



EPA Method 508.1: Determination of Chlorinated Pesticides, Herbicides, and Organohalides by Liquid-Solid Extraction and GC-ECD

UCT Part Numbers

CEC18156

Clean-Up® C18 endcapped
500mg/6mL

VMFSTFR12

Large volume sample
transfer tubes

VMF016GL

16 position glass block manifold

VMF02125

12 position large
volume collection rack

ECSS15M6

Enviro-Clean® Sodium Sulfate
5g/6mL w 50µm Teflon Frits

AD0000AS

Cartridge Adapters
(1, 3, 6, 10, and 15 mL cartridges)



Summary:

Most chlorinated pesticides, including herbicides and organohalides, have been banned in the U.S. due to the significant toxicity to plants, animals, and human beings [1]. These toxic compounds are lipophilic and could accumulate in adipose tissues causing many illnesses, such as mood disorders, learning and memory difficulties, diabetes, certain cancers, and heart disease [2]. Human exposure to chlorinated compounds includes digestion of the contaminated food, especially the animal originated food which may contain these bio-accumulated chemicals, as well as drinking water which contains chlorinated chemicals leached from contaminated soil into drinking water reservoirs. Besides the food and drinking water exposures, breastfeeding and trans-placental transfer are two additional exposure routes to infants [2]. The U.S. EPA has regulated chlorinated compounds to be tested by method 508.1, using liquid-solid extraction with C18 sorbent either in disk or cartridge format [3].

This application note outlines the liquid-solid extraction procedure using C18 cartridges to extract 21 representative chlorinated compounds in drinking water. A 1-liter sample is dechlorinated and acidified before passing through the pre-conditioned C18 cartridge, the retained analytes are then eluted with ethyl acetate (EtOAc) and dichloromethane (DCM). Instead of drying the eluate after elution, a drying tube containing 5 grams of sodium sulfate is attached to the bottom of the C18 cartridge with a cartridge adapter, providing simultaneous elution and eluate drying. Finally, the eluate is concentrated to 1 mL and analyzed by GC-ECD or GC/MS. With C18 cartridge extraction, all analytes passed the method QC acceptance criteria, 70-130% for recovery and $\leq 30\%$ for RSD, even for hexachlorocyclopentadiene, the notorious troublesome compound, with an 83.4% recovery obtained, which is much greater than the 21.7% listed in EPA method 508.1 (Table 6) or the 53% declared by 3M using 47mm C18 Empore™ disks [4].



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Sample Pretreatment:

1. Residual chlorine should be reduced by adding 50 mg/L of sodium sulfite.
2. After dechlorination, acidify the sample to $\text{pH} \leq 2$ using 6 N HCl.
3. Spike with appropriate volumes of surrogate and standard working solutions prepared in methanol.
4. Add 5 mL methanol and mix well.

Liquid-solid Extraction Procedure:

1. Cartridge Conditioning

- a) Attach the large volume sample transfer tubes (**VMFSTFR12**) to the top of the C18 cartridges (**CEC18156**) and position the connected cartridges onto a 16-position glass block manifold (**VMF016GL**).
- b) Insert the stainless steel ends of the transfer tubes into a beaker of EtOAc (5 mL per sample), apply a low vacuum to wet the sorbent, and let soak for 1 min before drawing to waste. Turn full vacuum on for 30 sec.
- c) Repeat 1.b) with 5 mL of DCM per sample.
- d) Insert the stainless steel ends of the transfer tubes into a beaker of methanol (10 mL per sample), and slowly draw methanol through the cartridge leaving a thin layer above the frit.
- e) Repeat 1.d) with deionized water (10 mL per sample) leaving a layer of about 1" above the frit.

2. Sample Loading

- a) Insert the stainless steel ends of the transfer tubes into each associated sample bottle.
- b) Adjust the vacuum so that the flow rate is approximate 20 mL/min (in a fast dropwise fashion).

3. Cartridge Drying

- a) Dry the C18 cartridges under full vacuum for 10 min.

4. Simultaneous Elution and Eluate Drying

- a) Attach the sodium sulfate drying tubes (**ECSS15M6**) to the manifold, and rinse with 5 mL of EtOAc.
- b) Attach the drying tubes to the bottom of the C18 cartridges using the cartridge adapters (**AD0000AS**).
- c) Insert the 12-position collection rack (**VMF02125**) with 40 mL VOA glass vials into the manifold to collect eluates.
- d) Rinse the sample bottles with 5 mL of EtOAc and elute the analytes from the cartridges by slowly pulling the 5 mL rinsate through the sample transfer tubes and the C18 cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion. After the elution solvent has been passed through, apply full vacuum for 30 seconds so that all of the elution solvent is collected.
- e) Repeat 4.d) with 10 mL of DCM.

5. Eluate Evaporation

- a) Remove the collection rack from the manifold.
- b) Evaporate the eluates to about 0.8 mL using TurboVap under a gentle stream of nitrogen (10 psi) at 40°C.
- c) Add internal standard, adjust the final volume to 1 mL with EtOAc.
- d) Transfer the extracts to 2-mL autosampler vials, and inject 1 μL to GC-ECD or GC/MS for analysis.



Results:

Comparison of Recoveries and RSD Obtained by EPA Method 508.1 and the UCT Method

Compound Name	EPA Method 508.1*		UCT Method**	
	Recovery%	RSD%	Recovery%	RSD%
Aldrin	73.6	8.9	78.6	3.9
Chlordane-alpha	91.9	4.9	94.0	4.2
Chlordane-gamma	74.5	7.5	99.9	4.5
4,4'-DDD	95.0	10.3	96.8	7.2
4,4'-DDE	96.5	5.0	105.9	5.1
4,4'-DDT	85.0	9.5	89.2	7.9
Dieldrin	92.3	9.9	109.2	4.4
Endosulfan I	91.8	8.6	99.3	4.9
Endosulfan II	92.0	7.4	109.4	5.5
Endosulfan sulfate	98.3	7.3	97.7	7.9
Endrin	102.0	10.3	114.3	7.3
Endrin aldehyde	69.8	5.2	107.5	6.8
HCH-alpha	95.4	6.7	98.5	3.6
HCH-beta	91.2	7.8	97.9	2.8
HCH-delta	97.6	5.9	103.5	3.5
HCH-gamma	106.0	7.1	97.9	3.2
Heptachlor	91.6	7.8	100.3	3.7
Heptachlor epoxide	71.4	9.8	103.5	4.0
Hexachlorobenzene	65.3	8.9	85.4	3.8
Hexachlorocyclopentadiene	21.7	40.1	83.4	3.5
Methoxychlor	96.1	12.0	119.4	9.1
QC Failures	3	1	0	0

*: Adopted from Table 6 in EPA method 508.1 using C18 disks

** : Obtained from 5 replicates of laboratory fortified blank samples spiked at 0.1 µg/L extracted using C18 cartridges

Conclusion:

A simple and effective liquid-solid extraction method using UCT's C18 cartridges has been demonstrated. Compared to the recovery and RSD values using C18 disk in EPA method 508.1, UCT's C18 cartridges showed much better analytical performance with all parameters passed the QC acceptance criteria set by the EPA method, even for one of the most troublesome compounds, hexachlorocyclopentadiene. In contrast, 4 parameters including 3 recoveries and 1 RSD failed the QC requirements when using C18 disks in method 508.1.

References:

[1] <http://dhss.delaware.gov/dhss/dph/files/organochlorpestfaq.pdf>

[2] <https://www.gdx.net/product/chlorinated-pesticides-test-serum>

[3] http://www.caslab.com/EPA-Methods/PDF/508_1.pdf

[4] http://solutions.3m.com/3MContentRetrievalAPI/BlobServlet?lmd=1139584675000&locale=en_US&assetType=MM

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