8976 Wellington Road
Manassas, VA 20109

September 5, 2019

Jessica LaClair
New York State Department of Environmental Conservation
Division of Environmental Remediation
Remedial Bureau E, 12th Floor
625 Broadway
Albany, New York 12233-7014

Re: B335 Area RCRA Facility Investigation Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York
NYSDEC Site No. 314054

Dear Ms. LaClair:

Per NYSDEC's request dated June 7, 2019, the enclosed Resource Conservation and Recovery Act (RCRA) Facility Investigation Work Plan presents the scope of work for assessing potential subsurface sources of tetrachloroethene in the area of Building 335 at the former IBM East Fishkill Facility in Hopewell Junction, New York. Building 335 is currently owned by iPark East Fishkill LLC.

If you have any questions, please contact me at (703) 257-2583.

Sincerely yours,
International Business Machines Corporation

Dean W. Chartrand
Program Manager
Corporate Environmental Affairs

Enclosure: B335 Area RCRA Facility Investigation Work Plan

Cc:	Julia Kenney	NYSDOH	(w/enclosure via e-mail)
	Mike Buckley	iPark	(w/enclosure via e-mail)
	Carl Monheit	iPark	(w/enclosure via e-mail)
	Gary Marone	Global Foundries	(w/enclosure via e-mail)
	David Shea	Sanborn Head	(w/enclosure via e-mail)

SANBORN HEAD ENGINEERING

Building Trust. Engineering Success.

B335 AREA RCRA FACILITY INVESTIGATION WORK PLAN

Former IBM East Fishkill Facility
Hopewell Junction, New York

Prepared for IBM Corporation
File No. 2999.06
September 2019

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20 Foundry Street
Concord, NH 03301

Dean Chartrand
IBM Corporate Environmental Affairs
8976 Wellington Road
Manassas, VA 20109

September 5, 2019
File No. 2999.06

Re: B335 Area RCRA Facility Investigation Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York
NYSDEC Site No. 314054

Dear Mr. Chartrand:

The enclosed Resource Conservation and Recovery Act (RCRA) Facility Investigation Work Plan presents the scope of work for assessing potential subsurface sources of tetrachloroethene in the area of Building 335 of the former IBM East Fishkill facility. Please contact us if you have any questions.

Very truly yours,
SANBORN, HEAD ENGINEERING, P.C.

David Shea

David Shea, P.E.
Principal Engineer

JHS/DS: ds

Encl. B335 Area RCRA Facility Investigation Work Plan

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B335 AREA RCRA FACILITY INVESTIGATION WORK PLAN

Former IBM East Fishkill Facility
Hopewell Junction, New York

Prepared for
IBM Corporation

Corporate Environmental Affairs

Prepared by
Sanborn, Head Engineering, P.C.

File 2999.06
September 2019

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1.0 INTRODUCTION

This document presents IBM's work plan for assessing potential subsurface sources of tetrachloroethene (PCE) and other volatile organic compounds (VOCs) in the area of Building 335 (B335) of the former IBM East Fishkill facility in Hopewell Junction, New York (the site). This work plan is intended to address the request from the New York State Department of Environmental Conservation (NYSDEC) for a Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan (WP) (hereinafter referred to as the work plan or B335 RFI WP) to investigate potential sources of PCE in the area of B335 and evaluate the extent of contamination, as conveyed in NYSDEC's June 7, 2019 letter to IBM and the other holders of the facility's RCRA Part 373 permit[1] (i.e., iPark and Global Foundries). IBM commissioned Sanborn, Head Engineering, P.C. (Sanborn Head) to prepare this B335 RFI WP.

In 2015, site ownership was transferred from IBM to Global Foundries, who subdivided the property into 8 lots and sold 6 lots to iPark East Fishkill LLC and iPark East Fishkill I LLC (collectively referred to as either iPark or National Resources [NR]) in September 2017. A site location plan is provided as Figure 1, and the lot lines and location of the area covered by the B335 RFI WP are shown on the site plan on Figure 2. B335 is located on Lot 7, which is owned by iPark.

In the summer of 2018, iPark expressed its intent to market B335 for lease. Therefore, IBM elected to proceed with an investigation of PCE presence in indoor air and subslab vapor, the results of which were summarized in an April 2019 report.[2] Based on its review of the April 2019 report, NYSDEC indicated in the June 7, 2019 letter that B335 requires mitigation based on the New York State Department of Health (NYSDOH) Soil Vapor/Indoor Air Matrix B. In addition, the letter states that the building is not suitable for occupancy without mitigation, and that vapor mitigation and additional sampling are required before this building can be used for occupancy. The letter also states that NYSDEC has determined the data presented in the April 2019 report indicates a newly discovered release that requires the need for further investigation in accordance with Module II Condition D of the current Part 373 permit, and it notifies the Permittee owners and operators to prepare the B335 RFI WP.

1.1 Purpose and Objectives

The purpose of this work plan is to present the assessment rationale and procedures used to identify and evaluate the potential sources of PCE detected during the previous B335 vapor intrusion (VI) assessment.

The main objectives of the scope of work presented in the work plan are to:

---

1 New York State Department of Environmental Conservation, *6 NYCRR Part 373 Permit, DEC Permit No. 3-1328-00025/00249*, November 2, 2011.

2 Sanborn, Head Engineering, P.C., *B335 Data Summary Report, Former IBM East Fishkill Facility, Hopewell Junction, New York*, April 23, 2019.

- Assess for potential interior and exterior sources of target VOCs, namely PCE and its breakdown products, in the subsurface;
- Evaluate the distribution of target VOCs in soil, upper bedrock, soil vapor, and groundwater;
- Evaluate contaminant fate and transport of target VOCs; and
- If new sources are identified, collect data to support a preliminary evaluation of remedial alternatives that could appreciably improve upon IBM's current site-wide bedrock groundwater extraction and treatment remediation approach.

1.2 Organization

This work plan is organized into six sections as described below:

Section 1 presents a general introduction to the work plan;

Section 2 presents an overview of background information and the conceptual site model, including hydrogeology, distribution of VOCs, and potential source areas in the area of B335 based on existing information;

Section 3 provides a summary of the scope of work for the source area assessment, including the rationale for sampling locations and analyte list, field procedures, and quality assurance/quality control (QA/QC) measures;

Section 4 provides a summary of the data assessment and visualization procedures including project work flow;

Section 5 provides a summary of the project team organization and roles; and

Section 6 provides a summary of the proposed work plan implementation schedule.

2.0 BACKGROUND AND CURRENT CONDITIONS

This section presents an overview of background information relevant to the work plan. This overview takes into consideration the large body of data and information collected since site investigations and groundwater monitoring were initiated at the site in 1979, when IBM discovered hazardous constituents in site soil and groundwater. IBM has conducted much of this past assessment work and monitoring as part of self-initiated investigations under the oversight of NYSDEC. More detailed information can be found in various reports, including the RFI reports for site subsurface contamination[3] and the

[3] Sanborn, Head & Associates, Inc., *Task VII Deliverable, Groundwater RFI Final Report, Building 330 Area of Concern, IBM East Fishkill Facility, East Fishkill, NY*, August 22, 1997.

annual corrective action status reports,[4] previously submitted to NYSDEC in accordance with the requirements of the facility's Part 373 Permit for the site.

2.1 Site Setting

The site is in south-central Dutchess County within the Town of East Fishkill, New York. The property comprises about 500 acres adjacent to and north of Interstate Route 84 and south of Route 52 as shown on Figure 1.

IBM owned the East Fishkill facility property from approximately 1962 until 2015. It was used for the manufacturing and development of semiconductor and electronic equipment. In 2015, the property was transferred to Global Foundries who subsequently subdivided the property into eight lots, as shown on Figure 2. Six of those lots were sold to iPark in September 2017. B335 is located on Lot 7, which is owned by iPark.

The assessment area covered by this work plan includes approximately 1.5 acres of land on the southern portion of the site as shown on the facility plan provided as Figure 2. The assessment area is located within the Area C (or B330) Area of Concern (AOC) as identified in the facility's Part 373 Hazardous Waste Management Permit. The assessment area was selected to focus on B335 and proximate areas that may contain potential subsurface sources of PCE based on review of historical documents and maps.

2.2 Potential Source Areas

This section presents an overview of information within the assessment area relevant to the proposed scope of work and establishment of investigatory drilling locations for VOC source area assessment. A discussion of the former building processes within B335 and exterior utilities is provided in the subsections below.

2.2.1 Former B335 Processes

B335 formerly housed the facility's PCE recycling operations for the B330C and B330D manufacturing complex. The building and its contents were decommissioned by IBM circa 2002. The entire scope of decommissioning is uncertain, but it included removal of interior process equipment, pipes and ducts, and roof-top equipment. Refer to Figure 3 for the approximate location of former process equipment, tanks, and piping.

Five main PCE aboveground storage tanks (ASTs) were located within B335: two approximately 10,000-gallon clean PCE ASTs, one 10,000-gallon spent PCE AST, and two approximately 5,000-gallon PCE buffer storage ASTs[5]. Additional smaller PCE ASTs were also located within B335 as shown on Figure 3. Based on information provided by a former site worker, spent PCE was piped to a series of stills within B335 where PCE was boiled off

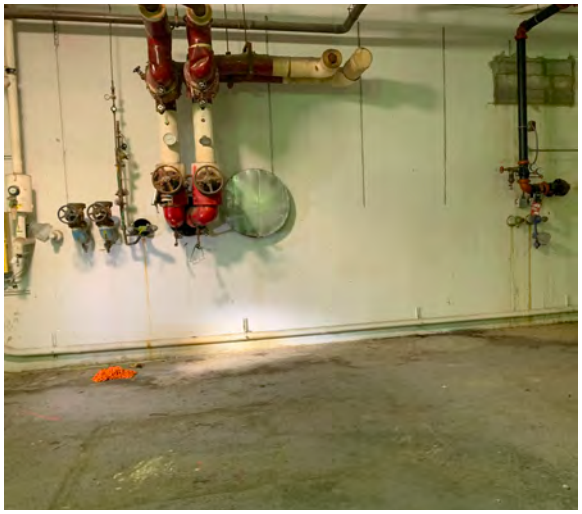
[4] Groundwater Sciences Corporation, *2018 Annual Corrective Action Status Report, Former IBM East Fishkill Facility, Town of East Fishkill, Dutchess County, New York, NYSDEC Site No. 314054, EPA ID No. NYD000707901*, May 30, 2019.

[5] Note that the PCE tank sizes and contents differ slightly between historical site plans and SWMU information in the Part 373 Permit. The information contained in the text is based on historical site plans. SWMU information from the Part 373 Permit is included in a table on Figure 3.

and re-condensed to remove the impurities. The condensed liquid went through a water separator, and the effluent PCE went to the clean PCE tanks. Exhaust from the stills was treated through activated carbon units, with waste condensate from the exhaust collected in a pit near the carbon beds. The waste condensate was then pumped back through the still system.

Sludge from the still bottoms was reportedly removed manually, and periodically shipped off-site for disposal. Subsurface PCE drain lines associated with the stills were hydrostatically tested in March 1986. Approximately 74 feet of the drain lines beneath the northeastern portion of B335 failed the hydrostatic test, and two locations along the pipe route were identified as having suspected structural defects, as shown on Figure 3. A filled trench was observed on the west side of the building that may be related to the solvent drain line, based on the location of piping and cleanouts on the solvent drain drawings. In addition, several below grade pipes were identified during the utility survey that have not been identified on historical drawings (labelled as unknown pipe on Figure 3). Capped pipes likely associated with the solvent drain lines were observed penetrating the floor slab on the north side of B335 along the solvent drain line route.

Capped pipes on the north side of B335 along the solvent drain line route



Pipe penetrations through north wall

An approximately 30-inch-diameter, capped metal duct was observed penetrating the northern wall of B335. A similar diameter, covered duct was observed to daylight at the ground surface to the north of B335. Based on historical site plans, the ducts are likely PCE exhaust ducts associated with the B335 recycling operations. Exterior utility plans show a large-diameter pipe labeled "PCE" that leads to B330C, which appears to tie into the exhaust duct. Several other smaller pipes that served as supply and return lines for chilled water (two pipes) and that may have conveyed PCE (two pipes) were observed to exit B335 through penetrations in the northern wall.

Three above-grade pipes that were used to drain the former PCE ASTs are located on the southern wall of B335. These pipes terminate and are capped outside of the building. It is uncertain where these pipes drained to when they were in use.

Five former below ground tanks were reportedly located in B335:[6,7] three PCE waste tanks, one recycled PCE tank, and one industrial waste (IW) tank. These below ground tanks have reportedly been removed, with the exception of the IW tank which is listed as active in the 2011 Part 373 permit. Several filled pits were observed in B335 during site reconnaissance visits. Although they do not line up in each case with the SWMU locations, they may have potentially been associated with the below ground tanks or other subsurface features that are not documented elsewhere.

A Loading/Unloading (L/UL) SWMU is located to the east of B335 at the top of the slope (SWMU L/UL 15). The L/UL is associated with former fuel oil tanks located to the south of B335. In addition, piping from the L/UL area enters B335 through the eastern wall where the PCE ASTs were located, indicating that this may have also been an L/UL area for PCE recycling operations.

2.2.2 Exterior Utilities and Storage Tanks

Exterior solvent-related chemical transfer and storage areas (e.g., subsurface utilities and storage tanks) in the vicinity of B335 relevant to the source area assessment are shown on Figure 4 and tabulated in Exhibit 2-1 below.

Exhibit 2-1: Exterior Solvent-Related Chemical Transfer and Storage Areas

Description	Figure 4 Designation/ SWMU Number	Location	Status in Table II-1 of 2011 Part 373 Permit
Below ground solvent waste transfer piping	Maroon utility lines	North and east of B335 (connecting to B330C and B338); B330D south dock area; between B335 and B330D south dock area	Varies (Inactive, abandoned, or closed); One segment of B330 listed as Active (unspecified location)
Below ground industrial wastewater transfer piping	Light blue utility line	South of B335	Varies (Inactive or removed)
Below ground PCE line	Dark blue utility line	North of B335	Not specifically listed in permit
Loading/unloading area	SWMU L/UL 13	B330D south dock area	Active
Loading/unloading area	SWMU L/UL 15	Between B335 and B338	Active
Former waste solvent AST	SWMU 257	Between B335 and B330C	Removed

[6] Appendix II-E, Solid Waste Management Unit Location Map, *6 NYCRR Part 373 Permit*, Effective Date 11/2/2011.

[7] IBM East Fishkill Solid Waste Management Unit Table, Dated 05/1995.

Description	Figure 4 Designation/ SWMU Number	Location	Status in Table II-1 of 2011 Part 373 Permit
Closed below ground solvent waste tanks (2)	SWMUs 86 and 168	B330D south dock area	Closed (see below for additional information)
Former above ground solvent waste tank	SWMU 3110		Active (note: AST has been removed, but closure documentation is unavailable)

Below ground waste solvent lines exited the west side of B335 and reportedly discharged to an 8,000-gallon mixed solvent waste UST (SWMU 86) and a 5,000-gallon mixed solvent waste UST (SWMU 168), both located in the B330D south dock area. SWMU 86 was reportedly exempt from certain permitting requirements including a written closure plan, but is noted as being closed in Table II-1 of the Part 373 permit. Notes from a 1995 SWMU table indicate that the tank was closed and filled with sand potentially in around 1987. According to a tank closure field report[8], SWMU 168 was a double-walled, steel UST that was installed in 1980, and cleaned and filled-in-place with gravel and concrete in 1997. Waste solvent from B330C and/or B330D also discharged to these tanks based on historical utility drawings and the 1998 tank closure field report.

In March 1995, the presence of PCE and lesser amounts of PCE breakdown products were discovered within groundwater infiltrating into stormwater drains in the B330D south dock area. PCE was detected in the storm drain system at concentrations of up to 16,599 and 16,870 micrograms per liter (µg/L), which were detected at manholes CS2849 and CS635, respectively (locations shown on Figure 4). IBM notified NYSDEC, removed liquid and solids present in the storm drain system using a vacuum truck, and sealed areas of apparent groundwater infiltration by placement of chemically resistant cured-in-place pipe lining. The presence of PCE was attributed to a historical release at the solvent loading/unloading area in the B330D South Dock Area. State Pollution Discharge Elimination System (SPDES) monitoring has indicated that this storm drain system is not a source of on-going VOC migration.

2.3 Conceptual Model of Hydrogeologic Conditions

Multiple subsurface assessments and hydrogeologic mapping projects have been undertaken at the site to support the following discussion of hydrogeologic features in the vicinity of B335. The key sources that were referenced are:

- *Supplemental Geologic Mapping, Buildings 322 and 330, IBM East Fishkill Facility, East Fishkill, New York*, prepared for IBM by Sanborn, Head & Associates, Inc., January 1996;

[8] William F. Cosulich Associates, P.C., "Closure of Tanks 157C, 168 and 169 and Sludge Dumpster Storage Area Field Report", prepared for IBM, dated January 1998.

- *RFI Task VII Deliverable, Groundwater RFI Summary Report, Building 330 Area of Concern, IBM East Fishkill Facility*, prepared for IBM by Sanborn, Head & Associates, Inc., August 1997; and

- *2016 through 2018 Annual Corrective Action Status Reports, Former IBM East Fishkill Facility,* prepared for IBM by Groundwater Sciences Corporation, various dates.

A compilation of prior mapping from the above reports that are relevant to this assessment are included in Appendix A and described throughout the work plan. In addition, the key hydrogeologic features in the B335 area of the site that impact groundwater flow and contaminant fate and transport are depicted on Figure 5 and summarized as follows:

- A northeast to southwest-oriented bedrock trough that underlies B335;

- Steeply upward sloping bedrock to the east of B335;

- An area potentially underlying and to the east of B335 with no saturated overburden; and

- Downward vertical hydraulic gradient from overburden to bedrock in the area of B335.

A cross-section depicting the vertical hydraulic gradient from overburden to bedrock in the area of B335 is included in Appendix A.2 (Sanborn Head, 1997).

Based on prior studies, groundwater in the soil and bedrock beneath the assessment area occurs as a result of infiltration of precipitation and groundwater flow from recharge at topographically higher areas to the north, east, southeast, and southwest. Two zones of groundwater saturation are present in the assessment area: (1) a localized perched zone associated with the presence of low permeability till soils, and (2) a deeper zone of saturation within the bedrock. These two "hydrogeologic units" are discussed further in the following sections.

2.3.1 Overburden Hydrogeology

Overburden within the assessment area is generally composed of three major stratigraphic units, as shown in cross-section KK-KK' included in Appendix A.1 (Sanborn Head, 1996). The major stratigraphic units are described in descending order as follows:

- Soil fill – Underlies much of the assessment area at thicknesses ranging from about 2 to 5 feet.

- Undifferentiated glacial outwash and ice-contact deposits – Consists of a mixture of poorly sorted and well-sorted sand and gravel with lesser amounts of silt, clay, cobbles, and boulders. These deposits have been observed typically within depressions in the underlying till and/or bedrock.

- Glacial till – This soil was deposited directly onto bedrock beneath the assessment area. Its thickness ranges from a few feet to approximately 20 ft to the east of B335. The till

generally consists of a heterogeneous mixture of sand, silt, and gravel, with generally greater density, and greater silt, clay, cobble, and boulder content than the outwash and ice contact deposits.

The till may act as an aquitard to vertical infiltration. In areas of significant recharge, such as in unpaved areas, the perched water table exists just above or near the top of the till layer. The overburden may be generally unsaturated in insignificant recharge areas, such as beneath B335.

Two other stratigraphic units may be present within the assessment area as follows:

- Glaciolacustrine deposits: Also referred to as the silt/clay aquitard, this layer is generally present in localized depressions within the glacial till. The silt/clay aquitard was observed in borings to the south of B335 and could potentially be encountered in the bedrock trough beneath B335.

- Colluvial/alluvial deposits – Consists of a variable sequence of silty sands, sand and gravel, and/or sand and silt, interlayered with peat and organic silt deposits. These deposits have been observed in limited areas beneath fill soil and above glacial till to the east of B330D, and could potentially be encountered in the B330D south dock area of the assessment area.

Groundwater elevation contour plans from the 2016 through 2018 Annual Corrective Action Status reports are included in Appendix A.3. Based on information provided in the 2018 Annual Corrective Action Status report, overburden groundwater (also referred to as soil groundwater) is present at depths ranging from approximately 2 to 5 feet below ground surface (bgs) in the B330D south dock area, and deeper in the vicinity of B335 (if present). Where present, overburden groundwater generally flows to the northwest. As described above and shown on the cross section in Appendix A.2, where the saturated thickness of overburden is limited or non-existent, groundwater flow may deviate from the northwesterly direction and/or be primarily downwards.

The approximate area of no saturated overburden based on previous studies is shown on Figure 5. It is uncertain whether overburden groundwater is present beneath B335. Groundwater elevation contour plans in Appendix A.3 demonstrate the variable groundwater flow pattern in this area, which may be due to the strong downward vertical gradient into bedrock near B335 as shown on the cross-section included as Figure 4 in Appendix A.2.

The overall contaminant mass flux from overburden to bedrock is expected to be small in the assessment area, limited by the relatively low permeability soils and limited recharge area. Underground utilities such as stormwater drain lines and the pipe bedding of other utility lines may influence groundwater migration pathways within the saturated overburden during seasonally high water level conditions.

Generalized conceptual depictions of the subsurface at B335 and the B330D south dock area are included below as Exhibits 2-2 and 2-3, respectively:

Exhibit 2-2: Generalized Subsurface Conditions at B335

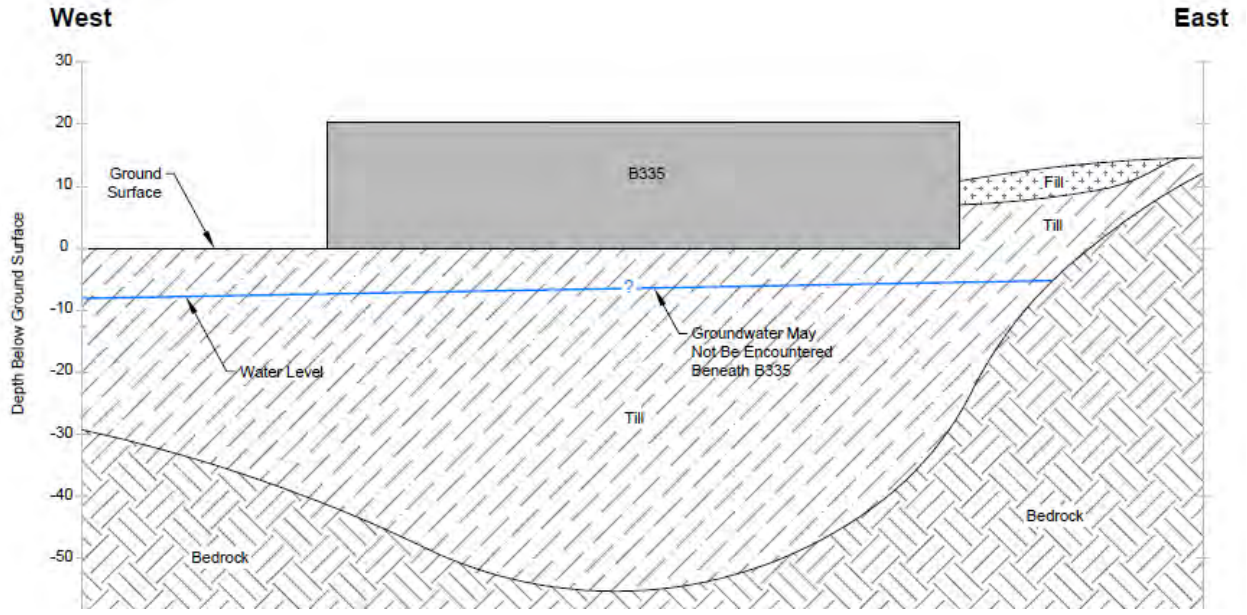
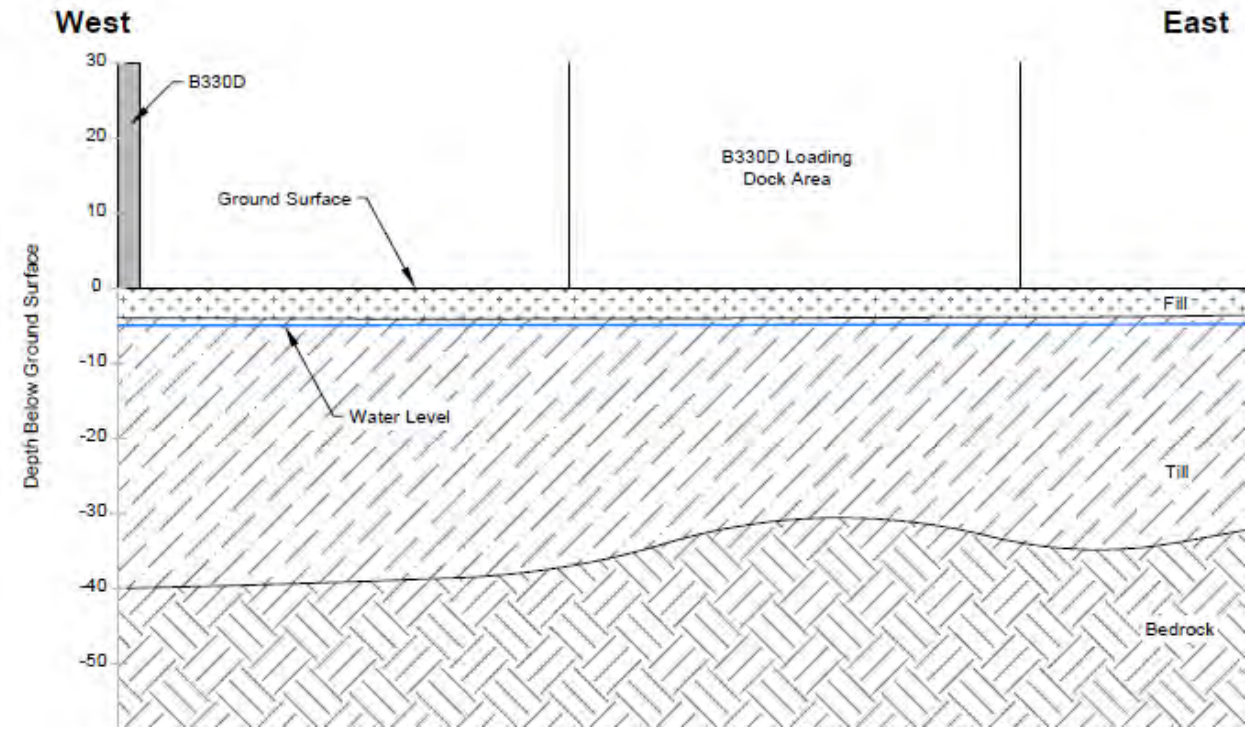


Exhibit 2-3: Generalized Subsurface Conditions at the B330D South Dock Area



2.3.2 Bedrock Hydrogeology

Bedrock beneath the assessment area consists of a sequence of dolostone interbedded with lesser amounts of limestone, fine-grained sandstone, dolomitic siltstone, and shale. The

rock is finely laminated to massive. Calcite and quartz veining are observed throughout the rock mass, oriented both parallel and perpendicular to bedding.

Areas of voids, possibly related to solution channels, have also been encountered in several locations. The degree of bedrock weathering varies greatly with location and depth from relatively fresh or faintly weathered to highly weathered and altered to a clay-rich soil-like state. The bedrock underlying the assessment area is likely to be characterized by a greater prevalence and continuity of slightly to highly weathered and fractured zones, as depicted on the cross-section included as Figure 4 in Appendix A.2.

The bedrock surface beneath the assessment area is characterized by knobs or broad mounds, and a series of narrow north-south to northeast-southwest oriented ridges and troughs. One such trough is potentially located beneath B335 as depicted on Figure 5 of the work plan.

Bedrock groundwater flow patterns beneath the assessment area include horizontal and vertical components which are influenced by variations in the extent and thickness of aquitard (glacial till) deposits, bedrock surface topography, bedrock weathering/structural variations, and the location of such hydrogeologic features relative to site production wells. Based on information provided in the 2018 Annual Corrective Action Status report, bedrock groundwater beneath the assessment area is present at depths ranging from approximately 20 to 50 feet bgs, and typically flows to the northwest towards the existing groundwater production well PW-25, which has been in operation since the early 2000s for facility groundwater supply and corrective action.

2.3.3 Contaminant Distribution

This section summarizes information regarding the distribution of VOCs in groundwater and soil vapor within and proximate to the assessment area.

VOCs observed within the assessment area consist predominantly of chlorinated ethenes (PCE, trichloroethene [TCE], cis-1,2-dichloroethene [cDCE], and vinyl chloride [VC]). PCE has been used at the site in bulk quantities. The presence of other chlorinated ethenes is attributed to chemical impurities in raw materials and/or wastes, and in-situ transformation processes.

As documented in the Annual Corrective Action Status Reports, the observed presence of VOCs in bedrock and overburden is controlled by IBM through its groundwater extraction and treatment systems.

2.3.3.1 VOCs in Overburden

VOCs present in the overburden groundwater in the assessment area predominantly consist of PCE and its breakdown products. In 2018, PCE was detected at concentrations ranging from 9.9 µg/L (MW 756) to 22 µg/L (MW 181), or parts per billion (ppb), in groundwater samples collected from existing overburden monitoring wells located within the assessment area. MW 756 is located to the south of B335, while MW 181 is located in the B330D south dock area. Low levels of PCE breakdown products were also detected in

MW 181 during the 2018 sampling event. PCE concentrations detected in 2018 are posted proximate to the monitoring wells on Figure 6. PCE concentration isopleths from the 2018 Annual Corrective Action Status Report are included in Appendix A.3.

2.3.3.2 VOCs in Bedrock

VOCs present in the bedrock predominately consist of PCE and its breakdown products and CFC-113. In 2018, PCE was detected at concentrations ranging from 300 µg/L (MW 929) to 7,300 µg/L (MW 607) in bedrock groundwater samples collected from existing monitoring wells located within the assessment area. MW 929 is located east of B330D, while MW 607 is located west of B335 in the B330D south dock area. Lower levels of PCE breakdown products were also detected in both monitoring wells during the 2018 sampling event. PCE concentrations detected in 2018 are posted proximate to the monitoring wells on Figure 6. PCE concentration isopleths from the 2018 Annual Corrective Action Status Report are included in Appendix A.3.

The bedrock groundwater VOC plume centered on Buildings 330C and 330D is currently being managed by groundwater withdrawals from production well PW-25 located to the north of the investigation area.

2.4 Summary of B335 Vapor Intrusion Investigation

A VI investigation was completed in B335 between November 2018 and February 2019 to assess impacts to subslab vapor and indoor air as a result of its former use as the facility's PCE recycling plant, and because the property owner intends to use this building. These investigations included indoor air grab sampling, passive diffusion sampling, building pressure testing and indoor air screening, subslab vapor sampling, and mass flux testing. Results of the VI assessment were presented in the April 2019 B335 Data Summary Report.[9]

The results of the VI investigation indicated that three transport mechanisms are likely contributing to the PCE presence in indoor air: off-gassing of PCE from the slab, VI through the slab by diffusion, and VI through floor cracks and other advective pathways.

PCE was detected at a concentration of 560 micrograms per cubic meter (µg/m^3) in an indoor air grab sample collected from the building in November 2018. In addition, PCE was detected in subslab vapor samples at concentrations ranging from 13,000 to 25,000,000 µg/m^3. PCE concentration isopleths in subslab vapor are depicted on Figure 7. The highest concentration of PCE in subslab vapor was detected in the area of the former stills and subgrade PCE drain pipes, including the area identified by the hydrostatic testing as containing piping having structural defects. Subslab vapor PCE concentrations were also elevated in the area of the former PCE ASTs. In general, PCE concentrations were higher in the northeastern half of the building where most of the PCE handling occurred.

[9] Sanborn, Head Engineering, P.C., *B335 Data Summary Report, Former IBM East Fishkill Facility, Hopewell Junction, New York*, April 19, 2019.

3.0 WORK PLAN ELEMENTS

This section describes the key elements of the work plan, which include a geophysical survey, shallow soil vapor screening assessment, and interior and exterior drilling and high-resolution, multi-media sampling and analysis. The work plan elements are described in detail in the following sub-sections. The scope of work was designed to meet the objectives outlined in Section 1.1 and repeated as follows:

- Assess for potential interior and exterior sources of target VOCs in the subsurface;
- Evaluate the distribution of target VOCs in soil, upper bedrock, soil vapor, and groundwater;
- Evaluate contaminant fate and transport of target VOCs; and
- If new sources are identified, collect data to support a preliminary evaluation of remedial alternatives that could appreciably improve upon IBM's current site-wide bedrock groundwater extraction and treatment remediation approach.

3.1 Work Plan Approach

The proposed scope of work will be completed in two phases. The first phase will include a subsurface utility survey and a shallow soil vapor screening program as a preliminary assessment of potential sources of PCE. Shallow soil vapor screening will be conducted at exterior locations along the presumed alignment of the below grade solvent waste lines and the locations of the USTs to obtain screening-level data on potential source areas, which will assist with identifying drilling locations for the second phase of the assessment. The approximate locations for temporary soil vapor screening samples are shown on Figure 6, and will likely be adjusted based on the results of the utility survey.

The second phase will include the advancement of borings into soil and bedrock (at a select subset of borings) at five interior (B335) and eight exterior locations as shown on Figures 6 and 7. The boring locations were placed in areas where potential solvent sources have been identified through historical documentation or existing data as described in Section 2.2. The boring locations may be adjusted to target areas of higher PCE concentrations identified during the shallow soil vapor screening. Table 1 presents the rationale for each proposed location, and Table 2 provides a summary of samples to be collected for laboratory analysis from each location.

Continuous Multichannel Tubing (CMT®) multilevel systems, manufactured by Solinst, will be installed in each boring. CMT® systems consist of a single tube with up to seven interior channels that can each be set at discrete depth intervals to sample either groundwater or soil vapor. Refer to the product sheet in Appendix B.1 for additional information on the CMT® systems, including a schematic. The purpose of installing multi-depth systems is to evaluate the vertical distribution of VOCs in the vadose zone, saturated overburden, and shallow bedrock (at select locations), and to evaluate vertical hydraulic gradients. The channel depths will be selected based on information obtained in the field as discussed in Section 3.4.3. Soil, bedrock (if encountered), groundwater (if encountered),

and soil vapor samples will be collected from each location. We expect that bedrock and groundwater may not be encountered at each location.

Soil and bedrock samples will be submitted to an on-site mobile laboratory for near real-time analysis and the results will be used evaluate the vertical extent of PCE-impacted soil or bedrock. A percentage of soil and bedrock samples will be split and submitted to an off-site fixed laboratory. Soil vapor and groundwater samples will be submitted to an off-site fixed laboratory.

3.1.1 Boring Depth Criteria

Because the primary goal of the assessment is to identify potential PCE source areas, the focus of the assessment will be on the upper approximately 20 feet of the subsurface. In addition, two to three borings will be advanced into shallow bedrock to evaluate whether dense non-aqueous phase liquid (DNAPL) and/or elevated concentrations of VOCs are located at the interface of overburden and bedrock and/or the upper portion of bedrock. Based on prior site data and location of subsurface utilities and storage tanks, these locations have been preliminarily selected as MLI-2, MLI-12, and MLI-13, but may be adjusted based on field observations.

The depth of the shallower borings will be adjusted in the field based on VOC results from the mobile laboratory and observed depths to groundwater and bedrock, as described below.

- Advance borings to 20 feet bgs;
- If PCE continues to be detected in soil at 20 feet bgs at concentrations greater than the mobile laboratory reporting limit, continue drilling until PCE is no longer detected by the mobile laboratory;
- If PCE is not detected in soil above the mobile laboratory reporting limit at 15 - 20 feet bgs, terminate boring and construct the CMT® multi-level system as described in subsequent sections;
- If bedrock is encountered before reaching the vertical extent of PCE in soil, continue drilling up to a depth of five feet into bedrock. A depth of five feet was selected to preliminarily assess PCE concentrations at and just below the till and bedrock interface.

3.1.2 Selection of Analyte List

PCE and its breakdown products were selected as the project-specific analyte list given that these compounds are the primary risk drivers for environmental media at the site. In addition, one of the primary objectives of the B335 Area RFI is to evaluate potential sources of PCE in the vicinity of B335. The analyte list will apply to soil, bedrock, groundwater, and soil vapor samples analyzed under this work plan. The project-specific analyte list is provided in Exhibit 3-2.

Exhibit 3-2: Project-Specific Analyte List

PCE
TCE
cis-1,2-DCE
trans-1,2-DCE
1,1-DCE
VC

3.2 Subsurface Utility Survey and Utility Clearance

A geophysical survey will be completed by Advanced Geological Services (AGS) of Malvern, Pennsylvania to locate and map subsurface utilities prior to the commencement of soil vapor screening and drilling operations. The purpose of the utility survey is to:

- Better understand the location and routes of potential sources such as subsurface waste solvent tanks and piping; and
- Assess for the presence of subsurface utilities at the proposed soil vapor screening and boring locations.

AGS will use a combination of the ground penetrating radar (GPR) geophysical method, radio frequency (RF) utility locating, and a hand-held electromagnetic method (HHEM). The purpose and anticipated measurement depth for each of the methods are presented below in Exhibit 3-3. Additional information on each technology is included in AGS's field standard operating procedures (SOPs) provided in Appendix B.2.

Exhibit 3-3: Utility Locating Techniques and Measurement Depths

Technology	Purpose/Use	Approximate Measurement Depth
GPR	Sweep the survey area for potential utilities; radial pattern centered on metallic anomalies identified by HHEM	5 – 10 ft bgs
RF	Trace active electric lines; trace other utilities via direct hookup or induction	5 – 10 ft bgs
HHEM	Locate buried metallic pipelines and buried metal objects	5 – 10 ft bgs

AGS will mark the subsurface features identified during the survey with spray paint and pin flags, and will provide a summary report at the conclusion of the project. The summary report will include a description of the services provided, equipment used during the project, a map of identified utilities, and overall results.

The drilling subcontractor will contact Dig Safely New York (8-1-1) at least 2 working days (not including the date of the call) prior to beginning intrusive work, as required by state law.

3.3 Shallow Soil Vapor Screening

Shallow soil vapor screening samples will be collected at the approximate locations shown on Figure 6. The temporary screening points will be installed using a hand-held Geoprobe® sampler with a Post-Run Tubing (PRT) set-up, which allows tubing to be inserted into the drive rods to the desired sampling depth. A Geoprobe® PRT product sheet is included in Appendix B.1. Sample depths will vary from approximately 2 to 5 feet bgs depending on refusal and the depth to water at each location, which is anticipated to be approximately 2 to 5 feet bgs in the B330D south dock area, and deeper at the top of the slope to the north and east of B335. A schematic of a soil vapor sampling set-up using PRT is provided as Exhibit 3-4.

Soil vapor will be sampled once the desired depth interval has been achieved and the probe rods have been retracted approximately six inches. Approximately one tubing volume will be purged using a peristaltic pump prior to sampling. Samples will be collected as short-duration grab samples using a 1-L SUMMA® canister.

After the sampling is complete, the equipment will be removed from the ground using a jack if needed. The down-hole components will be decontaminated between locations in accordance with Section 3.6.

Soil vapor screening samples will be submitted to Eurofins Air Toxics LLC (EATL) for analysis of the project-specific VOC analyte list via USEPA Method TO-15.

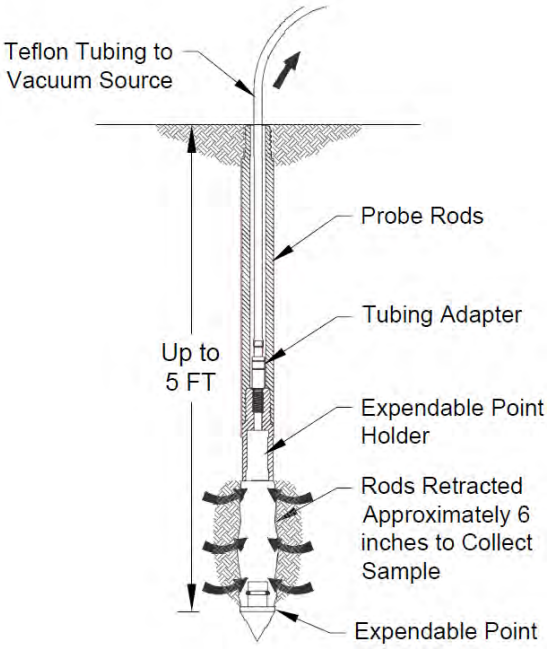


Exhibit 3-4: Schematic of PRT Soil Vapor Sampling Set-Up (Reference: Geoprobe® website)

3.4 Overburden and Bedrock Drilling

Borings are proposed at five locations inside B335, five locations immediately surrounding B335, and three locations in the B330D south dock area. The rationale for each boring location is provided in Table 1, and the locations are shown on Figures 6 and 7. Boring locations may be moved to areas of higher PCE soil vapor concentrations based on the results of the soil vapor screening.

3.4.1 Drilling Methods

Due to the presence of dense till in this area of the site, borings in both the overburden and upper bedrock will be advanced using a 3-7/8-inch outer diameter, triple-tube core barrel with rotary coring techniques and extracted using a wire-line cable system. The triple-tube core barrel system allows for the outer casing to remain in the borehole while a split inner

casing containing the soil or rock core is extracted using a wire-line system. The inner, split casing has a 3-inch inner diameter.

The purpose of coring the overburden is to decrease the amount of disturbance to the soil/bedrock core that would be expected with other drilling/sampling methods such as air or mud rotary, roto-sonic, or hollow stem augers with split spoon sampling.

Quik-Trol® Gold, a polymer manufactured by Baroid Industrial Drilling Products, will be added to the drilling water to lubricate the core bit and decrease the sediment build-up that could otherwise lock-up the core barrel components and damage the core bit. A specification sheet for Quik-Trol® Gold is included in Appendix B.3.

The drilling fluid will be recycled within each individual borehole. As the drilling fluid is delivered to the surface, it will be directed through a sand cone (desander) to remove sand from the fluid. The fluid will then be pumped back into the boring. After a boring is complete, the drilling fluid will be pumped to a frac tank where the polymer and sediment will be allowed to settle. Refer to Section 3.4.4 for a discussion of investigation derived waste (IDW) management.

Due to the presence of subgrade piping, soft-dig techniques utilizing a vacuum truck will be used to advance borings to a depth of 5 feet and 10 feet bgs at interior and exterior drilling locations, respectively.

3.4.2 Borehole Logging and Field Screening

Soil cores will be classified using the Modified Burmeister soil classification system using descriptors for color, soil components, and moisture. Bedrock cores will be classified based on visual-manual observation of the rock core for descriptors such as hardness, weathering, color, texture, rock type, rock structure, rock continuity, and rock quality designation. Photographs will be taken to visually document each core. Other field observations such as depth to water, odors, staining, and presence of non-aqueous phase liquid (NAPL) will be noted to the boring logs.

Soil/bedrock will be screened for the presence of total VOCs using a photoionization detector (PID). The soil or bedrock core will be scanned with the PID within minutes of opening the inner core barrel to assess for areas of relatively higher concentrations of VOCs. The PID readings will be recorded on the boring logs. The soil screening results along with soil/bedrock features will be used to select sample locations for mobile laboratory analysis, and to obtain a preliminary indication of relative VOC concentrations with depth.

Refer to Section 3.5.1 for a description of soil sampling for laboratory analysis.

3.4.3 Installation and Development of CMT® Multilevel Systems

CMT® multilevel systems will be installed at each of the boring locations with channels set at up to seven discrete depth intervals to sample either groundwater or soil vapor. Refer to

the product sheet in Appendix B.1 for additional information on the CMT® systems, including a schematic.

Alternating layers of filter sand and bentonite will be used to backfill the annular space to seal off the discrete intervals.

We anticipate that the CMT® groundwater sampling ports will be installed at no less than one port per ten feet of saturated drilling, and that soil vapor sampling ports will be installed at two depth intervals above groundwater.

Refer to Table 1 for the anticipated well screen intervals and soil vapor implant depths at each location.

CMT® groundwater ports will be developed prior to collecting groundwater samples. Water levels and well depths will be measured prior to development. Typically, CMT® ports will be developed based on the following criteria:

- Until field screening (e.g., pH, specific conductance) of the groundwater removed appears representative of "ambient" groundwater conditions;
- Removal of sediment inside the well screen and sump; and/or
- Production of visually clear purge water.

CMT® groundwater ports will be developed using a peristaltic pump or a small-diameter inertial pump. The pump/tubing will be lowered to the top of the port opening, and will be raised and lowered providing a surging action resulting in removal of particulates from the port and filter pack. Surging and pumping will be repeated to the bottom of the port to remove settled sediment and drilling fluids. Flow rates, duration of pumping, and visual description of the development water will be documented. For ports that go dry during development, flow rates will be reduced in an attempt to maintain a uniform development rate. No further development will be attempted for ports that are purged dry three times.

CMT® soil vapor ports will be set at two depth intervals at locations where groundwater is encountered at a depth of greater than 5 feet bgs. Where the depth to groundwater is 5 feet bgs or shallower, only one CMT® soil vapor port will be installed. The shallower soil vapor ports will be installed just above the estimated upper range of the water table. The deeper soil vapor ports will generally be installed between the estimated range of groundwater elevations and may be submerged and unable to be sampled during times of greater precipitation.

3.4.4 Investigation Derived Waste Management

Investigation derived waste to be generated during drilling activities, containment, and disposal procedures are included in Exhibit 3-5.

Exhibit 3-5: Drilling Investigation Derived Waste, Containment, and Disposal

IDW	Containment	Disposal
Soil cores	55-gallon drums	On-site reuse or off-site disposal depending on waste characterization sample results
Bedrock cores	55-gallon drums	On-site reuse or off-site disposal depending on waste characterization sample results
Soil/bedrock cuttings that settle out of the drilling fluids	Frac tank, then vacuum roll-off box or 55-gallon drums depending on volume	Off-site disposal
Sand from the desander and particulate filters from polishing prior to drilling/decon/groundwater discharge	55-gallon drums	Off-site disposal
Drilling water	Frac tank	On-site industrial wastewater treatment plant after being filtered for particulates and VOCs
Decontamination water	Frac tank with drilling water, or 55-gallon drums	On-site industrial wastewater treatment plant after being filtered for particulates and VOCs
Groundwater generated from well development	Frac tank with drilling water, or 55-gallon drums	On-site industrial wastewater treatment plant after being filtered for particulates and VOCs

As previously discussed, drilling fluids will be pumped to a frac tank after each boring has been completed. Water generated during decontamination and groundwater generated during well development will also be stored in the frac tank if possible. Otherwise, the water will be stored in 55-gallon drums.

System Floc-360®, a flocculant manufactured by Baroid Industrial Drilling Products, will be added to the frac tank upon completion of drilling activities to promote settlement of formation solids. A specification sheet for System Floc-360® is provided in Appendix B.3. The length of the settling process will depend on the size of the suspended solids, but is expected to take 1 to 2 days with the use of the flocculant. After the solids have settled, the remaining liquid will be filtered for particulates and VOCs (through activated carbon) prior to being discharged to the on-site industrial wastewater treatment plant via a nearby industrial waste pump station/manhole access port.

Bara-Kade®, a solidification agent manufactured by Bentonite Performance Minerals LLC, will be added to the sediment at the base of the frac tank for solidification purposes. A specification sheet for Bara-Kade® is provided in Appendix B.3. Alternatively, the sediment can either be pumped to a vacuum box equipped with a dewatering screen or

shoveled into 55-gallon drums, depending on the quantity. Sanborn Head will engage the on-site environmental waste management contractor to complete this process.

Drums containing solids (i.e., soil, bedrock) will be sampled for waste characterization parameters to assess whether the material can be reused on-site or if it needs to be disposed of at an off-site facility. The results will be compared to the industrial use soil cleanup objectives to assess whether the soils can be reused on-site.

Drums will be appropriately labelled, and lids will be secured when not in use. Drums will be staged near B335 at a location to be agreed upon by iPark and IBM.

3.5 Sampling and Analysis

The following sections explain the sampling procedures and analytical testing for soil, bedrock, groundwater, and soil vapor. A summary of analytical testing and sample counts by matrix is included in Table 2. Refer to the quality assurance project plan (QAPP) in Appendix C for details on field and laboratory QA/QC and analytical methods.

3.5.1 Soil and Bedrock Sampling Procedures

3.5.1.1 VOCs

Soil and bedrock samples will be collected at a frequency of approximately one sample every one to two feet of core for submittal to the on-site mobile laboratory for analysis of the list of VOCs in Exhibit 3-2.

A hand auger will be used to collect soil samples in the upper 5 to 10 feet while soft-dig procedures are being used. The vacuum truck will remove soil in one-foot intervals, and a hand auger will be advanced into the lower sidewall (if possible) or bottom of the boring to collect samples for soil logging and laboratory analysis.

Soil samples collected using the drill rig will be collected directly from the undisturbed soil core using plastic syringe samplers (e.g., Terracore or similar), and placed directly into volatile organic analysis (VOA) vials containing laboratory-grade methanol.

Bedrock samples will be collected by removing thin pieces of the core, which will be appropriately wrapped and labelled for further processing. The rock samples will be crushed on-site using a hydraulic press and transferred directly into VOA vials containing laboratory-grade methanol. Additional field sampling procedures are explained in Pace's field SOPs included in Appendix B.4.

Soil and bedrock samples submitted to the mobile laboratory will be analyzed using USEPA Method 8260 with methanol extraction (soil) or microwave assisted extraction (soil/bedrock). Soil samples may undergo microwave assisted extraction if they contain a mixture of unconsolidated and consolidated materials to provide a more aggressive extraction of VOC mass from the consolidated materials.

Approximately 10% of the soil and bedrock samples will be split and submitted to Eurofins Lancaster Laboratories of Lancaster, Pennsylvania (Lancaster) for fixed laboratory analysis. Because bedrock samples are being extracted using microwave assisted extraction, which Lancaster does not have the capability to conduct, the mobile laboratory will run the extraction and submit the extracted liquid to Lancaster for analysis.

3.5.1.2 Physical Parameters Testing

Soil and bedrock samples will be collected for the following physical parameters from two of the bedrock borings located in different portions of the assessment area: moisture content, total organic carbon (TOC), grain size distribution with hydrometer (soil only), bulk density, and porosity[10]. Samples will be collected that are representative of each of the stratigraphic units encountered while drilling. We anticipate that samples may be collected at a frequency of approximately one every five feet in up to two borings, but will be adjusted based on field observations. The purpose of collecting samples for the physical parameters testing is provided in Exhibit 3-6.

Exhibit 3-6: Purpose of Physical Parameters Testing

Parameter	Purpose
Moisture content	Evaluate fate and transport of VOCs
TOC	Evaluate fate and transport of VOCs
Grain size	Evaluate fate and transport of VOCs
Bulk density	PCE mass calculations
Porosity	Evaluate fate and transport of VOCs Mass flux calculations PCE mass calculations

Soil samples for physical parameter testing will be composited using a stainless-steel bowl and scoop and placed into laboratory-provided containers (moisture content and TOC) or plastic bags (grain size, bulk density, and porosity). Moisture content and TOC samples will be submitted to Lancaster, and grain size, bulk density, and porosity will be submitted to Geotesting Express of Acton, MA. Bedrock samples will be wrapped in foil, plastic wrap, and placed in plastic bags for shipment to the laboratory. Analytical methods are provided in Table 2.

3.5.2 Groundwater Sampling Procedures

Groundwater samples will be collected from each of the CMT® groundwater ports after they have been developed. Water levels will be measured using an electronic water level meter prior to sampling.

Groundwater samples will be collected using a peristaltic pump and dedicated silicone and polyethylene tubing. If pumps are not hydraulically capable of removing water from the port (i.e., depths of greater than about 25 ft bgs), a small-diameter inertial pump will be used to collect the sample. Where peristaltic pumps are used, groundwater samples will be

[10] Porosity will be calculated by the laboratory based on the results of bulk density, moisture content, and specific gravity testing.

collected using "modified low flow" techniques whereby select water quality parameters including specific conductance, pH, temperature, dissolved oxygen (DO), oxidation/reduction potential (ORP), and turbidity are monitored during purging; upon stabilization of the parameters, the samples will be collected. Where inertial pumps are used, samples will be collected after purging 3 to 5 port channel volumes.

Groundwater samples will be collected directly into laboratory-provided containers and shipped to Eurofins Lancaster Laboratories of Lancaster, Pennsylvania for the site-specific VOC analyte list by USEPA Method 8260C as indicated in Table 2.

3.5.3 Soil Vapor Sampling Procedures

Soil vapor samples will be collected from each of the soil vapor CMT® ports using 1-L stainless steel pre-evacuated SUMMA® canisters equipped with 1-hour flow controllers. Differential pressure measurements will be collected from each of the soil vapor CMT® ports using a digital manometer prior to collecting the soil vapor samples. SUMMA® canisters will be batch-certified clean by the analytical laboratory.

If groundwater is encountered in the implants, the implant will be purged using a peristaltic pump and a subsequent attempt will be made to collect a soil vapor sample from the implant.

Samples will be shipped to EATL for analysis of the site-specific VOC analyte list via USEPA Method TO-15.

3.5.4 Investigation Derived Waste Management

Investigation derived waste to be generated during sampling activities, containment, and disposal procedures are included in Exhibit 3-7.

Exhibit 3-7: Sampling Investigation Derived Waste, Containment, and Disposal

IDW	Containment	Disposal
Excess soil from mobile lab, including glass vials	55-gallon drums	Off-site disposal
Excess crushed bedrock from mobile lab, including glass vials	55-gallon drums	Off-site disposal
Methanol from mobile lab	10-gallon drums	Off-site disposal
Purged groundwater	Frac tank with drilling water, or 55-gallon drums	On-site industrial wastewater treatment plant after being filtered for particulates and VOCs if needed
Decontamination water	Frac tank with drilling water, or 55-gallon drums	On-site industrial wastewater treatment plant after being filtered for particulates and VOCs if needed

3.6 Equipment Decontamination

Decontamination procedures intended to limit the potential for cross-contamination are presented in Exhibit 3-8.

Exhibit 3-8: Equipment Decontamination Procedures

Equipment	Frequency	Decontamination Procedures
Drilling equipment (e.g., inner core barrel)	Before first use; Between sampling intervals	Before first use: Pressure washer on decontamination pad. Between sampling intervals: • Potable water rinse and scrub; • Non-phosphate detergent wash; and • Second potable water rinse.
Downhole tooling (e.g., casing)	Before first use; Between boring locations	Pressure washer set-up on a decontamination pad.
Well development/groundwater sampling	NA – Tubing and/or inertial pumps will be dedicated to each sampling port	
Non-dedicated soil sampling equipment (e.g., stainless steel bowls) and soil vapor screening implants	Before first use; After each use	• Potable water rinse and scrub; • Non-phosphate detergent wash; and • Second potable water rinse.
Soil vapor sampling	NA – Dedicated equipment will be used to collect soil vapor samples	

Wash and rinse water and water from pressure washer will be handled as investigation derived waste as described in prior sections of the work plan.

3.7 Horizontal and Vertical Survey

Multi-level implant locations and elevations (ground surface, road box/standpipe, and top of CMT®) will be surveyed by a licensed surveyor.

3.8 Quality Assurance/Quality Control (QA/QC)

Sampling and analysis will be conducted in accordance with the QAPP provided in Appendix C.

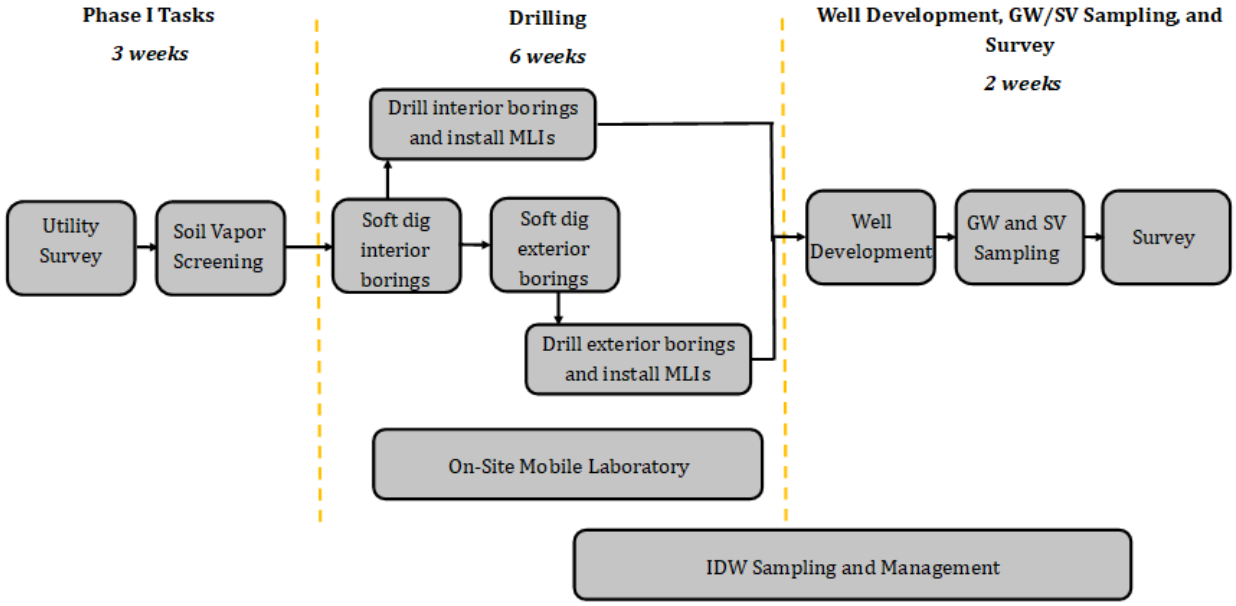
3.9 Air Monitoring

Air monitoring will be completed in accordance with the Community Air Monitoring Plan (CAMP) included as Appendix D, and the project-specific Health and Safety Plan (HASP) included in Appendix E.

3.10 Expected Work Flow

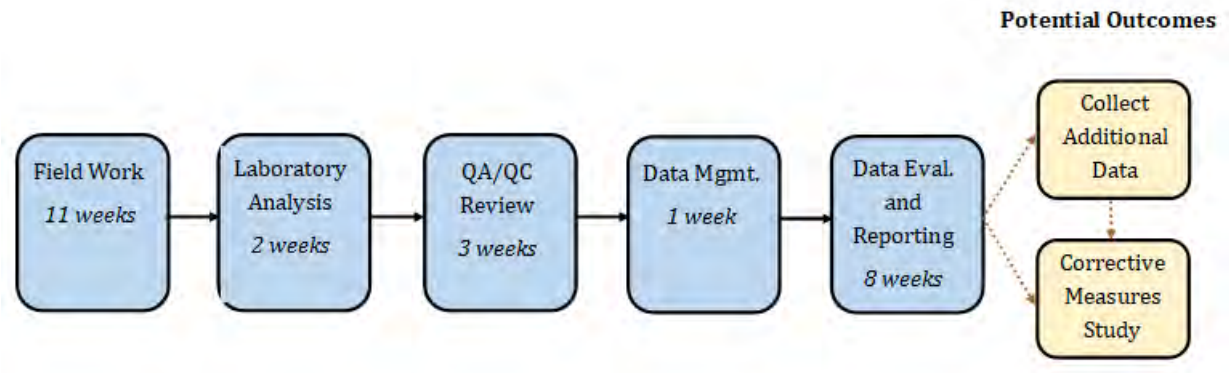
Exhibit 3-9 provides the anticipated work flow and duration for field activities, which we anticipate being approximately 11 weeks.

Exhibit 3-9: Work Flow for Field Activities



The expected work flow and duration for the RFI is provided in Exhibit 3-10 along with potential outcomes.

Exhibit 3-10: Work Flow for RFI and Potential Outcomes



3.11 Corrective Measures Engineering

If results of the RFI indicate that corrective measures are appropriate to address VOCs in the subsurface and/or indoor air, the next step would be a Corrective Measures Study (CMS). Additional field data may be required to support the CMS depending on the results of the initial program covered by this RFI Work Plan, such as additional investigation, and bench or field pilot studies to assess the feasibility of the technologies being evaluated.

Results of the previously completed VI investigation will be assessed in conjunction with the results of the RFI investigation to complete the CMS. Corrective measures may include a combination of remedial treatment technologies and mitigation technologies (i.e.,

engineering controls to address vapor intrusion). Corrective measures will depend on VOC sourcing (impacted location, depth, matrix, and concentrations), and may include one or a combination of source reduction measures and containment/control measures.

4.0 DATA MANAGEMENT, EVALUATION, AND REPORTING

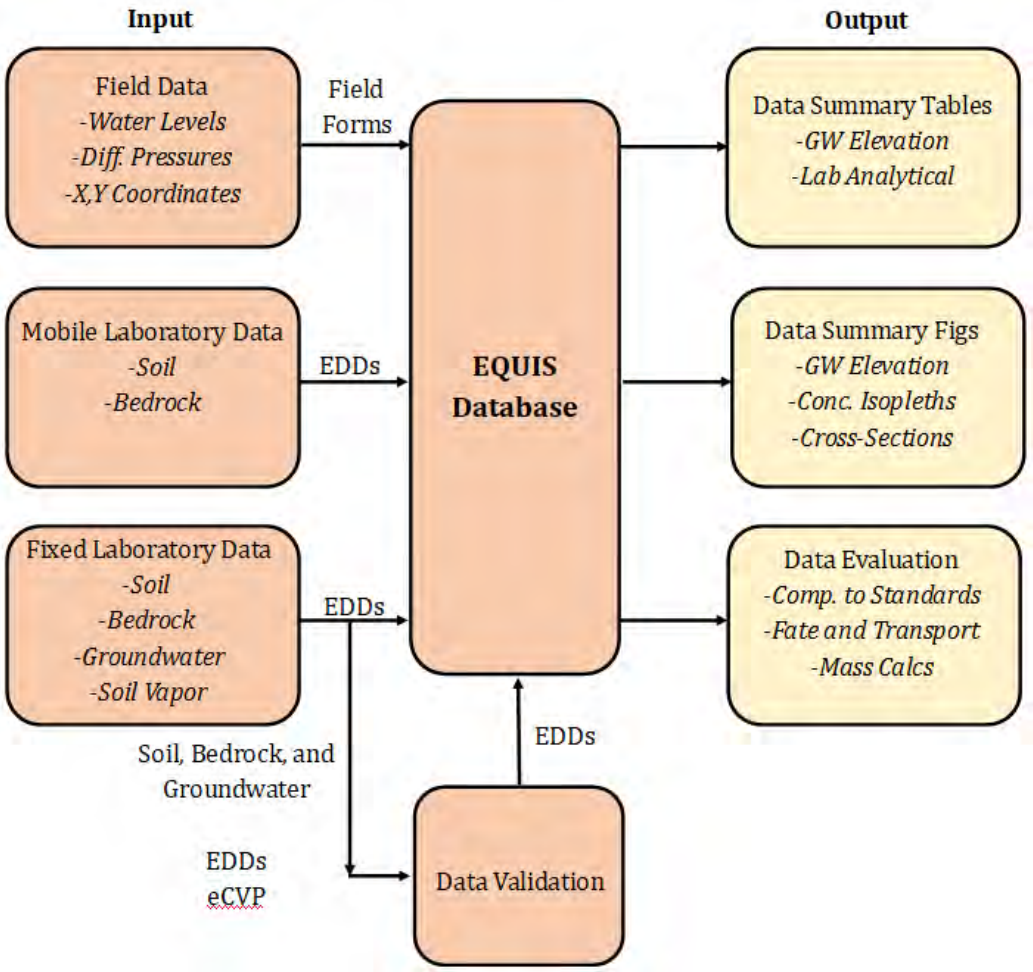
This section pertains to the management, evaluation, and reporting of data from the RFI field program.

4.1 Data Management and Visualization

Data generated as part of the proposed sampling activities will be managed in Sanborn Head's EQUIS database. The database will be used in conjunction with ArcGIS and/or Earth Volumetric Studio (EVS) to present and evaluate the spatial distribution of the data. Exhibit 3-11 presents a summary of the data management and visualization process. In general, data generated in the field will be provided to data management staff using electronic or hand-written field forms for entry into EQUIS.

Analytical data will be received from the mobile and fixed laboratories electronically and imported into EQUIS. A laboratory-generated electronic data deliverable (EDD) for the soil and groundwater samples will be sent to the data validators with columns/rows established to insert the results of the validation/usability assessment. Following completion of the validation/usability assessment, an EDD with the qualified data will be sent back to Sanborn Head electronically and inserted in place of the previously unqualified data.

Exhibit 3-11: Data Management and Visualization Process



4.2 Data Validation

In accordance with Section 2.2 of NYSDEC's DER-10,[11] A Data Usability Summary Report (DUSR) must be prepared by a party independent from the laboratory performing the analysis for all samples when Category B data deliverables are provided, including samples representing the final delineation of the nature and extent of contamination and correlation samples (i.e., split soil samples submitted to the fixed lab).

Therefore, data validation will be performed and a DUSR will be prepared by Sanborn Head's data validation subconsultant for soil, bedrock, and groundwater samples analyzed by the fixed laboratory, as these may provide information on the final delineation of the nature and extent of contamination, and/or are being analyzed as mobile laboratory correlation samples. A DUSR will not be prepared for soil vapor data, since that data is primarily intended as screening data to assess the distribution of VOCs in the subsurface, VOC mass calculations, and remedial alternatives assessments.

[11] *DER-10/Technical Guidance for Site Investigation and Remediation*, New York State Department of Environmental Conservation, DEC Program Policy, May 3, 2010.

A further discussion of data validation procedures is included in the project-specific QAPP provided in Appendix C.

4.3 Data Reporting and Communications Planning

An RFI Summary Report will be prepared and submitted to NYSDEC approximately 14 weeks after receiving the analytical data. The final report will also be provided to iPark and Global Foundries as property owners and Part 373 permit holders. We anticipate participating in a meeting with the Department to discuss the results of the RFI and proposed next steps.

5.0 PROJECT TEAM ORGANIZATION

IBM intends that the work described in this document be conducted through a project team consisting of IBM personnel and external consultants, laboratories, and contractors. Exhibit 5-1 below shows the general organization of the project team.

Exhibit 5-1: Project Team Organization

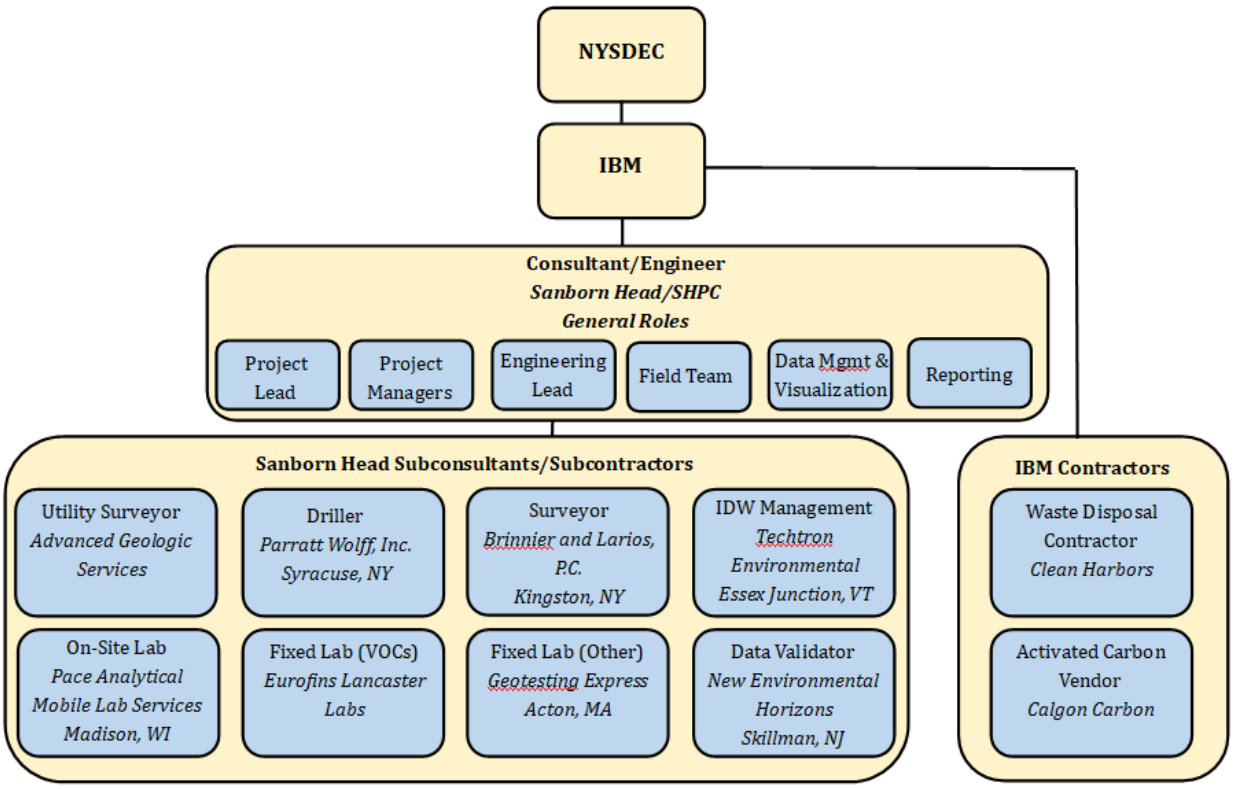


Table 4 provides further details regarding the anticipated roles of the team members along with contact information for project personnel. IBM reserves the right to modify the project team and its members while continuing to adhere to the objectives and procedures of the work plan.

6.0 SCHEDULE PROJECTION

IBM is prepared to begin the RFI field work in October 2019. This date is contingent upon approval of the work plan by the Department, subcontractor availability, and weather conditions. Given that the drilling work relies on the use of water, and the difficulties with water and IDW management during the winter months, it would be preferable to begin the field work as soon as possible.

Exhibit 6-1: Projected Schedule

Month of							
Sept 2019	**Oct 2019**	**Nov 2019**	**Dec 2019**	**Jan 2020**	**Feb 2020**	**March 2020**	**April 2020**
Dept. Review of RFI WP	Address Dept. Comments; Utility Survey; Soil Vapor Screening	Drilling/Soil Sampling IDW Management	Drilling/Soil Sampling GW and SV Sampling Laboratory Analysis IDW Management	QA/QC Review Data Management	Data Evaluation and Report Preparation		

\\conserv1\shdata\2900s\2999.06\Source Files\201909 B335 RFI Work Plan\201909-B335-RFIWP.docx

TABLES

Table 1
Summary of Proposed Explorations
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Exploration ID	Location Description	Potential Source(s) to be Assessed	Additional Objectives	Estimated Depth to Bedrock (ft bgs)	Target Boring Depth (ft bgs)	Estimated Depth to Overburden Groundwater (ft bgs) - Note 2		Target CMT® Groundwater Port Depths (ft bgs)		Target CMT® Soil Vapor Port Depths (ft bgs)	
B335 Interior						High (2018)	Low (2016)	Estimated Number of Ports	Estimated Depth	Upper	Lower
MLI-1	Former still area; North bldg	Subsurface solvent piping and PCE transfer/storage associated with former stills	Evaluate vertical gradients	47	20	4	16	2	8, 18	3	6
MLI-2	Former still area; North bldg	Subsurface solvent piping and PCE transfer/storage associated with former stills	Assess depth to bedrock; Assess for DNAPL and elevated dissolved phase PCE at bedrock surface; evaluate vertical gradients	47	52	4	14	5	10, 20, 30, top of bedrock, 5' into bedrock	3	8
MLI-3	Former PCE AST area; East bldg	Former PCE ASTs	Evaluate vertical gradients	42	20	4	17	2	8, 18	3	6
MLI-4	Near solvent pipe; West bldg	Subsurface solvent piping	Evaluate vertical gradients	41	20	3	10	2	8, 18	2	6
MLI-5	Former carbon area; South bldg	Former PCE UST	Evaluate vertical gradients	46	20	3	13	2	8, 18	2	6
Exterior - B335 Area											
MLI-6	Top of slope; East of bldg	Apparent former PCE AST filling station; Subsurface waste solvent line from B338	Evaluate vertical gradients	34	20	N/A	N/A	N/A	N/A	5	10
MLI-7	Near concrete pad containment area; South of bldg	Above grade PCE drains on southeast side of building; Apparent industrial waste line from B330D	Evaluate vertical gradients	56	20	3	18	2	10, 20	2	7
MLI-8	Near overhead door; West of bldg	Subsurface solvent piping from B335	Evaluate vertical gradients	40	20	3	9	2	8, 18	2	6
MLI-9	Base of slope; North of bldg	Subsurface solvent piping from B330C	Evaluate vertical gradients	41	20	4	9	2	8, 18	3	6
MLI-10	On slope; North of bldg	Subsurface solvent and PCE piping from B330C; Interior sources	Evaluate vertical gradients	52	30	14	23	2	15, 25	5	10
Exterior - B330D South Dock Area											
MLI-11	South of B330D south docks	Subsurface solvent piping from B330D; Former PCE release to storm drains	Evaluate vertical gradients	38	20	2	4	2	5, 15	2	N/A
MLI-12	East of B330D south docks	Subsurface solvent piping; Former waste solvent USTs and AST	Assess depth to bedrock; Assess for DNAPL and elevated dissolved phase PCE at bedrock surface; evaluate vertical gradients	30	35	3	5	5	5, 15, 25, top of bedrock, 5' into bedrock	2	N/A
MLI-13	Southeast of B330D south docks	Elevated concentration of PCE in bedrock groundwater at MW 607	Assess depth to bedrock; Assess for DNAPL and elevated dissolved phase PCE at bedrock surface; evaluate vertical gradients	32	37	3	9	5	5, 15, 25, top of bedrock, 5' into bedrock	2	N/A

Notes
1. Boring depths and CMT® sample port intervals may be adjusted in the field based on mobile laboratory results and depth to groundwater and bedrock, as discussed in the RFI Work Plan.
2. Note that the estimated depth to groundwater beneath B335 is based on monitoring wells located outside the building footprint. However, overburden groundwater may not be present beneath the building since no infiltration occurs in the building, and it is located close to the area of no saturated overburden to the east.

Table 2
Analytical Samples by Location and Matrix
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Matrix	Exploration ID	Estimated Target Depth (ft bgs)	Estimated Number of Samples						
			VOCs (Mobile Lab)	VOCs (Fixed Lab)	Physical Parameters Testing				
					Moisture Content	TOC	Grain Size w/Hydrometer	Bulk Density	Porosity
		Analytical Method	8260C	TO-15 (Vapor)/ 8260C (Soil, GW)	SM 2540 G-2011	Lloyd Kahn	ASTM D6913/D7928	Lab SOP	ASTM D854-14 and D2216-10
Soil	MLI-1	20	15	2	2	--	--	--	--
	MLI-2	52 (47 ft soil)	30	3	9	9	9	9	9
	MLI-3	20	15	2	2	--	--	--	--
	MLI-4	20	15	2	2	--	--	--	--
	MLI-5	20	15	2	2	--	--	--	--
	MLI-6	20	15	2	2	--	--	--	--
	MLI-7	20	15	2	2	--	--	--	--
	MLI-8	20	15	2	2	--	--	--	--
	MLI-9	20	15	2	2	--	--	--	--
	MLI-10	35 (30 ft soil)	20	2	6	6	6	6	6
	MLI-11	20	15	2	2	--	--	--	--
	MLI-12	30	20	2	2	--	--	--	--
	MLI-13	37 (32 ft soil)	21	2	2	--	--	--	--
	QA/QC - Field Duplicate	--	23	3	--	--	--	--	--
	QA/QC - Matrix Spike / Duplicate	--	11	2	--	--	--	--	--
	QA/QC - Equipment Rinseate Blank	--	--	--	--	--	--	--	--
	QA/QC - Trip Blank	--	--	15	--	--	--	--	--
Soil Total			***260***	***47***	***37***	***15***	***15***	***15***	***15***
Bedrock	MLI-2	52 (5 ft rock)	3	1	1	1	--	1	1
	MLI-12	35 (5 ft rock)	3	1	1	1	--	1	1
	MLI-13	37 (5 ft rock)	3	1	--	--	--	--	--
	QA/QC - Field Duplicate	--	1	1	--	--	--	--	--
	QA/QC - Matrix Spike / Duplicate	--	1	1	--	--	--	--	--
	QA/QC - Equipment Rinseate Blank	--	1	1	--	--	--	--	--
	QA/QC - Trip Blank	--	--	3	--	--	--	--	--
Bedrock Total			***12***	***9***	***2***	***2***	***--***	***2***	***2***
Groundwater	MLI-1	8, 18	--	2	--	--	--	--	--
	MLI-2	10, 20, 30, top of bedrock, 5' into bedrock	--	5	--	--	--	--	--
	MLI-3	8, 18	--	2	--	--	--	--	--
	MLI-4	8, 18	--	2	--	--	--	--	--
	MLI-5	8, 18	--	2	--	--	--	--	--
	MLI-6	N/A	--	--	--	--	--	--	--
	MLI-7	10, 20	--	2	--	--	--	--	--
	MLI-8	8, 18	--	2	--	--	--	--	--
	MLI-9	8, 18	--	2	--	--	--	--	--
	MLI-10	15, 25	--	2	--	--	--	--	--
	MLI-11	5, 15	--	2	--	--	--	--	--
	MLI-12	5, 15, 25, top of bedrock, 5' into bedrock	--	5	--	--	--	--	--
	MLI-13	5, 15, 25, top of bedrock, 5' into bedrock	--	5	--	--	--	--	--
	QA/QC - Field Duplicate	--	--	3	--	--	--	--	--
	QA/QC - Matrix Spike / Duplicate	--	--	2	--	--	--	--	--
	QA/QC - Equipment Rinseate Blank	--	--	3	--	--	--	--	--
	QA/QC - Trip Blank	--	--	3	--	--	--	--	--
Groundwater Total			***--***	***44***	***--***	***--***	***--***	***--***	***--***
Soil Vapor (Screening)	SV-1 through SV-16	2-5	--	16	--	--	--	--	--
	QA/QC - Field Duplicate	--	--	1	--	--	--	--	--
Soil Vapor	MLI-1	3, 6	--	2	--	--	--	--	--
	MLI-2	3, 8	--	2	--	--	--	--	--
	MLI-3	3, 6	--	2	--	--	--	--	--
	MLI-4	2, 6	--	2	--	--	--	--	--
	MLI-5	2, 6	--	2	--	--	--	--	--
	MLI-6	5, 10	--	2	--	--	--	--	--
	MLI-7	2, 7	--	2	--	--	--	--	--
	MLI-8	2, 6	--	2	--	--	--	--	--
	MLI-9	3, 6	--	2	--	--	--	--	--
	MLI-10	5, 10	--	2	--	--	--	--	--
	MLI-11	2	--	1	--	--	--	--	--
	MLI-12	2	--	1	--	--	--	--	--
	MLI-13	2	--	1	--	--	--	--	--
	QA/QC - Field Duplicate	--	--	3	--	--	--	--	--
Soil Vapor Total			***--***	***43***	***--***	***--***	***--***	***--***	***--***

Table 3 - Project Contact List
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Company / Name	Role	Phone	E-mail
IBM Corporate Environmental Affairs (CEA)			
Dean Chartrand	Primary IBM CEA contact for the RFI Work Plan	(703) 257-2583	chartd@us.ibm.com
Sanborn, Head Engineering, P.C. / Sanborn, Head & Associates, Inc.			
Consultant/Engineer – responsible for implementing field sampling programs, data evaluation/management, reporting, and mitigation engineering support, if necessary.			
Jennifer Sanborn	Project Lead	(603) 415-6137	jsanborn@sanbornhead.com
David Shea, P.E.	Engineering Lead	(603) 415-6130	dshea@sanbornhead.com
Joe Corsello	Project Manager/Field Implementation Lead	(603) 969-2324	jcorsello@sanbornhead.com
Sam Warner, P.G.	Project Manager/Data Management and Visualization Lead	(603) 415-6119	swarner@sanbornhead.com
Brad Green, P.G.	Technical review and support	(603) 415-6160	bgreen@sanbornhead.com
iPark			
Property Owner - responsible for site access and coordination			
Mike Buckley	Main Site Contact	(845) 765-2110	mbuckley@nationalresources.com
Jared Schlosser	Secondary Site Contact	(845) 642-6928	jschlosser@nationalresources.com
GlobalFoundries			
Former Property Owner – subcontractor access, IDW disposal coordination, medical emergencies			
Gary Marone	Snr. Engineering Manager	(845) 894-5700	gary.marone@globalfoundries.com
Eric Lutz	Env. Engineering and Operations Manager	(845) 892-3056	eric.lutz@globalfoundries.com
Emergency Control	Medical Emergency	(845) 894-3333	NA

FIGURES

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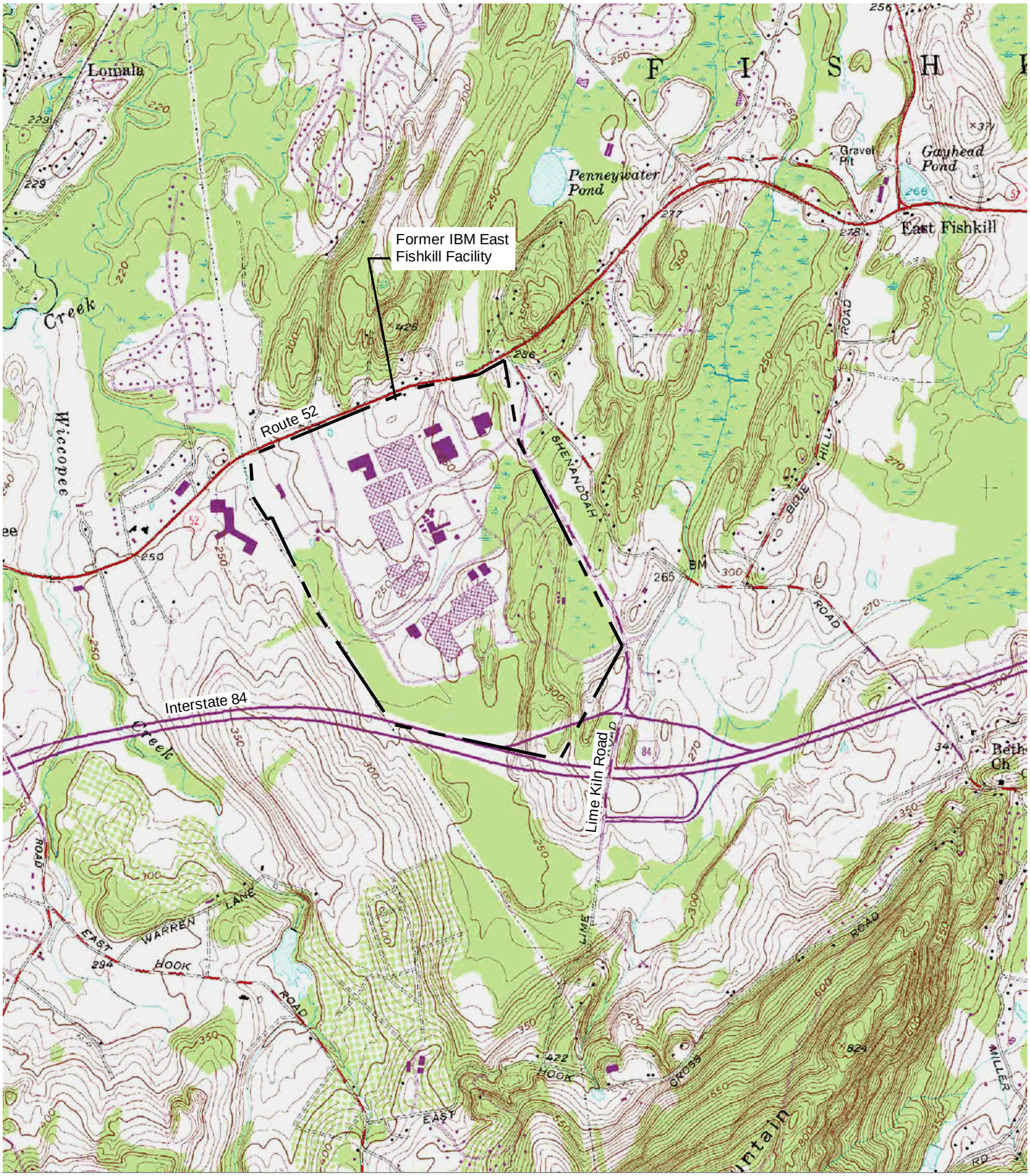

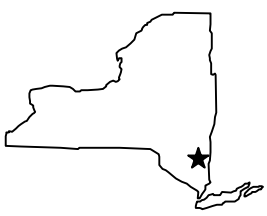

Notes:
Base map taken from 7.5 minute USGS Quadrangle Maps: Hopewell Junction, New York, Dated 1957, Photorevised in 1981.

Drawn By: E. Wright
Designed By: J. Sanborn
Reviewed By: D. Shea
Project No: 2999.06
Date: September 2019

1,000' 0 2,000' Feet

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Figure 1

Site Location Plan

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

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Figure 2

Site Plan

B335 Area RCRA Facility
Investigation Work Plan

Former IBM East Fishkill Facility
Hopewell Junction, New York

Drawn By: E. Wright
Designed By: J. Sanborn
Reviewed By: D. Shea
Project No: 2999.06
Date: September 2019

Figure Narrative

This figure shows the buildings at the former IBM East Fishkill facility, ownership information, and the approximate location of the proposed B335 Area RCRA Facility Investigation (RFI).

Legend

- Property Line
- Unlabeled features include wastewater treatment tanks, pump houses, trailers, and other structures and features not intended for human occupancy
- B320B — Indicates building number
- Approximate area of Proposed RFI
- 6 — Lot number

GlobalFoundries
- Lot 1 GlobalFoundries U.S 2 LLC
- Lot 5 GlobalFoundries U.S 2 LLC

i.Park
- Lot 2 i.Park East Fishkill I LLC
- Lot 3 i.Park East Fishkill I LLC
- Lot 4 i.Park East Fishkill LLC
- Lot 6 i.Park East Fishkill LLC
- Lot 7 i.Park East Fishkill LLC
- Lot 8 i.Park East Fishkill LLC

- Subdivision (GlobalFoundries U.S. 2 LLC)
- Subdivision (i.Park East Fishkill LLC)
- Subdivision (i.Park East Fishkill I LLC)

N W E S

200' 100' 0 200' 400' Feet

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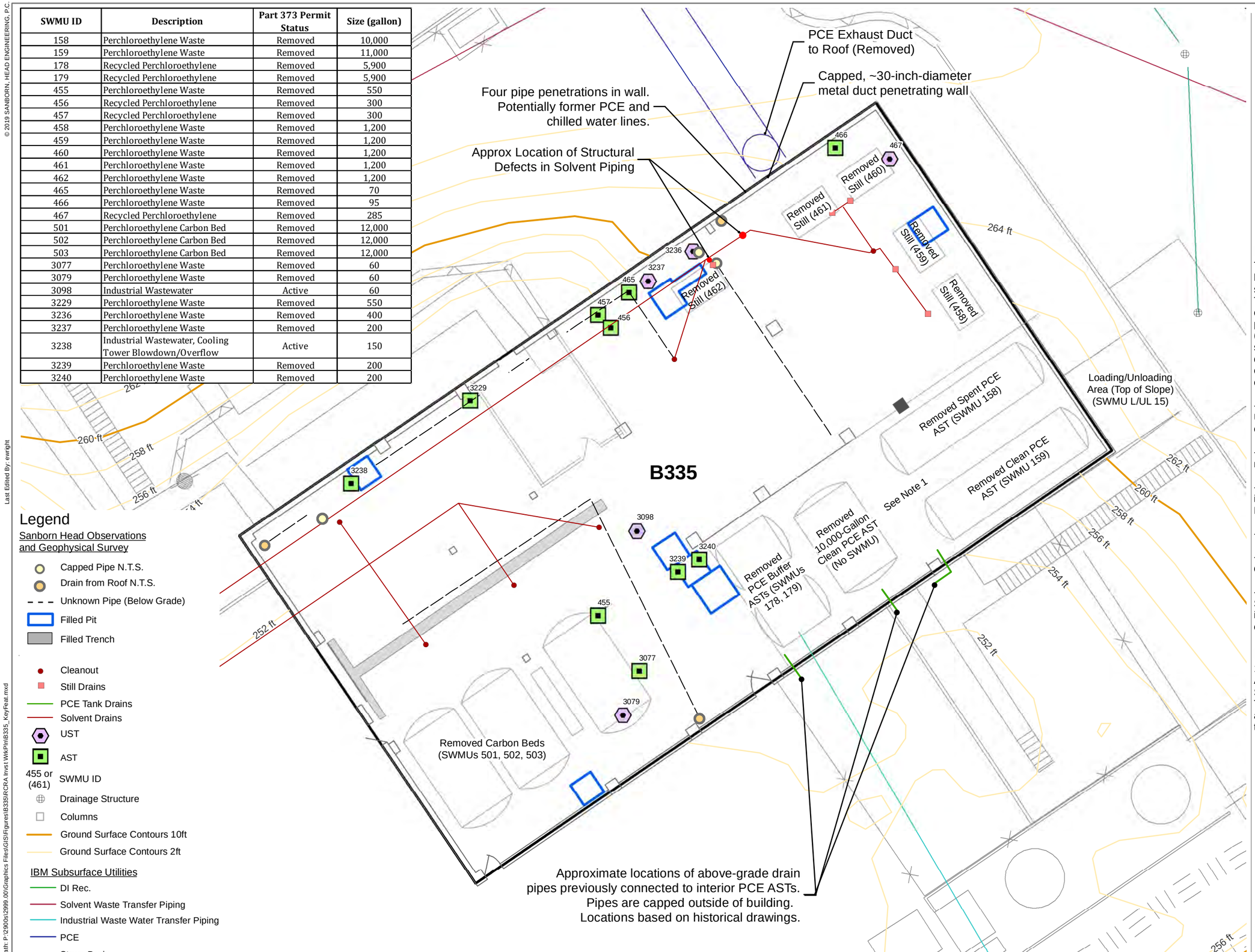
SWMU ID	Description	Part 373 Permit Status	Size (gallon)
158	Perchloroethylene Waste	Removed	10,000
159	Perchloroethylene Waste	Removed	11,000
178	Recycled Perchloroethylene	Removed	5,900
179	Recycled Perchloroethylene	Removed	5,900
455	Perchloroethylene Waste	Removed	550
456	Recycled Perchloroethylene	Removed	300
457	Recycled Perchloroethylene	Removed	300
458	Perchloroethylene Waste	Removed	1,200
459	Perchloroethylene Waste	Removed	1,200
460	Perchloroethylene Waste	Removed	1,200
461	Perchloroethylene Waste	Removed	1,200
462	Perchloroethylene Waste	Removed	1,200
465	Perchloroethylene Waste	Removed	70
466	Perchloroethylene Waste	Removed	95
467	Recycled Perchloroethylene	Removed	285
501	Perchloroethylene Carbon Bed	Removed	12,000
502	Perchloroethylene Carbon Bed	Removed	12,000
503	Perchloroethylene Carbon Bed	Removed	12,000
3077	Perchloroethylene Waste	Removed	60
3079	Perchloroethylene Waste	Removed	60
3098	Industrial Wastewater	Active	60
3229	Perchloroethylene Waste	Removed	550
3236	Perchloroethylene Waste	Removed	400
3237	Perchloroethylene Waste	Removed	200
3238	Industrial Wastewater, Cooling Tower Blowdown/Overflow	Active	150
3239	Perchloroethylene Waste	Removed	200
3240	Perchloroethylene Waste	Removed	200



Legend

Sanborn Head Observations and Geophysical Survey

- Capped Pipe N.T.S.
- Drain from Roof N.T.S.
- Unknown Pipe (Below Grade)
- Filled Pit
- Filled Trench
- Cleanout
- Still Drains
- PCE Tank Drains
- Solvent Drains
- UST
- AST
- 455 or (461) SWMU ID
- Drainage Structure
- Columns
- Ground Surface Contours 10ft
- Ground Surface Contours 2ft

IBM Subsurface Utilities

- DI Rec.
- Solvent Waste Transfer Piping
- Industrial Waste Water Transfer Piping
- PCE
- Storm Drain

Figure 3

B335 Key Features Plan

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

Drawn By:	E. Wright
Designed By:	J. Sanborn
Reviewed By:	D. Shea
Project No:	2999.06
Date:	September 2019

Figure Narrative

The purpose of this figure is to present the historical building layout, subsurface features, and solid waste management units (SWMUs) in B335. The location of former process equipment was obtained from an AutoCAD drawing B335_ALL.dwg provided by Groundwater Sciences Corporation (GSC) on January 24, 2019. The location of subsurface utilities, SWMUs, locations of structural defects in piping, and filled pits/trenches are considered approximate and are based on a combination of the following sources:

-a ground penetrating radar survey completed by Underground Surveying of Brookfield, Connecticut on January 19, 2019;

-Sanborn Head's field observations and measurements;

-Solid Waste Management Unit Location Map included in Appendix II-E of the facility's Part 373 RCRA Permit;

-Table II-1 of the facility's Part 373 RCRA Permit;

-Solid Waste Management Unit summary table dated May 1995; and

-Text excerpt and a figure ("Plate 17 - B/335 Solvent Drains") from a June 1986 report that included the results of hydrostatic testing completed for B335 subsurface solvent drains.

Notes

1. The sizes and contents of the five large PCE ASTs differ between historical site plans and SWMU information provided in the Part 373 Permit. The contents of the tanks provided on the figure are from site drawings, and the information in the table is from the Part 373 Permit.

N W E S

7.5 3.75 0 7.5 15 Feet

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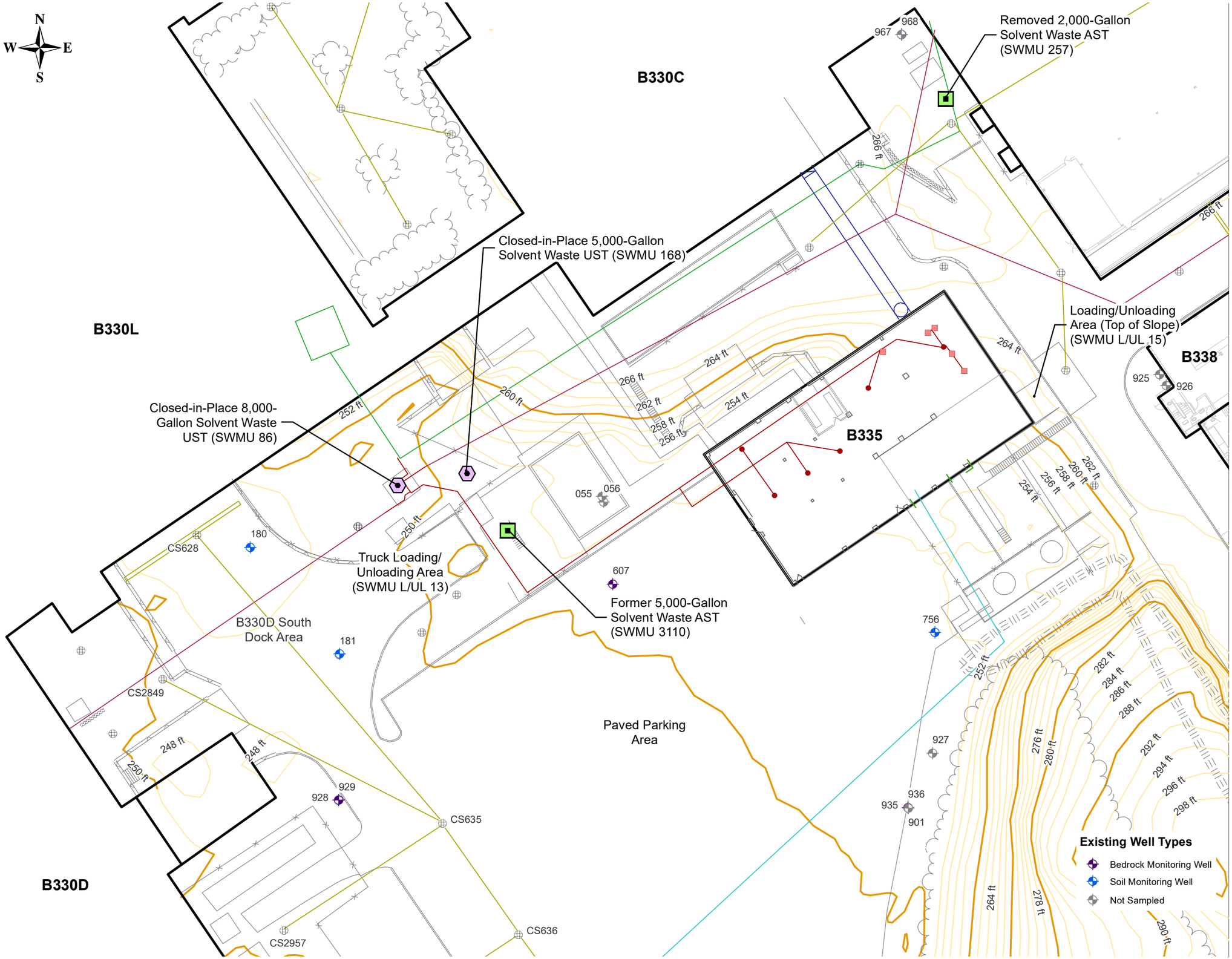

Figure 4

B335 Vicinity Plan

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

Drawn By:	E. Wright
Designed By:	J. Sanborn
Reviewed By:	D. Shea
Project No:	2999.06
Date:	September 2019

Figure Narrative

The purpose of this figure is to show key features in the vicinity of B335. The locations of exterior subsurface utilities and storage tanks are based on a combination of the following sources:

-Site utility plans provided historically by IBM;
-Solid Waste Management Unit Location Map included in Appendix II-E of the facility's Part 373 RCRA Permit;
-Solid Waste Management Unit summary table dated May 1995; and
-Text excerpt and a figure ("Plate 17 - B/335 Solvent Drains") from a June 1986 report that included the results of hydrostatic testing completed for B335 subsurface solvent drains.

All locations are approximate. Note that other subsurface utilities and tanks are or were present in the subject area; only those that are pertinent to the work being completed under the RFI Work Plan are shown.

Legend

- Cleanout
- Still Drains
- PCE Tank Drains
- Solvent Drains
- AST
- UST
- Drainage Structure
- Ground surface contours 10ft
- Ground surface contours 2ft

IBM Subsurface Utilities

- DI Rec.
- Solvent Waste Transfer Piping
- Industrial Waste Water Transfer Piping
- PCE
- Storm Drain

Existing Well Types

- Bedrock Monitoring Well
- Soil Monitoring Well
- Not Sampled

20 10 0 20 40 Feet

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Figure 5

Key Hydrogeologic Features

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

Drawn By:	E. Wright
Designed By:	J. Sanborn
Reviewed By:	D. Shea
Project No:	2999.06
Date:	September 2019

Figure Narrative

The purpose of this figure is to show key hydrogeologic features in the vicinity of B335. Bedrock surface contours, inferred area of no saturated soil, and extent of glaciolacustrine clay were taken from figures in a January 1996 report titled, "Supplemental Geologic Mapping", prepared by Sanborn Head (see Appendix A.1).

Soil (overburden) groundwater elevation contours are from Plate 3 of the "2018 Annual Corrective Action Status Report" (2018 Annual Report) prepared by Groundwater Sciences Corporation (GSC). (See Appendix A.3)

Bedrock groundwater elevation contours are provided on Plate 4 of the 2018 Annual Report, included in Appendix A.3.

B330C
B330L
B330D
B330D South Dock Area
B335
B338
Paved Parking Area
Loading/Unloading Area (Top of Slope) (SWMU L/UL 15)
180
247.44
181
246.50
607
055
056
967
968
925
926
756
248.89
927
928
929
935
936
210
230
245
190
250
260 ft
262 ft
264 ft
266 ft
284 ft
286 ft
288 ft
290 ft
292 ft
294 ft
296 ft
298 ft

Legend

- Bedrock Surface Elevation Contours 20 ft
- Soil (Overburden) Groundwater Elevation Contours (5 ft) - April 2018
- 247.44 Groundwater Elevation (feet AMSL)
- Inferred Areas of No Saturated Soil (Approximate)
- Extent of Glaciolacustrine Clay
- Ground Surface Contours 10ft
- Ground Surface Contours 2ft

Existing Well Types

- Bedrock Monitoring Well
- Soil Monitoring Well
- Not Sampled

20 10 0 20 40 Feet

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N
W E
S

Removed 2,000-Gallon Solvent Waste AST (SWMU 257)

Closed-in-Place 5,000-Gallon Solvent Waste UST (SWMU 168)

Closed-in-Place 8,000-Gallon Solvent Waste UST (SWMU 86)

Truck Loading/ Unloading Area (SWMU L/UL 13)

Loading/Unloading Area (Top of Slope) (SWMU L/UL 15)

Former 5,000-Gallon Solvent Waste AST (SWMU 3110)

B330D South Dock Area

Paved Parking Area

B335

PCE: 22 µg/l

PCE: 300 µg/l

PCE: 7,300 µg/l

PCE: 9.9 µg/l

SV-1 SV-2 SV-3 SV-4 SV-5 SV-6 SV-7 SV-8 SV-9 SV-10 SV-11 SV-12 SV-13 SV-14 SV-15 SV-16

MLI-1 MLI-2 MLI-3 MLI-4 MLI-5 MLI-6 MLI-7 MLI-8 MLI-9 MLI-10 MLI-11 MLI-12 MLI-13

CS628 CS2849 CS635 CS636 CS2957

180 181 055 056 607 756 925 926 927 928 929 935 936 901 967 968

266 ft 264 ft 262 ft 260 ft 258 ft 256 ft 254 ft 252 ft 250 ft 248 ft 282 ft 284 ft 286 ft 288 ft 292 ft 294 ft 296 ft 298 ft 276 ft 280 ft 290 ft

Figure 6

Proposed Exploration Location Plan

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

Drawn By:	E. Wright
Designed By:	J. Sanborn
Reviewed By:	D. Shea
Project No:	2999.06
Date:	September 2019

Figure Narrative

This figure shows the locations of proposed explorations to be completed as part of the B335 Area RCRA Facility Investigation Work Plan. The explorations consist of monitoring wells and soil vapor implants (collectively referred to as "Multi-Level Implants") installed at multiple depth intervals as described in the Work Plan.

Tetrachloroethene (PCE) concentrations in groundwater are from Groundwater Sciences Corporation's (GSC's) "2018 Annual Corrective Action Status Report" dated May 20, 2019 and represent the results of groundwater samples collected in April-May 2018.

Refer to previous figures for additional notes and legend.

Legend

MLI-11 — Proposed Multi-Level Implant (Monitoring Well and Soil Vapor Implants)

SV-1 — Proposed Soil Vapor Screening Locations (temporary)

PCE: 9.9 µg/l — PCE in Soil Groundwater

PCE: 300 µg/l — PCE in Bedrock Groundwater

Existing Well Types

- Bedrock Monitoring Well
- Soil Monitoring Well
- Not Sampled

IBM Subsurface Utilities

- DI Rec.
- Solvent Waste Transfer Piping
- Industrial Waste Water Transfer Piping
- PCE
- Storm Drain

20 10 0 20 40 Feet

SANBORN HEAD ENGINEERING

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Last Edited By: ewright

Path: P:\2900s\2999.00\Graphics Files\GIS\Figures\B335\RCRA Invst WkPlnB335_Bld_PELP.mxd

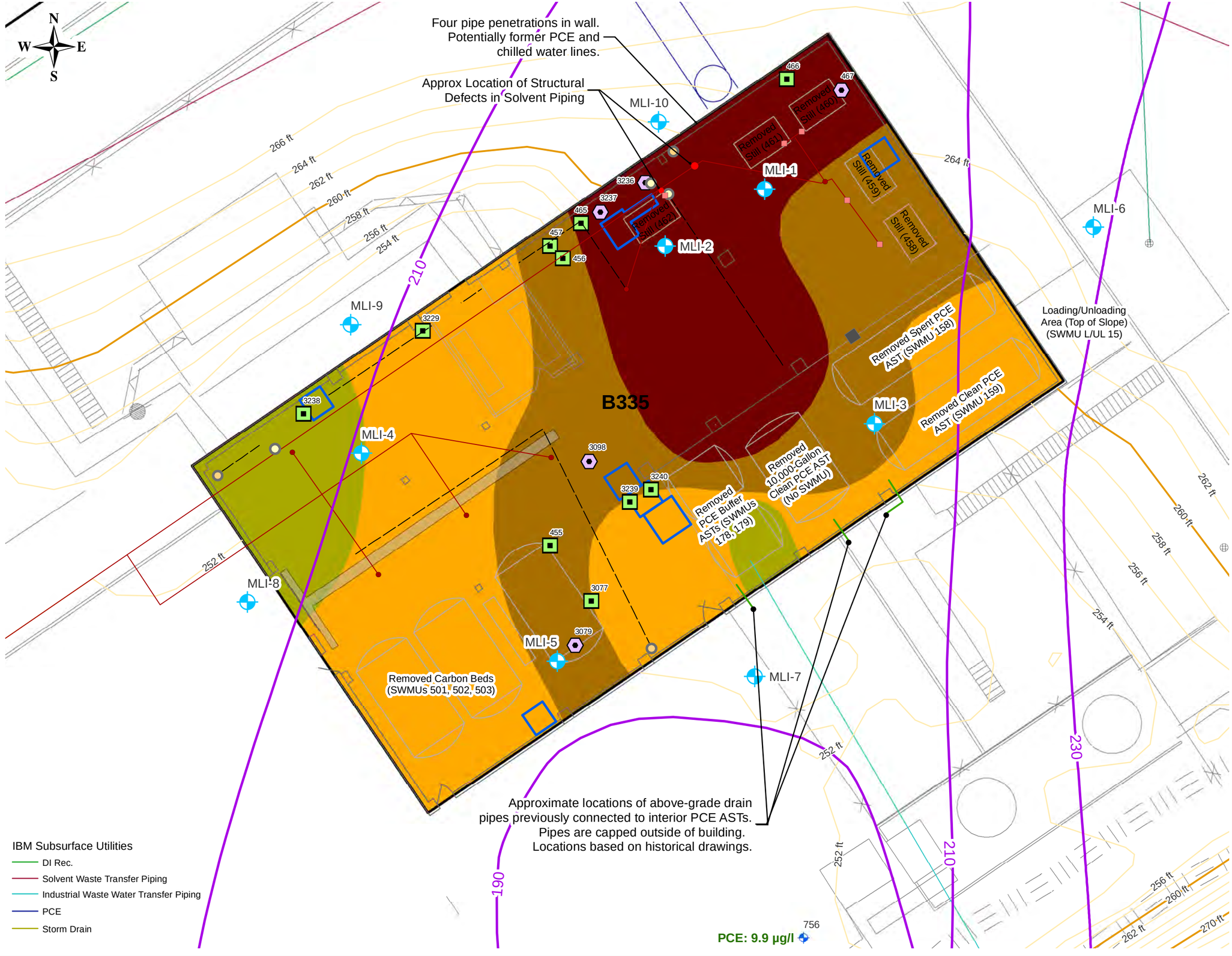

Figure 7

B335 Proposed Exploration Location Plan

B335 Area RCRA Facility Investigation Work Plan

Former IBM East Fishkill Facility

Hopewell Junction, New York

Drawn By:	E. Wright
Designed By:	J. Sanborn
Reviewed By:	D. Shea
Project No:	2999.06
Date:	September 2019

Figure Narrative

This figure shows the locations of proposed borings to be completed within and immediately adjacent to B335 as part of the B335 Area RCRA Facility Investigation Work Plan. The borings will be completed as monitoring wells and soil vapor implants (collectively referred to as "Multi-Level Implants") installed at multiple depth intervals as described in the Work Plan.

Bedrock surface elevation contours are from Figure 2 of Sanborn Head's "Supplemental Geologic Mapping Report", dated January 1996.

Tetrachlorethene (PCE) concentration isopleths in subslab vapor were taken from Figure 6 of Sanborn Head's "B335 Data Summary Report" dated April 2019 and are based on subslab vapor samples collected on January 30, 2019.

PCE concentrations in groundwater are from Groundwater Sciences Corporation's (GSC's) "2018 Annual Corrective Action Status Report" dated May 20, 2019 and represent the results of groundwater samples collected in April-May 2018.

Refer to previous figures for additional notes and legend.

Legend

MLI-1 — Proposed Multi-Level Implant (Monitoring Well and Soil Vapor Implants)

230 — Bedrock Surface Elevation Contours

PCE: 9.9 µg/l — PCE in Soil Groundwater

PCE Concentration in Subslab Vapor (µg/m³)

- <50,000
- 50,000 - 500,000
- 500,000 - 5,000,000
- >5,000,000

7.5 3.75 0 7.5 15 Feet

SANBORN HEAD ENGINEERING

APPENDIX A

REFERENCED FIGURES FROM HISTORICAL REPORTS

APPENDIX A.1

FIGURES 1 THROUGH 10 OF JANUARY 1996 SUPPLEMENTAL GEOLOGIC MAPPING REPORT

IBM EAST FISHKILL FACILITY

EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
MONITORING WELL LOCATION PLAN

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 1 OF 1

DRAWN BY: MRP

DESIGNED BY:

CHECKED BY:

REVIEWED BY:

PROJECT MGR:

PIC:

DATE: MAR 96

SCALE: 200 100 0 200 400

1 INCH = 200 FEET

SHA

Sanborn, Head & Associates

Consulting Engineers & Scientists

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY

NOTES:

1. THE BASE PLAN WAS DEVELOPED FROM AN AUTOCAD FILE OF A FIGURE ENTITLED "SOIL BORING LOCATION PLAN, IBM EAST FISHKILL FACILITY," ORIGINAL DATE UNKNOWN AND AUTHOR UNKNOWN.

2. THE LOCATIONS SHOWN FOR GROUNDWATER MONITORING WELLS, BEDROCK PRODUCTION WELLS, TEST BORINGS AND EXISTING GEOLOGIC CROSS-SECTIONS ARE BASED UPON AN AUTOCAD FILE ENTITLED "LINK0.DWG," DRAWN BY DAVID E. SPEED OF IBM CORPORATION AND DATED JUNE 5, 1992.

3. THE GEOLOGIC CROSS-SECTIONS WHICH WERE REVISED OR CREATED BY SHA WERE DEVELOPED WITH THE INTENT OF ILLUSTRATING CURRENT INTERPRETATIONS OF SUBSURFACE GEOLOGIC CONDITIONS TO SUPPORT REMEDIAL DESIGN CONSIDERATIONS IN THE GROUNDWATER RECOVERY ACT (GRA) FACILITY INVESTIGATIONS FOR THE AREAS OF BUILDINGS 322 AND 330.

4. THE APPROXIMATE AREAL LIMITS OF MAPPING ARE BASED UPON LIMITS OF PREVIOUS SURFICIAL MAPPING AND THE LOCATIONS OF GEOLOGIC CROSS-SECTIONS IN THE GRA FACILITY AREA OF BUILDING 330. PLEASE REFER TO APPENDIX A FOR LIMITATIONS CONCERNING THE REVISION / CREATION OF THE SURFACE MAPS AND CROSS-SECTIONS.

LEGEND:

- LOCATION AND DESIGNATION OF GROUNDWATER MONITORING WELL
- LOCATION AND DESIGNATION OF BEDROCK PRODUCTION WELL
- LOCATION AND DESIGNATION OF TEST BORING
- J — J' LOCATION AND DESIGNATION OF EXISTING GEOLOGIC CROSS-SECTION LINE (NOT UPDATED JANUARY 1996 (SEE NOTE 3)
- KK — KK' LOCATION AND DESIGNATION OF GEOLOGIC CROSS-SECTION LINE CREATED JANUARY 1996 (SEE NOTE 3)
- K — K' LOCATION AND DESIGNATION OF EXISTING GEOLOGIC CROSS-SECTION LINE (NOT UPDATED)

PREVIOUS SEGMENT OF CROSS-SECTION E–E'

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
BEDROCK SURFACE ELEVATION CONTOUR PLAN

PROJECT NUMBER: 11765
FIGURE NUMBER: 2 OF 10

DRAWN BY: MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

SCALE
200 100 0 200 400
1 INCH = 200 FEET

SHA
Sanborn, Head & Associates
Consulting Engineers & Scientists

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY

NOTES:

1. THE BEDROCK SURFACE AND REFUSAL ELEVATIONS AS DEPICTED WERE DETERMINED BASED UPON A REVIEW OF ORIGINAL LOGS OF TEST BORINGS AND MONITORING WELL LOGS AND A PHYSICAL WELL DATABASE FURNISHED BY IBM CORPORATION. THE ELEVATIONS ARE RELATIVE TO THE NATIONAL GEODETIC VERTICAL DATUM OF 1929.

2. THE LOCATIONS OF BEDROCK OUTCROPS AS SHOWN ARE GENERALLY FROM A PLAN ENTITLED "PLATE 1, GEOLOGIC MAP, IBM CORPORATION, EAST FISHKILL," PREPARED BY R. E. WRIGHT ASSOCIATES, INC., DATED FEBRUARY 17, 1987. ORIGINAL SCALE: 1" = 200'.

3. "BEDROCK SURFACE" ELEVATIONS IN AREAS OF BEDROCK OUTCROPS ARE BASED UPON TOPOGRAPHIC CONTOURS SHOWN ON A PLAN ENTITLED "BEDROCK STRUCTURE MAP – FIELD MEASUREMENTS," PREPARED BY R. L. LAFLEUR AND DATED AUGUST 1989. ORIGINAL SCALE: 1" = APPROXIMATELY 200'.

4. "CONFIRMED BEDROCK" SURFACE ELEVATIONS REPRESENT THE TOP OF WEATHERED OR COMPETENT BEDROCK AT EXPLORATION LOCATIONS WHERE BEDROCK WAS SAMPLED OR PENETRATED DURING DRILLING. "REFUSAL" ELEVATIONS REPRESENT LOCATIONS WHERE DRILLING WAS TERMINATED DUE TO DRILL STRING REFUSAL ON A HARD SURFACE.

5. THE BEDROCK SURFACE ELEVATION CONTOURS WERE DEVELOPED BASED UPON CONFIRMED BEDROCK SURFACE ELEVATIONS AND BEDROCK OUTCROP INFORMATION, SUPPLEMENTED WHEN APPROPRIATE WITH REFUSAL ELEVATION INFORMATION. THE CONTOURS ARE INTENDED TO SHOW INFERRED TRENDS IN BEDROCK SURFACE TOPOGRAPHY CONSISTENT WITH AVAILABLE INFORMATION. IN AREAS WHERE BEDROCK OUTCROPS ARE ABSENT, THE CONTOURS WERE ADJUSTED TO BE CONSISTENT WITH GEOLOGIC PROFILES SHOWN ON FIGURES 5 THROUGH 10.

6. SEE FIGURE 1 FOR ADDITIONAL NOTES AND LEGEND, AND APPENDIX A FOR LIMITATIONS.

LEGEND:

- PW-5 & 5A — LOCATION AND DESIGNATION OF BEDROCK PRODUCTION WELL
- MW-300 — LOCATION AND DESIGNATION OF GROUNDWATER MONITORING WELL
- 0855 — LOCATION AND DESIGNATION OF TEST BORING
- (184.4) — CONFIRMED BEDROCK SURFACE ELEVATION
- [212.7] — REFUSAL ELEVATION
- 230 — BEDROCK SURFACE ELEVATION CONTOUR
- 190 — DENOTES DEPRESSION IN BEDROCK SURFACE GREATER THAN CONTOUR INTERVAL OF 20 FEET
- LOCATION OF BEDROCK OUTCROP OR AREA OF CLOSELY SPACED BEDROCK OUTCROPS
- AREA WHERE BEDROCK IS INFERRED TO BE DIRECTLY BELOW FILL

FILE: M:\ARCH\11765\SHW\PMG.DWG
REF: FILES: M:\ARCH\11765\SHW\BEDSURF.DWG
XREF: DATE: 01-18-96

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
TILL SURFACE ELEVATION CONTOUR PLAN

PROJECT NUMBER: 1176.5
FIGURE NUMBER: 3 OF 10

DRAWN BY: MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

SCALE: 200 100 0 200 400
1 INCH = 200 FEET

Sanborn, Head & Associates
Consulting Engineers & Scientists

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY

NOTES:

1. THE "TILL SURFACE" ELEVATION CONTOUR PLAN WAS REVISED FROM AN AUTOCAD FILE ENTITLED "LINKBASE.DWG" OBTAINED FROM THE FILE AREA OF BUILDING 375 OF THE IBM CORPORATION, APPROVED BY DES / TGW OF IBM, AND DATED DECEMBER 1982.

2. THE "TILL SURFACE" ELEVATIONS SHOWN REPRESENT SURFACE ELEVATIONS FOR ARGILLACEOUS TILL (SILT, CLAY, AND VARIABLE AMOUNTS OF SAND AND GRAVEL) WHERE IT IS THE HIGHEST STRATIGRAPHIC UNIT. THE ELEVATIONS ARE RELATIVE TO THE NATIONAL GEODETIC VERTICAL DATUM OF 1929. THE INDIVIDUAL SPOT ELEVATIONS SHOWN ON THE FIGURE REPRESENT A SUBSET OF THE TOTAL EXPLORATIONS USED / CONSIDERED IN THE PREPARATION OF THE FIGURE. IN GENERAL, EXPLORATIONS USED / POSTED AS IN WHICH THE TILL SURFACE IS CONSIDERED TO BE THE HIGHEST REVISED OR EXTENDED (AREA SOUTH OF N8000 AND EAST OF E5000). THE REMAINDER OF EXPLORATIONS WERE OMITTED IN THE INTEREST OF CLARITY BUT CAN BE VIEWED ON THE ORIGINAL "LINKBASE.DWG" OR BY SUPERIMPOSITION OF THIS FIGURE ONTO FIGURE 1.

3. THE ELEVATION CONTOURS WERE DEVELOPED BASED UPON ELEVATIONS DETERMINED FROM EXPLORATION LOGS SUPPLEMENTED BY SURFACE TOPOGRAPHY IN UNDEVELOPED PORTIONS OF THE SITE AS SHOWN ON FIGURE 1. CONTOURS ARE INTENDED TO SHOW TRENDS IN TILL SURFACE TOPOGRAPHY CONSISTENT WITH AVAILABLE INFORMATION. IN AREAS WHERE EXPLORATIONS ARE ABSENT THE CONTOURS WERE ADJUSTED TO BE CONSISTENT WITH THE TOPOGRAPHY SHOWN ON FIGURE 1. THROUGHOUT MUCH OF THE CENTRAL BUILDING AREA (B. 330 AREA) WERE DEVELOPED ASSUMING TILL DEPOSITS AT ELEVATIONS ABOVE 260 FEET WERE EXCAVATED AS PART OF CONSTRUCTION OF THE BUILDINGS

4. SEE FIGURE 1 FOR ADDITIONAL NOTES AND LEGEND, AND APPENDIX A FOR LIMITATIONS.

LEGEND:

- MW-195 — LOCATION AND DESIGNATION OF GROUNDWATER MONITORING WELL
- B-58 — LOCATION AND DESIGNATION OF TEST BORING
- (223.0) — APPROXIMATE TILL SURFACE ELEVATION
- 235 — TILL SURFACE ELEVATION CONTOUR (INTERVAL VARIES)
- 220 — DENOTES DEPRESSION IN TILL SURFACE
- BEDROCK OUTCROP, AREA OF CLOSELY SPACED BEDROCK OUTCROPS, AND / OR AREA WHERE TILL IS INFERRED TO BE ABSENT

FILE: M:\MACRO\FY1176\FWPMG.DWG
REF: FILE: M:\MACRO\FY1176\TILSURF.DWG
PLOT DATE: 01-19-96

NOTES:

1. THE "CLAY SURFACE" ELEVATION CONTOUR PLAN WAS REVISED FROM AN AUTOCAD FILE ENTITLED "LINKBASE.DWG," DRAWN BY TLM / PAD OF IBM CORPORATION, APPROVED BY DES / TGW OF IBM, AND DATED DECEMBER 1989.

2. THE "CLAY SURFACE" ELEVATIONS WERE CHECKED OR IDENTIFIED BASED UPON A REVIEW OF BORING LOGS AND GROUNDWATER MONITORING WELL LOGS. THE ELEVATIONS SHOWN ARE INTERPRETED TO REPRESENT THE ELEVATION FOR THE TOP OF FINE-GRAINED GLACIOLACUSTRINE SOILS INCLUDING: CLAY, CLAYEY-SILT, OR SILTY-CLAY WITH LESSER AMOUNTS OF SAND. THE ELEVATIONS ARE RELATIVE TO THE NATIONAL GEODETIC VERTICAL DATUM OF 1929. THE INDIVIDUAL SPOT ELEVATIONS SHOWN ON THE FIGURE REPRESENT A SUBSET OF THE TOTAL EXPLORATIONS USED / CONSIDERED IN THE PREPARATION OF THE FIGURE. IN GENERAL, THE EXPLORATIONS ARE POSTED IN AREAS IN WHICH THE ORIGINAL FIGURE CONTOURS WERE REVISED OR EXTENDED. THE REMAINDER OF THE EXPLORATIONS WERE OMITTED IN THE INTEREST OF CLARITY BUT CAN BE VIEWED ON THE ORIGINAL "LINKBASE.DWG" OR BY SUPERIMPOSITION OF THIS FIGURE ONTO FIGURE 1.

   * THE "CLAY" DEPOSITS DESCRIBED ON EXPLORATION LOGS MW-87 / 88, MW-140 / 141 / 142 / 143, MW-166 / 167, MW-168 / 169, AND MW-178, WERE INTERPRETED TO CONSIST OF WEATHERED CLAYEY TILL AND WERE THEREFORE NOT INCLUDED ON THE PLAN.

   * THE "SILT AND CLAY" DEPOSITS DESCRIBED ON THE EXPLORATION LOG FOR MW-958 WERE DISCOUNTED BASED UPON A REVIEW OF EXPLORATION LOGS FOR MW-91 / 92 / 93 / 94, MW-526 / 527, AND MW-528 / 529.

   * THE "SILT, CLAY, AND SAND" DEPOSITS DESCRIBED IN EXPLORATION LOGS FOR MW-520 / 521 / 522, MW-857 / 858 / 859 / 860, AND MW-991 WERE DISCOUNTED AS THEY WERE INTERPRETED AS POST-GLACIAL COLLUVIUM / ALLUVIUM.

   * THE "SILTY-CLAY" DEPOSITS DESCRIBED IN BORING B18 WERE INTERPRETED TO CONSIST OF SILTY GLACIAL TILL BASED UPON A REVIEW OF NEARBY BORING AND MONITORING WELL LOGS.

3. THE CONTOURS ARE INTENDED TO SHOW TRENDS IN CLAY SURFACE TOPOGRAPHY CONSISTENT WITH AVAILABLE INFORMATION. IN AREAS WHERE EXPLORATIONS ARE ABSENT THE CONTOURS WERE ADJUSTED TO BE CONSISTENT WITH GEOLOGIC PROFILES SHOWN ON FIGURES 5 THROUGH 10.

4. THE CLAY LIMITS AND SURFACE ELEVATION CONTOURS IN THE AREA OF BUILDING 323 HAVE BEEN GENERATED ASSUMING THE BOTTOM OF EXCAVATION FOR BUILDING 323 IS AT AN ELEVATION OF 222 FEET AMSL.

5. SEE FIGURE 1 FOR ADDITIONAL NOTES AND LEGEND, AND APPENDIX A FOR LIMITATIONS.

LEGEND:

- MW-775 — LOCATION AND DESIGNATION OF GROUNDWATER MONITORING WELL
- B-87 — LOCATION AND DESIGNATION OF TEST BORING
- (215.3) — APPROXIMATE CLAY SURFACE ELEVATION
- 230 — CLAY SURFACE ELEVATION CONTOUR (INTERVAL VARIES)
- APPROXIMATE LIMITS OF MAPPING
- INFERRED LIMITS OF GLACIOLACUSTRINE CLAY (SEE NOTE 2)

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY

SCALE 200 0 200 400
1 INCH = 200 FEET

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
CLAY SURFACE ELEVATION CONTOUR PLAN

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 4 OF 10

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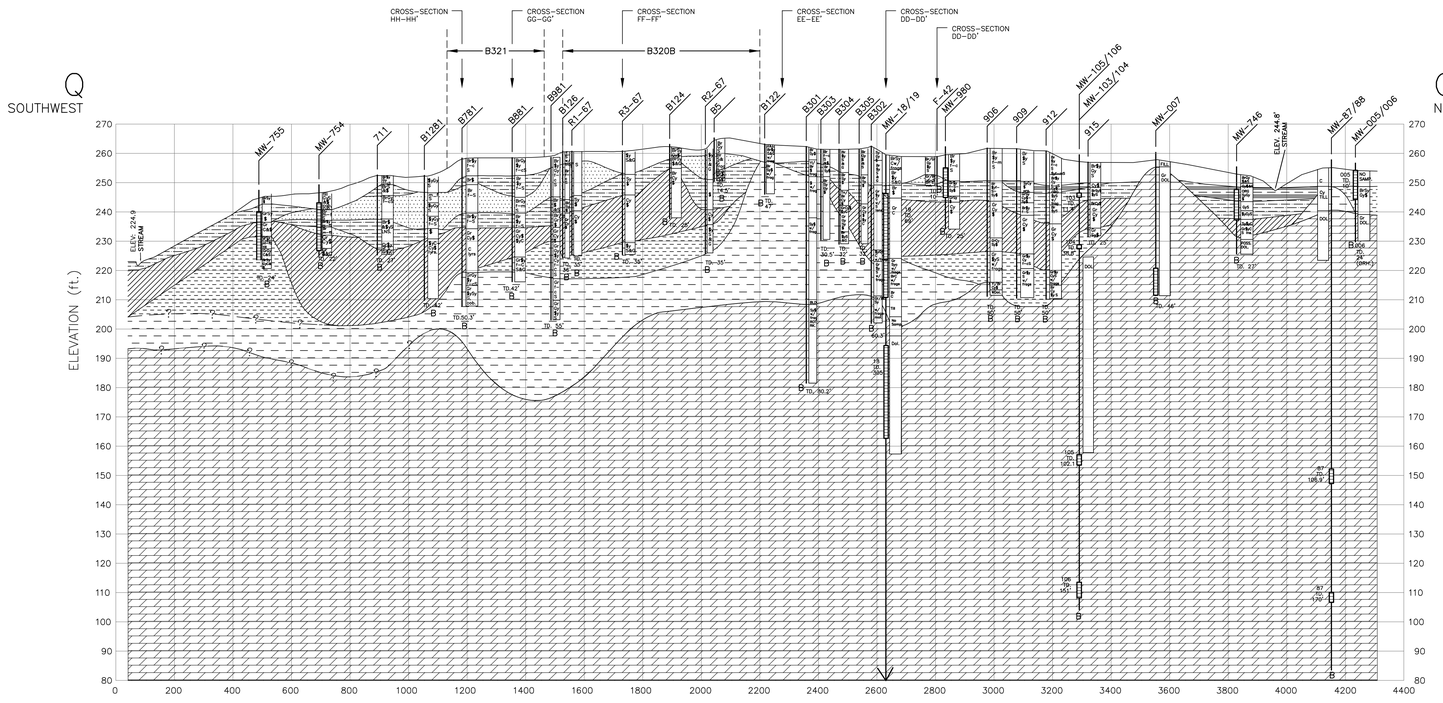

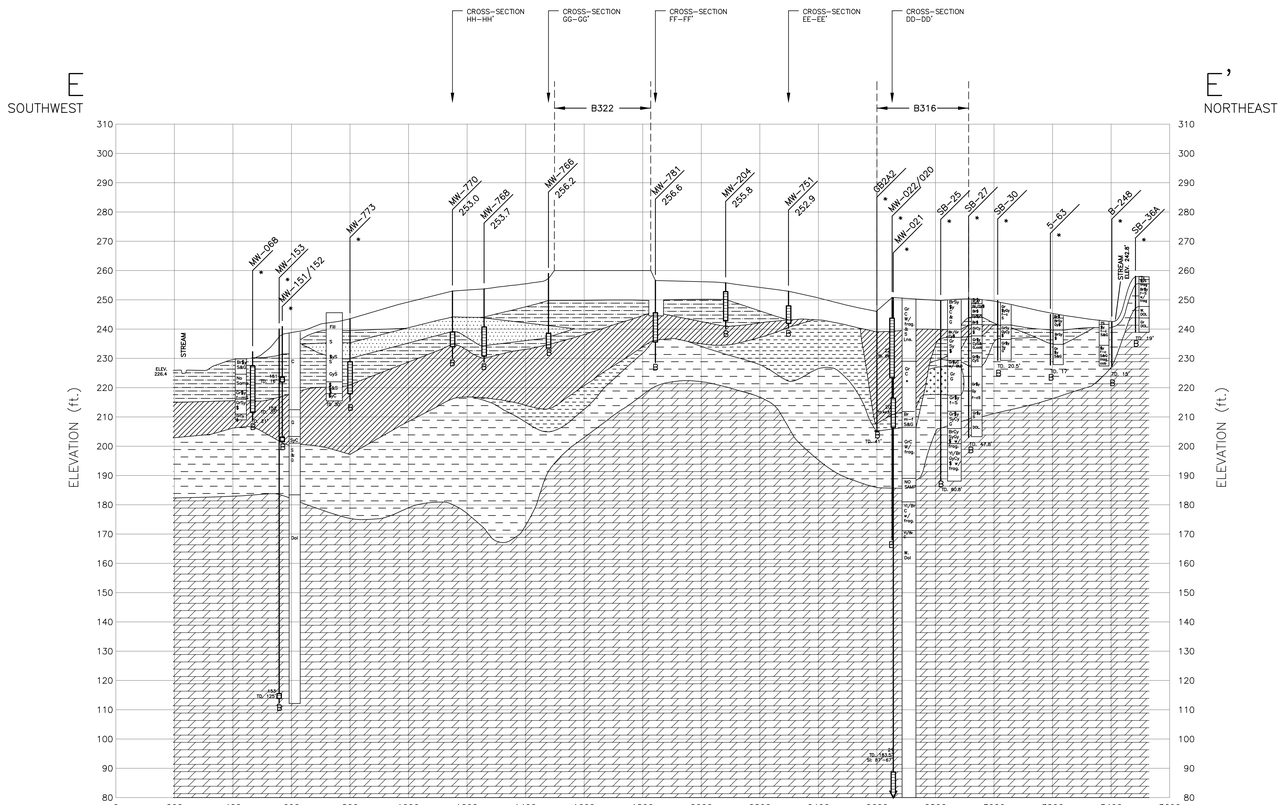

NOTES:

1. GEOLOGIC CROSS-SECTIONS Q-Q' AND E-E' WERE REVISED FROM EXISTING IBM "HIGH RESOLUTION" CROSS-SECTIONS WHICH WERE PROVIDED AS AUTOCAD FILES "EFQ-Q2.DWG" AND "EFE-E.DWG." AS PART OF REVISIONS TO CROSS-SECTION E-E' THE PORTION OF THE PROFILE BETWEEN MW-773 AND BORING GB2A2 WAS REPLACED WITH AN SHA INSERT. PLEASE REFER TO THE TITLE BLOCK FOR INFORMATION CONCERNING PREVIOUS REVISIONS TO THESE SECTIONS.

2. THE GEOLOGIC CROSS-SECTIONS WERE REVISED / UPDATED OR CREATED BASED UPON A REVIEW OF EXPLORATION LOGS AND A PHYSICAL WELL DATABASE FURNISHED BY IBM CORPORATION. THE BOUNDARIES BETWEEN DIFFERENT STRATA AS SHOWN WERE INTERPOLATED BETWEEN EXPLORATION LOCATIONS. THE APPROXIMATE STRATIGRAPHIC BOUNDARIES WERE CHECKED FOR CONSISTENCY AT CROSS-SECTION INTERSECTION POINTS AND ELEVATION CONTOURS ON SURFACE MAPS PROVIDED ON FIGURES 2 THROUGH 4. THE ELEVATIONS ARE IN FEET RELATIVE TO THE NATIONAL GEODETIC VERTICAL DATUM OF 1929.

3. PLEASE NOTE THAT OUR REVIEW OF IBM'S EXISTING SECTIONS WAS LIMITED TO "SPOT-CHECKING" GEOLOGIC DESCRIPTIONS AT CROSS-SECTION INTERSECTION POINTS AND AREAS MODIFIED BASED ON THE GEOLOGIC SURFACE MAPS.

4. THE CROSS-SECTIONS WERE REVISED OR CREATED WITH THE UNDERSTANDING THAT AS ADDITIONAL SUBSURFACE GEOLOGIC INFORMATION IS OBTAINED AND REVIEWED THEY WILL BE MODIFIED ACCORDINGLY.

5. ADDITIONAL NOTES BY IBM CORPORATION:
   - * CROSS-SECTION Q-Q' – "LOGS NOT USED FOR SECTION INTERPRETATION 103-106 (SOILS)."
   - * CROSS-SECTION E-E' – "LOGS FOR WELLS 123-126, AND 151-153 WERE NOT USED IN THE SECTION INTERPRETATION."

6. SEE FIGURE 1 FOR CROSS-SECTION LOCATIONS AND FOR ADDITIONAL NOTES AND LEGEND. SEE APPENDIX A OF THE REPORT FOR LIMITATIONS.

LEGEND

- FILL
- SILTY-SAND SANDY-SILT, SILT
- SILT WITH FRAGMENTS OR GRAVEL
- SAND, SAND-GRAVEL
- CLAY, CLAYEY-SILT/SILTY-CLAY
- ORGANIC LAYER/BURIED SOIL
- ABLATION TILL/ OUTWASH/ WEATHERED TILL
- LODGEMENT TILL
- BOULDER TILL
- GRAVEL (WITHIN LODGEMENT TILL)
- BEDROCK
- MW-751 252.9 — MONITORING WELL / TEST BORING DESIGNATION; GROUND SURFACE ELEVATION (FEET NGVD 1929)
- SCREENED INTERVAL
- B — BOTTOM OF BORING
- R — REFUSAL

HORIZONTAL SCALE
200 100 0 200 400
1 INCH = 200 FEET

VERTICAL SCALE
20 10 0 20 40
1 INCH = 20 FEET

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY
	3/90	Stratigraphy	PAD	7/90	DES
	11/90	Stratigraphy	JJW	11/91	DES
	1/93	Stratigraphy	SHA	–	–
		GEOLOGIC CROSS-SECTION E-E'			
	6/90	Stratigraphy	PAD	7/90	DES
	11/90	Last Update	–	–	–
	1/96	Stratigraphy/Layout	SHA	–	–
		GEOLOGIC CROSS-SECTION Q-Q'			

SCALE: AS DESIGNATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: HAV/MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
GEOLOGIC CROSS-SECTIONS Q-Q' AND E-E'

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 5 OF 10

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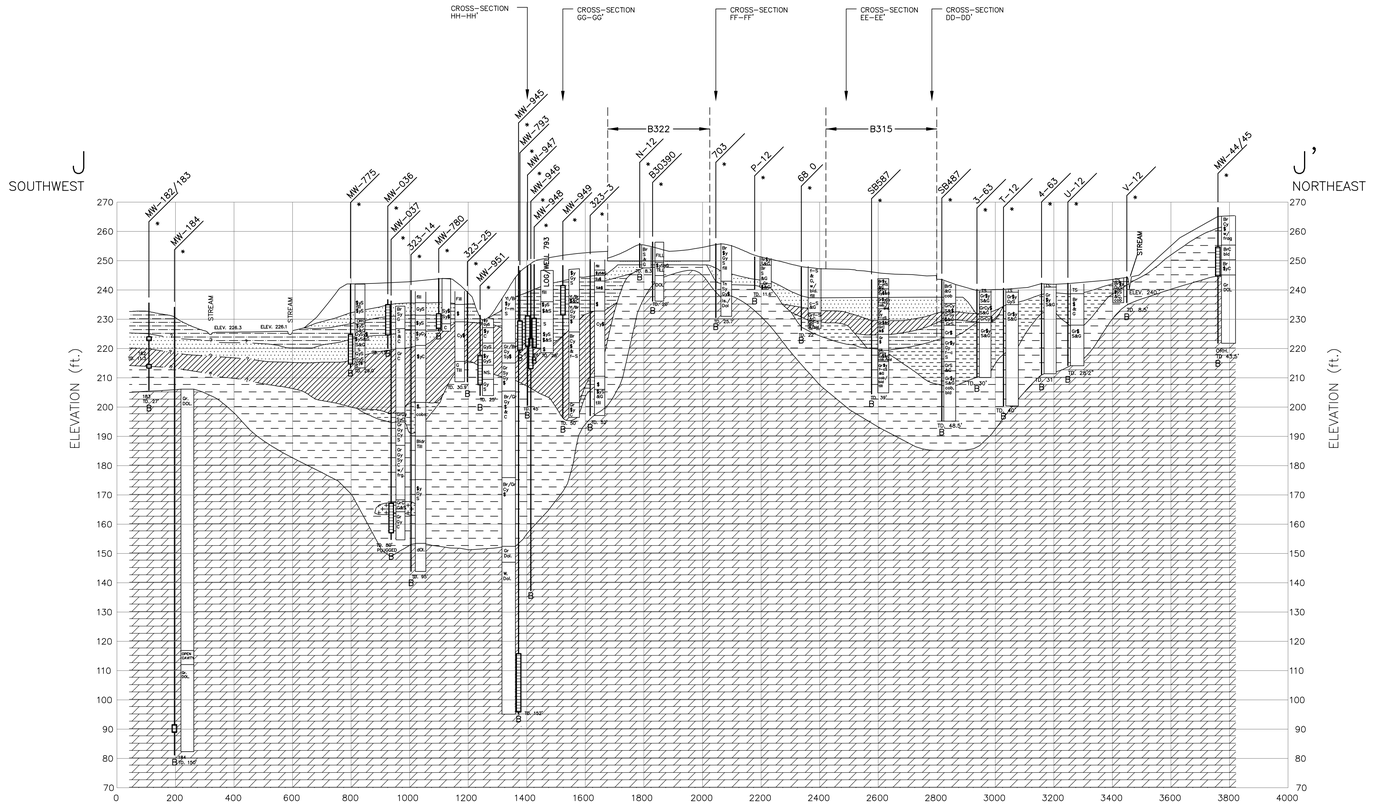
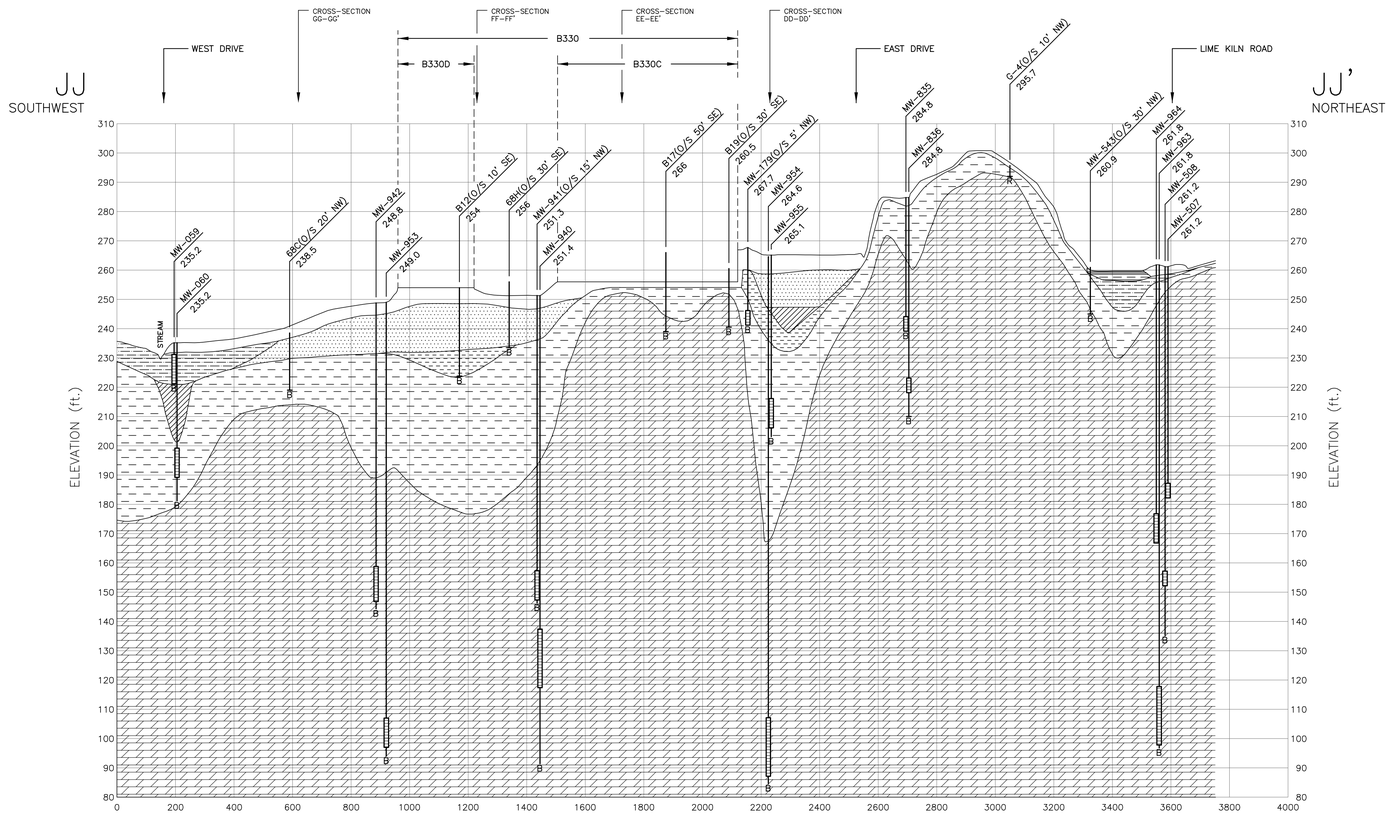
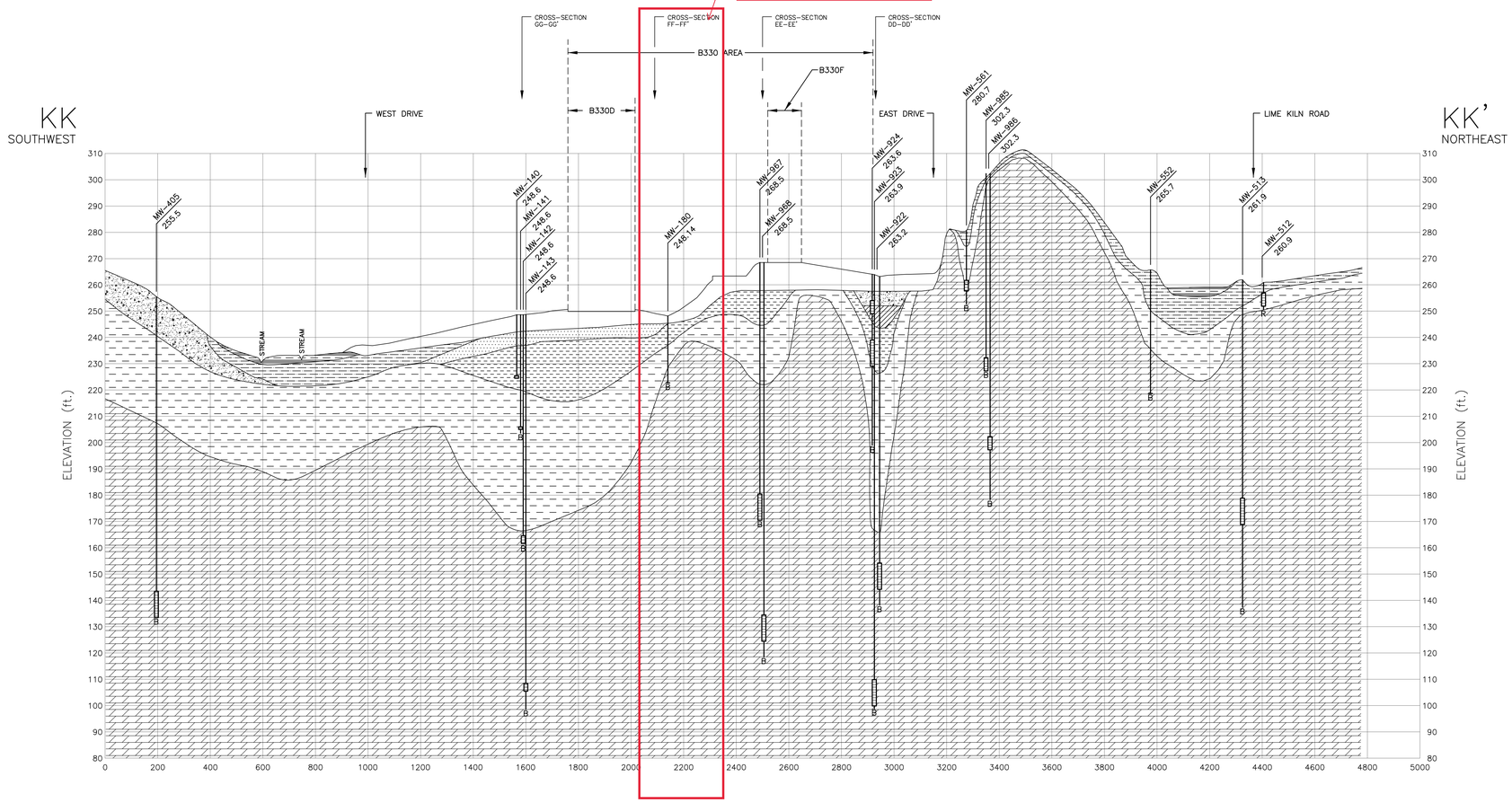
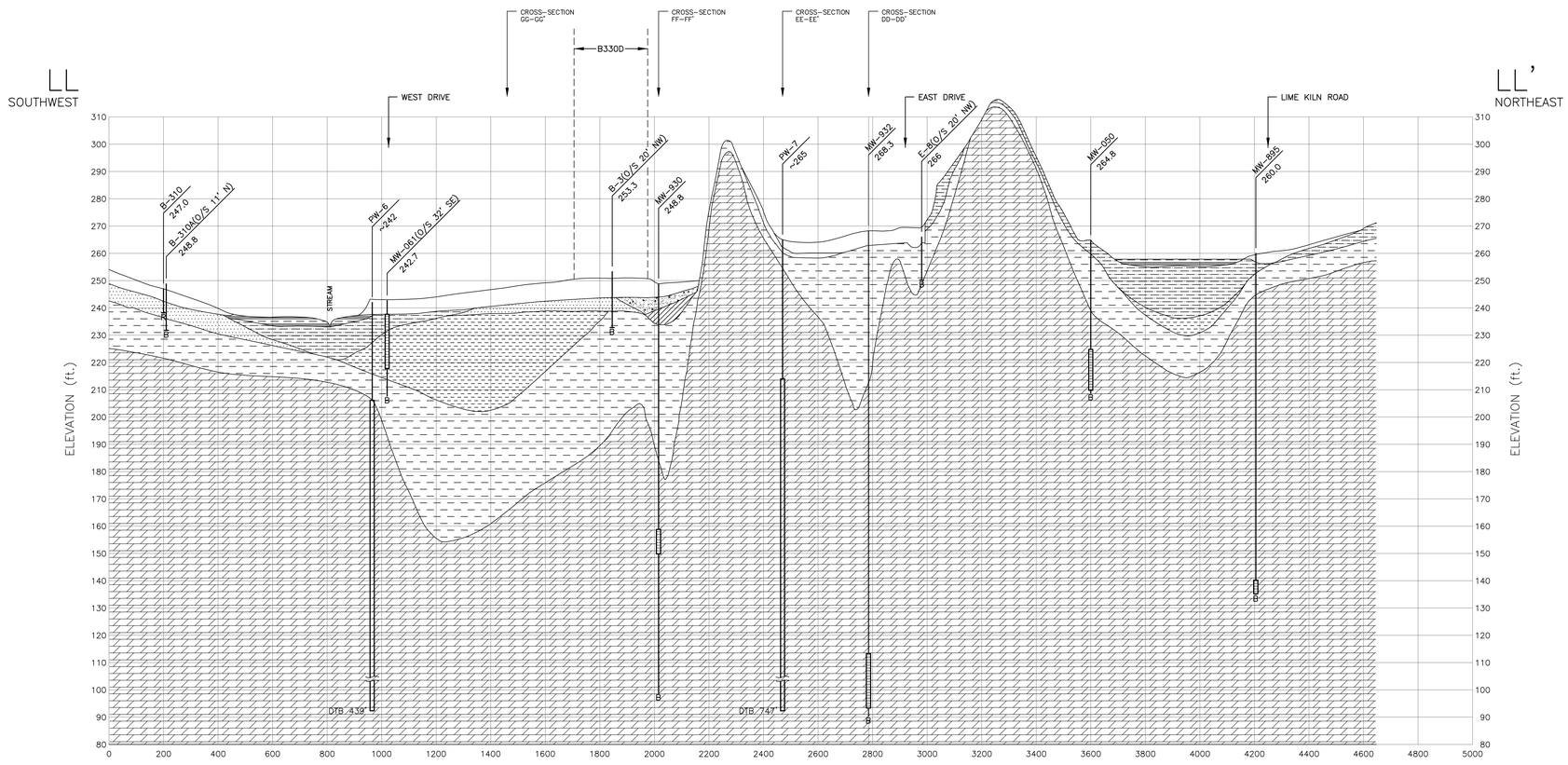



JJ
SOUTHWEST
JJ'
NORTHEAST
ELEVATION (ft.)
CROSS-SECTION GG-GG'
CROSS-SECTION FF-FF'
CROSS-SECTION EE-EE'
CROSS-SECTION DD-DD'
B330
B330D
B330C
WEST DRIVE
EAST DRIVE
LIME KILN ROAD
MW-059
235.2
MW-060
235.2
B6C10(S 20' NW)
238.5
MW-942
248.8
MW-953
248.0
B12(0/S 10' SE)
254
B6H(0/S 30' SE)
256
MW-941(0/S 15' NW)
251.3
MW-940
251.4
B17(0/S 50' SE)
266
B19(0/S 30' SE)
260.5
MW-179(0/S 5' NW)
257.7
MW-954
264.6
MW-955
265.1
MW-835
284.8
MW-836
284.8
G-4(0/S 10' NW)
295.7
MW-543(0/S 30' NW)
260.9
MW-964
261.8
MW-963
261.8
MW-508
261.2
MW-507
261.2
STREAM
B.R. 68.0'
B.R. 30.0'
B.R. 30.0'
B.R. 30

Approximate Area of B335 Assessment





NOTES:

1. GEOLOGIC CROSS-SECTIONS KK-KK' AND LL-LL' ARE NEW FIGURES CREATED BY SHA.

2. SEE FIGURES 1 AND 5 FOR ADDITIONAL NOTES AND LEGEND. SEE APPENDIX A OF THE REPORT FOR LIMITATIONS.

LEGEND

- FILL
- SILTY-SAND SANDY-SILT, SILT
- SILT WITH FRAGMENTS OR GRAVEL
- SAND, SAND-GRAVEL
- CLAY, CLAYEY-SILT/SILTY-CLAY
- ORGANIC LAYER/BURIED SOIL
- ABLATION TILL/ OUTWASH/ WEATHERED TILL
- LODGEMENT TILL
- BOULDER TILL
- GRAVEL (WITHIN LODGEMENT TILL)
- BEDROCK
- MW-898 260.0 — MONITORING WELL / TEST BORING DESIGNATION; GROUND SURFACE ELEVATION (FEET NGVD 1929)
- SCREENED INTERVAL
- B — BOTTOM OF BORING
- R — REFUSAL

HORIZONTAL SCALE
200 100 0 200 400
1 INCH = 200 FEET

VERTICAL SCALE
20 10 0 20 40
1 INCH = 20 FEET

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY
	1/96	GEOLOGIC CROSS-SECTION KK-KK'	SHA		
	1/96	Stratigraphy/Layout			
	1/96	GEOLOGIC CROSS-SECTION LL-LL'	SHA		
	1/96	Stratigraphy/Layout			

SCALE AS INDICATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: HAV/MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

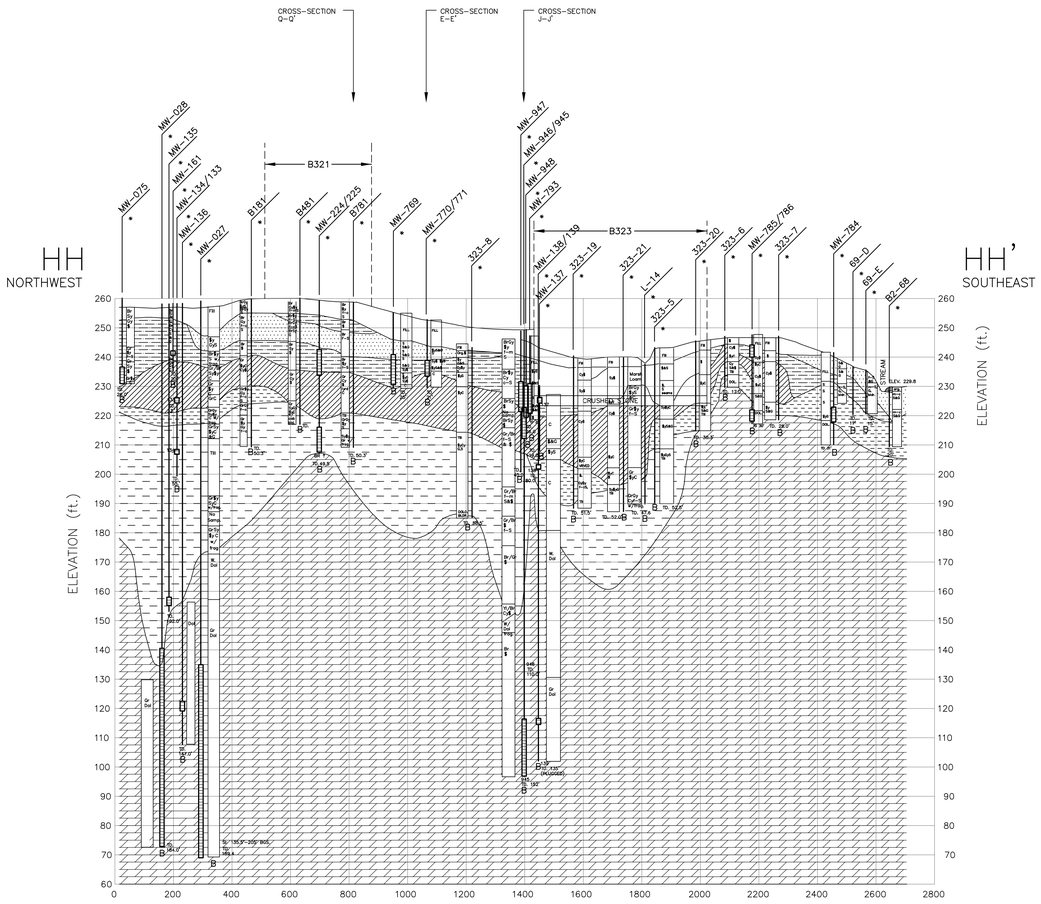
IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

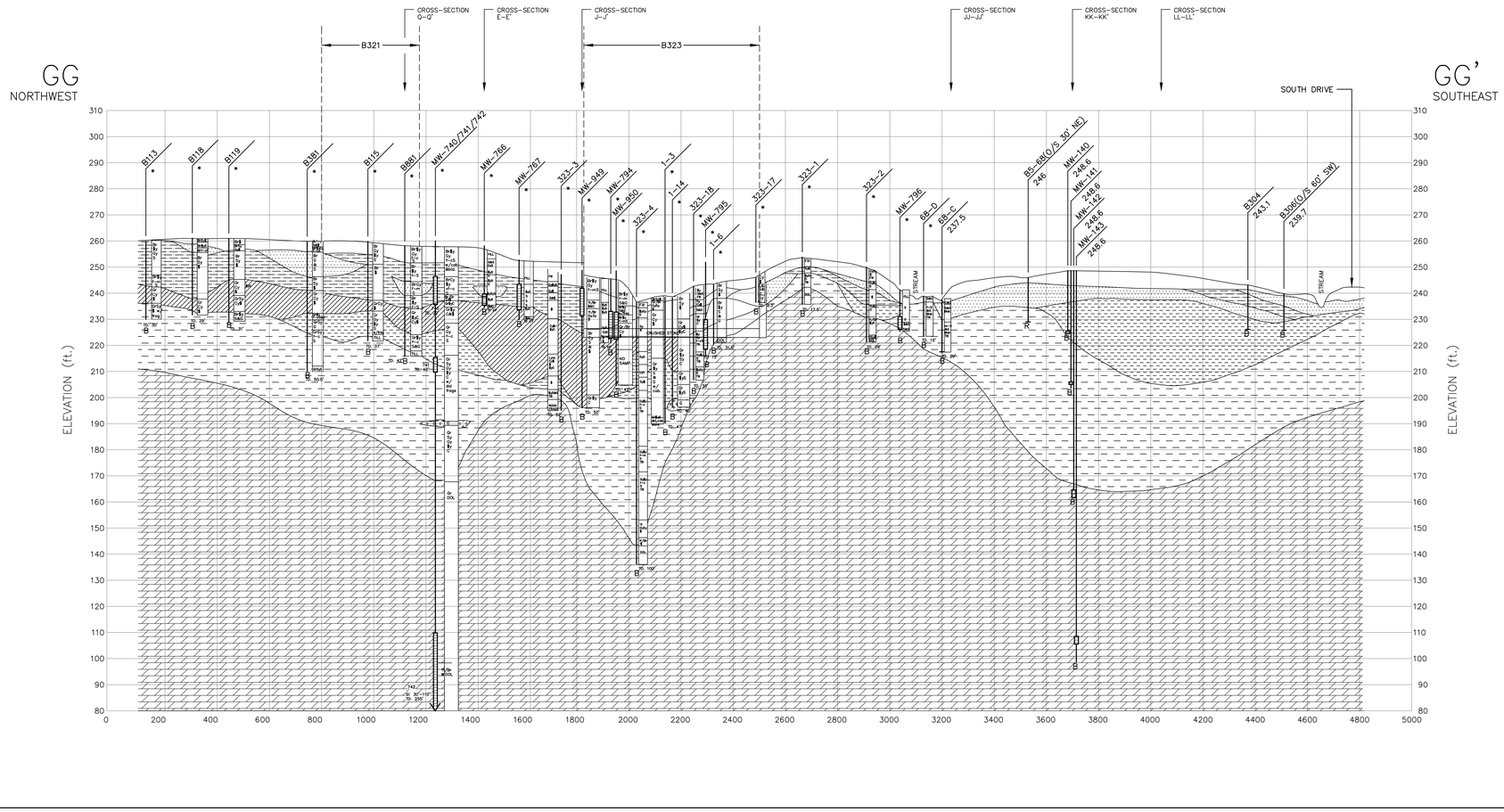
SUPPLEMENTAL GEOLOGIC MAPPING
GEOLOGIC CROSS-SECTIONS KK-KK' AND LL-LL'

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 7 OF 10

PLAN IMPORTANT\FIG7.DWG
XREF FILES: N:\PROJECT\1176.5\GEOL_M\MOD\FIG7-LL.DWG
N:\PROJECT\1176.5\GEOL_M\MOD\FIG7-KK.DWG
LAST SAVE: 01-19-96





NOTES:

1. GEOLOGIC CROSS-SECTIONS HH-HH' AND GG-GG' WERE REVISED FROM EXISTING IBM HIGH RESOLUTION CROSS-SECTIONS WHICH WERE PROVIDED AS AUTOCAD FILES "EFHH-HH.DWG" AND "EFGG-GG.DWG." CROSS-SECTION GG-GG' WAS EXTENDED BY SHA ABOUT 1,600 FEET SOUTHEAST OF BORING 68C.

2. ADDITIONAL NOTES BY IBM CORPORATION:

   * CROSS-SECTION HH-HH' - "LOGS NOT USED FOR SECTION REFERENCE DUE TO LITTLE OR NO OVERBURDEN DESCRIPTION (I.E. 133-166, 027)," "AIR ROTARY LOGS NOT USED: 945-949" AND "WELLS 137-139, DRILLED THROUGH B323 FLOOR."

   * CROSS-SECTION GG-GG' - NO NOTES.

3. SEE FIGURES 1 AND 5 FOR ADDITIONAL NOTES AND LEGEND. SEE APPENDIX A OF THE REPORT FOR LIMITATIONS.

LEGEND

- FILL
- SILTY-SAND SANDY-SILT, SILT
- SILT WITH FRAGMENTS OR GRAVEL
- SAND, SAND-GRAVEL
- CLAY, CLAYEY-SILT/SILTY-CLAY
- ORGANIC LAYER/BURIED SOIL
- ABLATION TILL/ OUTWASH/ WEATHERED TILL
- LODGEMENT TILL
- BOULDER TILL
- GRAVEL (WITHIN LODGEMENT TILL)
- BEDROCK

MW-141 / 248.6 — MONITORING WELL / TEST BORING DESIGNATION; GROUND SURFACE ELEVATION (FEET NGVD 1929)

SCREENED INTERVAL

B — BOTTOM OF BORING

R — REFUSAL

HORIZONTAL SCALE
200 100 0 200 400
1 INCH = 200 FEET

VERTICAL SCALE
20 10 0 20 40
1 INCH = 20 FEET

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY
		GEOLOGIC CROSS-SECTION HH-HH'	SHA	1/93	DES
1	1/96	Stratigraphy/Layout	JLB	11/91	DES
	1/93	Stratigraphy	JLW	1/93	DES
	11/90	Stratigraphy	JLB	11/91	DES
	6/90	Stratigraphy	PAD	7/90	DES
		GEOLOGIC CROSS-SECTION GG-GG'	SHA	-	-
	1/96	Stratigraphy/Layout	JLB	11/91	DES
	1/93	Well Info.	JLB	11/91	DES
	11/91	Stratigraphy	JLB	11/91	DES

SCALE AS INDICATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: HAV/MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

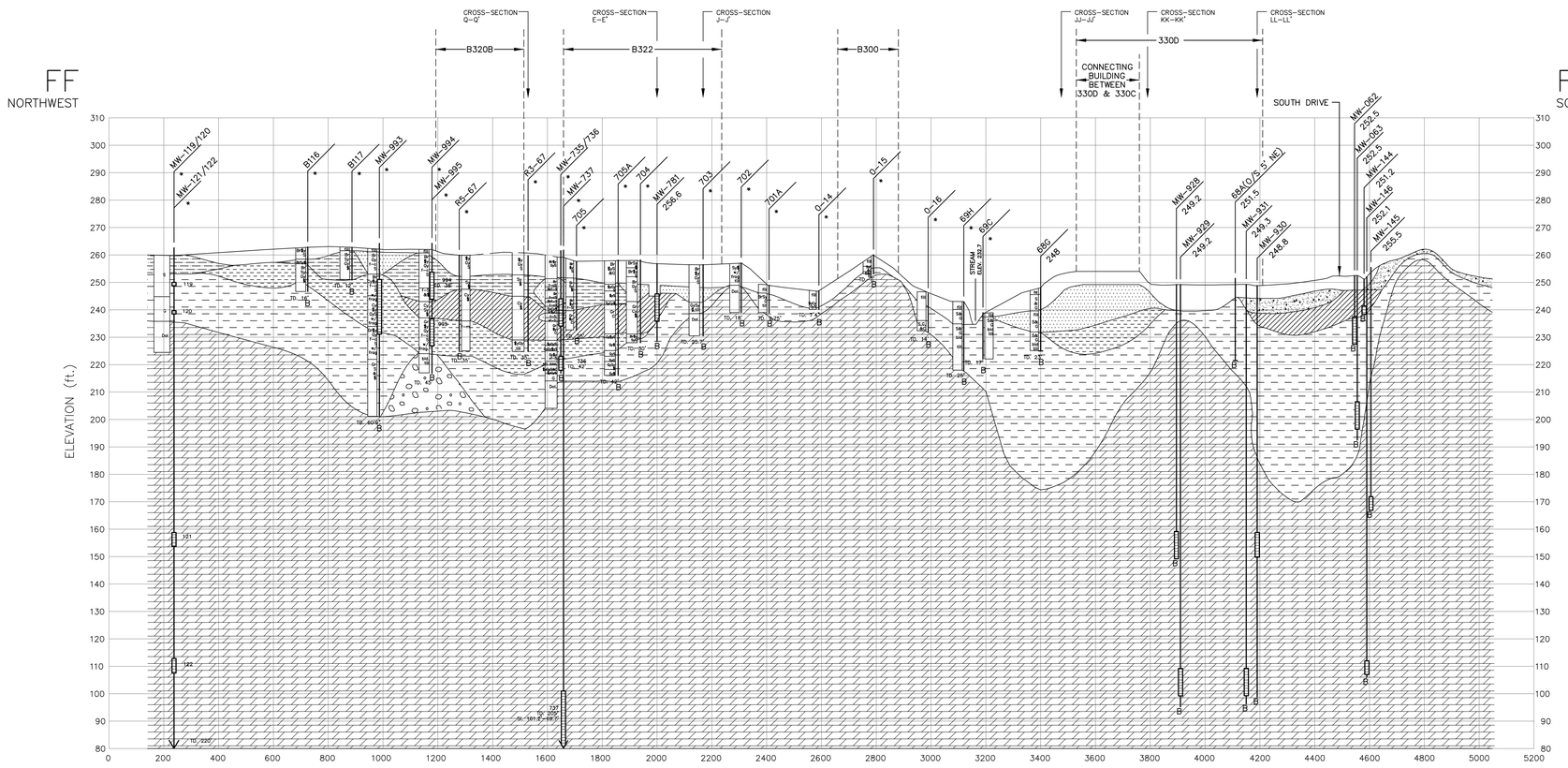
IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

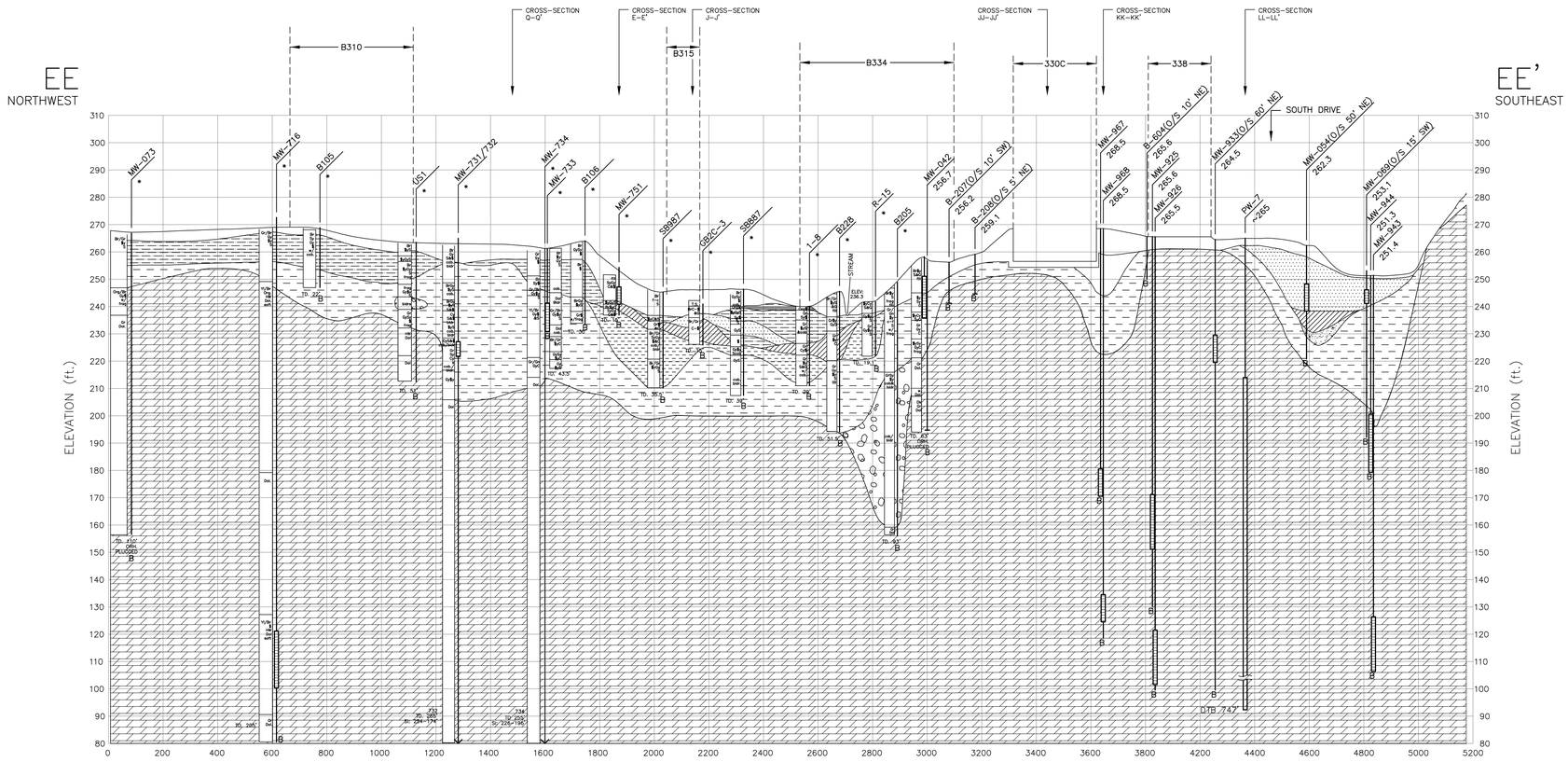
SUPPLEMENTAL GEOLOGIC MAPPING
GEOLOGIC CROSS-SECTIONS HH-HH' AND GG-GG'

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 8 OF 10

FILE: M:\MRP\FIGS\FIG8.DWG
PLOT: FEB 9, 96-09:15:57 (GROUND)
PLOT: FEB 9, 96-09:15:57 (GROUND) M:\MRP\FIGS\FIG8.DWG
LAST SAVE: 01:16:38





NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY
		GEOLOGIC CROSS-SECTION FF-FF'	SHA	-	-
1	1/96	Stratigraphy/Layout	-	-	-
	11/96	Slot Update	-	7/90	DES
	3/90	Stratigraphy	TLM		
		GEOLOGIC CROSS-SECTION EE-EE'	SHA	-	-
1	1/96	Stratigraphy/Layout	-	-	-
	11/96	Slot Update	-	7/90	DES
	3/90	Stratigraphy	TLM		

SCALE AS INDICATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: HAV/MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
GEOLOGIC CROSS-SECTIONS FF-FF' AND EE-EE'

PROJECT NUMBER: 1176.5

FIGURE NUMBER: 9 OF 10

FILE: M:\ARCHIVE\FIGURES\
PEN: FINE, M:\DRIVERS*.CTB
PLOT: 1:1, 0.0-10.0"
PLOTTED: 01-15-96

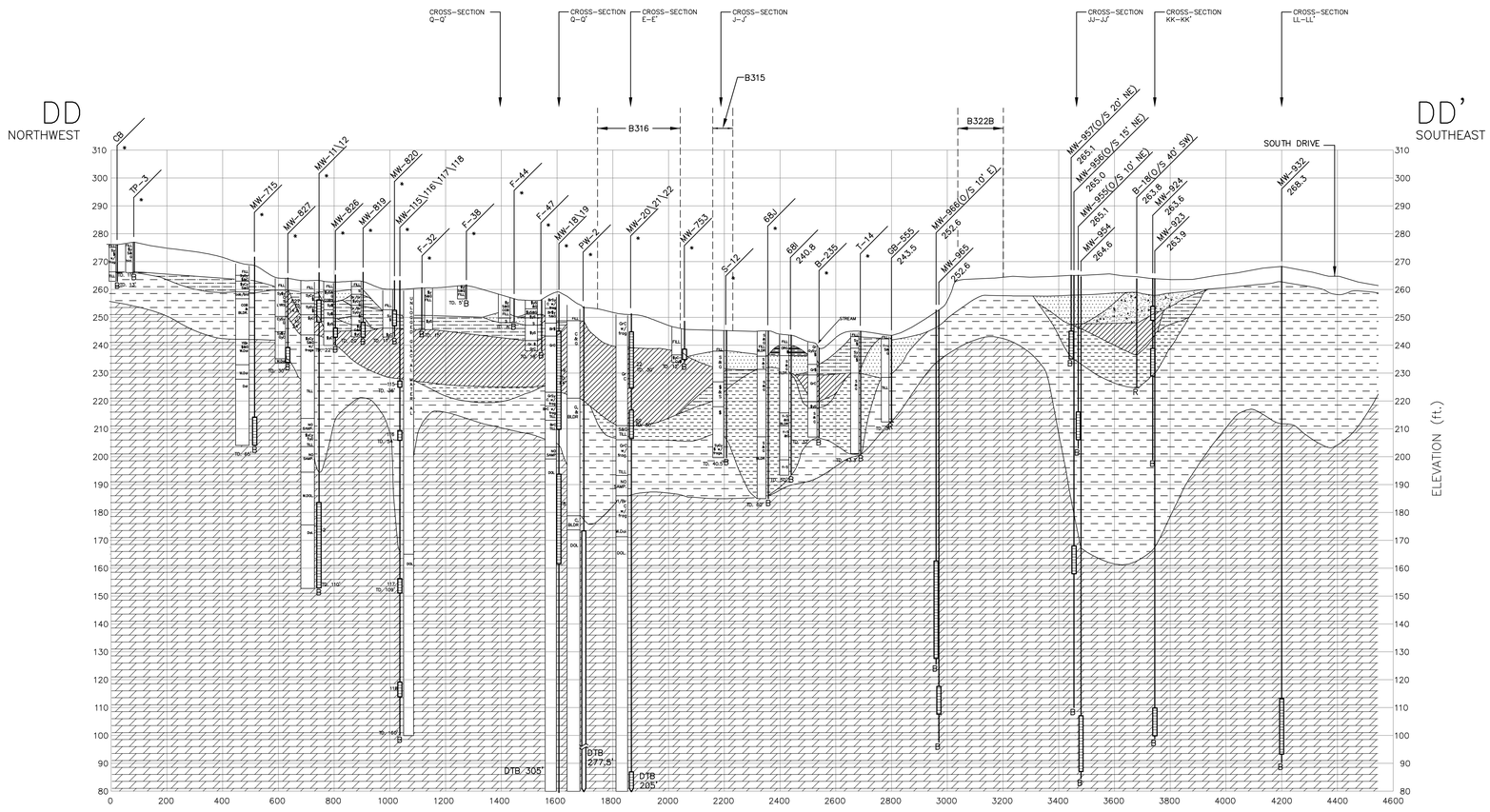
NOTES:

1. GEOLOGIC CROSS-SECTION DD-DD' WAS REVISED FROM AN EXISTING IBM HIGH RESOLUTION CROSS-SECTION WHICH WAS PROVIDED AS AUTOCAD FILE "EFDD-DD2.DWG." CROSS-SECTION DD-DD' WAS EXTENDED BY SHA ABOUT 1,750 FEET SOUTHEAST OF BORING GB-555.

2. ADDITIONAL NOTE BY IBM CORPORATION:

   * CROSS-SECTION DD-DD' -- "LOGS NOT USED FOR OVERBURDEN DESCRIPTION: 111-118, P02"

3. SEE FIGURES 1 AND 5 FOR ADDITIONAL NOTES AND LEGEND. SEE APPENDIX A OF THE REPORT FOR LIMITATIONS.



LEGEND

- FILL
- SILTY-SAND SANDY-SILT, SILT
- SILT WITH FRAGMENTS OR GRAVEL
- SAND, SAND-GRAVEL
- CLAY, CLAYEY-SILT/SILTY-CLAY
- ORGANIC LAYER/BURIED SOIL
- ABLATION TILL/ OUTWASH/ WEATHERED TILL
- LODGEMENT TILL
- BOULDER TILL
- GRAVEL (WITHIN LODGEMENT TILL)
- BEDROCK

MW-965 252.6 — MONITORING WELL / TEST BORING DESIGNATION; GROUND SURFACE ELEVATION (FEET NGVD 1929)

SCREENED INTERVAL

B — BOTTOM OF BORING

R — REFUSAL

HORIZONTAL SCALE
200 100 0 200 400
1 INCH = 200 FEET

VERTICAL SCALE
20 10 0 20 40
1 INCH = 20 FEET

NO.	DATE	DESCRIPTION/REVISION	BY	DATE APPROVED	BY
3/96	STRATIGRAPHY	TLM		7/96	DES
11/96	LAST UPDATE	-		-	
1/96	GEOLOGIC CROSS-SECTION DD-DD'	SHA		-	

SCALE: AS INDICATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: HAV/MRP
DESIGNED BY: RCW
CHECKED BY: RCW
REVIEWED BY: DBC
PROJECT MGR: MFD
PIC: CLH
DATE: JAN 96

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

SUPPLEMENTAL GEOLOGIC MAPPING
GEOLOGIC CROSS-SECTION DD-DD'

PROJECT NUMBER: 1176.5

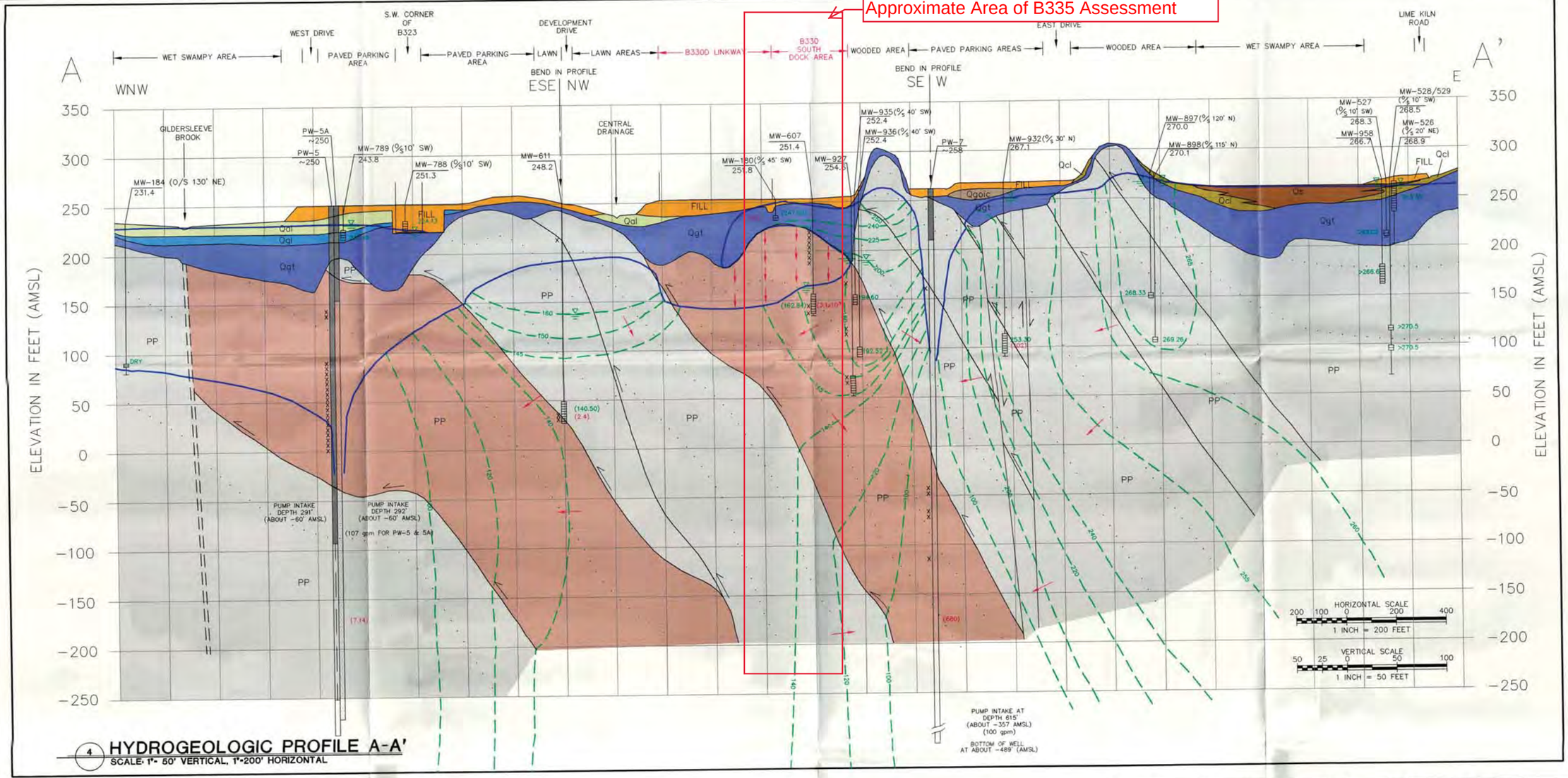
FIGURE NUMBER: 10 OF 10

FILE: M:\AUTOCAD\FISHKILL\EFDD-DD2.DWG
PLOTTED: 01/29/96 @ 07:19:36
PLOT SCALE: 1=1

APPENDIX A.2

FIGURE 4 OF AUGUST 1997 B330 GROUNDWATER RFI SUMMARY REPORT

Approximate Area of B335 Assessment



4 HYDROGEOLOGIC PROFILE A-A'

SCALE: 1"= 50' VERTICAL, 1"=200' HORIZONTAL

NOTES:

1) THE HYDROGEOLOGIC PROFILE DEPICTED ON THIS PLAN WAS DEVELOPED WITH THE INTENT OF ILLUSTRATING RELEVANT HYDROGEOLOGIC CONDITIONS AND CONCEPTS CONSISTENT WITH AVAILABLE DATA. PLEASE REFER TO THE INSET PROFILE LOCATION SKETCH MAP FOR THE LOCATION/ORIENTATION OF THIS PROFILE RELATIVE TO PROMINENT SITE FEATURES.

2) THE HYDROGEOLOGIC PROFILE WAS CREATED BASED UPON A REVIEW OF EXPLORATION LOGS AND TEST BORING/MONITORING WELL DATABASES FURNISHED BY IBM CORPORATION. THE BOUNDARIES BETWEEN DIFFERENT STRATA AS SHOWN WERE INTERPOLATED BETWEEN EXPLORATION LOCATIONS. THE APPROXIMATE STRATIGRAPHIC BOUNDARIES WERE CHECKED FOR CONSISTENCY WITH GEOLOGIC SURFACE MAPS AND OTHER HYDROGEOLOGIC PROFILES AT PROFILE INTERSECTION POINTS. THE ELEVATIONS ARE IN FEET RELATIVE TO THE NATIONAL GEODETIC VERTICAL DATUM OF 1929 – MEAN SEA LEVEL (MSL).

3) THE GROUNDWATER ELEVATIONS SHOWN ARE BASED UPON FIELD MEASUREMENTS RECORDED IN JANUARY 1997 BY IBM GROUNDWATER MONITORING PROGRAM PERSONNEL AND PROVIDED TO SHA IN A DATABASE FILE ENTITLED "FISH.DBF." FLUCTUATIONS IN THESE WATER LEVELS MAY OCCUR DUE TO VARIATIONS IN SEASONAL PRECIPITATION, GROUNDWATER WITHDRAWALS, TEMPERATURE, RUNOFF, AND OTHER FACTORS.

4) THE SCHEMATIC POTENTIOMETRIC SURFACE AND FLOW NET INFORMATION IS INTENDED TO DEPICT AN INTERPRETATION OF GENERAL TRENDS IN GROUNDWATER SURFACE ELEVATIONS AND FLOW CONDITIONS. AS THE PROFILE LINES ARE NOT NORMAL TO FLOW LINES, THIS INFORMATION IS NOT INTENDED FOR QUANTITATIVE PURPOSES.

5) THE APPROXIMATE JANUARY 1997 GROUNDWATER WITHDRAWAL RATES FOR THE BEDROCK PRODUCTION WELLS, AS SHOWN, ARE BASED UPON A MONTHLY PRODUCTION WELL PUMPING SUMMARY FOR THE PERIOD OF JANUARY 1997 THROUGH APRIL 1997.

6) THE GROUNDWATER QUALITY DATA SHOWN FOR "KEY" VOLATILE ORGANIC COMPOUNDS (VOCS) ARE BASED ON ROUTINE GROUNDWATER QUALITY MONITORING PERFORMED BY IBM DURING THE PERIOD OF JANUARY 1995 TO MARCH 1997. DATA WAS PROVIDED TO SHA IN DATABASE FILES ENTITLED "OR9596.DBF" AND "OR1Q97.DBF". UNLESS OTHERWISE NOTED, THE DATA FOR VOCS REPRESENT THE SUM OF MEDIAN VALUES RECORDED FOR KEY VOCS FOR THE B330 AOC WHICH INCLUDE PCE, TCE, C-1,2 DCE, VINYL CHLORIDE, FREON TF, AND FREON 123 (SEE FIGURES 10 AND 11).

7) SEE FIGURE 2 FOR ADDITIONAL NOTES AND LEGEND, AND APPENDIX A FOR LIMITATIONS.

LEGEND:

OVERBURDEN

- FILL — SOIL FILL
- Qs — SWAMP DEPOSITS
- Qcl — COLLUVIUM
- Qal — ALLUVIUM
- Qgt — GLACIOLACUSTRINE DEPOSITS
- Qgoic — UNDIFFERENTIATED GLACIOFLUVIAL OUTWASH AND ICE-CONTACT DEPOSITS
- Qgt — GLACIAL TILL

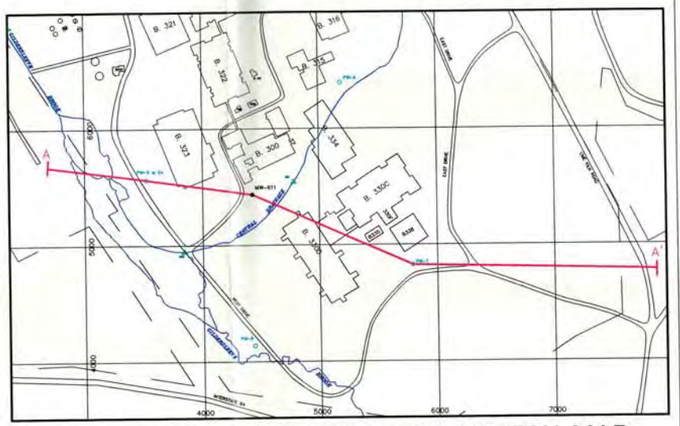
BEDROCK (PINE PLAINS FORMATION OF THE WAPPINGER GROUP)

- PP — BEDROCK – GENERALLY COMPETENT WITH RELATIVELY DISCRETE WEATHERED/FRACTURED ZONES
- PP — BEDROCK – CHARACTERIZED BY A GREATER PREVALENCE AND CONTINUITY OF SLIGHTLY TO HIGHLY WEATHERED/FRACTURED ZONES

- WATER LEVEL SURFACE (JANUARY 1997)
- 120 — EQUIPOTENTIAL CONTOUR
- INFERRED FLOW DIRECTION
- MW-607 / 251.4 — MONITORING WELL / TEST BORING DESIGNATION; GROUND SURFACE ELEVATION (IN FEET AMSL)
- DENOTES WEATHERED/ FRACTURED ZONES
- CASING (PROD. WELLS ONLY)
- SCREENED INTERVAL
- OPEN-HOLE (PROD. WELLS ONLY)
- GRAVEL-PACKED (PROD. WELLS ONLY)
- SLOTTED PIPE (PROD. WELLS ONLY)
- BOTTOM OF BORING
- GROUNDWATER ELEVATION MEASURED IN JANUARY 1997
- (225.73) — GROUNDWATER POTENTIOMETRIC LEVEL MEASURED IN JANUARY 1997
- 102 — SUM OF MEDIAN VALUES OF KEY VOCS (ug/l)
- (100 gpm) — JANUARY 1997 GROUNDWATER WITHDRAWAL RATE – PRODUCTION WELL

INFERRED BEDROCK STRUCTURAL ELEMENTS

- LOW ANGLE THRUST FAULT
- HIGH ANGLE NORMAL FAULT
- HIGH ANGLE STRIKE-SLIP FAULT
- APPROXIMATE ORIENTATION OF BEDDING



4 PROFILE LOCATION SKETCH MAP

SCALE: 1"=600'

NO.	DATE	DESCRIPTION	BY

SCALE: AS DESIGNATED

Sanborn, Head & Associates
Consulting Engineers & Scientists

DRAWN BY: FMC
DESIGNED BY: RCW
CHECKED BY: DBC
REVIEWED BY: PMS
PROJECT MGR: RCW
PIC: DBC
DATE: AUG 97

IBM EAST FISHKILL FACILITY
EAST FISHKILL, NEW YORK

BUILDING 330 GROUNDWATER RFI
SUMMARY REPORT
HYDROGEOLOGIC PROFILE A-A'

PROJECT NUMBER: 1386.5

FIGURE NUMBER: 4

FILE: G:\PROJECT\1386.5\GW\A330GW.DWG
PRINTED BY: (PROJECT.DGN) (MDL.DGN)
PLOT DATE: 08-26-97 PLOT SCALE: 1=1

APPENDIX A.3

VARIOUS FIGURES FROM 2016, 2017, AND 2018 ANNUAL CORRECTIVE ACTION STATUS REPORTS PREPARED BY GSC

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- GMP Soil Monitoring Well
- GMP Soil Monitoring Well (for groundwater elevations only)
- GMP Soil/Bedrock Monitoring Well
- GMP Soil/Bedrock Monitoring Well (for groundwater elevations only)
- Bedrock Extraction Well
- Inactive Bedrock Extraction Well
- Shallow Extraction Well
- Inactive Shallow Extraction Well
- OU – Operable Unit
- 220 – Contour of Constant Potentiometric Head (feet amsl)
- 228.08 – Groundwater Elevation (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- Extent of Clay Mapping
- Extent of Glaciolacustrine Clay
- Inferred Areas of No Saturated Soil (Approximate)

Area of Concern	Extraction Well(s)	Avg. October 2016 Flow Rate (GPM)
Area A	B384 Sump & GW-634	0.2
Area B	628	OFF LINE
Area D	032B	OFF LINE
B/322	612, 617	OFF LINE

AREA A (OU5)
AREA B (OU3)
AREA D (OU4)
B/322 (OU7)
B/133 (OU6)
SOUTHEAST QUADRANT (OU8)
NORTHEAST QUADRANT (OU9)
FORMER WEST COMPLEX
JOHN JAY HIGH SCHOOL
B384 Sump
PW-1
PW-2
PW-4
PW-5
PW-5A
PW-6
PW-7
PW-23
PW-25
B303
B304
B308
B309
B310
B316
B320A
B320B
B321
B322
B323
B323A
B324
B330C
B330D
B330E
B335
B336
B338
B339
B515
Route 52
Sheldon Hill Road
Lime Kiln Road
West Drive
North Drive
East Drive
Facility Drive
Administration Court
Service Road
Utility Road
Delivery Drive
Old Orchard Drive
South Drive
I-84
INTERSTATE 84

Plate 2c

Former IBM East Fishkill Facility

Elevation Contour Map
Soil Groundwater Table
October 5-7, 2016

DRAWN BY: MHM	DATE: 5/12/17	DRAWING NO. 95007-102-E2
CHECKED & APPROVED BY: CES		

GROUNDWATER SCIENCES CORPORATION

Scale 0 200' 400'

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- ▼ – GMP Bedrock Monitoring Well
- ▽ – GMP Bedrock Monitoring Well (for groundwater elevations only)
- ★ – Bedrock Extraction Well
- ☆ – Inactive Bedrock Extraction Well
- ☼ – Inactive Shallow Extraction Well
- OU – Operable Unit
- 200 – Contour of Constant Potentiometric Head (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- 168.13 – Groundwater Elevation (feet amsl) "<" symbol denotes a dry well. Elevation listed is the bottom elevation of the well

Note: Generally, the data used in contouring this plate is from the deepest of the bedrock wells in a well cluster and corresponds to a screened or open hole interval elevation between approximately 50 and 120 feet amsl (with the exception of PW-23). This elevation interval generally correlates with the principal water-bearing zones in the on-site deep bedrock production wells. (The principal water-bearing zone in PW-23 is at approximately 221 feet amsl, which is significantly higher than the elevation of the principal water-bearing zones in the other production wells.) Only groundwater elevation data from wells used in contouring is shown on this plate.

Extraction Well	Avg. January 2016 Flow Rate (GPM)
PW-1	99
PW-2	121
PW-4	101
PW-25	182

AREA A (OU5)
AREA B (OU3)
AREA D (OU4)
B/322 (OU7)
B/330 (OU8)
SOUTHEAST QUADRANT (OU6)
AREA C (LANDFILL) (OU6)
FORMER WEST COMPLEX
FORMER IBM RECREATION CENTER
JOHN JAY HIGH SCHOOL
INTERSTATE 84
ROUTE 52
LAKE KATRINE ROAD
SCHOOLHOUSE ROAD
NORTH DRIVE
WEST DRIVE
EAST DRIVE
FACILITY DRIVE
SERVICE ROAD
SOUTH DRIVE
GEORGE STREET
STEPHEN DRIVE
GEORGE STREET EXTENSION
CONFIDENCE COURT
B304, B303, B309, B308, B310, B320A, B320B, B321, B322, B323, B323A, B300, B324, B324A, B356, B317, B311e, B315, B330C, B330D, B338, B339, B339A, B339B, B339C, B339D, B339E, B339F, B339G, B339H, B339J, B339K, B339L, B339M, B339N, B339P, B339Q, B339R, B339S, B339T, B339U, B339V, B339W, B339X, B339Y, B339Z, B340, B341, B342, B343, B344, B345, B346, B347, B348, B349, B350, B351, B352, B353, B354, B355, B357, B358, B359, B360, B361, B362, B363, B364, B365, B366, B367, B368, B369, B370, B371, B372, B373, B374, B375, B376, B377, B378, B379, B380, B381, B382, B383, B384, B385, B386, B387, B388, B389, B390, B391, B392, B393, B394, B395, B396,

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- GMP Soil Monitoring Well
- GMP Soil Monitoring Well (for groundwater elevations only)
- GMP Soil/Bedrock Monitoring Well
- GMP Soil/Bedrock Monitoring Well (for groundwater elevations only)
- Bedrock Extraction Well
- Inactive Bedrock Extraction Well
- Shallow Extraction Well
- Inactive Shallow Extraction Well
- OU – Operable Unit
- 220 – Contour of Constant Potentiometric Head (feet amsl)
- 228.08 – Groundwater Elevation (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- Extent of Clay Mapping
- Extent of Glaciolacustrine Clay
- Inferred Areas of No Saturated Soil (Approximate)

Area of Concern	Extraction Well(s)	Avg. April 2017 Flow Rate (GPM)
Area A	B384 Sump & GW-634	0.8
Area B	628	OFF LINE
Area D	032B	OFF LINE
B/322	612, 617	OFF LINE

AREA A (OU5)
AREA B (OU3)
AREA D (OU4)
B/322 (OU7)
B330 (OU8)
SOUTHEAST QUADRANT (OU9)
LANDFILL (OU6)
PW-1
PW-2
PW-3
PW-4
PW-5
PW-6
PW-7
B384 Sump
FORMER WEST COMPLEX
FORMER IBM RECREATION CENTER
JOHN JAY HIGH SCHOOL
INTERSTATE 84
ROUTE 52
LAKE WALTON ROAD
SOUTH DRIVE
EAST DRIVE
WEST DRIVE
NORTH DRIVE
FACILITY DRIVE
UTILITY DRIVE
SERVICE ROAD
CENTER STREET
DEVELOPMENT DRIVE
PW-1 CONSTRUCTION COUNTY

Scale
0 200' 400'

Plate 3

Former IBM East Fishkill Facility

Elevation Contour Map
Soil Groundwater Table
April 24-26, 2017

DRAWN BY: MHM	DATE: 5/23/18	DRAWING NO.
CHECKED & APPROVED BY: CES		95007-102-G2

GROUNDWATER SCIENCES CORPORATION

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- ▼ – GMP Bedrock Monitoring Well
- ▽ – GMP Bedrock Monitoring Well (for groundwater elevations only)
- ★ – Bedrock Extraction Well
- ☆ – Inactive Bedrock Extraction Well
- ✱ – Soil Extraction Well
- OU – Operable Unit
- 200 – Contour of Constant Potentiometric Head (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- 164.54 – Groundwater Elevation (feet amsl) "<" symbol denotes a dry well. Elevation listed is the bottom elevation of the well

Note: Generally, the data used in contouring this plate is from the deepest of the bedrock wells in a well cluster and corresponds to a screened or open hole interval elevation between approximately 50 and 120 feet amsl (with the exception of PW-23). This elevation interval generally correlates with the principal water-bearing zones in the on-site deep bedrock production wells. (The principal water-bearing zone in PW-23 is at approximately 221 feet amsl, which is significantly higher than the elevation of the principal water-bearing zones in the other production wells.) Only groundwater elevation data from wells used in contouring is shown on this plate.

Extraction Well	Avg. April 2017 Flow Rate (GPM)
PW-1	70
PW-2	116
PW-4*	59
PW-25	226

*Based on the approved Production Well Optimization Report, flow rebalancing at the production wells commenced on April 17, 2017. PW-4 off-line beginning April 17, 2017. Average April 2017 flow rate prior to shutdown was 109 GPM.

Plate 4

Former IBM East Fishkill Facility

Elevation Contour Map
Deep Bedrock Potentiometric Surface
April 24-26, 2017

DRAWN BY: MHM	DATE: 5/24/18	DRAWING NO. 95007-064-S2
CHECKED & APPROVED BY: CES		

GROUNDWATER SCIENCES CORPORATION

Scale 0 200' 400'

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- GMP Soil Monitoring Well
- GMP Soil Monitoring Well (for groundwater elevations only)
- GMP Soil/Bedrock Monitoring Well
- GMP Soil/Bedrock Monitoring Well (for groundwater elevations only)
- Bedrock Extraction Well
- Inactive Bedrock Extraction Well
- Shallow Extraction Well
- Inactive Shallow Extraction Well
- OU – Operable Unit
- 220 – Contour of Constant Potentiometric Head (feet amsl)
- 228.08 – Groundwater Elevation (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- Extent of Clay Mapping
- Extent of Glaciolacustrine Clay
- Inferred Areas of No Saturated Soil (Approximate)

Area of Concern	Extraction Well(s)	Avg. April 2018 Flow Rate (GPM)
Area A	B384 Sump	OFFLINE

AREA A (OU5)
AREA B (OU3)
AREA D (OU4)
B/322 (OU7)
B/330 (OU8)
AREA C (LANDFILL) (OU6)
SOUTHEAST QUADRANT (OU9)
PW-1
PW-2
PW-25
B384 Sump
B309 sink
B308 sink
JOHN JAY HIGH SCHOOL
FORMER WEST COMPLEX
FORMER IBM RECREATION CENTER
INTERSTATE 84
ROUTE 52
SOUTH DRIVE
WEST DRIVE
EAST DRIVE
FACILITY DRIVE
UTILITY DRIVE
NORTH DRIVE
SERVICE ROAD
DOCUMENTATION DRIVE
OLD ROUNDTOP DRIVE
STORE STREET
LAKE HILL ROAD
SCHOOLHOUSE ROAD

Scale
0 200' 400'

Plate 3

Former IBM East Fishkill Facility

Elevation Contour Map
Soil Groundwater Table
April 2-6, 2018

DRAWN BY: MHM | DATE: 5/15/19 | DRAWING NO. 95007-102-K2
CHECKED & APPROVED BY: CES

GROUNDWATER SCIENCES CORPORATION

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- GMP Bedrock Monitoring Well
- GMP Bedrock Monitoring Well (for groundwater elevations only)
- Bedrock Extraction Well
- Inactive Bedrock Extraction Well
- Soil Extraction Well
- OU – Operable Unit
- 200 – Contour of Constant Potentiometric Head (feet amsl)
- Generalized Direction of Groundwater Flow
- Groundwater Divide
- 164.54 – Groundwater Elevation (feet amsl) "<" symbol denotes a dry well. Elevation listed is the bottom elevation of the well

Note: Generally, the data used in contouring this plate is from the deepest of the bedrock wells in a well cluster and corresponds to a screened or open hole interval elevation between approximately 50 and 120 feet amsl. This elevation interval generally correlates with the principal water-bearing zones in the on-site deep bedrock production wells. Only groundwater elevation data from wells used in contouring is shown on this plate.

Extraction Well	Avg. April 2018 Flow Rate (GPM)
PW-1	70
PW-2	118
PW-4	OFFLINE
PW-25	230

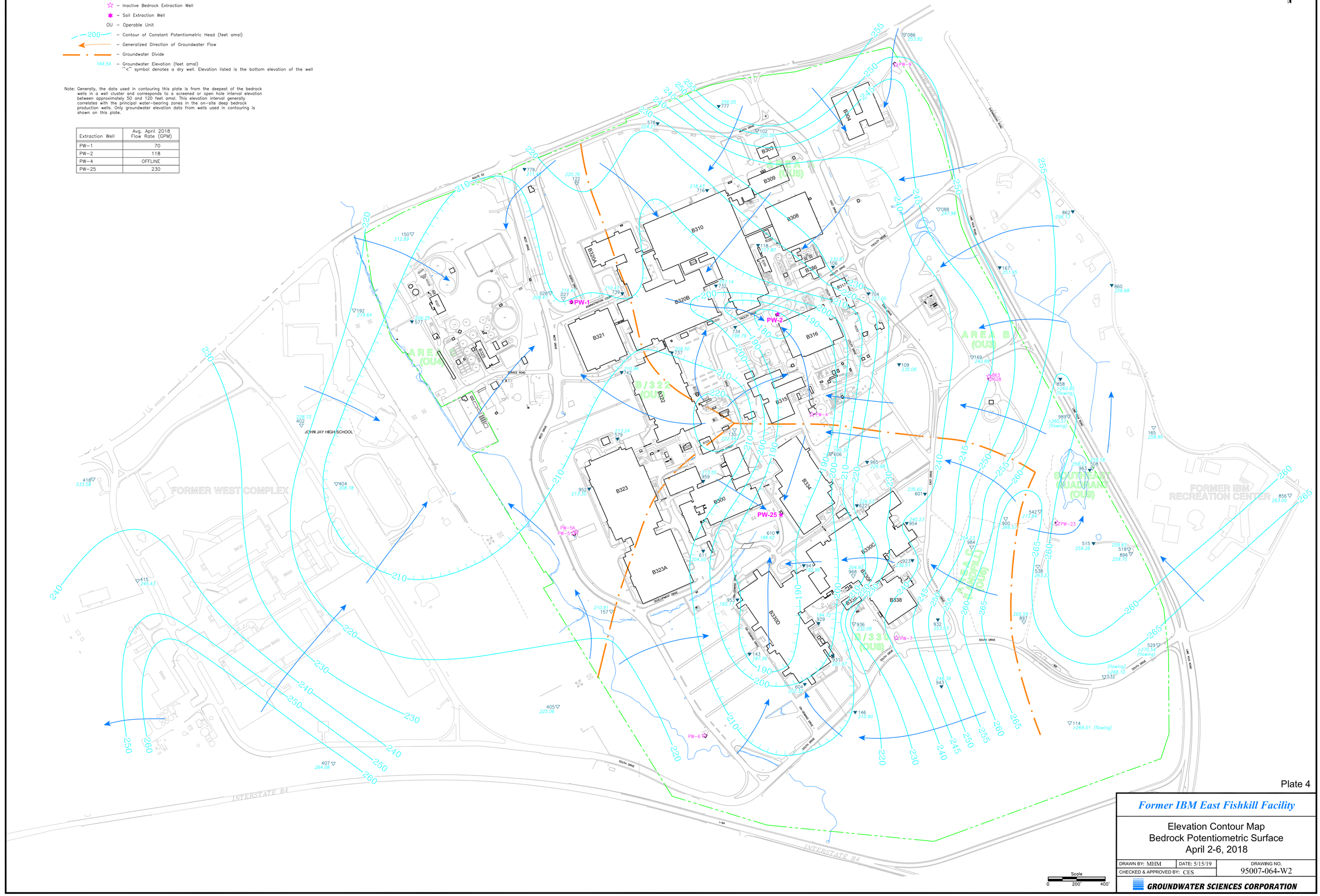


Plate 4

Former IBM East Fishkill Facility

Elevation Contour Map
Bedrock Potentiometric Surface
April 2-6, 2018

DRAWN BY: MHM	DATE: 5/15/19	DRAWING NO. 95007-064-W2
CHECKED & APPROVED BY: CES		

GROUNDWATER SCIENCES CORPORATION

Scale 0 200' 400'

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- ▲ - GMP Soil Monitoring Well
- ● - GMP Soil/Bedrock Monitoring Well
- ★ - Bedrock Extraction Well
- ☆ - Inactive Bedrock Extraction Well
- ✸ - Shallow Extraction Well
- ☼ - Inactive Shallow Extraction Well
- OU - Operable Unit
- PCE - Tetrachloroethene
- 5 - PCE Concentration Contour (ug/l)
- *49* - PCE Concentration (ug/l; April 9-May 4, 2018)
- *ND@"X"* - Not Detected at Laboratory Detection Limit "X"
- **Extent of Clay Mapping**
- **Extent of Glaciolacustrine Clay** (East Complex only) (Approximate)
- Inferred Areas of No Saturated Soil

AREA A (OU 5)
AREA B (OU 3)
AREA D (OU 4)
B/322 (OU 7)
B/330 (OU 8)
AREA C LANDFILL (OU 6)
SOUTHEAST QUADRANT (OU 9)
FORMER WEST COMPLEX
FORMER IBM RECREATION CENTER
JOHN JAY HIGH SCHOOL
INTERSTATE 84
ROUTE 52
NORTH DRIVE
WEST DRIVE
SOUTH DRIVE
EAST DRIVE
FACILITY DRIVE
UTILITY DRIVE
SERVICE ROAD
ADMINISTRATION COURT
DEVELOPMENT DRIVE
OLD ROUNDHILL DRIVE
STATE STREET
LAKE MILL ROAD
SCHOONMAKER DRIVE
B303, B304, B308, B309, B310, B316, B320A, B320B, B321, B322, B323, B323A, B324, B325, B330C, B330F, B335, B336, B338, B339, B375, B384 Sump, B300, B650, B660, B540, B560, Former B560
PW-1, PW-2, PW-3, PW-4, PW-5, PW-5A, PW-6, PW-7, PW-23, PW-25

Scale
0 200' 400'

Plate 5

Former IBM East Fishkill Facility

Isoconcentration Contour Map
Soil PCE
April-May 2018

DRAWN BY: MHM	DATE: 5/15/19	DRAWING NO.
CHECKED & APPROVED BY: CES		95007_PCE_2Q2018_soil_1

GROUNDWATER SCIENCES CORPORATION

LEGEND

- Former IBM East Fishkill Facility Property Line
- Former Structure
- ▼ - GMP Bedrock Monitoring Well
- ● - GMP Soil/Bedrock Monitoring Well
- ★ - Bedrock Extraction Well
- ☆ - Inactive Bedrock Extraction Well
- ☼ - Inactive Shallow Bedrock Extraction Well
- OU - Operable Unit
- PCE - Tetrachloroethene
- 5 - PCE Concentration Contour (ug/l)
- 47 - PCE Concentration (ug/l; April 6-May 4, 2018 unless otherwise noted)
- ND@"X" - Not Detected at Laboratory Detection Limit "X"

AREA A (OU5)
AREA B (OU3)
AREA C (LANDFILL) (OU6)
AREA D (OU4)
B/322 (OU7)
B/330 (OU8)
SOUTHEAST QUADRANT (OU9)
FORMER WEST COMPLEX
FORMER IBM RECREATION CENTER
JOHN JAY HIGH SCHOOL
INTERSTATE 84
ROUTE 52
SCHOONMAKER ROAD
LAKE KATRINE ROAD
NORTH DRIVE
WEST DRIVE
EAST DRIVE
FACULTY DRIVE
SERVICE ROAD
CENTER STREET
DOCKSHOVER DRIVE
SOUTH DRIVE
ADMINISTRATION DRIVE
B303, B304, B309, B310, B320A, B320B, B321, B322, B323, B3234A, B300, B330C, B330D, B334, B338, B315, B316, B317, B308, B305, B306, B307, B329, B331, B332, B333, B335, B336, B337, B339, B340, B650, Former B650

Scale: 0 200' 400'

Plate 10

Former IBM East Fishkill Facility

Isoconcentration Contour Map
Bedrock PCE
April-May 2018

DRAWN BY: MHM	DATE: 5/15/19	DRAWING NO.
CHECKED & APPROVED BY: CES		95007_PCE_2Q2018_BR_1

GROUNDWATER SCIENCES CORPORATION

APPENDIX B

PROCEDURES AND PROTOCOLS

APPENDIX B.1

FIELD TOOLING DATA SHEETS

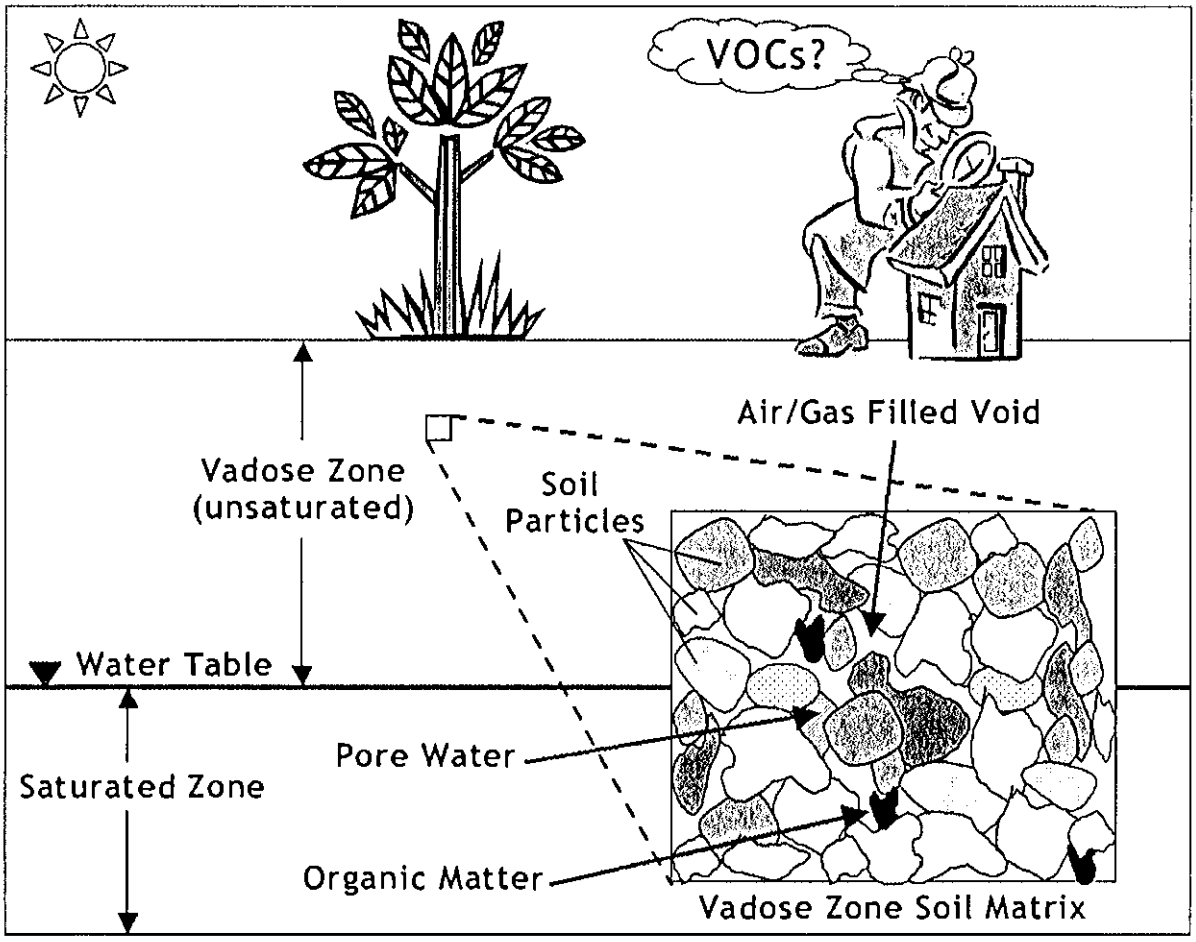
PRT Systems

geoprobe.com/**prt**

1.5 in. Probe Rod (60 in.) MN: ***201682*** PN: 11121	**1.5 in. Probe Rod (48 in.)** MN: ***202248*** PN: 13359	**1.5 in. Probe Rod (36 in.)** MN: ***207261*** PN: 35325	**1.5 in. Probe Rod (24 in.)** MN: ***202715*** PN: 15472	**1.5 in. Probe Rod (12 in.)** MN: ***202714*** PN: 15471	**1.5 in. Probe Rod (6 in.)** MN: ***205222*** PN: 27542
1.5 in. Probe Rod (1 m) MN: ***203193*** PN: 17899	**1.25 in. Probe Rod (60 in.)** MN: ***213773*** PN: AT1260	**1.25 in. Probe Rod (48 in.)** MN: ***213769*** PN: AT1248	**1.25 in. Probe Rod (36 in.)** MN: ***213762*** PN: AT1236	**1.25 in. Probe Rod (24 in.)** MN: ***213758*** PN: AT1224	**1.25 in. Probe Rod (12 in.)** MN: ***213753*** PN: AT1212
1.25 in. Probe Rod (6 in.) MN: ***213752*** PN: AT1206	**1.25 in. Probe Rod (1 m.)** MN: ***213763*** PN: AT1239	**1.0 in. Expendable Steel Point, 1.1 in. OD** MN: ***213778*** PN: AT14	**1.0 in. Expendable Steel Point, 1.1 in. OD (100 pack)** MN: ***213781*** PN: AT14K	**1.5 EXP PT HOLDER PRT THD** MN: ***204179*** PN: 22805	**1.25 EXP PT HOLDER PRT THD** MN: ***214202*** PN: PR1215
PRT EXP POINT POPPER MN: ***214207*** PN: PR15K	**PRT ADAPTER FOR 3/16 in. ID TUBING** MN: ***214208*** PN: PR17S	**PRT ADAPTER FOR 1/4 in. ID TUBING** MN: ***214211*** PN: PR25S	**PRT ADAPTER FOR 5/16 in. ID TUBING** MN: ***214212*** PN: PR30S	**PRT ADAPTER FOR 1/8 in. ID TUBING** MN: ***214203*** PN: PR12S	**Tube PE 1/4 in. OD x 3/16 in. ID** MN: ***601062*** PN: TB17L
TUBE PE 1/2 OD X 3/8 ID X 500 ft. MN: ***601067*** PN: TB37L	**TUBE PE 3/8 OD X 1/4 ID X 500 ft.** MN: ***601063*** PN: TB25L	**TUBE TEFLON 1/4 OD X 3/16 ID X 50 ft.** MN: ***214253*** PN: TB17T	**TUBE TEFLON 5/16 OD x 1/4 ID X 50 ft.** MN: ***601064*** PN: TB25T	**TUBE TEFLON 1/4 OD X 1/8 ID X 50 ft.** MN: ***601057*** PN: TB12T	**TUBE TEFLON 3/8 OD X 5/16 ID X 50 ft.** MN: ***601065*** PN: TB30T
TUBE NYLON 3/8 OD X 1/4 ID X 100 ft. MN: ***600144*** PN: 11633	**VAC VOL SYSTEM PORTABLE** MN: ***213739*** PN: AT1000	**O-RING PRT ADAPTERS (25)** MN: ***214210*** PN: PR25R	**O-RING 1.0 EXP PT (25)** MN: ***213782*** PN: AT14R	**GAS SAMPLING CAP 1.25 in. BOX** MN: ***213775*** PN: AT1266	**O-RING GAS SAMPLING CAP 1.25 in. (25)** MN: ***213776*** PN: AT1266R

Direct Push Installation of Devices for Active Soil Gas Sampling & Monitoring

Technical Bulletin No. MK3098
Prepared: May, 2006



Geoprobe Systems®

Section 1.0
Purpose

This document details the collection of representative soil gas samples with appropriate direct push (DP) methods to meet a range of data quality objectives, site-specific conditions, and regulatory requirements. New requirements for vapor intrusion investigations (U.S. EPA 2002, Cal. DTSC 2003, NYDOH 2005, etc.) establish higher sample data quality objectives to meet the needs for the human health risk assessment for this pathway. Downhole tools and sample collection methods needed to obtain the higher quality soil gas samples are reviewed.

Section 2.0
Background

What is soil gas? Where is the vadose zone? The ASTM Standard Guide D5314 for soil gas monitoring gives us the following definitions:

soil gas = vadose zone atmosphere
vadose zone = the hydrogeological region extending from the soil surface to the top of the principal water table

Soil gas is simply the gas phase (air) that exists in the open spaces between the soil particles in the unsaturated portion of the subsurface (Figure 1). Soil gas is normally comprised primarily of nitrogen and oxygen like the aboveground air it is in equilibrium with. However, when volatile organic compounds (VOCs) have been released in the subsurface due to spills or leaks, they will begin to evaporate from the fluid phase and become a part of the soil gas. Over time the VOCs can migrate through the soil and/or groundwater and present a potential hazard to human health or the environment. Intrusion of volatile contaminant vapors into an occupied building (home, office, school, etc.) can lead to exposure of the occupants to the potentially harmful contaminants (US EPA 2002).

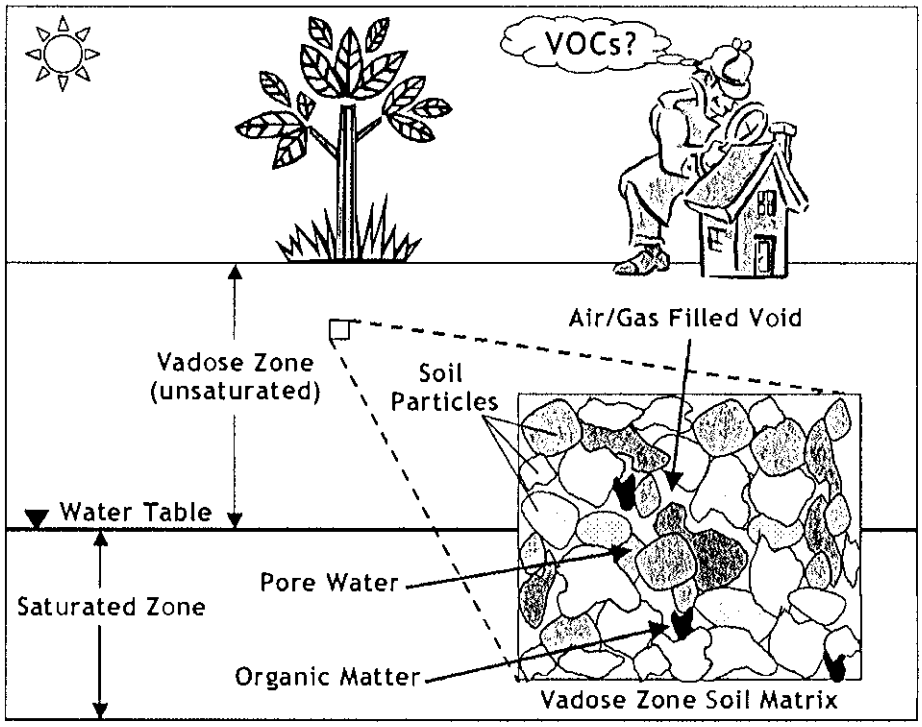


Figure 1: Schematic showing relationship of the vadose zone and saturated zone. Also exploded view showing the primary components of the soil matrix. VOCs will equilibrate with the solid, liquid, and gas phases of the soil matrix. Free product may also be present in the void space of the soil matrix.

Previously, soil gas sampling and monitoring were used primarily as cost- and time-effective means to track soil and groundwater contamination by VOCs and identify potential source areas (US EPA 1997). As our understanding of the vapor intrusion pathway develops, the use of soil gas data is being applied to evaluate this potential human health hazard. Because the vapor intrusion pathway deals with human health risk assessment, the sample and data quality objectives for soil gas samples are more stringent for this application than for simple plume tracking and source location activities. This technology overview will provide guidance on selection of the appropriate tools and methods for soil gas sampling and monitoring depending on the data quality objectives of the project.

It is useful to realize that VOCs will be partitioned between the different components (phases) of the vadose zone (Figure 1) (ASTM D5314, McCall et al. 2006) depending on their physical and chemical characteristics. The VOCs will be present in the soil gas phase, dissolved in any liquid water that may be present, and probably sorbed to the solid soil particles, especially any organic matter. Near the source of contamination, free product also may be present in the soil. The distribution of the contaminant(s) in different phases of the soil matrix is important to consider when sampling. If a strong vacuum is applied to the sample interval, VOCs that were partitioned into the liquid phase or sorbed to the solid matrix may be stripped and enter the vapor phase. This can result in an inaccurate measure of the amount of the contaminant actually in equilibrium with the vapor phase and lead to erroneous conclusions in a risk assessment.

3.0
VOCs and Other Soil Gases of Interest

The primary contaminants of interest in soil gas sampling and vapor intrusion studies are chlorinated volatile organic compounds (X-VOCs), aromatic compounds, and some landfill gases (Table 1). The volatility of a compound is primarily a function of its vapor pressure. The higher the compound's vapor pressure (Table 1) at ambient conditions the greater it's volatility. This simply means any spilled liquid will quickly evaporate and enter the gas phase. If spilled on soil or leaked into the subsurface, a compound will tend to enter the soil atmosphere. Don't forget these compounds also will be partitioned into the other phases of the soil matrix (Figure 1). In general, compounds with vapor pressures greater than about 0.5 mm Hg are considered to be detectable with active soil gas methods.

At many sites, VOC releases will contaminate the groundwater and then migrate down gradient with the flowing groundwater. Over time these groundwater contaminant plumes can become sources for VOC migration into the overlying soil gas that may potentially become a vapor intrusion source. The Henry's Law constant of a compound is used to describe its tendency to partition between water and vapor. One way to determine the Henry's Law constant of a compound is to divide the equilibrium concentration of the compound in air by it's concentration in water. This provides a unitless ratio. The higher the Henry's Law constant the more the compound tends to enter the gas phase. Usually compounds with Henry's law constants greater than 0.1 and sufficiently high vapor pressures are considered to be detectable with active soil gas sampling (EPA 1997).

4.0
Conceptual Site Model (CSM)

The conceptual site model (CSM) is a simplified picture and/or written description of what may be a complex system (U.S. EPA 2002). The purpose of the CSM is to provide the investigation team and other interested parties an initial idea about the subsurface conditions, possible contaminant migration pathways, potential receptors, local soils and geology, and potential sources for the volatile contaminants under investigation. The CSM should include preliminary site maps, geologic cross sections and other pertinent diagrams. This may include maps of sewer lines, water lines and other underground utility lines that may provide preferential conduits for migration of volatile contaminants to homes, schools, offices and other buildings where vapor intrusion could pose a health hazard. Sensitive environments and endangered species also should be evaluated.

A good CSM will help the investigation team identify additional information that will be required to complete the investigation and determine if the vapor intrusion pathway is complete. The CSM should be viewed as a dynamic tool that will be updated and modified as additional information, samples, and data are obtained that better characterize the site conditions. Soil samples may be required to verify local soil and geological conditions and identify potential source areas. Groundwater samples may be required to determine if a groundwater plume is acting as a source for volatiles across the site area and what the contaminant concentrations are. Measurements of hydraulic conductivity or soil permeability may be needed to evaluate the potential rate of contaminant migration. Several direct push methods are available to obtain these samples and other information needed to confirm or modify the CSM to accurately reflect site specific conditions. Direct push electrical conductivity logs, hydraulic profiling tool (HPT) logs, membrane interface probe (MIP) logs for volatiles, and CPT logs may provide methods to achieve a detailed site model in a relatively short time frame. In addition, ASTM Standards are available for particular methods and procedures for sampling and investigation that may provide useful insights and special knowledge. Some pertinent ASTM Standards particular to direct push methods include D6001, D6282, D6725, and D7242.

Section 5.0
Data Quality Objectives (DQOs) and Selection of Sampling Methods

As mentioned previously, the data quality objectives will determine how samples are obtained in the field and analyzed. The project manager and field team will have to determine which downhole tools are required and the specific sample collection and analysis methods that should be used to meet the DQOs of the project. But first, what are data quality objectives? DQOs are basically a set of rules that define the amount of uncertainty that is acceptable in the results obtained from an environmental investigation. How confident are you that the sample you collected is representative? Are you sure the analytical method applied accurately identifies the contaminant and what its concentrations are in the sample?

One of the first steps in determining your DQOs is to clearly define the regulatory requirements that must be met to determine if the potential hazard (e.g. vapor intrusion) is an actual threat to the population of concern. Review the state or federal regulations and guidance documents (Cal RWQCB 1997, Cal. DTSC 2003, NYDOH 2005, U.S. EPA 2002, etc.) to be sure you know what is required and how the data is to be used. The following outline of objectives for three different projects may help clarify where and when different levels of data quality are required.

Project 1:

- Are VOCs present in soil gas?
- What are gross concentration ranges?
- What is approximate extent of the contamination?

Project 2:

- What are the specific types of VOCs present in the soil gas?
- What are approximate concentrations of each contaminant type at each location?
- Define and map the extent of the contaminant plume and locate source areas.

Project 3:

- Determine the specific VOC compounds present in the soil gas.
- Determine the concentration of each compound at each location with good precision and accuracy.
- Define and map the plume for each analyte and locate sources.
- Determine if the contaminants present a threat to human health or the environment (is the vapor intrusion pathway complete?).
- Obtain data that can be used to determine appropriate remedial actions or if remedial actions are required.

Once you determine what level of data quality is required you can then determine the appropriate sampling tools, sample collection procedures, and analytical methods that are required to meet your objectives. Table 2 may be used to guide you in selecting the appropriate methods to use in the field to be sure the project manager, facility owners, and regulators are able to make the correct decisions. **Establishing project DQOs and selecting the appropriate field methods for sample collection and downhole tool systems is not a trivial process and should be completed well in advance of starting field work.** Details of the sample collection methods and downhole sampling systems listed in Table 2 are discussed in the following sections.

Of course the final data quality is controlled by the analytical method chosen and the analytical quality control that is used. You can collect a low quality sample and spend hundreds of dollars on an expensive analytical method with rigorous quality control, this would be a mistake. If you have low confidence in the sample quality (poorly representative) it will not matter how much you spend on the analysis, the final data will still be of low quality (poorly representative). You can't improve the quality of a bad sample by using expensive analytical methods. In the end the analytical result is only as good as the sample quality. For guidance on selection of the appropriate analytical methods review the appropriate federal or state guidance documents (Cal RWQCB 1997, Cal. DTSC 2003, NYDOH 2005, U.S. EPA 2002, etc.) and/or the SW846 manual (EPA 1986).

5.1 Analytical Methods

The EPA Draft Guidance for the vapor intrusion pathway includes a table listing more than a dozen analytical methods used to test for VOCs. The purge and trap methods such as 8260C were primarily written for soil and groundwater analysis. Purge and trap methods may be useful if you are conducting passive soil gas sampling and collecting VOCs in a sorbent trap. However, direct injection may be conducted with method 8260C and this provides a useful method for analysis of soil gas samples. Method TO-17 is also a method that requires the use of a sorbent trap for sample collection and later purge and trap gas chromatograph (GC) analysis. This is sometimes used for ambient air monitoring in buildings.

(continued on Page 6)

Table 1: Basic chemical properties[A,B,C,D] of selected VOCs and gases of interest in soil gas investigations.

Chemical Name	Molecular Formula	Molecular Weight	Density (g/cc)	Henry's Law Constant Ratio[C] ($atm \cdot m^3/mol$)[B]	Boiling Point (°C)	Water Solubility %^ (g/L @ 25°C)[B]	Vapor Pressure (mm Hg @ 68°F)	Viscosity[B] (mPa·s @ 25°C, 1 atm)	Ionization Potential (eV)
Aromatics and Aliphatics									
Benzene	C_6H_6	78.11	0.877	0.215	176°F	0.04% @ 54°F (1.77)	75	0.604	9.24
Toluene	$C_6H_5CH_3$	92.1	0.87	0.262	232°F	0.05% @ 61°F (0.53)	20 (at 65°F)	0.56	8.82
Ethyl Benzene	$C_6H_5CH_3CH_2$	106.2	0.87	0.322 (8.5E-3)	277°F	0.01% (0.16)	10 (at 79°F)	0.631	8.76
Xylenes (o,m,p isomers)	$C_6H_4(CH_3)_2$	106.2	0.88 to 0.86	0.304	269°F to 292°F	~ 0.01% (0.16 to 0.18)	7/9/9	0.760, 0.581, 0.603	8.44 to 8.56
(n-)Octane	$CH_3(CH_2)_6CH_3$	114.2	0.70		258°F	~insoluble	10	0.508	9.82
Methyl t-butyl ether (MTBE)[D]		88.15	0.74	0.018 (5.5E-4)	55.2	(51.2)	245	---	---
Chlorinated VOCs									
Vinyl Chloride	CH_2CHCl (chloroethene)	62.5	0.911	(1.1E-2)	-13.3 (7°F)	~0.1% @ 77°F (2.8)	>760	---	9.99
1,1 Dichloroethane (1,1-DCA)	$CHCl_2CH_3$	99.0	1.18	---	135°F	0.6%	230mm (at 77°F)	0.464	11.06
1,2-Dichloroethylene	$(CH)_2Cl_2$ (1,2-DCE)	97.0	1.27 (at 77°F)	(3.0E-2)	118° to 140°F	0.4% (2.4)	180 to 264	Cis = 0.445 Trans = 0.317	9.65
1,1,2-Trichloroethane	$CHCl_2CH_2Cl$ (TCA)	133.4	1.44	---	237°F	0.4% (4.4)	19	—	11.0
Trichloroethylene	$ClCH=CCl_2$ (TCE)	131.4	1.46	---	189°F	0.1% @ 77°F	58	0.545	9.45
Tetrachloroethylene	$Cl_2C=CCl_2$ (Perchloroethene)	165.8	1.62	---	250°F	0.02% @ 77°F (1.2)	14	0.844	9.32
Carbontetrachloride	CCl_4	153.8	1.59	---	170°F	0.05% (1.2)	91	0.908	11.47
Landfill Gases									
Methane	CH_4	16.04	0.4228^{-162}	---	-161.5	Soluble	-	---	12.98
Carbon dioxide	CO_2	44.0	—	---	sublimes	0.2% @ 77°F	>760	---	13.77

g/cc = grams per cubic centimeter. atm·m3/mol = atomosphere cubic meter per mole. °C = degrees centigrade. °F = degrees Fahrenheit. g/L = grams per liter.
mm Hg = millimeters of mercury. mPa·s = millipascal second = centipoise. atm = atmosphere. eV = electron volt
A) NIOSH Pocket Guide to Chemical Hazards, 1990. B) CRC Handbook of Chemistry and Physics, 1995. C) U.S. EPA, 1997. D) www.epa.gov/opptintr/chemfact/s_mtbe.txt

Table 2: Matrix for selection of field methods to achieve DQOs.

Downhole Sampling System		Sample Collection Method			
		Syringe	Tedlar Bag	Glass Bulb	Summa Canister
	Increasing Quality →				
Direct Sampling	↓	*Low / Low*			*Low / High*
PRT System					
Implants					
Gas Wells		*High / Low*			*High / High*

Dashed arrow shows general path for selection of methods that will result in sampling and collection with compatible quality procedures to achieve desired DQO level. If method pairs in the upper right or lower left corner are used the final sample analytical quality will be of low confidence regardless of the analytical method applied. To achieve intermediate DQOs for an initial survey the PRT system is often combined with collection in Tedlar bags or glass sampling bulbs and field analysis utilizing a mobile lab equipped with a gas chromatograph.

For active soil gas sampling you will probably be involved with one or more of the following methods:

Method #	Analytes	Instruments	Det. Limits
8260	Aromatic, Halogenated and other VOCs	GC/MS	50 ug/m³
8260 SIM	Aromatic, Halogenated and other VOCs	GC/MS	10 ug/m³
8021	Aromatic and Hologenated VOCs	PID/ELCD-GC	> 5 ug/m³
TO-15	VOCs in Air with Summa Canisters	GC/MS	1 to 10 ug/m³
TO-17	VOCs in Air with Sorbent Tubes	GC/MS	0.5 to 25 ppbv

GC = Gas Chromatograph MS = Mass Spectrometer PID = Photo Ionization Detector
ELCD = Electrolytic Conductivity Detector

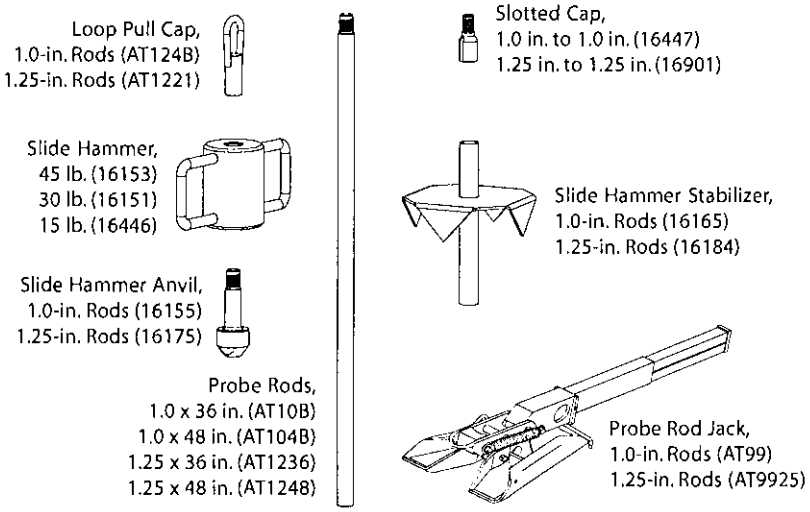
The 8021 Method is often modified for use in a mobile lab in the field. Air samples are direct injected on column for analysis. Sometimes an ECD (electron capture detector) is used instead of the ELCD(electrolytic conductivity detector) to detect the chlorinated VOCs. Field analysis with the 8021 method allows the field team to track/map plumes while in the field and locate source areas. The field analysis is also a useful way of screening samples to determine which are best submitted for more expensive off-site analysis. Knowing approximate sample concentrations also may be used to select the appropriate lab analysis.

For soil gas samples with elevated concentrations the standard 8260 method will provide sufficient sensitivity and detection limits. Samples with lower concentrations may be suitable for the 8260 SIM (selected ion monitoring) method. Low concentration samples requiring lower detection limits and high data quality may require the TO-15 method. Be aware that not all labs provide the same level of data quality for the same method. A cheaper price may mean less QA/QC. Be sure the final data package will meet your data quality requirements.

Section 6.0
Downhole Soil Gas Sampling Methods for Preliminary Assessments and Plume Tracking

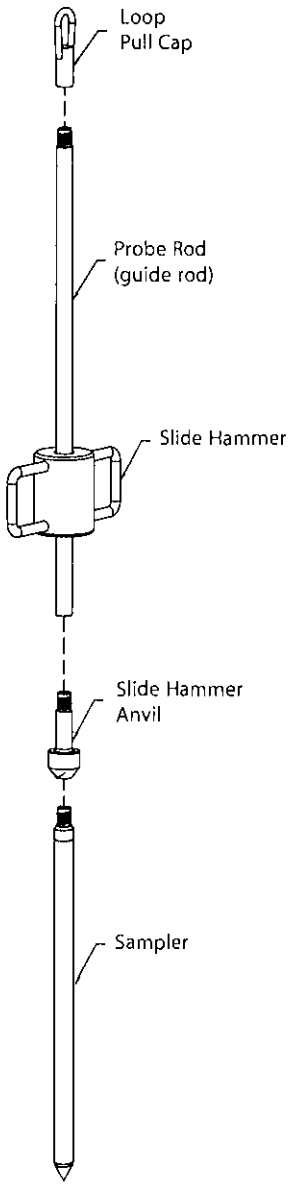
6.1 Methods to Advance Tool Strings

Several methods for soil gas sampling were developed in the 1980s and used to track VOC plumes and locate potential source areas. One of the earlier methods included manually driving galvanized steel pipe to the desired depth with a slam-bar or fence-post-driver. While physically demanding, these manual methods may still prove valuable in accessing delicate ground in residential areas (landscaped plant beds, etc.) or where access is otherwise limited. Improvements to this manual method have been made to improve sample quality and minimize the physical demands for driving and removing the probe rods (Figure 2) (Geoprobe Systems®, 2001). When site access conditions permit, soil gas sampling tools may be advanced into the subsurface with a Geoprobe® direct push machine (Figure 3) to minimize the physical effort required and speed up the sampling process.



A: Components of the manual slide hammer system.

C: With the tool string aligned for driving, lift the slide hammer to a comfortable height (as shown) and then quickly lower the hammer until it strikes the anvil. Maintain a constant hold on the hammer handles while driving.



B: Assembly view of slide hammer system.

Figure 2: Components, assembly, and operation of the manual sampling tools.

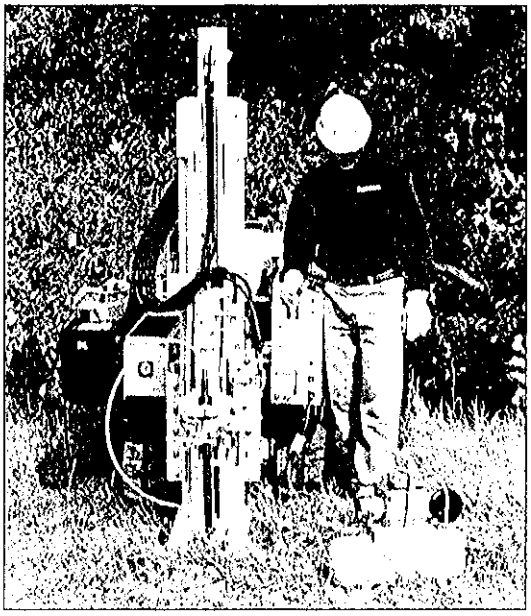


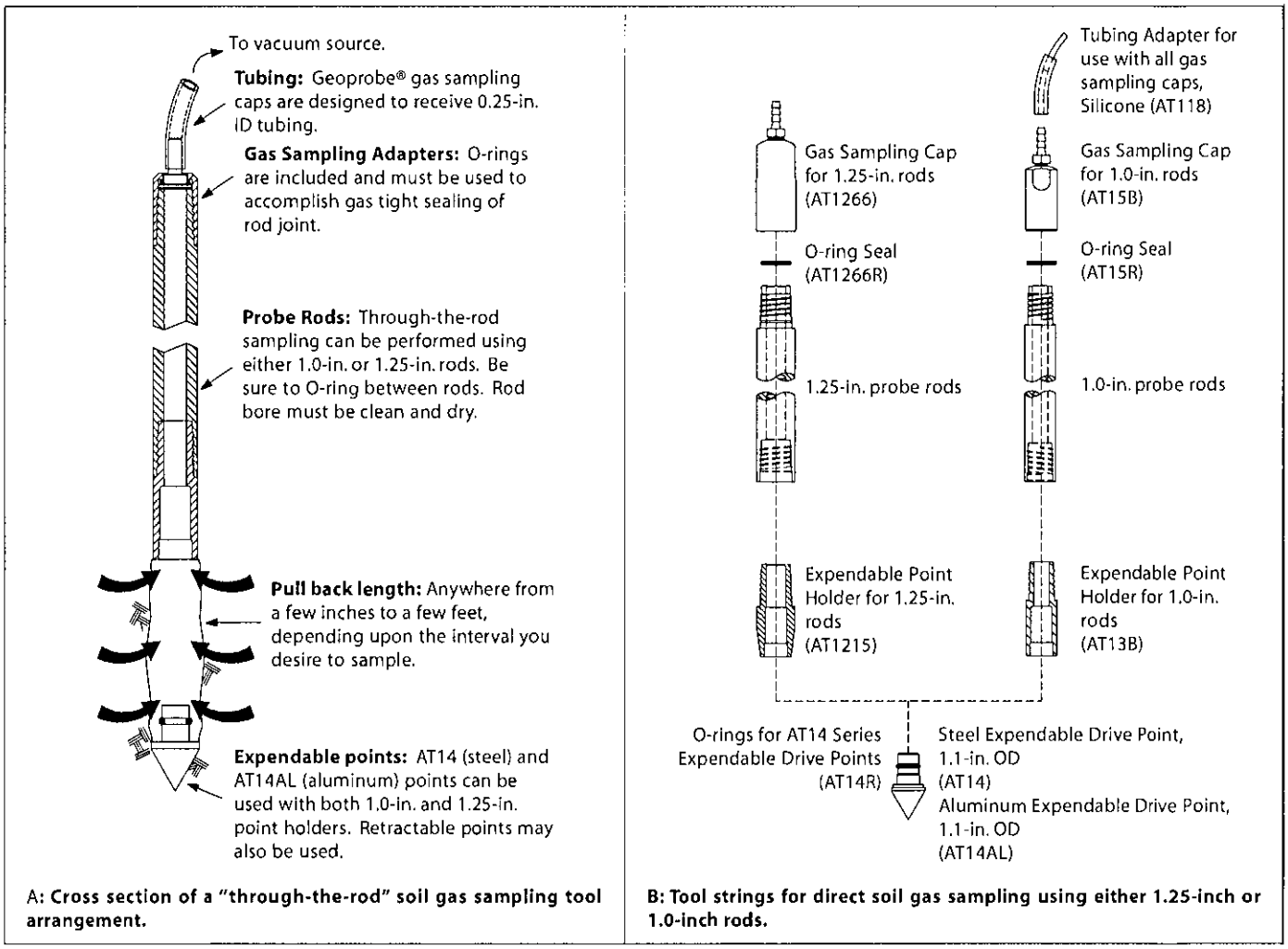
Figure 3: Using a Geoprobe® direct push machine to advance soil gas sampling tools to depth. Note use of portable vacuum/volume system for purging and sampling.

6.2 Direct Sampling System

Initially the collection of soil gas samples was conducted directly through the rods driven to depth (Figure 4). An expendable point was advanced to depth on the leading end of the drive rods. Then the rods were retracted a specified distance to release the expendable point and create an open void where soil gas could enter the rod string for sampling. A gas sampling cap with appropriate fitting was attached to the top of the drive rods and gas was purged and then collected for analysis. While this method is quick and cost effective there is potential for leaks at each rod joint that could compromise sample quality. This method also requires that the steel rods be thoroughly decontaminated between each use. In addition, some contaminants may be sorbed or degraded on the metal surface of the drive rods, especially if the rods are rusty.

6.3 PRT Sampling System

The Post Run Tubing (PRT) system was developed to help eliminate some of the problems with sampling directly through the steel rods. The PRT method uses an adapter and tubing to isolate the soil gas sample from the steel drive rods (Figure 5). This also eliminates possible leaks of ambient air from the rod joints into the gas being sampled. Tool configurations for soil gas sampling with the PRT system using different size probe rods are shown in Figure 6. PRT adapters (Table 3) must be selected to fit the desired tubing (Table 4) that will be used for sample collection.



A: **Cross section of a "through-the-rod" soil gas sampling tool arrangement.**

B: **Tool strings for direct soil gas sampling using either 1.25-inch or 1.0-inch rods.**

Figure 4: Direct sampling systems.

The basic steps to conduct soil gas sampling with the PRT system are as follows:

- An expendable point holder is attached to the lead rod.
- The expendable point is placed in the point holder, use O-ring.
- The tool string is advanced to the desired sampling depth. Additional probe rods are added as required.
- Next, the stainless steel PRT adapter with appropriate sized barb is attached to the selected tubing cut to length.
- The drive cap is removed from the tool string and the PRT Adapter is lowered on the tubing down the tool string.
- The PRT adapter is threaded into the reverse thread fitting in the top of the PRT expendable point holder by manually rotating the tubing counterclockwise until the O-ring on the adapter seals against the point holder. The top end of the tube may be sealed to minimize gas exchange with the ambient air and possible loss of VOCs.
- A leak test of the PRT tubing system may be conducted at this time by applying vacuum to the tubing. An in-line gauge may be used to verify that leaks do not occur after the vacuum is applied.
- The direct push machine is then used to retract the probe rods a specified interval, usually 6 to 12 inches (or 150 to 300 mm). This provides an open cylinder in the soil through which the soil gas may be purged and sampled.
- The upper end of the tube is connected to the purging/sampling system and the sample is collected.
- The direct push machine is used to remove the probe rods. Once at the surface, the O-ring seal between the PRT adapter and the expendable point may be visually inspected to further verify sample integrity.

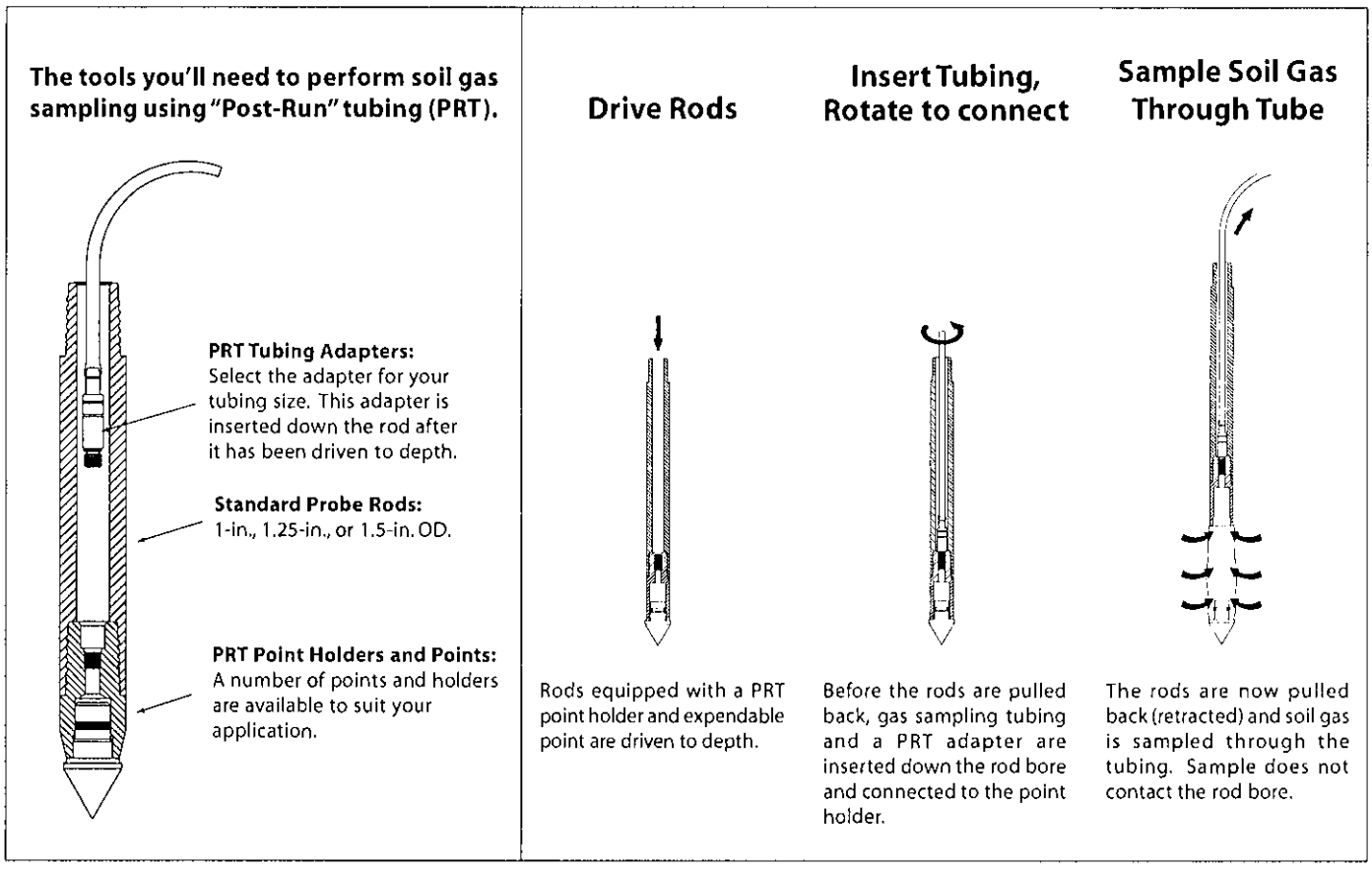


Figure 5: PRT sampling system.

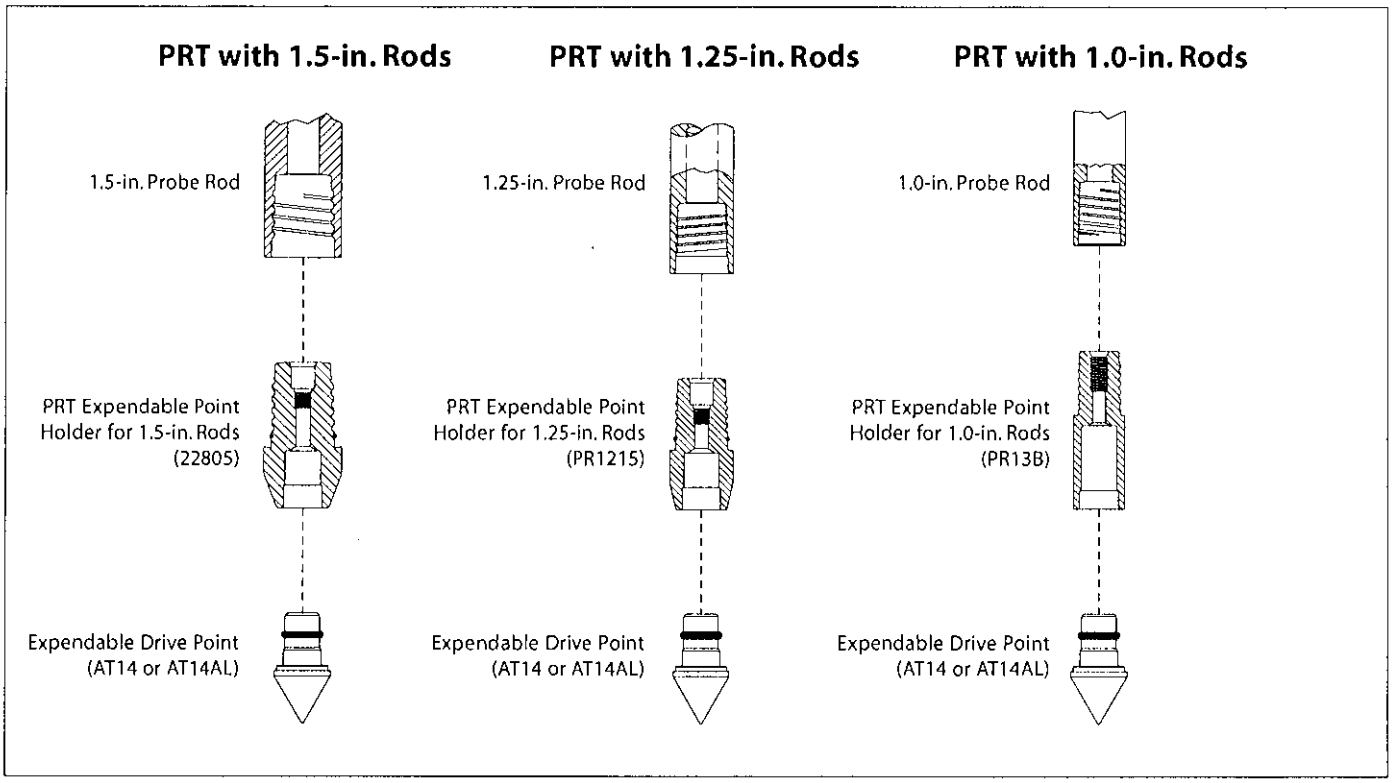
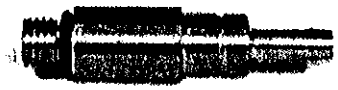
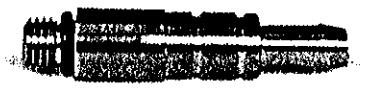


Figure 6: Tool configurations for PRT soil gas sampling.

Table 3: PRT adapter selection guide.

PRT Adapter Selection Guide			
PRT ADAPTER and PART NUMBER	**RECOMMENDED TUBING SIZE (ID)**	**GEOPROBE® TUBING PART NO.**	**TUBING INTERNAL VOLUME**
PR12S	1/8 in. (3.2 mm)	TB12T Teflon®	2.4 mL/ft (7.9 mL/m)
 PR17S	0.17 in. (4.3 mm)	TB17L LDPE	4.5 mL/ft (14.6 mL/m)
	3/16 in. (4.8 mm)	TB17T Teflon®	5.4 mL/ft (17.8 mL/m)
 PR25S	1/4 in. (6.4 mm)	TB25L LDPE	9.7 mL/ft (31.7 mL/m)
PR30S	5/16 in. (7.9 mm)	TB30T Teflon®	15.1 mL/ft (49.5 mL/m)
O-Rings for all PRT Adapters PR25R (pack of 25)			

NOTE: The sorption characteristics of certain tubing may not permit their use in all soil gas sampling applications.

Table 4: Tubing selection guide for PRT adapters and soil gas implants.

Part Number	Tubing Material	Inside Diameter, inches (mm)	Outside Diameter, inches (mm)	Wall Thickness, inches (mm)	Length, feet (m)	Tubing Internal Volume, mL/ft (mL/m	PRT Adapter Part Number	Implant Part Numbers[1]
TB17L	LDPE[2]	0.17 (4.8)	1/4 (6.4)	0.040 (1.0)	500 (152)	4.5 (14.6)	PR17S	AT8617S AT8717S
TB25L	LDPE[2]	1/4 (6.4)	3/8 (9.5)	1/16 (1.6)	500 (152)	9.7 (31.7)	PR25S	AT8625S AT8725S
TB37L	LDPE[2]	3/8 (9.5)	1/2 (12.7)	1/16 (1.6)	100 (31)	21.7 (71.2)	NA	AT9637[3] AT9638[4]
TB12T	Teflon®	1/8 (3.2)	1/4 (6.4)	1/16 (1.6)	50 (15)	2.4 (7.9)	PR12S	AT8612S AT8712S
TB17T	Teflon®	3/16 (4.8)	1/4 (6.4)	1/32 (0.8)	50 (15)	5.4 (17.8)	PR12S	AT8612S AT8712S
TB30T	Teflon®	5/16 (7.9)	3/8 (9.5)	1/32 (0.8)	50 (15)	15.1 (49.5)	PR30S	AT8630S AT8730S
TB45SL	Silicone	3/16 (4.8)	7/16 (11)	1/8 (3.2)	25 (8)	-	NA	NA
TB50TY	Vinyl	1/4 (6.4)	7/16 (11)	3/32 (2.4)	25 (8)	-	NA	NA
TB100TY	Vinyl	1/4 (6.4)	7/16 (11)	3/32 (2.4)	100 (31)	-	NA	NA
AT82	Stainless Steel	0.085 (2.2)	1/8 (3.2)	0.040 (1.0)	50 (15)	-	NA	AT86SW12

1 - 86 series implants are 6 in. (152 mm) long and 87 series implants are 21 in. (533 mm) long.
2 - Low-density polyethylene.
3 - 96 series implants are for use in 1.25-in. or 1.5-in. OD probe rods with an ID of 0.625 in. (15.9 mm). Screen is 12 in. (305 mm) long. Use with any plastic tubing with an ID ranging from 1/4 in. to 7/16 in. (6.4 mm to 11 mm).
4 - This implant is designed for air sparging only and is equipped with a porous polyethylene lining.

6.4 Vapor Implants / Small ID Rods

These simple devices allow the investigator to install a small screen at the desired depth to conduct long-term monitoring of soil gas in the subsurface. The implants may be installed through 1.0-, 1.25-, or 1.5-inch outside diameter probe rods for many applications. However, when installed through these smaller rods it may not be possible to achieve the high-integrity seal required for human health risk assessments as in evaluation for the vapor intrusion pathway. High-integrity seals can be obtained by installing the implants through larger diameter rods as discussed later in Section 7.0.

The implants consist of a stainless steel wire mesh screen with a threaded fitting on the bottom for anchoring and a fitting at the top to connect to tubing (Figure7). The AT86 series screens are 6 inches (152 mm) long, the AT87 series screens are 21 inches (533 mm) long, and both have an OD of less than 0.5 inches (13 mm). The AT96 series screens are 12 inches (305 mm) long with an OD of less than 0.625 inches (16 mm). All have a pore diameter of approximately 0.006 inches (0.15 mm) in the stainless screens (Table 5).

The implants are installed through the drive rods after they are advanced to the desired depth (Figure 8). An expendable implant anchor/drive point at the lead end of the rod string has a threaded fitting at the upper end and the implant is anchored in place by threading in counterclockwise. As the rods are retracted with the direct push machine, fine glass beads (P/N AT84) may be poured in around the screen to prevent clogging. Once the rods are above the screen, a bentonite powder mix (P/N AT85) may be added to the rod annulus to seal the probe hole. Do not hydrate the bentonite until the rod string is completely removed from the ground. The tubing should be capped to prevent venting to the ambient atmosphere until ready for sampling. A simple PVC pipe with cap may be installed over the implant tubing at the surface for protection. Conventional well protectors (P/N WP1771 or WP1741) may be used if necessary.

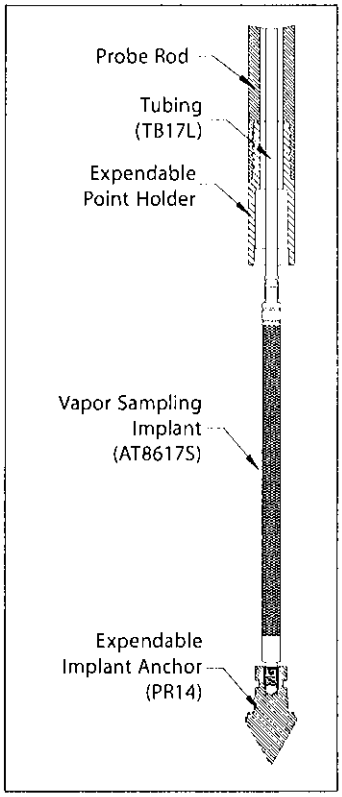


Figure 7: Schematic of a soil gas implant assembly.

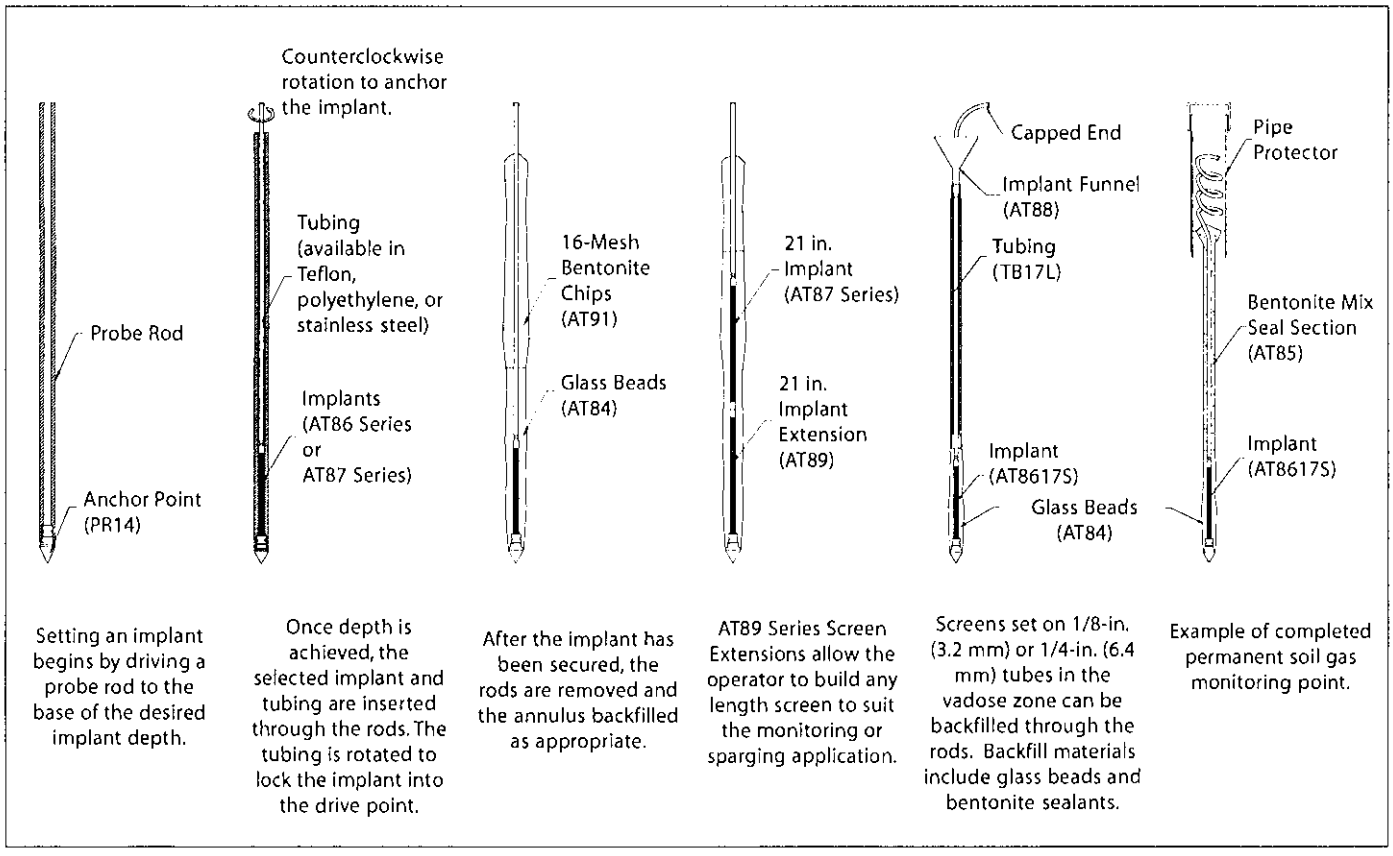


Figure 8: Installing implants through small-diameter probe rods.

Table 5: Implant selection guide.

END FITTING	SUGGESTED SAMPLING USE	RECOMMENDED TUBING SIZE	GEOPROBE® TUBING PART NO.	IMPLANT PART NUMBERS	
				6-in. (152 mm) SCREEN LENGTH	21-in. (533 mm) SCREEN LENGTH
Barbed	Soil Gas	1/8 in. ID (3.2 mm ID)	TB12T	AT8612S	AT8712S
Barbed	Soil Gas	0.17 in. ID (4.3 mm ID)	TB17L	AT8617S	AT8717S
		3/16 in. ID (4.8 mm ID)	TB17T		
Barbed	Soil Gas, Shallow Groundwater	1/4 in. ID (6.4 mm ID)	TB25L	AT8625S	AT8725S
Barbed	Soil Gas, Shallow Groundwater	5/16 in. ID (7.9 mm ID)	TB30T	AT8630S	AT8730S
Swagelok™	Soil Gas	1/8 in. OD (3.2 mm OD)	AT82 (1/8 in. stainless)	AT86SW12	—
		3/16 in. ID (4.8 mm ID)	—	AT86SW17	—
		1/4 in. OD (6.4 mm OD)	—	AT86SW25	AT87SW25
PRT Socket	Soil Gas, Shallow Groundwater	Screen Extension (for use with AT87 Series Implants)			AT89

For 1.25-in. and 1.5-in. Probe Rods Only		RECOMMENDED TUBING SIZE	GEOPROBE® TUBING PART NO.	12-in. (305 mm) SCREEN LENGTH
Tapered Screw Thread	Soil Gas, Shallow Groundwater, Air sparging (AT9638)	Any plastic tubing 1/4 in. ID to 7/16 in. ID (6.4 mm to 11 mm ID)	TB37L	AT9637 (sampling) AT9638 (sparging)

AT86 Series: Screen is 0.25 in. ID x 6 in. long (6.4 mm x 152 mm) with 0.15 mm pore openings. AT86 implants will fit through 0.5-in. (12.7 mm) bore tooling.

AT87 Series: Screen is 0.25 in. ID x 21 in long (6.4 mm x 533 mm) with 0.15mm pore openings. AT87 implants will fit through 0.5-in. (12.7 mm) bore tooling.

AT9637 and AT9638 Implants: Screen is 3/8 in. ID x 12 in. long (9.5 mm x 305 mm) with 0.15mm pore openings. These implants require a minimum 0.625-in. (15.9 mm) rod bore for passage.

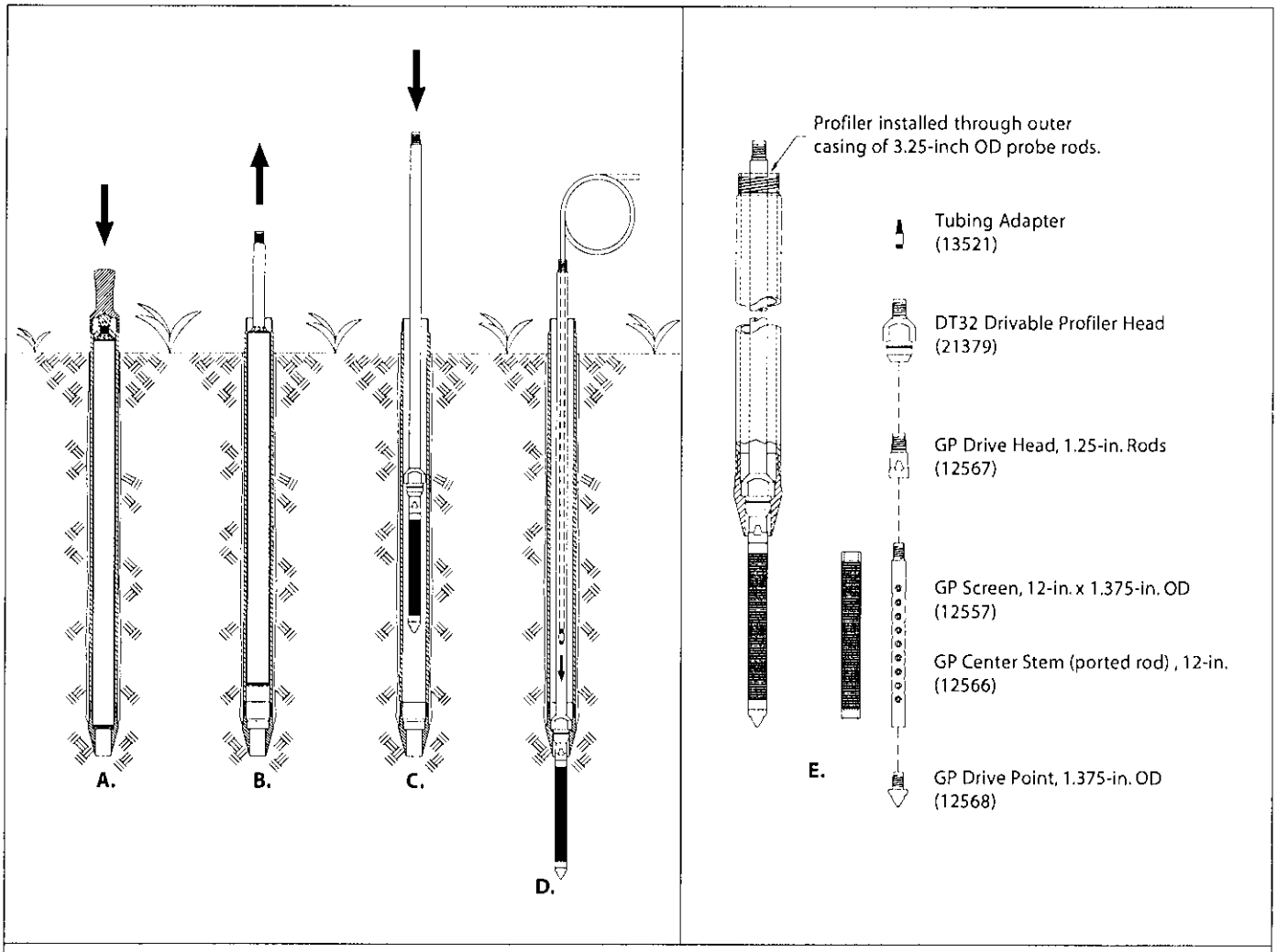
AT9638 implants are the only implants recommended for gas sparging (injection) as they have a porous polyethylene lining inside the implant screen. This interior filter prevents the implant from clogging with formation fines which may backflow into standard implants during periods of pressure shut down.

6.5 Soil Gas Profiling with Dual Tube Systems

In some situations it may be necessary to conduct vertical profiles of soil gas to determine if there is a significant change in concentration or contaminant type with depth. The methods discussed in previous sections could be used to obtain profiles, but a separate advancement would be required for each depth interval. Conversely, dual tube systems can provide access to soil gas or ground water at multiple depths during a single advancement of the tool string. The DT325 soil sampling system can be combined with a simple screen to conduct profiles of soil gas or groundwater if necessary (Figure 9). A summary of the procedure for soil gas profiling follows:

- The equipment operator may conduct continuous soil sampling as the tool string is advanced or install a solid drive point (P/N 28509) if soil sampling is not required.
- Once the cutting shoe is immediately above the desired soil gas sampling interval, the sample sheath and soil sample or solid drive point is removed.
- The profiler screen assembly (Figure 9) with a 12-inch (305 mm) screen (P/N 14402) or 6-inch (152 mm) screen (P/N 14401) is then attached to the inner drive rods and lowered through the annulus of the 3.25-inch rods.
- The screen is pushed through the open cutting shoe and advanced into the formation until the O-ring on the profiler head (P/N 21379) seals in the throat of the cutting shoe.
- At this point the tubing adapter (P/N 13521) is attached to the selected tubing (Table 4) and lowered through the bore of the inner rods.
- The inner tube and adapter are rotated counterclockwise to thread the tubing adapter into the top of the screen drive head (P/N 12567 or 16913).
- This then provides a sealed sampling system for collection of soil gas samples.
- Once the soil gas sample is collected, the tubing is removed and the inner rods and screen are retracted and removed.
- To advance to the next depth, either the solid drive point is installed in the cutting shoe or a soil sample barrel is installed for the next interval. The tool string is advanced to the next desired depth (with removal of soil cores as necessary).
- Installation of the screen and tubing is repeated for the next soil gas sample (Figure 9, Steps C and D).

Once the soil gas profiling is complete, it is possible to install permanent implants at multiple depths as the 3.25-inch rod string is retracted. See Section 7.3 for further discussion of this procedure. Be sure to soil core through any intervals where soil gas implants are to be installed. This will help minimize compression of the formation and loss of gas permeability. If permanent implants will not be installed, abandon the probe hole as required by local regulations (see Section 9.0).



A. Soil sampling as the tool string is advanced to minimize compression of the soil and loss of gas permeability.

B. Extracting the soil sample.

C. Installing the profiler screen through the rod bore.

D. The selected tubing (Table 4) with attached tubing adapter (P/N 13521) are lowered through the bore of the inner rod string and threaded into the Screen Drive Head (P/N 12567). The soil gas is purged and sample is collected.

E. Components of the profiler screen assembly for use with the DT325 system. The 12-inch (305 mm) screen assembly is shown here (P/N 14402). A 6-inch (152 mm) screen assembly (P/N 14401) is also available .

Figure 9: Soil gas profiling with the DT325 system.

Section 7.0
Downhole Soil Gas Monitoring Methods for Evaluation of the Vapor Intrusion Pathway

Data obtained for evaluation of the vapor intrusion pathway (VIP) will generally be used to conduct a human health risk assessment (HHRA). Because the data will be used for a HHRA, the data quality objectives are very high and most of the soil gas sampling methods described in previous sections may not meet the sample quality requirements. To achieve the higher DQOs and to permit long-term monitoring of soil gas, most regulatory agencies are requiring the use of permanent soil gas monitoring wells or implants with filter media around the screens and high-integrity seals. In some situations, multilevel monitoring is required to define the vertical distribution of soil gas in the subsurface and evaluate the potential for contaminant degradation. The following sections outline some field methods that will enable the investigator to achieve the higher DQOs when either single-level or multilevel monitoring of soil gas is required.

7.1 Single-Depth High-Integrity Soil Gas Implants for Monitoring

If the installation of implants with smaller probe rods as described previously will not meet the higher DQOs for your project, the DT21 dual tube system may be required. This system uses 2.125-inch (54 mm) OD by 1.5-inch (38 mm) ID outer probe rods and 1-inch (25 mm) OD inner rods to provide access to the desired sampling interval either by use of a solid drive point or soil coring system (Figure 10, see also Technical Bulletin # 982100). To minimize compression of the formation and reduction of gas permeability, it is best to stop the DT21 system just above the interval where soil gas is to be sampled. The following steps (Figure 11) should be followed to install an implant for long-term soil gas monitoring:

- Once at the desired depth, the inner rod string and any soil sample is removed.
- A small thin-walled sample tube (P/N 19484) with drive head (P/N DT4055) is lowered through the outer rods and advanced ahead of the cutting shoe to remove a soil core where the implant will be installed (Figure 11). This sample tube (P/N 19484) is designed for low-density sands and finer-grained materials only.
- Once the soil core is removed a stiff brush (P/N BU600) may be lowered by extension rods (P/N AT671, etc.) into the open core hole in fine-grained or otherwise cohesive formations. The brush is rotated and moved up and down gently through the cored interval to relieve any smearing and reestablish natural gas flow into the void where the implant will be installed.
- The appropriate 6-inch (152 mm) or 12-inch (305 mm) long implant (Table 5) is attached to the selected tubing (Table 4) and lowered into the open core hole below the probe rods. Cap or cover the end of the tube at the surface to prevent clogging and/or loss of volatiles.
- Next, either environmental sand (P/N AT95) or fine glass beads (P/N AT93) are poured through the rod bore to fill the open hole around the implant screen. The probe rods should be retracted slowly to keep the filter media below the cutting shoe. Measure with suitable depth indicator to confirm the filter media extends at least 6 to 12 inches (152 to 305 mm) above the top of the implant.
- Grouting of the annulus may be accomplished in one of two ways.
    1. Fine bentonite chips (P/N AT91) are gravity poured through the rod bore as the rods are slowly retracted to keep chips below the cutting shoe. The chips are hydrated with water every 4- to 6-inch (102 to 152 mm) increment. Water should be added through a tube extending below the cutting shoe. This will prevent wetting the ID of the rods and possible bridging.
    2. A grout slurry of either bentonite powder (P/N AT92) or neat cement may be pumped by tremie tube using an appropriate grout machine to fill the bore hole as the drive rods are retracted.
- Once grouting is completed, suitable surface protection (P/N WP1771 or WP1741) should be installed to meet local needs and regulatory requirements. In remote locations such as landfills, a simple PVC pipe with slip-cap may provide suitable protection for short-term monitoring requirements.

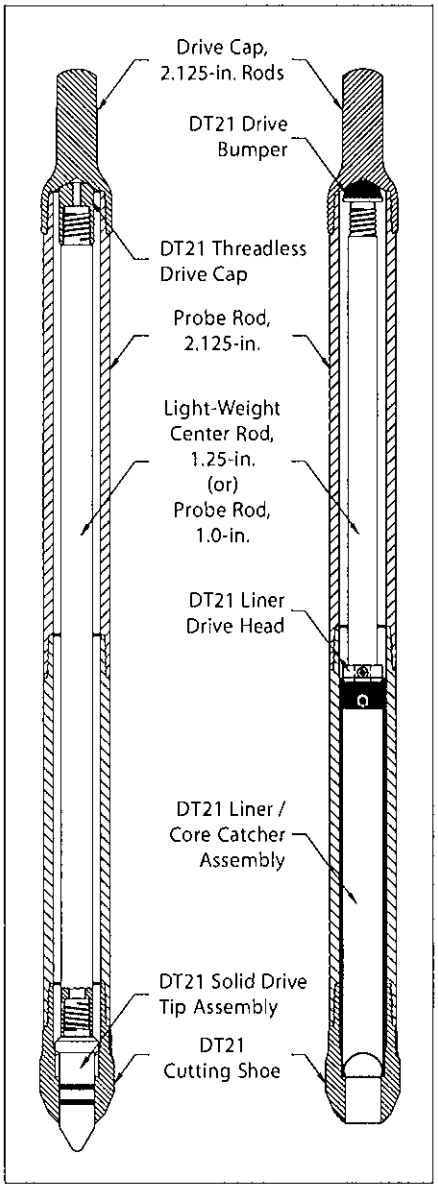
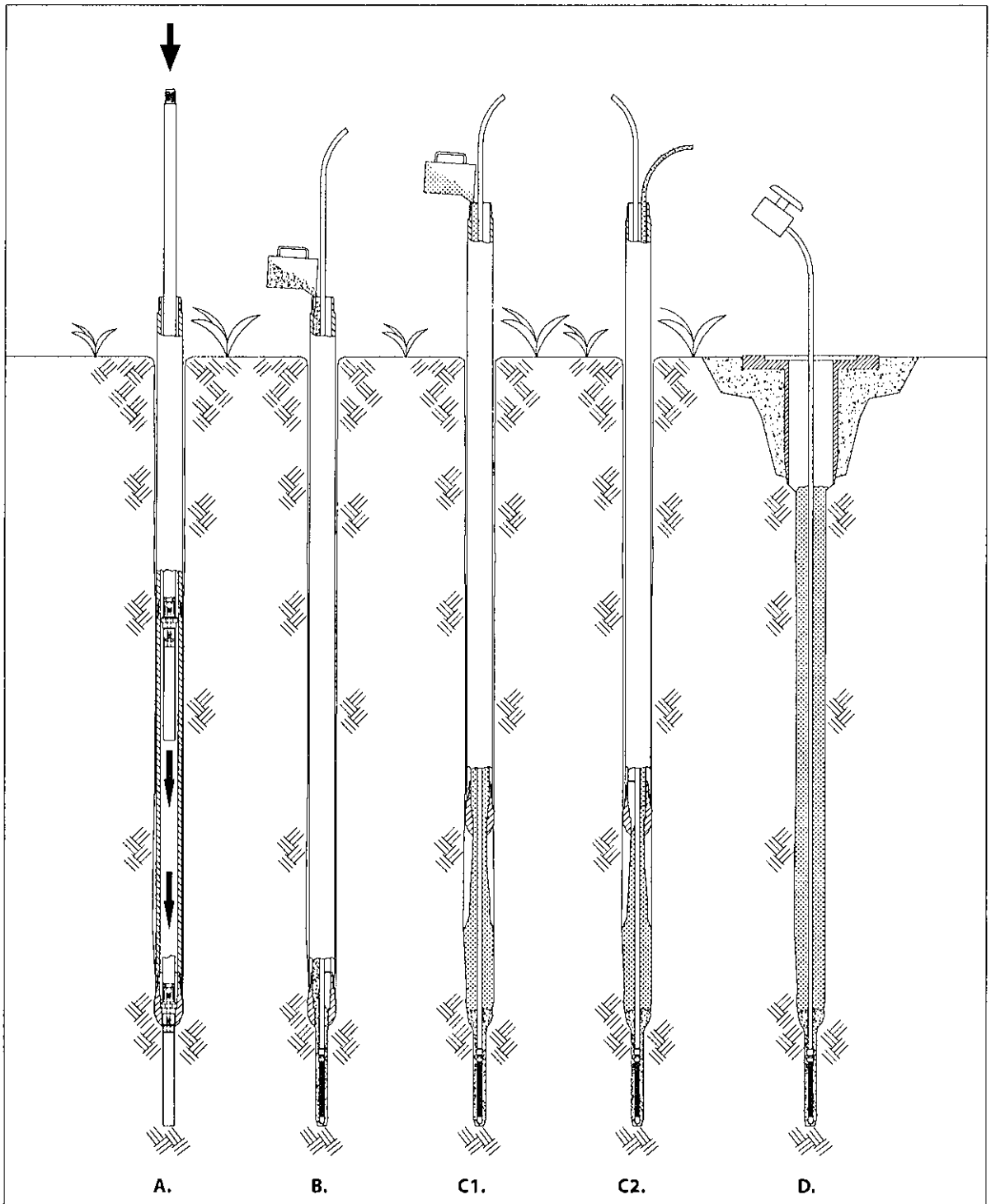


Figure 10: DT21 soil sampling tool string configurations.

The DT325 sampling system may also be used in a similar fashion to install high-integrity single-depth implants for soil gas sampling and long-term monitoring.



A. Advance the DT21 system to depth and remove the inner rod string. The pre-core assembly is attached to the lead inner rod and lowered through the outer rod bore. The thin-walled tube is advanced into the virgin soil ahead of the drive rods to remove a soil core.

B. The soil gas implant is assembled and lowered into the open core hole with the tubing. Fine sand or glass beads are added through the rods slowly to prevent bridging.

C1. Fine bentonite chips are added through the drive rods. A tube with funnel at the surface should be used to add water to hydrate the bentonite incrementally as the rods are retracted.

C2. Alternatively, grout slurry may be pumped through a tremie tube to fill the probe hole as the rods are retracted. Use appropriate pump (GS1000 or GS500) and nylon tremie tube (P/N 11633).

D. The sample tube is sealed with a suitable airtight cap or valve. Well protection is installed to meet local requirements

Figure 11: Installation of single-level implants for soil gas monitoring with the DT21 system.

7.2 Single-Depth, High-Integrity Soil Gas Wells

Under some conditions or to meet specific regulatory requirements, it may be necessary to install prepacked screen monitoring wells (Figure 12) for long-term soil gas monitoring. Smaller diameter wells (e.g. 0.5- or 0.75-inch ID) may be preferred for this option to minimize the amount of purging required before sampling is conducted. The DT21 system allows for this option and will provide high-integrity well construction with an excellent annular seal and well defined screened interval with filter media. This gas well installation process utilizes the same basic procedure as for the installation of prepacked screen wells for groundwater monitoring. See Geoprobe® Technical Bulletin No. 962000 for detailed discussion of this procedure or ASTM Standard Practice D6725. A different cap and seal mechanism will be required for the well to prevent venting of volatiles and permit for appropriate purging and sampling activities.

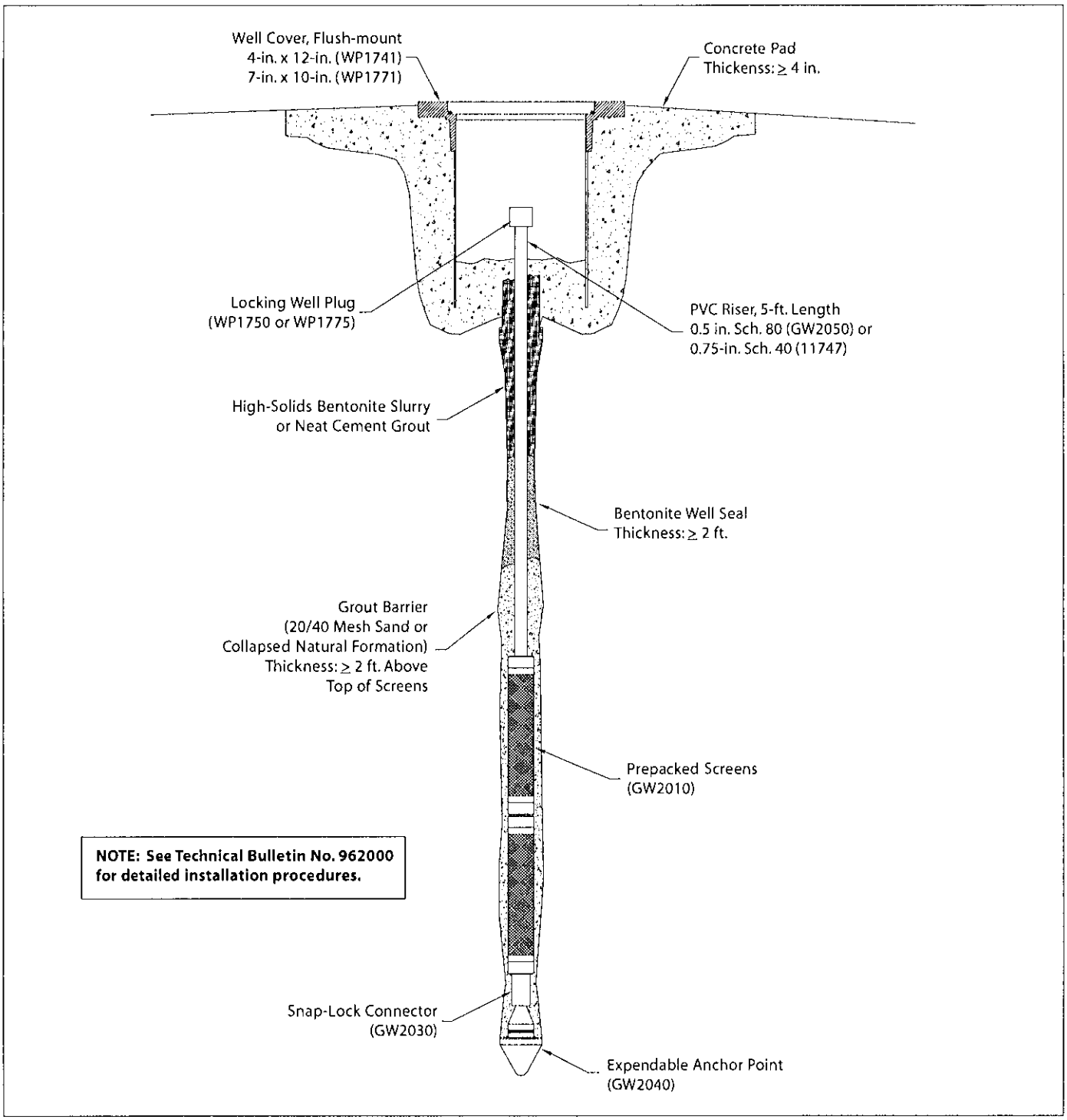


Figure 12: A properly installed Geoprobe® prepacked screen monitoring well.

7.3 Multiple-Depth High-Integrity Implants

Soil gas profiling with the DT325 system was discussed previously in Section 6.5 and Figure 9. As noted, this system may be used to install multilevel soil gas implants as the 3.25-inch diameter rods are retracted. It is recommended that DT325 soil sampling be conducted through the entire interval where soil gas implants are to be installed. Removal of the soil cores will minimize compression of the formation and loss of gas permeability where implants are to be installed. Once soil coring has been completed to the maximum desired depth, the multilevel soil gas implant installation is conducted as follows (refer to Figure 13):

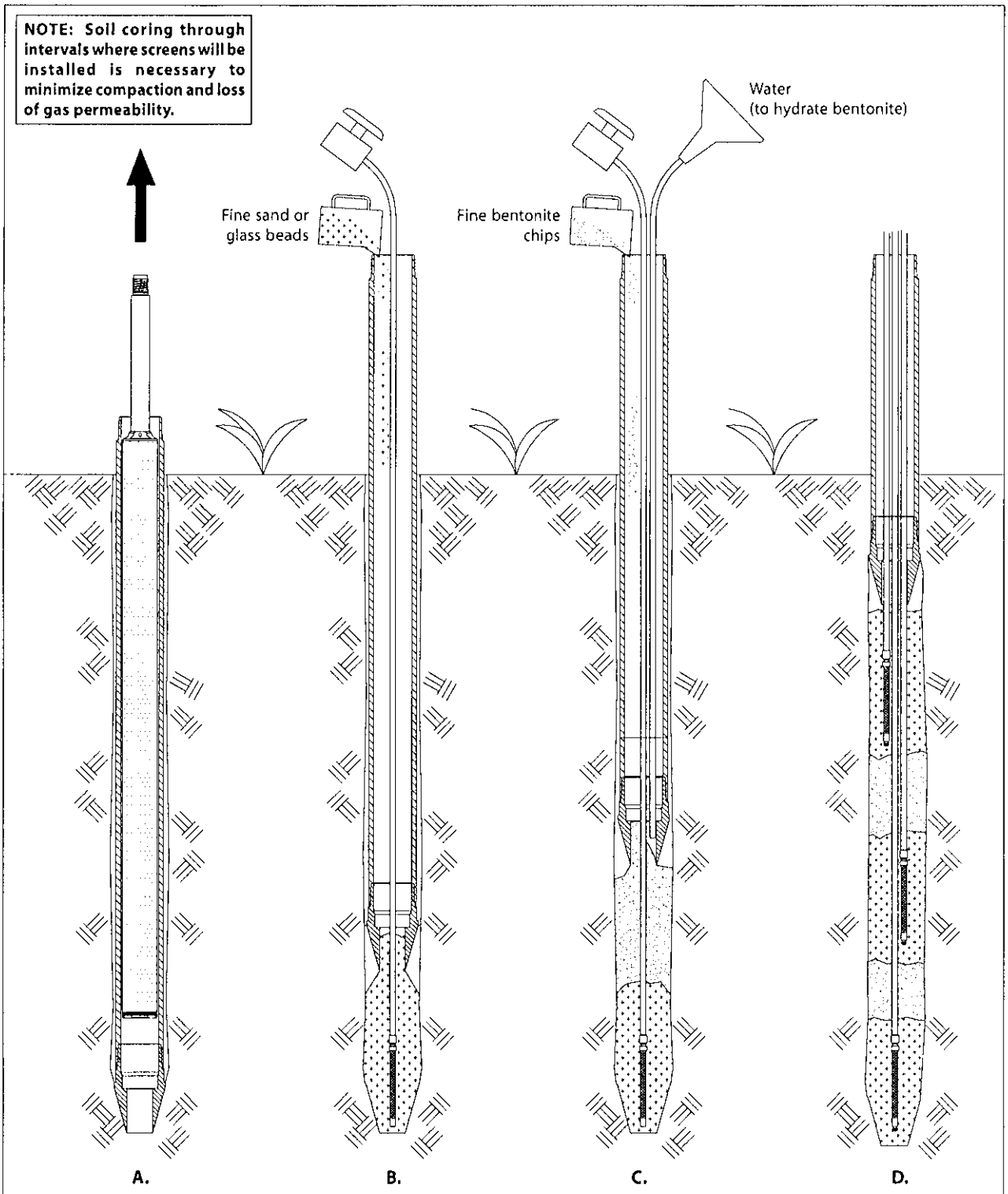
- The inner rod string and last soil core is removed.
- A soil core may be removed below the cutting shoe as described above (Figure 11) for installation of the deepest gas implant, if desired.
- The selected implant (Table 5) and tubing (Table 4) are assembled and lowered to the bottom of the bore hole.
- Filter media is poured through the rod bore as the rods are slowly retracted until filter media extends at least 6 to 12 inches (152 to 305 mm) above the implant. Measure depth to top of filter media and keep below rods and cutting shoe to prevent bridging.
- Fine bentonite chips are poured through the rod bore as the rods are slowly retracted. Water is added through a tube to hydrate each 4- to 6-inch (102 to 152 mm) increment of bentonite. Avoid wetting the interior of the rods to prevent bridging of sand and bentonite that could result in damage or loss of implants. (Alternately, grout slurry could be pumped by tremie tube and grout pump to install the seal at each level. See Figure 11, Part C2.)
- Repeat the last three steps for each soil gas implant as the rods are retracted.
- Grout from top of the filter pack for the uppermost implant to about 1 foot (0.3 m) below grade.
- Install surface protection as required.

Vapor monitoring implants installed following this procedure will enable you to achieve stringent data quality objectives for collection of soil gas samples to be used in human health risk assessments for the vapor intrusion pathway (Cal RWQCB 1997, Cal. DTSC 2003, NYDOH 2005, U.S. EPA 2002, etc.).

Section 8.0
Direct Push Equipment Decontamination and Equipment Blanks

Without appropriate decontamination of downhole tooling and other sampling accessories there is significant opportunity for cross contamination and erroneous results. Various methods may be appropriate for equipment decontamination (decon) depending on the contaminant types and concentrations encountered (ASTM D 5088, Parker and Ranney, 2003). Most often downhole tools such as probe rods and point holders are cleaned with a soap-and-water wash and one or more clean water rinses. This may be most effective for VOCs if a heated pressure sprayer is used to conduct the decontamination. Measures for the control and containment of the spray and wastewater must be implemented. Appropriate health and safety protocols should be followed and adequate personal protective equipment should be worn. Refer to the project specific work plan and health and safety plan for guidance on appropriate safety protocols. Smaller parts may be washed and heated in an oven to drive off volatiles when appropriate.

When field analyses are conducted as sampling is performed in the field the analytical data can be used to guide decontamination requirements. Equipment used to collect samples that are nondetect will require less stringent decontamination measures than equipment used to collect samples from grossly contaminated areas. To assure that cross contamination is controlled equipment blanks should be collected on a regular basis (e.g. once a day or once every 10 samples, as appropriate to meet project DQOs). Equipment blanks should be collected from tool strings and sampling trains assembled just as they are for a typical sample location. The equipment blanks should be labeled and handled as other samples and submitted to the laboratory for the same analytical protocol.



A. Advance DT325 system to depth with soil coring as needed. Remove last soil core.

B. Install the selected implant and tubing. Gravity install fine sand or glass beads to provide a filter pack. Extend filter pack at least 4 to 6 inches (102 to 152 mm) above the top of the implant.

C. Gravity install fine bentonite chips to create annular seal. Use funnel and tubing to incrementally hydrate the bentonite as the rods are retracted. Avoid wetting rod ID to prevent bridging and loss of boring. Alternatively, grout slurry can be installed with a grout pump and tremie tube.

D. Repeat Steps B and C at specified intervals to obtain a multilevel monitoring system. Install well protection to meet local requirements.

Figure 13: Use of the DT325 system to install multilevel soil gas implants.

Section 9.0
Abandonment and Grouting

To prevent migration of contaminants vertically along the probe hole to previously clean areas, proper abandonment and grouting techniques must be followed. For very shallow probe holes (e.g. < 5 ft (1.5 m)) in cohesive soils that remain open after the drive rods are removed, bentonite powder or fine bentonite chips may be added to the open hole incrementally and hydrated. In general, grout slurries should be pumped down the drive rods (Figure 14) or a tremie tube (Figure 11, Part C2) as the original drive rods are retracted from depths exceeding 5 feet (1.5 m). Pumps and accessories capable of injecting bentonite grout slurries or neat cement grout slurries through the small-diameter DP tools (Figure 15) will be required. Usually a 25% solids bentonite slurry is acceptable or a neat cement grout with 7 to 8 gallons (27 to 30 liters) of water per bag of cement may be required. Verify with state and local regulators the type and density of grout material necessary to meet local regulations. Proper abandonment and grouting is necessary to prevent substantial liability for contaminant migration if boreholes are left open.

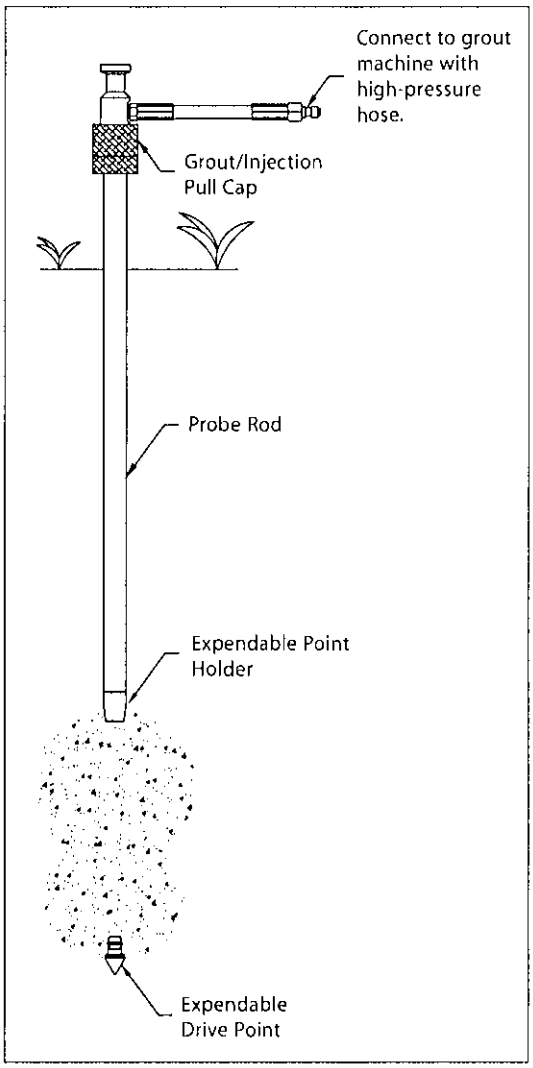


Figure 14: Grouting through tool string after soil gas sampling.

Section 10.0
Techniques for Collection of Soil Gas Samples

10.1 DQOs Select the Technique

As discussed previously in Section 5.0, you must clearly define the DQOs before going to the field to collect soil gas samples. Samples that will be used for a human health risk assessment require much more stringent DQOs than samples that will simply be used to qualitatively determine if VOCs are present in the soil gas. A simple matrix (Table 2) will help you select the appropriate downhole tools paired with the correct sample collection methods to achieve the required sample quality. Remember, an expensive analysis does not make a poor quality sample better, it just wastes your money and time.

10.2 Sample Collection Flow Rate and Purge Volume

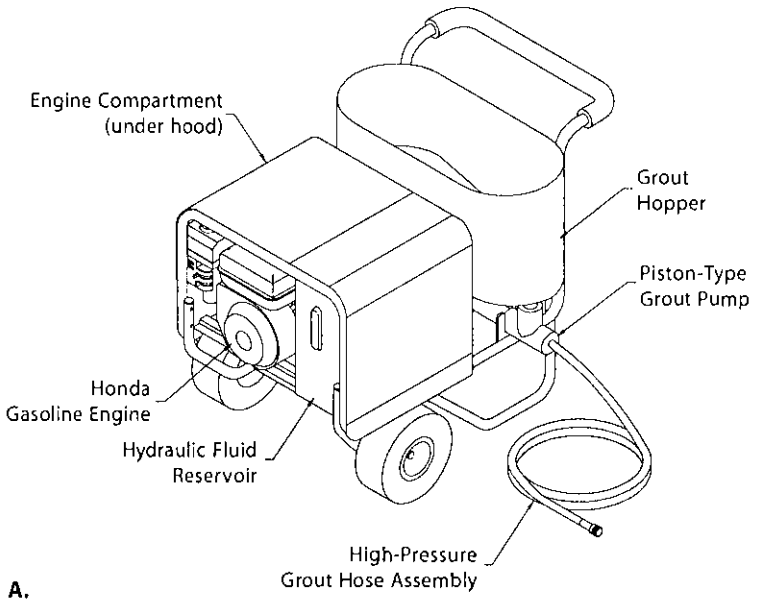
Another important consideration for sample collection is the actual flow rate at which the sample is purged from the ground. Some guidance documents indicate that the purge and sample flow rate for soil gas sample collection should not exceed 200 ml/min. (Cal RWQCB 1997, Cal. DTSC 2003, NYDOH 2005, U.S. EPA 2002, etc.). If very high flow rates or strong vacuum is applied, volatiles that were sorbed to solid phases or liquids in the pore space may be stripped out and enter the gas phase for sampling. Thus, the use of high flow rates or strong vacuum for soil gas sampling may result in a strong positive bias. This could cause an over estimation of the risk to human health and may result in expensive remedial actions being applied that are not necessary.

For most soil gas sampling activities it is common to purge at least one to three volumes from the sampling system before the sample is collected. For direct sampling methods this will simply be the volume inside the drive rods multiplied by three. For PRT methods this will essentially consist of the volume inside the tubing and the open cylinder in the ground created by retraction of the rods. The internal volume of common tubing sizes by foot or meter of length is provided in Table 4.

For permanent soil gas implants or wells, the volume of void space in the filter pack around the screen should be included in the calculation along with the open volume inside the screen and tubing to the surface. For a clean sand filter pack a porosity of 30% is usually considered representative. The following example of purge volume calculation may be useful.

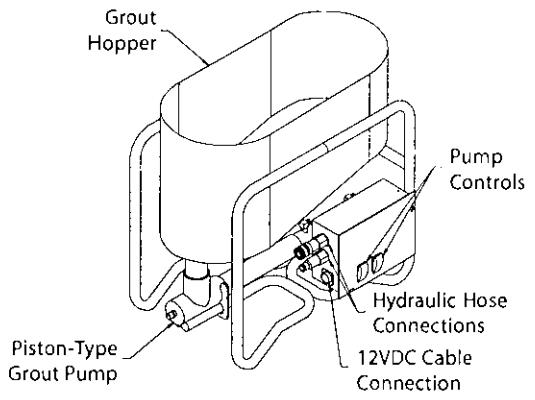
(continued on Page 23)

GS1000 Grout Machine



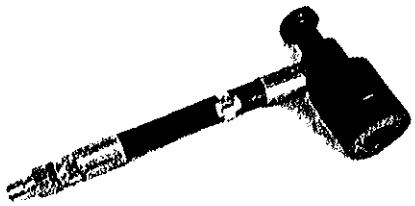
A.

GS500 Grout Machine



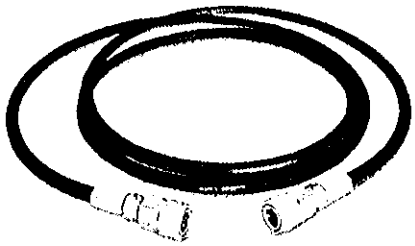
B.

C.



Grout/Injection Pull Cap
16698 - for 1.5-in. probe rods
16697 - for 1.25-in. probe rods

D.



High-Pressure Grout Hose Assembly
GS1051 - 10 ft. (3 m) long
GS1052 - 20 ft. (6 m) long
GS1053 - 30 ft. (9 m) long

A. Self-contained grouting system with gasoline engine and hydraulic fluid reservoir.
B. Portable grout machine (65 lb / 30 kg) that is powered by the hydraulic and electrical systems of a direct push machine.
C. Injection pull caps allow you to pump grout directly through the rod string during retraction.
D. High-pressure grout hoses used to connect the grout pump to the injection pull cap and probe rod string.

Figure 15: Typical grouting equipment for direct push soil gas operations.

Volume of a cylinder = π x radius^2 x height, where $\pi \sim 3.141$

For a 2.125-inch (5.4 cm) diameter borehole 24 inches (61 cm) long, the total volume in milliliters would be:

3.141 x (2.7 cm)^2 x 61 cm = 1397 cc or approximately 1400 ml

For a 0.375-inch (0.95 cm) diameter implant 12 inches (30.5 cm) long, the volume would be:

3.141 x (0.475)^2 x 30.5 cm = 21.6 cc or approximately 22 ml

Now subtract the implant volume from the total probe hole volume and then multiply the remainder by 0.30 to determine the 30% pore space in the filter media. This will be:

(1400 ml – 22 ml) x 0.30 = 413.3 ml

Add back in the total volume of the void in the implant and you have:

413.3 ml + 22 ml = 435.3 ml of void volume in the implant screen and filter media

If you have 12 feet (3.7 m) of 0.25-inch (6.4 mm) ID tubing, the volume per foot from Table 4 is 9.7 ml. One purge volume will be:

435.3 ml + (12 ft x 9.7 ml/ft) = 551.7 ml

and three purge volumes would be about 1650 ml. Purging at 200 ml/min, it would require approximately

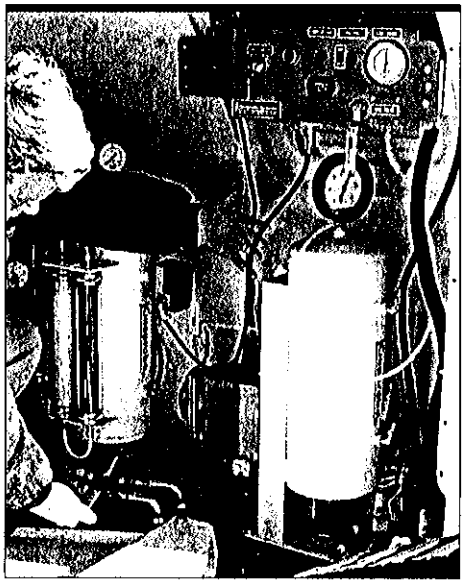
1650 ml / 200 ml/min = 8.25 minutes

to complete the three volume purge for this implant system before you would be ready to collect your sample. So you would want to estimate at least ten minutes for sample collection in your cost proposal when similar implants are to be sampled.

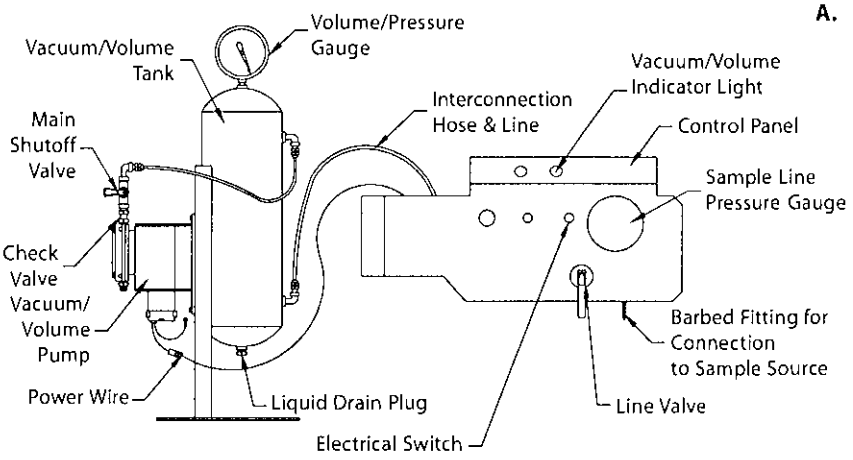
The purge volumes and time required to purge using the PRT sampling system with three different probe rod diameters, using a one-foot (30.5 cm) retraction/void is summarized below in Table 6.

Table 6: Purge volume and time with PRT system and selected rod diameters.

Rod Diameter, inches (cm)	1.0 (2.54)	1.25 (3.18)	1.5 (3.81)
Void Radius (r), inches (cm)	0.5 (1.27)	0.625 (1.59)	0.75 (1.91)
Void Lenght (L), inches (cm)	12 (30.5)	12 (30.5)	12 (30.5)
Single Void Volume (mL) (π x r^2 x L, where $\pi \sim 3.141$)	155	242	350
Tubing ID, inches (cm)	0.25 (0.64)	0.25 (0.64)	0.25 (0.64)
Tube Volume (mL/ft)	9.7	9.7	9.7
Tube Length (ft)	10	10	10
Total Tube Volume (mL)	97	97	97
Total System 1-volume (mL)	252	349	447
Approximate 3-System Volume (mL)	750	1050	1350
Required Purge Time at Flow Rate of 200 mL/min	3.75 min	5.25 min	6.75 min

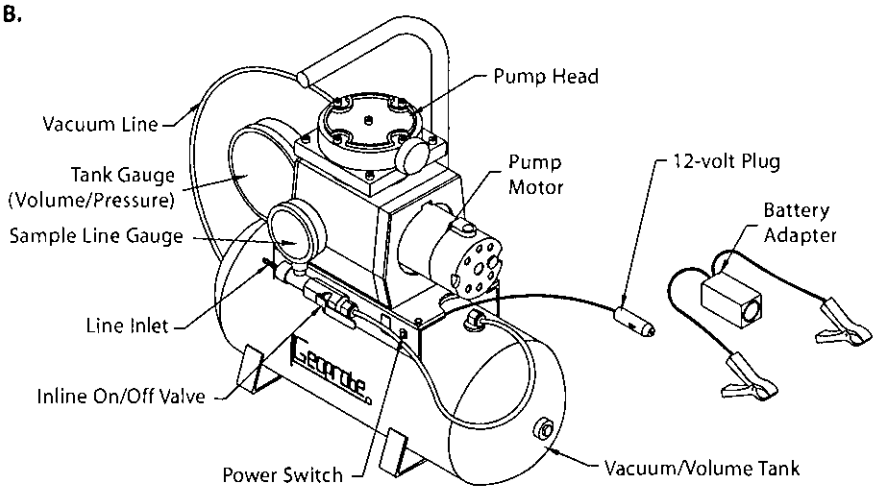


A.



AT1001 - Mountable Vacuum/Volume System
Weight: 35 lb (16 kg)

B.



AT1000 - Portable Vacuum/Volume System
Weight: 40 lb (18 kg)

A. This system mounts in the back of a truck or van, often along with a Geoprobe® Model 5410 direct push machine.

B. The portable 12V system may be powered from the vehicle 12V accessory outlet (cigarette lighter) or clipped directly to the vehicle battery.

NOTE: Both systems include an 11-liter (2.9 gal.) tank and can provide up to 21 in. of Hg (70 centibars) of vacuum.

Figure 16: Vehicle-mounted and field-portable vacuum/volume systems for soil gas sampling.

10.3 The Vacuum/Volume System

The vacuum/volume (V/V) system (Figure 16) has been used to good advantage for soil gas sampling during soil gas surveys, especially when large purge and sample volumes (liters) are required. The V/V system allows the field operator to perform three basic functions that are essential to successful soil gas sampling. These are measurement of the:

- volume of gas extracted at a sample point
- initial vacuum applied to a sample point
- amount of time for a sampling point to return to atmospheric pressure after a vacuum has been applied for sampling

The field operator may wish to incorporate an adjustable needle valve at the sample line inlet to control the flow rate to the desired level. The portable V/V system may be useful for periodic sampling of permanent wells or implants for monitoring purposes. Detailed operation instructions for the V/V system are available (Geoprobe® 2003).

10.4 Syringe sampling & purging

During initial surveys and plume tracking efforts, a mobile lab with onboard gas chromatograph (GC) may be used to conduct field analysis. This can significantly reduce analytical costs and the need for multiple mobilizations to determine the extent of the contaminant plume. When a van-mounted Geoprobe® direct push machine is equipped with a GC it is convenient to collect the soil gas sample directly from the PRT sample line with the syringe that will be used to inject the gas sample on the GC column (Figure 17). A short section of silicone tubing is placed in-line to allow for insertion of the GC syringe and collection of the sample. The sample is then immediately injected in the onboard GC for analysis. This collection and field analysis technique is especially amenable to use of the Triad approach for site characterization (EPA 2003).

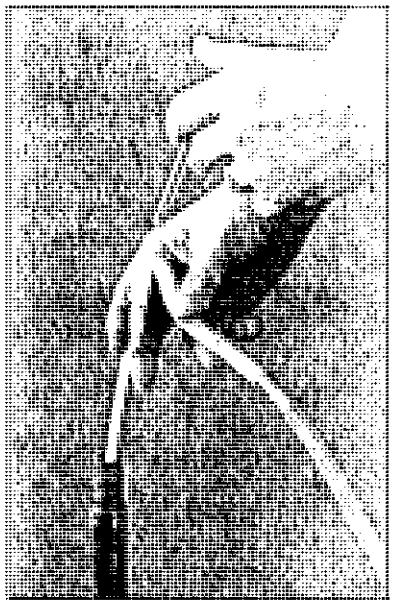


Figure 17: Syringe sampling for field analysis.

When smaller purge volumes are required, a large syringe may be used to purge the sample tube. Gas sampling syringes made of glass or plastics are available from scientific supply warehouses in sizes ranging from 50 ml volume to 250 ml volume. This may be a particularly useful approach when shallow sampling is done in areas that are difficult to access with larger tools and equipment. Usually a small on/off valve is fitted to the end of the sample tube and is closed as the syringe is removed to be emptied and reattached for additional purging. The field tech will have to manually control the purge and sample flow rate. After purging the syringe may be used to inject a sample into a Tedlar bag, or glass bulb for on-site analysis. If off-site analysis will be conducted, a small summa canister may be used to collect the sample.

10.5 Tedlar bags and glass sampling bulbs

These two sample collection options (Figure 18) have been widely used in soil gas surveys, especially when a mobile lab is stationed on-site to conduct the analyses as the samples are collected. These sample collection options also may be used when an off-site lab is to perform analyses but holding times for the samples may be limited. In addition, standard Tedlar bags and glass bulbs are transparent and exposure to sunlight is a concern. Some volatile organic contaminants may degrade when exposed to sunlight (especially ultraviolet light) so the sample containers must be stored out of the sunlight. A cooler or suitable box may be sufficient. Do not cool the samples if stored in a cooler as this may cause condensation and lead to biased sample results. Light-blocking Tedlar bags are available.

There are at least two ways to collect a sample in Tedlar bags. A large syringe may be used to extract the sample from the sample line and then it is injected into the Tedlar bag. Appropriate valves must be used on the syringe and bag to prevent ambient air contamination of the sample during transfer. Decontamination of the syringes between samples will be required to prevent cross contamination. An alternate method is to place the Tedlar bag inside a vacuum chamber with a port that allows connection to the sample line. A vacuum is applied outside the Tedlar bag and the bag expands and fills with gas from the sample line and downhole source. The downhole sample train must be purged before sample collection. Tedlar bags are generally considered a one-use disposable item, not to be decontaminated for reuse. Some regulatory agencies (Cal. EPA) discourage the use of Tedlar bags for soil gas sampling.

(continued on following page)

Alternatively, glass bulbs may be decontaminated for multiple reuse. However the glass bulbs are expensive and fragile, and decontamination requires additional equipment and man-hours. Blank analyses will also have to be performed periodically on the bulbs to verify no cross contamination occurs. The glass bulbs are usually placed in line during the purging process so they equilibrate with the gas being sampled. Smaller volume bulbs will require smaller system purge volumes and should still provide sufficient sample volume for duplicates and other QC measures if required. Decon may be accomplished by attaching the open bulbs to a vacuum pump and purging the bulbs while they are being heated in a small oven in the mobile lab on-site. A tubing manifold can connect several bulbs to one vacuum pump.

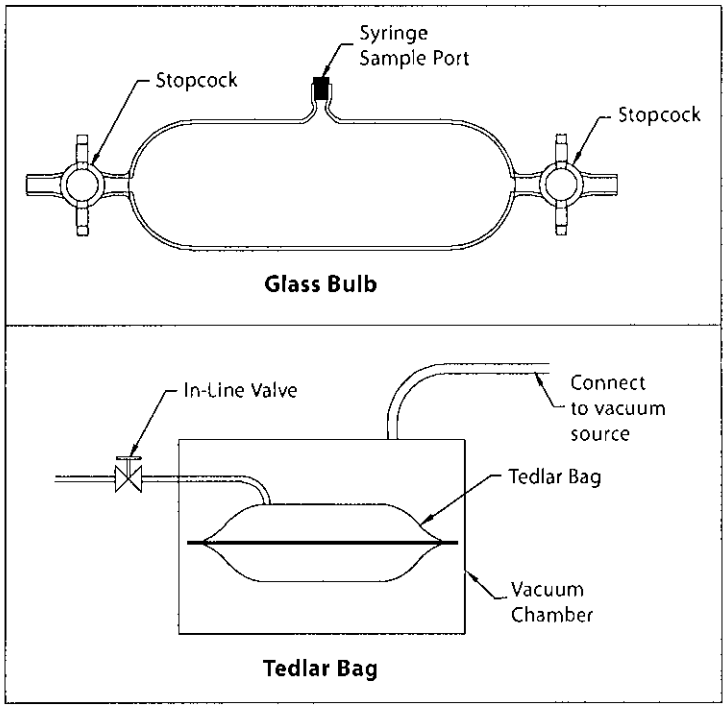


Figure 18: Tedlar bag and glass bulb for soil gas sampling.

10.6 Summa Canisters

For evaluation of the vapor intrusion pathway and human health risk assessments, summa canisters (Figure 19) are preferred for sample collection and transport to a fixed lab facility for analysis. These stainless steel devices are usually supplied by the lab clean and ready for sample collection. The canisters are under vacuum and are simply attached to the sample line for collection of the soil gas. (The three-volume pre-sampling purge is conducted before the summa canister is attached to the sampling train.)

A vacuum gauge and valve should be included on the canister inlet. The gauge will indicate when the canister is full and the valve can be used to control the sample flow rate from the downhole source at the 200 ml/min rate to prevent stripping of volatiles and biased sample results. Flow control valves may be set by the supplier at the requested flow rate. Smaller summa canisters (500 ml) may be preferable to minimize the sample collection time at the 200 ml/min flow rate.

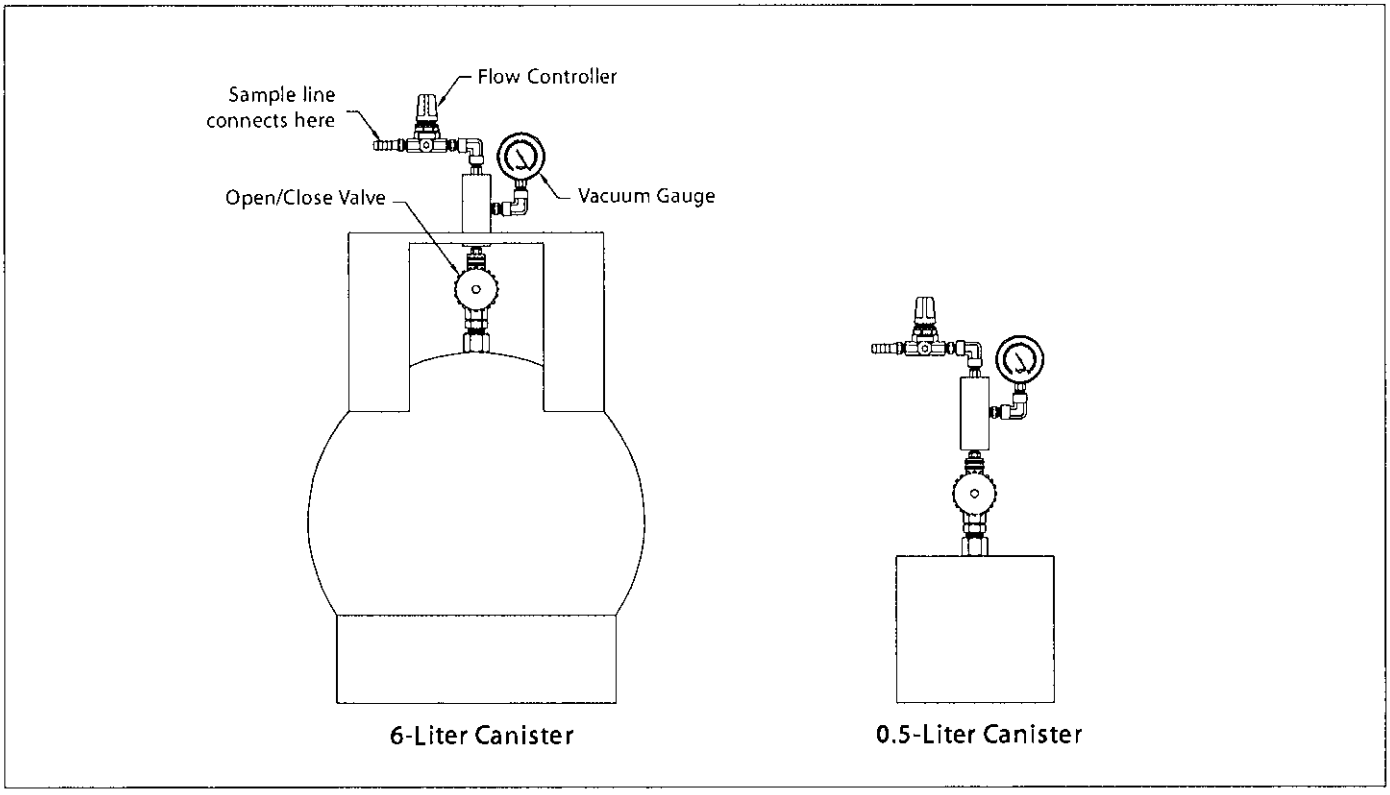


Figure 19: Summa canisters used for collection, storage, and transport of high-quality soil gas samples.

Section 11.0
System Leak Tests for Field QC

Leak testing is a critical step to perform in the field to assure that system leaks do not degrade sample quality and cause biased results. There are at least three categories of leak testing that should be considered. These are:

- Integrity of vacuum source
- Integrity of the sampling train
- Integrity of downhole tool system

The vacuum source may be as simple as a large syringe or as complex as a vacuum/volume system (see Figure 16). To verify that the vacuum supply itself does not have leaks, it should be tested periodically (e.g. once a day or every 10 samples). To test the integrity of your vacuum system the inlet valve should be closed or the inlet line should be plugged appropriately. The vacuum source is activated (syringe plunger retracted/vacuum pump turned on) and vacuum is applied. An in-line vacuum gauge should be included to quantitatively monitor loss of vacuum over time. Once the vacuum is applied, the system is "shut-in" so that loss of vacuum over time may be observed. For more complex systems the leak test may need to last 30 minutes to check for smaller leaks. Corrective action should be taken if leaks are detected.

Most often system leaks occur at aboveground plumbing connections where the sampling device is connected in-line between the vacuum supply and the sample line from the subsurface (sampling train). Probably the easiest and most effective way to determine if there are leaks during the sampling process is to apply a volatile compound around the fittings and analyze for that compound at the lab. One of the simplest and most effective ways to accomplish this is to wet paper towels with isopropyl alcohol and wrap them around the fittings and connections in the sampling train. Isopropyl alcohol may be purchased at the local pharmacy or department store. Remember, this material (liquid and vapor) is flammable and should be stored and handled appropriately.

Some downhole grab sampling systems can be tested for leaks before sampling is conducted. This may be accomplished for the direct sampling system (Figure 4) and the PRT sampling system (Figure 5). Expendable points with O-rings must be used so that an effective leak test can be accomplished. [Note: Neither monitoring nor grab sampling systems with downhole screens can be leak tested in this manner.] For the direct sampling system, the gas sampling cap with O-ring is installed on the drive rods and the tubing is connected between the cap and vacuum supply. Before the rods are retracted from the expendable point, a vacuum is applied to the sampling train and downhole system. The system is shut-in and the in-line vacuum gauge is observed for a period of time to determine if leaks are present.

For the PRT system, the PRT adapter (with fresh O-ring) is installed on the selected tubing. The adapter and tubing are lowered through the drive rods and the adapter is threaded into place. Before the drive rods are retracted off of the expendable point, vacuum is applied to the sampling train. The in-line vacuum gauge is observed to determine if leaks are present. Once the integrity of the sampling system is confirmed, the drive rods are retracted and soil gas purging and sampling is conducted.

A note of caution: System leak tests as described above will not allow you to determine if ambient air or soil gas from overlying zones is migrating into the interval being sampled. In order to prevent this problem, be sure that the expendable point and point holder are no larger in diameter than the outside diameter of the drive rods used in the tool string. Additionally, bentonite powder may be poured around the tool string at the ground surface and hydrated to help prevent ambient air intrusion during the sampling process.

Section 12.0
Mapping Soil Gas Plumes

During the initial stages of a soil gas or VIP investigation it is wise to construct site maps that plot the location of each soil gas sample collection point. Then once the sample analyses are completed, the results may be plotted on the map and contours drawn to define the probable extent of the plume and areas of differing concentration (Figures 20 and 21). In many situations this will help locate sources for the contaminant plumes and define the general direction of contaminant migration. Often, soil gas plumes will develop over groundwater contaminant plumes and roughly mimic the extent of contamination in the local groundwater. In many urban and suburban areas there may be multiple plumes of different contaminants originating from different sources. Sometimes the source area(s) may be located outside the initial boundaries set for the investigation.

Use of on-site analysis with a mobile laboratory will optimize the investigation and plume definition. When field analyses are plotted on site maps daily as results are obtained, the field team can determine if new sample locations should be added to better define the plume. Just as important, nondetect samples can indicate that additional sampling locations planned for some areas may be deleted to minimize cost and time requirements. Application of the Triad Approach (EPA 2003) can be used to obtain the data needed while minimizing costs and multiple mobilizations to the field.

Section 13.0
Health & Safety Considerations

All personnel working on an investigation where hazardous contaminants may be present must be OSHA 40 hour health and safety trained as required by 29 CFR 1910.120. The following discussion is provided as a brief overview of some of the health and safety issues that may be encountered during soil gas investigations. The field team should have a written health and safety plan that adequately addresses the possible physical and chemical exposure hazards that may be encountered during the investigation. Daily meetings to review health and safety conditions and any changes in the work environment should be conducted with the field team.

There are many types of health and safety issues to be considered during a soil gas investigation, or at any site where potentially hazardous contaminants may be present. These include physical hazards as well as potential exposure hazards to contaminants under investigation. Some of the physical hazards are associated with the use and operation of tools, equipment, and Geoprobe® direct push machines in the field. Basic precautions should be followed to prevent injury when operating tools and equipment such as wearing hard hats, steel-toe boots, appropriate gloves, eye protection, and hearing protection as necessary. Watch for pinch points on hydraulic equipment, machinery, and tools. Be familiar with the safety features of equipment being operated in the field, such as kill switches. Whenever subsurface sampling is to be conducted, all subsurface utilities must be professionally located and clearly marked to prevent accidents. Overhead utilities, especially high-voltage electrical lines, should be avoided. Adequate spacing between high-voltage lines and equipment masts or derricks should be maintained, check local requirements. When working in areas where vehicular traffic is a concern, wear appropriate high visibility clothing and use traffic cones to mark the work area.

The potential for exposure to hazardous contaminants is a reality whenever subsurface environmental investigations are conducted. The use of appropriate ambient air monitoring equipment is a must. Wearing appropriate clothing (e.g. tyvek, saranex, nitrile gloves) and air purification equipment (e.g. air purifying respirators (APR) or self contained breathing apparatus (SCBA)) for personal protection should be a routinely planned for and donned when necessary. The field team should have a written health and safety plan that reviews all these concerns and more that provides for appropriate measures when ambient air concentrations of contaminants exceeds the predetermined safety levels.

Section 14.0
Summary

There are several tools and methods available to obtain soil gas samples with direct push techniques. Single tube methods are generally more time efficient and are often used during surveys to define the extent of the plume and help with location of source areas. These methods primarily provide sampling at a single-depth with each advance of the tool string. Conversely, dual tube methods are generally a little more time consuming but allow the operator the option to conduct soil gas profiling at multiple depths during one advancement of the tool string. The dual tube systems also allow the investigator to install high-integrity soil gas implants or monitoring wells. The placement of filter media in the screened interval and annular seals by the dual tube methods allows the sampler to achieve sample quality levels to meet the needs of human health risk assessments for the vapor intrusion pathway. Selection of the appropriate sample collection device and procedure is an integral part of achieving the required DQOs. While on-site analysis with a well-equipped mobile laboratory can expedite completion of the field work and plume definition, off-site analytical work may be required under some conditions to meet the more stringent requirements of human health risk assessment for the vapor intrusion pathway.

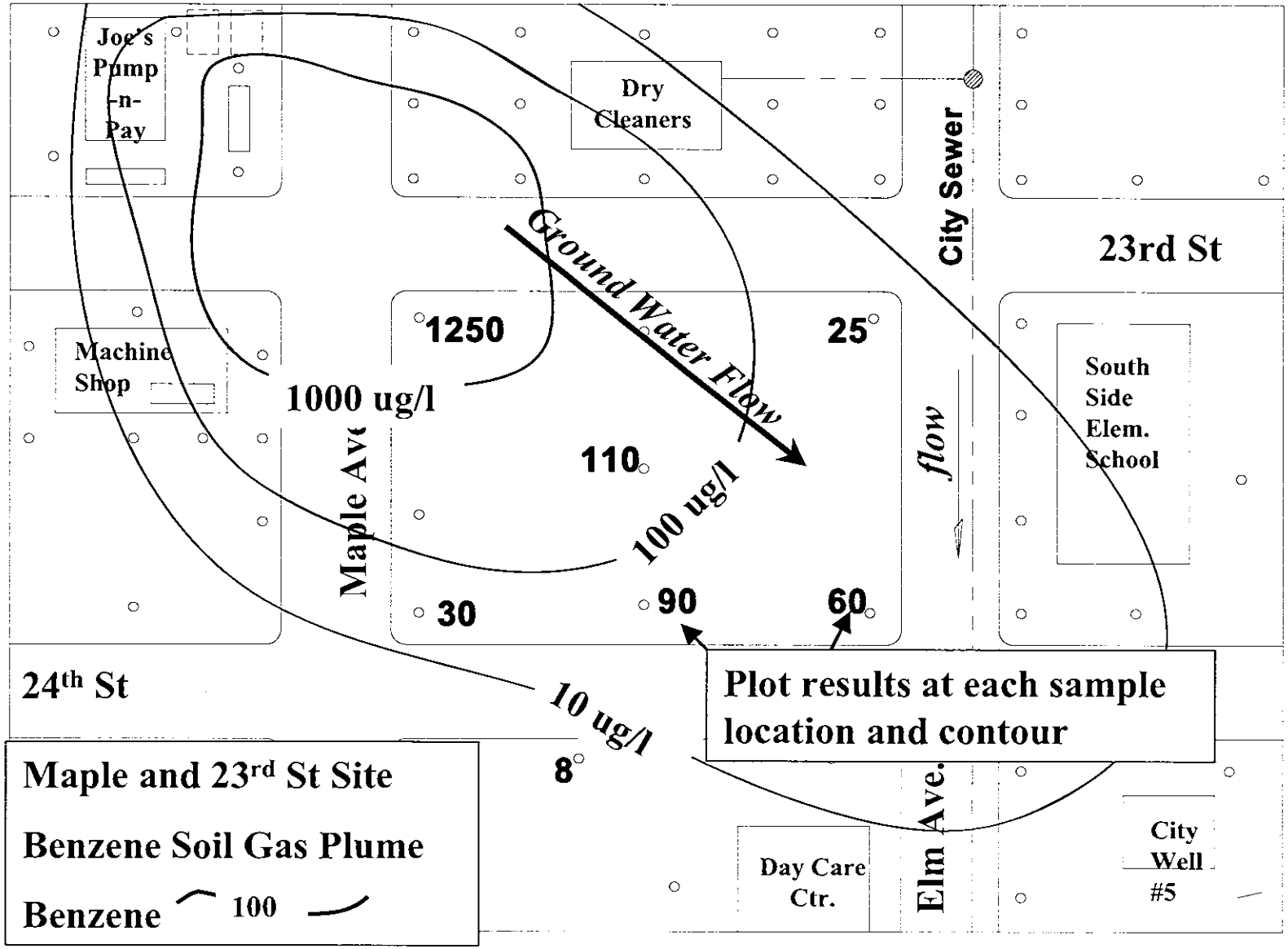


Figure 20: Mapping benzene plume for soil gas investigation.

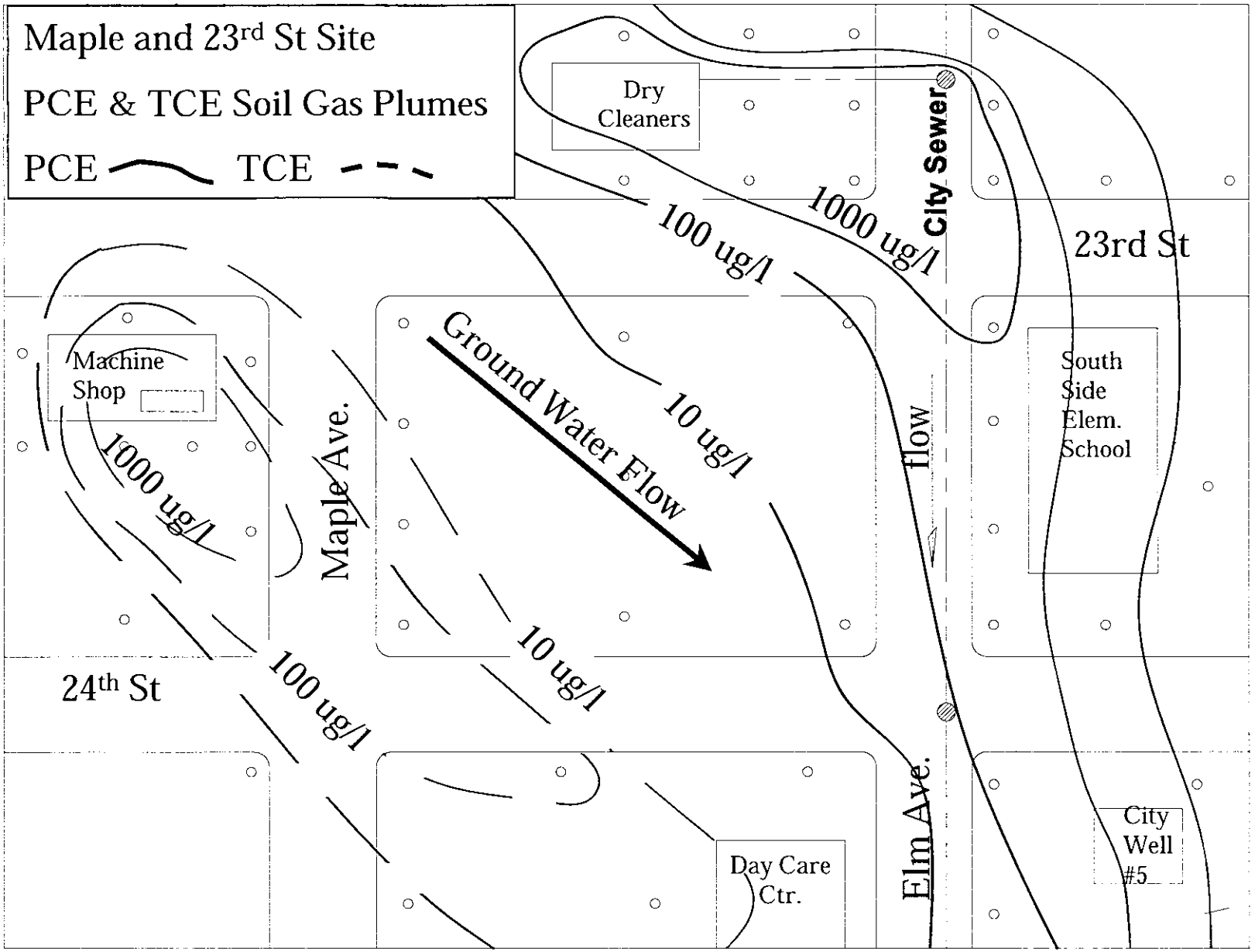


Figure 21: Mapping X-VOC plumes for soil gas investigation.

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Patented.

Typical 3 or 7-Channel CMT Installation using layers of bentonite and sand backfilled from surface.

APPENDIX B.2

ADVANCED GEOLOGICAL SERVICES FIELD PROCEDURES

SANBORN HEAD ENGINEERING

- **GEOPHYSICAL METHODS AND GENERAL FIELD PROCEDURES (UTILITY LOCATING SURVEY)**

Prior to conducting any on site utility mark out work AGS will review any as-built drawings of the facility that are available. Upon arrival at the work site a walk through of the site is conducted to identify locations of features such as pull boxes, water and gas valve, hydrants, PIV's or other features related to underground utilities. All of these features are good indicators of the configuration of underground utilities and can also provide a hook-up location where a tracing signal can be introduce into the utility. After the initial site walk, the tracing and marking out of utilities will be completed using multiple locating techniques.

Integrating two or more geophysical techniques provides a complementary effect resulting in higher confidence levels and more complete subsurface information by reducing the inherent ambiguities in interpretation that may exist if only one method was used. First, AGS will use the Radiodetection RD-8000 pipe and cable locating system to locate and trace buried metallic utilities such as water pipes and electrical cables. To facilitate locating, AGS will use an "inductive clamp" to apply a tracing signal to cables that can be accessed from pull boxes and junction boxes. The inductive clamp wraps around conduit and insulated cables so that the tracing signal can be safely applied without directly touching metallic components of the target utility.

Next, AGS will use the Fisher TW-6 M-Scope electromagnetic metal detector to search for localized buried metal masses that may not be detected with the RD-8000. Finally, AGS will scan the investigation area using GPR (GSSI SIR-3000 with a 270 MHz antenna). GPR profiles will be arranged in a grid pattern centered on the investigation area, and additional GPR profiles will be obtained as necessary to fully delineate any buried objects imaged.

The quality of GPR imaging depends on the nature of the fill soil, and the depth of the subsurface targets. Although dependent on local soil conditions, the typical depth of exploration of the GPR method is 3-to-6 feet below ground surface. AGS will interpret the RF, EM, and GPR data in the field and mark the locations of detected utilities on the ground surface with spray paint, stakes, or pin flags. It is recognized that not all buried utilities are detectable by geophysical methods. Please see below for a brief discussion of the limitations of geophysical utility locating methods.

Limitations of Geophysical Utility Locating Methods

In general, the limitations of utility locating methods stem from the fact that utility locating relies primarily on metal detection, using instruments that detect the magnetic fields associated with electrical current flowing through the utility, detect distortions in a magnetic field caused by metal objects, or detect radio signals traveling through metallic utilities (AGS uses all three

types of devices, in addition to GPR). Therefore, non-metallic utilities, especially when deeply-buried, can elude detection. Even metallic utilities can be difficult to detect if they are deeply-buried and lack a surface connection point where a tracing signal can be applied, or of they are discontinuous and do not allow for sustained current flow, lie in the vicinity of surface metal objects, or run beneath an energized electrical cable or near a high-voltage electrical system, or lie beneath another shallower metal pipeline. Signals from a shallow electric line or metallic utility can often mask the response from more deeply-buried metallic utilities that would otherwise be detected. Additionally, detecting corroded pipes or conduit and unenergized or low-voltage, small-diameter "wires," either directly buried or in PVC conduit, can be problematic if their presence in the area is not known beforehand and/or if they lack a surface connection point where a tracing signal can be applied. Finally, irrigation networks, particularly the smaller irrigation laterals, are not considered viable, cost-effective targets for standard utility locating investigations because they extremely difficult and time-consuming to detect and can be readily repaired or replaced if damaged.

GPR has the potential to detect non-metallic utilities and resolve multiple, closely-spaced utilities, and provide depth information; however, it is subject to investigation depth limitations; in electrically conductive soil (e.g., moist, fine-grained soil), the GPR signal may only penetrate 2 feet. Additionally, sites with heterogeneous fill material often produce "cluttered" GPR records that mask utility images. And finally, a subsurface target requires a certain minimum diameter to be imaged by GPR; a good rule-of-thumb is that a target requires a least 1 inch of diameter for each foot of burial to be imaged with GPR. In other words, a 2-inch pipe buried 4 feet deep probably will not be imaged.

APPENDIX B.3

PARRATT-WOLFF FIELD PROCEDURES

Designation: D 2113 – 99

Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation[1]

This standard is issued under the f xed designation D 2113; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the guidelines, requirements, and procedures for core drilling, coring, and sampling of rock for the purposes of site investigation. The borehole could be vertical, horizontal, or angled.

1.2 This practice is described in the context of obtaining data for the design, construction, or maintenance of structures, and applies to surface drilling and drilling from adits and exploratory tunnels.

1.3 This practice applies to core drilling in hard and soft rock.

1.4 This practice does not address considerations for core drilling for geo-environmental site characterization and installation of water quality monitoring devices (see Section 2).

1.5 The values stated in inch-pound units are to be regarded as standard. The SI values given in parentheses are provided for information purposes only.

1.6 This practice does not purport to comprehensively address all of the methods and the issues associated with coring and sampling of rock. Users should seek qualif ed professionals for decisions as to the proper equipment and methods that would be most successful for their site investigation. Other methods may be available for drilling and sampling of rock, and qualif ed professionals should have f exibility to exercise judgment as to possible alternatives not covered in this practice. This practice is current at the time of issue, but new alternative methods may become available prior to revisions; therefore, users should consult with manufacturers or producers prior to specifying program requirements.

1.7 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Also, the user must comply with prevalent regulatory codes, such as OSHA (Occupational Health and Safety Administration) guidelines, while using this practice. For good safety practice, consult applicable OSHA regulations and other safety guides on drilling **(1)**.

2. Referenced Documents

2.1 *ASTM Standards:*

D 420 Guide to Site Characterization for Engineering Design, and Construction Purposes[2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 4630 Test Method for Determining Transmissivity and Storage Coefficient of Low Permeability Rocks by In Situ Measurements Using the Constant Head Injection Test[2]

D 5079 Practices for Preserving and Transporting Rock Core Samples[2]

D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock[3]

D 5782 Guide for the Use of Direct Air-Rotary Drilling for Geoenvironmental Exploration and Installation of Subsurface Water Quality Monitoring Devices[3]

D 5783 Guide for Use of Direct Rotary Drilling With Water-Based Drilling Fluid for Geoenvironmental Exploration and Installation of Subsurface Water Quality Monitoring Devices[3]

D 5876 Guide for Use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices[3]

D 6032 Test Method for Determining Rock Quality Designation (RQD) of Rock Core[3]

D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling[3]

2.2 *American Petroleum Institute Standard:*

[1] This Practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

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[2] *Annual Book of ASTM Standards,* Vol 04.08.

[3] *Annual Book of ASTM Standards,* Vol 04.09.

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API RP 13B Recommended Practice Standard Procedure for Testing Drilling Fluids[4]

2.3 *NSF Standard:*

NSF 60-1988[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *blind hole*, *n*—borehole that yields no fluid recovery of the drilling fluids.

3.1.2 *casing*, *n*—hollow tubes of steel used to support bore hole walls or where fluid losses must be stopped.

3.1.3 *caving hole*, *n*—borehole whose walls or bottom are unstable and cave or collapse into the drilled borehole.

3.1.4 *core barrels*, *n*—hollow tubes of steel used to collect cores of drilled rock.

3.1.5 *core bits*, *n*—coring bits with surface set or impregnated diamonds in tungsten carbide mix of hardened steel, polycrystalline bits, or tungsten carbide (TC) inserts, mounted on a cylindrical coring bit that does the actual core cutting.

3.1.6 *drill rig*, *n*—includes drilling machine, mast or derrick, circulating pumps, and mounting platform.

3.1.7 *drill rod*, *n*—hollow steel tubes that are connected to the drill bit or core barrel and to the rotary head of the drilling machine.

3.1.8 *drill platform*, *n*—a platform for drilling rig.

3.1.8.1 *Discussion*—The platform may need to be constructed at the drilling site to provide a firm base upon which the drill rig is then placed. Platforms are also constructed in the vicinity of the drill hole for workers to hold equipment, serve as a datum, and to allow safe operations.

3.1.9 *drilling machine*, *n*—includes power unit, hoisting unit, controlled-feed rotary drill head, and water or mud pump.

3.1.10 *overshot*, *n*—a latching mechanism at the end of the hoisting line, specially designed to latch onto or release pilot bit or core barrel assemblies when using *wireline drilling*. **(D 5786)**

3.1.11 *pilot bit assembly*, *n*—designed to lock into the end section of drill rod for *wireline drilling* without sampling. The pilot bit can be either drag, roller cone, or diamond plug types. The bit can be set to protrude from the rod coring bit depending on the formation being drilled. **(D 5786)**

3.1.12 *squeezing hole*, *n*—borehole whose walls move into the drilled opening and squeeze on the drill rods.

3.1.13 *wireline drilling*, *n*—a rotary drilling process using special enlarged inside diameter drilling rods with special latching pilot bits or core barrels raised or lowered inside the rods with a wireline and overshot latching mechanism. **(D 5786)**

3.2 Additional terms are defined in Terminology D 653.

4. Summary of Practice

4.1 *Drilling*:

4.1.1 Drilling is accomplished by circulating a drilling medium through the drill bit while rotating and lowering or advancing the string of drill rods as downward force is applied to a cutting bit. The bit cuts and breaks up the material as it penetrates the formation, and the drilling medium picks up the cuttings generated by the cutting action of the bit. The drilling medium, with cuttings, then flows outward through the annular space between the drill rods and drill hole, and carries the cuttings to the ground surface, thus cleaning the hole. The string of drill rods and bit is advanced downward, deepening the hole as the operation proceeds.

4.1.1.1 Fluid drilling is accomplished by circulating water or a water-based fluid with additives. Additives such as bentonite or polymers are frequently added to water to lubricate and cool the bit and to circulate (transport) cuttings to the surface. Drill fluid can also act to prevent cave or collapse of the drill hole. After the drilling fluid reaches the surface, it flows to a ditch or effluent pipe and into a settling pit where the cuttings settle to the bottom. Cuttings are sometimes run through a shaker to remove the larger particles. From the settling pit, the drilling fluid overflows into the main pit, from which it is picked up by the suction line of the mud pump and recirculated through the drill string.

NOTE 1—The decrease of mud velocity upon entering the mud pit may cause gelling of the mud and prevent cuttings from settling. Agitation of the mud in the pit can remedy the problem.

4.1.1.2 Air drilling is performed where introduction of fluids is undesirable. Air rotary drilling requires use of an air compressor with volume displacement large enough to develop sufficient air velocity to remove cuttings. Cuttings can be collected at the surface in cyclone separators. Sometimes a small amount of water or foam may be added to the air to enhance return of cuttings. Air drilling may not be satisfactory in unconsolidated and cohesionless soils under the ground water table.

4.2 *Coring*:

4.2.1 Coring is the process of recovering cylindrical cores of rock by means of rotating a hollow steel tube (core barrel) equipped with a coring bit. The drilled core is carefully collected in the core barrel as the drilling progresses.

4.3 *Sampling*:

4.3.1 Once the core has been cut and the core barrel is full, the drill rods or overshot assembly are pulled and the core retrieved. Samples are packaged and shipped for testing (see Practices D 5079).

5. Significance and Use

5.1 Rock cores are samples of record of the existing subsurface conditions at given borehole locations. The samples are expected to yield significant indications about the geological, physical, and engineering nature of the subsurface for use in the design and construction of an engineered structure. The core samples need to be preserved using specific procedures for a stipulated time (Practices D 5079). The period of storage depends upon the nature and significance of the engineered structure.

5.2 Rock cores always need to be handled such that their properties are not altered in any way due to mechanical damage or changes in ambient conditions of moisture and temperature or other environmental factors.

[4] Available from American Petroleum Institute, 2101 L St. NW, Washington, DC

[5] Available from NSF International, P.O. Box 130140, Ann Arbor, MI 48113–0140.

6. Apparatus

6.1 *General*—Fig. 1 shows the schematic of a typical rock core drill setup **(2)**. Essential components of the drilling equipment include the drilling rig with rotary power, hoisting systems, casing, rods, core barrels, including bits and liners, and pumps with circulating system. In addition, equipment should include necessary tools for hoisting and coupling and uncoupling the drill string and other miscellaneous items such as prefabricated mud pits and racks for rod stacking and layout. Normally, a drilling platform of planking is built up around the drilling site.

6.1.1 Rock coring operations can proceed at high rotation rates. It is imperative the drill rig, rods, and core barrels are straight and have a balanced center of gravity to avoid whipping and resulting damage to cores and expensive bits.

6.2 *Drilling Rig*— The drill rig provides the rotary power and downward (or advance) force or hold-back force on the core barrel to core the rock. The preferred diamond drill coring equipments are designs with hydraulic or gear-driven variable speed hollow spindle rotary drill heads, although some core rigs are manufactured with gear or chain pulldown/retract systems. Precise control over bit pressure can best be accomplished by a variable setting hydraulic pulldown/retract system. Hydraulic systems are often equipped with a detent valve, which allows downfeed (or advance) rate to be set at a certain speed regardless of tool weight or down pressure exerted on the coring bit. Hydraulic feed drill rigs should be supplied with a hydraulic pressure gage that can be related to bit pressures. Deep hole drill rigs should be equipped with hydraulic hold-back control so, if required, the full weight of the drill rods is not exerted on the bit when drilling downward. Diamond drill rigs can apply high rotation rates as high as 1000 rpm as opposed to normal rotary drills operating at 60 to 120 rpm **(3)**. Most diamond core drills are equipped with a mast and powered hoist for hoisting heavy drill strings. A second wireline hoist is helpful for wireline drilling.

6.2.1 The drill machine frame is either skid or truck mounted and should be equipped with a slide base for ease in working around the drill hole. In special cases, the drilling machine may be mounted on a trailer, barge (for overwater drilling), or columns (for underground work). Some drill rigs are designed to be broken down into several pieces for transport into remote areas. The drilling machine may be powered by hydraulics, air, electricity, gas, or diesel. Most surface skid or truck mounted rigs are diesel or gas powered.

6.2.2 Drilling directions are rarely vertical in underground applications, and smaller rigs are frequently equipped with swivel heads to accommodate drilling at angles. Special accommodations must be made for holding and breaking rods when drilling at high angles into crowns of adits. Either top drive drill or column mount machines with hydraulic or pneumatic rod jacks are equipped to handle up holes. For

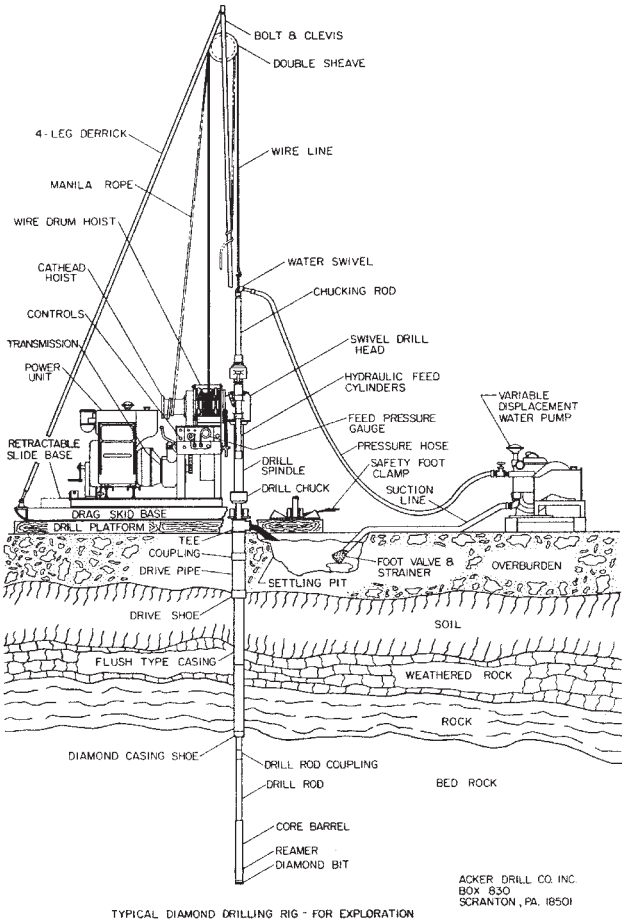


FIG. 1 Schematic of Typical Diamond Core Drill Set-up (2)

confined space drilling operations, drills are column mounted or mounted on small skids. Special power sources may be required for underground work due to air quality considerations. Remote power pack stations usually electric, hydraulic, compressed air, or a combination of the three. Electrically powered hydraulic systems are most common in underground use today

6.3 *Fluid or Air Circulation Systems*:

6.3.1 *Selection of Drill Media*—The two primary methods for circulating drill cuttings are water or water-based fluids or air with or without additives. The predominant method of drilling is water-based fluids. Water-based drilling is effective in a wide range of conditions both above and below the water table. Air drilling is selected when water-sensitive soils such as swelling clays or low density collapsible soils are encountered. Air drilling may also be required above the water table if special testing is required in the unsaturated zone. Air drilling is also convenient in highly fractured igneous rocks and porous formations where water-based fluid losses are unacceptable. The primary functions of the drill fluid are:

6.3.1.1 Remove drill cuttings,

6.3.1.2 Stabilize the borehole,

6.3.1.3 Cool and lubricate the bit,

6.3.1.4 Control fluid loss,

6.3.1.5 Drop cuttings into a settling pit,

6.3.1.6 Facilitate logging of the borehole, and

6.3.1.7 Suspend cuttings in the drill hole during coring.

6.3.1.8 No single drill fluid mixture can satisfy all of the above requirements perfectly. In the sections below, considerations for materials that could be used in drilling medium are given.

6.3.2 The pressure hose conducts the drilling fluid or air from the circulation pump or compressor to the swivel.

6.3.3 The swivel directs the drilling fluid or air to a rotating kelly or drill-rod column.

6.3.4 *Rotary Drilling with Water-based Drilling Fluids*:

6.3.4.1 The mud pit is a reservoir for the drilling fluid, and, if properly designed and used, provides sufficient flow velocity reduction to allow separation of drill cuttings from the fluid before recirculation. The mud pit can be a shallow, open metal tank with baffles or an excavated pit with some type of liner, and designed to prevent loss of drilling fluid. The mud pit can be used as a mixing reservoir for the initial quantity of drilling fluid, and, in some circumstances, for adding water and additives to the drilling fluid as drilling progresses. It may be necessary to have additional storage tanks for preparing fluids while drilling progresses.

6.3.4.2 The suction line, sometimes equipped with a foot valve or strainer, or both, conducts the drilling fluid from the mud pit to the fluid circulation pump.

6.3.4.3 The fluid circulation pump must be able to lift the drilling fluid from the mud pit and move it through the system against variable pumping heads at a flow rate to provide an annular velocity that is adequate to transport drill cuttings out of the drill hole.

6.3.4.4 *Water-based Drilling Fluids*—The four main classes of water-based drilling fluids are: (*1*) clean, fresh water, (*2*) water with clay (bentonite) additives, (*3*) water with polymeric additives, and (*4*) water with both clay and polymer additives. For commonly used materials added to water-based fluid, see Section 7 on Materials.

(*1*) Clean fresh water alone is often not acceptable for core drilling due to poor bit lubrication, erosion due to high velocities required for lifting cuttings, and excessive water loss. In water-sensitive soils, it is desirable to use drill additives to form drill hole wall cakes and prevent moisture penetration. In some cases, water may be required for piezometer installations where other fluid additives are not acceptable, but often newer synthetic polymer materials are acceptable for piezometer and well installations.

(*2*) Bentonitic drill muds are often used in rotary drilling applications. The bentonite should be added to water with vigorous mixing and recirculation to ensure uniform properties and to reach a dispersed deflocculated state. For diamond core drilling, low viscosity is usually required due to small clearances. The viscosity of a fluid-mud mixture is related to the solids content and particle shapes and alignments of the additives. During the high speed rotary drilling process, solids have a tendency to spin out and collect inside drill rods. For diamond drilling, low solids content is desirable. If mass is required to balance high hydrostatic pressures, additives such as barite or ilmenite (see 7.1.8) can be added to keep solids contents low.

(*3*) The need for low solids contents and good lubrication properties point to the use of polymer drill fluids. Natural or synthetic polymer fluids are the best additives for diamond core drilling. Polymer chains such as those from guar gum exhibit flow thinning characteristics in high velocity and shear conditions. Polymer fluids can be weighted with salts to balance hydrostatic pressures. Detergents or deflocculating agents can be added to discharge lines to assist in dropping cuttings to maintain fluid properties.

(*4*) Fluid management requires considerable experience for successful drilling and sampling. Important fluid parameters include viscosity and density, and these parameters can be tested to improve fluid properties. Test Method D 4380 and American Petroleum Institute (API) test procedures are available for testing drill fluids. Fluid design can be improved by consultation with manufacturers, suppliers, and by review of literature **(2-8)**. Because of a large number of suppliers, varying grades of drill fluid products, and varying requirements of each project, providing an exact procedure for design and mixing of drill fluids, is impossible.

6.3.5 *Rotary Drilling Using Air As the Circulation Medium*:

6.3.5.1 The air compressor should provide an adequate volume of air, without significant contamination, for removal of cuttings. Air requirements depend upon the drill rod and bit type, character of the material penetrated, depth of drilling below ground water level, and total depth of drilling. Airflow rate requirements are usually based on an annulus upflow (or outflow) air velocity of about 3000 to 4000 ft/min (1000 to 1300 m/min) although air upflow (or outflow) rates of less than 3000 ft/min (1000 m/min) are often adequate for cuttings transport. Special reaming shells may be required to maintain air circulation between the annulus of the hole wall and large diameter drill rods **(9)**. For some geologic conditions, air-blast

erosion may increase the borehole diameter in easily eroded materials such that the 1000 m/min (3000 ft/min) circulation rate may not be appropriate for cuttings transport.

6.3.5.2 Compressed air alone often can transport cuttings from the borehole and cool the bit. Pure air alone does not work well in very moist soils. In moist, clayey matrices, mud rings and bit balling may occur. For some geologic conditions, water injected into the air stream will help control dust or break down "mud rings" that can form on the drill rods. If water is injected, the depth(s) of water injection should be documented. In these cases, adding water and a foaming agent to make a misting mixture is desirable **(3)**. Under other circumstances, for example if the borehole starts to produce water, injection of a foaming agent may be required. The depth at which a foaming agent is added should also be recorded. If water infiltration into the borehole impedes circulation, the use of stiffer foams or slurries may be needed **(3)**. Air drilling may not be satisfactory in unconsolidated or cohesionless soils under the ground water table, and fluid drilling systems may be required.

6.3.5.3 The dust collector conducts air and cuttings from the borehole annulus past the drill rod column to an air cleaning device (cyclone separator).

6.3.5.4 The air cleaning device (cyclone separator) separates cuttings from the air returning from the borehole via the dust collector. A properly sized cyclone separator can remove practically all of the cuttings from the return air. A small quantity of fine particles is usually discharged to the atmosphere with the "cleaned" air. Some air cleaning devices consist of a cyclone separator alone and others use a cyclone separator combined with a power blower and sample collection filters. When foaming agents are used, a cyclone-type cuttings separator is not used and foam discharge is accumulated near the top of the borehole.

6.4 *Hole Diameters*— Selection of hole diameter and core size is the most important consideration when planning a coring program. Most rock coring operations are performed with casings and core barrels whose sizes have been standardized by the Diamond Core Drill Manufacturers Association (DCDMA) **(5,10)**. Table 1 provides a summary of nomenclature used for drill hole sizing. For each size of hole, there is a family of casings, core barrels, bits, casing bits, and drill rods with the same primary letter symbol (A through Z) whose design is compatible. Furthermore, the size steps are such that the next smaller size letter equipment can be used inside the next larger group. This nesting of casings, barrels, and rods allows for tapering or telescoping of a drill hole through difficult formations. Since the core barrel must pass through the casings selected, anticipating the necessity for telescoping the hole is important so a large enough diameter is selected at the start.

NOTE 2—Inclusion of the following tables and use of letter symbols in the foregoing text is not intended to limit the practice to use of DCDMA tools. The table and the text references are included as a convenience to the user since the majority of tools in use do meet the DCDMA dimensional standards. Similar equipment of approximately equal size on the metric standard system is acceptable unless otherwise stipulated by the engineer or geologist.

6.4.1 Core diameter, barrel design, bit design, and drilling method have a direct influence on sample quality. Usually when drilling in delicate formations,larger diameter samples provide higher quality samples. Often, obtaining samples of the weaker seams or joints in the rock is critical to design. A larger diameter core barrel can often reduce shearing stresses imparted to a seam or joint in the core and thus reduce mechanical breakage. For core operations related to most surface drilling project investigations, the minimum core size would correspond to "N" sized borings.

6.4.2 In concrete coring operations, the primary consideration for selecting a core diameter is the maximum size aggregate. For interface shear strength determinations on lift lines, the core diameter should be 2½ to 3 times the maximum size aggregate **(11)**.

6.4.3 In underground hard rock drilling, smaller cores may be used for ease of operation.

6.5 *Casing*—For most coring operations, setting casings in overburden materials will be necessary, especially near the surface to control drill fluid circulation. Typically, water-sensitive soils and loose overburden soils are protected by casings that are set in competent bedrock or to firm seating at an elevation below the water-sensitive formation. The casing used should allow for unobstructed passage of the largest core barrel to be used, and should be free of upsets in inside diameter. A listing of DCDMA casing sizes is shown in Table 1. For rock coring operations, the flush inside diameter "W" series casing is used to allow for use of the matching core barrel. In some cases, flush coupled drive pipe can be used to support the hole. Drive pipe is available in thickness schedules 40, 80, and 160.

6.5.1 Casing and drill rod selection should be based on uphole (or outflow) velocity of the circulation system selected. Uphole (or outflow) velocity should be sufficient to bring up all drill cuttings.

6.5.2 Casing or temporary drill hole support can be accomplished through several methods. One casing advancement technique is to drill incrementally ahead of the casing and then drive the casing to the previous depth. Driven casings should be equipped with a hardened shoe to protect end threads. The inside diameter of the shoe should be flush with the casing inside diameter to avoid hang-ups of the core barrel. In some cases, water-sensitive zones may require cementing for stabilization. Casing can be equipped with diamond casing shoes that allow the casing to be advanced with rotary drilling. The casing shoe should have the same inside diameter as the casing. Casing "shoes" should not be confused with casing "bits" **(10)**. Casing bits are only acceptable for temporary, rotary installation of casing where coring operations are not required, such as temporary installation of a large diameter telescoped casing. Casing "bits" have an inside diameter that is not large enough to pass a core barrel of the same nominal hole size. Hollow-stem augers may be used as casing through overburden soils. Liners may be used inside large diameter casings or augers to increase fluid circulation velocity and optimize cuttings return. If liners are used, they should not be driven and care should be taken to maintain true hole alignment.

6.6 *Drill Rods*—Drill rod selection should be based on consideration of the uphole (or outflow) velocity of the circulating fluids for the circulation system selected. Uphole

TABLE 1 Diamond Core Drill Manufacturers Association Casing Specifications (10)

NOTE 1—W series casing is known as "flush-coupled casing". W series casing has flush inside diameter throughout, while X series casing has upset diameter with coupling inside diameter equal to flush wall inside diameter.

DCDMA Casing Design										
Size	Outside Diameter		Inside Diameter W Series		Inside Diameter X Series		Gallons Per 100 ft	Mass Per ft	Threads Per Inch	
	in.	mm	in.	mm	in.	mm			W series	X series
RW, RX	1.44	36.5	1.20	30.5	1.20	302.0	5.7	1.8	5	8
EW, EX	1.81	46.0	1.50	38.1	1.63	41.3	9.2	2.8	4	8
AW, AX	2.25	57.2	1.91	48.1	2.00	50.8	14.8	3.8	4	8
BW, BX	2.88	73.0	2.38	60.3	2.56	65.1	23.9	7.0	4	8
NW, NX	3.50	88.9	3.00	76.2	3.19	81.0	36.7	8.6	4	8
HW, HX	4.50	114.3	4.00	100.0	4.13	104.8	65.3	11.3	4	5
PW, PX	5.50	139.7	5.00	127.0	5.13	130.2		14.0	3	5
SW, SX	6.63	168.3	6.00	152.4	6.25	158.8		16.0	3	5
UW, UX	7.63	193.7	7.00	177.8	7.19	182.6			2	4
ZW, ZX	8.63	219.1	8.00	203.2	8.19	208.0			2	4

velocity should be sufficient to bring up all drill cuttings. Most drilling operations are done with DCDMA drilling rods conforming to the dimensions given in Table 2. Drill rods are normally constructed of tubular steel and have a flush outside wall diameter. Drill rod sections usually have threaded female connections machined in each end. The rods are connected by either removable or welded pins (in one end) strengthened by addition of material at the inside walls. Some drill rod pins are constructed of high strength steel because the joints are a weak link and are subject to failure. Some larger rods are composed of composite materials to reduce weight. Nonmagnetic rods are available for drill holes requiring use of magnetic surveying equipment.

6.6.1 Tables 3 and 4 lists dimensions of wireline and API drill rods that also can be used. Wireline drill rod dimensions are not standardized and are specific to individual manufacturers. The API internal flush joint rods have upset walls on the outside joint and should not be used in air drilling, as air erosion of the formation could occur at the joints.

6.7 *Conventional Core Barrels*—Many types of core barrels are available. A conventional core barrel is attached to the drilling rods (see 6.6) and the complete set of connected rods and barrel must be removed from the hole at the end of each core run. Torque is applied to the drill rods while the circulating fluid is pumped through the center of the drill rods to the bit. Fluid returns along the annulus between the borehole wall and barrel and drill rods. Conventional barrels are used in smaller drilling operations, such as short underground holes, or when intermittent sampling is to be performed. Most continuous high production coring today is performed with wireline equipment.

6.7.1 Several series of conventional core barrels have standardized dimensions set by the DCDMA **(10)** in North America. Other organizations such as the British Standards Institute have adopted DCDMA size conventions, while others have different standard dimensions such as metric or Swedish (Craelius) **(4)**. The DCDMA WG, WM, WT series of barrels have standard dimensions as shown in Table 5. Most manufacturers make core barrels fitting the dimensional requirements of one of these series, but there may be variation of other design features such as inner liners, bearings, fluid routing, or

TABLE 2 Diamond Core Drill Manufacturers Association Drill Rod Specifications (10)

Drill Rods, W Series Drill Rod									
Rod Type	Outside Diameter		Inside Diameter		Coupling Identification		Mass Per Foot, lbm	Threads Per Inch	Thread Type
	in.	mm	in.	mm	in.	mm			
RW	1.094	27.8	0.719	18.3	0.406	10.3	1.4	4	Regular
EW	1.375	34.9	0.938	22.2	0.437	12.7	2.7	3	Regular
AW	1.750	44.4	1.250	31.0	0.625	15.9	4.2	3	Regular
BW	2.125	54.0	1.500	44.5	0.750	19.0	6.1	3	Regular
NW	2.625	66.7	2.000	57.4	1.38	34.9	7.8	3	Regular
HW	3.500	88.9	3.062	77.8	2.375	60.3	9.5	3	Regular
WJ Series Drill Rod									
AWJ	1.75	44.5	1.43	36.4	0.63	16.1	3.6	5	Taper
BWJ	2.13	54.0	1.81	46.0	0.75	19.3	5.0	5	Taper
NWJ	2.63	66.7	2.25	57.0	1.13	28.8	6.0	4	Taper
KWJ	2.88	73.0	2.44	61.9	1.38	34.9	...	4	Taper
HWJ	3.50	88.9	2.88	73.1	1.75	44.5	...	4	Taper
Old Standard									
E	1.313	33.3	0.844	21.4	0.438	11.1	...	3	Regular
A	1.625	41.3	1.266	28.6	0.563	14.3	...	3	Regular
B	1.906	48.4	1.406	35.7	0.625	15.9	...	5	Regular
N	2.375	60.3	2.000	50.8	1.000	25.4	...	4	Regular

TABLE 3 Wireline Drill Rod Dimensions

Wireline Drill Rods								
Rod Type	Outside Diameter		Inside Diameter		Gallons Per 100 ft	Weight Per lbm	Threads Per Inch	Thread Type
	in.	mm	in.	mm				
AQWL[A]	1.750	44.5	1.375	34.9	7.7	3.3	4	Taper
AXWL[B]	1.813	46.0	1.500	38.1	9.18	2.8	4	Regular
BQWL[A]	2.188	55.6	1.812	46.0	13.4	4.0	3	Taper
BXWL[B]	2.250	57.2	1.906	48.4	14.82	3.8	4	Regular
NQWL[A]	2.750	69.9	2.375	60.3	23.0	5.2	3	Taper
NXWL[B]	2.875	73.0	2.391	60.7	23.30	6.8	3	Regular
HQWL[A]	3.500	88.9	3.062	77.8	38.2	7.7	3	Taper
HXWL[B]	3.500	88.9	3.000	76.2	36.72	8.7	3	Regular
PQWL[A]	4.625	117.5	4.062	103.2	...	...	. . .	. . .
CPWL[B]	4.625	117.5	4.000	101.6	...	...	. . .	. . .

[A]Q Series rods are specific manufacturer's design.
[B]X Series rods are specific manufacturer's design.

TABLE 4 American Petroleum Institute Drill Rod Dimensions (12)

API Tool Joints—Regular External Flush (in.-lb System)				
Type/size	Rod o.d. (in.)	Rod o.d. (mm)	Rod i.d. (in.)	Rod i.d. (mm)
API 2-⅜	3.125	79.4	1	25.4
API 2-⅞	3.75	95.3	1.25	31.8
API 3-½	4.25	108.0	1.5	38.1
API 4	5.25	133.4	1.75	44.5
API 4-½	5.75	146.1	2.25	57.2
API 5 ½	6.75	171.5	2.75	69.9
API 6 ⅝	7.75	196.9	3.5	88.9
API 7 ⅝	8.88	225.6	4.0	101.6.
API 8 ⅝	10.0	25.4	4.75	120.7
API Tool Joints—Regular Internal Flush				
Type/size	Rod o.d. (in.)	Rod o.d. (mm)	Rod i.d. (in.)	Rod i.d. (mm)
API 2-⅜	3.375	85.7	1.75	44.5
API 2-⅞	4.125	104.8	2.125	54.0
API 3-½	4.75	120.7	2.687	68.3
API 4	5.75	146.1	3.25	82.6
API 4-½	6.125	155.6	3.75	95.3

TABLE 5 Approximated Core and Hole Diameters for Core Barrels

[A]Core barrel type/group	Set bit dimension inside diameter = core diameter		Set reaming shell = hole diameter	
	in.	mm	in.	mm
Conventional Core Barrels[B]				
RWT (d)	0.735	18.7	1.175	29.8
EWD_3	0.835	21.2	1.485	37.7
EWG (s.d.), EWM (d)	0.845	21.5	1.485	37.7
EWT (d)	0.905	23.0	1.485	37.7
AWD_3, AWD_4	1.136	28.9	1.890	48.0
AWG (s.d.), AWM (d)	1.185	30.1	1.890	48.0
AWT (d)	1.281	32.5	1.890	48.0
BWD_3, BWD_4	1.615	41.0	2.360	59.9
BWG (s.d.), BWM (d)	1.655	42.0	2.360	59.9
BWT (s.d.)	1.750	44.4	2.360	59.9
NWD_3, NWD_4	2.060	52.3	2.980	75.7
NWG (s.d.), NWM (d)	2.155	54.7	2.980	75.7
NWT (s.d.)	2.313	58.8	2.980	75.7
HWD_3, HWD_4	2.400	61.1	3.650	92.7
HWG (s.d.)	3.000	76.2	3.907	99.2
HWT (s.d.)	3.187	80.9	3.907	99.2
DCDMA Large Diameter—Double-Tube Swivel—Core Barrels				
2¾ × 3⅞	2.690	68.3	3.875	98.4
4 × 5½	3.970	100.8	5.495	139.3
6 × 7¾	5.970	151.6	7.750	196.8
Wireline Core Barrel Systems[C]				
AXWL (joy)	1.016	25.8	1.859	47.2
AQWL	1.065	27.1	1.890	48.0
BXWL	1.437	36.5	2.375	60.3
BQWL	1.432	36.4	2.360	60.0
BQ_3WL	1.313	33.4	2.360	60.0
NXWL	2.000	50.8	2.984	75.8
NQWL	1.875	47.6	2.980	75.7
NQ_3WL	1.75	44.4	2.980	75.7
HXWL	2.400	61.0	3.650	92.7
HQWL	2.500	63.5	3.790	96.3
HQ_3WL	2.375	60.3	3.790	96.3
CPWL	3.345	85.0	4.827	122.6
PQWL	3.345	85.0	4.827	122.6
PQ_3WL	3.25	82.6	4.827	122.6

[A]s = single tube; d = double tube.
[B]Conventional double-tube core barrels are available in either rigid or swivel designs. The swivel design inner barrel is preferred for sampling because it aids in preventing core rotation. In general, smallest core for given hole size results in best recovery in difficult conditions, that is, triple-tube core barrels. Use of double-tube-swivel type barrels with split liners is recommended in geotechnical investigations for best recovery and least sample damage.
[C]Wireline dimensions and designations may vary according to manufacturer.

core extrusion methods. Some manufacturers make core barrels that do not fit dimensional DCDMA standards for core diameters. An example is the" D_3 and D_4" series core barrels shown in Table 5. Use of other nonstandardized core barrels is acceptable if the type of barrel is appropriate for the drilling conditions and the type of barrel used is reported.

6.7.2 For most investigations and when rock types are unknown, it is desirable to specify a swivel type, double tube core barrel with a split inner barrel, or solid inner barrel with split liners (also know as "triple tube"). The barrel should be equivalent to, or better than, "M" series design to reduce fluid exposure. If the formation is poorly lithified, and contains soil-like layers such as shales with interbedded clay seams, a large diameter core barrel may be specified to aid in recovery. These desired components are discussed below.

6.7.3 Core barrels generally come in 5- or 10-ft core run lengths. Ten-foot core runs can be performed with good rock conditions. If soft, friable, or highly fractured formations are encountered, it may be necessary to select barrels with 5-ft core runs to reduce the possibility of blockages and improve core recovery.

6.7.4 Important design components of a conventional core barrel are tube type (triple, double, or single), inner tube rotation (rigid or swivel), core bit type, including fluid discharge locations (internal discharge - contacting core, or face discharge and waterway design), core lifter, and reaming shell.

6.7.5 *Single Tube Core Barrel*—The single tube core barrel

is the simplest in design (see Fig. 2). The core is subjected to drill fluid circulation over its entire length. Once the core in the barrel is broken from parent material, it will rotate with the assembly. These effects break up all but the most competent core **(4, 12)**. Because of fluid exposure and rotational effects, this barrel should not be used to sample weak, friable, and water-sensitive materials. Additional disadvantages of this core barrel include: poor diamond performance of the cutting bit in fractured or friable formations, frequent core blocking, and severe diamond erosion due to re-drilling of broken fragments. This system is only suitable for sampling massive, hard, competent, homogeneous rock or concrete. Due to these disadvantages, this core barrel type is not recommended for routine investigations.

6.7.5.1 In shallow applications, generally less than 5 ft (2 m) competent concrete or soil cement is cored with single tube masonry core barrels with portable drill rigs **(11)**. If there is evidence of excessive core erosion, breakage, or blocking, use of double tube swivel type barrels should be considered.

6.7.6 *Double Tube Core Barrel*—Double tube core barrels contain an inner barrel that protects the core from contact with drill fluid and from erosion or washing from the circulating

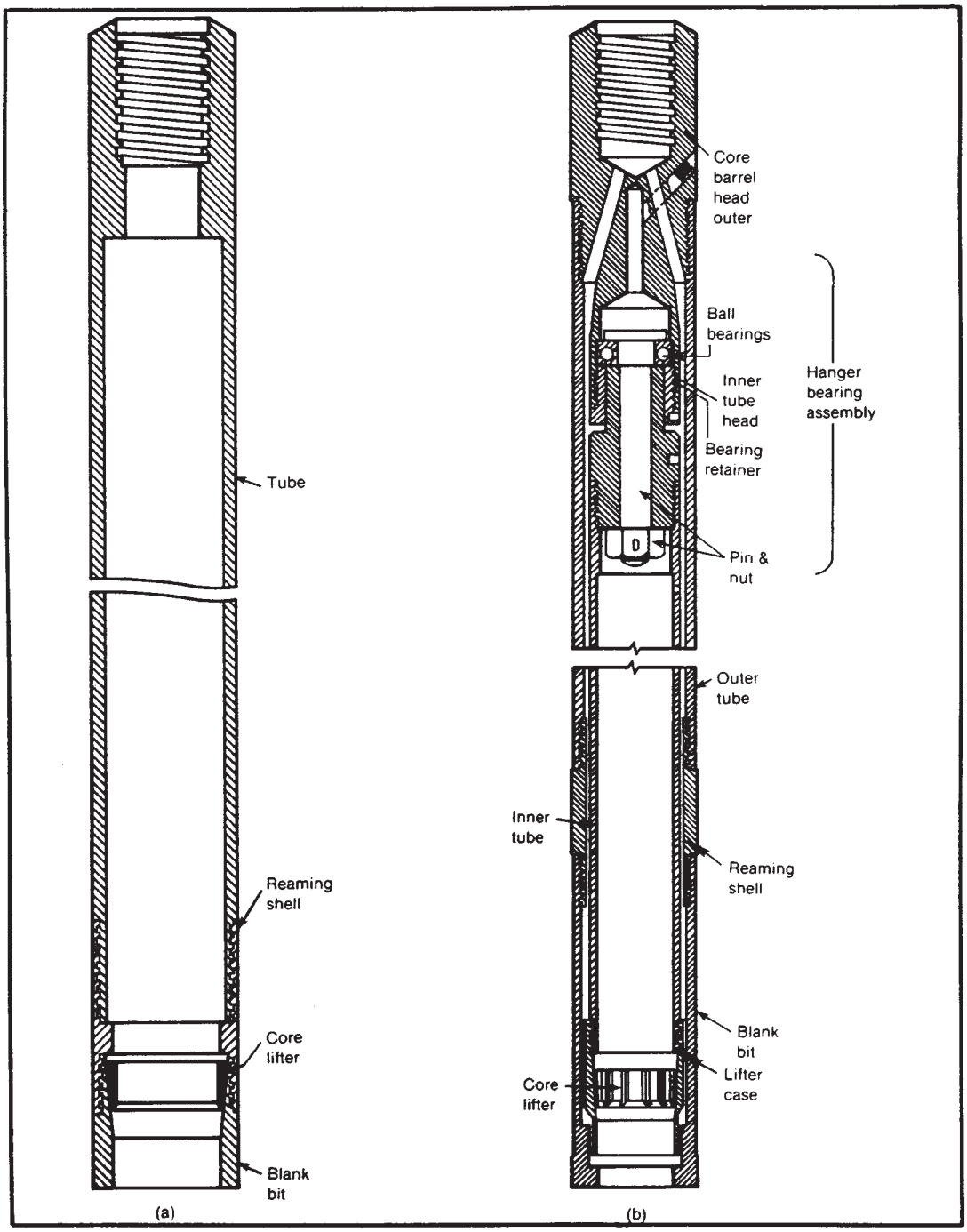


FIG. 2 Diagram of Two Types of Core Barrels: (a) Single Tube and (b) Double Tube

fluid. The bottom of the core may be subjected to fluid exposure depending on the locations of fluid discharge. Some barrel designs have fluid discharge near the lifter, near the bit, or on the bit face (see 6.7.7). The advantage of double tube design is greater protection of the core. Washing erosion is reduced and weaker zones can be recovered.

6.7.6.1 The inner barrel of double tube core barrels may be either solid or split. The barrel may be designed to accept split liners. Barrels accepting liners require a special inside diameter bit gage. Use of a split barrel or inner liners is preferred for easier handling of cores. Sections of the cores containing weak seams are more likely to remain intact. The cores may be rolled onto PVC half rounds. The use of split liners or PVC half rounds aids in placement of core in core boxes and handling of cores that require sealing for moisture preservation. In certain materials, such as expansive shales or blocked high fractured materials, the split liner may spring apart even though it is taped before sampling. In these cases, removing the inner barrel may be difficult. Remedies include use of a shorter core barrel, triple tube design with extruder (see 6.7.7.1), or the solid liner.

6.7.6.2 Double tube core barrels come in two designs, either rigid or swivel type.

(*1*) *Rigid Double Tube Barrel*—This barrel is rarely used in practice today due to limitations listed below. In the rigid barrel design, the inner barrel is fixed and it spins at the same rate as the outer barrel. Rigid tube barrels have fewer working parts, but suffer from similar disadvantages as single tube barrels. Core recovery is poor and diamond wear in friable and fractured formations is excessive. In softer deposits, there will be rotation of broken core, core blockage, and resulting crushing and grinding, which causes excessive bit wear. This type of design is not preferred for routine investigations where rock conditions are not known, as the equipment is only acceptable in hard competent formations.

(*2*) *Swivel Type Double Tube Barrels*—In the swivel type barrel (Fig. 3and Fig. 4show typical barrels) the inner barrel is connected to the drill string through a bearing that allows the inner barrel to remain stationary during coring. The core is completely protected once it enters the liner. This design reduces rock crushing and grinding and resulting blockages. Depending on the fluid discharge point, the core may be exposed to fluids near the bottom of the barrel and there could be erosion of soft or fractured formations.

(*3*) Double tube swivel type core barrels are the best selection for drilling rock of varying hardness and fracture. This type of barrel is typically the minimum requirement when drilling investigations are for engineering structures where varying conditions would be encountered.

6.7.7 *Triple Tube Core Barrels*—The triple tube barrel is essentially a double tube barrel with a liner inside the inner tube. The inner liner is made from either split metal half rounds or tubular acrylic. The use of split liners increases efficiency in handling and logging. If the purpose of the investigation is solely for logging of cores, the use of solid acrylic liners may be acceptable.

6.7.7.1 Many manufacturers offer the triple tube option and barrels are available that also have hydraulic core extrusion systems. These systems help with removing the inner liners by use of a piston in the top of the inner barrel. This feature is especially helpful if split liners are bowed apart by lateral expansion of the core. The extrusion systems allow for simple loading and unloading of liners.

6.7.8 *Conventional Barrel Standardized Designs*—DCDMA standardized barrels come in three designs, WG, WM, and WT series.

6.7.8.1 The “G” series barrels are the most simple in design and have a simple pin threaded bit into which the core lifter is inserted. Due to the simplicity of design, these barrels are the most rugged, with fewer parts and less maintenance. The only disadvantage is that the fluid exits above the lifter and the bottom of the core is exposed to fluids during drilling.

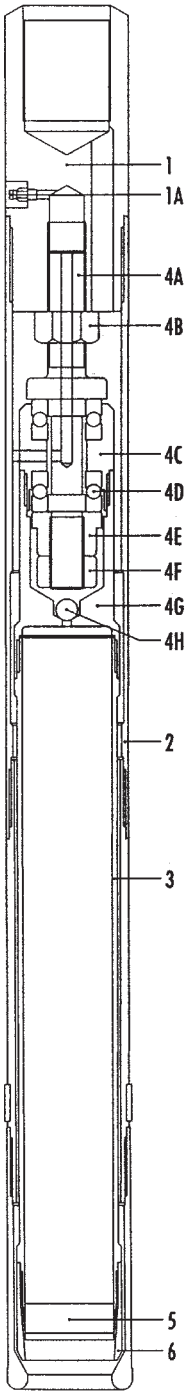
6.7.8.2 The “M” design core barrel is the best available tool for recovering of rock cores even in the most friable and caving stratums. The inner barrel is equipped with a lifter case that extends into the bit shank and therefore reduces exposure of the core to fluid during drilling. The fluid only contacts the core near the crown of the bit, and washing or eroding of the core is minimized. Face discharge bits are also available for almost no core exposure to fluids. The DCDMA “M” designs have been modified by individual manufacturers. Barrels such as the D_4 type barrels are equivalent to “M” design barrels.

6.7.8.3 The “T” series design stands for thin walled or thin kerf. This design provides larger core-to-hole size ratio. This barrel style has a thin kerf and requires fewer diamonds and less torque for drilling. It gives good performance in hard, dense, and friable shattered rock formations **(4)**. This type of core barrel is thin and lightweight and must be handled with care.

6.7.9 *Large Diameter, Double Tube, Swivel Design*—The large diameter conventional core barrel is similar in design to the double tube, swivel type, “WM” design, but with the addition of a ball valve in all the three sizes to control fluid flow. A sludge barrel to catch heavy cuttings is also incorporated on the two larger sizes (Fig. 5). The three sizes standardized by DCDMA are 2¾-in. (69.8 mm) by 3⅞-in. (98.4 mm), 4-in. (101.6 mm) by 5½-in. (139.7 mm), and 6-in. (152.4 mm) by 7¾-in. (196.8 mm). Other sizes such as 4⅝-in. (117.5 mm) by 3-in. (76.2 mm), 5¾-in. (146 mm) by 4-in. (101.6 mm), and 8-in. (203.2 mm) by 5⅞-in. (149.2 mm) have been designed by individual manufacturers. The larger barrels with increased annulus are suitable for larger rotary rig mud pumps and air compressors. Options include either conventional or face discharge bits with either conventional core lifter or spring finger basket retainers. Some core barrel systems can be converted to soil coring operations, but require carbide bit and a projecting cutting shoe. Some large diameter barrels are convertible from conventional to wireline coring operation.

6.8 *Wireline Core Barrels*—Wireline drilling for investigation in rock is a principal drilling system used for deep rock coring applications using surface mounted drill rigs (Fig. 6andFig. 7). In the wireline system, the drill rods are only removed from the hole to replace the coring bit, to free a stuck inner barrel, or to adjust the headspace of the inner barrel. The inner core barrel can be removed and replaced without removing the drill rods, allowing for continuous coring. The drill rods

NWD4-SERIES D4 CORE BARREL



NWD4 SERIES D4 CORE BARREL					
HOLE SIZE	CORE SIZE	OUTER TUBE O.D.	OUTER TUBE I.D.	INNER TUBE O.D.	INNER TUBE I.D.
2.980 in	2.060 in	2.906 in	2.562 in	2.375 in	2.135 in
75.7 mm	51.3 mm	73.8 mm	65.1 mm	60.3 mm	54.2 mm

NWD4 SERIES D4 CORE BARREL ASSEMBLIES
PART DESCRIPTION
Core Barrel Assembly - 5 ft (1.5 m)
Core Barrel Assembly - 10 ft (3.0 m)
Core Barrel Assembly - 5 ft (1.5 m) Split
Core Barrel Assembly - 10 ft (3.0 m) Split

SPARE PARTS FOR NWD4 SERIES D4 CORE B
1. Core Barrel Head, NW Rod
A. Grease Fitting
2. Outer Tube, 5 ft (1.5 m), C.P. Ends
Outer Tube, 10 ft (3.0 m), C.P. Ends
3. Inner Tube, 5 ft (1.5 m), C.P. ID
Inner Tube, 10 ft (3.0 m), C.P. ID
Inner Tube, Split, 5 ft (1.5 m) C.P. ID
Inner Tube, Split, 10 ft (3.0 m) C.P. ID
4. Bearing Assembly
A. Bearing Shaft
B. Lock Nut
C. Bearing Retainer
D. Bearing, Thrust Ball (2 Required)
E. Hex Nut, Flanged
F. Hex Nut, Regular
G. Inner Tube Connector
H. Ball
5. Core Lifter, Skirtless
6. Inner Tube Shoe/Case, C.P. ID
7. Thread Protector Sub

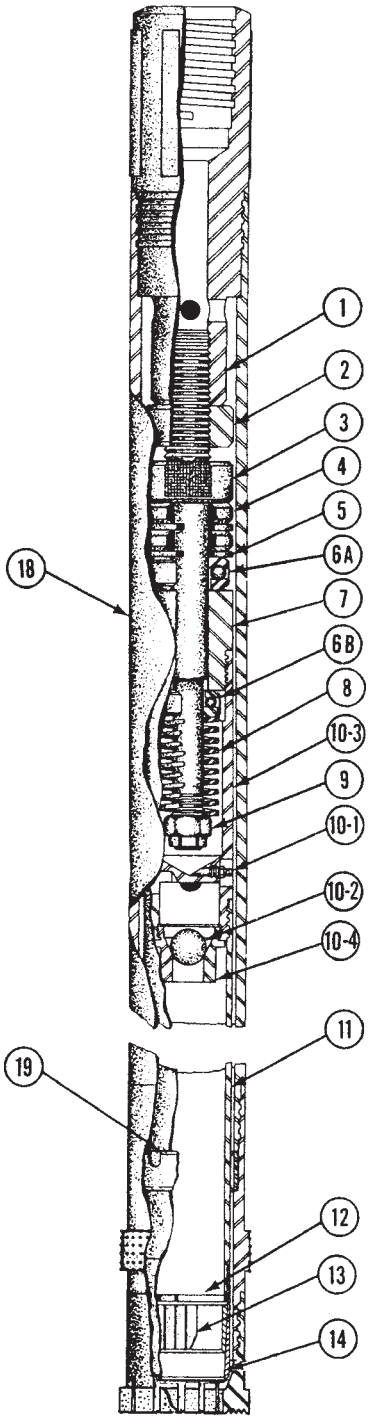
FIG. 3 Typical Double Tube Swivel Type-Conventional Core Barrel

also act as a casing, and f uid is circulated from the bit through the annulus between the drill hole wall and drill rod. Wireline drill rod dimensions are not standardized. Table 3 lists the typical wireline rod sizes and Table 5 lists core barrel sizes of predominant types of wireline equipment available. Other systems such as heavy duty systems with differing dimensions may be available and are acceptable for use if dimensions are reported.

6.8.1 The inner barrel assembly is locked into the lead section of wireline drill rod by means of a retrievable overshot latching mechanism. A latching device locks into a complementary recess in the wall of the leading outer tube such that the outer tube could be rotated without causing the rotation of the inner tube. After the core run, the overshot mechanism is lowered through the rods and latches onto a spearhead on the top of the core barrel assembly and is hoisted to the surface

Double-Tube Swivel Type **Parts List**
STANDARD CONVENTIONAL SYSTEM

NV2" Core Barrel



Item No.	Description	No. Req'd	Unit Weight lbs	kg
1-20	Core Barrel Assy 5 ft	—	76.0	34,5
1-20	Core Barrel Assy10 ft	—	126.0	57,2
-	Core Barrel Assy15 ft **	—	178.0	80,8
1-10	Head Assy	—	19.0	8,6
1	Outer Tube Head	1	8.7	3,9
2	Lock Nut	1	*	*
3	Spindle	1	3.1	1,4
4	Shut Off Valve	2	*	*
5	Valve Adjusting Washer	2	*	*
6A	Ball Thrust Bearing	1	*	*
6B	Hanger Bearing	1	*	*
7	Spindle Bearing	1	1.6	0,7
8	Compression Spring	1	*	*
9	Self Locking Nut	1	*	*
10	Inner Tube Cap Assy	—	3.6	1,6
10-1	Hydraulic Grease Fitting	1	*	*
10-2	Stainless Steel Ball	1	*	*
10-3	Inner Tube Cap	1	3.2	1,5
10-4	Check Valve Body	1	*	*
11	Inner Tube, 5 ft	1	12.3	5,6
11	Inner Tube, 10 ft	1	24.5	11,1
11	Inner Tube, 15 ft	1	38.0	17,2
12	Stop Ring	1	*	*
13	Fluted Core Lifter	1	*	*
14	Core Lifter Case	1	*	*
18	Outer Tube, 5 ft	1	44.0	20,0
18	Outer Tube, 10 ft	1	77.6	35,2
19	Inner Tube Stabilizer	1	*	*
20	Thread Protector (not shown)	1	2.5	1,1

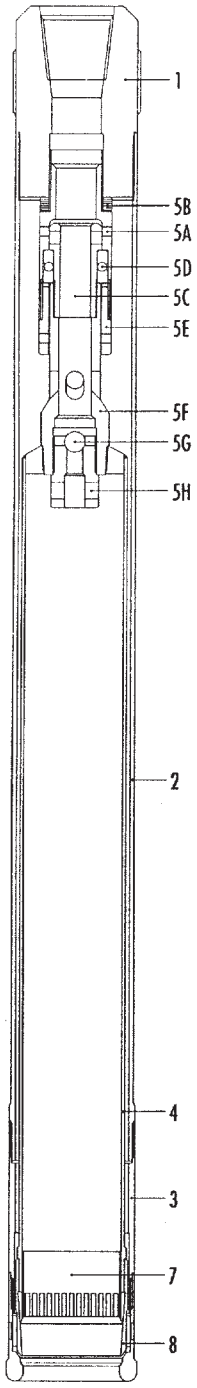
Weighs less than one pound (0,45 kg)
The Core Barrel Assembly 15 foot, consists of one Head Assembly, one each inner Tube 15 foot, Stop Ring, Core Lifter, Core Lifter Case, Outer Tube 5 foot, Outer Tube 10 foot, Thread Protector and two Inner Tube Stabilizers.

FIG. 4 Typical Double Tube Swivel Type-Core Barrel

with cable and wireline winch. The inner tube assembly consists of an inner tube with removable core lifter case and core lifter at one end, and a removable inner tube head swivel bearing, suspension adjustment, and a latching device with a release mechanism at the opposite end. If continuous coring is not required, the retrievable inner core barrel assembly can be replaced with a pilot bit for hole advancement.

6.8.2 Wireline coring systems are designed for long life bits with wide kerf and impregnated or surface set diamonds. Both internal discharge and face discharge bits are available. The

7-3/4 in X 5-7/8 in CORE BARREL



7-3/4 in X 5-7/8 in CORE BARREL					
HOLE SIZE	CORE SIZE	OUTER TUBE O.D.	OUTER TUBE I.D.	INNER TUBE O.D.	INNER TUBE I.D.
8.000 in	5.875 in	7.500 in	7.000 in	6.500 in	6.125 in
203.2 mm	149.2 mm	190.5 mm	177.8 mm	165.1 mm	155.6 mm

7-3/4 in X 5-7/8 in CORE BARREL ASSEMBLIES
PART DESCRIPTION
Core Barrel Assembly - 5 ft (1.5 m)
Core Barrel Assembly - 10 ft (3.0 m)
Core Barrell Assembly - 5 ft (1.5 m) Split
Core Barrell Assembly - 10 ft (3.0 m) Split
SPARE PARTS FOR 7-3/4 in X 5-7/8 in CORE
1. Core Barrel Head (4-1/2" A.P.I. Reg.)
2. Outer Tube, 5 ft (1.5 m)
Outer Tube 10 ft (3.0 m)
3. Outer Tube Sub
4. Inner Tube 5 ft (1.5 m), CP, ID
Inner Tube 10 ft (3.0 m), CP, ID
Inner Tube 5 ft (1.5 m) Split
Inenr Tube 10 ft (3.0 m) Split
5. Bearing Assembly
A. Cartridge Cap
B. Shim, Set
C. Bearing Retainer
D. Bearing, Thrust, Ball
E. Cartridge Plug
F. Inner Tube Plug
G. Ball
H. Pressure Relief Plug
6. Inner Tube Connector, Split
7. Core Lifter, HF
8. Inner Tube Shoe, BW
9. Thread Protector Sub
10. Tube Clamp, 7-1/2 in (190.5 mm) (outer)
OPTIONAL EQUIPME
11. Core Lifter, Skirtless, HF

Note: If you do not see the item or size you are looking for, please contact you for additional product information.

FIG. 5 Typical Large Diameter Series —Conventional Core Barrel

inner core barrel has a dual shut-off valve that stops fluid circulation to alert the driller of a core blockage.

6.8.3 The advantages of wireline drilling are:

6.8.3.1 Significant reduction in rod handling time compared with conventional core barrels.

6.8.3.2 Increased coring bit life with decreased diamond loss.

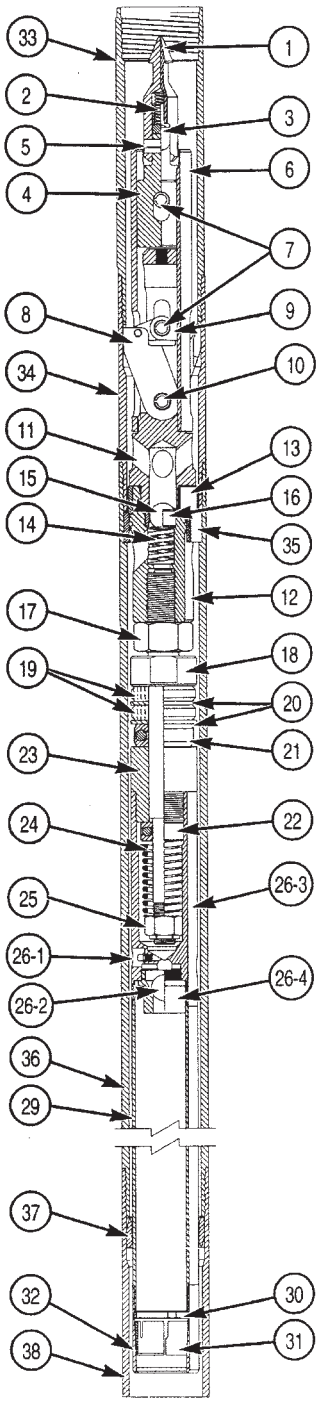
6.8.3.3 High core recovery.

6.8.3.4 Caving is reduced and rods aid to stabilize the drill hole walls.

6.8.3.5 Rods are flush or near flush both inside and outside and can be used as a temporary casing.

6.8.3.6 Various in-hole instrumentation packages can be sent through the end of the drill string to test the bottom of the hole. Wireline packer systems can be used for pressure water test (Test Method D 4630).

NQ Core Barrel



Item No.	Description	No. Req'd	Unit Weight lbs	kg
1-38	Core Barrel Assy 5 ft	—	-	-
1-38	Core Barrel Assy 10 ft	—	-	-
1-32	Inner Tube Assy 5 ft	—	-	-
1-32	Inner Tube Assy 10 ft	—	-	-
1-28	Head Assy	—	-	-
1	Spearhead Point	1	*	*
2	Compression Spring	1	*	*
3	Detent Plunger	1	*	*
4	Spearhead Base	1	1.3	0,6
5	Spirol Pin, 7/16" x 1"	1	*	*
6	Retracting Case	1	-	-
7	Spring Pin, 1/2" x 2"	2	*	*
8	Latch	2	*	*
9	Link	2	*	*
10	Spring Pin, 1/2" x 1-1/2"	1	*	*
11	Upper Latch Body	1	2.9	1,3
12	Lower Latch Body	1	3.1	1,4
13	Landing Shoulder	1	-	-
14	Dry Hole Spring, Light (not installed)			
15	Stainless Steel Ball, 22 mm (not installed)			
16	Bushing (not installed)			
17	Hex Nut	1	*	*
18	Spindle	1	3.1	1,4
19	Shut Off Valve, Hard	2	*	*
20	Valve Adjusting Washer	2	*	*
21	Thrust Bearing	1	*	*
22	Hanger Bearing	1	*	*
23	Spindle Bearing	1	1.6	0,7
24	Compression Spring	1	*	*
25	Self Locking Nut	1	*	*
26	Inner Tube Cap Assy (consists of 26-1, -2, -3, -4)	—	3.3	1,5
26-1	Hydraulic Grease Fitting	1	*	*
26-2	Stainless Steel Ball, 22 mm	1	*	*
26-3	Inner Tube Cap	1	2.9	1,3
26-4	Check Valve Body	1	*	*
27	Landing Indicator Bushing (not installed)			
28	Dry Hole Spring, Medium (not installed)			
29	Inner Tube, 5 ft	1	12.3	5,6
29	Inner Tube, 10 ft	1	24.5	11,1
30	Stop Ring	1	*	*
31	Core Lifter	1	*	*
32	Core Lifter Case	1	1.6	0,7
33	Full-Hole H/T Tanged Locking Coupling**	1	5.9	2,7
34	Adapter Coupling	1	3.2	1,5
35	Landing Ring	1	*	*
36	Outer Tube, 5 ft	1	44.0	20,0
36	Outer Tube, 10 ft	1	77.6	35,2
37	Inner Tube Stabilizer	1	*	*
38	Thread Protector	1	2.5	1,1

Weighs less than 1.0 lb (0,45 kg)
"H/T" means "Heat-Treated"

FIG. 6 Typical Wireline Rock Coring System

6.8.3.7 Two inner barrel assemblies can be used for maximum productivity in continuous coring operations.

6.8.4 The disadvantages of wireline drilling are:

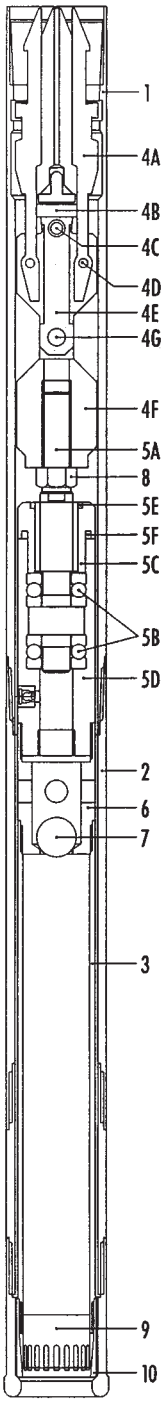
6.8.4.1 Equipment is more expensive than conventional equipment, and

6.8.4.2 Wireline systems are complicated and operations require additional training.

6.8.5 Table 5 lists common wireline core barrel sizes. They are available in “A” through “P” size. When rock samples are to be obtained for testing of engineering properties,“ H” size is recommended, “N” is minimum, and “P” size will result in better recovery of difficult formations.

6.8.6 The wireline core barrel is essentially a double tube swivel type core barrel. Since the core lifter is part of the inner barrel assembly, exposure of the core to drill f uid is minimized similar to “M” style conventional core barrels. Most systems can be adjusted for front discharge (inside above bit gage) or face discharge.

NXB WIRELINE CORE BARREL



NXB WIRELINE CORE BARREL					
HOLE SIZE	CORE SIZE	OUTER TUBE O.D.	OUTER TUBE I.D.	INNER TUBE O.D.	INNER TUBE I.D.
2.980 in	1.875 in	2.875 in	2.391 in	2.200 in	1.969 in
75.7 mm	47.6 mm	73.0 mm	60.7 mm	55.9 mm	50.0 mm

NXB WIRELINE CORE BARREL ASSEMBLIES
PART DESCRIPTION
Core Barrel Assembly - 5 ft Complete (1.5 m)
Core Barrel Assembly - 10 ft Complete (3.0 m)
Core Barrel Assembly 5 ft (1.5 m) Split
Core Barrel Assembly 10 ft (3.0 m) Split

SPARE PARTS FOR NXB WIRELINE CORE BA
1. Locking Coupling
Locking Coupling, NQ Box Thread
2. Outer Tube 5 ft (1.5 m)
Outer Tube 10 ft (3.0 m)
3. Inner Tube 5 ft (1.5 m)
Inner Tube 10 ft (3.0 m)
Inner Tube, Split, 5 ft (1.5 m)
Inner Tube, Split, 10 ft (3.0 m)
4. Quad-Latch Pivot Head Assembly
A. Quad latch (4 required)
B. Spring - Blue (Top)
C. Spring - Red (Bottom)
D. Pin (4 required)
E. Head Base (Top)
F. Head Base (Bottom)
G. Pin
5. Bearing Assembly
A. Bearing Shaft
B. Bearing (2 required)
C. Bearing Retainer
D. Housing w/Zerk & cap
E. O-Ring
F. O-Ring
6. Inner Tube Connector
InnerTube Connection, Split
7. Ball
8. Nut
9. Core Lifter, Skirtless, Buttress
10. Inner Tube Shoe/Case, CP

FIG. 7 Typical Wireline Rock Coring System

6.8.7 Most manufacturers offer a triple tube barrel, which is preferred for most operations. The third tube is a split inner liner that facilitates sample handling.

6.8.8 Some wireline barrels systems are convertible from soil sampling to rock sampling operation. These barrels are equipped with soil barrels that can lead in front of the core bit. Some systems are equipped with spring loaded soil core barrels. These systems are advantageous when soils or soft rock are encountered.

6.9 *Core Barrel Bits*— One of the most important equipment decisions in rock core drilling is the bit selection. Both the bit and reaming shell act together to cut the hole. There are many bit design factors involved when selecting the proper bit for good core recovery. Factors included in selection are:

6.9.1 Diamond type—source,

6.9.2 Diamond matrix (surface set or impregnated),

6.9.3 Rock hardness, grain size, and formation,

6.9.4 Drill power, and

6.9.5 Barrel type.

6.9.6 Bit selection is a trial and error process that may require several iterations. After a bit is selected, it is tried with the drill to be used. Penetration rate and bit life should be evaluated and a different design selected if necessary.

6.9.7 Important features of diamond drilling bits include crown shape, diamonds, and waterways. The crown is the end of the bit that contains a kerf equipped with cutting media. A round or semi-round kerf is frequently used with conventional core barrels. Flat, stepped, or v-ring designs are often used with wireline drilling with impregnated bits.

6.9.8 Important features of the diamonds themselves are the size, quality, quantity, setting, and matrix quality **(4)**. Surface set bits are used in most conventional coring operations. The size of diamonds is expressed in equivalent stones per carat (SPC). The typical SPC range is from 20 to 100 for surface set bits, with the f ner stones being used for harder rock matrixes. Larger diamonds are used for softer, more friable formations. Impregnated bits are fragments mixed with metal and pressed and sintered into the bit. The diamond fragments are throughout the crown and the bit is, in a sense, self-sharpening. Impregnated bits are used in more severe drilling conditions and in wireline drilling where long life is essential.

6.9.9 Waterway design is also an important aspect of bit selection. Surface channel routing of the f uids from inside gage to outside is typical in conventional and wireline drilling for most crystalline rocks that are not sensitive to f uid erosion. Face discharge bits should be used for soft friable formations where f uid erosion is detrimental. Step-face discharge conf gurations further inhibit core erosion. Special f ush discharge air ports are used with air and air-foam drilling operations. The softer the matrix, the larger the waterways must be to avoid blocking and plugging.

6.9.10 For very soft materials, it is not necessary to use diamonds as the cutting media. Diamonds can often be replaced with tungsten carbide or polycrystalline inserts for coring soft materials. Carbide and polycrystalline bits often have cutting teeth arranged in a sawtooth fashion. Polycrystalline bits (diamond grown to tungsten carbide substrates that are soldered or furnaced onto the crown) have replaced natural diamonds for coring in some softer sedimentary formations. Some core barrels, such as the large diameter series, can readily be changed from diamond rock coring to carbide soil sampling modes.

6.9.11 Manufacturers are an excellent resource to assist in selecting the matrix and style of bit, depending on rock conditions, drill power, and barrel type. Most manufacturers have a method for rating diamond matrices and bits into different series and groups **(3)**. DCDMA has selection codes for impregnated bits **(10)**. Manufacturers should be consulted for initial recommendations for your specif c drilling conditions, and as the project progresses, they can work with the driller to ref ne designs and drilling technique. Based on wear patterns, it may be possible to switch series or design to optimize results. In hard competent formations, bit selection may be drill rig-dependent (rotation rate and advance forces). It is beyond the scope of this practice to address drill bit selection. In addition to manufacturers' advice, there are several useful references to provide information in evaluating causes for wear **(3, 4, 10)**.

6.9.12 Most diamond bits have salvage value and should be returned to manufacturers for credit toward future purchases.

6.10 *Reaming Shells*— The reaming shell is a subassembly of a row or strip of material placed on the outside of the core barrel for some distance behind the core bit. It is designed to ream and enlarge the hole to a f nal diameter and must allow for adequate f uid circulation to the surface. The shell also acts as a collar or centralizer for the barrel. Manufacturers should be consulted for appropriate reaming shell designs for the formations to be drilled. They may be surface set with diamond bits, impregnated with diamond particles, inserted with tungsten carbide strips or slugs, hard faced with various types of hard surfacing materials, or furnished blank, as appropriate to the formation being cored.

6.11 *Core Lifters*— Core lifters are used to break the core from parent material at the end of a core run. As coring progresses, the lifter f oats up in its beveled recessed slot in the shoe. After an increment of core has been drilled that corresponds to core barrel length, the rotation is stopped. Fluid is circulated until cuttings are cleared, and then the drill pipe is steadily retracted. The lifter will slide down the beveled shoe and impart an increasing grip on the core. The core frequently breaks near the base of the hole, and often a snap can be felt in the drill pipe as the core breaks from parent material.

6.11.1 Core lifters of the split ring type, either plain or hard surfaced, are recommended and must be maintained in good condition, along with core lifter cases or inner tubes extensions or inner tube shoes. Basket or f nger type lifters with any necessary adapters may be used with core barrels if directed by the engineer or the geologist.

6.12 *Core Boxes*—These are durable waxed cardboard or wooden boxes with partitioned compartments for storing the core samples (see Practice D 5079).

6.13 *Auxiliary Equipment*—The following auxiliary equipment is typically used for the rock core drilling project:

6.13.1 *Drilling bits*— roller rock bits, drag bits, chopping bits, boulder busters, and f shtail bits;

6.13.2 *Tools*—pipe wrenches, core barrel wrenches, chain tong, strap wrench, watch, RPM counter, lubrication equipment, core splitters, rod wicking, extruders, hand sieves or strainers, and marking and packaging tools;

6.13.3 *Rod holding equipment*—closed pulling ring, open iron ring, knife edge holding dog, holding iron, chucking rods, safety foot clamp, manila rope, bolt, and clevis pins;

6.13.4 *Fluid circulation equipment*—hoses, positive displacement pump along with packing and seals, water swivel, pressure gages, f ow meters, water-level meters, and mud pit; and

6.13.5 *Drill area, platform, and leveling*—cribbing, planking, lumber, saw horses, metal saw horses with chain vise.

7. Reagents and Materials

7.1 *Water-Based Drill Fluid Additives*—Listed below are commonly used additives for water-based drilling f uids. Drilling additives in contact with drinking water aquifers should meet the requirements of NSF 60-1988.

NOTE 3—In some areas, certain types of drill fluid products are not allowed by state and local environmental authorities. Before using any drill fluid product, check with the authorities to determine its acceptability.

7.1.1 Beneficiated bentonite, a primary viscosifier and drill hole sealer, consists of montmorillonite with other naturally occurring minerals and various additives such as guar gum, sodium carbonate, or polyacrylates, or a combination thereof.

7.1.2 Unbeneficiated bentonite, a primary viscosifier and drill hole sealer, consists of montmorillonite with other naturally occurring minerals, but without additives such as guar gum, sodium carbonate, or polyacrylates.

7.1.3 Sodium carbonate powder (soda ash) is used to precipitate calcium carbonate from the drilling fluid water-base before adding other components. An increase in pH will occur with the addition of sodium carbonates. Sodium hydroxides (caustic soda) generally should not be used for this application.

7.1.4 Carboxylmethylcellulose powder (CMC) is sometimes used in a water-based fluid as a viscosifier and as an inhibitor to clay hydration. Some additives to water-based drilling-fluid systems retard clay hydration, thus inhibiting swelling of clays on the drill hole wall and "balling" or "smearing" of the bit.

7.1.5 Potassium chloride (muriated potash) or diammonium phosphate can be used as an inhibitor to clay hydration.

7.1.6 Polyacrylamide, a primary viscosifier and clay hydration inhibitor, is a polymer mixed with water to create a drilling fluid.

7.1.7 Guar gum, a primary viscosifier, drill-hole sealer, and hydration inhibitor, is a starch mixed with a water base. The water must be neutral to slightly acidic and hydrochloric acid is sometimes used to pre-treat the water base. Guar gum will degrade with time, but various chemicals can be used to accelerate decomposition.

7.1.8 Barium sulfate increases the density of water-based drilling fluids to help support the borehole wall. It is a naturally occurring high-specific gravity mineral processed to a powder for rotary drilling fluid applications.

7.1.9 Lost-circulation materials are added to the drilling fluid to seal the drill-hole wall when fluids are being lost through large pores, cracks, or joints. These additives usually consist of various coarse textured materials such as shredded paper or plastic, bentonite chips, wood fibers, or mica.

7.1.10 Attapulgite, a primary viscosifier for rotary drilling in high-salinity environments, is a clay mineral drilling-fluid additive.

8. Precautions

8.1 The drilling and sampling equipment shall be complete and in good order. A sufficient amount of drill rods, casings, drill bits, core barrels, core barrel liners, water meters, pumps, and pressure gages shall be in hand before the start of drilling. Measurement devices such as pressure gages and RPM counters shall be functioning in conformance with the manufacturer's specifications.

8.2 The use of fluid or air under high pressure may cause damage to formation materials by fracturing or excessive erosion if drilling conditions are not carefully maintained and monitored. If formation damage is evident and undesirable, other drilling method(s) should be considered.

8.2.1 Fluid pressure should be monitored during drilling. Fluid pressure at the bit should be as low as possible to maintain circulation and to reduce possibility of hydraulic fracturing or excessive erosion of surrounding materials. Normally, injection fluid pressures are fully monitored. Changes in fluid return and circulation pressure may indicate occurrence of excessive erosion, formation fluid loss, core blockage, or formation fracturing. Any abrupt changes or anomalies in the fluid pressure should be noted and documented, including the depth(s) of occurrence(s).

9. Procedure

9.1 Perform site inspections to determine locations of boreholes, and to select disposal sites for waste products during drilling.

9.1.1 Evaluate applicable methods for environmental protection and traffic regulation during core drilling.

9.1.2 Determine site accessibility and availability of water for core drilling operation. Check around the drill site for overhead obstructions or hazards, such as power lines, before raising the mast. A survey of underground and all other utilities is required before drilling to evaluate hazards.

9.2 Fabricate and assemble the drill mounting platform. The platform can take a variety of forms. The type of platform will depend on the terrain encountered, the stipulated depth of the borehole, and the accessibility of the drill site. Specialized mountings such as a barge or stilts or specially constructed towers are necessary to mount platforms for drilling over water.

9.3 For water-based fluid drilling operation, a mud pit is positioned to collect and filter fluid return flow. An initial quantity of drilling fluid is mixed, usually using the mud pit as the primary mixing reservoir.

9.3.1 For air-based circulation systems, the dust collector or cyclone separator is positioned and "sealed" to the ground surface.

9.4 Case any interval of the borehole that penetrates the overburden. This will prevent collapsing of loose materials into the borehole or loss of drill fluid. The casing should extend through the overburden and extend at least 5 ft (1.5 m) into the rock. Casing may be omitted if the borehole will stand open without caving. Deeper casing(s) or nested casing(s) may be required to facilitate adequate drill hole fluid circulation and hole control. Records of casing(s) lengths and depth intervals installed should be maintained and documented.

9.4.1 Surface casings can be installed using a variety of drilling methods. Hollow-stem auder drilling (D 6151) has been used successfully for surface casing and has an added benefit of obtaining samples of the overburden soils.The surface casing is normally backfilled, pressed, or sealed in place wit bentonite or cement, or both.

9.4.2 A datum for measuring drill hole depth should be established and documented. This datum normally consists of a stake driven into stable ground surface, the top of the surface casing, or the drilling deck. If there is possibility for movement of the surface casing, it should not be used as a datum. If the hole is to be later surveyed for elevation, record and report the height of the datum to the ground surface.

9.5 The core barrel is assembled following manufacturers' instructions. Keep core barrels cleaned and lubricated and free from damage, dents, or other defects that might affect core quality. Inspect barrels for wear, clearances, dents, or galls. Check condition of core lifters, fluid passages, relief holes, ball checks, valve rubbers, and inner barrel stabilizers, if present. Assemble and disassemble core barrels with the correct tools for the job.

9.5.1 The inner tube of double tube core barrels must be positioned correctly for proper operation. For both conventional and wireline systems where fluid circulates between the bit and lifter case, check that the proper clearance is maintained for circulation. For wireline systems, this will require engaging the inner barrel while the outer barrel is held vertically. If clearances are not correct, they should be adjusted using the adjustment screw on top of the barrel assembly.

9.5.2 For swivel type inner barrels, inspect the bearing assemblies and confirm the inner barrel is free to rotate smoothly when assembled.

9.6 An initial assembly of lead drill rod and core barrel is attached to the drill mechanism through a spindle or below the drill head, and placed within the top of the surface casing. Hole depth is determined by keeping track of the length of the rod-bit assemblies and comparing its position relative to the established surface datum. Hole depth for increments of drilling, coring, and sampling is recorded on the drill log.

9.7 The drilling-fluid circulation pump or air compressor is activated, causing drilling fluid or air to circulate through the system.

9.8 Drilling fluid or air circulation is initiated and rotation and axial force are applied to the drill rod and bit until drilling progresses to a depth where: (*1*) when the core sample has fully entered the core barrel or blockage is apparent, (*2*) sampling or in-situ testing will be performed, or (*3*) the length of the drill-rod column limits further penetration.

9.8.1 Maintain fluid circulation at a rate suitable for the formation to be drilled. Fluid should be circulated at a rate sufficient to circulate cuttings and cool the bit. Fluid pressures should be monitored during drilling. Avoid drilling at excessive advance rate, which could cause plugging of the bit and core blockage and damage. Changes in fluid return and circulation pressures may indicate occurrence of excessive erosion, formation fluid loss, or formation fracturing. Any abrupt changes or anomalies in fluid pressure should be noted and documented, including depth(s) of occurrence(s).

9.8.2 Maintain air circulation at a rate suitable for the formation, and avoid circulation losses. Add water or foam as necessary to maintain circulation. Zones of low air return or no air return should be documented. Should air-blast erosion occur, depth(s) of the occurrence(s) should be noted and documented. Air is particularly susceptible to blocking off circulation and causing uplift to occur very quickly.

9.8.3 Samples of drill cuttings can be collected for analysis of materials penetrated. If cuttings samples are obtained, the depth(s) and interval(s) should be documented.

9.8.4 The selection of proper rotation rate and down feed (advance) or hold-back pressure depends on many factors. The drilling process is iterative in nature. Diamond drilling in a harder matrix usually requires higher rotation rates and down force pressures. With the correct material and equipment configurations, diamond bit performance is generally optimum at rotation rates of at least 400 rpm or greater. Rates of up to 1000 rpm can be used, depending on the material. However, too high a rate can cause tearing of the formation and core recovery problems. Softer materials with other bits such as polycrystalline, require slower rotation rates. Vibration is extremely important to the drill hole and core quality and must be minimized. Sufficient thrust needs to be transmitted to the bit so that bit elements can cut the rock. The goal is to find the rotation rate and thrust that will result in high quality core and acceptable bit life.

9.8.4.1 Monitor advance or down force pressure, or hold-back pressures, and rotation rates of drill rods during drilling. Observe the penetration rate and drill cuttings as they relate to the geologic strata being penetrated. Document occurrences of any significant abrupt changes and anomalies during drilling.

9.9 Rotation is stopped, the advance or down force pressure is released, and circulation is continued for a short time until the drill cuttings are removed from the borehole annulus. Circulation is stopped and the barrel is rested on the hole bottom to determine hole depth.

9.10 Remove the core barrel and the core from the borehole. Disassemble the core barrel and remove the core.

9.10.1 With double tube barrels, when the barrel is retracted, the core lifter grips the core and the core is normally broken from the base material at the base of the hole. The core lifter is located inside the barrel above the bit typically about 0.1ft. When the core is broken there will be a small pedestal of core left in the hole. As long as the lifter is not slipping, and core is og goods quality, successive recoveries will be close to 100%. If for some reason, the lifter case slips, there will be low recovery recorded and there will be a longer pedestal on the subsequent sampling event. If there appears to be excessive core on the subsequent run, or there is visible evidence of the lifter slipping, these occurrences should be noted. If it is obvious the pedestal was present, the length can be accounted for in determination of recovery (see 10.1) of the previous ren. These occurrences and corrections should be appropriately noted in the drill report.

9.10.2 With single tube core barrels in shallow drilling operations, the core can be broken with a wedge and lifted by wire hoop to the surface.

9.11 Reassemble the core barrel and return it to the borehole. Check for proper barrel conditions as noted in 9.7. The use of two barrels can greatly speed coring operations; as one barrel is cleaned and reassembled the other is in use in coring.

9.12 Drilling depth is increased by attaching an additional drill-rod section(s) to the top of the previously advanced drill-rod column and resuming drilling operations in accordance with 9.8-9.12.

9.13 *Rock Core Handling*—Use of split inner barrel liners greatly increases the efficiency of handling of cores, especially in broken formations. Cores can be transferred into plastic half rounds for logging and sealing. Log, preserve, and place core samples in core boxes in accordance with Practice D 5079.

9.14 *Rock Core Recovery*—Rock core shall be recovered

continuously in the borehole. If recovery drops below 100 % (see Section 10), modify the drilling procedure, that is, adjust the drilling RPM, down feed pressure, the drilling fluid type and flow, or change the type and the size of core barrel or bit used, until core recovery is improved to a level acceptable to the project geologist or project engineer. Minimize mechanical breaks in the core during core drilling as much as possible.

9.14.1 *Poor Recovery*— Stop core drilling when recovery equals or falls below 50 %. If recovering samples is important, select a better core barrel or bit design. In some cases, it may be necessary to attempt soil sampling techniques **(11)**.

9.15 If conditions prevent advance of the drill hole to the stipulated depth, the borehole shall be cemented and redrilled, or reamed and cased, cased and advanced with a smaller size drill bit and core barrel, or abandoned, as directed by the engineer or geologist.

9.16 Prepare boring logs and place the rock sample in core boxes, and mark and pack them in accordance with Practice D 5079.

10. Calculation

10.1 *Calculate Percent Core Recovery*—Determine the rock core recovery as the ratio of length of core recovered to the length drilled, and express as a percent.

$$\text{Percent core recovery} = \frac{\text{Length of the recovered core}}{\text{Total length of the core run}} \times 100 \quad (1)$$

10.2 Calculate Rock Quality Designation (RQD) in accordance with D 6032.

11. Report

11.1 Report the following information:

11.1.1 *Site Conditions*— Site description: description of the site and any unusual circumstances.

11.1.2 *Personnel*—document all personnel at the site during the drilling process, including the driller, helpers, geologist or logger, engineer, and other monitors or visitors.

11.1.3 Weather conditions during drilling.

11.1.4 Working hours, operating times, break-down times, and sampling times. Report any long-term delays in the drilling and installation process.

11.1.5 Report any unusual occurrences that may have happened during the investigation.

11.1.6 *Drilling Methods*:

11.1.6.1 Description of the coring system including type, sizes, core barrels, fluid pump, fluid circulation, and discharge systems. Note intervals of equipment change or drilling method changes and reasons for change.

11.1.6.2 Type, quantities, and drill hole locations of use of additives added to the circulation media. If changes to the circulating medium are made, such as addition of water or conversion to foam, the depth(s) or interval(s) of these changes should be documented.

11.1.6.3 Descriptions of circulation rates, cuttings returns, including quantities, over intervals used. Note the quantity and locations of loss of circulation and probable cause.

11.1.6.4 Descriptions of drilling conditions related to drilling pressures, rotation rates, and general ease of drilling related to subsurface materials encountered. These descriptions can be very general, and should report how the sampling of different materials progressed.

11.1.6.5 *Records of casing installed*—Report type, amount and times of installation. Record water levels (dates and elevation) observed during drilling.

11.1.7 *Sampling*—When core sampling or undisturbed sampling at the base of the boring separate from coring operations, report condition of the base of the boring before sampling and any slough or cuttings present in the recovered sample. Samples of fluid circulation cuttings can be collected for analysis of materials being penetrated. If cuttings samples are taken, the depth(s) and interval(s) should be documented.

11.1.8 *In-situ Testing*:

11.1.8.1 For devices inserted below the base of the drill hole, report the depths below the base of the hole and any unusual conditions during testing.

11.1.8.2 For devices testing or seating at the drill hole wall, report any unusual conditions of the drill hole wall such as inability to seat pressure packers.

11.1.9 *Installations*— A description of completion materials and methods of placement, approximate volumes placed, intervals of placement, methods of confirming placement, and areas of difficulty or unusual occurrences.

11.2 *Boring Logs*— Boring logs should be completed in accordance with Guide D 5434. Some information that the boring log should include is:

11.2.1 Project identification, boring number, location, orientation, date boring began, date boring completed, and drillers' name(s).

11.2.2 Elevation coordinates of the top of the borehole.

11.2.3 Elevation of, or depth to, ground water surface, and any changes in water level, including the dates and times measured.

11.2.4 Elevations or depths at which the drilling fluid returns were lost and amount of return with depth. Report advance or down feed and hold-back pressures, rotation rates of drill rods, fluid/gas pressure, and circulations return during drilling. Report the penetration rate and drill cuttings as they relate to the geologic strata being penetrated. Document any significant abrupt changes and anomalies that occur during drilling.

11.2.5 Size, type, and design of core barrels used. Size, type, and set of core bits and reaming shells used. Size, type, and design and lengths of all casing used, and locations or elevations of casings used. Records of casing(s) lengths and depth intervals installed should be maintained and documented.

11.2.6 Length of each core run and the percentage of core recovery.

11.2.7 Driller's description of the core in each run, if no engineer or geologist was present.

11.2.8 Geologist's or engineer's description of the core recovered in each run. Subsurface description, including dip of strata, jointing, cavities, fissures, core loss, and any other observations made by the geologist, engineer, or the driller that could yield information about the formation encountered during drilling. Depth, thickness, and apparent nature of the filling of each soft seam or cavity encountered. Report the

calculated RQD and D 6032.

11.2.9 Any change in the character of the drilling fluid or drilling fluid return.

11.2.10 Reservoir, tidal, or current information, if the drilling is near or over a body of water.

11.2.11 Drilling time in minutes per foot and down feed gage pressure, when applicable, and the RPM of the drill rods.

11.2.12 Notations of character of drilling, that is, soft, slow, easy, smooth, and others.

12. Precision and Bias

12.1 This practice does not produce numerical data; therefore, a precision and bias statement is not applicable.

12.2 The boring log reflects the subjective opinions of the engineer, the geologist, or the driller. Therefore, the designer must exercise proper prudence when interpreting the boring logs.

12.3 This procedure produces data on rock type and rock quality and recovery factors such as RQD (D 6032) that may reflect the biases of the persons collecting the data. Precision for determination of RQD will be addressed inD 6032.

13. Keywords

13.1 diamond drilling; exploration; rock; rock coring; rock investigations; site characterization

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(**12**) Specifications for Drill Pipe and Drill String, Number 5D-92 and 7G-90, American Petroleum Institute.

(**13**) Core Recovery Techniques for Soft or Poorly Consolidated Materials, prepared for U.S. Bureau of Mines by Woodward Clyde Consultants, Contract J0275003, pp. 136. APL, 1978.

(**14**) API RP 13B "Recommended Practice Standard Procedure for Testing Drilling Fluids," American Petroleum Institute, 2101 L. St., N.W., Washington, DC.

SUMMARY OF CHANGES

This section identifies the principal changes to thispractice that have been incorporated since the last issue. There have been significant changes to the standard. The previous standard was develped in the 1970's. Since that time wireline systmens have gained preference in industry.

(*1*) Expanded Section 1 to add reference to other drilling standards and safety standards.

(*2*) Added logging and transport an storage standards to Section 2.

(*3*) Added a Terminology section and defined drilling-related terms.

(*4*) Added Section 4, Summary of Practice, to describe drilling, coring, and sampling processes.

(*5*) Expanded Apparatus section to discuss the advantages and disadvantages of selecting specific designs. Added sections on fluid and air drilling. Changed and expanded section on wireline drilling methods.

(*6*) Added section on materials and presented major drill fluid materials and additives.

(*7*) Removed section on transportation and storage of rock core and referred to Practice D 5079.

(*8*) Added new Section 8, Precautions, to warn against fracturing and drill hole disturbance.

(*9*) Added section on Calculations and added recovery and RQD. Referred to RQD as a separate D 6032.

(*10*) Changed section Boring Log to Report and referred to Guide D 5434.

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Designation: D5876 – 95 (Reapproved 2005)

Standard Guide for Use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices[1]

This standard is issued under the fixed designation D5876; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers how direct (straight) wireline rotary casing advancement drilling and sampling procedures may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

NOTE 1—The term "direct" with respect to the rotary drilling method of this guide indicates that a water-based drilling fluid or air is injected through a drill-rod column to rotating bit(s) or coring bit. The fluid or air cools the bit(s) and transports cuttings to the surface in the annulus between the drill rod column and the borehole wall.

NOTE 2—This guide does not include all of the procedures for fluid rotary systems which are addressed in a separate guide, Guide D5783.

1.2 The term "casing advancement" is sometimes used to describe rotary wireline drilling because at any time, the center pilot bit or core barrel assemblies may be removed and the large inside diameter drill rods can act as a temporary casing for testing or installation of monitoring devices. This guide addresses casing-advancement equipment in which the drill rod (casing) is advanced by rotary force applied to the bit with application of static downforce to aid in the cutting process.

1.3 This guide includes several forms of rotary wireline drilling configurations. General borehole advancement may be performed without sampling by using a pilot roller cone or drag bit until the desired depth is reached. Alternately, the material may be continuously or incrementally sampled by replacing the pilot bit with a core-barrel assembly designed for coring either rock or soil. Rock coring should be performed in accordance with Practice D2113.

1.4 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.5 Direct rotary wireline drilling methods for geoenvironmental exploration will often involve safety planning, administration, and documentation. This guide does not purport to specifically address exploration and site safety.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1452 Practice for Soil Exploration and Sampling by Auger Borings

D1586 Test Method for Penetration Test (SPT) and Split-Barrel Sampling of Soils

D1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D2113 Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

D3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils

[1] This guide is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Groundwater and Vadose Zone Investigations.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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D4630 Test Method for Determining Transmissivity and Storage Coefficient of Low-Permeability Rocks by In Situ Measurements Using the Constant Head Injection Test
D4631 Test Method for Determining Transmissivity and Storativity of Low Permeability Rocks by In Situ Measurements Using Pressure Pulse Technique
D5088 Practice for Decontamination of Field Equipment Used at Waste Sites
D5092 Practice for Design and Installation of Ground Water Monitoring Wells
D5099 Test Methods for Rubber—Measurement of Processing Properties Using Capillary Rheometry
D5782 Guide for Use of Direct Air-Rotary Drilling for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
D5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices

3. Terminology

3.1 *Definitions*—Terminology used within this guide is in accordance with Terminology D653 with the addition of the following:

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bentonite*—the common name for drilling fluid additives and well-construction products consisting mostly of naturally occurring montmorillonite. Some bentonite products have chemical additives that may affect water-quality analyses.

3.2.2 *bentonite pellets*—roughly spherical- or disk-shaped units of compressed bentonite powder (some pellet manufacturers coat the bentonite with chemicals that may affect the water-quality analysis).

3.2.3 *cleanout depth*—the depth to which the end of the drill string (bit or core barrel cutting end) has reached after an interval of cutting. The cleanout depth (or drilled depth as it is referred to after cleaning out of any sloughed material in the bottom of the borehole) is usually recorded to the nearest 0.1 ft (0.03 m).

3.2.4 *coefficient of uniformity*— $C_u(D)$, the ratio D_{60}/D_{10}, where D_{60} is the particle diameter corresponding to 60 % finer on the cumulative particle-size distribution curve, and D_{10} is the particle diameter corresponding to 10 % finer on the cumulative particle-size distribution curve.

3.2.5 *drill hole*—a cylindrical hole advanced into the subsurface by mechanical means. Also known as a borehole or boring.

3.2.6 *drill string*—the complete rotary drilling assembly under rotation including bit, sampler/core barrel, drill rods, and connector assemblies (subs). The total length of this assembly is used to determine drilling depth by referencing the position of the top of the string to a datum near the ground surface.

3.2.7 *filter pack*—also known as a gravel pack or primary filter pack in the practice of monitoring-well installations. The gravel pack is usually granular material, having selected grain-size characteristics, that is placed between a monitoring device and the borehole wall. The basic purpose of the filter pack or gravel envelope is to act as: a nonclogging filter when the aquifer is not suited to natural development or, as a formation stabilizer when the aquifer is suitable for natural development.

3.2.7.1 *Discussion*—Under most circumstances, a clean, quartz sand or gravel should be used. In some cases, a prepacked screen may be used.

3.2.8 *head space*—on a double- or triple-tube wireline core barrel it is the spacing adjustment made between the pilot-shoe leading edge and the inner kerf of the outer-tube cutting bit. Spacing should be about 1⁄16 in. or roughly, the thickness of a matchbook. (The head-space adjustment is made by removing the inner-barrel assembly, loosening the lock nut on the hanger-bearing shaft and either tightening or loosening the threaded shaft until the inner barrel is moved the necessary distance, up or down, to obtain the correct setting. Reassemble the inner- and outer-barrel assemblies, attach the barrel to the drill rod or a wireline and suspend vertically allowing the inner-barrel assembly to hang freely inside the outer barrel on the inner hanger-bearing assembly. Check the head space. It is imperative that the adjustment is correct to ensure that the inner barrel is free to rotate without contacting the outer barrel. If incorrectly adjusted, the inner barrel will ″hang up” and rotate with the outer barrel as the core is being cut. This will cause the core to break and block entry of core into the inner barrel.)

3.2.9 *grout shoe*—a drillable ″plug” containing a check valve that is positioned within the lowermost section of a casing column. Grout is injected through the check valve to fill the annular space between the casing and the borehole wall or another casing.

3.2.9.1 *Discussion*—The composition of the drillable ″plug” should be known and documented.

3.2.10 *grout packer*—an inflatable or expandable annular plug that is attached to a tremie pipe, usually positioned immediately above the discharge end of the pipe.

3.2.11 *intermittent sampling devices*—usually barrel-type samplers that are driven or pushed below the bottom of a borehole with drill rods or with a wireline system to lower, drive, and retrieve the sampler following completion of an increment of drilling. The user is referred to the following standards relating to suggested sampling methods and procedures: Practice D1452, Test Method D1586, Practice D3550, and Practice D1587.

3.2.12 *in-situ testing devices*—sensors or probes, used for obtaining mechanical- or chemical-test data, that are typically pushed, rotated, or driven below the bottom of a borehole following completion of an increment of drilling. However, some in-situ testing devices (such as electronic pressure transducers, gas-lift samplers, tensiometers, and so forth) may require lowering and setting of the device(s) in preexisting boreholes by means of a suspension line or a string of lowering rods or pipes. Centralizers may be required to correctly position the device(s) in the borehole.

3.2.13 *lead distance*—the mechanically adjusted length or distance that the inner-barrel cutting shoe is set to extend beyond the outer core-barrel cutting bit in order to minimize possible core-erosion damage that can be caused by the circulating drilling-fluid media. Lead distance is checked by vertically suspending the entire core-barrel assembly from a

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wireline or from a section of drill rod so that the inner-barrel can hang freely from the upper inner-barrel swivel assembly. The cutting shoe extension below the outer core-barrel cutting bit can then be checked. The ″stiffer” or more competent the formation to be cored, the less the extension of the inner-barrel cutting shoe is necessary to avoid core erosion.

3.2.14 *overshot*—a latching mechanism located at the end of the hoisting line. It is specially designed to latch onto or release pilot bit or core-barrel assemblies.

3.2.15 *pilot bit assembly*—design to lock into the end section of drill rod for drilling without sampling. The pilot bit can be either drag, roller cone, or diamond plug types. The bit can be set to protrude from the rod coring bit depending on formation conditions.

3.2.16 *sub*—a substitute or adaptor used to connect from one size or type of threaded drill rod or tool connection to another.

3.2.17 *subsurface water-quality monitoring device*— an instrument placed below ground surface to obtain a sample for analyses of the chemical, biological, or radiological characteristics of subsurface pore water or to make in-situ measurements.

3.2.18 *wireline drilling*—a rotary drilling process which uses special enlarged inside diameter drilling rods with special latching pilot bits or core barrels which are raised or lowered inside the rods with a wireline and overshot latching mechanism.

4. Summary of Practice

4.1 Wireline drilling is a rotary drilling process that uses special enlarged inside diameter drilling rods with special latching pilot bits or core barrels which are raised or lowered inside the rods with a wireline and overshot latching mechanism. The bottom section of rod has either a diamond or carbide coring bit at the end and is specially machined to accommodate latching of either pilot bits or core barrels. The overshot mechanism is designed to latch and unlatch bit or barrel assemblies. Bit cutting is accomplished by application of the combination rotary and static down forces to the bit. General drill-hole advancement may be performed without sampling by using either a pilot roller cone or drag bit until the desired depth is reached. Alternately, the material may be continuously or incrementally sampled by replacing the pilot bit with a core-barrel assembly designed for coring either rock or soil.

4.2 The pilot bit or core barrel can be inserted or removed at any time during the drilling process and the large inside diameter rods can act as a temporary casing for testing or installation of monitoring devices.

5. Significance and Use

5.1 Wireline casing advancement may be used in support of geoenvironmental exploration and for installation of subsurface monitoring devices in both unconsolidated and consolidated materials. Use of direct-rotary wireline casing-advancement drilling methods with fluids are applicable to a wide variety of consolidated or unconsolidated materials as long as fluid circulation can be maintained. Wireline casing-advancement drilling offers the advantages of high drilling-penetration rates in a wide variety of materials with the added benefit of the large-diameter drilling rod serving as protective casing. Wireline coring does not require tripping in and out of the hole each time a core is obtained. The drill rods need only be removed when the coring bit is worn or damaged or if the inner core barrel becomes stuck in the outer barrel.

5.1.1 Wireline casing advancers may be adapted for use with circulating air under pressure for sampling water-sensitive materials where fluid exposure may alter the core or in cavernous materials or lost circulation occurs **(1, 2)**.[3] Several advantages of using the air-rotary drilling method over other methods may include the ability to drill rather rapidly through consolidated materials and, in many instances, not require the introduction of drilling fluids to the borehole. Air-rotary drilling techniques are usually employed to advance the borehole when water-sensitive materials (that is, friable sandstones or collapsible soils) may preclude use of water-based rotary-drilling methods. Some disadvantages to air-rotary drilling may include poor borehole integrity in unconsolidated materials when casing is not used and the possible volatilization of contaminants and air-borne dust. Air drilling may not be satisfactory in unconsolidated or cohesionless soils, or both, when drilling below the groundwater table. In some instances, water or foam additives, or both, may be injected into the air stream to improve cuttings-lifting capacity and cuttings return. Use of water or other additives, or both, should be documented. The use of air under high pressures may cause fracturing of the formation materials or extreme erosion of the borehole if drilling pressures and techniques are not carefully maintained and monitored. If borehole damage becomes apparent, other drilling method(s) should be considered.

5.1.2 When air is used as the circulating fluid, the user should consult Refs **(1, 2)** and Guide D5782.

5.2 The application of wireline casing advancement to geoenvironmental exploration may involve sampling of groundwater, soil, or rock; or in-situ or pore-fluid testing; or installation of other casings for subsequent drilling activities in unconsolidated or consolidated materials.

5.3 The wireline drill rod can act as a temporary casing and may be used to facilitate the installation of a monitoring device. The monitoring device may be installed as the drill rod is removed from the drill hole.

NOTE 3—The user may install a monitoring device within the same drill hole wherein sampling or in-situ testing was performed.

5.4 Wireline casing-advancement rotary-drilling methods use fluid or air circulation to lubricate cutting bits and for removal of drill cuttings. In many cases, additives are added to improve circulation, cuttings return, borehole wall stabilization, and sealing of the borehole wall from fluid loss. The use of fluid or air under high pressures may allow for damage to formation materials by fracturing or excessive erosion if drilling conditions are not carefully maintained and monitored. If undesirable formation damage is occurring or evident, other drilling method(s) should be considered.

[3] The boldface numbers given in parentheses refer to a list of references at the end of the text.

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6. Apparatus

6.1 *General*—Direct rotary wireline casing advancement systems and procedures used for geoenvironmental exploration and subsurface water-quality monitoring device installations include direct air or mud-rotary drilling using wireline drill rods. The wireline drill rod has a large inside diameter and is equipped with either a wireline-retrievable center pilot bit for general hole advancement, or a rock- or soil-core barrel for sampling the borehole as it is advanced. Fig. 1(*a* through *d*) shows basic schematics of the components of a wireline-drilling assembly using a rock core-barrel assembly to sample formations as drilling progresses.

6.2 The basic mechanical components of wireline casing-advancement drilling systems include the drill rig with either hollow spindle or top-head drive, drill rods, coring or casing bits, overshot assembly, pilot bit, or core barrel. Water-based fluid circulation systems require drill fluid, mud pit, suction hose, drill fluid circulation pump, pressure hose, and swivel. Air circulation systems require an air compressor, dust collector, air cleaning device, pressure hose, and swivel.

6.2.1 *Drill Rig*—Most top-head drive or hollow-spindle drills are suitable for performing rotary wireline drilling. Rock-coring drills with smooth hydraulic operation and high RPM capability are desirable for rock-coring operations. Rotary table and kelly drills generally are not acceptable for wireline-drilling use due to difficulty or inability to raise and lower wireline assemblies. The drill unit should have the ability to rotate a drill-rod column and apply a controllable axial force on the drill bit appropriate to the drilling and sampling requirements and the geologic conditions.

6.2.2 *Drill Rods*, transfer force and rotation from the drill rig to the bit or core barrel. When rotary drilling is stopped, the large inside diameter wireline drill rod acts can as casing, that is, by preventing against hole collapse—to allow for testing or installation of monitoring devices for hole protection. Drill rods conduct drilling fluid to the bit or core barrel. Individual drill rods (that is, drill stem, drill string, drill pipe) should be straight so they do not contribute to excessive vibrations or “whipping” of the drill-rod column. All threaded connections should be in good repair and not leak significantly at the

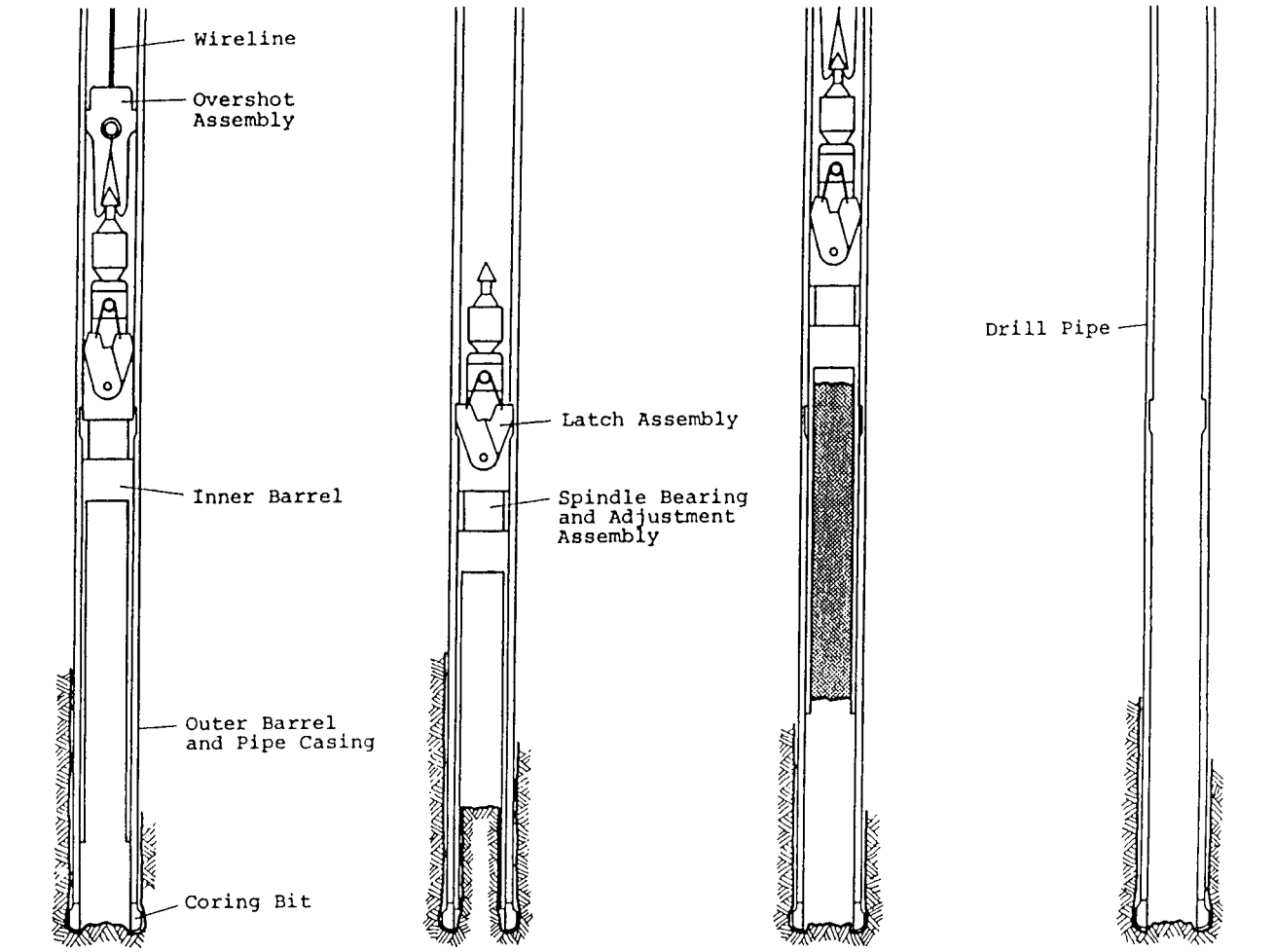

FIG. 1 Schematics of Wire-Line Drilling Assembly

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internal fluid pressure required for drilling. Drill rods should be made up securely by wrench tightening at the threaded joint(s) at all times to prevent rod damage.

6.2.2.1 Wireline drill rod dimensions are not fully standardized. The available sizes depend on the manufacturing sources **(3)**. General hole diameter available from most manufacturers follows the Diamond Core Drill Manufacturers Association (DCDMA) size conventions of A (48-mm), B (59.9-mm), N (75.7-mm), H (96.3-mm), and P (122.6-mm) sizes **(4)**. Inside diameter varies depending on the manufacturer.

NOTE 4—Sizes of casings, casing bits, drill rods, and core barrels are standardized by the DCDMA and the American Petroleum Institute (API). Refer to Ref **(4)** for available sizes and capacities of common use drilling equipments for soil and rock exploration.

6.2.2.2 The wireline lead rod contains shoulders for latching of pilot bits or core barrels. All wireline lead-rod sections are equipped with coring or casing bits. There are many configurations of wireline drilling equipment possible depending on the manufacturer. With rock-coring systems, the wireline lead rod is equipped with a reaming shell to ensure circulation and act as a stabilizer. Some multipurpose systems allow for latching of pilot bit, rock-core barrel, or soil-core barrel to the lead wireline rod section. In most coring operations the lead wireline rod is considered to be the outer barrel in a double- or triple-tube core barrel design (see Practice D2113). The bit is referred to as a core bit.

6.2.2.3 The wireline rod size is usually selected to provide a drill hole of sufficient diameter for the required sampling, testing, or insertion of instrumentation-device components, such as, the screened intake and filter pack and installation devices such as a tremie pipe. The inside diameter of the wireline rod should permit easy insertion and retraction of a sampler or a pipe with a sufficiently large inside diameter to accommodate the placement of completion materials adjacent to the screened intake and riser of an instrumentation device. Coring bits are selected to provide adequate required hole or core diameter, or both. Selection of protective casings, bits, and core barrels is made by considering size requirements listed above combined with: annulus circulation capabilities of drill rod used, and the need for tapering from larger to smaller diameters casings (“nesting” of casing) if difficult drilling conditions occur (lost circulation, zones of contamination, and so forth) requiring these problem zones in the borehole to be cased off.

NOTE 5—Drill rods usually require lubricants on the threads to allow easy threading and unthreading of the drill-rod tool joints. Some lubricants have organic or metallic constituents, or both, that could be interpreted as contaminants if detected in a sample. Various lubricants are available that have components of known chemistry. The effect of drill-rod lubricants on chemical analyses of samples should be considered and documented when using direct-rotary drilling. The same consideration and documentation should be given to lubricants used with water swivels, hoisting swivels, or other devices used near the drilling axis.

6.2.3 The casing bit or core bit provides the material cutting capability. In coring operations, the bit is referred to as a core bit. Rock coring should be performed in accordance with Practice D2113. Soil sampling or coring methods, some of which are listed in 2.2, can also be used to obtain samples. When drilling in a casing-advancement mode using a pilot bit, without sampling, the bit is referred to as a casing bit.

6.2.3.1 Numerous coring or casing bits can be selected depending on the properties of the formation to be drilled or cored. Since it is undesirable to remove the outer bit, design of this bit is extremely important. When coring, particularly in unconsolidated materials, it is important that the bit cuts the material and not merely tears it and pushes it aside. Some bit types successfully used include either carbide inserts coring bits or diamond core bits. In rock-coring operations the kerf design inner gage of bit, matrix cutting capacity, and location of drilling-fluid circulation ports are important. The inner gage of the bit can be selected so that the core is slightly undercut, thereby allowing it to move freely up the inner tube and not cause core blockage. If the core is over cut and air is present in the barrel, consideration should be given to possible alteration of the core that may occur during subsequent sealing and storage of the core obtained. It is beyond the scope of this guide to recommend bit styles. Bit selection can be aided by review of literature **(1, 2, 3, 5, 6)** and consultation with manufacturers.

6.2.3.2 The dimensions of the coring or casing bits often control the maximum diameter of testing or sampling device that can be inserted through the wireline drill rods. As mentioned previously, the bit size is usually selected to provide a drill hole of sufficient diameter for required borehole sampling or testing to be accomplished or for insertion of instrumentation device components such as the screened intake, riser pipe, filter pack, and well-completion installation devices such as a tremie pipe in the borehole.

6.2.4 *Wireline Retrievable Pilot-Bit Assembly*, used when no borehole coring/sampling is desired. The assembly is equipped with a receiver for pickup by the overshot latching assembly. Several pilot-bit styles are available including roller cone and drag bit configurations. Bit selection can be aided by review of literature **(1, 2, 3, 5, 6)** and consultation with manufacturers.

NOTE 6—Bottom-discharge bits are those having drill-fluid circulation discharge ports in direct contact with the base of the hole. If these bits are used to drill loose cohesionless materials, jetting or excessive erosion of the test intervals could occur.

6.2.5 *Overshot*, a latching retrieval assembly that is lowered into the hole with a wireline hoist cable to either retrieve or lower core-barrel inner assemblies or bits equipped with an upper retrieval spear and downhole latching assemblies.

NOTE 7—When lowering a latching bit assembly or retrievable inner-core barrel assembly into a dry hole, a retrievable dry-hole lowering tool should be employed to prevent damaging the outer bit kerf, matrix, or latching assemblies, or combination thereof. Inner tools should not be allowed to free-fall down the drill rod in a dry hole.

6.2.6 *Wireline Core Barrels*, available for obtaining continuous samples of soil or rock. The barrels for use in coring rock vary in design and manufacture from those barrels used for coring unconsolidated materials. The best core recovery in rock usually requires a double- or triple-tube, swivel-type design. The inner tube of the rock core barrel consists of a core-lifter case and core lifter that threads onto the lower end of the inner tube. On the upper end of the inner tube is a removable threaded inner-tube head swivel-bearing assembly

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with an inner-tube latching device and release mechanism. The inner-tube latching device locks into a complementary recess in the wall of the lead outer drill rod such that the drill rod may be rotated while the inner tube remains stationary. The use of split inner tubes or split inner-tube liners inside a solid inner-tube barrel facilitates easier handling, inspection, and removal of the core from the core barrel.

6.2.6.1 Several types of soil core barrels are also available. Most barrels have a cutting shoe that is either flush with the outer tube cutting bit or, it is made to extend past the outer tube core bit. Sample barrels may be of either the solid- or split-tube configuration. Some barrels may also be equipped with either a split tube or solid tube inner liner to minimize exposure of the core to fluids or other materials. Important considerations for optimum sampling results and maximum core recovery are: use of the "correct" lead distance of the inner barrel cutting shoe, using the "optimum" clearance ratio or head space of the cutting shoe, and prevention of inner-barrel rotation. (For optimum core recovery and minimum core damage the user is referred to 3.2.7 and 3.2.13 for making proper lead-distance or head-space adjustments of the inner-barrel cutting shoe.) The lead distance of the cutting shoe ahead of the cutting bit depends on the stiffness of the formations to be sampled. Stiffer materials require less lead distance.

6.2.6.2 The clearance ratio or head space of the cutting shoe with respect to inner barrel should be selected to result in core that fills the barrel without excessive compression of the core due to friction. If the clearance ratio is too great and the core is over cut, core-erosion damage may occur. If core is over cut and air is present in the barrel, alteration of the soil may occur during subsequent packaging, sealing, and storage of the core(s). Use of single-tube split barrels below the water table may expose soil cores to fluids present in the drill rod.

6.2.7 *Pressure Hose*, conducting the drilling fluid from the circulation pump to the swivel.

6.2.8 *Swivel*, directing the drilling fluid to the rotating kelly or drill-rod column.

6.3 *Rotary Wireline Drilling*, with water-based drilling fluids.

6.3.1 *Mud Pit*, a reservoir for the drilling fluid and, if properly designed and utilized, provides sufficient flow velocity reduction to allow separation of drill cuttings from the fluid before recirculation. The mud pit is usually a shallow, open metal tank with baffles; however, for some circumstances, an excavated pit with some type of liner, designed to prevent loss of drilling fluid and to contain potential contaminants that may be present in the cuttings, may be used. The mud pit can be used as a mixing reservoir for the initial quantity of drilling fluid and, in some circumstances, for adding water and additives to the drilling fluid as drilling progresses.

NOTE 8—Some drilling-fluid components must be added to the composite mixture before other components; consequently, an auxiliary mixing reservoir may be required to premix these components with water before adding to the mud pit. All quantities and types of drilling-fluid components and additives used in the composite drilling-fluid mixture should be documented.

6.3.2 *Suction Hose*, sometimes equipped with a foot valve or strainer, or both, conducts the drilling fluid from the mud pit to the drilling-fluid circulation pump.

6.3.3 *Drilling-Fluid Circulation Pump*, having the capability to lift the drilling fluid from the mud pit and move it through the system against variable pumping heads at a flow rate to provide an annular velocity that is adequate to transport drill cuttings out of the drill hole.

6.3.3.1 Fluid pressures at the bit should be as low as necessary to maintain circulation in order to minimize hydraulic fracturing or excessive erosion of the surrounding materials. Fluid pressures should be monitored during drilling. Normally, injection fluid pressures are readily monitored. Changes in fluid return and circulation pressures may indicate occurrence of excessive erosion, formation fluid loss, or formation fracturing. Any abrupt changes or anomalies in the fluid pressures should be duly noted and documented including the depth(s) of occurrence(s).

6.3.4 *Drilling Fluid*, usually consisting of a water-based circulation media and one or more additives that increase viscosity or provide other desirable physical or chemical properties. Principal functions of drilling fluid include: sealing the drill hole wall to minimize loss of drilling fluid, providing a hydraulic pressure against the drill-hole wall to support the open drill hole, removing cuttings generated at the bit, and lubricating and cooling of the bit. Drilling-fluid management requires considerable experience for successful use. Drilling-fluid program design can be aided by review of literature **(1, 3, 4, 5, 6)** and consultation with manufacturers. If changes to the circulating medium are made, the depth(s) or interval(s) of these changes should be documented. Samples of cuttings can be collected for analysis of materials being penetrated. If samples are taken, the depth(s) and interval(s) should be documented.

NOTE 9—Particular attention should be given to the drilling-fluid makeup-water source and the means used to transport the makeup water to the drilling site as potential sources of contamination in the drilling fluid. If the chemical makeup of the water is determined the test results should be documented.

6.3.4.1 Some commonly used additives for water-based drilling fluids are listed in 6.3.4.2-6.3.4.10.

6.3.4.2 Beneficiated bentonite, a primary viscosifier and borehole sealer, consists of montmorillonite with other naturally occurring minerals and various additives such as sodium carbonate or polyacrylates, or both.

6.3.4.3 Unbeneficiated bentonite, a primary viscosifier and drill hole sealer, consists of montmorillonite with other naturally occurring minerals but without additives such as sodium carbonate or polyacrylates.

6.3.4.4 Sodium carbonate powder (soda ash) is used to precipitate calcium carbonate hardness from the drilling fluid water-base before adding other components. An increase in pH will occur with the addition of sodium carbonate. Sodium hydroxide (caustic soda) generally should not be used in this application.

6.3.4.5 Carboxylmethylcellulose powder (CMC) is sometimes used in a water-based fluid as a viscosifier and as an inhibitor to clay hydration.

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NOTE 10—Some additives to water-based drilling-fluid systems retard clay hydration, thus inhibiting swelling of clays on the drill-hole wall and inhibiting "balling" or "smearing" of the bit.

6.3.4.6 Potassium chloride (muriated potash) or diammonium phosphate can be used as an inhibitor to clay hydration.

6.3.4.7 Polyacrylamide, a primary viscosifier and clay-hydration inhibitor, is a polymer that is mixed with water to create a drilling fluid.

6.3.4.8 Barium sulfate increases the density of water-based drilling fluids. It is a naturally occurring high specific gravity mineral processed to a powder for rotary drilling-fluid applications.

6.3.4.9 Lost-circulation materials are used to seal the borehole wall when fluids are being lost through large pores, cracks, or joints. These additives usually consist of various coarse-textured materials such as shredded paper or plastic, bentonite chips, wood fibers, or mica.

6.3.4.10 Attapulgite, a primary viscosifier for rotary drilling in high-salinity environments, is a clay mineral drilling-fluid additive.

NOTE 11—The listing and discussion of the above drilling-fluid additives does not imply general acceptance for geoenvironmental exploration. Some of the additives listed above may impact water-quality analyses. Some readily available, but not as common, drilling-fluid additives, not listed above, could cause significant contamination in a borehole or hydrologic unit. Each additive should be evaluated for each specific application. The types, amounts, and chemical compositions of all additives used should be documented. In addition, a hole log should document the depths where any new additives were introduced. Methods to break revertible fluids should be documented.

If drilling-fluid additives, such as any of those previously mentioned, cannot be tolerated it is recommended that other drilling method(s) be considered by the user.

6.4 *Rotary Wireline Drilling*, using air as the circulation medium:

6.4.1 *Air Compressor*, providing an adequate volume of air, without significant contamination, for removal of cuttings. Air requirements will depend upon the drill rod and bit configuration, the character of the material penetrated, the depth of drilling below groundwater level, and the total depth of drilling. The airflow rate requirements are usually based on an annulus upflow air velocity of about 1000 to 1300 m/min (about 3000 to 4000 ft/min) even though air-upflow rates of less than 1000 m/min are often adequate for cuttings transport. In order to maintain air circulation between the annulus of the hole wall and large-diameter drill rods, special reaming shells may be required **(2)**. For some geologic conditions, air-blast erosion may increase the borehole diameter in easily eroded materials such that 1000 m/min may not be appropriate for cuttings transport. Should air-blast erosion occur, the depth(s) of the occurrence(s) should be noted and documented so that subsequent monitoring equipment installation quality may be evaluated accordingly.

NOTE 12—The quality of compressed air entering the borehole and the quality of air discharged from the borehole and the cyclone separator must be considered. If not adequately filtered, the air produced by most oil-lubricated air compressors inherently introduces a significant quantity of oil into the circulation system. High-efficiency, in-line, air filters are usually required to prevent significant contamination of the borehole.

6.4.2 *Pressure Hose*, conducting the air from the air compressor to the swivel.

6.4.3 *Swivel*, directing the air to the rotating kelly or drill-rod column.

6.4.4 *Dust Collector*, conducting air and cuttings from the borehole annulus past the drill-rod column to an air cleaning device (cyclone separator).

6.4.5 *Air-Cleaning Device*, (cyclone separator) separates cuttings from the air returning from the borehole by means of the dust collector.

NOTE 13—A properly sized cyclone separator can remove practically all of the cuttings from the return air. A small quantity of fine particles, however, are usually discharged to the atmosphere with the "cleaned" air. Some air-cleaning devices consist of a cyclone separator alone; whereas, some utilize a cyclone separator combined with a power blower and sample-collection filters. It is virtually impossible to direct the return "dry" air past the drill rods without some leakage of air and return cuttings. Samples of drill cuttings can be collected for analysis of materials penetrated. If samples are obtained, the depth(s) and interval(s) should be documented.

NOTE 14—Zones of low air return also zones of no air return should be documented. Likewise, the depth(s) of sampled interval(s) and quality of samples obtained should be documented.

NOTE 15—Compressed air alone can often transport cuttings from the borehole and cool the bit. For some geologic conditions, injection of water into the air stream will help control dust or break down "mud rings" that tend to form on the drill rods. If water is injected the depth(s) of water injection should be documented. Under other circumstances, for example, if the borehole starts to produce water, the injection of a foaming agent may be required. The depth when a foaming agent is added should also be recorded. When foaming agents are used, a cyclone-type cuttings separator is not used and foam discharge accumulates near the top of the borehole. When contaminants are encountered during drilling and returning from the borehole at geoenvironmental-exploration sites, special measures should be taken to contain the foam and protect personnel and the environment. Therefore, added water and some available foaming agents could affect water-quality analyses. The need for chemical analysis of added water or foaming agents should be considered and documented.

7. Drilling Procedures

7.1 As a prelude to and throughout the drilling process, stabilize the drill rig and raise the drill-rig mast. Surface casings can be installed using a variety of drilling methods. The surface casing is normally backfilled, pressed, or sealed in place with bentonite or cement, or both. Establish and document a datum for measuring hole depth. This datum normally consists of a stake driven into the stable ground surface, the top of the surface casing, or the drilling deck. If there is a possibility for movement of the surface casing it should not be used as a datum. If the hole is to be later surveyed for elevation, record and report the height of the datum to the ground surface.

7.1.1 For water-based fluid drilling operation, position a mud pit to collect and filter fluid return flow. Mix an initial quantity of drilling fluid, usually using the mud pit as the primary mixing reservoir.

NOTE 16—The need for chemical analysis of samples of each drilling-fluid component and the final mixture should be documented.

7.1.2 For air-based circulation systems, position the dust collector or cyclone separator and "seal" to the ground surface. If air-monitoring operations are performed, consider the prevalent wind direction relative to the exhaust from the drill rig.

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Also, consider the location of the cyclone relative to the rig exhaust since air-quality monitoring will be performed at the cyclone separator discharge point.

NOTE 17—Deeper casing(s) or nested casing(s) may be required to facilitate adequate downhole fluid circulation and hole control. Records of casing(s) lengths and depth intervals installed should be maintained and documented if required. All casing used should be decontaminated according to Practice D5088 prior to use.

NOTE 18—Check above the drilling rig for overhead obstructions or hazards, such as power lines prior to raising the mast. In most cases, it is required to perform a survey of underground and all other utilities prior to drilling to evaluate possible hazards.

7.2 Drilling usually progresses as follows:

7.2.1 Attach an initial assembly of lead drill rod and a bit or core barrel below the top-head drive unit with the bit or drill head placed within the top of the surface casing. Record hole depth by knowing the length of the rod-bit assemblies and comparing its position relative to the established surface datum.

NOTE 19—The drill rig, drilling, hoisting and sampling tools, the rotary gear or chain case, the spindle and all components of the rotary drive above the drilling axis should be cleaned and decontaminated according to Practice D5088 prior to commencing drilling and sampling operations.

7.2.2 Activate the drilling-fluid circulation pump, causing drilling fluid to circulate through the system including the mud pit.

7.2.3 Initiate rotation of the bit and apply axial force to the bit.

7.2.4 Initiate drilling fluid or air circulation and apply rotation and axial force to the bit until drilling progresses to a depth where sampling or in-situ testing will be performed, the length of the drill-rod column limits further penetration, or (when core drilling) the core specimen has fully entered the core barrel or blockage is apparent. Monitor downfeed pressures, fluid/gas pressure, and circulations return during drilling. Observe the ease of drilling during drilling, and drill cuttings as they relate to the geologic strata being penetrated. Document occurrences of any significant abrupt changes and anomalies that occur during drilling.

7.2.4.1 During air drilling operations air-quality monitoring may be required. If air-quality monitoring is performed, document the sampled intervals and air-quality data.

NOTE 20—If circulation is lost and input fluid pressure is allowed to increase, the possibility of fracturing materials and excessive drill hole erosion exists.

NOTE 21—The time required to remove the cuttings from the drill hole will depend mainly upon the pumping rate, the cross-sectional area of the borehole annulus, the borehole depth, the viscosity of the drilling fluid, and the size of the cuttings.

7.2.5 Stop rotation, lift the bit slightly off the bottom of the hole to facilitate drill-cuttings removal, and continue circulation for a short time until the drill cuttings are removed from the borehole annulus. If sampling is to be done, stop circulation and rest the bit on the hole bottom to determine hole depth. Document the hole depth and amount of any caving that occurred.

7.2.6 Increase drilling depth by attaching an additional drill-rod section to the top of the previously advanced drill-rod column and resuming drilling operations according to 7.2.2-7.2.4.

NOTE 22—Drilling rates depend on many factors such as the density or stiffness of unconsolidated material and the existence of cobbles or boulders, the hardness or durability of the rock, or both, the swelling activity of clays or shales encountered in the borehole, and the erosiveness of the wall. Drilling rates can vary from a few mm/min (less than an in./min) to about a m/min (3 ft/min), depending on subsurface conditions. Other factors influencing drilling rates include the weight of the drill string, collar(s) weight and size of drill pipe, and the rig pulldown or holdback pressure. These data as well as any other drilling-rate information should be recorded.

7.2.7 In some cases it may be necessary to remove the pilot bit or core barrel during the drilling process. Remove core barrels when full or there is evidence of core blockage and loss of circulation. Remove pilot bits for sampling events, when worn, or when there is evidence of circulation blockage. When replacing the pilot bit or core barrel note the condition of the base of the boring and any difficulty latching the assembly. If heave is present it may be necessary to lift the drill rod to engage the locking mechanism. When drilling in unconsolidated materials it is recommended to use a vented hoisting plug when removing the inner bit or continued circulation to avoid “heaving,” “piping,” or “sanding in” problems.

7.2.8 In some cases it may be necessary to remove the drill-rod string to change the lead-rod bit. If the string is removed and replaced, check the depth to the base of the boring where the end of the string rests and compare to the cleanout depth to evaluate hole quality. If excessive hole cave or erosion is suspected, casing may be required to protect the borehole wall. As the drilling progresses, note and document drilling procedures such as circulation rates or losses, intervals where equipment is changed, intervals where casing is installed, or drilling method is changed.

7.2.9 *Continuous Sampling with Rock- or Soil-Core Barrels*—Continuous coring can be performed by using core barrels designed for rock or soil coring. Rock coring is performed in accordance with Practice D2113. Soil coring follows similar procedures. When replacing the core barrel, note the elevation of the base of the boring. Record the sampling length and measure core recovery. If blockage of core and circulation loss is noted during the sampling process, the sample should be recovered.

7.2.10 Sampling or in-situ testing can be performed at any depth by interrupting the advance of the bit, stopping the fluid circulation (after cleaning the annulus of cuttings) and removing pilot bit from the drill-rod column. If there are apparent cuttings or evidence of caving, these should be noted as part of the sample taken. Drill-rod removal is not necessary when a sample can be obtained or an in-situ test can be performed through the hollow axis of the drill rods and bit.

7.2.10.1 Some sampling method may be performed through the drill-rod column (see Test Method D1586, Practice D1587, Practice D2113, and Practice D3550). Compare sampling depth to the cleanout depth. Verify the depth-comparison data by first resting the sampler on the bottom of the hole and

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comparing that measurement with the cleanout-depth measurement. This should be done before every sampling or in-situ testing is performed in the hole. If bottom-hole sloughing is apparent from a depth measurement made prior to sampling (determined by comparing the hole-cleanout depth with the sampling depth) it may be necessary to reclean the hole by rotary recirculation. Record the depth of in-situ testing or sampling as well as the depth below the sampler/bit for evaluation of data quality. Decontaminate sampling and testing devices according to Practice D5088 prior to testing.

7.2.10.2 Perform in-situ testing at the base of the drill hole or at shallower depths if the hole is uncased. If in-situ testing is performed at the base of the borehole similar depth, perform checks to accurately document the location of the test.

7.2.10.3 Water testing is frequently performed in consolidated deposits by pulling back on the drill rods and passing inflatable packer(s) with pressure fittings for water injection through the drill rod to test the open borehole wall (see Test Methods D4630 and D4631). Record the depths or intervals of water tests performed.

7.2.10.4 Air drilling may not be satisfactory in unconsolidated or cohesionless soils, or both, under the groundwater table. One problem is “heaving” or “sanding in” of sands during pilot-bit removal. Another problem with wet clays is bit plugging and formation of mud rings which may cause loss of circulation. If the purpose of the drilling is to perform in-situ tests or undisturbed sampling, and soil instability is evident from unbalanced hydrostatic pressure or pilot-bit disturbance, give consideration to using fluids or other casing-advancement methods where disturbance may be more readily controlled.

7.3 When drilling must progress through material suspected of being contaminated, installation of single or multiple (nested) casings may be required to isolate zones of suspected contamination. Isolation casings are usually installed in a predrilled borehole or by using a casing-advancement method. A grout seal is then installed, usually by applying the grout at the bottom of the annulus with the aid of a grout shoe or a grout packer and a tremie pipe. Allow the grout to set before drilling activities are continued. Document complete casing and grouting records, including location(s) of nested casings for the hole.

8. Installation of Monitoring Devices

8.1 Subsurface water-quality monitoring devices are generally installed in boreholes drilled by wireline casing-advancement methods using a three-step procedure. The three steps are: (*1*) drilling, with or without sampling, (*2*) removal of the pilot bit or core barrel and placement of the monitoring device, and (*3*) addition of other materials such as filter packs, seals, and grouts as the drill rods are removed. During placement of filter packs and seals the drill rods may be lifted in increments to be backfilled. Place tremie rods between riser and drill-rod wall to extend past the end of the drill rods for placement increments. The retraction increments depend on the anticipated borehole wall stability. Alternately, if borehole conditions are stable the complete drill-rod column may be removed prior to placement of the monitoring device.

NOTE 23—Document the volumes of emplaced filter packs and seals and compare the results to actual volumes of cuttings return based on hole diameter for evaluation of hole quality.

8.2 Assemble water-quality monitoring devices, with attached fluid conductors (risers), and insert into the borehole with the least possible addition of contaminants. The user is referred to Practice D5092 for monitoring-well installation methods and Practice D5088 for suggested methods of field equipment decontamination.

NOTE 24—If the integrity of the borehole wall will not be compromised by removing the filter cake from the borehole in the vicinity of the screened-intake device the drilling mud should be removed using a well-development procedure as suggested in Practice D5099 prior to inserting a monitoring well or a water-quality monitoring device in the borehole.

8.2.1 Some materials, such as screens and risers, require cleaning or decontamination, or both, at the job site (see Practice D5088).

8.2.2 Prior to installation, store all monitoring-device materials undercover and place upwind and well away from the drill rig and any other sources of contamination such as electrical generators, air compressors, or industrial machinery.

8.2.3 Clean and decontaminate hoisting tools, particularly wire rope and hoisting swivels, according to Practice D5088 before using.

8.3 Select and install filter materials, bentonite pellets, granules and chips and grouts according to specific subsurface-monitoring requirements. Document this information.

NOTE 25—Filter packs for monitoring devices are usually installed in wireline casing-advancement drill holes using a tremie pipe inserted in the annulus formed between the drill rod or borehole wall (when drill rods are removed) and the monitoring device. The minimum annulus between riser pipe and drill rod or borehole wall should be about 1-in. (25.4 mm) completely around the riser pipe. The sizes of wireline drill rod are limited and the riser size may have to be reduced to accommodate placement clearances (see 6.2.2).

8.3.1 Monitoring devices installed in a saturated zone typically have sand-sized filter packs selected on the basis of the grain-size characteristics of the hydrologic unit adjacent to the screened intake. Filter-pack materials are usually selected with a coefficient of uniformity of less than 2.5. Filter packs for vadose-zone monitoring devices may be predominantly slit sized (however, soil-gas monitoring devices should not use silt-sized filter packs but typically use coarse sand or gravel filter packs). These filter materials are often mixed with water of known quality and then inserted through a tremie pipe and tamped into place around the device. The type(s) and volumes of filter materials used and the quality and quantities of mixing water should be documented. In most cases, a centralizer should be used to position the monitoring device in the borehole. The intake device and riser(s) should be suspended above the bottom of the borehole during installation of the filter pack(s), seal(s), and backfill to keep the riser(s) as straight as possible. Care should be taken when adding backfill or filter material(s), or both, so that the materials do not bridge. However, if bridging does occur during the installation procedure, tamping rods or other tamping devices may be used to dislodge the “bridge”.

8.4 Place sealing materials, consisting of either bentonite pellets, chips, or granules, directly above the filter pack.

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NOTE 26—It may be effective, when granular filter packs are used, to install a thin, fine sand, secondary filter either below the annular seal or both above and below the seal. These secondary filters protect both the monitoring-device filter and the seal from intrusion of grout installed above the seal.

8.5 The backfill that is placed above the annular seal is usually a bentonite or cement-base grout.

NOTE 27—Grouts should be designed and installed in consideration of the ambient hydrogeologic conditions. The constituents should be selected according to specific performance requirements and these data documented. Typical grout mixtures are given in Practice D5092 and Test Method D4630.

8.5.1 In most cases, the grout should be pumped into the annulus formed between the borehole wall and the monitoring device(s) riser(s) using a tremie pipe.

NOTE 28—Grouting equipment should be cleaned and decontaminated prior to use according to Practice D5088. Also, the equipment used for grouting should be constructed from materials that do not "leach" significant amounts of contaminants to the grout.

8.5.2 The initial position of the tremie pipe and grouting pressures should be controlled to prevent materials from being jetted into underlying seal(s) and filter(s) (use of a tremie pipe having a plugged bottom and side-discharge ports should be considered to minimize bottom-jetting problems).

8.5.3 In most cases, the grout should be discharged at a depth of approximately 1.5 to 3 m (5 to 10 ft) below the grout surface within the annulus (after the placement of the initial 1.5 to 3 m (5 to 10 ft) of grout above the underlying filter or seal). Additional grout should be discharged at a depth of approximately 1.5 to 3 m (5 to 10 ft) below the grout surface within the annulus. The tremie pipe should be periodically raised as grout is discharged to maintain the appropriate depth below the grout surface.

NOTE 29—The need for chemical analysis of samples of each grout component and chemical analysis of the final mixture should be documented. Also, it should be noted that if cements are used for grouting, they generate hydroxides and thereby, can cause a localized increase in the alkalinity and pH of the surrounding groundwater.

8.5.4 Install the grout from the bottom of the borehole to the top of the borehole so as to displace fluids in the borehole.

9. Development

9.1 Most monitoring-device installations should be developed to remove suspended solids from drilling fluids and disturbance of geologic materials during installation and to improve the hydraulic characteristics of the filter pack and the geologic unit adjacent to the monitoring-device intake. The method(s) selected and time expended to develop the installation and the changes in water quality discharged at the surface should be carefully observed and documented. For suggested well-development methods and techniques the user is referred to Practice D5099.

NOTE 30—Under most circumstances, development should be initiated as soon as possible following completion, however, time should be allowed for setting of grout.

10. Field Report and Project Control

10.1 The field report should include all information identified as necessary and pertinent to the needs of the exploration program. Information normally required for the project include exploration type and execution, drilling equipment and methods used, and subsurface conditions encountered including: groundwater conditions, sampling events, and installations.

10.2 Additional information should be considered as follows:

10.2.1 *Drilling Methods*:

10.2.1.1 Description of the wireline casing-advancer system including type, sizes, pilot bits, core barrels, fluid pump, fluid circulation, and discharge systems. Note and document intervals of equipment change or drilling method changes and reasons for change.

10.2.1.2 Type, quantities, and locations in the borehole of use of additives such as water or foaming agent(s) added to the circulation media.

10.2.1.3 Descriptions of circulation rates and cuttings return, including quantities, over intervals used. Locations and probable cause of loss of circulation in the borehole.

10.2.1.4 Descriptions of drilling conditions related to drilling pressures, rotation rates, and general ease of drilling related to subsurface materials encountered.

10.2.2 *Sampling*—Document conditions of the bottom of the borehole at the base prior to sampling, and report any slough or cuttings present in the recovered sample.

10.2.2.1 Samples of fluid circulation cuttings can be collected for analysis of materials being penetrated. If samples are taken, the depth(s) and interval(s) should be documented.

10.2.3 *In-Situ Testing*:

10.2.3.1 For devices inserted below the bottom of the borehole, document the depths below the bottom of the hole and any unusual conditions during testing.

10.2.3.2 For devices testing or seating at the borehole wall, report any unusual conditions of the borehole wall such as inability to seat borehole packers.

10.2.4 *Installations*—A description of well-completion materials and placement methods, approximate volumes placed, depth-intervals of placement, methods of confirming placement, and areas of difficulty of material placement or unusual occurrences.

11. Keywords

11.1 drilling; geoenvironmental exploration; groundwater; vadose zone; wireline drilling method

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Baroid Industrial Drilling Products
Product Service Line, Halliburton

Dear Sir or Madam:

Baroid Industrial Drilling Products, a product service line of Halliburton, does not knowingly introduce PFAS (per- and polyfluoroalkyl substances) into the following products during the manufacturing process:

Soda Ash
PERFORMATROL® 930 Polymer Fluid system
QUIK-TROL® GOLD Highly Dispersible Filtration Control Additive
QUIK-TROL® GOLD LV Highly Dispersible Low Viscosity Filtration Control Additive
EZ-MUD® GOLD Clay and Shale Stabilizer
SYSTEM FLOC-360® Flocculant
BARA-KADE® DWD Solidification Agent

If you have any further questions, do not hesitate to contact me.

Respectfully submitted,

Shantel Stone

Technical Service Manager

Baroid Industrial Drilling Products

3000 North Sam Houston Pkwy East

Houston, TX 77032

QUIK-TROL® GOLD

Highly Dispersible Filtration Control Additive

Description

QUIK-TROL® GOLD highly dispersible polyanionic cellulosic (PAC) polymer provides ease of mixing and improved filtration control in most water-based drilling fluids. QUIK-TROL GOLD highly dispersible polymer, when added to an AQUAGEL®, QUIK-GEL® or BORE-GEL® slurry, yields a low filtrate drilling fluid system suitable for drilling in water sensitive formations.

Applications/Functions

The use of QUIK-TROL GOLD filtration control additive promotes:

- Filtration control in water-based drilling fluids
- Borehole stability in water sensitive formations
- Minimized rotational torque and circulating pressure
- Improved hole cleaning and core recovery
- Enhanced foam properties to improve cuttings transport in air/foam drilling

Advantages

- Disperses readily in water, even with low shear
- Effective in fresh, salt and brackish water-based drilling fluids
- Efficiently improves filtration control- effective at low concentrations
- Non-fermenting
- Compatible with other Baroid drilling fluid additives
- NSF/ANSI Standard 60 certified

Typical Properties

• Appearance	Off white, granular powder
• pH (1% solution)	6.0 - 8.0

Recommended Treatment

- Using a Venturi Mixer, or into vortex of a high-speed stirrer, add slowly and uniformly to the entire circulating system. See treatment chart below or contact your local Baroid IDP Field Representative for dosing assistance.

Approximate Amounts of QUIK-TROL® GOLD filtration control additive Added to Water-Based Fluids		
lbs/bbl	**lbs/100 gallons**	**kg/m^3**
0.1 – 2.0	**0.25 – 4.75**	**0.3 – 5.7**

QUIK-TROL GOLD additive is designed for Industrial Use Only

Note:
Highly saline waters may require twice as much QUIK-TROL GOLD additive as fresh water. Preferably, QUIK-TROL GOLD additive should be mixed in fresh water before it is added to very salty water.

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Rev. 04/2011 · IDP-132

Because the conditions of use of this product are beyond the seller's control, the product is sold without warranty either express or implied and upon condition that purchaser make its own test to determine the suitability for purchaser's application. Purchaser assumes all risk of use and handling of this product. This product will be replaced if defective in manufacture or packaging or if damaged. Except for such replacement, seller is not liable for any damages caused by this product or its use. The statements and recommendations made herein are believed to be accurate. No guarantee of their accuracy is made, however.

Packaging

QUIK-TROL® GOLD filtration control additive is packaged in 40 pound (18.2 kg) pails and in 20-lb (9.1 kg) plastic cans containing 10 airtight sealed plastic bags. Each bag contains 2-lb (0.91 kg). QUIK-TROL GOLD filtration control additive is also available in 50-lb (22.7 kg) multiwall paper bags.

Availability

QUIK-TROL GOLD filtration control additive can be purchased through any Baroid Industrial Drilling Products Retailer. To locate the Baroid IDP retailer nearest you contact the Customer Service Department in Houston or your area IDP Sales Representative.

Baroid Industrial Drilling Products
Product Service Line, Halliburton
3000 N. Sam Houston Pkwy E.
Houston, TX 77032

Customer Service	(800) 735-6075 Toll Free	(281) 871-4612
Technical Service	(877) 379-7412 Toll Free	(281) 871-4613

SYSTEM FLOC-360®

Flocculant

Description

SYSTEM FLOC-360® polymeric flocculant is used to flocculate clays and shales encountered in drilling operations and facilitate recycling and/or disposal of water-based drilling fluids.

Applications/Functions

- Promotes flocculation and settling of dispersed formation solids in clear fluids and conventional drilling fluids

Advantages

- Can create aggressive water/solids separation
- Can improve clarity of residual fluids after treatment
- Effective in low to moderate concentrations

Typical Properties

Appearance	Amber Liquid
Specific gravity	1.14- 1.18
Freeze point, °F, (°C)	20°F (-7°C)
pH of 1% aqueous solution @ 25°C	5.5-6.5
Viscosity* @ 25°C (*Brookfield LVT, 30 rpm, Spindle #1)	80-120 cP
Solubility in water	100% miscible

Recommended Treatment

For flocculation treatment of fluid systems containing PHPA polymer:

- Recommended treatment concentration is between 1.0 - 2.0 % by volume
- Final product concentration can be influenced by method of introduction and efficiency of reaction created. SYSTEM FLOC-360 should be introduced with agitation to optimize speed and efficiency of reaction.
- Disposal of solids and water phase should always be done in accordance with all applicable federal, state and local regulations.
- Responsible handling and disposal of drilling fluids, water phase effluent and produced solids require that these materials should not be allowed to gain entry into any stream, waterway or body of water. If reusing water phase in fresh drilling fluid, effluent should be treated with bentonite to remove excess SYSTEM FLOC-360 prior to mixing.

Recommended Treatment (continued)

For clean-out of Drilled Shafts using PHPA polymers:

- Using the diameter of the drilled shaft calculate the volume of a 1-2 foot interval of the open hole.
- Add SYSTEM FLOC-360 flocculant at a concentration of 0.5 - 1.0 % by volume and place directly to the bottom of the shaft. Based on calculated volumes, the appropriate amount of SYSTEM FLOC-360 flocculant can be placed in collapsible bags allowing delivery to the bottom of the shaft or material can be tremied into place.
- After proper placement, use of the auger flights to create agitation is necessary for effective dispersion of the SYSTEM FLOC-360 flocculant and incorporation with existing solids.
- Crosslinked polymer and drilled solids can be removed using a vented clean-out bucket

Packaging

SYSTEM FLOC-360™ flocculant is packaged in 55-gallon (208.2 L) non-returnable steel-drums and 5-gallon (18.9 L) resealable plastic containers.

Availability

SYSTEM FLOC-360 flocculant can be purchased through any Baroid Industrial Drilling Products Retailer. To locate the Baroid IDP retailer nearest you contact the Customer Service Department in Houston or your area IDP Sales Representative.

Baroid Industrial Drilling Products
Product Service Line, Halliburton
3000 N. Sam Houston Pkwy E.
Houston, TX 77032

Customer Service	(800) 735-6075 Toll Free	(281) 871-4612
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BENTONITE
Performance Minerals LLC

BARA-KADE®

DWD

Description

BARA-KADE® DWD is a solidification agent designed for use with industrial and drilling waste streams.

Applications/Functions

- Drilling fluids and cuttings solidification agent
- Solidification and stabilization of complex waste water
- Engineered for solidification of fresh water streams, emulsions, and hydrocarbon-based stream

Advantages

- Solidification in minutes after application
- Meets landfill slump test (Paint Filter Test, EPA 9095)
- Meets EPA Liquid Release Test (EPA 9096)
- Forms stable agglomerates and will not release liquids after freezing and reheating to 160°F

Properties

	Typical
Moisture	10
Bulk Density (lbs/ft^3 uncompacted)	58
pH	9.5 - 11

Availability

BARA-KADE® DWD can be purchased through any Bentonite Performance Minerals LLC assigned Reseller. To locate the BPM Reseller nearest you, contact the Customer Service Department in Houston or your area BPM Regional Sales Manager.

Bentonite Performance Minerals LLC
A Halliburton Company
3000 N. Sam Houston Pkwy E.
Houston, TX 77032-3219
www.bentonite.com

Customer Service	(281) 871-7900	**Fax**	(281) 871-7940
Technical Service	(717) 432-3627	**Alternate**	(717) 645-0471

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APPENDIX B.4

PACE ANALYTICAL MOBILE LAB SERVICES FIELD PROCEDURES

Note: The mobile laboratory division of Cascade Drilling was recently purchased by Pace Analytical. The following SOPs are marked as draft as they are in the process of being converted from the Cascade SOP format to the Pace Analytical SOP format.

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1.0 SCOPE AND APPLICATION

The purpose of this procedure is to collect and process samples of bedrock aquifer material which can be subsequently analyzed to determine the concentration of analytes in the pore water within the rock matrix. This is important information in dual porosity bedrock environments where diffusion of contaminants into the rock matrix porosity can result in a large portion of the total contaminant mass residing in the relatively immobile matrix.

The procedures make up a portion of the CORE^{DFN} services that are provided under an exclusive license agreement between Pace Analytical Services and the University of Guelph.

2.0 SUMMARY OF METHOD

Rock cores are retrieved by a drilling team from fractured bedrock aquifers and sampled for volatile organic compounds (VOCs) along with representative samples for physical property analyses including porosity, bulk density, organic carbon content, chloride diffusion coefficient and matrix permeability.

VOC Samples need to be obtained, logged, and crushed into methanol with a minimal loss of volatile analytes.

3.0 INTERFERENCES

3.1 Not applicable to this SOP

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for commonly used terms and definitions.

5.0 HEALTH AND SAFETY

Proper Personal Protective Equipment (PPE) must be used when doing any type of sample collection. This includes at a minimum safety glasses and disposable gloves to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. Additional equipment including but not limited to boots, visors, aprons or hardhats may be necessary at some locations and should be used as needed.

If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed.

Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal; however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate Pace employee.

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The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

5.1 PPE Required

5.1.1 Nitrile gloves

5.1.2 Rubber gloves

5.1.3 Steel toe boots

5.1.4 Safety glasses

5.1.5 Hard Hat

5.1.6 Eye-wash bottle (1-2)

5.1.7 First aid kit

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Sample Collection: rock samples are collected into pre-weighed 40 mL vials with methanol. Details regarding this procedure are listed below in Section 9.6.

Pace will provide containers for the collection of samples upon client request for analytical services.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

6.1 Handling and Holding Times:

All shipments of samples should be opened in a fume hood or other negative pressure environment so that the sample handler is protected from any potential vapors or other exposure.

If samples are suspected to be of high concentration and are not to be immediately analyzed, they will be isolated in a separate cooler unit in the lab. If a separate cooler is not readily available in the lab, one will be purchased and kept outside the laboratory.

6.2 Storage

The following table lists the storage, preservative and holding times for the rock samples.

Sample type	Collection per sample	Preservation	Storage	Hold time

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Rock	One 15-20g aliquot in vial with 20 mL purge and trap grade MeOH.	Methanol	With methanol: 4 ± 2°C.	Preserved with methanol: 14 days

7.0 EQUIPMENT AND SUPPLIES

General materials: The following is a list of the primary pieces of equipment and supplies that are required to complete these procedures.

7.1 Equipment

- Panasonic U1 Toughbook handheld computer with field database
- Field computer with field database
- Printer/Copier– HP Envy 4500
- Dymo label printer– Label Writer (currently have a 450 Twin Turbo)
- Balance: Ohaus – SP402
- Camera – Fuji Film XP
- Tripod
- Small dry erase board (2)
- Re-pipettor – VWR International Lab Max 25mL
- Enerpac™ hydraulic system – E3007C
- Rock crushing press – custom made
- Sample trimming cells – custom made
- Complete sample crushing cells – custom made
- Rods (for knocking out crushing cell pucks) – custom made
- Vacuum sealer
- Decontamination wash tubs (4)
- Easy-up tent (1-2)
- Folding tables (2-3)
- Chairs (3)
- Crack hammer (2)
- Steel chisel 3” (4-6)

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- Steel spatulas (4-6)
- 6-foot plank (core tray) marked in 0.1 foot intervals
- 6-foot long, 6" PVC pipe cut in half lengthwise
- C clamps (3-4) – 8"
- Crescent wrench
- Small coolers (1-2)
- Freezer
- Tape measure
- Calculator
- Pick (for replacing o-rings on pucks)
- Scissors
- Squirt Bottles (4-6)
- 40mL VOA vial racks (4)
- 5-gallon buckets (3-4)

7.2 Supplies

- 40mL VOA vials with Teflon-lined septa and screw caps (solid cap) – C&G LV53-A020-A01D-ZM1
- Spare O-rings for crushing cell pucks - 328 Buna W 70 O-Ring
- Spare bolts for funnel/crushing cell base/collars – F593C THE
- Wood or Foam blocks 2" x 2"
- Paper towels
- Teflon tape
- Duct tape
- Packing tape
- Rubber bands
- Bubble wrap
- Aluminum foil (heavy duty)
- Plastic wrap
- Parafilm
- Vacuum sealer bags
- Printer paper

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- Dymo labels
- Ice packs or ice
- Fragile shipping labels
- Dangerous goods in excepted quantities labels
- Kim-wipes
- Trash bags
- Required field personnel protective equipment (PPE) – see section 4.0 for more details

8.0 REAGENTS AND STANDARDS

Purchase reagents from commercial vendors that provide a Certificate of Analysis (COA). Document the receipt of chemical materials and keep a copy of the COA for traceability. Rather than provide the COA with the shipment, some vendors now provide access to COA on their website. When this is the case, download a copy of the COA and retain the COA in accordance with the laboratory's SOP for record retention.

Store reagents in accordance with manufacturer recommendations and this SOP and replace on expiration. The expiration date is measured from date of preparation so long as the parent material does not expire sooner, in which case, use the earliest expiration date.

8.1 Reagents

8.1.1 Purge and Trap Grade Methanol – Fisher Chemical A453-1

8.1.2 HPLC Grade Methanol – Fisher ChemicalA452-4

8.1.3 Analyte Free Water

8.1.4 Alconox – Alconox Inc: Powdered Precision Cleaner

8.2 Standards

8.2.1 Not applicable to this SOP

9.0 PROCEDURE

9.1 Notification to proceed from the client:

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9.1.1 Upon receiving notification to proceed from the client about the project, the following information should be obtained, and a sampling plan created for each new sampling site.

9.1.1.1 Name of the client and/or responsible parties, and telephone numbers where they can be reached.

9.1.1.2 Directions to the facility and, if necessary, to the individual sampling boreholes within the facility.

9.1.1.3 Locations and names of the borehole locations and the analyses needed from each location.

9.1.1.4 Equipment that will be required for the sampling event.

9.1.1.5 Any project or site-specific instructions (i.e., call site contact when arriving at or leaving the site, sampling can only be done on certain days of the week or month, etc).

9.2 Preparation for sampling episode:

9.2.1 After the above information has been obtained and the project has been accepted and approved by the management of Pace, the sampler should follow the steps below before leaving the laboratory.

9.2.1.1 Compile a list of all analytical methods to be used for the project.

9.2.1.2 Discuss with the responsible laboratory Project Manager to ensure the client has been informed and approved of the scheduled Pace sampling event.

9.2.1.3 Retrieve all consumables and sampling equipment required.

9.2.1.4 The sampler should map the most direct route to the sampling site.

9.3 Rock Coring Technique and General Sample Selection Process:

Core runs are most often HQ-sized (2.5-inch diameter) collected in five-foot lengths. They are collected using a triple-tube core barrel system. NQ (1.87-inch diameter) or PQ (3.35-inch diameter) may also be used, but still need to be triple tube. HQ is the preferred size since it yields core that is large enough to sample (NQ is considerably smaller), but not so large to be unwieldy to work with (PQ core can be heavy and time-consuming to sample). Immediately upon retrieval, cores are removed from the core barrel and placed in aluminum foil-lined PVC tray (6-feet long, 4-inch diameter PVC pipe split lengthwise) and covered with a continuous sheet of clean aluminum foil to minimize volatilization of VOCs. The geologist and hydrogeologist inspect the core and select sample locations based on presence of fractures, lithology, weathering and evidence of groundwater and/or DNAPL fluid flow. The hydrogeologist will flag sections of the core to be subsamples for VOC analyses and physical property measurements. This is usually done by writing notes and symbols on a strip of duct tape on the core tray, using dry-erase marker. The drilling pace should be controlled by the on-site hydrogeologist to ensure that the core is not drilled or retrieved before the hydrogeologist and crusher are prepared to receive it (i.e. previous core run has been completely processed or nearly so).

9.4 Data Recording via Field Database:

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All information (drilling progression, sampling information, crushing information, shipping, etc.) will be recorded in the COREDFN Field Database. This Microsoft Access database has two components: a sample station copy and a crushing station copy. These two components are synchronized multiple times throughout the day and backed up frequently. Be sure to familiarize yourself with this database before beginning a CORE^{DFN} project. The hard copy field forms should be available in the event of database or computer malfunction.

9.5 Photographing Cores:

9.5.1 Before the core has been sampled, it needs to be photographed to provide a visual record of the core before pieces of it have been removed. It is important to do this rapidly to minimize VOC loss while core is exposed.

9.5.2 These photographs serve as a vital future reference. Since the rock is removed and destroyed during the sampling process there is no other way to see what the missing sections of core looked like.

9.5.3 The core should be photographed with a whiteboard (in each photo) that displays the following information: Site name, core hole ID, run number, run start/end depth, recovery, and rock quality designation (RQD). Arrows should be displayed to indicate core orientation (top and bottom). The core should be photographed from left to right (top to bottom) using a tripod. A five-foot core typically requires 3 photographs to capture. It is essential that no section of core is missed. In fact, a slight overlap in frames is preferred. To make this easier, it is useful to mark on the table where the tripod needs to be positioned for each frame, to ensure consistent photographs.

9.5.4 The photographs should also include the location of VOC, Physical Property (PP), and other samples. These can be shown either by notes written on the duct tape strip of the sample tray, or by placing the wooden blocks next to the sample locations (see subsection 9.6 below).

9.5.5 Try to minimize shadows and avoid having one section of core in bright sunlight, and another in shade. It is often helpful to spray the core with analyte free-water water (i.e. spring water) before photographing. If interesting features are noted take detailed photographs.

9.5.6 If allowed at the Site it can be helpful to photograph conditions at the Site, especially drilling operations or field setups. Assuming it is allowed, take photographs of the rig, return water setup, tooling, cutting shoes, etc.

9.6 VOC Sample Collection:

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9.6.1 Subsamples (i.e. core segments approximately 1 to 2-inches long) for determining VOC concentrations will be collected from the cores immediately to minimize chemical losses due to volatilization. The initial core logging will be performed to identify key features for subsampling purposes and samples will be quickly broken from the core using a hammer and chisel, and then wrapped in aluminum foil for VOC and moisture preservation and assigned a unique field ID. The foil wrapped sample is then placed in a zip-lock bag and labeled with the same unique field ID as the wood block. After these samples have been collected the core will be logged in more detail by the geologist. Additional sample types will then be selected and removed from the core. Wood blocks will be placed in the core boxes with a unique field ID where samples have been removed for future reference indicating the sample ID and the type of sample.

9.6.2 Samples for VOC analysis will typically be collected using the three criteria identified below unless the project work plan specifies another scheme:

9.6.2.1 Samples will be taken immediately adjacent to (including fracture surface) and 6 to 12 inches away from identified fractures, either above, or below these features. All breaks in core should be suspected as being fractures in-situ, however, emphasis will be made on features with additional lines of evidence for active fluid flow (i.e. secondary mineral coatings/staining, slickensides, report from drillers regarding fluid loss/gain at specific depths during coring). These samples are intended for measuring the extent of diffusion into the matrix away from fractures that may have once contained DNAPL or solute contamination.

9.6.2.2 Additional subsamples will be collected at distinct changes in lithology/mineralogy. Samples will be collected from both sides of such boundaries, referred to as lithology pairs so that representative samples are collected from the different matrix materials.

9.6.2.3 Field duplicate samples and matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a minimum frequency of 1 in 20, taken from the same length of core to provide samples from the same depth interval and lithology.

9.6.3 The Sampler will exercise judgement during sampling to provide an average sample frequency which adequately describes the system, or as specified in the work plan. An average sample spacing of one sample every 1 to 2 feet is recommended.

9.7 Physical Properties Sample Selection:

9.7.1 Physical Properties samples will be collected approximately every 20 feet. An effort will be made to obtain samples from different lithologies or where variations in lithology occur as assessed during core logging. Each sample will be a cylinder of the same diameter as the core retrieved from the core barrel, with a height of approximately 6 inches. These sections of core will be obtained such that the in-situ moisture conditions are retained: working quickly, keeping the core covered and out of direct sunlight, and immediately wrapping and sealing the samples. After the sample is broken from the core, it will be immediately wrapped tightly in clear plastic wrap twice around the circumference for complete coverage of the sample, followed by aluminum foil and a duct tape label. Each sample will be clearly labeled with the sample ID, core location, depth and date. The sample will then be placed in a vacuum sealable bag and vacuum sealed. Details and a full lithological description of each sample will be recorded in the field database.

9.8 VOC Sample Crushing and Preservation:

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9.8.1 VOC samples will be collected in 40mL clear glass VOA vials with Teflon-lined septa and screw caps (a solid cap). The rock core sampling requires both purge and trap grade methanol for rock sample extraction and preservation, and wash grade methanol for decontamination. Each labeled sample container and lid will be weighed empty and re-weighed once the purge and trap methanol has been added to accurately determine the weight and volume of methanol extract. Sample vials should be prepared with methanol within a few days of sampling when possible; if not the weight of the vial with methanol should be re-checked to ensure no significant methanol loss prior to use. At the end of each sampling round, or at the end of the drilling day, vials used for rock samples will be weighed to the exact weight of each rock sample is known. There should be approximately 20 grams (g) of rock in each sample vial. The crushed rock in the vial should never be above the level of the methanol. Sample bottles will be stored in a cooler with ice, in a fridge or in a freezer set to 4°C or less until they are shipped to the laboratory. The sample temperature will be verified upon receipt at the laboratory.

9.8.2 The following is a step-by-step outline of the process of rock core sampling for VOCs:

9.8.2.1 Lay core in an aluminum foil-lined split PVC tray. Note top/bottom of core and depth interval, measure length and quickly identify features (fractures, breaks, lithology/mineralogy changes, evidence of fluid flow (i.e. secondary minerals, precipitates, slickensides, etc.) to select sample locations. Cover with aluminum foil to minimize VOC volatilization and evaporation of pore water during any delays.

9.8.2.2 Collect VOC samples: break off a 1 to 2-inch section of core using a hammer and chisel, insert a wood spacer that specifies the field ID (e.g. A) and type of sample taken (VOC).

9.8.2.3 Completely wrap each sample in a piece of clean aluminum foil to minimize volatilization loss and place in a zip-loc bag with the unique field ID, and place in a cooler with ice for storage prior to crushing. Record sample information including a lithology description, sample type, depth, position relative to nearby fractures and other pertinent details.

9.8.2.4 Trimming the samples, remove the outer rind of the sample exposed to drilling fluids using a hammer and chisel and a steel trimming cell.

9.8.2.5 Place subsample in a clean, dry crushing cell and crush with the Enerpac™ system.

9.8.2.6 Place stainless steel funnel, crushing base/collar and VOA vial prepared with methanol below crushing cell. Empty crushed rock sample (approximately 20g) into 40 mL VOA sample vial containing a known volume (15mL) of purge and trap grade methanol, taking care to avoid splashing of methanol out of the vial. Sample vials and screw caps with septum have been previously labeled and weighed. Bottle threads should be wiped clean with a kim-wipe prior to screwing on the cap to remove any rock fragments that prevent the cap from sealing properly.

9.8.2.7 Decontaminate the rock crusher components using the procedure outlined in section 8.9. Multiple crushing cells should be used (at least four or five sets) to allow processing of all samples from a given core run before decontamination is required.

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9.8.3 Weigh vials with the rock samples so the mass of the rock is known. This data is recorded in the database.

9.8.4 If samples are to be analyzed off-site, wrap screw cap lid and shoulder of bottle with Teflon tape, wrap vials with bubble wrap, rubber band them and place individually in zip-lock bags for shipping. Place in cooler with trip blanks, which remain with specific batches of samples until arrival at the laboratory and keep on ice.

9.8.5 Sample shipping: Follow proper procedures for shipping samples as methanol is considered a dangerous good, which can be shipped in excepted quantities, 500 mL maximum per cooler.

9.8.6 Chain of custody (COCs) forms will be filled out at the end of each day of sample collection and shipped with each cooler to the laboratory.

9.9 Decontamination of Drilling and Subsampling Equipment:

9.9.1 Decontamination procedures are designed to remove all traces of contaminants from the equipment to prevent cross-contamination. Core barrels will be cleaned between runs using clean water and pressure washer. Sampling and trimming chisels will be sprayed off with wash grade methanol followed by distilled water and dried with a clean paper towel before re-use. Only those parts of the rock crusher that come into contact with the rock subsamples require decontamination, including the crushing cell, top and bottom plates (pucks), funnels, funnel/cell base/collars and spatulas/knives used to direct the crushed rock sample into the VOA vial. Trimming cells will also be decontaminated between samples. At least four or five sets of crushing cells will be used, which in most cases should allow processing of all of the samples collected from a 5-foot core run. The procedure for decontaminating these components consist for four steps, listed below:

9.9.1.1 Clean with a solution of water and a phosphate-free detergent (e.g. Alconox). Wash to get rid of obvious sediment and scrub with a scrub brush all surfaces that encountered the rock sample.

9.9.1.2 Fully immerse in a clean water rinse.

9.9.1.3 Rinse all surfaces with wash grade methanol using a squirt bottle.

9.9.1.4 Rinse with clean (analyte-free) water using a squirt bottle to remove all traces of methanol.

9.9.2 Parts are then dried using clean paper towels. Detergent and water baths will be changed on a regular basis and equipment blanks will be collected after every 20 samples by wiping the inside of a clean crushing cell with a kim-wipe pre-soaked in purge and trap methanol which is then placed back in the 40mL VOA vial to be submitted for VOC analysis along with the rock samples. Decontamination fluids and remnant rock fragments should be contained and properly disposed of on-site.

9.10 Documentation:

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9.10.1 Immediately following sample collection, identify each sample container with a unique sample ID using an indelible marking pen or by affixing the appropriate pre-printed label.

9.10.2 Include the sample ID, collection date and collection time on the label and transfer that information to the chain of custody form.

9.10.3 Record any sampling information that is specific to the site while still at the site.

9.10.4 Place the chain of custody form in a Ziploc bag and place it in the shipping container with the samples. The bag can alternatively be taped to the inside of the cooler lid.

9.10.5 When transporting samples, be sure that the samples are placed in a cooler and oriented such that they will not spill or fall out of the cooler. If a cooler of samples is left in a vehicle for any reason, the vehicle must be locked to ensure that proper chain of custody is maintained.

9.10.6 All individuals who handle and relinquish the sample containers must sign the transmittal form. The legal custody responsibilities of the field operations continue until the samples are relinquished to the laboratory.

9.11 Sampling site departure:

9.11.1 Verify that all equipment and safety gear has been cleaned and accounted for. Be sure all pertinent sampling paperwork and observation documentation is complete and correct. This includes signatures on the chain of custody, notes describing conditions at the site, and cross referencing of sample ID's, collection dates and times on the container labels and on the chain of custody.

10.0DATA ANALYSIS AND CALCULATIONS

10.1 Qualitative Identification

10.1.1 Not applicable to this SOP.

10.2 Quantitative Identification

10.2.1 Not applicable to this SOP.

10.3 Calculations

10.3.1 Not applicable to this SOP.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Field Quality Control

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11.1.1 Equipment blanks, collected as described above, will be taken every 20 samples. Trip blanks, consisting of purge and trap grade methanol in 40 mL vials will be placed with each batch of samples shipped in coolers. These are filled with methanol at the same time as the sample vials and handled with the prepared vials and are placed with each batch of methanol preserved VOC samples, and remain unopened until returned to the laboratory for analysis.

11.1.2 Field duplicate and MS/MSD samples will be taken every 20 samples ensuring the samples are from the same depth interval and lithology as the parent sample.

11.1.3 Methanol blanks, used to identify contamination of the purge and trap grade methanol, are taken at the start and end of each methanol bottle (typically 1L) used to fill sample vials and are stored/shipped separately from the rock samples.

11.1.4 All QA/QC samples will be identified on the COC forms and tracked throughout the sample handling and analysis process via the database.

11.2 Method Performance

11.2.1 Not applicable to this SOP.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Data review includes a series of checks performed at different stages of the process by different people to ensure the record is correct and complete and documented, that SOPs were followed, proper corrective actions were taken to QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The checks that naturally occur as employees review their own work is called primary review.

All data is also reviewed by an experienced peer or supervisor. This secondary review is performed to verify SOPs were followed, and QC criteria were met, proper corrective actions were taken, the analytical record is complete and traceable, and that results and data are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is complete and that project specifications were met.

12.2 Corrective Action

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12.2.1 Not applicable to this SOP.

13.0 POLLUTION PREVENTION AND MANAGEMENT

13.1.1 All waste created on site should remain on site, work with client to ensure this prior to mobilization.

14.0 MODIFICATIONS

14.1.1 Not applicable to this SOP

15.0 RESPONSIBILITIES

All Pace employees are responsible for following the procedures in this SOP and for seeking, obtaining and documenting management permission to depart from procedures in this SOP, when necessary.

Pace managers and supervisors are responsible for training employees on the procedures in this SOP, monitoring the implementation of this SOP in their work area, for review of documents used in their work area for completeness, correctness, suitability and compliance and for notifying QA when errors in document are found or when it is found that practice in the work area does not match the document.

16.0 ATTACHMENTS

Appendix A

17.0 REFERENCES

18.0 REVISION HISTORY

This Version:

Section	Description of Change

This document supersedes the following documents:

Document Number	Title	Version

DRAFT

STANDARD OPERATING PROCEDURE

CTS-10.17.2

MICROWAVE ASSISTED EXTRACTION OF VOLATILE ORGANIC COMPOUNDS FROM ROCK SAMPLES

NOTE: THIS SOP CONTAINS PROPRIETARY INFORMATION

DO NOT DISTRIBUTE OUTSIDE OF PROJECT TEAM

SOP Number: CTS-10.17.2 Date Issued: 07/02/08

Revision Number: 2 Date of Revision: 04/05/18

1.0 OBJECTIVE

The Microwave Assisted Extraction (MAE) method was designed at the University of Waterloo and is used to extract Volatile Organic Compounds (VOC) from rock samples.

2.0 SUMMARY OF METHOD

The microwave extraction is conducted in a microwave system. Cascade currently owns two different microwave systems. The first system (Cascade's Microwave Unit "MW1") is an Ethos Sel Labstation, built by Milestone Srl, Italy. This system utilizes the MPR-600/12S medium pressure, segmented rotor, which contains 12 vessels for solvent extraction. The second system (Cascade's Microwave Unit "MW1") is a MARS6, built by CEM Corporation, North Carolina, USA. The CEM system is equipped with the MARSXpress three-piece, 24-vessel assembly, which allows a high throughput and quick cooling.

Rock core samples are collected and immediately crushed using a device that was designed by Beth Parker's research team at the University of Waterloo. The crushed samples are preserved in purge and trap grade methanol and then undergo the microwave assisted extraction (MAE) process described in this standard operating procedure (SOP). Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass. By using MAE, this extraction time is reduced to fewer than 3 hours.

3.0 DEFINITIONS

Preparation Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or
other undesirable situation in order to prevent recurrence.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed, and which is accepted as the method for performing certain routine or repetitive tasks.

Preparation Blank (PBLK): a blank matrix that is the same matrix as the client samples in a preparation batch. The PBLK undergoes the preparation or extraction process with and in the same manner as client samples before being introduced onto the instrument for analysis.

Preparation Laboratory Control Sample (PLCS): a blank matrix that is of the same matrix as the client samples in a preparation batch and that is spiked with all target analytes. The PLCS undergoes the preparation or extraction process with and in the same manner as client samples before being introduced onto the instrument for analysis.

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank prepared from purge and trap methanol and carried through sampling and subsequent storage and handling must serve as a check on such interferences. Sulfur dioxide is a potential interferant in the analysis for vinyl chloride.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are extracted in sequence in the same vessel. Whenever an unusually concentrated sample is encountered, it is recommended to prepare methanol blanks in the vessels previously used for the extraction of highly concentrated samples. Extraction vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used, or by using analyte-free water. Clean, dry glassware should be stored in a clean environment.

5.0 SAFETY ISSUES

Laboratory personnel should take extra care when working with solvents and VOC standards. When working with standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and, if necessary, safety glasses.

If necessary and appropriate, a site-specific health and safety plan shall be created and followed for each study site. All samples, as well as standards, should be treated as a potential health hazard. Exposure to each should be reduced to the lowest level possible using gloves and a hood.

All chemicals are required to be received with a SDS. SDSs shall be made available to all personnel involved in the sampling and testing.

Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slips, trips, and falls, poisonous plants and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate CTS employee.

6.0 EQUIPMENT AND SUPPLIES

- Cascade uses two microwave systems:
    - Microwave Unit "MW1": Model Ethos Sel Labstation, built by Milestone Srl. (Italy). MW1 is equipped with the MPR-600/12S medium pressure, segmented rotor containing 12 vessels for solvent extraction. The vessels are made of TFM Teflon®, which is chemically inert to most organic solvents and combinations thereof. It is microwave transparent and its maximum working temperature for extended use is 260°C, and for brief use is 300°C. Each vessel is protected by a

safety shield, for which the maximum temperature for extended use is 250°C. The microwave system is controlled by a dedicated computer.

- Microwave Unit "MW2": Model MARS6, built by CEM Corporation. MW2 is equipped with a turntable and 24 MARSXpress Plus vessels. The vessels are made of TFM Teflon®, which is chemically inert to most organic solvents, and have a self-regulating pressure control to eliminate the risk of over pressurization. The three-piece vessel consists of a liner, plug and cap. The open turntable design and composite sleeves allow for fast cooling. The microwave system is controlled by a built-in computer.

- Analytical balance
- Glass disposable pipettes
- Sample glass vials (2 or 4 mL)
- Purge and trap grade methanol
- Wash grade methanol
- Analyte-free water
- Syringes
- Centrifuge
- Oven for baking/decontamination of vessels

7.0 REAGENTS AND STANDARDS

Organic-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized).

Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).

HPLC grade or equivalent grade methanol for decontamination of microwave vessels (demonstrated to be free of analytes and stored away from other solvents).

Stock standards for spiking Preparation LCS and Matrix Spike samples:

Stock standards may either be prepared from pure standard materials or purchased as certified solutions. Secondary dilution standards are prepared using stock standard solutions, which contain the compounds of interest, either as single compounds or mixed together. Typically, for this method, the stock standards are obtained from an approved vendor and mixed together in

secondary dilutions. A certificate of analysis (COA) is retained by the laboratory and maintained on file or on file with the vendor.

Cascade's SOP CTS-4.7.n, "Labeling, Preparation and Storage of Reagents, Solutions and Standards", provides procedures on properly labeling, preparing, and storing reagents and standards used in the mobile laboratory for analytical methods. This SOP should be reviewed by the analyst before labeling, preparing, and storing standards within the mobile laboratory.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection and Handling

Rock core samples are collected by trained geologists in accordance with CTS's SOP SEI-6.37.n. Approximately 15 g of crushed rock sample is transferred into a pre-weighed 40 mL VOA vial containing 15 ml of purge and trap grade methanol. The samples are typically stored in a cooler on ice or in a freezer until they are shipped or transported on ice to the laboratory. The samples are weighed upon receipt at the laboratory and the weights are recorded in the Laboratory Information Management System (LIMS). The crushed samples then undergo microwave assisted extraction (MAE) prior to analysis.

8.2 Handling and Holding Times

Samples should be received at the laboratory at a temperature of $\leq$6 º C; sample temperature shall be verified upon receipt at the laboratory. Once at the laboratory, samples are stored in a freezer before and after extraction. If methanol-preserved rock samples are stored in a refrigerator, the sample holding time is 14 days. If methanol-preserved rock samples are stored in a freezer (below -10C), the sample holding time is 28 days. Once extracted, extract holing time is 28 days, if stored in a freezer (below -10C). Samples are allowed to come to room temperature just prior to analysis.

9.0 PROCEDURE

Step 1: Sample Receipt

1. Use an infrared thermometer to measure the temperature of a representative sample from each cooler; record the receipt temperature on the associated chain of custody (COC).
2. Unwrap samples carefully, removing any Teflon tape from the sample vials.
3. Check all samples against the chain of custody forms for completion and accuracy.
4. Sign all COCs and record receipt date and time.
5. Weigh each sample vial and record the weight on the COC and/or directly into the LIMS.

6. Log each sample into the LIMS. The LIMS will assign a unique laboratory ID to each sample. Note: If using LIMS batch login function, weights may be recorded directly into the LIMS at login rather than on the COC, as long as the following steps are taken. Sample weight data MUST be exported from the LIMS (using "Review Sample Weights" button or running qrySampleWeights) to a separate excel sheet **immediately** after each batch is logged in. Save each excel file with a unique file name in the project directory after each batch login. Check the weights to make sure that each sample has a reasonable receipt weight. Print this record, initial and date to indicate that the weights have been reviewed and attach the weights sheet to the COC.
7. Store samples in a temperature-monitored laboratory freezer until they are ready for extraction.

Step 2: Preparing Samples for Extraction

For samples being analyzed via method 8260C, the methanol used for collection of samples would preferably be spiked with surrogate analyte bromofluorobenzene (BFB). If pre-spiking with BFB is not possible, each sample should be spiked with BFB at the appropriate concentration prior to extraction. Note that if the samples will be analyzed via GC/ECD following MAE rather than via method 8260C, samples should not be spiked with BFB, as the GC/ECD method does not include surrogates.

1. A Microwave Assisted Extraction (MAE) Log will be populated for each extraction, or preparation, batch. Each extraction batch should have a unique serial identifier (e.g., MW1-0001, MW1-0002, etc.). This batch ID will be identified at the top of the MAE Log, along with the project number, date, time, extraction chemist and vessel set (e.g., A, B, C). Lot numbers of solvents and spiking standards used in the extraction batch shall also be indicated on the MAE Log. The MAE Log indicates the contents of each of the vessels in that extraction batch. Choose a set of project samples from the freezer for the current extraction batch and record those samples on the MAE Log, assigning each to a specific microwave vessel listed on the MAE log (Figure 1).
2. Each preparation batch shall include at least one preparation blank sample (PBLK). With the Milestone system, the reference vessel (Vessel #1) is used to measure the temperature and adjust the power to achieve the proper temperature. This vessel is always used for the PBLK sample for each extraction batch. The CEM system uses a light emitting technology (LET) called iWave® to measure the sample temperature of each individual vessel. Therefore, there is no reference vessel and any vessel can be used for the PBLK. However, for consistency, the first vessel is typically used for the PBLK. To prepare the PBLK in a CEM vessel, fill the vessel with 15 g of clean sand (i.e., blank matrix) and 20 ml of the Purge & Trap grade methanol and spike with the appropriate amount of BFB (applicable to Method 8260C analysis only). As per manufacturer recommendation, the

Milestone reference vessel (Vessel #1) should contain solvent only (no solid matrix), so a PBLK in Milestone's reference vessel will be prepared in the same manner described for the CEM PBLK preparation above, with the exception of the sand matrix, which shall be omitted. Name the PBLK according to the extraction batch ID (e.g., PBLK-MW1-0001). Record on the MAE Log the concentration, volume, lot number of the BFB Spiking Standard used (if applicable).

3. Each preparation batch shall include one spiked preparation laboratory control sample (PLCS). Vessel #2 is typically used for the PLCS sample. To prepare the PLCS sample, fill the PLCS vessel with 15 g of clean sand (i.e., blank matrix) and 20 ml of Purge & Trap methanol and spike with a standard containing the project's target analytes and surrogate BFB (if applicable). Name the PLCS according to the extraction batch ID (e.g., PLCS-MW1-0001). Indicate on the MAE Log the concentration, volume, and lot number of the Spiking Standard used for the PLCS.

4. Any matrix spike (MS) or matrix spike duplicate (MSD) samples in the batch should be spiked in the same manner and concentration that the PLCS is spiked. PLCS and MS/MSD compound recoveries are evaluated against MAE-specific control limits that have been calculated by the laboratory.

5. Prepare samples one at a time for microwave extraction via the following steps:

   a. Ensure that each microwave vessel is clean and clearly labeled with its unique vessel ID. In the fume hood, transfer the entire contents of each sample vial into its assigned microwave vessel (as per the list on the MAE Log).

   b. A methanol rinse step is performed to transfer any remaining sample material out of the sample vial and into the microwave vessel. Using a syringe, introduce 5 mL of purge and trap methanol into the mostly empty sample vial. It is recommended to use about 3 mL of methanol to go around the sides of the vial to move remaining sample to the bottom of the vial. Then turn the vial upside down so that the contents pour into the MAE vessel. Use the remaining methanol by squirting the bottom of the vial while it in upside down so that most of the contents pour into the vessel.

   c. If methanol is not pre-spiked with BFB, spike each sample with the appropriate concentration of BFB (applicable to 8260C analysis only).

   d. Put the Teflon vessel lid (and plug in the case of MW2) onto the vessel and weigh the vessel.

   e. Record vessel weight on the MAE log.

   f. Assemble the vessel with its protection shield and tighten it either with the appropriate torque wrench. The protective shield for CEM vessels is part of the turntable. For CEM

vessels, ensure that the plug is on and then tighten the cap with the white torque wrench.

g. Place vessel into rotor/turntable.

h. Repeat until all samples have been transferred into vessels and all vessels have been properly closed and loaded into the rotor/turntable.

Step 3: Microwave Extraction

Milestone System:

1. Place the rotor into the microwave ensuring that it is properly seated on the spindle inside the microwave.
2. Install the temperature sensor by inserting the fiber optic sensor into the thermowell hole on the top of the reference vessel (vessel #1) until it is firmly set. Feed the cable of the temperature sensor through the hole of the guide arm on the top inside of the oven and attach the other end of the temperature sensor (it is blue) to the microwave by plugging it into the top hole on the left inside wall of the microwave. Again, press in until it is firmly set. Note that this fiber optic sensor is very delicate and extreme care should be taken to not bend or pinch any part of this cable.
3. Turn on the microwave at the power switch. This also turns on the control screen. On the control screen, select *Administrator* and type in the password (123456). Touch the *PressPrep* button and then go to the *Method* tab and the *Param* subtab. Make sure that the loaded method is *rockcore.mpr*. Check that the *Twist* and *Start-Param* buttons are checked and also make sure that the *Control* for the dropdown menu is set to *T1* and that *Ventilation* is set to 60 minutes. Use the vent button (looks like a fan) to rotate the rotor to access a certain vessel. Ensure that the thermometer probe is positioned correctly so that it has enough slack to move with the rotor without becoming kinked. The table below summarizes the parameter settings:

Method	rockcore.mpr
Twist	Checked
Start-Param	Checked
Control for	T1
Ventilation	60min
Level 1	T=00:07:00, E=1200 W, T1=120 °C
Level 2	T=00:40:00, E=500 W,

	T1=120 °C

4. To start the microwave extraction, press the green start button on the control screen. Before actually starting, the microwave will ask you two warning questions. The first asks you if you want to continue even if your pressure sensor is not monitoring. Say yes to continue, as we do not have a pressure sensor in place. The second warning wants you to check your stirrer. We do not have this function enabled, so continue through this message as well. At this point, the microwave program should begin and proceed according to the method. The program should take two hours to complete. This includes a ventilation step that will cool the vessels. **The vessels should not be opened until they have cooled to 25 °C or below.** To speed up the cooling process, the vessels in their protective shields can be removed from the rotor at approximately 50 °C and placed into a freezer. To check the temperature before opening the vessels, return the reference vessel (i.e., Vessel #1) to the microwave and reinsert the temperature sensor for a reading. NOTE: The temperature sensor is delicate. When you are done with it, disconnect it and place it in a secure location, out of the way of the microwave door and other equipment. On the control screen, the temperature reading will display 297 °C when the sensor is unplugged. This is ok.
5. Each sample will require one 4-ml glass vial. Each of these vials needs to be labeled with the sample id, project number, extraction date, extraction chemist's initials, batch number and vessel number. See Figure 2 for an example of an extraction vial label. The labeling process can be done during the microwave extraction or while the cooling is in progress. Use the Dymo label software to print out labels for the 4-mL vials. Attach the labels, cover each with a layer of clear cellophane tape, and organize the vials in a rack in the same order the vessels are in.
6. Once the vessels have reached 25 °C, you can begin to remove the protective shield from the vessels one sample at a time with the torque wrench or by hand and reweigh the vessels with the lids on.
7. Record the weight in the MAE log sheet.
8. In the hood, remove the Teflon lid. **NOTE: Use extra caution when removing the Teflon lid from the reference vessel. Lift it straight up to prevent cracking the thermowell.**
9. Using a fresh Pasteur pipette for each sample, transfer approximately 4 mL of sample (methanol extract only) from the vessel into its appropriately labeled 4-mL vial. Leave the rock material behind in the vessel.

10. Repeat these steps for each vessel until all extracted samples have been transferred to 4-mL extraction vials.
11. Place the 4-mL vials into the centrifuge and turn the centrifuge on until all the solid contents remain on the bottom of the vial, approximately 5-10 min.
12. Store the 4-mL sample vials in the freezer below -10 °C until ready for analysis. Label the sample vial rack with the project number and sample delivery group.

CEM System:

1. Turn on the CEM MARS6 system via the power switch located on the lower right side. This may take a minute or two.
2. Place the turntable into the microwave ensuring that it is properly seated on the spindle inside the microwave.
3. From the Home Screen, select "Classic Methods" on the touch screen. Then select "8260 MAE" method. The method parameters are:

Stage	Temp (°C)	Power Permitted (W)	Ramp Time (min)	Hold Time (min)	Total Time (min)
1	100	400	10	5	15
2	120	300	15	40	55
					70

4. Push the "Play" button on the top right of the screen and the microwave program should begin and proceed according to the method.
5. Each sample will require one 4-ml glass vial. Each of these vials needs to be labeled with the sample id, project number, extraction date, extraction chemist's initials, batch number and vessel number. See Figure 2 for an example of an extraction vial label. The labeling process can be done during the microwave extraction or while the cooling is in progress. Use the Dymo label software to print out labels for the 4-mL vials. Attach the labels, cover each with a layer of clear cellophane tape, and organize the vials in a rack in the same order the vessels are in.
6. Once the method has completed, the system will start to ventilate for 60 min. The MARS6's open turntable design and composite sleeves allow for quick cooling. The samples should reach 25 °C in less than 60 minutes and will not need to be cooled in a freezer.

7. Once the vessels have reached 25 degrees C or lower, press the "Stop" button. Remove the turntable from the microwave and weigh each of the vessels. Record post-extraction weights in the MAE Log.
8. Follow steps 8 through 12 of the Milestone Microwave Extraction System above.

Step 4: Decontamination of Teflon Vessels

1. Clean and decontaminate each Teflon vessel, cap, and plug. The other Milestone components do not need to be decontaminated, as they have not come in contact with the samples. Follow these steps:
    i. Working in the fume hood, decant the remaining methanol into the water/methanol hazardous liquid waste container.
    ii. Empty the rock into the solid hazardous waste container.
    iii. Rinse the vessels and lids 3x with analyte-free water and then wipe each vessel and lid with a clean paper towel. This rinsate water should be poured into the water/methanol hazardous waste container.
    iv. Rinse each vessel and lid 3x with HPLC grade methanol, pouring the rinsate into the water/methanol hazardous waste container.
    v. Allow the vessels to stay in the hood until any excess methanol has evaporated.
    vi. Place the Teflon vessels and lids in the oven and bake for 1 hour to remove any remaining VOC contamination. **Milestone vessels can be baked at 250 °C, however, CEM vessels should be baked at 160 °C.**

10.0 CALCULATIONS

For samples being analyzed via Method 8260C, target analytes (for PLCS and MS/MSD samples) and surrogate analyte BFB are typically spiked into the samples in the vessels prior to extraction at a concentration of 800 ppb, or 0.8 ppm. This assumes that the samples will be analyzed via method 8260 (as described in SOP CTS-10.15.n) following extraction; the default dilution factor for analyzing solid matrix samples via this method is 40x. Therefore, this would result in an on-column concentration of 20 ppb. The volume of spiking solution to be used for each sample is dependent on the concentration of the spiking solution. For example, if the concentration of surrogate BFB in the surrogate spiking solution is 200 ppm (C1) and the desired concentration in the vessel containing 20,000 uL of methanol (V2) is 0.8 ppm (C2), then the volume of surrogate spiking solution to be added to that vessel (V1) would be 80 uL. See equation below:

$$C_1 * V_1 = C_2 * V_2 \quad \text{or} \quad 200 \text{ ppm} * 80 \text{ uL} = 0.8 \text{ ppm} * 20{,}000 \text{ uL}$$

For samples being analyzed via GC/ECD rather than Method 8260C, surrogate is not used. PLCS and MS/MSD samples are typically spiked in the vessels prior to extraction at a concentration of 50 ppb for the GC/ECD method.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

Quality control samples are generated during sample preparation to determine if contamination, memory effects, or loss of VOCs are occurring.

11.1 Preparation Blank (PBLK)

A preparation blank (PBLK) is a methanol blank that is prepared with every MAE batch and goes through the entire process with the samples. The PBLK is prepared in vessel #1, which is used to measure the temperature and adjust the power to achieve the appropriate temperature in the case of MW1. It is prepared by adding 15 g of clean sand and 20 mL of purge and trap methanol and spiking with surrogate analyte BFB (if applicable). In addition to the PBLK prepared in vessel #1, additional PBLKs may be prepared and extracted in other vessels, particularly in those that have previously contained high concentration samples. This can demonstrate the cleanliness of these particular vessels and indicate that carryover did not occur from a previous extraction.

11.2 Preparation Laboratory Control Sample (PLCS)

A preparation laboratory control sample (PLCS) is prepared with every MAE batch and goes through the entire process with the samples. The PLCS is prepared in vessel #2 by adding 15 g of clean sand and 20 mL of purge and trap methanol. The methanol is fortified with a spiking standard containing surrogate BFB (if applicable) and all project-specific target analytes. Cascade has developed a set of statistically derived, MAE- specific control limits for the PLCS based on recovery data from past PLCS samples. PLCS recoveries are evaluated against these MAE-specific control limits.

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

Matrix spike/matrix spike duplicate samples field samples that are fortified with a spiking standard containing surrogate BFB (if applicable) and all project-specific target analytes and are used to determine interference from the matrix, as well as precision. An MS/MSD analysis is required every set of twenty samples or as specified by the client. A sample may be chosen by the client to perform a MS/MSD analysis. When submitted to the laboratory, one individual vial shall be submitted for the parent, the associate MS and the associated MSD sample so that each

can be spiked appropriately prior to extraction. Recovery limits for MS/MSD are evaluated against the MAE-specific control limits. The RPD criteria for rock samples shall be 50%.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

1. Yongdong Liu, "Microwave Assisted Rapid Extraction of VOCs from Low Permeability Media", M.Sc. thesis, University of Waterloo, 2005.

2. "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

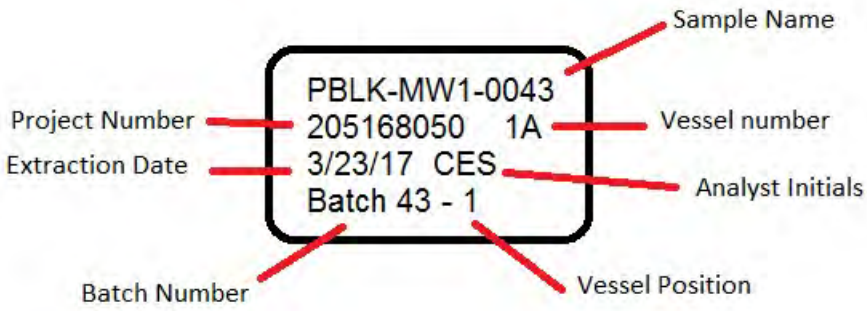
Figure 1. Example MAE Log

ROCK CORE LABORATORY MICROWAVE LOG

Project(s):				Date & Time:	
Extraction Chemist(s):				Extraction Batch#:	
PLCS/MS/MSD Spiking Solution Lot#:				Vessel Set (A, B, C, or 24):	
BFB Spiking Solution Lot#:					

Sample ID	Vessel Number/ Letter	Rotor Position	Pre-Microwave Weight (sample + teflon vessel and lid) (g)	Post-Microwave Weight (sample + teflon vessel and lid) (g)
		1		
		2		
		3		
		4		
		5		
		6		
		7		
		8		
		9		
		10		
		11		
		12		
		13		
		14		
		15		
		16		
		17		
		18		
		19		
		20		
		21		
		22		
		23		
		24		

Figure 2. Example MAE Label



15.0 AUTHORIZATION

Authored by: _____ Date: 4/8/2018

Michael Rossi, Director of Analytical Chemistry Services

Approved by: _____ Date: 4/8/2018

Morgan Greenwald, Laboratory Quality Assurance Manager

16.0 REVISIONS

Revision 1:

1) Added section on decontamination of vessels
2) Added section on temperature program for MAE

Revision 2:

1) Changed all "Stone" references to "Cascade" and all "SEI" references to "CTS." Replaced Stone logos with Cascade logos.
2) Included information on CEM MARS6 microwave system.
3) Indicated that the Milestone microwave unit is referred to as "MW1" and that the CEM MARS6 microwave unit is referred to as "MW2"
4) Changed the acceptable maximum sample receipt temperature from 10 deg C to 6 deg C.
5) Changed "MSDS" references to "SDS."
6) Added batch definition.
7) Added information on sample collection, handling, storage and holding times.
8) Added information on spiking and cleaning vessels.
9) Added information about spiking samples with BFB prior to extraction (indicated this is applicable to Method 8260C only)
10) Added information about the Quality Control samples.
11) Changed title of Michael Rossi
12) Added Morgan Greenwald

APPENDIX C

QUALITY ASSURANCE/QUALITY CONTROL PLAN

APPENDIX C
QUALITY ASSURANCE PROJECT PLAN (QAPP)
B335 Area RCRA Facility Investigation (RFI) Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

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ATTACHMENTS

Attachment 1 Laboratory Standard Operating Procedures

1.0 INTRODUCTION

This quality assurance project plan (QAPP) has been prepared to document sampling and monitoring procedures to be implemented during the Building 335 (B335) Area Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the Former IBM East Fishkill facility located in Hopewell Junction, New York (the site). The QAPP is included as an appendix to the B335 Area RFI Work Plan (hereinafter referred to as the RFI Work Plan).

The following activities are covered by the QAPP: sample collection, field measurements, sample analysis, and data validation. The purpose of the QAPP is to ensure that information, data, and resulting decisions are technically sound, statistically valid, and properly documented. The QAPP was prepared in general accordance with Appendix II-B of the 6 NYCRR Part 373 Permit for the facility.

2.0 DATA QUALITY OBJECTIVES

The data quality objectives (DQOs) were established to meet the following goals in the vicinity of and beneath B335:

- Assess for potential interior and exterior sources of target VOCs in the subsurface;
- Evaluate the distribution of target VOCs in soil, upper bedrock, soil vapor, and groundwater;
- Evaluate contaminant fate and transport of target VOCs; and
- If new sources are identified, collect data to support a preliminary evaluation of remedial alternatives that could appreciably improve upon IBM's current site-wide bedrock groundwater extraction and treatment remediation approach.

The first phase of the assessment will include the advancement and sampling of temporary soil vapor screening implants to preliminarily assess potential tetrachloroethene (PCE) source areas. The second phase of the assessment will include the advancement of soil borings within and in the vicinity of B335 to be completed as multi-level implants constructed using Continuous Multichannel Tubing (CMT®) for sampling groundwater and soil vapor at discrete depth intervals.

It is anticipated that soil and soil vapor samples will be collected from each location, and that groundwater and bedrock samples will be collected where encountered. Soil and bedrock samples will be processed and analyzed using an on-site mobile laboratory. Approximately 10% of the soil and bedrock samples will be submitted to a New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP)-certified fixed laboratory for VOC analysis (split sample, or correlation sample, analysis)[1]. Groundwater and soil vapor samples for VOC analysis will be submitted to a NYSDOH

[1] If microwave assisted extraction is used by the mobile lab, a split of the extract will be submitted to the fixed laboratory for analysis as opposed to the original soil or bedrock matrix.

ELAP-certified fixed laboratory. Samples will be collected using the procedures described in the RFI Work Plan and field standard operating procedures (SOPs) included in Appendix B. The laboratory analyses for the VOC target analyte list will be completed using methods provided in Table C.1.

Soil, bedrock, and groundwater off-site fixed laboratory analytical data for VOCs will undergo data validation and usability assessment prior to use of the data. Criteria for performance measures, including precision, accuracy/bias, representativeness, data comparability, sensitivity (quantitation limits), and completeness are discussed below and shown in Table C.1.

The soil and groundwater analytical data will be compared to applicable New York soil cleanup objectives and groundwater quality standards, respectively, as indicated in Table C.2. The lowest applicable regulatory standards were generally used to set the Project Action Limits (PALs) for soil and groundwater, with the exception of trans-1,2-dichloroethene (trans-1,2-DCE) and vinyl chloride in soil, which were set at the fixed laboratory reporting limit. Bedrock analytical data will be used to assess the vertical extent of contamination, and distribution of contaminant mass for the assessment of remedial alternatives. Therefore, PALs are not applicable for bedrock data. Soil vapor data will be used to assess the distribution of contaminant mass for the assessment of remedial alternatives. Soil vapor data will be compared to the NYSDOH Soil Vapor Matrix criteria and USEPA vapor intrusion screening levels (VISLs) as a means of rough screening indicators, as indicated in Table C.2. The lower of these two applicable standards (NYSDOH) were used to set the PALs.

PALs are not applicable for the physical parameters of moisture content, total organic carbon (TOC), grain size, bulk density, or porosity, based on the purpose of these measurements, as defined in the RFI Work Plan Section 3.5.1.2.

3.0 MEASUREMENT PERFORMANCE CRITERIA

This section describes the performance criteria defined for the analytical measurement systems so that the project DQOs, as defined above, are met for VOCs. Measurement Performance Criteria (MPC) for precision, accuracy/bias, representativeness, completeness, sensitivity, and comparability have been provided in Table C.1 for the proposed laboratory analysis of VOCs in soil, bedrock, groundwater, and soil vapor. The types and frequency of field quality control (QC) samples described in the following sections are included in Table C.3. No project-specific field or laboratory QC samples have been defined for the physical parameters. It is expected that the laboratories will analyze method-appropriate QC samples as defined in their SOPs for these parameters (moisture content, TOC, grain size, bulk density, and porosity).

3.1 Precision

Precision is the degree of agreement among repeated measurements of the same characteristic (analyte, parameter, etc.) under the same or similar conditions. Precision data indicate how consistent and reproducible the field sampling and/or analytical

procedures have been. Field matrix precision will be evaluated by collecting data from field duplicate (FD) samples for VOCs. Analytical laboratory precision for the method of analysis will be measured by analyzing laboratory control sample/laboratory control sample duplicate (LCS/LCSD) samples. Laboratory precision in the site matrix will be measured by analyzing matrix spike/matrix spike duplicate (MS/MSD) samples and laboratory duplicate (LD) samples. Inter-laboratory precision will be measured by comparing the mobile laboratory and fixed laboratory results for the split samples.

Comparing different measures of precision can help to identify sources of imprecision, such as possible laboratory error, change in sample integrity, or sample matrix heterogeneity, if such imprecision exists.

FD, LCS/LCSD, MS/MSD, and LD precision will be evaluated by calculating the relative percent difference (RPD) of the duplicate results using the following equation:

$$RPD = \frac{|x_1 - x_2|}{\frac{x_1 + x_2}{2}} \bullet 100\%$$

Where,

RPD	represents the relative percent difference;
x_1	indicates the original sample concentration; and
x_2	indicates a replicate sample concentration.

Acceptance criteria for LCS/LCSD, Sample/FD, split sample, lab duplicates (if performed), and MS/MSD RPDs are listed in Table C.1.

3.2 Accuracy/Bias

Accuracy is the extent of agreement between an observed value (sample result) and the accepted, or true, value of the parameter being measured. Accuracy is frequently used synonymously with bias. Specifically, the term "bias" describes the systematic or persistent error associated with a measurement process. Sources of error in the field and the laboratory that may contribute to poor accuracy include laboratory measurement error, sampling inconsistency, field contamination, laboratory contamination, and preservation and handling issues. The sample matrix may also lead to inaccurate results for a particular analysis.

Accuracy/bias will be evaluated using several different types of QC samples including, LCS, internal standard (IS) and surrogate spikes, and field and laboratory blank samples. In addition, method-required instrument tuning, initial calibration, and continuing calibration criteria provide for acceptable accuracy of the analytical measurements for this program. For the QC samples that have a "true" value (e.g., LCS and surrogates), the following equation will be used to calculate the accuracy or potential bias in the result as a "percent recovery:"

$$Accuracy/Bias = \%Recovery = \frac{Measured\ Value}{True\ Value} \bullet 100\%$$

Because environmental samples may contain interferences to analysis (i.e., other compounds that may interfere with the analysis of a specific analyte), the accuracy for a specific analyte will be evaluated in relation to the sample matrix. This will be done by analyzing MS/MSD samples for soils and waters and surrogate spikes for all sample matrices. Known concentrations of each target analyte are added to an aliquot of the sample for the MS/MSD. The difference between the concentration of the analyte in the unspiked sample and the concentration of the analyte in the spiked sample will be compared to the known amount of the spike added as a measure of accuracy in the sample matrix.

Surrogate spikes contain a known amount of compounds that are similar to the environmental compounds of interest but are not normally found in environmental samples. Surrogate spikes will be added to all field and QC samples for VOCs.

For the QC samples that measure the potential matrix effect on the accuracy of the result, including MS and MSD recoveries, the following equation will be used to calculate the potential bias in the result as a percent recovery of the spike added:

$$\%Recovery\ Accuracy/Bias = \frac{Spiked\ Sample\ Concentration - Unspiked\ Sample\ Concentration}{Spike\ Added} \bullet 100\%$$

Acceptance criteria for LCS/LCSD, Surrogate, and MS/MSD recoveries are listed in Table C.1.

3.3 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the site conditions. It takes into consideration the magnitude of the area represented by one sample and assesses the feasibility/reasonableness of that design rationale. Representativeness also reflects the ability of the sampling team to collect samples and laboratory personnel to analyze those samples in such a manner that the data generated accurately and precisely reflect the site conditions.

As a quantitative measure of representativeness, field duplicate samples will be collected and analyzed. See above section on precision for detailed approach to duplicate collection, analysis, and criteria. Overall representativeness will be assessed once the data are validated and reviewed to determine whether the sampling design was adequate for defining the site conditions.

3.4 Completeness

Completeness is a measure of the amount of valid/usable data resulting from data collection and analysis activities. Completeness can be calculated as a percentage of the number of valid/usable results obtained compared to the total number of results (usable and rejected) obtained during the course of the investigation. Theoretically, a completeness target is reached through adherence to the methods and QC requirements. Deficiencies in the data may be due to sampling techniques, poor accuracy or precision, or laboratory error. While these deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. The completeness objective for this

project is 90 percent (%) except if there are critical samples identified for which 100% completeness will be required.

3.5 Sensitivity

A list of six target VOCs has been identified for analysis for this RFI based on the rationale given in Section 3.1.2 of the RFI Work Plan. Sensitivity will be considered acceptable if non-detect data are reported accurately at levels at or below the Project Action Limits (PALs) given in Table C.2 for VOCs. Laboratory reporting limits (RLs) compared to PALs for VOCs are provided in Table C.4.

Sensitivity is the ability of the method or instrument to detect the target VOCs at the concentration of interest. Several QC samples and procedures will be used to provide sensitivity consistent with site DQOs. These include collection and analysis of field equipment and trip blanks, laboratory method blank samples, and instrument initial and continuing calibration criteria. The laboratory's lowest concentration initial calibration standard will be at a level at or below the project-required reporting limits, on a sample-equivalent basis. Adherence to method procedures, and field and laboratory instrument/equipment maintenance, testing, and inspection will also assist in providing the appropriate level of sensitivity.

Reporting limits for Method TO-15 analyses for a given sample are dependent on the sample residual vacuum at the time of receipt at the laboratory and upon sample-specific handling during TO-15 analysis. After the canister vacuum has been measured at the laboratory, the canister will be over-pressurized to approximately 5 pounds per square inch (psi) as a part of the sample preparation procedure, which will lead to an expected sample dilution factor of about 2.

For analysis of all matrices, if a compound is detected at a concentration above the instrument calibration range during analysis, a secondary dilution should be performed so that target compounds are quantitated within the accurate calibration range of the instrument.

If the laboratory identifies a target compound at a level below the reporting limit (equivalent to the sample-specific level associated with the lowest concentration initial calibration), they will report the result as an estimated value (flagged "J") due to uncertainty in quantitation at a level below the instrument calibration range. These results are qualitatively accurate and may be used for project decisions with the understanding of the uncertainty in the numerical value.

3.6 Comparability

Comparability is a qualitative parameter that expresses the confidence with which data sets can be compared. Comparable data allows for the ability to combine analytical results acquired from various sources (e.g., on-site and off-site laboratory analyses) taken during the period of the assessment. Comparability relies upon precision and accuracy within the individual data sets to be acceptable, to promote confidence in the data sets. The consistent use of the sampling and analytical methods defined in this plan will yield

comparable results. In addition, comparability can be affected by QA/QC criteria such as sample preservation, holding times, blank contamination, quantitation limits, and matrix issues. The QC criteria for these parameters have been defined in this plan to provide comparability of the data generated during the program. Refer to Table C.5 for a summary of container, preservation, and holding time requirements. Comparability of split/correlation samples from the on-site mobile lab and fixed lab will be evaluated as defined in Section 3.1, Precision.

4.0 SAMPLING AND FIELD MEASUREMENTS

4.1 Sampling Scope of Work and Procedures

Refer to Section 3.0 of the RFI Work Plan for a description of the scope of work. The following information for soil, bedrock, groundwater, and soil vapor sampling and analysis can be found in the referenced tables and figures from the RFI Work Plan:

- Proposed temporary soil vapor implant and boring locations (Figures 6 and 7);
- Summary and rationale for proposed boring locations (Table 1);
- Proposed sample depths (Table 1);
- Proposed analytical samples by matrix and location (Table 2);
- Number of analytical samples to be collected (Table 2);
- Summary of sample containers and preservation (Table C.5 of QAPP); and
- Field sample collection procedures (RFI Work Plan Section 3.0 and Appendix B).

4.2 Field QC Sampling Plan

The field QC sampling plan was developed in general accordance with New York State Department of Environmental Conservation DEC Program Policy, DER-10 / Technical Guidance for Site Investigation and Remediation (DER-10, May 3, 2010). The types and frequency of field QC samples described in the following sections are included in Table C.3, the required container, preservation, and holding time for the QC samples are included in Table C.5, and MPC are provided in Table C.1.

4.2.1 Mobile Laboratory Analysis

The Pace Analytical on-site mobile laboratory will analyze soil and bedrock samples for the project list of six VOCs. The following QC samples will be collected:

- Field duplicate samples;
- Split (correlation) samples for off-site fixed laboratory analysis;
- MS/MSD samples; and

- Equipment Rinseate Blanks (EB) for bedrock samples.

Field duplicate and mobile/fixed laboratory split samples will be collected without bias (e.g., will not select only detects for split samples). Field duplicate and split samples will be performed on approximately every tenth sample collected, selected in the order they are collected and presented for analysis. To ensure sufficient sample volume for the analyses, field duplicates will be collected initially on the 9th sample and thereafter on every tenth sample (e.g., field duplicates collected on the 9th sample, 19th sample, 29th sample, etc.) while split samples will be collected on every tenth sample (e.g., splits collected on 10th sample, 20th sample, 30th sample, etc.).

Samples to be used for contaminant delineation (e.g., to confirm the vertical extent of contamination) may be substituted for the split samples described above as needed.

For the bedrock samples, the mobile lab will prepare (crush) and extract (microwave) both the primary (i.e., for mobile laboratory analysis) and split (i.e., for fixed laboratory analysis) samples. After the samples undergo microwave extraction, bedrock split extracts will be transferred to the fixed laboratory for analysis. Procedures for bedrock crushing and extraction are provided in the RFI Work Plan Appendix B.4. If soil samples are extracted by the microwave extraction method, the split samples for fixed laboratory analysis will be provided as extracts.

For bedrock samples, equipment blanks will be collected and analyzed (as described in Pace SOP CORE DFN included in Appendix B.4 of the RFI Work Plan) at a frequency of 1 per 20 samples to assess the decontamination of the crushing equipment used to prepare the bedrock samples. Extracts of the EBs associated with the split samples will be transferred to the fixed laboratory along with the sample extracts. Note that though method blanks associated with the mobile lab extraction of bedrock are not "field QC", splits of a percentage of the method blanks associated with bedrock samples will also be transferred to the fixed laboratory for analysis to be associated with the split samples.

4.2.2 Fixed Laboratory Analysis for VOCs

The fixed laboratory (Lancaster and Eurofins Air Toxics [soil vapor]) will analyze split soil samples, split bedrock extract samples (and associated EBs and method blanks provided by the mobile laboratory), groundwater samples, and soil vapor samples for the project list of six VOCs. As shown in Table C.3, the following QC samples will be collected for fixed laboratory analysis:

- Field duplicate samples;
- Split (correlation) samples (split from mobile laboratory samples) for soils and bedrock only;
- MS/MSD samples;
- EBs for split bedrock samples and groundwater only; and

- Trip blanks.

Refer to Table C.3 for the type and frequency of field QC samples for each matrix.

4.2.3 Fixed Laboratory Analysis for Physical Parameters

Soil and bedrock samples will be submitted to Lancaster for moisture content and TOC, and to Geotesting Express for grain size, bulk density, and porosity. Analytical methods are listed in Table 2 of the RFI Work Plan. Bedrock samples will be crushed by the mobile laboratory prior to transfer to Lancaster for moisture content and TOC analysis. See QAPP Table C.5 for containers, preservation, and holding times for the physical parameters.

Based on the purpose of the physical parameters testing as described in the RFI Work Plan Section 3.5.1.2, no additional field or lab QC samples have been defined for these parameters (other than already performed by the laboratories as included in the methods and SOPs).

4.3 Decontamination Procedures

A description of decontamination procedures is included in Section 3.6 of the RFI Work Plan and further details can be found in the mobile lab SOPs included in Appendix B.4. Possible contamination from sample collection will be evaluated through collection of equipment rinsate blanks, as defined above, for bedrock samples and during groundwater sampling. The equipment rinsate blanks associated with collection of bedrock samples will be qualitatively compared to bedrock sample data since non-matrix matched equipment rinsate blanks are not quantitatively accurate. Matrix-matched trip blanks will be used to evaluate possible cross-contamination during the sample collection, handling, and shipping procedures.

4.4 Sampling Documentation and Tracking

The collection of field samples (including field duplicates and other field QC samples) will be documented with the use of media-specific field sampling summary forms. Information recorded on the forms generally includes:

- Identification of sample;
- Date and time of sample collection (for TO-15, start and stop dates/times required);
- Identity of sample collector(s);
- Weather conditions at the time of sample collection;
- Sampling equipment and sample containers (e.g., type, serial number) used;
- Field parameters measured (e.g., differential pressure, pH, etc.); and
- Starting and ending vacuums of SUMMA canisters, if applicable.

Information recorded on soil/bedrock boring logs generally includes:

- Location identification;
- Start and finish dates;
- Drilling and soil/rock sampling methods;

- Drilling company and foreman;
- Identity of field personnel observing the drilling;
- Depth to groundwater measurements;
- Field screening (e.g., PID); and
- Monitoring well construction materials.

The collection, transfer of custody, and shipping/transport of the samples to the mobile and fixed analytical laboratories will be documented using chain-of-custody (COC) forms. Information recorded on the COC form will include:

- Unique sample identification for primary and field QC samples;
- Sample matrix and numbers of container per sample (extra containers needed for MS/MSD analysis);
- Date and time of sample collection (for TO-15, start and stop dates/times required);
- Identity of sample collector(s);
- Starting and ending vacuums of SUMMA canisters, if applicable;
- Requested analyses;
- Sample preservation method;
- Extraction information (prep factors) for bedrock split samples that are prepared by the field lab and transferred to the fixed lab as extracts; and
- Additional notes or comments pertinent to analysis of the samples (e.g., need to perform MS/MSD on additional aliquots of sample).

PIDs and water quality meters will be calibrated at the beginning of each day and will be documented on a calibration field form or in a field notebook.

5.0 SAMPLE ANALYSIS

5.1 Laboratory Sample Management

When samples are received at the on-site mobile or fixed laboratory, the laboratory sample custodian will accept receipt of the samples and execute the COC by signing and dating it. The custodian will document the condition of the samples upon receipt and will ensure that all samples listed on the COC were received and that the sample ID for each sample container matches the ID listed on the COC. Any deviations for sample receipt will be brought to Sanborn Head's attention immediately so that corrective action can occur.

For fixed laboratory analysis, each sample will be logged into the Laboratory Information Management System (LIMS) that will assign a Sample Delivery Group (SDG) or Work Order number to the samples. The SDG or Work Order may contain only samples received in one shipment to the laboratory or may contain samples that are received within a few days of each other (i.e., the SDG or Work Order may open when one shipment comes to the laboratory and close once 20 field samples have been received, as long as doing so does not jeopardize the sample holding times). Each sample in the SDG or Work Order will be assigned a unique laboratory ID that is used by the laboratory to assign work and can be used to track the sample progress through laboratory processing of the sample. Analytical data are reported on an SDG or Work Order basis.

5.2 Laboratory Analytical Procedures

The laboratory SOPs provided in Attachment 1 give specific details on the analytical methods that will be used to analyze samples. Table C.1 indicates the project-specific Measurement Performance Criteria for VOCs that must be adopted by the laboratories for analysis. The SOPs give specifications for sample preparation and analysis and indicate the laboratory criteria for method required QC samples (e.g., frequency and acceptance of method blanks, LCS/LCSD, MS/MSD, calibration verification, etc.). Corrective action measures are also discussed in the laboratory SOPs.

Groundwater and soil vapor samples will be submitted for analysis to a NYSDOH ELAP-certified fixed laboratory. In addition, 10% of the soil and bedrock samples from the on-site mobile laboratory will be split and submitted to an ELAP-certified fixed laboratory. NYSDOH ELAP certification ensures that the laboratories performing VOC analysis are proficient in the methods for which they hold certification and have successfully passed proficiency testing for those methods. NYSDOH ELAP also requires that laboratories have formal Quality Assurance (QA) practices in place to ensure that testing is done properly and that the testing is fully documented.

Each NYSDOH ELAP-certified laboratory has a QA Officer who performs independent oversight of the laboratory operations. If a problem occurs during analysis of a sample, the analyst will inform their supervisor and corrective action will occur based on the requirements of the laboratory SOPs. The QA Officer will perform routine internal QA audits and will handle corrective actions that are beyond the scope of the analytical methods.

The first-level review of the analytical data occurs at the bench-level with the analyst evaluation of the gas chromatograph/mass spectrometer (GC/MS) results for accuracy (e.g., verifying that all detects fall within established retention time windows and that mass spectra of detects in sample match mass spectra from authentic standards). During this process, the QC DQIs for the analytical batch are reviewed and their results compared to project-specific criteria (see Table C.1). For any QC exceedance, corrective action occurs as necessary, as indicated in the laboratory SOPs. A second-level review of the data occurs at the Supervisor level before it is approved for reporting to the client.

A full data deliverable (e.g., NYSDEC Category B or equivalent) and Electronic Data Deliverable (EDD) in NYSDEC EQuIS version 4 format will be required for the fixed laboratory data for VOCs only. Physical parameters will be reported as a reduced deliverable. The on-site mobile laboratory will provide data in EDDs and will maintain records for the instrument controls.

6.0 DATA USABILITY SUMMARY REPORT

A NYSDEC Data Usability Summary Review (DUSR), as outlined in Appendix 2B of DER-10, "Technical Guidance for Site Investigation and Remediation" (May 2010), will be performed by New Environmental Horizons, Inc. (NEH) for soil, bedrock, and groundwater samples analyzed by the fixed laboratory for VOCs, as these may provide information on the final

delineation of the nature and extent of contamination, and/or are being analyzed as mobile laboratory correlation samples. A DUSR will not be prepared for soil vapor data, since that data is primarily intended to assess the distribution of VOCs in the subsurface, VOC mass calculations, and remedial alternatives assessments. A DUSR will not be prepared for the physical parameters either, based on the purpose of these measurements as defined in the RFI Work Plan.

The following QC elements, as applicable to the VOC analytical method, will be reviewed during this DUSR:

- Data package completeness and reporting protocols;
- Sample receipt, holding times, and preservation criteria;
- Calibration criteria (instrument tuning, initial and continuing calibration verifications);
- Method, field, and trip blank results;
- LCS and LCSD recoveries and RPDs;
- Surrogate or IS Recoveries;
- Sample/ LD, sample/ FD, and split sample RPDs;
- Sample result reporting (including reporting limits and units);
- Other method-specific QC if applicable and reported (e.g., internal standard areas);
- Selected result calculation verification; and
- Deficiencies or protocol deviations as noted in the Laboratory Narrative.

A Data Validation (DV) Checklist for each SDG or Work Order and for each matrix for VOCs will be completed to document the review and will explain the QC reviewed and any DV actions and professional judgments that may have been taken during the validation. Validation actions will also be documented in the EDD. The following data qualifiers will be applied in the EDD during data validation by NEH. Potential impacts on project-specific data quality objectives will be discussed in the DUSR.

Data Qualifier	Definition
U	The analyte is non-detect at or above the sample-specific reporting limit.
J	The result is an estimated quantity with indeterminate bias. The result is usable for project decisions.
J+	The result is an estimated quantity with possible high bias. The result is usable for project decisions.
J-	The result is an estimated quantity with possible low bias. The result is usable for project decisions.
UJ	The non-detect is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample result is rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

The DUSR will answer the seven questions required by DER-10 and summarize the actions taken during the review. The complete NYSDEC DUSR consists of this summary report, the DV Checklist, and validated EDD for each SDG or Work Order for VOCs.

TABLES

TABLE C.1

DATA QUALITY INDICATORS AND MEASUREMENT PERFORMANCE CRITERIA

TABLE C.1
Data Quality Indicators and Measurement Performance Criteria for Target VOCs
Site-Specific QAPP
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Matrix	Analytical Method	DQI	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
Soil, Bedrock, & Groundwater	8260C	Accuracy/Sensitivity	Results < RL	Method Blank, Trip Blank, and Equipment Blanks
		Accuracy/Precision	LCS &LCSD %Rec: 70-130% LCS/LCSD RPD ≤ 20%	LCS/LCSD
		Accuracy	Surrogate %Rec: 70-130%	Surrogates
		Precision	RPD ≤ 30% for values ≥ 2x RL; for values < 2x RL, use professional judgment	Lab Duplicate, if Split sample or Field Duplicate not available
Soil & Bedrock	8260C	Accuracy/Precision	MS&MSD %Rec: 70-130% MS/MSD RPD ≤ 30%	MS/MSD
		Precision/Representativeness	RPD ≤ 30% for values ≥ 2x RL; for values < 2x RL, use professional judgment	Split Samples
		Precision/Representativeness	RPD ≤ 30% for values ≥ 2 x RL; for values < 2x RL, use professional judgment	Field Duplicates
Groundwater	8260C	Accuracy/Precision	MS&MSD %Rec: 70-130% MS/MSD RPD ≤ 20%	MS/MSD
		Precision/Representativeness	RPD ≤ 30% for values ≥ 2x RL; for values < 2x RL, use professional judgment	Field Duplicates
Soil Vapor	TO-15	Accuracy	Tracer Gas < RL	Tracer Gas
		Accuracy/Sensitivity	Results < RL	Method Blank
		Accuracy/Precision	LCS &LCSD %Rec: 70-130% LCS/LCSD RPD ≤ 20%	LCS/LCSD
		Accuracy	Surrogate & IS %Rec: 70-130%	Surrogates & Internal Standards
		Precision	RPD ≤ 30% for values ≥ 5x RL; for values < 5x RL, use professional judgment	Lab Duplicate, if Field Duplicate not available
		Precision/Representativeness	RPD ≤ 30% for values ≥ 5x RL; for values < 5x RL, use professional judgment	Field Duplicates
All Matrices	All Methods	Completeness	>90% samples collected that are planned (100% for critical samples) >90% laboratory analyses are usable (100% for critical samples)	Field Activity Review & Data Validation

Abbreviations/Acronyms:
MS = matrix spike sample
MSD = matrix spike duplicate sample
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
IS = internal standards
RPD = relative percentage difference
RL = reporting limit
%Rec = percent recovery

TABLE C.2

TARGET COMPOUND LIST AND PROJECT ACTION LIMITS (PALs)

TABLE C.2
Target Compound List and Project Action Limits (PALs)
Site-Specific QAPP
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

		Soil Vapor			Soil[3]		Groundwater[4]
Target Compound	**CAS Number**	**2017 NYSDOH Categorization[1]**	**Soil Vapor Action Limit (µg/m^3)[1]**	**USEPA VISLs (Commercial ELCR 1E-6 HI 1.0)[2]**	**6 NYCRR Part 375 Restricted Use SCO, Protection of Groundwater (µg/Kg)**	**6 NYCRR Part 375 Restricted Industrial SCO (µg/Kg)**	**NYS Ambient Water Quality Standards, Water Class GA (TOGS 1.1.1) (µg/L)**
Tetrachloroethene (PCE)	127-18-4	Matrix B	1,000	1,570	1,300	300,000	5
Trichloroethene (TCE)	79-01-6	Matrix A	60	99.7	470	400,000	5
Dichloroethene (cis-1,2-)	156-59-2	Matrix A	60	--	250	1,000,000	5
Dichloroethene (trans-1,2-)	156-60-5	--	--	--	190	1,000,000	5
Dichloroethene (1,1-)	75-35-4	Matrix A	60	29,200	330	1,000,000	5
Vinyl Chloride (VC)	75-01-4	Matrix C	60	92.9	20	27,000	2

[1] New York State Department of Health (NYSDOH), Guidance for Evaluating Soil Vapor Intrusion in the State of New York, final October 2006 with updated Soil Vapor/Indoor Air Decision Matrices, May 2017.

[2] USEPA Vapor Intrusion Screening Levels (VISLs) were calculated using the Vapor Intrusion Screening Level Calculator (https://www.epa.gov/vaporintrusion/vapor-intrusion-screening-level-calculator; hazard quotient: 1.0; target risk: 10-6, commerical exposure assumptions).

[3] Soil Cleanup Objectives (SCOs) from 6 NYCRR Part 375 Environmental Remediation Programs Subpart 375-6, NYSDEC, Table 375-6.8(b) Restricted Use SCOs (SCOs effective December 14, 2006).

[4] NYS Division of Water Technical and Operational Guidance Series 1.1.1 (TOGS 1.1.1), Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, 1998 with updates through 2004.

TABLE C.3

FIELD QC SAMPLE FREQUENCY

TABLE C.3
Field QC Sample Frequency for Target VOCs
Site-Specific QAPP
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Field QC Sample Type	Frequency			
	Bedrock	**Soil**	**Groundwater**	**Soil Vapor**
Mobile Laboratory Analyses				
Field Duplicate (FD)	1 per 10	1 per 10	–	–
Matrix Spike (MS) / MS Duplicate (MSD)	1 set per 20 field samples	1 set per 20 field samples	–	–
Mobile Lab Split/Correlation (To Fixed Lab)	1 per 10	1 per 10	–	–
Equipment Rinseate Blanks (EBs)	1 per 20	-	–	–
Fixed Laboratory Analyses				
FD	1 per 10	1 per 10	1 per 10	1 per 10
MS / MSD	1 set per 20 field samples	1 set per 20 field samples	1 set per 20 field samples	–
Equipment Rinseate Blanks (EBs)	1 per 20 associated with split bedrock samples	-	1 per day	–
Trip Blanks (TBs)	1 per cooler containing VOC samples	1 per cooler containing VOC samples	1 per cooler containing VOC samples	–

Notes:

1. Split/correlation samples will be collected from mobile lab samples and sent to the fixed lab for analysis. For bedrock samples, mobile lab will prepare (crush) and extract (microwave) samples and submit extract as split sample to fixed lab for analysis. ***Along with bedrock field split samples, the mobile lab will provide the associated method blank extract and the associated equipment blank extract to the fixed lab for analysis.***

2. Frequency for fixed lab QC is based on number of samples sent for fixed laboratory analysis.

TABLE C.4

SUMMARY OF LABORATORY REPORTING LIMITS

TABLE C.4
Summary of Laboratory Reporting Limits & PALs for Target VOCs
Site-Specific QAPP
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Analyte	Cas No	Groundwater			Soil / Bedrock				Soil Vapor	
		PALs	Laboratory RLs		Soil PALs	Soil / Bedrock Fixed Laboratory RLs			PALs	Laboratory RLs
		NYS Ambient WQ Stds Class GA TOGS 1.1.1 (µg/L)	25ml Purge 8260C MDLs (µg/L)	25ml Purge 8260C RLs (µg/L)	6 NYCRRC Part 375 Restricted Use SCOs Protection of GW - *Unless Otherwise Noted* (µg/Kg)	8260C High Level (Methanol) MDLs (µg/kg)	8260C High Level (Methanol) RLs (µg/kg)	8260 Soil / Bedrock Mobile Laboratory RLs (µg/kg)	NYSDOH Soil Vapor Decision Matrices Action Limits 2017 (µg/m3)	TO-15 1 L canister (µg/m3)
Dichloroethene (1,1-)	75-35-4	5	0.06	0.5	330	25	250	20 - 40	60	4.8
Dichloroethene (cis-1,2-)	156-59-2	5	0.05	0.5	250	25	250	20 - 40	60	4.8
Dichloroethene (trans-1,2-)	156-60-5	5	0.06	0.5	250 (Note 4)	25	250	20 - 40	--	4.8
Tetrachloroethene (PCE)	127-18-4	5	0.06	0.5	1,300	25	250	20 - 40	1,000	8.2
Trichloroethene (TCE)	79-01-6	5	0.06	0.5	470	25	250	20 - 40	60	6.5
Vinyl Chloride	75-01-4	2	0.1	0.5	250 (Note 4)	30	250	20 - 40	60	3.1

1. This table summarizes the proposed analytes along with respective method detection limits (MDLs) and laboratory reporting limits (RLs).

   MDLs/RLs for United States Environmental Protection Agency (USEPA) Method 8260C for groundwater and soil were provided by Eurofins Lancaster Laboratories of Lancaster, Pennsylvania.

   RLs for USEPA Method TO-15 were provided by Air Toxics, Ltd. of Folsom, California. The RLs are based upon a presumed dilution factor of 2.5.

   RLs for USEPA Method 8260 Soil / Bedrock Mobile Laboratory were provided by Pace Analytical Mobile Lab Services on August 26, 2019.

2. Actual MDLs/RLs may be higher due to procedures associated with the preparation of field samples for analysis and will be a function of the actual sample volume provided to the laboratory, matrix effects, and other factors including dry-weight conversion for soil / bedrock.

3. See Table C.2 for full references for Project Action Limits (PALs) based on applicable regulatory standards. There are no PALs set for bedrock for this project.

4. The PALs for trans-1,2-DCE and vinyl chloride in soil were set at the laboratory reporting limit.

TABLE C.5

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

TABLE C.5
Sample Containers, Preservation, and Holding Times
Site-Specific QAPP
B335 Area RFI Work Plan
Former IBM East Fishkill Facility
Hopewell Junction, New York

Matrix	Method	Container(s)	Preservation	Preparation Holding Time	Analytical Holding Time	Analytical Standard Turn Around Time
Soil	8260C	1-40 mL pre-weighed vial w/ PTFE-faced silicone septum containing methanol	~1:1 ratio soil:preservative collected with soil completely covered by methanol preservative (High-level VOCs), ≤ 6°C	NA	14 days	10 business days
Bedrock	8260C	1-40 mL pre-weighed vial w/ PTFE-faced silicone septum containing methanol	Mobile lab: ~1:1 ratio bedrock:preservative (~20g crushed bedrock:15mL methanol preservative) prepared with microwave extraction Fixed Lab Split: extract containerized in 1-40 mL vial, ≤ 6°C	14 days to extraction	14 days from extract preparation to analysis	10 business days
Bedrock Equipment Blank	8260C	1-40 mL pre-weighed vial w/ PTFE-faced silicone septum containing methanol	Mobile lab: equip. wipe:15mL methanol preservative prepared with microwave extraction Fixed Lab Split: extract containerized in 1-40 mL vial, ≤ 6°C	14 days to extraction	14 days from extract preparation to analysis	10 business days
Soil / Bedrock Trip Blank	8260C	1-40 mL pre-weighed vial w/ PTFE-faced silicone septum containing methanol	≤ 6°C	NA	14 days	10 business days
Soil / Bedrock	TOC Lloyd Kahn	1-4 oz. glass jar [mobile lab will crush bedrock samples]	≤ 6°C	NA	28 days	10 business days
	Moisture / % Solids SM2540				7 days	10 business days
Soil	Grain Size Bulk Density Porosity (ASTM & Lab SOPs)	1-gal plastic ziplock bag or equivalent	NA	NA	NA	10 business days
Bedrock	Bulk Density & Porosity (ASTM & Lab SOPs)	Wrap in foil & plastic wrap; 1-gal plastic ziplock bag or equivalent	NA	NA	NA	10 business days
GW	8260C	3-40 mL VOA vials w/ PTFE-faced silicone septum	HCl, ≤ 6°C No headspace in vials. If effervescence occurs on contact with HCl, collect unpreserved.	NA	pH >2: 7 days pH <2: 14 days	10 business days
Water Equipment Blank	8260C	2-40 mL VOA vials w/ PTFE-faced silicone septum	HCl, ≤ 6°C No headspace in vials.	NA	pH >2: 7 days pH <2: 14 days	10 business days
Water Trip Blank	8260C	2-40 mL VOA vials w/ PTFE-faced silicone septum	HCl, ≤ 6°C No headspace in vials.	NA	pH >2: 7 days pH <2: 14 days	10 business days
Soil Vapor	TO-15	Batch-certified 1-L Summa canister	Vacuum prior to sample collection > 25 "Hg; Lab Receipt Vacuum < 15 "Hg, & Lab Receipt Vacuum < ± 5 "Hg of Final Field Vacuum	NA	30 days	10 business days

Abbreviations/Acronyms:
GW = Groundwater
VOA/VOC = volatile organic compound
PTFE = polytetrafluoroethylene (Teflon)
HCl = hydrochloric acid
mL = milliliter
oz = ounce
"Hg = inches mercury
°C = degrees Celsius

ATTACHMENT A

LABORATORY STANDARD OPERATING PROCEDURES

ANALYTICAL METHODS

Section 11.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds (Standard/Quad)

Eurofins Air Toxics SOP #6 Revision 38 Effective Date: May 9, 2018 Methods Manual Summary

Description: This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 0.2 liters of air is withdrawn from the canister utilizing a volumetric syringe, volumetric loop, or mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a secondary trap for further concentration and/or directly onto a GC/MS for separation and detection.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 (Standard or Quad).

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 0.5 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics takes no modifications of technical significance to Method TO-15 for the "Quad" configurations. Since Eurofins Air Toxics applies TO-15 methodology to all Summa canisters regardless of whether TO-14A or TO-15 is specified by the project, the laboratory performs a modified version of method TO-14A as detailed in Table 1. Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not

recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

Table 1. Summary of TO-14A Method Modifications

Requirement	TO-14A	Eurofins Air Toxics Modifications
Sample Drying System	Nafion Drier	Multibed hydrophobic sorbent.
Blank acceptance criteria	≤ 0.2 ppbv	≤ RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB	CCV internal standard area counts are compared to ICAL; corrective action when recovery is less than 60%.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	Follow TO-15 requirements of ≤ 30% RSD with 2 of Eurofins Air Toxics' 62 standard compounds allowed out to ≤ 40% RSD

The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 5.

Table 2. Method TO-14A/TO-15 Analyte List (Quad)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trichlorobenzene	2.0	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane (EDB)	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloropropane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3,5-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Benzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Bromomethane	5.0	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Carbon Tetrachloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	2.0	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Chlorotoluene (Benzyl Chloride)	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dichloromethane (Methylene Chloride)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Ethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 11 (Trichlorofluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 113 (Trichlorotrifluoroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexachlorobutadiene	2.0	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Styrene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Toluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Butadiene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dioxane	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Butanone (Methyl Ethyl Ketone)	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Hexanone	2.0	≤ 30%	70 – 130	70 – 130	± 25
4-Ethyltoluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
4-Methyl-2-Pentanone (MIBK)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Acetone	5.0	≤ 30%	70 – 130	70 – 130	± 25
Bromodichloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Bromoform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Carbon Disulfide	2.0	≤ 30%	70 – 130	70 – 130	± 25
Cyclohexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dibromochloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Ethanol	2.0	≤ 30%	70 – 130	70 – 130	± 25
Heptane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Isopropanol	2.0	≤ 30%	70 – 130	70 – 130	± 25
Methyl t-Butyl Ether (MTBE)	2.0	≤ 30%	70 – 130	70 – 130	± 25
Tetrahydrofuran	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
2,2,4-Trimethylpentane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Cumene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Propylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
3-Chloroprene	2.0	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene*	1.0	≤40%	60 – 140	60 – 140	± 25
TPH (Gasoline) **	50	1-Point Calibration	N/A	ICV only; 60 – 140	± 25
NMOC (Hexane/Heptane)**	10	1-Point Calibration	N/A	NA	± 25

*Due to its low vapor pressure, Naphthalene may exceed TO-15 performance requirements. The wider QC limits reflect typical performance. Although Naphthalene is not on Eurofins Air Toxics "standard" TO-15 list, it is commonly requested and included in Table 2.

**TPH and NMOC are not on Eurofins Air Toxics' "standard" TO-15 list, but are included in Table 2 due to common requests.

Table 2 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request.

Table 3. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table 4. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 5. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample analysis	% RSD ≤ 30 with 2 compounds allowed out to ≤ 40% RSD	Correct problem then repeat Initial Calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each Initial Calibration curve, and daily prior to sample analysis	Recoveries for 85% of "Standard" compounds must be 70–130%. No recovery may be < 50%. ICV evaluated on a full list basis at the time of calibration. If specified by the project, in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard compounds	Per client request or specific project requirements only	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each analytical clock after the tune check	70–130%	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70–130% or > 10% of VOCs if short list is used (20 compounds or less), corrective action will be taken. If any compound exceeds 60–140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV) for Non-standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60–140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	Inspect the system and re-analyze the blank. "B"-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogates	As each standard, blank, and sample is being loaded	70–130% If specified by the project, in-house generated control limits may be used.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound has >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

Technical Information

Reference Number:	---
Test Method Title:	Bulk Density of Soil
Test Property:	density
Test Specimen Size:	varies depending on largest grain size and type of soil
Number of Test Specimens:	1
Test Equipment:	Drying oven capable of maintaining 110+/- 5° C Balance Containers of known volume of various sizes

Standard Operating Procedure

1. Determine the mass of an appropriate size cylinder to the nearest 1 gram.

2. Place soil into a mixing tub and mix until soil is homogenous. Place the soil in three equal lifts into the cylinder, tamping each lift 10 times with a tamper.

3. Determine the mass of the cylinder and soil to the nearest 1 gram.

4. Obtain a representative sample of soil for moisture content determination (in accordance with ASTM D 2216).

5. Calculate dry mass of the soil sample in grams with the following equation:

   $M_3 = [(M_1-M_2)/(1 + w/100)]$

   where:

   M_1 = mass of the cylinder and wet soil sample, g
   M_2 = mass of the cylinder, g
   M_3 = mass of the dry soil sample, g
   w = moisture content, % as dry mass basis

6. Calculate the dry density of the sample in lbs/ft^3 as follows:

   $\rho_d = (M3/V)(3.810)$

   where:

   ρ_d = dry density of soils, lbs/ft^3
   V = volume of the cylinder, in^3

7. Report: sample identification, dry density, moisture content, dimensions and volume of cylinder, and visual description of soil.

Technical Information

Reference Number:	ASTM D854-14
Test Method Title:	Standard Test Method for Specific Gravity of Soil Solids by Water Pycnometer
Test Property:	Specific gravity
Test Specimen Size:	Specific gravity of soil passing the #4 sieve Specimen dry mass in grams when using a 500 ml pycnometer: SP, SP-SM 100 ± 10 SP-SC, SM, SC 75 ± 10 Silt or clay 50 ± 10
Number of Test Specimens:	1 (representative sample obtained by quartering)
Test Equipment:	500 ml volumetric pycnometer Drying oven capable of maintaining 110 ± 5 °C Balance readable to 0.01 gram with 1,000 gram capacity Thermometer readable to 0.1 °C System to apply heat and/or agitation Vacuum system Mortar and pestle Funnel Distilled water De-aired distilled water Squirt bottle Dessicator Insulated container

Standard Operating Procedure

METHOD A - Procedure for Moist Specimens

1a. Check the dry weight of your pycnometer against its calibrated dry weight. If it is off by more than 0.06g do not use pycnometer until a recalibration is performed. Determine recommended dry mass for test specimen. Collect sufficient wet mass of sample to satisfy dry mass criteria. Add soil to 100 ml of distilled water. Disperse the soil in the distilled water using the blender. Using a funnel, pour the slurry into a calibrated pycnometer. Rinse any soil particles remaining on the funnel into the pycnometer using a wash/spray squirt bottle of distilled water. Add distilled water into the pycnometer until the water level is about ½ the depth of the main body of the pycnometer. Rinse any soil adhering to the inside neck of the pycnometer into the slurry.

Proceed to Step 2.

METHOD B – Procedure for Oven-Dried Specimens

1b. Check the dry weight of your pycnometer against its calibrated dry weight. If it is off by more than 0.06g do not use pycnometer until a recalibration is performed. Dry the specimen to a constant mass in an oven maintained at 110+/- °5C. Break up any clods of soil using a mortar and pestle. If the soil will not easily disperse after drying or has changed composition, use Test Method A. Place funnel into pycnometer. The stem of the funnel must extend past the calibration mark. Spoon the soil solids directly into the funnel. Record the weight of the dry soil solids in the pycnometer. Add water until the water level is about ½ the depth of the main body of the pycnometer. Agitate the water until a slurry is formed. Rinse any soil adhering to the inside neck of the pycnometer into the slurry.

Proceed to Step 2.

2. Entrapped air needs to be removed by using a vacuum, boiling or both vacuum and boiling. The pycnometer needs to be continually agitated for at least two hours under vacuum or boiling alone. (This means the silt/clay particles need to remain in suspension and slurry is in constant motion.) The vacuum must remain relatively constant and be sufficient to cause bubbling at the beginning of the de-airing process. If both boiling and vacuum method is used, this must last a minimum of 1 hour.

3. Add de-aired distilled water to the pycnometer until it nearly reaches the calibration mark. Place pycnometer(s), a bottle of de-aired distilled water and thermometer in a closed insulated container and allow them to reach thermal equilibrium overnight. When thermal equilibrium is achieved, remove pycnometer by only touching the top and placing it on an insulated surface near the scale you will be using to weigh it on. Top the pycnometer up so the calibration mark is at the mid-height of the meniscus of the water in the pycnometer. Wipe drips above the calibration mark on the inside of the pycnometer and wipe and dry the entire outside of the pycnometer. Measure and record the mass of pycnometer, soil, and water. Measure and record the temperature of the slurry/soil-water mixture.

4. For both Method A and B –Weigh out an empty, clean dish for drying specimen in oven. Transfer the slurry to a dish using distilled water (be sure all soil particles are transferred from pycnometer to dish). Dry the specimen to a constant mass in an oven maintained at 110+/- 5 ºC. After the soil is oven-dried record the dry weight of soil and dish. Calculate and record the mass of dry solids to the nearest 0.01g.

5. Calculate the mass of the pycnometer and water at the test temperature:

$M_{pwt} = M_p + (V_p \bullet P_{wt})$

Where:

M_{pwt} = Mass of the pycnometer and water at the test temperature
M_p = the average calibrated mass of the dry pycnometer
V_p = the average calibrated volume of the pycnometer
P_{wt} = the density of water at the test temperature

6. Calculate the specific gravity as follows:

$G_t = M_s / (M_{pwt} – (M_{pwst} - M_s)$

Where:

G_t = specific gravity
M_s = weight of oven-dry soil, g
M_{pwt} = mass of water and pycnometer at test temperature
M_{pwst} = mass of water pycnometer, water, and soil solids at test temperature

7. Calculate the specific gravity of soil solids at 20 ºC as follows:

$$G_{20} = K \bullet G_t$$

Where:

K = the temperature coefficient given in Table 2

8. Report: sample identification, specific gravity at 20 °C to the nearest 0.01, test method used, maximum particle size of the specimen, if any portion of the original sample was not used for the test.

9. NOTE: If porosity is required, use results of a density test and specific gravity (ASTM D 854) to calculate as follows:

$$n = 1 - (\gamma_d / G \times \gamma_w)$$

Where:

n = porosity
γ_d = dry density of soil, lbs/ft^3
G = specific gravity of soil
γ_w = specific gravity of water

TABLE 2 Density of Water and Temperature Coefficient (K) for Various Temperatures[A]

Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)	Temperature (°C)	Density (g/mL)[B]	Temperature Coefficient (K)
15.0	0.99910	1.00090	16.0	0.99895	1.00074	17.0	0.99878	1.00057	18.0	0.99860	1.00039
.1	0.99909	1.00088	.1	0.99893	1.00072	.1	0.99876	1.00055	.1	0.99858	1.00037
.2	0.99907	1.00087	.2	0.99891	1.00071	.2	0.99874	1.00054	.2	0.99856	1.00035
.3	0.99906	1.00085	.3	0.99890	1.00089	.3	0.99872	1.00052	.3	0.99854	1.00034
.4	0.99904	1.00084	.4	0.99888	1.00067	.4	0.99871	1.00050	.4	0.99852	1.00032
.5	0.99902	1.00082	.5	0.99886	1.00066	.5	0.99869	1.00048	.5	0.99850	1.00030
.6	0.99901	1.00080	.6	0.99885	1.00064	.6	0.99867	1.00047	.6	0.99848	1.00028
.7	0.99899	1.00079	.7	0.99883	1.00062	.7	0.99865	1.00045	.7	0.99847	1.00026
.8	0.99898	1.00077	.8	0.99881	1.00061	.8	0.99863	1.00043	.8	0.99845	1.00024
.9	0.99896	1.00076	.9	0.99879	1.00059	.9	0.99862	1.00041	.9	0.99843	1.00022
19.0	0.99841	1.00020	20.0	0.99821	1.00000	21.0	0.99799	0.99979	22.0	0.99777	0.99957
.1	0.99839	1.00018	.1	0.99819	0.99998	.1	0.99797	0.99977	.1	0.99775	0.99954
.2	0.99837	1.00016	.2	0.99816	0.99996	.2	0.99795	0.99974	.2	0.99773	0.99952
.3	0.99835	1.00014	.3	0.99814	0.99994	.3	0.99793	0.99972	.3	0.99770	0.99950
.4	0.99833	1.00012	.4	0.99812	0.99992	.4	0.99791	0.99970	.4	0.99768	0.99947
.5	0.99831	1.00010	.5	0.99810	0.99990	.5	0.99789	0.99968	.5	0.99766	0.99945
.6	0.99829	1.00008	.6	0.99808	0.99987	.6	0.99786	0.99966	.6	0.99764	0.99943
.7	0.99827	1.00006	.7	0.99806	0.99985	.7	0.99784	0.99963	.7	0.99761	0.99940
.8	0.99825	1.00004	.8	0.99804	0.99983	.8	0.99782	0.99961	.8	0.99759	0.99938
.9	0.99823	1.00002	.9	0.99802	0.99981	.9	0.99780	0.99959	.9	0.99756	0.99936
23.0	0.99754	0.99933	24.0	0.99730	0.99909	25.0	0.99705	0.99884	26.0	0.99679	0.99858
.1	0.99752	0.99931	.1	0.99727	0.99907	.1	0.99702	0.99881	.1	0.99676	0.99855
.2	0.99749	0.99929	.2	0.99725	0.99904	.2	0.99700	0.99879	.2	0.99673	0.99852
.3	0.99747	0.99926	.3	0.99723	0.99902	.3	0.99697	0.99876	.3	0.99671	0.99850
.4	0.99745	0.99924	.4	0.99720	0.99899	.4	0.99694	0.99874	.4	0.99666	0.99847
.5	0.99742	0.99921	.5	0.99717	0.99897	.5	0.99692	0.99871	.5	0.99665	0.99844
.6	0.99740	0.99919	.6	0.99715	0.99894	.6	0.99689	0.99868	.6	0.99663	0.99842
.7	0.99737	0.99917	.7	0.99712	0.99892	.7	0.99687	0.99866	.7	0.99660	0.99839
.8	0.99735	0.99914	.8	0.99710	0.99889	.8	0.99694	0.99873	.8	0.99657	0.99836
.9	0.99732	0.99912	.9	0.98707	0.99887	.9	0.99681	0.99860	.9	0.99654	0.99833
27.0	0.99652	0.99831	28.0	0.99624	0.99803	29.0	0.99595	0.99774	30.0	0.99585	0.99744
.1	0.99649	0.99628	.1	0.99621	0.99800	.1	0.99592	0.99771	.1	0.99562	0.99741
.2	0.99646	0.99825	.2	0.99618	0.99797	.2	0.99589	0.99768	.2	0.99559	0.99738
.3	0.99643	0.99822	.3	0.99615	0.99794	.3	0.99586	0.99765	.3	0.99556	0.99735
.4	0.99641	0.99820	.4	0.99612	0.99791	.4	0.99583	0.99762	.4	0.99553	0.99732
.5	0.99638	0.99817	.5	0.99609	0.99788	.5	0.99580	0.99759	.5	0.99550	0.99729
.6	0.99635	0.99814	.6	0.99607	0.99785	.6	0.99577	0.99756	.6	0.99547	0.99726
.7	0.99632	0.99811	.7	0.99604	0.99783	.7	0.99574	0.99753	.7	0.99544	0.99723
.8	0.99629	0.99808	.8	0.99601	0.99780	.8	0.99571	0.99750	.8	0.99541	0.99720
.9	0.99627	0.99806	.9	0.99598	0.99777	.9	0.99568	0.99747	.9	0.99538	0.99716

[A]Reference: CRC Handbook of Chemistry and Physics, David R. Lide, Editor-in-Chief, 74th Edition, 1993–1994.
[B]mL = cm^3.

Technical Information

Reference Number:	ASTM D2216-10
Test Method Title:	Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock Mass
Test Property:	Moisture content
Test Specimen Size:	As per test method based on maximum particle size
Number of Test Specimens:	1 or, as required by other testing representative based on type of sample: Undisturbed: mix sample by hand or take random samples throughout test sample, combine and mix Disturbed: take a representative cross-section of material for test
Test Equipment:	Oven capable of maintaining 60 or 110 ±5 °C Balance readable to 0.01 gram with 200 gram capacity or readable to 0.1 grams with >200 gram capacity Numbered specimen containers Desiccator

Standard Operating Procedure

1. Determine and record mass of a clean, dry specimen container.

2. Select a representative test specimen based on largest particle size. (See chart in test standard for guidance.) Place moist test specimen into container. Determine and record mass of container and moist test specimen using proper balance based specimen size.

3. Place the container with moist specimen in the oven at 110 °C (unless highly organic or gypsum, in which case use 60°C). Dry material to constant mass.

4. Once specimen has dried to constant mass, remove from oven and place into desiccator. Allow cooling to room temperature (or until container can be held comfortably in hands).

5. Measure and record mass of dried specimen and container with the same type of balance used to measure the before drying mass.

6. Calculate the moisture content of the material as follows:

$$w = [(M_{cws} - M_{cs}) / (M_{cs} - M_c)] \times 100$$

where:

w = moisture content, %
M_{cws} = mass of container and wet specimen, g
M_{cs} = mass of container and dry specimen, g
M_c = mass of container, g

7. Report: sample identification, moisture content of specimens to the nearest 0.1% or 1 % based on sample size used, description of material, temperature of drying oven if other than 110 °C, size and amount of material (if any) that was excluded from the test specimen.

Technical Information

Reference Number: ASTM D6913 Method A– Composite Sieving (Use for samples containing particles larger than a #4 sieve.)

Test Method Title: Standard Test Methods for Particle-Size Distribution Gradation) of Soils Using Sieve Analysis

Test Property: Grain Size Analysis for large bulk samples

Test Specimen Size: Depends on test samples maximum particle size. See ASTM D6913 Table 2 below. For bulk samples sample size is generally on the order of 25 to 70 kg (55 to 150 lbs.)

D 6913 – 04$^{\varepsilon 2}$

TABLE 2 Minimum Mass Requirement for Specimen

Maximum Particle Size of Material (99 % or more passes)		Minimum Dry Mass of Specimen, g or kg[A]	
Alternative Sieve Designation	Maximum Particle Size, mm	Method A Results Reported to Nearest 1 %	Method B Results Reported to Nearest 0.1 %
No. 40	0.425	50 g	75 g
No. 10	2.00	50 g	100 g
No. 4	4.75	75 g	200 g[B]
⅜ in.	9.5	165 g[C]	[D]
¾ in.	19.0	1.3 kg[C]	[D]
1 in.	25.4	3 kg[C]	[D]
1-½ in.	38.1	10 kg[C]	[D]
2 in.	50.8	25 kg[C]	[D]
3 in.	76.2	70 kg[E]	[D]

[A] Specimen masses should not significantly exceed (by more than about 50 %) the presented values because excessively large specimens may result in sieve overloading, (see 11.3) and increase the difficulty of specimen processing.

Number of Test Specimens: 1 representative sample obtained by quartering, mixing or splitting

Test Equipment:

Coarse Sieve Screen Set:
3 in., 2in., 1.5 in., 1 in., ¾ in., ½ in., 3/8 in., No 4
Fine Sieve Set:
No. 4, No. 10, No. 20, No. 40, No. 60, No.100, No. 140, No.200
Washing Sieve, No. 200 (75-µm)
Washing Sink with Spray Nozzle
Large Mechanical Screen Shaker
Mechanical Sieve Shaker
Balances (readable to 1 g and 0.01 g)
Drying Oven 110° ± 5 ° C
Sample and Specimen Containers
Sieve Brushes
Wash bottle
Spatula
Stirring Rod

Optional Items
Splitter/Riffle Box or Quartering equipment
Mortar and Rubber-Covered Pestle (for breaking up aggregations of dried soil.)
Low Temperature Drying Oven not to exceed 60°C
Sodium hexametaphosphate dispersant solution:
Make a solution by using 40 g of sodium hexametaphosphate and 1,000 g distilled, deionized, or demineralized water

Standard Operating Procedure

Sample Splitting:

1. Split sample on a No. 4 sieve on large screen shaker.
2. Record the wet weight of the minus No. 4 fraction.
3. Record the weight of an empty tare.
4. Collect a 100–200 g representative sub-sample of the minus No. 4 fraction and place it in the tare weighed in step 3 above. This sample will be used as a moisture content sample and the fine sieve test sub-specimen. Record the wet weight of the [sub-specimen + tare] and oven dry at 110° ± 5 ° C.

Coarse Sieves:

1. Oven-dry the plus No. 4 fraction from Sample Splitting step 1 at 110° ± 5 ° C.
2. Sieve the oven-dried plus No. 4 fraction on the large screen shaker using the standard coarse sieve set, which includes the following screen sizes:

   3 in., 2in., 1.5 in., 1 in., ¾ in., ½ in., 3/8 in., No 4
   Additional screens may be added as requested by the client.

3. Avoid overloading individual sieve screens. Refer to ASTM Table 3 below for maximum screen loading. If necessary, shake overloaded screens in multiple batches.

4. Cumulatively record the weights of the oven-dried material retained on each of the coarse sieves. Place the material, sieve by sieve, into a tared weighing container and record the cumulative weight. At least one sieve in the sieving process shall have 100 percent passing.

Fine Sieves:

1. Record the weight of the [oven-dried minus No. 4 sub-specimen + tare] dried in Sample Splitting step 4 above.

2. Disperse the minus No. 4 sub-specimen by soaking in water for at least 5 minutes before washing. Difficult samples can be dispersed using a sodium hexametaphosphate solution and/or an ultrasonic bath.

3. Wash the dispersed specimen on a #200 wash sieve, using tap water. Collect the wash water and dispose of the fine soil properly. Do not let the fines run down the drain.

   The material in the wash sieve may be lightly manipulated by hand, to facilitate the washing, taking care not to lose any of the soil. Do not exert any downward pressure on the wash sieve as this may damage the sieve.

   Fine material can clog the screen. Care should be taken not to let the wash sieve fill with too much water when clogging occurs. Prevent clogging by lightly tapping the sides of the sieve with your hand or tapping on the bottom of the screen with a fingertip.

4. Transfer the washed +200 in. fraction of soil back into the same tare and oven dry at $110^{o} \pm 5^{o}$ C.

5. Sieve the dried specimen using the standard fine sieve set, which includes the following sieve sizes:

   No. 4, No. 10, No. 20, No. 40, No. 60, No.100, No. 140, No.200

   Additional screens may be added as requested by the client.

6. Shake the sieve sample for a minimum 10 minutes. (The "Standard Shaking Period" is verified annually based on the procedure in ASTM D 6913.)

7. Avoid overloading individual sieves. Refer to ASTM Table 3 below for maximum sieve loading. If necessary, shake overloaded sieves in multiple batches.

8. Cumulatively record the weights of the oven-dried material retained on each of the fine sieves. Place the material, sieve by sieve, into a tared weighing container and record the cumulative weight. At least one sieve in the sieving process shall have 100 percent passing.

TABLE 3 Overloading Limits for Standard Sieve Set

Alternative Sieve Designation	Standard Sieve Designation	Number of Grain Layers on Given Sieve	Maximum Mass Retained on 200-mm (8-in.) Diameter Sieve, g[A]	Maximum Mass Retained on 305-mm (12-in.) Diameter Sieve, g	Maximum Mass Retained on 370- by 580-mm (14.6- by 22.8-in.) Sieve, g
3 in.	75 mm	0.8	2 700	6 100	18 000
2 in.	50 mm	0.9	2 000	4 500	13 000
1-½-in.	37.5 mm	0.9	1 500	3 400	10 000
1 in.	25 mm	1	1 100	2 500	7 000
¾ in.	19.0 mm	1	900	2 000	6 000
⅜ in.	9.5 mm	1.25	550	1 200	3 600
No. 4	4.75 mm	1.5	325	730	2 000
No. 10	2.00 mm	2	180	410	1 000
No. 20	850 µm	3	115	260	800
No. 40	425 µm	4	75	170	500
No. 60	250 µm	5	60	140	400
No. 100	150 µm	6	40	90	300
No. 140	106 µm	6	30	70	200
No. 200	75 µm	6	20	50	100

Calculations:

1. Use initial moisture content and initial wet weight of test specimen to calculate initial dry weight of test specimen. Use reporting software to enter data and calculate % passing and retained for each sieve size.

Report:

1. The datasheet shall include the following:

   - Project identification, boring number, sample number, depth,
   - Visual classification of the soil being tested
   - Test method used (Method A)
   - Sample preparation - moist, air dried, or oven dried, etc.
   - Any material excluded from the specimen.
   - Indicate that composite sieving was used and the size of the designated separating sieve.
   - If material is retained on the designated separating sieve size in finer sieve set, then document that the percent retained does not exceed 2 % criterion.
   - Indicate if the ultrasonic bath or shaking apparatus or both were used during the dispersion process.
   - Any prior testing performed on specimen
   - All mass measurements.
   - Tabulation of percent passing for each sieve to 1%.

2. The report shall include the following:

   - Project identification, boring number, sample number, depth,
   - Classification or visual classification of the soil being tested
   - Any material excluded from the specimen.
   - Any prior testing performed on specimen
   - A graph of the percent passing versus log of particle size in mm

ASTM Comments:

Some materials that are not soils, but are made up of particles may be tested using this method.

This test method is ***not*** applicable for the following soils:

1. Soils containing fibrous peat that will change in particle size during the drying, washing, or sieving procedure.
2. Soils containing extraneous matter, such as organic solvents, oil, asphalt, wood fragments, or similar items. Such extraneous matter can affect the washing and sieving procedures.
3. Materials that contain cementitious components, such as cement, fly ash, lime, or other stabilization admixtures.

This test method may **not** produce consistent test results within and between laboratories for the following soils:

1. Friable soils in which the sieving processes change the gradation of the soil. Typical examples of these soils are some residual soils, most weathered shales and some weakly cemented soils such as hardpan, caliche or coquina.
2. Soils that will not readily disperse such as glauconitic clays or some dried plastic clays.

To test these soils, this test method must be adapted, or altered, and these alterations documented.

Standard Operating Procedure **ASTM D7928**

Technical Information

Reference Number:	ASTM D7928-17
Test Method Title:	Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis
Test Property:	Grain Size Analysis
Test Specimen Size:	Passing #10 sieve at least 50 g soil, containing no more than 60 g and no less than 15 g of minus #200 material. Amount varies see test standard Table 1 (based on largest particle size)
Number of Test Specimens:	Two representative samples obtained by quartering, mixing or splitting. One for grain size analysis and one for a representative moisture content determination.
Test Equipment:	Hydrometer (ASTM 152H –reads in grams of soil in suspension) Sedimentation Cylinder Sedimentation Cylinder Covers Stirring Apparatus (blender) Dispersion Cup Drying containers Balance readable to 0.01 gram Thermometer readable to 0.5 ºC Timing device Various sieves 250 mL glass beaker Drying oven capable of maintaining a temperature of 110 ± 5 ºC Dispersing agent mixture (40 g/L of Sodium Hexametaphosphate solution) Distilled Water Isopropyl alcohol Spray Bottle Wash pan Spatula

Standard Operating Procedure

Sampling

1. Collect a representative sample and perform a moisture content test in accordance with ASTM D2216. Enter data into the unique LIMS test file for this sample.
2. Collect another representative sample to be used for the particle size analysis. Base specimen size on test standard (based on largest particle size). Record empty beaker ID and weight. Record the weight of the wet specimen and beaker. Enter data into the unique LIMS test file for this sample.

   (Note: the two samples described above must be similar in grain size distribution and moisture content)

Splitting / Washing sample on #200 sieve

3. Add 125 ml of dispersing agent mixture or 5 g +/- 0.1 g of dry dispersing agent with at least 100 ml of distilled water to the test sample. Stir with spatula until all of the soil aggregations are broken-up.

   (Note: do not spill or lose any soil particles from the sample, rinse all surfaces off and back into sample)

4. Rinse sample into dispersion cup and use blender to further disperse sample for 1 minute.

5. Wash the test specimen from the dispersion cup, using distilled water, over the No. 200 sieve into a container. Be sure to collect all washings in the container. Use only 800 ml of distilled water for the washing operation.

6. Transfer the portion retained on the No. 200 into a tare and place in a drying oven. The dry weight will be needed for data entry and used in calculations for reporting. In addition, this portion could continue on for further grain size testing following any of a number of test standards for a full grain size analysis, upon request.

7. Wash the minus No. 200 sieve material into a Sedimentation cylinder and record ID of cylinder in the unique LIMS test file for this sample.

8. Add distilled water so the bottom of the meniscus is at the 1000 mL line of the Sedimentation cylinder. Place a rubber stopper securely (so no leaks or spills occur) over the open end and turn the cylinder upside down and shake in a manner that loosens all of the soil from the bottom and forms a slurry. Turn the cylinder upside down and back for a period of 1 minute (should be 60 turns per minute). Set the cylinder down.

9. Cover the cylinder, to prevent evaporation and allow the test specimen to sit overnight. This is the conditioning period which allows the temperature to equilibrate and the specimen to deflocculate.

Hydrometer Analysis of portion passing #200 sieve:

10. Using the Hydro Read Program of GTX LIMS system bring up this sample's unique test file and prepare to start this portion of the test.

11. Place a rubber stopper over the open end of the Sedimentation Cylinder and give the sample a pre-shake to loosen it from the bottom and make a slurry of the entire sample again.

12. Start the clock in the Hydro Read LIMS software. This will countdown the shake and time the test. Now turn the cylinder upside down and back for a period of 1 minute (should be 60 turns per minute) in coordination with the LIMS timer prompts. Set the cylinder down for sedimentation on a stable surface free from jarring and vibrations.

13. Remove stopper but DO NOT wash any adhering soil into the cylinder, let drips fall is necessary. If foaming on the surface occurs, spray with isopropyl alcohol to remove.

14. Begin to take and record hydrometer readings at the following intervals, using the LIMS timing prompts in the software: 1, 2, 4, 15, 30, 60, 120, 240, and approximately 1440 minutes. Enter your data into the unique LIMS test file for this sample

15. After the hydrometer reading, the temperature of the solution should be recorded. You may use the same temperature for the first few readings, and then for 30 min and those following take the temperature each time a reading is taken. Enter your data into the unique LIMS test file for this sample.

Report:

16. Enter all data required in the Hydrometer Header Data portion of the unique LIMS test file for this sample. The LIMS software will generate a complete grain size distribution report with tabulated and graphical data representations.

17. Record the following in the unique LIMS test file for this sample: Sample ID information including: Boring, Sample and Depth, Visual Description of sample including Munsell Book color and an initial "best guess" of grain sizes, Technician that performed each part of test, Balance ID, Oven ID, Thermometer ID, Separating Sieve ID and Sieve size, Wash Sieve ID, Hydrometer ID and type, Specimen Prep Method, Type of dispersant, Describe any excluded material, Describe any problems encountered and Indicate any prior testing on this specimen and if foam inhibitor was used.

18. Update your initial "best guess" sample description to correctly reflect the grain size descriptors corresponding to the as-tested data set following ASTM D2487 as guidance.

19. Calculate the % finer than the #200 sieve and add a sample comment to the unique LIMS test file as follows: Percent Passing #200 Sieve = 92%

eurofins	Always check on-line for validity. **Preparation of Vials for Field Preservation of Soils for Volatile Analysis**	Level: **Work Instruction**
Document number: **S-SS-WI11242**		
Old Reference: **1-P-QM-WI-9015073**		
Version: **17**		Organisation level: **5-Sub-BU**
Approved by: **UCSS** Effective Date **22-MAR-2018**	Document users: **5_EUUSLA_Sample Support_Manager, 6_EUUSLA_ Sample Support_VOA prep**	Responsible: **5_EUUSLA_Sample Support_Manager**

LIMS ID

Analysis DOD - 0388, 6119, 6169, 6647, 0405, 1169, 6171, 6172, 6173, 6645, 2392, 6176, 7579, 0069

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Revision Log

Revision 17	**Effective Date:** This version	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Applicable to the procedure	Updated frozen temp to ≤ -10°C

Revision: 16		**Effective Date:**	September 30, 2016
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356		Removed revision logs up to the previous version
Procedure	Reflects the current procedure		Referenced the option to use pre-purchased vials

Reference

1. USEPA, 40 CFR Part 60, Appendix A, Method 25D

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 5035, December 1996.

3. Test Methods for Evaluating Solid Wastes, SW-846, Method 5035A, July 2002.

4. *Method for the Field Extraction/Preservation of Soil Samples with Methanol for Volatile Organic Compounds*, New Jersey DEP, February 1997.

5. Method AK101 for the Determination of Gasoline Range Organics, April 8, 2002.

6. USEPA, 40 CFR Part 60, Appendix A, Method 25D

7. WI PVOC PUBL-SW-140 09/95, *Wisconsin DNR Modified GRO*.

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Purpose

The purpose of this SOP is to provide detailed instructions for the preparation of vials used for field-preservation of soil samples to be analyzed for volatiles.

Scope

This procedure applies to analysts who prepare pre-preserved containers used in the field for soil sampling of volatile analyses.

Basic Principles
An aliquot of preservative is placed in a volatile−free container and weighed. The weight of the container and preservative is then captured using the Volatile Preparation program in LIMS. When requested, the container is sent to the client for use in field preservation of a solid sample. When the container and the soil sample are returned to the lab, it is re−weighed and the actual sample weight is calculated. Samples are then spiked with the appropriate surrogate as determined by the analysis being performed.

Definitions
1. Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

2. VOA Prep Summary and VOA Prep Summary by SDG are reports that reside on the LIMS database. Using the Volatile Prep application and moisture results pulled from LIMS, the report is populated and calculations are performed to achieve a Final Extraction volume to be used when necessary by the appropriate Groups.

Interferences
Sample contamination can occur if the vial preparation is not performed in a volatile−free environment; therefore, this process must be performed in one of the designated volatile−free laboratories. Samples also become contaminated if volatiles diffuse through the sample vial septum. A trip blank carried through sampling, storage and handling acts as a check of such contamination.

Safety Precautions and Waste Handling
See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Methanol is flammable. Containers of this solvent must be kept away from any sources of open flames or sparks. Due to the potentially toxic nature of samples received from clients for analysis, safety precautions must be observed when handling samples. Safety glasses, lab coats, and gloves are required. Weighing out of the samples must always take place in a hood.

The solvents utilized in this procedure are disposed of in a solvent waste container which is transferred to the lab-wide disposal facility. Expired standards in methanol are disposed of as hazardous waste. Bulk sample containers and methanolic sample preparations are returned to the sample storage area for future disposal. Other wastes generated by Lancaster Labs are disposed of via incineration at EPA licensed facilities.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP.

The initial training consists of observing the procedure being carried out by an experienced analyst/technician. Next, the trainee performs the procedure while the experienced person watches, answers questions, and gives feedback. Following the initial training, experienced individuals are available as a resource until no longer required. Analysts are considered proficient when they are able to perform the procedure independently.

Sample Collection, Preservation, and Handling

Sample containers must be refrigerated at 0° to 6°C, not frozen, after preparation. Containers containing surrogates are not kept for more than 2 weeks before being discarded or sent into the field for use in sample collection. Packaging of sample containers must follow all DOT regulations. When returned, samples must be preserved and refrigerated unfrozen at 0° to 6° C or frozen in reagent water at ≤ −10° within 48 hours of collection. See individual technical area SOPs for method specific hold times.

Apparatus and Equipment

1. 40-mL vials with Teflon™−lined septa and screw caps
2. 40-mL vials with stir bars, Teflon™−lined septa, and screw caps
3. 125−mL amber glass wide mouth jar with Teflon™−lined septa and screw caps
4. Pipette capable of dispensing up to 25 ± 0.25 mL.
5. 50−µL syringe
6. 1000−mL volumetric flask, class A
7. Analytical balance capable of weighing ±0.01 g.
8. Label printer/labels

9. LIMS VOA prep application and Reagent, Solution, and Balance Applications which integrates a PC with an analytical balance to collect data directly from the balance. It organizes the data, performs calculations, and stores final results in the Laboratory Information Management System (LIMS).

Reagents and Standards

NOTE: A rinse using reagent water followed by methanol must be performed on the pipette dispensers before adding a new lot of solution or standards.

1. Methanol – Purge and trap grade.

   a. Store at room temperature and re−analyze yearly.

   b. Use methanol that has been previously tested and approved for use by the labs.

2. 8260A/B Surrogate Mix, Restek Catalog #30340 (2500 µg/mL) or equivalent.

   a. Store at ≤ -10°C for up to 1 year.

   b. Use standard as is, or diluted in methanol to a final concentration of 2.5 µg/mL. Diluted standard must be stored at ≤ -10°C for up to 6 months.

   c. This standard is used for the GC/MS analyses.

3. Custom α,α,α−trifluorotoluene (TFT), Restek Catalog #54357 (15,000 µg/mL) or equivalent.

   a. Dilute standard in methanol to a final concentration of 750 µg/L for the working solution. A prep dilution of 0.5 mL of TFT to 10 mL of methanol is used when spiking returned field preserved containers.

   b. Store at ≤ -10°C for up to 30 days.

   c. This standard is used for the GC analyses.

4. Sodium hydrogen sulfate anhydrous powder, Fluka, Catalog #2316657 or equivalent.

   a. Store at room temperature and re−analyze yearly.

   b. If compounds are detected above the method detection limit (MDL), prepare another vial and repeat the analysis. If compounds are still detected above the MDL, a new container must be tested and used.

5. Sodium Bisulfate Solution –

a. Prepare by diluting 200 ±0.5 g of the Sodium hydrogen sulfate anhydrous into 1000 mL of reagent water in 1000−mL volumetric flask. Cap and invert at least 10 times to mix.

b. Store at room temperature and re−analyze every 6 months if supply remains.

c. If compounds are detected above the method detection limit (MDL), repeat the analysis. If compounds are still detected above the MDL, remake the solution and test before using.

6. Reagent water – water in which target analytes are not detected at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

7. Polyethylene glycol (PEG) – Average molecular weight 400 amu, EM Science preferred.

a. Any lot/vendor must meet a cleanliness level of <50 mg/kg volatile content.

b. Store at room temperature and re-analyze each year if supply remains.

Calibration
Not applicable to this procedure.

Procedure
Use the VOA Prep application whenever possible for this procedure to facilitate data transfers and other tracking. However, it is also allowable to record data traditionally in a logbook.

A. Prepare preservative containers

NOTE: Pre-purchased vials can be used for selected preps. Vials containing 5 mL of DI Water, vials containing 5 mL of Sodium Bisulfate and vials containing 5, 10 and 15 mL of methanol are available for field preserved analyses. The vials contain the initial tare weight, vial lot number, and the preservative lot number.

1. Check to make sure the pipette calibration has been performed.

2. Use the pipette to add the appropriate amount of preservative to a clean container. A 40−mL vial is used unless otherwise indicated on Form 4580.

a. For analyses 6119 and 6172 the appropriately diluted surrogate solution must be added at this time using a pipette.

b. For analysis 1169 use a syringe to add the ampulated 8260A/B surrogate standard plus methanol into a 40−mL vial. Use 1 µL of 8260A/B surrogate for every 1 mL of methanol in the vial.

3. Seal the container with a screw cap and septum seal.

4. Label the container with the tracking number.

5. Check to make sure the balance has been calibrated each day before use.

6. Place the container on a zeroed balance and capture the weight electronically to the nearest 0.01 g using the VOA Prep application.

7. Store the prepared containers in the designated area in the Volatile Prep room. Vials containing surrogates must be stored in the freezer, vials containing just methanol are stored at room temperature or refrigerated.

8. Pull vials as requested via e−mail from client services and place on the designated shelf in the bottles room storage area. The containers are now ready to be sent into the field for sample collection.

9. Send a reply e−mail to the client service representative to notify them that the order has been completed.

B. Re−weigh preservative containers after return from the field

1. Using the Volatile Prep application, scan the tracking number on the container. This brings up the information documented from the preparation step described above.

NOTE: If using purchased pre-tarred vials insert the vial ID, preservative, and tare weight information on the Pre-Tarred tab within the Volatile Prep Application.

2. Check to make sure the balance has been calibrated each day before use. Place the container on a zeroed balance and capture the second weight electronically using the VOA Prep application.

3. The Volatile Prep application calculates the net weight. (weight of vial, solution, and soil minus the weight of the vial and solution).

4. If there are any holding−time issues or if weights are outside of the Action Requirement, the VOA Prep application automatically sends an e−mail to Client Services.

5. Record any unusual observations about the sample in the comment section.

C. Sample spiking

NOTE: Analyses 11014 and 11764 do not require a surrogate to be spiked into the methanol.

A 1:1 ratio of µL to mL is required when spiking surrogate into methanol, except for analysis 0388 MA−VPH samples which require 30 µL to the 15 mL of methanol.

1. Determine the amount of methanol in the sample. This is determined by the analysis number and the mL of methanol printed on the tracking number.

2. Fill the syringe with the appropriate surrogate slightly past the amount of µL needed.

a. Add the appropriate surrogate based on the bottle code entered and the department receiving the sample.

b. GC requires a TFT surrogate mix to be used, bottle code 64.

c. GC/MS requires an 8260A/B surrogate mix to be used, bottle codes 14 and 66.

3. Dispense the surrogate from the syringe until the required level of µL is reached. This assures no air bubbles are present and the syringe contains surrogate.

a. Tilt the vial so the methanol comes toward the top of the vial, spike through the septa, submerging the tip of the syringe into the methanol.

b. Shake the vial to disperse the surrogate throughout the sample.

c. Put an “S” on the vial label to indicate surrogate has been added.

d. Clean the syringe before proceeding to the next sample.

D. Deliver samples to the labs

Once the sample containers have been re−weighed, they must be transported to the laboratory for analysis. Each department has a designated refrigerated drop−off spot. All trip blanks and field blanks submitted must follow the sample containing the same preservative. Blanks submitted as waters are to be stored in the designated volatile refrigerator.

NOTE: Any sample designated for prescreen must be taken to the appropriate refrigerator and placed in a box sorted in numerical order in groups of 21 or 42 when applicable. If a methanol sample is designated for prescreen and a corresponding methanol trip blank or field blank is submitted the blanks must follow the associated samples through the entire process including sample prescreen.

Calculations

Calculation of sample weight : Wn = Ws - Wf

Where:

Wf = weight of container + solution (first weight)

Ws = weight of container + solution + soil (second weight)

Wn = net weight of soil sample

Statistical Information/Method Performance

Not applicable to this procedure.

Quality Assurance/Quality Control

The number of containers requested from Client Services includes the appropriate amount of extra bottles to serve as matrix quality control (QC) for volatile analyses requested. The matrix QC spiking for this process is noted in Procedure C. All other QC samples such as the LCS, LCSD, and method blanks are prepared as outlined in the individual technical area's SOPs.

End of document

Version history

Version	Approval	Revision information
16	30.SEP.2016	
17	08.MAR.2018	

eurofins	Always check on-line for validity. **TOC and TC in Solids and Sludges by Combustion by SM 5310B, EPA 415.1, SW-846 9060/9060A, Lloyd Kahn**	Level: **Work Instruction**
Document number: **T-WC-WI11627**		
Old Reference: **1-P-QM-WI-9013418**		
Version: **17**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **09-JUL-2019**	Document users: **6_EUUSLA_ Instrumental Water Quality _TOC Analyst, 6_EUUSLA_ Instrumental Water Quality _TOC Verifier**	Responsible: **5_EUUSLA_Instrumental Water Quality_Manager**

LIMS ID
Analysis 2079, 0383, 10065, 11356

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Revision Log

Revision:	17	**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version.
Scope	Current Process		Removed LIMS analyses 6623 and 11763
	Method Requirement		

Revision:	17	**Effective Date:**	This version
Reference Modifications			Removed 10% dup instead of a quadruplicate per batch
Procedure 8.	Current Process		Removed the process of analyzing three blanks prior to calibration.
Procedure 10.	Current Process		Removed the concentration of the CCV, this changes dependent on the CRM provided by the manufacturer
QA/QC	Method Requirement		One quadruplicate per batch for Lloyd Kahn

Revision:	16	**Effective Date:**	Oct 17, 2018
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Cross Reference	Reflect re-identification of documents in D4		Updated all document titles.
Reference	New instrumentation		Deleted the OI soil analyzer and added the Skalar information.
Apparatus and Equipment	New instrumentation		Deleted the OI soil analyzer and added the Skalar information.
Reference	Method Update		Updated the Standard Methods Edition to the 22nd Edition, 2011.
Subject	Addition		Added method references to title.
Calibration	Update		Updated to reflect current CCV process.
Reference 2.	Removal		Removed the term 'modified'.
Personnel Training and Qualifications	Addition		Added requirement of an annual Demonstration of Capability.
Throughout Document	Changed document control platform		Updated ETQ References to the D4 References.

Reference

1. Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 5310B-2011

2. *Method 415.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.*

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 9060, September 1986.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 9060A, November 2004

5. *The Primacs SNC-100 Analyzer Manual*, Skalar..

6. *Determination of Total Organic Carbon in Sediment, U. S. EPA, Region II, July 27, 1988. ("Lloyd Kahn Method.").*

7. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11188	Reagents and Standards

Scope

This method is applicable for the determination of total organic carbon (TOC) in soils and other samples not easily analyzed by the TOC waters method. The limit of quantitation (LOQ) for this method can be found in the analysis information file. Quantitative TOC results up to 1,000,000 mg/kg may be obtained by this method.

Analysis 0383 is used for samples referencing the Lloyd Kahn Method.

Analysis 10065 is used for the determination of Total Carbon in solids. This procedure does not eliminate the inorganic carbon by acidification prior to analysis.

Analysis 11356 is a calculation to determine the Total Inorganic Carbon (TIC) result.

Basic Principles

TOC is determined by acidifying a sample and heating it to remove the TIC. An aliquot of sample (1 mg to 1 g) is weighed into a sample cup. The sample is then heated to 900°C for combustion of the remaining TOC. The resulting carbon dioxide from the TOC is detected by a nondispersive infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. The mass is proportional to the mass of TOC in the sample. Samples analyzed by this method include solids such as soils or sediments, slurries, sludges, brines, and corrosives.

The TC measurement is identical to the TOC measurement, with the exception that there is not an acidification and heating step to remove the TIC.

TIC is the calculation subtracting the TOC soil result from the result obtained when analyzing the TC.

Reference Modifications

There is no referenced method for the determination of Total Carbon in solids. Analysis 10065 references a modified TOC method. The calibration standards, QC standards, and LCS employed for this analysis are composed of only organic sources of carbon (Sucrose for the calibration and QC standards, and a purchased standard for the LCS solid). Therefore, the Total Organic Carbon content is equal to the Total Carbon content in these standards.

Interferences

Carbon is ubiquitous in nature. Therefore, extra care must be taken to avoid contamination of reagents, glassware, and any other materials that come in contact with the sample. Samples which are light in weight may need to be analyzed at smaller aliquots to fit within a sample cup. These samples are reported with an elevated LOQ.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

1. Normal laboratory practices for safety must be followed.

2. Extreme caution must be used when handling sample cups after they have been analyzed.

3. Discard acid waste in acid waste containers.

4. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability. Analysts are considered proficient when they have successfully completed a Demonstration of Capability for the analysis. A Demonstration of Capability consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS and LCSD. Documentation for these studies are in each individual's training records.

Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples must be collected and stored in glass containers unpreserved. Samples must be stored under refrigeration at 0° to 6°C, not frozen.

Because very small amounts (1 mg to 1 g) of sample are used for the analysis, the sample must be as homogeneous as possible.

The holding time for analysis by SM 5310 B, EPA 415.1 and SW-846 9060/9060A is 28 days. The holding time for analysis by the Lloyd Kahn method (analysis 0383) TOC is 14 days.

Apparatus and Equipment

1. Skalar-Primacs SC100
2. PC and SNAccess software
3. 2-stage gas regulator (two required)
4. Sample cups
5. Fiber quartz wool
6. Analytical balance, capable of accurately weighing to 1. mg
7. Glassware – General laboratory glassware as needed for preparing reagents and standards
8. Microliter syringe, various volumes

Reagents and Standards

NOTE: All chemical used must be ACS reagent grade unless otherwise noted.
Different volumes or weights may be used provided the ratios remain equivalent.
See *QA-SOP11188*, for the appropriate labeling and documentation of reagent and standard preparation.

1. Ultra pure nitrogen gas (60 psi)
2. Ultra pure oxygen gas (60 psi)
3. Hydrochloric acid, concentrated (HCI) – Purchased. Store at room temperature.

4. 1+1 Phosphoric acid

Phosphoric acid (H3PO4)	50 mL
Reagent water	50 mL

Take 50 mL of H3PO4 and add slowly while swirling to a 100-mL volumetric flask containing 50 mL reagent water. Store at room temperature. Prepare fresh every 6 months.

5. TOC stock calibration standard (30% carbon), purchased. See label for expiration date. Store at room temperature.

6. TOC LCS standard, purchased. See label for expiration date, and the certified concentration. Store at room temperature.

7. TC/TOC QC Stock solution 15,000 mg/L.

Potassium Hydrogen Phthalate (KHP)	15.945 g ± 0.005 g
Phosphoric Acid, concentrated, 10 drops.	

Add 10 drops of Phosphoric acid to a 500 ml volumetric flask containing approximately 250 ml of reagent water. Add 15.945 g ± 0.005 g of KHP to the flask, dissolve and bring to full volume with reagent water. Store in an amber glass bottle at 0° to 6°C, not frozen. Prepare every 30 days.

Sample Cup Preparation

Sample cups must be conditioned prior to analysis. This is achieved by heating the cups to 900°C for a period of 2 to 5 minutes.

Calibration

1. Working standards for calibration

Pipette a volume of the TOC stock calibration standard into the sample cups as follows:

Working Std. (mg C)	Vol. Stock TOC (µL)
0.30	1.0
0.90	3.0
3.0	10.0
6.0	20.0

Prepare fresh daily. Store at room temperature.

2. Continuing calibration verification standard (CCV)

This is a purchased standard, and the acceptable range is specified by the manufacturer.

Procedure

1. Turn on nitrogen to 60 psig.

2. Turn on oxygen to 60 psig.

3. Add water and a drop of HCI to the scrubber.

4. Measure the carrier gas (oxygen) flow rate and adjust to 200 mL/min.

5. Measure the dryer gas (nitrogen) flow rate and adjust to 160 to 300 mL/min.

6. Power up instrument.

7. Program in a sequence file.

8. Run the calibration sequence as follows: 0.30, 0.90, 3.0, and 6.0 mg C (Performed monthly).

9. The instrument automatically calculates the R and list the calibration data.

10. Samples are analyzed along with the appropriate laboratory control standard and preparation blank. A check standard (CCV) and a continuing calibration blank (CCB) must be run at the beginning of each run and after every ten samples.

11. Add sufficient glass wool to each cup to cover the bottom surface.

12. Weigh samples into sample cups (up to 1000 mg). Add 1+1 H3PO4 drop wise until effervescence stops. Heat at 75°C for 15 minutes. Record the oven ID, time and temperature of this step in the log book.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample. The addition of H3PO4 and the heating to 75°C is not performed for analysis 10065.

13. Analyze the residue according to the instrument manufacturer's instructions for the remaining TOC result.

Calculations

1. To determine mg/kg TOC or TC

   mg/kg = (Raw result in mg C) x (1000/weight in mg) x (1000)

2. To determine LOQ/MDL factors

$$Factor = \frac{Maximum\ sample\ aliquot\ weight\ (1000.mg)}{weight\ of\ sample\ (mg)}$$

3. To determine Total Inorganic Carbon (TIC)

   TIC = Total Carbon (TC) - Total Organic Carbon (TOC)

Statistical Information/Method Performance

1. The method detection limit (MDL) is determined annually by following the procedure outlined in *QA-SOP11892*.

2. The quality control acceptance windows are generated annually by following the procedure outlined in *QA-SOP11896*.

Quality Assurance/Quality Control

1. A calibration must be performed every 30 days. The acceptable range for the calibration is R = 0.995 or greater. If this criteria is not met, the instrument must be recalibrated.

2. A batch must contain no more than 20 field samples.

3. A batch blank (PBS) must be analyzed every batch or each day samples are prepared (not to exceed 20 samples). An acceptable result is < the limit of quantitation. If the PBS does not meet this criterion, it must be rerun twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be repeated.

4. A laboratory control standard (LCSS) must be analyzed every batch or each day samples are analyzed (not to exceed 20 field samples). For TOC this is a purchased standard and the acceptable range is specified by the manufacturer. For TOC by 11763, the acceptance range for the LCS is 80% to 120%. For Total Carbon, spike 0.2 ml of the TC QC Stock solution onto glass wool. Assuming the nominal full volume of 1000 mg, the TC LCSS true value is 3000 mg/kg. The LCSS should undergo the same steps as the samples. If the LCSS does not meet the acceptable criterion, it must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be reanalyzed.

5. Based upon client requirements, a laboratory control standard duplicate (LCSSD) may need prepared and analyzed under the same conditions as the LCSS. The acceptance criterion for the LCSSD is the same as that of the LCSS. The relative percent difference between the LCSS and the LCSSD is calculated statistically.

6. Analysis 2079 - a duplicate is analyzed every 10 samples. Analysis 0383 - one sample is analyzed in quadruplicate every batch. The acceptable relative percent difference is statistically determined. The duplicate relative percent differences must be tracked to continually monitor method performance.

7. Analysis 2079 - A spike must be analyzed for every 10 samples. The sample is spiked with 3 ul of the purchased TOC stock calibration standard. The acceptance range is determined statistically. The spike recoveries must be tracked to continually monitor method performance.

8. Based upon client requirements, a matrix spike duplicate (MSD) may need prepared and analyzed under the same conditions as the MS. The acceptance criterion for the MSD is the same as that of the MS. The relative percent difference between the MS and the MSD is calculated statistically.

9. A check standard (CCV) and blank (CCB) must be run after every ten injections (including blanks and standards). The acceptable range for the CCV is ±10% of the true value. An acceptable CCB result is < the limit of quantitation. If either, or both, of these injections do not meet the acceptance criterion, the unacceptable original must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples since the last compliant CCV/CCB must be reanalyzed.

10. A CCV and CCB shall be analyzed at the beginning and the end of each run. At any time when the instrument has been idle for a period of 4 hours or more, a CCV and CCB must be analyzed. If either of these parameters cannot meet specifications, the instrument must be recalibrated.

QA-SOP11188 Reagents and Standards
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits

End of document

Version history

Version	Approval	Revision information
15	30.OCT.2013	
16	17.OCT.2018	
17	03.JUL.2019	

eurofins	Always check on-line for validity. **VOCs and GRO by GC/MS in Soils and Solids by EPA 8260C/D**	Level: **Work Instruction**
Document number: **T-VOA-WI8236**		
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Version: **6**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **12-APR-2019**	Document users: **6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Management, 6_EUUSLA_GC/MS Volatiles_SW846 8260B/C Soil**	Responsible: **5_EUUSLA_GC/MS Volatiles_Manager**

LIMS ID
Analysis 11995

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Revision Log

	Revision: 6	**Effective Date:** This version
Section	**Justification**	**Changes**
Title	Current practice	Updated title to reflect new methods
Revision Log	Formatting requirement per G-DC-SOP16244	Removed revision logs up to the previous version
Reference	Current practice	Added 8260D reference

Revision: 5		**Effective Date:** 21.MAR.2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Title		Shortened title for clarity
Reference	Update	Added 8000D
Cross Reference	Redundancy	Removed Level II procedures and updated to D4 numbers

Revision: 5	**Effective Date:** 21.MAR.2019	
Personnel Training and Qualifications	Requirement	Added reference to IDOC
Reagents and Standards	Current practice	Updated freezer temperature and clarified IS/SS concentrations. Updated secondary dilution standard expiration throughout section.
Calibration	Clarification	Updated verbiage concerning MDL detection. Added 8000D criteria. Quantified "scoop" amount for sodium bisulfate. Added "prepared daily" to CCV description. Clarified acceptance criteria by stating "No more than 20%"...
Procedure	Clarification	Added use of reagent water for method blank and reference to QA/QC section for specific requirements. Added statement that method blank must be analyzed before samples.
Statistical Information/ Method Performance	Current requirement	Added statement concerning MDL evaluation
Quality Assurance/ Quality Control	Current practice	Added LLOQ information Added sand requirement for low level method blank
Entire Document	Update	Removed references to Level II procedures and updated to D4 numbers
Entire Document	Formatting requirement	Added GRO information contained in Appendix A
Appendix A	Formatting requirement	Deleted entire section, information redistributed to appropriate sections

Reference

1. Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), SW-846 Method 8260C, August 2006.

2. Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), SW-846 Method 8260D, June 2018.

3. Determinative Chromatographic Separations, SW-846 Method 8000D, March 2018.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 5030B, December 1996.

5. Test Methods for Evaluating Solid Wastes, SW-846 Method 5030C, May 2003.

6. Test Methods for Evaluating Solid Wastes, SW-846 Method 5035, Revision 0, December 1996.

7. Test Methods for Evaluating Solid Waste, SW-846, Method 5035A, July 2002.

8. *Total Petroleum Hydrocarbons Analysis-Gasoline Method*, California Department of Health Services, LUFT Task Force.

9. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10682	GC/MS - Bulk Solid Matrix Sample Preparation
S-SS-WI11170	Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035
T-VOA-WI7619	GC and GC/MS Instrumentation Maintenance
T-VOA-WI7629	GC/MS Volatile Standards Traceability
T-VOA-WI7692	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
T-VOA-WI8373	GC/MS Volatiles Audit Process

Scope

This method is suitable for the determination of target compounds listed and maintained in the Laboratory Information Management System (LIMS) for soil and solid matrices. Samples consist of soils/solids collected or submitted in bulk in glass containers with Teflon-lined screw-caps or in field core sampling/storage containers (i.e., EnCore or equivalent). Associated MDLs/LOQs are listed in the LIMS under the analysis numbers and/or Project Information lists. Theoretical Standard Calibration (TSC) Sheets are included in the Appendix (Figures 1-6). These TSC sheets are to serve as examples only and may not reflect most current version in use. Non-target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration.

Compounds other than those listed in the LIMS for this group of master scans may be analyzed using USEPA SW-846 Method 8260C. Selected Ion Monitoring (SIM) parameters can be used to detect, identify and quantitate volatile organic compounds in methanol extracts of soils/solids, if lower quantitation limits are required (project- or client-specified) and/or matrix interferences are anticipated. Attachment I describes the proper analysis procedure for Gasoline Range Organics in soils and solids. Due to poor purging efficiency or poor chromatography, some analytes require calibration at higher levels and/or higher practical quantitation limits (PQLs). Any additional compounds must be added to the theoretical standard concentrations (TSC) sheet. Standards containing additional analytes must be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions must be prepared for use in analyzing additional compounds.

Basic Principles

The soil sample is prepared according to analysis *S-SS-WI10682* and *S-SS-WI11170* for 8260C. The sample is purged with an inert gas and the effluent gas passed through a sorbent trap where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed onto the head of a gas chromatographic column held at the appropriate initial temperature for the column in use. The gas chromatographic column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the MS, it is bombarded by high energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focused into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

The GRO analysis is typically performed in conjunction with the analysis of other volatile target compounds by SW-846 Method 8260C. The GRO quantitation range is 0.1 minutes before the peak apex of C6 (hexane) to 0.2 minutes after the peak apex of C12 (dodecane); however, other ranges can be established. By establishing a (C12) GRO window to 0.2 minutes following the elution of dodecane, the areas from a trio of unresolved peaks eluting near to the upper limit of the range must consistently be included in the total GRO area. In addition, the range remains tight enough to ensure that no C13 or greater compounds can be included in the total GRO area. The C4 range retention time is determined by selecting the first peak after the air and/or artifact peak minus 0.1 minutes in the first level of standard analyzed with the ICAL. The C5 range retention time is 0.1 minutes before the peak apex of pentane.This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies. The area of the total ion chromatogram for the GRO range is determined. The area of the internal standards and surrogate standards are found and subtracted from the total area of the chromatogram within the desired time range. The resulting area is then quantitated versus the internal standard, fluorobenzene.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated soils and solids can interfere with the internal

standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of SW-846 Method 8260C for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity of each reagent used has not been precisely determined. However, each reagent must be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves when working directly with reagents. Refer to the *Chemical Hygiene Plan* for specific details.

Personnel Training and Qualifications

Education Requirement: A 4–year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or 1 – 3 years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis must be documented by each analyst in their training record. Specifically, each new chemist must train with an experienced chemist for the first 12 weeks depending on the individual and their previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time may be less if the new chemist has prior relevant experience in GC/MS and analytical chemistry background.

During the training period, the new chemist must learn daily maintenance, calibration techniques, data and library search review, and forms generation. They are also required to read all relevant SOPs and EPA methods. All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP. Each analyst must perform an Initial Demonstration of Capability to measure proficiency. Thereafter, an annual Demonstration of Capability is performed and maintained in the analyst's training records.

NOTE: A separate Demonstration of Capability for GRO is required.

Sample Collection, Preservation, and Handling

The samples must be stored in a refrigerator at 0°C to 6°C, not frozen. Soils are collected/submitted in bulk in glass containers with Teflon-lined screw-caps or in field core sampling/storage containers (i.e., EnCore or equivalent). Samples are preserved in methanol and either frozen reagent water or Sodium Bisulfate. All samples must be analyzed within 14 days of collection.

Apparatus and Equipment

1. Gastight micro syringes – 10-µL and larger
2. 5-mL gastight syringes
3. Analytical balance, capable of accurately weighing ±0.0001 g
4. Top-loading balance capable of weighing ±0.01 g
5. Glassware
   a. Class A Volumetric flasks with ground-glass stopper
   b. Vials, 1.5-mL, 15-mL, and 40-mL screw cap, with Teflon/silicone septa
   c. Mininert vials, 1-mL, 2-mL, and 5-mL
6. Purge and trap device – Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging chamber must have the purge gas

passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

7. Autosampler – OI Analytical 4551, Archon, or equivalent meets the requirements of this method. The Archon has the capability to purge solid samples directly in the 40-mL vial (needed for samples prepared by analysis 8389).

8. Spiker units (optional) – OI Analytical Model 4551 SIM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551 autosampler to automatically introduce 10 µL of internal standard (ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

9. GC/MS system – The HP 5890GC/5972 MSD, HP 6890GC/5973MSD, HP 6890GC/5975MSD, and Shimadzu GC/MS QP5000 meet the requirements for this method.

10. Data System/Computer/Software– this is interfaced to the GC/MS system, which continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

11. GC Columns

a. Column 1 – 30M × 0.25 mm ID DB624 capillary column with a 1.4-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

b. Column 2 – 20M × 0.18 mm ID DB624 capillary column with a 1.0-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

Different sampling/analysis combinations are used based on how the sample was collected, expected level of VOCs in the sample, possible matrix interferences, list of target compounds, whether TICs were requested, the required quantitation limits and the type of equipment/instrumentation available.

NOTE: Refer to *T-VOA-WI7619* for instrumentation maintenance and troubleshooting.

Reagents and Standards

A. Reagents

1. Reagent water is defined as water in which an interferant is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory must meet these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Purge and Trap grade Methanol or equivalent

3. Sodium bisulfate – ACS grade

4. Sand- Ottawa Standard

B. Standards

See *T-VOA-WI7629* for standards traceability.

1. Stock standard solutions – Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at ≤-10°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

NOTE: For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate standards are purchased from a vendor, as well as the target compounds that are gases at room temperature. These gaseous standards have a 1-week expiration date, starting from the date they are opened.

2. Surrogate stock standard solution (for high level soils) – a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4-bromofluorobenzene, and 1,2-dichloroethane-d4 is prepared in methanol by a commercial supplier

3. 8260A Internal standard spiking solution (8260IS) – a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, and 12500 µg/mL deuterated tertiary butyl alcohol (tBA-d10) is prepared in methanol by a commercial supplier. Deuterated tertiary butyl alcohol (tBA-d10) is used sometimes as an auxiliary ISTD. If an autosampler is used, dilute the stock to 250 µg/mL (1250 µg/mL for tBA-d10) in methanol. This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained (50 µg/kg).

4. GRO calibration standard – a 5500-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier.

5. GRO QC standard – a 20,000-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier

6. To prepare stock standards from neat compounds:

a. Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

b. Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Reweigh the flask, record/note the amount, dilute to volume, stopper, and mix by inverting the flask at least 3 times. Calculate the concentration of the standard.

c. The stock standard solutions are stored in Teflon-sealed screw-capped vials at ≤-10°C. The compound name, concentration, date prepared, expiration date, preparer name and storage conditions must appear on the bottle.

d. Replace in house prepared stock standard solutions every 6 months.

7. Secondary dilution standards – Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required producing a given volume of a mixed working standard with a known concentration of each analyte. When custom mixes are used, these may be diluted down individually, or combined together with other mixes. The working standard is tested according to *T-VOA-WI7629*. The verified working standard is poured into Teflon-lined screw-capped GC vials or mininert vials and stored at ≤-10°C. A designator indicating the standard, month, and preparation date must be on the standard vials. The designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 2-4 weeks (1 week for standards containing gases) unless otherwise indicated.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50-µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used varies, depending on the actual stock concentration.

b. Surrogate standard spiking solution (high level only) – Prepare the surrogate standard spiking solution from the stock standard solutions at a concentration of 25 or 250 µg/mL in methanol. The requested soil preparation method dictates what concentration of surrogates are required for analysis.

c. Internal standard (IS) solution (high level only) – fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, and tert-Butyl Alcohol-d10. One mL of 8260IS is diluted with methanol to a total volume of 10 mL to give a final concentration of 250 µg/mL (1250 µg/mL for tBA-d10). This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained (50 µg/kg).

d. IS/SS spiking solution (low level only)– Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10-mL final volume (resulting in a concentration of 250 µg/mL, 1250 µg/mL for tBA-d10). This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained 50 µg/kg).

e. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 4, 10, 20, 50, 100, and 300 (µg/kg). Replace the calibration spiking solution every 2-4 weeks (1 week for standards containing gases).

f. Matrix spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates; therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace the matrix spiking solution every 2-4 weeks (1 week for standards containing gases). See Figures 2-5 for preparation information.

g. Store all standard solutions at ≤-10°C and protected from light.

Calibration

A. Instrument Conditions:

1. The purge and trap device must have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge gas	Helium
Purge flow	35 – 40 mL/min
Purge temperature	40°C for low level soils and ambient temp. for medium/high level soils
Purge time	11 min
Desorb temperature	190°-220°C
Desorb time	0.5 to 4 minutes **
Bake temperature	180°-220°C
Bake time	8 min (±3 min)

** - Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions may be changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument may be found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	Column 2
Column liquid phase	DB-624	DB-624
Carrier gas	Helium	Helium
Carrier gas flow	0.8 mL/min	0.6 mL/min
Make-up gas flow	None	None
Initial temperature	45°C	45°C
Initial hold time	4.5 min	2.5 min
Temperature ramp	12°/min to 100°C then 25°/min to 240° C	12°/min to 100°C then 25°/min to 235° C
Final temperature	240°C	235°C
Final hold time	None	.02 min

4. Recommended mass spectrometer (MS) operating conditions:

Ions	Positive
Electron energy	70 volts
Mass range	35 – 300 amu

H-P systems Scan time:

Number A/D Samples	22 (4)
Integration Time/Sample	50 microsec
Total Scan Time	0.6 sec

B. Tuning:

Tune the GC/MS system to meet the criteria in Table 1 following a 50-ng injection of BFB. The chromatographic conditions must be the same as those under which the samples are analyzed except that the temperature ramp may be increased and the initial temperature and flow rate may be different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same mass spectrometer parameters as those used to obtain a successful tune.

C. Initial Calibration:

1. Perform the initial calibration by analyzing at least six distinct levels of analyte concentration. For high level soils, the surrogates are also analyzed at each distinct level. Response factors for each analyte are determined from these levels. Six levels are required if second order regression fits are used. Calibration for GRO consists of analyzing six distinct levels of GRO area in order to produce a response factor for the GRO quantitation range of interest using the internal standard, fluorobenzene. The relative standard deviation of the response factor determines the suitability of the average relative response factor for calculation of the GRO concentration. Refer to Figure 1 for the preparation of calibration standards and Figure 6 for preparation of GRO standards. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte or GRO concentration. Prior to the analysis of any calibration level, retention time markers must be run for the GRO range of interest. The retention time markers are hexane (C6) and Dodecane (C12). Other markers can be used if different ranges are required by a project.

NOTE: 5 levels are required by the method.

2. A method detection limit (MDL) standard must be analyzed with each initial calibration. This standard is prepared at or near the departmental MDL and is not to be included in the calibration curve. All compounds must be detected at or below MDL standard.

3. For medium/high level soils, methanol is added to all calibration standards. For low-level soils prepared according to EPA Method 5035, 1 scoop (approximately .50 -.75 grams) of sodium bisulfate is added to all calibration standards.

4. When using an autosampler, blanks and standards are prepared and poured into 40-mL vials with Teflon-lined septa. For the high-level method, 5 mL is withdrawn from the vial and transferred to the sparge vessel along with the appropriate amount of the internal standard spiking solution. For the low-level method, the autosampler transfers 5 mL of reagent water along with the appropriate amount of IS/SS spiking solution to the 40-mL vial.

5. Purge and desorb according to conditions listed in Calibration A.2.

6. Collect GC/MS data until the end of the GC run.

7. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

8. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table 2) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section). The following table describes the guidelines for an acceptable initial calibration:

Frequency	Acceptance Criteria	Corrective Action
Initially and then when analytes in the daily calibration standard fail criteria.	1. % RSD of ≤20% is required for all analytes. 10% of the analytes may fail this criteria. 2. All compounds of interest must be detected in the MDL standard. 3. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance. 4. Minimum response factors must be met for select compounds. See Table 3.	1. Any target analyte with a %RSD of ≤20% must use the average RRF for quantitation. For any analyte in which the %RSD >20%, a first-degree linear regression can be used (providing that the correlation coefficient [CC] is ≥0.995). A quadratic fit ** (using 6 stds) can also be used (provided the coefficient of determination [CD] is >0.99). If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y-intercept quantifies to be greater than the LOQ, consult your supervisor

Frequency	Acceptance Criteria	Corrective Action
		immediately or recalibrate. If CC or CD is <0.995 for linear or <0.99 for quadratic, recalibrate. Supervisory approval is required for exceptions to these guidelines. If >10% target analytes fail, recalibration is required. If a linear or quadratic curve fit is used for a target analyte, the calibration data must be refit back into the calibration model for all calibration levels. Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest calibration point. 2. If a compound is not detected in the MDL standard, then report to the level of the lowest standard detected. 3,4. Perform system maintenance and recalibrate.

**Consult USEPA method 8000D for non-linear curve fitting techniques/guidelines

9. Each GRO level is analyzed as described above. Next, tabulate the area response for the GRO quantitation range minus the peak areas for the internal and surrogate standards that elute within the GRO range. Calculate the relative response factor (RRF) for GRO (see Calculation section) using the internal standard peak area for Fluorobenzene. Although four internal standard compounds are spiked for the 8260C analysis, only Fluorobenzene, is used for the quantitation of the GRO result.

10. Calculate the average relative response factors for the GRO quantitation range of interest. The calibration levels are evaluated on the basis of the relative standard deviation of the RRF values (%RSD). The %RSD for the GRO range of interest must be ≤20%. If the calibration meets this requirement, then the average RRF is used to calculate sample concentrations. If the %RSD is >20% then re-analysis of one or more levels can be necessary before the calibration is valid.

D. Initial Calibration Verification:

Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV is prepared according to the TSC sheet in Figure 5. The ICV acts as a second source standard to check against the initial calibration. All analytes must meet ICV acceptance windows of 70%-130%. If the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

For the GRO analysis, the ICV is prepared according to the TSC sheet in Figure 6 (QC prep). The ICV acts as a second source standard to check against the initial calibration. Results must quantitate within the 70-130% window. If the ICV does not meet the aforementioned criteria, a second ICV can be run before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

E. Continuing Calibration:

Continuing Calibration Verification (CCV) – The CCV is performed by analyzing a 50-ppb calibration standard (prepared daily due to aqueous nature) in subsequent tune periods after an initial calibration. The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours.		

Frequency	Acceptance Criteria	Corrective Action
	1. % Drift of ≤20% is required for all analytes. No more than 20% of analytes may fail this criteria if not detected in proceeding samples. 2. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 3. The extracted ion current profile (EICP) area for each internal standard must fall within the window of −50 % to +100 % from the mid-level standard area produced during the last initial calibration. 4. Minimum response factors must be met for select compounds. See Table 3.	1. - 4. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

A 1100-ppb standard is analyzed for the GRO analysis. The calibration is considered valid if the percent drift is ≤20%. Also, the internal standard peak area of fluorobenzene for the CCV is monitored against the mid-point standard of the initial calibration and must be −50% to +100% of the area counts. If any criteria listed above fails, the CCV is considered invalid. In the case where two consecutive CCVs fail, corrective action must be taken which can include re-analysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration is used for blank and sample calculations until the end of the 12-hour period.

F. MDL Sensitivity Check- A MDL Sensitivity Check must be analyzed in cases where compounds fail to meet the % drift criteria in the CCV and have decreased sensitivity (-20% drift or greater). Affected compounds can be reported as non-detects if it is demonstrated that there is adequate sensitivity to detect the compound at the MDL. If the failed compound is detected, the concentration must be reported as an estimated value.

Calibration Calculations

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression equations:

1st Order (linear) regression: $Y = Mx + B$

2nd order (quadratic) regression: $Y = Cx2 + Mx + B$

Where:

x = Area(Std) /Area(Istd)

Y = Conc.(Std)/Conc.(Istd)

M = 1st degree slope

C = 2nd degree slope

B = Y-intercept

3. Percent relative standard deviation (%RSD):

$$\%\ RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

4. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:
C(i) = Calibration check compound standard concentration
CI = Measured concentration using selected quantification method

Procedure

A. Method Blank:

Analyze the method blank as described above for the initial calibration standards using reagent water. Refer to table in QA/QC section for specific requirements. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the associated samples. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. The method blank must be analyzed before processing any samples.

B. Laboratory Control Sample/ Duplicate and Matrix Spike/Duplicate: Refer to table in QA/QC section for specific requirements.

C. Qualitative Analysis:

Sample analysis for soil, solids, and nonaqueous matrices proceeds as described in the Calibration section. The aqueous matrix is replaced by the soil sample, which is prepared according to *S-SS-WI10682* and *S-SS-WI11170*. A

compound is identified by comparison of the following parameters with those of a standard of this target compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The compound relative retention time must compare within ±0.06 RRT units of the RRT of the standard.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. (Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the above criteria and analyst discretion are used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, if a compound fails any of the criteria listed above but in the judgment of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed. The primary and secondary ions can be found in Table 2.

7. For the GRO analysis, the Total Ion Chromatogram (TIC) is reviewed to insure proper integration around the 8260 surrogates and internal standards. Also the TIC is checked to make sure all major peaks are integrated.

D. Quantitative Analysis:

Once a compound has been identified, quantitation of identified priority pollutant compounds is performed using the equations listed in the Calculations section of this SOP. The primary ions listed in Table 2 are used for quantitation. A secondary ion may be used if there is interference with the primary ion. All calculations must report concentrations in values of µg/kg. Any analyte with a calculated concentration above the highest standard must be reanalyzed at a dilution that brings the concentration in the solution within the calibration curve. It is desirable to have the dilution fall within the top half of the calibration curve, but it is not required.

The quantitation of the GRO range is performed using the equations listed in the Calculations section of this procedure. All calculations must report concentrations in values of µg/L. In the case where the total GRO concentration exceeds the calibration range, the sample is re-analyzed at a dilution that brings the GRO concentration within the calibration range of the GC/MS system.

Calculations

A. Analyte Concentration

1. Low level

$$\text{Concentration } (\mu g/kg) \frac{(Ax)(Is)}{(Ais)(RRF)(Ws)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured
Ais = Area of the quantitation ion peak for the appropriate internal standard
Is = Amount of internal standard added in nanograms
Ws = Weight of sample purged
RRF = Relative response factor from the initial calibration

2. Medium/high level

$$\text{Concentration } (\mu g/kg) \frac{(Ax)(Is)(Vt)}{(Ais)(RRF)(Ws)(Vi)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured
Ais = Area of the quantitation ion peak for the appropriate internal standard
Is = Amount of internal standard added in nanograms
Vt = Volume of the total extract in microliters
Vi = Volume of the extract used for purging in microliters
Ws = Weight of sample extracted
RRF = Relative response factor from the initial calibration

B. QC Calculations:

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

C. Relative percent difference (RPD)

$$RPD = \frac{MSR - MSDR}{(1/2)(MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

Statistical Information/Method Performance

The LCS must contain 80% to100% of the compounds in the calibration mix. LCS, MS, and surrogate recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived but must fall within 70% to 130% recovery for South Carolina compliance samples. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated on an annual basis in accordance with 40 CFR Part 136. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

Each analysis batch must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). Additional QC samples may be required to meet project or state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds.

The laboratory will establish the Lower Limit of Quantitation (LLOQ) at concentrations where both quantitative and qualitative requirements can consistently be met for 8000D. The LLOQ is the lowest concentration at which the

laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The LLOQ must be verified annually per instrument and is performed by the analysis of an LCS at 0.5-2 times the established LLOQ, using laboratory reagent water and following all preparation and analytical procedures as are used for environmental and QC samples. Acceptance criteria for target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% may be used for the LLOQ acceptance criteria.

The GC/MS system must be tuned to meet the criteria in Table 1 following BFB injection. The chromatographic conditions must be the same as those under which the samples are analyzed except that the rate of temperature ramping may be increased and the initial temperature and column flow may be different. The BFB tune must be verified every 12 hours.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards • Added to every sample, standard, method blank or QC sample	1. Peak areas within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.
Surrogates • Added to every sample, standard, method blank or QC sample	All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi -annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment. If recoveries are outside of specification high and no target compounds are detected then a reanalysis or comment is not required.
Method Blank (MB) Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples) • 1 scoop of Sodium Bisulfate is added if the preservative was added to the initial calibration. • Approximately 5 grams of sand is required for low level analysis.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1,2. Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they must be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) • LCS analyzed with each batch of ≤ 20 samples • LCSD analyzed if MS/MSD unavailable • Approximately 5 grams of sand is required for low level analysis. • 1 scoop of Sodium Bisulfate is added if the preservative was added to the initial calibration.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi -annual basis.	1,2. If non-compliant, check for calculation or preparation errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with an LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the

Quality Control Item	Acceptance Criteria	Corrective Action
• See Figures 2, 3 and 4 for preparation info.		sample must be analyzed with a compliant LCS.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) • MS/MSD analyzed with each batch of ≤ 20 samples (if sufficient sample volume available) • See Figures 2 and 3 for preparation info.	1. % Recoveries must fall within statistically derived QC limits, which are reviewed and updated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition, the method blank may not contain any target compound above the reporting limit for the associated samples. All method blanks must meet these criteria; otherwise, the system is considered out of control and corrective action must be taken.

If sufficient sample volume is not available to perform MS/MSD, LCS/LCSD are prepared and analyzed and must meet the above-mentioned criteria.

NOTE: Prior to release from the analytical laboratory, all data is reviewed in accordance with *T-VOA-WI8373*.

The sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution may be performed if:

1. Prescreening indicates a high volatile organic content in the sample

2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (see *T-VOA-WI7692* for information on when cleaning blanks must be run). See method SW-846 8260C for the recommended dilution procedures.

Table 1

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 2

Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

Continued
Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119

Compound Name	Primary Ion	Secondary Ion
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzene-d4	152	115

Table 3
Minimum Relative Response Factors For ICAL and CCV

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100

Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100

Table 3 Continued

Volatile Compounds	Minimum Response Factor
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	

eurofins	Always check on-line for validity. **VOCs and GRO by GC/MS in Waters and Wastewaters by EPA 8260C/D**	Level: **Work Instruction**
Document number: **T-VOA-WI8194**		
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Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **18-JUL-2019**	Document users: **6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Level II Peer Review, 6_EUUSLA_GC/MS Volatiles_Management, 6_EUUSLA_GC/MS Volatiles_SW846 8260B/C Water**	Responsible: **5_EUUSLA_GC/MS Volatiles_Manager**

LIMS ID
Analysis 11996, 11997, 13130

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Revision Log

	Revision: 8	**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per G-DC-SOP16244	Removed revision logs up to the previous version
Sample Collection, Preservation and Handling	Current practice	Added verbiage concerning Acrolein and Acrylonitrile in regard to pH and holding time.
Calibration	Process change	Removed MDL standard requirement from initial calibration

	Revision: 7	**Effective Date:** April 11 2019
Section	**Justification**	**Changes**
Title	Updated scope	Updated title to reflect new methods
Revision Log		Removed revision logs up to the previous version

	Revision: 7	**Effective Date:** April 11 2019
Section	**Justification**	**Changes**
	Formatting requirement per G-DC-SOP16244	
Reference	Updated scope	Added 8260D reference

Reference

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260C, August 2006.

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260D, June 2018.

2. Determinative Chromatographic Separations, SW-846 Method 8000D, Revision 4, July 2014.

3. Purge and Trap for Aqueous Samples, SW-846 Method 5030B, December 1996.

4. Purge and Trap for Aqueous Samples, SW-846 Method 5030C, Rev 3, May 2003.

5. *Total Petroleum Hydrocarbons Analysis-Gasoline Method*, California Department of Health Services, LUFT Task Force.

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-VOA-WI7691	Glassware Washing
T-VOA-WI7619	GC and GC/MS Instrumentation Maintenance
T-VOA-WI7629	GC/MS Volatile Standards Traceability
T-VOA-WI7692	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
T-VOA-WI8373	GC/MS Volatiles Audit Process
T-VOA-WI8400	Level II Review of GS/MS Volatiles

Scope

This method is suitable for the determination of the target compounds listed and maintained in the LIMS (Laboratory Information Management System) for aqueous matrices. Associated MDLs/LOQs are also listed in the LIMS under the analysis number and/or Project Information lists. Non-target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration. Compounds other than those listed in the LIMS for this group of master scans are analyzed using USEPA SW-846 Method 8260C. Theoretical Standard Calibration (TSC) Sheets are included in the Appendix (Figures 1-7). These TSC sheets are to serve as examples only and may not reflect most current version in use. Due to poor purging efficiency or poor gas chromatographic performance, some analytes require calibration at higher levels and higher practical quantitation limits (PQLs). Any additional compounds must be added to the theoretical standard concentrations (TSC) sheet. Standards containing additional analytes must be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions must be prepared for use in analyzing additional compounds.

Basic Principles

A 5-mL or 25-mL sample or a dilution of a sample is placed in a specially designed purge vessel. The sample is purged with an inert gas and the effluent gas passed through a sorbent tube where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed on to the head of a gas chromatographic (GC) capillary column. The GC column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the Mass Spectrometer, it is bombarded by high-energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focused into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

The GRO analysis is typically performed in conjunction with the analysis of other volatile target compounds by SW-846 Method 8260C. The GRO quantitation range is 0.1 minutes before the peak apex of C6 (hexane) to 0.2 minutes after the peak apex of C12 (dodecane); however, other ranges can be established. By establishing a (C12) GRO window to 0.2 minutes following the elution of dodecane, the areas from a trio of unresolved peaks eluting near to the upper limit of the range must consistently be included in the total GRO area. In addition, the range remains tight enough to ensure that no C13 or greater compounds can be included in the total GRO area. The C4 range retention time is determined by selecting the first peak after the air and/or artifact peak minus 0.1 minutes in the first standard analyzed in the ICAL. The C5 range retention time is 0.1 minutes before the peak apex of pentane. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies. The area of the total ion chromatogram for the GRO range is determined. The area of the internal standards and surrogate standards are found and subtracted from the total area of the chromatogram within the desired time range. The resulting area is then quantitated versus the internal standard, fluorobenzene.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated waters can interfere with the internal standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of SW-846 Method 8260C for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, safety glasses, lab coats, and gloves. Neat compound sources and stock solutions must be collected into a lab pack upon expiration. The lab pack is delivered to Safety personnel for appropriate disposal. Expired secondary standard solutions in methanol must be disposed of as solvent waste. Pour expired secondary standard solutions into the appropriate solvent waste collection container. Aqueous calibration standard mixes are disposed of as nonhazardous aqueous waste due to the low concentration. Samples with a pH ≤2 are taken to storage until disposal in an acid waste container.

Personnel Training and Qualifications

Education Requirement: A 4-year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or one to three years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis must be documented by each analyst in his/her training record. Specifically, each new chemist trains with an experienced chemist for the first 12 weeks depending on the individual and his/her previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time is less if the new chemist has prior relevant experience in GC/MS and/or analytical chemistry background.

During the training period, the new chemist learns daily maintenance, calibration techniques, data and library search review, and forms generation. He/she is also required to read all relevant SOPs and EPA methods. All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP. Each analyst must perform an Initial Demonstration of Capability to measure proficiency. Thereafter, an annual Demonstration of Capability is performed and maintained in the analyst's training records.

NOTE: A separate Demonstration of Capability for GRO is required.

Sample Collection, Preservation, and Handling

The samples to be analyzed with this method must be stored in a refrigerator at 0°C to 6°C, not frozen. Samples are collected in 40-mL vials with no headspace. Preserve samples to a pH of <2 in order to prevent degradation of aromatic compounds that are present in the sample. 1:1 HCL is the recommended preservative. Preserved samples must be analyzed within 14 days of collection; those that are not preserved must be analyzed within 7 days of collection. Samples to be tested for Acrolein and Acrylonitrile must be collected without headspace in vials that are preserved to a pH range of 4.0 to 5.0 and must be analyzed within 7 days of collection. Samples may also be submitted in unpreserved vials for Acrolein and Acrylonitrile, but must be analyzed within 3 days of collection. Samples submitted outside of the pH range or analyzed outside of the specified holding time windows will be reported with a comment on the final analytical report.

Apparatus and Equipment

1. Gastight micro-syringes – 1 to 1000 µL (various sizes)

2. 5-mL gastight syringes

3. Analytical balance, capable of accurately weighing ±0.0001 g

4. Glassware

   a. Class-A Volumetric flasks with ground-glass stopper

   b. Vials, 1.5-mL, 15-mL, and 40-mL screw cap, with Teflon™/silicone septa

   c. Mininert vials, 1 mL, 2 mL, and 5 mL

5. Purge and trap device – Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging chamber must have the purge gas passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

6. Autosampler – OI Analytical 4551, OI Analytical 4552, Archon, or equivalent meets the requirements of this method.

7. Spiker unit – OI analytical Model 4551/4552 SAM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551/4552 autosampler to automatically introduce 1 µL of internal standard (ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

8. GC/MS system – The Agilent 5890GC/5972 MSD, Agilent 6890GC/5973MSD, Agilent 6890GC/5975MSD and Shimadzu GC/MS QP5000 meet the requirements for this method.

9. Data System/Computer/Software – this is interfaced to the GC/MS system that continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

10. GC Columns

a. Column 1 – 30M × 0.25 mm ID DB624 capillary column with a 1.4-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

b. Column 2 – 20M × 0.18 mm ID DB624 capillary column with a 1.0-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

c. Column 3 – 20M × 0.18 mm ID DB-VRX capillary column with a 1.0-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

NOTE: Refer to *T-VOA-WI7619* for instrumentation maintenance and troubleshooting.

Reagents and Standards

A. Reagents

1. Reagent water is defined as water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol, Purge and Trap Grade or equivalent.

B. Standards

See *T-VOA-WI7629* for standards traceability.

1. Stock standard solutions – Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≤96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at ≤-10°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

a. For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate standards are purchased from a vendor, as well as the target compounds that are gases at room temperature. These gaseous standards have a 1-week expiration date, starting from the date they are opened.

b. 8260A Surrogate standard spiking solution (8260SS) – a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4-bromofluorobenzene, and 1,2-dichloroethane-d4 is prepared in methanol by a commercial supplier.

c. 8260A Internal standard spiking solution (8260IS) – a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, and 12500 µg/mL deuterated tertiary butyl alcohol (tBA-d10) is prepared in methanol by a commercial supplier. Deuterated tertiary butyl alcohol (tBA-d10) is used sometimes as an auxiliary ISTD.

d. GRO calibration standard – a 5500-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier.

e. GRO QC standard – a 20,000-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier

To prepare stock standards from neat compounds:

(1) Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

(2) Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Reweigh the flask, record/note the amount, dilute to volume, stopper, and mix by inverting the flask at least 3 times. Calculate the concentration of the standard.

(3) The stock standard solutions are stored in Teflon™-sealed screw-capped vials at ≤-10°C. The compound name, concentration, date prepared, expiration date, preparer name and storage method must appear on the bottle.

(4) Replace in-house prepared stock standard solutions every 6 months.

2. Secondary dilution standards - Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with a known concentration of each analyte. When custom mixes are used, these are diluted down individually or combined together with other mixes. The working standard is tested according to *T-VOA-WI7629*. The verified working standard is poured into Teflon-lined screw-capped GC vials or mininert vials and stored at ≤-10°C. A designator indicating the standard name, month, and day of preparation and expiration date must be on the standard vials. The designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 2-4 weeks (1 week for standards containing gases) unless otherwise indicated.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50-µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used varies depending on the actual stock concentration.

b. IS/SS spiking solution – Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10-mL final volume (resulting in a concentration of 250 µg/mL, 1250 µg/mL for tBA-d10). This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.(50& µg/L for 5-mL purge, 10 µg/L for 25-mL purge).

c. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 1, 4, 10, 20, 50, 100, and 300 ppb for 5-mL purge analysis. Suggested calibration levels are 0.2, 0.5, 1, 2, 5, 10, and 25 ppb for 25-mL purge analysis. A Theoretical Standard Concentration (TSC) sheet is filled out for all initial calibrations (see Figures 1, 2, and 5). Replace calibration spiking solution every 2-4 weeks (1 week for standards containing gases).

d. Matrix spiking solution – Prepare second source solutions in methanol that contain the compounds of interest at known concentrations. A TSC sheet is filled out for all quality control samples (see Figures 4 and 5). These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates. Therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace matrix spiking solution every 2-4 weeks (1 week for standards containing gases).

Store all standard solutions at ≤-10°C and protected from light.

Preparation of Glassware

All glassware is cleaned according to *T-VOA-WI7691*.

Calibration

A. Instrument conditions

1. The purge and trap device must have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge Gas	Helium
Purge Flow	35 - 45 mL/min
Purge Temperature	40°C for 8260C waters
Purge Time	11 minutes
Desorb Temperature	190°- 220°C
Desorb Time	0.5 to 4 minutes **
Bake Temperature	180°-220°C
Bake Time	5 – 16 min

**Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions are changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument is found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	Column 2	Column 3
Column liquid phase	DB-624	DB-624	DB-VRX
Carrier gas	Helium	Helium	Helium
Carrier gas flow	0.8 mL/min	0.6 mL/min	0.6 mL/min
Make-up gas flow	None	None	None
Initial temperature	45°C	45°C	45°C
Initial hold time	4.5 min	2.5 min	4 min
Temperature ramp	12°/min until 100°C then 25°/min until 240°C	12°/min until 100°C then 25°/min until 235°C	25°/min until 60°C then 36°/min until 240°C
Final temperature	240°C	235°C	240°C
Final hold time	None	.02 min	1 min

4. The recommended mass spectrometer operating conditions are listed below:

Mass range:	35 – 300 amu
Scan time:	One scan cycle per second or less and resulting in at least five scans per chromatographic peak

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

B. Tuning

Tune the GC/MS system to meet the criteria in Table 1 following a 50-ng injection of BFB. The chromatographic conditions must be the same as those under which the samples are analyzed except that the temperature ramp is increased and the initial temperature and flow rate is different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same MS parameters as those used to obtain a successful tune.

C. Initial calibration

1. The initial internal standard calibration consists of analyzing seven distinct levels of analyte concentrations and producing response factors for each compound (six levels are required if second order regression fits are used). Calibration for GRO consists of analyzing six distinct levels of GRO area in order to produce a response factor for the GRO quantitation range of interest using the internal standard, fluorobenzene. The relative standard deviation of the response factor determines the suitability of the average relative response factor for calculation of the GRO concentration. Refer to Figure 1, 2 or 5 for the preparation of the calibration standards and Figure 7 for preparation of GRO standards. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration. Prior to the analysis of any calibration level, retention time markers must be run for the GRO range of interest. The retention time markers are hexane (C6) and Dodecane (C12). Other markers can be used if different ranges are required by a project.

NOTE: 5 levels of standard are required by the method.

a. When using an OI 4552 or OI 4551 autosampler, the standards (including target and surrogate compounds) are prepared and poured into 40-mL vials with Teflon™-lined septa. A 5-mL or 25-mL aliquot is withdrawn from the vial by the autosampler. The aliquot is transferred through the spiker unit to add the IS/SS spiking solution and then transferred to the sparge vessel.

b. Purge and desorb according to instrument conditions listed in Calibration A.

c. Collect GC/MS data until the end of the GC run.

d. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

e. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table 2) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section). The calibration is valid for 12 hours from the injection of the BFB tune standard, at which time a new tune check and a continuing calibration check standard are evaluated prior to the analysis of additional samples. The following table describes the guidelines for an acceptable initial calibration:

Frequency	Acceptance Criteria	Corrective Action
Initially and then when analytes in the daily calibration standard fail criteria.	1. % RSD of≤20% is required for all analytes. 10% of the analytes may fail this criteria. 2. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance. 3. Minimum response factors must be met for select compounds. See Table 3.	1. Any target analyte with a %RSD of ≤20% must use the average RRF for quantitation. For any analyte in which the %RSD >20%, a first-degree linear regression can be used (providing that the correlation coefficient [CC] is ≥0.995). A quadratic fit ** (using 6 stds) can also be used (provided the coefficient of determination [CD] is >0.99). If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y-intercept quantifies to be greater than the LOQ, consult your supervisor immediately or recalibrate. If CC or CD is <0.995 for linear or <0.99 for quadratic, recalibrate. Supervisory approval is required for exceptions to these guidelines. If >10% target analytes fail, recalibration is required. If a linear or quadratic curve fit is used for a target analyte, the calibration data must be refit back into the calibration model for all calibration levels. Percent error between the calculated and expected amounts of an analyte should be ≤30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point. 2.-3. Perform system maintenance and recalibrate.

**Consult USEPA method 8000D for non-linear curve fitting techniques/guidelines

f. Each level is analyzed as described above. Next, tabulate the area response for the GRO quantitation range minus the peak areas for the internal and surrogate standards that elute within the GRO range. Calculate the relative response factor (RRF) for GRO (see Calculation section) using the internal standard peak area for fluorobenzene. Although four internal standard compounds are spiked for the 8260C analysis, only one, fluorobenzene, is used for the quantitation of the GRO result.

Calculate the average relative response factors for the GRO quantitation range of interest. The calibration levels are evaluated on the basis of the relative standard deviation of the RRF values (%RSD). The %RSD for the GRO range of interest must be ≤20%. If the calibration meets this requirement then the average RRF is used to calculate sample concentrations. If the %RSD is >20% then re-analysis of one or more levels can be necessary before the calibration is valid.

D. Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV acts as a second source standard to check against the initial calibration. All analytes must meet ICV acceptance windows of 70%-130%. If the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken. For the GRO analysis, the ICV is prepared according to the TSC sheet in Figure 7 (QC prep). The ICV acts as a second source standard to check against the initial calibration. Results must quantitate within the 70-130% window. If the ICV does not meet the aforementioned criteria, a second ICV can be run before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

E. Continuing calibration verification (CCV) – The CCV is performed by analyzing a CCV standard in subsequent tune periods after an initial calibration. The CCV (prepared daily due to aqueous nature) is analyzed at 50 ppb for 5-mL purge waters and 10 ppb for 25-mL purge waters. The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action
Every 12 hours.	1. % Drift of ≤20% is required for all analytes. No more than 20% of analytes may fail this criteria if not detected in proceeding samples. 2. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 3. The extracted ion current profile (EICP) area for each internal standard must fall within the window of –50 % to +100 % from the mid-level standard area produced during the last initial calibration. 4. Minimum response factors must be met for select compounds. See Table 3.	1.-4. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

A 1100-ppb standard (prepared daily due to aqueous nature) is analyzed for the GRO analysis. The calibration is considered valid if the percent drift is ≤20%. Also, the internal standard peak area of fluorobenzene for the CCV is monitored against the mid-point standard of the initial calibration and must be –50% to +100% of the area counts. If any criteria listed above fails, the CCV is considered invalid. In the case where two consecutive CCVs fail, corrective action must be taken which can include re-analysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration is used for blank and sample calculations until the end of the 12-hour period.

F. MDL Sensitivity Check- A MDL Sensitivity Check must be analyzed in cases where compounds fail to meet the % drift criteria in the CCV and have decreased sensitivity (-20% drift or greater). Affected compounds can be reported as non-detects if it is demonstrated that there is adequate sensitivity to detect the compound at the MDL. If the failed compound is detected, the concentration must be reported as an estimated value.

Procedure

A. Method Blank

Analyze the method blank as described above for the initial calibration standards using reagent water. Refer to table in QA/QC section for specific requirements. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the associated samples. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. The method blank must be analyzed before processing any samples.

B. Laboratory Control Sample/ Duplicate and Matrix Spike/Duplicate: Refer to table in QA/QC section for specific requirements.

C. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The compound relative retention time must compare within ±0.06 RRT units of the RRT of the standard.

3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

4. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the criteria listed above and the analyst's discretion is used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, if a compound fails any of the criteria listed above but in the judgment of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed.

The primary and secondary ions for the target compounds can be found in Table 2.

7. For GRO, the Total Ion Chromatogram (TIC) is reviewed to insure proper integration around the 8260 surrogates and internal standards. Also the TIC is checked to make sure all major peaks are integrated.

D. Quantitative analysis

Once a compound has been identified, quantitation is based on the internal standard technique and the integrated area from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions is listed in Table 2.

The quantitation of the GRO range is performed using the equations listed in the Calculations section of this procedure. All calculations must report concentrations in values of µg/L. In the case where the total GRO concentration exceeds the calibration range, the sample is re-analyzed at a dilution that brings the GRO concentration within the calibration range of the GC/MS system.

E. Sample Analysis

A 5-mL or 25-mL aliquot of the sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution is performed if:

1. Prescreening indicates a high volatile organic content in the sample

2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (See *T-VOA-WI7692* for information on when cleaning blanks must be run). See Section 11.5.6 in method SW-846 8260C for recommended dilution procedures.

Calculations

A. Calibration calculations

1. Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression Equations:

1st Order (linear) regression: Y = MX + B

2nd order (quadratic) regression: Y = CX2 + MX + B

Where:
x = Area(Std) /Area(Istd)
Y = Conc.(Std)/Conc.(Istd)
M = 1st degree slope
C = 2nd degree slope
B = Y-intercept

3. Percent relative standard deviation (%RSD):

$$\%\ RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

4. Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:
C(i) = Calibration check compound standard concentration
C(c) = Measured concentration using selected quantification method
B. QA Calculations

1. Calculation of percent recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

2. Relative percent difference (RPD):

$$RPD = \frac{MSR - MSDR}{(1/2)\ (MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

3. Analyte Concentration

$$Concentration\ (\mu g/L) = \frac{(Ax)\ (Is)}{(Ais)\ (RRF)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Concentration of internal standard added in µg/L

RRF = Relative response factor from the initial calibration

Statistical Information/method Performance

The LCS must contain 80% to100% of the compounds in the calibration mix. LCS, MS, and surrogate recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived but must fall within 70% to 130% recovery for South Carolina compliance samples. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated on an annual basis in accordance with 40 CFR Part 136. The MDLs are maintained in the LIMS. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

Each analysis batch (consisting of no more that 20 samples) must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), a laboratory control sample/laboratory control sample duplicate (LCS/LCSD) or a duplicate (DUP). The LCS serves as a 2nd source standard verification of the initial calibration (ICAL). Additional QC samples are required to meet project or

state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds at a concentration of 50 µg/L for a 5-mL purge or 10 µg/L for a 25-mL purge.

The laboratory will establish the Lower Limit of Quantitation (LLOQ) at concentrations where both quantitative and qualitative requirements can consistently be met for 8000D. The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be = the lowest point in the calibration curve. The LLOQ must be verified annually per instrument and is performed by the analysis of an LCS at 0.5-2 times the established LLOQ, using laboratory reagent water and following all preparation and analytical procedures as are used for environmental and QC samples. Acceptance criteria for target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% may be used for the LLOQ acceptance criteria.

The GC/MS system must be tuned to meet the criteria in Table 1 following BFB injection. The chromatographic conditions must be the same as those under which the samples are analyzed except that the rate of temperature ramping may be increased and the initial temperature and column flow may be different. The BFB tune must be verified every 12 hours.

Quality Control Item	Acceptance Criteria	Corrective Action
1. Internal Standards Added to every sample, standard, method blank and QC sample	1. Peak areas within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.
2 Surrogates Added to every sample, standard, method blank and QC sample	All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment. If recoveries are outside of specification high and no target compounds are detected, then a reanalysis or comment is not required.
3 Method Blank (MB) Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1.-2.Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they must be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)
Laboratory Control Sample/Laboratory Control	1. Must meet internal standard criteria.	1.-2. If non-compliant, check for calculation or preparation

Quality Control Item	Acceptance Criteria	Corrective Action
Sample Duplicate (LCS/LCSD) LCS analyzed with each batch of ≤ 20 samples LCSD analyzed if MS/MSD unavailable See Figure	2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with a LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the sample must be analyzed with a compliant LCS.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) MS/MSD analyzed with each batch of ≤ 20 samples (if sufficient sample volume available) See Figures 4 and 5 for preparation info.	1. % Recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

NOTE: Prior to release from the analytical laboratory, all data is reviewed in accordance with *T-VOA-WI8373* or *T-VOA-WI8400* (dual purge and trap).

Table 1

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 2

Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64

Compound Name	Primary Ion	Secondary Ion
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
trans-1,2-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77, 110
trans-1,3-Dichloropropene	75	77, 110
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

Table 3
Minimum Relative Response Factors For ICAL and CCV

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200

1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100

Table 3 - Continued

Volatile Compounds	Minimum Response Factor
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

T-VOA-WI7619 GC and GC/MS Instrumentation Maintenance
T-VOA-WI7629 GC/MS Volatile Standards Traceability
T-VOA-WI7691 Glassware Washing
T-VOA-WI7692 Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
T-VOA-WI8373 GC/MS Volatiles Audit Process
T-VOA-WI8400 Level II Review of GC/MS Volatiles

Attachment:
Figure 1
Figure 2
Figure 3
Figure 4
Figure 5
Figure 6
Figure 7

End of document

Version history

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QM Approval

Name/Signature	Title	Date	Meaning/Reason
Patrick Letterer (005125)	Quality Manager	22 Aug 2019, 02:11:33 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Patrick Letterer (005125)	Quality Manager	22 Aug 2019, 02:13:04 PM	Approved
Nicholas Nigro (005056)	General Manager	22 Aug 2019, 02:24:15 PM	Approved
Benjamin Claas (005049)	Laboratory Supervisor III	22 Aug 2019, 02:48:13 PM	Approved

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. The purpose of this method is to provide a procedure to measure the concentration of specified volatile organic compounds (VOCs) in water, soil, and rock samples using solid phase micro-extraction (SPME) and a Gas Chromatograph equipped with a capillary column and mass spectrometer. Specific VOCs of interest are outlined in Table 5.1. This method is based on USEPA 8260 C/D, Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS), and ASTM Method D6520, Solid Phase Micro-extraction of water and its headspace for analysis of Volatile and Semi-volatile Organic Compounds.

2. SUMMARY OF METHOD

2.1. This method provides gas chromatographic (GC) and mass spectrometer (MS) conditions for the detection of certain VOCs. Samples are prepared in 20 mL extraction vials where the SPME fiber is exposed to the headspace above the sample for eight minutes while the sample is agitated via either stirring or orbital shaking. The exposure time must be consistent for all standards and samples, which is accomplished using an autosampler. The absorbed analytes are thermally desorbed from the SPME fiber in the injection port of a GC and transferred to a narrow bore capillary column. A temperature program is used to enhance separation as the analytes are carried through the column to the ion source of the mass spectrometer (MS). The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by a quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion or selected ion chromatogram and mass spectra for every point on the chromatogram. The instrument's mass spectrometer is set to selective ion monitoring mode (SIM) to achieve lower reporting limits for the VOCs of interest.

3. SCOPE AND APPLICATION

3.1. This method is applicable to most organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates). The target compounds for this method are shown in Table 5.1.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interfere with sample analysis.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of SPME/GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. APPLICABLE MATRICES

4.1. This SOP is applicable to aqueous and solid matrices.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. The limits of quantitation (LOQ) for all analytes can be found Table 5.1. All current MDLs are listed in the LIMs and are available by request from the Quality Manager.

Table 5.1 – Limits of Detection and Quantitation

Analyte	CAS #	**Minimum RRF	LOQ/RL Water (µg/L)	LOQ/RL Soil/Rock (µg/kg)
Chloromethane	74-87-3	0.01	2	80
+Vinyl Chloride	75-01-4	0.01	2	80
+Chloroethane	75-00-3	0.1	1	40
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.1	1	40
1,1-Dichloroethene	75-35-4	0.01	1	40
+Carbon Disulfide	75-15-0	0.1	1	40
Methylene Chloride	75-90-2	0.01	1	40
+t-Butyl-methyl ether	1634-04-4	0.1	1	40
trans-1,2-Dichloroethene	156-60-5	0.01	1	40
1,1-Dichloroethane	75-34-3	0.1	1	40
cis-1,2-Dichloroethene	156-59-2	0.1	1	40
+2-Butanone	78-93-3	0.1	20	800
Chloroform	67-66-3	0.1	1	40
1,1,1-Trichloroethane	71-55-6	0.1	1	40
Carbon Tetrachloride	56-23-5	0.1	1	40
Benzene	71-43-2	0.1	1	40
1,2-Dichloroethane	107-06-2	0.01	1	40
Trichloroethene	79-01-6	0.1	1	40
1,2-Dichloropropane	78-87-5	0.1	1	40
+1,4-Dioxane	123-91-1	0.01	2	80
Bromodichloromethane	75-27-4	0.1	1	40
cis-1,3-Dichloropropene	10061-01-5	0.1	1	40
+4-Methyl-2-pentanone	108-10-1	0.1	20	800
Toluene	108-88-3	0.1	1	40
Trans-1,3-Dichloropropene	10061-02-6	0.1	1	40
1,1,2-Trichloroethane	79-00-5	0.01	1	40
Tetrachloroethene	127-18-4	0.1	1	40
+2-Hexanone	591-78-6	0.1	20	800
Dibromochloromethane	124-48-1	0.1	1	40
1,2-Dibromoethane	106-93-4	0.1	1	40
Chlorobenzene	108-90-7	0.1	1	40
Ethylbenzene	100-41-4	0.1	1	40
m+p-Xylenes[1]	108-38-3 106-42-3	0.1	1	40
o-Xylene	95-47-6	0.1	1	40
Styrene	100-42-5	0.1	1	40
Bromoform	75-252	0.1	1	40
Isopropylbenzene	98-82-8	0.1	1	40
p-Bromofluorobenzene (SS)	460-00-4	0.1	1	40
1,1,2,2-Tetrachloroethane	79-34-5	0.1	1	40

Analyte	CAS #	**Minimum RRF	LOQ/RL Water (µg/L)	LOQ/RL Soil/Rock (µg/kg)
1,3-Dichlorobenzene	541-73-1	0.1	1	40
1,4-Dichlorobenzene	106-46-7	0.1	1	40
1,2-Dichlorobenzene	95-50-1	0.1	1	40
+1,2-Dibromo-3-chloropropane	96-12-8	0.1	1	40
1,2,4-Trichlorobenzene	120-82-1	0.1	1	40
1,3,5-Trimethylbenzene	108-67-8	0.1	1	40
1,2,4-Trimethylbenzene	95-63-6	0.1	1	40
Naphthalene	91-20-3	0.1	1	40

+ Poor Performer, these compounds have wider QC tolerances for certain QC parameters – see Table 13.1 for details.
[1]Reference to o-xylene for all m,p-xylene RL and MDLs.
** Minimum response factors for a select number of compounds are lower than what is recommended in 8260C due to the fact that this method employs SPME as opposed to purge and trap. These minimum RFs are acceptable as demonstrated with demonstration of an acceptable LOQ standard. Minimum RF factors only apply to project work being conducted using the EPA 8260C criteria – EPA 8260D does not require that specific RFs are achieved.

6. INTERFERENCES

6.1. Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons, acetone, toluene, and methylene chloride) through the sample container septum during shipment and storage. A trip blank can be used as a check on this type of interference. However, a trip blank is not usually required if the analyses are being conducted onsite. A trip blank is prepared from analyte-free reagent water and carried through sampling and subsequent storage and handling. Equipment and field blanks are typically used for on-site analyses.

6.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the SPME fiber is left exposed in the inlet for 3 minutes. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a method blank or instrument blank to check for cross contamination.

6.3. All glassware and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Store clean dry glassware in a clean environment. SPME vials, caps, and solid reagents should be baked at 100 °C for at least 1 hour prior to use.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Sample Collection

7.1.1. Aqueous samples are collected in pre-cleaned 40 mL Teflon®-lined septum VOA vials filled to ensure that no headspace is present. If storage of these samples is expected to exceed seven days, aqueous samples should be preserved with hydrochloric acid (HCl) to a pH of < 2 in the field.

7.1.2. Soil and rock samples are collected into pre-weighed 40 mL vials with methanol. If required, a separate aliquot of soil or rock from the sample location will be collected for percent moisture determination. If storage of these samples is expected to exceed 14 days then they must

be kept frozen with client/ regulatory approval. Soil samples can also be collected using Lock N' Load syringes (or equivalent) in the field; these samples will be placed into methanol upon receipt at the laboratory.

7.2. Handling and Holding Times

7.2.1. Unpreserved water samples must be analyzed within 7 days of collection. Preserved (preserved with HCl to a pH of < 2) water samples must be analyzed within 14 days of collection. Soil and rock samples preserved in methanol must be analyzed within 14 days from collection. Samples shall be stored at ≤ 6 °C. As per site-specific project team decisions, longer holding times may be deemed acceptable if methanol-preserved solid samples are stored at < 0 °C. Samples should be allowed to come to room temperature just prior to analysis.

7.2.2. If water samples were preserved and analyses are performed after 7 days from collection, pH determination must be performed. Once the sample aliquots have been taken from the VOA vial for analysis, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field to a pH of < 2.

7.2.3. All shipments of samples or standards should be opened in a fume hood or other negative pressure environment so that the sample handler is protected from any potential vapors or other exposure. If samples are suspected to be of high concentration and are not to be immediately analyzed, they will be isolated in a separate cooler unit in the lab.

Sample Type	Collection per Sample	Preservation	Storage	Hold time
Aqueous	Two VOA vials	Acidified w/ 1:1 HCl (1-2 drops) to pH <2, no headspace. OR (on-site) Unpreserved	4 ± 2 °C	Unpreserved: 7 days pH Preserved: 14 days
Soil/rock	Soil: One 10-12g aliquot in vial with 10.0 mL purge and trap grade MeOH. Rock: One 15-20g aliquot in vial with 20 mL purge and trap grade MeOH. *OR (soils only)* Two (2) 10 g Lock N' Load syringes.	Methanol	With methanol: 4 ± 2 °C.	Preserved with methanol: 14 days

8. DEFINITIONS

8.1. Refer to Glossary section of the Pace Quality Manual (QM) for a comprehensive list of terms and definitions.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, column supplies, gases, syringes and data system to determine peak areas and perform integrations. Hewlett Packard 5890 Series II, Agilent 6890N or Agilent 7890A.

9.2. Merlin micro-seal (SPME Microseal – P/N 21-01)

9.3. Mass Spectrometer: Hewlett Packard/ Agilent 5972, 5973, 5975, or 5975T ,

9.4. Agilent ChemStation data acquisition and analyses software. Various revisions, depending on hardware configurations.

9.5. Typical Analytical Column: RTX-VMS 30m x 0.18mm ID, 1.0 um film or equivalent

9.6. SPME Autosampler: CTC Combi PAL configured for SPME

9.7. Sample introduction and preparation apparatus: SPME Fiber type 100 µm, 24 gauge polydimethylsiloxane (PDMS), Supelco Part #57301, Color RED.

9.8. Array of micro-syringes: range 5-1000 uL.

9.9. Balance: Top loading, capable of weighing accurately to 0.01 grams (soil samples only).

9.10. VOA vials: 40 mL collection containers with Teflon®-lined septum

9.11. SPME extraction vials, 20mLwith Teflon®-lined septum .

9.12. Two and four mL vials for standards storage with Teflon®-lined septum.

9.13. pH paper

9.14. Oven

9.15. Orbital Shaker

9.16. Centrifuge

10. REAGENTS AND STANDARDS

10.1. Reagents

Table 10.1 below lists the reagents required to perform this method along with pertinent information.

Table 10.1 Reagents

Reagent	Conc.	Purity	Manufacturer	Vendor	Catalog #
Methanol	100%	P&T grade	Fisher	Various	A45401
Helium gas	UHP	UHP	Praxair	Praxair	NA
Analyte-free spring water	100%	Organic Free	Various	Various	NA
Sand, baked at 400°C overnight	100%	Organic free	Various	Various	NA
Sodium chloride	100%	ACS Grade	EMD Millipore	Various	SX0420-3

10.2. Standards

The following sections discuss and present information regarding the stock and intermediate standard that are needed to perform this method.

10.2.1. Calibration Stock Standards and Intermediate Standard

Table 10.2 Calibration Stock Standards and Intermediate Standard – prepared in a 10 mL volumetric flask, filled to volume with methanol. Subdivide into 5 labeled 2 mL vials. Store in a freezer. Assign an expiration date of 1 month from date of preparation.

Table 10.2 Calibration Stock Standards and Intermediate Standards

Calibration Stock Standard Description	Concentration (µg/mL)	Vendor	Catalog #	Volume Added (µL)	Intermediate Standard, Final Concentration (µg/mL)
Custom VOC Solution (includes surrogate)	2,000	CPI	Z-122819-01-02	50	10
8260B Ketones	20,000	Absolute	82492	50	100
1,4-dioxane	2,000	Restek	30287	50	10

Volumes may be scaled up provided volume ratios are preserved.

10.2.2. Second Source Stock Standards and Second Source Intermediate Standard

Table 10.3 Second Source Stock Standards and Second Source Intermediate Standard - prepared in a 10 mL volumetric flask, filled to volume with methanol. Subdivide into 5 labeled 2 mL injections vials. Store in a freezer. Assign an expiration date of 1 month from date of preparation.

Table 10.3 Second Source Standards

Second Source Stock Standard Description	Concentration (µg/mL)	Vendor	Catalog #	Volume Added (µL)	Second Source Intermediate Standard, Final Concentration (µg/mL)
Custom VOC Solution (includes surrogate)	2000	CPI	Z-122819-01-02-SS	50	10
VOC Calibration Mix #1 Ketones	5000	Absolute	30006	200	100

Second Source Stock Standard Description	Concentration (µg/mL)	Vendor	Catalog #	Volume Added (µL)	Second Source Intermediate Standard, Final Concentration (µg/mL)
1,4-dioxane	1000	Accustandard	APP-9-096-10X	100	10

10.2.3. Internal Standard Stock Standards and Internal Standard

Table 10.4 Internal Standard Stock Standards and Internal Standard Intermediate Standard - prepared in a 10mL volumetric flask, filled to volume with methanol. Subdivide into 2 labeled 4 mL vials. Store frozen. Assign an expiration date of 3 months from date of preparation. The internal standards used for this method are fluorobenzene, 1,4-dioxane-d8 and chlorobenzene-d5.

Table 10.4 Internal Standards

Internal Standard Stock Standards Description	Concentration (µg/mL)	Vendor	Catalog #	Volume Added (µL)	Internal Standard Intermediate Standard, Final Concentration (µg/mL)
Internal Standard Mix	2,000	Accustandard	M-8260A/B-IS	100	20
1,4-dioxane-d8	2,000	Supelco	4M4829-U	200	40

10.2.4. Surrogate Stock Standard and Surrogate Intermediate Standards

Table 10.5 Surrogate Stock Standard and Surrogate Intermediate Standard: Subdivide into labeled 2 mL vials. Store in a freezer. Assign an expiration date of 3 months from date of preparation.

Table 10.5 Surrogate Standards

Surrogate Stock Standard Description	Concentration (µg/mL)	Vendor	Catalog #	Volume Added (µL)	Final Volume (mL)	Surrogate Intermediate Standard, Final Concentration (µg/mL)
Soil/ Rock Surrogate (4-BFB)	25,000	Accustandard	CLP-004-1000X	80	10	200
Water Surrogate (4-BFB)	2,5000	Accustandard	CLP-004-1000X	80	100	20

10.2.5. Working Calibration Standards

Table 10.6 Working Calibration Standards: Prepare as indicated in a 10 mL Leur lock syringe and transferred to a 20 mL SPME headspace vial with 1 teaspoon (approx. 2 grams) of salt. Load onto autosampler. Use a prepared standard on the same working day as it is prepared.

Table 10.6 Calibration Standards

Calibration Level	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	*CAL7	*CAL 8	ICV	TUN
Concentration (µg/L) VOC/Ketones	1/10	2/20	5/50	10/100	20/200	50/500	100/1000	200/2000	20/200	N/A
Volume of calibration intermediate (µL)	1	2	5	10	20	50	100	200	N/A	N/A
Volume of second source intermediate (µL)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	NA	20	N/A
Volume of working internal standard (µL)	10	10	10	10	10	10	10	10	10	10

*These calibration levels are typically not used for the aromatic compounds (i.e., dichlorobenzene isomers). Calibration data packages will specify the range that is used for each of the compounds.

11. CALIBRATION AND STANDARDIZATION

11.1. Allow the GC/MS system to stabilize under high vacuum when starting from a vented state. Before analysis, allow approximately 4 hours for the GC/MS to equilibrate after the pump down event is initiated.

11.2. The GC/MS system is software-tuned by successfully completing a BFB target tune. Perflurotributylamine (PFTBA) is the tuning compound used by the instrument. Further manual adjustments to the instrument optics settings may be also be necessary to successfully pass the following BFB tuning evaluation.

11.3. Introduce BFB using the same SPME process for all samples and QC runs to the GC/MS system. The sample used for this step is the same as a blank which has 20 µg/L of internal standard and BFB.

11.4. The mass spectra acquired for BFB must pass the acceptance criteria provided below in Table 11.3 Table 11.3 BFB Tune criteria

Mass (m/z)	Ion Abundance criteria – 8260 C
50	NA
75	NA
95	50-200% of mass 174
96	5-9% of m/z 95
173	<2% of m/z 174
174	50-200% of m/z 95
175	5-9% of m/z 174
176	95-105% of m/z 174
177	5-10% of m/z 176

11.5. Initial calibration (ICAL) is performed using the internal standard technique by injecting a minimum of 5 of the available calibration standards. Six calibration points are required if it is anticipated that a non-linear curve will be required for any analyte. The lowest calibration point must be at or below the reporting limit. Values below the lowest calibration point must be qualified as estimated values. Acceptance criteria for calibration curves are listed in Table 11.9.

11.5.1. Response factors (RF) establish the relationship of the instrument's response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard (IS) in an inverse manner, therefore, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis.

11.5.2. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_s C_{is}}{A_{is} C_s}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_s = Concentration of the compound being measured (µg/L).

11.5.3. The average response factor (RFavg) is determined by averaging the response factors for each target analyte over the calibration range. A calculation of the percent relative standard deviation (%RSD) for each analyte is used to determine the acceptability of the use of the average response factor for quantitation. If the percent relative standard deviation (%RSD) of the RFs for a compound is <20% over the calibration range, then linearity through the origin is assumed and the RFavg may be used to determine sample concentrations.

11.5.4. $\% \boldsymbol{RSD} = \left(\frac{RF_{STDEV}}{RF_{AVE}}\right) \times \mathbf{100}$

Where:

RF_{STDDEV} = Standard deviation of individual response factors

RF_{AVE} = Average of individual response factors

11.5.5. If the % RSD for any compound is >20%, the analyst may use a linear regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient (r) must be > 0.995. See Table 11.5 for calibration curve acceptance limits.

11.5.6. A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants, below the response of the lowest calibration point, may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r2. The COD or r2 from the regression equation must be > 0.99. As noted in Table 10.2.6, there are several aromatic compounds (i.e. dichlorobenzene isomers) that commonly require quadratic curve fitting and have reduced calibration ranges than that of the non-aromatic compounds.

11.6. Retention times and characteristic ions for each of the analytes is provided in Attachment A.

11.7. Initial Calibration Verification (ICV)

11.8. To assess the accuracy of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the primary standards. This step is referred to as an Initial Calibration Verification (ICV). This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The ICV is analyzed immediately after the calibration, prior to unknown sample analysis. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$\%\ \text{Difference} = [(\text{Result}_{ICV} - \text{TrueValue}_{ICV}) / \text{TrueValue}_{ICV}] * 100$

11.9. Continuing Calibration Verification (CCV)

11.9.1. The validity of the calibration is checked at the beginning of every analytical sequence, after every 10 samples and at the end of every analytical sequence.

11.9.2. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the criteria in Table 11.9 for the analysis to proceed.

11.9.3. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

% Difference: $[(RF_{CCV} - AvgRF_{CAL}) / AvgRF_{CALI}] \times 100$

% Drift: $[(Result_{CCV} - TrueValue_{CCV}) / TrueValue_{CCV}] \times 100$

Table 11.9 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	8260C Criteria	8260D Criteria	Comments/Corrective Actions
Calibration Curve Fit	Average Response Factor	% RSD ≤20%	% RSD ≤20%	If not met, linear or non-linear regression fits may be used. ***8260D** also requires that the low standard recalculates to within ±50% of the true value of the standard. Other standards are within ±30% of true value.
	Linear Regression	$R^2 \geq 0.990$, min. of 5 points	$R^2 \geq 0.990$, min of 5 points	
	Non-linear (quadratic)	$R^2 \geq 0.990$, min of 6 points	$R^2 \geq 0.990$, min of 6 points	
Second Source Verification Standard (ICV)	Immediately after each initial calibration	≤20% difference Bad Actors (See Table 5.1) ≤ 30% Diff.	≤20% difference Bad Actors (See Table 5.1) ≤ 30% Diff.	If analytes that are project primary compounds of interest meet ICV criteria, analysis may continue. Analytes that fail, but are less critical to project success, qualified in accordance with ENV-SOP-MAD1-0017, Qualification of Data" until a new calibration and ICV can be analyzed.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples, after 10 field samples and at end of sequence	≤20% difference Bad Actors (See Table 5.1) ≤ 30% Diff.	≤20% difference Bad Actors (See Table 5.1) ≤ 30% Diff.	If the criteria are not met a new calibration curve must be prepared or data qualified in accordance with ENV-SOP-MAD1-0017, Qualification of Data."
Internal Standard Response	All analyses	Response: 50 – 200% of mid-point of most recent calibration or most recent CCV on non-calibration days	Response: 50 – 200% of mid-point (or average) of most recent calibration or most recent CCV on non-calibration days	If internal standard responses fall outside of the acceptable limits, primary sources to investigate are the Merlin Microseal and accuracy of instrument tuning. Affected samples should be reanalyzed after correcting the issue. Occasionally this will require recalibration.
Internal Standard RT	All analyses	RT ± 10 sec of mid-point of most recent calibration	RT: NA	Retention time drift of more than 10 seconds is extremely uncommon with modern GC/MS systems. If this is observed, reanalyze the affected samples. Also ensure all connections and fittings within the GC are secure.

12. PROCEDURE

12.1. Typical Instrument Conditions

Component	Settings
Gas Chromatograph	Head Pressure: 10.0 psi Split Flow: splitless, split after 3 minutes at 30 mL/ min Purge Vent Flow: 3 mL/ min Injector Temperature: 240 °C Detector Temperature: 280 °C Oven Temperature Program Initial Temperature: 45°C Initial Hold: 4 min Ramp 1: 15°C / min to 200°C Ramp 2: 40°C / min to 240°C Final Hold: 1 minute at 240 °C
Mass Spectrometer	BFB target Tune EM Voltage 1500 - 2500, modifications (as timed events) to voltage are made to optimize sensitivity of mass spectrometer SIM windows established to optimize sensitivity of mass spectrometer Scan Range: 45-260 amu Scan time: approximate 2 scans/ second Note: Agilent 5975 is always operated in dual SIM/SCAN mode while the Agilent 5973 is operated in SIM mode with the exception of the BFB tune check which requires SCAN mode.
CTC Autosampler	Agitation Temperature: 32°C Agitation time: 8 minutes Desorb Time: 3 minutes

12.2. Sample Preparation

12.2.1. Water samples: An aliquot of sample, not to exceed 10 mL and sufficient to get the highest concentration constituents into the upper half of the linear range, is transferred via a 10 mL Leur lock syringe to a 20 mL extraction vial which contains 1 tsp. of sodium chloride (approx. 2 grams). The syringe is spiked with the working internal standard and water surrogate standard. Add 10 µL of the working internal standard as well as 10 µL of the water surrogate standard. The 10 mL volume allows for adequate headspace within the 20mL vial for the SPME process to occur. The extraction vial is then placed on the autosampler tray on the CTC autosampler and set for analysis in accordance with the CTC specifications. At that point, the CTC autosampler agitates the sample and the SPME fiber is then exposed to the headspace above the sample for 8 minutes while the sample is agitated. The exposure time is consistently measured by the autosampler. Extraction time may be modified depending on the anticipated equilibration response of the target analytes at given concentrations (if adjusted all samples and standards would be adjusted).

12.2.2. Soil samples: Preserved in methanol; approximately 10-12 g core of soil is added to 10 mL of methanol. Upon entering lab, surrogate is added to the mixture; **20 µL of the 200 mg/L** surrogate intermediate standard **is added to the soil/methanol mixture.** The methanol/soil mixture is then agitated to completely break up the solids, and then centrifuged if necessary. The level of agitation shall be

sufficient to completely disaggregate the solids and to maximize the contact between the methanol and the soil surfaces. This is typically achieved within 10 minutes using an aggressive orbital shaking procedure. If necessary, soil samples may be centrifuged to assist in the extraction process. Following this extraction step, an aliquot of the sample methanol (typically 0.5 mL), sufficient to get the highest concentration constituents into the upper half of the linear range but not to exceed 0.5 mL, is transferred to the SPME extraction vial via a Leur lock syringe. Prior to adding sample to syringe, the syringe is filled with enough DI water such that the end-volume is 10 mL. The syringe is also fortified with 10 µL of the working internal standard. This water/methanol mixture is further extracted via the headspace SPME technique in the same manner as a water sample.

12.2.3. Rock samples: Approximately 20 g core of crushed rock sample is preserved in 20 mL of methanol. Rock samples are typically extracted using a microwave assisted extraction (MAE) technique. Upon entering lab, and before the MAE step, surrogate is added to the mixture; **40 µL of the 200 mg/L surrogate intermediate standard is added to the rock/methanol mixture.** Following the MAE step, an aliquot of the sample methanol, sufficient to get the highest concentration constituents into the upper half of the linear range but not to exceed 0.5 mL, is transferred to a 20-mL SPME extraction vial, which contains 1 tsp salt (approx. 2 grams) , via a Leur lock syringe. Prior to adding sample to syringe, the syringe is filled with enough water such that the end-volume is 10 mL. The syringe is also fortified with 10 µL of the working internal standard. This water/methanol mixture is further extracted via the headspace SPME technique in the same manner as a water sample.

13. QUALITY CONTROL

13.1. Refer to Method 8000 for general quality control procedures for chromatography methods.

13.2. **Tune and BFB**: The instrument should complete a BFB target tune in Chemstation and must complete a BFB tune evaluation prior to calibration. Once calibrated, BFB must be evaluated at the beginning of a sequence and every 12 hours thereafter. 8260D only requires BFB verification prior to calibration. See Section 11 and Table 11.3.

13.3. **Initial Calibration (ICAL)**: The ICAL must consist of at least 5 points. The % RSD of response factors must meet the requirements in the referenced method, generally 20% or less. See Section 11 and Table 11.9 for calibration acceptance criteria.

13.4. **Analytical Batch**: An analytical batch consists of 20 or fewer samples. Batch quality control samples are run with every twenty samples, with a minimum of one per day: method blank (BLK), laboratory control sample (BS), matrix spike/ matrix spike duplicate (MS/MSD).

13.4.1. **Method Blank (BLK)**: A Method blank consists of an aliquot of laboratory reagent water or methanol that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. If target analytes or interferences are present at concentrations that impact the analytical results for samples, the samples (including quality control samples) should be reextracted or appropriately qualified in accordance with ENV-SOP-MAD1-0017, Qualification of Data. Prepare method blanks depending on the associated matrix as listed below.

13.4.2. Aqueous matrix: Add 10 µL of working internal standard and 10 µL of the water surrogate into 10 mL of water within the SPME extraction vial for subsequent analysis.

13.4.3. Soil matrix: Add 10 g sand to 10 mL methanol and spike with 20 µL of the 200 mg/L surrogate intermediate standard. Once extraction procedure is complete, a 0.5 mL aliquot of this mixture is diluted

into 10 mL of water and spiked with 10 µL of the working internal standard within the Leur lock syringe then transferred to the SPME extraction vial for subsequent analysis.

13.4.4. Rock matrix: Add 20 g sand to 20 mL methanol and spike with 40 µL of the 200 mg/L surrogate intermediate standard. Once extraction procedure is complete, a 0.5 mL aliquot of this mixture is diluted into 10 mL of water and spiked with 10 ul of the working internal standard within the SPME extraction vial for subsequent analysis.

13.4.5. If no method blank is run within a sequence, an instrument blank (-IBL) containing internal standards and surrogates must be run to demonstrate the cleanliness of the system.

13.4.6. **Laboratory control samples (BS)** are prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. Typical spike concentrations 20 µg/L are prepared as follows depending on the matrix:

13.4.7. Aqueous matrix: Add 20 µl of calibration intermediate standard into 10 ml of water within the SPME extraction vial for subsequent analysis.

13.4.8. Soil matrix: Add 10g sand to 10 ml methanol and spike with 20 µl of 200 µg/ml spike stock standard. Once extraction procedure is complete, a 0.5 ml aliquot of this mixture is diluted into 10 ml of water spiked with 10 µl of 20 µg/ml internal intermediate standard within the SPME extraction vial for subsequent analysis.

13.4.9. Rock matrix: Add 20g sand to 20 ml methanol and spike with 8 µl of 2000 µg/ml calibration stock standard. Once extraction procedure is complete, a 0.5 ml aliquot of this mixture is diluted into 10 ml of water spiked with 10 µl of 20 µg/ml internal intermediate standard within the SPME extraction vial for subsequent analysis.

13.4.10. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**: Matrix spikes are prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. Typical spike concentrations 20 µg/L are prepared as follows depending on the matrix:

13.4.11. Aqueous matrix: Add 20 µl of calibration intermediate standard into 10 ml of sample contained within the SPME extraction vial for subsequent analysis

13.4.12. Soil matrix: Add approximately 10g of soil sample to 10 ml methanol and spike with 20 µl of 200 µg/ml calibration stock standard. Once extraction procedure is complete, a 0.5 ml aliquot of this mixture is diluted into 10 ml of water spiked with 10 µl of 40 µg/ml internal intermediate standard within the SPME extraction vial for subsequent analysis.

13.4.13. Rock matrix: Add approximately 20g of rock sample to 20 ml methanol and spike with 40 µl of 200 µg/ml calibration stock standard. Once extraction procedure is complete, a 0.5 ml aliquot of this mixture is diluted into 10 ml of water spiked with 10 µl of 40 µg/ml internal intermediate standard within the SPME extraction vial for subsequent analysis.

Table 13.1 – Quality Control Acceptance and Verification Criteria

QC Type	Parameter / Frequency	8260C Criteria	8260D Criteria	Comments/Corrective Actions
Method Blank	One per preparation batch and at least one for every 20 samples.	Target Compounds < one-half LOQ.	Target Compounds < one-half LOQ	Investigate to determine source of contamination. Bake fiber to remove contamination or replace with new fiber. Re-prepare and reanalyze all associated samples. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all positive detections of the affected analyte(s) in samples analyzed in the affected batch.
Laboratory Control Samples (-BS)	One matrix-specific BS per analytical batch. BSD may be analyzed in lieu of a matrix spike duplicate (MSD) sample.	Initial QC limits are 70 to 130%, or 60 to 140% for poor performers for target compounds in water and soil analyses. Control limits will replace these limits once enough data are generated to calculate the control limits.	Control limits will replace these limits once enough data are generated to calculate the control limits	When a BS is prepared in the same manner as a CCV, the same standard can be used as both the BS and CCV. Reanalyze once; if still outside acceptance criteria, verify standard solution integrity, and instrument performance. Re-prepare and reanalyze all associated samples. If reanalysis cannot be performed, Data should be qualified in accordance with ENV-SOP-MAD1-0017, Qualification of data. Data should be qualified in accordance with ENV-SOP-MAD1-0017, Qualification of data..
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One set per 20 samples of a similar matrix or as specified by the Client.	Recovery Limits for the MS/MSD are 70-130% and 60-140% for poor performers. RPD not to exceed 30% for waters or 50% for soil samples. Control limits will replace these limits once enough data are generated to calculate the control limits.	Meets performance-based or project defined recovery criteria. Control limits will replace these limits once enough data are generated to calculate the control limits.	Reanalyze only if associated QC (i.e. LCS and ICV) results are also outside of tolerance. If BS or ICV are acceptable, narrate as possible matrix effect. Data should be qualified in accordance with ENV-SOP-MAD1-0017, Qualification of data.

14. DATA ANALYSIS AND CALCULATIONS

14.1. The PAS Quality Manual provides guidance on calculations used within the laboratory not covered in the previous sections.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. See Tables 11.9 and 13.1.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. See Tables 11.9 and 13.1.

16.2. Sections 13 (Corrective Action) and 14 (Preventative Action) of the Pace Analytical Services-Madison Quality Manual or its equivalent revision or replacement outlines the guidelines for out-of-control data.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. If not specifically listed in Tables 11.9 and 13.1, the contingencies are as follows: if there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager. Contingencies for out-of-control data should be evaluated on a case-by-case basis. Documentation of the out-of-control/non-conformance situation and resulting corrective action(s) taken should occur on the appropriate ENV-SOP-MAD1-0018 sequence checklist. The sequence checklist will be peer reviewed following the ENV-SOP-MAD1-0018, Data Review, SOP. Analytical results shall be qualified as necessary following the ENV-SOP-MAD1-0017, Qualification of Data, SOP.

18. METHOD PERFORMANCE

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Method Detection Limit (MDL) Study: An MDL study must be conducted (per the method) per ENV-SOP-MAD1-0021, Determination *of Detection and Quantitation Limits* (most current revision) for each matrix per instrument.

18.3. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per ENV-SOP-MAD1-0023, *Demonstration of Capability (DOC) Determinations* (most current version).

19. METHOD MODIFICATIONS

19.1. The analyst is permitted to modify GC columns, GC conditions, internal standards or surrogate standards, and GC/MS conditions. Each time such method modifications are made, the analyst must verify method criteria defined in Section 13 are met. Modifications to GC conditions should still produce conditions such that co-elution of the method analytes is minimized to allow for optimal GC/MS performance.

19.2. This method is developed to be compliant with the specific procedures and QA/QC metrics that are in EPA Method 8260 C and D.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1. General maintenance shall be conducted proactively to minimize the down time associated with equipment failure and to maintain the highest degree of data quality. This shall be accomplished through scheduled and symptomatic maintenance. Examples of symptomatic maintenance, in which the chromatography is monitored, include cleaning of inlet, replacement of septa and liner and mass spectrometer source cleaning. Examples of scheduled maintenance include replacement of diffusion and rough pump oils. All maintenance conducted on equipment shall be documented in the instrument maintenance logs. The primary pieces of instrumentation for the laboratory are listed below.

20.2. Routine instrument maintenance is critical to achieve optimum method sensitivity. All laboratory materials must be determined to be free of contamination to ensure potential background interferences are minimized.

20.2.1. Please refer to the instrument manual for maintenance procedures performed by the lab.

20.2.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3. GC maintenance – GC system components such as columns and injection ports must be maintained on a regular basis. The system is continuously checked as part of the ongoing analytical work and where problems are identified, actions to replace faulty instrumentation should be conducted.

20.4. Mass spectrometer maintenance – Please refer to the instrument manual for maintenance procedures performed by the lab. Common maintenance procedures are listed below.

20.4.1. Source cleaning – Clean the ion source parts according to the manufacturer's recommended practice as needed.

20.4.2. Rough pump oil – Check pump oil level and color periodically. Change pump oil every twelve months depending on usage by following the instruction manual. If the pump seems to need oil frequently, there may be a leak requiring further investigation.

20.4.3. When venting the MS system, follow the proper procedures as outlined in the instrument manual to prevent possible damage to the instrument as well as contamination of the vacuum chamber with oil.

20.5. SPME Autosampler CTC Combi PAL Maintenance – The autosampler requires periodic lubrication and bungy cord replacement. The lubrication of the x and y rails and the replacement of the bungy cords should occur every 6 months. If the unit is lightly used through a given period, this frequency can be reduced accordingly.

21. TROUBLESHOOTING

21.1. Any deviations from the norm encountered while conducting this analysis must be noted and brought to the attention of the group leader. This section contains information for troubleshooting basic system issues.

21.1.1. GC troubleshooting – Please refer to the instrument manual for troubleshooting procedures performed by the lab. Common GC issues are listed below.

21.1.2. Gas delivery system – checking for leaks with a helium leak detector is warranted if injection port pressure is not able to be achieved and/or significant water/air levels present, as indicated by the presence of ions 18 and 28 in the MS.

21.1.3. Peak shape issue –Split peaks, peak tailing, poor efficiency, and inconsistent response are usually associated with issues like injection port and column contamination or sample overload effects. Replacing, or removing a part of, the GC column and performing inlet maintenance maintenance usually helps to resolve this issue.

21.1.4. Background contamination – After multiple injections or long periods of operation, background interference may accumulate within the injection port. Performing inlet maintenance will help to remove the interferences.

22. SAFETY

22.1. Laboratory personnel should take extra care when working with standards. When working with standards, make certain that there is adequate ventilation and that safety glasses and Nitrile or Latex gloves are worn.

22.2. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed. Hazard assessments are listed in Attachment 3 of ENV-SOP-MAD1-001 *Contingency and Emergency Procedures Plan.*

22.3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal; however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate Pace employee.

22.4. All chemicals are required to be received with a Safety Data Sheets (SDS). Pace utilizes MSDSonline to provide SDS to employees in an online database that can be easily accessed at any time using Pace Connect. The corporate Safety Manual is available through Master Control®. Any questions or concerns should be taken to the laboratory Safety Officer.

22.5. Glassware that contains methanol is not allowed to be dried in an oven. Baking glassware to remove organic contaminants is only an acceptable practice when the glassware is dry and free of organic solvent.

23. WASTE MANAGEMENT

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual. Pollution Prevention

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

24. REFERENCES

24.1. ASTM6520, Solid Phase Micro-extraction of water and its headspace for analysis of Volatile and Semi-volatile Organic Compounds

24.2. EPA Method 8260C: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). August 2006.

24.3. EPA Method 8260D: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). June 2018.

24.4. Pace Quality Assurance Manual- most current version.

24.5. The NELAC Institute (TNI) Standard- 2009, or current revision.

24.6. Department of Defense (DoD) Quality Systems Manual- 2017, or current revision.

25. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

25.1. Attachment A: Absolute Retention Time Order and Characteristic Ions

26. REVISIONS

Document Number	Reason for Change	Revision
NA	Initial creation of PAS-Madison SOP from Cascade SOP CTS-10.15.16 EPA 8260C and inclusion of EPA 8260D criteria.	00

Attachment A - Absolute Retention Time Order and Characteristic Ions

Pace Analytical®
ECCS Mobile Lab Services

Document Name: Absolute Retention Time Order and Characteristic Ions

Compound	Absolute RT (min)	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Chloromethane	1.47	50	52
Vinyl chloride	1.55	62	64
Chloroethane	1.9	64	66
1,1,2-Trichloro-1,2,2-trifluoroethane	2.55	101	151
1,1-Dichloroethene	2.59	96	61
Acetone	2.68	58	
Carbon disulfide	2.77	76	78
Methylene chloride	3.03	84	86
Methyl-tert-Butyl ether	3.19	73	57
trans-1,2-Dichloroethene	3.21	96	61
1,1-Dichloroethane	3.58	63	65
cis-1,2-Dichloroethene	4.03	96	61
2-Butanone (MEK)	4.04	43	57
Chloroform	4.25	83	85
1,1,1-Trichloroethane	4.38	97	99
Pentafluorobenzene (IS)	4.4	168	
Carbon tetrachloride	4.48	117	119
Benzene	4.65	78	77
1,2-Dichloroethane	4.72	62	98
Fluorobenzene (IS)	4.94	96	77
Trichloroethene	5.12	95	97
1,2-Dichloropropane	5.33	63	112
Bromodichloromethane	5.5	83	85
1,4-Dioxane-d8		96	64
1,4-Dioxane		58	88
cis-1,3-Dichloropropene	5.83	75	77
Methyl isobutyl ketone (MIBK)	5.92	100	58,85
Toluene	6.05	92	91
trans-1,3-Dichloropropene	6.25	75	77
1,1,2-Trichloroethane	6.39	83	97
Tetrachloroethene	6.43	164	129
1,3-Dichloropropane	6.52	76	78
2-Hexanone	6.53	58	57
Chlorodibromomethane	6.67	127	129
1,2-Dibromoethane	6.78	107	109
Chlorobenzene-d5 (IS)	7.09	117	
Chlorobenzene	7.11	112	77
1,1,1,2-Tetrachloroethane	7.16	131	133
Ethylbenzene	7.15	91	106
m+p-Xylenes	7.24	106	91
o-Xylene	7.55	106	91
Styrene	7.56	104	78
Bromoform	7.74	173	175
Isopropylbenzene	7.8	105	120
p-Bromofluorobenzene (SURR)	7.96	95	174
1,1,2,2-Tetrachloroethane	8.07	83	131
1,3,5-Trimethylbenzene	8.24	105	120
1,2,4-Trimethylbenzene	8.53	105	120
1,3-Dichlorobenzene	8.65	146	111
1,4-Dichlorobenzene	8.79	146	111
1,2-Dichlorobenzene	8.79	146	111
1,2-Dibromo-3-chloropropane	9.77	75	155
1,2,4-Trichlorobenzene	10.38	180	182
Naphthalene	10.58	128	

APPENDIX D

COMMUNITY AIR MONITORING PLAN

APPENDIX D
Community Air Monitoring Plan (CAMP)
B335 Area RCRA Facility Investigation
Former IBM East Fishkill Facility
Hopewell Junction, New York

Introduction

This document summarizes the community air monitoring plan (CAMP) associated with the environmental assessment work to be completed for the Building 335 Area RCRA Facility Investigation (RFI) at the former IBM East Fishkill Facility. The air monitoring program associated with these activities will be performed by Sanborn, Head Engineering, P.C. (Sanborn Head).

The CAMP was developed in general accordance with the Generic Community Air Monitoring Plan (Generic CAMP) provided as Appendix 1A of DER-10 "Technical Guidance for Site Investigation and Remediation", dated May 2010, prepared by the New York State Department of Environmental Conservation (NYSDEC). In accordance with the Generic CAMP, the intent of this CAMP is to provide a measure of protection for the downwind community from potential airborne contaminant releases as a direct result of investigation and remedial work activities. Monitoring for worker exposure is covered under the project-specific Health and Safety Plan (HASP) included as a separate Appendix to the RFI Work Plan.

Volatile Organic Compounds (VOC) Monitoring, Response Levels, and Actions

Continuous monitoring for volatile organic compounds (VOCs) will be conducted during ground intrusive activities, including shallow soil vapor probe installation and sampling, soft-digging/vacuum excavation of borings, and drilling operations (including installation of CMT® multilevel systems). Continuous monitoring will be conducted at the downwind perimeter of the immediate work area (i.e., the exclusion zone). In addition, upwind concentrations will be measured at the start of each workday and periodically thereafter if wind direction changes significantly or if increased VOC concentrations increase significantly downwind.

Periodic monitoring will be completed during non-ground intrusive activities, including soil vapor and groundwater sampling from CMT® multilevel systems, and certain components of investigation derived waste (IDW) management (e.g., drilling water filtration). Periodic monitoring will generally consist of taking an instantaneous reading prior to beginning the work, at least once during the work when potentially-contaminated material is being sampled or handled, and after the work is complete. Periodic readings will either be collected at the location of the work being conducted to represent a worst-case scenario, or at the downwind perimeter of the immediate work area.

The monitoring work will be conducted using a photoionization detector (PID), which will be calibrated at the beginning of each day using an isobutylene standard. The PID will be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total VOCs at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities will be temporarily halted and monitoring continued. If total VOC concentrations readily decrease (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

2. If total VOC concentrations at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities will be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total VOC concentration 200 feet downwind of the exclusion zone, or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less, is below 5 ppm over background for the 15-minute average.

3. If total VOC concentrations are above 25 ppm at the perimeter of the work area, activities must be shutdown.

4. 15-minute readings will be recorded and available for NYSDEC and New York State Department of Health (NYSDOH) personnel to review upon request. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Continuous particulate monitoring with a dust meter is not proposed for this project because the proposed activities are unlikely to generate particulates for the reasons provided below:

- Shallow soil vapor probe installation: intermittent, low impact, manual activity;
- Soft digging/vacuum excavation: the vacuum activity itself is expected to capture particulates;
- Drilling operations: water or drilling fluid will saturate the soil and drill cuttings being brought to the ground surface; and
- Groundwater and soil vapor sampling: do not involve ground intrusive activities.

Visual dust monitoring and response actions will be completed in accordance with the project-specific health and safety plan.

APPENDIX E

PROJECT-SPECIFIC HEALTH AND SAFETY PLAN

SANBORN HEAD ENGINEERING

Building Trust. Engineering Success.

HEALTH AND SAFETY PLAN

B335 Area RCRA Facility Investigation
Former IBM East Fishkill Facility

Hopewell Junction, New York

File No. 2999.06
September 2019

EMERGENCY INFORMATION

Global Foundries Emergency Control (Medical/Fire/Safety and Health) ..... (845) 894-3333

Facility Ext: 4-3333

Global Foundries Security .....(845) 894-2000; Facility Ext: 4-2000

East Fishkill Fire District..... 911 or (845) 226-1652

East Fishkill Police Department ..... 911 or (845) 221-2111

St. Luke's Hospital..... (845)-568-2311

Poison Control Center.....1 800 222-1222

National Response Center.....1 800 424-8802

Site Superintendent/Site Safety Officer:
See Project Contacts Table

Safety Consultant: Rick Ecord .....(781) 278-3809

Directions from Global Foundries East Fishkill facility to St. Luke's Hospital:

1. Go east on NY-52 toward West Dr.
2. Turn right onto Lime Kiln Rd/CR-27.
3. Merge onto I-84 W towards Newburgh.
4. Take the Rt-32 exit, Exit 10S, toward US-9W S/Newburgh.
5. Take the ramp toward Newburgh/West Pt.
6. Turn slight right onto N Plank Rd/NY-32.
7. Turn right onto US-9W/NW-32/Albany Post Rd. Continue to follow US-9W/NY-32.
8. Turn left onto South St.
9. Turn Right onto Dubois St.
10. Destination is at 70 Dubois St., Newburgh, NY 12550.
    Total Time 22 minutes, total distance 15 miles.

MAPQUEST
Milton
Red Oaks Mill
Lagrangeville
Crown Heights
Ulster
Dutchess
Plattekill
Marlboro
Myers Corner
Wappingers Falls
Hillside Lake
New Hamburg
Hughsonville
Hopewell Junction
Ulster
Orange
Albany Post Rd
Chelsea
Castle Point
Brinckerhoff
Fishkill
Orange Lake
Gardnertown
Balmville
Newburgh
Stewart
Int'l
Airport
Dutchess
Putnam
New Windsor
North Highland
Vails Gate
Cornwall On Hudson
Firthcliffe
Salisbury Mills
Storm
King
State
Park
Nelsonville
Cold Spring
Mountainville
West Point
Garrison
Tompkins Corners
Kent Cliffs
Taconic State Pkwy
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PROJECT CONTACTS

Client/Site Contacts

Title	Name	Office Phone
IBM Project Coordinator	Dean Chartrand	703-257-2583
iPark Main Site Contact	Mike Buckley	203-430-9211
iPark Secondary Site Contact	Jared Schlosser	845-642-6928
Global Foundries Snr. Engineering Manager	Gary Marone	845-894-5700

Sanborn, Head & Associates, Inc.

Title	Name	Cell Phone	Office Phone
Principal-in-Charge	Dave Shea	603-219-8394	603-415-6130
Project Manager	Jennifer Sanborn	603-674-8752	603-415-6137
Project Manager	Joe Corsello	603-969-2324	857-327-9741
Site Superintendent/ Site Safety Officer (varies for each mobilization)	Erica Bosse	207-329-3878	978-577-1008
	Paula Pryor	845-699-5926	603-415-6129
	Jim Flood	203-673-4609	603-415-6143

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1.0 INTRODUCTION

This Site-Specific Health and Safety Plan (HASP) has been developed for use by employees who will be working on the B335 Area RCRA Facility Investigation (RFI) at the former IBM East Fishkill facility (hereinafter referred to as the site). For purposes of this plan, "employee(s)" means all Sanborn Head Engineering, P.C. and Sanborn, Head & Associates, Inc. (collectively, Sanborn Head) employees who will be working on this site. The work will be completed on property currently owned by iPark East Fishkill LLC (iPark). The HASP describes safety and health procedures and requirements for work that will be performed at this site. All corporate health and safety programs (i.e., Respiratory Protection, Hazard Communication, Personal Protective Equipment (PPE), Hearing Conservation, etc.) also apply to work performed on this Site.

All employees will comply with the requirements of this HASP and will sign the Acknowledgement on the last page of this HASP. This HASP will be provided to on-site subcontractors at their request for informational purposes only. Pursuant to Occupational Safety and Health Administration (OSHA) regulations and Sanborn Head's subcontract agreements, subcontractors are responsible for the health and safety of their employees. Similarly, the health and safety of employees of other entities (e.g., site owners) will be the responsibility of their respective employers, and this HASP will be provided to them at their request for informational purposes only.

Based on the results of site characterization and the history of the Site, Level D/D+ PPE is anticipated to be required (see Section 8.0, Table 4). However, the level of PPE required will depend on the field conditions observed and will be upgraded if appropriate at the discretion of the Site Safety Officer. If previously unidentified conditions which affect health or safety issues are discovered, work in the Exclusion Zone will cease and this HASP will be amended accordingly. Amendments are found in Appendix B of this HASP.

2.0 KEY PERSONNEL / CONTACTS

2.1 Contact Procedure – Non-Medical Emergency

For non-medical emergencies, the Site Superintendent should contact the Project Manager (PM), and if they are unavailable, the Site Superintendent should contact the Principal-in-Charge (PIC). Based on the issue(s) involved, the PM and/or PIC will determine how to proceed and if other project personnel should be contacted.

2.2 Contact Procedure – Medical Emergency

For a medical emergency, the Site Superintendent should call Global Foundries Emergency Control ((845) 894-3333) who will contact 911. If Global Foundries Emergency Control cannot immediately be reached, contact 911. The Site Superintendent should then contact the PM, and if they are unavailable, the Site Superintendent should contact the PIC.

For less severe medical situations, the Site Superintendent should make arrangements to get the injured/ill individual to the local hospital. Also contact Global Foundries Emergency Control ((845) 894-3333) for additional procedures that may be needed. Contact the PM,

and if they are unavailable, the Site Superintendent should contact the PIC. The PM and/or PIC will determine how to proceed, and if other project personnel should be contacted.

2.3 Organization, Responsibilities, and Authority

The following subsections describe the roles and responsibilities of the key personnel. Refer to the Project Contacts page for contact information for specific individuals.

2.3.1 Principal-in-Charge (PIC):

- Reviews and approves the HASP;
- Ultimate responsibility for project health and safety, and satisfactory completion of all tasks for this project; and
- Primary client contact.

2.3.2 Project Manager:

- Reports to PIC and day-to-day client contact;
- Oversees preparation and updates of HASP, and monitors that fieldwork proceeds according to HASP requirements; and
- Primary office contact of field staff.

2.3.3 Site Superintendent:

The Site Superintendent (or designee) is responsible for:

- Oversight of field aspects of the project; and
- Coordination of health and safety issues with the Site Safety Officer.

The Site Superintendent (or designee) will:

- Be present on-site during all work activities;
- Be available for regulatory personnel; and
- Be familiar with all aspects of this HASP.

2.3.4 Site Safety Officer:

The Site Safety Officer (or designee) is responsible for:

- Oversight of the health and safety of site employees;
- Implementing the provisions of this HASP;
- Performing site hazard analysis;
- Providing recommendations for PPE modifications; and
- Emergency response procedures in conjunction with the Site Superintendent.

The Site Safety Officer (or designee) will:

- Provide safety briefings to inform site employees of hazardous conditions; and
- In consultation with the Site Superintendent and the Project Manager, modify this HASP to provide adequate protection from physical, health or environmental hazards.

2.3.5 Subcontractor Responsibilities

All Sanborn Head subcontractors are responsible for their own health and safety and are therefore responsible for adhering to their own HASPs. All Sanborn Head subcontractors are also responsible for providing their own safety equipment and for ensuring a safe workplace for their employees. Information such as emergency routes and telephone numbers, should be provided to the Sanborn Head subcontractors.

3.0 SITE DESCRIPTION

The facility, formerly IBM's East Fishkill facility, is located on approximately 500 acres in the town of East Fishkill, Dutchess County, New York. The facility formerly developed and manufactured semiconductor logic and memory devices and modules for many of IBM's product lines. The facility is located in a semi-rural area with private residences adjacent to the north and eastern facility boundaries; John Jay High School is located in between the Main Site and West Complexes. The Wicoppee Creek and commercial property form the western perimeter, and I-84 is the southerly facility boundary.

Activities associated with this HASP are focused in and around B335, which is the former tetrachloroethylene (PCE) recycling facility for the facility. The volatile organic compounds (VOCs) found in soil, groundwater, soil vapor, and/or indoor air in this area of the site that are the subject of the RFI Work Plan include PCE and its breakdown products.

4.0 SCOPE OF WORK

The primary objective of the B335 RFI is to evaluate potential sources of PCE in the vicinity of B335. PCE and TCE were detected at concentrations of 560 µg/m^3 and 1.8 µg/m^3, respectively, in an indoor air grab sample collected from B335 in November 2018. PCE and TCE were detected at concentrations of up to 25,000,000 µg/m^3 and 70,000 µg/m^3, respectively, in subslab vapor beneath B335 in January 2019. Concentrations of PCE and TCE in the most recent overburden groundwater sampling round in 2018 were 22 µg/L and 10 µg/L, respectively, in nearby monitoring well 181.

The Scope of Work covered under this HASP includes:

- Conducting site walks;
- Installing shallow soil vapor sampling implants using a manual Geoprobe®;
- Observing drilling operations using vacuum excavation and wire-line coring techniques; and
- Collecting soil, groundwater, and soil vapor samples.

5.0 SITE CONTROL

Work zones may be established and marked prior to beginning work activities. The purpose of work zones is to:

- Reduce the migration of contaminants into "clean areas";
- Limit access of unauthorized personnel;
- Control chemical exposure; and
- Limit physical hazards.

The work zones if established will include, at a minimum the following:

- Exclusion Zone
- Contamination Reduction Zone
- Support Zone

5.1 Exclusion Zone

The Exclusion Zone is the area where work is being performed and where physical and chemical hazards may be present. At locations where the Site Superintendent/Site Safety Officer feels that risk to the public, co-workers, and the environment is relatively limited (e.g., inside vacant B335), cording off or physically marking the exclusion zone (designated with traffic cones and/or tape or other appropriate methods) will be discretionary. Only authorized employees who meet training and medical surveillance requirements may enter this zone.

5.2 Contamination Reduction Zone / Decontamination Zone

The Contamination Reduction Zone (CRZ) is the buffer zone between the Exclusion Zone and the Support Zone. If a CRZ is established, it will serve as the only means of entry or exit to the exclusion zone. Employees entering and leaving the exclusion zone must use this corridor. Only authorized employees who meet training and medical surveillance requirements may enter this zone.

The access corridor through the CRZ to the Exclusion Zone will be clearly delineated with traffic cones and/or tape and may serve as the Decontamination Zone, if one is required.

Only Sanborn Head and the construction subcontractor will have access to the CRZ.

5.3 Support Zone

This zone is the "clean area" and contains site offices, sanitation facilities, and clean equipment. Any function that need not or cannot be performed in hazardous or potentially hazardous area is performed here.

The following equipment will be available and will be located in the support zone:

- First aid kit (Located in personnel 'ready bags')

6.0 WORK HAZARDS – KNOWN AND ANTICIPATED

The following table summarizes the health risk analysis for substances noted in Section 3.0.

Table 1
Health Risk Analysis Data

Substance	OSHA PEL	ACGIH TLV	NIOSH IDLH	Action Level	Field Instrument To Be Used	Vapor Pressure	Principal Routes of Exposure	Target Organs and Effects	Known Carcinogen
PCE	100 ppm / C 200 ppm	25 ppm / STEL 100 ppm	150 ppm	12.5 ppm	PID	14 mm Hg	Inh, Ing, Abs, Cont	Liver, kidneys, skin, eyes, resp. sys, CNS	Yes
TCE	100 ppm / C 200 ppm	10 ppm / STEL 25 ppm	1,000 ppm	5 ppm	PID	58 mm Hg	Inh, Ing, Abs, Con	Resp. sys, heart, liver, eyes, CNS, skin	Yes
Dichloroethene (1,2-DCE) (cis- and trans- isomers)	200 ppm	200 ppm	1,000 ppm	100 ppm	PID	180-265 mm Hg	Inh, Ing, Con	CNS, eyes, resp. sys	No
Dichloroethene (1,1-DCE)	NE	5 ppm	NE	5 ppm	PID	500 mm Hg	Inh, Ing, Abs, Con	Eyes, skin, resp. sys, CNS, liver, kidneys	Yes
Vinyl Chloride (VC)	1 ppm / C 5 ppm	1 ppm	NE	5 ppm	PID	2,508 mm Hg	Inh, Con	Liver, resp. sys, CNS, blood, lymphatic sys.	Yes
Nuisance Dust	15 mg/m^3	NE	NE	2.5 mg/m^3	Visual and dust monitor	NA	Inh, Ing, Con	Eyes, skin, resp. sys	No

Notes:

OSHA = Occupational Safety and Health Administration
ACGIH = American Conference of Governmental Industrial Hygienists
NIOSH = National Institute for Occupational Safety and Health

CNS = Central Nervous System.

NE = Not Established.

PEL = Permissible Exposure Limit.

TLV = Threshold Limit Value.

IDLH = Immediately Dangerous to Life or Health

C = Ceiling Limit. An instantaneous/15 minute concentration which must not be exceeded without further engineering controls, work modifications, or PPE.

Carcinogen = An agent that is known or suspected to cause cancer.

Vapor Pressure indicates the volatility of a substance; for vapor pressure < 1 mmHg, the contaminant is not considered an inhalation hazard due to lack of volatility.

[Δ] Dust (i.e., particulates not otherwise regulated) refers to contaminants that are primarily an inhalation hazard, as dust particles. Dust can also be a contact hazard.

Early recognition of chemical exposure symptoms is essential in the prevention of serious chemical exposure incidents. Symptoms of exposure to the type of compounds potentially present at the Site include the following: fatigue, weakness; eye, nose, throat irritation; headache; dizziness; nausea; staggered gait; and dermatitis.

Each task to be performed as part of the scope of work for this site has been identified in Section 4.0. Table 2 provides a summary of tasks associated with the Work Plan for this project and the hazards and protective measures anticipated for each. Precautions will be taken to abate physical hazards as well as chemical hazards.

TABLE 2
HAZARD ANALYSIS FOR WORK ACTIVITIES

Task	Hazard (Chemical or Physical)	Hazard Control or Abatement
Site Walk	Slip/Trip/Fall	Maintain good housekeeping. Watch for slippery and uneven surfaces.
Installing shallow soil vapor sampling implants using a manual Geoprobe®	Particulate Matter/Dust Chlorinated Ethenes in Air (PCE Primarily) Noise Slip/Trip/Fall Use of Geoprobe® slide hammer Subsurface utilities	Ambient air monitoring as described in Section 7.0 and Table 3. PPE as described in Section 8.0 and Table 4. Watch for slippery and uneven surfaces. Wear heavy work gloves when using the slide hammer. Follow general construction safety practices. Maintain good housekeeping. Utility locating and mark-out.
Observing drilling operations using vacuum excavation and wire-line coring techniques, collecting soil samples	Particulate Matter/Dust Chlorinated Ethenes in Soil, Groundwater, and Air (PCE Primarily) Noise Slip/Trip/Fall Drilling hazards Subsurface utilities	Ambient air monitoring as described in Section 7.0 and Table 3. PPE as described in Section 8.0 and Table 4. Watch for slippery and uneven surfaces. Follow general construction safety practices. Maintain good housekeeping. Maintain a safe distance from the drill rig while in operation. Engineering controls, including use of exhaust hoses and fans, for interior drilling (and exterior drilling near HVAC intakes). Utility locating and mark-out.

Task	Hazard (Chemical or Physical)	Hazard Control or Abatement
Collecting groundwater and soil vapor samples	Chlorinated Ethenes in Groundwater and Air (PCE Primarily) Slip/Trip/Fall	Ambient air monitoring as described in Section 7.0 and Table 3. PPE as described in Section 8.0 and Table 4. Watch for slippery and uneven surfaces. Maintain good housekeeping.

7.0 AIR MONITORING

The purpose of air monitoring is to identify and quantify airborne contaminants in order to determine and verify the level of PPE required. Direct reading instruments will be used for initial and periodic air monitoring as required by this HASP. All instruments will be calibrated, operated and maintained according to manufacturer's instructions. Table 3 presents the air monitoring procedures for this Site.

An Air Monitoring Log is attached in Appendix C.

A Calibration Log is attached in Appendix D.

TABLE 3
AIR MONITORING PROCEDURES

Type of Monitoring (i.e., *Dust, Ambient Air*)	Instrument	Monitoring Frequency / Protocols	Action Level	Remedial Action
Dust	Visual	Continuous / as needed	Visible Dust	Moisten work area
Ambient Air	PID	Continuous during drilling operations Periodically during groundwater and soil vapor sampling	>5 ppm	Discontinue work, leave work area, and take appropriate measures to evaluate and mitigate the exceedance. Contact Site Safety Officer to upgrade to Level C if sustained PID readings >5 ppm.
Ambient Air	PID	Continuous during drilling operations Periodically during groundwater and soil vapor sampling	>100 ppm	Stop work immediately and contact site safety officer for further instruction.
Ambient Air (Inside Building)	O2/LEL/CO Meter	Continuous during interior drilling operations	>10% LEL Or <20% O2 Or >22% O2 Or >20 ppm CO	Stop work immediately and contact site safety officer for further instruction.

8.0 PERSONAL PROTECTIVE EQUIPMENT (PPE)

Based on analytical data available, it is anticipated that field activities will generally be performed in Level D or Level D+. However, the level of PPE required will depend on the field conditions observed and will be upgraded if appropriate at the discretion of the Site Safety Officer. Use Tables 1 and 3 to obtain Action Levels for each contaminant and Table 4 to determine appropriate PPE levels. Hard hats, boots, and safety glasses shall meet ANSI standards. Respirators shall meet MSHA standards.

TABLE 4
PPE LEVELS AND CORRESPONDING AIR LEVELS

PPE Level		Application
Level D/D+	■ Long pants ■ Long/Short sleeve shirts ■ Safety glasses with side shields, as required ■ Hard hat, as required ■ Steel-toed and shank work boots, as required ■ Hearing protection, as required ■ Chemical-resistant gloves, as required ■ Work gloves, as required ■ Outer boot covers, as required ■ Tyvek coveralls, as required	■ PID level ≤ 5 ppm ■ Dust < 2.5 mg/m^3 ■ Levels should be monitored above background readings.
PPE Level		**Application**
Level C	■ Respirator: TBD based on application ■ Long pants ■ Long/Short sleeve shirts ■ Safety glasses with side shields, as required ■ Hard hat, as required ■ Steel-toed and shank work boots ■ Hearing protection, as required ■ Chemical-resistant gloves, as required ■ Work gloves, as required ■ Outer boot covers, as required ■ Tyvek coveralls, as required	■ PID level greater than or equal to 5 ppm but less than 100 ppm ■ Dust > 2.5 mg/m^3 ■ Levels should be monitored above background readings. ■ Uncontrolled hazardous materials abatement in progress.

Notes:

1. This Table pertains to employees in the CRZ and Exclusion Zone.

2. Cartridge respirators can only be used when the contaminant has adequate warning properties to indicate breakthrough, oxygen levels are between 19.5% and 23.5%, the chemical is identified and a cartridge is available for that substance. If any of the listed conditions are not present, an air purifying respirator is not adequate protection.

3. This HASP will be revised if an upgrade to Level C is required.

In the unlikely event that employees encounter conditions which are immediately dangerous to life and health (IDLH) **all work in the Exclusion Zone will cease and this HASP will be revised.**

9.0 DECONTAMINATION PLAN

9.1 Decontaminating PPE

Decontamination of PPE is not expected to be needed, as disposable PPE will be used. Based on current data, the decontamination procedures anticipated for use on this site are doffing gloves using an outside-in approach and placing into sealed plastic bags for disposal as solid waste. If used, tyvek and rubber boot covers should be doffed while wearing rubber gloves and placed into sealed plastic bags for disposal as solid waste.

9.2 Decontaminating Equipment

Air Monitoring Equipment: If contamination is likely, wrap monitoring equipment in Ziploc bags to avoid the need for decontamination. If the equipment has to be decontaminated, follow the manufacturer's recommendations.

Non-Disposal Drilling Tools: Non-disposable down-hole drilling tools will be decontaminated between borings by the subcontractors using a pressure water on a decontamination pad.

Non-Disposable Sampling Equipment: Non-Disposal sampling equipment will be decontaminated between sample intervals using liquinox detergent and a potable water rinse.

Decontamination water shall be containerized in a frac tank or 55-gallon drums. All drums shall be properly labeled, transported, and treated on-site or disposed of off-site.

10.0 SITE RULES

In addition to all applicable Corporate Health and Safety Programs, the following general safety rules will be followed by all project employees:

- While in the exclusion and contamination reduction zones, do not eat, drink, chew gum, or tobacco, smoke or engage in any practice which increases the probability of hand-to-mouth transfer and ingestion of contaminants.
- Because medication can increase the effects of toxic chemicals in exposure situations, employees who must be on medication may discuss this issue with their supervisor prior to beginning work on site.
- When working in a "buddy system required" area/task, employees are paired with other employees or non-Sanborn Head personnel, and must always be in close proximity to each other.

- A tailgate health and safety meeting will be held before commencing a new phase of work; when there are changes to project environmental, chemical, and health hazards; the monitoring equipment requirements change; when PPE requirements change; or when there is a change in field personnel. In the meeting the Site Safety Officer shall:
  - Establish updates to evacuation routes.
  - Establish emergency meeting points.
  - Review any newly identified Hazards, PPE, or precautions to follow.

11.0 REPORTING INJURIES AND ILLNESSES

All employees on the site must immediately report injuries, and illnesses to their supervisor. If the injury or illness is a result of, or could result in, a chemical exposure, the supervisor will report it to the Site Superintendent/Site Safety Officer. He or she will then take appropriate action to prevent further exposure. The exposure will be reported in the Sanborn Head Incident/Injury Reporting Form.

Following an incident, a report will be completed by the supervisor and a copy given to the Site Superintendent/Site Safety Officer. All incident and illness reporting will follow corporate policies and procedures. Blank reports are available on Sanborn Head's HR Manager.

In the event of a hazardous material spill or chemical release above the reportable quantity, the Site Superintendent or designee will notify the appropriate Federal and State agencies.

ACKNOWLEDGEMENT OF SITE-SPECIFIC HEALTH AND SAFETY PLAN

Individuals signing below agree and acknowledge as follows:

I have read and understand the Site-Specific Health and Safety Plan for the B335 Area of the East Fishkill Facility. I agree to abide by these safety rules and understand that any violation may result in my removal from the Site.

Name (Please Print)	Signature	Date

APPENDIX A

SITE SIGN-IN LOG

APPENDIX A
SITE SIGN-IN LOG
B335 AREA RCRA FACILITY INVESTIGATION HASP
FORMER IBM EAST FISHKILL FACILITY

Name	Affiliation	Date & Time

APPENDIX B

AMENDMENTS TO THE SITE-SPECIFIC HEALTH AND SAFETY PLAN

APPENDIX B
AMENDMENTS TO THE SITE-SPECIFIC HEALTH AND SAFETY PLAN
B335 AREA RCRA FACILITY INVESTIGATION HASP
FORMER IBM EAST FISHKILL FACILITY

Date	Section	Page	By Whom	Received By

APPENDIX C

AIR MONITORING LOG

APPENDIX C
AIR MONITORING LOG
B335 AREA RCRA FACILITY INVESTIGATION HASP

Former IBM East Fishkill Facility

Date: _____ Project No: _____

Instrument: _____

Contaminants of Concern	PEL / Action Level	Time	Monitoring Results

APPENDIX D

AIR MONITORING EQUIPMENT CALIBRATION AND MAINTENANCE RECORDS

(Typically kept with equipment)