

Approved

**Enbridge Line 6B MP 608  
Marshall, MI Pipeline Release  
2013 Source Area Remedial Investigation Work Plan**

**Prepared for Michigan Department of Environmental Quality**

**Enbridge Energy, Limited Partnership**  
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## LIST OF ACRONYMS

COC	Chain of Custody
Enbridge	Enbridge Energy, Limited Partnership
ft bgs	Feet Below Ground Surface
GPS	Global Positioning System
Line 6B	The pipeline owned by Enbridge Energy, Limited Partnership that runs just south of Marshall, Michigan
LNAPL	Light Non-aqueous Phase Liquid
MDEQ	Michigan Department of Environmental Quality
ml	Milliliter
MP	Mile Post
NFA	No Further Action
Part 201	Part 201 of Michigan's Act 451 of 1994, as amended
PID	Photoionization Detector
PNAAs	Polynuclear Aromatic Hydrocarbons
PTS	PTS Labs of Santa Fe, California
PVC	Polyvinyl Chloride
QAPP	Quality Assurance Project Plan
RAWP	Response Action Work Plan
RI	Remedial Investigation
SARP	Source Area Response Plan
SOP	Standard Operating Procedure
TOC	Total Organic Carbon
USCS	Unified Soil Classification System
U.S. EPA	United States Environmental Protection Agency
UV	Ultraviolet
VOCs	Volatile Organic Compounds

## 1.0 INTRODUCTION

On July 26, 2010, Enbridge Energy, Limited Partnership (Enbridge) discovered a release of crude oil from the pipeline owned by Enbridge that runs just south of Marshall, Michigan (Line 6B) in the vicinity of its pump station. The crude oil was released below grade level via a break in Line 6B, emerged onto the ground surface, flowed over land following the natural topography into Talmadge Creek, and proceeded to flow downstream into the Kalamazoo River. Following the release, Enbridge performed response activities under the direction of the United States Environmental Protection Agency (U.S. EPA) and the Michigan Department of Environmental Quality (MDEQ) to remove oil from the system and respond to the release.

The area in which the break in the pipeline is located, the Source Area, is comprised of an approximate 5-acre parcel, adjacent to the Enbridge Line 6B Mile Post (MP) 608 pipeline release location, and divided into four Sub-Units designated as A-1 through A-4 (*Figure 1*).

### 1.1 Previous Investigations

Response activities at the Source Area were conducted in accordance with the *Source Area Response Plan* (Enbridge, 2010a) approved by the U.S. EPA on August 17, 2010 (SARP). Results of these activities were presented in the *Source Area Response Completion Report* (Enbridge, 2010b) submitted to the U.S. EPA on September 2, 2010. The *Remedial Investigation Work Plan Division A1 through A4* (Enbridge, 2010c) submitted to the MDEQ (formerly the Michigan Department of Natural Resources and Environment) on September 28, 2010 was developed to assess response activities and determine gaps in the analytical data at the Source Area with the presentation and interpretation of the data derived presented in the *Remedial Investigation Source Area (Divisions A1 through A4)* (Enbridge, 2011a) submitted to the MDEQ on March 21, 2011.

To further investigate residual oil impact in Sub-Unit A-1, the *Investigation Work Plan for Source Area (A1)* (Enbridge, 2012a) approved by the U.S. EPA on May 21, 2012 was developed. A courtesy copy was also submitted to the MDEQ. Field activities were performed from June 7 through and including June 8, 2012. Results from this investigation were presented in the memorandum *Summary of Findings for the Investigation Work Plan for Source Area (A1)* (Enbridge, 2012b) submitted to the U.S. EPA on July 6, 2012. Additional investigation activities (soil borings, temporary well installation, and groundwater sampling) in Sub-Units A-2 and A-3

were performed from July 18 through and including August 3, 2012 as an extension of the *Investigation Work Plan for Source Area (A1)* (Enbridge, 2012a).

At the request of Enbridge, the MDEQ and its professional services contractor, Weston Solutions, Inc., completed a review of available information from these previous investigations and response activities completed at the Source Area Sub-Units A-1 to A-4. The purpose of the July 25, 2012 review was to identify potential data gaps, recognized environmental conditions that may remain, and potentially unacceptable risks that may need to be addressed to comply with Part 201 of Michigan's Act 451 of 1994 as amended (Part 201).

The MDEQ review identified several areas of concern within each Sub-Unit where MDEQ asserted the nature and extent of submerged oil, residual oil, and the associated hazardous substance constituents were not clearly defined. In particular, the MDEQ expressed concerns regarding the following:

- Potential for submerged and residual oil along the pipeline corridor,
- A linear area in Sub-Units A-1 and A-2, and
- An isolated area in Sub-Unit A-3.

The activities in the *Response Action Work Plan for the Source Area* (Enbridge, 2012c) approved by the MDEQ on September 14, 2012 (RAWP) were proposed to address these areas of concern, as were the additional investigation activities performed in July through early August 2012 following the methods of the *Investigation Work Plan for Source Area (A1)* (Enbridge, 2012a) approved by the U.S. EPA on May 21, 2012.

RAWP activities were completed by September 27, 2012 and subsequently presented in the *Report of Findings for Response Action at the Source Area* (Enbridge, 2013) submitted to the MDEQ on January 25, 2013. The report found that concentrations of benzene and xylenes in two isolated areas (SSSA0025R0162 and SSSA0025R0154, respectively) of Sub-Unit A-1 indicated further investigation and evaluation would be necessary.

This Remedial Investigation (RI) work plan was developed to address these remaining residual impacts. The work plan was originally submitted to MDEQ for approval on April 19, 2013. A review was conducted by MDEQ and comments were supplied to Enbridge on April 29, 2013. Enbridge modified the work plan according to comments provided and resubmitted it to MDEQ as "Approved with Modifications" on May 6, 2013. In meetings following this submission, and as

detailed in a July 26, 2013 letter, MDEQ expressed some issues with the responses provided by Enbridge and indicated that the work plan was considered to be “Disapproved” by the Agency.

Excavation and line replacement activities were performed from May 15 through and including May 29, 2013, after submittal of the modified RI work plan. During the excavation, sheen, visible oil, and oil globules was observed by Enbridge and MDEQ oversight personnel at eight areas (MDEQ Observations A through H, *Figure 2, Sheet 2*) along the length of the excavation.

MDEQ Observations A through H compiled on May 20 and 21, 2013 were as follows:

- A – Visible oil and sheen on soil (2 to 4 feet below ground surface (ft bgs), visible oil and sheen on water within the excavation,
- B - Visible oil and sheen on soil (2 to 4 ft bgs) and odor present,
- C - Sheen on water within the excavation,
- D - Sheen on water within the excavation,
- E - Visible oil and sheen on soil at bottom of excavation (10 ft bgs),
- F – Sheen on surface water outside of the excavation,
- G - Visible oil and sheen on soil at bottom of the excavation (10 ft bgs) (possible slough), and
- H - Sheen and oil globules on water within the excavation.

Investigation activities associated with these eight additional areas have been proposed as part of this resubmitted document and are described in *Section 2.3* of this work plan.

## 1.2 Objective

In ongoing discussions regarding the Source Area, MDEQ has indicated the presence of data gaps within the current data set. In a meeting on May 15, 2013, MDEQ identified three distinct locations that warrant further investigation as follows:

- The pipeline corridor containing Line 6B and the adjacent Vector Pipeline (additional information to determine the potential for preferential pathways in the pipeline corridor),
- The location of the July 26, 2010 Line 6B pipeline break (vertical groundwater profiling to determine the vertical extent of impacts), and
- The locations of isolated exceedances of soil criteria (additional information to determine the extent of impacts).

The objective of this work plan is to gather sufficient information to define the nature and extent of remaining petroleum impacts in the identified locations at the Source Area. Information collected will be used to identify exposure pathways; evaluate the risk to human health and the environment posed by remaining residual oil impact in this section of the overbank area; assist in the characterization and evaluation of the ecological sensitivity of this area; and assess compliance with Part 201 rules and regulations.

The proposed investigation activities in this work plan are being performed for the MDEQ under the Administrative Consent Order and Partial Settlement Agreement entered *In the Matter of Enbridge Energy Partners, L.P., and Enbridge Energy, Limited Partnership*, proceedings under the Michigan Natural Resources and Environmental Protection Act, 1994 PA 451, as amended, MCL 324.101 et seq. signed November 1, 2010 (MDEQ, 2010).

Information obtained from these activities will be used to propose additional investigation and/or response activities, as necessary, and in the preparation of a No Further Action (NFA) Report for the Source Area and surrounding properties. Data gaps recognized through implementation of these investigations and by the evaluation of field observations and analytical results from this and previous investigations will be further investigated during this RI. Information collected during previous investigations at these locations will also be incorporated into the NFA Report.

### **1.3 Remedial Investigation Field Approach**

Implementation of the RI will be completed in Sub-Units A-1, A-2, A-3, and A-4 of the Source Area, the surrounding area, and Sub-Unit A-5 of Talmadge Creek (*Figure 1*). Work components in the field will include the following tasks:

- Use of global positioning system (GPS) to locate pre-determined sample locations,
- Visual identification and location of any remaining residual oil impact (e.g., oil globules, staining, etc.),
- Installation of soil borings at pre-determined and field-selected locations,
- Installation and development of temporary and permanent monitoring wells,
- Survey of the horizontal and vertical coordinates of newly installed permanent monitoring wells,
- Collection of soil and groundwater samples for laboratory analysis,
- Collection of groundwater elevation data from all permanent monitoring wells, and

- Removal of temporary wells and abandonment of soil borings.

The following paragraphs present the anticipated sequencing of field activities, the organization of RI sampling teams, media to be sampled, and sampling methods.

Field work will be generally sequenced at each of the proposed boring locations shown on *Figure 2* as follows:

1. Delineation of areas of previous investigations where analytical results exceeded Part 201.
2. Installation of soil borings, collection of soil cores, core logging, and soil sample collection.
3. Use of GPS units to collect horizontal coordinates of all field-selected boring locations.
4. Installation of temporary and permanent monitoring wells, well purging, and low-flow groundwater sample collection.
5. Survey of the horizontal coordinates of newly installed temporary wells.
6. Survey of the horizontal and vertical coordinates of newly installed permanent monitoring wells.
7. Temporary well removal and soil boring abandonment.

The RI will be conducted using a team approach in which teams will be assigned specific subsections of the investigation area. Team organization and responsibilities include:

- Team Lead/Geologist: Supervises field work at site, directs team, makes field decisions, measures settled core recovery, and logs soil cores,
- Yuma Operator: Locates pre-determined soil boring locations, captures geo-reference (horizontal coordinates) data of pre-determined and field-selected borings, and takes photographs (general locations and cores),
- Coring Team: Utilizes equipment to collect soil cores and measures field core recovery,
- Core Preparer/Sampler: Retrieves core from Coring Team, cuts core, collects field ultraviolet (UV) light screening information, and collects appropriate samples from core for laboratory analysis, and
- Groundwater Sampler: Measures depth to groundwater in all monitoring wells, and completes groundwater sampling utilizing low-flow techniques.

Actual team responsibilities will be dependent on field conditions and at the discretion of the Team Lead/Geologist. All team activities will be coordinated with MDEQ personnel to allow for oversight and the collection of split samples.

Activities and observations made by each team will be documented in log books, field forms, and/or electronic capture. Field sampling locations will be geo-referenced for incorporation into the comprehensive Geographic Information System that exists for this project.

## **2.0 REMEDIAL INVESTIGATION OF MEDIA**

This section describes the methods and approaches that will be used to collect representative samples from soil and groundwater from distinct locations identified by MDEQ. For this RI, investigation methods will include visual observation of the soil cores, field screening, and quantitative analysis. Samples will be collected using Standard Operating Procedures (SOPs) provided in the *Sampling and Analysis Plan* (Enbridge, 2011b) approved by the MDEQ on August 30, 2011 and the *Quality Assurance Project Plan (QAPP)* (Enbridge, 2011c) approved by the MDEQ on August 19, 2011 (QAPP). If field observations and/or analytical results indicate that additional investigative activities are required, those additional activities will be performed following the methods and SOPs referenced in this work plan. The following sections describe the rationale behind the collection of additional data by media type.

### **2.1 Soil Boring Installation**

#### **2.1.1 Step-out Borings**

Additional soil samples are proposed for collection from pre-determined locations in the investigation area where historic information (e.g., MDEQ review, response activities, etc.) indicates a concern (*Figure 2*).

Proposed soil boring locations are presented in *Figure 2* as step-out borings to those soil borings that exhibited exceedances of Part 201 criteria. Step-out soil borings are proposed as follows:

- Four borings at SSSA0025R0154: Total xylenes exceeded Part 201 criteria at 1.5 ft bgs,

- Three borings at SSSA0025R0162: Benzene exceeded Part 201 criteria at 1.0 ft bgs. One of these borings will also have a permanent monitoring well installed to screen for light non-aqueous phase liquid (LNAPL) (*Section 2.3*),
- Three borings at ESTC0000R020: Several volatile organic compounds (VOCs) exceeded Part 201 criteria at 3.5 ft bgs, and
- Three borings at ESTC0000R029: Several VOCs exceeded Part 201 criteria at 3.5 ft. bgs.

Note that locations with only three proposed step-out borings are delineated by existing borings. The spacing between soil borings for characterizing petroleum impacts will be dependent on field conditions and decided in the field by the Team Lead/Geologist. Field decisions on soil boring locations and number will be biased toward visual observations of residual oil, areas with the potential for residual oil impact, and areas of previous investigations. Proposed locations will be adjusted to maintain a safe working distance from the pipelines.

Installation of step-out borings and soil sample analysis, as well as potential groundwater sampling via temporary monitoring wells, will delineate the horizontal and vertical extent of impact at these locations. Permanent monitoring wells will be installed for the purposes of groundwater sampling, groundwater flow determination, and, if present, LNAPL screening (See *Section 2.2*).

### **2.1.2 Soil Coring**

Soil cores will be collected utilizing the direct push sampling method by a track mounted or marsh buggy Geoprobe® drilling rig, a hand probe, or other similar device. The drilling method for each location is dependent on access and site conditions, and will be completed in accordance with *SOP EN-301* (Enbridge, 2011b) and *SOP EN-305* (Enbridge, 2011b). Soil cores will be collected in 4-foot long intervals to the desired termination depth. The termination depth for each soil boring will be at a depth sufficient to define the vertical extent of contamination or to refusal. If visual or analytical data indicate that impact extends below the termination of the boring, alternate soil boring advancement techniques may be implemented to achieve the objectives of vertical delineation.

### **2.1.3 Soil Logging and Screening**

After the soil core is removed from the borehole, it will be opened, visually evaluated, screened with a photoionization detector (PID), photographed under white and UV light, visually screened

under UV light within a darkroom, measured for recovery, and logged using the Unified Soil Classification System (USCS). Discrete soil samples will be collected from each core for subsequent laboratory analysis from the interval with the greatest potential for petroleum hydrocarbon impact. Soil samples will be collected immediately following core opening and visual and UV light observations.

The observations made during this process for the presence of petroleum hydrocarbon impacts, UV fluorescence, and lithology will be recorded and the real-time data will be used to assist the Team Lead/Geologist in determining if further advancement of the soil boring and/or additional borings is required to achieve the RI objectives and lateral delineation. Lateral delineation will continue until the area is sufficiently characterized. If analytical results indicate further delineation is required, additional soil borings will be advanced and samples will be collected.

In the event that LNAPL, in the form of visible oil and/or UV-fluoresced oil is found in soil borings at the Source Area, up to three additional soil cores may be collected from the impacted location and submitted to PTS Labs of Santa Fe, California (PTS) for analysis of petro-physical parameters including UV photography, pore saturation (*Recommended Practices for Core Analysis (API RP40)* (API, 1998)), and LNAPL mobility testing (*Standard Method for Centrifuge Moisture Equivalent of Soils (ASTM D425-88)* (ASTM, 2008) (*Attachment A*)).

#### **2.1.4 Soil Sampling for Analysis**

Up to three soil samples will be collected for chemical analysis from each field sampling location. As a general guideline, samples will be collected as follows:

- One sample will be collected from above the water table that shows the highest potential for petroleum hydrocarbon impact (visual or UV observation). If no petroleum hydrocarbon impact is observed, the sample will be collected from the interval immediately above the water table (capillary fringe). Additional samples may be collected at the discretion of the Team Lead/Geologist based on lithology, adjacent borings, etc.
- To define vertical extent, one sample, that shows no petroleum hydrocarbon impact (visual or UV observation) will be collected from the interval directly below the interval that exhibits the highest potential for impact.
- At the discretion of the Team Lead/Geologist, one sample may also be collected from below the water table for subsequent laboratory analysis.

Soil sampling will be conducted in accordance with *SOP EN-301* (Enbridge, 2011b) and the QAPP.

Per RRD Operational Memorandum 2: “Neither soil nor water sample analyses methods are appropriate for comparison of saturated “soils” samples to generic soil or groundwater cleanup criteria” (MDEQ, 2004). As such, if saturated subsurface conditions are present, a temporary well will be installed and a groundwater sample will be collected and analyzed. As suggested in the Draft for Discussion “Enbridge issues and Potential Resolution Items” document provided by MDEQ for discussion at the 8/29/2013 meeting, in areas that exhibit evidence of NAPL impacts (e.g. fluorescence, visual oil, etc.), Enbridge will continue to collect water-saturated soil samples for comparison to the “dry” soil criteria. If there are compounds that exceed the “dry” soil criteria, additional risk assessments and/or presumptive remedies could be employed.

### **2.1.5 Soil Analyses**

Discrete soil samples will be collected from the core and sent to the laboratory for analysis. Soils will be analyzed for residual oil-related parameters including polynuclear aromatic hydrocarbons (PNAs), VOCs, and the select metals beryllium, molybdenum, nickel, and vanadium. Hold times and sample preservation methods will be followed as presented in the QAPP. Additional analyses (e.g., synthetic precipitation leaching procedure analysis (per *Attachment B* and RRD Operational Memorandum 2 Attachment 2), total organic carbon (TOC) content, bulk density, etc.) may also be conducted as appropriate.

The soil samples will be transported under chain of custody (COC) control in accordance with *SOP EN-102* (Enbridge, 2011b) to ALS Laboratory Services in Holland, Michigan for analyses.

In the event that LNAPL is found in soil borings at the Source Area and additional soil cores are collected, the cores will be sealed and frozen prior to submittal under COC control to PTS for analyses in accordance with the methods described in *Attachment A*. The *Standard Method for Centrifuge Moisture Equivalent of Soils (ASTM D425-88)* (ASTM, 2008) and *Recommended Practices for Core Analysis (API RP40)* (API, 1998) analyses will provide results of potential LNAPL mobility and percent residual LNAPL saturation within the collected soils.

## **2.2 Groundwater**

Groundwater will be sampled through the use of temporary and permanent monitoring wells. Previous exceedances of MDEQ groundwater cleanup criteria, current visual and/or UV

observations of residual oil below the observed depth of groundwater, and field observation of residual oil in the groundwater, if present, will be used to determine locations of the proposed wells and well screen intervals. In addition, vertical profiling of the groundwater will be utilized for the optimal placement of screened intervals. Locations of the proposed temporary and permanent monitoring wells are included in *Figure 2*. Benzene exceeded Part 201 criteria in temporary wells at SBSA0125 and SBSA0139. Permanent monitoring wells are proposed downgradient of these two wells based on groundwater flow maps presented in *Remedial Investigation Source Area (Division A1 through A4)* (Enbridge, 2011a) submitted to MDEQ on March 21, 2011.

Installation of additional permanent monitoring wells within the pipeline corridor will be used to further evaluate groundwater flow and the vertical and horizontal extent of residual oil impact within the corridor, and adjacent to and downgradient of the Line 6B release location.

Groundwater elevation measurements will be collected from all existing Source Area monitoring wells and groundwater flow directions will be determined prior to soil boring and well installations to assure that proposed monitoring wells are optimally located to achieve project objectives.

### **2.2.1 Well Installation**

Paired (deep/shallow) temporary monitoring wells will be installed at the proposed locations shown on *Figure 2*. Samples will be collected from these temporary wells and analyzed to vertically profile groundwater quality and determine potential impact from the residual oil.

The temporary wells will be constructed of 1-inch diameter, schedule 40 polyvinyl chloride (PVC) riser with 10-slot (0.010-inch) PVC well screen and an end cap. A deep well screen (1-foot in length) will be set at the bottom of the borehole (up to 10 feet bgs). A shallow well screen (5-foot in length) will be set to straddle the water table and capillary fringe. If field conditions require (i.e., limited or no natural collapse), a sand pack will be placed around the screen and bentonite chips will extend from the top of the filter pack to ground surface so that surface water does not enter the well from above the screened interval. Well screen lengths and intervals will be adjusted to the zone of greatest impact based on field observations of subsurface conditions.

Field procedures for temporary well installation will include:

- Measure the depth to groundwater in the borehole once the boring termination is reached.
- Assemble and install the deep temporary well.
- Assemble and install the shallow temporary well in a borehole adjacent to the deep well so that the PVC screen straddles the water table and capillary fringe. Field screening the borehole cuttings with a PID will be performed to aid in determining the shallow well screen interval. If depth to groundwater is less than 1 foot, the well screen will be set a minimum of 0.5 ft bgs.
- If necessary (i.e., limited or no natural collapse), place a sand/gravel pack in the annular space between screen and borehole to a level of approximately one to two feet above the top of screen. If the depth to groundwater is less than 1 ft bgs a sand/gravel pack extending only several inches above the top of the screen will be installed. A bentonite seal will be placed on top of the sand/gravel pack to grade.
- Purge temporary wells until visibly clear of sediment or until dry. If a temporary well purges dry, the well will be allowed to recharge for 12 to 24 hours prior to groundwater sample collection.

Once groundwater samples are collected, the temporary wells will be removed and the borehole will be backfilled with bentonite pellets to the ground surface.

Permanent monitoring wells will be constructed and installed per the approved *SOP EN-401* (Enbridge, 2011b). Depending on analytical results, additional temporary and/or permanent monitoring wells may prove necessary. A nested trio of permanent monitoring wells will be installed at the proposed locations to assess the horizontal and vertical nature of potential groundwater impact in the pipeline corridor and adjacent to the Line 6B break. Monitoring well screens will be set at 15 to 20 ft bgs to monitor groundwater below the Line 6B pipeline, 7 to 12 ft bgs to monitor groundwater at the level of the Line 6B pipeline, and a screen will be set straddling the water table. If depth to groundwater is less than 1 foot, the well screen will be set a minimum of 0.5 ft bgs.

Newly installed permanent wells will be developed per the approved *SOP EN-402* (Enbridge, 2011b) prior to groundwater sampling.

Newly installed permanent monitoring wells will be surveyed to the Michigan State Plane Coordinate System and the North American Datum 1983 international feet. The top of casing

elevation will be determined to the nearest 0.01 foot, and the x, y location and ground surface elevation will be determined to the nearest 0.1 foot.

### **2.2.2 Groundwater Sampling for Analysis**

Groundwater samples will be obtained from all existing and newly installed wells utilizing low flow sampling methods in accordance with the approved *SOP EN-406* (Enbridge, 2011b). As part of the groundwater sampling technique, each well will be evaluated for the presence of LNAPL with an oil-water interface probe. Static water levels in permanent monitoring wells will be measured prior to sample collection. These measurements will be utilized to interpret current and seasonal horizontal and vertical groundwater flow. The intake depth of sample tubing used in groundwater sampling will be adjusted based on the observed depth of impacted soil and the height of the water column in the monitoring well.

### **2.2.3 Groundwater Analysis**

Groundwater samples will be transported under COC control to ALS Laboratory Services in Holland, Michigan for analyses. The samples will be analyzed for PNAs, VOCs, and the select metals beryllium, molybdenum, nickel, and vanadium. Hold times and sample preservation methods are presented in the QAPP (Enbridge, 2011c).

If LNAPL is encountered during groundwater sampling in sufficient volume (150 milliliters (ml) minimum per PTS), up to three LNAPL samples will be submitted to PTS for hydrocarbon characterization and fluid property analyses, including specific gravity (*Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer (ASTM D1481-12)* (ASTM, 2012a), viscosity (*Standard Test Method Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity) (ASTM D445-12)* (ASTM, 2012b), and surface tension (*Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method (ASTM D971-12)* (ASTM, 2012c), to evaluate LNAPL behavior in the subsurface.

## **2.3 Additional Monitoring Wells**

Groundwater will be monitored, sampled, and screened for LNAPL through the use of additional permanent monitoring wells. Field observations of residual oil in the Line 6B replacement excavation as summarized by MDEQ oversight personnel will be used to determine locations for these additional monitoring wells. Proposed locations of these permanent monitoring wells are included in *Figure 2*. Each location is proposed downgradient of these observations based on

groundwater flow maps presented in *Remedial Investigation Source Area (Division A1 through A4)* (Enbridge, 2011a) submitted to MDEQ on March 21, 2011.

### **2.3.1 Additional Monitoring Well Installation**

Permanent monitoring wells will be installed at the proposed locations shown on *Figure 2*. Samples will be collected from these wells and analyzed to determine groundwater quality and potential impact from the residual oil. Vertical profiling of the groundwater will be conducted to determine the presence and vertical distribution of Line 6B crude oil contaminants and to assist in the optimal placement of screened intervals. Vertical profiling will be performed via direct-push methods utilizing a Geoprobe® Screen Point 22 Groundwater Sampler as described in *Geoprobe® Screen Point 22 Groundwater Sampler Standard Operating Procedure Technical Bulletin No. MK3173* (Geoprobe Systems®, 2010) (*Attachment C*). Alternate sampling equipment (e.g., ¾-inch to 1-inch PVC screens, Geoprobe® Screen Point 16 Groundwater Sampler, etc.) may be utilized based on subsurface conditions to optimize groundwater profiling.

Permanent monitoring wells will be constructed and installed per the approved *SOP EN-401* (Enbridge, 2011b). Stainless-steel well screens will be set to straddle the water table and capillary fringe with a minimum of 1 foot of screen above the groundwater table. If depth to groundwater is less than 1 foot, the well screen will be set a minimum of 0.5 ft bgs. Bottom of the well screen will correspond to the bottom of the Line 6B replacement excavation (minimum of 10 ft bgs).

Newly installed permanent wells will be developed per the approved *SOP EN-402* (Enbridge, 2011b) prior to groundwater sampling.

### **2.3.2 Groundwater Sampling for Analysis**

Groundwater samples will be obtained from the newly installed wells utilizing low flow sampling methods in accordance with the approved *SOP EN-406* (Enbridge, 2011b). As part of the groundwater sampling technique, each well will be evaluated for the presence of LNAPL with an oil-water interface probe. Static water levels in permanent monitoring wells will be measured prior to sample collection. These measurements will be utilized to interpret current and seasonal horizontal and vertical groundwater flow. The intake depth of sample tubing used in groundwater sampling will be adjusted based on the observed depth of impacted soil and the height of the water column in the monitoring well.

### 2.3.3 Groundwater Analysis

Groundwater samples will be transported under COC control to ALS Laboratory Services in Holland, Michigan for analyses. The samples will be analyzed for PNAs, VOCs, and the select metals; beryllium, molybdenum, nickel, and vanadium. Hold times and sample preservation methods are presented in the QAPP.

If LNAPL is encountered during groundwater sampling in sufficient volume (150 ml minimum per PTS), up to three LNAPL samples will be submitted to PTS for hydrocarbon characterization and fluid property analyses, including specific gravity (*Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer (ASTM D1481-12)* (ASTM, 2012a), viscosity (*Standard Test Method Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity) (ASTM D445-12)* (ASTM, 2012b), and surface tension (*Standard Test Method for Interfacial Tension of Oil Against Water by the Rind Method (ASTM D971-12)* (ASTM, 2012c), to evaluate LNAPL behavior as associated with groundwater.

### 2.3.4 Screening

Monitoring wells installed will be screened for LNAPL (per *Section 2.3.2* and *Section 2.3.3*) during groundwater sampling events. Based on the identification and delineation of the nature and extent of groundwater impact related to the Line 6B crude oil release, Enbridge will implement a groundwater monitoring strategy that complies with applicable MDEQ regulatory guidelines and regulations as well as industry standards. The groundwater monitoring strategy will be implemented to assess the risk to human health and the environment and to provide information necessary to develop a site closure strategy as applicable.

## 2.4 Survey of Monitoring Wells

The locations of all temporary monitoring wells will be recorded using a sub-meter accuracy GPS unit.

The locations of all newly installed permanent monitoring wells will be recorded using a sub-meter accuracy GPS unit and surveyed to the Michigan State Plane Coordinate System and the North American Datum 1983 international feet. The top of casing elevation will be determined to the nearest 0.01 foot, and the x, y location and ground surface elevation will be determined to the nearest 0.1 foot.

### 3.0 DATA EVALUATION AND REPORTING

As part of the data evaluation, the analytical data will be compiled and compared to relevant Part 201 criteria, Part 31 criteria, or screening levels. All lines of evidence, as observed and recorded by Enbridge personnel, and other readily available evidence, will be utilized during the evaluation of risk to both human health and the environment. The mobility of remaining residual oil will also be evaluated. Locations where criteria or screening levels are exceeded will be identified and exposure pathways where applicable criteria are exceeded will also be identified. Response recommendations will be evaluated with regard to the ecological sensitivity of the area. The data will be reviewed to identify data gaps. Based upon this evaluation, if data gaps are present they will either be addressed within the context of additional investigative activities or via remedial actions or engineering controls (e.g. further excavation, site restrictions, post closure plans/agreements, etc.).

The results of the work specified in this work plan and subsequent data evaluation activities, will be presented in a Remedial Investigation Report. The components will include, at a minimum: an introduction, objectives, methods, sample location map, cross sections (as appropriate), soil boring logs with monitoring well construction details, groundwater sampling logs, figures showing groundwater flow and extent of Line 6B crude oil impact (as appropriate), data tables, sample location coordinates, a discussion of the investigation-derived observations and results including nature and extent of Line 6B crude oil impacted soil and groundwater, recommendations, and a summary and conclusions section.

The methods section will present methods used or refer to standard methods or methods previously approved for this project. Deviations from the work plan will be identified and reasons for the deviations will be presented. For example, sample locations may need to be adjusted based on field conditions. Deviations that require an addendum to the work plan (e.g., bedrock investigation, etc.) will be proposed to the MDEQ for approval prior to implementation. Generic Part 201 Residential Cleanup Criteria will be used to evaluate the unsaturated soil, and groundwater data. Residential criteria will be used as a baseline for evaluation and may not be appropriate cleanup criteria at select areas. The laboratory data reports will be included as an attachment to the report in the form of a data disk.

The results of the work specified in this work plan and data evaluation activities will also be utilized in the development of a NFA Report as described in Part 201, Section 20114d and following the *Request for DEQ Review of No Further Action (NFA) Report* form.

#### 4.0 SCHEDULE

Field activities will be scheduled for completion within two weeks of MDEQ approval of this work plan and contingent on site conditions and access. Dates for the RI activities will be provided to the MDEQ to coordinate oversight activities. Should weather or Line 6B restoration activities create an obstacle to completion of the field activities, MDEQ will be notified of such conditions and their impact upon completion. When work activities are shut down due to weather, activities will begin again following site-specific health and safety procedures.

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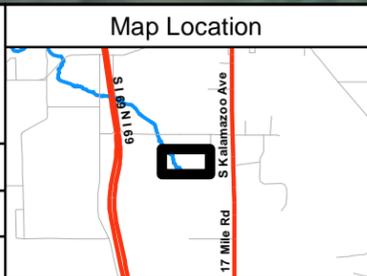
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## Figures



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 Project #: 60284509



**Legend**

- Line 6B Pipeline
- Vector Pipeline
- Approximate Replacement Pipeline
- Sub-Unit Boundary
- Parcel Boundary
- Quarter Mile Grid Segments
- A-1** Release Sub-Unit
- TC-A-001 Parcel Number

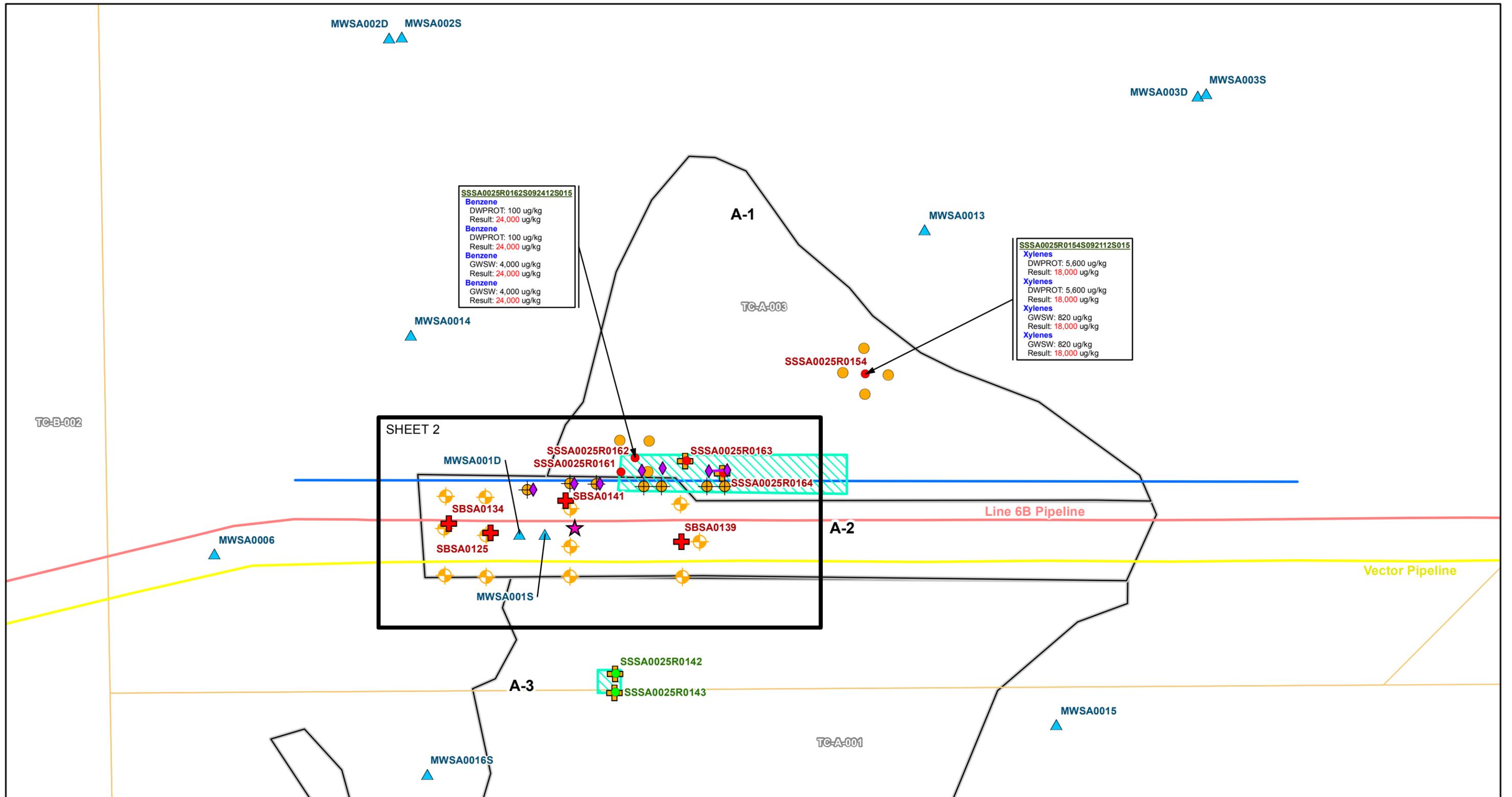
N

0 75 150 300

Scale in Feet

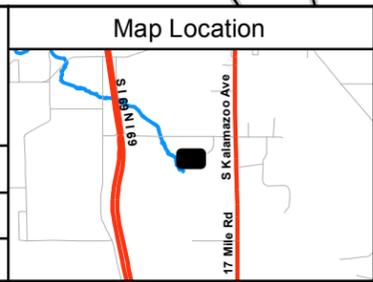
**FIGURE 1**  
**SITE LOCATION**  
**SOURCE AREA**

ENBRIDGE LINE 6B MP 608  
 MARSHALL, MI PIPELINE RELEASE  
 ENBRIDGE ENERGY, LIMITED PARTNERSHIP



**ENBRIDGE**

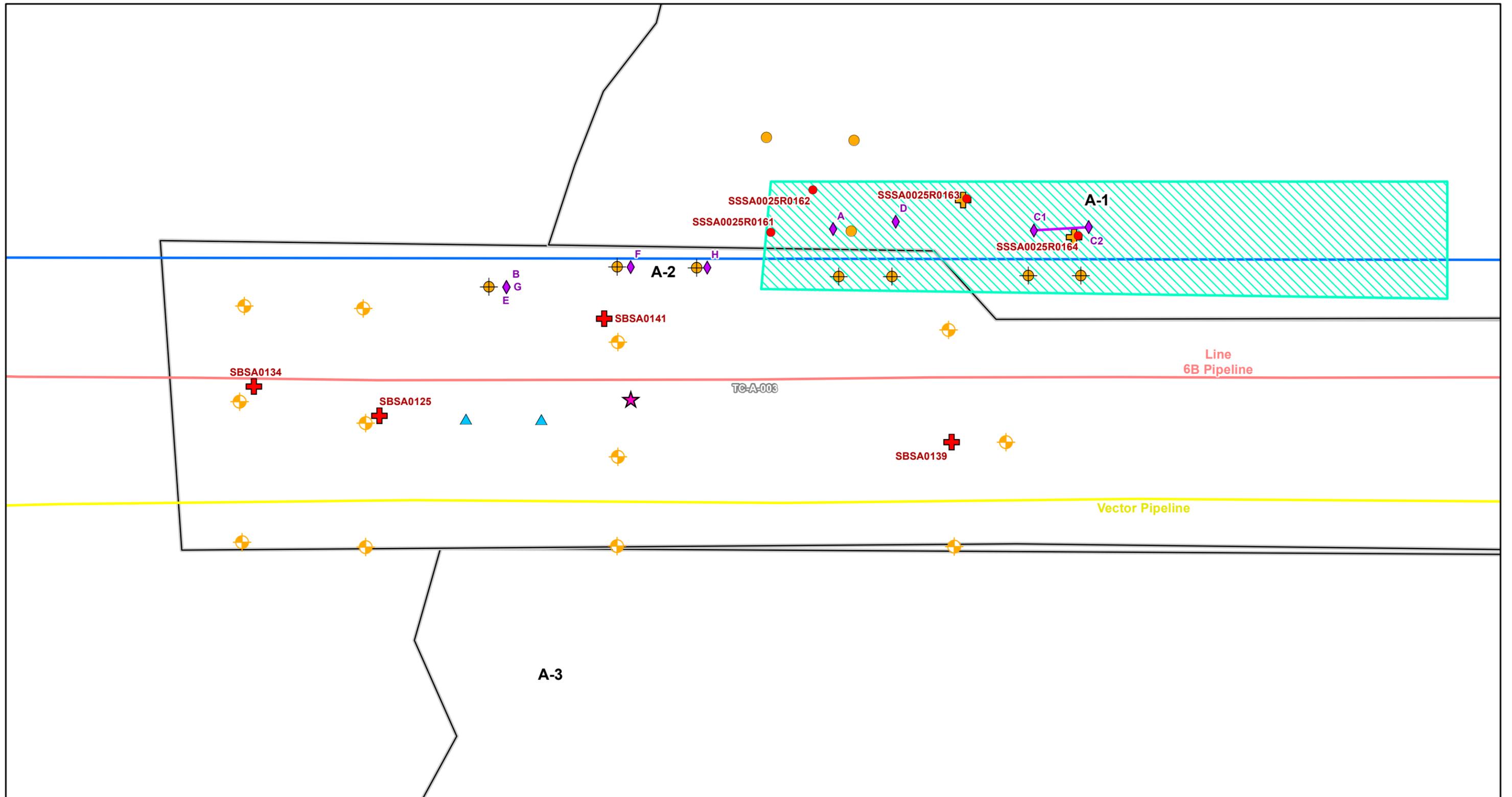
Drawn: NS 8/8/2013  
 Approved: JK 8/8/2013  
 Project #: 60284509



<ul style="list-style-type: none"> <li> Proposed Nested Monitoring Wells</li> <li> Proposed Paired Temporary Wells</li> <li> Proposed Soil Boring</li> <li> Proposed Monitoring Well</li> <li> Soil Sample (No Exceedance)</li> </ul>	<p>DWPROT - Drinking Water Protection        GWSW - Groundwater Surface Water Interface Protection</p>	<p><b>Sample With Exceedance of Criteria</b></p> <ul style="list-style-type: none"> <li> Soil (Existing)</li> <li> Soil (Excavated)</li> <li> Temporary Well (Abandoned)</li> <li> Monitoring Well to be Sampled (Existing)</li> <li> Monitoring Well (Abandoned)</li> </ul>	<ul style="list-style-type: none"> <li> Approximate Release Location</li> <li> Approximate Replacement Pipeline</li> <li> MDEQ Observation</li> <li> MDEQ Observation</li> <li> Area of MDEQ Concerns</li> <li> Quarter Mile Grid Segment</li> </ul>	<p> Sub-Unit Boundary</p> <p><b>A-1</b> Sub-Unit</p> <p> N</p> <p>Scale in Feet</p> <p>0 30 60 120</p>
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**FIGURE 2**  
 PROPOSED BORING/WELL LOCATIONS  
 SOURCE AREA  
 SHEET 1 OF 3

ENBRIDGE LINE 6B MP 608  
 MARSHALL, MI PIPELINE RELEASE  
 ENBRIDGE ENERGY, LIMITED PARTNERSHIP

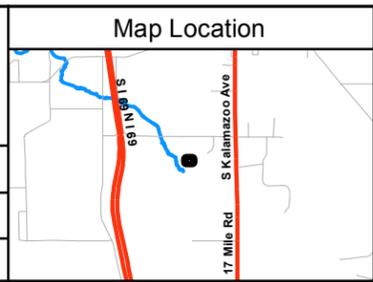


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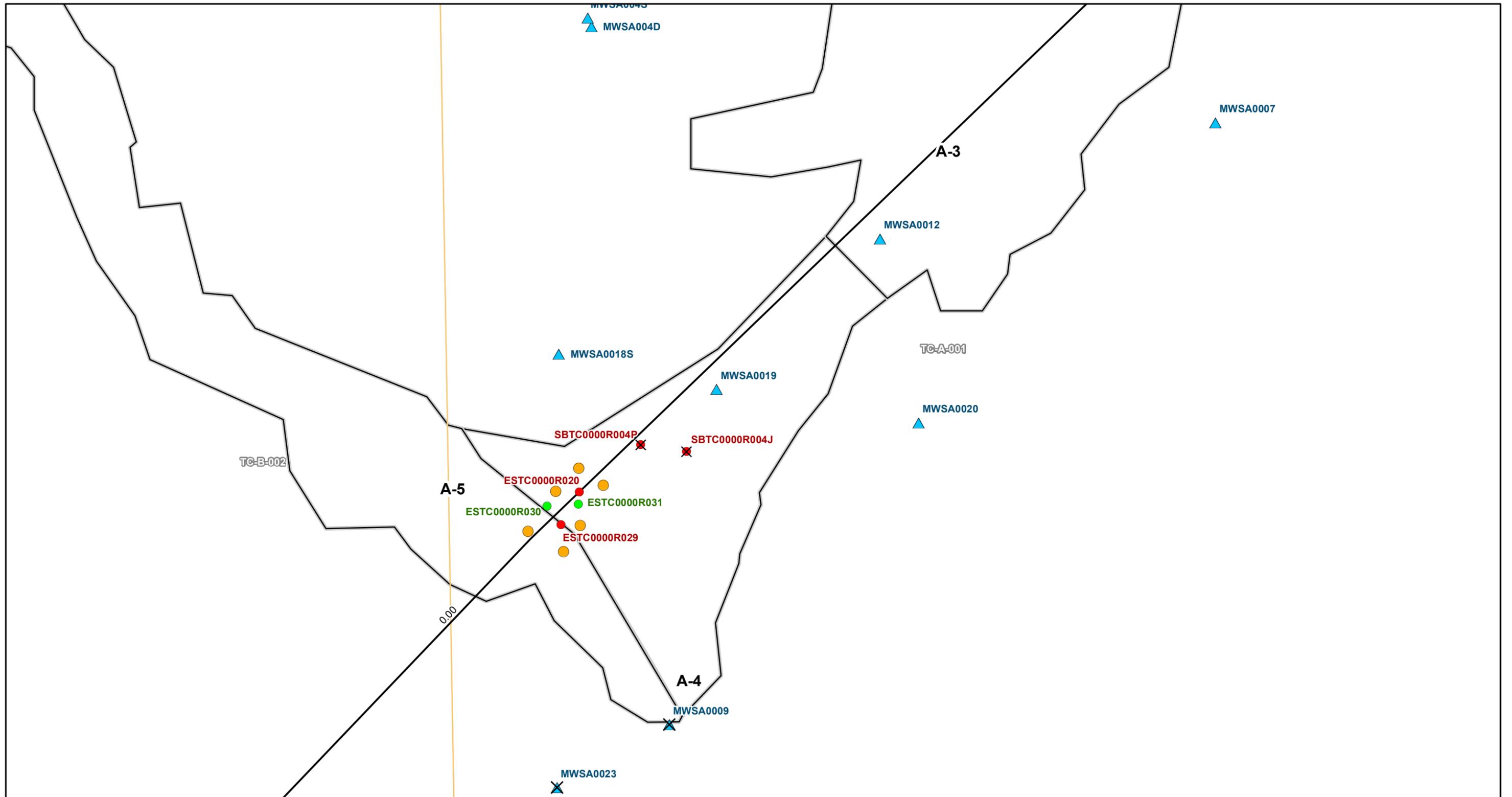
Project #: 60284509



<ul style="list-style-type: none"> <li> Proposed Nested Monitoring Wells</li> <li> Proposed Paired Temporary Wells</li> <li> Proposed Soil Boring</li> <li> Proposed Monitoring Well</li> <li> Soil Sample (No Exceedance)</li> </ul>	<p><b>Sample With Exceedance of Criteria</b></p> <ul style="list-style-type: none"> <li> Soil (Existing)</li> <li> Soil (Excavated)</li> <li> Temporary Well (Abandoned)</li> <li> Monitoring Well to be Sampled (Existing)</li> <li> Monitoring Well (Abandoned)</li> </ul> <p>DWPROT - Drinking Water Protection GWSW - Groundwater Surface Water Interface Protection</p>	<ul style="list-style-type: none"> <li> Approximate Release Location</li> <li> Approximate Replacement Pipeline</li> <li> MDEQ Observation</li> <li> Area of MDEQ Concerns</li> <li> Quarter Mile Grid Segment</li> </ul>	<p> Sub-Unit Boundary</p> <p><b>A-1</b> Sub-Unit</p> <p>N</p> <p>0 10 20 40</p> <p>Scale in Feet</p>
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**FIGURE 2**  
**PROPOSED BORING/WELL LOCATIONS**  
**SOURCE AREA**  
**SHEET 2 OF 3**

ENBRIDGE LINE 6B MP 608  
 MARSHALL, MI PIPELINE RELEASE  
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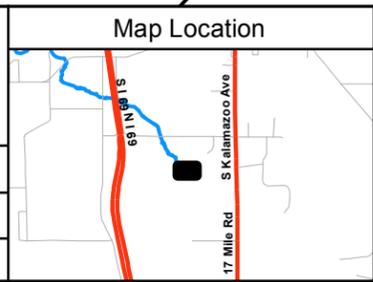


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Project #: 60284509



<ul style="list-style-type: none"> <li> Proposed Nested Monitoring Wells</li> <li> Proposed Paired Temporary Wells</li> <li> Proposed Soil Boring</li> <li> Proposed Monitoring Well</li> <li> Soil Sample (No Exceedance)</li> </ul>	<p><b>Sample With Exceedance of Criteria</b></p> <ul style="list-style-type: none"> <li> Soil (Existing)</li> <li> Soil (Excavated)</li> <li> Temporary Well (Abandoned)</li> <li> Monitoring Well to be Sampled (Existing)</li> <li> Monitoring Well (Abandoned)</li> </ul>	<ul style="list-style-type: none"> <li> Approximate Release Location</li> <li> Approximate Replacement Pipeline</li> <li> MDEQ Observation</li> <li> Area of MDEQ Concerns</li> <li> Quarter Mile Grid Segment</li> </ul>	<ul style="list-style-type: none"> <li> Sub-Unit Boundary</li> </ul> <p><b>A-1</b> Sub-Unit</p> <p>N</p> <p>0 30 60 120</p> <p>Scale in Feet</p>
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DWPROT - Drinking Water Protection  
GWSW - Groundwater Surface Water Interface Protection

**FIGURE 2**  
**PROPOSED BORING/WELL LOCATIONS**  
**SOURCE AREA**  
**SHEET 3 OF 3**

ENBRIDGE LINE 6B MP 608  
 MARSHALL, MI PIPELINE RELEASE  
 ENBRIDGE ENERGY, LIMITED PARTNERSHIP

**Attachment A**  
**Standard Operating Procedures for Synthetic Precipitation**  
**Leaching Procedure Analysis**





SYNTHETIC PRECIPITATION LEACHING PROCEDURE - ZERO HEADSPACE EXTRACTION

SW846 - 1312

SOPID: HN-EXT-007 Rev. Number: R02 Effective Date: 08/01/2012

Approved By: [Signature] Date: 7/24/12
Department Supervisor - Royce Hughes

Approved By: [Signature] Date: 7/24/12
Operations Manager - Joe Ribar

Approved By: [Signature] Date: 7/24/12
Quality Assurance - Dan Dehinger

Approved By: [Signature] Date: 7/20/12
Laboratory Director - Jeff Glaser

Archival Date: Doc Control ID#: Editor:

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature Title Date
Signature Title Date
Signature Title Date



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**SYNTHETIC PRECIPITATION LEACHING PROCEDURE – ZERO HEADSPACE EXTRACTION****1) Scope and Applicability**

- 1.1 Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

**2) Summary of Procedure**

- 2.1 For liquid samples (*i.e.*, those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 $\mu$ m glass fiber filter, is defined as the 1312 extract.
- 2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed for volatile analyses is reagent water. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8  $\mu$ m glass fiber filter.
- 2.3 If compatible (*i.e.*, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

**3) Definitions**

- 3.1 Organic Free Water: Deionized (DI) reagent water meeting purity characteristics of ASTM Type II laboratory distilled water (daily conductivity <1.0 umhos/cm).
- 3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.
- 3.3 Matrix Spike (MS/MSD): An aliquot of sample spiked with a known concentration of target analyte(s).
- 3.4 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing.

**4) Health and Safety Warnings****4.1 Lab Safety**

- 4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.
- 4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

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4.2 Waste Disposal

- 4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.
- 4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

- 4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.
- 4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

- 5.1 For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.
- 5.2 The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 30 minutes, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

6) Interferences

- 6.1 Interference may occur from a dirty ZHE (Zero Headspace Extractor) device and are eliminated by correct cleaning of the apparatus. All parts of the ZHE vessel should be thoroughly cleaned and dried. The ZHE O-rings should be rinsed with methanol and the extraction fluid.

7) Personnel Qualifications and Responsibilities

- 7.1 Each analyst must be trained, read and understand the method, read and understand the SOP, and prepare acceptable initial demonstration samples to establish method competency for this analysis.
- 7.2 Additionally, it is the responsibility of the analyst(s) to:
  - 7.2.1 The analyst is responsible for following this SOP as written. Any deviations or non-conformances must be documented and submitted to the department supervisor for approval.
  - 7.2.2 Produce contractually compliant data that meets all quality requirements using this procedure.
  - 7.2.3 Complete the required demonstration of proficiency before performing this procedure without supervision.



- 7.3 Section Supervisor - It is the responsibility of the section supervisor to:
  - 7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
  - 7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.
  - 7.3.3 The department supervisor is responsible for approving deviations and non-conformances.
- 7.4 QA Manager: The QA manager is responsible for verifying this SOP is followed as written through internal method and system audits.
- 7.5 Project Manager: The project manager must ensure that all contractual requirements for a client requiring this procedure are understood prior to initiating this procedure for a given set of samples.

8) Sample Collection, Handling, and Preservation

- 8.1 Samples should be collected using an appropriate sampling plan. A separate container of sample is required for the preliminary evaluations of the percent solids and the particle size. Quality control measures may require additional aliquots.
- 8.2 No preservation of samples is required prior to extraction. Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.3 When the handling the sample for the ZHE volatile preparation, care must be taken to minimize the loss of volatiles. Samples must be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).
- 8.4 SPLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for organic analyte determinations should not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Table 8.4 for holding time requirements.

Table 8.4	From Field Collection to SPLP Extraction	From SPLP Ext. to Prep. Extract.	From Prep. Extract. To Determinative analysis	Total Time
Volatiles	14	N/A	14	28

9) Equipment and Supplies

- 9.1 TCLP/SPLP Tumbler: The tumbling device must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
- 9.2 Zero-Headspace Extraction Vessel (ZHE): This device is for use only when the waste is being tested for the mobility of volatile analytes. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessels must have an internal volume of 500-600 mL, and be equipped to



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accommodate a 90 mm filter. The devices contain VITON™ O-rings that require periodic replacement.

9.2.1 The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi). NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

9.2.2 ZHE Materials of Construction: type 316 stainless steel equipment may be used when evaluating the mobility of volatile organics.

### 9.3 Filtration Devices:

9.3.1 For preparation of volatiles, using the ZHE performs this function.

9.3.2 For preliminary evaluations, the filter device for the metals/semi-volatiles may be used. The filter holder must be capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more.

9.3.3 For preliminary evaluations only, vacuum filtration can be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

9.4 Filters must be made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6 to 0.8  $\mu\text{m}$ , or equivalent. Pre-filters must not be used.

9.5 pH Meters: The meter should be accurate to  $\pm 0.05$  units at 25 °C.

9.6 ZHE Extract Collection Devices: A 40mL VOA vial containing HCl preservative is used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.

9.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit, or other ZHE device).

9.8 Laboratory Balance: Any laboratory balance accurate to within  $\pm 0.01$  grams may be used (all weight measurements are to be within  $\pm 0.1$  grams).

## 10) Standards and Reagents

10.1 Reagent grade chemicals must be used in all tests.

10.2 Reagent Water. ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, the reagent water is monitored periodically for impurities through use of method blanks analyzed by the GC/MS and through the preparation of batch SPLP extraction blanks.

10.3 Sulfuric acid/nitric acid (60/40 weight percent mixture)  $\text{H}_2\text{SO}_4/\text{HNO}_3$ . Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute  $\text{H}_2\text{SO}_4/\text{HNO}_3$  acid mixture may be prepared and used making it easier to adjust the pH of the extraction fluids.



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### 10.4 Extraction fluids.

- 10.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is  $4.20 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.
- 10.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is  $5.00 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.
- 10.4.3 **Extraction fluid #3: This fluid is reagent water and is used to determine cyanide and volatiles leachability.**

### 10.5 Notes:

- 10.5.1 Solutions are unbuffered and exact pH may not be attained.
- 10.5.2 These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

### 10.6 Prepare analytical standards according to the applicable analytical method.

## 11) Method Calibration

- 11.1 Perform support equipment calibration checks (balances, pH meter, etc.) as required for daily use.

## 12) Sample Preparation/Analysis

- 12.1 Preliminary Evaluations: Perform preliminary evaluations on a minimum 100g aliquot of waste. This aliquot may not be used to prepare the ZHE volatile extract. The preliminary evaluations include:

- 12.1.1 Determine the percent solids;
- 12.1.2 Determine whether the waste contains insignificant solids and is, therefore, its own extract after filtration;
- 12.1.3 Determine whether the solid portion of the waste requires particle size reduction

- 12.2 Determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described:

- 12.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids), proceed to Section 12.7.
- 12.2.2 If the sample is liquid or multiphasic, a liquid/solid separation is required to make the preliminary determination of percent solids. This involves use of a filtration device described in the next section

### 12.3 Filtration to determine % Solids:

- 12.3.1 Pre-weigh the filter and the container that will receive the filtrate.

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- 
- 12.3.2 Assemble the filter holder and filter following the manufacturer's instructions.
  - 12.3.3 Place the filter on the support screen and secure.
  - 12.3.4 Weigh out a subsample of the waste (100 gram minimum) and record the weight.
  - 12.3.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
  - 12.3.6 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.
    - 12.3.6.1 If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 12.3.11 to determine the weight of the waste sample that will be filtered.
  - 12.3.7 Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.
    - 12.3.7.1 Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.
  - 12.3.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.
  - 12.3.9 Weigh the filtrate and its container and record the weight.
  - 12.3.10 Determine the weight of the filtrate by subtracting the weight of the container from the total weight of the filtrate-filled container.
  - 12.3.11 Determine the weight of the solid phase by subtracting the weight of the filtrate phase from the weight of the total waste.
  - 12.3.12 Record the weight of the liquid and solid phases.
  - 12.3.13 Calculate and record the percent solids as follows:
  - 12.3.14 Percent solids = [Weight of solid phase / Total weight of waste ] X 100
  - 12.3.15 If a portion of the filtrate is retained on the filter, proceed to Section 12.4. Otherwise, proceed to Section 12.5.
- 12.4 Percent Dry Solids
- 12.4.1 Remove the solid phase and filter from the filter apparatus.



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- 
- 12.4.2 Dry the filter and solid phase at  $100 \pm 20^{\circ}\text{C}$  until two successive weight determinations yield the same value within  $\pm 1\%$ .
  - 12.4.3 Record the final weight.
  - 12.4.4 Calculate and record the % dry solids using the following equation:

$$\% \text{ dry solids} = [A - B] \times 100 / C$$

Where:

- A = (Weight of dry waste + filter)
- B = tared weight of filter
- C = Initial weight of waste

### 12.5 Determination of Particle Size Reduction:

- 12.5.1 Particle size reduction is required unless the solid has a surface area  $\geq 3.1 \text{ cm}^2$  or is smaller than 1 cm in its narrowest dimension.
- 12.5.2 If reduction of the solid phase is necessary, exposure of the waste to the atmosphere must be limited as much as possible. The method used to reduce the particle size must not generate heat in and of itself.

### 12.6 Preparation of the ZHE Extraction Vessel

- 12.6.1 Place the ZHE piston into the body of the ZHE. Remember to wet the piston O-rings slightly with extraction fluid.
- 12.6.2 If a sample size requirement is known, adjust the piston to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample. Secure the bottom gas inlet / outlet flange onto the ZHE body according to the manufacturer instructions.
- 12.6.3 Place a glass fiber filter between the support screens and place the top liquid inlet / outlet flange and set both aside.
- 12.6.4 Use a second aliquot of the waste to generate the volatile analysis extract. The portion used to determine % solids cannot be used.

### 12.7 Determination of Extraction Fluid

#### 12.7.1 Soils and wastes

- 12.7.1.1 Use Extraction Fluid #3 (reagent water)

### 12.8 Determination of Waste Weight Charged to ZHE

#### 12.8.1 When sample is 100% solids:

- 12.8.1.1 Weigh out a sub-sample (25gram maximum) of the waste to the nearest 0.1g and record the weight. Adjust particle size if necessary. Transfer the sample to the ZHE.
- 12.8.1.2 Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body.
- 12.8.1.3 Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom).
- 12.8.1.4 Attach the compressed air line to the gas inlet/outlet valve (on the bottom flange). With the liquid inlet/outlet valve (on top flange)



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- open, begin applying pressure (1-10 psi) to force all headspace out of the ZHE device.
- 12.8.1.5 Close the liquid inlet / outlet valve. Discontinue pressure to the piston. Determine the volume of extraction fluid #3 to add to the ZHE as follows:
- 12.8.1.6 Volume of Ext. Fluid #3 = 20 x wt. of waste in ZHE
- 12.8.1.7 Go to step 12.9.
- 12.8.2 When sample is < 0.5 % solids:
- 12.8.2.1 If the waste contains < 0.5 percent dry solids, the liquid portion of the waste after filtration is defined as the SPLP extract.
- 12.8.2.2 Quantitatively transfer sufficient sample to provide for volatile analysis, quickly to the ZHE (approximately 100-200 mL).
- 12.8.2.3 Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fittings and place the device in the vertical position with the gas inlet/outlet flange on the bottom. Do not attach the extraction collection device to the top plate.
- 12.8.2.4 Attach the compressed air line to the bottom gas inlet/outlet valve. With the top liquid inlet/outlet valve open, begin applying pressure (1-10 psi) to force all headspace out of the ZHE device.
- 12.8.2.5 At the first appearance of liquid from the liquid inlet / outlet valve, quickly close the valve and discontinue pressure.
- 12.8.2.6 Attach the pre-weighed gas tight syringe to the inlet /outlet-valve and open the valve. Apply 1-10 psi of pressure to force the liquid phase of the sample into the gas tight syringe.
- 12.8.2.7 Detach the syringe from the ZHE. Attach the transfer needle to the syringe. Insert the needle through the septum of the VOA vial in which the sample will be stored until volatile analysis is initiated.
- 12.8.2.8 Repeat steps 12.8.2.6 through 12.8.2.7 as necessary to provide two (2) 40 mL VOA vials for analysis.
- 12.8.2.9 Disassemble and clean the ZHE device. Discard the filter and suspended solids.
- 12.8.3 When sample is > 0.5% solids but < 5% solids
- 12.8.3.1 Pre-weigh a Tedlar bag collection container, record the weight and set aside.
- 12.8.3.2 Weigh a 500 gram sub-sample to the nearest 0.1 grams and quickly transfer it to the ZHE.
- 12.8.3.3 Secure the filter and support screens onto the top of the ZHE body and place the top flange to the ZHE body. Tighten all ZHE fittings and place the ZHE in the vertical position with the gas inlet/outlet on the bottom. Do not attach the extraction collection device to the top plate.



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- 12.8.3.4 Attach the compressed air line to the gas inlet/outlet valve at the bottom. With the liquid inlet / outlet valve at the top flange, open and begin applying pressure (1-10 psi) to force all headspace out of the ZHE.
- 12.8.3.5 As soon as liquid begins to escape from the top inlet / outlet valve, quickly close the valve and discontinue pressure.
- 12.8.3.6 Attach the evacuated pre-weighed Tedlar bag to the top inlet / outlet valve and open the valve. Apply 1-10 psi of pressure to force the liquid phase of the sample into the Tedlar bag. If no additional liquid has passed through the filter in a 2 minute interval following the initial pressurization of the vessel, slowly increase the pressure by an increment of 10 psi and apply the 2 minute interval at this pressure.
- 12.8.3.7 When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet / outlet valve and discontinue pressure to the piston.
- 12.8.3.8 Disconnect the Tedlar bag from the inlet / outlet valve. Weigh the Tedlar bag containing the filtrate.
- 12.8.3.9 The material remaining in the ZHE is defined as the solid phase and the filtrate is the liquid phase.

### 12.8.4 When sample is > 5% solids:

- 12.8.4.1 For wastes containing > 5% solids, use the percent solids information to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

$$12.8.4.1.1 \text{ Wt. of Waste to Charge ZHE} = [25\text{g} \times 100] / [\% \text{ Solids}]$$

- 12.8.4.2 Pre-weigh the Tedlar bag collection container, record the weight and set aside.
- 12.8.4.3 Follow steps 12.6.1 to 12.6.4 (ZHE Prep).
- 12.8.4.4 Weigh a sub-sample to the nearest 0.1 grams and quickly transfer it to the ZHE.
- 12.8.4.5 Follow steps 12.9.1 to 12.9.22.

### 12.9 ZHE Extraction

- 12.9.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line.
- 12.9.2 Release gas pressure on the ZHE piston (from the gas inlet/ outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE.
- 12.9.3 Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.
- 12.9.4 The 20:1 ratio of extraction fluid to waste is necessary.
- 12.9.5 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line.



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- 12.9.6 Check the ZHE to ensure that all valves are in their closed positions.
- 12.9.7 Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top.
- 12.9.8 Pressurize the ZHE to 5-10 psi and slowly open the liquid inlet / outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid.
- 12.9.9 This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve.
- 12.9.10 Re-pressurize the ZHE with 5-10 psi and check all ZHE fitting to ensure they are closed.
- 12.9.11 Record the initial pressure in the logbook.
- 12.9.12 Place the ZHE in the rotary extractor apparatus and rotate the ZHE at  $30 \pm 2$  rpm for  $18 \pm 2$  hours.
- 12.9.13 Record the starting and ending time of the extraction in the appropriate logbook.
- 12.9.14 The temperature of the extraction room shall be maintained at  $22 \pm 3^\circ \text{C}$ . throughout the agitation.
- 12.9.15 Record the initial and final temperature of the room in the logbook.
- 12.9.16 Following the 18 hour extraction, check the pressure behind the ZHE piston.
- 12.9.17 If the pressure has not been maintained (i.e. no gas release observed), the device has leaked and the extraction must be redone (using a new aliquot of waste).
- 12.9.18 If the pressure in the ZHE was maintained, attach a gas line to the gas inlet / outlet valve (bottom flange), and with the liquid inlet/outlet valve open (top flange), begin applying gentle pressure of 1 - 10 psi.
- 12.9.19 Force all the liquid extract out into the filtrate collection device (See Notes below).
- 12.9.20 Notes: If the original waste contained no initial liquid phase, the filtered liquid material obtained in 12.9.19 is defined as the SPLP extract. The filtrate will be collected in a gas tight syringe and transferred via a needle into two (2) 40 mL VOA vials. The transfer must not leave any headspace in the VOA vials.
- 12.9.21 If the original waste contained an initial liquid phase, the filtered liquid material obtained in 12.9.19 is combined with the liquid material from 12.8.3.8 (if the liquids are miscible) and the combination of these liquids is defined as the SPLP extract.
- 12.9.22 If the original waste contained an initial liquid phase that is not miscible with the filtered liquid material, the individual phases are collected SEPARATELY and analyzed separately. Determine the volume of each phase within 0.5% using the techniques described in this SOP and record the volume in the logbook. Provide this information to the appropriate department when delivering the extracts for analysis.

### 13) Troubleshooting

- 13.1 Refer to individual analytical SOPs for relevant information.

### 14) Data Acquisition

- 14.1 Sample preparation data recorded in preparation logbooks is manually entered into the LIMS for later use in analytical and QC calculations.



14.2 LIMS assigns a prep batch number for the data entered. Record the LIMS prep batch number in the prep log.

## 15) Calculation, and Data Reduction Requirements

15.1 LIMS uses the preparation data entered to perform calculations and reporting after analysis has been completed.

## 16) Quality Control, Data Assessment and Corrective Action

16.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

16.2 Matrix spikes should not be added prior to SPLP extraction of the sample. In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of SPLP extract as that which was analyzed for the unspiked sample. The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the SPLP extract when the recovery of the matrix spike is below the expected analytical method performance.

16.3 For matrix spike recovery preparation, calculation and QC: See determinative methods.

16.4 All quality control measures described in the appropriate analytical methods shall be followed.

16.5 If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

## 17) Data Records Management

17.1 Data hardcopy must be retained for a period of no less than 7 years. Electronic hardcopy must be retained for a period of no less than 7 years.

17.1.1 Hard copy documentation is maintained via logbooks for standard and chemical tracking, extraction procedures, instrument maintenance, and sample preparation logs.

17.1.2 All prepared samples are recorded in the preparation log, along with the LIMS batch ID, sample matrix, sample volume and/or weights, extraction fluid volumes, and extraction method.



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17.1.3 Prep Logbook data recording requires that each area is completed and if an item is not applicable, a line must be drawn through that section. No blank spaces should be left in the logbook. Uncompleted sections may be "Z'd" or "X'd" and then initialed.

17.2 All reagents and chemicals must be labeled and traceable back to the chemical inventory. Make sure that all reagents have a tracking number. Log all reagents and chemicals used into the extraction Prep Logbook. Make sure all reagents, chemicals and standards are dated.

17.3 All reagents prepared must be logged into the Chemical Prep Logbooks.

## 18) Quality Assurance and Quality Control

18.1 Logbooks must be reviewed monthly by the department supervisor.

18.2 Logbooks must be reviewed quarterly by the QA Staff.

## 19) Contingencies for Handling Out of Control Data

19.1 Data should never be reported when the associated mandatory QC data fail criteria. Should sample re-preparations and re-analysis be an impossibility, or method required corrective actions be an inadequate for the reporting of the data, the data can only be reported with the use of appropriate data qualifiers and project narration.

19.2 Deviations from this method or the quality control criteria outlined within must be documented via the NC/CA database for further evaluation.

## 20) Method Performance

20.1 N/A

## 21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R02	8/1/12	CES	Formatting

## 22) References and Related Documents

22.1 U.S. Environmental Protection Agency, "1312 Synthetic Precipitation Leaching Procedure (TCLP)", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.

22.2 40 CFR-Part 261.24, Toxicity Characteristic, Table 1

22.3 ALS Environmental Quality Assurance Manual, Revision 6.0 (or most current)





SYNTHETIC PRECIPITATION LEACHING PROCEDURE - NON-VOLATILE  
EXTRACTION

SW846 - 1312

SOPID: HN-EXT-008	Rev. Number: R02	Effective Date: 08/01/2012
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Approved By: Chad White Date: 7/24/12

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Operations Manager – Joe Ribar

Approved By: Dan Delinger Date: 7/24/12

Quality Assurance – Dan Delinger

Approved By: Jeff Glaser Date: 7/20/12

Laboratory Director – Jeff Glaser

Archival Date: _____	Doc Control ID#: _____	Editor: _____
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ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature _____	Title _____	Date _____
Signature _____	Title _____	Date _____
Signature _____	Title _____	Date _____



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**SYNTHETIC PRECIPITATION LEACHING PROCEDURE – NON-VOLATILE EXTRACTION****1) Scope and Applicability**

- 1.1 Method 1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

**2) Summary of Procedure**

- 2.1 For liquid samples (*i.e.*, those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8µm glass fiber filter, is defined as the 1312 extract.
- 2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.
- 2.3 If compatible (*i.e.*, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

**3) Definitions**

- 3.1 Organic Free Water: Deionized (DI) reagent water meeting purity characteristics of ASTM Type II laboratory distilled water (daily conductivity <1.0 umhos/cm).
- 3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.
- 3.3 Matrix Spike (MS/MSD): An aliquot of sample spiked with a known concentration of target analyte(s).
- 3.4 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing.

**4) Health and Safety Warnings****4.1 Lab Safety**

- 4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.
- 4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.



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### 4.2 Waste Disposal

- 4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.
- 4.2.2 Samples must be disposed according to Federal, State, and local regulations.

### 4.3 Pollution Prevention

- 4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.
- 4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

## 5) Cautions

- 5.1 Make sure all fittings of pressure filter device are soundly secured. Failure to do this may result in sample spraying out the side of the device.

## 6) Interferences

- 6.1 Semi-volatile extraction must be performed in glass or Teflon containers to prevent phthalate contamination.
- 6.2 Some paint and oil samples may appear liquid, but will not filter. These are to be treated as a soil for extraction purposes.

## 7) Personnel Qualifications and Responsibilities

- 7.1 Each analyst must be trained, read and understand the method, read and understand the SOP, and prepare acceptable initial demonstration samples to establish method competency for this analysis.
- 7.2 Additionally, it is the responsibility of the analyst(s) to:
  - 7.2.1 The analyst is responsible for following this SOP as written. Any deviations or non-conformances must be documented and submitted to the department supervisor for approval.
  - 7.2.2 Produce contractually compliant data that meets all quality requirements using this procedure.
  - 7.2.3 Complete the required demonstration of proficiency before performing this procedure without supervision.
- 7.3 Section Supervisor - It is the responsibility of the section supervisor to:
  - 7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
  - 7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.
  - 7.3.3 The department supervisor is responsible for approving deviations and non-conformances.



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- 7.4 QA Manager: The QA manager is responsible for verifying this SOP is followed as written through internal method and system audits.
- 7.5 Project Manager: The project manager must ensure that all contractual requirements for a client requiring this procedure are understood prior to initiating this procedure for a given set of samples.

### 8) Sample Collection, Handling, and Preservation

- 8.1 All samples should be collected using an appropriate sampling plan. A separate container of sample is may be required for the preliminary evaluations of the percent solids and the particle size. Quality control measures may require additional aliquots.
- 8.2 No preservation is required prior to extraction. Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.3 SPLP (1312) extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2. See Table 8.5 for holding time requirements.

<b>Table 8.5</b>	From Field Collection to SPLP Extraction	From SPLP Ext. to Prep. Extract.	From Prep. Extract. To Determinative analysis	Total Time
Semi Volatiles	14	7	40	61
Mercury	28	28	28	84
Metals, except Hg	180	180	28	388

### 9) Equipment and Supplies

- 9.1 TCLP/SPLP Tumbler: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at  $30 \pm 2$  rpm.
- 9.2 Filtration Devices:
- 9.2.1 For preliminary evaluations, the filter device for the metals/semi-volatiles may be used. The filter holder must be capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more.
- 9.2.2 For preliminary evaluations only, vacuum filtration can be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.
- 9.3 Filters must be made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6 to 0.8  $\mu\text{m}$ , or equivalent. Pre-filters must not be used. When evaluating the mobility of metals, filters must be acid-washed prior to use by



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rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended).

- 9.4 pH Meters: The meter should be accurate to  $\pm 0.05$  units at 25 °C.
- 9.5 Laboratory Balance: Any laboratory balance accurate to within  $\pm 0.01$  grams may be used (all weight measurements are to be within  $\pm 0.1$  grams).
- 9.6 Suitable pre-cleaned containers: Plastic is suitable for metals extraction. Glass or Teflon must be used for semi-volatile extraction.

### 10) Standards and Reagents

- 10.1 Reagent grade chemicals must be used in all tests.
- 10.2 Reagent Water. ASTM Type II water or equivalent meets the definition of reagent water.
- 10.3 Sulfuric acid/nitric acid (60/40 weight percent mixture)  $H_2SO_4/HNO_3$ . Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute  $H_2SO_4/HNO_3$  acid mixture may be prepared and used making it easier to adjust the pH of the extraction fluids.
- 10.4 Extraction fluids.
  - 10.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is  $4.20 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters.
  - 10.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is  $5.00 \pm 0.05$ . The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.
  - 10.4.3 Extraction fluid #3: This fluid is reagent water and is used to determine cyanide and volatiles leachability.
- 10.5 Notes:
  - 10.5.1 Solutions are unbuffered and exact pH may not be attained.
  - 10.5.2 These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid must be discarded and fresh extraction fluid prepared.
- 10.6 Prepare analytical standards according to the applicable analytical method.

### 11) Method Calibration

- 11.1 Perform support equipment calibration checks (balances, pH meter, etc.) as required for daily use.

### 12) Sample Preparation/Analysis



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- 12.1 Preliminary Evaluations: Perform preliminary evaluations on an aliquot of waste. The preliminary evaluations include:
- 12.1.1 Determine the percent solids;
  - 12.1.2 Determine whether the waste contains insignificant solids and is, therefore, its own extract after filtration;
  - 12.1.3 Determine whether the solid portion of the waste requires particle size reduction; and
  - 12.1.4 Determine which of the extraction fluids are to be used for the metals/semi-volatile SPLP extraction.
- 12.2 Determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described:
- 12.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (*i.e.*, is 100% solids), proceed to Section 12.7;
  - 12.2.2 If the sample is liquid or multiphasic, a liquid/solid separation is required to make the preliminary determination of percent solids. This involves use of a filtration device described in the next section
- 12.3 Filtration to determine % Solids:
- 12.3.1 Pre-weigh the filter and the container that will receive the filtrate.
  - 12.3.2 Assemble the filter holder and filter following the manufacturer's instructions.
  - 12.3.3 Place the filter on the support screen and secure.
  - 12.3.4 Weigh out a subsample of the waste (100 gram minimum) and record the weight.
  - 12.3.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
  - 12.3.6 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter.
    - 12.3.6.1 If waste material (>1% of original sample weight) has obviously adhered to the container used for transfer, determine the weight of this residue and subtract it from the sample weight determined in Section 12.3.11 for determination of the sample weight that will be filtered.
  - 12.3.7 Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (*i.e.*, filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.



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- 12.3.7.1 Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.
- 12.3.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.
- 12.3.9 Weigh the filtrate and its container and record the weight.
- 12.3.10 Determine the weight of the filtrate by subtracting the weight of the container from the total weight of the filtrate-filled container.
- 12.3.11 Determine the weight of the solid phase by subtracting the weight of the filtrate phase from the weight of the total waste.
- 12.3.12 Record the weight of the liquid and solid phases.
- 12.3.13 Calculate and record the percent solids as follows:
- 12.3.14 Percent solids = [Weight of solid phase / Total weight of waste ] X 100
- 12.4 If the % solids are < 0.5 %, SPLP extraction is not required and the filtered liquid is considered the SPLP extract. The pH must be taken immediately and recorded.
- 12.5 If the % solids are > 0.5 %, proceed with step 12.6 below.
- 12.6 Percent Dry Solids
- 12.6.1 Remove the solid phase and filter from the filter apparatus.
- 12.6.2 Dry the filter and solid phase at  $100 \pm 20^{\circ}\text{C}$  until two successive weighing's yield the same value within  $\pm 1\%$ .
- 12.6.3 Record the final weight.
- 12.6.4 Calculate and record the % dry solids using the following equation:

$$\% \text{ dry solids} = [A - B] \times 100 / C$$

Where:

A = (Weight of dry waste + filter)

B = tare weight of filter

C = Initial weight of waste

- 12.7 Determination of Particle Size Reduction: The use of a sieve or an appropriately graduated ruler is acceptable. If reduction of the solid phase is necessary, exposure of the waste to the atmosphere should be limited as much as possible. The method used to reduce the particle size must not generate heat in and of itself. Proceed with step 12.8 when complete.
- 12.8 Determination of Extraction Fluid
- 12.8.1 Soils
- 12.8.1.1 Use Extraction Fluid #1 if the sampling site is east of the Mississippi river.
- 12.8.1.2 Use Extraction Fluid #2 if the sampling site is west of the Mississippi river.
- 12.8.2 Wastes
- 12.8.2.1 Use Extraction Fluid #1
- 12.8.3 Cyanide Containing Soil/Wastes

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12.8.3.1 Use Extraction Fluid #3 (reagent water)

12.9 Sample Extraction

- 12.9.1 Plastic containers may be used for metals only. Glass or Teflon must be used for semi-volatile extraction.
- 12.9.2 Tare an appropriately sized container on a top loading balance.
- 12.9.3 Weigh and record an aliquot of sample (~50g) representative of the matrix.
- 12.9.4 Add the appropriate extraction fluid. The fluid to sample ratio must be maintained at 20:1.
- 12.9.5 Place extraction container in agitation apparatus and tumble for 18 +/- 2hr.
- 12.9.6 Record start and end time and temperature values.
- 12.9.7 Proceed to Section 12.10.

12.10 Extract Filtration

12.10.1 Metals

- 12.10.1.1 Vacuum filtration can be used for metal extracts only.
- 12.10.1.2 The metal extract must be filtered through a pre-rinsed 0.6 to 0.8µm glass fiber filter.

12.10.1.2.1 Pre-rinsing is completed by triple rinsing with 1N HNO<sub>3</sub>.

- 12.10.1.3 Filtered extract must be preserved to pH <2.
- 12.10.1.4 Transfer to the applicable department for digestion and analysis

12.10.2 Semi-volatiles

- 12.10.2.1 Semi-volatile extracts require pressure filtration.
- 12.10.2.2 Set up filtration apparatus per the manufacturer instructions, complete with 0.6 to 0.8µm glass fiber filter.
- 12.10.2.3 Place an appropriate sample collection container under apparatus.
- 12.10.2.4 Pour extract into the top using a glass funnel.
- 12.10.2.5 Seal apparatus with threaded cap.
- 12.10.2.6 Attach compressed air line to appropriate fitting.
- 12.10.2.7 Open valve on apparatus.
- 12.10.2.8 Slowly apply pressure to apparatus, not to exceed 50 psi.
- 12.10.2.9 Close valve and collect extraction fluid.
- 12.10.2.10 Transfer to the applicable department for sample preparation.

13) Troubleshooting

- 13.1 Refer to determinative SOPs for more information.

14) Data Acquisition

- 14.1 Sample preparation data recorded in preparation logbooks is manually entered into LIMS for later use in analytical and QC calculations.



14.2 LIMS assigns a prep batch number for the data entered. Record the LIMS prep batch number in the prep log.

## 15) Calculation, and Data Reduction Requirements

15.1 LIMS uses the preparation data entered to perform calculations and reporting after analysis has been completed.

## 16) Quality Control, Data Assessment and Corrective Action

16.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

16.2 Matrix spikes should not be added prior to SPLP extraction of the sample. In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of SPLP extract as that which was analyzed for the unspiked sample. The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the SPLP extract when the recovery of the matrix spike is below the expected analytical method performance.

16.3 For matrix spike recovery preparation, calculation and QC: See determinative methods.

16.4 All quality control measures described in the appropriate analytical methods shall be followed.

16.5 If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

## 17) Data Records Management

17.1 Data hardcopy must be retained for a period of no less than 7 years. Electronic hardcopy must be retained for a period of no less than 7 years.

17.1.1 Hard copy documentation is maintained via logbooks for standard and chemical tracking, extraction procedures, instrument maintenance, and sample preparation logs.

17.1.2 All prepared samples are recorded in the preparation log, along with the LIMS batch ID, sample matrix, sample volume and/or weights, extraction fluid volumes, and extraction method.



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- 17.1.3 Prep Logbook data recording requires that each area is completed and if an item is not applicable, a line must be drawn through that section. No blank spaces should be left in the logbook. Uncompleted sections may be “Z’d” or “X’d” and then initialed.
- 17.2 All reagents and chemicals must be labeled and traceable back to the chemical inventory. Make sure that all reagents have a tracking number. Log all reagents and chemicals used into the extraction Prep Logbook. Make sure all reagents, chemicals and standards are dated.
- 17.3 All reagents prepared must be logged into the Chemical Prep Logbooks.

## 18) Quality Assurance and Quality Control

- 18.1 Logbooks must be reviewed monthly by the department supervisor.
- 18.2 Logbooks must be reviewed quarterly by the QA Staff.

## 19) Contingencies for Handling Out of Control Data

- 19.1 Data should never be reported when the associated QC data fail criteria. Should sample re-preparations and re-analysis be an impossibility, or method required corrective actions be an inadequate for the reporting of the data, the data can only be reported with the use of appropriate data qualifiers and project narration.
- 19.2 Deviations from this procedure or any quality control criteria outlined within must be documented via the NC/CA database for further evaluation.

## 20) Method Performance

- 20.1 N/A

## 21) Summary of Changes

**Table 21.1 Summary of Changes**

Revision Number	Effective Date	Document Editor	Description of Changes
R02	8/1/12	CES	Formatting

## 22) References and Related Documents

- 22.1 U.S. Environmental Protection Agency, “1312 Synthetic Precipitation Leaching Procedure (TCLP)”, Revision 0, September 1994 Test Methods for Evaluating Solid Waste Physical/Chemical Methods.
- 22.2 40 CFR Part 261.24, Toxicity Characteristic, Table 1
- 22.3 ALS Environmental Quality Assurance Manual, Revision 6.0 (or most current)

**Attachment B**  
**Core Handling and Preservation Methods**

# Core Handling & Preservation

## Freezing Core with Dry Ice

- Second fastest way to freeze core and preserve fluid saturations
- “Best” for most applications
- Longer freeze time results in little alteration of core physical properties
- Used when fluid saturations and physical properties are of equal importance
  - Preserves volatile and semivolatile hydrocarbons
  - Keeps fluids from migrating in high-permeance materials

# Core Handling & Preservation

## Freezing Core with Dry Ice

1. Dry Ice is available at most industrial gas supply companies (some supermarkets).
2. Large marine ice chests are used to freeze and transport core.
  - a. Up to 72 quart coolers used for smaller core.
  - b. Large marine coolers (94 quarts and greater) used for core over two feet long.
3. Place approximately 50 to 75 pounds of dry ice in the bottom of the ice chest.
4. Label core, attach end caps, and place core in ice chest.
  - a. Close lid and core will freeze solid in 30 minutes.
  - b. Each ice chest freezes up to 22.5 feet of core using 50-75 pounds of dry ice.
5. Caution - Do not put dry ice in an unventilated or enclosed space due to risk of death by asphyxiation!



# Core Handling & Preservation

- Place 50 to 75 pounds of dry ice in the bottom of the ice chest.
- Put core in ice chest and close lid. Core will freeze solid within 30 minutes.
- Continue adding core and dry ice as necessary.



# Core Handling & Preservation

## Transporting Frozen Core

1. Caution - Do not transport dry ice in passenger compartments due to risk of death by asphyxiation!
2. Large ice chests are used for transporting frozen core.
3. Place a layer of foam, bubble wrap, or “Styrofoam peanuts” in the bottom of ice chest to absorb shock during transport.
4. Place one layer of core on top of packing material.
5. Place a layer of dry ice over the core.
  - a. Dry ice pellets can be used as a “packing material” to secure the core and fill any voids between cores.
  - b. Block dry ice can also be used by breaking into smaller pieces and packing around core.
  - c. Bubble wrap or styrofoam peanuts can be added to the core layers to cushion the core during transport.

# Core Handling & Preservation

## Transporting Frozen Core

6. Continue alternating layers of core, packing materials and dry ice. Do not exceed three layers of core.
7. Fill remaining space with dry ice.
8. Put Chain of Custody in a zip lock bag and tape inside lid.
9. Close lid and seal with tape to keep cold.
10. Attach Dry Ice Placards to ice chest.
11. Contact Overnight Courier for shipment to laboratory for next day delivery. Notify laboratory of shipment arrival time (tracking numbers).
12. Arrange shipment so core does not sit in hot warehouse or truck for days.

# Core Handling & Preservation

## Transporting Frozen Core

- Ice chest filled with cores, dry ice, and packing material for cushioning.



# Core Handling & Preservation

## Transporting Frozen Core

Dry Ice Placard:  
Overnight couriers  
can supply the  
placard stickers.

Shipper's Declaration not Required.

Part B is required

Dry Ice amount must be in kilograms.

Note: 2 lbs. = 1 kg.

Airwaybills/airbills must have the following:

1. "Dangerous Goods - Shipper's Declaration not required".
2. Dry Ice; 9; UN 1845;
3. \_\_\_\_\_ X \_\_\_\_\_ Kg 904 III  
(Number (wt)  
pkgs)

**Dry Ice**  
\_\_\_\_\_ kg.

**UN 1845**

Shipper's name and Address \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Consignee Name and Address \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

9

Logos # 106426

# Core Handling & Preservation

**FedEx USA Airbill Express** FedEx Tracking Number 8369 5105 3013 Form ID No. 0215 Sender's Copy

**1 From** Please print and print hard  
Date \_\_\_\_\_ Sender's FedEx Account Number 1669-5829-6  
Sender's Name \_\_\_\_\_ Phone (562) 907-3607  
Company P T S LABORATORIES  
Address 8100 SECURA WAY  
City SANTA FE SPRINGS State CA ZIP 9570-2116

**2 Your Internal Billing Reference**  
First 24 characters will appear on invoice.

**3 To**  
Recipient's Name \_\_\_\_\_ Phone ( ) \_\_\_\_\_  
Company \_\_\_\_\_  
Address \_\_\_\_\_  
City \_\_\_\_\_ State \_\_\_\_\_ ZIP \_\_\_\_\_

**4a Express Package Service** Packages up to 150 lbs.  
 FedEx Priority Overnight Next business morning  
 FedEx Standard Overnight Next business afternoon  
 FedEx First Overnight Earliest next business morning delivery to select locations  
 FedEx 2Day Second business day  
 FedEx Express Saver Third business day

**4b Express Freight Service** Packages over 150 lbs.  
 FedEx 1Day Freight\* Next business day  
 FedEx 2Day Freight Second business day  
 FedEx 3Day Freight Third business day

**5 Packaging** \*Declared value limit \$500  
 FedEx Envelope\*  
 FedEx Pak\* Includes FedEx Small Pak, FedEx Large Pak, and FedEx Specialty Pak  
 Other

**6 Special Handling** Includes FedEx address in Section 1.  
 SATURDAY Delivery  
 HOLD Weekday at FedEx Location  
 HOLD Saturday at FedEx Location  
Does this shipment contain dangerous goods?  
 No  Yes As per attached Shipper's Declaration  
 Dry Ice Dry Ice, 3, UN 1845 50  
 Cargo Aircraft Only

**7 Payment Method** Enter FedEx Acct. No. or Credit Card No. below.  
 Sender Acct. No. in Section 1 will be billed.  
 Recipient  
 Third Party  
 Credit Card  
 Cash/Check

Total Packages \_\_\_\_\_ Total Weight \_\_\_\_\_ Total Declared Value\* \$ \_\_\_\_\_ .00  
FedEx Use Only

**8 Release Signature** Sign or authorize delivery without obtaining signature.  
By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

0231229100 447

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By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.  
Questions? Visit our Web site at [fedex.com](http://fedex.com) or call 1.800.Go.FedEx® 800.463.3339.

Note Dry Ice Box

Paper Shipping Label: FedEx is the easiest to ship with, they routinely handle dry ice shipments.



# Core Handling & Preservation

**FedEx** Ship Track/History Address Book Preferences Fast Ship Reports My Profile

<< Log out Home Ship

**Special services**

- Saturday pickup
- Saturday delivery
- COD (Collect on Delivery)
- Hold at FedEx location
- Dry Ice
- Dangerous Goods

**FedEx Express reference information**

Your reference

P.O. number

Invoice number

Department number

**FedEx InSight<sup>SM</sup>** (a shipment visibility application) [Learn more.](#)

- Block shipment data (will prohibit the recipient and third party payer from viewing information about this shipment)
- Shipment content (shipment level detail for InSight customers only)

**FedEx<sup>®</sup> Delivery Signature Options**

Signature type

**Shipping Options**

Pickup/Drop-off

- Will use scheduled pickup at my location
- Will drop off at FedEx location
- Will contact FedEx to request pickup

**Please Note**

- Click the Continue button only once. Expect some delay due to transmission time. Do not click Stop or Reload; it may cause a duplicate shipment transaction.
- By clicking the Continue button, you agree to the FedEx Ship Manager at [fedex.com Terms of Use](#) and the FedEx terms of shipping in the applicable FedEx Service Guide Shipper's Terms and Conditions for FedEx Express international shipments.

Electronic Shipping: FedEx is the easiest to ship with, they routinely handle dry ice shipments.



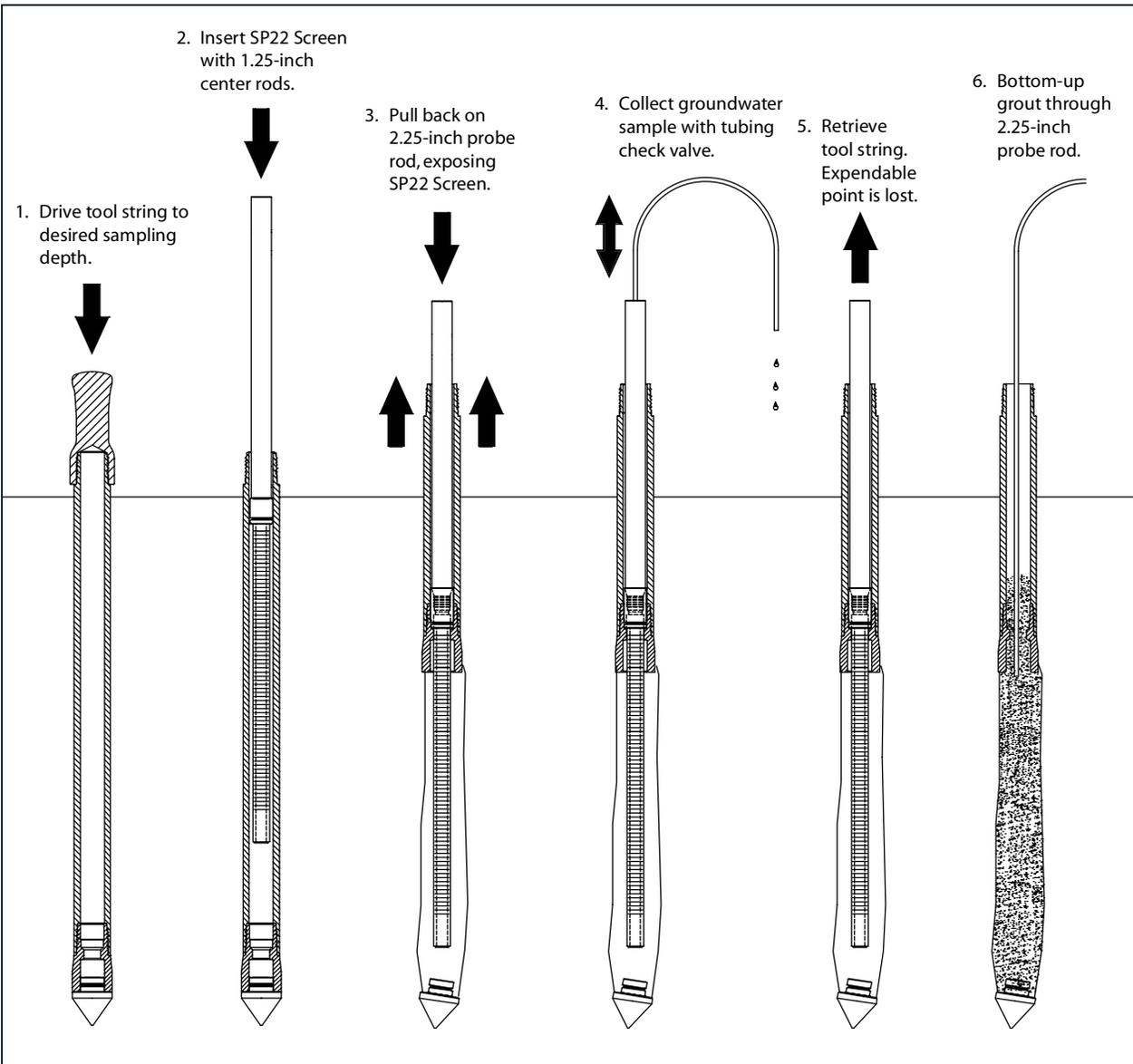
**Attachment C**  
***Geoprobe® Screen Point 22 Groundwater Sampler Standard***  
***Operating Procedure Technical Bulletin No. MK3173***

# GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER

## STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3173

PREPARED: April 2010



OPERATION OF THE GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER



**Geoprobe® and Geoprobe Systems®, Macro-Core® and Direct Image® are  
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**Screen Point 22 Groundwater Sampler is manufactured  
under U.S. Patent 5,612,498**

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## 1.0 OBJECTIVE

The objective of this procedure is to deploy a stainless steel or PVC screen at depth, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 22 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

## 2.0 BACKGROUND

### 2.1 Definitions

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

**Screen Point 22 (SP22) Groundwater Sampler:** A direct push device consisting of a PVC or stainless steel screen that is lowered (post-run) to depth within a sealed string of steel probe rods and then deployed for the collection of representative groundwater samples. Upon deployment, up to 48 inches (1219 mm) of screen can be exposed to the formation. There is also an optional 12-inch screen that can be used. The Screen Point 22 Groundwater Sampler is designed for use with 2.25-inch probe rods and machines equipped with the more powerful GH60 and GH80 series hydraulic hammers. Operators with GH40 series hammers may choose to use this sampler in soils where driving is easier.

**Rod Grip Pull System:** An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with probe rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

### 2.2 Discussion (Fig. 2.1)

In this procedure, 2.25-inch probe rods are advanced into the subsurface with a Geoprobe® subsurface machine (Fig. 2.1, Step 1). While the tool string is advanced to depth, O-ring seals at each rod joint, the expendable point holder, and the expendable drive point provide a watertight system. This eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once the leading end of the 2.25-inch probe rods reaches the desired sampling interval, an SP22 screen is lowered to the bottom of the rods using a string of either 1.25-inch outside diameter (OD) light-weight center rods, 1.25-inch probe rods, or 0.75-inch schedule 40 flush-thread PVC riser (Fig. 2.1, Step 2). The 2.25-inch rods are then retracted while the SP22 screen is held in place with the 1.25-inch rods or PVC riser (Fig 2.1, Step 3). As the 2.25-inch tool string is retracted, the expendable point is released from the expendable point holder. The tool string and expendable point holder may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

The SP22 Sampler can also be used with the Geoprobe® DT22 system. (Fig. 2.2)

(continued on following page)

## Expendable Drive Points

The SP22 system utilizes an SP22 Expendable Point Holder (33764) and standard 2.45-inch (62-mm) OD steel Expendable Drive Points for 2.25-inch probe rods (AT2015K). Extended Shank Expendable Drive Points (19442) are available for soft soil conditions where standard points may be advanced out of the point holder during percussion. A third option is to use a part number 43128 SP22 Expendable Point Holder along with 1.625-inch (41-mm) steel Expendable Drive Points (GW1555K). These smaller drive points are more economical to purchase and ship, but must not be used with GH80 Series Hydraulic Hammers as they may not stay seated during percussion.

## Screens

Two types of screens have been developed for use in the Screen Point 22 Groundwater Sampler - a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm). These screens are available in nominal 48- and 12-inch lengths. Effective screen lengths for the 48- and 12-inch PVC screens are 48 inches (1219 mm) and 12 inches (305 mm), while 48- and 12-inch stainless steel screens have effective screen lengths of 43 inches (1092 mm) and 14 inches (356 mm) respectively. Both types of screens are recovered with the tool string after sampling.

The SP22 PVC Screen Head Adapter (37871) provides yet another screen option for the SP22 sampler. Using this adapter, a section of slotted 0.75-inch Schedule 40 PVC pipe may be lowered through the 2.25-inch probe rods using a string of flush-threaded 0.75-inch Schedule 40 PVC Riser. An SP22 PVC Screen Plug (38968) is installed in the leading end of the slotted pipe prior to use. The slotted pipe may be cut and the screen plug installed to provide custom screen lengths.

An O-ring is located at the top of each stainless screen and on the screen adapters. When a screen is deployed, this O-ring maintains a seal between the top of the screen and the inner wall of the probe rods or expendable point holder as indicated in Figure 2.1. As a result, any liquid entering the tool string must first pass through the screen.

Screens are constructed such that equipment can be inserted into the screen cavity for sample collection as noted in the following section and illustrated in Figure 2.1, Step 4. This makes direct sampling possible from anywhere within the saturated zone.

The inner rod string and screen are generally removed prior to grouting through the 2.25-inch rod string as shown in Figure 2.1, Steps 5-6. However, a removable plug in the lower end of the screens allows for grouting through flexible tubing extending out the bottom of the screen as with the Geoprobe® SP15/16 Groundwater Samplers if desired.

## Sample Collection

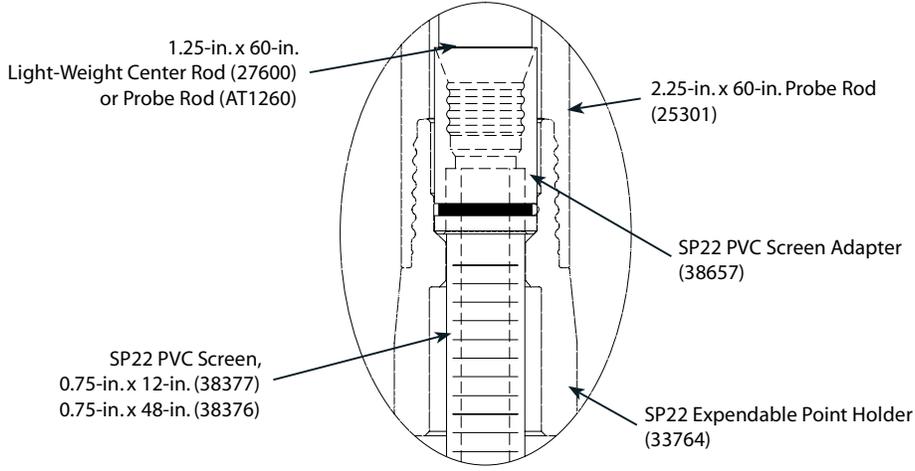
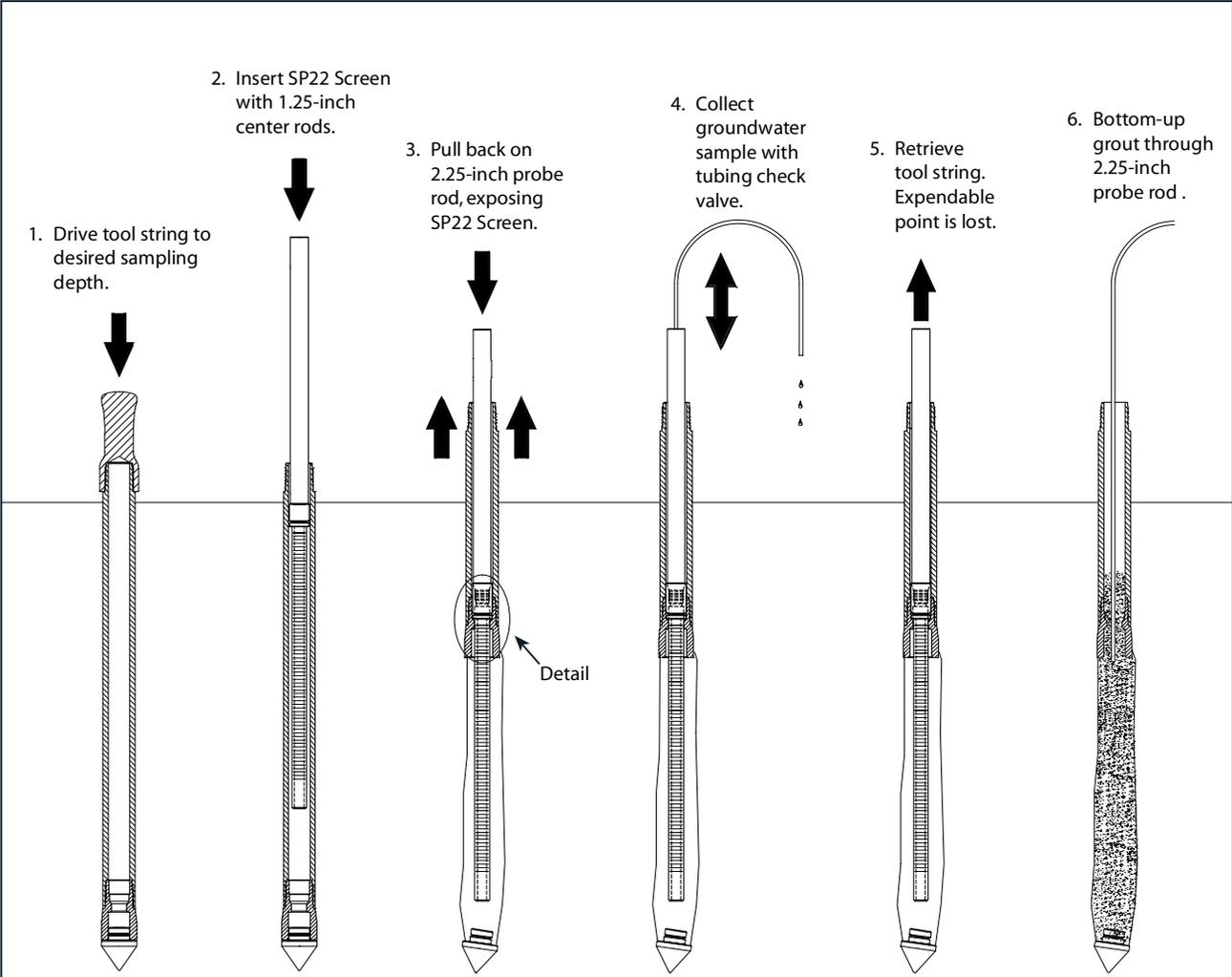
Groundwater samples can be obtained from the SP22 screen in a number of ways. A common method utilizes 0.375-inch OD polyethylene (TB25L) or Teflon® (TB25T) tubing and a check valve assembly. The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is then pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An SP22 Check Valve Assembly (37893) is recommended if sampling through 1.25-inch light-weight center rods. The SP22 Check Valve Assembly is approximately 20 inches long to enable it to pass through the stepped diameters at each rod joint that may cause problems for other, shorter check valves.

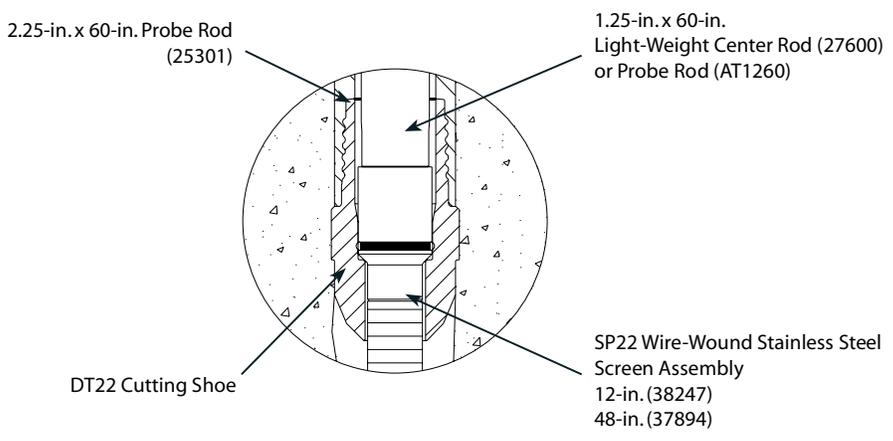
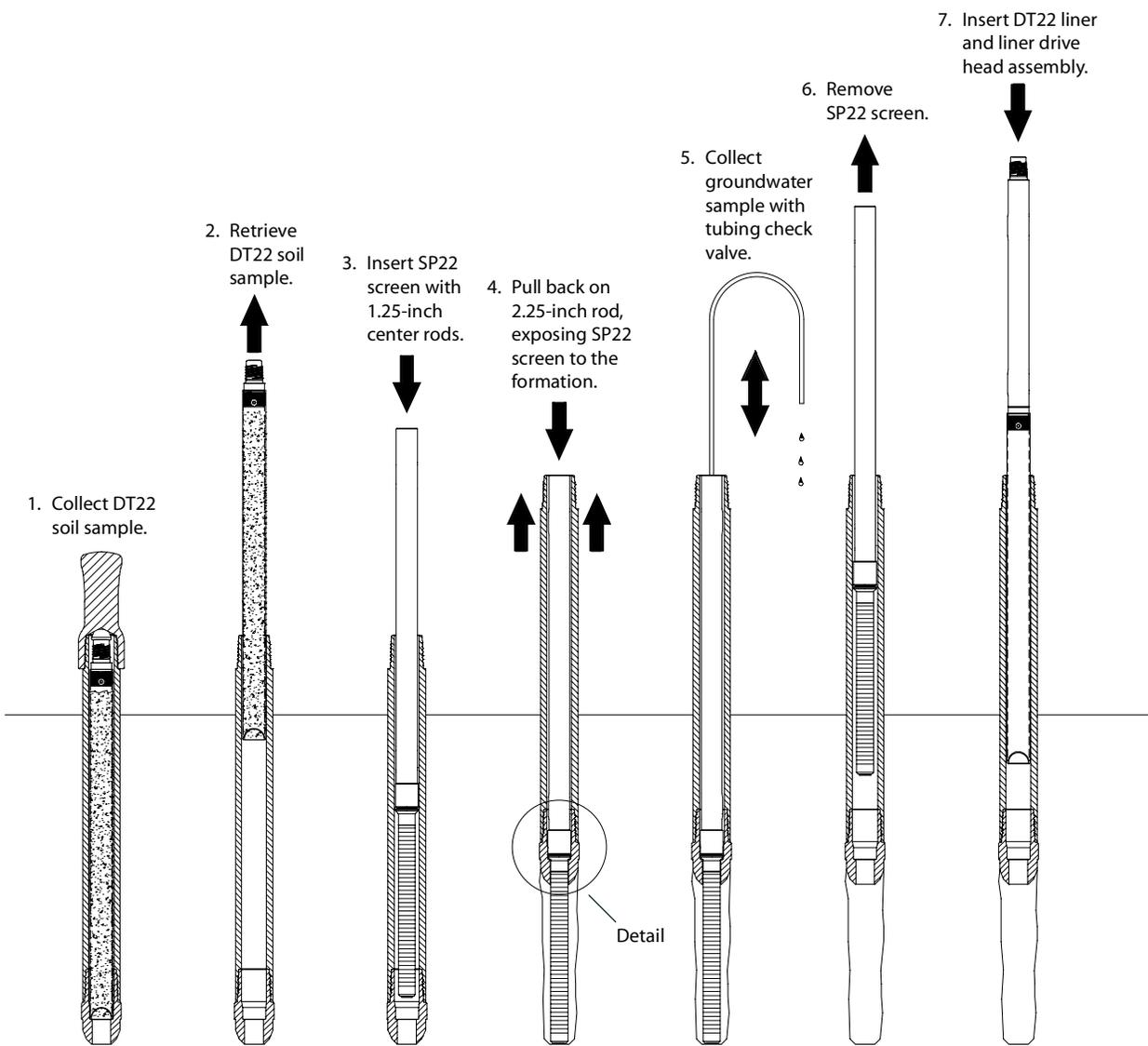
An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to tubing that is inserted through the inner rods to within the SP22 screen. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP22 Sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)\*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

*\*The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*



**FIGURE 2.1**  
**Screen Point 22 Groundwater Sampler - 0.75-inch PVC Screen Option**



**FIGURE 2.2**  
**Screen Point 22 Groundwater Sampler Operation with DT22 Sampling System**

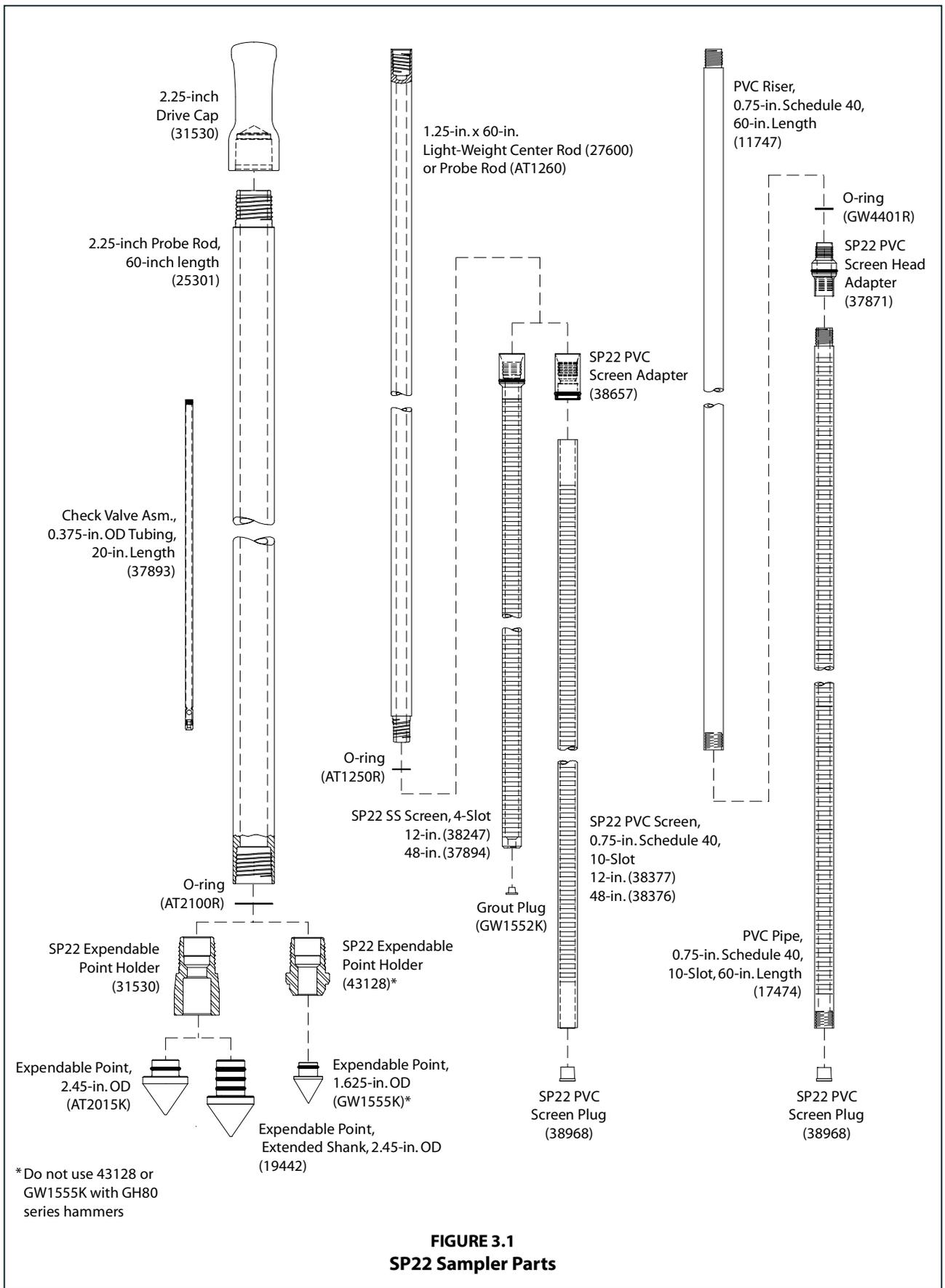
### 3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 22 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP22 / 2.25-inch probe rod configurations. Additional rod sizes and accessories are available. Contact Geoprobe Systems® for information regarding tools and equipment options.

<b>SP22 Sampler Parts</b>	<b>Part Number</b>
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (48-in.) .....	37894
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (12-in.) .....	38247
Grout Plugs, PE (Pkg. of 25) .....	GW1552K
SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-in. ....	38376
<i>SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-inch, Kit (includes 2 each of 38376 and 38429) .....</i>	<i>38664</i>
SP22 Screen, PVC, 10-Slot, 0.75-in. x 12-in. ....	38377
<i>SP22 Screen, PVC, 10-Slot 0.75-in. x 12-in., Kit (includes 2 each of 38377 and 38429) .....</i>	<i>38667</i>
SP22 PVC Screen Plug .....	38968
<i>SP22 PVC Screen Plug Kit (includes 10 of 38968) .....</i>	<i>38530</i>
SP22 PVC Screen Adapter, 0.75-in. PVC x 1.25-in. Probe Rod Box .....	38657
SP22 PVC Screen Head Adapter, 0.75-in. (for flush-threaded 0.75-in. Schedule 40 PVC) .....	37871
SP22 O-ring Kit (Pkg. of 10 O-rings for SP22 PVC screen adapters and stainless steel screens) ...	37853
O-rings, 0.75-in. PVC Riser (Pkg. of 25) .....	GW4401R
SP22 Expendable Point Holder, 2.25-in. Probe Rods, AT2045K and 19442 Points .....	33764
SP22 Expendable Point Holder, 2.25-in. Probe Rods, GW1555 Points* .....	43128
<b>Outer Casing (2.125-inch Probe Rods) and Inner Rod String</b>	<b>Part Number</b>
Probe Rod, 2.25-in. x 60-in. ....	25301
Expendable Drive Points, Steel, 2.45-in. OD (Pkg. of 25) .....	AT2015K
Expendable Drive Points, Steel, 2.45-in. OD, extended shank .....	19442
Expendable Points, steel, 1.625-in. OD (Pkg. of 25)* .....	GW1555K
Drive Cap, 2.25-in. Probe Rods, Threadless, (for GH60 and GH80 Series Hammers) .....	31530
O-Rings, 2.25-in. Probe Rods (Pkg. of 25) .....	AT2100R
Rod Grip Handle, 2.25-in. Probe Rods, (for GH60 and GH80 Series Hammers) .....	29385
Light-Weight Center Rod, 1.25-in. x 60-in. ....	27600
Probe Rod, 1.25-in. x 60-in. ....	AT1260
O-ring, 1.25-in. rods (Pkg. of 25) .....	AT1250R
Rod Grip Handle, 1.25/1.5-in. Rods, (for GH60 and GH80 Series Hammers) .....	15554
PVC Riser, 0.75-in. Schedule 40 x 60-inch .....	11747
PVC Pipe, 0.75-in. Schedule 40 x 60-inch, 10-Slot .....	17474
<b>Grout Accessories</b>	<b>Part Number</b>
High-Pressure Nylon Tubing, 0.375-in. OD / 0.25-in. ID, 100-ft. (30 m) .....	11633
Grout Machine, Auxiliary-Powered .....	GS2200
Grout System Accessories Package, 2.25-in. rods .....	GS1015
<b>Groundwater Purging and Sampling Accessories</b>	<b>Part Number</b>
Polyethylene Tubing, 0.375-in. OD, 500 ft. ....	TB25L
Check Valve Assembly, 0.375-in. OD Tubing x 20 in. Long .....	37893
Water Level Meter, 0.438-in. OD Probe, 100 ft. cable .....	GW2000
Mechanical Bladder Pump** .....	MB470
Mini Bailer Assembly, Stainless Steel .....	GW41

\* Not for use with GH80 Series Hydraulic Hammers

\*\* Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.



**FIGURE 3.1**  
**SP22 Sampler Parts**

## 4.0 OPERATION

### 4.1 Basic Operation

The SP22 Sampler utilizes a stainless steel or PVC screen which is lowered (post-run) through an alloy steel 2.25-inch OD probe rod tool string. An expendable drive point is placed in an expendable point holder on the leading 2.25-inch probe rod prior to advancement (Fig. 4.1). This expendable point is removed and stays in the subsurface as the rods are pulled back to exposes the SP22 screen. O-rings on the probe rods, the expendable point holder, and the expendable drive point provide a watertight tool string which keeps contaminants out of the system as the 2.25-inch rods are driven to depth in preparation for installation of the SP22 screen.

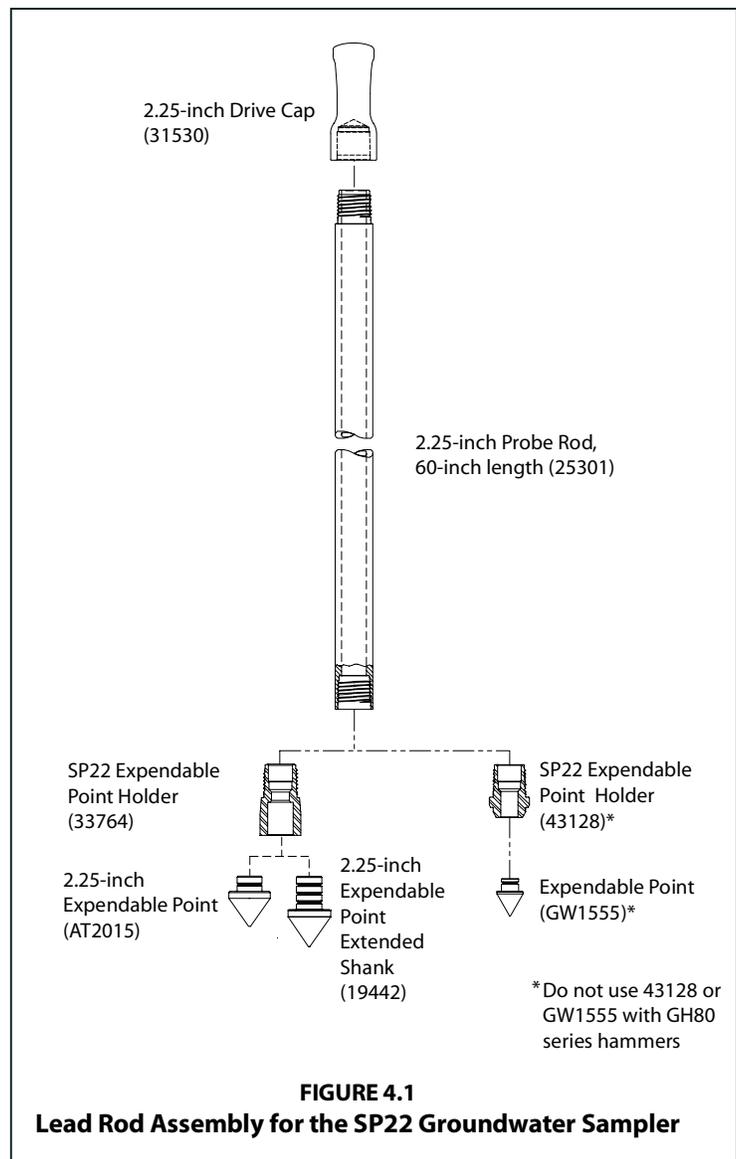
Once the sampling interval is reached with the 2.25-inch probe rods, the stainless steel or PVC screen is lowered through the rods using 1.25-inch probe rods, 1.25-inch light-weight center rods, or 0.75-inch PVC riser pipe. The 2.25-inch tool string is then retracted while the screen is held in place with the inner rods or riser. The system is now ready for groundwater sampling. When sampling is complete, the inner rods and screen are removed for grouting during retrieval or the 2.25-inch rods. Alternatively, a removable plug is located in the bottom of the screens to allow grouting directly through the inner tool string with high-pressure tubing during retrieval.

### 4.2 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

### 4.3 Lead Rod Assembly (Fig. 4.1)

1. Place an O-ring on the expendable point holder.
2. Thread expendable point holder into the 2.25-inch probe rod.
3. Place an O-ring on a steel expendable drive point.
4. Firmly seat the expendable point in the expendable point holder.
5. Place 2.25-inch Drive Cap (31530) on the top of the 2.25-inch probe rod. The lead rod assembly is now ready to be driven to depth.



#### 4.4 Advancing the Tool String (Fig. 4.2, step 1)

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Drive first 2.25-inch probe rod (as assembled in section 4.3).
2. Advance the tool string at a slow speed for the first few feet to ensure that the string is aligned properly.
3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the driven probe rod. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the tool string the entire length of the new rod.

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.
5. Remove the drive cap and retract the probe derrick away from the tool string.

#### 4.5 Screen Deployment (Fig 4.2, step 2 - 4)

1. Attach an SP22 stainless steel or PVC screen to a 1.25-inch probe rod, 1.25-inch light-weight center rod, or 0.75-inch flush-thread PVC riser using an SP22 PVC Screen Adapter (38657) or SP22 PVC Screen Head Adapter (37871) as shown in Figure 3.1. Note that the 38657 screen adapter is connected to the SP22 PVC screen using the setscrews provided with the adapter.

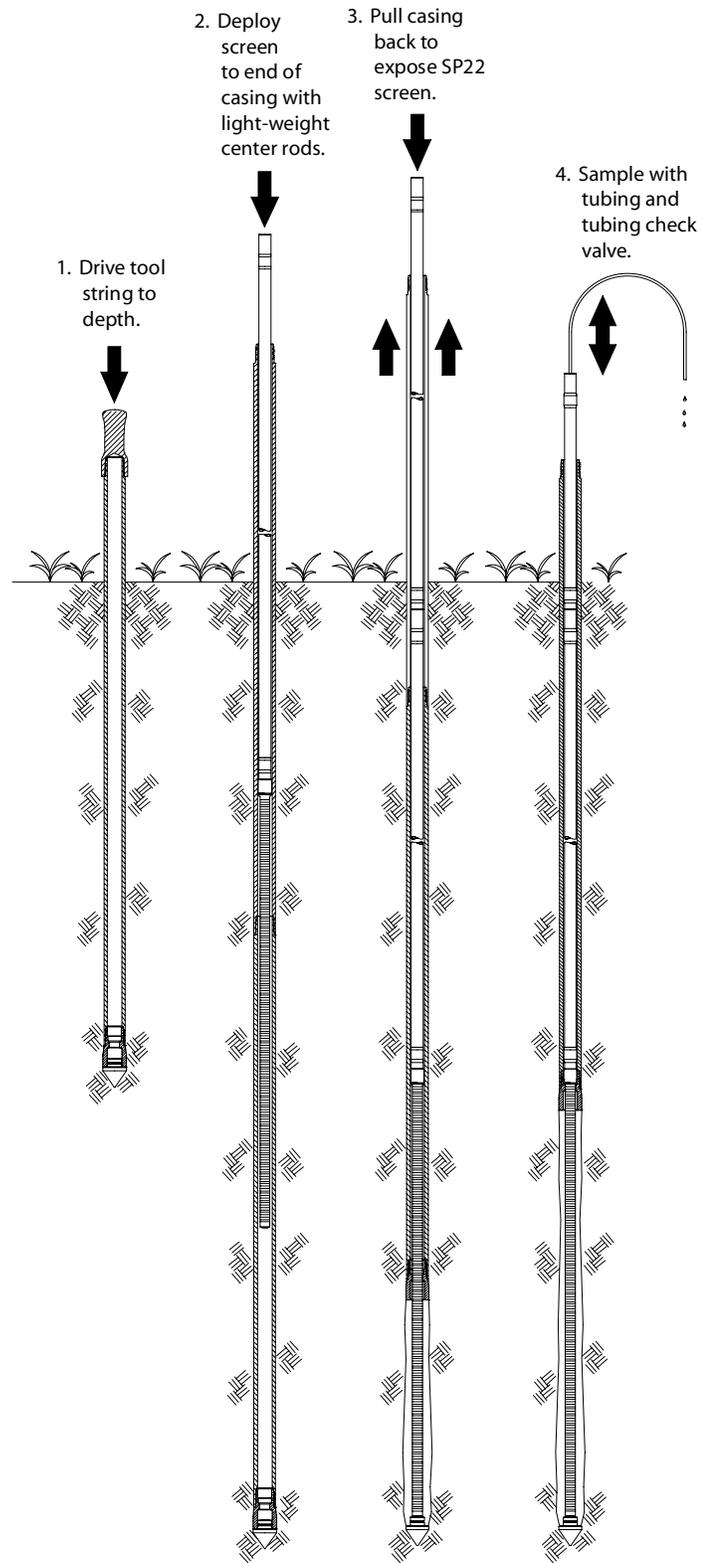
and lower it into the driven casing.

2. Lower the screen into the 2.25-inch probe rod casing and add rods or riser until the screen head contacts the bottom of the tool string.
3. Ensure that at least 48 inches (1219 mm) of rods or riser protrudes from the top 2.25-inch probe rod.
4. Maneuver the probe assembly into position for pulling.
5. Raise (pull) the outer 2.25-inch tool string while physically holding the screen in place with the inner 1.25-inch rods or 0.75-inch riser. A slight knock with the inner tool string will help to dislodge the expendable point and start the screen moving inside the probe rod.

Raise the hammer and outer tool string to expose the desired length of screen. The inner rods will begin raising with the outer rods when the screen adapter contacts the necked portion of the expendable point holder or DT22 Cutting Shoe. Use care when deploying a PVC screen so as not to break the screen when it contacts the expendable point.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top 2.25-inch probe rod.
7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.



**FIGURE 4.2**  
**Screen Deployment for SP22 Sampler**

## 4.6 Abandonment Grouting for SP22 Screens

The SP22 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of the SP22 screens, but the easiest method is to remove the inner string of rods; including the SP22 screen. A Grout Machine is then used to pump grout into the open probe hole as the outer casing is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations. (Figure 4.3)

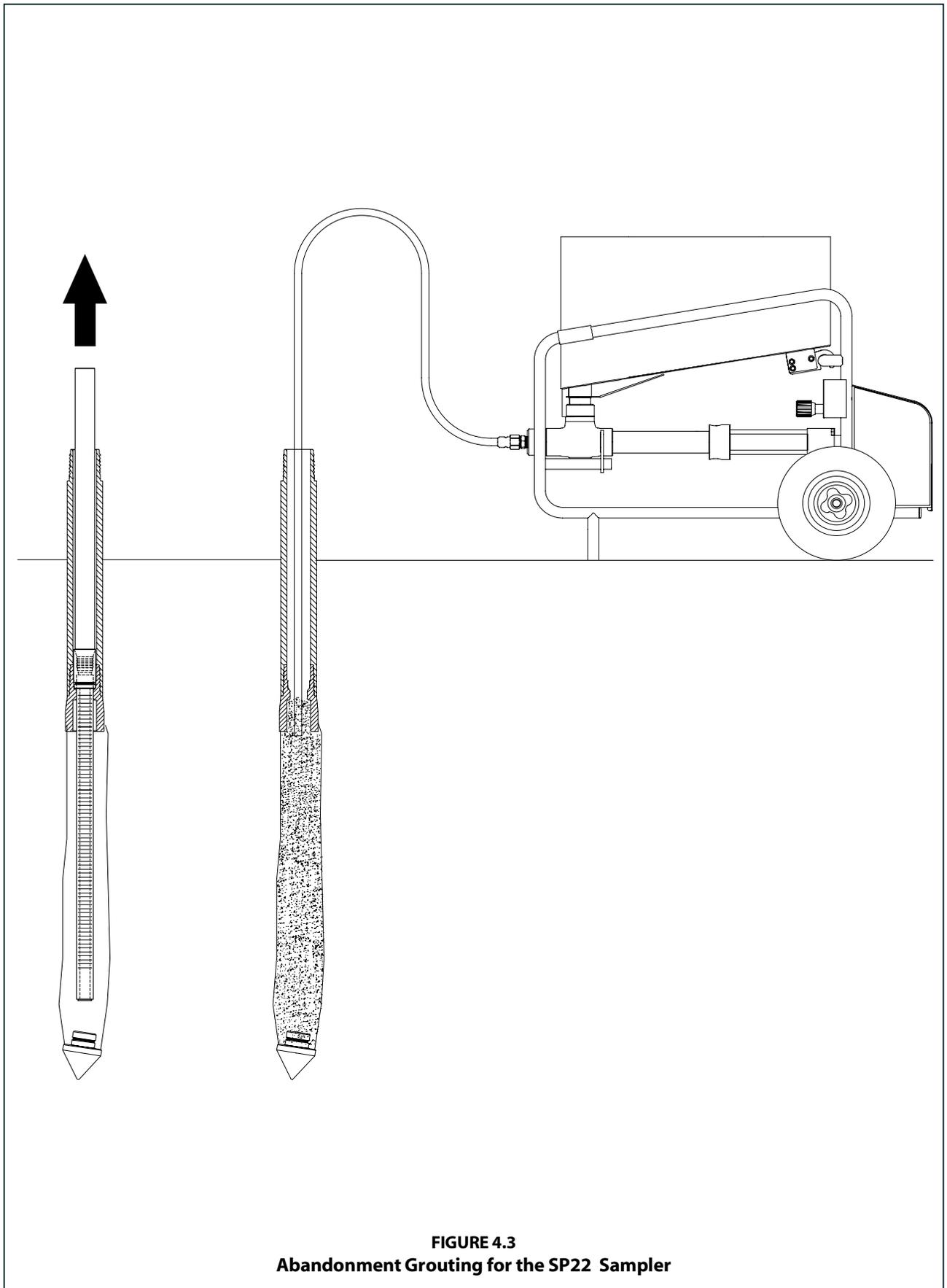
1. Maneuver the probe assembly into position for pulling.
2. High-Pressure Nylon Tubing (11633) is inserted down through the probe rods through the bottom of the expendable point holder (Fig. 4.3).

**Note:** All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.

3. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.
4. Repeat Step 5 until the tool string is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.
5. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

## 4.7 Retrieving the Screen Point 22 Sampler

If grouting is not required, the Screen Point 22 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (33622) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.



## 5.0 REFERENCES

- American Society of Testing and Materials (ASTM), 2003. D6771-02 Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations. ASTM, West Conshocken, PA. ([www.astm.org](http://www.astm.org))
- American Society of Testing and Materials (ASTM), 1993. ASTM 5299 *Standard Guide for Decommissioning of Groundwater Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities*. ASTM West Conshohocken, PA. ([www.astm.org](http://www.astm.org))
- Geoprobe Systems®, 2003, *Tools Catalog, V.6*.
- Geoprobe Systems®, 2006, *Model MB470 Mechanical Bladder Pump Standard Operating Procedure (SOP), Technical Bulletin No. MK3013*.
- Puls, Robert W., and Michael J. Barcelona, 1996. Ground Water Issue: Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures. EPA/540/S-95/504. April.
- U.S. Environmental Protection Agency (EPA), 2003. Environmental Technology Verification Report: Geoprobe Inc., Mechanical Bladder Pump Model MB470. Office of Research and Development, Washington, D.C. EPA/600R-03/086. August.

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.



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