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METHOD 525.3 DETERMINATION OF SEMIVOLATILE ORGANIC CHEMICALS IN DRINKING WATER BY SOLID PHASE EXTRACTION AND CAPILLARY COLUMN GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

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METHOD 525.3

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1. SCOPE AND APPLICATION

1.1. This is a gas chromatography/mass spectrometry (GC/MS) method for the determination of selected semivolatile organic compounds in drinking waters. Accuracy and precision data have been generated in reagent water, and in finished ground and surface waters for the compounds listed in the table below. This method was initially developed with full scan GC/MS, but performance has also been demonstrated in the selected ion monitoring (SIM) mode using a subset of analytes. SIM is useful when enhanced sensitivity is desirable. An example chromatogram which includes the entire analyte list is shown in Figs. 1a-1d.

Analyte	**Chemical Abstract Services Registry Number (CASRN)**
Acenaphthylene	208-96-8
Acetochlor	34256-82-1
Alachlor	15972-60-8
Aldrin	309-00-2
Ametryn	834-12-8
Anthracene	120-12-7
Atraton	1610-17-9
Atrazine	1912-24-9
Benzo[*a*]anthracene	56-55-3
Benzo[*b*]fluoranthene	205-99-2
Benzo[*k*]fluoranthene	207-08-9
Benzo[*a*]pyrene	50-32-8
Benzo[*g,h,i*]perylene	191-24-2
Bromacil	314-40-9
Butachlor	23184-66-9
Butylate	2008-41-5
Butylated hydroxytoluene (BHT)	128-37-0
Butylbenzylphthalate	85-68-7
Chlordane (tech grade)	12789-03-6
cis-chlordane	5103-71-9
trans-chlordane	5103-74-2
trans-nonachlor	39765-80-5
Chlorfenvinphos	470-90-6
Chloroneb	2675-77-6
Chlorobenzilate	510-15-6

Analyte	Chemical Abstract Services Registry Number (CASRN)
Chloropropham	101-21-3
Chlorothalonil	1897-45-6
Chlorpyrifos	2921-88-2
Chrysene	218-01-9
Cyanazine	21725-46-2
Cycloate	1134-23-2
Dacthal (DCPA)	1861-32-1
DDD, 4,4′-	72-54-8
DDE, 4,4′-	72-55-9
DDT, 4,4′-	50-29-3
DEET (N,N-Diethyl-meta-toluamide)	134-62-3
Dibenz[*a,h*]anthracene	53-70-3
Di-n-butylpthalate	84-74-2
Dichlorvos	62-73-7
Dieldrin	60-57-1
Diethylphthalate	84-66-2
Di(2-ethylhexyl)adipate	103-23-1
Di(2-ethylhexyl)phthalate	117-81-7
Diisopropyl methylphosphonate (DIMP)	1445-75-6
Dimethipin	55290-64-7
Dimethylphthalate	131-11-3
Dinitrotoluene, 2,4-	121-14-2
Dinitrotoluene, 2,6-	606-20-2
Diphenamid	957-51-7
Disulfoton	298-04-4
Endosufan I	959-98-8
Endosufan II	33213-65-9
Endosufan sulfate	1031-07-8
Endrin	72-20-8
EPTC (S-Ethyl dipropylthiocarbamate)	759-94-4
Ethion	563-12-2
Ethoprop	13194-48-4
Ethyl parathion	56-38-2
Etridiazole	2593-15-9
Fenarimol	60168-88-9
Fluorene	86-73-7
Fluridone	59756-60-4
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene (HCB)	118-74-1
Hexachlorocyclohexane, alpha (α-HCH)	319-84-6
Hexachlorocyclohexane, beta (β-HCH)	319-85-7

Analyte	Chemical Abstract Services Registry Number (CASRN)
Hexachlorocyclohexane, delta (δ-HCH)	319-86-8
Hexachlorocyclohexane, gamma (γ-HCH) (Lindane)	58-89-9
Hexachlorocyclopentadiene (HCCPD)	77-47-4
Hexazinone	51235-04-2
Indeno[1,2,3-*c,d*]pyrene	193-39-5
Isophorone	78-59-1
Methoxychlor	72-43-5
Methyl parathion	298-00-0
Metolachlor	51218-45-2
Metribuzin	21087-64-9
Mevinphos	7786-34-7
MGK 264	113-48-4
Molinate	2212-67-1
Napropamide	15299-99-7
Nitrofen	1836-25-5
Norflurazon	27314-13-2
Oxyfluorfen	42874-03-3
Pebulate	1114-71-2
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Permethrin, cis-	54774-45-7
Permethrin, trans-	51877-74-8
Prometon	1610-18-0
Phorate	298-02-2
Phosphamidon	13171-21-6
Profenofos	41198-08-7
Prometryn	7287-19-6
Pronamide	23950-58-5
Propachlor	1918-16-7
Propazine	139-40-2
Pyrene	129-00-0
Simazine	122-34-9
Simetryn	1014-70-6
Tebuconazole	107534-96-3
Tebuthiuron	34014-18-1
Terbacil	5902-51-2
Terbutryn	886-50-0
Tetrachlorvinphos	22248-79-9
Tribufos	78-48-8
Toxaphene	8001-35-2
Triadimefon	43121-43-3
Trifluralin	1582-09-8

Analyte	Chemical Abstract Services Registry Number (CASRN)
Vernolate	1929-77-7
Vinclozolin	50471-44-8
Polychlorinated Biphenyl (PCB) Congeners (IUPAC #)	
2-chlorobiphenyl (1)	2051-60-7
4-chlorobiphenyl (3)	2051-62-9
2,4′-dichlorobiphenyl (8)	34883-43-7
2,2′,5-trichlorobiphenyl (18)	37680-65-2
2,4,4′-trichlorobiphenyl (28)	7012-37-5
2,2′,3,5′-tetrachlorobiphenyl (44)	41464-39-5
2,2′,5,5′-tetrachlorobiphenyl (52)	35693-99-3
2,3′,4′,5-tetrachloroobiphenyl (70)	32598-11-1
2,3,3′,4′,6-pentachlorobiphenyl (110)	38380-03-9
2,3′,4,4′,5-pentachlorobiphenyl (118)	31508-00-6
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	35065-28-2
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	38380-04-0
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	35065-27-1
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	35065-29-3

1.2. The Minimum Reporting Level (MRL) is the lowest analyte concentration that meets Data Quality Objectives (DQOs) that are developed based on the intended use of this method. The single laboratory lowest concentration MRL (LCMRL) (Sect. 3.12) is the lowest true concentration for which the future recovery is predicted to fall, with high confidence (99 percent), between 50 and 150 percent recovery. The LCMRL is compound dependent and is also dependent on extraction efficiency, sample matrix, fortification concentration, and instrument performance. The procedure used to determine the LCMRL is described elsewhere.[1,2] During method development, LCMRLs were determined by two laboratories from the results of laboratory fortified blanks (LFBs) in full scan mode for all method analytes. LCMRLs were also determined for selected analytes in the SIM mode. These LCMRLs are provided in Tables 15 and 23 for full scan and SIM analyses, respectively.

1.3 Laboratories using this method are not required to determine the LCMRL for this method, but will need to demonstrate that their laboratory MRL for this method meets the requirements described in Sect. 9.2.4.

1.4 Determining the Detection Limit (DL) for analytes in this method is optional (Sect. 9.2.6). Detection limit is defined as the statistically calculated minimum concentration that can be measured with 99% confidence that the reported value is greater than zero.[3] The DL is compound dependent and is also dependent on extraction efficiency, sample matrix, fortification concentration, and instrument performance. DLs have been determined for all analytes in two laboratories in full

scan mode (Table 15) and by a single laboratory for selected analytes in SIM mode (Table 23).

1.5 This method is intended for use by analysts skilled in solid phase extractions, the operation of GC/MS instruments, and the interpretation of the associated data.

1.6 Analysts and laboratory managers are advised that analyzing for all of the method analytes on a continuing basis while maintaining quality control criteria may be difficult. It is highly recommended that analysts and laboratory managers consider analyzing a subset of the analyte list that will meet their data collection needs.

1.7 Multi-Component Analytes

1.7.1 Toxaphene is a pesticide manufactured by the chlorination of camphene. Over 1000 individual congeners, mainly chlorinated bornanes, have been identified in the technical mixture, although over 30,000 are theoretically possible. The most abundant of the individual components of the technical mixture are typically less than 1% by weight of the total mass. Using the instrument parameters in Method 525.3, technical toxaphene elutes as a poorly defined group of peaks between 21 and 26 min (Fig. 2). Toxaphene's complex composition introduces the potential for interferences that could negatively affect the method performance for other analytes. Therefore, all calibration standards and quality control procedures for toxaphene must be prepared and analyzed in separate samples from all the other method analytes. Toxaphene may be analyzed using this method in either full scan or SIM mode. However, the authors have demonstrated the method in SIM mode only. At the time of method development, the enhanced sensitivity provided by SIM was necessary to meet the drinking water monitoring trigger of 0.001 mg/L [40CFR 141.24 (h)(18)] and maximum contaminant level (MCL) of 0.003 mg/L [40CFR 141.61 (c)]. Detailed instructions for the identification and quantitation of toxaphene are in Sect. 12.3.3.

1.7.2 Aroclors - The PCB congeners selected as analytes in this method are representative of the major components (by weight percent) of Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260.[4,5] Analyzing for these specific congeners allows the analyst to screen samples for possible Aroclor contamination without complex pattern recognition protocols. This method is designed for Aroclor screening only and not for qualitative or quantitative analyses of specific Aroclors.

1.7.3 Technical chlordane - Technical chlordane is a multi-component analyte regulated under the Safe Drinking Water Act (SDWA) at the time of publication of this method. It contains at least 140 chlorinated components. At the concentrations likely to be encountered in drinking water samples, only two or three major components will typically be identified: cis- and

trans-chlordane and trans-nonachlor. These major components are listed as method analytes along with technical chlordane. Specific instructions for quantification of technical chlordane are provided in Sect. 12.3.1.

1.8 METHOD FLEXIBILITY – In recognition of technological advances in analytical systems and techniques, the laboratory is permitted to modify the GC inlet, inlet conditions, column, injection parameters, and all other GC and MS conditions. Changes may not be made to sample collection and preservation (Sect. 8), sample extraction (Sect. 11) or to the Quality Control (QC) requirements (Sect. 9). Method modifications should be considered only to improve method performance. Modifications that are introduced in the interest of reducing cost or sample processing time, but result in poorer method performance, should not be used. In all cases where method modifications are proposed, the analyst must perform the procedures outlined in the initial demonstration of capability (IDC, Sect. 9.2), verify that all QC acceptance criteria in this method (Sect. 9) are met, and that method performance in real sample matrices is equivalent to that demonstrated for Laboratory Fortified Sample Matrices (LFSMs) in Sect. 17.

Note: The above method flexibility section is intended as an abbreviated summation of method flexibility. Sects. 4-12 provide detailed information of specific portions of the method that may be modified. If there is any perceived conflict between the general method flexibility statement in Sect. 1.8 and specific information in Sects. 4-12, Sects. 4-12 supersede Sect.1.8.

2. SUMMARY OF METHOD

2.1. A 1-liter water sample is fortified with surrogate analytes and passed through a solid phase extraction (SPE) device (Sects. 6.9-6.13) to extract the target analytes and surrogates. The compounds are eluted from the solid phase with a small amount of two or more organic solvents. The solvent extract is dried by passing it through a column of anhydrous sodium sulfate, concentrated by evaporation with nitrogen gas, and then adjusted to a 1-mL volume with ethyl acetate after adding the internal standards. A splitless injection is made into a GC equipped with a high-resolution fused silica capillary column that is interfaced to an MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical GC/MS conditions. The GC/MS may be operated in the full scan, SIM, or selected ion storage (SIS) mode (Sects 3.19 and 3.20). The GC/MS may be calibrated using standards prepared in solvent or using matrix-matched standards (Sects. 3.15 and 7.2.4). The concentration of each analyte is calculated by using its integrated peak area and the internal standard technique. Surrogate analytes are added to all Field and Quality Control (QC) Samples to monitor the performance of each extraction and overall method performance.

2.2 Some analytes in this method can be affected by matrix induced chromatographic response enhancement (Sect. 3.14), when analyzed at low concentrations. Refer to

the "comments" section of Table 1 for potentially affected analytes, and to Sect. 13 for information regarding method performance.

3. DEFINITIONS

3.1. ANALYSIS BATCH - A set of samples that is analyzed on the same instrument during a 24-hour period that begins and ends with the analysis of the appropriate Continuing Calibration Check (CCC) Standards. Additional CCCs may be required depending on the length of the analysis batch and/or the number of Field Samples.

3.2. CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solution(s) and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration. In this method, traditional CAL standards prepared in ethyl acetate may be used or matrix-matched standards (Sect. 3.15) prepared in a concentrated laboratory reagent water (LRW) extract may be used. This procedure is described in Sect. 7.2.4.2.

3.3. CONTINUING CALIBRATION CHECK (CCC) STANDARD - A calibration standard containing one or more method analytes, which is analyzed periodically to verify the accuracy of the existing calibration for those analytes.

3.4. DETECTION LIMIT (DL) - The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. This is a statistical determination (Sect. 9.2.6), and accurate quantitation is not expected at this level.[3]

3.5. EXTRACTION BATCH - A set of up to 20 Field Samples (not including QC samples) extracted together by the same person(s) during a work day using the same lot of solid phase extraction devices, solvents, surrogate solution, and fortifying solutions. Required QC samples include Laboratory Reagent Blank, Laboratory Fortified Blank, Laboratory Fortified Sample Matrix, and either a Field Duplicate or Laboratory Fortified Sample Matrix Duplicate.

3.6. 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 provide an estimate of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

3.7. INTERNAL STANDARD (IS) - A pure compound added to an extract or standard solution in a known amount and used to measure the relative responses of the method analytes and surrogates. In this method, the internal standards are isotopically labeled analogues of selected method analytes.

3.8. LABORATORY FORTIFIED BLANK (LFB) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes and all the preservation compounds are added. The LFB is processed and 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.

3.9. LABORATORY FORTIFIED SAMPLE MATRIX (LFSM) - An aliquot of a Field Sample to which known quantities of the method analytes and all the preservation compounds are added. The LFSM is processed and 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 LFSM corrected for background concentrations.

3.10. LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (LFSMD) - A duplicate Field Sample used to prepare the LFSM, which is fortified, extracted and analyzed identically to the LFSM. The LFSMD is used instead of the Field Duplicate to assess method precision and accuracy when the occurrence of a method analyte is infrequent.

3.11. LABORATORY REAGENT BLANK (LRB) - An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, sample preservatives, internal standards, and surrogates that are used in the extraction batch. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the extraction apparatus.

3.12. LOWEST CONCENTRATION MINIMUM REPORTING LEVEL (LCMRL) - The single-laboratory LCMRL is the lowest true concentration for which the future recovery is predicted to fall, with high confidence (99 percent), between 50 and 150 percent recovery.[1,2]

3.13. MATERIAL SAFETY DATA SHEET (MSDS) - Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.14. MATRIX-INDUCED CHROMATOGRAPHIC RESPONSE ENHANCEMENT - This phenomenon occurs when, in the absence of matrix components, method analytes in calibration solutions are degraded or absorbed in the GC injector or column, resulting in poor peak shapes and low response. When subsequent sample extracts containing the analytes and components from a complex sample matrix are injected, peak shape and response improve. In this situation, quantitative data for field samples may exhibit a high bias.[6-13] Generally, overestimation of results is more pronounced at low analyte concentrations.

3.15. MATRIX-MATCHED CALIBRATION STANDARD - A calibration standard that is prepared by adding method analytes to a concentrated extract of a matrix (reagent water is used for this method) that has been prepared following all the extraction and sample preparation steps of the analytical method. The material extracted from the matrix reduces matrix-induced response enhancement effects and improves the quantitative accuracy of sample results.[12,13]

3.16. MINIMUM REPORTING LEVEL (MRL) - The minimum concentration that can be reported by a laboratory as a quantitated value for a method analyte in a sample following analysis. This concentration must meet the criteria defined in Sect. 9.2.4 and must not be any lower than the concentration of the lowest continuing calibration check standard for that analyte. The MRL may be determined by the laboratory based upon project objectives, or may be set by a regulatory body as part of a compliance monitoring program.

3.17. PRIMARY DILUTION STANDARD SOLUTION (PDS) - A solution containing method analytes, internal standards, or surrogate analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other analyte solutions.

3.18. QUALITY CONTROL SAMPLE (QCS) - A solution prepared using a PDS of method analytes obtained from a source external to the laboratory and different from the source of calibration standards. The second source PDS and the surrogate PDS are used to fortify the QCS at a known concentration. The QCS is used to verify the accuracy of the primary calibration standards.

3.19. SELECTED ION MONITORING (SIM) - An MS technique where only one or a few ions are monitored for each target analyte. When used with gas chromatography, the set of ions monitored is usually changed periodically throughout the chromatographic run, to correlate with the characteristic ions of the analytes, SURs and ISs as they elute from the chromatographic column. The technique is often used to increase sensitivity. Throughout this document, the term "SIM" will be used to include both SIM as described here and SIS as described in Sect. 3.20.

3.20. SELECTED ION STORAGE (SIS) - An MS technique typically used with ion trap mass spectrometers where only one or a few ions are stored at any given time point. When used with gas chromatography, the set of ions stored is usually changed periodically throughout the chromatographic run, to correlate with the characteristic ions of the analytes, SURs and ISs as they elute from the chromatographic column. SIS can be used to enhance sensitivity. Throughout this document the term "SIM" will be used to include both SIM (Sect. 3.19) and SIS.

3.21. STOCK STANDARD SOLUTION (SSS) - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.22. SURROGATE ANALYTE (SUR) - A pure analyte, which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in a known amount before extraction or other processing, and is measured with the same procedures used to measure other sample components. The purpose of the SUR is to monitor method performance with each sample.

4. INTERFERENCES

4.1. All glassware must be meticulously cleaned. Wash glassware with detergent and tap water, rinse with tap water, followed by reagent water. Rinse with methanol and/or acetone. Non-volumetric glassware may be heated in a muffle furnace at 400 °C for two hours as a substitute for solvent rinsing. Volumetric glassware should not be heated in an oven above 120 °C.

4.2. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All items such as these must be routinely demonstrated to be free from interferences (less than $^1/_3$ the MRL for each target analyte) under the conditions of the analysis by analyzing laboratory reagent blanks as described in Sect. 9.3.1. **Subtracting blank values from sample results is not permitted.**

4.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Water samples high in total organic carbon (TOC) may have elevated baselines or interfering peaks. Matrix components may directly interfere by producing a signal at or near the retention time of an analyte peak. They can also enhance the signal of method analytes (Sect. 3.14). Analyses of LFSMs are useful in identifying matrix interferences.

4.4. Relatively large quantities of the buffer and preservatives (Sect. 8.1.2) are added to sample bottles. The potential exists for trace-level organic contaminants in these reagents. Interferences from these sources should be monitored by analysis of laboratory reagent blanks, particularly when new lots of reagents are acquired.

4.5. Solid phase extraction media have been observed to be a source of interferences.[14] The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of solid phase extraction devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

4.6. Analyte carryover may occur when a relatively “clean” sample is analyzed immediately after a sample (or standard) that contains relatively high concentrations of compounds. Syringes and GC injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample (or standard) that contains high concentrations of compounds, a laboratory reagent blank should be analyzed to

ensure that accurate values are obtained for the next sample. The analyst should be especially careful to check for carryover of polycyclic aromatic hydrocarbons (PAHs) and PCBs.

4.7. Silicone compounds may be leached from punctured autosampler vial septa, particularly when particles of the septa sit in the vial. This can occur after repeated injections from the same autosampler vial. These silicone compounds, which appear as regularly spaced chromatographic peaks with similar MS fragmentation patterns, can unnecessarily complicate the total ion chromatograms and may cause interferences at high levels.

4.8. Quantitation of bromacil should be reviewed for potential common interferences. The quantitation ion (QI) *m/z* 205 suggested in Table 1 can be found in SPE media; therefore, method blanks should be carefully examined for this potential interference. The ion at *m/z* 207 may be used as an alternate QI; however, this ion is associated with column bleed. Laboratories should select the QI depending on the sorbent being used and the amount of bleed associated with the GC column. If both types of interferences are present, some laboratories may not be able to analyze for bromacil at low concentrations. Manual inspection of all bromacil data reported in field samples is mandatory.

4.9. There are many potential sources of phthalate contamination in the laboratory, especially from plastics and chemicals that may have been stored in plastic containers. If phthalates are to be reported as method analytes, care must be taken to minimize sources of contamination, and the QC criteria for LRBs must be met (Sect. 9.3.1). Special precautions must also be taken when creating calibration curves for analytes consistently found in LRBs (Sect. 10.2.5).

4.10. In cases where the SPE disks or cartridges are dried by pulling room air through the media using vacuum, it may be possible for the media to become contaminated by components in room air. This was not observed during method development. If laboratories encounter contamination problems associated with room air, compressed gas cylinders of high purity nitrogen may be used for drying SPE media during sample processing.

4.11. If samples are to be analyzed for DEET, sample collection personnel should be cautioned against the use of insect repellents containing DEET.

5. SAFETY

5.1. The toxicity or carcinogenicity of each reagent 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 an awareness of OSHA regulations regarding safe handling of chemicals used in this method. A reference file of MSDSs should be made available to all

personnel involved in the chemical analysis. Additional references to laboratory safety are available.[15-17]

5.2. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. EQUIPMENT AND SUPPLIES

References to specific brands or catalog numbers are included for illustration only, and do not imply endorsement of the product. Other brands of equivalent quality may be used. The SPE sorbents described in Sects. 6.9-6.11 are proprietary products that have been fully evaluated for use in this method by two laboratories. The sorbents described in Sects. 6.12 and 6.13 are proprietary products with demonstration data provided by the vendor. Due to their proprietary status, some aspects of the chemistry of all of these sorbents are unknown making equivalency difficult to determine. The EPA document "Technical Notes on Drinking Water Methods"[18] provides criteria for judging equivalency of SPE products. Before analyses are performed for compliance under the Safe Drinking Water Act, questions regarding equivalency of alternate sorbent materials must be addressed to the Office of Ground Water and Drinking Water Alternate Test Procedure Coordinator.[19]

6.1. SAMPLE CONTAINERS - 1-L or 1-qt amber glass bottles fitted with polytetrafluoroethylene (PTFE)-lined screw caps are preferred. Clear glass bottles with PTFE-lined screw caps may be substituted if sample bottles are wrapped with foil, stored in boxes, or otherwise protected from light during sample shipping and storage.

6.2. VIALS - Various sizes of amber glass vials with PTFE-lined screw caps for storing standard solutions and extracts. Amber glass 2-mL autosampler vials with PTFE-faced septa.

6.3. VOLUMETRIC FLASKS - Class A, suggested sizes include 1, 5, and 10 mL for preparation of standards and dilution of extract to final volume.

6.4. GRADUATED CYLINDERS - Suggested sizes include 5, 10, 250 and 1000 mL.

6.5. MICRO SYRINGES - Suggested sizes include 10, 25, 50, 100, 250, 500, and 1000 µL.

6.6. DRYING COLUMN - The drying column must be able to contain 10-12 g of anhydrous sodium sulfate (Na_2SO_4). The drying column should not leach interfering compounds or irreversibly adsorb method analytes. Any small glass or polypropylene column may be used, such as Supelco #57176.

6.7. COLLECTION TUBES - 15 to 50 mL, conical tubes (Fisher #05-569-6C) or other glassware suitable for collection of the eluent from the solid phase after extraction and for collecting extract from drying tube.

6.8. ANALYTICAL BALANCE - Capable of weighing to the nearest 0.0001 g.

6.9. SPE APPARATUS USING PTFE DISKS; MANUAL EXTRACTION

6.9.1. SPE DISKS - 47-mm diameter and 0.5-mm thick, manufactured with a styrene divinylbenzene (SDVB) sorbent (Empore SDB-XC, #2240 or equivalent).

Note: Several brands of SDVB and modified SDVB media in cartridge format were evaluated during method development and did not provide satisfactory performance. Therefore, SDVB cartridges are not included in this method.

6.9.2. SPE DISK EXTRACTION GLASSWARE – glass funnel, PTFE-coated support screen, PTFE gasket, base, and clamp used to support SPE disks and contain samples during extraction. May be purchased as a set (Fisher #K971100-0047, Kontes #971000-6047, or equivalent) or separately.

6.9.3. VACUUM EXTRACTION MANIFOLD – Designed to accommodate extraction glassware and disks (Kontes #971000-6047, or UCT #ECUCTVAC6, or equivalent).

6.9.4. An automatic or robotic system designed for use with PTFE disks may be used if all quality control requirements discussed in Sect. 9 are met. Automated systems may use either vacuum or positive pressure to process samples and solvents through the disk. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries.

6.10. SPE APPARATUS USING SPEEDISKS; MANUAL EXTRACTION

6.10.1. SPEEDISKS - J.T. Baker H_2O Phobic Divinylbenzene (DVB) Speedisk, regular capacity (part #8068-06).

6.10.2. VACUUM EXTRACTION MANIFOLD - J.T. Baker #8095-06

6.10.3. An automatic or robotic system designed for use with SPEEDISKS may be used if all quality control requirements discussed in Sect. 9 are met. Automated systems may use either vacuum or positive pressure to process samples and solvents through the disk. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries.

6.11. SPE APPARATUS USING SPE CARTRIDGES (6-mL COLUMNS); MANUAL EXTRACTION

6.11.1. SPE CARTRIDGES - Modified DVB polymer (not SDVB polymer-see note below).

Note: Several brands of SDVB and modified SDVB media in cartridge format were evaluated during method development and did not provide satisfactory performance. Therefore, SDVB cartridge products are not included in this method.

6.11.1.1. Waters Oasis HLB, 500 mg (Waters #186000115) - divinylbenzene N-vinylpyrrolidone copolymer

6.11.1.2. J.T. Baker Speedisk Column H_2O Phobic DVB, 200 mg (#8109-09) - divinylbenzene polymer

6.11.2. VACUUM EXTRACTION MANIFOLD - Equipped with flow/vacuum control (Supelco #57030-U or equivalent).

6.11.3. SAMPLE DELIVERY SYSTEM - Use of a transfer tube system (Supelco "Visiprep", #57275 or equivalent), which transfers the sample directly from the sample container to the SPE cartridge is recommended.

6.11.4. An automatic or robotic system designed for use with SPE cartridges may be used if all quality control requirements discussed in Sect. 9 are met. Automated systems may use either vacuum or positive pressure to process samples and solvents through the cartridge. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries.

6.12. SPE APPARATUS USING SPE CARTRIDGES (83-mL COLUMNS); MANUAL EXTRACTION

6.12.1. SPE CARTRIDGES - United Chemical Technologies (UCT) 525 Universal cartridge, #ECUNI525, 1500 mg octadecyl unendcapped bonded silica, carbon loading 15-18%, surface area approximately 500 m^2/g, or equivalent.

6.12.2. VACUUM EXTRACTION MANIFOLD - Designed to accommodate extraction glassware and cartridges (Varian #1214-6001 or UCT # ECUCTVAC6 or equivalent).

6.12.3. CARTRIDGE ADAPTER - UCT #ECUCTADP

6.12.4. BOTTLE HOLDER - UCT #ECUNIBHD

6.12.5. An automatic or robotic system designed for use with SPE cartridges may be used if all quality control requirements discussed in Sect. 9 are met. Automated systems may use either vacuum or positive pressure to process samples and solvents through the cartridge. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as

those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries.

6.13. SPE APPARATUS USING HORIZON ATLANTIC DVB DISKS WITH AUTOMATED OR MANUAL EXTRACTION

6.13.1. SPE DISKS - Horizon Atlantic DVB disk, 50 mm diameter, 4mm thick, #47-2346-06, or equivalent

6.13.2. DISK HOLDER ASSEMBLY – 50 mm disk holder with extended riser, Horizon #50-2629 or equivalent

6.13.3. VACUUM EXTRACTION MANIFOLD – Equipped with flow/vacuum control (Supelco #57030-U) or equivalent

6.13.4. SPE-DEX 4790 Automated Extraction System, Horizon Technology

6.14. EXTRACT CONCENTRATION SYSTEM - Extracts are concentrated by evaporation with nitrogen gas using a water bath set at 40 °C (N-Evap, Model 11155, Organomation Associates, Inc., or equivalent).

6.15. LABORATORY OR ASPIRATOR VACUUM SYSTEM - Sufficient capacity to maintain a vacuum of approximately 15 to 25 inches of mercury.

6.16. GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) SYSTEM

6.16.1. FUSED SILICA CAPILLARY GC COLUMN - 30 m x 0.25-mm inside diameter (i.d.) fused silica capillary column coated with a 0.25 μm bonded film of poly(dimethylsiloxy)poly(1,4-bis(dimethylsiloxy)phenylene)-siloxane (Restek RXI-5sil-MS or equivalent). Any capillary column that provides adequate capacity, resolution, accuracy, and precision may be used. A nonpolar, low-bleed column is recommended for use with this method to provide adequate resolution and minimize column bleed.

6.16.2. GC INJECTOR AND OVEN - Some of the target compounds included in this method are subject to thermal breakdown in the GC injection port. This problem is exacerbated when the injector and/or the injection port liner is

not properly deactivated or is operated at excessive temperatures. The injection system must not allow analytes to contact hot stainless steel or other metal surfaces that promote decomposition. The performance data in Sect. 17 were obtained using hot, splitless injection using a 4 or 5-mm i.d. glass deactivated liner. Other injection techniques such as temperature programmed injections, cold on-column injections and large volume injections may be used if the QC criteria in Sect. 9 are met. Equipment designed appropriately for these alternate types of injections must be used if these options are employed.

6.16.3. GC/MS INTERFACE – The interface should allow the capillary column or transfer line exit to be placed within a few millimeters of the ion source. Other interfaces are acceptable as long as the system has adequate sensitivity and QC performance criteria are met.

6.16.4. MASS SPECTROMETER (MS) - Any type of MS may be used (i.e., quadrupole, ion trap, time of flight, etc.) with electron impact ionization. The instrument may be operated in full scan mode or in SIM mode for enhanced sensitivity. The minimum scan range capability of the MS must be 45 to 450 *m/z*, and it must produce a full scan mass spectrum that meets all criteria in Table 2 when a solution containing 5 ng (or less) of decafluorotriphenylphosphine (DFTPP) is injected into the GC/MS (Sect. 10.2.1).

6.16.5. DATA SYSTEM - An interfaced data system is required to acquire, store, and output MS data. The computer software should have the capability of processing stored GC/MS data by recognizing a GC peak within a given retention time window. The software must allow integration of the ion abundance of any specific ion between specified time or scan number limits. The software must be able to construct linear regressions and quadratic calibration curves, and calculate analyte concentrations.

7. REAGENTS AND STANDARDS SUPPLIES (References to specific brands or catalog numbers are included for illustration only, and do not imply endorsement of the product.)

7.1. REAGENTS AND SOLVENTS - Reagent grade or better chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents will conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, if the reagents are demonstrated free of analytes and interferences, and all method requirements in the Initial Demonstration of Capability (IDC) are met.

7.1.1. HELIUM - 99.999 % or better, GC carrier gas.

7.1.2. LABORATORY REAGENT WATER (LRW) - Purified water which does not contain any measurable quantities of any target analytes or interfering compounds at or above $^1/_3$ the MRL for each compound of interest.

7.1.3. METHANOL (MeOH) (CASRN 67-56-1) - High purity, demonstrated to be free of analytes and interferences (Fisher Optima or equivalent).

7.1.4. ETHYL ACETATE (EtOAc) (CASRN 141-78-6) - High purity, demonstrated to be free of analytes and interferences (Tedia Absolv or equivalent).

7.1.5. DICHLOROMETHANE (DCM) (CASRN 75-09-02) - High purity, demonstrated to be free of analytes and interferences (Fisher GC Resolv or equivalent).

7.1.6. ACETONE (CASRN 67-64-1) - High purity, demonstrated to be free of analytes and interferences (Tedia Absolv or equivalent).

7.1.7. SODIUM SULFATE (Na_2SO_4), ANHYDROUS (CASRN 7757-82-6) - Soxhlet extracted with DCM for a minimum of four hours or heated to 400 °C for two hours in a muffle furnace. An "ACS grade, suitable for pesticide residue analysis," is recommended.

7.1.8. SAMPLE PRESERVATION REAGENTS – The following preservatives are solids at room temperature and may be added to the sample bottle before shipment to the field.

7.1.8.1. POTASSIUM DIHYDROGEN CITRATE (CASRN 866-83-1) - The sample must be buffered to pH 3.8 to inhibit microbial growth and analyte degradation.[20]

7.1.8.2. L-ASCORBIC ACID (CASRN 50-81-7) - Ascorbic acid reduces free chlorine at the time of sample collection (ACS Reagent Grade or equivalent).[21]

7.1.8.3. ETHYLENEDIAMINE TETRAACETIC ACID (EDTA), TRISODIUM SALT (CASRN 10378-22-0) - Trisodium EDTA is added to inhibit metal-catalyzed hydrolysis of analytes.[20,21]

7.2. STANDARD SOLUTIONS - Standard solutions of internal standards, surrogates and method analytes may be prepared gravimetrically or from commercially available stock solutions. When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of a gravimetrically prepared stock standard. Solution concentrations listed in this section were those used to develop this method and are included as an example only. Solution preparation steps may be modified as needed to meet the needs of the

laboratory. Often, standard mixes appropriate to the method become commercially available subsequent to method publication. **Even though stability times for standard solutions are suggested in the following sections, laboratories should use standard QC practices to determine when their standards need to be replaced. In addition, signs of evaporation and/or discoloration are indicators that a standard should be replaced.**

7.2.1. INTERNAL STANDARD (IS) SOLUTIONS - This method uses four IS compounds listed in the table below. ISs 1-3 are the same as those used in previous versions of this method and are available individually and as a mixture from many commercial sources. A commercial mixture of ISs 1-3 was used for method development, and it is highly recommended that other analysts use a commercial mix as well. However, if an analyst chooses to prepare a gravimetric stock solution, it should be prepared in acetone using a procedure similar to the preparation of analyte stocks as outlined in Sect. 7.2.3.1. The PDS mix for ISs 1-3 has been shown to be stable for at least one year when stored in amber glass screw cap vials at -5 °C or less. The PDS for IS 4, ^{13}C-pentachlorophenol, was prepared from neat material in methanol at 1000 µg/mL. The neat material was obtained from CDN Isotopes. This IS PDS has been shown to be stable for at least nine months when stored in amber glass screw cap vials at -5 °C or less. Using 10 µL of the PDS for ISs 1-3 and 4 µL of the PDS for IS 4 to fortify the final 1-mL extracts (Sect. 11.9) will yield a concentration of 5 µg/mL each for ISs 1-3 and 4 µg/mL of IS 4 for full scan analysis. Lower concentrations of ISs 1-3 should be used for SIM analysis. For SIM analysis during method development, ISs 1-3 were added to extracts such that their final concentration was 1 µg/mL. The IS 4 concentration remained at 4 µg/mL for most analyses due to the low abundance of the *m/z* 276 QI. **IS 4 should be omitted if pentachlorophenol is not being measured as an analyte.**

Note: Stock standard solutions and PDSs should be brought to room temperature and sonicated for few minutes prior to use. This ensures that components are dissolved and the solution is homogeneous.

Internal Standards	CASRN	Solvent	PDS conc.
acenaphthene-d_{10} (IS 1)	15067-26-2	acetone	500 µg/mL
phenanthrene-d_{10} (IS 2)	1517-22-2	acetone	500 µg/mL
chrysene-d_{12} (IS 3)	1719-03-5	acetone	500 µg/mL
^{13}C-pentachlorophenol (IS 4)	85380-74-1	methanol	1000 µg/mL

7.2.2. SURROGATE ANALYTE STANDARD SOLUTIONS - The surrogate analytes used in this method are listed in the table below. All SUR PDSs were used at the same concentration and were prepared in acetone. The

SURs may be prepared or purchased (if available from commercial suppliers) as individual PDSs or a single PDS. The SUR PDSs have been shown to be stable for at least one year when stored in amber glass screw cap vials at -5 ºC or less. For full scan analysis, 10 µL of each of these solutions was added to each 1L aqueous QC and Field Sample prior to extraction, for an expected final extract concentration of 5 µg/L of each SUR. For SIM analyses the QC and Field Samples were fortified such that the expected final extract concentration was 1 µg/L for each SUR.

Surrogates	CASRN	Solvent	PDS conc.
1,3-dimethyl-2-nitrobenzene (SUR 1)	81-20-9	acetone	500 µg/mL
triphenyl phosphate (SUR 2)	115-86-6	acetone	500 µg/mL
benzo[*a*]pyrene-d_{12} (SUR 3)	63466-71-7	acetone	500 µg/mL

Notes:

- Stock standard solutions and PDSs should be brought to room temperature and sonicated for a few minutes prior to use. This ensures that components are dissolved and the solution is homogeneous.
- Stock standard solutions that will be used for aqueous sample fortification generally should be prepared at a concentration such that only a small volume (e.g., 5-100 µL) needs to be added to achieve the desired final concentration. This will minimize the quantity of organic solvent added to aqueous samples.

7.2.3. ANALYTE STOCK SOLUTIONS

7.2.3.1. ANALYTE STOCK STANDARD SOLUTIONS (SSS) (0.5-5.0 mg/mL) - Analyte standards may be purchased commercially as ampulized solutions prepared from neat materials. Commercially prepared SSSs are widely available for most method analytes. Exceptions are DIMP, which at the time of method development was available only from Cerilliant, and the 14 PCB congeners. During method development, custom PCB mixes were obtained from Accustandard and Chem Service, Inc. Many groups of analytes can also be purchased as PDSs, making the preparation of individual SSSs unnecessary. To prepare gravimetric stock standard solutions, add 10 mg (weighed on analytical balance to 0.1 mg) of the pure material to 1.9 mL of solvent in a 2-mL volumetric flask, dilute to the mark, and transfer the solution to an amber glass vial. The suggested solvent for each analyte is identical to the PDS solvent listed in the "comments" portion of Table 1. If the neat material is only available in quantities less than 10 mg, reduce the volume of solvent accordingly. If compound purity is confirmed by the supplier to be ≥ 96%, the

weighed amount can be used without correction to calculate the concentration of the solution. Store at 4 ºC or less to guard against degradation and evaporation.

Note: Stock standard solutions and PDSs should be brought to room temperature and sonicated for a few minutes prior to use. This ensures that components are dissolved and the solution is homogeneous.

7.2.3.2. ANALYTE PRIMARY DILUTION STANDARD / ANALYTE FORTIFICATION SOLUTION (50 µg/mL for most compounds) - Prepare the 50-µg/mL Analyte PDS by volumetric dilution of the Analyte Stock Standard Solutions (Sect. 7.2.3.1) in acetone or methanol (see notes below) to make a 50-µg/mL solution. Analyte PDSs containing PCBs, PAHs, and other nonpolar compounds must be diluted in acetone rather than methanol. The PDS can be used to fortify the LFBs and LFSMs with method analytes and to prepare calibration solutions. Care should be taken during storage to prevent evaporation. The Analyte PDS/Analyte Fortification Solutions used during method development were stable for 6 months when prepared in the solvents indicated in the "comments" portion of Table 1, and stored in an amber glass screw cap vials at -5 °C or less.

Notes:

- It may be necessary to prepare multiple Analyte PDS mixtures based on the laboratory's specific compounds of interest. For example, the method was developed using an Analyte PDS mix containing analytes from Method 525.2 in acetone, a PCB PDS mix in acetone, a PDS mix containing analytes from EPA's Drinking Water Contaminant Candidate List 3 (CCL 3) in methanol, and a PDS mix containing the remaining analytes in methanol. The solvent used for the PDS solution for each analyte is listed in the "comments" portion of Table 1.
- During method development, PDS solutions contained most analytes at the same concentration. However, analytes can be combined in PDS solutions at any desired concentrations. It may be desirable to include some analytes at increased concentrations if they have a relatively low instrument response compared with other analytes. For example, during method development, pentachlorophenol was added to PDS solutions at four times the concentration of most other analytes.
- Standard solutions that will be used for aqueous sample fortification generally should be prepared at a concentration such that only a small volume (e.g., 5-100 µL) needs to be

added to achieve the desired final concentration. This will minimize the quantity of organic solvent added to aqueous samples.

- Stock standard solutions and PDSs should be brought to room temperature and sonicated for a few minutes prior to use. This ensures that components are dissolved and the solution is homogeneous.

7.2.4. CALIBRATION SOLUTIONS - Calibration standards may be prepared in EtOAc or as matrix-matched calibration standards (Sect. 3.15). This option is provided so that the analyst has the flexibility to prepare calibration curves that will be appropriate for the various types of analytes and the calibration range of interest. If the analyses to be performed include only those analytes that are not susceptible to matrix induced response enhancement, and/or the concentrations to be measured are relatively high (e.g., ≥ 5µg/L), it is likely that accurate data can be obtained with the use of traditional CAL standards prepared in EtOAc. If low concentrations of analytes susceptible to matrix induced response enhancement need to be measured, it is likely that matrix-matched standards will be required to obtain accurate quantitative data. Whichever type of CAL solutions are selected, those CAL solutions should be used for all calibration and QC procedures described in the method.

Note: Analytes observed to be susceptible to matrix induced response enhancement during method development are indicated in the "comments" portion of Table 1. However, the occurrence and degree of enhancement will depend upon the GC injector design, and the history of the injector, injector liner and GC column. It is highly recommended that prior to the initial demonstration of capability, separate calibration curves be generated using EtOAc CALs and matrix-matched CALs for each analyte to be measured. A careful evaluation of the relative peak areas using each type of CAL, especially low concentration CALs, can serve as a guide to the possible occurrence and extent of matrix enhancement, and thus an indicator of which type of standards should be used.

Note: Technical toxaphene CAL standards must be prepared so that they contain only technical toxaphene and ISs and SURs. Do not mix technical toxaphene with other analytes.

7.2.4.1. CALIBRATION SOLUTIONS PREPARED IN SOLVENT - Prepare a series of six concentrations of calibration solutions in EtOAc, which contain the analytes of interest. The suggested concentrations in this paragraph are a description of the concentrations used during method development, and may be modified to conform with instrument sensitivity. For full scan analyses, concentrations ranging from 0.10-5.0 ng/µL are

suggested for each analyte, with IS and SUR concentrations as described in Sect. 7.2.1 and 7.2.2. For SIM analysis, six concentrations in the range of 0.005-0.5 ng/µL are suggested, with reduced concentrations of the ISs and SURs (Sect. 7.2.1 and 7.2.2). The six CAL standards (CAL1 through CAL6) are prepared by combining appropriate aliquots of the Analyte PDS solution (Sect. 7.2.3.2) and the IS and SUR PDSs (Sects. 7.2.1. and 7.2.2). All calibration solutions should contain at least 60% EtOAc to avoid gas chromatographic anomalies such as poor peak shape, split peaks, etc. During method development, all analytes were prepared in a single set of calibration solutions. However, multiple sets of calibration standards may be prepared at the discretion of the analyst if more separation of the analytes is desired. Calibration solutions were stable for six months when stored at -5 ºC in amber screw top vials.

7.2.4.2. MATRIX-MATCHED CALIBRATION SOLUTIONS - Prepare a series of six calibration solutions in the same manner as in Sect. 7.2.4.1, but instead of preparation in EtOAc, calibration solutions are prepared in final solvent extracts derived from laboratory reagent water. One-liter aliquots of reagent water with sample preservatives added, are extracted using the sorbent selected for sample analysis, dried with sodium sulfate, and evaporated to <1 mL following the same procedure used for samples (Sects. 11.3-11.9). **However, the ISs, SURs, and analyte PDSs are added to the extract at appropriate concentrations immediately before the adjustment of the extract to 1 mL, i.e., they are <u>not</u> extracted.**

7.2.5. GC/MS TUNE CHECK SOLUTION (5 µg/mL or less) (CASRN 5074-71-5) - Prepare a DFTPP solution in DCM. DFTPP is more stable in DCM than in acetone or EtOAc. Store this solution in an amber glass screw cap vial at 4 °C or less.

7.2.6. 4,4′-DDT BREAKDOWN CHECK SOLUTION (5ng/µL in EtOAc) - Prepare a solution of 4,4′-DDT in EtOAc. Store this solution in an amber glass screw cap vial at 4 °C or less. The solution is used for the DDT breakdown check described in Sect. 10.2.2.

8. <u>SAMPLE COLLECTION, PRESERVATION, AND STORAGE</u>

8.1. SAMPLE BOTTLE PREPARATION

8.1.1. Grab samples must be collected using 1-liter or 1-quart sample bottles that meet the requirements in Sect. 6.1.

Note: Larger samples must not be collected. The performance of this method has not been verified with sample volumes greater than 1L. **Samples larger than 1L may not be portioned or split** because some analytes may adhere to the glass surface. These analytes are recovered in the SPE procedural steps that solvent rinse the sample bottle and include that rinsate in the extract. If only a partial sample were to be analyzed, the resulting data would be biased high if the bottle rinsate were added to the extract, and biased low if it was not.

8.1.2. Preservation reagents, listed in the table below, are added to each sample bottle as dry solids prior to shipment to the field (or prior to sample collection).

Compound	Amount	Purpose
L-Ascorbic acid	0.10 g/L	Dechlorination
Ethylenediaminetetraacetic acid, trisodium salt	0.35 g/L	Inhibit metal-catalyzed hydrolysis of targets
Potassium dihydrogen citrate	9.4 g/L	pH 3.8 buffer, microbial inhibitor

8.1.2.1. Residual chlorine must be reduced at the time of sample collection with 100 mg of ascorbic acid per liter.[21]

8.1.2.2. Trisodium EDTA must be added to inhibit metal-catalyzed hydrolysis of method analytes, principally, chlorpyrifos, parathion, vinclozolin, atrazine, and propazine.[20-22] There may be additional method analytes that also benefit from addition of EDTA.

8.1.2.3. The sample must be buffered to pH 3.8 using potassium dihydrogen citrate. This is added to inhibit microbial degradation of analytes, and to reduce base catalyzed hydrolysis of some of the method analytes.[20]

8.2. SAMPLE COLLECTION

8.2.1. Open the tap and allow the system to flush until the water temperature has stabilized (usually 3-5 min). Collect samples from the flowing system.

8.2.2. Fill sample bottles, taking care not to flush out the sample preservation reagents. Samples do not need to be collected headspace free.

8.2.3. After collecting the sample, cap the bottle and agitate by hand until preservatives are dissolved. Immediately place in ice or refrigerate.

8.3. SHIPMENT AND STORAGE - Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when they are received at the laboratory, with

the following exception. Samples arriving at the laboratory on the day of sampling may not have had time to achieve a temperature of less than 10 °C. This is acceptable as long as the cooling process has begun. Samples stored in the lab must be held at or below 6 °C until extraction, but should not be frozen. Sample holding time data are discussed in Sect. 13.4.

Note: Samples that are significantly above 10 ºC at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

8.4. SAMPLE AND EXTRACT HOLDING TIMES - Water samples should be extracted as soon as possible after collection but must be extracted within 14 days of collection. Exceptions to the 14 day sample holding time are samples being analyzed for dichlorvos and/or cyanazine which must be extracted within 7 days. All extracts must be stored at -5 °C or less, protected from light and analyzed within 28 days after extraction (Sect. 13.5).

9. QUALITY CONTROL

9.1. QC requirements include the Initial Demonstration of Capability (IDC) and ongoing QC requirements that must be met when preparing and analyzing Field Samples. This section describes QC parameters, their required frequency, and the performance criteria that must be met in order to meet EPA quality objectives. The QC criteria discussed in the following sections are summarized in Tables 24 and 25. These QC requirements are considered the minimum acceptable QC criteria. Laboratories are encouraged to institute additional QC practices to meet their specific needs.

Note: If toxaphene is being measured by this method in addition to other analytes, all QC steps described in Sect. 9 must be performed for toxaphene in fortified samples that are prepared and analyzed separately from other analytes. The frequency of each QC requirement and the acceptance criteria for toxaphene are the same as the general criteria for other analytes. Measured values for technical toxaphene to meet QC criteria should be obtained as described in Sect. 12.3.3.

9.2. INITIAL DEMONSTRATION OF CAPABILITY - The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in Sect. 10.2. **The IDC must be repeated if the laboratory changes the type or brand of SPE sorbent being used.**

9.2.1. INITIAL DEMONSTRATION OF LOW SYSTEM BACKGROUND - Any time a new lot of SPE cartridges or disks is used, it must be demonstrated that a Laboratory Reagent Blank is reasonably free of contamination and that the criteria in Sect. 9.3.1 are met.

9.2.2. INITIAL DEMONSTRATION OF PRECISION (IDP) - Prepare, extract, and analyze four to seven replicate LFBs fortified near the midrange of the initial calibration curve according to the procedure described in Sect. 11. Sample preservatives as described in Sect. 8.1.2 must be added to these samples. The relative standard deviation (RSD) of the results of the replicate analyses must be ≤ 20%.

9.2.3. INITIAL DEMONSTRATION OF ACCURACY - Using the same set of replicate data generated for Sect. 9.2.2, calculate average recovery. The average recovery expressed as the mean of the replicate values must be within 70-130 % of the true value for all analytes except dimethipin, HCCPD and HCB which may be within 60-130% of the true value.

9.2.4. MINIMUM REPORTING LEVEL (MRL) CONFIRMATION - Establish a target concentration for the MRL based on the intended use of the method. The MRL may be established by a laboratory for their specific purpose or may be set by a regulatory agency. Establish an Initial Calibration following the procedure outlined in Sect. 10.2. The lowest calibration standard used to establish the Initial Calibration (as well as the low-level Continuing Calibration Check standard) must be at or below the concentration of the MRL. Establishing the MRL concentration too low may cause repeated failure of ongoing QC requirements. Confirm the MRL following the procedure outlined below.

Note: Setting an MRL for method analytes that are consistently present in the background (e.g., phthalates) is particularly important so that false positive data are not reported for Field Samples. See Sect. 9.3.1 for guidance in setting an MRL for these analytes.

9.2.4.1. Fortify, extract, and analyze seven replicate Laboratory Fortified Blanks (LFBs) at the proposed MRL concentration. These LFBs must contain all method preservatives described in Sect. 8.1.2. Calculate the mean and standard deviation for these replicates. Determine the Half Range for the Prediction Interval of Results (HR_{PIR}) using the equation below

$$HR_{PIR} = 3.963S$$

where:

S = the standard deviation, and 3.963 is a constant value for seven replicates.[1]

9.2.4.2. Confirm that the upper and lower limits for the Prediction Interval of Result (PIR = Mean + HR_{PIR}) meet the upper and lower recovery limits as shown below:

The Upper PIR Limit must be ≤150 percent recovery.

$$\frac{Mean + HR_{PIR}}{Fortified\,Concentration} \times 100\% \leq 150\%$$

The Lower PIR Limit must be ≥ 50 percent recovery.

$$\frac{Mean - HR_{PIR}}{Fortified\,Concentration} \times 100\% \geq 50\%$$

9.2.4.3. The MRL is validated if both the Upper and Lower PIR Limits meet the criteria described above (Sect. 9.2.4.2). If these criteria are not met, the MRL has been set too low by the laboratory and must be demonstrated again at a higher concentration. If a required MRL set by a regulatory body has not been met, the analyst should evaluate possible problems in the execution of the extraction steps, and/or possible problems with instrument sensitivity. Reattempt MRL validation at the required MRL after problems have been addressed.

9.2.4.4. Confirmation of the MRL Using Fortified Matrix Samples *(optional)- This validation procedure may be used in addition to the reagent water confirmation described above. It may be useful in assessing any matrix induced quantitative bias at the MRL.*

Obtain replicate 1 L aliquots of a water sample similar in nature to the ones planned for analysis. If tap waters from both ground and surface water sources are to be analyzed, it is recommended that a surface water sample be selected for verification. Analyze one aliquot using the procedures in this method to verify the absence of analytes of interest. Fortify seven remaining aliquots with the analytes to be measured near the expected MRL, and verify the MRL as described in Sects. 9.2.4.1 through 9.2.4.3.

9.2.5. CALIBRATION CONFIRMATION - Analyze a Quality Control Sample as described in Sect. 9.3.9 to confirm the accuracy of the standards/calibration curve.

9.2.6. DETECTION LIMIT DETERMINATION *(optional) - While DL determination is not a specific requirement of this method, it may be required by various regulatory bodies associated with compliance monitoring. It is the responsibility of the laboratory to determine if DL determination is required based upon the intended use of the data.*

Replicate analyses for this procedure should be done over at least three days (both the sample extraction and the GC analyses should be done over at least three days). Prepare at least seven replicate LFBs at a concentration estimated to be near the DL. This concentration may be estimated by selecting a concentration at 2-5 times the noise level. The DLs in Tables 15 and 23 were calculated from LFBs fortified at various concentrations as indicated in the table. The appropriate fortification concentrations will be dependent upon the sensitivity of the GC/MS system used. All preservation reagents listed in Sect. 8.1.2 must also be added to these samples. Analyze the seven (or more) replicates through all steps of Sects. 11 and 12.

Note: If an MRL confirmation data set meets these requirements, a DL may be calculated from the MRL confirmation data, and no additional analyses are necessary.

Calculate the *DL* using the following equation:

$$DL = s \times t_{(n-1,\,1-\alpha=0.99)}$$

where:

$t_{(n\text{-}1,\ 1\text{-}\alpha=0.99)}$ = 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.

Note: Do not subtract blank values when performing DL calculations.

9.3. ONGOING QC REQUIREMENTS - This section summarizes the ongoing QC criteria that must be followed when processing and analyzing Field Samples.

9.3.1. LABORATORY REAGENT BLANK (LRB) - An LRB is required with each extraction batchto confirm that potential background contaminants are not interfering with the identification or quantitation of target analytes. If the LRB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, locate the source of contamination and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that interfere with the measurement of method analytes must be at or below $^1/_3$ of the MRL. Blank contamination may be estimated by extrapolation, if the concentration is below the lowest calibration standard. Although this procedure is not allowed for sample results as it may not meet data quality objectives, it can be useful in estimating background concentrations. If any of the method analytes are detected in the LRB at concentrations greater

than $^{1}/_{3}$ of the MRL, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

Note: It is extremely important to evaluate background values of analytes that commonly occur in LRBs. The MRL must be set at a value greater than three times the mean concentration observed in replicate LRBs. If LRB values are highly variable, setting the MRL to a value greater than the mean LRB concentration plus three times the standard deviation may provide a more realistic MRL.

9.3.2. CONTINUING CALIBRATION CHECK (CCC) - CCC Standards are analyzed at the beginning of each analysis batch, after every ten Field Samples, and at the end of the analysis batch. See Sect. 10.3 for concentration requirements and acceptance criteria.

9.3.3. LABORATORY FORTIFIED BLANK (LFB) - An LFB is required with each extraction batch. The fortified concentration of the LFB must be rotated between low, medium, and high concentrations from batch to batch. The low concentration LFB must be as near as practical to, but no more than two times the MRL. Similarly, the high concentration LFB should be near the high end of the calibration range established during the initial calibration (Sect. 10.2). Results of the low-level LFB analyses must be 50-150% of the true value. Results of the medium and high-level LFB analyses must be 70-130% of the true value for all analytes except dimethipin, HCCPD and HCB, which may be between 60-130% of the true value. If the LFB results do not meet these criteria for target analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.3.4. MS TUNE CHECK - A complete description of the MS Tune Check is found in Sect. 10.2.1. The acceptance criteria for the MS Tune Check are summarized in Table 2. The MS Tune Check must be performed each time a major change is made to the mass spectrometer, and prior to establishing and/or re-establishing an initial calibration (Sect. 10.2). Daily DFTPP analysis is not required.

Note: The tune check is performed in full scan mode, even if samples will be analyzed in SIM mode.

9.3.5. INTERNAL STANDARDS (IS) - The analyst must monitor the peak areas of the ISs in all injections during each analysis day. The peak area for each IS in any chromatographic run must not deviate by more than ±50% from the mean response in the CAL solutions analyzed for the initial analyte calibration. In addition, the peak areas of ISs 1-3 must not deviate by more than ± 30% from the most recent CCC. IS 4, ^{13}C-pentachlorophenol, is expected to have greater area count variability, and therefore is not subject

to the most recent CCC criterion. If the IS areas in a chromatographic run do not meet these criteria, inject a second aliquot of that standard or extract.

9.3.5.1. If the reinjected aliquot produces acceptable internal standard responses, report results for that aliquot.

9.3.5.2. If the reinjected aliquot is a sample extract and fails again, the analyst should check the calibration by evaluating the CCCs within the analysis batch. If the CCCs are acceptable, extraction of the sample may need to be repeated provided the sample is still available and within the holding time. Otherwise, report results obtained from the reinjected extract, but annotate as suspect. Alternatively, collect a new sample and reanalyze.

9.3.5.3. If the reinjected aliquot is a CAL standard, take remedial action (Sect. 10.3.3).

9.3.6. SURROGATE RECOVERY - Surrogate standards are fortified into the aqueous portion of all samples, LRBs, CCCs, LFSMs, and LFSMDs prior to extraction. They are also added to the calibration standards. The surrogates are a means of assessing method performance from extraction to final chromatographic measurement. Calculate the recovery (%R) for each surrogate using the equation

$$\%R = \left(\frac{A}{B}\right) \times 100$$

where:

A = calculated surrogate concentration for the QC or Field Sample, and
B = fortified concentration of the surrogate.

9.3.6.1. Surrogate recovery must be with 70-130% of the true value. When surrogate recovery from a sample, blank, or CCC is less than 70% or greater than 130%, check 1) calculations to locate possible errors, 2) the integrity of the surrogate analyte solution, 3) contamination, and 4) instrument calibration. Correct the problem and reanalyze the extract.

9.3.6.2. If the extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract.

9.3.6.3. If the extract reanalysis fails the 70-130% recovery criterion, the analyst should check the calibration by evaluating the CCCs within the analysis batch. If the CCCs fail the criteria of Sect. 9.3.6.1, recalibration is in order per Sect.10.2. If the calibration standard is

acceptable, extraction of the sample should be repeated provided the sample is still available and within the holding time. If the re-extracted sample also fails the recovery criterion, report all data for that sample as suspect/surrogate recovery to inform the data user that the results are suspect due to surrogate recovery.

9.3.7. LABORATORY FORTIFIED SAMPLE MATRIX (LFSM) - Within each analysis batch, analyze a minimum of one LFSM. The native concentrations of the analytes in the sample matrix must be determined in a second duplicate sample and subtracted from the measured values in the LFSM. If a variety of different sample matrices are analyzed regularly, for example, drinking water from ground water and surface water sources, performance data must be collected for each source.

9.3.7.1. Prepare the LFSM by fortifying a Field Duplicate with an appropriate amount of analyte PDS (Sect. 7.2.3.2). Select a fortification concentration that is greater than or equal to the matrix background concentration, if known. Selecting a duplicate sample that has already been analyzed aids in the selection of an appropriate fortification concentration. If this is not possible, use historical data. If historical data are unavailable, rotate the fortifying concentrations for LFSMs between low, medium and high concentrations based on the calibration range.

9.3.7.2. Calculate the percent recovery (%*R*) for each analyte using the equation

$$\%R = \frac{(A - B)}{C} \times 100$$

where:

A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration.

Note: LFSMs and LFSMDs fortified at concentrations near the MRL, where the associated Field Sample contains native analyte concentrations above the DL but below the MRL, should be corrected for the native levels in order the obtain meaningful %R values. This example, and the LRB extrapolation (Sect. 9.3.1), are the only permitted uses of analyte results below the MRL.

9.3.7.3. Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should be within 70-130% (60-130% for dimethipin, HCCPD and HCB), except for

low-level fortification near or at the MRL (within a factor of two times the MRL concentration) where 50-150% recoveries are acceptable. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCCs, the recovery is judged to be matrix biased. The quantitative result for that analyte in the unfortified sample is labeled "suspect/matrix" to inform the data user that the quantitative results may be suspect due to matrix effects.

9.3.8. FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or LFSMD) - Within each extraction batch, analyze a minimum of one Field Duplicate (FD) or Laboratory Fortified Sample Matrix Duplicate (LFSMD). Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If target analytes are not routinely observed in Field Samples, an LFSMD should be analyzed rather than an FD.

9.3.8.1. Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1* and *FD2*) using the equation

$$RPD = \frac{\left|FD1 - FD2\right|}{\left(FD1 + FD2\right)/2} \times 100$$

9.3.8.2. RPDs for Field Duplicates should be ≤ 30 %. Greater variability may be observed when Field Duplicates have analyte concentrations that are within two times the MRL. At these concentrations, Field Duplicates should have RPDs that are ≤ 50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCC, the recovery is judged to be affected by the matrix. The result for that analyte in the unfortified sample is labeled "suspect/matrix" to inform the data user that the quantitative results may be suspect due to matrix effects.

9.3.8.3. If an LFSMD is analyzed instead of a Field Duplicate, calculate the relative percent difference (RPD) for duplicate LFSMs (LFSM and LFSMD) using the equation

$$RPD = \frac{\left|LFSM - LFSMD\right|}{\left(LFSM + LFSMD\right)/2} \times 100$$

9.3.8.4. RPDs for duplicate LFSMs should be ≤ 30% for samples fortified at or above their native concentration. Greater variability may be observed when LFSMs are fortified at analyte concentrations that

are within two times the MRL. LFSMs fortified at these concentrations should have RPDs that are ≤ 50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCC, the recovery is judged to be affected by the matrix. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the quantitative results may be suspect due to matrix effects.

9.3.9. QUALITY CONTROL SAMPLES (QCS) - As part of the IDC (Sect. 9.2), each time a new Analyte PDS (Sect. 7.2.3.2) is prepared, or at least quarterly, analyze a QCS sample from a source different from the source of the calibration standards. If a second vendor is not available then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCC. Acceptance criteria for the QCS are identical to the CCCs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem. If the discrepancy is not resolved, one of the standard materials may be degraded or otherwise compromised and a third standard must be obtained.

9.4. METHOD MODIFICATION QC REQUIREMENTS - The analyst is permitted to modify GC columns, GC conditions, GC injection techniques, extract evaporation techniques, MS conditions and QIs. However, each time such method modifications are made, the analyst must repeat the procedures of the IDC (Sect. 9.2).

9.4.1. Each time method modifications are made, the analyst must repeat the procedures of the IDC (Sect. 9.2) and verify that all QC criteria can be met in ongoing QC samples (Sect. 9.3).

9.4.2. Each time method modifications are made, the analyst is also required to evaluate and document method performance for the proposed method modifications in real matrices that span the range of waters that the laboratory analyzes. This additional step is required because modifications that perform acceptably in the IDC, which is conducted in reagent water, can fail ongoing method QC requirements in real matrices. This is particularly important for methods subject to matrix effects. If, for example, the laboratory analyzes finished waters from both surface and groundwater municipalities, this requirement can be accomplished by assessing precision and accuracy (Sects. 9.2.2 and 9.2.3) in an analyte fortified surface water with moderate to high TOC (e.g., 2 mg/L or greater) and an analyte fortified hard groundwater (e.g., 250 mg/L or greater as calcium carbonate).

9.4.3. The results of Sects. 9.4.1 and 9.4.2 must be appropriately documented by the analyst and should be independently assessed by the laboratory's Quality Assurance (QA) officer prior to analyzing Field Samples. When implementing method modifications, it is the responsibility of the laboratory to closely review the results of ongoing QC, and in particular, the results associated with the LFSMs (Sect. 9.3.7), FDs or LFSMDs (Sect. 9.3.8), CCCs (Sect. 9.3.2), and the IS area counts (Sect. 9.3.5). If repeated failures are noted, the modification must be abandoned.

10. CALIBRATION AND STANDARDIZATION

10.1. Demonstration and documentation of acceptable mass spectrometer tune and initial calibration is required before performing the IDC and prior to analyzing Field Samples. The MS tune check and initial calibration must be repeated each time a major instrument modification is made, or maintenance is performed.

10.2. INITIAL CALIBRATION

10.2.1. MS TUNE/MS TUNE CHECK - Calibrate the mass and abundance scales of the MS with calibration compounds and procedures prescribed by the manufacturer with any modifications necessary to meet tuning requirements. Inject 5 ng or less of the DFTPP solution (Sect. 7.2.5) into the GC/MS system. Acquire a mass spectrum that includes data for *m/z* 45 to 450. The scan time should be set so that a minimum of five scans are acquired during the elution of the chromatographic peak. Seven to ten scans per chromatographic peak are recommended. Use a single spectrum at the apex of the DFTPP peak, an average spectrum of the three highest points of the peak, or an average spectrum across the entire peak to evaluate the performance of the system. If the DFTPP mass spectrum does not meet all criteria in Table 2, the MS must be retuned and adjusted to meet all criteria before proceeding with the initial calibration. The tune check should be conducted as described above for both full scan and SIM MS operation.

10.2.2. DDT BREAKDOWN CHECK -

Note: This procedure is only required if the DDT degradation products DDD and DDE are being reported. The DFTTP tune check and the DDT degradation check may be performed simultaneously with a single injection of a standard containing both analytes.

4,4′-DDT is subject to thermal degradation in the GC. Because the thermal degradation products 4,4′-DDE and 4,4′-DDD are also environmental contaminants and method analytes, it is important to determine their source if they are observed in sample chromatograms. Inject a standard of 4,4′-DDT at a concentration near 5 ng/µL, and acquire data in the full scan mode

using instrument conditions similar to those in Sect. 10.2.3. Evaluate the chromatogram for the presence of 4,4′-DDE and 4,4′-DDD using the retention times and ions listed in Table 1 as a guide. If either 4,4′-DDE or 4,4′-DDD are observed, calculate the percentage breakdown of 4,4′-DDT using peak areas from the Total Ion Current (TIC) with the following equation:

% DDT breakdown =

$$\frac{\sum \text{TIC area of DDT degradation peaks (DDE+DDD)}}{\sum \text{TIC area of total DDT peaks (DDT+DDE+DDD)}} \times 100$$

If the degradation of 4,4′-DDT exceeds 20%, perform maintenance on the GC injection port and possibly other areas of the GC prior to proceeding with the calibration. If GC maintenance does not correct the problem, a new 4,4′-DDT standard should be obtained from a different source to ensure that the standard material has not degraded.

10.2.3. INSTRUMENT CONDITIONS - Operating conditions used during method development are described below. Conditions different from those described may be used if QC criteria in Sect. 9 are met. Different conditions include alternate GC columns, temperature programs, MS conditions, and injection techniques and volumes, such as cold on-column and large volume injections. Equipment specifically designed for alternate types of injections must be used if these alternate options are selected.

10.2.3.1. GC Conditions - Inject a 1-µL aliquot into a hot, splitless injection port held at 275 ºC with a pressure pulse of 30 psi and a split delay of 1 min. The temperature program is as follows: initial oven temperature of 70 ºC, hold for 1.5 min, ramp at 10 ºC/min to 200 ºC, ramp at seven ºC/min to a final temperature of 320 ºC and hold for 3 min. The GC was operated at a constant flow rate of 1.2 mL/min. Total run time is approximately 32 min. Begin data acquisition at about seven min.

10.2.3.2. Full Scan MS Acquistion Parameters - Select a scan range that allows the acquisition of a mass spectrum for each of the method analytes, which includes all of the major fragments *m/z* 45 and above. Adjust the cycle time to measure at least five spectra during the elution of each GC peak. Seven to ten scans across each GC peak are recommended. The chromatogram may be divided into time windows, also known as segments or periods, with different scan ranges for each time window. Minimizing the scan range for each time window may enhance sensitivity. If the chromatogram is divided into time windows, the laboratory must ensure that each method analyte elutes entirely within the proper

window during each analysis. This can be achieved by carefully monitoring the retention times of all ISs and SURs in each sample, and carefully monitoring the retention times of all method analytes in CCCs, LFBs and LFMs. This requirement does not preclude continuous operation by sequencing multiple analysis batches; however, the entire analysis batch is invalid if one or more analyte peaks have drifted outside of designated time windows in the CCC at the beginning or end of the analysis batch.

10.2.3.3. SIM MS Acquistion Parameters - Prior to selecting SIM parameters, analyze a mid- to high-concentration CAL in full scan mode. Select one primary QI and at least one secondary ion for confirmation. If possible, select a second confirmation ion. Suggested QIs and secondary ions for all method analytes are designated in Table 1, but these may be modified. An internal standard for each analyte is also designated in Table 1. Verify that the primary ion is free from interferences due to an identical fragment ion in any overlapping peak(s). Selection of the QI should be based on the best compromise between the intensity of the signal for that ion and the likelihood and intensity of interferences. The most intense ion may not be the best QI. Adjust the cycle time to measure at least five spectra during the elution of each GC peak. If the chromatogram is divided into time windows, the laboratory must ensure that each method analyte elutes entirely within the proper window during each analysis. This can be achieved by carefully monitoring the retention times of all ISs and SURs in each sample, and carefully monitoring the retention times of all method analytes in CCCs, LFBs and LFMs. This requirement does not preclude continuous operation by sequencing multiple analysis batches; however, the entire analysis batch is invalid if one or more analyte peaks have drifted outside of designated time windows in the CCC at the beginning or end of the analysis batch. The SIM parameters used during method development for selected analytes are provided in Table 16 as an example.

10.2.3.4. Alternating Full and SIM Scan Modes - Alternating full and SIM scan modes during a single sample acquisition is permitted if the minimum number of scans across each GC peak acquired in each mode is maintained (as specified in Sect. 10.2.3.2 and 10.2.3.3), i.e., a minimum of five scans in full scan mode and a minimum of five scans in SIM mode. If the chromatogram is divided into time windows, the laboratory must ensure that each method analyte elutes entirely within the proper window during each analysis. This can be achieved by carefully monitoring the retention times of all ISs and SURs in each sample, and carefully monitoring the

retention times of all method analytes in CCCs, LFBs and LFMs. This requirement does not preclude continuous operation by sequencing multiple analysis batches; however, the entire analysis batch is invalid if one or more analyte peaks have drifted outside of designated time windows in the CCC at the beginning or end of the analysis batch.

10.2.4. CALIBRATION SOLUTIONS - To establish a calibration range extending two orders of magnitude, prepare a set of at least six calibration standards as described in Sect. 7.2.4. The lowest concentration CAL must be at or below the MRL for each method analyte. The MRL must be confirmed using the procedure outlined in Sect. 9.2.4 after establishing the initial calibration.

Note: This method contains many analytes that vary widely with regard to instrument sensitivity. If the analytes of interest differ in response, and the CAL standards have been prepared such that all analytes are at the same concentration, more standards may be needed to obtain the minimum five CAL points for each analyte. Analytes with poor response may not be observed in the low concentration standards, and the most responsive analytes may saturate the detector at the higher concentrations. It is likely that the calibration range for all analytes will not be the same. The use of custom calibration standards with varying analyte concentrations based on their relative instrument response is a possible alternative.

10.2.5. CALIBRATION - Calibrate the GC/MS system using the internal standard technique in either full scan, SIM or alternating full scan/SIM mode. Subsequent sample analysis must be performed in the same calibration mode using identical instrument conditions and parameters. Internal standard designations and suggested QIs for all method analytes are listed in Table 1. Table 16 contains example scanning parameters for selected analytes in SIM mode. Linear or quadratic calibrations may be used. Weighting may be used at the discretion of the analyst. In general, forcing zero as part of the calibration is not recommended. However, **zero must be forced for all analytes that are routinely observed as contaminants in LRBs.** Forcing zero allows for a better estimate of the background level of contaminants in the blank. An accurate estimate of background contamination is necessary to set MRLs for method analytes when blank levels are problematic (Sect. 9.3.1). For example, phthalates are a chemical class which is typically problematic with regard to background contamination.

10.2.5.1. Toxaphene calibration - Analyze a minimum of five calibration standards of technical toxaphene. These calibrations standards must contain only toxaphene and the ISs and SURs. Select a minimum of four chromatographic peaks from the toxaphene chromatograms to use for calibration. See Fig. 2 as an example of

suggested peak selection, and Table 16 as an example of suggested SIM parameters. Create a calibration curve for each selected peak, using the total technical toxaphene concentration in each CAL standard as the calibration concentration. See Sect. 12.3.3 for utilizing the curves to calculate the concentration of toxaphene in CCCs, LFBs, Field Samples, LFSMs, QCSs, FDs, and LFSMDs.

10.2.6. CALIBRATION ACCEPTANCE CRITERIA – Validate the initial calibration curves by using the regression equations to calculate the concentration of each analyte as an unknown in each of the analyses used to generate the curves. Calibration points that are ≤ MRL must calculate to be within ± 50% of their true value. All other calibration points must calculate to be within ± 30% of their true value. If these criteria cannot be met, the analyst may eliminate either the highest or lowest point on the curve and reassess the acceptance criteria. If the acceptance criteria still cannot be met, the analyst will have difficulty meeting ongoing QC criteria. **It is highly recommended that corrective action be taken before proceeding.** This may include one or more of the following actions: analyze the calibration standards, further restrict the range of calibration, or select an alternate method of calibration. The data presented in this method were obtained using either linear regression or quadratic fits. Quadratic fit calibrations should be used with caution, because the non-linear area of the curve may not be reproducible.

10.3. CONTINUING CALIBRATION CHECK (CCC) – Analyze a CCC to verify the initial calibration at the beginning of each analysis batch, after every tenth Field Sample, and at the end of each analysis batch. The beginning CCC for each analysis batch must be at or below the MRL. This CCC verifies instrument sensitivity prior to the analysis of samples. Alternate subsequent CCCs between the remaining calibration levels.

Notes:

- If standards have been prepared such that all analytes are not in the same calibration standard (or all low CAL points are not in the same CAL standard), it may be necessary to analyze more than one CCC to meet this requirement. Alternatively, it may be cost effective to prepare or obtain a customized standard to meet this criterion.
- Separate CCCs must be analyzed for toxaphene if it is one of the analytes being measured. The frequency of analysis, the concentration rotation requirements and acceptance criteria are the same for toxaphene as for other analytes.

10.3.1. Verify that the peak area of the QI of each IS has not changed by more than ± 50% from the mean peak area measured for that IS during initial calibration. In addition, verify that the peak area of the QI of each of the ISs 1-3 are within ± 30% from the most recently analyzed CCC. If these

limits are exceeded, remedial action must be taken (Sect. 10.3.3). Control charts are useful aids in documenting system sensitivity changes.

10.3.2. Calculate the concentration of each analyte and surrogate in the CCC. The calculated amount for each analyte for medium and high level CCCs must be ± 30% of the true value. The calculated amount for the lowest calibration level for each analyte must be within ± 50% of the true value. If these criteria are not met, then all data for the problem analyte must be considered invalid, and remedial action (Sect. 10.3.3) must be taken. Recalibration may be required. Any Field Sample extracts that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. If the CCC at the end of an analysis batch fails because the calculated concentration is greater than 130% (150% for the low-level CCC) for a particular target analyte, and Field Sample extracts show no detection for that target compound, non-detects may be reported without reanalysis.

10.3.3. REMEDIAL ACTION - Failure to meet CCC QC performance criteria may require remedial action. Major maintenance such as cleaning an ion source, cleaning the mass analyzer, replacing filament assemblies, or replacing the GC column, etc., will likely require returning to the initial calibration step (Sect. 10.2).

11. PROCEDURE

11.1. This section describes the procedures for sample preparation, SPE, final extract preparation and storage, and extract analysis. Important aspects of this analytical procedure include proper preparation of laboratory glassware, sample containers (Sect. 4.1), and sample collection and storage (Sect. 8). Procedures for data analysis and calculations are described in Sect. 12.

11.2. SAMPLE PREPARATION

11.2.1. Samples are preserved, collected and stored as described in Sect. 8. All field and QC samples, including LRBs and LFBs, must contain the preservatives listed in Sect. 8.1.2. Before extraction, verify that the sample pH is ≤ 4. If the sample pH does not meet this requirement, discard the sample. If the sample pH is acceptable, proceed with the analysis. Mark the level of the sample on the outside of the sample bottle for later sample volume determination. If using weight to determine volume (Sect. 11.10), weigh the bottle and sample contents before extraction.

11.2.2. Add an aliquot of the SUR PDS(s) to each sample to be extracted. For method development work, a 10-µL aliquot of each of the 500-µg/mL SUR

PDSs (Sect. 7.2.2) was added to 1 L samples for a final concentration of 5.0 µg/L.

11.2.3. If the sample is an LFB, LFSM, or LFSMD, add the necessary amount of Analyte Fortification Solution(s) (Sect. 7.2.3.2). Swirl each sample to ensure all components are mixed.

11.2.4. Proceed with sample extraction using one of the SPE options described in Sects. 11.3-11.7.

11.3. CARTRIDGE SPE (6 mL) PROCEDURE - This cartridge extraction procedure may be carried out in a manual mode or by using a robotic or automatic sample preparation device. This section describes the SPE procedure using the equipment outlined in Sect. 6.11 in its simplest, least expensive mode without the use of a robotic system. The manual mode described below was used to collect data presented in Sect. 17. The extraction steps are written for an individual sample, but multiple samples may be extracted simultaneously depending upon the extraction equipment used.

Automated systems may use either vacuum or positive pressure to process samples and solvents through the cartridge. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the cartridge and lead to poor recoveries. Compressed gas cylinders of high purity nitrogen may be used with systems that use positive pressure for sample processing.

11.3.1. CARTRIDGE CLEANUP - Install the SPE cartridge (Oasis HLB or J.T. Baker H_2O Phobic DVB as described in Sect. 6.11.1) into the vacuum manifold. Wash the cartridge with 5 mL of EtOAc by adding the solvent to the cartridge; draw about half through the sorbent, soak for about one min, then draw the remaining solvent through the cartridge.

11.3.2. CARTRIDGE CONDITIONING - Polymeric SPE sorbents are water wettable (unlike C-18 SPE sorbents). Many manufacturers of polymeric SPE media suggest that their products do not need to be kept wet during conditioning and sample processing. However, little data have been shown

to demonstrate performance under those conditions for the wide variety of environmental contaminants using these media. Therefore, in the interest of providing a single procedure for the sorbents and analytes in this method, the authors chose to use procedures similar to those used with C-18 where the sorbent is kept wet.

11.3.2.1. CONDITIONING WITH METHANOL – Add 10 mL MeOH to the cartridge and allow it to soak for about one min. Then draw most of the MeOH through. A layer of MeOH must be left on the surface of the cartridge. Do NOT let the cartridge go dry from this point on until the end of sample extraction, otherwise recondition the cartridge with 10 mL MeOH.

11.3.2.2. CONDITIONING WITH WATER - Rinse the cartridge by adding 10 mL of reagent water to the cartridge and drawing most through, again leaving a layer on the surface of the cartridge. Do NOT let the cartridge go dry from this point on until the end of sample extraction, otherwise recondition the cartridge starting with step 11.3.2.1.

11.3.3. SAMPLE EXTRACTION - Attach a PTFE transfer line to the top of the cartridge. Insert the opposite end of the transfer line into the sample to be extracted. Apply vacuum to begin the extraction. Adjust the vacuum so that the sample passes through the cartridge at a rate of about 10 mL/min. Pass the entire sample volume through the cartridge, draining as much water from the sample container as possible. Rinse the bottle with 10 mL LRW and transfer to the cartridge under full vacuum. Rinsing the sorbent with LRW prior to drying helps remove sample preservatives from the sorbent so they are not transferred to the extract. Remove the sample transfer line from the cartridge and dry by maintaining vacuum for about 10 min.

11.3.4. CARTRIDGE ELUTION - Remove the manifold lid (but do not remove the cartridge) and insert a suitable collection tube to contain the eluent (15 mL collection vial). Reassemble the apparatus. Add 5 mL of EtOAc to the sample bottle, and rinse the inside walls thoroughly. Allow the solvent to settle to the bottom of the bottle, and then transfer to the cartridge by applying vacuum. Draw about half of the solvent through the cartridge, cut off vacuum at the cartridge, and allow the cartridge to soak for one min. Draw the remaining solvent through the cartridge. Repeat the above step with DCM. Shut off vacuum, remove the transfer line, and remove the collection vial. Proceed to Sects. 11.8 and 11.9 to dry and concentrate the extract.

11.4. CARTRIDGE SPE (83 ML) PROCEDURE - This cartridge extraction procedure may be carried out in a manual mode or by using a robotic or automatic sample

preparation device. This section describes the SPE procedure using the equipment outlined in Sect. 6.12 in its simplest, least expensive mode without the use of a robotic system. The manual mode described below was used to collect data presented in Sect. 17.

Automated systems may use either vacuum or positive pressure to process samples and solvents through the cartridge. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the cartridge and lead to poor recoveries. Compressed gas cylinders of high purity nitrogen may be used with systems that use positive pressure for sample processing.

11.4.1. CARTRIDGE CLEANUP - Assemble the extraction system by adding cartridge adaptors, UCT Universal 525 cartridges and bottle holders to the six station manifold. Wash the bottle holders and cartridges with 5 mL 1:1 EtOAc:DCM, draw half through, soak the sorbent for one min, then draw the remaining solvent through, maintaining the vacuum for two additional min.

11.4.2. CARTRIDGE CONDITIONING

11.4.2.1. CONDITIONING WITH METHANOL – Add 10 mL MeOH to each cartridge. Soak for one min. Then draw most of the MeOH through, leaving a thin layer of MeOH on the surface of the cartridge. Do NOT let the cartridge go dry from this point on until the end of sample extraction, otherwise recondition the cartridge with 10 mL MeOH.

11.4.2.2. CONDITIONING WITH WATER – Add 10 mL LRW to each cartridge. Then draw most through, leaving a thin layer on the surface of the cartridge. Do NOT let the cartridge go dry from this point on until the end of sample extraction, otherwise recondition the cartridge starting with step 11.4.2.1.

11.4.3. SAMPLE EXTRACTION - Load the sample bottles onto the bottle holders. Turn on the vacuum; adjust the flow at a fast drop-wise fashion. A flow

rate of 10 mL/min provides optimum recoveries. After passing the entire volume of each sample through the cartridges, add 10 mL LRW to the sample bottle, rinse and pass the water through the cartridges. Rinsing the sorbent with LRW prior to drying helps remove sample preservatives from the sorbent so they are not transferred to the extract. Remove the sample bottles. Dry the cartridges under full vacuum for 10 min.

11.4.4. CARTRIDGE ELUTION - Remove cartridge adaptors and insert 40-mL glass vials into the manifold to collect the eluent. Replace the cartridge adaptors, cartridges and bottle holders on the manifold. Rinse each bottle holder and cartridge with 5 mL EtOAc, draw half through the sorbent, soak one min, then draw the remaining through. Repeat with 5 mL DCM. Again, rinse each sample bottle thoroughly with 5 mL EtOAc. Then pour the rinsing solvent into the cartridge. Repeat with 5 mL DCM. Proceed to Sects. 11.8 and 11.9 to dry and concentrate the extract.

11.5. SPEEDISK SPE PROCEDURE - The Speedisk extraction procedure may be carried out in a manual mode or by using a robotic or automatic sample preparation device. This section describes the SPE procedure using the equipment outlined in Sect. 6.10 in its simplest, least expensive mode without the use of a robotics system. The manual mode described below was used to collect data presented in Sect. 17. The extraction steps are written for an individual sample, but multiple samples may be extracted simultaneously depending upon the extraction equipment used.

Note: All automated extraction systems may not be suitable for use with Speedisks. See the warning concerning excessive water content in extracts in Sect. 13.6.6.

Automated systems may use either vacuum or positive pressure to process samples and solvents through the disk. <u>All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality.</u> Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries. Compressed gas cylinders of high purity nitrogen may be used with systems that use positive pressure for sample processing.

11.5.1. SPEEDISK CLEANUP - Insert a J.T. Baker Speedisk disk (DVB H_2O Phobic, 55 mm) onto a J.T. Baker Speedisk manifold apparatus. Wash the

disk with 5 mL of EtOAc by adding the solvent to the disk, drawing about half through the disk, allowing it to soak the disk for about one min, then drawing the remaining solvent through the disk.

11.5.2. SPEEDISK CONDITIONING

11.5.2.1. CONDITIONING WITH METHANOL - Pre-wet the disk with 10 mL MeOH by adding the MeOH to the disk and allowing it to soak for about one min, 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. Note that it may be necessary to briefly remove the disk from the manifold to prevent residual vacuum from pulling the remaining MeOH through and unintentionally allowing the disk to dry out.

11.5.2.2. CONDITIONING WITH WATER - Rinse the disk with 10 mL reagent water by adding the water to the disk and drawing most through, again leaving a layer on the surface of the disk. Add a sample reservoir adaptor to the top of the Speedisk.

11.5.3. SAMPLE EXTRACTION - Add the water sample to the reservoir and apply full vacuum to begin the extraction. Particulate-free water may pass through the disk in as little as five min without reducing analyte recoveries. Pass the entire sample through the disk, draining as much water from the sample container as possible. Rinse the bottle with 10 mL LRW and transfer to the disk under full vacuum. Rinsing the sorbent with LRW prior to drying helps remove sample preservatives from the sorbent so they are not transferred to the extract. Dry the disk by maintaining vacuum for about three min.

11.5.4. SPEEDISK ELUTION - Remove the reservoir adaptor and Speedisk disk, and add the collection vial adaptor and insert a suitable collection tube to contain the eluent (40 to 60-mL collection vial). Reassemble the apparatus. Add ~2 mL of acetone to the sample bottle, and rinse the inside walls thoroughly. Allow the solvent to settle to the bottom of the bottle, then transfer it to the disk. Draw the solvent through the disk by applying vacuum. Add 5 mL of EtOAc to the sample bottle, and rinse the inside walls thoroughly. Allow the solvent to settle to the bottom of the bottle, then transfer it to the Speedisk disk. A disposable pipette or syringe may be used to do this, rinsing the sides of the glass filtration reservoir in the process. Draw about half of the solvent through the disk, release the vacuum, and allow the disk to soak for one min. Draw the remaining solvent through the disk. Repeat the above step with 5 mL DCM. Proceed to Sects 11.8 and 11.9 to dry and concentrate the extract.

11.6. EMPORE DISK SPE PROCEDURE - The disk extraction procedure may be carried out in a manual mode or by using a robotic or automatic sample preparation device. This section describes the SPE procedure using the equipment outlined in Sect. 6.9 in its simplest, least expensive mode without the use of a robotics system. The manual mode described below was used to collect data presented in Sect. 17. The extraction steps are written for an individual sample, but multiple samples may be extracted simultaneously depending upon the extraction equipment used.

Automated systems may use either vacuum or positive pressure to process samples and solvents through the disk. All sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the manual procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries. Compressed gas cylinders of high purity nitrogen may be used with systems that use positive pressure for sample processing.

11.6.1. EMPORE DISK CLEANUP - Insert the disk (3M SDB-XC, 47mm) into the filter manifold apparatus. Wash the disk with 5 mL EtOAc by adding the solvent to the disk, drawing about half through the disk, allowing it to soak the disk for about one min, then drawing the remaining solvent through the disk.

11.6.2. EMPORE DISK CONDITIONING

11.6.2.1. CONDITIONING WITH METHANOL - Pre-wet the disk with 10 mL MeOH by adding the MeOH to the disk and allowing it to soak for about one min, 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.

11.6.2.2. CONDITIONING WITH WATER - Rinse the disk with 10 mL reagent water by adding the water to the disk and drawing most through, again leaving a layer on the surface of the disk.

11.6.3. SAMPLE EXTRACTION - Add the water sample to the reservoir and apply full vacuum to begin the extraction. Particulate-free water may pass

through the disk in as little as five min without reducing analyte recoveries. Pass the entire sample through the disk, draining as much water from the sample container as possible. Rinse the bottle with 10 mL LRW and transfer to the disk under full vacuum. Rinsing the sorbent with LRW prior to drying helps remove sample preservatives from the sorbent so they are not transferred to the extract. Dry the disk by maintaining vacuum for about 10 min.

11.6.4. DISK ELUTION - Remove the filtration top (but do not disassemble the reservoir and fritted base) and insert a suitable collection tube to contain the eluent (40-mL collection vial). Reassemble the apparatus. Add 5 mL EtOAc to the sample bottle, and rinse the inside walls thoroughly. Allow the solvent to settle to the bottom of the bottle, then transfer it to the disk. A disposable pipette or syringe may be used to do this, rinsing the sides of the glass filtration reservoir in the process. Draw about half of the solvent through the disk, release the vacuum, and allow the disk to soak for one min. Draw the remaining solvent through the disk. Repeat this step with DCM. Using a syringe or disposable pipette, rinse the filtration reservoir with 5 mL 1:1 EtOAc:DCM. Draw the solvent through the disk and into the collection vial. Proceed to Sects. 11.8 and 11.9 to dry and concentrate the extract.

11.7. HORIZON ATLANTIC DVB DISK PROCEDURE - This extraction procedure may be carried out in a manual mode or by using a robotic or automatic sample preparation device. The following section describes the SPE procedure using the automated extraction equipment outlined in Sect. 6.13. The automated procedure described below was used to collect data presented in Sect. 17. The use of a manual system is allowed; however, all sorbent washing, conditioning, sample loading, rinsing, drying and elution steps must be performed as closely as possible to the automated procedure. The solvents used for washing, conditioning, and sample elution must be the same as those used in the manual procedure; however, the amount used may be increased as necessary to achieve the required data quality. Solvent amounts may not be decreased. Note that in the automated procedure each solvent "prewet" step is approximately 10 mL and each solvent elution "rinse" is 4-5 mL. Sorbent drying times prior to elution may be modified to achieve the required data quality. Caution should be exercised when increasing solvent volumes. Increased extract volume will likely necessitate the need for additional sodium sulfate drying, and extended evaporation times which may compromise data quality. Caution should also be exercised when modifying sorbent drying times. Excessive drying may cause losses due to analyte volatility, and excessive contact with room air may oxidize some method analytes. Insufficient drying may leave excessive water trapped in the disk and lead to poor recoveries. Compressed gas cylinders of high purity nitrogen may be used with systems that use positive pressure for sample processing.

Step #	Description	Solvent	Soak Time (mm:ss)	Air Dry Time (mm:ss)
1	Prewet	EtOAc	1:00	0:30
2	Prewet	DCM	1:00	0:30
3	Prewet	MeOH	1:00	0.00
4	Prewet	LRW	0.05	0.00
5	Sample Load			
6*	Wash	LRW	0:10	0:30
7*	Wash	LRW	0:10	0:30
8	Air Dry			1:00
9	Rinse	EtOAc	1:30	0:30
10	Rinse	DCM	1:30	0:30
11	Rinse	DCM	1:30	0:30

* Steps 6 and 7 of this procedure were added subsequent to the collection of the data shown in Tables 13 and 14. The procedure has been tested with these additional steps, with no negative effect on the resulting data. These steps are optional, but highly recommended. Rinsing the sorbent with LRW before air drying and solvent elution removes any residual preservatives from the sorbent so that they are not eluted into the extract.

Proceed to Sects. 11.8 and 11.9 to dry and concentrate the extract.

11.8. DRYING THE EXTRACT - Transfer the combined eluent through a drying tube containing about 10 g of anhydrous sodium sulfate. Rinse the collection tube with 5 mL DCM, and then put the DCM through the sodium sulfate. Collect the dried extract and DCM rinse in a clean collection tube (15- or 40-mL tube depending on the extract volume).

Note: Speedisk disk extracts may contain higher amounts of water due to the acetone in the elution steps. If extracts appear wet after completing Sect. 11.8 or 11.9, additional sodium sulfate may be used. If additional sodium sulfate is used the 5 mL DCM rinse may need to be increased by a proportional amount.

11.9. EXTRACT CONCENTRATION - Concentrate the extract to about 0.7 mL under a gentle stream of nitrogen gas in a warm water bath (at ~ 40 ^{o}C). Do not blow down samples to less than 0.5 mL, because the more volatile compounds will exhibit diminished recovery. Transfer the extract to a 1-mL volumetric flask and add the internal standards (Sect. 7.2.1). Rinse the collection tube that held the dried extract with small amounts of EtOAc and add to the volumetric flask to bring the volume up to the 1-mL mark. Transfer to an autosampler vial. Store extracts at -5 ºC or less until analysis.

11.10. SAMPLE VOLUME OR WEIGHT DETERMINATION - Use a graduated cylinder to measure the volume of water required to fill the original sample bottle to the mark made prior to extraction (Sect. 11.2.1). Determine volume to the nearest 10 mL for use in the final calculations of analyte concentration (Sect. 12.2). If using weight to

determine volume, reweigh the empty sample bottle. Subtract the empty bottle weight from the weight of the original combined bottle/sample weight measured in Sect. 11.2.1. To calculate the sample volume from its weight , assume a sample density of 1g/mL. Use the calculated sample volume for analyte concentration calculations in Sect.12.2.

11.11. ANALYSIS OF SAMPLE EXTRACTS

11.11.1. Establish operating conditions as described in Sect. 10.2.3. Confirm that compound separation and resolution are similar to those summarized in Table 1 and Figures 1a-1d.

11.11.2. Establish a valid initial calibration following the procedures outlined in Sect. 10.2 or confirm that the calibration is still valid by running a CCC as described in Sect. 10.3. If establishing an initial calibration for the first time, complete the IDC as described in Sect. 9.2.

11.11.3. Analyze aliquots of Field and QC Samples at appropriate frequencies (Sect. 9) with the GC/MS conditions used to acquire the initial calibration and the CCC. At the conclusion of data acquisition, use the same software that was used in the calibration procedure to tentatively identify peaks in predetermined retention time windows of interest. Use the data system software to examine the ion abundances of components of the chromatogram.

12. DATA ANALYSIS AND CALCULATIONS

12.1. COMPOUND IDENTIFICATION - Identify sample components by comparison of their retention times and mass spectra to the reference retention times and spectra in the user-created data base as follows:

12.1.1. Establish an appropriate retention time window for each analyte, internal standard and surrogate analyte to identify them in QC and Field Sample chromatograms. Ideally, the retention time window should be based on measurements of actual retention time variation for each compound in standard solutions collected on each GC/MS over the course of time. The suggested variation is plus or minus three times the standard deviation of the retention time for each compound for a series of injections. The injections from the initial calibration and from the IDC (Sect. 9.2) may be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of an appropriate retention window size.

12.1.2. Each compound should be identified from its reference spectrum obtained during the acquisition of the initial calibration curve. The mass spectrum used for identification of each compound may have been acquired in the full

scan or SIM mode. In general, all ions that are present above 30% relative abundance in the mass spectrum of the reference standard obtained during calibration should be present in the mass spectrum of the sample component and should agree within an 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%.

12.1.3. 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. Comparing a background subtracted spectrum to the reference spectrum is suggested. If two or more analytes coelute but 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.

12.1.4. Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two isomer peaks is < 25% of the average height of the two isomer peaks. Otherwise, combine the peak areas of the isomers and quantify and identify as an isomeric pair.

12.1.5. Tribufos is the oxidation product of merphos. When analyzed by GC/MS under the conditions used during method development, the retention time and mass spectrum produced by merphos is identical to that of tribufos. Therefore, when tribufos is identified as a sample component in this method, it is possible that the original contaminant may have been merphos.

12.1.6. For specific instructions on identification and quantitation of the multi-component analytes chlordane and toxaphene, and for instructions on screening for Aroclors, see Sect. 12.3.

12.2. QUANTITATION AND CALCULATIONS

12.2.1. Calculate analyte and surrogate concentrations using the multipoint calibration established in Sect. 10.2. In validating this method, concentrations were calculated by measuring the characteristic ions listed in Table 1. Other ions may be selected at the discretion of the analyst. Do not use daily continuing calibration check data to quantitate analytes in samples. Adjust the final analyte concentrations to reflect the actual sample volume determined in Sect. 11.10. Field Sample extracts that require dilution should be treated as described in Sect. 12.2.2.

12.2.2. If the calculated amount of any analyte exceeds the calibration range of the curve, the extract must be diluted with EtOAc, with the appropriate amount of additional internal standard added to match the original concentration. Analyze the diluted extract. Acceptable surrogate performance (Sect. 9.3.6) should be determined from the undiluted sample extract. Incorporate the dilution factor into final concentration calculations. The resulting sample should be documented as a dilution, and MRLs should be adjusted accordingly. If matrix-matched calibration standards are being used, the dilution may be made with EtOAc but care should be taken to dilute just enough to position the analyte within the calibration range. Excessive dilution and resulting low concentration may affect the accuracy of the final measurement.

12.2.3. Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

Note: Some data in Sect. 17 of this method are reported with more than two significant figures. This is done to better illustrate the method performance data.

12.3. MULTI-COMPONENT ANALYTES

12.3.1. Technical Chlordane - Technical chlordane is a multi-component analyte regulated under the Safe Drinking Water Act (SDWA) at the time of publication of this method. It contains at least 140 chlorinated components. At the concentrations likely to be encountered in drinking water samples, only two or three major components will typically be identified: cis- and trans-chlordane and trans-nonachlor. If one or more of these components is identified in a Field Sample, and a quantitative value for technical chlordane is required, the analyst may choose from the following options:

Option 1: Obtain a standard of technical chlordane and use it to prepare calibration curves for each major component observed in the Field Sample. Assign the concentration of technical chlordane in the calibration standard to each peak selected for quantitation. Quantitate each of the major components observed in the Field Sample from the technical chlordane calibration and report a technical chlordane amount by averaging the values found for the components.

Option 2: If trans-chlordane is present as the major chlordane component, multiply the concentration of trans-chlordane (determined from calibration with a trans-chlordane standard) by seven to obtain a value for technical chlordane. (A multiplier of seven was determined from the average weight percent of trans-chlordane in multiple standards of technical chlordane.) If

trans-chlordane is not the major chlordane component observed, Option 1 must be used.

12.3.2. Aroclor screening - Aroclors are complex mixtures of polychlorinated biphenyls (PCBs). If any of the 14 PCB congeners in the analyte list are identified in a Field Sample, presume that one or more Aroclors are present. A quantitative value for "total PCBs" can be obtained by analyzing a duplicate sample by Method 508A[23].

12.3.3. Toxaphene - Identify the toxaphene peaks selected during calibration using the procedures in Sects. 12.1.1 and 12.1.2. Use the calibration curves created in Sect. 10.2.5.1 to quantitate each toxaphene peak. If all four peaks are present, use the mean concentration of the four as the reported concentration of technical toxaphene. All four peaks must be present in calibration standards and all fortified samples, i.e., LFBs and LFSMs. If one or more, but not all of the selected component peaks are present in a Field Sample, it may be an indication that weathering has taken place. In this case, calculate the amount of technical toxaphene to be reported as the mean of ONLY the target peaks present (do not include any user-defined value, or use zero in the mean calculation to represent missing peaks). Annotate the reported technical toxaphene concentration with the range of concentrations observed for the individual target peaks.

13. METHOD PERFORMANCE

13.1. METHOD DEVELOPMENT AND PERFORMANCE DATA COLLECTION - This method was developed by USEPA's National Exposure Research Laboratory (NERL) chemists and staff from Shaw Environmental and Infrastructure, Inc. (SHAW) and Industrial and Environmental Services, LLC (IES) under contract to USEPA's Office of Ground Water and Drinking Water (OGWDW). The method was developed using the four types of SPE media described in Sects. 6.9-6.11. Most data collected during the development phase of the method were duplicated in both the NERL lab and the SHAW/IES lab by different analysts using different instrumentation. This verified that the method procedures were transferable between labs. The majority of the data presented in Sect. 17 were generated with these four types of SPE media. Subsequent to the method development, Horizon Technologies, Inc. and United Chemical Technologies (UCT) each generated data using the method procedures with additional types of SPE media (Sects. 6.12-6.13). These additional SPE products were added to the method to further extend the SPE options available for use with this method. Data demonstrating the performance of these additional sorbents are in Tables 11-14.

13.1.1. GC/MS INSTRUMENTATION – The instruments described in this section were used to collect method performance data as cross-referenced in the footnotes of the tables in Sect. 17.

13.1.1.1. Thermo DSQ quadrupole mass spectrometer equipped with a Thermo Trace Ultra GC and a Restek RXI-5sil-MS 30 m x 0.25 mm x 0.25 µm column. A 5-mm i.d. single gooseneck injection port liner with Siltek deactivation was used (Restek #20946-214). Calibration standards and sample extracts were injected in the hot, splitless mode using a pressure pulse. See Sect. 10.2.3.1 for GC oven temperature program.

13.1.1.2. Agilent 5975C MSD equipped with a 7890A GC and a Restek RXI-5sil-MS 30 m x 0.25 mm x 0.25 µm column. A 4-mm i.d. single gooseneck injection port liner with intermediate polarity deactivation was used (Restek #20798). Calibration standards and sample extracts were injected in the hot, splitless mode using a pressure pulse. See Sect. 10.2.3.1 for GC oven temperature program.

13.1.1.3. Agilent 5973 MSD equipped with a 6890 GC and a Restek RXI-5sil-MS 30 m x 0.25 mm x 0.25 µm column. A drilled Uniliner (Restek #21055) was used in the injector, installed with the hole on top. One microliter pulsed (1 min) splitless injections were made with the injector at 275 °C. The GC oven temperature program was as follows: initial oven temperature 50 °C, hold for 1.5 min, ramp a 10 °C/min to 130 °C, ramp at 2 °C/min to 142 °C, ramp at 20 °C/min to 270 °C, ramp at 7 °C/min to 320 °C, hold for 1 min.

13.1.1.4. Agilent 5975C MSD with a 6890N GC, equipped with a Restek RXI-5sil-MS 30m x 0.25 mm x 0.25 µm column. A 4-mm i.d. splitless gooseneck injection port liner (UCT#GCLGN4MM) was used, with the injection port at 250 °C. One microliter injections were made with a 1 min split delay. The GC oven temperature program was as follows: initial oven temperature of 55 °C, hold for 1 min, ramp at 10 °C/min to 200 °C, ramp at 7 °C/min to a final temperature of 320 °C, hold for 0.36 min.

13.2. PRECISION AND ACCURACY DATA

13.2.1. FULL SCAN GC/MS - Precision and accuracy data were collected from LFBs at three concentration levels using each of the six sorbents described in Sects. 6.9-6.13. Precision and accuracy data were also collected at a single fortified concentration using two challenging water matrices. Water matrices were selected to be representative of ground water with high mineral content and surface water with a moderate level of TOC. Precision and accuracy data in both fortified reagent water and fortified matrices are presented in Tables 3-14. These data were generated using matrix-matched calibration standards. Performance is similar for most analytes on most sorbents, especially considering that the data were obtained by different

analysts using different instrumentation. A notable exception is dimethipin. The data generated for dimethipin using the J.T. Baker H_2O Phobic DVB Speedisk or the Oasis HLB cartridge provided the best performance. The other sorbent options do not retain dimethipin sufficiently to meet the method acceptance criteria for recovery. If dimethipin data are to be reported, either the Oasis HLB cartridge or the J.T. Baker H_2O Phobic DVB Speedisk must be used.

13.2.2. SIM GC/MS - The SIM GC/MS analysis option may be used with extracts from any of the options described in Sects. 6.9-6.13. To demonstrate the enhanced sensitivity and overall method performance using SIM, two sorbents were selected. Waters Oasis HLB cartridges and J.T. Baker H_2O-Phobic DVB Speedisks were specifically selected so that acceptable performance data could be obtained for dimethipin, a compound of particular interest to EPA at the time of method development. Precision and accuracy data were collected by two laboratories using LFBs fortified at three concentration levels. Additional precision and accuracy data were collected by the same laboratories using LFSMs created by fortifying two challenging tap water matrices with selected analytes at either one or two concentrations. Tap water matrices were selected to be representative of ground water with high mineral content and surface water with a moderate level of TOC. Precision and accuracy data in both fortified reagent water and fortified matrices are presented in Tables 17-20. These data were generated using matrix-matched calibration standards.

13.2.3. Toxaphene data - Precision and accuracy data for toxaphene in the SIM mode was generated using five sorbent options in reagent water (Table 21) and using four sorbents in challenging drinking water matrices (Table 22). Toxaphene data were not generated in the full scan mode due to poor sensitivity relative to regulatory requirements (Sect. 1.7).

13.3. LCMRLs and DLs

13.3.1. FULL SCAN GC/MS - The values in Table 15 were generated by the NERL and SHAW/IES laboratories. Sorbents were selected based on their convenience of use by each of the laboratories. The use of these specific brands of SPE media for generating LCMRLs and DLs does not imply any specific preference or endorsement of the products. Because the data were generated in two laboratories by different personnel using different sorbent options and instrumentation, minor variations between the data sets should be expected.

13.3.2. SIM GC/MS - The values for DLs in Table 23 were generated by the NERL using Oasis HLB cartridges. Table 23 also includes two sets of LCMRL data; one generated by NERL using Oasis HLB cartridges and one generated by SHAW/IES using J.T. Baker H_2O Phobic DVB Speedisks.

These sorbents were selected because they provided acceptable performance data for dimethipin, a compound of particular interest to EPA at the time of method development.

13.4. SAMPLE STORAGE STABILITY STUDIES - Drinking water samples from a chlorinated surface source were used as a representative matrix for an analyte holding time study in aqueous solution. Replicate samples in amber bottles were preserved as described in Sect. 8, fortified with method analytes, then stored for 48 hours at 10 ºC, followed by storage at 6 ºC until analysis. Randomly selected samples were analyzed in replicate (n=4) on day 0 and at several time points up to and beyond the 14 day holding time. Data from days 0, 7 and 14 are presented in Figs. 3a-3c. These data were used to establish the 14 day aqueous holding time for most method analytes (Sect. 8.4).

Notes:

- Surrogate analytes were not stored in this study, but added at the time of extraction. The data in Figs. 3a-c for SURs are same day data, obtained for QC purposes.
- Holding time studies conducted during method development indicated significant analyte losses (more than 20% in 14 days, with increasing loss at subsequent time points) for the two compounds listed below using the sample collection, preservation and holding time procedures in this method. Dichlorvos exhibited a loss of 25% at 7 days, 39% at 14 days and 56% at 28 days. Cyanazine exhibited a loss of 16% at 7 days, 22% at 14 days, and 44% at 28 days. (All losses are relative to the day 0 measurement.) Degradation of these compounds in aqueous samples has been previously reported.[24-26]
- Toxaphene holding time data were collected in separate LFSMs. After 14 days of storage under the conditions described above, the toxaphene recovery was 105% with an RSD of 7.1%. Aqueous holding time data for toxaphene are not shown in Fig. 3.

13.5. EXTRACT STORAGE STABILITY STUDIES - Replicate sample extracts (n=4) that were stored at -5 ºC and protected from light, were analyzed on day 0, and at five additional time points up to and beyond 28 days. Data from these analyses validate the 28 day extract holding time and are presented in Figs. 4a-4c. Extract holding time data for toxaphene were collected in separate sample extracts. After 28 days of storage under the conditions described above, the recovery of toxaphene was 104% with an RSD of 3.6. Extract holding time data for toxaphene are not shown in Fig. 4.

13.6. POTENTIAL PROBLEMS/ PROBLEM COMPOUNDS

13.6.1. MATRIX INDUCED CHROMATOGRAPHIC RESPONSE ENHANCEMENT - Some method analytes have the potential to exhibit a high bias. This has been attributed to this phenomenon. Examples of this are shown in Table 20a for oxyfluorfen and tebuconazole, which have

recovery data for a fortified tap water (from a surface water source) that is > 130% of the fortified amount. Data in Table 20b show that this phenomenon is related to analyte concentration, since the high bias is not observed at the higher fortified concentration. Compounds with the potential for matrix induced chromatographic response enhancement are noted in Table 1. Although the use of matrix-matched standards will improve quantitative accuracy for these analytes, a bias may still be observed. In addition to the use of matrix matched standards, "priming" the GC system by injecting one or more sample extracts at the beginning of each analytical sequence was found to reduce matrix enhancement effects. The data presented in Tables 19, 20a and 20b were obtained using this type of priming sequence. Literature citations suggest that temperature programmed or cold injections may also reduce matrix enhancement,[12,13,27] although trials of these types of injections during method development showed little or no improvement. The biased data was instrument dependent, and not related to the sorbent used. Similar tap water extracts generated using different sorbent options listed in this method, showed similarly high biased data on the same instrument.

Depending upon the intended use of the data, the analyst should consider performing the MRL verification (Sect. 9.2.4.4) in fortified matrices similar to the samples being analyzed.

13.6.2. HIGH BACKGROUND - Phthalates and adipates commonly appear in LRBs, either from their presence in laboratory reagent water, or in chemicals and solvents used in the sample preparation process. BHT was also noted to have intermittently high background values during method development. Compounds observed to have high background values are noted in Table 1.

13.6.3. HCCPD - This compound is sometimes recovered at lower rates than other method analytes. It is known to be highly reactive, and degrades relatively quickly via photolysis and hydrolysis.[28] The performance of this compound during method development was influential in establishing lower acceptance criteria for its recovery in LFBs.

13.6.4. HCB - This compound is sometimes recovered at lower rates than other method analytes. Although there are literature references to photodegradtion of HCB[29,30], there was no direct evidence during method development that this was the cause of low recovery. The performance of this compound during method development was influential in establishing lower acceptance criteria for its recovery in LFBs.

13.6.5. PENTACHLOROPHENOL - Pentachlorophenol is prone to peak tailing and changes in response due to changes in the GC column and injector. In this method the isotopically labeled ^{13}C-pentachlorophenol has been used as

an internal standard for pentachlorophenol to minimize inaccuracies in quantitation. Assuming that there is not complete chromatographic separation between pentachlorophenol and the isotopically-labeled IS, the QI used for ^{13}C-pentachlorophenol must be *m/z* 276 (Table 1). Although this ion has low abundance, it is the only ion free of interferences from the chlorine isotope ions of pentachlorophenol.

Some GC/MS instrumentation may have too many active sites for acceptable analysis of pentachloro-phenol by this method. Alternate methods that employ derivatization procedures to methylate pentachlorophenol to its methyl ether prior to analysis are available for drinking water compliance monitoring.[31,32]

13.6.6. EXCESSIVE WATER CONTENT IN EXTRACTS – This is most often seen using the Speedisk, and is exacerbated by the use of some automated extraction devices. Excessive water will typically result in low recoveries of triazines. Not all automated extraction systems may be suitable for use with the Speedisk option.

13.6.7. OVERDRYING SPE MEDIA PRIOR TO ELUTION – If SPE media is over dried between sample loading and solvent elution by drawing excessive amounts of room air through the media, analytes that can undergo oxidation may be observed to have low recoveries. Benzo(a) pyrene is an example of an analyte that may be affected.

13.7. MULTIPLE LABORATORY DEMONSTRATION - In addition to the two laboratories in which this method was developed, the performance of this method in the SIM mode for a subset of analytes (those listed in Table 18) was demonstrated by three independent laboratories (two commercial laboratories and a drinking water utility laboratory). These laboratories produced acceptable results and provided the authors with valuable comments and method performance data. The authors wish to acknowledge the assistance of the analysts and managers at the laboratories listed below for their participation in the multi-laboratory study.

13.7.1. Dr. Yongtao Li and Mr. William Davis of Underwriters Laboratories, South Bend, IN.

13.7.2. Mr. Kevin Durk and Ms. Annmarie Walsh of Suffolk County Water Authority, Hauppauge, NY.

13.7.3. Dr. Andrew Eaton, Mr. Charles Grady, Mr. Ali Haghani and Mr. Patrick Chapman of MWH Laboratories, Monrovia, CA.

14. POLLUTION PREVENTION

14.1. This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.2. For information about pollution prevention that may be applicable to laboratory operations, consult "Less is Better: Guide to Minimizing Waste in Laboratories" available on-line from the American Chemical Society at http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf accessed November 8, 2011.

15. WASTE MANAGEMENT

15.1. The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, the Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations, and that laboratories 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, particularly the hazardous waste identification rules and land disposal restrictions.

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17. TABLES, FIGURES AND VALIDATION DATA

Table 1. Retention Times (RTs), Suggested Quantitation Ions (QIs), Suggested Confirmation Ions, and Suggested Internal Standard Reference

Peak Identification #, Figure 1	RT (min)	Internal Standards, Analytes and Surrogates	IS Ref. #	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Comments
15	13.21	acenaphthene-d_{10} (IS 1)		162	164	a
41	16.35	^{13}C-pentachlorophenol (IS 4)		276	--	b,c,d
47	16.73	phenanthrene-d_{10} (IS 2)		188	160*	a
116	24.29	chrysene-d_{12} (IS 3)		240	236*	a
1	7.24	DIMP	1	97	123	b
2	8.20	isophorone	1	82	138*	a
3	9.13	1,3-dimethyl-2-nitrobenzene (SUR)	1	77	134	a
4	9.97	dichlorvos	1	109	185*	a,d,e
5	11.21	HCCPD	1	237	235, 239	a
6	11.53	EPTC	1	128	86	a
7	12.44	mevinphos	1	127	109*, 192*	a,d,e,f
8	12.48	butylate	1	57	146, 156	a
9	12.71	vernolate	1	128	86	a
10	12.74	dimethylphthalate	1	163	77*	a
11	12.77	etridiazole	1	211	183	a
12	12.84	2,6-dinitrotoluene	1	165	63, 89	a
13	12.87	acenaphthylene	1	152	--	a
14	12.88	pebulate	1	128	57, 72	a
16	13.43	2-chlorobiphenyl	1	188	152	a
17	13.45	BHT	1	205	220	b,g
18	13.47	chloroneb	1	193	191	a
19	13.69	tebuthiuron	1	156	171	a
20	13.79	2,4-dinitrotoluene	1	165	63, 89	a
21	13.92	molinate	1	126	55	a
22	14.31	DEET	1	119	91, 190	b
23	14.44	diethylphthalate	1	149	177*	a,g
24	14.46	4-chlorobiphenyl	1	188	152	a
25	14.53	fluorene	1	165	166	a
26	14.68	propachlor	1	120	77, 176	a

Table 1. Retention Times (RTs), Suggested Quantitation Ions (QIs), Suggested Confirmation Ions, and Suggested Internal Standard Reference

Peak Identification #, Figure 1	RT (min)	Internal Standards, Analytes and Surrogates	IS Ref. #	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Comments
27	15.00	ethoprop	1	97	126, 139, 158	a,d
28	15.03	cycloate	1	83	55, 154	a
29	15.27	chlorpropham	1	213	127	a
30	15.34	trifluralin	1	264	306	a,d
31	15.66	phorate	1	75	121	b,d
32	15.75	α-HCH	1	181	109, 183, 219	a
33	15.81	2,4'-dichlorobiphenyl	1	222	152, 224	a
34	15.83	hexachlorobenzene	1	284	142, 249	a
35	16.08	atraton	2	196	169, 211	a
36	16.20	simazine	2	201	173, 186	a
37	16.21	prometon	2	225	168, 210	a
38	16.28	dimethipin	2	54	53	b
39	16.29	atrazine	2	200	215	a
40	16.30	β-HCH	2	181	109, 183, 219	a
42	16.35	pentachlorophenol	4	266	264, 268	a,c,d
43	16.36	propazine	2	214	58, 229	a
44	16.46	γ-HCH (lindane)	2	183	109, 181, 219	a,e
45	16.66	pronamide	2	173	145	a
46	16.69	2,2',5-trichlorobiphenyl	2	256	186	a
48	16.79	phenanthrene	2	178	152*	a
49	16.81	chlorothalonil	2	266	264, 268	a,e
50	16.91	disulfoton	2	88	61*, 97*	a
51	16.91	anthracene	2	178	--	a
52	17.01	terbacil	2	117	161	a,d
53	17.06	δ-HCH	2	181	109, 183, 219	a
54	17.53	phosphamidon	2	127	72, 264	b,d
55	17.68	acetochlor	2	146	59, 162, 223	a
56	17.71	metribuzin	2	198	--	a
57	17.73	2,4,4'-trichlorobiphenyl	2	186	256	a,e
58	17.79	vinclozolin	2	212	124, 285	b,e

Table 1. Retention Times (RTs), Suggested Quantitation Ions (QIs), Suggested Confirmation Ions, and Suggested Internal Standard Reference

Peak Identification #, Figure 1	RT (min)	Internal Standards, Analytes and Surrogates	IS Ref. #	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Comments
59	17.84	methyl parathion	2	109	125, 263	b,d
60	17.87	alachlor	2	188	160	a
61	17.96	simetryn	2	213	155, 170	a
62	18.04	ametryn	2	227	212	a
63	18.05	heptachlor	2	100	272, 274	a
64	18.10	prometryn	2	241	184, 226	a
65	18.39	terbutryn	2	226	170, 185, 241	a
66	18.47	2,2',5,5'-tetrachlorobiphenyl	2	220	290, 292	a
67	18.53	dibutyl phthalate	2	149	--	a
68	18.56	bromacil	2	205, 207	207, 205	a
69	18.72	metolachlor	2	162	238	a
70	18.76	chlorpyrifos	2	97	197, 199	a,e
71	18.84	aldrin	2	66	79, 263	a
72	18.87	cyanazine	2	225	68, 172, 198	a
73	18.87	dacthal (DCPA)	2	301	332	a
74	18.91	2,2',3,5'-tetrachlorobiphenyl	2	220	255, 292	a
75	18.92	ethyl parathion	2	109	97, 291	b,d
76	19.01	triadimefon	2	208	57	a,d,e
77	19.27	diphenamid	2	72	167, 239*	a
78	19.30	MGK 264(a)	2	164	66, 111	a,h
79	19.59	MGK 264(b)	2	164	66, 111	a,h
80	19.72	heptachlor epoxide	2	353	81, 355	a,e
81	19.74	chlorfenvinphos	2	267	269, 323	b,d
82	19.85	2,3',4',5-tetrachlorobiphenyl	2	220	110, 292	a
83	20.27	trans-chlordane	2	375	237, 272	a
84	20.38	tetrachlorvinphos	2	109	329, 331	a,d
85	20.46	butachlor	2	176	57, 160	a,d
86	20.54	pyrene	2	202	--	a
87	20.58	cis-chlordane	2	375	373, 377	a
88	20.59	endosulfan I	2	241	195, 207	a

Table 1. Retention Times (RTs), Suggested Quantitation Ions (QIs), Suggested Confirmation Ions, and Suggested Internal Standard Reference

Peak Identification #, Figure 1	RT (min)	Internal Standards, Analytes and Surrogates	IS Ref. #	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Comments
89	20.65	trans-nonachlor	2	409	407, 411	a
90	20.76	napropamide	2	72	100, 128	a,d
91	21.00	profenofos	2	339	97, 139, 208	b,d
92	21.12	4,4'-DDE	2	246	176, 318	a
93	21.17	tribufos (+merphos)	2	57	169	b,d,i
94	21.23	dieldrin	2	79	81	a,e
95	21.25	oxyfluorfen	2	252	63, 361	b,d
96	21.26	2,3,3',4',6-pentachlorobiphenyl	2	326	184, 254	a
97	21.69	nitrofen	2	283	139, 202	b,d
98	21.72	endrin	2	263	81, 281	a
99	21.81	2,2',3,4',5',6-hexachlorobiphenyl	2	360	218, 290	a
100	21.90	chlorobenzilate	2	251	111, 139	a
101	21.91	2,3',4,4',5-pentachlorobiphenyl	2	326	184, 254	a
102	21.99	endosulfan II	2	195	207, 241	a
103	22.11	4,4'-DDD	2	235	165	a,d
104	22.11	ethion	2	231	97, 153	b,d,e
105	22.40	2,2',4,4',5,5'-hexachlorobiphenyl	2	360	218, 290	a
106	22.82	norflurazon	2	145	102, 303	a,d
107	22.91	butylbenzylphthalate	2	149	91, 206	a,g
108	22.91	endosulfan sulfate	2	272	237, 387	a,e
109	23.03	4,4'-DDT	2	235	165	a,e
110	23.06	2,2',3,4,4',5'-hexachlorobiphenyl	2	360	218, 290	a
111	23.17	hexazinone	2	171	83*	a,d
112	23.38	di(2-ethylhexyl)adipate	3	129	57, 70	a,g
113	23.39	tebuconazole	3	125	83, 250	b,d,e
114	23.48	triphenyl phosphate (SUR)	3	77	169, 326	a,e
115	24.26	benzo[*a*]anthracene	3	228	226*	a
117	24.37	chrysene	3	228	226*	a
118	24.40	methoxychlor	3	227	--	a,d
119	24.67	2,2',3,4,4',5,5'-heptachlorobiphenyl	3	394	252, 324	a

Table 1. Retention Times (RTs), Suggested Quantitation Ions (QIs), Suggested Confirmation Ions, and Suggested Internal Standard Reference

Peak Identification #, Figure 1	RT (min)	Internal Standards, Analytes and Surrogates	IS Ref. #	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Comments
120	24.93	di(2-ethylhexyl)phthalate	3	149	167	a,g
121	25.79	fenarimol	3	107	139, 219	a
122	26.64	cis-permethrin	3	183	163*	a,d,e
123	26.82	trans-permethrin	3	183	163*	a,d,e
124	27.49	benzo[*b*]fluoranthene	3	252	126*	a
125	27.54	benzo[*k*]fluoranthene	3	252	126*	a
126	28.22	benzo[*a*]pyrene-d_{12} (SUR)	3	264	132*	a
127	28.28	benzo[*a*]pyrene	3	252	126*	a
128	28.50	fluridone	3	328	329	a,c,d
129	30.72	indeno[1,2,3-*c,d*]pyrene	3	276	138*	a
130	30.80	dibenzo[*a,h*]anthracene	3	278	139*	a
131	31.20	benzo[*g,h,i*]perylene	3	276	138*	a

* Confirmation ions may be at or below 30% relative abundance depending on instrument tune.
-- Compounds without values do not have qualifier ions of significant relative abundance.

Comment Key

a. PDS solvent, acetone
b. PDS solvent, methanol
c. May exhibit peak tailing
d. Potential to exhibit matrix induced chromatographic response enhancement was observed during method development. This assessment was done based on a peak area comparison of a standard prepared in solvent compared to a matrix-matched standard, both at a concentration of 0.2 ng/µL. If the matrix-matched standard area was ≥ 130% of the solvent prepared standard, the analyte was flagged as having the potential for matrix induced chromatographic response enhancement.
e. Literature references 11 and 12 indicate that this compound may exhibit matrix induced response enhancement.
f. Mevinphos exists as two isomers, cis and trans that will partially or completely coelute in this method. Partial coelution results in distorted peak shape.
g. Commonly occurs in LRBs
h. MGK 264 consists of two isomers, endo and exo. In this method, a standard of mixed isomers was used. The resulting chromatographic peaks have been designated by the authors as (a) and (b) throughout this document, where (a) is the first eluting isomer and (b) is the second eluting isomer. The amounts of each isomer listed in subsequent tables are based on the relative peak areas of the two isomers. Users of this method who need to specifically identify each isomer, must obtain and analyze standards of each isomer.
i. Tribufos + merphos. Some fortified samples were fortified with tribufos and merphos together because of the configuration of standards used. Subsequent analysis of individual standards of merphos and tribufos showed an identical retention time, mass spectrum and response for each compound when analyzed at the same concentration. The mass spectrum observed matched the NIST MS library for tribufos.

Table 2. Ion Abundance Criteria for Decafluorotriphenyl Phosphine (DFTPP)[a]

Mass (*m/z*)	Relative Abundance Criteria	Purpose of Checkpoint[b]
68	< 2% of *m/z* 69	Low-mass resolution
69	present	Low-mass resolution
70	< 2% of *m/z* 69	Low-mass resolution
197	< 2% of *m/z* 198	Mid-mass resolution
198	present [c]	Mid-mass resolution and sensitivity
199	5-9% of *m/z* 198	Mid-mass resolution and isotope ratio
365	>1% of base peak	Baseline threshold
441	< 150% of *m/z* 443	High-mass resolution
442	present [c]	High-mass resolution and sensitivity
443	15-24% of *m/z* 442	High-mass resolution and isotope ratio

a. These ion abundance criteria have been developed specifically for target compound analysis as described in this method. Adherence to these criteria may not produce spectra suitable for identifying unknowns by searching commercial mass spectral libraries. If the analyst intends to use data generated with this method to identify unknowns, adherence to stricter DFTPP criteria as published in previous methods[22] is recommended.

b. All ions are used primarily to check the mass 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, followed by the correct setting of the baseline threshold, as indicated by the presence of *m/z* 365.

c. Either *m/z* 198 or 442 is typically the base peak.

Table 3. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	87.1	2.8	95.8	2.2	88.7	1.7
acetochlor	102	4.6	108	6.8	95.2	2.0
alachlor	106	3.6	107	5.6	102	3.0
aldrin	96.2	2.0	87.1	6.2	86.4	2.0
ametryn	105	4.6	102	7.1	95.0	3.0
anthracene	90.1	4.6	98.5	2.7	98.4	0.66
atraton	108	11	109	6.6	99.9	1.3
atrazine	103	1.3	106	7.2	102	1.1
benzo[*a*]anthracene	90.9	7.0	104	2.8	89.2	0.75
benzo[*a*]pyrene	95.4	5.5	96.4	5.7	89.0	5.1
benzo[*b*]fluoranthene	98.2	3.1	96.8	3.8	90.8	2.4
benzo[*g,h,i*]perylene	97.0	13	94.9	5.6	90.3	5.9
benzo[*k*]fluoranthene	103	5.6	102	4.5	92.8	3.3
BHT	ND[e]		ND[e]		92.8	1.3
bromacil	119	2.1	103	12	99.5	1.4
butachlor	106	4.8	106	3.5	94.5	2.5
butylate	91.2	4.2	104	6.0	88.6	1.9
butylbenzylphthalate	105	5.9	123	7.4	92.9	2.2
chlordane, cis	99.1	2.9	105	3.7	99.8	0.86
chlordane, trans	91.7	1.4	99.3	3.6	105	3.8
chlorfenvinphos	101	6.8	105	3.9	108	2.6
chlorobenzilate	109	1.1	109	4.5	96.8	2.0
chloroneb	91.8	5.5	97.5	5.0	94.9	2.4
chlorothalonil	102	2.7	101	2.9	106	2.6
chlorpropham	119	11	105	5.6	92.7	2.0
chlorpyrifos	112	13	99.8	6.3	94.4	2.3
chrysene	95.6	8.3	107	2.3	89.8	0.39
cyanazine	107	9.4	111	6.3	95.5	1.0

Table 3. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
cycloate	90.4	2.7	99.6	5.0	90.7	1.8
dacthal (DCPA)	91.5	7.3	103	4.4	101	1.5
DDD, 4,4′-	101	6.0	103	5.7	92.5	0.70
DDE, 4,4′-	98.1	1.2	100	5.9	95.0	2.4
DDT, 4,4′-	106	5.0	104	6.1	95.7	0.80
DEET	102	4.1	101	2.9	94.2	0.81
di(2-ethylhexyl)adipate	106	5.2	104	3.2	94.9	3.9
di(2-ethylhexyl)phthalate	144	27	112	4.6	91.3	1.8
dibenzo[*a*,*h*]anthracene	97.3	17	83.9	11	98.6	5.8
dibutyl phthalate	81.0	11	119	6.8	104	2.8
dichlorvos	101	9.2	98.3	5.9	91.2	2.5
dieldrin	97	2.0	100	5.0	91.6	2.1
diethylphthalate	108	7.2	104	2.6	94.2	1.4
dimethipin	107	15	95.2	5.5	61.1	7.3
dimethylphthalate	99.3	3.0	101	3.6	95.5	1.1
DIMP	104	5.3	96.2	1.6	90.6	2.1
dinitrotoluene, 2,4-	109	6.7	96.8	4.4	91.1	0.59
dinitrotoluene, 2,6-	ND[f]		110	5.2	90.7	2.4
diphenamid	104	4.4	109	6.6	93.0	1.4
disulfoton	85.2	1.3	85.6	4.9	89.3	1.9
endosulfan I	101	13	101	6.0	97.9	1.7
endosulfan II	106	7.0	113	9.6	93.9	0.70
endosulfan sulfate	98.7	6.2	106	6.2	96.5	1.5
endrin	94.7	14	107	5.9	95.8	3.2
EPTC	87.9	1.8	99.8	6.9	91.1	1.5
ethion	103	6.0	104	3.6	93.0	0.59
ethoprop	109	21	103	8.6	92.1	2.1
ethyl parathion	101	16	89.8	3.8	95.8	1.6

Table 3. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
etridiazole	97.7	4.6	96.0	4.7	93.7	0.41
fenarimol	108	14	112	7.5	95.6	1.2
fluorene	92.6	3.2	97.1	2.2	88.5	1.8
fluridone	116	2.6	95.9	11	122	3.5
HCCPD	61.7	12	80.6	4.2	79.4	1.4
HCH, α	101	1.9	101	6.1	93.8	2.7
HCH, β	103	4.4	106	4.5	98.9	1.4
HCH, δ	104	8.3	104	6.0	98.0	2.1
HCH, γ (lindane)	96.7	3.6	104	5.6	98.7	4.0
heptachlor	83.5	5.0	108	9.7	92.1	1.2
heptachlor epoxide	106	6.5	104	4.7	99.9	1.9
hexachlorobenzene	59.5	15	88.1	2.8	86.5	1.6
hexazinone	107	10	113	6.8	94.4	1.3
indeno[1,2,3-*c*,*d*]pyrene	101	23	90.8	12	84.9	7.1
isophorone	97.4	3.1	99.7	4.0	90.0	1.2
methoxychlor	100	6.9	108	3.7	98.0	1.1
methyl parathion	97.9	11	98.6	5.8	97.4	0.07
metolachlor	93.4	1.7	104	5.0	97.5	1.2
metribuzin	104	2.6	96.3	6.7	98.9	2.6
mevinphos	103	8.5	95.5	5.8	90.3	2.5
MGK 264(a)	95.7	11	104	5.4	98.6	2.5
MGK 264(b)	99	5.8	103	9.0	98.6	5.5
molinate	84.8	11	99.0	5.2	83.8	2.4
napropamide	103	3.2	109	6.8	92.1	1.3
nitrofen	102	13	102	3.0	92.7	1.6
nonachlor, trans	91.4	11	104	5.4	102	0.55
norflurazon	103	9.2	101	8.7	92.3	1.3
oxyfluorfen	106	2.6	107	2.9	91.6	0.23

Table 3. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
pebulate	92.2	2.7	101	4.3	92.7	1.1
pentachlorophenol	92.9	5.6	85.8	5.5	93.9	2.7
permethrin, cis	101	5.4	109	5.2	88.5	3.2
permethrin, trans	100	5.1	101	4.5	90.5	1.6
phenanthrene	84.2	7.7	101	2.2	95.6	1.1
phorate	95.8	5.3	89.8	2.4	91.0	1.5
phosphamidon	112	7.8	112	4.2	97.8	2.0
profenofos	104	16	109	7.1	99.7	0.92
prometon	93.5	13	110	7.5	92.4	2.1
prometryn	109	8.7	104	6.7	101	3.6
pronamide	102	2.4	104	6.8	100	1.9
propachlor	102	7.6	103	4.4	92.9	1.2
propazine	99.6	5.1	107	4.4	99.2	2.8
pyrene	95.7	2.6	102	2.4	93.6	1.3
simazine	96	1.2	84.8	5.2	100	2.7
simetryn	107	4.7	107	6.7	107	1.3
tebuconazole	105	1.6	107	5.0	96.1	0.87
tebuthiuron	105	4.0	109	5.0	96.8	2.1
terbacil	95.9	6.2	104	7.1	98.7	3.4
terbutryn	111	4.6	107	5.8	100	1.4
tetrachlorvinphos	109	6.3	104	7.8	94.8	2.4
triadimefon	99.6	7.5	110	6.8	98.0	2.7
tribufos+merphos	111	2.8	105	4.2	99.1	1.3
trifluralin	94.4	1.1	104	5.2	90.7	0.72
vernolate	95.3	1.6	96.5	5.1	92.3	0.82
vinclozolin	94.9	7.3	99.7	4.7	96.9	2.2
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	83.4	3.2	92.6	5.4	88.6	1.5

Table 3. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
4-chlorobiphenyl (3)	82.4	3.0	94.0	4.4	86.2	1.0
2,4′-dichlorobiphenyl (8)	78.5	3.5	92.2	4.7	90.8	2.1
2,2′,5-trichlorobiphenyl (18)	83.4	11	94.1	4.2	92.5	2.8
2,4,4′-trichlorobiphenyl (28)	83.2	6.0	93.6	6.9	90.3	1.1
2,2′,3,5′-tetrachlorobiphenyl (44)	91.2	5.3	99.4	4.3	94.6	1.8
2,2′,5,5′-tetrachlorobiphenyl (52)	84.3	4.4	102	3.7	94.5	1.5
2,3′,4′,5-tetrachloroobiphenyl (70)	89.9	7.3	98.5	5.2	95.0	0.73
2,3,3′,4′,6-pentachlorobiphenyl (110)	98.2	6.7	98.1	4.3	94.6	0.60
2,3′,4,4′,5-pentachlorobiphenyl (118)	92.5	5.1	99.3	4.4	96.2	1.5
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	94.5	4.0	98.7	5.0	95.1	1.3
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	96.1	5.8	103	4.5	97.6	0.74
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	92.2	0.9	101	4.3	99.1	2.0
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	88.5	4.1	107	3.6	100	3.0
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	91.5	3.7	94.0	2.5	91.3	1.7
benzo[*a*]pyrene-d_{12}	101	4.7	86.5	3.5	91.2	3.0
triphenyl phosphate	102	2.8	105	3.2	88.3	1.8

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, tebuconazole is 1.2 µg/L, c-permethrin is 0.14 µg/L, t-permethrin is 0.36 µg/L, MGK 264(a) is 0.20 µg/L and MGK 264(b) is 0.050 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, tebuconazole is 10.0 µg/L, c-permethrin is 1.1µg/L, t-permethrin is 2.9µg/L, and MGK 264(a) is 1.6 µg/L and MGK 264(b) is 0.40 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, tebuconazole is 25.0 µg/L, c-permethrin is 2.8 µg/L, t-permethrin is 7.2 µg/L, and MGK 264(a) is 4.0µg/L and MGK 264(b) is 1.0 µg/L.
e. ND = Not determined. Analyte could not be determined because of high laboratory reagent blank values.
f. ND = Not determined. Analyte could not be determined because of the low concentration.

Table 4. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	90.5	1.7	99.3	2.5
acetochlor	2.0	102	3.8	128	2.3
alachlor	2.0	104	6.2	115	3.8
aldrin	2.0	80.3	3.2	91.6	2.0
ametryn	2.0	91.8	6.7	97.8	4.7
anthracene	2.0	96.5	1.3	101	3.3
atraton	2.0	96.5	1.8	94.1	4.7
atrazine	2.0	100	2.2	106	3.6
benzo[*a*]anthracene	2.0	99.1	4.0	104	3.7
benzo[*a*]pyrene	2.0	91.1	4.2	99.9	1.2
benzo[*b*]fluoranthene	2.0	93.9	3.3	105	3.4
benzo[*g,h,i*]perylene	2.0	95.3	5.9	113	2.9
benzo[*k*]fluoranthene	2.0	97.2	2.7	96.7	4.6
BHT	2.0	ND[e]		ND [d]	
bromacil	2.0	89.4	2.3	108	2.9
butachlor	2.0	102	4.0	117	4.8
butylate	2.0	101	1.9	114	2.6
butylbenzylphthalate	2.0	110	4.7	99.8	4.0
chlordane, cis-	2.0	96.9	3.9	112	2.4
chlordane, trans	2.0	92.0	4.0	106	3.3
chlorfenvinphos	2.0	104	3.3	102	3.6
chlorobenzilate	2.0	105	4.1	116	4.0
chloroneb	2.0	87.9	3.7	95.5	2.3
chlorothalonil	2.0	98.1	3.4	108	2.2
chlorpropham	2.0	100	2.3	120	5.2
chlorpyrifos	2.0	97.7	3.1	110	3.7
chrysene	2.0	102	3.1	104	3.1
cyanazine	2.0	103	2.7	102	1.8
cycloate	2.0	92.9	2.2	105	2.0

Table 4. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
dacthal (DCPA)	2.0	98.7	2.8	108	2.7
DDD, 4,4′-	2.0	95.2	2.8	105	2.9
DDE, 4,4′-	2.0	93.7	3.5	104	2.7
DDT, 4,4′-	2.0	102	4.5	117	2.2
DEET	2.0	103	1.7	103	2.4
di(2-ethylhexyl)adipate	2.0	101	2.9	106	3.8
di(2-ethylhexyl)phthalate	2.0	101	3.7	99.8	6.3
dibenzo[*a*,*h*]anthracene	2.0	85.6	5.8	102	2.4
dibutyl phthalate	2.0	114	4.5	109	8.5
dichlorvos	2.0	94.3	1.6	118	2.2
dieldrin	2.0	96.0	3.0	101	3.8
diethylphthalate	2.0	90.5	1.2	102	2.5
dimethipin	2.0	94.9	6.7	85.3	2.9
dimethylphthalate	2.0	97.7	2.7	108	2.5
DIMP	2.0	93.4	2.0	98.4	4.5
dinitrotoluene, 2,4-	2.0	100	0.40	115	2.9
dinitrotoluene, 2,6-	2.0	103	2.6	124	3.8
diphenamid	2.0	102	3.1	109	4.1
disulfoton	2.0	73.0	5.1	82.6	5.9
endosulfan I	2.0	99.4	3.2	109	3.2
endosulfan II	2.0	106	2.8	108	4.0
endosulfan sulfate	2.0	94.3	9.1	102	2.9
endrin	2.0	107	5.2	118	2.0
EPTC	2.0	96.1	1.2	110	2.6
ethion	2.0	104	3.7	105	2.4
ethoprop	2.0	97.9	2.4	122	2.3
ethyl parathion	2.0	87.1	4.0	93.8	10
etridiazole	2.0	98.8	2.0	121	4.1
fenarimol	2.0	101	3.0	112	4.1

Table 4. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
fluorene	2.0	94.9	1.1	100	3.8
fluridone	2.0	88.8	3.9	116	4.4
HCCPD	2.0	99.1	2.8	111	4.3
HCH, α	2.0	96.3	2.6	111	2.9
HCH, β	2.0	86.8	2.6	110	2.8
HCH, δ	2.0	93.8	1.7	95.1	0.6
HCH, γ (lindane)	2.0	98.6	2.7	111	7.2
heptachlor	2.0	105	4.3	113	2.6
heptachlor epoxide	2.0	100	1.7	110	1.8
hexachlorobenzene	2.0	81.5	2.6	95.3	3.0
hexazinone	2.0	98.5	3.6	108	1.9
indeno[1,2,3-*c,d*]pyrene	2.0	96.0	4.8	111	1.1
isophorone	2.0	92.4	1.2	106	3.2
methoxychlor	2.0	102	1.8	120	4.4
methyl parathion	2.0	102	3.3	108	4.2
metolachlor	2.0	102	1.6	114	2.7
metribuzin	2.0	89.4	5.9	92.1	6.2
mevinphos	2.0	94.9	3.2	116	2.9
MGK 264(a)	1.6	98.1	3.0	110	2.7
MGK 264(b)	0.4	97.8	4.0	110	3.6
molinate	2.0	91.3	3.5	107	4.7
napropamide	2.0	103	1.3	112	3.3
nitrofen	2.0	96.8	3.5	102	3.4
nonachlor, trans	2.0	96.7	4.2	107	3.6
norflurazon	2.0	92.5	4.6	114	1.7
oxyfluorfen	2.0	106	4.1	112	3.0
pebulate	2.0	95.9	2.3	109	1.9
pentachlorophenol	8.0	81.9	13	96.3	7.3
permethrin, trans	1.1	95.8	3.0	107	2.9

Table 4. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
permethrin, trans	2.9	93.2	4.3	104	3.3
phenanthrene	2.0	97.0	2.5	99.6	3.7
phorate	2.0	92.5	2.3	96.9	1.5
phosphamidon	2.0	108	5.6	103	3.4
profenofos	2.0	105	2.5	110	4.3
prometon	2.0	94.7	1.9	88.3	5.5
prometryn	2.0	96.9	3.2	100	3.7
pronamide	2.0	101	2.8	114	2.8
propachlor	2.0	99.5	1.9	114	2.7
propazine	2.0	101	2.0	105	3.5
pyrene	2.0	97.1	4.3	100	3.2
simazine	2.0	78.9	2.0	79.9	2.9
simetryn	2.0	96.9	3.2	99.8	2.0
tebuconazole	10.0	112	1.1	107	1.2
tebuthiuron	2.0	105	2.5	106	3.3
terbacil	2.0	108	3.9	121	6.5
terbutryn	2.0	101	4.5	106	4.7
tetrachlorvinphos	2.0	102	4.2	110	4.4
triadimefon	2.0	104	3.1	109	3.7
tribufos+merphos	2.0	105	2.4	110	1.9
trifluralin	2.0	101	2.4	116	3.2
vernolate	2.0	91.3	0.8	106	2.6
vinclozolin	2.0	98.8	2.9	99.5	2.9
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	85.8	1.2	97.0	2.3
4-chlorobiphenyl (3)	2.0	96.0	1.7	99.5	4.4
2,4-dichlorobiphenyl (8)	2.0	87.8	2.3	96.6	3.3
2,2′,5-trichlorobiphenyl (18)	2.0	86.2	3.5	94.8	5.2
2,4,4′-trichlorobiphenyl (28)	2.0	87.8	7.4	100	2.8

Table 4. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	93.6	3.4	105	2.7
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	104	4.7	103	3.7
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	92.8	1.8	103	2.1
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	93.2	2.6	102	2.0
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	91.2	3.7	102	4.4
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	88.2	6.7	97.5	4.2
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	95.7	0.8	106	4.2
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	93.1	5.2	103	3.1
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	97.7	1.8	107	2.5
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene (SUR)	5.0	92.3	1.7	97.9	2.8
benzo[*a*]pyrene-d_{12}	5.0	88.4	3.2	88.0	3.9
triphenyl phosphate (SUR)	5.0	108	2.5	103	2.7

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 290 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.8 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.
e. ND= Not determined. Analyte could not be determined because of high laboratory reagent blank values.

Table 5. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	84.9	1.9	88.2	2.4	90.7	1.8
acetochlor	102	4.0	94.7	1.9	97.0	3.0
alachlor	96.0	5.0	94.6	2.2	103	3.2
aldrin	95.6	2.5	88.3	7.1	96.5	4.5
ametryn	97.6	3.2	95.7	3.9	96.0	4.1
anthracene	89.4	3.2	91.1	2.9	104	2.7
atraton	107	12	101	3.3	97.9	2.5
atrazine	100	6.7	99.3	1.0	100	3.4
benzo[*a*]anthracene	89.1	0.43	95.8	2.9	92.4	1.9
benzo[*a*]pyrene	89.9	11	89.7	5.2	100	3.5
benzo[*b*]fluoranthene	94.1	2.6	97.6	3.8	96.6	0.81
benzo[*g,h,i*]perylene	89.9	10	92.8	9.0	98.7	5.1
benzo[*k*]fluoranthene	92.5	7.4	92.9	3.9	99.7	1.6
BHT	81.7	11	86.0	3.2	86.0	0.53
bromacil	104	8.2	97.5	17	97.7	2.9
butachlor	103	2.1	92.0	2.2	99.6	1.3
butylate	91.0	2.6	95.5	1.6	92.7	0.59
butylbenzylphthalate	103	5.8	96.5	3.1	101	4.3
chlordane, cis	97.2	5.2	97.9	2.6	103	2.2
chlordane, trans	93.7	5.3	106	2.3	105	1.2
chlorfenvinphos	92.7	5.3	99.1	4.6	98.9	3.3
chlorobenzilate	99.9	4.7	98.2	3.7	102	3.8
chloroneb	92.9	2.9	95.9	2.5	95.2	1.9
chlorothalonil	90.9	5.6	97.6	2.8	99.5	3.2
chlorpropham	97.9	12	98.9	2.9	93.3	2.2
chlorpyrifos	92.2	13	94.7	2.2	99.5	4.4
chrysene	90.4	7.9	96.2	3.8	94.7	1.6
cyanazine	93.2	6.4	19.2	3.8	99.1	3.7

Table 5. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
cycloate	86.1	3.4	93.8	1.7	94.6	1.9
dacthal (DCPA)	91.2	8.3	100	3.0	99.6	2.8
DDD, 4,4′-	98.4	1.2	98.1	2.9	98.5	3.2
DDE, 4,4′-	90.9	4.9	93.4	2.5	103	2.8
DDT, 4,4′-	99.7	4.0	99.1	2.0	98.7	2.8
DEET	95.9	4.8	97.9	2.1	93.9	2.2
di(2-ethylhexyl)adipate	96.2	7.8	101	6.2	95.2	1.3
di(2-ethylhexyl)phthalate	ND[e]		106	9.1	92.5	3.0
dibenzo[*a*,*h*]anthracene	85.8	14	89.7	10	101	5.6
dibutylphthalate	94.7	32	105	4.2	102	3.3
dichlorvos	94.4	5.0	101	2.0	93.8	4.9
dieldrin	94.8	7.8	98.2	3.6	101	1.2
diethylphthalate	88.4	5.5	98.6	1.4	94.1	0.60
dimethipin	85.1	17	36.8	6.5	25.2	1.8
dimethylphthalate	97.6	6.7	96.8	1.8	95.1	2.6
DIMP	98.1	2.5	92.7	3.3	91.7	2.1
dinitrotoluene, 2,4-	ND		97.9	4.1	92.1	1.3
dinitrotoluene, 2,6-	ND		95.7	1.7	92.1	3.1
diphenamid	101	4.4	95.3	3.4	98.5	3.2
disulfoton	90.7	5.5	73.9	6.0	94.2	5.1
endosulfan I	98.5	2.6	95.3	4.2	100	2.5
endosulfan II	98.8	4.2	91.6	4.9	104	2.4
endosulfan sulfate	98.2	7.5	97.6	4.4	99.7	4.6
endrin	86.8	8.6	101	4.8	101	4.5
EPTC	88.2	5.7	96.5	2.6	92.8	1.3
ethion	97.5	7.0	94.0	3.0	97.4	3.8
ethoprop	85.9	4.2	98.6	3.1	93.3	3.3
ethyl parathion	79.2	14	99.9	2.4	96.1	3.0

Table 5. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
etridiazole	97.8	4.0	93.6	2.3	93.7	0.94
fenarimol	101	3.1	94.4	3.4	96.0	5.6
fluorene	84.0	4.0	92.2	1.7	93.3	0.71
fluridone	110	13	98.2	6.1	104	1.4
HCCPD	58.3	13	75.0	3.5	80.4	2.0
HCH, α	95.3	4.5	101	1.7	100	2.7
HCH, β	98.7	4.6	100	2.8	101	3.4
HCH, δ	98.0	2.6	97.5	2.8	105	2.2
HCH, γ (lindane)	98.1	5.6	97.4	3.2	101	1.7
heptachlor	85.6	4.6	89.2	3.6	95.5	2.3
heptachlor epoxide	97.5	3.2	99.2	3.5	102	0.66
hexachlorobenzene	68.1	5.9	90.0	3.3	95.6	0.80
hexazinone	98.8	9.7	96.4	3.2	99.3	2.0
indeno[1,2,3-*c*,*d*]pyrene	102	17	91.8	9.3	96.1	3.8
isophorone	90.6	2.0	93.1	2.3	93.7	1.6
methoxychlor	96.8	6.5	102	3.0	95.1	1.3
methyl parathion	93.8	8.3	98.2	3.2	95.6	4.0
metolachlor	91.1	6.4	95.3	2.0	99.6	2.4
metribuzin	110	3.2	87.4	5.6	97.2	3.8
mevinphos	94.3	10	97.7	1.4	91.3	1.7
MGK 264(a)	100	5.2	94.1	2.9	105	5.5
MGK 264(b)	92.5	4.4	93.9	8.0	104	5.3
molinate	77.2	9.8	88.8	2.1	92.1	2.7
napropamide	91.0	5.3	94.2	2.3	97.4	2.6
nitrofen	90.2	9.1	98.1	4.0	94.9	3.7
nonachlor, trans	84.5	3.7	98.0	4.2	102	2.7
norflurazon	94.1	10	93.0	4.8	99.9	4.3
oxyfluorfen	89.9	8.3	99.7	1.9	96.7	2.2

Table 5. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
pebulate	90.3	3.3	92.9	2.2	92.5	2.3
pentachlorophenol	85.8	6.0	95.4	2.5	98.5	4.5
permethrin, cis	91.6	4.4	99.3	3.2	91.2	2.8
permethrin, trans	93.6	3.8	92.8	3.2	92.2	1.7
phenanthrene	86.9	1.1	94.9	2.8	96.3	2.0
phorate	90.4	1.6	87.1	2.1	90.0	1.7
phosphamidon	109	8.6	97.2	4.0	95.2	2.8
profenofos	99.9	11	95.6	6.9	102	4.1
prometon	83.1	4.0	98.6	2.8	90.4	4.2
prometryn	103	4.7	99.8	2.5	99.3	2.5
pronamide	94.1	6.0	99.1	2.2	95.8	1.6
propachlor	94.5	0.94	98.6	1.2	96.5	0.77
propazine	107	4.1	98.9	2.2	97.3	3.3
pyrene	92.0	2.7	96.0	2.3	100	2.3
simazine	88.0	9.3	98.3	2.2	96.4	1.7
simetryn	92.4	6.0	97.3	2.1	108	3.5
tebuconazole	106	3.2	96.9	2.8	94.2	1.1
tebuthiuron	91.7	5.6	97.0	2.5	95.2	2.3
terbacil	82.6	9.0	102	5.7	97.7	3.0
terbutryn	99.4	2.8	99.0	1.7	100	3.1
tetrachlorvinphos	96.8	5.6	88.6	3.3	99.1	3.3
triadimefon	102	11	112	3.4	96.4	4.1
tribufos+merphos	119	2.2	90.4	2.7	100	3.4
trifluralin	92.5	3.3	98.9	1.3	93.9	0.71
vernolate	92.0	1.2	96.7	2.0	94.0	0.71
vinclozolin	98.8	6.6	101	3.1	97.9	3.8
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	84.7	4.6	92.9	2.4	90.5	0.78

Table 5. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
4-chlorobiphenyl (3)	81.8	6.5	92.8	2.2	90.0	1.2
2,4′-dichlorobiphenyl (8)	85.4	5.6	93.8	1.7	91.5	1.0
2,2′,5-trichlorobiphenyl (18)	80.4	2.3	93.9	3.9	92.9	4.2
2,4,4′-trichlorobiphenyl (28)	85.0	7.6	91.5	4.8	94.9	3.9
2,2′,3,5′-tetrachlorobiphenyl (44)	88.7	4.2	91.6	3.5	101	2.9
2,2′,5,5′-tetrachlorobiphenyl (52)	84.5	3.6	93.4	2.7	98.5	3.3
2,3′,4′,5-tetrachloroobiphenyl (70)	89.2	3.5	94.6	2.1	101	2.3
2,3,3′,4′,6-pentachlorobiphenyl (110)	93.6	3.0	99.1	1.8	102	2.0
2,3′,4,4′,5-pentachlorobiphenyl (118)	92.8	4.1	99.2	1.6	104	4.5
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	94.0	0.77	99.8	3.5	103	3.7
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	94.6	4.2	100	3.1	104	3.3
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	97.5	2.5	99.3	3.5	104	2.9
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	93.6	9.9	102	5.9	99.9	1.1
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	88.3	1.0	92.5	1.5	95.7	1.0
benzo[*a*]pyrene-d_{12}	92.2	3.7	88.8	4.3	94.6	3.9
triphenyl phosphate	99.7	2.2	94.0	2.2	91.1	2.1

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, tebuconazole is 1.2 µg/L, c-permethrin is 0.14 µg/L, t-permethrin is 0.36 µg/L, MGK 264(a) is 0.20 µg/L and MGK 264(b) is 0.05 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, tebuconazole is 10.0 µg/L, c-permethrin is 1.1µg/L, t-permethrin is 2.9µg/L, and MGK 264(a) is 1.6 µg/L and MGK 264(b) is 0.4 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, tebuconazole is 25.0 µg/L, c-permethrin is 2.8 µg/L, t-permethrin is 7.2 µg/L, and MGK 264(a) is 4.0µg/L and MGK 264(b) is 1.0 µg/L.
e. ND = Not determined. Analyte could not be determined because of the low concentration.

Table 6. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	84.8	1.5	91.5	2.0
acetochlor	2.0	106	2.2	103	2.7
alachlor	2.0	108	3.4	104	1.0
aldrin	2.0	90.6	4.0	85.5	4.2
ametryn	2.0	92.2	6.5	87.5	5.3
anthracene	2.0	97.7	3.2	99.3	2.8
atraton	2.0	94.2	2.7	86.0	4.8
atrazine	2.0	97.3	2.4	90.9	2.0
benzo[*a*]anthracene	2.0	101	2.6	96.4	2.3
benzo[*a*]pyrene	2.0	105	4.8	97.5	6.0
benzo[*b*]fluoranthene	2.0	101	3.1	100	2.1
benzo[*g*,*h*,*i*]perylene	2.0	121	2.4	118	3.2
benzo[*k*]fluoranthene	2.0	106	3.5	102	1.7
BHT	2.0	96.4	1.6	109	2.0
bromacil	2.0	104	2.2	123	16
butachlor	2.0	103	3.1	104	2.8
butylate	2.0	100	1.6	97.8	2.8
butylbenzylphthalate	2.0	116	3.9	208	94
chlordane, cis-	2.0	99.5	2.4	98.3	3.5
chlordane, trans	2.0	92.6	2.8	93.0	3.2
chlorfenvinphos	2.0	101	2.6	102	5.4
chlorobenzilate	2.0	106	3.9	105	5.8
chloroneb	2.0	84.4	1.0	83.4	1.8
chlorothalonil	2.0	97.2	1.8	96.2	3.2
chlorpropham	2.0	89.7	3.3	100	2.2
chlorpyrifos	2.0	104	6.4	99.9	4.4
chrysene	2.0	105	3.2	101	2.8
cyanazine	2.0	108	1.2	98.0	3.0

Table 6. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
cycloate	2.0	97.0	1.9	94.4	1.3
dacthal (DCPA)	2.0	96.0	4.0	97.8	2.0
DDD, 4,4′-	2.0	97.5	3.4	98.2	3.4
DDE, 4,4′-	2.0	94.4	1.8	95.0	3.7
DDT, 4,4′-	2.0	109	3.6	109	3.5
DEET	2.0	101	2.9	97.5	0.63
di(2-ethylhexyl)adipate	2.0	99.7	3.8	123	24
di(2-ethylhexyl)phthalate	2.0	106	1.7	126	29
dibenzo[*a*,*h*]anthracene	2.0	116	3.8	102	7.2
dibutyl phthalate	2.0	122	3.7	114	2.7
dichlorvos	2.0	82.3	3.2	92.1	2.0
diethylphthalate	2.0	94.3	3.5	94.4	3.5
dimethipin	2.0	49.2	11	34.9	4.6
dimethylphthalate	2.0	92.7	1.4	94.1	2.7
DIMP	2.0	91.2	2.3	87.7	1.9
dinitrotoluene, 2,4-	2.0	78.9	3.8	96.3	3.1
dinitrotoluene, 2,6-	2.0	102	4.8	111	4.7
diphenamid	2.0	109	3.9	98.2	4.7
disulfoton	2.0	81.4	2.4	74.5	1.9
endosulfan I	2.0	97.8	5.1	95.8	4.0
endosulfan II	2.0	98.9	3.0	103	8.3
endosulfan sulfate	2.0	101	4.8	98.6	3.7
endrin	2.0	105	5.7	110	6.2
EPTC	2.0	92.6	2.9	91.9	0.36
ethion	2.0	106	3.3	106	4.1
ethoprop	2.0	97.2	0.57	93.2	4.3
ethyl parathion	2.0	87.4	4.1	88.0	4.8
etridiazole	2.0	100	3.4	103	2.4

Table 6. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
fenarimol	2.0	100	3.2	98.6	1.9
fluorene	2.0	93.1	1.1	95.9	1.6
fluridone	2.0	94.4	5.8	98.0	1.8
HCCPD	2.0	105	2.4	105	5.2
HCH, α	2.0	97.9	0.92	95.5	2.9
HCH, β	2.0	101	3.4	97.0	2.9
HCH, δ	2.0	95.1	2.0	88.2	2.1
HCH, γ (lindane)	2.0	95.7	3.1	96.4	2.2
heptachlor	2.0	111	3.3	105	1.4
heptachlor epoxide	2.0	94.6	4.2	99.0	1.9
hexachlorobenzene	2.0	87.6	1.6	85.8	2.8
hexazinone	2.0	110	6.7	100	2.8
indeno[1,2,3-*c*,*d*]pyrene	2.0	115	1.5	112	4.8
isophorone	2.0	90.7	2.6	90.2	1.8
methoxychlor	2.0	99.0	2.2	106	2.0
methyl parathion	2.0	101	4.1	104	3.0
metolachlor	2.0	105	3.4	102	2.7
metribuzin	2.0	74.7	2.9	76.0	2.8
mevinphos	2.0	84.2	2.6	93.7	3.1
MGK 264(a)	1.6	102	4.0	102	3.1
MGK 264(b)	0.4	100	5.1	104	3.2
molinate	2.0	90.6	2.0	88.6	2.3
napropamide	2.0	105	2.4	102	6.2
nitrofen	2.0	105	4.7	107	7.1
nonachlor, trans	2.0	99.0	3.3	95.9	1.0
norflurazon	2.0	83.2	8.1	97.5	6.4
oxyfluorfen	2.0	109	3.0	110	4.1
pebulate	2.0	94.5	4.0	92.4	2.7

Table 6. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
pentachlorophenol	8.0	87.7	3.9	92.5	2.6
permethrin, cis	1.1	95.7	0.88	102	2.8
permethrin, trans	2.9	94.4	2.0	97.8	2.1
phenanthrene	2.0	98.6	2.4	98.5	1.5
phorate	2.0	89.1	0.39	97.3	2.7
phosphamidon	2.0	106	4.8	105	5.6
profenofos	2.0	109	3.8	102	13
prometon	2.0	92.1	3.9	83.6	3.2
prometryn	2.0	95.5	3.4	89.3	2.2
pronamide	2.0	105	2.5	99.8	1.6
propachlor	2.0	100	1.8	97.6	1.4
propazine	2.0	102	2.2	93.3	3.1
pyrene	2.0	105	5.6	102	4.0
simazine	2.0	75.4	0.82	74.0	2.1
simetryn	2.0	93.0	3.2	90.7	5.3
tebuconazole	10.0	108	3.9	108	1.9
tebuthiuron	2.0	92.1	2.5	84.2	2.8
terbacil	2.0	105	4.8	108	6.9
terbutryn	2.0	101	3.3	94.5	4.3
tetrachlorvinphos	2.0	98.9	2.7	101	4.3
triadimefon	2.0	102	3.7	99.1	5.5
tribufos+merphos	2.0	108	4.0	110	4.6
trifluralin	2.0	100	2.6	103	2.3
vernolate	2.0	92.9	0.68	90.4	2.1
vinclozolin	2.0	98.7	2.7	100	0.61
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	83.5	1.0	83.8	2.0
4-chlorobiphenyl (3)	2.0	91.9	2.6	89.3	2.3

Table 6. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE Columns (Cartridges); N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
2,4′-dichlorobiphenyl (8)	2.0	85.1	1.8	87.4	1.8
2,2′,5-trichlorobiphenyl (18)	2.0	89.5	3.9	90.9	2.8
2,4,4′-trichlorobiphenyl (28)	2.0	91.6	2.6	88.9	2.0
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	96.9	3.6	96.2	2.0
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	93.5	1.9	93.5	2.4
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	95.6	2.6	96.1	2.3
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	93.5	1.6	92.2	3.1
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	92.0	2.5	94.9	3.5
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	93.4	2.7	93.5	4.6
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	97.9	1.2	101	2.2
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	97.0	3.3	97.2	2.5
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	89.1	3.3	99.0	4.9
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	5.0	83.3	6.4	91.4	0.40
benzo[*a*]pyrene-d_{12}	5.0	94.0	4.2	90.5	2.6
triphenyl phosphate	5.0	104	4.0	107	1.9

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 342 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.8 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

Table 7. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	87.9	1.9	84.6	5.1	90.4	1.8
acetochlor	110	4.0	99.4	3.5	105	3.1
alachlor	107	5.0	92.5	3.8	110	2.4
aldrin	92.7	2.5	82.2	3.8	104	3.5
ametryn	96.6	3.2	89.5	7.3	99.2	5.7
anthracene	85.6	3.2	86.7	4.3	104	2.4
atraton	96.4	12	92.1	4.9	100	2.0
atrazine	96.8	6.7	94.2	2.6	103	2.9
benzo[*a*]anthracene	85.6	0.43	94.0	4.9	90.1	0.88
benzo[*a*]pyrene	94.5	11	81.3	5.9	94.6	3.5
benzo[*b*]fluoranthene	90.2	2.6	88.6	6.5	94.3	2.5
benzo[*g,h,i*]perylene	89.5	10	93.0	7.5	96.6	3.8
benzo[*k*]fluoranthene	103	7.4	90.1	4.7	94.1	2.8
BHT	50.4	11	107	2.6	85.4	1.8
bromacil	102	8.2	99.6	28	106	1.8
butachlor	103	2.1	95.5	2.7	105	2.0
butylate	91.5	2.6	87.6	3.6	95.8	1.4
butylbenzylphthalate	91.3	5.8	121	5.7	108	1.5
chlordane, cis	106	5.2	94.9	4.9	105	4.3
chlordane, trans	101	5.3	89.6	5.2	104	4.4
chlorfenvinphos	106	5.3	99.3	2.7	104	0.78
chlorobenzilate	106	4.7	96.7	4.4	107	1.7
chloroneb	95.5	2.9	82.0	6.0	94.3	1.8
chlorothalonil	96.0	5.6	83.8	5.6	103	2.5
chlorpropham	77.3	12	88.7	11	102	1.4
chlorpyrifos	101	13	98.7	4.2	110	3.0
chrysene	96.5	7.9	95.4	5.2	94.2	0.73
cyanazine	101	6.4	91.8	6.7	106	2.5

Table 7. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
cycloate	94.0	3.4	89.1	2.4	98.8	1.7
dacthal (DCPA)	93.2	8.3	89.8	3.5	101	1.9
DDD, 4,4′-	95.8	1.2	94.5	3.5	102	2.2
DDE, 4,4′-	95.2	4.9	89.2	3.2	106	3.4
DDT, 4,4′-	98.2	4.0	86.5	6.5	104	3.0
DEET	98.7	4.8	89.4	6.0	99.3	0.64
di(2-ethylhexyl)adipate	96.1	7.8	105	8.3	97.6	0.91
di(2-ethylhexyl)phthalate	ND[e]		104	9.3	94.4	3.7
dibenzo[*a*,*h*]anthracene	101	14	90.9	10	106	3.1
dibutylphthalate	99.1	32	110	3.5	107	2.9
dichlorvos	109	5.0	100	9.5	102	2.3
dieldrin	106	7.8	95.9	3.9	109	4.6
diethylphthalate	90.8	5.5	87.7	4.8	98.8	1.0
dimethipin	83.7	17	76.1	5.5	64.6	4.1
dimethylphthalate	93.3	6.7	87.2	5.8	96.2	1.2
DIMP	101	2.5	84.6	7.2	97.0	0.36
dinitrotoluene, 2,4-	77.5	3.3	87.4	16	99.8	3.5
dinitrotoluene, 2,6-	ND[f]		97.3	14	92.2	1.1
diphenamid	105	4.4	95.4	4.9	108	1.9
disulfoton	80.0	5.5	80.4	10	96.7	2.7
endosulfan I	109	2.6	92.3	5.6	107	2.4
endosulfan II	99.6	4.2	96.8	3.7	110	2.3
endosulfan sulfate	96.6	7.5	90.0	5.7	103	2.0
endrin	110	8.6	120	8.4	107	2.5
EPTC	93.8	5.7	85.0	5.7	94.8	0.59
ethion	98.4	7.0	98.7	2.8	101	1.1
ethoprop	108	4.2	95.9	7.7	103	2.8
ethyl parathion	90.6	14	90.0	9.6	104	2.3

Table 7. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
etridiazole	88.8	4.0	84.5	11	96.8	1.9
fenarimol	123	3.1	103	6.7	101	1.6
fluorene	82.5	4.0	85.5	5.2	94.1	0.86
fluridone	108	13	87.2	16	110	4.5
HCCPD	67.5	13	77.7	12	88.4	2.1
HCH, α	101	4.5	88.2	4.9	102	0.18
HCH, β	99.4	4.6	92.1	5.5	105	2.9
HCH, δ	104	2.6	91.4	5.0	112	2.6
HCH, γ (lindane)	95.1	5.6	92.9	3.2	104	1.9
heptachlor	87.3	4.6	91.1	5.8	104	2.9
heptachlor epoxide	98.9	3.2	98.0	5.2	104	2.6
hexachlorobenzene	79.4	5.9	76.4	5.6	95.7	0.52
hexazinone	107	9.7	89.1	4.2	103	1.3
indeno[1,2,3-*c*,*d*]pyrene	87.8	17	94.9	4.5	98.1	2.9
isophorone	96.4	2.0	85.1	4.3	97.5	0.52
methoxychlor	97.4	6.5	98.7	9.8	96.3	0.80
methyl parathion	103	8.3	92.1	4.2	102	3.5
metolachlor	99.0	6.4	94.4	4.5	106	2.7
metribuzin	93.7	3.2	81.0	2.8	99.8	2.5
mevinphos	94.2	10	88.3	13	100	3.9
MGK 246(a)	105	5.2	92.2	4.5	110	2.2
MGK 264(b)	95.5	4.4	98.0	5.0	107	2.2
molinate	93.2	9.8	87.2	4.8	98.4	3.6
napropamide	106	5.3	90.6	4.2	107	1.6
nitrofen	103	9.1	97.4	8.3	99.4	3.5
nonachlor, trans	90.1	3.7	86.0	4.5	103	1.5
norflurazon	102	10	96.1	14	110	0.76
oxyfluorfen	98.3	8.3	93.2	4.6	101	1.2

Table 7. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
pebulate	95.0	3.3	84.6	3.4	94.2	1.8
pentachlorophenol	93.5	6.0	111	3.9	101	4.2
permethrin, cis	89.5	4.4	96.3	7.0	93.8	1.5
permethrin, trans	91.6	3.8	90.4	6.7	96.1	2.7
phenanthrene	87.8	1.1	93.4	3.3	98.1	2.5
phorate	86.5	1.6	86.3	2.5	94.4	1.2
phosphamidon	103	8.6	105	4.3	108	1.9
profenofos	116	11	99.0	5.2	104	2.1
prometon	76.7	4.0	97.3	4.6	91.7	4.7
prometryn	99.7	4.7	91.4	5.0	101	2.4
pronamide	96.8	6.0	95.6	3.7	101	2.7
propachlor	98.3	0.9	88.0	4.4	103	1.0
propazine	95.0	4.1	88.5	3.9	102	2.6
pyrene	99.2	2.7	98.0	2.3	103	0.91
simazine	89.5	9.3	83.8	5.8	99.6	3.7
simetryn	99.1	6.0	91.2	3.9	112	2.9
tebuconazole	107	3.2	100	8.2	100	0.91
tebuthiuron	94.1	5.6	84.1	4.0	101	0.19
terbacil	98.2	9.0	95.1	3.6	110	2.6
terbutryn	103	2.8	91.2	4.7	105	2.9
tetrachlorvinphos	108	5.6	97.5	4.0	110	3.8
triadimefon	110	11	94.4	4.9	103	3.4
tribufos+merphos	126	2.2	96.3	5.3	107	2.0
trifluralin	84.3	3.3	89.6	8.7	95.5	0.67
vernolate	97.2	1.2	85.9	5.8	95.0	2.8
vinclozolin	95.0	6.6	96.1	3.5	102	1.4
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	90.2	4.6	78.9	4.7	91.0	2.2

Table 7. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b] n=4		Fortified Conc. 2.0 µg/L[c] n=8		Fortified Conc. 5.0 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
4-chlorobiphenyl (3)	87.9	6.5	80.5	5.8	92.7	0.82
2,4-dichlorobiphenyl (8)	85.3	5.6	78.6	5.5	94.2	1.5
2,2′,5-trichlorobiphenyl (18)	90.7	2.3	85.9	4.7	94.9	4.0
2,4,4′-trichlorobiphenyl (28)	93.2	7.6	90.0	6.7	103	2.1
2,2′,3,5′-tetrachlorobiphenyl (44)	97.5	4.2	89.5	3.9	108	2.4
2,2′,5,5′-tetrachlorobiphenyl (52)	89.0	3.6	95.0	3.1	103	1.7
2,3′,4′,5-tetrachloroobiphenyl (70)	102	3.5	89.6	3.8	106	3.6
2,3,3′,4′,6-pentachlorobiphenyl (110)	98.0	3.0	89.1	2.7	102	2.4
2,3′,4,4′,5-pentachlorobiphenyl (118)	96.0	4.1	90.3	3.5	105	1.6
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	93.7	0.8	90.9	3.3	105	3.4
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	96.4	4.2	92.4	2.0	103	2.8
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	98.3	2.5	90.0	5.3	104	3.5
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	87.2	9.9	88.4	6.5	95.3	2.2
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	98.0	1.0	95.5	3.9	102	2.4
benzo[*a*]pyrene-d_{12}	101	3.7	82.1	3.1	91.9	2.2
triphenyl phosphate	103	2.2	110	3.8	97.0	2.1

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, tebuconazole is 1.2 µg/L, c-permethrin is 0.14 µg/L, t-permethrin is 0.36 µg/L, MGK 264(a) is 0.20 µg/L and MGK 264(b) is 0.05 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, tebuconazole is 10.0 µg/L, c-permethrin is 1.1µg/L, t-permethrin is 2.9µg/L, and MGK 264(a) is 1.6 µg/L and MGK 264(b) is 0.4 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, tebuconazole is 25.0 µg/L, c-permethrin is 2.8 µg/L, t-permethrin is 7.2 µg/L, and MGK 264(a) is 4.0µg/L and MGK 264(b) is 1.0 µg/L.
e. ND = Not determined. Analyte could not be determined because of the low fortified concentration relative to the LRB.
f. ND = Not determined. Analyte could not be determined due to poor instrument sensitivity at this concentration.

Table 8. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	98.8	2.4	87.5	1.1
acetochlor	2.0	123	4.2	101	4.4
alachlor	2.0	116	5.8	91.6	3.8
aldrin	2.0	102	3.1	80.5	4.3
ametryn	2.0	113	2.0	82.7	2.0
anthracene	2.0	103	3.2	88.6	3.9
atraton	2.0	114	4.3	83.0	6.4
atrazine	2.0	119	5.9	93.0	4.8
benzo[*a*]anthracene	2.0	93.6	2.6	95.2	2.1
benzo[*a*]pyrene	2.0	80.2	4.5	85.6	11
benzo[*b*]fluoranthene	2.0	89.5	2.8	88.8	6.1
benzo[*g*,*h*,*i*]perylene	2.0	90.7	3.5	92.9	5.0
benzo[*k*]fluoranthene	2.0	88.6	2.3	87.8	3.4
BHT	2.0	121	3.6	109	0.46
bromacil	2.0	99.1	2.6	115	4.4
butachlor	2.0	114	3.3	99.1	5.6
butylate	2.0	104	4.0	92.0	4.6
butylbenzylphthalate	2.0	115	2.4	95.9	4.4
chlordane, cis-	2.0	118	7.2	93.1	6.0
chlordane, trans	2.0	114	4.3	85.1	4.4
chlorfenvinphos	2.0	116	3.7	95.9	7.4
chlorobenzilate	2.0	123	2.9	95.2	5.5
chloroneb	2.0	95.8	2.1	82.9	2.5
chlorothalonil	2.0	102	4.8	82.8	5.0
chlorpropham	2.0	100	5.5	99.3	1.1
chlorpyrifos	2.0	123	2.5	88.4	3.3
chrysene	2.0	95.0	6.3	93.9	2.9
cyanazine	2.0	121	4.4	84.4	6.2

Table 8. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
cycloate	2.0	105	3.2	91.9	1.9
dacthal (DCPA)	2.0	116	4.2	89.9	4.6
DDD, 4,4′-	2.0	119	3.4	94.1	4.9
DDE, 4,4′-	2.0	113	4.1	87.4	5.2
DDT, 4,4′-	2.0	112	3.5	87.5	6.0
DEET	2.0	106	1.9	95.4	1.2
di(2-ethylhexyl)adipate	2.0	112	6.4	102	1.9
di(2-ethylhexyl)phthalate	2.0	113	7.4	108	5.2
dibenzo[*a*,*h*]anthracene	2.0	90.4	3.8	90.0	5.3
dibutyl phthalate	2.0	132	4.2	102	5.1
dichlorvos	2.0	116	4.2	112	2.7
dieldrin	2.0	126	4.0	101	5.8
diethylphthalate	2.0	103	3.8	89.1	1.7
dimethipin	2.0	98.6	7.0	77.8	3.1
dimethylphthalate	2.0	103	4.1	89.1	2.6
DIMP	2.0	111	4.7	88.7	4.9
dinitrotoluene, 2,4-	2.0	87.9	3.7	100	4.9
dinitrotoluene, 2,6-	2.0	110	7.9	110	1.3
diphenamid	2.0	121	5.1	95.5	5.3
disulfoton	2.0	91.9	4.8	84.9	1.8
endosulfan I	2.0	121	1.7	90.4	5.8
endosulfan II	2.0	117	6.1	93.9	12
endosulfan sulfate	2.0	112	5.1	84.9	6.0
endrin	2.0	103	8.8	115	3.3
EPTC	2.0	100	3.1	89.5	3.1
ethion	2.0	112	1.4	102	4.4
ethoprop	2.0	112	2.3	104	1.2
ethyl parathion	2.0	95.4	5.7	99.8	1.1

Table 8. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
etridiazole	2.0	105	2.9	90.5	2.8
fenarimol	2.0	99.3	4.8	111	2.8
fluorene	2.0	99.6	3.5	88.9	1.6
fluridone	2.0	86.3	3.5	82.8	2.4
HCCPD	2.0	96.6	2.5	82.9	6.3
HCH, α	2.0	105	3.5	90.0	1.1
HCH, β	2.0	111	3.7	88.7	4.2
HCH, δ	2.0	107	5.2	84.4	5.2
HCH, γ (lindane)	2.0	118	4.3	86.3	2.7
heptachlor	2.0	116	5.1	84.5	7.2
heptachlor epoxide	2.0	121	3.4	93.0	2.8
hexachlorobenzene	2.0	94.8	4.0	78.1	2.8
hexazinone	2.0	104	4.1	79.3	5.6
indeno[1,2,3-*c*,*d*]pyrene	2.0	87.3	4.3	103	7.7
isophorone	2.0	103	3.7	89.8	3.1
methoxychlor	2.0	109	3.2	102	3.8
methyl parathion	2.0	107	4.7	100	5.0
metolachlor	2.0	120	3.0	94.9	4.2
metribuzin	2.0	101	5.0	80.6	2.7
mevinphos	2.0	94.3	2.6	94.2	2.6
MGK 246(a)	1.6	116	5.6	94.7	5.5
MGK 264(b)	0.4	114	4.3	89.4	9.9
molinate	2.0	101	3.6	89.3	1.8
napropamide	2.0	112	3.9	87.1	6.4
nitrofen	2.0	121	1.8	101	5.6
nonachlor, trans	2.0	108	3.9	83.8	3.4
norflurazon	2.0	106	2.4	102	6.8
oxyfluorfen	2.0	114	1.9	101	5.6

Table 8. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
pebulate	2.0	98.0	4.6	84.4	1.7
pentachlorophenol	8.0	118	2.3	110	3.8
permethrin, cis	1.1	93.9	4.8	93.4	4.8
permethrin, trans	2.9	94.8	4.7	92.7	2.5
phenanthrene	2.0	110	5.1	92.2	4.0
phorate	2.0	94.8	3.7	89.1	2.4
phosphamidon	2.0	126	5.2	97.6	3.9
profenofos	2.0	120	4.8	97.7	8.0
prometon	2.0	117	4.6	82.6	3.5
prometryn	2.0	118	3.7	86.2	4.9
pronamide	2.0	119	3.7	94.6	4.4
propachlor	2.0	104	1.9	94.2	1.3
propazine	2.0	113	4.1	85.2	3.2
pyrene	2.0	119	2.1	96.8	4.3
simazine	2.0	103	2.4	81.6	6.7
simetryn	2.0	116	4.0	88.3	6.5
tebuconazole	10.0	105	2.6	97.8	4.2
tebuthiuron	2.0	101	4.3	81.9	3.7
terbacil	2.0	120	4.1	101	4.7
terbutryn	2.0	113	2.5	85.9	3.6
tetrachlorvinphos	2.0	115	5.6	95.8	6.0
triadimefon	2.0	121	3.4	93.9	5.4
tribufos+merphos	2.0	121	2.1	102	6.1
trifluralin	2.0	110	2.7	94.8	2.7
vernolate	2.0	105	2.2	91.0	1.1
vinclozolin	2.0	117	4.6	96.1	5.1
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	94.2	2.8	81.3	2.0

Table 8. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Water from Ground and Surface Water Sources, and Extracted Using J.T. Baker Speedisk H_2O Phobic DVB SPE; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
4-chlorobiphenyl (3)	2.0	92.9	1.3	85.3	1.9
2,4-dichlorobiphenyl (8)	2.0	93.2	1.9	80.4	1.2
2,2′,5-trichlorobiphenyl (18)	2.0	109	4.7	86.4	4.9
2,4,4′-trichlorobiphenyl (28)	2.0	118	8.4	88.3	4.4
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	112	3.2	89.1	6.3
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	121	2.4	95.4	5.3
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	114	4.2	91.4	6.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	113	3.7	88.3	4.5
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	113	3.1	89.2	5.9
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	116	3.6	88.3	6.8
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	119	3.1	93.8	4.1
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	112	5.2	88.4	5.3
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	90.9	7.7	83.8	3.4
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	5.0	111	3.3	102	1.5
benzo[*a*]pyrene-d_{12}	5.0	79.8	3.4	86.0	2.8
triphenyl phosphate	5.0	115	3.7	115	2.3

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 342 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.8 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

Table 9. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Empore SDB-XC Disks; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.20 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	90.0	4.5	87.9	2.6	86.4	5.4
acetochlor	106	4.5	94.5	2.6	90.0	7.6
alachlor	98.8	6.4	92.0	1.9	92.3	8.2
aldrin	91.3	5.2	80.5	3.1	86.6	7.1
ametryn	106	5.9	90.8	4.6	94.3	9.2
anthracene	98.8	4.8	91.1	2.3	89.4	5.7
atraton	109	4.4	90.2	2.3	91.8	7.3
atrazine	105	3.9	91.1	3.5	91.6	8.0
benzo[*a*]anthracene	95.0	4.3	91.6	1.9	91.0	5.1
benzo[*a*]pyrene	96.3	7.8	88.0	3.5	88.9	5.9
benzo[*b*]fluoranthene	106	5.9	90.6	1.5	91.9	6.2
benzo[*g,h,i*]perylene	108	6.0	87.0	5.4	89.4	6.9
benzo[*k*]fluoranthene	111	4.3	89.4	1.6	89.4	5.6
BHT	97.5	8.9	81.0	4.4	81.7	7.0
bromacil	124	2.0	90.2	3.5	96.4	7.2
butachlor	115	3.5	92.1	3.7	93.2	8.0
butylate	100	4.1	88.6	3.6	87.1	6.9
butylbenzylphthalate	131	6.5	94.1	3.3	96.3	7.4
chlordane, cis	96.3	5.0	90.7	2.9	88.8	7.9
chlordane, trans	96.3	5.0	90.8	3.0	89.3	7.3
chlorfenvinphos	123	2.4	92.4	3.5	95.9	7.0
chlorobenzilate	105	3.9	92.9	3.7	92.9	8.4
chloroneb	283	10	101	4.1	96.6	7.1
chlorothalonil	98.8	2.5	88.6	2.8	88.4	7.3
chlorpropham	113	5.7	90.5	3.0	90.2	6.7
chlorpyrifos	96.3	5.0	93.3	3.1	89.6	7.3
chrysene	72.5	12	89.2	5.7	89.4	5.2
cyanazine	101	4.7	88.8	5.8	90.5	9.0
cycloate	90.0	4.5	90.1	0.99	91.6	7.1

Table 9. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Empore SDB-XC Disks; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.20 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
dacthal (DCPA)	95.0	4.3	92.7	2.7	89.6	7.5
DDD, 4,4′-	92.5	3.1	93.3	2.7	88.7	7.9
DDE, 4,4′-	98.8	6.4	90.8	2.8	90.5	7.2
DDT, 4,4′-	113	4.4	87.8	2.7	92.5	7.9
DEET	103	4.9	92.7	2.9	90.1	7.2
di(2-ethylhexyl)adipate	129	4.9	90.9	3.4	96.4	9.0
di(2-ethylhexyl)phthalate	160	5.1	98.9	2.7	102	8.5
dibenzo[*a*,*h*]anthracene	104	4.6	90.7	4.2	90.2	7.9
dibutyl phthalate	741	9.8	105	2.9	125	7.5
dichlorvos	101	4.7	86.6	3.3	88.6	6.5
dieldrin	103	4.9	91.0	3.0	90.4	7.7
diethylphthalate	97.5	6.6	90.7	3.0	88.6	7.3
dimethipin	23.8	11	8.52	9.0	6.7	11
dimethylphthalate	90.0	4.5	90.2	3.2	87.0	7.4
DIMP	103	6.3	64.0	3.5	67.8	6.1
dinitrotoluene, 2,4-	116	5.4	90.5	2.2	90.4	6.7
dinitrotoluene, 2,6-	108	6.0	88.9	3.3	90.1	6.7
diphenamid	111	4.3	93.3	2.9	92.2	7.3
disulfoton	98.8	4.8	87.1	4.1	85.4	7.2
endosulfan I	77.5	3.7	92.8	2.9	89.5	8.1
endosulfan II	83.8	5.7	93.5	3.7	91.0	7.3
endosulfan sulfate	104	4.6	90.9	3.3	90.0	8.1
endrin	111	5.7	94.5	1.8	93.4	7.0
EPTC	100	4.1	89.0	3.1	88.2	6.8
ethion	120	3.4	94.2	3.1	93.7	8.1
ethoprop	114	2.2	94.0	2.5	92.3	6.5
ethyl parathion	121	3.9	91.2	3.0	93.1	7.8
etridiazole	110	3.7	90.2	1.7	87.1	7.1
fenarimol	123	5.3	93.0	4.9	95.7	7.3

Table 9. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Empore SDB-XC Disks; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.20 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
fluorene	90.0	4.5	89.8	2.6	86.6	5.6
fluridone	116	5.4	94.1	2.3	100	8.5
HCCPD	75.0	5.4	78.3	2.6	70.5	6.3
HCH, α	88.8	2.8	88.7	2.7	85.9	7.2
HCH, β	95.0	6.1	91.9	3.3	89.4	8.2
HCH, δ	101	2.5	89.9	3.6	89.0	7.6
HCH, γ (lindane)	93.8	6.7	91.8	2.5	88.2	7.4
heptachlor	86.3	5.6	88.4	4.4	86.4	8.0
heptachlor epoxide	98.8	2.5	92.5	1.9	90.2	7.7
hexachlorobenzene	82.5	7.8	87.2	1.8	83.7	5.4
hexazinone	121	3.9	92.2	2.4	97.0	7.7
indeno[1,2,3-*c,d*]pyrene	111	4.3	87.7	4.3	91.9	7.1
isophorone	98.8	8.6	82.9	4.2	82.3	5.7
methoxychlor	118	4.3	92.3	3.2	93.1	6.9
methyl parathion	123	5.3	91.5	3.2	92.8	7.6
metolachlor	109	5.8	93.1	3.1	92.9	7.9
metribuzin	113	4.4	90.9	3.3	90.9	7.2
mevinphos	118	5.5	80.6	4.9	87.8	6.4
MGK 264(a)	128	4.8	89.6	4.8	91.3	8.1
MGK 264(b)	123	6.1	92.4	2.9	93.6	7.6
molinate	103	2.8	90.7	2.6	88.3	6.7
napropamide	111	4.3	94.1	3.4	94.9	8.0
nitrofen	111	2.2	91.4	1.7	93.6	6.9
nonachlor, trans	93.8	6.7	90.6	2.9	88.6	8.0
norflurazon	123	5.3	89.9	4.2	95.8	9.0
oxyfluorfen	110	3.7	95.4	2.9	94.3	7.8
pebulate	101	4.7	89.9	3.6	87.6	6.5
pentachlorophenol	95.0	5.4	95.1	4.0	88.2	6.7
permethrin, cis	112	6.3	92.2	3.4	99.7	7.5

Table 9. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Empore SDB-XC Disks; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.20 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
permethrin, trans	107	4.1	92.8	2.8	91.8	7.5
phenanthrene	95.0	4.3	92.2	2.1	90.0	5.9
phorate	121	3.9	88.2	2.1	85.7	7.5
phosphamidon	126	3.8	94.7	3.8	99.7	6.1
profenofos	125	3.3	93.4	2.5	94.9	7.3
prometon	111	5.7	91.4	3.1	93.5	7.7
prometryn	111	4.3	92.3	3.3	92.0	8.1
pronamide	118	5.5	94.4	3.6	93.9	7.6
propachlor	96.3	5.0	91.2	2.7	87.7	7.0
propazine	103	6.3	91.5	3.1	91.4	7.5
pyrene	98.8	4.8	92.5	2.1	91.0	6.0
simazine	108	2.7	90.7	3.3	91.0	7.9
simetryn	119	5.3	93.6	3.1	93.6	7.9
tebuconazole	126	2.0	89.5	1.5	95.6	9.9
tebuthiuron	116	7.3	94.0	4.4	100	5.0
terbacil	120	3.4	92.3	3.4	95.2	7.8
terbutryn	111	4.3	93.0	2.7	93.1	7.9
tetrachlorvinphos	125	3.3	92.1	3.4	97.4	7.2
triadimefon	105	8.7	93.9	2.4	94.5	7.6
tribufos+merphos	116	3.7	93.6	2.8	100	7.3
trifluralin	109	2.3	88.1	2.6	86.5	7.4
vernolate	100	4.1	90.0	2.9	87.2	6.9
vinclozolin	105	3.9	89.3	3.4	89.8	7.7
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	85.0	4.8	88.4	2.9	84.8	6.2
4-chlorobiphenyl (3)	90.0	4.5	89.2	2.6	85.6	6.1
2,4′-dichlorobiphenyl (8)	88.8	7.1	88.8	2.0	86.1	6.5
2,2′,5-trichlorobiphenyl (18)	82.5	6.1	90.3	3.1	88.3	6.8
2,4,4′-trichlorobiphenyl (28)	91.3	6.9	90.4	2.3	89.4	6.7

Table 9. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Empore SDB-XC Disks; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.20 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	91.3	6.9	91.1	3.1	89.9	7.6
2,2′,5,5′-tetrachlorobiphenyl (52)	95.0	4.3	90.6	2.9	89.6	6.6
2,3′,4′,5-tetrachloroobiphenyl (70)	91.3	6.9	91.1	2.9	90.5	7.0
2,3,3′,4′,6-pentachlorobiphenyl (110)	93.8	6.7	90.6	2.4	91.1	6.5
2,3′,4,4′,5-pentachlorobiphenyl (118)	97.5	6.6	91.2	2.3	91.4	6.7
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	90.0	4.5	91.3	3.1	91.1	7.6
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	93.8	5.1	91.1	3.0	91.5	7.0
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	95.0	4.3	91.2	1.7	91.6	7.6
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	88.8	5.4	92.5	1.9	90.9	5.2
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	93.8	3.6	93.3	3.5	87.9	7.1
benzo[*a*]pyrene-d_{12}	79.6	3.8	90.2	2.0	91.2	4.2
triphenyl phosphate	86.4	5.2	102	4.2	94.9	7.3

a. Data obtained on the instrumentation described in Sect. 13.1.1.2.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 0.80 µg/L, MGK 246(a) is 0.067 µg/L, MGK 246(b) is 0.13 µg/L, tribufos+merphos is 0.40 µg/L, c-permethrin is 0.12 µg/L, and t-permethrin is 0.29 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, MGK 246(a) is 0.67 µg/L, MGK 246(b) is 1.3 µg/L, tribufos+merphos is 4.0 µg/L, c-permethrin is 1.2 µg/L, and t-permethrin is 2.9 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20 µg/L, MGK 246(a) is 1.7 µg/L, MGK 246(b) is 3.3 µg/L, tribufos+merphos is 10 µg/L, c-permethrin is 2.9 µg/L, and t-permethrin is 7.2 µg/L.

Table 10. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Empore SDB-XC Disks; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	94.1	2.4	87.4	7.8
acetochlor	2.0	95.6	2.2	92.0	7.1
alachlor	2.0	98.1	3.3	92.4	7.2
aldrin	2.0	87.0	1.8	84.9	6.4
ametryn	2.0	101	2.2	92.0	9.2
anthracene	2.0	96.0	2.9	90.3	8.2
atraton	2.0	100	3.8	93.9	7.5
atrazine	2.0	99.1	3.1	91.4	7.1
benzo[*a*]anthracene	2.0	105	2.4	99.8	8.4
benzo[*a*]pyrene	2.0	95.1	3.4	91.5	9.8
benzo[*b*]fluoranthene	2.0	96.0	2.6	92.9	9.9
benzo[*g,h,i*]perylene	2.0	97.3	4.2	97.9	10
benzo[*k*]fluoranthene	2.0	124	3.5	118	8.2
BHT	2.0	91.3	3.7	87.5	7.3
bromacil	2.0	100	3.5	96.8	6.3
butachlor	2.0	98.3	2.5	94.3	6.9
butylate	2.0	96.4	3.0	90.5	7.8
butylbenzylphthalate	2.0	99.1	3.1	95.6	9.4
chlordane, cis-	2.0	94.9	2.3	89.6	6.7
chlordane, trans	2.0	94.0	2.6	89.8	6.3
chlorfenvinphos	2.0	102	3.5	96.8	8.0
chlorobenzilate	2.0	100	3.2	96.3	9.3
chloroneb	2.0	94.8	1.9	95.6	7.7
chlorothalonil	2.0	95.8	3.0	91.8	6.4
chlorpropham	2.0	97.4	2.8	91.9	7.5
chlorpyrifos	2.0	97.0	2.9	90.0	6.9
chrysene	2.0	84.8	2.8	79.5	9.3
cyanazine	2.0	101	2.3	79.4	4.9
cycloate	2.0	99.0	3.6	93.0	7.1

Table 10. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Empore SDB-XC Disks; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
dacthal (DCPA)	2.0	95.4	3.2	89.4	7.1
DDD, 4,4′-	2.0	92.8	3.1	86.5	7.0
DDE, 4,4′-	2.0	94.9	2.7	90.0	6.4
DDT, 4,4′-	2.0	102	3.6	102	7.8
DEET	2.0	97.9	2.7	92.9	7.3
di(2-ethylhexyl)adipate	2.0	97.0	3.1	93.3	10
di(2-ethylhexyl)phthalate	2.0	109	2.9	104	6.3
dibenzo[*a,h*]anthracene	2.0	100	3.3	102	11
dibutyl phthalate	2.0	106	3.0	96.8	7.4
dichlorvos	2.0	96.8	2.6	90.9	7.1
dieldrin	2.0	96.8	3.2	92.6	6.5
diethylphthalate	2.0	96.1	2.6	89.9	8.5
dimethipin	2.0	9.0	24	8.50	25
dimethylphthalate	2.0	94.5	3.1	88.1	7.9
DIMP	2.0	87.3	3.1	76.6	6.5
dinitrotoluene, 2,4-	2.0	97.5	2.7	93.3	7.6
dinitrotoluene, 2,6-	2.0	96.1	2.3	90.6	8.0
diphenamid	2.0	100	3.4	94.6	7.2
disulfoton	2.0	91.6	2.5	81.5	7.2
endosulfan I	2.0	95.0	3.7	90.4	6.3
endosulfan II	2.0	93.5	2.9	88.4	7.5
endosulfan sulfate	2.0	96.4	4.5	91.4	8.1
endrin	2.0	104	3.3	101	8.0
EPTC	2.0	97.3	2.5	90.6	7.6
ethion	2.0	98.3	3.6	95.6	8.1
ethoprop	2.0	103	1.1	98.9	9.5
ethyl parathion	2.0	96.8	3.7	92.9	8.4
etridiazole	2.0	99.9	2.7	95.8	7.4
fenarimol	2.0	106	2.4	103	9.4

Table 10. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Empore SDB-XC Disks; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
fluorene	2.0	94.4	2.3	88.5	8.0
fluridone	2.0	110	5.2	117	11
HCCPD	2.0	90.0	3.1	90.3	7.8
HCH, α	2.0	94.4	3.0	88.3	7.7
HCH, β	2.0	95.5	3.4	90.3	6.1
HCH, δ	2.0	96.4	2.9	92.3	6.6
HCH, γ (lindane)	2.0	95.4	3.3	91.3	5.6
heptachlor	2.0	93.1	3.5	88.5	6.9
heptachlor epoxide	2.0	95.8	3.3	90.6	8.0
hexachlorobenzene	2.0	90.0	2.1	84.0	7.5
hexazinone	2.0	102	3.2	100	9.2
indeno[1,2,3-*c*,*d*]pyrene	2.0	103	4.1	105	11
isophorone	2.0	93.4	2.7	86.9	7.2
methoxychlor	2.0	107	2.9	106	7.3
methyl parathion	2.0	98.8	3.0	94.5	6.9
metolachlor	2.0	98.6	3.2	93.1	6.6
metribuzin	2.0	98.4	2.9	89.9	6.7
mevinphos	2.0	99.8	3.4	97.0	8.2
MGK 264(a)	1.3	97.8	2.9	93.1	6.4
MGK 264(b)	0.67	96.9	4.4	95.4	6.8
molinate	2.0	97.6	2.8	91.3	7.6
napropamide	2.0	99.6	3.7	93.4	8.4
nitrofen	2.0	101	3.3	98.4	7.8
nonachlor, trans	2.0	94.4	2.9	89.4	7.4
norflurazon	2.0	102	4.4	99.6	8.8
oxyfluorfen	2.0	102	3.7	98.9	8.6
pebulate	2.0	96.8	2.9	90.4	7.7
pentachlorophenol	8.0	95.0	2.5	87.9	7.7
permethrin, cis	1.2	107	3.3	106	8.8

Table 10. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Empore SDB-XC Disks; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
permethrin, trans	2.9	101	2.9	96.9	9.1
phenanthrene	2.0	93.0	2.7	87.5	7.8
phorate	2.0	96.0	2.8	90.9	7.0
phosphamidon	2.0	105	3.7	101	7.3
profenofos	2.0	100	2.7	96.3	8.5
prometon	2.0	99.3	3.1	92.6	7.5
prometryn	2.0	102	3.2	90.8	7.3
pronamide	2.0	101	3.0	95.9	6.9
propachlor	2.0	96.4	3.0	91.1	8.0
propazine	2.0	98.4	2.8	91.4	7.1
pyrene	2.0	95.5	2.9	91.0	7.8
simazine	2.0	98.5	3.3	91.0	6.3
simetryn	2.0	99.8	3.4	92.6	6.7
tebuconazole	2.0	110	4.5	112	9.8
tebuthiuron	2.0	105	3.9	96.1	9.3
terbacil	2.0	104	2.2	99.4	5.9
terbutryn	2.0	101	2.7	95.3	7.5
tetrachlorvinphos	2.0	105	3.6	100	7.0
triadimefon	2.0	102	3.7	96.6	7.2
tribufos+merphos	4.0	95.9	3.2	91.4	7.0
trifluralin	2.0	95.0	3.5	90.5	7.9
vernolate	2.0	95.9	3.1	89.5	7.6
vinclozolin	2.0	93.0	3.1	88.4	6.6
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	93.3	2.9	86.1	8.0
4-chlorobiphenyl (3)	2.0	93.1	2.4	86.5	8.0
2,4′-dichlorobiphenyl (8)	2.0	93.3	2.9	87.0	8.4
2,2′,5-trichlorobiphenyl (18)	2.0	94.8	3.1	87.3	8.7
2,4,4′-trichlorobiphenyl (28)	2.0	94.6	3.4	88.6	7.3

Table 10. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Empore SDB-XC Disks; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	94.5	2.6	89.0	7.6
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	94.3	3.0	88.8	7.1
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	95.5	2.7	89.6	8.1
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	94.9	2.8	88.8	7.7
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	95.0	2.7	89.3	8.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	94.3	2.9	89.0	8.0
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	95.1	2.8	89.6	7.1
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	94.1	2.9	90.5	8.7
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	97.3	2.7	90.6	8.0
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	5.0	96.6	2.7	89.2	7.8
benzo[*a*]pyrene-d_{12}	5.0	95.0	4.0	91.0	11
triphenyl phosphate	5.0	100	2.7	91.2	8.4

a. Data obtained on the instrumentation described in Sect. 13.1.1.2.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 334 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.8 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

Table 11. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	101	2.0	93.6	0.51	99.8	0.88
acetochlor	99.0	3.9	93.6	2.1	104	2.1
alachlor	100	7.3	89.8	0.72	92.8	1.0
aldrin	77.0	5.0	78.4	2.9	85.0	3.4
ametryn	105	4.8	93.1	1.3	95.8	1.1
anthracene	106	3.8	92.3	1.0	104	0.71
atraton	112	2.9	90.3	4.1	96.8	2.2
atrazine	111	3.5	96.1	3.2	97.3	1.5
benzo[*a*]anthracene	112	5.1	99.1	3.8	112	3.6
benzo[*a*]pyrene	109	5.5	103	1.7	111	1.2
benzo[*b*]fluoranthene	119	5.0	102	1.2	114	2.4
benzo[*g,h,i*]perylene	112	2.9	102	4.3	113	2.8
benzo[*k*]fluoranthene	105	1.9	103	2.4	113	3.1
BHT	ND[e]		ND		ND	
bromacil	102	9.3	98.9	0.86	103	2.1
butachlor	107	3.6	86.3	1.1	99.4	1.4
butylate	85.0	7.1	83.0	2.2	84.0	3.0
butylbenzylphthalate	122	1.9	95.9	3.7	114	3.6
chlordane, cis	98.0	5.3	102	2.5	101	1.3
chlordane, trans	103	1.9	103	2.3	96.6	0.71
chlorfenvinphos	113	1.8	110	2.5	111	3.9
chlorobenzilate	82.0	9.3	99.8	5.1	94.1	1.3
chloroneb	93.0	2.2	92.0	2.9	100	1.4
chlorothalonil	116	2.8	106	3.8	105	1.5
chlorpropham	109	3.5	93.1	2.5	98.6	1.1
chlorpyrifos	102	5.1	93.4	3.3	97.2	2.5
chrysene	117	1.7	97.1	1.7	114	2.1
cyanazine	99.0	3.9	88.1	4.9	106	2.4
cycloate	102	3.9	87.4	1.0	88.8	1.2

Table 11. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
dacthal (DCPA)	105	3.6	102	3.5	101	1.7
DDD, 4,4′-	107	3.6	85.8	0.75	105	1.4
DDE, 4,4′-	99.0	3.9	82.3	1.3	101	1.0
DDT, 4,4′-	116	2.8	87.6	2.8	112	0.83
DEET	103	1.9	98.3	3.2	104	2.0
di(2-ethylhexyl)adipate	112	4.1	96.6	3.1	111	1.8
di(2-ethylhexyl)phthalate	137	3.7	97.6	1.3	110	2.4
dibenzo[*a*,*h*]anthracene	110	3.6	95.4	2.5	109	1.5
dibutyl phthalate	115	3.3	101	1.5	114	2.6
dichlorvos	104	3.1	91.6	1.7	88.8	2.7
dieldrin	103	1.9	87.4	0.55	98.1	0.39
diethylphthalate	111	1.8	111	2.3	114	1.4
dimethipin	24.0	14	29.5	6.5	24.9	2.5
dimethylphthalate	110	3.6	113	0.25	113	0.78
DIMP	112	5.8	90.0	1.9	93.7	3.0
dinitrotoluene, 2,4-	126	1.8	105	2.6	113	2.5
dinitrotoluene, 2,6-	121	1.7	106	0.71	111	0.67
diphenamid	106	2.2	95.1	0.90	97.8	0.69
disulfoton	79.0	2.5	91.5	8.9	85.3	1.5
endosulfan I	95.0	5.3	88.4	1.3	101	1.0
endosulfan II	103	1.9	89.6	3.5	103	1.0
endosulfan sulfate	112	7.1	96.5	2.4	106	0.75
endrin	89.0	5.7	82.9	3.4	91.3	4.0
EPTC	89.0	2.2	88.0	0.80	85.8	0.60
ethion	106	2.2	100	2.7	108	3.1
ethoprop	110	2.1	91.3	1.6	96.2	1.4
ethyl parathion	117	4.3	97.6	2.3	105	3.5
etridiazole	118	3.4	90.6	2.2	101	1.5
fenarimol	110	4.7	87.1	2.0	91.7	3.4

Table 11. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
fluorene	106	3.8	97.5	2.1	101	1.1
fluridone	92.0	5.0	103	4.9	98.6	2.9
HCCPD	92.0	3.5	65.6	1.7	68.0	5.8
HCH, α	101	3.8	92.5	1.3	95.2	0.47
HCH, β	101	3.8	94.0	4.0	102	1.7
HCH, δ	97.0	6.2	96.4	1.3	101	0.49
HCH, γ (lindane)	90.0	4.4	95.6	2.1	97.9	1.8
heptachlor	96.0	3.4	83.1	2.0	86.2	1.2
heptachlor epoxide	104	3.1	86.9	2.0	95.9	1.6
hexachlorobenzene	94.0	5.5	78.4	3.8	93.0	1.9
hexazinone	107	1.9	84.6	1.7	94.6	2.6
indeno[1,2,3-*c,d*]pyrene	113	4.5	95.0	2.1	112	2.4
isophorone	108	3.0	108	3.2	102	1.1
methoxychlor	122	1.9	89.9	1.5	109	0.72
methyl parathion	129	3.0	103	2.1	112	2.5
metolachlor	109	1.8	93.1	1.1	97.8	0.42
metribuzin	116	2.8	97.3	0.30	106	2.5
mevinphos	115	3.3	96.1	3.1	97.0	1.5
MGK 264(a)	94.0	2.5	75.5	1.9	88.3	4.1
MGK 264(b)	94.0	2.5	82.8	0.35	92.3	0.80
molinate	88.0	3.7	89.4	1.2	88.9	2.1
napropamide	105	3.6	89.9	2.4	99.2	1.5
nitrofen	129	3.0	106	2.7	113	3.9
nonachlor, trans	119	3.2	103	2.3	96.2	1.0
norflurazon	106	2.2	91.9	1.6	102	1.6
oxyfluorfen	129	1.6	93.9	2.9	111	3.5
pebulate	85.0	8.0	84.5	1.7	84.7	2.4
pentachlorophenol	104	4.1	100	1.3	96.0	3.6
permethrin, cis	110	3.6	107	1.3	107	2.2

Table 11. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
permethrin, trans	115	3.3	96.1	3.1	97.0	1.5
phenanthrene	94.0	2.5	75.5	1.9	88.3	4.1
phorate	94.0	2.5	82.8	0.35	92.3	0.80
phosphamidon	88.0	3.7	89.4	1.2	88.9	2.1
profenofos	105	3.6	89.9	2.4	99.2	1.5
prometon	129	3.0	106	2.7	113	3.9
prometryn	119	3.2	103	2.3	96.2	1.0
pronamide	106	2.2	91.9	1.6	102	1.6
propachlor	129	1.6	93.9	2.9	111	3.5
propazine	85.0	8.0	84.5	1.7	84.7	2.4
pyrene	104	4.1	100	1.3	96.0	3.6
simazine	110	3.6	107	1.3	107	2.2
simetryn	115	3.3	96.1	3.1	97.0	1.5
tebuconazole	94.0	2.5	75.5	1.9	88.3	4.1
tebuthiuron	94.0	2.5	82.8	0.35	92.3	0.80
terbacil	88.0	3.7	89.4	1.2	88.9	2.1
terbutryn	105	3.6	89.9	2.4	99.2	1.5
tetrachlorvinphos	129	3.0	106	2.7	113	3.9
triadimefon	119	3.2	103	2.3	96.2	1.0
tribufos+merphos	106	2.2	91.9	1.6	102	1.6
trifluralin	129	1.6	93.9	2.9	111	3.5
vernolate	85.0	8.0	84.5	1.7	84.7	2.4
vinclozolin	104	4.1	100	1.3	96.0	3.6
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	75.0	2.7	81.0	2.6	85.0	1.1
4-chlorobiphenyl (3)	85.0	2.4	84.4	2.8	88.9	1.5
2,4′-dichlorobiphenyl (8)	85.0	2.4	82.5	2.2	87.1	0.51
2,2′,5-trichlorobiphenyl (18)	104	3.1	89.3	2.9	95.3	4.0
2,4,4′-trichlorobiphenyl (28)	81.0	2.5	88.6	3.3	92.4	0.48

Table 11. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	85.0	9.7	91.1	3.1	93.4	1.6
2,2′,5,5′-tetrachlorobiphenyl (52)	84.0	3.9	90.3	4.4	96.4	1.1
2,3′,4′,5-tetrachloroobiphenyl (70)	84.0	3.9	92.4	3.4	97.0	0.94
2,3,3′,4′,6-pentachlorobiphenyl (110)	81.0	2.5	94.3	3.2	97.1	0.90
2,3′,4,4′,5-pentachlorobiphenyl (118)	90.0	2.6	94.5	3.7	98.8	1.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	86.0	4.7	99.0	3.2	105	1.3
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	87.0	5.8	96.5	4.3	101	1.2
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	79.0	2.5	96.9	2.8	101	1.1
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	101	2.0	93.1	2.9	91.6	2.2
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	92.9	4.0	98.9	3.1	88.8	4.0
benzo[*a*]pyrene-d_{12}	112	2.1	101	2.6	101	4.9
triphenyl phosphate	107	2.5	97.9	4.1	104	3.2

a. Data obtained on the instrumentation described in Sect. 13.1.1.4.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, c-permethrin is 0.13 µg/L, t-permethrin is 0.38 µg/L, MGK 264 (a) is 0.085 µg/L and MGK 264 (b) is 0.17 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, c-permethrin is 1.0 µg/L, t-permethrin is 3.0 µg/L, MGK 264 (a) is 0.67 µg/L and MGK 264 (b) is 1.3 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, c-permethrin is 2.5 µg/L, and t-permethrin is 7.5 µg/L, MGK 264 (a) is 1.7 µg/L and MGK 264 (b) is 3.3 µg/L.
e. ND = Not determined. Analyte could not be determined because of the low fortified concentration relative to the LRB.

Table 12. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	99.3	5.7	95.1	1.8
acetochlor	2.0	97.4	5.5	106	4.9
alachlor	2.0	93.5	3.5	95.0	3.3
aldrin	2.0	94.9	4.0	81.1	0.59
ametryn	2.0	98.9	5.7	93.4	6.2
anthracene	2.0	102	4.7	101	1.6
atraton	2.0	92.6	5.9	87.3	5.3
atrazine	2.0	100	4.6	95.6	1.5
benzo[*a*]anthracene	2.0	104	1.5	102	2.4
benzo[*a*]pyrene	2.0	106	3.2	100	2.7
benzo[*b*]fluoranthene	2.0	104	3.3	100	3.9
benzo[*g,h,i*]perylene	2.0	101	3.9	101	5.3
benzo[*k*]fluoranthene	2.0	103	3.0	101	2.6
BHT	2.0	95.5	1.2	114	1.9
bromacil	2.0	98.9	5.5	104	4.6
butachlor	2.0	96.4	2.9	95.6	3.3
butylate	2.0	87.6	3.2	83.6	1.8
butylbenzylphthalate	2.0	107	4.1	107	2.9
chlordane, cis-	2.0	96.1	6.2	98.0	5.9
chlordane, trans	2.0	94.5	5.9	98.3	7.3
chlorfenvinphos	2.0	93.1	3.9	111	3.4
chlorobenzilate	2.0	101	5.6	97.1	4.3
chloroneb	2.0	104	2.0	108	4.0
chlorothalonil	2.0	108	1.5	110	2.2
chlorpropham	2.0	95.9	5.6	98.1	2.4
chlorpyrifos	2.0	97.8	5.3	103	4.1
chrysene	2.0	106	2.2	100	3.2
cyanazine	2.0	97.4	5.2	91.0	11
cycloate	2.0	92.3	5.6	95.1	1.6

Table 12. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
dacthal (DCPA)	2.0	92.1	7.3	107	4.3
DDD, 4,4′-	2.0	93.1	3.7	91.0	2.0
DDE, 4,4′-	2.0	90.0	3.6	85.6	2.7
DDT, 4,4′-	2.0	91.4	3.7	90.4	3.5
DEET	2.0	101	1.8	109	1.1
di(2-ethylhexyl)adipate	2.0	102	5.8	106	3.4
di(2-ethylhexyl)phthalate	2.0	107	2.7	104	2.2
dibenzo[*a*,*h*]anthracene	2.0	106	6.2	102	2.2
dibutyl phthalate	2.0	110	3.7	107	1.5
dichlorvos	2.0	90.4	7.3	88.8	2.3
dieldrin	2.0	96.0	5.4	96.3	2.6
diethylphthalate	2.0	110	2.5	107	1.1
dimethipin	2.0	29.4	8.7	38.1	4.2
dimethylphthalate	2.0	111	2.4	111	1.0
DIMP	2.0	89.6	5.9	99.6	5.9
dinitrotoluene, 2,4-	2.0	95.6	7.0	105	4.1
dinitrotoluene, 2,6-	2.0	95.6	6.2	101	5.0
diphenamid	2.0	98.5	5.6	101	2.3
disulfoton	2.0	79.6	5.9	103	6.3
endosulfan I	2.0	96.9	7.0	93.0	4.5
endosulfan II	2.0	98.5	5.7	94.3	4.1
endosulfan sulfate	2.0	102	2.8	100	2.1
endrin	2.0	97.9	2.6	88.1	3.4
EPTC	2.0	89.0	7.2	85.8	3.0
ethion	2.0	95.8	6.9	98.9	4.3
ethoprop	2.0	94.9	5.7	103	3.9
ethyl parathion	2.0	97.6	5.9	103	3.0
etridiazole	2.0	104	2.4	104	3.1
fenarimol	2.0	93.5	4.4	86.6	3.3

Table 12. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
fluorene	2.0	101	5.1	104	1.8
fluridone	2.0	111	3.9	97.4	6.0
HCCPD	2.0	64.6	6.7	69.1	2.8
HCH, α	2.0	93.5	5.7	91.8	2.4
HCH, β	2.0	97.9	4.2	97.8	1.8
HCH, δ	2.0	102	6.4	94.3	3.7
HCH, γ (lindane)	2.0	100	6.8	90.0	2.1
heptachlor	2.0	91.0	3.0	86.1	1.5
heptachlor epoxide	2.0	98.3	6.2	91.6	1.6
hexachlorobenzene	2.0	90.4	4.0	89.0	2.7
hexazinone	2.0	93.4	3.1	97.0	8.1
indeno[1,2,3-*c*,*d*]pyrene	2.0	107	6.6	103	4.6
isophorone	2.0	104	3.9	101	2.6
methoxychlor	2.0	94.0	5.6	94.0	2.6
methyl parathion	2.0	100	3.7	110	2.5
metolachlor	2.0	97.5	5.6	97.8	2.1
metribuzin	2.0	102	2.6	120	0.77
mevinphos	2.0	89.9	5.3	96.3	4.5
MGK 264(a)	1.3	92.9	2.3	86.4	4.1
MGK 264(b)	0.67	95.9	4.1	92.6	3.9
molinate	2.0	94.9	5.0	89.1	2.3
napropamide	2.0	95.9	4.6	104	3.1
nitrofen	2.0	111	6.2	96.4	5.3
nonachlor, trans	2.0	105	4.0	98.4	5.3
norflurazon	2.0	98.1	3.8	101	6.4
oxyfluorfen	2.0	91.8	7.7	94.9	5.0
pebulate	2.0	89.3	4.4	84.3	1.0
pentachlorophenol	8.0	98.3	2.6	97.3	2.9
permethrin, cis	1.0	92.6	6.0	108	4.3

Table 12. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
permethrin, trans	3.0	91.4	7.1	100	2.3
phenanthrene	2.0	106	3.7	104	2.5
phorate	2.0	95.5	2.0	98.9	4.8
phosphamidon	2.0	100	3.3	114	2.8
profenofos	2.0	98.4	4.4	108	1.7
prometon	2.0	96.9	2.1	88.4	5.7
prometryn	2.0	98.8	4.9	94.6	4.9
pronamide	2.0	95.1	5.5	96.6	3.4
propachlor	2.0	106	1.4	110	3.4
propazine	2.0	101	2.8	96.4	2.5
pyrene	2.0	106	1.2	107	2.2
simazine	2.0	101	3.7	96.1	3.0
simetryn	2.0	98.6	4.2	87.9	3.8
tebuconazole	2.0	94.4	5.4	96.0	5.2
tebuthiuron	2.0	88.5	7.1	101	1.8
terbacil	2.0	101	5.6	95.8	10
terbutryn	2.0	97.3	5.1	87.4	5.1
tetrachlorvinphos	2.0	97.5	6.6	104	5.4
triadimefon	2.0	99.3	2.1	101	3.0
tribufos+merphos	4.0	96.6	7.3	107	2.0
trifluralin	2.0	90.8	2.2	90.0	4.5
vernolate	2.0	88.0	6.0	88.3	1.8
vinclozolin	2.0	98.6	7.0	111	1.7
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	90.8	1.7	96.3	5.6
4-chlorobiphenyl (3)	2.0	96.1	0.65	99.9	5.4
2,4′-dichlorobiphenyl (8)	2.0	97.9	0.87	86.6	6.3
2,2′,5-trichlorobiphenyl (18)	2.0	101	3.8	90.9	7.5
2,4,4′-trichlorobiphenyl (28)	2.0	101	2.2	86.1	4.9

Table 12. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT 525 Universal Cartridges; N=4 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	93.0	3.0	88.1	6.9
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	97.1	3.1	87.8	6.6
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	107	1.5	88.1	5.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	107	1.3	92.0	6.3
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	108	1.2	91.3	6.4
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	110	2.0	93.8	5.3
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	106	1.4	91.5	6.0
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	108	0.89	91.3	6.3
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	99.6	1.0	87.6	7.2
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	5.0	91.7	7.5	89.0	6.5
benzo[*a*]pyrene-d_{12}	5.0	103	2.7	104	3.0
triphenyl phosphate	5.0	104	0.58	112	3.5

a. Data obtained on the instrumentation described in Sect. 13.1.1.4.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 300 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.4 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

Table 13. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	87.3	3.4	86.6	1.1	90.5	1.3
acetochlor	92.3	4.0	93.9	2.6	95.2	1.4
alachlor	95.6	8.0	93.4	2.0	94.6	1.4
aldrin	85.6	1.2	84.7	5.7	90.4	1.2
ametryn	91.4	3.5	93.9	2.9	95.8	1.5
anthracene	87.2	4.6	90.2	2.2	92.9	1.4
atraton	93.2	5.2	94.9	5.3	98.5	2.4
atrazine	94.5	7.4	92.9	2.9	95.0	1.9
benzo[*a*]anthracene	93.6	2.5	90.6	2.1	91.3	1.7
benzo[*a*]pyrene	88.9	3.5	85.8	3.0	88.2	3.9
benzo[*b*]fluoranthene	87.9	3.6	85.1	3.0	89.0	4.0
benzo[*g,h,i*]perylene	88.3	4.2	79.8	3.9	84.5	3.8
benzo[*k*]fluoranthene	91.1	3.8	85.9	2.7	87.2	3.9
BHT	89.8	5.0	84.9	1.5	87.6	1.2
bromacil	96.9	1.9	94.3	2.8	96.6	3.6
butachlor	94.9	5.0	91.5	2.8	93.0	2.4
butylate	86.9	2.4	88.0	2.4	91.5	1.5
butylbenzylphthalate	104	19	94.5	3.9	93.6	1.2
chlordane, cis	92.1	6.5	89.2	4.9	89.7	2.5
chlordane, trans	91.2	5.0	89.4	4.2	88.9	2.3
chlorfenvinphos	89.1	7.7	94.0	4.3	95.3	2.4
chlorobenzilate	96.0	4.0	93.8	4.4	92.4	1.9
chloroneb	89.7	4.2	90.9	1.4	93.6	1.3
chlorothalonil	89.0	4.8	94.3	1.6	97.3	2.3
chlorpropham	93.7	7.7	92.5	2.3	95.8	0.75
chlorpyrifos	89.1	2.6	91.3	3.2	94.4	2.1
chrysene	90.7	2.3	92.0	2.6	90.9	1.4
cyanazine	101	8.6	108	3.3	114	3.7
cycloate	90.0	4.9	89.6	1.7	92.4	0.32

Table 13. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
dacthal (DCPA)	93.9	3.3	94.3	2.1	96.0	1.2
DDD, 4,4′-	91.0	3.4	91.8	3.4	88.3	1.8
DDE, 4,4′-	83.9	4.3	87.9	6.8	85.6	3.0
DDT, 4,4′-	88.8	6.0	88.3	4.2	86.4	2.4
DEET	91.6	5.5	92.9	2.0	94.5	0.82
di(2-ethylhexyl)adipate	84.1	8.6	76.2	3.3	76.5	3.1
di(2-ethylhexyl)phthalate	ND[e]		77.0	2.7	72.7	2.8
dibenzo[*a*,*h*]anthracene	85.3	5.0	76.7	3.7	81.5	4.5
dibutyl phthalate	ND		111	2.2	95.0	0.96
dichlorvos	93.2	4.4	86.7	0.98	89.3	2.3
dieldrin	93.6	9.2	91.3	6.2	91.1	2.8
diethylphthalate	93.6	5.4	91.4	2.2	94.2	0.90
dimethipin	36.0	13	32.8	25	28.4	9.6
dimethylphthalate	89.4	5.3	90.0	1.0	92.6	1.0
DIMP	89.9	5.3	83.9	2.7	86.8	1.8
dinitrotoluene, 2,4-	88.6	5.8	92.1	1.4	97.0	1.3
dinitrotoluene, 2,6-	89.3	5.7	89.1	2.1	94.0	0.67
diphenamid	93.8	3.2	94.7	2.1	96.2	1.4
disulfoton	71.0	5.0	77.4	4.9	77.8	4.8
endosulfan I	87.9	9.2	89.6	3.3	91.1	1.4
endosulfan II	94.8	5.8	91.5	6.7	90.6	3.0
endosulfan sulfate	95.1	4.8	93.4	2.7	93.4	1.7
endrin	88.1	2.1	92.5	4.7	93.0	2.3
EPTC	85.8	3.5	86.2	0.96	90.5	1.8
ethion	96.1	4.3	93.5	3.9	92.7	2.8
ethoprop	91.9	4.1	92.1	2.7	95.7	0.71
ethyl parathion	92.9	7.8	88.8	3.1	93.4	2.8
etridiazole	84.6	6.7	88.8	1.4	92.3	1.1
fenarimol	99.8	5.9	95.9	3.7	100	3.4

Table 13. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
fluorene	86.6	4.4	88.1	1.2	92.5	0.53
fluridone	95.4	8.2	96.1	5.9	101	4.6
HCCPD	72.6	6.9	73.5	2.4	76.5	4.4
HCH, α	91.5	5.9	91.1	1.3	93.3	0.71
HCH, β	96.1	6.1	94.4	2.8	97.0	2.3
HCH, δ	88.5	5.7	90.6	3.4	93.4	1.5
HCH, γ (lindane)	89.4	4.0	92.1	3.0	92.3	0.85
heptachlor	91.9	9.9	89.8	3.2	92.6	0.26
heptachlor epoxide	86.8	3.3	91.3	5.7	92.8	2.4
hexachlorobenzene	86.8	6.1	89.5	1.9	90.5	1.1
hexazinone	95.0	3.8	97.8	1.8	100	1.7
indeno[1,2,3-*c,d*]pyrene	85.3	5.5	80.2	3.7	86.1	4.3
isophorone	88.2	6.0	87.4	1.3	87.1	1.5
methoxychlor	94.1	4.0	93.0	2.6	92.2	1.3
methyl parathion	90.3	3.8	93.6	3.1	95.4	0.43
metolachlor	90.3	5.0	93.9	3.2	96.6	1.6
metribuzin	93.3	4.7	94.8	1.5	95.4	2.0
mevinphos	90.1	4.7	89.4	1.6	93.2	0.92
MGK 264(a)	87.5	4.0	92.3	2.6	94.5	1.5
MGK 264(b)	86.3	7.9	94.4	3.9	96.3	2.5
molinate	90.5	6.9	89.2	1.1	91.6	1.4
napropamide	97.3	7.6	93.3	3.8	92.9	1.2
nitrofen	90.4	5.1	94.4	3.3	94.0	1.3
nonachlor, trans	86.7	4.2	86.8	2.4	86.0	3.3
norflurazon	97.0	3.8	95.1	2.3	98.9	3.3
oxyfluorfen	94.3	5.2	93.2	4.5	92.9	1.9
pebulate	90.5	3.7	88.4	1.6	90.9	2.0
pentachlorophenol	93.0	4.4	97.4	3.9	94.8	3.4
permethrin, cis	87.7	2.0	79.8	1.3	80.9	3.5

Table 13. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
permethrin, trans	89.3	3.2	82.3	2.2	83.2	2.0
phenanthrene	88.6	3.9	90.1	2.4	92.6	1.7
phorate	83.7	4.2	88.8	2.3	91.1	0.97
phosphamidon	98.3	6.5	95.2	2.9	95.5	2.0
profenofos	92.0	8.6	93.7	2.8	94.7	4.2
prometon	90.0	3.8	93.9	3.2	96.0	0.79
prometryn	94.0	2.0	98.9	3.9	102	2.2
pronamide	92.3	4.0	93.5	1.7	96.1	1.9
propachlor	88.8	5.2	91.5	2.8	94.7	0.89
propazine	95.5	3.3	93.5	2.6	95.7	0.55
pyrene	92.2	4.3	91.8	3.8	92.3	2.1
simazine	93.9	3.8	94.2	1.8	98.7	2.9
simetryn	95.1	2.1	93.8	2.8	95.4	1.0
tebuconazole	93.2	5.3	94.7	2.0	97.0	2.3
tebuthiuron	95.4	11	91.3	9.5	99.6	8.9
terbacil	91.4	6.1	94.5	1.9	96.2	2.9
terbutryn	96.5	5.1	93.7	3.0	95.5	1.5
tetrachlorvinphos	95.6	4.9	92.3	3.1	93.0	2.8
triadimefon	94.5	2.4	96.1	3.3	96.7	2.6
tribufos	82.0	5.2	95.8	4.4	93.7	1.6
trifluralin	91.0	5.8	91.2	2.1	95.9	0.75
vernolate	87.3	2.5	87.0	1.1	90.7	1.8
vinclozolin	94.3	8.5	94.7	4.6	94.9	2.2
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	85.3	5.2	87.2	1.8	90.0	0.53
4-chlorobiphenyl (3)	88.2	4.0	88.1	1.3	92.0	1.1
2,4′-dichlorobiphenyl (8)	87.1	5.8	90.8	2.0	92.5	0.43
2,2′,5-trichlorobiphenyl (18)	89.4	2.5	91.0	2.9	92.0	0.54
2,4,4′-trichlorobiphenyl (28)	84.4	6.8	90.6	3.1	90.8	0.41

Table 13. Precision and Accuracy Data Obtained for Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Concentration; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	87.2	7.5	89.6	2.8	90.3	1.7
2,2′,5,5′-tetrachlorobiphenyl (52)	89.4	5.4	88.8	2.2	89.4	2.7
2,3′,4′,5-tetrachloroobiphenyl (70)	87.8	8.3	88.3	5.0	89.4	1.7
2,3,3′,4′,6-pentachlorobiphenyl (110)	87.1	3.2	90.5	4.1	88.5	3.4
2,3′,4,4′,5-pentachlorobiphenyl (118)	85.5	3.4	89.0	4.3	84.3	4.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	85.9	5.7	88.6	3.9	86.4	2.8
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	84.3	6.6	88.0	4.5	86.7	3.6
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	83.1	4.4	87.7	4.7	85.1	4.6
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	81.9	6.0	87.1	4.5	86.5	2.9
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	86.4	4.0	86.2	1.9	88.0	0.92
benzo[*a*]pyrene-d_{12}	89.6	3.5	93.5	5.1	98.9	4.2
triphenyl phosphate	87.8	2.6	93.3	2.9	99.9	1.8

a. Data obtained on the instrumentation described in Sect. 13.1.1.3.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, MGK 264 (a) is 0.200 µg/L and MGK 264 (b) is 0.050 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, , MGK 264 (a) is 1.6 µg/L and MGK 264 (b) is 0.4 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, , MGK 264 (a) is 4.0 µg/L and MGK 264 (b) is 1.0 µg/L.
e. ND = Not determined. Analyte could not be determined because of high laboratory reagent blank values relative to the fortification concentration.

Table 14. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Synthetic Hard Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	90.9	2.2	92.1	1.8
acetochlor	2.0	98.7	0.47	97.0	2.6
alachlor	2.0	96.9	1.2	95.0	1.6
aldrin	2.0	93.3	3.4	93.2	2.9
ametryn	2.0	90.8	4.2	90.0	3.2
anthracene	2.0	95.3	2.2	94.8	1.6
atraton	2.0	85.8	5.7	88.2	4.5
atrazine	2.0	92.2	3.2	93.3	0.82
benzo[*a*]anthracene	2.0	92.1	2.4	91.9	1.7
benzo[*a*]pyrene	2.0	87.4	2.3	86.8	2.4
benzo[*b*]fluoranthene	2.0	85.7	3.6	85.8	3.0
benzo[*g,h,i*]perylene	2.0	79.1	1.6	78.4	6.1
benzo[*k*]fluoranthene	2.0	87.9	3.3	85.1	3.5
BHT	2.0	92.6	1.6	93.8	2.4
bromacil	2.0	95.4	4.2	96.1	2.7
butachlor	2.0	93.5	3.6	93.4	2.1
butylate	2.0	95.0	2.0	95.8	3.4
butylbenzylphthalate	2.0	92.8	3.2	92.5	1.9
chlordane, cis-	2.0	89.0	1.7	89.8	2.3
chlordane, trans	2.0	89.7	1.6	90.3	1.3
chlorfenvinphos	2.0	95.2	4.1	95.1	2.3
chlorobenzilate	2.0	94.3	4.1	93.2	1.7
chloroneb	2.0	95.6	1.2	95.6	1.5
chlorothalonil	2.0	97.1	1.9	97.8	1.6
chlorpropham	2.0	97.1	1.4	97.4	2.0
chlorpyrifos	2.0	95.2	2.5	95.0	2.2
chrysene	2.0	94.8	2.7	91.8	2.3
cyanazine	2.0	98.7	7.1	97.4	5.4
cycloate	2.0	94.4	2.3	94.5	2.0

Table 14. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Synthetic Hard Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
dacthal (DCPA)	2.0	97.5	1.7	98.0	0.56
DDD, 4,4′-	2.0	86.5	3.5	87.5	1.4
DDE, 4,4′-	2.0	83.6	1.7	84.2	2.6
DDT, 4,4′-	2.0	82.4	2.1	82.5	0.57
DEET	2.0	99.3	2.4	99.3	2.9
di(2-ethylhexyl)adipate	2.0	72.1	1.4	73.4	2.0
di(2-ethylhexyl)phthalate	2.0	73.0	3.6	74.7	3.1
dibenzo[*a*,*h*]anthracene	2.0	75.8	2.1	75.2	6.3
dibutyl phthalate	2.0	116	2.3	114	1.0
dichlorvos	2.0	90.0	3.6	91.4	2.2
dieldrin	2.0	90.4	4.9	88.3	1.8
diethylphthalate	2.0	96.7	2.3	96.4	1.1
dimethipin	2.0	45.0	24	34.8	17
dimethylphthalate	2.0	94.3	1.8	95.2	1.9
DIMP	2.0	84.6	3.5	88.3	5.1
dinitrotoluene, 2,4-	2.0	93.4	5.6	98.2	3.0
dinitrotoluene, 2,6-	2.0	92.9	3.3	95.5	1.1
diphenamid	2.0	95.5	2.6	95.2	1.8
disulfoton	2.0	80.0	5.1	73.4	10
endosulfan I	2.0	68.1	2.7	59.4	7.7
endosulfan II	2.0	90.6	6.2	89.7	4.8
endosulfan sulfate	2.0	92.6	3.2	92.1	2.4
endrin	2.0	90.6	3.4	93.0	2.5
EPTC	2.0	93.0	2.0	94.6	2.0
ethion	2.0	92.8	3.2	89.3	1.1
ethoprop	2.0	99.1	2.1	98.7	2.6
ethyl parathion	2.0	95.4	1.9	96.3	6.8
etridiazole	2.0	96.1	2.6	95.1	2.8
fenarimol	2.0	98.7	4.5	94.4	1.8

Table 14. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Synthetic Hard Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
fluorene	2.0	93.3	1.7	93.0	2.4
fluridone	2.0	89.1	6.1	87.0	10
HCCPD	2.0	83.8	2.7	85.4	3.8
HCH, α	2.0	95.2	2.6	96.4	3.6
HCH, β	2.0	99.6	3.1	98.5	2.9
HCH, δ	2.0	94.8	3.3	94.2	2.4
HCH, γ (lindane)	2.0	92.8	1.8	94.1	2.8
heptachlor	2.0	90.5	3.7	91.4	3.7
heptachlor epoxide	2.0	95.4	4.5	92.3	3.0
hexachlorobenzene	2.0	93.6	1.3	92.8	1.8
hexazinone	2.0	95.1	1.6	94.9	4.4
indeno[1,2,3-*c*,*d*]pyrene	2.0	80.6	2.7	80.6	6.3
isophorone	2.0	90.9	2.3	91.9	1.4
methoxychlor	2.0	93.4	3.6	93.8	1.8
methyl parathion	2.0	95.4	2.6	98.3	4.9
metolachlor	2.0	97.0	1.6	98.0	1.6
metribuzin	2.0	93.7	0.99	93.6	1.5
mevinphos	2.0	95.0	2.5	95.6	1.3
MGK 264(a)	1.6	95.7	2.3	93.9	2.2
MGK 264(b)	0.4	98.4	3.6	98.1	4.0
molinate	2.0	93.9	2.2	94.5	1.7
napropamide	2.0	91.3	4.4	93.1	1.9
nitrofen	2.0	93.2	3.3	95.6	1.8
nonachlor, trans	2.0	86.7	3.7	86.3	2.9
norflurazon	2.0	97.1	3.1	96.0	1.5
oxyfluorfen	2.0	93.5	3.2	95.3	0.75
pebulate	2.0	92.8	2.3	94.3	2.4
pentachlorophenol	8.0	96.8	2.7	96.6	2.2
permethrin, cis	2.0	77.7	3.1	77.3	2.1

Table 14. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Synthetic Hard Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
permethrin, trans	2.0	80.1	2.9	79.1	1.8
phenanthrene	2.0	93.8	1.2	94.3	2.3
phorate	2.0	92.0	1.5	93.0	4.1
phosphamidon	2.0	92.7	5.3	94.6	1.7
profenofos	2.0	94.2	2.9	95.7	2.0
prometon	2.0	87.4	6.1	86.1	3.6
prometryn	2.0	93.3	4.5	91.5	2.8
pronamide	2.0	95.7	2.3	96.4	1.7
propachlor	2.0	97.1	2.8	96.3	1.3
propazine	2.0	93.8	1.8	93.0	3.0
pyrene	2.0	93.0	2.5	93.7	1.4
simazine	2.0	91.8	2.7	92.9	2.7
simetryn	2.0	88.4	3.8	89.6	3.4
tebuconazole	2.0	97.0	2.3	96.8	2.4
tebuthiuron	2.0	97.7	6.1	98.8	8.0
terbacil	2.0	96.1	2.0	96.5	2.6
terbutryn	2.0	92.4	5.4	89.6	4.2
tetrachlorvinphos	2.0	91.7	3.9	91.8	3.1
triadimefon	2.0	97.6	3.9	93.8	4.2
tribufos	2.0	95.2	3.7	93.5	2.3
trifluralin	2.0	100	1.8	99.1	2.0
vernolate	2.0	92.6	2.4	94.0	2.5
vinclozolin	2.0	97.5	1.1	97.6	3.9
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	90.8	2.0	92.0	2.4
4-chlorobiphenyl (3)	2.0	91.8	2.3	92.4	3.2
2,4′-dichlorobiphenyl (8)	2.0	94.9	2.0	95.0	2.7
2,2′,5-trichlorobiphenyl (18)	2.0	95.2	2.2	94.4	1.8
2,4,4′-trichlorobiphenyl (28)	2.0	93.6	1.9	92.9	3.4

Table 14. Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Horizon Atlantic DVB Disks; N=5 for Each Matrix; Full Scan GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Synthetic Hard Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	94.4	3.8	92.1	2.1
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	91.1	3.6	91.4	2.5
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	87.8	1.2	89.4	1.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	86.6	1.3	88.0	1.8
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	83.3	2.9	86.9	1.7
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	82.6	1.8	84.7	2.2
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	82.5	2.0	85.9	2.8
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	81.5	1.8	83.4	2.9
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	79.3	2.6	81.3	1.6
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	2.0	88.7	2.9	90.6	3.8
benzo[*a*]pyrene-d_{12}	2.0	94.1	2.4	93.4	3.2
triphenyl phosphate	2.0	92.2	1.8	92.4	1.7

a. Data obtained on the instrumentation described in Sect. 13.1.1.3.
b. A synthetic hard water sample was prepared as described in Sect. 16, Reference 33 by adding calcium carbonate, magnesium carbonate, and sodium bicarbonate to tap water. The resulting synthetic hard water was ≥ 350 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.6 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

Table 15. DLs and LCMRLs (µg/L) Calculated from Replicate Analyses of Fortified Reagent Water Samples Analyzed in Full Scan GC/MS Mode

Analytes	Laboratory 1 Oasis HLB SPE[a,b]			Laboratory 2 Empore PSDVB SPE[c]		
	DL	Fortified conc. of DL replicates	LCMRL	DL	Fortified conc. of DL replicates	LCMRL
acenaphthylene	0.015	0.050	NC[d]	0.0029	0.025	0.028
acetochlor	0.037	0.050	0.19	0.011	0.025	0.054
alachlor	0.035	0.10	0.25	0.0084	0.025	0.097
aldrin	0.056	0.10	0.30	0.010	0.025	0.420
ametryn	0.038	0.050	0.17	0.017	0.025	0.12
anthracene	0.018	0.050	0.12	0.0078	0.025	0.059
atraton	0.016	0.10	0.12	0.0055	0.025	0.12
atrazine	0.030	0.050	0.29	0.010	0.025	0.086
benzo[*a*]anthracene	0.021	0.050	0.12	0.0062	0.025	0.043
benzo[*a*]pyrene	0.043	0.050	0.18	0.035	0.025	0.11
benzo[*b*]fluoranthene	0.031	0.050	0.14	0.013	0.050	0.11
benzo[*g,h,i*]perylene	0.021	0.10	0.31	0.011	0.025	0.047
benzo[*k*]fluoranthene	0.048	0.050	0.18	0.026	0.025	0.190
BHT	0.092	0.10	0.32	0.0064	0.025	0.50
bromacil	ND[e]		0.31	0.037	0.050	0.10
butachlor	0.028	0.050	0.39	0.0043	0.025	0.089
butylate	0.024	0.050	0.16	0.0053	0.025	0.089
butylbenzylphthalate	0.068	0.10	0.40	0.016	0.025	0.20
chlordane, cis	0.10	0.10	0.33	0.0094	0.025	0.068
chlordane, trans	0.027	0.050	0.28	0.010	0.025	0.043
chlorfenvinphos	0.095	0.10	0.52	0.0062	0.025	0.098
chlorobenzilate	0.034	0.050	0.30	0.0042	0.025	0.091
chloroneb	0.068	0.10	0.16	0.19	0.025	0.90
chlorothalonil	0.036	0.10	0.17	0.0074	0.025	0.11
chlorpropham	0.026	0.050	0.21	0.013	0.025	0.14
chlorpyrifos	0.091	0.10	0.45	0.026	0.025	0.076
chrysene	0.017	0.050	0.15	0.010	0.050	0.035
cyanazine	0.032	0.050	0.33	0.19	0.050	0.15

Table 15. DLs and LCMRLs (µg/L) Calculated from Replicate Analyses of Fortified Reagent Water Samples Analyzed in Full Scan GC/MS Mode

Analytes	Laboratory 1 Oasis HLB SPE[a,b]			Laboratory 2 Empore PSDVB SPE[c]		
	DL	Fortified conc. of DL replicates	LCMRL	DL	Fortified conc. of DL replicates	LCMRL
cycloate	0.039	0.050	0.27	0.018	0.025	0.14
dacthal (DCPA)	0.018	0.050	0.44	0.0055	0.025	0.040
DDD, 4,4′-	0.019	0.050	0.35	0.0031	0.025	0.092
DDE, 4,4′-	0.033	0.050	0.32	0.0023	0.025	0.088
DDT, 4,4′-	0.036	0.050	0.25	0.022	0.025	0.12
DEET	0.019	0.050	0.14	0.0042	0.025	0.066
di(2-ethylhexyl)adipate	0.079	0.10	0.38	0.013	0.025	0.083
di(2-ethylhexyl)phthalate	ND	0.050	0.29	0.020	0.025	0.40
dibenzo[*a*,*h*]anthracene	0.014	0.050	0.30	0.014	0.025	0.14
dibutyl phthalate	ND	0.050	0.88	0.13	0.025	1.4
dichlorvos	0.021	0.050	0.46	0.0051	0.025	0.081
dieldrin	0.064	0.10	0.28	0.017	0.025	0.084
diethylphthalate	0.037	0.050	0.074	0.018	0.025	0.13
dimethipin	0.045	0.050	0.27	ND	0.025	NC
dimethylphthalate	0.026	0.050	0.11	0.0048	0.025	0.046
DIMP	0.039	0.050	0.19	0.0036	0.025	>5.0
dinitrotoluene, 2,4-	0.026	0.050	0.23	0.0076	0.025	0.19
dinitrotoluene, 2,6-	0.056	0.10	0.31	0.053	0.075	0.20
diphenamid	0.033	0.050	0.29	0.010	0.025	0.089
disulfoton	0.12	0.10	0.12	0.0066	0.025	0.11
endosulfan I	0.039	0.050	0.62	0.015	0.050	0.11
endosulfan II	0.14	0.10	0.38	0.024	0.050	NC
endosulfan sulfate	0.068	0.10	0.39	0.025	0.050	0.12
endrin	0.045	0.050	0.40	0.033	0.050	0.12
EPTC	0.037	0.050	0.13	0.0048	0.025	0.068
ethion	0.010	0.050	0.29	0.013	0.025	0.13
ethoprop	0.13	0.10	0.39	0.0048	0.025	0.12
ethyl parathion	0.15	0.10	0.46	0.013	0.050	0.19

Table 15. DLs and LCMRLs (µg/L) Calculated from Replicate Analyses of Fortified Reagent Water Samples Analyzed in Full Scan GC/MS Mode

Analytes	Laboratory 1 Oasis HLB SPE[a,b]			Laboratory 2 Empore PSDVB SPE[c]		
	DL	Fortified conc. of DL replicates	LCMRL	DL	Fortified conc. of DL replicates	LCMRL
etridiazole	0.024	0.050	0.24	0.012	0.025	0.17
fenarimol	0.10	0.10	0.28	0.041	0.050	0.20
fluorene	0.016	0.050	0.08	0.0058	0.025	0.051
fluridone	0.11	0.10	0.33	0.045	0.050	0.27
HCCPD	0.012	0.050	0.24	0.0066	0.025	0.084
HCH, α	0.036	0.050	0.31	0.0037	0.025	0.062
HCH, β	0.10	0.10	0.24	0.021	0.050	0.11
HCH, δ	0.019	0.050	0.31	0.015	0.025	0.099
HCH, γ (lindane)	0.036	0.050	0.24	0.014	0.025	0.078
heptachlor	0.034	0.050	0.27	0.0032	0.025	0.029
heptachlor epoxide	0.039	0.10	0.11	0.0053	0.025	0.063
hexachlorobenzene	0.016	0.050	0.40	0.0094	0.025	0.087
hexazinone	0.025	0.050	0.26	0.012	0.025	0.14
indeno[1,2,3-*c*,*d*]pyrene	0.11	0.10	0.40	0.039	0.025	0.20
isophorone	0.014	0.050	0.11	0.0043	0.025	0.063
methoxychlor	0.024	0.050	0.23	0.0064	0.025	0.061
methyl parathion	0.036	0.050	0.28	0.0088	0.050	0.16
metolachlor	0.019	0.050	0.11	0.0042	0.025	0.14
metribuzin	0.036	0.050	0.076	0.0070	0.025	0.11
mevinphos	0.035	0.10	0.21	0.0040	0.025	0.093
MGK 264(a)	0.072	0.080	0.32	0.0026	0.025	0.031
MGK 264(b)	0.023	0.020	0.09	0.0096	0.025	0.083
molinate	0.062	0.10	0.29	0.0036	0.025	0.074
napropamide	0.077	0.10	0.44	0.025	0.050	0.11
nitrofen	0.093	0.10	0.39	0.042	0.050	0.13
nonachlor, trans	0.035	0.050	0.35	0.0062	0.025	0.049
norflurazon	0.057	0.10	0.26	0.062	0.050	0.14
oxyfluorfen	0.037	0.050	0.34	0.015	0.050	0.28

Table 15. DLs and LCMRLs (µg/L) Calculated from Replicate Analyses of Fortified Reagent Water Samples Analyzed in Full Scan GC/MS Mode

Analytes	Laboratory 1 Oasis HLB SPE[a,b]			Laboratory 2 Empore PSDVB SPE[c]		
	DL	Fortified conc. of DL replicates	LCMRL	DL	Fortified conc. of DL replicates	LCMRL
pebulate	0.020	0.050	0.20	0.0053	0.025	0.040
pentachlorophenol	0.060	0.20	0.56	0.069	0.025	0.16
permethrin, cis	0.015	0.028	0.13	0.0026	0.025	0.036
permethrin, trans	0.031	0.072	0.10	0.012	0.025	0.087
phenanthrene	0.025	0.10	0.097	0.0069	0.025	0.51
phorate	0.032	0.050	0.20	0.052	0.050	0.15
phosphamidon	0.040	0.10	0.23	0.0029	0.025	0.11
profenofos	0.11	0.10	0.31	0.055	0.050	0.11
prometon	0.065	0.10	0.22	0.010	0.025	0.12
prometryn	0.021	0.050	0.24	0.012	0.025	0.10
pronamide	0.017	0.050	0.13	0.0077	0.025	0.13
propachlor	0.029	0.050	0.27	0.0076	0.025	0.068
propazine	0.023	0.050	0.20	0.0065	0.025	0.055
pyrene	0.013	0.050	0.17	0.0045	0.025	0.062
simazine	0.023	0.050	0.12	0.0059	0.025	0.083
simetryn	0.037	0.050	0.28	0.013	0.025	0.12
tebuconazole	0.24	0.50	2.1	0.053	0.050	0.18
tebuthiuron	0.027	0.050	0.21	0.060	0.075	0.13
terbacil	0.082	0.10	0.41	0.039	0.075	0.13
terbutryn	0.029	0.050	0.18	0.0051	0.025	0.093
tetrachlorvinphos	0.048	0.10	0.35	0.0042	0.025	0.14
triadimefon	0.053	0.050	0.35	0.048	0.050	0.11
tribufos+merphos	0.12	0.10	0.53	0.013	0.025	0.18
trifluralin	0.027	0.050	0.28	0.019	0.025	0.12
vernolate	0.038	0.050	0.11	0.0051	0.025	0.080
vinclozolin	0.029	0.050	0.34	0.016	0.050	0.12
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	0.021	0.050	0.14	0.0061	0.025	0.054

Table 15. DLs and LCMRLs (µg/L) Calculated from Replicate Analyses of Fortified Reagent Water Samples Analyzed in Full Scan GC/MS Mode

Analytes	Laboratory 1 Oasis HLB SPE[a,b]			Laboratory 2 Empore PSDVB SPE[c]		
	DL	Fortified conc. of DL replicates	LCMRL	DL	Fortified conc. of DL replicates	LCMRL
4-chlorobiphenyl (3)	0.019	0.050	0.24	0.0059	0.025	0.065
2,4′-dichlorobiphenyl (8)	0.024	0.050	0.18	0.0037	0.025	0.088
2,2′,5-trichlorobiphenyl (18)	0.024	0.050	0.086	0.028	0.025	0.14
2,4,4′-trichlorobiphenyl (28)	0.036	0.050	0.069	0.0073	0.025	0.036
2,2′,3,5′-tetrachlorobiphenyl (44)	0.029	0.050	0.23	0.0079	0.025	0.041
2,2′,5,5′-tetrachlorobiphenyl (52)	0.043	0.10	0.092	0.0055	0.025	0.078
2,3′,4′,5-tetrachloroobiphenyl (70)	0.043	0.050	0.30	0.0044	0.025	0.046
2,3,3′,4′,6-pentachlorobiphenyl (110)	0.026	0.050	0.36	0.0030	0.025	0.084
2,3′,4,4′,5-pentachlorobiphenyl (118)	0.046	0.050	0.36	0.0050	0.025	0.095
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	0.19	0.10	0.41	0.0071	0.025	0.058
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	0.020	0.050	0.37	0.0039	0.025	0.056
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	0.029	0.050	0.38	0.0033	0.025	0.092
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	0.058	0.10	0.31	0.0066	0.025	0.036

a. DL calculated from eight replicates.
b. Data obtained on the instrumentation described in Sect. 13.1.1.1.
c. Data obtained on the instrumentation described in Sect. 13.1.1.2.
d. NC= Not calculated. A sufficient number of data points were not available to calculate a valid LCMRL.
e. ND= Not determined. DLs were not determined when a response was not detected in all replicates at a given concentration, or when a high background concentration or an interference was present that prevented accurate quantitation at the fortified concentration.

Table 16. Example SIM Parameters for Selected Method Analytes

SIM Segment	Compound	RT (min)	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Dwell Time (ms)	Scan Time (sec/scan)
# 1	1,3-dimethyl-2-nitrobenzene (SUR)	8.94	77	134	80	0.49
	HCCPD	11.03	237	235, 239		
# 2	acenaphthene-d_{10} (IS #1)	13.05	162	164	80	0.58
	2-chlorobiphenyl	13.26	188	152		
# 3	molinate	13.72	126	55	225	0.49
# 4	4-chlorobiphenyl	14.26	188	152	100	0.59
	ethoprop	14.81	97	139, 158		
# 5	α-HCH	15.55	181	183, 219	50	0.60
	2,4'-dichlorobiphenyl	15.61	222	152, 224		
	hexachlorobenzene	15.62	284	142, 249		
# 6	simazine	15.99	201	173, 186	25	0.57
	dimethipin	16.04	54	53		
	atrazine	16.10	200	215		
	pentachlorophenol	16.15	266	264, 268		
	^{13}C-pentachlorophenol (IS #4)	16.17	276	--		
	γ-HCH (lindane)	16.27	183	181, 219		
# 7	2,2',5-trichlorobiphenyl	16.50	256	186	75	0.37
	phenanthrene-d_{10} (IS #2)	16.55	188	160*		
# 8	acetochlor	17.48	146	162, 223	40	0.62
	2,4,4'-trichlorobiphenyl	17.52	186	256		
	vinclozolin	17.59	212	124		
	alachlor	17.66	188	160		
	heptachlor	17.84	100	272		
# 9	2,2',5,5'-tetrachlorobiphenyl	18.25	220	292	60	0.61
	metolachlor	18.50	162	238		
	2,2',3,5'-tetrachlorobiphenyl	18.69	220	255, 292		
# 10	heptachlor epoxide	19.50	353	81, 355	50	0.47
	2,3',4',5-tetrachlorobiphenyl	19.62	220	292		
# 11	trans-chlordane	20.02	375	373, 377	50	0.60
	cis-chlordane	20.35	375	373, 377		
	trans-nonachlor	20.42	409	407, 411		

Table 16. Example SIM Parameters for Selected Method Analytes

SIM Segment	Compound	RT (min)	QI (*m/z*)	Confirmation Ion(s) (*m/z*)	Dwell Time (ms)	Scan Time (sec/scan)
# 12	profenofos	20.77	339	97, 139	45	0.61
	tribufos	20.91	57	169		
	oxyfluorfen	21.02	252	361		
	2,3,3',4',6-pentachlorobiphenyl	21.03	326	254, 256		
# 13	endrin	21.46	263	81, 281	50	0.60
	2,2',3,4',5',6-hexachlorobiphenyl	21.57	360	218, 290		
	2,3',4,4',5-pentachlorobiphenyl	21.68	326	254, 256		
	toxaphene, peak 1	21.74	159	125, 305*		
	2,2',4,4',5,5'-hexachlorobiphenyl	22.18	360	218, 290		
	toxaphene, peak 2	22.47	305	125		
	toxaphene, peak 3	22.67	159	125, 305*		
	2,2',3,4,4',5'-hexachlorobiphenyl	22.81	360	218, 290		
# 14	tebuconazole	23.12	125	83, 250	50	0.60
	di(2-ethylhexyl)adipate	23.17	129	57, 70		
	triphenyl phosphate (SUR)	23.26	77	169, 325		
# 15	chrysene-d_{12} (IS #3)	24.03	240	236*	75	0.55
	methoxychlor	24.18	227	--		
	2,2',3,4,4',5,5'-heptachlorobiphenyl	24.44	394	252, 324		
# 16	toxaphene, peak 4	24.55	159	125, 305*	125	0.57
	di(2-ethylhexyl)phthalate	24.72	149	167		
	c-permethrin	26.42	183	163*		
	t-permethrin	26.60	183	163*		
# 17	benzo(a)pyrene-d_{12} (SUR)	27.90	264	132*	125	0.57
	benzo(a)pyrene	28.03	252	126*		

* Confirmation ions may be at or below 30% relative abundance depending on instrument tune.

Table 17. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. 0.025 µg/L[b] n=4		Fortified Conc. 0.10 µg/L[c] n=4		Fortified Conc. 0.25 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	Mean % Recovery	Mean % Recovery	RSD
acetochlor	103	1.8	109	1.9	105	2.2
alachlor	114	2.7	112	1.1	104	3.1
atrazine	117	5.7	118	3.6	111	1.8
benzo[*a*]pyrene	81.0	4.8	103	2.2	97.4	7.5
chlordane, cis	110	4.1	102	2.9	100	3.0
chlordane, trans	89.9	1.4	99.4	5.3	100	2.8
di(2-ethylhexyl)adipate	ND[e]		114	4.2	111	1.7
di(2-ethylhexyl)phthalate	ND		113	33	118	7.8
dimethipin	91.5	4.1	108	7.4	112	3.2
endrin	97.5	6.1	105	3.7	100	1.6
ethoprop	89.4	19	109	3.2	112	1.7
HCCPD	102	7.6	108	1.2	100	4.3
HCH, α	112	5.6	114	3.3	99.6	4.5
HCH, γ (lindane)	104	6.9	110	2.0	100	6.2
heptachlor	104	9.3	99.9	3.4	89.2	2.7
heptachlor epoxide	108	3.8	103	4.6	98.2	3.3
hexachlorobenzene	90.2	9.6	95.3	2.1	90.6	2.7
methoxychlor	113	8.4	116	2.8	104	1.6
metolachlor	114	1.0	115	4.3	107	1.7
molinate	104	5.7	109	2.3	109	1.4
nonachlor, trans	98.9	2.8	96.4	5.5	99.9	3.4
oxyfluorfen	114	12	115	5.2	124	6.3
pentachlorophenol	79.0	7.9	96.3	6.7	97.2	2.0
permethrin, cis	91.5	9.7	113	3.2	105	2.0
permethrin, trans	94.6	2.5	104	2.0	103	1.5
profenofos	102	8.4	92.0	2.3	112	1.7
simazine	85.2	12	101.3	13.0	124	1.9
tebuconazole	82.6	7.4	116.0	1.9	117	0.69
tribufos	96.2	7.8	101.2	3.5	105	3.9
vinclozolin	93.1	4.4	110	2.0	104	2.4

Table 17. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using Oasis HLB SPE Cartridges; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. 0.025 µg/L[b] n=4		Fortified Conc. 0.10 µg/L[c] n=4		Fortified Conc. 0.25 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	Mean % Recovery	Mean % Recovery	RSD
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	99.9	2.7	106	1.1	98.8	2.6
4-chlorobiphenyl (3)	96.8	4.8	105	1.0	109	1.8
2,4′-dichlorobiphenyl (8)	109	12	110	7.1	102	2.4
2,2′,5-trichlorobiphenyl (18)	101	6.1	106	3.0	99.9	0.82
2,4,4′-trichlorobiphenyl (28)	90.8	10.0	105	1.7	101	1.3
2,2′,3,5′-tetrachlorobiphenyl (44)	94.5	4.2	108	5.7	98.5	3.1
2,2′,5,5′-tetrachlorobiphenyl (52)	97.0	4.6	105	1.3	101	2.5
2,3′,4′,5-tetrachloroobiphenyl (70)	88.3	15	103	6.5	100	2.5
2,3,3′,4′,6-pentachlorobiphenyl (110)	92.0	3.7	99.9	4.9	99.2	1.6
2,3′,4,4′,5-pentachlorobiphenyl (118)	86.2	8.8	96.5	3.7	97.6	2.5
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	86.7	3.7	96.1	4.4	96.3	1.5
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	95.3	4.7	97.4	3.1	95.4	2.3
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	86.8	2.0	98.7	0.79	97.2	1.4
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	99.7	9.5	97.6	4.3	95.7	2.0
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	101	2.2	105	3.6	93.6	4.4
benzo[*a*]pyrene-d_{12}	87.9	3.0	92.1	2.4	94.5	8.8
triphenyl phosphate	103	1.9	106	3.2	101	1.4

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 0.10 µg/L, tebuconazole is 0.12 µg/L, c-permethrin is 0.014 µg/L, and t-permethrin is 0.036 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 0.40 µg/L, tebuconazole is 0.50 µg/L, c-permethrin is 0.056 µg/L, and t-permethrin is 0.144 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 1.0 µg/L, tebuconazole is 1.02 µg/L, c-permethrin is 0.14 µg/L, and t-permethrin is 0.36 µg/L.
e. ND = Not determined. Analyte could not be determined because of the low concentration relative to the LRB.

Table 18. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acetochlor	0.10	109	9.6	NA[e]	
alachlor	0.10	110	5.9	112	3.6
atrazine	0.10	112	11	101	0.45
benzo[*a*]pyrene	0.10	86.3	2.7	104	7.6
chlordane, cis	0.10	98.1	3.4	97.9	3.9
chlordane, trans	0.10	101	4.3	102	3.7
di(2-ethylhexyl)adipate	0.10	116	7.4	114	4.8
di(2-ethylhexyl)phthalate	0.10	ND[f]		ND	
dimethipin	0.10	96.0	7.4	103	18
endrin	0.10	112	4.4	107	3.3
ethoprop	0.10	111	9.6	102	3.6
HCCPD	0.10	122	7.4	82.8	7.3
HCH, α	0.10	111	8.4	84.5	7.2
HCH, γ (lindane)	0.10	100	5.5	108	4.6
heptachlor	0.10	90.9	5.9	85.3	5.8
heptachlor epoxide	0.10	96.5	4.5	103	1.4
hexachlorobenzene	0.10	90.4	4.9	83.8	3.3
methoxychlor	0.10	116	4.1	128	1.3
metolachlor	0.10	112	6.3	NA	
molinate	0.10	120	7.2	NA	
nonachlor, trans	0.10	93.3	3.8	NA	
oxyfluorfen	0.10	122	6.5	123	4.6
pentachlorophenol	0.40	90.5	8.2	94.9	0.88
permethrin, cis	0.06[g]	106	5.0	102	5.1
permethrin, trans	0.14[h]	101	3.6	108	3.0
profenofos	0.10	107	3.5	110	6.8
simazine	0.10	101	10	105	3.0
tebuconazole	0.50	115	5.6	99.9	4.5
tribufos	0.10	107	5.9	110	3.5

Table 18. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using Oasis HLB SPE Cartridges; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
vinclozolin	0.10	103	8.3	104	4.2
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	0.10	87.8	9.6	95.8	4.5
4-chlorobiphenyl (3)	0.10	104	6.4	96.5	3.5
2,4′-dichlorobiphenyl (8)	0.10	110	5.9	90.0	4.9
2,2′,5-trichlorobiphenyl (18)	0.10	93.0	4.0	92.7	3.2
2,4,4′-trichlorobiphenyl (28)	0.10	97.2	6.1	91.4	2.1
2,2′,3,5′-tetrachlorobiphenyl (44)	0.10	96.8	3.9	81.6	5.2
2,2′,5,5′-tetrachlorobiphenyl (52)	0.10	94.2	4.4	95.3	5.0
2,3′,4′,5-tetrachloroobiphenyl (70)	0.10	95.4	4.9	98.8	0.85
2,3,3′,4′,6-pentachlorobiphenyl (110)	0.10	92.8	4.6	97.5	3.0
2,3′,4,4′,5-pentachlorobiphenyl (118)	0.10	91.5	5.6	92.3	3.0
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	0.10	101	4.6	96.9	3.5
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	0.10	93.6	4.4	96.6	1.5
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	0.10	92.1	4.8	94.5	0.86
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	0.10	92.1	3.7	92.6	2.1
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	1.0	106	5.4	95.3	5.7
benzo[*a*]pyrene-d_{12}	1.0	84.4	2.6	100	5.6
triphenyl phosphate	1.0	103	2.7	106	2.0

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 393 mg/L as calcium carbonate.
c. Tap water from a surface water source. TOC of 2.0 mg/L.
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.
e. NA = Not analyzed. Matrix sample was not fortified with this analyte.
f. ND = Not determined. Recovery could not be determined because high LRB concentrations were observed relative to the fortified concentration.
g. The concentration in ground water is as listed in the table. The concentration in surface water is 0.030 µg/L.
h. The concentration in ground water is as listed in the table. The concentration in surface water is 0.070 µg/L.

Table 19. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker H_2O Phobic Speedisks; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. 0.025µg/L[b] n=4		Fortified Conc. 0.10 µg/L[c] n=4		Fortified Conc. 0.25 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	Mean % Recovery	Mean % Recovery	RSD
acetochlor	99.0	1.7	98.7	1.9	98.8	0.67
alachlor	111	2.3	100	1.1	100	1.0
atrazine	106	5.0	98.6	6.0	99.3	1.2
benzo[*a*]pyrene	89.6	2.4	93.3	2.9	91.8	2.3
chlordane, cis	100	1.3	98.0	2.4	99.2	1.6
chlordane, trans	100	0.60	96.5	2.5	97.5	1.6
di(2-ethylhexyl)adipate	102	1.1	94.9	5.1	96.2	2.8
di(2-ethylhexyl)phthalate	ND[e]		ND		ND	
dimethipin	86.0	9.9	105	6.9	98.8	1.5
endrin	105	3.6	105	1.5	104	0.76
ethoprop	104	4.1	101	2.7	98.0	1.0
HCCPD	85.8	5.1	86.6	3.5	87.4	1.3
HCH, α	101	2.0	96.8	2.0	96.2	2.0
HCH, γ (lindane)	102	1.8	96.3	3.3	97.0	2.1
heptachlor	96.7	3.2	90.6	3.4	89.9	1.5
heptachlor epoxide	103	2.0	99.3	1.9	101	1.9
hexachlorobenzene	94.7	2.1	88.0	2.2	91.2	1.8
methoxychlor	104	2.9	101	4.0	101	2.1
metolachlor	104	1.5	101	2.3	101	1.3
molinate	110	13	95.7	2.6	96.1	1.3
nonachlor, trans	99.5	1.0	97.3	2.6	97.4	1.6
oxyfluorfen	107	1.4	107	5.7	105	2.2
pentachlorophenol	99.5	1.4	93.6	2.4	92.8	1.6
permethrin, cis	97.0	5.0	95.6	5.4	93.1	2.4
permethrin, trans	95.8	2.5	95.5	5.1	96.0	2.1
profenofos	109	1.7	105	3.6	105	1.5
simazine	105	0.71	99.7	5.1	100	0.74
tebuconazole	125	2.8	114	4.1	112	2.3
tribufos	95.0	5.2	96.9	4.3	98.7	1.2
vinclozolin	89.2	6.7	82.7	6.3	87.1	6.6

Table 19. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using J.T. Baker H_2O Phobic Speedisks; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. 0.025µg/L[b] n=4		Fortified Conc. 0.10 µg/L[c] n=4		Fortified Conc. 0.25 µg/L[d] n=4	
	Mean % Recovery	RSD	Mean % Recovery	Mean % Recovery	Mean % Recovery	RSD
PCB congeners (by IUPAC#)	96.4	1.6	94.5	2.8	95.3	2.9
2-chlorobiphenyl (1)	97.3	1.7	93.4	2.5	93.2	1.1
4-chlorobiphenyl (3)	95.0	1.8	93.2	2.9	92.4	1.8
2,4′-dichlorobiphenyl (8)	97.2	2.0	94.6	2.7	93.7	1.5
2,2′,5-trichlorobiphenyl (18)	94.8	1.2	92.9	2.3	94.8	1.3
2,4,4′-trichlorobiphenyl (28)	98.4	2.2	94.3	2.3	95.5	1.7
2,2′,3,5′-tetrachlorobiphenyl (44)	96.5	2.2	93.6	2.4	95.2	1.5
2,2′,5,5′-tetrachlorobiphenyl (52)	97.6	1.8	95.7	2.4	96.8	3.6
2,3′,4′,5-tetrachloroobiphenyl (70)	98.7	1.4	95.0	2.3	96.1	1.4
2,3,3′,4′,6-pentachlorobiphenyl (110)	97.9	1.5	95.4	2.5	96.3	1.7
2,3′,4,4′,5-pentachlorobiphenyl (118)	101	1.1	91.9	2.3	94.6	2.8
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	96.8	1.3	94.7	2.4	95.2	1.7
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	95.9	1.0	94.2	2.4	94.5	1.9
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	92.8	1.5	87.9	2.1	87.1	2.6
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	96.2	2.5	96.4	1.2	95.4	2.4
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	89.0	2.2	92.0	1.8	91.6	2.1
benzo[*a*]pyrene-d_{12}	100	2.6	101	2.0	100	2.9
triphenyl phosphate	99.0	1.7	98.7	1.9	98.8	0.67

a. Data obtained on the instrumentation described in Sect. 13.1.1.2.
b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 0.10 µg/L, c-permethrin is 0.014 µg/L, and t-permethrin is 0.036 µg/L.
c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 0.40 µg/L, c-permethrin is 0.058 µg/L, and t-permethrin is 0.14 µg/L.
d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 1.0 µg/L, pentachlorophenol is 1.0 µg/L, c-permethrin is 0.14 µg/L, and t-permethrin is 0.36 µg/L.
e. ND = Not determined. Matrix spike recovery could not be determined due to the low fortification concentration relative to the background concentration.

Table 20a. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using J.T. Baker H_2O Phobic Speedisks; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery [c]	RSD
acetochlor	NA[d]				
alachlor	0.10	90.4	6.6	104	1.4
atrazine	0.10	112	5.3	125	0.94
benzo[*a*]pyrene	0.10	84.8	5.1	83.1	6.1
chlordane, cis	0.10	91.0	4.5	98.4	1.8
chlordane, trans	0.10	90.4	4.1	98.5	1.8
di(2-ethylhexyl)adipate	0.10	67.0	7.9	81.0	8.2
di(2-ethylhexyl)phthalate	0.10	ND[e]		ND[e]	
dimethipin	0.10	69.6	9.7	70.8	6.7
endrin	0.10	95.3	4.2	108	2.9
ethoprop	0.10	91.0	4.4	109	1.8
HCCPD	0.10	82.4	8.9	163	4.2
HCH, α	0.10	90.9	4.2	96.9	1.7
HCH, γ (lindane)	0.10	88.2	4.8	91.9	0.73
heptachlor	0.10	90.6	5.1	101	1.3
heptachlor epoxide	0.10	93.8	4.3	104	1.5
hexachlorobenzene	0.10	87.3	5.6	90.8	1.9
methoxychlor	0.10	98.9	5.6	121	1.8
metolachlor	NA				
molinate	NA				
nonachlor, trans	NA				
oxyfluorfen	0.10	100	6.2	141	1.3
pentachlorophenol	0.40	90.2	5.5	89.0	1.8
permethrin, cis	0.058	87.1	4.5	108	4.3
permethrin, trans	0.14	86.3	4.9	105	3.5
profenofos	0.10	96.8	5.5	119	2.1
simazine	0.10	84.9	7.1	97.2	2.7
tebuconazole	0.50	109	5.6	138	2.5
tribufos	0.10	95.9	9.0	117	3.2

Table 20a. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using J.T. Baker H_2O Phobic Speedisks; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery [c]	RSD
vinclozolin	0.10	90.3	4.9	104	1.5
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	0.10	85.8	4.7	88.1	1.4
4-chlorobiphenyl (3)	0.10	85.3	4.8	89.1	1.7
2,4′-dichlorobiphenyl (8)	0.10	87.0	4.7	89.9	1.8
2,2′,5-trichlorobiphenyl (18)	0.10	90.2	2.2	90.4	1.3
2,4,4′-trichlorobiphenyl (28)	0.10	87.7	4.3	91.0	1.8
2,2′,3,5′-tetrachlorobiphenyl (44)	0.10	87.7	3.9	89.9	2.0
2,2′,5,5′-tetrachlorobiphenyl (52)	0.10	87.3	4.0	90.8	1.8
2,3′,4′,5-tetrachloroobiphenyl (70)	0.10	84.1	3.6	88.2	2.1
2,3,3′,4′,6-pentachlorobiphenyl (110)	0.10	84.8	3.4	88.7	2.2
2,3′,4,4′,5-pentachlorobiphenyl (118)	0.10	83.6	2.4	88.1	3.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	0.10	77.4	2.6	79.9	4.3
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	0.10	81.6	2.7	84.7	3.6
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	0.10	76.9	2.9	77.8	4.7
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	0.10	67.9	4.4	67.9	8.7
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	1.0	102	1.0	109	0.60
benzo[*a*]pyrene-d_{12}	5.0	119	2.0	112	5.0
triphenyl phosphate	1.0	99.1	3.4	110	2.6

a. Data obtained on the instrumentation described in Sect. 13.1.1.2.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 325 mg/L as calcium carbonate, TOC was 0.73 mg/L.
c. Tap water from a surface water source. Tap water hardness was 137 mg/L as calcium carbonate, TOC was 2.52 mg/L.
d. NA = Not analyzed. Matrix sample was not fortified with this analyte.
e. ND = Not determined. Recovery could not be determined because high LRB concentrations were observed relative to the fortified concentration.

Table 20b. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using J.T. Baker H_2O Phobic Speedisks; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery [c]	RSD
acetochlor	NA[d]				
alachlor	0.50	90.8	1.1	95.8	3.3
atrazine	0.50	90.5	0.85	92.1	3.7
benzo[*a*]pyrene	0.50	92.3	2.8	79.6	6.6
chlordane, cis	0.50	92.5	1.1	94.6	2.8
chlordane, trans	0.50	91.9	0.98	94.1	2.6
di(2-ethylhexyl)adipate	0.50	73.5	1.1	76.3	9.9
di(2-ethylhexyl)phthalate	0.50	ND[e]		ND[e]	
dimethipin	0.50	72.3	6.0	69.7	7.8
endrin	0.50	96.1	1.3	102	3.0
ethoprop	0.50	91.7	1.6	97.7	2.9
HCCPD	0.50	89.1	2.2	114	1.6
HCH, α	0.50	92.4	1.4	92.6	3.6
HCH, γ (lindane)	0.50	91.3	1.0	90.7	3.0
heptachlor	0.50	92.8	1.2	97.7	2.3
heptachlor epoxide	0.50	94.6	1.4	97.6	2.7
hexachlorobenzene	0.50	89.3	1.7	90.0	3.2
methoxychlor	0.50	99.7	1.3	104	2.6
metolachlor	NA				
molinate	NA				
nonachlor, trans	NA				
oxyfluorfen	0.50	93.0	0.83	107	2.7
pentachlorophenol	2.0	92.3	1.3	89.7	2.8
permethrin, cis	0.15	81.1	1.4	86.1	4.4
permethrin, trans	0.35	82.2	0.97	84.9	3.8
profenofos	0.10	93.3	2.1	99.4	3.0
simazine	0.50	82.8	1.0	85.0	3.3
tebuconazole	2.5	96.5	1.3	103	2.7
tribufos	0.50	91.4	1.0	98.0	2.5

Table 20b. Precision and Accuracy Data Obtained for Selected Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using J.T. Baker H_2O Phobic Speedisks; N=4 for Each Matrix; SIM GC/MS Analyses[a]

Analytes	Fortified Conc. (µg/L)	Ground Water[b]		Surface Water[c]	
		Mean % Recovery[d]	RSD	Mean % Recovery [c]	RSD
vinclozolin	0.50	91.3	1.0	94.7	2.7
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	0.50	89.7	1.8	87.8	4.7
4-chlorobiphenyl (3)	0.50	87.7	1.9	87.5	3.7
2,4′-dichlorobiphenyl (8)	0.50	89.7	1.9	89.4	3.3
2,2′,5-trichlorobiphenyl (18)	0.50	90.9	1.5	90.2	3.0
2,4,4′-trichlorobiphenyl (28)	0.50	90.6	1.1	90.8	2.9
2,2′,3,5′-tetrachlorobiphenyl (44)	0.50	90.5	1.1	90.0	3.1
2,2′,5,5′-tetrachlorobiphenyl (52)	0.50	90.2	1.4	90.1	2.9
2,3′,4′,5-tetrachloroobiphenyl (70)	0.50	90.6	1.7	88.8	3.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	0.50	88.5	2.2	86.6	3.7
2,3′,4,4′,5-pentachlorobiphenyl (118)	0.50	86.1	1.2	86.0	3.2
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	0.50	80.6	0.86	78.9	3.1
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	0.50	83.5	0.84	81.5	3.0
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	0.50	79.1	0.82	76.1	3.2
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	0.50	71.0	0.72	64.9	2.8
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	1.0	103	0.65	108	0.93
benzo[*a*]pyrene-d_{12}	5.0	117	2.6	112	5.1
triphenyl phosphate	1.0	102	1.8	112	0.84

a. Data obtained on the instrumentation described in Sect. 13.1.1.2.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 325 mg/L as calcium carbonate, TOC was 0.73 mg/L.
c. Tap water from a surface water source. Tap water hardness was 137 mg/L as calcium carbonate, TOC was 2.52 mg/L.
d. NA = Not analyzed. Matrix sample was not fortified with this analyte.
e. ND = Not determined. Recovery could not be determined because high LRB concentrations were observed relative to the fortified concentration.

Table 21. Precision and Accuracy Data for Toxaphene Extracted from Fortified Reagent Water; Sample Extracts Analyzed in SIM GC/MS Mode

Sorbent	Fortified Conc. 1.0 µg/L n=4		Fortified Conc. 10 µg/L n=4		Fortified Conc. 25 µg/L n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
Oasis-HLB[a]	82.9	5.0	101	3.3	89.5	8.8
J.T. Baker H_2O Phobic cartridge[a]	100	3.63	104	3.8	98.5	1.7
J.T. Baker H_2O Phobic Speedisk[b]	114	1.0	103	3.4	98.5	2.8
Empore SDB-XC[a]	107	5.3	111	2.1	101	0.15
UCT 525 Universal Cartridge[a]	NA[c]		111	1.8	NA	

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Data obtained on the instrumentation described in Sect. 13.1.1.2.
c. NA = Not analyzed. Data using the UCT 525 Universal cartridge was obtained at a single concentration of 10 µg/L.

Table 22. Precision and Accuracy Data for Toxaphene Extracted from Fortified Drinking Water Samples; Sample Extracts Analyzed in SIM GC/MS Mode

Sorbent	Ground Water [a] Fortified Conc. 10 µg/L, n=4		Surface Water [b] Fortified Conc. 10 µg/L, n=4	
	Mean % Recovery	RSD	Mean % Recovery	RSD
Oasis-HLB[c]	95.4	3.7	94.2	5.9
J.T. Baker H_2O Phobic cartridge[c]	102	5.6	103	3.5
J.T. Baker H_2O Phobic Speedisk[d]	120	2.4	124	1.9
Empore SDB-XC[c]	106	4.6	104	0.46

a. Tap water from a ground water source with high mineral content. Hardness was determined to be 323-360 mg/L as calcium carbonate.
b. Tap water from surface water sources. Fortified water samples extracted on Oasis-HLB cartridges had a TOC of 2.05 mg/L. Fortified water samples extracted on other sorbents had a TOC of 3.12 mg/L.
c. Data obtained on the instrumentation described in Sect. 13.1.1.1.
d. Data obtained on the instrumentation described in Sect. 13.1.1.2.

Table 23. DLs and LCMRLs Calculated from Replicate Analyses of Fortified Reagent Water Samples Extracted on Oasis HLB Cartridges, and LCMRLs Calculated from Replicate Analyses of Fortified Reagent Water Samples Extracted on J.T. Baker H_2O Phobic Speedisks, All Samples Analyzed in SIM Mode, All concentrations in µg/L

Analytes	Oasis HLB[a]			Baker Speedisk[b]
	DL[c]	Fortified conc. of DL replicates	LCMRL	LCMRL
acetochlor	0.0047	0.010	0.011	0.0091
alachlor	0.0045	0.010	0.016	0.013
atrazine	0.0062	0.010	0.023	0.015
benzo[*a*]pyrene	0.0069	0.010	0.036	0.0060
chlordane, cis	0.0016	0.010	0.0073	0.0039
chlordane, trans	0.0028	0.010	0.0097	0.0020
di(2-ethylhexyl)adipate	ND[d]		0.38	0.020
di(2-ethylhexyl)phthalate	ND		0.43	ND
dimethipin	0.0093	0.010	0.022	0.065
endrin	0.0081	0.010	0.014	0.011
ethoprop	0.0082	0.010	0.036	0.0084
HCCPD	0.0055	0.010	0.014	0.0026
HCH, α	0.0068	0.010	0.021	0.0040
HCH, γ (lindane)	0.0040	0.010	0.031	0.0071
heptachlor	0.0034	0.010	0.010	0.0024
heptachlor epoxide	0.0026	0.010	0.017	0.0031
hexachlorobenzene	0.0092	0.010	0.014	0.0065
methoxychlor	0.0025	0.010	0.021	0.0011
metolachlor	0.0035	0.010	0.019	0.004
molinate	0.0057	0.010	0.010	0.029
nonachlor, trans	0.0029	0.010	0.0061	0.0066
oxyfluorfen	0.0038	0.010	0.035	0.031
pentachlorophenol	0.047	0.100	0.068	0.0086
permethrin, cis	0.0041	0.006	0.012	0.0015
permethrin, trans	0.0080	0.014	0.020	0.0023
profenofos	0.0081	0.010	0.029	0.012
simazine	0.010	0.025	0.048	0.018
tebuconazole	0.042	0.12	0.20	0.037

Table 23. DLs and LCMRLs Calculated from Replicate Analyses of Fortified Reagent Water Samples Extracted on Oasis HLB Cartridges, and LCMRLs Calculated from Replicate Analyses of Fortified Reagent Water Samples Extracted on J.T. Baker H_2O Phobic Speedisks, All Samples Analyzed in SIM Mode, All concentrations in µg/L

Analytes	Oasis HLB[a]			Baker Speedisk[b]
	DL[c]	Fortified conc. of DL replicates	LCMRL	LCMRL
toxaphene	0.32	1.0	1.4	NF[e]
tribufos	0.0033	0.010	0.023	0.0063
vinclozolin	0.0064	0.010	0.0098	0.095
PCB congeners (by IUPAC#)				
2-chlorobiphenyl (1)	0.0024	0.010	0.0080	0.0063
4-chlorobiphenyl (3)	0.0049	0.010	0.0073	0.0056
2,4′-dichlorobiphenyl (8)	0.0045	0.010	0.023	0.0022
2,2′,5-trichlorobiphenyl (18)	0.0038	0.010	0.013	0.0012
2,4,4′-trichlorobiphenyl (28)	0.0056	0.010	0.014	0.0084
2,2′,3,5′-tetrachlorobiphenyl (44)	0.0063	0.010	0.025	0.0020
2,2′,5,5′-tetrachlorobiphenyl (52)	0.0027	0.010	0.0078	0.0012
2,3′,4′,5-tetrachloroobiphenyl (70)	0.013	0.025	0.036	0.0022
2,3,3′,4′,6-pentachlorobiphenyl (110)	0.0030	0.010	0.0092	0.0035
2,3′,4,4′,5-pentachlorobiphenyl (118)	0.0077	0.010	0.013	0.0026
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	0.0081	0.010	0.016	0.015
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	0.0063	0.010	0.012	0.0021
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	0.0092	0.010	0.013	0.0038
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	0.0029	0.010	0.015	0.0026

a. Data obtained on the instrumentation described in Sect. 13.1.1.1.
b. Data obtained on the instrumentation described in Sect. 13.1.1.2.
c. DL calculated from eight replicates.
d. ND = Not determined. DLs and LCMRLs were not determined when a high background concentration or an interference was present that prevented accurate quantitation at the fortified concentration.
e. NF = Not fortified.

Table 24. Initial Demonstration of Capability (IDC) and Quality Control (QC) Requirements (Summary)

Method Reference	Requirement	Specification and Frequency	Acceptance Criteria
Sect. 9.2.1 & 9.3.1	Initial Demonstration of Low Background	Analyze LRB prior to any other IDC steps. When a new lot of SPE media is obtained, verify that background is at acceptable limits.	Demonstrate that the method analytes are ≤ 1/3 the MRL, and that possible interferences from extraction media do not prevent the identification and/or quantification of any analytes, SURs or ISs. **Note:** This includes the absence of interferences at both the QIs and confirmation ions at the RTs of interest.
Sect. 9.2.2	Initial Demonstration of Precision (IDP)	Analyze 4-7 replicate LFBs fortified near the midrange calibration concentration.	%RSD must be ≤ 20%
Sect. 9.2.3	Initial Demonstration of Accuracy (IDA)	Calculate average recovery for replicates used in IDP.	Mean recovery ± 30% of the true value for all analytes except dimethipin, HCCPD and HCB which must be within 60-130% of the true value
Sect. 9.2.4	Minimum Reporting Limit (MRL) Confirmation	Fortify, extract and analyze 7 replicate LFBs at the proposed MRL concentration. Calculate the mean, standard deviation and HR_{PIR} for each analyte. Confirm that the upper and lower limits for the Prediction Interval of Result (Upper PIR, and Lower PIR, Sect. 9.2.4.2) meet the recovery criteria.	Upper PIR ≤ 150% Lower PIR ≥ 50%
Sect. 9.2.5 & 9.3.9	Calibration Confirmation, Quality Control Sample (QCS)	Analyze a standard from a second source (QCS) to verify the initial calibration curve.	± 30% of the expected value.

NOTE: Table 24 is intended as an abbreviated summary of QC requirements provided as a convenience to the method user. Because the information has been abbreviated to fit the table format, there may be issues that need additional clarification, or areas where important additional information from the method text is needed. In all cases, the full text of the QC in Sect. 9 supersedes any missing or conflicting information in this table.

Table 25. Ongoing Quality Control (QC) Requirements (Summary)

Method Reference	Requirement	Specification and Frequency	Acceptance Criteria
Sect. 8.4	Sample Holding Time	14 days for most analytes with appropriate preservation and storage as described in Sects. 8.1-8.3. See Sect 8.4 for exceptions.	Sample results are valid only if samples are extracted within sample hold time.
Sect. 8.4	Extract Holding Time	28 days stored at -5 °C and protected from light	Sample results are valid only if extracts are analyzed within extract hold time.
Sect. 9.3.1	Laboratory Reagent Blank (LRB)	One LRB with each extraction batch of up to 20 Field Samples.	Demonstrate that the method analyte concentration is $\leq {}^{1}/_{3}$ the MRL, and confirm that possible interferences do not prevent quantification. If the background concentration exceeds $^{1}/_{3}$ the MRL, results for the extraction batch are invalid.
Sect. 9.3.3	Laboratory Fortified Blank (LFB)	One LFB is required for each extraction batch of up to 20 Field Samples. Rotate the fortified concentrations between low, medium, and high amounts.	Results of LFB analyses at medium and high fortifications must be ± 30% of the true value for all analytes except dimethipin, HCCPD and HCB which may be 60-130% of the true value. Results of the low-level LFB must be ± 50% of the true value.
Sect. 9.3.5	Internal Standard (IS)	Compare IS area to the mean IS area from the analysis of each CAL in the initial calibration and the area in the most recent CCC.	Peak area counts for all ISs in all injections must be within ± 50% of their mean peak area calculated during the initial calibration. Peak areas of ISs 1-3 must also be ± 30% from the most recent CCC. If the ISs do not meet these criteria, target analyte results are invalid. Consult Sect. 9.3.5 for further information.
Sect. 9.3.6	Surrogate (SUR) Standards	The SUR standards are added to all calibration standards and samples, including QC samples prior to extraction. Calculate SUR recoveries.	SUR recovery must be ± 30% of the true value. If any SUR fails this criterion, report all results for sample as suspect/SUR recovery.
Sect. 9.3.7	Laboratory Fortified Sample Matrix (LFSM)	Analyze one LFSM per extraction batch (of up to 20 Field Samples) fortified with the method analytes at a concentration close to but greater than the native concentration. Calculate LFSM recoveries.	See Sect. 9.3.7.3 for instructions on the interpretation of LFSM results.

Table 25. Ongoing Quality Control (QC) Requirements (Summary) (Continued)

Sect. 9.3.8	Laboratory Fortified Sample Matrix Duplicate (LFSMD) or Field Duplicates (FD)	Extract and analyze at least one FD or LFSMD with each extraction batch of up to 20 Field Samples. An LFSMD may be substituted for a FD when the frequency of detects for analytes of interest are low. Calculate RPDs.	Method analyte RPDs for the LFSMD or FD should be ≤ 30% at mid and high levels of fortification and ≤ 50% at concentrations within 2 times the MRL. Failure to meet this criterion may indicate a matrix effect.
Sect. 9.3.9	Quality Control Sample (QCS)	Analyze a QCS during the IDC, and each time new CAL solutions or PDSs are prepared. A QCS must be analyzed at least quarterly.	Results must be ±30% of the expected value.
Sect. 10.2	Initial Calibration	Use the IS calibration technique to generate a linear or quadratic calibration curve for each analyte. A minimum of six standards should be used for a calibration range of two orders of magnitude. Suggested concentrations can be found in Sect. 7.2.4. Check the calibration curve against the acceptance criteria in Sect. 10.2.6.	When each calibration standard is calculated as an unknown using the calibration curve, the result should be ± 30% of the true value for all except the lowest standard, which should be ± 50% of the true value. If this criterion is not met, reanalyze CALs, select a different method of calibration or recalibrate over a shorter range.
Sects. 10.1 and 10.2.1	MS Tune Check	Analyze DFTPP to verify the MS tune after instrument maintenance and each time the instrument is mass calibrated. The MS tune must also be verified prior to analyzing CAL stds and establishing calibration curves for method analytes.	Acceptance criteria are given in Table 2.
Sect. 10.3	Continuing Calibration Check (CCC)	Verify initial calibration by analyzing a calibration standard at the beginning of each analysis batch prior to analyzing samples, after every 10 Field Samples, and after the last sample of each analysis batch. The first CCC daily must be at or below the MRL. Subsequent CCCs alternate between medium and high concentrations. Low CCC – at or below the MRL concentration Mid CCC – near midpoint in the initial calibration curve High CCC – near the highest calibration standard.	Low: ±50% of true value Mid: ±30% of true value High: ±30% of true value

Note: Table 25 is intended as an abbreviated summary of QC requirements provided as a convenience to the method user. Because the information has been abbreviated to fit the table format, there may be issues that need additional clarification, or areas where important additional information from the method text is needed. In all cases, the full text of Sects. 8-10 in the method supersedes any missing or conflicting information in this table.

Figure 1a. Example chromatogram of a calibration standard (concentration of 5 ng/µL injected for most analytes). Peak identification numbers correspond to those in Table 1.

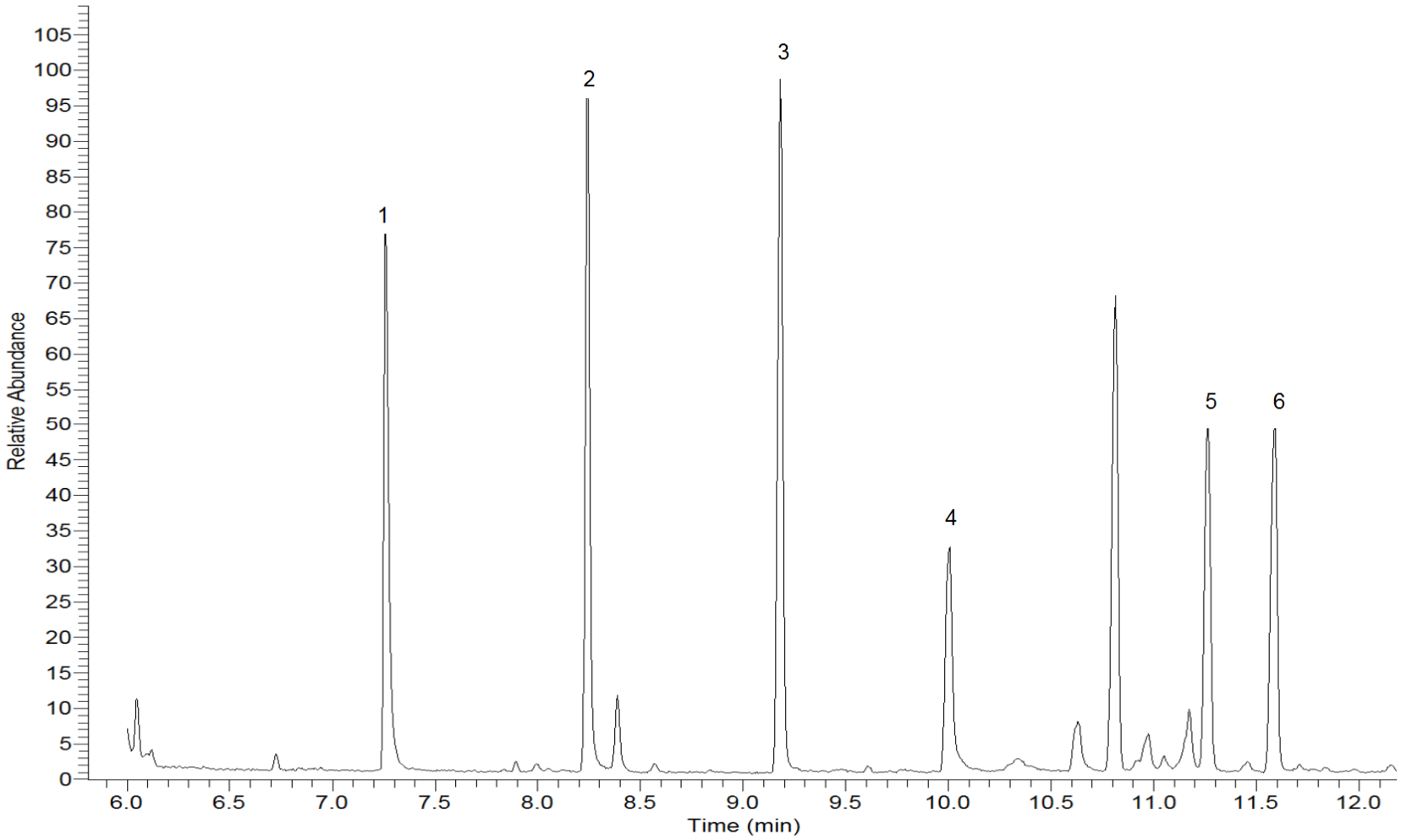

Figure 1b. Example chromatogram of a calibration standard (concentration of 5 ng/µL injected for most analytes). Peak identification numbers correspond to those in Table 1.

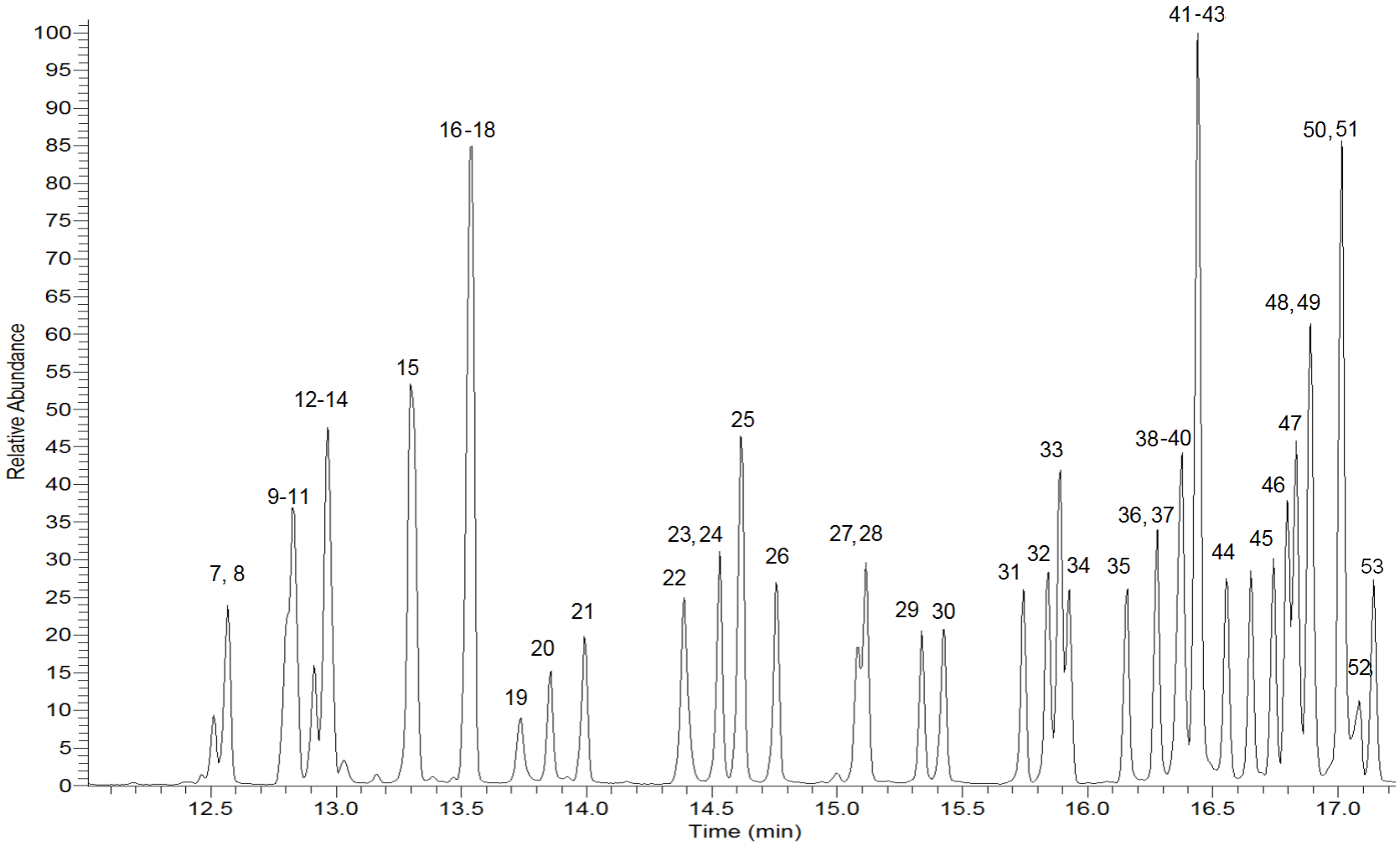

Figure 1c. Example chromatogram of a calibration standard (concentration of 5 ng/µL injected for most analytes). Peak identification numbers correspond to those in Table 1.

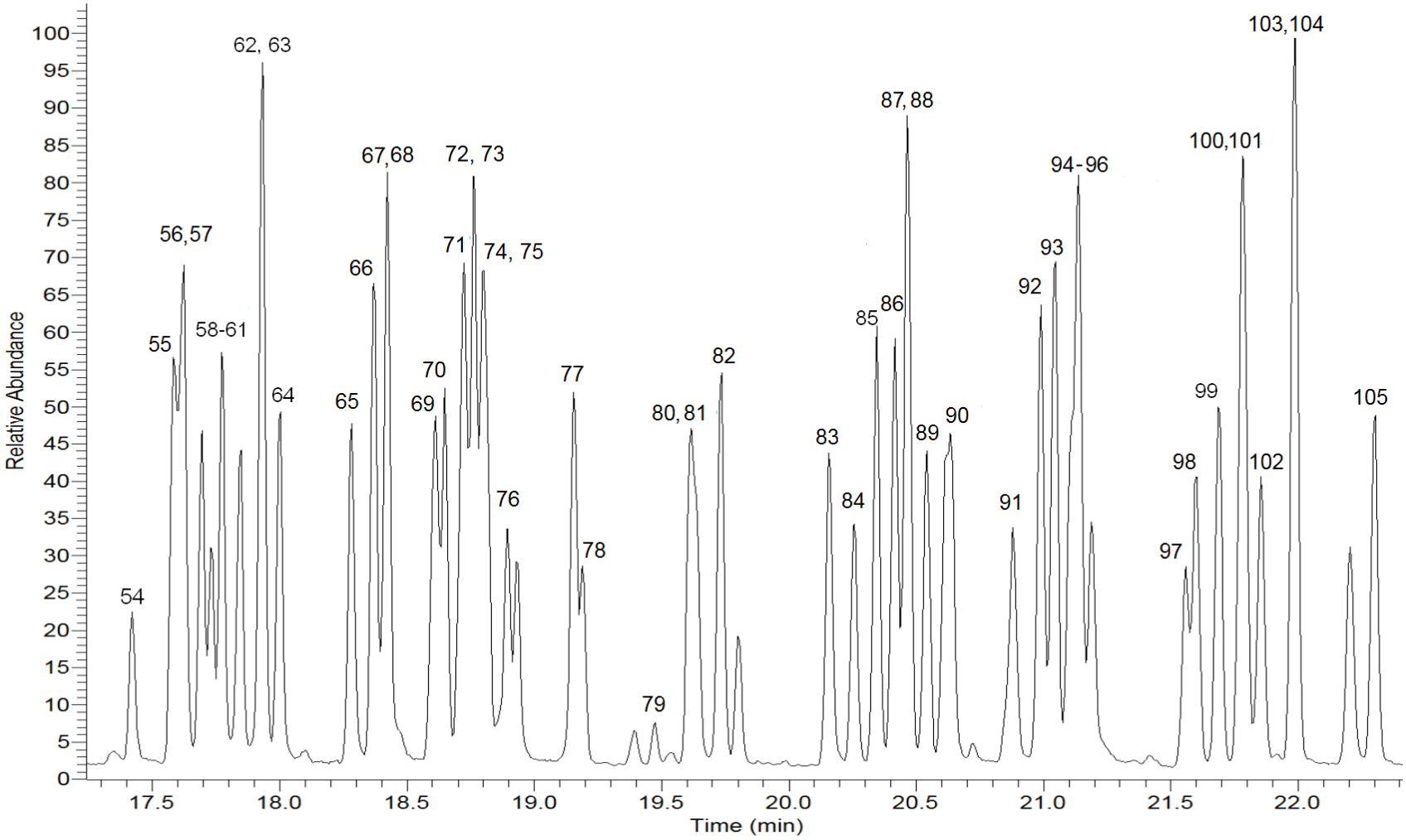

Figure 1d. Example chromatogram of a calibration standard (concentration of 5 ng/µL injected for most analytes). Peak identification numbers correspond to those in Table 1.

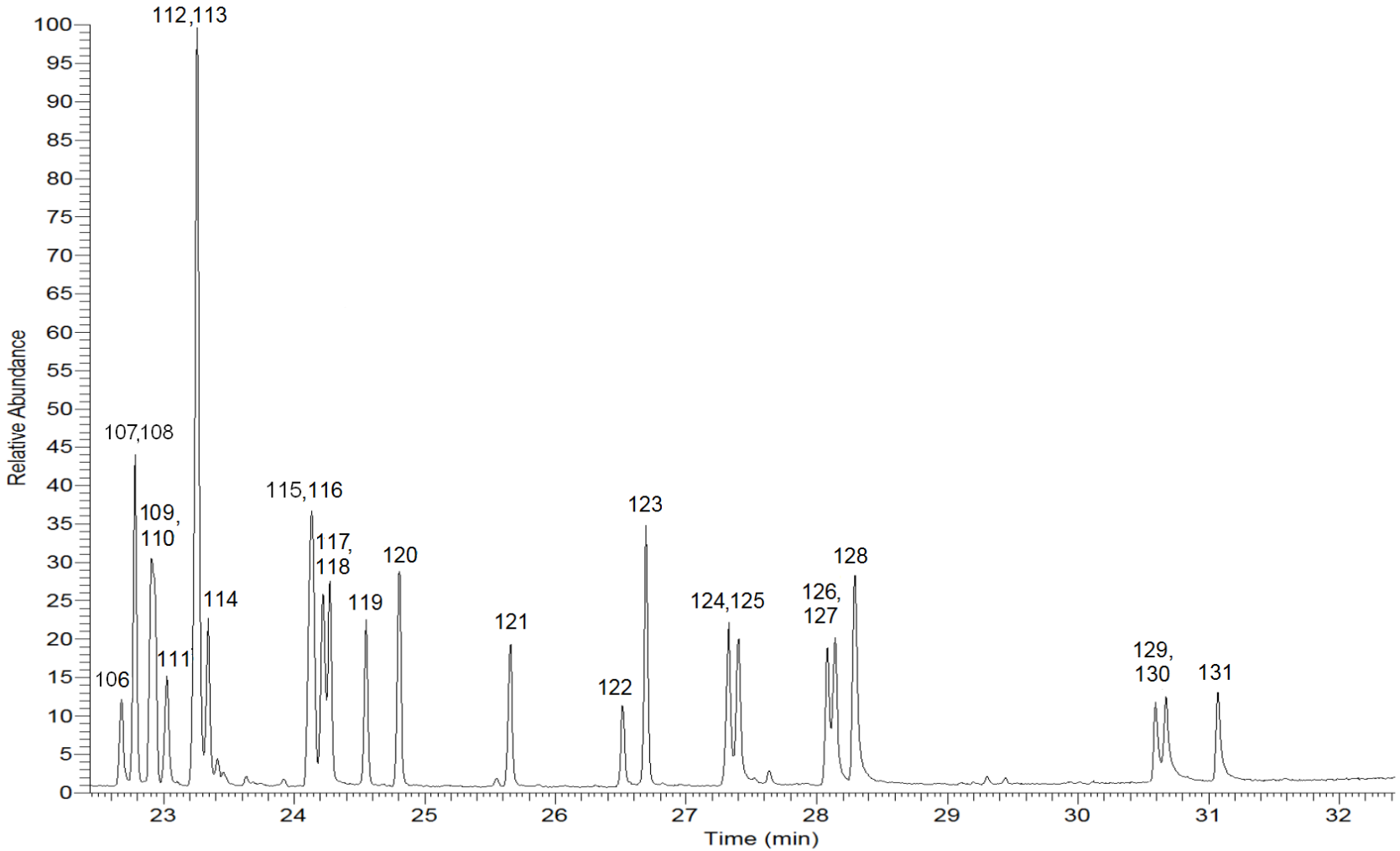

Figure 2. Example extracted ion current profiles of toxaphene at m/z 125, 305, and 159.

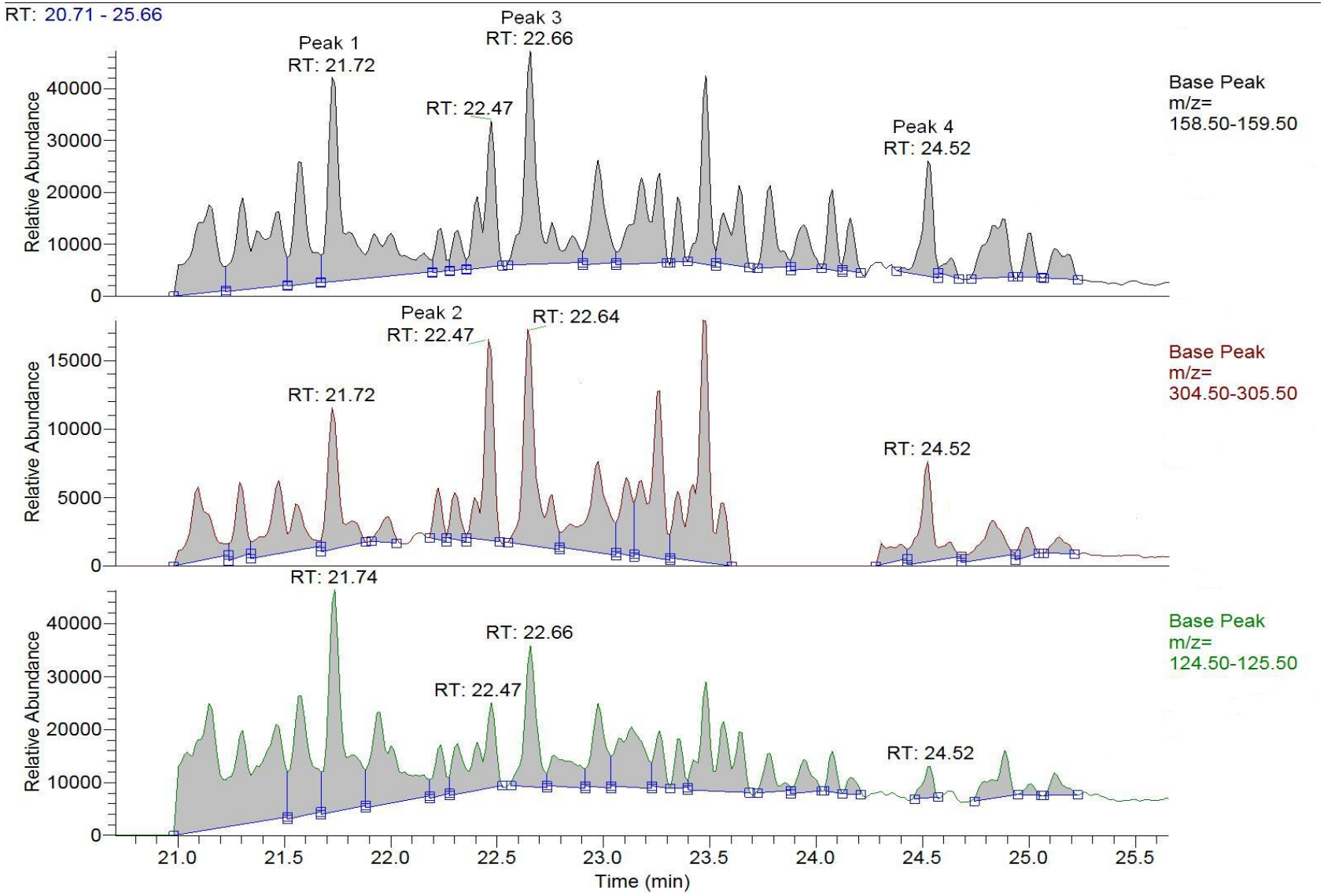

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Figure 3a. Results of Aqueous Holding Time Study (Sect. 13.4)

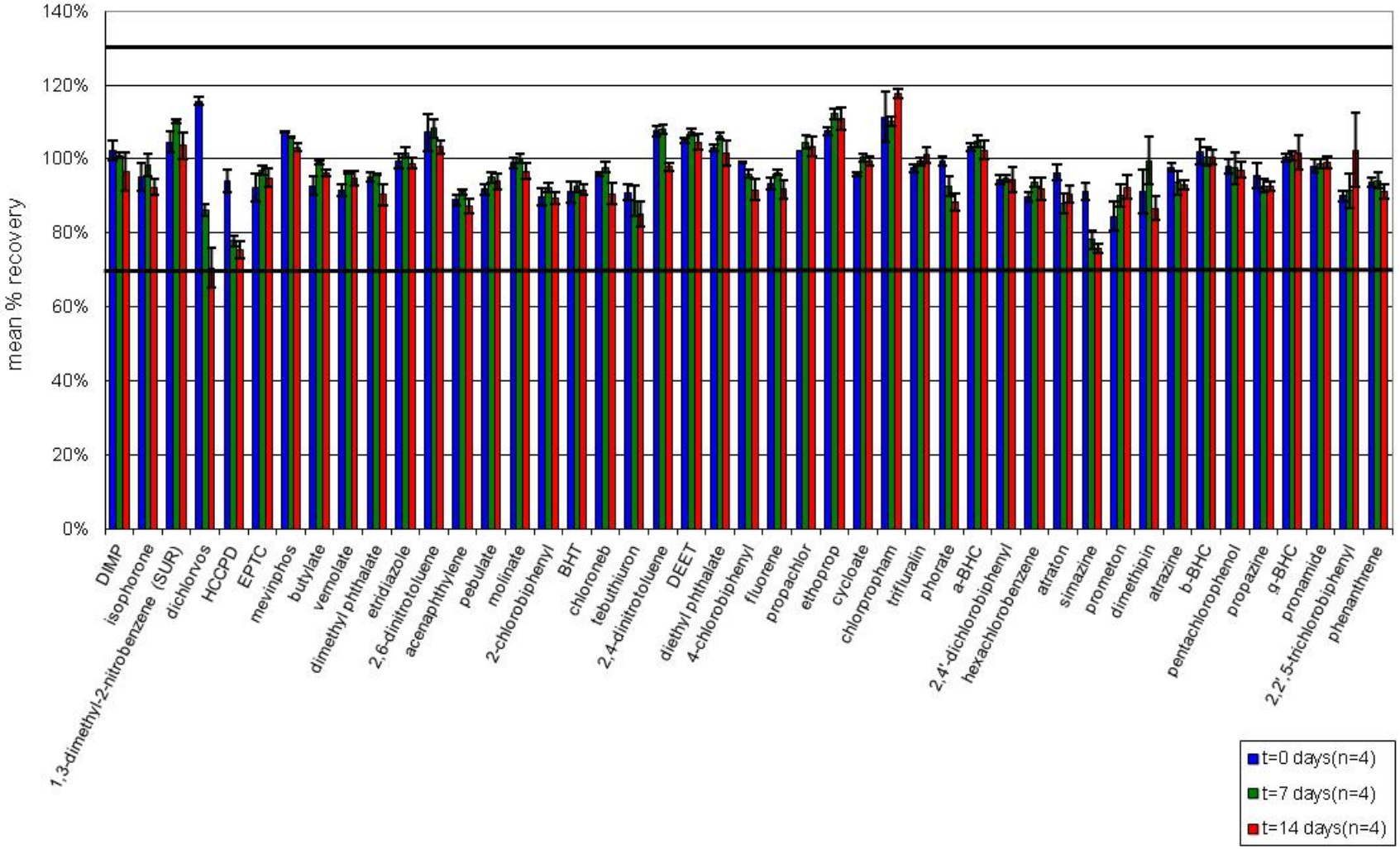

Figure 3b. Results of Aqueous Holding Time Study (Sect. 13.4)

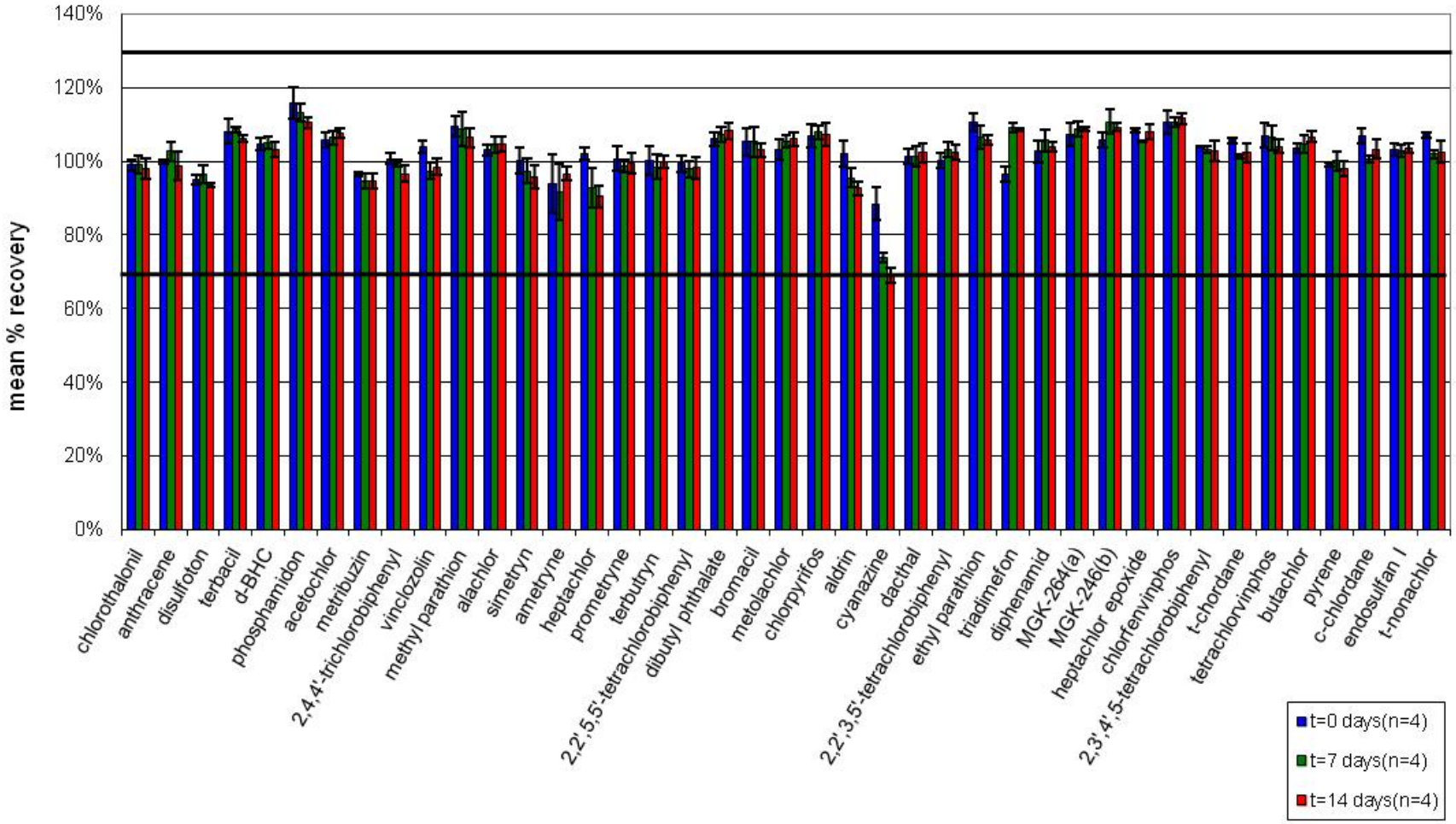

Figure 3c. Results of Aqueous Holding Time Study (Sect. 13.4)

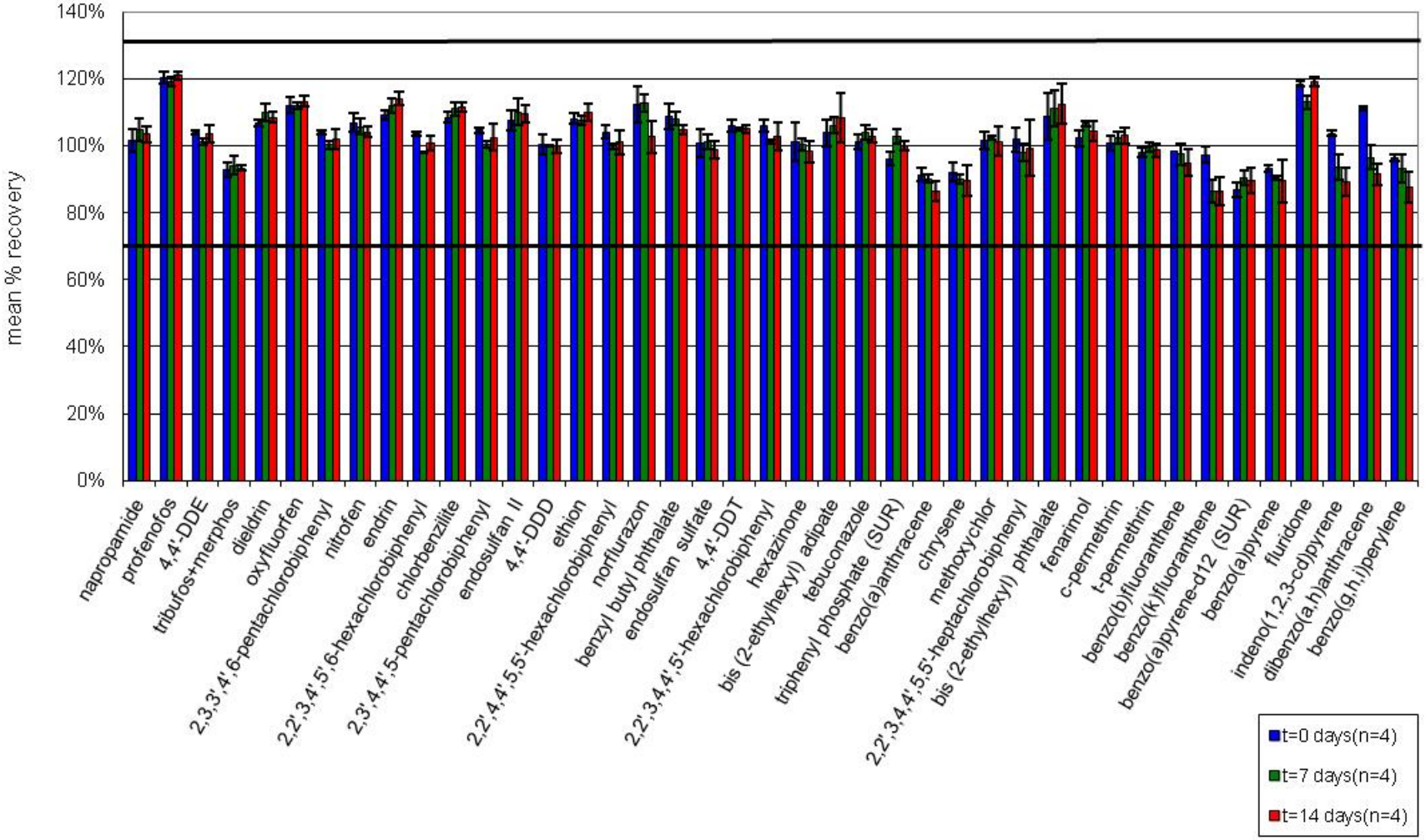

Figure 4a. Results of Extract Holding Time Study (Sect. 13.5)

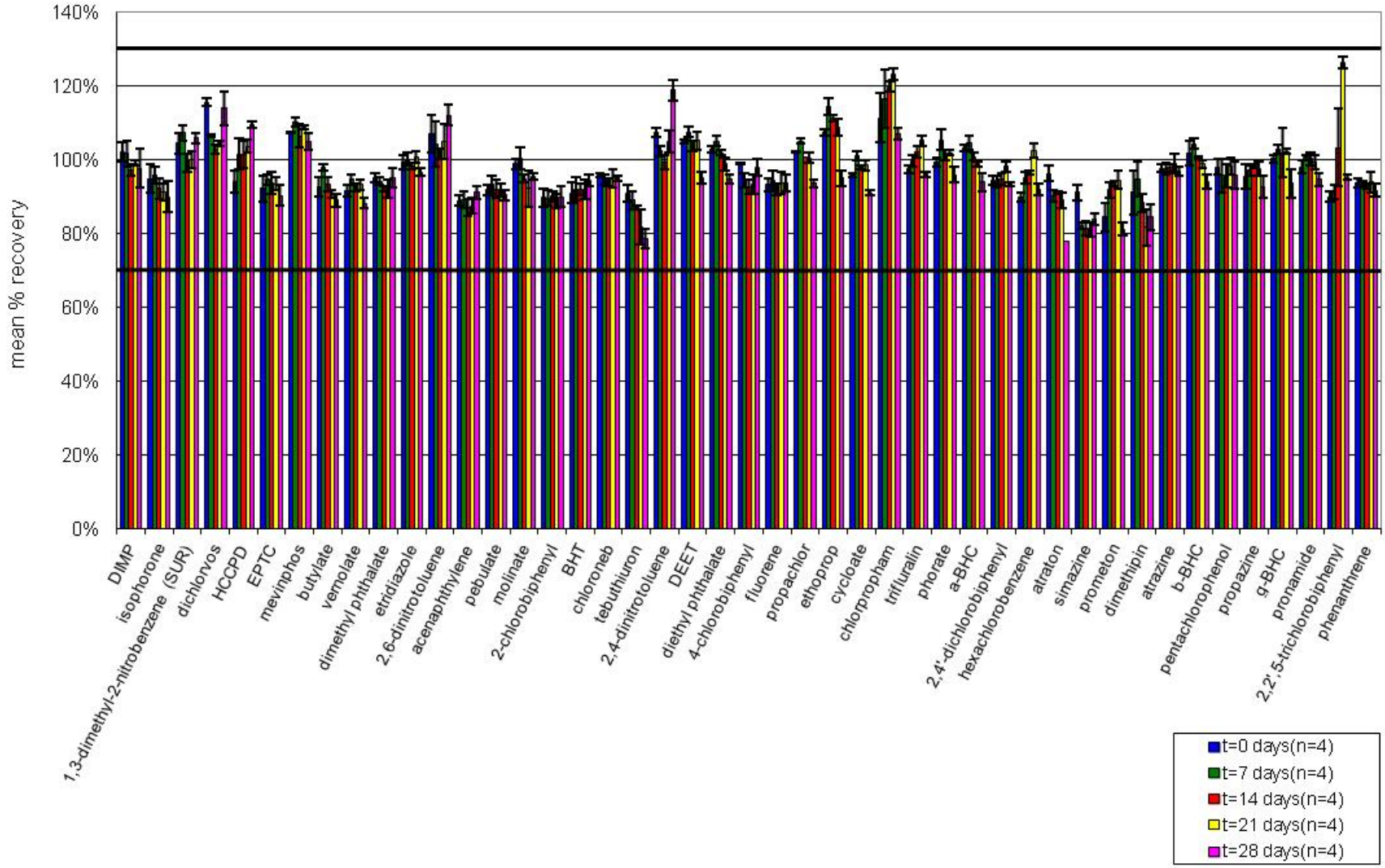

Figure 4b. Results of Extract Holding Time Study (Sect. 13.5)

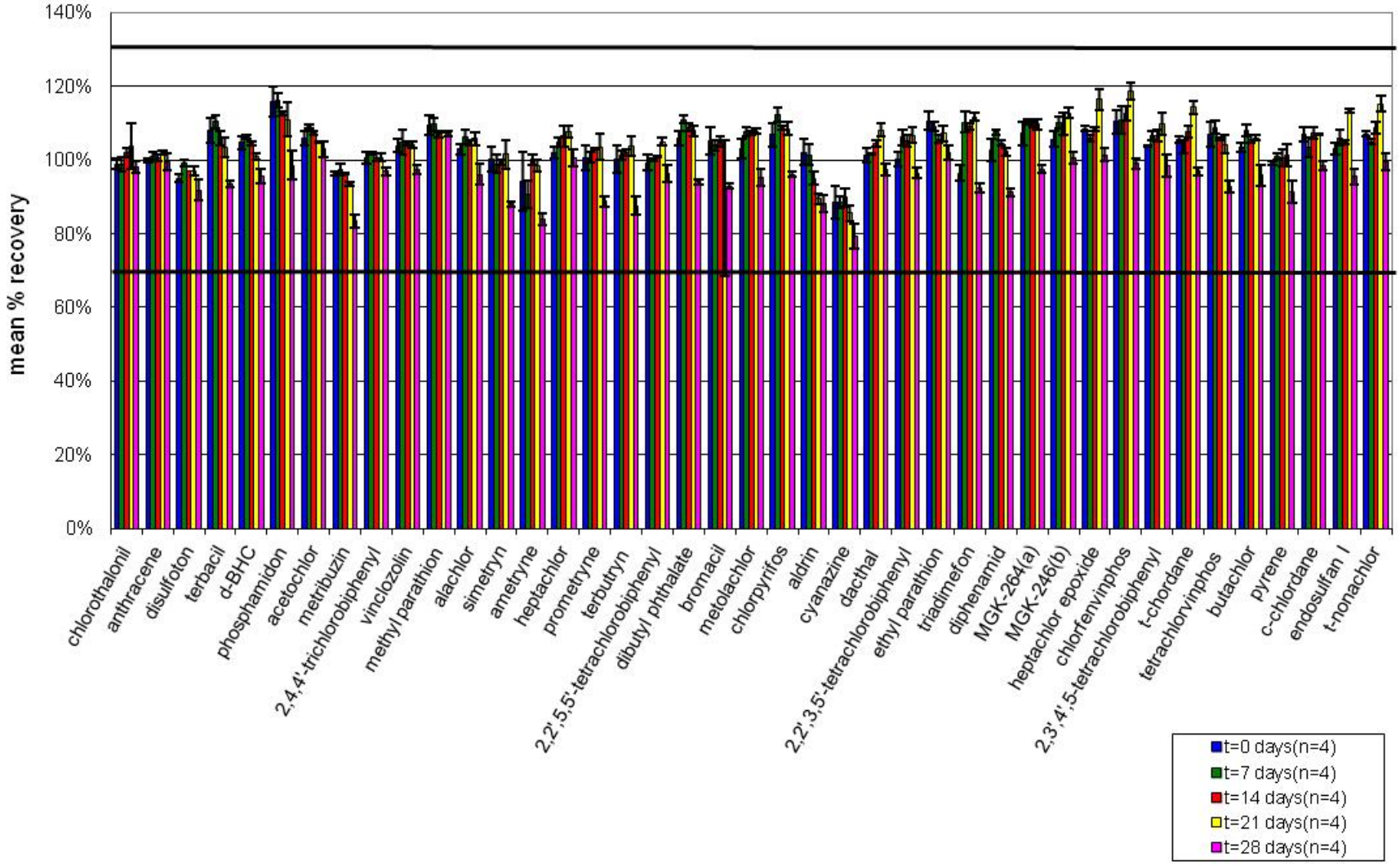

Figure 4c. Results of Extract Holding Time Study (Sect. 13.5)