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The Effect of Equilibration Time and Tubing Material on Soil Gas Measurements

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ABSTRACT

The collection of soil vapor samples representative of *in-situ* conditions presents challenges associated with the unavoidable disturbance of the subsurface and potential losses to the atmosphere. This article evaluates the effects of two variables that influence the concentration of volatile organic compounds in soil vapor samples: equilibration time and tubing material. The time for three types of soil vapor probes (i.e., macro-purge, mini-purge, and post-run tubing probes [PRT]) to equilibrate with subsurface conditions was assessed by installing probes and collecting multiple samples over a 72-hour period. The effect of tubing material was evaluated by collocating soil vapor probes constructed with different tubing material and collecting samples over several months. We recommend that soil vapor probes constructed with a sand filter-pack and bentonite seal (i.e., macro-purge probe) equilibrate for 24 to 48 hours prior to sample collection. Post-run tubing (PRT) probes equilibrated within one to two hours while a new probe design, (i.e., mini-purge probe) equilibrated and could be sampled after only 30 minutes for screening assessments. Nylaflow[®], Teflon[®], polyetheretherketone (PEEK), and stainless-steel tubing had comparable trichloroethene (TCE) concentrations over all sampling time frames. We recommend that copper tubing be avoided and polyethylene only be used for screening assessments.

KEYWORDS

Soil gas; equilibration time; tubing type; trichloroethylene; TCE; volatile organic compounds

Introduction

Soil vapor data are widely used in site investigation and remediation projects to delineate volatile organic compound (VOC) vapor plumes (Cal/EPA, 2012; AZDEQ, 2011), as a screening tool to refine soil and groundwater sampling efforts, to track the progress of soil remediation, and to assess the vapor intrusion pathway (AZDEQ, 2011; MDNR, 2013). A critical facet of any sampling program is the collection of representative data; that is, data that accurately reflect the *in-situ* conditions of the media being sampled. The collection of environmental samples necessarily disturbs the sampled media, so care must be taken to reduce the disturbance to the extent possible (AZDEQ, 2011; Cal/EPA, 2012).

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The collection of vadose zone soil vapor samples requires that a sampling tool be advanced to the target sample depth, typically using direct-push drilling equipment (e.g., a GeoProbe[®] rig), or less commonly with other drilling methodologies, such as hollow-stem augers, hand-augers, or sonic drilling. All of these techniques result in some disturbance to the subsurface. After advancing the drill rod to the target sampling depth, the drill rod is then withdrawn a small amount to create a void space and vapor samples can be collected through the drill rod (e.g., “post-run-tubing”). Semi-permanent vapor probes can be constructed in the borehole after completely removing the drill rod, which further disturbs the subsurface and exposes the vapor column to the atmosphere. Therefore, it is important to know how long the vapor probe must be allowed to equilibrate in the subsurface in order to obtain a representative sample.

Various agencies have provided recommendations on equilibration time, including: the California Environmental Protection Agency (Cal/EPA): two hours for probes installed using direct push drilling; American Society for Testing and Materials (ASTM): 30 minutes for probes installed using direct-push and two hours for probes installed using hollow-stem auger; Interstate Technology & Regulatory Council (ITRC): “a few hours”; and the New York State Department of Health: 24 hours after the installation of permanent probes and shortly after the installation of temporary probes (Cal/EPA, 2012; ASTM, 2011; ITRC, 2007; NYDOH, 2006). However, little empirical data are available to support these recommendations.

A further consideration during the collection of soil vapor samples is the impact of the sampling apparatus on the measurement. Most soil vapor sampling systems consist of a length of tubing that is either open or connected to a permeable tip at the target sampling depth, and a surface termination that facilitates the extraction of vapor. The largest surface area that the vapors come into contact with during sampling is the tubing interior and the vapor may interact with the tubing material during sample collection.

The objective of this investigation was to assess the time required following installation for vapor probes to equilibrate with the *in-situ* environment and yield representative samples (i.e., the equilibration time) and to compare the performance of an assortment of tubing materials commonly used during soil vapor sample collection.

Experimental design

To achieve the objectives of this study, a number of soil vapor probes were installed at the study site and were sampled at set time intervals until the measured VOC concentrations had stabilized. In addition, a set of vapor probes constructed with a variety of tubing materials were installed at the same depth in the same borehole and sampled to evaluate the differences in measured VOC concentrations due to tubing material. An initial field investigation and a second follow-up field investigation to repeat the experiments and test the mini-purge and post-run tubing (PRT) probe types were conducted.

Study site

The study was conducted at Installation Restoration Program (IRP) Site 14, located on Naval Air Station (NAS) Lemoore, located in the California Central Valley, approximately 40 miles

south of Fresno and 180 miles north of Los Angeles, California (US EPA, 2009). Chlorinated VOCs, predominantly trichloroethene (TCE), are the primary contaminants that have been found in soil, soil gas, and groundwater at IRP Site 14.

Geologic deposits beneath IRP Site 14 consist of an alluvial aquifer composed of sand, silty sand, and sandy silt interfingered with less permeable deposits of clayey silt and silty clay. The alluvial assemblage is interrupted by clay interbeds of lacustrine origin at various intervals.

Several partially connected groundwater zones are present beneath IRP Site 14. The shallow uppermost groundwater zone is designated as the A zone aquifer. The A-Clay underlies the A-zone aquifer at a depth of approximately 45 to 50 feet, forming a semi-impermeable barrier that the A-zone groundwater is perched on. The depth to A zone groundwater ranges from 10 to 14 feet bgs.

Alluvium in the A zone aquifer (~12 to 45 feet bgs) consists largely of granular alluvium (predominantly sands). The vadose zone soils consisted predominantly of a homogeneous mixture of silts and clays. Limited soil physical property data for the vadose zone and A-zone aquifer soils at Site 14 are presented in Table 1. Trichloroethene is the primary chemical of concern in groundwater. Concentrations in excess of 1,000 micrograms per liter ($\mu\text{g/L}$) have been measured in groundwater samples from Site 14; however, concentrations directly beneath the study site are less than 100 $\mu\text{g/L}$ (US EPA, 2009).

Vapor probes

Three types of vapor probes were evaluated for this study. They are referred to here as macro-purge probes, mini-purge probes, and post-run tubing (PRT) probes, as detailed in the following. All of the probes were installed at depths of 2.1 or 3 meters (7 or 10 feet) bgs in the vadose zone soils.

Macro-purge probes

Macro-purge probes consisted of a length of Nylaflo tubing attached to a 2.5-cm-long, gas-permeable probe tip, buried in sand filter pack and sealed with hydrated bentonite (Figure 1).

Table 1. Typical physical properties of the vadose zone and A-zone aquifer.

PARAMETER	RESULT	
	Vadose zone	A-zone aquifer
Clay (%)	—	4.2 – 6.1
Dry Bulk Density (g/cm^3)	—	1.53 – 1.54
Bulk Density (g/cm^3)	—	1.82
Moisture Content (%)	12 – 37	18.3 – 18.8
Fraction Organic Carbon (%)	0.28 – 0.48	0.80 – 0.90
Percent Gravel (%)	—	0.0 – 0.6
Percent Sand (%)	—	73 – 80
Percent Silt or Percent Clay (%)	—	19.7 – 27.1
Porosity, Effective	—	0.15 – 0.18
Porosity, Total	0.45 – 0.60	0.40 – 0.41
Permeability, Effective (millidarcy)	4.3 – 3.7	—
USCS Classification (field)	clay and silt	medium sand
Geotechnical Analysis Classification	—	silty sand

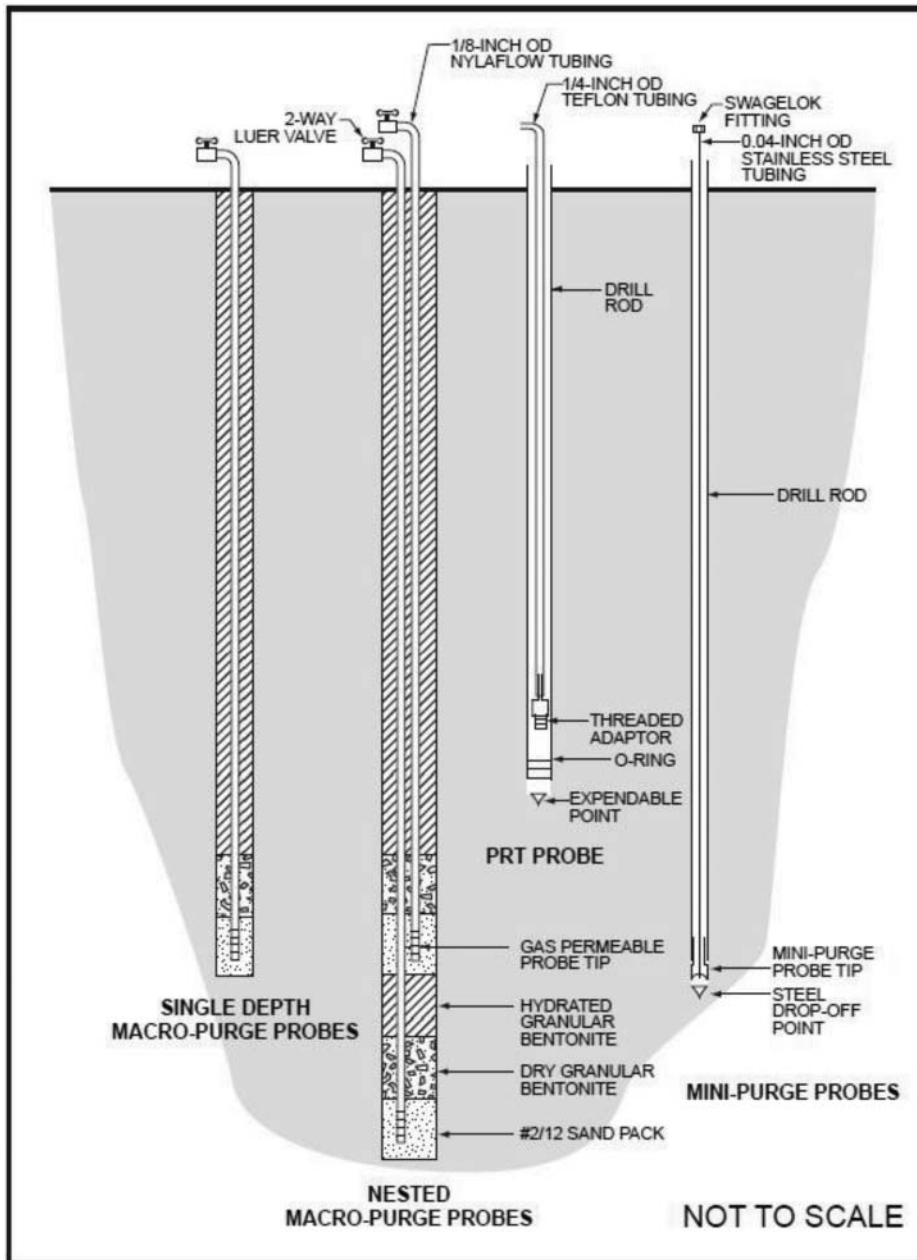


Figure 1. Schematic diagram of macro-purge, PRT, and mini-purge probes.

These are industry-standard probes often referred to as semi-permanent probes or vapor implants. The macro-purge probes were installed in pilot holes advanced using a direct-push drilling rig equipped with 3.2-cm-diameter probe rods. The probes were installed as follows. Approximately 7.6 to 15.2 cm of #2/12 sand was poured into the bottom of the pilot holes. The probe tip, attached to 0.3-cm-diameter Nylaflo tubing, was lowered into the

Table 2. Macro-purge vapor probe installation details.

Probe ID ¹	Installation Date	Installation Type	Probe Depth (m bgs)	Length of Sand Pack (cm)
A-SV-7	15 July 2009	Single	2.1	30.5
A-SV-10	15 July 2009	Single	3	30.5
A-SVN-7	15 July 2009	Nested	2.1	30.5
A-SVN-10	15 July 2009	Nested	3	30.5
B-SV-7	13 July 2009	Single	2.1	30.5
B-SV-10	13 July 2009	Single	3	30.5
C-SV-7	14 July 2009	Single	2.1	30.5
C-SV-10	14 July 2009	Single	3	30.52
C-SVN-7	14 July 2009	Nested	2.1	30.52
C-SVN-10	14 July 2009	Nested	3	30.5
ST3-NF-7	19 May 2010	Single	2.1	15.2
ST3-NF-10	18 May 2010	Single	3	15.2

Note: bgs = below ground surface.

pilot hole to the top of the sand. Additional sand was then poured into the pilot hole, filling the annulus between the hole and the tubing until it extended 7.6 to 15.2 cm above the probe tip to form a 15.2- to 30.5-cm-long sand pack. Approximately 30 cm of dry bentonite was then placed on top of the sand pack, followed by hydrated bentonite to approximately 15.2 cm below the next sampling depth or to the surface. For nested probes, this process was repeated at the next sampling depth. The sampling probes were completed at the surface with approximately 18 inches of tubing extending out of the ground and a two-way Luer valve attached to the end of the tubing. Relevant details of the macro-purge probe installations are provided in [Table 2](#).

Mini-purge probes

The mini-purge probe consists of a 0.1-cm-diameter stainless-steel tubing epoxied into a 3.2-cm-diameter steel probe tip, which was, in turn, threaded into the end of a 3.2-cm-diameter direct push drill rod ([Figure 1](#)). The end of the probe rod is equipped with a stainless-steel drop-off point. The probe rod, with the stainless-steel tubing running through the center, was advanced to the target sampling depth, and the rod was withdrawn approximately 2.5 cm to expose the tubing to the subsurface vapors. The mini-purge sampling was performed to evaluate a sampling procedure that only requires a very small volume of soil gas to be purged and withdrawn during sampling and, thus, the collected sample would be more representative of the immediate subsurface environment. The probe identification numbers for mini-purge probes consist of a location number, followed by “-MIN-,” followed by the probe depth in feet (e.g., probe ST9-MIN-10 is at location ST9 and installed at 10 feet bgs).

Post-run tubing probes

The PRT probe was developed by Geoprobe Systems[®] (Salina, Kansas) and is widely used in soil vapor investigations. PRT probes were constructed by driving a direct-push drill rod equipped with a reverse-threaded adapter and drop-off point to the target sampling depth, threading 0.64-cm-diameter Teflon[®] tubing into the bottom of the drill rod, and pulling the rod back up by approximately 7.6 cm. The end of the tubing is equipped with an O-ring, which seals against the adaptor at the bottom of the drill rod, ensuring that vapors drawn

through the tubing come from the target sampling depth. Probe identification numbers for PRT probes are like the mini-purge probes, but with “-PRT-.”

Tubing material cluster probes

The tubing material cluster probes consisted of a collocated bundle of six different tubing materials, each with a gas-permeable tip, installed in the same manner as macro-purge probes, and measured six times during an 18-month time span. The tubing materials tested were stainless steel, copper, polyetheretherketone (PEEK), Teflon[®], Nylaflo, and polyethylene. All of the tubing materials were 0.32 cm diameter with the exception of the polyethylene, which was only available in 0.64 cm diameter. The tubing material cluster probes were installed in a single borehole at a depth of approximately 1.8 m bgs with a 45.7-cm-long sand filter pack.

Sample collection

Sample collection consisted of purging a vapor probe with a syringe to remove any stagnant/ambient air, followed by collection of a vapor sample. Prior to collection of the first sample from each macro-purge probe, a volume of gas equal to three times the void volume of the sand filter pack plus the tubing and probe tip was purged. Macro-purge and PRT probes were purged and sampled at a rate of approximately 200 milliliters per minute (mL/min), while mini-purge probes were purged and sampled at approximately 5 mL/min (US EPA, 2007). For all probe types, a volume of gas equal to three times the volume of the tubing and probe tip was purged from each probe prior to collection of every sample. Samples were collected in glass airtight syringes and immediately analyzed on-site. The sample volume from macro-purge and PRT probes was 20 mL and from mini-purge probes was 5 mL.

The minimum sampling objective was to sample each probe at 17 and 30 minutes after installation as well as 1, 2, 4, 8, 24, 36, and 72 hours after installation or until equilibrium was attained. Equilibrium was initially defined as two or more successive measurements from the same probe having a relative percent difference (RPD) of less than 25. However, a majority of measurements met the 25% criterion, even as the measured concentrations were steadily increasing with time. Therefore, plots of the measured concentrations were made as the samples were analyzed, and equilibrium was identified by a plateauing of the concentration curves.

Sample analysis

Soil gas samples were analyzed on-site using a mobile laboratory operated by H&P Mobile Geochemistry/Hartman Environmental Geoscience (Carlsbad, CA). Analyses were conducted using a modified version of EPA SW-846 Method 8021 (EPA, 1996). Method 8021 was modified to use an electron capture detector instead of the electrolytic conductivity detector. This method is faster, more sensitive, and has a larger linear dynamic operating range than gas chromatography/mass spectrometry (GC/MS) methods. Other modifications to Method 8021 consisted of the absence of matrix spike samples and surrogates and the use of a four-point instead of five-point calibration curve. The analytical instrument was an SRI 8610 gas chromatograph equipped with a 30-meter DB-61 megabore capillary column operated at a temperature of 80°C. Nitrogen was used as the carrier flow gas at a rate of 10 mL/min. The detection limit for TCE was 5 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$). With this

analytical arrangement, the run time for each sample was approximately five minutes, which permitted high-frequency on-site analysis and the collection of numerous data points for each vapor probe.

A “final calculated concentration” for each probe was calculated as the mean concentration from the last three samples collected at each probe. The final calculated concentration is a relative term used to represent the TCE concentration at which a steady state (i.e., no more potential influence from probe installation) has been reached. Results for each sample are shown as the concentration of TCE detected and the percent of the final calculated concentration.

Temporal control

The temporal control was a macro-purge probe located adjacent to the equilibration study probes that had previously been installed at 10 feet bgs. The temporal control probe was periodically sampled and analyzed during the initial field investigation. The concentrations of TCE in samples from the temporal control probe were stable with relative percent differences between successive measurements ranging from 0 to 25% (Figure 2). These data indicate that while there is some limited temporal variability in measured TCE concentrations, it is random and does not account for the consistent trends observed in the equilibration study results. Due to the negligible temporal variability observed during the initial field investigation, the temporal control sampling was not repeated during the final field sampling.

Mobile laboratory quality control

All of the mobile laboratory quality control (QC) sample results were compliant with the QAPP-specified criteria (TetraTech, 2008). Five laboratory blanks were analyzed and all results were non-detect. All of the initial calibrations passed the QAPP specified criteria of a minimum correlation coefficient 0.990 or a percent relative standard deviation of $\pm 20\%$. The daily calibration verifications passed the QAPP specified criteria of $\pm 20\%$ difference from the average response factor determined from the calibration. These results confirmed that the soil gas results were within the acceptance criteria and that the data set was of high quality and suitable for its intended use (Tetra Tech, 2010a; 2010b).

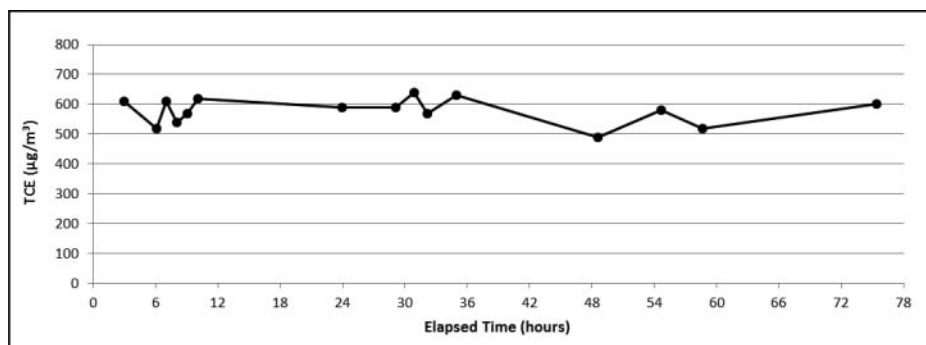


Figure 2. TCE in temporal control probe.

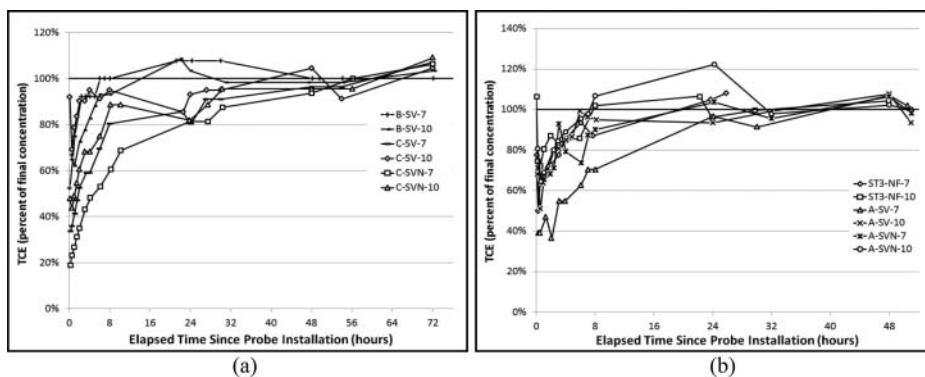


Figure 3. TCE in macro-purge probes, percent of final concentration vs. time after installation: (a) concentrations above $1000 \mu\text{g}/\text{m}^3$; (b) concentrations below $1000 \mu\text{g}/\text{m}^3$.

Discussion

Macro-purge probes

All 12 of the macro-purge probes had detectable TCE concentrations. Within four hours of installation, seven of the 12 probes reached 80% of the final determined concentration (Figures 3a and b). A minimum of eight hours was needed for the TCE concentration to consistently be at or above 80% of the final determined concentration in 10 of the 12 probes (including the seven discussed earlier). Within 24 hours of installation, 10 of the 12 probes had reached 90% of their final concentrations while two of the 12 probes required 48 hours to reach 90% of their final concentration.

The concentrations of TCE measured from the macro-purge probes generally increased steadily over approximately the first eight hours after installation (Figures 4a and b). Concentrations in most of the probes continued to increase, but at a distinctly slower rate, from approximately 8 to 24 hours. In general, concentrations stabilized after approximately 24 to 48 hours, although, in some probes, TCE concentrations continued to increase through the 72-hour sampling period. For example, C-SV-7 yielded a TCE concentration of $630 \mu\text{g}/\text{m}^3$

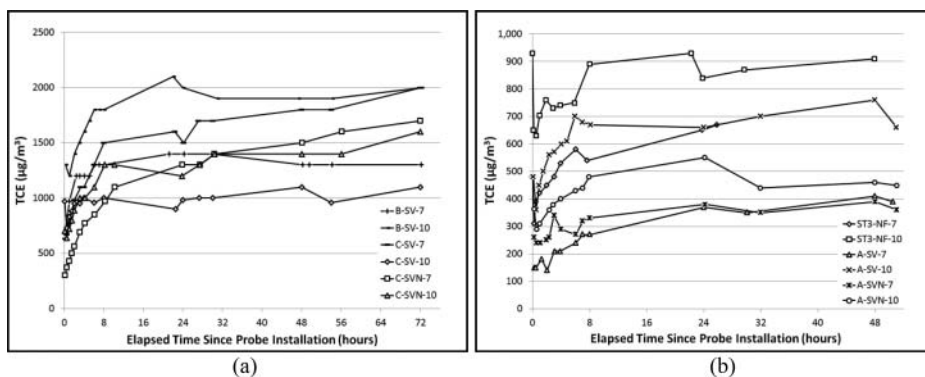


Figure 4. TCE concentration in macro-purge probes vs. time after installation: (a) concentrations above $1000 \mu\text{g}/\text{m}^3$; (b) concentrations below $1000 \mu\text{g}/\text{m}^3$.

(34% of the final determined concentration) 13 minutes after installation, $770 \mu\text{g}/\text{m}^3$ (41%) after one hour, $980 \mu\text{g}/\text{m}^3$ (53%) after two hours, $1,100 \mu\text{g}/\text{m}^3$ (59%) after three and four hours, $1,500 \mu\text{g}/\text{m}^3$ (80%) after 8 and 24 hours, $1,800 \mu\text{g}/\text{m}^3$ (96%) after 48 and 54 hours, and $2,000 \mu\text{g}/\text{m}^3$ (106%) after 72 hours. Some probes, particularly ST3-NF-10, yielded a relatively high concentration in the first sample collected, and then immediately decreased in the second sample before gradually increasing again.

Mini-purge probes

The mini-purge probes yielded concentrations that were similar to, or higher than, the final calculated concentrations almost immediately after installation (Figure 5a). Of the five mini-purge probes, four yielded concentrations over 80% of the final calculated concentration within 30 minutes of installation, while the other probe required one hour (Figure 5b). Three of the probes (ST2-MIN-4, ST2-MIN-10, and ST9-MIN-7) showed decreasing concentrations after the first eight hours following installation. The reason for this behavior is unclear; however, it is likely that the higher concentrations were more representative of *in-situ* concentrations. For example, probe ST2-MIN-10 yielded TCE concentrations of $850 \mu\text{g}/\text{m}^3$ (36%) immediately after installation; $2,200 \mu\text{g}/\text{m}^3$ (93%) 10 minutes after installation; $3,500 \mu\text{g}/\text{m}^3$ (148% and the maximum detected in this probe) after one hour; $3,100 \mu\text{g}/\text{m}^3$ (131%) after two hours, $3,000 \mu\text{g}/\text{m}^3$ (127%) after six and eight hours, and $2,600 \mu\text{g}/\text{m}^3$ (110%) after 24 hours.

Post-run tubing probes

The data set for the PRT probes was relatively limited (three probes); however, the limited number of PRT probes yielded all three patterns of concentration changes as identified for the two other types of probes (Figure 6a). Probe C10-CRTb had TCE concentrations that increased and then plateaued after two to four hours, while probe ST3-PRT-10 had TCE concentrations that increased for about an hour and then decreased with an ultimate rise in concentration, and probe ST3-PRT-7 increased and then plateaued after about an hour (Figure 6c). The PRT probe sample TCE concentrations were within 20% of the final concentrations quicker than the macro-purge probes but not as quick as the mini-purge probes.

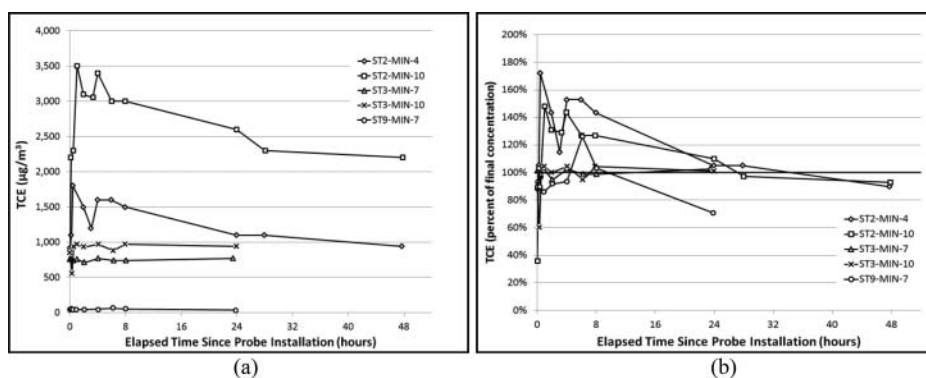


Figure 5. TCE concentration (a) and percentage of final concentration (b) in mini-purge probes vs. time.

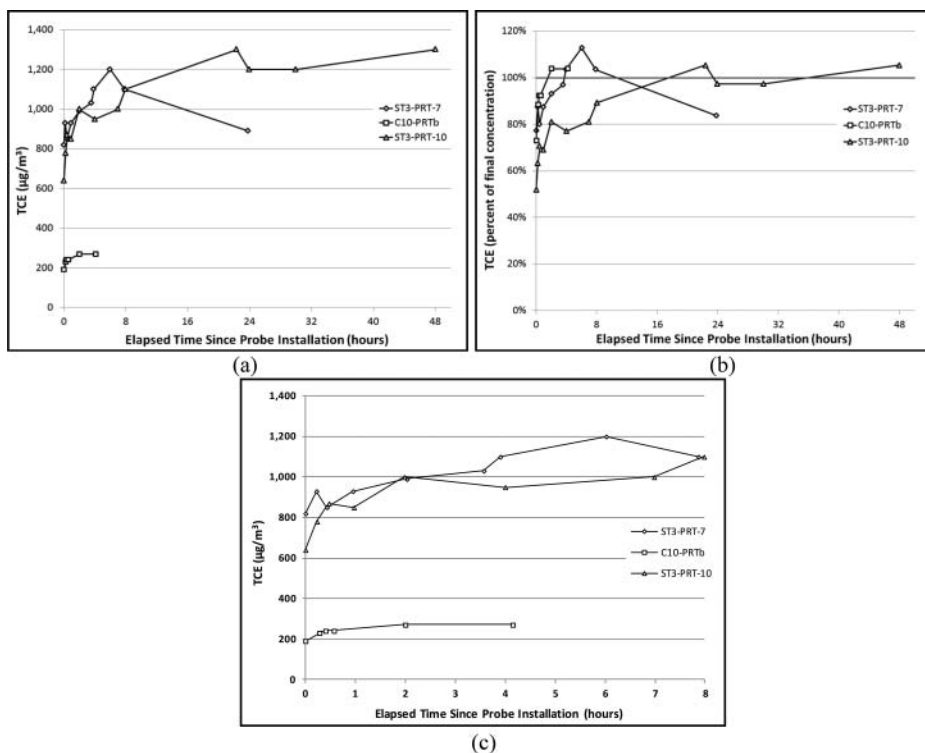


Figure 6. TCE concentrations in PRT probes vs. time.

(Figure 6b). Two of the PRT probes yielded concentrations over 80% of the final calculated concentration within one hour of installation, while the third probe required two hours. For example, Probe ST3-PRT-7 yielded TCE concentrations of $820 \mu\text{g}/\text{m}^3$ (77%) immediately after installation; $930 \mu\text{g}/\text{m}^3$ (87%) after 13 minutes and one hour; $990 \mu\text{g}/\text{m}^3$ (93%) after two hours; $1,100 \mu\text{g}/\text{m}^3$ (103%) after four and eight hours; and $890 \mu\text{g}/\text{m}^3$ (84%) after 24 hours (Figures 6a and b). Similar to some of the mini-purge probe results, this probe showed decreasing concentrations after eight hours.

tubing material cluster probes

The tubing material cluster probes were sampled six times over an 18-month period following installation (Table 3). The probes constructed with stainless steel, Nylaflow, PEEK,

Table 3. TCE concentrations ($\mu\text{g}/\text{m}^3$) in tubing cluster samples.

Tubing	26 Feb 2008	29 Feb 2008	13 Jun 2008	14 Nov 2008	18 Mar 2009	20 May 2009
Nylaflow	400	390	670	230	131	690
Teflon	380	410	810	200	112	670
PEEK	460	340	750	230	116	680
Stainless Steel	470	350	840	240	128	640
Copper	ND	170	685	220	94	630
Polyethylene	310	310	560	110	62	420

Note: PEEK = polyetheretherketone.

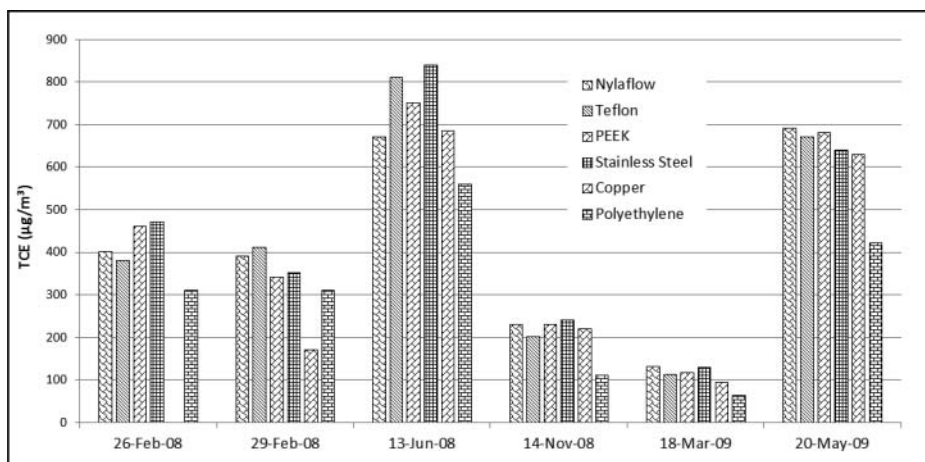


Figure 7. Histogram of TCE in tubing material samples.

polyethylene, and Teflon[®] all yielded comparable TCE concentrations, although polyethylene consistently yielded the lowest concentrations among these five materials (Figure 7). The probe constructed with copper tubing yielded significantly lower concentrations in the first two sampling periods, collected over the first 36 hours following installation; however, subsequent samples collected several months later yielded TCE concentrations comparable to the other probes. These data suggest that TCE may have been sorbing to, or reacting with, the copper tubing, resulting in lower TCE concentrations until the interior tubing surface was “saturated” with TCE or oxidized.

Conclusions

TCE concentrations collected from macro-purge probes generally increased over the first eight hours after installation and typically achieved steady-state concentrations after approximately 24 to 48 hours. Most of the macro-purge probes sampled achieved 80% of their final calculated concentrations of TCE after 24 hours, but many required 48 hours to achieve 90%. In contrast, the mini-purge and PRT probes generally reached 80 to 90% of their final concentrations within one to two hours and were within 70% within 30 minutes. This relatively fast equilibration, in comparison to the macro-purge probes, is likely because the mini-purge and PRT systems result in much less disturbance to the subsurface and exposure to the atmosphere during installation. While the macro-purge probe installation requires drilling the borehole, removing the drill rod, and then pouring the probe construction materials (e.g., sand and bentonite), the mini-purge probes are placed directly into the subsurface with the drill rod, and the rod is left in the ground during sampling.

Due to the nature of the mini-purge and PRT probe installation, it is not possible to log the soil types in which these probes were installed. However, the vadose zone soils at the site are a fairly homogenous mixture of silts and clays; all of the probes, regardless of type, were installed at depths of 2.1 and 3 meters bgs; all were installed within an area approximately 30 × 20 meters; and all of the probe types were installed at locations distributed across the study area. Therefore, it is implausible that the differences observed

in the equilibration time for the various probe types were due to differences in the soil type at the probe locations.

The appropriate time to wait between probe installation and sample collection is, therefore, dependent upon the type of probe being used, but also on the quality of data needed. For site screening, where the objective is to quickly assess the extent of a vapor plume, mini-purge or PRT probes may be the most appropriate type of probes. These two types of probes are installed more quickly than macro-purge probes, and can be sampled relatively soon after installation. However, mini-purge and PRT probes may not be suitable for long-term monitoring as they have a tendency to show decreasing concentrations in the long term after installation.

To obtain reliable data, it is recommended that macro-purge probes be installed and allowed to equilibrate for a minimum of 48 hours prior to sampling. In general, macro-purge probes are used for permanent or semi-permanent installations for risk assessment or long-term monitoring purposes, where multiple samples will be collected from the same probe weeks or months apart. Therefore, ensuring that the probe has fully equilibrated prior to the first sample collection is important, as collecting the first sample prior to equilibration could erroneously result in an apparent increasing concentration trend producing falsely low biased sample concentrations.

Six tubing materials were evaluated for their relative performance: Nylaflow, Teflon[®], PEEK, stainless steel, copper, and polyethylene. Of these materials, polyethylene, Nylaflow, and Teflon[®] are the most commonly used in environmental investigations. Our results indicate that polyethylene consistently yields lower results than other tubing materials and, therefore, should be avoided when highly accurate data are required. For screening-level assessments, polyethylene may provide adequate data, but the results should be used with caution as they may underestimate the actual concentrations present. Nylaflow, Teflon[®], PEEK, and stainless steel all yielded similar results and no consistent bias was observed. Copper yielded similar results to the other tubing materials after several months; however, results obtained within the first few days after installation were significantly lower than other tubing materials. Based on these results, copper tubing should generally be avoided, while stainless steel is a better option when metal tubing is required.

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