



Empore™ Extraction Disks

Method Summary

EPA Method 1613 Revision B Solid Phase Disk Extraction

Tetra- Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS

Method 1613 contains extraction directions for several matrices including aqueous, solid, and tissue samples. The 3M Empore™ disk extraction procedure is applicable only to aqueous matrices. The method is for use in EPA's data gathering and monitoring programs associated with the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation and Liability Act, and the Safe Drinking Water Act.

Issued October 1994 by US EPA Office of Water as document number EPA 821-B-94-005, copies of the publication can be obtained from: Water Resource Center; Mail Code RC-4100; 401 M Street SW; Washington, D.C. 20460. Telephone: (202) 260-7786 or -2814.

Summary of Method

One liter aqueous samples are spiked with isotopically labeled analogs of the Chlorinated DibenzoDioxins/Chlorinated DibenzoFurans (CDDs/CDFs). Those samples containing sediment can either be filtered off-line or in-situ (a prefilter placed above the Empore disk in the extraction glassware) with the extraction disk. Samples (pH 2, 5 mL methanol) are extracted with a C18 disk. Both the prefilter and the C18 disk are placed in a Soxhlet thimble and extracted 16-24 hours with toluene. The extract is subjected to the standard clean-up and concentration techniques as in the previous version of Method 1613.

Performance Data

Validation studies for this method were performed on four types of wastewater provided by EPA. The effluents included POTW final effluent, petroleum refinery API separator and secondary treatment effluent, and a river water. Validation data on these samples were generated in a single laboratory with additional recovery data provided by pulp and paper mills on various matrices from their respective plants. The study was performed on triplicate samples of each matrix. One liter of each wastewater type was also analyzed for background levels. In addition, the post-extraction filtrate from the triplicate spikes was combined and liquid/liquid extracted to document the efficiency of extraction. In all cases, the combined filtrates were "not detected" for all analytes. Using the in-situ prefilter, settle/decant method described herein, all sample extractions were completed in under 20 minutes.

Validation Data

Refinery Effluent

Analyte	Spike Level pg/L	POTW Effluent %R (RSD)	API %R (RSD)	Final %R (RSD)	River Water %R (RSD)
Native					
2378-TCDD	200	101 (5.7)	109 (11)	110 (5.6)	112 (2.6)
12378-PeCDD	1000	88 (1.0)	110 (9.1)	110 (0.6)	115 (1.5)
123478-HxCDD	1000	99 (9.0)	114 (4.9)	108 (2.5)	116 (1.5)
123678-HxCDD	1000	99 (9.0)	105 (3.0)	106 (1.5)	123 (2.6)
123789-HxCDD	1000	99 (5.6)	118 (15)	109 (1.5)	115 (6.1)
1234678-HpCDD	1000	102 (4.9)	112 (4.2)	108 (0.6)	110 (2.1)
OCDD	2000	111 (3.6)	B 119 (3.5)	B 119 (2.6)	B 116 (1.0)
12378-PeCDF	1000	90 (1.7)	115 (6.8)	111 (1.5)	116 (0.6)
23478-PeCDF	1000	98 (4.0)	113 (6.6)	111 (2.7)	114 (1.0)
123478-HxCDF	1000	100 (3.6)	110 (6.0)	108 (6.0)	118 (0.6)
123678-HxCDF	1000	100 (5.0)	110 (2.6)	105 (2.1)	116 (1.0)
234678-HxCDF	1000	94 (1.7)	108 (2.5)	101 (1.5)	114 (1.0)
123789-HxCDF	1000	95 (4.2)	107 (5.0)	104 (2.1)	118 (2.6)
1234678-HpCDF	1000	B 110 (2.1)	109 (8.6)	100 (3.0)	119 (2.0)
1234789-HpCDF	1000	103 (4.0)	106 (10)	100 (1.5)	119 (2.0)
OCDF	2000	105 (9.8)	119 (13)	115 (3.5)	119 (7.2)
Other Standards					
37C1 -TCDD		62 (6.2)	64 (1.8)	71 (9.1)	63 (1.5)
Internal Standards					
13C12-2378-TCDD		56 (6.7)	64 (2.8)	71 (8.5)	59 (1.9)
13C12-PeCDD 123		82 (12)	61 (12)	72 (15)	83 (5.0)
13C12-HxCDD 478		76 (2.9)	87 (18)	99 (5.2)	80 (4.2)
13C12-HxCDD 678		72 (2.3)	70 (12)	76 (5.0)	76 (2.5)
13C12-HpCDD 678		71 (4.2)	87 (6.7)	83 (20)	83 (2.6)
13C12-OCDD		69 (6.0)	80 (14)	86 (4.9)	79 (8.4)
13C12-PeCDF 123		67 (7.2)	61 (9.3)	77 (13)	74 (3.4)
13C12-PeCDF 234		61 (5.3)	63 (9.3)	79 (14)	80 (4.7)
13C12-HxCDF 478		82 (7.2)	81 (14)	96 (1.8)	71 (6.8)
13C12-HxCDF 678		69 (4.0)	72 (6.4)	79 (1.4)	69 (6.3)
13C12-HxCDF 234		80 (2.4)	76 (7.3)	85 (1.4)	73 (0.9)
13C12-HxCDF 789		73 (1.6)	78 (7.7)	85 (5.7)	75 (1.4)
13C12-HpCDF 678		63 (2.5)	78 (9.4)	77 (14)	69 (11)
13C12-HpCDF789		68 (1.1)	85 (5.7)	80 (20)	81 (2.4)

B = Present in Blank Sample

n = 3

2,3,7,8-TCDF Not Determined

Average Recovery Native Compounds = 109%

Method

1. This extraction option is applicable only to aqueous samples. Measure sample volume. Add isotopically labeled analogs, 5 mL methanol, and pH to 2. Allow the sample to sit and the sediment settle as much as is practical.
2. Assemble an all glass filtration assembly using a 90 mm 3M Empore™ extraction disk containing C18. For samples with no particulates, a 47mm disk can be used. Use of a manifold for multiple extractions is acceptable.

If samples contain significant quantities of particulates, the use of an in-situ glass micro-fiber prefilter (Whatman GMF 150, 1 micron pore size or equivalent) is advisable. The glass fiber prefilter is placed on top of the Empore disk prior to placement of the glass reservoir and clamp.

3. **Prewash.** Wash the extraction apparatus and disk by adding 15 mL of toluene to the reservoir washing down the sides of the glass reservoir in the process. Pull a small amount through the disk with a vacuum; turn off the vacuum and allow the disk to soak for about one minute. Pull the remaining solvent through the disk and allow the disk to dry. Repeat the wash step using 15 mL of acetone.
4. **Condition.** Pre-wet the disk by adding 15 mL methanol (MeOH) to the reservoir, pulling a small amount through the disk then letting it soak for about one minute. Pull most of the remaining MeOH through the disk, leaving 3-5 mm on the surface of the disk, which should not be allowed to go dry from this point until the sample extraction has been completed. **THIS IS A CRITICAL STEP FOR A UNIFORM FLOW AND GOOD RECOVERY.** The disk is composed of hydrophobic materials. To make them amenable to a water solution, they must be pre-wetted with a water miscible solvent (MeOH) or they will not allow water to pass through the materials. Should the material accidentally dry before the sample is added, simply repeat the pre-wetting step.
5. Rinse the disk by adding 50 mL of reagent water to the disk and drawing most through, again leaving 3-5 mm of water on the surface of the disk. Repeat using a second 50 mL aliquot of water.
6. **Extraction.** Add the water sample to the reservoir and, under full vacuum, filter as quickly as the vacuum will allow. Drain as much water from sample bottle as possible. Particulate-free water may pass through the disk in as little as 10 minutes without reducing analyte recoveries. Allow the entire sample to pass through the disk then dry the disk by maintaining vacuum for about 3 minutes.

With heavily particle-laden samples, allow the sediment to settle, decant as much liquid as is practical into the reservoir. Allow most of the liquid to filter then swirl the sediment portion and add it to the reservoir. Before the entire sample has filtered, rinse the sample bottle with reagent water and add to the reservoir to transfer any particulates remaining in the bottle to the extraction. Drain as much water as possible from the sample bottle.

7. Disassemble the extraction glassware and carefully transfer both the Empore disk and the particle-laden GMF filter to the thimble of a Soxhlet Dean-Stark apparatus. Place a glass wool plug over the filters. Add about 50 mL toluene to the sample bottle, replace the cap and agitate well to rinse all remaining residues into the toluene. Transfer the toluene to the Soxhlet apparatus. Repeat the bottle rinse three times. Using 10 mL toluene, rinse the extraction glassware reservoir and transfer to the Soxhlet.
8. Soxhlet extract for 16 - 24 hours. The data for this validation was generated using a 16 hour Soxhlet extraction.
9. **Concentrate sample and proceed to clean-up and analysis as per the Method 1613 directions.**

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