EPA Method 8141B Determination of Organophosphorus Pesticides and Triazine Herbicides in Water by Solid Phase Extraction

UCT Part Numbers:
ECHLD156-P – Enviro-Clean® HL DVB 500 mg, 6 mL cartridge, PE Frit
ECSS15M6 - 5 g anhydrous sodium sulfate in 6 mL cartridge
AD0000AS - Cartridge adaptor

Summary:

This application note describes a solid phase extraction (SPE) method for the determination of EPA method 8141B analytes, including organophosphorus compounds and triazine herbicides in water. Target analytes are extracted onto a polymeric sorbent from water samples at neutral pH. Proper sample pH is critical for good analyte recoveries as organophosphorus esters hydrolyze under acidic or basic conditions. The adsorbed analytes are eluted with acetone and dichloromethane (DCM). A drying cartridge is attached downstream from the SPE cartridge in the elution step, eliminating the need for an additional extract drying step. The eluate is evaporated and exchanged to n-hexane. A gas chromatograph (GC) equipped with a 30-meter capillary column along with either a FPD, NPD or MS detector is employed for analyte separation and detection. Excellent recoveries and relative standard deviations (RSD) were obtained for 13 representative compounds (11 organophosphorus pesticides and 2 triazine herbicides).

Experimental:

SPE procedure:

1. To 1 L* portions of neutral water samples add appropriate amounts of surrogates. Surrogates and target analytes are spiked into fortified samples.
2. Attach the SPE cartridges to a glass block manifold.
3. Cartridge Conditioning

a. Wash the SPE cartridges with 5 mL DCM, pass 1/3 through to wet the sorbent, soak 1 min before draw the remaining through, turn full vacuum on for 1 min. Repeat the wash with additional 2 aliquots of 5 mL DCM.

b. Condition the cartridges with 2 aliquots of 5 mL methanol. Do not allow the sorbent go dry unless instructed so in the cartridge drying step.
c. Equilibrate the cartridges with 2 aliquots of 5 mL DI water. Leave about 4 mL of water in the cartridge, and connect sample transfer tubes to the SPE cartridges (**ECHLD156-P**).

4. Sample Loading

a. Insert the stainless steel ends of the sample transfer tubes into the sample containers and completely draw the samples through the SPE cartridges in a fast drop-wise fashion (10-15 mL/min).
b. Remove the transfer tubes from the SPE cartridges and dry the SPE cartridges under full vacuum for 10 min.

5. Eluate Drying and Elution

a. Attach the drying cartridges (**ECSS15M6**) to the bottom of the SPE cartridges with the cartridge adaptors (**AD0000AS**).
b. Insert a collection tube or vial into the manifold underneath each SPE cartridge.
c. Rinse each sample bottle with 5 mL acetone, and pull the rinsates through the SPE cartridges slowly using the transfer tubes. Turn full vacuum on for 1 min to pull all of the elution solvent into the collection container.
d. Repeat Step 5c. with 10 mL DCM.
e. Remove the transfer tubes from the SPE cartridges. Add 5 mL DCM into the SPE cartridges, pass 1/3 though, soak 1 min and draw the remaining through in a slow drop-wise fashion.

6. Eluate Concentration

a. Concentrate the eluates to about 0.5 mL under a gentle stream of nitrogen at 40 ºC.
b. Rinse the wall of the eluate containers with 3 mL of n-hexane, and continue concentrating to about 2 mL.
c. Transfer the extracts to 2-mL auto-sampler vials, and adjust the final volume to 2 mL with n-hexane.
d. Add internal standard, the samples are ready for GC analysis.

**Use of a smaller sample volume is permitted if method sensitivity is not an issue.*

GC/MS method:

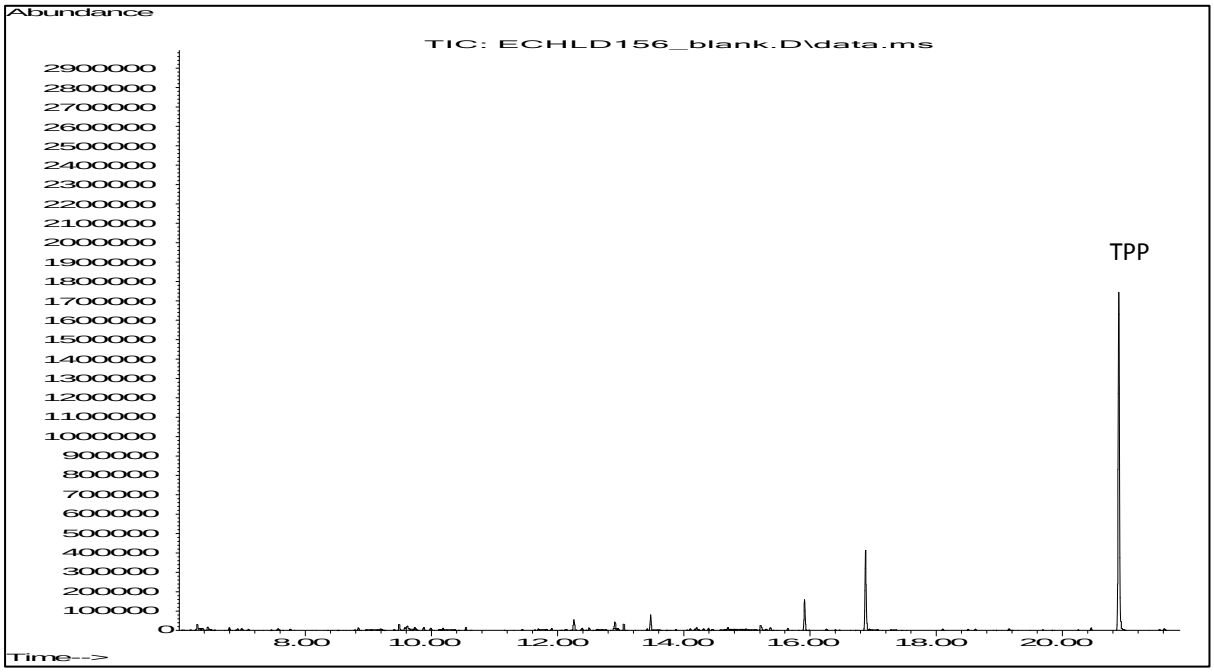
GC/MS	Agilent 6890N GC coupled with 5975C MSD
Injector Temp	250 ºC
Injection Volume	2 µL splitless
GC column	Restek Rxi®-5sil MS 30mx0.25mm, 0.25µm with 10m guard column
Carrier gas	Helium at a constant flow of 1.2 mL/min
Oven	Initial temperature at 60 ºC, hold for 1 min; ramp at 10 ºC/min to 300 ºC, hold for 2 min.
Transfer line	280 ºC
Tune	dftpp.u
Full Scan	45-450 amu

Results:

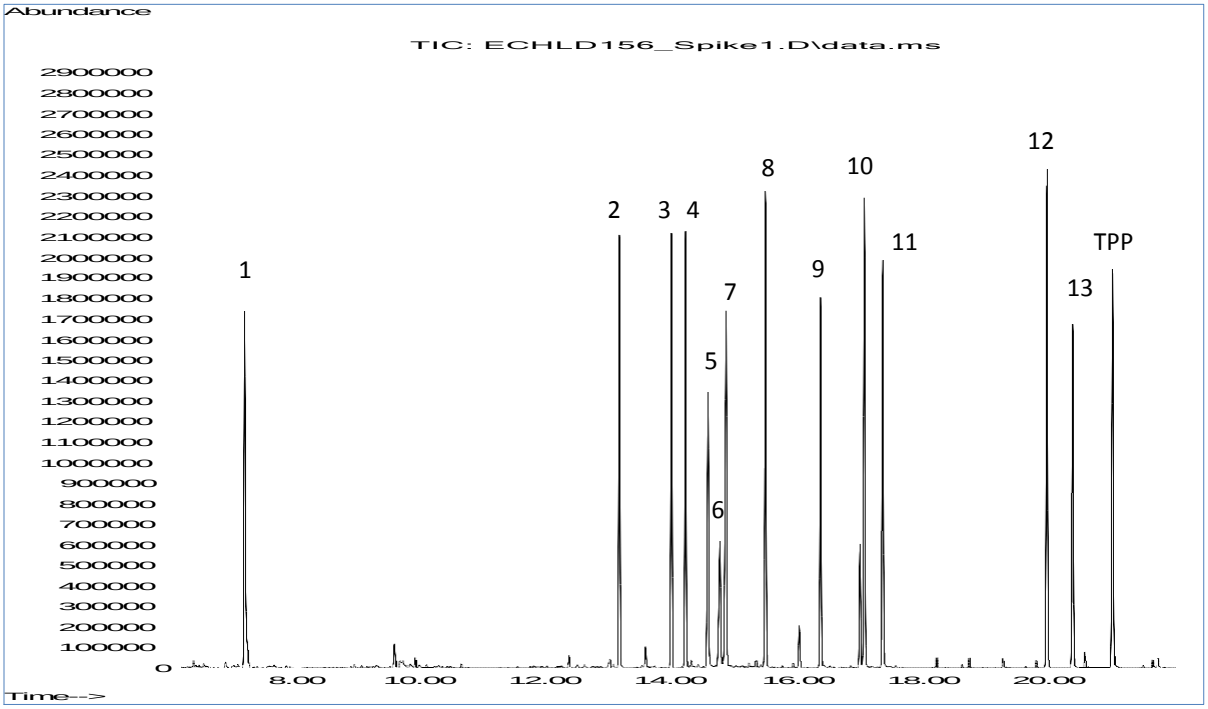
Accuracy and Precision Data

Compound	Class	Spiked (µg/L)	Recovery%	RSD% (n=5)
o,o,o-Triethyl phosphorothioat	Organophosphorus	10	**90.7**	1.8
Thionazin	Organophosphorus	10	**98.3**	1.8
Sulfotep	Organophosphorus	10	**100.5**	1.0
Phorate	Organophosphorus	10	**94.2**	1.2
Dimethoate	Organophosphorus	10	**80.4**	7.4
Simazine	Triazine	10	**100.1**	2.1
Atrazine	Triazine	10	**103.5**	1.1
Disulfoton	Organophosphorus	10	**88.1**	1.7
Methyl	Organophosphorus	10	**105.9**	1.8
Malathion	Organophosphorus	10	**109.4**	1.3
Parathion	Organophosphorus	10	**106.0**	1.3
Ethion	Organophosphorus	10	**108.0**	0.7
Famphur	Organophosphorus	10	**105.0**	1.9
		Overall mean	**99.2**	**1.9**

Chromatogram - Blank Sample



Chromatogram - Sample spiked with 10 µg/L analytes



Peak list: 1. o,o,o-Triethyl phosphorothioate; 2. Thionazin; 3. Sulfotep; 4. Phorate; 5. Dimethoate; 6. Simazine; 7. Atrazine; 8. Disulfoton; 9. Methyl parathion; 10. Malathion; 11. Parathion; 12. Ethion; 13. Famphur.

UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 • 800.385.3153 • 215.781.9255 • www.unitedchem.com • Email: methods@unitedchem.com