Determination of Explosives in Drinking Water by Solid Phase Extraction and GC/MS Detection

UCT Part Numbers

ECHLD156-P
500 mg Enviro-Clean® HL DVB
in 6 mL cartridge

ECSS15M6
5 g anhydrous sodium sulfate
in 6 mL cartridge

AD0000AS
Cartridge adaptor

Summary:

EPA method 529 determines a variety of explosives and related compounds in finished drinking water. The analytes have sufficient volatility and thermal stability for GC/MS analysis and can be partitioned from aqueous samples onto a DVB solid phase extraction (SPE) sorbent. UCT has developed a novel DVB sorbent for the extraction of explosives in water. One liter of sample is passed through a 6-mL SPE cartridge packed with 500 mg of the DVB sorbent using a sample transfer tube. The explosives are retained on the DVB sorbent and then eluted with ethyl acetate (EtOAc). A drying cartridge packed with 5 grams of anhydrous sodium sulfate is attached to the bottom of the SPE cartridge using a cartridge adaptor in the elution step, eliminating the need of an additional extract drying step. Excellent recoveries and minimum lot-to-lot variations were obtained using this new DVB sorbent.

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Procedure:

1. Cartridge Conditioning

a) Preserve 1 L of the water sample with 50 mg sodium sulfite (dechlorinating agent), 0.5 g copper sulfate pentahydrate (microbial inhibitor) and 5 g pH 7 Trizma Pre-set Crystals (pH buffer).
b) Spike with appropriate amounts of surrogates, and target analytes for fortified samples.
c) Attach the SPE cartridges onto a multi-position manifold.
d) Wash the SPE cartridges with 5 mL EtOAc - pass 1/3 through the cartridge to wet the sorbent, allow the EtOAc to soak for 1 min before drawing the remaining solvent through. Repeat this process 2 additional times. Dry under full vacuum for 1 min.
e) Condition the cartridges with 3 aliquots of 5 mL methanol each. During this step and in subsequent steps do not allow the sorbent to go dry until instructed to do so in the drying step (step 8).
f) Equilibrate the cartridges with 2 aliquots of 10 mL DI water. After the second addition leave about 4 mL of water in the cartridge. Attach sample transfer lines (available from Restek p/n 26250) to the top of the SPE cartridges (**ECHLD156-P**).

2. Sample Extraction

a) Insert the weighted ends of the transfer lines into the 1 L sample bottles and draw the entire sample through the SPE cartridge in a fast, drop-wise fashion (10-15 mL/min).
b) Remove the transfer lines from the SPE cartridges and dry the SPE cartridges under full vacuum for 10 min.
c) Attach the drying cartridges (**ECSS15M6**) to the bottom of the SPE cartridges with the cartridge adaptors (**AD0000AS**).

3. Cartridge Elution

a) Insert a collection tube or vial into the manifold underneath each SPE cartridge.
b) Rinse each sample bottle with 5 mL EtOAc, and pull the rinsate through the SPE cartridges slowly using the transfer line. Turn full vacuum on for 1 min to pull all of the elution solvent into the collection container.
c) Remove the transfer line from the SPE cartridge. Add 5 mL EtOAc to the SPE cartridge, pass 1/3 though the cartridge, allow the cartridge to soak for 1 min and then draw the remainder through in a slow drop-wise fashion.
d) Concentrate the eluate to about 0.9 mL under a gentle stream of nitrogen at 40 °C.
e) Add internal standard and adjust the final volume to 1 mL with EtOAc. The samples are ready for GC/MS analysis.

4. Sample Analysis

GC/MS Method	
GC/MS	Agilent 6890N GC coupled with 5975C MSD
Injector	1-2 µL PTV or cold on-column injection
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 50 °C, hold for 1 min; ramp at 8 °C/min to 210 °C; ramp at 20 °C/min to 250 °C, hold for 2 min.
Tune	bfb.u
Full Scan	45-250 amu

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Results:

Accuracy and Precision Data

		Single lot		Multiple lots (5)	
Analyte	Spiked (µg/L)	Ave Recovery %	RSD % (n=5)	Ave Recovery %	RSD % (n=25)
Nitrobenzene d5 Surr	5	**92.4**	3.5	**88.9**	4.3
Nitrobenzene	5	**86.8**	2.7	**88.8**	4.6
2-Nitrotoluene	5	**87.6**	3.6	**89.1**	4.7
3-Nitrotoluene	5	**86.6**	3.6	**87.7**	4.6
4-Nitrotoluene	5	**84.4**	3.3	**87.2**	4.9
1,3-Dinitrobenzene	5	**102.4**	5.3	**99.7**	4.2
2,6-Dinitrotoluene	5	**98.2**	5.7	**97.3**	4.8
2,4-Dinitrotoluene	5	**91.2**	5.3	**92.9**	4.2
1,3,5-Trinitrobenzene	5	**100.0**	9.1	**100.4**	5.5
2,4,6-Trinitrotoluene	5	**103.0**	6.3	**100.9**	5.3
RDX	5	**107.0**	1.7	**111.1**	5.8
4-Amino-2,6-Dinitrotoluene	5	**100.1**	7.5	**99.6**	5.8
3,5-Dinitroaniline	5	**104.3**	5.6	**103.6**	6.3
2-Amino-4,6-Dinitrotoluene	5	**103.3**	5.2	**105.7**	5.0
Tetryl	5	**102.2**	3.7	**105.4**	4.7
	Overall Mean	**96.6**	**4.8**	**97.2**	**5.0**

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4102-36-02

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