May 30, 2019 2011-2678-15

Mr. Peter Ramanauskas
United States Environmental Protection Agency - Region 5
Waste, Pesticides, Toxics Division
77 West Jackson Boulevard (DW-8J)
Chicago, IL 60604-3590

RE: Response to Additional USEPA Comments on Supplemental Site Investigation (SSI) Work Plan
Exide Technologies, 555 Hoke Avenue, Frankfort, Indiana

Dear Peter:

Please find the following letter presenting general responses to comments received from your emails dated March 13, April 5, April 11 and May 13, 2019 that pertain to your further review of the SSI Work Plan. The comments provided by the United States Environmental Protection Agency (USEPA) are in **bold** font and the general response follows in regular font. Specific revisions have been made to the attached Revised SSI Work Plan.

One significant change to note in the revised SSI Work Plan is that the HPT-GWS sampler will not be used for discreet groundwater sample collection. Instead, a hydropunch tool will be used at a location immediately adjacent to the MiHPT location and a sample(s) will be collected at the saturated zone(s) determined by the MiHPT data.

Comment 1: Shouldn't the same decision rules apply to work done under Section 2.1.1 and 2.1.2?

Response: Yes, the decision rules for MiHPT and hydropunch sampling will apply to both 2.1.1 and 2.1.2. These sections have been updated in the revised SSI Work Plan.

Comment 2: What is the rationale for stopping at 20 to 30 feet? What if real time results at 30 feet indicate VOC presence? It is my understanding that depending on local soils and geologic conditions tools can be advanced to depths of 50 to 100+ feet.

Response: Based on our previous observations of confining layers we do not expect to see evidence of contamination at depths greater than 20 to 30 feet bgs. However, our proposed subcontractor has indicated that the HPT equipment we are proposing can go deeper. If we see evidence of contamination (XSD response > 2 ppm, PID response > 10 ppm) at depths in excess of 20-30 feet we will continue to advance the probe to greater depths accordingly. The sentence in decision rule #2 that previously stated "maximum boring depth will not exceed 30 feet" was removed.

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Comment 3: The decision rules state that "Offset borings will not be performed beyond the Exide property line." How will this define "existing area of contamination" if it appears that VOC contamination extends off-site from the VOC present at the facility?

Response: Offset borings will not be performed beyond the Exide property line at this time due to presence of utility conflicts (particularly on Kelley Avenue near MW-4) as well as right-of-way access, private property access, and the related time considerations. If it is determined that off-site delineation is required, Exide will engage in further discussions regarding the scope of such additional investigation..

Comment 4: Referring to Decision Rule 3, what if the saturated zones are such that multiple water samples can be collected within that zone at different depths?

Response: If we see distinct saturated zones capable of being sampled using the hydropunch techniques then additional groundwater samples will be collected.

Comment 5: I did notice that the text states that "Actual locations, termination depths, and target groundwater sample depths will be determined by the Indiana Professional Geologist (P.G.) responsible for the investigation based on results of the real-time observations during implementation." Please see the attached examples of a decision logic diagram (DLD) and use of multiple sampling depth intervals to generate a high resolution TCE concentration vs. depth profile. Perhaps such a DLD can be generated for this project.

Response: We have reviewed the DLD that was provided. We are anticipating that a XSD response of 2 ppm or greater (or PID response of 10 ppm or greater) will indicate potential groundwater impacts and trigger additional investigation. The decision rules proposed for Section 2.1.1 (and now 2.1.2) are modified to match with the responses to these comments. We did find that DLD can be a useful tool on more complex projects, but feel that the proposed approach described for Section 2.1.1 (and now 2.1.2) is appropriate for this sampling event based on our current understanding of the conditions.

Comment 6: Referring to decision rule 3, collecting a groundwater sample from a deeper saturated interval even if a detector reading does not indicate elevated VOC may be beneficial in providing a more overall informed data set under this mobilization.

Response: Our intention is to collect a groundwater sample from the lowest saturated zone we encounter. Because we are advancing the boring until XSD <2 ppm/PID <10 ppm we expect this will serve as a "clean" bounding point.

Comment 7: With respect to the VOC detector, would use of a XSD detector in place of or in tandem with the PID be better for the detection of chlorinated VOCs or a mixed plume of hydrocarbon & chlorinated VOC if that is of concern in the former UST areas?

Response: The MiHPT rig we are planning to use will have XSD (or ECD) and PID.

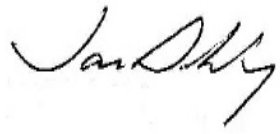
Comment 8: Can you further describe what analyte of interest you will use to calibrate the MIP?

Response: Cascade uses 2 ppm standard for TCE to response test the XSD and 10 ppm standard for benzene or toluene that they use to response test the PID. The MIP will provide information that shows areas over 2 ppm for chlorinated VOCs and where areas are over 10 ppm for BTEX/fuel related VOCs as the MIP is advanced vertically.

We believe that the responses provided above address the questions and comments contained in your March 13, April 5 and April 11, 2019 emails. If you have any questions or concerns, please call Jan Dobinsky at 610-840-9136 or Paul Stratman at 610-840-9122.

Sincerely,

ADVANCED GEOSERVICES CORP., a Montrose Environmental Group company



Jan S. Dobinsky
Associate Project Professional

Paul G. Stratman, P.E., P.G.
Consultant

JSD: PGS: vm

Enclosures

cc: Brad Weaver, Exide
Scott Ward, AGC

SUPPLEMENTAL SITE INVESIGATION
WORK PLAN

EXIDE TECHNOLOGIES
555 North Hoke Avenue
Frankfort, Indiana
EPA ID No. IND001647460

Prepared For:

EXIDE TECHNOLOGIES
Milton, Georgia

Prepared By:

ADVANCED GEOSERVICES CORP.
West Chester, Pennsylvania

Project No. 2011-2678-15
December 21, 2018
Revised May 30, 2019

ADVANCED GeoServices
a Montrose Environmental Group company

SUPPLEMENTAL SITE INVESTIGATION (SSI) WORK PLAN (WP)

EXIDE TECHNOLOGIES
555 North Hoke Avenue
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Revised May 30, 2019

PROFESSIONAL GEOLOGIST — PAUL GERARD STRATMAN — No. 2112 — STATE OF INDIANA

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5/30/19

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1.0 INTRODUCTION

1.1 PURPOSE

This Revised Supplemental Site Investigation (SSI) Work Plan (WP) has been prepared by Advanced GeoServices Corp. (Advanced GeoServices), on behalf of Exide Technologies (Exide), to specify the proposed investigation activities and additional work elements requested by United States Environmental Protection Agency (USEPA) as part of the RCRA Facility Investigation (RFI) process at 555 North Hoke Avenue in Frankfort, Indiana (the Site).

The Site is a former battery manufacturing facility that has been demolished to grade and is currently proceeding through the RCRA Corrective Action process, under the purview of the USEPA and in accordance with the Administrative Order on Consent (Consent Order), dated May 10, 2017. This SSIWP was developed to close data gaps identified in the RFI Report (Advanced GeoServices, September 14, 2018) and allow Exide and the USEPA to proceed with selection, design and implementation of appropriate interim measures and/or corrective measures for the Site. The Facility is located as shown on the Site Location Map (Figure 1). The Site is bounded by North Hoke Avenue to the west, Kelley Avenue to the east, Washington Avenue (a.k.a. Michigantown Road) to the north, and Norfolk Southern railroad tracks to the south. The Site is located within Clinton County.

The Site consists of eighteen (18) contiguous/adjacent parcels owned by Exide which encompass approximately 13.7 acres (Figure 2). All but three of the parcels are located within a perimeter security fence. The majority of the area within the fence perimeter is paved (12.1 acres). The three parcels (approximately 1.6 acres) outside the fence are open grassy lots that were not associated with the battery manufacturing activities.

The RFI was performed between May and July of 2018 in accordance with the RFI Work Plan (Advanced GeoServices, February 26, 2018). The purpose of the RFI is to identify and delineate the nature and extent of impacts to Site soils, groundwater, and sediment by Constituents of Concern (COCs) that may be associated with previous battery manufacturing activities.

During the previously completed RFI sampling work, several areas of the Site were found to have elevated concentrations of COCs that may have been associated with historic production and manufacturing processes relating to battery manufacturing or previous operations. Elevated concentrations of Trichloroethene (TCE) were observed in perched groundwater at and in the vicinity of monitoring well MW-4. TCE was also detected above screening levels in MW-1 and MW-7. Screening and sampling will be performed around these wells during the proposed SSI to further delineate the extent of soils and groundwater impacted by TCE. 1,4-Dioxane (p-Dioxane) was observed above screening levels in MW-8 during the May and July groundwater sampling events. Additional sampling around MW-8 will help determine the extent of any impacts related to 1,4-Dioxane (p-Dioxane).

Sediment sampling identified elevated concentrations of lead in sediment in several of the storm sewer manholes at the Site. In the comments issued on the RFI, USEPA questions if sediment water present in the manholes in proximity to MW-4 could be impacted by TCE or related VOCs.

Elevated lead in shallow fill in the southeast portion of the Site were the highest concentrations encountered during the RFI sampling. Additional sampling in the shallow fill in the area surrounding borings R-14 and R-15 will be completed to delineate elevated lead concentrations around these borings.

Field investigation activities in the SSI are expected to take place over approximately two weeks and will implement various forms of technology to obtain data necessary to help determine which corrective measures or remedies are appropriate to address the sources of contamination.

Although not an investigation-related task, in conjunction with the field work for the Supplemental Investigation, Exide intends to perform specific onsite activities to address issues identified during the previously completed RFI. These activities will consist of the following:

- Removal of accumulated sediment from impervious Site surfaces and installation of sediment traps in on-site storm water manholes/inlets.
- Installation of silt socks/silt fence at low points along the fence line where potentially impacted sediment could be leaving the Site. Figure 4 shows the tentative silt sock/silt fence locations. Additional locations identified in the field during implementation, if any will be added as appropriate.

If dry conditions exist at the time of removal, potentially lead impacted sediment will be sprayed with water until damp (to limit dust) and then removed from the Site surfaces using industrial scale HEPA vacuums and hand tools, until visibly clean or no additional sediment can be liberated using the proposed techniques. The filter on these vacuums will prevent airborne particulates during the sediment removal. Waste generated during the cleaning of the onsite surfaces will be placed in 55 gallon steel drums, or placed into water tight roll-off containers with cover, characterized and stored onsite until removed offsite disposal.

If wet conditions exist at the time of removal, sediment will be cleaned using HEPA vacuums or a vacuum truck (with HEPA filters) capable of transferring the collected sediment into drums or water tight roll-off containers. Pressure washing may be used to assist in cleaning sediment that cannot be removed with vacuum or hand tools alone.

The intent is to perform cleaning until visibly clean or no additional sediment can be liberated using the proposed techniques. If bulk transportation and disposal is used (instead of drums), the sediment will be solidified with a drying agent such as cement kiln dust (CKD) or lime kiln dust (LKD) to absorb free liquids sufficiently to allow for transport (i.e., pass paint filter test). After solidification the storage container contents will be characterized and then removed for off-site disposal.

If at the time of sediment cleaning it is observed that potentially impacted sediment has migrated off-site, the sidewalks and adjacent road surfaces believed to be impacted with sediment from the Site will be cleaned in the same manner as the Site surfaces.

1.2 WORK PLAN ORGANIZATION

This Work Plan is organized into five sections with supporting appendices as follows:

Section 1	Organization
Section 2	Supplemental Investigation Objectives
Section 3	Field Procedures
Section 4	Quality Control and Quality Assurance
Section 5	Reporting
Section 6	Schedule

2.0 SUPPLEMENTAL INVESTIGATION OBJECTIVES

The overall purpose of the SSI is to fill data gaps and allow characterization of risk associated with elevated concentrations of lead and TCE observed in previous RFI sampling. The SSI will include the following activities:

- In-situ screening of soil and groundwater VOCs.
- Soil and groundwater sampling guided by the results of the in-situ screening.
- Soil sampling for lead in the vicinity of the previously completed RFI borings R-14 and R-15.
- One round of groundwater sampling at all existing wells.
- Purging/bailing of MW-4 (after completion of low flow groundwater sampling) and visual evaluation of resulting purge water for DNAPL.
- Sampling perched groundwater adjacent to storm system manholes MH-4 and MH-5, and analysis for VOC.
- Sampling of sediment and/or standing water in storm system manholes MH-4 and MH-5, and analysis for VOCs.

Field procedures are discussed in the following sections of this document. Task specific data quality objectives are presented in the Sampling and Analysis Plan (SAP) in Appendix A and the Quality Assurance Project Plan (QAPP) in Appendix B of the previously approved RFI Work Plan. A Standard Operating Procedure (SOP) for proposed Soil and Groundwater Screening is provided as an attachment to this submission.

2.1 SOIL AND GROUNDWATER SCREENING

Advanced GeoServices will utilize a membrane interface probe (MIP) equipped with Electron Capture (ECD), Photo-ionization (PID), Flame Ionization (FID) and Halogen-specific XSD sensors to field screen shallow soil and perched groundwater for VOCs. The MIP will be deployed using a direct push drill rig which pushes the probe into the ground to the beginning target depth. Once the beginning depth has been reached, the probe heats the surrounding soil/groundwater, captures the vapor sample, and transports the gas to the surface to be field analyzed. The MIP collects continuous measurements from the beginning target depth through the completion depth. MIPs technology is typically used to optimize the placement and depth of subsequent samples.

Direct push drilling techniques will be utilized to advance the MIP to the top of the clay aquitard in the vicinity of elevated concentrations of VOCs in groundwater detected during the RFI sampling. The MIP will provide a qualitative indication of VOC concentrations in the saturated and unsaturated soil that will assist in targeting additional soil and groundwater sampling. Advanced GeoServices is proposing to complete1.5 days of MIPs sampling around MW-4 and an additional 2 days of MIP sampling around monitoring wells MW-1, MW-7 and MW-8. MIPs sampling is not proposed around MW-6 because the VOCs observed at MW-6 in the May 2018 sampling event are believed to be the result of cross-contamination from sampling equipment and not representative of actual groundwater quality at MW-6. This belief is supported by the MW-6 results during the July 2018 sampling event in which TCE was not detected.

In addition to using the MIP technology, we are also proposing the use of a hydraulic profiling tool (HPT) technology; or MiHPTwhich measures the pressure required to inject a flow of water into soil as the probe is advanced into the subsurface. The HPT provides a continuous representation of the hydraulic behavior of subsurface soil for the intervals tested. The information provided by the HPT will help estimate the permeability of the subsurface fill/soils. Based on the MiHPT data, a hydropunch sampling tool will be used

to collect groundwater samples from discrete intervals within a sample point immediately adjacent to the MIP location.

2.1.1 MiHPT Screening at MW-4

MW-4 was installed in the northeastern portion of the Site as part of the initial RFI site activities. Groundwater sampling was performed in two separate sampling events at MW-4 which yielded elevated TCE concentrations ranging from 214,000 ug/L to 350,000 ug/L. In July 2018 hydropunch sampling was performed at nine (9) locations surrounding MW-4 to attempt to delineate the extent of TCE around this well. TCE detections in the hydropunch samples ranged from 89.8 ug/L to 75,000 ug/L but did not fully define the limits of TCE above the screening level (5 ug/L) in this area.

In the area surrounding MW-4, the MiHPT (and associated hydropunch locations) will be advanced to approximately 20 to 30 feet below ground surface (bgs). Approximately six (6) MiHPT locations will be completed at offset distances of 100 – 150 feet from MW-4, and each location will include 1 to 2 target depths for GWS sample collection. The tentative locations of the Mi/HPT-GWS borings are shown on Figure 3. Actual locations, termination depths, and target groundwater sample depths will be determined by the Indiana Professional Geologist (P.G.) responsible for the investigation based on results of the real-time observations during implementation.

The following decision rules for the termination depths, groundwater sampling and lateral off-sets are as follows.

1. MiHPT (and associated hydropunch) borings will be terminated if the Geologist or driller has reason to believe that the boring has encountered dense non-aqueous phase liquid compounds.
2. MiHPT borings will be terminated when the boring has reached a minimum depth of 20 feet and real-time MIP results no longer indicate the presence

of notable VOC impacts (>2 ppm response on XSD or >10 ppm response on PID).

3. Groundwater samples for laboratory analysis will be collected from the first saturated groundwater zone > 4 inches in thickness in each hydropunch location. If MiHPT data indicates the presence of additional saturated horizons and the deeper zone exhibits elevated VOC concentrations (>2 ppm response on XSD or > 10 ppm response on PID) within the depth intervals being investigated then additional discreet groundwater samples will be collected using the hydropunch tooling. In addition, a groundwater sample will be collected from the deepest saturated horizon encountered regardless of XSD/PID results.
4. Off-set borings will be selected at the discretion of the field Geologist. In general, off-set borings will be performed when notable VOC impacts are observed (>2 ppm response on XSD or > 10 ppm response on PID). Off-set borings will not be performed beyond the Exide property line at this time due to the presence of utility conflicts in Kelley Avenue, right-of-way access, private property access and related time considerations.

2.1.2 MiHPT Screening at MW1, MW-7 and MW-8

TCE was detected at MW-1 and MW-7 during the May 2018 and July 2018 groundwater sampling. The TCE concentrations detected at MW-1 were 15.8 ug/L and 19.9 ug/L, and TCE concentrations detected at MW-7 were 16 ug/l and 9.9 ug/L, respectively. TCE was not detected in any of the other onsite wells other than MW-6 during the May 2018 sample event when equipment cross-contamination was identified as the source of elevated TCE detections.

During the May 2018 groundwater sampling event, 1,4-Dioxane (p-Dioxane) was detected at a concentration of 11.3 ug/L at the MW-8 location but was not detected in the other seven (7) wells onsite. In the July 2018 groundwater sampling event, 1,4-Dioxane

(p-Dioxane) was detected at a concentration of 8.4 ug/L at the MW-8 location but was not detected in the other seven (7) wells onsite.

MiHPT (and associated hydropunch) sampling will be performed during the SSI in the areas surrounding MW-1, MW-7 and MW-8 to define elevated concentrations of VOCs in groundwater. Approximately nine (9) locations in total will be sampled in the areas around these wells for VOC delineation; based on three (3) offset locations onsite at approximately 50-feet from each well. Target depths in the proposed MiHPT (and hydropounch sampling) will be from approximately 2 feet bgs to the top of the aquitard (MW-1- 16 ft., MW-4- 20 ft., and MW-8- 20 ft.). The tentative locations of the MiHPT-GWS borings are shown on Figure 3. The hydropunch groundwater samples will be collected from 1 to 2 depths at each location for VOC analysis.

2.2 GROUNDWATER SAMPLING

2.2.1 MiHPT Hydropunch Groundwater Sampling

The MiHPT related hydropunch groundwater sampling is proposed to refine delineation of the TCE above the MCL detected in groundwater surrounding MW-1, MW-4 and MW-7 as well as 1,4-Dixoane (p-Dioxane) above the MCL at MW-8.

These discrete groundwater samples will be collected from each location at a depth equivalent to “first water”. Additional groundwater samples will be collected using the hydropunch if HPT results identify a distinct saturated permeable zone (i.e. saturated zone separated from first water by a clay layer). The water from these sample horizons will be placed in individual laboratory supplied containers. Based on previous observations during the RFI sampling events, groundwater is expected to be encountered at a depth between 4 feet to 18 feet. Hydropunch samples associated with the area around MW-8 will be analyzed for VOCs and 1,4-Dioxane (p-Dioxane). All other hydropunch samples collected onsite will be analyzed for VOCs only.

2.2.2 Hydropunch Groundwater Sampling

In addition to the MiHPT-related hydropunch locations, hydropunch samples will be collected immediately adjacent to MH-4 and MH-5 to address USEPA concerns related to possible VOC impacts to the storm sewer or possible transport along the storm sewer and process lines in the northeastern portion of the Site. Hydropunch samples will also be collected in the vicinity of MW-2 and MW-6. The locations of Hydropunch sample points are shown on Figure 3. Hydropunch sample collection will be performed in accordance with the procedures utilized during previous RFI investigations.

2.2.3 Low Flow Groundwater Sampling

One (1) additional round of groundwater sampling will be performed for all of the wells onsite using low-flow purging methods at groundwater monitoring wells installed during previous investigations. Groundwater samples at monitoring wells MW-2, MW-3, MW-4, MW-5 and MW-6 will be analyzed for VOCs. Groundwater samples at monitoring wells MW-1, MW-7 and MW-8 will be analyzed for VOCs and 1,4-Dioxane (p-Dioxane). Specific conductivity, dissolved oxygen, temperature oxidation-reduction potential (ORP), turbidity and pH will be measured in the field. Water levels will be obtained in all wells prior to sampling. An equipment blank will be collected from the sampling equipment for each day that non-disposable equipment is used for low flow sampling.

2.2.4 Manhole MH-4 and MH-5 Sampling

Grab water samples will be collected for VOC analysis at manholes MH-4 and MH-5. The Advanced GeoServices field representative will access this water by removing the manhole lids, scanning the atmosphere with a 4-gas meter and entering the manhole using the manhole ladder. Grab samples will be collected from the manholes during "base" flow conditions (i.e. not during or immediately following a storm). This will allow the water to be representative of shallow/perched groundwater that is entering the storm

sewer system without “dilution” from surface water drainage. Under these sampling conditions the depth of the water in the manholes is anticipated to be shallower than 1-foot and “dipping” will be employed to collect the samples. Surface water samples will be collected using laboratory supplied containers and analyzed for VOCs. If sediment is present in MH-4 and MH-5 it will also be sampled at this time.

2.3 SOIL SAMPLING

Advanced GeoServices proposes to sample at six additional geoprobe locations to a depth of 4 feet bgs to further delineate the elevated lead concentrations observed in the shallow fill at soil borings R-14 and R-15. Samples will be collected from each one foot interval: 0-1ft.; 1-2 ft., 2-3 ft. and 3-4ft. and placed into laboratory supplied containers. These samples will be analyzed for total lead.

Soil sampling will also be performed at 3 locations (2 samples/location; 6 samples total) selected based on VOC information obtained during the MIP sampling to provide a correlation between MIP results and soil VOC concentration. One location shall be near MW-8. In general, soil samples will be selected from the boring based on the following criteria:

- Soil interval with the highest PID readings during the MIP sampling;
- One boring shall be performed in proximity to MW-8; and,
- Soil horizon 4 to 6 feet below first soil sample.

Soil samples collected in the vicinity of MW-8 will also be analyzed for 1,4-dioxane.

3.0 FIELD PROCEDURES

Details of the proposed field procedures to be followed during the SSI are presented in the SAP in Appendix A. The laboratory that will perform the analytical testing is Pace Analytical of Indianapolis, Indiana.

3.1 LOW FLOW GROUNDWATER SAMPLING

The following tasks will be undertaken for the groundwater:

- All existing wells will be inventoried to establish their conditions and determine what repairs, if any are needed.
- The depth to the well bottom and depth to groundwater will be measured in all wells.
- Groundwater sampling and analysis summary is shown on Table 1. Sampling will be performed in all 8 onsite wells using low flow sampling techniques and samples analyzed for VOCs. In addition, MW-1, MW-7 and MW-8 will be analyzed for 1,4-Dioxane (p-Dioxane).
- The repairs identified as necessary in the well inventory will be made to the wells that will be used for future monitoring.

Detailed procedures for these groundwater sampling activities are laid out in Section 2.4 of the SAP (Appendix A).

3.2 HYDROPUNCH SAMPLING

The following tasks will be undertaken for the hydropunch sample locations:

- Utilities will be marked in the areas where drilling will take place using both the public (i.e., Indiana one call) and private utility locating services.
- The hydropunch locations will be staked out in the field using spray paint markings on asphalt or concrete surfaces and modifying locations to avoid utilities.
- If necessary, concrete surfaces will be sawcut or cored prior to drilling.
- Samples collected using direct push samplers will be advanced to depths corresponding to the first saturated zone adjacent to the manhole. An Advanced GeoServices field representative will screen the groundwater samples using a PID. Excess water will be containerized for characterization and disposal.
- Representative samples of the water produced at each hydropunch location will be analyzed for VOCs.
- After the hydropunch sampling is completed, the hole will be backfilled using bentonite chips or grout and the surface restored to its original condition.

3.3 MiHPT SAMPLING

The following tasks will be undertaken for VOC screening purposes to assist in targeting additional soil and groundwater sampling around the vicinity of elevated concentrations of VOCs identified in the RFI. The following field activities will take place for the MIPs/HPT sample locations:

- Utilities will be marked in the areas where drilling will take place using both the public (i.e., Indiana one call) and private utility locating services.

- The MiHPT locations will be laid out in the field using spray paint markings on asphalt or concrete surfaces and modifying locations to avoid utilities.
- If necessary, concrete surfaces will be sawcut or cored prior to drilling.
- See MiHPT SOP which can be found in Appendix C for additional information.

3.4 SOIL BORINGS

The following field activities will take place for the soil borings in the vicinity of RFI boring locations R-14 and R-15 to attempt to refine the horizontal limits of the notably elevated lead concentrations:

- Utilities will be marked in the areas where drilling will take place using both the public (i.e., Indiana one call) and private utility locating services.
- The boring locations will be staked out in the field using spray paint markings on asphalt or concrete surfaces and modifying locations to avoid utilities.
- If necessary, concrete surfaces will be sawcut or cored prior to drilling.
- Samples collected using Geoprobe™ direct push samplers will be advanced in one foot intervals to a depth of 4 feet using acetate sleeves. An Advanced GeoServices field representative will log the borings noting the soil types and presence of free water. Excess soils will be containerized for characterization and disposal.
- Representative samples of the soils and fill materials encountered will be taken from the acetate sleeves and analyzed for total lead. Sample homogenization will take place in the laboratory.
- After the soil boring is completed, the hole will be backfilled using bentonite chips or grout and the surface restored to its original condition.

The following field activities will take place for soil borings performed to retrieve soil samples as part of the VOC investigation:

- Utilities will be marked in the areas where drilling will take place using both the public (i.e., Indiana one call) and private utility locating services.
- The boring locations will be staked out in the field using spray paint markings on asphalt or concrete surfaces and modifying locations to avoid utilities.
- If necessary, concrete surfaces will be sawcut or cored prior to drilling.
- Samples collected using direct push samplers in the area surrounding MW-4 and the areas surrounding MW-1, MW-7 and MW-8 will be advanced to depths based on VOC detections during the MIPs sampling. An Advanced GeoServices field representative will log the borings noting the soil types, PID screening results and presence of free water. Excess soils will be containerized for characterization and disposal.
- Representative samples of the soils and fill materials encountered will be taken from the acetate sleeves at and analyzed for VOCs. Soil and fill materials collected from the vicinity of MW-8 will be limited to VOCs and 1,4-Dioxane.
- After the soil boring is completed, the hole will be backfilled using bentonite chips or grout and the surface restored to its original condition.

Details of the field procedures to be followed for the soil borings can be found in Section 3.0 of the SAP (Appendix A).

3.5 MANHOLE SAMPLING

Grab water samples and/or sediment samples will be collected for VOC analysis at MH-4 and MH-5. The Advanced GeoServices field representative will access this water and/or sediment by removing the manhole lids, scanning the atmosphere with a 4-gas meter and, if deemed safe will enter the manhole using the manhole ladder. Surface water samples will be collected using laboratory supplied containers and analyzed for VOCs. The depth of water typically observed in the manholes is on the order of 3-6 inches in depth. VOCs are not expected to stratify over this shallow depth. Surface water samples will be collected by dipping the bottle into the water. If sediment is observed in MH-4 and MH-5, samples will be collected using laboratory supplied containers and analyzed for VOCs. Clean single use sample collection equipment such as disposable plastic trowels or clean stainless steel tools may be utilized to facilitate sample collection. Reusable tools will be decontaminated before reuse.

3.6 INVESTIGATION DERIVED WASTES

The investigation derived wastes (IDW) expected to be generated include excess soils and acetate sleeves from Geoprobe® samplers, decontamination fluids and used disposable equipment. The excess soils and/or collected decontamination fluids will be containerized and representative samples taken for waste characterization purposes based on the landfill requirements. Following review of the waste characterization data, the excess soil will be treated as necessary and disposed of off-site at an appropriate disposal facility by Exide. Spent disposable equipment will be collected in plastic trash bags and disposed of along with facility trash by Exide.

4.0 QUALITY CONTROL AND QUALITY ASSURANCE

To evaluate whether field or laboratory conditions may be impacting analytical samples, equipment blanks, matrix spike/matrix spike duplicate and field duplicate samples will be utilized and evaluated as part of the data review. Pace Analytical's Quality Assurance Manual is included on disk as Attachment B-1 in Appendix B.

4.1 FIELD DUPLICATE SAMPLES

Field duplicate samples allow for determination of sampling precision of the sampler and the analytical laboratory. One field duplicate sample will be taken for every 20 samples of each medium tested. At least one duplicate will be taken for each medium. For the groundwater, the field duplicate will be taken by filling two sample containers from the same sample location. For the soil samples, two samples will be taken side by side from the sample sleeve and placed into separate containers.

4.2 EQUIPMENT BLANKS

An equipment blank will be prepared when a particular piece of reusable sampling equipment was used for sample collection and subsequently decontaminated for use in additional sampling. The equipment blank will be created in the field by collecting, in the appropriate pre-preserved container, a blank water rinse from the equipment (e.g. Geoprobe® drive head) after execution of the last step of the field decontamination protocol. One equipment blank shall be collected for every twenty samples collected using the specific piece of equipment. The equipment blanks will be tested for metals and VOCs.

4.3 MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES

A matrix spike/matrix spike duplicate (MS/MSD) sample will be collected as a split for every twenty samples of each medium sampled. The sample will be given the same designation as the parent sample with the addition of MS/MSD. MS/MSD samples determine the accuracy by recovery rates of the compounds added by the laboratory as defined by the analytical method. The MS/MSD samples also monitor any possible matrix effects specific to samples and sample medium and the extraction/digestion efficiency. In addition, the analyses of MS and MSD samples check precision by comparison of the two spike recoveries.

4.4 QUALITY ASSURANCE REVIEW

Upon receipt of the electronic data packages, the data will undergo a qualitative QA review to verify the reliability of the data as it is presented. This review is defined in more detail in the QAPP in Appendix B. Based on the QA review, qualifier codes will be placed next to specific sample results on the sample data tables, if necessary. A completeness of 90% or greater of unrejected analytical data is required for each medium.

5.0 REPORTING

Following approval of this Work Plan, field activities will begin with obtaining an Indiana licensed driller to perform MIPs sampling, soil borings and hydropunch sampling.

USEPA will be notified ten days in advance of the start of sampling activity. The field work is expected to take approximately two weeks to complete (this may or not be consecutive weeks).

Approximately 60 days following validation of all of the sample data, a revised RFI Report including data obtained during the SSI activities will prepared and submitted to USEPA for approval. The report will include results of the sampling activities, a discussion of the subsurface fill/soil conditions and groundwater conditions, and responses to the EPA comments on the original RFI Report.

6.0 SCHEDULE

The approximate schedule for implementation of the SSIWP is as follows:

- 0-45 Days: Retain Driller/MiHPT operator (MiHPT equipment is not available untilmid June)
- 45-75 Days: Perform field work (soil and groundwater sampling pending driller availability);
- 75-105 Days:Receive, evaluate and validate soils and groundwater data;
- 105-150 Days: Prepare SSI Report and submit to USEPA.

If any observations are made during the SSI period which Exide believes warrant a modification (additional investigation) to the SSI, the USEPA will be notified. The 150 day schedule does not anticipate/include delays due to weather, permitting, driller availability, or additional scope.

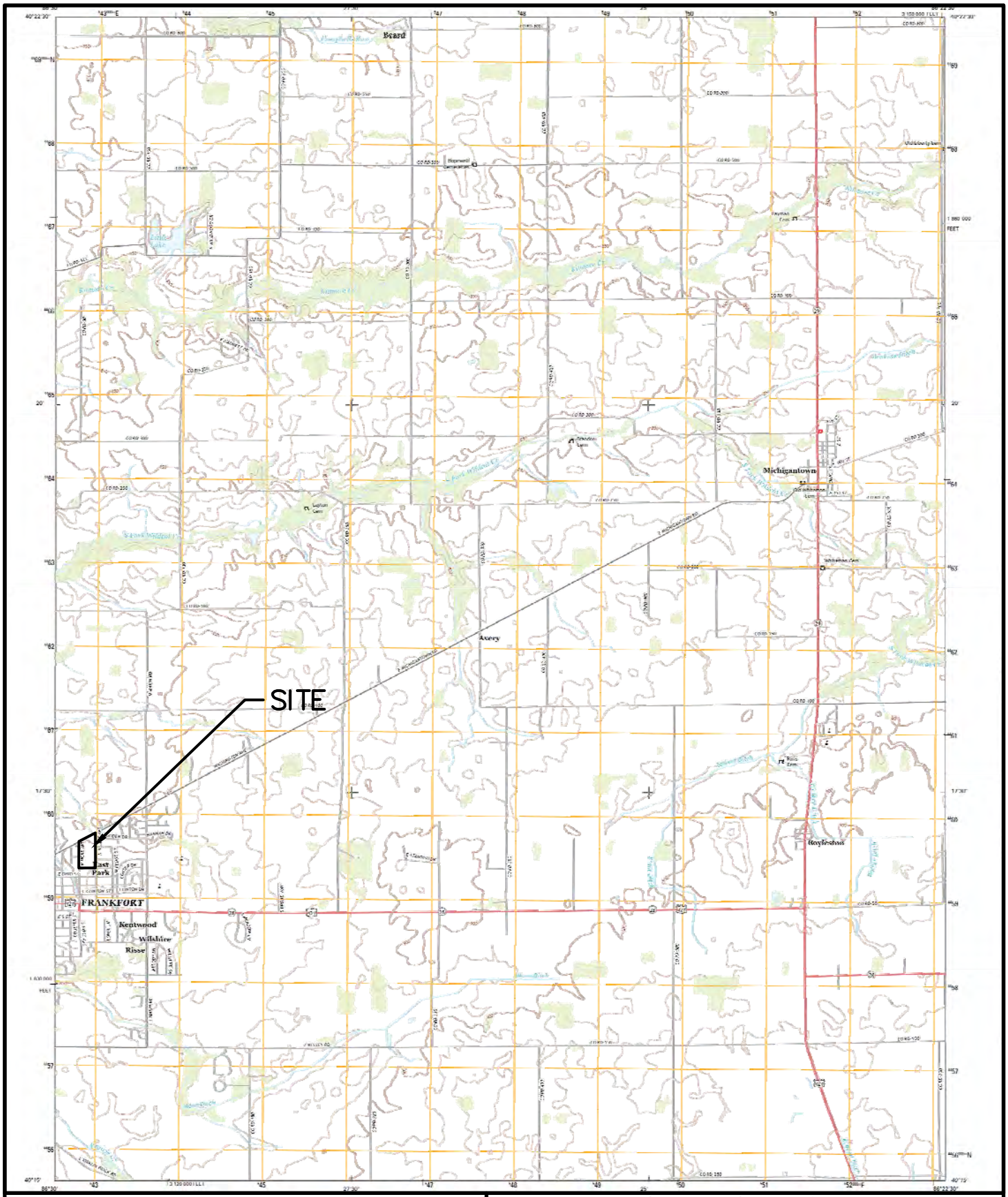
TABLE

TABLE 1
MONITORING WELL ANALYSIS SUMMARY
Supplemental Site Investigation Plan
Frankfort, Indiana

Sampling Event	Matrix	Analytical Parameters	Analytical Method	Geotechnical Testing
SSI Groundwater	Groundwater	TCL Volatiles	8260C	NA

Notes:
1. All monitoring wells MW-1, MW-2, MW-3, MW-4, MW-5, MW-6, MW-7 and MW-8 will be sampled during the SSI.
2. MW-5 was the only monitoring well location where no VOC or 1,4-dioxane detections were observed in the May and July 2018 goundwater sampling events.
3. Monitoring wells MW-1, MW-7 and MW-8 will also be sampled for 1,4-dioxane in addition to VOCs.

FIGURES



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USGS TOPOGRAPHIC MAP

EXIDE TECHNOLOGIES
FRANKFORT, INDIANA

PROJECT ENGINEER:	PGS	SCALE:	NTS
CHECKED BY:	JSD	PROJECT NUMBER:	2011-2678
DRAWN BY:	KEZ	DATE:	FIGURE: 1

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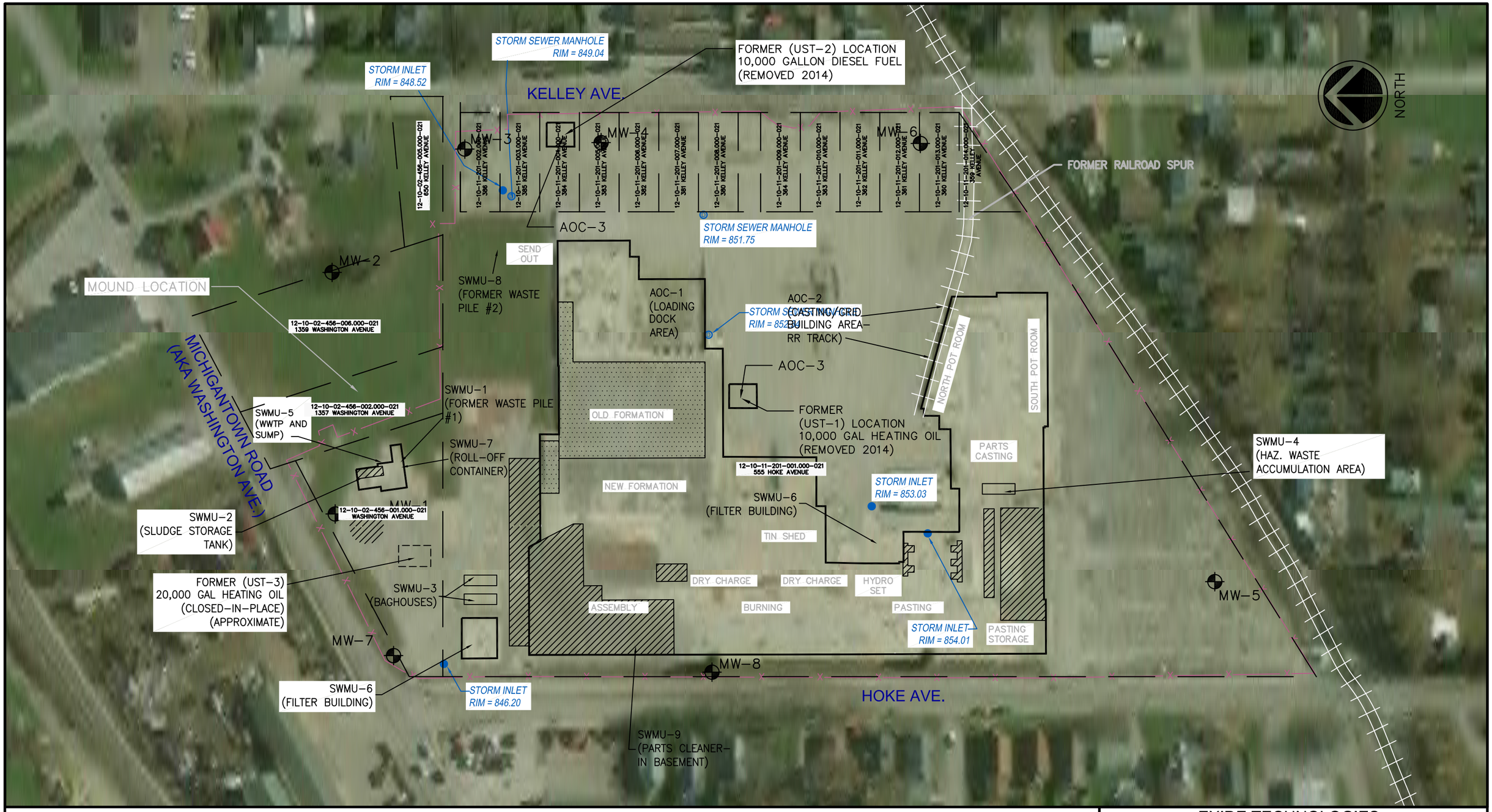

LEGEND:

- REMAINING CONCRETE SLAB PERIMETER
- CRUSHED RUBBLE/MASONRY FILL
- GEOMEMBRANE/STONE CAP
- EXISTING FENCE (LOCATION APPROXIMATE)
- APPROXIMATE PARCEL BOUNDARY
- STORM INLET
- MW-1 RFI MONITORING WELL LOCATION (INSTALLED MAY 2018)
- RAILROAD TRACKS

AOC: AREA OF CONCERN

SWMU: SOLID WASTE MANAGEMENT UNIT

0 50 100 200

1 INCH = 100 FT

EXIDE TECHNOLOGIES

555 HOKE AVENUE
FRANKFORT, INDIANA

SITE PLAN

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Originated By:	S.D.W.
Project No.:	2011-2678
Drawing Date:	6/13/2018
Sheet No.:	1 OF 1
Revision Number:	0

FIGURE 2

F:\Projects\2011\20112678-Exide Frankfort Decon DemO\Cad\RFI soil boring locations.dwg

FORMER (UST-2) LOCATION
10,000 GALLON DIESEL FUEL
(REMOVED 2014)

KELLEY AVE.

NORTH

MW-3 MW-4 MW-6

AOC-3

SEND OUT

MW-2

MOUND LOCATION

SWMU-8
(FORMER WASTE
PILE #2)

AOC-1
(LOADING
DOCK
AREA)

AOC-2
(CASTING/GRID
BUILDING AREA-
RR TRACK)

(ABANDONED)

NORTH PART ROOM

SOUTH POT ROOM

MICHIGANTOWN ROAD
(AKA WASHINGTON AVE.)

SWMU-1
(FORMER WASTE PILE
#1)

SWMU-5
(WWTP AND
SUMP)

SWMU-7
(ROLL-OFF
CONTAINER)

OLD FORMATION

NEW FORMATION

FORMER
(UST-1) LOCATION
10,000 GAL HEATING OIL
(REMOVED 2014)

PARTS
CASTING

SWMU-4
(HAZ. WASTE
ACCUMULATION AREA)

SWMU-6
(FILTER BUILDING)

MW-1

SWMU-2
(SLUDGE STORAGE
TANK)

TIN SHED

FORMER (UST-3)
20,000 GAL HEATING OIL
(CLOSED-IN-PLACE)
(APPROXIMATE)

SWMU-3
(BAGHOUSES)

DRY CHARGE

DRY CHARGE

HYDRO
SET

ASSEMBLY

BURNING

PASTING

PASTING
STORAGE

MW-5

MW-7

MW-8

SWMU-6
(FILTER BUILDING)

HOKE AVE.

SWMU-9
(PARTS CLEANER-
IN BASEMENT)

0 50 100 200

1 INCH = 100 FT

REMAINING CONCRETE SLAB PERIMETER

CRUSHED RUBBLE/MASONRY FILL

GEOMEMBRANE/STONE CAP

R-1 RFI SOIL BORING LOCATION (APRIL/MAY 2018)

RFI HYDROPUNCH LOCATION (JULY 2018)

MW-1 RFI MONITORING WELL LOCATION (INSTALLED MAY 2018)

S-1 PROPOSED SSI SOIL BORING LOCATION. ADDITIONAL SOIL BORINGS WILL BE FIELD LOCATED BASED ON RESULTS OF MIPS SAMPLING.

PROPOSED MIPS SAMPLE LOCATION

PROPOSED HYDROPUNCH SAMPLE LOCATION. ADDITIONAL LOCATIONS WILL BE FIELD LOCATED BASED ON RESULTS OF MIPS SAMPLING.

STORM SEWER/MANHOLE/FLOW DIR.

SAN. SEWER/MANHOLE/FLOW DIR.

10,000 - 10,000 ug/L TCE ISOCONCENTRATION LINE

50,000 - 50,000 ug/L TCE ISOCONCENTRATION LINE

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SSI PROPOSED SAMPLE LOCATIONS

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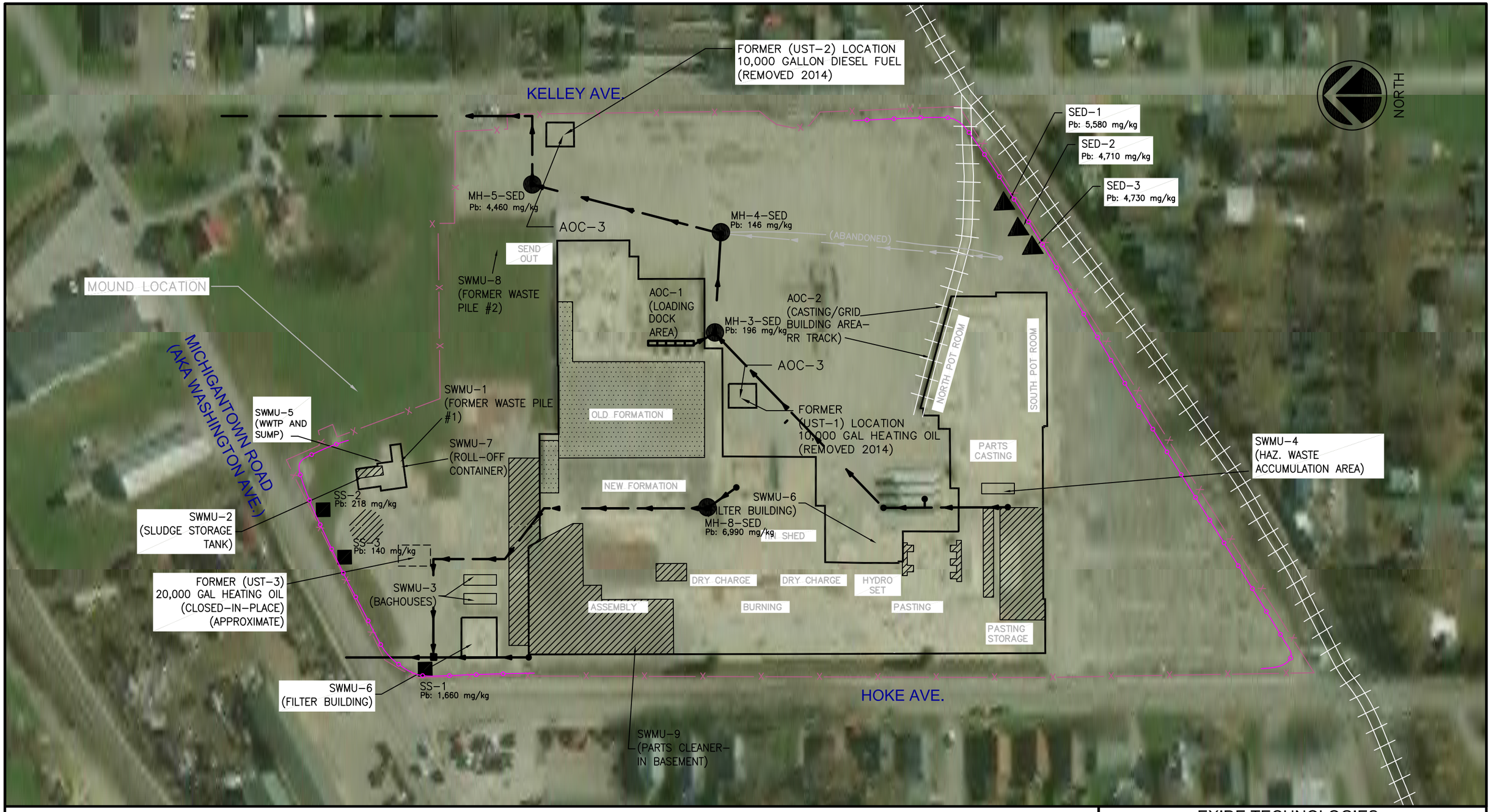
Originated By:	S.D.W.
Project No.:	2011-2678
Drawing Date:	12/18/2018
Sheet No.:	1 OF 1
Revision Number:	0

FIGURE 3

F:\Projects\2011\20112678-Exide Frankfort Decon DemO\Cad\SSI\SSI sample locations.dwg



MH-5-SED
Pb: 4,460 mg/kg
LOCATION OF SEDIMENT SAMPLE COLLECTED IN BOTTOM OF MANHOLE. LEAD CONCENTRATION LISTED IF SAMPLE EXCEEDED 800 mg/kg.

SED-1
Pb: 5,580 mg/kg
LOCATION OF SEDIMENT SAMPLE COLLECTED ON SURFACE OF ASPHALT. LEAD CONCENTRATION LISTED IF SAMPLE EXCEEDED 800 mg/kg.

SS-1
Pb: 1,660 mg/kg
LOCATION OF SEDIMENT SAMPLE COLLECTED ON BARE SOIL SURFACE (0-1 FT INTERVAL.). LEAD CONCENTRATION LISTED IF SAMPLE EXCEEDED 800 mg/kg.

REMAINING STORMWATER COLLECTION PIPING

REMAINING CONCRETE SLAB PERIMETER

CRUSHED RUBBLE/MASONRY FILL

GEOMEMBRANE/STONE CAP

PROPOSED SILT SOCK/SILT FENCE

0 50 100 200
1 INCH = 100 FT

EXIDE TECHNOLOGIES
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FRANKFORT, INDIANA

RFI SEDIMENT SAMPLE LOCATIONS AND PROPOSED E&S CONTROLS

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Originated By:	S.D.W.
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Drawing Date:	2/11/2019
Sheet No.:	1 OF 1
Revision Number:	0

FIGURE 4

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APPENDIX A

Sampling and Analysis Plan (SAP)

SAMPLING AND ANALYSIS PLAN
FOR THE
RCRA FACILTY INVESTIGATION
FORMER EXIDE MANUFACTURING FACILITY
Frankfort, Indiana

Prepared for:

EXIDE TECHNOLOGIES
Milton, Georgia

Prepared by:

ADVANCED GEOSERVICES
West Chester, Pennsylvania

Project No. 2011-2678-11
October 12, 2017

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1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been developed for the RCRA Facility Investigation (RFI) Work Plan (WP) at the former Exide Technologies (Exide) battery manufacturing facility (the Facility) located at 555 North Hoke Avenue in Frankfort, Indiana (the Site). This SAP provides the specific descriptions of field groundwater and soil sampling procedures to be performed during the RFI activities. The Quality Assurance Project Plan (QAPP), provided as Appendix B, provides the quality assurance procedures for the field sampling and laboratory analysis of the samples. Some of the field QA procedures have been included in each section of the SAP, where appropriate.

The SAP consists of sections for each type of sampling that will occur followed by general procedures as follows:

Section 2	Groundwater
Section 3	Soil Borings
Section 4	Sample Handling Procedures

1.1 FACILITY BACKGROUND INFORMATION

Based on available information contained in the Consent Order and a November 28, 2011 Letter Report prepared by USEPA, Prest-O-lite Manufacturing owned the Site during the World War II era. Prest-O-Lite was a car equipment manufacturer (including lead acid batteries). Based on Polk's City Directory in 1959 a telephone exchange registered to P.R. Mallory & Co. Inc. was listed for the address. P.R. Mallory & Co. manufactured electronics including dry cell batteries and eventually became Duracell. General Battery Corporation purchased the Site in 1963, which is consistent with additional information from Polk's City Directory which shows the telephone exchange for the address belonging to General Battery & Ceramic Co. Exide assumed ownership of the Site during the acquisition of General Battery Corporation in 1988. Exide currently owns the parcels that make up the Site. At its peak the facility produced over 12,000

automotive batteries per day. The Standard Industry Classification (SIC) code for the facility was 3691, Battery manufacturing. Battery manufacturing operations ceased in 1997. The Site was used by Exide for equipment storage until the demolition project in October 2012.

In October 2012, Exide voluntary performed a decontamination of the facility followed by demolition of the above grade structures. The decontamination and demolition project was completed in January 2013. Aside from UST removal and a limited soil investigation performed in 2014 the Site has been inactive since the completion of demolition. The Site buildings have been demolished and it is currently vacant.

1.2 FACILITY OPERATIONS

During the battery manufacturing process, metallic lead was received at the facility, melted, and cast into grids and posts. Lead oxide paste was also manufactured at the facility and subsequently applied to the grids. Lead oxide was created by feeding molten lead into a reactor and mixing it with air to oxidize the lead. Pasted grids were placed in stacks that formed the core of the battery. The cores were placed in battery cases that were produced offsite. Then the remaining components were added. Electrolyte (dilute sulfuric acid) was then added to the battery and a charge was applied (formation). After formation, the battery was cleaned, finished, labelled, and packaged for shipment to retailers and distributors.

Water was used to cool batteries after charging and also to wash batteries prior to shipment. Cooling water was collected in floor drains and emptied to a sump in the wastewater treatment plant (WWTP) located on the northwest corner of the Site. The sump collected wastewater from the entire plant, including acidic water from the cooling and washing process, and lead from washing and dry charge operations. The corrosive wastewater (containing primarily dilute sulfuric acid and lead) was pumped into two above ground holding tanks outside the building and then into reactor tanks located inside the WWTP building (SWMU-5).

Wastewater was treated with lime to neutralize the pH and precipitate dissolved metals. Following neutralization, the wastewater flowed into a large clarifier immediately adjacent to the WWTP building. Precipitated solids settled to the bottom and were pumped to a sludge holding tank (SWMU-2). From June 1986 until operations ceased the sludge was dewatered using a filter press. Sludge cake generated by the filter press was collected in a roll-off container (SWMU-7) for offsite disposal. Extracted water was recycled back to the clarifier. Prior to the use of the filter press, sludge was dewatered using vacuum pan filters and the filter cake was temporarily stored in an enclosure building (SWMU-1) prior to offsite disposal. Clarified water was then discharged to the City of Frankfort sanitary sewer system in accordance with a discharge permit.

Lead emissions were generated from molten lead handled in melting pots and casting machines. Plastic fumes were also generated from the battery case heat sealing process. Air handling equipment was used to vent these emissions to baghouses on the southeast side of the building. Lead oxide dust also was vented through ducting to a baghouse (SWMU-3). In general, air from the Facility was cycled through a filtering system called the OSI in the filter building (SWMU-6).

2.0 GROUNDWATER

The objective of the groundwater investigation during the RFI is to quantify the groundwater quality at the downgradient limits of the Site (expected to be north-northeast). Soil and groundwater sampling completed as part of the UST closure in 2014 identified chlorinated solvents and naphtha compounds in the vicinity of former UST-2 and UST-3. No site-wide groundwater network exists.

During the UST closure in 2014 geoprobe groundwater samples were collected from depths of up to 12 feet below ground surface (bgs). Groundwater was encountered at approximately 10 feet bgs. The results were not definitive as to the hydraulic gradient onsite, although expectations are that shallow groundwater flow is toward the north-northeast.

Five new monitoring wells will be installed at the locations shown on the Monitoring Well Location Plan, attached as Figure 4 of the RFI Work Plan to serve as upgradient and downgradient monitoring locations. Following installation of the new monitoring wells, a round of groundwater elevations will be collected from all accessible wells to create the piezometric maps for the facility. Two rounds of low flow sampling spaced approximately three months apart and analysis of the samples for total and dissolved RCRA 8 metals, and TCL VOCs/SVOCs. Specific conductivity, pH, oxidation-reduction potential (ORP), turbidity, dissolved oxygen, and temperature will be measured in the field during groundwater sampling.

2.1 DATA QUALITY OBJECTIVES

Groundwater monitoring will be conducted to determine whether site-related constituents are present at the Site perimeter at concentrations above the Indiana Department of Environmental Management (IDEM) Office of Land Quality (OLQ) Screening Levels for Residential Tap Water (RCGs) and to confirm the groundwater flow conditions. To meet this goal, the following data quality objectives (DQOs) have been established. To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection

necessary to meet the applicable decision criteria as defined in USEPA *Guidance for the Data Quality Objective Process* dated August 2000 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process are presented below.

2.1.1 State the Problem

There is a potential that heavy metals identified in site soils could be released to the groundwater. Based on limited chemical data they can contain lead and/or other heavy metals at elevated concentrations. Infiltration into these fill materials is limited by the buildings and the asphalt and concrete pavements at the ground surface. Furthermore, lead and the other heavy metals associated with facility operations are soluble only in very low or very high pH conditions.

During the UST closure groundwater samples detected low levels of naphtha compounds and elevated levels of chlorinated solvents at concentrations exceeding the RCGs. Chlorinated solvents were not known to be used on the Site and the source of the detections observed in previous groundwater samples has not been determined (on or offsite). Monitoring well locations around the former USTs and in likely upgradient perimeter locations are intended to help identify/delineate the source for the chlorinated solvents.

Low flow sampling techniques will be used that minimize the resuspension of sediments and yield results that are representative of the groundwater conditions.

2.1.2 Identify the Decision

Groundwater monitoring will be conducted to determine if site contaminants, if present, have been released beyond the facility perimeter as well as to assist in determining a location of the source.

2.1.3 Identify Inputs to the Decision

The Performance Standards are the IDEM 2017 OLQ Screening Levels established for groundwater. These are referred to as RCGs. The most stringent groundwater criteria is for residential tap water and those standards will be used for screening. No industrial groundwater standards exist for tap water. Residential and commercial/industrial standards for groundwater vapor exposure exist, but are less stringent than the tap water screening level.

Pace Analytical is expected to be utilized for analysis of groundwater samples. The laboratory that was selected participates regularly in performance evaluation audits as part of their laboratory certification efforts. Pace is a NELAP accredited laboratory. USEPA SW-846 methods will be used in the sample analysis of total and dissolved metals (RCRA 8) and TCL list VOCs and SVOCs; see Table 3 in the QAPP (see Appendix B) for the specific methods.

2.1.4 Define the Study Boundaries

Groundwater sampling will occur at the following wells:

Downgradient (Site):	MW-1, MW-2, MW-3
Upgradient (former USTs):	MW-4
Upgradient (Site):	MW-5

2.1.5 Develop a Decision Rule

Total and dissolved RCRA 8 metals concentrations will be compared to the RCGs. If the results exceed the RCGs at the downgradient perimeter, then additional evaluation and investigation may be conducted to identify the source of the elevated concentrations. If groundwater results exceed the RCGs, a fate and transport study will be conducted along with a risk assessment in order to determine the need for corrective action, if any.

VOC/SVOC concentrations (notably chlorinated solvents and fuel related compounds) will be compared to the RCGs. Upgradient wells (MW-4 and MW-5) will provide important information on the direction of groundwater flow and potential source of the contaminants (i.e., onsite or offsite).

2.1.6 Specify Limits of Decision Errors

The sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as "total study error." The two contributors to the total study error are the statistical sampling error and the measurement error. The statistical sampling error occurs when the data collection plan fails to provide a limited variability within the decision unit necessary for accurate decision. Measurement errors are introduced during the processes to which the sample is subjected such as physical collection, sample handling, sample preparation, sample analysis, and data reduction. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors.

The following are some ways to minimize the statistical sampling error:

- Use low-flow purging techniques when collecting the groundwater samples to minimize the turbidity of the samples.
- Use passive sampling techniques (i.e. Hydrasleeves) if field conditions such as well constrictions, poor recharge or easily mobilized sediments prevent or affect low-flow purging techniques.

The following are some ways to minimize measurement errors:

- Have the samplers collect the samples in a standardized manner;
- Ensure that each sample is properly preserved, labels, and transported to the laboratory under chain-of-custody;

- Specify that the laboratory use accepted USEPA Methods and report the data using the proper units;
- Specify that the laboratory participate in regular performance testing and also has NELAP certification;
- Receive the analytical data from the laboratory in an electronic format to minimize transcription errors; and,
- Perform a qualitative review (data validation) of the analytical data, as specified in the QAPP (see Appendix B), to verify the reliability of the data.

2.1.7 Optimize the Design for Obtaining Data

Upon review of the analytical data, future groundwater monitoring may be proposed to optimize the long-term program design. The design options will be evaluated based on cost and the ability to meet the DQOs and presented to the USEPA for approval. Design options may include additional monitoring well locations and/or additional groundwater parameters to aid in fate and transport modelling.

2.2 SAMPLE LOCATION AND FREQUENCY

Two groundwater monitoring events will be conducted after the installation of five proposed wells onsite. The groundwater monitoring events will be completed approximately 3 months apart with the first event being at least 2 to 4 weeks after well development. The well locations for sampling are shown on Figure 4 of the RFI Work Plan.

2.3 SAMPLE DESIGNATION

Groundwater sampling will be conducted at the wells identified in Section 2.1.4 of this SAP. Samples will be labeled by the well identification (e.g., MW-1, MW-2, etc.).

2.4 WELL INSTALLATION

Three shallow zone wells (MW-1, MW-2, and MW-3) will be installed along the northern property boundary. An upgradient (inferred) shallow zone well (MW-5) will be installed in the southwest corner of the Site inset from Hoke Avenue. Another upgradient (inferred) shallow zone well (MW-4) will be installed on the eastern side of the Site south of the UST area.

The wells will be installed using hollow-stem auger techniques (direct push methods may also be accepted). During drilling, soil samples will be collected from the 0-4 foot bgs, 4-8 foot bgs, and 8-12 foot bgs intervals. Soil conditions will be recorded and provided on boring logs.

The proposed monitoring well construction detail is shown on Figure A-1. Hollow stem auger drilling techniques using 6-inch (minimum) inner diameter augers will be used to drill and install the monitoring wells. The monitoring wells will be finished as stick-up wells (flush mount may be considered depending on the conditions where they are installed).

Five monitoring wells will be constructed. All wells will be constructed of two-inch diameter Schedule 40 PVC screen and riser. A 5 to 10-foot long 0.010" slot screen will be installed in the first water bearing zone encountered to intercept the uppermost part of the aquifer. The bottom of the screen set is expected to be set approximately 18 feet below the ground surface.

Specific monitoring well installation procedures include:

- Design of monitoring well construction criteria;
- Approval of construction detail by Advanced GeoServices Indiana Professional Geologist;
- Installation of PVC screen with riser;
- Installation of shallow well sand pack in preferred shallow zone strata;
- Installation of seal layer; and,
- Installation of outer casing stick-up.

The Advanced GeoServices field technician will design the monitoring well based on the soil/sediment conditions encountered. The field technician will contact the Advanced GeoServices Indiana Professional Geologist prior to installation of the monitoring well for confirmation of design parameters.

Based on the measurements of the total depth and depth to water, the borehole bottom will be plugged with a 2 feet of Portland Type II and bentonite (5%) if necessary to reach two feet below the design elevation for the bottom of the well. A two-foot bentonite layer using 3/4-inch, coated bentonite pellets will be installed over the top of the grout. The bentonite will be hydrated using approximately three gallons of potable water after installation. The bentonite will be hydrated for a minimum period of fifteen minutes prior to any other installation procedures.

The screen will be installed above the bottom bentonite layer. A sand pack (#1 sand) will be installed in the borehole by tremie pipe to approximately two feet above the screen. A two-foot thick seal of bentonite pellets will be installed above the sand pack and hydrated (using approximately three gallons of potable water) for 15 minutes. An outer steel casing will be installed and grout will be installed using a tremie pipe above the bentonite seal using a 5% bentonite/Portland Type II mixture to approximately 0.5-feet above surface grade for stickup wells or 0.5 feet below surface grade for flush-mount wells, within the annular space of the inner and outer casings.

Locking caps and locks will be installed on the top of each PVC inner casings. Aluminum locking covers will be properly bolted to the outer casing upon completion (cast iron lids will be used for any flush mount wells). All monitoring well construction procedures will be documented in the field notebook. Monitoring well designations (i.e., MW-1, MW-2, etc.) will be clearly labeled using a permanent marker on the lid (inside and outside). The location of the well and the elevation of the top of the inner well casing will be determined by an Indiana licensed surveyor.

Following installation and development of the new monitoring wells, a round of groundwater elevations will be collected from all accessible on-site wells to generate peizometric maps for the Facility. The new monitoring wells will be sampled in the two groundwater sample events planned as part of the RFIWP as noted above in Section 2.2 and will follow sampling procedures as described below in Section 2.5

2.5 FIELD PROCEDURES

2.5.1 Well Development

Following installation of the wells and at least 2 weeks prior to groundwater sampling, development will be performed in the new wells. Redevelopment will consist of agitation of the well bottom to suspend silt and removal of silt laden water via air-lifting followed by purging with submersible pump until purge water generally has low levels of turbidity (less than 20 NTUs) and a true bottom has been established. It is anticipated that approximately 3 well volumes may need to be purged to meet the redevelopment turbidity goals but in no case will more than 5 well volumes be purged. Purge water will be collected and stored onsite until it can be characterized and disposed of by Exide.

2.5.2 Sampling Equipment

The following equipment will be used for the groundwater well sampling:

- Low-flow bladder pump and control box capable of sampling groundwater at depths expected at the Site;(peristaltic or electric pumps are acceptable alternatives)
- Nitrogen tank;
- Electronic depth to water meter;
- Flow-through-cell consisting of pH/temperature meter, conductivity meter, dissolved oxygen meter and an oxidation-reduction potential meter;

- Turbidity Meter;
- Laboratory supplied containers for the collection of samples; and,
- Teflon® tubing.

The groundwater sampling is comprised of synoptic water level measurements, field analysis, well purge techniques, low-flow sample collection, and decontamination procedures.

2.5.3 Synoptic Water Levels

Prior to all groundwater sampling events, depth-to-water will be measured in each well using an electronic water level indicator. The synoptic measurements will include the measurement of water levels and well depths in the monitoring wells in as short a time frame as possible to determine the potentiometric surface across the Site. The field personnel will measure the water levels in the wells to the nearest 0.01 foot using the surveyed point at the top of the inner well casing for reference.

2.5.4 Field Measurements

Field measurements that will be performed during well purging will include pH, specific conductivity, temperature, oxidation/reduction potential (ORP), dissolved oxygen (DO), and turbidity. Measurements will be collected by inserting the appropriate probe in a closed non-dedicated plastic container (flow-through-cell) that is rinsed with deionized water prior to purging the well. Turbidity samples will be collected from the flow-through-cell outflow.

Calibration of the instruments will be completed at the beginning of each sampling day, checked in the middle of the day, and as otherwise necessary based on the functioning of the meters and equipment. The following items outline the calibration of each meter. Each meter will be field calibrated in accordance with the manufacturer's specifications and appropriate calibration solutions. All calibrations will be recorded in the field logbook. Field calibration procedures at a minimum will include the following:

- Calibration of the field instruments will be performed by trained technicians prior to the mobilization of equipment to the Site. All the instruments will be calibrated as specified by the manufacturer. Standard solutions will also be checked to determine stability and operating conditions. All results of field calibrations and measurements will be maintained in bound field logbooks at least daily when the instrument is in use. The recorded calibration information will include date and time of calibration results.

- pH meters will be calibrated according to the manufacturer's instructions prior to each use and will, at a minimum, consist of three standard buffer solutions (pH 4, 7 and 10) obtained from chemical supply houses. The pH values of the buffers will be compensated for the temperature at which the pH sample is measured. Verification checks will be completed at least once per day using a standard solution. The verification check results must agree within ±0.05 pH standard units or re-calibrations will be performed.

- All temperature measurements will be measured using a field thermometer and recorded to ±0.2ºC.

- Dissolved oxygen meters will be calibrated to ambient air conditions.

- Specific conductance meters will be calibrated prior to each use using a potassium chloride solution (1,000 µmhos or 1,423 µmhos) as prepared by a qualified laboratory or chemical supplier.

- Turbidity meters will be calibrated daily prior to use by a minimum of two standards of known turbidity as prepared by the manufacturer of the instrument.

- Oxidation – Reduction Potential probes will be checked against standard solutions (at least one) prepared by a qualified laboratory or chemical supplier.

All calibration procedures performed will be documented in the field logbook and will include the date and time of calibration, name of the person performing the calibration, reference standards used and instrument readings.

If equipment fails calibration or equipment malfunction is noted during calibration or use, the equipment will be tagged and removed from service.

2.5.5 Purging Procedures

Sampling procedures will include water level measurements, calculation of well volumes, purging, and sampling activities. The following step-by-step procedures are in adherence to the Pennsylvania Department of Environmental Protection (PADEP) *Groundwater Monitoring Guidance Manual* (December 1999) and USEPA Region III groundwater sampling protocols for low flow pump purging and sampling, which are based upon the method of Puls and Barcelona (EPA/540/S-9S/504).

Step 1 Measure depth-to-water of every well at the Site.

Step 2 Calculate one well volume of the screened or open interval.

Step 3 Lower the low-flow pump in the well to collect groundwater samples. The pump intake will be placed at the approximate mid-point of the screened or open borehole section of the well. Likely groundwater sample interval depth/elevations are presented in the Well Construction Details (see Figure A-1).

Step 4 Calibrate meters.

Step 5 Begin to purge well. USEPA recommends a purge rate of 100 to 300 milliliters/minute (ml/min). The purge rate should not exceed the recharge rate (i.e., less than 0.3 feet of draw down from the static water level).

Step 6 Measure purging parameters at a minimum of 1 measurement every 3 to 5 minutes. Measurements will be collected via a flow-through-cell for pH, temperature, specific conductivity, ORP, and DO. Turbidity will also be measured at the outflow of the flow through cell every 3 to 5 minutes. All measurements will be recorded in the field logbook.

Step 7 After conductivity and temperature have stabilized to within 3% over three readings, pH readings differ <0.1 standard pH units, ORP readings differ within 10 mV, and turbidity measurements differ within ±10%, sampling can begin after the flow-through-cell is disconnected.

Step 8 The sample will be collected out of the discharge line. The date and time of the sample collection will be recorded in the field logbook.

The pump and sampling equipment will be decontaminated before and between each well. Decontamination procedures are presented in Section 4.2.5.

2.5.6 Sample Collection Procedures

Groundwater samples will be collected for total and dissolved RCRA 8 metals and TCL VOCs/SVOCs using the low-flow pump and tubing at a rate of 100 to 300 ml/min with the flow-through-cell disconnected. For total metal, VOC, and SVOC analyses, groundwater will be collected directly into a laboratory prepared, pre-preserved container. For dissolved metal analyses, groundwater will be transferred through a new 0.45 micron filter and then collected into a laboratory supplied, pre-preserved container. As per QAPP Table B-3 (see Appendix B), metal analyses samples will be collected in 500 ml HDPE bottles that are preserved with nitric

acid to a pH value of less than 2 standard units. SVOC analysis samples will be collected in 100 mL amber jars. VOC analysis samples will be collected in three (3) 40 mL VOA vials that are preserved with hydrochloric acid to a pH value of less than 2 standard units. VOC and SVOC samples will also be refrigerated or preserved with ice to maintain a temperature of less than 6 °C. Pump and tubing will be decontaminated while filters will be discarded after each use.

The selected analytical laboratory will perform all analyses in accordance with accepted USEPA publication SW-846 methods so that the detection limits are lower than the applicable RCG.

Sample handling will be in accordance with the procedures outlined in Section 5.0. The appropriate methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

2.5.7 Decontamination and Waste Handling

The pump will be disassembled and pump components and tubing will be decontaminated in the following manner.

- Alconox and water wash,
- Distilled water rinse,
- Nitric acid rinse (10% solution),
- Distilled water rinse, and,
- Air dry and store in plastic.

After decontamination of the pump components and tubing, two volumes of distilled water will be flushed through the pump assembly and tubing to ensure all decontamination fluids have been removed. Decontamination water will be collected for disposal with collected purge water.

2.5.8 Alternative (Passive) Groundwater Sampling

Site conditions sometimes prevent representative aquifer samples from being collected when employing low-flow sampling techniques. This typically occurs when constrictions in wells prevent low flow pumps from being deployed, there is poor recharge, or when easily mobilized sediments are continually entrained in pump purge water. If such conditions are encountered at the Site, passive samplers, such as Hydrasleeves, may be used to collect groundwater samples from the site wells.

Disposable one-time use Hydrasleeves would be deployed to the screened intervals of wells and allowed to remain in the wells for a minimum of 2 weeks before being retrieved. Hydrasleeve remain empty until time of collection, allowing wells to equilibrate with the surrounding aquifer. Sample material is taken directly from the Hydrasleeve via a disposable straw that pierces the Hydrasleeve. Once samples are collected, another Hydrasleeve is deployed and left in the well until the next sample event. Since wells are at equilibrium with the surrounding aquifer when Hydrasleeves are collected, there is no requirement to purge wells until stabilization is attained. All sampling equipment is dedicated or disposable so there is no decontamination required.

2.5.9 Purge/Waste Water Sampling

If groundwater sample data is not considered acceptable for disposal characterization of purge water; samples of containerized purged water will be collected using a bailer or a dip sampler and analyzed in accordance with disposal facility requirements.

2.6 QUALITY ASSURANCE/QUALITY CONTROL

The field quality control/quality assurance sample results will be used to assess the variability of the groundwater sample results at the end of the sampling period when considering whether corrective action is needed.

2.6.1 Temperature Blank

Temperature blanks (indicator) are designed to address sample preservation. The temperature blank is a vial (usually plastic) filled with water. Each cooler submitted to the laboratory will contain a temperature blank. Upon receipt by the laboratory, the sample custodian opens the cooler and places a NIST-traceable thermometer into the temperature blank, closes the lid and after a few minutes measures the cooler temperature. The temperature blank is used for analytical analysis. Samples for all analytical parameters, except for metals, are required to be cooled to 4ºC ± 2ºC after the sample has been collected into the appropriate container(s). For samples that are received above 6ºC, the laboratory will contact the Advanced GeoServices QA Manager to determine the appropriate corrective action.

An alternate method of measuring the cooler temperature is through the use of an infrared digital thermometer. The sample custodian in this case, opens the cooler, aims the thermometer at the inside of the cooler, pulls the trigger and reads the temperature from the digital display. These kinds of thermometers are not NIST-traceable, but are calibrated by the manufacturer.

2.6.2 Equipment (Rinsate) Blank

Equipment blanks will be collected to ensure that the sampling equipment has been properly decontaminated and that the potential for cross contamination has been minimized by the decontamination procedures. These blanks will be collected by decontaminating the sampling device and pouring ultrapure de-ionized water (provided by the laboratory) over the device and collected into the appropriate sample containers. One equipment blank per sampling event will be collected whenever non-dedicated or non-disposable equipment is used. The equipment blanks will be analyzed for identical parameters as the samples.

2.6.3 Trip Blank

Trip blanks are designed to address sample contamination from ambient/environmental sources outside of the Site and laboratory. The trip blank typically three (3) 40-mL VOA vials filled with deionized water. Each grouping of samples submitted to the laboratory for VOC analysis will contain a trip blank.

2.6.4 Field Duplicates

These samples will be collected to allow for the determination of analytical and sampling precision. Two field duplicate samples per sampling event will be collected and submitted for the identical parameters as the true sample. In order to prevent bias, these samples will be submitted blind to the laboratory and will not be identified as field duplicates on the Chain of Custody (CHOC).

2.6.5 Matrix Spike/Matrix Spike Duplicates

These samples will also be submitted as further QC checks. These will be collected at the frequency of two MS/MSD pairs per sampling event. These will allow for the accuracy to be determined by the percent recovery of the compounds spiked. The laboratory will perform the spiking of the MS/MSDs. Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to the samples collected from the Site. MS/MSD samples will be identified on the CHOC.

2.7 REPORTING

The groundwater monitoring program will generate data concerning the groundwater quality and hydrogeologic conditions at the Site. The data will be tabulated and transmitted to the USEPA in the Phase III RFI Report after the data have been validated. The form of the evaluation and reporting to be included in the report is detailed in the following outline:

- **Introduction** - including date and wells monitored.

- **Groundwater Sampling and Analysis Procedures** - This section will summarize well purging, sample collection procedures, field parameters, sample preparation and preservation, and decontamination procedures.

- **Groundwater Flow Direction** - Depth-to-water information will be converted to groundwater elevation data using the top-of-casing as the reference point and presented in tabular form. Elevations will be plotted on a Site map to illustrate the potentiometric surface of the groundwater. .

- **Groundwater Quality** - Groundwater quality data will be tabulated, and validated. A brief validation quality report consisting of general information and qualifying statements that describe the usability of the data, difficulties encountered, and qualifier codes defining the reliability of the samples will be presented in data summary tables. Data deliverables from the laboratory will be paginated, and include at a minimum:

  - a page including the facility name and address, laboratory certification number, date of analytical report preparation, and signature of laboratory director or group leader;
  - a listing of the field sample identification number and the corresponding laboratory sample identification numbers;

- a listing of analytical methods used;
- detection limits of each analyte;
- tabulated sample results, including date of analysis;
- method blank results;
- instrument raw data; and,
- chain of custody documents.

Any issues noted in the data validation will be reviewed to determine its impact on the data interpretation. Groundwater analytical data will be compared to historic groundwater data for the Site and the appropriate Pennsylvania water quality criteria.

- **Conclusion and Recommendations** - Will include a statement about the general Site groundwater conditions, comparison of data to the performance standards and recommendations for any follow-up activities.

3.0 SOIL BORINGS

The objective of the soil borings proposed for the RFI is to supplement the existing limited subsurface information by obtaining samples of the fill/soil materials encountered for chemical analysis. Soil borings will be performed across the Site (random sampling) with respect to inorganic contaminants (RCRA 8 metals) with an emphasis on certain SWMUs (SWMU-2, 3, 5, and 9) and AOCs (focused sampling) where impacts from past operations is possible. Soil sampling for VOCs/SVOCs will be performed for soil samples collected from the proposed groundwater monitoring wells.

The following field activities will take place for the soil borings:

- Utilities will be marked in the areas where drilling will take place.
- The boring locations will be staked out in the field using spray paint markings on asphalt or concrete surfaces and modifying the location to avoid utilities.
- If necessary, concrete surfaces will be saw cut or cored prior to drilling.
- Plastic will be used on the ground surface around the borehole to contain the drill cuttings.
- The soil borings will be advanced using direct-push (Geoprobe™) techniques. Soil sampling for monitoring wells will be collected using hollow stem augers. Samples will be obtained using acetate sleeves (direct push) or a split spoon sampler (hollow stem auger) on a continuous basis until the end of the boring or refusal. An experienced field technician working under the supervision of and Indiana Professional Geologist will log the borings, for split spoon sampling the number of blows required to advance the sampler 6 inches will be recorded, and the soil/fill types encountered and the presence of any free water in the subsurface will be documented. Drill cuttings and excess sample volume will be containerized for proper disposal.

- Representative samples of soils and fill materials encountered will be taken from the acetate sleeve or split spoon sampler and analyzed. Sample homogenization will take place in the laboratory (inorganics only).
- After the soil boring is completed, the hole will be grouted and the surface restored to its original condition.
- Reusable sampling equipment that is contact with soils will be decontaminated between borings using DI water and a scrub brush. The split spoon sampler will be dry brushed to remove visible soil between samples and decontaminated using a water-Alconox solution between boring locations.
- The boring location and ground surface elevation will be surveyed.

3.1 DATA QUALITY OBJECTIVES

Soil borings will be conducted to obtain samples of the soils/fill material encountered for chemical analysis. To meet this goal, the following data quality objectives (DQOs) have been established. To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection necessary to meet the applicable decision criteria as defined in USEPA *Guidance for the Data Quality Objective Process* dated August 2000 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process are presented below.

3.1.1 State the Problem

There is a potential that soils/fill materials onsite associated with the former manufacturing area (SWMUs and AOCs) and on the overall site outside of the manufacturing areas may contain lead and other heavy metals at elevated concentrations. Previous investigations have provided some indication that these contaminants are only present in shallow fill areas.

Impacts from VOCs/SVOCs requiring further investigation have been observed in the vicinity of the former fuel shed during removal of UST-2. Soils with impacts above the residential screening level were removed during the UST removal, although groundwater impacts from VOC/SVOCs may remain. Soil sampling will be performed to confirm that these contaminants are not present in soil at concentrations above the RCGs.

3.1.2 Identify the Decision

Soil borings will be conducted to characterize site subsurface conditions. Results of soil sampling will be utilized in conjunction with results from previous environmental investigations to characterize conditions and delineate limits of historic impacts. Depending on the magnitude of those impacts, further evaluation, corrective measures and/or risk evaluation may be necessary.

3.1.3 Identify Inputs to the Decision

Site specific performance standards have not been established for soils or fill materials at the site. Results of soil sampling will be compared to IDEM 2017 OLQ Screening Levels established for soil to determine whether or not further evaluation under the RCRA Corrective Action Process is necessary. The Site is a commercial/industrial property and the screening levels for this use are the most appropriate. These are referred to as the RCGs. VOC results will also be compared to the migration to groundwater screening level.

Pace Analytical is proposed for analysis of soil boring samples. The proposed laboratory participates regularly in performance evaluation audits as part of their laboratory certification requirements. Pace Analytical is a NELAP certified laboratory. USEPA SW-846 methods will be used in the sample analysis of priority pollutant metals; see Table B-3 in the QAPP (Appendix B) for the specific methods.

3.1.4 Define the Study Boundaries

Soil borings using a hollow-stem augur will be completed to depths of up to 15 to 20 feet bgs (new monitoring well depth) at five (5) locations throughout the site. Soil samples will be obtained continuously in the borings by means of the Standard Penetration Test (ASTM D 1586) using split spoon samplers. Samples for laboratory analysis of VOCs/SVOCs will be collected from intervals of 0-4 feet bgs, 4-8 feet bgs, 8-12 feet bgs, 12-16 feet bgs, and 16-20 feet bgs (if depth exceeds 15 feet).

At sixty five (65) locations (R-1 through R-55 and F-1 through F-10) within the Site boundary (see Figure 3 of RFI Work Plan) geoprobe soil borings will be performed to a depth of eight feet below the ground surface (top of pavement) to collect soil samples from the following intervals: 0-1 feet bgs. 1-2 feet bgs, 3-4 feet bgs, 5-6 feet bgs, and 7-8 feet bgs.

In the vicinity of the former UST-2, six (6) geoprobe soil borings (U-1 through U-6) will be performed to a depth of eight feet bgs to collect one sample each from the 0-4 feet bgs and 4-8 feet bgs intervals. The specific location of the sample will target the highest observed PID measurement and/or areas with staining or odors observed. At any given boring location, the boring depth may be increased if evidence of deeper contamination is observed (discoloration, odors, debris, PID measurments). Soil samples from the UST-2 borings will be analyzed for VOCs/SVOCs and diesel (DRO) and gasoline range (GRO) total petroleum hydrocarbons (TPH).

Soil sampling during the RFI will follow the sampling protocols presented below. See Figure 3 in the RFI Work Plan for the proposed soil boring locations.

3.1.5 Develop a Decision Rule

All split spoon and geoprobe samples with sufficient recovery will be logged for soil characteristics. Results of the laboratory analysis will be tabulated and presented in the RFI Report.

3.1.6 Specify Limits of Decision Errors

The sample data are subject to random and systematic errors during field collection and sample analysis. The combination of errors is referred to as "total study error." The two contributors to the total study error are the statistical sampling error and the measurement error. The statistical sampling error occurs when the data collection plan fails to provide a limited variability within the decision unit necessary for accurate decision. Measurement errors are introduced during the processes to which the sample is subjected such as physical collection, sample handling, sample preparation, sample analysis, and data reduction. Since the total study error directly affects the possibility of making a decision error, the total decision error must be managed by minimizing the sample design and measurement errors.

The following are some ways to minimize the statistical sampling error:

- use standardized sampling techniques when performing the soil borings to minimize variations in recovery over the sampling intervals.

The following are some ways to minimize measurement errors:

- have the samplers collect the samples in a standardized manner;
- ensure that each sample is properly preserved, labels, and transported to the laboratory under chain-of-custody;
- specify that the laboratory has to use accepted USEPA Methods and reports the data using the proper units;
- specify that the laboratory participates in regular performance testing and also has NELAP certification;
- receive the analytical data from the laboratory in an electronic format to minimize transcription errors; and,
- perform a qualitative review (data validation) of the analytical data, as specified in the QAPP (see Appendix B), to verify the reliability of the data.

3.1.7 Optimize the Design for Obtaining Data

Upon review of the analytical data, the sampling frequency may be changed to optimize the design. The design options will be evaluated based on cost and the ability to meet the DQOs and presented to the USEPA for approval.

3.2 SAMPLE LOCATION AND FREQUENCY

The proposed soil borings are shown on Figure 3 of the RFI Work Plan. Samples will be collected as described in Section 3.1.4. Physical characteristics of soil samples will be logged on standard geotechnical boring logs. Samples representative of the various soil and fill materials encountered in each sample interval will be sent for laboratory analysis.

3.3 SAMPLE DESIGNATION

Soil boring samples will be identified using the boring identification and depth interval (e.g., R17-3-4, etc.).

3.4 FIELD PROCEDURES

3.4.1 Utility Location and Mark-out

Prior to drilling, Indiana One-Call system will be notified to mark out the underground public utilities in the areas of the study. Coordination of the field work with Exide's caretaker will also be conducted during one site visit with the driller prior to the start of work to locate the borings in the field and to adjust boring locations as needed based upon site restrictions or the presence of utilities. The assistance of the Site caretaker during the site visit will also be requested to identify any private underground utilities/structures in the areas of study. A private utility locating service will also be contracted to identify utilities where test borings are being performed.

3.4.2 Sampling Equipment

The following equipment will be used for the soil boring sampling:

- Concrete coring equipment;
- Plastic sheeting;
- Standard drilling rig with Hollow Stem Augur capable of drilling to depths at least 40 feet;
- Split spoon sample equipment;
- Geoprobe ™ direct push boring equipment (with acetate sleeves) capable of boring to depths of at least 20 feet;
- Photo-ionization Detector (PID);
- En-Core or Terra-Core samplers;
- Electronic depth to water meter; and,
- Laboratory supplied containers for the collection of samples.

The soil boring sampling is comprised of drilling or boring, split spoon or acetate sleeve sample collection, field characterization of physical features, homogenization for metals analysis, and decontamination procedures.

3.4.3 Field Measurements

In cases where groundwater is encountered during drilling activities, depth-to-water will be measured in the boring using an electronic water level indicator. The field personnel will measure the water levels in the borings to the nearest 0.01 foot from the ground surface for reference.

3.4.4 Sample Collection Procedures

Soil samples from borings R-1 through R-55, F-1 through F-6, and F-10 will be collected for total RCRA 8 metals. Soil samples from borings R-20, R-21, R-28, R-29, R-30, and R-37 will also be analyzed for pH.

In borings F-7 through F-9, U-1 through U-6, and MW-1 through MW-5 VOC and SVOC analysis will be performed. Samples from borings U-1 through U-6 will also be analyzed for DRO/GRO TPH.

As per QAPP Table B-3 found in Appendix B, the samples will be collected in 4-oz glass jars (or larger) or resealable bags (inorganic analysis) that are unpreserved. VOC samples will be collected using En-Core or Terra Core techniques. The selected analytical laboratory will perform all analyses in accordance with USEPA publication SW-846 methods.

Sample handling will be in accordance with the procedures outlined in Section 4.0. The appropriate methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

3.4.5 Decontamination and Waste Handling

Sample equipment will have soil particles removed via dry brushing between various sample depths at the same boring location. Split spoons and geoprobe drive shoes will be disassembled and decontaminated between soil boring locations in the following manner.

- Alconox and water wash;
- Distilled water rinse;
- Distilled water rinse, and,
- Air dry.

Investigation derived waste (IDW) includes decontamination water, soil spoils, trash , and PPE generated during the investigation. Decontamination water will be collected and stored onsite until it can be disposed of offsite. Soil spoils will be collected in a drum and stored onsite until it can be disposed of offsite. Trash and PPE will be placed in plastic trash bags. Disposal of IDW will be performed by Exide.

3.5 QUALITY ASSURANCE/QUALITY CONTROL

The field quality control/quality assurance sample results will be used to assess the variability of the soil sample results at the end of the sampling period when considering whether corrective action is needed.

3.5.1 Temperature Blank

Temperature blanks will be utilized for samples (groups of samples) that are designated for VOC/SVOC analysis, but are not required for soil samples designated for metals analysis.

3.5.2 Equipment (Rinsate) Blank

Equipment blanks will be collected to ensure that the sampling equipment has been properly decontaminated and that the potential for cross contamination has been minimized by the decontamination procedures. These blanks will be collected by decontaminating the sampling device and pouring ultrapure de-ionized water (provided by the laboratory) over the device and collected into the appropriate sample containers. One equipment blank per sampling day will be collected whenever non-dedicated or non-disposable equipment is used. The equipment blanks will be analyzed for identical parameters as the samples collected on that day.

3.5.3 Trip Blank

Trip blanks are designed to address sample contamination by VOCs from ambient/environmental sources outside of the Site and laboratory. The trip blank typically three (3) 40-mL VOA vials filled with deionized water. Each grouping of samples submitted to the laboratory for VOC analysis will contain a trip blank.

3.5.4 Field Duplicates

These samples will be collected to allow for the determination of analytical and sampling precision. One field duplicate sample per twenty samples will be split from the parent sample after homogenization and submitted for the identical parameters as the parent sample. In order to prevent bias, these samples will be submitted blind to the laboratory and will not be identified as field duplicates on the Chain of Custody (CHOC).

3.5.5 Matrix Spike/Matrix Spike Duplicates

These samples will also be submitted as further QC checks. These will be collected at the frequency of one MS/MSD pair per twenty samples collected and will be split from the parent sample after homogenization. The MS/MSD samples will allow for the accuracy to be determined by the percent recovery of the compounds spiked. The laboratory will perform the spiking of the MS/MSDs. Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. The purpose of these laboratory spikes is to monitor any possible matrix effects specific to the samples collected from the Site. MS/MSD samples will be identified on the CHOC.

3.6 REPORTING

The soil boring sampling will generate data concerning the subsurface conditions and extent of fill material at the Site. The data will be tabulated and transmitted to the USEPA in the RFI Report after the data has been validated. The form of the evaluation and reporting to be included in the report is detailed in the following outline:

- **Introduction** - including date, number and depth of borings completed.

- **Soil Boring Sampling and Analysis Procedures** - This section will summarize drilling, sample collection procedures, geotechnical characterization, and decontamination procedures.

- **Subsurface Profile** – Auger/Geoprobe refusal depth information and nature of materials encountered will be converted to elevation data using the ground surface as the reference point. The soil boring information generated in the RFI will be combined with subsurface information from previous investigations conducted at the Facility to gain an understanding of the extent and character of the soil and fill at the site.

- **Soil Analytical** - Soil data will be tabulated, and validated. A brief validation quality report consisting of general information and qualifying statements that describe the usability of the data, difficulties encountered, and qualifier codes defining the qualitative and quantitative reliability of the samples will be presented in data summary tables. Data deliverables from the laboratory will be paginated, and include at a minimum:

  - a page including the facility name and address, laboratory certification number, date of analytical report preparation, and signature of laboratory director or group leader;

- a listing of the field sample identification number and the corresponding laboratory sample identification numbers;
- a listing of analytical methods used;
- detection limits of each analyte;
- tabulated sample results, including date of analysis;
- method blank results;
- instrument raw data; and,
- chain of custody documents.

Any issues noted in the data validation will be reviewed to determine its impact on the data interpretation.

- **Conclusion and Recommendations** – This section will include a description of the general Site subsurface soil conditions, and recommendations for follow-up activities, if any.

4.0 SAMPLE HANDLING PROCEDURES

4.1 SAMPLE HANDLING

All sample containers will be identified by the use of sample labels with the sample identification. Each sample label will be filled out by the sampler to avoid any possibility of sample misidentification and attached to the sample container. Indelible ink will be used to complete the sample labels. Each sample label will be labeled at the time of collection with, at a minimum, the following information:

- Sample identification;
- Initials of the sample collector;
- Time and date of the sample collection;
- Site name and location number (if any);
- Requested analyses;
- Any preservative added or field preparation performed; and,
- Sample designation if this sample is a quality assurance sample.

Each member of the sampling team will use a new pair of gloves at each sample location; however, the same pair of gloves can be used when collecting samples for compositing.

The field sampler will maintain custody of the samples following the procedures outlined in the following sections until the samples are properly relinquished to the laboratory or a common carrier for delivery to the laboratory or are archived on-site. Once at the laboratory, each sample will be assigned a unique laboratory identification number that will be used for analysis assignment, sample tracking, and data reporting while the samples are at the laboratory.

4.2 SAMPLE CUSTODY/SAMPLE CONTROL

A sample is physical evidence collected from the Site. Due to the evidentiary nature of the data generated from sampling, sample custody must be traceable from the time the empty sample containers are prepared by the laboratory through the reporting of the results of the analyses. Therefore, sample control procedures have been established to ensure sample integrity. All sample containers and samples will be maintained under strict custody procedures throughout the investigation. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

A sample, sample container, or evidence file will be considered under custody if:

- An item is in the actual possession of the person; or
- The item is in the view of the person, after being in actual possession of the person; or
- The item was in the person's actual physical possession but is now locked up or sealed in a tamper-proof manner; or
- The item is placed in a designated secured restricted area.

4.2.1 Field Custody Procedures

The field personnel in charge of collecting the samples will maintain custody of the samples collected. The field personnel will be responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory or archived. The appropriate sample containers, preservatives, methods for extraction and analysis and required holding times to be met are given in the QAPP in Appendix B.

4.2.2 Field Data Documentation/Field Logs

A system of logging all pertinent data collected during sampling operations will be maintained using a dedicated field logbook(s). Each page will be numbered, dated and initialed by the person making the entry. All entries will be made in indelible ink. Incorrect entries will be crossed out with a single line and verified with the recorder's initials. At the completion of the day, if a page is not complete, a diagonal line will be drawn through the remainder of the page with the recorder's signature at the bottom.

All sample locations will be recorded in the field logbook and referenced to the site map so that each location is permanently established. Samples will be tagged with all pertinent site information at the time of sampling. Pertinent site information to be supplied in the field logbook for each task is listed below:

- Signature of recorder;
- Name and location of sample;
- Date and time of arrival and departure;
- Names of all personnel on-site and their affiliation;
- Purpose of the visit/description of the field activity;
- All field instruments used, date and time of calibration and calibration checks, method of calibration, and standards used;
- All field measurement results;
- Date, time, and location of all sampling;
- Method of sample collection;
- Any factors which could affect sample integrity;
- Name of sampler(s);
- Sample identification, sample description, and sample preservation, if any;
- Documentation of all conversations with the client, agency personnel, field decisions and approval; and,
- Weather conditions.

Field logbooks should contain only factual information entered as real-time notes, which will enable the user to recreate events on-site. In addition, chain-of-custody records will be prepared and kept as part of the field records.

4.2.3 Chain-of-Custody Procedures

The following chain-of-custody procedures will be used for this project:

- New, certified clean sample containers will be prepared and relinquished by the laboratory on a chain-of-custody record. The chain-of-custody record will be kept as part of the permanent record.
- Any transfer of custody of containers of samples will be noted on a chain-of-custody record.
- Each sample collected for the event will be entered on the chain-of-custody record.
- The chain-of-custody will be completed as soon as possible after sample collection. The following information must be supplied to complete the chain-of-custody record.

  - Site specific project name and number;
  - Signature of sampler(s);
  - For each sample, sampling station number, date and time (military is preferred) of collection, grab or composite sample designation, and a brief description of the type of sample and sampling location;
  - Number of sample containers per each sample location;
  - Analysis required;
  - Type of preservative;
  - Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time they received the samples/cooler on the record; and,

- Type of carrier service.

The original chain-of-custody record will accompany the sample containers during transport to document their custody.

If custody is relinquished through a common carrier for delivery to the laboratory, the following protocol will be followed:

- In the space for the sample receiver, the name of the common carrier and the date relinquished will be written. In addition, if known, the tracking number will be included on the chain-of-custody record;
- The original completed chain-of-custody record will be placed inside the shipping package; and,
- The shipping package will be sealed with tape and custody seals affixed. The seals will be placed on the package in such a manner that the package cannot be opened without breaking the seals. The seals will serve to document that the shipping container was not opened during the shipment through the common parcel carrier.

4.2.4 Sample Shipment Procedures

Prior to expiration of holding times for any parameters, all samples scheduled for analysis will be packaged in shipping containers for shipment to the analytical laboratory using the following steps:

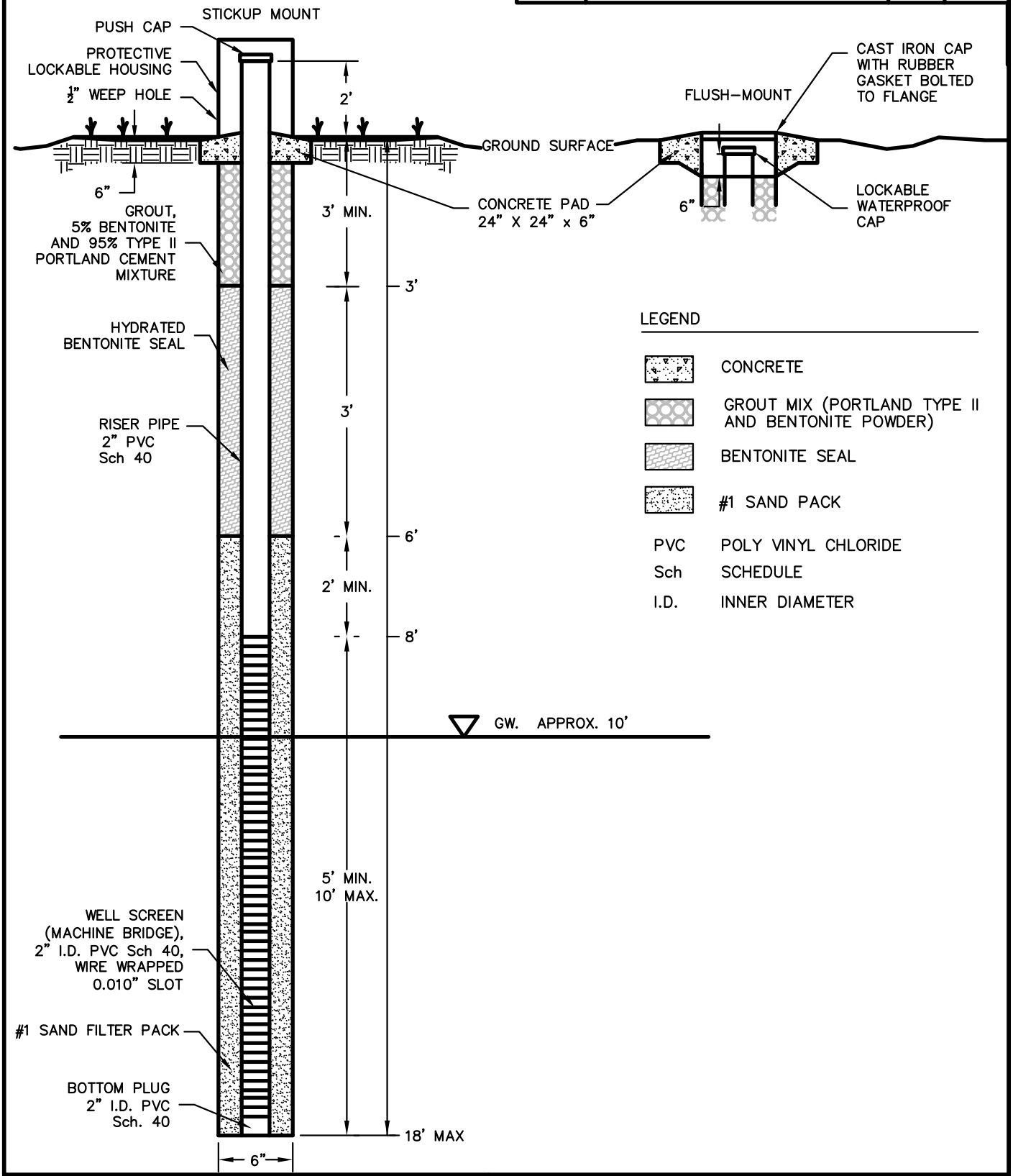
1. Check each sample container for a properly completed sample identification label.
2. Ship the samples in a large capacity cooler, or specific laboratory prepared sample-shipping container. Place packing material on the bottom of the cooler to prevent sample bottle breakage.

3. Place the sample bottles in the shipping container in a manner such that they do not touch and will not touch during shipment. Secure with packing material as needed to fill void space.
4. A cooler with samples to be analyzed for metals only do not need to be cooled (iced) during shipping.
5. Place the original chain-of-custody record in a plastic bag, seal, and tape it to the inside of the shipping container lid.
6. Retain the pink copy of the chain-of-custody for the Advanced GeoServices QA Manager.
7. Tape the cooler drain shut. Tape the cooler or shipping container closed at a minimum of two locations.
8. Place two signed and dated custody seals across each edge of the shipping container.
9. Attach the completed shipping label to the top of the shipping container.
10. Relinquish the cooler to the courier with the required signed and dated handbill/waybill.
11. Retain receipt of the handbill/waybill as part of the permanent documentation.

If the sample coolers are not shipped but instead picked-up by the laboratory courier, step number 6 and 12 will be omitted and the chain-of-custody will be handed to and signed by the laboratory courier. The pink copy of the chain-of-custody will be maintained by the sampler and submitted to the Advanced GeoServices QA Manager.

FIGURE

Revision	Description	Date	By



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MONITORING WELL CONSTRUCTION DETAILS
RFI SAP
EXIDE TECHNOLOGIES
FRANKFORT, INDIANA

PROJECT ENGINEER:	P.G.S.	SCALE:	N.T.S.
CHECKED BY:	P.G.S.	PROJECT NUMBER:	2011-2678-11
DRAWN BY:	J.S.D.	DATE:	FIGURE: A-1

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APPENDIX B

Quality Assurance Project Plan (QAPP)

QUALITY ASSURANCE PROJECT PLAN
FOR THE
RCRA FACILTY INVESTIGATION
EXIDE TECHNOLOGIES FORMER MANUFACTURING FACILITY
Frankfort, Indiana

Prepared for:

EXIDE TECHNOLOGIES
Milton, Georgia

Prepared by:

ADVANCED GEOSERVICES
West Chester, Pennsylvania

Project No. 2011-2678-11
October 12, 2017

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Project No. 2011-2678-11
October 12, 2017

Paul G. Stratman, P.E.
Project Manager

Amy K. Graham
Quality Assurance Scientist

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ATTACHMENT

B-1 Laboratory Quality Assurance Manual

1.0 PROJECT MANAGEMENT

1.1 PROJECT/TASK ORGANIZATION

This Quality Assurance Project Plan (QAPP) has been developed to present the quality assurance measures that will be used during the RFI field activities at the Exide Technologies' former manufacturing facility property located at 555 North Hoke Avenue in Frankfort, Indiana (the Site). The QAPP has been prepared based on guidance presented in the "United States Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans," (QA/R-5, EPA/240/B-01/003, March 2001) and the "Guidance on Systematic Planning Using the Data Quality Objective Process" (QA/G-4, EPA/240/B-06/001, February 2006).

While all personnel involved in an investigation and in the generation of data are implicitly a part of the overall project and quality assurance program, certain individuals have specifically delegated responsibilities. The RFI supervising professional firm is Advanced GeoServices Corp (Advanced GeoServices). The Advanced GeoServices personnel with quality assurance/quality control (QA/QC) responsibilities are the Project Coordinator, QA Manager, QA Scientist, and the field technicians. For samples collected by Advanced GeoServices personnel, the analyses of the samples will be performed by Pace Analytical located in Indianapolis, Indiana. The laboratory retains the responsibility for analytical data quality assurance, however; specific laboratory personnel with QA/QC responsibilities include the Laboratory QA Officer and Laboratory Sample Custodian.

1.1.1 Project Coordinator

The Project Coordinator is an experienced manager and technical professional who assists in the coordination of the RFI, participates in major meetings and regulatory negotiations and provides upper level contact for the client. The designated Project Coordinator is Paul Stratman, P.E, P.G.

1.1.2 QA Manager

The QA Manager will work on all projects requiring the collection of data, and as such is not directly involved in the routine performance of the technical aspects of the investigations. The QA Manager's responsibilities include the development, evaluation, and implementation of the QAPP and procedures appropriate to the investigation. Additional responsibilities include reviewing project plans and revising the plans to ensure proper QA is maintained. The QA Manager is also responsible for all data processing activities, data processing QC, and final analytical data quality review.

It is a primary responsibility of the QA Manager to ensure that all personnel have a good understanding of the QAPP, and understanding of their respective roles relative to one another, and an appreciation of the importance of the roles to the overall success of the program.

1.1.3 QA Scientist

The QA Scientist has primary responsibility for analytical data validation and review. In this capacity, the QA Scientist will prepare data validation reports describing data usability and analytical QC problems encountered.

1.1.4 Field Technicians

Experienced Advanced GeoServices Field Technicians will conduct all sampling tasks to be conducted by Advanced GeoServices. One of the Field Technicians will be designated as the Field Team Leader. Their responsibilities will include the documentation of the proper sample collection protocols, sample collection, field measurements, calibration of field equipment, equipment decontamination, and logbook and CHOC documentation.

1.1.5 Analytical Laboratory QA Officer

The QA Officer has the responsibility for maintenance of all laboratory QA activities and documentation. The role and responsibilities of the laboratory's QA Manager has been included as part of the laboratory quality assurance manual (QAM). The QAM from Pace Analytical, Indianapolis has been included in an electronic format as Attachment B-1

1.1.6 Analytical Laboratory Sample Custodian

The Sample Custodian's responsibilities include ensuring proper sample entry and sample handling procedures by laboratory personnel.

1.2 QUALITY OBJECTIVES AND CRITERIA

Data quality objectives (DQOs) have been established as described in the sampling and analysis plan. DQOs are qualitative and quantitative statements specifying the quality of the environmental data required to support the decision making process. Separate DQOs are designed for field sampling and laboratory analysis so that clear distinctions can be isolated with respect to cause between any problems found in the system. Conversely, the DQOs are also designed to provide an indication in the form of data quality indicators (DQIs) of the variability of the overall system. The overall QA objective is to keep the total uncertainty within an acceptable range that will not hinder the intended use of the data. To achieve this, specific data requirements are required such as precision, accuracy, sample representativeness, data comparability, data completeness, and sensitivity.

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 also characterizes the natural variation of the matrix and how the contamination

exists or varies within that matrix. Precision is measured by comparison of the field duplicate with the original parent sample and by comparison of the specific compound recoveries for the matrix spike/matrix spike duplicate samples. For this project, the DQO for precision is to meet the performance criteria listed with the method as shown in Table B-1.

Accuracy

Accuracy is defined as the degree of agreement of a measurement or an average of measurements with an accepted reference value. Accuracy measures the bias in a measurement system which 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, sample handling, matrix interference, and sample preservation. The field component of accuracy will be reduced if the sampling, preservation and handling techniques described in the SAP and this QAPP are followed. Accuracy in the laboratory methods and procedures will be evaluated through the use of matrix spike and matrix spike duplicate samples. For this project the DQI for accuracy is to meet the performance criteria listed within the method.

Representativeness

Representativeness expresses the degree to which sample data represents the characteristics of the environment from which they are collected. Samples that are considered representative are properly collected to accurately characterize the contamination at a sample location. Therefore, the samples will be collected in a standardized method. Representativeness will be measured by a review of the precision obtained from the field and laboratory duplicate samples.

Comparability

Comparability expresses the confidence with which one data set can be compared with another data set from a different phase or from a different program. Data comparability will be ensured by the control of sample collection methodology, analytical methodology, and data reporting.

Completeness

Completeness is defined as the percentage of data that is judged 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, or poor accuracy, precision and laboratory error. While deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. The level of completeness, with respect to usable data, will be measured during the data assessment process by comparing the total number of data points to the number of data points determined to be usable. A completeness of 90% or greater of unrejected analytical data is the objective for the project.

Sensitivity

Sensitivity is used broadly to describe the instrument detection/method detection/reporting limits established to meet project-specific DQIs. Limits have been established to describe project sensitivity requirements, such as method detection limits (MDLs) and reporting limits (RLs). All off-site laboratory analysis will be performed using USEPA methods. Table B-2A and Table B-2B contains the laboratory reporting limits and method detection limits for the analytical parameters. Specific reporting limits are highly matrix-dependent and may not always be achievable.

1.2.1 Project Data Quality Objectives

To determine the project DQOs, a series of planning steps are used. The DQO development process is used to optimize the data collection necessary to meet the applicable decision criteria as defined in USEPA *Guidance on Systematic Planning Using the Data Quality Objective Process* dated February 2006 and USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* dated January 2000. The seven steps of the DQO process for each sampling activity have been integrated into the individual sampling plans.

To achieve the DQOs, this QAPP is designed to ensure that a sufficient number of samples will be collected using technically valid, scientific procedures. The DQOs for this project require qualitative validation. Substantiating the results of the data provides the level of confidence in the data necessary so decisions can be made regarding the monitoring activities and long-term concerns.

1.2.2 Field Investigation Quality Objectives

The main field investigation DQO is to collect high quality data using the proper collection techniques in a repeatable and consistent manner. The SAP discusses the boundaries and the decision rule for each sampling activity. To reduce the random and systematic errors that are introduced in the measurement process during physical sample collection, sample handling, and sample analysis, field duplicates, equipment blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples will be collected.

- Field duplicates are independent samples collected in such a manner that they are equally representative of the sampling point and parameters of interest at a given point in space and time. Field duplicate samples provide precision information of homogeneity, handling, shipping, storage, preparation, and analysis. One field duplicate will be collected for every twenty (20) samples per matrix.

- Trip blanks serve to detect possible cross-contamination of aqueous samples by volatile organic compounds (VOCs) resulting from handling, storage and shipment procedures. Trip Blanks will be submitted for VOC analysis whenever VOC samples are collected at a rate of one per shipment.

- Equipment blanks are designed to address cross-contamination between sample sources in the field due to deficient equipment decontamination procedures. This blank also addresses field preservation procedures, environmental Site interferences, and the integrity of the source water for field

decontamination/cleaning. The equipment blanks will be analyzed for identical parameters as the samples. One equipment blank will be collected per day per matrix or per 20 samples per matrix per day (whichever is more frequent) when sampling equipment is decontaminated.

- The MS/MSD samples monitor any possible matrix effects specific to samples collected from the Site. In addition, the analysis of MS/MSD pairs checks precision by comparison of the two spike recoveries Precision will also be assessed by comparison of the MS/MSD relative percent duplicate results. One MS/MSD pair will be collected for every twenty (20) samples collected per matrix.

The acceptance criteria for the above QA/QC samples are presented in Table B-1.

1.2.3 Laboratory Data Quality Objectives

The "ideal objective" for the laboratory is to analyze the samples by the most appropriate analytical method to meet the cleanup criteria, which in turn will provide the highest quality data possible. However, it is not probable that the analyses will be performed flawlessly and without any need to re-extract or re-analyze samples due to necessary dilutions, sample matrix, poor surrogate recoveries, analyst error, etc. To reduce the random and systematic errors that are introduced in the measurement process during sample handling, sample preparation, sample analysis, and data reduction; method blanks, laboratory control samples (LCSs), MS/MSD, and laboratory duplicate samples will be included during preparation and analysis of the Site samples.

- Method blanks are generated within the laboratory during the processing of the actual samples. These blanks will be prepared using the same reagents, procedures, and at the same time as the project samples are being analyzed. If contamination is found in the method blank, it indicates that similar contamination found in associated samples may have been introduced in the laboratory and may not have been present in the samples themselves. Guidelines for accepting or rejecting data based on the level of contamination found in the method blank are presented in the

specified analytical method. A minimum of one method blank per twenty (20) samples will be analyzed or, in the event that a quality control batch consists of less than twenty (20) samples, one method blank sample will be analyzed per batch.

- MS/MSD samples determine the accuracy by the recovery rates of the compounds added by the laboratory (the spiking compounds are defined in the analytical methods). The MS/MSD samples also monitor any possible matrix effects specific to samples collected from the Site and the extraction/digestion efficiency. In addition, the analysis of MS/MSD samples check precision by comparison of the two spike recoveries.

- The laboratory control sample (LCS) is prepared by the laboratory by adding analytes of known concentrations to solution (de-ionized water for metals) for the selected analyses. The LCS is prepared, analyzed, and reported once per quality control batch. The LCS must be prepared and analyzed concurrently with the samples in the batch using the same instrumentation as the samples. The LCS is designed to access the capability of the laboratory to perform the analytical methods. If the analytes present in the LCS are not recovered within the criteria defined in the specified analytical methods, the samples will be re-analyzed or the laboratory will qualify the data.

The acceptance criteria for the laboratory QC checks are presented in Table B-1.

1.2.4 Criteria Objectives

Criteria objectives are presented in Table B2. The table also contains the laboratory reporting limits (RLs) for organics and method detection limits (MDLs) for inorganics to show that the analytical methods selected are below or meet the criteria objectives. The laboratory will be expected to report the RLs or MDLs for all samples in the appropriate statistical reporting units for all analytes.

1.2.5 Data Management Objectives

It is a data management objective that all aspects of the investigation from the sample design, collections, shipment, analysis use/decisions, etc. be performed in conjunction with rigorous QA/QC documentation. The specific details of this documentation can be found throughout this document.

It is expected that by the design of separate data quality requirements for field sampling and laboratory analysis, clear distinctions can be made such that any problems found in the system can be isolated with respect to the cause. Conversely, the data quality requirements are also designed to provide an indication of the variability inherent to the overall system. The overall data management objective is to provide a complete database with a high degree of confidence through the use of a phased approach of sampling, analysis, data assessment (data review), data qualification, and feedback.

1.3 SPECIAL TRAINING/CERTIFICATION

One or more technicians, with at least one of the technicians having at least two (2) years of field sampling experience, will perform the field sampling. A Senior Technician will be matched to the project based on the field sampling being performed and the sampling-specific experience level of the technician(s) performing the field sampling.

The training and/or certification for the laboratory personnel are presented in the laboratory QAMs (Attachment B-1).

Data Validation will be performed by a trained QA Scientist. The QA Scientist will have experience validating inorganic and organic laboratory data packages according to Federal guidelines outlined in Section 4.2

1.4 DOCUMENTS AND RECORDS

The documentation of sample collection will include the use of bound field logbooks in which all information on sample collection and field instrument calibration will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including Site name (top of each page), sample identification, brief description of the sample, date and time of collection, sampling methodology, field measurements and observations, and sampler's initials (bottom of page with date).

The following documents will be collected and filed: logbooks, field data records, correspondence, chain-of-custody records, analytical reports, data packages, photographs, computer disks, and reports.

2.0 DATA GENERATION AND ACQUISITION

2.1 ANALYTICAL METHODS

The samples should be analyzed using the most current USEPA SW-846 Methods. The appropriate methods for extraction and analysis and required holding times to be met are given in Table B-3. These methods are the most appropriate to achieve all DQOs. Laboratory SOPs for the methods to be used are included in Attachment B-1.

2.2 QUALITY CONTROL

Quality control checks for the analytical laboratory and the field are presented below.

2.2.1 Laboratory Internal QC Checks

Laboratory QC Checks are presented in the laboratory QAM (Attachment B-1). These will be a continuation of the field QC checks presented below. Some of the laboratory internal QC checks include the use of method blanks, surrogates, internal standard compounds, laboratory control samples, laboratory duplicates, interference check samples, and serial dilutions.

2.2.2 Field QC Checks

The specific field QC Checks that will be utilized during this investigation have been included in each individual sampling and analysis plan section. The frequency of collection for the field QC samples has also been included in each individual sampling and analysis plan section.

2.3 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

2.3.1 Laboratory Maintenance

Laboratory preventative maintenance programs and requirements are described in the laboratory QAM (Attachment B-1).

2.3.2 Field Maintenance

The routine daily maintenance procedures conducted in the field have been included for each planning sampling activity and can be found in the SAP.

2.4 INSTRUMENT EQUIPMENT CALIBRATION AND FREQUENCY

2.4.1 Laboratory Calibration

Laboratory calibration and frequency is specified in the USEPA SW-846 Methods for both inorganic and organic parameters and is summarized in the laboratory QAM (Attachment B-1).

2.4.2 Field Calibration

In addition to the laboratory analyses being conducted, field measurements will also be taken. Field personnel will be responsible for making sure the field equipment is properly calibrated prior to use. Any calibrations performed in the field will be recorded directly into the field logbook. If equipment fails calibration or equipment malfunction is noted during calibration or use, the equipment will be tagged and removed from service.

The specific field calibration procedures conducted in the field have been included for each sampling activity and can be found in the SAP if calibration of a field meter/instrument is required.

2.5 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Supplies and consumables are inspected upon receipt to check for damage during shipment to confirm that no potential cross contamination occurred, and to confirm the items ordered were shipped. Spare and replacement parts stored at the Advanced GeoServices office equipment room; on-Site; and/or in the Advanced GeoServices company vehicle(s) to minimize downtime include the following:

- Appropriately sized batteries;
- Locks;
- Extra sample containers and appropriate preservatives;
- Extra sample coolers, packing material, and sample location flags/stakes;
- Additional supply of health and safety equipment, i.e., gloves, hardhats, safety glasses, etc.; and,
- Additional equipment as necessary for the field tasks.

2.6 NON-DIRECT MEASUREMENTS

There will be no non-direct measurements.

2.7 DATA MANAGEMENT

All field data is documented in bound, pre-numbered logbooks. Once a logbook has been filled, the logbook is returned to the office and filed in the project files. Samples collected are documented in the logbook and on the CHOC record for submittal to the laboratory.

All laboratory data is submitted to Advanced GeoServices in both a hard copy report and as an electronic data deliverable (EDD).

A copy of the CHOC is used to hand enter the sample identification into the database. All hand entries are 100% checked and validated by a designated individual that did not enter the CHOC originally. The EDD is used to enter the analytical results into the database. No modifications of the data are made to the EDD. Once the field data and laboratory data have been entered into the database, tables are made for use in the validation of the data. Through the process of data validation, the tables are checked to the laboratory report to confirm that the EDD was accurately reported by the laboratory.

Any qualifiers assigned during the validation are entered by the QA Scientist and 100% checked by a designated individual who did not enter the qualifiers originally.

3.0 ASSESSMENT AND OVERSIGHT

3.1 LABORATORY ASSESSMENTS AND RESPONSE ACTIONS

The purpose of a QA assessment or audit is to provide an objective, independent assessment of a measurement effort. The QA audit determines whether the laboratory's data generating, data gathering, and measurement activities produce reliable and valid results. There are two forms of QA audits: performance evaluation audits and system audits.

3.1.1 Performance Evaluation Audits

The purpose of performance evaluation audits is to quantitatively measure the quality of the data. These audits provide a direct evaluation of the various measurement systems' capabilities to generate quality data.

The laboratory regularly participate in performance evaluation audits as part of their laboratory certification efforts. Performance audits are conducted by introducing control samples in addition to those routinely used. The results for the performance audits are summarized and maintained by the Laboratory QA Supervisor and distributed to the Section Supervisors who must investigate and respond to any out of control results.

Pace Analytical is a NELAP accredited laboratory.

3.1.2 Technical System Audits

A technical system audit is an on-site, qualitative review of various aspects of a total sampling and/or analytical system. The purpose of the technical audit is to assess the overall effectiveness, through an objective evaluation, of a set of interactive systems with respect to strength, deficiencies, and potential areas of concern. Typically, the audit consists of observations and documentation of all aspects of sample analyses. External and internal audits are conducted of the

laboratory throughout each year. Corrective actions for the laboratory are presented in their QAM. The Section Supervisors will provide documentation as to what, if any, corrective actions were initiated concerning external and/or internal audits and are reported to the Laboratory QA Supervisor.

4.0 DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VERIFICATION, AND VALIDATION

Data validation practices will be followed to ensure that raw data are not altered and that an audit trail is developed for those data which required reduction. All field data, such as those generated during field measurements, observations, and field instrument calibrations, will be entered directly into a bound field logbook.

Upon receipt of the final laboratory report, the laboratory data will be qualitatively validated by the QA Scientist.

Analytical data for soil/solid matrices will be reported as micrograms per kilogram (µg/kg) or milligrams per kilogram (mg/kg); for aqueous matrices will be reported as microgram per liter (µg/L) or milligram per liter (mg/L). Sample concentrations detected between the reporting limit and the method detection limit will be reported by the laboratory and flagged as estimated with a "J" qualifier. Data packages associated with the analyses of samples collected during the investigation will be prepared utilizing USEPA Contract Laboratory Program (CLP) similar deliverable formats.

All raw field data will be summarized, reduced, or tabulated for use in the investigation reports. All laboratory analytical data will be summarized and tabulated upon receipt, validated, and qualified (see Section 4.2) and the final data submitted to the project team for use in the investigation reports.

The following documents will be collected and filed: logbooks, field data records, correspondence, CHOC records, analytical reports, data packages, photographs, computer disks/CDs, and reports.

To maintain control in the transfer of data, all copies of raw data from the field notebooks and the data as received from the laboratory, will be entered into a data file and assigned an appropriate document control identification number. The data file will serve as the ultimate archive for all information and data generated during this investigation.

The documentation of sample collection will include the use of bound field logbooks in which all information on sample collection and field instrument calibration will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including the Site name (top of each page), sample identification, brief description of the sample, date and time of collection, sampling methodology, field measurements and observations, and sampler's initials (bottom of each page with date).

4.2 VERIFICATION AND VALIDATION METHODS

All analytical data generated during the investigation will undergo data review. This review will be performed in accordance with the USEPA *National Functional Guidelines for Organic Superfund Methods Data Review, January 2017* and USEPA *National Functional Guidelines for Inorganic Superfund Methods Data Review, January 2017.*

A preliminary review will be performed on all analysis to verify the necessary paperwork (chain-of-custodies, traffic reports, analytical reports, laboratory personnel signatures) and deliverables are present.

A detailed QA review will be performed on the soil borings data and groundwater analyses by the QA Scientist to verify the reliability of the data as it is presented. This review will include a detailed review and interpretation of all data generated by the laboratory. The primary tools which will be used by the experienced QA Scientist will be guidance documents, established (contractual) criteria, and professional judgment. The items that will be examined during the detailed QA review of organic and/or inorganic data will consist of: holding times; sample receipt condition, preservation, and cooler temperature; initial calibration; initial calibration verification; continuing

calibration; CRDL standards; blanks (method, preparation, trip, initial, continuing, and equipment); GC/MS tune summaries; ICP interference check samples; surrogate recoveries; laboratory duplicates; field duplicates; internal standards; MS/MSD pairs; laboratory control samples; serial dilutions; GC/ECD instrument performance checks; and GC/ECD and GC/MS target compound identification; and overall system performance.

Based upon the detailed review of the analytical data a data validation report will be prepared which will state in a technical, yet "user-friendly" fashion the qualitative and quantitative reliability of the analytical data. The report will consist of a general introduction section, followed by qualifying statements that should be taken into consideration for the analytical results to best be utilized. Based upon the QA review, qualifier codes will be placed next to specific sample results on the sample data tables, if necessary. These qualifier codes will serve as an indication of the qualitative and quantitative reliability of the data. Common qualifier codes include:

- "U" – the analyte was not detected at or above the reporting limit;
- "J" – the analyte was positively identified and detected; however, the concentration is an estimated value because the result is less than the reporting limit or quality control criteria were not met;
- "UJ" – the analyte was not detected, the associated reporting limit is an estimated value;
- "R" – data are rejected due to significant exceedance of quality control criteria. The analyte may or may not be present. Additional sampling and analysis are required to determine;

The data tables and data validation reports will be signed and dated by the QA Scientist.

4.3 RECONCILIATION WITH USER REQUIREMENTS

Completeness will be calculated to reconcile the useable validated data to the entire data set. A completeness of 90% or greater of unrejected analytical data is the objective for the project.

Specific reconciliation with the user requirements has been included in their respective sampling sections of the SAP.

TABLES

TABLE B-1
FIELD AND LABORATORY ACCEPTANCE CRITERIA
Exide Frankfort
Frankfort, Indiana

ANALYSIS	Field Criteria				Laboratory Criteria					
	Trip Blank	Equipment Blank	Field Duplicate RPD (Aqueous)	Field Duplicate RPD (Soil)	Laboratory Blank	MS/MSD %R	MS/MSD RPD *	Laboratory Duplicate	Surrogate %R	LCS %R
Gasoline Range Organics (8015D)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	46-134% aqueous 17-153% soil	20%	NA	68-136% aqueous 29-151% soil	65-133% aqueous 68-122% soil
Diesel Range Organics (8015D)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	37-101% aqueous 10-175% soil	20%	NA	35-99% aqueous 10-162% soil	35-99% aqueous 68-122% soil
Volatile Organic Compounds (8260B)	< MDL	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	Vary	20%	NA	Vary	Vary
Semivolatile Organic Compounds (8270C/8270C SIM)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	Vary	20%	NA	Vary	Vary
Inorganics (6010B)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	75-125%	20%	NA	NA	80-120%
Mercury (7471/7470A)	NA	< MDL	<30% for results >5 x RL <±RL for results <5 x RL	<40% for results >5 x RL <±2xRL for results <5 x RL	< MDL	75-125%	20%	NA	NA	80-120%
pH (9045C)	NA	NA	NA	<40%	NA	NA	2%	2%	NA	NA

RL - Reporting Limit
NA - Not applicable
%R - Percent recovery
RPD - Relative percent difference
LCS - Laboratory control sample

The MS/MSD, LCS, and Surrogate percent recoveries are statistically calculated and are subject to change.

The semivolatile and volatile laboratory criteria vary for the indivdual parameters and surrogates recoveries based on in house control limits and method criteria.

TABLE B-2A
Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Volatile Organic Compounds						
Acetone	ug/L	67-64-1	14000	8260C	100	50
Benzene	ug/L	71-43-2	5	8260C	5.0	1.0
Bromodichloromethane	ug/L	75-27-4	80	8260C	5.0	2.5
Bromoform	ug/L	75-25-2	80	8260C	5.0	2.5
Bromomethane	ug/L	74-83-9	7.5	8260C	5.0	3.9
Bromochloromethane	ug/L	74-97-5	83	8260C	5.0	2.5
2-Butanone (MEK)	ug/L	78-93-3	5600	8260C	25	12
Carbon disulfide	ug/L	75-15-0	810	8260C	10	5.0
Carbon tetrachloride	ug/L	56-23-5	5	8260C	5.0	2.5
Chlorobenzene	ug/L	108-90-7	100	8260C	5.0	2.5
Chloroethane (Ethyl Chloride)	ug/L	75-00-3	21000	8260C	5.0	2.5
Chloroform	ug/L	67-66-3	80	8260C	5.0	2.5
Chloromethane	ug/L	74-87-3	190	8260C	5.0	2.5
Cyclohexane	ug/L	110-82-7	13000	8260C	100	50
Dibromochloromethane	ug/L	124-48-1	80	8260C	5.0	2.5
1,2-Dibromoethane (EDB)	ug/L	106-93-4	0.05	8260C	5.0	2.5
1,2-Dibromo-3-chloropropane (DBCP)	ug/L	96-12-8	0.2	8260C	10	5.0
1,2-Dichlorobenzene	ug/L	95-50-1	600	8260C	5.0	2.5
1,3-Dichlorobenzene	ug/L	541-73-1	NA	8260C	5.0	2.5
1,4-Dichlorobenzene	ug/L	106-46-7	75	8260C	5.0	2.5
Dichlorodifluoromethane	ug/L	75-71-8	200	8260C	5.0	5.0
1,1-Dichloroethane	ug/L	75-34-3	28	8260C	5.0	2.5
1,2-Dichloroethane (EDC)	ug/L	107-06-2	5	8260C	5.0	2.5
1,1-Dichloroethene	ug/L	75-35-4	7	8260C	5.0	2.5
cis -1,2-Dichloroethene	ug/L	156-59-2	70	8260C	5.0	2.5
trans -1,2-Dichloroethene	ug/L	156-60-5	100	8260C	5.0	2.5
1,2-Dichloropropane	ug/L	78-87-5	5	8260C	5.0	2.5
cis -1,3-Dichloropropene	ug/L	10061-01-5	4.7	8260C	5.0	2.5
trans -1,3-Dichloropropene	ug/L	10061-02-6	4.7	8260C	5.0	2.5
Ethylbenzene	ug/L	100-41-4	700	8260C	5.0	2.5
2-Hexanone	ug/L	591-78-6	38	8260C	25	12
lsopropylbenzene (Cumene)	ug/L	98-82-8	450	8260C	5.0	2.5
Methylacetate	ug/L	79-20-9	20000	8260C	50	25
Methylcyclohexane	ug/L	108-87-2	NA	8260C	50	25
Methylene Chloride	ug/L	75-09-2	5	8260C	5.0	3.2
4-Methyl-2-pentanone (MIBK)	ug/L	108-10-1	6300	8260C	25	12
Methyl-tert-butyl-Ether (MTBE)	ug/L	1634-04-4	140	8260C	4.0	2.1
Styrene	ug/L	100-42-5	100	8260C	5.0	2.5
1,1,2,2-Tetrachloroethane	ug/L	79-34-5	0.76	8260C	5.0	2.5
Tetrachloroethene (PCE)	ug/L	127-18-4	5	8260C	5.0	1.2
Toluene	ug/L	108-88-3	1000	8260C	5.0	2.5
1,2,3-Trichlorobenzene	ug/L	87-61-6	7	8260C	5.0	2.5
1,2,4-Trichlorobenzene	ug/L	120-82-1	70	8260C	5.0	2.5
1,1,1-Trichloroethane (TCA)	ug/L	71-55-6	200	8260C	5.0	2.5
1,1,2-Trichloroethane	ug/L	79-00-5	5	8260C	5.0	2.5
1,1,2-Trichloro-1,2,2-trifluoroethane	ug/L	76-13-1	55000	8260C	5.0	2.5
Trichloroethene (TCE)	ug/L	79-01-6	5	8260C	5.0	1.9
Trichlorofluoromethane	ug/L	75-69-4	5200	8260C	5.0	2.5
Vinyl Chloride	ug/L	75-01-4	2	8260C	2.0	2.0
Xylenes, Total	ug/L	1330-20-7	190	8260C	10	5.0
Semivolatiles Organic Compounds						
Acenaphthene	ug/L	83-32-9	530	8270C SIM	1.0	0.012
Acenaphthylene	ug/L	208-96-8	NA	8270C SIM	1.0	0.012

TABLE B-2A
Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria
Exide Technologies
Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Acetophenone	ug/L	98-86-2	1900	8270C	10.0	5
Anthracene	ug/L	120-12-7	1800	8270C SIM	0.10	0.021
Atrazine	ug/L	1912-24-9	3	8270C	10.0	5
Benzaldehyde	ug/L	100-52-7	190	8270C	50	25
Benzo[a]anthracene	ug/L	56-55-3	0.12	8270C SIM	0.10	0.023
Benzo[a]pyrene	ug/L	50-32-8	0.2	8270C SIM	0.10	0.013
Benzo[b]fluoranthene	ug/L	205-99-2	0.34	8270C SIM	0.10	0.026
Benzo[g,h,i]perylene	ug/L	191-24-2	NA	8270C SIM	0.10	0.014
Benzo[k]fluoranthene	ug/L	207-08-9	3.4	8270C SIM	0.10	0.018
1,1-Biphenyl	ug/L	92-52-4	0.83	8270C	10.00	5
Bis(2-chloroethoxy) methane	ug/L	111-91-1	59	8270C	10	5.0
Bis(2-chloroethyl) ether	ug/L	111-44-4	0.14	8270C	10	5.0
Bis(2-ethylhexyl) phthalate	ug/L	117-81-7	6	8270C	10	5.0
4-Bromophenyl phenyl ether	ug/L	101-55-03	NA	8270C	10	5.0
Butyl benzyl phthalate	ug/L	85-68-7	160	8270C	10	5.0
Caprolactam	ug/L	105-60-2	9900	8270C	10	5.0
Carbazole	ug/L	86-74-8	NA	8270C	10	5.0
4-Chloroaniline	ug/L	106-47-8	3.7	8270C	10	10
4-Chloro-3-methylphenol	ug/L	59-50-7	1400	8270C	10	7.0
2-Chloronaphthalene	ug/L	91-58-7	750	8270C	10	5.0
2-Chlorophenol	ug/L	95-57-8	91	8270C	10	5.0
4-Chlorophenyl phenyl ether	ug/L	7005-72-3	NA	8270C	10	5.0
Chrysene	ug/L	218-01-9	34	8270C SIM	0.50	0.025
1,4 - Dioxane (p-Dioxane)	ug/L	123-91-1	4.6	8270C SIM	3	0.3
Dibenz[a,h]anthracene	ug/L	53-70-3	0.034	8270C SIM	0.10	0.020
Dibenzofuran	ug/L	132-64-9	7.9	8270C	10	5.0
Di-*n* -butyl phthalate	ug/L	84-74-2	900	8270C	10	5.0
3,3'-Dichlorobenzidine	ug/L	91-94-1	1.3	8270C	20	10
2,4-Dichlorophenol	ug/L	120-83-2	46	8270C	10	5.0
Diethyl phthalate	ug/L	84-66-2	15000	8270C	10	5.0
2,4-Dimethylphenol	ug/L	105-67-9	360	8270C	10	5.0
Dimethylphthalate	ug/L	131-11-3	NA	8270C	10	5.0
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	ug/L	534-52-1	1.5	8270C	50	25
2,4-Dinitrophenol	ug/L	51-28-5	39b	8270C	50	25
2,4-Dinitrotoluene	ug/L	121-14-2	2.4	8270C	10	5.0
2,6-Dinitrotoluene	ug/L	606-20-2	0.49	8270C	10	5.0
Di-*n* -octyl phthalate	ug/L	117-84-0	200	8270C	10	5.0
Fluoranthene	ug/L	206-44-0	800	8270C SIM	1.0	0.022
Fluorene	ug/L	86-73-7	290	8270C SIM	1.0	0.022
Hexachlorobenzene	ug/L	118-74-1	1	8270C	10	5.0
Hexachloro-1,3-butadiene	ug/L	87-68-3	1.4	8270C	10	5.0
Hexachlorocyclopentadiene	ug/L	77-47-4	50	8270C	10	5.0
Hexachloroethane	ug/L	67-72-1	3.3	8270C	10	5.0
lndeno[1,2,3-cd]pyrene	ug/L	193-39-5	0.34	8270C SIM	0.10	0.015
Isophorone	ug/L	78-59-1	780	8270C	10	5.0
2-Methylphenol (o-Cresol)	ug/L	95-48-7	930	8270C	10	5.0
3 & 4-Methylphenol (m & p Cresols)	ug/L	108-39-4, 106-44-5	930	8270C	20	10
2-Methylnaphthalene	ug/L	91-57-6	36	8270C SIM	1.0	0.062
N -Nitroso-di-*n* -propylamine	ug/L	621-64-7	0.11	8270C	10	5.0
N -Nitrosodiphenylamine	ug/L	86-30-6	120	8270C	10	5.0
Naphthalene	ug/L	91-20-3	1.7	8270C SIM	1.0	0.098
2-Nitroaniline	ug/L	88-74-4	190	8270C	50	25
3-Nitroaniline	ug/L	99-09-2	NA	8270C	50	25
4-Nitroaniline	ug/L	100-01-6	38	8270C	50	25

TABLE B-2A

Groundwater reporting limits and minimum detection limits versus 2017 Remediation Closure Guide Criteria

Exide Technologies

Frankfort, Indiana

Analysis	Units	CAS Number	2017 RCG Ground Water Tap Limit	Method	RL	MDL
Nitrobenzene	ug/L	98-95-3	1.4	8270C	10	5.0
2-Nitrophenol	ug/L	88-75-5	NA	8270C	10	5.0
4-Nitrophenol	ug/L	100-02-7	NA	8270C	50	50
2,2'-Oxybis(1-Chloropropane)	ug/L	108-60-1	710	8270C	10	5
Pentachlorophenol	ug/L	87-86-5	1	8270C	50	25
Phenanthrene	ug/L	85-01-8	NA	8270C SIM	1.0	0.03
Phenol	ug/L	108-95-2	5800	8270C	10	5.0
Pyrene	ug/L	129-00-0	120	8270C SIM	1.0	0.015
1,2,4,5-Tetrachlorobenzene	ug/L	95-94-3	1.7	8270C	10.0	5
2,3,4,6-Tetrachlorophenol	ug/L	58-90-2	240	8270C	10.0	5
2,4,5-Trichlorophenol	ug/L	95-95-4	1200	8270C	10	5.0
2,4,6-Trichlorophenol	ug/L	88-06-2	12	8270C	10	5.0
Inorganics						
Arsenic	ug/L	7440-38-2	10	6010B	10	3.18
Barium	ug/L	7440-39-3	2000	6010B	10	0.83
Cadmium	ug/L	7440-43-9	5	6010B	2.0	0.24
Chromium	ug/L	7440-47-3	100	6010B	10	4.439
Lead	ug/L	7439-92-1	15	6010B	10	4.47
Mercury	ug/L	7439-97-6	2	7471A	2.0	0.10
Selenium	ug/L	7782-49-2	50	6010B	10	3.76
Silver	ug/L	7440-22-4	94	6010B	10	8.64
Analysis	**Units**	**CAS Number**	**2009 RISC Closure Level[1]**	**Method**	**RL**	**MDL**
Total Petroleum Hydrocarbons						
Gasoline Range Organics C5-C12	mg/L	NA	14	8015D	0.20	0.10
Diesel Range Organics C8-C28	mg/L	NA	2.5	8015D	0.10	0.038

Notes:

[1]TPH Closure Level - RISC Announcements July 06, 2009

TABLE B-3
SAMPLE CONTAINERS AND PRESERVATIVES
Exide Technologies
Frankfort, Indiana

Sampling Event	Matrix	Analysis	Extraction Method	Analytical Method	Container	Preservative	Holding Time
RFI Soil	Soil	TCL Volatiles	5035 (prep.)	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
		TCL Semivolatiles	3550C (prep.)	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis
		GRO	NA	8015D	Terracore Sampling Kit	Ice	14 days
		pH	NA	9045C	4 oz WMG	Ice	ASAP (24 hours)
		DRO	NA	8015D	4 oz WMG	Ice	14 days
		RCRA 8 Metals	3050B (prep.)	6010B/7471A	4 oz WMG	Ice	180 days (Hg-28 days)
RFI Groundwater	Groundwater	TCL Volatiles	NA	8260C	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
		TCL Semivolatiles	3510C (prep.)	8270C/8270C SIM	100 ml Amber	Ice	7 days to extraction, then 40 days to analysis
		1,4-Dioxane	3510C (prep.)	8270C	1 liter Amber	Ice	7 days to extraction, then 40 days to analysis
		GRO	NA	8015D	3 40mL VOA vials	pH < 2, HCl, Ice	14 days
		DRO	NA	8015D	2 L Amber	Ice	7 days
		Total RCRA 8 Metals	3010A (prep.)	6010B/7470A	500 ml HDPE	HNO3	180 days (Hg-28 days)
		Dissolved RCRA 8 Metals	3010A (prep.)	6010B/7470A	500 ml HDPE	Field Filtered, HNO3	180 days (Hg-28 days)
Well Installation	Soil	TCL Volatiles	5035 (prep.)	8260C	Terracore Sampling Kit	Ice	48 hours to extraction, 14 days to analysis
		TCL Semivolatiles	3550C (prep.)	8270C	4 oz WMG	Ice	14 days to extraction, then 40 days to analysis

NA - Not applicable. Extraction method is included in the analytical procedures.
WMG - Wide mouth glass
Sample containers are based on individual sampling analysis. The laboratory may combine and increase sample container sizes if multiple analysis is requested.

Attachment B-1

Laboratory Quality Assurance Manual
(Provided on CD)

QUALITY ASSURANCE MANUAL

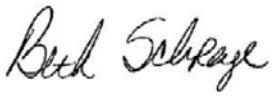
Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services, LLC – Indianapolis
7726 Moller Road
Indianapolis, IN 46268
(317)228-3100

APPROVAL

April 18, 2017
Date

Steve Sayer
General Manager
(317)228-3100



April 18, 2017
Date

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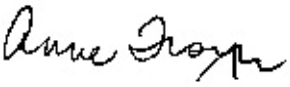

April 18, 2017
Date

Anne Troyer
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1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1. Introduction to Pace

1.1.1. Pace Analytical Services, LLC is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the NELAC Standard, the TNI Standard, and ISO standards and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**

- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**:

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct:**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development

matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environment where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Extractions
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis

1.7.7. The organizational structure for Pace – Indianapolis is listed in Attachment II. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its

accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.4. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The QM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews select laboratory data and final reports;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Reviews and maintains records of proficiency testing results;

- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains calibration of support equipment such as balances and thermometers;
- Maintains the currency of the Quality Manual.

1.8.5. **Technical Director**

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- May review tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.6. **Administrative Business Manager**

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. **Client Services Manager**

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;

- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.8. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody;
- Enters project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes.

1.8.9. Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Trains analysts or oversees training of analysts in laboratory operations and analytical procedures;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.10. Quality Assurance Analyst

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;
- Reviews select laboratory data and final reports;
- Generates and reviews QC data validation packages;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system.

1.8.11. Group Supervisor/Leader

- Trains analysts in laboratory operations and analytical procedures;
- Organizes and schedules analyses with consideration for sample holding times;
- Implements data verification procedures by assigning data verification duties to appropriate personnel;
- Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs;
- Reports non-compliance situations to laboratory management including the SQM/QM.

1.8.12. Laboratory Analyst

- Performs detailed preparation and analysis of samples according to published methods and laboratory procedures;
- Processes and evaluates raw data obtained from preparation and analysis steps;
- Generates final results from raw data, performing primary review against method criteria;
- Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks;
- Reports data in LIMS, authorizing for release pending secondary approval;
- Conducts routine and non-routine maintenance of equipment as required;
- Performs or is capable of performing all duties associated with that of Laboratory Technician.

1.8.13. Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures;
- Performs preparation and analytical steps for basic laboratory methods;
- Works under the direction of a Laboratory Analyst on complex methodologies;
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies;
- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.14. Field Technician

- Prepares and samples according to published methods, PACE Quality Assurance Manual and/or customer directed sampling objectives;
- Capable of the collection of representative environmental or process samples;
- Reviews project documentation for completeness, method compliance and contract fulfillment;
- Train less experienced environmental technicians and provide guidance on sampling and analysis;
- Responsible for project initiation and contact follow-up;
- Develop sampling plans and prepare test plan documents.

1.8.15. **Sample Receiving Personnel**

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments;
- May enter project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes;
- Manages sample storage areas and sample disposal procedures.

1.8.16. **Systems Administrator or Systems Manager**

- Assists with the creation and maintenance of electronic data deliverables (EDDs);
- Coordinates the installation and use of all hardware, software and operating systems;
- Performs troubleshooting on all aforementioned systems;
- Trains new and existing users on systems and system upgrades;
- Maintains all system security passwords;
- Maintains the electronic backups of all computer systems.

1.8.17. **Safety/Chemical Hygiene Officer**

- Maintains the laboratory Chemical Hygiene Plan;
- Plans and implements safety policies and procedures;
- Maintains safety records;
- Organizes and/or performs safety training;
- Performs safety inspections and provides corrective/preventative actions;
- Assists personnel with safety issues.

1.8.18. **Hazardous Waste Coordinator**

- Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
- Maintains complete records of waste disposal including waste manifests and state reports;
- Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.;
- Conducts a weekly inspection of the waste storage areas of the laboratory.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through web-based training systems. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;
- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);

- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;
- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in SOP S-IN-Q-153 *Training Procedures* or its equivalent revision or replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff. Keyless door locks are accessible only to authorized personnel through the use of assigned key fobs. All visitors, including PACE staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out.

1.11.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees.

1.11.3. All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.1.2. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-IN-C-006 *Review of Analytical Requests* or its equivalent revision or replacement.

2.2. Sampling Materials and Support

2.2.1. Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed.

2.2.2. Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the “relinquished” and “received by” sections. All information except signatures is printed.

2.3.4. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents, that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2^{o}C of the required temperature, or within the method-specified range. For samples with a required temperature of 4^{o}C, samples with a temperature ranging from just above freezing to 6^{o}C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
- Samples for **drinking water** analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly

preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).

- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to ± 0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ± 0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤ 6°C limit. Please reference the Support Equipment SOP for more information.

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.4.5. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.5.3. The LIMS automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of 50XXXXXX. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.5.4. Sample labels are printed from the LIMS and affixed to each sample container.

2.5.5. Additional information can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in SOP S-IN-C-001 *Sample Management* or its equivalent revision or replacement and in SOP S-IN-W-002 *Waste Handling and Management* or its equivalent revision or replacement.

2.6.2. **Storage Conditions**

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Storage blanks are stored with volatile samples and are used to measure cross-contamination acquired during storage. Laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.6.2.3. Additional information can be found in SOP S-IN-Q-018 *Monitoring Temperature Controlled Units***.**

2.6.3. **Temperature Monitoring**

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

2.6.3.2. The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ but above freezing unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at $\leq -10^{o}C$ unless state, method or program requirements differ. The temperature of each storage area is checked and documented each day of use. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after a period of time, usually two hours, to verify temperature exceedance. Corrective action is initiated and documented if necessary.
- The SQM/QM and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified and/or documentation is provided on the final report, if necessary.

2.6.3.3. Additional information can be found in SOP S-IN-Q-018 *Monitoring Temperature Controlled Units*.

2.6.4. **Hazardous Materials**

2.6.4.1. Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.6.5. **Foreign/Quarantined Soils**

2.6.5.1. Foreign soils and soils from domestic USDA quarantined areas must be adequately segregated to prevent cross-contamination and enable proper sample disposal. The USDA requires these samples and by-products to be properly identified and handled and to be treated by an approved procedure prior to disposal or as part of disposal.

2.6.5.2. Additional information regarding USDA regulations and sample handling can be found in the laboratory's SOP S-IN-C-007 *USDA Regulated Soil Handling and Disposal* or its equivalent revision or replacement.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-IN-C-003 *Subcontracting Samples* or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in SOP S-IN-C-003 *Subcontracting Samples* or its equivalent revision or replacement.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.8.2. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be moved to ambient temperature storage when the hold time is expired, when the report has been delivered, and/or when allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposal of **hazardous** samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in SOP S-IN-W-002 *Waste Handling and Management* and SOP S-IN-C-001 *Sample Management* or their equivalent revisions or replacements.

3.0. QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or the final report must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Each method blank is evaluated for contamination. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are affected by contamination in a method blank.

3.2.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains all analytes required by a specific method or by the customer or regulatory agency, which may not include the full list of target compounds. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
    - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
    - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;

- For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. The LCS is evaluated against the method default or laboratory-derived acceptance limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any sample containing a compound that was 'out-of-control' in the associated LCS must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the result of the LCS exceeds the upper control limit, indicating high bias, associated samples determined to be non-detect may be reported without qualification.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

3.3.5. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria. When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS when possible or reported with appropriate data qualifiers.

3.3.6. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section.

3.4.3. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method or when limited sample volume or weight prohibits the analysis of an MS/MSD set.

3.4.4. The MS and MSD are evaluated against the method or laboratory derived limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance; however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site-specific information.

3.4.5. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. The sample and duplicate are evaluated against the method or laboratory limits for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

3.5.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are added to samples for most organic analyses to measure the extraction or purge efficiency and to monitor the effect of the sample matrix on compound recovery.

3.6.2. The surrogates are evaluated against the method or laboratory derived acceptance limits. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systemic error. An exception to this would be samples that have surrogate recoveries that exceed the upper control limit but have no reportable hits for target compounds. These samples would be reported and qualified to indicate the implied high bias would not affect the final results.

3.6.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. Additional information can be found in SOP S-IN-Q-004 *Determination of LOD and LOQ* or its equivalent revision or replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. The RL may or may not be based on the lowest calibration standard concentration used in the initial calibration. Results below the lowest calibration level may not be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. Additional information can be found in SOP S-IN-Q-004 *Determination of LOD and LOQ* or its equivalent revision or replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling or sample matrix. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples (LCS). The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-IN-Q-031 *Estimation of Measurement Uncertainty* or its equivalent revision or replacement.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST-approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

3.11.2. The laboratory initiates an investigation whenever PT results are determined to be "Not Acceptable" by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and, when required, this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

3.11.3. Additional information can be found in SOP S-IN-Q-010 *Proficiency Testing Program* or its equivalent revision or replacement.

3.12. Rounding and Significant Figures

3.12.1. In general, the Pace laboratories report data to no more than three significant figures. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a variable that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace - Indianapolis follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

3.12.3.1. Pace - Indianapolis follows the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant figures
Values ≤ 10 – Reported to 2 significant figures

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within appropriately determined retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute

retention time windows must be calculated, unless instrument maintenance fixes the problem. New retention time windows must be established when column geometry is affected by maintenance.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, when the laboratory develops or modifies a method, or when the laboratory brings new instrumentation online, the laboratory validates the method and/or instrument prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method or instrument validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in SOP S-IN-Q-002 *Document Control and Management* or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 *Document Numbering*.

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template then applicable lab staff will sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum; although, a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in SOP S-IN-Q-001 *Preparation of SOPs* or its equivalent revision or replacement.

4.2. Document Change Control

4.2.1. Additional information can be found in SOP S-IN-Q-002 *Document Control and Management* or its equivalent revision or replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After

revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logs so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. Prepared standard or reagent containers include the Pace identification number, the standard or chemical name, and expiration date. The date of preparation, concentration with units, and the preparer's initials can be determined by tracing the standard or reagent ID through the standard log database.

5.1.5. All initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Additional information concerning the procurement of standards and reagent and their traceability can be found in the SOP S-IN-Q-025 *Standard and Reagent Management and Traceability* or its equivalent revision or replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. All applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative.

5.2.3. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

5.2.3.1. The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve;

5.2.3.2. The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve;

5.2.3.3. Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, whichever is appropriate, must be adjusted accordingly;

5.2.3.4. Results from a concentration level between the lowest and highest calibration levels can only be excluded from an initial calibration curve for a documentable and acceptable cause with approval from the SGM/GM, AGM, or the local SQM/QM. An acceptable cause is defined as an obvious sample introduction issue that resulted in no response or very low response, documentation of an incorrectly prepared standard, or a documented response of a single standard that is more than twice the expected value of that standard. The results for all analytes in the calibration standard are to be excluded and the remaining number of concentration levels must meet the minimum number of standards established by the method and standard operating procedure.

5.2.4. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

5.2.5. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.6. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.2.7. Please reference method-specific SOPs for more detailed calibration information.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in SOP S-IN-Q-157 *Support Equipment* or its equivalent revision or replacement.

5.3.2. On each day of use, balances, ovens, refrigerators, incubators, freezers and water baths are checked in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately. This applies mainly to thermometers within temperature-controlled units and balances.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Working calibration weights are ASTM Class 1 or other class weights that have been calibrated against a reference weight set that is re-certified every 5 years, at a minimum, by the manufacturer or other qualified vendor, against a NIST traceable reference. If balances are calibrated by an external agency, verification of their weights must be available. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the local Quality department.

5.3.4. **Thermometers**

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified every 3 years, at a minimum by the manufacturer or other qualified vendor with equipment directly traceable to NIST.

5.3.4.2. Working thermometers and temperature sensors are compared with the reference thermometers annually according to corporate metrology procedures. Alternatively, working thermometers may be replaced with new thermometers annually in lieu of verification or may be verified annually by the manufacturer or other qualified vendor. Each thermometer is individually numbered and assigned a correction factor, when applicable, based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. **pH/Electrometers**

5.3.5.1. The meter is calibrated before use each day, at a minimum, using fresh buffer solutions.

5.3.5.2. The pH electrode is inspected daily and cleaned, filled or replaced as needed.

5.3.6. **Spectrophotometers**

5.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

5.3.7. **Mechanical Volumetric Dispensing Devices**

5.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers dispensing critical volumes, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-IN-Q-157 *Support Equipment* or its equivalent revision or replacement.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

5.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical, field, or product testing data is generated, it is documented appropriately. The resulting logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. Data review checklists, either hardcopy or electronic, are used to document the primary data review process. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in SOP S-IN-Q-016 *Data Review* or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 *Manual Integration* or its equivalent revision or replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer, which must be someone other than the primary analyst. The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations, data quantitation and applicable data qualifiers. The reviewer validates the data entered into the LIMS and documents approval of manual integrations. Data review checklists, either hardcopy or electronic, are used to document the secondary data review process.

6.2.3. Additional information regarding data review procedures can be found in SOP S-IN-Q-016 *Data Review* or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 *Manual Integration* or its equivalent revision or replacement.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. A standard Pace final report consists of the following components:

6.3.2.1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.;

6.3.2.2. Name and address of laboratory and/or subcontractor laboratories, if used;

6.3.2.3. Phone number and name of laboratory contact to where questions can be referred;

6.3.2.4. A unique identification number for the report. The pages of the report are numbered and a total number of pages is indicated;

6.3.2.5. Name and address of customer and name of project;

6.3.2.6. Unique identification of samples analyzed as well as customer sample IDs;

6.3.2.7. Date and time of sample collection, sample receipt and sample analysis;

6.3.2.8. Identification of the test methods used;

6.3.2.9. Qualifiers to the analytical data, if needed or applicable;

6.3.2.10. Identification of whether results are reported on a dry-weight or wet-weight basis;

6.3.2.11. Reporting limits used;

6.3.2.12. Final results or measurements;

6.3.2.13. A signature and title, electronic or otherwise, of person accepting responsibility for the content of the report;

6.3.2.14. Date report was issued;

6.3.2.15. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory;

6.3.2.16. A statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

6.3.3. Any changes made to a final report shall be designated as “Revised” or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.4. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.5. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

6.3.6. Additional information regarding final reports and data deliverables can be found in SOP S-IN-Q-046 *Final Report and Data Deliverable Contents* or its equivalent revision or replacement.

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. TNI-related records will be made readily available to accrediting authorities. Access to archived data is controlled by the Quality Department.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports, raw analytical data, logs or logbooks, and electronic files.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing, assigning and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in SOP S-IN-Q-011 *Internal and External Audits* or its equivalent revision or replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections, at a minimum:

- Raw Data Review audits- conducted according to a schedule per local SQM/QM. A certain number of these data review audits may be conducted per quarter to accomplish this yearly schedule;
- Quality System audits- considered the traditional internal audit function and includes analyst interviews to help determine whether practice matches method requirements and SOP language;
- Final Report reviews;
- Corrective Action Effectiveness Follow-up

7.1.2.2. Internal systems audits are conducted annually at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and identification of final reports that were re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.3. **Internal Audit Reports and Corrective Action Plans**

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. The Quality Department auditor writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

7.1.3.3. Additional information can be found in SOP S-IN-Q-011 *Internal and External Audits* or its equivalent revision or replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in SOP S-ALL-Q-015 *Review of Laboratory Management System* or its equivalent revision or replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, staffing, and safety/waste activities.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.0. CORRECTIVE ACTION

Additional information can be found in SOP S-IN-Q-012 *Corrective and Preventive Actions* or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using Pace's LabTrack system or other system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The following items are examples of sources of laboratory deviations or non-conformances that may warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- Proficiency Testing Sample Results
- Internal and External Audits
- Data or Records Review
- Client Complaints
- Client Inquiries
- Holding Time violations

8.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency or it may be a more formal documentation. This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

8.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and any other pertinent information. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.4. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack.

8.1.5. Based on the determined root cause(s), the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented

laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any ramifications to data quality if impacted.

8.2. Corrective Action Completion

8.2.1. Internal Laboratory Non-Conformance Trends

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting/Data Entry error

8.2.2. PE/PT Sample Results

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.2.2. Additional information, such as requirements regarding time frames for reporting failures to states, makeup PTs, and notifications of investigations, can be found in SOP S-IN-Q-010 *Proficiency Testing Program* or its equivalent revision or replacement.

8.2.3. Internal and External Audits

8.2.3.1. The SQM/QM or designee is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM or designee is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. **Data Review**

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews, errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. **Client Complaints**

8.2.5.1. Project Managers are responsible for issuing corrective action requests, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

8.2.6. **Client Inquiries**

8.2.6.1. When an error on the customer's final report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

8.2.7. **Holding Time Violations**

8.2.7.1. In the event that a holding time has been missed due to laboratory error, the analyst or supervisor must complete formal corrective action. The Project Manager and the SQM/QM must be made aware of all holding time violations due to laboratory error.

8.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- 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 that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.

American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- 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.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- 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 are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- 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.

Batch	TNI- Environmental samples that 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 to 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.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- 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).
Blank	TNI and DoD- 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 (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
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's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that 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. 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 or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- 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 Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- 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 type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Confirmation	TNI- 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. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
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.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
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.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	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	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.

Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.

In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI and DoD- 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.

International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element 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	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), 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 and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) 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.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result 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.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
May	EPA – The word "may" is used to provide guidance on aspects of the method that are useful but not essential.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- 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.
Method Blank	TNI- 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.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance 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 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.

Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- 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.
Must	EPA – The word "must" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.

Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
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.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- 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.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- 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.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.

Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
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.
Quality Control (QC)	TNI- 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 for 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.
Quality Control Sample (QCS)	TNI- 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.

Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	TNI and DoD- 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 and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **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 sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater 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 potentially 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.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.

Raw Data	TNI- 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.
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.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".

Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
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	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	EPA – The word "shall" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
Should	EPA – The word "should" is used to provide guidance on aspects of the method that are useful but not essential.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.

Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a 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.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.

Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
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.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- 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 conditions 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.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where $k > 1$).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. 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.

Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

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10.4. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.

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10.11. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

10.12. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

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10.14. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

10.15. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.

10.16. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.

10.17. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

10.18. ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories- most current version.

10.19. Department of Defense Quality Systems Manual (QSM), most current version.

10.20. TNI (The NELAC Institute) Standard- 2003 and 2009.

10.21. UCMR Laboratory Approval Requirements and Information Document, most current version.

10.22. US EPA Drinking Water Manual, most current version.

11.0. REVISIONS

The Pace Corporate Environmental Quality Office files an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to previous versions of the Quality Assurance Manual. This document is available upon request. All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 19.0	General: made administrative edits that do not affect the policies or procedures within the document (including revising company name to Pace Analytical Services, LLC). Cover page: removed corporate approval signature lines and revised document control format. Table of Contents: added Attachment VII – Pace COC Old Section 3: moved to other sections of the QAM as applicable and deleted entire section (All section references below reflect the new section numbers). Section 1.1.2: replaced with section 3.1.1. Sections 1.3, 1.4, 1.11: removed extraneous language. Sections 1.5: added language from old section 1.6. Section 1.6: revised anonymous reporting information. Section 1.8: removed job descriptions for non-applicable personnel. Section 1.8.4: added tasks to QM job description. Section 1.8.8: added tasks to PM job description. Section 1.11.1: added keyless entry using key fobs detail. Section 2: rearranged existing sections. Section 2.4: reworded to match existing Sample Acceptance policy document. Section 2.6.3.2: added some detail regarding temperature monitoring corrective action. Section 2.6.5.1: added by-products of USDA soils. Section 3.2.2: added basic evaluation criteria. Section 3.4.3: added MS and Dup as optional alternative to MS/MSD. Section 3.5.2: added basic evaluation criteria. Section 3.9.1: added that RL may be based on calibration standard. Section 3.14: added new instrumentation as requiring validation. Section 4: in general, for each QC type, removed language regarding frequency and corrective actions and referenced lab-specific SOPs. Section 5: in general, removed extraneous language and Management of Change section. Section 5.1, 5.2: reorganized into Primary and Secondary Review sections and removed extraneous language. Section 5.3.2: specified types of support equipment to be monitored daily. Section 5.3.3.1: specified “working” weights. Section 5.3.4.2: added temperature sensors and added alternatives to annual in-house verification. Section 5.3.5: added pH electrode inspection/maintenance. Section 6: removed extraneous language including Quarterly Report section. Section 8.2.3.1: added “or designee”. Section 9 (glossary): revised and added definitions based on 2016 TNI Standard. Added “may, must, shall and should” based on SW-846 definition. Section 10: Added EPA DW Manual and revised references as applicable. Attachment III: updated corporate organizational chart. Old Attachment IV: removed floor plan attachment. Old Attachment VII: removed COC (available in SOPs). Indy added back in.	22Mar2017

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

where:
n = number of data points
X_i = individual data point

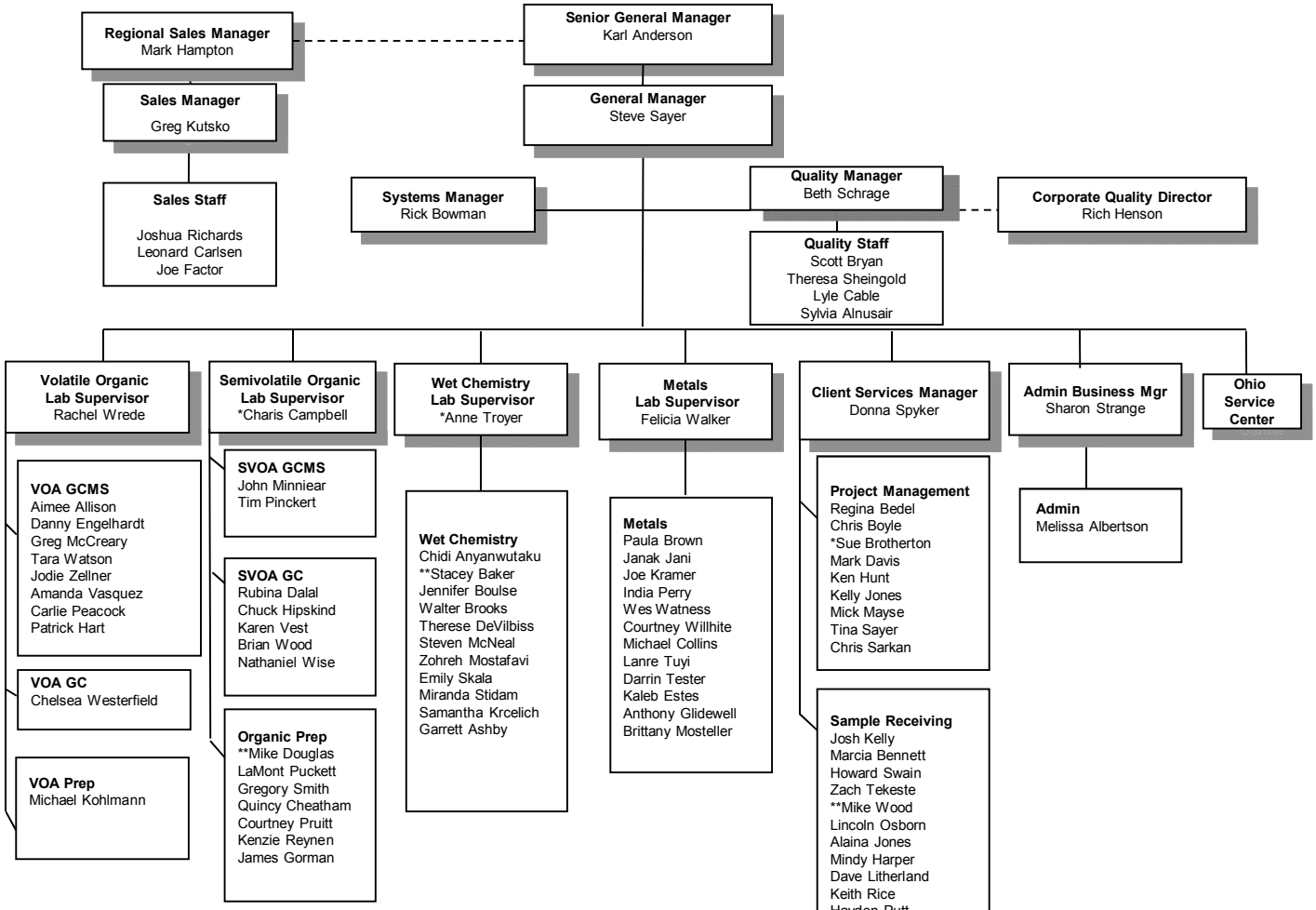
RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

ATTACHMENT II- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)

PACE ANALYTICAL SERVICES - INDIANAPOLIS

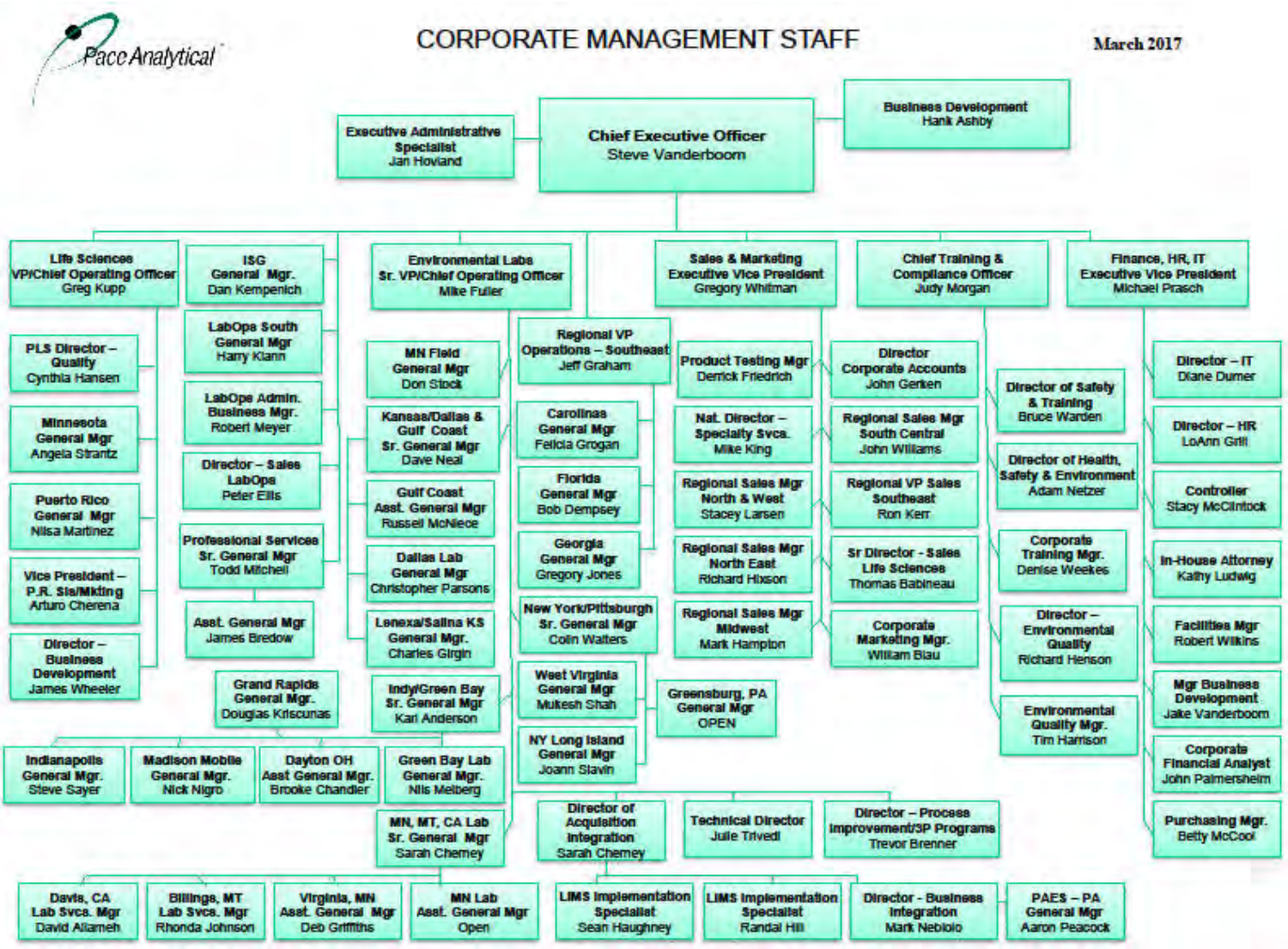


*TNI TECHNICAL DIRECTOR

**DEPT LEAD

Last Revised 3/22/2017

ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)

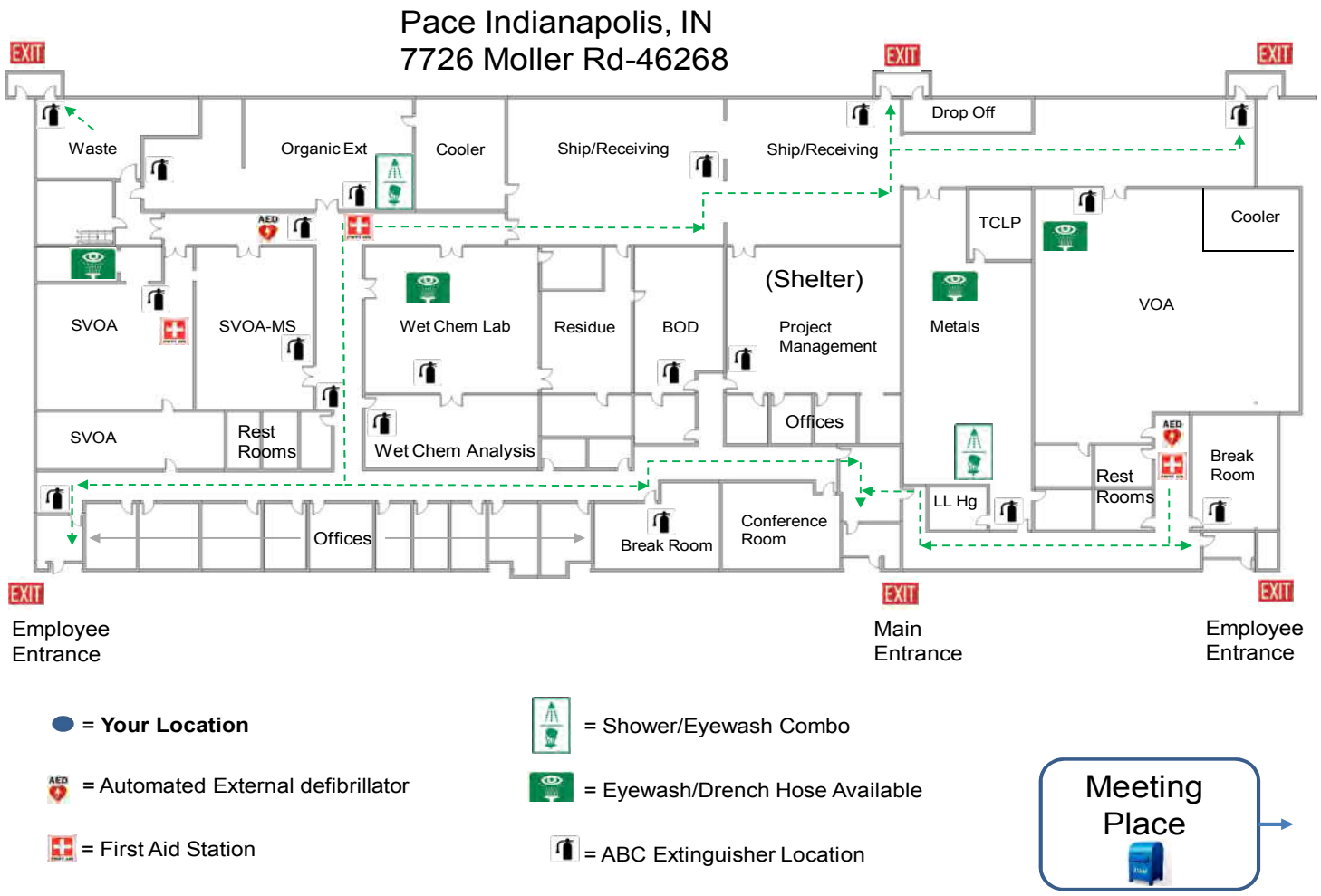


ATTACHMENT IV- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

Pace Indianapolis Equipment/Instrumentation List

INSTRUMENT	MANUFACTURER	MODEL NUMBER	DETECTOR	AUTOSAMPLER	SERVICE ANALYSIS	YEAR
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6890	MS 5973	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2004
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624 VOC	2010
GC/MS	Agilent	6890	MS 5973	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	7890	MS 5975C	Archon	8260	2008
GC/MS	Agilent	6890	MS 5975	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	5975	Centurion	8260/624 VOC	2008
GC/MS	Hewlett-Packard	6890	MS 5973	7683	8270 PAH SIM	2000
GC/MS (2)	Agilent	7890	MS 5975	7683	8270/625 BNA	2008
GC/MS (2)	Agilent	6890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (3)	Agilent	6890	MS 5973	7683	8270/625 BNA	2008
GC/MS	Agilent	7890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (2)	Hewlett-Packard	5890	MS 5971	7673	Solvent Screen	2007
Gas Chromatograph	Agilent	6890	FID	7683	8015 Alcohols	2006
Gas Chromatograph	Hewlett-Packard	6890	FID	6890	8015 Glycols	2008
Gas Chromatograph	Agilent	7890A	FID	7693	8015 DRO/ERO	2009
Gas Chromatograph	Agilent	7890A	Dual ECD	7693	8082/608 PCBs/8011 EDB/DBCP	2009/2013
Gas Chromatograph	Hewlett-Packard	5890	FID	6890	Benzene	2006
Gas Chromatograph	Hewlett-Packard	5890	FID	8100	8015 GRO	2011
Gas Chromatograph	Hewlett-Packard	5890	FID	EST LGX50	RSK175 Dissolved gases	2006
Gas Chromatograph	Agilent	6890N	FID	8100	8015 GRO	2008
Gas Chromatograph	Agilent	6890	Dual NPD	7683	Pesticides	2008
Gas Chromatograph (2)	Agilent	6890	Dual ECD	7683	PCBs	2008
Gas Chromatograph	Hewlett-Packard	6890	Dual ECD	7683	Herbicides	2008
Gas Chromatograph	Agilent	7890	Dual ECD	7693	Pesticides	2010
Microwave Extractors (2)	CEM	230/60	n/a	n/a	soil extraction	2008/2011
Spe-Dex	Horizon	4790	n/a	n/a	1664A Oil & Grease	2008
Trace ICP (3)	Thermo Scientific	ICAP 6500	n/a	n/a	6010/200.7 Metals	2008/2011
ICP/MS (2)	Agilent	7700	n/a	n/a	6010/200.7 Metals	2012/2014
Mercury Analyzer	CETAC	M-6100	n/a	n/a	7470/7471/245 Mercury	2012/2010
Mercury Analyzer	Teledyne Leeman	M-7600	n/a	n/a	7470/7471/245 Mercury	2016
Low-Level Mercury Analyzer	CETAC	M-8000	n/a	n/a	Low-Level Mercury	2015
Auto Analyzer (2)	Lachat	Quick Chem	n/a	n/a	NO3,Cl,Phenol, NH3,TKN	2010/2012
Titrosampler	Metrohm	855	n/a	n/a	Alkalinity, Acidity	2014
Automated Flash Point	Tanaka	APM-8	n/a	n/a	flash point	2010
Spectrophotometer	Spec 20	Labtronics	n/a	n/a	Sulfide	2002
Spectrophotometer	Hach	DR5000	n/a	n/a	Sulfate,Cr6+,Fe2+, PO4	2007
Spectrophotometer	Thermo	AquaMatePlus	n/a	n/a	Surfactants, COD	2005
pH/ISE Meter (2)	Accumet	AR25/XL25	n/a	n/a	pH, Fluoride, Redox	2003/2010
pH/ISE Meter	Thermo Orion Star	A214	n/a	n/a	pH, Fluoride, Redox	2013
Dissolved Oxygen/pH Meter	Hach	HQ440d	n/a	n/a	BOD, cBOD	2014
BOD Analyzer	Thermo	AutoEz	n/a	n/a	BOD, cBOD	2013
TOC Analyzer	Shimadzu	TOC-Vwp	n/a	n/a	TOC, DOC	2008
TOC Analyzer	Teledyne	Phoenix 8000	n/a	n/a	TOC, DOC	2005
Discrete Analyzer	Smart Chem	200	n/a	n/a	Cyanide, Phosphorus	2006
Ion Chromatogram	Dionex	IC3000	n/a	n/a	Cl-, F-, SO4-, Br-, NO3/NO2	2008

ATTACHMENT V- LABORATORY FLOOR PLAN (CURRENT AS OF ISSUE DATE)



ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)

Pace Analytical – Indianapolis Certifications

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Illinois	Hazardous Waste	IL-EPA	003971	10/12/2017
Illinois	Non-Potable Water	IL-EPA	003971	10/12/2017
Indiana	Drinking Water	ISDH	C-49-06	05/06/2018
Kansas (TNI)	Hazardous Waste	KDHE	E-10177	04/30/2017
Kansas (TNI)	Non-Potable Water	KDHE	E-10177	04/30/2017
Kentucky	UST	KDEP	80226	04/30/2017
Kentucky	Wastewater	KDEP	98019	12/31/2017
Ohio VAP	Hazardous Waste	OH-EPA	CL0065	01/28/2018
Ohio VAP	Non-Potable Water	OH-EPA	CL0065	01/28/2018
Oklahoma	Non-Potable Water	OK DEQ	2016-075	08/31/2017
Oklahoma	Solids	OK DEQ	2016-075	08/31/2017
Texas	Non-Potable Water	TX CEQ	T104704355-16-10	01/31/2018
Texas	Solid Chemical Mat.	TX CEQ	T104704355-16-10	01/31/2018
West Virginia	Hazardous Waste	WV-DEP	330	10/31/2017
West Virginia	Non-Potable Water	WV-DEP	330	10/31/2017
Wisconsin	Non-Potable Water	WI DNR	999788130	08/31/2017
Wisconsin	Waste, Soil, Tissue	WI DNR	999788130	08/31/2017
USDA	Foreign Soil	USDA	P330-16-00257	08/19/2019

ATTACHMENT VII- PACE CHAIN-OF-CUSTODY (CURRENT AS OF ISSUE DATE)

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

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Page: of

Section A Required Client Information:
Company:
Address:
Email To:
Phone: Fax:
Requested Due Date/TAT:

Section B Required Project Information:
Report To:
Copy To:
Purchase Order No.:
Project Name:
Project Number:

Section C Invoice Information:
Attention:
Company Name:
Address:
Pace Quote Reference:
Pace Project Manager:
Pace Profile #:

REGULATORY AGENCY
☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER
Site Location STATE:

Section D Required Client Information

SAMPLE ID (A-Z, 0-9 / ,) Sample IDs MUST BE UNIQUE

Matrix Codes (MATRIX / CODE): Drinking Water DW; Water WT; Waste Water WW; Product P; Soil/Solid SL; Oil OL; Wipe WP; Air AR; Tissue TS; Other OT

ITEM #	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test Y/N	Analysis Test	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS	Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY):

ATTACHMENT VIII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	$\leq 6^oC$	14 Days
Alkylated PAHs		Water	1L Amber Glass	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	$\leq 10^oC$	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; $\leq 6^oC$; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)
Biomarkers		Solid	$\leq 10^oC$	1 Year/40 Days	$\leq 10^oC$
BOD/cBOD	SM5210B	Water	Plastic/Glass	$\leq 6^oC$	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	$\leq 6^oC$	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	28 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; $\leq 6^oC$	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; $\leq 6^oC$	7/40 Days
Carbamates	8318	Solid	Glass	$\leq 6^oC$	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6°C, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	≤ 6°C; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10°C	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	$\leq 6^oC$; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	$\leq 6^oC$; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	$\leq 6^oC$	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	$\leq 6^oC$; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	$\leq 6^oC$ in MeOH	21 Days
Glyphosate	547	Water	Glass	$\leq 6^oC$; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; $\leq 6^oC$	14/7 Days if extracts stored $\leq 6^oC$ or 14/14 Days if extracts stored at $\leq -10^oC$

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C	Water	Plastic/Glass	$\leq 6^oC$	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid	Glass	$\leq 6^oC$	30 Days from collection to extraction and 7 days from extraction to analysis
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Lipids	Pace Lipids	Tissue	Plastic/Glass	$\leq$ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	$\leq$ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	$\leq$ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	20mL vials	HCl; or trisodium phosphate or benzalkonium chloride and $\leq$ 6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	$\leq$ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	$\leq$ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6°C	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6°C	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6°C	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; $\leq 6^oC$	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	$\leq 6^oC$	14/30 Days
Perchlorate	331	Water	Plastic/Glass	$\geq$0-6^oC, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and $\leq 6^oC$	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	$\leq -10^oC$	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	$\leq 6^oC$	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	$\leq -10^oC$	1 Year if frozen/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	≤ 6°C	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	$\leq 6^oC$	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; $\leq 6^oC$	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	$\leq 6^oC$	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; $\leq 6^oC$	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	$\leq 6^oC$	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	$\leq 6^oC$	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, $\leq 6^oC$	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	$\leq 6^oC$	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	$Na_2CO_3/NaHCO_3$/ $(NH_4)_2SO_4$; pH>8	14 Days
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6°C	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	≤ 6°C	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	≤ 6°C with benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	28 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	TO-15	Air	Summa Canister or Tedlar Bag	None	28 Days
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	$\leq 6^oC$	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; $\leq 6^oC$; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil	ASTM D3328 (prep); ASTM D5739	Product	10mL glass vials	$\leq 6^oC$	N/A

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4^oC, **and** one vial methanol preserved at $\leq 6^oC$ **and** one container of unpreserved sample stored at $\leq 6^oC$.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

APPENDIX C

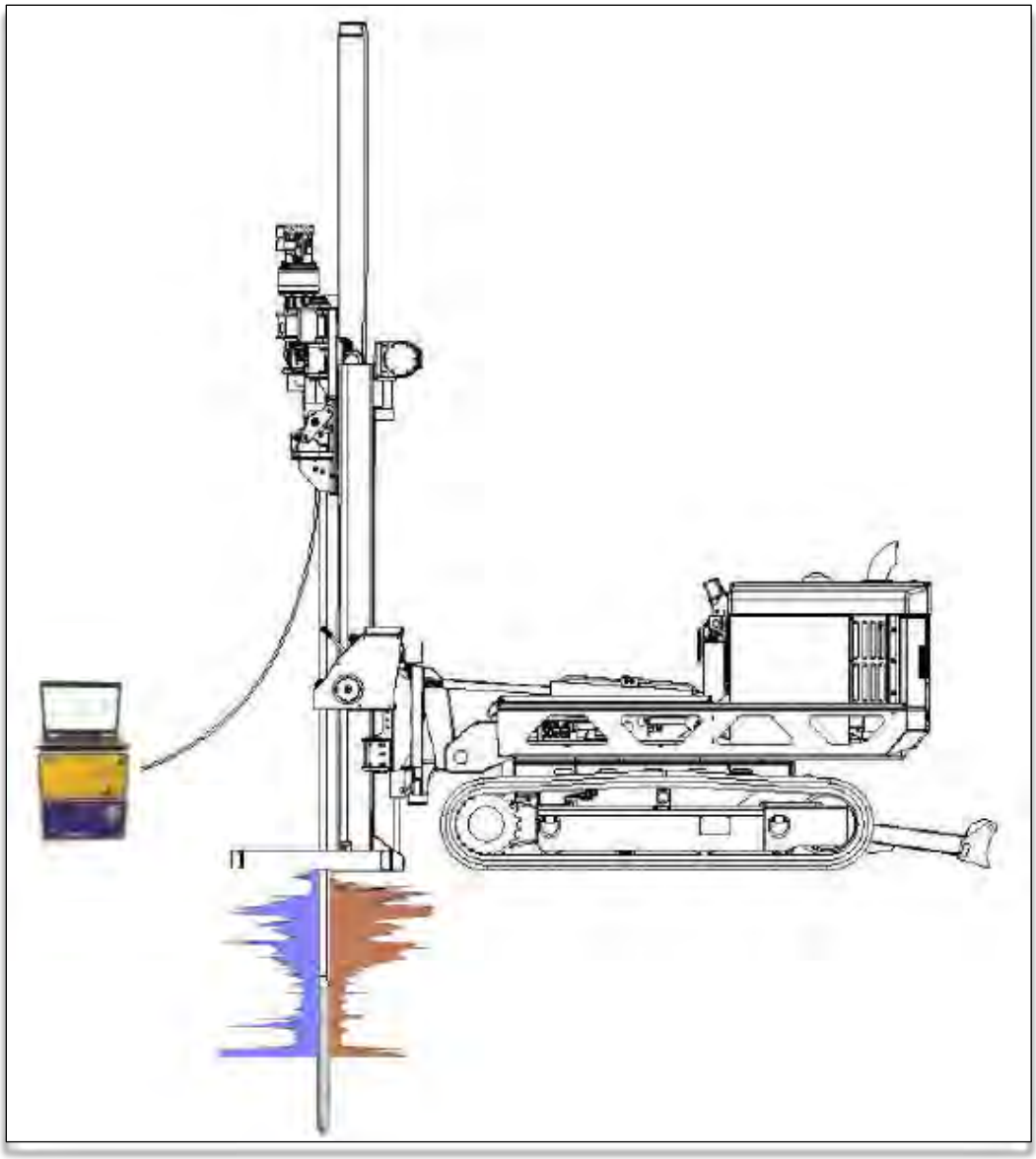
HPT/MIP SOPs

Geoprobe® Hydraulic Profiling Tool (HPT) System

Standard Operating Procedure

Technical Bulletin No. MK3137

Prepared: January 2015



1.0 Objective

This document serves as the standard operating procedure for the Geoprobe® Hydraulic Profiling Tool (HPT) system. In this procedure, the HPT system is used to measure the pressure response of soil to injected water for identifying potential flow paths and to assist with characterization of soil type.

2.0 Background

2.1 Definitions

Geoprobe®*: A brand of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, electrical conductivity and contaminant logging, grouting, and materials injection.

**Geoprobe® and Geoprobe Systems® are registered trademarks of Kejr, Inc., Salina, Kansas.*

Hydraulic Profiling Tool (HPT) System: A system manufactured by Geoprobe Systems® to evaluate the hydraulic behavior of subsurface soil. The tool is advanced through the subsurface at a constant rate while water is injected through a screen on the side of the probe. An in-line pressure sensor measures the pressure response of the soil to water injection. The pressure response identifies the relative ability of a soil to transmit water. Both pressure and flow rate are logged versus depth.

2.2 Introduction

The HPT system has been developed by Geoprobe Systems® for the geohydrologic characterization of soils. The HPT probe and logging system is able to quickly provide logs that are easily interpreted. HPT logs are used to indicate hydraulic conductivity, EC, hydrostatic profile, and areas of EC/permeability anomalies.

The HPT system is designed to evaluate the hydraulic behavior of unconsolidated materials. As the probe is pushed or hammered at 2cm/s, clean water is pumped through a screen on the side of the HPT probe at a low flow rate, usually less than 300mL/min. Injection pressure, which is monitored and plotted with depth, is an indication of the hydraulic properties of the soil. That is, a low pressure response would indicate a relatively large grain size, and the ability to easily transmit water. Conversely, a high HPT pressure response would indicate a relatively small grain size and the lack of ability to transmit water.

An electrical conductivity measurement array is built into the HPT probe. This allows the user to collect soil electrical conductivity (EC) data for lithologic interpretation. In general, the higher the electrical conductivity value, the smaller the grain size, and vice versa. However, other factors can affect EC, such as mineralogy and pore water chemistry (brines, extreme pH, contaminants). In contrast, HPT pressure response is independent of these chemical and mineralogical factors.

There are four primary components of the HPT system: the probe assembly, trunkline, HPT Flow Controller (K6300 Series), and Field Instrument (FI6000 series). These primary components are shown in Figure 2.1.

The probe assembly consists of the HPT probe and connection section. This assembly houses the downhole HPT pressure transducer, water and electrical connections, and the probe body with the injection screen and electrical conductivity array.

Injecting water at a constant rate is integral to system operation. The HPT Flow Module houses the pump and associated hand crank mechanism used for adjusting the output flow of the HPT pump. The flow module also contains the HPT flow measurement and injection line pressure transducers. HPT flow can be adjusted from approximately 50 to 500ml/min. The HPT pump is a positive displacement pumping device with minimal decrease in flow over the HPT operating pressure range. The flow module is equipped with an internal bypass that is factory set to open and return flow to the supply reservoir at a pressure of 120psi. When the soil resistance to water injection becomes sufficiently great, the HPT Flow Module bypass will open, returning some or all of the pumped flow to the supply reservoir. The flow meter only measures flow leaving the module to the HPT probe. The HPT Flow Module is connected to the Field Instrument via a data cable.

Water and power are transmitted from the controller to the probe assembly via the HPT trunkline. The probe rods must be pre-strung with the trunkline before advancing the probe.

Data collection occurs in real time by connecting the controller to the field instrument. The field instrument collects, stores and displays transducer pressure, flow rate and electrical conductivity, line pressure, probe rate, and diagnostic parameters, with depth via the field laptop.

Since the HPT pressure response is analogous to the soil's ability to transmit water (and therefore the to the soil's dominant grain size), the HPT system can be used to identify potential contaminant migration pathways. Similarly, it can help identify zones for remedial material injection or provide qualitative guidance on how difficult injection may be in different zones of the formation.

The HPT system may be used to direct other investigation methods, such as soil and groundwater sampling and slug testing. HPT pressure response and EC data can help target zones of geologic and hydraulic interest, minimizing the number of soil and groundwater samples required to adequately develop a site conceptual model. When hydraulic conductivity values are required, the

HPT system can also help the user identify zones to slug test, as well as the length of the screen required to adequately test the zone.

The HPT system also can be used to collect static water pressure data at discrete intervals during the logging process. These static pressure data can be used to calculate static water levels or to create a hydrostatic profile for the log.

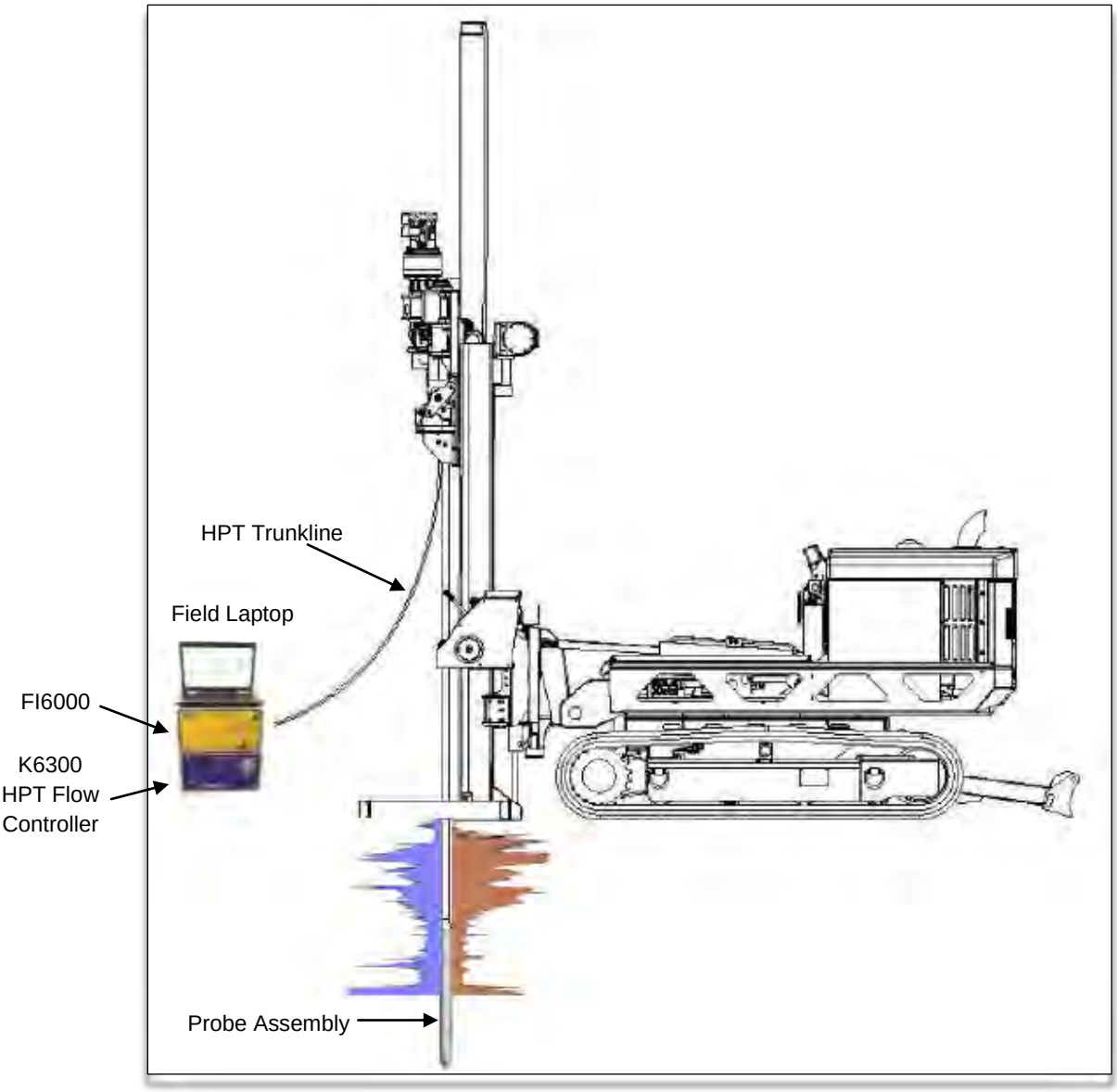


Figure 2.1: HPT Components

3.0 Tools and Equipment

The following equipment is required to perform and record an HPT log using a Geoprobe® 66- or 78-Series Direct Push Machine. Refer to Appendix I for identification of the specified parts.

Basic HPT System Components	Quantity	Material Number
Field Instrument, 120V (Model FI6000)	-1-	213940
Field Instrument, 220V (Model FI6003)	*	213941
HPT Acquisition Software	-1-	214128
HPT Flow Module, 120V (Model K6300)	-1-	214091
HPT Flow Module, 220V (Model K6303)	*	214093
HPT Probe, 1.75 inch	-1-	215667
MIP/HPT Connection Tube	-1-	206304
MIP/HPT Adapter 1.5 Pin x LB Box	-1-	203794
MIP/HPT Adapter 1.75ML Pin x LB Box	**	220966
HPT Probe, 2.25 inch	**	214097
2.25 Connection Tube	**	219455
2.25 Inch Water Seal Drive Head	**	212089
2.75 Inch Water Seal Drive Head	**	209796
HPT Reference Tube 1.75 in HPT Probe	-1-	212689
HPT Reference Tube 2.25 in HPT Probe	**	211762
HPT Trunkline 150 ft.	-1-	214095
HPT Trunkline 200 ft.	(optional)	214096
HPT Service Kit	-1-	205599
HPT Test Load	-1-	206552
EC Probe Test Jig	-1-	214237
EC Test Load	-1-	*208075*
EC Bypass Cable	-1-	204025
Stringpot, 100-inch	-1-	214227
Stringpot Cordset, 65-feet (19.8 m)	-1-	202884

*Use in place of 120V components if desired.

**Use in place of 1.75 inch probe and components if desired.

4.0 HPT Assembly

Refer to Appendix I

Threading the Rods

- Protect the end of the trunkline to be threaded through the rods with electrical tape or shrink tubing.
- Probe rods must alternate directions prior to threading the trunkline.
- The end of the HPT trunkline with chrome connectors is the downhole or probe end.
- The probe end of the trunkline will always enter the male end and exit the female end of the probe rods.
- The instrument end (no chrome connectors) will always enter the female end and exit the male end of the probe rods.
- After the trunkline is through the probe rods make sure the downhole end is threaded through the male end of the drive head and connection tube prior to connecting to the probe.
- The trunkline is now ready to connect to the instrument and HPT pressure sensor and probe.

5.0 Field Operation

5.1 Instrument Setup

1. Connect the HPT Controller (K6300), Field Instrument (FI6000) and laptop (Fig. 5.1) to an appropriate power source.

2. Connect the FI6000 to the K6300 using the 62-pin serial cable inserted into the acquisition port of each instrument.

3. Secure the EC wires into the Green terminal block connector and insert into the FI6000. The wires match to the EC dipoles in the following top down order when the probe tip is on the ground – white, black, yellow and blue (Fig 5.2).

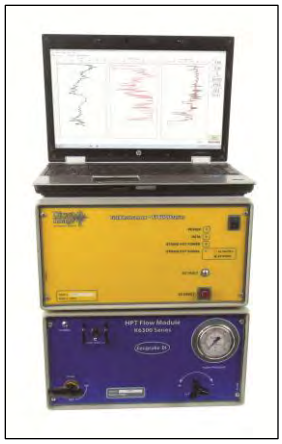


Figure 5.1: HPT Instrument Setup

4. Secure the HPT sensor wires to the appropriate inputs on the green terminal block connector and connect to the rear of the K6300. The top down order of the wires which is listed on the back of the instrument is: brown, orange, red and reserved (open).

5. Insert the nylon water line tubing from the trunkline into the water output connector on the back of the K6300.

6. Connect the HPT water supply hose into the input port on the rear of the K6300 and insert the filtered end of the supply line into a water supply tank. The bypass line connects to the bypass port and will follow the supply line back to the supply tank.

7. Connect the USB cable between the USB interface port on the rear of the FI6000 to USB input on the field laptop computer.

Figure 5.2: EC Wire Connections

8. A stringpot is required to measure depth. Bolt the stringpot onto the machine and the stringpot onto the bracket. Connect the plastic connector end of the stringpot cable to the "Stringpot" connector on the back of the Field Instrument and the metal connector to the stringpot. Pull the stringpot cable and attach to the stringpot piston weight which should be mounted to the probe machine foot and pull the keeper pin so the weight is free to move.

5.2 Starting the Software

1. Make sure the FI6000 and K6300 are connected together with the 62 pin cable, powered on and connected to the computer by the USB cable for the software to load properly.

2. Start the DI Acquisition Software which should open in HPT mode.

3. Select "Start New Log". The software will request log information and have you browse for a storage location and create and save a file name for the log (Fig. 5.3).

4. Select "Next". If the software has been run before it will show a list of previous settings including Probe Type, EC Configuration, Stringpot length, rod length and HPT Transducer. If any of these have changed or you are unsure select "No" but if they are all the same select "yes". If you select "No" the software will have you select the proper settings after the EC Load Test, if you selected "Yes" the selection of these settings will be bypassed.

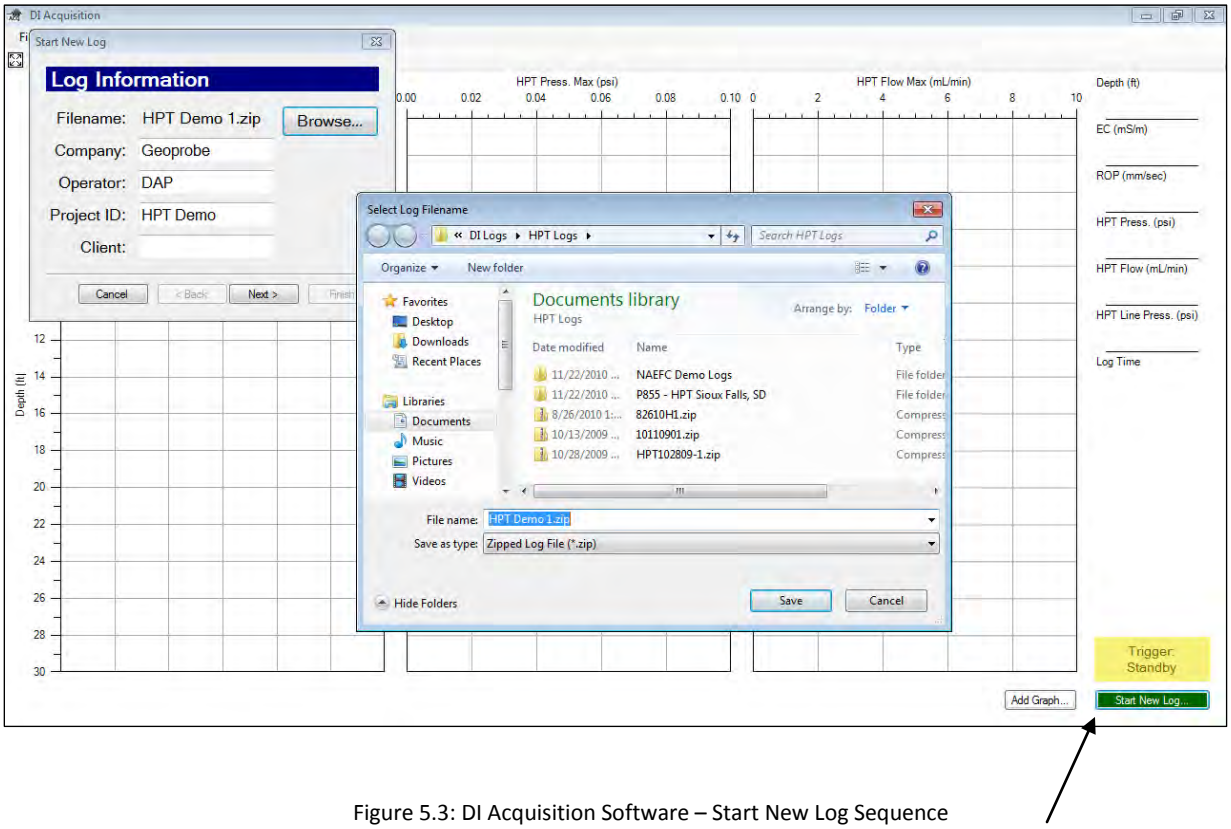


Figure 5.3: DI Acquisition Software – Start New Log Sequence

5.3 QA Testing the EC and HPT Systems

Both the EC and HPT components must be tested before and after each log. This is required to ensure that the equipment is working properly and capable of generating good data before and after the log.

A. Electrical Conductivity Load Test

1. Secure the EC 3 position test load connector (208075) to the test input jack on the back of the Field Instrument.

2. Secure the EC Probe Test Jig into the input on the EC 3 position test load.

3. Clean and dry the EC dipoles as well as several inches of the probe body above the pins.

4. Place the EC Test Jig (214237) so that the four springs on the test jig touch the four dipoles of the Wenner EC array (Fig. 5.4). Make sure the trunkline and test jig wires go in the same direction. The other spring on the test jig will ground the probe body above the Wenner array. Make sure the springs are pulled out far enough to make a solid contact on the dipoles.

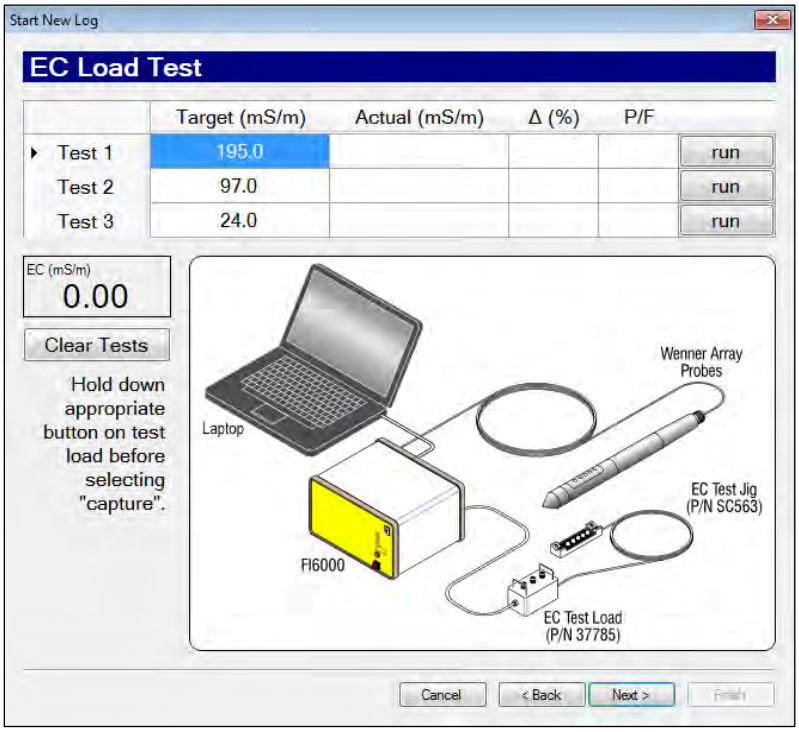


Figure 5.4: EC Load Test Screen

5. When you get to the EC Load Test Screen and the EC test load and test jig are in place on the probe press down on the test 1 button on the test load and select “run” of Test 1 (Fig. 5.4). After 5 seconds the actual value will acquire and will pass if within 10% of the target value. Continue on with Test 2 and 3.

6. If any of the EC load tests fail do not pass within the allowed 10% acceptance range you can make adjustments on the test jig and rerun the test by just re-clicking the “run” button for an individual test.

7. If the tests continue to fail, select “Next” and the software will conduct the “EC Troubleshooting Tests.” The Instrument Calibration Tests (Fig. 5.5) checks of the calibration within the FI6000. If these are far out of range it will influence the EC Test load values and will need to return to Geoprobe® for repair. The “Probe Continuity and Isolation Tests” confirm each of the wires is a complete circuit and is fully isolated from one another. If a probe continuity test fails just outside the target range of <8ohms this is typically a contact issue with the test jig and the dipoles. If the continuity is in the thousands of ohms this is a break in the EC wire circuit – either in the probe, the trunkline or the connection between them.

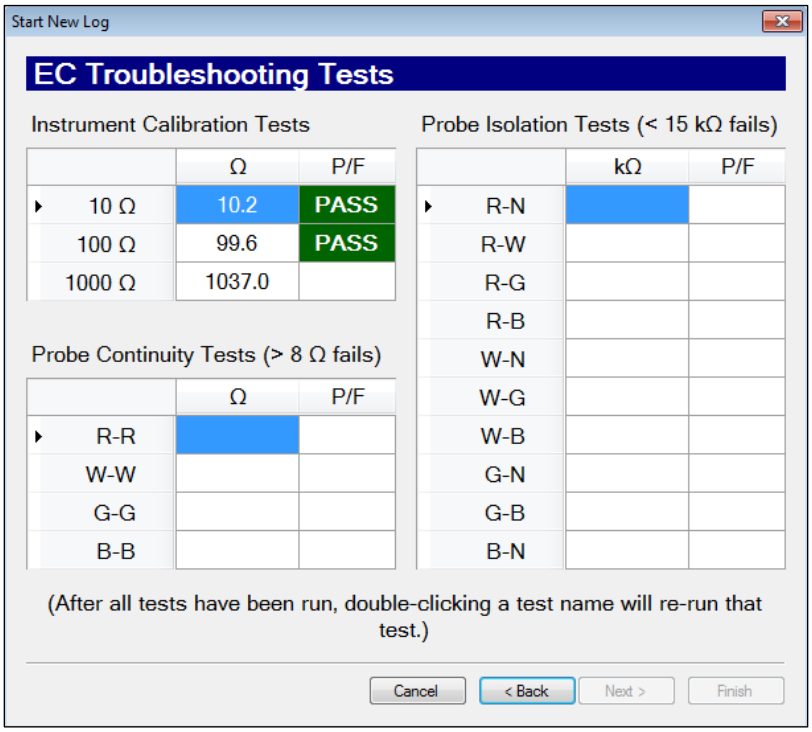


Figure 5.5: EC Troubleshooting Test Screen

8. When these tests are complete select next. In the next screen, the software will provide an EC option, if one is available. The EC Load Test will only work if EC can be operated in Wenner array meaning all of the EC wires in the continuity test pass with results <8ohms on the individual circuits. EC can be operated and collect good data in one of the dipole areas: top, middle or bottom dipole. If the R-R test fails but the others pass the software will provide the option in the next screen to run either middle dipole or bottom dipole arrays. If R-R and G-G are both an incomplete circuit then no EC array is available to run and a new probe must be connected or the problem fixed. In the Wenner configuration it requires 2 adjacent dipoles to operate in dipole mode. If an EC array is chosen and run in this last manner then all of the EC information collected will be bad data.

B. HPT Reference Testing

Reference testing is done to ensure that the HPT pressure sensor is in working order and to evaluate the condition of the HPT injection screen. The HPT reference test calculates atmospheric pressure which is required to obtain static water level readings and to determine the estimated K values for the log in our post log processing software the DI Viewer.

Reference Test Procedure

1. Connect a clean water source to the HPT controller and turn on the pump.

2. Allow water to flow through the system long enough so that no air remains in the trunkline or probe (air in the system can cause inaccurate flow and pressure measurements).

3. Insert the probe into the HPT reference tube and allow the water to flow out the valve adjusting the flow rate to between 250-300ml/min (Fig. 5.5). Ensure that the reference tube is close to vertical.

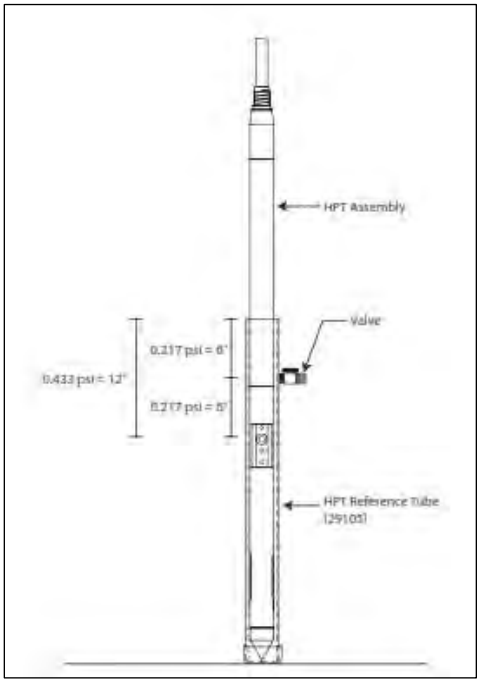


Figure 5.5: HPT Reference Test Setup

4. With a stable pressure reading and the water flowing out of the valve select "capture" - bottom with flow (Fig. 5.6)

5. Close the valve and allow the water to overflow the top of the tube. When the pressure stabilizes select "capture" - top with flow.

6. Shut off the water flow. When the pressure stabilizes select "capture" - top flow = 0.

7. Open the valve and allow the water to drain out. When the pressure stabilizes select "capture" - bottom flow = 0.

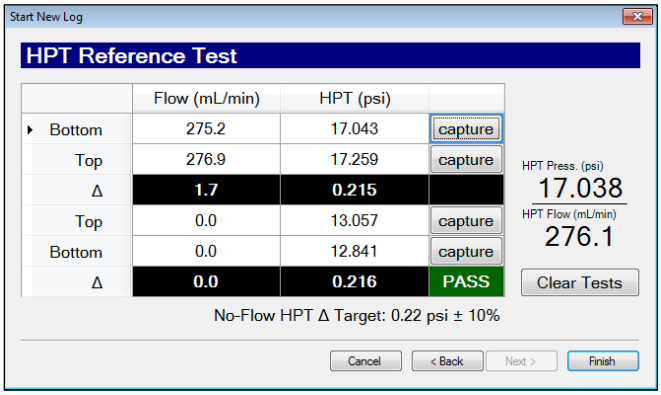


Figure 5.6: HPT Reference Test Screen

The HPT reference test reading flow = 0 is the true test of the condition of the pressure sensor and is the only sensor test to have a pass/fail reading on it. Ideally, the pressure difference between the top and bottom values will be 0.22psi (1.52kPa). Typical pressure readings of the sensor will be in the 12-15psi (83-104kPa) range.

5.4 Running an HPT Log

1. Place the rod wiper on the ground over the probing location and install the drive cushion in place of the anvil of the probing machine.

2. Place the probe tip in the center of the rod wiper, and place the slotted drive cap on top of the HPT probe.

3. Start the HPT water flow. **Note**: It is important that there is always water flowing when the probe is advanced to avoid soil particles from moving through the screen and causing problems with the pressure readings or causing a blockage behind the screen.

4. Adjust the probe so that it is vertical and advance the probe until the HPT screen is at the ground surface.

5. Click the trigger button in the lower right hand corner of computer screen. (The Trigger label will flash and the background will change from yellow to green).

9. Advance the probe at a rate of 2cm/s. If necessary, feather the hammer to maintain this advance rate.

10. Perform a dissipation test (Section 5.4) in a zone of higher permeability indicated by lower HPT pressure.

11. After completing the log, press the trigger button again and select “Stop Log”.

12. Pull the rod string using either the rod grip pull system or a slotted pull cap. Run a post-log EC test and HPT response test (Section 5.2).

5.5 Performing a Dissipation Test

At least one dissipation test must be performed in order to calculate the static water level and estimated K readings from the log. Dissipation tests need to be performed below the water table and are best in zones of high permeability where the injection pressure can dissipate off quickly once the flow is shut off.

1. Stop in a zone of higher permeability which is indicated by lower HPT inject pressure.

2. Switch the DI Acquisition display view from the depth screen to the time screen by pressing the F10 key (F9 and F10 toggle between the depth and time screen of the acquisition software).

3. The screen will be grayed out which means that the data up to that point has not been saved. Select “Start Dissipation Test” which will turn the screen from gray to a white background indicating that you are now saving the time data.

4. Now shut the pump switch off and when the line pressure reaches zero, turn the flow valve off.

5. The HPT Pressure will begin to drop (dissipate the hydrostatic increase) and allow it to stabilize so very little visible drop in pressure is seen. When the pressure has fully dissipated turn the flow valve and the pump switch back on. When the flow and pressure are reestablished select “End Dissipation test.”

6. Select F9 to return to the depth screen and advancing the tool into the ground.

   Note: Performing a dissipation test in zones of higher permeability may only take 30 seconds or so but if the HPT pressure was higher to start with it may take a long time up to several hours to dissipate off to equilibrium. This is why targeting the most permeable zone to perform the dissipation tests is most desirable.

6.0 HPT Log and Interpretation

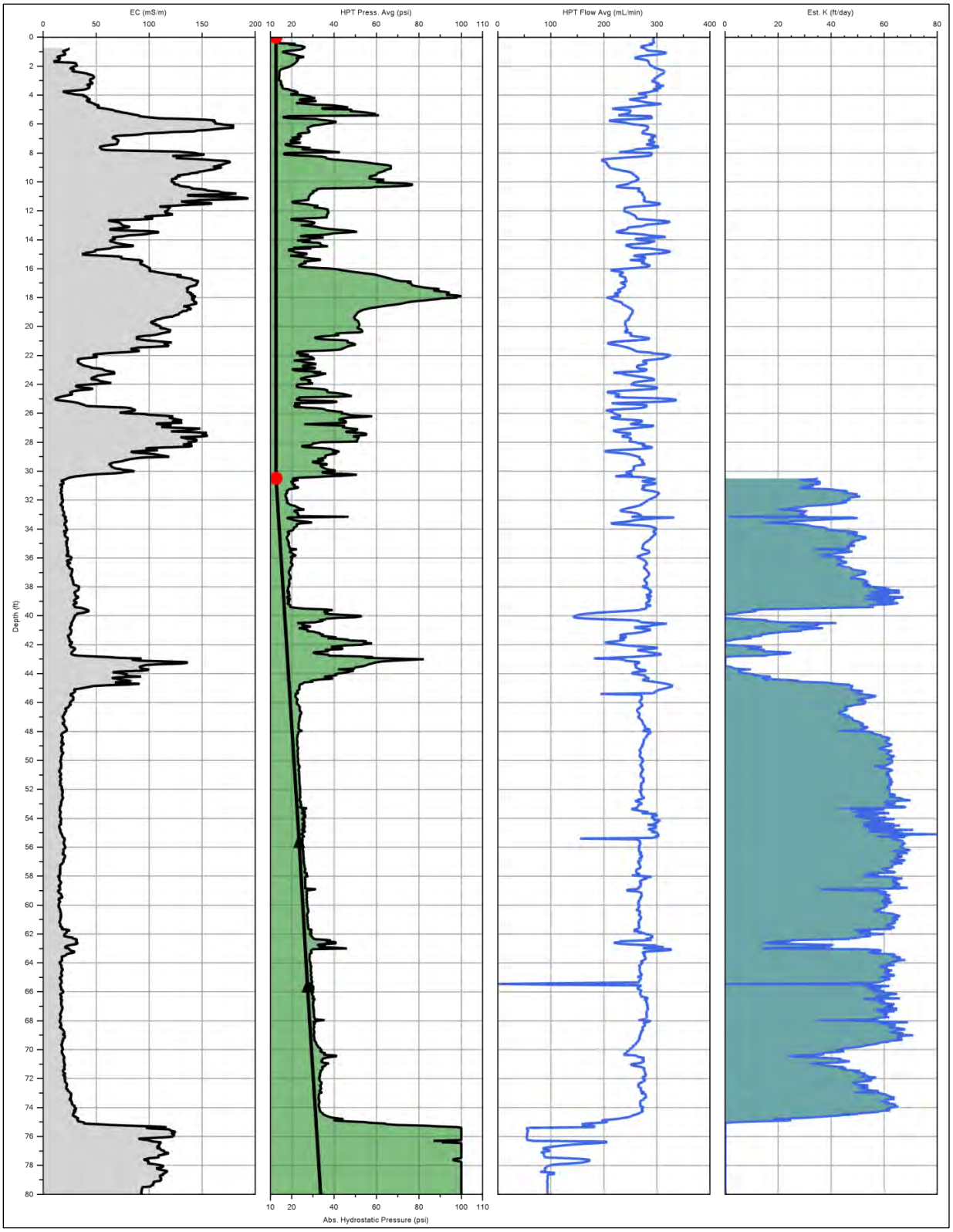


Figure 6.1: HPT Log file showing (left to right):
Electrical Conductivity (EC), HPT Injection Pressure with Hydrostatic Profile, HPT Flow, and Estimated K

A typical HPT log is shown in figure 6.1, which consists of both the HPT pressure response and electrical conductivity. In general, both HPT pressure and EC values increase with decreasing grain size, and decrease with increasing grain size. The log in Figure 6.1 shows good consistency between EC and HPT pressure for the majority of the log. It is only between 32'-42'bgs that we see some divergence of the graphs with higher HPT pressure while the EC readings remained low. This can happen for reasons such as poor mineralogy of the soil. Refusal was encountered in a shale layer beginning at 75'bgs and it can be noted that as we enter this layer the HPT flow gets suppressed as the pressure reaches a maximum value of 100psi (690kPa). The second graph of the log shows the hydrostatic profile on the secondary series of the graph. The hydrostatic profile has 2 black triangles which indicate where dissipation tests were run and used to calculate the profile. The red circle indicates the calculated water table based upon where the hydrostatic profile intersects atmospheric pressure. The fourth graph is the estimate K or groundwater flow graph. This is calculated based upon HPT pressure and HPT flow relationships. Less permeable soil will have less groundwater flow.

It is fairly common to see zones where EC readings and HPT pressure contradict one another. In cases where EC readings are low and HPT pressure trends higher as in the log in Figure 6.1 the following are possible reasons:

- Poor mineralogy of the soil particles resulting in silt and clay soils with very low EC readings. This is seen in many locations along the east coast of the United States.
- Silts intermixed with sand particles.
- Weathered bedrock may have low EC but would have low permeability.

Where we have cases of higher EC and lower HPT pressure typically is due to an ionic influence in the soil or groundwater. These higher EC readings can range from very slight to higher than typical soil readings. Very high EC readings can occur when the probe contacts metallic objects in the soil which will ground them out and typically will cause hard sharp spikes in the EC data.

- Chloride or other ionic contaminant (sea water, injection materials)
- Sea Water intrusion
- Wire, metal objects or Slag

In cases where HPT and EC do not confirm one another it is important to take confirmation soil and/or groundwater samples to help understand the difference between the two graphs.

7.0 Troubleshooting

7.1 Using the HPT Controller Test Load

The HPT Controller Test Load (206552) is included with the HPT Controller to help troubleshoot the HPT pressure sensor, trunkline, and controller. If there is a major problem with the HPT pressure sensor or the system wiring the system will not read anywhere close to atmospheric pressure with the probe at the surface. Commonly if the HPT sensor has broken the software will read either a maximum or minimum value which would be 100psi or 0psi (690kPa or 0kPa). If there is damaged wiring or nothing is connected to the controller the system typically reads 50psi (345kPa).

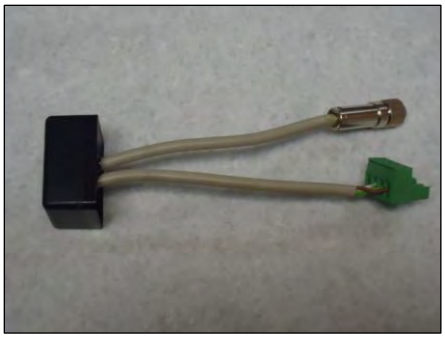


Figure 7.1: HPT Test Load (206552)

To use the test load, set up the system as previously described. Turn on both the field instrument and HPT controller and start the HPT software. Plug the green wire connector of the test load into the HPT sensor connector on the back of the HPT controller. If the pressure sensor value reads between 25-35psi (172 – 241kPa) the controller is able to properly read pressures so the problem is in the trunkline or the HPT sensor. If HPT controller has not moved from what it was reading or is way out from the expected value of the load test the HPT controller may require servicing. Contact Geoprobe Systems® for service.

Next, connect the HPT sensor wires of the trunkline to the controller with the green connector and then connect the chrome connector side of the test load to the female chrome connector on the downhole end of the trunkline in place of the pressure sensor. Again, the pressure value displayed on the field instrument should read between 25-35psi (172 – 241kPa) and should be the same as what was seen with the load test connected into the controller. If the load test read the expect value 25-35psi (172 – 241kPa) at both locations then both the trunkline and the controller are working properly and the problem is in the HPT sensor. If the test load read the expected value at the controller but not at the end of the trunkline, the trunkline may be defective and should be replaced. Before restringing another HPT trunkline, first connect the new trunkline sensor wires into the HPT controller and the downhole end into the test load. If the system now reads in the expected test load range the original trunkline needs replacing.

Finally, connect the pressure sensor to the trunkline. If it reads atmospheric pressure, approximately 12-15psi (83-104kPa), then the pressure transducer is functioning properly. However, if it does not, replace the sensor with a new one and re-check the pressure reading. Be sure to enter the new sensor calibration values into the software prior to starting the new log. Additional pressure sensors may be purchased from Geoprobe®.

7.2 Common Problems

Problem: The pressure transducer is connected to the trunkline, but the software is reporting a reading of ~ 50psi (345kPa).
Solution: Make sure all trunkline wires are secured to the green terminal blocks and plugged in to the back of the HPT controller and sensor chrome connectors are secure. Check components using the HPT Controller Test Load (Section 7.1).

Problem: The pressure transducer is connected to the trunkline, but the software is reporting a reading of 100psi or 0psi (690kPa or 0kPa).
Solution: Make sure all of the connections are good and recheck the pressure reading. If still bad connect a new HPT pressure sensor onto the trunkline and see if it reads atmospheric pressure. If not check all the components using the HPT Controller Test Load (Section 7.1).

Problem: The pressure with flow values keep drifting when water is flowing out the port or over the top of the reference tube.
Solution 1: If the trunkline was just connected and flow was just started air may still be in the lines. Allow the water to continue to flow through system which will purge out the remaining air. When it appears that most of the air is out of the lines press your thumb over the injection screen for a few seconds to help drive out any remaining air from the trunkline.
Solution 2: There may be debris behind the screen. Remove the HPT screen with the membrane wrench and turn the water flow on, use a small screwdriver to scrap out any debris in the screen socket as well as any that might be behind the screen. Replace the screen and retry the reference test with flow.
Solution 3: If the with flow pressure values continue to not settle down and provide close to the expected difference for a 6" water column then the problem may be inside the HPT control box. When you remove the cover of the HPT controller there will be a brass filter located on the left side when viewing from the front of the instrument (Fig 7.2). Particulates and precipitates can collect inside this filter causing problems with HPT pressure stability. Remove this filter and open up using appropriate wrenches. The filter can be easily cleaned by rinsing water over the screen. Reassemble and return to its proper location inside the control box. Resume reference testing the system.

Figure 7.2: Location of Inline Filter in K6300 and buildup of particulates in filter.

Problem: Atmospheric pressure values are way off from normal (12-15psi (83-104kPa)) after installing a new HPT sensor.
Solution: Check the calibration values that were entered into the software to ensure that they are correct.

Problem: Winterizing the HPT system for subfreezing work or air transport.
Solution: Pump RV antifreeze through the HPT pump and bypass pathway which can be done by blocking off the inject line. The trunkline can either be purge free of water by the pump or with an air compressor. NOTE: Never purge the HPT Controller of water using an air compressor this will damage sensor components in the controller.

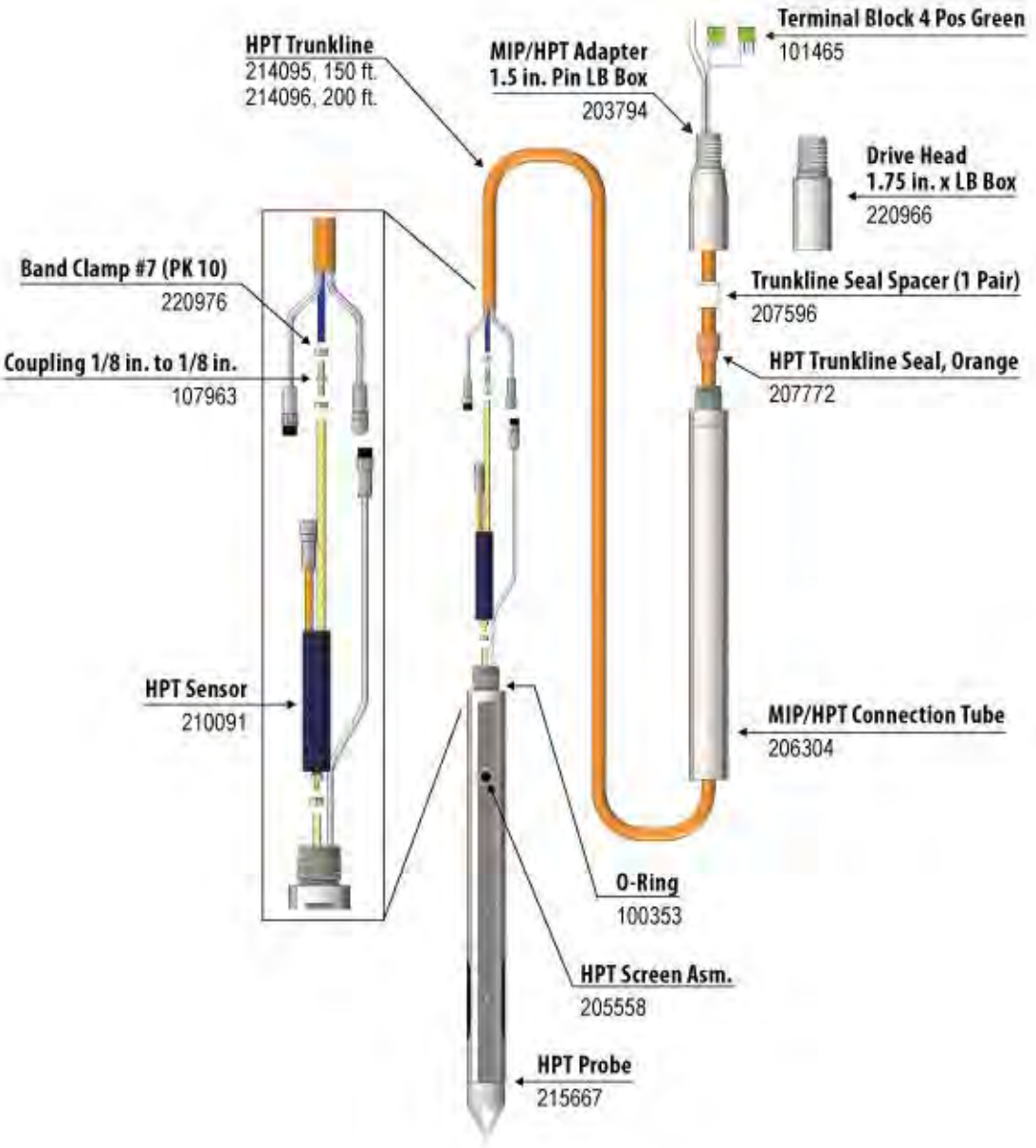
Problem: HPT flow sensor reading 0ml/min
Solution: If the flow sensor reads 0 or some other stabile number that does not correspond to actual water flow out the controller likely the flow sensor has been damaged. The flow sensor is very susceptible to damage from freezing. To repair the HPT flow sensor contact Geoprobe-DI technical support.

Problem: EC won't pass the QA tests.
Solution: Check the trunkline to probe EC connections ensuring they are tight. Run the troubleshooting tests (Section 5.3A), test EC on a new probe. If multiple probes and trunklines do not pass EC isolate the FI6000 instrument using the EC bypass cable (204025). The bypass cable is a six inch long cable that connects between the Test input and the EC probe connections on the back of the FI6000. Once connected start an EC or HPT log and fail the EC test load tests on purpose and run the EC troubleshooting tests (Figure 5.5). If the EC calibration or the EC continuity readings fail there could be an issue in the FI6000. In this case contact Geoprobe-DI technical support. If all of the troubleshooting tests pass then the problem is not in the instrument but in the trunkline, probe or their connections.

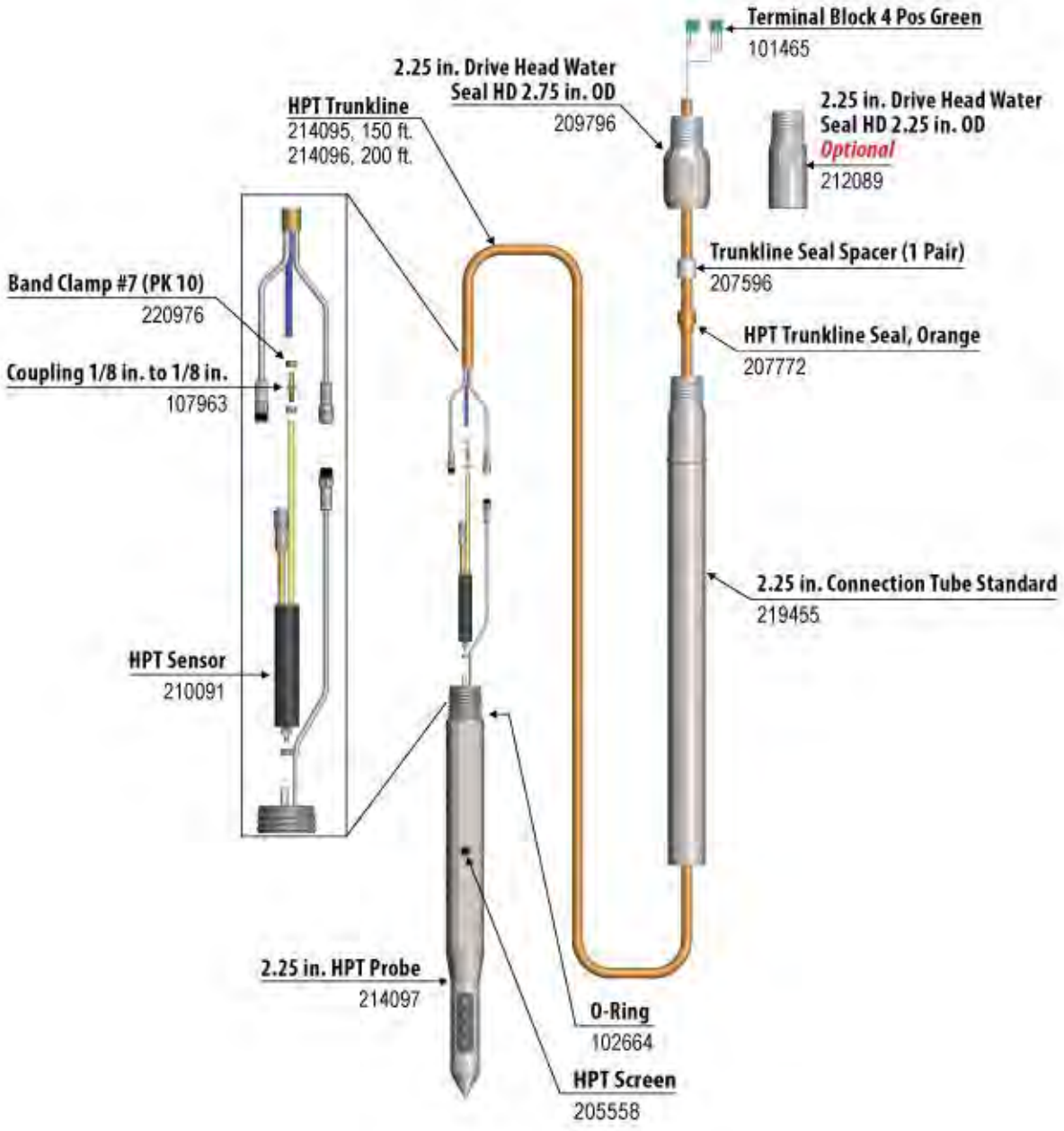
APPENDIX I

HPT Tool Configurations

HPT – K6050 (1.5 in / 1.75 in. system)



HPT – K8050 (2.25 in. system)



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Geoprobe® Membrane Interface Probe (MIP)

Standard Operating Procedure

Technical Bulletin No. MK3010
PREPARED: May, 2003
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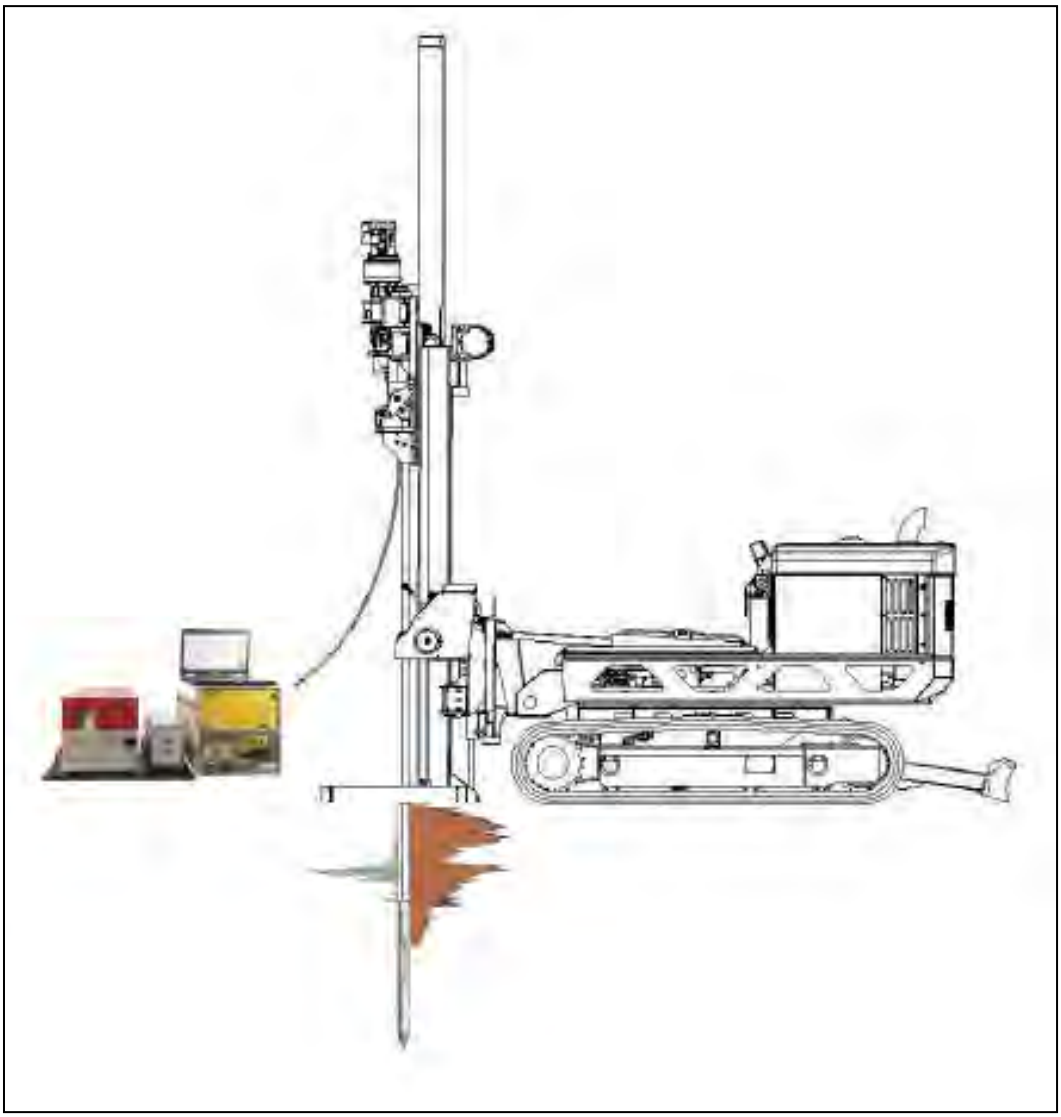

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1.0 Objective

This document serves as the standard operating procedure for use of the Geoprobe Systems® Membrane Interface Probe (MIP) used to detect volatile organic compounds (VOCs) at depth in the subsurface.

2.0 Background

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.
**Geoprobe® is a registered trademark of Kejr, Inc., Salina, Kansas.*

Membrane Interface Probe (MIP): A system manufactured by Geoprobe Systems® for the detection and measurement of volatile organic compounds (VOCs) in the subsurface. A heated probe carrying a permeable membrane is advanced to depth in the soil. VOCs in the subsurface cross the membrane, enter into a carrier gas stream, and are swept to gas phase detectors at ground surface for measurement.

2.2 Discussion

The MIP is an interface between contaminates in the soil and the detectors at ground surface. It is a mapping tool used to find the depth at which the contamination is located, but is not used to determine concentration of the compound. Two advantages of using the MIP are that it detects contamination in situ and can be used in all types of soil conditions.

The MIP is a logging tool used to make continuous measurements of VOCs in soil. Volatile compounds outside the probe diffuse across a membrane and are swept from the probe to a gas phase detector at ground surface. A log is made of detector response versus probe depth. In order to speed diffusion, the probe membrane is heated to approximately 121°C. (Refer to Figure 2.1).

Along with the detection of VOCs in the soil, the MIP also measures the electrical conductivity of the soil to give a probable lithology of the subsurface. This is accomplished by using a dipole measurement arrangement at the end of the MIP probe so that both conductivity and detector readings may be taken simultaneously. A simultaneous log of soil electrical conductivity is recorded with the detector response.

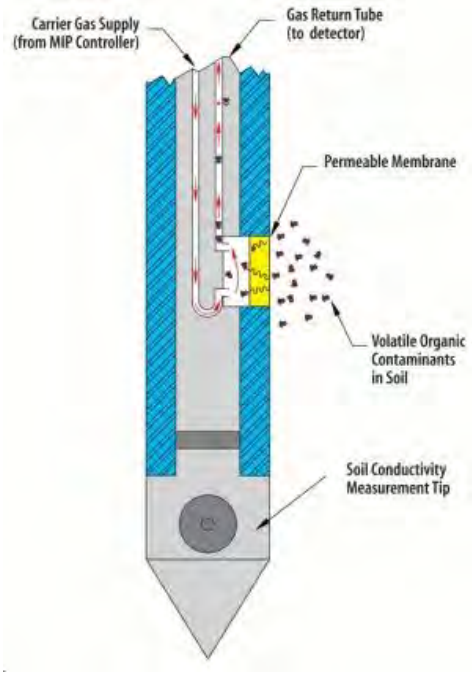


Figure 2.1: Diffusion across the membrane

Interpretation of electrical conductivity (EC) logs comes with field experience. It is very important that soil core samples are taken to confirm lithologic changes as each EC log is unique per site. As a generalization, a high conductivity reading indicates a smaller grain size and a low conductivity reading indicates a larger grain size (See Fig. 2.2).

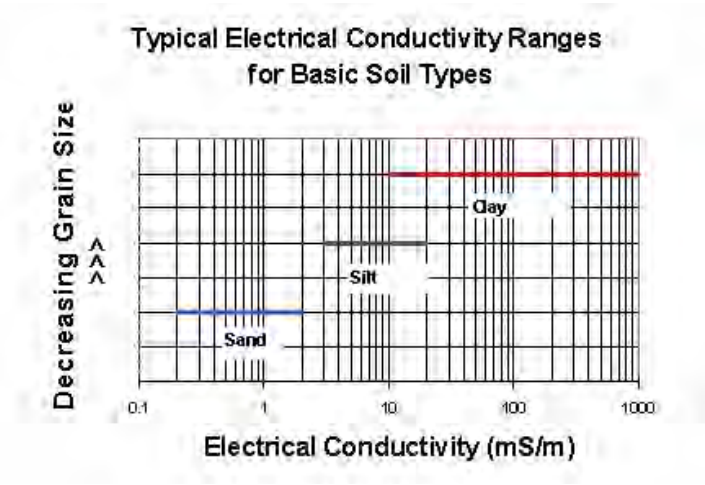


Figure 2.2: Generalized Electrical Conductivity Readings

3.0 MIP/EC Interferences

3.1 Detector saturation may require a short period of time for the detector to return to baseline after a log has been performed in higher concentrations. The MIP system can be used in free product environments with the operator monitoring and making the necessary adjustments to the detector and software gain/attenuation settings to account for the higher voltage readouts.

3.2 The MIP system can be operated in a wide range of contaminant concentrations from low dissolved phase to free phase materials. During a log and the removal of the tool string, contaminants can absorb onto the surface of the membrane and trunkline material causing elevated detector baseline signals. It is very important that the probe and trunkline system is clean enough to see the low concentrations typically used in the chemical response test. Not adequately decontaminating the probe prior to performing a response test can elevate the concentration of the standard causing an inaccurate high response to the specific concentration of standard that was prepared for the test.

3.3 Electrical conductivity can provide false positives or higher than expected readings when the soil is impacted by ionic plumes (chloride, nitrate) originating from, but not limited to: agriculture practices, seawater, salt storage, mining practices. Encountering metallic objects in the subsurface can also result in high EC readings.

3.4 Some silt and clay soils will not have the typical ionic composition that an operator may be used to for similar soils. This can result in lower than expected readings and perhaps cause misidentification of the associated soil zone based on typical response of a courser grain material. This can occasionally be found in clays that have had the minerals leached out or in intermixed silt-sand zones.

4.0 Tools and Equipment

The following equipment is needed to perform and record MIP logs. Basic MIP system components are listed in section 4.1 and shown in Figure 4.1. Additional equipment needed to run MiHPT logs is listed in section 4.2 with optional equipment listed in section 4.3. Refer to Appendix V for a detailed illustration of the GC1000 setup configuration. Appendix VI shows the common MIP probe tool string diagrams. There may be more required tools as determined by your specific model of Geoprobe® direct push machine.

4.1 Basic MIP System Components

Description	Quantity	Material Number
Field Instrument, 120V (Model FI6000)	-1-	213940
Field Instrument, 220V (Model FI6003)	*	213941
MIP Acquisition Software	-1-	214128
MIP Controller, 120V (Model MP6505)	-1-	214137
MIP Controller, 220V (Model MP6507)	*	214139
Gas Chromatograph, 120V with PID, FID and XSD	-1-	213946
Gas Chromatograph, 220V with PID, FID and XSD	*	213947
MIP Probe, 1.75 inch	-2-	214143
MIP/HPT Connection Tube	-2-	220912
MIP/HPT Drive Head 1.5in. rods	-2-	203794
Slotted 1.5” Drive Cap, Threadless	-2-	206609
MIP Probe, 2.25 inch	**	214152
2.25 Connection Tube	**	219455
2.25 Inch Water Seal Drive Head	**	212089
2.75 Inch Water Seal Drive Head	**	209796
Slotted 2.25” Drive Cap	**	211798
MIP Trunkline 100 ft.	(optional)	204077
MIP Trunkline 150 ft.	-2-	202570
MIP Trunkline 200 ft.	(optional)	204655
Agilent ADM 1000 Digital Flow Meter	-1-	600227
Hydrogen Gas Regulator	-1-	600137
Nitrogen Gas Regulator	-1-	600175
Vertical Gas Bottle Rack	-1-	214121
MIP Service Kit	-1-	214142
EC Bypass Cable	-1-	204025
Stringpot, 100-inch	-1-	214227
Stringpot Ground Plate	-1-	220775
Stringpot Cordset, 65-feet (19.8 m)	-1-	202884
Stringpot Mounting Bracket (6600/7700)	(optional)	202947
Stringpot Mounting Bracket (78 Series)	-1-	209511

Stringpot Foot Bracket (6600/7700)	(optional)	201816
Stringpot Foot Bracket (78 Series)	-1-	209533
Stringpot Piston Weight	-1-	214225
Drive Cushion (GH60)	-1-	204278
Rod Wiper, 1.25/1.5” Rods	-1-	600341
Rod Wiper Weldment	-1-	204387

4.2 Additional MiHPT System Components

Description	Quantity	Material Number
HPT Flow Module, 120V (Model K6300)	-1-	214091
HPT Flow Module, 220V (Model K6303)	*	214093
HPT Reference Tube 1.75 in HPT Probe	-1-	212689
HPT Reference Tube 2.25 in HPT Probe	**	211762
MiHPT Probe, 1.75 inch	-2-	219228
MiHPT Probe, 2.25 inch	**	214117
MiHPT Connection Tube	-2-	219594
MiHPT Drive Head for 1.5” rods	-2-	220545
MiHPT Trunkline 100 ft	(optional)	214113
MiHPT Trunkline 150 ft	-2-	214114
MiHPT Trunkline 200 ft	(optional)	214115
Coupling 1/8 to 1/8 Tube	-5-	107963
Coupling 0.135 to 0.150 Tube	-5-	220978
Oetiker Band Clamp 4.7mm x 5.7mm	-10-	220977
Oetiker Band Clamp #7	-10-	220976
HPT Sensor Module	-2-	210091
Heated Trunkline Seal Asm.	-4-	211768
HPT Test Load	-1-	206552
HPT Service Kit	-1-	205599

4.3 Optional Accessories

Description	Quantity	Material Number
Heated Trunkline Controller, 120V (Model MP7000)	-1-	214147
Heated Trunkline Controller, 220V (Model MP7003)	*	214148
MIP Heated Trunkline 100 ft.	(optional)	214150
MIP Heated Trunkline 150 ft.	-1-	214151
MIP Heated Transfer Line 8 ft.	-1-	214149
MIP Breakout Connection Panel.	-1-	211100
Roll-out Rod Rack (30-1.5in rods)	-1-	213627
Water Transport System.	-1-	203450

*Use in place of 120V components if desired.

**Use in place of 1.75 inch probe and components if desired.

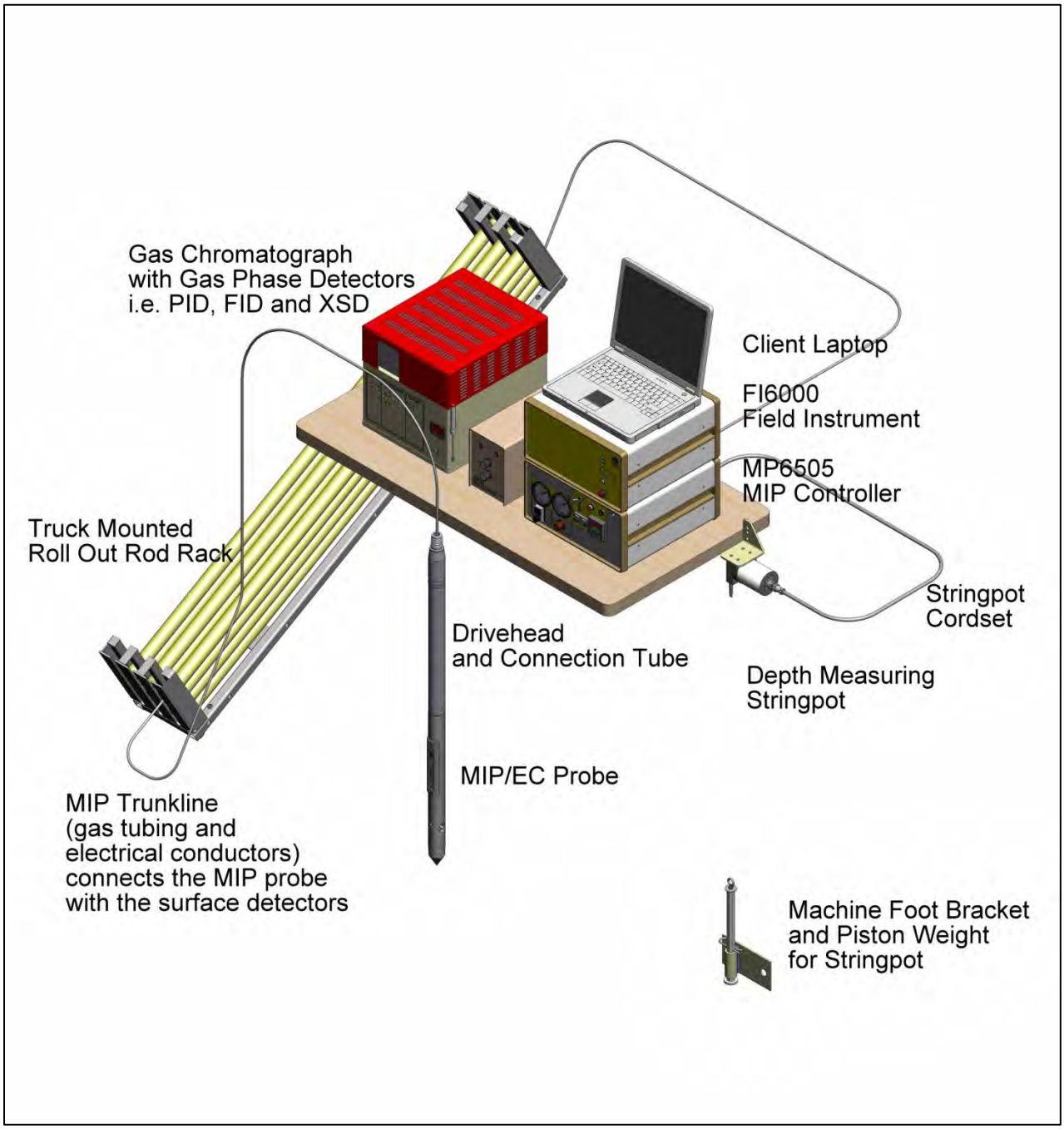


Figure 4.1: MIP System Components

5.0 Quality Assurance/Quality Control

Quality assurance (QA) testing of each of the sensors on the probe is too be performed before and after each log to validate that the equipment is capable of generating good data. The MIP tool includes chemical response tests (Section 5.1.3) which are performed to ensure that the probe membrane, trunkline and detectors are capable to providing ample signal over baseline noise to a known site contaminant at a given concentration. The electrical conductivity (EC) sensor is tested using an EC dipole test (Section 5.2) with low and high readings typical to EC values of the soil. The HPT sensor is included on the MiHPT probe and is tested using the HPT reference test (Section 5.3) which confirms the sensors ability to accurately measure a 6" column of water and provides an accurate measurement of atmospheric pressure.

Quality control (QC) is performed during and after each log is generated. Log QC will answer the following questions to ensure that the data is good and makes sense:

1. Does the log look correct? Does the elctrical conductivity appear to be in an acceptable range? Is there anything seen in the log that would make you suspect that the system wasn't working correctly, ie. a loss of temperature or gas pressure of the system.
2. Response consistency? As more logs are completed do they show general consistency of EC and contaminant response? Review a cross section of logs in the DI-Viewer (Appendix IV).
3. Repeatablity? Replicate logs may be run every 10 to 20 locations to verify repeatability.
4. Are my lithogy changes consistant with physical soil cores? Take continuous or discreet confirmation soil samples to confirm your lithogy changes in EC.
5. Do the MIP detector responses make sense for contaminant concentration. This must be verified by the collection of water or soil samples for lab analysis to confirm contaminants and their concentrations.

5.1 MIP Chemical Response Test:

Chemical response testing is an important quality assurance measure used to validate each log by proving that the integrity of the detector system is intact. With the chemical response test the operator introduces a working standard (known site contaminant of concern) at a known concentration to the membrane for a set time of 45 seconds which should match the residence or holding time at each sampling interval. Two acceptable methods of introducing the standard to the membrane are shown in Figures 5.4 and 5.5.

Typical site contaminant of concerns which are used in MIP chemical response tests include but are not limited to Benzene, Toluene, Trichloroethylene or Perchloroethylene. The stock standard should be made up from one of these or an appropriate mix of chemicals.

5.1.1 Preparation of the Stock Standard

Preparation of the stock standard is critical to the final outcome of the concentration to be placed into the testing cylinder. The following items are required for preparing the stock standard:

- Neat sample of the analyte of interest (i.e.: Benzene, Toluene, TCE, PCE, etc.) purchased from a chemical vendor
- Microliter syringes (recommended to have: 25μL, 100μL and a 500μL or 1,000μL syringes).
- 25-mL or 50-mL Graduated cylinder
- Several 40-mL VOC vials with labels
- 25mL Methanol

1. The total volume of methanol and the compound added should equal 25mL.
2. Pour methanol into graduated cylinder to the 23.5-24mL mark, the volume depends upon the compound density (Table 5.1).
3. Pour the methanol from the graduated cylinder into a 40mL VOC vial.
4. Add the appropriate volume of desired neat analyte into 40mL VOC vial containing methanol. The required volume of neat analyte for seven common compounds is listed in Column 3 of Table 5.2. The equation in table 5.1 shows how to calculate the appropriate neat analyte volume for other compounds of interest given the appropriate density.
5. Label the vial with the name of the stock standard (i.e. Benzene, Toluene, TCE, PCE), concentration (50mg/mL), date created, and created by (your name or initials).
6. Stock standards need to be kept cold in a refrigerator or freezer to ensure they can last up to one month otherwise they should be made up more frequently as often as every 3 days if there is not cooling during the summer months. The more volatile the compound the quicker it will lose its concentration.

Stock Standard Calculations
25mL (methanol) x 50mg/mL = 1250mg 1250mg x 1/density of analyte = amount of neat material to be placed with methanol to make up 25mL total volume *Example: Preparation of 50mg/mL Benzene standard.* *1250mg x 1/0.8765mg/μL = 1426μL* *Use 1426μL of neat Benzene in 23.5mL of Methanol to get a 50mg/mL stock standard.*

Table 5.1: Stock Standard Preparation Calculations

Compound	**Density (mg/μL)**	**Volume (μL) of Neat Standard Required to prepare Working Standard (0.5 L)**
Benzene	0.876	1426
Toluene	0.867	1442
Xylenes	0.860	1453
Methylene Chloride	1.335	936
Carbon Tetrachloride	1.594	784
Chloroform	1.480	845
Trichloroethylene	1.464	854
Perchloroethylene	1.623	770

Table 5.2: Density and required volumes of neat (pure ~100%) compounds used to make a 50mg/mL stock standard into 25 ml of methanol

5.1.2 Preparation of Working Standards

The following items are required to perform response testing:

- Microliter syringes (recommended to have: 10, 25, 100 & 500µL syringes).
- Freshly made 50ml/ml Stock standard
- Testing cylinder made from a nominal 2-in. PVC pipe with a length of 24 in. or 40ml vial
- 0.5 L plastic beaker or pitcher
- Supply of fresh water, 500mL needed per test
- Stopwatch

Volume of 50mg/ml Stock Standard (µL)		Final Concentration mg/L = ppm
10		1.0
100		10
1000		100

Table 5.3: Volumes of stock standard and final concentrations

Figure 5.1: Working standard

5.1.3 Performing the Chemical Response Test

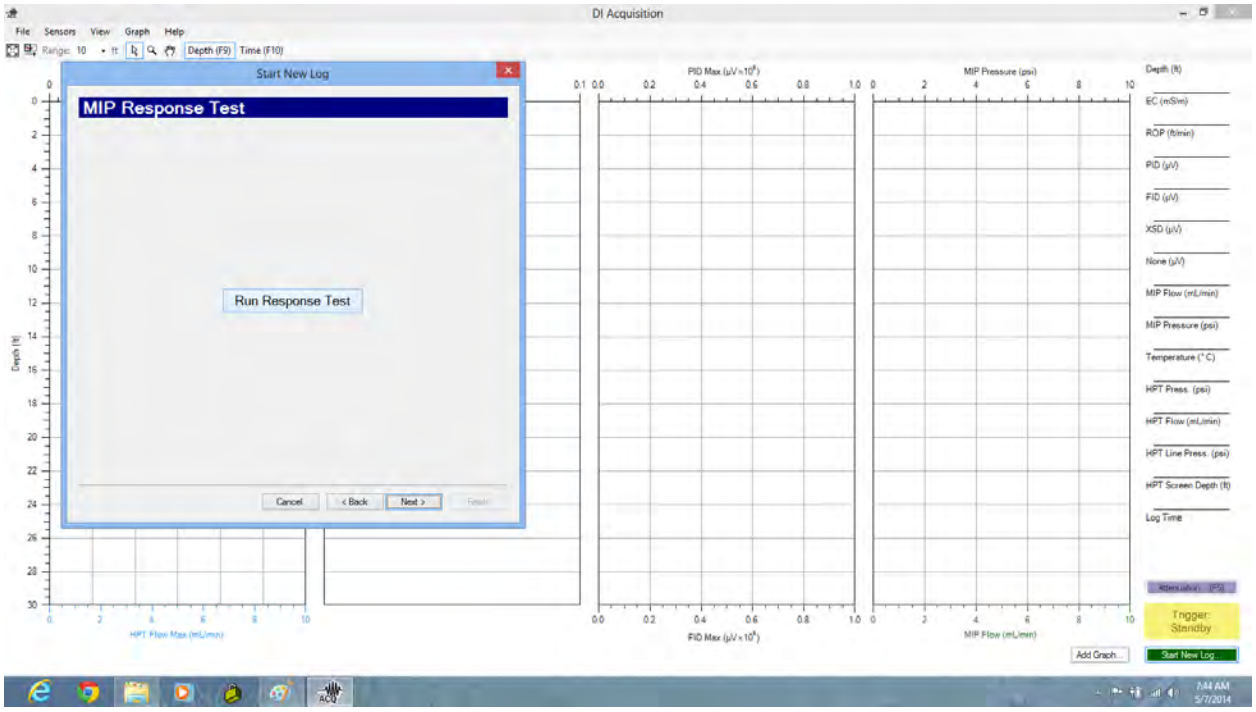


Figure 5.2: DI Acquisition Response Test Screen

1. Begin a new log in the DI-Acquisition software and proceed to the response test screen. The detector signals should be stable before proceeding with the test.
2. Measure out 500mL of tap or distilled water in a graduated beaker.
3. Using Table 5.3, determine the desired volume of stock standard to place into the 500ml measured volume of water to make up the working standard.
4. If the detector baselines have been monitored while the standard was being prepared select “Clear Response Test”.
5. When ready with the working standard prepare to run the response test by exposing the membrane to the working standard. Two acceptable methods are to pour the standard into a nominal 2-inch x 24-inch PVC pipe and insert the probe into the tube (Fig. 5.4) and the other method is to pour the working standard into a 40ml/vial and invert onto the membrane (Fig. 5.5).
6. Start the response test by clicking on the “Run Response Test” button (Fig. 5.2) and immediately expose the MIP membrane to the test solution (Figures 5.4 or 5.5).

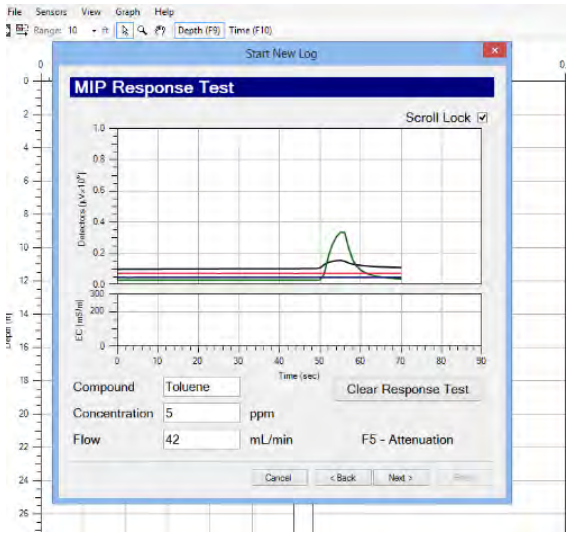


Figure 5.3: Running the Chemical Response Test

7. Leave the membrane exposed to the test solution for 45 seconds. This time is to be equal to the resonance time at each depth interval during probe advancement.
8. Starting the response test time file as the membrane is exposed to the test solution allows the trip time (Section 5.1.4) to be easily calculated by when response begins to climb which is approximately 50seconds in Fig. 5.3.
9. Fresh working standards need to be made for each test, they cannot be reused.
10. After the response has come through the detectors and adequate detector response is seen the operator may select “next” to move to the EC QA test.

Acceptable methods for performing the MIP Chemical Response Test

Figure 5.4 Probe immersed in steel or PVC pipe containing working standard

Figure 5.5: 40ml vial of working standard inverted onto membrane

5.1.4 Determination of Contaminant Trip Time:

Response testing also enables the operator to measure the chemical trip time which needs to be entered into the MIP software to accurately plot the contaminants depth position. The trip time is the time it takes for the contaminant to travel through the trunkline from the membrane to the detectors. The contaminant trip time is influenced primarily by trunkline length and carrier gas flow rates as well as the contaminant makeup specifically boiling point. The chemical response trip time can be determined from the results on the Pre-Log Response Test. Using Fig. 5.6 the Benzene trip time (TT) would be approximately 55 seconds. This response test would need to have been started right when the chemical used in the response test was exposed to the membrane. Additional typical response test graphs are located in Appendix I.

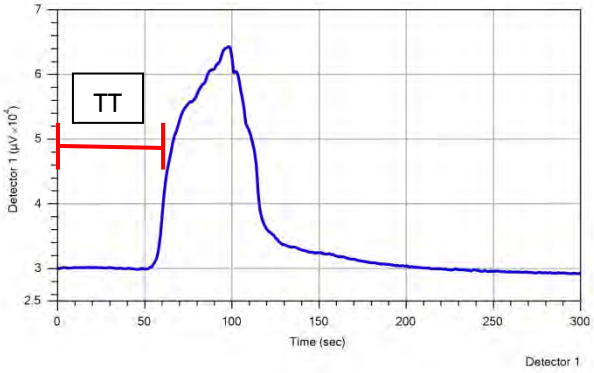


Figure 5.6: 5ppm Benzene on PID

5.1.5 Appropriate Chemical Response Test Concentrations and Response

The compound used in a chemical response test should be the site contaminant of concern or similar which will give you the most accurate response magnitude for that chemical as well as an accurate trip time. If the site objective is to delineate the extent of a dry cleaner plume then the operator should use PCE for the response tests at the lowest possible concentrations ~1ppm or less. If the site objective is to delineate the extent of the petroleum plume from a gas station then the operator should use one of the BTEX compounds or a gasoline mixture at or near the detection limit. If the site objective is to map out a plume source and high contaminant concentrations are expected then the response tests should be run at higher concentrations such as 10ppm-50ppm. This should reduce the possibility of trunkline/membrane carryover masking the chemical response tests.

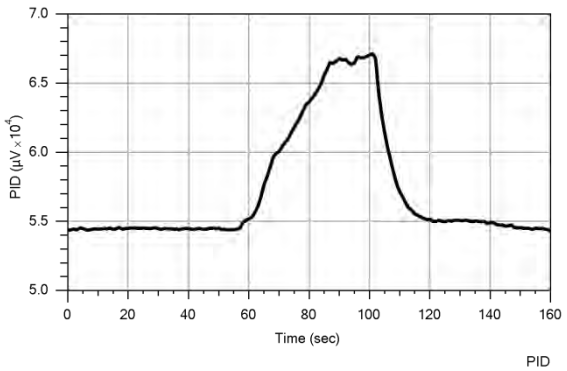


Figure 5.7: 2.5ppm Benzene on the PID

h

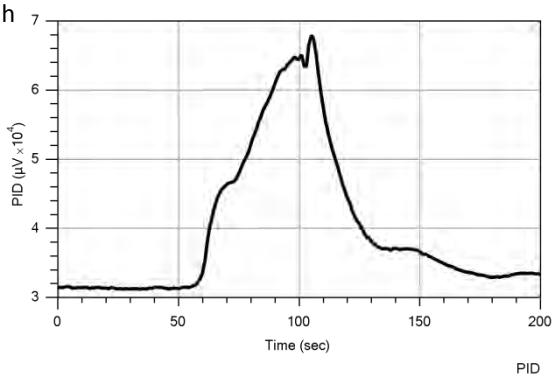


Figure 5.8: 5ppm TCE on the XSD

Figure 5.7 shows a benzene response over baseline on the PID of approximately 12,500μV on a 2.5ppm standard for a 5,000μV/1ppm. Figure 5.8 shows TCE responses over baseline on the XSD of 33,000μV on a 5ppm standard for approximately a 6,500μV/1ppm response.

5.1.6 Minimum Acceptable MIP Response Test Levels and Maintenance Tips

Geoprobe Systems specifies the following guidelines as minimum MIP response test values for performing MIP logging.

Detector systems can vary in the level of response for a given chemical concentration depending on detector age, model, and maintenance performed. However, it should be expected that the detector system would be able to provide at least a 5:1 signal to noise ratio (see Appendix I) for 1ppm of Benzene or TCE. Other compounds or concentration may be performed at the client requests however they may have different response magnitudes and signal to noise ratios at 1ppm. These specifications are required with operation of the PID and XSD (ECD or DELCD as well as alternative halogen detectors). The FID is a less sensitive detector typically used as a confirmation detector and one used for mapping natural gas components.

If the minimum response test levels are not achievable or throughout the day or project the detector sensitivity falls below these levels, the operator must stop and perform maintenance on the system to enhance the sensitivity of the detectors. Corrective actions could include:

- Changing MIP membrane (see section 9.0)
- Making a fresh chemical stock standard (see section 5.1.1). It does not take long for a volatile chemical standard to lose the original concentration.
- Decreasing trunkline carrier gas flow from 40ml/min to 30 or 20ml/min. This will lower the pressure in the trunkline and at the membrane which will increase system sensitivity. If this is corrective action is taken the operator must update the system trip time which has changed.
- Performing detector maintenance
    - Cleaning or replacing the PID bulb
    - Replacing the XSD probe assembly or reactor core
- Checking and adjusting detector gas flows - especially in the FID.
- Replacing the trunkline (an old trunkline can be a source of contaminant phase buildup. This will reduce detector sensitivity by causing contaminant dispersion in the trunkline which results in reduced response levels as well as delayed trip times).

It is wise for the MIP operator to monitor the detector response heights from the chemical response tests to evaluate membrane performance. With increased membrane footage, detector response will fall off indicating that it is time to change the membrane (see Appendix III). It may be possible to rejuvenate a MIP membrane by scrubbing with a wire brush.

5.2 EC Dipole Test

On the FI6000 and the DI-Acquisition software the EC dipole test screen (Fig. 5.10) will open up after the chemical response test is completed. When ready place the low (brass) side of the EC Dipole test jig (Fig. 5.9) between the EC dipole and body of the probe and start the low level test, hold for 5 sec until the system captures the data (Fig. 5.10). Repeat for the high (stainless steel) EC test. These tests should result in readings of 55mS/m and 290mS/m ± 10%.

Figure 5.9: EC Dipole Test Jig

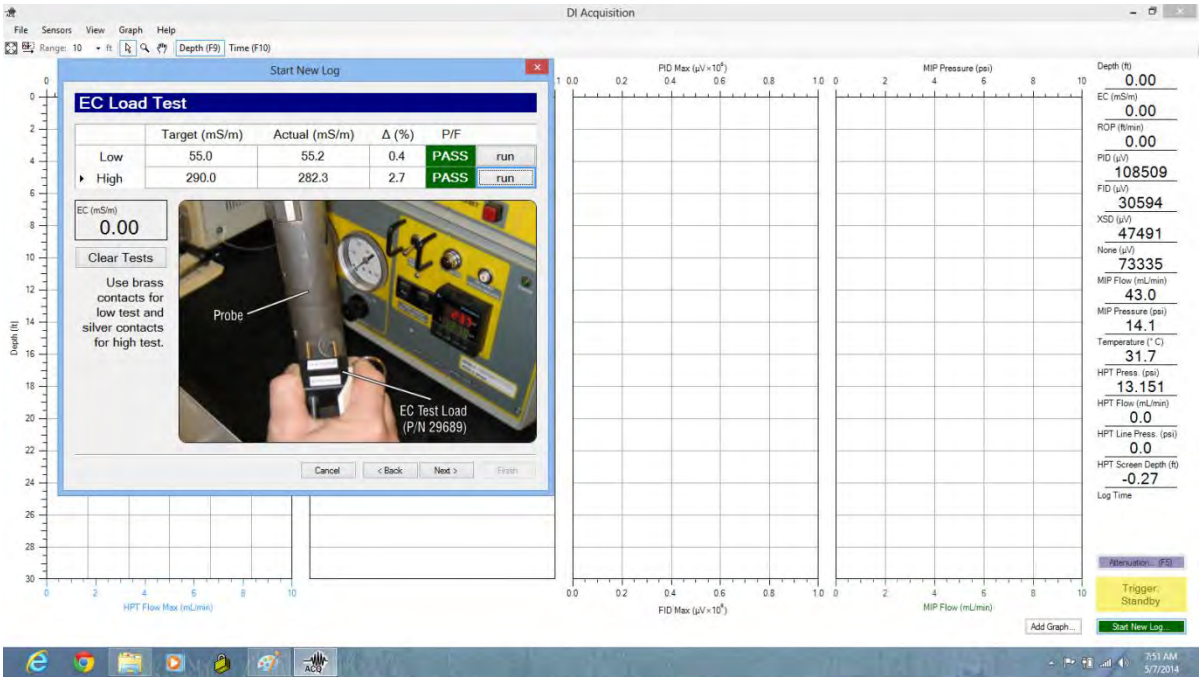


Figure 5.10: EC Dipole Test QA Screen

If the EC readings do not pass, the DI Acquisition (FI6000) software will prompt the user to proceed through a series of troubleshooting tests (Fig. 5.11). These tests will check the EC calibration (Fig. 5.12) to determine if the reason EC Test loads have failed was an issue internal to the FI6000 or if it is external in the trunkline-probe circuit. From here the operator should have an idea where to focus their attention to fix the problem.

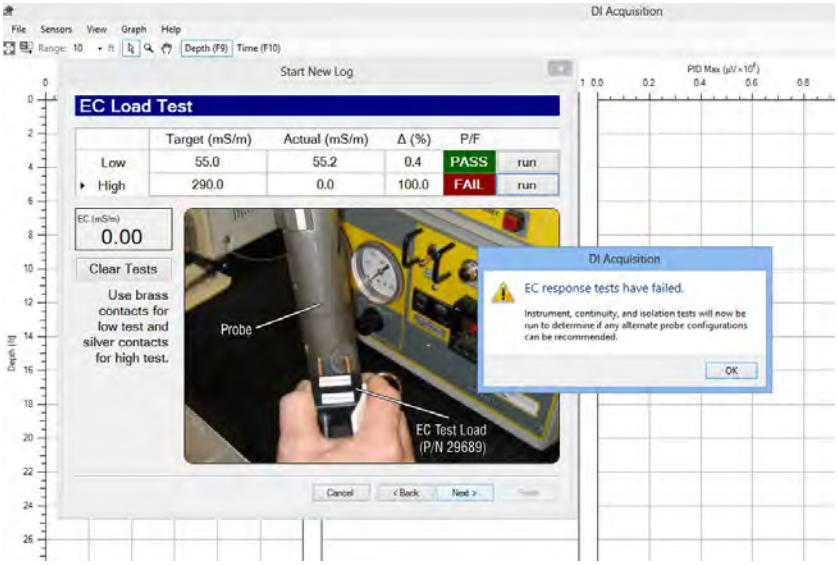


Figure 5.11: Failed EC Dipole Test Error Screen

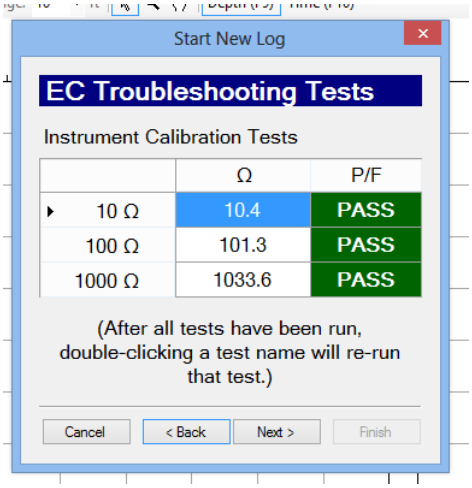


Figure 5.12: EC Calibration Check

5.3 HPT Reference Test – (MiHPT)

Reference testing is done to ensure that the HPT pressure sensor is in working order and to evaluate the condition of the HPT injection screen. The HPT reference test calculates atmospheric pressure which is required to obtain static water level readings and to determine the estimated K values for the log in our post log processing software the DI Viewer.

Reference Test Procedure

1. Connect a clean water source to the HPT controller and turn on the pump.
2. Allow water to flow through the system long enough so that no air remains in the trunkline or probe (air in the system can cause inaccurate flow and pressure measurements).
3. Insert the probe into the HPT reference tube and allow the water to flow out the valve adjusting the flow rate to between 200-300ml/min (Fig. 5.13). Ensure that the reference tube is close to vertical.
4. With a stable pressure reading and the water flowing out of the valve select “capture” - bottom with flow (Fig. 5.14)

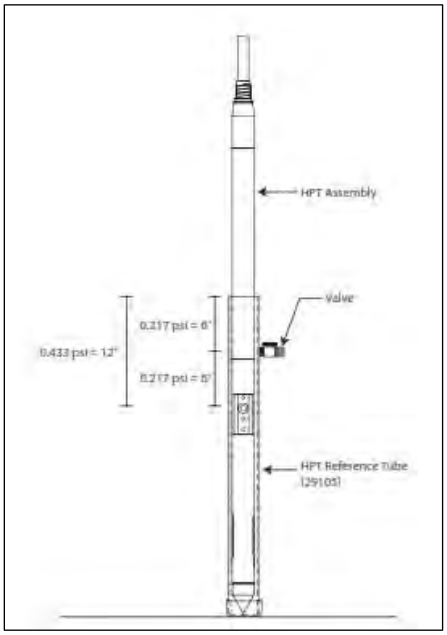


Figure 5.13: HPT Reference Test Setup

5. Close the valve and allow the water to overflow the top of the tube. When the pressure stabilizes select “capture” - top with flow.
6. Shut off the water flow. When the pressure stabilizes select “capture” - top flow = 0.
7. Open the valve and allow the water to drain out. When the pressure stabilizes select “capture” - bottom flow = 0.

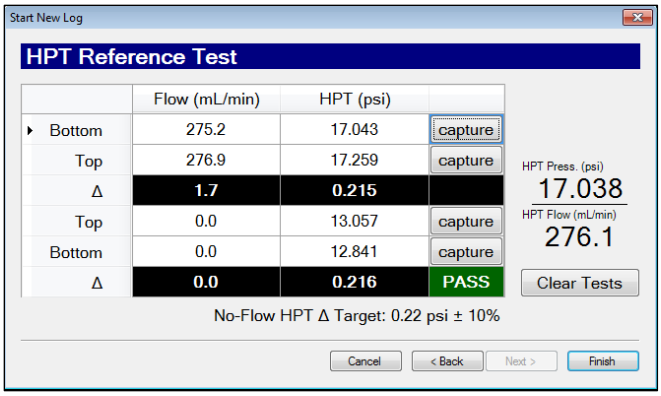


Figure 5.14: HPT Reference Test Screen

The HPT reference test reading flow = 0 is the true test of the condition of the pressure sensor and is the only sensor test to have a pass/fail reading on it. Ideally, the pressure difference between the top and bottom values will be 0.22 psi (1.52kPa). Typical pressure readings of the sensor will be in the 12PSI-15PSI (83kPa-104kPa) range.

6.0 Equipment Preparation for Site Work

The biggest issues to the performance of any specific MIP system is inadequate project preparation and system review, too heavy of a workload which reduces the ability to perform needed maintenance and inexperienced operators how do not fully understand the steps of troubleshooting.

When a MIP system is stored for a period of time between projects, operators must review the equipment and give it a full system checkup which includes checking detector gas flow rates, running response chemical response tests with known chemicals at concentrations at or near required site detection limits. This needs to be performed 1-2 weeks in advance of project work so there is time to obtain required supplies that might be needed for proper operation. A final checkout needs to be performed within 7 days of the project. If the MIP site contaminant of concern is an obscure chemical not normally tested for the operator should run some of that chemical for response tests to confirm it can be detected and to determine reasonable detection limits. The operator should be able to supply the consultant with pre-project performance data of all sensor information to be performed at the site which might include EC, MIP chemical response tests, and HPT reference test information.

If a MIP system is scheduled on a long job or has a number of jobs strung together it is in the best interest of the MIP service company to schedule a maintenance day at least every 3 weeks to allow the operator time to go through system and service the components that need attention. This will help to be able to keep to system performing well for the company and their clients. Pre project performance must still be able to be produced.

New operators will always be needed as the MIP community continues to grow, however it is imperative that operators who are running the MIP systems on their own have been properly trained by experts from their own company or at Geoprobe Systems® headquarters. An inadequately trained operator who faced difficulties onsite and does not understand the system and how to troubleshoot it will quickly bring frustration upon themselves, their company and clients. It is important that each operator is properly trained, is able to spend consistent time with the equipment and the software, and whenever possible operate of the equipment under the guidance of a mentor "MIP specialist."

7.0 MIP Field Operation

1. Power on the generator.
2. Open the gas cylinders that will be used for the MIP system (i.e. nitrogen, hydrogen, air, etc.).
3. Power on the GC and detectors and allow them to warm up (min. 20 minutes) to set temperature.
4. Power on the MIP controller, field instrument and laptop computer.
5. Check the trunkline supply and return flows of the system and MIP pressure. Compare these numbers to previous work.
6. Start the Acquisition software and start a new log.
7. Perform the chemical response test (Section 5.1.2) and record the height of the peak response and the trip time into a field notebook. Refer to Figure 5.4 and Appendix I and III.
8. Complete the EC dipole test (Section 5.3) and finish setting up the log.
9. Record the system parameters in a field notebook at this time (i.e. flow, pressure, trip time, detector baseline voltages).
10. Connect the stringpot cable to the stringpot and the stringpot wire to the weight located on the probe foot and pull keeper pin so the weight will drop to the ground.

NOTE: Do not allow the stringpot cable to snap back into the stringpot housing at a high rate of speed. This will ultimately damage the stringpot transducer.

11. Place the drive cushion onto the probing machine head.
12. Place a slotted drive cap to the MIP drive head.
13. Place the rod wiper on the ground and insert the point of the MIP probe into rod wiper opening.
14. Start the HPT water flow if running MiHPT.

    Note: It is important that there is always water flowing when the probe is moving to avoid soil particles from moving through and plugging up the screen.
15. Align the probe exactly straight and advance the probe to the starting depth: MIP membrane even with the ground surface.
16. Click the trigger button in the lower right hand corner of computer screen. (The Trigger label will flash and the background will change from yellow to green).
17. Standard advancement the probe is at a rate of 1ft/min meaning: advance 1 ft (30 cm) in 15 seconds and then hold at depth for 45 seconds, then advance to the next depth interval (1 foot) over 15 seconds and wait for 45 seconds. Do this until the predetermined log depth or until refusal is attained.

Advancement the MIP probe can be performed using a continuous push method with no stopping intervals which may be desirable in source level contamination. Data collected by this method will result in higher detection limits and is not directly comparable to data collected by the standard advancement method previously discussed.

NOTE: If the there is a loss in MIP pressure or temperature during the logging process, stop and evaluate the problem using the troubleshooting guide located in Appendix II.

NOTE: Refusal is attained when it takes longer than 1 minutes of continuous hammering to advance the probe one foot. This is the maximum time to reach one foot of probe travel.

18. Perform an HPT dissipation test (Section 7.1) in a zone of higher permeability indicated by lower HPT pressure if you are operating the MiHPT probe.
19. When the MIP log is complete, turn the trigger off and slowly return the stringpot cable into the stringpot housing.
20. Turn off the heater switch to the probe during tool string retraction so no as few contaminants as possible are diffused through the membrane and into the trunkline during retraction.
21. Raise the probe foot of the direct push machines foot assembly and place the rod wiper weldment under the foot assembly to keep it in place during rod retraction.
22. Pull the probe rod string using either the Geoprobe® rod grip pull system or a slotted pull cap.
23. When the MIP probe reaches the surface, clean the probe and membrane well with a detergent/water mix and rinse off well.
24. Now turn the probe heat back on to back off the membrane. Make sure the probe membrane and trunkline are clean of contaminants and the detector baselines are stable prior to running a post log response test. View the detector activity in the response test screen.
25. When the baselines are stable run a post log response test. These response test results should be written down in the field notes and compared to the initial test. This system check ensures the data for that log is valid.
26. When using the FI6000, the data will be saved into your designated folder on your laptop in a compact .zip file. Data from the MIP log can now be graphed and printed using the DI-Viewer software (Appendix IV).

7.1 Performing an HPT Dissipation Test

At least one dissipation test must be performed in order to calculate the static water level and estimated K readings from the HPT log. Dissipation tests need to be performed below the water table and are best in zones of high permeability where the injection pressure can dissipate off quickly once the flow is shut off. The following are the steps for running an HPT Dissipation test.

1. Stop in a zone of higher permeability which is indicated by lower HPT inject pressure.
2. Switch the DI Acquisition display view from the depth screen to the time screen by pressing the F10 key (F9 and F10 toggle between the depth and time screen of the acquisition software).

3. The screen will be grayed out which means that the data up to that point has not been saved. Select “Start Dissipation Test” which will turn the screen from gray to a white background indicating that you are now saving the time data.
   Now shut the pump switch off and when the line pressure reaches zero, turn the flow valve off.
4. The HPT Pressure will begin to drop (dissipate the hydrostatic increase) and allow it to stabilize so very little visible drop in pressure is seen. When the pressure has fully dissipated turn the flow valve and the pump switch back on. When the flow and pressure are reestablished select “End Dissipation test.”
5. Select F9 to return to the depth screen and advancing the tool into the ground.

Note: Performing a dissipation test in zones of higher permeability may only take 60 seconds or so but if the HPT pressure was higher to start with it may take a long time up to several hours to dissipate off to equilibrium. This is why targeting the most permeable zone to perform the dissipation tests is most desirable.

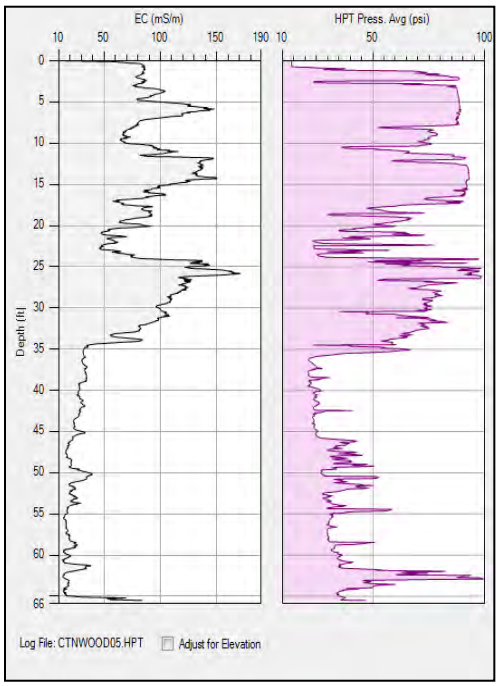


Figure 7.1: EC and HPT Pressure Graphs

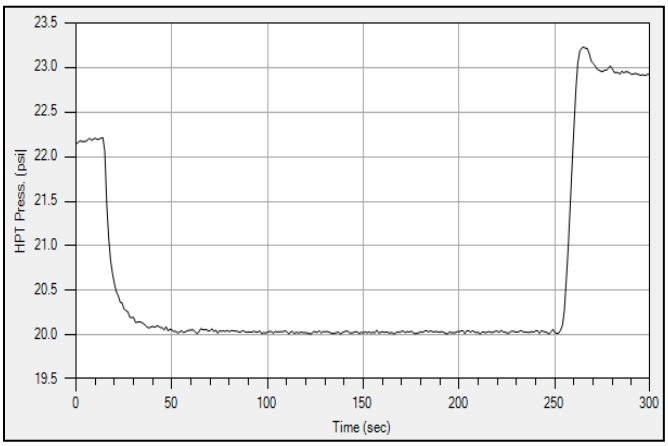


Figure 7.2: HPT Dissipation Test Screen.

The dissipation test shown in Figure 7.2 was performed in the lower pressure zones located at 39.5’ of the log shown in Figure 7.1. With HPT sands and gravels are indicated by lower injection pressure which is primarily seen below 35’ in the above log. The dissipation test in Figure 7.2 shows a higher pressure at the start of the test which falls off which is a result of shutting off the water flow. A good dissipation test will run for a period of time approximately 30-60seconds at a stabilized pressure and then turn the water flow back on during the saved log.

7.2 Detector Gain/Software Attenuation Changes

While mapping volatile contaminants with the MIP system operators commonly encounter highly contaminated/free product zones that can result in the detector signal climbing to the point of saturation or "flat lining." This occurs because the GC or detector system has a limited signal output range. What that range is varies depending upon the GC model or detector controller. Typical signal out limits for are 0-5VDC for SRI and Shimadzu GC models and 0-1VDC for HP/Agilent GCs and the OI XSD. The attenuation settings (software multipliers) for SRI and Shimadzu GCs and the XSD are based on a 10^x multiplication factor. The attenuation settings for detectors operated through an HP GC are based on a 2^x multiplication scale x = HP GC Range with the sum being the corresponding attenuation for the MIP software (Table 7.1).

As the probe is being advanced into higher concentration petroleum hydrocarbon soils the operator, if using an SRI GC, will want/need to adjust the GC gain on the PID and probably the FID from a gain setting of high to medium which takes those detector signals and divides them by a factor of 10 (Table 7.1). This reduction in the signal can be seen in the software both in the digital display as well as on the time graph. After the signal has been reduced the operator will need to select the attenuation tab/F5 in the DI Acquisition software and input a 10x multiplier in for the PID and the FID if both gain switches were changes to the medium setting.

If the operator chooses to go back to the highest sensitivity on those detectors after passing through the high contamination zone they need to first remove the multiplier in the software (F5) and then change the gain setting from medium to high on the GC – removing the signal divider. If either of these is performed in reverse fashion the log will see a very larger false positive peak because the signal is multiplied up without having a signal divider in place. The operator always wants to add the signal divider in first as they go into higher reading soils and remove it last as they come out of them.

Gain/Attenuation Settings on the GC detectors and the DI Acquisition software

HP GC* Range	DI Acq. Attenuation		SRI GC Gain	XSD Gain	DI Acq. Attenuation
0	1		High	High	1
1	2		Medium	Medium	10
2	4		Low	Low	100
3	8				

Table 7.1: GC gain/range settings and associated software multipliers.

*- The detectors on the HP GC can have attenuation settings up to a range of 7 on the GC corresponding to an acquisition software multiplication value of 128.

8.0 Replacing a Membrane on the MIP Probe

A probe membrane is considered in good working condition as long as two requirements are met:

1. Adequate signal response is achieved during the chemical response tests to see the required detection limits.
2. The difference between the supply and return flow has not increased by more than 3mL/min from the original settings. (A digital or bubble flow meter should be kept with the system at all times).

If either one of these requirements are not met, a new membrane must be installed as follows.

1. Turn the heater off and allow the block to cool to less than 50° C on the control panel readout.
2. Clean the entire heating block with water and a clean rag to remove any debris.
3. Dry the block completely before proceeding.
4. Remove the membrane using the membrane wrench (Fig. 8.1). Keep the wrench parallel to the probe while removing the membrane to ensure proper engagement with socket head cap screw.

NOTE: Do <u>not</u> leave the membrane cavity open for extended periods. Debris can become lodged in the gas openings in the plug.

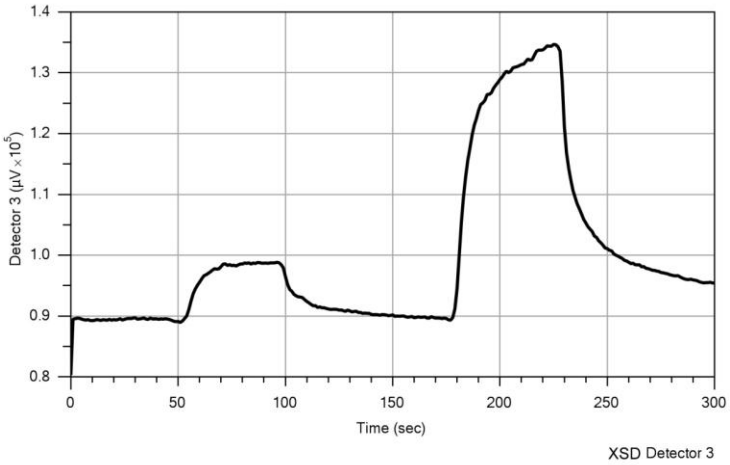
5. Remove and discard the copper washer as shown in Figure 8.2. Each new membrane is accompanied by a new copper washer. **Do not reuse the copper washer**.
6. Clean the inside of the membrane socket with a q-tip and methanol removing dirt and debris that will be present.
7. Insert the new copper washer around the brass plug making sure that it sits flat on the surface of the block.
8. Install the new membrane by threading it into the socket. Thread the membrane into the socket by hand, do not use the membrane wrench until the membrane is nearly all the way threaded. Use the membrane wrench to tighten the membrane to a snug fit. Do not over-tighten.
9. Turn the carrier gas on and leave the heater off. Apply soapy water to the membrane and surrounding area to check for leaks. If bubbles form in the water around the edges of the membrane or in the wrench holes use the membrane wrench to further tighten the membrane.
10. Use a flow meter to check carrier flow. The difference between the supply flow from the MP6505 and the return flow from the trunkline should be less than 3ml/min. Record the values in a field notebook.

Figure 8.1: Unthread the membrane from the probe block.

Figure 8.2: Remove and discard the copper washer.

APPENDIX I

Typical Response Test Data

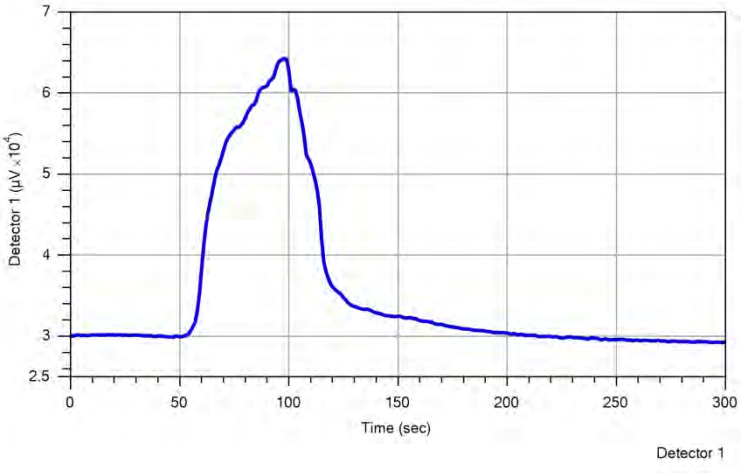


System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
XSD Temperature of 1,100°C

System Response:
1ppm – 9,000µV
5ppm- 45,000µV

Figure 1: Chemical Response Test: TCE 1 & 5ppm on XSD



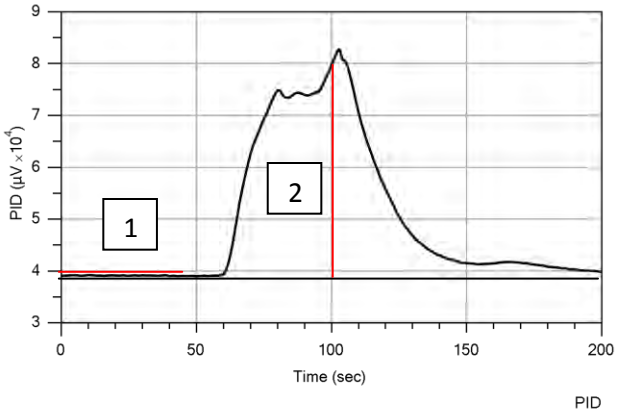
System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
PID Lamp intensity

System Response: 5ppm- 35,000µV

7,000µV/1ppm

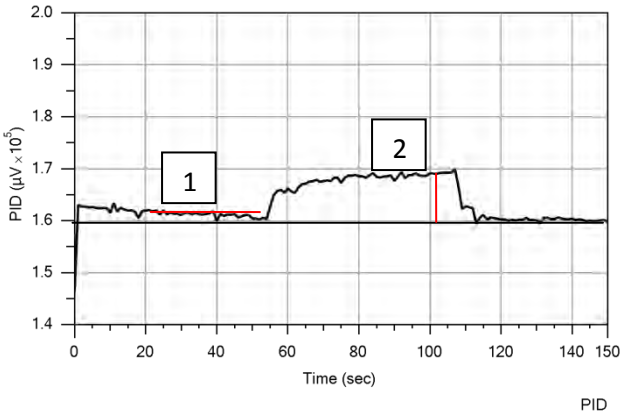
Figure 2: Chemical Response Test: Benzene 5ppm on PID



Response test - PID	5ppm Benzene
Response magnitude (2)	~40,000µV
Response/1ppm	~8,000µV
Baseline noise (1)	<500µV

Parameters: 150'TL/40ml/min flow/12PSI

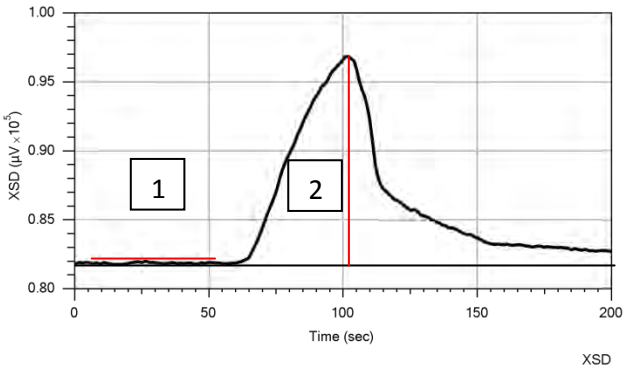
Acceptable response test. Response to baseline noise ratio is >5:1 at 1ppm



Response test - PID	1ppm Benzene
Response magnitude (2)	~8,000µV
Baseline noise (1)	~2,000µV

Parameters: 150'TL/39ml/min flow/12PSI

Not Acceptable response test
Response to baseline noise ratio is <5:1 for 1ppm Benzene
Quick Fix: Lowering carrier flow rate to 25-30ml/min will improve signal response 50% or more.



Response test - XSD	2.5ppm TCE
Response magnitude	15,000µV
Response/1ppm	6,000µV
Baseline noise	<300µV

Parameters: 150'TL/40ml/min flow/11.4PSI
Acceptable response test, Response to baseline noise ratio >5:1 for 1ppm TCE

1. Baseline noise is the amount of variation in baseline signal over a given time.
2. Signal response is the amount of rise in baseline over the stable baseline level.

APPENDIX II

Troubleshooting Guide

Loss of Pressure 1-2 PSI

- If the pressure loss has been gradual, and your MIP controller has a flow sensor check to see if the MIP supply flow has gradually dropped over the course of the log. This can happen due to the control box warming up and will be indicated by a gradual drop of both MIP pressure and flow. To resolve this increase the mass flow controller to bring the supply flow back to its original set point.
- Punctured membrane: Are there any obvious holes in the membrane with bubbles streaming out of them? Replace membrane.
- Membrane leaking out of the face – heavy frothing of bubbles on membrane face but no obvious punctures in membrane. **With the heat off**, place your thumb over the membrane**,** if the pressure goes back up to the gas pressure prior to the boring the pressure and flow loss is due to a leak in at the membrane face. Replace the membrane.
- Swagelok fitting connecting one of the trunkline gas lines to stainless steel gas line of the probe is loose. Check with soapy water, if bubbles build, fix by slowly tighten the gas line 1/16" nut to the probe.
- Examine for cuts, kinks & cracks in the length of the observable gas line. Expect to see bubbling when MEOH or soapy water is placed on it. Cut gas line prior to this and replace nut and ferrule and reconnect onto the probes steel gas line connection.
- Broken gas line somewhere else up the trunkline. Confirmed when trunkline connections are removed from the probe and close coupled. The carrier gas supply and return should be within 2ml/min, if it is >5ml/min first check with soapy water at the connecting nuts and exposed gas line then look for cuts in throughout the trunkline and see if they will show bubbles with soapy water placed on them. If this is seen you will likely need to change the trunkline.

Loss of Pressure >5 PSI

- Large puncture in membrane. Either visible puncture or observable streaming bubble when soapy water or methanol placed on membrane. Replace membrane.
- Loosen the 1/16" Swagelok nut on gas line. Check and carefully tighten.
- Broken gas line in the probe. Compare the supply versus return flow values (should $\leq$ 2/ml/min) of trunkline connected with the probe and with a close coupled trunkline. If close coupled supply/return flow is good but connected to the probe shows a big leak, there is a break is in the probe. This may be seen with soapy water placed on the edges of the heater block or on the top of the probe where the connections come out. If this produces bubbles it confirms a broken internal line or connection point. Replace the probe.

DI Acquisition - Flash Warnings:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – MIP pressure out of Range - to the operator if the probe pressure (PSI) changes over 1 PSI from the initial starting MIP pressure of the log. This alerts the operator that something in the system has changed and the operator can take the necessary precautions for a punctured membrane, broken gasline or a plug in the system.

Increase in Pressure (clearing a blockage)

- After setting the mass flow, an increase of more than 3 PSI over the original set pressure indicates a potential blockage, especially if you can verify that the pressure first dropped a 2-5 PSI prior to rising toward 20PSI.
  - Shut off the Nitrogen carrier gas flow ASAP. Do this by turning off the black regulator knob on the MIP controller or removing the carrier gas supply line from the breakout panel or the back of the MIP controller.
  - Remove the tools from the ground.
  - Look for a hole in the membrane and water or dirt got into the up-hole gas line just behind the membrane.
  - Remove connection tube and membrane.
  - Remove the trunkline gas lines from the top of the probe. Take note of which one had the gas flow coming out because this is the line that will be plugged.
  - Look for any obvious particles in either holes behind the membrane or in the gas line at the top of the probe. If any are evident attempt to remove them.
  - Take the return gas line at the surface and connect it to the supply gas connection on the breakout panel or on the back of the MIP controller.
  - Place the probe end of this line into a jar of methanol to see if the line is clear which is evident by streaming bubbles. If there are no bubbles, increase the flow to try to expel the blockage. If this does not work you may need to cut back the trunkline.
  - To clear out the probe take a 5 ml plastic syringe (or a 3 foot section of Teflon/PEEK gas line will work) filled with methanol and attempt to inject through the plugged gas line at the top of the probe. If it clears it will shoot the methanol in an arcing stream out one of the ports in the plug that sits behind the membrane.
  - The probe must be dried of the methanol which can be accelerated by heating the probe. Don't reconnect the trunkline to the detectors until you are sure the blockage is clear and the methanol is out of the system.
  - If the blockage cannot be cleared a new probe or trunkline will need to be connected.

Blinking Temperature Light

- If the temperature light on the MP6505 begins blinking in an unreadable number, it means that there is an open thermocouple in the system.
  - To complete the log in progress, replace the thermocouple for the trunkline with a thermocouple wire and twist-tie the wires together. This will fool the system to thinking there is continuity of the thermocouple wire and allow you to finish a log. The probe will continually heat set up this way and if left on when out of the ground it will overheat. When the log is complete remove the tricked thermocouple and remove tools from the ground.

- When you have the probe out of the ground, replace the thermocouple as follows.
  - Remove the connection tube from the probe.
  - Check the crimp connections of the thermocouple wires from the trunkline to the probe.
    - If one of the crimp connections has broken then strip back the wire on both sides of the thermocouple – probe and trunkline ends and reconnect in a new crimp connection and see if the probe temperature comes back.
    - If the thermocouple connection is good, the thermocouple wire in the probe has likely broken. Cut off the crimp connections of the thermocouple wires between the probe and the trunkline Check the resistance between the red and yellow thermocouple wires coming out of the probe. A resistance reading of approximately 40ohms indicates that the thermocouple is good reconnect. If they are open (O.L.) or mega ohms then the leads are broken on the thermocouple. Replace the thermocouple.
- To check the trunkline thermocouple wires, measure each wire from top to bottom. The resistances will be different between the two colored wires but should be somewhere approximately 50 ohms – 150ohms for the length of the trunkline. The resistances will also increase with an increase in trunkline length.
  - If they are open (no resistance) then there is a break in the trunkline. Replace the trunkline.

Blinking temperature readout or
Spiking in the Pressure and/or Temperature Data

- If spikes show up in the temperature or pressure data especially when related to hammer strikes it is likely an intermittent break in the thermocouple connection. Spiking of the temperature may reach single point readings of 250^{o}C in the data but may not be visible when watching the temperature display on the MIP controller.
  - When you check the resistance between the two thermocouple wires they may check out at approximately 40 ohms, however there likely is an intermittent break in the wire.
  - Replacing the thermocouple should eliminate the pressure and temperature data spikes.

Probe Not Reaching Temperature

- If the heater light is on but the temperature seems low (<100^{o}C with a set point of 120^{o}C) a heater may have broken in the probe.
  - Check the resistance of the heater wires.
    - If a heater is broken the resistance will be over 40 ohms. The probe needs to be replaced.
    - Two good heaters will read approximately 22 ohms on the MP6520, MP8520 and MK6530.
      - Check to see if the thermocouple has pulled of few inches out of the probe.
        - If the thermocouple duct has broken and pulled back away from the probe, the probe will need to be replaced and rebuilt.
        - A thermocouple can unscrew and vibrated loose out of the thermocouple duct connection if it is not secured with shrink tubing or electrical tape. Reseated back into the leur-lock connection and secure. When the thermocouple pulls away from the probe it measures the probe temperature in the wrong location.

Flash Warning:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – Temperature out of Range - to the operator if the temperature goes outside of a set range from the setpoint temperature of 121^{o}C. This alerts the operator that something in the system has failed and the operator can take the necessary precautions for a broken probe heater or thermocouple problem.

System explanations and warnings

MIP Flow

MIP flow is the carrier gas flow set by the MIP controller. This flow is supplying carrier gas to the trunkline and probe and is typically set to approximately 42ml/min. This parameter may be monitored by the DI-Acquisition system if the operator has the necessary components in their MIP Controller. The return flow, or Flow-R, is the flow coming back to the GC up the return gas line. Flow-S and Flow-R should be within 3-4ml/min and are usually much closer.

MIP Pressure

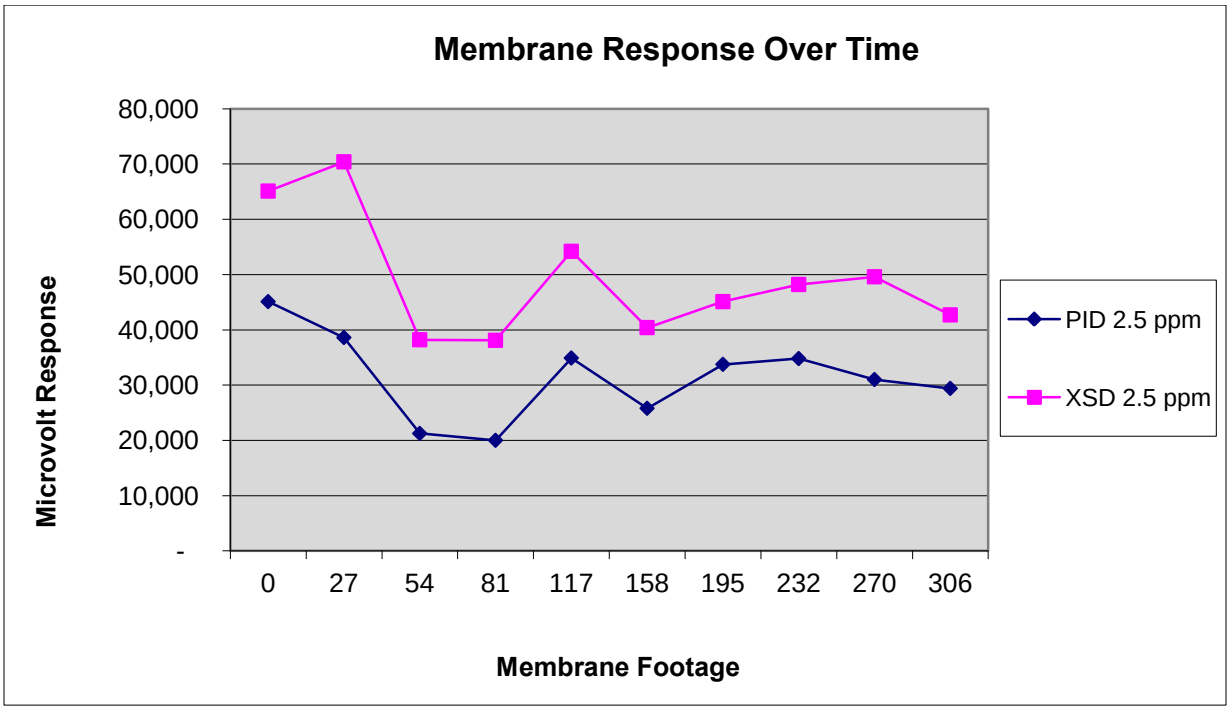
The MIP pressure is the back pressure of the carrier gas as it moves through the trunkline and probe. This is monitored digitally on the DI-Acquisition screen as well as by an analog pressure gauge on the front of the MIP controller. The MIP pressure is directly related to the MIP return flow (Flow-R) and the length of the trunkline. If the MIP pressure falls, the return flow has also dropped, if the MIP flow (Flow-S) has remained the same then there is likely a punctured membrane of problem with the gas lines.

APPENDIX III

Membrane Performance Control Charts

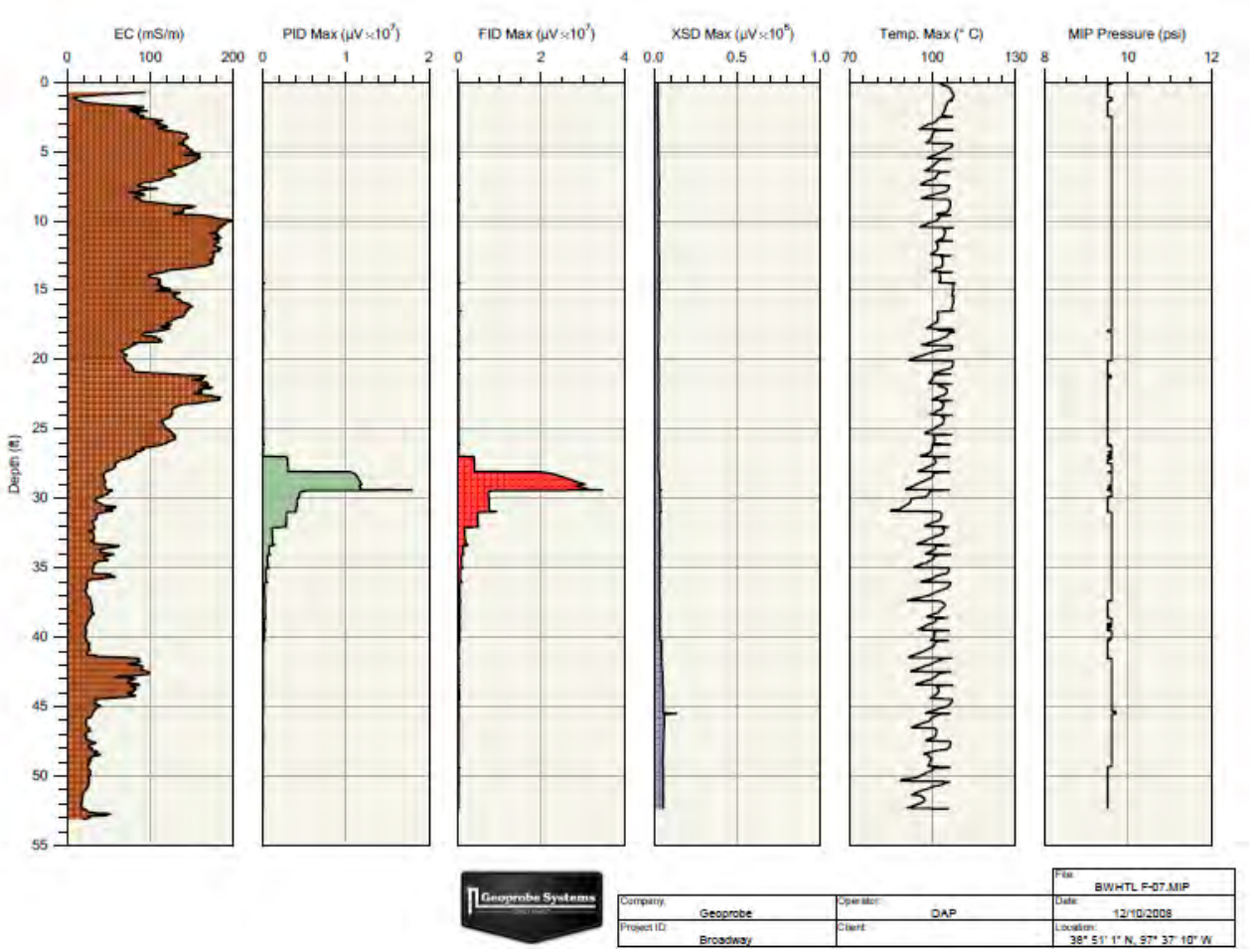
Response Tests using TCE

Pre/Post Log Response Test	Log ID:	PID Response 2.5ppm	XSD Response 2.5ppm	Log Footage	Membrane Footage
Pre-Log	MIP01	45,100	65,100	27	0
Pre-Log	MIP02	38,600	70,400	27	27
Pre-Log	MIP03	21,250	38,200	27	54
Pre-Log	MIP04	20,000	38,100	36	81
Pre-Log	MIP05	34,900	54,200	41	117
Pre-Log	MIP06	25,800	40,400	37	158
Pre-Log	MIP07	33.750	45,100	37	195
Pre-Log	MIP08	34,800	48,200	37	232
Pre-Log	MIP09	31,000	49,600	36	270
Post-Log	MIP09	29,400	42,700		306



APPENDIX IV

Sample Logs and Interpretation



Here is a MIP log with the graphs left to right: electrical conductivity, detectors (PID, FID and XSD), probe temperature and trunkline carrier gas pressure.

The above log shows contamination from 27ft to 33ft bgs. The main detector response is on the PID and FID with minimal response on the XSD (Halogen Specific Detector). This indicates that the main contaminant would not contain halogenated (Cl-, Br-, Fl-) atoms, but would likely be hydrocarbon based. The contaminants are present in the lower electrical conductivity formations which typically indicate courser grained formations of higher permeability. The temperature deflections of the MIP block heater are indicative of the probe heat cycling and the trunkline carrier gas maintains a constant stable pressure which indicates no leak or plug issues occurred with the gas line or membrane during the log.

Detector Interpretation

Standard MIP systems are able to identify compound families and determine general compound classes. The identification of individual compounds is not possible. Standard MIP systems have a continuous carrier gas flow that is brought to the detectors from the down-hole probe. To be able to effectively speciate (determine specific contaminant chemicals) the operator would need a highly modified system in place. The carrier gas stream would need to be run through a mass spectrometer or trapped and run a secondary GC onsite.

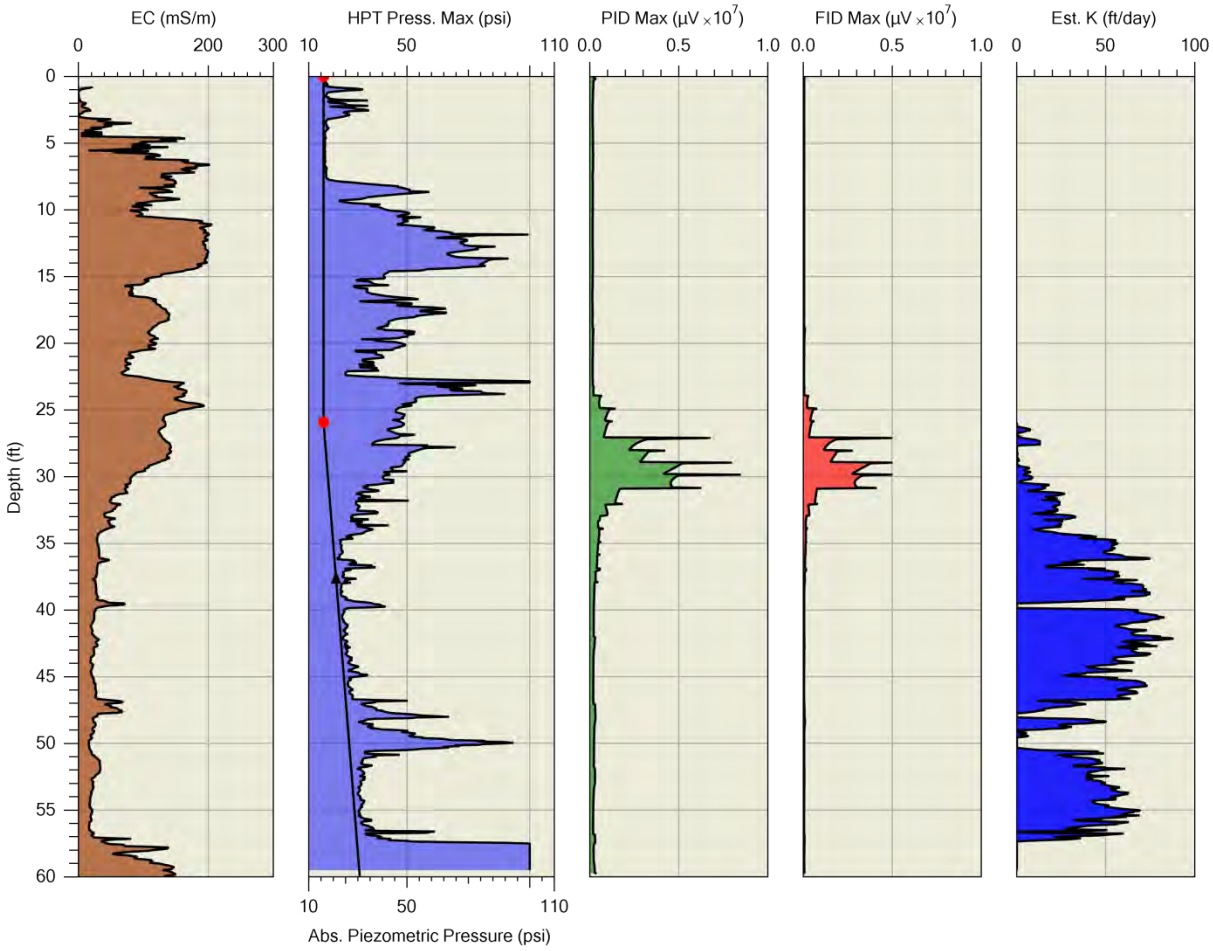
Typical standard MIP configurations use 3 gas phase detectors: a photo-ionization detector (PID), flame-ionization (FID) and a halogen specific detector (XSD). The PID responds to compounds which have an ionization potential $\leq$ electron voltage of the PID bulb. These compounds include both chlorinated and non-chlorinated hydrocarbons. A typical PID bulb has a 10.6eV lamp. The FID will respond when organic compounds (anything containing carbon) are present in the carrier gas stream in high enough concentration burn up in the flame which increases the flames ionization voltage. The XSD responds only to halogenated compounds which are made up of chlorinated (most typical halogen environmental contaminant), brominated and fluorinated compounds. Based upon which detector or detector series a contaminant responds on, we can determine if the contaminants are halogenated or petroleum based.

Petroleum hydrocarbons will respond on the PID and FID but not on the XSD. Fresh gasoline primarily contains aromatic hydrocarbons such as benzene, toluene, ethyl benzene and xylenes, which respond strongly on a photo-ionization detector (PID) and not so well on the FID. As gasoline breaks down or weathers the molecular structure changes from primarily aromatic to mainly straight chain hydrocarbons (single bonded hydrocarbons). Straight chain hydrocarbons typically do not show up on the PID do having a higher ionization potential but will respond on a flame ionization detector (FID). Weathered petroleum will still have a decent signal on the PID but may show a stronger FID signal.

Chlorinated compounds such as trichloroethylene and perchloroethylene are detected by the XSD and PID and respond in a similar profile. This is typical of the common double bonded chlorinated compounds seen in the subsurface which have an ionization potential that the PID can see. Chlorinated compounds without multiple bonds such as chloroform, methylene chloride and 1,1,1,-trichloroethane have an ionization potential higher than the PID electron voltage which results in a solid response on the XSD but will not show up on the PID.

The only sure way of determining contaminant concentration from MIP responses is to take confirmation soil and/or groundwater samples for laboratory analysis. After obtaining the results the actual concentrations can be compared to the MIP detector responses and concentrations may be estimated across the site.

MiHPT Log Example– Combined MIP & HPT



The addition of the HPT sensor to the MIP detectors and EC has provided valuable information to the subsurface lithogy encountered by the MIP operator. The above log shows graphs left to right: electrical conductivity, HPT injection pressure with the absolute piezometric pressure profile on the secondary axis, detectors (PID and FID) and estimated hydraulic conductivity (K).

The above log shows contamination from 24ft to 31ft bgs both on the PID & FID at similar magnitudes which is likely from petroleum hydrocarbons but without showing the XSD we cannot tell for sure that there are no halogenated (Cl-, Br-, Fl-) compounds present. The contaminants are present in the higher electrical conductivity and HPT pressure formations which indicate finer grained formations of lower permeability. The second graph with the Absolute Peizometric profile graph has a triangle on the increasing line at approximately 37 feet which indicates that an HPT dissipation test was performed at that depth. By taking the hydrostatic pressure at that interval and subtracting off the weight of water (0.445psi/ft) until the atmospheric pressure (calculated in the pre log HPT reference test) we can see the static water table is approximately 26 feet indicated by the red dot. Estimated hydraulic conductivity (K) is shown as the final graph which is a relationship between HPT injection pressure and HPT flow.

APPENDIX V

GC1000 Configuration and Operating Parameters

Figure 1: GC1000: SRI 310GC with XSD Controller

GC1000 Configuration

SRI310 GC with PID, FID & OI Analytical XSD
(all standalone detectors)

Flows:

TL Carrier (N_2): 40ml/min
Detector split 60:40 – 24ml/min-XSD
16ml/min-FID

Nafion Dryer (installed in GC Oven)
80ml/min (2x carrier flow rate)

A built in air compressor is split underneath the GC between the XSD & FID. The XSD & FID air supply is controlled through the GC air pressure screw control on front of GC and with different air line sizes and lengths to provide 250ml/min to the FID and 30 ml/min to the XSD.

Detectors front of GC to back: XSD, FID & PID

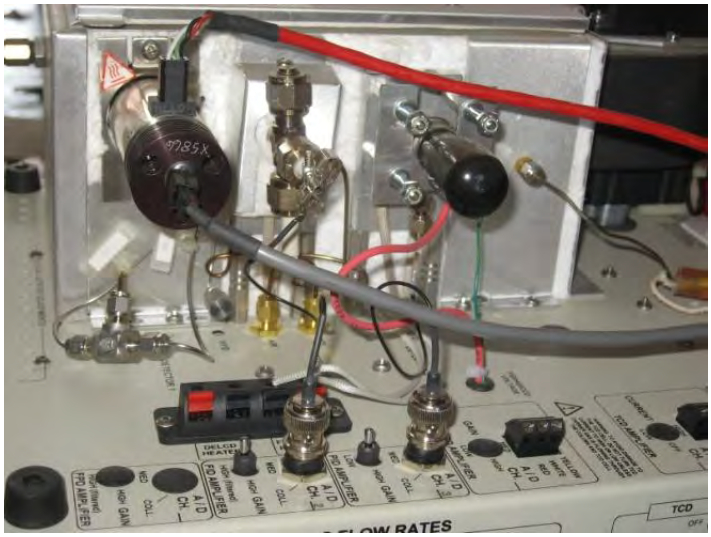


Figure 2: GC Detectors – left to right - XSD, FID, PID

SRI 310 GC Detector 1 position – XSD
(not controlled by GC)

SRI 310 GC Detector 2 position – FID

SRI 310 GC Detector 3 position – PID

Nafion dryer installed inside GC oven

GC Oven set to 85°C – 130°C max temp.

Flow comes into the GC oven via a 1/16" bulkhead fitting located in the 4th detector position furthest back (upper right inside oven) behind the PID detector. The trunkline will connect to this bulkhead and a 1/16" stainless steel line transports flow into the Nafion dryer. Silco steel takes this to the PID lamp which is inserted up to the lamp and backed off a 1/16" and tightened. A 1/16" stainless steel line brings it back into the GC oven where it is split between the FID and XSD and sent to them via a silco-steel line to the XSD and a stainless steel line to the FID.

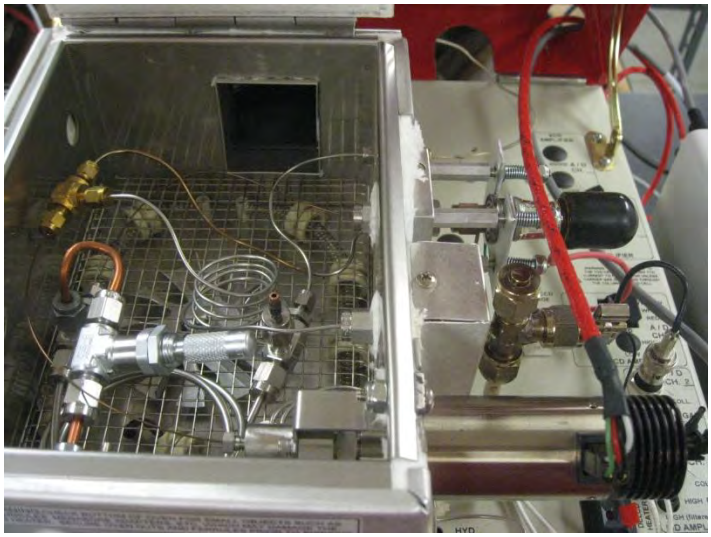


Figure 3: GC Oven Configuration

Detector Operating Parameters:

PID:

- MIP Carrier Flow (N_2) – 100% - 40ml/min
- Carrier return back into oven split between XSD & FID
- Detector Temperature setting – 150°C
- PID current 70 (0.70ma)

FID:

- Carrier N_2 MIP effluent – 40% - 16ml/min
- Hydrogen – 25ml/min
- AIR – 250ml/min
- Detector Temperature setting – 250°C
- FID igniter set at -600 (6.0V)

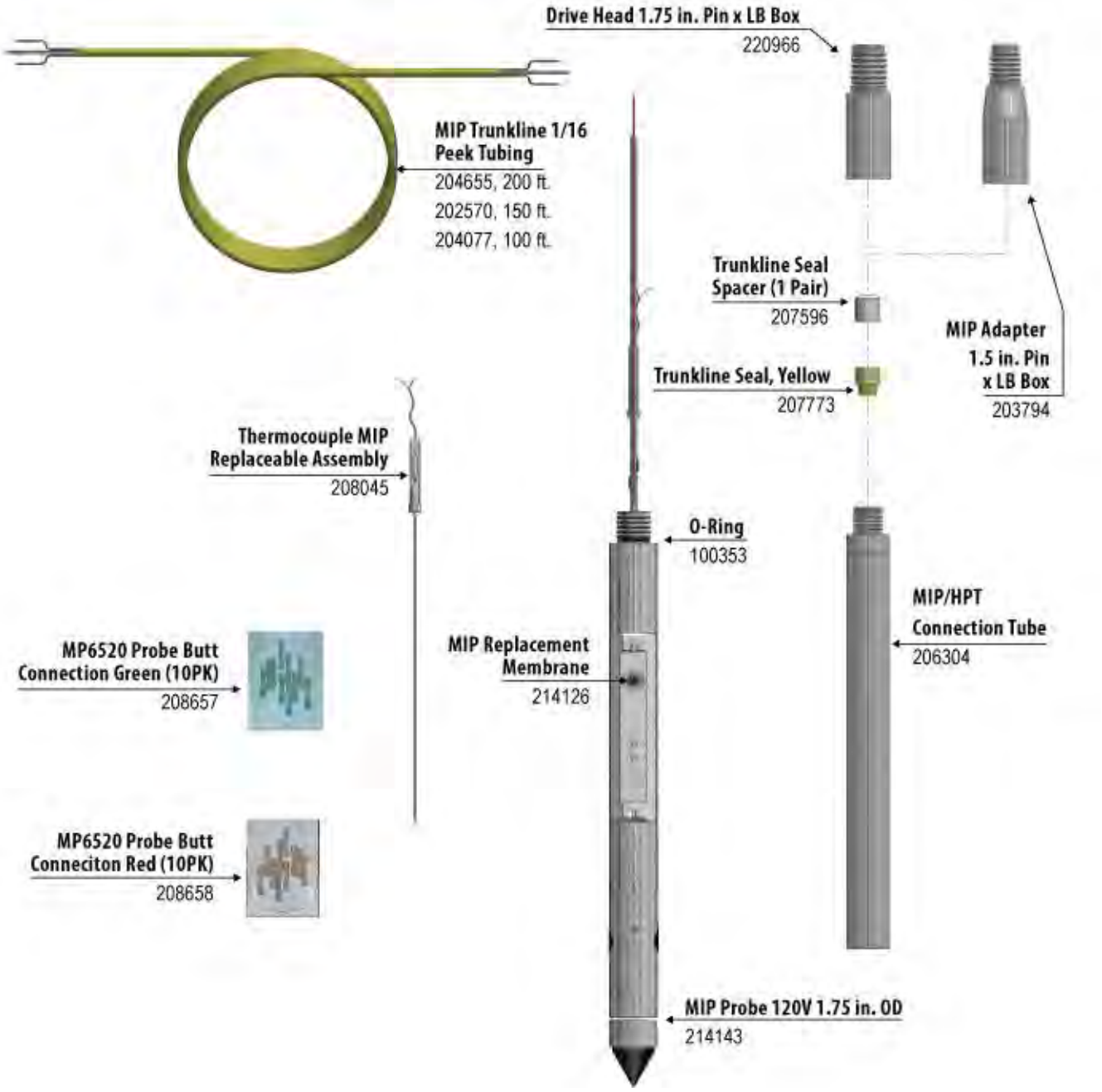
XSD:

- Carrier N_2 MIP effluent – 60% - 24ml/min
- Air – 30ml/min (split 50:50 wall & jet input of XSD)
- Detector Temperature setting – 1,100°C

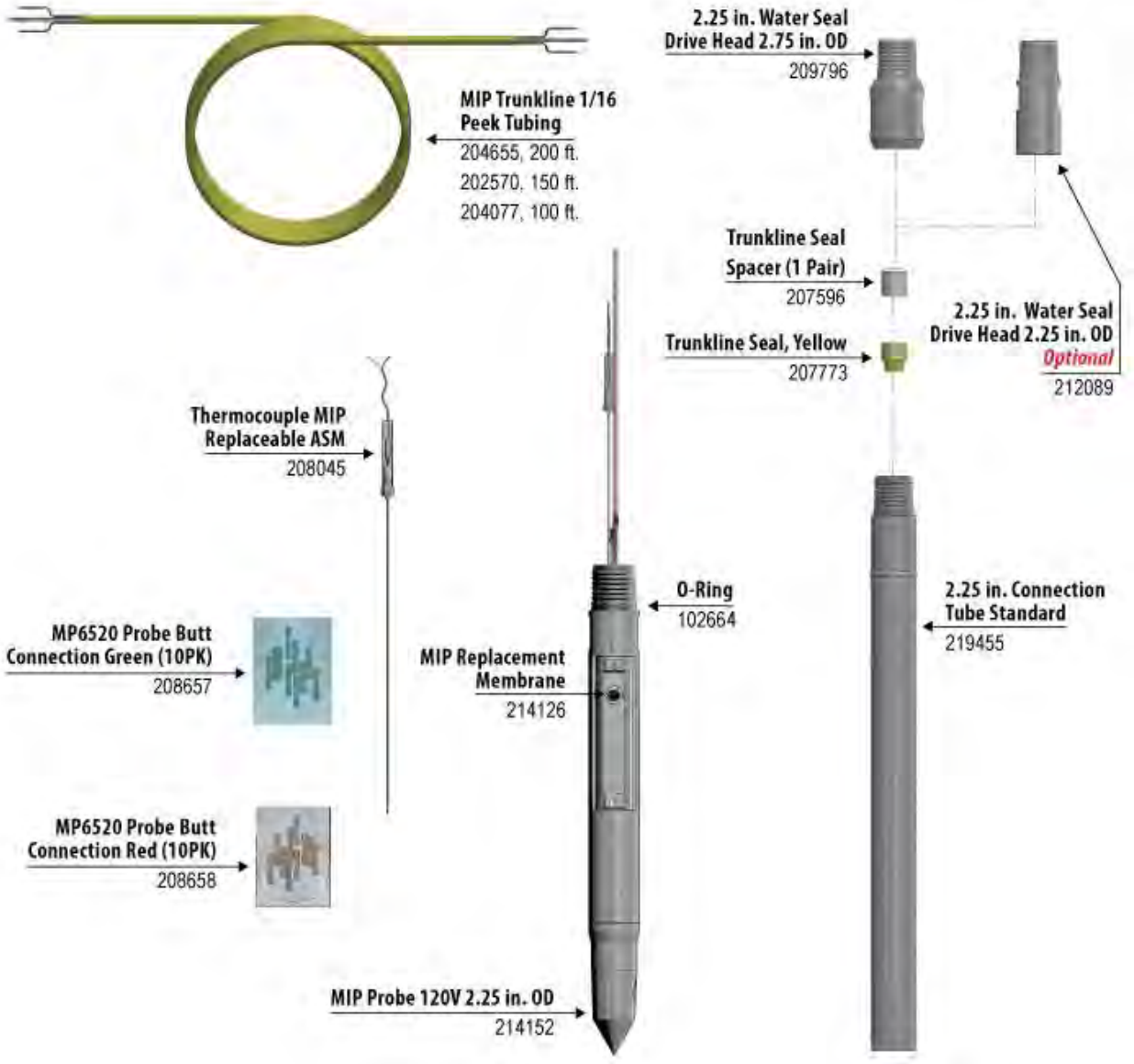
APPENDIX VI

MIP Tool Configurations

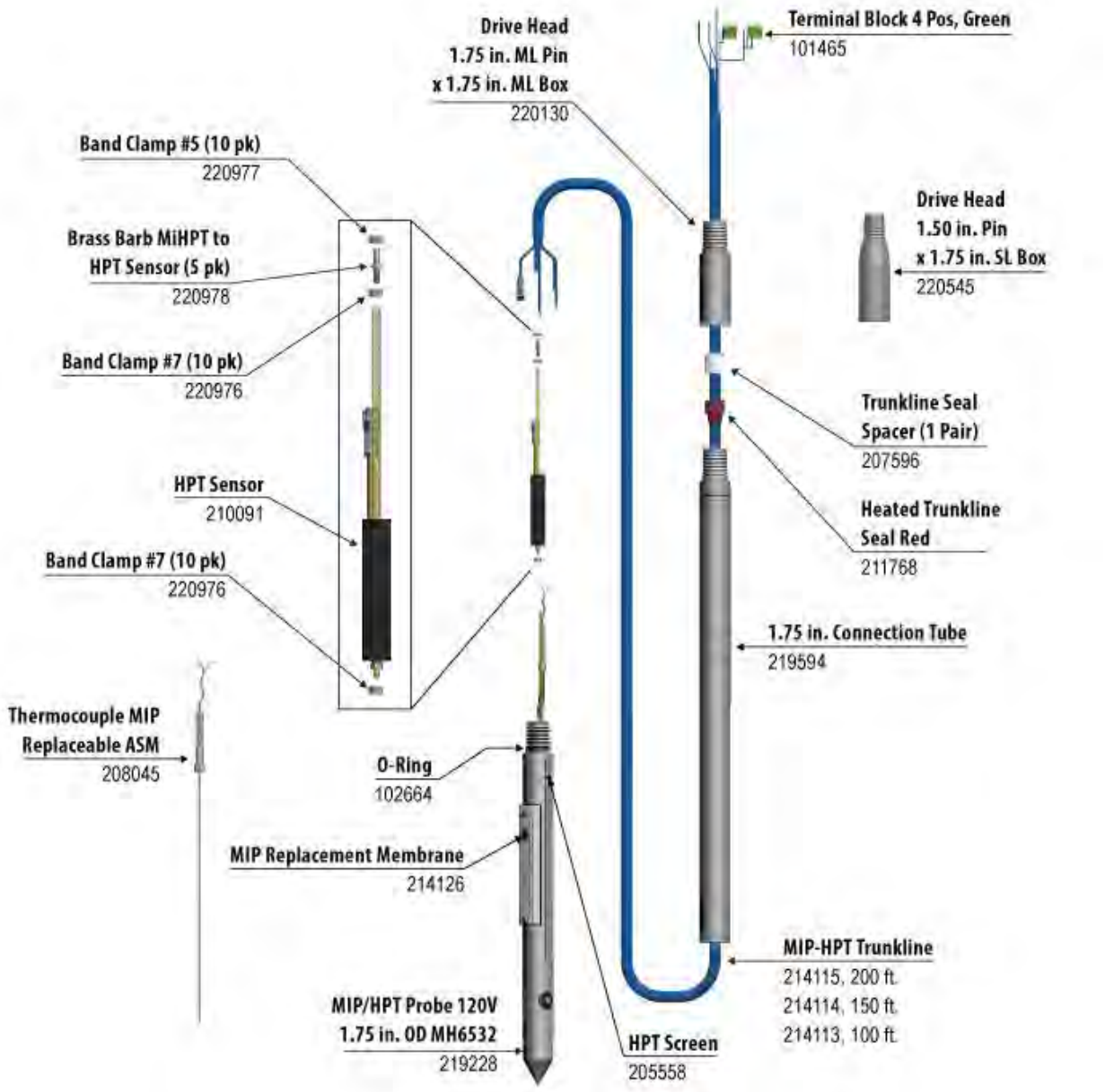
MIP - MP6520 (1.5 in. / 1.75 in. system)



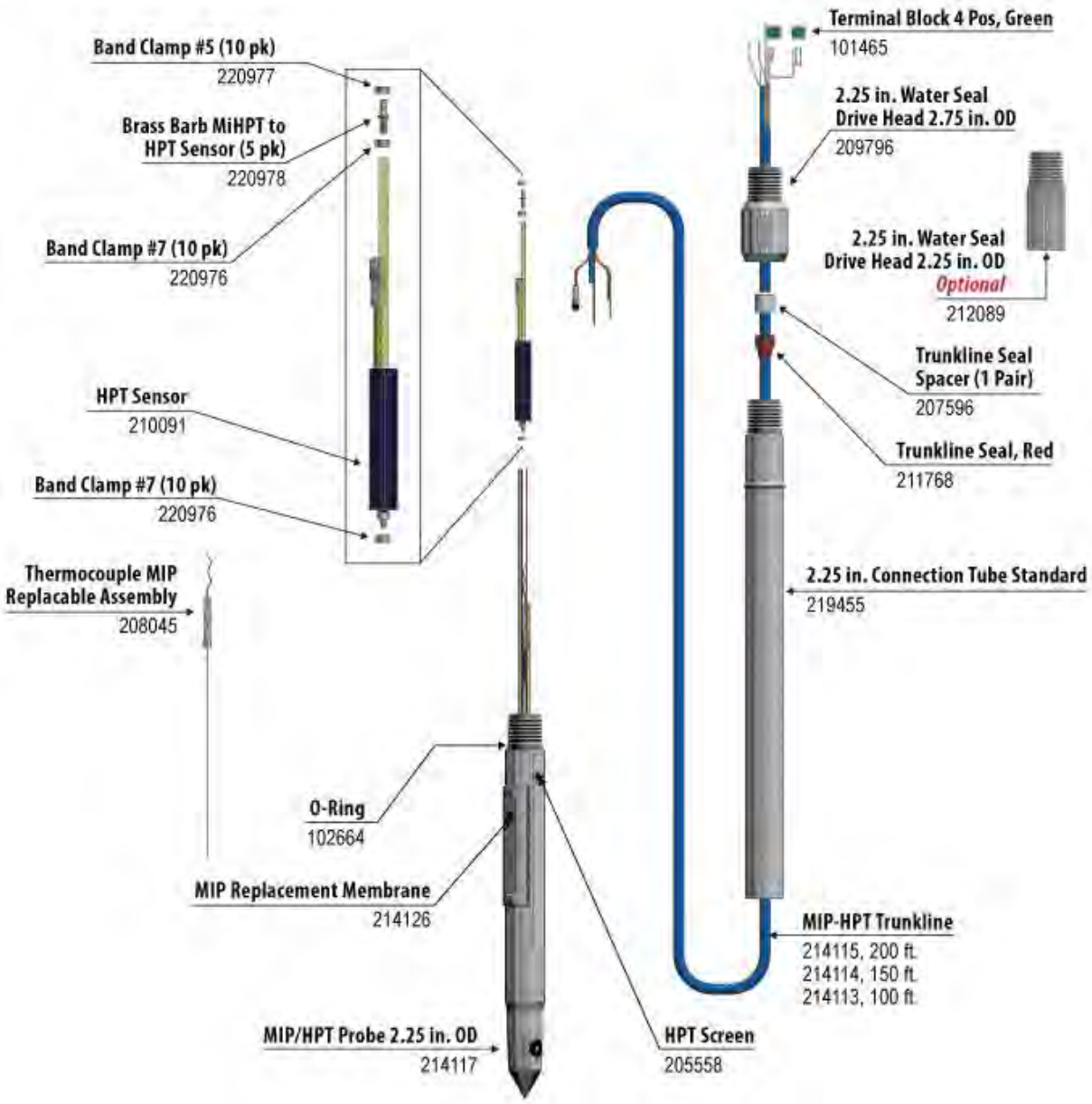
MIP – MP8520 (2.25 in. System)



MiHPT – MH6532 (1.5 in. / 1.75 in. system)



MiHPT – MH8530 (2.25 in. System)



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