



Empore™ Extraction Disks

Method Summary

EPA Method 515.2

Determination of Chlorinated Acids in Water Using Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector

Issued August, 1992 as a part of Methods for the Determination of Organic Compounds in Drinking Water, Supplement II; Revision 1.0; Author: J.W. Hodgeson, Environmental Monitoring Systems Laboratory, Office of Research and Development, US Environmental Protection Agency, Cincinnati, Ohio 45268.

The complete method is available as a part of Supplement II from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703, (800) 553-6847.

Summary

This method determines chlorinated acids in ground water and finished drinking water. Surface water is also included in recovery and precision data. Salts and esters are hydrolyzed to the corresponding acid before the analysis. The sample prep uses 47 mm SDB solid phase extraction disks to extract the analytes, diazomethane to form the methyl esters, and GC-ECD for final analysis.

Analytes

Acifluorfen	Dichlorprop
Bentazon	Dionseb
2,4-D	5-Hydroxydicamba
2,4-DB	Pentachlorophenol (PCP)
Dacthal	Picloram
Dicamba	2,4,5-T
3,5-Dichlorobenzoic Acid	2,4,5-TP (Silvex)

MDLs in reagent water range from 0.06 to 1.23 µg/L with an average of 0.35 µg/L.

Method Detection Limits

Method

1. Mix 250 ml of sample with 50 g sodium sulfate and 4 ml of 6N NaOH (to pH 12) in a separatory funnel. Shake periodically for 1 hour.
2. Extract organic interferences from the aqueous layer using 3 x 15 ml methylene chloride. Discard the methylene chloride. Drain the aqueous layer into a 500 ml beaker and adjust the pH to 1 using concentrated sulfuric acid.
3. Assemble Empore™ 47 mm poly(styrene-divinylbenzene) extraction disks in an all-glass filtration apparatus (manifolds are appropriate for multiple samples).
4. Wash the disks with 20 ml of 10% methanol in methyl-t-butyl ether by allowing the solvent to soak into the disk for two minutes. Use vacuum to pull air through the disk for five minutes.

5. Using a vacuum of about 5 inches of Hg, add the following series to the disk: a. 20 ml methanol; b. 20 ml reagent water; c. sample. Don't allow the disk to dry between the steps. (For samples with suspended solids, higher vacuum is appropriate once the flow slows down.)
6. Once the sample has passed through the disk, apply maximum vacuum for 20 minutes to dry the disk.
7. Place an appropriate elution tube in the vacuum apparatus to collect the eluant. Use 2 x 2 ml of 10% methanol in methyl-t-butyl ether to elute the disk. Allow each aliquot to soak into the disk for one minute before applying vacuum.
8. Rinse the 500 ml beaker with 4 ml of pure methyl-t-butyl ether and elute the disk with this, as in step 7.
9. Dry the extract with sodium sulfate. If a water layer is in the elution tube, leave it behind, rinsing the elution tube with 2 x 1 ml methyl-t-butyl ether and adding these rinsings to the drying tube. Finally, rinse the drying tube with 1 ml of methyl-t-butyl ether. The final volume should be 6-9 ml.
10. Derivatize the acids in a diazomethane generator and analyze by GC-ECD.

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