



SIMULTANEOUS EXTRACTION OF PESTICIDES, SEMI-VOLATILES AND PCB CONGENERS USING ONE UNIVERSAL SPE CARTRIDGE

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INTRODUCTION

Pesticides, Semi-volatiles and PCB congeners belong to different groups of contaminants in drinking water. It is difficult to extract them all with one single solid phase extraction as they have a variety of polarities. UCT has successfully developed the universal octadecyl (C₁₈) bonded silica-based sorbent that can simultaneously extract all these compounds in a single extraction, which can make it faster and more cost-effective for environmental laboratories to detect and quantify them.

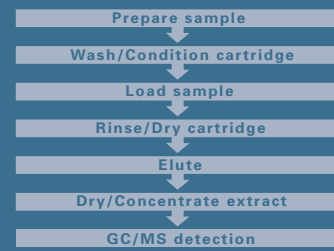
The EPA is proposing modifications to Method 525.2 including additional analytes of varying polarities, organic acids as preservatives and modified surrogates and internal standards. The UCT universal cartridge (ECUNI525) has demonstrated excellent performance for the proposed method. Extraction of 127 target analytes from reagent water, surface water with high total organic carbon, and groundwater with significant hardness, yielded recoveries between 80-120% and RSDs <10%.

PROCEDURES

SPE Procedure:

1. Weigh 0.1 g L-ascorbic acid, 0.35 g ethyl enediaminetetra acetic acid trisodium salt, and 9.4 g potassium monobasic citrate into a 1 L amber bottle. Fill the bottle with 1 L sample.
2. Assemble the extraction system by attaching cartridge adaptors, cartridges and bottle holders to the manifold.
3. Wash with 5 mL 1:1 EtOAc:MeCl₂, soak for 1 min, draw the solvent through, keep the vacuum on for 2 mins.
4. Condition the cartridge with 10 mL MeOH, soak for 1 min, draw most of the MeOH through, leave a thin layer of MeOH on the surface of the cartridge. Condition with 10 mL reagent water, draw most through, leave a thin layer on the surface of the cartridge.
5. Apply sample at 10 mL/min. After passing the entire sample through, add 10 mL reagent water into the sample bottle, rinse and pass the water through the cartridge. Dry the cartridge under full vacuum for 10 mins.
6. Insert a 40 mL vial into the manifold. Elute the dried cartridge with 5 mL EtOAc. Repeat with 5 mL MeCl₂. Add 10 g muffled sodium sulfate into the cartridge. Rinse the sample bottle thoroughly with 5 mL EtOAc then pour the rinsed solvent into the cartridge. Repeat with 5 mL MeCl₂.
7. Dry the extract by passing it through fritted reservoirs holding 10 g muffled sodium sulfate. Rinse the vial with EtOAc and pass the rinse through the sodium sulfate. Repeat with MeCl₂.
8. Concentrate the extract to 1 mL under a gentle N₂ stream at 40 °C using TurboVap evaporator. Add internal standard and load to GC/MS for analysis.

FLOW CHART:



Universal cartridge with C₁₈ sorbent



6 station manifold extraction system (amber bottle should be used)

MATERIALS

SPE cartridge:
83 mL UCT Universal cartridge with 1500 mg C18 (ECUNI525)
Vacuum pump: (ECROCKER400)
6-station manifold: (ECUCTVAC6)
Cartridge adaptor: (ECUCTADP)
Bottle holder: (ECUNIBHD)
20L waste trap: (ECUCTTRAP20)
Fritted reservoirs: 50 µm Teflon frit (ERTFT1FUNIP)
Sodium sulfate: anhydrous, ACS, Granular 60 Mesh (ECSS05K)

INSTRUMENTAL

GC/MS: Agilent 6890N GC coupled with 5975C MSD, equipped with 7683 auto sampler. GC capillary column: Restek Rxi-5sil MS 30m*0.25mm*0.25µm
Injector: 1µL splitless injection at 250 °C, with a split delay of 1 min.
Linier: 4 mm splitless gooseneck, 4mmID*6.5mmOD*78.5mm (GCLGN4MM)
Temperature program: Initial oven temperature of 55 °C, hold for 1 min, ramp at 10 °C/min to 200 °C, ramp at 7 °C/min to a final temperature of 320 °C and hold for 0.36 min.

Solvent delay: 5 min.

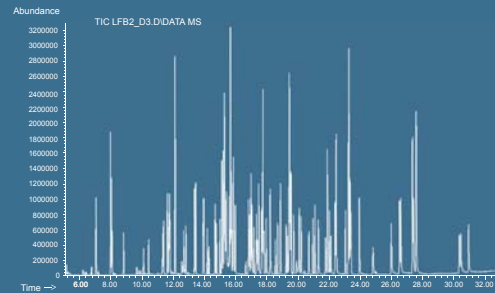
Carrier gas: Helium at a constant flow of 1.2 mL/min. MSD condition: Aux temperature: 280 °C, MS Source: 230 °C, MS Quad: 150 °C

Full scan: 45-500 amu

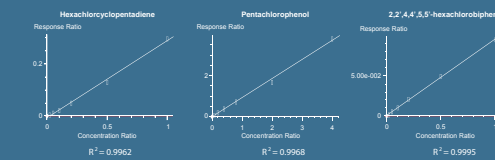
Aux temperature: 280 °C, MS Source: 230 °C, MS Quad: 150 °C

Full scan: 45-500 amu

CHROMATOGRAM: Laboratory reagent blank fortified at 2 µg/L



SELECTED CALIBRATION CURVES:



RESULTS

Table 1. Accuracy and Precision Data obtained for Pesticides: Fortified at Three Concentrations in Reagent Water and One Concentration in Ground Water and Surface Water

Analyte	Reagent Water		Reagent Water		Reagent Water		Ground Water ¹		Surface Water ¹	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
Azinphos-methyl	100	1.4	100	2.1	100	1.1	100	1.5	100	1.4
Bifenthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos	100	1.4	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Acetamiprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorantraniliprol	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
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Chlorpyrifos-methyl	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Cyfluthrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Imidacloprid	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Permethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Spinetoram	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
Triazophos	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
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Deltamethrin	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
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Thiamethoxam	100	1.2	100	1.2	100	1.2	100	1.5	100	1.2
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