

Quality Assurance Document

Standard Operating Procedure

TITLE: Extraction of Water Samples for Organochlorine Pesticides/PCBs

DEPARTMENT: Semivolatile Organic Extractions

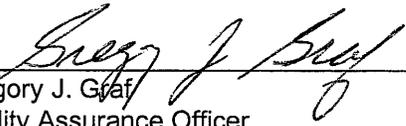
REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 method 3510C (Dec. 1996)

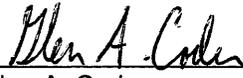
Code of Federal Regulations
USEPA 40CFR (1988), Pt.136, App. A, Method 608

PROCEDURE SUMMARY:

A measured volume of water sample, approximately one liter, is serially extracted with methylene chloride at a neutral pH using a separatory funnel. The methylene chloride extracts are dried and exchanged to hexane and concentrated to a volume of 10 mL. The samples are analyzed by either Method 8081A or Method 8082.

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QUALITY CONTROL:

Note: If sample extracts are to be analyzed by **both** Method 8081A and 8082, a separate LCS and MS/MSD should be extracted at the frequencies described below.

- The sample holding time is 7 days from date of sampling.
- One method blank is extracted per 20 samples OR per extraction batch whichever is more frequent. Reagent method blanks are prepared from laboratory de-ionized water.
- A laboratory control spike is extracted per 20 samples OR per extraction batch whichever is more frequent. Control spikes are prepared from laboratory de-ionized water spiked with the appropriate solution.
- Surrogate standards should be added to all samples, laboratory control spikes, matrix spikes, and method blanks. Surrogates are used to monitor unusual matrix effects, sample processing problems, etc.
- A matrix spike and a matrix spike duplicate will be performed for every 20 samples. The time frame for the 20 samples cannot extend beyond 14 days. If insufficient volume or matrix problems do not allow this, two laboratory control spikes may be prepared instead. Matrix spike compounds are used to indicate the presence or absence of unusual matrix effects.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

MATERIALS AND APPARATUS:

- | | |
|--------------------|---|
| Separatory funnel: | 2000 mL with Teflon stopcock. |
| Concentrator tube: | Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be demonstrated prior to use. |
| Evaporation flask: | Kuderna-Danish 300 mL (Reliance G-9601-001 or equivalent). Attach to concentrator tube with clips. |
| Snyder column: | Kuderna-Danish, three-ball macro (Kontes K-503000-0121 or equivalent). |
| Vials: | Amber glass, 12 mL capacity with teflon-lined screw cap. |
| Funnel: | 150-gram capacity. |

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Glass wool:	Rinsed with methylene chloride.
Boiling chips:	Solvent rinsed and dried, approximately 10/40 mesh(silicon carbide or equivalent).
Sodium Sulfate:	Preheated at 400 ⁰ C for 4 hours in a crucible.
Water bath:	Heated, with concentric ring cover, capable of temperature control of ± 5 ⁰ C. The bath should be used in a hood.
Tube heater:	Kontes 720000-0000.
Pipets:	Disposable, 2 mL short-stem.
Syringes:	250-1000 L Gastight syringes (Hamilton 1000 series or equivalent).
Graduated Cylinder:	1000 mL.
pH paper:	Wide range pH paper.

REAGENTS:

Surrogate Spiking Solution:

The mixture contains the following components at 2.0 mg/mL:
DCB (decachlorobiphenyl)
TCMX (tetrachloro-m-xylene)

Matrix Spiking Solution: See Appendix A

Sodium hydroxide (NaOH): (10 Normal), dissolve 80 g of NaOH pellets in reagent water and dilute to 200 mL.

Sulfuric Acid (H₂SO₄): (1:1), slowly add 50 mL concentrated H₂SO₄ (specific gravity: 1.84) to 50 mL reagent water.

Methylene chloride, acetone, and hexane (pesticide grade).

EXTRACTION PROCEDURE OUTLINE:

- 1 Allow the sample to warm to room temperature, mark the sample volume on the sample container, then invert sample several times.
- 2 Set up and label separatory funnel, K-D apparatus, and funnel with glass wool and sodium sulfate.
- 3 Transfer sample volume to separatory funnel.

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- 4 Add 60 mL methylene chloride to sample container, swirl container several times, then transfer solvent to separatory funnel. **Record methylene chloride lot # on the extraction form.**
- 5 Add the required amount (500 μ L) of surrogate solution to each separatory funnel with a 1000 μ L syringe. **Record the amount of surrogate solution used and the reference number on the surrogate solution vial on the extraction form.**
- 6 Add the required volume (800 μ L of the pesticide matrix spiking solution or 1000 μ L of the PCB matrix spiking solution) to only those required samples using a 1000 μ L syringe. **Record the amount of spiking solution used and the reference number on the matrix spiking solution vial on the extraction form.**
- 7 Adjust sample pH between 5-9 with either 5 N sodium hydroxide or sulfuric acid (1:1). Check with wide range pH paper.
- 8 Shake separatory funnel for 1 to 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase. If an emulsion interface occurs between the phases, the technician must employ mechanical techniques to complete phase separation.

NOTE: Methylene chloride creates excessive pressure very rapidly, therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Venting of the separatory funnel should be into a hood to avoid needless exposure of the technician to solvent vapors.
- 9 Drain the extract through the funnel containing glass wool and sodium sulfate. Collect the extract in the K-D apparatus. Rinse the sodium sulfate with methylene chloride.
- 10 Add 60 mL methylene chloride to the separatory funnel and repeat steps 8 through 9. This process is repeated one more time. The total extract volume in the K-D apparatus should be approximately 200 mL.
- 11 Attach a Snyder three-ball column to the K-D apparatus and set aside for future concentration of sample extract.
- 12 Determine initial sample volume. Fill the original sample container from step 1 to the mark with water. Transfer contents to a 1000 mL graduated cylinder. Record the initial sample volume on the extraction form to the nearest 5 mL.

SAMPLE EXTRACT CONCENTRATION:

- 1 Place the K-D apparatus on a hot water bath (80° to 90° C) so that the concentrator tube is totally immersed in the hot water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 2-5 mL, add 50 mL hexane and continue concentration to an apparent volume of 4-6 mL. **Record the lot # for hexane on the extraction form.** Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

- 2 Rinse the Snyder column with 1-2 mL of hexane. Remove Snyder column and rinse the evaporating flask with 1-2 mL hexane.
- 3 Remove the evaporating flask from the concentrator tube and adjust the final extract volume to 10.0 mL with hexane.
- 4 Using a disposable pipette, transfer extract to a 12 mL amber vial.
- 5 Label the vial with project name, extraction date, sample number, and final volume.
- 6 Log sample into the analysts' extract storage refrigerator and complete all paperwork.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

Appendix A

MATRIX SPIKING SOLUTION

PCB Matrix Spike

10.0 mg/mL

Aroclor 1016, 1221, 1232, 1242, 1248, 1254, or 1260, (rotated periodically).

Pesticide Matrix Spike

0.5 mg/mL

g-BHC
a-BHC
Endosulfan I
Heptachlor
Aldrin
b-BHC
d-BHC
a-Chlordane
g-Chlordane
Heptachlor epoxide

1.0 mg/mL

4,4'-DDD
4,4'-DDT
Dieldrin
Endrin
4,4'-DDE
Endosulfan II
Endosulfan sulfate
Endrin aldehyde
Endrin ketone

5.0 mg/mL

Methoxychlor