United States Environmental Protection Agency

Office of Research and Development

The Children's Total Exposure to Persistent Pesticides and Other Persistent Organic Pollutants (CTEPP) Study

Extracting and Preparing Drinking Water Samples for Analysis of Persistent Organic Pollutants

Battelle
Columbus, OH 43201
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Standard Operating Procedure **CTEPP-SOP-5.23**

Title: Extracting and Preparing Drinking Water Samples for Analysis of Persistent Organic Pollutants

Source: Battelle

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Exposure Measurements & Analysis Branch

STANDARD OPERATING PROCEDURE (SOP) FOR EXTRACTING AND PREPARING DRINKING WATER SAMPLES FOR ANALYSIS OF PERSISTENT ORGANIC POLLUTANTS

Prepared by: _____ Date: _____

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1.0 Scope and Applicability

This standard operating procedure (SOP) describes the method for extracting and preparing drinking water samples for analysis of persistent organic pollutants (POP).

2.0 Summary of Method

The method for extracting and preparing a drinking water sample for analysis of target POP is summarized in this SOP. It covers the extraction and concentration of samples that are to be analyzed by gas chromatography/mass spectrometry (GC/MS).

3.0 Definition

3.1 Internal Standard (IS): The compounds that are added to sample extracts just prior to GC/MS analysis. The ratio of the detector signal of the native analyte to the detector signal of the corresponding IS is compared to ratios obtained for calibration curve solutions where the IS level remains fixed and the native analyte levels vary. The IS is used to correct for minor run-to-run differences in GC injection, chromatographic behavior, and MS ionization efficiency.

3.2 Matrix Spike Standard (MSS): The compounds that are used for QA/QC purposes to assess the recovery efficiency obtained for the individual samples. Known amounts of atrazine (typical 20 ng) are spiked into the water sample prior to extraction. The matrix spikes (MSs) are quantified at the time of analysis and their recoveries indicate the probable extraction and recovery efficiency for atrazine. The MSs are only generated for duplicate aliquots of selected water samples.

4.0 Cautions

Standard laboratory protective clothing, gloves, and eye covering is required.

5.0 Responsibilities

5.1 The project staff who performs the sample extractions will be responsible for obtaining samples from the sample coordinator, entering relevant information in the extraction/preparation laboratory record books, and sending final extracts for analyses.

5.2 The CTEPP Laboratory Team Leader (LTL), the QA Officer, and Task Order Leader (TOL) will oversee the sample extraction operation and ensure that SOPs are followed by all project staff.

6.0 Apparatus and Materials

6.1 Materials

6.1.1 Silylated small Soxhlet extraction apparatus consisting of condenser, extractor (31 mm id x 135 mm length), and flask (250 mL round bottom); (Kontes 585000-0021)

6.1.2 Analytical syringes

6.1.3 Silylated wide-neck glass funnels (muffled)

6.1.4 Large Kim-wipes (15" x 15")

6.1.5 Latex gloves

6.1.6 Silylated glass vials with Teflon-lined screw caps; muffled

6.1.7 Silylated 1.8 mL glass GC vials with Teflon-lined screw caps; muffled

6.1.8 Silylated Kuderna-Danish concentrators (large 24/40 3-ball Snyder condenser, 125 mL reservoir flask and 25 mL tube); (Kontes 570000)

6.1.9 Silylated small 19/22 3-ball Snyder condensers

6.1.10 Disposable glass pipettes (muffled and stored in clean glass jar)

6.1.11 Vortex mixer (American Scientific Products)

6.1.12 Graduated cylinders

6.1.13 Heated water bath

6.1.14 Quartz fiber filters

6.1.15 C_{18} solid phase extraction (SPE) cartridges

6.2 Reagents

6.2.1 Dichloromethane (DCM); distilled-in-glass

6.2.2 n-Hexane (distilled-in-glass)

6.2.3 Boiling chips (Hengar crystals)

6.2.4 Matrix Spiking Solution

6.2.5 Internal Standard Solution

6.2.6 Distilled, deionized water (DI water)

6.2.7 Diethyl ether (distilled-in-glass)

7.0 Procedure

7.1 Extraction and Concentration.

7.1.1 Remove an aliquot (100 mL) of the drinking water sample and place in a clean container. For matrix spike sample, know amount of matrix spiking solution will be spiked into the water sample.

7.1.2 Place reverse phase C_{18} SPE cartridges on the SPE manifold and condition each cartridge in sequence with 6 mL of 100% DCM, 6 mL of 100% of methanol, and 12 mL of distilled water. Close the valve stem on the manifold to prevent the cartridge from going dry between solvents.

7.1.3 Transfer the water sample to the adaptor of a C_{18} SPE cartridge.

7.1.4 After all the water sample is processed through the C_{18} SPE cartridge, the cartridge is air-dried for one hour under a slight vacuum (< 1" of mercury) and for another one hour under no vacuum. Elute the cartridge with 12 mL of 50% DCM in hexane.

7.1.5 Dry the collected sample extract with sodium sulfate, and filter it through a quartz fiber filter.

7.1.6 K-D concentrate the extract to 0.6 to 0.8 mL and rinse down the sides of the tube with hexane to bring the volume to 1 mL.

7.1.7 Spike the extract with 50 L of the Internal Standard solution (atrazine-d_5, 1.0 ng/µL), and vortex for 3 s to mix.

7.1.8 Transfer the extract, using a muffled disposable glass Pasteur pipette, to a clean prelabeled 1.8 mL GC vial for GC/MS analysis. Mark the volume on the side of the vial and label the sample with the laboratory notebook number and field sample ID.

7.2 Store the extract in a $\leq$-10˚C freezer until GC/MS analysis.

8.0 Records

8.1 Records of the field blank levels and field spike recovery values will be retained in a project laboratory record book that is kept in the extraction laboratory. This record book will serve as a continuing file for reference on expected performance of the methods and likely contaminant levels that will arise as a result of field handling. These samples will be identified in the laboratory record book by field sample ID and the assigned laboratory analysis number (a unique number that combines the 5 digit laboratory book number-2 digit page number-2 digit line number), the date of extraction, and the lot number of solvent used.

8.2 The record of the extraction of samples will be maintained in a project laboratory notebook that is kept in the extraction laboratory. This record book will contain the field sample ID, the assigned laboratory analysis number (see above), the date of extraction, and the lot number of solvent used for extraction. The record book will be retained in the laboratory where these operations are performed until the conclusion of the study and will be archived in a secure room for three years after completion of the study.

9.0 Quality Control and Quality Assurance

9.1 A field blank, laboratory method blank, and laboratory fortified blank that will be extracted together with the field samples. The field blank analyses are performed to verify that minimal contamination occurs through sample handling during shipping and field operations. The laboratory method blank analyses are performed to verify that minimal contamination occurs through sample preparation. The laboratory fortified blank analyses are performed to verify the recoveries of analytes preparation procedures.

9.2 Field crews will be reminded to wear clean clothing and shoes, to remove all pesticide products from their residences that may contain these analytes, and to refrain from using these materials while part of the field crew. Field crews will also be reminded to obtain clean clothing after visiting a home where they know or suspect that these pesticides have been applied within the previous week. Cigarette smoking is not permitted during the field sampling. Field crews should store the samples in a clean environment away from any known combustion sources.

9.3 Surrogate recovery values of 50-150% in blanks, and actual samples will be deemed acceptable, and no correction to the data will be made. For recoveries less than 50% or greater than 150%, the data will be flagged. For recoveries greater than 130%, the concentration of the surrogate spiking solution will be checked against a calibration curve to determine whether inadvertent solvent loss has resulted in higher spike levels. If this has occurred, the surrogate spiking solution will be re-prepared.

9.4 One laboratory method blank that is analyzed as a sample concurrently with a field sample set will be analyzed for typically every 50 samples processed. If significant target analyte levels (>0.01 µg) are found in the field blanks, the source of contamination must be identified and more laboratory blanks, together with additional field blanks, trip blanks, and storage blanks, will be analyzed.

10.0 Reference

10.1 J. C. Chuang, C. Lyu, Y-L Chou, P. J. Callahan, M. Nishioka, K. Andrews, M. A. Pollard, L. Brackney, C. Hines, D. B. Davis, and R. Menton, "Evaluation and Application of Methods for Estimating Children's Exposure to Persistent Organic Pollutants in Multiple Media." EPA/600/R-98/164a, EPA/600/R-98/164b, and EPA/600/R-98/164c (Volume I, II, and III), 1999.