TITLE: Removal of Sulfur from Sample Extracts Using Copper

MATRICES:

This SOP pertains to removing sulfur from any type of sample extract to be analyzed for PCBs.

DETECTION LIMITS:

Not applicable.

1.0 **SCOPE AND APPLICATION:**

1.1 Sulfur co-extracted from samples can cause difficulties during the analysis of organic compounds by gas chromatography due to co-elution. Sulfur may be removed from the sample extract with the use of copper granules. Caution: This method should not be used on extracts to be analyzed for pesticides.

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 Copper granules are added to sample extracts during the extraction procedure or after concentration. The mixture is agitated or shaken to allow the copper to react with the sulfur and precipitate out.

3.0 **DEFINITIONS**:

3.1 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the extraction and analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

3.2 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): Aliquots of an environmental sample to which a known quantity of the analyte(s) of interest is added in the laboratory. The MS and MSD are analyzed exactly like a sample. Their purpose is to quantify the bias and precision caused by the sample matrix.

3.3 Lab Control Spike (LCS) and Lab Control Spike Duplicate (LCSD): A method blank to which a known quantity of the analyte(s) of interest is added. The LCS, and LCSD when needed, is processed simultaneously with and under the same conditions as samples through all steps of the extraction and analytical procedures. Their purpose is to show complete extraction technique.

3.4 Surrogate: A substance with properties that mimic the analyte(s) of interest that is unlikely to be found in environment samples. Surrogate should be added to all samples, laboratory control spikes, matrix spikes, and method blanks and are used to monitor unusual matrix effects, sample processing problems, etc.

4.0 **INTERFERENCES:**

4.1 Solvents, reagents, glassware, and other sample processing may yield artifacts and /or interferences to sample analysis. All these materials are demonstrated to be free from interferences under the conditions of the procedure by analyzing method blanks.

5.0 **SAFETY:**

5.1 Safety is everyone's business at En Chem. Everyone is responsible for reducing unsafe and unhealthy working conditions or potential hazards whenever and wherever possible. En Chem provides its staff with a safe place to work, but it takes everyone's cooperation to keep it safe. If an employee sees something that does not look safe, or sees someone else working in an unsafe manner, he/she mentions it to a Supervisor.

5.2 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. Respirators and dust masks should be worn as needed. The toxicity or carcinogenic affect of each reagent and standard used in this method has not been precisely defined in this SOP. Therefore, each chemical compound used should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique and by working in a hood. A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis.

5.3 Required Safety Equipment is listed in Appendix A Table A.

6.0 **SUPPLIES:**

6.1 See Appendix A Table B for a summary.

7.0 **REAGENTS:**

7.1 See Appendix A Table C for a summary.

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE:**

8.1 Not applicable.

9.0 **QUALITY CONTROL:**

9.1 When this cleanup procedure is implemented, all samples, method blanks, laboratory control spike and matrix spike extracts undergo this process.

10.0 COPPER PREPARATION:

10.1 Place approximately 500 g of copper granules in a clean 250 ml beaker and add 150 ml of de-ionized (DI) water. Add 5 ml 65% nitric acid. Stir copper in solution with a spatula 3 minutes. Decant dilute acid mix from the copper granules and rinse four times with 200 ml DI water, again decanting after each rinse. Rinse and decant the copper granules twice with approximately 100mL of Acetone followed by a final 100mL rinse of Hexane. Remove copper from the beaker and place into a Pyrex baking dish. Allow to air dry and then transfer the copper to an appropriate storage container with a lid.

11.0 SULFUR REMOVAL FROM EXTRACTS:

Copper is introduced into a sample extract and allowed to react with any sulfur present. This may be done as follows:

11.1 **During Soxhlet or Automated Soxhlet Extraction**: If the sample is to be soxhlet extracted, 3 to 4 grams of copper granules may be added to the solvent vessel prior to heating where it serves a dual purpose: as boiling chips and to remove sulfur during the extraction process. More copper may be utilized if the samples are expected to have relatively high concentrations of sulfur, particularly sediment samples.

11.2 After extraction, the solvent extract may be poured from the vessel leaving the copper behind. The extract may need to be filtered or centrifuged to remove precipitate as needed. Further removal of sulfur from the extracts may be required.

11.3 **Sample Extracts**: Cleanup procedures such as gel permeation chromatography or column chromatography with Florisil or silica gel should be performed on samples prior to removing sulfur with copper mesh. In an 8 mL scintillation vial, add 3 to 4 grams of 20-30 mesh copper to about 2 ml of sample extract.

11.4 Cap the vial tightly and shake the vial by hand for one minute or swirl on a vortex mixer. For PCB analyses, the extract may be allowed to stand with the copper until the time of analysis.

11.5 Using a disposable pipette, transfer the extract into an injection vial and cap. If the extract has precipitate filter the extract using a 0.45 um syringe filter or centrifuge. If high concentrations of sulfur are expected or present, repeat this procedure until the sulfur is removed and no precipitation is present.

11.6 Sample is now ready for further cleanup if needed or analysis.

12.0 **CALCULATIONS:**

12.1 Not applicable.

13.0 **METHOD PERFORMANCE:**

13.1 Method performance is validated through the study of performance check and calibration standards and by the analysis of laboratory reagent blanks put through the copper cleanup method to meet quality control criteria for this procedure.

14.0 **POLLUTION PREVENTION:**

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff order, whenever possible, acceptable non-toxic alternative supplies. Staff also prepares only those quantities of reagents or standards that will be used prior to the expiration date. Any appropriate measures to minimize waste generation are brought to the attention of laboratory management.

15.0 **DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:**

15.1 Not applicable.

16.0 **CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA:**

16.1 Assessment of quality control measures provides a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. Corrective actions are found in the determinative SOP.

17.0 **CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA:**

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, based on received sample volumes, data generated that do not meet statistical goals are re-analyzed to see if the statistical goal can be achieved. When environmental samples do not meet statistical goals, unacceptable data is generated. These events are different from those pertaining to instrument operating conditions and occur when the instruments are operating under ideal conditions.

18.0 **WASTE MANAGEMENT:**

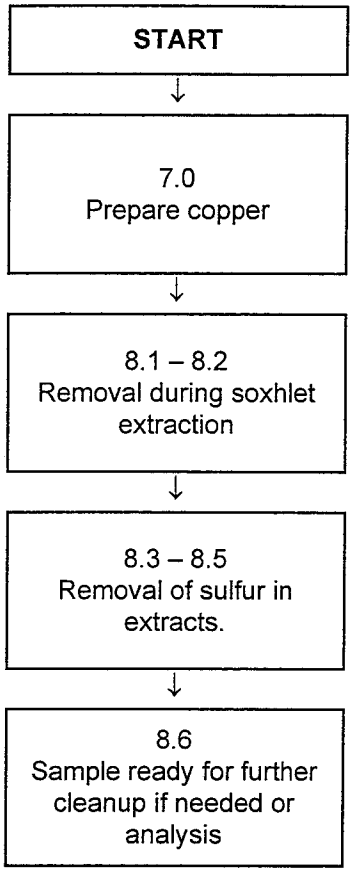
18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment, as fewer materials need to be disposed.

REFERENCES:

The procedure described in this SOP was based on all or parts of the following authoritative sources of methods for chemical assessment of environmental samples:

Test Methods for Evaluating Solid Waste, Third Edition. SW-846 3500B Organic Extraction and Preparation, Revision 2, December 1996.
Test Methods for Evaluating Solid Waste, Third Edition. SW-846 3660B Sulfur Cleanup, Revision 2, December 1996.

FLOWCHART


START
7.0
Prepare copper
8.1 – 8.2
Removal during soxhlet extraction
8.3 – 8.5
Removal of sulfur in extracts.
8.6
Sample ready for further cleanup if needed or analysis

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: Julie Trivedi — 4/25/03
Julie Trivedi
Quality Assurance Officer — Date

APPROVED BY: Glen A. Coder — 6/25/03
Glen Coder
Laboratory Manager — Date

Periodic Review Record

Review Date					
Initials					

Appendix A
TABLES

Table A
REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	√	
2	Lab Coat	√	
3	Gloves	√	
4	Respirator		√

Table B
CLEANUP SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
2 mL autosampler vials	VWR	VWR	66020-953
12 mL screw cap vials with caps		Fisher Scientific	03-391-7D
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free (large)	Best	Fisher Scientific	7005L
9" disposable pipettes	Fisher Scientific	Fisher Scientific	13-678-20-C

Table C
REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Di-ionized Water	Type I ASTM	----	----	----
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Hexane	Pesticide Quality	Burdick & Jackson	Fisher Scientific	217-4
Nitric Acid	65% huey	JT Baker	MG Scientific	
Copper 20-30mesh	ACS	Aldrich	Aldrich	

All reagents are stored at room temperature and environmental conditions.