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## **METHOD 508.1**

### **I. SCOPE AND APPLICATION:**

This method utilizes disk liquid/solid extraction (LSE) disks and gas chromatography with an electron capture detector (ECD) to determine 29 chlorinated pesticides, three herbicides, and four organohalides in drinking water, groundwater, and drinking water in any treatment stage. LSE cartridges may also be used to carry out sample extractions. Single laboratory accuracy, precision and method detection limit data have been determined for the following compounds:

<b><u>Analyte</u></b>	<b><u>Chemical Abstract Services Registry Numbers (CASRN)</u></b>
Aldrin	309-00-2
Chlordane-alpha	5103-71-9
Chlordane-gamma	5103-74-2
Chlorneb	2675-77-6
Chlorobenzilate (a)	501-15-6
Chlorothalonil	2921-88-2
DCPA	1897-45-6
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Etridiazole	2593-15-9
HCH-alpha	319-84-6

<u>Analyte</u>	<u>Chemical Abstract Services Registry Numbers (CASRN)</u>
HCH-beta	319-85-7
HCH-delta (a)	319-86-8
HCH-gamma (Lindane)	58-89-9
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Methoxychlor	72-43-5
cis-Permethrin	52645-53-1
trans-Permethrin	52645-53-1
Propachlor	1918-16-7
Trifluralin	1582-09-8
Arochlor 1016*	12674-11-2
Arochlor 1221*	11104-28-2
Arochlor 1232*	11141-16-5
Arochlor 1242*	53469-21-9
Arochlor 1248*	12672-29-6
Arochlor 1254*	11097-69-1
Arochlor 1260*	11096-82-5
Toxaphene*	8001-35-2
Chlordane*	57-74-9

\* The extraction conditions of this method are comparable to U.S. EPA Method 608, which does measure the multi component constituents: commercial polychlorinated biphenyl (PCB) mixtures (Aroclors), toxaphene, and chlordane.

(a) These compounds are only qualitatively identified in the National Pesticide Survey (NPS) Program. These compounds are not quantitated because control over precision has not been demonstrated.

## II. REAGENTS:

- Sodium Thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) solution

### III. MATERIALS:

- 1-liter amber borosilicate sample bottle fitted with screw caps lined with TFE-fluorocarbon. Collect duplicate samples.
- Latex gloves
- Folding card table
- Pool and Spa 3-Way Test Strips (Chem Lab Products, Inc.)
- Paper towels
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Protective eyewear
- Pliers

### IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.
2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.
3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.
4. Remove the cap from the 1-liter container. Do not rinse the container as it has already been acid rinsed and may already contain sodium thiosulfate as a preservative.
5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.
6. Remove the bottle from the flow and recap. Invert the sample bottle five times.
7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

#### **IV. PROCEDURE (continued):**

8. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C. Collect duplicate samples.
9. If chlorine is present, add 5 drops of sodium thiosulfate solution, recap the bottle firmly and invert 5 times. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.
10. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C. Remember to collect duplicate samples.
11. Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

#### **V. SAMPLE TRANSPORT:**

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each 1-liter bottle. The 1-liter bottle must be amber colored to reflect sunlight since some of the pesticides analyzed for in this method are light sensitive and degrade when exposed to ultraviolet radiation. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use “blue ice” as the voa samples will never adequately chill. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

## VI. SAMPLE PRESERVATION:

Preservation study results indicate that most of the target analytes present in the samples are stable for 7 days when stored according to the above referenced conditions. Preservation data for the analytes chlorthalonil, alpha-HCH, delta-HCH, gamma-HCH, cis-permethrin, trans-permethrin, and trifluralin are nondefinitive, and therefore if these are analytes of interest, it is recommended that the samples be analyzed immediately.

## VII. DEFINITIONS:

- A. *Sodium Thiosulfate* ( $Na_2S_2O_3$ ): A preservative use to dechlorinate water samples. Reduces free chlorine into acid.

## VIII. SAFETY:

In the past, Method 508 required that the sample be preserved using mercuric chloride ( $Cl_2Hg$ ) so that the samples final  $Cl_2Hg$  concentration was 10 mg/l. The purpose of this preservation test was to prevent microbial degradation of the pesticides. But recently EPA has issued a decree that the use of mercuric chloride for preservation is no longer required. EPA has addressed the potential problem of microbial degradation by adjusting the holding times of the sample. The driving force for this change came about because of the highly toxic nature of mercuric chloride. Not only was it an additional hazard to sample handlers and the analysts but it also created more stringent requirements with respect to sample disposal.

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

## IX. SUMMARY OF METHOD:

METHOD 508.1--The sample volume is measured and spiked with 5 ml of methanol (a wetting agent for the cartridges), and with a surrogate (and any target compounds for quality control purposes) then transferred into a 2-liter separatory funnel with a solvent reservoir connected beneath it. The solvent reservoir has the liquid/solid extraction (LSE) cartridge attached below it and the LSE cartridge leads into a vacuum flask. (see figure 1A). As the sample passes from the separatory funnel through the LSE cartridge, the LSE cartridge extracts the target compounds from the water. The target compounds are then desorbed from the LSE cartridge using ethyl acetate, passed through an anhydrous sodium sulfate drying column and collected in an evaporation flask. The drying column is washed with at least 2 ml of methylene chloride in which the eluant is also collected in the evaporation flask. The "dried" eluant is then concentrated (evaporated) to a volume of approximately 0.8 ml and brought to a volume of 1.0 ml using ethyl acetate.

**IX. SUMMARY OF METHOD (continued):**

Once the extraction is complete, 2  $\mu$ l of the extract is injected into the gas chromatograph and the analytes in the extract are separated in the column of a GC having a temperature program. The chlorinated pesticides, herbicides, and organohalides are detected and quantitated using an electron capture detector (ECD).