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Analysis of trichloroethene vapour in soil-gas samples using solid-sorbent tubes with gas chromatography/mass spectrometry

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Abstract

A sampling method for the determination of chlorinated contaminant vapor concentrations present in the vadose zone, specifically TCE, has been developed, and was applied at the Tucson International Airport Authority (TIAA) Superfund site. The method, modified from the NIOSH Manual of Analytical Methods (NMAM) # 1022 for ambient-air sampling of TCE, is targeted to situations requiring cost effective sample collection, particularly for cases when concentrations are at or below maximum contaminant levels (MCLs). In the modified NIOSH method, TCE vapor is sampled using a solid sorbent tube. Gas Chromatography with Mass Spectrometry is used to confirm and quantify the presence of TCE. The results of laboratory tests demonstrate a maximum TCE vapor load of approximately 22 mg before breakthrough to the secondary sorbent tube section, and an extraction efficiency of approximately 97%. The results of a performance comparison test conducted in the field demonstrated that concentrations obtained with the sorbent tube samplers (~5 ug/L) were similar to those obtained with the use of standard Summa canisters (~3 ug/L). The quantitative detection limit for the new method was 0.03 ug/L under the operative conditions, a significant improvement to current regulatory analytical methods. The results indicate that use of the sorbent tube method will be effective for vapor sample collection at VOC-contaminated sites, particularly in characterizing low concentrations.

Keywords

trichloroethene; vapor phase; soil gas; VOC sampling method

Introduction

Many sites contaminated by volatile organic compounds (VOCs) contain source zones within the vadose zone. Two major concerns are associated with sites containing vadose-zone contaminant sources. First, discharge of contaminant vapor from the vadose-zone source may impact the underlying groundwater.^[1,2] Second, contaminant vapor from the

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vadose-zone source may migrate to the land surface and transfer into buildings, thereby causing vapor intrusion.^[2,3] Measuring VOC distributions and concentrations for vadose-zone systems is an important component of characterizing risk and developing remediation plans for these sites.

Analytical methods quantifying VOCs in the vadose zone are established.^[4–8, 13–16] Regulatory agencies and commercial labs primarily use gas chromatography (GC) coupled with detectors (i.e. MS, ECD, FID, etc.) as the standard analytical method for VOC vapor and liquid samples. These analytical methods are internationally approved and provide the best results given varying site conditions. Compound-specific isotope analysis (CSIA) has been used for more than a decade to characterize the impact of transformation reactions and other processes on contaminant transport and fate in groundwater. CSIA has recently begun to see use for determining contaminant concentrations for vadose-zone applications.^[9–12] One example is its use to distinguish contamination originating from in-home products from subsurface soil sources (vapor intrusion).^[13]

Both the standard (GCMS) and newer (CSIA) methods require the collection of robust vapor samples from the vadose zone. Currently, summa canisters are the industry standard device used for (active) soil gas sampling in the vadose zone. Soil gas sampling guidance documents (i.e., EPA Method 0040 and Compendium Method TO-15)^[14, 15] suggest summa canisters and tedlar bags exclusively for soil gas sampling in the vadose zone. These devices require a large sample volume (i.e., 1, 4, 15 or >20 L of gas) for analysis. Resultant sampling times vary from minutes to hours based on flow rate requirements. In addition, summa canisters and tedlar bags can be relatively expensive to use, particularly for sites requiring large sample sets. In some cases, their use may become problematic as the vapor concentrations of the relevant constituents become sufficiently low versus the method QDL. These limitations illustrate the need for alternative vapor sampling methods. One possible approach is the use of sorbent tubes.

Current ambient air sampling methods use sorbent tubes for both active and passive methods.^[4,7, 16–17] For example, NIOSH method #1022 uses a solid sorbent tube for the analysis of TCE. Vapor phase TCE is sorbed to the charcoal material and subsequently analyzed. This NIOSH method is one of a few methods using sorbent material as the primary ambient-air sampling device for VOCs. Sorbent tubes have been demonstrated to perform very effectively for a range of ambient air sampling and monitoring applications. However, the sampling conditions for ambient-air systems are quite different from subsurface conditions. For example, while sample collection periods typically span many hours or days for the former, sampling times of minutes to a few hours are generally the norm for subsurface systems. In addition to sample collection periods, the sample matrices for ambient-air and subsurface systems can be quite different. The ambient air system contains particulates that can affect the sampling protocol whereas the vapor in subsurface systems typically is void of particulate matter. Hence, specific testing of the sorbent tube approach is required before it can be adapted for use for vadose-zone sampling applications.

Improving upon current sample-collection methods is paramount as analytical detection limits (MDLs) are lowered and in recognition of the observation that many hazardous-waste

sites require many decades to attain site closure.¹⁸ This requires updating current methods or creating new innovative methods that can provide rapid collection of many samples at low cost. This study investigates the use of a sorbent tube as an active soil gas collection device for vadose-zone systems. Laboratory experiments are conducted to determine extraction efficiency and maximum holding capacity. The performance of the sorbent tubes is compared to that of summa canisters through a field test at a hazardous waste site.

Experimental

Materials

An ORBO-32 (large) sorbent tube with 400 mg of coconut charcoal in section A and 200 mg in section B was used for sorption of TCE vapors (Supelco Analytical; Sigma-Aldrich Corp., St. Louis, MO). The flow rate limitation of the cartridge is 50–1000 mL/min. Sections A and B were extracted and analyzed separately to determine TCE concentrations and maximum sorption capacity for each section. Section A is designed to collect the compounds of interest while section B is designed as a backup to section A. Section B is used in this research to determine breakthrough occurrence from section A. Although other sampling sorbent devices such as activated carbon, silica gel, porous polymers, resins and mixed media exist for ambient air and soil gas sampling, Harper (1994)^[17] suggests that coconut charcoal has a very broad range of uses due to its surface area in reference to silica and hydrophobicity. Pore size and uniformity of a sorbent material can affect its ability to adsorb VOCs optimally. Due to TCE's planar shape and less than 7 angstrom size, it will adsorb into micropores of a carbon based adsorbent.^[19] Thus, the coconut charcoal sorbent material is acceptable for use of VOC sorption.

Trichloroethene (TCE) for standard stock solutions was obtained from Sigma-Aldrich (Sigma-Aldrich Corp., St. Louis, MO) as reagent grade (> 99.4%). Dichloromethane (DCM) used for sorbent extraction was obtained from Sigma-Aldrich and contained amylene as a stabilizer as ACS reagent (>99.5%). Hexane (CHROMASOLV, HPLC grade) from Sigma-Aldrich (Sigma-Aldrich Corp., St. Louis, MO) was used as an alternative extractant in initial tests.

Experiments

Two laboratory tests were conducted. The first, referred to as Laboratory Test A, was designed to determine the extraction efficiency of the cartridge sample processing method. Known volumes of vapor containing measured concentrations of TCE were injected into the sorbent tube. The sorbent was then removed from the tube and subjected to solvent extraction with DCM. The second test (Laboratory Test B) was designed to determine the TCE holding capacity of section A of the sorbent tube. A known volume of TCE vapor was injected into the cartridge. Each section was then subjected to extraction and analysis separately. Different masses of TCE were loaded until quantifiable concentrations were detected in section B, indicating attainment of maximum loading of section A. Lastly, a field test was conducted to validate the sorbent tube method by providing a comparative test to the standard summa canister method. This test will be referred to as the Field Test.

Methods

Laboratory Test A.—A stock volume of TCE vapor was created by placing 5 ml of pure TCE into a 20 mL 28×60 cm Kimble Chase EPA vial with septa and polypropylene cap. The pure liquid TCE was allowed to equilibrate with the gas phase over the span of 24 h. The calculated vapor solubility of TCE is approximately 500 mg/L based on the ideal gas law (equation 1 in “*Calculations*” section). The TCE-saturated gas served as the stock vapor in preparation for Laboratory Test A and B. Calculation of recoveries (equation 2 in “*Calculations*” section) was used to assess the extraction efficiency and the precision of the sampling methodology.

Known volumes of the TCE saturated vapor were injected onto the sorbent tube. The sorbent tubes were opened following manufacturers’ instructions and the charcoal fractions were placed in 20 mL 28×60 cm Kimble Chase EPA vials with septa and polypropylene cap. The samples were then extracted using DCM. The masses recovered were compared to the known input masses.

Laboratory Test B.—A series of experiments was conducted to determine the maximum holding capacity of TCE for section A. Select volumes of saturated TCE gas ranging from 10–60mL were loaded onto the solid sorbent tubes. These volume ranges equate to 5–30 mg of TCE mass loaded. The masses recovered were calculated for sections A and B. Exceedance of the holding capacity of section A was deemed to occur when the mass recovered for section B was greater than approximately 1% of the total mass load.

Field Test.—The field test of the sorbent tube-based sampling method was conducted at the Tucson International Airport Area Superfund site in Tucson, Arizona in 2013 (Figure 1) [21] A sample port is connected to a GAST diaphragm type DOA-P704-AA 1/8HP 115Vac purging vacuum pump and purged for a duration of 2 min. After the purge, a sorbent tube is connected via Teflon tube to the sampling port and in line with a Schwarzer precision SP 402 SA-R-V collection vacuum pump. The sampling flowrate is set and controlled using an Omega FL-3802ST flowmeter, installed in-line between the sorbent tube and the sampling vacuum pump inlet. All electric equipment is taken to the sampling port and powered using a generator. Sampling target duration is 2 min. The sampling target flowrate is 150 ml/min but was adjusted in the field for constant optimal flow for each sampling port. Both sample time and flow rate values are recorded. After sampling, the sorbent tube is disconnected, capped and stored in a cooler for transport to the laboratory where it is immediately prepped for analysis. The sorbent tubes were prepared, sub-sampled, and analyzed by University of Arizona staff in the Arizona Laboratory for Emerging Contaminants of the Soil, Water and Environmental Science Department.

Commercial summa canisters were used to collect samples for comparison. The summa canisters were employed one week, less than 24 hours, and one week apart from the sorbent tube sampling, respectively, for the January, May and August dates. The Summa canisters were deployed by personnel from Conestoga-Rover Associates (CRA), and delivered to TestAmerica Laboratories, Inc. in Phoenix, Arizona for analysis. Hence, the use of the summa canisters was completely independent of our study. The samples were collected

following the protocol described in EPA TO-15 method previously mentioned. Note that the sorbent tubes and Summa canisters were sampled under closed environments from sample origin to sampling device. Hence, sample volatilization or dilution issues were minimized.

Chemical Analysis

Standard stock solutions for development of calibration curves were prepared on a Mettler AE 240 analytical precision balance in 100 ml volumetric flasks. A 135 ml volume of dichloromethane was added to the volumetric flask and weighed. 20 μ l of TCE was added using a 50 μ l syringe mixed with a stir bar for 24 hours. Using the mass, volume and density measurements of TCE and dichloromethane, stock solution concentrations of TCE were calculated prior to preparation of serial dilution samples. All calibration, check standards and samples collected were stored at 4°C.

For all tests, sorbent from sections A and B of each tube was extracted with 5 mL of dichloromethane, mixed for 30 minutes, agitated, and then sub-sampled. A 2 mL aliquot was placed into a 2 mL headspace-free auto-sampler vial for TCE analysis by gas chromatography mass spectrometry (GCMS) using an Agilent Technologies 7890A gas chromatograph attached to a Quattro micro mass spectrometer (Waters-Micromass, Beverly, MA). For GC, a J&W DB-5MS (30 m, 0.25 mm, 0.25 μ m) column was used with 99.999% helium carrier gas at 1.1 ml min⁻¹ constant flow rate. Sample extracts were stored in 12 $\times$ 32 mm, 2 ml, threaded, glass auto-sampler vials with plastic caps and Teflon/silicone septa from Microliter Analytical Supplies, Inc. Liquid extracts (1 μ l) were injected by an Agilent 7683 auto-sampler into the GC inlet using a split mode of 10:1 at 250°C. The transfer line temperature was 275°C. The GC temperature program held the oven at 30°C for 4 min and then increased the temperature at a rate of 30°C min⁻¹ up to 75°C followed directly by another temperature increase of 45°C min⁻¹ up to 275°C. All analyses were conducted using positive ion electron impact (EI) at 70 eV in selected ion monitoring mode. A single channel was monitored at m/z = 94.84 for detection of trichloroethene, which consistently eluted at 3.1 min. Results were processed using the MassLynx (TargetLynx) software provided by Waters-Micromass.

TCE concentrations for the liquid extractant were calculated using the calibration curve. Limits of detection (LOD) and quantitation (LOQ) were determined by measuring a signal to noise ratio (by root mean squared method) of 3:1 and 10:1, respectively, for the selected ion chromatogram peak for TCE. Using serial dilution standard samples spanning 0.05 – 98 ppm, a calibration curve (r^2 value of 0.993) was produced by regression. All reported results fell within this range. A weighting function of 1/X was applied in order to add weight to calibration points with a low response. This allowed the curve to fit closer to low-concentration points, thereby reducing relative error at these points. Solvent blanks and standard samples for quality control were run every 6 samples for QA/QC purposes. The liquid TCE concentrations were converted to gas phase concentrations based on sampling flow rates and collection times.

A UV-VIS spectrophotometer (Shimadzu Scientific Instruments, Inc., Columbia, MD) was used for the analysis of TCE in solvent extracts for Test B.

Calculations

Using the NIOSH Manual of Analytical Methods (NMAM)⁴ as a template, known concentrations of TCE vapor were created. The vapor phase solubility of TCE was calculated and expressed by equation (1):

$$C_{ig} = p^*/RT \quad (1)$$

where C_{ig} is the concentration of TCE (i) in the gas phase (g), p^* is the vapor pressure of TCE, R is the ideal gas constant and T is the temperature.

Recovery of TCE was calculated using equation (2):

$$\% \text{ Recovery} = (\text{mass TCE recovered} / \text{mass TCE loaded}) \times 100 \quad (2)$$

Upon acceptable recovery in Sections A and B, optimization of the quantitative detection limit (QDL) was performed. The concentration of TCE at the QDL was determined using equation (3):

$$C_{DL}(\text{mg/L}) = M(\text{mg})/Q(\text{L/min.}) \times t(\text{min.}) \quad (3)$$

where M is the maximum determined mass of TCE at breakthrough into section B, Q is flow rate and t is the exposition time. Therefore, if given the QDL of the method, with a fixed flow rate and TCE breakthrough mass being known (M), the exposure time can be calculated prior to use in the field. This is illustrated in Figure 3.

Results and Discussion

Test A- TCE Extraction Efficiency

This test was conducted to determine the efficiency of TCE recovery for the solvent extraction of the charcoal sorbent. The mean recovery of TCE for Section A is 97% (see Table 1). The mean recovery of TCE for Section B is 10% ($n=14$ for Section A and $n=12$ for Section B). This indicates acceptable TCE mass recovery from Section A, with little to no breakthrough of TCE vapor into Section B. As section B was designed for sample overflow, this test shows the precision and accuracy of the sampling method.

Test B- Holding Capacity

Specific volumes of saturated TCE vapor were injected into the sorbent tube and subsequent mass loadings were determined to evaluate the maximum holding capacity of section A, based on breakthrough into section B. The maximum mass load of section A was determined to be approximately 22 mg (Figure 2). The reported load range for the standard 100/50 sorbent tube used in NIOSH method 1022 is 0.5 – 10 mg. This equates to a range of 2 – 40 mg for the 400/200 sorbent tube used herein, assuming proportionality. Hence, our results are consistent with those reported for the standard sorbent tube.

Sample Collection Time

The results obtained from Test B can be used to determine maximum and minimum sample collection times. The maximum time for which a single sorbent tube can be used before its capacity is exceeded depends the extant analyte concentration and the sample-collection flow rate. The maximum sample collection time can be determined as a function of analyte target concentration for selected flow rates. This function is presented in Figure 3 for two selected flow rates. As shown, the collection times required to reach maximum capacity are quite long for most conditions. For example, a sample with TCE concentration of 1 ug/L requires 1500 hours at low flow rates and 375 hours for high flow rates. These times are extremely long for standard field sampling applications, indicating that it is unlikely that the holding capacity would be exceeded under typical site conditions and sample-collection times.

The minimum mass load of Section A required to obtain detection above the QDL was calculated using the instrument detection limit of 0.01 ug/L, and determined to be 0.00001 mg. With a known minimum mass load for TCE, the minimum required sample collection time can be determined (Figure 4). The minimum sample collection times are quite small, ranging from 4 minutes for low flow rates and 1 minute for high flow rates (concentration at 0.01 ug/L). These results indicate that the modified method allows one to quantify soil gas concentrations while employing very reasonable (short) sampling times.

For example, current method sampling times can range from minutes to hours. Compendium method To-17 reports a typical sampling time of 3 hours at 0.01 L/min. to collect 1.8 liters. The NIOSH method recommends a mass load of 0.5 – 10 mg per sample as flow rates range from 0.01 – 0.2 L/min. This equates to a 50-minute sample time at 0.01 L/min. rate to collect 0.5 liters. Conversely, Figure 3 shows a sampling time of 1.5 hours for a low (0.25 L/min.) flow rate to collect 22L at a target concentration of 1 mg/L. Figure 4 shows a sampling time of 0.0004 minutes for a low flow rate to collect 0.01 mL at a target concentration of 1mg/L. This research method shows mass loads, flow rates and sampling times improved.

Field Test

The field test was conducted to evaluate performance of the sorbent tube device by comparing reported TCE concentrations to those obtained with summa canisters. Several factors in general may potentially produce disparities between the two methods. One factor may be sample volume. Sample volume is reflected in the zone of influence and subsequent concentrations therein. Referenced sample volumes range from 1 to greater than 20 liters. The sample volumes collected for the sorbent tube and summa canister for this test were 1.6 L and 1 L, respectively. Thus, the zone of influence between the two methods is comparable. Therefore, it is unlikely to be an issue for this application. Relative humidity can affect both sampling methods. At humidity greater than 50%, certain sorbent tubes are hydrophilic and water vapor prevents the adsorption of select VOCs.^[17] In addition, increased water vapor can inhibit pre-analytical steps with summa canisters.^[20] The field test site is in an arid environment with corresponding very low humidity. Therefore, humidity effects were likely minimal for this test.

The TCE concentrations obtained with the sorbent tube protocol averaged ~5 ug/L (Table 2). They are quite comparable to those obtained with the summa canister protocol (~ 3 ug/L). This result supports the accuracy of the sorbent tube method for use in collecting VOC samples for vadose-zone applications.

The sorbent tube method QDL is 0.03 ug/L based on the protocol employed in this study (800 ml/min. flow rate, 2-minute collection time, and dilution factor of 5). It is also significantly lower than the current EPA method MDL of 0.5 ug/L for ambient air sampling (i.e., EPA Method TO-17) and for subsurface vapor sampling (i.e., EPA Method TO-15). Improving upon the detection limit is invaluable when assessing VOC concentrations at or below the MCL.

Conclusion

A sorbent tube method currently used for sampling chlorinated compounds in the ambient air has been adapted for the sampling of VOCs in the vadose zone. The results obtained with this method compared well to those obtained with the standard method for the field site test. The use of this sampling device adds to the toolkit of methods available for collecting soil gas samples.

The maximum and minimum load of Section A was determined to be 22 mg and 0.00001 mg, respectively, for TCE. These values can be used to estimate sample-collection time required for the sorbent tube when the desired DL is known. The modified sampling method in this study has minimum and maximum sampling times that are quite comparable to current methods requiring sampling times of fractions of minutes to less than an hour for high flow rates. The minimum and maximum sorbent tube exposure (collection) times reported above are nominal values. As such, the values do not account for purge time typically used to account for dead volumes.

The QDL for the modified method is 0.03 ug/L for the protocol used herein and is an improvement to those for existing state and federal analytical methods. The improved QDL values are approximates and are dependent on site conditions, current soil gas concentrations and sorbent tube extraction efficiency. The sampling method is viable for a wide range of vapor concentrations, based on the minimum and maximum loadings determined herein. Significantly, the QDL is well below the MCL for TCE (5 ug/L). Conversely, the maximum mass load translates to an approximate 10 second sampling time for conditions of a 0.25 mL/min flow rate and 474 mg/L TCE concentration.

A critical question of concern is when should an SVE system be closed down, or replaced with another remedial method. The application of the sorbent tube method will aide responsible parties in making this very important decision. By improving upon the quantitative detection limit of the standard method, the sorbent tube method is an additional sampling tool to help address the SVE system closure question.

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Figure 1.
Tucson International Airport Area superfund site map.

Figure 2.
Determination of maximum TCE mass loaded for breakthrough into Section B.

Figure 3.
Variation of maximum sorbent-tube sampling time with target concentration.

Figure 4.
Variation of minimum sorbent-tube sampling time with target concentration.

Table 1.

Mean recovery of TCE for sections “A” and “B”.

Sorbent Tube Section	Average Mean Recovery (%)	Standard Deviation of Recovery	Covariance	Average TCE removed (mg)	Approximate TCE mass loaded (mg)
A (n=14)	97.15	15.71	0.16	4.86	5
B (n=12)	0.19	0.04	0.23	0.01	0.14
background correction	0.15	0.0003	0.002	0.007	0

Table 2.

TCE air concentrations for comparison of Summa canister and Sorbent tube protocol.

Sampling date	Summa canister protocol TCE air concentration (µg/L)	Sorbent tube protocol TCE air concentration (µg/L)
Jan/Feb 2013	3.8	7.0
May 2013	2.1	5.6
August 2013	2.4	3.7
Mean	2.8 (1.7–3.8) ^A	5.4 (3.5–7.3) ^A

^A 95% confidence intervals.