EPA Method 528 Determination of Phenols 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 - 5g anhydrous sodium sulfate in 6 mL cartridge
AD0000AS - Cartridge adaptor

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Summary:

EPA method 528 determines 12 phenols in finished drinking water using solid phase extraction (SPE) and GC/MS detection. Among the 12 phenols, 10 are listed as priority pollutants by U.S. EPA, the two exceptions are 2-methylphenol and 4-chloro-3-methylphenol. UCT has developed a new polystyrene divinylbenzene material for EPA method 528. One liter of drinking water sample is passed through the SPE cartridge using a large sample delivery tube. Phenols in the water samples are retained to the sorbent by hydrophobic interaction, and are eluted thereafter with dichloromethane (DCM). A drying cartridge packed with anhydrous sodium sulfate is attached to the end of the SPE cartridge in the elution step, eliminating the eluate drying step. This cross linked DVB sorbent demonstrates superior extraction performance with recoveries ranging from 85.1 to 108.4% and minimum lot-to-lot variations < 10% for 7 tested sorbent lots.

Experimental:

Sample pretreatment:

a) De-chlorinate 1 L of drinking water sample with 40-50 mg sodium sulfite if free chlorine is present, and acidify the sample to pH ≤ 2 with 6 N HCl.
b) Spike with appropriate amounts of surrogates, and target analytes for fortified samples.

SPE procedure:

a) Connect the large sample delivery tubes to the top of the SPE cartridges (**ECHLD156**-**P**), and attached the cartridges to a SPE manifold.
b) Wash the cartridges with 3 aliquots of 5 mL DCM. Condition the cartridges with 3 aliquots of 5 mL methanol. Do not let the sorbent go dry after applying the 3rd aliquot of methanol. Equilibrate the cartridges with 10 mL of 0.05 N HCl.
c) Insert the stainless steel ends of the sample delivery tubes into the sample containers, and draw the entire sample through the SPE cartridge at a fast drop-wise fashion (about 20 mL/min).
d) Remove the sample delivery tubes from the SPE cartridges and dry the cartridges under full vacuum for 15 min.
e) Attach the drying cartridges (**ECSS15M6**) to the bottom of the SPE cartridges with cartridge adaptors (**AD0000AS**) such that the elution solvent will pass through the SPE cartridge first and then the drying cartridge.
f) Insert test tubes or glass vials into the manifold, elute the SPE cartridges with 5 mL DCM, and repeat with sample bottle rinse using 10 mL DCM.
g) Concentrate the eluate to between 0.7 and 0.9 mL under a gentle stream of nitrogen at 35 ºC.
h) Add internal standards and adjust final volume to 1 mL with DCM. The samples are ready for GC/MS analysis.

GC/MS method:

GC/MS: Agilent 6890N GC with 5975C MSD
Injector: 1 µL splitless injection at 200 ºC
Liner: 4 mm splitless gooseneck liner with deactivated glass wool (**GCLGN4MM**)
GC column: Restek Rxi®-5sil MS 30m*0.25mm*0.25µm with 10 m guard column
Carrier gas: Helium at a constant flow of 1.0 mL/min
Oven: Initial temperature of 40 ºC, hold for 6 min; ramp at 8 ºC/min to 250 ºC.

Solvent delay: 10 min

Tune: dftpp.u

Full Scan: 45-350 amu

Results:

Accuracy and Precision Data

Target analytes	Spiked (µg/L)	Single lot		Multiple lots (7)	
		Ave Recovery%	RSD% (n=5)	Ave Recovery%	RSD% (n=35)
Phenol	10	**88.2**	2.2	**86.4**	4.0
2-chlorophenol	10	**87.4**	1.3	**85.3**	3.5
2-methylphenol	10	**88.6**	1.5	**86.8**	3.6
2-nitrophenol	10	**85.6**	0.8	**85.5**	3.8
2,4-dimethylphenol	10	**88.4**	1.1	**85.1**	6.5
2,4-dichlorophenol	10	**87.4**	1.3	**86.5**	3.8
4-chloro-3-methylphenol	10	**90.4**	1.0	**89.5**	2.9
2,4,6-trichlorophenol	10	**88.3**	0.6	**87.8**	3.2
2,4-dinitrophenol	10	**103.2**	7.6	**108.4**	5.6
4-nitrophenol	10	**96.5**	1.2	**97.4**	4.2
2-methyl-4,6-dinitrophenol	10	**92.9**	2.5	**97.9**	6.7
Pentachlorophenol	10	**94.3**	1.1	**95.8**	4.7
Surrogates					
2-chlorophenol d4	2.5	**88.7**	1.6	**87.3**	4.7
2,4-dimethylphenol d3	2.5	**88.5**	1.4	**86.9**	6.6
2,4,6-tribromophenol	5.0	**88.7**	0.9	**89.5**	4.3

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