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METHOD 507

I. SCOPE AND APPLICATION:

This is a gas chromatographic (GC) method applicable to the determination of certain nitrogen- and phosphorous-containing pesticides in groundwater and finished drinking water. The following compounds can be determined using this method.

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Alachlor	15972-60-8
Ametryn	834-12-8
Atraton	1610-17-9
Atrazine	1912-24-9
Bromacil	314-40-9
Butachlor	23184-66-9
Butylate	2008-41-5
Carboxin	5264-68-5
Chlorpropham	101-21-3
Cycloate	1134-23-2
Diazinon**(a)***	333-41-5
Dichlorvos	62-73-7
Diphenamid	957-51-7
Disulfoton****	298-04-4
Disulfoton sulfone****	2497-06-5
Disulfoton sulfoxide **(a)***	2497-07-6
EPTC	759-94-4
Ethoprop	13194-48-4
Fenamiphos	22224-92-6
Fenarimol	60168-88-9
Fluridone	59756-60-4

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Hexazinone	51235-04-2
Merphos*	150-50-5
Methyl paraoxon	950-35-6
Metolachlor	51218-45-2
Metribuzin	21087-64-9
Mevinphos	7786-34-7
MGL 264	113-48-4
Molinate	2212-67-1
Napropamide	15299-99-7
Norflurazon	27314-13-2
Pebulate	1114-71-2
Prometon	1610-18-0
Prometryn	7287-19-6
Pronamide **(a)***	23950-58-5
Propazine	139-40-2
Simazine	122-34-9
Simetryn	1014-70-6
Stirofos	22248-79-9
Tebuthiuron	34014-18-1
Terbacil	5902-51-2
Terbufos **(a)***	13071-79-9
Terbutryn	886-50-0
Triademefon	43121-43-3
Tricyclazole	41814-78-2
Vernolate	1929-77-7

***** 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 (Arochlors), 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 ($Na_2S_2O_3$) solution

III. MATERIALS:

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

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.

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. Thoroughly 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/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.

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 semi-volatile samples for transportation. Never use “blue ice” as the samples may not chill adequately. 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 method analytes present in the samples are stable for 14 days when stored according to the above referenced conditions. The analytes disulfoton sulfoxide, diazinon, pronamide, and terbufos exhibited significant aqueous instability, and samples to be analyzed for these compounds must be extracted immediately. The analytes carboxin, EPTC, fluridone, metolachlor, napropamide, tebuthiuron, and terbacil exhibited recoveries of less than 60% after 14 days.

VII. DEFINITIONS:

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

B. *Aqueous:* A solution or solvent that is water based.

VIII. SAFETY:

In the past, Method 507 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 was no longer mandatory. 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 and the 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 507--The sample volume is spiked with a surrogate (and a known amount of any target compounds used for quality control purposes), measured and then transferred into a 2-liter separatory funnel. The pH is adjusted to 7 using a phosphate buffer, and 100g of NaCl are added to “salt out” the mixture. The extraction process begins by adding 300 ml of methylene chloride to the sample bottle that is capped and mixed to rinse the inner walls of the bottle and then transferred to a separatory funnel. The sample is extracted by shaking the separatory funnel in a mechanical tumbing device for 1 hour. The organic layer of methylene chloride is allowed to separate from the water sample. The organic extract layer is removed and its volume measured.

IX. SUMMARY OF METHOD (continued):

The methylene chloride extract is concentrated through evaporation to a volume of about 10-20 ml and passed through a drying column containing about 10 cm of anhydrous sodium sulfate and collected in an evaporation flask. The drying tube is rinsed with about 20-30 ml of methylene chloride to remove any target compounds that may have adhered to the drying column and the rinsate added to the evaporation flask. The "dried" extract is then concentrated to a volume of 2 ml and 5-10 ml of methyl-tert-butyl ether (MTBE) is added to the evaporation flask. This procedure is referred to as solvent exchange. MTBE is not as volatile as methylene chloride so the methylene chloride solvent containing the target compounds is replace by the MTBE solvent. The resulting mixture is concentrated to about 2 ml and enough MTBE is added to bring the volume up to 5 ml.

The analysis begins by the injection of 2µl the extract into a gas chromatograph. from which conditions are selected which permit the separation and measurement of the analytes by a capillary column GC with a nitrogen phosphorous detector (NPD).