



State of New Mexico

ENVIRONMENT DEPARTMENT

TA-00  
JUDITH M. ESPINOSA  
SECRETARY

RON CURRY  
DEPUTY SECRETARY

BRUCE KING  
GOVERNOR

UNDERGROUND STORAGE TANK BUREAU

FAX #: 827-0310

TELECOPIER/FACSIMILE COVER SHEET

TO: BRUCE SWANTON

OFFICE: HRM B

PHONE: 827-4308 FAX #: 827-4361

ACTION REQUIRED: HIGH PRIORITY  LOW PRIORITY

NUMBER OF PAGES INCLUDING THIS COVER SHEET

3

FROM: JERRY SCHOEPPNER

OFFICE: DMED/USTB

PHONE: 827-0214

DATE SENT: 11/28/94

COMMENTS:

Bruce:  
I haven't received a response from James as to who should take the lead (out for a couple of days). Until it is resolved I put together a draft letter outlining comments we discussed. Please feel free to make any corrections / comments.

*Jerry*

PAGE 1 OF 3



DRAFT

November 28, 1994

Mr. Theodore J. Taylor, Program Manager  
 Environmental Restoration Program  
 Department of Energy  
 Los Alamos Area Office  
 Los Alamos, New Mexico 87544

**RE: Work Plan for Operable Unit 1071, Solid Waste  
 Management Unit 0-039, Former Underground Solvent  
 Storage Tanks, Community Center, Los Alamos, NM**

Dear Taylor:

The New Mexico Environment Department (Department),  
 Underground Storage Tank Bureau (Bureau) has received and  
 reviewed the above-referenced document dated October 20, 1994.  
 The Bureau approves the work plan with the following  
 revisions/comments:

1. Samples shall be collected every 5 feet rather than alternating 5 foot intervals.
2. Samples collected may not be composites. Rather discrete samples shall be screened/analyzed.
3. The Stoddard contaminant plume shall be delineated to non-detect (ND) rather than 100 parts per million.
4. Additional borings/sampling shall be completed near the rear of the present dry cleaning facility. This is where the fill pipes to the former underground storage tanks (UST's) are located.
5. Welded Bandalier Tuff was encountered at a depth of approximately 19 feet below grade during previous investigations. The work plan calls for each boring to go to a total depth of 100 feet below grade. An alternative plan needs to be addressed in case auger refusal is encountered.

(APPENDIX 5.19-B)

6. It is not clear that the proposed method of vapor sampling ~~in the most cost effective~~. Split-spoon samples screened with a photo-ionization detector (PID) and confirmation samples collected and submitted to a laboratory for analysis is adequate. If the proposed soil vapor sampling method is used background readings for the soil vapor work have to be defined.

used to define <sup>✓</sup> or <sup>14</sup> extent of contaminant

7. PID readings <sup>used to define</sup> must be confirmed with laboratory analysis along with verification that an 11.8 electron volt lamp is

will provide useful data.

DRAFT

Mr. Taylor  
November 28, 1994  
page 2

used in the field.

8. Shallow soil screening under the pipelines using a PID shall be continuous rather than every 20 feet. Confirmation laboratory analysis is also required. <sup>should</sup>

These revisions/comments along with any comments from the EPA, Region 6 must be incorporated into the final work plan for the site.

Please contact me at (505)827-0214 if you have any questions.

Sincerely,

Jerry Schoeppner  
Geologist  
Underground Storage Tank Bureau

cc: Joseph Vozella, DOE  
Bonnie Koch, Sciencetech, LAAO  
NMED District II Office  
NMED Espanola Field Office  
Anna Richards, Manager, Remedial Action Program

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## 5.19 SWMU 0-039 Former Underground Solvent Storage Tanks

SWMU 0-039 consists of the site of underground storage tanks, formerly used to store dry cleaning solvent, and the connecting pipes. Soil at the SWMU is contaminated and potentially contaminated. The SWMU is located on private property in the Community Center, Los Alamos, NM (Figure 5-101).

### 5.19.1 Description and History

As part of environmental assessments conducted by the previous property owner prior to sale of the property, five underground storage tanks (USTs) were removed from the Community Center in Los Alamos, New Mexico, in May and June 1993. The site location and site plan are shown in Figure 5-101. Three of the USTs contained petroleum hydrocarbons (i.e., gasoline, diesel fuel, or used oil), and were approved for closure by the Underground Storage Tank Bureau of the New Mexico Environment Department (NMED). The other two tanks contained dry cleaning solvents, and solvents were detected in the subsurface near the former tanks. Soil/tuff containing solvents near the former dry cleaning solvent USTs was excavated; however, the extent was greater than expected and the excavation was backfilled, leaving soil/tuff containing solvents in the vicinity of the former tanks. To date, the former dry cleaning solvent USTs have not been approved for closure by the NMED.

The former dry cleaning solvent USTs were installed and used during the Los Alamos National Laboratory's (the Laboratory's) ownership of the Community Center. As part of their permit to operate under the Resource Conservation and Recovery Act (RCRA), the Laboratory must take corrective action to mitigate releases from current or past operations. Therefore, on 5 July 1994, the Department of Energy (DOE) requested that the U.S. Environmental Protection Agency (EPA) add the former dry cleaning solvent USTs, designated Solid Waste Management Unit (SWMU) 0-039, to the Laboratory's operating permit (DOE, 1994). This request was granted by the EPA on 21 July 1994 (EPA, 1994). This work plan for additional investigation of this SWMU is being submitted as an addendum to the investigation activities for Operable Unit (OU) 1071, and is intended to comply with both the NMED's UST regulations and the RCRA corrective action process.

This sampling plan describes the methods to be used to: 1) determine the nature and extent of solvents in the subsurface in the vicinity of the former dry cleaning solvent USTs and any appurtenant structures, 2) perform a screening assessment as described in Appendix J of the Laboratory's Installation Work Plan (IWP; LANL, 1993), and 3) if necessary based on the results of the screening assessment, perform a preliminary assessment of the potential health effects posed by the site in the absence of further remediation.

### 5.19.2 Nature and Extent of Contamination

Our current understanding of the nature and extent of chemicals in subsurface soil/tuff in the vicinity of the former dry cleaning solvent USTs is based on analytical data collected during the initial tank excavation conducted by Glorieta Geoscience, Inc. (GGI, 1993), correspondence from the NMED UST Bureau, and information provided by the current owners of the Community Center. Based on this information, we have identified potential migration pathways through which chemicals released from the USTs may reach a receptor and the potential health and environmental impacts that may occur. Work conducted previously at the site, potential migration pathways, and potential health and environmental impacts are described in the following sections.

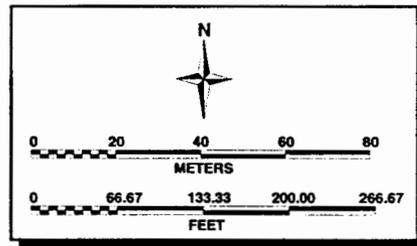
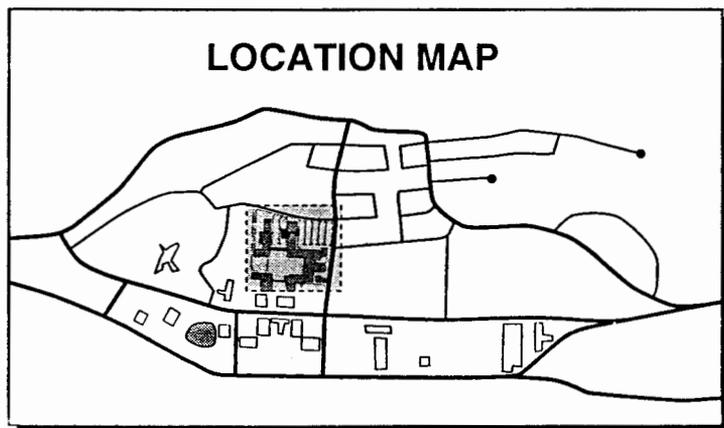
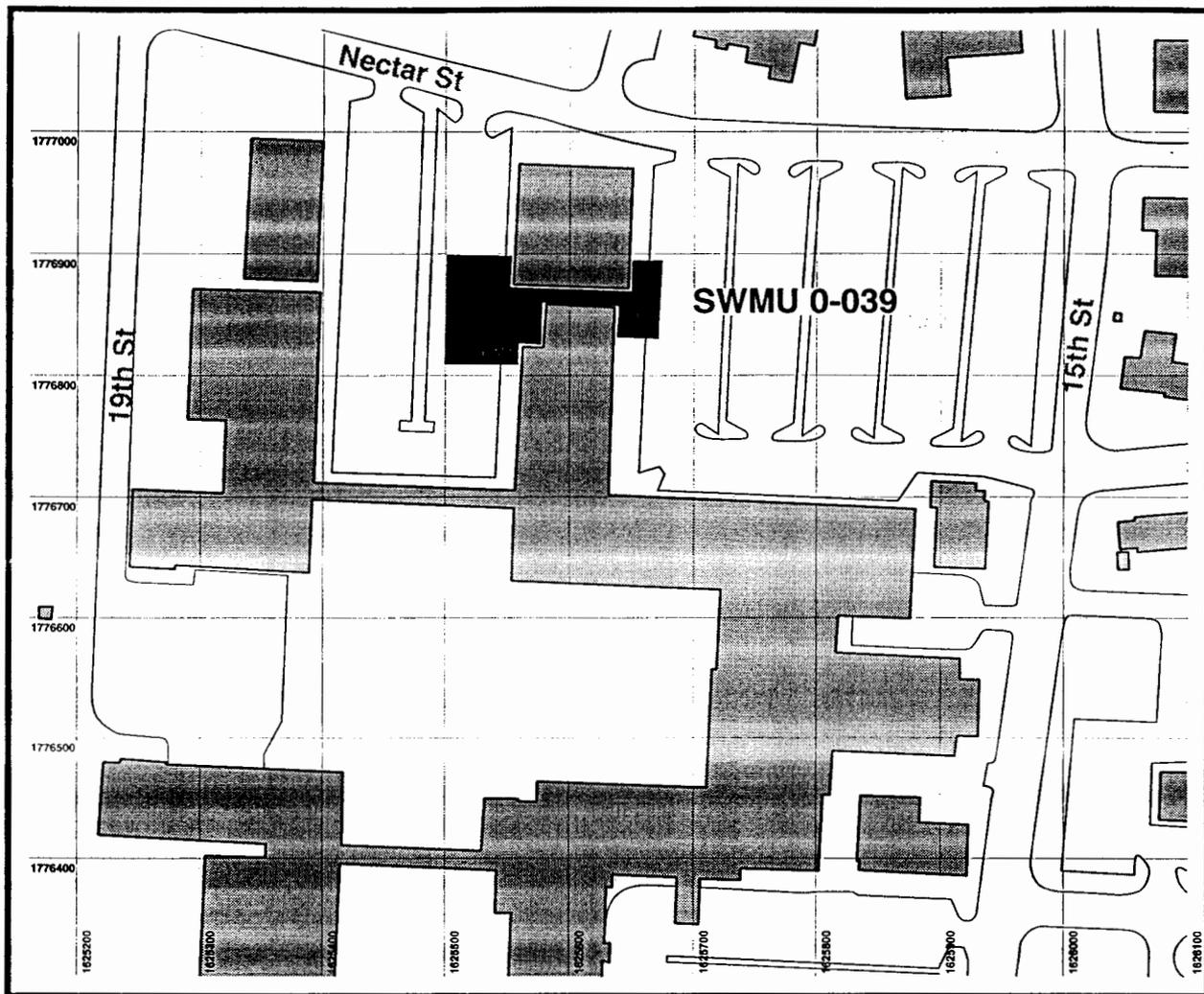


Figure 5-101. Site location of former underground dry cleaning solvent tanks and pipes (SWMU 0-039).

### 5.19.2.1 Summary of Previous Work

On 2 June 1993, two vertical 700-gallon USTs, located about four feet apart, were removed from an area northwest of the dry cleaning establishment shown on Figure 5-101 (GGI, 1993). These tanks reportedly had been used to store solvents associated with dry cleaning, although the tanks have not been used since sometime before 1969. Prior to removal, absorbent material was placed in each tank to absorb residual liquid. Organic vapor readings and bulk soil samples were collected from underneath each tank, the four walls of the excavation, and an area of "dark" soil between the two tanks on the bottom of the excavation. All of these samples were collected between 10 and 11 feet below ground surface. The soil samples and a sample of the absorbent material were submitted for laboratory analysis of polynuclear aromatic hydrocarbons (PNAs; EPA Method 8310) and halogenated hydrocarbons (EPA Method 8010). The analytical results from this investigation are summarized in Table 5.19.1. Tetrachloroethylene was detected in four of the seven soil samples at concentrations ranging from 0.011 to 0.78 milligrams per kilogram (mg/kg). Three PNAs, naphthalene, 1-methylnaphthalene, and 2-methylnaphthalene, were detected in a single soil sample at concentrations ranging from 0.05 to 0.21 mg/kg. Tetrachloroethylene and naphthalene were also detected in the absorbent material at concentrations of 6.6 and 0.3 mg/kg, respectively (GGI, 1993).

Excavation of soil from around the former dry cleaning UST excavation continued on 17 July 1993 (NMED, 1994). Higher concentrations of unspecified chemicals were reportedly discovered as the excavation progressed, and further excavation was abandoned so that additional investigation of the solvent release could be conducted (NMED, 1994; DOE, 1994). The current property owner stated that one of the chemicals detected was Stoddard solvent (Tom Netuschil, property owner, pers. commun., August 1994), which is a mixture of petroleum hydrocarbons that was used in the past as a dry cleaning solvent. No other information regarding the second phase of excavation was available for review; therefore, these observations could not be verified. The excavation was backfilled in September 1993, and no additional work has occurred at the site (Mary Anne Netuschil, property owner, pers. commun., July 1994).

In addition to the former USTs, several underground pipelines reportedly extend in an easterly direction away from the former USTs (Mary Anne Netuschil, property owner, pers. commun., July 1994). As part of the initial excavation activities, these underground pipelines were filled with cement and abandoned in place. To our knowledge, no investigation of possible chemical releases from these pipelines has been conducted to date.

### 5.19.2.2 Potential Migration Pathways

Based on previous sampling results and other available information, chemicals associated with dry cleaning processes are present in subsurface soil/tuff. These chemicals may leach/disperse through the vadose zone, migrate upward by vapor phase diffusion, and enter the atmosphere (Figure 5-102).

### 5.19.2.3 Potential Health and Environmental Impacts

Chemicals detected at the site may migrate further through the soil/tuff and upward into ambient air. As a result, human exposure to site chemicals could occur through inhalation of vapors, incidental ingestion of soil/tuff, or dermal contact with soil/tuff.

*will migrate away  
not just up*

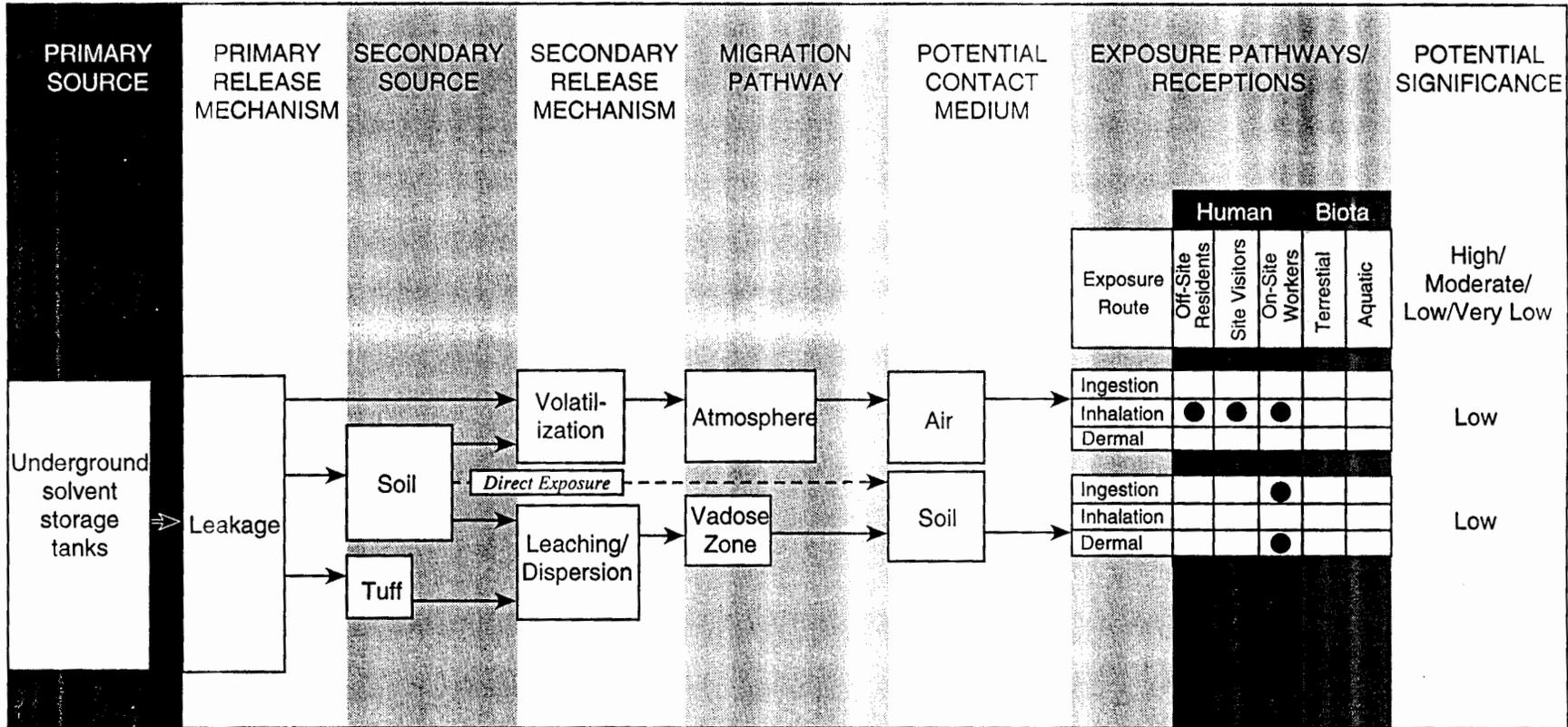


FIGURE 5-102. Conceptual model of SWMU 0-039.

Table 5.19.1

ANALYTICAL RESULTS OF SOIL SAMPLES AT DRY CLEANER SITE<sup>1, 2</sup>

Sample Description	PID (HNU Meter) (parts per million)	EPA Method 8310	EPA Method 8010
		Polynuclear Aromatics (milligrams per kilogram)	Purgeable Halocarbons/Aromatics (milligrams per kilogram)
Sample 1: Dark soil	270.0	naphthalene:0.21 1-methylnaphthalene:0.05 2-methylnaphthalene:0.6	tetrachloroethylene:0.66
North tank bottom	0.6	BDL	BDL
South tank bottom	36.0	BDL	tetrachloroethylene:0.78
South wall 10' depth	140.0	BDL	BDL
North wall 10' depth	4.0	BDL	tetrachloroethylene:0.01 <sup>1</sup>
West wall 10' depth	130.0	BDL	tetrachloroethylene:0.17
East wall 10' depth	2.6	BDL	BDL
Absorbent material	NA	naphthalene:0.30	tetrachloroethylene:6.6

## Notes:

NA = Not analyzed.

BDL = Below detection limit.

<sup>1</sup> Reproduced from GGI, 1993.<sup>2</sup> Soil Screening Action Levels (SALs) from Appendix J of the Installation Work Plan:

1-methylnaphthalene:	not available
2-methylnaphthalene:	not available
naphthalene:	3,200 mg/kg
tetrachloroethylene:	5.9 mg/kg

It should be noted, however, that incidental ingestion or dermal contact would require direct contact with soil/tuff, which could occur only through significant disturbance of the soil/tuff.

## 5.19.3 Decisions, Domain, and Approach

The domain for this SWMU is an area around the former UST excavation that extends eastward to encompass the underground pipelines (Figure 5-101). Previous investigations indicate that dry cleaning solvents were released from the former USTs to the environment; therefore, this initial site investigation is designed to collect the information necessary to:

- determine the nature and extent of solvents pursuant to Sections 1205C and 1305C of the New Mexico UST Regulations (NMED, 1988) and proposed Subpart S to RCRA (EPA, 1990),
- perform a screening assessment as described in Appendix J of the IWP, and
- if necessary based on the findings of the screening evaluation, conduct a preliminary risk assessment of the potential health effects posed by the site in the absence of further remediation.

*Sample handling problem - how will be prevented?*

In addition, the underground pipelines will be removed, and the underlying soil will be investigated to determine if a release has occurred. The site is located on private property; therefore, other remedial action will be proposed, if necessary, such that the property is suitable for unrestricted use and residual chemicals in the subsurface do not present an unacceptable risk to public health or the environment.

*non-hazy VOC's*

**5.19.3.1 Investigation Approach**

To assess the nature and extent of dry cleaning solvents in subsurface soil/tuff, multiple core samples will be collected from alternating 5-foot intervals (e.g., 10-15 ft, 20-25 ft, etc.) from several boreholes in the vicinity of the former dry cleaning solvent USTs. Each core sample will be composited and submitted for field screening-level and fixed laboratory chemical analysis as described in Section 5.19.5.4. The analytical results from the composite core samples will represent the average concentration of chemicals within the borehole interval, and will be appropriate for delineating the extent of solvents in the subsurface.

For petroleum hydrocarbons, the limits of this investigation will be based on guidance provided by the New Mexico UST regulations. Part XII of these regulations addresses corrective actions for petroleum UST systems (NMED, 1988). Section 1205C requires that the horizontal and vertical extent of petroleum hydrocarbons be determined, and defines it as a concentration of total petroleum hydrocarbons of less than 100 mg/kg in soil. For this investigation, 100 mg/kg total petroleum hydrocarbons will be used to define the extent of Stoddard solvent in soil, if present.

*are there any?  
OK if confirmatory  
samples taken*

Part XIII of the New Mexico UST regulations addresses corrective action for hazardous substance (i.e., non-petroleum) UST systems (NMED, 1988). Section 1305C specifies that the horizontal and vertical extent of hazardous substances in soil must be determined; however, it does not provide guidance or criteria for defining horizontal and vertical extent. For the other volatile organic compounds (VOCs), the limits of the investigation will be determined in the field based on factors such as the results of field screening-level chemical analyses, the locations of the samples, etc.

*background*

Chemical transport through the tuff is likely to be affected by heterogeneities within the tuff, such as variations in lithologic and structural characteristics, including degree of welding and fracturing. To provide additional information regarding the distribution of solvents at the site, soil vapor samples, which may be more representative of a larger volume of the heterogeneous subsurface, also will be collected at each composite core sample interval.

*no-need  
quartz, hi.  
QA/QC data*

To facilitate comparisons between the two data sets, the soil vapor concentrations will be converted to equivalent concentrations in tuff according to the following equation (EPA, 1992):

$$\text{Soil} = \frac{\text{Soil Vapor} \times k_{OC} \times f_{OC}}{H}$$

Where:

- Soil = Chemical concentration in tuff
- Soil Vapor = Chemical concentration in soil vapor
- $k_{OC}$  = Chemical-specific organic carbon partition coefficient
- $f_{OC}$  = Fraction of organic carbon in tuff
- H = Chemical-specific, dimensionless Henry's Law Constant

*no  
need direct  
measurement*

Chemical-specific values for  $k_{OC}$  and H that are recommended by the U.S. EPA be used for this conversion (EPA, 1990). As described in Section 5.19.5.4,

fraction of organic carbon ( $f_{OC}$ ) in core samples collected at the site will be measured by an analytical laboratory as part of this site investigation.

The equivalent concentrations in tuff will be used in addition to the composite core sample results in the screening and preliminary risk assessments described below. The analytical results from these samples also will be used to evaluate the correlation between the core sample and soil vapor data.

### 5.19.3.2 Screening Assessment

The Screening Assessment Methodology described in Appendix J of the IWP consists of comparing a measured chemical concentration, generally the maximum, in an environmental medium (e.g., air, water, or soil) to a Screening Action Level (SAL) for the same medium. A SAL is a medium-specific concentration that is calculated using chemical-specific toxicity information and conservative default exposure parameters based on a residential exposure scenario. The methodology used to calculate SALs is described in Appendix J of the IWP; the SALs for chemicals previously detected at the site are presented in Table 5.19.2. If the measured concentration in a medium is less than the relevant SAL, then, in conjunction with other information collected during the site investigation, no further action may be proposed. If the measured concentration is greater than the SAL, then additional evaluation (e.g., baseline risk assessment) is generally required. Exceptions to these guidelines that take into account multiple chemical constituents and natural background concentrations of some chemicals are described in Appendix J of the IWP.

*may be proposed,  
but may not be  
accepted*

### 5.19.3.3 Preliminary Health Risk Assessment

In the event that the results of the screening assessment suggest that chemicals in the subsurface at the site may pose an unacceptable potential risk to human health, a preliminary health risk assessment may be conducted to further evaluate whether additional action is required or if no further action at the site can be supported. The scope of this preliminary assessment will be defined, if necessary, following completion of the initial site investigation and screening assessment. The preliminary

**TABLE 5.19.2**  
**SCREENING ACTION LEVELS**  
**FOR CHEMICALS PREVIOUSLY DETECTED**  
**IN THE VICINITY OF THE FORMER DRY CLEANING SOLVENT TANKS<sup>1</sup>**

Chemical	Soil (mg/kg)	Water ( $\mu\text{g/l}$ )	Air ( $\mu\text{g/m}^3$ )
1-methylnaphthalene <sup>2</sup>	NA	NA	NA
2-methylnaphthalene <sup>2</sup>	NA	NA	NA
Naphthalene	3,200	1,400	NC
Tetrachloroethylene	5.9	5	1.8

NA = Not available.

NC = Not calculated.

<sup>1</sup> The methodology used to calculate screening action levels is described in Appendix J of the Installation Work Plan.

<sup>2</sup> Toxicity criteria are not available; screening action levels not calculated.

health risk assessment will be based on data collected according to this work plan, and will be conducted in a manner that is consistent with the Human Health Risk Assessment Methodology presented in Appendix K of the IWP.

#### **5.19.4 Data Requirements**

##### **5.19.4.1 Source Characterization**

Specific data requirements for source characterization in this initial investigation include:

- determining the location of the underground pipelines and confirming the presence or absence of other underground structures by means of a geophysical survey, and
- determining the nature and extent of chemicals in the vicinity of the former dry cleaning solvent USTs and, if present, in the vicinity of the underground pipelines and/or additional underground structures by means of laboratory analysis of core and soil vapor samples.

The methods through which these data requirements will be met are described in Section 5.19.5.

##### **5.19.4.2 Environmental Setting**

Environmental setting is defined by topography, climate, geology, and hydrogeology, all of which affect the movement of chemicals in the environment. The screening assessment, which consists of comparing a measured concentration to the appropriate SAL, does not require site-specific data on environmental setting. However, if conducted, the preliminary health risk assessment may use environmental transport models to predict the movement of chemical vapors in the subsurface. These models take into account a number of physical parameters that characterize the subsurface environment. To support this modeling effort, several discrete-depth core samples of backfill material and tuff will be collected for analysis of bulk density, moisture content, and organic content.

If additional phases of investigation are required, more detailed hydrogeologic and geologic characterization of the soil/tuff may be required. These data, if collected, will be used to evaluate the potential risk associated with release and migration of chemicals or to design and implement corrective measures.

##### **5.19.4.3 Potential Receptors**

Comparison of measured and equivalent soil concentration data with SALs will be used as the initial basis for determining whether there is a potential for adverse human health effects associated with chemical releases from the former dry cleaning solvent USTs or appurtenant structures. SALs are based on conservative default assumptions; thus, no specific information regarding the activities, behavior, or location of actual receptors is required for the screening assessment. Specific information regarding actual receptors also is not required for the preliminary health risk assessment, if conducted, because the site is located on private property, and remedial action will be proposed, if necessary, such that the property is suitable for unrestricted use.

### 5.19.5 Field Sampling Plan

Fieldwork for SWMU 0-039 will be conducted under four tasks: a background review, a geodetic and surface geophysical survey, an underground pipeline removal and investigation program, and an exploratory boring, core sampling, and soil vapor investigation in the vicinity of the former dry cleaning solvent USTs. Fieldwork conducted for this study, including pipeline removal, soil excavation work, and drilling activities, will be performed under permit(s) from the applicable state and local agencies. Before subsurface work begins, the Los Alamos County Utilities will be notified and a private utility locator will be subcontracted to clear the underground pipeline area (Task 2) and borehole locations (Task 4) of underground utilities.

Fieldwork will be performed in accordance with applicable Environmental Restoration (ER) program Standard Operating Procedures (SOPs). Except for the samples collected for field screening purposes, the bulk soil/tuff samples and soil vapor samples collected during this investigation will be analyzed in accordance with the Contract Laboratory Program (CLP) Level III or IV data package. Fieldwork is described below by task.

#### 5.19.5.1 Task 1 - Background Review

Records of past and present use of the site will be reviewed to identify the locations of underground utilities/conduits (e.g., underground pipelines) and appurtenant structures within the SWMU domain, and to evaluate chemical use at the dry cleaning facility. This information will be obtained by conducting interviews with the current and past owners/operators of the dry cleaning facility and by reviewing the Los Alamos Fire Department's and the Laboratory's UST and chemical use records/permits, engineering plans, and other historical records available at the Laboratory. The results of this task will be used to assess the presence of other underground features used for chemical handling or that may act as a conduit for chemical migration at the site, and to confirm the identity of chemicals used at the site.

In addition, readily-available information pertaining to the previous UST site investigation work (e.g., soil analytical data, descriptions, and/or photographs of the USTs removed) that is not presented in the GGI site investigation report (GGI, 1993) will be obtained and reviewed. This information may be obtained from documents provided by and/or interviews with the owners of the site, and GGI and NMED representatives. Review of the information obtained during this task is anticipated to provide a more complete description of the former excavation dimensions, an estimate of the volume of soil excavated and transported off site, laboratory analytical results or field screening measurements of solvents in soil samples, and the condition and type of USTs removed from the site.

#### 5.19.5.2 Task 2 - Geodetic and Surface Geophysical Survey

An initial geodetic survey will be conducted at the site to develop a site plan with horizontal and vertical control points. In conjunction with existing laboratory survey data, the geodetic survey will be performed by surveying the existing dry cleaning building and adjacent pertinent structures, including other buildings, parking areas, fence lines, utilities, and other surface features, as needed. Additional surveying of site investigation features identified during the geophysical survey and subsequent field tasks, such as boreholes, excavations, and underground utilities, also will be performed as needed. The geodetic survey work will be conducted in accordance with the "Draft Interim Guidance on Geodetic Surveying For Townsite Operations."

The screening results provided by Chem-Van will be used to determine if additional excavation is warranted. If VOCs, Stoddard solvent, and/or PNAs are detected in the field, the field team will determine whether additional investigation (as described in Section 5.19.5.4.3) or excavation would be warranted. The decision to determine the nature and extent of chemicals in the subsurface will be based on consideration of factors such as the presence of Stoddard solvent, and/or PNAs in the field screening results; the depths at which these chemicals are detected; and the presence of existing buildings or other structures to the excavation.

After the excavation program has been completed, soil samples will be submitted to the Laboratory's Chemical Science and Engineering for fixed laboratory analysis to confirm the field screening results. Soil samples will be analyzed for VOCs by EPA Method 8150, for Stoddard solvent by EPA Modified Method 8015, and PNAs by EPA Method 8153 (5-103). Additional chemical analyses may be specified if additional chemical use is identified.

OK -  
as long as these  
are taken least determine  
presence of them

#### 5.19.5.4 Task 4 - Exploratory Boring, Core Investigation

This task will consist of drilling up to 12 boreholes (B1-B12) to collect core samples for chemical analysis to further assess the presence of Stoddard solvent in the vicinity of the former dry cleaning solvent USTs (5-104). Low concentrations (less than 1 mg/kg) of noncarcinogenic and of low toxicity; the soil SAL for VOCs is less than 1 mg/kg. Therefore, no additional analysis of PNAs will be conducted for the former dry cleaning solvent USTs.

No comparison

In addition to the composite core samples, discrete samples will be collected at selected sampling intervals for physical and chemical data for the preliminary health risk assessment described in Section 5.19.5.4. Finally, soil vapor samples will be collected for chemical analysis where composite core samples are obtained for chemical analysis. The results of the soil vapor samples will be used to:

- Provide a qualitative comparison of the composite core sample results.
- Evaluate the degree of heterogeneity of the distribution of VOCs.
- Evaluate which sampling method may be more appropriate at other Laboratory sites where similar chemicals are present.

The composite core samples will be submitted to the Laboratory for chemical analysis. Duplicate composite core samples will be delivered to CST-9 for fixed laboratory analysis to confirm the screening results. The Chem-Van screening-level results will be used to guide the field investigation and the location of the boreholes, while the fixed laboratory results will be used to determine the presence of VOCs and Stoddard solvent in the subsurface.

Sample Location	No. of Samples	Sample Description	Field Surveys			Field Screening (Level I)			Field Mobile Laboratory <sup>1</sup>			Laboratory Analysis (Level III/IV) <sup>1</sup>												
			Site Survey/Mapping	Geophysical Survey (Level I)	Soil Gas Survey (Level III)	Gross Alpha, Beta, Gamma (Level I)	Lithologic Logging	Combustible Gas/Oxygen	Organic Vapor	Gross Alpha, Beta, Gamma	Volatile organics (VOA) (SW 8240)	TPH (Modified SW 8015)	PNA (SW 8310 <sup>2</sup> )	Number of samples analyzed depends on results of field screening and/or field survey. * =50%, + =30%										
												Gamma Spectrometry	Total U ( <sup>234</sup> U, <sup>238</sup> U)	<sup>238</sup> Pu, <sup>239/240</sup> Pu	Volatile Organics (VOA) (SW 8240)	BTEX (SW 8020)	Semivolatile Organics (BNA) (SW 8270)	TPH (Modified SW8015)	Target Analyte List Metals	TCLP Metals	PCB and Pesticides (SW 8080)	PNA (SW8310 <sup>2</sup> )	Physical properties <sup>3</sup>	Organic carbon (ASTM D4129-93)
Underground pipelines	4-8	Discrete soil / core samples	•	•				•		•	•				•			•				•		
Boreholes	20-90	Composite core samples								•	•				•			•						
Boreholes	6-36	Discrete - Depth Core																					•	•
Boreholes	20-90	Soil vapor samples			•			•							•			•						

**Notes:**

- 1 The typical laboratory detection limits for tetrachloroethylene (PCE) and Stoddard solvent in soil/core samples, the only volatile chemicals known or suspected to be present at the site, are 0.005 and 0.05 milligrams per kilogram (mg/kg), respectively. However, the soil/core samples to be analyzed for these compounds by EPA Methods 8015 and 8240 will be field extracted using methanol in accordance with ASTM Standard Method D4547-91. As a result, laboratory detection limits for the respective analytical methods likely will be 10 to 100 times higher than the laboratory's typical detection limit when a gas chromatograph/Hall detector is used (e.g., the detection limit for PCE may be up to 0.5 mg/kg).
- 2 Soil/core samples analysed for PNAs by EPA Method 8310 will not be field extracted. These samples will be collected in brass sleeves and delivered to CST-9 in accordance with the appropriate laboratory standard operating procedures. Typical laboratory detection limits for EPA Method 8310 range from 0.16 to 0.33 mg/kg.
- 3 Discrete-depth core samples collected for physical testing will be analyzed in accordance with LANL-ER-SOP-11.01, RO, "Measurement of Bulk Density, Dry Density, Water Content, and Porosity in Soil."

**FIGURE 5-103. Screening and analysis for Investigations at SWMU 0-039, Underground Solvent Storage Tanks**

The screening results provided by Chem-Van will be used to guide the field investigation and to determine if additional excavation should be conducted. If VOCs, Stoddard solvent, and/or PNAs are detected in the field screening samples, the field team will determine whether additional investigation (i.e., borings, as described in Section 5.19.5.4.3) or excavation would be the most practical approach to determining the nature and extent of chemicals in the subsurface. This decision will be based on consideration of factors such as the concentration of VOCs, Stoddard solvent, and/or PNAs in the field screening samples; the locations and depths at which these chemicals are detected; and site constraints (e.g., proximity of existing buildings or other structures to the excavation).

After the excavation program has been completed, soil samples will be collected and submitted to the Laboratory's Chemical Science and Technology Division (CST-9) for fixed laboratory analysis to confirm the field screening results. The confirmation soil samples will be analyzed for VOCs by EPA Method 8240, TPH as Stoddard solvent by EPA Modified Method 8015, and PNAs by EPA Method 8310 (Figure 5-103). Additional chemical analyses may be specified based on the findings of Task 1 if additional chemical use is identified.

#### 5.19.5.4 Task 4 - Exploratory Boring, Core Sampling, and Soil Vapor Investigation

This task will consist of drilling up to 12 boreholes (B1 - B12) and obtaining composite core samples for chemical analysis to further assess the extent of VOCs and Stoddard solvent in the vicinity of the former dry cleaning solvent USTs (Figure 5-104). Low concentrations (less than 1 mg/kg) of naphthalenes were detected in only one of seven soil samples previously analyzed for PNAs. Naphthalenes are noncarcinogenic and of low toxicity; the soil SAL for naphthalene is 3,200 mg/kg. Therefore, no additional analysis of PNAs will be conducted in the vicinity of the former dry cleaning solvent USTs.

In addition to the composite core samples, discrete-depth core samples will be collected at selected sampling intervals for physical testing to provide site-specific data for the preliminary health risk assessment described in Section 5.19.3.3. Finally, soil vapor samples will be collected for chemical analysis at each interval where composite core samples are obtained for chemical analysis. The analytical results of the soil vapor samples will be used to:

- Provide a qualitative comparison of the composite core and soil vapor sample results.
- Evaluate the degree of heterogeneity of the tuff with respect to the distribution of VOCs.
- Evaluate which sampling method may be more appropriate for site characterization at other Laboratory sites where similar chemicals may be present.

The composite core samples will be submitted to Chem-Van for screening-level chemical analysis. Duplicate composite core samples and discrete-depth soil vapor samples will be delivered to CST-9 for fixed laboratory analysis and confirmation of the screening results. The Chem-Van screening-level results for the composite core samples will be used to guide the field investigation and to determine the total depth of the boreholes, while the fixed laboratory results will be used to verify the extent of VOCs and Stoddard solvent in the subsurface.

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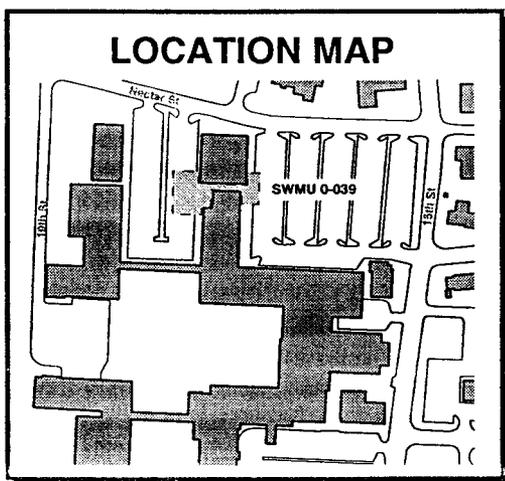
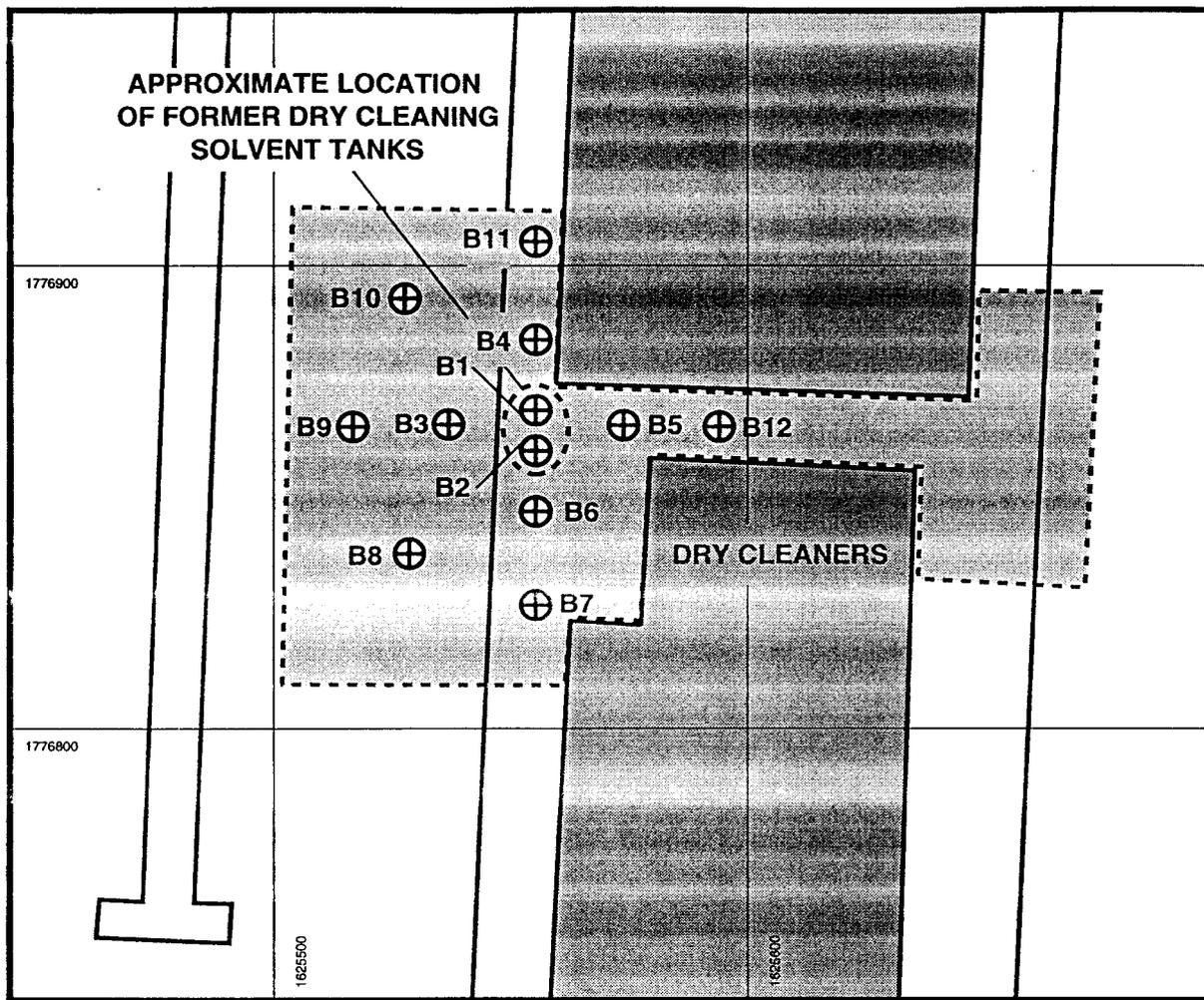


FIGURE 5-104. Boring locations at the former underground dry cleaning solvent tank site (SWMU 0-039).

5.19.5.4.1 Drilling and Core Sampling

The first two borings (B1 and B2) to be drilled will be located within the former excavation; therefore, the total depth of these borings, to be determined based on the results of the Chem-Van screening-level analyses of the composite core samples, likely will represent the maximum depth that VOCs or Stoddard solvent have migrated in the tuff. The total depths of borings B1 and B2 will be used to establish the minimum total depth that the remaining borings will be advanced. For planning purposes, a maximum total depth of 100 feet below ground surface (bgs) is assumed. After drilling and sampling borings B1 and B2, the first round of perimeter borings (B3 - B6) will be drilled and sampled. Additional perimeter borings will be drilled and sampled as needed to assess the extent of VOCs and Stoddard solvent beneath the former USTs.

The borings will be advanced using 8-inch outside-diameter hollow stem augers. To record lithologic descriptions for the soil borings, continuous core samples will be collected from each boring using a 5-foot-long, stainless steel split-barrel sampler and wireline system, or another appropriate continuous dry sampling method. The drill rig and down-hole drilling and sampling equipment will be decontaminated in accordance with a site-specific waste management plan. Upon retrieval, the core samples will be screened initially for health and safety monitoring for organic vapors using a PID and appropriate radiation meters/probes. The core samples will be described visually in accordance with LANL-ER-SOP-12.01, R0, "Field Logging, Handling, and Documentation of Borehole Materials." The borings will be drilled in accordance with LANL-ER-SOP-04.01, R0, "Drilling Methods and Drill Site Management" and LANL-ER-SOP-04.04, R0, "General Borehole Logging."

5.19.5.4.1.1 Collection of Composite Core Samples for Chemical Analysis

Composite core samples will be collected for chemical analysis from each borehole at alternating 5-foot intervals (e.g., 10-15 ft, 20-25 ft, etc.) using the methanol field extraction method in accordance with ASTM Method D4547-91 (Appendix 5.19-A). For borings B1 and B2, the first sampling interval will begin approximately 5 feet below the contact of the excavation backfill and the native tuff. Subsequent samples will be collected at alternating 5-foot intervals. Sampling intervals in the remaining boreholes (i.e., B3 to B12) will begin at 10 feet bgs and then will be adjusted to coincide with the B1/B2 sample intervals. The total depth of the boreholes will be based on the results of the field-screening chemical analyses, as described below.

Representative sub-samples of each 1-foot interval within the 5-foot sample interval will be composited in the field. For example, given a typical 5-foot core interval, a total of five sub-samples would be collected from the cored interval and placed in a methanol container for field extraction and preservation.

An additional composite core sample should be collected from core intervals originally not planned to be sampled if free solvent product is visually observed in a core sample interval and/or if the PID readings measured for health and safety monitoring are significantly higher than the PID readings of the previous core interval. Sampling then would continue as described above.

The composite core samples should be collected with minimum disturbance of the core samples and transferred immediately to the appropriate methanol container for field extraction and preservation. At each sampling interval, the methanol will be split into two subsamples. One subsample will be submitted to Chem-Van for on-site

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screening-level chemical analysis for VOCs by EPA Method 8240 and for TPH as Stoddard solvent by Modified EPA Method 8015 (Figure 5-103). The other subsample will be submitted to CST-9 for fixed laboratory analysis of VOCs by EPA Method 8240 and TPH as Stoddard solvent by EPA Modified Method 8015 to confirm the field screening results.

Analytical results of the composite core samples from Chem-Van will be used in the field to decide whether the boreholes should be drilled deeper. If it is decided to stop drilling, then the borehole will be grouted with a cement-bentonite grout from the bottom of the boring to the ground surface. However, if it is decided to continue drilling, then the boring will be advanced 10 feet, and a composite core sample will be collected at the next interval and submitted to Chem-Van for screening-level analysis and to CST-9 for fixed laboratory analysis as described above. This process will be repeated until the vertical extent of chemicals in the subsurface is adequately defined.

#### 5.19.5.4.1.2 Collection of Discrete-Depth Core Samples for Physical Testing

A maximum of three discrete-depth core samples will be collected in brass sleeves from the first six borings at depths of approximately 10 ft, 25 ft, and 50 ft bgs or the bottom of the borehole, whichever is encountered first, for laboratory geotechnical analysis (Figure 5-103). These samples will be collected in accordance with LANL-ER-SOP-06.24, R0, "Sample Collection from Split-Spoon Samplers and Shelby Tube Samplers," and analyzed for bulk density, dry density, water content, and porosity in accordance with LANL-ER-SOP-11.01, R0, "Measurement of Bulk Density, Dry Density, Water Content, and Porosity in Soil." In addition, these core samples will be analyzed for organic carbon by ASTM Method D4129-93, or another appropriate laboratory method.

#### 5.19.5.4.2 Soil Vapor Sampling

Soil vapor samples will be collected from each boring at the same interval that composite core samples are collected. The field procedures for collecting soil vapor samples in conjunction with hollow-stem auger drilling are described in Appendix 5.19-B. At each sample interval, soil vapor will be screened in the field with a PID. One soil vapor sample will be collected from each sample interval and submitted to CST-9 for fixed laboratory analysis for VOCs by EPA Method 8240 and for TPH as Stoddard solvent by EPA Modified Method 8015.

#### 5.19.5.4.3 Additional Drilling, Core Sampling, and Soil Gas Investigation

If the analytical results of the screening-level core samples from the former UST area indicate that VOCs and/or Stoddard solvent in the tuff extend laterally beyond the location of the proposed borings, the field program will be expanded to include drilling additional borings and collecting composite core samples as needed to define the lateral and vertical extent of VOCs and/or Stoddard solvent. The location(s) of the additional borings will be based on an evaluation of the screening-level composite core results. Drilling, core sampling, and soil vapor sampling will proceed as described in Sections 5.19.5.4.1 and 5.19.5.4.2.

Following evaluation of the screening-level results of soil/tuff samples analyzed by Chem-Van during the shallow soil sampling program (Section 5.19.5.3.2) conducted in the underground pipeline area, an exploratory boring, core sampling, and soil vapor sampling program may be implemented near the underground pipelines as

needed to assess the extent of VOCs, Stoddard solvent, and/or PNAs beneath the site. The location of exploratory borings will be based on the field screening and laboratory analytical results obtained during the shallow soil sampling program described in Section 5.19.5.3. Additional drilling, core sampling, and soil vapor sampling will proceed as described in Sections 5.19.5.4.1 and 5.19.5.4.2.

#### 5.19.6 Report Preparation

A RCRA Facility Investigation (RFI) report will be prepared following completion of the field sampling program, screening assessment, and, if necessary, preliminary health risk assessment. The RFI report will include the following:

- a summary of the background information obtained in Task 1 of the field sampling program,
- documentation of the pipeline removal activities,
- logs of boreholes drilled,
- a summary of field screening and/or laboratory chemical and physical results of bulk core samples, composite core samples, and soil vapor samples collected,
- results of the screening assessment, including a description of methods used, and
- if necessary based on the results of the screening assessment, results of a preliminary health risk assessment, including a description of methods used.

Based on the results of the field sampling program, screening assessment, and, if necessary, preliminary health risk assessment, recommendations regarding the need for corrective action will be made.

#### 5.19.7 References

Department of Energy (DOE), 1994, Letter to Mr. William K. Honker, Chief, RCRA Permits Branch, U.S. Environmental Protection Agency, Region VI from Mr. Theodore J. Taylor, Program Manager, Environmental Restoration Program, 5 July.

EPA, 1990, Corrective Action for Solid Waste Management Units at Hazardous Waste Management Facilities; Proposed Rule, Federal Register, Volume 55, pp. 30798-30884, 27 July.

EPA, 1992, Air/Superfund National Technical Guidance Study Series, Assessing Potential Indoor Air Impacts for Superfund Sites: EPA-451/R-92-002, Office of Air Quality, Planning and Standards, Research Triangle Park, NC.

EPA, 1994, Letter to Mr. Theodore J. Taylor, Program Manager, Environmental Restoration Program, Department of Energy from Mr. William K. Honker, Chief, RCRA Permits Branch, Region VI, 21 July.

Glorieta GeoScience, Inc. (GGI), 1993, 7 & 30-Day Reports, Community Center, Los Alamos, New Mexico, July.

Los Alamos National Laboratory (LANL), 1993, Installation Work Plan for Environmental Restoration, Revision 3, Report LA-UR-93-3987.

New Mexico Environment Department (NMED), 1988, Underground Storage Tank Regulations, 15 March, amended 12 September 1988 and 14 February 1989.

NMED, 1994, Letter to Mr. Tom Netuschil from Mr. Jerry Schoeppner, Geologist, Underground Storage Tank Bureau, 7 February.

**APPENDIX 5.19-A  
ASTM STANDARD METHODS**

ASTM Method D 4129-88

Standard Test Method for Total and Organic Carbon in Water by High  
Temperature Oxidation and by Coulometric Detection

ASTM Method D 4547-91

Standard Practice for Sampling Waste and Soils for Volatile Organics



## Standard Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection<sup>1</sup>

This standard is issued under the fixed designation D 4129; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This test method covers the determination of total and organic carbon in water and waste water, including brackish waters and brines in the range from 2 to 20 000 mg/L. This test method has the advantages of a wide range of concentration which may be determined without sample dilution and the provision for boat or capillary introduction of samples containing sediments and particulate matter where syringe injection is inappropriate.

1.2 This procedure is applicable only to that carbonaceous matter in the sample that can be introduced into the reaction zone. When syringe injection is used to introduce samples into the combustion zone, the syringe needle opening size limits the maximum size of particles that can be present in samples. Sludge and sediment samples must be homogenized prior to sampling with a micropipetor or other appropriate sampler and ladle introduction into the combustion zone is required.

1.3 The precision and bias information reported in this test method was obtained in collaborative testing that included waters of the following types: distilled, deionized, potable, natural, brine, municipal and industrial waste, and water derived from oil shale retorting. Since the precision and bias information reported may not apply to waters of all matrices, it is the user's responsibility to ensure the validity of this test method on samples of other matrices.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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. For specific cautionary statements, see 9.1 and 10.2.1.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 513 Test Methods for Total and Dissolved Carbon Dioxide in Water<sup>2</sup>

D 1129 Terminology Relating to Water<sup>2</sup>

D 1193 Specification for Reagent Water<sup>2</sup>

D 3370 Practices for Sampling Water<sup>2</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

Current edition approved June 24, 1988. Published August 1988. Originally published as D 4129 – 82. Last previous edition D 4129 – 82.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

D 4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data<sup>2</sup>

### 3. Definitions

3.1 For definitions of terms used in this test method, refer to Definitions D 1129.

### 4. Summary of Test Method

4.1 The sample is homogenized or diluted, or both, as necessary. If the sample does not contain suspended particles or high-salt level a 0.200-mL portion is injected into the reaction zone. For samples containing solids or high salt levels, portions are placed in combustion boats containing tungsten trioxide ( $WO_3$ ) or quartz capillaries and introduced into the reaction zone using a ladle. In the reaction zone the heat, oxidation catalyst and oxygen atmosphere convert carbonaceous matter to carbon dioxide ( $CO_2$ ). The oxygen gas stream sweeps the gaseous reaction products through a series of scrubbers for potentially interfering gases and then to the absorption/titration cell. The  $CO_2$  is determined by automatic coulometric titration. Calibration by testing known carbon content standards is not required, however, standards are analyzed periodically to confirm proper operation.

4.2 Carbon dioxide is liberated from carbonates as well as from organic matter under the reaction conditions. Organic carbon is determined by difference between the total carbon and the inorganic carbon determined separately or by acidifying a portion of the sample to a pH of 2 or less and sparging with carbon dioxide-free gas to remove carbonates, bicarbonates, and dissolved carbon dioxide prior to total carbon determination. To determine organic carbon by difference the inorganic carbon is determined by acid release of carbon dioxide from a portion of the sample or other methods as given in Test Methods D 513. For discussion of the limitations and guidelines for the use of the sparge technique see 5.4 and the paper by Van Hall.<sup>3</sup>

4.3 Because of the various properties of carbon-containing compounds in water, any preliminary treatment of a sample prior to injection dictates a definition of the carbon measured. Filtration of the sample prior to injection will limit the carbon measured to dissolved carbonates and dissolved organic matter. Homogenizing permits determination of the

<sup>3</sup> Van Hall, C. E., Barth, D., and Stenger, V. A., "Elimination of Carbonates from Aqueous Solutions Prior to Organic Carbon Determinations," *Analytical Chemistry*, Vol 37, 1965, pp. 769–771.

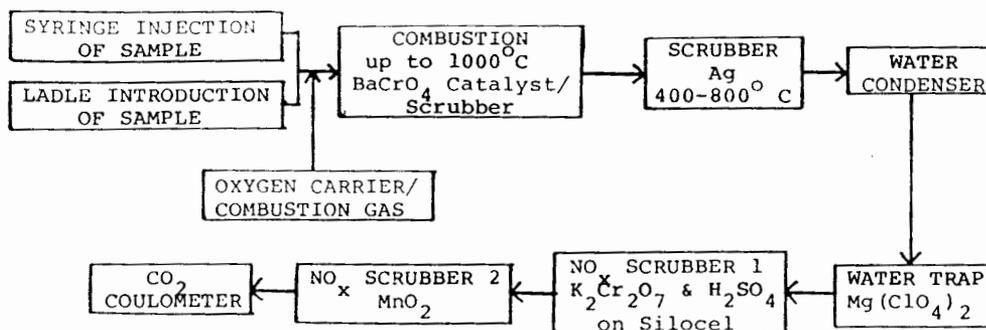


FIG. 1 Total Carbon and TOC Apparatus

carbon in insoluble carbonates and insoluble organic materials.

## 5. Significance and Use

5.1 This test method is necessary because of the need for rapid reliable tests for carbonaceous material in waters and sediments.

5.2 It is used for determining the concentration of organic carbon in water that comes from a variety of natural, domestic, and industrial sources. Typically, these measurements are used to monitor organic pollutants in domestic and industrial waste water.

5.3 When a sample is homogenized so that particulate, immiscible phases, and dissolved carbon from both organic and inorganic sources is determined, the measurement is called total carbon (TC). When inorganic carbon response is eliminated by removing the dissolved  $\text{CO}_2$  prior to the analysis or the dissolved  $\text{CO}_2$  concentration subtracted from the total carbon concentration, the measurement is called total organic carbon (TOC). When particulates and immiscible phases are removed prior to analysis the measurement is called dissolved carbon (DC), or dissolved organic carbon (DOC) if inorganic carbon response has been eliminated.

5.4 Homogenizing or sparging of a sample, or both, may cause loss of volatile organics, thus yielding a negative error. The extent and significance of such losses must be evaluated on an individual basis. If significant quantities of volatile carbonaceous materials are present or may be present in samples organic carbon should be determined by the difference between the total carbon and the inorganic carbon concentrations. When organic carbon determined both by difference and by sparging agree it is acceptable to determine organic carbon by sparging for similar samples.

5.5 The relationship of TOC to other water quality parameters such as COD and BOD is described in the literature.<sup>4</sup>

## 6. Interferences

6.1 Any acidic or basic gas formed in the oxidation of the sample and not removed by the scrubbers will interfere with the test. Potentially interfering gases that are removed by the scrubbers include hydrogen sulfide ( $\text{H}_2\text{S}$ ), hydrogen chloride

( $\text{HCl}$ ), hydrogen bromide ( $\text{HBr}$ ), hydrogen iodide ( $\text{HI}$ ), sulfur dioxide ( $\text{SO}_2$ ), sulfur trioxide ( $\text{SO}_3$ ) free halogens, halogen oxides, and nitrogen oxides. Hydrogen fluoride ( $\text{HF}$ ) may be removed by bubbling the gas stream through water in the water vapor condenser.

6.2 The capacity of the scrubbers for potentially interfering gases may vary with the type of samples being analyzed. If the scrubber capacity is exceeded it can be recognized by the titration continuing beyond the normal analysis time at a higher rate than the blank and high results for known carbon content standards as well as by appearance changes in the scrubbers. If the scrubber capacity is exceeded during an analysis the scrubbers should be replaced and the analysis repeated. Samples containing all concentrations of the potentially interfering species can be analyzed if the analyst uses great care to ensure that the scrubbers are and remain effective for his samples. The frequency of replacing the scrubbers will depend on the nature of the samples.

## 7. Apparatus

7.1 Apparatus for total carbon, organic carbon, and inorganic carbon determinations—combustion furnace with gas supply, gas purification train, flow control, acid reaction train, and carbon dioxide coulometer.<sup>5</sup> Figures 1 and 2 show block diagrams of the apparatus.

7.2 *Sampling Devices*—A spring-loaded .200-mL syringe<sup>6</sup> (carbon analyzer syringe) having an all metal tip and a 50 mm long, 0.5-mm inside diameter needle with a square end is recommended for water samples containing little or no particulate matter.

7.3 *Homogenizing Apparatus*—A household blender with glass mixing chamber is generally satisfactory for homogenizing immiscible phases in water.

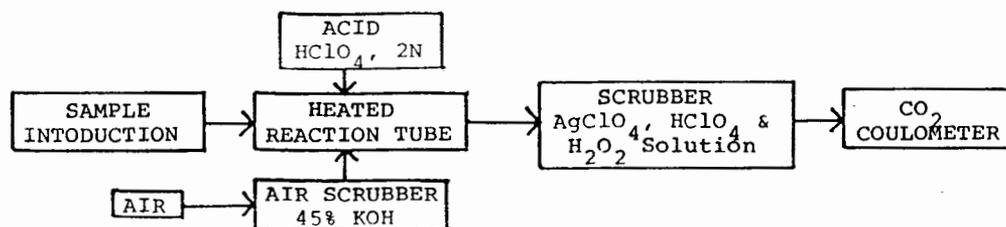
## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chem-

<sup>4</sup> Handbook for Monitoring Industrial Wastewater, U.S. Environment Protection Agency, August 1973, pp. 5-10 to 5-12.

<sup>5</sup> Instruments marketed by Coulometrics, Inc., a subsidiary of UIC Inc., P.O. Box 563, Joliet, IL, 60434, or an equivalent, have been found satisfactory.

<sup>6</sup> Syringes manufactured by Hamilton Co., P.O. Box 10030, Reno, NV 89510, or an equivalent, have been found satisfactory for this purpose.

FIG. 2 CO<sub>2</sub> Evolution Apparatus

ical Society.<sup>7</sup> Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to the Specification D 1193, Type II. Where specified, carbon dioxide-free water is to be prepared by boiling distilled water in a conical flask for 20 min. The boiled water is cooled in the flask stoppered with a one-hole rubber stopper fitted to a soda lime-Ascarite drying tube. For large (10 to 20 L) volumes of carbon dioxide-free water, the absorbed carbon dioxide may be removed by inserting a fritted-glass gas-dispersion tube to the bottom of the container and vigorously bubbling nitrogen through the water for at least 1 h. Carbon dioxide-free water may be stored if properly protected from atmospheric contamination.

NOTE 1—Glass containers are preferred for the storage of reagent water and most standard solutions. It is necessary to provide protection against changes in quality due to the absorption of gases or water vapor from the laboratory air. As volumes of fluid are withdrawn from the container, the replacement air should be passed through a drying tube filled with equal parts of 8 to 20-mesh soda lime, oxalic acid, and 4 to 8-mesh anhydrous calcium chloride, each product being separated from the other by a glass-wool plug.

8.3 *Gas Supply*—Use oxygen of at least 99.6 % purity.

8.4 *Scrubber Tubes and Catalyst Packings* as well as instructions for their preparation are available from the equipment manufacturer.<sup>8</sup> Figure 1 illustrates the flow diagram and names the reagents used.

8.5 *Carbon Dioxide Coulometer Reagents*—Cell solutions to absorb CO<sub>2</sub> from the gas stream and convert it to a titratable acid and permit 100 % efficient coulometric titration.<sup>8</sup>

8.6 *Acid*—Various acids may be used for acidification of samples. Hydrochloric acid is recommended. Phosphoric and sulfuric acids are suitable if they do not cause materials to precipitate from the sample. Nitric acid is not recommended because it may cause premature oxidation of organics in the sample.

8.7 *Organic Carbon Standard Solutions*—Although the method does not require sample standardization, proper

operation of the instrument should be confirmed by injection of standards of similar composition and concentration to the unknown. Standards should be stable water soluble compounds such as KHP or benzoic acid of suitable purity.

## 9. Hazards

9.1 Injection of samples containing over 25 000 mg/L TOC or 0.5 mL water may cause explosion of the combustion tube.

## 10. Sampling

10.1 Collect the sample in accordance with Practices D 3370 or other applicable ASTM method(s).

### 10.2 Preservation:

10.2.1 To preserve samples for this analysis, store or ship samples in glass at or below 4°C. **Caution**—Head space in the sample bottle or freezing the sample may contribute to the loss of volatile organics from some samples.

10.3 For monitoring of waters containing solids or immiscible liquids of interest, use a mechanical homogenizer or ultrasonic disintegrator to homogenize samples.

10.4 For waste water streams where carbon concentrations are greater than the desired range of instrument operation, provide on-stream dilution of the sample if possible.

10.5 Annex A1.1 gives additional guidelines for preparing heavily contaminated water samples when using the sparge technique.

10.6 Annex A1.2 gives additional guidelines for samples containing solids and immiscible liquids.

## 11. Calibration and Standardization

11.1 Set up the analyzer and fill coulometer cell in accordance with the manufacturer's specifications. Adjust the gas flow to 80 to 100 mL/min. Set the readout to milligrams per litre except when other than 0.200-mL samples are used in which case set the readout to micrograms.

11.2 Analyze samples of carbon dioxide-free water as instructed in Section 12 for samples to determine the instrument blank, *B*.

11.3 Calibration is not required, however, inject appropriate standard(s) prior to and following analysis of samples to confirm proper operation. The standard concentration(s) should be approximately that of the samples to be analyzed. If the recovery of standards is unacceptable the cause of poor results should be determined and corrected. Low results suggest a leak or exhausted combustion tube packing. High results suggest contamination of the samples or exhausted

<sup>7</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the Am. Chemical Soc., see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

<sup>8</sup> Satisfactory reagents available from Coulometrics, Inc., a subsidiary of UIC Inc., P.O. Box 563, Joliet, IL, 60434 use ethanolamine to absorb CO<sub>2</sub> forming hydroxycarbanic acid that is titrated coulometrically using a color indicator for end-point detection.

scrubber or combustion tube fillings.

## 12. Procedure

12.1 Condition each sample to bring the homogenous carbon content within range. Although analyses of samples containing up to 20 000 mg/L TOC is possible, dilution to TOC levels below 1000 is preferable if the sample contains salts or forms a precipitate upon acidification.

12.2 See 12.3, 12.4, and 12.5 for total carbon and TOC by sparging; 12.6 applies to carbonate carbon determinations when organic carbon is to be determined by difference.

12.3 *Syringe Injection of Samples*—Rinse the syringe several times with the solution to be analyzed, then fill to precise volume (0.200 mL). Wipe off the excess from the needle tip with soft paper tissue, taking care that no lint adheres to the needle. Insert the sample syringe into the injection port, inject the sample, and reset the coulometer to zero. Leave the syringe in the holder until the analysis is completed.

### 12.4 Ladle Introduction of Samples:

12.4.1 When a micropipet can be used with the sample, place 0.200 mL of the sample into a platinum boat or a capillary tube containing approximately 20 mg of  $WO_3$ . Position the boat (capillary) in the ladle and place the ladle in the cool portion of the combustion tube through the introduction port. After closing the introduction port, allow 60 s for the oxygen gas stream to sweep out air that entered then move the ladle into the furnace.

NOTE 2—The  $WO_3$  is used to minimize potential difficulties caused by salts in samples. Use of the  $WO_3$  will minimize the splattering of the sample which allows salts in the sample to degrade the combustion tube. The  $WO_3$  also helps prevent salts from reacting with  $CO_2$  forming carbonates which then decompose slowly lengthening the analysis time and increasing the instrument blank.

12.4.2 When a precise volume (0.200 mL) of the sample cannot be obtained, weigh the sample into the combustion boat or capillary tube and introduce it into the combustion zone as described in 12.4.1.

NOTE 3—When weighing samples the size of the sample may be increased. The carbon content must not exceed 4 mg or the water content exceed 0.4 mL.

NOTE 4—The density of the sample must be known to report the results if the result is to be given in mg/L when samples are weighed into combustion boats.

12.5 Consistent analysis times must be used for all samples and blanks. The time must be sufficient for all  $CO_2$  to be swept from the combustion tube to the coulometer and titrated as evidenced by stable coulometer readings. The time required will depend upon the nature of the samples and is normally 3 to 7 min. High-level samples require longer analysis times than low level samples and may result in higher blank levels, especially if the samples are high in salts. If samples of a large concentration range are being analyzed care must be used when analyzing lower level samples following much higher level samples. Additional blanks must be run to confirm that the blank is reasonable and consistent.

12.6 Carbonate carbon may be determined using the methods given in Test Methods D 513 or as instructed by equipment manufacturer.

## 13. Calculation

13.1 Read total carbon values of 0.200-mL samples di-

rectly from the digital display. Correct these values by subtracting the blank value,  $B$ , obtained with carbon dioxide-free water and correct for any dilutions made to obtain original sample values.

13.2 For organic carbon values of acidified and sparged 0.200-mL samples, read the values directly from the digital display. Correct these values by subtracting the blank value  $B$ , and correct for any dilutions in acidifying or other steps to obtain original sample values.

13.3 For organic carbon values determined by difference the result is obtained as follows:

$$O = T - C$$

where:

$O$  = organic carbon for original sample, mg/L

$T$  = total carbon corrected for any blank and dilutions mg/L, and

$C$  = carbonate carbon corrected for any blank and dilutions calculated in accordance with the instructions for the method used, mg/L.

NOTE 5—The digital display of the coulometer is set for readout in milligrams of carbon per litre for 0.200-mL samples. When other sample volumes are used the readout may be changed to compensate for the volume change or the correct value calculated using the new sample volume without changing the display units. The readout may also be set to be in micrograms of carbon.

13.4 When total carbon or organic carbon is determined by introduction in a boat, capsule or capillary tube, the concentration of carbon is determined as follows if the volume used is not 0.200 mL:

$$c = m/V \text{ or } md/W$$

where:

$c$  = concentration of carbon, mg/L,

$m$  = micrograms of carbon in sample, corrected for instrument blank,

$V$  = volume of sample, mL,

$d$  = density of sample, g/mL, and

$W$  = weight of sample used, g.

NOTE 6—In some cases, such as for sediments, it may be desirable to know the concentration per gram of sample instead of per litre, in which case the following may be used:  $c = m/W \times 1000$  where  $c$  is now the concentration of carbon in milligrams per litre.

## 14. Report

14.1 The results may be reported in milligrams per litre or other units as desired. The units used must be clearly noted in the report.

## 15. Precision and Bias<sup>9</sup>

15.1 The overall and single-operator precision of this test method varies with the concentration. The precision for standards and for laboratory samples of interest are shown in Fig. 3.

15.2 The observed precision and bias for a series of potassium hydrogen phthalate in water standards were as shown in Table 1.

15.3 The recoveries from standard solutions and samples of interest were as shown in Table 2.

<sup>9</sup> Supporting data are available from ASTM Headquarters. Request RR: D19-1094.

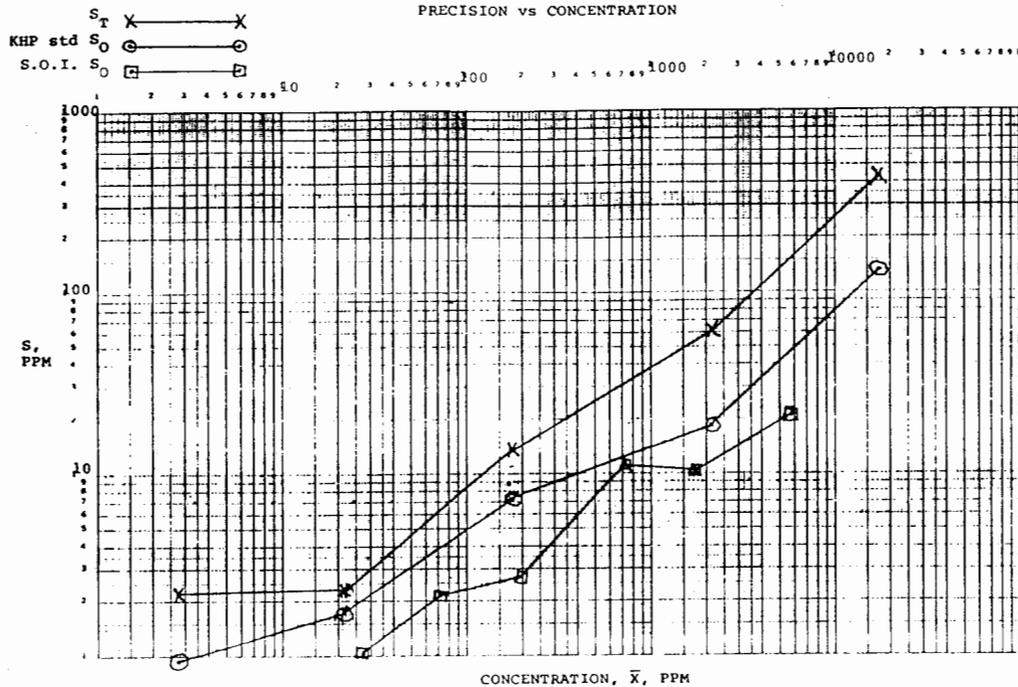


FIG. 3 Precision Versus Concentration

TABLE 1 Precision and Bias

Sample	Added Amount, mg/L	$\bar{X}$ , mg/L	$S_O$ , mg/L	% $S_O$	$S_T$ , mg/L	% $S_T$	Bias, mg/L	% Bias	Significant?
102	22	21.2 <sup>A</sup>	1.8	8.3	2.4	11.2	-0.8	-3.6	no
103	180	178. <sup>A</sup>	7.3	4.1	13.5	7.6	-2.	-1.1	no
104	2200	2160. <sup>A</sup>	19.	0.9	61.	2.8	-40.	-1.8	yes <sup>B</sup>
104	18 000	17800. <sup>A</sup>	130.	0.7	420.	2.3	-200.	-1.1	no

<sup>A</sup> Value after subtracting result for blank, sample 101 which had an  $\bar{X}$  of 2.7 mg/L and an  $S_O$  of 0.9 mg/L.

<sup>B</sup> This bias is statistically significant at the 95 % confidence level. The user of the method may not find the bias significant in his application.

15.4 Nine laboratories participated in the collaborative testing using syringe injection of sparged samples. Samples of interest included: distilled, deionized, municipal tap, natural, brine, waste, oil shale retort, and industrial process waters. For other matrices or other techniques (ladle introduction, TOC calculated by difference between total carbon and dissolved CO<sub>2</sub>) the precision and accuracy may vary. It is the user's responsibility to determine the validity of the method and the resulting precision and accuracy.

15.5 The testing program required laboratories to test samples covering the full concentration range of the method, 2 to 20 000 mg TOC/L (five samples of potassium hydrogen phthalate in distilled water and three samples of interest analyzed before and after spike additions).

NOTE 7—The full concentration range is seldom encountered in normal practice and presented some difficulties for the participants. The problems were maintaining a stable blank in going from extremely high concentrations to low concentrations and use of appropriate standards. The procedure calls for determination of the instrument blank and testing of standard(s) before running samples. When a standard appropriate for the high concentrations is tested the instrument requires some time to stabilize sufficiently for the lowest concentration samples. In normal laboratory practice the extreme concentration range is not likely

to be encountered and standards can be more easily chosen and tested. The reported precision and bias may be poorer than a laboratory will achieve working in a narrower concentration range.

### 16. Quality Assurance/Quality Control (QA/QC)

16.1 Before this test method is applied to the analysis of samples of unknown concentration, the analyst should conduct six duplicate analyses of known and nearly the same concentration by the procedure.

16.2 Calculate the standard deviation of the data according to Practice D 4210. If the value obtained is within that given in the procedure for single operator precision, construct a preliminary control chart.

16.3 Continue to analyse samples in duplicate until at least 40 data points have been accumulated. Follow suggestions in Section 5 of Practice D 4210.

16.4 Prepare a control chart with upper and lower limits from data obtained from analysing replicate standard solutions following Section 6 of Practice D 4210.

16.5 To monitor precision and bias, a minimum of one standard and one sample of known value or a spiked sample must be analysed each day.

**TABLE 2 Recoveries<sup>a</sup>**

Matrix	Concentration Range, mg/L			
	22	50+	500+	4500+
Deionized water, avg	98.2%	99.0%	98.2%	99.0%
S <sub>avg</sub>	4.7	6.9	2.8	2.3
Sample of interest, avg	N/A	103.0	98.7	97.3
S <sub>avg</sub>	N/A	10.9	1.3	5.4

<sup>a</sup> None of the S<sub>avg</sub> are significantly different from 100 % at the 95 % confidence level.

16.6 Other QA/QC portions of this test method have not been completely established at this time. Analysts performing this test method will be required to measure their performance against the performance level achieved by the laboratories that participated in the ASTM round-robin study done on this test method. These formal QA/QC procedures will be incorporated at such time as they have been officially accepted by the Society.

## ANNEXES

### (Mandatory Information)

#### A1. SAMPLE PREPARATION FOR HEAVILY CONTAMINATED WATERS

A1.1 Improper preparation of heavily contaminated water samples may yield erroneously low results. Acidification of such water can cause separation of organics that may be lost during subsequent sampling and injection. For example, an organic acid may be soluble in a high-pH water but, because of its high concentration, not soluble upon acidification. This problem can be solved by first diluting the sample and then acidifying slowly while stirring. Dilution tends to keep the organics in solution and minimizes problems if salts are present, while slow acidification of the diluted sample tends to keep the insolubles formed small in particle size and well dispersed. If the TOC concentration is not much smaller than the TC concentration, determination of TOC by difference may be preferred over determination of TOC by use of the sparge technique.

A1.2 The procedure for acidifying and sparging samples is as follows:

A1.2.1 Blend the water sample, if necessary, to produce a homogeneous sample suitable for dilution.

A1.2.2 Dilute the blended sample sufficiently with water to improve solubility and suspension of potentially insoluble organics. **CAUTION—DO NOT REDUCE ORGANIC**

**CARBON CONCENTRATION BELOW THE RANGE OF THE METHOD.** Determine the approximate level initially if uncertain.

A1.2.3 While stirring, acidify the diluted sample to pH 2 to 3 slowly so as to keep any particles formed small in size and well dispersed. A dilute acid may be required to accomplish this. Note the quality of acid added for later volumetric correction of results if a known sample volume is not being diluted to a known final volume.

A1.2.4 Sparge the sample to complete removal of dissolved CO<sub>2</sub>.

A1.2.5 Blend the acidified sample, if necessary, and while stirring, take an aliquot for analysis using an appropriate syringe or micropipet.

NOTE A1.1—For some samples it has been found convenient to add sufficient alkali to cause the organic acids to redissolve. If this is done the sample should be analyzed immediately afterwards to minimize the absorption of CO<sub>2</sub> from the air.

A1.2.6 The blank correction for the instrument should be performed on CO<sub>2</sub> free water treated identically to the samples to compensate for organic contaminants in the reagents added to the samples.

#### A2. SAMPLE CONDITIONING FOR SUSPENDED SOLIDS AND IMMISCIBLE LIQUIDS

A2.1 If the sample is relatively homogeneous, no conditioning will be required except for possible dilution and mixing.

A2.2 Samples containing solids of no interest should be filtered prior to analysis. Sedimentation or centrifugation may also be employed for solids removal if desired.

A2.3 For laboratory analysis of samples containing immiscible liquid or solid phases of interest, homogenize the sample in a glass household-type blender or an ultrasonic disintegrator. Reproducibility of results will indicate when homogenization of the sample is complete.

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*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.*



## Standard Practice for Sampling Waste and Soils for Volatile Organics<sup>1</sup>

This standard is issued under the fixed designation D 4547; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This practice describes field sampling of solid wastes for subsequent volatile organics analysis in the laboratory. This practice is also intended to apply to soils and sediments that may contain volatile waste constituents.

1.2 Both the collection of the sample and the method of containing the sample for shipment to the laboratory are considered.

1.3 This practice concerns only sampling methods to be used in the field; it does not cover laboratory preparation of containers or solutions or other laboratory techniques related to processing or analysis of the samples.

1.4 It is recommended that this standard be used in conjunction with Guide D 4687.

1.5 *This standard does not purport to address all of the safety problems, 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.* For specific precautionary statements, see Section 6.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 3550 Practice for Ring-Lined Barrel Sampling of Soils<sup>2</sup>

D 4687 Guide for General Planning of Waste Sampling<sup>3</sup>

### 3. Terminology

#### 3.1 Description of Terms Specific to This Standard:

3.1.1 *material*—for the purposes of this practice, material covers any soil or wastes that are solid.

3.1.2 *sample*—the portion of the waste or soil material that is initially collected using the techniques described in this practice; portions of the waste or soil, in generic terms.

3.1.3 *subsample*—the portion of the waste or soil that is collected by subdividing or trimming of the initial sample.

3.1.4 *waste*—for the purposes of this practice, waste covers any discarded material that is solid in form.

### 4. Summary of Practice

4.1 Samples of soils or wastes can be obtained with minimal loss of volatile organic constituents. Materials may be either sampled from ground surface or test pits or obtained by using down-hole coring devices. These samples may be shipped in metal rings (that is, hollow metal

cylinders) directly to the laboratory, or they may be subsampled by trimming or by using a small coring cylinder. With the coring method, the coring cylinder is driven into the waste or soil surface to remove the solid material without exposure to the air. The subsample is then extruded from the cylinder directly into a sample container. This method does not apply to cemented material or material with fragments coarse enough to interfere with proper coring techniques; such samples are trimmed before handling.

4.2 Subsamples obtained in the field are contained so as to prevent loss of volatiles using one of the two following methods: (1) the subsample is stored in a glass bottle with methanol; or (2) the subsample is stored in a vial designed to minimize loss of volatiles (for example, a specially adapted VOA vial). Advantages and disadvantages to both methods are discussed in Sections 7 and 8.

### 5. Significance and Use

5.1 The objective of this practice is to provide procedures for obtaining samples which will result in analytical data representative of the concentrations and compositions of volatile compounds actually present in the waste or soil. The procedure also allows for correlation of the analytical data with other properties of the waste or soil materials. Several factors are identified that influence the objective of this procedure.

#### 5.1.1 Loss of Volatiles:

5.1.1.1 Loss of volatile organics during sample collection, handling, and shipping affects the concentrations detected by the laboratory. Comparison of the field testing of volatiles (using a gas chromatograph) with subsequent laboratory testing of the solids or ground water from the same zone suggests that losses can be significant, but are not necessarily due to one particular part of the sampling and analysis process. The principal mechanisms of loss are volatilization of the compounds and biodegradation. Susceptibility of the various compounds to these losses varies. Both the actual concentrations and the relative amounts of the compounds detected can be affected. In some cases, the loss of a compound or the formation of other compounds not actually present in the waste can occur. Compound gain and loss will result in analyses that are both unrepresentative of field conditions and subject to ambiguities in interpretation.

5.1.2 *Selection of Samples for Analysis*—The choice of representative samples is of particular concern in waste materials and soils in which heterogeneities are significant. Interpretation of the analytical data is generally improved if the individual(s) most familiar with the site can describe and select the sample(s) to be analyzed in the laboratory.

5.1.3 *Analytical Considerations*—The method of sample handling and containment is dependent on the method to be

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.01 on Sampling and Monitoring.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 04.08.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.04.

used in the laboratory for analysis of the volatile compounds. The laboratory methods are addressed here only insofar as they affect the collection method and influence the objective stated above.

5.1.4 Other factors affecting the interpretation of data are as follows: sample size, sample matrix, whether the subsample analyzed is representative of the entire sample, potential losses during handling of the sample using the laboratory procedure, and detection limit.

5.2 This practice should be used in conjunction with Practice D 3550 and Guide D 4687.

## 6. Safety

6.1 Proper safety precautions must always be observed when sampling solid waste or contaminated soil. For general guidelines to safety precautions, refer to Guide D 4687 and Practice D 3350. These standards, however, should only complement the judgement of an experienced professional.

## 7. Sampling

### 7.1 Introduction:

7.1.1 This section is intended to define general sampling guidelines to be applied to a variety of possible materials and conditions. Many of the specific materials and conditions, however, are not addressed in this document. Specific sampling methods are presented for granular materials (for example, contaminated soils and non-cemented solid wastes) and materials that are cemented or of sufficient cohesion to make driven samplers impractical. The procedures are intended to allow flexibility in the following:

7.1.1.1 The means of collection (for example, from test pits, surface sampling, and sampling during drilling),

7.1.1.2 The selection of a method suited for the individual requirements of a project or the conditions encountered at a particular site, and

7.1.1.3 The design and dimensions of the actual sampling equipment.

### 7.2 General Methods:

7.2.1 The sampling procedure should be completed in a minimum amount of time, with the least possible handling of the sample before it is sealed in a container.

7.2.2 Rough trimming of the sample in the field should be considered if cross-contamination of the surface of the sample from other waste or soil strata is likely to occur during collection. Significant contamination of the sample surfaces can lead to redistribution of the volatiles throughout the sample during shipping and storage, which will result in misleading analytical data. The reduction of surface contamination errors by trimming should be balanced with the potential losses from volatilization during trimming operations.

7.2.3 If possible, the sample should be inspected visually and its characteristics logged. Adjacent samples that appear to have similar physical properties should be retained for testing to determine or verify the relevant properties of the solid materials (for example, grain size distribution). Ideally, the sample itself or another sample of the same material should be available for inspection and notation of the following: general appearance and color, presence of oils or other visible signs of contamination, grain-size distribution, volatile organics, and so forth. In the case of samples that are

collected directly into metal rings for shipment to the laboratory such inspection may not be possible (see 7.3.3), and alternative procedures (examination of the exposed ends of the core or adjacent samples) should be used. Selection of representative samples for volatiles analysis is aided greatly by information obtained from field testing of other samples from the same stratum.

### 7.3 Granular or Uncemented Materials:

7.3.1 Granular materials may be collected from the ground surface, the walls of test pits, blocks of wastes or soils, or by using split barrel or other sampling devices during the drilling of soil borings.

7.3.2 In the case of samples collected from test pits, ground surface, or larger blocks of soils or waste, the surface of the sample should be trimmed to remove contaminants from other waste or soil strata or to remove surface layers that may have already lost volatiles. This removal of surface layers can be accomplished by scraping the surface using a clean spatula or knife.

### 7.3.3 Collection of Samples in Metal Rings:

7.3.3.1 Samples from split barrels or similar devices may be collected in precleaned metal rings inserted in the sampling barrel, such as those described in Practice D 3550. The exposed ends of the solid or waste in the ring or in adjacent samples are used to log the sampled materials. The ends of the ring containing the soil or waste are then covered quickly and sealed in the field using an inert material (for example, TFE-fluorocarbon sheets or aluminum foil with tightly wrapped sealing tape or screw-on metal caps). Extrusion of a subsample for analysis is accomplished by the analytical laboratory and does not allow for logging or field testing of interior layers of the sample by on-site personnel.

7.3.3.2 This method should not be used in the case of poor sample recovery (that is, when the metal rings are not completely filled with the material to be sampled), due to the potential losses of compounds by volatilization into the headspace of the rings.

### 7.3.4 Subsampling in the Field Using Metal Coring Cylinders:

7.3.4.1 Samples taken from test pits, ground surface, or larger blocks of soils or wastes, or samples that have been removed from down-hole sampling devices, are subsampled using a small metal coring cylinder. Removal of the subsample to be sent to the laboratory is accomplished using a cleaned metal cylinder, open at both ends, which is driven into the solid material. This method allows the field sampling personnel to inspect the surrounding solid material, log its properties, and perform field tests on the excess sample. However, this method may be impractical for certain types of solid materials that are difficult to core or extrude.

7.3.4.2 The metal coring cylinder used for subsampling should be sharpened by grinding to allow greater ease in driving the sampler. The optimum diameter of the cylinder depends on the following: size of the sampling jar, dimensions of the original sample, particle size of the solid materials (for example, gravel size particles would require larger samplers), and volume of subsample required. It is anticipated that a number of cylinders of different diameters should be available to the field personnel for selection of the optimum size.

7.3.4.3 In general, the outside diameter of the coring

cylinder should be smaller than the inside diameter of the mouth of the sampling container to avoid contamination of the outside threads of the bottle, which may result in a bad seal.

7.3.4.4 The coring cylinder containing the subsample of material for analysis can be removed by excavation (surface or test pit) or by cleaning away the excess sample with a spatula or a clean, disposable towel. The solid materials around the cylinder are used (1) to log the properties of the sample, (2) to aid in determining whether the subsample is representative of the horizon to be sampled, or (3) for additional tests (for example, grain size analysis, field testing of total volatiles, field gas chromatography). If the subsample is not extruded from the cylinder immediately, it should be sealed temporarily, by covering the ends with aluminum foil and TFE-fluorocarbon tape, and stored on ice or similarly cooled.

NOTE 1—Aluminum foil may be unsuitable in very alkaline environments.

7.3.4.5 The subsample is extruded using a cleaned rod to push the subsample out of the cylinder. The subsample is extruded directly into the sampling container. If the subsample is to be placed in a 40-mL vial, ideally, the length of subsample collected in the cylinder should be greater than the height of the vial, so that the vial can be filled in one operation. Extrusion should be performed rapidly and soon after sampling to reduce volatilization and redistribution of volatiles, which may result from contaminated subsample ends.

NOTE 2—Extrusion of cooled subsamples under controlled conditions is preferred if it can be performed on-site or within a short period of time (that is, four to six h) after sample collection.

#### 7.4 Cemented Solid Wastes:

7.4.1 *Subsampling Cemented Material by Trimming*—The solid wastes or contaminated soils may be so hard that the coring cylinder cannot be driven into the wastes. Subsamples of such wastes and soils may be collected by trimming the larger sample with a cleaned tool to a size that can be placed in the sampling jar. Although some loss of volatiles can be expected, the losses should be less than in loose, granular solids due to the lower surface area exposed to the atmosphere. Collection, trimming, and containment of the subsample should be accomplished in the least amount of time practical.

## 8. Handling

### 8.1 General:

8.1.1 In the case of materials not subsampled in the field (that is, those collected in metal rings inserted in down-hole sampling devices), the sample is retained in the metal ring and sealed as described in 6.3.3. Both the sample and the metal ring are then shipped to the laboratory, where the subsample is extruded for analysis.

8.1.2 In the case of materials subsampled in the field, glass containers with inert caps should be used for storage and shipment. To retard volatilization and biodegradation, subsamples should be placed on ice or similarly cooled as soon as practical. Two alternative methods of containing subsamples for shipment to the laboratory are outlined.

These methods have different applications, and advantages and disadvantages.

### 8.2 Method 1—Methanol Container:

8.2.1 This method can be used for a wide range of cases, but it is particularly useful for situations in which (1) larger samples are desired to obtain a composite representation of the volatiles concentration and composition, (2) high detection limits can be tolerated, or (3) biodegradation is a concern.

8.2.2 Sample containers consist of wide mouth, 8-oz. glass jars with TFE-fluorocarbon-lined lids. An aliquot of 100 mL of an appropriate analytical grade of methanol is added to the organic-free jar by the laboratory that supplies the jar, by the sample collector, or by a third party. The solid waste or soil is added to the jar containing the methanol to a specified level in the jar. This level is defined by the party responsible for preparing the sampling jars and is equivalent to the level that would correspond to the addition of approximately 100 g of the soil or waste (at an assumed specific gravity). The actual mass of material added to the jar is determined later by comparison with a tare weight, by the analytical laboratory. The jar containing the methanol should not be left open unnecessarily.

8.2.3 The volatile compounds are more soluble in the methanol than in the soil water, which results in transfer of the volatiles from the solid to the methanol for analysis. Addition of methanol to the subsample in the field allows a longer contact time with the subsample, which improves the extraction efficiency. In addition, extraction of volatiles is performed with a larger subsample than used in some methods (100 g versus 5 g with a heated He purge), which results in a more representative determination of the concentrations of the volatiles.

8.2.4 This method permits splitting of the sample into several jars, or compositing by placing several aliquots of the solid waste (from the coring cylinder described above) in one sample jar. The methanol reduces volatilization during repeated opening and closing of the jar for each subsample and serves as a medium for extracting volatiles from each subsample added to the jar. The physical mixing used in other types of analyses, with its potential for volatilization and incomplete mixing, is avoided with this method. This method also allows for multiple laboratory analyses of the same sample.

8.2.5 Since the partial pressure of the volatiles over the methanol is very low, losses by volatilization are reduced. The methanol also inhibits microbial activity, reducing losses from biodegradation.

8.2.6 The primary disadvantages of this method are (1) the need for laboratory cooperation in preparing tared sample jars, (2) possible shipping restrictions (if the methanol volume is sufficient to qualify the samples as flammable materials), and (3) the reduction of sensitivity of the gas chromatograph/Hall detector (if such detectors are used by the method).

### 8.3 Method 2—Dry Container:

8.3.1 This method involves placement of a subsample of the solid in a tared 40-mL glass container (or a size compatible with the analytical instrumentation) for shipment to the laboratory. This container is modified by the addition of a cap that allows direct connection of the

container with the purge and trap device in the laboratory, so that removal of the subsample is not required for analysis. Subsample weights are determined in the laboratory.

8.3.2 The samples are extruded into the clean, organic-free jar from the coring cylinder, ideally in one operation, to minimize opening and closing of the jar and the potential loss from volatilization. This method is preferable for cases in which the methanol method is not desired (due to shipping limitations, detection limit requirements, or laboratory restrictions) or if only small samples are available for analysis (for example, if only a thin horizon of contaminated material exists or a limited zone of contaminated material is the target for analysis).

8.3.3 The primary disadvantages of this method are (1) the requirement for specialized containers, (2) the inability to perform additional analyses of the same sample, and (3) the small size of the sample, which can reduce the representativeness of the sample.

## 9. Packaging and Package Marking

### 9.1 An indelible label identifying the sample should be

secured to the container. The label should contain or reference the following information:

- 9.1.1 Name and location of site,
- 9.1.2 Date and time of sampling,
- 9.1.3 Location of sampling,
- 9.1.4 Sample number,
- 9.1.5 Description and disposition of sample,
- 9.1.6 Name of sampling personnel,
- 9.1.7 Type of preservative, and
- 9.1.8 Sampling conditions and analytical requirements.

9.2 Pack the sample container securely in a shipping container. The sample container should be packed in ice and cooled to 4°C. A min/max thermometer should be packed with the samples.

9.3 Follow DOT (Department of Transportation)<sup>4</sup> shipping regulations.

9.4 Make arrangements for handling, logging in, adequate storage and analysis of the sample or subsample at its destination. If warranted, follow chain-of-custody protocol.

<sup>4</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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## APPENDIX 5.19-B

**DISCRETE-DEPTH SOIL VAPOR SAMPLING  
IN CONJUNCTION WITH HOLLOW-STEM AUGER DRILLING****5.19-B.1 Introduction**

This appendix describes field procedures for collecting discrete-depth soil vapor samples in conjunction with hollow-stem auger drilling during the investigation of the former solvent underground storage tanks (USTs) at SWMU 0-039. A summary of the field sampling plan, including the objectives and rationale for collecting the soil vapor samples, is presented in the RFI Addendum work plan for the site. The field procedures presented herein are intended to be of a general nature and, where necessary, appropriate modifications may be made to suit specific field conditions or project requirements.

**5.19-B.2 Sampling Equipment**

The equipment to be used for collecting discrete-depth soil vapor samples in conjunction with hollow-stem auger drilling is listed below:

- a specialized packer assembly
- threaded sections of nominal 2-inch pipe to lower the packer assembly through the hollow-stem auger
- packer inflation equipment
- Teflon or other appropriate tubing for vapor sampling
- valved ports/inlets for photoionization detector (PID) screening and gas canister sampling
- a vacuum gauge
- a valve for the vapor sampling line
- a flow meter for the vapor sampling line
- a vacuum pump suitable for vapor sampling
- two-way control valves
- gas canisters
- a PID

A schematic of the soil vapor sampling equipment is presented on Figure 5-105. Descriptions of the main components follow.

**5.19-B.2.1 Packer Assembly**

The packer assembly consists of an inflatable packer mounted on a nominal 2-inch-diameter steel or aluminum pipe with threaded ends. The deflated packer assembly must pass freely through the nominal 4-inch-inside diameter hollow-stem auger. When inflated to its maximum pressure, the packer will expand against the borehole wall, providing a seal that isolates the sample interval from the atmosphere.

A threaded cap fitted with a vapor sampling inlet is attached to the bottom of the packer assembly pipe to ensure that the vapor samples are obtained from the bottom of the borehole rather than from the inside of the packer assembly riser pipe. The threaded cap provides an air-tight seal between the cap and packer assembly pipe. On the inside of the packer assembly pipe, a tube connects between fittings at the vapor sampling inlet and through the wall of the packer assembly pipe just above the

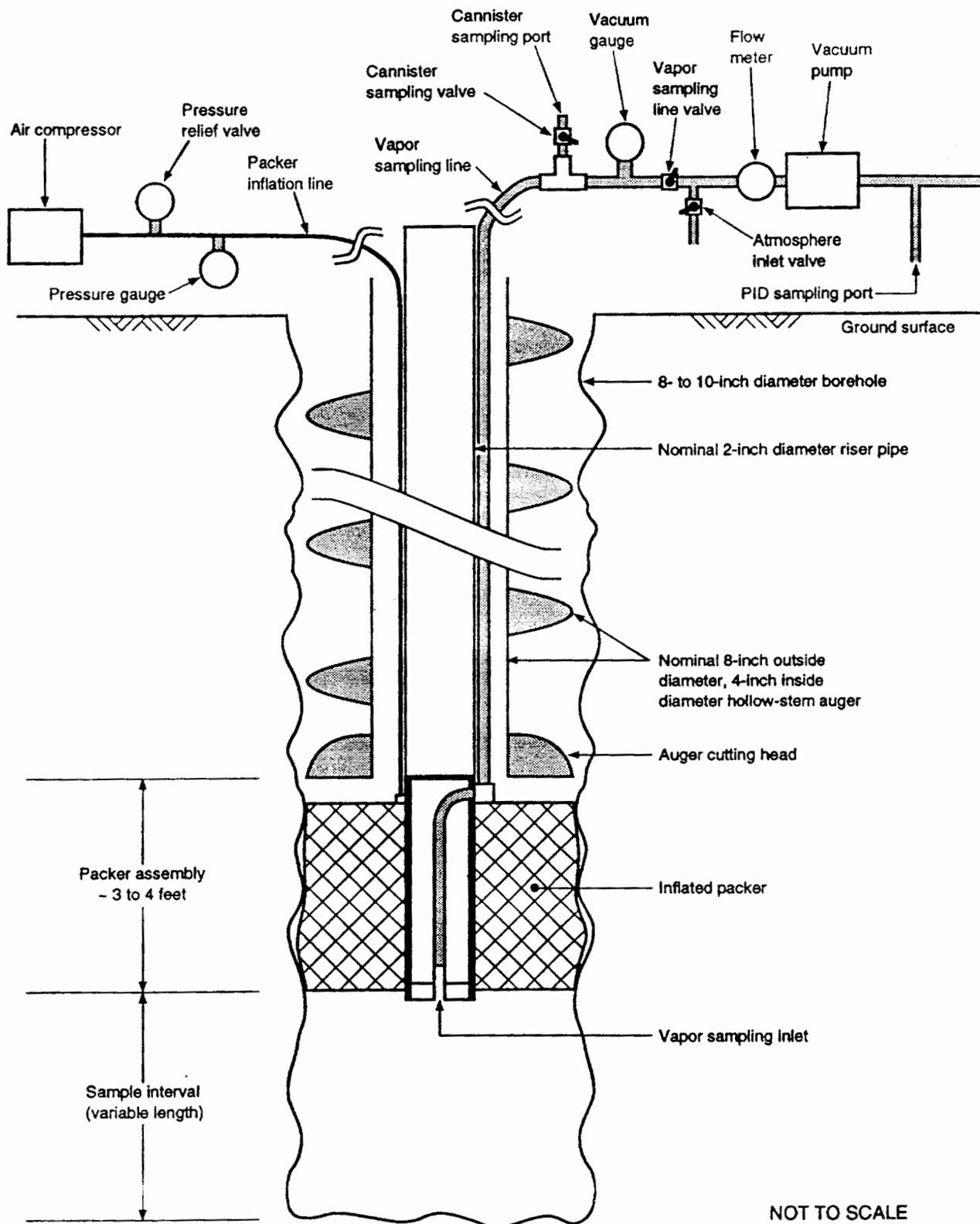


FIGURE 5-105. Schematic of soil vapor sampling equipment (SWMU 0-039).

packer. The packer assembly is threaded onto the first section of nominal 2-inch riser pipe. As the packer assembly is lowered into the hollow-stem auger, cable ties or tie-wraps are used to secure the vapor sampling and packer inflation lines to the riser pipe.

#### **5.19-B.2.2 Packer Assembly Riser Pipe**

Threaded pipe with a nominal 2-inch-diameter is used to lower the packer assembly into the hollow-stem auger. This riser pipe should be of suitable strength to lower and support the packer assembly to the required sampling depths estimated to be a maximum of 100 feet below ground surface.

#### **5.19-B.2.3 Packer Inflation Equipment**

Compressed gas (nitrogen or other appropriate gas) or an air compressor equipped with an in-line filter will be used to inflate the packer. Filtered air or inert gas are used for packer inflation to extend the life of the packer. A pressure relief valve with a tolerance just below the maximum inflation pressure will prevent damage to the packer. A pressure gauge will be used to monitor the inflation pressure.

#### **5.19-B.2.4 Sampling and Monitoring Equipment**

A vacuum pump suitable for sampling VOCs will be used to extract vapor through the sampling tube. The pump should be capable of purging the combined volume of the sampling system and borehole sample interval within approximately 10 to 20 minutes.

Teflon or other appropriate tubing that minimizes the potential for cross-contamination by adsorption will be used for the vapor sampling line. Air-tight fittings, such as swageloks, are to be used for all vapor sampling line connections.

A series of ports/inlets, valves, and gauges are fitted to the vapor sampling line. To minimize potential for cross-contamination and provide efficient operation, this equipment should be installed along the sampling line in the order shown on Figure 5-105 and described below. All fittings should be air-tight. A canister sampling port will be used to collect soil vapor samples. This sampling port should be fitted in a position on the sampling line before (upstream of) any other valves or gauges. The gas canister sampling port is followed by a vacuum gauge that is used to monitor vacuum pressures attained during purging and sampling. The vacuum gauge is followed by a sampling line valve, which is used to shut-off the sampling line or regulate the flow through the sampling line. The sampling line valve is followed by a valved atmospheric inlet, which is used to provide air to the vacuum pump when the vapor sampling line is closed during initial adjustment of the vacuum pump flow rate. A flow meter that is used to monitor flow rate and the volume extracted follows the atmospheric inlet valve. In a position after the vacuum pump (downstream), a PID sampling port is provided to screen the soil vapor concentrations in the field.

#### **5.19-B.3 Sampling Methods**

A target sample interval will be identified prior to collecting a vapor sample as described in the RFI Addendum work plan. While drilling through the sample interval, excessive rotation of the auger is avoided to minimize generation of frictional heat. After reaching the bottom of the sample interval, the auger is retracted for a distance equal to the sum of the sample interval, the inflated packer height, and at least

several additional inches to assure clearance between the packer and the auger cutting head. Field data collected during the discrete-depth soil vapor sampling program can be recorded on the field record form shown on Figure 5-106.

After cleaning the packer and sampling system, as described in Section 5.19-B.4, the packer assembly is lowered into the hollow-stem auger by adding sections of threaded riser pipe. The packer is lowered below the bottom of the auger cutting head and positioned so that after inflation the bottom of the packer is at the top of the sample interval and adequate clearance is assured between the top of the packer and the auger cutting head. After the packer is set at the target sample depth, the inflation line is attached to the air compressor and the packer is inflated to the pressure specified by the manufacturer.

The vapor sampling line valve before the vacuum pump is closed and the atmospheric inlet valve is opened. The vacuum pump is turned on and the flow rate is adjusted so that 1.5 sample interval volumes would be purged in approximately 10 to 20 minutes. For example, to purge 1.5 sample interval volumes for a 5-foot sample interval in a 10-inch-diameter boring (approximate volume =  $1.5 \times 77$  liters = 115 liters = 30 gallons), a flow rate of 7.5 liters/minute would purge 1.5 sample volumes in 15 minutes. A slow purge rate is necessary to assure that the vapor in the borehole sample interval is representative of the vapor in the surrounding tuff. The PID is attached to the PID sampling port to obtain field-screening data. The vapor sampling line valve is then opened and the atmospheric inlet valve is closed to begin purging. The PID readings, the vacuum gauge, and the flow rate of vapor entering the pump are monitored and recorded at least every 5 minutes.

After purging a volume equivalent to the approximately 1.5 times the sample interval plus the sampling system, a vacuum reading is recorded. A gas canister sample is then collected and a PID reading is recorded. The vapor sampling line valve is then closed and the vacuum pump is turned off. The gas canister will be submitted to CST-9 for fixed laboratory analysis for VOCs by EPA Method 8240 and for TPH as Stoddard solvent by Modified EPA Method 8015 to confirm the field screening results. Soil vapor samples collected as described herein should be handled in accordance with applicable Environmental Restoration (ER) Program Standard Operating Procedures (SOPs).

Before deflating the packer, the atmospheric inlet valve and the vapor sampling line valve are opened to bleed air into the sampling line and sample interval, if the vacuum gauge shows that a negative pressure still remains in the sample interval. The packer is then deflated. Deflation may require extracting air from the packer, as specified by the manufacturer, to pass the packer assembly back inside the hollow stem auger.

#### **5.19-B.4 Equipment Decontamination**

After use at each sample interval, the packer assembly is decontaminated using steam-cleaning and/or laboratory grade detergent and potable water wash, followed by a deionized-water rinse. To prevent water from entering the vapor sampling line inside the packer assembly, the vapor sampling inlet must be plugged during decontamination. The packer assembly must be dry before it can be used again.

After use at each sample interval, the vapor sample tubing and vacuum pump are cleaned by purging with ambient air for a minimum of 100 sampling system volumes. For example, 100 feet of 1/4-inch diameter tubing will require approximately 10



minutes to purge 100 volumes at a rate of 10 liters per minute. The sampling system can be purged while drilling to the next sample interval.

To confirm that the system is adequately purged, a PID reading of the system equipment (a system equipment blank reading) will be taken while pumping ambient air through the sampling system, before lowering the sampling system into the auger. If the system equipment blank is above background levels, purging will continue. If elevated PID readings persist, corrective action will be taken, such as cleaning the pump or replacing the tubing.

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