METHOD 504.1

1,2-DIBROMOETHANE (EDB), 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP), AND 1,2,3-TRICHLOROPROPANE (123TCP) IN WATER BY MICROEXTRACTION AND GAS CHROMATOGRAPHY

T.W. Winfield -- Method 504, Revision 1.0 (1986)

T.W. Winfield -- Method 504, Revision 2.0 (1989)

James W. Eichelberger -- Method 504.1 (1993)

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

METHOD 504.1

1,2-DIBROMOETHANE (EDB), 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TRICHLOROPROPANE (123TCP) IN WATER BY MICROEXTRACTION AND GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 This method[1-4] is applicable to the determination of the following compounds in finished drinking water and groundwater:

Analyte	Chemical Abstract Services Registry Number
1,2-Dibromoethane	106-93-4
1,2-Dibromo-3-Chloropropane	96-12-8
1,2,3-Trichloropropane	96-18-4

1.2 For compounds other than the above mentioned analytes, or for other sample sources, the analyst must demonstrate the usefulness of the method by collecting precision and accuracy data on actual samples[5] and provide qualitative confirmation of results by gas chromatography/mass spectrometry (GC/MS)[6].

1.3 The experimentally determined method detection limits (MDL)[7] for EDB and DBCP were calculated to be 0.01 µg/L and for 123TCP was calculated to be 0.02 µg/L. The method has been useful for these analytes over a concentration range from approximately 0.03-200 µg/L. Actual detection limits are highly dependent upon the characteristics of the gas chromatographic system used.

2.0 SUMMARY OF METHOD

2.1 Thirty-five mL of sample are extracted with 2 mL of hexane. Two µL of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis. Aqueous calibration standards are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.2 The extraction and analysis time is 30-50 minutes per sample depending upon the analytical conditions chosen.

2.3 Confirmatory evidence can be obtained using a dissimilar column. When component concentrations are sufficiently high, Method 524.1 or 524.2 may be employed for improved specificity.

3.0 DEFINITIONS

3.1 Laboratory Duplicates (LD1 and LD2) -- Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.2 Field Duplicates (FD1 and FD2) -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.3 Laboratory Reagent Blank (LRB) -- An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.4 Field Reagent Blank (FRB) -- An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

3.5 Instrument Performance Check Solution (IPC) -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.6 Laboratory Fortified Blank (LFB) -- An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.7 Laboratory Fortified Sample Matrix (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.8 Stock Standard Solution (SSS) -- A concentrated solution containing one or method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.9 Primary Dilution Standard Solution (PDS) -- A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

3.10 Calibration Standard (CAL) -- A solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.11 Quality Control Sample (QCS) -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

4.0 INTERFERENCES

4.1 Impurities contained in the extracting solvent usually account for the majority of the analytical problems. Solvent blanks should be analyzed on each new bottle of solvent before use. Indirect daily checks on the extracting solvent are obtained by monitoring the reagent water blanks (Section 7.2.4). Whenever an interference is noted in the reagent water blank, the analyst should reanalyze the extracting solvent. Low level interferences generally can be removed by distillation or column chromatography[4]. When a solvent is purified, preservatives put into the solvent by the manufacturer are removed thus potentially making the shelf-life short. It is generally more economical to obtain a new source of solvent. Interference-free solvent is defined as a solvent containing less than 0.1 µg/L individual analyte interference. Protect interference-free solvents by storing in an area free of organochlorine solvents.

4.2 This liquid/liquid extraction technique efficiently extracts a wide boiling range of non-polar organic compounds and, in addition, extracts polar organic components of the sample with varying efficiencies.

4.3 Current column technology suffers from the fact that EDB at low concentrations may be masked by very high levels of dibromochloromethane (DBCM), a common disinfection byproduct of chlorinating drinking waters.

5.0 SAFETY

5.1 The toxicity and carcinogenicity of chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations

regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available[7-9] for the information of the analyst.

5.2 EDB, DBCP, and 123TCP have all been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled in a hood or glovebox. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 EQUIPMENT AND SUPPLIES

6.1 Sample Containers -- 40 mL screw cap vials each equipped with a Teflon-lined cap. Individual vials shown to contain at least 40.0 mL can be calibrated at the 35.0 mL mark so that volumetric, rather than gravimetric, measurements of sample volumes can be performed. Prior to use, wash vials and septa with detergent and rinse with tap and distilled water. Allow the vials and septa to air dry at room temperature, place in a 105°C oven for one hour, then remove and allow to cool in an area free of organic solvent vapors.

6.2 Vials -- auto sampler, screw cap with Teflon faced septa, 1.8 mL.

6.3 Micro Syringes -- 10, 25, and 100 µL.

6.4 Pipettes -- 2.0 mL and 5.0 mL transfer.

6.5 Standard Solution Storage Containers -- 15 mL bottles with Teflon lined screw caps.

6.6 Gas Chromatography System

6.6.1 The gas chromatograph must be capable of temperature programming and should be equipped with a linearized electron capture detector and a capillary column split/splitless injector.

6.6.2 Two gas chromatography columns are recommended. Column A provides separation of the method analytes without interferences from trihalomethanes (Section 4.3). Column A should be used as the primary analytical column unless routinely occurring analytes are not adequately resolved. Column B is recommended for use as a confirmatory column when GC/MS confirmation is not viable. Retention times for the method analytes on these columns are presented in Table 1.

6.6.3 Column A (primary column) -- DB-1, 30 m x 0.25 mm ID, 1.0 µm film thickness fused silica capillary column or equivalent. The linear velocity of the helium carrier gas should be about 25 cm/sec at 100°C. The column temperature is programmed to hold at 40°C for four

minutes, to increase to 240°C at 10°C/min, and hold at 240°C for five minutes or until all expected compounds have eluted.

6.6.4 Column B (alternative column) -- DB-624, 30 m x 0.32 mm ID, 1.8 µm film thickness fused silica capillary column or equivalent. The linear velocity of the helium carrier gas should be about 25 cm/sec at 100°C. The column temperature is programmed as described in Section 6.6.3.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Hexane extraction solvent -- UV Grade, distilled in glass.

7.1.2 Methyl alcohol -- ACS reagent grade, demonstrated to be free of method analytes above the MDLs.

7.1.3 Sodium chloride, NaCl -- ACS reagent grade, for pretreatment before use, pulverize a batch of NaCl and place in a muffle furnace at room temperature. Increase the temperature to 400°C for 30 minutes. Place in a bottle and cap.

7.1.4 Sodium thiosulfate, $Na_2S_2O_3$ -- ACS reagent grade, for preparation of solution (40 mg/mL), dissolve 1 g of $Na_2S_2O_3$ in reagent water and bring to 25 mL volume in a volumetric flask.

7.2 Reagent Water -- Reagent water is defined as water free of interferences above the analyte MDLs.

7.2.1 Reagent water can be generated by passing tap water through a filter bed containing activated carbon. Change the activated carbon when there is evidence that volatile organic compounds are breaking through the carbon.

7.2.2 A Millipore Super-Q Water System or its equivalent may be used to generate deionized reagent water.

7.2.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water at 100 mL/min for one hour. While still hot, transfer the water to a narrow mouth screw cap bottle with a Teflon seal.

7.2.4 Test reagent water each day it is used by analyzing it according to Section 11.0.

7.3 Stock Standard Solutions -- These solutions may be purchased as certified solutions or prepared from pure standard materials using the following procedures:

7.3.1 Place about 9.8 mL of methanol into a 10 mL ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes and weigh to the nearest 0.1 mg.

7.3.2 Use a 100 µL syringe and immediately add two or more drops of standard material to the flask. Be sure that the standard material falls directly into the alcohol without contacting the neck of the flask.

7.3.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight.

7.3.4 Store stock standard solutions in 15 mL bottles equipped with Teflon lined screw caps. Methanol solutions prepared from liquid analytes are stable for at least four weeks when stored at 4°C.

7.4 Primary Dilution Standard Solutions -- Use stock standard solutions to prepare primary dilution standard solutions that contain all three analytes in methanol. The primary dilution standards should be prepared at concentrations that can be easily diluted to prepare aqueous calibration standards (Section 10.1.1) that will bracket the working concentration range. Store the primary dilution standard solutions with minimal headspace and check frequently for signs of deterioration or evaporation, especially just before preparing calibration standards. The storage time described for stock standard solutions in Section 7.3.4 also applies to primary dilution standard solutions.

7.5 Laboratory Fortified Blank (LFB) Sample Concentrate (0.25 µg/mL) -- Prepare an LFB sample concentrate of 0.25 µg/mL of each analyte from the stock standard solutions prepared in Section 7.3.

7.6 MDL Check Sample Concentrate (0.02 µg/mL) -- Dilute 2 mL of LFB sample concentrate (Section 7.5) to 25 mL with methanol.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection

8.1.1 Replicate field reagent blanks (FRB) must be handled along with each sample set, which is composed of the samples collected from the same general sampling site at approximately the same time. At the laboratory, fill a minimum of two sample bottles with reagent water, seal, and ship to the sampling site along with sample bottles. Wherever a set of samples is shipped and stored, it must be accompanied by the FRB.

8.1.2 Collect all samples in 40 mL bottles into which 3 mg of sodium thiosulfate crystals have been added to the empty bottles just prior to shipping to the sampling site. Alternately, 75 µL of freshly prepared sodium thiosulfate solution (40 mg/mL) may be added to empty 40 mL bottles just prior to sample collection.

8.1.3 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Adjust the flow to about 500 mL/min and collect samples from the flowing stream.

8.1.4 When sampling from a well, fill a wide-mouth bottle or beaker with sample, and carefully fill 40 mL sample bottles.

8.2 Sample Preservation

8.2.1 The samples must be chilled to 4°C or less at the time of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be ≤4°C on arrival at the laboratory.

8.2.2 Sodium thiosulfate -- A dechlorinating agent, must be added to each sample to avoid the possibility of reactions that may occur between residual chlorine and indeterminant contaminants present in some solvents, yielding compounds that may subsequently interfere with the analysis. The presence of sodium thiosulfate will arrest the formation of DBCM (See Section 4.3). Adjustment of sample pH to prevent biological degradation of method analytes has not been tested. Therefore, the addition of acid to each sample is not recommended.

8.3 Sample Storage

8.3.1 Store samples and field reagent blanks together at 4°C until analysis. The sample storage area must be free of organic solvent vapors.

8.3.2 Because 1,2,3-trichloropropane has been added to the analyte list in this method and has been found to have a 14-day maximum holding time in studies conducted for Method 524.2, all samples must be analyzed within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

9.0 QUALITY CONTROL

9.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of instrument performance check solutions (IPC), laboratory reagent blanks (LRB),

laboratory fortified blanks (LFB), laboratory fortified sample matrix (LFM), and quality control samples (QCS) to evaluate and document data quality. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst must make an initial determination of the method detection limits and demonstrate the ability to generate acceptable precision with this method. This is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 9.2.

9.1.3 Each day, the analyst must analyze a laboratory reagent blank (LRB) and a field reagent blank, if applicable (Section 8.1.1), to demonstrate that interferences from the analytical system are under control before any samples are analyzed. In general, background interferences co-eluting with method analytes should be below the method detection limits.

9.1.4 The laboratory must, on an ongoing basis, demonstrate through the analyses of laboratory fortified blanks (LFB) that the operation of the measurement system is in control. This procedure is described in Section 9.3. The frequency of the LFB analyses is equivalent to 10% of all samples analyzed.

9.1.5 The laboratory should demonstrate the ability to analyze low level samples weekly. The procedure for low level LFB samples is described in Section 9.4.

9.2 To establish the ability to achieve low detection limits and generate acceptable accuracy and precision, the analyst should perform the following operations:

9.2.1 Prepare four to seven samples at 0.02 µg/L by fortifying 35 µL of the MDL check sample concentrate (Section 7.6) into 35 mL aliquots of reagent water in 40 mL bottles. Cap and mix well.

9.2.2 Analyze the well-mixed MDL check samples according to the method beginning in Section 11.0.

9.2.3 Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentrations in µg/L, for each analyte. Then calculate the MDL for each analyte.

9.2.4 For each analyte, X should be between 70% and 130% of the true value. Additionally, the calculated MDL should meet data quality objectives. If the results for all three analytes meet these criteria, the system performance is acceptable and analysis of actual samples can begin. If any analyte fails to meet the data quality objectives on the basis of high variability, correct the source of the problem and repeat the test. It is recommended that the laboratory repeat the MDL determinations on a regular basis.

CAUTION: No attempts to establish low detection limits should be made before instrument optimization and adequate conditioning of both the column and the GC system. Conditioning includes the processing of LFB and LFM samples containing moderate analyte concentrations.

9.3 The laboratory must demonstrate on a frequency equivalent to 10% of the sample load that the measurement system is in control by analyzing an LFB of the analytes at 0.25 µg/L concentration level.

9.3.1 Prepare an LFB sample (0.25 µg/L) by adding 35 µL of LFB concentrate (Section 7.5) to 35 mL of reagent water in a 40 mL bottle.

9.3.2 Immediately analyze the LFB sample according to Section 11.0 and calculate the recovery for each analyte. The recovery should be between 60% and 140% of the expected value.

9.3.3 If the recovery for either analyte falls outside the designated range, the analyte fails the acceptance criteria. A second LFB containing each analyte that failed must be analyzed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test.

9.4 The laboratory should demonstrate the ability to analyze low level samples weekly.

9.4.1 Prepare an MDL check sample (0.02 µg/L) as outlined in Section 9.2.1 and immediately analyze according to the method in Section 11.0.

9.4.2 The instrument response must indicate that the laboratory's MDL is distinguishable from instrument background signal. If it is not, correct the problem and repeat the MDL test in Section 9.2.

9.4.3 For each analyte, the recovery must be between 60% and 140% of the expected value.

9.4.4 When either analyte fails the test, the analyst should repeat the test for that analyte. Repeated failure, however, will confirm a general problem with the measurement system or faulty samples and/or standards. If

this occurs, locate and correct the source of the problem and repeat the test.

9.5 At least quarterly, a quality control sample (QCS) should be analyzed. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem source.

9.6 At least once in every 20 samples, fortify an aliquot of a randomly selected routine sample with known amounts of the analytes. The added concentration should not be less than the background concentration of the sample selected for fortification. To simplify these checks, it would be convenient to use LFM concentrations ≈10X MDL. Over time, recovery should be evaluated on fortified samples from all routine sources.

9.7 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. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration and Standardization

10.1.1 At least three calibration standards are needed; five are recommended. One should contain the method analytes at a concentration near to but greater than the method detection limit (Table 1) for each compound; the other two should be at concentrations that bracket the range expected in samples. For example, if the MDL is 0.01 µg/L, and a sample expected to contain approximately 0.10 µg/L is to be analyzed, aqueous standards should be prepared at concentrations of 0.02 µg/L, 0.10 µg/L, and 0.20 µg/L.

10.1.2 To prepare a calibration standard (CAL), add an appropriate volume of a primary dilution standard solution to an aliquot of reagent water in a volumetric flask. If <20 µL of an alcoholic standard is added to the reagent water, poor precision may result. Use a 25 µL micro syringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask several times. Discard the contents contained in the neck of the flask. Aqueous standards should be prepared fresh and extracted immediately after preparation unless sealed and stored without headspace as described in Section 8.0.

10.1.3 Each day, analyze each calibration standard according to Section 11.0 and tabulate peak area response versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of concentration to response (calibration factor) is a constant over the working range (<20% relative standard deviation), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

10.2 Instrument Performance -- Check the performance of the entire analytical system daily using data gathered from analyses of laboratory reagent blanks, standards, and the instrument performance check standard.

10.2.1 Significant peak tailing of the target compounds in the chromatogram must be corrected. Tailing problems are generally traceable to active sites on the GC column, improper column installation, or problems with the operation of the detector.

10.2.2 Check the precision between replicate analyses. A properly operating system should perform with an average relative standard deviation of <10%. Poor precision is generally traceable to pneumatic leaks, especially at the injection port.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 Remove samples and standards from storage and allow them to reach room temperature.

11.1.2 For samples and field reagent blanks, contained in 40 mL bottles, remove the container cap. Discard a 5 mL volume using a 5 mL transfer pipette or 10 mL graduated cylinder. Replace the container cap and weigh the container with contents to the nearest 0.1 g and record this weight for subsequent sample volume determination (Section 11.3).

11.1.3 For calibration standards, laboratory fortified blanks and laboratory reagent blanks, measure a 35 mL volume using a 50 mL graduated cylinder and transfer it to a 40 mL sample container.

11.2 Microextraction and Analysis

11.2.1 Remove the container cap and add 6 g NaCl (Section 7.1.3) to the sample.

11.2.2 Recap the sample container and dissolve the NaCl by shaking for about 20 seconds.

11.2.3 Remove the cap and, using a transfer pipette, add 2.0 mL of hexane. Recap and shake vigorously for one minute. Allow the water and hexane phases to separate. (If stored at this stage, keep the container upside down.)

11.2.4 Remove the cap and carefully transfer 0.5 mL of the hexane layer into an autoinjector using a disposable glass pipette.

11.2.5 Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second autoinjector vial. Reserve this second vial at 4°C for a reanalysis if necessary.

11.2.6 Transfer the first sample vial to an autoinjector set up to inject 2.0 µL portions into the gas chromatograph for analysis. Alternatively, 2 µL portions of samples, blanks and standards may be manually injected, although an autoinjector is recommended.

11.3 Determination of Sample Volume

11.3.1 For samples and field blanks, remove the cap from the sample container.

11.3.2 Discard the remaining sample/hexane mixture. Shake off the remaining few drops using short, brisk wrist movements.

11.3.3 Reweigh the empty container with original cap and calculate the net weight of sample by difference to the nearest 0.1 g. This net weight (grams) is equivalent to the volume of water (in mL) extracted (Section 12.3).

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Identify the method analytes in the sample chromatogram by comparing the retention time of the suspect peaks to retention times of the calibration standards and the laboratory control standards analyzed using identical conditions.

12.2 Use the calibration curve or calibration factor (Section 10.1.3) to directly calculate the uncorrected concentration (C_i) of each analyte in the sample (e.g., calibration factor x response).

12.3 Calculate the sample volume (V_s) as equal to the net sample weight: V_s = gross weight (Section 11.1.2) - bottle tare (Section 11.3.3).

12.4 Calculate the corrected sample concentration as:

$$\text{Concentration } (\mu g/L) = C_i \times \frac{35}{V_s}$$

12.5 Results should be reported with an appropriate number of significant figures. Experience indicates that three significant figures may be used for concentrations above 99 µg/L, two significant figures for concentrations between 1-99 µg/L, and one significant figure for lower concentrations.

13.0 METHOD PERFORMANCE

13.1 Single laboratory accuracy and precision data are presented for the three method analytes in reagent water at concentrations of 0.1 µg/L and 0.2 µg/L[11]. Table 2 lists the data generated using Column A and Table 3 lists data gathered using Column B. The method detection limits are presented in Table 1.

14.0 POLLUTION PREVENTION

14.1 This method utilizes a microextraction procedure that requires the use of very small volumes of hexane, thus making this method safe for use by the laboratory analyst and harmless to the environment. For information concerning pollution prevention that may be applicable to laboratory operations, consult "Less is Better: Laboratory Chemical Management for Waste Reduction" available from the American Chemical Society's Department of Government Relations, and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

15.0 WASTE MANAGEMENT

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing the waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations. For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel," also available from the American Chemical Society at the address in Section 14.1.

16.0 REFERENCES

1. Winfield, T.W., Longbottom, J.E., Graves R.L., and Cohen, A.L. "Analysis of Organohalide Pesticides and Commercial PCB Products in Drinking Water by Microextraction and Gas Chromatography," U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

2. Glaze, W.W. and Lin, C.C. "Optimization of Liquid-Liquid Extraction Methods for Analysis of Organics in Water," EPA-600/S4-83-052, January 1984.

3. Henderson, J.E., Peyton, G.R., and Glaze, W.H. (1976). In Identification and Analysis of Organic Pollutants in Water (L.H. Keith ed.), pp. 105-111. Ann Arbor Sci. Publ., Ann Arbor, Michigan.

4. Richard, J.J. and Junk, G.A. "Liquid Extraction for Rapid Determination of Halomethanes in Water," Journal AWWA, 69, 62, January 1977.

5. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory - Cincinnati, Ohio 45268, March 1979.

6. Budde, W.L. and Eichelberger, J.W. "Organic Analyses Using Gas Chromatography/Mass Spectrometry," Ann Arbor Science, Ann Arbor, Michigan 1979.

7. Glaser, J.A., Forest, D.L., McKee, G.D., Quave, S.A., and Budde, W.L. "Trace Analyses for Wastewaters," Environmental Science and Technology, 15, 1426 (1981).

8. "Carcinogens-Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute of Occupational Safety and Health, Publication No. 77-206, August 1977.

9. OSHA Safety and Health Standards, (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206.

10. Safety in Academic Chemistry Laboratories, American Chemical Society Publication, Committee on Chemical Safety, 4th Edition, 1985.

11. Report on the Feasibility of Including 1,2,3-Trichloropropane in Method 504, Technology Applications, Inc., August 27, 1992.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS FOR METHOD ANALYTES USING CONDITIONS IN SECTION 6.6.3

Analyte	Retention Time (min)		MDL (µg/L)
	Column A	Column B	
EDB	9.37	12.47	0.01
123TCP	12.00	15.37	0.02
DBCP	17.3	15.0	0.01

MDLs were calculated from 8 replicate samples fortified at a concentration of 0.04 µg/L of each analyte.

TABLE 2. ACCURACY AND PRECISION USING COLUMN A

Analytes Fortified at 0.10 µg/L				Analytes Fortified at 0.20 µg/L			
	Recovered Concentration (µg/L)				Recovery Concentration (µg/L)		
Replicate #	**EDB**	**123TCP**	**DBCP**	**Replicate #**	**EDB**	**123TCP**	**DBCP**
1	0.1098	0.1157	0.1093	1	0.2473	0.2175	0.2171
2	0.1121	0.1090	0.1108	2	0.2640	0.2232	0.2209
3	0.1109	0.1144	0.1114	3	0.2767	0.2214	0.2160
4	0.1125	0.1041	0.1118	4	0.3114	0.2186	0.2196
5	0.1133	0.1101	0.1088	5	0.3146	0.2186	0.2160
6	0.1228	0.1085	0.1122	6	0.2838	0.2307	0.2162
7	0.1370	0.1139	0.1090	7	0.3126	0.2258	0.2257
mean	0.1169	0.1108	0.1105	mean	0.2872	0.2223	0.2188
STD DEV (n-1)	0.0098	0.0041	0.0014	STD DEV (n-1)	0.0266	0.0047	0.0036
Spk Lev, µg/L	0.1000	0.1000	0.1000	Spk Lev, µg/L	0.2000	0.2000	0.2000
% RECOVERY	116.9	110.8	110.5	% RECOVERY	143.6	111.1	109.4
% RSD	8.41	3.69	1.28	% RSD	9.25	2.13	1.65

TABLE 3. ACCURACY AND PRECISION USING COLUMN B

Analytes Fortified at 0.10 µg/L				Analytes Fortified at 0.20 µg/L			
	Recovered Concentration (µg/L)				Recovery Concentration (µg/L)		
Replicate #	**EDB**	**123TCP**	**DBCP**	**Replicate #**	**EDB**	**123TCP**	**DBCP**
1	0.1010	0.0718	0.0989	1	0.2361	0.1789	0.2164
2	0.1086	0.0915	0.1085	2	0.2486	0.1859	0.2309
3	0.1068	0.1091	0.1140	3	0.2784	0.2051	0.2199
4	0.1055	0.0894	0.1197	4	0.3099	0.1934	0.2211
5	0.1124	0.0920	0.1129	5	0.3138	0.1979	0.2173
6	0.1182	0.0835	0.1062	6	0.2641	0.2171	0.2205
7	0.1374	0.1060	0.1117	7	0.2924	0.1994	0.2303
mean	0.1128	0.0919	0.1103	mean	0.2776	0.1968	0.2223
STD DEV (n-1)	0.0121	0.0128	0.0066	STD DEV (n-1)	0.0298	0.0125	0.0059
Spk Lev, µg/L	0.1000	0.1000	0.1000	Spk Lev, µg/L	0.2000	0.2000	0.2000
% RECOVERY	112.8	91.9	110.3	% RECOVERY	138.8	98.4	111.2
% RSD	10.74	13.88	5.98	% RSD	10.7	6.36	2.65

TABLE 4. INTERLABORATORY STUDY OF METHOD 504 REGRESSION EQUATIONS FOR RECOVERY AND PRECISION*

Water Type Applicable Conc. Range	1,2-Dibromoethane (0.05 - 6.68) µg/L	1,2-Dibromo-3-chloropropane (0.05 - 6.40) µg/L
Reagent Water		
Single-Analyst Precision	SR = 0.041X + 0.004	SR = 0.065X + 0.000
Overall Precision	S = 0.075X + 0.008	S = 0.143X - 0.000
Recovery	X = 1.072C - 0.006	X = 0.987C - 0.000
Ground Water		
Single-Analyst Precision	SR = 0.046X + 0.002	SR = 0.076X - 0.000
Overall Precision	S = 0.102X + 0.006	S = 0.160X + 0.006
Recovery	X = 1.077C - 0.001	X = 0.972C + 0.007

X = Mean recovery
C = True value for the concentration
*No interlaboratory method validation data is available for 1,2,3-Trichloropropane using Method 504, Revision 3.0.