and records of all non-conforming events requiring corrective action shall include the determined cause(s) and corrective action taken. Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and prevent recurrence. Corrective actions shall be appropriate to the magnitude and risk of the problem.

9.3.4 After the best corrective/preventative action is chosen, it will be implemented immediately and monitored for effectiveness through follow-up investigations and if warranted put into the policing program. If deemed necessary, an internal audit will be conducted in that area of activity the issue occurred as soon as possible.

10.0 Analytical Methods

The following is a listing of analytical methods commonly utilized by Con-Test Analytical Laboratory. Deviations from tests and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client. Deviations will be noted on the final report.

Please note: This is not a complete listing of analytical method. For information concerning other utilized analytical methods contact one of our project chemists.

Analytical Methodology
Water/Wastewater

Bacteriological Analyses	Method Number	Reference
Coliform, Total	SM 9222B	8
	SM 9223 (Colisure)	8
Coliform, Fecal	SM 9222D	8
Enterococci	SM 9223 (Enterolert)	8
Inorganic Mineral Analyses	**Method Number**	**Reference**
Alkalinity (Titrimetric)	SM 2320 B	8
Chloride (Argentometric)	SM 4500-Cl B	8

Chloride (IC)	EPA 300.0	21
Chromium, Hexavalent	SM 3500-Cr B	8
(Manual, Colorimetric)	SW-846 7196 A	3
Conductivity (Wheatstone Bridge)	SM 2510 B	8
Dissolved Oxygen (Membrane electrode method)	SM 4500-O G	8
Ferrous Iron (Spectrophotometric)	SM 3500-Fe D	8
Fluoride (Ion Selective Electrode)	SM 4500-F C	8
Fluoride (IC)	EPA 300.0	21
Hardness (Titrimetric, EDTA)	SM 2340 C	8
pH (Electrode)	SM 4500-H B	8
	SW-846 9040 B	3
Solids, Total	SM 2540 B	8
Solids, Total Dissolved	SM 2540 C	8
Solids, Total Suspended	SM 2540 D	8
Solids, Settleable	SM 2540 F	8
Sulfate (Turbidimetric)	ASTM D516	15
Sulfate (IC)	EPA 300.0	21
Sulfide (Iodometric Back Titration)	SM 4500-S^2 F	8

Nutrient Analyses	**Method Number**	**Reference**
Ammonia-N	SM 4500-NH_3 C -titration	8
Nitrite-N (Manual Spectrophotometer)	SM 4500-NO_2 B	8
Nitrite (IC)	EPA 300.0	21
Nitrite (Lachat Auto analyzer)	SM 4500-NO_3 F	8
Nitrate/Nitrite (Lachat Auto-analyzer)	SM 4500-NO_3 F	8
Nitrate (IC)	EPA 300.0	21
Nitrate-N (Cadmium Reduction Lachat)	SM 4500-NO_3 F	8
(NO_3 + NO_2) – NO_2		

Total Kjeldahl Nitrogen (Organic N) (Titrimetric)	SM (19-21) 4500-N_{org} B, C	8
MBAS (Surfactants) (Colorimetric)	SM 5540 C	8
Phosphate, Ortho (Colorimetric)	SM 4500-P E	8
Phosphate, Ortho (IC)	EPA 300.0	21
Phosphate, Total (Colorimetric)	SM 4500-P E	8

Demand Analyses	**Method Number**	**Reference**
BOD (5 day) (Dissolved Oxygen Consumption)	SM 5210 B	8
CBOD (5 day) (Carbonaceous DO Consumption)	SM 5210 B	8
COD (Colorimetric)	EPA 410.4	1
Chlorine, Total Residual (Colorimetric)	SM 4500-Cl G	8
TOC (Total Organic Carbon)	SM 5310 B	8

Physical Analyses	**Method Number**	**Reference**
Color (Visual Comparison)	SM 2120 B	8
Odor (Threshold Odor)	SM 2150 B	8
Turbidity (Nephelometric)	EPA 180.1	1

Other	**Method Number**	**Reference**
Bromide (IC)	EPA 300.0	21
Oil and Grease (FOG) (Hexane Extraction)	SW-846 1664B	22
Phenols (Colorimetric)	EPA 420.1	1
Cyanide, Total (Manual Spectrophotometer)	SM 4500-CN E	8
	SW-846 9014	3
Physiologically Available Cyanide (Manual Spec.)	SW-846 9014	16

Volatile Organics	Method Number	Reference
Purgeable Aromatics (GC)	EPA 602	4
Purgeables (GC/MS)	EPA 624	4
Drinking Water Purgeables (GC/MS)	EPA 524.2	5
EDB/DBCP	EPA 504.1	5

Semi-Volatile Organics	Method Number	Reference
Base/Neutrals & Acids	EPA 625	4
Priority Pollutants Pesticides/PCB's	EPA 608	4
CT Extractable Petroleum Hydrocarbons	CT ETPH	12
MA Volatile Petroleum Hydrocarbons	MA VPH	13
MA Extractable Petroleum Hydrocarbons	MA EPH	14

Other Organics	Method Number	Reference
PFAA's (LC/MS/MS)	EPA 537	23
PFAA's (LC/MS/MS)	ISO 25101	24

Metals Analyses

Waters, soils and other materials may be analyzed for metals by Inductively Coupled Argon Plasma – Atomic Emission Spectroscopy (ICP) and/or Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Non-aqueous samples are generally treated as a solid waste and SW-846 methods are applied.

Analyte	Water/ Wastewater (Ref 3, 18, 20)	Drinking Water (Ref 18, 20)	Non-Aqueous (Solids/Wastes) (Ref 3)
Aluminum (Al)	200.7/6010C+D 6020B	200.7	6010C+D/6020[a]+B
Antimony (Sb)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B

Arsenic (As)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B
Barium (Ba)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Beryllium (Be)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B
Boron (B)	200.7/6010C+C	200.7	6010C+D
Cadmium (Cd)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Calcium (Ca)	200.7/6010C+D	200.7	6010C+D
Chromium (Cr)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Cobalt (Co)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Copper (Cu)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Iron (Fe)	200.7/6010C+B 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Lead (Pb)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B
Magnesium (Mg)	200.7/6010C+D	200.7	6010C+D
Manganese (Mn)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Mercury (Hg)	245.1/7470A	245.1	7471B
Molybdenum	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Nickel (Ni)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Potassium (K)	200.7/6010C+D	200.7	6010C+D

Selenium (Se)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B
Silver (Ag)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Sodium (Na)	200.7/6010C+D	200.7	6010C+D
Thallium (Tl)	200.7/6010C+D 200.8/6020A+B	200.8	6010C+D/6020A+B
Tin (Sn)	200.7/6010C+D	200.7	6010C+D
Vanadium (V)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B
Zinc (Zn)	200.7/6010C+D 200.8/6020A+B	200.7/200.8	6010C+D/6020A+B

Note: EPA Method 200.7 and SW-846 Method 6010C+D are "Inductively Coupled Plasma – Atomic Emission Spectroscopy" (ICP) methods.

Note: EPA Method 200.8 and SW-846 Method 6020A+B are "Inductively Coupled Plasma Mass Spectrometry" (ICP-MS) methods.

Analytical Methodology
Test Methods for Evaluating Solid Wastes; SW-846

Waste Evaluation	Method Number	Reference
Paint Filter Liquids Test	SW-846 9095A	3
Corrosivity (pH solid)	SW-846 9045	3
Flashpoint	SW-846 1010A	3
Ignitability	SW-846 1030	3
Reactivity: Cyanide and Sulfide	SW-846 Chapter 7.3.3.2	3
TCLP (Toxicity Char. Leaching Procedure)	SW-846 1311	3
SPLP (Synthetic Precipitation Leaching Procedure)	SW-846 1312	3
PCB in Oil	EPA 600/4-81-045	17

Total Organic Carbon (TOC)	SW-846 9060A	3
Cyanide	SW-846 9014	3
Hexavalent Chromium (FCr+6)	SW-846 7196A	3

Sample Preparation Methods

Inorganic Prep Techniques	**Method Number**	**Reference**
Acid digestion of Aqueous samples	3005A/3010C	3
Acid digestion for Oils, Greases, and Waxes	3051A	3
Acid digestion of Sediments and Sludges	3050B	3
Microwave Extraction	3051A	3
Organic Prep Techniques	**Method Number**	**Reference**
Separatory Funnel Liquid-Liquid Extraction	3510C	3
Sonication Extraction	3550B	3
Pressurized Fluid Extraction	3545	3
Microwave Extraction	3546	3
Soxhlet Extraction	3540C	3
Organic Analytical Methods	**Method Number**	**Reference**
Priority Pollutant Pesticides/PCB's	8081B/8082A	3
GC/MS Method for Volatile Organics	8260B/C	3
GC/MS Method for Semi-Volatile Organics	8270C/D	3
Fuel Hydrocarbons	8015M	3*
Fuel Hydrocarbons	8100M	3*
GRO and DRO	8015C/D	3
Herbicides	8151A	3

CT Extractable Petroleum Hydrocarbons	CT ETPH	12
Volatile Petroleum Hydrocarbons	MA VPH	13
Extractable Petroleum Hydrocarbons	MA EPH	14

M = Modified * = In-house Standard Operating Procedure

Environmental Lead
Commonly Utilized Methodology

Air	NIOSH 7303, Lead ICP-AES
Paint	SW-846 Modified Method 6010C (3050B), Lead ICP-AES
Dust Wipes	SW-846 Modified Method 6010C (3050B), Lead ICP-AES
Soil	SW-846 Method 6010C (3050B), Lead ICP-AES

Analytical Methodology – Air

Analyte	**Collection Media**	**Method No.** Modified NIOSH
Metals:		
Arsenic (As)	37 mm Cassette w/MCE Filter	7303 (6)
Beryllium (Be)	37 mm Cassette w/MCE Filter	7303 (6)
Cadmium (Cd)	37 mm Cassette w/MCE Filter	7303 (6)
Chromium (Cr)	37 mm Cassette w/MCE Filter	7303 (6)
Copper (Cu)	37 mm Cassette w/MCE Filter	7303 (6)
Lead (Pb)	37 mm Cassette w/MCE Filter	7303 (6)
Nickel (Ni)	37 mm Cassette w/MCE Filter	7303 (6)
Zinc (Zn)	37 mm Cassette w/MCE Filter	7303 (6)
Metals by ICP	37 mm Cassette w/MCE Filter	7303 (6)

Note: Only the most commonly requested Air Analyses Methods are listed. Other analytes and alternative methods are available. Please check with project chemist for more details.

Note: It is required that a blank be submitted for all wipe and air analyses

Analytical Methodology – Air

Analyte	**Collection Media**	**Method No.**
Dust, Total	37 mm Cassette w/PVC Filter	0500 (6)
Dust, Respirable	37 mm Cassette w/PVC Filter	0600 (6)
PCB'S in Air	Florisil Sorbent Tubes	5503 (6)
Hg in Air	Hopcalite Sorbent Tube	6009 (6)
TO-4	PUF	10
TO-10A	PUF	10
TO-13A	PUF	10

TO-14/TO-15	Summa Canister	10
APH	Summa Canister	11
Method 3C (Fixed Gases)	Summa Canister	9
TO-17	Sorbent Tube	10

References

1.0 USEPA – "Methods for Chemical Analysis of Water and Wastes" – EPA 600/4-79-020, Revised 1983.

2.0 APHA – "Standard Methods for the Examination of Water and Wastewater", 17th edition, 1989.

3.0 USEPA – "Methods for Evaluating Solid Waste, Physical/Chemical Methods", 3rd edition, USEPA November, 1986 (SW846), and updates. (Update V, Rev 5, July 2014)

4.0 "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act", 40CFR Part 136.

5.0 USEPA – "Methods for the Determination of Organic Compounds in Drinking Water", EPA 600/4-88/039, December 1988, and updates.

6.0 NIOSH Manual of Analytical Methods

7.0 OSHA Manual of Analytical Methods

8.0 APHA – "Standard Methods for the Examination of Water and Wastewater", 18th, 19th, 20th, 21st, 22nd editions

9.0 EPA Technology Transfer Network Emission Measurement Center. CFR Promulgated Test Methods http://www.epa.gov.ttn/emc/promgate.html

10.0 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.

11.0 Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH). Public Comment Draft 1.0, Massachusetts DEP, ORS, BWSC. February 2000.

12.0 Analysis of Extractable Petroleum Hydrocarbons (ETPH) Using Methylene Chloride GC/FID. University of Connecticut ERI. March 1999.

13.0 Method for Determination of Volatile Petroleum Hydrocarbons (VPH). Massachusetts DEP, ORS, BWSC. Revision 1.1, May 2004

14.0 Method for the Determination of Extractable Petroleum Hydrocarbons (EPH). Massachusetts DEP, ORS, BWSC. Revision 1.1, May2004.

15.0 ASTM, "American Society of Testing and Materials", 2002

16.0 "Method for the Determination of Physiologically Available Cyanide (PAC)", Massachusetts DEP, ORS, BWSC, August 2004.

17.0 "The Determination of PCB's in Transformer, Fluid and Waste Oils", USEPA Method EPA 600/4-81-045, September 1982.

18.0 USEPA Supplement 1 of "Methods for the Determination of Metals in Environmental Samples", EPA 600R-94-11, May 1994, Method EPA 200.7, Rev 4.4. EMMC Version 1994.

19.0 USEPA Supplement 1 of "Methods for the Determination of Metals in Environmental Samples", EPA 600R-94-11, May 1994, Method EPA 245.1, Rev 3.0 EMMC Version 1994.

20.0 USEPA Supplement 1 of "Methods for the Determination of Metals in Environmental Samples", EPA 600R-94-11, May 1994, Method EPA 200.8, Rev 5.4. EMMC Version 1994.

21.0 USEPA "Methods for Determination of Inorganic Substances in Environmental Samples", EPA 300.0 (Determination of Inorganic Anions by Ion Chromatography"), Rev 2.1, August 1993.

22.0 "Method 1664, Revision A: N-Hexane Extractable Material (HEM: Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-Polar Material) by Extraction and Gravimetry", EPA-821-R-98-002; PB99-121949, February 1999.

23.0 EPA Method 537, "Determination of Selected Perfluorinated Alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", Version 1.1, September 2009.

24.0 Method ISO 25101:2009, "Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry", April 30, 2009.

11.0 Sampling and Preservation Requirements

Information shall be available to clients through Con-Test Analytical Laboratory. Con-Test Analytical Laboratory will assist clients in obtaining information regarding recommended procedure, sampling materials, sampling containers, preservatives, and shipping instructions. This includes laboratory request for client submittal of field blanks or blank sampling media. Con-Test will also direct clients to the appropriate agencies (Federal, State, Local Officials, Field Services, and Consulting Services, etc.) or channels when information is unavailable through the laboratory. This information is available upon request, through our project chemists. Multiple tests may be able to be combined in one container as long as sufficient sample amount is submitted or method dictates otherwise. Please consult the laboratory on which ones.

Sampling and Preservation Requirements
Water

Key:

Holding Time = Time allowable between time of sampling and before specified analysis begins.

Parameter = Test	*mL = Milliliters*	*P = Polyethylene Container*
G = Glass Container	*P/G = Either P or G*	*HNO_3 = Nitric Acid*
H_2SO_4 = Sulfuric Acid	*NaOH = Sodium Hydroxide*	*$Na_2S_2O_3$ = Sodium Thiosulfate*

Parameter	Container	Preservative	Holding Time
Alkalinity	100 mL P/G (Needs its own container with no headspace)	Cool to 4°C	14 Days
Ammonia (as N)	100 mL P/G	Cool to 4°C, H_2SO_4 to pH<2	28 Days
Bacteria Tests (T.Coliform) And Enterococci	100 mL P Bacteria cup	0.008% $Na_2S_2O_3$ Cool to 4°C	30 Hours
Fecal Coliform	100 mL P Bacteria cup	0.008% $Na_2S_2O_3$ Cool to 4°C	6 Hours
Biological Oxygen Demand (BOD)	1000 mL P/G	Cool to 4°C	48 Hours
Carbonaceous BOD	1000 mL P/G	Cool to 4°C	48 Hours
Chemical Oxygen Demand (COD)	50 mL P/G	Cool to 4°C, H_2SO_4 to pH<2	28 Days
Chloride	200 mL P/G	None required	28 Days
Chlorine, Residual	100 mL P/G	None required	Analyze Immediately
Color	100 mL P/G	Cool to 4°C	48 Hours
Cyanide, Total and Amenable	500 mL P/G	Cool to 4°C	14 Days[2]

		NaOH to pH>12 0.6g Ascorbic[1]	
PAC	500 mL G	NaOH to pH>12	14 Days
Ferrous Iron	500 mL P/G	HNO_3 to pH<2	6 months (48 hrs. if Unpreserved)
Fluoride	100 mL P	None required	28 Days
Hardness	100 mL P/G	HNO_3or H_2SO_4 to pH<2	6 months
Ignitability	50 mL P/G	Cool to 4°C	ASAP/7 days
Metals 500 mL P(A)/G(A)	HNO_3 to pH<2	6 months	
Chromium, Hexavalent	200 mL, P(A)/G(A) MCP soils need its own container	Cool to 4°C	24 Hours
Mercury	200 mL, P(A)/G(A)	HNO_3 to pH<2	28 Days
TKN	200 mL P/G	H_2SO_4 to pH<2	28 Days
Nitrate/Nitrite as N	50 mL P/G	Cool to 4°C H_2SO_4 to pH<2	28 Days
Nitrate as N	50 mL P/G	Cool to 4°C	48 Hours
Nitrite as N	50 mL P/G	Cool to 4°C	48 Hours
Odor	200 mL G only	Cool to 4°C	Analyze Immediately
Oil and Grease	1 Liter G only	Cool to 4°C H_2SO_4 to pH<2	28 Days
Total Organic Carbon (TOC)	25 mL P/G or 40mL VOA vial	Cool to 4°C H_2SO_4 or HCL to pH<2	28 Days
Orthophosphate	100 mL P(A)/G(A)	Cool to 4°C	48 Hours Field Filtered within 15 minutes
Dissolved Oxygen	300 mL G	None required	Analyze Immediately
pH	30 mL P/G	Cool to 4°C	Analyze Immediately
Total Phenol	500 mL Amber G	Cool to 4°C H_2SO_4 to pH<2	28 Days
Total Phosphate	100 mL P/G	Cool to 4°C H_2SO_4 to pH<2	28 Days
Settleable Solids	1000 mL P/G	Cool to 4°C	48 Hours
Total Solids (TS)	100 mL P/G	Cool to 4°C	7 Days

Total Suspended Solids (TSS)	100 mL P/G	Cool to 4°C	7 Days
Total Dissolved Solids (TDS)	100 mL P/G	Cool to 4°C	7 Days
Specific Conductance (Conductivity)	100 mL P/G	Cool to 4°C	28 Days
Sulfate	250 mL P/G	Cool to 4°C	28 Days
Sulfide	100 mL P/G	Cool to 4°C Add 4 drops of 2N Zinc acetate, NaOH pH>9	7 Days
Surfactants (MBAS)	500 mL P/G	Cool to 4°C	48 Hours
Turbidity	50 mL P/G	Store in a dark place Cool to 4°C	48 Hours

[1] Should only be used in the presence of residual chlorine.
[2]Maximum holding time is twenty-four hours when sulfide is present. Optionally, Sulfide can be removed by the Addition of cadmium nitrate (etc.) powder before preservation until a negative spot test is obtained on lead acetate test paper.

Parameter	**Container**	**Preservative**	**Holding Time**
Volatile Organics (602, 624)	(2) 40 mL VOA Vials w/Teflon lined lid	Cool to 4°C HCL to pH<2, Zero headspace	14 Days
Base Neutral/Acid extractables (625)	(2) Liter amber G, w/Teflon lined lid	Cool to 4°C	7 Days until extraction 40 Days after ext.
Pesticide extraction (608/8081B)	(2) 1 Liter amber G, w/Teflon lined lid	Cool to 4°C pH 5-9	7 Days until extraction 40 Days after ext.
Herbicide extraction	(2) 1 Liter amber G,	Cool to 4°C	7 Days until extraction 40 Days after ext.
EDB/DBCP (504.1)	(2) 40 mL VOA Vials w/Teflon lined lid	Cool to 4°C HCL to pH<2, Zero headspace	28 Days
Polychlorinated Biphenyls (PCBs) (608/8082A)	(2) 1-Liter amber G, w/Teflon lined lid	Cool to 4°C	CT – 7 Days until ext. MA – 1 yr. until ext. 40 Days after ext. NO HT under SW-846
Purgeable Aromatic Hydrocarbons (602)	(2) 40 mL VOA Vials w/Teflon lined lid	Cool to 4°C HCL to pH<2, Zero headspace	14 Days
Benzene, Toluene, Xylene (BTEX, 602)	(2) 40 mL VOA Vials w/Teflon lined lid	Cool to 4°C HCL to pH<2, Zero headspace	14 Days
Total Organic Halogens (TOX)	250 mL amber G, w/Teflon lined lid	Cool to 4°C Zero headspace	14 Days

Total Petroleum Hydrocarbons (TPH)	1 Liter amber G, w/Teflon lined lid	Cool to 4°C H_2SO_4 to pH<2	14 Days
PFAA's (EPA 537)	250 mL Polypropylene bottle With polypropylene screw cap	Cool to 4°C	14 days to extract 28 days from ext

Sampling and Preservation Requirements
Solids

Parameter	Container	Preservative	Holding Time
Volatile Organics (8260B/C)	(1) 8 oz. Amber G, w/Teflon lined lid	Cool to 4°C	14 Days
Volatile Organics (8260B/C with 5035)	(3) 40 mL VOA Vials w/Teflon lined lid	Cool to 4°C (2)vials preserved w/ Na Bisulfate (1)vial preserved w/ methanol	14 Days
Base Neutral/Acid Extractables (8270D)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	7 Days until ext. 40 Days after ext.
Herbicides (8151A)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	14 Days
Pesticides (8081B)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	7 Days until ext. 40 Days after ext.
PCB (8082A)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	CT – 7 Days until ext. MA – 1 year until ext. 40 Days after ext. No HT under SW-846
Benzene, Toluene, Xylenes (BTEX)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	14 Days
Total Petroleum Hydrocarbons (TPH)	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	14 Days
Cyanide	20 Grams P/G	Cool to 4°C	N/A
Metals, Total	(1) 8 oz. G w/Teflon lined lid	Cool to 4°C	6 Months
TKN	20 Grams P/G	Cool to 4°C	N/A
Total Organic Carbon (TOC)	10 Grams P/G	Cool to 4°C	N/A
pH	50 Grams P/G	Cool to 4°C	N/A

Hazardous Waste Characterization

TCLP Metals
Reactivity PCB's
Volatile Organics
Corrosivity
Reactivity
-Cyanide
-Sulfide

(2) 8 oz. glass containers with a Teflon lined lid is sufficient sample amount to perform Hazardous Waste characterization

EPA Method 1311 – TCLP Sampling Requirements

Aqueous Liquid Samples (approx. 100% liquid)

Volatile Organics: (2) 40 mL VOA vials with no head space
8 RCRA Metals: (1) 500 mL Nalgene Bottle
BNA's: (2) One liter amber wide mouth glass bottles with Teflon lined lid
Pesticides/Herbicides: (2) One liter amber wide mouth glass bottles with Teflon lined lid

Solid Samples (approx. 100% solid or paint)

(1) 8 oz. wide mouth glass jar with Teflon lined cap packed tightly

Mixed Samples (solid mixed with water or mostly water)

Please contact laboratory as to the nature of the material so that appropriate sample amounts will be provided.

Non-Aqueous Liquid (mostly solvent)

(1) 4 oz. wide mouth glass jar (with Teflon lined cap)

Note: TCLP analysis is generally inappropriate; sample will be run to determine percent of suspected solvents.

12.0 Personnel Qualifications: Training

12.1 Personnel

Con-Test is committed to producing and utilizing technically competent, well trained individuals. Each new analyst undergoes a Quality Assurance/Control Policy Orientation and reads the current copy of the Quality Assurance Manual. They must sign off that they have read the current QA Manual as well as any other appropriate controlled SOP's. Analysts will also read any applicable method that corresponds to the SOP's they've read. A Data Integrity and Ethics class is provided which they will have annual refreshers of and they will receive any needed supplies.

12.2 Employee Training

It is the responsibility of the Laboratory Director to ensure that the staff is competent to perform laboratory analysis. Laboratory staff is trained by experienced analysts and supervisors in techniques where proficiency has been demonstrated by past performance. New analysts continue to perform under the supervision and direction of experienced analysts until sufficient information is obtained for Demonstration of Capability (DOC). See section 12.6.

AIHA-LAP, LLC IHLAP/ELLAP trainees must have a training period of 20 business day's duration, prior to completing a DOC and working independently on client samples. This 20-day period must be clearly documented on the IDOC training form.

A Demonstration of Capability must be performed prior to using any test method, and any time there is a change in instrument type, personnel, or method. The laboratory, through QC charting, has historical data adequately demonstrating current analyst capability to meet laboratory generated acceptance criteria.

Where the analyst has demonstrated capability through analysis and QC charting of Laboratory Control Samples with acceptable results, this procedure for demonstrating continued proficiency to perform the test method will be used for the DOC Certification Statement. All new analysts will perform an initial DOC. Continued proficiency can also be demonstrated through acceptable performance on proficiency samples.

Laboratory staff performing in-house calibrations and verifications shall have received documented training. This includes in-house verifications of thermometers and Eppendorf's. All personnel concerned with testing and calibration activities within the laboratory will familiarize themselves with the quality documentation and implement the policies and procedures in their work.

12.3 Training Documentation

Laboratory personnel performance is documented throughout training and the time spent at Con-Test. Employees are evaluated on a regular basis and their performance on external and internal proficiency samples documented.

The laboratory will maintain a training file, which contains:

1) A statement from each employee that they have read, understood, and is using the latest version of the laboratory Quality Assurance Manual and SOP's. The statement will be signed and dated.
2) A statement from each employee that they have read acknowledged and understood their personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions. The statement will be signed and dated.

3) A Demonstration of Capability (DOC) for each employee for each accredited method.
4) Documentation of any training courses, seminars, and/or workshops.
5) Documentation of each employee's continued proficiency to perform each test method by one of the following annually:

    a) Acceptable performance of a blind sample (single blind to the analyst) for each method;

    b) Another Demonstration of Capability;

    c) Successful analysis of a blind performance sample on a similar test method using the same technology (e.g. GC/MS volatiles by purge and trap for Methods 524.2, 624, or 8260) would only require documentation for one of the test methods;

    d) At least four consecutive Laboratory Control Samples with acceptable levels of precision and accuracy;

    e) If a-d cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results.

12.4 Metals Analysis Training Program (As required per ELLAP)

Environmental Lead Analysis

Prospective analysts for Lead in environmental samples shall have training and aptitude in chemistry, biology, physics, or a related physical science. Analysts receive specific training in the techniques required for analysis either formally from an instrument manufacturer, an educational institution or on-the-job (in house).
In house (on-the job) training proceeds as follows:

New analysts are required to read the instrument manual regarding operation of the instrument, calibration, hardware and software. The analyst is also required to read the relevant Laboratory Standard Operating Procedures and review reference methods in the appropriate methods manuals. This is done under the supervision of the metals section supervisor.

After a mandatory QA orientation, each analyst is then taught, one-on-one, the daily operating procedures for the respective methods concerning preparation of standards, order of analysis, QC criteria, operation of the instrument from start-up to shut-down, as well as the operation of any auxiliary software for the calculation of results. Conditions for data rejection are discussed along with the proper procedure to be followed when an out of control event occurs.

Each analyst is required to run external reference samples to determine his/her proficiency in the operation of the instrument before he/she is allowed to do sample determinations. Sample preparation personnel must be trained in the proper use of the analytical balance, preparation of glassware, and volumetric techniques. Previous training is acceptable as long as the metals section supervisor evaluates the performance of the prospective analyst through observation and comparison of standard reference preparations with known values. Analysts and technicians are trained using AIHA-LAP, LLC Policy:

All analysts and technicians shall be trained with the SOP's in use in the laboratory and with the instrument and equipment operation manuals. All analysts and technicians shall complete a minimum of four (4) independent test runs of sample preparation and/or instrumental analysis ***for each matrix****.*

Independent runs are defined as analytical runs consisting of at least five (5) samples of known content, one of which is a certified reference material (CRM) or proficiency testing material, separated by a period of time sufficient to evaluate the performance of any previous independent run. For sample preparation training, the recoveries of the associated reference materials or proficiency training samples for each run must be within ± 20% of the certified value, 75% of the time. For instrumental analysis training, the recoveries of the associated reference materials or proficiency training samples for each run must be within ± 10% of the certified value, 75% of the time. The reference/proficiency test samples utilized shall be representative of the matrices and mass ranges that the analyst will encounter during routine sample analysis.

Training checklists are completed for each person by the metals supervisor to ensure competency of individuals in each applicable area. This documentation is to be kept in the laboratory records.

The minimum total experience required before complete independent operation is allowed (i.e. absence of the instructor or immediate supervisor >30% of the time during work operations) is listed below.

Sample Preparation:	3 Months per method
Sample Analysis:	6 Months per instrument

12.5 Education and Training in Ethical and Legal Responsibilities Including the Potential Punishments and Penalties for Improper, Unethical, or Illegal Action

An employee handbook is distributed to each employee upon hire; ethical and legal responsibilities are addressed within. New employees are trained in the Laboratory Ethics and Data Integrity policy as specified in Section 3.2.2 of this manual.

12.6 Procedure for Demonstration of Capability

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel, or test method.

In general, this demonstration does not test the performance of the method in real world samples, but in applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g. water, solids, biological tissue and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e. at least four consecutive matrix spikes within the last twelve months. In addition, for analytes, which do not lend themselves to spiking, e.g. TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in section 12.7.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed if required by mandatory test method or regulation. Note: for analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. It is the responsibility of the laboratory to document that other approaches to DOC are adequate; this shall be documented in the laboratory's Quality Manual.

a) A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery (x) in the appropriate reporting units (such as ug/L) and the standard deviations of the population samples (n-1) (in the same units) for each parameter of interest.

e) When it is not possible to determine mean and standard deviations, such as for presence/absence tests and logarithmic values, the laboratory will assess performance against established and documented criteria.

f) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin.

g) If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

h) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

12.6.1 Certification Statement

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certificate statement shall be retained in the personnel records of each affected employee.

See next page for DOC

CON-TEST ANALYTICAL LABORATORY
TRAINING and IDOC (Initial Demonstration of Capability)
(Must be completed BEFORE any client samples are analyzed)

Analyst: _____
Analyte/Method Reference: _____
ex. Metals ICP EPA 200.7, Sulfide SM4500 S^2 E,VOA EPA 624

Matrix: _____

The analyst attests that they have read, understood, and/or performed the following:

	Initial and date as reviewed
SOP _____ Rev# ___	_____
Method Reference(s): (Example: SW846 8260C)	Initial and date as reviewed
_____	_____
_____	_____
_____	_____
_____	_____
Instrument Manual(s):	
_____	_____
_____	_____
_____	_____
MA DEP CAM (if any)	
_____ Rev# ____	_____
CT RCP (if any)	
_____ Ver# ____	_____

On-the-Job Training (provide dates: minimum 20-business-days for IHLAP/ELLAP)
_____
_____

We, the undersigned, CERTIFY that:
The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the NELAP program, have met the Demonstration of Capability.
The test method(s) was performed by the analyst identified on this certification.
A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Method-Specific IDOC (attached and completed with reduced data) OR **4 LFBs** (attached and completed with reduced data)

ANALYST SIGNATURE: _____ _____
Analyst has reviewed all checked materials SIGNATURE Date

SUPERVISOR APPROVAL: _____ _____
signature authorization date

LAB. DIRECTOR APPROVAL: _____ _____
signature authorization date

QA OFFICER APPROVAL: _____ _____
signature authorization date

13.0 Other Quality Considerations

13.1 Communication with Clients

Effective communication between the laboratory and its clients is of crucial importance to our ability to generate quality results. Con-Test will immediately notify clients of any problems when and if they arise.

Con-Test also encourages clients to contact the laboratory for technical assistance and to evaluate Con-Test services rendered, as a part of our continuous improvement (TQM) policy.

Any drinking water analysis where the amount detected exceeds the regulatory level (MCL) needs to be reported to the project chemist immediately, that is, as soon as realized by the analyst, for client notification, which must occur within 24 hours of obtaining the valid data. The laboratory must identify, in writing, those samples needing special reports (MCL exceedance) when the laboratory subcontracts with another laboratory. All reports, with the exception of reports submitted to the EPA in a format approved by the Department, for finished drinking water analysis, must indicate the maximum contaminant level for each analyte measured where a maximum contaminant level has been established by the Department.

Communication to the subcontracting laboratory of any special report requirements, like immediately notifying Con-Test of MCL exceedances is facilitated by the chain of custody. For MCL exceedances, the following is stamped on all subcontracting chain of custodies: "Subcontracted lab must notify Con-Test Analytical Lab of any MCL exceedance within 24 hours of obtaining valid data".

MCL exceedances and Data reporting must meet MA 310 CMR 42.13 (5) requirements:

"A certified laboratory shall be required to have current knowledge of all Federal and Massachusetts standards for all categories in which it has been certified or provisionally certified, and to report analytical results in a timely manner.

(a) Upon obtaining Valid data, a certified laboratory shall notify its clients of the results of all samples that exceed any EPA – or Department – established maximum contaminant level (MCL), maximum residual disinfection level or reportable concentration, or that identify the presence of regulated microbiological organisms in potable water. Notification must clearly indicate that a regulatory limit has been exceeded. The date, time, and manner of notification must be documented and kept on file.

(b) A laboratory that accepts potable water samples for analysis must notify its client public water system of the results of all samples that exceed a

regulatory limit. Data indicating an exceedance of a regulatory limit must be validated and the validated data reported as soon as possible, not to exceed 24 hours after the completion of sample analysis. Such notification must be given within 24-hours of the completion of the analysis of the sample whether or not the laboratory accepting the sample subcontracted the analysis to another laboratory.

(c) Laboratories must identify, in writing, those samples needing special reports (e.g. MCL exceedance) when the laboratory subcontracts with another laboratory.

(d) Laboratories accepting samples to be analyzed for the purpose of determining regulatory compliance must ensure that analytical data are reported in a timely manner to meet their clients' reporting requirements. A laboratory that has had regulatory compliance samples subcontracted to it by another laboratory must release analytical data to the client laboratory within the timeline arranged by the laboratories.

(e) Laboratories must have written standard operating procedures to ensure that the requirements of 310 CMR 42.13 (5)(a)-(d) are met."

CON-TEST PROCEDURE TO HANDLE THIS REQUIREMENT:

-Con-test ensures that once all known drinking water MA samples submitted for regulatory compliance have been analyzed, the data is reviewed (validated), and reported in a timely manner to meet the clients' needs, and any MCL exceedance is immediately relayed from the analyst to the Project manager as soon as sample is verified. The project manager then immediately contacts the client's public water system and client (within the 24-hour from analysis requirement), typically by email and relays the MCL exceedance. This email is retained to show date and time of the notification. Drinking water MA samples are not loaded onto instruments on Friday nights and over the weekend, as the process of notification is difficult. Special requests can be done with permission by supervisors. The client must supply contact information for the client and the clients' public water system that can be used over the weekend by the analyst if a notification is needed due to a MCL exceedance.

-Con-Test rarely sub contracts Drinking water MA samples for regulatory compliance, however if the situation comes up, Con-Test will relay to sub lab that MA 310 CMR regulations must be followed and have their data reported in a timely manner to meet our clients' needs as well as be given ample time to be able to let our clients know of a MCL exceedance in the required 24- hour time frame. This will be documented in the Project manager's email. Likewise, if another lab subcontracts to Con-Test we will ensure they have analytical results to report to their clients in a timely manner and within enough time to notify their client within 24 hours from analysis for any MCL exceedance.

When necessary the client is notified and work is recalled when any aspect of the testing and/or calibration work, or the results of the work, do not conform to the procedures or the agreed requirements of the client. Deviations from test and calibration methods shall only occur if the deviation has been documented, technically justified, authorized, and accepted by the client.

13.2 Communication with Regulatory Agencies

It is imperative to maintain effective communication with various Federal, State, and Local regulatory agencies. Con-Test maintains close contact and is constantly reviewing pertinent sources of information such as publications and periodicals etc. for changes in legislation and approved methodology.

13.3 Complaints/Client Inquiries

Complaints/Client Inquiries about testing data are handled immediately. All inquiries are documented on Client Inquiry Forms by project chemists who have initial client contact. Any supporting data (e.g. final reports) are attached to the inquiry form, which is forwarded to the QA department for entry into a client inquiry tracking database. The QA staff assigns an investigator, who returns the form with a response.

The investigator checks all appropriate paperwork, computer printouts, log book entries, and calculations associated with the results in question. Sometimes the sample is reanalyzed.

If through the client inquiry investigation, there is an issue with the data, a corrective action is immediately initiated by the QA department. The corrective action shall start with an investigation to determine the root cause(s) of the problem. See section 9.3 and/or the Corrective/Preventative Action SOP for more detail on corrective actions.

The inquiry resolution after being signed off from the supervisor is forwarded back to the QA department where the resolution is logged into the client inquiry tracking database, and the form (with supporting data) is returned to the initiating project chemist. The client is then notified by the project chemist of the response.

If no errors or reasons to suspect the data are found and the sample has not been removed from the laboratory the sample may still be re-analyzed, at the request of the client. If the reanalysis yields substantially different results, there will be no charge for the entire test: otherwise the reanalysis will be charged to the client at the normal rate as a separate sample. Any and all problems or complaints which arise are brought to the Laboratory Director's attention immediately.

14.0 References

14.1 Code of Federal Regulations (CFR), Protection of Environment, Title 40, Section 136 & 141, Revised July 1, 1993, 94, 95, 2012, 2016.

14.2 USEPA – Supplemental I of "Methods for the Determination of Metals in Environmental Samples", EPA/600R-94-11, Revised May 1994.

14.3 USEPA – "Methods for Evaluating Solid Waste, Physical/Chemical Methods", Quality Control, 3rd Edition, USEPA November 1990 (SW846).

14.4 USEPA – "Methods for Evaluation Solid Waste, Physical/Chemical Methods", Quality Control, 3rd Edition Proposed Update, USEPA December 1987 (SW846)

14.5 USEPA, Good Automated Laboratory Practices, December 1990.

14.6 APHA – "Standard Methods for the Examination of Water and Wastewater", 1010, 1020, 1030, & 1040, 17th edition, 1989.

14.7 APHA – "Standard Methods for the Examination of Water and Wastewater", 18th, 19th, and 21st, 22nd Editions, 1992, 1995, 2005, 2012.

14.8 AIHA-LAP, LLC Policy Modules reference, September 13, 2011 and 2017 updates.

14.9 ACIL Data Integrity Initiative, American Council of Independent Laboratories, January 2003.

14.10 NELAC 2003 Standard Quality Systems Section 5.5.2.7

14.11 Preventing Improper Laboratory Practices, Advanced Systems, Inc. September 2005.

14.12 ISO/IEC 17025:2005, "General Requirements for the Competence of Testing and Calibration Laboratories", 2005 Revision.

14.13 NELAC TNI Standard – The NELAC Institute, 2009

Appendix D

Chain of Custody Forms

NEW YORK CHAIN OF CUSTODY

ALPHA ANALYTICAL

Westborough, MA 01581
8 Walkup Dr.
TEL: 508-898-9220
FAX: 508-898-9193

Mansfield, MA 02048
320 Forbes Blvd
TEL: 508-822-9300
FAX: 508-822-3288

Service Centers
Mahwah, NJ 07430: 35 Whitney Rd, Suite 5
Albany, NY 12205: 14 Walker Way
Tonawanda, NY 14150: 275 Cooper Ave, Suite 105

Page 1 of 1

Date Rec'd in Lab

ALPHA Job #

Client Information

Client: Mott MacDonald
Address: 111 Wood Ave. South
Iselin, NJ 08830
Phone: 973 912 3347
Fax: 973 912 2400
Email: robert.treppjr@mottmac.com

Project Information

Project Name: Amtrak Penn Station NY
Project Location: AOI1 - Track 13
Project # 372088 RI02
(Use Project name as Project #) ☐
Project Manager: Robert Trepp
ALPHAQuote #:

Turn-Around Time

Standard ☒ 5 Day Due Date:
Rush (only if pre approved) ☐ # of Days:

Deliverables

☐ ASP-A ☐ ASP-B
☐ EQuIS (1 File) ☐ EQuIS (4 File)
☒ Other - Category B

Regulatory Requirement

☐ NY TOGS ☒ NY Part 375
☐ AWQ Standards ☒ NY CP-51
☒ NY Restricted Use ☒ Other - EPA - NY EDDs
☒ NY Unrestricted Use
☐ NYC Sewer Discharge

Billing Information

☒ Same as Client Info
PO #

Disposal Site Information

Please identify below location of applicable disposal facilities.

Disposal Facility:
☐ NJ ☐ NY
☐ Other:

These samples have been previously analyzed by Alpha ☐

Other project specific requirements/comments:

* Do not open or break custody seal before reaching lab.

Please specify Metals or TAL.

ANALYSIS

PCBs (SOXHLET 3540C)

Sample Filtration
☐ Done
☐ Lab to do
Preservation
☐ Lab to do
(Please Specify below)

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler's Initials	PCBs (SOXHLET 3540C)	Sample Specific Comments	Total Bottle
	AOI1-Track 13-E107-CS-C	10/15/17	1:55 AM	Concrete	DAF	X		1
	AOI1-Track 13-E10-CS-C		2:12 AM			X		1
	AOI1-Track 13-W293-CS-C		2:33 AM			X		1
	AOI1-Track 13-W326-CS-CD		2:50 AM			X		1
	AOI1-Track 13-W368-CS-CD		3:00 AM			X		1
	AOI1-Track 13-W393-CS-C		3:13 AM			X		1
	AOI1-Track 13-W487-CS-C		3:27 AM			X		1
	AOI1-Track 13-W193-CS-C	↓	9:45 AM	↓	↓	X		1

Preservative Code:
A = None
B = HCl
C = HNO_3
D = H_2SO_4
E = NaOH
F = MeOH
G = $NaHSO_4$
H = $Na_2S_2O_3$
K/E = Zn Ac/NaOH
O = Other

Container Code
P = Plastic
A = Amber Glass
V = Vial
G = Glass
B = Bacteria Cup
C = Cube
O = Other
E = Encore
D = BOD Bottle

Westboro: Certification No: MA935
Mansfield: Certification No: MA015

Container Type	A									
Preservative	A									

Relinquished By:	Date/Time	Received By:	Date/Time
Daniel Fischer	10.15.2017 @1130	Matt MacDonald/Cole Stone	10.15.2017 @ 1130
Matt MacDonald/Cole Stone	10/16/2017 @14:20	Kevin Silva	10/16/2017 @ 14:20
Kevin Silva	10/16/2017 @14:23	Daniel Fischer AAL	10/16/17 14:23

Please print clearly, legibly and completely. Samples can not be logged in and turnaround time clock will not start until any ambiguities are resolved. BY EXECUTING THIS COC, THE CLIENT HAS READ AND AGREES TO BE BOUND BY ALPHA'S TERMS & CONDITIONS. (See reverse side.)

Form No: 01-25 HC (rev. 30-Sept-2013)

con-test ANALYTICAL LABORATORY
Phone: 413-525-2332
Fax: 413-525-6405
Email: info@contestlabs.com
http://www.contestlabs.com
39 Spruce Street
East Longmeadow, MA 01028

16E0427

CHAIN OF CUSTODY RECORD (AIR)

Company Name: TRC
Address: 1430 Broadway Fl 10
Phone: 212 221 7842
Project Name: Amtrak - Penn Station
Project Location: Penn Station
Project Number: 257705.9990.0000
Project Manager: Jack Springston
Con-Test Bid:
Invoice Recipient: Jack Springston
Sampled By: C. Walter

Requested Turnaround Time
7-Day ☐ 10-Day ☐
Other:

Rush-Approval Required
1-Day ☐ 3-Day ☐
2-Day ☒ 4-Day ☐

Data Delivery
Format: PDF ☒ EXCEL ☐
Other:
Enhanced Data Package Required: ☐
Email To: jspringston@trcsolutions.com
Fax To #: cwalter@

ANALYSIS REQUESTED

TO-10A

" Hg

Lab Use	Client Use	Collection Data		Duration	Flow Rate	Matrix	Volume		Initial Pressure	Final Pressure	Lab Receipt Pressure	Summa Can ID	Flow Controller ID
Con-Test Work Order#	Client Sample ID / Description	Beginning Date/Time	Ending Date/Time	Total Minutes Sampled	☒ m³/min ☐ L/min	Code	☒ Liters ☐ m³						
01	04-west WA-E5	5/9/16 2238	5/10/16 0102	322	4.2188	IA	1358	X					
02	06 east WA-mezzdecon	1 2234	1 0356	322	4.7715	|	1536	|					
03	13-Field Blank	–	–	–	–		–	L					

Please fill out completely, sign, date and retain the yellow copy for your records

Summa canisters and flow controllers must be returned within 15 days of receipt or rental fees will apply

For summa canister and flow controller information please refer to Con-Test's Air Media Agreement

Comments:

Please use the following codes to indicate possible sample concentration within the Conc Code column above:
H - High; M - Medium; L - Low; C - Clean; U - Unknown

Matrix Codes:
SG = SOIL GAS
IA = INDOOR AIR
AMB = AMBIENT
SS = SUB SLAB
D = DUP
BL = BLANK
O = Other_____

	Date/Time:
Relinquished by: (signature)	5/10/16 1600
Received by: (signature)	5/11/16 944
Relinquished by: (signature)	Date/Time:
Received by: (signature)	Date/Time:
Relinquished by: (signature)	Date/Time:
Received by: (signature)	Date/Time:

Detection Limit Requirements
MA
CT
Other:

Special Requirements
☐ MA MCP Required
☐ CT RCP Required
☐ Enhanced Data Package Required

NELAC and AIHA-LAP, LLC Accredited

TURNAROUND TIME (BUSINESS DAYS) STARTS AT 9:00 AM THE DAY AFTER SAMPLE RECEIPT UNLESS THERE ARE QUESTIONS ON THIS CHAIN. IF THIS FORM IS NOT FILLED OUT COMPLETELY OR IS INCORRECT, TURNAROUND TIME CANNOT START UNTIL ALL QUESTIONS HAVE BEEN ANSWERED.

PLEASE BE CAREFUL NOT TO CONTAMINATE THIS DOCUMENT

Table of Contents

Appendix E

Data Validation Plan Prepared by Stantec dated November 30, 2017

Technical Memorandum

Data Validation Plan and Standard Operating Procedure

To:	Craig Caldwell, Rich Mohlenhoff. Jack Springston, Robert Trepp, Kevin Koch	From:	Theresa Kalaghan
File:	AMTRAK Penn Station	Date:	November 30, 2017

1.0 INTRODUCTION

AMTRAK is engaged in the collection of environmental data to support ongoing investigative activities at the Penn Station located in New York, New York. The purpose of this Data Validation Plan is to set forth the standard operating procedures for validating and assessing usability of data that are consistent with guidelines set forth in NYC DER-10/Technical Guidance of Site Investigation and Remediation (NYDEC, 2010). The data is anticipated to be utilized for evaluations to be conducted consistent with Code of Federal Regulations (CFR) 761.61(Toxic Substances Control Act [TSCA]). This Plan is intended to be included in the site-specific Quality Assurance Project Plan (QAPP). A flow chart illustrating the validation and reporting process is shown in Figure 1.

2.0 LABORATORY CERTIFICATION

The laboratory will be certified by the New York State Department of Health to perform the requested analyses. All analyses will be performed according to the requirements of CFR 761 Subpart N (761.272) and other applicable sections of TSCA. The designated project laboratories are Alpha Analytical and Contest.

3.1 LABORATORY DATA DELIVERABLES

The laboratory will provide Category B Data Deliverable in electronic format that includes the following elements as specified in Appendix 2B of NYDEC, 2010.

- Sample Delivery Group Narrative
- Lab Sample Information Sheets
- DEC Data Package Summary Forms
- Chain-of-Custody forms
- Analytical results
- Calibration standards
- Surrogate recoveries
- Blank results
- Spike recoveries
- Duplicate results
- Confirmation (lab check/QC) samples
- Internal standard area and retention time summary
- Chromatograms
- Raw data files

MOTT MACDONALD

Penn Station, New York
Data Screening Checklist

Project Name: _____

Project Manager: _____

Project Number: _____

Laboratory Name: _____

Laboratory (Certification #): _____

Laboratory Job Number: _____

Sample Location: _____

Sample Received Date: _____ Deliverable Package: NY Cat B ☐Yes ☐No

Samples Delivered to Laboratory By: _____

COC Present: _____

Container Status Upon Arrival: ☐ Intact ☐ Broken ☐ Open ☐ Other _____

All Sample Containers Accounted For: ☐Yes ☐No _____

Were Extra Samples Received: ☐Yes ☐No _____

Did Sample Labels and COC Agree: ☐Yes ☐No _____

Were Samples Received Within Holding Time: ☐Yes ☒No _____

Were Samples Properly Preserved: ☐Yes ☐No ☐NA _____

Were Samples Received with a Seal: ☐Yes ☐No _____

Were Samples Received at an Appropriate Temperature (4 ± 2ºC): ☐Yes ☐No _____

Laboratory Conformance Summary/Case Narrative Provided: ☐Yes ☐No _____

Were Samples Analyzed as Requested: ☐Yes ☐No _____

Were Samples Extracted and Analyzed within Holding Times: ☐Yes ☐No _____

Were Extraction and Analysis Dates Provided: ☐Yes ☐No _____

Were Requested Reporting Limits Met: ☐Yes ☐No _____

Were Results "At or Below" Reporting Limits Clearly Identified: ☒Yes ☐No _____

Sample Matrix:
☐Ground Water ☐Surface Water ☐Soil ☐Sediment ☐Drinking Water
☐Air (☐Indoor ☐Sub-Slab ☐Ambient)
☐Other _____
Analyses/Methods: _____ ☐Yes ☐No

Note 1: The Client ID for L1746832-10 was incorrectly logged as "AOI3-TRACK2/3-DUP-D" in the report issued on 12/28/2017. Mott MacDonald requested that Alpha Analytical Inc. (Alpha) revise the report so that the Client ID for L1746832-10 reads "AOI5-TRACK2/3-DUP-D". The report was reissued on 1/5/2018.

Reviewed By: _____ Date: _____

4.0 DATA VALIDATION

Data validation will generally conform to the guidelines set forth in National Functional Guidelines for Superfund Organic Methods Data Review (USEPA, 2016), National functional Guidelines for Inorganic Superfund Methods Data Review (USEPA, 2016b), and DER-10 / Technical Guidance for Site Investigation and Remediation (NYDEC, 2010). It is anticipated that 100 percent of the data collected will be reviewed, verified and validated unless instructed otherwise at a future date. Validation is a multiple step process that includes evaluation of the completeness of the sample delivery group, conformance with sample related quality control (QC) data and acceptance criteria, and conformance with instrument-related QC data and acceptance criteria. The data validation steps are described in the following sections.

4.1 DATA REVIEW

Data review or "Commercial Screening" is the initial in-house examination of the data to verify the completeness of the Sample Delivery Group (all data recorded, transmitted and processed correctly). The following items will be checked as part of the initial data review process by the consultant responsible for collecting the samples (TRC and Mott MacDonald) and documented using the *Data Screening Checklist Form* (Attachment A).

- Verify that samples were received by the laboratory and that the condition upon receipt was documented (temperature, preservation, container breakage, etc).
- Verify that the requested analyses were performed and the dates of analysis were provided.
- Verify that the requested results were reported along with the original laboratory data qualifiers.
- Verify that the requested reporting limits were included for all samples, and results at or below the reporting limits were clearly identified.
- Verify conformance with contract requirements (laboratory licensure, pricing, turnaround time, etc).
- Other screening activities as determined necessary for preliminary use of the non-validated data.

4.2 DATA VERIFICATION AND VALIDATION

Data verification is the process of evaluating the completeness, correctness, and conformance of a specific data set against the method procedure or contractual specifications. Data validation is an analyte and sample specific process that extends evaluation of the data beyond method, procedure and contract compliance to determine the quality of the data relative to the end use. The following sample and instrument related QC data and acceptance criteria are reviewed by the consultant responsible for validating the data (Stantec).

- Sample holding time
- Method blank analysis
- Surrogate spike and system monitoring compound recovery
- Laboratory control sample/duplicate analysis
- Matrix spike/duplicate analyses
- Field duplicate sample results
- Trip/equipment/field blank results
- Instrument performance checks
- Initial and continuing calibration checks
- Internal standards
- Recalculation of results only if warranted by laboratory QA/QC data.

4.3 DATA QUALIFIERS

The following qualifiers will be applied to the tabulated data based on the results of the data validation process.

Qualifier	Definition
U	The analyte was analyzed for but not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be imprecise or inaccurate.
NJ	The analyte has been tentative identified or presumptively identified as present and the associated numerical result is the estimated concentration in the sample.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria.
B	The analyte was detected in the method, field and/or trip blank.

5.0 DATA USABILITY REPORT

A *Data Usability Summary Report (DUSR)* will be prepared for each laboratory data package (includes the summary laboratory report) upon completion of the data validation process and will generally conform to the guidelines set forth in NYDEC, 2010. The purpose of the DUSR is to provide a narrative evaluation of the data quality with respect to the project specific criteria for data quality and use.

The DUSR will include a discussion of sample and analytical parameters, data deficiencies, analytical protocol deviations, and quality control problems, that result in a "qualifying action" and the respective effect on the usability of the data. The DUSRs and data tables (with qualifiers) will be distributed to the consulting firm responsible for collecting the samples and other designated end users of the data at completion of the data validation process.

6.0 DATA TRACKING AND ARCHIVE

An archive will be created and maintained by Stantec to track all data, reports, and other deliverables. Laboratory data and reports will be indexed and archived for future project use. Tracking for the data validation scope of work will be initiated by Mott MacDonald using the *Data Validation Tracking Form* (Attachment B) when the samples are delivered to the laboratory. The *Data Validation Tracking Form* will reside on the SharePoint site and will be updated by the respective consultant (Mott McDonald, TRC and Stantec) as the analyses are completed, laboratory reports are received, the data are validated, DUSRs are completed, and the validated data are distributed to end users. In addition to the laboratory data, all communications with the laboratory necessary to complete the validation process will be tracked and archived.

7.0 REVISION HISTORY

Revisions to this plan will be documents and tracked in the following table.

Revision	Description	Prepared By	Date
Rev1	Revision to include inorganic methods	Theresa Kalaghan	11/30/2017

8.0 REFERENCES

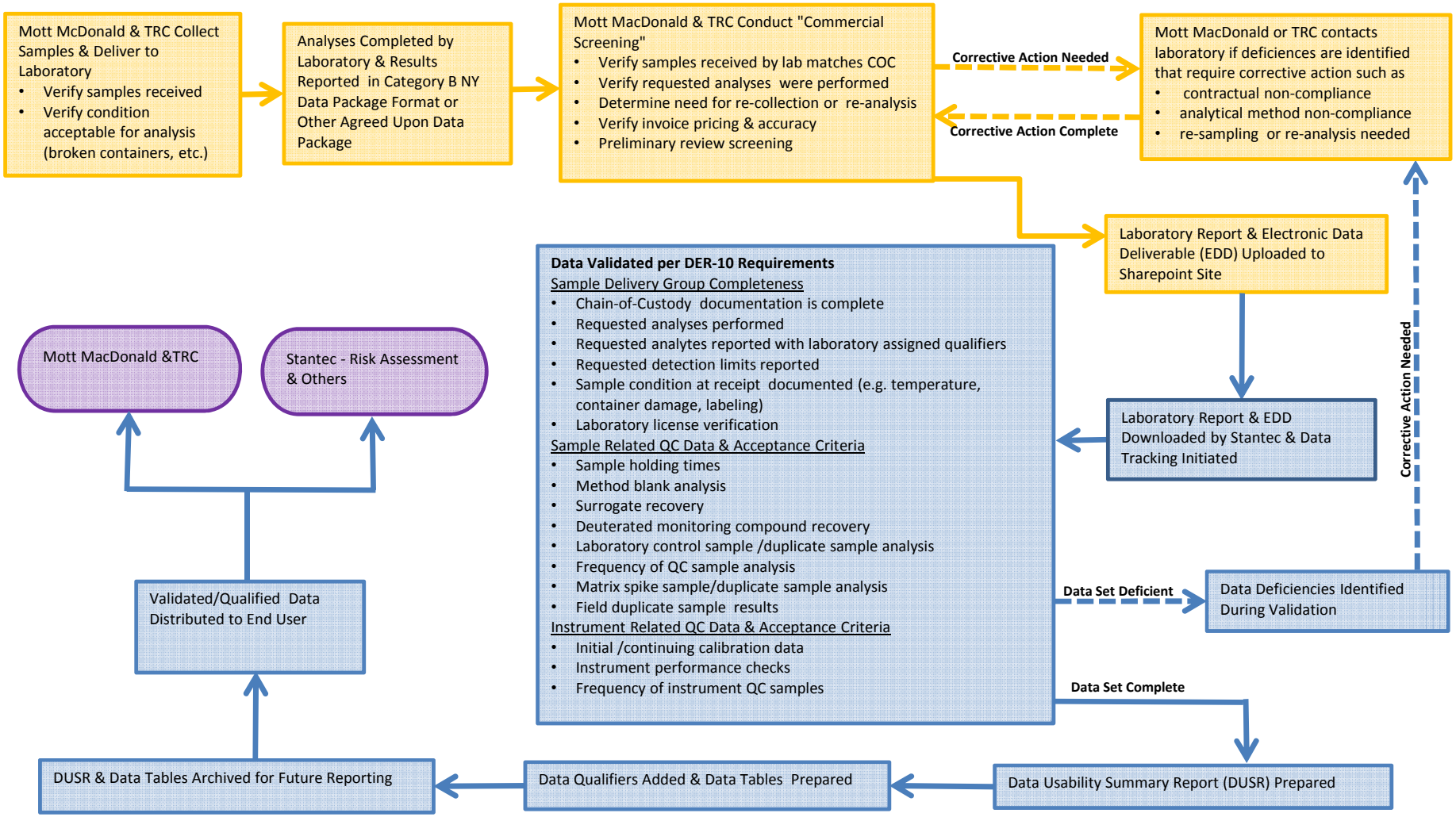
USEPA, 2016, National Functional Guidelines for Superfund Organic Methods Data Review, EPA-540-R-2016-002, September 2016.

USEPA, 2016, National Functional Guidelines for Superfund Inorganic Methods Data Review, EPA-540-R-2016-001, September 2016.

NYDEC, DER-10, Technical Guidance for Site Investigation and Remediation, May 3, 2010.

FIGURES

Figure 1
Data Validation and Reporting Process
Amtrak Penn Station, NY



ATTACHMENT A

MOTT MACDONALD

Penn Station, New York
Data Screening Checklist

Project Name: _____

Project Manager: _____

Project Number: _____

Laboratory Name: _____

Laboratory (Certification #): _____

Laboratory Job Number: _____

Sample Location: _____

Sample Received Date: _____ Deliverable Package: NY Cat B ☐Yes ☐No

Samples Delivered to Laboratory By: _____

COC Present: _____

Container Status Upon Arrival: ☐ Intact ☐ Broken ☐ Open ☐ Other _____

All Sample Containers Accounted For: ☐Yes ☐No _____

Were Extra Samples Received: ☐Yes ☐No _____

Did Sample Labels and COC Agree: ☐Yes ☐No _____

Were Samples Received Within Holding Time: ☐Yes ☐No _____

Were Samples Properly Preserved: ☐Yes ☐No ☐NA _____

Were Samples Received with a Seal: ☐Yes ☐No _____

Were Samples Received at an Appropriate Temperature (4 ± 2ºC): ☐Yes ☐No _____

Laboratory Conformance Summary/Case Narrative Provided: ☐Yes ☐No _____

Were Samples Analyzed as Requested: ☐Yes ☐No _____

Were Samples Extracted and Analyzed within Holding Times: ☐Yes ☐No _____

Were Extraction and Analysis Dates Provided: ☐Yes ☐No _____

Were Requested Reporting Limits Met: ☐Yes ☐No _____

Were Results "At or Below" Reporting Limits Clearly Identified: ☐Yes ☐No _____

Sample Matrix:
☐Ground Water ☐Surface Water ☐Soil ☐Sediment ☐Drinking Water
☐Air (☐Indoor ☐Sub-Slab ☐Ambient)
☐Other _____
Analyses/Methods: _____ ☐Yes ☐No

Reviewed By: _____ Date: _____

ATTACHMENT B

Table 1
Amtrak
Data Validation Tracking Summary
Penn Station, New York
DRAFT

Sample Collection Information (Mott MacDonald/TRC)					**Data Review (Mott MacDonald / TRC)**									**Data Usability (Stantec)**					
Sample Date(s)	AOI #	Track (Area or Project)	Sample Media	Sample Description	Date Samples Received (picked up) by Laboratory	Laboratory Name	Laboratory Job # and Data Link	Date Data Available From Laboratory	Date Data Downloaded From Laboratory	Date Data Checklist Screening Completed	Data Checklist Link	Corrective Action Needed? (YES / NO) ^	Date Corrective Action Completed	Date Data Obtained for Data Usability	Corrective Action Needed? (YES / NO) ^	Corrective Action Completed (YES / NO)	Is Data Usable (YES / NO) ^	DUSR Complete Date	DUSR Link
2016-12-19	1	16	Concrete	Post Concrete Cleaning Concrete Sample	2016-12-21	Alpha Analytical	L1641729	2016-12-27	2016-12-28		L1641729_VV	YES	NA		NO	NA	YES	2016-01-12	L1641729_DUSR

Notes and Abbreviations:
^ Follow the link to the associated report for details regarding corrective actions needed and completed
DUSR: Data Usability Summary Report

Appendix F

Project Assessment Documentation

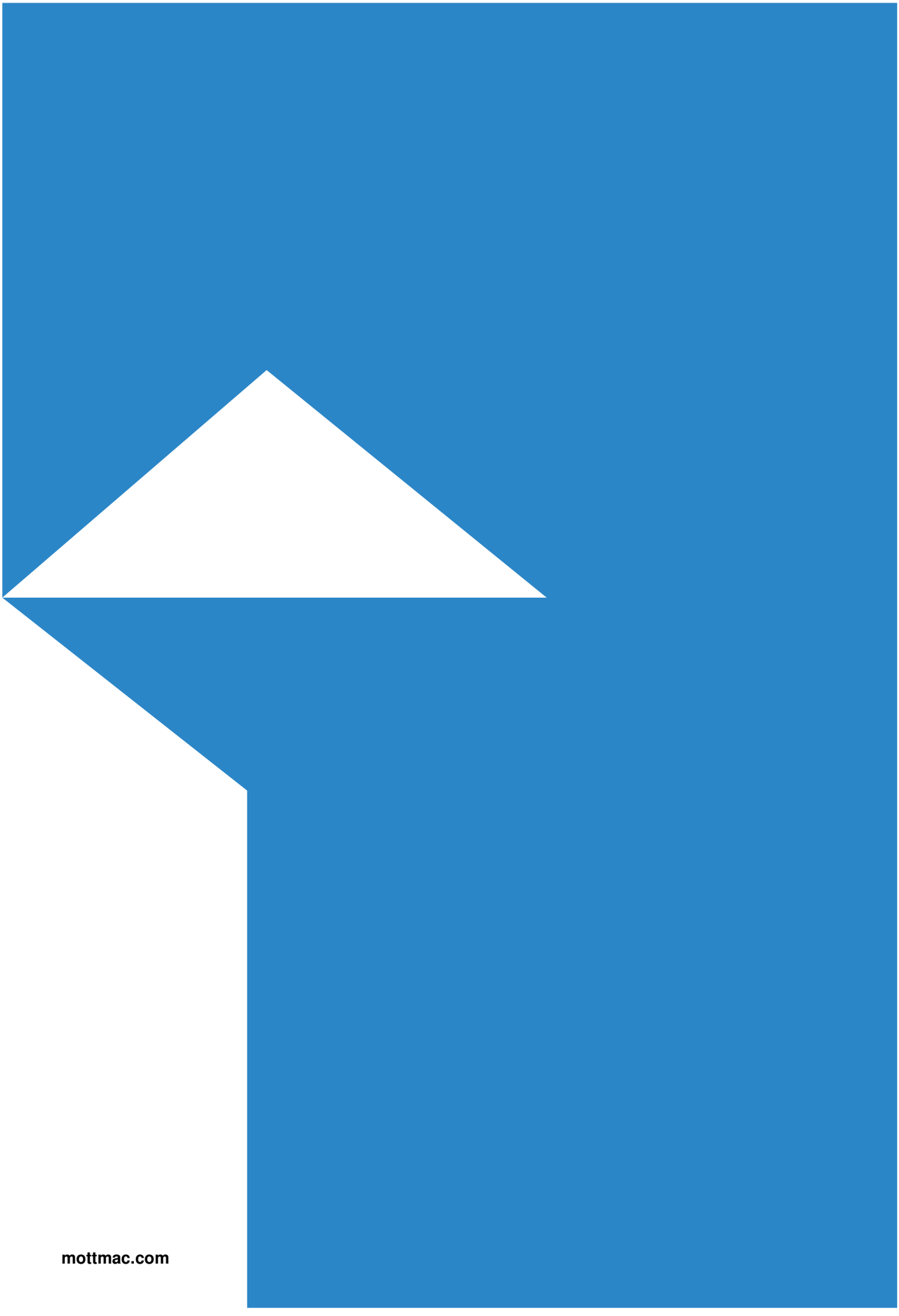
MOTT MACDONALD

Corrective Action Form
Penn Station, New York

Reviewer:		Date of Audit:	
Project Name:			
Project Number:			
Audit Type:			
Summary:			
Corrective Action(s) Taken:	☐ Y ☐ N	Corrective Action(s) Implemented:	☐ Y ☐ N
Description of Corrected Actions:			
Date of Next Audit:			
Reviewer Signature:		Date:	
Quality Assurance Officer Signature:		Date:	

Internal Progress Assessment Tracking Form
Penn Station, New York

Internal Quality Control	Frequency	Staff Member	Date	Corrective Action Needed? (Y/N)	Corrective Action Implemented? (Y/N)	Corrective Action Details
Field Staff Training	Annual					
Field Staff Audit	Quarterly					
Sample Documentation Audits	Quarterly					
Data Quality Audits	Monthly					
SOP Review	Annual					



mottmac.com