METHOD 8111

HALOETHERS BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8111 is a gas chromatographic method used to determine the concentration of haloethers. It describes wide-bore, open tubular, capillary column gas chromatography procedures using a dual column/dual detector approach. However, a single column/single detector approach is acceptable. The following Resource Conservation and Recovery Act (RCRA) analytes have been determined by this method:

Compound	CAS No.[a]
Bis(2-chloroethoxy) methane	111-91-1
Bis(2-chloroethyl) ether	111-44-4
Bis(2-chloro-1-methylethyl) ether[b]	108-60-1
4-Chlorophenyl phenyl ether	7005-72-3

[a] Chemical Abstracts Service (CAS) Registry Number

[b] Chemical name was changed by the Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This compound is also known as 2,2'-oxybis(1-chloropropane) (CAS index name). See the link at http://www.epa.gov/iris/subst/0407.htm, Section VII for the "Revision History" and Section VIII, for "Synonyms" of this chemical.

1.2 The following additional haloether compounds may also be analyzed by this method:

Compound	CAS No.
4-Bromophenyl phenyl ether	101-55-3
2-Chlorophenyl 4-nitrophenyl ether	209-61-4
3-Chlorophenyl 4-nitrophenyl ether	2303-23-3
4-Chlorophenyl 4-nitrophenyl ether	1836-74-4
2,4-Dibromophenyl 4-nitrophenyl ether	2671-93-4
2,4-Dichlorophenyl 3-methyl-4-nitrophenyl ether	42488-57-3
2,6-Dichlorophenyl 4-nitrophenyl ether	2093-28-9
3,5-Dichlorophenyl 4-nitrophenyl ether	NA
2,5-Dichlorophenyl 4-nitrophenyl ether	391-48-7
2,4-Dichlorophenyl 4-nitrophenyl ether	1836-75-5
2,3-Dichlorophenyl 4-nitrophenyl ether	82239-20-1
3,4-Dichlorophenyl 4-nitrophenyl ether	22532-80-5
4-Nitrophenyl phenyl ether	620-88-2
2,4,6-Trichlorophenyl 4-nitrophenyl ether	1836-77-7
2,3,6-Trichlorophenyl 4-nitrophenyl ether	NA
2,3,5-Trichlorophenyl 4-nitrophenyl ether	NA
2,4,5-Trichlorophenyl 4-nitrophenyl ether	22532-68-9
3,4,5-Trichlorophenyl 4-nitrophenyl ether	NA

2,3,4-Trichlorophenyl 4-nitrophenyl ether	NA

NA = CAS numbers not assigned at this time.

1.3 This method is restricted to use by, or under supervision of, analysts experienced in the use of a gas chromatograph (GC) and in the interpretation of gas chromatograms.

2.0 SUMMARY OF METHOD

2.1 Method 8111 provides GC conditions for the detection of ppb concentrations of haloethers in water and soil or ppm concentrations in waste samples.

2.2 Prior to use of this method, appropriate sample extraction techniques must be used for environmental samples (refer to Chapter Two and Method 3500).

2.3 Both neat and diluted organic liquids (Method 3580) may be analyzed by direct injection.

2.4 Analysis is accomplished by GC utilizing an instrument equipped with a wide-bore capillary column and an electron capture detector (ECD).

3.0 DEFINITIONS

Refer to Chapter One, the individual determinative methods, and the manufacturer's instructions for definitions that may be relevant.

4.0 INTERFERENCES

4.1 Refer to Methods 3500, 3600, and 8000.

4.2 The ECD responds to all electronegative compounds. Therefore, interferences are possible by other halogenated compounds, as well as phthalates and other oxygenated compounds such as organonitrogen, organosulfur, and organophosphorus compounds. Second column confirmation or gas chromatography/mass spectrometry (GC/MS) confirmation is necessary to ensure proper analyte identification unless previous characterization of the sample source will ensure proper identification.

4.3 Dichlorobenzenes are known to co-elute with haloethers under some GC conditions. If these materials are present in a sample, it may be necessary to analyze the extract with two different column packings to completely resolve all of the compounds.

4.4 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. To reduce carryover, the syringe used for injection must be thoroughly rinsed between samples with solvent. Whenever a highly concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Additional solvent blanks interspersed with the sample extracts should be considered whenever the analysis of a solvent blank indicates cross-contamination problems.

4.5 Some compounds co-elute using this procedure. In these cases, the compounds must be reported as co-eluting. The mixture may be reanalyzed for peak confirmation by GC/MS techniques if concentration permits (see Method 8270).

4.6 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the condition of the analysis, by analyzing reagent blanks.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Health and Safety Administration (OSHA) regulations regarding the safe handling of the chemicals and instrumentation included in this method. A reference file of material safety data sheets should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC – An analytical system complete with a GC suitable for on-column and split/splitless injection, and all necessary accessories, including syringes, analytical columns, gases, and two ECDs. A data system for measuring peak areas and/or peak heights is recommended.

6.2 Suggested GC columns – Alternative columns may be used to provide the separation required to resolve all target analytes listed in Sec. 1.1 of this method. Refer to Chapter 1 and Method 8000 for additional information regarding quality control (QC) requirements and column performance.

6.2.1 Column 1 – 30 m x 0.53 mm ID fused silica open tubular column, cross-linked and chemically bonded with 95 percent dimethyl and 5 percent diphenyl-polysiloxane (DB-5, RT_x-5, SPB-5, or equivalent), 0.83 µm or 1.5 µm film thickness

6.2.2 Column 2 – 30 m x 0.53 mm ID fused silica open tubular column cross-linked and chemically bonded with 14 percent cyanopropylphenyl and 86 percent dimethyl-polysiloxane (DB-1701, RT_x-1701, or equivalent), 1.0 µm film thickness

6.3 Column rinsing kit (optional) – Bonded-phase column rinse kit (Agilent, Catalog no. 430-3000 or equivalent)

6.4 Microsyringes – 100 µL, 50 µL, 10 µL (Hamilton 701 N or equivalent), and 50 µL (Blunted, Hamilton 705SNR or equivalent)

6.5 Balances – Analytical, capable of accurately weighing 0.0001 g, and top-loading, capable of accurately weighing 0.01 g

6.6 Volumetric flasks, Class A – 10 mL to 1000 mL

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Solvents – All solvents must be pesticide quality or equivalent.

7.2.1 Hexane, C_6H_{14}

7.2.2 Acetone, CH_3COCH_3

7.2.3 Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$

7.3 Stock standard solutions (1000 mg/L) – May be prepared from pure standard materials or can be purchased as certified solutions

7.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially prepared stock standard solutions may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.3.2 For those compounds which are not adequately soluble in hexane or isooctane, dissolve the compound initially with a small volume of toluene, ethyl acetate or acetone and dilute to volume with isooctane or hexane.

7.4 Composite stock standard – This standard can be prepared from individual stock solutions. For composite stock standards containing less than twenty-five components, transfer exactly 1 mL of each individual stock solution at 1000 mg/L, add solvent, mix the solutions, and bring to volume in a 25 mL volumetric flask. For example, for a composite containing twenty individual standards, the resulting concentration of each component in the mixture, after the volume is adjusted to 25 mL, will be 40 mg/L. This composite solution can be further diluted to obtain the desired concentrations.

7.5 Calibration standards – These should be prepared at a minimum of five concentrations with dilution of the composite stock standard with isooctane or hexane. The standard concentrations should correspond to the expected range of concentrations present in the field samples and should bracket the linear range of the detector.

7.6 Recommended internal standard – Prepare a solution of 1000 mg/L of 4,4'-dibromobiphenyl. For spiking, dilute this solution to 50 ng/µL. (This concentration may need to be more dilute depending on the detector sensitivity. The internal standard response should be approximately 50 to 90% of full scale.) Use a spiking volume of 10 µL/ml of extract. The spiking concentration of the internal standards should be kept constant for all samples and calibration standards. Store the internal standard spiking solutions at 4 °C in polytetrafluoroethylene (PTFE) sealed containers in the dark.

7.7 Recommended surrogate standards – The performance of the method should be monitored using surrogate compounds. Surrogates are added to all samples, method blanks, matrix spikes, and calibration standards. Prepare a solution of 1000 mg/L each of 2,4-dichlorodiphenyl ether and 2,3,4-trichlorodiphenyl ether and dilute them to 20 ng/µL. Use a

spiking volume of 100 µL for a 1 L aqueous sample. (This concentration may need to be adjusted depending on the detector sensitivity. The surrogate standard response should be approximately 100% of full scale.)

7.8 Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4°C or cooler in PTFE-sealed containers in the dark. All standard solutions must be replaced after six months or sooner if routine QC (Sec. 9.0) indicates a problem.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See Chapter 4, Organic Analytes, Sec. 4.1 for more information on this topic.

8.2 Extracts should be stored in the dark at or below 4 °C and be analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 General Guidance

Refer to Chapter One for guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results.

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged, as described in Sec. 9.6 of Method 8000. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency (IDP)

Prior to implementation of a method, each laboratory must perform an IDP consisting of at least four replicate reference samples spiked into a clean matrix taken through the entire sample preparation and analysis. Whenever a significant change to instrumentation or procedure occurs, the laboratory must demonstrate that acceptable precision and bias can still be obtained by the changed conditions. Whenever new staff members are trained, an analyst IDP must be performed.

9.2.1 Demonstration of proficiency for new analysts

Each laboratory should have a training program which documents that a new analyst is capable of performing the method, or portion of the method, for which the analyst is responsible. This demonstration should document that the new analyst is capable of successfully following the Standard Operating Procedure (SOP) established by the laboratory.

9.3 QC procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 11 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples.

9.4 Sample quality control for preparation and analysis – The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

9.4.1 Before processing any samples, the analyst should demonstrate (through the analysis of a method blank) that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

9.4.2 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. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on 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, laboratories should use a matrix spike and matrix spike duplicate pair.

9.4.3 An 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 indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that that the laboratory can perform the analysis in a clean matrix.

9.4.4 See Method 8000, Sec. 9 for the details on carrying out sample QC procedures for preparation and analysis.

9.5 Surrogate recoveries – The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 9 for information on evaluating surrogate data and developing and updating surrogate limits.

9.6 It is recommended that the laboratory adopt additional QA 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.

9.7 Lower Limit of Quantitation (LLOQ)

The laboratory shall establish the LLOQ as the lowest point of quantitation, which in most cases, is the lowest concentration in the calibration curve. LLOQ verification is recommended for each project application to validate quantitation capability at low analyte concentration levels. This verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated data quality objectives.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare calibration standards using the procedures in Sec. 7.0. Refer to Method 8000 for proper calibration procedures. The procedure for internal or external calibration may be used.

10.2 Refer to Method 8000 for procedures for establishing retention time windows.

11.0 PROCEDURE

11.1 Extraction and cleanup

11.1.1 Refer to Chapter Two and Method 3500 for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral, or as-is, pH with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using any of the extraction methods for solids listed in Method 3500, as appropriate.

11.1.2 If necessary, the samples may be cleaned up using Method 3620 (Florisil) and/or Method 3640 (Gel Permeation Chromatography). See Chapter Two and Method 3600 for general guidance on cleanup and method selection. Method 3660 may be used for sulfur removal.

11.1.3 Prior to gas chromatographic analysis, the extraction solvent should be exchanged to hexane. The exchange is performed during the Kuderna Danish (KD) procedures listed in each of the extraction methods. Any methylene chloride remaining in the extract will cause a very broad solvent peak.

NOTE: Some of the haloethers are very volatile and significant losses will occur in concentration steps, if care is not exercised. It is important to maintain a constant, gentle evaporation rate and not to allow the liquid volume to fall below 1 to 2 mL before the K-D apparatus is removed from the hot water bath.

11.2 GC conditions – Retention time information for each of the target analytes is presented in Table 1. Retention times of additional haloether compounds are presented in Table 2. GC operating conditions under which these retention times were obtained are provided in the appropriate table. Figures 1 and 2 illustrate typical chromatography of the haloethers.

11.3 Calibration

11.3.1 Prepare calibration standards using the procedures in Sec. 7. Refer to Method 8000 for proper calibration procedures. The procedure for internal or external calibration may be used.

11.3.2 Refer to Method 8000 for procedures for establishing retention time windows.

11.4 Gas chromatographic analysis

11.4.1 Method 8000 provides instructions on calibration, establishing retention time windows, the analysis sequence, appropriate dilutions, and identification criteria.

11.4.2 Automatic injections of 1 µL are recommended. Hand injections of no more than 2 µL may be used if the analyst demonstrates quantitation precision of ≤10 percent relative standard deviation (RSD). The solvent flush technique may be used if the amount of solvent is kept at a minimum. If the internal standard calibration technique is used, add 10 µL of the internal standard to each 1 mL of sample extract prior to injection.

11.4.3 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Normally, confirmation is required. Confirmation techniques include analysis on a second column with dissimilar stationary phase, by GC/MS (full scan or selected ion monitoring (SIM)), or by using a different detector and getting comparable data. See Method 8000 for further information.

11.4.3.1 If partially overlapping or co-eluting peaks are present, install columns with a dissimilar liquid phase or use a GC/MS technique.

11.4.3.2 Interferences that prevent analyte identification and/or quantitation may possibly be removed by the cleanup techniques mentioned above.

11.4.4 Record both the volume injected (to the nearest increment) and the resulting peak size in area units or peak height. Using either the internal or the external calibration procedure (Method 8000), determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

11.4.5 If the responses exceed the linear range of the system, dilute the extract and reanalyze. Peak height measurements are recommended, rather than peak area integration, when overlapping peaks may cause errors in area integration.

11.4.6 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

11.4.7 Determine the concentration of each identified analyte using the calculation formulas in Sec. 11 of Method 8000.

11.5 Instrument maintenance

11.5.1 Injection of sample extracts from waste sites often leaves a high boiling residue in the injection port area, splitters (when used), and the injection port end of the chromatographic column. This residue affects chromatography in many ways (e.g., peak tailing, retention time shifts, analyte degradation, etc.). Therefore, instrument maintenance is very important. Residue buildup in a splitter may limit flow through one leg and, therefore, change the split ratios. If this occurs during an analytical run, the quantitative data may be incorrect. Proper cleanup techniques will minimize the problem and instrument QC will indicate when instrument maintenance is required.

11.5.2 Suggested chromatograph maintenance – Corrective measures may require remedial actions.

11.5.2.1 Column rinsing – The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone; methylene chloride is a satisfactory final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to remain flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen passing through the column.

11.5.2.2 See Method 8000 for additional guidance on corrective action for capillary columns and the injection port.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 See Method 8000, Sec. 11 for information regarding data analysis and calculations.

12.2 Results must be reported in units commensurate with their intended use and all dilutions must be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 Table 1 lists the retention times and recoveries of the target analytes. The recoveries presented were obtained from the analysis of spiked sandy loam soils. No recovery data is currently available on Bis(2-chloro-ethoxy) methane and Bis(2-chloroethyl) ether.

13.2 Table 2 lists the compounds that may be determined by this method and their retention times. Figure 1 shows a chromatogram of the target analytes eluted from a pair of DB-5/DB-1701 columns and detected with ECD under the GC conditions listed in Table 2.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The Environmental Protection Agency (EPA) has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, a free publication available from the ACS, Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the

letter and spirit of any sewer discharge permits and regulations and complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the ACS at the web address listed in Sec. 14.2 above.

16.0 REFERENCES

1. V. Lopez-Avila, E. Baldin, J. Benedicto, J. Milanes and W. F. Beckert, "Application of Open-Tubular Columns to SW-846 GC Methods," final report to the U.S. EPA on Contract 68-03-3511, Mid-Pacific Environmental Laboratory, Mountain View, CA, 1990.

2. S. Tsang, P. Marsden and N.Chau, "Performance Data for Methods 8041, 8091, 8111, and 8121A," draft report to U.S. EPA on Contract 68-W9-0011, Science Applications International Corp., San Diego, CA, 1992.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method.

TABLE 1

RETENTION TIMES AND RECOVERIES OF TARGET HALOETHERS

Analyte	RT (min)	Spiking Conc. (µg/g)	Recovery (%)	RSD (%)
Bis(2-chloro-1-methylethyl) ether	3.06	2.5	112	4.3
4-Chlorophenyl phenyl ether	15.75	5.0	91.5	3.5
4-Bromophenyl phenyl ether	18.21	0.5	97.0	2.1

Column:	DB-5, 30 m x 0.53 mm ID
Temperature program:	125 °C (1.0 min hold) to 135 °C at 2 °C/min 135 °C to 200 °C at 5 °C/min 200 °C to 275 °C at 10 °C/min, (3.5 min hold)
Injector:	Packed, wide-bore liner
Injector temperature:	200 °C
Detector:	ECD
Detector temperature:	320 °C
Nitrogen carrier gas:	5 mL/min
Nitrogen makeup gas:	55 mL/min

TABLE 2

RETENTION TIMES OF ADDITIONAL HALOETHER COMPOUNDS

Peak[a]	Compound	Retention Time (min)	
		DB-5	DB-1701
1	4-Bromophenyl-phenyl ether	4.28	5.57
2	Phenyl 4-nitrophenyl ether	6.85	10.86
3	2-Chlorophenyl 4-nitrophenyl ether	10.44	16.31
4	3-Chlorophenyl 4-nitrophenyl ether	10.78	16.70
5	4-Chlorophenyl 4-nitrophenyl ether	11.37	17.68
6	2,6-Dichlorophenyl 4-nitrophenyl ether	14.02	20.84
7	3,5-Dichlorophenyl 4-nitrophenyl ether	14.55	21.33
8	2,5-Dichlorophenyl 4-nitrophenyl ether	14.55	21.54
9	2,4-Dichlorophenyl 4-nitrophenyl ether	15.08	22.30
10	2,3-Dichlorophenyl 4-nitrophenyl ether	16.11	23.87
11	3,4-Dichlorophenyl 4-nitrophenyl ether	16.65	24.54
12	2,4,6-Trichlorophenyl 4-nitrophenyl ether	17.89	24.93
13	2,3,6-Trichlorophenyl 4-nitrophenyl ether	19.40	27.27
14	2,3,5-Trichlorophenyl 4-nitrophenyl ether	19.70	27.56
15	2,4,5-Trichlorophenyl 4-nitrophenyl ether	20.03	28.05
16	2,4-Dibromophenyl 4-nitrophenyl ether	21.63	30.03
17	3,4,5-Trichlorophenyl 4-nitrophenyl ether	21.83	30.42
18	2,3,4-Trichlorophenyl 4-nitrophenyl ether	22.28	31.18
19	2,4-Dichlorophenyl 3-methyl-4-nitrophenyl ether	21.83	31.60
IS	4,4'-Dibromobiphenyl	9.44	12.66
SU-1	2,4-Dichlorodiphenyl ether	4.82	6.17
SU-2	2,3,4-Trichlorodiphenyl ether	8.31	10.95

[a] Peak numbers refer to the chromatogram in Figure 2.

IS=Internal Standard
SU=Surrogate

The GC operating conditions for the above analysis were as follows:

Columns:	30 m x 0.53 mm ID DB-5 (0.83 µm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0 µm film thickness) connected to an 8 in. injection tee (Supelco, Inc.).
Temperature program:	180 °C (0.5 min hold) to 260 °C (1.0 min hold) at 2 °C/min
Injector temperature:	250 °C
Detector temperature:	320 °C
Helium carrier gas:	6 mL/min
Nitrogen makeup gas:	20 mL/min

FIGURE 1

GC/ECD CHROMATOGRAM OF TARGET ANALYTE HALOETHERS
ANALYZED ON A DB-5 CAPILLARY COLUMN FOR RECOVERY STUDIES

The GC operating conditions are listed in Table 1.

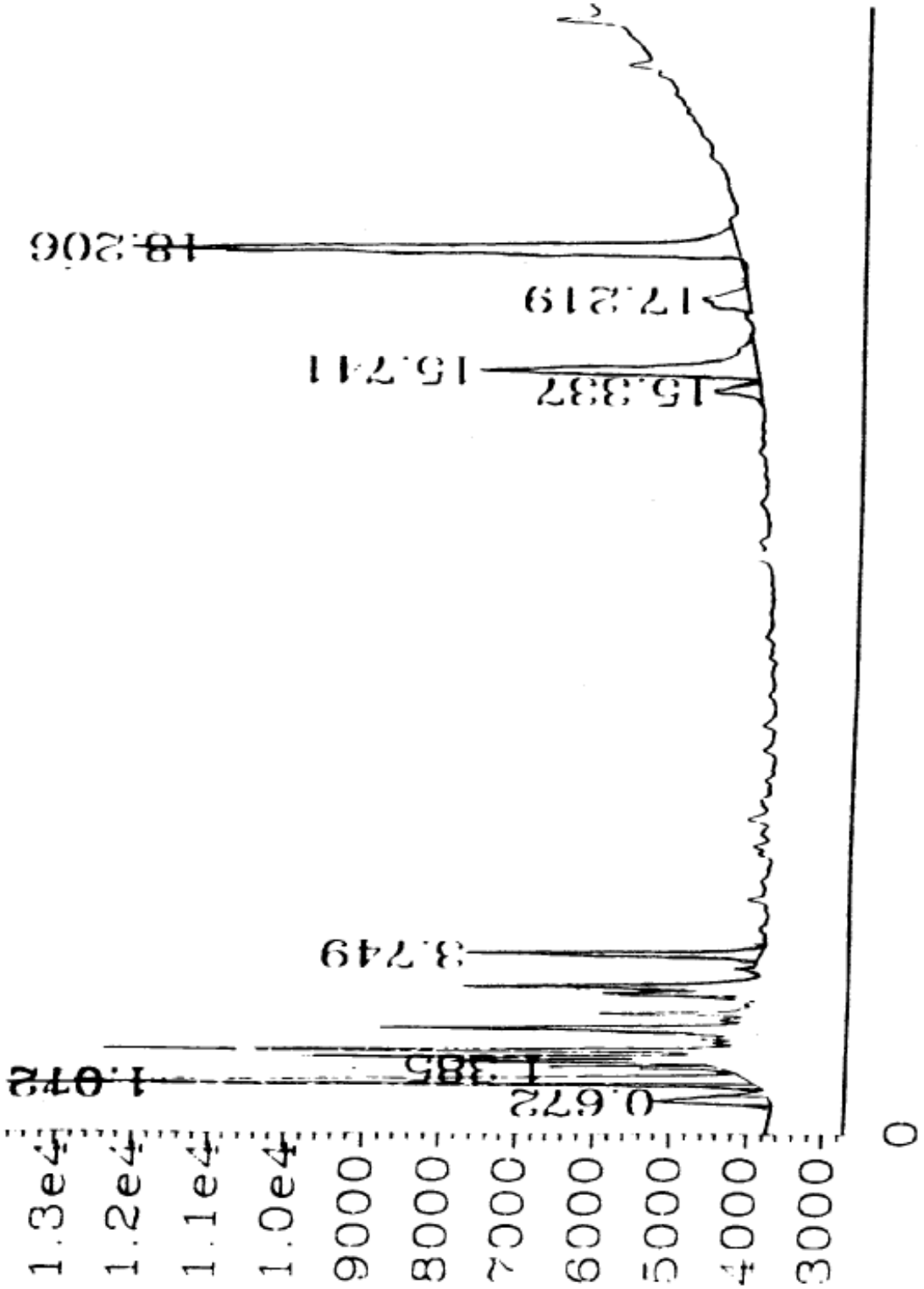
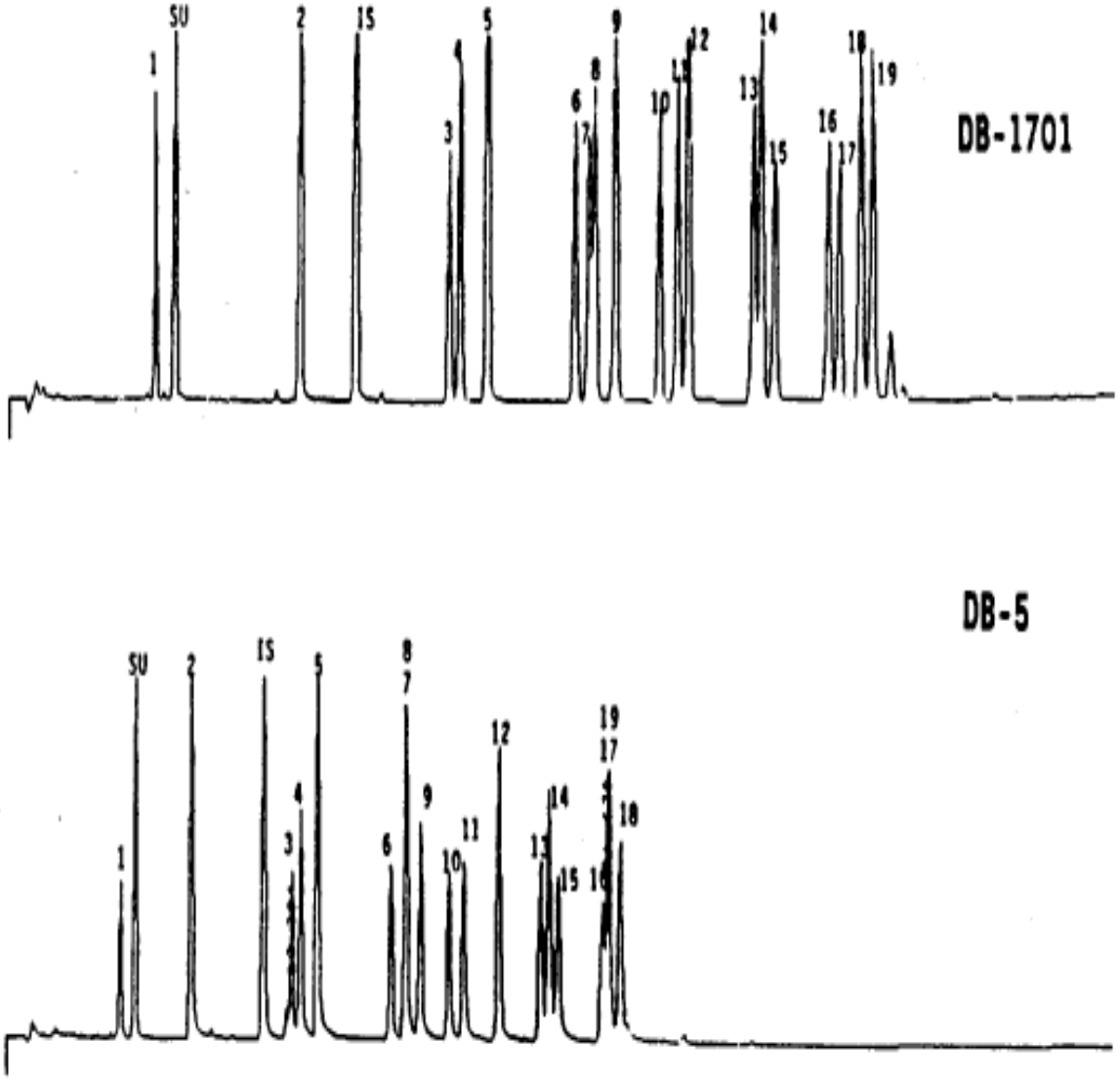
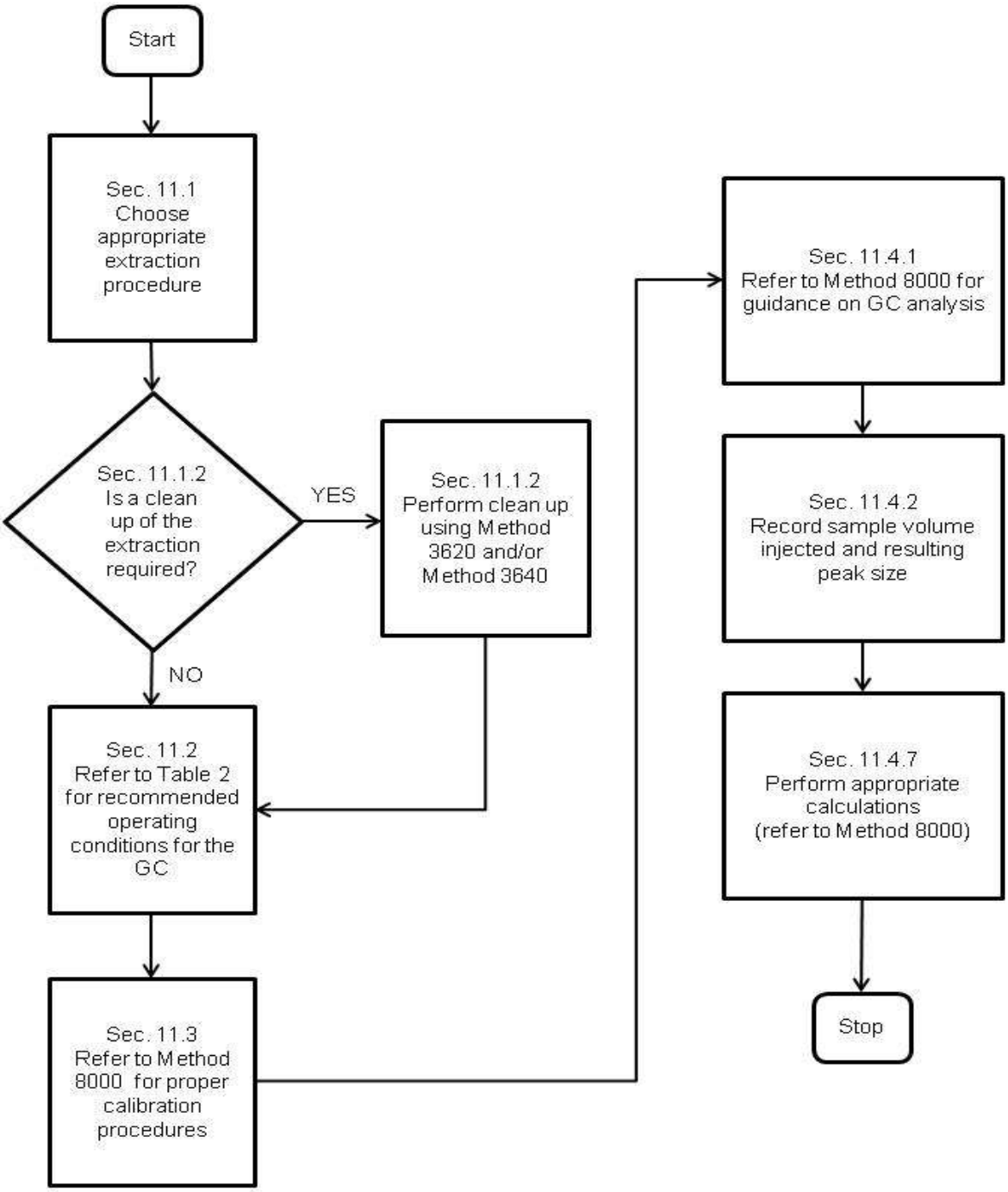


FIGURE 2

GC/ECD CHROMATOGRAM OF HALOETHERS ANALYZED ON A
DB-5/DB-1701 FUSED-SILICA OPEN-TUBULAR COLUMN PAIR

The GC operating conditions are listed in Table 2.




Start
Sec. 11.1 Choose appropriate extraction procedure
Sec. 11.1.2 Is a clean up of the extraction required?
YES
Sec. 11.1.2 Perform clean up using Method 3620 and/or Method 3640
NO
Sec. 11.2 Refer to Table 2 for recommended operating conditions for the GC
Sec. 11.3 Refer to Method 8000 for proper calibration procedures
Sec. 11.4.1 Refer to Method 8000 for guidance on GC analysis
Sec. 11.4.2 Record sample volume injected and resulting peak size
Sec. 11.4.7 Perform appropriate calculations (refer to Method 8000)
Stop

Appendix A
Changes in this version from the December 1996, Revision 0.

1. Improved overall method formatting for consistency with new SW-846 methods style guidance. The format was updated to Microsoft Word .docx. This includes the additions of all sections required by the new format.

2. Many minor editorial and technical revisions were made throughout to improve method clarity.

3. The revision number was changed to 1 and the date published was changed to July 2014.

4. This appendix was added showing changes from the previous revision.

5. Chemical name was changed by the Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This compound is also known as 2,2'-oxybis(1-chloropropane) (CAS index name). See the link at http://www.epa.gov/iris/subst/0407.htm, Section VII for the "Revision History" and Section VIII, for "Synonyms" of this chemical.

6. The method flowchart on page 15 was updated to reflect the current section numbers.