



EPA Method 529 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

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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.

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 through 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

Results:

Accuracy and Precision Data

Target analytes	Spiked (µg/L)	Single lot		Multiple lots (5)	
		Ave Recovery%	RSD% (n=5)	Ave Recovery%	RSD% (n=25)
Nitrobenzene d5 (Surrogate)	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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