Application Note

EXTRACTION OF CHLORINATED ACIDS FROM WATER (EPA METHOD 515.2)

This is a summary of Section 11 of EPA Method 515.2 entitled "Determination of Chlorinated Acids in Water Using Liquid-Solid Extraction And Gas Chromatography With An Electron Capture Detector."

Field	Environmental
Sample	Chlorinated Acids
Matrix	Drinking Water
Extraction Disk	BAKERBOND Speedisk® with divinylbenzene polymer (DVB), 50 mm, Part Number 8059-06.
Safety and Protective Equipment	Goggles and face shield, lab coat and apron, gloves, vent hood, Type B fire extinguisher
Sample Preparation	Mix 250 mL sample, an appropriate amount of surrogate standards, 4 mL of 6N NaOH and 50 g hydrated sodium sulfate in a separatory funnel. Check the pH of the sample. If the pH is lower than 12, adjust the pH by adding more 6N NaOH. Let the sample stand for 1 hour at room temperature. Shake periodically. Extract the sample with 15 mL methylene chloride. Discard the methylene chloride phase. Repeat the extraction procedure with 2 × 15 mL methylene chloride. Drain the contents of the separatory funnel into a 500 mL beaker and adjust the pH to 1.0 ± 0.1 by the dropwise addition of concentrated sulfuric acid.
Extraction Disk Conditioning	Assemble a Speedisk® extraction station. (Other types of vacuum stations can also be used.) Mount disk on vacuum port. Wash the disk with 20 mL of 10% by volume of methanol in methyl-t-butyl ether (MTBE). Soak the disk for 2 minutes. Pull the solvent through the disk. Dry the disk for 5 minutes using full vacuum. Adjust the vacuum to about 5" Hg and add 20 mL of methanol. Without turning the vacuum off , immediately add 20 mL of water. From this point until the end of the sample addition, keep the disk wet.
Sample Addition	Add the sample at 5" Hg. A higher vacuum pressure may be used to maintain the desired flow rate. After extraction of the entire sample, apply maximum vacuum and dry the disk for 20 minutes.
Sample Elution	Remove the BAKERBOND Speedisk® from the extraction station and insert a collection tube. Apply 2 mL of 10% by volume methanol in MTBE without vacuum, Let soak 1 minute. Apply vacuum to elute. Rinse the 500 mL sample beaker with 4 mL of pure MTBE. With vacuum off, transfer the solvent to the disk. Soak the disk for 1 minute before pulling through the MTBE. Transfer the organic layer from the collection tube to a drying tube containing 3 g anhydrous sodium sulfate. Keep the drying tube wet between this and subsequent steps. Rinse the aqueous layer in the collection tube with 2 × 1 mL MTBE and repeat the drying step. Finally, rinse the drying tube with 1 mL MTBE. The final volume should be 6-9 mL. Derivatize the acids in a diazomethane generator.
Final Analytical Method	GC-ECD (Refer to EPA Method 515.2.)
Analytes	1. Dalapon, 2. 3, 5-Dichlorobenzoic Acid, 3. 4-Nitrophenol, 4. DCAA, 5. Dicamba, 6. Dichlorprop, 7. 2,4-D, 8. DBOB (internal gtandard), 9. Pentachlorophenol, 10. 2,4,5-TP, 11. 2,4,5- T, 12. 2,4-DB, 13. Dinoseb, 14. Bentazon, 15. Pichloram, 16. DCPA, 17. Acifluorfen

Product List

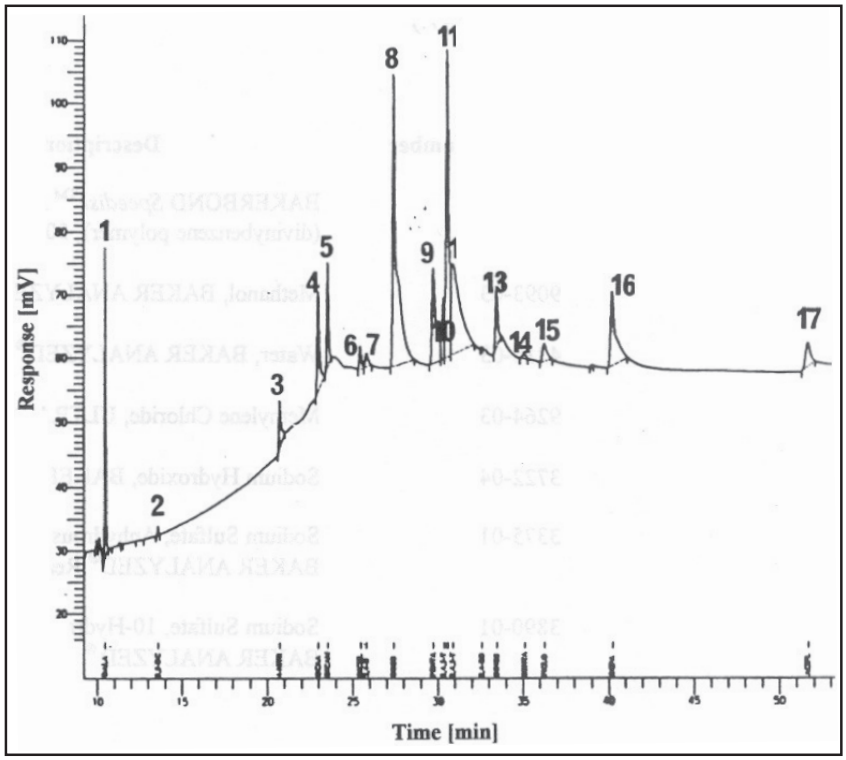
Product Number	Description
8068-06	BAKERBOND Speedisk® DVB (divinybenzene polymer), 50 mm
8402	Methanol, BAKER ANALYZED® HPLC
4218	Water, BAKER ANALYZED® HPLC
9264	Methylene Chloride, ULTRA RESI-ANALYZED®
9043	Methyl tert-Butyl Ether (MTBE) ULTRA RESI-ANALYZED®
5672	Sodium Hydroxide, 6N Volumetric Solution
3375	Sodium Sulfate, Anhydrous, Granular, BAKER ANALYZED® Reagent
3890	Sodium Sulfate, 10-Hydrate, Crystal, BAKER ANALYZED® ACS
9681	Sulfuric Acid BAKER ANALYZED® Reagent

Chemicals used in Downstream Process (2)

Column: DB-1, 60 m, 0.32 mm ID, 5.0 µm film

Oven Temp: 80°C for 1 minute,
to 200°C at 20°C minute,
to 280°C at 5°C/minute,
hold for 44 minutes.

Injector Temp: 250°C
Detector Temp: 290°C
Detector. ECD



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