

# SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

## W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA, INC 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

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### **1.0 INTRODUCTION**

On February 26, 2008, the United States Environmental Protection Agency (U.S. EPA) issued a Final Decision (U.S. EPA Final Decision) to implement soil vapor extraction (SVE) in unsaturated soil at the Former Chlorobenzene and Benzene Storage Area and Central Plant Process Area of the W.G. Krummrich facility that are impacted with volatile organic compounds (VOCs). Specifically, SVE will be applied to a total of six treatment areas: Big Mo, Former Chlorobenzene Storage Area, North Tank Farm, near Little Mo, Former Steamer Overhead Tank, and Former Benzene Pipeline (refer to Figure 1 for treatment area locations).

The U.S. EPA Final Decision requires a work plan to be developed for the implementation of SVE in all six treatment areas and establish the objectives and location of any pilot tests to be performed. A pilot test is necessary in order to develop an effective design and establish performance objectives for the full-scale SVE remedies. Therefore, this work plan focuses on the performance of a SVE pilot test in the Big Mo treatment area, which has the highest measured concentrations of VOCs of the six treatment areas and an unsaturated soil profile that is generally consistent with the other treatment areas. Upon completion of the SVE pilot test, a pilot test data evaluation report and a full-scale SVE implementation design work plan for the six treatment areas will be developed and submitted to the U.S. EPA for review and approval prior to the installation and operation of the full-scale system.

### **1.1 BACKGROUND**

The W.G. Krummrich facility (Site) is a 314-acre facility located at 500 Monsanto Avenue, Sauget, Illinois. The Site is approximately one mile east and in the floodplain of the Mississippi River. The Site is located in a heavily industrialized area, and has a history of approximately 100 years of industrial operations.

The Resource Conservation and Recovery Act (RCRA) Corrective Measures Study (CMS) Report dated August 27, 2004, included geological and VOC distribution data for the Site. Prior to the development of this work plan for pilot testing, pre-work plan data collection activities

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were conducted in each of the six treatment areas to aid in the design of the pilot testing program. The pre-work plan data collection event was conducted from June 23 to July 3, 2008, and included:

- a) soil borings to obtain more specific data on the geology of the unsaturated soils for each treatment area, and to understand the VOC distribution within the varying geologic layers observed at each treatment area; and
- b) vapor probe installation and testing to obtain preliminary data on the concentrations of the VOCs in the soil vapor and to obtain air permeability data for the significant (greater than 1 foot thickness) geologic layers observed.

Figure 1 shows the pre-work plan data collection soil borings and vapor probe installation locations. Data collected during the pre-work plan data collection event is presented in Appendix A (including soil boring logs, vapor probe installation logs, and soil and soil vapor samples analytical data). Appendix B provides the standard operating procedure (SOP) for collecting and evaluating air permeability data and summarizes the calculated air permeability values for each significant geologic layer. The collected data along with the historical data presented in the RCRA CMS Report were used to develop a conceptual model of the treatment areas, as presented in sub-section 1.2. The conceptual model is used as the basis for the pilot test design.

### **1.2 CONCEPTUAL MODEL OF THE TREATMENT AREAS**

Generally, the unsaturated natural soils across the treatment areas include silty sand layers measured from ground surface to approximately 15 feet below grade (bg) with an intermediate silty clay layer ranging in thickness from less than a foot to six feet at approximately 8 feet bg. Overlying fill is present in some areas at thicknesses up to 10 feet. Depth to groundwater at the Site typically ranges from less than 10 feet to 20 feet bg; however, depth to water is highly variable and depends upon Mississippi River stage. A representative geological cross-section of the Big Mo treatment area is presented in Figure 2 (geological cross-sections for each of the other five treatment areas are provided in Appendix A).

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During the pre-work plan data collection, field screening of continuous soil cores [using a photoionization detector (PID)] showed that VOC impacts occur generally throughout the unsaturated soils (refer to boring logs in Appendix A). Field observations indicated a petroleum sheen impacting a subset of soils collected from the lower silty sand layer (SB-A01, SB-A03, SB-A05, SB-C01, and SB-E01), which indicates the potential for light non-aqueous phase liquid (LNAPL) to exist within the soil pores in the Big Mo area. Table 1 summarizes the soil analytical results, and presents the VOCs and their maximum concentrations detected within each treatment area. Of the VOCs detected, benzene and chlorobenzene are measured at relatively high concentrations in the treatment areas [up to 19,000,000 micrograms per kilogram (ug/kg)]. These compounds, in addition to ethylbenzene, xylenes, toluene, and 2-hexanone (methyl butyl ketone or MBK), have relatively high vapor pressures, and therefore, are amenable to removal by SVE.

Dichlorobenzenes are measured at significantly lower concentrations (up to 10,000 ug/kg). The vapor pressures of dichlorobenzenes are approximately an order of magnitude lower than that of chlorobenzene, which makes them amenable to SVE, albeit at a much lower removal rate.

The presence of VOCs in the lower permeability (silty clay) layer within the higher permeability (silty sand) layers in the treatment areas will pose an engineering challenge to effective mass removal from the lower permeability layer by SVE. The higher permeability silty sand layers will provide preferential pathways for the induced airflow with proportionally lesser airflow occurring in the lower permeability silty clay layers. The preferential airflow will cause a significant reduction in the rate of VOC mass removal from the lower permeability layers. VOC mass removal from the low permeability layers will likely be mass transfer limited, and will likely be dominated by diffusion from the lower permeability layer into the higher permeability layers through which the majority of airflow will be occurring. Therefore, the permeability differences between the geologic layers, the placement of SVE well screens and possible use of air injection wells are important parameters to evaluate from the pilot test to optimize system performance, determine full-scale system design parameters, and to understand realistic soil clean-up expectations from SVE operation as a function of time.

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SVE systems typically achieve the majority of VOC mass removal from soils within two to three years of operation. Following the initial period of rapid mass removal, the remaining mass removal rates become asymptotic, typically based on mass transfer limitations in the subsurface.

While both aqueous solubilities and Henry's Law constants of the VOCs can impact removal rates via SVE, the effect is generally seen at lower soil concentrations where the levels within the soil moisture are at or below solubility. Considering the three month duration (Section 2.2.2) of the SVE pilot testing and the elevated COC concentrations in the subsurface, it is unlikely that this effect will be dominant. However, the pilot test data evaluation report will contain an evaluation of SVE remedial time-frames and the potential effect of COC solubilities and Henry's Law constants relative to other Site specific factors.

### **1.3 PILOT TEST OBJECTIVES**

Based on U.S. EPA Final Decision and the above conceptual model of the treatment areas, the following objectives have been identified for the SVE pilot test.

• Develop a SVE system design to effectively treat the unsaturated soils (i.e., fill, upper silty sand, intermediate silty clay and lower silty sand) and determine the expectations for full-scale SVE system operation as a function of time: While it is expected that an appropriately designed SVE system will be effective in removal of VOCs from the higher permeability soil layers, as discussed in Section 1.2, the intermediate silty clay layer between the two silty sand layers poses engineering challenges that will affect SVE performance. The pilot test is designed to provide information to allow an evaluation of the treatment of each of the geological layers and to predict performance over time in both the higher and lower permeability soils. Remedial objectives for the full-scale SVE treatment systems will be developed based on the results of the pilot test. Depending on predicted performance of the full-scale SVE systems, remedial objectives will either be:

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- mass reduction [for geologic units with mass transfer rates too low to allow Illinois' Tiered Approach to Corrective Action Objectives (TACO) Tier III criteria to be achieved within a two to three year time period],
- TACO Tier III criteria (for geologic units with mass transfer rates high enough to achieve the criteria within a two to three year time period), or
- o a combination of these two performance measures.
- Develop full-scale SVE design and operation parameters: The pilot test is designed to collect data to determine a number of necessary full-scale design and operational parameters, such as effective radius of influence (ROI), vapor extraction/air injection strategy, achievable airflow and vacuum/pressures, soil vapor velocities, process equipment requirements, VOCs in soil vapor discharge and vapor-phase VOC treatment requirements prior to discharge to the atmosphere, and water handling requirements.



### 2.0 PILOT TEST APPROACH

### 2.1 PILOT TEST LOCATION

The data presented in Table 1 shows that the highest measured concentrations of VOCs are in the Big Mo treatment area. Further, the geology in Big Mo is generally consistent with the other treatment areas. Therefore, the Big Mo treatment area is selected for performance of the pilot test. Table 2 presents the soil analytical VOC data for samples collected from the unsaturated soils in Big Mo. The data shows that borings S0607 and SB-A01 are the most impacted locations, and therefore, an area surrounding these two borings is selected for the SVE pilot test (refer to Figure 1 for soil boring locations).

Figure 2 presents a geologic cross-section of the Big Mo treatment area. The unsaturated soils profile in Big Mo is generally consistent with the other treatment areas. Based on the cross-section, a sandy fill with gravel exists from the surface gravel cover to 4 to 6 feet bg, an upper silty sand layer is present under the fill from approximately 6 to 10 feet bg, underlain by an intermediate silty clay layer ranging in thickness from less than a foot to 4 feet. Underneath the silty clay layer, a lower silty sand layer is present to a depth of approximately 16 feet bg. The lower silty sand layer has an interbedded clay lens/thin layer.

Depth to groundwater was difficult to ascertain during the pre-work plan data collection event due to unusually heavy rain and flooding in the region that resulted in a significant amount of groundwater in the treatment areas. Historical data suggests that the depth to groundwater in Big Mo is approximately 15 feet bg.

### 2.2 PILOT TEST DESIGN

### 2.2.1 DESIGN BASIS

A key parameter in SVE system design is the relative air permeabilities of the soil layers within, and of the surface cover over, the proposed treatment area. The relative air permeabilities of each of the soil layers and surface cover dictate the achievable airflow through the layers and the effective ROI for a given vacuum applied to a SVE well. The following average relative air



permeabilities were determined for the soil layers within the treatment areas based on the data collected during the pre-work plan data collection event (details provided in Appendix B):

- Sandy fill layer<sup>1</sup>  $1.9 \times 10^{-8}$  centimeter squared (cm<sup>2</sup>)<sup>2</sup>
- Upper silty sand layer<sup>1</sup>  $1.8 \times 10^{-8} \text{ cm}^2$
- Intermediate silty clay layer<sup>3</sup>  $3.9 \times 10^{-9} \text{ cm}^2$
- Lower silty sand layer<sup>1</sup>  $2.1 \times 10^{-8} \text{ cm}^2$

The fill, upper silty sand and lower silty sand layers have relatively higher permeabilities compared to that of the intermediate clay layer. Based on the above-determined relative air permeabilities, and considering that it would require between approximately 500 to 1,000 pore volume exchanges with clean air per year for aggressive treatment of the impacted soils by SVE, an effective ROI of between 10 to 15 feet is calculated for the higher permeability layers (fill and upper and lower silty sand layers) based on the above-mentioned air permeability data and preliminary airflow modeling using 2-D analytical model<sup>4</sup>. Similarly, a relatively small effective ROI of 2 to 3 feet is calculated for the lower permeability intermediate silty clay layer. It is noted that these ROI values are used for the design of the SVE pilot test, and data collected during the pilot test will be used in the full-scale SVE design.

### 2.2.2 TECHNICAL APPROACH

The SVE testing will be performed by applying vacuum to SVE wells to extract soil vapor, and is designed to target both the higher and lower permeability layers observed in the unsaturated soils. Air availability in the lower silty sand layer will likely be limited due to the overlying intermediate silty clay layer. Therefore, air injection wells are proposed to supply and promote

<sup>&</sup>lt;sup>1</sup> Presence of groundwater during testing likely influenced the permeability data, and the permeabilities of the fill, upper silty sand and lower silty sand layers may be higher under drier conditions.

<sup>&</sup>lt;sup>2</sup> The relationship between air permeability and hydraulic conductivity is a factor of approximately  $10^{-5}$ , i.e., an air permeability of 1 x  $10^{-8}$  cm<sup>2</sup> is approximately equal to a hydraulic conductivity of  $10^{-3}$  cm/second for comparison of soil type for water flow.

<sup>&</sup>lt;sup>3</sup> Per field observations, the intermediate silty clay layer is highly plastic and low-permeable. However, the air permeability results show higher than expected permeability for this layer. This higher estimated permeability may be due to short-circuiting between the silty clay layer and the adjacent silty sand layers.

<sup>&</sup>lt;sup>4</sup> Baehr, A.L. and Joss, C.J. 1995. "An updated model of induced air flow in the unsaturated zone," Water Resources Research vol 31, n 2, pp 417-421.



airflow in the lower silty sand layer. Air injection wells will also help direct air in the upper silty sand layer due to the presence of the overlying fill. The air injection wells also aid in expediting the removal of any water in the treatment area. The air injection wells will be constructed similarly to the SVE wells, and air injection will be accomplished by connecting the injection wells to a clean air supply system (discussed in Section 3 of the work plan).

During the initial two-week period of operations of the pilot test, various well configurations will be tested to develop the data (primarily airflow, soil vapor discharge concentrations, and vacuum/pressure distribution data) required to develop the design parameters for an effective SVE treatment system to remediate the treatment areas (refer to Section 4.1 for details). The need for air injection for effective treatment by SVE will also be evaluated during the initial phase of the pilot test.

After the initial design parameter evaluation phase, the pilot test will be run for an extended period of time (three months) at a selected vapor extraction/air injection well configuration to develop performance data on VOC mass removal in the target geological layers within the test area (refer to Section 4.2 for details). The performance data will be collected to project the performance of the proposed full-scale SVE system over time. Baseline, intermediate and postpilot test soil sampling will be completed as part of the performance monitoring for the pilot test, and extracted soil vapor concentrations will be used to estimate the mass removal from the operation of the pilot test. Soil samples will also be collected from within the pilot test area to determine the mass removal rate in the fill and upper silty sand layers, intermediate silty clay layer and lower silty sand layer. The locations for the soil samples (discussed in Section 4.3.1) are selected to provide data in areas where, over the duration of the pilot test, VOC mass removal is expected to be good (e.g., near air injection wells), and where mass removal is not expected to be good (e.g., in the intermediate silty clay layer). The three month performance evaluation testing period was estimated based on preliminary calculations from the pre-work plan data collection results and experience at other sites to provide meaningful data for predicting fullscale SVE system performance.

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Two blowers will be used for the pilot testing; one for the higher permeability layers (fill, upper silty sand and lower silty sand) and the other for the lower permeability layer (intermediate silty clay). Vacuum/pressure and corresponding airflow rates at the extraction and injection wells will be documented (refer to Section 4.3 for monitoring details). Vacuum/pressure measurements will also be collected at surrounding monitoring points (e.g., vapor probes) to assess the distribution of vacuum/pressure in the test area under the various test conditions. The testing will also include soil vapor monitoring and analysis to assess VOC removal rates.

Ideally, the pilot test should be conducted during a relatively dry (Fall) season with seasonally low water table conditions for effective performance of SVE, to reduce interference of groundwater on the pilot testing, and for the effectiveness of the data collected; however, the pilot test process components are designed to accommodate high groundwater level conditions. Further, asphalt paving, or equivalent, will be installed over the pilot test area to prevent rain water from infiltrating into the treatment area and influencing the effectiveness and performance of the pilot test.

### 2.2.3 PILOT TEST LAYOUT

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Figure 3 shows the proposed pilot test layout. The layout includes a total of 22 SVE wells to target the higher permeability soil layers (11 in the fill and upper silty sand layers and 11 in the lower silty sand layer)<sup>5</sup> within a pilot test area of 60 feet long by 80 feet wide. The spacing between the SVE wells targeting the higher permeability layers ranges from 10 feet to 30 feet. The range of spacing is to allow evaluation of effective ROI during the pilot testing. Five SVE wells (SVE-04D, SVE-05D, SVE-06D, SVE-07D, SVE-08D) in the lower silty sand layer and two SVE wells (SVE-05S, SVE-07S) in the fill and upper silty sand layer will also be manifolded to allow use as both air injection or vapor extraction wells (refer to Figure 3 for locations). The remaining 15 wells in the high permeability layers will be used as vapor extraction only wells. This configuration allows for testing a range of ROI/well spacing, as well

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<sup>&</sup>lt;sup>5</sup> Wells SVE-01 through -11 will be nested SVE wells and will contain two well screens (shallow – S and deep – D) and casings in one drilled boring. As shown in Figure 4, one screen (S) will be installed in the fill and upper silty sand layers, and the second screen (D) will be installed in the lower silty sand layer.

as the impacts of air injection on system design and performance. The pilot testing procedures are discussed in Section 4.0.

Figure 3 also shows a 10 feet long by 10 feet wide area for SVE pilot testing in the intermediate silty clay layer. A total of 4 SVE wells will be installed in the 10 foot by 10 foot area at a spacing of 5 feet.

Vapor probes installed during the pre-work plan data collection event will be used in the pilot test performance monitoring program. Additionally, 19 vapor points (8 in the fill and upper silty sand layers, 8 in the lower silty sand layer and 3 in the intermediate silty clay layer – refer to Figure 3 for locations) will be installed for performance monitoring.

### 2.2.4 PILOT TEST DESIGN/OPERATIONAL PARAMETERS

The SVE pilot test design/operational parameters are presented in Table 3. The design airflow rate per well in the upper and lower high permeability soil layers is calculated to be 8 to 15 standard cubic feet per minute (scfm) with a vacuum at the individual wellheads of between 5 and 12 inches of mercury (Hg). The 2-D analytical model (refer to Section 2.2.1) was used with the air permeability data collected during the pre-work plan data collection event to calculate the expected flow rates and vacuums. Similarly, the design airflow rate per well in the intermediate clay layer is calculated to be 1 to 2.5 scfm with a vacuum at the individual wellheads of approximately 23 inches of Hg. Air injection pressure will be regulated to avoid fracturing the geology and creating preferential pathways for air movement.

Actual operational parameters (i.e., flow and vacuum/pressure at individual wellheads) will be determined in the field during the design parameter evaluation phase of the SVE pilot test. For the extended SVE performance evaluation phase of testing, five SVE wells (SVE-04D, SVE-05D, SVE-06D, SVE-07D, SVE-08D) in the lower silty sand layer and two SVE wells (SVE-05S, SVE-07S) in the fill and upper silty sand layer will be operated as air injection wells, and the remaining 19 SVE wells will run as vapor extraction wells (six in the lower silty sand layer, nine in the fill and upper silty sand layer, and four in the intermediate silty clay layer). Section



4.2 provides details of the performance evaluation phase of the pilot testing. This configuration would achieve a total SVE airflow rate ranging from 124 to 235 scfm, and total air injection flow ranging from 56 to 105 scfm (refer to Table 3).

### 2.3 DATA QUALITY OBJECTIVES

A detailed presentation of the project data quality objectives (DQOs), including measurement parameters, field measurement equipment, analytical methods and intended use of the data is provided in the project Quality Assurance Project Plan (QAPP) provided as Appendix C.

Specific objectives for each data set are established to develop sampling protocols, applicable documentation, sample handling procedures, measurement system procedures and quality assurance (QA)/quality control (QC) procedures that will be used during the pilot test activities. Each DQO may require different levels of data quality depending on the end uses of that data. Qualitative and/or quantitative statements specify the quality of data required for each data collection activity.

Work plan specific QA/QC samples, media-specific project quality objectives, and data validation specifics are provided in QAPP. DQOs are used to design a field pilot test program that allows for the proper collection of data in order to satisfy the objectives of the testing.



### 3.0 PILOT TESTING COMPONENTS

The system components presented in the following sub-sections are selected to meet the pilot test design presented in Section 2.2. The primary pilot testing components include:

- System wells and manifold,
- System process equipment, and
- Low permeability rain cover.

### **3.1 SYSTEM WELLS AND MANIFOLD**

System wells include SVE wells for vapor extraction and/or air injection, the existing vapor probes installed in the test area during the pre-work plan data collection activities (nested vapor probes VP-A01, VP-A02 and VP-A03) and the additional vapor points to be installed for performance monitoring, as described below in Section 3.1.3. Figure 4 shows construction details for the typical SVE well and vapor point installations for the test area. The installation logs for the existing vapor probes are included in Appendix A.

### 3.1.1 SVE/Air Injection Wells

Figure 3 shows the proposed SVE and/or air injection well locations. Wells SVE-01 through -11 will be nested SVE wells and will contain two well screens (shallow – S and deep – D) and casings in one drilled boring. As shown in Figure 4, one screen (S) will be installed in the fill and upper silty sand layers, and the second screen (D) will be installed in the lower silty sand layer. The shallow screens with sand pack targeting the fill and upper silty sand layers will be located from the top of the intermediate silty clay layer to 5 feet below ground surface (approximately 5-foot long), whereas the deep screens with sand pack targeting the lower silty sand layer will be located from the bottom of the intermediate silty clay to about 2 feet above the water table (approximately 1.5-foot long). Wells SVE-04D, SVE-05S/D, SVE-06D, SVE-07S/D and SVE-08D will be fitted to operate as either SVE or air injection wells.

The intermediate silty clay layer will be targeted using individual SVE wells (SVE-12 through 15) installed at approximately 5 foot on-center spacing. These wells will be installed in the



intermediate silty clay layer with a 0.5 to a 1-foot screen interval (refer to Figure 4). Please note that the SVE well depths and screen intervals presented in this sub-section and Figure 4 are subject to change based on field conditions observed during the well installation (e.g., observed thickness of target geologic layer).

The SVE and/or air injection wells will be two-inch in diameter with galvanized steel well screen and riser. All wells will be connected to above grade piping (Figure 5) and fitted with wellhead controls (as shown in Figure 6). All SVE and/or air injection wells will be installed by an Illinois licensed driller using sonic drilling techniques. Soil cuttings will be drummed and staged on-site for appropriate disposal.

### 3.1.2 EXISTING VAPOR PROBES

Existing vapor probes VP-A01, VP-A02 and VP-A03 will be used during the SVE pilot testing to monitor vacuum/pressure distribution and SVE performance (refer to Figure 3 for the existing vapor probe locations and Appendix A for the existing vapor probe installation logs). Vapor probe VP-A01 consists of a cluster of three probes located within the fill/upper silty sand and lower silty sand layers (nested) and the intermediate silty clay layer. Vapor probe VP-A02 is a single probe located in the upper silty sand layer. Vapor probe VP-A03 consists of two nested vapor probes located within the fill and lower silty sand layers.

### 3.1.3 VAPOR POINTS

As shown in Figure 3, an additional 19 vapor points (eight in each of the fill/upper silty sand and the lower silty sand layers, and three in the intermediate silty clay layer) will be installed within the pilot test area to monitor vacuum/pressure distribution and SVE performance. The additional vapor points will be GeoProbe® permanent implants that are 1/2-inch in diameter stainless steel with a 6-inch screen. Refer to Figure 4 for typical construction details. The implants will be installed using direct push drilling techniques.



### 3.1.4 System Manifold

Figure 5 shows the pilot test manifold layout. Four inch diameter mainlines will be used for the fill, upper silty sand and lower silty sand layers, and a two inch diameter mainline will be used for the intermediate silty clay layer and the air injection manifold. The system manifold will consist of four aboveground main manifold lines that will run from the process equipment shed/container. Individual branches will run from the mainlines to each SVE/air injection wellhead with associated wellhead controls, as shown in Figure 6. Three of the main manifold lines will be connected to SVE wells in the fill/upper silty sand, lower silty sand and intermediate silty clay layers. The fourth main manifold line will be used for air injection, and will be connected to the wells (SVE-04D, SVE-05S/D, SVE-06D, SVE-07S/D, SVE-08D) as discussed in Section 3.1.1.

Wellhead controls will be located at each SVE and/or air injection well, and will include flow control gate valve, flow meter, and pressure/vacuum gauge. At the seven well screens that will also be used as air injection wells, "T" fittings with two gate valves will be used at the wellheads to allow the well operation to switch from vacuum to pressure injection as required.

### **3.2 SYSTEM PROCESS EQUIPMENT**

The SVE and air injection process equipment and associated control panel will be housed in an equipment shed/container (approximate dimensions of 40 feet by 10 feet) to be located in the vicinity of the pilot test area (refer to Figure 5 for approximate location). The process equipment will be fabricated offsite. Figure 6 shows a process and instrumentation (P&ID) diagram of the process equipment. The following sub-sections identify and provide a brief description of the main components of the process equipment.

### 3.2.1 SVE PROCESS EQUIPMENT

The SVE process equipment consists of two vacuum blowers, air intake gate valves, oil water separator, air-moisture separators, in-line air particulate filters, equalization tank, shallow tray air stripper, and a thermal oxidation (THERMOX) unit. Two separate blowers and associated process equipment are proposed to effectively evaluate the fill, upper silty sand, lower silty sand



and the intermediate silty clay geologic layers, respectively (refer to Figure 6). The SVE main manifold lines associated with the fill, upper silty sand and lower silty sand layers will flow through gate valves to the associated process equipment. The SVE mainline from the intermediate silty clay layer will flow through a separate gate valve and separate associated process equipment.

The major SVE process equipment consists of the following components:

- <u>Air-Moisture Separator System</u> Each soil vapor stream (from the main manifold lines) will have an air-moisture separator to remove entrained water from the air stream before it enters the vacuum blower. The air-moisture separator system will have adequate volume capacity and a three-switch float control. The air-moisture separators will be equipped to drain manually or through the use of a transfer pump.
- <u>Oil-Water Seperator</u> A oil-water separation tank will be installed to receive fluids from the water separation tank. Any separated and stored LNAPL will be separated prior to discharging process water to the equalization tank.
- <u>In-Line Air Particulate Filter</u> A high efficiency particulate filter will be used for each soil vapor stream prior to the downstream blower to remove fine particle solids that could damage the blowers.
- <u>Vacuum Relief Valve</u> A vacuum relief valve will be installed for each soil vapor stream to prevent excessive system vacuum build-up.
- <u>Vacuum Blower</u> Two separate vacuum blowers will be used for the fill, upper silty sand and lower silty sand and for the intermediate silty clay layers. The soil vapor stream from the fill and silty sand layers will be extracted using a positive displacement blower with a capacity of 275 scfm at 16 inches of mercury vacuum at the inlet. The soil vapor stream from the silty clay layer will be equipped with a rotary vane pump with a capacity of 15 scfm at 24 inches of mercury vacuum at the inlet.
- <u>Equalization Tank</u> An equalization tank will used to store water generated by the water separation tank and air moisture separators and provide a steady flow of water to the air stripper

- <u>Air Stripper</u> A shallow tray air stripper fed from the equalization tank. VOC impacted air from the air stripper will be treated via the THERMOX unit.
- <u>THERMOX Unit</u> The air stream will be treated through a THERMOX unit with a design capacity of 450 scfm to handle the system total flow and VOC load. Pressure gauges and sample ports located upstream and downstream of the THERMOX unit will be installed to monitor pressure drop across the units and to collect pre-treatment and post-treatment vapor samples, respectively.

### 3.2.2 AIR INJECTION PROCESS EQUIPMENT

The air injection process equipment will be located in the equipment shed/container with an intake pipe to the air compressor located externally. After the process equipment, the airflows through the air injection main manifold to the appropriate wellheads (SVE-04D, SVE-05S/D, SVE-06D, SVE-07S/D, SVE-08D). The major components of the air injection process equipment are described below and are shown in the P&ID presented in Figure 6.

- <u>In-Line Air Particulate Filter</u> A high efficiency particulate filter will be used to remove fine particle solids to prevent damage to compressor.
- <u>Air Compressor</u> Rotary vane air compressor with a capacity of 125 scfm at 13 pounds per square inch (psi) at the outlet line will be used.
- <u>Pressure Relief Valve</u> A pressure relief valve will be installed on the air injection line to prevent excessive pressure build-up in the system and to regulate injection pressure so as to avoid fracturing the geology, which would create preferential pathways for air movement.

### 3.2.3 CONTROL PANEL

A control panel will be housed in the equipment shed/container for the SVE and air injection systems. A programmable logic controller (PLC) will be used to control the systems operation. An auto-dialer telemetry system will be included to notify operator of alarm conditions (i.e., system shutdown). System control would be programmed so that air injection could only occur while vapor extraction is operating.



### **3.3 LOW PERMEABILITY RAIN COVER**

A low permeability cover was installed over the pilot test area in October 2008 to prevent the infiltration of rain water during the pilot testing. Asphalt paving will likely reduce the groundwater level and improve the airflow through the unsaturated zone soils.



#### 4.0 SVE PILOT TESTING PROCEDURES

The pilot test startup will begin after the installation of the pilot test components discussed in Section 3.0, and following the baseline soil and soil vapor sampling discussed in Section 4.3. Upon startup, initial system operation will involve removal of recoverable soil moisture from the test area soils. Subsequently, the pilot testing will begin, and the test will be conducted in two phases as discussed in Section 2.2.2: i) design parameter (ROI and achievable airflow) evaluation testing conducted over a period of two weeks, followed by ii) performance (mass removal) evaluation testing conducted over a period of three months. The following sub-sections describe the design parameter and performance evaluation testing, and associated monitoring program.

### 4.1 DESIGN PARAMETER EVALUATION TESTING

The design parameter evaluation testing will be performed primarily to evaluate air/vapor flow characteristics (i.e., ROI, well spacing, achievable airflow and vacuum/pressure) in each of the unsaturated geological layers. Individual wells and various combinations of well configurations will be tested to develop the data for the design of an aggressive SVE treatment system to remediate the treatment areas. Air injection to promote airflow in the higher permeability layers to facilitate SVE will also be tested as part of the design parameter evaluation testing. This phase of testing will be conducted over a period of two weeks.

### 4.1.1 INDIVIDUAL WELL TESTING

This testing will be conducted at approximately half of the SVE wells installed for the pilot test. A total of 12 SVE wells [five in the fill and upper silty sand layer (SVE-02S, SVE-04S, SVE-06S, SVE-08S, SVE-10S); five in the lower silty sand layer (SVE-02D, SVE-04D, SVE-06D, SVE-08D, SVE-10D); and two in the intermediate silty clay layer (SVE-12, SVE-14)] will be tested by applying vacuum at each test well separately using the system process equipment while measuring the changes in vacuum and the corresponding vapor flow rate at the test well. A total of four SVE wells [two in the fill and upper silty sand layer (SVE-05S, SVE-07S); and two in the lower silty sand layer (SVE-05D, SVE-07D)] connected to the air injection manifold will also be



tested by applying pressure using the system process equipment while measuring the changes in pressure and corresponding airflow at the test well. Vacuum/pressure measurements will also be collected at the nearby SVE wells and vapor points/probes in order to monitor the vacuum/pressure changes and distribution in the subsurface.

Testing is to be performed at a minimum of two different flow rates at the selected wells. The well will be tested first at the maximum airflow rate that can be achieved at the vacuum/pressure capacity of the process equipment. The wellhead vacuum/pressure will then be set to create an intermediate airflow rate (e.g., approximately half of the maximum flow observed at the high vacuum/pressure condition). Each test will run until vacuum/pressure measurements at the nearby wells/vapor probes/points have stabilized, anticipated to be approximately one to two hours. In addition to barometric pressure and ambient temperature readings, the following data will be collected during this phase of testing (also summarized in Table 4 and Section 4.3):

- Baseline vacuum/pressure at the test well and nearby wells/vapor probes/points,
- Vapor/air flow rate and vacuum/pressure at the test well,
- Vacuum/pressure and groundwater elevations at the nearby wells/vapor points/probes,
- Vacuum/pressure, airflow rate and temperature at the system process equipment,
- Soil vapor concentrations of VOCs at the test well (only for vacuum testing),
- Pre- and post-treatment effluent air sampling, and
- Groundwater elevation monitoring at PSMW-05.

### 4.1.2 Well-Field Configurations & Flow Optimization Testing

After conducting tests at individual wells, ROI and airflow testing will be performed by applying vacuum and pressure at multiple wells in the following well-field configurations to confirm the full-scale SVE design parameters for the fill and upper and lower silty sand layers, and to determine effective flow conditions and well-field configuration for the performance evaluation phase of pilot testing. During the testing of different well-field configurations, vapor flow rates and vacuum/pressure distribution in the test wells and surrounding vapor probes/points and SVE wells will be monitored. These well-field configurations will be tested for both the fill and upper



silty sand layers, and the lower silty sand layer separately. Figure 7 presents the following well-field configurations for the fill and silty sand layers:

- SVE configurations designed to examine varying radii of influence (ROI):
  - <u>Configuration 1</u>: Operating wells SVE-01, -02 and -03 as extraction wells 15foot (ft) ROI;
  - <u>Configuration 2</u>: Operating wells SVE-04 and -08 as extraction wells 20-ft ROI;
  - <u>Configuration 3</u>: Operating wells SVE-04 and -07 as extraction wells 15-ft ROI; and
  - <u>Configuration 4</u>: Operating wells SVE-04, -06 and -08 as extraction wells 10-ft ROI.
- SVE configurations designed to examine varying radii of influence under subsurface air injection conditions:
  - <u>Configuration 5</u>: Operating wells SVE-04 and -08 as extraction wells, and SVE-06 as an injection well;
  - <u>Configuration 6</u>: Operating wells SVE-02, -04, -08 and -10 as extraction wells, and SVE-06 as an injection well; and
  - <u>Configuration 7</u>: Operating wells SVE-04, -06 and -08 as extraction wells, and SVE-05 and -07 as a injection wells.

Subsequently, the following additional well-field configurations for the lower silty sand layer will be tested to estimate the preferred configuration for the performance evaluation phase of the pilot test. Figure 8 presents the following well-field configurations for the lower silty sand layer:

- SVE configurations designed to examine varying radii of influence:
  - <u>Configuration 8</u>: Operating all wells in the lower silty sand layer as extraction wells;
- SVE configurations designed to examine varying radii of influence under subsurface air injection conditions:
  - <u>Configuration 9</u>: Operating well SVE-06D as an injection well and the remaining wells in the lower silty sand layer as extraction wells;
  - <u>Configuration 10</u>: Operating wells SVE-04D, SVE-06D and SVE-08D as injection wells and the remaining wells in the lower silty sand layer as extraction wells; and



• <u>Configuration 11</u>: Operating wells SVE-04D, SVE-05D, SVE-06D, SVE-07D and SVE-08D as injection wells and the remaining wells in the lower silty sand layer as extraction wells.

Similarly, the following additional well-field configurations for the fill and upper silty sand layers will be tested to estimate the preferred configuration for the performance evaluation phase of testing:

- <u>Configuration 12</u>: Operating all wells in the fill and upper silty sand layers as extraction wells; and
- <u>Configuration 13</u>: Operating wells SVE-05S and SVE-07S as injection wells and the remaining wells in the fill and upper silty sand layers as extraction wells.

Each of the well-field configuration test will run for approximately two to three hours. Based on the results of the well-field configuration/flow option testing, an appropriate well-field configuration (the one that results in the maximum total vapor flow rate from the test area and the highest mass removal rate) will be selected for the SVE performance evaluation testing.

Additional well-field configuration testing will not be conducted for SVE wells in the intermediate silty clay layer as all four SVE wells in this layer (SVE-12, SVE-13, SVE-14 and SVE-15) will be operated as vacuum wells during the SVE performance evaluation phase of testing.

### **4.2 PERFORMANCE EVALUATION TESTING**

Upon completion of the design parameter evaluation testing phase, the extended SVE performance evaluation testing will begin using the optimal vapor extraction/air injection well-field configuration determined from the well-field configuration/flow testing. The system will run under this configuration for a period of three months to assess VOC removal rates for the system as a whole, and to evaluate the treatability of each of the unsaturated geological layers.

Monitoring will be performed to collect data (refer to Table 4 and Section 4.3) to estimate VOC mass removal rates in the vapor phase and to estimate the reduction in soil VOC mass in the



higher and lower permeability geological layers in the pilot test area. The data will be used to project the performance of the proposed full-scale SVE system in the six treatment areas as a function of time.

### **4.3 MONITORING**

During the pilot testing, monitoring will be conducted to collect data to allow the design of an aggressive SVE system for the treatment areas, to ensure proper operation of the pilot testing, to evaluate the VOC mass removal from the test area, and to determine the overall effectiveness of the system. A summary of the pilot test sampling and monitoring parameters and the frequency of sampling is provided in Table 4. Specifically, the monitoring will include collection of soil and soil vapor samples for analysis, gauging of operation parameters (e.g., vacuum/pressure monitoring, airflow rate) and sampling of air discharge stream for compliance with air discharge criteria. Detailed procedures for sample collection and analysis are provided in the Sampling and Analysis Plan (SAP) provided as Appendix D of this work plan.

### 4.3.1 SOIL SAMPLING

Soil sampling will be conducted as one measure to assess the effectiveness of SVE in treating the various geologic layers that constitute the unsaturated soils in the test area. Prior to the pilot test startup, a baseline soil sampling event will be conducted to establish baseline conditions. Two additional soil sampling events, one at approximately the mid-point of operation of the 3-month long SVE performance evaluation testing, and the other within a week of the conclusion of the pilot testing is proposed to evaluate the degree and rate of treatment in each geologic layer over the period of the pilot test performance. The soil samples will be sent to Test America Laboratories of Savannah, GA, for VOC analysis via U.S. EPA Method 8260B. Soil sampling and analysis procedures are discussed in detail in the SAP (Appendix D).

A total of 26 soil samples (one per each SVE well screen - 11 from the fill and upper silty sand layers, 4 from the intermediate silty clay layer and 11 from the lower silty sand layer) will be collected from a total of 15 borings during the baseline sampling event (refer to Figure 9). The location of these 15 borings (26 soil samples) were selected at varying distances from the



SVE/air injection wells with an emphasis on soils that are expected to have significant pore volume exchanges with clean air and therefore, a higher level of treatment over the duration of the pilot testing (e.g., near air injection wells). A few borings/sampling locations were also selected to focus on soils that are not likely to be significantly treated over the duration of the pilot testing (e.g., in the intermediate silty clay layer).

#### 4.3.2 SOIL VAPOR SAMPLING

Soil vapor sampling is proposed to assess the changes in soil vapor concentrations in the subsurface over the period of pilot test performance, and to estimate the vapor-phase VOC mass removal from the pilot test area vicinity. Prior to the pilot test startup, baseline soil vapor samples will be collected from all SVE wells and vapor probes/points to establish baseline conditions. The baseline soil vapor samples will be collected using an air-sampling pump in Tedlar<sup>™</sup> bags, and screened in the field using a PID capable of measuring down to 1.0 part per billion (ppb). Additionally, one soil vapor sample with the highest PID reading from SVE wells and vapor probes/points in each geological layer (i.e., three vapor samples from SVE wells and three vapor samples from vapor probes/points) will be sent to Test America Laboratories of Knoxville, TN, for VOC laboratory analysis via U.S. EPA Method TO-15.

Additional soil vapor monitoring will be conducted throughout the pilot test period as proposed in Table 4 using the sampling protocols provided in the SAP, attached as Appendix D. During the operation of the SVE pilot test, samples of extracted soil vapors from individual SVE mainlines and the combined SVE mainline will be collected for field PID screening and analytical laboratory analysis (refer to Table 4 for the frequency and number of samples) to monitor changes in VOC concentrations over the period of the pilot test performance, and to calculate the total mass of vapor-phase VOCs removed during the pilot test.

As shown in Table 4, additional soil vapor samples will be collected from the vapor probes/points during the SVE performance evaluation phase of the pilot test to evaluate SVE performance.



### 4.3.3 **OPERATION PARAMETER MONITORING**

SVE system operation parameters will be monitored during the design parameter evaluation and SVE performance evaluation phases of testing, primarily to collect data to develop the design of the full-scale SVE system, and to ensure effective system operation during pilot testing, respectively. The operation parameters include vapor/air flow rates at individual SVE wells, LNAPL removal rates, vacuum/pressure readings at vapor probes/points, and temperature measurements. Additionally, gauging of water elevation in available SVE wells (those not in use for vapor extraction/air injection) and vapor probes and monitoring of the volume of extracted water/condensate will be conducted.

The Site water table is relatively shallow and areas of elevated groundwater were observed during the pre-work plan data collection event. Additionally, the operation of the SVE system may affect the water table elevation which is correlated to the local vacuum/pressure levels in the soil near the water table.

During the design parameter evaluation phase of testing, operation parameters will be monitored on an hourly basis for tests that last less than a day, and on a daily basis for tests that last more than a day. Operation parameters will be measured on a daily basis during the first week of the performance evaluation phase of testing, weekly during the first month of operation and biweekly thereafter (refer to Table 4).

Vapor/air flow rates at the test wells will be measured using either a Pitot Tube<sup>™</sup> flow sensor combined with a differential pressure gauge or a thermal anemometer connected to the sample port on the wellhead/manifold. These devices are acceptable for measuring flow velocities of 1,000 feet/minute or greater (approximately 20 scfm in a 2 inch diameter pipe). For lower flow rates, a large rotometer or ERDCO orifice plate meter may be used to provide more accurate measurements. The devices will be installed according to the manufacturers' specifications. All flow rates will be corrected to standard temperature and ambient pressure conditions (i.e., scfm).



Vacuum gauges capable of reading up to 29 inches of mercury will be installed at each SVE wellhead and a pressure gauge capable of reading up to 15 pounds per square inch (psi) will be installed at each air injection wellhead (refer to Figure 6). These gauges will be used to monitor vacuum/pressure measurements at the test wells. Pressure and vacuum measurements will be collected at the vapor points/probes using a set of Magnehelic<sup>TM</sup> (or equivalent) gauges, capable of reading 0.005 to 150 inches of water. Tygon<sup>TM</sup> (or equivalent) tubing will be used to connect the pressure/vacuum gauge set to the appropriate monitoring ports. Pressure/vacuum gauges are available in a variety of ranges, and the same gauge can be used to measure either vacuum or pressure by switching inlet ports. Gauges are sealed and calibrated at the factory and will be re-zeroed before each test. Temperature of the vapor streams will be measured using thermometers installed at the wellhead and thermocouples installed in the manifolding associated with the process equipment (refer to Figure 6 for locations).

### 4.3.4 AIR PERMIT COMPLIANCE SAMPLING

The expected initial VOC vapor concentrations from the SVE system is approximately 3,300 parts per million by volume (ppmv) based on the maximum soil vapor concentration detected during the pre-work plan data collection (refer to Table 5). Based on the rate of VOC discharge during the pilot testing, it was determined that the SVE vapor stream should be treated with a THERMOX unit (target treatment efficiency of 99%) prior to discharge to atmosphere. Treatment with the THERMOX unit will ensure that VOC emissions from the system are within the limits specified in the air discharge permit.

As described in Section 4.6, an air operating permit will be required prior to the construction of the SVE pilot test system. Compliance sampling will be performed throughout the duration of system operation as required by the air discharge permit. Discharge vapors from the THERMOX unit will be collected for laboratory analysis for VOCs per U.S. EPA Method TO-15 to confirm compliance. Air emissions will be measured periodically (twice a week during design parameter evaluation and monthly during SVE performance evaluation) using a PID before and after the THERMOX unit for screening purposes to assess treatment efficiency.



### 4.4 HEALTH AND SAFETY

All work detailed in this work plan will be performed in accordance with the site-specific Health and Safety Plan (HASP – refer to Appendix E), which provides details on potential health and safety hazards, administrative and engineering controls, environmental monitoring and personal protection equipment required during field activities. The HASP will also ensure that work is performed in compliance with Occupational Safety and Health Administration (OSHA) regulations and guidelines (29 CFR 1910.120).

#### 4.5 WASTE DISPOSAL

Waste generated during the pilot test activities will include soil cuttings and decontamination water generated during drilling for well installation and soil sampling, debris and trash generated during the process equipment and manifold installation, and soil vapors and extracted water/condensate generated during the pilot testing.

All waste generated from the drilling activities (i.e., soil cuttings and decontamination water) will be containerized, stored in a location designated by Solutia and disposed off site in accordance with regulations. General trash and debris will be disposed of in accordance with facility practices. Soil vapors from SVE operation will be treated using a THERMOX unit (refer to Section 3.2) prior to discharge to atmosphere.

A significant amount of water from the test area may be extracted during the operation of the SVE system. The quantity of water generated will depend on the season when the pilot test is conducted (e.g., ideally, the pilot test should be conducted during the relatively dry season), amount of precipitation (rain fall) prior to and during the testing, and potential horizontal movement of water into the pilot test area after the installation of the low permeability rain cover. The approach for handling and disposal of the extracted water will depend on the quantity of water generated during the SVE pilot testing. If the water quantity is relatively small (a few to several hundred gallons), the water will be containerized and stored in a location designated by Solutia prior to disposal. If the water quantity is relatively large (a few thousand gallons or



more), the water will be treated on-site and discharged via plant sewers to the American Bottoms Regional Treatment facility [a publicly owned treatment works (POTW)].

#### **4.6 PERMIT REQUIREMENTS**

All applicable federal, state, local and Site permits required for the pilot testing activities including drilling, SVE well installation and construction and operation of an SVE system will be acquired prior to commencing the pilot test. This sub-section identifies the federal, state and local permits, required. Permits required from the W.G. Krummrich facility will be coordinated as appropriate with Solutia personnel.

### Air Permit: Construction Permit Application

Table 5 shows an estimate of the maximum potential vapor-phase VOC discharge from the SVE pilot test system. The maximum potential vapor-phase VOC discharge values are calculated using the maximum concentrations of each VOC in the soil vapor samples collected during the pre-work plan data collection event and the maximum design flow of the SVE pilot test system provided in Table 3. Further, to support a worst-case condition, it is assumed that the concentrations of the VOCs in soil vapor would remain constant throughout the pilot test.

An Air Operating Permit with the Illinois Environmental Protection Agency (IEPA) will be required prior to the construction of the SVE pilot test system. Based on the estimated maximum potential air emission from the SVE pilot system of 43 tons of hazardous air pollutants (HAP) per year (Table 5), the system is considered a "potential major source." The goal of the proposed SVE vapor discharge treatment using a THERMOX unit (refer to Section 3.2) is to reduce the emissions to less than 10 tons per year for a single HAP and less than 25 tons per year for total HAPs.

### Water Permit for Discharge to POTW

**US EPA ARCHIVE DOCUMENT** 

Water generated during the SVE pilot test will be treated on-site and discharged via Site sewers to the American Bottoms Regional Treatment Facility POTW. A permit from the POTW will be required.



### 4.7 DATA ANALYSIS

Data collected during the SVE pilot testing will be used to evaluate treatment of each of the higher and lower permeability geological layers in the unsaturated zone in order to develop performance expectations for implementation of full-scale SVE system for the six treatment areas as a function of time. As stated previously, SVE systems typically achieve the majority of VOC mass removal from soil within two to three years of operation. Remedial objectives (either mass removal, TACO Tier III criteria or a combination of the two) for the SVE system will be developed based on the pilot test results. Data collected during the pre-work plan data collection activities and pilot test will also be used to determine the optimal SVE system design to effectively remediate the six treatment areas to meet the remedial objectives. The following subsections outline how the data acquired during the pilot testing will be utilized for the above-mentioned evaluations and for the full-scale SVE system design.

### 4.7.1 EVALUATION OF SVE PERFORMANCE

The extracted soil vapor sampling data (refer to Section 4.3.4) and the baseline, intermediate and post-pilot test soil sampling data (refer to Section 4.3.1) collected during the SVE performance evaluation testing phase will be used to evaluate the treatability of each of the unsaturated geological layers present in the pilot test area.

As each geological layer has a separate SVE mainline that will be analyzed for VOC mass removal during the pilot testing, the extracted soil vapor sampling data will be used to calculate the total VOC mass removed during the operation of the pilot test, from each of the geological layers in the test area.

The baseline soil sampling data will be used to estimate the initial VOC mass present in each of the geological layers within the pilot test area. Similarly, the intermediate and post-pilot test soil data will be used to estimate the amount of VOC mass remaining in each geological layer at the sampling time periods. A comparison of the baseline, intermediate and post-pilot test soil sampling data in conjunction with the extracted soil vapor sampling data will be used to evaluate the overall performance of the system, to provide an estimate of the level and rate of treatment of

each layer during the SVE pilot test, and to predict (Section 5.1.2) the treatability of each of the unsaturated geologic layers.

### 4.7.2 PREDICTION OF SVE PERFORMANCE

As discussed in Section 5.1.1, the extracted soil vapor and soil sampling data will be used to evaluate the VOC mass removal and mass removal rates from the unsaturated geological layers in the pilot test area. The VOC mass removal rate for each geologic layer in conjunction with the available VOC mass data for each of the six treatment areas will be used to predict the performance of a full-scale SVE system (through extrapolation of the mass removal rate over time, including the diffusive mass flux from the lower permeability silt/clay layers, minor coal or ash materials in the overlying fill, and VOCs contained within the residual soil moisture). The predicted system performance will include expected VOC mass removal in the SVE discharge as a function of time and a projection of the reduction in soil VOC mass within each geologic layer over time. From these analyses, the expected performance (based on mass removal, TACO Tier III criteria or a combination of both) of the SVE system at the six treatment areas as a function of time will be estimated.

### 4.7.3 DETERMINATION OF FULL-SCALE SVE DESIGN AND OPERATIONAL PARAMETERS

Key elements in the design of an effective SVE system are the understanding and control of the airflow field that will be developed in the subsurface soils. The developed airflow field is largely a function of soil bulk permeability, the permeability differences between the geologic layers as a result of both soil heterogeneities and the degree of saturation of the soil pores with water or VOCs, and the air extraction/injection strategy used. Data collected during the pre-work plan data collection activities and SVE pilot testing along with the results of the subsequent data analysis and evaluations, as described in Sections 5.1.1 and 5.1.2, will be used to determine the full-scale SVE design and operational parameters for each of the six treatment areas. The key design and operational parameters that will be developed using airflow modeling<sup>6</sup> are outlined below:

<sup>&</sup>lt;sup>6</sup> These parameters will be determined using the AIR-3D computer model, a three-dimensional airflow model, developed by Baehr and Joss (1993) and distributed by the American Petroleum Institute (API, 1994). Modeling



- Air permeabilities of the geologic layers
- Vapor extraction/air injection strategy and effective well configurations
- Vertical and lateral placement of SVE and air injection well screens
- Achievable airflow as a function of operating vacuum/pressure
- Achievable air pore volume exchanges (i.e., effective ROI and projected mass removal as a function of time from each geologic layer) and soil vapor velocities as a function of distance from the vapor extraction and air injection wells as a function of operating configuration
- Effective zone of influence (ZOI) of the full-scale SVE system
- Process equipment requirements and sizing
- VOCs in air discharge and treatment requirement prior to discharge to atmosphere
- Low permeability cover and water handling needs
- System water and LNAPL handling requirements

The SVE systems will be designed to effectively and aggressively remediate the six treatment areas at the Site to meet the remedial objectives.

### 4.8 REPORTING

Upon completion of the SVE pilot testing, data validation and data analysis, a pilot test data evaluation report and a full-scale SVE implementation design for the six treatment areas will be developed and submitted to the U.S. EPA for review and approval prior to the installation and operation of the full-scale SVE system.

may include simple one-dimensional diffusion models (based on Fick's Laws) to estimate mass removal from mass transfer limited soil layers.



### 5.0 PILOT TEST SCHEDULE

This section provides a tentative schedule for the SVE pilot activities. This proposed schedule may change based on the results of the initial phases of the testing and actual duration required to complete various activities.

Milestone

#### Timeframe

November 13, 2008 December 13, 2008 (Within 30 days of work plan approval)

February 11, 2009 (Within 90 days of work plan approval)

March 13, 2009 (Within 120 days of work plan approval) April 12, 2009 (Within 150 days of work plan approval) August 10, 2009 (Within 270 days of work plan approval) December 8, 2009 (Within 360 days of work plan approval)

# Work plan approval from U.S. EPA Submit air permit application to IEPA Initiate discharge permit discussions with POTW Finalize process equipment design/specifications Complete baseline soil sampling Install SVE wells and vapor points

Issuance of air emissions permit by IEPA Issuance of discharge permit by POTW Delivery and installation of process equipment Equipment shakedown and startup Complete SVE pilot test

Submit SVE pilot test report



TABLES

### **SVE PILOT TESTING WORK PLAN**

### Table 1Soil VOC Data SummarySVE Pilot Testing Work PlanW.G. Krummrich Facility, Sauget, ILDecember 2008

Treatment Areas         Big Mo <sup>a</sup> Big Mo <sup>a</sup> Former Chlorobenzene Storage         Area <sup>b</sup> North Tank Farm <sup>c</sup> Near Little Mo <sup>d</sup> Former Steamer Overhead         Tank <sup>e</sup>	Maximum Detected Soil Concentrations (ug/kg)													
	Benzene	Chlorobenzene	Ethylbenzene	Toluene	Xylenes	1,2- Dichloro- benzene	1,4-Dichloro- benzene	2-Hexanone						
Big Mo <sup>a</sup>	13,000,000	5,100	BDL	BDL	BDL	1,200	2,400	17,000						
. 0	370	19,000,000	BDL	BDL	BDL	BDL	BDL	BDL						
North Tank Farm <sup>c</sup>	7,900	560,000	6.2	9.9	81	10,000	BDL	BDL						
Near Little Mo <sup>d</sup>	1,100,000	520	2,600	BDL	BDL	BDL	BDL	BDL						
	1.3	1,900	990	5.6	BDL	BDL	BDL	19,000						
Former Benzene Pipeline <sup>f</sup>	1,600,000	1,100,000	BDL	2,100	BDL	BDL	BDL	BDL						

Notes:

ug/kg – micrograms per kilogram

BDL - below detection limit

VOC - volatile organic compound

SVE - soil vapor extraction

<sup>a</sup> Big Mo is characterized by URS soil borings S0601, S0607, DNAPL K-8, and XDD soil borings SB-A01 and SB-A03

<sup>b</sup> Former Chlorobenzene Storage Area is characterized by URS soil boring S0610, and XDD soil boring SB-B01

<sup>c</sup> North Tank Farm is characterized by URS soil borings S0502, S0501 and S0513

<sup>d</sup> Near Little Mo is characterized by URS soil borings S0506, S0516, and XDD soil boring SB-D01

<sup>e</sup> Former Steamer Overhead Tank is characterized by URS soil borings Sump-277, S0514, S0503, and XDD soil boring SB-E01

<sup>f</sup> Former Benzene Pipeline is characterized by URS soil borings S0428, S0413, and XDD soil boring SB-F01



### Table 2Big Mo Soil VOC DataSVE Pilot Testing Work PlanW.G. Krummrich Facility, Sauget, ILDecember 2008

Sample	Sampling				Soil Con	centrations (ug/kg)	)			
Sample LocationInter belowS06018S06018S06079S06079	Interval (feet below grade)	Benzene	Chlorobenzene	Ethylbenzene	Toluene	Xylenes	1,2-Dichloro- benzene	1,4-Dichloro- benzene	2-Hexanone	
S0601	8-10	23,000 J	590	ND (160)	ND (160)	ND (320)	ND (440)	ND (440)	ND (800)	
S0601	8-10 Duplicate	55,000 J	680	ND (310)	ND (310)	ND (610)	ND (440)	ND (440)	ND (1,500)	
S0607	1-3	2,400,000	ND (1,500)	ND (1,500)	ND (1,500)	ND (3,000)	1,200	2,400	ND (7,400)	
S0607	9-11	1,200,000	1,400	ND (1,000)	ND (1,000)	ND (2,000)	ND (440)	ND (440)	ND (5,100)	
S0607	14-16	1,700,000	5,100	ND (1,200)	ND (1,200)	ND (2,400)	ND (380)	ND (380)	ND (5,900)	
DNAPL K-8	5-8	43,000	ND (1,300)	ND (1,300)	ND (1,300)	ND (2,600)	ND (430)	ND (430)	ND (6,600)	
DNAPL K-8	15-18	2,000,000	ND (21,000)	ND (21,000)	ND (21,000)	ND (42,000)	ND (390)	ND (390)	ND (110,000)	
SB-A01	11-12	13,000,000	ND (830,000)	ND (830,000)	ND (830,000)	ND (1,700,000)	ND (830,000)	ND (830,000)	ND (4,200,000)	
SB-A03	11-12	960,000	ND (33,000)	ND (33,000)	ND (33,000)	ND (65,000)	ND (33,000)	ND (33,000)	17,000 J	

Notes:

ug/kg - micrograms per kilogram

ND (160) - not detected at the detection limit of 160 ug/kg

J-estimated

VOC – volatile organic compounds

SVE - soil vapor extraction

S0601, S0607, DNAPL K-8 soil samples were collected by URS

SB-A01 and SB-A03 soil samples were collected by XDD during the pre-work plan data collection event





### Table 3 Pilot Test Design Summary SVE Pilot Testing Work Plan W.G. Krummrich Facility, Sauget, IL December 2008

Wells	Typical Screen Interval (feet bg)	Target Geology	Expected ROI <sup>2</sup> (feet)	Approximate Well Spacing (feet on-center)	Design Air Flow Rate <sup>3</sup> [per well] (scfm)	Design Vacuum <sup>3</sup> [at individual wellhead] (inches of Hg)	No. of Wells	
SVE in the Higher P	ermeability Laye	rs						
Upper SVE <sup>1</sup>	4.5 - 9.5	Fill and Upper Silty Sand	10 - 15	10 - 30	8 - 15	5 - 12	22	
Lower SVE <sup>1</sup>	12.5 - 14.5	Lower Silty Sand	10 15	10 50	0 13	5 12	22	
SVE in the Lower P	ermeability Layer							
Intermediate SVE	10.5 - 11.5	Intermediate Silty Clay	2 - 3	5	1 - 2.5	22 - 27	4	

### OTHER DESIGN PARAMETERS

SVE Target Area:

=> 60 feet x 80 feet = 4,800 feet<sup>2</sup> (in the fill, upper silty sand and lower silty sand layers)
 => 10 feet x 10 feet = 100 feet<sup>2</sup> (in the intermediate silty clay layer)

• SVE Target Depth (total) = 15 feet bg

• Total No. of  $SVE^1$  Wells = 26

SVE Design Air Flow Rate (total) = 124 - 235 scfm (assuming 19 wells will operate as vapor extraction wells)
Air Injection Design Flow Rate (total) = 56 - 105 scfm (assuming 7 wells will operate as air injection wells)

### Notes:

The conceptual operating parameters (i.e., air flow rate and vacuum/pressure at individual wellheads) may change based on the actual field conditions.

<sup>1</sup> Five (5) SVE wells in the lower silty sand layer and two (2) SVE wells in the fill and upper silty sand layer will also be fitted with air injection manifold, and can be used as both air injection and vapor extraction wells. During the Design Parameter Evaluation phase, these seven (7) wells will be tested for air injection and vapor extraction. However, during the extended Performance Evaluation phase, these seven (7) wells will likely be operated as air injection wells.

<sup>2</sup> The estimated ROI values are based upon the data collected during the June 2008 pre-work plan data collection event. These ROI values are estimated for the design of the SVE pilot test only, and the data collected during the pilot test will be used in the full-scale SVE design.

<sup>3</sup> The design air flow rate and vacuum at individual wellheads are based on the data collected during the June 2008 pre-work plan data collection event. These conceptual operating parameters are estimated for the SVE pilot test only, and the data collected during the pilot test will be used in the full-scale SVE design.

SVE = Soil vapor extraction ROI = Radius of influence bg = below grade scfm = standard cubic feet per minute feet<sup>2</sup> = square feet inches of Hg = inches of mercury



### Table 4 Monitoring Plan SVE Pilot Testing Work Plan W.G. Krummrich Facility, Sauget, IL December 2008

Event	Task	Description	Sampling Location	Measurement Location	Frequency	Samples per Event	Analytical Method
Baseline	Sampling						
	Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once	26	8260B
	Soil Vapor Sampling	Soil vapor samples from SVE wells	SVE wells	Field measurement Laboratory analysis	once	26 3	PID TO-15
		Soil vapor samples from vapor probes/points	Vapor probes/points	Field measurement Laboratory analysis	once	25 3	PID TO-15
Design I	Parameter Evaluation Tes	l sting					
	Operation Parameter Monitoring	Vapor extraction rates at individual SVE wells Vacuum at individual SVE wells Air injection rates at individual wells Pressure at individual air injection wells Vacuum/pressure at vapor probes/points Air/vapor flow, pressure, vacuum, temperature Extracted perched water/condensate volume Groundwater elevation gauging	SVE wells SVE wells SVE wells SVE wells Vapor probes/points Process equipment Process equipment SVE wells/vapor probes/PSMW-05	Flow meters and pressure, vacuum & temperature gauges Water level indicator	hourly to daily reac	visual reading	
	Extracted Soil Vapor	SVE stream from higher and lower permeability	A A		daily	2	PID
	Sampling	layers	Process equipment	Field measurement	daily	2	PID
	Derameter Evaluation Testing           Operation Parameter Monitoring         Vapor extraction rates at individual SVE wells Air injection rates at individual air injection wells Pressure at individual air injection wells Vacuum/pressure at vapor probes/points Air/vapor flow, pressure, vacuum, temperatu Extracted perched water/condensate volume Groundwater elevation gauging           Extracted Soil Vapor         SVE stream from higher and lower permeabi layers           Air Treatment Sampling         Air permit compliance To confirm treatment effectiveness           formance Evaluation Testing         Vapor extraction rates at individual SVE wells Air injection rates at individual are injection wells Vacuum/pressure at vapor probes/points Air/vapor flow, pressure, vacuum, temperatu Extracted perched water/condensate volume Total vapor extraction flowrate Groundwater elevation gauging           Soil Vapor Sampling         Soil vapor samples from SVE wells           Soil vapor samples from vapor probes/points         Soil vapor samples from vapor probes/points		post-treatment	Laboratory analysis	per Peri	TO-15	
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	twice a week	1	PID
	formance Evaluation Tex Operation Parameter Monitoring Soil Vapor Sampling Extracted Soil Vapor Sampling	Vapor extraction rates at individual SVE wells Vacuum at individual SVE wells Air injection rates at individual wells Pressure at individual air injection wells Vacuum/pressure at vapor probes/points Air/vapor flow, pressure, vacuum, temperature Extracted perched water/condensate volume Total vapor extraction flowrate Groundwater elevation gauging Soil vapor samples from SVE wells Soil vapor samples from vapor probes/points SVE streams from the three SVE mainlines (fill and upper silty sand layers, intermediate silty clay layer and lower silty sand layer) + combined SVE	SVE wells SVE wells SVE wells SVE wells SVE wells Vapor probes/points Process equipment Process equipment SVE wells/vapor probes/PSMW-05 SVE wells Vapor probes/points Process equipment	Flow meters and pressure, vacuum & temperature gauges Water level indicator Field measurement Laboratory analysis Field measurement Laboratory analysis	daily - 1st week weekly - 1st month biweekly - afterwards once at starup once at mid-point once at end once at end daily - 1st week biweekly - 1st month monthly - afterwards	19 19 7 7 25 7 1 1 6 19 3 25 3 4 4	visual reading PID TO-15 PID TO-15 PID TO-15
	Soil Sampling	Soil samples via Terrcore at Test mid-point	See Figure 9	Laboratory analysis	once	26	8260B
	Air Treatment Sampling	Air permit compliance	post-treatment	Laboratory analysis	per Peri	mit	TO-15
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	monthly	1	PID
	ot Test Sampling Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once - one week after pilot test concluded	26	8260B

### Summary of Sampling for Laboratory Analysis

	Samples per Event	Frequency of Events	Total Number of Samples
Soil Sample Analysis via 8260B Method			
Baseline sampling	26	1	26
Sampling at mid-point of Performance Evaluation Testing	26	1	26
Post-pilot test sampling	26	1	26
Total Number of Soil Analyticals (excluding QA	A/QC samples)		78
Soil Vapor Sample Analysis via TO-15			
Baseline SVE well sampling	3	1	3
Baseline vapor probe/point sampling	3	1	3
SVE well sampling during Performance Evaluation Testing	3	3	9
Vapor probe/point sampling during Performance Evaluation Testing	3	1	3
Extracted soil vapor sampling during Performance Evaluation Testing	4	7	28
Total Number of Soil Vapor Analyticals (exclude	ling QA/QC samples)		46
Air Permit Compliance Monitoring			
Vapor discharge sampling per air permit requirements	1	per Permit	to be determined
Total Number of Vapor Discharge Analyticals	excluding OA/OC samples)		to be determined

Notes:

PID - photoionization detector 8260B - U.S. EPA Method 8260B for VOC analysis TO-15 - U.S. EPA Method TO-15 for VOC analysis Permit - Permit required for air discharge SVE - soil vapor extraction



### Table 5 Soil Vapor Data & Projected Vapor Emissions SVE Pilot Testing Work Plan W.G. Krummrich Facility, Sauget, IL December 2008

Primary VOCs	Extracted Soil Vapor Concentration (ppmv) <sup>a</sup>	Potential Discharge to Atmosphere <sup>b</sup> (lbs/hr) (ton/yr)			
Benzene	3,300	9.4	41.3		
Chlorobenzene	14 U	0.06	0.25		
Ethylbenzene	14 U	0.05	0.24		
Xylenes	14 U	0.05	0.24		
1,2-Dichlorobenzene	14 U	0.08	0.33		
1,4-Dichlorobenzene	14 U	0.08	0.33		
	TOTAL <sup>c</sup>	10	43		

Notes:

U - not detected

ppmv - parts per million by volume

a - maximum measured concentration of soil vapor at Big Mo Area during pre-pilot test design data collection

b - assumed SVE system flow of 235 scfm; and not detected VOC concentrations = detection limit (Table 3)

c - total hazardous air pollutant (HAP) loading to air is 43 ton/yr, which is above the Illinois Bureau of Land 25 ton/yr limit for a minor source

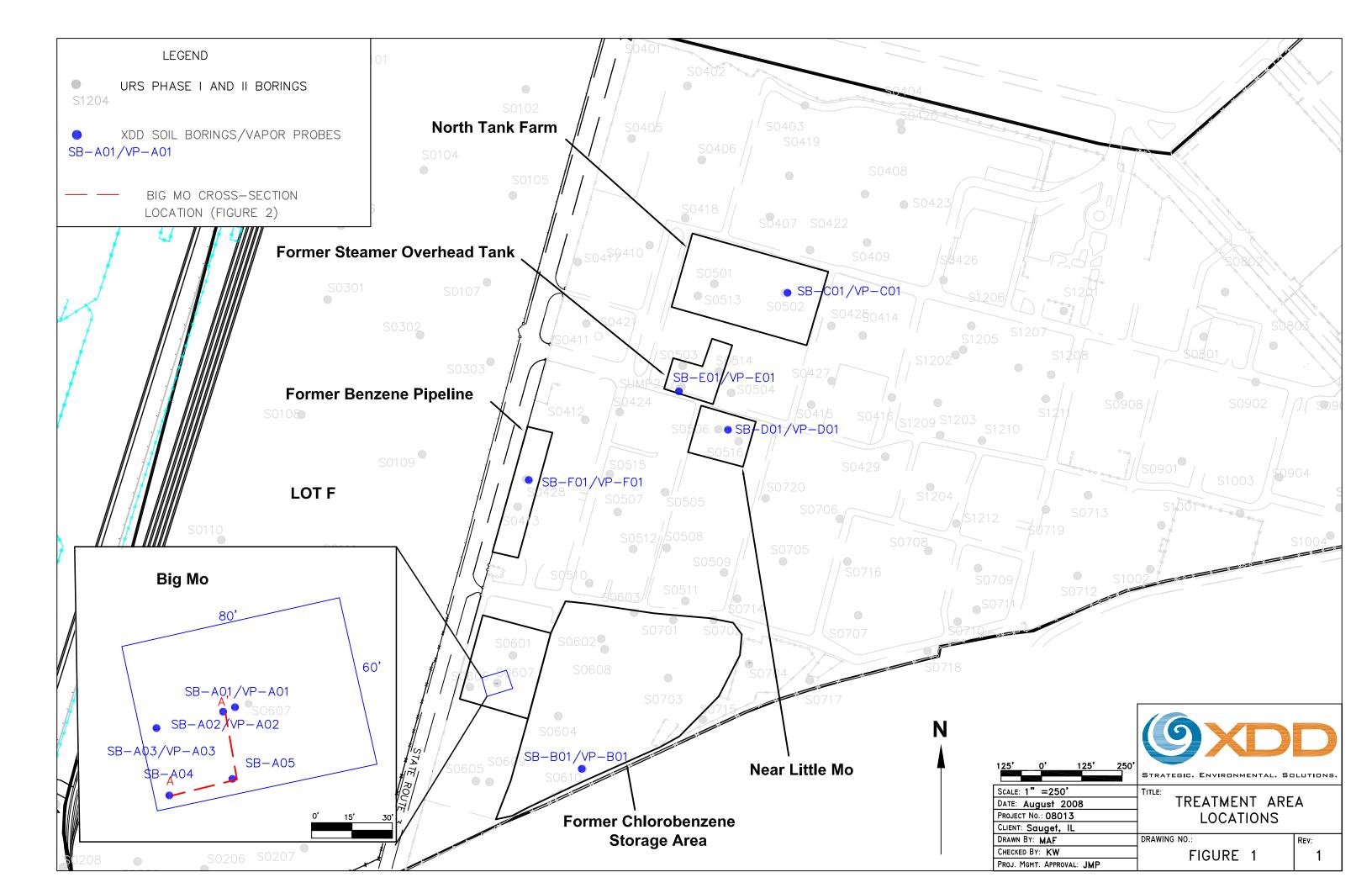
additionally the estimated maximum benzene loading to air is 41 ton/yr, which is above the Illinois Bureau of Land 10 ton/yr limit for a single HAP

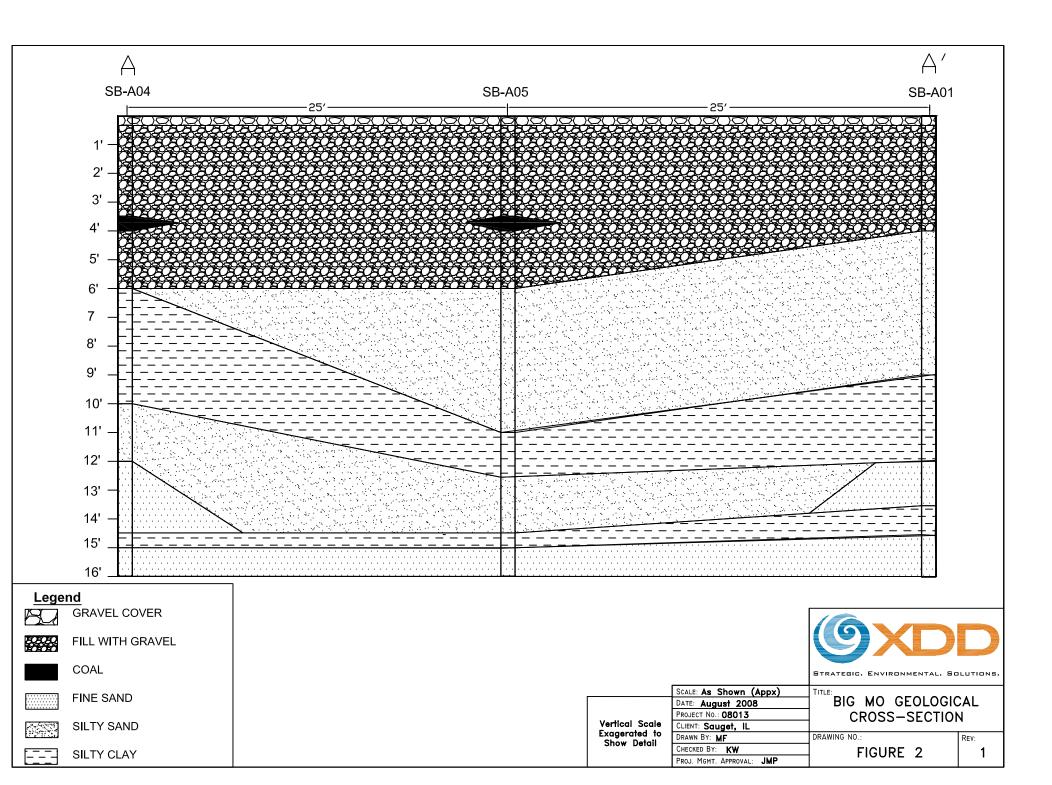


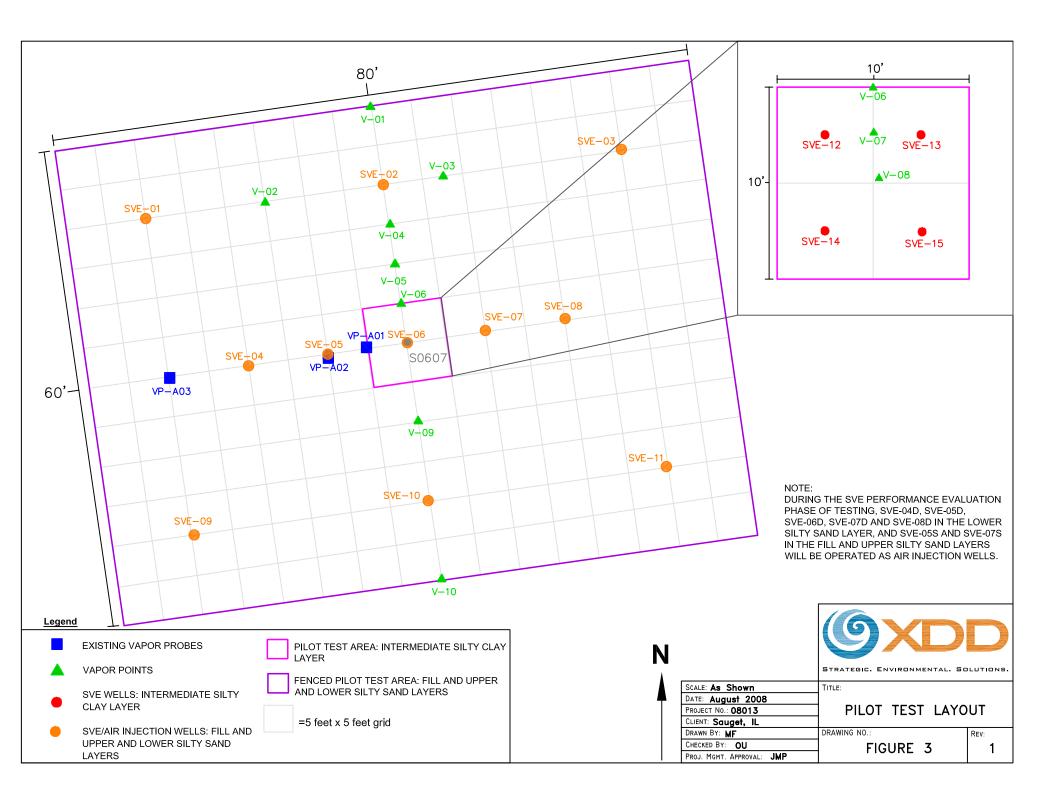


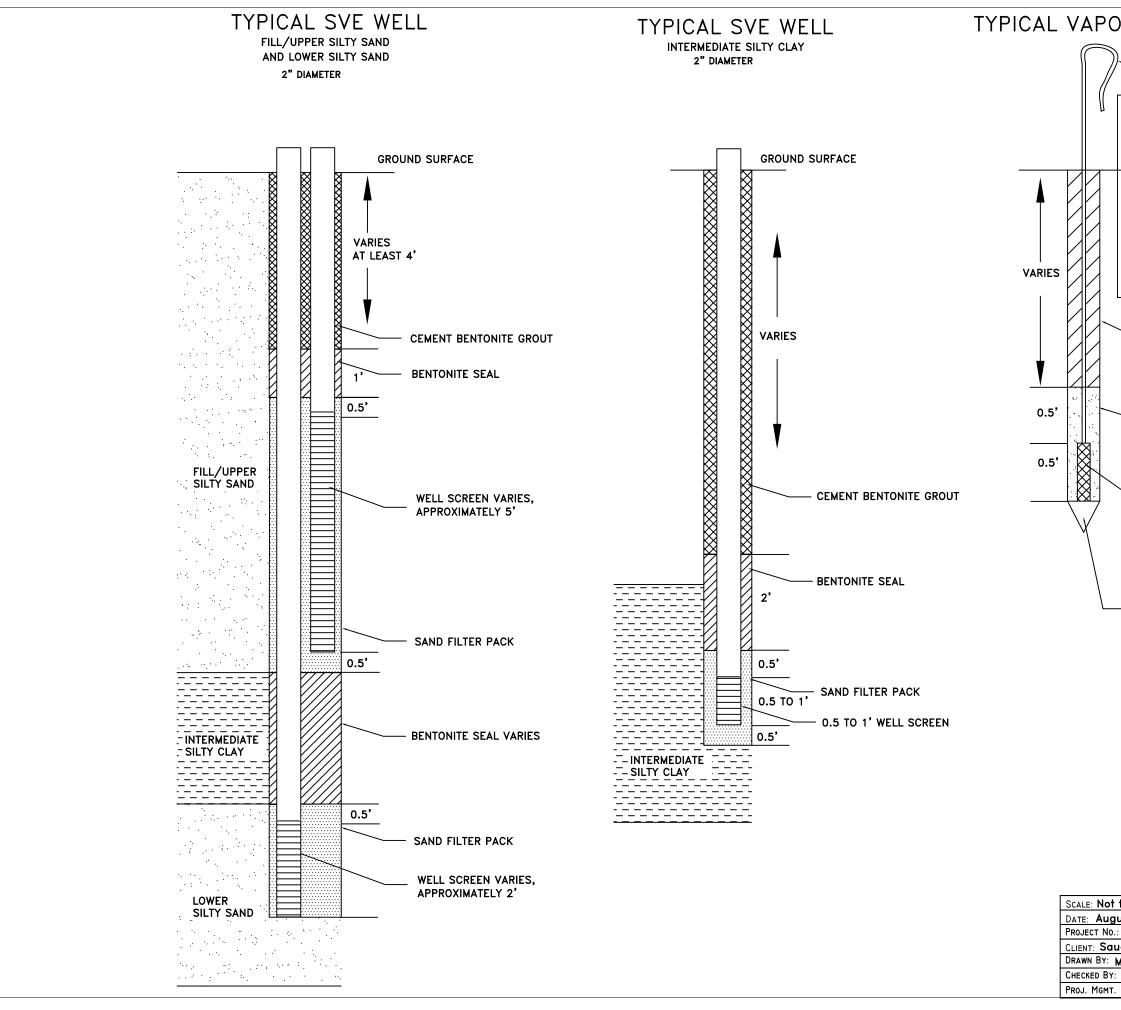
### FIGURES

### **SVE PILOT TESTING WORK PLAN**

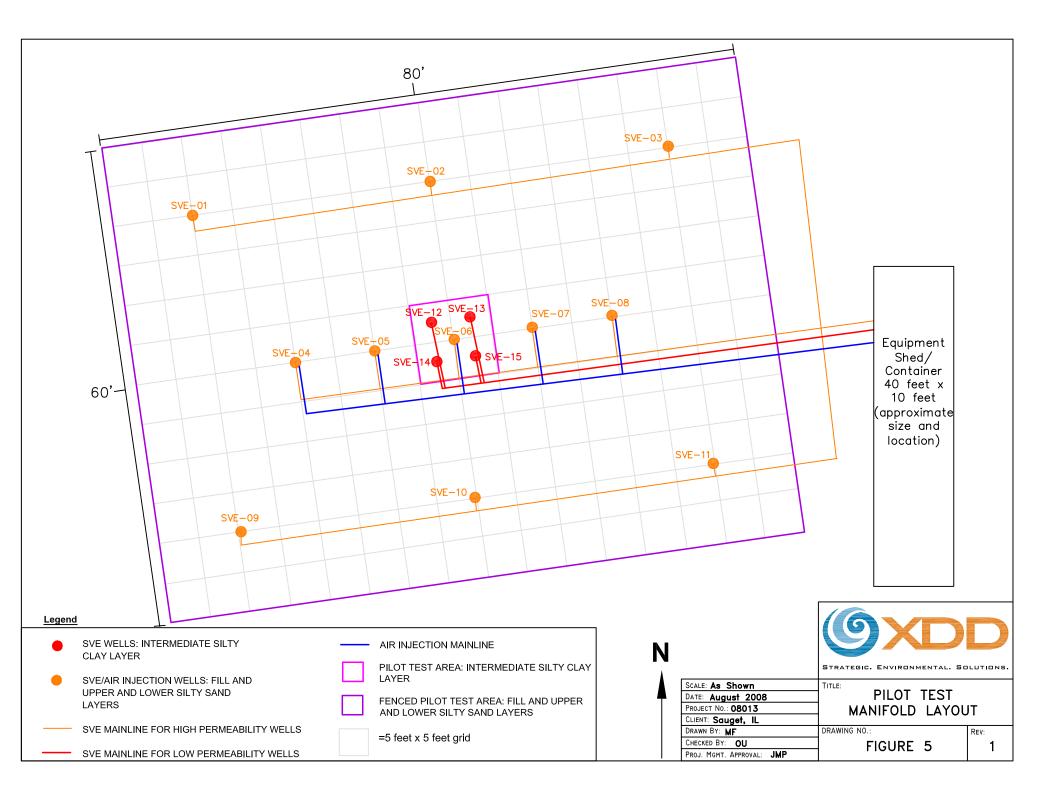


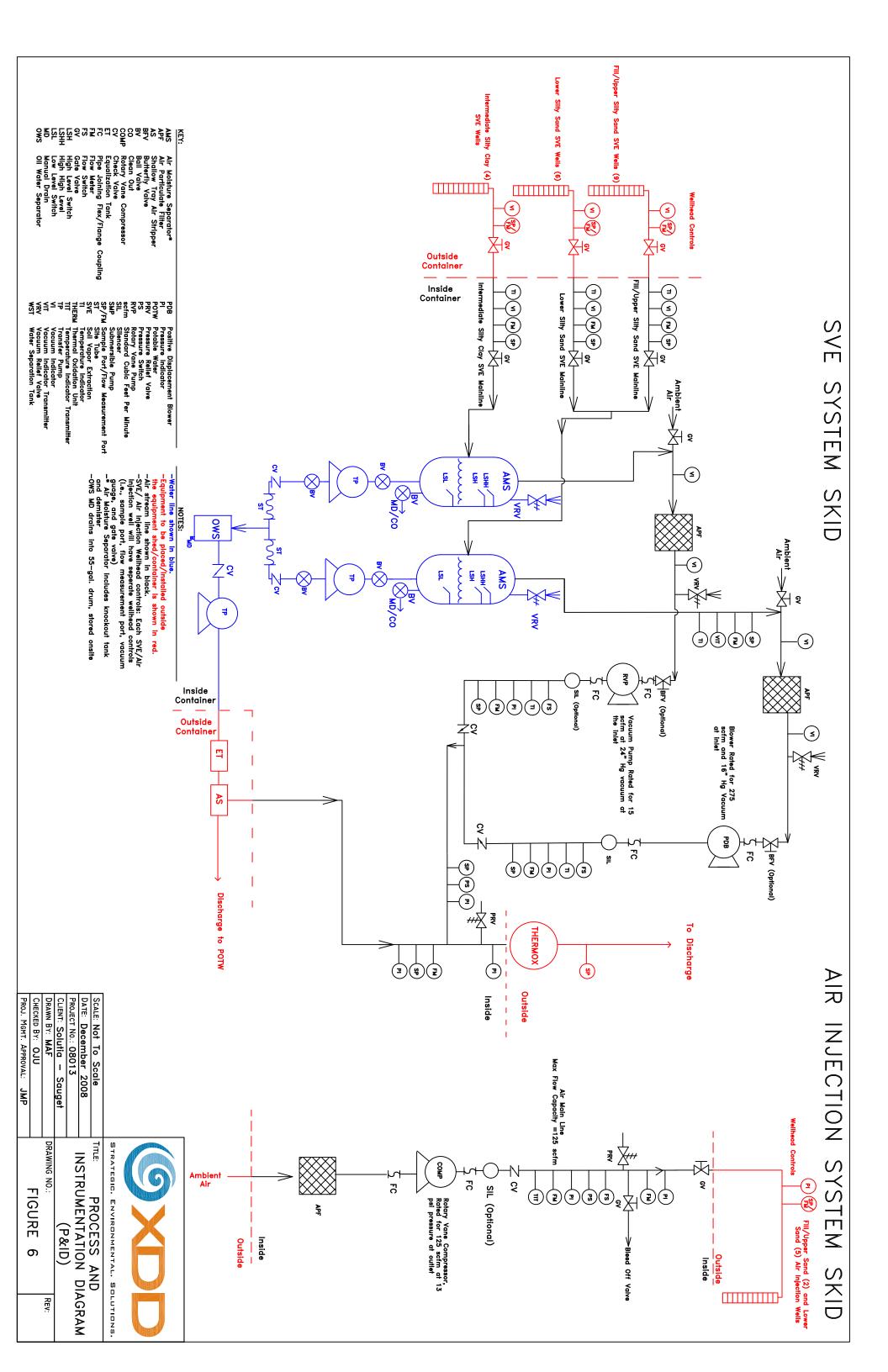


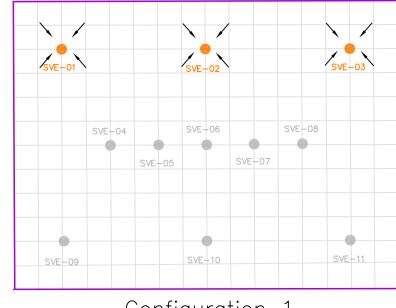




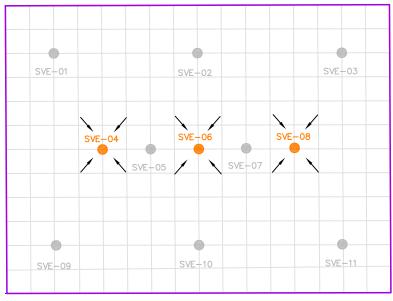
OR POINT		
	3	
	BAR FOR SUPPORT	
	RFACE	
BENTONITE S	SEAL	
SAND/GLAS	S BEADS	
GEOPROBE	PERMANENT IMPLANT	
ANCHOR POINT		
	<b></b>	
	STRATEGIC. ENVIRONMENTAL. SO	
ot to Scale ugust 2008	TITLE: TYPICAL SVE WELL	
No.: 08013 Sauget, IL	VAPOR POINT	
(: MF By: OU mt. Approval: JMP	DRAWING NO.: FIGURE 4	Rev: 1







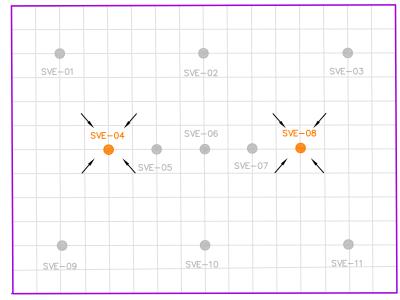
Configuration 1



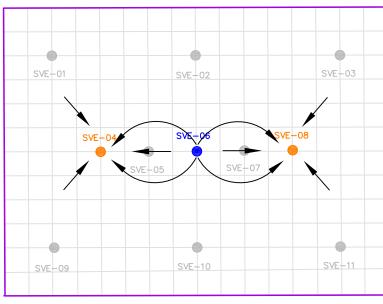
### Configuration 4

Note: Well-field configurations 1 through 7 will be tested separately in both the fill and upper silty sand layers and the lower silty sand layer.

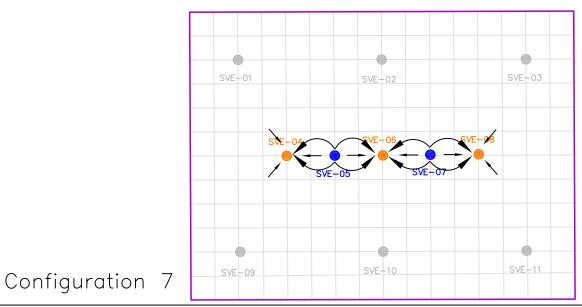
### Legend SVE WELLS IN OPERATION AS VACUUM WELLS ۲ SVE WELLS IN OPERATION AS PRESSURE WELLS SVE WELLS NOT IN OPERATION ۲ FENCED PILOT TEST AREA: FILL AND UPPER AND LOWER SILTY SAND LAYERS 5 FEET X 5 FEET AREA AIR FLOW DIRECTION

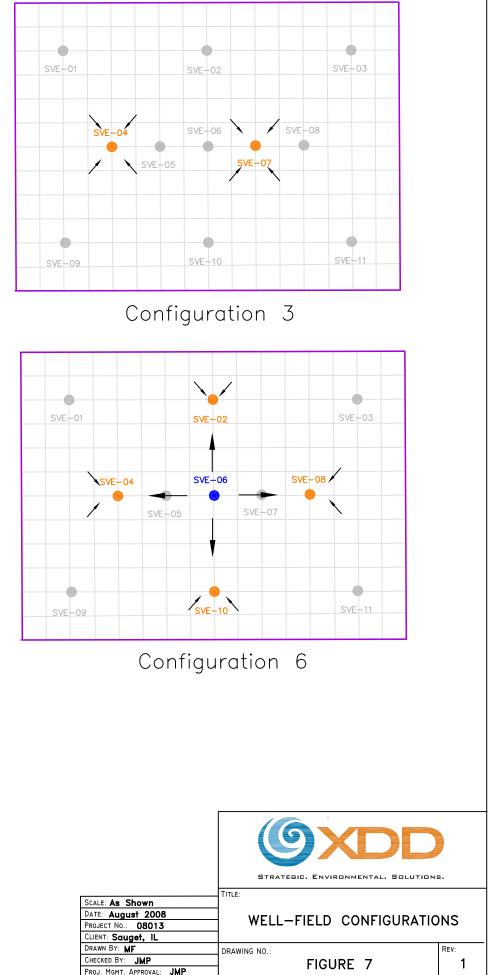


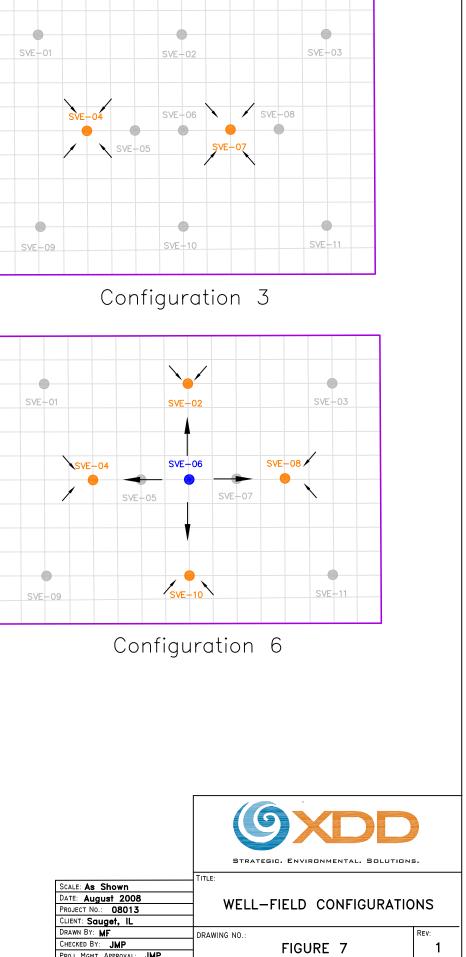
Configuration 2

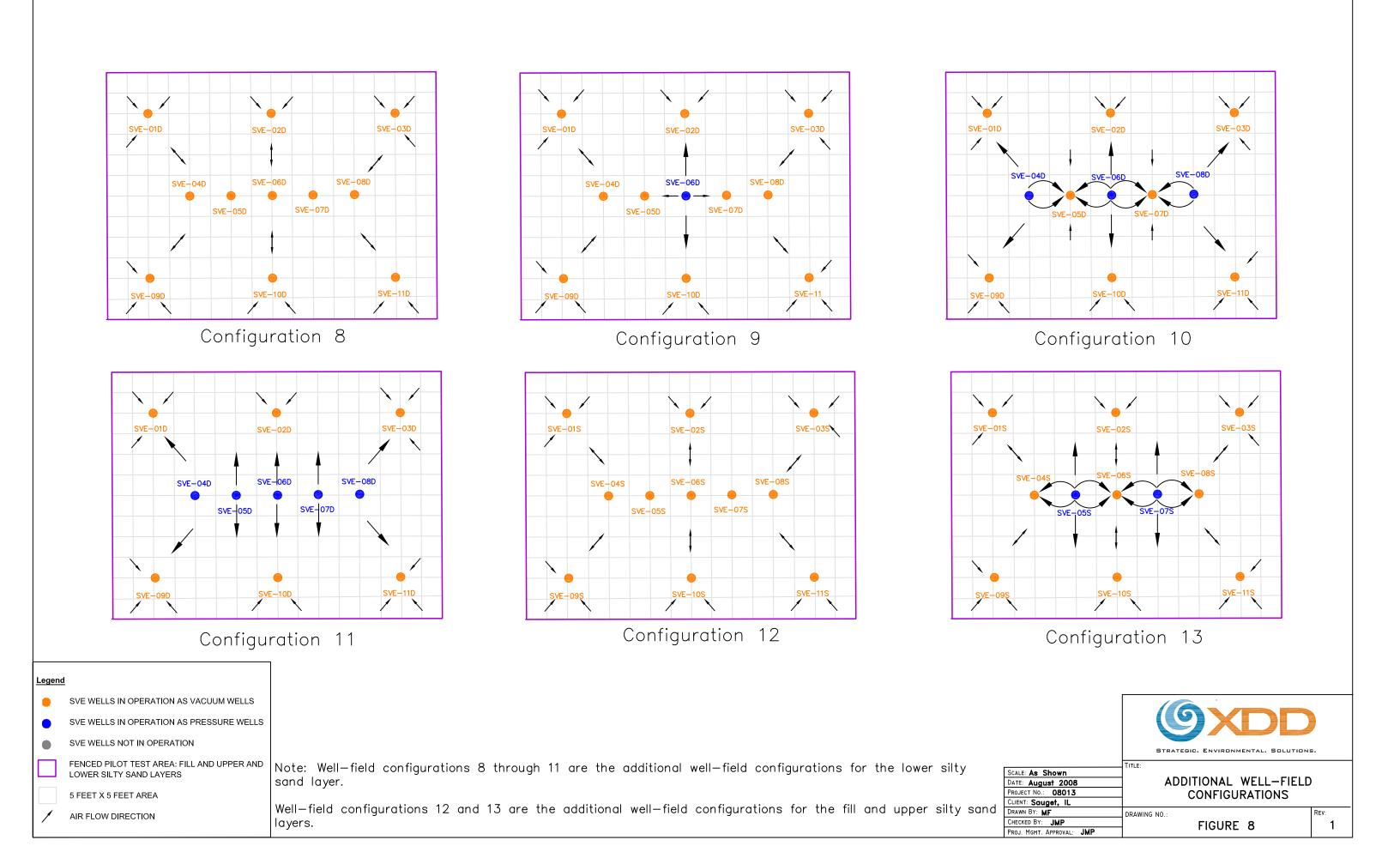


Configuration 5









### **APPENDIX** A

### PRE-WORK PLAN DATA COLLECTION SUMMARY

### SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA INC. 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

### **August 2008**



### PRE-WORK PLAN DATA COLLECTION SUMMARY

The pre-work plan data collection event was conducted by XDD, LLC from June 23 to July 3, 2008, prior to the development of the soil vapor extraction (SVE) pilot testing work plan. Prework plan data collection activities were conducted in each of the six treatment areas to aid in the design of the pilot testing program and included:

- a) soil borings to obtain more specific data on the geology of the unsaturated soils for each treatment area, and to understand the VOC distribution within the varying geologic layers observed at each treatment area; and
- b) vapor probe installation and testing to obtain preliminary data on the concentrations of the VOCs in the soil vapor and to obtain air permeability data (air permeability data provided in Appendix B) for the significant (greater than 1 foot thickness) geologic layers observed.

The following attachments provide figures (i.e., geologic cross-sections, boring logs) prepared as part of the pre-work plan data collection event and laboratory analytical data collected during the event. Refer to Figure 1 of the SVE Pilot Testing Work Plan for soil boring and vapor probe locations.

Attachment A: Geological Cross-Sections of the Treatment Areas
(based on the historical soil boring data collected from URS Corporation)
Attachment B: Soil Boring Logs/Vapor Probe Installation Logs
(from 2008 pre-work plan data collection event)
Attachment C: Soil VOC Laboratory Analytical Data
(from 2008 pre-work plan data collection event)
Attackment D. Soil Van an VOC Lakenstern. Analytical Data

Attachment D: Soil Vapor VOC Laboratory Analytical Data (from 2008 pre-work plan data collection event)



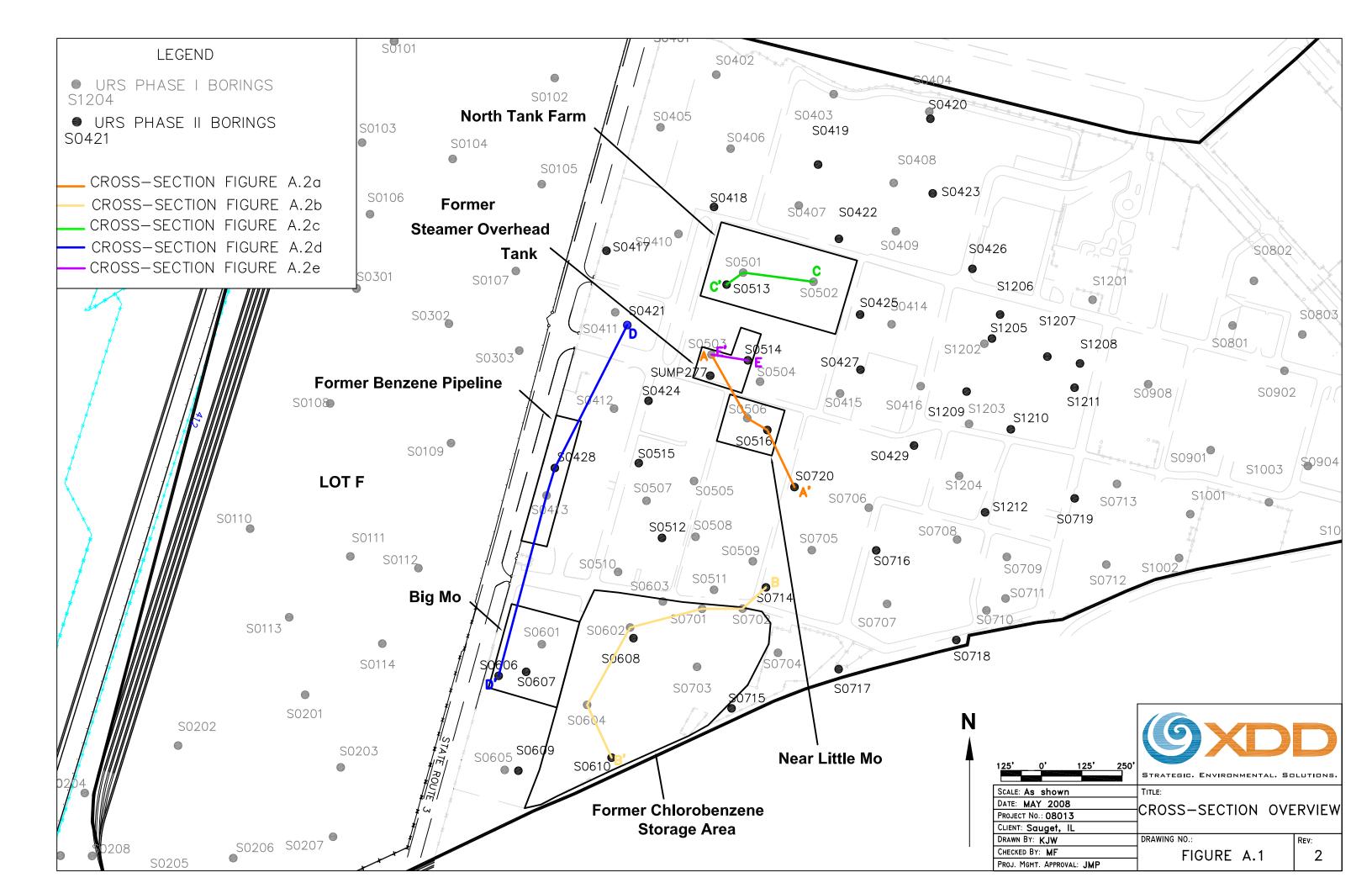
### ATTACHMENT A GEOLOGICAL CROSS-SECTIONS OF THE TREATMENT AREAS

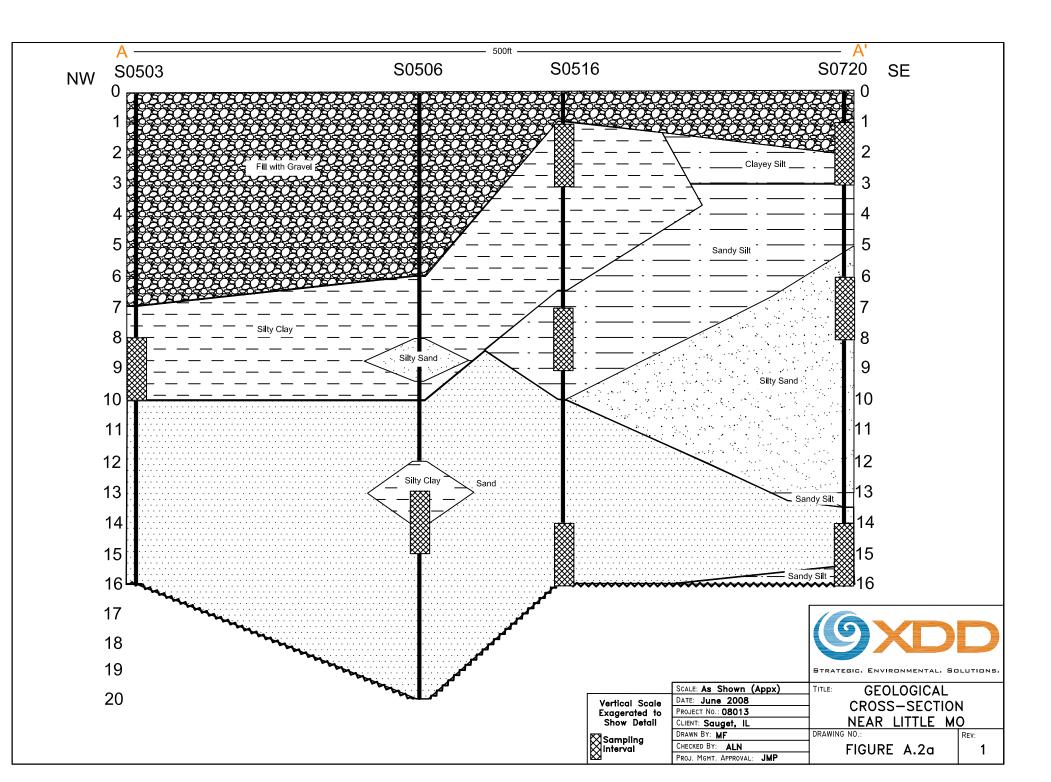
Figure A.1: Cross-Section Overview Figure A.2a: Geological Cross-Section Near Little Mo Area Figure A.2b: Geological Cross-Section Former Chlorobenzene Storage Area Figure A.2c: Geological Cross-Section North Tank Farm Area Figure A.2d: Geological Cross-Section Former Benzene Pipeline and Big Mo Areas Figure A.2e: Geological Cross-Section Former Steamer Overhead Tank Area

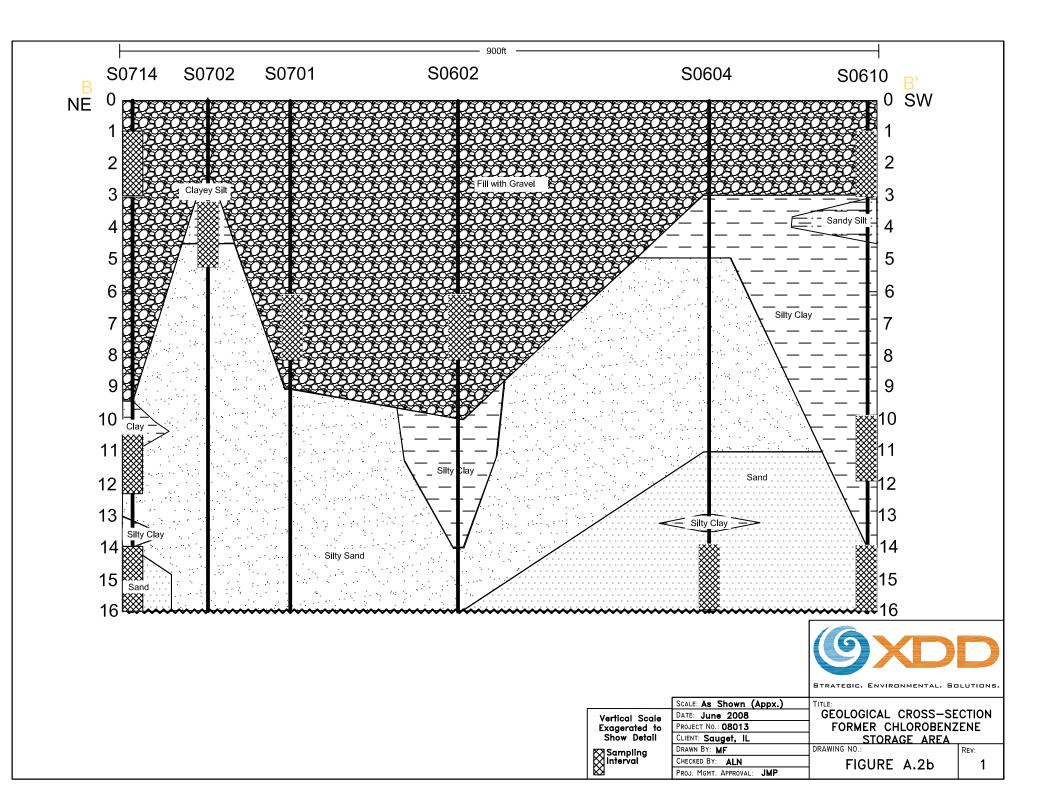
### APPENDIX A PRE-WORK PLAN DATA COLLECTION SUMMARY

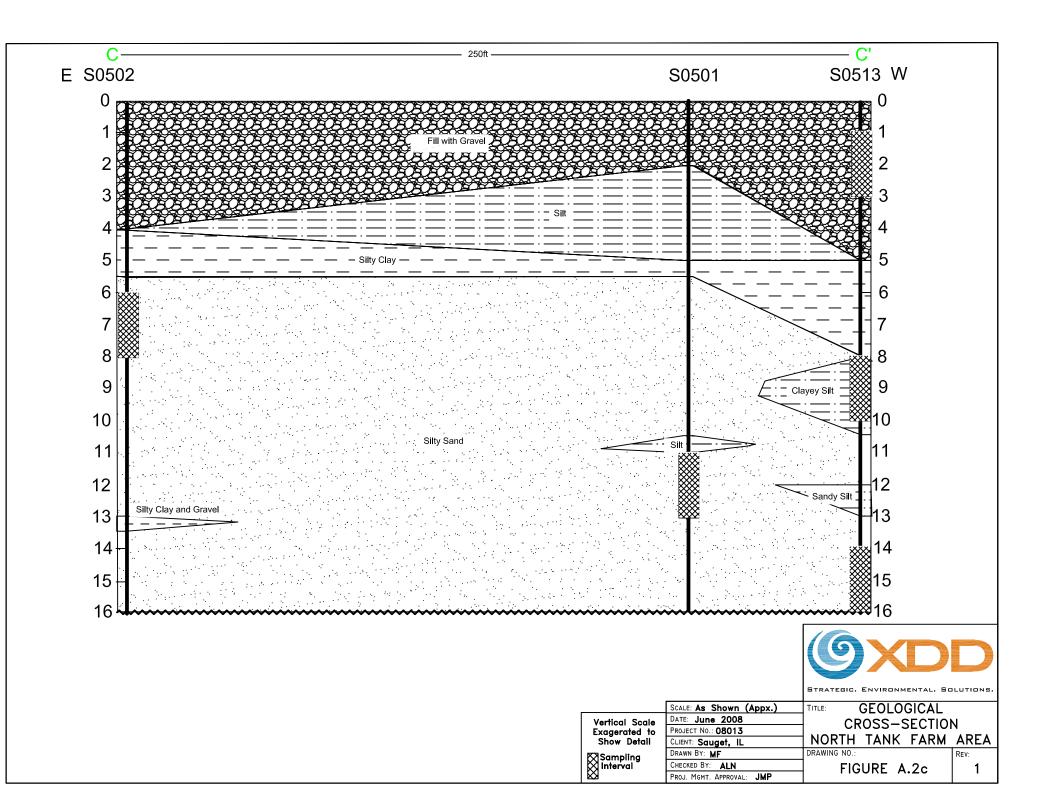
### SVE PILOT TESTING WORK PLAN

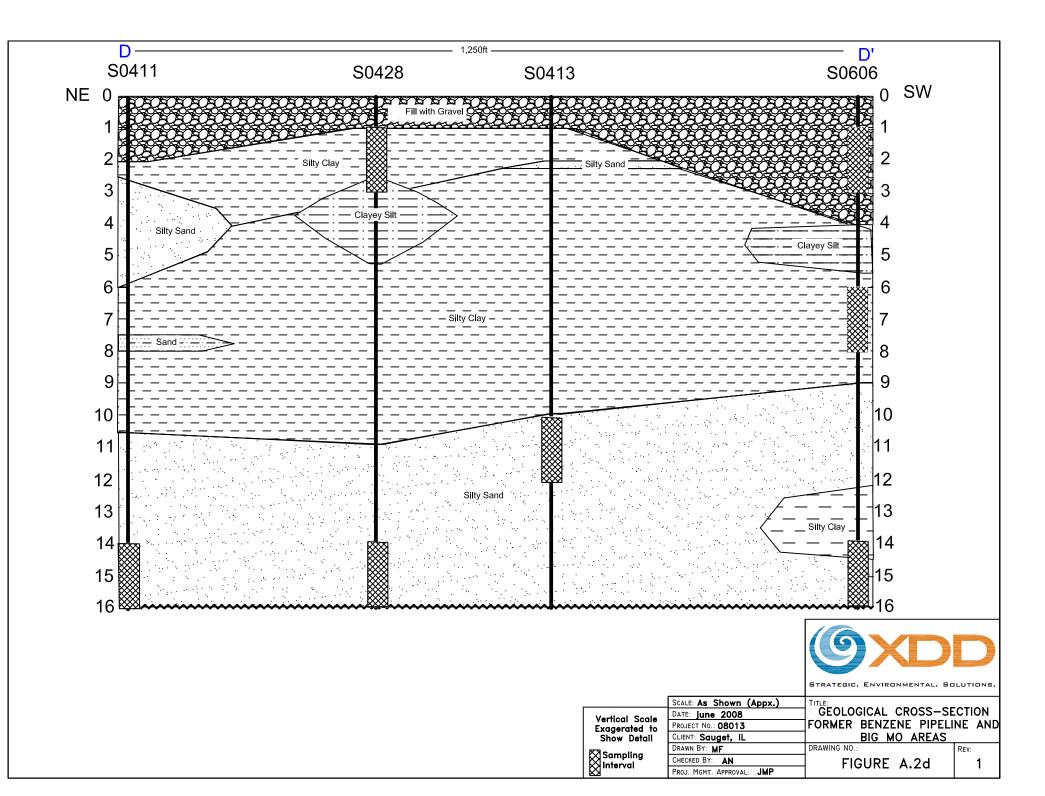
APPENDIX A: PRE-WORK PLAN DATA COLLECTION SUMMARY Soil Vapor Extraction Pilot Testing Work Plan

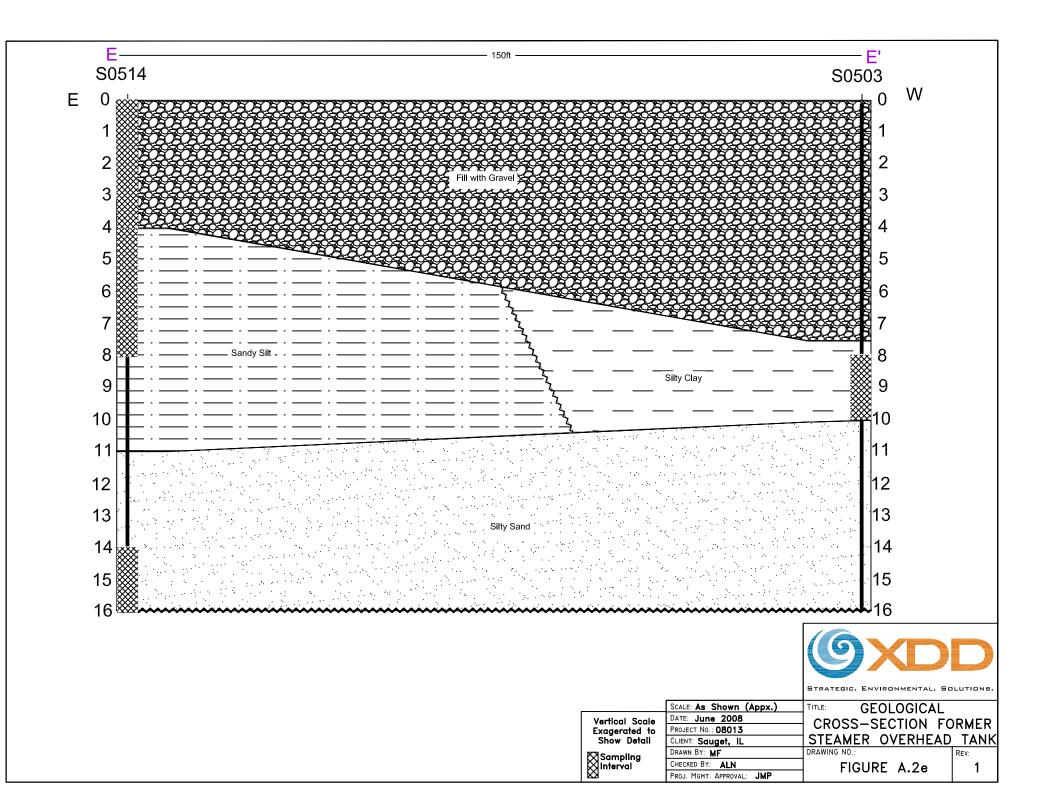












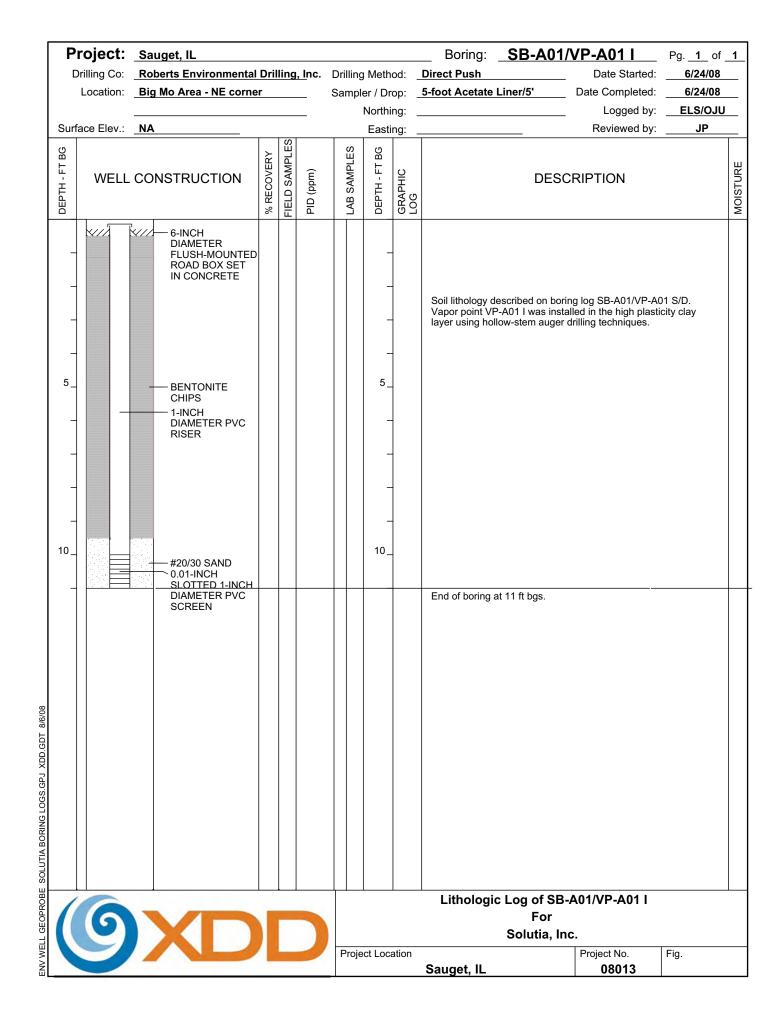


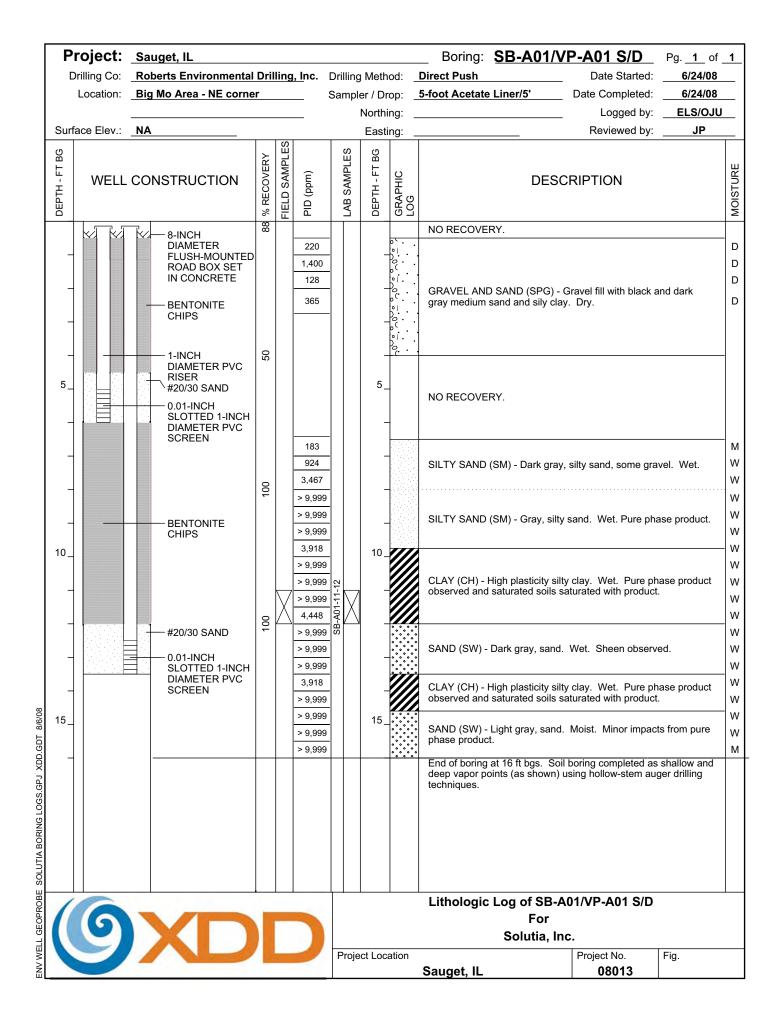
### ATTACHMENT B SOIL BORING LOGS/ VAPOR PROBE INSTALLATION LOGS

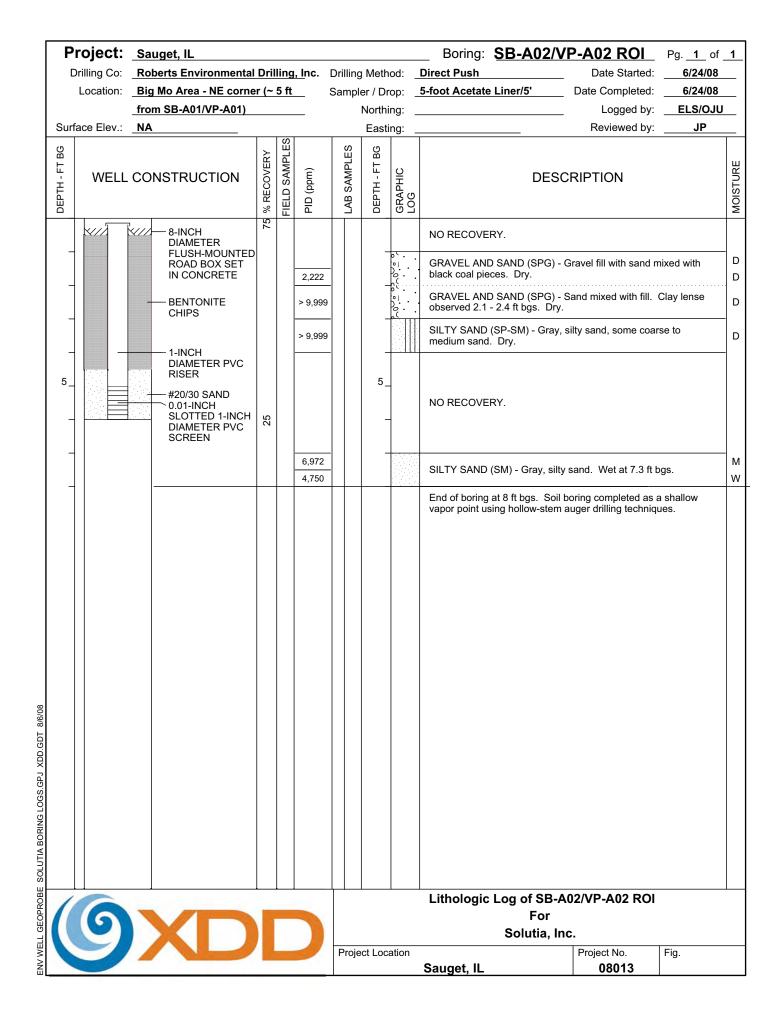
SB-A01/VP-A01 Shallow/Deep (S/D) SB-A01/VP-A01 Intermediate (I) SB-A02/VP-A02 ROI SB-A03/VP-A03 S/D SB-A03/VP-A03 S/D SB-A04 SB-A04 SB-A05 SB-B01/VP-B01 S/D SB-B01/VP-B01 I SB-C01/VP-C01 S/D SB-D01/VP-D01 S/D SB-E01/VP-E01 S/D SB-F01/VP-F01 I

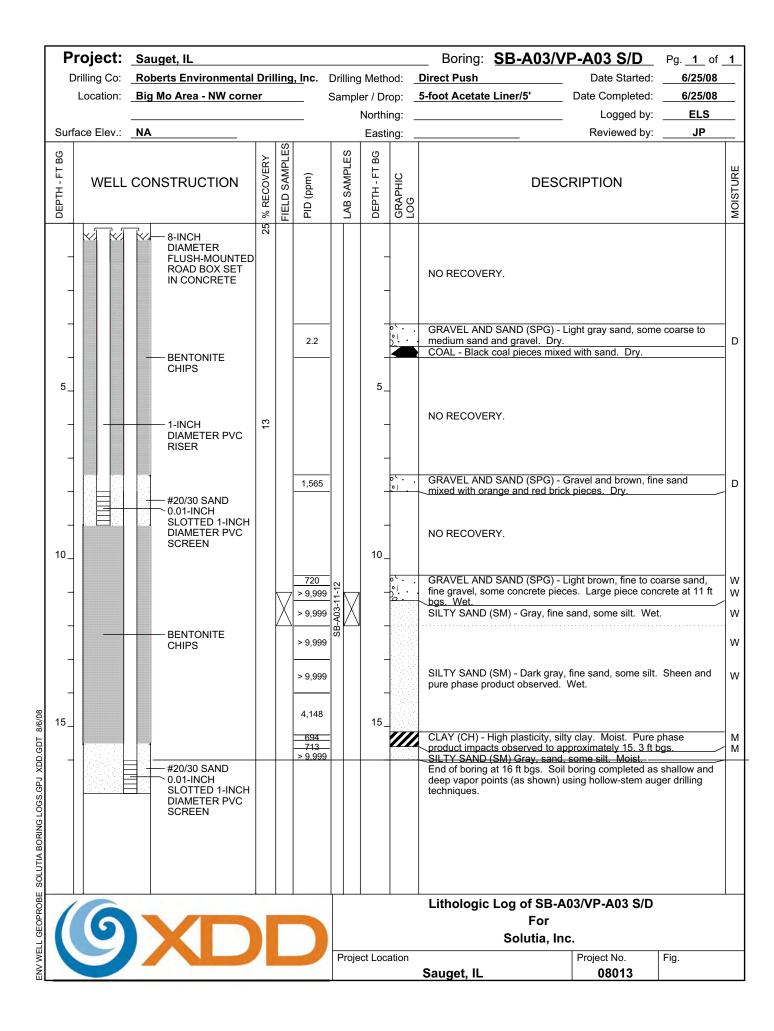
### APPENDIX A PRE-WORK PLAN DATA COLLECTION SUMMARY

### SVE PILOT TESTING WORK PLAN





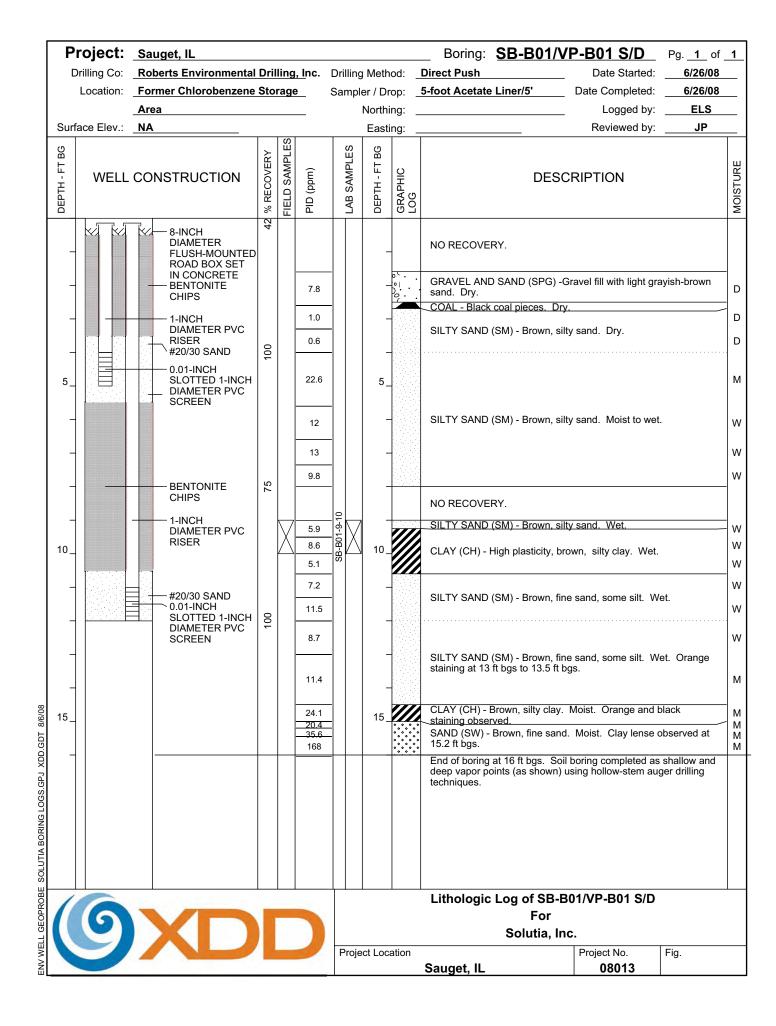


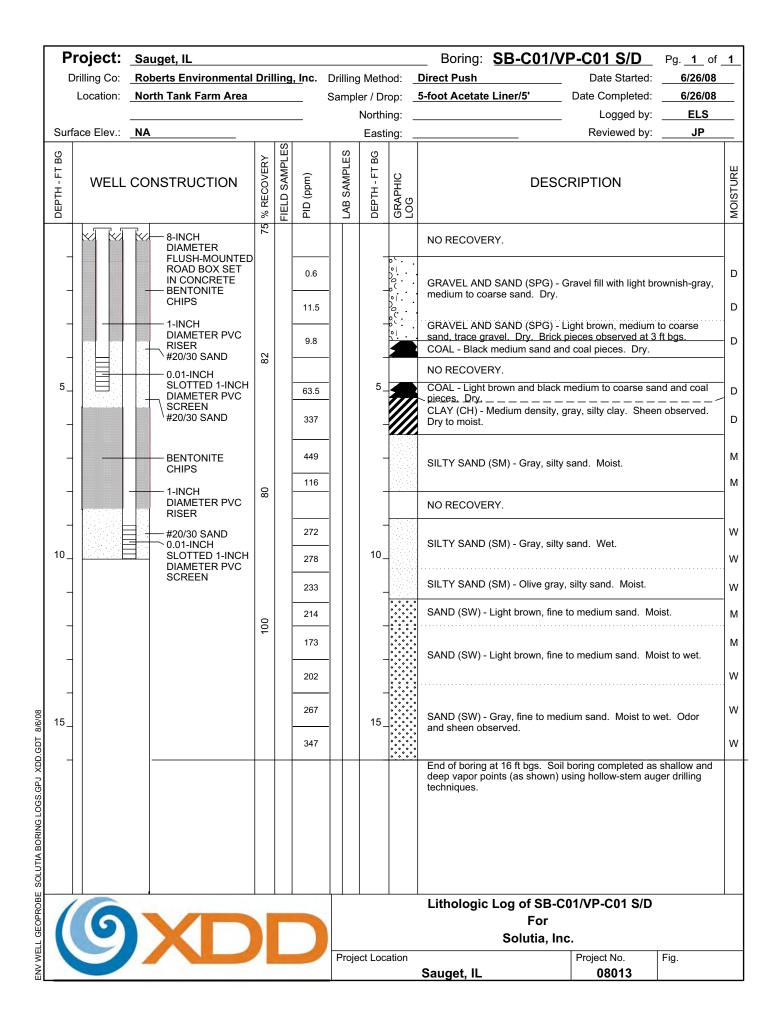


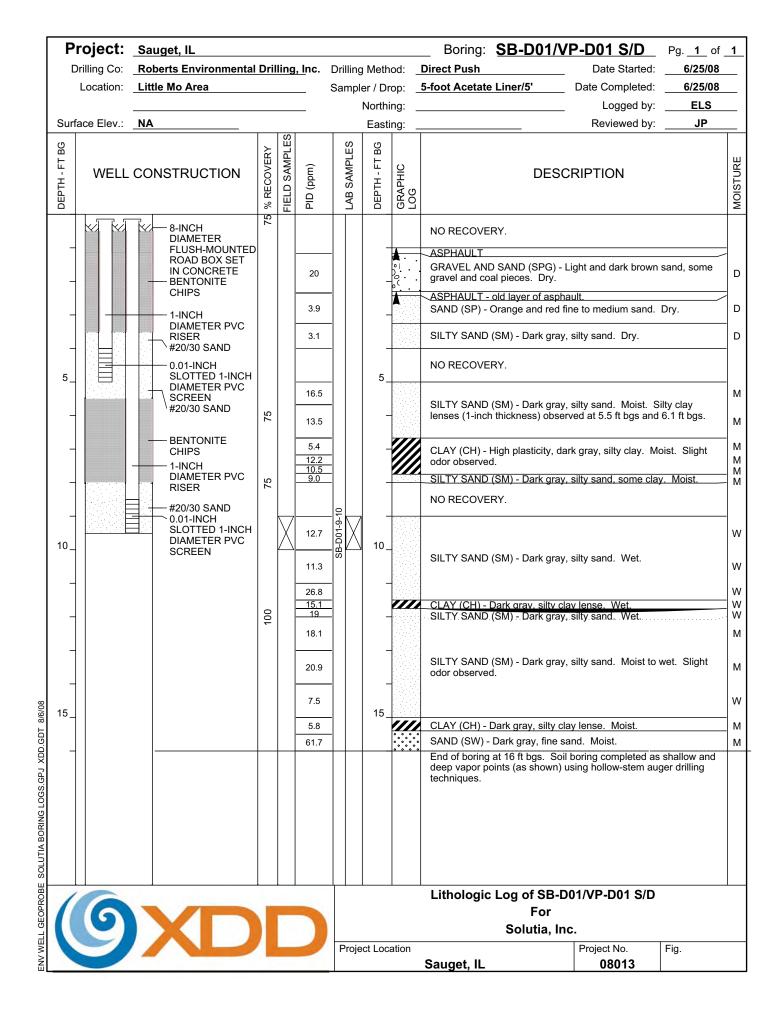
Proj	ect:	Sauget, IL							Boring: <b>SB-A04</b> Pg1 of		
							-		Direct Push Date Started: 6/24/08		
Loc	ation: _	Big Mo Area - SW cor	ner						5-foot Acetate Liner/5' Date Completed: 6/24/08 Logged by: ELS/OJ		
Surface	Elev.: _	NA				Easting:			Reviewed by:		
DEPTH			38 % RECOVERY	FIELD SAMPLES	PID (ppm)	LAB SAMPLES	DEPTH - FT BG	GRAPHIC LOG	DESCRIPTION		
			r r				-	-	NO RECOVERY.		
- 0 - 0					1.8	-	-		GRAVEL AND SAND (SPG) - Gravel fill and light gray, sand. Dry.		
0					1,480				COAL - Black coal pieces mixed with sand. Dry.		
5_ °	000	No vapor probe installed.	63				5		GRAVEL AND SAND (SPG) - Orange brick and rock pieces. Dry. NO RECOVERY.	/	
					3,227	_		o`· . 01	GRAVEL AND SAND (SPG) - Medium-coarse sand, trace gravel and brick pieces. Dry.	_	
					1,310				CLAY (CH) - Gray, silty clay. Moist.		
	0,00		75		45.5	_	-				
	000	2					-		NO RECOVERY.		
100					862		10_		CLAY (CH) - High plasticity, grayish-brown, silty clay. Moist.		
		0			2,810	-	-		SILTY SAND (SM) - Dark gray, fine sand, some silt. Wet.		
	0,00		100		4,056	-	-	••••• •••••			
	0 0	0			> 9,999	9	-		SAND (SW) - Dark gray, fine sand. Wet.		
15	00	0			3,173		15_		CLAY (CH) - High plasticity, dark gray, silty clay. Moist.		
	0°0	<u> </u>			2,070			•••••	SAND (SW) - Dark gray, fine sand. Wet.		
									End of boring at 16 ft bgs.		
									Lithologic Log of SB-A04		
	5	YC							For Solutia, Inc.		
	-					Proj	ect Loc	ation	Project No. Fig.		
									Sauget, IL 08013	_	

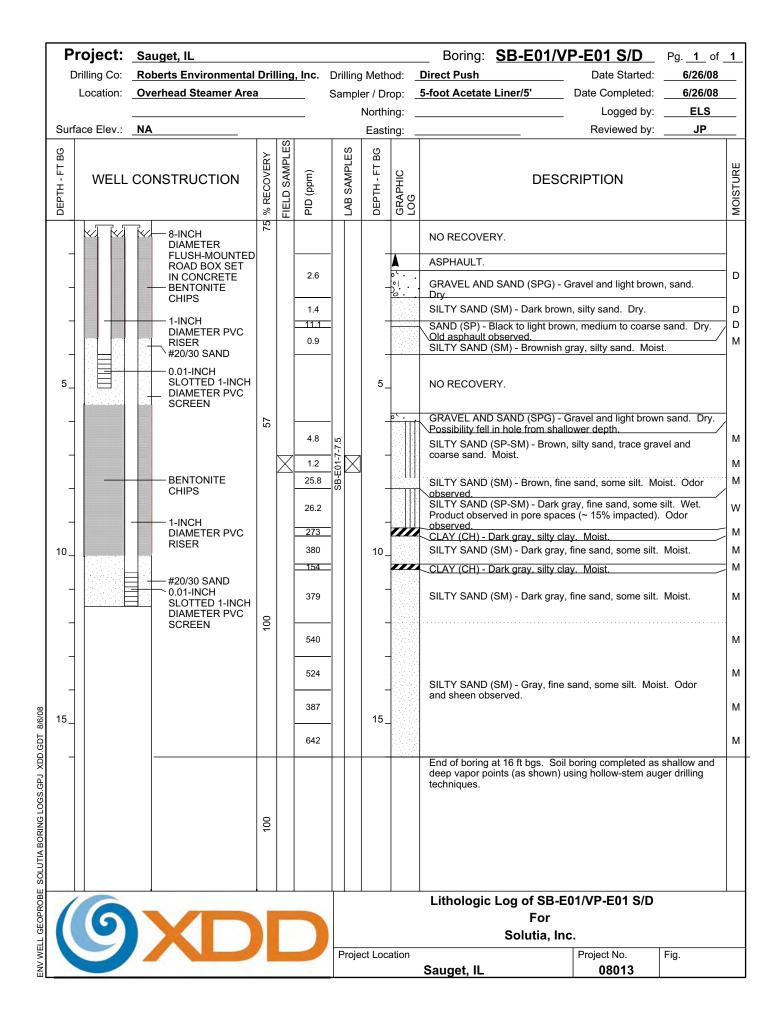
Project: <u>Sauget, IL</u> Drilling Co: <u>Roberts Environ</u>	ental Di	rillin	<u>g, I</u> nc.	Drilling	g Meth	od: _	-	SB-A05 Date Started:			
Location: <u>Big Mo Area - SE</u>								5' Date Completed: Logged by:	6/24/08 ELS/OJU		
Surface Elev.: NA				1	Easti	ng: _		Reviewed by:	JP	_	
DEPTH	WELL CONSTRUCTION WELL CONSTRUCTION					GRAPHIC LOG		DESCRIPTION			
	2 Y	00	1.0         10.6         475         666         621         1,061         > 9,999		- - - 5_ - - - - - - - - - - - - - - - -		Dry. COAL - Black coal pie SAND (SP-SC) - Ligh Fibrous material. NO RECOVERY. SILTY SAND (SP-SM Gravel at 6.5 ft bgs. ft SILTY SAND (SP-SM some silt. Wet. SILTY SAND (SP-SM some silt. Wet. SILTY SAND (SP-SM some silt. Wet. SILTY SAND (SP-SM coarse sand, some gr sitting on silty clay lay CLAY (CH) - Silty clay SAND (SW) - Gray, fti SILTY SAND (SM) - C sheen observed. CLAY (CH) - High pla	(SPG) - Gravel fill, with black ces mixed with medium-fine s t brown and orange sand, som ) - Gray, silty sand, some coa Moist to 6 ft bgs. Wet at 6.5 ft ) - Dark gray, medium to coar ) - Dark gray, medium to coar ) - Dark gray, medium to coar ) - Dark gray, silty sand, some avel. Wet. Significant layer of er below this layer. /. Wet. he sand. Moist. Gray, fine sand, some silt. We sticity, soft, gray, silty clay. We	sand. Dry. ne clay. Dry. rse sand. bgs. se sand, se sand, se sand, if pure phase 		
			)	Proje	oct Loca	ation	-	Log of SB-A05 For utia, Inc. Project No. 08013	Fig.		

C	Drilling Co: Location:	Bect:       Sauget, IL         ng Co:       Roberts Environmental Drilling, Inc.         cation:       Former Chlorobenzene Storage         Area					ampl	er / Dr Northi	op: _ ng: _	Direct Push 5-foot Acetate Li	ner/5'		6/26/08 6/26/08 ELS	1
DEPTH - FT BG	WELL CONSTRUCTION										JP	MOISTURE		
	5						Soil lithology described on boring log SB-B01/VP-B01 S/D. Vapor point VP-B01 I was installed in the high plasticity clay layer using hollow-stem auger drilling techniques.			01 S/D. city clay				
			)				Proje	ect Loca	ation	_	Log of SB-I For Solutia, Inc	B01/VP-B01 I S. Project No. 08013	Fig.	



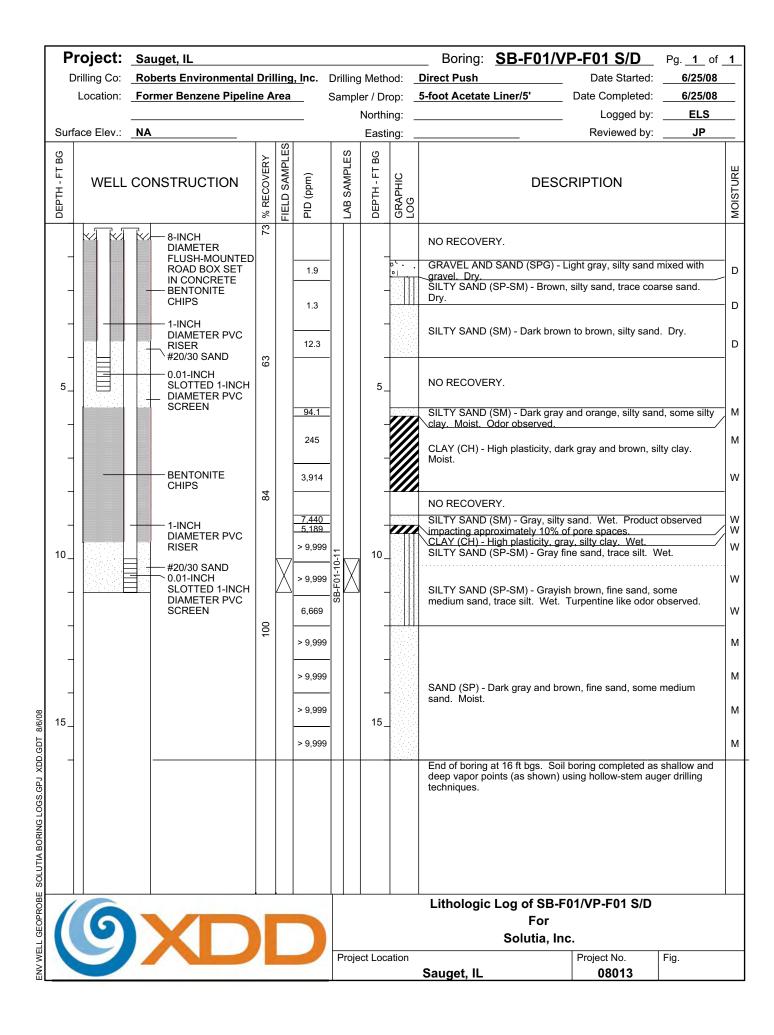






Pro	oject:	Sauget, IL							Boring: <b>SB-F0</b>	I/VP-F01 I	Pg. <u>1</u> of
	-	Roberts Environmental					-			_ Date Started: _	6/25/08
Lo	Location: Former Benzene Pipeline Area Sampler / Drop: 5-foot Acetate Liner/5' Date Northing:										
Surface	e Elev.:	NA			_		Easti	-			
BB				ES		S	BB	<u> </u>			
E I	WELL		% RECOVERY	FIELD SAMPLES	PID (ppm)	LAB SAMPLES	DEPTH - FT B	GRAPHIC LOG	DES	CRIPTION	
		<ul> <li>6-INCH DIAMETER FLUSH-MOUNTED ROAD BOX SET IN CONCRETE</li> <li>BENTONITE CHIPS</li> <li>1-INCH DIAMETER PVC RISER</li> <li>#20/30 SAND</li> <li>0.01-INCH SLOTTED 1-INCH DIAMETER PVC SCREEN</li> </ul>					- - 5_ -		Soil lithology described on bo Vapor point VP-F01 I was ins layer using hollow-stem auge	talled in the high plasticit r drilling techniques.	ty clay
	6								Lithologic Log of SI For Solutia, I		

# **US EPA ARCHIVE DOCUMENT**



S EPA ARCHIVE DOCUMENT



# ATTACHMENT C SOIL VOC LABORATORY ANALYTICAL DATA JOB NUMBER 680-38109-1

SB-A01-11'-12' SB-E01-7'-7.5' SB-F01-10'-11' SB-A03-11-12 SB-D01-9'-10' SB-B01-9'-10' SB-G01-9'10' (Duplicate of SB-D01-9'-10') Trip Blank

# APPENDIX A PRE-WORK PLAN DATA COLLECTION SUMMARY

SVE PILOT TESTING WORK PLAN

APPENDIX A: PRE-WORK PLAN DATA COLLECTION SUMMARY Soil Vapor Extraction Pilot Testing Work Plan



# ANALYTICAL REPORT

Job Number: 680-38109-1 SDG Number: KSX01 Job Description: WGK Point Permeability Soils 6/24-26/08

> For: Solutia Inc. 575 Maryville Centre Dr. Saint Louis, MO 63141 Attention: Mr. Bruce Yare

Lideja gricia

Lidya Gulizia Project Manager I lidya.gulizia@testamericainc.com 07/29/2008

c: Mr. Jerry Rinaldi Erin Stanisewski

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TestAmerica Laboratories, Inc. TestAmerica Savannah 5102 LaRoche Avenue, Savannah, GA 31404 Tel (912) 354-7858 Fax (912) 352-0165 www.testamericainc.com



#### Job Narrative 680-J38109-1 / SDG No. KSX01

#### Receipt

All samples were received intact ahnd within temperature requirements.

## GC/MS VOA

Method(s) 5035: The preserved volatile sample vials submitted for the project samples contained significantly greater than 5 grams for both the deionized and the methanol preserved samples. Please refer to the individual analysis sheets for the gram weights of the preserved samples.

Method(s) 8260B: Due to the submitted sample weights in the deionized (DI) preserved vials, the final results may be somewhat biased low due to possible poor purging efficiency as low-level analysis was performed on the DI vials for the following samples: SB-E01-7'-7.5' and SB-B01-9'-10' (680-38109-6).

Method(s) 8260B: Due to the level of dilution required for the following sample(s), surrogate recoveries are not reported: SB-A01-11'-12' (680-38109-1); SB-F01-10'-11' (680-38109-3), SB-A03-11-12 (680-38109-4).

No other analytical or quality issues were noted.

#### General Chemistry (Moisture)

No analytical or quality issues were noted.

**Comments** No additional comments.

## **METHOD SUMMARY**

Client: Solutia Inc.

Job Number: 680-38109-1 Sdg Number: KSX01

Description	Lab Location	Method	Preparation Method	
Matrix Solid				
Volatile Organic Compounds by GC/MS	TAL SAV	SW846 8260B		
Closed System Purge & Trap/Field Preservation	TAL SAV		SW846 5035	
M-4-1 14/-4				
Matrix Water				
Volatile Organic Compounds by GC/MS	TAL SAV	SW846 8260B		

#### Lab References:

TAL SAV = TestAmerica Savannah

## Method References:

SW846 = "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

## METHOD / ANALYST SUMMARY

Client: Solutia Inc.

Job Number: 680-38109-1 Sdg Number: KSX01

Method	Analyst	Analyst ID
SW846 8260B	LeSeane, Latika Rene	LL
SW846 8260B	Lui, Chung	CL

TestAmerica Savannah

# SAMPLE SUMMARY

Client: Solutia Inc.

Job Number: 680-38109-1 Sdg Number: KSX01

			Date/Time	Date/Time
Lab Sample ID	Client Sample ID	Client Matrix	Sampled	Received
680-38109-1	SB-A01-11'-12'	Solid	06/24/2008 1515	06/28/2008 0845
680-38109-2	SB-E01-7'-7.5'	Solid	06/26/2008 0930	06/28/2008 0845
680-38109-3	SB-F01-10'-11'	Solid	06/26/2008 1605	06/28/2008 0845
680-38109-4	SB-A03-11-12	Solid	06/26/2008 1600	06/28/2008 0845
680-38109-5	SB-D01-9'-10'	Solid	06/25/2008 1700	06/28/2008 0845
680-38109-6	SB-B01-9'-10'	Solid	06/26/2008 1400	06/28/2008 0845
680-38109-7	SB-G01-9'-10'	Solid	06/25/2008 1445	06/28/2008 0845
680-38109-8TB	Trip Blank	Water	06/26/2008 0000	06/28/2008 0845

# SAMPLE RESULTS

Client Sample ID:	SB-A01-11'-12	•					Sdg Number: k
Lab Sample ID:	680-38109-1					Date Sampled:	06/24/2008 1515
Client Matrix:	Solid	%	Moisture:	29.4		Date Received:	06/28/2008 0845
		8260B Volatile	Organic Co	mpounds	by GC/MS		
Method:	8260B	Analysis I	Batch: 680-1	10956	Ir	nstrument ID: 0	GC/MS Volatiles - M
Preparation:	5035		ch: 680-1104		L	.ab File ID: n	n0046.d
Dilution:	100000				lı	nitial Weight/Volume	e: 8.5 g
Date Analyzed:	07/08/2008 2328					inal Weight/Volume	-
Date Prepared:	07/01/2008 1459						5
Analyte		DryWt Corrected: Y	Result (ug/Kg	1)	Qualifier	MDL	RL
Acetone		-	1500000		JB	730000	8300000
Acetonitrile			33000000	1	U	7500000	33000000
Carbon disulfide			830000		U	200000	830000
Acrolein			17000000	1	U	3200000	17000000
Acrylonitrile			17000000	1	U	3800000	17000000
Benzene			13000000	1		130000	830000
Dichlorobromometha	ine		830000		U	140000	830000
Bromoform			830000		U	180000	830000
Bromomethane			830000		U	270000	830000
2-Butanone (MEK)			4200000		U	450000	4200000
Carbon tetrachloride			830000		U	170000	830000
Chlorobenzene			830000		U	120000	830000
Chloroethane			830000		U	200000	830000
Chloroform			830000		U	83000	830000
Chloromethane			830000		U *	120000	830000
-Chloro-1,3-butadie	ne		830000		U	95000	830000
-Chloro-1-propene			830000		U	250000	830000
Chlorodibromometha			830000		U	83000	830000
,2-Dibromo-3-Chlor	opropane		1700000		U	470000	1700000
Ethylene Dibromide			830000		U	250000	830000
bibromomethane	hutana		830000		U	200000	830000
rans-1,4-Dichloro-2- Dichlorodifluorometh			1700000		U	520000 150000	1700000 830000
,1-Dichloroethane	alle		830000 830000		U	83000	830000
,2-Dichloroethane			830000		U	170000	830000
,1-Dichloroethene			830000		U	90000	830000
,2-Dichlorobenzene	1		830000		U	110000	830000
rans-1,2-Dichloroeth			830000		U	160000	830000
,2-Dichloropropane			830000		U	180000	830000
,3-Dichlorobenzene	:		830000		U	140000	830000
,4-Dichlorobenzene			830000		U	85000	830000
is-1,3-Dichloroprop	ene		830000		U *	140000	830000
rans-1,3-Dichloropro			830000		U	140000	830000
thylbenzene			830000		U	120000	830000
thyl methacrylate			830000		U	370000	830000
2-Hexanone			4200000		U	350000	4200000
odomethane			830000		U	170000	830000
sobutyl alcohol			33000000		U	11000000	33000000
/lethacrylonitrile			17000000	1	U	4000000	17000000
Methylene Chloride			830000		U	170000	830000
lethyl methacrylate			830000		U	620000	830000
-Methyl-2-pentanon	e (MIBK)		4200000		U	480000	4200000
Pentachloroethane			4200000		U	370000	4200000
Propionitrile			17000000		U	3500000	17000000

Client: Solutia In	IC.			J	ob Number: 680-38109-1
Client Sample ID:	SB-A01-11'-12				Sdg Number: KSX01
Lab Sample ID:	680-38109-1			Date Sampled:	06/24/2008 1515
Client Matrix:	Solid	% Moisture: 29.4		Date Received:	06/28/2008 0845
		8260B Volatile Organic Compound	s by GC/MS		
Method:	8260B	Analysis Batch: 680-110956	Ir	nstrument ID: GC	C/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110469	L	ab File ID: m0	)046.d
Dilution:	100000		Ir	nitial Weight/Volume:	8.5 g
Date Analyzed:	07/08/2008 2328		F	inal Weight/Volume:	10 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifier	MDL	RL
Styrene		830000	U	110000	830000
1,1,1,2-Tetrachloroe	ethane	830000	U	110000	830000
1,1,2,2-Tetrachloroe	ethane	830000	U	230000	830000
Tetrachloroethene		830000	U	120000	830000
Toluene		830000	U	130000	830000
1,1,1-Trichloroethar	ne	830000	U	97000	830000
1,1,2-Trichloroethar	ne	830000	U	200000	830000
Trichloroethene		830000	U	170000	830000
Trichlorofluorometh	ane	830000	U	250000	830000
1,2,3-Trichloropropa	ane	830000	U	230000	830000
Vinyl acetate		1700000	U	250000	1700000
Vinyl chloride		830000	U	97000	830000
Xylenes, Total		1700000	U	380000	1700000
Surrogate		%Rec		•	ance Limits
4-Bromofluorobenzo		0		65 - 1	
Dibromofluorometha	ane	0		65 - 1	
Toluene-d8 (Surr)		0	65 - 132		

Client: Solutia Ind	С.				Job Number: 680-38109-
Client Sample ID:	SB-E01-7'-7.5'	,			Sdg Number: KSX0 <sup>-</sup>
Lab Sample ID: Client Matrix:	680-38109-2 Solid	% Moisture:	18.7	Date Sampled: Date Received	
		8260B Volatile Organic C	ompounds by GC/	MS	
Method:	8260B	Analysis Batch: 680-			GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110		Lab File ID:	m0075.d
Dilution:	1.0	·		Initial Weight/Volum	ie: 11.2 g
Date Analyzed:	07/10/2008 1806			Final Weight/Volum	
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/K			RL
Acetone		11	JB	2.4	27
Acetonitrile		110	U	25	110
Carbon disulfide		2.7	U	0.66	2.7
Acrolein		55	U	10	55
Acrylonitrile		55	U	13	55
Benzene		1.3	J	0.43	2.7
Dichlorobromometha	ane	2.7	U	0.46	2.7
Bromoform		2.7	U	0.60	2.7
Bromomethane		2.7	U	0.88	2.7
2-Butanone (MEK)		3.2	J	1.5	14
Carbon tetrachloride		2.7	U	0.55	2.7
Chlorobenzene		2.7	U	0.40	2.7
Chloroethane		2.7	U	0.66	2.7
Chloroform		2.7	U	0.27	2.7
Chloromethane		2.7	U	0.39	2.7
2-Chloro-1,3-butadie	ene	2.7	U	0.31	2.7
3-Chloro-1-propene		2.7	U	0.82	2.7
Chlorodibromometha		2.7	U	0.27	2.7
1,2-Dibromo-3-Chlor	opropane	5.5	U	1.5	5.5
Ethylene Dibromide		2.7	U	0.82	2.7
Dibromomethane		2.7	U	0.66	2.7
trans-1,4-Dichloro-2-		5.5	U	1.7	5.5
Dichlorodifluorometh	lane	2.7	U	0.49	2.7
1,1-Dichloroethane		2.7	U	0.27	2.7
1,2-Dichloroethane		2.7	U	0.55	2.7
1,1-Dichloroethene		2.7	U	0.30	2.7
1,2-Dichlorobenzene		2.7	U	0.36	2.7
trans-1,2-Dichloroeth		2.7	U	0.53	2.7
1,2-Dichloropropane		2.7	U	0.60	2.7
1,3-Dichlorobenzene		2.7	U	0.46	2.7
1,4-Dichlorobenzene		2.7	U	0.28	2.7
cis-1,3-Dichloroprop		2.7	U	0.48	2.7
trans-1,3-Dichloropro	opene	2.7	U	0.48	2.7
Ethylbenzene		2.7	U	0.41	2.7
Ethyl methacrylate		2.7	U	1.2	2.7
2-Hexanone		14	U	1.2	14
lodomethane		2.7	U	0.55	2.7
Isobutyl alcohol		110	U	38	110 55
Methacrylonitrile		55	U	13	55
Methylene Chloride		2.7	U	0.55	2.7
Methyl methacrylate		2.7	U	2.0	2.7
4-Methyl-2-pentanor	ie (IVIIBK)	14	U	1.6	14
Pentachloroethane		14	U	1.2	14
Propionitrile		55	U	12	55

Client: Solutia In	С.				Job Number: 680-38109-1 Sdg Number: KSX01
Client Sample ID:	SB-E01-7'-7.5				
Lab Sample ID:	680-38109-2			Date Sampled	: 06/26/2008 0930
Client Matrix:	Solid	% Moisture: 18.7		Date Received	1: 06/28/2008 0845
		8260B Volatile Organic Compour	ds by GC/I	NS	
Method:	8260B	Analysis Batch: 680-111218		Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110469		Lab File ID:	m0075.d
Dilution:	1.0			Initial Weight/Volum	ne: 11.2 g
Date Analyzed:	07/10/2008 1806			Final Weight/Volum	e: 5 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifi	er MDL	RL
Styrene		2.7	U	0.36	2.7
1,1,1,2-Tetrachloroe	ethane	2.7	U	0.35	2.7
1,1,2,2-Tetrachloroe	ethane	2.7	U	0.77	2.7
Tetrachloroethene		2.7	U	0.40	2.7
Toluene		2.7	U	0.43	2.7
1,1,1-Trichloroethan	ie	2.7	U	0.32	2.7
1,1,2-Trichloroethan	e	2.7	U	0.66	2.7
Trichloroethene		2.7	U	0.55	2.7
Trichlorofluorometha	ane	2.7	U	0.82	2.7
1,2,3-Trichloropropa	ane	2.7	U	0.77	2.7
Vinyl acetate		5.5	U	0.82	5.5
Vinyl chloride		2.7	U	0.32	2.7
Xylenes, Total		5.5	U	1.3	5.5
Surrogate		%Rec			eptance Limits
4-Bromofluorobenze		93			- 124
Dibromofluorometha	ane	86			- 124
Toluene-d8 (Surr)		86		65	- 132

Client: Solutia Ind	С.				Job Number: 680-38109-1 Sdg Number: KSX01
Client Sample ID:	SB-F01-10'-11'				·
Lab Sample ID:	680-38109-3			Date Sample	ed: 06/26/2008 1605
Client Matrix:	Solid	% Moisture:	15.8	Date Receive	ed: 06/28/2008 0845
		8260B Volatile Organic C	ompounds by G	GC/MS	
Method:	8260B	Analysis Batch: 680-	111225	Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110	469	Lab File ID:	m0077.d
Dilution:	2000			Initial Weight/Volu	ıme: 9.5 g
Date Analyzed:	07/10/2008 1851			Final Weight/Volu	
Date Prepared:	07/01/2008 1459			0	C C
Analyte		DryWt Corrected: Y Result (ug/K	g) Qu	alifier MDL	RL
Acetone		16000	J E	3 11000	120000
Acetonitrile		500000	U	110000	
Carbon disulfide		12000	U	3000	12000
Acrolein		250000	U	47000	250000
Acrylonitrile		250000	U	57000	250000
Benzene		40000		2000	12000
Dichlorobromometha	ane	12000	U	2100	12000
Bromoform		12000	U	2700	12000
Bromomethane		12000	U	4000	12000
2-Butanone (MEK)		62000	U	6700	62000
Carbon tetrachloride	!	12000	U	2500	12000
Chlorobenzene		230000		1800	12000
Chloroethane		12000	U '		12000
Chloroform		12000	U	1200	12000
Chloromethane		12000	U	1800	12000
2-Chloro-1,3-butadie	ene	12000	U U	1400 3700	12000
3-Chloro-1-propene	222	12000 12000	U	1200	12000 12000
Chlorodibromometha 1,2-Dibromo-3-Chlor		25000	U	7000	25000
Ethylene Dibromide	opioparie	12000	U	3700	12000
Dibromomethane		12000	U	3000	12000
trans-1,4-Dichloro-2-	-hutene	25000	U	7700	25000
Dichlorodifluorometh		12000	U	2200	12000
1,1-Dichloroethane		12000	U	1200	12000
1,2-Dichloroethane		12000	U	2500	12000
1,1-Dichloroethene		12000	U	1300	12000
1,2-Dichlorobenzene	9	12000	U	1600	12000
trans-1,2-Dichloroeth	nene	12000	U	2400	12000
1,2-Dichloropropane		12000	U	2700	12000
1,3-Dichlorobenzene	9	12000	U	2100	12000
1,4-Dichlorobenzene	9	12000	U	1300	12000
cis-1,3-Dichloroprop	ene	12000	U	2200	12000
trans-1,3-Dichloropro	opene	12000	U	2200	12000
Ethylbenzene		12000	U	1900	12000
Ethyl methacrylate		12000	U	5500	12000
2-Hexanone		62000	U	5200	62000
lodomethane		12000	U	2500	12000
Isobutyl alcohol		500000	U	170000	
Methacrylonitrile		250000	U	60000	250000
Methylene Chloride		12000	U	2500	12000
Methyl methacrylate		12000	U	9200 7200	12000
4-Methyl-2-pentanor		62000 62000	U	7200	62000
Pentachloroethane Propionitrile		62000 250000	U U	5500 52000	62000 250000
Propionitrile		250000	U	52000	200000

Client: Solutia In	С.			J	lob Number: 680-38109-1	
Client Sample ID:	SB-F01-10'-11	,			Sdg Number: KSX01	
Lab Sample ID:	680-38109-3			Date Sampled:	06/26/2008 1605	
Client Matrix:	Solid	% Moisture: 15.8		Date Received:	06/28/2008 0845	
		8260B Volatile Organic Compound	s by GC/MS	5		
Method:	8260B	Analysis Batch: 680-111225		Instrument ID: GO	C/MS Volatiles - M	
Preparation:	5035	Prep Batch: 680-110469		Lab File ID: m0	0077.d	
Dilution:	2000			Initial Weight/Volume:	9.5 g	
Date Analyzed:	07/10/2008 1851			Final Weight/Volume:	10 g	
Date Prepared:	07/01/2008 1459					
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifier	MDL	RL	
Styrene		12000	U	1600	12000	
1,1,1,2-Tetrachloroe	ethane	12000	U	1600	12000	
1,1,2,2-Tetrachloroe	ethane	12000	U	3500	12000	
Tetrachloroethene		12000	U	1800	12000	
Toluene		2100	J	2000	12000	
1,1,1-Trichloroethan	e	12000	U	1400	12000	
1,1,2-Trichloroethan	e	12000	U	3000	12000	
Trichloroethene		12000	U	2500	12000	
Trichlorofluorometha	ane	12000	U	3700	12000	
1,2,3-Trichloropropa	ane	12000	U	3500	12000	
Vinyl acetate		25000	U	3700	25000	
Vinyl chloride		12000	U	1400	12000	
Xylenes, Total		25000	U	5700	25000	
Surrogate		%Rec			ance Limits	
4-Bromofluorobenze		0	65 - 124			
Dibromofluorometha	ane	0		65 - 1		
Toluene-d8 (Surr)		0		65 - 1	32	

Client Sample ID:	SB-A03-11-12				Sdg Number: KS
Lab Sample ID:	680-38109-4			Date Sampled	: 06/26/2008 1600
Client Matrix:	Solid	% Moisture:	15.5	Date Received	
		8260B Volatile Organic Com	pounds by GC/	MS	
Method:	8260B	Analysis Batch: 680-11	1225	Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-11046		Lab File ID:	m0078.d
Dilution:	5000	· · · · · · · · · · · · · · · · · · ·		Initial Weight/Volun	ne: 9.1 g
Date Analyzed:	07/10/2008 1914			Final Weight/Volum	=
Date Prepared:	07/01/2008 1459				
Buter repured.					
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualif	ier MDL	RL
Acetone		41000	JB	29000	330000
Acetonitrile		1300000	U	290000	1300000
Carbon disulfide		33000	U	7800	33000
Acrolein		650000	U	120000	650000
Acrylonitrile		650000	U	150000	650000
Benzene		960000		5100	33000
Dichlorobromometha	ane	33000	U	5400	33000
Bromoform		33000	U	7200	33000
Bromomethane		33000	U	10000	33000
2-Butanone (MEK)		160000	U	18000	160000
Carbon tetrachloride		33000	U	6500	33000
Chlorobenzene		33000	U	4700	33000
Chloroethane		33000	U *	7800	33000
Chloroform		33000	U	3300	33000
Chloromethane		33000	U	4600	33000
2-Chloro-1,3-butadie	ne	33000	U	3700	33000
3-Chloro-1-propene		33000	U	9800	33000
Chlorodibromometha		33000	U	3300	33000
1,2-Dibromo-3-Chlor	opropane	65000	U	18000	65000
Ethylene Dibromide		33000	U	9800	33000
Dibromomethane		33000	U	7800	33000
rans-1,4-Dichloro-2-		65000	U	20000	65000
Dichlorodifluorometh	ane	33000	U	5800	33000
1,1-Dichloroethane		33000	U	3300	33000
1,2-Dichloroethane		33000	U	6500	33000
1,1-Dichloroethene 1,2-Dichlorobenzene		33000 33000	U U	3500 4200	33000 33000
rans-1,2-Dichloroeth		33000	U	6300	33000
1,2-Dichloropropane		33000	U	7200	33000
1,3-Dichlorobenzene		33000	U	5400	33000
1,4-Dichlorobenzene		33000	U	3300	33000
cis-1,3-Dichloroprop		33000	U	5700	33000
rans-1,3-Dichloropro		33000	U	5700	33000
Ethylbenzene		33000	U	4900	33000
Ethyl methacrylate		33000	U	14000	33000
2-Hexanone		17000	J	14000	160000
odomethane		33000	U	6500	33000
sobutyl alcohol		1300000	Ŭ	450000	1300000
Methacrylonitrile		650000	U	160000	650000
Methylene Chloride		33000	U	6500	33000
Methyl methacrylate		33000	U	24000	33000
4-Methyl-2-pentanor	e (MIBK)	160000	U	19000	160000
Pentachloroethane		160000	U	14000	160000
Propionitrile		650000	U	140000	650000

Client: Solutia In	С.			·	lob Number: 680-38109-1	
Client Sample ID:	SB-A03-11-12				Sdg Number: KSX01	
Lab Sample ID:	680-38109-4			Date Sampled:	06/26/2008 1600	
Client Matrix:	Solid	% Moisture: 15.5		Date Received:	06/28/2008 0845	
		8260B Volatile Organic Compound	ls by GC/M	s		
Method:	8260B	Analysis Batch: 680-111225		Instrument ID: G	C/MS Volatiles - M	
Preparation:	5035	Prep Batch: 680-110469		Lab File ID: m	0078.d	
Dilution:	5000			Initial Weight/Volume:	9.1 g	
Date Analyzed:	07/10/2008 1914			Final Weight/Volume:	10 g	
Date Prepared:	07/01/2008 1459					
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifie	r MDL	RL	
Styrene		33000	U	4300	33000	
1,1,1,2-Tetrachloroe	ethane	33000	U	4200	33000	
1,1,2,2-Tetrachloroe	ethane	33000	U	9100	33000	
Tetrachloroethene		33000	U	4700	33000	
Toluene		33000	U	5100	33000	
1,1,1-Trichloroethan	e	33000	U	3800	33000	
1,1,2-Trichloroethan	e	33000	U	7800	33000	
Trichloroethene		33000	U	6500	33000	
Trichlorofluorometha	ane	33000	U	9800	33000	
1,2,3-Trichloropropa	ane	33000	U	9100	33000	
Vinyl acetate		65000	U	9800	65000	
Vinyl chloride		33000	U	3800	33000	
Xylenes, Total		65000	U	15000	65000	
Surrogate		%Rec			ance Limits	
4-Bromofluorobenze		0	65 - 124			
Dibromofluorometha	ane	0	65 - 124			
Toluene-d8 (Surr)		0		65 - 1	32	

Client: Solutia Inc	<b>).</b>						Job Number: 680-3	
Client Sample ID:	SB-D01-9'-1	0'					Sdg Number:	KSX01
Lab Sample ID:	680-38109-5	5				Date Sampled:	06/25/2008 1700	
Client Matrix:	Solid		% Moisture:	22.6		Date Received:	06/28/2008 0845	
		82	860B Volatile Organic Co	ompounds	by GC/MS			
Method:	8260B		Analysis Batch: 680-				GC/MS Volatiles - M	
Preparation:	5035		Prep Batch: 680-110	469			n0055.d	
Dilution:	40					iitial Weight/Volume	-	
Date Analyzed:	07/09/2008 124				Fi	inal Weight/Volume	: 10 g	
Date Prepared:	07/01/2008 14	59						
Analyte			prrected: Y Result (ug/K	a)	Qualifier	MDL	RL	
Acetone		Diywico	360	9)	J	220	2500	
Acetonitrile			10000		U	2300	10000	
Carbon disulfide			250		U	61	250	
Acrolein			5100		U	960	5100	
Acrylonitrile			5100		U	1200	5100	
Benzene			80		J	40	250	
Dichlorobromometha	ne		250		U	40	250	
Bromoform			250		U	56	250	
Bromomethane			250		U	81	250	
2-Butanone (MEK)			1300		U	140	1300	
Carbon tetrachloride			250		U	51	250	
Chlorobenzene			520		0	37	250	
Chloroethane			250		U	61	250	
Chloroform			250		U	25	250	
Chloromethane			250		U	36	250	
2-Chloro-1,3-butadie	ne		250		U	29	250	
3-Chloro-1-propene			250		U	76	250	
Chlorodibromometha	ine		250		U	25	250	
1,2-Dibromo-3-Chlor			510		U	140	510	
Ethylene Dibromide	opropune		250		U	76	250	
Dibromomethane			250		U	61	250	
trans-1,4-Dichloro-2-	hutene		510		U	160	510	
Dichlorodifluorometh			250		U	45	250	
1,1-Dichloroethane	une		250		U	25	250	
1,2-Dichloroethane			250		U	51	250	
1,1-Dichloroethene			250		U	27	250	
1,2-Dichlorobenzene			250		U	33	250	
trans-1,2-Dichloroeth			250		U	49	250	
1,2-Dichloropropane			250		U	56	250	
1,3-Dichlorobenzene			250		U	42	250	
1,4-Dichlorobenzene			250		U	26	250	
cis-1,3-Dichloroprope			250		U *	44	250	
trans-1,3-Dichloropro			250		U	44	250	
Ethylbenzene			250		U	38	250	
Ethyl methacrylate			250		U	110	250	
2-Hexanone			1300		U	110	1300	
Iodomethane			250		U	51	250	
Isobutyl alcohol			10000		U	3500	10000	
Methacrylonitrile			5100		U	1200	5100	
Methylene Chloride			250		U	51	250	
Methyl methacrylate			250		U	190	250	
			1300		U	150	1300	
4-Methyl-2-pentanon			1000		0			
4-Methyl-2-pentanon Pentachloroethane			1300		U	110	1300	

Client: Solutia In	С.				Job Number: 680-38109-1
Client Sample ID:	SB-D01-9'-10'				Sdg Number: KSX01
Lab Sample ID:	680-38109-5			Date Sampled	1: 06/25/2008 1700
Client Matrix:	Solid	% Moisture: 22.6		Date Receive	d: 06/28/2008 0845
		8260B Volatile Organic Compound	s by GC/M	S	
Method:	8260B	Analysis Batch: 680-111103		Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110469		Lab File ID:	m0055.d
Dilution:	40			Initial Weight/Volur	ne: 10.2 g
Date Analyzed:	07/09/2008 1248			Final Weight/Volum	ne: 10 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifie	r MDL	RL
Styrene		250	U	33	250
1,1,1,2-Tetrachloroe	ethane	250	U	32	250
1,1,2,2-Tetrachloroe	ethane	250	U	71	250
Tetrachloroethene		250	U	37	250
Toluene		250	U	40	250
1,1,1-Trichloroethan	ie	250	U	29	250
1,1,2-Trichloroethan	e	250	U	61	250
Trichloroethene		250	U	51	250
Trichlorofluorometha	ane	250	U	76	250
1,2,3-Trichloropropa	ane	250	U	71	250
Vinyl acetate		510	U	76	510
Vinyl chloride		250	U	29	250
Xylenes, Total		510	U	120	510
Surrogate		%Rec			eptance Limits
4-Bromofluorobenze		112			5 - 124
Dibromofluorometha	ane	100			5 - 124
Toluene-d8 (Surr)		102		65	5 - 132

**US EPA ARCHIVE DOCUMENT** 

Client: Solutia Ind	С.				Job Number: 680-38109-1
Client Sample ID:	SB-B01-9'-10'				Sdg Number: KSX01
Lab Sample ID:	680-38109-6			Date Sampled	
Client Matrix:	Solid	% Moisture:	26.0	Date Received	1: 06/28/2008 0845
		8260B Volatile Organic Co	ompounds by GC/N	MS	
Method:	8260B	Analysis Batch: 680-7	111218	Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-1104	469	Lab File ID:	m0079.d
Dilution:	1.0			Initial Weight/Volum	ne: 9.9 g
Date Analyzed:	07/10/2008 1936			Final Weight/Volum	ie: 5 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg		-	RL
Acetone		4.3	JB	3.0	34
		140	U	31	140
Carbon disulfide		3.4	U	0.82	3.4
Acrolein		68	U	13	68
Acrylonitrile		68	U	16	68
Benzene		1.8	J	0.54	3.4
Dichlorobromometha	ane	3.4	U	0.57	3.4
Bromoform		3.4	U	0.75	3.4
Bromomethane		3.4	U	1.1	3.4
2-Butanone (MEK)		17	U	1.8	17
Carbon tetrachloride		3.4	U	0.68	3.4
Chlorobenzene		2.9	J	0.50	3.4
Chloroethane		3.4	U	0.82	3.4
Chloroform		3.4	U	0.34	3.4
Chloromethane		3.4	U	0.48	3.4
2-Chloro-1,3-butadie	ene	3.4	U	0.39	3.4
3-Chloro-1-propene		3.4	U	1.0	3.4
Chlorodibromometha		3.4	U	0.34	3.4
1,2-Dibromo-3-Chlor	opropane	6.8	U	1.9	6.8
Ethylene Dibromide		3.4	U	1.0	3.4
Dibromomethane		3.4	U	0.82	3.4
trans-1,4-Dichloro-2-		6.8	U	2.1	6.8
Dichlorodifluorometh	lane	3.4	U	0.61	3.4
1,1-Dichloroethane		3.4	U	0.34	3.4
1,2-Dichloroethane		3.4	U	0.68	3.4
1,1-Dichloroethene		3.4	U	0.37	3.4
1,2-Dichlorobenzene		3.4	U	0.44	3.4
trans-1,2-Dichloroeth		3.4	U	0.66	3.4
1,2-Dichloropropane		3.4	U	0.75	3.4
1,3-Dichlorobenzene		3.4	U	0.57	3.4
1,4-Dichlorobenzene		3.4	U	0.35	3.4
cis-1,3-Dichloroprop		3.4	U	0.59	3.4
trans-1,3-Dichloropro	opene	3.4	U	0.59	3.4
Ethylbenzene		3.4	U	0.51	3.4
Ethyl methacrylate		3.4 17	U U	1.5	3.4
2-Hexanone Iodomethane				1.4	17
		3.4 140	U U	0.68 47	3.4 140
Isobutyl alcohol		68	U	47 16	68
Methacrylonitrile		3.4	U	0.68	3.4
Methylene Chloride					
Methyl methacrylate		3.4	U	2.5 2.0	3.4
4-Methyl-2-pentanor		17	U		17
Pentachloroethane Propionitrile		17 68	U U	1.5 14	17 68

Client: Solutia In	С.				Job Number: 680-38109-1
Client Sample ID:	SB-B01-9'-10'				Sdg Number: KSX01
Lab Sample ID:	680-38109-6			Date Sampled:	06/26/2008 1400
Client Matrix:	Solid	% Moisture: 26.0		Date Received:	06/28/2008 0845
		8260B Volatile Organic Compou	nds by GC/I	MS	
Method:	8260B	Analysis Batch: 680-111218	3	Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110469		Lab File ID:	m0079.d
Dilution:	1.0			Initial Weight/Volume	e: 9.9 g
Date Analyzed:	07/10/2008 1936			Final Weight/Volume	e: 5 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifi	ier MDL	RL
Styrene		3.4	U	0.45	3.4
1,1,1,2-Tetrachloroe	ethane	3.4	U	0.44	3.4
1,1,2,2-Tetrachloroe	ethane	3.4	U	0.96	3.4
Tetrachloroethene		3.4	U	0.50	3.4
Toluene		3.4	U	0.54	3.4
1,1,1-Trichloroethan	ie	3.4	U	0.40	3.4
1,1,2-Trichloroethan	ie	3.4	U	0.82	3.4
Trichloroethene		3.4	U	0.68	3.4
Trichlorofluorometha	ane	3.4	U	1.0	3.4
1,2,3-Trichloropropa	ane	3.4	U	0.96	3.4
Vinyl acetate		6.8	U	1.0	6.8
Vinyl chloride		3.4	U	0.40	3.4
Xylenes, Total		6.8	U	1.6	6.8
Surrogate		%Rec			ptance Limits
4-Bromofluorobenze		89			- 124
Dibromofluorometha	ane	89			- 124
Toluene-d8 (Surr)		85		65 -	· 132

Client: Solutia Inc	).				Job Number: 680-38109-1
Client Sample ID:	SB-G01-9'-10'				Sdg Number: KSX01
Lab Sample ID:	680-38109-7			Date Sampled	
Client Matrix:	Solid	% Moisture:	22.1	Date Received	1: 06/28/2008 0845
		8260B Volatile Organic	Compounds by GC/I	MS	
Method:	8260B	Analysis Batch: 680		Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-11	0469	Lab File ID:	m0056.d
Dilution:	40			Initial Weight/Volum	ne: 11.3 g
Date Analyzed:	07/09/2008 1311			Final Weight/Volum	ie: 10 g
Date Prepared:	07/01/2008 1459				
Analyta					
Analyte		DryWt Corrected: Y Result (ug/			RL
Acetone		260	J	200	2300
Acetonitrile		9100	U	2000	9100
Carbon disulfide		230	U	55	230
Acrolein		4500	U	860	4500
Acrylonitrile		4500	U	1000	4500
Benzene		92	J	36	230
Dichlorobromometha	ine	230	U	38	230
Bromoform		230	U	50	230
Bromomethane		230	U	73	230
2-Butanone (MEK)		1100	U	120	1100
Carbon tetrachloride		230	U	45	230
Chlorobenzene		300		33	230
Chloroethane		230	U	55	230
Chloroform		230	U	23	230
Chloromethane		230	U	32	230
2-Chloro-1,3-butadie	ne	230	U	26	230
3-Chloro-1-propene		230	U	68	230
Chlorodibromometha		230	U	23	230
1,2-Dibromo-3-Chlor	opropane	450	U	130	450
Ethylene Dibromide		230	U	68	230
Dibromomethane		230	U	55	230
trans-1,4-Dichloro-2-		450	U	140	450
Dichlorodifluorometh	ane	230	U	40	230
1,1-Dichloroethane		230	U	23	230
1,2-Dichloroethane		230	U	45	230
1,1-Dichloroethene		230	U	25	230
1,2-Dichlorobenzene		230	U	30	230
trans-1,2-Dichloroeth	iene	230	U	44	230
1,2-Dichloropropane		230	U	50	230
1,3-Dichlorobenzene		230	U	38	230
1,4-Dichlorobenzene		230	U	23	230
cis-1,3-Dichloroprope		230	U *	40	230
trans-1,3-Dichloropro	pene	230	U	40	230
Ethylbenzene		230	U	34	230
Ethyl methacrylate		230	U	100	230
2-Hexanone		1100	U	95	1100
Iodomethane		230	U	45	230
Isobutyl alcohol		9100	U	3100	9100
Methacrylonitrile		4500	U	1100	4500
Methylene Chloride		230	U	45	230
				170	
Methyl methacrylate		230	U	170	230
4-Methyl-2-pentanon	e (MIBK)	1100	U	130	1100
	e (MIBK)				

Client: Solutia In	С.				Job Number: 680-38109-1
Client Sample ID:	SB-G01-9'-10'				Sdg Number: KSX01
Lab Sample ID:	680-38109-7			Date Sampled	1: 06/25/2008 1445
Client Matrix:	Solid	% Moisture: 22.1		Date Received	d: 06/28/2008 0845
		8260B Volatile Organic Compound	ds by GC/M	S	
Method:	8260B	Analysis Batch: 680-111103		Instrument ID:	GC/MS Volatiles - M
Preparation:	5035	Prep Batch: 680-110469		Lab File ID:	m0056.d
Dilution:	40			Initial Weight/Volun	ne: 11.3 g
Date Analyzed:	07/09/2008 1311			Final Weight/Volum	ne: 10 g
Date Prepared:	07/01/2008 1459				
Analyte		DryWt Corrected: Y Result (ug/Kg)	Qualifie	er MDL	RL
Styrene		230	U	30	230
1,1,1,2-Tetrachloroe	ethane	230	U	29	230
1,1,2,2-Tetrachloroe	ethane	230	U	64	230
Tetrachloroethene		230	U	33	230
Toluene		230	U	36	230
1,1,1-Trichloroethar	ie	230	U	26	230
1,1,2-Trichloroethar	ie	230	U	55	230
Trichloroethene		230	U	45	230
Trichlorofluorometha	ane	230	U	68	230
1,2,3-Trichloropropa	ane	230	U	64	230
Vinyl acetate		450	U	68	450
Vinyl chloride		230	U	26	230
Xylenes, Total		450	U	100	450
Surrogate		%Rec			eptance Limits
4-Bromofluorobenze		121			5 - 124
Dibromofluorometha	ane	102			5 - 124
Toluene-d8 (Surr)		111		65	5 - 132

Olient Course 1: 10					Sdg Number: KS
Client Sample ID:	Trip Blank				00/00/0000 0000
Lab Sample ID: Client Matrix:	680-38109-8TB Water			Date Sampled: Date Received:	06/26/2008 0000 06/28/2008 0845
		8260B Volatile Organic Compound	s by GC/M	S	
Method:	8260B	Analysis Batch: 680-111182			GC/MS Volatiles - P C2
Preparation:	5030B				p1130.d
Dilution:	1.0			Initial Weight/Volume	
	07/10/2008 1402				
Date Analyzed:	07/10/2008 1402			Final Weight/Volume	. JIL
Date Prepared:	07/10/2006 1402				
Analyte		Result (ug/L)	Qualifie	r MDL	RL
Acetone		8.9	J	5.0	25
Acetonitrile		40	U	15	40
Acrolein		20	U	18	20
Acrylonitrile		20	U	3.8	20
Benzene		1.0	U	0.32	1.0
Bromodichlorometha	ane	1.0	U	0.34	1.0
Bromoform		0.60	J	0.41	1.0
Bromomethane		1.0	Ŭ	0.50	1.0
2-Butanone		4.3	J	0.60	10
Carbon disulfide		2.0	Ŭ	0.60	2.0
Carbon tetrachloride	<b>`</b>	1.0	U	0.27	1.0
Chlorobenzene	•	1.0	U	0.34	1.0
Chloroprene		1.0	U	0.35	1.0
Chloroethane		1.0	U	1.0	1.0
Chloroform		1.0	U	0.29	1.0
Chloromethane		1.0	U	0.28	1.0
3-Chloro-1-propene		1.0	U	0.20	1.0
Dibromochlorometh	220	0.54	J	0.30	1.0
1,2-Dibromo-3-Chlo		1.0	U	0.48	1.0
1,2-Dibromoethane	Toproparie	1.0	U	0.30	1.0
Dibromomethane		1.0	U	0.30	1.0
1,2-Dichlorobenzen		1.0	UU	0.33	1.0
,3-Dichlorobenzen		1.0	•	0.31	1.0
,4-Dichlorobenzen		1.0	U	0.33	1.0
rans-1,4-Dichloro-2		2.0	U	0.83	2.0
Dichlorodifluorometh	lane	1.0	U	0.33	1.0
I,1-Dichloroethane		1.0	U	0.32	1.0
I,2-Dichloroethane	h	1.0	U	0.31	1.0
rans-1,2-Dichloroet	nene	1.0	U	0.30	1.0
I,1-Dichloroethene		1.0	U	0.36	1.0
1,2-Dichloropropane		1.0	U	0.36	1.0
cis-1,3-Dichloroprop		1.0	U	0.37	1.0
rans-1,3-Dichloropr	opene	1.0	U	0.27	1.0
		1.0	U	0.30	1.0
Ethyl methacrylate		1.0	U	1.0	1.0
2-Hexanone		10	U	0.68	10
odomethane		5.0	U	1.0	5.0
sobutanol		40	U	19	40
Aethacrylonitrile		20	U	6.6	20
Methylene Chloride		5.0	U	1.0	5.0
Methyl methacrylate		1.0	U	0.38	1.0
I-Methyl-2-pentanor	ne	10	U	0.60	10
Pentachloroethane		5.0	U	1.3	5.0
Propionitrile		20	U	9.2	20

Client: Solutia In	С.				Job Number: 680-38109-1 Sdg Number: KSX01
Client Sample ID:	Trip Blank				
Lab Sample ID: Client Matrix:	680-38109-8TB Water			Date Sampled: Date Received:	06/26/2008 0000 06/28/2008 0845
		8260B Volatile Organic Compound	s by GC/M	s	
Method:	8260B 5030B	Analysis Batch: 680-111182			C/MS Volatiles - P C2
Preparation: Dilution:	5030B 1.0			Initial Weight/Volume:	1130.d 5 mL
Date Analyzed:	07/10/2008 1402			Final Weight/Volume:	5 mL
Date Prepared:	07/10/2008 1402			r indi vvelgno volume.	o me
Analyte		Result (ug/L)	Qualifie	r MDL	RL
Styrene		1.0	U	0.36	1.0
1,1,1,2-Tetrachloroe	ethane	1.0	U	0.29	1.0
1,1,2,2-Tetrachloroe	ethane	1.0	U	0.26	1.0
Tetrachloroethene		1.0	U	0.28	1.0
Toluene		1.0	U	0.31	1.0
1,1,1-Trichloroethan		1.0	U	0.39	1.0
1,1,2-Trichloroethan	le	1.0	U	0.51	1.0
Trichloroethene		1.0	U	0.40	1.0
Trichlorofluorometha		1.0	U	0.29	1.0
1,2,3-Trichloropropa	ane	1.0	U	0.42	1.0
Vinyl acetate		2.0	U	0.62	2.0
√inyl chloride Xylenes, Total		1.0 2.0	U U	0.20 0.87	1.0 2.0
Surrogate		%Rec		Accept	ance Limits
4-Bromofluorobenze	ene	94		75 - 7	120
Dibromofluorometha	ane	100		75 - 1	
Toluene-d8 (Surr)		99		75 - 1	120

## DATA REPORTING QUALIFIERS

Client: Solutia Inc.

Job Number: 680-38109-1 Sdg Number: KSX01

Lab Section	Qualifier	Description
GC/MS VOA		
	В	Compound was found in the blank and sample.
	U	Indicates the analyte was analyzed for but not detected.
	*	LCS or LCSD exceeds the control limits
	J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.

# **QUALITY CONTROL RESULTS**

Job Number: 680-38109-1 Sdg Number: KSX01

Client: Solutia Inc.

# **QC Association Summary**

		Report			
Lab Sample ID	Client Sample ID	Basis	Client Matrix	Method	Prep Batch
GC/MS VOA					
Prep Batch: 680-110469					
680-38109-1	SB-A01-11'-12'	Т	Solid	5035	
680-38109-2	SB-E01-7'-7.5'	Т	Solid	5035	
680-38109-3	SB-F01-10'-11'	Т	Solid	5035	
580-38109-4	SB-A03-11-12	Т	Solid	5035	
80-38109-5	SB-D01-9'-10'	Т	Solid	5035	
80-38109-6	SB-B01-9'-10'	Т	Solid	5035	
80-38109-7	SB-G01-9'-10'	Т	Solid	5035	
Analysis Batch:680-1109	56				
_CS 680-110956/10	Lab Control Spike	Т	Solid	8260B	
/IB 680-110956/4	Method Blank	Т	Solid	8260B	
680-38109-1	SB-A01-11'-12'	Т	Solid	8260B	680-110469
Analysis Batch:680-1111	03				
CS 680-111103/14	Lab Control Spike	Т	Solid	8260B	
/IB 680-111103/12	Method Blank	Т	Solid	8260B	
80-38109-5	SB-D01-9'-10'	т	Solid	8260B	680-110469
80-38109-7	SB-G01-9'-10'	Т	Solid	8260B	680-110469
Analysis Batch:680-1111	82				
CS 680-111182/6	Lab Control Spike	Т	Water	8260B	
.CSD 680-111182/7	Lab Control Spike Duplicate	т	Water	8260B	
/IB 680-111182/9	Method Blank	т	Water	8260B	
80-38109-8TB	Trip Blank	Т	Water	8260B	
Analysis Batch:680-1112	18				
.CS 680-111218/5	Lab Control Spike	Т	Solid	8260B	
/IB 680-111218/6	Method Blank	Т	Solid	8260B	
80-38109-2	SB-E01-7'-7.5'	т	Solid	8260B	680-110469
80-38109-6	SB-B01-9'-10'	Т	Solid	8260B	680-110469
Analysis Batch:680-1112	25				
.CS 680-111225/4	Lab Control Spike	Т	Solid	8260B	
/IB 680-111225/5	Method Blank	т	Solid	8260B	
80-38109-3	SB-F01-10'-11'	т	Solid	8260B	680-110469
580-38109-4	SB-A03-11-12	т	Solid	8260B	680-110469

Report Basis

T = Total

# **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## **Surrogate Recovery Report**

## 8260B Volatile Organic Compounds by GC/MS

## Client Matrix: Solid

		BFB	DBFM	TOL
Lab Sample ID	Client Sample ID	%Rec	%Rec	%Rec
680-38109-1	SB-A01-11'-12'	0	0	0
680-38109-2	SB-E01-7'-7.5'	93	86	86
680-38109-3	SB-F01-10'-11'	0	0	0
680-38109-4	SB-A03-11-12	0	0	0
680-38109-5	SB-D01-9'-10'	112	100	102
680-38109-6	SB-B01-9'-10'	89	89	85
680-38109-7	SB-G01-9'-10'	121	102	111
MB 680-110956/4		92	87	82
MB 680-111103/12		84	86	73
MB 680-111218/6		95	94	88
MB 680-111225/5		80	74	70
LCS 680-110956/10		111	120	114
LCS 680-111103/14		108	110	98
LCS 680-111218/5		93	88	91
LCS 680-111225/4		104	100	92

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Surrogate	Acceptance Limits
BFB = 4-Bromofluorobenzene	65-124
DBFM = Dibromofluoromethane	65-124
TOL = Toluene-d8 (Surr)	65-132

Job Number: 680-38109-1 Sdg Number: KSX01

## **Surrogate Recovery Report**

## 8260B Volatile Organic Compounds by GC/MS

## Client Matrix: Water

		BFB	DBFM	TOL
Lab Sample ID	Client Sample ID	%Rec	%Rec	%Rec
680-38109-8	Trip Blank	94	100	99
MB 680-111182/9		96	98	96
LCS 680-111182/6		96	100	100
LCSD 680-111182/7		95	100	96

Surrogate	Acceptance Limits
BFB = 4-Bromofluorobenzene	75-120
DBFM = Dibromofluoromethane	75-121
TOL = Toluene-d8 (Surr)	75-120

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method Blank - Batch: 680-110956

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-110956/4	Analysis Batch: 680-110956	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq019.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/08/2008 2019		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
Acetone	230	J	180	2000
Acetonitrile	8000	U	1800	8000
Acrolein	4000	U	760	4000
Acrylonitrile	4000	U	920	4000
Benzene	200	U	32	200
Carbon disulfide	200	U	48	200
Dichlorobromomethane	200	U	33	200
Bromoform	200	U	44	200
Bromomethane	200	U	64	200
2-Butanone (MEK)	1000	U	110	1000
Carbon tetrachloride	200	U	40	200
Chlorobenzene	200	U	29	200
Chloroethane	200	U	48	200
Chloroform	200	U	20	200
Chloromethane	200	U	28	200
2-Chloro-1,3-butadiene	200	U	23	200
3-Chloro-1-propene	200	U	60	200
Chlorodibromomethane	200	U	20	200
1,2-Dibromo-3-Chloropropane	400	U	110	400
Ethylene Dibromide	200	U	60	200
Dibromomethane	200	U	48	200
trans-1.4-Dichloro-2-butene	400	U	120	400
Dichlorodifluoromethane	200	U	36	200
1,1-Dichloroethane	200	U	20	200
1,2-Dichloroethane	200	U	40	200
1,1-Dichloroethene	200	U	22	200
1,2-Dichlorobenzene	200	U	26	200
trans-1,2-Dichloroethene	200	U	39	200
1,2-Dichloropropane	200	Ŭ	44	200
1,3-Dichlorobenzene	200	U	33	200
1,4-Dichlorobenzene	200	U	20	200
cis-1,3-Dichloropropene	200	U	35	200
trans-1,3-Dichloropropene	200	U	35	200
Ethylbenzene	200	U	30	200
Ethyl methacrylate	200	U	88	200
2-Hexanone	1000	U	84	1000
lodomethane	200	U	40	200
Isobutyl alcohol	8000	U	2800	8000
Methacrylonitrile	4000	U	960	4000
Methylene Chloride	200	U	40	200
Methyl methacrylate	200	U	40 150	200

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

# Method Blank - Batch: 680-110956

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-110956/4	Analysis Batch: 680-110956	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq019.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/08/2008 2019		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
4-Methyl-2-pentanone (MIBK)	1000	U	120	1000
Pentachloroethane	1000	U	88	1000
Propionitrile	4000	U	840	4000
Styrene	200	U	26	200
1,1,1,2-Tetrachloroethane	200	U	26	200
1,1,2,2-Tetrachloroethane	200	U	56	200
Tetrachloroethene	200	U	29	200
Toluene	200	U	32	200
1,1,1-Trichloroethane	200	U	23	200
1,1,2-Trichloroethane	200	U	48	200
Trichloroethene	200	U	40	200
Trichlorofluoromethane	200	U	60	200
1,2,3-Trichloropropane	200	U	56	200
Vinyl acetate	400	U	60	400
Vinyl chloride	200	U	23	200
Xylenes, Total	400	U	92	400
Surrogate	% Rec		Acceptance Limits	
4-Bromofluorobenzene	92		65 - 124	
Dibromofluoromethane	87		65 - 124	
Toluene-d8 (Surr)	82		65 - 132	

Client Matrix:

Date Analyzed: Date Prepared:

Dilution:

**US EPA ARCHIVE DOCUMENT** 

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Lab Control Spike - Batch: 680-110956

Lab Sample ID: LCS 680-110956/10

Solid

07/08/2008 2357

40

N/A

## Method: 8260B Preparation: N/A

Instrument ID:	GC/MS V	olatiles - M
Lab File ID:	mq020.d	
Initial Weight/Ve	olume: 5	g
Final Weight/Vo	olume: 5	mL

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Acetone	5000	3780	76	16 - 202	
Benzene	2500	2500	100	63 - 130	
Carbon disulfide	2500	2300	92	46 - 134	
Dichlorobromomethane	2500	1760	70	64 - 137	
Bromoform	2500	2080	83	66 - 127	
Bromomethane	2500	2930	117	54 - 146	
2-Butanone (MEK)	5000	3850	77	19 - 192	
Carbon tetrachloride	2500	2220	89	60 - 136	
Chlorobenzene	2500	2730	109	77 - 120	
Chloroethane	2500	1050	42	26 - 166	
Chloroform	2500	3000	120	68 - 127	
Chloromethane	2500	3680	147	46 - 137	*
Chlorodibromomethane	2500	1980	79	70 - 126	
1,2-Dibromo-3-Chloropropane	2500	1550	62	62 - 140	
Ethylene Dibromide	2500	2030	81	61 - 138	
Dibromomethane	2500	1940	77	61 - 138	
Dichlorodifluoromethane	2500	2060	83	17 - 163	
1,1-Dichloroethane	2500	2810	112	65 - 130	
1,2-Dichloroethane	2500	2040	82	62 - 140	
1,1-Dichloroethene	2500	2240	90	59 - 137	
1,2-Dichlorobenzene	2500	2870	115	75 - 123	
trans-1,2-Dichloroethene	2500	2080	83	66 - 127	
1,2-Dichloropropane	2500	1890	76	66 - 135	
1,3-Dichlorobenzene	2500	2700	108	74 - 123	
1.4-Dichlorobenzene	2500	2570	103	75 - 122	
cis-1,3-Dichloropropene	2500	1270	51	66 - 137	*
trans-1,3-Dichloropropene	2500	1800	72	64 - 138	
Ethylbenzene	2500	2810	112	77 - 121	
2-Hexanone	5000	4030	81	47 - 151	
Methylene Chloride	2500	1950	78	65 - 126	
4-Methyl-2-pentanone (MIBK)	5000	2830	57	50 - 148	
Styrene	2500	2710	108	75 - 123	
1,1,1,2-Tetrachloroethane	2500	2460	98	72 - 124	
1,1,2,2-Tetrachloroethane	2500	2030	81	65 - 130	
Tetrachloroethene	2500	2890	116	76 - 120	
Toluene	2500	2870	115	67 - 132	
1,1,1-Trichloroethane	2500	2230	89	56 - 140	
1,1,2-Trichloroethane	2500	1890	76	62 - 138	
Trichloroethene	2500	2120	85	68 - 133	
Trichlorofluoromethane	2500	2460	98	33 - 152	
1,2,3-Trichloropropane	2500	2130	85	65 - 132	

Analysis Batch: 680-110956

Prep Batch: N/A

Units: ug/Kg

Lab Control Spike - Batch: 680-110956

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID: Client Matrix: Dilution: Date Analyzed: Date Prepared:	LCS 680-110956/10 Solid 40 07/08/2008 2357 N/A	Analysis Batch: Prep Batch: N/A Units: ug/Kg	680-110956	Lab Fil Initial \	le ID: mq020.d Weight/Volume: {	/olatiles - M 5 g 5 mL
Analyte		Spike Amount	Result	% Rec.	Limit	Qual
Vinyl acetate		5000	3860	77	10 - 254	
Vinyl chloride		2500	2820	113	56 - 139	
Xylenes, Total		7500	8230	110	76 - 122	
Surrogate		% R	lec	Acc	ceptance Limits	
4-Bromofluorobe	enzene	11	1		65 - 124	
Dibromofluorom	ethane	12	0		65 - 124	
Toluene-d8 (Sur	r)	11	4		65 - 132	

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

# Method Blank - Batch: 680-111103

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111103/12	Analysis Batch: 680-111103	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq029.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/09/2008 1125		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
Acetone	2000	U	180	2000
Acetonitrile	8000	U	1800	8000
Acrolein	4000	U	760	4000
Acrylonitrile	4000	U	920	4000
Benzene	200	U	32	200
Carbon disulfide	200	U	48	200
Dichlorobromomethane	200	U	33	200
Bromoform	200	U	44	200
Bromomethane	200	U	64	200
2-Butanone (MEK)	1000	U	110	1000
Carbon tetrachloride	200	U	40	200
Chlorobenzene	200	U	29	200
Chloroethane	200	U	48	200
Chloroform	200	U	20	200
Chloromethane	200	U	28	200
2-Chloro-1,3-butadiene	200	U	23	200
3-Chloro-1-propene	200	U	60	200
Chlorodibromomethane	200	U	20	200
1,2-Dibromo-3-Chloropropane	400	U	110	400
Ethylene Dibromide	200	U	60	200
Dibromomethane	200	U	48	200
rans-1.4-Dichloro-2-butene	400	U	120	400
Dichlorodifluoromethane	200	U	36	200
1,1-Dichloroethane	200	U	20	200
1,2-Dichloroethane	200	U	40	200
1,1-Dichloroethene	200	U	22	200
1.2-Dichlorobenzene	200	U	26	200
rans-1,2-Dichloroethene	200	U	39	200
1,2-Dichloropropane	200	U	44	200
1.3-Dichlorobenzene	200	U	33	200
1.4-Dichlorobenzene	200	U	20	200
cis-1,3-Dichloropropene	200	U	35	200
rans-1,3-Dichloropropene	200	U	35	200
Ethylbenzene	200	U	30	200
Ethyl methacrylate	200	U	88	200
2-Hexanone	1000	U	84	1000
odomethane	200	U	40	200
sobutyl alcohol	8000	U	2800	8000
Methacrylonitrile	4000	U	960	4000
Methylene Chloride	200	U	40	200
Methyl methacrylate	200	U	150	200

Date Prepared: N/A

Method Blank - Batch: 680-111103

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111103/12	Analysis Batch: 680-111103	Instrument ID: G	GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: m	nq029.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volu	ume: 5 g
Date Analyzed:	07/09/2008 1125		Final Weight/Volu	me: 5 mL

Analyte	Result	Qual	MDL	RL
4-Methyl-2-pentanone (MIBK)	1000	U	120	1000
Pentachloroethane	1000	U	88	1000
Propionitrile	4000	U	840	4000
Styrene	200	U	26	200
1,1,1,2-Tetrachloroethane	200	U	26	200
1,1,2,2-Tetrachloroethane	200	U	56	200
Tetrachloroethene	200	U	29	200
Toluene	200	U	32	200
1,1,1-Trichloroethane	200	U	23	200
1,1,2-Trichloroethane	200	U	48	200
Trichloroethene	200	U	40	200
Trichlorofluoromethane	200	U	60	200
1,2,3-Trichloropropane	200	U	56	200
Vinyl acetate	400	U	60	400
Vinyl chloride	200	U	23	200
Xylenes, Total	400	U	92	400
Surrogate	% Rec Acceptance Limits			
4-Bromofluorobenzene	84 65 - 124			
Dibromofluoromethane	86		65 - 124	
Toluene-d8 (Surr)	73		65 - 132	

Client Matrix:

Date Analyzed: Date Prepared:

Dilution:

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Lab Control Spike - Batch: 680-111103

Lab Sample ID: LCS 680-111103/14

Solid

07/09/2008 0901

40

N/A

## Method: 8260B Preparation: N/A

Instrument ID:	GC/MS	Vo	latiles - M
Lab File ID:	mq025	d	
Initial Weight/Ve	olume:	5	g
Final Weight/Vo	Final Weight/Volume:		

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Acetone	5000	4700	94	16 - 202	
Benzene	2500	2500	100	63 - 130	
Carbon disulfide	2500	2170	87	46 - 134	
Dichlorobromomethane	2500	1750	70	64 - 137	
Bromoform	2500	2280	91	66 - 127	
Bromomethane	2500	2860	115	54 - 146	
2-Butanone (MEK)	5000	4600	92	19 - 192	
Carbon tetrachloride	2500	1880	75	60 - 136	
Chlorobenzene	2500	2640	106	77 - 120	
Chloroethane	2500	1050	42	26 - 166	
Chloroform	2500	2860	114	68 - 127	
Chloromethane	2500	3130	125	46 - 137	
Chlorodibromomethane	2500	2340	93	70 - 126	
1,2-Dibromo-3-Chloropropane	2500	1940	77	62 - 140	
Ethylene Dibromide	2500	2150	86	61 - 138	
Dibromomethane	2500	1930	77	61 - 138	
Dichlorodifluoromethane	2500	1750	70	17 - 163	
1,1-Dichloroethane	2500	2610	104	65 - 130	
1,2-Dichloroethane	2500	2090	84	62 - 140	
1,1-Dichloroethene	2500	2200	88	59 - 137	
1,2-Dichlorobenzene	2500	2830	113	75 - 123	
trans-1,2-Dichloroethene	2500	2130	85	66 - 127	
1,2-Dichloropropane	2500	2230	89	66 - 135	
1,3-Dichlorobenzene	2500	2560	102	74 - 123	
1,4-Dichlorobenzene	2500	2490	100	75 - 122	
cis-1,3-Dichloropropene	2500	1480	59	66 - 137	*
trans-1,3-Dichloropropene	2500	1990	80	64 - 138	
Ethylbenzene	2500	2620	105	77 - 121	
2-Hexanone	5000	5070	101	47 - 151	
Methylene Chloride	2500	2140	86	65 - 126	
4-Methyl-2-pentanone (MIBK)	5000	3240	65	50 - 148	
Styrene	2500	2580	103	75 - 123	
1,1,1,2-Tetrachloroethane	2500	2630	105	72 - 124	
1,1,2,2-Tetrachloroethane	2500	2200	88	65 - 130	
Tetrachloroethene	2500	2720	109	76 - 120	
Toluene	2500	2460	98	67 - 132	
1,1,1-Trichloroethane	2500	1930	77	56 - 140	
1,1,2-Trichloroethane	2500	2150	86	62 - 138	
Trichloroethene	2500	2290	92	68 - 133	
Trichlorofluoromethane	2500	1540	62	33 - 152	
1,2,3-Trichloropropane	2500	2350	94	65 - 132	

Analysis Batch: 680-111103

Prep Batch: N/A

Units: ug/Kg

Lab Control Spike - Batch: 680-111103

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID: Client Matrix: Dilution: Date Analyzed: Date Prepared:	LCS 680-111103/14 Solid 40 07/09/2008 0901 N/A	Analysis Batch: Prep Batch: N/A Units: ug/Kg	680-111103	Lab Fil Initial V	e ID: mq025.d Weight/Volume: 5	olatiles - M g mL
Analyte		Spike Amount	Result	% Rec.	Limit	Qual
Vinyl acetate		5000	4270	85	10 - 254	
Vinyl chloride		2500	2670	107	56 - 139	
Xylenes, Total		7500	7680	102	76 - 122	
Surrogate		% R	lec	Acc	ceptance Limits	
4-Bromofluorobe	enzene	10	8		65 - 124	
Dibromofluorom	ethane	11	0		65 - 124	
Toluene-d8 (Sur	r)	98	}		65 - 132	

Method Blank - Batch: 680-111182

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: 5030B

Lab Sample ID:	MB 680-111182/9	Analysis Batch: 680-111182	Instrument ID: GC/MS Volatiles - P C2
Client Matrix:	Water	Prep Batch: N/A	Lab File ID: pq462.d
Dilution:	1.0	Units: ug/L	Initial Weight/Volume: 5 mL
Date Analyzed:	07/10/2008 1328		Final Weight/Volume: 5 mL
Date Prepared:	07/10/2008 1328		

Acetone				
Acelone	25	U	5.0	25
Acetonitrile	40	U	15	40
Acrolein	20	U	18	20
Acrylonitrile	20	U	3.8	20
Benzene	1.0	U	0.32	1.0
Bromodichloromethane	1.0	U	0.34	1.0
Carbon disulfide	2.0	U	0.60	2.0
Bromoform	1.0	U	0.41	1.0
Bromomethane	1.0	U	0.50	1.0
2-Butanone	10	U	0.60	10
Carbon tetrachloride	1.0	U	0.27	1.0
Chlorobenzene	1.0	U	0.34	1.0
Chloroethane	1.0	U	1.0	1.0
Chloroform	1.0	U	0.29	1.0
Chloromethane	1.0	U	0.28	1.0
Chloroprene	1.0	U	0.35	1.0
3-Chloro-1-propene	1.0	U	0.46	1.0
Dibromochloromethane	1.0	U	0.30	1.0
1,2-Dibromo-3-Chloropropane	1.0	U	0.48	1.0
1.2-Dibromoethane	1.0	U	0.30	1.0
Dibromomethane	1.0	U	0.29	1.0
trans-1,4-Dichloro-2-butene	2.0	U	0.83	2.0
Dichlorodifluoromethane	1.0	U	0.33	1.0
1,1-Dichloroethane	1.0	U	0.32	1.0
1,2-Dichloroethane	1.0	U	0.31	1.0
1,1-Dichloroethene	1.0	U	0.36	1.0
1,2-Dichlorobenzene	1.0	U	0.33	1.0
trans-1,2-Dichloroethene	1.0	U	0.30	1.0
1,2-Dichloropropane	1.0	U	0.36	1.0
1.3-Dichlorobenzene	1.0	U	0.31	1.0
1,4-Dichlorobenzene	1.0	U	0.33	1.0
cis-1,3-Dichloropropene	1.0	U	0.37	1.0
trans-1,3-Dichloropropene	1.0	U	0.27	1.0
Ethylbenzene	1.0	U	0.30	1.0
Ethyl methacrylate	1.0	U	1.0	1.0
2-Hexanone	10	U	0.68	10
lodomethane	5.0	U	1.0	5.0
Isobutanol	40	U	19	40
Methacrylonitrile	20	U	6.6	20
Methylene Chloride	5.0	U	1.0	5.0
Methyl methacrylate	1.0	U	0.38	1.0

Method Blank - Batch: 680-111182

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: 5030B

Lab Sample ID:	MB 680-111182/9	Analysis Batch: 680-111182	Instrument ID: GC/MS Volatiles - P C2
Client Matrix:	Water	Prep Batch: N/A	Lab File ID: pq462.d
Dilution:	1.0	Units: ug/L	Initial Weight/Volume: 5 mL
Date Analyzed:	07/10/2008 1328		Final Weight/Volume: 5 mL
Date Prepared:	07/10/2008 1328		

Analyte	Result	Qual	MDL	RL
4-Methyl-2-pentanone	10	U	0.60	10
Pentachloroethane	5.0	U	1.3	5.0
Propionitrile	20	U	9.2	20
Styrene	1.0	U	0.36	1.0
1,1,1,2-Tetrachloroethane	1.0	U	0.29	1.0
1,1,2,2-Tetrachloroethane	1.0	U	0.26	1.0
Tetrachloroethene	1.0	U	0.28	1.0
Toluene	1.0	U	0.31	1.0
1,1,1-Trichloroethane	1.0	U	0.39	1.0
1,1,2-Trichloroethane	1.0	U	0.51	1.0
Trichloroethene	1.0	U	0.40	1.0
Trichlorofluoromethane	1.0	U	0.29	1.0
1,2,3-Trichloropropane	1.0	U	0.42	1.0
Vinyl acetate	2.0	U	0.62	2.0
Vinyl chloride	1.0	U	0.20	1.0
Xylenes, Total	2.0	U	0.87	2.0
Surrogate	% Rec		Acceptance Limits	
4-Bromofluorobenzene	96		75 - 120	
Dibromofluoromethane	98		75 - 121	
Toluene-d8 (Surr)	96		75 - 120	

## Lab Control Spike/ Lab Control Spike Duplicate Recovery Report - Batch: 680-111182

07/10/2008 1200

07/10/2008 1200

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: 5030B

LCS Lab Sample ID: Client Matrix:	LCS 680-111182/6 Water	Analysis Batch: 680-111182 Prep Batch: N/A	Instrument ID: GC/MS Volatiles - P C2 Lab File ID: pq454.d
Dilution:	1.0	Units: ug/L	Initial Weight/Volume: 5 mL
Date Analyzed:	07/10/2008 1124		Final Weight/Volume: 5 mL
Date Prepared:	07/10/2008 1124		
LCSD Lab Sample ID	: LCSD 680-111182/7	Analysis Batch: 680-111182	Instrument ID: GC/MS Volatiles - P C2
Client Matrix:	Water	Prep Batch: N/A	Lab File ID: pq456.d
Dilution:	1.0	Units: ug/L	Initial Weight/Volume: 5 mL

Final Weight/Volume: 5 mL

	o	% Rec.					
Analyte	LCS	LCSD	Limit	RPD	RPD Limit	LCS Qual	LCSD Qual
Acetone	134	130	17 - 175	3	50		
Benzene	99	95	77 - 119	4	30		
Bromodichloromethane	105	102	78 - 127	3	30		
Carbon disulfide	94	90	55 - 131	4	30		
Bromoform	103	104	62 - 133	0	30		
Bromomethane	142	135	12 - 184	5	50		
2-Butanone	119	113	33 - 157	5	30		
Carbon tetrachloride	104	101	71 - 135	3	30		
Chlorobenzene	101	98	85 - 116	3	30		
Chloroethane	123	123	40 - 165	0	50		
Chloroform	104	102	82 - 120	2	30		
Chloromethane	91	87	48 - 142	5	50		
Dibromochloromethane	106	102	75 - 133	3	30		
1,2-Dibromo-3-Chloropropane	100	101	49 - 140	1	30		
1,2-Dibromoethane	103	102	80 - 121	1	30		
Dibromomethane	103	102	78 - 119	1	30		
Dichlorodifluoromethane	115	111	34 - 154	4	30		
1,1-Dichloroethane	98	98	74 - 127	0	30		
1,2-Dichloroethane	102	102	66 - 132	1	30		
1,1-Dichloroethene	97	94	62 - 141	3	30		
1,2-Dichlorobenzene	103	102	79 - 124	1	30		
trans-1,2-Dichloroethene	93	93	72 - 131	0	30		
1,2-Dichloropropane	100	96	73 - 124	3	30		
1,3-Dichlorobenzene	100	100	78 - 125	0	30		
1,4-Dichlorobenzene	102	101	81 - 122	2	30		
cis-1,3-Dichloropropene	95	92	76 - 126	3	30		
trans-1,3-Dichloropropene	97	94	73 - 128	3	30		
Ethylbenzene	97	95	86 - 116	3	30		
2-Hexanone	114	108	34 - 161	5	30		
Methylene Chloride	97	95	70 - 125	2	30		
4-Methyl-2-pentanone	102	99	40 - 151	4	30		
Styrene	101	98	82 - 122	3	30		

Calculations are performed before rounding to avoid round-off errors in calculated results.

Date Analyzed:

Date Prepared:

Date Prepared:

## Lab Control Spike/ Lab Control Spike Duplicate Recovery Report - Batch: 680-111182

07/10/2008 1200

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: 5030B

LCS Lab Sample ID: Client Matrix: Dilution: Date Analyzed: Date Prepared:	LCS 680-111182/6 Water 1.0 07/10/2008 1124 07/10/2008 1124	Analysis Batch: 680-111182 Prep Batch: N/A Units: ug/L	Instrument ID: GC/MS Volatiles - P C2 Lab File ID: pq454.d Initial Weight/Volume: 5 mL Final Weight/Volume: 5 mL
LCSD Lab Sample ID Client Matrix: Dilution: Date Analyzed:	: LCSD 680-111182/7 Water 1.0 07/10/2008 1200	Analysis Batch: 680-111182 Prep Batch: N/A Units: ug/L	Instrument ID: GC/MS Volatiles - P C2 Lab File ID: pq456.d Initial Weight/Volume: 5 mL Final Weight/Volume: 5 mL

	0	<u>% Rec.</u>					
Analyte	LCS	LCSD	Limit	RPD	RPD Limit	LCS Qual	LCSD Qual
1,1,1,2-Tetrachloroethane	104	102	81 - 128	1	30		
1,1,2,2-Tetrachloroethane	94	91	69 - 129	3	30		
Tetrachloroethene	100	97	76 - 126	3	30		
Toluene	103	98	81 - 117	5	30		
1,1,1-Trichloroethane	104	98	76 - 127	5	30		
1,1,2-Trichloroethane	101	100	75 - 121	2	30		
Trichloroethene	105	101	84 - 115	4	30		
Trichlorofluoromethane	120	113	58 - 149	6	50		
1,2,3-Trichloropropane	98	98	70 - 130	0	30		
Vinyl acetate	118	116	10 - 217	2	30		
Vinyl chloride	105	100	59 - 144	5	50		
Xylenes, Total	97	94	84 - 118	4	30		
Surrogate	L	CS % Rec	LCSD %	Rec	Accep	tance Limits	
4-Bromofluorobenzene	ç	6	95		7	5 - 120	
Dibromofluoromethane	1	00	100		7	5 - 121	
Toluene-d8 (Surr)	1	00	96		7	5 - 120	

EPA ARCHIVE DOCUMENT

Date Prepared: N/A

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method Blank - Batch: 680-111218

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111218/6	Analysis Batch: 680-111218	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq047.d
Dilution:	1.0	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/10/2008 1737		Final Weight/Volume: 5 mL

Analyte	Result	Qual	MDL	RL
Acetone	8.3	J	4.4	50
Acetonitrile	200	U	45	200
Acrolein	100	U	19	100
Acrylonitrile	100	U	23	100
Benzene	5.0	U	0.79	5.0
Carbon disulfide	5.0	U	1.2	5.0
Dichlorobromomethane	5.0	U	0.83	5.0
Bromoform	5.0	U	1.1	5.0
Bromomethane	5.0	U	1.6	5.0
2-Butanone (MEK)	25	U	2.7	25
Carbon tetrachloride	5.0	U	1.0	5.0
Chlorobenzene	5.0	U	0.73	5.0
Chloroethane	5.0	U	1.2	5.0
Chloroform	5.0	U	0.50	5.0
Chloromethane	5.0	U	0.71	5.0
2-Chloro-1,3-butadiene	5.0	Ŭ	0.57	5.0
3-Chloro-1-propene	5.0	U	1.5	5.0
Chlorodibromomethane	5.0	Ŭ	0.50	5.0
1,2-Dibromo-3-Chloropropane	10	U	2.8	10
Ethylene Dibromide	5.0	U	1.5	5.0
Dibromomethane	5.0	U	1.2	5.0
trans-1,4-Dichloro-2-butene	10	U	3.1	10
Dichlorodifluoromethane	5.0	U	0.89	5.0
1,1-Dichloroethane	5.0	U	0.50	5.0
1,2-Dichloroethane	5.0	U	1.0	5.0
1,1-Dichloroethene	5.0	U	0.54	5.0
1,2-Dichlorobenzene	5.0	U	0.65	5.0
trans-1,2-Dichloroethene	5.0	U	0.97	5.0
1,2-Dichloropropane	5.0	U	1.1	5.0
1,3-Dichlorobenzene	5.0	U	0.83	5.0
1,4-Dichlorobenzene	5.0	U	0.51	5.0
cis-1,3-Dichloropropene	5.0	U	0.87	5.0
trans-1,3-Dichloropropene	5.0	U	0.87	5.0
Ethylbenzene	5.0	U	0.75	5.0
Ethyl methacrylate	5.0	U	2.2	5.0
2-Hexanone	25	U	2.1	25
lodomethane	5.0	U	1.0	5.0
Isobutyl alcohol	200	U	69	200
Methacrylonitrile	100	U	24	100
Methylene Chloride	5.0	U	24 1.0	5.0
Methyl methacrylate	5.0	U	3.7	5.0

Method Blank - Batch: 680-111218

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111218/6	Analysis Batch: 680-111218	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq047.d
Dilution:	1.0	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/10/2008 1737		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
4-Methyl-2-pentanone (MIBK)	25	U	2.9	25
Pentachloroethane	25	U	2.2	25
Propionitrile	100	U	21	100
Styrene	5.0	U	0.66	5.0
1,1,1,2-Tetrachloroethane	5.0	U	0.64	5.0
1,1,2,2-Tetrachloroethane	5.0	U	1.4	5.0
Tetrachloroethene	5.0	U	0.73	5.0
Toluene	5.0	U	0.79	5.0
1,1,1-Trichloroethane	5.0	U	0.58	5.0
1,1,2-Trichloroethane	5.0	U	1.2	5.0
Trichloroethene	5.0	U	1.0	5.0
Trichlorofluoromethane	5.0	U	1.5	5.0
1,2,3-Trichloropropane	5.0	U	1.4	5.0
Vinyl acetate	10	U	1.5	10
Vinyl chloride	5.0	U	0.58	5.0
Xylenes, Total	10	U	2.3	10
Surrogate	% Rec		Acceptance Limits	
4-Bromofluorobenzene	95		65 - 124	
Dibromofluoromethane	94		65 - 124	
Toluene-d8 (Surr)	88		65 - 132	

Client Matrix:

Date Analyzed: Date Prepared:

Dilution:

**US EPA ARCHIVE DOCUMENT** 

Lab Sample ID: LCS 680-111218/5

Solid

07/10/2008 1534

1.0

N/A

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Lab Control Spike - Batch: 680-111218

## Method: 8260B Preparation: N/A

Instrument ID:	GC/MS	Vc	latiles - M
Lab File ID:	mq044.	d	
Initial Weight/Vo	olume:	5	g
Final Weight/Vo	olume:	5	mL

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Acetone	100	119	119	16 - 202	
Benzene	50.0	46.0	92	63 - 130	
Carbon disulfide	50.0	47.5	95	46 - 134	
Dichlorobromomethane	50.0	47.0	94	64 - 137	
Bromoform	50.0	47.9	96	66 - 127	
Bromomethane	50.0	44.9	90	54 - 146	
2-Butanone (MEK)	100	105	105	19 - 192	
Carbon tetrachloride	50.0	50.0	100	60 - 136	
Chlorobenzene	50.0	45.5	91	77 - 120	
Chloroethane	50.0	49.0	98	26 - 166	
Chloroform	50.0	45.6	91	68 - 127	
Chloromethane	50.0	47.0	94	46 - 137	
Chlorodibromomethane	50.0	47.0	94	70 - 126	
1,2-Dibromo-3-Chloropropane	50.0	52.5	105	62 - 140	
Ethylene Dibromide	50.0	47.3	95	61 - 138	
Dibromomethane	50.0	48.0	96	61 - 138	
Dichlorodifluoromethane	50.0	48.6	97	17 - 163	
1,1-Dichloroethane	50.0	46.3	93	65 - 130	
1,2-Dichloroethane	50.0	47.5	95	62 - 140	
1,1-Dichloroethene	50.0	46.8	94	59 - 137	
1,2-Dichlorobenzene	50.0	45.3	91	75 - 123	
trans-1,2-Dichloroethene	50.0	44.3	89	66 - 127	
1,2-Dichloropropane	50.0	46.4	93	66 - 135	
1,3-Dichlorobenzene	50.0	45.5	91	74 - 123	
1,4-Dichlorobenzene	50.0	45.0	90	75 - 122	
cis-1,3-Dichloropropene	50.0	46.4	93	66 - 137	
trans-1,3-Dichloropropene	50.0	45.3	91	64 - 138	
Ethylbenzene	50.0	43.7	87	77 - 121	
2-Hexanone	100	110	110	47 - 151	
Methylene Chloride	50.0	43.5	87	65 - 126	
4-Methyl-2-pentanone (MIBK)	100	107	107	50 - 148	
Styrene	50.0	46.5	93	75 - 123	
1,1,1,2-Tetrachloroethane	50.0	47.7	95	72 - 124	
1,1,2,2-Tetrachloroethane	50.0	49.9	100	65 - 130	
Tetrachloroethene	50.0	48.2	96	76 - 120	
Toluene	50.0	46.3	93	67 - 132	
1,1,1-Trichloroethane	50.0	49.8	100	56 - 140	
1,1,2-Trichloroethane	50.0	48.8	98	62 - 138	
Trichloroethene	50.0	47.0	94	68 - 133	
Trichlorofluoromethane	50.0	49.6	99	33 - 152	
1,2,3-Trichloropropane	50.0	49.9	100	65 - 132	

Analysis Batch: 680-111218

Prep Batch: N/A

Units: ug/Kg

Lab Control Spike - Batch: 680-111218

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID: Client Matrix: Dilution: Date Analyzed: Date Prepared:	LCS 680-111218/5 Solid 1.0 07/10/2008 1534 N/A	Analysis Batch: Prep Batch: N/A Units: ug/Kg	680-111218	Lab Fil Initial \	Weight/Volume: 5	
Analyte		Spike Amount	Result	% Rec.	Limit	Qual
Vinyl acetate		100	90.1	90	10 - 254	
Vinyl chloride		50.0	48.9	98	56 - 139	
Xylenes, Total		150	141	94	76 - 122	
Surrogate		% R	lec	Acc	ceptance Limits	
4-Bromofluorobe	nzene	93	}		65 - 124	
Dibromofluorome	ethane	88	5		65 - 124	
Toluene-d8 (Suri	-)	91			65 - 132	

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method Blank - Batch: 680-111225

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111225/5	Analysis Batch: 680-111225	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq046.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/10/2008 1714		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
Acetone	330	J	180	2000
Acetonitrile	8000	U	1800	8000
Acrolein	4000	U	760	4000
Acrylonitrile	4000	U	920	4000
Benzene	200	U	32	200
Carbon disulfide	200	U	48	200
Dichlorobromomethane	200	U	33	200
Bromoform	200	U	44	200
Bromomethane	200	U	64	200
2-Butanone (MEK)	1000	U	110	1000
Carbon tetrachloride	200	U	40	200
Chlorobenzene	200	U	29	200
Chloroethane	200	U	48	200
Chloroform	200	U	20	200
Chloromethane	200	U	28	200
2-Chloro-1,3-butadiene	200	Ŭ	23	200
3-Chloro-1-propene	200	U	60	200
Chlorodibromomethane	200	Ŭ	20	200
1,2-Dibromo-3-Chloropropane	400	U	110	400
Ethylene Dibromide	200	U	60	200
Dibromomethane	200	U	48	200
trans-1,4-Dichloro-2-butene	400	U	120	400
Dichlorodifluoromethane	200	U	36	200
1,1-Dichloroethane	200	U	20	200
1,2-Dichloroethane	200	U	40	200
1,1-Dichloroethene	200	U	22	200
1,2-Dichlorobenzene	200	U	26	200
trans-1,2-Dichloroethene	200	U	39	200
1,2-Dichloropropane	200	U	44	200
1,3-Dichlorobenzene	200	U	33	200
1,4-Dichlorobenzene	200	U	20	200
cis-1,3-Dichloropropene	200	U	35	200
trans-1,3-Dichloropropene	200	U	35	200
Ethylbenzene	200	U	30	200
Ethyl methacrylate	200	U	88	200
2-Hexanone	1000	U	84	1000
lodomethane	200	U	40	200
Isobutyl alcohol	8000	U	2800	8000
Methacrylonitrile	4000	U	960	4000
Methaciyonnine Methylene Chloride	200	U	40	200
Methylene Chloride Methyl methacrylate	200	U	40 150	200

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method Blank - Batch: 680-111225

## Method: 8260B Preparation: N/A

Lab Sample ID:	MB 680-111225/5	Analysis Batch: 680-111225	Instrument ID: GC/MS Volatiles - M
Client Matrix:	Solid	Prep Batch: N/A	Lab File ID: mq046.d
Dilution:	40	Units: ug/Kg	Initial Weight/Volume: 5 g
Date Analyzed:	07/10/2008 1714		Final Weight/Volume: 5 mL
Date Prepared:	N/A		

Analyte	Result	Qual	MDL	RL
4-Methyl-2-pentanone (MIBK)	1000	U	120	1000
Pentachloroethane	1000	U	88	1000
Propionitrile	4000	U	840	4000
Styrene	200	U	26	200
1,1,1,2-Tetrachloroethane	200	U	26	200
1,1,2,2-Tetrachloroethane	200	U	56	200
Tetrachloroethene	200	U	29	200
Toluene	200	U	32	200
1,1,1-Trichloroethane	200	U	23	200
1,1,2-Trichloroethane	200	U	48	200
Trichloroethene	200	U	40	200
Trichlorofluoromethane	200	U	60	200
1,2,3-Trichloropropane	200	U	56	200
Vinyl acetate	400	U	60	400
Vinyl chloride	200	U	23	200
Xylenes, Total	400	U	92	400
Surrogate	% Rec		Acceptance Limits	
4-Bromofluorobenzene	80		65 - 124	
Dibromofluoromethane	74		65 - 124	
Toluene-d8 (Surr)	70		65 - 132	

Client Matrix:

Date Analyzed: Date Prepared:

Dilution:

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## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Lab Control Spike - Batch: 680-111225

Lab Sample ID: LCS 680-111225/4

Solid

07/10/2008 1557

40

N/A

## Method: 8260B Preparation: N/A

Instrument ID:	GC/MS	S Vo	latiles - M
Lab File ID:	mq045	.d	
Initial Weight/Ve	olume:	5	g
Final Weight/Vo	olume:	5	mL

Analyte	Spike Amount	Result	% Rec.	Limit	Qual
Acetone	5000	6350	127	16 - 202	
Benzene	2500	2200	88	63 - 130	
Carbon disulfide	2500	1850	74	46 - 134	
Dichlorobromomethane	2500	2130	85	64 - 137	
Bromoform	2500	2550	102	66 - 127	
Bromomethane	2500	2090	84	54 - 146	
2-Butanone (MEK)	5000	5420	108	19 - 192	
Carbon tetrachloride	2500	2290	92	60 - 136	
Chlorobenzene	2500	2500	100	77 - 120	
Chloroethane	2500	507	20	26 - 166	*
Chloroform	2500	2530	101	68 - 127	
Chloromethane	2500	2500	100	46 - 137	
Chlorodibromomethane	2500	2480	99	70 - 126	
1,2-Dibromo-3-Chloropropane	2500	2340	94	62 - 140	
Ethylene Dibromide	2500	2240	90	61 - 138	
Dibromomethane	2500	1990	80	61 - 138	
Dichlorodifluoromethane	2500	1490	60	17 - 163	
1,1-Dichloroethane	2500	2350	94	65 - 130	
1,2-Dichloroethane	2500	2010	81	62 - 140	
1,1-Dichloroethene	2500	1960	79	59 - 137	
1,2-Dichlorobenzene	2500	2500	100	75 - 123	
trans-1,2-Dichloroethene	2500	1990	80	66 - 127	
1,2-Dichloropropane	2500	2190	88	66 - 135	
1,3-Dichlorobenzene	2500	2430	97	74 - 123	
1,4-Dichlorobenzene	2500	2480	99	75 - 122	
cis-1,3-Dichloropropene	2500	1990	79	66 - 137	
trans-1,3-Dichloropropene	2500	1990	80	64 - 138	
Ethylbenzene	2500	2300	92	77 - 121	
2-Hexanone	5000	5470	109	47 - 151	
Methylene Chloride	2500	1960	78	65 - 126	
4-Methyl-2-pentanone (MIBK)	5000	4570	91	50 - 148	
Styrene	2500	2530	101	75 - 123	
1,1,1,2-Tetrachloroethane	2500	2570	103	72 - 124	
1,1,2,2-Tetrachloroethane	2500	2310	92	65 - 130	
Tetrachloroethene	2500	2480	99	76 - 120	
Toluene	2500	2320	93	67 - 132	
1,1,1-Trichloroethane	2500	2200	88	56 - 140	
1,1,2-Trichloroethane	2500	2200	88	62 - 138	
Trichloroethene	2500	2150	86	68 - 133	
Trichlorofluoromethane	2500	2010	80	33 - 152	
1,2,3-Trichloropropane	2500	2340	93	65 - 132	

Analysis Batch: 680-111225

Prep Batch: N/A

Units: ug/Kg

Lab Control Spike - Batch: 680-111225

## **Quality Control Results**

Job Number: 680-38109-1 Sdg Number: KSX01

## Method: 8260B Preparation: N/A

Lab Sample ID: Client Matrix: Dilution: Date Analyzed: Date Prepared:	LCS 680-111225/4 Solid 40 07/10/2008 1557 N/A	Analysis Batch: Prep Batch: N/A Units: ug/Kg	680-111225	Lab Fil Initial V	Veight/Volume: 5	
Analyte		Spike Amount	Result	% Rec.	Limit	Qual
Vinyl acetate		5000	5510	110	10 - 254	
Vinyl chloride		2500	2430	97	56 - 139	
Xylenes, Total		7500	7230	96	76 - 122	
Surrogate		% R	lec	Acc	ceptance Limits	
4-Bromofluorobe	enzene	10	14		65 - 124	
Dibromofluorome	ethane	10	0		65 - 124	
Toluene-d8 (Sur	r)	92	2		65 - 132	

er 007274	Website: www.testamericainc.com Phone: (912) 354-7858 Fax: (912) 352-0165		Phone: Fax:	PAGE / / OF			UELIVEHY (SURCHARGE)	PER N	REMARKS				· · · · · · · · · · · · · · · · · · ·					L'X ANU		DATE	DATE	sample SB ADS 71-8 not an COL.	rantservsk Anis Ra Shi Rochicelov Ra	A Marled W/ there the man
			Phon Fax:	REQUIRED ANALYSIS					NUMBER OF CONTAINERS SUBMITTED											TIME RELINQUISHED BY: (SIGMATURE)	ത	. 3	pylo	s guard cluspage
DOCUMENT	IRD X TestAmerica Savannah 5102 LaRoche Avenue Savannah GA 3100	Alternate Laboratory Name/Location		MATRIX TYPE	90 (*** 1N	7722	סרוס	OR SEMISC OR SEMISC	RIA ANON	N 4		-2		<u>,</u> 2	<u>~</u>	2	0			E) DATE	DATE		CUSTODY SAVANNAH SEAL NO. LOG NO.	divation pachage
VE	ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD			3 (STATE) JL	CONTRACT NO.	00 607782121	C. CON	Shuthiam NH 03		11-12, 6	-7.5'	)/-1('	11-12	- (0*	1 - 10'	1,-101				TIME RELINQUISHED BY: (SIGNATURE)	IME RECEIVED BY: (SIGNATURE)		OUT YES OUT	1 II data validi
<b>US EPA ARCHI</b>	ANALYSIS REQUEST AN	estamerica	THE LEADER IN ENVIRONMENTAL TESTING	PROJECT NO.		60778 W		1	SAMPLE ID	SB- A01 - 11'-		1 80 - 501 - 10	0 54- 403 -1	1 SU-DOI-91	0 SQ - B01-9	59-1001-9	Trin Bunk			TON 6124 A	EV DATE 1		D DATE OGZZOD	report level
D		estar	THE LEADER IN ENVI	PROJECT REFERENCE	TAL (LAB) PEDUECT MANAGER	CLIENT NAME	Jayder Park	COMPANY CONTRACTING THIS WORK (II applicable)	SAMPLE TIME	26 N	12/21/08/0930	<u> </u>		UCH KOLSKU	c/2/07 1400	1611 410 14 15	- Innia			RECINQUISHED BY (SOMATUR	RECEIVED TY: (SIGNATIVES)		RECEIVED FOR LABORATORY BY	* Please

## Login Number: 38109

## Creator: Hall, Karl I

## List Number: 1

Question	T / F/ NA	Comment
Radioactivity either was not measured or, if measured, is at or below background	False	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	4.9 C
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
There are no discrepancies between the sample IDs on the containers and the COC.	False	Rec. SB-A05-7'-8' not on COC, per E.Stanisewski lab not to analyze
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	MS/MSD not reqesuted
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	

#### List Source: TestAmerica Savannah



## ATTACHMENT D SOIL VAPOR VOC LABORATORY ANALYTICAL DATA PROJECT NUMBER 680-38216

VP-A01 Intermediate VP-A03 Shallow VP-A02 ROI Duplicate (VP-A03 Shallow)

# APPENDIX A PRE-WORK PLAN DATA COLLECTION SUMMARY

SVE PILOT TESTING WORK PLAN

APPENDIX A: PRE-WORK PLAN DATA COLLECTION SUMMARY Soil Vapor Extraction Pilot Testing Work Plan



THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

## ANALYTICAL REPORT

PROJECT NO. 680-38216

Solutia Vapor Sampling

Lot #: H8G030101

Lidya Gulizia

TestAmerica Savannah 5102 Laroche Avenue Savannah, GA 31404

TESTAMERICA LABORATORIES, INC.

Gernzlebelhurbbonn P

Terry Wasmund Project Manager

July 23, 2008

## ANALYTICAL METHODS SUMMARY

#### H8G030101

PARAMETER		ANALYTICAL METHOD
Volatile	Organics by TO15	EPA-2 TO-15
Reference	s:	
EPA-2	"Compendium of Methods for	the Determination of Toxic

PA-2 "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air", EPA-625/R-96/010b, January 1999.

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## SAMPLE SUMMARY

## H8G030101

				SAMPLED	SAMP
WO #	SAMPLE#	CLIENT	SAMPLE ID	DATE	TIME
KQ2FX	001	VP-A01	INTERMEDIATE	07/01/08	15:52
KO2F0	002	VP-A03	SHALLOW	07/01/08	12:42
KO2F1	003	VP-A02	ROI	07/01/08	14:25
KO2F2	004	DUPLICA	TE	07/01/08	12:00
~					

#### NOTE(S):

- The analytical results of the samples listed above are presented on the following pages.

- All calculations are performed before rounding to avoid round-off errors in calculated results.

- Results noted as "ND" were not detected at or above the stated limit.

- This report must not be reproduced, except in full, without the written approval of the laboratory.

- Results for the following parameters are never reported on a dry weight basis: color, corrosivity, density, flashpoint, ignitability, layers, odor,

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paint filter test, pH, porosity pressure, reactivity, redox potential, specific gravity, spot tests, solids, solubility, temperature, viscosity, and weight.

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## PROJECT NARRATIVE H8G030101

The results reported herein are applicable to the samples submitted for analysis only.

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The original chain of custody documentation is included with this report.

## **Sample Receipt**

Custody seals were not present.

## **Quality Control and Data Interpretation**

Unless otherwise noted, all holding times and QC criteria were met and the test results shown in this report meet all applicable NELAC requirements.

The EPA method requires that all target analytes in the continuing calibration verification standard be within 30% difference from the initial calibration. The laboratory standard operating procedure allows up to four analytes in the calibration verification to be  $\leq 40\%$  difference from the initial calibration. The calibration verification analyzed on 07/16/08 exhibited a %difference of >30% but  $\leq 40\%$  for benzene and 1,2-dichloroethane.

Although benzene is flagged as being outside recovery limits in the laboratory control sample for batch 8199127, the laboratory control sample is in control. The standard operating procedure allows for two nonpolar analyte recoveries between 60% and 140% and two polar analyte recoveries between 45% and 155%.

The samples were received on 7/03/08 in Tedlar bags and transferred into Summa Canisters within 72 hours of sampling.

TestAmerica Knoxville maintains the following certifications, approvals and accreditations: Arkansas DEQ Cert. #05-043-0, California DHS ELAP Cert. #2423, Colorado DPHE, Connecticut DPH Cert. #PH-0223, Florida DOH Cert. #E87177, Georgia DNR Cert. #906, Hawaii DOH, Illinois EPA Cert. #000687, Indiana DOH Cert. #C-TN-02, Iowa DNR Cert. #375, Kansas DHE Cert. #E-10349, Kentucky DEP Lab ID #90101, Louisiana DEQ Cert. #03079, Louisiana DOHH Cert. #LA030024, Maryland DHMH Cert. #277, Massachusetts DEP Cert. #M-TN009, Michigan DEQ Lab ID #9933, New Jersey DEP Cert. #TN001, New York DOH Lab #10781, North Carolina DPH Lab ID #21705, North Carolina DEHNR Cert. #64, Ohio EPA VAP Cert. #CL0059, Oklahoma DEQ ID #9415, Pennsylvania DEP Cert. #68-00576, South Carolina DHEC Lab ID #84001001, Tennessee DOH Lab ID #02014, Utah DOH Cert. # QUAN3, Virginia DGS Lab ID #00165, Washington DOE Lab #C120, West Virginia DEP Cert. #345, Wisconsin DNR Lab ID #998044300, Naval Facilities Engineering Service Center and USDA Soil Permit #S-46424. This list of approvals is subject to change and does not imply that laboratory certification is available for all parameters reported in this environmental sample data report.

# Sample Data Summary

Client Sample ID: VP-A01 INTERMEDIATE

#### GC/MS Volatiles

Lot-Sample #: H8	3G030101-001 W	Nork Order #:	KQ2FX1AA	Matrix:	AIR
Date Sampled: 07	7/01/08 D	ate Received:	07/02/08		
Prep Date: 07	7/17/08 A	malysis Date	07/17/08		
Prep Batch #: 81	199311				
Dilution Factor: 12	225.26 M	lethod:	EPA-2 TO-15		

REPORTING PARAMETER RESULT LIMIT UNITS Dichlorodifluoromethane ND 250 ppb(v/v)1,2-Dichloro-ND 250 ppb(v/v)1,1,2,2-tetrafluoroethane Chloromethane ND 610 ppb(v/v)Vinyl chloride ND250 ppb(v/v)Bromomethane ND 250 ppb(v/v)Chloroethane ND 250 ppb(v/v)Trichlorofluoromethane ND 250 ppb(v/v)1,1-Dichloroethene ND 250 ppb(v/v)1,1,2-Trichloro-ND 250 ppb(v/v)-1,2,2-trifluoroethane Methylene chloride ND 610 ppb(v/v)1,1-Dichloroethane ND 250 ppb(v/v)cis-1,2-Dichloroethene ND 250 ppb(v/v)Chloroform ND 250 ppb(v/v) 1,1,1-Trichloroethane  $\mathbf{ND}$ 250 ppb(v/v)Carbon tetrachloride ND 250 ppb(v/v)Benzene 37000 250 ppb(v/v)1.2-Dichloroethane ND 250 ppb(v/v)Trichloroethene ND ppb(v/v)250 1,2-Dichloropropane ND 250 ppb(v/v)cis-1,3-Dichloropropene ND 250 ppb(v/v)Toluene ND 250 ppb(v/v)trans-1,3-Dichloropropene ND 250 ppb(v/v)1,1,2-Trichloroethane ND 250 ppb(v/v)Tetrachloroethene ND 250 ppb(v/v)1,2-Dibromoethane (EDB) ND 250 ppb(v/v)Chlorobenzene ND 250 ppb(v/v)Ethylbenzene ND 250 ppb(v/v)m-Xylene & p-Xylene ppb(v/v)ND 250 o-Xylene ND 250 ppb(v/v)Styrene ND 250 ppb(v/v)1,1,2,2-Tetrachloroethane ND 250 ppb(v/v)1,3,5-Trimethylbenzene ND 250 ppb(v/v)1,2,4-Trimethylbenzene ND 250 ppb(v/v)1,3-Dichlorobenzene ND 250 ppb(v/v)1,4-Dichlorobenzene ppb(v/v)ND 250 1,2-Dichlorobenzene ND 250 ppb(v/v)Benzyl chloride ND 490 ppb(v/v)

(Continued on next page)

## Client Sample ID: VP-A01 INTERMEDIATE

## GC/MS Volatiles

Lot-Sample #...: H8G030101-001 Work Order #...: KQ2FX1AA Matrix...... AIR

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PARAMETER	RESULT	REPORTING LIMIT	UNITS
1,2,4-Trichloro- benzene	ND	1200	ppb (v/v)
Hexachlorobutadiene	ND	1200	ppb(v/v)
	PERCENT	RECOVERY	
SURROGATE	RECOVERY	LIMITS	
1,2-Dichloroethane-d4	97	(70 - 130	))
Toluene-d8	102	(70 - 130	)
4-Bromofluorobenzene	92	(70 - 130	))

and the second second

Client Sample ID: VP-A03 SHALLOW

## GC/MS Volatiles

Lot-Sample #: H80 Date Sampled: 07 Prep Date: 07 Prep Batch #: 81	/01/08 Date /16/08 Analy	Order #: Received: vsis Date:	07/02/08	Matrix:	AIR
Dilution Factor: 37		d	EPA-2 TO-15		

		REPORTING	
PARAMETER	RESULT	LIMIT	UNITS
Dichlorodifluoromethane	ND	7400	ppb(v/v)
1,2-Dichloro-	ND	7400	ppb(v/v)
1,1,2,2-tetrafluoroethane			
Chloromethane	ND	19000	ppb(v/v)
Vinyl chloride	ND	7400	ppb(v/v)
Bromomethane	ND	7400	ppb(v/v)
Chloroethane	ND	7400	ppb(v/v)
Trichlorofluoromethane	ND	7400	ppb(v/v)
1,1-Dichloroethene	ND	7400	ppb(v/v)
1,1,2-Trichloro-	ND	7400	ppb(v/v)
1,2,2-trifluoroethane		·	
Methylene chloride	ND	19000	ppb(v/v)
1,1-Dichloroethane	ND	7400	ppb(v/v)
cis-1,2-Dichloroethene	ND	7400	ppb(v/v)
Chloroform	ND	7400	ppb(v/v)
1,1,1-Trichloroethane	ND	7400	ppb(v/v)
Carbon tetrachloride	ND	7400	ppb(v/v)
Benzene	1200000	7400	ppb(v/v)
1,2-Dichloroethane	ND	7400	ppb(v/v)
Trichloroethene	ND	7400	ppb(v/v)
1,2-Dichloropropane	ND	7400	ppb(v/v)
cis-1,3-Dichloropropene	ND	7400	ppb(v/v)
Toluene	ND	7400	ppb(v/v)
trans-1,3-Dichloropropene	ND	7400	ppb(v/v)
1,1,2-Trichloroethane	ND	7400	ppb(v/v)
Tetrachloroethene	ND	7400	ppb(v/v)
1,2-Dibromoethane (EDB)	ND	7400	ppb(v/v)
Chlorobenzene	ND	7400	ppb(v/v)
Ethylbenzene	ND	7400	ppb(v/v)
m-Xylene & p-Xylene	ND	7400	ppb(v/v)
o-Xylene	ND	7400	ppb(v/v)
Styrene	ND	7400	ppb(v/v)
1,1,2,2-Tetrachloroethane	ND	7400	ppb(v/v)
1,3,5-Trimethylbenzene	ND	7400	ppb(v/v)
1,2,4-Trimethylbenzene	ND	7400	ppb(v/v)
1,3-Dichlorobenzene	ND	7400	ppb(v/v)
1,4-Dichlorobenzene	ND	7400	ppb(v/v)
1,2-Dichlorobenzene	ND	7400	ppb(v/v)
Benzyl chloride	ND	15000	ppb(v/v)

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## Client Sample ID: VP-A03 SHALLOW

## GC/MS Volatiles

Lot-Sample #...: H8G030101-002 Work Order #...: KQ2F01AA Matrix...... AIR

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		REPORTING	
PARAMETER	RESULT	LIMIT	UNITS
1,2,4-Trichloro- benzene	ND	37000	ppb (v/v)
Hexachlorobutadiene	ND	37000	ppb(v/v)
	PERCENT	RECOVERY	
SURROGATE	RECOVERY	LIMITS	_
1,2-Dichloroethane-d4	103	(70 - 130)	-
Toluene-d8	96	(70 - 130)	
4-Bromofluorobenzene	95	(70 - 130)	

Client Sample ID: VP-A02 ROI

## GC/MS Volatiles

Lot-Sample #: H8G03	0101-003 Work Order #	: KQ2F11AA I	Matrix: AIR
Date Sampled: 07/01	/08 Date Received	: 07/02/08	
Prep Date: 07/17	//08 Analysis Date	: 07/17/08	
Prep Batch #: 81993	11		

Dilution Factor: 68352.36 Method.....: EPA-2 TO-15

		REPORTIN	-
PARAMETER	RESULT	LIMIT	<u>UNITS</u>
Dichlorodifluoromethane	ND	14000	ppb (v/v)
1,2-Dichloro-	ND	14000	ppb(v/v)
1,1,2,2-tetrafluoroethane			
Chloromethane	ND	34000	ppb(v/v)
Vinyl chloride	ND	14000	ppb(v/v)
Bromomethane	ND	14000	ppb(v/v)
Chloroethane	ND	14000	ppb (v/v)
Trichlorofluoromethane	ND	14000	ppb(v/v)
1,1-Dichloroethene	ND	14000	ppb(v/v)
1,1,2-Trichloro-	ND	14000	ppb-(v/v)
1,2,2-trifluoroethane			
Methylene chloride	ND	34000	ppb (v/v)
1,1-Dichloroethane	ND	14000	ppb(v/v)
cis-1,2-Dichloroethene	ND	14000	ppb(v/v)
Chloroform	ND	14000	ppb(v/v)
1,1,1-Trichloroethane	ND	14000	ppb(v/v)
Carbon tetrachloride	ND	14000	ppb(v/v)
Benzene	3300000	14000	ppb(v/v)
1,2-Dichloroethane	ND	14000	ppb(v/v)
Trichloroethene	ND	14000	ppb(v/v)
1,2-Dichloropropane	ND	14000	ppb(v/v)
cis-1,3-Dichloropropene	ND	14000	ppb(v/v)
Toluene	ND	14000	ppb(v/v)
trans-1,3-Dichloropropene	ND	14000	ppb(v/v)
1,1,2-Trichloroethane	ND	14000	ppb(v/v)
Tetrachloroethene	ND	14000	ppb(v/v)
1,2-Dibromoethane (EDB)	ND	14000	ppb(v/v)
Chlorobenzene	ND	14000	ppb(v/v)
Ethylbenzene	ND	14000	ppb(v/v)
m-Xylene & p-Xylene	ND	14000	ppb(v/v)
o-Xylene	ND	14000	ppb(v/v)
Styrene	ND	14000	ppb(v/v)
1,1,2,2-Tetrachloroethane	ND	14000	ppb(v/v)
1,3,5-Trimethylbenzene	ND	14000	(v/v) dqq
1,2,4-Trimethylbenzene	ND	14000	ppb(v/v)
1,3-Dichlorobenzene	ND	14000	ppb(v/v)
•		14000	ppb(v/v)
1,4-Dichlorobenzene	ND	14000	ppb(v/v) ppb(v/v)
1,2-Dichlorobenzene	ND		ppb(v/v) ppb(v/v)
Benzyl chloride	ND	27000	PPp(()))

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## Client Sample ID: VP-A02 ROI

## GC/MS Volatiles

Lot-Sample #...: H8G030101-003 Work Order #...: KQ2F11AA Matrix.....: AIR

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		REPORTING	
PARAMETER	RESULT	LIMIT	UNITS
1,2,4-Trichloro- benzene	ND	68000	ppb(v/v)
Hexachlorobutadiene	ND	68000	ppb(v/v)
	PERCENT	RECOVERY	
SURROGATE	RECOVERY	LIMITS	
1,2-Dichloroethane-d4	98	(70 - 130)	<b>-</b> .
Toluene-d8	99	(70 - 130)	
4-Bromofluorobenzene	98	(70 - 130)	

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Client Sample ID: DUPLICATE

## GC/MS Volatiles

Lot-Sample #:	H8G030101-004	Work Order #:	KQ2F21AA	Matrix:	AIR
Date Sampled:		Date Received:			
Prep Date:	07/16/08	Analysis Date:	07/16/08		
Prep Batch #:	8199127				
Dilution Factor:	36836.1	Method	EPA-2 TO-15		

		REPORTIN	G
ביי האת את היו גיו	RESULT	LIMIT	UNITS
PARAMETER Dichlorodifluoromethane	ND	7400	ppb(v/v)
1.2-Dichloro-	ND	7400	ppb(v/v)
1,1,2,2-tetrafluoroethane	****		
Chloromethane	ND	18000	ppb(v/v)
Vinyl chloride	ND	7400	ppb(v/v)
Bromomethane	ND	7400	ppb(v/v)
Chloroethane	ND	7400	ppb(v/v)
Trichlorofluoromethane	ND	7400	ppb(v/v)
1.1-Dichloroethene	ND-	7400	ppb-(v/v)
1,1,2-Trichloro-	ND	7400	ppb(v/v)
1,2,2-trifluoroethane		,	
	ND	18000	ppb(v/v)
Methylene chloride	ND	7400	ppb(v/v)
1,1-Dichloroethane cis-1,2-Dichloroethene	ND	7400	ppb(v/v)
	ND	7400	ppb(v/v)
Chloroform	ND	7400	ppb(v/v)
1,1,1-Trichloroethane Carbon tetrachloride	ND	7400	ppb(v/v)
	2300000	7400	ppb(v/v)
Benzene	ND	7400	ppb(v/v)
1,2-Dichloroethane	ND	7400	ppb(v/v)
Trichloroethene	ND	7400	ppb(v/v)
1,2-Dichloropropane	ND	7400	ppb(v/v)
cis-1,3-Dichloropropene	ND	7400	ppb(v/v)
Toluene	ND ND	7400	ppb(v/v)
trans-1,3-Dichloropropene	ND ND	7400	ppb(v/v)
1,1,2-Trichloroethane	ND	7400	ppb(v/v)
Tetrachloroethene		7400	ppb(v/v)
1,2-Dibromoethane (EDB)	ND	7400	ppb(v/v)
Chlorobenzene	ND	7400	ppb(v/v)
Ethylbenzene	ND	7400	ppb(v/v)
m-Xylene & p-Xylene	ND		ppb(v/v)
o-Xylene	ND	7400	ppb(v/v)
Styrene	ND	7400	ppb(v/v) ppb(v/v)
1,1,2,2-Tetrachloroethane	ND	7400	
1,3,5-Trimethylbenzene	ND	7400	ppb(v/v)
1,2,4-Trimethylbenzene	ND	7400	ppb(v/v)
1,3-Dichlorobenzene	ND	7400	ppb(v/v)
1,4-Dichlorobenzene	ND	7400	ppb(v/v)
1,2-Dichlorobenzene	ND	7400	ppb(v/v)
Benzyl chloride	ND	15000	ppb(v/v)

(Continued on next page)

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## Client Sample ID: DUPLICATE

## GC/MS Volatiles

Lot-Sample #...: H8G030101-004 Work Order #...: KQ2F21AA Matrix...... AIR

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PARAMETER	RESULT	REPORTING LIMIT	UNITS	
1,2,4-Trichloro- benzene	ND	37000	ppb(v/v)	
Hexachlorobutadiene	ND	37000	ppb(v/v)	
	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS	-	
1,2-Dichloroethane-d4	98	(70 - 130)		
Toluene-d8	99	(70 - 130)		
4-Bromofluorobenzene	98	(70 - 130)	)	

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## METHOD BLANK REPORT

## GC/MS Volatiles

Client Lot #:		Work Order #:	KRM2F1AA	Matrix AIR
MB Lot-Sample #:	H8G170000-127	Prep Date:	07/16/08	
Analysis Date: Dilution Factor:		Prep Batch #:	8199127	

REPORTING

PARAMETER Dichlorodifluoromethane	RESULT	LIMIT	UNITS	METHOD
Dichlorodifluoromethane				
	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichloro-	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2,2-tetrafluoroethane				
Chloromethane	ND	0.50	ppb(v/v)	EPA-2 TO-15
Vinyl chloride	ND	0.20	ppb(v/v)	EPA-2 TO-15
Bromomethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Trichlorofluoromethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1-Dichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
-1,-1,2-Trichloro-	ND	-0.20	ppb (-v/-v)	-EPA-2-TO-15
1,2,2-trifluoroethane				
Methylene chloride	ND	0.50	ppb(v/v)	EPA-2 TO-15
1,1-Dichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
cis-1,2-Dichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chloroform	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,1-Trichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Carbon tetrachloride	ND	0.20	ppb(v/v)	EPA-2 TO-15
Benzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Trichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichloropropane	ND	0.20	ppb(v/v)	EPA-2 TO-15
cis-1,3-Dichloropropene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Toluene	ND	0.20	ppb(v/v)	EPA-2 TO-15
trans-1,3-Dichloropropene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2-Trichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Tetrachloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dibromoethane (EDB)	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Ethylbenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
m-Xylene & p-Xylene	ND	0.20	ppb(v/v)	EPA-2 TO-15
o-Xylene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Styrene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2,2-Tetrachloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,3,5-Trimethylbenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2,4-Trimethylbenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,3-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,4-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Benzyl chloride	ND	0.40	ppb(v/v)	EPA-2 TO-15
1,2,4-Trichloro-	ND	1.0	ppb(v/v)	EPA-2 TO-15
benzene				

#### METHOD BLANK REPORT

## GC/MS Volatiles

		REPORTING		
PARAMETER	RESULT	LIMIT	UNITS	METHOD
Hexachlorobutadiene	ND	1.0	ppb(v/v)	EPA-2 TO-15
	PERCENT	RECOVERY		
SURROGATE	RECOVERY	LIMITS		
1,2-Dichloroethane-d4	98	(70 - 130	)	
Toluene-d8	100	(70 - 130	)	
4-Bromofluorobenzene	96	(70 - 130	)	

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## NOTE(S):

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Calculations are performed before rounding to avoid round-off errors in calculated results.

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## LABORATORY CONTROL SAMPLE EVALUATION REPORT

#### GC/MS Volatiles

Client Lot #		Work Order	₿: KRM2F1AC	Matrix AIR
LCS Lot-Sample#: 1 Prep Date: Prep Batch #: Dilution Factor:	07/16/08 8199127	Analysis Da	te: 07/16/08	
		PERCENT	RECOVERY	
PARAMETER		RECOVERY	LIMITS	METHOD
1,1-Dichloroethen	e	109	(70 - 130)	BPA-2 TO-15
Benzene		133 a	(70 - 130)	EPA-2 TO-15
Trichloroethene		120	(70 - 130)	EPA-2 TO-15
Toluene		118	(70 - 130)	BPA-2 TO-15
Chlorobenzene		115	(70 - 130)	BPA-2 TO-15
			PERCENT	RECOVERY
SURROGATE			RECOVERY	LIMITS
1,2-Dichloroethan	e-d4		115	(70 - 130)
Toluene-d8			.94	(70
4-Bromofluorobenz	ene		96	(70 - 130)

## NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

Bold print denotes control parameters

a Spiked analyte recovery is outside stated control limits.

#### LABORATORY CONTROL SAMPLE DATA REPORT

#### GC/MS Volatiles

Matrix....: AIR Client Lot #...: H8G030101 Work Order #...: KRM2F1AC LCS Lot-Sample#: H8G170000-127 Prep Date....: 07/16/08 Analysis Date ..: 07/16/08 Prep Batch #...: 8199127 Dilution Factor: 1 SPIKE MEASURED PERCENT PARAMETER AMOUNT UNITS AMOUNT RECOVERY METHOD 1,1-Dichloroethene ppb(v/v)2.72 2.50 109 **EPA-2** TO-15 Benzene 2.50 3.33 a ppb(v/v)133 **EPA-2** TO-15 Trichloroethene 2.50 3.00 ppb(v/v)120 EPA-2 TO-15 Toluene 2.95 2.50 ppb(v/v)118 EPA-2 TO-15

SURROGATE	PERCENT RECOVERY	RECOVERY LIMITS
1,2-Dichloroethane-d4	115	(70 - 130)
Toluene-d8	. 94	(70 - 130)
4-Bromofluorobenzene	96	(70 - 130)

2.88

ppb(v/v)

115

2.50

#### NOTE(S):

Chlorobenzene

Calculations are performed before rounding to avoid round-off errors in calculated results.

Bold print denotes control parameters

a Spiked analyte recovery is outside stated control limits.

EPA-2 TO-15

## METHOD BLANK REPORT

## GC/MS Volatiles

Client Lot #: MB Lot-Sample #:		Work Order #: KRNQA1AA	Matrix AIR
Analysis Date: Dilution Factor:	• •	Prep Date: 07/17/08 Prep Batch #: 8199311	

		REPORTI	NG	
PARAMETER	RESULT	LIMIT	UNITS	METHOD
Dichlorodifluoromethane	ND	0.20	$\overline{ppb(v/v)}$	EPA-2 TO-15
1,2-Dichloro-	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2,2-tetrafluoroethane				
Chloromethane	ND	0.50	ppb(v/v)	EPA-2 TO-15
Vinyl chloride	ND	0.20	ppb(v/v)	EPA-2 TO-15
Bromomethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Trichlorofluoromethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1-Dichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2-Trichloro-	ND		ppb(v/v)	EPA-2_TO-15
1,2,2-trifluoroethane				
Methylene chloride	ND	0.50	ppb(v/v)	EPA-2 TO-15
1,1-Dichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
cis-1,2-Dichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chloroform	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,1-Trichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Carbon tetrachloride	ND	0.20	ppb(v/v)	EPA-2 TO-15
Benzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Trichloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichloropropane	ND	0.20	(v/v) dqq	EPA-2 TO-15
cis-1,3-Dichloropropene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Toluene	ND	0.20	ppb(v/v)	EPA-2 TO-15
trans-1,3-Dichloropropene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2-Trichloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
Tetrachloroethene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dibromoethane (EDB)	ND	0.20	ppb(v/v)	EPA-2 TO-15
Chlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Ethylbenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
m-Xylene & p-Xylene	ND	0.20	ppb(v/v)	EPA-2 TO-15
o-Xylene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Styrene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,1,2,2-Tetrachloroethane	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,3,5-Trimethylbenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2,4-Trimethylbenzene	ND	0,20	ppb(v/v)	EPA-2 TO-15
1,3-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,4-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
1,2-Dichlorobenzene	ND	0.20	ppb(v/v)	EPA-2 TO-15
Benzyl chloride	ND	0.40	ppb(v/v)	EPA-2 TO-15
1,2,4-Trichloro-	ND	1.0	ppb(v/v)	EPA-2 TO-15
benzene				

(Continued on next page)

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## METHOD BLANK REPORT

## GC/MS Volatiles

Client Lot #: H8G030101	Work Order #	: KRNQA1AA	Mar	trix AIR
PARAMETER	RESULT	REPORTING	UNITS	METHOD
Hexachlorobutadiene	ND	1.0	ppb(v/v)	EPA-2 TO-15
SURROGATE	PERCENT RECOVERY	RECOVERY LIMITS	_	
1,2-Dichloroethane-d4	99	(70 - 130)		
Toluene-d8	98	(70 - 130)		
4-Bromofluorobenzene	99	(70 - 130)		

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## NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

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#### LABORATORY CONTROL SAMPLE EVALUATION REPORT

#### GC/MS Volatiles

Client Lot #: H8G030101 LCS Lot-Sample#: H8G170000-311 Prep Date: 07/17/08 Prep Batch #: 8199311 Dilution Factor: 1		#: KRNQA1AC	
PARAMETER 1,1-Dichloroethene Benzene Trichloroethene Toluene Chlorobenzene	PERCENT <u>RECOVERY</u> 84 105 81 115 106	RECOVERY LIMITS (70 - 130) (70 - 130) (70 - 130) (70 - 130) (70 - 130)	METHOD KPA-2 TO-15 KPA-2 TO-15 KPA-2 TO-15 KPA-2 TO-15 KPA-2 TO-15
SURROGATE 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene	• • • • • • • • •	PERCENT <u>RECOVERY</u> 102 101 91	RECOVERY LIMITS (70 - 130) (70 - 130) (70 - 130)

#### NOTE(S):

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Calculations are performed before rounding to avoid round-off errors in calculated results.

Bold print denotes control parameters

#### LABORATORY CONTROL SAMPLE DATA REPORT

#### GC/MS Volatiles

Client Lot #: H8G030101 LCS Lot-Sample#: H8G170000-311 Prep Date: 07/17/08 Prep Batch #: 8199311 Dilution Factor: 1		#: KRNQA1AC te: 07/17/08		ix	: AIR
	SPIKE	MEASURED		PERCENT	
PARAMETER	AMOUNT	AMOUNT	UNITS	RECOVERY	METHOD
1,1-Dichloroethene	2.50	2.10	ppb(v/v)	84	BPA-2 TO-15
Benzene	2.50	2.63	ppb(v/v)	105	<b>EPA-2 TO-15</b>
Trichloroethene	2.50	2.02	ppb(v/v)	81	EPA-2 TO-15
Toluene	2.50	2.88	ppb(v/v)	115	EPA-2 TO-15
Chlorobenzene	2.50	2.65	ppb(v/v)	106	RPA-2 TO-15
		PERCENT	RECOVERY		
SURROGATE		RECOVERY	LIMITS		
1,2-Dichloroethane-d4		102	(70 - 130)		
Toluene-d8	an an antice of the second	. 101	(70 - 130)		
4-Bromofluorobenzene		91	(70 - 130)		

#### NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

Bold print denotes control parameters

### **APPENDIX B**

### **AIR PERMEABILITY DATA SUMMARY**

### SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA INC. 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

### **August 2008**



#### AIR PERMEABILITY DATA SUMMARY

Point permeability testing (PPT) was conducted in each of the six treatment areas at the Site from June 30 to July 3, 2008 as part of the pre-work plan data collection event. The standard operating procedures (SOPs) for collecting and evaluating point permeability data and the calculated relative air permeability values for each significant geologic layer are provided below. PPT was conducted by XDD, LLC per the attached SOP (Attachment A: XDD SOP No. POINTPERM). A combination of extraction and injection data from a variety of locations and depths were collected to calculate an average permeability for each of the four geologic layers. Figure 1 of the SVE Pilot Testing Work Plan shows the vapor probe locations, and Table B.1 provides a summary the PPT results. The air permeability values were calculated using the MDFIT<sup>1</sup> modeling software. Review of the PPT results revealed several representative subsets of data to be used in the MDFIT modeling for air permeability calculations. Vapor point sites that were not functioning properly during the extraction or injection testing, or, where vacuums could not be measured due to the extraction of liquid during the extraction test were not included in the point permeability evaluation. A total of four representative PPT result data sets within each geologic layer (sandy fill, upper silty sand, intermediate silty clay, and lower silty sand) were selected for the MDFIT modeling for permeability calculation.

For the MDFIT modeling, the soils in each geologic layer were assumed to be isotropic (homogenous in the horizontal and vertical directions). In addition, the model assumed that confining layers were not present above or below each of the sandy fill, upper silty sand, and intermediate silty clay layers. The soils in the lower silty sand layer were modeled with an upper confining layer due to the presence of the relatively lower permeability silty clay layer above this layer. A summary of the average relative air permeability values calculated for each geologic layer is provided in Table B.2.

<sup>&</sup>lt;sup>1</sup> A two-dimensional analytical air flow model developed by Marley, Li and Droste (August 1994) based on the Baehr and Hault paper titled "Evaluation of Unsaturated Zone Air Permeability through Pneumatic Test" (Water Resources Research, Vol. 27, October 1991).



TABLES

### APPENDIX B AIR PERMEABILITY DATA SUMMARY

### **SVE PILOT TESTING WORK PLAN**

## Table B.1 Summary of Point Permeability Testing Results SVE Pilot Testing Work Plan, Appendix B

W.G. Krummrich Facility, Sauget, IL

August 2008

							Extraction	Test Results	Injection	Test Results	VOC Vapor Measuremen
Area	Vapor Probe ID	Screen Interval	Depth to TOS (ft. bg)	Depth to BOS (ft. bg)	Depth to Perched Water (ft. bg)	Geologic Layer	Air Flow Rate (scfm)	Wellhead Vacuum ("Hg)	Air Flow Rate (scfm)	Wellhead Pressure (psi)	C <sub>i</sub> (ppmv)
Big Mo	VP-A01	Intermediate	10	11	5	Silty Clay	1.35	28	1.4	4	315
-						(high plasticity)	nc	nc	3.25	4.2	
	VP-A02	Shallow	5	6	4	Silty Sand	1.75 nc	26 nc	2 4.75	0.39 0.55	1,133
	VP-A03	Shallow	8	9	4.5	Fine Sand, some silt and gravel	1 1.85	22 23	1.5 3.1	3.6 4.8	2,500
Former Chlorobenzene	VP-B01	Shallow	4	5	dry	Silty Sand	1.75 3.25	5 5.5	1.7 4.2	0.2 0.75	23
Storage Area		Intermediate	9	10	6	Silty Clay (high plasticity)	1.5	20	nc	nc	6
North Tank Farm	VP-C01	Shallow	4	5	dry	Sand (some gravel & coal pieces)	1.75 3.5	3.5 4	2 4.1	0.25 0.5	108
		Deep	9	10	6.5	Silty Sand	2 3.5	4 4.1	2.2 4.1	0.5 1	36
Near Little Mo	VP-D01	Shallow	4	5	dry	Silty Sand	2 4.1	6.1 8.5	2 4	0.4 1	53
		Deep	8.5	9.5	5.5	Silty Sand	nc nc	nc nc	2 3.85	2.6 3.2	n/a
Former Steamer Overhead Tank	VP-E01	Shallow	4	5	4.5	Silty Sand with some Gravel Fill	2 3.8	6.5 8	nc nc	nc nc	100
		Deep	10.5	11.5	6	Fine Sand (some silt, clay lenses)	nc nc	nc nc	1.5 2.9	4 5	n/a
Former Benzene Pipeline	VP-F01	Shallow	4	5	2.5	Silty Sand	0.75 1.25	20.5 22	1.5 3.25	2.8 4.25	1,301
i ipenne		Intermediate	6.5	7.5	2.5	Silty Clay	0.75 1.5	10 20	1.5 3.25	3 4.5	4,698

Notes:

1. BOS = Bottom of Screen; TOS = Top of screen

2. "Hg = inches of mercury; "H<sub>2</sub>O = inches of water

3.  $cm^2 = square centimeters$ 

4. PID = photoionization detector

5.  $C_i$  = Initial PID reading;  $C_f$  = Final PID Reading

6. ppmv = parts per million by volume

7. nc = not completed due to perched water

8. scfm = standard cubic feet per minute

9. psi = pounds per square inch

10. ft bg = feet below grade

11. n/a = not available

12. A total of four representative PPT result data sets within each geologic layer were selected from this table to be used in air permeability calculations using MDFIT modeling.

or Discharge aents via PID					
	C <sub>f</sub> (ppmv)				
	94				
	2,701				
	1,962				
	13				
	6				
	68				
	36				
	29				
	n/a				
	34				
	n/a				
	770				
	1,800				



## Table B.2Summary of Calculated Air Permeability Results

SVE Pilot Testing Work Plan, Appendix B

W.G. Krummrich Facility, Sauget, IL

#### August 2008

			Air Permeability <sup>b</sup>
Geologic Layer	Location	Vapor Probe ID	$(cm^2)$
Sandy Fill <sup>a</sup>	North Tank Farm	VP-C01	6.58E-09 1.16E-08 5.08E-08
	Former Steamer Overhead Tank	<b>VP-E01</b>	7.01E-09
		Average:	1.90E-08
	Big Mo	VP-A02	6.04E-08
Upper Silty Sand <sup>a</sup>	Former Chlorobenzene Storage	VP-B01	2.63E-09
	Area		4.47E-09
	Near Little Mo	VP-D01	5.01E-09
		Average:	1.81E-08
	Big Mo	VP-A01	1.33E-09
Intermediate Silty Clay <sup>c</sup>	Former Chlorobenzene Storage Area	VP-B01	1.65E-09
	Former Benzene Pipeline	VP-F01	5.97E-09
		Average:	3.86E-09
			1.42E-08
Laura Cilta Can J <sup>a</sup>	North Tank Farm	VP-C01	3.34E-08
Lower Silty Sand	$ \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$		
	Former Steamer Overhead Tank	VP-E01	4.09E-09
		Average:	2.09E-08

Notes:

a. Presence of perched water during testing likely influenced the permeability data, and the permeabilities of the fill, upper silty sand and lower silty sand layers may be higher under drier conditions.

b. The relationship between air permeability and hydraulic conductivity is a factor of approximately  $10^{-5}$ , i.e., an air permeability of 1 x  $10^{-8}$  cm<sup>2</sup> is approximately equal to a hydraulic conductivity of  $10^{-3}$  cm/second for comparison of soil type for water flow.

c. Per field observations, the intermediate silty clay layer is highly plastic and low-permeable. However, the air permeability results show higher than expected permeability for this layer. This higher estimated permeability may be due to short-circuiting between the silty clay layer and the adjacent silty sand layers.





### ATTACHMENT A XDD SOP No.: POINTPERM

### APPENDIX B AIR PERMEABILITY DATA SUMMARY

### SVE PILOT TESTING WORK PLAN

#### XDD, LLC

#### STANDARD OPERATING PROCEDURE (SOP)

#### DETERMINATION OF VADOSE ZONE AIR PERMEABILITY UTILIZING POINT PERMEABILITY TEST PROCEDURES

#### **XDD SOP NO.: POINTPERM**

#### **1.0 OBJECTIVE**

To establish specific guidelines for the measurement, collection and determination of vadose zone air permeabilities utilizing point permeability test (PPT) procedures.

#### 2.0 **DEFINITIONS**

- 1. Vadose Zone the zone of unsaturated soil (geological unit) above the capillary fringe of the ground water table.
- 2. Vadose Zone Air Permeability a measure of the air permeability within a given vadose zone soil unit. As is applies to vapor intrusion evaluation and soil vapor extraction (SVE), air permeability is an in-situ measurement of the intrinsic potential for air movement within a given soil unit.
- 4. Point Permeability in contrast to the determination of vadose zone permeability utilizing standard SVE parameter evaluation test (PET) procedures, point permeability allows for the determination of soil air permeability at specific locations or "points" within the vadose zones and is typically conducted in conjunction with soil gas sampling surveys. Point permeability analysis offers the following advantages over standard PET Procedures: 1) depending upon site-specific conditions, the procedure is typically quick and inexpensive and thereby allows for the collection and evaluation of a matrix of air permeabilities; 2) offers the ability for enhanced evaluation of specific, finite locations; zones within the vadose zone. These "points" can be analyzed/modeled collectively to develop a more accurate total representation of a site this is particularly applicable for vadose zones suspected of significant soil heterogeneities.

#### 3.0 EQUIPMENT DESCRIPTION

Key equipment necessary to collect point permeability data includes:

- 1. Sample Pump used to draw air from the sample point within the vadose zone. Sample pump should be capable of moving 5 cubic feet per minute (cfm) of air at vacuums of up to 25 inches of mercury (in Hg).
- 2. Air Flow Rotometer used to measure the airflow on the pressure side of the sample pump. Airflow will be measured on the pressure side of the pump in order to avoid the discrepancies between actual cubic feet per minute (acfm) and standard cubic feet per minute (scfm) at high vacuum(s). The flow meter should be capable of measuring airflow rates up to 5 cfm.
- 3. Vacuum Gauges should include a "kit" of several Magnehelic differential pressure gauges (reading inches of water, in H<sub>2</sub>O). The kit should be capable of reading from approximately 0.005 in H<sub>2</sub>O to 25 in Hg. A gauge used for a specific monitoring point must have a range allowing for the accurate reading of vacuum or pressure.
- 4. Vapor Probe Vapor probes are permanent/temporary vadose zone soil vapor monitoring points.
- *Note:* Due to the extent of time required to achieve steady state conditions and the potential for the sampling pump to work under high vacuums, it is recommended that sampling pump be operated from a generator or non-exhaustable source of power.

#### 4.0 **PROCEDURE**

- 1. Prepare the PPT system by attaching PPT manifold to the vacuum side of the sample pump.
- 2. Attach the PPT system to the vapor probe with appropriate sized tubing and fittings. As a general rule, the inside diameter of tubing and fittings should be greater than 3/16 inch in order to avoid significant headlosses across the equipment.

(Note: To measure the headloss associated with the PPT equipment, operate the PPT system in air (i.e., without a connection to the vapor probe) using the same dimension piping (length and diameter) as the vapor probe. Measure the associated vacuum and flow rate at several flow rates and record the data. The measured vacuum for a particular flow rate is the headloss inherent in the equipment, tubing and piping. If several vapor probes are to be measured, measure the headloss over the range of expected piping lengths.)

- 3. Turn the pump on with the flow valve open completely.
- 4. Allow the system to stabilize and measure air flow rates and pressures under these conditions.

- 5. At this point there will likely be one of two conditions. Either higher flow at low vacuum or lower flow at high vacuum. Depending on which condition, try and reduce the flow or the vacuum by 50% by closing the flow control valve to the system.
- 6. Repeat the previous step if possible. Try to get 3 readings per location. Keep in mind that this may not be possible depending on soil type.
- 7. Following stabilization of point permeability test parameters, be sure to record the measured vacuum and flow rate in association with the sample location's grid and depth coordinate(s).
- 8. Attach the PPT manifold on the pressure side of the sample pump, and repeat the test with air injection. Be sure to start with the lowest flow rate recorded during the PPT test under vacuum conditions. Do not apply excessive pressure as it may create fractures in the formation, depending on soil type.

### **APPENDIX C**

### **QUALITY ASSURANCE PROJECT PLAN**

### SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA INC. 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

### **August 2008**



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#### 1.0 PROJECT DESCRIPTION

This plan describes the Quality Assurance Project Plan (QAPP) procedures to be conducted as part of the Soil Vapor Extraction (SVE) pilot test activities at the W.G. Krummrich plant (Site) in Sauget, Illinois. This plan has been developed in accordance with the general guidelines provided by the United States Environmental Protection Agency (U.S. EPA) documents "Guidance for the Data Quality Objectives Process" EPA QA/G-4 and "Guidance for Quality Assurance Project Plans" EPA QA/G-5, February 1998.

#### 1.1 SAMPLING MEDIA AND PARAMETERS

The purpose of this QAPP is to establish the quality requirements for the sampling and analytical data to be collected during the pilot testing activities. Table 4 of SVE Pilot Testing Work Plan summarizes the monitoring plan. Table C.1 presented in this QAPP presents only the sampling plan portion of the table (excluding SVE operation parameter monitoring). The Sampling and Analysis Plan (SAP) attached as Appendix D of the SVE Pilot Testing Work Plan provides detailed sampling and analytical procedures. Specifically, the sampling media and parameters include:

- a) Field-screening of soil cores using a photo-ionization detector (PID);
- b) Soil sample collection and analysis at Test America Laboratories of Savannah, Georgia (Test America) for volatile organic compounds (VOCs) via U.S. EPA Method 8260B (primary VOCs detected in the Big Mo treatment area include benzene, chlorobenzene, 1,2-dichlorobenzene and 1,4-dichlorobenzene);
- c) Soil vapor sample collection (from SVE wells and vapor probes/points; from SVE manifold during system operation; and discharge air pre- and post-treatment) and field-screening using a PID; and
- d) Soil vapor sample collection (from SVE wells and vapor probes/points; from SVE manifold during system operation; and discharge air pre- and post-treatment) and analysis at Test America for VOCs via U.S. EPA Method TO-15 (primary VOCs detected in the Big Mo treatment area include benzene, chlorobenzene, 1,2-dichlorobenzene and 1,4-dichlorobenzene).

Further, as discussed in the Health and Safety Plan (HASP) attached as Appendix E of the SVE Pilot Testing Work Plan, ambient air will be monitored using a PID and if required, by Draeger Tubes for benzene concentrations.

#### **1.2 DATA QUALITY OBJECTIVES**

#### 1.2.1 Soil Screening by PID

Soil field-screening data by PID is a screening level data that will be used to semi-quantitatively estimate the level of VOCs in soil, and therefore is considered Level I data quality objectives (DQO) data. The field-screening data will also be considered with laboratory analytical data to develop a rough estimate of soil concentrations and associated VOC mass. Field screening of soil will be completed during the baseline, intermediate and post-pilot test characterization of soil VOCs.

#### 1.2.2 Soil Laboratory Analysis

Select soil samples will be submitted to Test America for VOC analysis via U.S. EPA Method 8260B, and the data will be used to evaluate the effectiveness of the SVE pilot test. Therefore, this data is considered Level IV DQO data. The laboratory analysis of select soil samples will occur during the baseline, intermediate and post-pilot test characterization of soil VOCs.

#### 1.2.3 SOIL VAPOR ANALYSIS BY PID

Data collected by the analysis of soil vapor (from SVE wells and vapor probes/points; from SVE manifold during system operation; and discharge air pre- and post-treatment) by a PID is a considered a Level I DQO data that will be used to semi-quantitatively estimate the level of VOCs. The PID data will be considered with laboratory data to estimate the level of VOC removal by SVE, and assess the level of treatment by the soil vapor treatment unit.



Field analysis of soil vapor will be conducted during baseline soil vapor sampling from SVE wells and vapor probes/points prior to the start of the SVE system. Additional field analysis of soil vapor will be conducted during SVE design parameter testing and performance evaluation testing frequencies provided in Table C.1.

#### 1.2.4 Soil Vapor Laboratory Analysis

Select soil vapor samples will be submitted to Test America for VOC analysis via U.S. EPA Method TO-15, and the data will be used to evaluate the effectiveness of the SVE pilot test. Therefore, this data is considered Level IV DQO data. The laboratory data for vapor will be used to estimate the VOC mass removal by the SVE system. Also, data collected from the air treatment effluent will be used to ensure compliance with the applicable air permit requirements. The laboratory analysis of select soil vapor samples will be conducted during the baseline sampling, design parameter evaluation testing and SVE performance evaluation testing.

#### 1.2.5 Ambient Air Monitoring by PID and Draeger Tubes

As outlined in the HASP included as Appendix E of the SVE Pilot Testing Work Plan, ambient air quality will be monitored during the drilling (well installation and soil sampling) work using a PID and draeger tubes (for benzene data), if required. Additional ambient air monitoring will be conducted when the air injection equipment is operational during the SVE pilot test. This data is collected for health and safety screening, and therefore, considered Level I DQO data. This data will be used to assess the level of VOCs in breathing space for the evaluation of the appropriate level of personal protection equipment (PPE), as outlined in the HASP.

#### 2.0 QA/QC REQUIREMENTS

The overall quality assurance/quality control (QA/QC) objective is to develop and implement procedures for field sampling, laboratory analysis and reporting to obtain scientifically valid results and to meet DQOs identified in Section 1.2.

#### 2.1 LEVEL OF QUALITY CONTROL EFFORT

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.

Field analytical activities include a) field screening of soil samples for VOCs (using PID); b) field screening of soil vapors for VOCs (using PID); and c) ambient air monitoring for VOCs (using PID) and benzene (using draeger tubes), if required. Direct reading field instrumentation will be used for these measurements per manufacturers' instructions. Quality control will be provided by field calibration (provided in Section 3.1) and equipment maintenance. Where applicable, two types of samples will be used: equipment blanks and field duplicates. No additional quality control measures are deemed necessary.

Field sampling quality control will be monitored through the collection and laboratory analysis of quality control samples. Where applicable, two types of samples will be used: trip blanks and field duplicates. Equipment blanks are not proposed as all samples for laboratory analysis will use dedicated equipment. Additionally, matrix spike/matrix spike duplicate (MS/MSD) samples will be collected for laboratory use. The rationale and frequency of sample collection are listed below for each type of QA sample.

• Field duplicate analysis: one per ten samples to assess precision (i.e., sampling and analytical reproducibility). Field duplicates are required for each matrix sampled.

- Trip blank analysis: one per cooler of samples to assess the integrity of the samples during shipment to the laboratory and ensure that no cross-contamination of samples occurs during shipment.
- Equipment blank analysis: one per ten samples, or at least one per day, to assess the decontamination procedures at the site. Equipment blanks are required for each type of non-dedicated equipment used at the Site.
- MS/MSD analysis: one per twenty samples to assess the effect of the sample matrix on laboratory analytical results.

#### 2.1.1 LABORATORY QUALITY CONTROL

Analytical quality control will be monitored through laboratory quality control checks. The DQO level for the laboratory analysis will be 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 quality assurance 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 the SAP appendix of the SVE Pilot Testing Work Plan.
- 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.

The method detection limit (MDL), practical quantitation limit, and holding time for the laboratory VOC analysis according to U.S. EPA Method 8260B and TO-15 are presented in Table C.2.

#### 2.1.2 CONTROL LIMITS

Control limits are the maximum and/or minimum values defining a range for a specific parameter, as outlined within each analytical procedure that is considered to satisfactorily meet quality control criteria. When the parameter falls outside that range, the procedure is considered to be out-of-control. Whenever the analytical procedure is or becomes out-of-control, corrective action must be taken to bring the analysis back into control. The corrective action will include: (1) finding the cause of the problem, (2) correcting the problem, (3) demonstrating the problem has been corrected by reanalyzing appropriate laboratory reference samples, and (4) repeating the analyses of any investigative samples that may have been affected by the control problem.

Exceptions may be made on a case-specific basis. If the control limit is technically impracticable for a particular sample or analysis, documentation and narrative explanation will be submitted with the data report and raw data. The documentation will include evidence that a good faith effort [generally two attempts to re-analyze the sample(s)] was made to meet the control limit.

#### 2.2 DATA QUALITY INDICATORS

Data quality indicators (DQIs) that will be evaluated upon completion of both field activities and laboratory analysis include precision, accuracy, representativeness, completeness, comparability, and adequacy, as defined below:



• **Precision:** An indicator of the reproducibility of measurements under a given set of conditions, precision is usually stated in terms of relative standard deviation or relative percent difference. 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. Precision will be expressed as RPD:

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

Where,

RPD = Relative Percent Difference

D<sub>1</sub> = First Duplicate Value (percent recovery)

D<sub>2</sub> = Second Duplicate Value (percent recovery)

• 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 MS/MSD samples and/or laboratory control spike (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 U.S. EPA Quality Control Samples or Standard Reference Materials. Accuracy is expressed as percent recovery:

Percent Recovery =  $Q_d/Q_a \times 100\%$ 

Where,

 $Q_d$  = quantity determined by analysis

 $Q_a$  = true or accepted reference value



- 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. Sensitivity will be evaluated by comparing the results to the designated quantitation limits presented on Table C.2.
- **Representativeness:** This parameter expresses the degree to which the sample data accurately and precisely represent a characteristic of the environmental condition. Representativeness is a qualitative parameter best addressed by ensuring that the proposed sampling techniques and the rationale used to select sampling locations are consistent with the overall project objectives.

Sampling representativeness for baseline, intermediate, and post pilot test soil sampling will be achieved for the remediation program by collecting multiple samples within the pilot test treatment areas, as discussed in Section 4 of the SVE Pilot Testing Work Plan.

• **Completeness:** Completeness (%C) is defined as the number of measurements judged valid compared to the number of measurements needed to achieve a specific level of confidence in decision-making, as calculated by the following formula:

$$\% C = \frac{V}{N} \times 100\%$$

Where: V = number of measurements judged valid; and

N = number of measurements planned

Based on the number of samples to be collected and the complexity of the sampling analysis program, the completeness goal will be >90%. Should this goal not be achieved, the data set will be evaluated to determine the impact on overall data quality.

• **Comparability:** This qualitative parameter indicates the level of confidence with which one data set may be compared with another. Comparability will be achieved by using standard operating procedures (SOPs) developed for the analytes for the matrices collected in



accordance with U.S. EPA guidance, and reporting analytical data in appropriate units. The specific comparisons that need to be made are with the air discharge requirements. In order to be comparable, the reporting detection limits of air discharge analytical results for each analyte must be lower than the discharge requirements for that analyte.

#### 3.0 MEASUREMENTS AND DATA ACQUISITION

Measurements and data acquisition systems encompass the procedures by which samples are collected and analyzed. Quality control measures associated with the sampling process design and implementation are described in the SAP attached as Appendix D of the SVE Pilot Testing Work Plan. The following discussion addresses the procedures to be followed in the field and analytical laboratory.

#### 3.1 CALIBRATION PROCEDURES AND FREQUENCY

Equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specification.

#### 3.1.1 FIELD INSTRUMENTS/EQUIPMENT

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment problems are not overlooked and all necessary repairs to equipment have been carried out.

Calibration of field instruments will be performed at intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments include a PID. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service.

#### 3.1.1.1 Portable MiniRae 2000 Photo-ionization Detector (or Equivalent)

The MiniRAE 2000 is a PID equipped with a 10.6 electron-volt lamp and will be calibrated using a 100 ppmv isobutylene calibration gas standard in air.

The response of the MiniRAE 2000 in the field will change with environmental (humidity, temperature, shock/vibration) or operational conditions. In order to qualify the performance of the PID, calibrations or calibration checks should be performed on a regular basis based on the conditions for which it will be used.

The field calibration check is designed to allow a rapid, yet controlled, one-point evaluation of the response of the MiniRAE 2000 and to recalibrate the instrument if necessary. The procedures are as follows:

- 1. Using a short length of Teflon tubing, connect the calibration cylinder regulator to the on/off valve of a Tedlar bag.
- Fill the Tedlar bag with approximately one liter of the gas standard (100 ppmv isobutylene). Close the cylinder, close the bag and disconnect the canister from the regulator valve.
- 3. Connect the sampling probe of the PID to the Tedlar bag using a short section of Teflon tubing and open the Tedlar bag valve.
- 4. Record the total VOC reading from the instrument once a steady reading is observed. A dedicated instrument calibration logbook should be used to record all calibration activities.
- 5. Compare the instrument's response to the calibration gas concentration. If the response is in agreement within 10%, clean up materials and record the result in the calibration logbook. If not, recalibrate the instrument once more to verify that the reading is within 10% of the gas standard and record the results in the logbook.

#### 3.1.2 LABORATORY INSTRUMENTS

All calibration procedures that will be used by Test America are detailed in the quality manuals and/or SOPs, which are included in the SAP attached as Appendix D of the SVE Pilot Testing Work Plan.



#### 3.1.3 LABORATORY QUALIFICATIONS

Test America Laboratories will be used for soil and soil vapor sample analytical testing. Test America will conduct analyses in accordance with the demonstrated method proficiency requirements prescribed in the "Test Methods for Evaluating Solid Waste", 3<sup>rd</sup> edition with updates, 1986 and 40 CFR Part 60 Appendix B, where applicable.

#### 3.1.4 ANALYTICAL TEST METHODS

The analytical test methods and parameters to be run for this sampling and analysis program are listed in Table C.2.

#### 3.1.5 CALIBRATION AND MAINTENANCE PROCEDURES

Laboratory instruments will be calibrated in accordance with the laboratory's applicable standard operating procedures, the manufacturer's recommendation for the equipment, and the promulgated U.S. EPA methodology. Equipment will be calibrated using reference standards having known relationships to nationally recognized standards or accepted values of physical constants. The laboratory will document all calibration procedures conducted during analytical testing of samples obtained during this sampling program and provide this documentation upon request.

The laboratory will be responsible for maintaining all analytical testing equipment as specified in the appropriate test methods. Trained personnel will operate, calibrate, and provide maintenance on the equipment or instruments, and qualified laboratory personnel or service technicians will perform repairs. All maintenance and repair procedures will be documented in the instrument logbooks and archived at the laboratory.



#### 3.1.6 INTERNAL QUALITY CONTROL

Internal QC measures will be performed through the collection and evaluation of both field and laboratory QC samples. Field QC samples will include field duplicates that are discussed in the SAP. Laboratory QC samples will include method blanks, laboratory control spike (LCS) samples, and matrix spike and matrix spike duplicate (MS/MSD) analyzed in accordance with the approved U.S. EPA analytical protocols.

#### 4.0 ASSESSMENT AND OVERSIGHT

Assessment and oversight activities will be conducted to evaluate the effectiveness of the implementation of the QAPP. The quality assurance (QA) activities described below relate to field sampling only. Since the subcontractor laboratory is routinely assessed according to the audit/certification programs, no additional oversight of this facility is deemed necessary for the SVE pilot testing.

#### 4.1.1 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 the QAPP. 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.

#### 4.1.2 Reports to Management

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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. Quality assurance reports ensure that QA/QC objectives are followed. These reports will be submitted to management and will include:

- Individuals submitting and receiving reports;
- Type of report;
- Written or oral frequency;
- Interim or final QA/QC reporting for final reports;
- Report contents;



- Summary of QA/QC programs, training and accomplishments;
- Results of technical systems and performance evaluations;
- Significant QA/QC problems, recommended solutions and results of corrective actions;
- Data quality assessment in terms of precision, accuracy, representativeness, completeness, comparability, and MDL;
- Indication of whether the QA objectives were met; and
- Limitations on use of the measurement data.

#### 5.0 DATA VERIFICATION AND REPORTING

The data validation process for chemical analyses will begin upon receipt of data results from Test America. This process involves evaluating the analytical data with respect to the DQRs in this section. The QA manager will be responsible for resolving any outstanding data issues and determining the certainty with which data may be used in making remedial effectiveness decisions. All data verification and usability activities will be conducted in accordance with this QAPP.

#### 5.1.1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

Data review and verification of data packages will be performed. Specific criteria from all QA/QC measures supporting the analytical results will be used to determine whether to accept, reject or qualify the analytical results.

#### 5.1.2 VALIDATION AND VERIFICATION REQUIREMENTS

Validation of the analytical data will be performed by the project QA Officer or designee utilizing the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-99/008, October 1999. This protocol will provide the basis for data review by establishing the specific objectives, defining the evaluation process, and identifying the actions while incorporating the specific quality control limits for the analytical protocols.

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, the Level IV data packages will be independently validated using U.S. EPA-based guidelines. The validated project data will be evaluated for overall usability with the DQIs presented in Section 2.2.

#### 5.1.3 DATA REPORTING

The laboratory data will be provided to XDD in both electronic and printed 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<sup>®</sup> Access database, or similar.



TABLES

### APPENDIX C QUALITY ASSURANCE PROJECT PLAN

SVE PILOT TESTING WORK PLAN

#### Table C.1 Sampling Plan Quality Assurance Project Plan W.G. Krummrich Facility, Sauget, IL August 2008

Event	Task	Description	Sampling Location	Measurement Location	Frequency	Samples per Event	Analytical Method
Baseline	e Sampling						
	Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once	26	8260B
	Soil Vapor Sampling	Soil vapor samples from SVE wells	SVE wells	Field measurement Laboratory analysis	once	26 3	PID TO-15
		Soil vapor samples from vapor probes/points	Vapor probes/points	Field measurement Laboratory analysis	once	25 3	PID TO-15
Design I	Parameter Evaluation Tes	ting					
	Extracted Soil Vapor Sampling	SVE stream from higher and lower permeability layers	Process equipment	Field measurement	daily	2	PID
	Air Treatment Sampling	Air permit compliance	post-treatment	Laboratory analysis	per Perr	nit	TO-15
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	twice a week	1	PID
SVE Per	rformance Evaluation Tes	ting					
	Soil Vapor Sampling	Soil vapor samples from SVE wells	SVE wells	Field measurement	once at starup once at mid-point	19	PID
				Laboratory analysis	once at end	3	TO-15
		Soil vapor samples from vapor probes/points	Vapor probes/points	Field measurement Laboratory analysis	once at end once at end	25 3	PID TO-15
	Extracted Soil Vapor	SVE streams from the three SVE mainlines (fill and upper silty sand layers, intermediate silty clay	Process equipment	Field measurement	daily - 1st week biweekly - 1st month monthly - afterwards	4	PID
	Sampling	layer and lower silty sand layer) + combined SVE stream prior to the air treatment unit		Laboratory analysis	3 times - 1st week biweekly - 1st month monthly - afterwards	4	TO-15
	Soil Sampling	Soil samples via Terrcore at Test mid-point	See Figure 9	Laboratory analysis	once	26	8260B
	Air Treatment Sampling	Air permit compliance	post-treatment	Laboratory analysis	per Perr	nit	TO-15
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	monthly	1	PID
Post-Pil	lot Test Sampling						
	Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once	26	8260B

#### Summary of Sampling for Laboratory Analysis

	Samples per Event	Frequency of Events	Total Number of Samples
Soil Sample Analysis via 8260B Method			
Baseline sampling	26	1	26
Sampling at mid-point of Effectiveness Testing	26	1	26
Post-pilot test sampling	26	1	26
Total Number of Soil Analyticals (excluding (	QA/QC samples)		78
Soil Vapor Sample Analysis via TO-15			
Baseline SVE well sampling	3	1	3
Baseline vapor probe/point sampling	3	1	3
SVE well sampling during Effectiveness Testing	3	3	9
Vapor probe/point sampling during Effectiveness Testing	3	1	3
Extracted soil vapor sampling during Effectiveness Testing	4	7	28
Total Number of Soil Vapor Analyticals (excl	uding QA/QC samples)		46
Air Permit Compliance Monitoring			
Vapor discharge sampling per air permit requirements	1	per Permit	to be determined
Total Number of Vapor Discharge Analytical	s (excluding QA/QC sample	s)	to be determined

Notes:

PID - photoionization detector

8260B - U.S. EPA Method 8260B for VOC analysis TO-15 - U.S. EPA Method TO-15 for VOC analysis Permit - Air permit

SVE - soil vapor extraction



# Table C.2 Project Laboratory Analyte List Quality Assurance Project Plan W.G. Krummrich Facility, Sauget, IL

August 2008

Analysis	Matrix	DQO	Method	MDL*	PQL*	Holding Time	Preservation	Sample Container
VOCs	soil	IV	US EPA 8260B	0.32 ug/kg	1 ug/kg	14 days	Deionized water, methanol, ice (< 4°C)	5 gram Glass Jar
VOCs	soil vapor	IV	EPA-2 TO-15	0.20	ppbv	72 hours	ice (< 4°C)	Tedlar Bag

Notes:

DQO = data quality objective

MDL = method detection limit

PQL = practical quantitation limit

\* =for benzene

VOCs = volatile organic compounds

ug/kg = micrograms per kilogram

ppbv = parts per billion by volume

°C = degrees Celcius



### **APPENDIX D**

### **SAMPLING AND ANALYSIS PLAN**

### SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA INC. 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

### **August 2008**

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## 1.0 PROJECT DESCRIPTION

This plan describes the Sampling and Analysis Plan (SAP) procedures to be conducted as part of the Soil Vapor Extraction (SVE) pilot test activities at the W.G. Krummrich plant (Site) in Sauget, Illinois.

## **1.1 SCOPE AND OBJECTIVE**

The objectives of the SVE pilot testing work are to: a) Evaluate an optimal SVE system design to treat the unsaturated soils (i.e., fill, upper silty sand, intermediate silty clay and lower silty sand) and determine the expectations for full-scale SVE system operation; and b) Evaluate full-scale SVE design and operation parameters. Table 4 of the SVE Pilot Testing Work Plan shows the monitoring plan designed to collect data for evaluation to achieve the pilot test objectives.

The specific objectives of the sampling program are to:

- Evaluate the effectiveness of the SVE technology: This evaluation will be completed through the collection of representative soil samples before, during and after treatment. Baseline representative soil samples will be collected to characterize pre-pilot test soil volatile organic compound (VOC) concentrations (primary VOCs detected in the Big Mo include benzene, chlorobenzene, 1,2-dichlorobenzene 1.4treatment area and dichlorobenzene). Similarly, intermediate and post-pilot test representative soil samples will be collected to characterize intermediate and post-treatment soil VOC concentrations, respectively. A comparison of the baseline, intermediate and post-pilot test soil data will provide information related to the effectiveness of the SVE technology. Additionally, extracted soil vapor sampling from the SVE mainlines will occur during the operation to provide an estimate of mass removal from the system. Soil vapor samples from SVE wells and vapor probes/points to evaluate the change in VOC data over the performance of the pilot test compared to the baseline condition.
- Monitor air discharge: Air sampling will be performed to assess treatment of extracted soil vapor prior to discharge to atmosphere and demonstrate compliance with the air permit.



To achieve these project objectives, the sampling and analysis program presented in Table D.1 will be implemented.

#### **1.2 NETWORK DESIGN AND RATIONALE**

The sample network and rationale are presented in Section 4 of the SVE Pilot Testing Work Plan. Sample locations for each area are presented in Figure D.1. Sample locations have been selected to achieve two goals: 1) provide data to refine the mass estimate of the VOCs in the test area; and 2) provide data to evaluate the effectiveness of SVE in treating the unsaturated soils in the test area.

## **1.3 SAMPLING PARAMETERS AND FREQUENCY**

Table D.1 summarizes the sampling parameters and frequency for the SVE pilot testing work. Sampling will occur as part of the four phases of the pilot test: baseline sampling, during design parameter evaluation testing, during SVE performance evaluation testing, and post-pilot test sampling.



#### 2.0 SAMPLING PROCEDURES

Overall sampling procedures, including field documentation requirements, quality assurance/quality control (QA/QC) sampling procedure, detailed 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 provided in the Quality Assurance Project Plan (QAPP) attached as Appendix C of the SVE Pilot Testing Work Plan.

## **2.1 SAMPLE COLLECTION**

## 2.1.1 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, 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 its own unique sample number; the description should include the unique sample number of its duplicate.



## 2.1.2 QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Several types of QA/QC samples will be collected, including field duplicates, trip blanks, equipment blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples. The following subsections present the sample collection procedures for each of these samples.

## 2.1.2.1 Field Duplicates

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 or soil vapor samples.

## 2.1.2.2 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.



## 2.1.2.3 Equipment Blank

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).

## 2.1.2.4 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 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.

## 2.1.3 Soil Sampling in Pilot Test Area

The field screening procedures, soil sampling procedures and decontamination procedures for the baseline, intermediate, and post-pilot test sampling are discussed in the following subsections. The XDD standard operating procedure (SOP) for Geoprobe soil sampling will be followed and is provided in Attachment A.

## 2.1.3.1 Field Screening Procedures

Field screening of soil samples will be performed using a Portable MiniRAE 2000 photoionization detector (PID). 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).

## 2.1.3.2 Soil Sampling Procedures

Soil sample collection will be performed from Geoprobe cores using a disposable Terra-core sampling device. Soil sampling locations are indicated in Figure D.1.

During baseline soil characterization, field screening techniques (i.e., PID) will be used to screen each six-inch interval and a representative grab sample for laboratory analysis will be collected using Terra-core sampling devices from the interval with the maximum PID reading in each of the three target soil intervals (i.e., the upper silty sands, intermediate siltyclay, and the lower silty sands). During the intermediate and post-pilot test soil characterization, PID readings from each six-inch interval will be recorded; however, grab samples for laboratory analysis will be collected from the same interval as the pre-treatment soil characterization samples. Although there is inherent variability in soil sampling, this sampling methodology will allow for more accurate baseline, intermediate and post-treatment soil data for comparison.

Soil samples will be submitted to the laboratory for VOC analysis. The ice will be contained within sealable plastic bags and the coolers will contain enough ice to maintain the contents at or less than 4 degrees Celsius during transport. Samples will be shipped to the laboratory at the end of each sampling event for VOC analysis with standard turnaround time.

# 2.1.3.3 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.

# 2.1.4 VAPOR SAMPLING

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

# 2.1.4.1 Field Screening Procedures

Field screening of vapor samples will be performed using a MiniRAE 2000 PID. 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 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.

# 2.1.4.2 Vapor Sampling Procedures

Representative vapor samples will be collected from sampling ports installed throughout the SVE system using an air sample pump as described in the field screening procedures. 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 with standard turnaround time.

## 2.1.4.3 Decontamination Procedures

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

## 2.2 SAMPLE IDENTIFICATION AND LABELING

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

#### WGK-BIGMO-ZZZ-YYY-BB-CC-A

Where: WGK is the site identification for the XDD project location

BIGMO is the treatment unit area (e.g. Big Mo)

ZZZ is the field sample identification (e.g. baseline, intermediate or post-pilot test soil)

YYY is the unique sampling location number (e.g. sample location)

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

A is the sample type (S = soil, V = vapor, A = air, 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

# 2.3 CHAIN-OF-CUSTODY RECORDS

Chain-of-custody records will be initiated by the samplers in the field. The field portion of the 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.

# 2.4 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.

## 3.0 ANALYTICAL PROCEDURES

## **3.1 FIELD SCREENING ANALYTICAL PROTOCOLS**

Field screening of soil samples will be performed using a Portable MiniRAE 2000 PID. After calibrating the PID, a moisture filter should be attached to the PID to avoid moisture buildup on the PID lamp. 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 ppmv.

Field screening of vapor samples will be performed using a MiniRAE 2000 PID. Flexible Tygon tubing will be used to connect the Tedlar bags to the MiniRAE 2000 PID for screening. 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.

## **3.2 LABORATORY PROCEDURES**

All laboratory analyses will follow approved analytical methods as well as approved preparatory procedure methods, as applicable. Details on the USEPA Method 8260B and TO-15 laboratory analyses are presented in Test America's SOPs in Attachment B. All analyses will be performed by Test America unless otherwise stated.



TABLES

# APPENDIX D SAMPLING AND ANALYSIS PLAN

SVE PILOT TESTING WORK PLAN

#### Table D.1 Sampling Plan Sampling and Analysis Plan W.G. Krummrich Facility, Sauget, IL August 2008

Event	Task	Description	Sampling Location	Measurement Location	Frequency	Samples per Event	Analytical Method
Baseline	e Sampling						
	Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once	26	8260B
	Soil Vapor Sampling	Soil vapor samples from SVE wells	SVE wells	Field measurement Laboratory analysis	once	26 3	PID TO-15
		Soil vapor samples from vapor probes/points	Vapor probes/points	Field measurement Laboratory analysis	once	25 3	PID TO-15
Design I	Parameter Evaluation Tes	ting					
	Extracted Soil Vapor Sampling	SVE stream from higher and lower permeability layers	Process equipment	Field measurement	daily	2	PID
	Air Treatment Sampling	Air permit compliance	post-treatment	Laboratory analysis	per Permit		TO-15
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	twice a week	1	PID
SVE Pe	erformance Evaluation Tes	ting					
	Soil Vapor Sampling	Soil vapor samples from SVE wells	SVE wells	Field measurement	once at starup once at mid-point	19	PID
				Laboratory analysis	once at end	3	TO-15
		Soil vapor samples from vapor probes/points	Vapor probes/points	Field measurement Laboratory analysis	once at end once at end	25 3	PID TO-15
	Extracted Soil Vapor	SVE streams from the three SVE mainlines (fill and upper silty sand layers, intermediate silty clay	Process equipment	Field measurement	daily - 1st week biweekly - 1st month monthly - afterwards	4	PID
	Sampling	layer and lower silty sand layer) + combined SVE stream prior to the air treatment unit		Laboratory analysis	3 times - 1st week biweekly - 1st month monthly - afterwards	4	TO-15
	Soil Sampling	Soil samples via Terrcore at Test mid-point	See Figure 9	Laboratory analysis	once	26	8260B
	Air Treatment Sampling	Air permit compliance	post-treatment	Laboratory analysis	per Permit		TO-15
		To confirm treatment effectiveness	pre- & post-treatment	Field measurement	monthly	1	PID
Post-Pil	lot Test Sampling						
	Soil Sampling	Soil samples via Terracore	See Figure 9	Laboratory analysis	once	26	8260B

#### Summary of Sampling for Laboratory Analysis

	Samples per Event	Frequency of Events	Total Number of Samples
Soil Sample Analysis via 8260B Method			
Baseline sampling	26	1	26
Sampling at mid-point of Effectiveness Testing	26	1	26
Post-pilot test sampling	26	1	26
Total Number of Soil Analyticals (excluding QA/QC samples)			78
Soil Vapor Sample Analysis via TO-15			
Baseline SVE well sampling	3	1	3
Baseline vapor probe/point sampling	3	1	3
SVE well sampling during Effectiveness Testing	3	3	9
Vapor probe/point sampling during Effectiveness Testing	3	1	3
Extracted soil vapor sampling during Effectiveness Testing	4	7	28
Total Number of Soil Vapor Analyticals (excluding QA/QC samples)			
Air Permit Compliance Monitoring			
Vapor discharge sampling per air permit requirements	1	per Permit	to be determined
Total Number of Vapor Discharge Analytica	ls (excluding QA/QC sample	s)	to be determined

Notes:

PID - photoionization detector

8260B - U.S. EPA Method 8260B for VOC analysis TO-15 - U.S. EPA Method TO-15 for VOC analysis Permit - Air permit SVE - soil vapor extraction

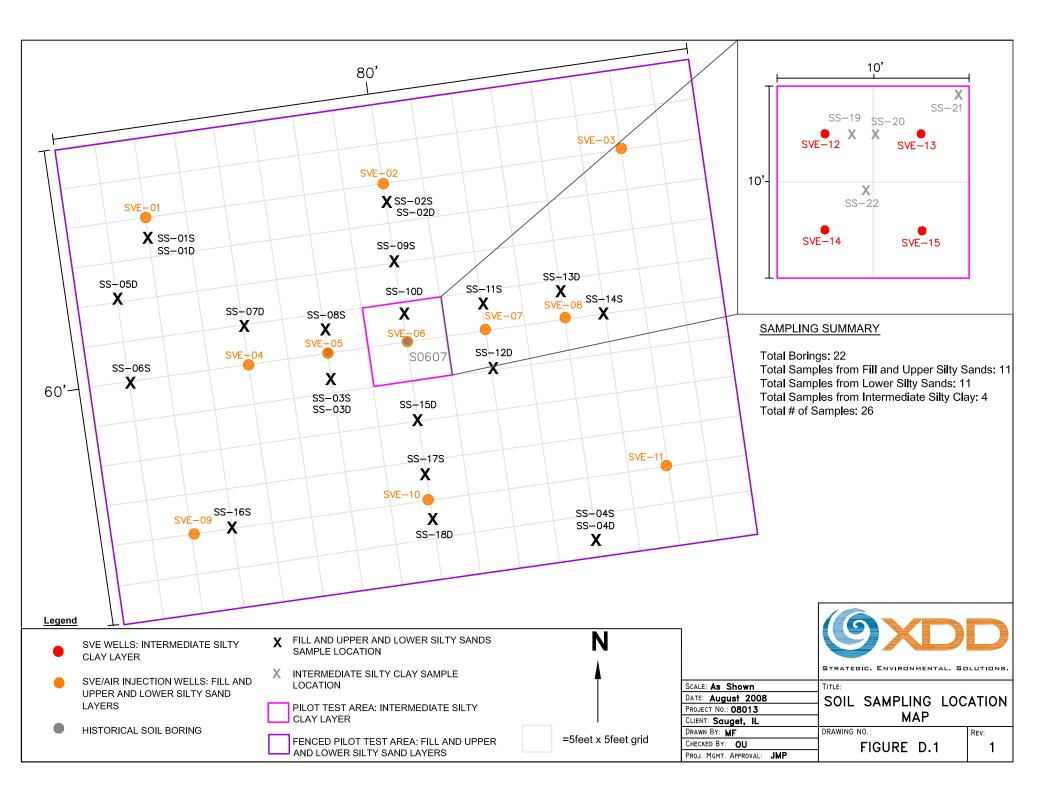




**FIGURES** 

# APPENDIX D SAMPLING AND ANALYSIS PLAN

SVE PILOT TESTING WORK PLAN





# ATTACHMENT A XDD SOPS FOR

# **GEOPROBE SOIL SAMPLING**

# VAPOR SAMPLING WITH TEDLAR BAGS

# APPENDIX D SAMPLING AND ANALYSIS PLAN

SVE PILOT TESTING WORK PLAN

### XDD, LLC (XDD)

#### STANDARD OPERATING PROCEDURE (SOP)

# STANDARD OPERATION PROCEDURE FOR SOIL SAMPLING VIA DIRECT PUSH (E.G., GEOPROBE<sup>®</sup>) 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.

#### 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.

#### 3.0 METHOD

A 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
- stainless steel soil mixing pans
- stainless steel soil sampling spatulas
- soil boring log
- Unified Soil Classification System (USCS) chart
- Munsell soil color chart
- measuring tape
- wide mouth sample jars
- ZipLock<sup>®</sup> 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 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.

One equipment blank shall be collected 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.

One field blank will also be collected from each water source (potable and distilled/deionized water) used for sampling equipment decontamination and analyzed 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 thermal oxidation system (Thermox) discharge. Vapor samples are collected in 1-liter Tedlar air sample bags for VOC, O<sub>2</sub>, CO<sub>2</sub>, 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  $\frac{1}{4}$  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  $\frac{3}{16}$ " 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.



# ATTACHMENT B TEST AMERICA SOPS FOR U.S. EPA METHOD 8260B AND TO-15

# APPENDIX D SAMPLING AND ANALYSIS PLAN

SVE PILOT TESTING WORK PLAN

APPENDIX D: SAMPLING AND ANALYSIS PLAN SVE Pilot Testing Work Plan, August 2008



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# **VOLATILE COMPOUNDS BY GC/MS**

# (Method: EPA 8260B)



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TestAmerica Savannah



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# SOP REVISION / REVIEW SUMMARY

SOP Number: <u>VM20:03.04.04:5</u> Review Date: <u>March 4, 2004</u> Effective Date: <u>April 4, 2004</u>

## **Review / Revision Type:**

Analytical/procedural revisions

- Revision # change. Completed SOP Training Forms are required.

## Summary of Changes:

- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information to be consistent with current STL format
- Added information regarding expiration dates for standards
- Added trap check standard information in Section 10
- Added method performance and waste management information
- Updated instrument conditions
- Removed requirement to analyze liquid field QC samples using heated purge and report as  $\mu$ g/kg. No longer analyzed as soils.
- Changed number of sample containers provided from 3 to 4
- Removed option of performing 25mL purge
- Removed option of adding preservative to samples upon receipt if pH<2
- Removed requirement to change carbon traps every 3 months
- Updated current standard concentrations and volumes
- Added requirement to run ICV per SOP AN67
- Added guidance for evaluating non-CCC compounds in CCV, (use <50% as guidance) difference

## SOP Number: <u>VM20:03.04.04:5 A</u> Review / Effective Date: <u>01.26.06</u>

# Review / Revision Type:

Minor text, grammatical, and/or formatting changes

- Alpha character revision, notification is required. SOP training is not required.

# Summary of Changes:

- Minor grammatical and editorial changes.
- Revised all referenced SOP titles to be consistent with current revisions.
- Clarification of 7 day holding time for unpreserved samples taken from EPA 624. (Section 5.1)
- Removed references to hard-copy records to be maintained for 4-BFB. This data is automatically stored electronically in the Target data processing system. References to recording data in an analysis log changed to recording in the data acquisition system.
- Added LCS to analytical sequence to correspond with current practice. (Appendix)
- Corrected LCS spiking list from subset spike to TCL list as is current practice. (Appendix)



THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Savannah

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- Added note to perform 2<sup>nd</sup> source ICV as stated in body of SOP. (SOP Summary Table)
- Corrected ISTD area requirement in CCV to be -50% to +100% as listed in body of SOP and is currently performed. This requirement was previously listed incorrectly as -50% to +200%. (SOP Summary Table)

Approval:

Quality Assurance Manager: andre Sal Date: January 26, 2006

Andrea Teal

SOP Number: <u>VM20:03.04.04:5 B</u> Review / Effective Date: <u>March 14, 2007</u>

# **Review / Revision Type:**

Minor text, grammatical, and/or formatting changes

- Alpha character revision, notification is not required. SOP training is not required.

# Summary of Changes:

- Minor grammatical and editorial changes.
- Revised all referenced SOP titles to be consistent with current revisions.

Approval:

**Quality Assurance Manager:** 

andrea.

Date: March 14, 2007

Andrea Teal



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#### 1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes the procedures used to determine the concentration of volatile organic compounds (VOC) in water, wastewater, soils/sediments, wastes, oils, sludges, and solids. Appendix A lists the routine target compounds, an example of the retention time order of each target compound, the quantitation and confirmation ions of the target compounds, and the internal standard assignments.
- 1.2 The complete list of target analytes, reporting limits (RL), method detection limits (MDL), and the accuracy and precision criteria for each target compound associated with this procedure are listed in the Method Limit Groups in STL LIMS.

#### 2.0 SUMMARY OF METHOD AND DEFINITIONS

- 2.1 Volatile organic compounds (VOC) are purged from the sample matrix with helium. The VOC are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOC are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOC, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion. Appendix B is an SOP Summary that highlights the major QC requirements of this procedure.
- 2.2 Aqueous samples may be purged at ambient conditions (recommended) or at 40°C (optional). Five milliliter aliquots of the sample may be purged. The calibration standards and the associated QC must be analyzed under the same conditions and volume.
- 2.3 Low-level (nominally<1mg/kg) soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards are purged under the same conditions.
- 2.4 High level soils (nominally>1mg/kg) and waste samples are extracted with methanol (1mL of methanol per gram of sample). An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.
- 2.5 Definitions Refer to SOP AN99: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions.
  - VOC volatile organic compound(s) VOA – volatile organic analytes (analysis)
- 2.6 This procedure is based on the guidance in SW-846 Methods 8260B, 5030, and 5035.

#### 3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual (CSM), the STL Savannah Addendum to the CSM, and this document.



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#### 3.1 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and disposed of in accordance with SOP CA70: *Waste Disposal*.

All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. The analyst must wear protective clothing (lab coat or apron), eye protection (glasses or face shield), and disposable gloves when handling these samples.

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

#### 3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. MSDS can be found in electronic format on Oasis on the EH&S webpage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure		
Methanol	Flammable	200 ppm-TWA	A slight irritant to the mucous membranes.		
	Poison		Toxic effects exerted upon nervous system,		
	Irritant		particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.		
1 – Always add acid to water to prevent violent reactions.					
2 – Exposure limit refers to the OSHA regulatory exposure limit.					



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#### 4.0 INTERFERENCES

- 4.1 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.
- 4.2 The volatiles lab must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator blanks should be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in SOP AN70: *Homogenization, Compositing, and Segregation of Samples.*
- 4.3 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-tetrachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the lab can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.
- 4.4 The analysis of highly contaminated samples (>1mg/L or >1mg/kg) can affect succeeding analyses. Carry-over can occur when low concentration samples are analyzed after high concentration samples. Trap replacement and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when carryover is suspected to demonstrate that the system is free from contamination.
- 4.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If contamination of the fittings is suspected, the fittings may be heated at 105°C for one hour or replaced.

#### 5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Liquid samples are collected with no headspace in 40-mL vials equipped with Teflon-lined caps. The samples are acidified at the time of collection with about 0.10mL of concentrated HCl per 40mL of sample. The acid prevents the biological degradation of the aromatic compounds and prevents the dehydrohalogenation of some of the chlorinated alkanes. The sample must be iced at the time of collection and refrigerated at 4°C (less than 6°C with no frozen samples) in the lab until analysis.

Check each sample vial at the time of receipt for the presence of "bubbles". If the bubbles are less than 3mm in diameter, the vial is acceptable. If the bubble is greater than 3mm, use another vial. Notify the department supervisor or project manager if there are no acceptable vials for analysis.

A "sacrificial" vial or the vial used for screening analysis is used to check the sample pH. If the sample pH is greater than 2 notify the department supervisor or project manager.

The holding time for samples preserved with HCl is 14 days for all target compounds. Although not specifically stated in the reference method, the laboratory has taken guidance from EPA Method 624 and adopted a 7day default holding time for unpreserved or improperly preserved samples

5.2 Soils: Soils are routinely collected in triplicate in Encore samplers. A "bulk" sample is also routinely collected in a 125-mL jar fitted with a Teflon-lined cap. The bulk sample can be used for the methanol extraction if the concentration of the sample collected in the Encore exceeds the working range of the

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analytical system.

Soils collected in Encore samplers must be analyzed within 48 hours of collection or must be transferred within 48 hours of collection to sealed vials containing sodium bisulfate preservation solution or methanol. If the sample contains high levels of carbonates, the sample is preserved with water and frozen until the time of analysis. The procedure for preparing soil samples is given in Section 9.2.

The holding time for the preserved sample is 14 days from the date of collection. The holding time for frozen samples is 14 days from the date of collection.

- 5.3 High level soil and waste samples are collected in glass containers (usually 125-mL clear glass) equipped with Teflon-lined caps. Soil samples may also be submitted as core samples contained in Encore samplers, metal or plastic "tubes", or in 40-mL VOA vials. The samples are iced at the time of collection and stored at 4°C (less than 6°C with no frozen samples). The holding time for soil and waste samples subjected to methanol extraction is14 days from date of collection; that is, the extraction and analysis must be completed within 14 days of collection.
- 5.4 TCLP leachate samples are collected with no headspace in Tedlar bags or syringes. The leachate samples are acidified at the time of collection (after the leaching procedure) with about 0.10mL of concentrated HCl per 40mL of sample and stored at 4°C (less than 6°C with no frozen samples) from the time leaching is completed until the analysis. The acidified leachate sample must be analyzed within 14 days of the leaching procedure. If the sample is not acidified, the leachate must be analyzed within 7 days of the leaching procedure.

**NOTE:** Samples that are suspected of having very high concentrations of VOC should be segregated from the "routine" samples and stored in a manner that will minimize sample and laboratory contamination. See SOP AN70: *Sample Composting, Homogenization, and Segregation of Low/High Concentration* for guidance. If possible, keep the field QC in the same storage refrigerator as the samples.

#### 6.0 APPARATUS AND MATERIALS

- 6.1 Mass spectrometer: equipped with a capillary direct interface and a split/splitless injector or molecular jet separator
- 6.2 Gas chromatograph, compatible with the MS and purge and trap systems. If the GC is equipped with an injector that is operated in the split mode, the exit vent must have a carbon trap in-line to collect the volatile compounds that are vented during the transfer from the purge and trap device.
- 6.3 Purge and trap device Tekmar 3000 Liquid Concentrator or equivalent
- 6.4 Supelco Vocarb 3000 trap or equivalent Other traps may be used as long as the target compounds can be detected at the required quantitation limit.
- 6.5 Archon soil analyzer for low level soils, compatible with Tekmar purge and trap instruments The instrument must be capable of automatically adding water and internal standard to the container while maintaining the septum seal, heating the sample to 40°C, and spinning the stir bar to mix the sample during the purging step.
- 6.6 Data System compatible with the analytical system
- 6.7 Microsyringes: 10μL, 25μL, 50μL, 100μL, 250μL, 500μL, 2.5mL



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- 6.8 Gastight syringe: 5mL
- 6.9 Volumetric flasks: 1.0mL, 10mL, 100mL
- 6.10 Recommended Column: J&W DB-624: 20m x 0.18mm ID, 1.8μm film

#### 7.0 REAGENTS

The preparation and tracking of reagents must be performed in accordance with SOP AN41: *Reagent and Standard Materials Traceability.* 

- 7.1 Reagent water free of volatile contaminants (obtained by purging with inert gas or carbon filtration)
- 7.2 Methanol purge and trap grade
- 7.3 Sodium bisulfate reagent grade. This salt is hydroscopic and should be stored in a dessicator.
- 7.4 Soil preservation solution Slowly add, while stirring, 200g of sodium bisulfate to a 1.0-L volumetric containing about 700mL of reagent water. After the salt has dissolved, dilute to volume with reagent water, transfer to a storage container, and purge the solution with nitrogen for several hours. Store the solution in an area free from VOC especially water-soluble solvents such as acetone. The reagent should be tested prior to use by the analysis of a blank containing 5mL of the solution. The reagent is acceptable if it meets the same criteria as a method blank.

#### 8.0 STANDARDS

Calibration and spike solutions are prepared from either certified stock solutions purchased from vendors or from stock standards prepared from neat materials. Certificates of analysis or purity must be received with all stock solutions or neat compounds. The preparation of the calibration standards must be tracked in accordance with SOP AN41: *Reagent and Standard Materials Traceability*.

#### 8.1 Expiration Dates

Maximum hold times (time from initial use or receipt to final use) for standard materials should be set according to the guidance in this SOP. The analyst is cautioned that these are maximum hold times and not to be considered an absolute guarantee of standard quality. The analyst must use sound judgment when deciding whether to use a standard. If the analyst is in doubt about the quality of a standard material, a new material must be obtained or the standard material verified. Do not compromise data quality to extend a standard's life - when in doubt, throw it out.

The expiration date of any standard may not exceed the expiration date of the material or stock that was used to prepare the standard; that is, the "children may not outlive the parents".

#### **Neat Materials**

Neat materials with no manufacturer's expiration date should be considered acceptable for use as long as they are stored in their original, unopened container. Once opened, standard materials should be verified against an independent source annually. The acceptance criteria for the verification are to meet the most stringent calibration criteria of the procedure(s) being employed.

Flame Sealed Ampoules



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Flame sealed ampoules with no manufacturer's expiration date should be considered acceptable for use for 1 year from receipt.

Non-Flame Sealed Containers – Stock Standards

VM 1 month from opening/prep VM Gases 1 week to one month from opening/prep (see NOTE 1)

Non-Flame Sealed Containers – Calibration/Working Standards

VM	1 month from prep
VM Gases	1 week to one month (see NOTE 2)

Internal standard, Spike, and Surrogate Solutions:SVOC3 months from preparationVOC/A1 month from preparation

Secondary non-routine standards 3 months from preparation

\*NOTE 1: The expiration data for volatile gas standards may be extended beyond the recommended one week period if the lab can demonstrate that the standards are stable for a longer time.

\*NOTE 2: It is recommended that a sufficient volume of this VOC standard be prepared when the ampoule is opened to cover a period of one week. For example, when a 2000µg/mL standard containing the gases is opened, twenty-five milliliters of the  $25\mu$ g/mL solution are prepared and aliquoted into 1-mL vials. This would give the lab enough working gas standard to prepare the initial calibration and two CCV each day for one week.

8.2 Preparation of Stock Standards from Neat Compounds

The lab should attempt to obtain a certified primary standard or secondary standard before preparing stock standards from neat materials. If primary stock standards must be prepared in-house, the target concentration range is from  $2000\mu$ g/mL to  $10000\mu$ g/mL. SOP AN41: *Reagent and Standard Materials Traceability* gives the general instructions for the preparation of the stock solutions from neat materials.

8.3 Preparation of the Working Standard from Stock Standards

The working standard is prepared from the primary stock standards that are either prepared from neat compounds or purchased as certified solutions. The working standard contains one or more of the target compounds at a concentration suitable for preparing the calibration standards, generally 10- $200\mu$ g/mL. A known volume of the working standard is then added to a known volume of reagent water to make the calibration standard.

The laboratory must document the "recipes" used to prepare the standards in the standard traceability log, as a controlled posting, or other appropriate log.

8.4 Preparation of the Calibration Standards from the Working Standards

The calibration standards are the standards that are analyzed on the instrument. The calibration standard is made by adding a known volume of the working standard to a known volume of reagent water. The instrument must be calibrated using a minimum of five calibration standards. The lowest level standard must be at the reporting limit and the rest of the standards will define the working range



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of the analytical system. The routine calibration concentrations are 1, 5, 10, 25, 50, 100, and  $200\mu g/L$ . Ketones are calibrated at 2, 10, 20, 50, 100, 200, and  $400\mu g/L$ . Internal standards are maintained at  $50\mu g/L$  in each calibration standard.

- 8.4.1 Add 5.0mL of reagent water to a 5mL-glass syringe.
- 8.4.2 Add a known volume of the working standard to 5.0mL of reagent water.

NOTE: The calibration standards for the low level soils are prepared using the same procedures as for the 5mL water purge except that the standards are purged at 40°C. The lab has the option of using blank sand in the calibration standards.

NOTE: The standard concentrations given in this SOP are provided for guidance. The laboratory must document the standard preparation procedures and concentrations in the standard traceability log, as a controlled posting, or other appropriate log.

#### 9.0 SAMPLE PREPARATION

Composite samples can be prepared using the guidance provided in SOP AN70: *Homogenization, Compositing, and Segregation of Samples.* 

- 9.1 Aqueous samples are analyzed directly by purge and trap/GC-MS. No sample preparation is necessary except to homogenize the sample prior to subsampling. The pH of liquid samples is checked and recorded upon receipt into the department to determine if the sample has been properly preserved.
- 9.2 Preparation of Soil Samples (5035)

The preparation of soil samples must be performed upon arrival in the laboratory and within 48 hours of collection. Three Encore devices and one bulk container are routinely received for each sample. Two of the Encores are prepared for low level analysis and one is extracted in methanol to be used if the low-level samples exceed the calibration range. The bulk container is used for determining the type of preservation for the low-level samples and for screening.

- 9.2.1 Test an aliquot of the bulk sample for the presence of carbonates. At the same time, transfer a 5-g aliquot of the sample to a vial for screening by headspace analysis.
  - Transfer approximately 5g of sample from the bulk sample to a 40mL vial.
  - Add approximately 5mL of the sodium bisulfate solution and shake the vial.
  - If the sample exhibits effervescence, the Encore samples are preserved by adding 5mL of volatile-free water and placing in a freezer at -10°C. If no effervescence is noted, the Encore samples are preserved with 5mL soil preservation solution.
- 9.2.2 Add a stir bar to a 40-mL vial. Weigh the vial, and record its tare weight (or tare the vial and stir bar weight by pressing the autotare button).
- 9.2.3 Transfer the sample from the Encore sampler to the tared vial and record the weight of the sample log.

If the sample effervesced during the carbonate test (Section 9.2.1), add 5.0mL of reagent water and freeze at -10°C. The holding time is 14 days from collection. If the sample did not effervesce, add 5.0mL of the soil preservation solution, seal the vial, and store the sample at 4°C until the time of analysis. The preserved sample must be analyzed within 14 days of collection.



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9.3 A methanol extraction is prepared from the third Encore device or from the bulk container when the third Encore is unavailable. Carry out the preparation quickly to minimize the loss of volatiles.

- Mix the sample with a stainless steel spatula and transfer the sample to 20-mL glass vial.

- Add  $8\mu$ L of the surrogate spiking solution (2500 $\mu$ g/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 10g and 100% percent solids, is calculated:

 $Ct(\mu g / kg, dw) = \frac{0.008mL \otimes 2500ug / mL}{0.010g \otimes solids} = 2000\mu g / kg, dw$ 

- Shake the sample for approximately two minutes. Allow the solvent to separate from the solids portion of the sample and transfer a 1-2mL aliquot of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4°C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

- For each batch of twenty or fewer extracted samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding  $40\mu$ L of the surrogate spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze  $25\mu$ L of the extract.

The lab control standard is prepared by adding  $40\mu$ L of the surrogate spiking solution and  $40\mu$ L of the matrix spiking solution to 10mL of purge and trap grade methanol. Assume a sample weight of 10g. Analyze  $25\mu$ L of the extract.

The matrix spikes are prepared by adding  $40\mu$ L of the surrogate spiking solution ( $2500\mu$ g/mL) and  $40\mu$ L of the matrix spiking solution ( $2500\mu$ g/mL) to 10-g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for approximately two minutes. Analyze  $25\mu$ L of the extract or a smaller volume if the VOC concentration is high.

- Add  $25\mu$ L of the extract (or a smaller volume if the VOC concentration exceeds the linear range of the system with  $25\mu$ L) to 5.0mL of water. Add the internal standard solution and analyze the sample using the ambient water calibration.

9.4 Methanol Extraction for Wastes

Carry out the preparation quickly to minimize the loss of volatiles.

- 9.4.1 Mix the sample with a stainless steel spatula and transfer 1g ( $\pm$  0.2g) to a glass vial.
- 9.4.2 Add 10µL of the surrogate spiking solution (2500µg/mL) to the sample and quickly add 10mL of purge and trap grade methanol. The theoretical concentration of the surrogates in the sample, assuming a sample weight of 1.0g, is calculated:

$$Ct(\mu g / kg) = \frac{0.010mL \otimes 2500\mu g / mL}{0.0010g \otimes solids} = 25000\mu g / kg$$

9.4.3 Shake the sample for approximately one minute. Allow the solvent to separate from the



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solids portion of the sample and transfer 1mL to 2mL of the extract to a storage vial. The vial should be sealed with no headspace. Store the methanol extract at 4°C until the time of analysis. The extract must be analyzed within 14 days of sample collection.

For each batch of twenty or fewer extracted samples, prepare a method blank and a lab control standard. Prepare a matrix spike and matrix spike duplicate at a frequency of 5% of all samples.

The method blank is prepared by adding  $8\mu$ L of the surrogate spiking solution (2500 $\mu$ g/mL) to 10mL of purge and trap grade methanol. Assume a sample weight of 1.0g. Analyze 100 $\mu$ L of the extract.

The lab control standard is prepared by adding  $10\mu$ L of the surrogate spiking solution (2500 $\mu$ g/mL) and  $10\mu$ L of the matrix spiking solution (2500 $\mu$ g/mL) to 5.0mL of purge and trap grade methanol. Assume a sample weight of 5.0g. Analyze 100 $\mu$ L of the extract.

The matrix spikes are prepared by adding  $10\mu$ L of the surrogate spiking solution ( $2500\mu$ g/mL) and  $10\mu$ L of the matrix spiking solution ( $2500\mu$ g/mL) to 1g aliquots of the sample selected for the MS/MSD. Quickly add 10mL of purge and trap grade methanol to each sample and shake for approximately one minute.

Add  $100\mu$ L of the extract (or a smaller volume) to 5.0mL of water. Add the internal standard solution and analyze the sample using the ambient water calibration.

NOTE: Waste samples may require significant dilution prior to analysis.

#### 10.0 ANALYTICAL PROCEDURES

The following instrument conditions are recommended. The lab must document the instrument conditions in the maintenance log, the data system, or on the analysis log.

#### 10.1 Instrument Conditions

#### 10.1.1 GC/MS Conditions

GC/MS conditions may vary according to the environment and condition of each instrument. The lab must document the instrument conditions to assure consistent results and to aid in trouble-shooting the analytical system. The laboratory is responsible for assuring that the conditions necessary to achieve adequate separation and sensitivity of the target analytes are maintained.

Column: 20m x 0.18mm ID x 1.0μm Column: Flow: 0.5-1.0ml/min Injector: Split/Splitless operated in the split mode with 1mm ID quartz insert Split ratio (desorb to column flow): 40:1 or 80:1 Mass spectrometer interface: 240°C (direct column interface) Mass spectrometer source temperature: 250°C Mass scan range: 35-300amu, with a minimum scan cycle of 1 scan per second

Temperature Program:

Initial column temperature: 50°C Column temperature program: 18°C per minute



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Final column temperature: 200°C

10.1.2 Purge and Trap Conditions

The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

VOCARB 3000 trap Purge Time: 11 minutes Purge temperature: aqueous-ambient; soils-heated 40°C Desorb time: 0.75 minutes Desorb temperature: 250°C Bake time: 8 minutes at 260°C Purge flow: Approximately 20-30mL/minute Valve temperature: 150°C Transfer line: 150°C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets - ketones, ethers, bromoform - may be low and the reporting limit may not be achieved on a routine basis.

10.1.3 Purge and Trap Device Cleanup

Prior to analysis of calibration standards and samples, the purge and trap system should be purged with helium with blank tubes attached to the port. The recommended program for a "purging cycle" is:

Purge Time: 2 minutes Purge flow: 25-40m/min (same flow as is routinely used) Desorb for one minute at routine temperature Set Purge Ready temperature to 100-150°C to cycle P/T unit more quickly

A purging cycle is also recommended if high level samples are analyzed and carry over hits ("J" values) are detected in the same port or in subsequent sample ports.

#### 10.2 BFB Tune Check

- 10.2.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each 12-hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical the mass range, scan rate, and multiplier voltage. If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.
- 10.2.2 Evaluation of the 4-BFB peak

10.2.2.1The chromatogram should exhibit acceptable baseline behavior and the 4-BFB peak

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should be symmetrical. A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

- 10.2.2.2 The spectrum of the 4-BFB must meet the criteria listed in the attached SOP Summary. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans ±1 scan from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.
- 10.2.2.3 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, or other anomalies.
- 10.2.2.4 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

#### 10.3 Trap Check Standard

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds.

- 10.3.1 Prepare the trap check standard by injecting 2µL of a 50µg/mL bromoform standard into 5mL of reagent water. Other sample volumes may be used but the sample must transfer 100ng of bromoform to the column.
- 10.3.2 Add the internal standards and surrogates.
- 10.3.3 Analyze the sample using the same analytical system conditions used for samples and standards.
- 10.3.4 Evaluate the chromatogram for the presence of chloromethane and bromomethane. Compare the response to  $1.0\mu g/L$  standard. The response must be less than or equal to one half of the response of the  $1.0\mu g/L$  standard and the trap check standard must quantify less than  $0.5\mu g/L$  compared to the initial calibration curve.

NOTE: Make sure that the spectra match the reference spectra and that the most abundant ions are present for both compounds - chloromethane (m/z 50, 52) and bromomethane (94, 96)

10.3.5 If the trap check standard does not meet the acceptance criteria, the trap must be replaced, conditioned, and system re-calibrated prior to the analysis of samples.

#### 10.4.1 Initial Calibration

Initial calibration is performed in accordance with SOP AN67: *Evaluation of Calibration Curves*. After the 4-BFB criteria have been met, the initial calibration standards are analyzed. Prepare the initial



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calibration standards according to the example recipes in the SOP appendices or lab-specific recipe. The lab must document the "recipe" used to prepare the calibration standards. The lowest level calibration standard must be at or below the routine RL and the other calibration standards will define the working range of the system.

- 10.4.1 Remove the plunger from the syringe and fill the barrel to overflowing with reagent water.
  - 10.4.2 Replace the plunger, and force any airspace out of the syringe. Adjust the volume to the syringe volume (5mL).
  - 10.4.3 Inject the standards and internal standards into the syringe.

NOTE: Use the internal standard (IST) mix when preparing the calibration standards for analysis. The surrogates are already included in the standard mixes.

10.4.4 Load the standards onto the purge and trap device and begin the analysis. All pertinent standard traceability information must be recorded within the analytical data system. The standards must be clearly identified and traceable to the preparation steps.

NOTE: The standards for low-level soil samples are prepared in the same manner as the 5mL standards. The standards for the low-level soils are purged at 40°C. The lab has the option of using blank sand or soil in the calibration standards and the blank in the low level soil analysis.

- 10.4.5 After the acquisition has taken place, evaluate the calibration standards to ensure each target compound, surrogate, and internal standard has been correctly identified. The analyst must be careful to complete this step before proceeding.
- 10.4.6 After each target compound, surrogate, and internal standard has been correctly identified, the relative response factor for each target compound and surrogate is calculated using the data system as follows:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where

Ax = area of the characteristic ion for the compound being measured

Ais = area of the characteristic ion for the internal standard associated with the compound being measured (see the attached quantitation report for a list of the compounds that are associated with the various internal standards)

Cx = concentration or mass on-column of the target compound being measured ( $\mu$ g/L or  $\mu$ g/kg OR ng or  $\mu$ g on-column)

Cis = concentration or mass on-column of the internal standard ( $\mu$ g/L or  $\mu$ g/kg OR ng or  $\mu$ g on-column)

The average relative response factor (RRFavg) is calculated for each target compound and each surrogate compound as follows:

$$RRFavg = \frac{RRF1 + RRF2 + \dots + RRFn}{n}$$

where n = number of calibration levels



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Calculate the standard deviation (SD) for the target compounds and surrogates at all calibration levels as follows:

$$SD = \sqrt{\frac{\frac{n}{\sum}}{\frac{i=1}{n-1}}(RFi - RFavg)^2}$$

where

Rfi = response factor of a target compound in the individual calibration level Rfavg = average response factor n= number of calibration levels

10.4.7 Calculate the relative standard deviation (% RSD) of the calibration levels for each target as follows:

$$\% RSD = \frac{standard \ deviation}{RRFavg} \otimes 100$$

10.4.8 The results of the initial calibration are evaluated against the Calibration Check Compound (CCC) criteria and the System Performance Check Compound (SPCC) criteria, which are listed below. The CCC and SPCC criteria must be met before samples can be analyzed.

#### **Calibration Check Compounds – CCC**

Vinyl chloride; 1,1-dichloroethene; chloroform; 1,2-dichloropropane; toluene; ethylbenzene

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds - SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30

NOTE: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

10.4.9 After the initial calibration criteria (CCC and SPCC) have been met, each target is evaluated for linearity.



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If the %RSD of the target compound is less than or equal to 15%, the average response factor can be used for quantitation of samples. If the %RSD of the target compound is greater than 15%, a regression curve (linear, quadratic, etc) must be used for the quantitation of samples. A regression curve may also be used for the compounds that have %RSD less than 15%. The results can be used to plot a calibration curve of response ratios - Ax/Ais is plotted on the y-axis; Cx/Cis is plotted on the x-axis where:

Ax = area of the characteristic ion for the compound being measured

Ais = area of the characteristic ion for the internal standard associated with the compound being measured (See attached quantitation report for a list of the compounds that are associated with the correct internal standard)

Cx = concentration or mass on-column of the target compound being measured ( $\mu g/L$  or  $\mu g/kg$  OR ng or  $\mu g$ )

Cis = concentration of the internal standard ( $\mu$ g/L or  $\mu$ g/kg OR ng or  $\mu$ g)

If the regression coefficient of the regression curve is greater than 0.99, the curve can be used to quantify samples. Regression curves may be forced through zero but it is recommended that the curve be evaluated without forcing through zero first and then with the curve forced through the origin. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range.

When more calibration levels are analyzed than required, individual compounds may be eliminated from the lowest or highest calibration levels(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual analytes in the middle of the calibration curve be eliminated without eliminating the entire level. The lowest calibration point must be at or below the project RL.

NOTE: Linear regression curves must be used for South Carolina DHEC compliance samples. See pre-project plans and client QAPPs for other exceptions to using non-linear curve fitting.

Method *8000B* allows for the use of the evaluation of the "grand mean". That is, if the average %RSD of ALL (all targets including CCC and SPCC) compounds in the initial calibration is less than 15%, the average response factor can be used for quantitation of all target compounds. The recommended course is to use regression curves, as described above, to quantify targets where the %RSD criterion (<=15%) is exceeded.

Each calibration curve must be verified with the analysis of a second source initial calibration verification standard (ICV). Refer to SOP AN67: *Evaluation of Calibration Curves* for guidance on the analysis and evaluation of ICV.

10.4.10 After the initial calibration criteria have been met, the method blank is analyzed. 5.0mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits (RL) in the MLG or other specified QAP.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet these criteria.

If the concentration of any target compound is above the RL in the MLG, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of samples can take place.



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If the method blank repeatedly fails to meet the criteria, contact the immediate supervisor to determine the cause of the problem and to determine a course of action. This action may include re-cleaning the sparging tubes (with soap, hot water, and methanol), purging the effected autosampler ports with heated methanol, flushing the purge and trap ALS concentrator with methanol, replacing the trap, changing the transfer line, and changing the column. A method blank is then analyzed after taking the corrective action to demonstrate that the contamination has been eliminated. Once the system is determined to be free from contamination, sample analysis may begin. Method blanks may be required after the analysis of samples that contain very high levels of VOC.

#### 10.5 Continuing Calibration Verification

At the beginning of each 12-hour clock, the tune of the instrument must be checked by the analysis of 50ng of 4-BFB. These criteria must be met before the analysis of the calibration check standards can take place.

- 10.5.1 After the tune criteria have been met, a continuing calibration check standard is analyzed. The continuing calibration standard should be at a nominal concentration of 20μg/L for liquid and 20μg/L for soils with ketones and poor purgeables at higher concentrations.
- 10.5.2 The CCC and SPCC criteria (Section 10.3.8) must be met in the calibration check standard before the analysis of the method blank and samples can take place. The percent difference (%D) is calculated as follows:

$$\%D = \frac{RRFavg - RRFccv}{RRFavg} \otimes 100$$

where

RRFavg = average response factor from initial calibration RRFccv = response factor from the check (12-hour) standard-calibration verification

The percent drift (%Drift) may also be used to evaluate the change/deviation of the curve:

$$\%Drift = \frac{Ci - Cccv}{Ci} \otimes 100$$

where

Ci = Calibration Check Compound standard concentration Cccv = measured concentration using the selected quantitation method

NOTE: The SPCC criteria (Section 10.3.8) must be met even if the regression curve option is used for quantitation. If these criteria are not met, corrective action must be taken. The corrective action may include reanalysis of the calibration check standard or preparation of a new secondary stock standard and reanalysis of the calibration check standard. If subsequent analysis of the standard is still out of criteria, a new initial calibration curve must be analyzed and evaluated.

Although the method only specifies %D criteria for the CCC compounds, all compounds in the continuing calibration verification must be evaluated. The laboratory has adopted a general rule of thumb to hold the non-CCC compounds to 50%D. If these criteria are not met, corrective action should be taken. The department manager or technical manager can provide guidance if these criteria are not met.



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10.5.3 The calibration standard (CCV) must also be evaluated for internal standard retention time and response.

If the retention time of any internal standard changes by more than 30 seconds from the retention times of the internal standards in the initial calibration, the analytical system must be inspected for problems and corrective action instituted.

If the extracted ion current profile (EICP) area for any of the internal standards changes by more than a factor of two (-50% to +100%) from the last calibration check standard, the analytical system must be inspected for problems and corrective action instituted. If the CCV is the first one after the initial calibration, compare the ISTD response to the corresponding level in the ICAL.

10.5.4 After the continuing calibration criteria have been met, the method blank is analyzed. 5.0mL of reagent water is spiked with the internal standard/surrogate and analyzed. The concentrations of the target compounds in the method blank are calculated and the results are compared to the reporting limits in the MLG.

If the concentrations of all target compounds are below the RL, analysis of client samples can take place. Note that all target compounds must meet the criteria.

If the concentration of any target compound is above the RL in the MLG, the method blank must be reanalyzed. The analytical system must be demonstrated to be free from contamination before the analysis of client samples can take place.

10.6 Aqueous Sample Analysis

Samples are analyzed only after the tune criteria, the calibration (initial or continuing) criteria have been met, and the method blank criteria have been met. See the SOP Summary for the analytical sequence.

- 10.6.1 Remove the samples to be analyzed from the refrigerator and allow the samples to come to ambient temperature.
- 10.6.2 Put on a pair of gloves before transferring the sample from the vial to the syringe. The sample is most likely preserved with acid or may contain toxic or hazardous chemicals or biologically active components that may cause skin irritations. *Gloves must be worn when handling samples.*
- 10.6.3 Mix the contents of the vial by inverting the vial several times. Check to see if there are air bubbles present in the sample. If air bubbles are present, use another vial if available. Make a note on the analysis log if the sample used contained bubbles and notify the supervisor and/or the project manager.
- 10.6.4 Remove the plunger from the glass syringe.
- 10.6.5 Open the vial of the well-mixed sample and gently pour the sample into the syringe barrel. The sample should fill the barrel of the syringe and overflow to allow trapped air bubbles to escape.
- 10.6.6 Replace the plunger into the syringe barrel. Try not to let air bubbles get into the barrel. If air bubbles are present, turn the syringe up, open the syringe valve, and expel the air while adjusting the volume to 5.0mL. If no air bubbles were trapped, adjust the syringe to volume.



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- 10.6.7 Inject the internal standard/surrogate (ISSU) mix into the sample.
- 10.6.8 Transfer the sample from the syringe to the purge and trap device. Record all of the sample identification information data acquisition system.
- 10.6.9 Analyze the samples using the purge and trap and GC/MS conditions used for the initial and continuing calibration standards.
- 10.6.10 Determine the concentration of the samples and QC items. If the concentration of a sample is above the highest calibration standard, the sample must be diluted and reanalyzed.

After the sample analysis sequence is completed, evaluate the results to determine if targets can be attributed to preceding samples or to samples analyzed in the same port in a previous sequence. This may be especially critical if samples are being evaluated to the method detection limit.

NOTE: Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags should be used or qualification in a case narrative provided to the client. For TCLP analyses, every reasonable effort should be made to achieve the regulatory level without instrument overload.

For clients who require lower detection limits, a general guide is to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if a sample analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower RLs.

A dilution is made when a volume of the sample is mixed with the reagent water to a final volume of 5.0mL. The dilution factor is calculated by dividing the volume of sample into the volume used for the calibration curve, as follows.

$$DF = \frac{\text{final volume of dilution}(mL)}{\text{volume of sample used}(mL)}$$

Example: if 1.0mL of sample is diluted to final volume of 5.0mL, the dilution factor is 5. (5.0/1.0 = 5).

Volume of Sample (mL)	Volume of Reagent Water (mL)	Final Volume (mL)	Dilution Factor
5.0	0	5.0	1
2.5	2.5	5.0	2
1.0	4.0	5.0	5
0.5	4.5	5.0	10
0.10	4.9	5.0	50

The following table gives some dilution factors:

NOTE: The same volume of internal standard/surrogate mix (ISSU) is added to the dilution as was added to the undiluted sample.

10.7 Low Level Soil Samples by Heated Purge and Trap (Method 5035)

The soil analytical system is calibrated using the same concentrations as the 5-mL purge. The tune,



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initial and continuing calibration criteria, and the method blank criteria must be met before samples are analyzed. Standards and QC items must be analyzed under the same heated purge and trap conditions.

Remove the samples to be analyzed (Section 9.2) from the refrigerator or freezer and allow the samples to come to ambient temperature. Inspect the vial for cracks or obvious breaches in the septum. Load the samples on to the soil-purging unit and analyze according to the sequence described in Appendix B.

10.8 Analysis of Methanol Extracts of Soils and Wastes

The methanol extraction is used when the concentration of one or more target compounds exceeds the linear range of the low-level purge technique (>1000 $\mu$ g/kg) or if the concentration of VOC in the soil or waste samples is high. Samples are analyzed only after the 4-BFB criteria, the calibration criteria (initial and continuing), and the method blank criteria have been met. Medium level soil extracts are quantitated using the ambient purge calibration curve. Sample preparation steps are included in Section 9.

- 10.8.1 Remove the plunger from the 5.0-mL syringe and fill the barrel to overflowing with reagent water. Replace the plunger, and force any airspace out of the syringe. Adjust the volume to the syringe volume (5mL).
- 10.8.2 Inject the sample extract and 5μL of the internal standard (IST) solution into the syringe. Use 25μL of the extract for soils and 20μL of the extract for wastes. Smaller aliquots are used if the concentration of target analytes exceeds the working range of the system.

NOTE: Use the internal standard (IST) mix when preparing the medium level samples. Recall that the surrogates have already been added to the sample during the methanol extraction step (See Section 9).

- 10.8.3 Load the sample onto the purge and trap device and begin the analysis. All pertinent information concerning the samples must be recorded in the data acquisition software. The samples must be clearly identified and traceable to the extraction log. These conditions must be the same as were used for the initial and continuing calibration standards ambient purge for aqueous samples.
- 10.8.4 Determine the concentration of the samples and QC items using the procedures in Section 11. If the concentration of a sample is above the highest calibration standard, a smaller aliquot of the methanol extract is reanalyzed to bring the highest target within the upper half of the calibration curve. Follow the guidelines in Section 10.4.10 for reporting dilutions.

NOTE: It is possible to dilute the surrogates in the sample extract below the linear range of the calibration curve. The minimum extract aliquot that can be used to provide a quantifiable result for the surrogates and matrix spikes is  $0.0025mL (2.5\mu L)$ .

SOIL: 10g to 10mL MeOH	WASTES: 1g to 10mL MeOH	Surrogates - Theoretical ng on-column
125uL (25mL)	100uL (20mL)	250
62.5uL (12.5mL)	50uL (10mL)	125
25uL (5.0mL)	25uL (5.0mL)	50
12.5uL (2.5mL)	10uL (2.0mL)	25
<2.5uL	<2.0uL	<5.0ng - below the quantitation limit-diluted out

NOTE: Some instrument quantitation limits may be higher than the limit listed in the table. The volume of extract should be adjusted accordingly.



## 11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Analysis of Target Compounds

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

- 11.1.1 Two criteria must be met in order to identify a target compound:
  - 1) Elution of the sample component within ±0.06 RRT (relative retention time) units of the daily standard containing that compound.

 $RRT = \frac{retention time of the target compound}{retention time of the associated internal standard}$ 

2) Correspondence of the target compound spectrum and the standard component mass spectrum

- 11.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. The ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.
- 11.1.3 The relative intensities of the ions present in the sample component spectrum should agree within  $\pm$  30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.
- 11.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.
- 11.2 Tentatively Identified Compounds

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification.

- 11.2.1 Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum.
- 11.2.2 The relative intensities of the major ions should agree within  $\pm 30\%$ .
- 11.2.3 Molecular ions present in the spectrum should be present in the sample spectrum.
- 11.2.4 lons present in the sample spectrum but not in the reference spectrum should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.



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- 11.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of coeluting peaks.
- 11.2.6 If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria are not met exactly, the peak may be considered tentatively identified. The analyst should consult the department supervisor or technical manager if there are any questions concerning an interpretation of spectra.
- 11.2.7 The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area of the nearest internal standard that has no interferences. The calculation is as follows:

Aqueous

$$TIC(ug/L) = \frac{Cis}{AREAis} \otimes AREAtic \otimes DF$$

where

Cis = concentration of the internal standard,  $\mu$ g/L AREAis = total ion peak area of the internal standard AREAtic= total ion peak area of the TIC DF = dilution factor

Soils by Heated P/T

$$TIC ( \mu g/kg, dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{5.0g}{(W)(solids)}$$

where

Cis = concentration of the internal standard,  $\mu g/kg$ AREAis = total ion peak area of the internal standard AREAtic= total ion peak area of the TIC W = weight of sample analyzed, g solids = decimal equivalent of percent solids

Soils by Methanol Extraction

$$TIC(ug/kg,dw) = \frac{Cis}{AREAis} \otimes AREAtic \otimes \frac{Vcal}{(W)(solids)}$$

where

Cis = concentration of the internal standard,  $\mu g/kg$ AREAis = total ion peak area of the internal standard AREAtic= total ion peak area of the TIC Vcal = volume that calibration curve is based on (5mL) solids = decimal equivalent of the percent solids(percent solids/100) W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

where

Wext = weight of sample extracted (g)



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Vf = final volume of the extract (mL) Vext = volume of extract added to the water (mL)

11.3 Calculations for Samples - Internal Standard Technique

Aqueous Samples - relative response factor:

$$concentration(ug/L) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes DF$$

where

Ax = area of the characteristic ion of the compound being measured Ais = area of the characteristic ion of the internal standard Cis = concentration of the internal standard ( $\mu$ g/L) RRFavg = average response factor of the compound being measured DF = dilution factor

Aqueous Samples - regression curve

concentration( $\mu g/L$ ) = concentration(curve)  $\otimes DF$ 

where DF = dilution factor

The reporting limit (RL) is calculated:

$$RL(\mu g/L) = RL_{MLG} \otimes DF$$

where

DF = dilution factor. The MLG RL (RL MLG) assumes a DF of 1.

Soils by Heated P/T- relative response factor:

concentration(
$$\mu g/kg, dw$$
) =  $\frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{5.0g}{(W)(solids)}$ 

where

Ax = area of the characteristic ion of the compound being measured Ais = area of the characteristic ion of the internal standard Cis = concentration of the internal standard ( $\mu$ g/kg) RRFavg = average response factor of the compound being measured W = weight of sample added to the sparging vessel (g) solids = (percent solids)/100)

Soils by Heated P/T - regression curve

$$conc(\mu g/kg, dw) = Ccurve(\mu g/kg) \otimes \frac{5.0g}{(W)(solids)}$$

where

Ccurve = concentration from curve ( $\mu$ g/kg) W = weight of sample added to the sparging vessel (g) solids = (percent solids)/100)

The reporting limit (RL) is calculated:



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$$RL = RL_{MLG} \otimes \frac{5.0g}{(W)(solids)}$$

where

W = weight of sample added to the sparging vessel (g) solids = (percent solids)/100)

The MLG assumes W= 5.0g and solids = 1.

Methanol Extraction Soils and Wastes - relative response factor

 $concentration(\ \mu g/kg, dw) = \frac{Ax}{Ais} \otimes \frac{Cis}{RRFavg} \otimes \frac{Vcal}{(W)(solids)}$ 

where

Ax = area of the characteristic ion of the compound being measured Ais = area of the characteristic ion of the internal standard Cis = concentration of the internal standard ( $\mu$ g/L) RRFavg = average response factor of the compound being measured Vcal = volume that calibration curve is based on (5mL) solids = (percent solids)/100) W = weight of sample added to the reagent water (g)

This weight is determined using the following equation:

$$W = \frac{Wext(g)}{Vf(mL)} \otimes Vext(mL)$$

Wext = weight of sample extracted (g) Vf = final volume of the extract (mL) Vext = volume of extract added to the water (mL)

Methanol Extraction of Soils and Solids- regression curve:

$$conc(\mu g, kg, dw) = Ccurve(\mu g/L) \otimes \frac{Vcal}{(W)(solids)}$$

where

Vcal = volume that calibration curve is based on (0.005L)W = weight of sample added to the reagent water (g) - defined above

The reporting limit (RL) is calculated:

$$RL = RL_{MLG} \otimes \frac{5.0g}{(W)(solids)}$$

where

W = weight of sample added to the reagent water (g) solids = (percent solids)/100)

The MLG assumes W= 5.0g and solids = 1.

#### 12.0 QUALITY CONTROL AND DATA ASSESSMENT



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#### 12.1 Analytical Batching

- 12.1.1 QC data must be evaluated against the precision and accuracy criteria set forth in the Method Limit Groups in STL LIMS and SOP AN02: *Analytical Batching and Evaluation of QC Data*. SOP AN02: *Analytical Batching* describes the procedure for evaluating batch-specific QC. These criteria are summarized in Appendix B.
- 12.1.2 The analytical batch consists of up to twenty client samples and the associated QC items that are analyzed together. The matrix spike and LCS frequency is defined in SOP AN02. Note that the method blank for liquid samples and low-level soils is clock-specific and that the method blank for medium level soil samples is extraction batch-specific.
- 12.1.3 SOP AN02 also contains the calculations for accuracy and precision and the calculations for the theoretical concentrations of surrogates, lab spikes, and matrix spikes.
- 12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP QA05: *Preventive and Corrective Action Procedures*. SOP QA05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

#### 13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with this procedure are given in the Method Limit Groups in STL LIMS.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP QA06: *Training Procedures*.

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP QA07: *Determination of Detection Limits (MDLs and IDLs).* 

#### 14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

See SOP QA09: *Maintenance Procedures for Laboratory Instrumentation* for guidance on routine instrument maintenance. See instrument manufacturers' manuals for guidance on locating and repairing instrument problems.

#### 15.0 WASTE MANAGEMENT AND POLLUTION CONTROL

All waste will be disposed of in accordance with Federal, State and Local regulations. Follow the guidance for disposal in the STL Savannah Addendum to the Corporate Safety Manual. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

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Excess samples, reagents, and standards must be disposed in accordance with the STL Savannah Addendum to the Corporate Safety Manual.

The following waste streams are produced when this method is carried out.

- Methanolic waste from rinsings and standards. Transfer to satellite container for methanolic (flammable) waste. Transfer to hazardous waste section when satellite container is full.
- Excess aqueous samples Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

#### 16.0 REFERENCES

STL Savannah's Laboratory Quality Manual (LQM), current revision

Severn Trent Laboratories' Quality Management Plan (QMP), current revision

Methods 5035, 8000B, and 8260B. *Test Methods for Evaluating Solid Wastes, Third Edition, SW-*846.including Update II<u>I</u>U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.

#### 17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

- Appendix A contains a list of routine target analytes and their respective ions and internal standards.
- Appendix B contains the SOP Summary.



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#### THE LEADER IN ENVIRONMENTAL TESTING

#### Appendix A – Routine Target Compounds\*

RT	Compound	CAS	ISTD	Quant Ion	Secondary	/ Ions
		Standard				
3.023	1,2-DICHLOROETHANE-d4	17060-07-0	1	65	67	102
3.236	1,4-DIFLUOROBENZENE	540-36-3	2	114	63	88
5.048	CHLOROBENZENE-d5	3114-55-4	3	82	117	119
0.040		mpounds	0	02	117	115
1.198	DICHLORODIFLUOROMETHANE	75-71-8	1	85	87	101
1.198	CHLOROMETHANE	74-87-3	1	50	52	101
1.344	VINYL CHLORIDE	74-87-3	1	62	64	
1.490	BROMOMETHANE	73-01-4	1	96	94	79
1.533	CHLOROETHANE	74-83-9	1	90 64	94 66	19
-			1			105
1.654	TRICHLOROFLUOROMETHANE	75-69-4		101	103	105
1.818		107-02-8	1	56	55	100
1.867	TRICHLOROTRIFLUOROETHANE(113)	76-13-1	1	101	151	103
1.879	1 1-DICHLOROETHENE	75-35-4	1	96	61	98
1.873	ACETONE	67-64-1	1	58	43	
	IODOMETHANE	74-88-4	1	142	127	
1.995	CARBON DISULFIDE	75-15-0	1	76	78	
1.989	ACETONITRILE	75-05-8	1	41	40	
2.019	3-CHLORO-1-PROPENE	107-05-1	1	76	41	
2.068	METHYLENE CHLORIDE	75-09-2	1	84	49	86
2.153	ACRYLONITRILE	107-13-1	1	53	52	51
2.190	trans-1 2-DICHLOROETHENE	156-60-5	1	96	61	98
2.178	METHYL T-BUTYL ETHER	1634-04-4	1	73	57	
2.366	1 1-DICHLOROETHANE	75-34-3	1	63	65	83
2.360	VINYL ACETATE	108-05-4	1	43	86	
2.414	CHLOROPRENE	126-99-8	1	53	88	
2.628	cis-1 2-DICHLOROETHENE	156-59-2	1	96	61	98
2.634	2 2-DICHLOROPROPANE	594-20-7	1	77	41	
	2-BUTANONE	78-93-3	1	43	72	
2.639	PROPIONITRILE	107-12-0	1	54	55	
2.719	METHACRYLONITRILE	126-98-7	1	67	52	
2.743	BROMOCHLOROMETHANE	74-97-5	1	49	128	130
2.768	CHLOROFORM	67-66-3	1	83	85	47
	1 1 1-TRICHLOROETHANE	71-55-6	2	97	99	61
2.956	1 1-DICHLOROPROPENE	563-58-6	2	75	110	77
2.968	CARBON TETRACHLORIDE	56-23-5	2	117	119	121
2.900	ISOBUTANOL	78-83-1	2	43	41	121
3.066	BENZENE	71-43-2	2	78	50	
3.060	1 2-DICHLOROETHANE	107-06-2	2	62	49	64
3.060	TRICHLOROETHENE	79-01-6	2	130	49 95	132
3.516		78-87-5	2	63	76	65
3.540		80-62-6	2	69	41	05
3.583		74-95-3	2	93	174	95
3.656		75-27-4	2	83	85	129
3.808	2-CHLOROETHYL VINYL ETHER	110-75-8	2	63	65	106
3.911	cis-1,3-DICHLOROPROPENE	10061-01-5	2	75	77	110



THE LEADER IN ENVIRONMENTAL TESTING

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RT	Compound	CAS	ISTD	Quant Ion	Secondar	y lons
	Target Comp	ounds, cont'	d			
3.990	4-METHYL-2-PENTANONE (MIBK)	108-10-1	2	43	57	58
4.136	TOLUENE	108-88-3	2	92	91	65
4.246	trans-1,3-DICHLOROPROPENE	10061-02-6	2	75	77	110
4.288	ETHYL METHACRYLATE	97-63-2	3	69	41	39
4.368	1 1 2-TRICHLOROETHANE	79-00-5	2	83	97	99
4.495	TETRACHLOROETHENE	127-18-4	3	164	166	168
4.483	1 3-DICHLOROPROPANE	142-28-9	2	76	78	41
4.514	2-HEXANONE	591-78-6	3	43	58	
4.641	DIBROMOCHLOROMETHANE	124-48-1	3	129	127	131
4.733	1 2-DIBROMOETHANE	106-93-4	2	107	109	
5.073	CHLOROBENZENE	108-90-7	3	112	77	51
5.116	1 1 1 2-TETRACHLOROETHANE	630-20-6	3	131	133	119
5.140	ETHYL BENZENE	100-41-4	3	91	106	51
5.225	m,p-XYLENE	108-38-3	3	106	91	77
5.517	o-XYLENE	95-47-6	3	106	91	77
5.523	STYRENE	100-42-5	3	104	78	103
5.657	BROMOFORM	75-25-2	3	173	171	175
5.785	ISOPROPYLBENZENE	98-82-8	3	105	120	77
6.028	BROMOBENZENE	108-86-1	3	156	77	158
5.992	1 1 2 2-TETRACHLOROETHANE	79-34-5	3	83	85	168
6.028	1 2 3-TRICHLOROPROPANE	96-18-4	3	110	112	100
6.034	trans-1,4-DICHLORO-2-BUTENE	110-57-6	3	53	88	89
6.101	n-PROPYLBENZENE	103-65-1	3	120	91	65
6.174	2-CHLOROTOLUENE	95-49-8	3	126	91	63
6.235	1 3 5-TRIMETHYLBENZENE	108-67-8	3	105	120	77
6.253	4-CHLOROTOLUENE	106-43-4	3	126	91	63
6.491	tert-BUTYLBENZENE	98-06-6	3	119	91	134
6.502	PENTACHLOROETHANE	76-01-7	3	167	130	104
6.527	1 2 4-TRIMETHYLBENZENE	95-63-6	3	107	120	77
6.661	sec-BUTYLBENZENE	135-98-8	3	105	134	91
6.752	1 3-DICHLOROBENZENE	541-73-1	3	146	148	111
6.777	p-ISOPROPYLTOLUENE	99-87-6	3	119	134	91
6.825	1 4-DICHLOROBENZENE	106-46-7	3	146	148	111
7.123	1 2-DICHLOROBENZENE	95-50-1	3	146	148	111
7.125	n-BUTYLBENZENE	104-51-8	3	91	92	134
7.738	1 2-DIBROMO-3-CHLOROPROPANE	96-12-8	3	157	75	154
8.498	1 2 4-TRICHLOROBENZENE	120-82-1	3	180	182	145
8.687	HEXACHLOROBUTADIENE	87-68-3	3	225	223	-
8.748	NAPHTHALENE	91-20-3	3	128	102	190 51
			3			51
9.021	1 2 3-TRICHLOROBENZENE	87-61-6 ogates	3	180	182	I
2.841		1868-53-7	1	113	81	111
4.094	TOLUENE-d8	2037-26-5	2	98	100	70
5.901	p-BROMOFLUOROBENZENE	460-00-4	3	98 95	174	176
0.901	p-BROMOFLUOROBENZENE	460-00-4		95		0/1

\* For a complete list of target analytes refer to the Method Limit Groups in LIMS.



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#### Appendix B SOP SUMMARY

### SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME SUMMARY

MATRIX	Preservative/ Storage*	Container	Holding Time
Aqueous	None; 4ºC	4 x 40-mL no headspace	7 days
	HCl pH<2; 4⁰C	4 x 40-mL no headspace	14 days
Soil/solid(low level) -low or no carbonates	Iced at collection; 5mL sodium bisulfate added upon arrival in lab within 48 hours of collection; store at 4°C	3 x 5-g Encore Sampler	14 days
Soil/solid(low level) -high carbonates	Iced at collection; 5mL water added upon arrival in lab within 48 hours of collection; store at -10°C	3 x 5-g Encore Sampler	14 days
Soil/solid(high level)	None; 4ºC	Glass 125-mL	14 days
TCLP	HCl pH<2; 4⁰C	Tedlar bag or syringe	14 days

\*storage temperature is 4°C with a control criteria of less than 6C with no frozen samples

#### ANALYSIS SEQUENCE

INITIAL CALIBRATION	CONTINUING CALIBRATION
4-BFB 50ng on column	4-BFB 50ng on column
Clock starts at injection	Clock starts at injection
Trap Check Standard	Trap Check Standard
Calibration standards -	Mid point calibration verification (50ug/L or 50ug/kg))
Minimum of five cal levels	RL Standard - low point on cal curve (if necessary)
Laboratory Control Sample	Laboratory Control Sample
Method blank	Method blank
Samples analyzed until the 12-hour clock expires	Samples analyzed until 12-hour clock expires

#### **Recommended Internal Standards:**

1,2-dichloroethane-d4; 1,4-difluorobenzene; chlorobenzene-d5; 1,4-dichlorobenzene-d4

#### Surrogates/System Monitoring Compounds:

dibromofluoromethane; toluene-d8; 4-bromofluorobenzene

LCS/MS/MSD: Routine list is TCL List



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#### Appendix B SOP SUMMARY

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)				
m/e	Abundance Criteria			
50	8.0-40.0% of mass 95			
75	30.0-66.0% of mass 95			
95	Base peak, 100% relative abundance			
96	5.0-9.0% of mass 95			
173	< 2.0% of mass 174			
174	50-120%% of mass 95			
175	4.0-9.0% of mass 174			
176	93.0-101.0% of mass 174			
177 5.0-9.0% of mass 176				

(1) \*8260 criteria taken from CLP OLMO4.0 (January 1998)

## CALIBRATION ACCEPTANCE CRITERIA

#### Calibration Check Compounds - CCC

Vinyl chloride, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene

Initial Calibration	Continuing Calibration
Less than or equal to 30% RSD	Less than or equal to 20% difference or drift from initial calibration

#### System Performance Check Compounds - SPCC

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30

See Sections 10.3 and 10.4 for ICAL and CCV linearity checks and criteria.



#### THE LEADER IN ENVIRONMENTAL TESTING

## Appendix B-SOP Summary

# TestAmerica Savannah

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QC Check	Frequency	Acceptance Criteria	Corrective Action
MS Tune Check – 50ng 4-BFB	Before initial and continuing calibration standards - every 12 hours	Mass abundances within method acceptance criteria	-Evaluate chromatogram and spectrum - Reanalyze - Retune MS and reanalyze - Remake standard and reanalyze - Perform instrument maintenance and reanalyze
Trap Check Standard – 100ng Bromoform, internal standards, and surrogates	Before initial and continuing calibration standards - every 12 hours	Chloromethane and bromomethane less than 0.5ug/L	<ul> <li>Reanalyze</li> <li>Change trap and recalibrate</li> </ul>
Initial Calibration – minimum five point curve with lowest point at or below the Reporting Limit (RL)	Initially; after major instrument maintenance; whenever continuing calibration check fails. Prior to analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria Table presented earlier in this document) ICAL must be verified with 2 <sup>nd</sup> source ICV per SOP AN02	<ul> <li>Evaluate chromatograms, spectra, and integrations</li> <li>Reanalyze standard(s)</li> <li>Remake and reanalyze standard(s)</li> <li>Perform instrument maintenance and recalibrate</li> </ul>
Continuing Calibration check – midpoint standard	Every 12 hours before analysis of method blank and samples	Method criteria for CCC/SPCC (see Calibration Acceptance Criteria Table presented earlier in this document)	<ul> <li>Evaluate chromatogram, spectra, integrations</li> <li>Reanalyze standard</li> <li>Remake and reanalyze standard</li> <li>Recalibrate</li> <li>Perform instrument maintenance and recalibrate</li> </ul>
Method Blank	Every 12 hours (per clock) before sample analyses	All reported targets <rl< td=""><td>-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze</td></rl<>	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze - Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze



#### THE LEADER IN ENVIRONMENTAL TESTING

# TestAmerica Savannah

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### Appendix B-SOP Summary

QC Check	Frequency	Acceptance Criteria	Corrective Action			
Lab Control Sample (LCS)	Each batch	MLG	-Evaluate chromatogram and integrations. Check calculations. -Follow guidance in SOP AN02 -Perform instrument or column maintenance, recalibrate, and reanalyze			
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Each batch	MLG	<ul> <li>-Evaluate chromatogram and integrations. Check calculations.</li> <li>-Follow guidance in SOP AN02</li> <li>-Perform instrument or column maintenance, recalibrate, and reanalyze</li> <li>-Evaluate chromatogram and integrations. Check calculations.</li> <li>-Reanalyze</li> <li>- Follow guidance in SOP AN02</li> <li>-Perform instrument or column maintenance, recalibrate, and reanalyze</li> </ul>			
Surrogates	All samples, blanks, LCS, MS	MLG				
Internal Standard Area	Evaluate all standards and samples	-Areas in continuing calibration verification must be 50% to +100% of previous initial calibration sequence -RT of internal standard must be +/-30 seconds from internal standard in initial calibration -Areas in samples should be	-Evaluate chromatogram and integrations. Check calculations. -Reanalyze			
		evaluated for gross error. -Consult supervisor.				
Reporting Limit Standard -1x to 2x the RL	(Optional) Daily. Required for Florida DEP	Detected with reasonable response	-Evaluate chromatogram, spectra, and integrations -Reanalyze -Remake standard and reanalyze -Retune and recalibrate -Perform instrument maintenance and recalibrate			
Demonstration of Capability	Initially and then annually per analyst	Method criteria See SOP CA01	-Reanalyze targets that do not meet criteria			



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QC Check	Frequency	Acceptance Criteria	Corrective Action
Method Detection Limit (MDL)	See SOP QA07	See SOP QA07	-Reanalyze and re-evaluate



# **TestAmerica Knoxville SOP Cover Page**

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QA088R0, 12/20/07

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# STL KNOXVILLE

## STANDARD OPERATING PROCEDURE

# **TITLE: VOA CANISTER ANALYSIS**

(SUPERSEDES: KNOX-MS-0001, Revision 8)

Prepared By:	Holly 2.
Reviewed By:	The Star w/s/00
Approved By:	Technical Specialist Muchantur Much 10/05/04
	Quality Assurance Manager B · O. July 10-5-06
Approved By:	Equironmental, Health and Safety Coordinator 10 - 6 - 26
Approved By:	Laboratory Director

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# 1. Scope and Application

- 1.1. The purpose of this standard operating procedure is to define the procedures and quality control necessary to analyze samples collected in "SUMMA<sup>TM</sup> passivated" stainless steel canisters.
- 1.2. This procedure is applicable to the analysis of ambient air, indoor air, landfill gases, soil gases, vapor intrusion, and other gaseous samples. It is based on EPA Methods TO-14, TO14A and TO-15.

Position	Responsibilities
Analyst	- Prepares and analyzes samples
	<ul> <li>Summarizes/assembles data package</li> <li>Reviews the data package</li> </ul>
Team/Group Leader	<ul> <li>Schedules/assigns analyses</li> <li>Reviews data package</li> </ul>

1.3. Responsibilities to perform this procedure in the lab are as follows:

# 2. Summary of Method

- 2.1. Microscale Purge and Trap (MSPT): A precisely measured aliquot is removed from the canister or Tedlar bag and concentrated on a cryogenic trap. The cryogenic trap is desorbed. Polar and nonpolar compounds are quantitatively transferred to a subambient Tenax<sup>™</sup> trap. Most of the water remains on the Cryotrap and CO2 passes through the Tenax trap and is vented. The Tenax<sup>™</sup> trap is thermally desorbed to the on-column cryofocuser. Sample components are separated by temperature programmed gas chromatography and detected with a quadrupole mass spectrometer.
- 2.2. The compounds analyzed by this method are listed in Tables 1, 2 and 3.

# 3. Definitions

- 3.1. Canister a stainless steel container, typically 6-liter volume, equipped with a stainless steel shut-off valve, suitable for use from vacuum to 40 psig. 1-L cans are available for reduced volume analysis.
- 3.2. SUMMA<sup>TM</sup> Passivation a proprietary treatment process used to deactivate stainless steel surfaces. It produces a pure chrome/nickel oxide surface that features a high level of inertness.

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- 3.3. Absolute pressure pressure measured with reference to absolute zero pressure, expressed as kpa, mmHg, or psia.
- 3.4. Gauge pressure pressure above atmospheric pressure as measured by a standard gauge. Zero gauge pressure is equal to ambient atmospheric pressure, expressed as mmHg, inches Hg, or psig.
- 3.5. Polar compound Oxygen-containing compound capable of forming hydrogen bonds in water; compound having significant solubility in water.
- 3.6. Batch A batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same 24 hour time period. The Quality Control batch must contain a blank and a Laboratory Control Sample (LCS). Refer to the QC Program document (QA-003, current revision) for further details of the batch definition.
- 3.7. Additional definitions can be found in the STL Knoxville LQM glossary and in the STL Quality Management Plan.
- 3.8. Tedlar bag Tedlar bags are manufactured from PVF (Tedlar) film with a polypropylene valve and septum. Various volume capacities available.

# 4. Interferences

- 4.1. Only compounds having both a similar mass spectrum and GC retention time would be expected to interfere in the method. The most common occurrence of this would be with structural isomers.
- 4.2. Large concentrations of water, methane, or carbon dioxide may limit the size of the aliquot that can be effectively cryotrapped. This may elevate the quantitation limits obtainable for samples of this type.
- 4.3. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.
- 4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination, or evaluate the next sample for blank acceptance criteria. The autosampler and concentrator may require extensive bake-out and cleaning after a high-level sample.

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## 5. Safety

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2. Procedures shall be carried out in a manner that protects the health and safety of all associates. Exposure to chemicals and samples will be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. The preparation of all standards, reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit.
- 5.3. All work must be stopped in the event of a known or potential compromise to the health and safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.
- 5.4. Specific Safety Concerns or Requirements
  - 5.4.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.
  - 5.4.2. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
  - 5.4.3. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
  - 5.4.4. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power
- 5.5. Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetone	Flammable	1000 ppm (TWA)	Inhalation may cause coughing, dizziness, dullness, and headache. Contact causes redness, pain, drying and cracking of the skin. Vapors cause eye irritation. Eye splashes may cause severe irritation, with stinging, tearing, redness and pain.
Benzene	Carcinogen Flammable Poison	1 ppm-TWA 5 ppm-STEL	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness, weakness and breathing difficulties. This material is irritating on contact with the skin and eyes and may cause permanent eye damage.
Carbon Tetrachloride	Carcinogen Poison	10 ppm-TWA 200 ppm-STEL	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness and narcosis. Contact with skin or eyes may cause irritation. Consumption of alcohol may increase toxic effects
Chloroform	Carcinogen Irritant	50 ppm Ceiling	Acts as a relatively potent anesthetic. Irritates respiratory tract and causes central nervous system effects, including headache, drowsiness, dizziness. Causes skin irritation resulting in redness and pain. Removes natural oils. May be absorbed through skin. Vapors cause pain and irritation to eyes. Splashes may cause severe irritation and possible eye damage.
1,4- Dichlorobenzene	Irritant	75 ppm-TWA	Can cause irritation by ingestion and inhalation. Causes nausea, vomiting and diarrhea. Contact with material or vapors can cause irritation to skin and eyes.
Vinyl Chloride	Carcinogen Flammable Poison	1 ppm TWA	Toxic by inhalation, ingestion and absorption. Can cause respiratory irritation, dizziness, weakness, fatigue, nausea and headache. Contact with the material can cause eye and skin irritation.
1 – Exposure limit	refers to the OSHA	A regulatory exposu	

# 6. Equipment and Supplies

- 6.1. Canisters, 1, 6-, 15-, and 30-liter sizes, preferably equipped with two valves and integral vacuum/pressure gauge, Scientific Instrumentation Specialists or equivalent.
- 6.2. Static gas dilution bottles (SGDB), nominally 2000 ml, with mininert valves, Tekmar Co., or equivalent.
- 6.3. Syringes, gas-tight, 10 uL, 50 uL, 500 uL, 1000 uL, 2.5 mL, 50 mL, 500 mL, all side port needle, Hamilton, Inc., or equivalent.

- 6.4. Gas Chromatograph/Mass Spectrometer System, Agilent HP 6890 GC and 5973 MSD or equivalent.
- 6.5. Fused silica capillary column, 60 m x 0.32 x 1um film DB-5, J&W Scientific, or equivalent.
- 6.6. Vacuum pump, Model 726.3 TTP, KNF Newberger, or equivalent.
- 6.7. Canister concentrator system, Model 7100 or 7100A, Entech Co., with a Model 7016CA, 16-position auto sampler.
- 6.8. Gauges: The following gauges are certified annually
  - 6.8.1. Test gauge, 0 to 30 in. Hg vacuum, Ashcroft Co., or equivalent
  - 6.8.2. Test gauge, 0-60 psi, Ashcroft Co., or equivalent
  - 6.8.3. Test gauge, 0 to 100 in. Hg pressure, Ashcroft Co., or equivalent
  - 6.8.4. Digital gauge 0 to 30" Hg vacuum, Dwyer or equivalent
  - 6.8.5. Digital gauge 0 to -29.9" Hg vacuum, 0 to 99.9 psi, Dwyer or equivalent
- 6.9. Tedlar Bags: Variety of sizes. SKC or equivalent.

# 7. Reagents and Standards

- 7.1. Helium, ultra high purity, 99.999+%, Air Products, or equivalent.
- 7.2. Liquid nitrogen, Air Products, or equivalent.
- 7.3. Nitrogen, ultra high purity, Air Products or equivalent
- 7.4. Internal/Surrogate Standard (all at 100 ppb) in nitrogen, 2000 psig, Scott Specialty Gases, or equivalent:

CAS NUMBER	Internal Standards	MOLECULAR WEIGHT (ng/n mole)				
74-97-5	bromochloromethane	129.4				
540-36-3	1,4-difluorobenzene	114.1				
3114-55-4	chlorobenzene-d5	117.6				
	Surrogates					
17060-07-0	1,2-dichloroethane-d4	103.0				
2037-26-5	toluene-d8	100.2				
460-00-4	4-bromofluorobenzene	175.0				

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- 7.4.1. A sufficient volume from the internal/surrogate standard cylinder is transferred to the 15-liter summa canister to produce a positive pressure.
- 7.4.2. The working internal/surrogate standard may be used as long as the pressure in the canister remains above ambient pressure and is not past its expiration date.
- 7.4.3. The Entech is programmed to add 20 mL of the internal standard/surrogate can. This results in a concentration of 4 ppb/v of internal standard/surrogate (based on 500 mL volume).
- 7.5. Primary Target and Laboratory Control Sample Gaseous Standards: target compounds, 1000 ppb v/v, vendor-certified high-pressure aluminum cylinder.
  - 7.5.1. An expiration date of one year from the date of vendor certification is assigned to the standard cylinder. This expiration date may be extended through comparison against an unexpired standard that meets the second source standard criteria in Section 10.4.
- 7.6. Initial Calibration Verification Standard (2<sup>nd</sup> source) stock cylinders (ICV): Target compounds, 300 ppb v/v, vendor-certified high-pressure aluminum cylinders.
  - 7.6.1. These cylinders have been used for over 10 years and no significant degradation in response has been observed. Due to this stability, the calibration verification (second source) standard stock cylinders may be used for 20 years from date of certification or until the vendor supplied expiration date, whichever is earlier. It is also subject to ongoing monitoring of target analytes against the primary calibration standard.
- 7.7. Standard grade neat compounds of hexachlorobutadiene (HCBD) and naphthalene, 99+% or of known purity, Chem Service, or equivalent.
  - 7.7.1. Naphthalene/Hexachlorobutadiene (HCBD) Stock Standard: Approximately 3.6 mg of naphthalene is weighed into a glass container small enough to be dropped into a SGDB. The glass container is introduced into the SGDB and the bottle is capped with a mininert valve. Approximately 4.4 uL of HCBD is added to the SGDB using a 10 uL syringe. The SGDB is placed in an oven at approximately 60°C. The exact weight/volume to be added is calculated from the volume of the SGDB such that adding 2.5 milliliters from the SGDB to a 15-L can (or 1

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mL in a 6-L can) pressurized to 30 psig yields a concentration of 25 ppb (v/v) of HCBD and Naphthalene in the working standard. See section 12.7 and 12.8 for calculations.

- 7.8. Prepared Standard, polar compounds, vendor certified mix containing methanol, ethyl ether, acetone, acrylonitrile, vinyl acetate, 2-butanone, 1-butanol, 4-methyl-2-pentanone, 2-hexanone, methyl-tert-butyl ether, acrolein, acetonitrile, 99+% or of known purity, Ultra Scientific or equivalent.
- 7.9. Polar stock standard and 1,2,3-trichloropropane, 99+% or of known purity, Chem Service or equivalent.
  - 7.9.1. SGDB stock method: Approximately 27 μL of the polar standard mix (section 7.8) and approximately 1.3 uL of 1,2,3-trichloropropane is injected into a SGDB. The exact volume to be added is calculated from the volume of the SGDB such that adding 5 mL from the SGDB to the working standard yields a concentration of 100 ppb (v/v) for methanol and 50 ppb (v/v) for the other polar compounds and 25 ppb (v/v) of 1,2,3-trichloropropane in the working standard.
  - 7.9.2. Water stock method: 67.5 uL of the polar standard mix and 3.25 uL of 1,2,3-trichloropropane are dissolved in water to a final volume of 10 mL.
- 7.10. Additional Standards: Neat materials, not contained in the certified cylinders, can be added to a SGDB either individually or as a mix.
  - 7.10.1. If the desired compound is a gas at room temperature, a measured volume is injected into an evacuated summa canister and pressurized. See section 12.9 and 12.10 for calculation. If the desired compound is a liquid or solid at room temperature, the volume of each compound to be added to the SGDB should be back calculated to the desired final concentration in the canister. See section 12.7 and 12.8 for calculation.
- 7.11. 50ppb v/v Canister Working Standard (for a 15-L can. For a 6-L can, reduce the volume of standards appropriately).
  - 7.11.1. 100ul of reagent water is injected through a septum (inserted into a ¼ in. nut) into a clean evacuated 15-L canister.
  - 7.11.2. The canister is then brought up to 0" gauge pressure with UHP nitrogen
  - 7.11.3. 3 in (Hg) of the 1000 ppb v/v high pressure gas standard from Section 7.5 is added to the 15-liter canister. The can is pressurized to 30 in (Hg) with

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UHP nitrogen for a final volume of 30 L and final concentration of 50ppb v/v.

- 7.11.4. Alternate concentrations of the working standards may be made as long as the calculations, concentrations and volumes are adjusted appropriately and preparation is clearly documented in the standard preparation logbook.
- 7.12. Low Standard Preparation: Typically 1 ppb v/v or 2 ppb v/v calibration point
  - 7.12.1. 40  $\mu$ L of reagent water is injected through a septum (inserted into a 1/4 inch nut) into a clean evacuated 6-liter canister. The canister is allowed to stand for at least 20 minutes to allow all the water to evaporate from the valve area.
  - 7.12.2. 240 mL (2.4" Hg) (for a 1 ppb v/v standard) or 480 mL (4.8" Hg) (for a 2 ppb v/v standard) of the 50 ppb (v/v) standard is transferred to a 6-liter canister.
  - 7.12.3. The canister is pressurized to 30 in. Hg with UHP nitrogen.
- 7.13. 50 ppb v/v Canister Second Source Standard (for a 15-L can. For a 6-L can, reduce the volume of the standards appropriately).
  - 7.13.1. 100 uL of reagent water is injected through a septum (inserted into a 1/4in. nut) into a clean evacuated 15-liter canister. The canister is allowed to stand for at least 20 minutes to allow all the water to evaporate from the valve area.
  - 7.13.2. 10 uL from the water standard in 7.9.2 (or 5 mL of the polar stock from the SGDB in 7.9.1) and 2.5 mL of the naphthalene/HCBD stock in 7.7.1 are injected through the septum. This step should be done quickly and the syringe for the naphthalene/HCBD stock should be heated in the oven along with the SGDB. The canister is then brought up to exactly zero inches gauge pressure with UHP nitrogen
  - 7.13.3. 5 in. (Hg) of each 300 ppb v/v high-pressure gas standard from section 7.5 is added to the 15-liter canister. The can is pressurized to 30 in (Hg) with UHP nitrogen for a final volume of 30L and final concentration of 25 ppb v/v
  - 7.13.4. Alternate concentrations of the working standards may be made as long as the calculations, concentrations and volumes are adjusted appropriately and preparation is clearly documented in the standard preparation logbook

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- 7.14. Approved SGDB and canister stock standards (section 7.7, 7.8, 7.9 and 7.10) may be used for 6 months from the date of preparation or the earliest expiration of parent standard, whichever comes first. Working canister standards (7.11 and 7.12 and 7.13) may be used for two months from the date of preparation or the earliest expiration of parent standard, whichever comes first.
- 7.15. The HCBD/naphthalene SGDB is stored at approximately 60°C. Other SGDB and canister standards are stored at room temperature. If the analytes prove to be plating/condensing in the SGDB at room temperature, then the SGDB should be stored at approximately 60°C. Mixes and neat compounds (that are not in SGDB, cans, or cylinders) are stored at the manufacturer's recommended storage conditions.
- 7.16. Approval of Stock and Working Standards
  - 7.16.1. When a new stock is prepared, it can either be verified at the SGDB stage or at the working level stage.
    - 7.16.1.1. To compare the SGDB standards, humidify two 6 liter canisters with 40 μL of water each, and spike with equal known volumes (typically 5 10 mL) from the SGDB, and bring to an equal final pressure of nominally 15 psig.
    - 7.16.1.2. The two standards must agree to within 20 percent difference after taking into account nominal volume differences between the two bottles.
  - 7.16.2. Working canister standards are approved for use by passing the daily standard acceptance criteria given in Section 10.5 or the initial calibration verification in section 10.4. The working LCS canister is approved for use by passing the LCS acceptance criteria in section 9.3.

## 8. Sample Collection, Preservation and Storage

8.1. Sampling is not performed for this method by STL Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, Receipt and Log In of Commercial Samples, current revision.

Container Type	Preservative	Holding Time
SUMMA canister	None	30 days
Tedlar bag	None	72 hours from collection to analysis or transfer to a can. After transfer to a can, the holding time is 30 day from sample collection.

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## 9. Quality Control

- 9.1. Internal/Surrogate Standards
  - 9.1.1. Internal standards and surrogates are added to each analytical standard, blank and sample. The acceptance criteria for each internal standard's area for every analysis must be  $\pm$  40% recovery of the internal standard area from the continuing calibration standard. The acceptance criteria for each internal standard's retention time in every analysis must be within  $\pm$  20 seconds (0.33 minutes) of the internal standard retention time from the continuing calibration standard.
  - 9.1.2. Surrogate recoveries must fall within 70% to 130%, or within laboratory historical control limits if available.
  - 9.1.3. If the internal standard areas or surrogate recoveries for a sample are outside their limits, the cause is determined. If it is a result of a system problem, then the problem must be corrected and the sample reanalyzed with acceptable results. If it is the result of a matrix effect, the sample must be reanalyzed to confirm this, unless the effect is caused by high levels of target or non-target compounds co-eluting with or interfering with the surrogates or internal standards.
  - 9.1.4. If the sample surrogate recoveries are biased high outside acceptance limits and no target analytes are detected above the reporting limit, the sample data may be reported with qualification in the project narrative.
- 9.2. System Blanks
  - 9.2.1. For each 24-hour tune in which samples are analyzed or every 20 samples, whichever is more frequent, an acceptable system blank must be analyzed before samples analysis may begin.
    - 9.2.1.1. A system blank is defined as a cleaned canister, humidified with reagent water and filled with UHP nitrogen.
    - 9.2.1.2. Typically, a 30L canister, humidified with 200 uL of reagent water and pressurized to 25-30 psi with nitrogen is used for the blank. A lot check from the can cleaning system can be used as a system blank (See section 9.5).
    - 9.2.1.3. An acceptable system blank is one with all target analytes less than 0.2 ppbv. The data may still be reported if the concentration of the analyte is less than the laboratory

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reporting limit (see Tables 1, 2 and 3), and meets internal standard and surrogate requirements in section 9.1. Any samples associated with a method blank with results above 0.2 ppbv are flagged in the data report. If a blank has a reportable result between the RL and the MDL, the associated samples are also flagged.

- 9.2.2. If a system blank does not meet the above criteria, then the blank must be reanalyzed or a new blank prepared and analyzed with acceptable results.
- 9.3. Laboratory Control Standard (LCS)
  - 9.3.1. The LCS is defined as a working standard made by the same method as analytical standards, using the same source materials. It is used to assess analytical control of this procedure. The LCS is analyzed every 24 hour tune or every 20 samples, whichever is more frequent.
    - 9.3.1.1. The daily calibration verification may also serve as the LCS as long as it meets the criteria of both the LCS and the daily calibration verification.
  - 9.3.2. All non-polar analytes in the LCS must be within 70-130% recovery with the allowance of up to two non-polar analytes having 60-140% recovery. All polar analytes in the LCS must be within 60-140% recovery with the allowance of up to two polar analytes having 45-155% recovery.
  - 9.3.3. The internal standards and surrogates must pass criteria specified in section 9.1.
  - 9.3.4. If the above criteria cannot be met, corrective action must take place. Corrective action may include: a reanalysis of the LCS, performing instrument maintenance, preparation of a new working standard, or recalibration of the instrument. Corrective action is followed by reanalysis of any samples associated with the LCS that failed acceptance criteria.
  - 9.3.5. Note: If the LCS recovery for a target analyte is biased high outside acceptance limits and that target analyte is not detected in any of the associated samples above the reporting limit, the sample data may be reported with qualification in the project narrative.
- 9.4. Duplicate Analysis
  - 9.4.1. A duplicate is analyzed with every 20 samples. It is not reported unless specifically requested.

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- 9.4.2. The acceptance criteria for the duplicate analysis are  $\leq 25\%$  RPD for target compounds that are greater than 5 times the RL. No criteria for methanol and n-butanol. The calculations are given in section 12.16.
- 9.4.3. If the RPD is outside acceptance criteria for the duplicate, the sample is rerun once. If upon reanalysis, the duplicate does not meet acceptance criteria, the original sample data is qualified in the project narrative.
- 9.4.4. Due to limited sample volume, duplicates are not performed for Tedlar bags unless otherwise specified in the project requirements.
- 9.5. Canister Blank Checking
  - 9.5.1. From each cleaned lot of canisters, a canister is selected, humidified with 40 μL reagent water, and pressurized with UHP nitrogen. (See SOP KNOX-SC-0001, current revision, "Canister Cleaning and Preparation").
  - 9.5.2. A blank check is analyzed within 24 hours of a valid tune check and calibration.
  - 9.5.3. A blank check passes if there are no target analytes above the reporting limit, and the internal standards and surrogates pass criteria in section 9.1. Cans are considered certified "clean" if the result for all analytes are below 0.2 ppbv. However the can may still be used to collect samples if the concentration of the target analyte is less than the reporting limit. If analytes are detected in the can being certified as clean above 0.2 ppbv and below the reporting limit, this will be noted on the blank check quantitation report.
  - 9.5.4. If a blank check canister does not pass, the can may be re-analyzed. If the acceptance criteria are still not met, the entire lot of canisters must be re-cleaned, and a blank check from the re-cleaned lot must pass.
- 9.6. Nitrogen check
  - 9.6.1. Before a new nitrogen cylinder is used for pressurization of samples or standards, it must be analyzed as a blank and pass all the criteria in section 9.2.1.3.
- 9.7. Annual gauge calibration: The gauges that are used in calculations to measure cylinder and canister pressure or vacuum must be certified annually.

## 10. Calibration and Standardization

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- 10.1. Instrument Conditions: The following steps are part of the software's automatic tuning procedure and are performed as needed.
  - 10.1.1. Mass assignments of the mass spectrometer are checked and adjusted using perfluorotributylamine (PFTBA FC43).
  - 10.1.2. The mass spectrometer is tuned to meet the criteria for BFB (see Figure 1).
  - 10.1.3. The mass spectrometer is adjusted to minimize noise (see instrument manufacturer instruction manuals).
  - 10.1.4. See Appendix III for examples of GC/MS and GC instrument parameters.
- 10.2. Daily Tune Check
  - 10.2.1. 50 ng or less of BFB is analyzed for each 24-hour time tune period; the 24-hour time period begins at the moment of injection of BFB. All abundance criteria for BFB in Figure 1 must be met before the analysis of standards, QC samples or client samples.
  - 10.2.2. The BFB must be acquired in the following manner: Three scans (the apex scan, and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.
  - 10.2.3. Once the BFB passes criteria, the same mass spectral conditions used for the BFB must be used to acquire the data in that 24-hour tune period, until the next BFB event.
- 10.3. Initial Calibration
  - 10.3.1. The GC/MS system must be calibrated with at least 5 concentrations that span the monitoring range of interest. The dynamic range of the curve is generally 0.2 ppb v/v to 30 ppb v/v based on 200 mL sample analysis for normal level reporting limits for most analytes, and 0.01 ppb v/v to 1 ppb v/v based on 500 mL sample analysis for low level reporting limits, for a limited set of analytes. The concentration of the low standard of the calibration must be at or below the reporting limit. If quadratic fit is required, there must be at least 6 points. See Appendix IV for the recommended calibration amounts.
  - 10.3.2. See chart below to obtain the typical desired levels of quantitation. This is a typical schematic of the calibration; however the standard can

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concentration, calibration levels and calculated concentrations may be different, as long as the calibration rules in 10.3.1 and 10.3.11 are followed. See Appendix IV and V for the table of analytes. If the actual standard amount trapped is greater than 5% from the programmed volume, the actual volume trapped is documented and used in calculations.

Normal Level Reporting Limit (calculation based on 200ml sample analysis) Volumes (mls) taken from the working stock canisters to prepare the calibration series concentrations listed (ppby/y)

			COLLECT	manon	s natu	(ppuv/v	)		
50 ppb v/v	240	160	80	40	20	-	-	-	-
can									
1 ppb v/v can	-	-	-	-	-	160	80	40	20
Calculated concentratio	60	40	20	10	5	0.8	0.4	0.2	0.1
n ppb v/v									

Low Level Reporting Limit (calculation based on 500ml sample analysis) Volumes (mls) taken from stock canisters to prepare the calibration series concentrations listed (ppbv/v)

2 ppb v/v can	250	125	50	-	-	-	-
0.1 ppb v/v can	-	-	-	500	200	100	50
Calculated	1.0	0.5	0.2	0.1	0.04	0.02	0.01
concentration ppb v/v							

10.3.3. See Tables 1, 2 and 3 for suggested quantitation ions.

- 10.3.4. A calibration curve is valid for all target analytes if the relative standard deviation (RSD) of the relative response factors is  $\leq$ 30% for each target analyte, with the following allowance: up to two target analytes may have an RSD  $\leq$  40%.
- 10.3.5. The internal standard area response at each calibration level must be within 40% of the mean area response over the initial calibration range for each internal standard.
- 10.3.6. The retention time (RT) shift for each of the internal standards at each calibration level must be within 20 seconds of the retention time of the mean calibration for each internal standard.
- 10.3.7. Each analyte at each level must be within 0.06 RRT units of the mean RRT.
- 10.3.8. If the curve is acceptable and there is time remaining in the 24-hour tune, blanks, LCSs and samples may be analyzed.
- 10.3.9. The concentrations in the samples, LCSs and blanks are calculated using the response factors from the initial calibration curve.

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- 10.3.10.Linear or quadratic curve fits may be used. Use of 1/Concentration<sup>2</sup> weighting may be used to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. The correlation coefficient (coefficient of determination for non-linear curves,  $r^2$ ) must be  $\geq 0.990$ .
- 10.3.11.Analyst may elect to drop points from the calibration to improve subsequent quantitation. The rules for dropping points are:
  - May drop points below the RL as long as there is a point remaining at or below the RL.
  - May drop high points, decreasing linear range.
  - May <u>NOT</u> drop a point between points.

For more guidance see "Selection of Calibration Points" Policy P-T-001, current revision.

Rules for curve use/acceptability:

- The Y intercept must be below the RL.
- The r^2 value obtained from Target must be  $\geq 0.990$ .
- At least 5 points must be used for average or linear curve.
- At least 6 points must be used for a quadratic curve.
- Do not include the origin or force the curve through the origin.
- For quadratic curves, the tangent line to the slope of the curve must be continuous and positive (i.e. no parabola's or breaks in the curve).
- 10.4. Initial Calibration Verification (ICV)
  - 10.4.1. The ICV is a second source standard containing the TO-14 list compounds at 10 ppb (Table 1) and is analyzed after the initial calibration and before any samples are analyzed. For each analyte, a percent recovery (%R) is calculated using the response factor from the initial calibration.
  - 10.4.2. A working standard from an independently prepared stock containing all analytes is also analyzed as the ICV for analytes not included in the TO-14 list.
  - 10.4.3. The ICV is valid for all analytes if the %R is between 65% and 135% for each TO-14 list analyte in the ICV. Benzyl chloride ICV acceptance criteria is 20-180%.
- 10.5. Daily Calibration Verification
  - 10.5.1. A mid-level standard is analyzed following the daily tune check (section 10.2) as the calibration verification standard. Typically, this is 80 mL of

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the 25-ppb (v/v) can. For all target analytes, a percent difference (%D) or percent drift is calculated using the response from the calibration verification standard and compared to the current initial calibration curve.

- 10.5.2. A calibration verification standard is acceptable if the %D or % drift is ≤30% for all target analytes. However, data may be reported from a calibration verification standard with up to four target analytes with % drift up to ≤40%. These analytes must be clearly noted in the data report.
  - 10.5.2.1. For samples from New Jersey, the target analytes with drift >30% must be noted in the narrative.
- 10.5.3. The daily calibration verification may also serve as the LCS as long as it meets the criteria of both the LCS and the daily calibration verification (section 9).
- 10.5.4. If the calibration verification standard does not meet the above criteria, corrective action must be taken and/or a new initial calibration performed unless project specific analytes or client specified QC criteria are met. Corrective action may include a reanalysis of the calibration verification standard. If reanalysis of the standard does not meet acceptance criteria, further corrective action may include performing instrument maintenance, or preparation of a new working calibration verification standard. Either of these corrective actions must be followed by successful analysis of the calibration verification standard and reanalysis of any affected samples. If these corrective actions do not result in an acceptable calibration verification verification a new initial calibration must be performed.

#### 11. Procedure

11.1. Canister Preparation

11.1.1. Use the following guidelines when checking a sample upon receipt:

• Tedlar bags are inspected to ensure that the valve is closed and the bags are not leaking. Bags must be analyzed or transferred to a can within 72 hours of collection. Tedlar bags are analyzed directly from the bag or transferred to an evacuated SUMMA can within 72 hours of sampling. If the entire bag is transferred to a can, the bag is attached to a short line and the entire contents transferred to a 1-L or a 6-L evacuated can. If only a portion of the bag is to be transferred, a measured aliquot of the bag is transferred via syringe through a septa attached to the top of a 1-L or a 6-L humidified can. After transfer, the can is then pressurized to a positive pressure and the pressure is recorded. The lab default is to analyze tedlar bags at a 20x dilution. Based on a default dilution factor

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at the bench, the RLs and MDLs will be 20 times higher for tedlar bag analysis. (If a client wants lower RLs than 20x the standard this will need to be communicated to the lab via special instructions.) If a client wants RLs lower than 20x and the client is supplying the tedlar bags, the PM should request that the client send an unused bag to be logged in and run along with their samples as a media blank check. If a client wants RLs lower than 20x and STL Knoxville is supplying the tedlar bags, the PM should have sample receiving set aside and log in a tedlar bag from the same lot as a media check.

- 1-L cans received between -10" Hg vacuum and a positive pressure are ready for a 20 mL analysis. If more volume is expected to be analyzed, the can will have to be pressurized in order to obtain more volume from the can.
- 6-liter cans received between -10" Hg vacuum and a positive pressure are ready for 200 mL analysis
- If any can is near zero psi (approximately -1" Hg to 1 psi), nonconformance the can as "suspect improper sampling event" and proceed with analysis. The project manager will discuss this with the client.
- Cans received -10" Hg or more vacuum should be pressurized to no more than approximately 5 psi.
- Cans below -20" Hg: non-conformance the can as "suspect improper volume sampled" and proceed with pressurizing the can to no more than approximately 5 psi for analysis. The project manager will discuss with the client that there may have not been enough sample collected for analysis and inform the analyst if sample analysis is to proceed or if the test is to be cancelled.
- Cans received at high vacuum (near -28" Hg or lower) should be inspected to determine if it is a trip blank. If the can is a trip blank, pressurize the can and use a dilution factor of one in analysis. If the sample cannot be determined to be a trip blank, non-conformance the can as "suspect improper sampling event" and proceed with pressurizing the can for analysis. The project manager will discuss with the client that there was not enough sample collected for analysis, and inform the analyst if sample analysis is to proceed or if the test is to be cancelled.
- 11.1.2. Measure the initial and final pressure/vacuum of the canister using an NIST traceable, certified vacuum or pressure gauge.
- 11.1.3. The barometric pressure, initial pressure/vacuum and final pressure/vacuum are recorded in a laboratory notebook, and used to calculate the dilution factor caused by pressurizing the can to working conditions

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- 11.1.4. The canister is allowed to equilibrate for approximately one hour. If the canister was pressurized to greater than 15 psig, pressure should be released from the canister to bring the pressure below 15 psig. For autosampler volumes scheduled to be below 50 mL, the can pressure must be reduced to below 7 psig to more accurately measure the volume injected.
- 11.1.5. This canister may be further diluted, if necessary, by the dilution methods discussed in sections 11.3, 11.5 and 11.6.
- 11.2. Following a successful initial or calibration verification and prior to analysis of actual samples, an acceptable system blank and LCS must be analyzed (see sections 9.2 and 9.3). Following successful system blank and LCS analysis, actual sample analysis may begin. The LCS and blank are analyzed every 24 hour tune or every 20 samples, whichever is more frequent.
  - 11.2.1. The desired sample size of each sample to be analyzed is determined by screening the cans according to SOP KNOX-MS-0010, current revision, Volatile Analyte Screening By Purge and Trap. The standard aliquot size is 200 mL for standard reporting limit work or 500 mL for low-level work. Sample volume injected can range from 10 mL to 1000 mL. For sample volumes below 50 mL, the can pressure must be reduced to below 7 psig to more accurately measure the volume injected. Volumes larger than 1000 mL can cause trap freeze-up when high humidity samples are trapped. If samples have been adequately pressurized with nitrogen, have been diluted, or only a small amount of sample collected in the can, then volumes larger than 1000 mL may be trapped, and the internal standards and surrogates monitored closely for breakthrough or freeze-up problems.
  - 11.2.2. The pressure of each sample canister is checked. If the pressure is above 15 psig, the excess pressure is vented.
  - 11.2.3. Each sample name, volume (aliquot), method, and autosampler position are typed into the Entech sequence table.
  - 11.2.4. If necessary, the automated flush function is used to sweep each autosampler line in the name list with helium.
  - 11.2.5. The cans are then securely tightened onto the autosampler with the canister valves closed.
  - 11.2.6. An automated leak check is run on each position. A hard copy of the leak check results is included with the daily calibration package. This is only a

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check to ensure that the valves are in proper working order and that the cans are attached securely to the autosampler.

- 11.2.7. If all positions pass the leak check, the canister valves are opened.
- 11.2.8. A name list similar to the Entech name list (section 11.2.3) is typed into the GC/MS sequence table. The sample volume programmed, the can number and a notation for in-can or serial dilution are noted in the analytical run log.
  - 11.2.8.1. If the actual sample amount trapped is greater than 5% from the programmed volume, the actual volume trapped is documented and used in calculating the results.
- 11.2.9. The Entech autosampler is started and the GC/MS acquisition program is started. (Note: The scan and GC parameters are controlled by the GC/MS method.)
- 11.2.10. 20 ml of the surrogate/internal standard is trapped on the Entech concentrator prior to sample introduction.
- 11.2.11. The analysis proceeds automatically for each name in the Entech autosampler program.
- 11.2.12. The internal standards and surrogates must pass all the criteria specified in section 9.1.
- 11.3. Autosampler Dilutions
  - 11.3.1. Volumes of 10 to 1000 mL may be analyzed by the autosampler (see section 11.2.1). The standard aliquot is 200 mL for standard reporting limit work and 500 mL for low-level work.
  - 11.3.2. If an analyte found in the sample is over the curve by less than a factor of twenty (based on 200 ml nominal volume) or fifty (based on 500 ml nominal volume), then the aliquot size of the sample may be reduced to a volume as low as 10 mL. This dilution factor is multiplied with all other dilution factors for this sample to obtain the final dilution factor.
  - 11.3.3. If a dilution is performed to bring one or more analytes within the calibration range, the analyte having the highest concentration should not be diluted to less than 20% of the calibration range unless there are significant amounts of non-target compounds present.

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- 11.3.4. If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non target peaks are less than five times the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution (up to the nominal volume). This requirement is approximate and subject to analyst judgment.
- 11.3.5. Only the most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.
- 11.3.6. The internal standards and surrogates must pass all the criteria specified in section 9.1.
- 11.4. Water addition
  - 11.4.1. The analyst should be aware that humidity plays an important role in the recovery of certain target compounds, particularly polar compounds, and should be prepared to add humidity to canisters where appropriate. The addition of water helps to stabilize the behavior of these compounds, which might otherwise interact with the interior surface of the summa canister or with the stainless-steel lines of the sample manifold.
  - 11.4.2. Since it is not practical to know the relative humidity of all canisters received at the laboratory, the analyst should assume that canisters are received at approximately 80 percent relative humidity. When making canister dilutions (see Sections 11.5, and 11.6), the analyst should attempt to preserve the relative humidity of canisters at a level that will minimize recovery loss due to low canister relative humidity.
  - 11.4.3. Under normal laboratory conditions, a 6 liter summa canister at ambient pressure will have a relative humidity of 100 percent if approximately 100 uL of water is in the canister.
    - 11.4.3.1. The minimum relative humidity at which canisters containing polar analytes can be analyzed before polar target recovery is negatively affected is approximately 20 30 percent.
    - 11.4.3.2. The minimum relative humidity at which canisters containing nonpolar analytes can be analyzed before nonpolar target recovery is negatively affected is approximately 10 percent.

#### 11.5. Serial Dilution

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- 11.5.1. High-level samples, for example, are those containing ppm levels of volatile organic compounds.
- 11.5.2. The original sample canister must have a positive pressure. If the pressure is less than 0 psig, then proceed to Section 11.1.
- 11.5.3. A septum cap is attached to the sample canister and a gas-tight syringe is purged with UHP nitrogen. A septum cap is attached to a clean evacuated 6-liter canister (the dilution canister).
- 11.5.4. 40 uL of deionized water is added to the canister through the septum of the evacuated can (See Section 11.4 for guidance on addition of water).
- 11.5.5. The syringe is inserted into the septum cap of the canister containing the sample and the canister valve is opened. The syringe is purged twice with sample and vented. The desired volume is then withdrawn and transferred into the dilution canister. The dilution canister is then pressurized using UHP nitrogen.
- 11.5.6. The final pressure is measured in the serial dilution canister using a NIST traceable, certified gauge.
- 11.5.7. If the canister was pressurized to greater than 15 psig, pressure should be released from the canister to bring the pressure below 15 psig.
- 11.5.8. The barometric pressure, the aliquot volume, final canister pressure and canister serial number are recorded in a laboratory notebook. The serial dilution factor is calculated.
- 11.5.9. If a high level dilution is performed to bring one or more analytes within the curve, the analyte having the highest concentration should not be diluted to less than 20% of the upper calibration range, unless there are significant amounts of non-target compounds present. It is imperative that high levels of target and non-target analytes not contaminate the analytical system.
- 11.5.10. This serial dilution canister may be further diluted, if necessary, by another serial dilution or in-can dilution (see section 11.6) or on the autosampler (see section 11.3). The final dilution factor is the product of all the dilution factors for the sample.
- 11.6. In-canister Dilutions

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- 11.6.1. If an analyte found or suspected to be in the sample is over the calibration range, to a level that an autosampler dilution would be insufficient, an incanister dilution may be performed.
- 11.6.2. The canister vacuum/pressure is checked. If the can is under vacuum, then record the vacuum reading and proceed to section 11.6.3. If the canister is under pressure, then the can is bled to ambient pressure, then proceed to section 11.6.3.
- 11.6.3. The canister is pressurized to the desired pressure. The pressure should be no more than 40 psig.
- 11.6.4. The final pressure is measured using an NIST traceable, certified gauge.
- 11.6.5. If the canister was pressurized to greater than 15 psig, pressure should be released from the canister to bring the pressure below 15 psig.
- 11.6.6. The barometric pressure and the final pressures are recorded in a laboratory notebook and the in-can dilution factor is calculated.
- 11.6.7. If an in-canister dilution is performed to bring one or more analytes within the curve, the analyte having the highest concentration should not be diluted to less than 20% of the upper calibration range, unless there are significant amounts of non-target compounds present. Care should be taken to avoid over-dilution for in-canister dilutions since the original sample is affected.
- 11.6.8. This in-can dilution canister may be further diluted, if necessary, by another in-can dilution or a serial dilution (see section 11.6) or on the autosampler (see section 11.3). This dilution factor is multiplied with all other dilution factors for this sample to obtain the final dilution factor.
- 11.7. Major Maintenance
  - 11.7.1. A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing moisture or Tenax traps.
- 11.8. Minor Maintenance
  - 11.8.1. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments (instrument contains two filaments under vacuum), replacing valves or rotors,

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change/refill the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps or transfer lines.

- 11.9. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager and QA Manager. If contractually required, the client shall be notified.
- 11.10. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

#### 12. Data Analysis and Calculations

- 12.1. Refer to Figure 2 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.
- 12.2. Tentatively Identified Compounds (TICs): Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the STL Knoxville SOP KNOX-MS-0014, current revision, "Determination of Tentatively Identified Compounds (TICs)"
- 12.3. Calculation legend:

А	=	amount of neat compound, uL
CB	=	concentration in SGDB, ug/mL
CC	=	concentration in canister, ppb v/v
CS	=	concentration in mix, ug/uL
Cx	=	the value determined by vendor certification analyses is
		used in the following calculations, ppb v/v (300 ppb nominal)
d	=	density of neat compound, g/mL
DF	=	dilution factor, unitless
FV	=	final volume in a pressurized canister, liters
GC	=	gas constant at 25°C and standard pressure, 24.45 nL/n mole
MW	=	molecular weight, ng/n mole
PB	=	barometric pressure
PF	=	final pressure, units specified
PI	=	initial pressure, units specified
PT	=	transfer pressure, units specified
Px	=	pressure in $X =$ inches, psia or mmHg
ΤK	=	temperature in Kelvin

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TV =	transfer volume, liters or uL
Vbottle=	volume of static gas dilution bottle, mL
Vmix =	volume of mix, µL

#### 12.4. Calculations:

12.4.1. Final Canister Volume

$$FV = \frac{Canister \ size \ (L) \ x \ P_F(mm \ Hg \ Abs)}{P_B(mm \ Hg \ Abs)}$$

Pmm Hg = P inches x 25.4P inches = Ppsi \* 2.036 $Pmm Hg = P_{psi} x 51.7149$ 

12.5. Polar stock:

12.5.1. 
$$CS \ \mu g \ / \ uL = \frac{A^* d^* 1000}{V_{mix}}$$

12.5.2. 
$$CB \ \mu g \ / \ mL = \frac{CS * TV, \ \mu L}{V_{bottle}}$$

12.6. Polar concentration in target dilution standard

$$CC, ppb v / v = \frac{TV, \ \mu L * CB x GC}{MW * FV}$$

#### 12.7. Stock standards in SGDB

$$CB_{LIQUID}, \, \mu g/mL = \frac{\# \, \mu L * d * 1000 * 0.8829}{V_{bottle}}$$

where 0.8829 is a temperature correction factor (from 21  $^{\circ}$ C to 60 $^{\circ}$ C)—only used if the SGDB standard is in the oven at 60 C; = 294  $^{\circ}$ K / 333  $^{\circ}$ K.

12.7.1. Liquid formula

12.7.2. Solid formula

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$$CB_{SOLID}$$
,  $\mu g/mL = \frac{\# mg * 1000 * 0.8829}{V_{bottle}}$ 

where 0.8829 is a temperature correction factor (from 21 °C to 60°C)—only used if the SGDB standard is in the oven at 60 C; = 294 °K / 333 °K.

12.8. Concentration of standards in primary target standard made from SGDB

$$CC, ppb v/v = \frac{TV, mL * CB \times 1000 * GC}{FV * MW}$$

12.9. Concentration of Cylinder Standards: Concentration of Analytes in Primary Target Standard

$$CC, ppb v / v = \frac{(P_T - P_I, psi)(Cx)}{(P_F, psi + PB, psi)}$$

12.10. Target Dilution Standard (CC is the concentration of the Primary Target Standard).

$$CC, ppb v / v = \frac{(P_T - P_I)(CC)}{(P_F + PB, psi)}$$

12.11. Dilution Factors of original sample canisters

12.11.1. In Can Dilution Factor

$$DF = \frac{P_{f(mmAbs)}}{P_{i(mmAbs)}}$$

12.11.2.Serial Dilution Factor

DF = FV/TV

12.11.3. Instrument Dilution Factor

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 $DF = \frac{Nominal Sample Volume}{Sample Volume Injected}$ 

12.12. Response Factor (RF)

$$RF = \frac{Ax * Cis}{Ais * Cx}$$

where:

Х	=	area of the characteristic ion for the target compound.
Ais	=	area of the characteristic ion for the internal standard.
Cx	=	amount of the target compound.
Cis	=	amount of the internal standard.

12.13. Average Response Factor (ARF)

$$ARF = \frac{RF_1 + RF_2 + \dots + RF_n}{n}$$

where:

n

= the number of calibration points

12.14. Standard deviation of the ARF:

$$S = \sqrt{\frac{\sum_{i}^{n} (ARF - RF_{n})^{2}}{n - 1}}$$

12.15. Relative standard deviation (RSD) of the ARF:

$$RSD = \frac{S}{ARF} * 100\%$$

12.16. Calibration Verification : Percent deviation (% D) of the daily RF values as compared with the initial ARF values:

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$$\% D = \frac{/RF - ARF/}{ARF} * 100\%$$

12.17. Laboratory Control Sample percent recovery (%R):

 $\% R = \frac{FoundAmount, ppb}{SpikeAmount, ppb} * 100\%$ 

12.18. Duplicate relative percent difference (RPD):

$$RPD = \frac{|A_1 - A_2|}{\overline{A}} \times 100\%$$

where:

$A_1$	=	amount determined in first analysis
$A_2$	=	amount determined in second analysis
А	=	average determination, $(A_1 + A_2)/2$

12.19. Calibration verification percent drift and difference from the initial calibration:

% Drift = 
$$\frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

 $C_{expected} = Known concentration in standard$ 

C<sub>found</sub> = Measured concentration using selected quantitation method

$$\% Difference = \frac{RF - RF}{\overline{RF}} \times 100$$

- $\overline{RF}$  = Average Analyte Response Factor from Initial Calibration RF = Measured Analyte Response Factor from Calibration Verification
- 12.19.1.Target analyte concentrations in samples are typically calculated using the average response factor from the initial calibration. Quantitation may also be determined using linear or second order curves at the analyst's discretion to improve the quantitation of target analytes.

12.19.1.1.Calculation of concentration using Average Response Factors

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$$C_{pv} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.19.1.2.Calculation of concentration using Linear fit

$$C_{pv} = A + B \frac{(R_x C_{is})}{R_{is}}$$

 $C_{pv}$  = Concentration, ppb (v/v)

 $R_x$  = Response for analyte (area of quantitation ion)

 $R_{is}$  = Response for internal standard (area of quantitation ion)

 $C_{is}$  = Concentration of internal standard

A = Intercept

B = Slope

The corresponding Target software calculation is as follows:

$$C_{pv} = C_{is}(b + \frac{1}{ml} \times \frac{R_x}{R_{is}})$$

b = Concentration Ratio Intercept

m1 = Inverse of Slope

12.19.1.3.Calculation of concentration using Quadratic fit

$$C_{pv} = A + B\left(\frac{R_xC_{is}}{R_{is}}\right) + C\left(\frac{R_xC_{is}}{R_{is}}\right)^2$$

$$C = Curvature$$

The corresponding Target software calculation is as follows:

$$Cpv = Cis\left(b+m1 \times \frac{Rx}{Ris} + m2 \times \left(\frac{Rx}{Ris}\right)^{2}\right)$$

m1 = First order coefficient

m2 = Curvature (Second order coefficient)

12.20. Sample Quantitation: The amount of target compound detected is determined using the average RF or calibration curve values from the initial calibration (not the continuing calibration):

Amount = 
$$Cpv * DF$$

12.21. Unit conversions

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Amount, 
$$\mu g/m^3 = \frac{Amount, ppb(v/v) * MW}{GC}$$

12.21.2.

Amount, ppm v/v = amount, 
$$\frac{\text{ppb}(v/v)}{1000}$$

- 12.22. Quantitation of Unknowns
  - 12.22.1.If required, nontarget peaks are reported with probable identifications as Tentatively Identified Compounds (TICs). These are quantitated using the nearest internal standard and assuming a response factor of 1; correction for dilution factor is also made. Search criteria are those in the STL Knoxville SOP KNOX-MS-0014, current revision, Tentatively Identified Compounds (TICs).

#### **13.** Method Performance

- 13.1. Method Detection Limit (MDL) An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP S-Q-003 current revision based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit. MDL summaries are stored on the local area network.
- 13.2. Initial Demonstration of Capability Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in STL Knoxville SOP KNOX-QA-0009. Recovery limits must be 70-130% and RSD must be less than or equal to 25%. Recovery limits for Methanol are 60-140% and RSD must be less than or equal to 30%.
- 13.3. Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and ongoing demonstrations of capability.

#### 14. Pollution Prevention

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14.1. All attempts will be made by laboratory personnel to minimize the use of solvents when performing this procedure.

## 15. Waste Management

- 15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 15.2. The following waste streams are produced when this method is carried out.
  - Expired solid and liquid standards are stored in metal closed-top containers.

## 16. References

- Compendium Method TO-14, "The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMATM Passivated Canister Sampler and Gas Chromatographic Analysis," U.S. EPA 600/4-89/017, June 1988.
- 16.2. Compendium Method TO-14A, "Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters With Subsequent Analysis by Gas Chromatography," U.S. EPA 625/R-96/010b, January 1999.
- 16.3. Compendium Method TO-15, "Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GCMS)", U.S. EPA 625/R-96/010b, January 1999.
- 16.4. STL Quality Management Plan (QMP), current revision.
- 16.5. STL Knoxville Laboratory Quality Manual (LQM), current revision.
- 16.6. Entech Instruments Inc. 7100 Operators Manual. Version 2.0 for the 7100 Preconcentrator and Accessories.

## 17. Miscellaneous

- 17.1. Other SOPs cross-referenced in this SOP: KNOX-SC-0001, "Canister Cleaning and Preparation", latest revision.
- 17.2. Modification from the referenced methods

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- 17.2.1. The TO-15 tune criteria were not used in this procedure. The tune criteria listed in TO-14 is tighter and thus was used in this procedure. This SOP also allows for 50 ng or less of BFB to verify tuning of the instrument.
- 17.2.2. The continuing calibration listed in this procedure allows up to 4 target analytes with a %D of  $\leq$  40%, with a narrative note (or data review checklist note for non-NJ DEP analysis) of those target analytes that are over 30% D, but  $\leq$  40% D.
- 17.2.3. This procedure uses purified nitrogen in place of zero humid air specified in the reference methods.
- 17.2.4. TO-14 requires that the RT shift for the internal standards at each calibration level must be within 20 seconds of the RT of the midlevel calibration for each internal standard. TO-15 specifies that the comparison is made to the mean RT over the initial calibration range for each internal standard. This SOP uses the TO-15 criteria.
- 17.2.5. Section 7.13 Method TO-15 states that the working standard may be stored for 30 days. This laboratory experience has allowed the standard expiration date to be 2 months with no significant degradation of the standards.
- 17.2.6. Surrogates are not required by the methods. This SOP adds surrogates to every sample to help monitor for matrix effects and method performance.
- 17.2.7. The TO-15 method states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument. See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.
- 17.2.8. EPA Method TO-14A specifies that the relative accuracy of the field sampler or sample delivery system must meet 90-110% for a standard at 8 ppb v/v. The laboratory Control Sample (LCS) summary data is evaluated against alternate acceptance criteria based on this laboratory procedure for method TO-14A. When TO-14A work is performed, this must be noted in the case narrative.
- 17.3. List of Appendices

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17.3.1. Appendix I: Target Analyte Tables

17.3.1.1.Table 1: Target Analytes - TO-14 and TO-15 Compounds

17.3.1.2. Table 2: Target Analytes - Other Nonpolar Compounds

17.3.1.3.Table 3: Target Analytes - Other Polar Compounds

17.3.2. Appendix II: Figures

17.3.2.1.Figure 1: BFB Tuning Criteria

17.3.2.2.Figure 2: Example of a Data Review Checklist

17.3.2.3.Figure 3: Flow Chart

17.3.3. Appendix III: Example Instrument Parameters

17.3.4. Appendix IV: Recommended Calibration Levels (normal reporting limits)

17.3.5. Appendix IV: Recommended Calibration Levels (low level reporting limits).

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CAS	METHOD TO-14	REPORTING	REPORTING	Low Level	MOLECULAR	SUGGESTED
NUMBER	COMPOUND	LIMIT	LIMIT	Reporting Limit	WEIGHT	ION
		(ppb, v/v) (i)	(ug/m <sup>3</sup> ) (i)	(ppb, v/v) (j)	(ng/n mole)	
75-71-8	Dichlorodifluoromethane (b)	0.2	0.99	0.020	120.9	85
76-14-2	1,2-Dichlorotetrafluoroethane (c)	0.2	1.40	0.020	170.9	135
74-87-3	Chloromethane	0.5	1.03	n/a	50.49	52
75-01-4	Vinyl Chloride	0.2	0.51	0.020	62.50	62
74-83-9	Bromomethane	0.2	0.78	0.060	94.94	94
75-00-3	Chloroethane	0.2	0.53	0.030	64.51	64
75-69-4	Trichlorofluoromethane (d)	0.2	1.12	0.030	137.4	101
75-35-4	1,1-Dichloroethene	0.2	0.79	0.090	96.94	96
76-13-1	1,1,2-Trichlorotrifluoroethane (e,f)	0.2	1.53	n/a	187.4	101
75-09-2	Methylene Chloride (f)	0.5	1.74	n/a	84.93	84
75-34-3	1,1-Dichloroethane	0.2	0.81	0.020	98.96	63
156-59-2	cis-1,2-Dichloroethene	0.2	0.79	0.050	96.94	96
67-66-3	Chloroform	0.2	0.98	0.020	119.4	83
71-55-6	1,1,1-Trichloroethane	0.2	1.09	0.020	133.4	97
56-23-5	Carbon Tetrachloride	0.2	1.26	0.020	153.8	117
71-43-2	Benzene	0.2	0.64	0.030	78.11	78
107-06-2	1,2-Dichloroethane	0.2	0.81	0.020	98.96	62
79-01-6	Trichloroethene	0.2	1.07	0.020	131.4	130
78-87-5	1,2-Dichloropropane	0.2	0.92	0.030	113.0	63
10061-01-5	cis-1,3-Dichloropropene	0.2	0.91	0.030	111.0	75
108-88-3	Toluene	0.2	0.75	0.10	92.14	91
10061-02-6	trans-1,3-Dichloropropene	0.2	0.91	0.050	111.0	75
79-00-5	1,1,2-Trichloroethane	0.2	1.09	0.040	133.4	97
127-18-4	Tetrachloroethene	0.2	1.36	0.020	165.8	129
106-93-4	1,2-Dibromoethane (EDB)	0.2	1.54	0.020	187.9	107
108-90-7	Chlorobenzene	0.2	0.92	0.020	112.6	112
100-41-4	Ethylbenzene	0.2	0.87	0.020	106.2	91
IT5-30-5	m/p-Xylene (g, h)	0.2	0.87	0.10	106.2	91
95-47-6	o-Xylene (h)	0.2	0.87	0.030	106.2	91
100-42-5	Styrene	0.2	0.85	0.020	104.2	104
79-34-5	1,1,2,2-Tetrachloroethane	0.2	1.37	0.020	167.8	83
108-67-8	1,3,5-Trimethylbenzene	0.2	0.98	0.020	120.2	120
95-63-6	1,2,4-Trimethylbenzene	0.2	0.98	n/a	120.2	105
541-73-1	1,3-Dichlorobenzene	0.2	1.2	n/a	147.0	146
106-46-7	1,4-Dichlorobenzene	0.2	1.2	n/a	147.0	146
100-44-7	Benzyl Chloride	0.4	2.07	n/a	126.6	91
95-50-1	1,2-Dichlorobenzene	0.2	1.2	n/a	147.0	146
120-82-1	1,2,4-Trichlorobenzene	1.0	7.42	n/a	181.4	180
87-68-3	Hexachlorobutadiene	1.0	10.67	n/a	260.8	225

Appendix I: Target Analyte Tables Table 1: Target Analytes – Method TO-14A Target Compounds

b) Freon 12

Π

c) Freon 114

d) Freon 11

e) Freon 113

f) This is a common laboratory solvent

g) m-xylene and p-xylene coelute

h) Total xylenes (CAS # 1330-20-7) is the sum of m/p-xylenes and o-xylene.

i) Normal reporting limits (RLs) based on 200 mL sample volume. The ug/m3 values are example RLs using the value listed in the table.

j) Low level reporting limits based on 500 mL sample volume

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### Table 2: Target Analytes - Other Nonpolar Compounds

CAS NUMBER	OTHER NON-POLAR COMPOUNDS	REPORTING LIMITS (ppb, v/v) (i)	REPORTING LIMIT (ug/m <sup>3</sup> ) (i)	Low Level Reporting Limit (ppb, v/v) (j)	MOLECULAR WEIGHT (ng/n mole)	SUGGESTED ION
75-45-6	Chlorodifluoromethane (b)	0.2	0.71	n/a	86.47	51
106-97-8	n-Butane	0.4	0.95	n/a	58.12	43
106-99-0	1,3-Butadiene	0.4	0.88	0.030	54.09	54
109-66-0	Pentane	1.0	2.95	n/a	72.15	57
75-15-0	Carbon Disulfide	0.5	1.56	n/a	76.14	76
107-05-1	3-Chloropropene	0.2	0.63	0.060	76.52	39
156-60-5	trans-1,2-Dichloroethene	0.2	0.79	0.10	96.94	96
110-54-3	n-Hexane	0.5	1.76	0.10	86.18	56
110-82-7	Cyclohexane	0.5	1.72	0.040	84.16	69
540-84-1	2,2,4-Trimethylpentane	0.5	2.34	0.020	114.2	57
142-82-5	n-Heptane	0.5	2.05	0.020	100.2	43
74-95-3	Dibromomethane	0.4	2.84	n/a	173.8	93
75-27-4	Bromodichloromethane	0.2	1.34	0.020	163.8	83
111-65-9	n-Octane	0.4	1.87	n/a	114.2	85
124-48-1	Dibromochloromethane	0.2	1.7	0.020	208.3	129
111-84-2	Nonane	0.5	2.62	n/a	128.3	57
75-25-2	Bromoform	0.2	2.07	0.020	252.7	173
98-82-8	Cumene	0.4	1.97	n/a	120.2	105
103-65-1	n-Propylbenzene	0.4	1.97	n/a	120.2	120
95-49-8	2-chlorotoluene	0.4	2.07	n/a	126.6	126
622-96-8	4-ethyltoluene	0.4	1.97	0.020	120.2	105
124-18-5	Decane	1.0	5.82	n/a	142.3	57
98-83-9	alpha-Methylstyrene	0.4	1.93	n/a	118.2	118
135-98-8	sec-butylbenzene	0.4	2.20	n/a	134.2	105
104-51-8	n-Butylbenzene	0.4	2.20	n/a	134.2	91
1120-21-4	n-Undecane	1.0	6.39	n/a	156.3	57
112-40-3	n-Dodecane	1.0	6.97	n/a	170.3	57
91-20-3	Naphthalene	0.5	2.62	n/a	128.2	128

b) Freon 22

i) Normal reporting limits (RLs) based on 200 mL sample volume. The ug/m3 values are example RLs using the value listed in the table.

j) Low level reporting limits based on 500 mL sample volume.

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CAS NUMBER	OTHER POLAR COMPOUNDS	REPORTING LIMITS	REPORTING LIMIT	Low Level Reporting Limit	MOLECULAR WEIGHT	SUGGESTED ION
		(ppb, v/v) (i)	(ug/m <sup>3</sup> ) (i)	(ppb, v/v) (j)	(ng/n mole)	
67-56-1	Methanol	10	13.1	n/a	32.04	31
593-60-2	Vinyl Bromide	0.2	0.88	0.050	107.0	106
60-29-7	Ethyl Ether	2.0	6.06	n/a	74.12	31
67-64-1	Acetone	5.0	11.88	n/a	58.08	58
75-65-0	Tert-Butanol	2.0	6.06	n/a	74.12	59
107-13-1	Acrylonitrile	2.0	4.34	n/a	53.06	53
108-05-4	Vinyl Acetate	1.0	3.52	n/a	86.09	43
78-93-3	2-Butanone	1.0	2.95	n/a	72.11	72
71-36-3	1-Butanol	2.0	6.06	n/a	74.12	31
108-10-1	4-Methyl-2-Pentanone	0.5	2.05	n/a	100.2	43
591-78-6	2-Hexanone	0.5	2.05	n/a	100.2	58
1634-04-4	Methyl-t-Butyl ether	1.0	3.61	0.050	88.15	73
107-02-8	Acrolein	0.8	1.83	n/a	56.06	56
75-05-8	Acetonitrile	1.0	1.68	n/a	41.05	40
96-18-4	1,2,3-Trichloropropane	0.5	3.01	n/a	147.4	110

### **Table 3: Target Analytes – Other Polar Compounds**

- i) Normal reporting limits (RLs) based on 200 mL sample volume. The ug/m3 values are example RLs using the value listed in the table.
- j) Low level reporting limits based on 500 mL sample volume

### **Appendix II: Figures**

#### Figure 1: BFB Tuning Criteria

Mass	Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 to 9% of mass 174
176	95% to 101% of mass 174
177	5 to 9 % of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though m/z 174 may be over 100 % of m/z 95.

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## Figure 2: Example Data Review Checklist

Analysis Date: Instrument::				ICAL Ba	tch/Sear	1 Name	:		Scanned	7
Review Items				N/A	Yes	No	IEN	o, why is data reportable?		2d
1. Did BFB me	et tune criteria?			IN/A	165	INO	IIN	o, why is data reportable?		
1. DIG DI D IIIO	or tano ornoria.									
2. Were all star	ndards injected wit	hin 24 hr of BFB:	?							
3. Was date/tim	ne of analysis verif	fied between anal	vsis header	-						
and logbook		inou o como cinu	J010 11000001							
4. Is low level a	std at or <rl :<="" and="" td=""><td>are the remaining</td><td>points</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></rl>	are the remaining	points							
consecutive?	)	-	-							
5. Were at least	t 5 levels of each o	compound analyz	ed?							
	all target analytes: with RSD <u>&lt;</u> 40%)	$s \leq 30\%$ ? (with up	p to 2							
	ks been auto ident	ified? If not, list:								
8 If oursian mar	re used, is correlat	ion coefficient 🗸	1 9902							
	nsecutive points u			-	<u> </u>					
	ecutive points for l		्या १७०, साथ श							
	quadratic: is a tar		he curve							
	tive or negative ar									
	quadratic: origin 1		forced?							
	tercept less than t									
	IS ±20 sec avg. R'									
	h IS + 40% avg. ar									
15. Each analyte	e ± 0.06 RRT of a	vg. RRT?								
	tegrations were pe		/ clearly				Reas	sons: 1)Corrected split peak; 2)Unresolve	d peak;	
	itialed, dated and i		, v				3)ta:	iling; 4)RT shift; 5)wrong peak selected; 6	ó)other	
17. Have alterna	te hits/manual inte	egrations been ve	rified as							
correct and a	are correct RFs list	ed in ICAL summ	nary?							
18. Was ICAL s and files?	ummary form proc	cessed using corr	ect methods							
19. Are the ICA summary?	L start and end da	tes/times correct	on ICAL							
	r checked on isom	eric nairs?								
	fluoromethane / 1,		uoroethane							
	, uoromethane / 1,1									
<ul> <li>vinyl aceta</li> </ul>		<i>′</i>								
<ul> <li>cis- and tra</li> </ul>										
	ene / m/p -xylene /	/o-xylene			İ					
	luene/1,3,5-trimet		4-							
trimethylb		•								
• 1,3-, 1,4-	, and 1,2-dichloro	benzene								
21. Is the second	l source analysis o 135% R; 20-180%	f a reference stan								
	ere not met, was a									
supervisor, a	nd copy included i	in folder?								
	AL folder contain		the following							
order: Data r	review checklist, a	ι complete runlog	, Entech	1						
	info, ICAL summ									
	matograms, manu	al integrations] in	n increasing							
amount orde	r.									
					-		1.5			
Analyst:			Date:					riewer : Dat	e:	
Comments:					C0	mmei	uts:			

#### STL Knoxville GC/MS Air Initial Calibration Data Review / Narrative Checklist Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 9

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## Figure 2: Example Data Review Checklist (continued)

#### STL Knoxville GC/MS Air Continuing Calibration Review / Narrative Checklist Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 9

Review Items       N/A       Yes       No       If No, why is data reportable?         1. Did BFB meet tune criteria?       Image: Comparison of the standards injected within 24 hr of BFB?       Image: Comparison of the standards injected within 24 hr of BFB?       Image: Comparison of the standards injected within 24 hr of BFB?         3. Have the Entech position no. & vol. been verified with run log & sample vol. corrected if actual amount differs >5%?       Image: Comparison of the standards injected with run log & sample vol. corrected if actual amount differs >5%?       Image: Comparison of the standards injected with run log & sample vol. correct ICAL?         4. Was date/time of analysis verified between analysis header and logbook as correct?       Image: Compared to the correct ICAL?       Image: Compared to the correct ICAL?         5. Was the CCAL compared to the correct ICAL?       Image: Compared to the correct ICAL?       Image: Compared to the correct ICAL?         6. Is the %D = 30% for all target analytes? Up to 4 analytes allowed over 30% but = 40% D (Narrative req'd.).       Image: Compared to the correct ICAL?       Image: Compared to the correct ICAL?         7. Have all peaks been auto identified? If not, list:       Image: Compared to the correct ICAL?       Image: Compared to the standard and reason given?       Image: Compared to the correct ICAL?         8. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?       Image: Compared to the correct of the standards and reason given?       Image: Compared to the correct RFs listed in CCAL summary?	Scanned 7
1. Did BFB meet tune criteria?	
2. Were all standards injected within 24 hr of BFE?	
3. Have the Entech position no. & vol. been verified with run log & sample vol. corrected if actual amount differs >5%?         4. Was date/time of analysis verified between analysis header and logbook as correct?         5. Was the CCAL compared to the correct ICAL?         6. Is the %D = 30% for all target analytes? Up to 4 analytes allowed over 30% but = 40% D (Narrative req'd.).         7. Have all peaks been auto identified? If not, list:         8. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?         9. Have alternate hits/manual integrations been verified as	
4. Was date/time of analysis verified between analysis header and logbook as correct?	
6. Is the %D = 30% for all target analytes? Up to 4 analytes allowed over 30% but = 40% D (Narrative req'd.).	
allowed over 30% but = 40% D (Narrative req'd.).         7. Have all peaks been auto identified? If not, list:         8. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?         9. Have alternate hits/manual integrations been verified as	
8. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?       Reasons: 1)Corrected split peak; 2)Unresolved performed, are they clearly 3)tailing; 4)RT shift; 5)wrong peak selected; 6)oth         9. Have alternate hits/manual integrations been verified as       Image: selected; 6)oth	
identified, initialed, dated and reason given? 3)tailing; 4)RT shift; 5)wrong peak selected; 6)oth 9. Have alternate hits/manual integrations been verified as	
10. Is the first IS documented correctly on the log?	
11. Is the ICAL date & time on the CCAL correct?	
12. Elution order checked on isomeric pairs? • dichlorodifluoromethane / 1,2-dichlorotetrafluoroethane	
trichlorofluoromethane / 1, 1, 2-trichlorotrifluoroethane	
vinyl acetate / hexane	
cis- and trans- isomers	
ethyl benzene / m/p -xylene / o-xylene	
• 4-ethyl toluene/1,3,5-trimethylbenzene/1,2,4- trimethylbenzene	
• 1,3-, 1,4-, and 1,2-dichlorobenzene	
13. Did the LCS meet criteria (nonpolar target analytes 70- 130%, with up to 2 nonpolars 60-140%; polar target analytes 60-140%, with up to 2 polars 45-155%)?       ? [Ics6] LCS analyte(s) flagged as being outside control but SOP allows 2 polars and 2 nonpolars outside 70-1309	
14. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?	
15. Does the CCAL folder contain complete data in the following order: data review checklist, a complete runlog, Entech report, tune pass/fail page, m/z list, tune chromatogram, Target CCAL summary, Quan report, chromatogram, manual integrations and leak check report.	
Analyst: Date: 2nd Level Reviewer : Date:	
Comments: Comments:	

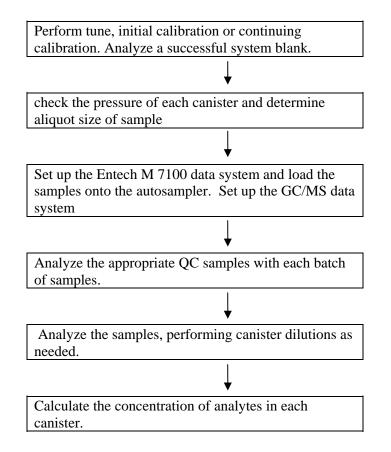
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## Figure 2: Example Data Review Checklist (continued)

Instrument:				
Scanned File:				
eview Items				
. Tune / Continuing Calibration Were all samples injected within 24 hr of BFB?	N/A	Yes	No	Why is data reportable?
Has a Continuing Calibration Checklist been completed for each				
analytical batch?				
Was the correct ICAL used for quantitation?				
CLIENT SAMPLE AND QC SAMPLE Results Were all special project requirements met?	N/A	Yes	No	Why is data reportable?
Were dilution factors/can prep information verified?				
Have the can number & lab ID been verified between the analysis log				
& sample prep log?				
Were samples received in cans?				? [Tedlar1] Samples rec'd on (date) in Tedlar bags & ana by
				TO-14 (TO-15) within 72 hours from sampling. ? [Tedlar2] Samples rec'd on (date) in Tedlar bags & transferred
				into Summa canisters within 72 hours.
Sample analyses done within analytical holding time (HT)?				// [ht2] Client requested analysis after HT expired.
no, list samples:				2 Other:
Are surrogates and internal standards within QC limits? (70-130% R			-	/ [Sur1] DUP surr. %R demonstrated same effect.
for surr.; 60-140%R from CCAL for IS)				<ul> <li>[Sur2] Reanalysis demonstrated same effect.</li> </ul>
no, list samples/reason (e.g., <b>sur1</b> ):				? [sur5] At client's request, data was flagged as estimated &
imple Reason Sample Reason				released without further investigation.*
				2 [is1] Per client, reanalysis was not performed *
				<ul> <li>? [is2] Reanalysis confirmed a matrix effect.</li> <li>? Obvious matrix effect</li> </ul>
Were all positive results and false negatives on quan report verified to				7 COVIOLS MALIA CITOCE
be correct in LIMS?				
For dilutions, is highest concentration hit $\geq$ 20% cal range and not				? [elev1] Elevated RL for (ANALYTE) due to sample matrix.
above calibration range?				interferences.
ist samples and reason (e.g., elev1): ample Reason Sample Reason				P [elev3] Elevated RLs for all analytes due to difficult sample matrix.
				? [elev4] Elevated RLs based on screening
				? [elev5] Elevated RLs for all analytes due to presence of non-
				target compounds.
If manual integrations were performed, are they clearly identified, initialed, dated and reason given?				Reasons: 1)Corrected split peak; 2)Unresolved peak; 3)tailing; 4)RT shift; 5)wrong peak selected; 6)other
). Have alternate hits/manual integrations been verified as correct?				fill billing by whole pour boloood, sjouloi
. Preparation QC				
System blank run every 24 hours prior to samples?				
System blank surrogate recoveries within QC limits (70-				? [mb1] All sample surrogates OK and there is no analyte
130% R) ? Are all analytes present in the system blank < RL?				>RL in samples associated with blank.* / [mb3] No analyte > RL in associated samples.*
If no, list blank ID:				? [mb4] Sample results > 20x higher than blank.
DUP done per 20 samples and are all RPDs within limits? (for target				
analytes >5x RL, <25% RPD; no criteria for methanol and n-butanol)				
If no, list DUP ID: Are all LCS analytes on final report within limits?				? [LCS6] Flagged out but within SOP limits.
Are an Eost analyses on thrat report within minus:				LCS ID:
. Other				
Final report acceptable? (Results correct, RLs calculated correctly,				
units correct, surrogate %R correct, appropriate flags used, dilution factor correct, analysis dates correct.)				
Are all nonconformances documented appropriately and copy included				
with deliverable?				
Were the standards scanned properly?				
Was a narrative prepared and all deviations noted?			<u> </u>	
TO14A Autotext included in narrative (for TO14A samples only). All target analytes on c.cal >30%D but <40%D noted in the narrative?				
All target analytes on c.cal >30%D but <40%D noted in the narrative?				following analytes:
nalyst: Date:		2 <sup>nd</sup>	Level l	Reviewer: Date:

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#### **Figure 3: Flow Chart**



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#### **Appendix III: Example Instrument parameters**

TOPLEVEL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\T014.M

Method Sections To Run:

Save Copy of Method With Data Pre-Run Cmd/Macro = }

(X) Data Acquisition
 () Data Analysis
 () Post-Run Cmd/Macro =

Method Comments: TO14 METHOD USING HP-DB-5 60M X 0.32MM X 1.0 FILM THICKNESS

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS Sample Inlet: GC Injection Source: Manual Injection Location: Front Mass Spectrometer: Enabled HP6890 GC METHOD OVEN Initial temp: 35 'C (On) Initial time: 5.00 min Ramps: # Rate Final temp Final time 1 6.00 65 0.00 2 12.00 155 0.00 3 25.00 220 7.00 4 0.0(Off) Post temp: 35 'C Post time: 0.00 min Run time: 27.10 min Maximum temp: 230 'C Equilibration time: 0.00 min FRONT INLET (UNKNOWN) BACK INLET () ONT INLET (UNKNOWN) Mode: Split Initial temp: 200 'C (On) Pressure: 7.89 psi (On) Split ratio: 2:1 Split flow: 3.0 mL/min Total flow: 7.3 mL/min Gas saver: Off Gas type: Helium COLUMN 2 (not installed) COLUMN 1 LUMN 1 Capillary Column Model Number: HP 19091J-216 HP-5 5% Phenyl Methyl Siloxane Max temperature: 325 °C Nominal length: 59.0 m Nominal film thickness: 1.00 um Mode: constant flow Initial flow: 1.5 mL/min Nominal init pressure: 7.90 psi Average velocity: 31 cm/sec Method: TO14.M Wed Apr 30 14:03:13 2003 Page: 1

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Inlet: Front Inlet Outlet: MSD Outlet pressure: vacuum FRONT DETECTOR (NO DET) BACK DETECTOR (NO DET) SIGNAL 1 SIGNAL 2 Data rate: 20 Hz Type: test plot Save Data: Off Zero: 0.0 (Off) Range: 0 NAL 1 Data rate: 20 Hz Type: test plot Save Data: Off Zero: 0.0 (Off) Range: 0 Fast Peaks: Off Attenuation: 0 Range: 0 Fast Peaks: Off Attenuation: 0 COLUMN COMP 1 (No Detectors Installed) COLUMN COMP 2 (No Detectors Installed) THERMAL AUX 2 SHAAL AUX 2 Use: MSD Transfer Line Heater Description: Initial temp: 150 'C (On) Initial time: 0.00 min # Rate Final temp Final time 1 0.0(Off) POST RUN Post Time: 0.00 min TIME TABLE Time Specifier Parameter & Setpoint 7673 Injector Front Injector: ont Injector: Injector not configured, use these parameters if it becomes configured Sample Washes 2 Sample Pumps 4 Injection Volume 1.0 microliters Syringe Size 10.0 microliters PostInj Solvent A Washes 4 PostInj Solvent B Washes 0 Viscosity Delay 0 seconds Viscosity Delay Plunger Speed 0 seconds Fast Back Injector: No parameters specified MS ACOUISITION PARAMETERS General Information Tune File : bfb.u Acquistion Mode : Scan MS Information Solvent Delay : 3.80 min EM Absolute : False : 0 : 1858.8 EM Offset Resulting EM Voltage [Scan Parameters] : 28.5 : 260.5 : 200 : 3 Low Mass High Mass Threshold Sample # A/D Samples 8 Plot 2 low mass Plot 2 high mass : 28.5 : 260.5 [MSZones] MS Quad MS Source : 106 C : 230 C maximum 200 C maximum 250 C END OF MS ACOUISITION PARAMETERS PostRun InstCntl macro(s) exist: msacq2.mac END OF INSTRUMENT CONTROL PARAMETERS

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Appendix IV: Recommended Calibration levels (normal reporting limit based on 200 mL sample analysis)

sample analysis)	Level, ppb v/v*										
Compound	1	2	3	4	4A	5	6	7	8		
Bromochlormethane	10	10	10	10	10	10	10	10	10		
1,4-Difluorobenzene	10	10	10	10	10	10	10	10	10		
Chlorobenzene-d5	10	10	10	10	10	10	10	10	10		
1,2-Dichloroethane-d4	10	10	10	10	10	10	10	10	10		
Toluene-d8	10	10	10	10	10	10	10	10	10		
4-Bromofluorobenzene	10	10	10	10	10	10	10	10	10		
Chlorodifluoromethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Dichlordifluoromethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Chloromethane	-	-	0.4	0.8	2.5	5	10	15	30		
1,2-Dichlorotetrafluoro-methane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Vinyl Chloride	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Methanol	-	-	-	3.2	10	20	40	60	120		
1,3-Butadiene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
n-Butane	-	0.2	0.4	0.8	2.5	5	10	15	30		
Bromomethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Chloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Trichlorofluoromethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Acrolein	-	0.4	0.8	1.6	5.0	10	20	30	60		
Acetonitrile	-	-	0.8	1.6	5.0	10	20	30	60		
Acetone	-	-	-	1.6	5.0	10	20	30	60		
Pentane	-	-	0.4	0.8	2.5	5	10	15	30		
Ethyl Ether	-	0.4	0.8	1.6	5.0	10	20	30	60		
1,1-Dichloroethene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Acrylonitrile	-	0.4	0.8	1.6	5.0	10	20	30	60		
1,1,2-Trichlorotrifluoroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Methylene Chloride	-	-	0.4	0.8	2.5	5	10	15	30		
3-chloropropene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Carbon Disulfide	-	0.2	0.4	0.8	2.5	5	10	15	30		
Trans-1,2-Dichloroethene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Methyl-t-butyl Ether	0.1	0.4	0.8	1.6	5.0	10	20	30	60		
1,1-Dichloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Vinyl Acetate	-	0.4	0.8	1.6	5.0	10	20	30	60		
2-Butanone	-	-	0.8	1.6	5.0	10	20	30	60		
Hexane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
cis-1,2-Dichloroethene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Chloroform	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
1,1,1-Trichloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
1,2-Dichloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Benzene	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
1-Butanol	-	0.4	0.8	1.6	5.0	10	20	30	60		
Cyclohexane	-	0.2	0.4	0.8	2.5	5	10	15	30		
Carbon Tetrachloride	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
Heptane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		
1,2-Dichloropropane	0.1	0.2	0.4	0.8	2.5	5	10	15	30		

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Appendix IV: Recommended Calibration levels (normal reporting limit based on 200 mL
sample analysis), continued

Level, ppb v/v*									
Compound	1	2	3	4	4A	5	6	7	8
Trichloroethene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Dibromomethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Bromodichloromethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
4-Methyl-2-Pentanone	-	0.4	0.8	1.6	5.0	10	20	30	60
cis-1,3-Dichloropropene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
trans-1,3-	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Dichloropropene									
Toluene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
1,1,2-Trichloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
2-Hexanone	-	0.4	0.8	1.6	5.0	10	20	30	60
Octane	-	0.2	0.4	0.8	2.5	5	10	15	30
Dibromochloromethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
1,2-Dibromoethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Tetrachloroethene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Chlorobenzene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Ethylbenzene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
m/p-Xylene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Bromoform	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Nonane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Styrene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
o-Xylene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
1,1,2,2-Tetrachloroethane	0.1	0.2	0.4	0.8	2.5	5	10	15	30
1,2,3-Trichloropropane	-	-	0.4	0.8	2.5	5	10	15	30
Cumene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
n-Propylbenzene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
4-Ethyltoluene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
1,3,5-Trimethylbenzene	0.1	0.2	0.4	0.8	2.5	5	10	15	30
Alpha-Methylstyrene	-	0.2	0.4	0.8	2.5	5	10	15	30
Decane	-	0.2	0.4	0.8	2.5	5	10	15	30
1,2,4-Trimethylbenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
1,3-Dichlorobenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
Benzyl Chloride	-	0.2	0.4	0.8	2.5	5	10	15	30
1,4-Dichlorobenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
1,2-Dichlorobenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
Undecane	-	0.2	0.4	0.8	2.5	5	10	15	30
Dodecane	-	0.2	0.4	0.8	2.5	5	10	15	30
1,2,4-Trichlorobenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
Naphthalene	-	0.2	0.4	0.8	2.5	5	10	15	30
Hexachlorobutadiene	-	0.2	0.4	0.8	2.5	5	10	15	30
Sec-butylbenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
n-Butylbenzene	-	0.2	0.4	0.8	2.5	5	10	15	30
Vinyl Bromide	-	0.2	0.4	0.8	2.5	5	10	15	30
Tert-Butanol	-	0.4	0.8	1.6	5.0	10	20	30	60
2,2,4-Trimethylpentane	-	0.2	0.4	0.8	2.5	5	10	15	30
2-Chlorotoluene	-	0.2	0.4	0.8	2.5	5	10	15	30

\* See section 10.3.11.

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Appendix V: Recommended Low Level Calibration levels (normal reporting limit based on 500 mL sample analysis)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
For compounds listed in Tables 1, 2, and 3 that are noted for Low Level analysis	0.010	0.020	0.040	0.100	0.200	0.500	1.0

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# STL KNOXVILLE ATTACHMENT TO SOP KNOX-MS-0001 TITLE: PROJECT SPECIFIC ANALYTE LIST FOR GIVAUDAN

	(SUPERSEDES: NONE)
Prepared By:	Loch 11 Ar
Reviewed By:	Technical Specialist
Approved By:	Murpy Un hu stpilos
	Quality Assurance Manager
Approved By:	Environmental, Health and Safety Coordinator
Approved By	- 1-21-05
	Laboratory Director

The following additional analytes will be target analytes for the Givaudan project.

CAS NUMBER	COMPOUNDS	REPORTING LIMITS (ppb, v/v)	MOLECULAR WEIGHT (ng/n mole)	SUGGESTED ION
79-29-8	2,3-Dimethyl butane	0.2	86.2	43
80-56-8	a-Pinene	0.2	136.2	93
79-92-5	Camphene	0.2	136.2	93
127-91-3	b-Pinene	0.2	136.2	93
5989-27-5	d-Limonene	0.2	136.2	68
99-87-6	p-Cymene	0.2	134.2	119
98-51-1	4-tert-Butyltoluene	0.2	148.2	133
464-49-3	Camphor*	5.0	152.2	95

\* The initial demonstration of capability indicated poor precision for this analyte by this method as evidenced by the variable recovery of this compound. Any reported values for this analyte should be considered estimated.

# **APPENDIX E**

# **HEALTH AND SAFETY PLAN**

## SOIL VAPOR EXTRACTION PILOT TESTING WORK PLAN

W.G. Krummrich Facility Sauget, Illinois

Prepared For:

SOLUTIA INC. 575 Maryville Centre Drive St. Louis, MO 63141

Prepared By:



STRATEGIC. ENVIRONMENTAL. SOLUTIONS.

101 East Mill Street, Suite D Quakertown, PA 18951 Tel: (800) 486-3575 Fax: (215) 538-2780

# **August 2008**



STRATEGIC. ENVIRONMENTAL, SOLUTIONS.

## HEALTH AND SAFETY PLAN

Project Name: Site Location Project Manager: Site Health and Safety Officer Plan Preparer: Preparation Date:

**Krummrich Facility** Sauget, IL Jaydeep Parikh **Omer Uppal Katherine** Woolhouse August 2008

**Approved:** Health and Safety Plan Preparer:

8/19/2008 Śignature Date

Site Health and Safety Officer:

<u>8/19/2008</u> Date

Signature

**Project Manager:** 

Milan 820/08 Reel Date Signature



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Attachment B	Information Regarding Slip, Trip and Fall Hazards/Heat and Cold Stress
	Symptoms
Attachment C	Data Sheets for Chemicals that may be Encountered at the Site
Attachment D	Exposure/Injury Incident Report

## **1.0 INTRODUCTION**

# **1.1 SCOPE AND APPLICABILITY**

This plan has been prepared to serve as the Health and Safety Plan (HASP) for the Soil Vapor Extraction (SVE) pilot test activities at the W.G. Krummrich plant (Site) in Sauget, Illinois. The work covered by this HASP is being performed on behalf of Solutia, Inc. (Solutia).

This document provides comprehensive procedures to protect the health and safety of project personnel and the plant personnel during implementation of the SVE pilot testing activities, and is compliant with the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120). All activities covered by this HASP must be conducted in complete compliance with this HASP and with all applicable federal, state and local health and safety regulations and Solutia Site specific health and safety requirements. Personnel covered by this HASP who cannot or will not comply will be excluded from Site activities. Each employee working on-site during this project is first required to read this HASP and acknowledge his or her review by signing the Sign-Off Form, which is attached at the conclusion of this document (Attachment A).

# **1.2 SITE VISITORS**

All visitors entering the contamination reduction zone and exclusion zone at the Site will be required to read and comply with the provisions of this HASP. In addition, visitors will be expected to comply with relevant OSHA requirements such as medical monitoring, training and respiratory protection. Visitors will also be expected to provide their own personal protective equipment.

In the event that a visitor does not adhere to the provisions of the HASP, he/she will be requested to leave the work area. All nonconformance incidents will be recorded in the Site log.



# **1.3 MODIFICATIONS TO HASP**

The procedures in this HASP have been developed based on information provided by Solutia and the scope of work. Every effort has been made to address the chemical hazards that may be encountered during the implementation of the SVE pilot testing work. Similarly, this document also discusses the physical hazards associated with the data collection activities. However, unanticipated Site-specific conditions or situations may occur during the implementation of this work. Also, XDD and/or subcontractors may decide to perform certain tasks in a manner that is different from what was originally intended due to a change in field conditions. As such, this HASP must be considered a *working document* that is subject to change to meet the needs of the project.

# 2.0 KEY PERSONNEL

# 2.1 KEY ORGANIZATIONS

The organizational structure for the project and designated responsibilities are as follows:

Organization	Project Responsibility
Solutia, Inc	Coordination with plant personnel/U.S. EPA Review of data and reports
XDD, LLC (XDD)	Execution of project tasks Preparation of reports Other assistance to Solutia
Analytical Laboratory	
Test America	Laboratory analysis of soil and soil vapor samples
Installation Subcontractor	
To be determined	Installation of process equipment, piping and electrical connections
Drilling Contractor	
Roberts Environmental Drilling, Inc.	SVE test well, vapor probe and injection well installation

# 2.2 ORGANIZATIONAL RESPONSIBILITIES

The following section outlines the primary roles and responsibilities of key personnel.

Name/Title	Role/Responsibilities	Telephone
Maureen Lein, Health and Safety Manager	Oversees health and safety program, OSHA compliance program	603-548-0114
Jaydeep Parikh, Project Manager	Overall project supervision	603-778-1100
Omer Uppal, Site Supervisor/ Site Health and Safety Officer (SS/SHSO)	Oversees on-site activities	603-778-1100
Erin Stanisewski, Alternate SS/SHSO	Substitute for SS/SHSO as needed	603-778-1100
XDD project staff/project contractors	Perform work/follow HASP	603-778-1100



Name/Title	Role/Responsibilities	Telephone
Ron Dill, Solutia CMR	Oversee safe work practices consistent with Plant requirements	618-978-3366
Jerry Rinaldi, Solutia Project Manager	Overall project management	314-674-3312

## 2.2.1 HEALTH AND SAFETY MANAGER

The Health and Safety Manager (HSM) has overall responsibility for XDD personnel health and safety on the project. Specific responsibilities include:

- Reviewing and approving the project HASP for clarity and completeness;
- Confirming that the project HASP is implemented in accordance with the XDD Health and Safety Program and all applicable health and safety regulations as established by OSHA and Site specific requirements established by Solutia;
- Ensuring that all accidents, injuries and other incidents are reported by field personnel, investigated and then reported to clients and others (as applicable);
- Coordinate and develop corporate health and safety management system; and
- Coordinate all mandated health and safety training.

The HSM for this project is Maureen Lein of XDD's Stratham, New Hampshire office. Ms. Lein has extensive experience implementing and supervising similar work at hazardous waste sites and has served as the HSM on numerous projects.

# 2.2.2 Project Manager

The Project Manager (PM) is responsible for the implementation of the project. This includes supervision of the preparation and implementation of the project HASP, and conformance with all health and safety practices established by the project HASP, OSHA regulations, and Solutia requirements. In addition, the PM's responsibilities include:

- Selecting a SS/SHSO with the necessary qualifications and experience to direct Site health and safety;
- Selection of qualified contractors

- Ensuring the full implementation of the project HASP by field personnel and project contractors;
- Establishing responsibility for health and safety with project contractors, including reviewing the contractor's HASP(s) and ensuring the XDD HASP has been read, understood and signed by all on-site project personnel;
- Investigating all accidents, injuries and other incidents reported by field personnel and reporting the occurrences and investigation results to the XDD HSD, clients and others (as applicable); and
- Reviewing project contractor HASP(s).

The PM for this project is Jaydeep Parikh of XDD's Quakertown, Pennsylvania office. Mr. Parikh has extensive experience implementing and supervising similar work at hazardous waste sites and has served as the PM on numerous projects.

# 2.2.3 SITE SUPERVISOR/SITE HEALTH AND SAFETY OFFICER

The Site Supervisor/Site Health and Safety Officer (SS/SHSO) is responsible for ensuring that the provisions of this HASP have been approved and are implemented at the Site for XDD personnel, subcontractors and the plant personnel. The SS/SHSO is also responsible for monitoring health and safety during the project work. Changing field conditions may require judgments to be made regarding adequate health and safety protection programs. It is the responsibility of the SS/SHSO to recognize changes in field conditions that may require revisions to the HASP and to notify the PM of these changes. The SS/SHSO must have completed the OSHA 8-hour Hazardous Waste Site Operations Supervisor training in addition to the 40-hour Hazardous Waste Site Operations training and have at least 24 hours of site supervision experience in order to serve in this role.

Specific responsibilities of the SS/SHSO include:

• Overseeing the performance of all field tasks and standard operating procedures (SOPs) to verify compliance with the project HASP;

- Conducting safety meetings with XDD personnel and project contractors to ensure safe working conditions. Safety meetings will be held by the SS/SHSO to ensure that the XDD HASP has been read, understood and signed. Project contractor HASP(s) will be on-site and reviewed;
- Ensuring that all monitoring equipment is calibrated on a daily basis and that the results are properly recorded and filed;
- Confirming that appropriate personal protective equipment (PPE) is properly utilized and reviewing the daily use of PPE (as applicable);
- Monitoring the performance of all project personnel to ensure that safe work practices are employed;
- Identifying Site personnel with special medical problems;
- Reporting any deviations from the anticipated conditions described in the HASP to the PM and HSD if necessary;
- Monitoring and recording results of exposure evaluations;
- Reporting and investigating all accidents, injuries, or other occurrences;
- Halting Site operations, if necessary, to correct potential unsafe work practices; and
- Performing Site safety inspections (refer to Attachment A for a Site Safety Inspection Form).

The SS/SHSO for this project is Omer Uppal of XDD's Stratham, New Hampshire office. Mr. Uppal has extensive experience implementing and supervising similar work at hazardous waste sites. Mr. Uppal has also completed the OSHA 40-hour Hazardous Waste Site Operations Course and the 8-hour Hazardous Waste Site Operations Supervisors Course.

# 2.2.4 Alternate Site Supervisor/Site Health and Safety Officer

The alternate SS/SHSO shall assist the SS/SHSO in the implementation of the project HASP. Specific responsibilities may include all the duties listed above for the SS/SHSO.

The alternate SS/SHSO for this project is Erin Stanisewski. Ms. Stanisewski has supervised similar work at hazardous waste sites and has also completed the OSHA 40-hour Hazardous



Waste Site Operations Course and the 8-hour Hazardous Waste Site Operations Supervisors Course.

# 2.2.5 XDD PROJECT STAFF/PROJECT CONTRACTORS

XDD project personnel and project contractors involved in on-site work and operations are responsible for:

- Attending the safety meetings and making certain that the XDD HASP, as well as any project contractor HASPs, are read, understood and signed;
- Implementing the procedures set forth in the HASP and reporting any accidents, unsafe conditions and/or deviations from the procedures in the HASP to the SS/SHSO (or alternate SS/SHSO);
- Properly using the PPE specified in the HASP;
- Taking all reasonable precautions to prevent self-injury as well as injury to fellow workers;
- Performing only those tasks that can be implemented with confidence and safety; and
- Notifying the SS/SHSO of any special medical problems (i.e., allergies, etc.) and ensuring that on-site personnel are aware of and prepared for such problems.

## 3.0 TASK/OPERATION HEALTH AND SAFETY RISK ANALYSIS

## **3.1 SITE NARRATIVE**

Solutia's W.G. Krummrich plant (Site) is located at 500 Monsanto Avenue (Section 14, T. 2 N., R. 10 W.) in the Village of Sauget, St. Clair County, Illinois, across the Mississippi River from the City of St. Louis. The Site is located approximately one mile east of the Mississippi River in an industrial/commercial area. The treatment area selected for the SVE pilot test was formerly used for benzene and chlorobenzene storage.

## **3.2 SVE PILOT TESTING AREA**

The area of interest for the SVE pilot test is Big Mo (see Figure E.1). A 60-foot by 80-foot area has been designated for the SVE pilot testing (see Figure E.2). The unsaturated soils will be targeted for SVE treatment, and are therefore the focus of the SVE pilot testing. The primary constituents present in this area are benzene, chlorobenzene, 1,2-dichlorobenzene, and 1,4-dichlorobenzene.

## 3.3 RESPONSE ACTIVITY (SCOPE OF WORK)

The scope of work covered by this HASP is 1) drilling for the installation of SVE/air injection wells and vapor points, and soil sampling, 2) installation of process equipment and piping, 3) operation of the SVE pilot test, and 4) monitoring activities associated with the SVE pilot test. The overview of the pilot test location is shown in Figure E.2.

# 3.3.1 DRILLING

Activities:

SVE and/or air injection test wells will be installed at 15 locations around the pilot test target area using a hollow stem auger drilling techniques.Soil borings will be completed to up to 15 feet below grade (bg). SVE and/or air injection test wells will be installed in the fill and upper silty



sand layer and the lower silty sand layer at 11 locations (22 nested wells). Four additional SVE test wells will be installed in the intermediate silty clay layer (4 individual wells). A total of 26 SVE and/or air injection test wells will be installed in the pilot test target area. Vapor points will be installed at 10 locations around the pilot test target area. At eight locations, vapor points will target the fill and upper silty sand layer and the lower silty sand layer (16 vapor points) and at 3 locations, vapor points will target the intermediate silty clay layer. A total of 19 additional vapor points will be installed in the pilot test target area. Soil sampling will be conducted at a total of 16 locations in the pilot test area. Soil samples will be collected from continuous soil cores, and soil samples will be collected from select locations.

Health & Safety: Level D (steel toe boots, hard hat, safety glasses, hearing protection, pants, long-sleeve shirt). Chemical resistant gloves shall also be worn during sample collection. XDD will provide personnel and equipment for breathing zone monitoring. If sustained PID readings exceeding 1 ppm above the background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the analysis of benzene will be drawn to determine if action levels are being exceeded. In the event that the action level for benzene (1ppm) is exceeded, the Site personnel will also be required to wear a respirator with organic cartridges. Driller shall provide additional PPE as needed (e.g., Tyvek suits, chemical resistant gloves).

#### 3.3.2 INSTALLATION

Activities: SVE process equipment including blowers, air-moisture separators and a thermal oxidation unit will be installed near the pilot test area. The process equipment, except for the thermal oxidation unit, will be housed in



an equipment shed/container. Piping will be installed from the equipment shed/container to individual SVE wells.

Health & Safety: Level D (steel toe boots, hard hat, safety glasses, hearing protection, pants, long-sleeve shirt). Additional PPE includes work gloves as needed for handling pipes and equipment. Since intrusive subsurface activity will not be conducted during this task, XDD does not anticipate a need to continuously monitor the breathing zone. If deemed necessary by the SSO or project personnel, breathing zone monitoring may be conducted periodically. XDD will provide personnel and equipment for breathing zone monitoring. If sustained PID readings exceeding 1 ppm above the background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the analysis of benzene will be drawn to determine if action levels are being exceeded. In the event that the action level for benzene (1ppm) is exceeded, the Site personnel will also be required to wear a respirator with organic cartridges.

#### 3.3.3 OPERATION OF THE SVE PILOT TEST

Activities: General operation and maintenance on the SVE system.

Health & Safety: Level D (steel toe boots, hard hat, safety glasses, hearing protection, pants, long-sleeve shirt). XDD will provide personnel and equipment for breathing zone monitoring as deemed necessary by the site personnel. If sustained PID readings exceeding 1 ppm above the background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the analysis of benzene will be drawn to determine if action levels are being exceeded. In the event that the action level for benzene (1ppm) is exceeded, the Site personnel will also be required to wear a respirator with organic cartridges.



#### 3.3.4 MONITORING ACTIVITIES ASSOCIATED WITH THE SVE PILOT TEST

- Activities: Soil sampling activity is described in Section 3.3.1. This sub-section discusses additional monitoring activities associated with the SVE pilot test including soil vapor sampling from SVE wells and vapor probes/points; operation parameter monitoring; extracted soil vapor sampling; and air treatment sampling. Soil vapor samples will be collected using sample ports installed in the system equipment and an air sampling pump. A PID will also be used for soil vapor sampling and air quality monitoring. Operation parameter monitoring will be conducted by direct reading of various pressure, vacuum, temperature and flow gauges.
- Health & Safety: Level D (steel toe boots, hard hat, safety glasses, hearing protection, pants, long-sleeve shirt). Chemical resistant gloves shall also be worn during sample collection. XDD will provide personnel and equipment for breathing zone monitoring as deemed necessary by the site personnel. If sustained PID readings exceeding 1 ppm above the background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the analysis of benzene will be drawn to determine if action levels are being exceeded. In the event that the action level for benzene (1ppm) is exceeded, the Site personnel will also be required to wear a respirator with organic cartridges.

## **3.4 PHYSICAL HAZARDS**

The SVE pilot testing work will be completed at an operating chemical plant. All pilot test workers will be required to attend plant safety orientation, be aware of the plant's safety requirements and follow safety rules. Workers will also be required to be aware of facility-specific danger and evacuation signals and know the location of assembly points and evacuation routes.



Most of the physical hazards associated with the scope of work are directly related to slips, trips and falls. Guidelines for avoiding these hazards are presented in Attachment B and are considered to be part of the HASP. Other potential hazards include injury due to lifting heavy objects. Regular equipment inspections, caution and awareness during equipment operation can limit these occurrences.

The weather presents a variety of physical hazards that will vary from season to season and must be addressed accordingly. High temperatures present the potential for heat stress. Therefore, when conditions dictate, an appropriate heat-stress monitoring program must be implemented and documented on-site. Warmer weather also presents the possibility of thunderstorms, lightning and sudden/severe weather. On-site work may also take place during periods of extreme cold weather. Extreme cold weather presents the potential for frostbite as well as slippery conditions when ice and/or snow is present. Site personnel must be aware of the hazards associated with heat and cold stress. Attachment B provides further information on preventing, recognizing and treating both heat-stress and cold-stress.

## **3.5 CHEMICAL HAZARDS**

Contact and/or proximity to contaminated materials during drilling and testing present a potential for exposure, although the risk of exposure is anticipated to be very low. Potential exposure can be minimized through personal monitoring and the use of appropriate PPE as described in Section 5.0 of this HASP. For all Site activities, personnel must minimize contact with impacted media and safe work practices should be exercised at all times.

Previous sampling and chemical analysis at the Site has identified the following primary constituents that could be present in the unsaturated soils in the test areas: benzene, chlorobenzene, 1,2-dichlorobenzene, and 1,4-dichlorobenzene.

The highest reported concentrations for these constituents in soil in the areas of proposed activity are presented in the following table. Information regarding the properties and hazards associated with each of these constituent is provided in Attachment C of this HASP. All activities and

associated levels of protection described herein are subject to actual field conditions and as such, protection levels may be modified to reflect the actual field conditions.

## Maximum Reported Constituent Concentrations (ug/kg) in the Big Mo Area

	Benzene	Chlorobenzene	1,2-Dichlorobenzene	1,4-Dichlorobenzene
Big Mo	13,000,000	5,100	1,200	1,400

Notes:

ND - not detected

## 3.5.1 CHEMICAL EXPOSURE LIMITS

Exposure limits for the constituents are typically expressed as time-weighted average (TWA) exposure limits and ceiling limits. Different agencies have adopted slightly different methods for expressing exposure limits. OSHA expresses exposure limits as Permissible Exposure Limits (PELs). The National Institute for Occupational Safety and Health (NIOSH) publishes Recommended Exposure Limits (RELs). The American Conference of Governmental Industrial Hygienists (ACGIH) has adopted values for exposure limits referred to as Threshold Limit Values (TLVs). Attachment C presents OSHA PELs, NIOSH RELs, and ACGIH TLVs for the constituents present at the Site.

The ionization potentials, concentrations that are considered Immediately Dangerous to Life and Health (IDLH) and other important data for the constituents are also presented in Attachment C. This chemical information was used to determine appropriate action levels/PPE for activities being performed at the Site.

# 3.5.2 ACTION LEVELS

The action level for an upgrade in respiratory protection from Level D to Level C was established by using the lowest exposure limit for each constituent (i.e., the most conservative value comparing the PEL, REL and the TLV for each of the constituent). Benzene was selected as the reference compound for establishing an action level since it has the lowest exposure limit

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(see Attachment C). The PEL for benzene is 1 part per million (ppm). The lowest PEL for the remaining constituents (chlorobenzene, 1,2-dichlorobenzene and 1,4-dichlorobenzene) is 50 ppm.

Air concentrations will be measured using a PID, which measures total VOCs. PID measurements will be obtained at each field location before the commencement of work to determine the background concentrations. PID measurements will be continually monitored. A change in the current conditions, such as a shift in wind direction, may require additional background readings to be obtained.

If sustained PID readings exceeding 1 ppm above the background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the screening of benzene will be drawn to determine if action levels are being exceeded. In the event that the action level for benzene (1ppm) is exceeded, the Site personnel will be required to upgrade to Level C PPE which includes donning a respirator with organic vapor cartridges. The organic vapor cartridges are rated to provide more than nine hours of protection at 25 ppm benzene concentration.

When air concentrations exceed the specified action level for benzene, the SS/SHSO will initiate an upgrade in the level of respiratory protection. All breathing zone readings are then compared to the reference compound exposure limit (i.e., 1 ppm for benzene). Air concentrations will be continually measured using a PID, which measures total VOCs. If sustained PID readings exceeding 25 ppm above background in the breathing space are observed, Draeger-Tubes<sup>®</sup> for the screening of benzene will be drawn. If the benzene concentration is less than 25 ppm, Site personnel will continue work in Level C PPE. If the screened concentration of benzene is 25 ppm or greater, the SS/SHSO will immediately stop work and evacuate the work area. The SS/SHSO will contact the HSD, and the Project Manager to determine appropriate response actions.

ACTION LEVEL	PPE RESPONSE
Background Concentration <sup>[1]</sup>	Level D
Background Concentration + 1-25 ppm as Benzene <sup>[2]</sup>	Level C <sup>[3]</sup>
Background + >25 ppm as Benzene <sup>[2]</sup>	Evacuate Work Zone <sup>[4]</sup>

# Action Levels for Upgrading Respiratory Protection

Notes:

- [1] Assumed to be zero ppm. If background readings are greater than zero, check calibration of instrument.
- [2] Benzene concentrations measured using Draeger-Tubes<sup>®</sup>.
- [3] Action level is background concentration plus 1 ppm as Benzene, upgrade to Level C.
- [4] If sustained concentrations in breathing zone increase to above 25 ppm as total VOCs measured by a PID, sample the ambient air with a Draeger-Tube<sup>®</sup> for benzene. If the benzene concentration exceeds 25 ppm, immediately stop work and evacuate the work area. Contact the HSM, and the Project Manager to determine appropriate response actions.

Organic vapors respirator cartridges will be worn if respirators are required. These cartridges provide protection against dusts, mist, fumes and atmospheres containing less than 1,000 ppm (0.1 percent) of organic vapors, according to Mine Safety Appliance specifications.

## 3.5.3 POTENTIAL CHEMICAL EXPOSURE PATHWAYS

Many of the VOCs can enter the body by inhalation, ingestion, eye and skin contact and absorption through the skin. Due to the nature of the constituents and the scope of work being performed at the Site, the primary potential routes of exposure include inhalation, ingestion and dermal absorption. This includes inhalation of the chemical vapors and ingestion of impacted groundwater as well as dermal contact with groundwater. The air monitoring program, presented in Section 7.0, was designed to protect against exposure. Compliance with the PPE program presented in Section 5.0 will protect against ingestion and dermal exposure.

The information included in Attachment C indicates exposure pathways and short-term effects such as dizziness or eye and skin irritations, which can be identified promptly. However, long-term effects such as kidney and liver damage may not be easily detected until chronic damage has occurred. It is important that all personnel involved in field activities adhere to the

recommended personal protective procedures advised by the SS/SHSO to reduce the potential for exposure.

# 3.6 BIOLOGICAL HAZARDS

The potential for serious injury through biological sources such as insects, snakes, rodents and poisonous plants is not anticipated for this project since the facility is located in a developed industrial/residential area. However, during the spring and summer months at the Site, the possibility of exposure to certain insects (e.g., ticks, bees, etc.) and poisonous vegetation (e.g., poison ivy) could be present. Biological hazards will be addressed in the field, as necessary, through proper identification of the potential hazards and initiation of protective measures (e.g., avoiding plants and bees nests, etc.) to reduce the potential impacts of the hazard to workers. In addition, pertinent personnel medical information, such as allergies to insect stings, or severe allergies to poisonous plants, will be requested before Site activities are initiated. This information will be kept on-site and used in case of medical emergencies. In addition, the appropriate first aid equipment will be made available on-site.

## **3.7 NOISE HAZARDS**

Noise hazards may be encountered during performance of the scope of work. The drilling work would produce high levels of noise that could cause damage to hearing. Hearing protection must be worn during the drilling work, and during other work if equipment noise levels are anticipated to exceed a time-weighted average of 90 decibels (dB), or a peak sound pressure level exceeding 140 dB. The noise reduction ratio of such hearing protection should be 32 dB or better.

# **3.8 LEVEL OF PROTECTION**

The scope of work includes activities associated with SVE pilot testing. As previously discussed, the potential hazards include inhalation of vapors, ingestion of groundwater, dermal contact with impacted groundwater and physical hazards (e.g., slips, trips and falls). Potential hazards related to damage to underground utilities will be essentially eliminated by proper utility



marking/identification prior to the start of drilling. Any underground utilities located near the work area will be uncovered by hand digging to ensure that utilities are not disturbed by project activities. Care will be taken when working in the vicinity of overhead utilities and roads.

The hazard assessment was the basis for selecting an initial level of protection to be used for the tasks being performed. Equipment and clothing required for each level of protection is described in detail in Section 5.0. The level of protection will be modified by the SS/SHSO, as necessary, based on actual field conditions, the results air monitoring programs (see Section 7.0), predetermined action levels and weather conditions.

## 3.8.1 INITIAL LEVEL OF PPE

All activities will be initiated in Level D PPE (see Section 5.0). It includes, steel toe boots, hard hat, safety glasses, long-sleeved cotton shirts and full pants. Hearing protection will be worn during the drilling work, and chemical resistant gloves will be worn during the sampling work.

## 3.8.2 UNDERGROUND/ABOVEGROUND UTILITIES

All underground piping and utility systems will be located with the designated plant personnel on the day before the commencement of drilling. This will prevent potential equipment contact with underground utilities. Aboveground utilities must be located immediately prior to (at a minimum) commencement of work. All underground utilities and piping near the work area will be uncovered by hand digging as necessary to ensure that utilities and piping are not disturbed by the project activities.

# 3.8.3 Respiratory Protection

Air monitoring for VOCs will be conducted prior to and during Site activities to ensure that ambient concentrations do not pose a threat to the on-site workers and plant personnel. Refer to Sections 3.6 and 7.0 of this HASP for detailed air monitoring program, including exposure levels/action levels. Ambient air monitoring will be conducted using a PID. Before Site



activities begin each day, background VOC levels will be measured upwind and downwind of sampling locations. Continuous monitoring will be performed during sampling activities.

## 3.8.4 SITE CONTROL

During the Site operations, it is necessary to restrict entry of the plant employees into the work areas. Caution tape will be used to identify the area as a restricted work area. General public is not expected as the facility is restricted to general public.

## **3.9 ACCIDENT PREVENTION**

## 3.9.1 FIRE AND EXPLOSION PREVENTION

The following precautions will be taken to minimize and/or address potential fire and explosion hazards and accidents:

- Fire extinguishers rated for Class A, B and C fires will be maintained on any construction equipment used for Site activities. Personnel will be trained in the proper use of fire extinguishers. All extinguishers will be routinely inspected and recharged after use;
- No smoking or open fires/flames will be permitted near the work area; and
- All overhead and underground utilities will be identified before initiating any intrusive activities.

A tri-gas meter will be used to monitor extracted soil vapor for explosive conditions due to the high level of potentially explosive contaminants in the target area.

# 3.9.2 SAFE LIFTING PROCEDURES

To avoid injuries, the following lifting procedures should be used when lifting heavy objects:

- Inspect the ground around the object and the route over which it will be carried;
- Remove all obstructions from the path of travel;

- Make sure that clearances between pieces of equipment are sufficient for removal of the object;
- Inspect the object to determine the safest way to grasp it;
- Dry any wet or greasy objects;
- Position the body with feet parted;
- Use a sit-down position, keeping the back straight;
- Draw the load close to the body, keeping arms and elbows tucked to the side of the body;
- Extend the fingers and hand around the object using the full palm;
- Tuck in the chin, keeping the neck and head in line with the back;
- Position the body so weight is centered over the feet; start the lift with a thrust of the rear foot; and
- Use hand-trucks, forklift trucks, or hoists to move any objects that cannot be lifted comfortably.

# 4.0 PERSONNEL TRAINING REQUIREMENTS

All XDD personnel and project contractors performing activities described within this HASP must be trained and experienced at performing their assigned work tasks. Individuals assigned to the project that may be exposed to physical and chemical hazards shall undergo training including Plant's pre-job safety training to:

- Understand the scope and objectives of the project;
- Provide awareness of hazards and corresponding health and safety protocols to mitigate these hazards;
- Ensure that the health and safety of XDD employees, contractors, other agencies and the plant personnel are maintained; and
- Safeguard the health and safety of all employees and the public by complying with all applicable laws, rules and regulations.

# 4.1 TRAINING

All personnel, including visitors, entering the exclusion or contamination reduction zones are required to have certifications of completion for training in accordance with 29 CFR 1910.120(e).

# 4.2 TAILGATE SAFETY MEETINGS

XDD will require tailgate safety meetings to be conducted by the SS/SHSO on a daily basis during this project. Tailgate safety meetings will be conducted in order to discuss Site activities and apprise field personnel of the potential hazards present on-site (refer to Attachment A for the tailgate safety meeting form). These meetings will serve as a forum for dialogue between the SS/SHSO and field personnel regarding Site safety. Daily safety meetings will emphasize the health and safety issues applicable to the day's activities. Physical and chemical hazards will be identified and all safeguards will be discussed including monitoring and PPE. Emergency telephone numbers, directions to the nearest hospital and the emergency contingency plan will be discussed.



## **4.3 RESPIRATOR FIT TESTING**

All persons entering an area that will potentially require the use of any negative pressure respirator must have a qualitative fit test (at a minimum). This test must have been administered in accordance with OSHA 29 CFR 1910.134 or American National Standards Institute standards within the last 12 months. Documentation of the fit testing will be required if respirator use is warranted.

## 4.4 MEDICAL REQUIREMENTS

All personnel (including visitors) entering the exclusion or contamination reduction zones must be certified as medically fit to work. Personnel must be certified fit to wear a respirator, if appropriate, in accordance with 29 CFR 1910.120.

## 4.5 XDD PERSONNEL TRAINING INFORMATION

Current training information is presented in the table below for XDD Site personnel:

Name	Title	Medical Surveillance	Fit Test Current		Annual 8- Hour Heath and Safety	Supervisor Training	First Aid /
		Current	Qualitative	Quantitative	(OSHA) Current	(OSHA)	CPR
Omer Uppal	SS/SHSO	Y	Y	Ν	Y	Y	Y
Erin Stanisewski	Alternate SS/SHSO	Y	Y	Ν	Y	Y	Y

# 5.0 PERSONAL PROTECTIVE EQUIPMENT

A description of the PPE required for Site activities is presented in this section. If the action level (see Section 7.0) for the task is exceeded, the level of protection will be upgraded according to the requirements listed below.

# 5.1 LEVEL D PPE

The initial level of protection for Site personnel and contractors is Level D PPE. The Level D PPE consists of most or all of the following equipment:

- Steel toe work boots;
- Long pants, long sleeved cotton shirt;
- Hard hat;
- Tyvek coveralls (as needed when there is a potential for contact with impacted media);
- Earplugs or Earmuffs (when working near drilling equipment or if noise levels exceed 90 dBA/140 dB peak);
- Chemical resistant work gloves if necessary during sampling;
- Nitrile or latex inner disposable gloves during sampling; and/or
- Safety goggles or safety glasses.

# **5.2 LEVEL C PPE**

Level C PPE includes a full-face or half-mask air-purifying respirator (NIOSH approved) with appropriate organic vapor cartridges, in addition to the Level D PPE. Level C protection should be used when air monitoring indicates the need for air-purifying respirators. The action levels/criteria for upgrading to Level C are defined in Section 7.0.

If conditions change so that Level C PPE is not adequate, work at the Site will stop and the HASP will require revision.



# **5.3READY BAG**

All XDD personnel will be issued a ready bag containing PPE equivalent to Level C equipment. Each employee is responsible for ensuring his/her individual ready bag contains all required PPE before leaving for the Site (see Attachment A for the Ready Bag checklist form).

# 5.4 ACTION LEVELS FOR CHANGING LEVELS OF PROTECTION

As previously discussed in Section 3.4.2, the following action levels have been established for upgrading from Level D to Level C PPE.

# Action Levels for Upgrading Respiratory Protection

ACTION LEVEL	PPE RESPONSE
Background Concentration <sup>[1]</sup>	Level D
Background Concentration + 1-25 ppm as Benzene <sup>[2]</sup>	Level C <sup>[3]</sup>
Background + >25 ppm as Benzene <sup>[2]</sup>	Evacuate Work Zone <sup>[4]</sup>

Notes:

- [1] Assumed to be zero ppm. If background readings are greater than zero, check calibration of instrument.
- [2] Benzene concentrations measured using Draeger-Tubes<sup>®</sup>.
- [3] Action level is background concentration plus 1 ppm as Benzene, upgrade to Level C.
- [4] If sustained concentrations in breathing zone increase to above 25 ppm as total VOCs measured by a PID, sample the ambient air with a Draeger-Tube<sup>®</sup> for benzene. If the benzene concentration exceeds 25 ppm, immediately stop work and evacuate the work area. Contact the HSM, and the Project Manager to determine appropriate response actions.



# 6.0 MEDICAL SURVEILLANCE REQUIREMENTS

# 6.1 MEDICAL SURVEILLANCE PROGRAM

All XDD and contractor personnel performing any on-site activities within the contaminant exclusion zone are under a Medical Surveillance Program in accordance with OSHA 1910.120 requirements. The purpose of the Medical Surveillance Program is to:

- Identify illnesses or medical conditions that could put an employee at an unusual risk from potential exposures and/or situations;
- Ensure that each employee can safely utilize respirators; and
- Establish and maintain an employee medical database to monitor any abnormalities related to exposure.

Any personnel of child-bearing age will be informed of potential or known Site constituents that are suspected to be teratogens or mutagens. The XDD Medical Surveillance Program includes:

- A baseline physical examination;
- Medical determination of fitness for duty, including work restrictions after any jobrelated injury, illness, or non-job-related absence lasting more than three working days;
- The review of potential exposure to determine the need for specific biological and medical monitoring;
- Medical evaluation after known or suspected hazardous exposures;
- Physical examinations for field personnel on an annual basis; and
- Exit physical examinations with attention given to specific exposures and/or symptoms.

# 6.2 ACCIDENT AND INJURY REPORTING

A list of procedures for reporting accidents/incidents is provided below. The procedures are to be performed in the order indicated.

1. Contact appropriate emergency services numbers listed in Section 10.1. Be prepared to provide the following information when reporting an accident:

- Your name and present location;
- Location of accident/incident;
- Name and affiliation of injured party or parties;
- Description of injuries/exposure;
- First aid presently being administered;
- Pertinent information regarding chemicals/hazardous materials involved (if applicable);
- Description of accident, including time accident occurred and suspected cause; and
- Temporary measure taken to minimize further risk to personnel (Site evacuation, etc.).
- 2. Call the XDD PM and provide the information listed under item one (1).
- 3. Complete the Exposure/Injury Incident Report (Attachment D) and promptly send to XDD SS/SHSO.

The above information is not to be released to personnel other than the Solutia/XDD PM, the SS/SHSO, emergency response personnel and responding medical personnel.

# 6.3 RECORD KEEPING

All documents will be maintained by XDD in accordance with OSHA specifications. Documents will be maintained at XDD's Stratham, NH office and on-site, as applicable. Medical records are also kept at XDD's Stratham office.

Documents that will be maintained by XDD in the Stratham, NH office include:

- Medical surveillance records;
- Illness and injury reports;
- Accident and incident reports;
- Training documentation/certificates;
- Fit test results;
- Medical authorization records for respirator use;
- Safety equipment calibration records;



- Safety meetings records;
- Material Safety Data Sheets (as applicable) for all potentially hazardous materials; and
- Original project HASP.

Documents that will be maintained by XDD on-site include:

- Illness and injury reports;
- Accident and incident reports;
- Safety meetings records;
- Material Safety Data Sheets (as applicable) for all potentially hazardous materials;
- Fire extinguisher inspection records;
- Emergency contacts list and hospital map;
- Copy of project HASP; and
- Equipment calibration and air monitoring data.

## 7.0 AIR MONITORING

# 7.1 HAZARD MONITORING PROGRAM

Air monitoring will be conducted by the SS/SHSO, the alternate SS/SHSO, or an appointed designee during the Site work activities. Air monitoring with direct-reading instrumentation will be the means of determining the adequacy of the current level of protection relative to existing Site conditions. Monitoring equipment will be calibrated daily prior to the start of field activities. Monitoring results and calibration logs will be completed and filed with the HASP.

## 7.1.1 MONITORING EQUIPMENT

A PID equipped with a 10.6 electron-volt bulb will be used to monitor employee exposures during project activities. All constituents listed in Attachment C have ionization potentials below the PID lamp strength.

## 7.1.2 AIR MONITORING PROTOCOL

Prior to the start of work, PID readings will be taken upwind of the work area, within the work area and downwind of the work area to establish baseline ambient air levels.

Once baseline air quality conditions have been determined, personnel may begin work using the appropriate level of PPE. Upon entering the work area and initiating intrusive activities, air monitoring in the breathing zone will be performed when:

- Site work begins;
- Operations change;
- Invasive Site activity begins (i.e., start of drilling);
- Constituents other than those previously identified are being handled;
- Personnel begin to handle obviously contaminated materials; and/or
- Personnel are handling leaky drums or containers.



All breathing zone and ambient air readings will be recorded in the field notebook. Instruments must be calibrated daily (at a minimum), or as specified by the manufacturer in the calibration protocol. Calibration readings will be logged using the forms supplied in Attachment A of this HASP.

## 8.0 SITE CONTROL MEASURES

# 8.1 BUDDY SYSTEM

When performing work in Level C PPE, or when Site conditions present a risk to personnel, the implementation of a buddy system is mandatory. A buddy system requires at least two people working together as a team and looking out for each other.

# **8.2 SITE COMMUNICATIONS PLAN**

Successful communications between the field teams and contact with personnel in the support zone is essential. The following communications will be available during activities at the Site:

- Cellular phone
- Vehicle horn
- 2-way radio

# 8.3 EMERGENCY ALARM PROCEDURES

In the event of an emergency, the following signals will be used to indicate the respective emergencies:

Emergency Stop Work – Upgrade to Level C Fire Man Down Signal 1 blast on vehicle horn

2 blasts on vehicle horn 3 blasts on vehicle horn

# **8.4 SITE SECURITY**

The XDD SS/SHSO or appointed designee will be responsible for preventing unauthorized access into the work area and for accounting for project team members on-site at all times. The immediate work area boundaries may be delineated with signage, barricades, fences, cones and/or caution tape.

## 8.5 SITE MAP

A Site map is provided on Figure E.1. The map will be updated throughout the course of Site operations to reflect changes in Site activities and conditions.

## 8.6 SITE CONTROL AND WORK ZONES

The objectives of Site control will be to prevent the spread of potential contamination to clean areas of the Site and to protect Site personnel and the public from contact with potential Site hazards. The immediate work area, where contact with Site hazards is possible as previously discussed, shall be deemed the exclusion area. Access to the exclusion area will be limited, as determined by the SS/SHSO, to only those individuals who:

- are authorized to be on-site by XDD or Solutia;
- possess the required health and safety training certifications and necessary documentation of medical monitoring; and
- are properly protected against the anticipated Site hazards.

Every effort will be made to physically demarcate the exclusion area using banner tape, barricades, fencing and/or signs. The exclusion area will be large enough to ensure protection of persons outside of the area. The SS/SHSO or appointed designee will be responsible for keeping unauthorized persons from entering the exclusion area. All locations outside the exclusion area will be considered to be the clean area and will serve as the support location for Site activities.

# 8.7 CPR TRAINING

All on-site XDD employees will have current certifications in both First Aid and CPR.



## 8.8 NEAREST MEDICAL ASSISTANCE

The nearest medical assistance to the Site is the Kenneth Hall Regional Hospital in East Saint Louis, Illinois, approximately 2.2 miles from the Site. The directions and map to the hospital are included in Section 10 and Figure E.3 of this HASP and will be posted at the Site during remediation activities. A dispensary staffed by a registered nurse during the normal business hours (7 am to 4 pm, Monday to Friday) is located on the plant property.

## 9.0 DECONTAMINATION PLAN

## 9.1 EQUIPMENT DECONTAMINATION

Decontamination will include the inspection of all equipment and vehicles to ensure contaminated materials are not tracked to areas outside of the exclusion area. Drilling equipment will be decontaminated by steam cleaning after use at the Site and prior to demobilization. All such decontamination will be performed over a bermed, 6-millimeter (mil) plastic liner and decontamination fluids will be stored in 55-gal drums. The 55-gal drums will be stored in the existing waste storage warehouse south of Big Mo area.

## 9.2 PERSONAL DECONTAMINATION

There is a potential that certain tasks will require implementation of the personal decontamination procedures outlined below. Such a determination will be made by the SS/SHSO and will typically be presented during the daily tailgate meeting. If necessary, a decontamination area will be set up outside of the exclusion zone. This area will be called the contaminant reduction zone. Prior to commencing work, all personnel will be trained in routine decontamination procedures by the SS/SHSO or appointed designee.

Personal decontamination procedures, if deemed necessary by the SS/SHSO, will be as follows:

- 1. Enter contaminant reduction zone.
- 2. Wash, rinse and dispose of outer disposable protective clothing and boots.
- 3. Remove outer non-disposable protective clothing (boots, etc.).
- 4. If a respirator was used, remove, wash, rinse, sanitize and place in designated area for drying.
- 5. Exit contaminant reduction zone.
- 6. Wash face, hands and forearms.

Respirators will be decontaminated in the contamination reduction zone. The respirators will be sanitized in the manner recommended by the respirator manufacturer to ensure that they are not



damaged by the decontamination process. After sanitizing, the respirators will be hung to dry and then placed into dedicated storage devices. In the event that respirator accessories or components are grossly contaminated and/or are difficult to decontaminate, the accessory/component will be discarded and replaced. Each person is responsible for the decontamination and maintenance of his/her assigned respirator. Personnel will be trained in respirator maintenance as part of the health and safety training program.

Personnel will wash face and hands immediately after removing PPE. Personal hygiene will be performed prior to entering project vehicles, eating, drinking, smoking or departing the work area. In the event that running water is not available for this practice, towelettes will be utilized.

## 9.3 DISPOSAL OF WASTE

All materials associated with decontamination, such as water used for decontamination and the 6-mil plastic liner, will be drummed for proper disposals off-site. Similarly, all soil cuttings generated during the drilling activities will be stored in 55-gallon drums for characterization and appropriate disposal at a later date. All 55-gal drums will be stored in the existing waste storage warehouse south of Big Mo area.

## 10.0 EMERGENCY RESPONSE/CONTINGENCY PLAN

## **10.1 EMERGENCY CONTACTS**

Site emergency communications must be pre-arranged and understood by all on-site personnel. Means of such communications will include using hand signals and air horns (such as a vehicle horn). See Section 8.3 for description of the primary signals that will be used. An external communication system between Site personnel and off-site response agencies shall be made by contacting the agencies listed below.

Agency	Contact	Number
		(10, 400, 7117
Local Medical Emergency Facility	Kenneth Hall Regional Hospital	618-482-7117
5	Edward Droste	603-778-1100
XDD Health and Safety Manager	Maureen Lein	603-548-0114 (cell)
		603-887-3335 (home)
Ambulance, Fire and Police	Solutia	618-482-2000
Solutia Headquarters	Jerry Rinaldi	314-674-3312
State Poison Control Center	Illinois Poison Control	800-222-1222
Nearest Telephone	XDD cell phone	603-498-4411
Solutia Headquarters State Poison Control Center	Solutia Jerry Rinaldi Illinois Poison Control	603-887-3335 (home) 618-482-2000 314-674-3312 800-222-1222

# 10.2 LOCAL MEDICAL EMERGENCY FACILITY INFORMATION AND DIRECTIONS

Name of Hospital:

Kenneth Hall Regional Hospital

Phone No.:

618-482-7117

Route to Hospital: (also see Figure E.3)

- 1. Head West on Monsanto Ave toward Mississippi Ave.
- 2. Turn RIGHT onto **IL-3** and follow for 0.9 miles.
- 3. Take the I-70 E/I-55 N/I-64 E exit toward Indianapolis/Chicago/IL-3N in 0.6 miles.
- Keep RIGHT at the fork, follow signs for E St Louis/Business District/Fourth St and merge onto S 4<sup>th</sup> St in 0.3 miles.



- 5. Slight RIGHT to stay on **S** 4<sup>th</sup> St in 0.1 miles.
- 6. Turn RIGHT at **E Broadway** in 0.3 miles.
- 7. The hospital is located on the left side of the road.

### **10.3 CONTINGENCY PLANS**

### 10.3.1 PRE-EMERGENCY PLANNING

During the daily Tailgate Safety Meetings (discussed in Section 4.2) all employees will be trained in and reminded of provisions of the emergency response plan, communications systems and evacuation routes. The hazardous conditions associated with the Site activities are discussed in Section 3.0 and should be reviewed and revised, if necessary, on a regular basis by the SS/SHSO. Continued review of hazardous conditions will ensure that the HASP is adequate and consistent with the prevailing Site conditions.

### 10.3.2 PERSONNEL ROLES AND LINES OF AUTHORITY

The SS/SHSO has the primary responsibility for responding to and correcting emergency situations. This includes employing necessary measures to ensure the safety of the Site personnel and the plant personnel, ensuring that corrective measures are implemented and notifying the appropriate authorities.

### 10.3.3 Emergency Response and Contingency Plan

If an injury occurs, the following actions should be taken:

- Get medical attention for the injured person immediately (see Section 10.1 for emergency telephone numbers);
- Notify the SS/SHSO, XDD PM and Solutia CMR. The SS/SHSO will assume charge during a medical emergency;
- Notify the injured person's office;

- Depending on the type and severity of the injury, notify the corporate consulting physician or the occupational physician for the injured person; and
- Prepare the Exposure/Injury Incident Report (attached in Attachment D). The XDD PM is responsible for its preparation and submittal to the Human Resources Department.

### 10.3.4 FIRE/EXPLOSION AND SPILL/RELEASE PROCEDURES

The initial response to minor fire/explosion incidents that are controllable by using on-site equipment (e.g., fire extinguisher) will be performed by Site personnel. However, if the emergency is determined to be uncontrollable, local emergency agencies will be contacted.

### 10.3.5 MEDICAL RESPONSE

In case of medical emergencies, personnel trained in first aid will initially respond to non-lifethreatening injuries. Follow-up treatment shall be provided by professional medical services for all but minor injuries. Should a life-threatening emergency occur, professional medical services will be contacted immediately.

In the case of certain non-life threatening emergencies, decontamination procedures may be modified in accordance with the specific circumstances. Outer protective clothing shall only be removed if doing so does not delay the treatment or aggravate the injury. Respirators shall not be removed unless the victim has stopped breathing or has been removed from the hazardous breathing zone area. Standard decontamination procedures shall be followed when possible.

Injury victims will be administered first aid treatment on-site in the following manner:

- An individual with current first aid and/or CPR certifications, depending on the nature of the injury, will administer the victim first aid using on-site emergency equipment; and
- The local first aid rescue unit, the local hospital, and the XDD and Solutia PMs will be notified as to the nature of the emergency.



Signs, symptoms and treatment of heat and cold related illness are discussed in the standard safety procedures presented in Attachment B. The SS/SHSO will be trained in monitoring, recognizing and treating heat and cold related stress. If the victim is not obviously contaminated, decontamination procedures shall be minimized so that treatment may be administered as soon as possible.

### 10.3.6 SPILL/RELEASE RESPONSE

All liquid-phase hazardous materials will be properly contained in a designated area on-site. PPE will be utilized as appropriate and under the direction of the SS/SHSO.

### 10.3.7 SITE EVACUATION

Although it is very unlikely, evacuation of Site personnel may be necessary during a severe emergency. Unusual events that may require evacuation include: flooding, tornado, earthquake, severe weather, fire or spills. If evacuation is required, the Solutia CMR and/or SS/SHSO will give the signal to shutdown operations and begin evacuation. Evacuation routes will be discussed at the daily tailgate meetings prior to the commencement of work. It is the responsibility of each individual to evacuate the area(s) in an orderly manner. Personnel will assemble in an area that will be pre-designated by the SS/SHSO. Further instructions will be provided at the assembly area.



### 11.0 HAZARD COMMUNICATION

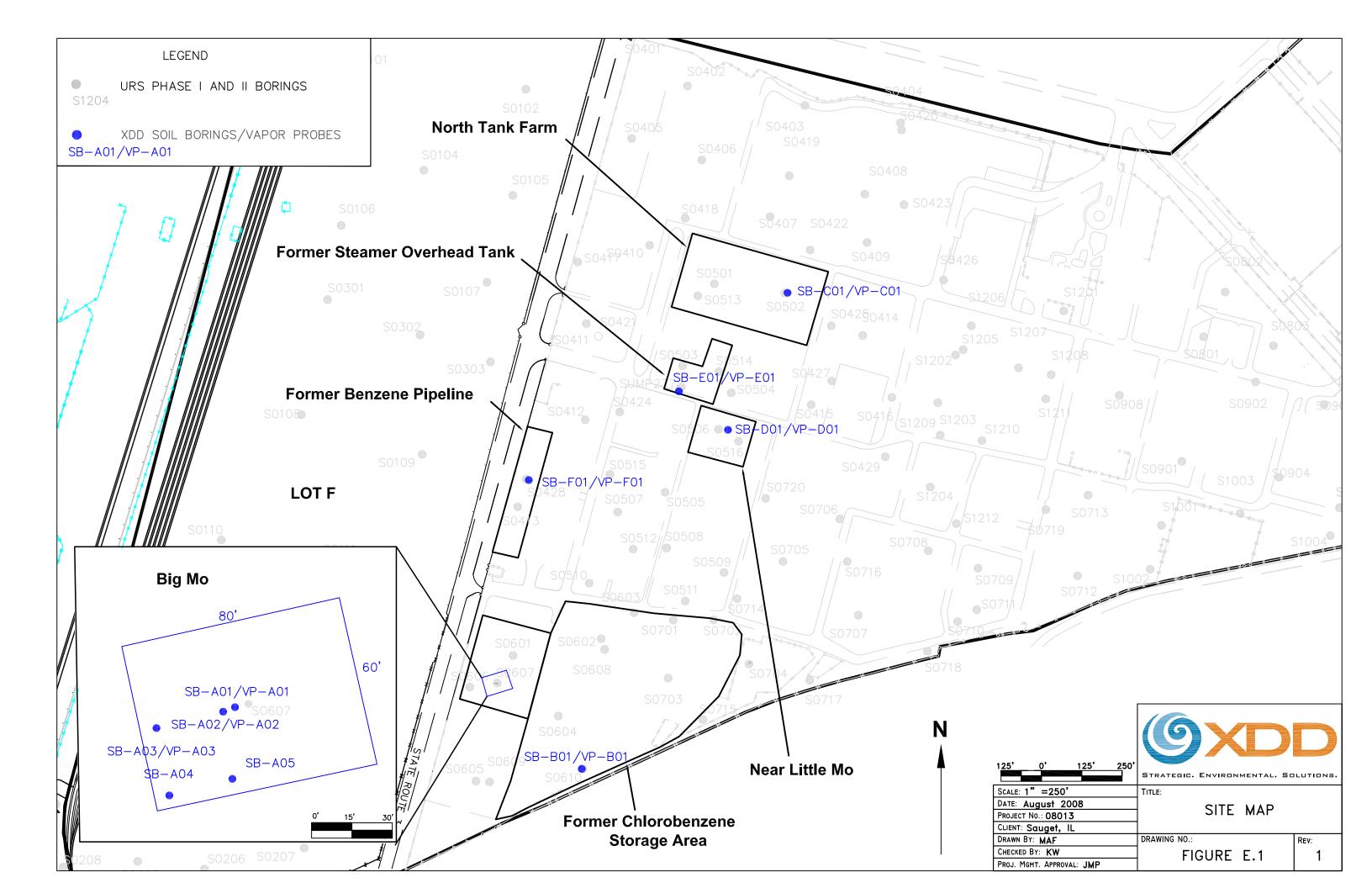
All XDD personnel and subcontractors will be required to read and sign-off on the HASP before beginning work on the project. In addition, all personnel should follow the OSHA standards on hazard communication as listed in 29 CFR 1910.120. A tailgate safety meeting will be held prior to commencing work each day. All XDD personnel and subcontractors will be informed of all potential hazards associated with the project. The HASP will be kept on-site at all times and the SS/SHSO shall ensure that all personnel will have access to the HASP at any time during Site activities, upon request.



**FIGURES** 

### APPENDIX E HEALTH AND SAFETY PLAN

SVE PILOT TESTING WORK PLAN



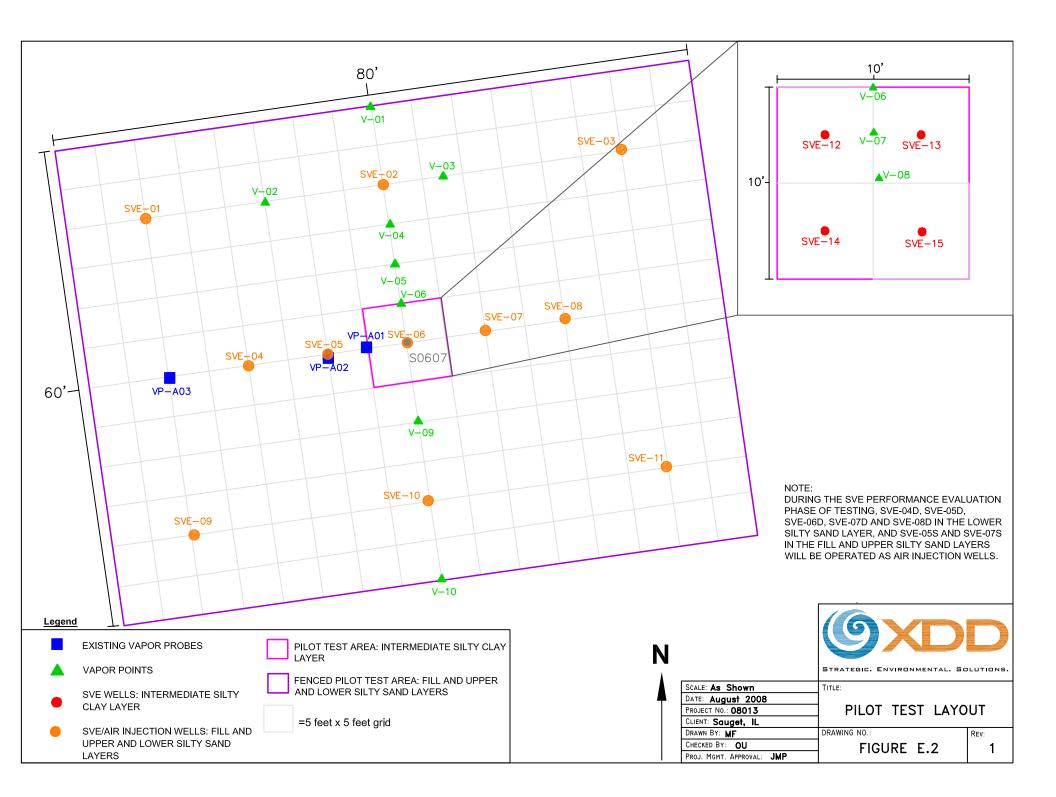
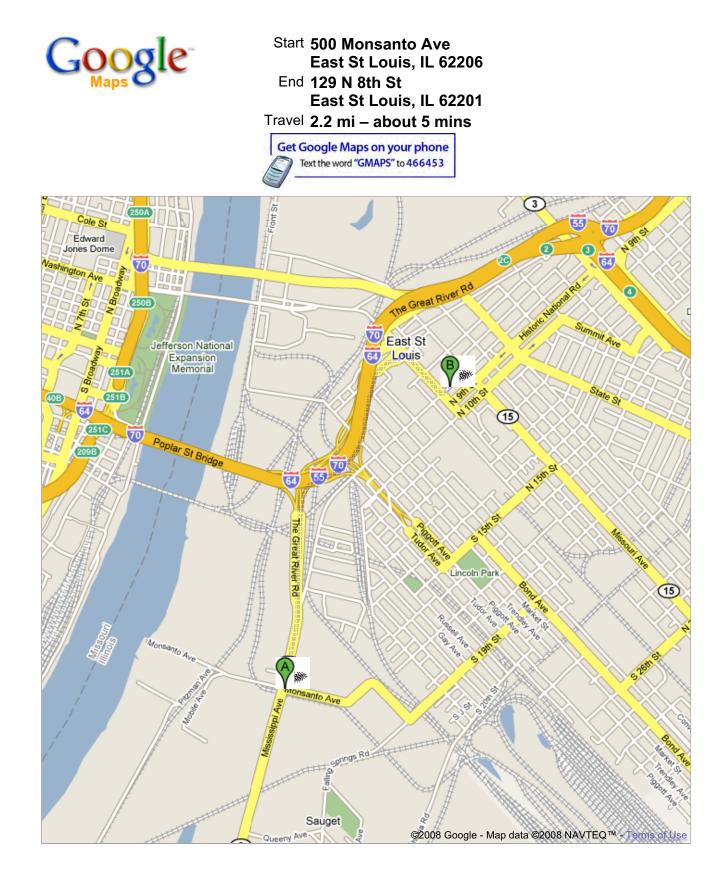


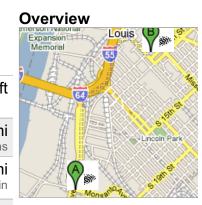
Figure E.3 - Directions to Nearest Hospital



500 Monsanto Ave East St Louis, IL 62206 Drive: 2.2 mi – about 5 mins	
<ol> <li>Head west on Monsanto Ave toward Mississippi Ave</li> </ol>	39 ft
➡ 2. Turn right at IL-3	<b>0.9 mi</b> 2 mins
<ol> <li>Take the I-70 E/I-55 N/I-64 E exit toward Indianapolis/Chicago/IL-3 N</li> </ol>	<b>0.6 mi</b> 1 min
<ul> <li>4. Keep right at the fork, follow signs for E St Louis/ Business District/Fourth St and merge onto S 4th St</li> </ul>	0.3 mi
➡ 5. Slight right to stay on S 4th St	0.1 mi
➡ 6. Turn right at E Broadway	0.3 mi 1 min
<ul> <li>← 7. Turn left at N 8th St</li> <li>(B) 129 N 8th St</li> </ul>	249 ft
Ϋ́East St Louis, IL 62201	

These directions are for planning purposes only. You may find that construction projects, traffic, or other events may cause road conditions to differ from the map results.

Map data ©2008 NAVTEQ™







Map data ©2008 NAVTEQ™



### ATTACHMENT A SITE HEALTH AND SAFETY FORMS

### APPENDIX E HEALTH AND SAFETY PLAN

SVE PILOT TESTING WORK PLAN



### STRATEGIC. ENVIRONMENTAL. SOLUTIONS. TAILGATE SAFETY MEETING

Date:	_ Time:	_ Page:	_ of
Client:		Project #:	
Project #:	_ Work Description:		
TODAY'S WORK:	-		

### **SAFETY TOPIC(S) PRESENTED** (attach outline/notes as necessary) Specific Topic(s)

Air monitoring equipment: \_\_\_\_ Incidents/Accidents: SAFETY ISSUES/CONCERNS RAISED BY SITE PERSONNEL:

SAFETY ACTIONS TO BE TAKEN TODAY:							
Action Item	Assigned To						

### **ATTENDEES:**

Signature	Company	Name (print)	Signature	Company
	Signature	Signature Company 	Signature       Company       Name (print)         Image: Signature       Image: Signature       Image: Signature         Image: Signature       Image: Signature       Image: Signature       Image: Signature         Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature         Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature         Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature         Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature       Image: Signature	Signature     Company     Name (print)     Signature       Image: Signature     Image: Signature     Image: Signature       Image: Signature     I

### **MEETING CONDUCTED BY:** Health & Safety Officer:

Name (Printed)

Signature

Site Superintendent:

Name (printed)

Signature

A-1

22 MARIN WAY, UNIT 3, STRATHAM, NH 03885 • WWW.XDD-LLC.COM 603-778-1100 • F 603-778-2121

### PLAN ACCEPTANCE FORM

### PROJECT HEALTH AND SAFETY PLAN

INSTRUCTIONS: This form is to be completed by each person who works on the subject work site and returned to the Health and Safety Manager.

Job Number

Client/Project:

Date:

I represent that I have read and understand the contents of the above Plan and agree to perform my work in accordance with it.

Signature

Print Name

Company Name

Date

### HEALTH AND SAFETY

### PLAN FEEDBACK FORM

Job Number:

Job Name:

Date:

Problems with plan requirements:

Unexpected situations encountered:

Recommendations for future revisions:

### DAILY INSTRUMENT CALIBRATION CHECK SHEET

Instrument:

Serial #:

Date Remarks	Pure Air Y/N	Calibration Gas (ppm)	Battery Check (good/bad)	Calibrated

### **READY-BAG CHECKLIST**

- 1. Full Face of Half Face Respirator. Includes: respirator, carbon filters, nose-cup insert, protective eye lens cover.
- \_\_\_\_\_2. Boot covers or chemical resistant boots
- \_\_\_\_\_3. Eye wash kit
- \_\_\_\_4. Hard Hat
- \_\_\_\_\_5. Insulated steel-toed work shoes.
- \_\_\_\_\_6. Insulated coveralls.
- \_\_\_\_\_7. Insulated Work Gloves.
- \_\_\_\_\_8. Disposable ear plugs.
- \_\_\_\_\_9. Safety Glasses with side-shields or Splash Goggles
- \_\_\_\_\_10. Plastic Disposal Bags.
- \_\_\_\_\_11. Latex or Nitrile Gloves.
- \_\_\_\_\_12. Duct Tape.
- \_\_\_\_13. Emergency First Aid Kit.

**Note:** The equipment listed above comprises the standard "Ready Bag", and will provide appropriate protection from chemical exposures and noise in most situations encountered. However, for a particular job, certain items that are not included in the standard ready bag may be required, for example, a different type of coverall or respirator cartridge. Therefore, the Site-Specific Health and Safety Plan should always be consulted to make sure the proper equipment is brought to the site.

### WEEKLY FIRE EXTINGUISHER AND FIRST AID KIT CHECK SHEET

Fire Extinguisher Serial #:\_\_\_\_\_

Date Remarks	Fire Extinguisher on-site (Y/N)	Fire extinguisher pin in place (Y/N)	First aid kit on-site (Y/N)	First aid kit Contents current (Y/N)



### BTRATEGIC, ENVIRONMENTAL SOLUTIONS. SITE SAFETY INSPECTION

Project Name:	Date:
Project Number:	Inspector:
Instructions: Check	off every item as "Yes" or "No", or "Not Applicable". All "No" items must have
an explanation of co	prrective actions undertaken to correct the problem.

Inspection Item	Yes	No	N/A	<b>Corrective Actions</b>	Date of Correction
1		MENTA	TION	SIGNS	Goncetion
Documentation such as Safety Plan,					
MSDS's, air monitoring logs, training					
records, and inspections are up to					
date.					
Postings such as emergency numbers,					
hospital map, permits, OSHA poster,					
and speed limit signs are maintained.					
H	<b>JUSEK</b>	EEPIN	G/WOR	<b>KAREA</b>	
Work areas are kept clean and					
orderly, and exits and walkways are					
not blocked.					
Materials are stored in proper and					
stable configurations.					
Hazards are not created by sharp					
surfaces, open pits, wet surfaces, or					
tripping hazards.					
A clean break area is provided to					
employees					
Adequate lighting is provided indoors					
and for outdoor work after dark.					
Employees are working carefully and					
without horseplay.					
Emergency equipment such as					
eyewashes, first aid kits, and fire					
extinguishers are full, inspected and					
accessible					
	LADD	ERS & S	<b>SCAFF</b>	DLDS	
Ladders have no damage, defects, or					
missing parts, including support feet.					
Ladders extend three feet above the					
top surface being climbed, and are					
secured at the top and stable at					
bottom.					
Only wooden or fiberglass ladders are					
used near electrical lines.					
Scaffolds are on level, stable surfaces,					
floorboards are tight fitting, and has					
no damage or missing parts.					
Employees working on platforms					
higher than 6					



### SITE SAFETY INSPECTION

Inspection Item	Yes	No	N/A	<b>Corrective Actions</b>	Date of Correction
Gas or diesel machinery is only used with proper ventilation					Concetion
with proper ventilation	ELE	CTRIC/	AL SAFE	ETY	
Electrical equipment is in good					
condition, grounded, protected from					
damage, and protected by a Ground					
Fault Circuit Interrupter.					
Lockout/tag out procedure is utilized					
whenever work is performed on					
electrical/mechanical equipment.					
EXCAVATIO	ONS,TI	RENCH	ES, CO	NFINED SPACES	
The Competent Person has inspected					
all trenches and excavations at least					
every shift.					
Excavation and trenches are					
adequately sloped, benched or shored					
based on soil conditions.					
Shoring systems have been approved					
by a Registered Professional					
Engineer, and shoring design. Soil stockpiles are placed no closer					
than two feet from the edge of					
excavations.					
Excavations/trenches are protected					
from water berms, drainage and/or					
pumps.					
Underground Service Alert and all					
entities potentially affected by the					
excavation have been notified					
Excavations and trenches are					
protected by fencing, barricades, ect.					
Surface encumbrances such as					
buildings, roads, ect. Have been					
protected.					
Ladders are located in excavation so					
that employees have access to them					
within 25 feet at all times.					
Underground utilities have been located, and exposed utilities are					
protected.					
All excavations and trenches have					
been evaluated for confined space					
conditions, and a checklist/permit is					
utilized for all entries.					



### SITE SAFETY INSPECTION

Inspection Item	Yes	No	N/A	<b>Corrective Actions</b>	Date of				
					Correction				
Feet are wearing properly secured									
lifelines and safety harnesses.									
PERSONAL PROTECTIVE EQUIPMENT									
Employees are utilizing proper									
personal protective equipment and									
respirators based on hazards, and									
equipment is in good condition.									
CHE	MICAL	STORA	GE/FII	RE SAFETY					
Chemicals, gases, and oily rags are									
stored properly in the appropriate									
labeled containers, and only with									
compatible materials.									
Stored compressed gas cylinders are									
secured and upright, with caps on.									
Chemicals are used with adequate									
ventilation									
Bonding/grounding is used during									
flammable liquid transfers.									
Compressed gas cylinders are moved									
with caps on, with dollies whenever									
possible.									
Flammable liquid storage areas have									
containment, and fire extinguishers									
within 25 feet.									
Hot work (welding, cutting) is performed with permit, fire watch,									
and extinguisher.									
Air monitoring equipment is									
maintained and calibrated daily, and									
monitoring is performed and									
documented properly.									
	HICIE	S/HFAV	VY FOI	JIPMENT					
Vehicles and heavy equipment are in		5 <b>/111</b> 2/1							
good condition, including: tires,									
mirrors, windshields, and back up									
alarms.									
Operators and ground personnel are	-	-							
cautious and maintain line of sight.									
Seat belts are used by operators									
Equipment is parked with									
buckets/forks in the down position.									
Drill rigs and cranes are moved with									
the boom down and rigging secured.									
All chains, slings, and hoists are in									
good condition and are inspected									
before use									



### ATTACHMENT B INFORMATION REGARDING SLIP, TRIP AND FALL HAZARDS/HEAT AND COLD STRESS SYMPTOMS

### APPENDIX E HEALTH AND SAFETY PLAN

SVE PILOT TESTING WORK PLAN

Awareness of how to prevent slips, trips and falls is an important part of our safety program.

### Slips, trips and falls from the same level:



Walking surfaces must be free of anything that can cause slipping, tripping or falling. This includes, water, oil or other fluids, hoses, electrical cords, tools, stock or scrap material, paper, etc. Surfaces should not have any sunken or raised pavement. Additionally, wood surfaces should be free of loose boards, holes, and protruding nails/screws. Watch for loose carpeting or mats.

Mats with waffle grids are excellent for drainage and provide extra safety in areas where oil, water, solvents or chemicals are present.

Shoes are a very important component in prevention. Shoes with heels and/or leather soles are particularly prone to slips. Remember when walking on slippery surfaces – bend your knees, lean slightly forward and slowly walk flat-footed with toes pointed outwards.

### Slips, trips and falls from different levels:

Equipment – Equipment surfaces can be very slippery. Take extra care when climbing on to or off of equipment. Assure you have secure footing when climbing or moving on equipment. A good rule to follow when climbing is to always have **three points of contact** (two feet and a hand or two hands and a foot) on either a good handhold or on solid footing before reaching/stepping up or down.

Ladders – Choose the right ladder for the job. Read and follow the manufacturer's instructions posted on the ladder. Always check the ladder prior to using. Open ladder completely (braces locked), place on firm footing, and never climb past the point of the ladder that the manufacturer had marked as the last point to stand on (usually, the next to the top step).

Fall Protection – Remember, when working at elevations above six feet off the ground, a fall protection lanyard and harness is required when standard guardrails are not available.

### THE COLD STRESS EQUATION

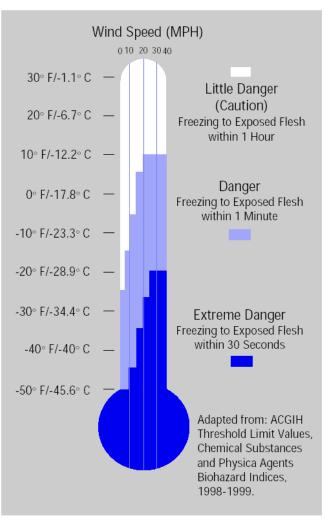


U.S. Department of Labor Occupational Safety and Health Administration

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0SHA 3 1998

When the body is unable to warm itself, serious coldrelated illnesses and injuries may occur, and permanent tissue damage and death may result. Hypothermia can occur when land temperatures are above freezing or water *temperatures* are below 98.6°F/ 37°C. Coldrelated illnesses can slowly overcome a person who has been chilled by low temperatures, brisk winds, or wet clothing.



### **FROST BITE**

### What Happens to the Body:

FREEZING IN DEEP LAYERS OF SKIN AND TISSUE; PALE, WAXY-WHITE SKIN COLOR; SKIN BECOMES HARD and NUMB; USUALLY AFFECTS THE FINGERS, HANDS, TOES, FEET, EARS, and NOSE.

### What Should Be Done: (land temperatures)

- Move the person to a warm dry area. Don't leave the person alone.
- Remove any wet or tight clothing that may cut off blood flow to the affected area.
- **DO NOT** rub the affected area, because rubbing causes damage to the skin and tissue.
- **Gently** place the affected area in a warm (105°F) water bath and monitor the water temperature to **slowly** warm the tissue. Don't pour warm water directly on the affected area because it will warm the tissue too fast causing tissue damage. Warming takes about 25-40 minutes.
- After the affected area has been warmed, it may become puffy and blister. The affected area may have a burning feeling or numbness. When normal feeling, movement, and skin color have returned, the affected area should be dried and wrapped to keep it warm. Note: If there is a chance the affected area may get cold again, do not warm the skin. If the skin is warmed and then becomes cold again, it will cause severe tissue damage.
- Seek medical attention as soon as possible.

### HYPOTHERMIA - (Medical Emergency)

### What Happens to the Body:

NORMAL BODY TEMPERATURE (98.6° F/37°C ) DROPS TO OR BELOW 95°F (35°C); FATIGUE OR DROWSINESS; UNCONTROLLED SHIVERING; COOL BLUISH SKIN; SLURRED SPEECH; CLUMSY MOVEMENTS; IRRITABLE, IRRATIONAL OR CONFUSED BEHAVIOR.

### What Should Be Done: (land temperatures)

- Call for emergency help (i.e., Ambulance or Call 911).
- Move the person to a warm, dry area. Don't leave the person alone. Remove any wet clothing and replace with warm, dry clothing or wrap the person in blankets.
- Have the person drink warm, sweet drinks (sugar water or sports-type drinks) if they are alert. **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) or alcohol.
- Have the person move their arms and legs to create muscle heat. If they are unable to do this, place warm bottles or hot packs in the arm pits, groin, neck, and head areas. **DO NOT** rub the person's body or place them in warm water bath. This may stop their heart.

### What Should Be Done: (water temperatures)

- Call for emergency help (Ambulance or Call 911). Body heat is lost up to 25 times faster in water.
- **DO NOT** remove any clothing. Button, buckle, zip, and tighten any collars, cuffs, shoes, and hoods because the layer of trapped water closest to the body provides a layer of insulation that slows the loss of heat. Keep the head out of the water and put on a hat or hood.
- Get out of the water as quickly as possible or climb on anything floating. DO NOT attempt to swim unless a floating object or another person can be reached because swimming or other physical activity uses the body's heat and reduces survival time by about 50 percent.
- If getting out of the water is not possible, wait quietly and conserve body heat by folding arms across the chest, keeping thighs together, bending knees, and crossing ankles. If another person is in the water, huddle together with chests held closely.

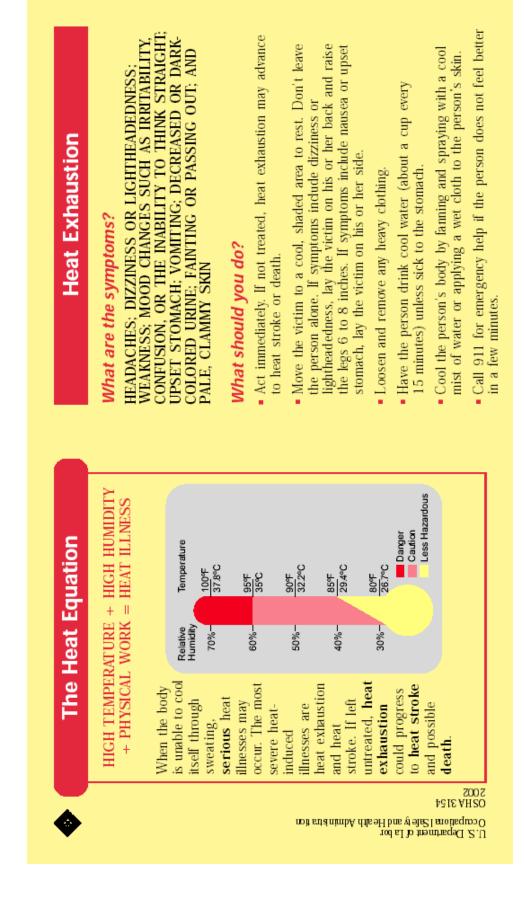
### How to Protect Workers

- Recognize the environmental and workplace conditions that lead to potential cold-induced illnesses and injuries.
- Learn the signs and symptoms of cold-induced illnesses/injuries and what to do to help the worker.
- Train the workforce about cold-induced illnesses and injuries.
- Select proper clothing for cold, wet, and windy conditions. Layer clothing to adjust to changing environmental temperatures. Wear a hat and gloves, in addition to underwear that will keep water away from the skin (polypropylene).
- Take frequent short breaks in warm dry shelters to allow the body to warm up.
- Perform work during the warmest part of the day.
- Avoid exhaustion or fatigue because energy is needed to keep muscles warm.
- Use the buddy system (work in pairs).
- Drink warm, sweet beverages (sugar water, sports-type drinks). Avoid drinks with caffeine (coffee, tea, or hot chocolate) or alcohol.
- Eat warm, high-calorie foods like hot pasta dishes.

### Workers Are at Increased Risk When...

- They have predisposing health conditions such as cardiovascular disease, diabetes, and hypertension.
- They take certain medication (check with your doctor, nurse, or pharmacy and ask if any medicines you are taking affect you while working in cold environments).
- They are in poor physical condition, have a poor diet, or are older.

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# **US EPA ARCHIVE DOCUMENT**

# Heat Stroke–A Medical Emergency

### What are the symptoms?

DRY, PALE SKIN WITH NO SWEATING; HOT, RED SKIN THAT LOOKS SUNBURNED; MOOD CHANGES SUCH AS IRRITABILITY, CONFUSION, OR THE INABILITY TO THINK STRAIGHT; SEIZURES OR FITS; AND UNCONCIOUSNESS WITH NO RESPONSE

### What should you do?

- Call 911 for emergency help immediately
- Move the victim to a cool, shaded area. Don't leave the person alone. Lay the victim on his or her back. Move any nearby objects away from the person if symptoms include seizures or fits. If symptoms include nausea or upset stomach, lay the victim on his or her side.
- Loosen and remove any heavy clothing.
- Have the person drink cool water (about a cup every 15 minutes) if alert enough to drink something, unless sick to the stomach.
- Cool the person's body by fanning and spraying with a cool mist of water or wiping the victim with a wet cloth or covering him or her with a wet sheet.
- Place ice packs under the armpits and groin area.

# How can you protect yourself and your coworkers?

- Learn the signs and symptoms of heat-induced illnesses and how to respond.
  - Train your workforce about heat-induced illnesses.
- · Perform the heaviest work during the coolest part of the day
- Build up tolerance to the heat and the work activity slowly. This usually takes about 2 weeks.
- Use the buddy system, with people working in pairs.
- Drink plenty of cool water, about a cup every 15 to 20 minutes.
- Wear light, loose-fitting, breathable clothing, such as cotton.
   Take frequent, short breaks in cool, shaded areas to allow
  - the body to cool down.
     Avoid eating large meals before working in hot environments.
    - Avoid caung targe means before working in not environments
       Avoid alcohol or beverages with caffeine. These make the
      - Avoid alcohol or beverages with callence. These make the body lose water and increase the risk for heat illnesses.

## What factors put you at increased risk?

- Taking certain medications. Check with your health-care provider or pharmacist to see if any medicines you are taking affect you when working in hot environments.
  - Having a previous heat-induced illness.
- Wearing personal protective equipment such as a respirator or protective suit.



### ATTACHMENT C DATA SHEETS FOR CHEMICALS THAT MAY BE ENCOUNTERED AT THE SITE

### APPENDIX E HEALTH AND SAFETY PLAN

SVE PILOT TESTING WORK PLAN

### Table C-1. Chemical Hazard Data for Compounds of Concern

Compound of Concern	and	Chemical d Physical Properties	Incompatibilities and Reactivities	Exposure Limits		Routes of Exposure	Symptoms of Exposure
Benzene	State:	colorless to light-yellow liquid Aromatic Odor	Reacts violently with	REL:	0.1 ppm Ca	Inhalation	Irritation to eyes, skin,
71-43-2	VP:	75 mm Hg	oxidants, nitric acid, sulfuric acid and		1 ppm STEL	Ingestion	nose, and the respiratory system. Nausea.
	BP:	176°F	halogens causing fire	PEL:	1 ppm	ingestion	Potential carcinogen.
	FRZ:	42°F	and explosion hazard.		5 ppm STEL		
	Sol (77°F):	0.07%	Attacks plastic and				
	FI.P:	12°F	rubber.				
	IP:	9.24 eV		TLV:	10 ppm		
	UEL (77°F):						
	LEL (77°F):	1.2%		IDLH:	3000 ppm		
	Sp.Gr:	0.88					
Chlorobenzene	State:	Colorless liquid Almond-like odor	The substance decomposes on heating,	REL:	N/A	Ingestion Skin Absortion	Irritation to the eyes and skin. Aspiration into the
	VP:	9 mmHg	on contact with hot			Inhalation	lung may acause
	BP:	270°F	surfaces or flames	PEL:	75ppm		chemical pneumonitis.
	FRZ:	-50°F	producing toxic and				May affect central
	Sol:	0.05%	corrosive fumes Reacts				nervous system. Defats
	FI.P:	82 °F	violently with strong oxidants causing fire and	TLV:	10 ppm		skin. May damage the liver and kidneys
	IP: UEL:	9.07 eV 9.6%	explosion hazard.				
	LEL:	9.6% 1.3%	Attacks rubber and	IDLH:	1000 ppm		
	Sp.Gr:	1.11	some plastic.				



### Table C-1. Chemical Hazard Data for Compounds of Concern

Compound of Concern	Chemical and Physical Properties		Incompatibilities and Reactivities	Exposure Limits		Routes of Exposure	Symptoms of Exposure
1,2-dichlorobenzene 95-50-1	State: VP:	Colorless to yellow liquid Aromatic odor 1 mm Hg	The substance decomposes on burning producing toxic and	REL:	50 ppm	Inhalation Skin Absorbtion Ingestion	Irritation eyes, nose; liver, kidney damage; skin blisters
	BP: FRZ: Sol:	357°F 1°F 0.01%		PEL:	50 ppm	Skin Contact	
	FI.P: IP:	151°F 9.06 eV	aluminium oxidants Attacks plastic and rubber.	TLV:	25 ppm		
	UEL: LEL: Sp.Gr (77°F	9.2% 2.2% <b>):</b> 1.3		IDLH:	200 ppm		
1,4-dichlorobenzene 106-46-7	State:	Colorless to white crystals Mothball-like odor	On combustion, forms toxic and corrosive fumesincludinghydrogen	REL:	N/A	Inhalation Skin Absorbtion Ingestion	Eye irritation, swelling periorbital (situated around the eye); profuse
	VP: BP: FRZ:	1.3 mmHg 345⁰F N/A		PEL:	75ppm	Skin Contact	rhinitis; headache, anorexia, nausea, vomiting; weight loss,
	Sol (77°F): FI.P: IP:	0.008% 150°F 8.98 eV		TLV:	10 ppm		jaundice, cirrhosis; in animals: liver, kidney injury; [potential
	UEL: LEL:	N/A 2.5%		IDLH:	150 ppm		occupational carcinogen]



### Table C-1. Chemical Hazard Data for Compounds of Concern

### NOTES:

DOCUMENT

EPA ARCHIVE

\* data taken from the Niosh Pocket Guide to Chemical Hazards (June, 1997), U.S. Department of Health and Human Services.
 NIOSH = National Institute for Occupational Safety and Health
 OSHA = Occupational Safety and Health Administration
 ACGIH = American Conference of Governmental Industria Hygienists

VP = vapor pressure at 68°F BP = boiling point at 1 atmosphere, °F FRZ = freezing point Sol = solubility in water at 68°F unless otherwise specified; units are % by weight (i.e., g/100 ml) FL.P = flash point NA = not available IP = ionization potential UEL = upper explosive (flammable) limit in air, % by volume (at room temperature unless otherwise specified) LEL = lower explosive (flammable) limit in air, % by volume (at room temperature unless otherwise specified) Sp.Gr = specific gravity at 68°F referenced to water at 39.2°F ( $4^{\circ}$ C) RGasD = relative density of gases referenced to air = 1 (indicates how many times a gas is heavier than air at the same temperature) Class IA Flammable Liquid = FL.P. below 73°F and BP below 100°F Class IB Flammable Liquid = FL.P. below 73°F and BP at or above 100°F mm Hg = millimeters of mercury atm = atmosphere <sup>o</sup>F = degrees Fahrenheit

REL = NIOSH recommended exposure limit; unless otherwise noted, RELs are time-weighted average (TWA) concentrations for up to a 10-hour workday during a 40-hour workweek PEL = OSHA permissible exposure limit; unless otherwise noted, PELs are time-weighted average (TWA) concentrations for up to an 8-hour workday during a 40-hour workweek TLV = ACGIH threshold limit value IDLH = immediately dangerous to life and health STEL = short-term exposure limit (15-minute TWA) ppm = parts per million Ca = potential occupational carcinogen LFC = lowest feasible concentration N.D. = not determined

Inh = inhalation Abs = skin absorption Ing = ingestion Con = skin and/or eye contact



### ATTACHMENT D EXPOSURE/INJURY INCIDENT REPORT

### APPENDIX E HEALTH AND SAFETY PLAN

SVE PILOT TESTING WORK PLAN



This report must be completed and submitted to the H&S staff within 24 hours of the incident. □ Verbal notification must be made immediately in case of serious accident or injury. Complete Pages 1 & 3 for all incidents, and appropriate section(s) of Page 2.

GENERAL INFORMATION: (complete for	or all incidents)
Report Completed by:	Title:
Date Completed:	Date Submitted:
	Project Number:
Project Location:	
Date & Time of Accident:	Work Activity:
Where Occurred:	Work Activity: □ On-Site □ Off-Site
Type of Incident: □ Personal Injury/Exposure	Property Damage/Fire     Vehicle Accident
XDD Employee(s) Involved:	
Subcontractor(s)/ Visitor(s)/ Third parties Inve	olved:
<b>DESCRIPTION OF INCIDENT:</b> Describe	e any equipment or materials involved or damaged,
or chemicals/hazardous materials involved or r	eleased. Attach additional pages and drawings as
necessary.	
Diagram of Accident:	
NOTIFICATION MADE: (date & time):	
Project Manager:	Health & Safety:
Client:	Other (specify):
Agency Responding:	Report No
Agency Responding:	Report No
Photos taken? (Y/N, sent to):	
WITNESSES: List below. Attach signed states	ments.
Name:	Phone Number:
Employer:Ad	ddress:
Name:	Phone Number:
Employer: Ac	ddress:



### ACCIDENT/LOSS REPORT

ACCIDENT INVESTIGATION: Direct cause of accident: Contributing Factors: (Mark "Yes" and explain ALL factors which contributed to the incident)											
								Item	Yes	No	Explain
								Safety policies/regulations-lack of			
understanding of policies or regulations. List											
applicable.											
Job hazard assessment-hazard assessment not											
preformed, or hazards not identified.											
Safe work procedures-not developed for the											
operation or not adequate.											
Training- employees did not have the											
appropriate or updated training for the											
operation.											
Communication-hazards, safe work											
procedures, and/or work rules not											
communicated to workers.											
Enforcement- work rules not enforced											
adequately or consistently.											
Employee behavior- employee inattention,											
speeding, or horseplay involved in the incident.											
Housekeeping- housekeeping was not											
performed initially, regularly, and/or											
thoroughly.											
Maintenance- equipment not maintained											
regularly or adequately.											
Proper Equipment- the proper tools or											
equipment were not provided or not used for											
the operation.											
Equipment or material failure- equipment or material that failed contributed to incident.											
Personal Protective Equipment- not provided, not used, or not used properly by individual											
Personal physical factors- such as strength,											
physical condition, and prior injuries were a											
factor.											
Drugs or alcohol- drug or alcohol use may											
have been a factor in the incident.											
Environmental or chemical factors-vapors,		1									
dust, noise, glare, or heat/cold were a factor.											
Inspections- site or equipment inspections not											
preformed initially, regularly, and/or											
thoroughly											
Other:											