VOLATILE ORGANIC COMPOUNDS IN SOILS AND OTHER SOLID MATRICES USING EQUILIBRIUM HEADSPACE ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 Method 5021 is a general purpose method for the preparation of volatile organic compounds (VOCs) in soils/sediments and solid wastes for determination by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS). The method is applicable to a wide range of organic compounds that have sufficiently high volatility to be effectively removed from soil samples using an equilibrium headspace procedure. The following compounds have been determined in soils using Method 5021.

Compound	CAS No.[a]
Benzene	71-43-2
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
Methylene chloride	75-09-2
Naphthalene	91-20-3
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,4-Trichlorobenzene	120-82-1

(continued)

Compound	CAS No.[a]
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
Vinyl chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3
Gasoline Range Organics	

[a] Chemical Abstract Service Registry Number

1.2 Method detection limits, using Method 8260, are compound-, matrix-, and instrument-dependent, and vary from approximately 0.1 to 3.4 µg/kg. The applicable concentration range of this method is approximately 10 µg/kg to 200 µg/kg. Analytes that are inefficiently extracted from the soil will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present in sufficient concentrations.

1.3 The following compounds may also be analyzed by this procedure or may be used as surrogates.

Compound	CAS No.[a]
Bromobenzene	108-86-1
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
cis-1,2-Dichloroethene	156-59-4
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
Isopropylbenzene	98-82-8
4-Isopropyltoluene	99-87-6
n-Propylbenzene	103-65-1
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8

[a] Chemical Abstract Service Registry Number

1.4 Alternatively, the method may be utilized as an automated sample introduction device as a means for screening samples for volatile organics. A suggested configuration is to employ it with Method 8021 but use very minimal calibration and quality control, i.e., a reagent blank and a single calibration standard, to obtain semiquantitative data.

1.5 Method 5021 may be applicable to other compounds that have sufficient volatility to be removed from the soil matrix using the conditions described in this method. It may also be applicable to both listed and non-listed target analytes in other matrices. For solid samples that contain more than 1% organic matter or for compounds with high octanol/water partitioning coefficients, the equilibrium headspace technique may yield slightly lower results then either dynamic purging or methanol extraction followed by dynamic purging.

1.6 This method is restricted to use by, or under the supervision of, analysts experienced in volatile organic analysis in general and specifically the use of equilibrium headspace devices interfaced to the determinative method selected by the analyst.

2.0 SUMMARY OF METHOD

2.1 At least 2 g of a soil sample are placed into a crimp-seal or screw-top glass headspace vial at time of sampling.

2.2 A matrix modifying solution is added to each soil sample to act as a chemical preservative. In addition, each sample is fortified with internal standards and surrogate compounds. These additions may be done either in the field or in the laboratory upon receipt of samples.

2.3 Additional sample volume is collected in a VOA vial for dry weight determination and for high concentration determination if the sample concentration requires it.

2.4 In the laboratory, the vials are rotated to allow for diffusion of the internal standards and surrogates throughout the matrix. The vials are placed in the autosampler carousel of the headspace analyzer and maintained at room temperature. Approximately 1 hour prior to analysis, the individual vials are moved to a heated zone and allowed to equilibrate. The sample is then mixed by mechanical vibration while the elevated temperature is maintained.

2.5 The autosampler then pressurizes the vial with helium which forces a portion of the headspace gas mixture through a heated transfer line onto the GC column.

2.6 Determinative analysis is performed using the appropriate GC or GC/MS method.

3.0 INTERFERENCES

3.1 Volatile organic analyses are subject to major interference problems because of the prevalence of volatile organics in a laboratory. See Method 5000, Sec. 3.0, for common problems and precautions to be followed.

3.2 The sample matrix itself can cause severe interferences by one of several processes or a combination of these processes. These include, but are not necessarily limited to, the absorption potential of the soil, the biological activity of the soil, and the actual composition of the soil. Soils high in oily material and organic sludge wastes inhibit the partitioning of the volatile target analytes

into the headspace, therefore, recoveries will be low. This so- called "matrix effect" can be difficult, if not impossible, to overcome. It is recommended that surrogates or additional deuterated compounds (for GC/MS methods) be added to a matrix and analyzed to determine the percent recovery of these compounds. The calculated percent recovery can give some indication of the degree of the matrix effect, but not necessarily correct for it. Alternatively, the use of the high concentration procedure in this method should minimize the problem with oily waste and other organic sludge wastes.

4.0 EQUIPMENT AND SUPPLIES

4.1 Sample Containers - Clear glass, 22-mL soil vials, compatible with the analytical system. The vial must be capable of being hermetically sealed in the field (either crimp-cap or screw-cap) and be equipped with a polytetrafluoroethylene (PTFE)-lined septum which demonstrates minimum bleed at elevated temperatures while maintaining the seal. Ideally, the vials and septa should have a uniform tare weight. Prior to use, wash the vials and septa with detergent solution, then rinse with tap water followed by distilled water. Dry the vials and septa in an oven at 105°C for 1 hour, then remove and allow to cool. Store in an area free of organic solvents. Vials of other sizes may be employed, provided that they can be hermetically sealed and equipped with a suitable septum.

4.2 Headspace System - This method was developed using a totally automated equilibrium headspace analyzer, the Tekmar Model 7000 Equilibrium Headspace Autosampler and a Tekmar 7050 Carousel (Tekmar Co., 7143 East Kemper Road, Cincinnati, OH 45249). Similar systems are available from several commercial sources. The system used must meet the following specifications.

4.2.1 The system must be capable of holding samples at elevated temperatures and establishing a reproducible equilibrium between a wide variety of sample types and the headspace.

4.2.2 The system must be capable of accurately transferring a representative portion of the headspace into a gas chromatograph fitted with a capillary column. This must be accomplished without adversely affecting the chromatography or the detector.

4.2.3 The operating conditions listed in Sec. 7.0 are those selected for the equipment used in developing this method. Other equipment and conditions may be employed, provided that the laboratory demonstrates performance for the analytes of interest using the determinative method appropriate for the intended application.

4.3 Field Sampling Equipment

4.3.1 A soil sampler which delivers at least 2 g of soil is necessary, e.g., Purge-and-Trap Soil Sampler Model 3780SPT (Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314), or equivalent.

4.3.2 An automatic syringe or bottle-top dispenser calibrated to deliver 10.0 mL of matrix modifier solution, e.g., Automatic Vaccinator Model C1377SN (NASCO, 901 Jamesville Ave., P.O. Box 901, Fort Atkinson, WI 53538), or equivalent.

4.3.3 An automatic syringe calibrated to deliver internal standards and surrogate analytes.

4.3.4 Crimping tool for sample vials. If using screw-top vials, this is not needed.

4.4 Miscellaneous Equipment

4.4.1 VOA vials - 40 or 60 mL VOA vials with PTFE-faced septa and crimp-seal caps or screw-top caps. These vials will be used for sample screening, high concentration analysis (if needed) and dry weight determination.

5.0 REAGENTS

5.1 Organic-Free Reagent Water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.2 Methanol, CH_3OH - Pesticide quality or equivalent. Store away from other solvents. Purchase in small quantities (½ Liter or 1 Liter size) to minimize contamination.

5.3 See the determinative method and Method 5000 for guidance on the preparation of stock standards and a secondary standard for internal standards, calibration standards, and surrogates.

5.3.1 Calibration spiking solutions - Prepare five spiking solutions in methanol that contain all the target analytes and the surrogate standards. The concentrations of the calibration solutions should be such that the addition of 1.0 µL of each to the 22 mL vials will bracket the analytical range of the detector, e.g., for Method 8260 the suggested concentration range for target analytes and surrogates is 5, 10, 20, 40 and 50 mg/L. The suggested concentration of internal standards is 20 mg/L (internal standards may be omitted for the GC methods if desired). The internal standard may be added separately using 1.0 µL or premixed with the calibration standards maintaining a 20 mg/L concentration in each calibration standard. These concentrations may vary depending on the relative sensitivity of the GC/MS system or any other determinative method that is utilized.

5.3.2 Internal and surrogate standards - Follow the recommendations of the determinative methods for the selection of internal and surrogate standards. A concentration of 20 mg/L in methanol for both internal and surrogate standards will be needed for spiking each sample. If determination is by GC, external standard calibration may be preferred and the internal standard is omitted. The concentration may vary depending on the relative sensitivity of the GC/MS system or any other determinative method that is utilized.

5.4 Blank Preparation - Transfer 10.0 mL (Sec. 5.6) of matrix modifying solution to a sample vial. Add the prescribed amounts of the internal standards and surrogate compounds, and seal the vial. Place it in the autosampler and analyze in the same manner as an unknown sample. Analyzing the blank in this way will indicate possible problems with the autosampler as well as the headspace device.

5.5 Preparation of Calibration Standards - Prepare calibration standards in the same manner as the blanks (Sec. 5.4) using the standards prepared in Sec. 5.3.1.

5.6 Matrix Modifying Solution - Using a pH meter, add concentrated phosphoric acid (H_3PO_4) dropwise to 500 mL of organic-free reagent water until the pH is 2. Add 180 g of NaCl. Mix well until all components are dissolved. Analyze a 10.0 mL portion from each batch per Sec. 5.4 to verify that the solution is free of contaminants. Store in a sealed bottle in an area free of organic chemicals at 4°C.

WARNING: The matrix modifying solution may not be appropriate for soil samples having organic carbon content. See Sec. 6.2.4.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Three alternative procedures are presented for the collection of low concentration samples in special headspace sample vials. One procedure includes the addition of a matrix modifying solution and standards in the field while the other two procedures do not. The choice between these alternatives should be based on knowledge of the field conditions, the organic carbon content of the soil, the specific volatile analytes of interest, and the intended use of the analytical results. Whichever alternative is used, collect 3 or 4 vials of sample from each sampling point to allow for sample reanalyses if necessary. In addition, separate portions of sample are taken for dry weight determination and for high concentration analysis (if necessary).

The addition of the matrix modifying solution and the internal and surrogate standards at the time of sampling (Sec. 6.2) is the preferred option unless high concentrations of volatile organics are expected. The matrix modifying solution eliminates biodegradation of the analytes, minimizes losses of analytes by volatility since the vial is not opened in the laboratory, and minimizes dehydrohalogenation reactions through pH adjustment.

The downside is increased opportunity for contamination of the matrix modifier and standards in a field sampling situation. Also, skilled personnel are required to precisely and accurately add the matrix modifying solution, and especially the internal and surrogate standards.

These problems are minimized when these solutions are not added in the field (Sec. 6.1), however, there is the likelihood of significant losses of volatile analytes when the vial is reopened in the laboratory.

If high concentrations of volatile organics are expected (greater than 200 µg/kg), collection of the sample in the 22-mL vial without the addition of the matrix modifying solution allows direct addition of methanol to the vial, as described in the high concentration method in Sec. 7.5.

6.1 Sample collection without the addition of matrix modifying solution and standards

6.1.1 Use standard 22-mL crimp-cap or screw-top glass headspace vials with PTFE-faced septa (other vials may be used, as described in Sec. 4.1).

6.1.2 Using the purge-and-trap soil sampler (Sec. 4.3.1), add 2-3 cm (approximately 2 g) of the soil sample to a tared 22-mL headspace vial and seal immediately with the PTFE side of the septum facing toward the sample. The samples should be introduced into the vials gently to reduce agitation which might drive off volatile compounds.

6.2 Sample collection with the addition of matrix modifying solution and standards

6.2.1 Use standard 22-mL crimp-cap or screw-top glass headspace vials with PTFE-faced septa (other vials may be used, as described in Sec. 4.1).

6.2.2 Add 10.0 mL of matrix modifying solution to the vial prior to adding the sample.

6.2.3 Using the purge-and-trap soil sampler (Sec. 4.3.1), add 2-3 cm (approximately 2 g) of the soil sample to a tared 22-mL headspace vial. The samples should be introduced

into the vials gently to reduce agitation which might drive off volatile compounds. Seal the vial immediately with the PTFE side of the septum facing toward the sample.

6.2.4 Using an appropriate size syringe (e.g., 10 µL), carefully puncture the septum and add the amount of internal and surrogate standards called for in the determinative method.

WARNING: Preliminary indications are that soil samples containing over 1% organic carbon may yield low recoveries when the matrix modifying solution is used. The matrix modifying solution may not be appropriate for these samples.

6.3 The third alternative is to add the soil sample to a vial containing 10.0 mL of organic-free reagent water. This organic-free reagent water may be added to the vial either in the field or in the laboratory prior to shipping the vials to the field.

6.3.1 Use standard 22-mL crimp-cap or screw-top glass headspace vials with PTFE-faced septa (other vials may be used, as described in Sec. 4.1).

6.3.2 Using the purge-and-trap soil sampler (Sec. 4.3.1), add 2-3 cm (approximately 2 g) of the soil sample to a tared 22-mL headspace vial containing 10.0 mL of reagent water. The samples should be introduced into the vials gently to reduce agitation which might drive off volatile compounds. Seal immediately with the PTFE side of the septum facing toward the sample.

6.4 Field blanks should be prepared, regardless of which alternative is employed for the soil samples. If the matrix modifying solution is not added in the field, then the field blank should be prepared by adding 10.0 mL of organic-free reagent water to a clean vial and immediately sealing the vial. If the matrix modifying solution and standards are added in the field, then prepare a field blank by adding 10.0 mL of matrix modifying solution plus internal and surrogate standards to a clean vial.

6.5 Fill a 40- or 60-mL VOA vial with soil from each sampling point to use for dry weight determination, sample screening, and for high concentration analysis (if necessary). Sample screening is optional since there is no danger of contaminating the headspace device because of carryover from a high concentration sample.

6.6 Sample Storage

6.6.1 Store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.6.2 All samples should be analyzed within 14 days of collection. Samples not analyzed within this period must be identified to the data user and the results are considered minimum values.

7.0 PROCEDURE

7.1 Sample screening

This method (using the low concentration approach), used in conjunction with either Methods 8015 (GC/FID) or 8021 (GC/PID/ELCD), may be used as a sample screening method prior to any

of the sample introduction - GC/MS configurations to assist the analyst in determining the approximate concentration of volatile organics present in a sample. This is especially critical prior to the use of volatile organic analysis by purge-and-trap to prevent the contamination of the system by high concentration samples. It can also be helpful prior to the use of this headspace method, to determine whether to proceed with the low concentration method or the high concentration method. High concentrations of volatiles will not contaminate the headspace device. However, it may create contamination problems in the GC or GC/MS system. Whenever this method is utilized for sample screening, very minimal calibration and QC are suggested. In most cases, a reagent blank and a single point calibration are sufficient.

7.2 Determination of sample % dry weight

In certain cases, sample results are desired based on dry-weight basis. When such data are desired, a portion of sample for this determination should be weighed out from the 40 or 60 mL VOA vial (Sec. 6.5.3).

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the % dry weight of the sample by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

7.3 The Low Concentration Method utilizing an equilibrium headspace technique is found in Sec. 7.4 and sample preparation for the High Concentration Method is found in Sec. 7.5. The high concentration method is recommended for samples that obviously contain oily material or organic sludge waste (see Sec. 3.3). See Method 5000, Sec. 7.0, for guidance on the selection of a GC or GC/MS determinative method. For the analysis of gasoline, use Method 8021 with GC/PID for BTEX in series with Method 8015 with the GC/FID detector for hydrocarbons. If GC/MS analysis is preferred for BTEX in gasoline, follow Method 8260.

7.4 Low concentration method for soil/sediment and solid waste amenable to the equilibrium headspace method. (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.4.1 Calibration

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods because of possible interference problems with internal standards. If interferences are not a problem, based on historical data, internal standard calibration is acceptable. The GC/MS methods normally utilize internal standard calibration. The GC/MS methods require instrument tuning prior to proceeding with calibration.

7.4.1.1 GC/MS tuning

If a GC/MS determinative method is employed, prepare a 22-mL vial containing reagent water and the amount of BFB specified in the determinative method.

7.4.1.2 Initial calibration

Prepare five 22-mL vials, as described in Sec. 5.5, and a reagent blank (Sec. 5.4), and proceed according to Sec. 7.4.2 and the determinative method selected. The mixing step is eliminated since no soil is present in the vial.

7.4.1.3 Calibration verification

Prepare a 22-mL vial, as described in Sec. 5.5, by spiking with the mid-concentration calibration standard. Proceed according to Sec. 7.4.2.4 (beginning by placing the vial into the autosampler) and the determinative method. If a GC/MS determinative method is employed, prepare a second 22-mL vial containing reagent water and the amount of BFB specified in the determinative method.

7.4.2 Headspace operating conditions

The conditions described throughout Sec. 7.4 were experimentally optimized using the equipment described in Sec. 4.2.1 and employing Method 8260 as the determinative method. If other headspace systems and determinative methods are utilized, it is recommended that the manufacturer's headspace operating conditions be followed, provided that they are appropriate for the determinative method to be employed.

7.4.2.1 This method is designed for a 2-g sample size. The sample is prepared in the field by adding 2 g of the soil sample to the 22 mL crimp-seal or screw-top glass headspace vial as described in Sec. 6.0.

7.4.2.2 Prior to analysis, weigh the sealed vial and its contents to 0.01 g. If the matrix modifying solution was added at the time of sampling (Sec. 6.2), the tare weight does not include the 10 mL of matrix modifying solution. Therefore, weigh the field blank and determine the weight of the matrix modifying solution in the field blank and use that weight as the weight of the matrix modifying solution in the samples. (Although this approach may introduce some error into the sample results, that error should be much less than the changes that will occur in an unpreserved sample shipped to the laboratory without the modifier).

7.4.2.3 If the matrix modifying solution was not added at the time of sampling (Sec. 6.1), unseal the vial, rapidly add 10.0 mL of matrix modifying solution and the amount of internal standard and surrogate standards called for in the determinative method, and immediately reseal the vial. As noted in the introductory text in Sec. 6.0, volatilization losses will occur as a result of opening the vial and displacing 10 mL of air.

NOTE: Only open and prepare one vial at a time to minimize loss of volatile organics.

7.4.2.4 Mix the samples (on a rotator or shaker) for at least 2 min. Place the vials in the autosampler carrousel at room temperature. The individual vials are heated to 85°C and allowed to equilibrate for 50 min. Each sample is mixed by mechanical

vibration for at least 10 min during this equilibrium period. Each vial is pressurized with helium carrier gas to a minimum pressure of 10 psi.

7.4.2.5 A representative and reproducible sample of the pressurized headspace is transferred to the GC column through a heated transfer line according to the manufacturer's instructions.

7.4.2.6 Proceed with the analysis as per the determinative method of choice.

7.5 High concentration method

7.5.1 If the sample was collected as described in Sec. 6.1 without the addition of matrix modifying solution or organic-free reagent water to the vial, then weigh the sample to the nearest 0.01 g, add 10.0 mL of methanol to the sample in the tared 22-mL vial, and immediately reseal the vial. Open only one vial at a time to minimize the loss of volatile organics.

7.5.2 If the procedures in either Sec. 6.2 or 6.3 were employed for sample collection and either the matrix modifying solution or organic-free reagent water were added to the vial, then the sample for high concentration analysis should be taken from the separate 40- or 60-mL VOA vial filled in the field. Transfer approximately 2 g of sample from the 40- or 60-mL VOA vial into a tared 22-mL sample vial. Immediately add 10.0 mL of methanol to the 22-mL vial and seal both the 22-mL and the VOA vials. Open only one vial at a time to minimize the loss of volatile organics.

7.5.3 Mix by shaking for 10 min at room temperature. Decant 2 mL of the methanol to a screw-top vial with PTFE-faced septa and seal. Withdraw 10 µL, or appropriate volume of extract from Table 2, and inject into a 22 mL vial containing 10.0 mL of matrix modifying solution and internal standards (if required) and surrogates. Analyze by the headspace procedure by placing the vial into the autosampler and proceeding with Sec. 7.4.2.4.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 5000 for sample preparation QC procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Sec. 8.0 of Methods 5000 and 8000 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - See Sec. 8.0 in Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each

set of samples to be analyzed. This includes the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS) and the addition of surrogates to each sample and QC sample.

8.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

Single laboratory accuracy and precision data were obtained for the method analytes in two soil matrices: sand and a surface garden soil. These data are found in tables in Method 8260.

10.0 REFERENCES

1. Flores, P., Bellar, T., "Determination of Volatile Organic Compounds in Soils using Equilibrium Headspace Analysis and Capillary Column Gas Chromatography/Mass Spectrometry," U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH, December, 1992.

2. Ioffe, B.V., Vitenberg, A.G., "Headspace Analysis and Related Methods in Gas Chromatography," John Wiley and Sons, 1984.

TABLE 1

DETERMINATIVE METHODS COMPATIBLE WITH METHOD 5021

Method Number	Method Title
8015	Nonhalogenated Volatile Organics Using GC/FID
8021	Aromatic and Halogenated Volatiles by GC with Detectors in Series
8260	Volatile Organics by GC/MS

TABLE 2

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000 µg/kg	100 µL
1,000-20,000 µg/kg	50 µL
5,000-100,000 µg/kg	10 µL
25,000-500,000 µg/kg	100 µL of 1/50 dilution [b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 µL added to the syringe.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

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