SAMPLING AND ANALYSIS PLAN

SOIL VAPOR EXTRACTION SYSTEM
BIG MO AND FORMER BENZENE PIPELINE AREAS

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1.0 PROJECT DESCRIPTION

This Sampling and Analysis Plan (SAP) describes procedures for performing routine sampling and compliance monitoring as part of operation of the soil vapor extraction (SVE) remedy in the Big Mo and Former Benzene Pipeline treatment areas (**Figure 1**) at the W.G. Krummrich facility in Sauget, Illinois (site). Detailed monitoring information is presented in the Operation, Maintenance, and Monitoring (OM&M) Manual by XDD, LLC (XDD), dated June 2012.

1.1 SITE BACKGROUND

For a detailed site background and description of the overall system design, refer to the Soil Vapor Extraction Pilot Test Report (Pilot Test Report), dated November 2010; the Work Plan for Full-Scale Soil Vapor Extraction (Work Plan), dated November 2010; and the 100% Soil Vapor Extraction System Design (100% design), dated September 2011.

The SVE system is designed to treat unsaturated zone soil impacts in the Big Mo and Former Benzene Pipeline areas. Based on soil sampling conducted in the Big Mo and Former Benzene Pipeline areas, the primary constituent of concern (COC) is benzene. The extents of the Big Mo and Former Benzene Pipeline treatment areas are presented on **Figure 2**.

Three general soil layers are observed in the target treatment area – sandy fill/upper silty sands, intermediate silty clay, and lower silty sands. The sandy fill/upper silty sand layer generally extends from ground surface to approximately 10 feet below ground surface (ft bgs) and the lower silty sand layer is encountered below the intermediate silty clay layer (present between 5 and 12 ft bgs) to approximately 15 ft bgs. Refer to the Work Plan for comprehensive geological cross-sections of the Big Mo and Former Benzene Pipeline areas.

The target depths for the SVE treatment are shallow (5 to 10 ft bgs) and deep (10 to 15 ft bgs) intervals within the unsaturated portion of the Shallow Hydrogeologic Unit (SHU). The SVE system is not designed to treat the unsaturated zone intermediate silty clay layer or soils below the groundwater table. Due to fluctuations of groundwater levels, portions of the lower silty

sand layer will be saturated on a seasonal basis (typical groundwater level ranges from 10 ft bgs to greater than 15 ft bgs). As the water table fluctuates and the lower silty sand layer is exposed, the system will be reconfigured to target that interval.

1.2 SCOPE AND OBJECTIVE

Performance and process monitoring will be conducted during SVE system operation to evaluate overall COC mass reduction and system effectiveness. The specific scope and objectives of the sampling program are summarized below. Sampling and analysis procedures are described in detail in **Section 2.0** and summarized on **Tables 1 and 2**. Analytical sampling parameters are summarized on **Table 3**.

1.2.1 PERFORMANCE MONITORING:

The objective of performance monitoring is to monitor and assess the remedial performance of the SVE system. Performance monitoring includes:

1. SVE vapor stream monitoring: Performance monitoring data from the SVE vapor stream will be used to assess COC mass removal rates and calculate cumulative COC mass removed in the vapor phase. Monitoring will consist of:
    a. Measurement of COC concentrations in the SVE well field vapor stream.
    b. Measurement of the total SVE well field flowrate.
    c. Calculation of the COC mass removal rates for each monitoring event using the COC vapor concentration and SVE flowrate.
    d. Calculation of the cumulative volatile organic compound (VOC) mass removed using the average COC mass removal rate and the length of time elapsed between each monitoring event.

2. COC mass estimates: Soil sampling data will be used to assess reductions in soil concentration and soil COC mass during SVE system operation. As described in the Work Plan, soil data collected in 2009 and 2010, and the corresponding COC mass estimate will serve as the estimated COC mass baseline for the Big Mo and Former Benzene Pipeline Treatment areas. This prior work was completed as part of a site-wide

soil characterization project (refer to the "Soil Vapor Extraction Treatment Area Characterization Report" submitted to the United States Environmental Protection Agency in November 2010). Grab samples were selected for laboratory analysis from the depth intervals exhibiting elevated total VOC vapor concentrations (as measured using a field photoionization detector [PID]) for the purposes of COC characterization.

To satisfy the performance monitoring objectives, the soil sampling procedure will involve a multi-leveled approach:

- **Approach 1**: Discrete soil sampling for performance monitoring consistent with the approach used during the baseline characterization of the Big Mo and Former Benzene Pipeline areas[1]. This sampling method will monitor the system performance based on remediation of the treatment depths with the highest level of contamination.
- **Approach 2**: Using the same soil boring as Approach 1, additional samples will be collected at 2-foot discrete intervals from 3 ft bgs to 15 ft bgs, determined by the treatment interval(s) targeted during the previous year of operation.

Because the distribution of COCs within the soils are often heterogeneous, a single grab sample selected from a short soil interval exhibiting the highest field screening result (Approach 1) is typically not representative for mass estimation purposes. Approach 1 is expected to yield a value that may initially underestimate the performance of the SVE system, as areas with the most elevated soil concentrations can be the last to show significant improvement due to the mechanics of the pore volume exchanges and the potential for NAPL in the subsurface. By collecting soil samples over a consistent vertical interval (Approach 2), the bias that is introduced using screened grab samples is significantly reduced. Results from Approach 1 will be compared to historical data and mass estimates. Results from the Approach 2 will be used to calculate a more accurate estimate of the remaining benzene mass in the soil.

[1] Refer to the *SVE Treatment Area Characterization Report* by URS Corporation, submitted to USEPA in November 2012.

To calculate the remaining benzene mass annually, an average of the results may be used if the subset of borings sampled annually (a maximum of 15) are equally distributed throughout the treatment area. The sampling plan after the second year may be focused on areas that have shown sub-optimal performance relative to previous soil sampling results and vapor concentrations. Therefore, the use of an average to calculate the benzene mass may not be applicable for the entire treatment area and in these instances an estimate may be derived or the total mass calculation will be deferred until the next or final soil sampling event. The final sampling event will target all the borings used for delineation and mass estimation purposes. The SVE system is not designed to directly target the silty clay; however, samples will be collected from the intermediate silty clay layer during the final soil sampling event only to assess the overall level of COC mass remaining in this unit.

The procedure for estimating COC mass reduction on the soils will consist of the following steps:

a. Annual soil sampling will only be conducted within the treatment intervals that have been targeted by the SVE system during the prior year of operation. For example, if the SVE system resources are entirely focused on the deep treatment interval during a given year, then only the deep treatment interval will be sampled (i.e., groundwater elevation fluctuations can limit the availability of this interval for SVE treatment, so during periods of lower groundwater elevations deep interval treatment will be the priority). XDD will evaluate the system operation and determine the applicable soil depth interval(s) for soil sampling at the time of the annual sampling. Soil samples will typically be co-located with the baseline sampling locations.
b. Calculation of the COC mass remaining will be conducted following each annual soil sampling event (for the appropriate intervals).
c. COC mass reduction on the soils will be compared to the cumulative COC mass removed based on vapor concentration data (see "SVE vapor stream monitoring" above).

3. Soil vapor sampling: Soil vapor sampling will be conducted at SVE wells and vapor probes at startup and during the SVE system operation. Data will be used to evaluate changes in VOC concentrations over the operating duration of the SVE system, which will be used to indicate treatment progress both vertically and spatially across the site. Soil vapor sampling will consist of:
    a. Quarterly field screening for total VOCs at each SVE wellhead and vapor probe using a PID.
    b. Annual sampling of soil vapors for laboratory analysis at select SVE wellheads and vapor probes.

XDD Standard Operating procedures are presented in **Appendix A**.

1.2.2 PROCESS MONITORING:

The objective of process monitoring is to evaluate the mechanical performance of the system to ensure that equipment is operating as designed, track system operating conditions, and identify potential areas for system optimization. Process and Instrumentation Diagrams for the SVE system are provided in **Appendix B**. Process monitoring includes:

1. SVE system monitoring: Process monitoring data from the SVE process will be used to evaluate the mechanical performance of the equipment. SVE process monitoring will consist of:
    a. Monitoring of system vacuums/pressures, flowrates, and temperatures
    b. Monitoring vacuum/pressures and flowrates at wellheads.
    c. Monitoring vacuum/pressures observed at vapor points.
    d. Monitoring water/condensate (e.g. totalizer readings) and non-aqueous phase liquid (NAPL) recovery rates.
    e. Monitoring of the operating parameters of the thermal oxidizer (ThermOx) units (see **Appendix C**).

2. Water treatment monitoring: The treatment effectiveness of the liquid granular activated carbon (LGAC) treatment system will be evaluated monthly to determine activated carbon usage rates and the carbon change-out timeframe (see **Appendix D**).

3. Groundwater level monitoring: Groundwater levels will be monitored monthly at BSA-MW-1S (see **Figure 2**) to determine the optimal timeframe to focus the SVE system on the deep interval which will periodically be below the water table.

1.2.3 COMPLIANCE MONITORING

The objective of compliance monitoring is to satisfy the air and water discharge permit requirements; see **Appendices C and D**. The compliance monitoring generally includes:

1. Vapor treatment sampling: Vapor/air discharge sampling will be performed to assess treatment of extracted soil vapor by the ThermOx unit(s) prior to discharge to atmosphere and demonstrate compliance with the Illinois Environmental Protection Agency permit.

2. Water sampling: Pre- and post-treatment water discharge samples will be performed to assess treatment of condensate collected by the SVE process equipment by liquid granular activated carbon (LGAC). Influent and effluent samples will be collected and analyzed for VOCs to confirm treatment efficiency of the LGAC units and demonstrate compliance with the American Bottoms water discharge permit.

2.0 QUALITY ASSURANCE PROJECT PLAN

2.1 QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVE

The overall QA/QC objective is to develop and implement procedures for field sampling, laboratory analysis, and reporting to obtain scientifically valid results and to meet Data Quality Objectives (DQOs). Procedures for sampling, chain-of-custody documentation, laboratory analysis, data reporting, and internal quality control are described in this SAP. The purpose of this section is to state the expected level of QC effort for both field and laboratory activities and to present the specific, required data quality indicators (DQI) for accuracy, precision, sensitivity, representativeness, comparability, and completeness of data.

2.2 DATA QUALITY REQUIREMENTS

2.2.1 ACCURACY

Accuracy is measured by the difference between the measured or observed value and the true or assigned value. Accuracy in the field is assessed through the adherence to all sample handling, preservation, and holding times. Laboratory accuracy is assessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD) samples and/or laboratory control samples (LCS) for the determination of percent recoveries. The measurement of "standards", or materials of accepted reference values, provides an assessment of the accuracy of laboratory instruments and analytical methods. Accuracy will be evaluated through the use of USEPA Quality Control Samples or Standard Reference Materials. Accuracy is expressed as percent recovery:

$$\text{Percent Recovery} = Q_d/Q_a \times 100\%$$

where,

Q_d = quantity determined by analysis

Q_a = true or accepted reference value

2.2.2 PRECISION

Precision will be assessed through the collection and measurement of field duplicates at a rate of one duplicate per ten samples and through the calculation of relative percent difference (RPD) for duplicate samples and between MS and MSD samples. The total number of duplicates for this project will depend on the number of field measurements collected.

In addition to the field-collected duplicate samples, additional laboratory duplicates may be analyzed. Precision will be expressed as RPD:

$$RPD = [(D_1-D_2)/((D_1+D_2)/2)] \times 100\%$$

where,

RPD = Relative Percent Difference

D_1 = First Duplicate Value (percent recovery)

D_2 = Second Duplicate Value (percent recovery)

2.2.3 SENSITIVITY

Sensitivity is the capability of a laboratory instrument or test method to discriminate between measurement responses representing different levels (concentrations) of a compound or constituent. The internal laboratory procedures are used to optimize an instrument's sensitivity to the lowest possible concentration levels. Sensitivity will be evaluated by comparing the results to the designated quantitation limits.

2.2.4 REPRESENTATIVENESS

Sample representativeness will be reviewed qualitatively through the review of the proper use of specific sampling and/or analysis procedures, through internal field sampling audits and through the use of field QC data (including field duplicates and equipment blanks). RPD will be used to quantitatively assess representativeness.

2.2.5 COMPARABILITY

Maximum comparability will be achieved by using consistent sampling and sample handling procedures. Documentation of such sampling events will provide the means necessary to compare data from different events.

2.2.6 COMPLETENESS

Completeness is the percentage of valid/usable data compared to the total number of data:

$$C = V/T \times 100\%$$

where,

C = Percent completeness

V = Number of measurements judged valid

T = Total number of measurements

The QA objective is to obtain acceptable data for 90 to 95 percent of the samples received. Completeness also implies the ability of the final laboratory report to answer XDD questions.

2.3 INTERNAL QUALITY CONTROL

Quality control requirements include both field and laboratory samples and procedures designed to ensure and document the overall quality of the data. Quality control check samples are controlled samples, introduced into the analytical system at specific points. The results of the quality control checks are used during data validation to evaluate the precision, accuracy, sensitivity and representativeness of the overall sampling and analytical program.

2.3.1 FIELD ANALYTICAL QUALITY CONTROL

Field duplicates will be collected to provide a measure of the reproducibility of the sampling procedures. When a field duplicate is required, a second set of samples will be collected from a

single sampling location in addition to the initial field samples. The collection of the duplicate sample should immediately follow the collection of the field sample.

Each field duplicate sample will be labeled with a unique identification number and submitted with the field sample. Field duplicate samples will be collected and submitted for volatile organic carbon (VOC) analysis and will be collected at a frequency of one per ten soil, soil vapor, or water samples.

2.3.2 TRIP BLANKS

Trip blanks will consist of VOA vials that are filled with the laboratory blank water and preserved to a pH of less than 2. Trip blank results are used as indicators of contamination originating from the proximity of sample containers to one another during shipment and storage. Trip blanks will be prepared by the laboratory and delivered to the field personnel to be included with shipments of field samples to the laboratory. The trip blanks will not be opened until they are analyzed. There must be at least one trip blank in every cooler used to ship samples to the laboratory for VOC analysis.

2.3.3 EQUIPMENT BLANKS

Equipment blank analysis: one per ten samples, or at least one per day, to assess the decontamination procedures. Equipment blanks are required for each type of non-dedicated equipment used at the Site (i.e., reuse of Tedlar bag to collect soil vapor samples).

2.3.3.1 MS/MSD Samples

MS/MSD analysis results reflect the ability of the laboratory and method to accurately determine the quantity of an analyte in a particular sample. Additional volumes for MS/MSD samples will be collected as quality control samples for the VOC analysis only for Level IV validation. MS/MSD samples will be collected in the field at a single sampling location as described for field duplicate samples. MS/MSD samples will be collected and submitted for VOC analysis

and spikes will be added in the laboratory. MS/MSD samples will be collected at a frequency of one for every twenty soil samples.

2.3.4 LABORATORY QUALITY CONTROL

Analytical QC will be monitored through laboratory quality control checks. The data quality objective (DQO) level for the laboratory analysis varies from Level III to Level IV. The minimum required QA/QC procedures that will be conducted by the laboratory for DQO Level IV data include the following:

- Laboratory duplicate analysis to assess reproducibility of measurement. Laboratory duplicates may also be run for internal laboratory QA purposes.
- MS/MSD sample analysis (one per twenty samples) provides information about the effect of the sample matrix on the digestion procedure and measurement methodology. All MS samples will be performed in duplicate and are referred to as MS/MSD samples. MS/MSD samples will be analyzed for each matrix sampled.
- The laboratory will run a method (preparation) blank at the beginning of each analytical run per sample matrix and per analytical method each day.
- Upon initiation of an analytical run, the laboratory will perform calibration procedures as dictated by the analytical method(s) used. In addition, calibrations will be performed according to instrument manufacturer specifications. Continuing calibrations will be performed at the frequency specified during the length of the run. Where applicable, calibration blanks will be included in the calibration procedure.
- Surrogate standards will be added to all samples for organic analysis. Surrogate recovery will be used to assess accuracy of organic analyses.
- Sample chains-of-custody will be maintained and documented as outlined in this SAP. Copies of the chain-of-custody sheets will be submitted to USEPA with the data sheets in their respective report submittals.
- Laboratory data, documentation, reports, and any other project records must be kept on file for at least three years after the date of submission of the final report or as required by regulations, whichever is longer.

2.4 ASSESSMENTS AND RESPONSE ACTIONS

Assessments will be conducted by XDD QA personnel. These activities will be conducted to assess the implementation of the SAP and verify compliance with all aspects of quality assurance. Any deficiencies identified by the assessment team will be documented, reported to the XDD Project Manager and QA Officer, and resolved.

During the assessment process, deficiencies and items of concern related to field sampling procedures will be brought to the immediate attention of the sampling personnel so that appropriate response actions can be taken as soon as practicable. This will minimize potential field error and ensure the usability of data from subsequent sampling locations.

2.5 REPORTS TO MANAGEMENT

Following the completion of any assessment activities, the assessment team will submit a report to the XDD Project Manager and QA Officer. Sufficient information will be provided so that the potential impact on data quality can be evaluated.

2.6 DATA REDUCTION

The selected analytical laboratory will be responsible for reducing analytical results to concentrations using the analytical procedures and equations specified in each method SOP. All calculations shall be checked by senior analytical staff.

2.7 DATA VALIDATION

Analytical data will be evaluated and accepted or rejected based on a set of criteria. The following criteria will be used in the validation of laboratory data:

- Adherence to published or approved analytical procedures;
- Properly operating and calibrated instruments;

- Comparable precision and accuracy achieved in an initial demonstration of capability or similar analytical program(s); and
- Completeness of data set.

Occasionally, a single result is found that differs from the rest of the relevant data by more than would be reasonably expected on the basis of good analytical practice. Such a value is referred to as an "outlying" value. Records of all data will be maintained, even those judged to be "outlying" values. All data will be validated by laboratory supervisors prior to being released for reporting purposes to the project manager. The persons validating the data will have sufficient knowledge of the technical work to identify questionable values.

In addition to the internal laboratory validation procedures, validation of the analytical data will be performed by the project QA Officer or designee according to the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-99/008, October 1999.. The validated project data will be evaluated for overall usability using data quality indicators (accuracy, precision, sensitivity, representativeness, comparability, and completeness).

2.8 DATA REPORTING

The laboratory data will be provided to XDD in electronic format. Data reports submitted from the laboratory will include:

- Sample results with concentration units;
- Data qualifiers, where applicable;
- Statement of methods for each parameter;
- Date of sample receipt;
- Initialed chain of custody form;
- MDL for each method; and
- Sample extraction and analysis dates.

The reports will include a cover page and/or case narrative outlining the case specifics and any problems or corrective actions. Laboratory data reports will be imported to and maintained in a Microsoft® Access database, or similar.

3.0 SAMPLING AND MONITORING PROCEDURES

Overall sampling procedures, including field documentation requirements, quality assurance/quality control (QA/QC) procedures, detailed soil and vapor sampling procedures, sample locations, chain-of-custody records and calibration records are presented in the following subsections. Data quality objectives, QA/QC requirements and field equipment calibration procedures are also provided in this section.

3.1 SAMPLE COLLECTION

3.1.1 FIELD DOCUMENTATION

Sample collection information may be recorded in a bound field notebook with pre-numbered pages or on a pre-printed form. Field records must be written in indelible ink. At a minimum, all documentation errors shall be corrected by drawing a single line through the error so that it remains legible; the error must then be initialed by the responsible individual and the date of the change noted. The correction shall be written adjacent to the error. The following information will be documented in the field notebook or on designated field forms.

A. *For the sampling event:* the Site name and location, date, starting and ending times, weather, names of all the people involved in sampling activities, level of personal protective equipment (PPE) used, documentation of adherence to protocol, any changes made to planned protocol, names of visitors to the Site during sampling and reason for their visit, unusual observations and signature of the person recording the information.
B. *For each individual sample:* a detailed description of location, any measurements made, the unique sample number assigned, the time the sample was taken, physical description of sample, (as appropriate) depth from which the sample was collected, equipment used to collect the sample, volume and number of sample containers, how the sample is preserved and signature of sampler. Each field duplicate must be given a unique sample number; the description should include the unique sample number of its duplicate.

3.1.2 SOIL SAMPLING

The field screening, soil sampling, and decontamination procedures for soil sampling are discussed in the following subsections. The XDD standard operating procedure (SOP) for direct push soil sampling will be followed and is provided in **Appendix A**.

3.1.2.1 Field Screening Procedures

Field screening of soil samples for total VOCs will be performed using a Portable MiniRAE 2000 PID (or equivalent). Samples will be placed in a sealed plastic bag and allowed to equilibrate with the bag atmosphere for at least two minutes. Where ambient temperatures are below 32°F, headspace development will occur within a heated vehicle or building.

After at least two minutes have elapsed, the PID tip is used to quickly puncture the plastic bag, taking care that the PID tip does not contact soil within the plastic bag. The highest PID response, which should occur within 2 to 5 seconds, is recorded as the headspace concentration. Between sample readings, the PID response should return to near zero parts per million by volume (ppmv).

3.1.2.2 Soil Sampling Procedures

Soil sample collection will be performed from direct push cores using a disposable Terracore sampling device. Soil sampling locations are indicated in **Figure 3**. Field screening techniques described in **Section 3.1.2.1** (i.e., total VOCs measured with a PID) will be used as needed based on field observations.

Soil sampling will be conducted annually beginning after approximately one year of system operation. Samples will be co-located with borings that were used to establish the estimated contaminant mass in the Big Mo and Former Benzene Pipeline areas (**Figure 3**). Soil boring logging will be conducted at each location to determine if the sample is directly co-located with the previous borings. If grout remnants are observed, the location will be offset by one foot to the east and an additional attempt will be conducted. At up to 15 of these locations, soil

samples will be taken from the sandy fill/upper silty sand and lower silty sand layers. As described in **Section 1.2.1**, soil sampling will target the depth interval(s) in which the system operated during the preceding year and a two-tiered approach (Approach 1 and Approach 2) will be used to collect data to estimate mass reduction. XDD will evaluate operation of the SVE system prior to the soil sampling event to determine the applicable sample target intervals.

Each soil core will be field screened as described in **Section 3.1.2.1**. Approach 1 soil samples will be collected from the depth with the highest PID reading within each treatment interval. The depth intervals may vary between borings and sample depths will be based upon the actual depths of the target soils being sampled.

In the same soil core, additional soil samples will be collected at two-foot intervals from a minimum depth of 3 feet to a maximum depth of 15 feet within the targeted treatment intervals (shallow and deep) for Approach 2.

During the final soil sampling event, which will commence after asymptotic conditions are reached (approximately 3 to 4 years), all of the soil borings used for delineation and mass estimation purposes will be targeted for sampling via both Approach 1 and Approach 2. Additionally, soil samples from the intermediate silty clay layer will be collected.

Continuous soil cores will be collected using a 2-inch diameter stainless steel sampling device from ground surface to 15 ft bgs. Boreholes will be backfilled with hydrated bentonite chips. After backfilling, sample locations will be marked with a 2-inch field marker to ensure that those locations will not be selected for sampling during subsequent sampling events.

Soil samples will be collected using a Terracore (or equivalent) sample kit and shipped on ice contained within sealable plastic bags. The sample coolers will contain enough ice to maintain the contents at or less than 4°C during transport. Samples will be transported to the laboratory at the end of each sampling event for analysis of VOCs.

3.1.2.3 Decontamination Procedures

Disposable sampling equipment will be used wherever possible. However, if reusable sampling devices are used, the following decontamination procedure will be followed:

1. Rinse with potable water;
2. Scrub with a brush to remove soil, groundwater and/or residual contamination material;
3. Wash or rinse with laboratory-grade non-phosphate detergent;
4. Rinse with dilute isopropanol;
5. Double rinse with potable water;
6. Rinse with deionized water;
7. Air dry or dry with a lint-free paper towel; and
8. Wrap in clean aluminum foil for storage, if necessary.

Decontamination water will be collected and drummed with labels for on-site storage until proper disposal.

3.1.3 VAPOR SAMPLING

The field screening procedures, vapor sampling procedures and decontamination procedures for sampling in the treatment areas is discussed in the following subsections. The XDD SOP for vapor sampling with Tedlar bags is provided in **Appendix A**.

3.1.3.1 Field Screening Procedures

Field screening of vapor samples will be performed using a MiniRAE 2000 PID (or equivalent). Samples will be collected in a Tedlar bags filled using an air sample pump connected to sample ports, which will be installed throughout the SVE system. The sample pump will be thoroughly flushed with ambient air prior to sample collection. Tedlar bags will be filled with ambient air and flushed three times prior to reuse. Flexible Tygon tubing will be used to connect the sample port to the sample pump inlet and the sample pump outlet to the Tedlar bag. The Tedlar bag will be filled and emptied and then filled and screened using the MiniRAE 2000 PID (or equivalent) connected to the Tedlar bag. The highest PID response, which should occur within 2 to

5 seconds, is recorded as the vapor concentration. Between sample readings, the PID response should return to near zero.

3.1.3.2 Vapor Sampling Procedures

Representative vapor samples will be collected from the following sampling ports installed throughout the SVE system using an air sample pump as described in the field screening procedures:

- Flow from wellfield (SP-001)
- Influent for PDB-101 (SP-101)
- Effluent from PDB-101 (SP-102)
- Influent for PDB-201 (SP-201)
- Effluent from PDB-201 (SP-202)
- Influent for PDB-301 (SP-301)
- Effluent from PDB-301 (SP-302)
- Effluent to ThermOx units (SP-401)

Select vapor samples will be submitted to the laboratory for VOC analysis. Care will be taken to provide adequate sample volume for laboratory analysis requirements. Samples will be shipped to the laboratory at the end of each sampling event for VOC analysis.

3.1.3.3 Decontamination Procedures

Disposable sampling equipment will be used wherever possible. However, if reusable sampling devices are used, the equipment shall be flushed with either ambient air (in the case of the sample pump) or filled with three volumes of sample air and emptied, prior to sample collection (in the case of the Tedlar bag).

3.2 SAMPLE IDENTIFICATION AND LABELING

Each sample submitted for laboratory analysis will be assigned a unique field identification number according to the following codes:

BF-MMYY-AAA-BB-CC-D

Where:

BF is the treatment area (e.g. **B**ig Mo and **F**ormer Benzene Pipeline)

MMYY is the abbreviated field sample date (e.g. month and year)

AAA is the unique sampling location number (e.g. boring location)

BB-CC is the depth interval over which the soil sample was collected (e.g. 04-05 ft bgs)

D is the sample type (S = soil, V = vapor, A = air, W = water, AD = analytical duplicate, EB = equipment blank, TB = trip blank, MS = matrix spike and MD = matrix duplicate)

Field sample identification numbers will be assigned in the field by the sampling crew. A label will be affixed to each individual sample container with the following information written legibly in waterproof ink:

- Field sample identification number
- Date and time of sample collection
- Sample matrix
- Preservative
- Analysis to be performed
- Name and initials of sampler

3.3 CHAIN-OF-CUSTODY RECORDS

Chain-of-custody records will be initiated by the samplers in the field. Custody documentation will include: (1) the project name; (2) signature of samplers; (3) the sample number, date and time of collection and whether the sample is grab or composite; (4) signatures of individuals involved in sample, including laboratory receiving personnel; and (5) if applicable, air bill or other shipping number.

3.4 LABORATORY PROCEDURES

All laboratory analyses will follow approved procedures and preparatory methods, as applicable. All analyses will be performed by Pace Analytical of Lenexa, Kansas unless otherwise stated.

Pace Analytical's Quality Assurance Manual includes details on the laboratory's quality assurance and quality control policies and procedures (**Appendix E**). Details on the USEPA Method 8260B and TO-14 laboratory analyses are presented in Pace Analytical's SOPs (**Appendix E**).

3.5 CALIBRATION RECORDS

For all field analyses with field instrumentation, calibrations will be performed and documented. Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program shall include provisions for documentation of frequency, conditions, standards and records reflecting the calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used.

Calibration of field instruments will be performed daily, at intervals specified by the manufacturer, or more frequently as conditions dictate. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service and/or replacement. Calibration results will be recorded in the site Health and Safety Plan.

TABLES

TABLE 1
Sampling Plan Summary
SVE System Sampling and Analysis Plan
W.G. Krummrich Facility, Sauget, Illinois

Purpose	Sample Type	Minimum Frequency	Measurement Type	Details/Rationale
Soil Sampling	Performance Monitoring	Annually	Field Screening	Soil cores will be field screened for total VOCs.
			Laboratory Analysis	Samples will be collected to assess reductions in soil concentration and soil COC mass during the SVE system operation.
Soil Vapor Sampling	Performance Monitoring	Pre-startup Quarterly (after startup/shakedown)	Field Screening	Field screening for total VOCs at all SVE wells and vapor probes during startup and at select active SVE wells and vapor probes thereafter to evaluate the SVE system effectiveness.
		Annually	Laboratory Analysis	Samples will be collected from select SVE wells and vapor probes to assess subsurface air flow patterns and dynamic contaminant distributions and evaluate changes in VOC concentrations over the operation of the SVE system.
SVE Well Field Monitoring	Performance Monitoring	Quarterly	Field Screening	Field measurements including vac/press and extraction/injection flowrates.
SVE System Monitoring	Process Monitoring	Monthly	Field Measurements	Process measurements (i.e., temperature, vac/press, air flow, condensate collection rates, and volume extracted) will be collected throughout the system process equipment and used to optimize system performance.
	Performance Monitoring	Monthly	Field Screening	Vapor samples will be collected from the SVE process equipment for measurements of vapor concentrations throughout the SVE system.
			Laboratory Analysis	Samples will be collected from the combined SVE stream prior to the ThermOx units to estimate the total mass removal rate.
SVE System Monitoring	Performance Monitoring	As needed during startup (~ 1 mo.) Bi-weekly (up to 2 mos.) Monthly (thereafter)	Field Measurements	Vapor samples will be collected from the SVE process equipment for measurements of vapor concentrations throughout the SVE system.
		Monthly	Laboratory Analysis	Post-blower/pre-treatment combined SVE vapor samples collected for compliance (see below) and performance monitoring.
	Process Monitoring	As needed during startup (~ 1 mo.) Bi-weekly (up to 2 mos.) Monthly (thereafter)	Field Measurements	Process measurements (i.e., vac/press, extraction/injection flow rates) will be collected at various points throughout the SVE process equipment. Process monitoring also includes water/condensate and NAPL system monitoring, and groundwater level monitoring.
Water Level Monitoring	Process Monitoring	Monthly	Field Measurements	Water levels will be monitored to determine the optimal timeframe to focus the SVE system on the deeper treatment interval, which may periodically be below the water table.
Air Discharge Compliance Monitoring	Permit Compliance and Treatment Monitoring	Monthly	Laboratory Analysis	Samples will be collected pre- and post-treatment at the ThermOx units to confirm treatment effectiveness.
Water Discharge Compliance Monitoring	Permit Compliance and Treatment Monitoring	Monthly	Laboratory Analysis	Samples will be collected pre-, mid-, and post-treatment at the LGAC units to confirm treatment effectiveness and determine the change out schedule for the LGAC.

SVE = soil vapor extraction
VOC = volatile organic compound
COC = constituents of concern
vac/press = vacuum/pressure
LGAC = liquid-phase granular activated carbon
ThermOx = thermal oxidizer

TABLE 2

Sampling Plan Details

SVE System Sampling and Analysis Plan

W.G. Krummrich Facility, Sauget, Illinois

Task	Sampling Location	Measurement Type	Minimum Frequency	Samples Per Event	Sample Type/Collection	Analytical Method
Baseline Sampling[1]						
Soil Vapor Sampling	SVE wells (Figure 2)	Field screening	Once (2012 startup)	158	Vapor samples will be collected using a vacuum or hand pump and screened for total VOCs. Vapor samples for analytical analysis will be collected into dedicated 1-L tedlar bags.	PID
	Vapor probes (Figure 2)			32		
Performance Monitoring						
Soil Sampling	Co-Located at URS boring locations (Figure 3)	Field screening	Annually	As needed[2]	Headspace analysis per SAP	PID
		Laboratory analysis		15[3]	Soil samples via Terracore[4]	SW846 8260B
Soil Vapor Sampling	SVE wells (Figure 2)	Field screening	Quarterly	All active SVE wells	Vapor samples will be collected using a vacuum or hand pump and screened for total VOCs in the field using a PID.	PID
		Laboratory analysis	Annually	10		EPA-21 TO-14[5]
	Vapor probes (Figure 2)	Field screening	Quarterly	32		PID
		Laboratory analysis	Annually	3		EPA-21 TO-14
SVE Well field Monitoring	SVE wells (Figure 2)	Field measurements	Quarterly	All active SVE wells	Field measurements including vac/press and extraction/injection flowrates	NA
	Vapor probes (Figure 2)	Field measurements	Quarterly	32	Field measurements including vac/press measurements	NA
System Monitoring	SVE process equipment including the air treatment ThermOx unit(s) (pre-SVE blowers, post-SVE blowers, and post treatment by ThermOx units) (Appendix C)	Field screening	As needed during startup (up to 1 mo.)	Up to 12 locations	Vapor samples will be collected using a vacuum or hand pump and screened for total VOCs in the field using a PID.	PID
			Biweekly (up to 2 mos.)			
			Monthly (thereafter)			
	Combined post-blower SVE stream (Appendix C)	Laboratory analysis	Monthly	1	The post-blower/pre-treatment combined SVE vapor samples collected will be used for compliance monitoring (see below) and to calculate system mass removal rates.	EPA-21 TO-14
	ThermOx unit(s) post-treatment (Appendix C)	Laboratory analysis	Monthly	2	The post-treatment ThermOx samples collected will be used for compliance monitoring (see below).	EPA-21 TO-14
Water Treatment Monitoring	Pre-, mid-, and post-treatment samples from LGAC units (Appendix C)	Laboratory analysis	Monthly	3	The pre- and post-treatment samples collected will be used for compliance monitoring (see below). The mid-point sample will be used to determine activated carbon usage rates and the carbon change out schedule.	EPA 624 and EPA 625
Process Monitoring						
SVE System Equipment	Process equipment (Appendix C)	Field measurements	As needed during startup (up to 1 mo.)	Throughout SVE process stream	Field measurements including temp., vac/press, flowrates, and operating parameters will be collected throughout the process equipment	NA
			Biweekly (up to 2 mos.)			
			Monthly (thereafter)			
ThermOx Equipment	ThermOx unit(s) (Appendix C)	Field measurements	Monthly	NA	Field measurements including temp., vac/press, flowrates, and operating parameters will be collected throughout the ThermOx equipment.	NA
Water/condensate Monitoring	Water/condensate storage and treatment equipment (Appendix C)	Field measurements	Monthly	NA	Field measurements including vac/press, flowrates, and totalizer readings through the water/condensate process equipment.	NA

TABLE 2

Sampling Plan Details

SVE System Sampling and Analysis Plan

W.G. Krummrich Facility, Sauget, Illinois

Task	Sampling Location	Measurement Type	Minimum Frequency	Samples Per Event	Sample Type/Collection	Analytical Method
Process Monitoring (cont.)						
NAPL Monitoring	NAPL storage and transfer equipment (Appendix C)	Field Measurements	Monthly	NA	Field measurements including NAPL recovery rates,	NA
Water Level Monitoring	Select SVE wells and monitoring well BSA-MW-1S	Field Measurements	Monthly	NA	Field measurements of the depth to water in monitoring well(s) within the treatment area.	water level indicator
Compliance Monitoring						
Air Discharge	Pre- and post-treatment vapor samples from combined SVE stream and ThermOx units (Appendix C)	Laboratory analysis	Monthly	2	The vapor streams will be field screened for total VOCs using a PID. Vapor samples for analytical analysis will be collected into dedicated 1-L tedlar bags using a vacuum or hand pump.	EPA-21 TO-14
Water Discharge	Pre- and post-treatment from LGAC701 and 702 (Appendix C)	Laboratory analysis	Monthly	2	Pre- and post-treatment condensate samples will be collected from pressurized sample ports into VOA vials	EPA 624 and EPA 625

SVE = soil vapor extraction
L = liter
SAP = Sampling and Analysis Plan prepared by XDD, LLC dated January 2012
PID = photoionization detector
VOC = volatile organic compound
vac/press = vacuum/pressure
ThermOx = thermal oxidizer
P&ID = process and instrumentation diagram
LGAC = liquid-phase granular activated carbon
AI = air injection
NAPL = non-aqueous phase liquid
VOA = volatile organic analysis
URS = URS Corporation

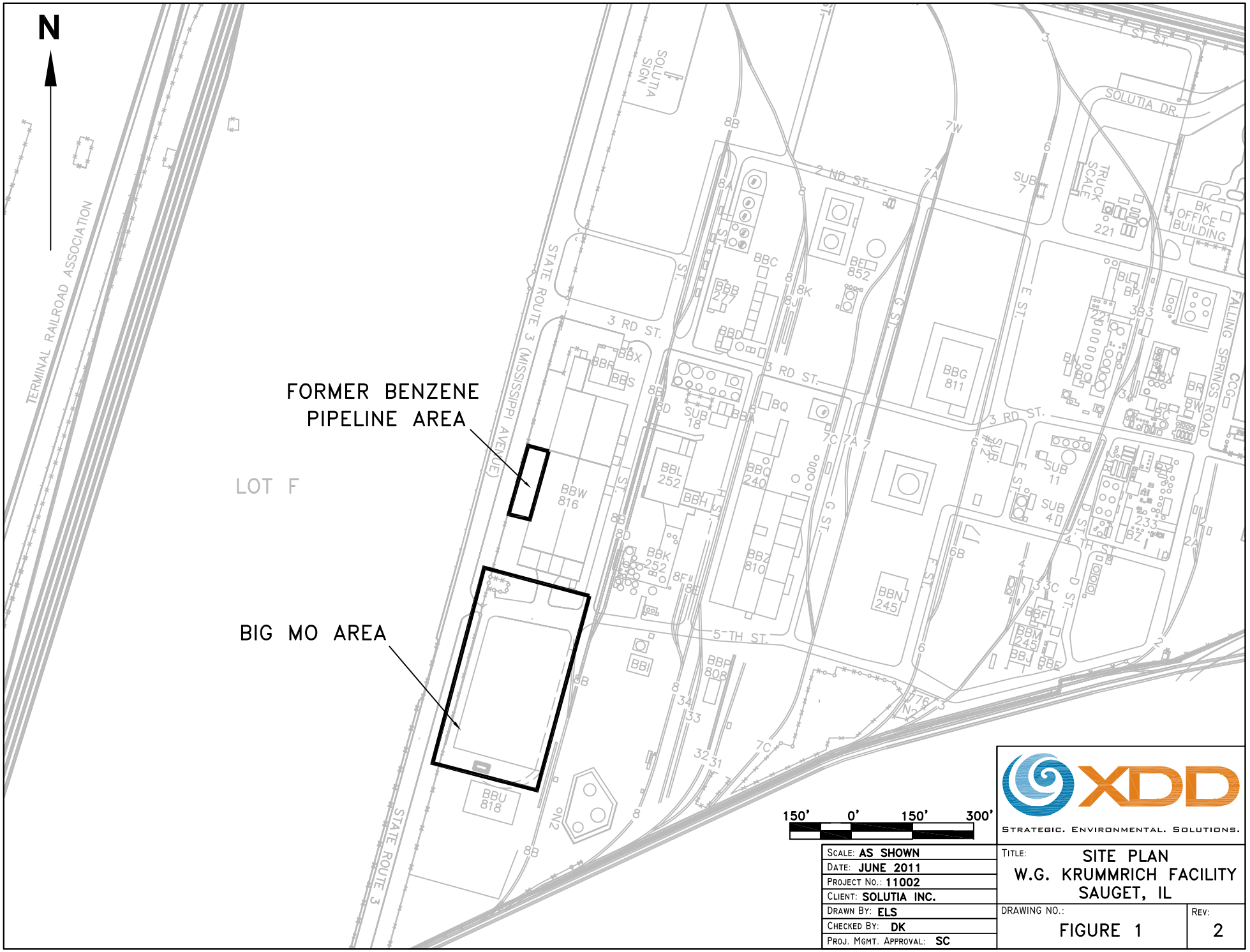
Notes:

1. The baseline soil sample data is based on the treatment area characterization study conducted by URS Corporation in 2009 and 2010 and presented in the "Soil Vapor Extraction Treatment Area Characterization Report" submitted to the USEPA in November 2010.
2. Field screening will be used as needed based on field observations.
3. Up to 15 soil sampling locations will be sampled depending on the system operation in each target treatment interval (maximum 30 samples). Sampling frequency and quantities may be adjusted based on the actual system performance.
4. Terracore sample kits are purchased from the analytical laboratory.
5. EPA-21 = “Compendium of Methods for the Determination of Toxic Organic Compounds In Ambient Air”, Second Edition, EPA/625/R-96/010B, January 1999.

TABLE 3
Analytical Sampling Parameters
SVE System Sampling and Analysis Plan
W.G. Krummrich Facility, Sauget, Illinois

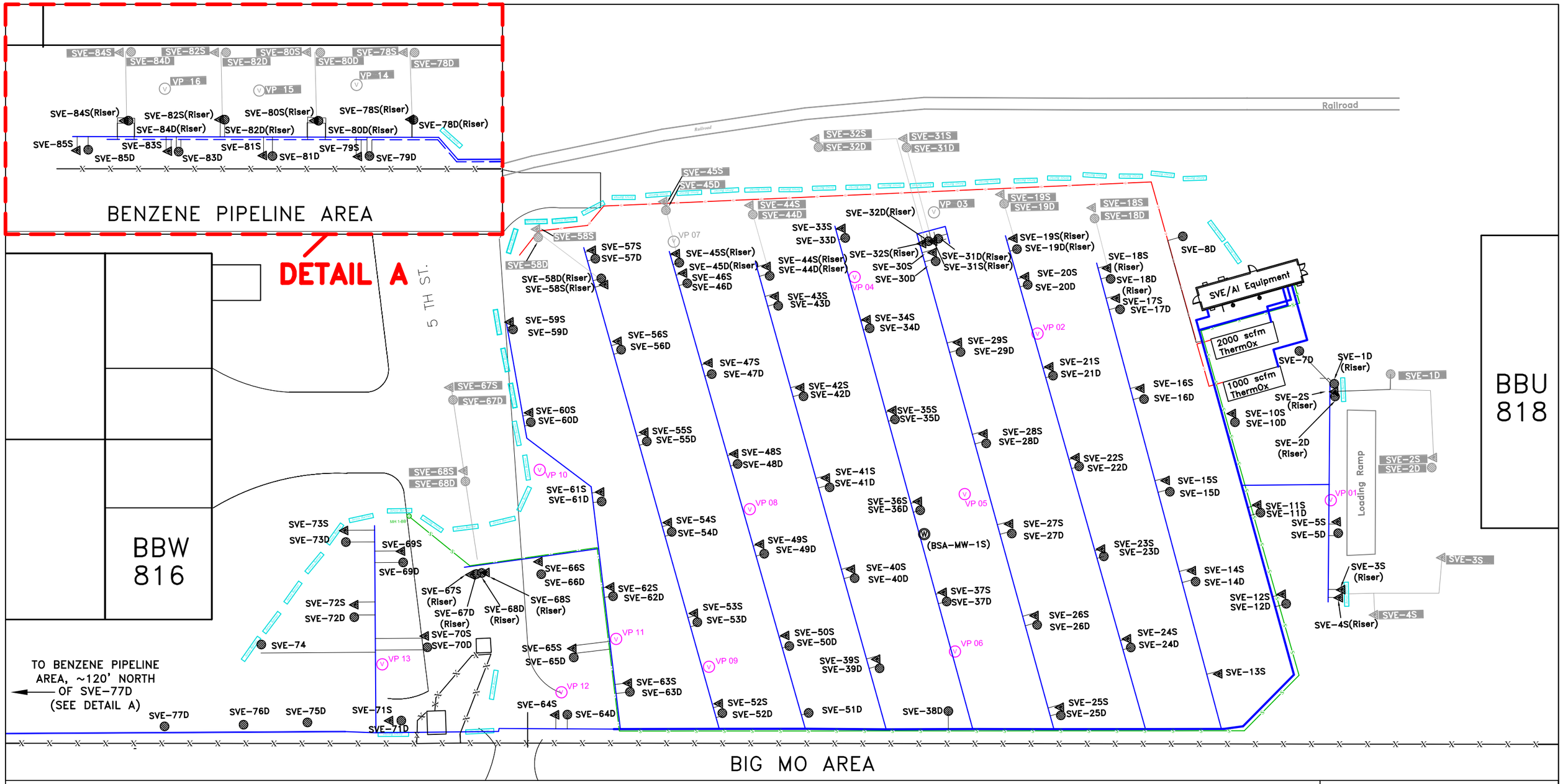
TASK	ANALYTICAL METHOD	ANALYTE LIST
Soil Vapor Sampling	EPA TO-14a	Benzene Chlorobenzene Ethylbenzene m&p-Xylene o-Xylene Toluene 1,4-Dichlorobenzene
Soil Sampling	EPA 8260B	Full Pace Analytical 8260B analyte list (89 compounds)
Water Treatment Monitoring	EPA 624	1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Benzene Chlorobenzene Ethylbenzene Toluene
	EPA 625	1,2,4-Trichlorobenzene 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol 2,4-Dichlorophenol 2-Chloroaniline 2-Chlorophenol 2-Nitroaniline 4-Nitroaniline Aniline Naphthalene Pentachlorophenol Phenol

FIGURES



XDD
STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

SCALE: AS SHOWN	TITLE: SITE PLAN W.G. KRUMMRICH FACILITY SAUGET, IL
DATE: JUNE 2011	
PROJECT NO.: 11002	
CLIENT: SOLUTIA INC.	
DRAWN BY: ELS	DRAWING NO.: FIGURE 1
CHECKED BY: DK	REV: 2
PROJ. MGMT. APPROVAL: SC	



LEGEND

- = ABOVE GRADE DEEP WELL
- = ABOVE GRADE SHALLOW WELL
- = BELOW GRADE DEEP WELL
- = BELOW GRADE SHALLOW WELL
- = MONITORING WELL
- = VAPOR PROBE
- = BELOW GRADE VAPOR PROBE
- = SEWER MANHOLE
- = FENCELINE
- = GAS LINE
- = BELOW GRADE 2" CARBON STEEL PIPING (APPROXIMATE LOCATION)
- = 2" ABOVE GRADE INSULATED AND HEAT TRACED SCHEDULE 40 PVC PIPING
- = 6" ABOVE GRADE INSULATED AND HEAT TRACED SVE SCHEDULE 40 PVC PIPING
- = 12" ABOVE GRADE INSULATED AND HEAT TRACED SVE SCHEDULE 40 PVC PIPING
- = JERSEY BARRIER
- = SEWER LINE
- = BELOW GRADE WELL/VAPOR PROBE ID

NOTES:

– ALL ABOVE GRADE BRANCH AND MANIFOLD PIPING WAS HEAT TRACED AND INSULATED
– CLEANOUTS WERE INSTALLED AT THE TERMINUS OF ALL BRANCH AND MAIN SVE LINES

20' 0' 20' 40'

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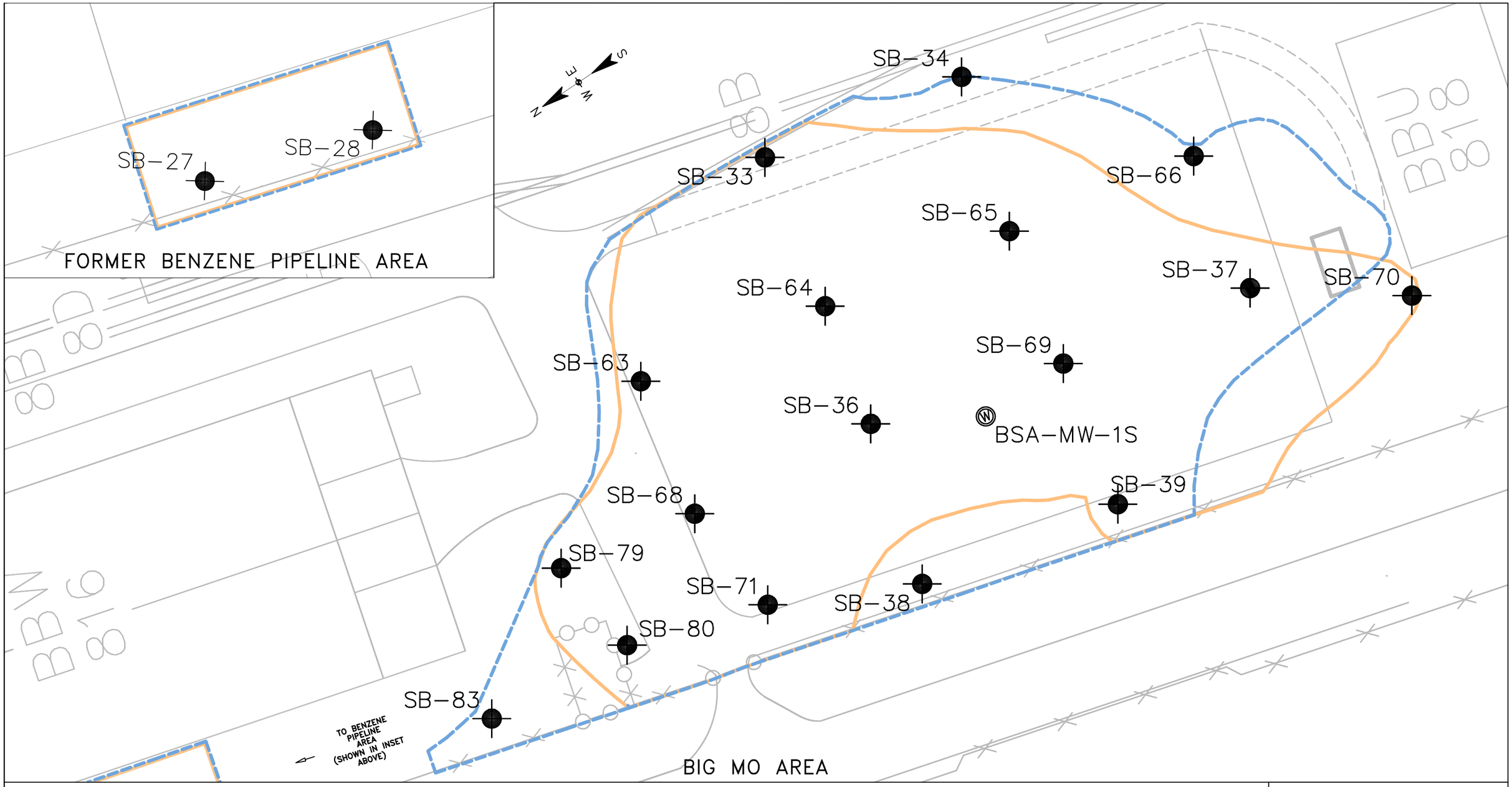
SCALE: AS SHOWN	TITLE: SVE WELL AND VAPOR PROBE LAYOUT BIG MO AND FORMER BENZENE PIPELINE TREATMENT AREAS W.G. KRUMMRICH FACILITY, SAUGET, IL
DATE: JUNE 2012	
PROJECT NO.: 11002	
CLIENT: SOLUTIA INC.	
DRAWN BY: JDA	DRAWING NO.: FIGURE 2
CHECKED BY: DK	REV: 2
PROJ. MGMT. APPROVAL: DK	



LEGEND

- APPROXIMATE EXTENT OF SHALLOW TARGET TREATMENT AREA
- APPROXIMATE EXTENT OF DEEP TARGET TREATMENT AREA
- APPROXIMATE LOCATION OF VEHICLE ACCESS ROAD
- LOADING DOCK
- SB-36 SOIL BORING LOCATION
- MONITORING WELL LOCATION

NOTE:

- SOIL BORING LOCATIONS ARE BASED ON THE TREATMENT AREA CHARACTERIZATION STUDY CONDUCTED BY URS CORPORATION IN 2009 AND 2010 AND PRESENTED IN THE "SOIL VAPOR EXTRACTION TREATMENT AREA CHARACTERIZATION REPORT" SUBMITTED TO THE USEPA IN NOVEMBER 2010.

25' 0' 25' 50'

SCALE: AS SHOWN	
DATE: MAY 2012	
PROJECT NO.: 11002	
CLIENT: SOLUTIA INC.	
DRAWN BY: JDA	
CHECKED BY: ELS	
PROJ. MGMT. APPROVAL: DK	

XDD
STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

TITLE: SOIL SAMPLING LOCATIONS
BIG MO AND FORMER BENZENE PIPELINE TREATMENT AREAS
W.G. KRUMMRICH FACILITY, SAUGET, IL

DRAWING NO.: FIGURE 3

REV: 1

APPENDIX A

XDD SOPs

XDD, LLC (XDD)

STANDARD OPERATING PROCEDURE (SOP)

STANDARD OPERATION PROCEDURE FOR SOIL SAMPLING VIA DIRECT PUSH (E.G., GEOPROBE®) METHODS

XDD SOP NO. DIRECT PUSH SAMPLER

1.0 OBJECTIVE

The purpose of this standard operating procedure (SOP) is to define the requirements for the collection of soil samples using the direct-push sampler method specific to the sampling protocol described in the Sampling and Analysis Plan for the Soil Vapor Extraction System – Big Mo and Former Benzene Pipeline Areas at the W.G. Krummrich Facility in Sauget, Illinois (SAP).

2.0 BACKGROUND

Collection of soil samples from soil borings is required to characterize the nature of soils, geology, geochemistry, and contamination. Soil handling and sampling will be performed in accordance with standard ASTM methodology.

The soil sampling program described in the SAP outlines a multi-leveled approach in which multiple soil samples will be collected from each soil boring. One soil sample will be collected from the depth exhibiting the highest total volatile organic compound (VOC) concentration based on field screening results and additional samples will be collected from each 2-foot intervals in the target treatment area.

3.0 METHOD

A track or truck mounted direct push drill rig will be used to advance a 2 7/8" I.D. steel casing. The casing will be advanced in two-foot intervals from the ground surface and soils within the casing sampled every two feet with a direct-push sampler.

3.1 Equipment

- 2-inch I.D. diameter, 2-foot long direct-push sampler with clear plastic sleeve
- Stainless steel knife to slice open clear plastic sleeve
- plastic sheeting
- soil boring log
- Unified Soil Classification System (USCS) chart
- Munsell soil color chart
- measuring tape
- Terracore® sampling kit (or similar)
- wide mouth sample jars
- ZipLock® Bags
- labels and shipping products for samples

- sample cooler and ice
- photoionization detector (PID) or equivalent
- logbook
- personal protective equipment specified in the Site Health & Safety Plan

3.2 Procedures

The following describes the sampling procedures for direct-push soil sampling using direct-push samplers. Personal protective equipment will be donned in accordance with the requirements of the Site Health and Safety Plan (HASP).

1. Set up a sample preparation area by placing plastic sheeting over the designated area. Place soil mixing pan, spatulas, stainless steel knife and sample jars on plastic sheeting. Prepare a disposal area for used plastic sample sleeves and remaining soil not placed in analytical containers.
2. Advance steel drive casing to desired soil sampling interval. The casing will be cleaned out using the appropriate size roller bit.
3. Drive clean, dedicated direct-push sampler inside drive casing through the desired soil sampling interval.
4. Retrieve direct-push sampler from within drive casing, loosen the cutting shoe and head assembly.
5. Remove the clear plastic sampling sleeve.
6. Record the soil interval depth and total length recovered on soil boring log.
7. Slice the clear plastic sampler in half, disposing of the clear halved sleeve. Slice the sampled soil core in half. Scan the recovered soil halved sample with the PID and record reading on soil boring log.
8. Collect soil from the one side of the halved core which had the greatest PID reading and place into a sealed plastic bag. The samples in the sealed plastic bags will be allowed to equilibrate with the bag atmosphere for at least two minutes. Where ambient temperatures are below 32°F, headspace development shall occur within a heated vehicle or building.
9. After at least two minutes have elapsed, the PID tip is used to quickly puncture the plastic bag, taking care that the PID tip does not contact soil within the plastic bag. The highest PID response, which should occur within 2 to 5 seconds, is recorded as the headspace concentration. Between sample readings, the PID response should return to near zero parts per million by volume (ppmv). Record reading on soil boring log.
10. Describe and classify the recovered soil sample using the USCS and Munsell charts and record observation on soil boring log.
11. For VOCs collect samples which had the highest observed headspace PID reading within the core. Label all required fields on the sampling container and chain of custody.
12. Place sample in cooler with ice.
13. Dispose of remaining soil on 6 ml. plastic and cover with same 6 ml. plastic for future disposal. Weigh down the edges of the plastic to prevent from wind disturbance.

3.3 Decontamination

Direct-Push samplers, soil mixing pans, and spatulas will be decontaminated by the following:

1. Steam clean prior to initial use at the site.
2. Scrub the equipment with a brush in a five-gallon bucket filled with potable water to remove soil.
3. Transfer the equipment to a second five-gallon bucket filled with potable water/alconox solution and brush a second time.
4. Rinse the equipment with deionized water.

If using non-dedicated equipment and conducted decontamination operations, collect one equipment blank each day soil sampling is performed and analyzed for VOCs via EPA Method 8260b to monitor the decontamination procedures as part of the quality assurance/quality control (QA/QC) protocol. In addition, collected one field blank from each water source (potable and deionized water) used for sampling equipment decontamination and analyze for VOCs via EPA Method 8260b as part of the QA/QC program.

One Duplicate soil sample will be collected for ever ten soil samples collected and analyzed for VOCs via EPA Method 8260b as part of the QA/QC protocol.

A trip blank (provided by the laboratory) will accompany every sample delivery group and be and analyzed for VOCs via EPA Method 8260b as part of the QA/QC program.

4.0 RESIDUAL MANAGEMENT

Residual soil, pavement, and concrete (if any) generated during site activities will be drummed, sampled and disposed off-site in a permitted landfill in accordance with the facilities waste management plan. Little residual soil is anticipated since the direct push drilling method does not generate soil cuttings.

5.0 REFERENCES

ASTM D 6282 – 98 Standard Test Method for Direct-Push Sampling of Soils.

ASTM D 2488 – 93 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure).

XDD, LLC

STANDARD OPERATING PROCEDURES (SOP)

PROCEDURES FOR OBTAINING VAPOR SAMPLES IN TEDLAR™ BAGS FROM SOIL VAPOR PROBES/SVE WELLS, SVE SYSTEM INLETS, AND OFF-GAS TREATMENT SYSTEMS

XDD SOP NO.: SMP-TEDLAR

1.0 OBJECTIVE

To establish procedures for the collection of soil vapor samples from sub-surface soil vapor probes, soil vapor extraction systems (SVES), carbon canister discharge, or catalytic oxidation system (CATOX) discharge. Vapor samples are collected in 1-liter Tedlar air sample bags for VOC, O_2, CO_2, or CO analyses via laboratory or field instrumentation.

2.0 DEFINITIONS

Vapor Probe – Vapor probes are vadose zone soil vapor "sampling ports". Vapor probes are installed to provide access to sampling of soil gas for VOC analyses and pressure measurements.

SVES – Soil Vapor Extraction System.

Sample Pump – A relatively small-scale (1/8 to ¼ HP) diaphragm or rotary vane vacuum pump. The sample pump is capable of achieving a vacuum pressure of >27 inches of mercury ("Hg). The sample pump is equipped with 3/16" I.D. by 1 to 2 feet of Teflon tubing at the inlet and outlet of the pump. A glass wool filter installed at the sample pump inlet will prevent particulates from blocking and/or damaging the sample pump.

Tedlar air sample bag - Bags manufactured from polyvinyl fluoride PVF (Tedlar) film. They are generally considered inert and can be used to collect samples containing common solvents, hydrocarbons, chlorinated solvents, and many other classes of compounds. Tedlar bags can be used for VOC concentrations down to approximately 5 parts per billion (ppb), below which, either Tedlar bags manufactured from fluorinated ethylene propylene (FEP) or Summa canisters should be used.

VOC – Volatile organic compound.

3.0 DIRECT SAMPLING METHOD

3.1 Equipment

1. Sample Pump – 12 VDC, 120VAC, or hand driven.

2. 1 liter Tedlar air sample bag(s) equipped with sample valve(s) with 3/16" O.D. by 1 inch tubing extending from the sample valve.
3. Extra lengths and sizes of Teflon and/or Tygon tubing.
4. Charged 12 V battery, car battery adapter/extension cord, or 115 VAC extension cord to supply power to sample pump (if necessary).
5. Health and safety gear as specified in the Health and Safety Plan.
6. Field notebook with pen, and/or appropriate data logging form.
7. Sample air flow meter, appropriately sized (if necessary).

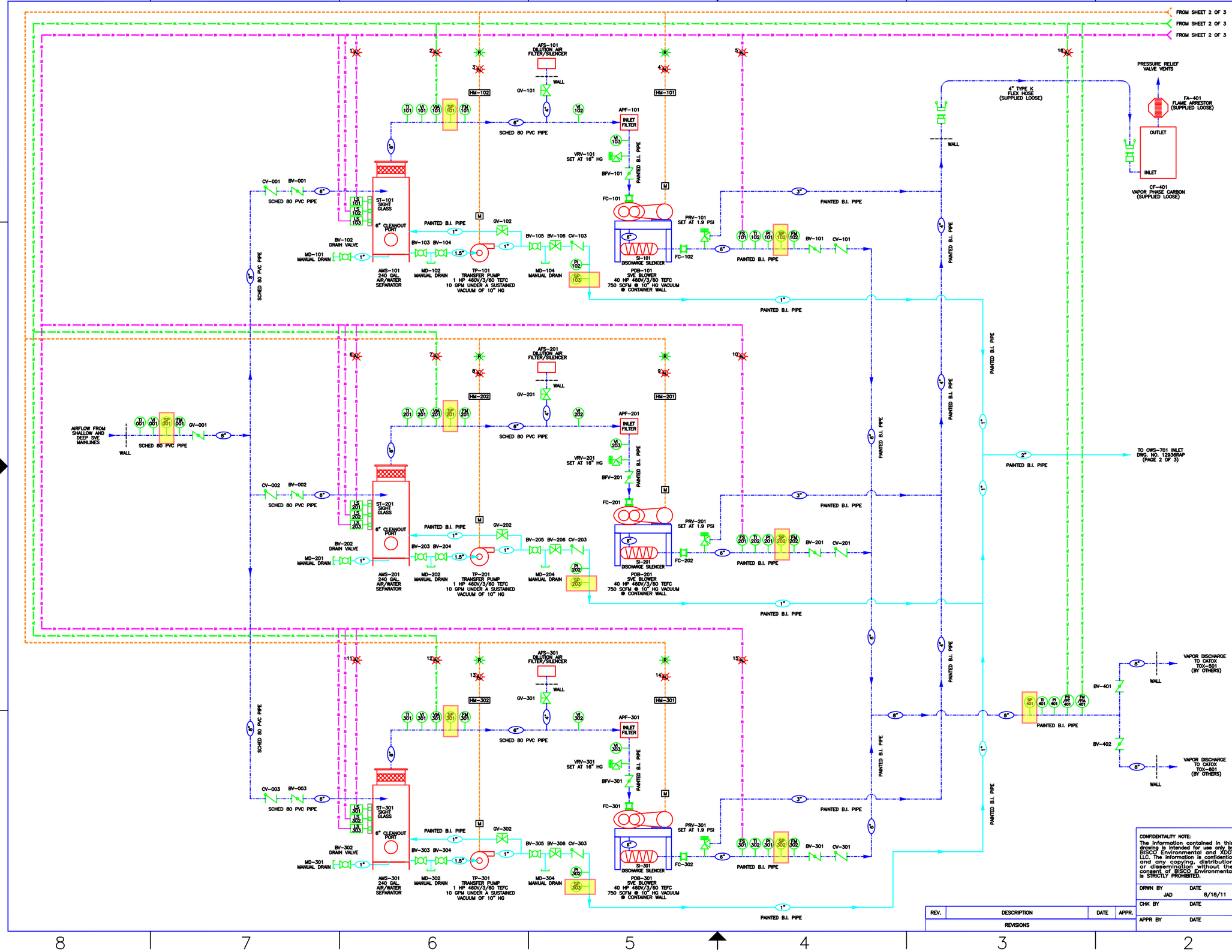
3.2 Procedure

1. Access the vapor probe, SVE well, or pre-/post carbon sample tubing. In some instances, a sampling valve may be provided.
2. Connect the sample pump inlet tubing to the sample port. If the sample port tubing size is not compatible with the sample pump tubing, use a small piece of Teflon or Tygon tubing as a connector.
3. Turn on the sample pump and purge the vapor probe tubing for 10 to 20 seconds (approximately 3 volumes). If the sample is collected from an operating system, then purging of the sampling lines is required. *If the soil vapor is known to contain high VOC concentrations, it is recommended that the purged air be discharged down-wind through a 10 to 15 foot length of 5/16" I.D. Tygon tubing. If the VOCs are known to exist at concentrations two orders of magnitude above the TLV, a hand held total hydrocarbon analyzer is to be used to monitor the ambient air. If ambient air concentrations exceed the TLV, take proper health and safety measures and/or revise the sampling method.*
4. Open the valve to the Tedlar sample bag and connect the Tedlar sample bag to the sample pump discharge tubing. The sample pump discharge tubing should be sized to snugly fit the sample bag stem.
5. Fill the sample bag (2/3 to 3/4 full) with the soil vapor from the vapor probe.
6. Stop the sample pump. Disconnect the sample bag from the sample pump discharge and purge the sample bag by gently squeezing the bag until it is empty. *Be sure to hold the sample bag such that the purged air is discharged downwind from yourself and other workers.*
7. Reconnect the Tedlar sample bag to the sample pump discharge tubing. Fill the sample bag to approximately 2/3 to 3/4 full.
8. Stop the sample pump, close the sample bag valve, and disconnect the sample bag from the sample pump discharge tubing.
9. Disconnect the sample pump inlet tubing from the vapor probe. Turn on the sample pump and run ambient air through the sample system in between samples in order to purge the sample pump and tubing of residual VOCs.

NOTE: To ensure that VOC cross-contamination is not occurring, periodically obtain and analyze ambient air samples from the sample pump in between vapor probe samples. This is to be done prior to obtaining the initial sample, and periodically thereafter, depending on the relative VOC concentrations, constituents and analytical methods being used to evaluate the soil vapor samples.

APPENDIX B

PIPING AND INSTRUMENTATION DIAGRAMS



LEGEND

AC	AIR COMPRESSOR
AFS	AIR FILTER/SILENCER
AMS	AIR MOISTURE SEPARATOR
APF	AIR PARTICULATE FILTER
BFV	BUTTERFLY VALVE
BPF	BAG PARTICULATE FILTER
BV	BALL VALVE
CF	CARBON FILTER (VAPOR PHASE)
CO	CLEAN OUT
CV	CHECK VALVE
DD	DRY DISCONNECT
FA	FLAME ARRESTOR
FC	FLEX CONNECTOR
FH	FLEXIBLE HOSE
FIA	FLOW INDICATOR ANALOG
FM	FLOW METER
FS	FLOW SWITCH
FT	FLOW TRANSMITTER
GV	GATE VALVE
HM	HOUR METER
LEL	LOWER EXPLOSIVE LIMIT
LGAC	LIQUID PHASE CARBON
LS	LEVEL SWITCH
M	MOTOR
MCP	MASTER CONTROL PANEL
MD	MANUAL DRAIN
NS	NAPL STORAGE
NT	NAPL TANK
OIT	OPERATOR INTERFACE TERMINAL
OWS	OIL WATER SEPARATOR
PDB	POSITIVE DISPLACEMENT BLOWER
PI	PRESSURE INDICATOR
PLC	PROGRAMMABLE LOGIC CONTROLLER
PS	PRESSURE SWITCH
PT	PRESSURE TRANSMITTER
PRV	PRESSURE RELIEF VALVE
QCV	QUICK CONNECT VALVE
SI	SILENCER
SP	SAMPLE PORT
ST	SITE TUBE
TI	TEMPERATURE INDICATOR
TIA	TEMPERATURE INDICATOR ANALOG
TOX	THERMAL OXIDIZER
TP	TRANSFER PUMP
TSH	TEMPERATURE SWITCH HIGH
VI	VACUUM INDICATOR
VIA	VACUUM INDICATOR ANALOG
VRV	VACUUM RELIEF VALVE
PS/PT	PRESSURE SWITCH / PRESSURE TRANSMITTER

BALL VALVE
BUTTERFLY VALVE
CHECK VALVE
GATE VALVE
RELIEF VALVE
VACUUM BREAKER
MALE CAMLOCK
FEMALE CAMLOCK
PLUGGED TEE (MANUAL DRAIN)
FLAME ARRESTER
ALARM LIGHT
WARNING LIGHT
EMERGENCY STOP
RUN LIGHT
AIR LINE
INTRINSICALLY SAFE ANALOG LINE
INTRINSICALLY SAFE LINE
CONTROL LINE
WATER LINE
BREAK

ALARMS

1. AIR MOISTURE SEPARATOR (AMS-101) HIGH HIGH LEVEL
2. SVE BLOWER (PDB-101) LOW VACUUM
3. TRANSFER PUMP (TP-101) MOTOR OVERLOAD
4. SVE BLOWER (PDB-101) MOTOR OVERLOAD
5. SVE BLOWER (PDB-101) LOW FLOW
6. AIR MOISTURE SEPARATOR (AMS-201) HIGH HIGH LEVEL
7. SVE BLOWER (PDB-201) LOW VACUUM
8. TRANSFER PUMP (TP-201) MOTOR OVERLOAD
9. SVE BLOWER (PDB-201) MOTOR OVERLOAD
10. SVE BLOWER (PDB-201) LOW FLOW
11. AIR MOISTURE SEPARATOR (AMS-301) HIGH HIGH LEVEL
12. SVE BLOWER (PDB-301) LOW VACUUM
13. TRANSFER PUMP (TP-301) MOTOR OVERLOAD
14. SVE BLOWER (PDB-301) MOTOR OVERLOAD
15. SVE BLOWER (PDB-301) LOW FLOW
16. COMBINED SVE SYSTEM DISCHARGE HIGH PRESSURE

REV.	DESCRIPTION	DATE	APPR.
	REVISIONS		

BISCO Environmental
Soil & Groundwater Remediation Equipment
Taunton, Massachusetts 02780

DRWN BY	JAD
DATE	8/18/11
CHK BY	
DATE	
APPR BY	
DATE	

TITLE
VAPOR EXTRACTION & AIR INJECTION SYSTEM
PROCESS & INSTRUMENTATION DIAGRAM

XDD, LLC.
W.G.KRUMMRICH FACILITY – SAUGET, IL

JOB NO. 12936

SCALE	SIZE	DWG NO.	SHEET	REV
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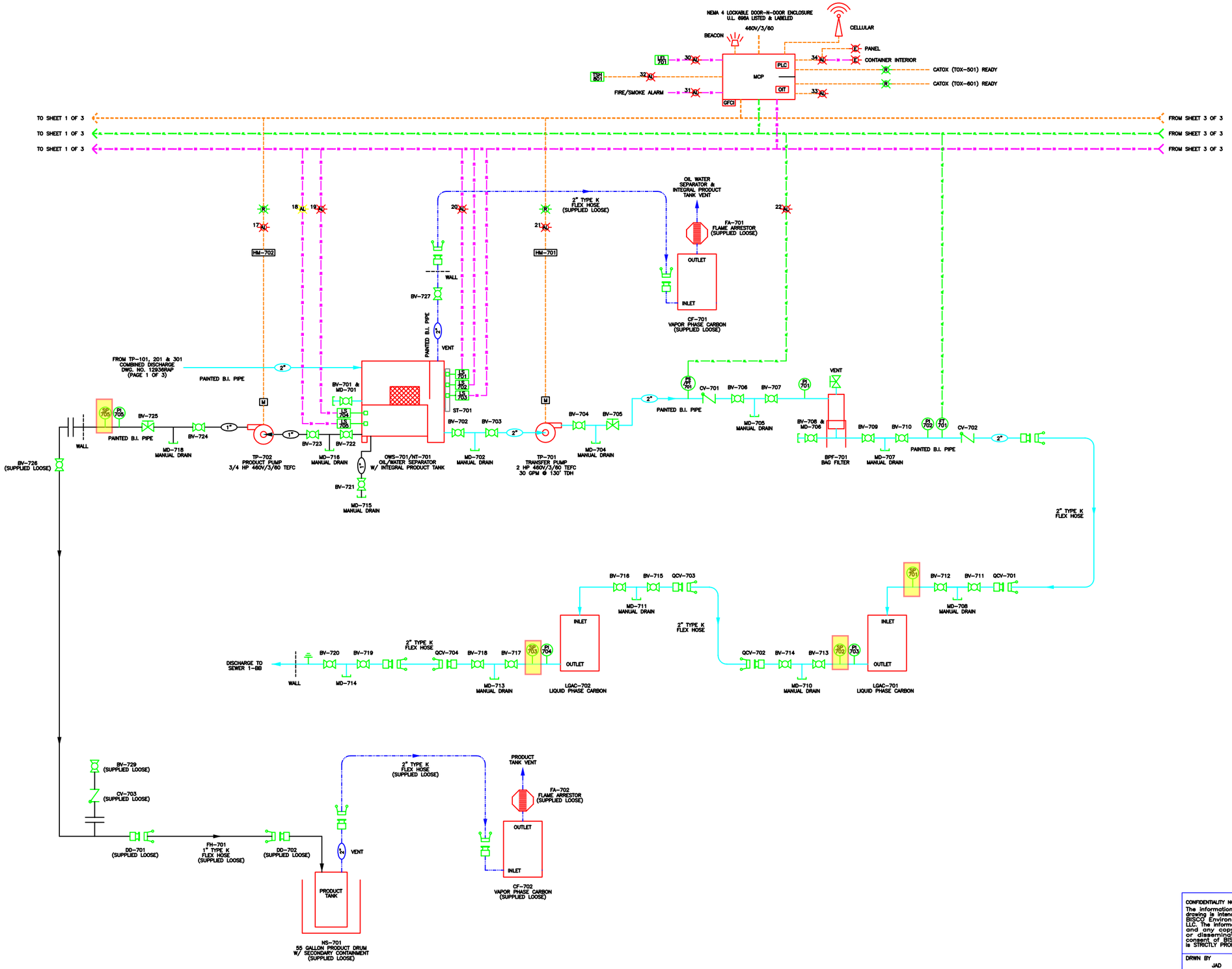


LEGEND

AC	AIR COMPRESSOR
AFS	AIR FILTER/SILENCER
AMS	AIR MOISTURE SEPARATOR
APF	AIR PARTICULATE FILTER
BFV	BUTTERFLY VALVE
BPF	BAG PARTICULATE FILTER
BV	BALL VALVE
CF	CARBON FILTER (VAPOR PHASE)
CO	CLEAN OUT
CV	CHECK VALVE
DD	DRY DISCONNECT
FA	FLAME ARRESTOR
FC	FLEX CONNECTOR
FH	FLEXIBLE HOSE
FIA	FLOW INDICATOR ANALOG
FM	FLOW METER
FS	FLOW SWITCH
FT	FLOW TRANSMITTER
GV	GATE VALVE
HM	HOUR METER
LEL	LOWER EXPLOSIVE LIMIT
LGAC	LIQUID PHASE CARBON
LS	LEVEL SWITCH
M	MOTOR
MCP	MASTER CONTROL PANEL
MD	MANUAL DRAIN
NS	NAPL STORAGE
NT	NAPL TANK
OIT	OPERATOR INTERFACE TERMINAL
OWS	OIL WATER SEPARATOR
PDB	POSITIVE DISPLACEMENT BLOWER
PI	PRESSURE INDICATOR
PLC	PROGRAMMABLE LOGIC CONTROLLER
PS	PRESSURE SWITCH
PT	PRESSURE TRANSMITTER
PRV	PRESSURE RELIEF VALVE
QCV	QUICK CONNECT VALVE
SI	SILENCER
SP	SAMPLE PORT
ST	SITE TUBE
TI	TEMPERATURE INDICATOR
TIA	TEMPERATURE INDICATOR ANALOG
TOX	THERMAL OXIDIZER
TP	TRANSFER PUMP
TSH	TEMPERATURE SWITCH HIGH
VI	VACUUM INDICATOR
VIA	VACUUM INDICATOR ANALOG
VRV	VACUUM RELIEF VALVE
PS/PT	PRESSURE SWITCH / PRESSURE TRANSMITTER

BALL VALVE
BUTTERFLY VALVE
CHECK VALVE
GATE VALVE
RELIEF VALVE
VACUUM BREAKER
MALE CAMLOCK
FEMALE CAMLOCK
PLUGGED TEE (MANUAL DRAIN)
FLAME ARRESTER
ALARM LIGHT
WARNING LIGHT
EMERGENCY STOP
RUN LIGHT
AIR LINE
INTRINSICALLY SAFE ANALOG LINE
INTRINSICALLY SAFE LINE
CONTROL LINE
WATER LINE
BREAK

ALARMS

17. TRANSFER PUMP (TP-702) MOTOR OVERLOAD
18. INTEGRAL PRODUCT TANK HIGH LEVEL WARNING
19. INTEGRAL PRODUCT TANK HIGH HIGH LEVEL
20. OIL WATER SEPARATOR (OWS-701) HIGH HIGH LEVEL
21. TRANSFER PUMP (TP-701) MOTOR OVERLOAD
22. BAG FILTER (BPF-701) HIGH PRESSURE
30. HIGH LEL
31. FIRE/SMOKE ALARM (FUTURE)
32. BUILDING INTERIOR HIGH TEMPERATURE
33. VOLTAGE FAULT
34. EMERGENCY STOP

REV.	DESCRIPTION	DATE	APPR.
REVISIONS			

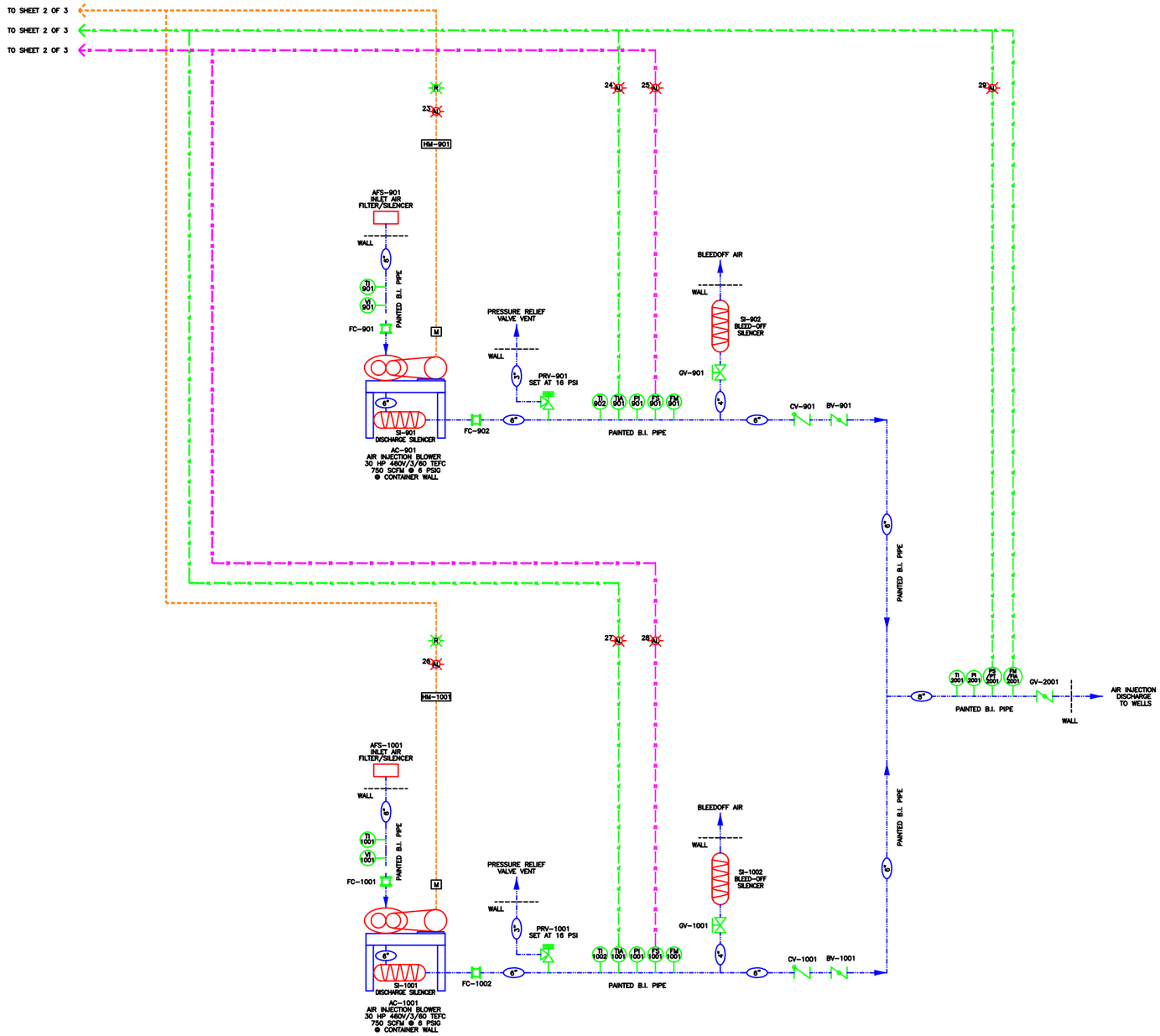
DRWN BY JAD DATE 8/18/11
CHK BY DATE
APPR BY DATE

BISCO Environmental
Soil & Groundwater Remediation Equipment
Taunton, Massachusetts 02780

TITLE
VAPOR EXTRACTION & AIR INJECTION SYSTEM
PROCESS & INSTRUMENTATION DIAGRAM

XDD, LLC.
W.G.KRUMMRICH FACILITY – SAUGET, IL

JOB NO. 12936
SCALE N/A | SIZE D | DWG NO. 12936RAP | SHEET 2 OF 3 | REV



LEGEND	
AC	AIR COMPRESSOR
AFS	AIR FILTER/SILENCER
AMS	AIR MOISTURE SEPARATOR
APF	AIR PARTICULATE FILTER
BFV	BUTTERFLY VALVE
BPF	BAG PARTICULATE FILTER
BV	BALL VALVE
CF	CARBON FILTER (VAPOR PHASE)
CO	CLEAN OUT
CV	CHECK VALVE
DD	DRY DISCONNECT
FA	FLAME ARRESTOR
FC	FLEX CONNECTOR
FH	FLEXIBLE HOSE
FIA	FLOW INDICATOR ANALOG
FM	FLOW METER
FS	FLOW SWITCH
FT	FLOW TRANSMITTER
GV	GATE VALVE
HM	HOUR METER
LEL	LOWER EXPLOSIVE LIMIT
LGAC	LIQUID PHASE CARBON
LS	LEVEL SWITCH
M	MOTOR
MCP	MASTER CONTROL PANEL
MD	MANUAL DRAIN
NS	NAPL STORAGE
NT	NAPL TANK
OIT	OPERATOR INTERFACE TERMINAL
OWS	OIL WATER SEPARATOR
PDB	POSITIVE DISPLACEMENT BLOWER
PI	PRESSURE INDICATOR
PLC	PROGRAMMABLE LOGIC CONTROLLER
PS	PRESSURE SWITCH
PT	PRESSURE TRANSMITTER
PRV	PRESSURE RELIEF VALVE
QCV	QUICK CONNECT VALVE
SI	SILENCER
SP	SAMPLE PORT
ST	SITE TUBE
TI	TEMPERATURE INDICATOR
TIA	TEMPERATURE INDICATOR ANALOG
TOX	THERMAL OXIDIZER
TP	TRANSFER PUMP
TSH	TEMPERATURE SWITCH HIGH
VI	VACUUM INDICATOR
VIA	VACUUM INDICATOR ANALOG
VRV	VACUUM RELIEF VALVE
PS/PT	PRESSURE SWITCH / PRESSURE TRANSMITTER

BALL VALVE
BUTTERFLY VALVE
CHECK VALVE
GATE VALVE
RELIEF VALVE
VACUUM BREAKER
MALE CAMLOCK
FEMALE CAMLOCK
PLUGGED TEE (MANUAL DRAIN)
FLAME ARRESTER
ALARM LIGHT
WARNING LIGHT
EMERGENCY STOP
RUN LIGHT
AIR LINE
INTRINSICALLY SAFE ANALOG LINE
INTRINSICALLY SAFE LINE
CONTROL LINE
WATER LINE
BREAK

ALARMS

23. AIR INJECTION BLOWER (AC-901) MOTOR OVERLOAD
24. AIR INJECTION BLOWER (AC-901) HIGH TEMPERATURE
25. AIR INJECTION BLOWER (AC-901) LOW FLOW
26. AIR INJECTION BLOWER (AC-1001) MOTOR OVERLOAD
27. AIR INJECTION BLOWER (AC-1001) HIGH TEMPERATURE
28. AIR INJECTION BLOWER (AC-1001) LOW FLOW
29. COMBINED AIR INJECTION SYSTEM HIGH PRESSURE

REV.	DESCRIPTION	DATE	APPR.
	REVISIONS		

BISCO Environmental
Soil & Groundwater Remediation Equipment
Taunton, Massachusetts 02780

DRWN BY	JAD
DATE	8/18/11
CHK BY	
DATE	
APPR BY	
DATE	

TITLE
VAPOR EXTRACTION & AIR INJECTION SYSTEM
PROCESS & INSTRUMENTATION DIAGRAM

XDD, LLC.
W.G.KRUMMRICH FACILITY – SAUGET, IL

JOB NO. 12936

SCALE	SIZE	DWG NO.	SHEET	REV
N/A	D	12936RAP	3 OF 3	

Motor Control Center
Power Input:
480V
60 Hz
3 Phase

Main Control Panel
Power Input:
120V
60 Hz

E-STOP

Dilution Air

To Atm.

Process Inlet:
10" Dia
150 lb Style Flange
2000 SCFM
@ Negative 2"-5" WC

Heat Exchange Mode
Thermal / Catalytic - Stack Position 2.

Blanking Plate
Heat Exchange
Bypass Mode

Exhaust to Atmosphere

Exhaust to Atmosphere

Sample Port

Heat Exchanger

Insulation

Duct Plug

Heat Exchange Bypass
Thermal Mode - Stack Position 1.

Blanking Plate
Heat Exchange Mode

Remove Duct Plug for
Heat Exchange Mode

System Air Proving Switch

0.4 to 4" WC

SP = 250°F

0 to 5" WC

0 to 10" WC

Flame Arrestor

Damper 1

Expansion Joint

High Limit

PROCESS BLOWER
20 HP

Damper 2

Remove Catalyst for
Thermal Mode Operation

Catalyst

2 to 20" WC

0-100% LEL
SP = 40% LEL

0-2 cf/hr

Combustion Air

0 to 20" WC

Expansion Joint

Calibration Gas Connection

Ignition Transformer

Spark Ignitor

Sight Glass

UV Flame Sensor

Burner

Combustion Chamber

(Spare)

Flame Safety Programmer

Pilot Gas

0 to 30" WC

To Atm.

Primary

Combustion Air Proving Switch

0.4 to 4" WC

MICRO RATIO VALVE

40 to 200" WC
SP = 100" WC

12 to 60" WC
SP = 14" WC

Vent Valve

Vent Valve

To Atm.

To Atm.

To Atm.

To Atm.

Secondary

Main Gas

Blocking Valve

Main Shut Off

Primary

0 to 15 PSIG

2" FNPT

Gas Meter Totalizer

Trap

Shipped Loose

3" 150# Flange inlet and outlet

COMBUSTION BLOWER
10 HP

To Atm.

Electrical Input- 480VAC/60Hz /3 Phase

Input BTU Rating - 6,000,000 BTU/Hour

Max. Process Inlet Flow Rate - 2000 SCFM

Catalyst Volume - 3.0 Cubic Feet

Stack Outer Diameter - 27.8 Inches

Stack Inner Diameter - 27.66 Inches

Discharge Height - 20 Feet AGL

Site Elevation - 500 ft

Fuel:
Natural Gas
or Propane
5 PSIG Operating
to 7 PSIG Maximum

Note: Damper Valve Positions
Heat Exchange Mode - Damper 1 - Open, Damper 2 - Closed
Heat Exchange Bypass Mode - Damper 1 - Closed, Damper 2 - Open

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1198-001

TITLE: 2000 CFM Thermal/Catalytic Oxidizer
INSTRUMENTATION LAYOUT

DRAWING NO. N-11-1198-001
SCALE: NTS
REV. D
DRAWN: SAL 5/15/2018
CHECKED:
APPROVED:
CUSTOMER: XDD Solutia
W. G. Krummerich Facility, Sauget, IL
LAST DRAWN BY: Admin 7/26/2011
SOURCE:
PAGE 1 OF 3
LAST PLOTTED BY: XXX DATE: XXX
FILE NO.

Measured Variable		Succeding Letters		Valve Bodies		Miscellaneous		Operators	
A	Analysis	A	Alarm		Valve (N.O. or Unspecified)		Centrifugal Blower	M	Electric Motor
F	Flow Rate	C	Controller		Valve Normally Closed			S	Solenoid
I	Current	D	Differential		Ball Valve		Flow Orifice		Manual
L	Level	E	Primary Element		Check Valve			S/G	Sight Glass
M	Motor	H	High		Butterfly Valve		Electrical Line		
P	Pressure	I	Indicator		Damper		Pneumatic Line		
T	Temperature	L	Light		Orifice Valve		Process Ductwork		Vortex Damper
Z	Position	L	Low				Panel Mounted Instrument		
		R	Record		Pressure Regulator (Self Contained)		Locally Mounted Instrument		Filter
		S	Switch				Mounted Inside Panel	AI	Analog Input
		T	Transmitter		Pressure Regulator (External Backload)		Alarm	DI	Digital Input
		V	Valve				Transition	I	Interlock
				T	Sample Port				

REV	BY	DATE	CHANGE
D	SAL	12-5-11	UPDATE AS BUILT
C	SAL	10-11-11	REMOVE FE 3 STACK FLOW ELEMENT. ADD DPT 1 TO TAKE DIFF PRESSURE ACROSS BURNER ORIFICE TO DETERMINE COMBUSITON AIR FLOW
B	SAL	8-31-11	UPDATE PER CUSTOMER COMMENTS.
A	SAL	8-30-11	UPDATE GAS METER POSITION

Revision Table

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1198-001

TITLE: 2000 CFM Thermal/Catalytic Oxidizer
INSTRUMENTATION LEGEND

DRAWING NO.: N-11-1198-001
SCALE: NTS
DRAWN: SAL DATE: 8/5/2011
CHECKED: DATE:
APPROVED: DATE:
LAST DRAWN BY: Admin DATE: 7/5/2011
CUSTOMER: XDD Solutio
SOURCE: W. G. Krummrich Facility, Sauget, IL
REV: D
PAGE: 2 OF 3
LAST PLOTTED BY: XXX DATE: XXX
FILE PATH

MODE	OXIDIZER READY CONTACT	OXIDIZER ALARM CONTACT	INLET VALVE M5	DILUTION VALVE M6	SYSTEM FAN M1	SYSTEM FAN SPEED CONTROL SC1	OXIDIZER COMBUSTION BLOWER M2	FIRING RATE VALVE M3	FUEL TRAIN BLOCKING VALVES S1,S2,S3,S4	FUEL TRAIN VENT VALVES S5,S6	OXIDIZER FLAME SAFETY FSP1
SYSTEM STOP	DISABLED	ENABLED	CLOSED	OPEN	DISABLED	DISABLED	DISABLED	CLOSED	CLOSED	OPEN	DISABLED
SYSTEM START PURGE	DISABLED	DISABLED	CLOSED	OPEN	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	CLOSED	CLOSED	OPEN	DISABLED
SYSTEM PRE-HEAT	DISABLED	DISABLED	CLOSED	OPEN	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	OPEN	OPEN	CLOSED	ENABLED
SYSTEM RUN	ENABLED	DISABLED	OPEN	ENABLED	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	OPEN	OPEN	CLOSED	ENABLED
SYSTEM COOL-DOWN	DISABLED	DISABLED	CLOSED	OPEN	ENABLED 240 SEC.	PRE-SET VALUE 2000 SCFM	ENABLED 240 SEC.	CLOSED	CLOSED	OPEN	DISABLED
SYSTEM ALARM	DISABLED	ENABLED	CLOSED	OPEN	ENABLED 240 SEC.	PRE-SET VALUE 2000 SCFM	ENABLED 240 SEC.	CLOSED	CLOSED	OPEN	DISABLED
POWER FAULT	DISABLED	ENABLED	CLOSED	OPEN	DISABLED	DISABLED	DISABLED	CLOSED	CLOSED	OPEN	DISABLED

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1198-001

TITLE: 2000 CFM Thermal/Catalytic Oxidizer
MODE CHART

DRAWING NO.: N-11-1198-001
SCALE: NTS
DRAWN: SAL DATE: 4/20/11
CHECKED: DATE:
APPROVED: DATE:
CUSTOMER: KDD Solutia
W. G. Krummerich Facility, Sauget, IL
REV.: D
LAST DRAWN BY: Aamir DATE: 7/20/2011
SOURCE:
PAGE: 3 OF 3
LAST PLOTTED BY: XXX DATE: XXX
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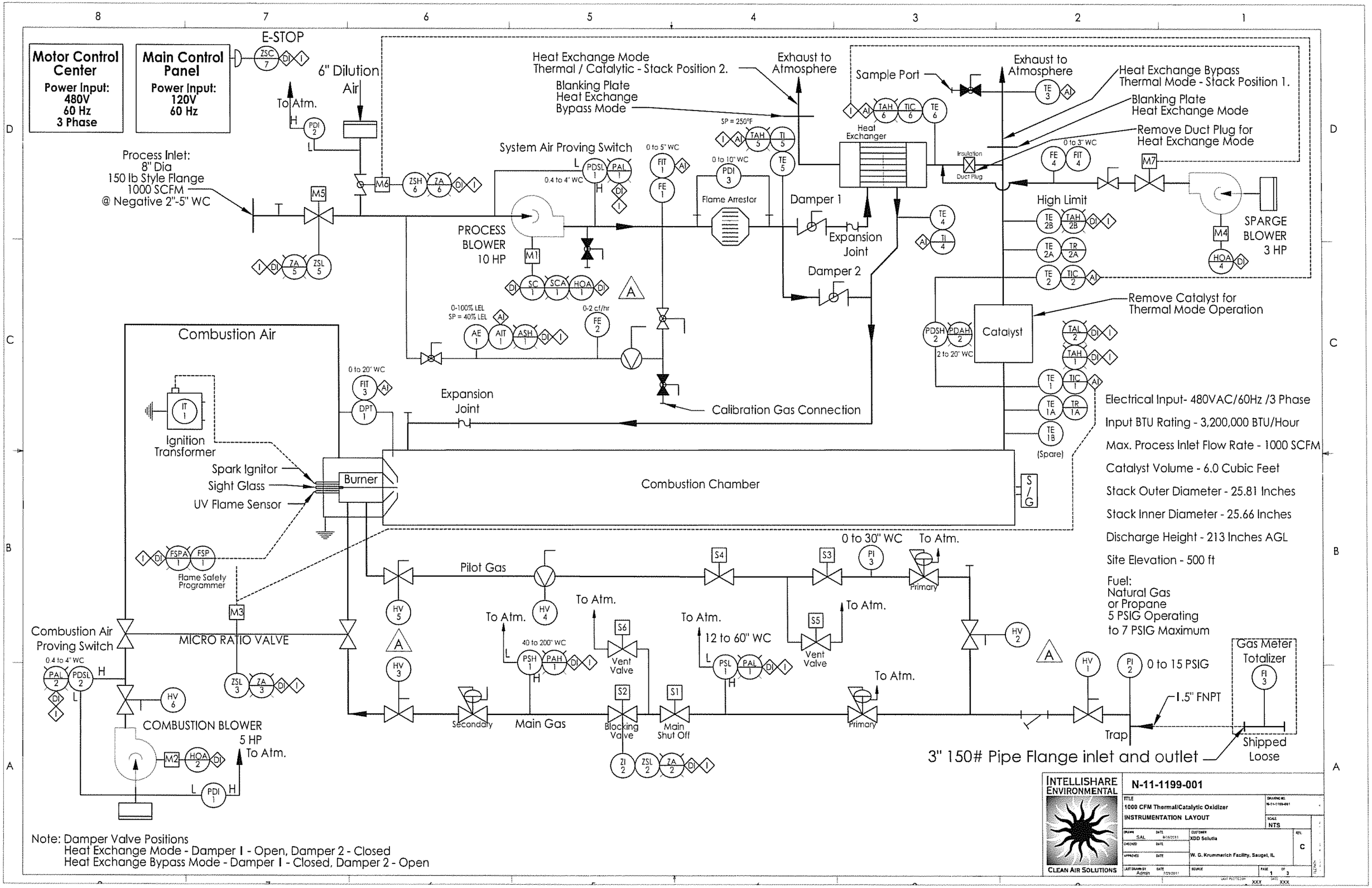


Note: Damper Valve Positions
Heat Exchange Mode - Damper 1 - Open, Damper 2 - Closed
Heat Exchange Bypass Mode - Damper 1 - Closed, Damper 2 - Open

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1199-001

1000 CFM Thermal/Catalytic Oxidizer
INSTRUMENTATION LAYOUT

SCALE: NTS
DRAWN: SAL 8/16/2011
CUSTOMER: XDD Solutia
REV: C
W. G. Krummerich Facility, Sauget, IL
LAST DRAWN BY: Admin 7/29/2011
SOURCE
PAGE 1 OF 3
LAST PLOTTED BY: XXX DATE: XXX

Measured Variable		Succeding Letters		Valve Bodies		Miscellaneous		Operators	
A	Analysis	A	Alarm		Valve (N.O. or Unspecified)		Centrifugal Blower	M	Electric Motor
F	Flow Rate	C	Controller		Valve Normally Closed			S	Solenoid
I	Current	D	Differential		Ball Valve		Flow Orifice		Manual
L	Level	E	Primary Element		Check Valve			S/G	Sight Glass
M	Motor	H	High		Butterfly Valve		Electrical Line		
P	Pressure	I	Indicator		Damper		Pneumatic Line		
T	Temperature	L	Light		Orifice Valve		Process Ductwork		Vortex Damper
Z	Position HV 2	L	Low				Panel Mounted Instrument		
		R	Record		Pressure Regulator (Self Contained)		Locally Mounted Instrument		Filter
		S	Switch				Mounted Inside Panel	AI	Analog Input
		T	Transmitter		Pressure Regulator (External Backload)		Alarm	DI	Digital Input
		V	Valve				Transition	I	Interlock
				T	Sample Port				

REV	BY	DATE	CHANGE
C	SAL	12-9-11	UPDATE AS BUILT
B	SAL	10-11-11	REMOVE FE 3 STACK FLOW ELEMENT. ADD DPT 1 TO TAKE DIFF PRESSURE ACROSS BURNER ORIFICE TO DETERMINE COMBUSITON AIR FLOW.
A	SAL	9-2-11	UPDATE GAS METER POSITION

Revision Table

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1199-001

TITLE: 1000 CFM Thermal/Catalytic Oxidizer
INSTRUMENTATION LEGEND

DRAWING NO.: N-11-1199-001
SCALE: NTS
REV: C
DRAWN: SAL DATE: 8/8/2011
CHECKED: DATE:
APPROVED: DATE:
CUSTOMER: XDD Solutia
W. G. Krummerich Facility, Sauget, IL
LAST DRAWN BY: ASHH DATE: 10/6/2011
SOURCE:
PAGE: 2 OF 3
LAST PLOTTED BY: XXX DATE: XXX
FILE PATH

MODE	OXIDIZER READY CONTACT	OXIDIZER ALARM CONTACT	INLET VALVE M5	DILUTION VALVE M6	SYSTEM FAN M1	SYSTEM FAN SPEED CONTROL SC1	OXIDIZER COMBUSTION BLOWER M2	FIRING RATE VALVE M3	HEAT EX. SPARGE BLOWER M4	FUEL TRAIN BLOCKING VALVES S1,S2,S3,S4	FUEL TRAIN VENT VALVES S5,S6	OXIDIZER FLAME SAFETY FSP1
SYSTEM STOP	DISABLED	ENABLED	CLOSED	OPEN	DISABLED	DISABLED	DISABLED	CLOSED	DISABLED	CLOSED	OPEN	DISABLED
SYSTEM START PURGE	DISABLED	DISABLED	CLOSED	OPEN	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	CLOSED	ENABLED	CLOSED	OPEN	DISABLED
SYSTEM PRE-HEAT	DISABLED	DISABLED	CLOSED	OPEN	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	OPEN	ENABLED	OPEN	CLOSED	ENABLED
SYSTEM RUN	ENABLED	DISABLED	OPEN	ENABLED	ENABLED	PRE-SET VALUE 2000 SCFM	ENABLED	OPEN	ENABLED	OPEN	CLOSED	ENABLED
SYSTEM COOL-DOWN	DISABLED	DISABLED	CLOSED	OPEN	ENABLED 240 SEC.	PRE-SET VALUE 2000 SCFM	ENABLED 240 SEC.	CLOSED	ENABLED 240 SEC.	CLOSED	OPEN	DISABLED
SYSTEM ALARM	DISABLED	ENABLED	CLOSED	OPEN	ENABLED 240 SEC.	PRE-SET VALUE 2000 SCFM	ENABLED 240 SEC.	CLOSED	ENABLED 240 SEC.	CLOSED	OPEN	DISABLED
POWER FAULT	DISABLED	ENABLED	CLOSED	OPEN	DISABLED	DISABLED	DISABLED	CLOSED	DISABLED	CLOSED	OPEN	DISABLED

INTELLISHARE ENVIRONMENTAL
CLEAN AIR SOLUTIONS

N-11-1199-001

TITLE: 1000 CFM Thermal/Catalytic Oxidizer
MODE CHART

DRAWING NO.: N-11-1199-001
SCALE: NTS
REV.: C
DRAWN: SAL DATE: 4/2011
CHECKED: DATE:
APPROVED: DATE:
CUSTOMER: XDD Solutia
W. G. Krummerich Facility, Sauget, IL
LAST DRAWN BY: Admin DATE: (10:00:)
SOURCE:
PAGE: 3 OF: 3
LAST PLOTTED BY: XXX DATE: XXX
FILEPATH:

APPENDIX C

AIR EMISSION PERMIT

ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

1021 NORTH GRAND AVENUE EAST, P.O. BOX 19506, SPRINGFIELD, ILLINOIS 62794-9506 - (217) 782-2113

PAT QUINN, GOVERNOR LISA BONNETT, INTERIM DIRECTOR

217/782-2113

CONSTRUCTION PERMIT

PERMITTEE

Solutia Inc. - W.G. Krummrich Plant
Attn: Brett Shank
500 Monsanto Avenue
Sauget, Illinois 62206

Application No.: 11040037 I.D. No.: 163121AAC
Applicant's Designation: SVE Date Received: April 20, 2011
Subject: Soil Vapor Extraction System - Former Benzene Storage & Pipeline Area
Date Issued: May 17, 2011
Location: 500 Monsanto Avenue, Sauget

Permit is hereby granted to the above-designated Permittee to CONSTRUCT emission source(s) and/or air pollution control equipment consisting of an in-situ soil vapor extraction system with oxidizers, as described in the above-referenced application. This Permit is subject to standard conditions attached hereto and the following special condition(s):

1a. This permit authorizes construction of an in-situ soil vapor extraction system (the affected system) that will be used for a soil remediation project at the former benzene storage and pipeline area at the source. Vapor extraction wells will be installed for applying vacuum to the contaminated soil within this area with selected wells periodically used for air injection to promote air flow. Groundwater extracted by the system will be processed using an air stripper.

Extracted vapor and the vapor discharge of the air stripper will be ducted to oxidizer(s) to control emissions of volatile organic material (VOM). When remediation begins, two oxidizers may be used in parallel to handle the volume of extracted vapor.

b. This permit only authorizes the treatment of extracted vapor and groundwater due to the affected system's operation for in-situ treatment of soil that is in its original location and will remain in that location following treatment, except for soil displaced from drilling wells, which must be appropriately handled.

2. This permit only authorizes in-situ treatment of soils that are only contaminated with volatile and semi-volatile organic materials.

Note: Soils classified as hazardous waste pursuant to Section 3.15 of the Illinois Environmental Protection Act (Act) cannot be treated without written approval from the Illinois EPA, Bureau of Land, including permits in accordance with Section 21(f) of the Act. The Permittee must make the determination that the soil to be treated is non-hazardous using the criteria under 35 IAC 721.111.

3a. This remediation project is subject to 35 IAC 212.301, which provides that no person shall cause or allow any visible emissions of fugitive particulate matter from any process, including any material handling or storage activity beyond the property line of the source.

b. i. The affected system is subject to the control requirements of 35 IAC 219, Subpart TT, so that the oxidizers must achieve at least 81 percent reduction in VOM emissions.

ii. The affected system is subject to 35 IAC 219.301 and 219.302, which requires that emissions of organic materials be no more than 8 pounds per hour or be controlled by at least 85 percent.

4a. This permit is issued based on the affected system not being subject to the National Emission Standard for Hazardous Air Pollutants (NESHAP) for Site Remediation, 40 CFR 63, Subpart GGGGG. This is because the project meets the exemption under this NESHAP for remedial activities conducted under the federal Resource Conservation and Recovery Act (RCRA). In particular, this project is being undertaken pursuant to an Administrative Order on Consent issued to the Permittee by USEPA pursuant to RCRA (R8H-5-00-003; May 3, 2000).

b. The Permittee shall comply with 35 IAC 219.105(d)(2)(A), which requires use of an approved continuous monitoring system that is installed, calibrated, maintained and operated according to vendor specification at all times an oxidizer is in use. For a thermal oxidizer, the continuous monitoring equipment shall monitor the combustion chamber temperature of the oxidizer. For a catalytic oxidizer, the equipment shall monitor the temperature rise across each catalytic afterburner bed or VOM concentration of the exhaust.

5. At all times that the affected system is generating vapors, emissions shall be controlled by an oxidizer. An oxidizer shall operate at a temperature of at least 1450°F (thermal) and in catalytic mode at 600°F.

6a. Emissions of nitrogen oxides (NO_x), carbon monoxide (CO), and volatile organic material (VOM) from the affected system shall not exceed the following limits:

Pollutant	(Lbs/Hour)	(Tons/Year)
NO_x	0.8	3.5
CO	0.4	1.5
VOM	2.4	10.4

b. This permit is issued based on negligible emissions of particulate matter and sulfur dioxide from the affected system. For this purpose, emissions of each pollutant shall not exceed nominal emission rates of 0.1 lb/hour and 0.44 tons/year.

c. This permit is issued based on the emissions of hazardous air pollutants (HAPs) from the affected system being less than 7.9 tons per

year of any single HAP and 10.4 tons per year of any combination of HAPs.

8a. The affected system and associated oxidizers shall be operated in accordance with good air pollution control practice to minimize emissions.

b. The affected system shall not begin operation until construction, including construction of the oxidizers, is complete, and reasonable measures short of actual operation have been taken to verify proper operation.

c. In the event that the operation of the affected system and any associated activities results in an odor nuisance, the Permittee shall take all appropriate and necessary actions, including but not limited to, reduction in the operating rate, in order to eliminate the nuisance.

9. The Permittee shall maintain records of the following items:

a. A file containing the manufacturer's specifications for the performance of each oxidizer, percent destruction or exhaust concentration for hydrocarbons, and recommended operating and maintenance procedures.

b. i. Operating hours of the affected system (hours/month); and

ii. Amount of groundwater processed (gallons/month).

c. The following records for the operation and maintenance of the affected system:

i. An operating log or other records for the affected system that at a minimum shall address:

Any unusual occurrences during each malfunction of the affected system that significantly impairs emission performance, including the nature and duration of the event, corrective actions taken, any deviations from the established procedures for such a malfunction, and preventative actions taken to address similar events.

ii. Inspection, maintenance and repair log(s) for the affected system that at a minimum shall identify such activities that are performed related to components that may affect emissions; the reason for such activities, i.e., whether planned or initiated due to a specific event or condition; and any failure to carry out the established maintenance procedures, with explanation.

d. Records for any period during which the affected system deviated from an applicable requirement.

e. Records of the VOM and HAP emissions of the affected system (tons/month and tons/year), with supporting calculations.

10. All records and logs required by this permit shall be retained at a readily accessible location at the source for at least three years from the date of entry and shall be made available for inspection and copying by the Illinois EPA upon request. Any records retained in an electronic format (e.g., computer) shall be capable of being retrieved and printed on paper during normal source office hours so as to be able to respond to an Illinois EPA request for records during the course of a source inspection.

11a. The Permittee shall promptly notify the Illinois EPA of deviations of the affected system from requirements set by this permit as follows. These notifications shall provide for each such incident, a description of the incident, the date and duration of the incident, and whether it occurred during startup, malfunction, breakdown, or shutdown.

i. Failure of the affected system, (including associated control systems) that is accompanied by the direct release of emissions of VOM to the atmosphere shall be reported within 15 days.

ii. Other deviations shall be reported in a quarterly report.

b. With these quarterly reports the Permittee shall also report any deviations from applicable compliance procedures for the affected system established by this permit, including specifying periods during which the continuous monitoring system was not in operation.

12. Two copies of required reports and notifications concerning equipment operation or repairs, performance testing or a continuous monitoring system shall be sent to:

Illinois Environmental Protection Agency
Division of Air Pollution Control
Compliance Section (#40)
P.O. Box 19276
Springfield, Illinois 62794-9276

and one copy shall be sent to the Illinois EPA's regional office at the following address unless otherwise indicated:

Illinois Environmental Protection Agency
Division of Air Pollution Control
2009 Mall Street
Collinsville, Illinois 62234

13. Operation of the affected system, as addressed by this Construction Permit, is authorized until the CAAPP permit for this source is revised or renewed, to address this system.

If you have any questions on this permit, please contact Bob Smet at 217/782-2113.

Edwin C. Bakowski

Edwin C. Bakowski, P.E.
Manager, Permit Section
Division of Air Pollution Control

Date Signed: May 17, 2011

ECB:RPS:jws

cc: Region 3
T. Dragovich, DLPC

STATE OF ILLINOIS
ENVIRONMENTAL PROTECTION AGENCY
DIVISION OF AIR POLLUTION CONTROL
P. O. BOX 19506
SPRINGFIELD, ILLINOIS 62794-9506

STANDARD CONDITIONS FOR CONSTRUCTION/DEVELOPMENT PERMITS ISSUED BY THE ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

July 1, 1985

The Illinois Environmental Protection Act (Illinois Revised Statutes, Chapter 111-1/2, Section 1039) authorizes the Environmental Protection Agency to impose conditions on permits which it issues.

The following conditions are applicable unless susperseded by special condition(s).

1. Unless this permit has been extended or it has been voided by a newly issued permit, this permit will expire one year from the date of issuance, unless a continuous program of construction or development on this project has started by such time.

2. The construction or development covered by this permit shall be done in compliance with applicable provisions of the Illinois Environmental Protection Act and Regulations adopted by the Illinois Pollution Control Board.

3. There shall be no deviations from the approved plans and specifications unless a written request for modification, along with plans and specifications as required, shall have been submitted to the Agency and a supplemental written permit issued.

4. The permittee shall allow any duly authorized agent of the Agency upon the presentation of credentials, at reasonable times:

   a. to enter the permittee's property where actual or potential effluent, emission or noise sources are located or where any activity is to be conducted pursuant to this permit,

   b. to have access to and to copy any records required to be kept under the terms and conditions of this permit,

   c. to inspect, including during any hours of operation of equipment constructed or operated under this permit, such equipment and any equipment required to be kept, used, operated, calibrated and maintained under this permit,

   d. to obtain and remove samples of any discharge or emissions of pollutants, and

   e. to enter and utilize any photographic, recording, testing, monitoring or other equipment for the purpose of preserving, testing, monitoring, or recording any activity, discharge, or emission authorized by this permit.

5. The issuance of this permit:

   a. shall not be considered as in any manner affecting the title of the premises upon which the permitted facilities are to be located,

   b. does not release the permittee from any liability for damage to person or property caused by or resulting from the construction, maintenance, or operation of the proposed facilities,

   c. does not release the permittee from compliance with other applicable statutes and regulations of the United States, of the State of Illinois, or with applicable local laws, ordinances and regulations,

   d. does not take into consideration or attest to the structural stability of any units or parts of the project, and

e. in no manner implies or suggests that the Agency (or its officers, agents or employees) assumes any liability, directly or indirectly, for any loss due to damage, installation, maintenance, or operation of the proposed equipment or facility.

6. a. Unless a joint construction/operation permit has been issued, a permit for operation shall be obtained from the Agency before the equipment covered by this permit is placed into operation.

   b. For purposes of shakedown and testing, unless otherwise specified by a special permit condition, the equipment covered under this permit may be operated for a period not to exceed thirty (30) days.

7. The Agency may file a complaint with the Board for modification, suspension or revocation of a permit:

   a. upon discovery that the permit application contained misrepresentations, misinformation or false statements or that all relevant facts were not disclosed, or

   b. upon finding that any standard or special conditions have been violated, or

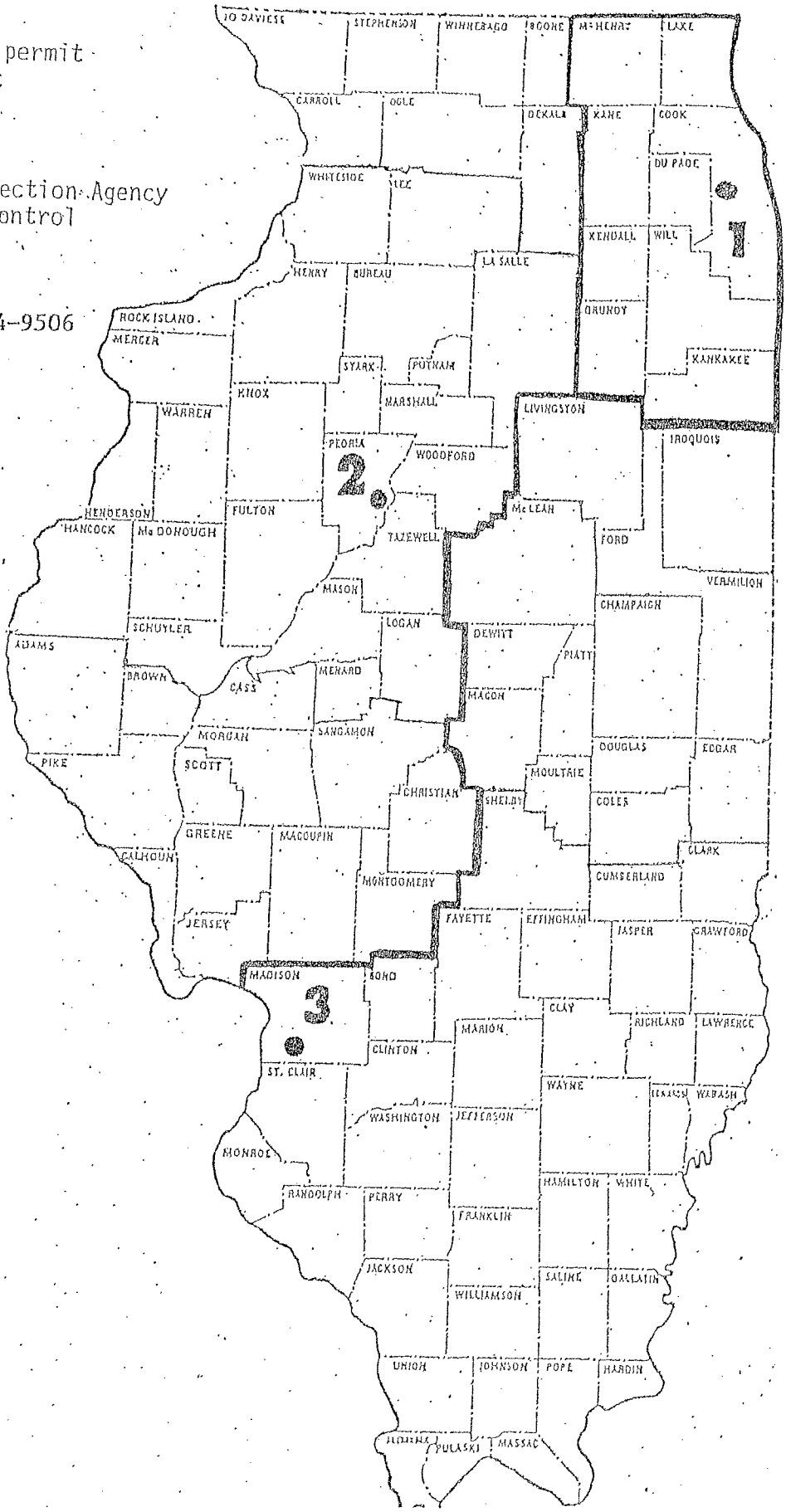
   c. upon any violations of the Environmental Protection Act or any regulation effective thereunder as a result of the construction or development authorized by this permit.

ENVIRONMENTAL PROTECTION AGENCY
BUREAU OF AIR

For assistance in preparing a permit application contact the Permit Section.

Illinois Environmental Protection Agency
Division of Air Pollution Control
Permit Section
1021 N. Grand Ave E.
P.O.Box 19506
Springfield, Illinois 62794-9506

or a regional office of the Field Operations Section. The regional offices and their areas of responsibility are shown on the map. The addresses and telephone numbers of the regional offices are as follows:



Illinois EPA
Region 1
Bureau of air, FOS
9511 West Harrison
Des Plaines, Illinois 60016
847/294-4000

Illinois EPA
Region 2
5415 North University
Peoria, Illinois 61614
309/693-5463

Illinois EPA
Region 3
2009 Mall Street
Collinsville, Illinois 62234
618/346-5120

APPENDIX D

WASTEWATER DISCHARGE PERMIT

AMERICAN BOTTOMS
REGIONAL WASTEWATER TREATMENT FACILITY
1 AMERICAN BOTTOMS ROAD
SAUGET, ILLINOIS 62201-1075

(618) 337-1710
FAX (618) 337-8919

CERTIFIED MAIL & ELECTRONIC MAIL
7004 2510 0000 6909 1705

January 5, 2012

Mr. Brett Shank
Environmental Specialist
Solutia Inc. - W.G. Krummrich Plant
500 Monsanto Avenue
Sauget, Illinois 62206-1198

Subject: Discharge Permit No. 10C-105

Dear Mr. Shank:

Attached is the permit modification for the purpose of incorporating changes to pretreatment permit limitations at your facility. The final permit is issued pursuant to the e-mail response you sent on December 30, 2011, waiving the thirty day comment period. We agree to the changes regarding the typographical errors that you pointed out. We also agree to your suggestion of making the samples for semi-volatile and chloride analysis a grab sample with the understanding that the samples will be collected during a batch discharge so that they are representative of the daily discharge. We have added additional language to clarify that if multiple grabs all grabs will be weighted equally in the preparation of an in-lab site composite sample. In your e-mail you asked us to provide clarification on the acceptable analytical methods to be performed for chloride. The acceptable methods can be found in 40 CFR 136 Table 1B. See the table below for acceptable chloride methods.

Parameter	Methodology[58]	Reference (method number or page)					
		EPA[35,52]	Standard methods (18th, 19th)	Standard methods (20th)	Standard methods online	ASTM	USGS/AOAC/other
Chloride, mg/L	Titrimetric: (silver nitrate) or		4500–Cl–B	4500–Cl–B	4500–Cl–B–97	D512–89(99) (B)	I–1183–85[2]
	(Mercuric nitrate)		4500–Cl–C	4500–Cl–C	4500–Cl–C–97	D512–89 (99) (A)	973.51[3], I–1184–85[2]

	Colorimetric: manual or						I–1187–85[2]
	Automated (Ferricyanide)		4500–Cl–E	4500–Cl–E	4500–Cl–E–97		I–2187–85[2]
	Potentiometric Titration		4500–Cl–D	4500–Cl–D	4500–Cl–D–97		
	Ion Selective Electrode					D512–89(99)(C)	
	Ion Chromatography	300.0, Rev 2.1 (1993) and 300.1, Rev 1.0 (1997)	4110 B	4110 B	4110 B–00	D4327–97, 03	993.30[3]

American Bottoms laboratory has the capability to run chloride tests if Solutia would like to utilize the service.

We are reviewing your e-mail and determining if we have enough information to approve the design of the pretreatment systems. We may request additional information to determine if the pretreatment system is sufficient in design.

All discharges from this facility and related actions and reports shall be in accordance with the terms and conditions of this permit and the Ordinance. If you wish to appeal any effluent limitations, pretreatment requirements, or conditions imposed in this wastewater discharge permit, a written notice of appeal should be filed within thirty days after the effective date of the permit. Your written notice of appeal, if filed, should be mailed or delivered to:

Village Clerk
Village of Sauget
2897 Falling Springs Road
Sauget, Illinois 62206

If you have any questions related to this permit, please call me at 337-9749.

Sincerely,

Adam Rossel
Pretreatment Coordinator

AMERICAN BOTTOMS
REGIONAL WASTEWATER TREATMENT FACILITY
1 AMERICAN BOTTOMS ROAD
SAUGET, ILLINOIS 62201-1075

(618) 337-1710
FAX (618) 337-8919

CERTIFIED MAIL & ELECTRONIC MAIL
RETURN RECIEPT REQUESTED
7004 1350 0005 0762 4376

May 7, 2012

Mr. Brett Shank
Environmental Specialist
Solutia Inc. - W.G. Krummrich Plant
500 Monsanto Avenue
Sauget, Illinois 62206-1198

Subject: Typographical Error in Discharge Permit No. 10C-105

Dear Mr. Shank:

While reviewing your permit, 10C-105, we discovered a typographical error in Part 4.E.6. This part describes the monitoring parameters and frequencies for the site remediation locations, SVE Site-13 and TSVE Site-14. It was our intention to require sampling and testing for benzene at both locations, but only require testing for acrolein at Site 14. According to the permit Solutia was required to test for acrolein at Site 13, but testing for benzene was not required. Benzene is the main constituent of concern at Site 13 so it is important that monitoring for this parameter occurs.

Please see the attached permit that corrects the typographical errors described above and incorporates our original intentions and.

We apologize for any confusion. Please contact me at (618) 337-9749 or at Adamr@AmericanBottoms.com with any comments or questions regarding this matter.

Sincerely,

Adam Rossel
Pretreatment Coordinator

VILLAGE OF SAUGET

AMERICAN BOTTOMS REGIONAL

WASTEWATER TREATMENT FACILITY

WASTEWATER DISCHARGE PERMIT

FOR

SOLUTIA INC.

PERMIT NO. 10C-105

AMERICAN BOTTOMS
REGIONAL WASTEWATER TREATMENT FACILITY
1 AMERICAN BOTTOMS ROAD
SAUGET, ILLINOIS 62201-1075

(618) 337-1710
FAX (618) 337-8919

May 7, 2012

Mr. Wojciech Klim
Plant Manager
Solutia Inc. - W.G. Krummrich Plant
500 Monsanto Avenue
Sauget, Illinois 62206-1198

Wastewater Discharge Permit No. 10C-105

Dear Sirs:

In accordance with all the terms and conditions of Ordinance 632 of the Village of Sauget; the 1977 Regional Agreement as amended; Section 46 of the Illinois Environmental Protection Act of 1970 (Ill. Rev. Stat. 1987. Ch. 1111/2, Sec. 1046) as amended; and Ill. Rev. Stat. 1987, Ch 24, Sec. 11-141-7; permission is hereby granted to Solutia Inc., operating under the Standard Industrial Classification (SIC) Code Nos. 2819, 2842, and 2869, and subject to the National Categorical Pretreatment Standard (NCPS) No. 40 CFR 414, Subpart H, to discharge industrial wastewater into sewer lines tributary to the American Bottoms Regional Wastewater Treatment Plant (ABRWTP) in accordance with and subject to the provisions of attached American Bottoms Regional Wastewater Discharge Permit No. 10C-105 ("Permit").

This Permit is modified to correct a typographical error in permit 10C-105. This Permit modification is in accordance with the Village of Sauget Ordinance 632 Part 4, Section 4.10. Please affix the modified sections to your current permit.

Nothing herein shall be construed as a permit or as permission for the permittee to violate the provisions of any sewer use ordinance in effect within the jurisdiction of any unit of local government in which the permittee's facility is located.

This Permit revision will expire on March 1, 2015.

VILLAGE OF SAUGET

By: Heather Taraghes
Executive Director

Solutia Permit Modification
Permit 10C-105
PART 4 - EFFLUENT LIMITATIONS, SAMPLING AND TESTING REQUIREMENTS

A. Local Limits: The Village of Sauget reserves the right, in Ordinance 632 and as amended, to establish limitations or requirements on discharges to the wastewater disposal system if deemed necessary to comply with the objectives presented in Section 1.4 of the Ordinance.

Parameter	Limitations	Effective Date
Total Kjeldahl Nitrogen (TKN)	9,000 lbs/day (7-day Moving average)	November 1, 2010

Total Kjeldahl Nitrogen (TKN) is the concentration or mass of Nitrogen, as measured by an analytical method for TKN approved by USEPA in 40 CFR 136.

TKN Temporary Variance

A temporary variance may be granted by the General Manager when necessary to allow a Discharge of TKN at levels in excess of the discharge limitations in section 4.A, above, up to a maximum TKN discharge of 15,000 lbs/day. A TKN temporary variance may be granted for a period of not more than two consecutive calendar days. The TKN temporary variance is for the purpose of addressing temporary shutdowns and start-ups of industrial production processes that generate an elevated Discharge of TKN at the Solutia – W.G. Krummrich Plant. A TKN temporary variance does not authorize a Bypass of pretreatment units.

The decision whether to grant a TKN temporary variance shall be within the sole discretion of the General Manager, using best professional judgment and in consideration of acceptable ambient and operational conditions, including but not limited to, the availability of adequate storage capacity at the POTW for the TKN discharge.

A TKN temporary variance may be requested by prior verbal notice to the General Manager and shall be confirmed in writing. No discharge above the TKN limitations in section 4.A, above, is authorized without the prior approval of the General Manager or his designee.

TKN temporary variances shall be limited to a maximum duration of 12 discharges within any 360-day period. The amount of TKN discharged during the authorized period of a TKN temporary variance will not be used for purposes of determining compliance with the TKN limitations in section 4.A, above.

B. State Limits: These limits are stated in 35 Ill. Adm. Code Part 307. This Part 307 places restrictions on the types, concentrations, and quantities of contaminants which can be discharged into sewer systems in the State.

Limitations (mg/l)

Parameters	Monthly Avg. (mg/l)	Daily Composite (mg/l)	Grab sample (mg/l)	Applicable Monitoring Location
Mercury	0.003	0.006	0.015	1AA
Cyanide (Total)	--	--	10	1AA

Any sample tested shall not release more than 2 mg/l of cyanide when tested at a pH of 4.5 and at a temperature of 66°C (150°F) for a period of 30 minutes.

The mercury limitation shown is the alternate limitation based on 35 Illinois Adm. Code 307.1102(c). Subject to the averaging rule of Ill Adm. Code 304.104, the monthly average shall be the numerical average of all daily composites taken during a calendar month. A monthly average must be based on at least three daily composites.

C. Site Specific Limits: [RESERVED]

D. National Categorical Pretreatment Standards (NCPS):Solutia's operations are subject to 40 CFR 414 - Organic Chemicals, Plastics, and Synthetic Fibers (OCPSF), Subpart H (Specialty Organic Chemicals), which has a final compliance date for Pretreatment Standards for Existing Sources (PSES) of November 5, 1990. Sources must comply with 40 CFR Part 403 and achieve discharges not exceeding the quantity (mass) determined by multiplying the process wastewater flow subject to the OCPSF regulation times the concentration listed in the regulation, subject to application of the Combined Wastestream Formula pursuant to 40 CFR part 403.6(e). The Village of Sauget, as the local Control Authority, hereby exercises its discretion to calculate and apply a fixed alternative mass limit applicable to all regulated and unregulated process flows as set forth below. The applicable regulatory concentration reference values and the mass limits for your facility, determined by using average flow data you have previously provided, are set forth as follows:

NCPS - 40 CFR 414	Regulatory Concentration Reference Values (ug/l)		Plant Mass Limits (lbs/day)	
Parameter	Max. One Day	Max. Mo. Avg.	Max. One Day	Max. Mo. Avg.
1,1,1-Trichloroethane	59	22	0.19	0.069
1,1,2-Trichloroethane	127	32	0.398	0.10

Solutia Permit Modification
Permit 10C-105

NCPS - 40 CFR 414

Parameter	Regulatory Concentration Reference Values (ug/l)		Plant Mass Limits (lbs/day)	
	Max. One Day	Max. Mo. Avg.	Max. One Day	Max. Mo. Avg.
1,1-Dichloroethane	59	22	0.19	0.069
1,1-Dichloroethene	60	22	0.19	0.069
1,2,4-Trichlorobenzene	794	196	0.245	0.0604
1,2-Dichlorobenzene	794	196	2.49	0.615
1,2-Dichloroethane	574	180	1.80	0.564
1,2-Dichloropropane	794	196	2.49	0.615
1,2-trans-Dichloroethene	66	25	0.21	0.078
1,3-Dichlorobenzene	380	142	1.19	0.445
1,3-Dichloropropene	794	196	2.49	0.615
1,4-Dichlorobenzene	380	142	1.19	0.445
2-Nitrophenol	231	65	0.0712	0.020
4,6-Dinitro-o-cresol	277	78	0.0854	0.024
4-Nitrophenol	576	162	0.178	0.0499
Acenaphthene	47	19	0.014	0.0059
Anthracene	47	19	0.014	0.0059
Benzene	134	57	0.420	0.18
Bis (2-ethylhexyl) phthalate	258	95	0.0795	0.029
Carbon Tetrachloride	380	142	1.19	0.445
Chlorobenzene	380	142	1.19	0.445
Chloroethane	295	110	0.925	0.345
Chloroform	325	111	1.02	0.348
Di-n-butyl phthalate	43	20	0.013	0.0062
Diethyl phthalate	113	46	0.0348	0.014
Dimethyl phthalate	47	19	0.014	0.0059
Ethylbenzene	380	142	1.19	0.445
Fluoranthene	54	22	0.017	0.0068
Fluorene	47	19	0.014	0.0059
Hexachloroethane	794	196	0.245	0.0604
Hexachlorobenzene	794	196	0.245	0.0604
Hexachlorobutadiene	380	142	0.117	0.0438
Methyl Chloride	295	110	0.925	0.345
Methylene Chloride	170	36	0.533	0.11
Naphthalene	47	19	0.014	0.0059
Nitrobenzene	6402	2237	1.974	0.6897

Effective May 7, 2012

Solutia Permit Modification
Permit 10C-105

NCPS - 40 CFR 414	Regulatory Concentration Reference Values (ug/l)		Plant Mass Limits (lbs/day)	
Parameter	Max. One Day	Max. Mo. Avg.	Max. One Day	Max. Mo. Avg.
Phenanthrene	47	19	0.014	0.0059
Pyrene	48	20	0.015	0.0062
Tetrachloroethene	164	52	0.514	0.16
Toluene	74	28	0.23	0.088
Trichloroethylene	69	26	0.22	0.082
Vinyl Chloride	172	97	0.539	0.30
Total Cyanide	1200	420	N/A	N/A
Total Lead	690	320	N/A	N/A
Total Zinc	2610	1050	N/A	N/A

1. The Regulatory Concentration Reference Values are included in this Permit for reference purposes only. An exceedance of a Regulatory Concentration Value, without an exceedance of the corresponding mass limit, shall not be deemed a violation of this Permit.

2. The volatile mass limits are based on an average flow from regulated and non-regulated processes of 0.376 MGD; the semi-volatile mass limits are based on an average flow from regulated processes of 0.0370 MGD. The mass limits are calculated using the Combined Wastestream Formula as defined in 40 CFR 403.6(e)(1)(i).

3. Cyanide, lead and zinc are not regulated in OCPSF regulated streams at Solutia due to the absence of cyanide and metal-bearing waste streams as listed in 40 CFR 414.

4. Compliance with each OCPSF parameter standard will be determined by adding the mass loadings contributed by individual processes at five (5) locations designated as sites 8, 9, 10, 11, and, for volatile parameters, ACL site 12 in Part 4 - Section E.5. of this Permit.

5. The monthly average shall be the numerical average of the results of all sampling events during a calendar month. If only one sampling event occurs during a given month, the monthly average limits will apply to the results of that single sampling event.

6. Mass limits are subject to change based on changes in process flows. The regulated facility shall notify the POTW in writing at least sixty (60) days prior to any proposed material and/or substantial changes in process flows to allow for the calculation of revised limits.

E. Monitoring Schedule

1. The monitoring schedule requirements are required as of the effective date of this Permit.

2. Monitoring locations are shown in the attached diagram - Figure 1, which is incorporated into and made a part of this Permit.

3. Monitoring frequencies:

Parameter (Units)	Monitoring Location	Monitoring Frequency	Sample Type
TKN (mg/L)	1-AA	See 4. below	Composite
Mercury (mg/L)	1-AA	1/6-months	Composite
Cyanide (mg/L)	1-AA	See 4. below	Grab
Flow (mgd)	1-AA	Daily	Meter
Flow (gpd)	13	Daily	Meter
Flow (gpd)	14	Daily	Meter

4. Compliance with the TKN local limit and the cyanide state limit will be determined by sampling and testing performed by the POTW. Permittee self-monitoring for TKN and cyanide is not required.

5. Monitoring frequencies for OCPSF regulated monitoring locations and parameters are as follows:

OCPSF Parameters	Sample Type	8	9	10	11	12
1,1,1-Trichloroethane	grab	M	M	M	M	M
1,1,2-Trichloroethane	grab	M	M	M	M	M
1,1-Dichloroethane	grab	M	M	M	M	M
1,1-Dichloroethene	grab	M	M	M	M	M
1,2,4-Trichlorobenzene	comp	M	M	M	M	-
1,2-Dichlorobenzene	grab	M	M	M	M	M
1,2-Dichloroethane	grab	M	M	M	M	M
1,2-Dichloropropane	grab	M	M	M	M	M
1,2-Trans-Dichloroethene	grab	M	M	M	M	M
1,3-Dichlorobenzene	grab	M	M	M	M	M
1,3-Dichloropropene	grab	M	M	M	M	M
1,4-Dichlorobenzene	grab	M	M	M	M	M
2-Nitrophenol	comp	M	M	M	M	-
4,6-Dinitro-o-cresol	comp	M	M	M	M	-

Solutia Permit Modification
Permit 10C-105

OCPSF Parameters	Sample Type	8	9	10	11	12
4-Nitrophenol	comp	M	M	M	M	-
Acenaphthene	comp	M	M	M	M	-
Anthracene	comp	M	M	M	M	-
Benzene	grab	M	M	M	M	M
Bis (2-ethylhexyl) phthalate	comp	M	M	M	M	-
Carbon Tetrachloride	grab	M	M	M	M	M
Chlorobenzene	grab	M	M	M	M	M
Chloroethane	grab	M	M	M	M	M
Chloroform	grab	M	M	M	M	M
Di-n-butyl phthalate	comp	M	M	M	M	-
Diethyl phthalate	comp	M	M	M	M	-
Dimethyl phthalate	comp	M	M	M	M	-
Ethylbenzene	grab	M	M	M	M	M
Fluoranthene	comp	M	M	M	M	-
Fluorene	comp	M	M	M	M	-
Hexachloroethane	comp	M	M	M	M	-
Hexachlorobenzene	comp	M	M	M	M	-
Hexachlorobutadiene	comp	M	M	M	M	-
Methyl Chloride	grab	M	M	M	M	M
Methylene Chloride	grab	M	M	M	M	M
Naphthalene	comp	M	M	M	M	-
Nitrobenzene	comp	M	M	M	M	-
Phenanthrene	comp	M	M	M	M	-
Pyrene	comp	M	M	M	M	-
Tetrachloroethene	grab	M	M	M	M	M
Toluene	grab	M	M	M	M	M
Trichloroethylene	grab	M	M	M	M	M
Vinyl Chloride	grab	M	M	M	M	M

Monitoring locations are identified as follows:
KEY M = Monthly Q= Quarterly '-'= Not Required

8-4ADPA/Santoflex – Sump
9-Santoflex pastilles – Trench 12–ACL
10-Laboratory East
11-Laboratory West

Effective May 7, 2012

6. Monitoring frequencies for site remediation monitoring locations and parameters are as follows:

Remediation Parameters	Sample Type	13	14
1,2-Dichlorobenzene	Grab	M	M
1,2,4-Trichlorobenzene	*	M	M
1,3-Dichlorobenzene	Grab	M	M
1,4-Dichlorobenzene	Grab	M	M
2,4,5-Trichlorophenol	*	M	M
2,4,6-Trichlorophenol	*	M	M
2,4-Dichlorophenol	*	M	M
2-Chloroaniline	*	M	M
2-Chlorophenol	*	M	M
2-Nitroaniline	*	M	M
4-Nitroaniline	*	M	M
Aniline	*	M	M
Acrolein	Grab	-	M
Benzene	Grab	M	M
Chlorobenzene	Grab	M	M
Ethylbenzene	Grab	M	M
Naphthalene	*	M	M
Pentachlorophenol	*	M	M
Phenol	*	M	M
Toluene	Grab	M	M
Chloride	*	-	M

*Single grabs taken during a batch discharge may be used for semi-volatile analysis at the SVE Site-13 and TSVE Site-14 remediation sites. Where more than one grab sample is taken, all grabs will be weighted equally in the preparation of an in-lab site composite sample.

13-Soil Vapor Extraction Site (SVE)
14-Thermally Enhanced Soil Vapor Extraction Site (TSVE)

7. Time Compositing - Twenty-four hour time-composited site samples are required for all semi-volatile analysis. Single grabs may be used for volatile analysis. Where more than one grab sample is taken, all grabs will be weighted equally in the preparation of an in-lab site composite sample.

8. Site Compositing - For OCPSF compliance monitoring, sampling shall be conducted and monitoring results obtained from sites 8, 9, 10, 11 and, for volatile parameters ACL site

12, except for those sites which are not discharging on the sampling date. The individual site samples may either be analyzed as individual sites or as a flow-weighted laboratory composite of the individual sites samples.

For compliance monitoring at SVE Site 13 and TSVE Site 14 remediation sites, sampling must be conducted at each individual site. The SVE Site 13 and TSVE Site 14 samples shall be analyzed separately.

9. OCPSF Flow Monitoring Reporting

a. OCPSF Monitoring Events

For each OCPSF self-monitoring event, the monthly self-monitoring report shall contain the following information:

i. Sites 8 and 9 – Date, Time, depth measurement(s) (inches or feet), corresponding gallons per minute (gpm), and corresponding gallons per day (gpd);
ii. Sites 10 and 11 – Estimated average flow in gpm and gpd;
iii. Site 12
-Measured or calculated total site 12 gallons per minute and corresponding gallons per day;
-Internal flow meter measurements and calculated internal flows used in the calculation of the daily total site 12 flow;
-Other data and estimations used to calculate internal flows used in the calculation of the daily site 12 flow;
-Formulae used in the determination of calculated internal flows and total daily site 12 flow.

b. Upon request, Solutia shall provide within 24 hours the site 12 flow data information identified in 8.a.iii of this paragraph for POTW OCPSF events. This flow data information shall also be included in the Solutia monthly self-monitoring report.

c. OCPSF Monthly Flow Summary Report

The monthly self-monitoring report shall include a summary of OCPSF flow information for the calendar month including the following:

i. The monthly average total flows and dilution flows for sites 8 and 9;
ii. The monthly average total flows for sites 10 and 11;
iii. The monthly average total site 12 flow and individual dilution flows, including supporting information as follows:
-Internal flow meter measurements and calculated internal flows used in the calculation of the total monthly average site 12 flow and monthly average dilution flows;

-Other data and estimations used to calculate internal flows used in the calculation of the monthly average site 12 flow and monthly average dilution flows;
-Formulae used in the determination of calculated internal flows and total monthly average site 12 flow and monthly average dilution flows

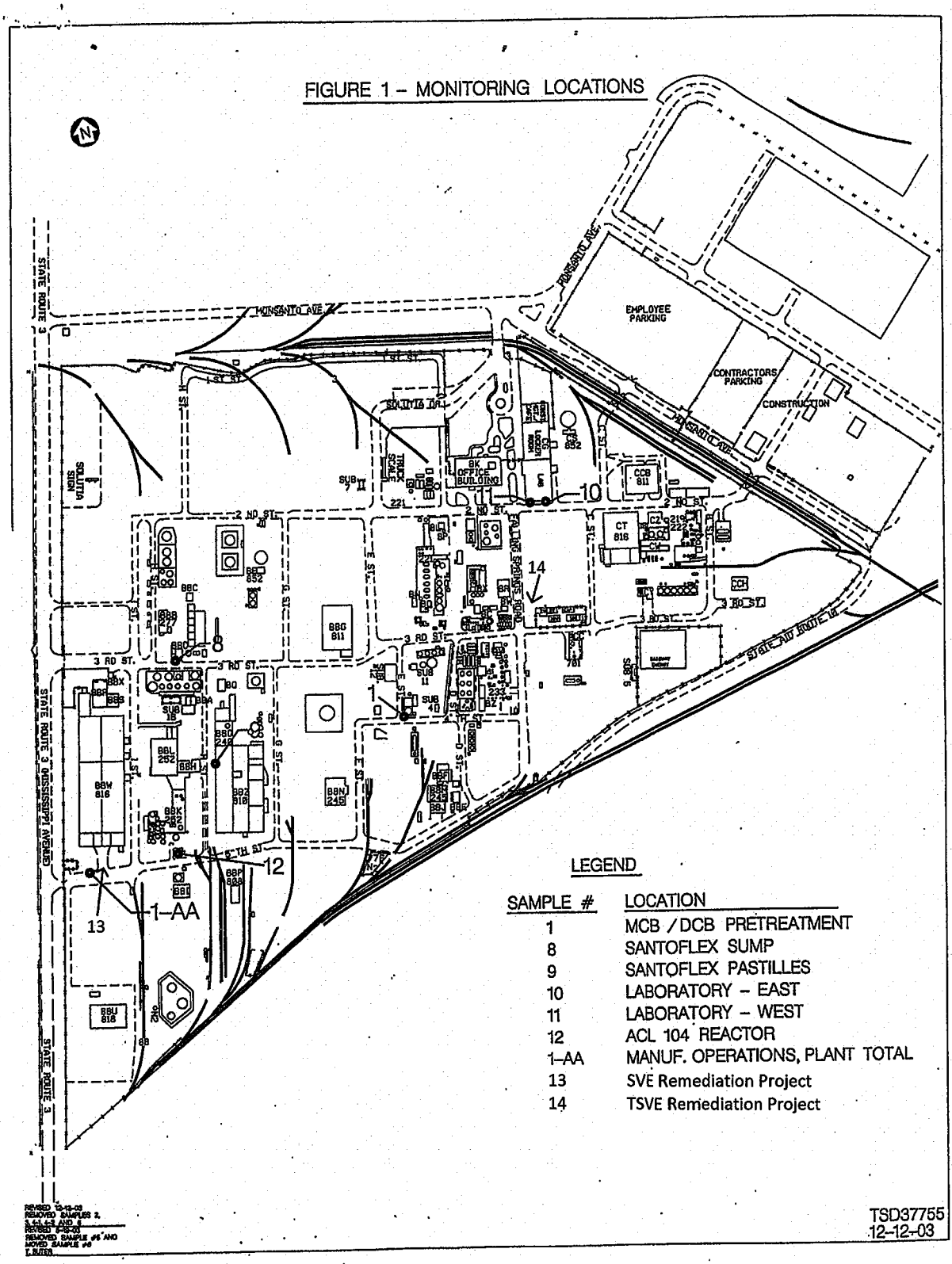


FIGURE 1 – MONITORING LOCATIONS
EMPLOYEE PARKING
CONTRACTORS PARKING
CONSTRUCTION
MONSANTO AVE.
SOLUTIA INC.
STATE ROUTE 3
LEGEND
SAMPLE # LOCATION
1 MCB / DCB PRETREATMENT
8 SANTOFLEX SUMP
9 SANTOFLEX PASTILLES
10 LABORATORY – EAST
11 LABORATORY – WEST
12 ACL 104 REACTOR
1-AA MANUF. OPERATIONS, PLANT TOTAL
13 SVE Remediation Project
14 TSVE Remediation Project
TSD37755
12-12-03

AMERICAN BOTTOMS REGIONAL TREATMENT PLANT
INDUSTRIAL WASTEWATER DISCHARGE PERMIT
FACT SHEET-PERMIT MODIFICATION
FULL-SCALE SVE AND TSVE REMEDIATION PROJECTS
Date: January 5, 2012

Section 1 Description of the Permittee

Facility Name: **Solutia Inc. W.G. Krummrich Plant**
Facility Site Address: **500 Monsanto Avenue, Sauget, IL 62206**
Facility Mailing Address: **Same**

Total Number of Monitoring connections: **8**

Monitoring Location	Reg. Process Y/N	Unregulated Process Y/N	Dilution Y/N	Locally regulated Y/N
1-AA	**N**	**N**	**N**	**Y**
Site 8-OCPSF	**Y**	**N**	**Y**	**N**
Site 9-OCPSF	**Y**	**N**	**Y**	**N**
Site 10-OCPSF	**Y**	**N**	**N**	**N**
Site 11-OCPSF	**Y**	**N**	**N**	**N**
Site 12-OCPSF	**N**	**Y**	**Y**	**N**
Site 13-SVE	**N**	**Y**	**N**	**N**
Site 14-TSVE	**N**	**Y**	**N**	**N**

Brief description of the facility processes: **Organic and inorganic chemical manufacturing operations whose products are marketed worldwide, supporting the automotive, water treatment, commercial dyes and agricultural industries.**

MODIFICATION – Solutia intends to perform two full-scale remediation projects at the W.G. Krummrich site in Sauget, IL. The basis for these remediation projects are the pilot projects that were done at the facility. Soil vapor extraction (SVE) will be used to remediate the area that formerly contained the "Big Mo" benzene storage tank. Thermally enhanced soil vapor extraction (TSVE) will be used to remediate the former chlorobenzene manufacturing area. These remediation activities are not subject to 40 CFR 414 OCPSF manufacturing category that many of Solutia's operations are subject to. Therefore, the discharges from the remediation activities are not subject to the categorical limits.

The groundwater collected in the SVE area may contain volatile organic compounds (VOC) and non-aqueous phase liquids (NAPL). An oil/water separator will be utilized to remove NAPL, which will be collected and stored in 55-gallon drums for disposal. The effluent from the oil/water separator will be treated using granular activated carbon (GAC) prior to discharge to the sewer.

The vapors collected in the TSVE area may contain benzene and chlorinated volatile organic compounds (CVOC). A thermal oxidizer will be used to treat the CVOC vapors, per an IEPA air permit, producing HCL vapor. The HCL will be removed prior to atmospheric discharge using a wet scrubber. The wet scrubber employs a water spray and packed bed media to strip the hydrogen chloride from the thermal oxidizer effluent vapor stream. The resulting HCL will be discharged to the process sewer. The groundwater collected may contain CVOC and NAPL. The groundwater will be treated with a oil-water separator and air stripper prior to discharge to the sewer. The NAPL collected in the oil/water separator will be collected and stored in 55-gallon drums for offsite disposal.

Categorical Industry: Yes **X** No____
If Yes, 40 CFR 414 Subpart(s) H
SIC Code(s): **2869, 2842, and 2819**

Section 2 **Type and Quantity of Discharge - NO CHANGES TO OCPSF SITES 8, 9, 10, 11 or 12;**

The flow of the SVE remediation project is estimated at 7200 gpd with a maximum discharge of 7,200 gpd. Approximately 28,800 gpd from the wet scrubber and 7200 gpd from the air-stripper is expected for a total of 36,000 gpd from the TSVE project. Due to the treatment processes planned at these sites, very low concentrations of organics are expected from either location. The discharge from the remediation activities will flow through the fenceline location, 105 1-AA, compliance with local limits will be addressed during routine sampling at 1-AA.

Section 3 **Rationale for Permit Limits – NO CHANGES TO THIS SECTION**

Section 4 **Special Conditions**

Groundwater Remediation Projects

- The groundwater from the SVE project must be treated by oil/water separator and granular activated carbon prior to discharge to the POTW. Other wastewater generated from the SVE project groundwater sampling and equipment decontamination wastewater from remediation activities must also be treated by the oil/water separator and granular activated carbon prior to discharge.

- The vapors generated from the TSVE project will be treated using a thermal oxidizer resulting in the generation of HCl vapor. The HCl will be removed from the vapor using a wet-scrubber and discharged to the sewer. Other wastewater generated from the TSVE project, sampling and equipment decontamination wastewater from remediation activities will be treated with an oil/water separator and air-stripper prior to discharge.

- The discharge of wastewater from the SVE project without oil/water separation and granular activated carbon treatment is subject to the bypass provisions of Section 3.9 of Ordinance 632.

- The discharge of wastewater from the TSVE project without oil/water separation and air-stripping is subject to the bypass provisions of Section 3.9 of Ordinance 632.

- The discharge of condensed vapor generated at the TSVE project without thermal oxidation and wet-scrubbing is subject to the bypass provisions of Section 3.9 of Ordinance 632.

- Solutia must maintain onsite and make available for inspection records of the wastestreams collected and shipped offsite for disposal.

- Solutia must submit discharge logs in its monthly monitoring report showing the days and volume of discharge from the SVE and TSVE remediation projects.

- No discharge shall occur from the SVE project until Solutia has received written approval from the Pretreatment Coordinator and installed the pretreatment system, flow monitoring device and sampling location to be used.

- No discharge shall occur from the TSVE project until Solutia has received written approval from the Pretreatment Coordinator and installed of the pretreatment system, flow monitoring device and sampling location to be used.

Solutia Permit Modification
For Remediation Projects

January 4, 2012

Section 5 **Monitoring Requirements – NO CHANGES TO OCPSF OR 1AA FENCELINE MONITORING.**

MODIFICATIONS FOR REMEDIATION PROJECTS INCLUDE:

Monitoring Location: **105-13 SVE Project and 105-14 TSVE Project**
Monitoring Frequencies: **Monitoring of the SVE project discharge must be conducted monthly during any calendar month during which a discharge occurs at the SVE project.**

Monitoring of the TSVE project discharge must be conducted monthly during any calendar month during which a discharge occurs at the TSVE project.

Monitoring Parameters:

Parameter	Sample Type	13	14
1,2-Dichlorobenzene	Grab	M	M
1,2,4-Trichlorobenzene	*	M	M
1,3-Dichlorobenzene	Grab	M	M
1,4-Dichlorobenzene	Grab	M	M
2,4,5-Trichlorophenol	*	M	M
2,4,6-Trichlorophenol	*	M	M
2,4-Dichlorophenol	*	M	M
2-Chloroaniline	*	M	M
2-Chlorophenol	*	M	M
2-Nitroaniline	*	M	M
4-Nitroaniline	*	M	M
Aniline	*	M	M
Acrolein	Grab	M	M
Benzene	Grab	-	M
Chlorobenzene	Grab	M	M
Ethylbenzene	Grab	M	M
Naphthalene	*	M	M
Pentachlorophenol	*	M	M
Phenol	*	M	M
Toluene	Grab	M	M
Chloride	*	-	M

***Single grabs taken during a batch discharge shall be used for semi-volatile analysis at the SVE Site-13 and TSVE Site-14 remediation sites. Where more than one grab sample is taken, all grabs will be weighted equally in the preparation of an in-lab site composite.**

Rationale for Monitoring Frequency:
Monitoring compounds were chosen based on the expected presence as indicated by Solutia in the application and the detection during the pilot plant discharges. It is the POTW's policy to require monthly monitoring for all parameters from new discharges.

AMERICAN BOTTOMS
REGIONAL WASTEWATER TREATMENT FACILITY
1 AMERICAN BOTTOMS ROAD
SAUGET, ILLINOIS 62201-1075

(618) 337-1710
FAX (618) 337-8919

January 13, 2012

VIA ELECTRONIC MAIL

Mr. Brett Shank
Solutia Inc.
500 Monsanto Drive
Sauget, IL 62206-1198

Re: SVE Pretreatment System

Dear Mr. Shank,

Solutia submitted specifications for the pretreatment system at the SVE remediation site, which includes an oil water separator, two granular activated carbon units, an effluent flow meter, and to American Bottoms Wastewater Treatment Facility. This letter serves as the approval from the Pretreatment Coordinator of the SVE pretreatment system, flow meter, and sampling location described above, required by permit 10C-105 prior to discharge. This letter does not approve the TSVE pretreatment system. Solutia is responsible for all corrective and preventative maintenance to keep the system working properly.

In addition to the testing that is required by the permit, we request that the results from the intermediate testing that is performed to determine the efficiency of the granular activated carbon units be submitted with Solutia's monthly monitoring reports.

If you have any questions feel free to contact me at 618.337.9749.

Thank You,

Adam Rossel
Pretreatment Coordinator

APPENDIX E

PACE ANALYTICAL QAM AND SOPs

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control
Policies and Procedures

Pace Analytical Services – Kansas Laboratory

Lenexa	Frontenac
9608 Loiret Blvd	808 W McKay
Lenexa, KS 66219	Frontenac, KS 66763
913-599-5665	620-235-0003

CORPORATE APPROVAL

May 14, 2010

Steve A. Vanderboom
President, CEO
1700 Elm Street SE, Suite 200
Minneapolis, MN 55414
(612) 607-1700

Date

May 14, 2010

Bruce Warden
Director of Quality, Safety, and Training
1700 Elm Street SE, Suite 200
Minneapolis, MN 55414
(612) 607-1700

Date

Any printed documents in use within a Pace Analytical Services, Inc. laboratory have been reviewed and approved by the persons listed on the cover page. They can only be deemed official if proper signatures are present.

This is **COPY#**_____ distributed on _____ by _____and is _____**CONTROLLED or** _____**UNCONTROLLED.**

PACE ANALYTICAL SERVICES – *KANSAS LABORATORY*

LOCAL APPROVAL

This document has been approved as the Quality Assurance Manual, effective October 1, 2010, as indicated by the following signatures:

Laboratory General Manager David N. Neal, 913-563-1425	9-17-10 Date
Laboratory Quality Manager Charles E. Girgin, 913-563-1444	9/22/10 Date
Laboratory Technical Director, Frontenac Tim D. Harrell, 913-563-1456	9/21/10 Date

The Technical Director(s) and Quality Manager, however named on the approval page of this document, nominate the following personnel to act as deputies in case of absence.

GC/MS Semivolatile Group leader Steve McGreal, 913-563-1430	9/22/10 Date
Inorganic Manager Kathleen White, 913-563-1427	9/22/10 Date
Client Services Manager Mary Jane Walls, 913-563-1401	9/22/10 Date
Quality Assurance Analyst Robert Perez, 913-563-1445	9/22/2010 Date

Table of Contents

1.0 INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"

Pace Analytical Services Inc. - Mission Statement

1.1 Introduction to PASI

Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, industrial hygiene testing, explosives, high resolution mass spectroscopy (including dioxins, furans and coplanar PCB's), radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

1.2 Statement of Purpose

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

The PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system. The overall objective of this quality system is to provide reliable data through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

All personnel within the PASI network are required to be familiar with all facets of the quality system and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel comply with all current applicable state, federal, and industry standards (such as the NELAC, NVLAP and ISO 17025 standards).

1.4 Pace Analytical Services Core Values

- **INTEGRITY**
- **VALUE EMPLOYEES**
- **KNOW OUR CUSTOMERS**
- **HONOR COMMITMENTS**
- **FLEXIBLE RESPONSE TO DEMAND**
- **PURSUE OPPORTUNITIES**
- **CONTINUOUSLY IMPROVE**

1.5 Code of Ethics

PASI's fundamental ethical principles are as follows:

- Each PASI employee is responsible for the propriety and consequences of his or her actions.
- Each PASI 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 PASI does business or seeks to do business.
- Each PASI employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

Strict adherence by each PASI employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of PASI.

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.

1.6 Standards of Conduct

1.6.1 Data Integrity

The accuracy and integrity of the analytical results produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values and puts PASI and its employees at grave financial and legal risk. Therefore, 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 (e.g., dates, calculations, results or conclusions).

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.

1.6.2 Confidentiality

PASI employees must not (directly or indirectly) use or disclose confidential or proprietary information except when in connection with their duties at PASI. This is effective over the course of employment and for a period of two years thereafter.

Confidential or proprietary information, belonging to either PASI 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.

1.6.3 Conflict of Interest

PASI employees must avoid situations that might involve a conflict of interest or appear questionable to others. The employee must be careful in two general areas:

- Participation in activities that conflict or appear to conflict with PASI responsibilities.
- Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced. This includes bribes, kickbacks or illegal payments.

Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other questionable 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.

1.6.4 Compliance

All employees are required to read, understand and comply with the various components of the standards listed in this document. As confirmation that they understand this responsibility, each employee is required to sign an acknowledgment form (either hardcopy or in electronic database) annually (or as revisions become finalized) that becomes part of the employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

1.7 Laboratory Organization

The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

Each laboratory within the system operates with local management, but all share common systems and receives support from the Corporate Office.

A General Manager (GM) supervises each regional laboratory. Some operations may have an Assistant General Manager (AGM) in situations where the General Manager is responsible for

multiple laboratory facilities and is not necessarily in the facility on a regular basis. Quality Managers (QM) at each lab report directly to their General Manager (or Assistant General Manager) but receive guidance and direction from the Director of Quality.

The General Manager bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of the General Manager (and an Assistant General Manager), the Quality Manager serves as the next in command. He or she assumes the responsibilities of the GM until the GM is available to resume the duties of their position. In the absence of the GM and 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 GM or QM. The most senior department manager in charge may include the Client Services Manager or the Administrative Business Manager at the discretion of the General Manager.

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 General Manager or Quality Manager 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.

The Quality Manager 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 Quality Manager has the authority to halt laboratory operations should he or she deem such an action necessary. The QM will immediately communicate the halting of operations to the GM and keep him or her posted on the progress of corrective actions. In the event the GM and QM are not in agreement as to the need for the suspension, the Chief Operating Officer and Director of Quality will be called in to mediate the situation.

Under the direction of the General Manager, the technical staff of the laboratory is generally organized into the following functional groups:

- Organic Sample Preparation
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Microbiology/Bioassay

Appropriate support groups are present in each laboratory. The actual organizational structure for PASI –*Kansas* is listed in Attachment IIA. In the event of a change in General Manager, Quality Manager or Technical Director(s), the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key personnel will also be noted

by the additional signatures on the QAM Local Approval page. In any case, 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 General Managers 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, and
- Ensures compliance with all applicable state, federal and industry standards.

1.8.2 General Manager (local lab)

- Oversees all functions of the 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 Quality Manager accordingly, and
- Ensures compliance with all applicable state, federal and industry standards.

1.8.3 Assistant General Manager / Operations Manager

- In the absence of the GM, performs all duties as listed above for the General Manager,
- Oversees the daily production and quality activities of all departments,
- Manages all departments and works with staff to ensure department objectives are met,
- Works with all departments to ensure capacity and customer expectations are accurately understood and met,
- Works with General Manager to prepare appropriate budget and staffing plans for all departments,
- Responsible for prioritizing personnel and production activities within all departments, and
- Performs formal and informal performance reviews of departmental staff.

1.8.4. Quality Manager

- Oversees the laboratory Quality Systems while functioning independently from laboratory operations. Reports directly to the General Manager,
- Monitors Quality Assurance policies and Quality Control procedures to ensure that the laboratory achieves established standards of quality,

- 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 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, and
- Maintains the laboratory training records, including those in the Learning Management System (LMS).

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,
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project,
- Serves as the general manager of the laboratory in the absence of the GM, AGM and QM, and
- 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 General Manager 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 (mileposts) 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 General Manager 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, and
- 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, and
- 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 PASI,
- 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 PASI 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, and
- Verifies login information relative to project requirements and field sample Chains-of-Custody.

1.8.9 Project Coordinator

- Responsible for preparation of project specifications and provides technical/project support,
- Coordinates project needs with other department sections and assists with proposal preparation,
- Prepares routine proposals and invoicing,
- Responsible for scanning, copying, assembling and binding final reports, and
- Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

1.8.10 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, and
- Ensures compliance with all applicable state, federal and industry standards.

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, and
- Reports non-compliance situations to laboratory management including the Quality Manager.

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, and
- 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, and

- 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, PASI Quality Assurance Manual and/or customer directed sampling objectives,
- Capable of the collection of representative environmental or process related air samples,
- Use computer software to compile, organize, create tables, create graphics and write test reports,
- 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, and
- Develop sampling plans and prepare test plan documents.

1.8.15 Field Analyst

- Analyzes field samples according to published methods, PASI Quality Assurance Manual and/or customer directed sampling objectives,
- Capable of the collection and analysis of representative environmental or process related air samples,
- Proficient in a variety of analytical tests; specifically on-site gas-phase organic and inorganic compounds by extractive fourier transform infrared spectroscopy (FTIR),
- Train less experienced staff and provide guidance on FTIR sampling and analysis,
- Assist in reporting tasks and project management responsibilities, and
- Perform back-up support for manager tasks such as reporting needs and customer concerns.

1.8.16 Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain-of-Custody forms,
- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting,
- Stages samples according to EPA requirements, and
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

1.8.17 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, and
- Maintains the electronic backups of all computer systems.

1.8.18 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 (e.g. personal protective equipment).

1.8.19 Program Director/Hazardous Waste Coordinator (or otherwise named)

- 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 lab.

1.9 Training and Orientation

Each new employee receives a five part orientation: human resources, ethics and data integrity, safety, Quality Systems, and departmental.

The human resources orientation includes benefits, salary, and company policies. All records are stored with Human Resources.

The ethics and data integrity training covers the obligations of each employee to ensure the defensibility of laboratory data. Employees are provided with general policies related to ethics in the laboratory and specific examples of improper practices that are unacceptable in any PASI facility. The employee is trained to make the right decisions with regards to laboratory practices and where to go for answers in circumstances where they may be unclear as to the correct protocol.

The safety orientation includes an in-depth review of the PASI Chemical Hygiene Plan/Safety Plan, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200) and other pertinent regulations.

The Quality Systems orientation provides the new employee with information through an introduction to the Quality Assurance Manual and SOPs, acceptable record keeping practices, and the individual's responsibility to data quality. Quality Systems training is reinforced with the new employee as specific topics are covered during the departmental or analytical method training. Quality Systems training will address policies and practices that ensure the quality and defensibility of the analytical data. These topics include but are not limited to traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation and root cause analysis.

The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position.

Supervised training uses the following techniques:

- Hands-on training
- Training checklists/worksheets (e.g. from LMS new hire workbooks)
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability (see Section 3.4 for details on Demonstration of Capability requirements). Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the Learning Management System (LMS).

All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by lab management. Additional information can be found in SOP S-ALL-Q-020 ***Training Procedures*** or its equivalent revision or replacement.

1.10 Laboratory Safety

It is the policy of PASI to make safety and health 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 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 corporate Safety Manual and Chemical Hygiene Plan.

1.11 Security and Confidentiality

Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facility 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 GM, QM or TD 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. The last staff member to leave their department for the day should ensure that all outside access points to that area are secure.

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 in secure offsite storage. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out the associated internal Chain-of-Custody records.

Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for internal identification of samples and data by number only are implemented as required under contract-specific Quality Assurance Project Plans (QAPPs).

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 (i.e. federal or state subpoena). Additional information can be found in SOP S-KS-Q-007 ***Lab Security Procedures*** or its equivalent revision or replacement.

2.0 SAMPLE CUSTODY

2.1 Sampling Support

Each individual PASI laboratory provides shipping containers, sample containers (including applicable chemical preservatives), custody documents, and field quality control samples (e.g., trip blanks) to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VIII. Note that all analyses listed are not necessarily performed at all PASI laboratories and there may be additional laboratory analyses performed that are not included in these tables. PASI - *Kansas* may provide pick-up and delivery services to their customers when needed and also has a field services department which provides field sampling services.

All sampling activities conducted by laboratory's field personnel are conducted with the expectation that they will be made for routine monitoring purposes, unless specifically stated to the contrary prior to the field investigation. Therefore, the use of proper sampling procedures cannot be overemphasized. The collection of representative samples depends upon:

- Ensuring that the samples taken are representative of the material or medium being sampled;
- Using proper sampling, sample handling, preservation, and quality control techniques;
- Properly identifying the collected samples and documenting their collection in field records;
- Maintaining sample chain-of-custody; and
- Protecting the collected samples by properly packing and transporting them to the laboratory for analysis.

Additional information can be found in the ***Field Procedures Manual*** S-KS-F-001 or its equivalent revision or replacement.

2.2 Field Services

Pace Analytical has a large Field Services Division that is based in their Minneapolis facility as well as limited field service capabilities in some of the other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Stormwater and Surface Water Monitoring

- Soil and Waste Sampling
- Mobile Laboratory Services

Field Services operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services for a unit specific Quality Program. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.3 Project Initiation

Prior to accepting new work, the laboratory reviews performance capability. The laboratory establishes that sufficient resources (personnel, equipment capacity, analytical method capability, etc.) are available to complete the required work. The customer needs and data quality objectives are defined and appropriate environmental test methods are assured to meet customer's requirements by project managers or sales representative. Project Managers review laboratory certifications. 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.

The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the General Managers and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews are maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

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

2.4 Chain-Of-Custody

A chain-of-custody (COC) (see Attachment VII) document provides the legal documentation of samples from time of collection to completion of analysis. Importance is stressed on completeness of COCs. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

Field personnel or client representatives complete a chain-of-custody form for all samples. Samples are received by the laboratory accompanied by these forms.

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.

The sampler is responsible for providing the following information on the chain-of-custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample type (matrix)
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks (if applicable)
- Custody Seal Number (if applicable)
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

The record 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.

Additional information can be found in SOP S-KS-C-001 ***Sample Management*** and SOP S-KS-C-002 ***Assembly of Sample Container Kits*** or its equivalent revisions or replacements.

2.5 Sample Acceptance Policy

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

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 qualified on the final report.

All samples must:

- Have unique customer identification that are clearly marked with durable waterproof labels on the sample containers and that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature
- Have the requested analyses clearly marked

- Have clear documentation of any special analysis requirements (data deliverables, etc.);
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer permission.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but ≤6°C (See Note 1), unless program requirements or customer contractual obligations mandate otherwise (see Note 2). The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the lab immediately after collection are considered acceptable if there is evidence that the chilling process has been started, for example by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data will be appropriately qualified on the final report.

  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 (i.e. 6.2°C rounded and recorded as 6°C).

  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.

  Note 3: Biological Tissue Samples must be received frozen at ≤0°C.

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
- Sample holding time
- Sample pH when required
- Appropriate containers

Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

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

2.6 Sample Log-in

After sample inspection, all sample information on the chain-of-custody is entered into the Laboratory Information Management System (LIMS).

This permanent record documents receipt of all sample containers including:

- 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 lab receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

All samples received are logged into the LIMS system within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. 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.

The Laboratory Information Management System (EPIC Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The prefix 60 designates the PASI-Kansas laboratory region number, XXXXXX designates a number assigned in numerical order from 1 to 999999, and the suffix 001 designates the physical number of samples within the project work order. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represent the total number of containers of that particular type. Together the LIMs sample number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. 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 client's field identification; it will be a permanent reference number for all future interactions.

Current region codes are noted below. More may be added without updating this document.

10 = Minnesota	35 = Florida
92 = Asheville and Charlotte	20 = Gulf Coast
60 = Kansas	30 = Pittsburgh
50 = Indianapolis	40 = Green Bay
3038 = Pittsburgh Radiological	17 = Pace Life Sciences
25 = Seattle	

Sample labels are printed from the LIMS system and affixed to each sample container. Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or General Manager.

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

2.7 Sample Storage

2.7.1 Storage Conditions

Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross-contamination (e.g. volatile samples are stored separate 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

Additional information can be found in SOP S-ALL-Q-018 ***Monitoring Storage Units*** or its equivalent revision or replacement.

2.7.2 Temperature Monitoring

Samples are taken to the appropriate storage location (ambient, refrigerator, freezer) 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.

The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ unless state or program requirements differ. The temperature of each freezer storage area is maintained at $< -10^{o}C$ unless state or program requirements differ. The temperature of each storage area is monitored and recorded each workday. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after two hours to verify temperature exceedance. Corrective action is initiated if necessary.
- The Quality Manager 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.
- Documentation is provided on analytical report.

Additional information can be found in SOP S-ALL-Q-018 ***Monitoring Storage Units*** or its equivalent revision or replacement.

2.7.3 Hazardous Materials

Pure product or potentially heavily contaminated samples may be tagged as "hazardous" or "lab pack" and are stored separately from other samples.

Additional information can be found in the ***Waste Handling*** SOP S-KS-S-002 or its equivalent revisions or replacements.

2.7.4 Foreign/Quarantined Soils

Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are segregated. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure. Additional information regarding USDA regulations and sample handling can be found in applicable local laboratory SOPs.

Additional information can be found in SOP S-KS-C-001 ***Sample Management*** and S-KS-Q-020 ***USDA Regulated Soil***, and ***Waste Handling*** SOP S-KS-S-002 or its equivalent revisions or replacements.

2.8 Sample Protection

PASI laboratory facilities are operated under controlled access to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted.

Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

Upon customer request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For example, some projects may require complete documentation of sample custody within the secure laboratory.

Additional information can be found in SOP S-KS-C-001 ***Sample Management*** and SOP S-KS-Q-007 ***Lab Security Procedures*** or its equivalent revisions or replacements.

2.9 Subcontracting Analytical Services

Every effort is made to perform chemical analyses for PASI customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory (inside or outside the PASI network) becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. Customers are notified in writing of the lab's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations.

Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions (quick turn-around, required detection or reporting limits, unusual information known about the samples or analytical procedure).
- Signature in "Relinquished By"

All subcontracted sample data reports are sent to the PASI Project Manager.

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

Additional information can be found in SOP S-ALL-Q-027 ***Evaluation & Qualification of Vendors*** and SOP S-KS-C-003 ***Subcontracting Samples*** or its equivalent revision or replacement.

2.10 Sample Retention and Disposal

Samples (and sample by-products) must be retained by the laboratory for a period of time necessary to protect the integrity of the sample or sample by-product (e.g. method holding time) and to protect the interests of the laboratory and the customer.

Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The sample retention time is a minimum of 45 days from receipt of the samples. Samples requiring storage beyond this time due to special requests or contractual obligations will not be stored under temperature controlled conditions unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

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

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

3.0 ANALYTICAL CAPABILITIES

3.1 Analytical Method Sources

PASI 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. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, A2LA, A-Class, NVLAP and State Agencies. Section 11 (References) is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2 Analytical Method Documentation

The primary form of documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3 Analytical Method Validation

In some situations, PASI develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods (e.g. methods other than EPA, NIOSH, ASTM, AOAC, etc.) are required for specific projects or analytes of interest, or when the laboratory develops a method, or modifies a standard method, the laboratory validates the method 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 validation include determination of the limit of detection and limit of quantitation, evaluation of precision and bias, and evaluation of selectivity of each analyte of interest.

Additional information can be found in SOP S-ALL-Q-004 ***Method Detection Limit Studies*** or its equivalent revision or replacement.

3.4 Demonstration of Capability (DOC)

Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel or test method (when a defined 'work cell' is in operation, the entire work cell must meet the criteria). The mean recovery and

standard deviation of each analyte, taken from 4 replicates of a quality control standard is calculated and compared to method criteria (if available) or established lab criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability and corresponding raw data for future reference and must document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4 replicate approach listed above. For methods or procedures that do not lend themselves to the "4 replicate" approach, the demonstration of capability requirements will be specified in the "Method Performance" section of the applicable SOP.

Additional information can be found in SOP S-ALL-Q-020 ***Training Procedures*** or its equivalent revision or replacement.

3.5 Regulatory and Method Compliance

PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the Chain-of-Custody submitted with samples.

PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision-making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

It is PASI 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 QUALITY CONTROL PROCEDURES

4.1 Data Integrity System

The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to providing a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1. A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics within this training include:
   a. Need for honesty in analytical reporting
   b. Process for reporting data integrity issues
   c. Specific examples of unethical behavior and improper practices
   d. Documentation of non-conforming data that is still useful to the data user
   e. Consequences and punishments for unethical behavior
   f. Examples of monitoring devices used by management to review data and systems
2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.
3. In-depth, periodic monitoring of data integrity: including peer data review and validation, internal data audits, proficiency testing studies, etc.
4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be available for review for lab assessors and must be retained for a minimum of five years.

PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over-scheduling, and working condition pressures.

Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. **The message line voice mail box number is available in the Pace Employee Handbook.**

4.2 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system. The method blank is processed through all preparation and analytical steps with its associated samples.

A method blank is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The method blank consists of a matrix similar to the associated samples that is known to be free of the analytes of interest. Laboratories will characterize a representative matrix as "clean" if the matrix contains contaminants at less than ½ the laboratory's reporting limit.

Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater then 1/10 of the amount of that analyte found in any associated sample. Corrective actions include the re-preparation and re-analysis of all the samples (where possible) along with the full set of required quality control samples. Data qualifiers must be applied to any result reported that is associated with a contaminated method blank. Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.3 Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

An LCS is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency (which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix compounds when a full Appendix list of compounds is requested). In the absence of specified components, the lab will spike 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 lab will spike with all compounds
    - For methods with 11-20 target compounds, the lab will spike with at least 10 compounds or 80%, whichever is greater
    - For methods with greater than 20 compounds, the lab will spike with at least 16 compounds.

The LCS is evaluated against the method default or laboratory-derived acceptance criteria. For those methods that require laboratory-derived limits, method default control limits may be used until the laboratory has a minimum of 20 (preferably greater than 30) data points from which to derive internal criteria. Any compound that is outside of these limits is considered to be 'out of

control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier.

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). NELAC has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but less 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)

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 (this is a NELAC allowance). 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 (if possible) or reported with appropriate data qualifiers.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

Additional information for the generation of control limits and charts can be found in SOP S-KS-Q-003 ***Control Chart Generation and Trend Analysis*** or its equivalent revision or replacement.

4.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch (see LCS) unless the MS is actually used as the LCS.

A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per general matrix (i.e. soil, water, biota, etc.) per method.

The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Lab personnel spike customer samples that are specifically designated as

MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible. The MS and MSD contain all analytes specified by a specific method or by the customer or regulatory agency. In the absence of specified components, the lab will spike with the same number of compounds as previously discussed in the LCS section.

The MS and MSD are evaluated against the method or laboratory-derived criteria. 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. 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. Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.5 Surrogates

Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

Surrogates are added to each customer sample (for organics), method blank, LCS and MS prior to extraction or analysis. The surrogates are evaluated against the method or laboratory-derived acceptance criteria. 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 systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.6 Sample Duplicate

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.

The sample and duplicate are evaluated against the method or laboratory-derived criteria 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.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.7 Internal Standards

Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.8 Field Blanks

Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinseate blanks, or equipment blanks. The lab analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.9 Trip Blanks

Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the lab. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.10 Limit of Detection (LOD)

PASI laboratories are required to use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. All sample-processing steps of the preparation and analytical methods are included in this determination. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

Where specifically stated in the published method, LODs (or MDLs) will be performed at the listed frequency.

The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available (e.g. temperature).

The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the lab can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the lab must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The lab must verify the LOD on each instrument used for the reporting of sample data.
- The lab must be able to identify all target analytes in the verification standard (distinguishable from noise).

Additional information can be found in SOP S-ALL-Q-004 ***Method Detection Limit Studies*** or its equivalent revision or replacement.

4.11 Limit of Quantitation (LOQ)

A limit of quantitation (LOQ) for every analyte of concern must be determined. For PASI laboratories, this LOQ is referred to as the RL, or Reporting Limit. This RL is based on the lowest calibration standard concentration that is used in each initial calibration. Results below this level are not allowed to 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 out below the LOQ but above the LOD if they are properly qualified (e.g. J flag).

There must be a sufficient buffer between the LOD and the limit of quantitation (LOQ). The LOQ must be higher than the LOD.

To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with target analytes at the concentration(s) equivalent to or less than the RL(s). This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits or as specified by the method.

Additional information can be found in SOP S-ALL-Q-004 ***Method Detection Limit Studies*** or its equivalent revision or replacement.

4.12 Estimate of Uncertainty

PASI laboratories 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. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. 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.

Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-KS-Q-022 ***Estimation of Measurement Uncertainty*** or its equivalent revision or replacement.

4.13 Proficiency Testing (PT) Studies

PASI laboratories participate in the NELAC-defined proficiency testing program. PT samples are obtained from NIST and/or A2LA accredited approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix. The PT studies are reported directly to the required accreditation agencies (e.g. A2LA, Kansas Department of Health, etc.). A PT plan is required for A2LA and is maintained on file.

The lab initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the Quality Manager. A corrective action plan (including re-analysis of similar samples) is initiated and this report is sent to the appropriate state accreditation agencies for their review.

PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

Additional information can be found in SOP S-ALL-Q-010 ***PE/PT Program*** or its equivalent revision or replacement.

4.14 Rounding and Significant Figures

In general, the PASI laboratories report data to no more than three significant digits. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter 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 (Excel, etc.).

Rounding

PASI-*Kansas* 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).

Significant Digits

PASI-*Kansas* 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 digits
- Values ≤ 10 – Reported to 2 significant digits

5.0 DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1 Document Management

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

Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures, Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system.

A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

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***.

SOPs, specifically, are available to all lab staff via the Learning Management System (LMS), which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the lab's local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Electronic documents (i.e. pdf's) must be locked from printing. All hardcopy SOPs must be obtained from the Quality Department.

5.1.1 Quality Assurance Manual (QAM)

The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the regional Quality Managers. The regional management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality, the General Manager, Quality Manager and Technical Director(s) sign the Quality Assurance Manual. Each regional Quality Manager 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 by all of the PASI Quality Managers and revised accordingly by the Director of Quality.

5.1.2 Standard Operating Procedures (SOPs)

SOPs fall into two categories: company-wide documents (starting with the prefix S-ALL-) and facility-specific documents (starting with the individual facility prefix).

The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the regional Quality Managers. The regional management personnel sign the company-wide SOPs. The regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies.

Regional PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The regional facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The regional facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the regional Quality Manager according to the corporate document management policies.

SOPs are reviewed every two years at a minimum (a more frequent review may be required by state or federal agencies or customers). A review of the document does not necessarily constitute a re-issue of a new revision. Documentation of this review and any applicable revisions are made in the last section of each SOP. This provides a historical record of all revisions.

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 PASI employees use the most current version of each SOP and provides the Quality Manager with a historical record of each SOP.

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

5.1.3 Field Service Procedures Manual (FSM)

The Field Service Procedures Manual is a document that describes all aspects of the field test and sampling procedures performed by PASI-*Kansas*. This document is distributed to all field personnel and maintained by the Quality Assurance department.

5.2 Document Change Control

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. Copies may be kept for audit or knowledge preservation purposes.

All controlled copies of the previous document are replaced with controlled 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.

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

5.3 Management of Change

The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large-scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-ALL-Q-034 ***Management of Change*** or its equivalent revision or replacement.

6.0 EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the PASI- *Kansas* facility.

6.1 Standards and Traceability

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

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.

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 PASI identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

If a second source standard is required to verify an existing calibration or spiking standard, this standard is purchased from a different supplier. If no second source is available, a second standard from a different lot may be purchased from the same supplier if the lot can be demonstrated as prepared independently from other lots.

Additional information concerning standards and reagent traceability can be found in the SOP S-ALL-Q-025 ***Standard and Reagent Preparation and Traceability***, SOP S-KS-Q-026 ***Purchasing of Lab Supplies***, and S-KS-Q-006 ***Receipt and Storage of Lab Supplies,*** **and the** ***A2LA Policy on Measurement Traceability P102*** or its equivalent revisions or replacements.

6.2 General Analytical Instrument Calibration Procedures

All types of support equipment and instrumentation are calibrated or checked before use to ensure proper functioning and verify that the laboratory's 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.

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 (e.g. J flag) 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 (e.g. E flag) or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

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.

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. If the investigation indicates sample results have been impacted, the customer is notified within 30 days. 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 replaced.

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.

6.2.1 General Organic Calibration Procedures

Calibration standards are prepared at a minimum of five concentrations for organic analyses. Results from all calibration standards must be included in constructing the calibration curve with the following exceptions:

- 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.
- 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.

- 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 remain as established by method or standard operating procedure. The reporting limit or quantitation range, which is appropriate, must be adjusted accordingly.
- Results from a concentration level between the lowest and highest calibration levels can be excluded from the calibration curve for an acceptable cause with approval from the responsible department supervisor if the results for all analytes are excluded and the point is replaced by re-analysis. Re-analysis must occur within the same 12 hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed.

Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. Calibration verification is performed at the beginning and end of each analytical batch (except if an internal standard is used only one verification at the beginning of the batch is needed), whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that contain a calibration verification requirement. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence is continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated. Sample analysis cannot begin until after documented corrective action has been completed and two consecutive CCVs have been analyzed (NELAC). (If required by specific state, program, or customer specification, the instrument is re-calibrated after two consecutive CCV failures.) All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

When instruments are operating unattended, the autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. (The 12-hour clock begins with the injection of the second CCV.)
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.

- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples preceded by the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

6.2.2 General Inorganic Calibration Procedures

The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range is established,
- Zero point and single point calibration standards are analyzed with each batch
- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards. A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs.

Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3 Support Equipment Calibration Procedures

All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications.

6.3.1 Analytical Balances

Each analytical balance is checked and (if necessary) calibrated annually by a qualified service technician. The calibration of each balance is checked each day of use with weights traceable to NIST. Calibration weights are ASTM Class 1 (or other class weights that have been calibrated against a NIST standard weight) and are re-certified annually against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.2 Thermometers

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, at a minimum, every 3 years with equipment directly traceable to NIST.

Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

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

6.3.3 pH/Electrometers

The meter is calibrated before use each day, using fresh buffer solutions.

6.3.4 Spectrophotometers

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

6.3.5 Mechanical Volumetric Dispensing Devices

Mechanical volumetric dispensing devices including bottle top dispensers, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. The accuracy of glass microliter syringes is verified and documented prior to use.

Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-ALL-Q-013 ***Support Equipment*** or its equivalent revision or replacement.

6.4 Instrument/ Equipment Maintenance

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.

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

To minimize downtime and interruption of analytical work, preventative maintenance is 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.

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.

All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation are, 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

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

When maintenance is performed to repair an instrument problem, depending on the initial problem, demonstration of return to control may be satisfied by the successful analysis of a reagent blank or continuing calibration standard. The 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.

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 satisfactorily perform.

Additional information regarding instrument transport can be found in SOP S-KS-Q-024 ***Instrument Transport*** or its equivalent revision or replacement.

7.0 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 well-defined, well-documented multi-tier review process prior to being reported to the customer. This section describes procedures used by PASI for translating raw analytical data into accurate, final sample reports and PASI data storage policies.

7.1 Analytical Results Processing

When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g. Run log or Instrument log) or copies of computer-generated printouts are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the lab chooses to minimize or eliminate its paper usage, these records can be kept as electronic records. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting discrepancies in logbooks and as footnotes or narratives, and uploading analytical results into the LIMS.

The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain-of-custody forms, and logbook copies.

Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

Additional information regarding laboratory documentation requirements can be found in SOP S-ALL-Q-009 ***Laboratory Documentation*** or its equivalent revision or replacement.

7.2 Data Verification

Data verification 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 discrepancies are properly documented.

Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS.

The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data reviewer, the reviewer must:

1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note
2. Have a DOC on file for a similar method/technology (i.e. GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note
3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,
4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

Note: Secondary reviewer status must be approved personally by the Quality Manager or General Manager in the event that this person has no prior experience on the specific method or general technology (i.e. GC/MS).

This 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 and data quantitation. The reviewer also validates the data entered into the LIMS.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

The data verification procedures are further supplemented by the LIMS Data Checker program that was developed to review test results for chemical incongruence (i.e. Total Metals >Dissolved Metals) and quality control outliers. This evaluation is performed and summarized during the generation of the final report and reviewed by the Project Manager.

Additional information regarding data verification can be found in SOP S-KS-Q-005 ***Data Reduction, Review, and Reporting***, S-ALL-Q-016 ***Manual Integrations***, and SOP S-ALL-Q-030 ***EpicPro Data Checker*** or its equivalent revisions or replacements.

7.3 Data Reporting

All data segments pertaining to a particular PASI project number are delivered to the Client Services Department (Project Manager) for assembly into the final report. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

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 PASI final report consists of the following components:

1. A title which designates the report as "Final Report", "Laboratory Results", "Certificate of Results", etc.
2. Name and address of laboratory (or subcontracted laboratories, if used).
3. Phone number and name of laboratory contact where questions can be referred.
4. A unique number for the report (project number). The pages of the report shall be numbered and a total number of pages shall be indicated (usually in the cover letter).
5. Name and address of customer and name of project (if applicable).
6. Unique identification of samples analyzed (including customer sample numbers).
7. Identification of any sample that did not meet acceptable sampling requirements (from NELAC or other governing agency), such as improper sample containers, holding times missed, sample temperature, etc.
8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less.
9. Identification of the test methods used.
10. Identification of sampling procedures if sampling was conducted by the laboratory.
11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data.
12. Identification of whether calculations were performed on a dry or wet-weight basis.
13. Reporting limits used.
14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.
15. A signature and title of person accepting responsibility for the content of the report (can be an equivalent electronic identification) and date report was issued.
16. 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.
17. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory.
18. Identification of all test results provided by a subcontracted laboratory or other outside source.
19. Identification of results obtained outside of quantitation levels.

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

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

The following positions are the only approved signatories for PASI final reports:

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

Additional information regarding data reporting can be found in SOP S-KS-Q-005 ***Data Reduction, Review, and Reporting*** or its equivalent revision or replacement.

7.4 Data Security

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

Additional information regarding data reporting can be found in SOP S-ALL-IT-001 ***System Security and Integrity*** or its equivalent revision or replacement.

7.5 Data Archiving

All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable 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. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis and personnel involved. NELAP-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the Quality Manager or a designated Data Archivist.

Records that are computer-generated have either a hard copy or electronic write-protected 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.

In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

Additional information regarding data reporting can be found in SOP S-KS-Q-019 ***Lab Data Filing and Archiving*** or its equivalent revision or replacement.

7.6 Data Disposal

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.

Additional information regarding data security, archival, and disposal can be found in SOP S-ALL-IT-001 ***System Security and Integrity***, SOP S-ALL-IT-002 ***Server Backup***, and SOP S-KS-IT-001 ***Target Data Backup*** or its equivalent revisions or replacements.

8.0 QUALITY SYSTEM AUDITS AND REVIEWS

8.1 Internal Audits

8.1.1 Responsibilities

The Quality Manager is responsible for designing 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 functionally 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 Quality Manger 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 execution of the Quality System as outlined in this manual but may also include other quality programs applicable to each laboratory.

8.1.2 Scope and Frequency of Internal Audits

The complete internal audit process consists of the following four sections:

- Raw Data Review audits- conducted according to a schedule per local Quality Manager. A certain number of these data review audits are 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

Internal systems audits are conducted yearly 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.

Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual and all applicable addenda
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, standards, and associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.
- General procedures for data security, review, documentation, reporting and archiving.
- Data integrity issues such as proper manual integrations.

When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain-of-custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

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

A representative number of data audits are completed annually. The report format of any discrepancy is similar to that of other internal audits.

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 lab and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and impact to final results is assessed.

8.1.3 Internal Audit Reports and Corrective Action Plans

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. Although other personnel may assist with the performance of the audit, the Quality Manager writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

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 3 business days, if investigations show that the laboratory results may have been affected.

Once completed, the internal audit report is issued jointly to the Laboratory General Manager and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The Quality Manager may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

The Quality Manager reviews the audit responses. If the response is accepted, the Quality Manager uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the Quality Manager determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

To complete the audit process, the Quality Manager performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

Additional information can be found in SOP S-ALL-Q-011 ***Audits and Inspections*** and SOP S-ALL-Q-029 ***MintMiner© Data File Review*** or its equivalent revision or replacement.

8.2 External Audits

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

Audit teams external to the company review the laboratory to assess the existence of systems and degree of technical expertise. The Quality Manager and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the Quality Manager. The Laboratory General Manager provides the necessary resources for staff to develop and implement the corrective action plans. The Quality Manager collates this information and provides a written report to the audit team. The report contains the corrective action plan and expected completion dates for each element of the plan. The Quality Manager follows-up with the laboratory staff to ensure corrective actions are implemented.

8.3 Quarterly Quality Reports

The Quality Manager is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Company-wide 3P Document implementation (internal program)
- External audit findings
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Corrective action activities

- Training activity status
- Other significant Quality System items

The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the Quality Systems of the company as a whole. Each General Manager utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

Additional information can be found in SOP S-ALL-Q-014 ***Quality System Review*** or its equivalent revision or replacement.

8.4 Annual Managerial Review

A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

The managerial review must include the following topics of discussion:

- Policy and procedure suitability
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventative 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 and staffing.

The Quality Manager must document this managerial review for future reference and copies of the report are distributed to laboratory staff. Results should feed into the laboratory planning system and should 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 timescale.

Additional information can be found in SOP S-ALL-Q-014 ***Quality System Review*** or its equivalent revision or replacement.

8.5 Customer Service Reviews

As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys. The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the lab management in order for them to evaluate and improve their management system, testing activities and customer service.

In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the lab's performance in relation to the work being performed for the customers.

9.0 CORRECTIVE ACTION

During the process of sample handling, preparation and analysis, 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 PASI provides systematic procedures for documentation, monitoring, and completion of corrective actions and follow-up verification of the effectiveness of these corrective actions. This is tracked using PASI's Lab Track system that lists among other things, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

9.1 Corrective Action Documentation

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

- Internal Laboratory Non-Conformance Trends
- PE/PT Sample Results
- Internal and External Audits
- Data Review
- Client Complaints
- Client Inquiries
- Holding Time violations

Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g. matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventative Action report or Lab Track ticket. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name and the sample matrix involved. 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.

In the event that the laboratory is unable to determine the cause, 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 problem, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance problem. 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 Lab Track or on the Corrective/Preventative Action Report.

After all the documentation is completed, the routing of the Corrective/Preventative Action Report or Lab Track ticket will continue from the person initiating the corrective action, to their immediate supervisor or the Project Manager and finally to the Quality Manager, who is responsible for final review and signoff of all formal corrective/preventative actions.

Additional information can be found in SOP S-ALL-Q-012 ***Corrective Action/Preventive Action Process*** or its equivalent revision or replacement.

9.2 Corrective Action Completion

9.2.1 Internal Laboratory Non-Conformance Trends

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:

- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Lab accident
- Spike Failure
- Instrument Failure
- Final Reporting error

9.2.2 PE/PT Sample Results

Any PT result returned to the Quality Manager 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 Quality Manager 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 Quality Manager and reported to the applicable regulatory authorities.

9.2.3 Internal and External Audits

The Quality Manager 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 reporting back to the auditing body, the root cause of the issue, and the corrective action taken to resolve the findings. The Quality Manager is also responsible for providing any back-up documentation used to prove that a corrective action has been completed.

9.2.4 Data Review

In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g. by the Quality Manager), 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.

9.2.5 Client Complaints

Project Managers are responsible for issuing corrective action forms 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. After the corrective actions have been listed, the Project Manager reviews the corrective action to determine if the customer needs or concerns are being addressed.

9.2.6 Client Inquiries

When an error on the customer 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, 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.

9.2.7 Holding Time Violations

In the event that a holding time requirement has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the Quality Manager must be made aware of these holding time violations.

The Project Manager must contact the customer for appropriate decisions to be made with the resolution documented and included in the customer project file. The Quality Manager includes a list of all missed holding times in their Quarterly Report to the corporate office.

9.3. Preventive Action Documentation

Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems (technical, managerial, quality, etc.). These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems are discovered. These improvements can be made within a department or lab-wide.

- Annual managerial reviews- part of this NELAC-required and NVLAP-required review is to look at all processes and procedures used by the lab over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

When improvement opportunities are identified or if preventive action is required, the lab can develop, implement, and monitor preventive action plans.

Additional information can be found in SOP S-ALL-Q-012 ***Corrective Action/Preventive Action Process***, S-ALL-Q-029 ***3P Program: Continuous Process Improvement***, SOP S-KS-Q-028 ***Customer Complaint Resolution, and*** SOP S-ALL-Q-028 ***Lab Track System*** or its equivalent revisions or replacements.

10.0 GLOSSARY

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity and Performance. Best Practices are identified that can be used by all PASI labs.
Accuracy	The 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.
Aliquot	A portion of a sample taken for analysis.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analyte	The specific chemical species or parameter an analysis seeks to determine.
Analytical Uncertainty	A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Audit	A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	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 NELAC-defined 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) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Bias	The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	A sample for submitted 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 analyst or laboratory 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	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard must bracket the range of planned or expected sample measurements.
Calibration Curve	The graphic representation of known values, such as concentrations for a series of calibration standards and their instrument response.
Calibration Verification	The process of verifying a calibration by analysis of standards and comparing the results with the known amount.
Chain-of-Custody (COC)	A record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, 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.
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	Verification of the identity of a component through the use of an alternate scientific approach from the original method. These may include, but are not limited to: • second-column confirmation • alternate wavelength • derivatization derivative • mass spectral interpretation • additional cleanup procedures
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 (CCV)	Also referred to as a 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.
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.
Corrective Action	The action taken to eliminate the causes of a non-conformity, defect, or other undesirable situation in order to prevent recurrence.
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.
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	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
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).
Document Control (Management)	Procedures to ensure that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled (managed) to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate or Replicate Analysis	The identically performed measurement on two or more sub-samples of the same sample within a short interval of time
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.

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.
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	Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Finding	An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.
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	The maximum time that samples may be held prior to preparation and/or analysis as defined by the method.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
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-AES 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 Verification (ICV)	A standard (usually from a second source or otherwise required vendor) analyzed after the initial calibration curve to verify that the curve is valid.
Internal Standards	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.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Laboratory Control Sample (LCS)	(however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes 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 assess the performance of all or a portion of the measurement system.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
LabTrack	Database used by Pace Analytical to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A training database used by Pace Analytical to train their employees. This system is a self-paced system which is capable of tracking all employee training requirements and documentation.

Legal Chain-of-Custody	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 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 of Detection (LOD)	A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. An LOD is analyte and matrix specific and may be lab-dependent.
Limit of Quantitation (LOQ)	The minimum levels, concentrations or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
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 lab's learn centers are maintained by a local administrator.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix Duplicate	A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	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)	A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte.
Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis) systematically presented in the order in which they are to be executed.
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures: and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Limit of Detection (LOD); 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.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.

National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
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.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
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.
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.
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 lab's ability to reproduce those conditions.
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	Any conditions under which a sample must be kept in order to maintain the chemical and/or biological integrity of the sample.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Sample	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.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.
Quality Assurance (QA)	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 required by a specific project.

Quality Control (QC)	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)	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 System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.
Quality System Matrix	These matrix definitions are to be used for purposes of batch and quality control requirements: • **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 or 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 saltwater source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
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	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, printouts of chromatograms, instrument outputs, and handwritten records.

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.
Reference Standard	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.
Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the lab'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.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical 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	Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective 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.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels (concentrations) of a variable of interest.
Standard	A substance or material with properties known with sufficient accuracy to permit its use to evaluate the same property in a sample.
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 Operating Procedure (SOP)	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

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.
Surrogate	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.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
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.
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	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.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Verification	Confirmation by examination and objective evidence that specified requirements have been met.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

11.0 REFERENCES

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
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- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (September 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
- Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988
- National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent
- ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.

12.0 REVISIONS

The PASI Corporate Quality and Safety Manager files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual Revision 12.0	General: replaced the word 'client' with 'customer', where applicable. Section 1.6.4: added language for clarity Added new section 1.8.1; responsibilities of Senior General Managers. Section 1.8.3: added reference to LMS. Added new section 1.8.17: responsibilities of Waste Coordinators. Section 1.9, last paragraph: changed 'annually' to 'periodically'. Next to last paragraph- added reference to LMS. Added new section 2.2 entitled Field Services. Section 2.3: added reference to the new Review of Analytical Requests SOP. Section 2.7.2: changed freezer temp requirement to match SOP. Section 4.10: revised and added language regarding LOD studies, initial verification and annual verification, where applicable. Section 4.11: changed PRL to RL. Section 4.13: added editable line regarding PT study information. Section 4.14: added sentence regarding rounding rules listed applying only to LIMS. Section 5.1, last bullet point: changed language to reflect that SOPs must be locked from printing if controlled electronically. Section 6.3.1: adjusted language about classes of weights potentially used. Section 6.3.3: removed customer-specific requirement to re-calibrate every four hours but added space for this to be added back in where applicable. Added reference to Attachment III in the introductory paragraph to section 6. Sections 7.1-7.3: added language for those labs that are minimizing or eliminating the need for paper copies. Section 7.2: clarified language in numbered items so that it does not appear that all 4 criteria must be applicable at one time. Section 7.3: added list of approved signatories for final reports. Section 8.1.2, last paragraph: revised language regarding data integrity issues and added a timeframe to notify customers of affected data. Added section 8.5 "Customer Service Reviews"- ISO requirement Added new section 9.3 regarding Preventive Action. Attachment IIb: updated corporate org chart Attachment VIII: revised to match the current Analytical Guides.	13Nov2008
Quality Assurance Manual revision 13.0	Increased font size of entire document. Section 1.7, fifth paragraph: changed length of time Technical Director can be gone before contacting primary authority (from 65 down to 35 days per TNI standard). Section 1.8.2: Reworded definition for Assistant GM to say "all departments". Fixed numbering issue with sub-sections for section 1.8 and used bullet points instead of numbers. Section 1.8.19: revised position title to capture requirement of some labs. Section 1.9: added language to second bullet point regarding LMS. Section 1.9: added bullet point for on-line courses. Section 2.5: added third note per request from GB (in red text). Section 2.6: Added chart of 2-digit codes (lab designations) per audit finding from GB lab (matches corporate SOPs). Section 2.7.4: added reference for Waste Handling and Management SOT. Section 3.1: added more method agency references. Section 3.4: added reference to Training SOP at end of section. Section 4.1: fixed numbering issue. Removed anonymous phone number	30Apr2010

Document Number	Reason for Change	Date
	and added reference to the Employee Handbook. Section 4.2: added paragraph of Ohio VAP required language (red text). Section 4.3, fifth paragraph: reworded second sentence for clarity. Section 4.3: added paragraphs of Ohio VAP and DoD required language (red text). Section 4.4, first paragraph: added qualifier to end of paragraph that MS limits are used to assess the batch if the MS is used in place of the LCS. Section 4.4: added paragraph of Ohio VAP required language (red text). Section 4.6: added paragraph of Ohio VAP required language (red text). Section 4.7: added paragraph of Ohio VAP required language (red text). Section 4.10: added paragraph of Ohio VAP required language (red text). Section 4.11: added paragraph of DoD required language (red text). Section 5.1, fifth paragraph: changed wording from LAN/WAN to local server (as opposed to hardcopies) and added language about LMS access. Section 5.1.2: added paragraphs of Ohio VAP and DoD required language (red text). Added new section 5.3- Management of Change. Section 6.2.1: added paragraph of Ohio VAP required language (red text). Section 6.3.2: changed NIST thermometer calibration frequency to every 3 years to match current practice. Section 7.3: added comment about Ohio VAP reporting (red text). Section 8.1.2, last sentence: reworded to match current practice. Section 8.1.3, last paragraph: reworded sentences regarding verification of corrective actions. Section 8.3: revised list of Quarterly Quality report items to match the revised SOP. Section 8.4: added last two bullet points and added second line of last paragraph to match ISO language. Section 9.1: changed bullet point items to match CAR SOP. Section 9.2.1: revised language to match SOP. Section 9.2.2: moved language from old 9.2.8 to 9.2.2. Section 9.2.4: added language to data review section. Glossary: Added definitions for analytical uncertainty, audit, bias, field of accreditation, finding, legal COC, matrix duplicate, method, PT sample, sampling, verification (per TNI standard). Glossary: Added definitions for reporting limit verification standard and initial calibration verification per request. Glossary: revised the following definitions to match new TNI language: DOC, LCS, LOD, MS, MSD, preservation, QA, QC, QC sample, raw data, reference standard, SOPs, and traceability. Also revised language within the definition for Quality System Matrix (previously just called Matrix). Glossary: deleted definition for 'detection limit'. Glossary: added definitions from company Acronym form from IT. Glossary: added definitions for LabTrack and MintMiner. Attachment VIII: added more tests to the chart per QM input including a line item for concentrated waste matrix for VOA 8260. General: changed all references to "Director of Quality, Safety, and Training" to "Director of Quality". General: revised document references to SOTs for Waste Handling and Management and Sample Management. Removed corporate org chart from Attachment IIB and will provide this as a separate document to the QMs. In this way, revisions to the corporate org chart will not necessitate a new QAM revision.	

ATTACHMENT I

Quality Control Calculations

PERCENT RECOVERY (%REC)

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

NOTE: The SampleConc is zero (0) for theLCS 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 (CONTINUED)

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

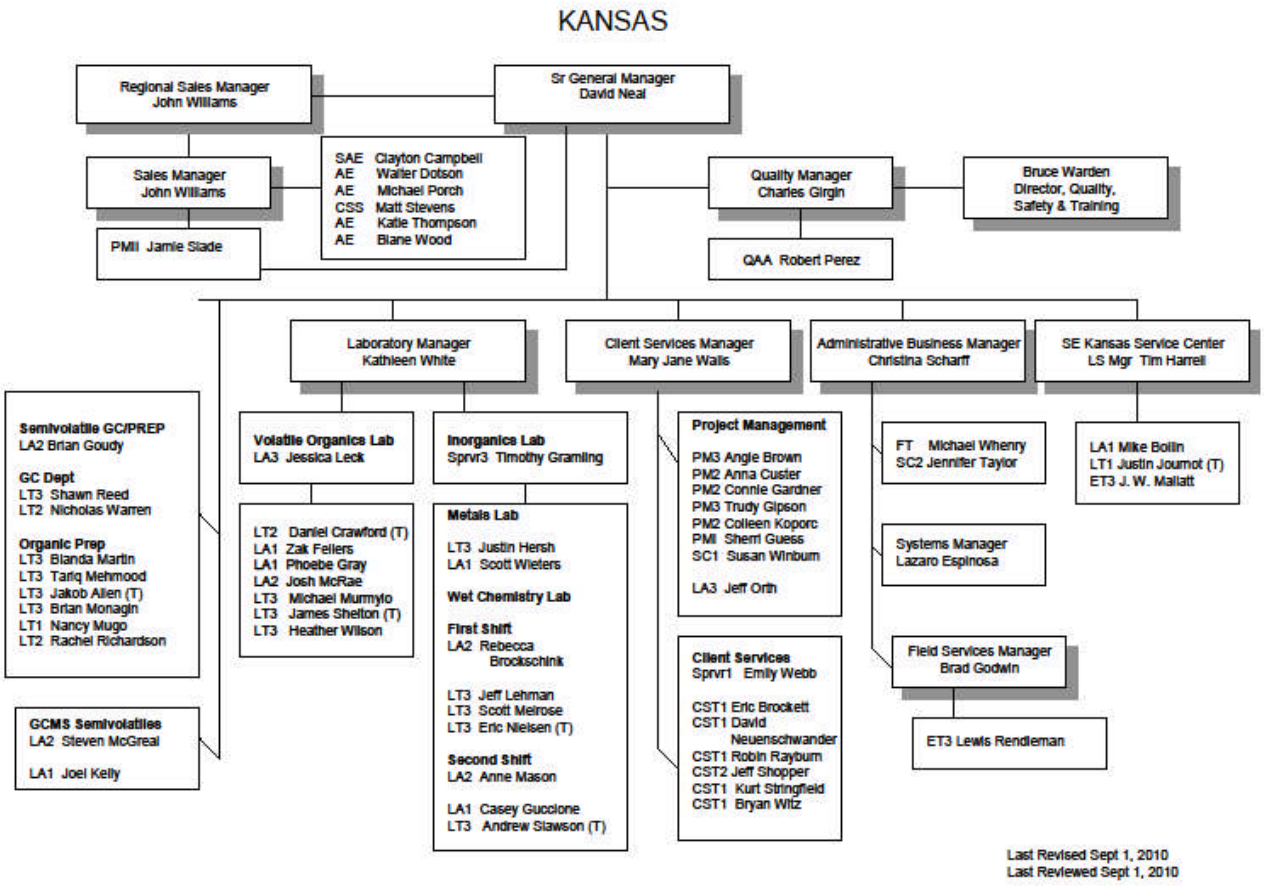
RELATIVE STANDARD DEVIATION (RSD)

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

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

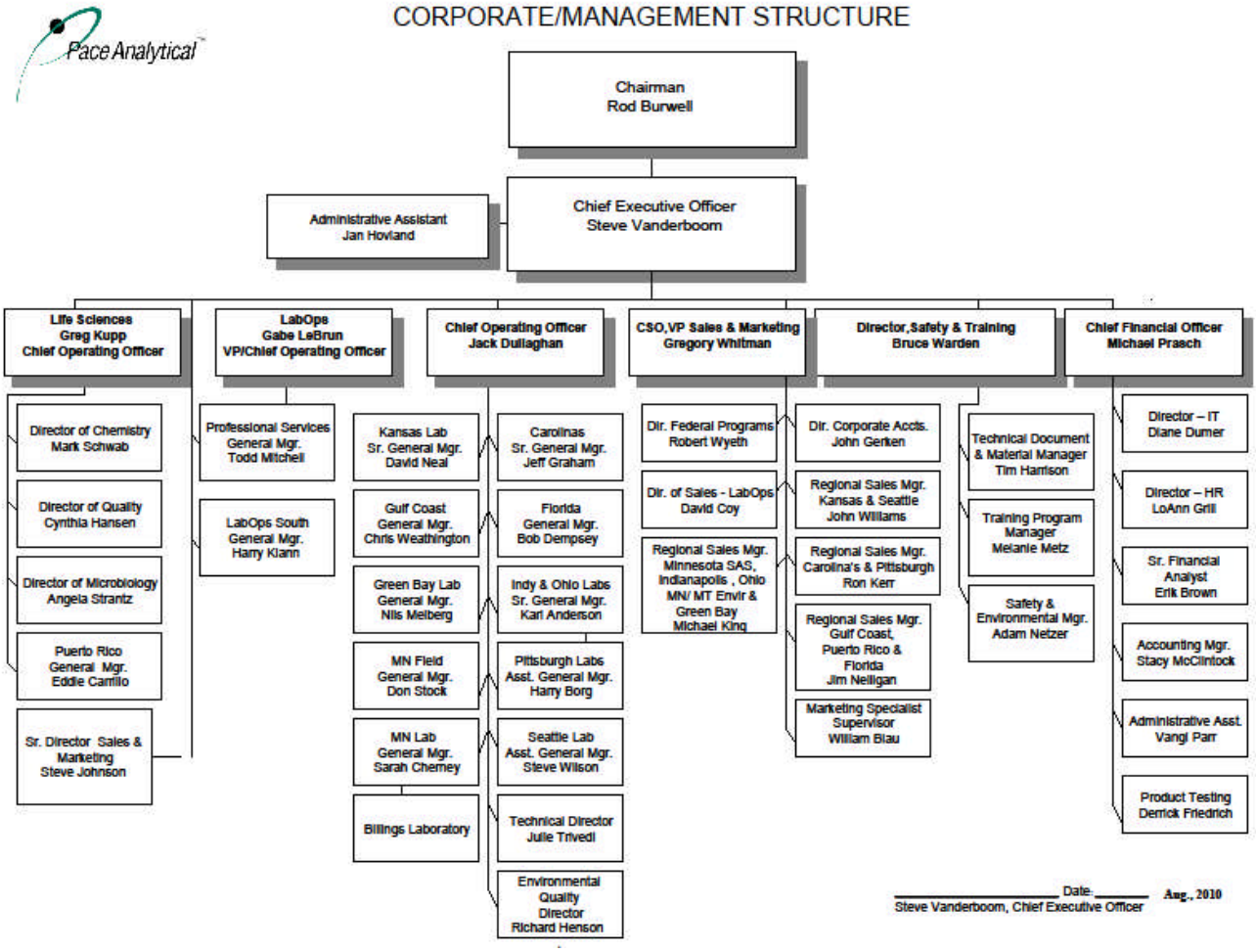
ATTACHMENT IIA

PASI – KANSAS ORGANIZATIONAL CHART



ATTACHMENT IIB

PASI – CORPORATE ORGANIZATIONAL CHART



ATTACHMENT III

PASI – *KANSAS* EQUIPMENT LIST
Lenexa, Kansas Laboratory

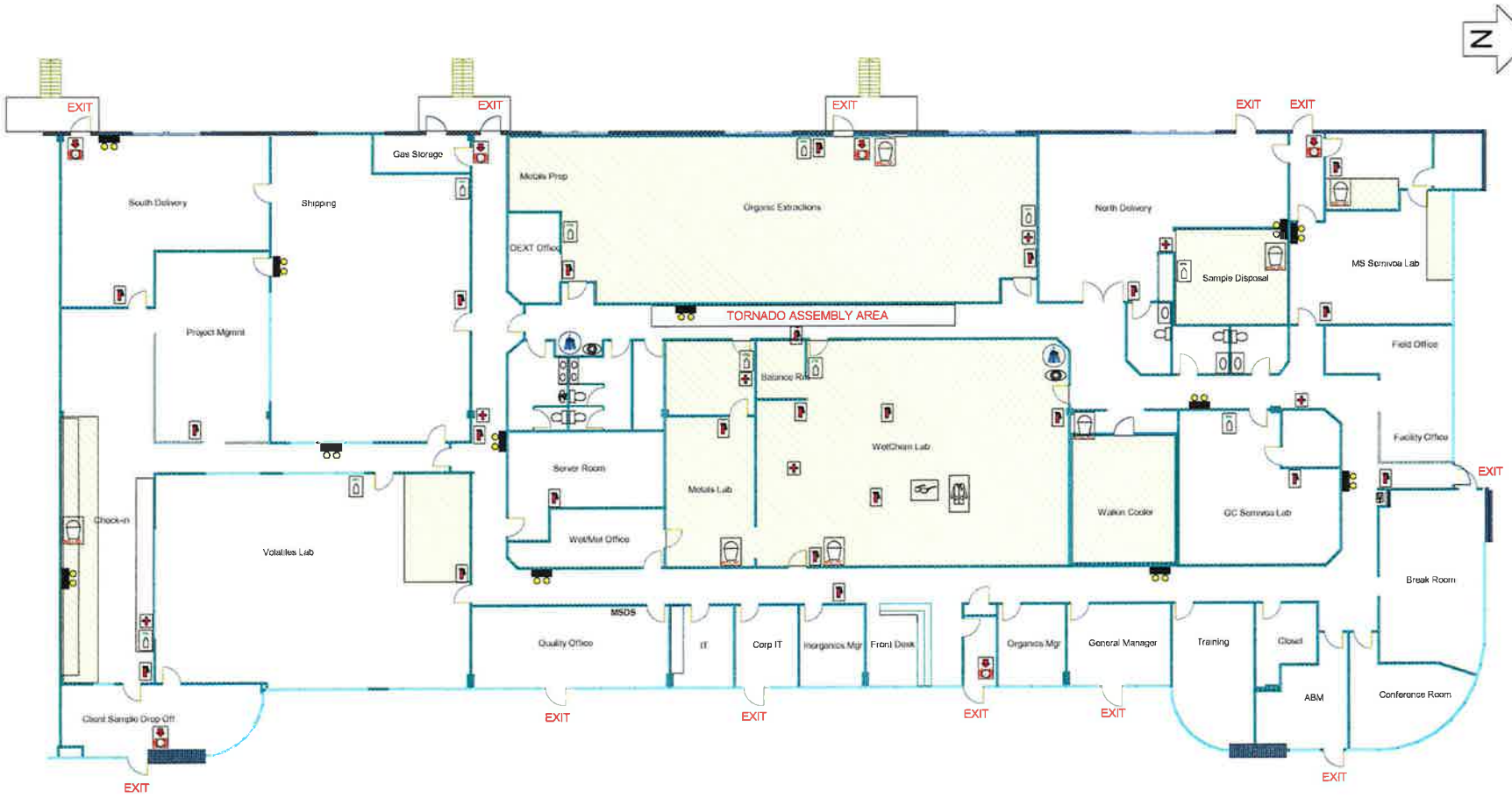
Instrument	Age	Manufacturer	Model Number	Detector	Total Analyses
GC/MS	2008	Agilent	6890/5975C	MS	8260 water, ML soil, GRO
GC/MS	2010	Agilent	6850/5975C	MS	8260 water, ML soil, GRO
GC/MS	2009	Agilent	6850/5975B	MS	8260 water, ML soil, GRO
GC/MS	1998	Hewlett-Packard	5890/5972	MS	8260 water, ML soil, GRO
GC/MS	2010	Agilent	6890/5975C	MS	8260 soil, GRO
GC/MS	2002	Hewlett-Packard	6890/5973	MS	8260 water, ML soil, TCLP, GRO
GC/MS	2007	Agilent	6850/5975C	MS	8260 water, 624, GRO
GC/MS	2009	Agilent	7890A/5975C	MS	8270 SIM, 8270 Full Scan, 625
GC/MS	2002	Hewlett-Packard	6890/5973	MS	8270 SIM
GC/MS	2007	Agilent	7890A/5975C	MS	8270 DRO/ORO
GC	1991	Hewlett-Packard	5890	PID/FID	8021, OK GRO, 8015, 602, OA1
GC	1990	Hewlett-Packard	5890	ECD	8082
GC	2008	Agilent	7890A	Dual FID	DRO (8015, IA-OA2, OK)
GC	1991	Hewlett-Packard	5890	FID	DRO (8015, IA-OA2, OK,)
GC	1991	Hewlett-Packard	5890	FID	DRO (8015, IA-OA2, OK, TNRCC 1005)
GC	2005	Hewlett-Packard	6890	Dual ECD	8081, 8082
Microwave Extractor	2006	CEM	MARS 230/60 907501	Microwave	TPH DRO, 8270C, 8081A, 8082
Ion Analyzer (pH)	2002	Corning	Model 10	Electrolytic	pH
Ion Analyzer (pH)	2010	Thermo Orion	350	Electrolytic	pH
Dissolved Oxygen	1998	Accumet	AB15	Electrolytic	Dissolved Oxygen
Conductivity Meter	1998	Fisher Scientific	Fisher	Electrolytic	BOD, Specific Conductance
TCLP Rotator	1990	Associate Design	3740-48BRE	NA	TCLP/ZHE
ICP	2009	Thermo	6500	ICP	ICP Metals 200.7, 6010
ICP	2000	Thermo Jarrel Ash	61E Trace	ICP	ICP Metals 200.7, 6010
CVAA Hg Analyzer	2007	Perkin-Elmer	FIMS400	Cold Vapor	Mercury
Auto Analyzer	2009	QuikChem	8500	Colorimetric	Ammonia, TKN, Total Phos, Ortho Phos, Nitrate/Nitrite, Cyanide, Chloride, Phenol
Ion Chromatograph	1992	Dionex	DX-100	Electrolytic	Fluoride, Chloride, Nitrite, Bromide, Nitrate, Ortho Phos, Sulfate
Ion Chromatograph	2008	Dionex	ICS-2000	Electrolytic	Fluoride, Chloride, Nitrite, Bromide, Nitrate, Ortho Phos, Sulfate
Discrete Analyzer	2006	West Co	Smartchem	Colorimetric	Phenol, TKN,
Spectrophotometer	1992	Shimadzu	2101 PC	UV	MBAS, Silica, Manual NH3
Spectrophotometer	2008	Shimadzu	UV-1800	UV	COD, Residual Chlorine, Hexavalent Chromium, Phenols, Ferrous Iron
Chlorine Analyzer	2003	HACH	AutoCat9000	Amperometric	Residual Chlorine, Total
Autotitrator	1993	Radiometer	VIT90	Electrolytic	Alkalinity
TOC Analyzer	1993	Astro	2000	UV/FID	Water TOX, UV/Persulfate oxidation
Autoextractor, ASE	2000	Dionex	ASE-200	NA	1664, TPH in Soil
Flashpoint Analyzer	1990	Pensky-Martins	Flashpoint	Combustion	Flashpoint
Flashpoint Analyzer	2010	Pensky-Martins	Flashpoint	Combustion	Flashpoint
Oven	2001	Fisher Scientific	Isotemp 750F	NA	Solids methods
Oven	2001	Fisher Scientific	Isotemp	NA	Solids methods
Incubator	2000	Fisher Scientific	307	NA	3 BOD units
Water Purifier	2002	Milli-Q Water System	Synergy 185	NA	Reagent Water Purification

Frontenac, Kansas Laboratory

Instrument	Age	Manufacturer	Model Number	Detector	Total Analyses
Ion Analyzer (pH)	2001	Accumet	AP61	Electrolytic	pH
Dissolved Oxygen	2006	YSI	550A	Electrolytic	Bioassay
Conductivity Meter	2001	Accumet	AB30	Electrolytic	Specific Conductance
Autoclave	2001	Tutnauer Brinkman	3870E	NA	Microbiology General use
Incubator, water bath	2002	Precision	Precision	NA	Microbiology
Incubator, thermal	1995	Equatherm	C1574	NA	Microbiology
Bioassay Water Baths	2001	ISO Temp	2100	NA	Total 5 units for Bioassay
Balance	1990	Metler	AE-240	Gravimetric	Bioassay

ATTACHMENT IVA

PASI – LENEXA KANSAS FLOOR PLAN



LEGEND

- SAFETY SHOWER
- EYE WASH
- FIRST AID KIT
- FIRE EXTINGUISHER
- EMERGENCY LIGHTING
- EYE WASH BOTTLE
- FIRE ALARM PULL
- SPILL KIT
- WORKING LAB DEPENDENT PPE

EMERGENCY PHONE NUMBERS

911 FIRE POLICE AMBULANCE

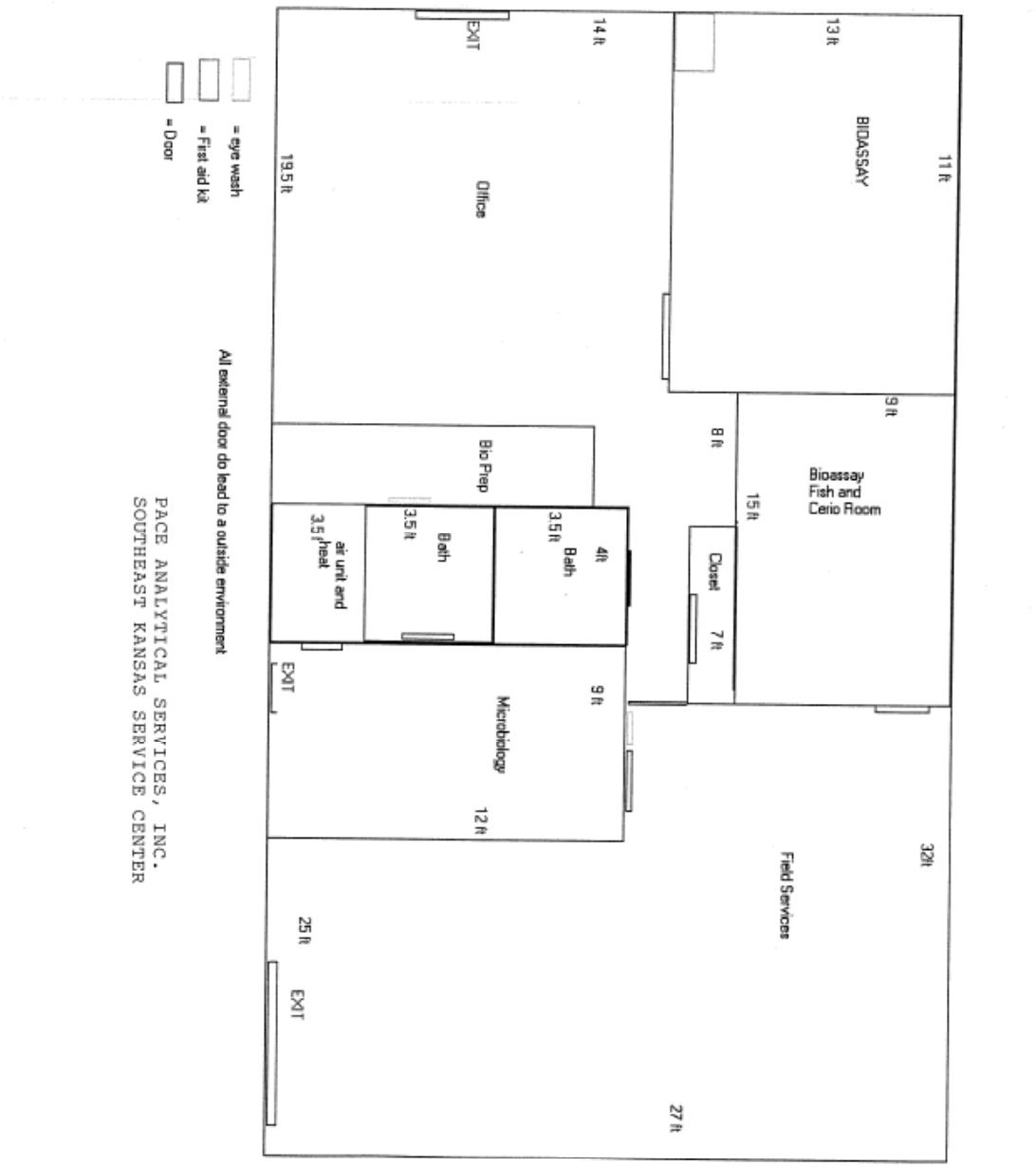
Poison 1 (800) 222-1222
KU Medical Center (913) 588-6633
Children's Mercy Hospital (816) 234-3000

Hospitals Shawnee Mission Med Center (913) 676-2000
I-35 and 75th Street
24-HR Emergency Services (913) 676-2215
Overland Park Regional Med Ctr (913) 541-5000
I-435 and Quivira
24-HR Emergency Services (913) 541-5338
Centennial (913) 594-6000
15525 W 99th Street
Lenexa KS

Evacuation Plan: In the event evacuation becomes necessary, assemble East of building, across the street

ATTACHMENT IVB

PASI – *FRONTENAC, KANSAS* FLOOR PLAN



ATTACHMENT V
PASI – *KANSAS* SOP LIST

SOP Number	SOP Title	DEPT
S-ALL-C-002-rev.1	BOTTLE ORDER DATABASE	CORP
S-ALL-C-005-rev.1	PACEPORT CUSTOMER FEEDBACK FORM	CORP
S-ALL-IT-001-rev.2	SYSTEM SECURITY AND INTEGRITY	CORP
S-ALL-IT-002-rev.2	SERVER BACKUP	CORP
S-ALL-KS-Q-013-rev.1	SUPPORT EQUIPMENT (KS ADDENDUM)	QA
S-ALL-Q-001-rev.9	PREPARATION OF SOPS	CORP
S-ALL-Q-002-rev.2	DOCUMENT MANAGEMENT	CORP
S-ALL-Q-003-rev.4	DOCUMENT NUMBERING	CORP
S-ALL-Q-004-rev.5	METHOD DETECTION LIMIT STUDIES	CORP
S-ALL-Q-007-rev.2	EPIC Pro: ACODE VALIDATION	CORP
S-ALL-Q-008-rev.1	EPIC Pro: ACODE ADDITION/MODIFICATION	CORP
S-ALL-Q-009-rev.2	LAB DOCUMENTATION	CORP
S-ALL-Q-010-rev.3	PROFICIENCY TESTING PROGRAM	CORP
S-ALL-Q-011-rev.3	AUDITS AND INSPECTIONS	CORP
S-ALL-Q-012-rev.2	CORRECTIVE AND PREVENTIVE ACTIONS	CORP
S-ALL-Q-013-rev.1	SUPPORT EQUIPMENT	CORP
S-ALL-Q-014-rev.2	QUALITY SYSTEM REPORTING	CORP
S-ALL-Q-016-rev.3	MANUAL INTEGRATION	CORP
S-ALL-Q-018-rev.2	MONITORING STORAGE UNITS	CORP
S-ALL-Q-020-rev.3	TRAINING PROCEDURES	CORP
S-ALL-Q-021-rev.3	SUB-SAMPLING (SAMPLE HOMOGENIZATION)	CORP
S-ALL-Q-025-rev.2	STANDARD AND REAGENT PREPARATION AND TRACEABILITY	CORP
S-ALL-Q-026-rev.1	SOFTWARE VALIDATION	CORP
S-ALL-Q-027-rev.1	VENDOR QUALIFICATION	CORP
S-ALL-Q-028-rev.1	LABTRACK SYSTEM	CORP
S-ALL-Q-029-rev.1	MINTMINER© DATA FILE REVIEW	CORP
S-ALL-Q-029-rev.1	3P PROGRAM: CONTINUOUS PROCESS IMPROVEMENT	CORP
S-ALL-Q-030-rev.2	EPIC PRO: DATA CHECKER	CORP
S-ALL-Q-033-rev.0	MCL VIOLATION REPORTING	CORP
S-ALL-S-001-rev.2	HAZARD ASSESSMENT	CORP
S-ALL-T-002-rev.1	LMS ADMIN TASK GUIDE	CORP
S-KS-C-001-rev.2	SAMPLE MANAGEMENT	CS
S-KS-C-002-rev.3	ASSEMBLY OF SAMPLE CONTAINER KITS	CS
S-KS-C-003-rev.2	SUBCONTRACTING SAMPLES	CS
S-KS-F-001-rev.2	FIELD MANUAL	FLD
S-KS-I-001-rev.7	ACIDITY	WET
S-KS-I-002-rev.9	ALKALINITY	WET
S-KS-I-003-rev.7	AMMONIA, NITROGEN BY 350.1	WET
S-KS-I-004-rev.8	BOD/CBOD	WET
S-KS-I-005-rev.7	CHEMICAL OXYGEN DEMAND	WET
S-KS-I-006-rev.3	CHLORINE (AT)	WET
S-KS-I-007-rev.7	CHLORINE (DPD)	WET

ATTACHMENT V (CONTINUED)
PASI – *KANSAS* SOP LIST

SOP Number	SOP Title	DEPT
S-KS-I-008-rev.9	HEXAVALENT CHROMIUM	WET
S-KS-I-010-rev.8	DISSOLVED OXYGEN	WET
S-KS-I-011-rev.3	FERROUS IRON	WET
S-KS-I-013-rev.2	TKN BY 351.2	WET
S-KS-I-014-rev.6	OIL AND GREASE BY 1664A	WET
S-KS-I-015-rev.2	HEM/SGT-HEM BY 9071B MOD	WET
S-KS-I-016-rev.7	TOTAL ORGANIC CARBON	WET
S-KS-I-017-rev.3	TURBIDITY	WET
S-KS-I-018-rev.7	pH IN WATER, SOIL, AND WASTE	WET
S-KS-I-019-rev.5	TOTAL RECOVERABLE PHENOLICS	WET
S-KS-I-020-rev.9	TOTAL SOLIDS	WET
S-KS-I-021-rev.9	TOTAL DISSOLVED SOLIDS	WET
S-KS-I-022-rev.10	TOTAL SUSPENDED SOLIDS	WET
S-KS-I-023-rev.7	SETTLEABLE SOLIDS	WET
S-KS-I-024-rev.7	VOLATILE AND FIXED SOLIDS	WET
S-KS-I-025-rev.7	CONDUCTANCE	WET
S-KS-I-027-rev.6	SULFIDE BY METHOD 9034	WET
S-KS-I-028-rev.7	SULFITE	WET
S-KS-I-029-rev.8	METHYLENE BLUE ACTIVE SUBSTANCES	WET
S-KS-I-030-rev.6	IGNITABILITY	WET
S-KS-I-031-rev.5	REACTIVE SULFIDE	WET
S-KS-I-032-rev.5	REACTIVE CYANIDE	WET
S-KS-I-033-rev.5	PAINT FILTER LIQUIDS TEST	WET
S-KS-I-034-rev.6	TOTAL HARDNESS	WET
S-KS-I-036-rev.6	TOTAL AND AMENABLE CYANIDE	WET
S-KS-I-037-rev.9	AUTOMATED CHLORIDE	WET
S-KS-I-038-rev.2	ORTHOPHOSPHATE	WET
S-KS-I-039-rev.8	NITRATE/NITRITE BY 353.2	WET
S-KS-I-040-rev.3	TOTAL PHOSPHORUS	WET
S-KS-I-042-rev.2	POLYQUAT	WET
S-KS-I-043-rev.5	ANIONS BY IC	WET
S-KS-I-044-rev.1	COLOR ANALYSIS	WET
S-KS-I-045-rev.1	SPECIFIC OXYGEN UPTAKE RATE	WET
S-KS-I-046-rev.0	TOTAL/VOLATILE/FIXED SOLIDS IN SLUDGES	WET
S-KS-I-047-rev.0	SULFIDE BY METHYLENE BLUE METHOD (SM4500-$S2^{-}$ D)	WET
S-KS-I-048-rev.0	SULFIDE BY IODOMETRIC TITRATION (SM 4500-$S2^{-}$ F)	WET
S-KS-IT-001-rev.2	TARGET DATA BACKUP	IT
S-KS-M-001-rev.1	MICRODIGESTION BY 3010A	MET
S-KS-M-002-rev.7	ACID DIGESTION BY 3010A	MET
S-KS-M-003-rev.7	ACID DIGESTION OF SOILS	MET
S-KS-M-004-rev.4	ACID DIGESTION OF WIPES	MET
S-KS-M-005-rev.9	METALS BY ICP-AES	MET
S-KS-M-006-rev.5	MERCURY PREP AND ANALYSIS	MET
S-KS-M-007-rev.3	CATION EXCHANGE CAPACITY	MET

ATTACHMENT V (CONTINUED)
PASI – *KANSAS* SOP LIST

SOP Number	SOP Title	DEPT
S-KS-M-008-rev.2	ICP METALS by 6010C	MET
S-KS-MB-001-rev.6	FECAL COLIFORM	MB
S-KS-MB-003-rev.6	HETEROTROPHIC PLATE COUNT	MB
S-KS-MB-004-rev.5	FECAL STREPTOCOCCUS AND ENTEROCOCCUS	MB
S-KS-MB-005-rev.5	SALMONELLA	MB
S-KS-MB-006-rev.5	SUITABILITY TEST	MB
S-KS-MB-007-rev.4	INHIBITORY RESIDUE TEST	MB
S-KS-MB-008-rev.4	ACUTE TOXICITY, C. DUBIA	TOX
S-KS-MB-009-rev.4	ACUTE TOXICITY, P. PROMELAS	TOX
S-KS-MB-010-rev.4	CHRONIC TOXICITY, C. DUBIA	TOX
S-KS-MB-011-rev.4	CHRONIC TOXICITY, P. PROMELAS	TOX
S-KS-MB-012-rev.4	PREPARING YTC	TOX
S-KS-MB-013-rev.4	CULTURING BRINE SHRIMP	TOX
S-KS-MB-014-rev.4	ACUTE REF TOX, C. DUBIA	TOX
S-KS-MB-015-rev.4	ACUTE REF TOX, P. PROMELAS	TOX
S-KS-MB-016-rev.4	CHRONIC REF TOX, P. PROMELAS	TOX
S-KS-MB-017-rev.4	CHRONIC REF TOX, C. DUBIA	TOX
S-KS-MB-018-rev.4	LIGHT INTENSITY/PHOTOPERIOD	TOX
S-KS-MB-019-rev.2	CULTURING OF C. DUBIA AND D. PULEX	TOX
S-KS-MB-020-rev.2	HATCHING AND CARE OF FATHEAD MINNOWS	TOX
S-KS-MB-021-rev.0	TOTAL COLIFORM AND E.COLI (QUANTI-TRAY)	MB
S-KS-MB-022-rev.0	BIOASSAY CHEMICAL TESTS	TOX
S-KS-O-001-rev.7	TCLP BY METHOD 1311	OEXT
S-KS-O-002-rev.3	SPLP BY METHOD 1312	OEXT
S-KS-O-003-rev.4	ORGANIC EXTRACTION SPIKE FORTIFICATION VERIFICATION	OEXT
S-KS-O-004-rev.7	EDB AND DBCP IN WATER BY EPA 8011	SVOA
S-KS-O-005-rev.10	DRO BY MODIFIED 8015	SVOA
S-KS-O-007-rev.8	PCBs IN WATER AND SOIL	SVOA
S-KS-O-008-rev.7	PCBs IN OIL AND WIPES	SVOA
S-KS-O-012-rev.10	VOLATILE ORGANIC COMPOUNDS BY 8260B	VOA
S-KS-O-013-rev.10	BNAs BY METHOD 8270C	SVOA
S-KS-O-014-rev.7	EPH BY METHOD OA-2	SVOA
S-KS-O-015-rev.4	EXTRACTABLE HYDROCARBONS BY OKLAHOMA DRO	SVOA
S-KS-O-016-rev.3	PURGABLE HYDROCARBONS BY OKLAHOMA GRO	VOA
S-KS-O-017-rev.6	PERCENT MOISTURE IN SOIL	OEXT
S-KS-O-018-rev.6	EDB & DBCP IN WATER BY 504.1	SVOA
S-KS-O-019-rev.8	VOCs IN WATER BY 602	VOA
S-KS-O-020-rev.5	ORGANOCHLORINE PEST/PCB BY EPA 608	SVOA
S-KS-O-022-rev.3	VOCs IN WATER BY 624	VOA
S-KS-O-023-rev.4	BNAs BY METHOD 625	SVOA
S-KS-O-024-rev.2	TPH-DRO/ORO BY 8270C	SVOA
S-KS-O-025-rev.2	TPH-GRO BY 8260B	VOA
S-KS-O-026-rev.1	VOLATILE AROMATICS/GRO BY 8021B AND 8015B/C	VOA
S-KS-O-027-rev.1	DRO BY 8015B/C	SVOA

ATTACHMENT V (CONTINUED)
PASI – *KANSAS* SOP LIST

SOP Number	SOP Title	DEPT
S-KS-O-028-rev.2	PAHS BY SIM 8270C	SVOA
S-KS-O-029-rev.1	SEPARATORY FUNNEL EXTRACTION	OEXT
S-KS-O-032-rev.1	MICROWAVE SOIL EXTRACTION	OEXT
S-KS-O-033-rev.1	VPH BY OA-1	VOA
S-KS-O-035-rev.0	WASTE DILUTION	OEXT
S-KS-O-036-rev.0	PCB EXTRACT CLEANUP	OEXT
S-KS-O-037-rev.0	TNRCC 1005	SVOA
S-KS-O-038-rev.0	SILICA GEL CLEANUP	OEXT
S-KS-Q-001-rev.5	LAB GLASSWARE WASHING	QA
S-KS-Q-003-rev.6	CONTROL CHART GENERATION AND ANALYSIS	QA
S-KS-Q-005-rev.5	DATA REDUCTION, REVIEW AND REPORTING	QA
S-KS-Q-006-rev.2	RECEIPT AND STORAGE OF LAB SUPPLIES	QA
S-KS-Q-007-rev.5	LABORATORY SECURITY PROCEDURES	QA
S-KS-Q-011-rev.3	REAGENT WATER QUALITY	QA
S-KS-Q-012-rev.3	SIGNIFICANT FIGURES AND ROUNDING	QA
S-KS-Q-019-rev.3	LAB DATA FILING AND ARCHIVING	QA
S-KS-Q-020-rev.2	USDA REGULATED SOIL	QA
S-KS-Q-022-rev.2	ESTIMATION OF UNCERTAINTY	QA
S-KS-Q-024-rev.2	INSTRUMENT TRANSPORT	QA
S-KS-Q-025-rev.2	A2LA TERMS AND SYMBOLS	QA
S-KS-Q-026-rev.3	PURCHASING OF LAB SUPPLIES	QA
S-KS-Q-027-rev.0	SAMPLE COMPOSITING	QA
S-KS-Q-028-rev.3	CUSTOMER COMPLAINT RESOLUTION	QA
S-KS-Q-038-rev.0	LABORATORY HOUSEKEEPING	QA
S-KS-S-002-rev.2	WASTE HANDLING	WASTE
S-KS-S-003-rev.0	WASTE MGMT TRAINING REQUIREMENTS	WASTE
S-KS-SM-rev.4	PACE KANSAS SAFETY MANUAL REV.4	SAFETY

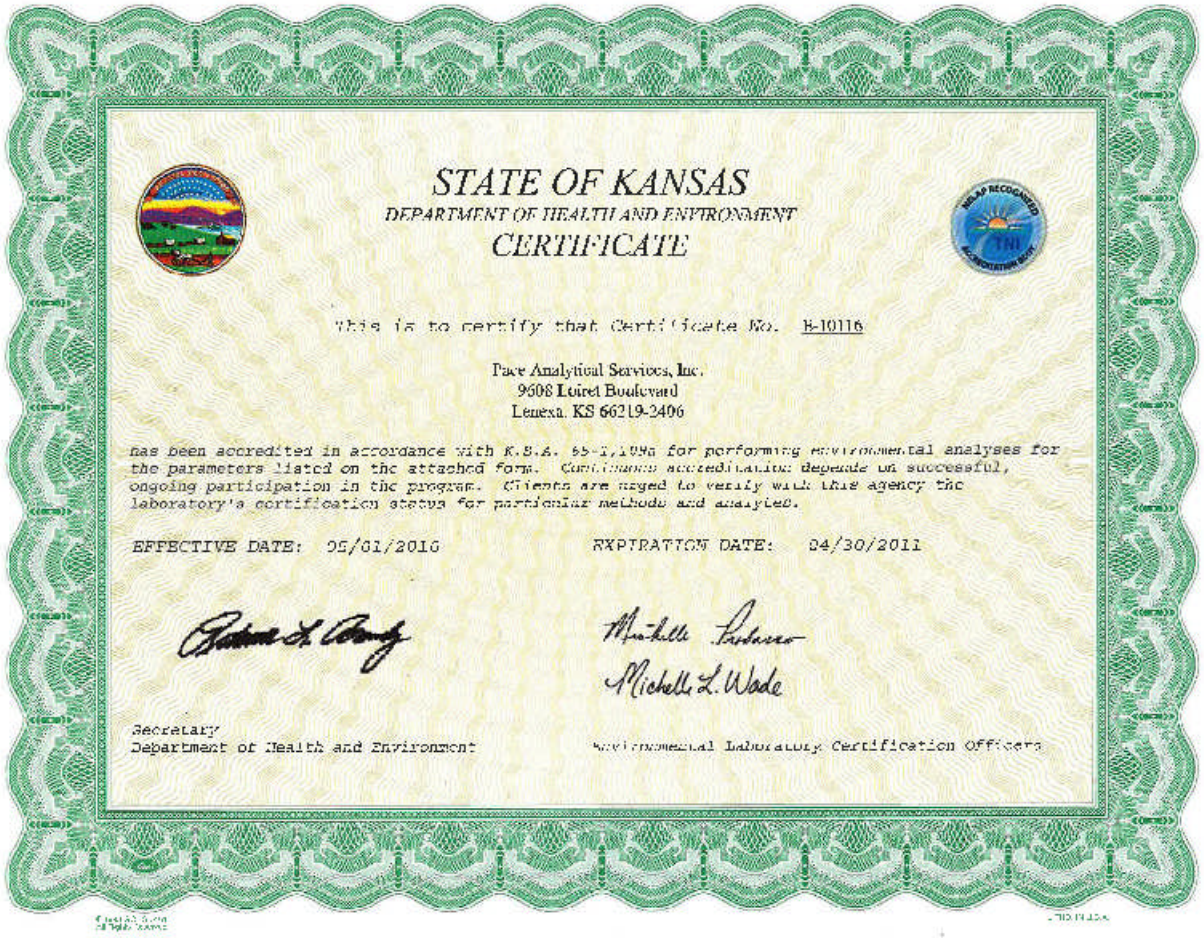
ATTACHMENT VI

PASI – *KANSAS* CERTIFICATION LIST

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
A2LA	Wyoming STR	A2LA	2456.01	7/31/2012
Arkansas	Hazardous Waste	Dept of Environmental Quality	02-05532	2/2/2011
Arkansas	Waste Water	Dept of Environmental Quality	02-05532	2/2/2011
Arkansas	Bioassay-WET	Dept of Environmental Quality	02-05532	2/2/2011
Illinois	Hazardous Waste	Illinois EPA	200030	2/4/2011
Illinois	Waste Water	Illinois EPA	200030	2/4/2011
Iowa	Hazardous Waste	Dept of Natural Resources	118	7/1/2012
Iowa	Waste Water	Dept of Natural Resources	118	7/1/2012
Iowa	UST Program	Dept of Natural Resources	118	7/1/2012
Kansas	Drinking Water	Dept of Health & Environment	E-10116	4/30/2011
Kansas	Hazardous Waste	Dept of Health & Environment	E-10116	4/30/2011
Kansas	Microbiology	Dept of Health & Environment	E-10116	4/30/2011
Kansas	Waste Water	Dept of Health & Environment	E-10116	4/30/2011
Kansas	Bioassay-WET	Dept of Health & Environment	E-10116	4/30/2011
Louisiana	Hazardous Waste	Dept of Environmental Quality	3055	6/30/2011
Louisiana	Waste Water	Dept of Environmental Quality	3055	6/30/2011
Louisiana	Bioassay-WET	Dept of Environmental Quality	3055	6/30/2011
Nevada	Waste Water	Division of Environmental Protection	KS00021208A	7/31/2011
Nevada	Hazardous Waste	Division of Environmental Protection	KS00021208A	7/31/2011
Oklahoma	Waste Water/Sludge	Dept of Environmental Quality	9205	8/31/2011
Oklahoma	Microbiology	Dept of Environmental Quality	9935	8/31/2011
Oklahoma	Bioassay-WET	Dept of Environmental Quality	9935	8/31/2011
Texas	Bioassay-WET	Texas Commission on Environmental Quality	T104704407-08-TX	6/30/2011
Texas	Hazardous Waste	Texas Commission on Environmental Quality	T104704407-08-TX	6/30/2011
Texas	Waste Water	Texas Commission on Environmental Quality	T104704407-08-TX	6/30/2011
USDA	Foreign Soil Import	USDA (Renewal in Process)	S-64040	1/13/2012
Utah	Hazardous Waste	Dept of Health	PASKS	5/1/2011
Utah	Bioassay-WET	Dept of Health	PASKS	5/1/2011

ATTACHMENT VII

PASI – *KANSAS* NELAP CERTIFICATION LIST



STATE OF KANSAS
DEPARTMENT OF HEALTH AND ENVIRONMENT
CERTIFICATE

This is to certify that Certificate No. E-10116

Pace Analytical Services, Inc.
9608 Loiret Boulevard
Lenexa, KS 66219-2406

has been accredited in accordance with K.S.A. 65-1,109a for performing environmental analyses for the parameters listed on the attached form. Continuous accreditation depends on successful, ongoing participation in the program. Clients are urged to verify with this agency the laboratory's certification status for particular methods and analytes.

EFFECTIVE DATE: 05/01/2010 EXPIRATION DATE: 04/30/2011

Secretary
Department of Health and Environment

Michelle L. Wade
Environmental Laboratory Certification Officers

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY ACCREDITATION
SAFE DRINKING WATER ACT PROGRAM - DRINKING WATER MATRIX

This certificate supersedes all previous certificates

Pace Analytical Services, Inc.
9608 Loiret Boulevard
Lenexa, KS 66219-2406

Certificate Number: E-10116
Effective Date: 05/01/2010
Expiration Date: 04/30/2011
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory accreditation in accordance with K.S.A. 65-1, 109a for the following:

****DEMANDS**
Dissolved Organic Carbon {SM 5310 C}
Total Organic Carbon {SM 5310 C}

****METALS**
Aluminum {EPA 200.7}
Barium {EPA 200.7}
Beryllium {EPA 200.7}
Cadmium {EPA 200.7}
Calcium {EPA 200.7}
Chromium {EPA 200.7}
Copper {EPA 200.7}
Iron {EPA 200.7}
Magnesium {EPA 200.7}
Manganese {EPA 200.7}
Mercury {EPA 245.1}
Nickel {EPA 200.7}
Silver {EPA 200.7}
Sodium {EPA 200.7}
Zinc {EPA 200.7}

****MICROBIOLOGY - ESWTR2**
E. Coli {Colilert System}
E. Coli {SM 9223 B}

****MICROBIOLOGY - Filtration**
Heterotrophic Plate Count {SM 9215 B}
Total Coliforms {SM 9223 B}

****MICROBIOLOGY - GWR**
E. Coli {Colilert System}
E. Coli {SM 9223 B}

****MICROBIOLOGY - TCR**
Total Coliform & E. Coli {Colilert System}
Total Coliform & E. Coli {SM 9223 B}

****MINERALS**
Alkalinity {SM 2320 B}
Bromide {EPA 300.0}
Chloride {EPA 300.0}
Fluoride {EPA 300.0}
Hardness {EPA 200.7}
Hardness {SM 2340 B}
Hardness {SM 2340 C}
Sulfate {EPA 300.0}

****MISCELLANEOUS**
Chlorine - Total {SM 4500-Cl E}
Chlorine - Total {SM 4500-Cl G}
Color {SM 2120 B}

Conductivity {SM 2510 B}
Cyanide {SM 4500-CN E}
Cyanide, Amenable to CL {SM 4500-CN G}
Hydrogen Ion (pH) {EPA 150.1}
Hydrogen Ion (pH) {SM 4500-H B}
Ortho-phosphate {EPA 365.1}
Temperature {SM 2550 B}
Turbidity {EPA 180.1}
Turbidity {SM 2130 B}

****NUTRIENTS**
Nitrate {EPA 300.0}
Nitrate {EPA 353.2}
Nitrite {EPA 300.0}
Nitrite {EPA 353.2}

****ORGANIC CHEMISTRY DBCP/EDB**
{EPA 504.1} Dibromochloropropane (DBCP)
{EPA 504.1} Ethylene dibromide (EDB, 1,2-Dibromoethane)

****RESIDUES**
Residue, Filterable (TDS) {SM 2540 C}

End of Parameter List

This certificate supersedes all previous certificates

Pace Analytical Services, Inc.
9608 Loiret Boulevard
Lenexa, KS 66219-2406

Certificate Number: E-10116
Effective Date: 05/01/2010
Expiration Date: 04/30/2011
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory accreditation in accordance with K.S.A. 65-1, 109a for the following:

****BIOMONITORING**
Ceriodaphnia dubia, acute {EPA 2002.0}
Ceriodaphnia dubia, chronic {EPA 1002}
Daphnia pulex, acute {EPA 2021.0}
Pimephales promelas, acute {EPA 2000.0}
Pimephales promelas, chronic {EPA 1000}
Pimephales promelas, chronic {EPA 1001}

****DEMANDS**
BOD {SM 5210 B}
cBOD {SM 5210 B}
COD {EPA 410.4}
COD {SM 5220 D}
Oxygen, Dissolved {SM 4500-O G}
Specific Oxygen Uptake {SM 2710 B}
Total Organic Carbon {SM 5310 B, C, or D}

****METALS**
Aluminum {EPA 200.7}
Antimony {EPA 200.7}
Arsenic {EPA 200.7}
Barium {EPA 200.7}
Beryllium {EPA 200.7}
Boron {EPA 200.7}
Cadmium {EPA 200.7}
Calcium {EPA 200.7}
Chromium {EPA 200.7}
Chromium, VI {SM 3500-Cr D (18th, 19th)}
Cobalt {EPA 200.7}
Copper {EPA 200.7}
Iron {EPA 200.7}
Iron {SM 3500-Fe B}
Lead {EPA 200.7}
Magnesium {EPA 200.7}
Manganese {EPA 200.7}
Mercury {EPA 245.1}
Molybdenum {EPA 200.7}
Nickel {EPA 200.7}
Potassium {EPA 200.7}
Selenium {EPA 200.7}
Silver {EPA 200.7}
Sodium {EPA 200.7}
Thallium {EPA 200.7}
Tin {EPA 200.7}
Titanium {EPA 200.7}
Vanadium {EPA 200.7}
Zinc {EPA 200.7}

****METALS - 503 Regs**
Arsenic {EPA 6010}
Cadmium {EPA 6010}

Chromium, Total {EPA 6010}
Copper {EPA 6010}
Lead {EPA 6010}
Mercury {EPA 7470}
Mercury {EPA 7471}
Molybdenum {EPA 6010}
Nickel {EPA 6010}
Selenium {EPA 6010}
Zinc {EPA 6010}

**MICROBIOLOGY
E. Coli {Colilert System}
E. Coli {SM 9223 B}
Fecal Coliforms {SM 9222 D (MF)}
Fecal Streptococci {SM 9230 C (MF)}

**MINERALS
Acidity {SM 2310 B}
Alkalinity {SM 2320 B}
Chloride {EPA 300.0}
Chloride {SM 4500-Cl E}
Fluoride {EPA 300.0}
Hardness {EPA 200.7}
Hardness {SM 2340 B or C}
Sulfate {EPA 300.0}
Sulfide {SM 4500-S-2 D}
Sulfide {SM 4500-S-2 F}
Sulfite {SM 4500-SO3-2 B}

**MISCELLANEOUS
Available Cyanide {SM 4500-CN G}
Bromide {EPA 300.0}
Chlorine - Total {SM 4500-Cl G}
Color {SM 2120 B}
Conductivity {EPA 120.1}
Conductivity {SM 2510 B}
Cyanide {SM 4500-CN E}
Hydrogen Ion (pH) {SM 4500-H B}
Oil & Grease {EPA 1664}
Phenolics {EPA 420.1}
Surfactants {SM 5540 C}
Temperature {SM 2550 B}
Turbidity {EPA 180.1}
Turbidity {SM 2130 B}

**NUTRIENTS
Ammonia {EPA 350.1}
K Nitrogen {EPA 351.2}
Nitrate-Nitrite {EPA 300.0}
Nitrate-Nitrite {EPA 353.2}
Nitrate {EPA 300.0}
Nitrate {EPA 353.2}
Nitrite {EPA 300.0}
Nitrite {EPA 353.2}
Organic-Nitrogen {TKN-NH3-CAL}
Ortho-phosphate {EPA 365.1}
Phosphorus {EPA 365.4}

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC)
{EPA 602} Benzene
{EPA 602} Ethylbenzene

{EPA 602} Toluene

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 624} 1,1-Dichloroethane
{EPA 624} 1,1-Dichloroethene
{EPA 624} 1,1,1-Trichloroethane
{EPA 624} 1,1,2-Trichloroethane
{EPA 624} 1,1,2,2-Tetrachloroethane
{EPA 624} 1,2-Dichlorobenzene
{EPA 624} 1,2-Dichloroethane
{EPA 624} 1,2-Dichloropropane
{EPA 624} 1,3-Dichlorobenzene
{EPA 624} 1,4-Dichlorobenzene
{EPA 624} 2-Chloroethyl vinyl ether
{EPA 624} Acrolein
{EPA 624} Acrylonitrile
{EPA 624} Benzene
{EPA 624} Bromodichloromethane
{EPA 624} Bromoform
{EPA 624} Bromomethane (Methyl bromide)
{EPA 624} Carbon tetrachloride
{EPA 624} Chlorobenzene
{EPA 624} Chloroethane
{EPA 624} Chloroform
{EPA 624} Chloromethane (Methyl chloride)
{EPA 624} cis-1,3-Dichloropropene
{EPA 624} Dibromochloromethane
{EPA 624} Ethylbenzene
{EPA 624} Methylene chloride (Dichloromethane)
{EPA 624} Tetrachloroethene (Perchloroethylene)
{EPA 624} Toluene
{EPA 624} trans-1,2-Dichloroethene
{EPA 624} trans-1,3-Dichloropropene
{EPA 624} Trichloroethene (Trichloroethylene)
{EPA 624} Trichlorofluoromethane
{EPA 624} Vinyl chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 608} PCB-1016
{EPA 608} PCB-1221
{EPA 608} PCB-1232
{EPA 608} PCB-1242
{EPA 608} PCB-1248
{EPA 608} PCB-1254
{EPA 608} PCB-1260

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 625} 1,2,4-Trichlorobenzene
{EPA 625} 2-Chloronaphthalene
{EPA 625} 2-Chlorophenol
{EPA 625} 2-Methyl-4,6-dinitrophenol
{EPA 625} 2-Nitrophenol
{EPA 625} 2,2'-Oxybis(1-chloropropane)
{EPA 625} 2,4-Dichlorophenol
{EPA 625} 2,4-Dimethylphenol
{EPA 625} 2,4-Dinitrophenol
{EPA 625} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 625} 2,4,6-Trichlorophenol
{EPA 625} 2,6-Dinitrotoluene
{EPA 625} 3,3'-Dichlorobenzidine
{EPA 625} 4-Bromophenyl phenyl ether

{EPA 625} 4-Chloro-3-methylphenol
{EPA 625} 4-Chlorophenyl phenyl ether
{EPA 625} 4-Nitrophenol
{EPA 625} Acenaphthene
{EPA 625} Acenaphthylene
{EPA 625} Anthracene
{EPA 625} Benzidine
{EPA 625} Benzo(a)anthracene
{EPA 625} Benzo(a)pyrene
{EPA 625} Benzo(b)fluoranthene
{EPA 625} Benzo(g,h,i)perylene
{EPA 625} Benzo(k)fluoranthene
{EPA 625} Benzyl butyl phthalate
{EPA 625} Bis(2-chloroethoxy)methane
{EPA 625} Bis(2-chloroethyl)ether
{EPA 625} Bis(2-ethylhexyl)phthalate
{EPA 625} Chrysene
{EPA 625} Di-n-butyl phthalate
{EPA 625} Di-n-octyl phthalate
{EPA 625} Dibenzo(a,h)anthracene
{EPA 625} Diethyl phthalate
{EPA 625} Dimethyl phthalate
{EPA 625} Fluoranthene
{EPA 625} Fluorene
{EPA 625} Hexachlorobenzene
{EPA 625} Hexachlorobutadiene
{EPA 625} Hexachlorocyclopentadiene
{EPA 625} Hexachloroethane
{EPA 625} Indeno(1,2,3-cd) pyrene
{EPA 625} Isophorone
{EPA 625} N-nitroso-di-n-propylamine (NDPA)
{EPA 625} N-nitrosodimethylamine (NDMA)
{EPA 625} N-Nitrosodiphenylamine
{EPA 625} Naphthalene
{EPA 625} Nitrobenzene
{EPA 625} Pentachlorophenol
{EPA 625} Phenanthrene
{EPA 625} Phenol
{EPA 625} Pyrene

**RESIDUES
Fixed & Volatile Solids in Solids & Semi-solid samples, Total {SM 2540 G}
Residue, Filterable (TDS) {SM 2540 C}
Residue, Non Filterable (TSS) {SM 2540 D}
Residue, Settleable {SM 2540 F}
Residue, Total {SM 2540 B}
Residue, Volatile {EPA 160.4}

End of Parameter List

This certificate supersedes all previous certificates

Pace Analytical Services, Inc.
9608 Loiret Boulevard
Lenexa, KS 66219-2406

Certificate Number: E-10116
Effective Date: 05/01/2010
Expiration Date: 04/30/2011
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory accreditation in accordance with K.S.A. 65-1, 109a for the following:

****CHARACTERISTICS**
Ignitability {EPA 1010}
Synthetic-Precipitation Leaching Procedure {EPA 1312}
Toxic-Characteristic-Leaching Procedure {EPA 1311}

****DEMANDS**
Total Organic Carbon {EPA 9060}

****METALS**
Aluminum {EPA 6010}
Antimony {EPA 6010}
Arsenic {EPA 6010}
Barium {EPA 6010}
Beryllium {EPA 6010}
Boron {EPA 6010}
Cadmium {EPA 6010}
Calcium {EPA 6010}
Chromium {EPA 6010}
Chromium, VI {EPA 7196}
Cobalt {EPA 6010}
Copper {EPA 6010}
Iron {EPA 6010}
Lead {EPA 6010}
Lithium {EPA 6010}
Magnesium {EPA 6010}
Manganese {EPA 6010}
Mercury {EPA 7470}
Mercury {EPA 7471}
Molybdenum {EPA 6010}
Nickel {EPA 6010}
Potassium {EPA 6010}
Selenium {EPA 6010}
Silica {EPA 6010}
Silver {EPA 6010}
Sodium {EPA 6010}
Strontium {EPA 6010}
Thallium {EPA 6010}
Tin {EPA 6010}
Titanium {EPA 6010}
Vanadium {EPA 6010}
Zinc {EPA 6010}

****MINERALS**
Bromide {EPA 9056}
Chloride {EPA 9056}
Fluoride {EPA 9056}
Sulfate {EPA 9056}
Sulfide {EPA 9030}
Sulfide {EPA 9034}

**MISCELLANEOUS
Cation-Exchange Capacity of Soils {EPA 9081}
Conductivity {EPA 9050}
Cyanide {EPA 9010}
Cyanide {EPA 9012}
Cyanide, Amenable to CL {EPA 9010}
Cyanide, Amenable to CL {EPA 9012}
Hydrogen Ion (pH) {EPA 9040}
Hydrogen Ion (pH) {EPA 9045}
Oil & Grease {EPA 9070}
Oil & Grease {EPA 9071}
Paint Filter Liquids Test {EPA 9095}
Phenolics {EPA 9065}
Phenolics {EPA 9066}

**NUTRIENTS
Nitrate {EPA 9056}
Nitrite {EPA 9056}
Ortho-phosphate {EPA 9056}

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC)
{EPA 8021} Benzene
{EPA 8021} Ethylbenzene
{EPA 8021} meta-Xylene
{EPA 8021} ortho-Xylene
{EPA 8021} para-Xylene
{EPA 8021} Toluene

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 8260} 1-Chlorohexane
{EPA 8260} 1,1-Dichloroethane
{EPA 8260} 1,1-Dichloroethene
{EPA 8260} 1,1-Dichloropropene
{EPA 8260} 1,1,1-Trichloroethane
{EPA 8260} 1,1,1,2-Tetrachloroethane
{EPA 8260} 1,1,2-Trichloroethane
{EPA 8260} 1,1,2,2-Tetrachloroethane
{EPA 8260} 1,2-Dichlorobenzene
{EPA 8260} 1,2-Dichloroethane
{EPA 8260} 1,2-Dichloropropane
{EPA 8260} 1,2,3-Trichlorobenzene
{EPA 8260} 1,2,3-Trichloropropane
{EPA 8260} 1,2,4-Trichlorobenzene
{EPA 8260} 1,2,4-Trimethylbenzene
{EPA 8260} 1,3-Dichlorobenzene
{EPA 8260} 1,3-Dichloropropane
{EPA 8260} 1,3,5-Trimethylbenzene
{EPA 8260} 1,4-Dichlorobenzene
{EPA 8260} 1,4-Dioxane
{EPA 8260} 2-Chloroethyl vinyl ether
{EPA 8260} 2-Chlorotoluene
{EPA 8260} 2-Hexanone
{EPA 8260} 2-Nitropropane
{EPA 8260} 2,2-Dichloropropane
{EPA 8260} 4-Chlorotoluene
{EPA 8260} 4-Isopropyltoluene
{EPA 8260} 4-Methyl-2-Pentanone (MIBK)
{EPA 8260} Acetone
{EPA 8260} Acetonitrile
{EPA 8260} Acrolein
{EPA 8260} Acrylonitrile

{EPA 8260} Benzene
{EPA 8260} Bromobenzene
{EPA 8260} Bromochloromethane
{EPA 8260} Bromodichloromethane
{EPA 8260} Bromoform
{EPA 8260} Bromomethane (Methyl bromide)
{EPA 8260} Carbon disulfide
{EPA 8260} Carbon tetrachloride
{EPA 8260} Chlorobenzene
{EPA 8260} Chloroethane
{EPA 8260} Chloroform
{EPA 8260} Chloromethane (Methyl chloride)
{EPA 8260} cis-1,2-Dichloroethylene
{EPA 8260} cis-1,3-Dichloropropene
{EPA 8260} cis-1,4-Dichloro-2-butene
{EPA 8260} Dibromochloromethane
{EPA 8260} Dibromochloropropane (DBCP)
{EPA 8260} Dibromofluoromethane
{EPA 8260} Dibromomethane
{EPA 8260} Dichlorodifluoromethane
{EPA 8260} Dichloromethane (Methylene chloride)
{EPA 8260} Diethyl ether
{EPA 8260} Ethanol
{EPA 8260} Ethyl acetate
{EPA 8260} Ethylbenzene
{EPA 8260} Ethylene dibromide (EDB, 1,2-Dibromoethane)
{EPA 8260} Hexachlorobutadiene
{EPA 8260} Iodomethane
{EPA 8260} Isobutyl Alcohol
{EPA 8260} Isopropylbenzene
{EPA 8260} meta-Xylene
{EPA 8260} Methyl ethyl ketone
{EPA 8260} Methyl tert-butyl ether (MTBE)
{EPA 8260} n-Butyl alcohol
{EPA 8260} n-Butylbenzene
{EPA 8260} n-Propylbenzene
{EPA 8260} Naphthalene
{EPA 8260} ortho-Xylene
{EPA 8260} para-Xylene
{EPA 8260} Pyridine
{EPA 8260} sec-Butylbenzene
{EPA 8260} Styrene
{EPA 8260} t-Butyl alcohol
{EPA 8260} tert-Butylbenzene
{EPA 8260} Tetrachloroethene (Perchloroethylene)
{EPA 8260} Toluene
{EPA 8260} trans-1,2-Dichloroethylene
{EPA 8260} trans-1,3-Dichloropropene
{EPA 8260} trans-1,4-Dichloro-2-butene
{EPA 8260} Trichloroethene (Trichloroethylene)
{EPA 8260} Trichlorofluoromethane
{EPA 8260} Vinyl Acetate
{EPA 8260} Vinyl chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 8011} Dibromochloropropane (DBCP)
{EPA 8011} Ethylene dibromide (EDB, 1,2-Dibromoethane)
{EPA 8082} PCB-1016
{EPA 8082} PCB-1221
{EPA 8082} PCB-1232
{EPA 8082} PCB-1242

{EPA 8082} PCB-1248
{EPA 8082} PCB-1254
{EPA 8082} PCB-1260

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 8270} 1-Chloronaphthalene
{EPA 8270} 1,2-Dichlorobenzene
{EPA 8270} 1,2-Diphenylhydrazine
{EPA 8270} 1,2,4-Trichlorobenzene
{EPA 8270} 1,3-Dichlorobenzene
{EPA 8270} 1,4-Dichlorobenzene
{EPA 8270} 2-Chloronaphthalene
{EPA 8270} 2-Chlorophenol
{EPA 8270} 2-Methyl-4,6-Dinitrophenol
{EPA 8270} 2-Methylnaphthalene
{EPA 8270} 2-Methylphenol (o-Cresol)
{EPA 8270} 2-Nitroaniline
{EPA 8270} 2-Nitrophenol
{EPA 8270} 2,3,4,6-Tetrachlorophenol
{EPA 8270} 2,4-Dichlorophenol
{EPA 8270} 2,4-Dimethylphenol
{EPA 8270} 2,4-Dinitrophenol
{EPA 8270} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 8270} 2,4,5-Trichlorophenol
{EPA 8270} 2,4,6-Trichlorophenol
{EPA 8270} 2,6-Dinitrotoluene
{EPA 8270} 3-Methylphenol (m-Cresol)
{EPA 8270} 3-Nitroaniline
{EPA 8270} 3,3'-Dichlorobenzidine
{EPA 8270} 4-Bromophenyl phenyl ether
{EPA 8270} 4-Chloro-3-methylphenol
{EPA 8270} 4-Chloroaniline
{EPA 8270} 4-Chlorophenyl phenyl ether
{EPA 8270} 4-Methylphenol (p-Cresol)
{EPA 8270} 4-Nitroaniline
{EPA 8270} 4-Nitrophenol
{EPA 8270} 7,12-Dimethylbenz(a)anthracene
{EPA 8270} Acenaphthene
{EPA 8270} Acenaphthylene
{EPA 8270} Acetophenone
{EPA 8270} Aniline
{EPA 8270} Anthracene
{EPA 8270} Benzidine
{EPA 8270} Benzoic acid
{EPA 8270} Benzo(a)anthracene
{EPA 8270} Benzo(a)pyrene
{EPA 8270} Benzo(b)fluoranthene
{EPA 8270} Benzo(g,h,i)perylene
{EPA 8270} Benzo(k)fluoranthene
{EPA 8270} Benzyl alcohol
{EPA 8270} Bis(2-chloroethoxy)methane
{EPA 8270} Bis(2-chloroethyl)ether
{EPA 8270} Bis(2-chloroisopropyl)ether
{EPA 8270} Bis(2-ethylhexyl)phthalate
{EPA 8270} Butyl benzyl phthalate
{EPA 8270} Chrysene
{EPA 8270} Di-n-butyl phthalate
{EPA 8270} Di-n-octyl phthalate
{EPA 8270} Dibenzofuran
{EPA 8270} Dibenzo(a,e)pyrene
{EPA 8270} Dibenzo(a,h)anthracene

{EPA 8270} Dibenz(a,j)acridine
{EPA 8270} Diethyl phthalate
{EPA 8270} Dimethyl phthalate
{EPA 8270} Diphenylamine
{EPA 8270} Fluoranthene
{EPA 8270} Fluorene
{EPA 8270} Hexachlorobenzene
{EPA 8270} Hexachlorobutadiene
{EPA 8270} Hexachlorocyclopentadiene
{EPA 8270} Hexachloroethane
{EPA 8270} Indeno(1,2,3-cd) pyrene
{EPA 8270} Isophorone
{EPA 8270} N-nitroso-di-n-butylamine (NDBA)
{EPA 8270} N-nitroso-di-n-propylamine (NDPA)
{EPA 8270} N-nitrosodimethylamine (NDMA)
{EPA 8270} N-Nitrosodiphenylamine
{EPA 8270} N-Nitrosomethylethylamine
{EPA 8270} Naphthalene
{EPA 8270} Nitrobenzene
{EPA 8270} Pentachlorophenol
{EPA 8270} Phenanthrene
{EPA 8270} Phenol
{EPA 8270} Pyrene
{EPA 8270} Pyridine
{EPA 8270} Thiophenol

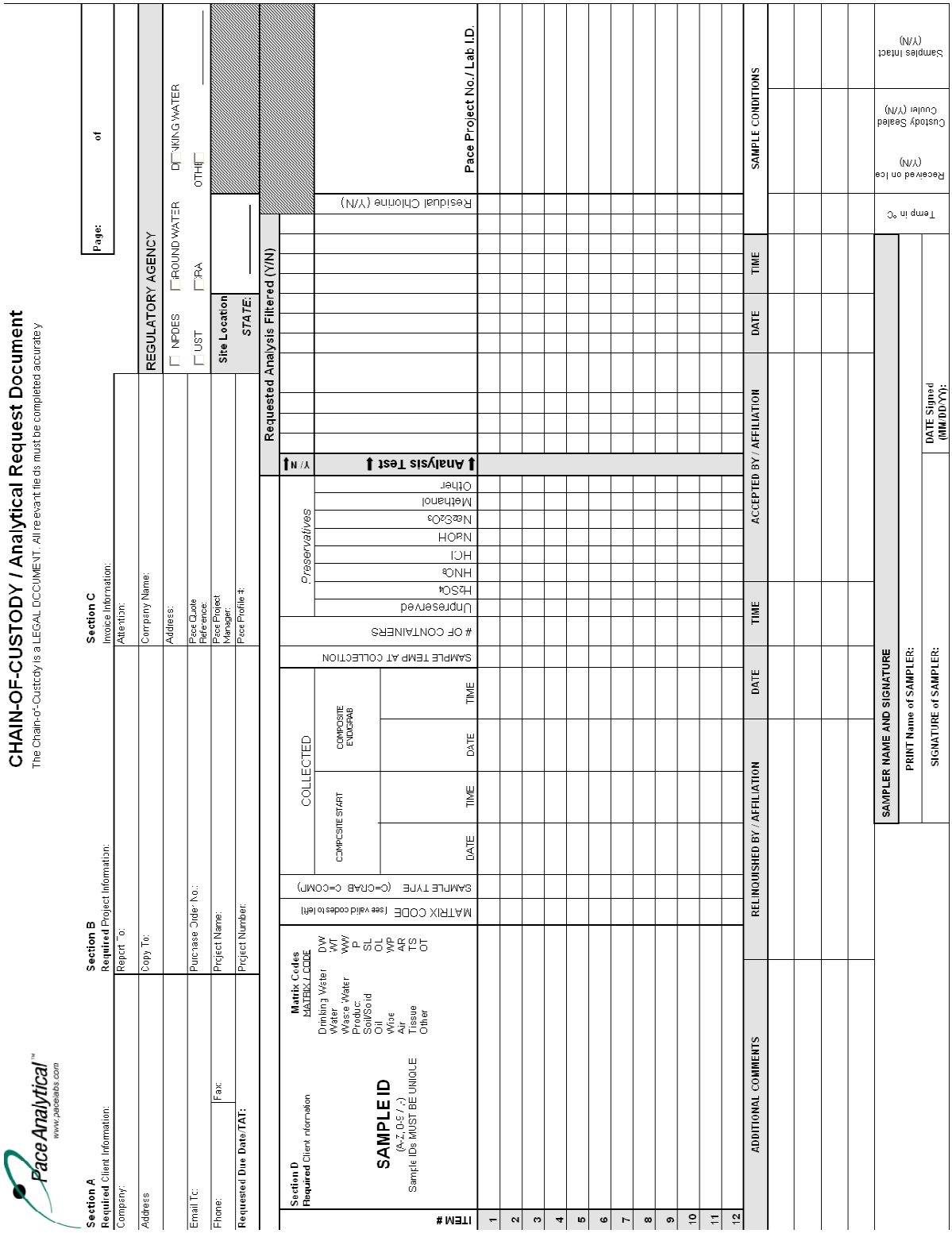
**TOTAL PETROLEUM HYDROCARBONS
{EPA 8015} DRO
{EPA 8015} GRO

End of Parameter List

US EPA ARCHIVE DOCUMENT

ATTACHMENT VIII

PASI – CHAIN OF CUSTODY



ATTACHMENT IX
METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

Parameter Name	Method (s)	Container[1]	Volume	Preservation[2]	Maximum Holding Time
Bacterial Tests:					
Coliform, total, fecal, and *E. coli*	SM 9222D, SM 9223B Colilert©	PA, G	120 mL	Cool, <10 °C, Na2S2O3	6 hours
Fecal streptococci	SM 9230C	PA, G	120 mL	Cool, <10 °C, Na2S2O3	6 hours
Enterococci	SM 9230C	PA, G	120 mL	Cool, <10 °C, Na2S2O3	6 hours
Heterotrophic Plate Count	SM 9215B	PA, G	120 mL	Cool, <10 °C, Na2S2O3	8 hours
Aquatic Toxicity Tests:					
Toxicity, acute	EPA 2002, 1002	P, FP, G	1 Gallon	Cool, ≤6 °C	36 hours
Toxicity, chronic	EPA 1000, 1002	P, FP, G	2.5 Gallon	Cool, ≤6 °C	36 hours
Inorganic Tests Aqueous:					
Acidity	SM 2310B	P, FP, G	250 mL	Cool, ≤6 °	14 days
Alkalinity	SM 2320B	P, FP, G	250 mL	Cool, ≤6 °C	14 days
Ammonia	EPA 350.1	P, FP, G	250 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Biochemical oxygen demand	SM 5210B	P, FP, G	1 L	Cool, ≤6 °C	48 hours
Boron	EPA 200.7, 200.8	P, FP, or Quartz	250 mL	HNO3 to pH<2	6 months
Bromide	EPA 300.0, 9056	P, FP, G	250 mL	None required	28 days
Biochemical oxygen demand, carbonaceous	SM 5210B	P, FP G	1 L	Cool, ≤6 °C	48 hours
Chemical oxygen demand	EPA 410.4	P, FP, G	250 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Chloride	300.0, SM 4500-Cl E, 9056	P, FP, G	250 mL	None required	28 days
Chlorine, total residual	SM 4500-Cl G	P, G	250 mL	None required	Analyze within 15 minutes
Color	110.2, SM 2120B	P, FP, G	250 mL	Cool, ≤6 °C	48 hours
Cyanide, total	SM 4500-CN E, 9012A	P, FP, G	250 mL	Cool, ≤6 °C, NaOH to pH>12, reducing agent, ascorbic acid	14 days
Fluoride	EPA 300.0, 9056	P	250 mL	None required	28 days
Hardness	200.7, SM 2340B	P, FP, G	250 mL	HNO3 to pH<2	6 months
Hydrogen ion (pH)	150.1, SM 4500-H+B	P, FP, G	250 mL	None required	Analyze within 15 minutes
Kjeldahl and organic N	EPA 351.2	P, FP, G	250 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Chromium VI, Hexavalent	SM 3500-Cr D, 7196A	P, FP, G	250 mL	Cool, ≤6 °C	24 hours
Mercury (CVAA)	245.1	P, FP, G	250 mL	HNO3 to pH<2	28 days

Note: Contact your Pace Analytical Project Manager for bottle order assistance.

ATTACHMENT IX (CONTINUED)
METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

PASI – KANSAS

Parameter Name	Method (s)	Container[1]	Volume (mL)	Preservation[2]	Maximum Holding Time
Metals, except boron, chromium VI, and mercury.	EPA 200.7, 200.8 6010B, 6010C	P, FP, G	250 mL	HNO3 to pH<2, or at least 24 hours prior to analysis	6 months
Nitrate	300.0, 353.2, 9056	P, FP, G	250 mL	Cool, ≤6 °C	48 hours
Nitrate-nitrite	353.2	P, FP, G	250 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Nitrite	300.0, 353.2, 9056	P, FP, G	250 mL	Cool, ≤6 °C	48 hours
Oil and Grease	1664A	G	1 L	Cool to ≤6 °C, HCl or H2SO4 to pH<2.	28 days
Organic Carbon, Total	SM 5310C, 9060	P, FP, G	500 mL	Cool, ≤6 °C, H2SO4 pH<2.	28 days
Orthophosphate	365.1	P, FP, G	250 mL	Cool, ≤6 °C	Analyze within 48 hours
Oxygen, Dissolved Probe	SM 4500-O G	G	250 mL	None required	Analyze within 15 minutes
Phenols	420.1, 420.4, 9065/9066	G	500 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Phosphorous, total	365.4	P, FP, G	250 mL	Cool, ≤6 °C, H2SO4 to pH<2.	28 days
Residue, total	SM 2540B	P, FP, G	250 mL	Cool, ≤6 °C	7 days
Residue, Filterable	SM 2540C	P, FP, G	250 mL	Cool, ≤6 °C	7 days
Residue, Nonfilterable (TSS)	SM 2540D	P, FP, G	1 L	Cool, ≤6 °C	7 days
Residue, Settleable	SM 2540F	P, FP, G	1 L	Cool, ≤6 °C	48 hours
Residue, Volatile	160.4	P, FP, G	250 mL	Cool, ≤6 °C	7 days
Silica	200.7, 6010B	P or Quartz	250 mL	Cool, ≤6 °C	28 days
Specific conductance	120.1	P, FP, G	250 mL	Cool, ≤6 °C	28 days
Sulfate	300.0, 9056	P, FP, G	250 mL	Cool, ≤6 °C	28 days
Sulfide	SM 4500-S-2 F, SM 4500-S-2 D, 9030, 9034	P, FP, G	250 mL	Cool, ≤6 °C, add zinc acetate plus sodium hydroxide to pH>9.	7 days
Sulfite	SM 4500-SO3-2 B	P, FP, G	250 mL	None required	Analyze within 15 minutes
Surfactants	SM 5540C	P, FP, G	500 mL	Cool, ≤6 °C	48 hours
Temperature	SM 2550B	P, FP, G	250 mL	None required	Analyze
Turbidity	180.1	P, FP, G	250 mL	Cool, ≤6 °C	48 hours

(1) ‘‘P’’ is polyethylene; ‘‘FP’’ is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon), or other fluoropolymer, unless stated otherwise in this Table; ‘‘G’’ is glass; ‘‘PA’’ is any plastic that is made of a sterlizable material (polypropylene or other autoclavable plastic); ‘‘LDPE’’ is low density polyethylene.

(2) Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agent shown to be most effective is sodium thiosulfate (Na2S2O3).

Note: Contact your Pace Analytical Project Manager for bottle order assistance.

ATTACHMENT IX (CONTINUED)
METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE
PASI – KANSAS

Parameter	Method (s)			Container	Volume Needed (mL)	Preservation	Max Hold Time
	EPA Drinking Water	EPA Water	EPA Waste SW-846				
Aromatic and Halogenated Volatiles		601/602	8021B	40-mL vial	3 X 40 mL	pH<2 HCl, Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Volatiles	524.1/524.2			40-mL vial	3 X 40 mL	pH<2 HCl, Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Volatiles		624		40-mL vial	3 X 40 mL	pH<2 HCl, Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present, 2-chloro-ethylvinyl ether collect as unpreserved	14 Days (7 unpreserved)
Volatiles			8260B	40-mL vial	3 X 40 mL	pH<2 HCl, Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Volatiles, GRO, and Oxygenates, Missouri RBCA			8260B	40-mL vial	3 X 40 mL	pH> 11 TSP, Cool, ≤6 °C	14 Days
Gas Range Organics (GRO)			8015B/8015C, OK DEQ GRO	40-mL vial	3 X 40 mL	pH<2 HCl	14 Days
Volatile Petroleum Hydrocarbons (VPH)			OA1	40-mL vial	3 X 40 mL	pH<2 HCl	14 Days
EDB & DBCP	504.1		8011	40-mL vial	3 X 40 mL	Cool, ≤6°C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Base/Neutrals and Acids		625	8270C	G FP-lined amber	2 X 1000 mL	Cool, ≤6°C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.1/525.2			G FP-lined amber	2 X 1000 mL	Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	7/30 Days
Organochlorine Pesticides and PCB's		608	8081/8082	G FP-lined amber	2 X 1000 mL	Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Organophosphorous Pesticides			8141A	G FP-lined amber	2 X 1000 mL	Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Polynuclear Aromatic Hydrocarbons		610	8310	G FP-lined amber	2 X 1000 mL	Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Chlorinated Herbicides	515.1		8151	G FP-lined amber	2 X 1000 mL	Cool, ≤6 °C, $Na_2S_2O_3$ if Cl_2 present	14/28 Days**
Haloacetic Acids	552.1/552.2			40-mL vial, amber	3 X 40 mL	NF,C1, Cool, ≤6 °C	14/7 Days
Diesel Range Organics			8015B/8015C	G FP-lined amber	2 X 1000 mL	Cool, ≤6	7/40 Days
Diesel Range Organics			OK DEQ DRO	G FP-lined amber	1 X 1000 mL	Cool, ≤6, 1:1 HCL	7/40 Days
Extractable Petroleum Hydrocarbons			OA2	G FP-lined amber	1 X 500 mL	Cool, ≤6	7/40 Days
Explosives			8330	G FP-lined amber	2 X 1000 mL	Cool, ≤6	7/40 Days
2, 3, 7, 8-TCDD	1613B		8280	G FP-lined amber	2 X 1000 mL	none required	90/40 Days
Methane, Ethane, & Ethene			3810M	20-mL vial	3 X 40 mL Vials	pH<2 HCl, Cool, ≤6 °C	14 Days

ATTACHMENT IX (CONTINUED)

METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

PASI – KANSAS

Parameter	EPA Method	Container	Volume Needed	Preservative	Max Hold Time
Metals	6010B/C or 6020	G	4 oz Jar	None required	6 months
Mercury	7471A	G	4 oz Jar	Cool, ≤6 °C	28 days
Chromium, Hexavalent	7196A	G	4 oz Jar	Cool, ≤6 °C	30/4 Days
Aromatic and Halogenated Volatiles	5035A/8021B	5035 vial kit	1 kit or 4 oz Jar	See Note 1,3	14 days
Volatiles	5035A/8260B	5035 vial kit	1 kit or 4 oz Jar	See Note 1,3	14 days
Volatiles, GRO, and Oxygenates for Missouri RBCA	5035A/8260B	5035 vial kit	1 kit	See Note 2	14 days
Volatile Petroleum Hydrocarbons	OA1	G	4 oz Jar	Cool, ≤6 °C	14 days
Gasoline Range Organics	5035A/8015B	5035 vial kit	1 kit or 4 oz Jar	See Note 1,3	14 days
Base/Neutrals and Acids	8270C	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Organochlorine Pesticides and PCBs	8081A/8082	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Organophosphorous Pesticides	8141A	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Polynuclear Aromatic Hydrocarbons	8310	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Chlorinated Herbicides	8151	G, amber	4 oz Jar	Cool, ≤6 °C	14/40 Days
Diesel Range Organics	8015B/8015C, OK DRO	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Extractable Hydrocarbons	OA2	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
Diesel Range Organics	Oklahoma DEQ DRO	G	4 oz Jar	Cool, ≤6 °C	7/40 Days
Explosives	8330	G	4 oz Jar	Cool, ≤6 °C	14/40 Days
2, 3, 7, 8-TCDD	1613B/8280	G	4 oz Jar	None required	90/40 Days

Note 1: 5035 and 5035A Kit 2 vials (5 mL) water, preserved by freezing ≤-10 °C
2 vials aqueous sodium bisulfate (NaHSO4), Cool, ≤6 °C
1 vial (5 mL) methanol, Cool, ≤6 °C
1 bulk container for percent moisture determination

Note 2: 5035A MO RBCA Kit 2 vials (5 mL) water, preserved by freezing ≤-10 °C
2 vials aqueous trisodium phosphate (TSP), Cool, ≤6 °C
1 vial (5 mL) methanol, Cool, ≤6 °C
1 bulk container for percent moisture determination

Note 3: 5035 Preservation Volatile soil preservation will be conducted in accordance with state specific mandated regulations or method requirements unless otherwise specified contractually or by request.

Note: Contact your Pace Analytical Project Manager for bottle order assistance.

ATTACHMENT IX (CONTINUED)

METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

PASI – KANSAS

Parameter	EPA Method	Container	Maximum Holding Time
Permanent Gases	3C	Summa Canister	14 Days
Permanent Gases	3C	Tedlar Bag	48 Hours
Methane, Ethane, Ethene	3C-M	Summa Canister	14 Days
Methane, Ethane, Ethene	3C-M	Tedlar Bag	48 Hours
Non-Methane Organics	25C	Summa Canister	14 Days
Non-Methane Organics	25C	Tedlar Bag	48 Hours
BTEX Total Hydrocarbons	TO-3	Summa Canister	14 Days
BTEX Total Hydrocarbons	TO-3	Tedlar Bag	48 Hours
Organochlorine Pesticides & PCBs	TO-4	PUF	7/40 Days
Dioxins & Furans	TO-9	PUF	30/45 Days
Polynuclear Aromatic Hydrocarbons	TO-13	PUF	7/40 Days
Volatiles	TO-14	Summa Canister	14 Days
Volatiles	TO-14	Tedlar Bag	48 Hours
Volatiles	TO-15	Summa Canister	14 Days
Ozone Precursors	TO-15	Summa Canister	14 Days
Particulates	PM10	Filters	6 Months
Metals	10-3.5	Filters	6 Months
Stationary Source Particulates	5	Filter/Solutions	6 Months
Lead Emissions	12	Filter/Solutions	6 Months
Stationary Source Dioxins & Furans	23	XAD Trap	30/45 Days
Stationary Source Metals	29	Filters	6 Months, 28 Days for Ha
Stationary Source Mercury	101	Filters	6 Months, 28 Days for Hq
Stationary Source PM10	201A	Filters	6 Months
Condensable Particulate Emissions	202	Solutions	6 Months
Hydrogen Halide & Halogen Emissions	26	Solutions	6 Months

Note: Contact your Pace Analytical Project Manager for bottle order assistance.

ATTACHMENT IX (CONTINUED)

METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

PASI – KANSAS

Parameter	Method			Container	Volume Needed	Preservative	Max Hold Time
	EPA Water	Standard Methods	EPA SW-846				
Gross Alpha and Gross Beta	900.0		9310	P, G	1000 mL	pH<2 HNO_3	180 days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6			P, G	1000 mL	pH<2 HNO_3	48 Hrs
Gamma Emitting Radionuclides	901.1			P, G	1000 mL	pH<2 HNO_3	180 days
Alpha Emitting Radium Isotopes	903.0		9315	P, G	1000 mL	pH<2 HNO_3	180 days
Radium-226 Radon Emanation Technique	903.1			P, G	1000 mL	pH<2 HNO_3	180 days
Radium-228	904.0		9320	P, G	1000 mL	pH<2 HNO_3	180 days
Radioactive Strontium	905.0			P, G	1000 mL	pH<2 HNO_3	180 days
Tritium	906.0			G	1000 mL	pl-l<2 HNO_3	180 days
Uranium Radiochemical Method	908.0	D5174-97		P, G	1000 mL	pH<2 HNO_3	180 days

Note: Contact your Pace Analytical Project Manager for bottle order assistance.

Pace Analytical Services, Inc.
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STANDARD OPERATING PROCEDURE

Analysis of Whole Air Samples for Volatile Organic Compounds by GC/MS

Reference Method: EPA Compendium Method TO-15/ TO-14 (ppbv)

SOP NUMBER:	S-MN-A-013-Rev.10
EFFECTIVE DATE:	Upon Final Signature
SUPERSEDES:	S-MN-A-013-Rev.09

APPROVALS

Laboratory General Manager — Date: 27JUL2011

Laboratory Quality Manager — Date: 27 Jul 2011

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL. SOP IS VALID FOR ONE YEAR FROM DATE OF LAST SIGNATURE.

Signature	Title	Date

This is **COPY#** **18** Distributed on **28-Dec-11** by LRM and is ____ **CONTROLLED or** **X** **UNCONTROLLED**

Table of Contents

1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to provide quality control and analytical guidance for the analysis of whole air samples and soil vapor samples contained in Summa ® passivated canisters or Silco ® lined canisters (or equivalent) using gas chromatography/mass spectrometry. This SOP is based on EPA Compendium Method TO-15.

2. SCOPE AND APPLICATION

2.1 This procedure is designed to analyze whole air samples collected in Summa ® canisters or Silco ® lined canisters (or equivalent) for some of the volatile organic compounds (VOCs), or hazardous air pollutants (HAPs), found in Title III of the Clean Air Act Amendments of 1990. This SOP is related to only those VOCs that have been found to be stable when collected in Summa ® polished stainless steel canisters or Silco ® lined canisters (or equivalent). VOCs are defined as organic compounds having a vapor pressure greater than 10^{-1} Torr. Attachment I lists target VOCs applicable to this method.

2.2 This SOP is based on the EPA Compendium Method TO15 which can also be applied to TO14. As such, this SOP serves to cover both analyses. See EPA Compendium Method TO15 Section 3 and Attachment V for compound list.

3. SUMMARY OF METHOD

3.1 Samples are received in Summa ® canisters or Silco ® lined canisters (or equivalent). The gauge pressure upon arrival is measured and recorded. The canister is then pressurized to 5 psi gauge pressure using an inert gas. The canister is connected to an autosampler tree, which concentrates the sample prior to injection into a GC/MS. The data is then analyzed for the desired volatile organic compounds.

3.2 This method addresses an extensive set of VOCs by incorporating a multisorbent, dry purge technique for water management.

3.3 An aliquot of the whole air sample is concentrated prior to gas chromatographic (GC) separation and mass spectrometry (MS) full scan detection. Samples expected to contain VOCs in a range of 0.1 parts per billion by volume (ppbv) to 500 ppbv can be analyzed by this technique.

4. INTERFERENCES

4.1 Carrier gas potentially contains small amounts of contaminants and is filtered prior to use in instrumentation. Other interferences are sample specific and are dealt with as they occur.

4.2 Interferences in samples can result from contamination of the canisters. To minimize this problem, processes must be implemented to ensure that the canisters are contamination free. See SOP S-MN-A-004 - Procedure for Cleaning, Certification, Leak Checking, and Preparation for Shipment of SUMMA Passivated Canisters, or equivalent replacement.

4.3 Contamination of analytical equipment can also occur when samples containing high concentrations of VOCs are analyzed. The resulting “carryover” contamination varies from system to system. The analyst needs to use best judgment when evaluating sample data following samples with large detection levels.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as reasonably achievable. Cautions are included for known extremely hazardous materials

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) must be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advised.

5.3 A reference file of MSDS is maintained by the lab and available to all personnel to review at any time needed.

6. DEFINITIONS

6.1 Absolute canister pressure = $P_g + P_a$, where P_g = gauge pressure in the canister (kPa, psig) and P_a= barometric pressure.

6.2 Absolute pressure - Pressure measured with reference to absolute zero as opposed to atmospheric pressure, usually expressed as kPa, mm Hg or psia.

6.3 Cryogen - A refrigerant used to obtain very low temperatures for sample concentration. A typical cryogen is liquid nitrogen (bp - 195.8°C).

6.4 Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

6.5 Gauge pressure - Pressure measured above ambient atmospheric pressure as opposed to absolute pressure. Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

6.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

6.7 MS-SIM - The GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly.

6.8 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

6.9 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

6.10 Additional definitions are also found in the glossary of the current Pace Analytical Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 The holding time indicated below is the maximum allowable time from collection to analysis per the analytical method.

7.2 Samples are collected into evacuated Summa ® canisters and Silco ® canisters (or equivalent). The canisters are then shipped back to Pace Analytical Services, Inc. for analysis.

7.3 Samples collected in Summa ® canisters, Silco ® canisters (or equivalent) must be analyzed within 28 days from collection.

7.4 Samples collected in Minnesota are to be collected in canisters and must be analyzed within 14 days of collection per the MPCA.

7.5 If samples have been collected in Tedlar bags, the samples need to be transferred to a Summa Canister within 48 hours to maintain a 28 day holding time. The holding time is potentially extended to 72 hours per client specific QAPPS. Collection in a Tedlar bag results in higher reporting limits. See Attachments VIII-X for instructions and documentation.

7.5.1 Ohio VAP samples must be transferred to a Summa Canister within 48 hours from collection to extend the holding time to 28 days.

8. EQUIPMENT AND SUPPLIES

8.1 Standard preparation materials for static dilution technique:

8.1.1 0.010, 0.025, 0.05, 0.1, 0.25, 0.5, 1, 5, and 10 mL gas tight syringes

8.1.2 Neat liquid standards of at least 95%

8.1.3 2L glass static dilution flask equipped with a Mini-inert cap

8.1.4 Oven capable of maintaining a temperature of 65^{o}C

8.1.5 Summa ® passivated canisters or Silco ® lined canisters (or equivalent), six liter or fifteen liter capacity

8.1.6 High accuracy dual pressure/vacuum gauge

8.1.7 Nitrogen

8.1.8 Organic free water

8.2 Analytical Instrumentation

8.2.1 Agilent Technologies 6890N gas chromatograph equipped with a split/splitless injection port and electronic pressure control (EPC) or equivalent.

8.1.2.1 Gas chromatograph.Suggested Operating Parameters:

1) Initial temp: 40°C for 2.0 min.
2) Ramp A: 8°C/min to 150°C
3) Ramp B: 15°C/min to 200°C
4) Hold 2 min
5) EPC Pressure: 9 psi
6) Temp 250°C
7) Split Flow 20mL/min

8.1.2.2 Injection port parameters.

1) EPC pressure: 9 psi
2) Temperature: 250°C
3) Purge valve:Initial value On, Off time 0.0 min.
4) Split flow: 20 mL/min.

8.2.2 J & W Scientific DB-5 60m x 0.32mm capillary column or DB-624 60m x 0.32mm with a 1.8 µm film thickness or equivalent.

8.2.3 High purity grade high-pressure helium cylinder for column carrier gas equipped with a dual stage pressure regulator.

8.2.4 Hewlett Packard 5973 Mass Selective Detector, or equivalent with Chemstation operating software and WinTarget data processing software or equivalent.

8.2.4.1 Suggested Mass spectrometer parameters:

1) Electron volts: 70 nominal
2) Scan range: 29 to 300 amu

3) Scan time: At least 2 scans/peak, not to exceed 1 sec/scan
4) Interface temp: 250°C
5) The GC/MS system must be set up to meet manufacturer's specification. The mass calibration and resolution of the GC/MS are verified by the analysis of the tune standard, p-bromofluorobenzene (BFB). For more information refer to the Chemsystem User's Guide and the GC/MS User's Guide.

8.2.5 Entech 7100A pre-concentrator with 7016 canister manifold autosampler.

8.2.6 Entech Pre-Concentrator suggested settings:

8.2.6.1

During Concentration	Temperature (°C)
Module No. 1, Glass Bead Cryotrap	-150
Module No. 2, Sorbent Packed Cryotrap	-20
Focusing Trap	-160

8.2.6.2

Desorb/Transfer/Inject	Preheat (ºC)	Final Temp(ºC)
Module No. 1, Glass Bead Cryotrap	10	10
Module No. 2, Sorbent Packed Cryotrap	50	180
Focusing Trap	N/A	N/A

8.2.6.3

Media Concentrated/Transferred	Volume (cc)	Flow Rate (sccm)
Internal Standard & Surrogate	50	200
Sample	25 to 500	250
Sweep/Dry Purge	75	100
Transfer to Packed Column	40	10

8.2.6.4 Sample Transfer

Line Conditioning Sample Flush Before Trapping	20 sec
Carrier Flush Before Trapping	2 to 4 min.
Sample Transfer to Focusing Trap	2 to 4 min.
Sample Injection	2 to 5 min.

8.2.6.5

System Bakeout	Temperature (ºC)	Time (min.)
Module No. 1	150	10
Module No. 2	190	10

8.2.6.6

Regulated Zones	Temperature (ºC)
8-Port Valve	100
GC Transfer Line	110
Manifold Transfer Line	100
16-Position Select Valve	100
Sample Container	Ambient

8.3 Data System:

8.3.1 Chemstation – data acquisition software

8.3.2 Target – data processing software

8.3.3 Horizon – data reporting software

9 REAGENTS AND STANDARDS

9.1 Calibration Standard: The calibration standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, INC. This is a custom mix that includes all compounds of interest at 1ppmv.

9.1.1 2 PPBV: Using the 1000cc gas tight syringe, pull 90cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 2 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.1.2 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50 µL H_2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.1.3 Suggested Ical levels and preparation:

Ical Level	Concentration	Calibration Standard Used	Amt of Cal Standard Used
Level 1	0.10 ppbv	2.0 ppbv std	25 cc
Level 2	0.20 ppbv	2.0 ppbv std	50 cc
Level 3	0.50 ppbv	2.00 ppbv std	125 cc
Level 4	1.00 ppbv	2.00 ppbv std	250 cc
Level 5	10.00 ppbv	20.00 ppbv std	250 cc
Level 6	20.00 ppbv	20.00 ppbv std	500 cc
Level 7	30.00 ppbv	20.00 ppbv std	750 cc

9.2 Initial Calibration Verification (second source standard): The laboratory control standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas Solutions, or equivalent). This is a custom mix that includes all compounds of interest at 1ppmv. See Attachment I for the list of compounds present in the standard.

9.2.1 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.2.2 The ICV is prepared by taking 250cc from the 10 ppbv standard (see above table) and delivering it to a 15 L canister that has been humidified according to 9.2.1.

9.3 Internal Standard/ Surrogate/ BFB Standard 200ppbv: The internal, surrogate, and BFB standards are purchased as neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent.

9.3.1 To prepare a neat/cocktail standard: Using Entech Static Dilution software, enter barometric pressure, room temperature, flask temperature, flask volume, canister pressure, canister volume, flask concentration and desired final concentration (100ppbv). The software calculates approximate transfer volume 1 (vial to flask), then transfer volume 2 (flask to canister)*. **Note**: Standard canister (6 L or 15 L) must be cleaned, evacuated, and humidified with 50 µL H_2O before being used.

Example: Barometric Pressure: 29.92
Room Temperature : 24 °C
Flask Temperature : 65 °C
Flask Volume : 2000 mL
Canister Pressure : 30 psig
Canister Volume : 15,000 mL (15 L)
Flask Concentration : 520.015 PPM

*The software calculates transfer volume to the $1/10000^{th}$. The volumetric syringes are calibrated to $1/10^{th}$ of a decimal place. Therefore, the analyst must adjust the volume to a measurable amount prior to standard preparation.

9.3.2 Next, pressurize the 15 L canister 30 psig with clean nitrogen. This yields a final concentration of 200 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.3.3 The tune standard, Bromofluorobenzene (BFB), must be 50ng or less on column.

9.3.3.1 The tune standard can be combined with the CCV standard so long as all criteria can be evaluated and met.

9.3.4 Internal standard compounds and surrogate standard compounds are used in the analysis.

9.3.4.1 Internal Standards: 1,4-Difluorobenzene and Chlorobenzene-d5

9.3.4.2 Surrogates: Hexane-d14, Toluene-d8, and 1,2-Dichlorobenzene-d

9.4 Standard Canister Preparation:

9.4.1 Static Dilution Technique

9.4.1.1 Summary: Standard preparation is accomplished by injecting an aliquot of liquid standard cocktail into a static dilution vessel. The static dilution vessel is held at a temperature of 60°C. The liquid standard vaporizes and is quickly vented to come to equilibrium. An aliquot is removed and injected into a canister. The canister is then pressurized with nitrogen to a pre-established final pressure.

9.4.1.2 Procedure

9.4.1.2.1 The volume of a clean 2L round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density of 1 g/mL for water, the weight of the flask in grams when filled with water is taken as the volume of the flask in milliliters.

9.4.1.2.2 The dried flask is flushed with nitrogen. After a few minutes, the glass neck is immediately capped with a Mininert septum cap.

9.4.1.2.3 The flask is placed in a 60°C oven and allowed to equilibrate at that temperature for about 30 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 60°C.

9.4.1.2.4 The contents are allowed to equilibrate in the oven for at least 30 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots.

9.4.1.2.5 Sample aliquots are then to be taken from the static dilution flask for introduction into a clean, evacuated canister. The canister is then filled to a final predetermined pressure. An aliquot or aliquots totaling greater than 1 percent of the flask volume are to be avoided.

9.4.1.2.6 The concentration of each component in the flask is calculated using Equation 1:

Equation 1

$$\text{Concentration}(\text{mg/L}) = \frac{(V_i)(d)}{V_f}$$

where: V_i =Volume of liquid neat standard injected into the flask in mL;
d=Density of the liquid neat standard in mg/mL;
V_f =Volume of the flask in liters.

Caution: In the preparation of standards by this technique, make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask..

9.4.1.2.7 The concentration in ppbv of each component in the flask is determined using Equations 2 and 3 as follows:

9.4.1.2.7.1 First determine the volume of the compound as a gas using Equation 2:

Equation 2

$$V = \frac{nRT}{P} \quad \text{where,} \qquad n = \frac{(V_i)(d)}{M}$$

where, V=Volume of injected compound at STP in liters;
n=Moles;
R=Gas constant (0.08206 L-atm/mole ^{o}K);
T=Ambient temperature in ^{o}K;
P=Ambient pressure in atm;
V_i=Volume of liquid neat standard injected into the flask in mL;
d=Density of the neat standard in g/mL;
M=Molecular weight of the compound in g/mole.

9.4.1.2.7.2 Now calculate the concentration in the flask in ppbv using Equation 3:

Equation 3

$$\text{ppbv} = \frac{V}{V_f}\left(10^9\right)$$

where: V=Gas volume of compound as determined in Eq. 8 in liters;
V_f=Volume of static dilution flask in liters.

9.4.1.2.8 The concentration in ppbv of each compound in the canister can be determined using Equation 4:

Equation 4

$$\text{ppbv} = \frac{(V_i)(C_x)}{V_c}$$

where: V_i=Volume removed from static dilution flask and injected into the canister in liters;
C_x=Concentration of compound x in the static dilution flask in ppbv;
V_c=Final canister volume in liters.

9.4.1.2.9 Entech Standards Preparation has a database of compounds and their properties. The program does the necessary conversions of units and calculations (equations 1-4) to yield the amounts of neat standard put in the standard cocktail, the amount of cocktail spiked into the 2L flask, and the aliquot taken from the 2L flask to the final canister. This program can be used to make any gas standard from neat liquid standards.

9.4.1.2.10 See Attachment VI for a single sheet summary of Air Standard Preparation.

10 CALIBRATION

10.1 Instrument Tune

10.1.1 It is necessary to establish that the GC/MS system can produce tuning and standard mass spectral criteria prior to analyzing standards or samples. The GC/MS is set up according to the manufacturer's specifications. The MS source and mass filter are adjusted by monitoring the mass spectra of PFTBA.

10.1.2 Before any standard, blank, or sample analysis can occur using the GC/MS system, it must be demonstrated that the GC/MS is capable of producing compliant spectra when p-bromofluorobenzene (BFB) is analyzed. Attachment II lists the required spectral criteria.

10.1.3 Instrument Tune Procedure

10.1.3.1 Prepare a standard solution of BFB at a concentration that allows the collection of 50ng or less under the optimized concentration parameters (see Section 9.3)

10.1.3.2 The BFB is introduced into the system through microscale purge and trap.

10.1.3.3 Evaluate the BFB spectrum.

10.1.3.3.1 The spectrum of BFB must be acquired by averaging three scans; the apex and the scans that immediately proceed and follow the apex.

10.1.3.3.2 Background subtraction is accomplished using a single scan taken before the BFB peak.

10.1.3.3.3 The instrument performance check must be analyzed initially and once every 24-hour period. The tune period begins at the time of injection of the BFB.

10.1.3.4 If the BFB spectrum meets the criteria listed in Attachment II, proceed with standard and sample analysis.

10.1.3.5 If the BFB spectrum fails to meet the criteria listed in Attachment II, the MS must be retuned. Repeated failures potentially indicate the need for MS maintenance such as cleaning the ion source.

10.2 Initial Calibration

10.2.1 All standards, blanks, spikes, and samples must be analyzed using the same conditions. A calibration curve must consist of a minimum of 5 standards (6 for quadratic) and spans the expected monitoring range established for each compound of interest to determine instrument response and linearity. The lowest level of the curve must be at or below the reporting limit for each analyte. A typical calibration curve can cover a range from 0.1 to 20 ppbv. Section 9.1.3 contains standard preparation information.

10.2.1.1 Initial calibrations are not meant to be a replacement of necessary instrument maintenance.

10.2.1.2 Calibration curve fits are possible indicators of instrument performance or deterioration. Analytes that traditionally are average or linear responders that suddenly display quadratic curve fits could be a sign of a system that is deteriorating. Quadratic cannot be used to extend the calibration range for compounds that normally exhibit a linear response, perform necessary maintenance to return the system to good working order. This is not to eliminate the use of quadratic curve fits, some analytes always present a quadratic response and that is acceptable.

10.2.2 Calibration is performed using the internal standard technique. See Attachment III for internal standard groups. The data is evaluated using WinTarget. See 10.2.7 for acceptance criteria.

10.2.3 Initial Calibration Verification (Section 9.2): a second source standard must be analyzed following an initial calibration curve which contains all the analytes of interest. The spike level of the ICV must be near the midpoint level of the calibration curve. The ICV is considered acceptable if the recoveries of the analytes fall within 60-140%.

10.2.3.1 If ICV fails criteria, the analyst must consult with his or her supervisor or manager before moving forward. Possible corrective actions include:

- •Analyze modified list for compounds that were ICV compliant.
- •Stop instrument and verify standard
- •Remake ICV and reanalyze.
- •Perform instrument maintenance.
- •Recalibrate and reanalyze the ICV.
- •Technical justification provided for analysis to continue

10.2.4 The ENTECH 7000 Concentrator automatically adds a specified concentration of internal standards and surrogates (Section 9.3) to each analysis during trapping.

10.2.5 Using the Target data processing software, evaluate the calibration data.

10.2.6 Calculations

10.2.6.1 Relative Response Factor (RRF): Tabulate the area response of the primary ion (Attachment III) for each compound and the associated internal standard. Use the internal standard, which has a retention time nearest to the compound of interest. Calculate the relative response factors (RRF) for each compound using Equation 5:

Equation 5

$$\text{Relative Response Factor (RRF)} = \frac{(A_x)(C_i)}{(A_i)(C_x)}$$

where, A_x=Area of the primary ion for compound x to be measured;
A_i=Area of the primary ion for the internal standard associated with compound x;
C_i=Concentration of the internal standard in ppbv;
C_x =Concentration of compound x to be measured in ppbv.

10.2.6.2 Mean Relative Response Factor. Calculate the mean RRF for each compound using the RRF from the five (or six, where n=6)-point calibration using Equation 6:

Equation 6

$$\overline{R_f} = \frac{\sum_{n=5} R_f}{n}$$

where, $\overline{R_f}$ =Average relative response factor;
R_f=Relative response factor from calibration curve;
n=Number of data points.

10.2.6.3 Standard Deviation ($\sigma_{(n-1)}$).

Equation 7

$$\sigma_{(n-1)} = \sqrt{\sum_{i-1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

10.2.6.4 %Relative Standard Deviation (%RSD). Using the average RRF from Equation 6 and the standard deviation from Equation 7, calculate the %RSD using Equation 8:

Equation 8

$$\%RSD = \frac{S_{(n-1)}}{\overline{R_f}} x100$$

10.2.6.5 Mean area response for Internal Standard:

Equation 9

$$\bar{y} = \sum_{i=1}^{n} \frac{y_i}{n}$$

where $\bar{y}$ = mean area response
y = Area response for the internal standard for each initial calibration standard

10.2.6.6 If a linear regression is used, the regression produces the slope and intercept terms for a linear equation according to Equation 10:

Equation 10

$$y = ax + b$$

where: y = instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = the intercept, do not include the origin (0) as a calibration point

10.2.6.7 To calculate the sample concentration by the internal standard method using the linear regression equation, use Equation 11:

Equation 11:

$$C_s=[(A_sC_{is}/A_{is})-b]/a$$

where: As = Area of the peak for the target analyte in the sample
Ais = Area of the peak of the internal standard
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (also called the coefficient of Cs)
b = The intercept

10.2.6.8 To calculate the coefficient of determination (or r^2) for a quadratic curve fit, use Equation 12:

Equation 12:

$$COD = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - Y_i)^2}{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2}$$

where: y_{obs} = Observed response for each concentration from each initial calibration standard
y = Mean observed response from the initial calibration (See equation 6)
Y_i = Calculated response at each concentration from the initial calibration (See Equation 5)
n = Total number of calibration points in the equation, 6 points for quadratic
p = Number of adjustable parameters in the polynomial equation

10.2.6.9 Calculate the sample concentration by the internal standard method using the quadratic regression by comparing peak heights to the calibration curve.

Regression equation (quadratic):

$$y = ax^2 + bx + c$$

10.2.7 Technical Acceptance Criteria.

10.2.7.1 The %RSD for all calibrated target compounds must be ±30% with no more than 2 compounds at ±40%. Alternately, linear regression is used with an r^2 value of 0.995 or greater. A quadratic curve is utilized if the r^2(equals COD in Equation 12) value is 0.990 or greater and six calibration points are included in the curve. Curves must not be forced through zero. *For Ohio VAP: quadratic curve fit is only to be used for analytes that have historically exhibited nonlinear response.*

10.2.7.2 The area response for each internal standard in each calibration level must be within 40% of the mean area response over the calibration range. The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve.

10.2.7.3 Per MDH Rules, the reporting limit standard must be evaluated to determine if the curve fit is presenting bias. The level corresponding to the reporting limit must be quantitate back after processing the curve and be ± 40% of the expected true value. If the criteria is not met, the instrument must be recalibrated or the reporting limit adjusted to the next level that meets the criteria

10.2.8 Corrective Action

10.2.8.1 If the technical acceptance criteria fail for the initial calibration curve, inspect the system for any possible leaks. A high baseline and reduced response potentially indicates a leak.

10.2.8.2 Examine the response factors of each calibration level. If the response factors of all the compounds for one level appear to be significantly different, analyze that same level calibration standard again.

10.2.8.3 If the same results occur after reanalysis, a new standard canister must be made and analyzed.

10.2.8.4 If a leak or other system problem cannot be found, try to clean the ion source or perform column maintenance.

10.2.8.5 No samples can be analyzed until a compliant initial calibration curve has been established and verified against a second source standard or technical justification given for the analysis to continue.

10.2.8.6 Recalibration must be performed if any major change has been made to the GC/MS system such as replacing the GC column, cleaning the MS source or repair.

10.3 Continuing Calibration Verification

10.3.1 The initial calibration curve for each compound of interest must be checked and verified before sample analysis can occur each day. This is accomplished by analyzing continuing calibration verification (CCV) standard at 10 ppbv. (See Section 9.1)

10.3.2 The CCV is analyzed after a compliant tune, as described in 10.1, once every 24-hour period during sample analysis.

10.3.3 Calculations

10.3.3.1 Calculate the RRF for each target compound from the continuing calibration standard using Equation 5.

10.3.3.2 Percent Difference (%D). The % D in the RRF of the daily RRF of an individual compound compared to the mean RRF for that compound in the most recent calibration curve is determined as follows:

Equation 13

$$\%D = \frac{|R_i - R_c|}{R_i}(100)$$

where, R_i=The average RRF from the initial calibration curve for compound *x*;
R_c=RRF for compound *x* from the daily calibration standard.

10.3.4 Technical Acceptance Criteria

10.3.4.1 The %D for each target compound in the daily calibration standard must be less than or equal to 30 percent.

10.3.4.2 The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve

10.3.4.3 For TO14 only analysis, the CCV criteria must be ±10%.

10.3.5 Corrective Action

10.3.5.1 If the CCV does not meet criteria, the system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second CCV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance.

10.3.5.1.1 Check pressure on the standard canister.

10.3.5.1.2 Check system for leaks.

10.3.5.1.3 Check to see that standards were made correctly.

10.3.5.2 If corrective action attempts fail or two consecutive CCV do not meet criteria, then a new calibration curve must be analyzed.

10.3.5.3 Samples are not to be analyzed until CCV criteria has been met or technical justification given for the analysis to continue.

11 PROCEDURE

11.1 Analytical Sequence

The following is the GC/MS analytical sequence for samples each 24-hour period:

11.1.1 Instrument tune using (BFB); see Section 10.1

11.1.2 Initial Multi-Point Calibration or CCV; see Section 10.2 or 10.3

11.1.3 ICV or Laboratory Control Sample (LCS); see Section 10.2.3 and 11.3

11.1.4 Laboratory Method Blank

11.1.5 20 field samples

11.1.6 Sample duplicate, minimum of one in 20 samples

11.1.7 Any necessary dilutions from previously analyzed samples (see the dilution preparation section of Attachment V).

11.1.8 In the event that time remains in the 24 hour tune period, an additional blank and LCS must be analyzed in order to analyze additional reportable samples.

11.2 Blank Analysis

11.2.1 A clean canister filled with humidified nitrogen is analyzed on the GC/MS system to demonstrate that the system is free of interferences.

11.2.2 The Method Blank is prepared in the same manner as any standard or sample and analyzed in the same manner.

11.2.3 A Method Blank is analyzed once every 24-hour period or every 20 samples, whichever comes first.

11.2.4 The Method Blank is analyzed after the daily calibration standard.

11.2.5 An instrument blank analysis is allowed after any sample that has known VOCs present that exceed the upper calibration limit of the method to demonstrate that the system is free of possible carryover effects. When possible, historical data can be used to determine if there are high levels of contaminants present, possibly causing carry over in the system.

11.2.6 See Equation 16 for the calculation on how to determine the concentration present.

11.2.7 See Section 12 for technical acceptance criteria and corrective actions.

11.3 Laboratory Control Sample (LCS)

11.3.1 The laboratory control standard is prepared from the same standard as the calibration standard (20ppbv) as outlined in section 9.1.2. The LCS is to be analyzed at a minimum of 1 in every 20 samples.

11.3.2 Calculations

11.3.2.1 Field sample calculations in Section 11.7 also apply to the LCS.

11.3.2.2 Calculate the percent recovery of the LCS using Equation 14:

Equation 14

$$\text{Percent Recovery} = \frac{C_q}{C_a}(100)$$

where: C_q=Quantitated concentration of compound *x* in ppbv;
C_a=Actual concentration of compound *x* in ppbv.

11.3.3 See section 12 for technical acceptance criteria and corrective actions.

11.4 Sample Analysis

11.4.1 Upon receipt, the canister pressure of each sample is measured and recorded on the canister sample tag.

11.4.1.1 If the canister pressure is less than 5 psig, the canister pressure must be increased before analysis can occur.

11.4.1.1.1 Add clean nitrogen or helium gas to the sample canister. For a six liter canister, 5 psig is the desired final pressure. A one liter canister requires a final pressure of 10 psig for adequate sample volume for analysis.

11.4.1.1.2 Record the final canister pressure on the canister sample tag noting which gas was added. Also, note the information in the final analytical results report.

11.4.1.1.3 Calculate the resultant dilution factor using Equation 15:

Equation 15

$$\text{Dilution Factor} = \frac{P_f}{P_i}$$

where: P_f=Final canister pressure in psig;

P_i=Initial canister pressure in psig.

See attachment V for the application of dilution factors for filling canisters.

11.4.1.1.4 This dilution factor is applied to Equation 16.

11.4.2 Once the GC/MS system is demonstrated to be in control, an aliquot of the air sample is removed from the canister and pre-concentrated using the Entech 7100A pre-concentrator and 7016 autosampler manifold.

11.4.3 Analyze the samples under the same operating conditions as the instrument calibration and quality control samples.

11.4.4 Analyze a duplicate sample for every 20 samples analyzed.

11.4.5 If time remains in the 24-hour tune period in which an initial calibration was performed, it is possible to continue to analyze samples without the analysis of a daily calibration standard.

11.4.6 If the tune period has expired, an instrument performance check standard and daily calibration standard must be analyzed before samples can be analyzed.

11.4.7 If time remains in the tune period after a batch of no more than 20 samples and its re-runs have been analyzed, it is possible to analyze additional samples after a new LCS and method blank have been analyzed.

11.4.8 Technical Acceptance Criteria can be found in Section 11.9

11.4.9 Procedures for the determination of Air Phase Petroleum Hydrocarbons (APH) can be found on attachment XII.

11.5 Qualitative Analysis

11.5.1 The compounds listed in Attachment I are identified by an analyst competent in the interpretation of mass spectra. Sample mass spectrum is compared to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the target compound identifications: (1) elution of the sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

11.5.2 The relative retention time (RRT) of the sample component must agree within ± 0.06 RRT units of the RRT of the standard component using the continuing check standard as reference.

11.5.3 Standard and sample mass spectra are compared using reference spectra obtained on the GC/MS system being used. The mass spectra used for comparison are from the same standard as that being used for RRT comparison. Mass spectral requirements are as follows:

11.5.3.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.5.3.2 The relative intensities of ions specified above must agree within ± 20% between the standard and sample spectra.

11.5.3.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process favors false positive.

11.5.4 Non-target sample components are library searched using the latest NIST library for the purpose of tentative identification. These components are referred to as TICs - Tentatively Identified Compounds) and are noted as such in any final report with a qualifier of "J" unless the client specifies differently. The "J" qualifier indicates an estimated value. Guidelines for identification are as follows:

11.5.4.1 Characteristic ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample.

11.5.4.2 The relative intensities of the major ions must agree within ± 20%.

11.5.4.3 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for background contamination or presence of co-eluting peaks.

11.5.4.4 If in the technical judgment of the analyst, no valid identification can be made, the compound is to be reported as an unknown with possible classification, such as hydrocarbon.

11.5.4.5 TIC searches are reported only upon client request.

11.6 Identified target analytes are quantitated using the internal standard method using the EICP area of the characteristic ions of analytes listed in Attachment III. This ion is referred to as the quantitation ion.

11.7 The RRF from the continuing calibration standard analysis is used to quantitate samples and blanks. Calculate the concentration of the sample component using Equation 16:

Equation 16

$$C_x = \frac{(A_x)(C_i)(D_f)}{(A_i)(R_x)}$$

where: C_x=Concentration of compound x in ppbv;
A_x=EICP area of the quantitation ion for compound x;
C_i= Concentration of the internal standard associated with compound x in ppbv;
D_f=Dilution factor from Equation 12 (if no dilution was performed, D_f equals 1.)
A_i=EICP area of the quantitation ion for the internal standard associated with compound x;
R_f=Average RRF for compound x from the most recent calibration curve.

11.7.1 Additional curve fit equations are in 10.2.6.

11.8 The internal standard method of quantitation is also used to determine an estimated concentration for Tentatively Identified Compounds (TIC). The nearest internal standard to the TIC is used as a reference to estimate the concentration of the TIC. If the nearest internal standard exhibits interferences, the next closest internal is used. The estimated concentration is obtained using Equation 16 with the following exceptions:

A_x = Total ion chromatogram area of the TIC,
A_i = Total ion chromatogram area of the specific internal standard;
R_f = 1.0

Estimated TIC concentrations are flagged with a qualifier of "J" which indicates that the quantitated amount is an estimate.

11.9 General Technical Acceptance Criteria

11.9.1 For data to be reported without qualification, the following criteria must be met for all samples, CCVs, method blanks, and quality control samples:

11.9.1.1 The EICP area response for each internal standard must be within ±40% of the EICP area response in the most recent CCV. See Attachment III for a list of analytes and assigned internal standards.

11.9.1.2 The retention time for each of the internal standards must be ±0.33 minutes of each of the IS retention times in the most recent CCV.

11.9.1.3 Recoveries for surrogate standard compounds (where required) must fall within ±30% of the true value.

11.9.2 If the technical acceptance criteria are not met, the instrument calibration, laboratory quality control samples and/or associated samples must be reanalyzed to confirm results. See Section 10 for corrective action for calibration failures and Section 12 for all other samples (including QC).

11.9.2.1 If the surrogates don't fall within laboratory generated limits, the system must be checked to determine the cause of the failures. The sample must be reanalyzed to confirm the results unless there is definitive proof of matrix interference. The data is qualified accordingly.

12 QUALITY CONTROL

12.1 Three performance criteria are used to demonstrate method validity which are: (1) method detection limit (MDL), (2) replicate precision, and (3) accuracy - % recovery of LCS.

12.1.1 The MDL is determined following the guidelines set forth in S-ALL-Q-004, or equivalent replacement.

12.1.2 Replicate precision is based upon the relative difference between replicate measurements of the same sample expressed as a percentage,

[(Measurement #1 - Measurement #2) x 100%]/Average of 2 measurements.

12.2 A Method Blank analyzed once every 24-hour period or every 20 samples, whichever comes first.

12.2.1 Technical Acceptance Criteria

12.2.1.1 The blank must not contain any target analyte at a concentration greater than its reporting limit and must not contain additional compounds with elution characteristics and mass spectral features that interfere with identification and measurement of a method analyte.

12.2.1.2 The internal standard must be within ± 40% of the mean area response of the IS in the most recent calibration. The retention time of each of the internal standards must be within ± 0.33 minutes between the method blank and the most recent calibration standard.

12.2.2 Corrective Action

12.2.2.1 If a Method Blank fails acceptance criteria, the source of the contamination must be identified and eliminated.

12.2.2.2 If a source of contamination is corrected, another Method Blank must be prepared and analyzed to verify that the problem has been resolved.

12.2.2.3 However, if the contaminant cannot be eliminated and samples are analyzed, samples containing the same artifact as that found in a blank must be flagged accordingly.

NOTE: For Ohio VAP samples, if the detection is above the reporting limit and corrective actions do not result in acceptable data, the samples must be re-analyzed undiluted. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate

12.3 A LCS must be analyzed once every 24-hour period or every 20 samples, whichever is more frequent.

12.3.1 Technical Acceptance Criteria

12.3.1.1 The percent recovery for each analyte in the LCS must be within the internally generated QC limits.

12.3.2 Corrective Action

12.3.2.1 If a LCS fails to meet the recovery limit criteria, inspect the system for the possibility of a poor sampling.

12.3.2.2 If the LCS fails and no error in sampling was found, the system must be recalibrated. The preparation of new calibration standards is also considered.

12.3.2.3 If the samples cannot be reanalyzed, qualify the data accordingly.

12.3.2.4 For Ohio VAP samples, if the outlier is an analyte of interest and corrective actions do not result in acceptable data, the samples must be re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate

12.4 Duplicate sample analysis is performed once per 20 samples. See Attachment VII for exception to this criteria.

12.4.1 The RPD between the sample and the sample duplicate must be < 25% and can be calculated using Equation 17:

Equation 17

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

Where: RPD = Relative Percent Difference

A = Sample Value
B = Duplicate Value

12.4.2 If the RPD fails to meet criteria, the instrument must be evaluated to determine if there was an error with the analysis. If there is not evidence of malfunction, the samples must be reanalyzed to confirm results. If the data confirms, report the original data and qualify accordingly.

12.5 Internal Standards

12.5.1 Technical Acceptance Criteria – See 11.9.1.

12.5.2 Corrective Action

12.5.2.1 Examine the instrument for possible errors or malfunctions and correct any that are discovered. Re-analyze the samples and QC and report the acceptable data

12.5.2.2 If there is no evidence of error or malfunction, re-analyze the affected QC and samples. If the data confirms, report the original data and qualify accordingly.

12.5.2.3 Unless a matrix interference was detected, Ohio VAP samples must be re-analyzed undiluted.

12.6 Surrogates

12.6.1 Technical Acceptance Criteria

12.6.1.1 Surrogates are not required by the TO15 method. Therefore they are only be analyzed upon client request.

Note: Ohio VAP samples must not include surrogates.

12.6.1.2 If surrogates are requested by the client, they must meet internally generated limits (±30%).

12.6.2 Corrective action

12.6.2.1 If surrogates do not meet the recovery limits, re-analysis is performed to confirm the outlier and data is qualified as necessary.

12.6.2.2 One exception to the above corrective action occurs if the sample is non-detect for the analytes of interest and the surrogate fails above the recovery limits. In this situation, the data is biased high and is unaffected by the high surrogate recoveries. The sample is qualified accordingly without re-analysis.

13 METHOD PERFORMANCE

13.1 There are several requirements that must be met to ensure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications are found in the Laboratory Quality Manual and specific Standard Operating Procedures.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file within the QA office

13.1.2 An initial demonstration of capability (IDC) must be performed per SOP ALL-Q-020. A record of the IDC is maintained in his/her file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual minimum detection limit (MDL) study following SOP S-MN-Q-269 is completed for this method and whenever there is a major change in personnel or equipment. Results are stored in the QA Office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed to demonstrate continuing competence according to SOP S-ALL-Q-258 – Proficiency Testing Program.

14 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 The Environmental Protection Agency (USEPA), state and federal law requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. For further information on waste management consult SOP S-MN-S-003, Waste Handling (or equivalent replacement).

15 REFERENCES

15.1 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO15.

15.2 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO14A

15.3 Pace Quality Assurance Manual- most current version.

15.4 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

15.5 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

16 TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA, ETC

16.1 ATTACHMENT I: Target Compound List

16.2 ATTACHMENT II: Required BFB Key Ions and Ion Abundance Criteria

16.3 ATTACHMENT III: Characteristic Ions for Target Compounds

16.4 ATTACHMENT IV: Calibration of THC as Gas

16.5 ATTACHMENT V: Canister Dilution Factors

16.6 ATTACHMENT VI: Air Laboratory Standard Preparation Procedures

16.7 ATTACHMENT VII: Procedures for Analyzing MPCA Samples

16.8 ATTACHMENT VIII: Procedure for Tedlar Bags

16.9 ATTACHMENT IX: Tedlar Sign-off Logbook

16.10 ATTACHMENT X: Tedlar Bag Transfer Log

16.11 ATTACHMENT XI: Common Logbook Abbreviations

16.12 ATTACHMENT XII: Determination of Air Phase Petroleum Hydrocarbons (APH)

16.13

17 REVISIONS

Revision Number	Reason for Change	Date

MN-A-013-rev3	Sec. 9-Remove heating band. Sec. 10 Add surrogate standard compounds standard.Sec. 11. Add second source standard to verify calibration and preparation of the surrogate standard canister. Sec. 12 Add second source verification of calibration to analytical sequence and that surrogate standard compounds will fall within internally generated limits. Attachment IV reformatted	10/31/05
MN-A-013-rev.2	Reformat to conform to corporate model. Add reference to MDL SOP: SOP ALL-P-04 to section 14.	3/28/05
S-MN-A-013-rev.04	Reformat Section 7 to new style Re-worded multiple sections to make SOP more concise Eliminated multiple sections to reduce repetition. Combined with TO14 SOP since one SOP covered both via the TO15 Compendium Method Attachment I was edited to reflect TO14 compounds and reporting limits were removed. Added attachments V and VI.	6/08/07
S-MN-A-013-Rev.05	Complete rearrangement of sections. Deleted Section 7 Responsibilities and Distribution based on SOP of SOP Preparation Moved Instrument condition to Section 8, move all standard preparation to Section 9, moved all calibration and tuning information to Section 10, moved all QC acceptance and corrective action to Section 12. Added corrective actions throughout Added attachments VIII-X for Tedlar bags Added corrective actions for internal standards and surrogates (12.5 and 12.6).	21April2008
S-MN-A-013-Rev.06	Changed the holding time for Tedlar bag from 72 to 48 hrs for Ohio VAP, except for client specific QAPPS Added that BFB can be combined with CCV analysis CCV criteria for TO-14 is 10% Section 1 edited to include soil vapor samples Clarification about differences in Ohio VAP samples vs. other clients Section 12.5; Ohio VAP samples must be reanalyzed undiluted	02Jun2009
S-MN-A-013-Rev.07	Added Benzyl Chloride to the target compound list. Changed location of MSDS to Groupwise Added reference to Method TO-14 Formatting updates Changed heading of table in 8.2.5.3 to “Sample Transfer” Remove “LCS” from 9.2.2 LCS info updated in 11.3.1 “LCS” removed from Attachment VI	03Dec2009
S-MN-A-013 Rev.08	Updated Methyl Ethyl Ketone – primary ion: 72 secondary ion: 43	31Mar2010
S-MN-A-013 Rev.09	Added Reporting limit standard in formation to Section 10 Added software to section 9 Updated 10.2.3 to allow the ICV spike level to pass if all analytes are +/-40% (no outliers) and added 10.2.3.1 Updated attachments XI and X. Added dilution info to V.	02 Aug 2010
S-MN-A-013 Rev.10	Added 11.4.9 to refer to Attachment XII Added Attachment XII	27Jul2011

S-MN-A-013 Rev.10	5.3 – Updated MSDS location 8.3 – Software added 9.1.3 – Added 7th calibration level 10.2.3.1 – Added 10.2.7.3 – 40% rule added 11.3.1. – LCS information updated 11.4 – Changed "may be" to "it is possible to" within section 13.1.3-4, 14.2 – SOP references updated 15.3-5 – References added Updated and normalized formatting. Added 7.4 – MPCA holding time of 14 days Clarified the IS criteria in the Mblank section per the method in 12.2.1.2 Updated Dup criteria to 25% per 11.3.2 of TO15 Updated 9.4.1.1 and 9.4.1.2 to 60C and 30 minutes for standard prep Added additional equations for curve fits	09May 2011

ATTACHMENT I - Target Compound List

Compound	CAS RN	TO14 compounds
1,1,1-Trichloroethane	71-55-6	X
1,1,2,2-Tetrachloroethane	79-34-5	X
1,12-trichloroethane	79-00-5	
1,1-Dichloroethane	75-34-3	X
1,1-Dichloroethene	75-35-4	X
1,2,4-Trichlorobenzene	95-63-6	X
1,2,4-Trimethylbenzene	95-63-6	X
1,2-Dibromoethane	106-93-4	X
1,2-Dichlorobenzene	95-50-1	X
1,2-Dichloroethane	107-06-2	X
1,2-Dichloropropane	78-87-5	X
1,3,5-Trimethylbenzene	108-67-8	X
1,3-Butadiene	106-99-0	
1,3-Dichlorobenzene	541-73-1	X
1,4-Dichlorobenzene	106-46-7	X
4-Ethyltoluene	622-96-8	
Acetone	67-64-1	
Acrolein	107-02-8	
Acrylonitrile	107-13-1	
Benzene	71-43-2	X
Benzyl Chloride	100-44-7	
Bromodichloromethane	75-27-4	
Bromoform	75-25-2	
Bromomethane	74-83-9	X
Carbon Disulfide	75-15-0	
Carbon Tetrachloride	56-23-5	X
Chlorobenzene	108-90-7	X
Chloroethane	75-00-3	X
Chloroform	67-66-3	X
Chloromethane	74-87-3	X
Cis-1,2-Dichloroethene	156-59-2	X
Cis-1,3-Dichloropropene	10061-01-5	X

ATTACHMENT I (continued)

Compound	CAS RN	TO14 compounds
Cyclohexane	110-82-7	
Dibromochloromethane	124-48-1	
Dichlorodifluoromethane	75-71-8	X
Dichlorotetrafluoroethane	76-14-2	X
Ethanol	64-17-5	
Ethyl Acetate	141-78-6	
Ethyl Benzene	100-41-4	X
Freon 113	76-13-1	X
Heptane	142-82-5	
Hexachlorobutadiene	87-68-3	X
Hexane	110-54-3	
Isopropyl Alcohol	67-63-0	
M,P Xylene	106-42-3	X
O-Xylene	95-47-6	X
Methyl Butyl Ketone	591-78-6	
Methyl Ethyl Ketone	78-93-3	
Methyl Isobutyl Ketone	108-10-1	
Methyl Tert Butyl Ether	1634-04-4	
Methylene Chloride	75-0902	X
Napthalene	91-20-3	
Propylene	115-07-1	
Styrene	100-42-5	X
Tetrachloroethene	127-18-4	X
Tetrahydrofuran	109-99-9	
Toluene	108-88-3	X
Trans-1,2-Dichloroethene	156-60-5	
Trans-1,3-Dichloropropene	10061-02-6	X
Trichloroethene	79-01-6	X
Trichlorofluoromethane	75-69-4	X
Vinyl Acetate	108-05-4	
Vinyl Chloride	75-01-4	X

*Current reporting limits can be found in Horizon

ATTACHMENT II - Required BFB Key Ions And Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (See note)
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

ATTACHMENT III - Characteristic Ions For Target Compounds

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Propylene	41	39	1
Dichlorodifluoromethane	85	87	1
Chloromethane	50	52	1
Dichlorotetrafluoroethane	85	135,87	1
Vinyl Chloride	62	64	1
1,3-Butadiene	54	39	1
Bromomethane	94	96	1
Chloroethane	64	66	1
Ethanol	31	45	1
Trichlorofluoromethane	101	103,105	1
Acetone	43	58	1
Isopropyl Alcohol	45	43	1
1,1-Dichloroethene	61	96	1
Freon 113	101	103,151	1
Methylene Chloride	49	84,86	1
Carbon Disulfide	76	44,78	1
Trans-1,2-Dichloroethene	96	61,98	1
Methyl Tert Butyl Ether	73	41	1
Vinyl Acetate	43	86	1
1,1-Dichloroethane	63	65	1
Methyl Ethyl Ketone	72	43	1
Hexane	57	41,43	1
Cis-1,2-Dichloroethene	96	61,98	1
Ethyl Acetate	43	61,70	1
Chloroform	83	85,47	1
Tetrahydrofuran	42	41,72	1
1,1,1-Trichloroethane	97	99,61	1
1,2-Dichloroethane	62	64	1
Benzene	78	77,50	1
Carbon Tetrachloride	117	119	1
Cyclohexane	56	84,41	1
Heptane	43	41	1
1,2-Dichloropropane	63	41,62	1
Trichloroethene	130	132,95	1

ATTACHMENT III (continued)

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Bromodichloromethane	83	85	1
Napthalene	128	127	1
Methyl Isobutyl Ketone	43	58,100	1
Cis-1,3-Dichloropropene	75	39,77	1
Trans-1,3-Dichloropropene	75	39,77	1
Toluene	91	92	1
1,12-trichloroethane	97	83,61	1
Methyl Butyl Ketone	43	58	2
Dibromochloromethane	129	127	2
1,2-Dibromoethane	107	109	2
Tetrachloroethene	166	164,131	2
Chlorobenzene	112	77,114	2
Ethyl Benzene	91	106	2
M,P,& O Xylene	91	106	2
Bromoform	173	171	2
Styrene	104	78,103	2
1,1,2,2-Tetrachloroethane	83	85	2
4-Ethyltoluene	105	120,79	2
1,3,5-Trimethylbenzene	105	120	2
1,2,4-Trimethylbenzene	105	120	2
1,3-Dichlorobenzene	146	111,148	2
Benzyl Chloride	91	126	2
1,4-Dichlorobenzene	146	148,111	2
1,2-Dichlorobenzene	146	111,148	2
1,2,4-Trichlorobenzene	180	182,184	2
Hexachlorobutadiene	225	227,223	2
1,4-Difluorobenzene	114	88	IS #1
Chlorobenzene	117	82	IS #2
Hexane-d14 (surr)	66	64	1
Toluene-d8 (surr)	98	100	1
1,4-Dichlorobenzene-d4 (surr)	150	152	2

ATTACHMENT IV - Calibration of THC as Gas

IV-1 THC as gas is calibrated by using the same calibration runs that are used for all other compounds, as well as using the same acceptance criteria.

IV-2 The original calibration files are copied to a target batch. This does not change the raw data in any way, it merely allows the same data to be processed against two different methods

IV-3 The area response is obtained by summing the area in the total ion chromatogram from the first eluting compound of interest till the end of the run. The internal standard is included as part of this value, the response factor is not calculated using the internal standard method. It is solely based on area response and calibration concentration

IV-4 The calibration concentration at each level is obtained by summing the values of the individual compounds present in the calibration standard.

IV-5 A response factor is obtained as detailed earlier in this SOP. Calibration criteria are the same as stated earlier in this SOP.

IV-6 Custom THC values may be obtained and are noted as such on final reports. These custom values can be based on calibrating using a select list of compounds or a select time frame for example. Requests for these custom values are to be evaluated on an individual basis for analytical feasibility.

ATTACHMENT V - Canister Dilution Factors (6L)

6 Liter Canister

Units (inches Hg or PSIG)	Initial Pressure	Initial Pressure Converted to PSIA	Final Pressure (PSIG)	Pressure Converted to PSIA	Dilution Factor
Hg	0	14.69	5	19.69	1.34
Hg	-1	14.22	5	19.69	1.38
Hg	-2	13.75	5	19.69	1.43
Hg	-3	13.28	5	19.69	1.48
Hg	-4	12.81	5	19.69	1.54
Hg	-5	12.35	5	19.69	1.59
Hg	-6	11.88	5	19.69	1.66
Hg	-7	11.41	5	19.69	1.73
Hg	-8	10.94	5	19.69	1.80
Hg	-9	10.47	5	19.69	1.88
Hg	-10	10	5	19.69	1.97
Hg	-11	9.53	5	19.69	2.07
Hg	-12	9.06	5	19.69	2.17
Hg	-13	8.59	5	19.69	2.29
Hg	-14	8.12	5	19.69	2.42
Hg	-15	7.66	5	19.69	2.57
Hg	-16	7.19	5	19.69	2.74
Hg	-17	6.72	5	19.69	2.93
Hg	-18	6.25	5	19.69	3.15
Hg	-19	5.78	5	19.69	3.41
Hg	-20	5.31	5	19.69	3.71
Hg	-21	4.84	5	19.69	4.07
Hg	-22	4.37	5	19.69	4.5
Hg	-23	3.9	5	19.69	5.04
Hg	-24	3.43	5	19.69	5.73
Hg	-25	2.97	5	19.69	6.64
Hg	-26	2.5	5	19.69	7.89
Hg	-27	2.03	5	19.69	9.71
Hg	-28	1.56	5	19.69	12.64
Hg	-29	1.09	5	19.69	18.08
PSIG	1	15.69	5	19.69	1.25
PSIG	2	16.69	5	19.69	1.18

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

1 Liter Canister

Initial Pressure Units (inches Hg or PSIG)	Initial Pressure	Initial Pressure Converted to PSIA	Final Pressure (PSIG)	Final Pressure Converted to PSIA	Dilution Factor
Hg	0	14.69	10	24.69	1.68
Hg	-1	14.22	10	24.69	1.74
Hg	-2	13.75	10	24.69	1.80
Hg	-3	13.28	10	24.69	1.86
Hg	-4	12.81	10	24.69	1.93
Hg	-5	12.35	10	24.69	2.00
Hg	-6	11.88	10	24.69	2.08
Hg	-7	11.41	10	24.69	2.16
Hg	-8	10.94	10	24.69	2.26
Hg	-9	10.47	10	24.69	2.36
Hg	-10	10	10	24.69	2.47
Hg	-11	9.53	10	24.69	2.59
Hg	-12	9.06	10	24.69	2.73
Hg	-13	8.59	10	24.69	2.87
Hg	-14	8.12	10	24.69	3.04
Hg	-15	7.66	10	24.69	3.22
Hg	-16	7.19	10	24.69	3.43
Hg	-17	6.72	10	24.69	3.67
Hg	-18	6.25	10	24.69	3.95
Hg	-19	5.78	10	24.69	4.27
Hg	-20	5.31	10	24.69	4.65
Hg	-21	4.84	10	24.69	5.10
Hg	-22	4.37	10	24.69	5.65
Hg	-23	3.9	10	24.69	6.33
Hg	-24	3.43	10	24.69	7.20
Hg	-25	2.97	10	24.69	8.31
Hg	-26	2.5	10	24.69	9.88
Hg	-27	2.03	10	24.69	12.2
Hg	-28	1.56	10	24.69	15.8
Hg	-29	1.09	10	24.69	22.7
PSIG	1	15.69	10	24.69	1.57
PSIG	2	16.69	10	24.69	1.48

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

AIR CANISTER DILUTIONS

When a sample is over the linear range of calibration for a compound of interest, several compounds of interest, or the matrix of the sample interferes with internal standard detections, a dilution is performed.

SYSTEM DILUTION
The pre-concentrator uses a digital mass flow controller to pull volume of the air sample onto the system.

1x = 500cc
2x = 250cc
5x = 100cc
10x = 50cc
20x = 25cc

SERIAL DILUTION
For samples that may require a dilution greater than 20x, the lab performs serial dilutions by emptying the pressurized air in the sample back to ambient conditions (0psig) and refilling the can to 15psig.
This doubles the volume once inside the can and is a 2x

As you multiply this process, the resultant dilution factor is multiplied out.

1. Flush to 0psig fill to 15 = **2x**
2. Flush to 0 and fill again to 15 = **4x**
3. **8x**
4. **16x**
5. **32x**
6. **64x**

ATTACHMENT VI - Air Laboratory Standard Preparation Procedures

CALIBRATION STANDARD

The calibration standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, INC. This is a custom mix that includes all compounds of interest at 1ppmv.

2 PPBV:

Using the 1000cc gas tight syringe, pull 90cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 2 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

20 PPBV:

Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

SECOND SOURCE VERIFICATION

The laboratory control standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas Solutions, or equivalent). This is a custom mix that includes all compounds of interest at 1ppmv.

20 PPBV:

Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

Internal Standard/ Surrogate/ BFB Standard 100ppbv:

The internal, surrogate, and bfb standards are purchased as neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent.

To prepare a neat/cocktail standard: Using Entech Static Dilution software, enter barometric pressure, room temperature, flask temperature, flask volume, canister pressure, canister volume, flask concentration and desired final concentration (100ppbv). The software calculates approximate transfer volume 1 (vial to flask), then transfer volume 2 (flask to canister)*. Note: Standard canister (6 L or 15 L) must be cleaned, evacuated, and humidified with 50ul H2O before being used.

Example:
Barometric Pressure: 29.92
Room Temperature : 24 degree C
Flask Temperature : 65 degree C
Flask Volume : 2000 ml
Canister Pressure : 30 psig
Canister Volume : 15,000 ml (15 L)
Flask Concentration : 520.015 PPM

*The software calculates transfer volume to th $1/10000^{th}$. The volumetric syringes are calibrated to $1/10^{th}$ of a decimal place. Therefore, the analyst adjusts the volume to a measurable amount prior to standard preparation.

Then, pressurize the 15 L canister 30 psig with clean nitrogen. This yields a final concentration of 100 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

ATTACHMENT VII - Procedures For Analyzing MPCA Samples

VII-1 Samples must be carefully monitored for carryover from previous samples with large detections. Analysts and data reviewers need to verify that each analysis has been evaluated for potential carryover.

- If a compound of interest has an on-column concentration that is greater than 10% of the previous sample, it is assumed that this value is not due to carryover.
- If the compound of interest has an on-column concentration between 2 and 10% of the previous sample, then the analyst carefully examines other factors relating to sample analysis (i.e. the concentration of related components, the overall concentration of constituents in each sample, etc.). When in doubt, the analyst must re-analyze the sample to confirm that the results are not due to carryover.
- When the compound of interest has an on-column concentration which is less than 2% of the previous sample's concentration, but greater than the method reporting limit, the sample must be analyzed to confirm or eliminate possible carryover.

VII-2 Sample duplicate analysis must be performed at a minimum of 1 in 10 samples analyzed.

VII-3 The relative detection limit for MPCA samples is 0.200 ppbv for all analytes except m&p xylene which has a relative detection limit of 0.400 ppbv.

ATTACHMENT VIII - Procedure for Tedlar Bags

Transfer of Tedlar Bags to SUMMA Canisters

In the event that a sample is collected into a tedlar bag, the client has 48 hours to get the bag to the facility for analytical testing. Pace Analytical Services recognizes a 48 hour holding time for all samples collected in tedlar bags. Upon receipt at the laboratory, the sample in the tedlar bag is transferred into a batch certified, evacuated one liter SUMMA canister for analysis. The sample is subsequently analyzed by the appropriate method within 28 days of transfer.

Procedure for transfer:

- Tedlar bag is received and logged for analysis by Pace Analytical Services
- The sample is delivered to the Air Lab, and the laboratory numbers assigned to the sample is recorded in a logbook (as delivered; see Attachment IX).
- The bag is connected to a clean, evacuated canister (105mTorr).
    - The tip of the bag valve is placed into tubing, connected by a ¼" nut to the sample valve of the canister, secured with a wrench to insure all sample is pulled into the can.
- The bag is opened first. Second, the can is opened.
    - By opening the canister second, the sample is transferred into the can through vacuum (since the can is evacuated to 150mTorr, and the bag is at ambient room pressure).
- After the sample is transferred the sample data and canister number, time and date, is recorded into the transfer logbook (Attachment X).
- Sample is submitted to the laboratory for analysis.
- A data qualifier is added to the report, notifying the client of the transfer.

ATTACHMENT IX - Tedlar Sign-off Logbook (example)

Pace Analytical

Tedlar Signoff Logbook

Date	Time	Project Number	Sample Number(s)	Method	Initials

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ATTACHMENT X - Tedlar Bag Transfer Log (example)

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Tedlar Bag Transfer Log

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Sample ID	Can ID	Date Collected	Date when Tedlar Bag was evacuated to the can	Comments

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Pace Analytical Services, Inc. – MN

Reviewed by:________________________
Date:________________________

ATTACHMENT XI – Common Logbook Abbreviations

RR	**Reanalysis for previously analyzed sample**
DIL	**Dilution for over-range compounds from a previously reported sample**
CONF	**Confirms results from a previously analyzed sample**
C/O	**Possible carryover from a prior sample**
OK	**Analysis is acceptable and sample is reported**

ATTACHMENT XII – Determination of Air Phase Petroleum Hydrocarbons (APH)

This method is designed, based on the Massachusetts APH method, to measure the gaseous-phase concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in air and soil gas. Volatile aliphatic hydrocarbons are collectively quantitated within two carbon number ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantitated within the C_9 to C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 28°C and 245°C. This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance.

Collective Aliphatic/Aromatic ranges: Relative Response Factors are calculated for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons based upon a correlation between the TOTAL mass of aliphatic APH Component Standards eluting within the range of interest and the total ion area count. A Relative Response Factor is calculated for C_9-C_{10} Aromatic Hydrocarbons based upon a correlation between the TOTAL mass of aromatic APH Component Standards eluting within this range and the total area count of extracted ions 120 and 134. Specified APH Component Standards are designated "marker" compounds to define the beginning and end of the hydrocarbon ranges.

- **C_5 through C_8 Aliphatic Hydrocarbon**s are defined as all aliphatic hydrocarbon compounds which elute from isopentane to just before n-nonane (C_9).
- **C_9 through C_{12} Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute from n-nonane to just after 1-methylnaphthalene.
- **C_9 through C_{10} Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds which elute from just after o-xylene to just after 1-methylnaphthalene, excluding naphthalene and 2-methylnaphthalene, which are quantitated and evaluated separately as Target APH Analytes.

Hydrocarbon Range	Beginning Marker	Ending Marker
C_5-C_8 Aliphatic Hydrocarbons	0.1 min. before isopentane	0.01 min. before n-Nonane
C_9-C_{12} Aliphatic Hydrocarbons	0.01 min. before n-Nonane	0.1 min. after 1-Methylnaphthalene
C_9-C_{10} Aromatic Hydrocarbons	0.1 min. after o-xylene	0.1 min. after 1-Methylnaphthalene

Standard Information: All APH standards are purchased as 30 component mixtures from a known vendor, such as SPEX CertiPrep or O2Si, in methanol.

Initial Calibration (*Suggested Parameters*)

- Standard Concentration: All components 10 ppmv
- Prepare a **20 ppbv** working standard by adding 36cc to a clean, evacuated 6L canister. Fill to 30psig.

Second Source Verification

- Standard Concentration: Components range from 30-70ug/ml
- Prepare working standard by adding 7.2ul to a clean, evacuated 6L canister. Fill to 30psig.

ATTACHMENT XII (continued) – Determination of Air Phase Petroleum Hydrocarbons (APH)

Initial Calibration and SSV Table:

	Volume (cc)	C5-C8	C9-C12	C9-C10
ICAL-1	10	5.2	6.4	2.4
ICAL-2	25	13	16	6
ICAL-3	50	26	32	12
ICAL-4	125	65	80	30
ICAL-5	250	130	160	60
ICAL-6	500	260	320	120
SSV	250	165	145	59

all expressed in ppbv

Component Mixture		Ions	
Compound	**CAS NO**	**Quant**	**Qual.**
1,3-Butadiene	106990	54	39
Isopentane	78784	43	42
MTBE	1634044	73	41
n-Hexane	110543	57	41/43
Benzene	71432	78	77/50
Cyclohexane	110827	56	84/41
2,3-Dimethylpentane	565593	56	43
n-Heptane	142825	43	41
Toluene	108883	91	92
n-Octane	111659	43	85/57
Ethylbenzene	100414	91	106
2,3-Dimethylheptane	3074713	43	84/85
m-Xylene	108383	91	106
p-Xylene	106423	91	106
o-Xylene	95476	91	106
n-Nonane	111842	43	57
Isopropylbenzene	98828	105	120
1-Methyl-3-ethylbenzene	620144	105	120
1,3,5-Trimethylbenzene	108678	105	120
n-Decane	124185	57	85
1,2,3-Trimethylbenzene	526738	105	120
p-Isopropyltoluene	99876	119	105
Indene	95136	115	116
Butylcyclohexane	1678939	83	55
n-Undecane	1120214	57	42
Naphthalene	91203	128	127
n-Dodecane	112403	57	43
Hexylcyclohexane	4292755	83	82
2-Methylnaphthalene	91576	142	141
1-Methylnaphthtalene	90120	142	141

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STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY 8260

REFERENCE METHOD: SW-846, METHOD 8260

SOP Number:	S-KS-O-012-rev.12
Effective Date:	Date of Final Signature
Supersedes:	S-KS-O-012-rev.11

APPROVALS

Laboratory General Manager — Date: 9/21/11

Laboratory Quality Manager — Date: 9/21/11

Group Leader — Date: 9/22/11

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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TABLE OF CONTENTS

1. Purpose

This Standard Operating Procedure (SOP) documents the procedures used by PASI –Kansas to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed SW-846 Methods 5030B, 5035A and 8260B.

2. Scope and Application

2.1 This method is applicable to most organic compounds that have boiling points below 200ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates).

2.2 This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data

2.3 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

2.4 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3. Summary of Method

Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract by purging with helium. The purged analytes are collected in a trap. At the completion of the purge time, the trap is rapidly heated and backflushed with helium to drive the trapped analytes into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

4. Interferences

4.1 Interferences can occur from a variety of sources including the purging gas, tubing, trap materials, flow controllers, and valves that use rubber components. When potential contaminants are present in method blanks the system requires maintenance.

4.2 Contamination may occur in a sample analyzed immediately after a sample containing high concentrations of volatile organic compounds (VOC). Rinsing the autosampler sparging vessels and lines between analyses can reduce this system carryover. Blanks should also be scheduled for analysis immediately after highly concentrated samples to demonstrate that the system is free from contamination.

4.3 Samples containing high levels of water soluble analytes, suspended solids, or high boiling compounds may require cleaning the sparging vessel with detergent, followed by rinsing with reagent water, and baking in an oven. Foaming samples or suspended solids may require dismantling the purge and trap system to clean or replace parts. Contamination may reach the GC, requiring cleaning the injector and liner, and either cutting or replacing the column.

4.4 Samples containing analytes with low purge efficiencies may result in significant amounts of the sample remaining in the sparging vessel after analysis. The sparging vessel may require additional rinsing in reagent water and baking in an oven.

4.5 Methylene chloride can permeate through PTFE tubing. All purge gas plumbing and GC gas carrier lines should be made from steel or copper tubing. Laboratory clothing exposed to methylene chloride is another potential source of contamination.

4.6 Diffusion of volatile organics through the septum seal of sample vials into the sample can occur during shipping and handling. A trip blank prepared from reagent water and carried through the entire process can serve as a check against this potential source of contamination.

4.7 HVAC systems and sample drains can be potential sources of contamination. Filters and traps can limit this potential source of contamination. Loading additional method blanks at every sample prep event can serve as a check against this source of contamination.

4.8 A mass spectrometer capable of very low detection limits increases the chance of detecting laboratory contamination as interferences.

4.9 Semivolatile peaks can elute after target analytes. The oven temperature program must include a bake-out period to ensure that semivolatile peaks elute.

5. Safety

5.1 Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2 Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

5.3 Equipment

Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

6. Definitions

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

6.2 Run Sequence Log – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

6.3 Toxicity Characteristic Leaching Procedure (TCLP) – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

6.4 Synthetic Precipitation Leaching Procedure (SPLP) – An extraction procedure meant to simulate the leaching of contaminants when exposed to acid rain.

6.5 Tune Period – The period after the BFB instrument tune check within which analyses may be performed.

6.6 EPIC Pro – LIMS developed by Pace Analytical (Environmental Project Information Control.

6.7 Holding Time – The elapsed time from the date/time of sample collection by the field personnel until the date/time of its processing/analysis. Holding time requirements are dictated by the method or QAPP.

6.8 Initial Calibration – The process of analyzing standards prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method.

6.9 Laboratory Control Sample – A control sample of known composition spiked with a known concentration of analytes of interest. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and analytical methods employed for field samples.

6.10 LIMS – Laboratory Information Management System.

6.11 Matrix – The predominant material of which the sample to be analyzed is composed.

6.12 Matrix Spike – Aliquot of sample fortified (spiked) with known quantities of specified target compounds or analytes and subjected to the entire sample preparation and analysis procedure in order to assess the appropriateness of the method for the sample matrix by measuring recovery.

6.13 Matrix Spike Duplicate – A second aliquot of the sample that is treated the same as the original matrix spike sample. The relative percent difference between the matrix spike and matrix spike duplicate is calculated and used to assess analytical precision.

6.14 Method Blank – An analytical control consisting of a blank matrix containing all reagents, internal standards and surrogate standards that are carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and contamination, and to demonstrate that this level does not exceed acceptance limits. Acceptable levels of contamination are defined by project specific data quality objectives.

6.15 Method Detection Limit (MDL)– The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. Method Detection Limits are determined using replicate spike samples prepared by the lab and taken through all preparation and analysis steps of the method. The method detection limit is calculated using the appropriate Student's t-parameter times the standard deviation of a series of spiked samples.

6.16 Precision – The measurement of agreement of a set of replicate results among themselves without any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis.

6.17 PQL – The practical quantitation limit (PQL) is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

6.18 Quality Assurance (QA) – A system of policies and procedures whose purpose is to ensure, confirm and document that the product or service rendered fulfills the requirements of Pace Analytical and it client. Quality Assurance includes quality planning, quality control, quality assessment (auditing), quality reporting and corrective action.

6.19 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.

6.20 Replicate Samples – Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between samples.

6.21 Reporting Limit (RL) – The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. Reporting limits are generally two times the MDL.

6.22 Standard Operating Procedure –A procedure adopted for repetitive use when performing specific measurement or sampling operation. It may be an industry accepted standard method or one developed by the user.

6.23 Traceability – The ability to trace the source and accuracy of a material (i.e. standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA.

7. Responsibilities and Distribution

7.1 Corporate Officers

7.1.1 **Chief Operating Officer (COO**) – The COO has oversight responsibility for Pace Analytical environmental laboratory operations, including compliance with all quality system requirements.

7.1.2 **Director of Quality, Safety and Training** – The Director of Quality, Safety and Training has oversight responsibility for PASI's quality programs, including establishing and monitoring compliance with all quality system requirements.

7.2 **Laboratory General Manager** – The Laboratory General Manager has overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the region. The General Manager will review and approve all SOPs.

7.3 **Quality Manager (QM)** – The QM is responsible for monitoring the implementation of the SOPs and the associated good laboratory practice. The QM will participate in the revision of the SOP and make sure it is current. The QM will review and approve all SOPs.

7.4 **Department Supervisor** – The supervisor must ensure that all analysts are properly trained and qualified to use this procedure. The supervisor is also responsible to ensure that the SOP is followed. The supervisor is responsible for reviewing the SOP and communicating recommended changes to the QM.

7.5 **Analyst** – Any analyst using this procedure is responsible for reading, understanding and following this SOP. Any deviation from this SOP must be reported to the appropriate Supervisor. The analyst must make their recommendations for changing the SOP to their supervisor or the QM in writing.

7.6 Revision – This SOP will be reviewed biennially at a minimum. It will also be revised as needed if procedures or methods change. In the event no changes have been made to the procedures, the cover page of this SOP will be signed and the expiration date will be extended for one year from the date of the reviewer's signature.

7.7 Distribution – The official version of this SOP is the hardcopy version bearing original signatures. A copy of the SOP shall be kept in the appropriate department and online for reference.

8. Sample Collection, Preservation and Handling

8.1 Aqueous

8.1.1 Collect in 40-mL VOA vials with PTFE-lined septa and screw cap (or equivalent) and store at ≤6°C. Prior to filling the vials with sample, sufficient 1:1 HCL (1-2 drops) is added to each vial to preserve the sample at pH<2. NOTE: Samples collected at Missouri Risk-Based Corrective Action for Petroleum Storage Tank sites must be preserved with sufficient trisodium phosphate dodecahydrate (TSP) to a pH >11.

8.1.2 Each sample vial is carefully filled and capped to eliminate headspace. Sample vials with bubbles larger than 6 mm should be noted and client notified. It is recommended to collect (3) vials for each sample and up to 9 for one of every 20 samples for MS/MSD analyses.

8.1.3 Samples must be analyzed within 14 days of the sample collection.

8.2 Soils/solids

8.2.1 Low-level aliquots are collected using specially prepared sampling kits containing a Terracore sampler and 40-mL VOA vials with PTFE-lined septa and screw cap. Three different kit options are utilized.

8.2.1.1 For general-purpose sampling a kit containing (5) 40-mL VOA vials and one sample container for moisture determination is utilized. Two 40-mL vials are empty, one vial contains 5-mL of methanol, and two vials contain sodium bisulfate.

8.2.1.2 Samples collected at Missouri Risk-Based Corrective Action for Petroleum Storage Tank sites must be preserved with sufficient trisodium phosphate dodecahydrate (TSP) to a pH >11. For these sites a sampling kit containing (5) 40-mL VOA vials and one sample container for moisture determination is utilized. Two 40-mL VOA vials are empty, one vial contains 5-mL of methanol, and two vials contain TSP.

8.2.1.3 For sites needing only medium-level analysis (e.g., UST sites) a kit with (3) 40-mL VOA vials containing 5-mL of methanol and one sample container for moisture determination is utilized.

8.2.2 All vials contained in these kits are tared and the weight recorded before being sent to the field for sample collection. The additional container is a bulk sample used for moisture determination.

8.2.3 If a sample demonstrates a high level of carbonates by effervescing on contact with the preservative, the sodium bisulfate should be eliminated for that sample. These samples should be noted on the chain of custody and the unpreserved vials used to analyze these samples.

8.2.4 Soil/solid samples collected in preserved containers must be stored at ≤6°C and analyzed within 14 days after collection.

8.2.5 Samples collected in unpreserved containers must placed in a freezer and be stored at –7 to -20 °C within 48 hours of collection and analyzed with 14 days of collection.

8.2.6 Upon customer request, four-ounce soil jars will be provided for sample collection. If samples are collected in four-ounce jars, they will be handled in one of two ways:

8.2.6.1 Standard procedure – 5-gram aliquots of the sample will be taken added to a 40-mL VOA vial at the time of analysis and 5 mL of reagent water or methanol added to the vial.

8.2.6.2 Special request – upon request the sample will be aliquotted from the 4 oz. jar into the appropriate sample kit described in Section 8.2.1 above and stored and analyzed as appropriate for the sample kit used.

8.3 Terracore or Encore sampling device - Low-level aliquots may also be collected in the sampling. The devices must be stored at ≤6°C and extruded into the proper low- or medium-level container vials within 48 hours of collection.

8.4 TCLP/SPLP Extracts - Extracts are collected in 40-mL VOA vials with PTFE-lined septa and screw cap with minimal headspace, preserved with 1:1 HCl and stored at ≤6°C until analysis. Samples must be analyzed no more than 14 days after collection from the Zero Headspace Extractor.

8.5 See Tables 8.1 and 8.2 for supplemental information regarding Trip Blank associations and sample collection, storage, preservation and holding time information.

Table 8.1 – Sample Collection, Preservation, Storage and Holding Time

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Two (2) VOA vials	Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace *Note:* 2-Chloroethylvinyl ether requires an unpreserved sample.	≤6°C	Unpreserved: 7 days pH Preserved: 14 days
Low-Level Aliquot Soil/Solid	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g aliquots in vials with magnetic stir bar, 5.0 mL reagent water and 1.0 g sodium bisulfate as needed. *OR (alternative):* Two (2) EnCore, TerraCore or similar sampling tubes.	No preservation *OR* sodium bisulfate *Note:* If sample effervesces on contact with the preservative, the sodium bisulfate should be eliminated for that sample. *OR* trisodium phosphate (TSP) for Missouri RBCA sites	≤6°C Without preservation (including EnCore, TerraCore or similar): ≤6°C for up to 48 hours before storing <-10°C, until analysis.	Unpreserved or **not** stored frozen: 48 hours Preserved with sodium bisulfate or stored frozen: 14 days Stored Frozen: 14 days
Medium-Level Aliquot Soil/Solid	One (1) 5-g aliquot in vial with 5.0 mL purge and trap grade MeOH. *OR (alternative)* One (1) 5-g aliquot in empty vial	Methanol - if sample was collected in empty vial it must be transferred into 10 mL of purge & trap grade MeOH within 48 hours of collection	With methanol: ≤6°C	Unpreserved: 48 hours Preserved with methanol: 14 days
TCLP/SPLP Extracts	Tedlar bag or three (3) VOA vials.	Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace Vials with bubbles larger than 5 mm should be discarded.	≤6°C	14 days from end of leaching procedure

Table 8.2 - Trip Blank Requirements

Sample type	Sample Preservation	Trip Blank Type	Quantity per cooler*
Aqueous	Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace.	40-mL vials per cooler - DI water acidified w/ 1:1 HCl (1-2 drops), no headspace.	2
	Basified w/ 0.6 g of Trisodium phosphate (TSP)	40-mL vials per cooler - DI water baseified w/ 0.6 g of TSP, no headspace.	2
	Unpreserved	40-mL vials per cooler - DI water, no headspace.	2
	Sodium thiosulfate	40-mL vials per cooler - DI water containing 3 mg sodium thiosulfate, no headspace.	2
Low-Level Aliquot Soil/Solid	Sodium bisulfate kit	40-mL vials – Containing a stir bar and 5 mL of DI water.	2
	TSP kit	40-mL vials – Containing a stir bar and 5 mL of DI water.	2
	Jar (4-oz.)	40-mL vials – Containing a stir bar and 5 mL of DI water.	2
	EnCore® Sampler	40-mL vials – Containing a stir bar and 5 mL of DI water.	2
Medium-Level Aliquot Soil/Solid	Methanol	Methanol Kit	1

* The quantity of trip blanks assembled may vary depending upon customer request.

9. Equipment and Supplies

Table 9.1 - Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatograph	Agilent	6890	or equivalent
Mass Spectrometer	Agilent	5973	or equivalent
Concentrator	Tekmar	LSC-3000	or equivalent
Autosampler	Tekmar	Aquatek	or equivalent
Data System	Thruput Systems, Inc.	Target, version 3.4	Pentium processor, 1GB HD, 500MB RAM (minimum)

Table 9.2 - Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Agilent	DB-624	121-1324	20m, 0.18mm, 1.0 um df
Trap	Supelco	Vocarb 3000	24940-U	Purge Trap K

Table 9.3 - Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	5-, 10-, 20-, 25-, 50-, 100-mL	Class A
VOA Vials	40-mL, clear, screw cap w/PTFE-lined septa	EP Scientific / 140-40C

Table 9.4 - General Supplies

Item	Description	Vendor/ Item # / Description
Microsyringes	10-, 25-, 100-, 500-, 1000-uL	Hamilton Series 1700
LVI vials	amber, 0.3-mL capacity w/screw cap	Fisher / 03-410-454
Helium	5.0 Ultra-High Purity	Praxair / HE 5.0UH

10. Reagents and Standards

10.1 Reagents

Table 10.1 – Reagents

Reagent	Concentration/ Description	Requirements / Vendor/ Item #
Methanol	Purge & Trap Grade (P&T)	Fisher / A453
Reagent water	purged with inert gas	ASTM Type II, Organic-free
Antifoam B® Silicone Emulsion		JT Baker / B531-05
Ottawa sand		Fisher / S23

10.2 Internal Standard / Surrogate Solution

Table 10.2 - Internal Standard / Surrogate Standard

Standard	Concentration	Vendor / Item #
8260 Internal/Surrogate Solution	2.5 mg/mL	o2si / 121500-01

10.2.1 SOLAtek 72 – Add 800 uL of the above standard to 19.2 mL of P&T methanol. This will yield a final concentration of 100 ug/mL.

10.2.2 AQUAtek 70 – Add 2.0 mL of the above standard to 18.0 mL of P&T methanol. This will yield a final concentration of 250 ug/mL.

10.2.3 Centurion - Add 200 uL of the above standard to 9.8 mL of P&T methanol. This will yield a final concentration of 50 ug/mL.

10.2.4 Atomx - Add 400 uL of the above standard to 9.6 mL of P&T methanol. This will yield a final concentration of 100 ug/mL.

Transfer solution to reservoir tube, affix to the autosampler and assign a one-month expiration date (not to exceed manufacturer's expiration date).

10.3 Calibration Stock Standard

Table 10.3 – Calibration Stock Constituents

Standard	Concentration	Vendor / Item #	Aliquot (uL)
Purgeable Gases, Mix B	2.0 mg/mL	Spex / 5022-BH	1000
Volatile Organics Combination Mix	2.0 mg/mL	Spex / 5242-VCX	1000
A-Mix	2.0 mg/mL	Spex / PCLKS-3	1000
Reactives	2.0 mg/mL	Spex / PCLKS-4	1000

Prepare by adding the indicated aliquots to a 20-mL, Class A volumetric flask containing P&T methanol, diluting to the mark and inverting three times to mix. Pipet into amber LVI vials and store at <–10ºC; assign a six-month expiration date (not to exceed manufacturer's expiration date).

10.4 Working Calibration Standard Preparation

Working calibration standards are prepared in reagent water for the purpose of direct analysis by the analytical instrument. These standards must be prepared in Class A volumetric flasks, with the exception of those standards with a 5-mL final volume, which are prepared in gastight syringes. Internal standard/surrogate compounds are added by the autosamplers during analysis.

10.4.1 Volumetric Flask Preparation

- Fill the expanded area of a Class A volumetric flask with reagent water.

- Use a microsyringe to rapidly inject the methanol solution into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection and bring to the mark with reagent water.
- Mix by inverting the flask three times only.
- Discard the contents contained in the neck of the flask. Aqueous standards are not stable and must be transferred to a VOA vial (without headspace) and sealed immediately.

10.4.2 Gastight Syringe Preparation (5-mL final volumes only)

- Fill a 5 mL gastight syringe with reagent water and adjust to the mark (no air bubbles).
- Inject an appropriate volume of the Calibration Stock Standard into the reagent water.
- Immediately transfer to a VOA vial and cap.

Table 10.4 – Working Standard Dilutions and Concentrations (10-mL purge volume)

Standard	Calibration Stock Standard Amount (uL)	Solvent	Final Volume (mL)	Final Concentration (ug/L)
Calibration Std 1	1.0	Water	250	0.4
Calibration Std 2	1.0	Water	100	1.0
Calibration Std 3	2.0	Water	100	5.0
Calibration Std 4	5.0	Water	50	10
Calibration Std 5	10	Water	50	20
Calibration Std 6	25	Water	50	50
Calibration Std 7	50	Water	50	100
Calibration Std 8	100	Water	50	200
Continuing Calibration Verification Standard	10	Water	50	20

Table 10.5 – Working Standard Dilutions and Concentrations (5-mL purge volume)

Standard	Calibration Stock Standard Amount (uL)	Solvent	Final Volume (mL)	Final Concentration (ug/L)
Calibration Std 1	2.0	Water	100	2.0
Calibration Std 2	2.5	Water	50	5.0
Calibration Std 3	5.0	Water	50	10
Calibration Std 4	10	Water	50	20
Calibration Std 5	2.5	Water	5	50
Calibration Std 6	5	Water	5	100
Calibration Std 7	15	Water	5	300
Continuing Calibration Verification Standard	5	Water	5	100

Note: When analyzing low-level soil samples, add 5 grams of Ottawa sand and a magnetic stirbar to each VOA vial before filling with standard.

10.5 LCS/MS Stock Standard

Table 10.6 – LCS/MS Stock Constituents

Standard	Concentration	Vendor / Item #	Aliquot (uL)
VOC Gas Mixture	2.0 mg/mL	Ultra Scientific / DWM-544	500
MegaMix	varied	Ultra Scientific / CUS-12313	500
1-,2-Methylnaphthalene	2.0 mg/mL	Ultra Scientific / CUS-12314	500
Reactives	varied	Ultra Scientific / CUS-12315	500

Prepare by adding the indicated aliquots to a 10-mL, Class A volumetric flask containing P&T methanol, diluting to the mark and inverting three times to mix. Pipet into amber LVI vials and store at <–10ºC; assign a six-month expiration date (not to exceed manufacturer's expiration date).

10.6 Second-Source Verification

Table 10.7 – Second-Source Verification (10-mL purge volume)

Standard	LCS/MS Stock Standard Amount (uL)	Solvent	Final Volume (mL)	Final Concentration (ug/L)
Second Source Verification Standard	10	Water	50	20

Prepare in reagent water using a volumetric flask.

Table 10.8 – Second-Source Verification (5-mL purge volume)

Standard	LCS/MS Stock Standard Amount (uL)	Solvent	Final Volume (mL)	Final Concentration (ug/L)
Second Source Verification Standard	5	Water	5	100

Prepare in reagent water using a gastight syringe.

11. Calibration

11.1 Tune Verification

The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see Table 12.2), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures.

Refer to Section 12.2 for details on the analysis and evaluation of this standard.

11.2 Analysis of Standards

11.2.1 An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to Tables 10.3 and 10.4 for compound concentrations.

11.2.2 An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 12.7.3. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

11.3 Calibration Response Factors

11.3.1 Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.3.2 To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (ug/L).
C_x = Concentration of the compound being measured (ug/L).

Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations.

The 8260B method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

11.4 Calibration Curve Fit

The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

Average Response Factor ($\overline{RF}$): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y-axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the $\overline{RF}$ (see Table 11.1).

The % RSD is calculated as follows: $\%RSD = \frac{SD \times 100}{\overline{RF}}$

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The **Calibration Check Compounds (CCCs)** are a subset of the target analyte list that must meet specific criteria (see Table 11.1) for the calibration to be acceptable. The %RSD for each of the CCCs is compared to the method criteria. If any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of y=ax+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y-axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve (see Table 11.1)

Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

Refer to Table 11.1 for curve fit criteria. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety and reanalyzed. Re-analysis should be within the same 12-hour time window and must occur within 8 hours of the original analysis.

11.5 Calibration Verification

11.5.1 Second Source Verification (SSV)

In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the "fit" of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as Secondary Source Verification or, alternatively as Initial Calibration Verification. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\%\text{Difference} = \frac{(\text{Result}_{SSV} - \text{True Value}_{SSV})}{\text{True Value}_{SSV}} \times 100$$

See Section 10.6 for details on the preparation of this standard. See Table 11.1 for control criteria

11.5.2 Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as ***Continuing Calibration Verification***. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method-specified criteria for the analysis to proceed for an additional 12 hours.

The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\%\text{Difference} = \frac{(RF_{CCV} - \overline{RF})}{\overline{RF}} \times 100 \quad (\text{curves based on } \overline{RF})$$

$$\%\text{Drift} = \frac{(\text{Result}_{CCV} - \text{True Value}_{CCV})}{\text{True Value}_{CCV}} \times 100 \text{ (curves based on linear or quadratic regression)}$$

Table 11.1 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD ≤ 15%	If not met, try linear regression fit
	Linear Regression	r ≥ 0.99	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	Chloromethane 1,1-Dichloroethane Bromoform Chlorobenzene 1,1,2,2-Tetrachloroethane	> 0.100 > 0.100 > 0.100 > 0.300 > 0.300	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system. See Appendix 1 for additional client-specific criteria.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Check Compounds (CCCs)	1,1-Dichloroethene Toluene Chloroform Ethylbenzene 1,2-Dichloropropane Vinyl chloride	%RSD ≤ 30%	%RSD for the calibration check compounds (CCC's) must be ≤30% regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a ≤15% RSD criterion. See Appendix 1 for additional client-specific criteria.
Second Source Verification Standard	Immediately after each initial calibration	% Diff ±30%	See Appendix 1 for additional client-specific criteria.
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	See Appendix 1 for additional client-specific criteria.
	Internal Standard RT	RT ± 30 sec	Use the mid-point standard level of the most recent initial calibration sequence as reference.
	Internal Standard Area	50 – 200%	Use the mid-point standard level of the most recent initial calibration sequence as reference.
	CCCs	RF ± 20% Diff. Result ± 20% Drift	Use for Avg RF calibration curves Use for linear and quadratic calibration curves

11.6 Calibration Corrective Actions

- Check all calculations including internal standard area counts and integrations.
- Check instrument performance against historical information
- Retune the instrument and run another initial calibration standard curve.
- Clean or replace traps, transfer lines, and valves on the autosampler and concentrator.
- Replace the injection port liner and cut 6-12" from the front of the column.
- Clean the ion source of the mass spectrometer.
- Prepare another working standard if there are indications that analyte concentrations have dropped or standard has become concentrated.

12. Procedure

12.1 Purge-Trap GC/MS System Preparation

12.1.1 Operating Parameters - Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 12.1 –Instruments and Operating Parameters

Instrument IDs	Component	Settings and Consumables	
60MSV##	Gas Chromatograph	Column: Agilent DB-624, 20m x 0.18mm x 1.0 um	Flow: 1.0 mL/min (constant) Purge Vent: On @ 0 min Split Flow: 38.9 mL/min Initial Temperature: 45ºC Initial Time: 2 min Rate: 22ºC/min Final Temperature: 225ºC Final Time: 0 min Injector Temperature: 225ºC Detector Temperature: 240ºC
	Mass Spectrometer	Tune File: bfb.u Electron Energy: 70 eV Scan Range: 35-450 amu Threshold: 150	Samples: 2 Solvent Delay: 0.60 min Scans/sec: 3.46 Acq. Mode: Scan
	Purge & Trap Concentrator	Trap: VOCARB 3000 Purge Ready: 60ºC Purge Temp: 0ºC Purge Time: 5 min Dry Purge: 0.5 min Desorb Preheat: 220ºC	Desorb: 4 min @ 225ºC Bake: 10 min @ 225ºC BGB: 0 min Valve: 150ºC Mount: 40ºC Transfer Line Temp: 150ºC
	Autosampler	Pressurize: 0.20 Fill IS: 0.02 ON Transfer Sample: 0.50 Rinse Lines: 0.25	Purge Lines: 0.50 Bake Rinse: 0.75 Bake Transfer: 0.50 Bake Rinses: 1

12.2 Tune Status Verification

At the beginning of each analytical sequence, prior to the analysis of any samples, the mass spectrometer tune conditions must be verified by analyzing a standard containing 4-bromofluorobenzene. The tune verification standard is combined with the CCV standard in this procedure

After the analysis of this standard, the mass spectrum of BFB must be evaluated against the following criteria.

Table 12.2 - BFB Abundance Criteria

Mass (m/z)	Ion Abundance Criteria
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95 (see note)
173	<2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0 to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120.0 percent that of m/z 95.

To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for bromofluorobenzene. If the software has a program or

macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order.

- Using an average of three scans, centered on the apex of the peak; or,
- Using an average of all scans across the width of the peak, taken at half height; or,
- Using an average of all scans taken across the width of the peak from baseline to baseline.

A background scan taken immediately before, but not including, the BFB peak must be subtracted.

Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours from the injection time for the BFB tune verification. After that, the tune must be verified again to establish a new analytical window.

12.3 Initial Calibration

12.3.1 Prepare Working Calibration Standards 1-8 (10-mL purge instrument) or Calibration Standards 1-7 (5-mL purge instrument) as specified in Section 10.4 and analyze.

12.3.2 Confirm that $\overline{RF}$ for the following System Performance Check Compounds (SPCC) meet the minimum response factors specified in Table 11.1. If the minimum response factors are not met then the chromatographic system is too reactive for sample analysis. Take corrective action before attempting recalibration.

12.3.3 Confirm that the RSD $\leq$ 30% for the Calibration Check Compounds (CCC). RSD >30% for any CCC may be indicative of system leaks or reactive sites on the column. Take corrective action before attempting recalibration.

12.3.4 If the RSD of any target analyte is 15% or less, then that compound's $\overline{RF}$ is assumed to be constant over the calibration range, and the $\overline{RF}$ may be used for quantitation.

12.3.5 If the RSD for any target analyte is greater than 15%, a calibration curve using linear (first-order) regression fit must be used for that compound, provided the correlation coefficient (r) is 0.99 or greater.

12.3.6 If the correlation coefficient for any target analyte is greater than 0.99, a quadratic (second-order) regression model must be used for that compound, provided the coefficient of determination (r^2) is 0.99 or greater and a minimum of six (6) initial calibration points are used.

12.3.7 Prepare the Second Source Verification as specified in Section 10.6 and analyze. Confirm that the Second Source Verification meets the criteria in Table 11.1. See Appendix 1 for additional client specific criteria.

12.4 Calibration Verification

The Calibration Verification consists of three steps that are performed at the beginning of each 12-hour analytical shift, in the following order: Tune status evaluation (Section 12.2), CCV Standard, Blank.

If the instrument meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed Section 11.6.

Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within Section 6 – Equipment and Measurement Traceability.

12.4.1 CCV Standard

12.4.1.1 Prepare a Continuing Calibration Verification Standard as specified in Section 10.4 and analyze.

12.4.1.2 Confirm that the SPCC compounds meet the minimum relative response factor criteria in Table 11.1.

12.4.1.3 Confirm that the responses of the CCC compounds have not varied from those in the initial calibration by more than 20%. Evaluate this by using percent difference (%D) for compounds that utilize the average response factor and percent drift for compounds that utilize linear or quadratic regression.

12.4.1.4 Evaluate the retention times and area responses for all internal standards in the CCV standard.

12.4.1.4.1 Confirm that the retention times have not varied by more than 10 seconds from those of the mid-point standard level of the most recent initial calibration sequence as reference.

12.4.1.4.2 Evaluate the area responses for all internal standards in the CCV standard. Confirm that they have not varied by more than a factor of two (-50 to +100%) from those of the mid-point standard level of the most recent initial calibration sequence as reference.

12.4.1.5 Blank

After every CCV a method blank or an instrument blank will be analyzed to demonstrate the analytical system is free of contamination.

12.5 Sample Analysis

All samples must be at room temperature when prepared for analysis. The system must be tuned, calibrated, and free of contamination before samples are analyzed.

12.6 Aqueous Samples

12.6.1 10-mL purge volume - Schedule the analysis on the autosampler according to the manufacturer's instructions and pace the sample vial into the scheduled slot

12.6.2 5-mL purge volume - Open the sample bottle and carefully pour the sample into a 5-mL gastight syringe just short of overflowing. Replace the syringe plunger. Adjust the sample volume to 5 mL. Transfer the contents of the syringe to a 40-mL VOA vial and cap.

12.6.3 TCLP/SPLP extracts are diluted by a factor of ten prior to analysis.

12.6.3.1 10-mL purge volume – Add 5 mL of extract to a 50-mL volumetric flask containing reagent water, bring to volume, invert three times only, discard the contents in the neck and transfer to a VOA vial.

12.6.3.2 5-mL purge volume – Add 0.5 mL of extract to a 5-mL syringe containing 4.5 mL of reagent water and transfer to a 40-mL VOA vial.

12.6.4 If any analyte concentration exceeds the calibration range of the instrument, the sample must be reanalyzed at a dilution so that the most concentrated analyte will be within the upper half of the calibration range.

12.6.5 Aqueous LCS preparation is identical to that of the Second Source Verification. See Section 10.6 for procedure.

12.6.6 MS/MSD (10-mL purge volume) Randomly select two vials of a client sample for matrix spiking. Uncap vials; spike each with 8 uL of the LCS/MS Stock Standard and recap.

12.6.7 MS/MSD (5-mL purge volume) Randomly select a client sample for matrix spiking. Fill a 5-mL syringe with sample and adjust to the mark. Inject 5 uL of LCS/MS Stock Standard, immediately transfer to a VOA vial and cap. Repeat for the Matrix Spike Duplicate.

12.6.8 Preservation pH Determination

12.6.8.1 10-mL purge volume - The pH of the sample is checked after analysis. An aliquot from the used sample vial is poured directly onto a pH strip. Excess liquid is to be expelled from the strip before the pH value is read and recorded in the instrument run logbook.

12.6.8.2 5-mL purge volume - The pH of the sample is checked before analysis. Residual liquid from the syringe used to load the sample is poured onto a pH strip. Excess liquid is to be expelled from the strip before the pH value is read and recorded in the instrument run logbook.

12.6.8.3 If the pH is less than or equal to 2 for an HCl-preserved sample, no further action is required provided that the sample was analyzed within 14 days.

12.6.8.4 If the pH is greater than 2 and the sample was analyzed within 7 days then the analysis is within method compliance.

12.6.8.5 If the pH is greater than 2 and the sample was analyzed after 7 days from the collection date, then a footnote must be added to the sample results indicating method noncompliance.

NOTE: Samples collected at Missouri Risk-Based Corrective Action for Petroleum Storage Tank sites must be preserved with sufficient trisodium phosphate dodecahydrate (TSP) to a pH ≥11.

12.6.8.6 Foaming samples

Samples will occasionally foam when purging, which creates the potential for severe instrument contamination if the foaming sample enters the analytical trap, and possibly into the GC column. Take corrective action (i.e. replace trap, rinse or replace concentrator/transfer lines, etc.) to return the instrument to control. Dilute the sample and add Antifoam B® Silicone Emulsion prior to reanalysis:

- Warm antifoam agent to 40ºC (e.g., in GC oven).
- Shake vigorously for 3 minutes.
- Wet bore of a 10-uL syringe with methanol (draw/dispense 10uL methanol 3 times).
- Very slowly draw 1uL antifoam agent into syringe (should take 10 - 30 seconds).
- Inject antifoam agent into 5-mL water sample.
- Draw/dispense 10uL sample/antifoam mix 3 times, to transfer all antifoam agent to sample.
- Always include an antifoam-treated blank, LCS, MS/MSD with antifoam-treated samples.

12.6.9 Low-Level Soils Collected in Sampling Kits

12.6.9.1 Weigh the sample and vial to the nearest 0.01 gram. Subtract the tare weight from the weight measured to determine the sample weight.

12.6.9.2 If the samples were placed in the freezer within 48 hours of collection analyze one of the unpreserved vials; otherwise, analyze a preserved vial.

12.6.9.3 Prepare the LCS by injecting 5 uL of LCS/MS Stock Standard into a gastight syringe containing 5 mL of reagent water and transferring to a VOA vial containing 5g of Ottawa sand and a magnetic stirbar.

12.6.9.4 Randomly select two vials of a client sample for matrix spiking. Spike each with 5 uL of the LCS/MS Stock Standard by injecting through the septa.

12.6.9.5 Schedule the analysis on the autosampler according to the manufacturer's instructions. Have the autosampler add 5 mL of reagent water plus internal standards and surrogates to the sample prior to analysis.

12.6.9.6 Place the sample vials into the scheduled slots.

12.6.9.7 If any analyte concentration exceeds the calibration range of the instrument, a medium-level analysis must be performed using the methanol-preserved vial.

12.6.10 Low-Level Soils Collected in 4-oz. Jars

12.6.10.1 Weigh 5.0 ± 0.1 g sample into a 40-mL VOA vial, recording the sample weight to the nearest 0.1 g in the balance logbook, add 5.0 mL reagent water and a magnetic stirbar and cap.

12.6.10.2 Prepare the LCS by injecting 5 uL of LCS/MS Stock Standard into 5 mL of reagent water in a gastight syringe and adding to a VOA vial containing 5g of Ottawa sand and a magnetic stirbar.

12.6.10.3 Randomly select a client sample for matrix spiking. Weigh two additional 5.0 g ± 0.1 g sample aliquots into 40-mL VOA vials, recording the sample weight to the nearest 0.1 g in the balance logbook. Add 5.0 mL reagent water and magnetic stirbar to each and spike with 5 uL of the LCS/MS Stock Standard by injecting through the septa.

12.6.10.4 Schedule the analysis on the autosampler according to the manufacturer's instructions. Have the autosampler add 5 mL of reagent water plus internal standards and surrogates to the sample prior to analysis.

12.6.10.5 Place the sample vials into the scheduled slots.

12.6.10.6 If any analyte concentration exceeds the calibration range of the instrument, a smaller weighed portion of the sample must be analyzed. If the aliquot size will be less than 1.0 g, proceed to the medium-level soil analysis in the following section.

12.6.11 Medium-Level Soils in Sampling Kits

12.6.11.1 Weigh the sample and vial to the nearest 0.01 gram. Subtract the tare weight from the weight measured to determine the sample weight.

12.6.11.2 If the sample contact time with methanol is less than 24 hours, mix the contents of the vial on the sonicator for 20 minutes. Allow the contents of the vial to settle for several minutes.

12.6.11.3 10-mL purge volume - Fill the expanded area of a 50-mL Class A volumetric flask with reagent water. Inject 500 uL of the methanol extract and dilute to the mark. Mix by inverting the flask three times only; discard the contents in the neck of the flask and transfer to a 40-mL VOA vial.

Randomly select a client sample for matrix spiking. Dilute 500 uL of the methanol extract and 10 uL of LCS/MS Stock Standard in 50 mL reagent water using a volumetric flask. Repeat for the Matrix Spike Duplicate.

12.6.11.4 5-mL purge volume - Fill a 5-mL gastight syringe barrel to 4.9 mL with reagent water. Add a 100-uL aliquot of the methanol extract to the contents of the syringe. Transfer the entire syringe contents to a 40-mL VOA vial.

Randomly select a client sample for matrix spiking. Fill a 5-mL gastight syringe barrel to 4.9 mL with reagent water. Add 5 uL of the LCS/MS Stock Standard and 100-uL aliquot of the methanol extract to the contents of the syringe. Transfer the entire syringe contents to a 40-mL VOA vial. Repeat for the Matrix Spike Duplicate.

12.6.11.5 Analyze the sample as if it were a water sample, having the autosampler add internal standards and surrogates.

12.6.12 Medium-Level Soils Collected in 4 oz. Jars

12.6.12.1 Weigh 10.0 g ± 0.1 g sample into a 40-mL VOA vial. Record the sample weight to the nearest 0.1 g in the balance logbook.

12.6.12.2 Using the methanol dispenser, add 10.0 mL of methanol to the contents of the vial.

12.6.12.3 Mix the contents of the vial in the sonicator bath for 20 minutes. Allow the contents of the vial to settle for several minutes.

12.6.12.4 10-mL purge volume - Fill the expanded area of a 50-mL Class A volumetric flask with reagent water. Inject 500 uL of the methanol extract and dilute to the mark. Mix by inverting the flask three times only; discard the contents in the neck of the flask and transfer to a 40-mL VOA vial.

Randomly select a client sample for matrix spiking. Dilute 500 uL of the methanol extract and 10 uL of LCS/MS Stock Standard in 50 mL reagent water using a volumetric flask. Repeat for the Matrix Spike Duplicate.

12.6.12.5 5-mL purge volume - Fill a 5-mL gastight syringe barrel to 4.9 mL with reagent water. Add a 100-uL aliquot of the methanol sample extract to the contents of the syringe. Transfer the entire syringe contents to a 40-mL VOA vial.

Randomly select a client sample for matrix spiking. Fill a 5-mL gastight syringe barrel to 4.9 mL with reagent water. Add 5 uL of the LCS/MS Stock Standard and 100-uL aliquot of the methanol extract to the contents of the syringe. Transfer the entire syringe contents to a 40-mL VOA vial. Repeat for the Matrix Spike Duplicate.

12.6.12.6 Analyze the sample as if it were a water sample, having the autosampler add internal standards and surrogates.

12.7 Data Interpretation

12.7.1 Qualitative Analysis

Suspected target compounds will be identified by comparison of the sample mass spectrum to that of the NIST library reference spectrum. Three criteria must be satisfied to verify the identification:

12.7.1.1 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component

12.7.1.2 The intensities of the characteristic ions of a compound must maximize within one scan of each other. The characteristic ions are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum.

12.7.1.3 The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. For example, for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

12.7.1.4 See Appendix 1 for additional client-specific criteria.

12.7.2 Quantitative Analysis

Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

Raw Data Results: The GC/MS data system will calculate the concentration of each analyte as ug/L (or ng/mL). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the calibration curve.

12.7.2.1 Calculations

$$\text{Concentration}\left(\text{ug/L}\right) = \frac{\left(X_s\right)\left(D\right)}{\left(V_s\right)}$$

$$\text{Concentration}\left(\text{ug/kg}\right) = \frac{\left(X_s\right)\left(V_t\right)\left(D\right)}{\left(V_i\right)\left(W_s\right)}$$

Where:

X_s = Calculated mass of the analyte (in nanograms) in the sample aliquot introduced into the instrument. The type of calibration model used determines the derivation of X_s.

V_t = Total volume of the methanol extract (in uL).

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, then D=1. The dilution factor is always dimensionless.

V_s = Volume of the aqueous sample purged, in milliliters (mL).

V_i = Volume of the extract added to reagent water and purged (in uL). Any dilutions made to the initial volume of the solvent extract are accounted for in the dilution factor (D).

W_s= Weight of sample extracted or purged (in grams). The wet weight or dry weight may be used, depending upon the specific applications of the data.

12.7.3 Tentatively Identified Compounds (TIC)

Non-target analytes may be tentatively identified and reported upon request. A library search is performed for the purpose of tentative identification. Guidelines for TIC identification are:

12.7.3.1 All ions present in the reference mass spectrum at a relative intensity >10% should be present in the sample spectrum.

12.7.3.2 The relative intensities of the major ions should agree within ± 20%. For example, an ion with an abundance of 50% in the reference spectrum must be present in the sample between 30 and 70% abundance.

12.7.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.7.3.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.7.3.5 Ions present in the reference spectrum but not the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

12.7.3.6 If, in the opinion of the analyst, no valid tentative identification can be made, the compound should be reported as an unknown. The analyst should give additional classification of the unknown compound, if possible.

12.7.4 An estimated concentration for TICs and unknowns shall be calculated by the internal standard method. Use the total ion count of the nearest internal standard free from interferences, assuming a response factor of 1.

13. Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples or 12-hour window (whichever is most frequent).	1) Target analytes must be less than reporting limit. 2) If results are reported to MDL, target analytes in MB should be non-detect	1) Re-analyze associated samples. ***Exceptions:*** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value.

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	Method-specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene *OR (alternative)* Full Target List compounds	One per 20 samples or 12-hour window (whichever is most frequent).	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	1) Analyze a new LCS 2) If problem persists, check spike solution 3) Perform system maintenance prior to new LCS run ***Exceptions:*** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Method-specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene *OR (alternative)* Full Target List compounds	One set per 20 samples or 12-hour window (whichever is most frequent). Must include at least one TCLP/SPLP MS when any analyzed in batch.	% Recovery/ %RPD: Laboratory derived limits	1) If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences

Table 13.2 – Sample Quality Control criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Fluorobenzene, Chlorobenzene-d5 1,4-dichlorobenzene-d4	Added to all samples, spikes, control samples and method blanks prior to purging.	IS peak area within 50-200% of associated CCV standard. IS retention time within ±30 seconds of the mid-point standard level of the most recent initial calibration sequence.	**Recovery Failure:** 1) Re-analyze sample to confirm failure 2) Assess impact of sample matrix 3) In the absence of obvious matrix interference, reanalyze sample. **Retention Time Failure:** 1) If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting. 2) Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Dichloroethane-d4 Dibromofluoromethane Toluene-d8 4-Bromofluorobenzene	Added to all samples, spikes, control samples and method blanks prior to purging.	SS recoveries within laboratory-derived limits	**Recovery Failure:** 1) Check system parameters 2) Identify and correct likely cause 3) Re-run samples ***Exceptions:*** Surr rec above criteria and target compounds < RL, result may be reported with appropriate footnote. Surr rec out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

14. Method Performance

14.1 Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months per S-ALL-Q-004, Method Detection Limit Studies, for each matrix per instrument. Study results constitute the Limits of

Detection (LODs) for the analytes in this procedure and can be accessed through the laboratory's EPIC Pro LIMS system.

14.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per ALL-Q-020, Training Procedures.

14.2.1 Aqueous - Analysis of four replicates of reagent water spiked with LCS/MS Spiking Standard at a concentration of 20 ug/L or equivalent to the LCS.

14.2.2 Low-Level Soil - Analysis of four 5-g replicates of Ottawa sand spiked with LCS/MS Spiking Standard at a concentration of 100 ug/kg or equivalent to the LCS.

14.2.3 Medium-Level Soil – Extraction and analysis of four 5-g replicates of Ottawa sand spiked with LCS/MS Spiking Standard at a concentration of 1000 ug/kg or equivalent to the LCS.

14.2.4 If the recoveries are within the matrix-specific LCS recovery limits and the RSDs are <30%, system performance is acceptable and analysis of samples may begin. If any recovery falls outside the acceptance range, or an RSD exceeds the precision limit, system performance is unacceptable. In this event, correct the problem and repeat the test.

15. Method Modifications

Method modifications for EPA method 8260B are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- Stock standards containing gas components are given a six-month expiration date

16. Pollution Prevention and Waste Management

16.1 Procedures for handling waste generated during this analysis are addressed in S-ALL-S-002, Waste Handling.

16.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

16.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, Etc.

17.1 Table 1 - 8260B Quantitation Limits

17.2 Table 2 - 8260B TCLP/SPLP Quantitation Limits

17.3 Table 3 - Primary Ions of Target Analytes and Reference Compounds

17.4 Appendix 1 – Client-Specific Requirements

18. References

18.1 EPA Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Update IV, 2/2007, Chapters 1-4, Methods 5030B, 5035A, 8000B, 8260B.

18.2 USEPA CLP Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration OLM04.2, 5/1999.

18.3 Procedure for Using Antifoam Agent, Restek Corporation, 400-00 [002].

19. Revisions

Document Number	Change	Date
KS-O-012-rev.8	Grammatical/Removal of outdated information. Changed BFB tuning criteria from 8260B to CLP SOW OLM04.2.	July 15, 2005
KS-O-012-rev.9	Section 5.3 – Location of MSDS sheets changed. Section 8.1.2 – Changed 5 mm to 6 mm. Section 11.1.3 – Combined LCS and MS/MSD Standard sections. Changed concentration to 50 ug/mL. Section 12.12.2.3 – Changed 4 grams to 10 grams. Section 13 – Added notations for WYDEQ STP batching requirements. Section 13.2.1 – Added "20 samples" Section 13.2.2 – Changed "per day" to "within a 12-hour period. Add 5 grams Ottawa sand to soil LCS. Section 13.2.3 – Changed "per day" to "within a 12-hour period. Add 5 grams sample to soil MS. Section 16.3 – Doubled aliquots and concentrations. Added "113" to Freon. Table 1 – Deleted Medium Soil PRLs. Section 16.4 - Changed "2.0 ug/mL" to "2.0 ug/mL." Combined A and B Calibration stds. Final volume now 2.0 mL. Added tert-Butyl Alcohol.	March 22, 2006
KS-O-012-rev.10	Section 6 - Removed unnecessary definitions. Section 7.4 - Revised review frequency from biennial to annual Section 8 - Revised 4 C to ≤6 C, added tables 8.1 and 8.2. Section 11 – Changed amount of BFB for tuning. Changed CCV internal standard RT criteria to 10 sec. Section 14 – Added ½ PRL blank criteria. Table 1 – Changed TBA PQL. Appendix 3 – Changed purge time Appendix 4 – Revised standard prep. Appendix 6 - Added IDOC criteria.	August 23, 2008
S-KS-O-012-rev.11	SOP – Updated format and content to corporate template Section 7 – Revised distribution. Section 8 – Changed soil freeze temp to <–10ºC. Section 10 – Added sand and antifoam. Revised standard prep/storage/exp. date procedure. Section 11 – Revised SSV criteria. Added thermal decomp standard. Section 12 – Revised calculations. Added thermal decomp standard. Added SPLP dilution.	August 23, 2010
S-KS-O-012-rev.12	SOP – Removed thermal decomposition standard. Section 12 – Changed IS RT criteria to 30 sec. Section 13 – Removed Wyoming UST batching requirements. Appendix 1 – Client-specific criteria for internal use only.	September 21, 2011

Table 1 – 8260B Quantitation Limits

Analytes	CAS #	Water (ug/L)	Low-Level Soil (ug/kg)	Medium-Level Soil (ug/kg)	TCLP/ SPLP (ug/L)
1,1,1,2-Tetrachloroethane	630-20-6	1	5	50	—
1,1,1-Trichloroethane	71-55-6	1	5	50	—
1,1,2,2-Tetrachloroethane	79-34-5	1	5	50	—
1,1,2-Trichloroethane	79-00-5	1	5	50	—
1,1,2-Trichlorotrifluoroethane	76-13-1	1	5	50	—
1,1-Dichloroethane	75-34-3	1	5	50	—
1,1-Dichloroethene	75-35-4	1	5	50	50
1,1-Dichloropropene	563-58-6	1	5	50	—
1,2,3-Trichlorobenzene	87-61-6	1	5	50	—
1,2,3-Trichloropropane	96-18-4	2.5	5	125	—
1,2,4-Trichlorobenzene	120-82-1	1	5	50	—
1,2,4-Trimethylbenzene	95-63-6	1	5	50	—
1,2-Dibromo-3-chloropropane	96-12-8	2.5	10	125	—
1,2-Dibromoethane (EDB)	106-93-4	1	5	50	—
1,2-Dichlorobenzene	95-50-1	1	5	50	—
1,2-Dichloroethane	107-06-2	1	5	50	50
1,2-Dichloroethene (Total)	156-60-5	1	5	50	—
1,2-Dichloropropane	78-87-5	1	5	50	—
1,3,5-Trimethylbenzene	108-67-8	1	5	50	—
1,3-Dichlorobenzene	541-73-1	1	5	50	—
1,3-Dichloropropane	142-28-9	1	5	50	—
1,4-Dichlorobenzene	106-46-7	1	5	50	—
1,4-Dioxane (p-Dioxane)	123-91-1	100	100	5000	—
1-Methylnaphthalene	90-12-0	10	10	500	—
2,2-Dichloropropane	590-20-7	1	5	50	—
2-Butanone (MEK)	78-93-3	10	10	500	1000
2-Chloroethylvinyl ether	110-75-8	10	5	500	—
2-Chlorotoluene	95-49-8	1	5	50	—
2-Hexanone	591-78-6	10	20	500	—
2-Methylnaphthalene	91-57-6	10	10	500	—
4-Chlorotoluene	106-43-4	1	5	50	—
4-Methyl-2-pentanone (MIBK)	108-10-1	10	10	500	—
Acetone	67-64-1	10	20	500	—
Acetonitrile	75-05-8	20	100	1000	—
Acrolein	107-02-8	100	100	5000	—
Acrylonitrile	107-13-1	20	100	1000	—
Benzene	71-43-2	1	5	50	50
Bromobenzene	108-86-1	1	5	50	—
Bromochloromethane	74-97-5	1	5	50	—
Bromodichloromethane	75-27-4	1	5	50	—
Bromoform	75-25-2	1	5	50	—
Bromomethane	74-83-9	1	5	50	—
Carbon disulfide	75-15-0	5	5	250	—
Carbon tetrachloride	56-23-5	1	5	50	50
Chlorobenzene	108-90-7	1	5	50	50
Chloroethane	75-00-3	1	5	50	—
Chloroform	67-66-3	1	5	50	200
Chloromethane	74-87-3	1	5	50	—
cis-1,2-Dichloroethene	156-59-2	1	5	50	—
cis-1,3-Dichloropropene	10061-01-5	1	5	50	—
Cyclohexane	110-82-7	5	10	250	—
Cyclohexanone	108-94-1	10	20	500	—
Dibromochloromethane	124-48-1	1	5	50	—

Analytes	CAS #	Water (ug/L)	Low-Level Soil (ug/kg)	Medium-Level Soil (ug/kg)	TCLP/ SPLP (ug/L)
Dibromomethane	74-95-3	1	5	50	
Dichlorodifluoromethane	75-71-8	1	5	50	—
Diethyl ether	60-29-7	1	5	50	—
Diisopropyl ether	108-20-3	1	5	50	—
Ethyl tert-butyl ether	637-92-3	1	5	50	—
Ethylbenzene	100-41-4	1	5	50	—
Hexachloro-1,3-butadiene	87-68-3	1	5	50	—
Iodomethane	74-88-4	10	10	500	—
Isopropylbenzene (Cumene)	98-82-8	1	5	50	—
m&p-Xylene	108-38-3 / 106-42-3	2	5	100	—
Methyl acetate	79-20-9	20	100	5000	—
Methyl tert-butyl ether	1634-04-4	1	5	50	—
Methylcyclohexane	108-87-2	5	10	250	—
Methylene chloride	75-09-2	1	5	50	—
Naphthalene	91-20-3	10	10	500	—
n-Butylbenzene	104-51-8	1	5	50	—
n-Heptane	142-82-5	5	10	250	—
n-Hexane	110-54-3	5	10	250	—
n-Propylbenzene	103-65-1	1	5	50	—
o-Xylene	95-47-6	1	5	50	—
p-Isopropyltoluene	99-878-6	1	5	50	—
sec-Butylbenzene	135-98-8	1	5	50	—
Styrene	100-42-5	1	5	50	—
tert-Amyl methyl ether	994.05-8	1	5	50	—
tert-Butyl alcohol	75-65-0	10	25	500	—
tert-Butylbenzene	98-06-6	1	5	50	—
Tetrachloroethene	127-18-4	1	5	50	50
Toluene	108-88-3	1	5	50	—
trans-1,2-Dichloroethene	156-60-5	1	5	50	—
trans-1,3-Dichloropropene	10061-02-6	1	5	50	—
trans-1,4-Dichloro-2-butene	110-57-6	20	20	1000	—
Trichloroethene	79-01-6	1	5	50	50
Trichlorofluoromethane	75-69-4	1	5	50	—
Vinyl acetate	108-05-4	20	100	1000	—
Vinyl chloride	75-01-4	1	5	50	100
Xylene(s), Total	1330-20-7	3	5	150	—

Table 2 - Primary Ions of Target Analytes and Reference Compounds

Compounds	Primary Ion	Secondary Ion	Internal Standard
Internal Standards			
1,4-Difluorobenzene	114	-	1
Chlorobenzene-d5	117	-	2
1,4-Dichlorobenzene-d4	152	115, 150	3
Surrogates			
Dibromofluoromethane	113	111	1
1,2-Dichloroethane-d4	65	102	1
Toluene-d8	98	100	2
4-Bromofluorobenzene	95	174, 176	3
Target Analytes			
1,1,1-Trichloroethane	97	99	1
1,1,2-Trichlorotrifluoroethane	101	151	1
1,1-Dichloroethane	63	65, 83	1
1,1-Dichloroethene	96	61, 63	1
1,1-Dichloropropene	75	77	1
1,2-Dichloroethane	62	64	1
1,2-Dichloroethene (each isomer)	96	61, 98	1
1,2-Dichloropropane	63	62	1
1,4-Dioxane (p-Dioxane)	88	58	1
2,2-Dichloropropane	77	97	1
2-Butanone (MEK)	43	72	1
2-Chloroethylvinyl ether	63	65	1
Acetone	43	58	1
Acetonitrile	41	40,39	1
Acrolein	56	55	1
Acrylonitrile	53	52	1
Benzene	78	57, 77	1
Bromochloromethane	128	130	1
Bromodichloromethane	83	85	1
Bromomethane	94	96	1
Carbon disulfide	76	78	1
Carbon tetrachloride	117	119	1
Chloroethane	64	66	1
Chloroform	83	85	1
Chloromethane	50	52	1
cis-1,2-Dichloroethene	96	61,98	1
cis-1,3-Dichloropropene	75	110	1
Cyclohexane	56	84	1
Dibromomethane	93	95, 174	1
Dichlorodifluoromethane	85	87	1
Diethyl ether	74	45,59	1
Diisopropyl ether	45	59, 87	1
Ethyl tert-butyl ether	59	87	1
Iodomethane	42	127	1
Methyl acetate	43	74	1
Methylcyclohexane	83	55,98	1
Methylene chloride	84	49	1
Methyl-tert-butyl ether	73	57	1
n-Heptane	57	41	1
n-Hexane	57	41	1

Compounds	Primary Ion	Secondary Ion	Internal Standard
tert-Amyl methyl ether	73	55	1
tert-Butyl alcohol	59	51	1
trans-1,2-Dichloroethene	96	61, 98	1
trans-1,4-Dichloro-2-butene	75	53, 124	1
Trichloroethene	95	130, 132	1
Trichlorofluoromethane	101	151	1
Vinyl acetate	43	86	1
Vinyl chloride	62	64	1
1,1,1,2-Tetrachloroethane	131	133	2
1,1,2-Trichloroethane	83	97, 85	2
1,2-Dibromoethane (EDB)	107	109	2
1,3-Dichloropropane	76	78	2
2-Hexanone (MBK)	43	58, 100	2
4-Methyl-2-pentanone (MIBK)	43	58	2
Bromoform	173	175	2
Chlorobenzene	112	77, 114	2
Cyclohexanone	55	42,98	2
Dibromochloromethane	129	127	2
Ethylbenzene	106	91	2
Isopropylbenzene	105	120	2
m,p-Xylene	106	91, 105	2
o-Xylene	106	91, 105	2
Styrene	104	78	2
Tetrachloroethene	166	168	2
Toluene	92	91	2
trans-1,3-Dichloropropene	75	77	2
Xylene(s), Total	106	91, 105	2
1,1,2,2-Tetrachloroethane	83	85	3
1,2,3-Trichlorobenzene	180	182, 145	3
1,2,3-Trichloropropane	110	77	3
1,2,4-Trichlorobenzene	180	182, 145	3
1,2,4-Trimethylbenzene	105	120	3
1,2-Dibromo-3-chloropropane	75	155, 157	3
1,2-Dichlorobenzene	146	111, 148	3
1,3,5-Trimethylbenzene	105	120	3
1,3-Dichlorobenzene	146	148	3
1,4-Dichlorobenzene	146	111, 148	3
1-Methylnaphthalene	142	141	3
2-Chlorotoluene	126	125	3
2-Methylnaphthalene	142	141	3
4-Chlorotoluene	126	125	3
Bromobenzene	156	77, 158	3
Hexachloro-1,3-butadiene	225	223, 227	3
Naphthalene	128	127	3
n-Butylbenzene	91	92, 134	3
n-Propylbenzene	120	91	3
p-Isopropyltoluene	119	134, 91	3
sec-Butylbenzene	105	134	3
tert-Butylbenzene	119	91, 134	3