

1 system allowed for the collection of a sample that is more representative of the soil atmosphere
2 surrounding the sampling point. That is, a sample that is not does not contain an atmosphere that
3 has migrated from distance through the geologic material or from the surface in response to the
4 vacuum induced during purging soil gas concentrations. It is, thus, recommended that when soil
5 gas sampling is conducted using temporary probes in fine-grained soils, that the sampling system
6 uses the smallest practical ID soil gas tubing and minimize purge volume to obtain the soil gas
7 sample with minimal risk of leakage so that proper decisions, based on more representative soil
8 gas concentrations, about the site can be made.

9

10 **Introduction**

11 Soil gas data are widely used in site investigation and remediation projects to delineate
12 volatile organic compound (VOC) vapor plumes, as a screening tool to refine soil and
13 groundwater sampling efforts, to track the progress of soil remediation, and to identify potential
14 risks from the inhalation of indoor air associated with soil vapor migration and intrusion into
15 buildings and homes (EPA 2007). The overall goal of any monitoring or sampling program is to
16 collect representative samples; that is, samples that are representative of the environmental,
17 chemical, and physical conditions present during the time of sample collection. However, due to
18 numerous environmental, as well as sampling and analytical variables, the representativeness of
19 a sample can often be compromised, the degree to which is often not well understood or
20 quantified. The definition of a representative sample is also dependant upon the data quality
21 objectives of the sampling effort. In some cases, a larger sample and corresponding larger
22 sampling area is desired to account for heterogeneity in the subsurface. In other cases, a discrete

1 sample that best characterizes the VOC concentrations at that sampling point and depth is
2 desired.

3 A number of research groups and local, state, and federal agencies have developed
4 guidance concerning sampling and analytical protocols for active soil vapor sampling with the
5 overall objective of facilitating a technically correct approach for site investigations (EPA 2007).
6 Guidance documents from a variety of sources including the: American Petroleum Institute (API
7 2005); American Society for Testing and Materials (ASTM 2001); California Environmental
8 Protection Agency, Department of Toxic Substances Control and California Regional Water
9 Quality Control Board, Los Angeles Region (CaEPA 2003); Interstate Technology and
10 Regulatory Council (ITRC 2007); Missouri Department of Natural Resources (MoDNR 2005);
11 New Jersey Department of Environmental Protection (NJDEP 2005); New York State
12 Department of Health (NYSDH 2005); and the U.S. Environmental Protection Agency (EPA
13 2006), all generally recommend that purge volume be minimized to increase the likelihood that
14 the collected sample is representative of conditions immediately surrounding the sampling probe
15 and to reduce the potential of short-circuiting (i.e., the intrusion of surface air into the sample via
16 channeling down the probe tube) the sampling system. However, few of the documents provide
17 specific recommendations for purge volumes. CaEPA (2003) guidance stipulates that a step
18 purge test be conducted by collecting samples after one, three, and seven line volumes have been
19 purged. MoDNR (2005) and NJDEP (2005) recommend three line volumes be purged prior to
20 sampling while NYSDH (2005) recommends one to three line volumes.

21 The system volume refers to the total volume of gas purged prior to sample collection
22 (EPA 2007). Most soil vapor sampling protocols require the purging of a system volume
23 consisting of multiple line volumes prior to collection of the final sample for analysis. However,

1 during purging, the ambient soil atmosphere in the immediate vicinity of the probe tip, which is
2 in theoretical equilibrium with the all soil fractions (e.g., air, water, mineral and organic matter),
3 is removed. The resultant soil gas collected for analysis migrated from the geologic material
4 immediately surrounding the probe tip in response to the vacuum induced during purging, unless
5 the geologic material has a lower permeability than the annulus between the probe and the
6 geologic materials, in which case, there may be short circuiting with soil gas flowing from the
7 next shallower higher permeability geologic interval. There may also be complexities in
8 fractured media or where soil macropores act as a preferential flow pathway, if purge volumes
9 are very large.

10 Soil gas sampling protocols also recognize that large system volumes in sampling trains
11 require correspondingly large purge volumes (EPA 2007). As the purge volumes increase,
12 associated with larger sampling tube diameters and deeper sampling depths, more and more of
13 the ambient soil atmosphere is lost prior to sample collection and the size of the recharge zone
14 increases. The resultant analytical samples may represent the “average” conditions within the
15 larger purged area; however, the area of influence is not known. In areas with very little
16 localized vapor phase VOCs or relatively tight soils (i.e., high clay content soils) using
17 temporary probes, there is a greater chance of short-circuiting to shallower more permeable
18 intervals, such that an analytical false negative can result. Conversely, limiting system volume
19 purging requirements increases the likelihood that the sample is representative of a discrete,
20 limited volume immediately adjacent to the sampling location that is representative of the
21 ambient VOC concentrations found at the location prior to purging. This paper describes the
22 influence of system volume removal during purging, as affected by sampling tube diameters and

1 sampling depth, on the concentrations of VOCs found at several contaminated waste sites with
2 silt and clay-rich soils using temporary driven probes.

3

4 **Materials and Methods**

5

6 **Sampling Locations**

7 Three sites contaminated with volatile organic compounds were selected to test the
8 macro- and micro-purge soil gas sampling systems. These sites were located at Mare Island
9 Naval Shipyard (MINS) in Vallejo, CA; Grants Chlorinated Solvents Plume (GCSP) in Grants,
10 NM; and Vandenberg Air Force Base (VAFB) Installation Restoration Project Area 3, near
11 Lompoc, CA. At each site, multiple locations were sampled. The distance between each pair of
12 macro- and micro-purge system sampling points was approximately 30 cm.

13

14 **Probe Design**

15 The design of the macro-purge sampling system remained the same at all three sites while
16 the design of the micro-purge sampling system was the same at the MINS and GCSP sites and
17 altered, to allow for greater depth of sampling, at the VAFB site. Although not specifically
18 designed for use in fine-grained soils, the Post-Run Tubing (PRT) system by Geoprobe®
19 (Geoprobe, 2003) was used for the macro-purge soil-vapor sampling system. A hollow 3.2 cm
20 OD drive probe with a retractable sampling point was inserted to the appropriate depth (1m
21 below ground surface [bgs] at MINS and GCSP and between 2.4 and 3.7 m bgs at VAFB). The
22 drive probes were then raised approximately 2.5 – 5.0 cm to create a void in the subsurface. A
23 section of FEP Teflon® tubing (0.49 cm ID x 0.64 cm OD) was attached to a threaded PRT

1 adapter, inserted down the hollow probe tube and threaded into the retractable probe tip housing.
2 The other end of the Teflon® tubing was attached to a septum port constructed from a 0.6 cm
3 Swagelok® elbow fitting through which a syringe was inserted to collect a sample. The internal
4 volume of the macro-system at 1m bgs was calculated as 18.1 mL for the tubing, plus 6.4 mL to
5 account for the internal volume of the retractable drive-point, for a total internal volume of 24.5
6 mL.

7 To collect the micro-purge vapor samples at the MINS and GCSP sites, the soil-vapor
8 sampling system designed by Hewitt (1998) was used (Figure 1). The probe design consisted of
9 a small-diameter stainless steel tubing (0.16 cm OD and 0.10 cm ID) epoxied into a groove in a
10 1.2 m steel drive rod. The upper end of the stainless steel tubing extending out the top of the
11 probe and was attached to a Swagelok® septum port. On the bottom end of the probe, a
12 disposable, conical, brass drive point was fitted onto the end of the sampling tube to assist in
13 probe placement and to prevent soil from clogging the stainless steel tubing. After the probe was
14 driven to a 1 m depth, the probe was raised approximately 2.5 cm to create a void in the
15 subsurface. The internal volume of the sampler was approximately 1.2 mL at a 1m sampling
16 depth.

17 To allow for sampling at greater depths, such as found at the VAFB site, a variation of
18 the Hewitt micro-volume system was necessary. The modified probe design (Figure 2) consisted
19 of a small-diameter stainless steel tubing (0.0254 cm ID) epoxied into a Geoprobe® PRT point
20 holder such that the open end of the steel tubing (Sigma Aldrich® part number 20552) was
21 approximately 2.8 cm below mouth of the point holder (i.e., the depth necessary for the insertion
22 of the expendable drive point). For sampling, the stainless steel tubing was run through the
23 hollow shaft of the probe rod during insertion and then capped with a low dead volume

1 Swagelok[®] union with Teflon[®] lined septa. After the probe was driven to the appropriate depth,
2 the probe was raised approximately 2.5 cm to create a void in the subsurface. The internal
3 volume of the sampler was approximately 7.05 mL at 1m bgs, 0.05 mL for the stainless steel
4 tubing and 7.0 mL the internal volume of the expendable point holder.

6 **Sample Collection**

7 At each site, soil vapor samples were collected after purging 3 system volumes. A gas-
8 tight glass syringe with on/off valve (SGE 8970 for 10 mL and SGE 9770 for 100 mL) was used
9 to collect a volume equal to one purge system volume for analysis. To collect the samples, the
10 syringe plungers were to drawn back at a rate to minimize the creation of a vacuum. At the
11 VAFB site, after soil vapor samples were collected, the on/off valve was closed, the end of the
12 syringe needle capped via insertion into a septa, and taken directly to an on-site mobile
13 laboratory for analysis.

14 At the MINS and GCSP sites, the collected gas samples were transferred to thermal
15 desorption tubes (TD) tubes (Pankow et al. 1998). The TD tubes were stainless steel, 8.9 cm
16 long by 0.64 cm diameter, sealed at each end with brass Swagelok[®] endcaps fitted with Teflon[®]
17 ferrules. Each TD tube contained 180 mg Carbotrap B on the inlet side, followed by 70 mg
18 Carboxen 1000 (Supelco Inc., Bellefonte, PA). During sampling, the end caps were removed
19 and an on/off valve, with a septum port, was attached to the front end of the TD tube and a
20 constant flow air sampling pump (Sensidyne, Clearwater, FL) on the tail end. The pump
21 maintained a constant flow of 50 mL/min of filtered ambient air through the TD tube. To
22 transfer the sample, with the pump running, the valve was opened and the sample injected near
23 the front of the TD tube. The air flow through the tube was maintained for a minimum of 15 min

1 before the valve was closed. The TD tubes were capped and stored chilled (4° C) prior to
2 analysis.

3 Bulk soil samples were collected at the MINS and GCSP sites using a 60 mm diameter
4 core sampler (Geoprobe® RS60) driven to the appropriate depth. The core tube was then brought
5 to the surface and an 8 cm segment, containing the sampling depth, was collected for the
6 determination of particle-size distribution and total organic carbon content (TOC).

7 8 **Sample Analysis**

9 Soil vapor samples were analyzed using a modified version of EPA Method TO-17 (EPA
10 1997a) and quantified using the gas chromatography/mass spectrometry technique described in
11 SW-846 Method 8260B (EPA 1997b) at the MINS and GCSP sites. At the VAFB site, soil
12 vapor samples were analyzed by direct injection using EPA SW-846 method 8021B (EPA 1996).

13 Soil particle-size analyses were determined using the hydrometer method as specified in
14 Gee and Bauder (1986). TOC determinations were performed following the high-temperature
15 induction furnace method of Nelson and Sommers (1996).

16 17 **Quality Assurance/Quality Control**

18 Quality assurance/quality control (QA/QC) measures included trip and field blanks;
19 spiked samples; field duplicate samples (sequential collection of a second system volume after
20 purging); and initial and ongoing calibration checks. All field and laboratory quality control
21 sample results were compliant with the project specific data quality objectives. Leak testing was
22 performed at the VAFB site using a rag saturated with 1,1-difluoroethane (DFA), placed over the
23 surface completion of the probe. No DFA was detected in any of the samples associated with the

1 leak checks indicating no short circuiting to the surface although cross communication between
2 different depth intervals below the surface could still be possible.

3

4 **Results and Discussions**

5

6 Mare Island Naval Shipyard

7 The soil at the MINS site had a silt loam texture and a TOC of 1.5% (Table 1). VOCs
8 identified at the MINS site included toluene, ethylbenzene, and xylenes (Table 2 and Figure 3).
9 At both locations, the relative order of abundance of the contaminants was the same between the
10 micro-purge and macro-purge systems. When the contaminants were identified in both the
11 micro-purge and macro-purge systems, at location 1, the concentrations were about 5 times
12 greater in the micro-purge samples than the macro-purge samples while at location 2, the
13 concentrations were between 15 and 22 times greater in the micro-purge samples than the macro-
14 purge samples.

15

16 Grants Chlorinated Solvent Plume

17 The soil at the GCSP site had a silt loam texture and a TOC of 0.4% (Table 1). VOCs
18 identified at the GCSP site included tetrachloroethene (PCE), trichloroethene (TCE), *cis*-1,2-
19 dichloroethene (DCE), toluene, ethylbenzene, and xylenes (Table 2 and Figure 3). PCE was the
20 dominant contaminant and had a concentration of 27.8 µg/L when collected using the micro-
21 purge system and 3.1 µg/L when collected using the macro-purge system. When the
22 contaminants were identified in both micro-purge and macro-purge samples, concentrations were
23 markedly higher in the micro-purge samples. The concentration differences ranged from the

1 same for o-xylene to 23 times higher for m, p-xylenes in the micro-purge samples than the
2 macro-purge samples. All contaminants found in the macro-purge samples were identified in the
3 micro-purge samples. Conversely, toluene was not identified in the macro-purge samples yet
4 was identified in the micro-purge samples. Possible explanations for the lack of toluene in the
5 macro-purge samples include analytical loss/error or loss during purging combined with the
6 subsequent lack of recharge from the surrounding soil.

7

8 Vandenberg Air Force Base

9 The soil at the VAFB site had a clayey sand texture (G. Swanson, personal
10 communication, July 21, 2008). The primary contaminant identified at all three locations on
11 VAFB was TCE. DCE was found only at locations 2 and 3. TCE concentrations ranged from
12 270 to 1500 µg/L in the micro-purge samples (Table 2) and from 26 to 120 µg/L in the macro-
13 purge samples. When identified, the concentrations of DCE were low relative to the
14 concentrations of TCE. At location 2, DCE was found at 2.5 and 1.3 µg/L in the micro-purge
15 and macro-purge samples, respectively. Similarly, at location 3, the concentrations of DCE
16 found in the micro-purge and macro-purge samples were 12 and 2.6 µg/L, respectively. The
17 TCE concentrations were 9 to 27 times greater in the micro-purge samples than the macro-purge
18 samples system (Figure 3). When DCE was identified, the micro-purge sample contained about
19 2 to 5 times as much as the macro-purge sample.

20

21 **Conclusions**

22 At each of the sites and each location within the sites, although there were variations in
23 texture and total organic carbon contents, the same contaminants (with few exceptions) were

1 identified using either the micro-purge or macro-purge temporary probe systems. In general, the
2 relative abundances of the identified contaminants were identical until VOC concentrations
3 approached the detection limits. When the relative abundances orders were different between the
4 two sampling systems, it was generally the case where the contaminant was identified with the
5 micro-purge sampling system and not detected with the macro-purge sampling system. These
6 results indicate that the same soil atmosphere was being collected by both sampling systems.
7 However, when a larger purge volume was required, the larger purge volumes may have led to
8 the loss of any detectable levels of toluene as these contaminants were identified only when
9 using the micro-purge sampling system at the GCSP site. Macro-purge sample volumes were
10 about 20 times greater at the MINS and GCSP sites and 6 to 10 times greater at the VAFB site
11 than the micro-probe sample volumes collected at the same depths.

12 The concept that the larger purge volumes required when using a macro-purge sampling
13 system led to a greater loss and subsequent reduction in contaminant concentrations is supported
14 by the markedly higher concentrations of each contaminant found in the micro-purge samples.
15 Concentrations ranged from the same to 27 times greater in the micro-purge samples than their
16 corresponding macro-purge samples. The higher concentrations are the result of a minimal
17 disturbance of the ambient soil atmosphere during purging or less cross-communication along
18 the outer annulus of the drive casing. The minimal disturbance allows for the collection of a
19 sample that is more representative of the soil atmosphere surrounding the sampling point and a
20 sample that does not contain an atmosphere that has migrated from distance through the geologic
21 material or from the surface in response to the vacuum induced during purging soil gas
22 concentrations. It is thus, recommended that when soil gas sampling is performed using
23 temporary probes in fine-grained soils, that the sampling system uses the smallest practical ID

1 soil gas tubing and minimize the purge volume to obtain a final soil gas sample that has minimal
2 risks of atmospheric air leakage so that proper decisions, based on more representative soil gas
3 concentrations, about the site can be made. Otherwise, probes should be installed with sand-
4 packs around the screened interval and bentonite slurry above the sandpack through the annulus
5 between the probe and the borehole wall.

6

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16

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23 recommendation for use.

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7 Agency, Washington, D.C.

8

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1

SITE	% Sand	% Silt	% Clay	% TOC
MINS[†]	15	78	7	1.5
GCSP[†]	13	79	8	0.4

Note: MINS = Mare Island Navel Shipyard, GCSP = Grants Chlorinated Solvent Plume.
† - n = 3-5 replicates per site.

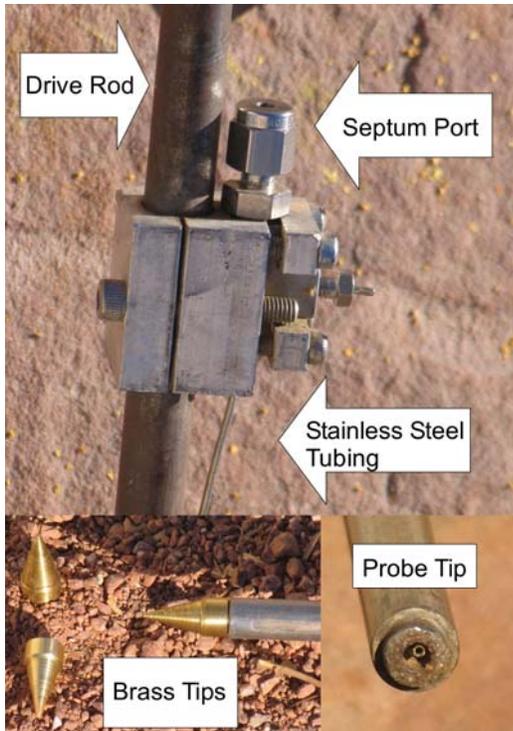
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Table 2. Sample Data for the MINS, GCSP, and VAFB sites.

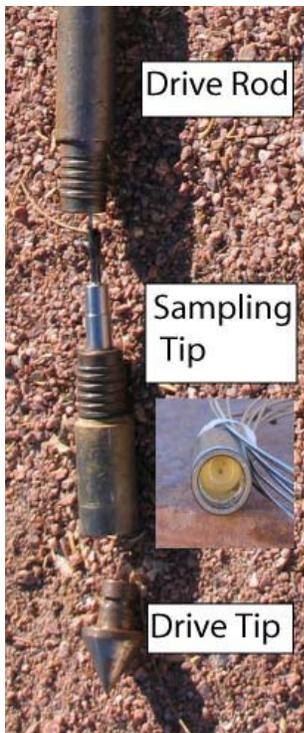
Sample ID	cis-12-dichloroethene	"Trichloroethylene	Toluene	Tetrachloroethylene	Ethylbenzene	m,p-Xylene	o-Xylene
MINS1I	ND	ND	5.0	ND	ND	1.5	0.78
MINS2I	ND	ND	3.4	ND	0.70	2.4	1.1
GCSP4I	0.80	0.42	1.4	27.8	0.23	13	0.44
VAFB1I	ND	270	ND	ND	ND	ND	ND
VAFB2I	2.5	700	ND	ND	ND	ND	ND
VAFB3I	12	1500	ND	ND	ND	ND	ND
MINS1A	ND	ND	0.68	ND	0.11	0.44	0.15
MINS2A	ND	ND	0.23	ND	0.041	0.14	0.05
GCSP4A	0.44	0.044	ND	3.1	0.10	0.56	0.44
VAFB1A	ND	29	ND	ND	ND	ND	ND
VAFB2A	1.3	26	ND	ND	ND	ND	ND
VAFB3A	2.6	120	ND	ND	ND	ND	ND

4 **Note:** MINS = Mare Island Naval Shipyard, GCSP = Grants Chlorinated Solvent Plume, VAFB = Vandenberg Air Force Base, ND = non-detect.



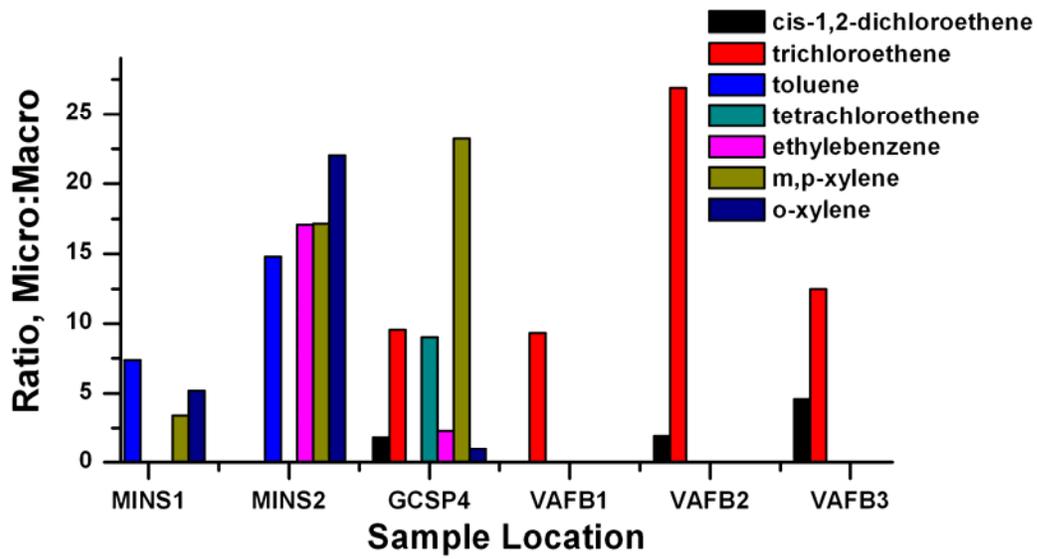
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Figure 1. MINS and GCSP Micro-Purge Sampling System



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Figure 2. VAFB Micro-Purge Sampling System



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Figure 3. Ratios of VOC concentrations between micro- and macro-purge sampling systems. (Note: Ratios only shown when VOC identified by both systems.)