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

## *INTRODUCTION*

Method 525.2, determination of organic compounds in drinking water by SPE and capillary column gas chromatography/mass Spectrometry, is a drinking water method for the determination of a wide variety of semi-volatile organic compounds including PAHs, pesticides, and PCBs.

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 C18 packing), and a Varian Saturn Ion Trap GC/MS.

## *AUTOTRACE EXTRACTION PROCEDURES*

**Loading Procedure (Method One):**

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|---|---|
| Step 1: | Process 6 samples using the following procedure |
| Step 2: | Wash syringe with 2 mL of $CH_3OH$ |
| 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 $CH_3OH$ 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 |

**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 (Method Two):**

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|---|---|
| Step 1: | Process 6 samples using the following procedure |
| Step 2: | Manually rinse the sample container with 7 mL to collect. Use Ethyl acetate to rinse the sample container. See Elution details section. |
| Step 3: | Manually rinse sample container with 10 mL to collect. Use Dichloromethane to rinse the sample container. See Elution details section. |
| Step 4: | Elute to soak 3 mL fraction using EtOAc |
| Step 5: | Elute to collect 2 mL fraction into sample tube using EtOAc |
| Step 6: | Elute to soak 3 mL fraction using DCM |
| Step 7: | Elute to collect 2 mL fraction into sample tube using DCM |

**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 |

**Manual Rinse Details:**

In Step 2, add 5 mL of EtOAc to sample container, rinsing the walls, and reposition tube, and press COND. Again in Step 3, add 5 mL of DCM to sample container, rinsing the walls, and reposition tube, and press COND. The extra volume pulled allows air to follow each rinsing. Note that the "Load Flow" parameter is different for the the eluting procedure (Method Two) and should be set at 5mL/min.

### *VARIAN SATURN ION TRAP CONDITIONS*

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|---|---|
| Column: | Restek Rtx®-5MS 0.25 mm ID by 30 m with 0.25 µm df. |
| Injection Temp.: | 245 °C, splitless for 1 min. |
| Temp. Prog.: | 70 °C (1 min), 50 °C/min, 130 °C (0.8 min), 12 °C/min, 180°, 7 °C/min, 240 °C, 12 °C/min, 320 C° (1.61 min) for total of 24 min. |
| Pressure Temp: | 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. |

| Table 1. Analytical Results of Spiked Samples | | | | | |
|---|---|---|---|---|---|
| **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 AutoTrace® system effectively automates sample extraction using a cartridge.

Solid Phase Extraction provides reduced solvent consumption and sample handling when compared to traditional liquid-liquid extraction techniques. The AutoTrace instrument automates the conditioning loading rinse and eluting steps of Manual SPE that normally require extensive involvement by an operator. AutoTrace provides both Automation of the SPE steps and positive pressure flow of sample and solvent through the cartridges. Positive pressure flow of the sample and solvent ensure an uniform extraction and elution. These features contribute greatly to increased laboratory productivity and reliable results for determination of organic pollutants in drinking water.



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