



US EPA Method 525.2 — Determination of Semi-Volatile Organics in Water by Solid Phase Extraction and GC/MS Detection

Introduction

Method 525.2 is a drinking water method for the determination of a wide variety of semi-volatile organic compounds including PAHs, pesticides and PCBs. It uses solid phase extraction (SPE) for isolation and concentration and Gas Chromatography/Mass Spectroscopy (GC/MS) for determination.

Method 525.2 is performed by loading a one liter sample of water onto a conditioned C-18 SPE cartridge, the cartridge is dried and eluted with a combination of ethyl acetate (EtOAc) and methylene chloride (DCM). The extract is dried by passing through 5-7 g of fired sodium sulfate and concentrated under nitrogen to 1 mL. For this work, we used the optional cartridge SPE (6 mL syringe body, 1000 mg of C-18 packing) and a Varian Saturn Ion Trap GC/MS.

AutoTrace Extraction Procedures

Loading Procedure:

- Step 1:** Process 6 samples using the following procedure
- Step 2:** Wash syringe with 2 mL of MeOH
- Step 3:** Rinse column with 5 mL of EtOAc into SOLVENT WASTE
- Step 4:** Rinse column with 5 mL of DCM into SOLVENT WASTE
- Step 5:** Condition column with 10 mL of MeOH into SOLVENT WASTE
- Step 6:** Condition column with 10 mL of Water into AQUEOUS WASTE
- Step 7:** Load 1000 mL of sample onto column
- Step 8:** Dry column with gas for 10 minutes
- Step 9:** END

Flow Rate:

- Cond Flow:** 40 mL/min
- Load Flow:** 20 mL/min
- Rinse Flow:** 40 mL/min
- Elute Flow:** 5 mL/min
- Cond air push:** 15 mL/min
- Rinse air push:** 20 mL/min
- Elute air push:** 5 mL/min

Eluting Procedure:

- Step 1:** Process 6 samples using the following procedure
- Step 2:** Manually rinse sample container with 7 mL to collect
- Step 3:** Manually rinse sample container with 10 mL to collect
- Step 4:** Soak and collect 3 mL fraction using EtOAc
- Step 5:** Collect 2 mL fraction into sample tube using EtOAc
- Step 6:** Soak and collect 3 mL fraction using DCM
- Step 7:** Collect 2 mL fraction into sample tube using DCM
- Step 8:** END

In Step 2, add 5 mL of EtOAc to sample container, rinsing the walls and repositioning tube and pressing COND. Again in Step 3, add 5 mL of DCM to sample container, rinsing the walls and repositioning tube and pressing COND. The extra volume pulled allows air to follow each rinsing. Note that the "Load Flow" parameter is different for the two procedures.

Flow Rate:

- Cond Flow:** 40 mL/min
- Load Flow:** 5 mL/min
- Rinse Flow:** 40 mL/min
- Elute Flow:** 5 mL/min
- Cond air push:** 15 mL/min
- Rinse air push:** 20 mL/min
- Elute air push:** 5 mL/min

Varian Saturn Ion Trap Conditions

COLUMN: Restek Rtx-5MS 0.25 mm ID by 30 m with 0.25 μ m df

INJECTION TEMPERATURE: 245°, splitless for 1 min.

TEMPERATURE PROGRAM: 70° (1 min), 50°/min, 130°(0.8 min), 12°/min, 180°, 7°/min, 240°, 12°/min, 320° (1.61 min) for total of 24 min.

PRESSURE PROGRAM: 20 psig(1 min), -3.6 psig/min, 13.1 psig, 0.6 psig/min, 15.7 psig, 0.4 psig/min, 18.7 psig, 0.5 psig/min, 22.7 psig (1.2 min) for total of 24 min.

Analytical Results

COMPOUND	%REC.	%RSD	COMPOUND	%REC.	%RSD
Chlordane	110%	7.8%	Methoxychlor	111%	4.5%
Toxaphene	92%	7.4%	Heptachlor	109%	3.4%
Aroclor 1248	104%	3.6%	Heptachlor epoxide	106%	2.8%
Atrazine	119%	12.8%	Hexachlorobenzene	120%	5.2%
Alachlor	109%	4.8%	Hexachlorocyclopentadiene	85%	7.6%
Simazine	142%	5.8%	Aldrin	104%	3.1%
Metolachlor	128%	2.8%	Dieldrin	106%	4.0%
Propachlor	147%	5.4%	DDT	91%	4.6%
Butachlor	102%	14.3%	Benzo(a)pyrene	71%	3.3%
Metribuzin	126%	7.2%	bis(2-ethylhexyl)adipate	120%	3.4%
Lindane	119%	4.1%	bis(2-ethylhexyl)phthalate	79%	2.7%
Endrin	111%	4.3%			

Summary

Using Caliper Life Sciences' AutoTrace system effectively automates sample extraction using a cartridge. Cartridges provide a greater variety of chemistries than are available for disks. The positive flow features mean that each extraction is uniform and well controlled. Compared with manual SPE procedures, the AutoTrace increases production and minimizes error by relieving the operator of tedium.

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