

METHOD 5000

SAMPLE PREPARATION FOR VOLATILE ORGANIC COMPOUNDS

1.0 SCOPE AND APPLICATION

1.1 Method 5000 provides general guidance on the selection of sample preparation methods (purge-and-trap, extraction, azeotropic distillation, vacuum distillation, dilution, headspace, etc.) for introducing volatile organic compounds into a detection device (outlined in the determinative methods). The matrices include aqueous, soil/sediment, solid waste, organic solvents, air, and oily waste. Other waste matrices may be adaptable to one or more of the listed preparation methods.

1.2 Method 5000 also provides specific information pertaining to analyte interferences, preparation of calibration and spiking standards, and specific quality control that should be applied to each preparative method.

1.3 The following table is presented as a guide to the sample preparation techniques for volatile organic compounds:

SAMPLE PREPARATION METHODS FOR VOLATILE ORGANICS

| Method No. | Matrix | Sample Preparation | Analytes |
|------------|--------------------------------------|------------------------------|---------------------------------------|
| 3585 | Oily waste | Solvent dilution | Volatile organics |
| 5021 | Solids | Automated headspace | Volatile organics |
| 5030 | Aqueous | Purge-and-trap | Volatile organics |
| 5031 | Aqueous | Azeotropic distillation | Polar volatile organics |
| 5032 | Aqueous & solids | Vacuum distillation | Non polar and polar volatile organics |
| 5035 | Solids, organic solvents, oily waste | Closed system purge-and-trap | Volatile organics |
| 5041 | Air sampled by VOST | Purge-and-trap from VOST | Volatile POHCs |

VOST = Volatile Organic Sampling Train
POHCs = Principal Organic Hazardous Constituents

1.4 Method 3585 provides guidance for dilution and direct injection of oily waste samples (e.g., waste oil or oily waste that passes through the filter during TCLP sample preparation) for volatile organic analysis.

1.5 The following table is presented as a guide to the air sampling methods found in Chapter Ten that can be used in conjunction with the volatile organic determinative methods:

AIR SAMPLING METHODS FOR VOLATILE ORGANIC COMPOUNDS
FROM CHAPTER TEN OF SW-846

| Method No. | Air Sampling Method | Sample Preparation | Analytes |
|------------|--------------------------|----------------------------------|---------------------------------------|
| 0011 | Aqueous solution of DNPH | Solvent extraction | Formaldehyde plus aldehydes & ketones |
| 0030 | Resin/charcoal | Purge-and-trap by 5041 | Volatile organics |
| 0031 | Resin/Anasorb 747 | Purge-and-trap by 5041 | Volatile organics |
| 0040 | Tedlar® bag | Direct analysis with sample loop | Volatile organics |
| 0100 | DNPH-coated silica gel | Solvent extraction | Formaldehyde plus aldehydes & ketones |

DNPH = Dinitrophenylhydrazine

1.6 Prior to employing this method, analysts are advised to consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the allowed flexibility in the choice of apparatus, reagents, and supplies. In addition, unless specified in a regulation, the use of SW-846 methods is not mandatory in response to Federal testing requirements. The information contained in this procedure is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to meet the data quality objectives or needs for the intended use of the data.

2.0 SUMMARY OF METHOD

2.1 Method 5000 provides general information that is common to each of the methods listed in Sec. 1.0. Specifically, this includes: interference problems that are common to any volatile organic sample preparation method; preparation of calibration standards, internal standards, surrogate spikes, laboratory control samples (LCSs), and matrix spikes; a brief summary of each of the methods; and the specific quality control procedures that should be applied to each of the preparative methods.

2.2 Table 1 provides guidance on which sample preparation methods can be employed in conjunction with each volatile organic determinative method.

3.0 INTERFERENCES

3.1 Samples requiring analysis for volatile organic compounds can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method for specific guidance on quality control procedures and to Chapter Four for guidance on the cleaning of glassware.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample purging device must be thoroughly rinsed between samples with an appropriate solvent. Purge and trap devices or headspace devices should be thoroughly baked out between samples. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents.

3.4.1 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result.

3.4.2 Since methylene chloride will permeate through polytetrafluoroethylene (PTFE) tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing.

3.4.3 Laboratory workers' clothing that has been previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

3.4.4 The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and similar precautions must be taken to minimize this problem..

3.5 Interference problems specific to the sample preparation methods are discussed in the individual methods.

4.0 APPARATUS AND MATERIALS

Refer to the specific method of interest for a description of the apparatus and materials needed.

5.0 REAGENTS

5.1 Refer to the specific method of interest for a description of the solvents and other reagents needed.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water as defined in Chapter One.

5.3 Stock standards for spiking solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. The stock solutions may be used as calibration standards if dilutions are made in a water-miscible solvent. However, the quality control check sample stock concentrate must be prepared independently from the calibration because it serves as a check on the accuracy of the calibration solution.

5.3.1 Purgeable stock standards - Prepare stock standards in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. The following sections outline one approach to preparing stock standards.

5.3.1.1 Place about 9.8 mL of methanol in a 10-mL, tared, ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.3.1.2 Using a 100- μ L syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.3.1.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.1.4 Transfer the stock standard solution into a PTFE-sealed screw-cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light, or as recommended by the supplier of the standard.

5.3.1.5 Refer to the determinative method for holding times of the stock solutions.

5.3.2 Non-purgeable stock standards - Non-purgeable stock solutions may be prepared from pure standard materials or purchased as certified solutions. Refer to the individual determinative method for additional guidance.

5.4 Surrogate standards - A surrogate standard (i.e., a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample) should be added to each sample, blank, laboratory control sample and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated by comparing the measured concentration with the amount added to the sample.

5.4.1 Recommended surrogates for certain analyte groups may be listed in the determinative methods. For methods where no recommended surrogates are listed, the laboratory is free to select compounds that fall within the definition provided above. Even compounds that are on the method analyte list may be used as surrogates as long as historical

data are available to ensure their absence at a given site. Normally, one or more surrogates are added for each analyte group.

5.4.2 Prepare a surrogate spiking concentrate by mixing stock standards prepared above and diluting with a water-miscible solvent. Commercially-prepared spiking solutions are acceptable. The concentration for volatile organic analysis by purge-and-trap should be such that a 10 µL aliquot when added directly to 5 mL of sample provides the concentrations listed in the determinative method. The spiking volumes are normally listed in each preparation method. Where concentrations are not specified, a concentration in the sample of 10 times the estimated quantitation limit is recommended. If the surrogate quantitation limit is unknown, the average estimated quantitation limit of method target analytes may be utilized to estimate a surrogate quantitation limit.

5.5 Matrix spike standards - Prepare a matrix spike concentrate by mixing stock standards as prepared above and diluting with a water-miscible solvent. Commercially-prepared spiking solutions are acceptable. The stock standards are to be independent of the calibration standard.

5.5.1 A few methods provide guidance on concentrations and the selection of compounds for matrix spikes. For example, the recommended purgeable matrix spiking solution for Methods 8021 and 8260 is as follows: Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25 mg/L.

Purgeable organics

1,1-Dichloroethene
Trichloroethene
Chlorobenzene
Toluene
Benzene

The suggested matrix spiking solution in any method may be replaced with a project-specific list of analytes of concern. The spiking concentration employed should correspond to either the applicable regulatory limit or action level for the compound or a concentration in the middle of the calibration range, unless the regulatory limit or action level is lower.

5.5.2 For methods with no guidance, select five or more analytes (select all analytes for methods with five or less) from each analyte group for use in a spiking solution. Where matrix spike concentrations in the sample are not listed it should be at or below the regulatory limit or action level concentration or 1 to 5 times higher than the background concentration, whichever concentration would be larger.

5.6 Laboratory control spike standard - Use the matrix spike standard prepared in Sec. 5.5 as the spike standard for the laboratory control sample (LCS). The LCS should be spiked at the same concentration as the matrix spike.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See Chapters Two and Four for guidance on sample collection, preservation, and handling.

7.0 PROCEDURE

Water, soil/sediment, sludge, and waste samples requiring analysis for volatile organics are extracted and/or introduced into the GC and/or GC/MS system by various methods (see Table 1). This manual contains method choices that are dependent on the matrix, the physical properties of the analytes, the sophistication and cost of equipment available to a given laboratory, and the turn-around time required for sample preparation. The following is a brief summary of each of the sample preparation/introduction techniques:

7.1 Method 3585: This method describes a solvent dilution (hexadecane) technique followed by direct injection into a sensitive GC/MS system for the analysis of volatiles in waste oils. Method 3585 has adequate sensitivity to determine the regulatory concentrations for TCLP oily wastes that pass through the filter. Direct injection is very simple, provides quick turnaround, and requires no special hardware. However, the GC/MS system must be quite sensitive, and direct injection has the potential for instrument contamination and is more subject to matrix difficulties. Method 3585 is best when performing analyses for small groups of samples.

7.2 Method 5021: This method describes an automated headspace analysis for soils and other solid matrices. The solid sample is placed in a tared septum-sealed vial at time of sampling. A matrix modifier is added containing internal and/or surrogate standards. The sample vial is placed into an automated equilibrium headspace sampler which heats the entire sample to 85°C and mixes it by mechanical vibration. A measured volume of headspace is automatically introduced into a GC or GC/MS system for volatile organic analysis. The method is automated and causes no equipment contamination, however, it does require an automated headspace device.

7.3 Method 5030: This method describes the technique of purge-and-trap for the introduction of purgeable organics into a gas chromatograph. This procedure is applicable to aqueous samples and water-miscible extracts prepared by Method 5035. An inert gas is bubbled through the sample, which will efficiently transfer the purgeable organics from the aqueous phase to the vapor phase. The vapor phase is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. Purge-and-trap is easily automated, provides good precision and accuracy, but, is limited to analytes that purge efficiently from water and requires a purge-and-trap device. The system is easily contaminated by samples containing compounds at mg/L concentrations. This procedure may be used for the analysis of gasoline in various aqueous matrices.

7.4 Method 5031: This method describes an azeotropic distillation technique for the analysis of nonpurgeable, water-soluble, volatile organics in aqueous samples. The sample is distilled in an azeotropic distillation apparatus (guidance for an optional micro-distillation apparatus is also included) followed by direct aqueous injection of the distillate into a GC or GC/MS system. The method is not readily automated except for the GC/MS analysis, requires a 1-hour distillation, and is applicable to a limited group of analytes.

7.5 Method 5032: This method describes a closed-system vacuum distillation technique for the analysis of volatile organics including nonpurgeable, water-soluble, volatile organics in aqueous samples, solids and oily waste. The sample is introduced into a sample flask which is then attached to the vacuum distillation apparatus. The sample chamber pressure is reduced and remains at approximately 10 torr (vapor pressure of water) as water is removed from the sample. The vapor is passed over a condenser coil chilled to a temperature of -10°C or less, which results in the condensation of water vapor. The uncondensed distillate is cryogenically trapped on a section of 1/8 inch stainless steel tubing chilled to the temperature of liquid nitrogen (-196°C). After an

appropriate distillation period, which may vary due to matrix or analyte group, the condensate contained in the cryogenic trap is thermally desorbed and transferred to the gas chromatograph using helium carrier gas. This method very efficiently extracts organics from a variety of matrices. The method requires a vacuum system, cryogenic cooling, and is not readily automated, except for the GC/MS analysis.

7.6 Method 5035: This method describes a closed-system purge-and-trap for the analysis of volatile organics that are purgeable from a solid matrix at 40°C. It is amenable to soil/sediment and any solid waste sample of a consistency similar to soil. It differs from the original soil method in Method 5030 in that a sample (normally 5 g) is placed into the sample vial at time of sampling along with a matrix-modifying solution. The sample remains hermetically sealed from sampling through analysis as the closed-system purge-and-trap device automatically adds standards and then performs the purge-and-trap process. The method provides more accurate data than the original method because the sample container is never opened, minimizing the loss of volatiles through sampling handling. However, it does require a special purge-and-trap device. It also includes a technique for the extraction of oily waste using methanol. This procedure may be used for the analysis of gasoline in various solid matrices.

7.7 Method 5041: This method is applicable to the analysis of sorbent cartridges from a volatile organic sampling train (VOST). The sorbent cartridges are placed in a thermal desorber which, in turn, is attached to a standard purge-and-trap device. Analysis may be by GC or GC/MS.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control procedures. Each laboratory using SW-846 methods should maintain a formal quality assurance program. Each sample preparation batch of 20 or less samples should contain: a method blank; either a matrix spike/matrix spike duplicate or a matrix spike and duplicate samples; and a laboratory control sample, unless the determinative method provides other guidance.

8.2 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 reference matrix. This will include a combination of the sample preparation method (usually a 5000 series method for volatile organics) and the determinative method (an 8000 series method). The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

8.2.1 The reference samples are prepared from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate must be made using stock standards prepared independently from those used for calibration.

8.2.2 The procedure for preparation of the reference sample concentrate is dependent upon the method being evaluated. Guidance for reference sample concentrations for certain methods are listed below. In other cases, the determinative methods contain guidance on preparing the reference sample concentrate and the reference sample. If no guidance is provided, prepare a reference sample concentrate in methanol. Spike the reference sample at the concentration on which the method performance data are based. The spiking volume added to water should not exceed 1 mL/L so that the spiking solvent will not decrease purging

efficiency. If the method lacks performance data, prepare a reference standard concentrate at such a concentration that the spike will provide a concentration in the clean matrix that is 10 - 50 times the MDL for each analyte in that matrix.

The concentration of the target analytes in the reference sample may need to be adjusted to more accurately reflect the concentrations that will be analyzed in the laboratory. If the concentration of an analyte is being evaluated relative to a regulatory limit, see Sec. 8.3.3 for information on selecting an appropriate spiking level.

8.2.3 To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. Use a clean matrix for spiking purposes (one that does not have any target or interference compounds) e.g., organic-free reagent water for the water matrix or sand or soil (free of organic interferences) for the solid matrix. Because of the volatility of these compounds, the spike must be introduced directly into the matrix while the matrix is in a sealed container (e.g., a gas-tight syringe or purge device).

8.2.4 Preparation of reference samples

The following sections provide guidance on the QC reference sample concentrates for many SW-846 determinative methods. The concentration of the target analytes in the QC reference sample for the methods listed below may need to be adjusted to more accurately reflect the concentrations of interest in different samples or projects. If the concentration of an analyte is being evaluated relative to a regulatory limit or action level, see Sec. 8.3.3 for information on selecting an appropriate spiking level. In addition, the analyst may vary the concentration of the spiking solution and the volume of solution spiked into the sample. However, because of concerns about the effects of the spiking solution solvent on the sample, the total volume spiked into a sample should generally be held to no more than 200 μL .

8.2.4.1 When analyzing aqueous samples by purge-and-trap Method 5030, prepare reference sample concentrates containing each target analyte at a concentration of 10 mg/L in methanol. For water samples, spike 100 mL of organic-free reagent water with 200 μL which provides a 20 $\mu\text{g/L}$ concentration in the reference sample. Quickly transfer the spiked water to four, 5-mL gas-tight syringes. The samples are ready for analysis using Method 5030 and the appropriate determinative method.

8.2.4.2 When analyzing soil or other solid samples by purge-and-trap by Method 5035, add 10 μL of reference sample concentrate directly to the purge device as specified in Sec. 7.0. For oily waste analysis by Method 3585 or the high concentration technique in Method 5035, add 10 μL of reference sample concentrate (dissolved in methanol) directly to the oily waste just prior to the addition of the extraction solvent. The concentration in the oily waste should be 10 - 50 times greater than the determinative method MDL for each analyte. Prepare four replicates.

8.2.4.3 When analyzing matrices using equilibrium headspace by Method 5021 or vacuum distillation by Method 5032, prepare the reference sample concentrate as per Sec. 8.2.4.1. Reference sample concentrates and spiking solutions used in azeotropic distillation Method 5031 should be prepared in water, not alcohol or acetone. Add sufficient reference sample concentrate to the volume of organic-free reagent water specified in these methods to provide a concentration in the water that is 10 - 50 times greater than the determinative method MDL for each analyte. Prepare four replicates.

8.2.4.4 For Methods 8031, 8032, 8315 and 8316, analyze four portions of the water sample volume specified in each method, spiked at a concentration that is 10 - 50 times greater than the determinative method MDL for each analyte.

8.2.5 Analyze replicate aliquots (at least four) of the well-mixed reference samples by the same procedures used to analyze actual samples (Sec. 7.0 of each of the methods). This will include a combination of the sample preparation method (usually a 5000 series method for volatile organics) and the determinative method (an 8000 series method). Follow the guidance on data calculation and interpretation presented in Method 8000, Sec. 8.0.

8.3 Sample Quality Control for Preparation and Analysis

8.3.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair per analytical batch. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. See Sec. 5.5 for additional guidance on matrix spike preparation. Sec. 8.3.3 provides guidance on establishing the concentration of the matrix spike compounds in the sample chosen for spiking. The choice of analytes to be spiked should reflect the analytes of interest for the specific project. Thus, if only a subset of the list of target analytes provided in a determinative method are of interest (e.g., Method 8260 is used for the analysis of only aromatics), then these would be the analytes of interest for the project. In the absence of project-specific analytes of interest, it is suggested that the laboratory periodically change the analytes that are spiked with the goal of obtaining matrix spike data for most, if not all, of the analytes in a given determinative method.

8.3.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

For the laboratory control sample, use a clean matrix for spiking purposes (one that does not have any target or interference compounds) e.g., organic-free reagent water for the water matrix or sand or soil (free of organic interferences) for the solid matrix. Because of the volatility of these compounds, the spike must be introduced directly into the matrix while the matrix is in a sealed container (e.g., a gas-tight syringe or purge device).

8.3.3 The concentration of the matrix spike sample and/or the LCS should be determined as described in the following sections.

8.3.3.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit or action level, the spike should be at or below the regulatory limit or action level or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher.

8.3.3.2 If historical data are not available, it is suggested that an uncontaminated sample of the same matrix from the site be submitted for matrix spiking

purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries.

8.3.3.3 If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the spike should be at the same concentration as the reference sample (Sec. 8.2.4) or 20 times the estimated quantitation limit (EQL) in the matrix of interest. It is again suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.

8.3.4 Analyze these QC samples (the LCS and the matrix spikes or the optional matrix duplicates) following the procedure (Sec. 7.0) of the selected determinative method. Calculate and evaluate the QC data as outlined in Sec. 8.0 of Method 8000.

8.3.5 Blanks - Use of method blanks and other blanks are necessary to track contamination of samples during the sampling and analysis processes. Refer to Chapter One for specific quality control procedures.

8.3.6 Surrogates - A surrogate standard is a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample. Surrogate standards should be added to all samples when specified in the appropriate determinative method. See Sec. 5.4 for additional guidance on surrogates.

8.4 The laboratory must have procedures in place for documenting and charting the effect of the matrix on method performance. Refer to Chapter One and Method 8000 for specific guidance on developing method performance data.

9.0 METHOD PERFORMANCE

9.1 The recovery of surrogate standards is used to monitor unusual matrix effects, sample processing problems, etc, in each sample. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.

9.2 The performance of each 5000 series method will be dictated by the overall performance of the sample preparation in combination with the analytical determinative method.

10.0 REFERENCES

None required.

TABLE 1

COMBINATIONS OF VOLATILE ORGANIC SAMPLE PREPARATION
AND DETERMINATIVE METHODS FOR SW-846

| Determinative Method | | Preparation Methods | | | |
|----------------------|--|---------------------|------------------------|---------------|-----------------------|
| No. | Method Title | Aqueous Samples | Soil/Solid Samples | Waste Samples | Air Samples |
| 8011 | EDB & DBCP by GC/ECD | 8011 | None listed | None listed | None listed |
| 8015 | Nonhalogenated VOCs by GC/FID | 5030, 5031, 5032 | 5021, 5031, 5032, 5035 | 5032, 5035 | None listed |
| 8021 | Aromatic and Halogenated VOCs by GC/ELCD & PID | 5030, 5032 | 5021, 5032, 5035 | 5032, 5035 | None listed |
| 8031 | Acrylonitrile by GC/NPD | 8031, 5030, 5032 | 5032, 5035 | 5032, 5035 | None listed |
| 8032 | Acrylamide by GC/ECD | 8032 | None listed | None listed | None listed |
| 8033 | Acetonitrile by GC/NPD | 5031 | None listed | None listed | None listed |
| 8260 | Volatile Organic Compounds by GC/MS | 5030, 5031, 5032 | 5021, 5031, 5032, 5035 | 5032, 5035 | 0030, 0031/5041, 0040 |
| 8315 | Carbonyl Compounds by HPLC | 8315 | 8315 | 8315 | 0011, 0100/8315 |
| 8316 | Acrylamide and Acrylonitrile by HPLC | 8316 | None listed | None listed | None listed |

DBCP = 1,2-Dibromo-3-chloropropane
 EDB = Ethylene dibromide (1,2-dibromoethane)
 VOCs = Volatile Organic Compounds
 GC = Gas Chromatography
 ECD = Electron Capture Detector
 ELCD = Electrolytic Conductivity Detector
 FID = Flame Ionization Detector
 HPLC = High Performance Liquid Chromatography
 MS = Mass Spectrometry
 PID = Photoionization Detector