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METHOD 3511

ORGANIC COMPOUNDS IN WATER BY MICROEXTRACTION

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria or for the purpose of laboratory accreditation.

1.0 SCOPE AND APPLICATION

For a summary of changes in this version, please see Appendix A at the end of this document.

1.1 Method 3511 is a procedure for extracting selected volatile and semivolatile organic compounds from water. The microscale approach minimizes sample size and solvent usage, thereby reducing the supply costs, health and safety risks, and waste generated.

1.2 This method was validated for several mono- and poly-cyclic aromatic hydrocarbons (MAHs and PAHs) and can be applied to any combination of these compounds.

1.3 This method also may be used to extract selected volatile organic compounds (VOCs) or semivolatile organic compounds (SVOCs) which are slightly soluble or insoluble in water at neutral pH if the extraction performance is demonstrated to be satisfactory using an appropriate analytical technique.

1.4 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly required in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained personnel. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Samples are prepared by liquid:liquid extraction with organic solvent in field sampling containers. Careful manipulation of the sample, solvent, and spiking solutions during the procedure minimizes loss of volatile compounds while maximizing extraction of volatile, semivolatile, and nonvolatile compounds.

2.2 Since volatile compounds are included in the method, their extraction from water requires special handling and particular attention to detail. Samples should be removed from the refrigerator shortly before extraction, and should not be allowed to warm to room temperature before the extraction solvent is added.

2.3 Samples should be prepared one at a time to the point of solvent addition (i.e., do not prepare a number of samples then add the solvent). Pay particular attention to minimizing the exposure of the sample and/or extract to air.

2.4 Determinative analysis is performed using the appropriate GC or GC/MS method (*e.g.*, 8015, 8021 and 8270). Reference 2 shows an example of a GC configuration used for the entire list of target analytes that may help reduce solvent tailing to prevent interference with any early-eluting target compounds in this method.

3.0 DEFINITIONS

Refer to the SW-846 chapter of terms and acronyms for potentially applicable definitions.

4.0 INTERFERENCES

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

4.2 Refer to Method 3500 for additional information on interferences.

5.0 SAFETY

There are no significant safety issues specific to this method. However, SW-846 methods do not purport to address all safety issues associated with their use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

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6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 VOA vials - 40-mL capacity, disposable, pre-cleaned with Polytetrafluoroethylene (PTFE)-lined caps

6.2 Vials - Amber glass, 2-mL capacity, with PTFE-lined screw or crimp top

6.3 Centrifuge - Capable of at least 500 G's

6.4 Syringes - gastight, contaminant-free, 2.0-mL, 1.0-mL, 10-μL

6.5 Analytical balance - Capable of weighing to 0.01 g

6.6 Pasteur glass pipettes - 1mL, disposable

7.0 REAGENTS AND STANDARDS

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

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

7.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400 °C for four hours in a shallow porcelain bowl. Store unused portion of sodium sulfate in a desiccator or sealed container.

7.4 Extraction and exchange solvents

The choice of solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

7.4.1 Methylene chloride, CH_2Cl_2.

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8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

Samples should be extracted as soon as after collection as possible, but no longer than 14 days from the date of collection for acid preserved samples. If samples are not acidified, the extraction should be performed within 7 days from date of collection.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and the criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency and lower limit of quantitation (LLOQ)

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000D, Sec. 9.3 for information on how to accomplish a demonstration of proficiency.

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

In order to demonstrate the entire sample preparation and analysis process at the lower limit of quantitation (LLOQ), a LLOQ check standard (not part of an initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at the predicted LLOQ concentration level(s). Alternatively, a representative sample matrix may be spiked with the analytes of interest at the predicted

LLOQ concentration levels. The LLOQ check is carried through the same preparation procedures as environmental samples and other QC samples.

Recovery of target analytes in the LLOQ check standard should be within established in-house limits, or other such project-specific acceptance limits, to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% may be used for the LLOQ acceptance criteria. This acknowledges the poorer overall response at the low end of the calibration curve. Historically-based LLOQ acceptance criteria should be determined as soon as practical once sufficient data points have been acquired.

9.4 Refer to Method 3500 for additional quality control procedures.

9.5 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. Each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination.

9.6 Any method blanks, matrix spike samples, or replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.7 Each extraction batch of twenty or fewer samples should include a minimum of a method blank, a laboratory control sample (LCS), a matrix spike sample, and a matrix spike duplicate or laboratory duplicate sample.

9.8 All field and QC samples should be spiked with an appropriate mix of surrogate compounds in order to track extraction efficiency.

9.9 Any QC samples should be subjected to exactly the same analytical procedures as those used on field samples.

10.0 CALIBRATION AND STANDARDIZATION

There are no calibration or standardization steps directly associated with this extraction procedure.

11.0 PROCEDURE

Refer to Sec. 9 for a list of number and types of QC samples that should be included with each batch of 20 or fewer field samples.

11.1 For each QC sample to be extracted and analyzed, prepare one vial as described below (Sec. 11.3).

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11.2 If volatile compounds are target analytes of interest, the QC vials must be prepared far enough in advance that the water has time to chill before extraction. However, these vials should not be allowed to sit in the refrigerator for more than 24 hours, or their integrity may be suspect.

11.3 For each method blank or LCS QC sample, fill a 40 mL glass VOA vial with a PTFE-lined septum screw top with reagent water.

11.4 If volatile compounds are target analytes of interest, make sure that all field and QC samples are chilled to less than 4°C before proceeding.

11.5 Working with a single field or QC sample at a time, remove the cap from the VOA vial, and use a disposable pipette to remove approximately 5 mL of water. Alternatively, remove enough sample volume from the vial to prevent vessel overflow, however, too much volume removed will create a potential for headspace and loss of volatile constituents. In addition, the amount of time used to process volatile compound extracts up to the point of solvent addition needs to be kept to a minimum in order to avoid further volatile constituent losses. Replace the vial cap.

The volume removed should be disposed of according to the guidelines set forth by the laboratory for the disposal of laboratory wastes.

11.6 After removing the 5 mL, weigh the capped VOA vial for each field and QC sample. Record the weight to the nearest 0.1 grams.

11.7 Remove the cap and add 10 μg of the surrogate standard compounds in acetone. The surrogates recommended are fluorobenzene, 2-fluorobiphenyl, and 5-a-androstane. Other compounds may be used as surrogates, depending upon the desired target analytes and project requirements.

11.7.1 It should be noted that more surrogate standard may be added to the water if the sample is suspected to be highly contaminated (i.e., the contamination may interfere with the recovery of lower concentration levels of surrogate standard).

11.7.2 The LCS and matrix spike QC samples should have 10 μg of the appropriate compounds of interest added.

11.8 Add exactly 2.0 mL of methylene chloride (DCM) (using a class A volumetric pipette or gas-tight syringe) and approximately 12 g of anhydrous sodium chloride to the VOA vial. Replace the vial cap. At this point, the next sample may be processed to the point of solvent addition and the subsequent steps performed for each sample batch.

11.9 Shake each vial vigorously for 5 minutes, or until the sodium chloride dissolves completely.

11.10 Allow the phases to separate. Centrifugation may be useful for separating the phases. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst should employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.11 Using a 2.0-mL gas-tight syringe, transfer approximately 1.5 mL of the lower (DCM) layer to a 2 mL vial fitted with a PTFE lined screw cap, taking precautions to exclude any

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water from the syringe. Add a small amount (~50 mg) of anhydrous sodium sulfate to the vial, cap, and shake for 2 minutes.

11.12 Using a 1.0 mL gas-tight syringe, transfer exactly 1.0 mL of the dried extract to a 2 mL vial fitted with a PTFE lined screw cap. Add 50 µg of the internal standard compounds to each vial, using an appropriate volume not to overflow the vial or dilute the extract. The internal standards recommended are *o*-terphenyl and 2,4-difluorotoluene. Other compounds may be used as internal standards, depending upon the desired target analytes and project requirements. Cap the vial and invert several times to mix the contents.

11.13 Discard the remaining contents of the VOA vials according to laboratory waste disposal guidelines. Shake off the last few drops with short, brisk wrist movements. If needed, rinse the vial with a water soluble solvent to ensure that the extraction solvent is removed. Reweigh the capped vial, and record the weight to the nearest 0.1 g. The difference between this weight, and the weight determined in Sec. 11.6 is equal to the volume of water extracted, in mL.

11.14 Extracts should be stored in the freezer or over ice until analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

There are no data analysis and calculation steps directly associated with this procedure. Follow the directions given in the determinative method.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method.

13.2 Single-laboratory precision data were obtained for MAHs and PAHs at four different spiking concentrations in reagent water. Three replicates were prepared at three levels, and six replicates at one level. Extracts were analyzed by Method 8100, set up to include the MAHs along with the PAHs. Data summary tables are included in this method. For guidance purposes, the data are reported in detail in Reference 1.

14.0 POLLUTION PREVENTION

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

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste*

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Reduction, a free publication available from the American Chemical Society (ACS), Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. "Simultaneous Analysis of Monocyclic Aromatic Hydrocarbons in Water by Microextraction." EPRI Report TR-Research Project, April 1997.

2. D. M. Munro. “Simultaneous Determination of Volatile and Semivolatile Organic Compounds in Soil.” *Remediation Journal,* 10, pp. 65-81, 2000. http://onlinelibrary.wiley.com/doi/10.1002/rem.3440100408/pdf

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method.

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TABLE 1

EXAMPLE MICROEXTRACTION GC/FID RECOVERY DATA FOR MAHs AND PAHs IN REAGENT WATER SPIKED AT 8.57 μg/L[1]

Compound	Sample A		Sample B		Sample C		Std. Dev.
	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	(Conc.[2])
Benzene	9.59	112	7.40	86	8.80	103	1.1
Toluene	9.03	105	9.44	110	9.36	109	0.22
Ethylbenzene	8.60	100	8.81	103	8.87	103	0.14
m/p-Xylene	17.0	99	18.1	106	18.1	106	0.60
Styrene	6.86	80	7.17	84	6.99	82	0.16
o-Xylene	9.94	116	10.3	120	10.5	123	0.31
1,2,4-Trimethylbenzene	7.44	87	8.53	100	8.35	97	0.59
Naphthalene	7.02	82	7.36	86	7.48	87	0.24
2-Methylnaphthalene	7.11	83	7.76	91	7.96	93	0.45
1-Methylnaphthalene	7.98	93	8.45	99	8.80	103	0.41
Acenaphthylene	8.44	98	8.45	99	8.74	102	0.17
Acenaphthene	9.23	108	9.73	114	9.61	112	0.26
Dibenzofuran	6.72	78	7.01	82	6.99	82	0.16
Fluorene	6.17	72	6.62	77	7.35	86	0.60
Phenanthrene	7.43	87	7.90	92	7.74	90	0.24
Anthracene	4.76	56	6.19	72	6.24	73	0.84
Fluoranthene	8.12	95	8.53	100	8.57	100	0.25
Pyrene	8.49	99	8.78	102	8.92	104	0.22
Benz(a)anthracene	6.59	77	6.88	80	6.46	75	0.21
Chrysene	5.78	67	5.92	69	6.14	72	0.18
Benzo(b)fluoranthene[3]	14.8	172	14.8	172	12.0	141	1.6
Benzo(k)fluoranthene[3]	16.6	194	10.4	122	8.60	100	4.2
Benzo(a)pyrene	6.60	77	6.24	73	6.94	81	0.35
Indeno(1,2,3-cd)pyrene	5.45	64	6.16	72	5.37	63	0.43
Dibenz(a,h)anthracene	5.62	66	5.90	69	5.10	60	0.40
Benzo(g,h,i)perylene	6.15	72	6.41	75	6.50	76	0.18

[1] Three reagent water samples were spiked at 8.57μg/L with all target compounds (in acetone), and then extracted and analyzed by the method. A small spiking volume at high analyte concentration was used to maximize the analyte/water interaction prior to the extraction step.

[2] Concentration units are μg/L.

[3] High compound recovery was caused by an unidentified interference, possibly a silicone compound.

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TABLE 2

EXAMPLE MICROEXTRACTION GC/FID RECOVERY DATA FOR MAHs AND PAHs IN REAGENT WATER SPIKED AT 42.9 µg/L[1]

Compound	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F		Std. Dev.
	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	(Conc.[2])
Benzene	45.0	105	41.3	96	29.5	69	44.9	105	43.0	100	43.6	102	5.9
Toluene	44.5	104	45.8	107	43.9	102	45.4	106	46.0	107	46.3	108	0.93
Ethylbenzene	45.9	107	47.1	110	45.7	107	47.5	111	47.7	111	48.4	113	1.1
m/p-Xylene	92.3	108	95.1	111	91.9	107	95.3	111	95.7	112	98.0	114	2.3
Styrene	40.7	95	41.9	98	40.9	95	42.1	98	42.4	99	43.4	101	1.0
o-Xylene	49.6	116	51.2	120	49.3	115	50.8	119	51.3	120	52.4	122	1.2
1,2,4-Trimethylbenzene	44.0	103	45.9	107	44.8	104	45.6	106	45.4	106	46.5	109	0.88
Naphthalene	42.8	100	44.0	103	43.2	101	43.8	102	43.7	102	43.8	102	0.45
2-Methylnaphthalene	43.3	101	44.8	105	43.6	102	43.9	103	44.3	103	43.7	102	0.54
1-Methylnaphthalene	45.4	106	46.9	109	46.0	107	46.0	107	45.7	107	45.2	106	0.58
Acenaphthylene	46.0	107	47.4	111	46.6	109	46.3	108	46.5	109	46.0	107	0.51
Acenaphthene	47.7	111	48.8	114	48.2	112	47.7	111	48.2	112	46.9	109	0.67
Dibenzofuran	43.6	102	45.0	105	44.3	103	44.0	103	44.0	103	43.3	101	0.60
Fluorene	44.8	104	45.6	106	45.4	106	44.5	104	44.7	104	44.3	103	0.53
Phenanthrene	46.1	108	47.5	111	47.3	110	46.4	108	46.9	109	46.1	107	0.61
Anthracene	37.4	87	38.6	90	38.4	90	38.0	89	37.7	88	37.4	87	0.51
Fluoranthene	46.3	108	48.0	112	48.0	112	46.9	109	47.0	110	45.9	107	0.84
Pyrene	46.5	109	48.6	113	47.7	111	47.2	110	47.4	111	47.4	111	0.68
Benz(a)anthracene	41.9	98	44.8	105	44.3	103	42.8	100	43.0	100	42.6	99	1.1
Chrysene	42.8	100	44.8	105	44.1	103	43.5	102	43.9	102	43.1	101	0.72
Benzo(b)fluoranthene	47.2	110	50.6	118	49.8	116	46.9	109	48.4	113	50.8	119	1.7
Benzo(k)fluoranthene	52.2	122	50.5	118	46.8	109	46.9	109	47.5	111	46.7	109	2.3
Benzo(a)pyrene	39.2	91	41.3	96	40.7	95	40.0	93	40.0	93	40.5	95	0.73
Indeno(1,2,3-cd)pyrene	49.8	116	45.7	107	38.4	89	42.7	100	42.5	99	41.3	96	3.9
Dibenz(a,h)anthracene	35.4	83	36.7	86	35.3	82	35.5	83	35.4	83	35.7	83	0.52
Benzo(g,h,i)perylene	38.3	89	39.7	93	39.2	91	38.7	90	38.5	90	38.9	91	0.50

[1] Six reagent water samples were spiked at 42.9 µg/L with all target compounds (in acetone), and then extracted and analyzed by the method. A small spiking volume at high analyte concentration was used to maximize the analyte/water interaction prior to the extraction step.

[2] Concentration units are µg/L.

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TABLE 3

EXAMPLE MICROEXTRACTION GC/FID RECOVERY DATA FOR MAHs AND PAHs IN REAGENT WATER SPIKED AT 857 µg/L[1]

Compound	Sample A		Sample B		Sample C		Std. Dev.
	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	(Conc.[2])
Benzene	819	96	866	101	797	93	35
Toluene	905	106	931	109	937	109	17
Ethylbenzene	922	108	933	109	978	114	30
m/p-Xylene	1840	107	1860	109	1950	114	59
Styrene	878	102	878	102	935	109	33
o-Xylene	946	110	951	111	998	116	28
1,2,4-Trimethylbenzene	933	109	928	108	991	116	35
Naphthalene	908	106	951	111	939	110	22
2-Methylnaphthalene	909	106	955	111	940	110	23
1-Methylnaphthalene	909	106	955	111	940	110	23
Acenaphthylene	905	106	962	112	937	109	29
Acenaphthene	913	107	958	112	945	110	23
Dibenzofuran	906	106	949	111	938	109	22
Fluorene	910	106	956	112	941	110	24
Phenanthrene	909	106	954	111	943	110	23
Anthracene	887	103	930	108	922	108	23
Fluoranthene	909	106	954	111	946	110	24
Pyrene	902	105	943	110	948	111	26
Benz(a)anthracene	907	106	947	110	942	110	22
Chrysene	937	109	992	116	968	113	28
Benzo(b)fluoranthene	894	104	875	102	930	108	28
Benzo(k)fluoranthene	924	108	1120	131	961	112	104
Benzo(a)pyrene	898	105	967	113	937	109	34
Indeno(1,2,3-cd)pyrene	895	104	942	110	933	109	25
Dibenz(a,h)anthracene	900	105	1010	118	941	110	56
Benzo(g,h,i)perylene	908	106	974	114	949	111	33

[1] Three reagent water samples were spiked at 857 µg/L with all target compounds (in acetone), and then extracted and analyzed by the method. A small spiking volume at high analyte concentration was used to maximize the analyte/water interaction prior to the extraction step.

[2] Concentration units are µg/L.

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TABLE 4

EXAMPLE MICROEXTRACTION GC/FID RECOVERY DATA FOR MAHs AND PAHs IN REAGENT WATER SPIKED AT 8570 μg/L[1]

Compound	Sample A		Sample B		Sample C		Std. Dev.
	Conc.[2]	% Rec.	Conc.[2]	% Rec.	Conc.[2]	% Rec.	(Conc.[2])
Benzene	9470	111	8770	102	9070	106	351
Toluene	8440	98	8710	102	8510	99	140
Ethylbenzene	8750	102	8860	103	8860	103	64
m/p-Xylene	17500	102	17700	103	17700	103	115
Styrene	NA	NC	NA	NC	NA	NC	NC
o-Xylene	8810	103	8910	104	8910	104	58
1,2,4-Trimethylbenzene	8840	103	8940	104	8950	104	61
Naphthalene	8520	99	8800	103	8480	99	174
2-Methylnaphthalene	8400	98	8880	104	8540	100	247
1-Methylnaphthalene	8420	98	8890	104	8540	100	244
Acenaphthylene	8490	99	8750	102	8440	98	167
Acenaphthene	8520	99	8790	103	8470	99	172
Dibenzofuran	8450	99	8930	104	8600	100	246
Fluorene	8550	100	8820	103	8510	99	169
Phenanthrene	8680	101	8940	104	8640	101	163
Anthracene	8230	96	8480	99	8150	95	172
Fluoranthene	8560	100	8820	103	8520	99	163
Pyrene	8630	101	8910	104	8600	100	171
Benz(a)anthracene	8630	101	8890	104	8610	100	156
Chrysene	8450	99	8750	102	8400	98	189
Benzo(b)fluoranthene	8850	103	9110	106	8810	103	163
Benzo(k)fluoranthene	8420	98	8660	101	8380	98	151
Benzo(a)pyrene	8700	102	8960	105	8660	101	163
Indeno(1,2,3-cd)pyrene	9480	111	9670	113	9480	111	110
Dibenz(a,h)anthracene	8550	100	8970	105	8540	100	245
Benzo(g,h,i)perylene	8990	105	9310	109	9030	105	174

NA = Not available
NC = Not calculated

[1] Three reagent water samples were spiked at 8570 μg/L with all target compounds (in acetone), and then extracted and analyzed by the method. A small spiking volume at high analyte concentration was used to maximize the analyte/water interaction prior to the extraction step.

[2] Concentration units are μg/L.

Appendix A:

Summary of Revisions to Method 3511 (as compared to previous Revision 0, November 2002)

1. Improved overall method formatting for consistency with new SW-846 methods style guidance. The format was updated to Microsoft Word.docx.
2. Minor editorial and technical revisions were made throughout to improve method clarity.
3. The revision number was changed to1 and the date published was changed to July 2014.
4. This appendix was added showing changes from the previous revision.
5. Added Updated IDP language and LLOQ verification standard language to Secs. 9.2 and 9.3.
6. Added a reference in Sec. 16.

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