FINAL REPORT

FIELD SCREENING FOR HALOGENATED VOLATILE ORGANIC COMPOUNDS: THE NEW X-WAND[TM] HVOC SCREENING DEVICE

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DISCLAIMER

ABSTRACT

Western Research Institute (WRI) has developed new methodology and a test kit to screen soil or water samples for halogenated volatile organic compounds (HVOCs) in the field. The technology has been designated the X-Wand[TM] screening tool. The new device uses a heated diode sensor that is commonly used to detect leaks of refrigerants from air conditioners, freezers, and refrigerators. This sensor is selective to halogens. It does not respond to volatile aromatic hydrocarbons, such as those in gasoline, and it is not affected by high humidity.

In the current work, the heated diode leak detectors were modified further to provide units with rapid response and enhanced sensitivity. The limit of detection for trichloroethylene TCE in air is 0.1 mg/m^3 (S/N = 2). The response to other HVOCS relative to TCE is similar. Variability between sensors and changes in a particular sensor over time can be compensated for by normalizing sensor readings to a maximum sensor reading at 1,000 mg/m^3 TCE. The soil TCE screening method was expanded to include application to water samples. Assuming complete vaporization, the detection limit for TCE in soil is about 1 ug/kg (ppb) for a 25-g sample in an 8-oz jar. The detection limit for TCE in water is about 1 ug/L (ppb) for a 25-mL sample in an 8-oz jar. This is comparable to quantitation limits of EPA GC/MS laboratory methods.

A draft ASTM method for screening TCE contaminated soils using a heated diode sensor was successfully submitted for concurrent main committee and subcommittee balloting in ASTM Committee D 34 on Waste Management. The method was approved as ASTM D 7203-05, Standard Test Method for Screening Trichloroethylene (TCE)-Contaminated Soil Using a Heated Diode Sensor.

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EXECUTIVE SUMMARY

Western Research Institute (WRI) has developed new methodology and a test kit to screen soil or water samples for halogenated volatile organic compounds (HVOCs) in the field. The technology has been designated the X-Wand[TM] screening tool (patent pending). The new device uses a heated diode sensor that is commonly used to detect leaks of refrigerants from air conditioners, freezers, and refrigerators. The sensor is selective to halogens and it does not respond to volatile aromatic hydrocarbons or water vapor. The limit of detection (S/N = 2) for the heated diode sensor is about 0.1 mg/m^3 trichloroethylene (TCE) in air. The maximum reading is obtained at about 1,000 mg/m^3. Variability between sensors and changes in a particular sensor over time can be compensated for by normalizing sensor readings to a maximum sensor reading at 1,000 mg/m^3 TCE in air and by providing an additional calibration point at 100 mg/m^3 TCE in air.

The device can be used to screen headspace above either soil or water. Assuming complete vaporization, the detection limit for TCE in soil is about 1 ug/kg (ppb) for a 25-g sample in an 8-oz jar. The detection limit for TCE in water is about 1 ug/L (ppb) for a 25-mL sample in an 8-oz jar. The variable volume headspace technique can be used to compensate for incomplete VOC partitioning between a sample and air. This variable volume calculation is more suited for application to water, since there is typically a single coefficient for water and headspace partitioning. The results show that the modified heated diode leak detector can be used to screen HVOC concentrations in soil and water with quantitation limits comparable to the EPA GC/MS laboratory methods.

ASTM Committee D 34 on Waste Management approved the development of a new draft ASTM standard for screening TCE-contaminated soils using a heated diode sensor. The initial draft method was balloted within ASTM subcommittee D 34.01 and was revised based on reviewer comments. The revised draft was successfully submitted for concurrent main committee and subcommittee balloting in ASTM Committee D 34. The method was approved as ASTM D 7203-05, Standard Test Method for Screening Trichloroethylene (TCE)-Contaminated Soil Using a Heated Diode Sensor.

OBJECTIVES

The ultimate goal of the effort is to develop and commercialize a field-portable kit for screening halogenated volatile organic compounds (HVOCs). The X-Wand[TM] screening tool is based on a refrigerant leak detector from Bacharach Inc., New Kensington, PA, modified to provide a numerical readout.   A method for screening trichloroethylene (TCE)-contaminated soil was developed, and balloted in ASTM International. A water screening method is being developed, and the ASTM method will be revised to include water screening in the future.

INTRODUCTION

Halogenated Volatile Organic Compounds

HVOC contamination is a widespread problem at U.S. Department of Energy (DOE), commercial, and military sites.   Compounds, such as carbon tetrachloride, TCE, tetrachloroethylene, etc., are also referred to as dense nonaqueous phase liquids (DNAPLs). These were used extensively in degreasing and equipment cleaning operations in the past, with disposal practices that led to their release into the environment. Many HVOCs are still in use as degreasing solvents in petroleum refining and other industries (U.S. DOE 1998). VOCs are the most significant organic contaminants in groundwater associated with disposal sites in the U.S. (Plumb 1992). These represent 75% of events involving organic contamination in contaminated sites. Similar observations were made for sites in Germany (Kerndorff et al. 1992).

Background and Prior Approaches to HVOC Screening

HVOC contamination is a common issue encountered by many state agencies and environmental engineering firms. Over the last decade, research at WRI included work with photoionization detection (PID) with various VOCs in soil and water (Schabron et al. 1996). PID is the most common VOC field screening tool in use today. Typical PID lamp energy is 10.6 electron volts (eV), which is sufficient for ionizing compounds containing double bonds. However, HVOCs without double bonds, such as carbon tetrachloride or dichloromethane, require energy of 11.7 eV for ionization (Driscoll and Becker 1979).   This can only be accomplished with a PID equipped with a lithium fluoride window, which has a short lifetime due to the solubility of lithium fluoride in water. Field screening with a PID probe can involve placing a soil sample in a plastic bag or a glass jar, sealing the bag or covering the jar with aluminum foil, then inserting the probe tip into the headspace in the container (Hewitt and Lukash 1997). In some cases, field engineers simply move the PID probe above the surface of the soil to determine if target VOCs such as volatile fuels are present. PID detectors have a disadvantage in that they cannot discriminate between halogenated and non-halogenated species. A more detailed analysis technique that also provides for some speciation information involves a portable gas chromatograph, which requires skilled operators (Myers et al. 1995, Linenberg

1995). Immunoassay kits allow for field analysis, however, this approach requires controlled temperature and critical timing, as well as involving several steps (Hudak et al. 1995).

There is a need for a new type of simple field monitor that is selective to HVOCs. WRI has developed a new environmental monitoring tool based on a heated diode refrigerant leak detector platform. The device can be used to screen headspace above soil or water.

Several novel approaches have been proposed for surface or down-hole screening of HVOCs (Schabron et al. 1991). One approach uses refractive index attenuation on coated optical fibers (Le Goullon and Goswami 1990, Oxenford et al. 1989). Another technology uses a chemical reaction in a basic media to form a color in the presence of trichloroethylene (Milanovich et al. 1994, 1986). A LaF_2-doped element heated to 600 °C (1,112 °F) has been used to measure volatile chlorine-containing compounds (Buttner et al. 1995, Stetter and Cao 1990). A synthetic nose consisting of an array of different chemicals that give a different optical response to various volatile analytes has been proposed (Walt 1998). Other approaches include Raman spectroscopy (Ewing et al. 1995, Haas et al. 1995), electrochemical cells (Adams et al. 1997), acoustic wave devices (Frye et al. 1995), and ion mobility spectrometry (Stach et al. 1995). The above devices all represent some progress toward the problem of monitoring for various VOC indicator compounds at varying levels; however, these are not commercially available.

The detector system that is used must to operate in an environment of varying and often high relative humidity. Since a common non-halogenated VOC contaminant is gasoline, interferences from aliphatic or aromatic hydrocarbons must also be minimal. The detector must demonstrate a significant selectivity to halogenated VOCs in the presence of non-halogenated VOCs.

New Methodology

The current work has led to the development of new commercial technology to provide a cost-effective means to rapidly screen for HVOCs in the field. This involves taking existing refrigerant detectors, and with slight hardware modification and comprehensive analytical method development, launching them into a new commercial application with significant use to the environmental industry.

In prior work, commercially available heated diode and corona discharge leak detectors were modified to provide readouts that correspond to the concentration of HVOCs in air (Schabron et al. 2002, 2003, 2004). Sensor response was evaluated for several HVOCs with and without double bonds. The response characteristics were determined for the VOCs in air in Tedlar bags with no soil present. Potential interferences from volatile fuel hydrocarbons such as toluene and heptane were evaluated and found to be nonexistent. Work with the corona discharge detector was discontinued because the detector was found to have a response to

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humidity that could not be zeroed out. Method development work was conducted with the heated diode sensor.

<div align="center">

EXPERIMENTAL DETAILS

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Chemicals

The HVOCs used in this work were reagent grade or better and were obtained from Aldrich Calibration gases and from Scott Specialty Gases.

X-Wand Heated Diode Sensor Unit

The X-Wand heated diode unit is a modified version of a model H-10PM refrigerant leak detector from Bacharach Inc., New Kensington, PA (Figure 1). The diode sensor is heated between temperatures ranging from about 600–1,000 $^{\circ}$C (1,100–1,800 $^{\circ}$F). The voltage readout is proportional to a microamp current from the diode, which results from the reaction of halogen with alkali metal vapor. The on-board sampling pump provides an unrestricted air flow through the sensor at about 0.38 L/minute. It is important not to block the flow of air since the baseline reading can be affected. A 14-gauge 4-inch long, blunt stainless steel needle is attached to the wand tip to allow the probe to be used to sample air from various containers. Five modified heated diode sensor units were provided by Bacharach Inc., New Kensington, PA for the development work. These are equipped with on-board LCD voltmeters which provide a numerical voltage readout that is related to the current produced by the diode in the presence of halogens. The units are equipped with zero and span knobs. The zero knob is manually adjusted to provide a zero reading in the absence of HVOCs in air. The span knob is set to provide a voltage readout of 9 – 12 V when exposed to a 1,000 mg/m^3 TCE in air standard.

Gas Chromatography and Gas Chromatography/Mass Spectrometry Analysis

Gas chromatography (GC) analyses were performed using a Hewlett-Packard 5890A GC equipped with an electron capture detector (ECD). The column was a J&W DB-624 30 m x 0.53 mm i.d. x 3 micron film thickness. Six GC calibration standards for each VOC were prepared from certified standard solutions in methanol from Supelco. Calibration was performed daily using 1-microliter injections of standard solutions. The GC/MS analyses were performed using an Agilent Model 6890 GC with Agilent Model 5973 MSD equipped with Agilent Chemstation software using a Tekmar-Dohrmann Aqua Tek 70 liquid auto sampler and a Supelco Purge Trap K (VOCARB 3000) installed in a Tekmar-Dohrmann Model 3100 Sample Concentrator. Calibration standards were prepared in methanol using aliquot volumes consistent with those used for the samples.

X-Wand Calibration and Sample Screening

Headspace vapors of 1,000 and 100 mg/m^3 TCE in air were obtained by connecting a 1-L

Tedlar bag to Scott Specialty Gases calibration cylinders using a 1-inch length of ¼-inch id Viton tubing. The calibration standards were custom made by Scott Specialty Gases in 48-L aluminum cylinders or 221-L steel cylinders. Since the cylinders were certified in terms of ppmv, which is typical of gas calibration standards, the ppmv value was converted to mg/m^3 using the ideal gas equation PV = nRT. Additional concentrations of standards were obtained by preparing solutions of the pure standard in methanol, and pipetting 5 uL of a particular standard directly into a 250 mL septum jar, and immediately capping the jar. Response of the X-Wand was found to be essentially identical to standards provided in either manner. The LCD readout was initially adjusted to zero with the zero control knob, and the span knob was adjusted to provide a reading of about 10V for 1,000 mg/m^3 TCE.

A portion of sample (soil or water) was placed into a 250 mL jar with a septum lid. The samples were shaken prior to screening. A sharp 10 gauge needle 0.75 inches long was used to puncture the septum, and the 14-gauge probe tip was inserted into the jar through the 10-gauge needle. A maximum voltage reading was obtained within a few seconds, after which it dropped. Headspace concentrations in mg/m^3 were calculated from the voltage readings.

RESULTS AND DISCUSSION

X-Wand Calibration

At HVOC concentrations between 0 and 10 mg/m^3, the relative voltage response is linear with concentration. This has been observed using many different sensors throughout the course of the development work. Between 10 mg/m^3 and 1,000 mg/m^3, the response is an approximate linear function of the log of analyte concentration in air. The response profiles can vary slightly for different sensors. Normalized relative response profiles for TCE for the modified heated diode prototype are shown in Figure 2. All measurements are relative to the voltage response for a 1,000 mg/m^3 TCE standard. In this manner, compensation can be made for different response profiles for different sensors.

The response of the heated diode detector is similar to the response of an electron capture detector, which uses a radioactive nickel beta radiation source to generate electrons which react with electronegative compounds. As with the heated diode detector, response is approximately logarithmic, indicating first order reaction rate characteristics. Log response suggests that the sensor output current is a function of a first order reaction between the HVOC and the alkali metal vapor. Since at low concentrations (<10 mg/m^3) the response shows linear characteristics, the interaction with the halogenated compound at low levels with the alkali metal vapor results is probably due to near complete reaction, and at higher levels (>10 mg/m^3), the interaction is incomplete and the voltage signal is a function of the reaction rate, which is a function of the log of analyte concentration. Above 1,000 mg/m^3 TCE, the response of the sensor levels out and

becomes erratic. Sensor noise is about 3 mV. If a more conservative number such as 5 mV is used, the quantitation limit (S/N = 2) for TCE is 0.1 mg/m^3.

To accurately interpret the signal from the heated diode sensor, two calibration gas cylinders should be used. The procedure described below can be used for either TCE or CCl_4. It could possibly be used for other HVOCs however this would require additional study. For TCE in air, the cylinders have a concentration of about 220 ppmv and 22 ppmv. This corresponds to about 1,000 mg/m^3 and 100 mg/m^3, respectively. The conversion from ppmv to mg/m^3 uses the ideal gas law, and requires knowledge of the temperature and atmospheric pressure.

After the X-Wand is set to zero in clean air, the span knob is set to about 10V when reading the 1,000 mg/m^3 standard C_{Ha}. This gives a reference voltage. All other voltage readings are divided by this reference voltage to provide a relative response value R_x. The relative response R_a for the 1,000 mg/m^3 standard (or whatever the exact concentration is) is 1.00 by definition. To compensate for different sensor characteristics, the response from a second standard, near 100 mg/m^3 (C_{Hb}) is obtained by dividing the voltage into the voltage for the 100 mg/m^3 standard by the voltage for the 1,000 mg/m^3 standard to give a relative response value R_b. For different sensors, this value can range from near 0.80 to 0.35. Many sensors give a value near 0.55.

Signal Interpretation

Three lines are defined to interpret the signal from an unknown sample. These are shown in Figure 2. Development work with many sensors for over a year indicates that for R values ranging from 0.00 to 0.10, the sensor response is approximately linear with the TCE concentration in the headspace above the sample. The concentration in the headspace above the sample in mg/m^3, C_H, can be calculated with the simple line equation:

$$C_H = R \times 100$$

For values of R between 0.10 and 1.0, two straight line equations relating the response to the log of concentration are used. This is called the "two point method". In this concentration range, the response is approximately linear with the log of concentration in air, suggesting a first order kinetics mechanism in which the response is a function of the rate of reaction between the halogen and the alkali metal vapor. The slopes and intercepts for the two equations must be calculated from the X-Wand response for the two gas standards. An upper line is defined between $logC_{Ha}$ and $logC_{Hb}$, where C_{Ha} is about 1,000 mg/m^3 and C_{Hb} is about 100 mg/m^3. The lower line is defined between $logC_{Hb}$ and the point where R = 0.1 and log C_H = 1.0. The line equation y = mx + b is used. Calculations for the upper and lower lines are provided below.

Upper Line Equation:

Slope = m_u = (R_a − R_b) / ($logC_{Ha}$ − $logC_{Hb}$)

Since by definition $R_a = 1.0$, this becomes

$m_u = (1.0 - R_b) / (\log_{CHa} - \log C_{Hb})$

Intercept $= b_u = y - (m_u)(x) = R_a - [(m_u)(\log C_{Ha})]$, this becomes

$b = 1.0 - [(m_u)(\log C_{Ha})]$

To calculate the concentration of TCE in the headspace above an unknown sample (x) that gives a sample response, R_x, (sample voltage / voltage for 1,000 mg/m3 standard) with a value from R_a to R_b:

$\log C_{Hx} = (R_x/m_u) - (b_u/m_u)$, then\

$CH_x = 10^{\log CHx}$

Lower Line Equation:

Slope $= m_l = (R_b - 0.10) / (\log C_{Hb} - 1.0)$

Intercept $= b_l = y - m_l x = 0.1 - m_l$

To calculate the concentration of TCE in the headspace of an unknown sample (x) that gives a sample response (R_x) that is greater than 0.1 but less than R_b:

$\text{Log} C_{Hx} = R_x/m_l - b_l/m_l$, then

$CH_x = 10^{\log CHx}$

For an ideal single straight line between R = 1.0 (1,000 mg/m^3) and R = 0.1 (10 mg/m^3), the value of R_b is 0.55. If a sensor gives an R_b value between 0.5 and 0.6, a single line equation between 10 mg/m^3 and 1,000 mg/m^3 can be used. In reality, not all the current refrigerant leak detector sensors behave identically, so the above two-line routine compensates well for these differences. The error that can be caused by not using the two-point calibration method can be calculated for three cases, as shown below.

For an unknown sample giving a response value R_x, the results vary depending on the R_b mid-point standard calibration reading. These cases assume that $\log(C_{Ha}) = 3.0$, and $\log(C_{Hb}) = 2.0$.

Case 1 $R_b = 0.75$

Upper line (R = 0.75 to 1.0)	$\log(C_{Hx}) = R_x/0.25 - 1.0$
Lower line (R = 0.1 to 0.57)	$\log(C_{Hx}) = R_x/0.65 + 0.85$

Case 2 (ideal) $R_b = 0.55$

Upper line (R = 0.55 to 1.0)

And lower line (R – 0.10 to 0.55) $\log(C_{Hx}) = R_x/0.45 + 0.78$

Case 3 $R_b = 0.35$

Upper line (R = 0.35 to 1.0) $\log(C_{Hx}) = R_x/0.65 + 1.46$
Lower line (R = 0.1 to 0.35) $\log(C_{Hx}) = R_x/0.25 + 0.60$

Applying the three cases to an unknown sample that gives an X-Wand R_x value of 0.55 can provide different results:

Case 1, the lower line is used: Calculated $C_{Hx} = 50$ mg/m^3

Case 2, the single line is used: Calculated $C_{Hx} = 100$ mg/m^3

Case 3, the upper line is used: Calculated $C_{Hx} = 200$ mg/m^3

These results indicate that calibration should always be performed with two concentrations of TCE in air, 100 mg/m^3 and 1,000 mg/m^3.

Once the calibration is performed, the concentration of TCE in the headspace above the soil can be calculated using the line equations. The mg/m^3 value is converted to concentration of TCE in soil or water using the equation below.

$Cs = C_HV_H/1000Ms$

Where Cs = concentration in the sample, mg/kg

C_H = concentration in the headspace, mg/m^3

V_H = volume of the headspace, L

Ms = mass of the sample, kg

Units of the 1000 are L/m^3

For water samples, a density of 1.0g/mL is assumed and the mass of sample in kg is considered to be identical with the volume in L, therefore mg/kg = mg/L.

Relative Response to other HVOCS

Sensitivities relative to TCE for the heated diode X-Wand prototype and eight chlorinated VOCs at a single concentration (100 mg/m^3) using a single sensor are provided in Table 1. Response is within a factor of two for the VOCs relative to the TCE response. The relative response numbers are a function of the particular sensor used and the actual HVOC concentrations measured. These must not be used to calculate concentrations of a different HVOC by using a simple response factor ratio. If calibration of the system is with TCE, any other HVOC present during screening analysis will be reported as TCE.

Henry's Law and Partition Coefficients

Prior research at WRI has involved studies of the partitioning of VOCs between air and water in a sealed system as a function of temperature and the concentration of VOC species in water (Schabron et al. 1996, Schabron and Rovani 1997). Henry's law states that the partial pressure P_i, or concentration of a volatile component in the headspace, is proportional to its concentration in the aqueous solution C_i:

$$P_i = H_i \times C_i$$

where H_i is the Henry's law constant for component i, with units of ppmv / mg/L. The assumptions in using this approach for determining VOCs are that they have not exceeded their solubility in water, and that they partition into the headspace according to Henry's law. For example, H_i relates the vapor parts per million (ppmv) level in the headspace to the mg/L concentration in water. Thus, the vapor concentration of TCE in equilibrium with a 1 mg/L aqueous TCE solution at 21 °C (70 °F) is about 70 ppmv. By measuring the ppmv of volatile organics in the headspace above aqueous solutions, field screening personnel often assume that the aqueous level can be established. However, this is an erroneous assumption since H_i is only defined at infinite dilution, and the partitioning varies significantly with total VOC water concentration and with temperature. In prior work, partition coefficients were measured for TCE at various temperatures and concentrations (Schabron et al. 1996) (Figure 3).

The Variable Volume Headspace Technique

The variable volume headspace technique can be applied only if C_H in mg/m^3 determined for the larger sample amount (50-mL sample in a 250-mL jar) is greater than the value for C_H for the corresponding smaller sample amount (25-mL sample in a 250-mL jar).

The variable volume headspace technique is based on the equation for a line plot of $1/C_H$ vs. V_H/V_S (Figure 4). The slope of the line is $1/C_O$, where C_O is the concentration of the analyte of interest in the sample in mg/m^3 compensated for equilibrium partitioning (Markalov and Bershevits 2001).

$$1/C_H = (1/Co) \cdot (V_H/V_S) + K/Co$$

Where Co = original concentration in the sample, mg/m^3

C_H = concentration in the headspace, mg/m^3

V_H = volume of the headspace, L

V_S = volume of the sample

K = partition coefficient (this is not determined in the procedure, however it must be identical for both sample portions)

This approach can compensate for equilibrium partitioning in samples only in cases where the partition coefficient is constant for both sample portions and the original concentration in each sample portion is identical. The variable volume headspace technique does not provide information as to whether or not partitioning is fully compensated for.

Theoretical Headspace Recovery Calculations

Using the partition coefficient data in Figure 3, maximum theoretical recoveries of TCE in headspace above initial aqueous solutions of 1.0 mg/L in 250-mL jars were calculated using iterative calculations. For the calculations, the two assumptions were that the partition coefficient is identical for each condition, and that the total amount of TCE in the system was constant. The results of the calculations are listed in Table 2. Different conditions were considered for the calculations to provide H_i values of 20, 70, and 120 ppmv / mg/L at sea level (1.0 atm) and at a typical 0.75 atm in Laramie, WY. Maximum theoretical recovery values in the headspace range from 48 to 95 percent. The results show that for a particular pressure and temperature, a greater percentage of TCE is found in the headspace as smaller sample sizes are screened. However, this can affect the detection limit. For example, assuming a detection limit of 0.1 mg/m^3 TCE in air, the theoretical detection limit (assuming full volatilization) for TCE for 50 mL water in a 250-mL jar is about 0.0004 mg/kg while the detection limit for a 10-mL sample is about 0.002, about five times higher.

By applying the variable volume headspace calculation to the maximum theoretical recoveries that were calculated, the original concentration of 1.0 mg/L could be determined (Table 3). Theoretically, the compensation technique works well.

Preliminary Water Screening Experiments

Preliminary testing was performed to determine the feasibility of developing a method for screening water for HVOC-contamination. High-purity, organic-free water was added to eight 250-mL glass jars having septum caps. Portions of 25-mL of water were added to four of the containers, and 50-mL portions of water were added to the other four jars. Three of the 25-mL water samples were spiked with 5 uL portions of a methanol solution of TCE to give TCE concentrations of 0.5, 1, and 5 mg/L in the water samples. Three of the 50-mL water samples were spiked with 10 uL portions of a methanol solution of TCE to give TCE concentrations of 0.5, 1, and 5 mg/L in the water samples. A 50-mL water sample with 10 uL methanol added served as a blank in the testing. The methanol level in all of the solutions was about 160 mg/L. TCE spike concentrations were verified by GC/MS analysis. After the water samples were spiked with TCE, they were swirled for one minute and then allowed to sit for 10 minutes prior to being screened using the X-Wand. Ten minutes was selected as the time for the TCE to partition into the headspace for screening, because this is the equilibration time used in the X-Wand soil method. The two-point procedure using 108 and 1,059 mg/m^3 TCE in air gas

standards was used to calculate the concentrations of TCE in the water. The results in Table 4 show recoveries ranging from 21 to 32 percent. These results are significantly below the calculated maximum recovery values in Table 2, suggesting that full equilibration had not occurred for these samples.   The results show the feasibility of extending the soil screening method to water; however, further testing will be required for the water method to determine the appropriate equilibration time, agitation procedure, and calculation method for screening water. Prior results suggest that vigorous shaking can achieve equilibrium between the air and headspace within minutes (Schabron et al 1996).

Water Samples from a Contaminated Site

A second set of experiments was performed with two TCE contaminated water samples that were being used in a TCE treatability project at WRI (Table 5). The samples were originally from F.E. Warren Air Force Base in Cheyenne, WY. Portions of these samples were poured into 250-mL septa jars and screened with the portable X-Wand device. Sample amounts were determined gravimetrically. Concurrent analysis of other sample portions was performed using purge and trap gas chromatography/mass spectrometry. The samples for screening were vigorously shaken for about 30 seconds, and allowed to sit in the laboratory for about 2 hours prior to screening to ensure that they were all at the same temperature as the laboratory.

X-Wand calibration was with Scott Specialty Gases custom TCE standards at 221 ppmv and 22.3 ppmv in air. Using the laboratory temperature of 78 ºF and the atmospheric pressure in Laramie of 0.781 atm, this converts to 926 mg/m^3 and 93.5 mg/m^3, respectively. The average reading for the 926 mg/m^3 standard was 10.076V, and this was the reference voltage for R = 1.000. The average reading for the 93.5 mg/m^3 standard was 5.126V, R = 0.5087. Replicate readings varied by less than 5%.

The two-point calibration was used to interpret the data from the samples. Recovery values for the initial screening analysis ranged from 41 to 93 percent, indicating that vigorous shaking results in more rapid equilibrium partitioning than swirling. The variable volume headspace calculation was applied to the X-Wand results from each sample pair, since it was assumed that the partition coefficient was the same for each of the sample pairs, and that the exchange mechanism between air and water is due only to partitioning.   The results show that the incomplete partitioning into the headspace can be compensated for using the variable volume headspace calculation (Table 5). The successful application of the variable volume technique indicates that partitioning equilibration had occurred for these samples prior to screening, and that the partition coefficient was indeed the same for each sample pair.

New ASTM Standard

In March 2004, ASTM Committee D 34 on Waste Management approved the

development of a new draft ASTM standard for screening soils for halogenated VOCs using a heated diode sensor. The standard was written and submitted for initial D 34.01 subcommittee technical review balloting in October 2004. The method was revised based on reviewer comments and committee instructions. The revised draft method was submitted to ASTM for Subcommittee D 34.01 balloting in April 2005. Additional comments were received, and a revised version was submitted for concurrent balloting within ASTM Subcommittee D 34.01 and ASTM Main Committee D 34 in August 2005. There were no negative votes, and the method was approved in September 2005. The draft method that was approved in September 2005 is provided in the Appendix. The method was approved as ASTM D 7203-05, Standard Test Method for Screening Trichloroethylene (TCE)-Contaminated Soil Using a Heated Diode Sensor.

CONCLUSIONS

Commercially available heated diode leak detectors were evaluated further for screening for HVOCs. Sensor responses to eight chlorinated VOCs relative to the response of TCE were obtained. The limit of detection (S/N = 2) for the heated diode sensor is about 0.1 mg/m^3 TCE. The maximum reading is obtained at about 1,000 mg/m^3 TCE. Sensor to sensor variability, or changes within a sensor over time can be compensated for by normalizing sensor readings to a maximum sensor reading at 1,000 mg/m^3 TCE and obtaining an additional calibration reading at 100 mg/m^3. The soil screening method was submitted as a draft for ASTM balloting. Experiments were performed to expand the soil method for screening water samples. Assuming complete volatilization into headspace, the detection limit for TCE in soil or water is about 1 ug/kg (ppb) for a 25-g sample in an 8-oz jar.

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Table 1. Approximate Responses Relative to TCE With Prototype X-Wand at 100 mg/m^3, Single Sensor

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Compound	Relative Response
Dichloromethane	1.2
Cis-1,1-Dichloroethylene	1.2
trans-1,2-Dichloroethylene	1.4
1,2 Dichloroethane	1.1
1,1,1-Trichloroethane	1.5
1,1,2-Trichloroethylene (TCE)	1.0
Trichloromethane	2.1
Tetrachloroethylene (PCE)	0.67
Carbon Tetrachloride	1.6

_____

Table 2. Theoretical 1.0 mg/L TCE Partition into Headspace from Water in a 250-mL Jar

Volume, mL	P, atm.	T, ºF	H_i, ppmv/mg/L	Percent of Total in Headspace	mg/m^3 in Headspace
50	1.0	70	70	60	150
25	1.0	70	70	78	85
5.0	1.0	70	70	95	19
50	0.75	70	70	51	130
25	0.75	70	70	72	80
5.0	0.75	70	70	93	19
25	1.0	70	20[a]	48	56
25	1.0	90	120	85	93

a. Lower K with other organic compounds present

Table 3. Theoretical Variable Volume Headspace Workup for 1.0 mg / L TCE in 250-mL Jar, 1.0 atm, 70 ºF , H_i = 70 ppmv / mg/L (From Table 2 Data)

Sample Volume, mL	Calculated C_H, mg/m^3	V_H, cc	V_S, cc	V_H / V_S	$1 / C_H$
a. 50	150	200	50	4.0	0.00667
b. 25	85	225	25	9.0	0.0118
c. 5.0	19	245	15	29	0.526

$$1 / C_H = (1 / C_O)(V_H / V_S) + K / C_O$$

Slope = $1 / C_O$ Calculations:

ab: = 0.00103 , C_O = 970 mg/m^3 = 0.97 mg/L

ac: = 0.00102 , C_O = 980 mg/m^3 = 0.98 mg/L

bc: = 0.00102 , C_O = 980 mg/m^3 = 0.98 mg/L

Table 4. Preliminary TCE Spiking Experiments with Water Using the Two Point Calibration and the Variable Volume Headspace Technique; Samples Swirled Only

	TCE Concentration, mg/L		
Volume, mL	Spike Level	Headspace Screening	% Recovery
25	0.52	0.15	29
50	0.52	0.11	21
25	1.1	0.29	26
50	1.1	0.27	24
25	5.3	1.7	32
50	5.3	1.1	21
50 mL Water Blank, 10 uL MeOH (with 160 mg/L methanol)		0.006	

Table 5. Screening and GC/MS Results from F.E. Warren Air Force Base Site; Samples Shaken Vigorously

Sample ID	Volume, mL	R	TCE Concentration, mg/L		
			Headspace Screening	Variable Volume	GC/MS
151	18.30.	0.687	2.8		
	48.2	0.887	2.3		
				3.0	3.22
190	28.5	0.214	0.14		
	58.3	0.295	0.095		
				0.23	0.230
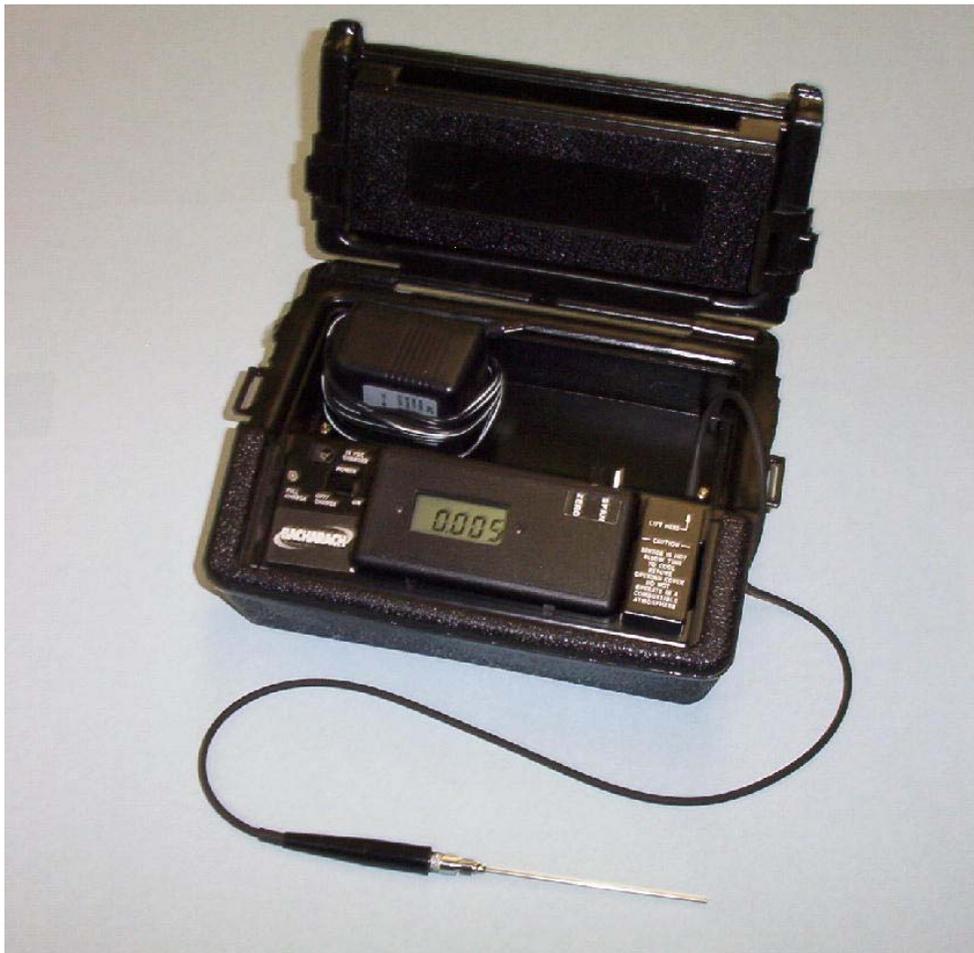
**Figure 1. X-Wand Heated Diode Prototype**
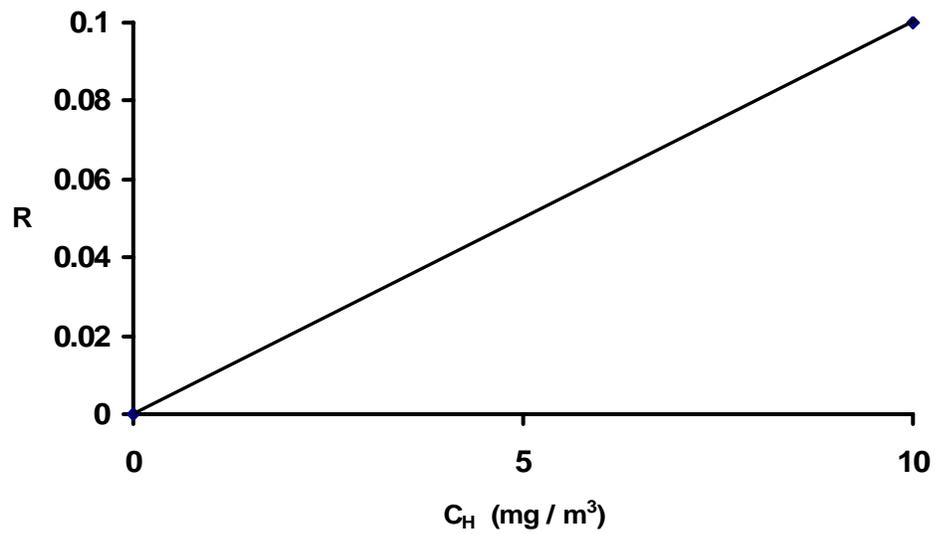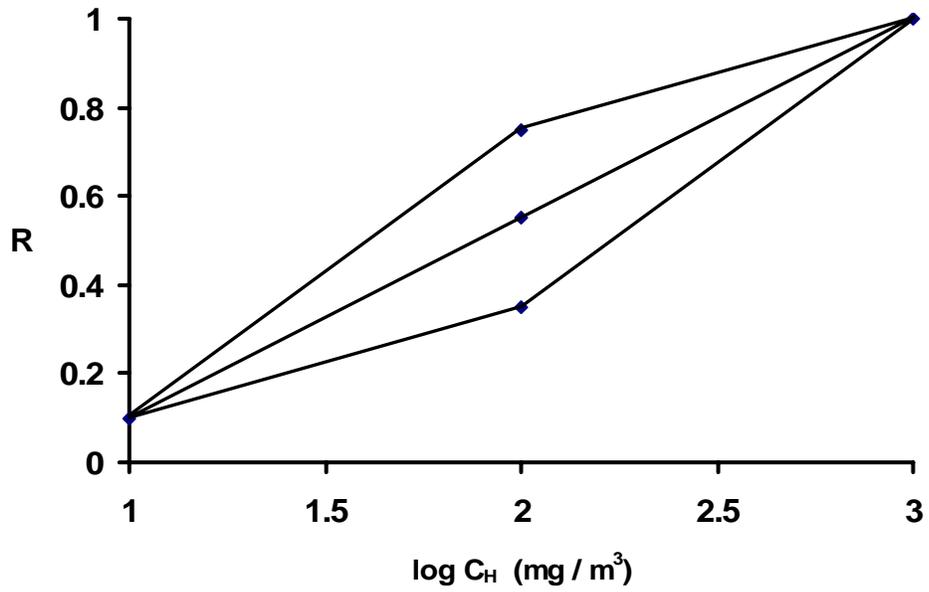
Figure 2. TCE Calibration Profiles from 10 to 1,000 mg/m^3 Showing 2-point Compensation for Sensor to Sensor Variability (top) and Linear Response below 10 mg/m^3 (bottom)
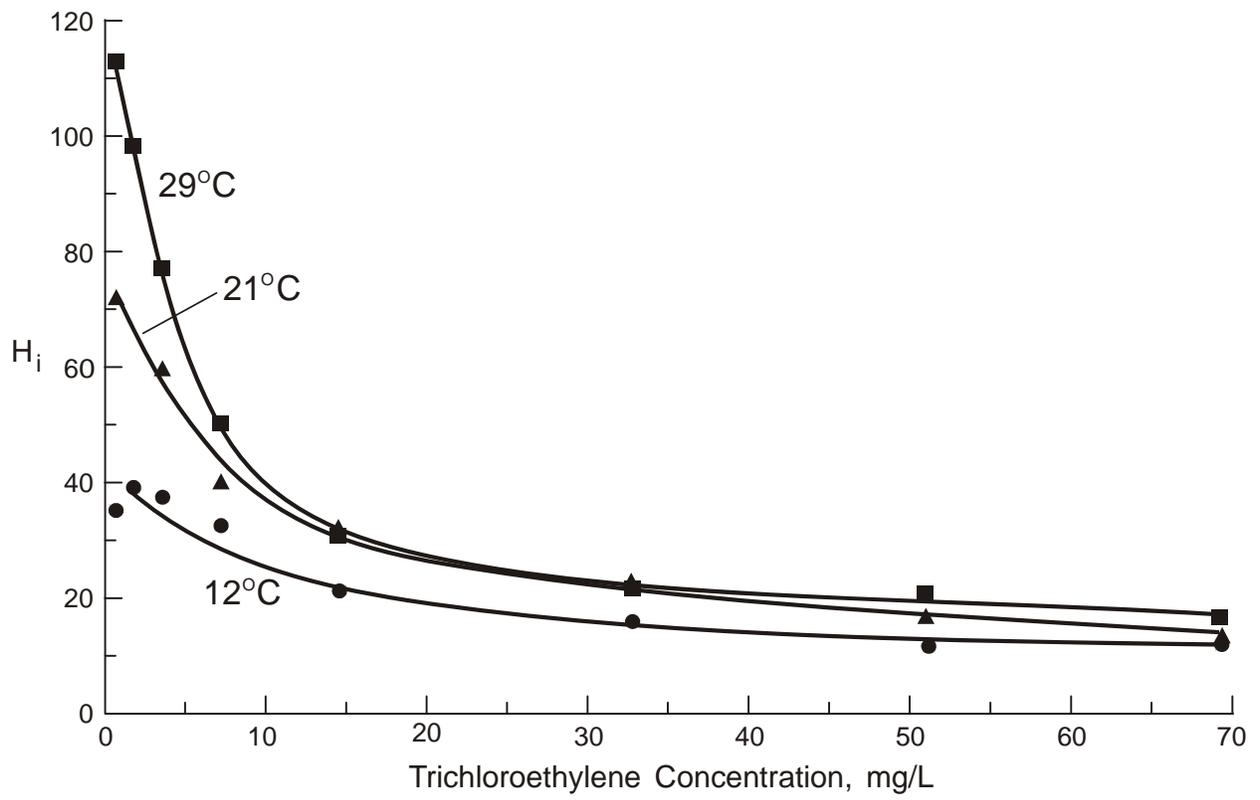
Figure 3. Partition Coefficient Profiles for TCE in Water
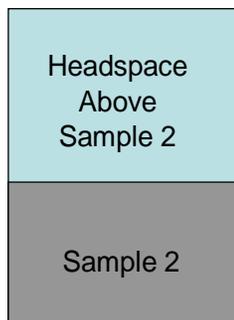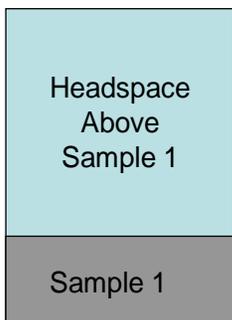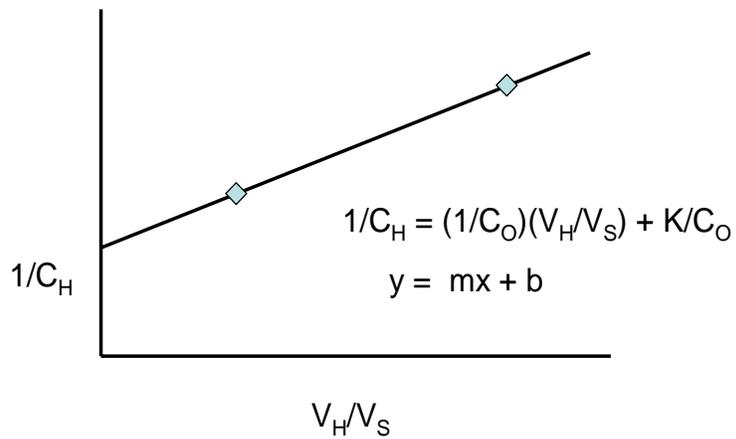
$$1/C_H = (1/C_O)(V_H/V_S) + K/C_O$$

$$y = mx + b$$

C_O = original concentration in the sample, mg/m^3
C_H = concentration in the headspace, mg/m^3
V_H = volume of the headspace, L
V_S = volume of the sample, L
K = partition coefficient, concentration in sample/concentration in headspace

Figure 4. Variable Volume Headspace Technique

APPENDIX

DRAFT ASTM METHOD

Standard Test Method for Screening Trichloroethylene (TCE)-Contaminated Soil Using a Heated Diode Sensor[1]

1. Scope

1.1 This test method describes a procedure for screening soil known to contain the halogenated volatile organic compound (HVOC), trichloroethylene (TCE), by measuring the TCE concentration in the headspace above a sample of the soil using a heated diode sensor device. From this measurement, an estimated concentration of TCE in the soil can be determined. Any TCE remaining in the soil sample is not measured by this method. Any other HVOC present in the soil will be reported as TCE.

1.2 This method can also be used for screening the headspace above a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination in the soil. Any HVOC contamination remaining in the soil is not detected by this method.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the application of regulatory limitations prior to use.

NOTE 1—The diode sensor is heated to temperatures ranging between approximately 600 and 1,000 $^\circ$C (see section 6.6) and as a result could be a source of ignition.

2. Referenced Documents

2.1 ASTM Standards:[2]

 D 4547 Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

3. Terminology

3.1 *Definitions*–For definitions of terms used in this screening test method, refer to Terminology E 131.

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[1] This method is under the jurisdiction of ASTM Committee D 34 on Waste Management and is the direct responsibility of Task Group D 34.01.05 on Screening Methods.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org.   For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4. Summary of Test Method

4.1 To estimate the concentration of TCE in a soil known to contain TCE contamination, a sample of the soil is added to a glass jar having an open-top cap with a PTFE-bonded silicone septum. At the time of screening, the temperature of the soil in the jar should be approximately 50 to 120 $^{\circ}$F (10 to 49 $^{\circ}$C). The soil in the jar is shaken and allowed to settle for 10 minutes, so the TCE can partition into the headspace above the soil. After 10 minutes, the TCE concentration in the headspace is measured using a heated diode sensor device, which gives a numerical voltage reading. The voltage reading from the device is converted to a mg/m^3 value of TCE in the headspace in the container. Using this value, an estimated concentration of TCE in the soil in mg/Kg can be calculated. Any TCE remaining in the soil sample is not measured by this method. Any other HVOC present in the soil will be reported as TCE.

4.2 To use this method to screen a soil suspected of containing HVOC contamination, a sample of the soil is added to a glass jar having an open-top cap with a PTFE-bonded silicone septum. At the time of screening, the temperature of the soil in the jar should be approximately 50 to 120 $^{\circ}$F (10 to 49 $^{\circ}$C). The soil in the jar is shaken and allowed to settle for 10 minutes, so the HVOC can partition into the headspace above the soil. After 10 minutes, the heated diode sensor device is used to screen the headspace in the container. The numerical voltage reading from the device indicates the presence or absence of HVOC contamination in the soil. Any HVOC contamination remaining in the soil is not detected by this method.

5. Significance and Use

5.1 The heated diode sensor device used in this method is selective for HVOCs. Other electronegative compounds, such as alcohols, ketones, nitrates, and sulfides, may cause a positive interference with the performance of the heated diode sensor to detect HVOCs, but to do so they must be present at much higher concentrations than the HVOCs.

NOTE 2—For volatile organic compound (VOC) screening purposes, a flame ionization detector (FID) selectively responds to flammable VOCs; a photoionization detector (PID) selectively responds to VOCs having a double bond; and a heated diode sensor selectively responds to halogenated VOCs.

5.2 This method can be used for screening soil known to contain TCE to estimate the concentration of TCE in the soil. The method measures the TCE concentration in the headspace above a sample of the TCE-contaminated soil. An estimated concentration of TCE in the soil can be determined. Any TCE remaining in the soil sample is not measured by this method. Any other HVOC present in the soil will be reported as TCE.

5.3 This method can also be used for screening the headspace above a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination in the soil. Any HVOC contamination remaining in the soil is not detected by this method.

5.4 The quantitation limit of the screening method is dependent on the soil-to-headspace ratio in the sample container and the HVOC in the soil. For a 25-gram TCE-contaminated soil sample in a 250-mL container, the quantitation limit for TCE is 0.005 mg/Kg, based on a signal-to-noise ratio of 10.

5.5 The detection limit of the heated diode sensor for TCE is 0.1 mg/m^3 in air, based on a signal-to-noise ratio of 2. For a 25-gram TCE-contaminated soil sample in a 250-mL container, the detection limit of the screening method for TCE is 0.001 mg/Kg, assuming complete partitioning of TCE into the headspace.

5.6 This method can be used to screen moist soil samples. Water vapor does not interfere with the performance of the heated diode sensor.

5.7 Hydrocarbon fuels, including fuels containing aromatic compounds, such as gasoline, are not detected by the method.

6. Apparatus

6.1 *Metal or rigid plastic coring tools*, designed for collecting and transferring a 25-g soil VOC sample (see Guide D 4547 and section 8.1).

6.2 *Glass Jars*, 250-mL (8-oz), approximately 14 cm (5½ inches) tall, with open-top caps having PTFE-bonded silicone septa.

6.3 *Scale*, capable of weighing to 0.1 g.

6.4 *Thermometer*, with temperature given in divisions of 0.1 ℃

6.5 *Barometer*, such that pressure in atmospheres can be determined to 0.001 atm.

6.6 *Heated Diode Sensor Device*, a device having a diode sensor that is heated between temperatures ranging from approximately 600 to 1,000 ℃ generating an alkali metal vapor stream that selectively reacts with halogens present in HVOC molecules, creating ionized product species that cause a current to flow between a cathode and an anode. The numerical output from the sensor in volts is proportional to a microamp current from the diode and ranges from 0.001 to 20 volts with a resolution of 0.001 volt. The HVOC molecules in the headspace above the soil sample are drawn through a probe to the heated diode sensor by a pump in the device. The heated diode sensor device should have a needle attached to the probe of the device so the septum in the cap of the sample jar can be pierced and the needle can be inserted into the headspace above the sample. This needle must be designed to allow make-up air to enter the

sample jar from the top so that back pressure will not build up within the jar. Back pressure in the jar will change the air flow rate of the device and in turn affect the voltage reading.

6.7 *Tedlar Bags*, 1-L in volume and having a stainless steel valve with a nipple fitting that can be opened and closed.

6.8 *Gas Regulators*, for use with the TCE standard gas cylinders (see section 7.1). Each regulator should have a short length, about 1¼-inch, of ¼-inch inner diameter Viton tubing attached to the nipple fitting.

7. Reagents and Materials

7.1 *TCE Standard Gas Cylinders*—These are transportable cylinders containing certified concentrations of TCE in air pressurized to about 320 psi. If the method is being used to screen a TCE-contaminated soil to estimate the concentration of TCE in the soil, two concentrations of TCE are required. One concentration is 220 ± 10 vapor part per million (ppmv) TCE in air. This is the high concentration TCE standard gas. The other concentration is 22 ± 1 ppmv TCE in air, which is the mid concentration TCE standard gas. These concentrations correlate with the upper and mid range of sensor response for the device. A 220 ppmv TCE standard gas at 25 ºC corresponds to about 890 mg/m^3 TCE in air at 0.75 atm of pressure and to about 1,200 mg/m^3 TCE in air at 1 atm of pressure. A 22 ppmv TCE standard gas at 25 ºC corresponds to about 90 mg/m^3 TCE in air at 0.75 atm of pressure and to about 120 mg/m^3 TCE in air at 1 atm of pressure. See Notes 3 and 4. If the method is being used to screen a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination, only the high concentration TCE standard gas, 220 ± 10 ppmv TCE in air, is required.

NOTE 3—For HVOC concentrations in air that are greater than 10 mg/m^3, the current that is generated by the reaction between the halogen and the alkali metal vapor when the sensor is exposed to the HVOC is a function of the log of the concentration of the halogen in air. The log of 890 is 2.9, and the log of 1,200 is 3.1, showing that the log values of these concentrations vary only slightly. Similarly, the log of 90 is 1.9, and the log of 120 is 2.1. These values show that the log of the TCE concentration in air varies only slightly at different elevations (atmosphere of pressure).

NOTE 4—The mg/m^3 concentrations of the TCE in the standard gas cylinders can be calculated using the ppmv concentrations of TCE in the cylinders, the atmospheric pressure, the temperature, and equations 5 and 6, which are given in section 13.6.

7.1.1 Transportation of the TCE gas cylinders must comply with current Department of Transportation (DOT) regulations.

8.   Sample Collection and Preparation

8.1 Collect a soil sample of approximately 25 g using a metal or rigid plastic coring tool. See D 4547 for recommended devices. If the sample will be stored in the coring tool prior to screening, it should be collected using a coring tool designed for sample storage as described in D 4547 and should be stored as specified in D 4547.

8.2 Preweigh a 250-mL glass jar with an open-top cap having a PTFE-bonded silicone septum. Record the mass of the jar with the cap to ±0.1 g.

8.3 Extrude the soil sample from the coring tool into the pre-weighed 250-mL glass jar and immediately seal the jar making sure that there are no soil particles on the sealing surfaces.

8.4 Weigh the jar-plus-soil sample and record the mass of the jar-plus-soil sample to ±0.1 g.

8.5 Determine the mass of soil added to the jar, and record the mass of the soil sample to ±0.1 g.

8.6 The temperature of the soil in the sample jar should be approximately 50 to 120 $^{\circ}$F (10 to 49 $^{\circ}$C) prior to screening the sample using the heated diode sensor, so that HVOC partitioning into the headspace above the sample will occur (see Note 5). The sample jar should not be opened to determine the soil temperature. The ambient temperature where the screening is to be performed should be in the range of 50 to 120 $^{\circ}$F (10 to 49 $^{\circ}$C), and the soil sample should be allowed to come to approximately that temperature prior to screening (section 12).

NOTE 5—The temperature at which the screening is performed may affect the HVOC concentration in the headspace. For example, the vapor pressure of TCE at 120 $^{\circ}$F (49 $^{\circ}$C) is about seven times greater than the vapor pressure of TCE at 50 $^{\circ}$F (10 $^{\circ}$C). Therefore, more TCE would be expected in the headspace at higher temperatures.

NOTE 6—The ambient temperature where the screening is to be performed will be recorded as specified in section 11.3

9. Operation of the Heated Diode Sensor Device

9.1 The heated diode sensor device used to perform this method should be calibrated and operated according to the manufacturer's instructions.

9.2 After a sample or standard gas has been screened using the heated diode sensor device, the voltage reading of the device must be allowed to return to zero, indicating that the sensor has been flushed with intake air so that any contamination from the previous sample or standard gas has been removed from the system.

10. Preparation of Tedlar Bags Containing the TCE Standard Gases

10.1 If the method is being used to screen a TCE-contaminated soil to estimate the concentration of TCE in the soil, a 1-L Tedlar bag containing each of the TCE standard gases (section 7.1) should be prepared. If the method is being used to screen a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination, a 1-L Tedlar bag containing the high concentration TCE standard gas, 220 ± 10 ppmv TCE in air, (section 7.1) should be prepared.

10.1.1 To prepare a Tedlar bag containing the TCE standard gas, attach a regulator (see section 6.8) to the TCE standard gas cylinder.

10.1.2 Open the valve on a 1-L Tedlar bag. Press the bag against a flat, hard surface to make sure the bag is empty.

10.1.3 With the valve on the Tedlar bag in the open position, attach the bag to the regulator by connecting the nipple fitting on the valve with the tubing on the regulator. Carefully fill the Tedlar bag with the standard gas. Remove the bag from the regulator. With the valve on the Tedlar bag in the open position, once again press the bag against a flat, hard surface to empty the bag. After the bag has been flushed with the standard gas, it should be refilled with the standard gas, as described above, and the valve on the bag should be closed.

11. Heated Diode Sensor Device Calibration Check and Voltage Measurement of TCE Standard Gases

11.1 After the heated diode sensor device has been calibrated (see section 9.1), open the valve on the Tedlar bag containing the high concentration TCE standard gas and connect the nipple fitting on the bag to the probe of the heated diode sensor device. The device is calibrated if the voltage reading for the high concentration TCE standard gas is between 9 and 12 volts. If the voltage reading is 9 volts or less or 12 volts or greater, the device should be re-calibrated until the reading for the high concentration TCE standard gas is between 9 and 12 volts. Record this voltage reading from the device for the high concentration TCE standard gas as $V_{HighTCEStd}$.

11.2 If the method is being used to screen a TCE-contaminated soil to estimate the concentration of TCE in the soil, a voltage reading for the mid concentration TCE standard gas is required. This reading is made after it is determined that the heated diode sensor device is calibrated (section 11.1), the voltage reading from the device for the high concentration TCE standard gas ($V_{HighTCEStd}$) has been recorded, and the voltage reading of the heated diode sensor device has returned to zero. The valve on the Tedlar bag containing the mid concentration TCE standard gas (section 10) should be opened and the nipple fitting on the bag should be connected to the probe of the heated diode sensor device. The voltage reading from the device for the mid

concentration TCE standard gas should be recorded as $V_{MidTCEStd}$. If the method is being used to screen a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination, a voltage reading for the mid concentration TCE standard gas is not required

11.3 If the method is being used to screen a TCE-contaminated soil to estimate the concentration of TCE in the soil, record the temperature in $^{\circ}$C to 0.1 $^{\circ}$C and pressure in atmospheres to 0.001 atm at the time that the voltage readings for the high concentration and mid concentration TCE standard gases are recorded. If the method is being used to screen a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination, record the temperature in $^{\circ}$C to 0.1 $^{\circ}$C at the time that the voltage reading for the high concentration TCE standard gas is recorded.

11.4 The voltage reading of the high concentration TCE standard gas should be checked after 10 samples have been screened, or if the sensor is exposed to a sample that gives a voltage reading above the reading for the high concentration TCE standard gas, $V_{HighTCEStd}$, recorded in section 11.1 (see Note 7). If during this check, the reading for the high concentration TCE standard gas is not within 10% of the value recorded for the standard gas during the previous check, the device must be re-calibrated.

NOTE 7—If a sensor is exposed to a sample that gives a voltage reading above the reading for the high concentration TCE standard gas, VHighTCEStd, indicating that the concentration of TCE in the headspace above the sample is greater than the TCE concentration in the high concentration standard gas, the sensor response may become unstable.

12. Sample Screening

12.1 When the sample temperature is estimated to be 50 to 120 $^{\circ}$F (10 to 49 $^{\circ}$C), shake the jar-plus-soil sample for approximately 20 seconds. Allow the jar to sit for a minimum of 10 minutes for HVOC partitioning into the headspace above the soil sample.

12.2 After 10 minutes, insert the needle attached to the probe of the heated diode sensor device through the septum in the cap of the sample jar and into the headspace above the sample. The tip of the needle should be approximately 3 inches below the cap of the jar. The tip of the needle should never contact the surface of the soil. The voltage reading for the headspace above the soil sample should be recorded as V_s.

12.3 If the method is being used to screen a soil suspected of containing HVOC contamination, the voltage reading for the headspace above the 25-g soil sample, V_s, determined above in section 12.2 can be used to indicate the presence or absence of HVOC contamination in the soil (see Note 8). If the method is being used to screen a TCE-contaminated soil to estimate

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the concentration of TCE in the soil, the calculations provided in section 13 should be performed.

NOTE 8—The voltage reading of a heated diode sensor device is specific to the particular device, the particular sensor, and the amount of use the sensor has encountered. For this reason, the voltage reading for a soil sample, V_s, can be used to indicate the presence or absence of HVOC contamination; however, to compare readings made during different screening episodes, the relative response values for the samples, R_s, (see section 13.4) should be calculated and compared.

13. Calculations

13.1 Data interpretation in this method is based on the response characteristic of the particular sensor used in the device. Figure 1 is a, qualitative plot of the response, R, versus the base 10 log of the concentration of TCE in air, $\log C_H$ (mg/m^3). This figure shows three possible heated diode sensor response profiles for a concentration of 100 mg/m^3 TCE in air ($\log C_H$ = 2.00): R greater than 0.550 (profile 1); R equal to 0.550 (profile 2); and R less than 0.550 (profile 3). These profiles are specific to TCE and any heated diode sensor meeting the specifications given in section 6.6.

13.2 Calculate the relative response for the mid concentration TCE standard gas, $R_{MidTCEStd}$, using the voltage reading for the mid concentration TCE standard gas, $V_{MidTCEStd}$ (section 11.2), and the voltage reading for the high concentration TCE standard gas, $V_{HighTCEStd}$ (section 11.1), as shown in equation 1.

$$R_{MidTCEStd} = V_{MidTCEStd} / V_{HighTCEStd} \tag{1}$$

13.3 The relative response, $R_{HighTCEStd}$, for the high concentration TCE in air standard gas is equal to 1.00, based on the calculation shown in equation 2.

$$R_{HighTCEStd} = V_{HighTCEStd} / V_{HighTCEStd} = 1.00 \tag{2}$$

13.4 Calculate a relative response, R_s, for the sample using the voltage reading for the 25-g sample (V_s, section 12.2) and the voltage reading for the high concentration TCE in air standard ($V_{HighTCEStd}$) as shown in equation 3.

$$R_s = V_s / V_{HighTCEStd} \tag{3}$$

13.5 If R_s calculated in section 13.4 is less than or equal to 1.00, go to section 13.6 and continue the calculations. If R_s is greater than 1.00, then the concentration of TCE in the headspace above the sample is greater than the TCE concentration in the high concentration standard gas. The concentration of TCE in mg/m^3 in the high concentration TCE standard gas should be calculated using equation 5 shown in section 13.6. Then the estimated concentration

of TCE in the soil sample, C_s, mg/Kg, should be reported as the greater than value calculated using equation 4 shown below.

C_s, mg/Kg > ($C_{HighTCEstd}$, mg/m^3)(Vol. of headspace in the container, L)/(Kg of soil)(1000 L/m^3)

(4)

Where: $C_{HighTCEstd}$, mg/m^3, is the concentration of TCE in the high concentration standard gas, calculated using Eq 5 in section 13.6;

Vol. of headspace in the sample container, L, is 0.235 L for a 25.0-g sample in a 250-mL jar, assuming a soil density of 1.7 g/mL;

Mass of soil sample in Kg: for a 25.0-g sample, this is 0.025 Kg.

13.6 Calculate the concentration of TCE in mg/m^3 in the high concentration and mid concentration TCE in air standard gas cylinders using equations 5 and 6, respectively.

$C_{HighTCEStd}$, mg/m^3 = (ppmv$_{HighTCEStd}$) (131.39 g/mol) (P) / (R) (T)　　　　　(5)

$C_{MidTCEStd}$, mg/m^3 = (ppmv$_{MidTCEStd}$ (131.39 g/mol) (P) / (R) (T)　　　　　(6)

where: ppmv$_{HighTCEStd}$ = the TCE concentration in the high concentration TCE standard gas in ppmv ppmv$_{MidTCEStd}$ = the TCE concentration in the mid concentration TCE standard gas in ppmv

P = pressure in atmospheres (recorded in section 11.3)
R = the gas constant, 0.082056 ppmv L atm/mol $^{\circ}$K
T = temperature in $^{\circ}$K (T $^{\circ}$C + 273.15) (T $^{\circ}$C recorded in section 11.3)

13.7 To interpret the data, equations corresponding to specific line segments, such as those shown in Figure 2 for concentrations of TCE in the headspace above the soil sample (C_H) ranging from greater than 10 to 1,000 mg/m^3, and the line shown in Figure 3 for concentrations of TCE in the headspace above the soil sample (C_H) ranging from 0 to 10 mg/m^3, are used depending on the relative response for the sample, R_s.

13.7.1 If the relative response, R_s, for the sample (calculated in section 13.4) is equal to or between the relative response values, $R_{MidTCEStd}$ and $R_{HighTCEStd}$ (1.00) (Eq 1 and Eq 2, respectively), calculate the slope and y intercept of the line segment appropriate for the sample response (segments a shown in Figure 2) using equations 7 and 8. Then calculate the base 10 log of the concentration of TCE in the headspace above the soil sample (log C_H) using equation 9.

slope, m_a = (1.00 – $R_{MidTCEStd}$) / (log $C_{HighTCEStd}$ – log $C_{MidTCEStd}$)　　　　　(7)

y intercept, b_a = 1.00 - [(m_a) (log $C_{HighTCEStd}$)]　　　　　(8)

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$$\log C_H = (R_s/m_a) - (b_a/m_a) \tag{9}$$

13.7.2 If the relative response, R_s, for the sample (calculated in section 13.4) is between $R_{midTCEStd}$ (Eq 1) and 0.100, calculate the slope and y intercept of the line segment appropriate for the sample response (segments b shown in Figure 2) using equations 10 and 11. Then calculate the base 10 log of the concentration of TCE in the headspace above the soil sample (log C_H) using equation 12.

$$\text{slope, } m_b = (R_{MidTCEStd} - 0.100) / (\log C_{MidTCEStd} - 1.00) \tag{10}$$

$$\text{y intercept, } b_b = 0.100 - m_b \tag{11}$$

$$\log C_H = (R_s/m_b) - (b_b/m_b) \tag{12}$$

13.7.3 If the relative response, R_s, for the sample (calculated in section 13.4) ranges from 0 to 0.100 (line shown in Figure 3), calculate the concentration of TCE in the headspace above the soil sample (C_H) in mg/m^3 using equation 13.

$$C_H = (R_s) (100) \tag{13}$$

13.7.4 If a log C_H value was determined in either section 13.7.1 or 13.7.2, the concentration of TCE in the headspace above the soil sample (C_H) in mg/m^3 can be calculated using equation 14.

$$C_H = 10^{\log CH} \tag{14}$$

13.8 Using the C_H value calculated in either section 13.7.3 or 13.7.4, the estimated concentration of TCE in the soil sample, C_s, in mg/Kg, can be calculated using equation 15.

$$C_s, \text{mg/Kg} = (C_H, \text{mg/m}^3) \text{ (Vol. of headspace in the container, L) / (Kg of soil) (1000 L/m}^3) \tag{15}$$

Where: C_H, mg/m^3, is the concentration of TCE in the headspace above the soil sample determined in section 13.7.3 or in section 13.7.4; Vol. of headspace in the sample container, L, is 0.235 L for a 25.0-g sample in a 250-mL jar, assuming a soil density of 1.7 g/mL; Mass of soil sample in Kg: for a 25.0-g sample, this is 0.025 Kg.

14. Record

14.1 Record the following information:

14.1.1 Mass of the empty 250-mL sample jar with the cap, g,

14.1.2 Mass of the sample jar with cap-plus-soil sample, g,

14.1.3 Mass of the soil sample, g,

14.1.4 Voltage reading for the high concentration TCE standard gas, $V_{HighTCEStd}$,

14.1.5 Voltage reading for the mid concentration TCE standard gas, $V_{MidTCEStd}$ (see Note 9),

14.1.6 Temperature in $^{\circ}$C and pressure in atmospheres at the time that the voltage readings for the mid concentration and high concentration TCE standard gases are recorded (see Note 9), and

14.1.7 Voltage reading for the headspace above the 25-g soil sample, V_s

NOTE 9—If the method is being used to screen a soil suspected of containing HVOC contamination to indicate the presence or absence of HVOC contamination in the soil, a reading for the mid concentration TCE standard gas, $V_{MidTCEStd}$, and a pressure reading at the time that the voltage reading for the high concentration TCE standard gas is recorded are not required.

15. Report

15.1 If the test method is used to screen soil known to contain TCE to estimate the concentration of TCE in the soil, report the estimated TCE concentration in mg/Kg to two significant figures. Any other HVOC present in the soil will be reported as TCE.

15.2 If the method is used for screening soil to indicate the presence or absence of HVOC contamination, report the voltage reading recorded for the soil and the indicated presence or absence of HVOC contamination.

16. Precision and Bias

16.1 *Precision*—Data on the precision of this screening method are to be generated in the near future.

16.2 *Bias*—No information can be given on the bias of the screening method because there is no standard reference material for screening soil for TCE in the field; however, data have been generated to provide information on the performance of the method to screen soil collected at a TCE-contaminated site. The screening method was evaluated based on a comparison of the screening data with data generated from analysis of samples collected side-by-side from the same location and analyzed in the laboratory using gas chromatography/mass spectrometry (GC/MS). These data are shown in Appendix X1[3].

17. Keywords

13.1 Halogenated volatile organic compounds (HVOCs); heated diode sensor; trichloroethylene (TCE); field screening; soils

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[3]A copy of the research report on the study described in Appendix X1 is available from ASTM Headquarters. Request RR: D34-XXXX.
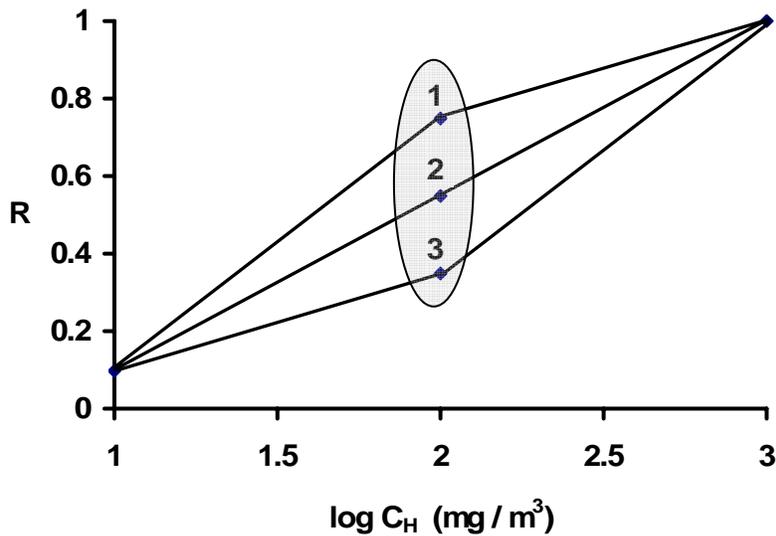
Figure 1. Qualitative heated diode sensor response profiles for TCE headspace concentrations (C_H) ranging from greater than 10 to 1,000 mg/m^3 showing three possible R values for C_H = 100 mg/m^3 (log C_H = 2.00)
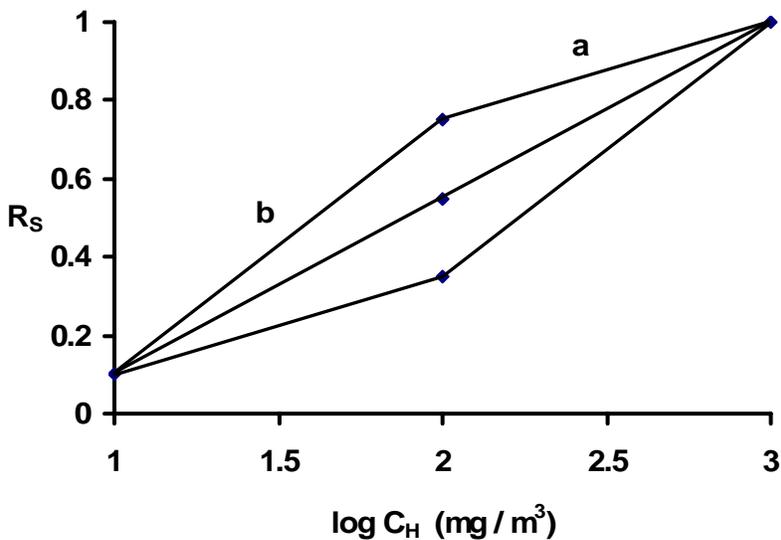
Figure 2. Qualitative heated diode sensor response profiles for sample TCE headspace concentrations (C_H) ranging from greater than 10 to 1,000 mg/m^3 showing specific line segments for TCE concentrations equal to or between 100 and 1,000 mg/m^3 (line segments a) and TCE concentrations between 10 and 100 mg/m^3 (line segments b)
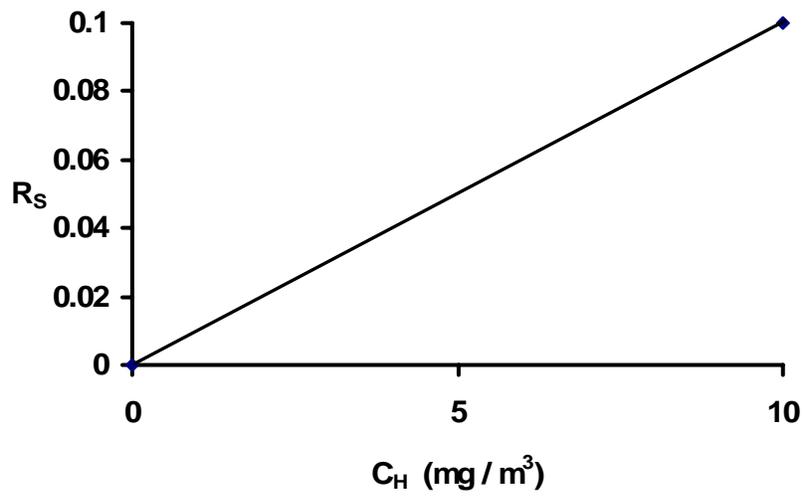
**Figure 3. Heated diode sensor response profile for sample TCE headspace concentrations (C_H) ranging from 0 to 10 mg/m^3**

APPENDIX

(Non-mandatory Information)

X1. Application of the Method for Screening Trichloroethylene (TCE)-Contaminated Soil Using a Heated Diode Sensor to Soil Collected at a TCE-Contaminated Field Site

X1.1 A study was conducted to evaluate the performance of the method to screen soil collected at a TCE-contaminated site. The screening method was evaluated by comparing the screening data with data generated from analysis of samples collected side-by-side from the same location and analyzed in the laboratory using gas chromatography/mass spectroscopy (GC/MS).

X1.1.1 The site selected for the study was contaminated with TCE more than 20 years ago. Contamination is near the soil surface and is known to be relatively uniform so that adjacent samples having similar contaminant levels can be collected. The soil at the site is characterized as a cohesive silty-clay with an organic carbon content of less than 1.0% and ranging in moisture from 10 to 20%.

X1.1.2 For the study, contaminated soil samples were collected at depths ranging from approximately 2 to 12 inches at 18 locations within the site. At each of the contaminated locations, three side-by-side samples were collected in 25-gram En Core® samplers to evaluate the screening method. One of these samples was placed in a cooler for shipment to a commercial laboratory for TCE analysis by GC/MS; one of these was placed in a cooler for shipment to a research laboratory for TCE analysis by GC/MS; and one of the samples was placed in a third cooler for on-site screening. The sample coolers contained frozen cold packs to maintain cooler temperatures at 4 ± 2 °C during sample storage.

X1.1.3 In addition to the 18 contaminated locations; two locations believed to be uncontaminated were also sampled. Three side-by-side samples were collected at each of these locations, two samples for laboratory analysis and one sample for on-site screening.

X.1.1.4 After sample collection, the samples for laboratory analysis were shipped for next day delivery to the laboratories. Each cooler contained a mini digital temperature data logger so storage temperatures could be monitored. Upon receipt by the laboratories, the samples were extruded from the En Core samplers and analyzed for TCE using EPA SW-846 Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)[4.] SW-846

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[4] U.S. EPA, 1996, Method 8260B: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol 1B, Final Update III. Method 5035A, Closed-System Purge-And-Trap and Extraction for Volatile Organics in Soil and Waste Samples[5] was used as the sample introduction technique for analysis.

X1.1.5 On-site screening was performed in a laboratory at the site. The soil samples were extruded into 250-mL glass jars having septum caps. The exact sample mass in each container was determined using a portable balance. The samples were screened as specified in the method. Because they were stored in a cooler during sample collection, the samples were allowed to sit at room temperature (20.0 °C) for approximately 30 minutes prior to being shaken. After shaking, the samples were allowed to sit for 10 minutes before screening. Due to recent rains in the area, the samples collected on the first day were extremely wet and did not break apart with shaking. Instead, the wet soil formed one or more round clumps in the containers, with free moisture condensed on the inner walls. The samples collected on the second day were not as wet and easily broke apart with shaking. An X-Wand heated diode sensor device was used to screen the samples in this study[6].

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[5] U.S. EPA, 2002, Method 5035A: Closed-System Purge-And-Trap and Extraction for Volatile Organics in Soil and Waste Samples. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol 1B, Draft Revision 1.

[6] X-Wand is a trademark of Western Research Institute, Laramie, WY. The X-Wand device is covered by a pending patent.

X1.1.6 The data generated in this study are specific to the experimental design of the study. The data give information on the performance of the heated diode screening method to estimate TCE concentrations in soil collected at a TCE-contaminated site and also on use of the method to determine the presence or absence of HVOC contamination at a site suspected of containing HVOC contamination. The data generated in this study are also specific to the soil used in the study, TCE concentrations, and the heated diode sensor device that was used to screen the soil samples. For other soil types, analyte concentrations, and heated diode sensor devices, these data may not apply.

X1.1.7 The TCE concentrations determined in the three side-by-side soil samples collected from the sample locations are listed in Table 1. The first column of data shows the estimated concentrations of TCE determined by screening the samples using the heated diode screening method. The second and third columns of data show the TCE concentrations determined in the samples by laboratory GC/MS analysis. The data reported by the laboratories are on an as received basis. There were no moisture content corrections performed on the data.

X1.1.7.1 The data given in Table 1 show that the method performs well for screening TCE-contaminated soil samples to give an estimated TCE concentration in the samples. The screening method gave no false negative or false positive results and provided estimated TCE concentrations that as screening data correlate well with the laboratory data, especially considering that the data for each of the sample locations were determined for three different

samples taken within close proximity to one another. In addition, as discussed in section X1.1.5, some of the samples were extremely wet. These are samples 1-11 listed in Table 1. There is no apparent difference in the data for samples 1-11, as compared with the data for samples 12-20, which were much drier samples. The results show that moisture does not interfere with the performance of the heated diode sensor for use in screening TCE-contaminated soil for TCE.

X1.1.8 Table 2 shows the percent of TCE determined in the TCE-contaminated soil samples using the screening method as compared with the average concentrations of TCE determined in the samples by the commercial and research laboratory GC/MS analysis. As shown in Table 2, these values range from 15 to 124%. For all but one of the samples, the values are less than 100% due to TCE remaining in the soil during screening.

X1.1.9 The heated diode sensor method can also be used to screen soil suspected of containing HVOC contamination for the presence or absence of contamination. The voltage reading from the device is used to indicate the presence or absence of HVOC contamination. The voltage reading of a heated diode sensor device is specific to the particular device, the particular sensor, and the amount of use the sensor has encountered. For this reason, the voltage reading can be used to indicate the presence or absence of HVOC contamination; however, to compare readings made during different screening episodes, the relative response values for the samples, R_s, (see section 13.4 of the method) should be calculated and compared.   The voltage readings and the relative response values for the samples that were screened from each of the sampling locations at the TCE-contaminated site are listed in Table 3.

X1.1.9.1 The data given in Table 3, show the usefulness of the screening method for indicating the presence or absence of HVOC contamination in soil. The soil samples containing TCE contamination gave sample voltage readings ranging from 0.237 to 7.818 volts, indicating the presence of HVOC contamination. One of the soil samples believed to be uncontaminated, sample 11, gave a voltage reading of 0.029, indicating an extremely low concentration of HVOC in the soil. The second soil sample believed to be uncontaminated, sample 20, gave a voltage reading of 0.000 volts, indicating no HVOC contamination in the soil.

Table 1. TCE Concentrations Determined in TCE-Contaminated Soil Samples, mg/Kg

Sample Location	Sample Screened using a Heated Diode Sensor Device[a] (Estimated Concentration)	Sample Analyzed by Commercial Laboratory GC/MS Analysis	Sample Analyzed by Research Laboratory GC/MS Analysis
1	0.091	0.32	0.27
2	0.23	1.0	2.0
3	0.13	0.47	0.55
4	0.71	1.6	1.8
5	1.4	2.0	2.0
6	0.12	0.66	0.67
7	0.30	1.3	1.2
8	0.14	0.58	0.63
9	0.091	0.10	0.11
10	0.13	0.40	0.41
11[b]	<0.005[c]	<0.05[d]	0.011
12	0.31	0.88	1.0
13	2.1	1.5	1.9
14	0.023	0.059	0.057
15	0.30	0.77	0.54
16	0.16	0.40	0.41
17	0.10	0.40	0.41
18	0.14	0.17	0.17
19	1.3	2.8	2.4
20[b]	<0.005[c]	<0.05[d]	<0.01[e]

[a] The X-Wand heated diode sensor device was used to screen samples in this study. X-Wand is a trademark of Western Research Institute, Laramie, WY. The X-Wand device is covered by a pending patent.

[b] Sample locations 11 and 20 were believed to be uncontaminated.

[c] Method quantitation limit is 0.005 mg/Kg.

[d] Commercial laboratory estimated quantitation limit is 0.05 mg/Kg.

[e] Research laboratory estimated quantitation limit is 0.01 mg/Kg.

Table 2. Percent TCE Determined in TCE-Contaminated Soil Samples Using the Heated Diode Sensor Device as Compared with GC/MS Data

Sample Location	Concentration Determined Using Heated Diode Sensor Device[a] (Estimated Concentration, mg/Kg)	Average Concentration Determined by Laboratory GC/MS Analysis, mg/Kg[b]	Percent TCE Determined by Heated Diode Sensor Device[c]
1	0.091	0.30	30%
2	0.23	1.5	15
3	0.13	0.51	25
4	0.71	1.7	42
5	1.4	2.0	70
6	0.12	0.67	18
7	0.30	1.3	23
8	0.14	0.61	23
9	0.091	0.11	83
10	0.13	0.41	32
11[d]	---	---	---
12	0.31	0.94	33
13	2.1	1.7	124
14	0.023	0.058	40
15	0.30	0.66	45
16	0.16	0.41	39
17	0.10	0.41	24
18	0.14	0.17	82
19	1.3	2.6	50
20[d]	---	---	---

[a] The X-Wand heated diode sensor device was used to screen samples in this study. X-Wand is a trademark of Western Research Institute, Laramie, WY. The X-Wand device is covered by a pending patent.

[b] Average concentration determined using commercial and research laboratory data

[c] Percent of average concentration determined using commercial and research laboratory data

[d] Sample locations 11 and 20 were believed to be uncontaminated.

Table 3. Sample Voltage Readings[a] and Sample Relative Response Values Determined using the Heated Diode Sensor Screening Method for Indicating the Presence or Absence of HVOC Contamination

Sample Location	Sample Voltage Reading, V_s	Sample Relative Response, R_s ($R_s = V_s/V_{HighTCEStd}$)
1	0.960	0.0971[b]
2	3.377	0.3416[b]
3	1.807	0.1828[b]
4	5.443	0.5126[c]
5	6.825	0.6428[c]
6	2.056	0.2079[b]
7	4.153	0.4200[b]
8	2.337	0.2364[b]
9	1.078	0.1015[c]
10	1.967	0.1853[c]
11[d]	0.029	0.0029[b]
12	4.252	0.4475[e]
13	7.818	0.8229[e]
14	0.237	0.0249[e]
15	3.718	0.3913[e]
16	2.248	0.2366[e]
17	0.996	0.1048[e]
18	2.121	0.2232[e]
19	7.200	0.7578[e]
20[d]	0.000	0.0000[e]

[a] The X-Wand heated diode sensor device was used to screen samples in this study. X-Wand is a trademark of Western Research Institute, Laramie, WY. The X-Wand device is covered by a pending patent.

[b] $V_{HighTCEStd} = 9.887$

[c] $V_{HighTCEStd} = 10.618$

[d] Sample locations 11 and 20 were believed to be uncontaminated.

[e] $V_{HighTCEStd} = 9.501$