

Method 525.1
Determination of Organic Compounds
in Drinking Water
by Liquid-Solid Extraction
and Capillary Column
Gas Chromatography/Mass Spectrometry

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Method 525.1

Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry

1. Scope and Application

1.1 This is a general purpose method that provides procedures for determination of organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage. The method is applicable to a wide range of organic compounds that are efficiently partitioned from the water sample onto a C₁₈ organic phase chemically bonded to a solid silica matrix in a cartridge or disk, and sufficiently volatile and thermally stable for gas chromatography. Single-laboratory accuracy and precision data have been determined at two concentrations with two instrument systems for the following compounds:

<i>Compound</i>	<i>MW^a</i>	<i>CAS No.</i>
Acenaphthylene	152	208-96-8
Alachlor	269	15972-60-8
Aldrin	362	309-00-2
Anthracene	178	120-12-7
Atrazine	215	1912-24-9
Benz[a]anthracene	228	56-55-3
Benzo[b]fluoranthene	252	205-99-2
Benzo[k]fluoranthene	252	207-08-9
Benzo[a]pyrene	252	50-32-8
Benzo[g,h,i]perylene	276	191-24-2
Butylbenzyl phthalate	312	85-68-7
<i>Chlordane Components</i>		
α-Chlordane	406	5103-71-9
γ-Chlordane	406	5103-74-2
trans-Nonachlor	440	39765-80-5
2-Chlorobiphenyl	188	2051-60-7
Chrysene	228	218-01-9
Dibenz[a,h]anthracene	278	53-70-3
Di-n-butyl phthalate	278	84-72-2
2,3-dichlorobiphenyl	222	16605-91-7
Diethyl phthalate	222	84-66-2
Bis(2-ethylhexyl) adipate	222	103-23-1
Bis(2-ethylhexyl) phthalate	390	117-81-7
Dimethyl phthalate	194	131-11-3
Endrin	378	72-20-8
Fluorene	166	86-73-7
Heptachlor	370	76-44-8
Heptachlor epoxide	386	1024-57-3
2,2',3,3',4,4',6-Heptachlorobiphenyl	392	52663-71-5

Compound	MW^a	CAS No.
Hexachlorobenzene	282	118-74-1
2,2',4,4',5,6'-Hexachlorobiphenyl	358	6-145-22-4
Hexachlorocyclopentadiene	270	77-47-4
Indeno[1,2,3,c,d]pyrene	276	193-39-5
Lindane	288	58-89-9
Methoxychlor	344	72-43-5
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	426	40186-71-8
2,2,3',4,6-Pentachlorobiphenyl	324	60233-25-2
Pentachlorophenol	264	87-86-5
Phenanthrene	178	85-01-8
Pyrene	202	129-00-0
Simazine	201	122-34-9
2,2',4,4'-Tetrachlorobiphenyl	290	2437-79-8
Toxaphene mixture		8001-35-2
2,4,5-Trichlorobiphenyl	256	15862-07-4

^aMonoisotopic molecular weight calculated from the atomic masses of the isotopes with the smallest masses.

A laboratory may use this method to identify and measure additional analytes after the laboratory obtains acceptable (defined in Section 10) accuracy and precision data for each added analyte.

- 1.2 Method detection limit (MDL) is defined as the statistically calculated minimum amount that can be measured with 99% confidence that the reported value is greater than zero.¹ The MDL is compound dependent and is particularly dependent on extraction efficiency and sample matrix. For the listed analytes, MDLs vary from 0.01-15 µg/L. The concentration calibration range of this method is 0.1-10 µg/L.

2. Summary of Method

- 2.1 Organic compound analytes, internal standards, and surrogates are extracted from a water sample by passing 1 L of sample water through a cartridge or disk containing a solid inorganic matrix coated with a chemically bonded C₁₈ organic phase (liquid-solid extraction, LSE). The organic compounds are eluted from the LSE cartridge or disk with a small quantity of methylene chloride, and concentrated further by evaporation of some of the solvent. The sample components are separated, identified, and measured by injecting an aliquot of the concentrated methylene chloride extract into a high resolution fused silica capillary column of a gas chromatography/mass spectrometry (GC/MS) system. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a data base. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard. Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure.

3. Definitions

- 3.1** Internal standard: A pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.
- 3.2** Surrogate analyte: A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of a surrogate analyte is to monitor method performance with each sample.
- 3.3** Laboratory duplicates (LD1 and LD2): Two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 3.4** 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.
- 3.5** Laboratory reagent blank (LRB): An aliquot of reagent water 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.
- 3.6** Field reagent blank (FRB): Reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including 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.
- 3.7** Laboratory performance check solution (LPC): A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 3.8** Laboratory fortified blank (LFB): An aliquot of reagent water 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 at the required method detection limit.
- 3.9** 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.

- 3.10** Stock standard solution: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assayed reference compound. Stock standard solutions are used to prepare primary dilution standards.
- 3.11** Primary dilution standard solution: 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.
- 3.12** 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.
- 3.13** Quality control sample (QCS): A sample matrix containing method analytes or a solution of method analytes in a water miscible solvent which is used to fortify reagent water or environmental samples. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance with externally prepared test materials.

4. Interferences

- 4.1** During analysis, major contaminant sources are reagents and liquid-solid extraction columns. Analyses of field and laboratory reagent blanks provide information about the presence of contaminants.
- 4.2** Interfering contamination may occur when a sample containing low concentrations of compounds is analyzed immediately after a sample containing relatively high concentrations of compounds. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample containing high concentrations of compounds, a laboratory reagent blank should be analyzed to ensure that accurate values are obtained for the next sample.

5. Safety

- 5.1** The toxicity or carcinogenicity of chemicals used in this method has 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 cited.²⁻⁴
- 5.2** Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin, eyes, etc.

6. Apparatus and Equipment

- 6.1** All glassware must be meticulously cleaned. This may be accomplished by washing with detergent and water, rinsing with water, distilled water, or solvents, air-drying, and heating (where appropriate) in an oven. Volumetric glassware is never heated.
- 6.2** Sample containers. 1 L or 1 qt amber glass bottles fitted with a Teflon-lined screw cap. (Bottles in which high purity solvents were received can be used as sample containers without additional cleaning if they have been handled carefully to avoid contamination during use and after use of original contents.)
- 6.3** Separatory funnels. 2 L and 100 mL with a Teflon stopcock.
- 6.4** Liquid chromatography column reservoirs. Pear-shaped 100 mL or 125 mL vessels without a stopcock but with a ground glass outlet joint sized to fit the liquid-solid extraction column. (Lab Glass, Inc. part no. ML-700-706S, with a 24/40 top outer joint and a 14/35 bottom inner joint, or equivalent). A 14/35 outlet joint fits some commercial cartridges.
- 6.5** Syringe needles. No. 18 or 20 stainless steel.
- 6.6** Vacuum flasks. 1 L or 2 L with solid rubber stoppers.
- 6.7** Volumetric flasks, various sizes.
- 6.8** Laboratory or aspirator vacuum system. Sufficient capacity to maintain a slight vacuum of 13 cm (5 in.) of mercury in the vacuum flask.
- 6.9** Micro syringes, various sizes.
- 6.10** Vials. Various sizes of amber vials with Teflon-lined screw caps.
- 6.11** Drying column. Approximately 1.2 cm x 40 cm with 10 mL graduated collection vial.
- 6.12** Analytical balance. Capable of weighing 0.0001 g accurately.
- 6.13** Fused silica capillary gas chromatography column. Any capillary column that provides adequate resolution, capacity, accuracy, and precision (Section 10) can be used. A 30 m x 0.25 mm id fused silica capillary column coated with a 0.25 μm bonded film of polyphenylmethylsilicone is recommended (J&W DB-5 or equivalent).
- 6.14** Gas chromatograph/mass spectrometer/data system (GC/MS/DS)
- 6.14.1** The GC must be capable of temperature programming and be equipped for splitless/split or on-column capillary injection. The injection tube liner should be quartz and about 3 mm in diameter. The injection system must not allow the analytes to contact hot stainless steel or other metal surfaces that promote decomposition.

- 6.14.2** The GC/MS interface should allow the capillary column or transfer line exit to be placed within a few mm of the ion source. Other interfaces, for example the open split interface, are acceptable as long as the system has adequate sensitivity (see Section 9 for calibration requirements).
- 6.14.3** The mass spectrometer must be capable of electron ionization at a nominal electron energy of 70 eV. The spectrometer must be capable of scanning from 45-450 amu with a complete scan cycle time (including scan overhead) of 1.5 seconds or less. (Scan cycle time = Total MS data acquisition time in seconds divided by number of scans in the chromatogram). The spectrometer must produce a mass spectrum that meets all criteria in Table 1 when 5 ng or less of DFTPP is introduced into the GC. An average spectrum across the DFTPP GC peak may be used to test instrument performance.
- 6.14.4** An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software must have the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectra from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software must also allow integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as defined in Section 9.2.6 (or construction of a second or third order regression calibration curve), calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the equation in Section 12.
- 6.15** Millipore Standard Filter Apparatus, All Glass. This will be used if the disks are to be used to carry out the extraction instead of the cartridges.

7. Reagents And Consumable Materials

- 7.1** Helium carrier gas, as contaminant free as possible.
- 7.2** Liquid-solid extraction (LSE) cartridges. Cartridges are inert non-leaching plastic, for example polypropylene, or glass, and must not contain plasticizers, such as phthalate esters or adipates, that leach into methylene chloride. The cartridges are packed with about 1 gram of silica, or other inert inorganic support, whose surface is modified by chemically bonded octadecyl (C₁₈) groups. The packing must have a narrow size distribution and must not leach organic compounds into methylene chloride. One liter of water should pass through the cartridge in about two hours with the assistance of a slight vacuum of about 13 cm (5 in.) of mercury. Section 10 provides criteria for acceptable LSE cartridges which are available from several commercial suppliers. The extraction disks contain approximately 0.5 grams of 8 μm octadecyl bonded silica uniformly enmeshed in a matrix of inert PFTE fibrils. The size of the disks is 47mm x 0.5mm. As with cartridges, the disks should not contain any organic compounds, either from the PFTE or the bonded silica, which will leach into the methylene chloride eluant. One liter of reagent water should pass through the disks

in 5-20 minutes using a vacuum of about 66 cm (26 in.) of mercury. Section 10 provides criteria for acceptable LSE disks which are available commercially.

7.3 Solvents

7.3.1 Methylene chloride, acetone, toluene and methanol. High purity pesticide quality or equivalent.

7.3.2 Reagent water. Water in which an interferent is not observed at the method detection limit of the compound of interest. Prepare reagent water by passing tap water through a filter bed containing about 0.5 kg of activated carbon or by using a water purification system. Store in clean, narrow-mouth bottles with Teflon-lined septa and screw caps.

7.4 Hydrochloric acid. 6N.

7.5 Sodium sulfate, anhydrous. (Soxhlet extracted with methylene chloride for a minimum of four hours)

7.6 Stock standard solutions. Individual solutions of analytes, surrogates, and internal standards may be purchased as certified solutions or prepared from pure materials. To prepare, add 10 mg (weighed on an analytical balance to 0.1 mg) of the pure material to 1.9 mL of methanol or acetone in a 2 mL volumetric flask, dilute to the mark, and transfer the solution to an amber glass vial. If the analytical standard is available only in quantities smaller than 10 mg, reduce the volume of solvent accordingly. Some polycyclic aromatic hydrocarbons are not soluble in methanol or acetone, and their stock standard solutions are prepared in toluene. Methylene chloride should be avoided as a solvent for standards because its high vapor pressure leads to rapid evaporation and concentration changes. Methanol and acetone are not as volatile as methylene chloride, but their solutions must also be handled with care to avoid evaporation. Compounds 10, 11, and 35 in Table 2 are soluble in acetone. Compounds 12, 13, and 20 in Table 2 are soluble in toluene. If compound purity is certified by the supplier at >96%, the weighed amount can be used without correction to calculate the concentration of the solution (5 µg/µL). Store the amber vials in a dark cool place.

7.7 Primary dilution standard solution. The stock standard solutions are used to prepare a primary dilution standard solution that contains multiple analytes. The recommended solvent for this dilution is acetone. Aliquots of each of the stock standard solutions are combined to produce the primary dilution in which the concentration of the analytes is at least equal to the concentration of the most concentrated calibration solution, that is, 10 ng/µL. Store the primary dilution standard solution in an amber vial in a dark cool place, and check frequently for signs of deterioration or evaporation, especially just before preparing calibration solutions.

7.8 Fortification solution of internal standards and surrogates. Prepare a solution of acenaphthene-D₁₀, phenanthrene-D₁₀, chrysene-D₁₂, and perylene-D₂ in methanol or acetone at a concentration of 500 µg/mL of each. This solution is used in the preparation of the calibration solutions. Dilute a portion of this solution by 10-50 µg/mL and use this solution to fortify the actual water samples (see

Section 11.2). Other surrogates, for example, caffeine-¹⁵N₂ and pyrene-D₁₀ may be included in this solution as needed (a 100 µL aliquot of this 50 µg/mL solution added to 1 liter of water gives a concentration of 5 µg/L of each internal standard or surrogate). Store this solution in an amber vial in a dark cool place.

- 7.9** MS performance check solution. Prepare a 5 ng/µL solution of DFTPP in methylene chloride. Store this solution in an amber vial in a dark cool place.
- 7.10** Calibration solutions (CAL1 through CAL6). Prepare a series of six concentration calibration solutions in acetone which contain all analytes except pentachlorophenol and toxaphene at concentrations of 10 ng/µL, 5 ng/µL, 2 ng/µL, 1 ng/µL, 0.5 ng/µL, and 0.1 ng/µL, with a constant concentration of 5 ng/µL of each internal standard and surrogate in each CAL solution. CAL1 through CAL6 are prepared by combining appropriate aliquots of the primary dilution standard solution (Section 7.7) and the fortification solution (500 µg/mL) of internal standards and surrogates (Section 7.8). Pentachlorophenol is included in this solution at a concentration four times the other analytes. Toxaphene CAL solutions should be prepared as separate solutions at concentrations of 250 ng/µL, 200 ng/µL, 100 ng/µL, 50 ng/µL, 25 ng/µL, and 10 ng/µL. Store these solutions in amber vials in a dark cool place. Check these solutions regularly for signs of deterioration, for example, the appearance of anthraquinone from the oxidation of anthracene.
- 7.11** Reducing agents. Sodium sulfite or sodium arsenite. Sodium thiosulfate is not recommended as it may produce a residue of elemental sulfur that can interfere with some analytes.
- 7.12** Fortification solution for optional recovery standard. Prepare a solution of terphenyl-D₁₄ in methylene chloride at a concentration of 500 µg/mL. An aliquot of this solution may be added (optional) to the extract of the LSE cartridge to check on the recovery of the internal standards in the extraction process.

8. Sample Collection, Preservation, and Handling

- 8.1** Sample collection. When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about two to five minutes). Adjust the flow to about 500 mL/min and collect samples from the flowing stream. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach analytes into water. Automatic samplers that composite samples over time must use refrigerated glass sample containers.
- 8.2** Sample dechlorination and preservation. All samples should be iced or refrigerated at 4°C from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of a reducing agent. Add 40-50 mg of sodium sulfite or sodium arsenite (these may be added as solids with stirring until dissolved) to each liter of water. Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in unchlorinated water. The sample pH

is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of pentachlorophenol.

8.3 Holding time. Samples must be extracted within seven days and the extracts analyzed within 30 days of sample collection.

8.4 Field blanks

8.4.1 Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sampling site along with the empty sample containers. Return the FRB to the laboratory with filled sample bottles.

8.4.2 When hydrochloric acid is added to samples, use the same procedures to add the same amount to the FRB.

9. Calibration

9.1 Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analysis as dictated by results of continuing calibration checks. After initial calibration is successful, a continuing calibration check is required at the beginning of each eight-hour period during which analyses are performed. Additional periodic calibration checks are good laboratory practice.

9.2 Initial Calibration

9.2.1 Calibrate the mass and abundance scales of the MS with calibration compounds and procedures prescribed by the manufacturer with any modifications necessary to meet the requirements in Section 9.2.2.

9.2.2 Inject into the GC a 1 μL aliquot of the 5 ng/ μL DFTPP solution and acquire a mass spectrum that includes data for m/z 45-450. Use GC conditions that produce a narrow (at least five scans per peak) symmetrical peak. If the spectrum does not meet all criteria (Table 1), the MS must be retuned and adjusted to meet all criteria before proceeding with calibration. An average spectrum across the GC peak may be used to evaluate the performance of the system.

9.2.3 Inject a 1 μL aliquot of a medium concentration calibration solution, for example 0.5-2 $\mu\text{g}/\text{L}$, and acquire and store data from m/z 45-450 with a total cycle time (including scan overhead time) of 1.5 seconds or less. Cycle time should be adjusted to measure at least five or more spectra during the elution of each GC peak.

- 9.2.3.1** Multi-ramp temperature program GC conditions. Adjust the helium carrier gas flow rate to about 33 cm/sec. Inject at 45°C and hold in splitless mode for one minute. Heat rapidly to 130°C. At three minutes, start the temperature program: 130-180°C at 12°/min; 180-240°C at 7°/min; 240-320°C at 12°/min. Start data acquisition at five minutes.
- 9.2.3.2** Single ramp linear temperature program. Adjust the helium carrier gas flow rate to about 33 cm/sec. Inject at 40°C and hold in splitless mode for one minute. Heat rapidly to 160°C. At three minutes, start the temperature program: 160-320°C at 6°/min; hold at 320° for two minutes. Start data acquisition at three minutes.
- 9.2.4** Performance criteria for the medium calibration. Examine the stored GC/MS data with the data system software. Figure 1 shows an acceptable total ion chromatogram.
- 9.2.4.1** GC performance. Anthracene and phenanthrene should be separated by baseline. Benz[a]anthracene and chrysene should be separated by a valley whose height is less than 25% of the average peak height of these two compounds. If the valley between benz[a]anthracene and chrysene exceeds 25%, the GC column requires maintenance. See Section 9.3.6.
- 9.2.4.2** MS sensitivity. The GC/MS/DS peak identification software should be able to recognize a GC peak in the appropriate retention time window for each of the compounds in calibration solution, and make correct tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required. See Section 9.3.6.
- 9.2.4.3** Lack of degradation of endrin. Examine a plot of the abundance of m/z 67 in the region of 1.05-1.3 of the retention time of endrin. This is the region of elution of endrin aldehyde, a product of the thermal isomerization of endrin. Confirm that the abundance of m/z 67 at the retention time of endrin aldehyde is <10% of the abundance of m/z 67 produced by endrin. If more than 10% endrin aldehyde is observed, system maintenance is required to correct the problem. See Section 9.3.6.
- 9.2.5** If all performance criteria are met, inject a 1 µL aliquot of each of the other CAL solutions using the same GC/MS conditions.
- 9.2.6** Calculate a response factor (RF) for each analyte and surrogate for each CAL solution using the internal standard whose retention time is nearest the retention time of the analyte or surrogate. Table 2 contains suggested internal standards for each analyte and surrogate, and quantitation ions for all compounds. This calculation is supported in acceptable GC/MS data system software (Section 6.14.4), and many other software programs. RF is a unitless number, but units used to express quantities of analyte and internal standard must be equivalent.

Equation 1

$$RF = \frac{(A_x) (Q_{is})}{(A_{is}) (Q_x)}$$

where:

- A_x = integrated abundance of the quantitation ion of the analyte.
 A_{is} = integrated abundance of the quantitation ion internal standard.
 Q_x = quantity of analyte injected in ng or concentration units.
 Q_{is} = quantity of internal injected in ng or concentration units.
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9.2.6.1 For each analyte and surrogate, calculate the mean RF from the analyses of the six CAL solutions. Calculate the standard deviation (SD) and the relative standard deviation (RSD) from each mean: $RSD = 100 (SD/M)$. If the RSD of any analyte or surrogate mean RF exceeds 30%, either analyze additional aliquots of appropriate CAL solutions to obtain an acceptable RSD of RFs over the entire concentration range, or take action to improve GC/MS performance. See Section 9.2.7.

9.2.7 As an alternative to calculating mean response factors and applying the RSD test, use the GC/MS data system software or other available software to generate a linear, second, or third order regression calibration curve.

9.3 Continuing calibration check. Verify the MS tune and initial calibration at the beginning of each eight hour work shift during which analyses are performed using the following procedure.

9.3.1 Inject a 1 μ L aliquot of the 5 ng/ μ L DFTPP solution and acquire a mass spectrum that includes data for m/z 45–450. If the spectrum does not meet all criteria (Table 1), the MS must be retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.

9.3.2 Inject a 1 μ L aliquot of a medium concentration calibration solution and analyze with the same conditions used during the initial calibration.

9.3.3 Demonstrate acceptable performance for the criteria shown in Section 9.2.4.

9.3.4 Determine that the absolute areas of the quantitation ions of the internal standards and surrogate(s) have not decreased by more than 30% from the areas measured in the most recent continuing calibration check, or by more than 50% from the areas measured during initial calibration. If these areas have decreased by more than these amounts, adjustments must be made to restore system sensitivity. These adjustments may require cleaning of the MS ion source, or other maintenance as indicated in Section 9.3.6, and recalibration. Control charts are useful aids in documenting system sensitivity changes.

- 9.3.5** Calculate the RF for each analyte and surrogate from the data measured in the continuing calibration check. The RF for each analyte and surrogate must be within 30% of the mean value measured in the initial calibration. Alternatively, if a second or third order regression is used, the point from the continuing calibration check for each analyte and surrogate must fall, within the analyst's judgement, on the curve from the initial calibration. If these conditions do not exist, remedial action must be taken which may require reinitial calibration.
- 9.3.6** Some possible remedial actions. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.
- 9.3.6.1** Check and adjust GC and/or MS operating conditions; check the MS resolution, and calibrate the mass scale.
- 9.3.6.2** Clean or replace the splitless injection liner; silanize a new injection liner.
- 9.3.6.3** Flush the GC column with solvent according to manufacturer's instructions.
- 9.3.6.4** Break off a short portion (about 1 meter) of the column from the end near the injector; or replace GC column. This action will cause a change in retention times.
- 9.3.6.5** Prepare fresh CAL solutions, and repeat the initial calibration step.
- 9.3.6.6** Clean the MS ion source and rods (if a quadrupole).
- 9.3.6.7** Replace any components that allow analytes to come into contact with hot metal surfaces.
- 9.3.6.8** Replace the MS electron multiplier, or any other faulty components.

10. Quality Control

- 10.1** Quality control (QC) requirements are the initial demonstration of laboratory capability followed by regular analyses of laboratory reagent blanks, laboratory fortified blanks, and laboratory fortified matrix samples. The laboratory must maintain records to document the quality of the data generated. Additional quality control practices are recommended.
- 10.2** Initial demonstration of low system background and acceptable particle size and packing. Before any samples are analyzed, or any time a new supply of cartridges or disks is received from a supplier, it must be demonstrated that a laboratory reagent blank (LRB) is reasonably free of contamination that would prevent the determination of any analyte of concern. In this same experiment, it must be demonstrated that the

particle size and packing of the LSE cartridge or disk are acceptable. Consistent flow rate is an indication of acceptable particle size distribution and packing.

- 10.2.1** A major source of potential contamination is the liquid-solid extraction (LSE) cartridge which could contain phthalate esters, silicon compounds, and other contaminants that could prevent the determination of method analytes.⁵ Although disks are made of a teflon matrix, they may still contain phthalate materials. Generally, phthalate esters will be leached from the cartridges into methylene chloride and produce a variable background that is equivalent to <2 µg/L in the water sample. If the background contamination is sufficient to prevent accurate and precise analyses, the condition must be corrected before proceeding with the initial demonstration. Figure 2 shows unacceptable background contamination from a poor quality commercial LSE cartridge. The background contamination is the large broad peak, and the small peaks are method analytes present at a concentration equivalent to 2 µg/L. Several sources of LSE cartridges may be evaluated before an acceptable supply is identified.
- 10.2.2** Other sources of background contamination are solvents, reagents, and glassware. Background contamination must be reduced to an acceptable level before proceeding with the next section. In general, background from method analytes should be below the method detection limit.
- 10.2.3** One liter of water should pass through a cartridge in about two hours with a partial vacuum of about 13 cm (5 in.) of mercury. The flow rate through a disk should be about 5-20 minutes for a liter of drinking water, using full aspirator or pump vacuum. The extraction time should not vary unreasonably among a set of LSE cartridges or disks.
- 10.3** Initial demonstration of laboratory accuracy and precision. Analyze four to seven replicates of a laboratory fortified blank containing each analyte of concern at a concentration in the range of 2-5 µg/L (see regulations and maximum contaminant levels for guidance on appropriate concentrations).
- 10.3.1** Prepare each replicate by adding an appropriate aliquot of the primary dilution standard solution, or another certified quality control sample, to reagent water. Analyze each replicate according to the procedures described in Section 11 and on a schedule that results in the analyses of all replicates over a period of several days.
- 10.3.2** Calculate the measured concentration of each analyte in each replicate, the mean concentration of each analyte in all replicates, and mean accuracy (as mean percentage of true value) for each analyte, and the precision (as relative standard deviation, RSD) of the measurements for each analyte. Calculate the MDL of each analyte using the procedures described in Section 13.1.2.¹
- 10.3.3** For each analyte and surrogate, the mean accuracy, expressed as a percentage of the true value, should be 70-130% and the RSD should be <30%. Some analytes, particularly the polycyclic aromatic hydrocarbons with molecular weights >250, are measured at concentrations below 2 µg/L, with a mean

accuracy of 35-130% of true value. The MDLs should be sufficient to detect analytes at the regulatory levels. If these criteria are not met for an analyte, take remedial action and repeat the measurements for that analyte to demonstrate acceptable performance before samples are analyzed.

- 10.3.4** Develop and maintain a system of control charts to plot the precision and accuracy of analyte and surrogate measurements as a function of time. Charting of surrogate recoveries is an especially valuable activity since these are present in every sample and the analytical results will form a significant record of data quality.
- 10.4** Monitor the integrated areas of the quantitation ions of the internal standards and surrogates in continuing calibration checks (see Section 9.3.4). In laboratory fortified blanks or samples, the integrated areas of internal standards and surrogates will not be constant because the volume of the extract will vary (and is difficult to keep constant). But the ratios of the areas should be reasonably constant in laboratory fortified blanks and samples. The addition of 10 μL of the recovery standard, terphenyl- D_{14} (500 $\mu\text{g}/\text{mL}$), to the extract is optional, and may be used to monitor the recovery of internal standards and surrogates in laboratory fortified blanks and samples. Internal standard recovery should be in excess of 70%.
- 10.5** Laboratory reagent blanks. With each batch of samples processed as a group within a work shift, analyze a laboratory reagent blank to determine the background system contamination. Any time a new batch of LSE cartridges or disks is received, or new supplies of other reagents are used, repeat the demonstration of low background described in Section 10.2.
- 10.6** With each batch of samples processed as a group within a work shift, analyze a single laboratory fortified blank (LFB) containing each analyte of concern at a concentration as determined in Section 10.3. If more than 20 samples are included in a batch, analyze a LFB for every 20 samples. Use the procedures described in Section 10.3.3 to evaluate the accuracy of the measurements, and to estimate whether the method detection limits can be obtained. If acceptable accuracy and method detection limits cannot be achieved, the problem must be located and corrected before further samples are analyzed. Add these results to the on-going control charts to document data quality.
- 10.7** Determine that the sample matrix does not contain materials that adversely affect method performance. This is accomplished by analyzing replicates of laboratory fortified matrix samples and ascertaining that the precision, accuracy, and method detection limits of analytes are in the same range as obtained with laboratory fortified blanks. If a variety of different sample matrices are analyzed regularly, for example, drinking water from groundwater and surface water sources, matrix independence should be established for each. A laboratory fortified sample matrix should be analyzed for every 20 samples processed in the same batch.
- 10.8** With each set of field samples a field reagent blank (FRB) should be analyzed. The results of these analyses will help define contamination resulting from field sampling and transportation activities.

- 10.9** At least quarterly, replicates of laboratory fortified blanks should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality (as in Section 10.3).
- 10.10** At least quarterly, analyze a quality control sample from an external source. If measured analyte concentrations are not of acceptable accuracy (Section 10.3.3), check the entire analytical procedure to locate and correct the problem source.
- 10.11** Numerous other quality control measures are incorporated into other parts of this procedure, and serve to alert the analyst to potential problems.

11. Procedure

11.1 Cartridge Extraction

- 11.1.1** Setup the extraction apparatus shown in Figure 3A. The reservoir is not required, but recommended for convenient operation. Water drains from the reservoir through the LSE cartridge and into a syringe needle which is inserted through a rubber stopper into the suction flask. A slight vacuum of 13 cm (5 in.) of mercury is used during all operations with the apparatus. The pressure used is critical as a vacuum greater than 13 cm may result in poor precision. About two hours is required to draw a liter of water through the system.
- 11.1.2** Pour the water sample into the 2 L separatory funnel with the stopcock closed, add 5 mL methanol, and mix well. Residual chlorine should not be present as a reducing agent should have been added at the time of sampling. Also the pH of the sample should be about 2. If residual chlorine is present and/or the pH is >2, the sample may be invalid. Add a 100 μ L aliquot of the fortification solution (50 μ g/mL) for internal standards and surrogates, and mix immediately until homogeneous. The concentration of these compounds in the water should be 5 μ g/L.
- 11.1.3** Flush each cartridge with two 10 mL aliquots of methylene chloride, followed by two 10 mL aliquots of methanol, letting the cartridge drain dry after each flush. These solvent flushes may be accomplished by adding the solvents directly to the solvent reservoir in Figure 3A. Add 10 mL of reagent water to the solvent reservoir, but before the reagent water level drops below the top edge of the packing in the LSE cartridge, open the stopcock of the separatory funnel and begin adding sample water to the solvent reservoir. Close the stopcock when an adequate amount of sample is in the reservoir.
- 11.1.4** Periodically open the stopcock and drain a portion of the sample water into the solvent reservoir. The water sample will drain into the cartridge, and from the exit into the suction flask. Maintain the packing material in the cartridge immersed in water at all times. After all of the sample has passed through the LSE cartridge, wash the separatory funnel and cartridge with 10 mL of reagent water, and draw air through the cartridge for 10 minutes.

11.1.5 Transfer the 125 mL solvent reservoir and LSE cartridge (from Figure 3A) to the elution apparatus (Figure 3B). The same 125 mL solvent reservoir is used for both apparatus. Wash the 2 L separatory funnel with 5 mL of methylene chloride and collect the washings. Close the stopcock on the 100 mL separatory funnel of the elution apparatus, add the washings to the reservoir and enough additional methylene chloride to bring the volume back up to 5 mL and elute the LSE cartridge. Elute the LSE cartridge with an additional 5 mL of methylene chloride (10 mL total). A small amount of nitrogen positive pressure may be used to elute the cartridge. Small amounts of residual water from the LSE cartridge will form an immiscible layer with the methylene chloride in the 100 mL separatory funnel. Open the stopcock and allow the methylene chloride to pass through the drying column packed with anhydrous sodium sulfate (1 in) and into the collection vial. Do not allow the water layer to enter the drying column. Remove the 100 mL separatory funnel and wash the drying column with 2 mL of methylene chloride. Add this to the extract. Concentrate the extract to 1 mL under a gentle stream of nitrogen. If desired, gently warm the extract in a water bath to evaporate to between 0.5-1.0 mL (without gas flow). Do not concentrate the extract to less than 0.5 mL (or dryness) as this will result in losses of analytes. If desired, add an aliquot of the recovery standard to the concentrated extract to check the recovery of the internal standards (see Section 10.4).

11.2 Disk Extraction (This may be manual or automatic)

11.2.1 Preparation of Disks

11.2.1.1 Insert the disk into the 47 mm filter apparatus as shown in Figure 4. Wash the disk with 5mL methylene chloride (MeCl₂) by adding the MeCl₂ to the disk, drawing about half through the disk, allowing it to soak the disk for about a minute, then drawing the remaining MeCl₂ through the disk.

11.2.1.2 Pre-wet the disk with 5 mL methanol (MeOH) by adding the MeOH to the disk and allowing it to soak for about a minute, then drawing most of the remaining MeOH through. A layer of MeOH must be left on the surface of the disk, which should not be allowed to go dry from this point until the end of the sample extraction. **THIS IS A CRITICAL STEP FOR A UNIFORM FLOW AND GOOD RECOVERY.**

11.2.1.3 Rinse the disk with 5 mL reagent water by adding the water to the disk and drawing most through, again leaving a layer on the surface of the disk.

11.2.2 Add 5 mL MeOH per liter of water sample. Mix well.

11.2.3 Add the water sample to the reservoir and turn on the vacuum to begin the extraction. Full aspirator vacuum may be used. Particulate-free water may pass through the disk in as little as ten minutes or less. Extract the entire sample, draining as much water from the sample container as possible.

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- 11.2.4** Remove the filtration top from the vacuum flask, but do not disassemble the reservoir and fritted base. Empty the water from the flask, and insert a suitable sample tube to contain the eluant. The only constraint on the sample tube is that it fit around the drip tip of the fritted base. Reassemble the apparatus.
- 11.2.5** Add 5 mL methylene chloride to the sample bottle, and rinse the inside walls thoroughly. Allow the methylene chloride to settle to the bottom of the bottle, and transfer to the disk with a pipet or syringe, rinsing the sides of the glass filtration reservoir in the process. Draw about half of the methylene chloride through the disk, release the vacuum, and allow the disk to soak for a minute. Draw the remaining methylene chloride through the disk.
- 11.2.6** Repeat the above step twice. Pour the combined eluates through a small funnel with filter paper containing three grams of anhydrous sulfate. Rinse the test tube and sodium sulfate with two 5 mL portions of methylene chloride. Collect all the extract and washings in a concentrator tube.
- 11.2.7** Concentrate the extract to 1 mL under a gentle stream of nitrogen. If desired, gently warm the extract in a water bath or heating block to concentrate to between 0.5 mL and 1 mL. Do not concentrate the extract to less than 0.5 mL, since this will result in losses of analytes.
- 11.3** Analyze a 1-2 μ L aliquot with the GC/MS system under the same conditions used for the initial and continuing calibrations (Section 9.2.3).
- 11.4** At the conclusion of data acquisition, use the same software that was used in the calibration procedure to tentatively identify peaks in retention time windows of interest. Use the data system software to examine the ion abundances of components of the chromatogram. If any ion abundance exceeds the system working range, dilute the sample aliquot and analyze the diluted aliquot.
- 11.5** Identification of analytes. Identify a sample component by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time of the sample component should be within 10 seconds of the time observed for that same compound when a calibration solution was analyzed.
- 11.5.1** In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.
- 11.5.2** Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between

two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.5.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. See Section 9.2.4.1. Acceptable resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the average height of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Benzo[b] and benzo[k]fluoranthene are measured as an isomeric pair.

11.5.4 Phthalate esters and other background components appear in variable quantities in laboratory and field reagent blanks, and generally cannot be accurately measured at levels below about 2 µg/L. Subtraction of the concentration in the blank from the concentration in the sample at or below the 2 µg/L level is not recommended because the concentration of the background in the blank is highly variable.

12. Calculations

12.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations if unique ions with adequate intensities are available for quantitation. For example, although two listed analytes, dibenz[a,h]anthracene and indeno[1,2,3,c,d]pyrene, were not resolved with the GC conditions used, and produced mass spectra containing common ions, concentrations (Tables 3-6) were calculated by measuring appropriate characteristic ions.

12.1.1 Calculate analyte and surrogate concentrations.

Equation 2

$$C_x = \frac{(A_x) (Q_{is})}{(A_{is}) RF V}$$

where:

C_x = concentration of analyte or surrogate in µg/L in the water sample.

A_x = integrated abundance of the quantitation ion of the analyte in the sample.

A_{is} = integrated abundance of the quantitation ion of the analyte in the sample.

Q_{is} = total quantity (in micrograms) of internal standard added to the water sample.

V = original water sample volume in liters.

RF = mean response factor of analyte from the initial calibration.

12.1.2 Alternatively, use the GC/MS system software or other available proven software to compute the concentrations of the analytes and surrogates from the second or third order regression curves.

12.1.3 Calculations should utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty). 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. Method Performance

13.1 Single laboratory accuracy and precision data (Tables 3-7) for each listed analyte was obtained at two concentrations with the same extracts analyzed on more than two different instrument systems. Seven 1 L aliquots of reagent water containing 2 µg/L of each analyte, and five to seven 1 L aliquots of reagent water containing 0.2 µg/L of each analyte were analyzed with this procedure. Tables 8-10 list data gathered using C-18 disks. These data were results from different extracts generated by a volunteer laboratory, Environmental Health Laboratories.

With these data, method detection limits (MDL) were calculated using the formula:

$$MDL = S t_{(n-1, 1-\alpha = 0.99)}$$

where:

$t_{(n-1, 1-\alpha = 0.99)}$ = student's *t* value for the 99% confidence level with *n*-1 degrees of freedom

n = number of replicates

S = standard deviation of replicate analyses

13.2 Problem Compounds

13.2.1 The common phthalate and adipate esters (Compounds 14, 21, and 23-26), which are widely used commercially, appear in variable quantities in laboratory and field reagent blanks, and generally cannot be accurately or precisely measured at levels below about 2 µg/L. Subtraction of the concentration in the blank from the concentration in the sample at or below the 2 µg/L level is not recommended because the concentrations of the background in blanks is highly variable.

13.2.2 Some polycyclic aromatic hydrocarbons are rapidly oxidized and/or chlorinated in water containing residual chlorine. Therefore residual chlorine must be reduced before analysis.

13.2.3 In water free of residual chlorine, some polycyclic aromatic hydrocarbons (for example, Compounds 9, 12, 13, 20, and 35) are not accurately measured because of low recoveries in the extraction process.

13.2.4 Pentachlorophenol No. 40 and hexachlorocyclopentadiene No. 34 may not be accurately measured. Pentachlorophenol is a strong acid and elutes as a broad

weak peak. Hexachlorocyclopentadiene is susceptible to photochemical and thermal decomposition.

References

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Table 1. Ion Abundance Criteria for Bis(perfluorophenyl)phenyl Phosphine (Decafluorotriphenylphosphine, DFTPP)

Mass (M/z)	Relative Abundance Criteria	Purpose of Checkpoint¹
51	10-80% of the base peak	Low mass sensitivity
68	<2% of mass 69	Low mass resolution
70	<2% of mass 69	Low mass resolution
127	10-80% of the base peak	Low-mid mass sensitivity
197	<2% of mass 198	Mid-mass resolution
198	base peak or >50% of 442	Mid-mass resolution and sensitivity
199	5-9% of mass 198	Mid-mass resolution and isotope ratio
275	10-60% of the base peak	Mid-high mass sensitivity
365	>1% of the base peak	Baseline threshold
441	Present and < mass 443	High mass resolution
442	base peak or >50% of 198	High mass resolution and sensitivity
443	15-24% of mass 442	High mass resolution and isotope ratio

¹All ions are used primarily to check the mass measuring accuracy of the mass spectrometer and data system, and this is the most important part of the performance test. The three resolution checks, which include natural abundance isotope ratios, constitute the next most important part of the performance test. The correct setting of the baseline threshold, as indicated by the presence of low intensity ions, is the next most important part of the performance test. Finally, the ion abundance ranges are designed to encourage some standardization to fragmentation patterns.

Table 2. Retention Time Data, Quantitation Ions, and Internal Standard References for Method Analytes

Compound	Compound Number	Retention Time (min:sec)		Quantitation Ion (m/z)	Internal Standard Reference
		A^a	B^b		
Internal standard					
Acenaphthene-D ₁₀	1	4:49	7:45	164	-
Phenanthrene-D ₁₀	2	8:26	11:08	188	-
Chrysene-D ₁₂	3	18:14	19:20	240	-
Surrogate					
Perylene-D ₁₂	4	23:37	22:55	264	3
Target analytes					
Acenaphthylene	5	4:37	7:25	152	1
Aldrin	6	11:21	13:36	66	2
Anthracene	7	8:44	11:20	178	2
Atrazine	8	7:56	10:42	200/215	1/2
Benz[a]anthracene	9	18:06	19:14	228	3
Benzo[b]fluoranthene	10	22:23	22:07	252	3
Benzo[k]fluoranthene	11	22:28	22:07	252	3
Benzo[a]pyrene	12	23:28	22:47	252	3
Benzo[g,h,i]perylene	13	27:56	26:44	276	3
Buthylbenzyl phthalate	14	16:40	18:09	149	2/3
Chlordane Components					
α-Chlordane	15	13:44	15:42	375	2/3
γ-Chlordane	16	13:16	15:18	375	2/3
trans-Nonachlor	17	13:54	15:50	409	2/3
2-Chlorobiphenyl	18	4:56	7:55	188	1
Chrysene	19	18:24	19:23	228	3
Dibenz[a,h]anthracene	20	27:15	25:57	278	3
Di- <i>n</i> -butyl phthalate	21	10:58	13:20	149	2
2,3-Dichlorobiphenyl	22	7:20	10:12	222	1
Diethyl phthalate	23	5:52	8:50	149	1
Di(2-ethylhexyl) phthalate	24	19:19	20:01	149	2/3
Di(2-ethylhexyl) adipate	25	17:17	18:33	129	2/3
Dimethyl phthalate	26	4:26	7:21	163	1
Endrin	27	15:52	16:53	81	2/3
Fluorene	28	6:00	8:53	166	1
Heptaclor	29	10:20	12:45	100/160	2
Heptachlor epoxide	30	12:33	14:40	81/353	2
2,2',3,3',4,4',6-Hepta-chlorobiphenyl	31	18:25	19:25	394/396	3

Table 2. Retention Time Data, Quantitation Ions, and Internal Standard References for Method Analytes

Compound	Compound Number	Retention Time (min:sec)		Quantitation Ion (m/z)	Internal Standard Reference
		A^a	B^b		
Hexachlorobenzene	32	7:37	10:20	284/286	1/2
2,2',4,4',5,6'-Hexachlorobiphenyl	33	14:34	16:30	360	2
Hexachlorocyclopentadiene	34	3:36	6:15	237	1
Indeno[1,2,3,c,d]pyrene	35	27:09	25:50	276	3

Table 2. Retention Time Data, Quantitation Ions, and Internal Standard References for Method Analytes (cont.)

Compound	Compound Number	Retention Time (min:sec)		Quantitation Ion (m/z)	Internal Standard Reference
		A^a	B^b		
Lindane	36	8:17	10:57	181/183	1/2
Methoxychlor	37	18:34	19:30	227	3
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	38	18:38	19:33	430	3
2,2',3',4,6-Pentachlorobiphenyl	39	12:50	15:00	326	2
Pentachlorophenol	40	8:11	10:51	266	2
Phenanthrene	41	8:35	11:13	178	2
Pyrene	42	13:30	15:29	202	2/3
Simazine	43	7:47	10:35	201	1/2
2,2',4,4'-Tetrachlorobiphenyl	44	11:01	13:25	292	2
Toxaphene	45	11:30–23:30	13:00–21:30	159	2
2,4,5-Trichlorobiphenyl	46	9:23	11:59	256	2
Alachlor	47	--	13:19	160	2

^aSingle ramp linear temperature program conditions (Section 9.2.3.2).

^bMulti-ramp linear temperature program conditions (Section 9.2.3.1).

Table 3. Accuracy and Precision Data from Seven Determinations of the Method Analytes at 2 µg/L With Liquid-Solid Extraction and the Ion Trap Mass Spectrometer

Compound Number (Table 2)	True Conc. (µg/L)	Mean Observed Conc. (µg/L)	Std. Dev. (µg/L)	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) (µg/L)
4	5	5.0	0.3	6.0	100	– ^a
5	2	1.9	0.2	11.	95	– ^a
6	2	1.6	0.2	13.	80	– ^a
7	2	1.7	0.1	5.9	85	– ^a
8	2	2.2	0.3	14.	110	– ^a
9	2	1.8	0.2	11.	90	– ^a
10	2	not separated from No. 11; measured with No. 11				
11	2	4.2	0.3	7.1	105	– ^a
12	2	0.8	0.2	25.	40	– ^a
13	2	0.7	0.1	14.	35	– ^a
14	2	2.0	0.3	15.	100	– ^a
15	2	2.0	0.2	10.	100	– ^a
16	2	2.2	0.3	14.	110	– ^a
17	2	2.7	1.0	37.	135	– ^a
18	2	1.9	0.1	5.2	95	– ^a
19	2	2.2	0.1	4.5	110	– ^a
20	2	0.3	0.3	100.	15	– ^a
21	2	2.2	0.3	14.	110	– ^a
22	2	2.3	0.1	4.3	115	– ^a
23	2	2.0	0.3	15.	100	– ^a
24	2	1.9	0.2	11.	95	– ^a
25	2	1.6	0.3	19.	80	– ^a
26	2	1.9	0.2	11.	95	– ^a
27	2	1.8	0.1	5.5	90	– ^a
28	2	2.2	0.2	9.1	110	– ^a
29	2	2.2	0.3	14.	110	– ^a
30	2	2.3	0.2	8.7	115	– ^a
31	2	1.4	0.2	14.	70	– ^a
32	2	1.7	0.2	12.	85	– ^a
33	2	1.6	0.4	25.	80	– ^a
34	2	1.1	0.1	9.1	55	– ^a
35	2	0.4	0.2	50.	20	– ^a
36	2	2.1	0.2	9.5	105	– ^a
37	2	1.8	0.2	11.	90	– ^a
38	2	1.8	0.2	11.	90	– ^a
39	2	1.9	0.1	5.3	95	– ^a
40	8	8.2	1.2	15.	102	– ^a
41	2	2.4	0.1	4.2	120	– ^a
42	2	1.9	0.1	5.3	95	– ^a
43	2	2.1	0.2	9.5	105	– ^a
44	2	1.5	0.1	6.7	75	– ^a
45	25	28.	4.7	17.	112	15.
46	2	1.7	0.1	5.9	85	– ^a
Mean ^b	2	1.8	0.2	15.	91	0.6

^aSee Table 4.^bCompounds 4, 40, and 45 excluded from the means.

Table 4. Accuracy and Precision Data from Five to Seven Determinations of the Method Analytes at 0.2 µg/L with Liquid-Solid Extraction and the Ion Trap Mass Spectrometer

Compound Number (Table 2)	True Conc. (µg/L)	Mean Observed Conc. (µg/L)	Std. Dev (µg/L)	Rel Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) (µg/L)
4	0.5	0.45	0.6	13.	90	0.1
5	0.2	0.13	0.03	23.	65	0.1
6	0.2	0.13	0.03	23.	65	0.1
7	0.2	0.13	0.01	7.7	65	0.04
8	0.2	0.24	0.03	13.	120	0.1
9	0.2	0.14	0.01	7.1	70	0.04
10	0.2	not separated from No. 11; measured with No. 11				
11	0.2	0.25	0.04	16.	62	0.2
12	0.2	0.03	0.01	33.	15	0.04
13	0.2	0.03	0.02	67.	15	0.1
14	0.2	0.32	0.07	22.	160	0.3
15	0.2	0.17	0.04	24.	85	0.2
16	0.2	0.19	0.03	16.	95	0.1
17	0.2	0.17	0.08	47.	85	0.3
18	0.2	0.19	0.03	16.	95	0.1
19	0.2	0.21	0.01	4.8	105	0.04
20	0.2	0.03	0.02	67.	150	0.1
21	0.2	0.48	0.09	19.	240	0.3
22	0.2	0.20	0.03	15.	100	0.1
23	0.2	0.45	0.21	47.	225	0.8
24	0.2	0.39	0.16	41.	195	0.6
25	0.2	0.31	0.16	52.	155	0.6
26	0.2	0.21	0.01	4.8	105	0.04
27	0.2	0.12	0.12	100.	60	0.5
28	0.2	0.21	0.05	24.	105	0.2
29	0.2	0.22	0.01	4.5	110	0.04
30	0.2	0.19	0.04	21.	95	0.2
31	0.2	0.19	0.03	16.	95	0.1
32	0.2	0.16	0.04	25.	80	0.1
33	0.2	0.19	0.03	16.	95	0.1
34	0.2	0.04	0.01	25.	20	0.03
35	0.2	0.04	0.03	75.	20	0.1
36	0.2	0.22	0.02	9.1	110	0.1
37	0.2	0.11	0.01	9.1	55	0.04
38	0.2	0.19	0.05	26.	95	0.2
39	0.2	0.13	0.02	15.	65	0.1
40	0.2	0.78	0.08	10.	97	0.3
41	0.2	0.20	0.004	2.0	100	0.01
42	0.2	0.18	0.005	2.8	90	0.02
43	0.2	0.25	0.04	16.	125	0.02
44	0.2	0.14	0.04	29.	70	0.1
45		not measured at this level				
46	0.2	0.13	0.02	15.	65	0.06
Mean ^a	0.2	0.18	0.04	25.	95	0.16

^aCompounds 4, 40, and 45 excluded from the means.

Table 5. Accuracy and Precision data From Five to Seven Determinations of the Method Analytes at 0.2 µg/L with Liquid-Solid Extraction and a Magnetic Sector Mass Spectrometer

Compound Number (Table 2)	True Conc. (µg/L)	Mean Observed Conc. (µ/L)	Std. Dev (µg/L)	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) (µg/L)	
4	5	5.7	0.34	6.0	114	– ^a	
5	2	1.9	0.22	12.	95	– ^a	
6	2	1.6	0.18	11.	80	– ^a	
7	2	2.2	0.67	30.	110	– ^a	
8	2	2.4	0.46	19.	120	– ^a	
9	2	2.2	0.87	40	110	– ^a	
10	2	not separated from No. 11; measured with No. 11					
11	2	4.0	0.37	9.3	100	– ^a	
12	2	0.85	0.15	18.	43	– ^a	
13	2	0.69	0.12	17.	35	– ^a	
14	2	2.0	0.20	10.	100	– ^a	
15	2	2.2	0.41	19.	110	– ^a	
16	2	2.1	0.38	18.	105	– ^a	
17	2	1.9	0.10	5.2	95	– ^a	
18	2	2.0	0.29	14.	100	– ^a	
19	2	2.1	0.32	15.	105	– ^a	
20	2	0.75	0.18	24.	38	– ^a	
21	2	2.5	0.32	13.	125	– ^a	
22	2	2.0	0.23	12.	100	– ^a	
23	2	3.5	1.8	51.	175	– ^a	
24	2	2.0	0.28	14.	100	– ^a	
25	2	1.4	0.16	11.	70	– ^a	
26	2	2.9	0.70	24.	145	– ^a	
27	2	1.7	0.45	26.	85	– ^a	
28	2	2.6	1.0	38.	130	– ^a	
29	2	1.2	0.10	8.3	60	– ^a	
30	2	2.6	0.42	16.	130	– ^a	
31	2	1.5	0.19	13.	75	– ^a	
32	2	1.5	0.35	23.	75	– ^a	
33	2	1.9	0.17	8.9	95	– ^a	
34	2	0.89	0.11	12.	45	– ^a	
35	2	0.83	0.072	8.7	42	– ^a	
36	2	2.2	0.10	4.5	110	– ^a	
37	2	2.0	0.88	44.	100	– ^a	
38	2	1.5	0.11	7.3	75	– ^a	
39	2	1.6	0.14	8.8	80	– ^a	
40	8	12.	2.6	22.	150	– ^a	
41	2	2.3	0.18	7.8	115	– ^a	
42	2	2.0	0.26	13.	100	– ^a	
43	2	2.5	0.34	14.	125	– ^a	
44	2	1.6	0.17	11.	80	– ^a	
45	25	28.	2.7	10.	112	9.	
46	2	1.9	0.073	3.8	95	– ^a	
Mean ^a	2	1.8	0.32	16.	88	1.	

^aSee Table 4.^bCompound 4, 40, and 45 excluded from the means.

Table 6. Accuracy and Precision Data from Six or Seven Determinations of the Method Analytes at 0.2 µg/L with Liquid-Solid Extraction and a Magnetic Sector Spectrometer

Compound Number (Table 2)	True Conc. (µg/L)	Mean Observed Conc. (µ/L)	Std.Dev (µg/L)	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) (µg/L)	
4	0.5	0.67	0.07	9.4	134	0.2	
5	0.2	0.11	0.03	24.	55	0.1	
6	0.2	0.11	0.02	21.	56	0.1	
7	0.2	0.14	0.02	17.	70	0.1	
8	0.2	0.26	0.08	31.	130	0.3	
9	0.2	0.24	0.06	26.	120	0.2	
10	0.2	not separated from No. 11; measured with No. 11					
11	0.2	0.40	0.10	25.	100	0.3	
12	0.2	0.08	0.02	27.	38	0.1	
13	0.2	0.07	0.01	22.	33	0.1	
14	0.2	0.33	0.16	48.	160	0.5	
15	0.2	0.19	0.02	13.	95	0.1	
16	0.2	0.17	0.08	45.	85	0.3	
17	0.2	0.19	0.04	18.	95	0.1	
18	0.2	0.17	0.02	13.	85	0.1	
19	0.2	0.27	0.08	28.	135	0.3	
20	0.2	0.09	0.01	15.	46	0.1	
21	0.2	1.1	1.2	109.	550	4.	
22	0.2	0.18	0.05	30.	90	0.2	
23	0.2	0.29	0.17	59.	145	0.6	
24	0.2	0.42	0.23	55.	210	0.8	
25	0.2	0.32	0.16	50.	160	0.5	
26	0.2	0.20	0.09	47.	100	0.3	
27	0.2	0.53	0.30	57.	265	1.	
28	0.2	0.18	0.03	15.	90	0.1	
29	0.2	0.11	0.05	42.	55	0.2	
30	0.2	0.33	0.08	26.	165	0.3	
31	0.2	0.17	0.01	7.1	85	0.04	
32	0.2	0.11	0.04	40.	55	0.2	
33	0.2	0.17	0.03	15.	85	0.1	
34	0.2	0.05	0.02	35.	24	0.1	
35	0.2	0.08	0.06	8.1	40	0.02	
36	0.2	0.27	0.03	11.	135	0.1	
37	0.2	0.24	0.09	39.	120	0.3	
38	0.2	0.15	0.02	12.	75	0.1	
39	0.2	0.13	0.02	13.	65	0.1	
40	0.8	1.8	0.82	46.	225	3.	
41	0.2	0.21	0.07	33.	105	0.2	
42	0.2	0.19	0.04	23.	95	0.1	
43	0.2	0.27	0.07	27.	135	0.2	
44	0.2	0.13	0.03	22.	65	0.1	
45		not measured at this point					
46	0.2	0.16	0.04	23.	80	0.12	
Mean ^a	0.2	0.21	0.09	28.	102	0.3	

^aCompounds 4, 40, and 45 excluded from the means.

Table 7. Accuracy and Precision Data from Seven Determinations at 2 µg/L with Liquid-Solid Extraction and a Quadrupole Mass Spectrometer

Compound Number (Table 2)	True Conc. (µg/L)	Mean Observed Conc. (µ/L)	Std. Dev (µg/L)	Rel. Std. Dev. (%)	Mean Method Accuracy (% of True Conc.)	Method Detection Limit (MDL) (µg/L)
47	2	2.4	0.4	16.	122	1.0

Table 8. Accuracy and Precision Data from Seven Replicates at 0.2 $\mu\text{g/L}$ with Liquid-solid C-18 Disk Extraction and an ion Trap Mass Spectrometer

Compound Number (Table 2)	Target Concentration ($\mu\text{g/L}$)	Standard Deviation ($\mu\text{g/L}$)	Relative Deviation (%)	Mean ($\mu\text{g/L}$)	Accuracy (% of target)
1	0.2	0.01	5.3	0.22	110
4	5.0	0.37	7.4	5.55	111
6	0.2	0.03	13.2	0.26	130
7	0.2	0.03	13.7	0.22	108
8	0.2	0.04	22.4	0.21	105
9	0.2	0.07	33.2	0.29	147
10	0.2	0.16	77.6	0.40	199
11	0.2	0.03	13.7	0.21	107
12	0.2	0.04	21.7	0.26	128
13	0.2	0.03	14.9	0.23	115
14	0.2	0.07	32.5	0.37	183
15	0.2	0.12	61.1	0.19	95
16	0.2	0.06	31.9	0.19	93
17	0.2	0.18	91.3	0.55	276
18	0.2	0.01	7.2	0.16	78
19	0.2	0.02	10.9	0.27	136
20	0.2	0.05	22.9	0.18	90
21	0.2	0.08	40.3	0.47	233
22	0.2	0.02	9.7	0.17	87
23	0.2	0.02	11.9	0.27	133
24	0.2	0.50	252.0	1.54	771
25	0.2	0.04	20.8	0.36	180
26	0.2	0.02	7.6	0.23	117
27	0.2	0.05	25.4	0.23	117
28	0.2	0.01	7.3	0.20	101
29	0.2	0.05	22.9	0.28	139
30	0.2	0.08	38.9	0.36	181
31	0.2	0.08	38.0	0.28	141
32	0.2	0.04	17.7	0.22	109
33	0.2	0.06	31.9	0.19	96
34	0.2	0.01	5.2	0.34	170
35	0.2	0.05	27.0	0.29	143
36	0.2	0.01	6.5	0.22	110
37	0.2	0.03	13.4	0.20	100
38	0.2	0.04	21.1	0.20	99
39	0.2	0.03	15.1	0.17	84
40	2.0				
41	0.2	0.02	12.2	0.20	102
42	0.2	0.02	10.2	0.24	121
43	0.2	0.04	18.8	0.19	94
44	0.2	0.06	28.1	0.21	107
45	20.0	2.47	12.3	24.80	123
46	0.2	0.04	21.4	0.19	95
47	0.2	0.03	14.7	0.11	55

Table 9. Accuracy and Precision Data from Seven Replicates at 2.0 $\mu\text{g/L}$ with Liquid-Solid C-18 Disk Extraction and an ion Trap Mass Spectrometer

Compound Number (Table 2)	Target Concentration ($\mu\text{g/L}$)	Standard Deviation ($\mu\text{g/L}$)	Relative Deviation (%)	Mean ($\mu\text{g/L}$)	Accuracy (% of target)
1	2	0.18	9.2	2.00	100
4	5	0.45	9.1	5.22	104
6	2	0.30	14.8	2.14	107
7	2	0.17	8.6	2.25	112
8	2	0.47	23.5	2.78	139
9	2	0.21	10.4	2.21	111
10	2	0.62	30.9	2.84	142
11	2	0.57	28.7	2.30	115
12	2	0.31	15.6	2.61	130
13	2	0.28	13.9	2.28	114
14	2	0.33	16.7	2.92	146
15	2	0.62	31.1	1.21	61
16	2	1.02	51.2	1.92	96
17	2	1.39	69.3	3.29	164
18	2	0.22	11.2	2.52	126
19	2	0.23	11.6	1.99	100
20	2	0.27	13.4	2.25	113
21	2	0.23	11.3	2.45	123
22	2	0.38	18.9	2.35	117
23	2	0.22	11.1	2.23	111
24	2	0.38	19.1	3.25	163
25	2	0.26	12.8	2.49	124
26	2	0.69	34.6	1.80	90
27	2	0.12	6.1	1.97	98
28	2	0.19	9.7	2.15	108
29	2	0.30	15.0	2.10	105
30	2	0.15	7.4	2.41	121
31	2	0.64	32.2	2.46	123
32	2	0.85	42.3	1.96	98
33	2	0.52	25.9	2.05	102
34	2	0.22	11.0	1.42	71
35	2	0.37	18.3	2.31	115
36	2	0.42	21.2	2.69	134
37	2	0.34	16.8	2.34	117
38	2	0.77	38.5	0.97	49
39	2	0.29	14.7	2.11	106
40	20	15.16	75.8	19.51	98
41	2	0.20	9.9	2.20	110
42	2	0.17	8.3	2.34	117
43	2	0.27	13.3	2.37	119
44	2	0.15	7.4	2.11	106
45	100	3.36	3.4	98.33	98
46	2	0.58	28.8	1.65	82
47	2	0.07	3.5	1.55	77

Table 10. Minimum Detection Limits From Seven Replicates Using Liquid-Solid Extraction C-18 Disks and an Ion Trap Mass Spectrometer

<i>Chemical Name</i>	<i>Minimum Detection Limits</i>
Acenaphthylene	0.033
Alachlor	0.092
Aldrin	0.083
Anthracene	0.086
Atrazine	0.140
Benz[a]anthracene	0.224
Benzo[b]fluoranthene	0.488
Benzo[k]fluoranthene	0.086
Benzo[a]pyrene	0.137
Benzo[g,h,i]perylene	0.094
Butylbenzyl phthalate	0.204
Chlordane- α	0.384
Chlordane- γ	0.200
Chlordane (trans-Nonachlor)	0.574
Chrysene	0.068
Dibenz[a,h]anthracene	0.144
Di- <i>n</i> -butyl phthalate	0.253
Diethyl phthalate	0.075
Di(2-ethylhexyl) phthalate	1.584
Di(2-ethylhexyl) adipate	0.131
Dimethyl phthalate	0.048
Endrin	0.160
Fluorene	0.046
Heptachlor	0.144
Heptachlorepoxyde	0.244
Hexachlorobenzene	0.111
Hexachlorocyclopentadiene	0.039
Indeno[1,2,3, <i>c,d</i>]pyrene	0.170
Lindane	0.041
Methoxychlor	0.084
PCB-mono-Cl-isomer	0.045
PCB-di-Cl-isomer	0.061
PCB-tri-Cl-isomer	0.135
PCB-tetra-Cl-isomer	0.177
PCB-penta-Cl-isomer	0.200
PCB-hepta-Cl-isomer	0.239
PCB-octa-Cl-isomer	0.133
Pentachlorophenol	47.648
Phenanthrene	0.076
Pyrene	0.064
Simazine	0.118
Toxaphene	7.763

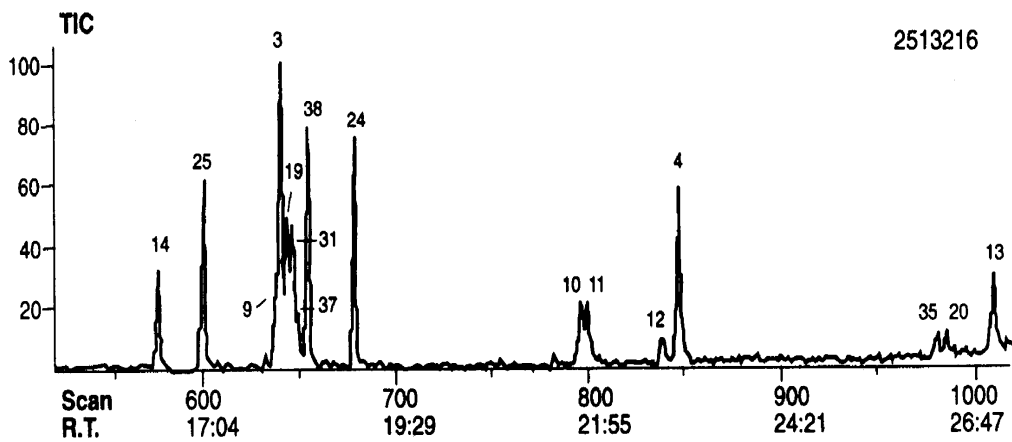
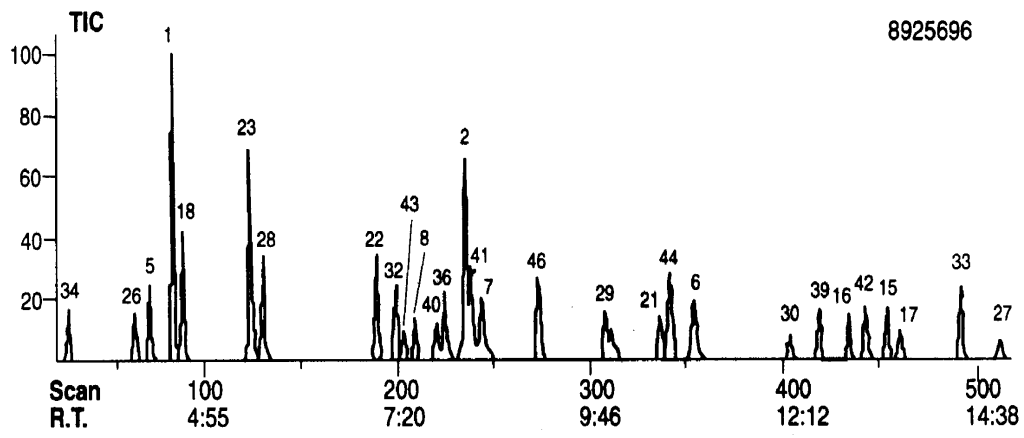
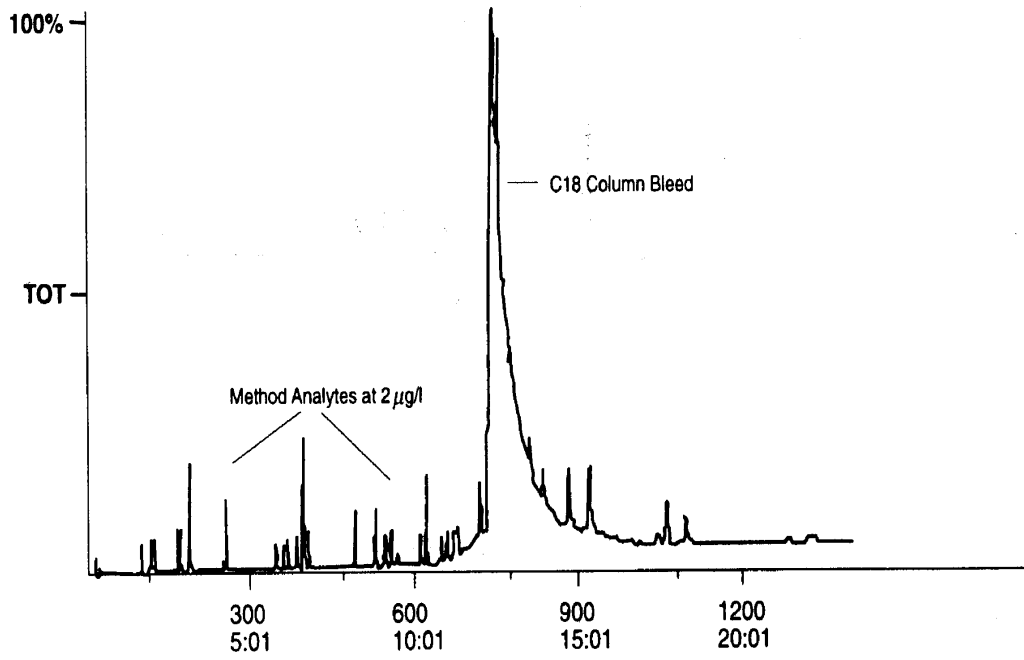


Figure 1. Total Ion Chromatogram of Two Nanograms of Analytes And Five Nanograms of Surrogates and Internal Standard



52-015-13

Figure 2. Total Ion Chromatogram from a Laboratory Blank With an Unacceptably High Background

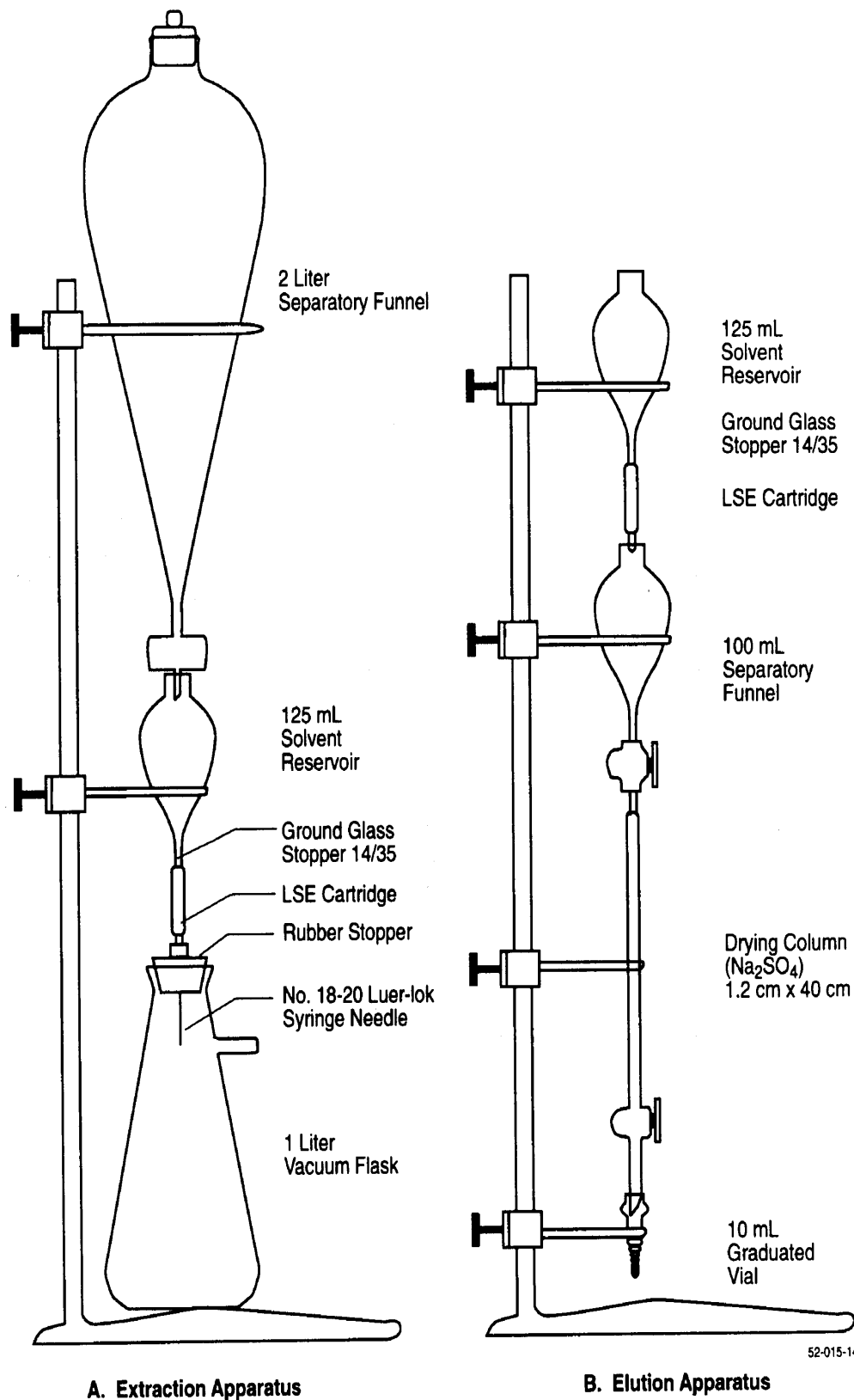
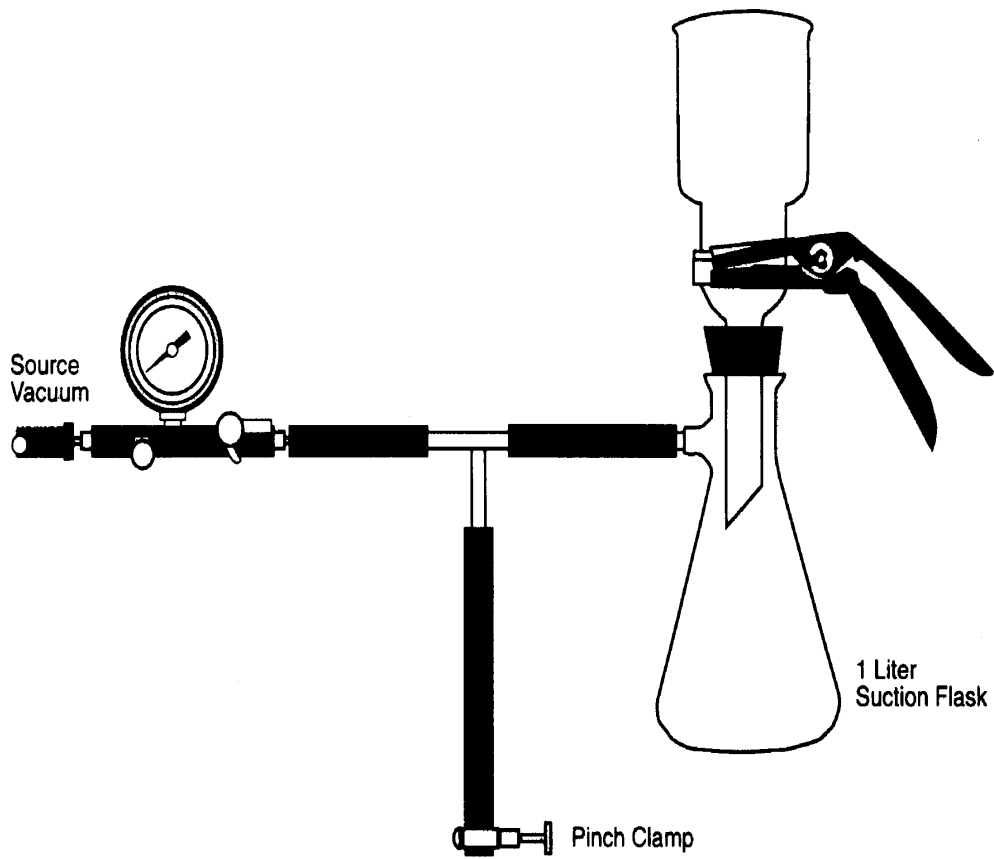


Figure 3.



52-015-15

Figure 4. Disk Extraction Apparatus

Appendix

Detection Limits for Precision and Accuracy for the Analysis of Pesticide Compounds by EPA Method 525.1

Table 7. Method 525.1 Detection Limits—507 & 508 Compounds

Compound	Target ($\mu\text{g/L}$)	# Reps	% Rec	SD	MS MDL	ECD-NPD MDL
Alachlor	0.2	7	73	7	0.044	0.380
Aldrin	0.2	7	33	10	0.060	0.075
Ametryn	0.1	7	68	14	0.043	2.000
Atraton	2.0	7	51	8	0.533	0.600
Atrazine	0.2	7	78	7	0.041	0.130
BHC, α	0.1	7	143	31	0.970	0.025
BHC, β	0.1	7	76	9	0.027	0.010
BHC, δ	0.1	7	78	9	0.029	0.010
BHC, γ	0.2	7	73	12	0.072	0.015
Bromacil	0.1	7	117	13	0.041	2.500
Butachlor	0.1	7	98	8	0.025	0.380
Butylate	0.1	7	78	23	0.071	0.150
Carboxin	0.1	7	65	9	0.027	0.600
Chlordane, α	0.2	7	61	11	0.069	0.002
Chlordane, γ	0.2	7	61	11	0.066	0.002
Chloroneb	0.1	7	62	8	0.026	0.500
Chlorobenzilate	0.1	7	315	25	0.077	5.000
Chlorothalonil	0.1	7	77	9	0.028	0.025
Chlorpropham	0.1	7	84	9	0.028	0.500
Cycloate	0.1	7	67	14	0.043	0.025
DCPA	0.1	7	78	23	0.071	0.025
DDD, 4,4'-	0.1	7	73	14	0.045	0.003
DDE, 4,4'-	0.1	7	143	16	0.051	0.010
DDT, 4,4'-	0.1	7	74	17	0.052	0.060
Diazinon	0.1	7	98	14	0.043	0.250
Dichlorvos	0.1	7	75	28	0.089	2.500
Dieldrin	2.0	7	86	4	0.265	0.020
Diphenamid	0.1	7	87	7	0.022	0.600
Disulfoton	0.1	7	75	16	0.050	0.300
Disulfoton sulfone	0.1	7	86	8	0.025	3.800
Disulfoton sulfoxide	2.0	7	131	11	0.723	0.380
Endosulfan I	0.1	7	72	10	0.031	0.015
Endosulfan II	0.1	7	101	30	0.096	0.024
Endosulfan sulfate	0.1	7	79	13	0.042	0.015
Endrin	0.2	7	85	16	0.102	0.015
Endrin aldehyde	0.1	7	92	11	0.036	0.025
EPTC	0.1	7	56	20	0.061	0.250
Ethoprop	0.1	7	152	2	0.007	0.250
Etridiazole	0.1	7	61	24	0.076	0.190
Fenamiphos	0.1	7	140	12	0.037	1.000
Fenarimol	0.1	7	136	9	0.028	0.380
Fluridone	2.0	7	105	19	1.166	3.800
Heptachlor	0.2	7	54	12	0.074	0.010

Table 7. Method 525.1 Detection Limits—507 & 508 Compounds

Compound	Target ($\mu\text{g/L}$)	# Reps	% Rec	SD	MS MDL	ECD-NPD MDL
Heptachlor epoxide	0.2	7	71	10	0.061	0.015
Hexachlorobenzene	0.2	7	80	16	0.102	0.008
Hexazinone	0.1	7	124	12	0.038	0.760
Merphos	0.1	7	119	15	0.049	0.250
Methoxychlor	0.2	7	15	3	0.020	0.050
Methyl paraoxon	0.1	7	122	11	0.036	2.500
Metolachlor	0.1	7	81	5	0.015	0.750
Metribuzin	0.1	7	49	14	0.045	0.150
Mevinphos	0.1	7	98	14	0.044	5.000
MKG-264	0.1	7	64	4	0.013	0.500
Molinate	0.1	7	64	13	0.041	0.150
Napropamide	0.1	7	89	7	0.021	0.250
Norflurazon	0.1	7	108	7	0.023	0.500
Pebulate	0.1	7	72	19	0.061	0.130
Permethrin, cis-	0.05	7	76	20	0.031	0.500
Permethrin, trans-	0.15	7	80	20	0.095	0.500
Prometon	0.1	7	78	58	0.181	0.300
Prometryn	0.1	7	98	16	0.050	0.190
Pronamide	0.1	7	73	7	0.021	0.760
Propachlor	0.2	7	92	11	0.071	0.500
Propazine	0.1	7	77	5	0.015	0.130
Simazine	0.1	7	81	8	0.026	0.075
Simetryn	0.1	7	82	34	0.108	0.250
Stirofos	0.1	7	101	8	0.025	0.760
Tebuthiuron	0.1	7	114	14	0.044	1.300
Terbacil	0.1	7	104	6	0.019	4.500
Terbufos	0.1	7	162	19	0.060	0.500
Terbutryn	0.1	7	83	12	0.038	0.250
Triademefon	0.1	7	208	84	0.264	0.650
Tricyclazole	2.0	7	150	77	4.861	1.000
Trifluralin	0.2	7	68	13	0.082	0.025
Vernolate	0.1	7	54	14	0.045	0.130
		Avg	91	15	0.16	0.67

Table 9. Method 525.1—Laboratory Fortified Blank Data, 1 L

<i>Compound</i>	<i>Target (µg/L)</i>	<i># Reps</i>	<i>Mean Obs.</i>	<i>SD</i>	<i>% RSD</i>	<i>Accuracy</i>
Alachlor	1.0	13	1.039	0.15	13	104
Aldrin	1.0	13	1.114	0.58	15	111
Ametryn	1.0	13	0.850	0.09	13	85
Atraton	1.0	13	0.313	0.10	13	31
Atrazine	1.0	13	0.932	0.13	14	93
BHC, α	1.0	13	1.070	0.64	12	107
BHC, β	1.0	13	1.075	0.61	13	108
BHC, δ	1.0	13	1.136	0.66	12	114
BHC, γ	1.0	13	1.105	0.67	12	110
Bromacil	1.0	13	0.879	0.21	13	88
Butachlor	1.0	13	1.126	0.27	9	113
Butylate	1.0	13	0.843	0.23	18	84
Carboxin	1.0	13	0.757	0.07	16	76
Chlordane, α	1.0	13	1.017	0.69	9	102
Chlordane, γ	1.0	13	1.144	0.61	9	114
Chlorneb	1.0	13	1.018	0.60	10	102
Chlorobenzilate	1.0	13	1.211	0.65	11	121
Chlorothalonil	1.0	13	1.211	0.65	13	121
Chlorpropham	1.0	13	1.121	0.67	15	112
Cycloate	1.0	13	0.639	0.24	10	64
DCPA	1.0	13	0.822	0.16	11	82
DDD, 4,4'-	1.0	13	1.084	0.63	9	108
DDE, 4,4'-	1.0	13	1.221	0.60	13	122
DDT, 4,4'-	1.0	13	1.128	0.66	13	113
Diazinon	1.0	13	1.335	0.84	13	134
Dichlorvos	1.0	13	0.749	0.09	28	75
Dieldrin	1.0	13	0.819	0.12	11	82
Diphenamid	1.0	13	1.383	0.79	12	138
Disulfoton	1.0	13	0.978	0.12	16	98
Disulfoton sulfone	1.0	9	0.880	0.11	17	88
Disulfoton sulfoxide	1.0	13	0.490	0.47	19	49
Endosulfan I	1.0	13	1.229	1.47	24	123
Endosulfan II	1.0	13	1.358	0.67	12	136
Endosulfan sulfate	1.0	13	1.194	0.56	15	119
Endrin	1.0	13	1.223	0.66	11	122
Endrin aldehyde	1.0	13	1.331	0.77	10	133
EPTC	1.0	13	1.159	0.70	14	116
Ethoprop	1.0	13	1.134	0.19	29	113
Etridiazole	1.0	13	0.911	0.11	12	91
Fenamiphos	1.0	13	1.088	0.58	17	109
Fenarimol	1.0	13	1.074	0.47	11	107
Fluridone	1.0	13	1.108	0.20	14	111
Heptachlor	1.0	13	1.102	0.22	14	110
Heptachlor epoxide	1.0	13	1.218	0.63	12	122
Hexachlorobenzene	1.0	13	1.024	0.65	9	102
Hexazinone	1.0	13	0.988	0.60	24	99
Merphos	1.0	13	0.955	0.28	10	95
Methoxychlor	1.0	13	1.182	0.31	20	118

Table 9. Method 525.1—Laboratory Fortified Blank Data, 1 L

<i>Compound</i>	<i>Target (µg/L)</i>	<i># Reps</i>	<i>Mean Obs.</i>	<i>SD</i>	<i>% RSD</i>	<i>Accuracy</i>
Methyl paraoxon	1.0	13	2.334	0.83	34	233
Metolachlor	1.0	13	0.888	0.18	14	89
Metribuzin	1.0	13	1.043	0.15	13	104
Mevinphos	1.0	13	0.727	0.13	18	73
MKG-264	1.0	13	0.822	0.13	21	82
Molinate	1.0	13	0.751	0.19	12	75
Napropamide	1.0	13	0.827	0.18	12	83
Norflurazon	1.0	13	0.972	0.12	12	97
Pebulate	1.0	13	1.043	0.15	11	104
Permethrin, cis-	1.0	13	1.123	0.24	16	112
Permethrin, trans-	1.0	13	0.860	0.96	18	86
Prometon	1.0	13	1.124	0.22	11	112
Prometryn	1.0	13	0.562	0.11	8	56
Pronamide	1.0	13	1.118	0.13	18	112
Propachlor	1.0	13	0.932	0.13	14	93
Propazine	1.0	13	2.179	0.90	14	218
Simazine	1.0	13	0.853	0.20	14	85
Simetryn	1.0	13	0.708	0.06	9	71
Stirofos	1.0	13	1.043	0.21	13	104
Tebuthiuron	1.0	13	1.036	0.23	30	104
Terbacil	1.0	13	1.119	0.18	17	112
Terbufos	1.0	13	0.913	0.13	13	91
Terbutryn	1.0	13	0.908	0.10	13	91
Triademefon	1.0	13	1.172	0.19	15	117
Tricyclazole	1.0	13	0.931	0.42	54	93
Trifluralin	1.0	13	2.215	0.78	15	221
Vernolate	1.0	13	0.901	0.18	16	90
		Avg	1.05	0.40	15	105