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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF:
5HR-JCK-13

JUL 11 1990

CERTIFIED MAIL (P 185 141 072)
RETURN RECEIPT REQUESTED

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Mr. James E. Conley
Assistant Public Works Officer
Department of the Navy
Naval Weapons Support Center
Mailcode 5090/H11.8 0924
Crane, Indiana 47522-5000

RE: RFI Phase II Soils Work Plan
Naval Weapons Support Center
Crane, Indiana
IN5 170 023 498

Dear Mr. Conley:

The United States Environmental Protection Agency (U.S. EPA) has reviewed the RFI Phase II Soils Work Plan, dated December 7, 1989. Enclosed are our comments on areas that need to be revised and updated. Please submit a revised work plan to this Agency within 60 days of the date of this letter.

If you have any questions regarding this matter, please contact Ms. Carol Witt-Smith of my staff, at (312) 886-6146 for assistance.

Sincerely,

Lisa A. Pierard

Lisa Pierard
Acting Chief
RCRA Permitting Branch

Enclosure

cc: Jim Hunsicker, USNC
Bill Murphy, USAEWES
M. R. Beringer, Northern Div., Restoration Tech. Section
Jeff Ciocco, Northern Division
Dan Sparks, U.S. Fish & Wildlife Service
Tom Linson, IDEM

Comments on the
RCRA RFI Phase II Release Assessment
Work Plan for Soils
Naval Weapons Support Center (NWSC)
Crane, Indiana
IN5 170 023 498

1. Part B. RFI Phase II Sampling and Analysis Plan, Page 1

   NWSC should make sure volatiles are not impaired by the equipment used.

2. Table 1

   This table identifies the units for Phase II Soil Release Assessments. If this generalized Quality Assurance Plan (QAP) is to be used for all soil sampling, a second table should be added showing Phase II Soil Release Characterization is required for the Ammunition Burning Grounds (ABG).

3. Part B. RFI Phase II Sampling and Analysis Plan, Page 2

   a. Samples may not be composited, except for duplicate samples.

   b. For the purposes of decontamination and protection of the samples, sheet plastic or some other method of preventing samples and equipment from touching the ground should be used.

   c. The latest revision of EPA SW-846 must be used.

   d. Practical Quantification Limits (PQLs) should also be stated and included in Table 2.

   e. The bottle equipment list should be included.

   f. NWSC should tabulate the parameter, test method, detection limit, and PQL.

4. Table 2

   The methods should be updated to include the latest version of EPA SW-846.

5. Page 3

   The method for doing sieve analysis should be appendixed.

6. Page 3, Sample Types

   "...Samples will be collected randomly at depth....", should read, "...based on approved site specific locations for Phase II sampling...." instead of randomly.

7. Page 3, Subsurface Sampling

The following should be deleted: "Following the State of Indiana Department of Environmental Management (IDEM) guidelines, the first drive sample of each boring site will be split lengthwise and one half discarded."

When a core is taken, if the core is solid, the outer edges should be scraped and discarded to assure no contamination from the equipment has occurred. The sample should be a composite of the 6-inch lift, then another sample a composite of the next 6-inch lift, etc. If the edges cannot be scraped, the same compositing procedure applies without scraping. Only if stratigraphic samples are being taken along with analytical samples, should you cut the core in half, and use one half for stratigraphy and the other for analysis, yet assuring that the analytical sample is the most likely to confirm a release or not.

8. Page 4, Sampler Decontamination

The equipment should be brushed clean and then triple-rinsed.

9. Page 4, Paragraph 2

a. When taking samples, disposable gloves should be worn and changed after sampling, or decontaminated prior to continued use. Samples should be cut with a stainless steel knife.

b. The type of water used to decontaminate equipment should be identified.

10. Page 4, Environmental Conditions at the Time of Sampling

Environmental conditions must be logged in a field notebook. "Not applicable" is incorrect.

11. Figure 5

The sample label should include: identification for a composite sample under the grab heading, reference to the lab sample number, state that the sampler names should be signatures, and include the following parameters: TOC, Pesticides, Volatile Organics, Phenolics, Mercury, Cyanide, Organics GC/MS, and Energetics. See Attachment II CLP Protocols.

12. Figure 4

Identify that this figure is an example of a sample seal. A copy of the cooler seal should also be included.

13. Page 5, Decontamination Procedures

Again, any decontamination water used should be uncontaminated at the start and, triple-rinsing should be done.

14. Page 5, Documentation

a. Copies of all chain-of-custody papers must be included in the final report.

b. Computer printouts or logbooks of calibration documentation must be kept.

15. Figure 6

We would like to see modifications of the chain-of-custody to include information from EPA's standard CLP form (See Attachment II).

16. Page 7

Contingency plans should either be approved for the whole site, or included in the site specific plans.

17. Page 9

Before beginning the decontamination process, be sure the source water is uncontaminated. An explanation of handling of decontamination water should be included, especially final disposal.

18. Appendix 2

a. The SW-846 third edition must be used for Method 3540.

b. Method 8260 has been changed to Method 8270 in the SW-846 third edition. The method must be revised.

EPA SW 846 3rd Ed.

METHOD 3500

ORGANIC EXTRACTION AND SAMPLE PREPARATION

1.0 SCOPE AND APPLICATION

1.1 The 3500 Methods are procedures for quantitatively extracting nonvolatile and semivolatile organic compounds from various sample matrices. Cleanup and/or analysis of the resultant extracts are described in Chapter Four, Sections 4.2.2 and 4.3, respectively.

1.2 Method 3580 describes a solvent dilution technique that may be used on non-aqueous nonvolatile and semivolatile organic samples prior to cleanup and/or analysis.

1.3 The 5000 Methods are procedures for preparing samples containing volatile organic compounds for quantitative analysis.

1.4 Refer to the specific method of interest for further details.

2.0 SUMMARY OF METHOD

2.1 3500 Methods: A sample of a known volume or weight is solvent extracted. The resultant extract is dried and then concentrated in a Kuderna-Danish apparatus. Other concentration devices or techniques may be used in place of the Kuderna-Danish concentrator if the quality control requirements of the determinative methods are met (Method 8000, Section 8.0).

2.2 5000 Methods: Refer to the specific method of interest.

3.0 INTERFERENCES

3.1 Samples requiring analysis for volatile organic compounds, can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

3.2 Solvents, reagent, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Refer to Chapter One for specific guidance on quality control procedures.

3.3 Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary. Refer to Method 3600 for guidance on cleanup procedures.

3.4 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

3.5 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

4.0 APPARATUS AND MATERIALS

4.1 Refer to the specific method of interest for a description of the apparatus and materials needed.

5.0 REAGENTS

5.1 Refer to the specific method of interest for a description of the solvents needed.

5.2 Stock standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions.

5.2.1 **Purgeable stock standards:** Prepare stock standards in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood.

5.2.1.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.2.1.2 Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.2.1.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.1.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.2.1.5 All standards must be replaced after 1 month, or sooner if comparison with check standards indicates a problem.

5.2.2 **Semivolatile stock standards:** Base/neutral and acid stock standards are prepared in methanol. Organochlorine pesticide standards are prepared in acetone.

5.2.2.1 Stock standard solutions should be stored in Teflon-sealed containers at 4°C. The solutions should be checked frequently for stability. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicate a problem.

5.3 Surrogate standards: A surrogate standard (i.e., a chemically inert compound not expected to occur in an environmental sample) should be added to each sample, blank, and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits. Recommended surrogates for different analyte groups follow; however, these compounds, or others that better correspond to the analyte group, may be used for other analyte groups as well. Normally three or more standards are added for each analyte group.

5.3.1 **Base/neutral and acid surrogate spiking solutions:** The following are recommended surrogate standards.

Base/neutral	Acid
2-Fluorobiphenyl	2-Fluorophenol
Nitrobenzene-d_5	2,4,6-Tribromophenol
Terphenyl-d_{14}	Phenol-d_6

5.3.1.1 Prepare a surrogate standard spiking solution in methanol that contains the base/neutral compounds at a concentration of 100 ug/mL, and the acid compounds at 200 ug/mL for water and sediment/soil samples (low- and medium-level). For waste samples, the concentration should be 500 ug/mL for base/neutrals and 1000 ug/mL for acids.

5.3.2 **Organochlorine pesticide surrogate spiking solution:** The following are recommended surrogate standards for organochlorine pesticides.

Organochlorine pesticides

Dibutylchlorendate (DBC)
2,4,5,6-Tetrachloro-meta-xylene (TCMX)

5.3.2.1 Prepare a surrogate standard spiking solution at a concentration of 1 ug/mL in acetone for water and sediment/soil samples. For waste samples, the concentration should be 5 ug/mL.

5.3.3 **Purgeable surrogate spiking solution:** The following are recommended surrogate standards for volatile organics.

Purgeable organics

p-Bromofluorobenzene
1,2-Dichloroethane-d_4
Toluene-d_8

5.3.3.1 Prepare a surrogate spiking solution (as described in Paragraph 5.2.1 or through secondary dilution of the stock standard) in methanol containing the surrogate standards at a concentration of 25 ug/mL.

5.4 Matrix spike standards: Select five or more analytes from each analyte group for use in a spiking solution. The following are recommended matrix spike standard mixtures for a few analyte groups. These compounds, or others that better correspond to the analyte group, may be used for other analyte groups as well.

5.4.1 **Base/neutral and acid matrix spiking solution:** Prepare a spiking solution in methanol that contains each of the following base/neutral compounds at 100 ug/mL and the acid compounds at 200 ug/mL for water and sediment/soil samples. The concentration of these compounds should be five times higher for waste samples.

Base/neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

5.4.2 **Organochlorine pesticide matrix spiking solution:** Prepare a spiking solution in acetone or methanol that contains the following pesticides in the concentrations specified for water and sediment/soil. The concentration should be five times higher for waste samples.

Pesticide	Concentration (ug/mL)
Lindane	0.2
Heptachlor	0.2
Aldrin	0.2
Dieldrin	0.5
Endrin	0.5
4,4'-DDT	0.5

5.4.3 **Purgeable matrix spiking solution:** Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25 ug/mL.

Purgeable organics

1,1-Dichloroethene
Trichloroethene
Chlorobenzene
Toluene
Benzene

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to the Organic Analyte Chapter, Section 4.1.

7.0 PROCEDURE

7.1 Semivolatile organic sample extraction: Water, soil/sediment, sludge, and waste samples requiring analysis for base/neutral and acid extractables and/or organochlorine pesticides must undergo solvent extraction prior to analysis. This manual contains four methods that may be used for this purpose: Method 3510; Method 3520; Method 3540; and Method 3550. The method that should be used on a particular sample, is highly dependent upon the physical characteristics of that sample. Therefore, review these four methods prior to choosing one in particular. Appropriate surrogate standards and, if necessary, matrix spiking solutions are added to the sample prior to extraction for all four methods.

7.1.1 **Method 3510:** Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is solvent extracted using a separatory funnel. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. Method 3520 should be used if an emulsion forms between the solvent-sample phases, which can not be broken up by mechanical techniques.

7.1.2 **Method 3520:** Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is extracted with an organic solvent in a continuous liquid-liquid extractor. The solvent must have a density greater than that of the sample. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis. The limitations of Method 3510 concerning solvent-sample phase separation do not interfere with this procedure.

7.1.3 **Method 3540:** This is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. A solid sample is mixed with anhydrous sodium sulfate, placed into an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis.

7.1.4 **Method 3550:** This method is applicable to the extraction of nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes using the technique of sonication. Two procedures are detailed depending upon the expected concentration of organics in the sample; a low concentration and a high concentration method. In both, a known weight of sample is mixed with anhydrous sodium sulfate and solvent extracted using sonication. The extract is dried, concentrated and, if necessary, exchanged into a solvent compatible with further analysis.

7.1.5 **Method 3580:** This method describes the technique of solvent dilution of non-aqueous waste samples. It is designed for wastes that may contain organic chemicals at a level greater than 20,000 mg/kg and that are soluble in the dilution solvent.

7.2 Volatile organic sample preparation: There are three methods for volatile sample preparation: Method 5030; Method 5040; and direct injection. Method 5030 is the most widely applicable procedure for analysis of volatile organics, while the direct injection technique may have limited applicability to aqueous matrices.

7.2.1 **Method 5030:** This method describes the technique of purge-and-trap for the introduction of purgeable organics into a gas chromatograph. This procedure is applicable for use with aqueous samples directly and to solids, wastes, soils/sediments, and water-miscible liquids following appropriate preparation. An inert gas is bubbled through the sample, which will efficiently transfer the purgeable organics from the aqueous phase to the vapor phase. The vapor phase is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. Prior to application of the purge-and-trap procedure, all samples (including blanks, spikes, and duplicates) should be spiked with surrogate standards and, if required, with matrix spiking compounds.

7.2.2 **Method 5040:** This method is applicable to the investigation of sorbent cartridges from volatile organic sampling train (VOST).

7.3 Sample analysis: Following preparation of a sample by one of the methods described above, the sample is ready for further analysis. For samples requiring volatile organic analysis, application of one of the three methods described above is followed directly by gas chromatographic analysis (Methods 8010, 8015, 8020, or 8030). Samples prepared for semivolatile analysis may, if necessary, undergo cleanup (See Method 3600) prior to application of a specific determinative method.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific guidance on quality control procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of a reagent water blank that all glassware and reagents are interference free. Each time a set of samples are processed, a method blank(s) should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Surrogate standards should be added to all samples when specified in the appropriate determinative method in Chapter Four, Section 4.3.

8.4 A reagent blank, a matrix spike, and a duplicate or matrix spike duplicate must be performed for each analytical batch (up to a maximum of 20 samples) analyzed.

8.5 For GC or GC/MS analysis, the analytical system performance must be verified by analyzing quality control (QC) check samples. Method 8000, Section 8.0 discusses in detail the process of verification; however, preparation of the QC check sample concentrate is dependent upon the method being evaluated.

8.5.1 **Volatile organic QC check samples:** QC check sample concentrates containing each analyte of interest are spiked into reagent water (defined as the QC check sample) and analyzed by purge-and-trap (Method 5030). The concentration of each analyte in the QC check sample is 20 ug/L. The evaluation of system performance is discussed in detail in Method 8000, beginning with Paragraph 8.6.

8.5.2 **Semivolatile organic QC check samples:** To evaluate the performance of the analytical method, the QC check samples must be handled in exactly the same manner as actual samples. Therefore, 1.0 mL of the QC check sample concentrate is spiked into each of four 1-L aliquots of reagent water (now called the QC check sample), extracted, and then analyzed by GC. The variety of semivolatile analytes which may be analyzed by GC is such that the concentration of the QC check sample concentrate is different for the different analytical techniques presented in the manual. Method 8000 discusses in detail the procedure of verifying the detection system once the QC check sample has been prepared. The concentrations of the QC check sample concentrate for the various methods are as follows:

8.5.2.1 Method 8040 - Phenols: The QC check sample concentrate should contain each analyte at a concentration of 100 ug/mL in 2-propanol.

8.5.2.2 Method 8060 - Phthalate esters: The QC check sample concentrate should contain the following analytes at the following concentrations in acetone: butyl benzyl phthalate, 10 ug/mL; bis(2-ethylhexyl)phthalate, 50 ug/mL; di-n-octylphthalate, 50 ug/mL; and any other phthalate at 25 ug/mL.

8.5.2.3 Method 8080 - Organochlorine pesticides and PCBs: The QC check sample concentrate should contain each single-component analyte at the following concentrations in acetone: 4,4'-DDD, 10 ug/mL; 4,4'-DDT, 10 ug/mL; endosulfan II, 10 ug/mL; endosulfan sulfate, 10 ug/mL; and any other single-component pesticide at 2 ug/mL. If the method is only to be used to analyze PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multicomponent parameter at a concentration of 50 ug/mL in acetone.

8.5.2.4 Method 8090 - Nitroaromatics and Cyclic Ketones: The QC check sample concentrate should contain each analyte at the following concentrations in acetone: each dinitrotoluene at 20 ug/mL; and isophorone and nitrobenzene at 100 ug/mL.

8.5.2.5 Method 8100 - Polynuclear aromatic hydrocarbons: The QC check sample concentrate should contain each analyte at the following concentrations in acetonitrile: naphthalene, 100 ug/mL; acenaphthylene, 100 ug/mL; acenaphthene, 100 ug/mL; fluorene, 100 ug/mL; phenanthrene, 100 ug/mL; anthracene, 100 ug/mL; benzo(k)fluoranthene 5 ug/mL; and any other PAH at 10 ug/mL.

8.5.2.6 Method 8120 - Chlorinated hydrocarbons: The QC check sample concentrate should contain each analyte at the following concentrations in acetone: hexachloro-substituted hydrocarbons, 10 ug/mL; and any other chlorinated hydrocarbon, 100 ug/mL.

9.0 METHOD PERFORMANCE

9.1 The recovery of surrogate standards is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds indicates the presence or absence of unusual matrix effects.

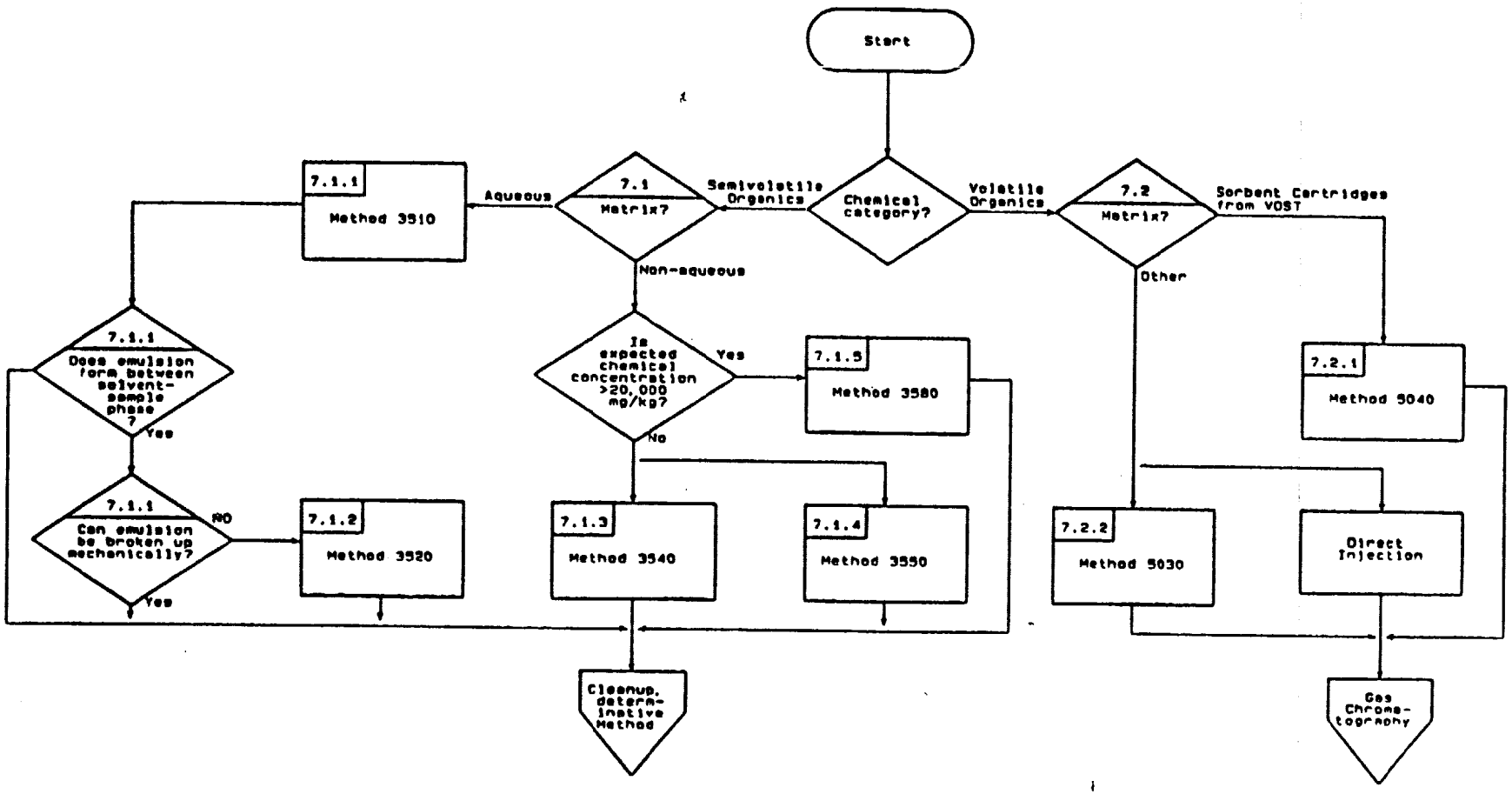
9.2 The performance of this method will be dictated by the overall performance of the sample preparation in combination with the analytical determinative method.

10.0 REFERENCES

10.1 None required.

METHOD 3500

ORGANIC EXTRACTION AND SAMPLE PREPARATION



EPA SW846 3rd Ed.

METHOD 3540

SOXHLET EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 Method 3540 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor: 40-mm I.D., with 500-mL round-bottom flask.

4.2 Drying column: 20-mm I.D. Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.3 Kuderna-Danish (K-D) apparatus:

4.3.1 Concentrator tube: 10-mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.3.2 Evaporation flask: 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.

4.3.3 Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.3.4 Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.4 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.5 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.6 Vials: Glass, 2-mL capacity, with Teflon-lined screw cap.

4.7 Glass or paper thimble or glass wool: Contaminant free.

4.8 Heating mantle: Rheostat controlled.

4.9 Syringe: 5-mL.

4.10 Apparatus for determining percent moisture:

4.10.1 Oven: Drying.

4.10.2 Desiccator.

4.10.3 Crucibles: Porcelain.

4.11 Apparatus for grinding: If the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, it should be processed into a homogeneous sample that meets these requirements. Fisher Mortar Model 155 Grinder, Fisher Scientific Co., Catalogue Number 8-323, or an equivalent brand and model, is recommended for sample processing. This grinder should handle most solid samples, except gummy, fibrous, or oily materials.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.2 Sodium sulfate: (ACS) Granular anhydrous (purified by washing with methylene chloride followed by heating at 400°C for 4 hr in a shallow tray).

5.3 Extraction solvents:

5.3.1 Soil/sediment and aqueous sludge samples shall be extracted using either of the following solvent systems.

5.3.1.1 Toluene/Methanol: 10:1 (v/v), pesticide quality or equivalent.

5.3.1.2 Acetone/Hexane: 1:1 (v/v), pesticide quality or equivalent.

5.3.2 Other samples shall be extracted using the following:

5.3.2.1 Methylene chloride: pesticide quality or equivalent.

5.4 Exchange solvents: Hexane, 2-propanol, cyclohexane, acetonitrile (pesticide quality or equivalent).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Sample handling:

7.1.1 **Sediment/soil samples:** Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.1.2 **Waste samples:** Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two before extraction. This procedure is for solids only.

7.1.3 **Dry waste samples amenable to grinding:** Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

7.2 Determination of percent moisture: In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \%\ \text{moisture}$$

7.3 Blend 10 g of the solid sample with 10 g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0 mL of the surrogate standard spiking solution onto the sample (See Method 3500 for details on the surrogate standard and matrix spiking solutions.) For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/uL of each base/neutral analyte and 200 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.4 Place 300 mL of the extraction solvent (Section 5.3) into a 500-mL round-bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hr.

7.5 Allow the extract to cool after the extraction is complete.

7.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

7.7 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 mL of extraction solvent to complete the quantitative transfer.

7.8 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min.

7.9 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip, and re-attach the Snyder column. Concentrate the extract as described in Paragraph 7.6, raising the temperature of the water bath, if necessary, to maintain proper distillation.

7.10 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Paragraph 7.9 or adjusted to 10.0 mL with the solvent last used.

TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040[a]	as received	2-propanol	hexane	1.0	1.0, 10.0[b]
8060	as received	hexane	hexane	2.0	10.0
8080	as received	hexane	hexane	10.0	10.0
8090	as received	hexane	hexane	2.0	1.0
8100	as received	none	cyclohexane	2.0	1.0
8120	as received	hexane	hexane	2.0	1.0
8140	as received	hexane	hexane	10.0	10.0
8250[a,c]	as received	none	-	-	1.0
8270[a,c]	as received	none	-	-	1.0
8310	as received	acetonitrile	-	-	1.0

[a]To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

[b]Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

[c]The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

7.11 If further concentration is indicated in Table 1, add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Prewet the column by adding 0.5 mL of methylene chloride or exchange solvent to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 0.2 mL of solvent. Adjust the final volume to 1.0-2.0 mL, as indicated in Table 1, with solvent.

7.12 The extracts obtained may now be analyzed for analyte content using a variety of organic techniques (see Section 4.3 of this chapter). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days, it should be transferred to a Teflon-sealed screw-cap vial and labeled appropriately.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

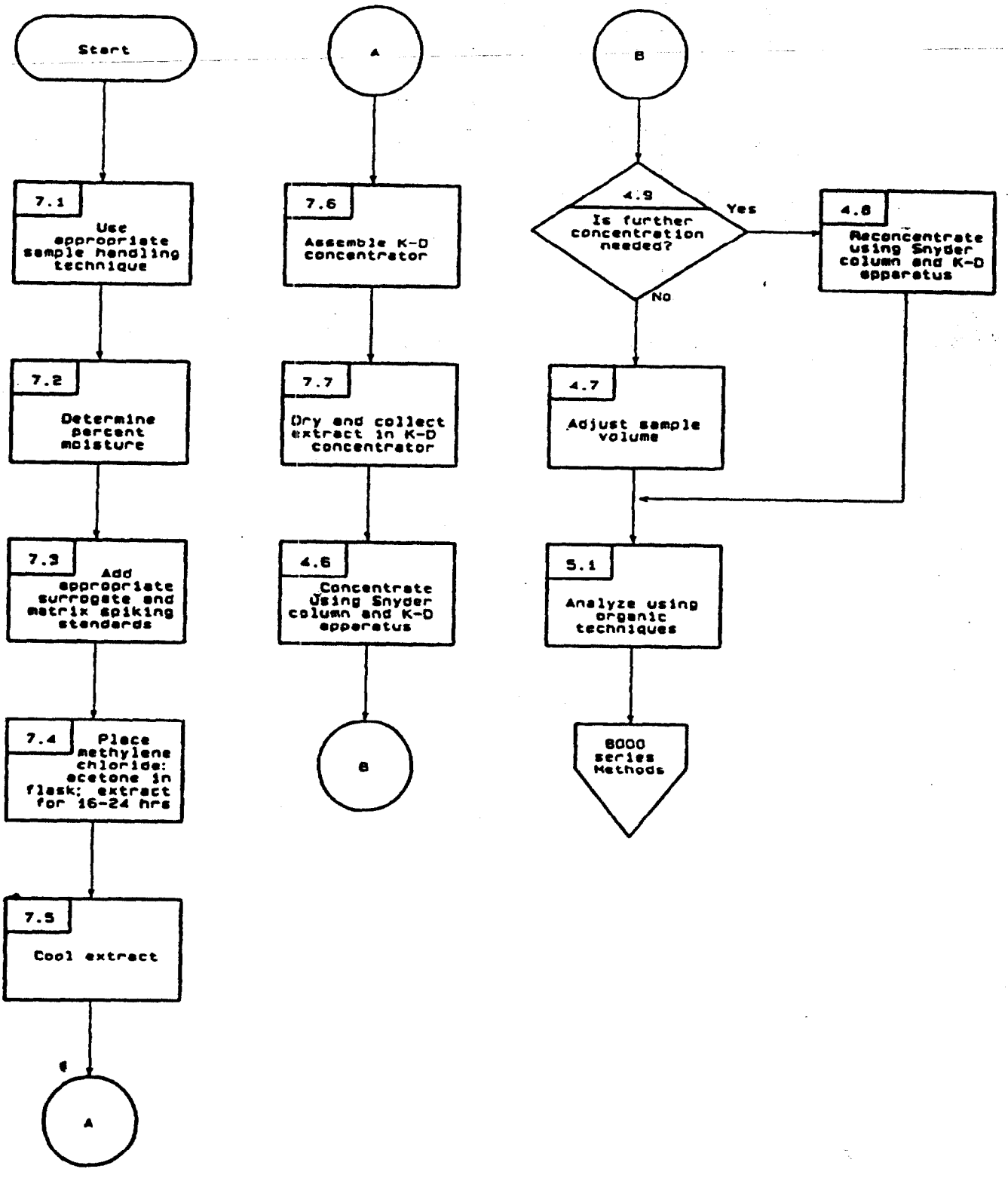
9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

METHOD 3540

SOXHLET EXTRACTION



Revision 0
Date September 1986

received from CK
3/27/87

EPA CLP
PAPERWORK REQUIREMENTS

II. Paperwork Requirements

aperwork requirements include Chain of Custody, Traffic Reports, SAS Packing List and CRL Sample Data Report. All of these forms, except for the CRL Sample Data Report are composed of 3-4 different colored carbon copies. The destination of each copy is specified on the bottom of each form. The Central Regional Laboratory Sample Data Report is a single copy form which is to be submitted to the CRL.

Paperwork required by the contract laboratory is sent in a plastic bag taped to the inside top of the cooler with the samples.

The SMO copies of the Traffic Reports and Packing Lists(for SASs) should be sent within one week of sampling.

It is required that the CRL (regional) copy of the Traffic Reports, Chain of Custody, SAS Packing List, and Central Regional Laboratory Sample Data Report be received at the CRL within 5 days of sample shipment. Late paperwork can delay data review and will affect contractor fee award reviews. It is the samplers responsibility to make sure that paperwork is filled out accurately and submitted within the timeframes mentioned above.

Immediately upon shipment of samples, samplers must call SMO with the following information: Case or SAS number; name of laboratory; date of shipment; carrier, airbill(shipment) numbers, and type of service; number and matrices of samples shipped; information on changes, delays, continuations, etc.,pertainent to the case.
SMO must be notified by 3:00 PM on Friday for samples intended for Saturday delivery/pickup.

SAMPLE TAG

1. Enter the first six digits of the CRL sample identification.
2. Enter the last three digits of the CRL identification code.
3. Enter date of sampling.
4. Enter time of sampling (military time only).
5. Specify "grab" or "composite" sample with an "X".
6. Insert sample identification code.
7. Obtain signature of sample team leader.
8. Indicate presence of preservative with an "X".
9. Specify parameters for analysis with an "X".

10a. Indicate traffic report type and serial number.

10b. Indicate case number.

11. Leave BLANK (for laboratory use only).
12. Enter any desired analyses not listed on menu provided (e.g., PCB's, ammonia, sulfide, etc.) and mark box with an "X".

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

EPA

Project Code (1)	Station No. (2)	Month/Day/Year (3)	Time (4)	Designate (5): Comp. / Grab

Station Location (6)

Samplers (Signatures) (7)

Tag No. 5- 48246

Lab Sample No. (11)

Remarks: (10a) (10b)

ANALYSES	
BOD Anions	
Solids (TSS) (TDS) (SS)	
COD, TOC, Nutrients	
Phenolics	
Mercury	
Metals	
Cyanide	
Oil and Grease	
Organics GC/MS	
Priority Pollutants	
Volatile Organics	
Pesticides	
Microbiology	
Bacteriology	

(9)

Preservative: Yes ☐ No ☐ (8)

(12)

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION V
OFFICIAL SEAL
№ 16004

Project Code	Station No.	Month/Day/Year	Time	Designate: Comp.	Designate: Grab

Station Location

Samplers (Signatures)

Preservative:
Yes ☐ No ☐

ANALYSES

BOD Anions Solids (TSS) (TDS) (SS)	
COD, TOC, Nutrients	
Phenolics	
Mercury	
Metals	
Cyanide	
Oil and Grease	
Organics GC/MS	
Priority Pollutants	
Volatile Organics	
Pesticides	
Mutagenicity	
Bacteriology	

Remarks:

Tag No. 5- 54505

Lab Sample No.

CHAIN-OF-CUSTODY FORM

1. Enter first six digits of the CRL sample identification code.
2. Enter site name.
3. Obtain full signature of sample team leader.
4. Enter last three digits of the CRL sample identification code (eg. S01, DC2, R01, etc.).
5. List sampling dates for all samples.
6. List sampling times for all samples.
7. Indicate "grab" or "composite" sample with an "X."
8. List sample numbers.
9. Enter number of containers per sample and container volume (e.g., 2-40 ml).
10. List analyses individually.
11. Construct column heading for traffic report number and list serial numbers for corresponding sample identification codes.
12. Construct column heading for "tag number" and list tag numbers for each sample container.
13. Obtain signature of sample team leader and carry out chain of custody procedures.
14. State carrier service and air bill number, lab service, and custody seal numbers.
15. Write in the words "CASE #:" and enter the case number.

*NOTE: One Chain-Of-Custody should be filled out per shipping container. Also, the COC seal numbers should be recorded on Chain-Of-Custody form.

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

CHAIN OF CUSTODY RECORD

PROJ. NO. (1) | PROJECT NAME (2)

SAMPLERS: (Signature) (3)

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	REMARKS (10) (11) (12) (15)

(4) (5) (6) (7) (8) (9)

Relinquished by: (Signature) (13)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks (14)	

Distribution: White — Accompanies Shipment; Pink — Coordinator Field Files; Yellow — Laboratory File

FIGURE 15
Page 1 of 2

INORGANIC TRAFFIC REPORT

1. Insert assigned laboratory case number.
2a. Insert sample number.
2b. Insert CRL log number.
3. Insert EPA region number (e.g. V)
4. Insert sample team leader's name.
5. Insert sample team leader's office telephone number (do not use field office telephone number).
6. Insert date sample was taken and time sampling began and ended.
7. Specify sample description with an "X".
8. Insert corresponding organic traffic report number for sample (if any).
9. Specify the sample concentration with an "X".
10. Indicate the sample matrix with an "X".
11. Indicate "Federal Express" (or other approved carrier).
12. Indicate date of shipment.
13. Indicate air bill number.
14. Check required analyses: Metals, Mercury, and Cyanide are RAS analytes.
15. Insert the phrase "QC lot number" and indicate the quality control lot number(s) of the container(s).
16. Insert laboratory name and address.
17 Indicate name of laboratory contact.
18. Leave blank.

Sample Number
MS 1535

ENVIRONMENTAL PROTECTION AGENCY
INORGANICS TRAFFIC REPORT

(1) Case Number: (1)
Sample Site Name/Code:
(2a)
(2b)

(2) SAMPLE CONCENTRATION
(Check One) (9)
___ Low Concentration
___ Medium Concentration

(3) SAMPLE MATRIX
(Check One)
___ Water
___ Soil/Sediment (10)

(4) Ship To: (16)
Attn: (17)
Transfer Ship To: (18)

(5) Sampling Office: (3)
Sampling Personnel:
(Name) (4)
(Phone) (5)
Sampling Date:
(Begin) ___ (End) ___ (6)

(6) Shipping Information:
Name Of Carrier: (11)
Date Shipped: (12)
Airbill Number: (13)

(7) Sample Description:
(Check One)
___ Surface Water
___ Ground Water
___ Leachate
___ Mixed Media
___ Solids
___ Other ___ (specify) (7)

(8) Mark Volume Level
On Sample Bottle
Check Analysis required
___ Task 1 & 2
___ Task 3 Ammonia
Sulfide
Cyanide (14)

(15)

MATCHES ORGANIC SAMPLE NO. ___ (8)

SMO COPY

MS 1535 - Task 1 & 2
MS 1535 - Task 1 & 2
MS 1535 - Task 3
MS 1535 - Task 3
MS 1535 - Task 3
MS 1535 - Task 3
MS 1535 - Task 3

ORGANIC TRAFFIC REPORT

1. Insert assigned laboratory case number.
2a. Insert sample number.
2b. Insert CRL log number.
3. Insert EPA region number (e.g. V)
4. Insert sample team leader's name.
5. Insert sample team leader's office telephone number (do not use field office telephone number).
6. Insert date sample was taken and time sampling began and ended.
7. Indicate "Federal Express" (or other approved carrier).
8. Indicate date of shipment.
9. Indicate air bill number.
10. Specify sample description with an "X".
11. Insert the phrase "QC lot number" and indicate the quality control lot number(s) of the container(s).
12. Insert the phrase "Matches ITR number" and indicate the corresponding inorganics traffic report number for the sample (if any).
13. Specify the sample concentration with an "X".
14. Indicate the sample matrix with an "X".
15. Insert the number of containers.
16. Insert an estimated sample volume in appropriate box.
17. Insert laboratory name and address.
18. Indicate name of laboratory contact.
19. Leave blank.
20. Leave blank (or make reference notes for future use).

Sample Number: S 4601

ORGANIC TRACE REPORT

① Case Number: (1)

Sample Site Name/Code: (2a) (2b)

② SAMPLE CONCENTRATION (Check One) (13)

___ Low Concentration
___ Medium Concentration

③ SAMPLE MATRIX (Check One) (14)

___ Water
___ Soil/Sediment

④ Ship To: (17)

Attn: (18)

Transfer Ship To: (19)

⑤ Regional Office: (3)

Sampling Personnel: (4)

(Name) (5)

(Phone)

Sampling Date: (6)

(Begin) (End)

⑥ For each sample collected specify nu of containers used and mark volume l on each bottle.

	Number of Containers	Approx Total V
Water (Extractable)		
Water (VOA)		
Soil/Sediment	(15)	
Water (Ext/VOA)		(16)
Other		

⑦ Shipping Information (7)

Name of Carrier: (8)

Date Shipped: (9)

Airbill Number:

S 4601	Water (Extractable)
S 4601	Water (Extractable)
S 4601	Water (Extractable)
S 4601	Water (Extractable)
S 4601	Water (VOA)
S 4601	Water (VOA)
S 4601	Soil/Sediment (Ext & VOA)
S 4601	Soil/Sediment (Ext & VOA)
S 4601	Water (Ext & VOA)
S 4601	Water (Ext & VOA)

⑧ Sample Description (10)

___ Surface Water ___ Mixed Media
___ Ground Water ___ Solids
___ Leachate ___ Other (specify) _____

⑨ San (20)

⑩ Special Handling Instructions:
(e.g., safety precautions, hazardous nature)

(11)

High Hazard Traffic Report

1. Insert assigned laboratory case number

2a. Insert CRL sample identification code

2b. Insert sample number.

3. Insert EPA region number (e.g. V)

4. Insert sample team leader's name

5. Insert sample team leader's office telephone number (do not use field office telephone number)

6. Insert date sample was taken and time sampling began and ended.

7. Insert "Federal Express" (or other approved carrier)

8. Indicate date of shipment

9. Indicate air bill number on which shipment was made

10. Insert the phrase "QC lot number: " and indicate the quality control lot number(s) of the container(s)

11. Indicate sample description with an "X"

12. List known or suspected hazards

13. Indicate volume of sample

14. Specify desired organic parameters to be analyzed for

15. Specify desired inorganic parameters to be analyzed for (strong acid anions include Cl^-, $SO_4^=$, NO_3^-, F^-)

16. Insert lab name and address

17. Insert name of laboratory contact

18. Leave BLANK (or make reference notes for future use)

HIGH HAZARD TRACE RECORD

Sample Number S 5429

FIELD SAMPLE RECORD

① Case Number: 1

Sample Site Name/Code:

(2a)

(2b)

② Field Sample Description:

__ Drum
__ Aqueous Liquid
__ Sludge
__ Solid
__ Oil
__ Other

11

③ Ship To: 16

Attn: 17

④ Sampling Office: 3

Sampling Personnel: 4

(name) 5

(phone)

Sampling Date: 6

(begin) (end)

⑤ Known or Suspected Hazards:

12

⑥ Sample Location: 18

⑦ Preparations Requested: (check below)

Sample Volume: 13

__ Organics
__ Volatile Organics
__ Base/Neutral Acid
TCDD
__ Pesticides PCB

14

__ Inorganics
__ Total Metals
__ Total Mercury
__ Strong Acid Anions

15

S 5429

S 5429

S 5429

S 5429

S 5429

⑧ Shipping Information: 7

(name of carrier)

8

(date shipped)

9

(airbill number)

⑨ Special Handling Instructions:

10

SMO Copy

SAS PACKING LIST

1. Insert assigned SAS case number.
2. Insert EPA region number (e.g., V).
3. Insert sample team leader's name.
4. Insert sample team leader's office telephone number (do not use field office telephone number).
5. Insert date sample was taken.
6. Indicate date of shipment.
7. Insert site name, or site code.
8. Insert laboratory name and address.
9. Indicate name of laboratory contact.
10. List SAS sample numbers, which should include the SAS number.
11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag number 5-48246).
12. Leave BLANK - for laboratory use only.

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

(1) SAS Number:

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office: (2)	Sampling Date(s): (5)	Ship To: (8)	For Lab Use Only
Sampling Contact: (3) (name)	Date Shipped: (6)		Date Samples Rec'd:
(4) (phone)	Site Name/Code: (7)	Attn: (9)	Received By:

	Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.	(10)	(11)	(12)
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT

1. Insert assigned laboratory case number.
2. Insert site name.
3. Insert laboratory names, indicating which lab will receive the organic samples and which lab will receive the inorganic samples.
4. Insert date of shipment.
5. Insert DU code (either Y905 for site inspection or remedial, or Y306 for enforcement, including PRP sites).
6. Insert name of RPM (the RPM will know what the site DU code is).
7. Insert page number and total number of pages.
8. Insert CRL log number, which consists of the fiscal year, EPA assigned contractor code, sampler code, round of sampling, sample type designation and sample number.

   eg. 8 7 S W 0 1 S 0 1
   (a) (b) (c) (d) (e) (f)

a.	b.	c.	d.	e.	f.
FY	contractor code	this could be the 1st letter of surname of the sampler (sampler code)	round	sample type, could be: S-sample D-duplicate R-field blank	sample number ie. 01,02,03,et

   87SW01S01 would be a sample.
   87SW01D01 would be a field duplicate of sample 87SW01S01.
   87SW01R01 would be a field blank.

9. Insert organic traffic report number.
10. Insert inorganic traffic report number.
11. Indicate the analyses required (eg. acid-base neutral cpds, volatile organic analysis, etc.) for each sample in the appropriate section (for waters or soils) with an "X".

Note: All samples should have a unique CRL log number. If the same location will be sampled at a site two weeks in a row, the sample number for the first week could be 87SW01S01, and for the second week 87SW02S01.
If a sample is collected for total and dissolved metals analyses, a separate ITR should be filled out for each bottle(the filtered and unfiltered). Each one of these samples would then be assigned a unique CRL log number. The dissolved metals analyses can be requested by writing "Dissolved Metals" in one of the free columns and checking this analysis off for the samples that have been filtered. Metals analysis for the unfiltered samples would have an "X" in the metals column (already printed on the form).

1/87

This form must be filled out for all SF samples which will go to contract labs.

ENVIRONMENTAL PROTECTION AGENCY
Central Region-1 Laboratory Analysis Request Form

DIVISION/BRANCH - (1)
DO/ACTIVITY - (3)

SAMPLING DATE - (2)
DATASET NO. -

ARRIVAL DATE -
STUDY - (4)

DUE DATE -
PRIORITY - (5)

CRL LOG NUMBER	SAMPLE DESCRIPTION	WATER SULFATE 375.2 D mg/L	WATER CHLORIDE 325.2 D mg/L	WATER TURBIDITY 180.1 JTU	WATER RESIDUE-FILTERABLE 160.1 mg/L	WATER
(6)	(7)	(8)				

OBSERVATIONS and DEVIATIONS

The analytical systems performed as outlined in the appropriate method summary statement except as listed under "Observations and Deviations".

ENVIRONMENTAL PROTECTION AGENCY
Central Regional Laboratory Analysis Request Form

1. Insert sampler name, i.e., CDM, Fit, E&E, Weston, or RPM name.
2. Insert sampling date.
3. Insert DU number (Y905 or Y306).
4. Insert site name.
5. Insert priority code (if any).*
6. Insert CRL log numbers.
7. Insert well number and/or sample tag number.
8. Indicate analyses required by placing an "X" in the appropriate column for each sample.

Note: The analytes that are routinely analyzed for at the Central Regional Laboratory are listed in Appendix [illegible] which was included in the Transmittal of Latest CRL Method Capabilities and Requirements, sent from the CRL Director on September 10, 1986 to All Superfund Sample Requestors and QAPP Preparers.

* Normal turnaround time for the CRL is 21 days from receipt of samples. Priority 1 analysis requires a 5 day turnaround, and must be requested via a memo from Bill Constantelos (WMD) to Bill Sanders (ESD) in advance of sampling.
Requests for shorter than the 21 day (and greater than 5 days) turnaround must be addressed to Curtis Ross (CRL).

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT
ORGANICS/INORGANICS

THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

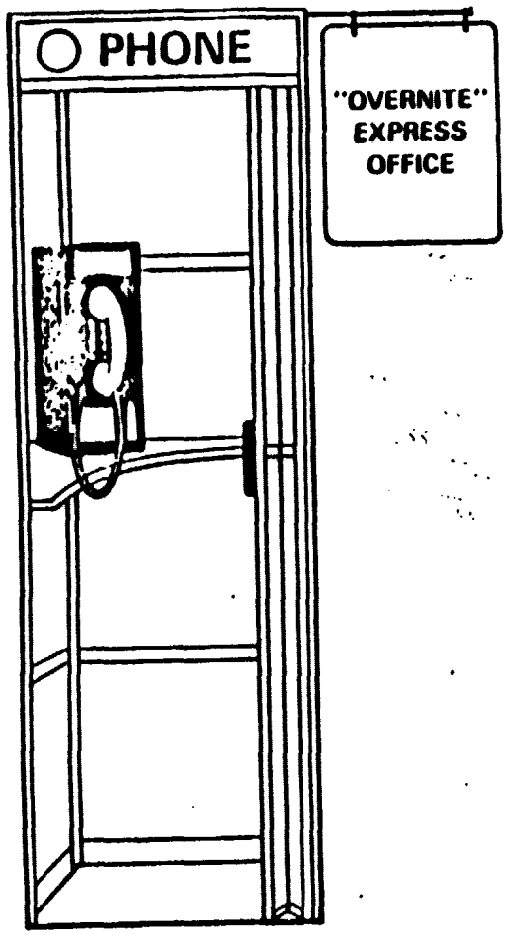
SE NUMBER (1) _____ SITE NAME (2) _____ LABORATORY (3) _____ DATE SHIPPED (4) _____

PERFUND CU NUMBER (5) _____ EPA RPM (S.M.S.)/(CES) (6) _____ PAGE (7) _____ OF _____

CTIVITY NUMBER _____

CRL LOG NUMBER	ORGANIC TRAFFIC REPORT NUMBER	INORGANIC TRAFFIC REPORT NUMBER	(11) WATER OR LIQUIDS												SEDIMENTS										
			ACID BASE NEUTRAL ORGANIC SCAN UG/L TOX17574	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN UG/L TOX17584	WATER POLYCHLORINATED BIPHENYLS UG/L PES 17144	WATER CHLORINATED PESTICIDES UG/L PES17135	TOTAL METALS IN WATER UG/L MET111	WATER CYANIDE UG/L MIN74919	NITRATE-NITRITE MG/L MIN7294	AMMONIA MG/L MIN7294	RESIDUE, FILTERABLE TSS MG/L MIN7382	RESIDUE, NON-FILT TSS MG/L MIN7372			ACID BASE NEUTRAL CPOS ORGANIC SCAN MG/KG TOX215722	VOLATILE ORGANIC ANALYSIS SCAN MG/KG TOX219622	SEDIMENTS POLYCHLORINATED BIPHENYLS MG/KG PES211422	SEDIMENT CHLORINATED PESTICIDES MG/KG 211322	TOTAL METALS MG/KG MET213	CYANIDE MG/KG MIN44930	NITRATE/NITRITE MG/KG	AMMONIA MG/KG MIN42925				
(8)	(9)	(10)																							

SAMPLE SHIPMENT COORDINATION CHECKLIST



IMMEDIATELY UPON SHIPMENT OF SAMPLES, SAMPLERS CALL SMO WITH THE FOLLOWING INFORMATION:

- CASE OR SAS NUMBER
- NAME OF LABORATORY
- DATE OF SHIPMENT
- CARRIER, AIRBILL (SHIPMENT) NUMBERS AND TYPE OF SERVICE
- NUMBER AND MATRICES OF SAMPLES SHIPPED
- INFORMATION ON CHANGES, DELAYS, CONTINUATIONS, ETC., PERTINENT TO THE CASE
- SMO MUST BE NOTIFIED BY 3:00 PM ON FRIDAY FOR SAMPLES INTENDED FOR SATURDAY DELIVERY/PICKUP

SAS REQUESTS (Special Analytical Services Request)

These forms must be completed and included in the QAPP for a site when requesting non-routine analytes, routine analytes with fast turnaround by the contract laboratory, or routine analytes in sample matrices other than soil or water.
Guidance in filling out these forms will be provided by the CPMS/CRL on request.
It must be noted that SASs are separate contracts from the IFBs for routine work and therefore must be explicit and concise in definition of work, special technical instructions, quality control and documentation requirements.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: _____

B. RSCC Representative: _____

C. Telephone Number: _____

D. Date of Request: _____

E. Site Name: _____

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability to your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: _____

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: _____

6. Number of days analysis and data required after laboratory receipt of samples:

_____

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

_____

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): _____

_____

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

_____

10. Other (use additional sheets or attach supplementary information, as needed):

_____

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions

CLP Sample Collection Requirements

ANALYSIS	REQUIRED VOLUME	# OF CONTAINERS	CONTAINER TYPE	PRESERVATIVE	COMMENTS
WATER SAMPLES					
Metals-low level (includes mercury)	1 Liter	1	1 liter polyethylene bottle	HNO_3 to a pH<2	
*Metals-medium level (Hg included)	1 Liter	1	16 oz. wide mouth glass jar	HNO_3 to a pH<2	
Cyanide-low level	1 Liter	1	1 liter polyethylene bottle	Cool, 4°C NaOH to a pH>12 **1.2 g ascorbic acid	
*Cyanide-medium level	1 Liter	1	1 16 oz. wide mouth glass jar	Cool, 4°C NaOH to a pH>12 **1.2 g ascorbic acid	
Extractables-low level	1 gallon	2	80 oz. amber glass bottles	Cool, 4°C	
		or 4	1 liter amber glass bottles		
*Volatile-low or medium level	80 ml	2	40 ml glass vials	Sample vial must be full and free of headspace. Cool, 4°C	
SOIL SAMPLES					
*Metals and cyanide, low or medium level	6 oz.	1	8 oz. wide mouth glass jar		
		or 2	4 oz. wide mouth glass jars		
*Extractables-low or medium level	6 oz.	1	8 oz. wide mouth glass jar	Cool, 4°C	
		or 2	4 oz. wide mouth glass jars		
*Volatiles-low or medium level	240 ml	2	120 ml wide mouth glass vials	Cool, 4°C; vial must be full and free of headspace.	
HIGH HAZARD SAMPLES					
*Liquid Samples-organic and inorganic	6 oz.	1	8 oz. wide mouth glass jar		
*Solid Samples-organic and inorganic	6 oz.	1	8 oz. wide mouth glass jar		
DIOXIN SAMPLES					
2,3,7,8-TCDD	4 oz.	1	4 oz. or 8 oz. wide mouth glass jar		

*ALL MEDIUM LEVEL, HIGH HAZARD, AND DIOXIN SAMPLES MUST BE SEALED IN METAL PAINT CANS FOR SHIPMENT. THE OUTER METAL CAN MUST BE LABELED WITH THE NUMBER OF SAMPLES CONTAINED INSIDE.
**Should only be used in the presence of residual chlorine.

CLP Sample Collection Requirements (continued)

All low level sample containers must be enclosed in clear plastic bags before placing in the cooler for shipment.

All samples should be shipped in ice chests or coolers packed with non-combustible, absorbent packing material (vermiculite) surrounding the plastic enclosed sample bottles (or metal paint cans containing samples).

Traffic Reports, Dioxin Shipment Records, SAS Packing Lists, Chain of Custody Records, and any other shipping/sample documentation accompanying the shipment must be enclosed in a waterproof plastic bag and taped to the underside of the cooler lid.

Coolers must be sealed with custody seals in such a manner that the custody seal would be broken if the cooler were opened. Waterproof tape must cover the custody seal.

Water samples for duplicate analysis must be collected at double the volume specified for Extractables and triple the volume specified for Volatiles.

For water samples, one field blank should be supplied for per Case and one volatile trip blank should be supplied per shipment.

No additional volume is required for soil sample duplicate analyses.

oil blanks will be supplied to the Regions by EMSL/LV.

CRL Sample Collection Requirements

ANALYSIS	REQUIRED VOLUME	# OF CONTAINERS	CONTAINER TYPE	PRESERVATIVE	COMMENTS
Metals	1 Liter	1	1 Liter polyethylene bottle	HNO_3 to a pH<2	
Cyanide	1 Liter	1	1 Liter polyethylene bottle	NaOH to a pH>12 Cool, 4°C **Special handling if residual chlorine or sulfide is suspected.	
Mercury	500 ml	1	1 Liter polyethylene bottle	10 ml of preservative containing 0.05% (w/v)$K_2Cr_2O_7$ and 0.5% (w/v) HNO_3	
Pest/PCB (Organics)	1 Liter	1	1 80 oz. amber glass bottle filled half-way*	Cool, 4°C	
Acid/Base/Neutral Organics	1 Liter	1	1 80 oz. amber glass bottle filled half-way*	Cool, 4°C	
Volatile Organics	80 ml	2	40 ml glass vials	Cool, 4°C Samples must be free of headspace.	

*Or 1 - 1 liter amber bottle, full.

Two extra bottles each are required for the Matrix Spike/Matrix Spike Duplicate analyses of Pest/PCBs and Acid/Base/Neutrals; these can be full bottles.

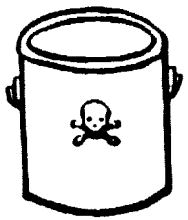
Four extra vials are required for the Matrix Spike/Matrix Spike Duplicate analyses of Volatiles.

No extra volume is required for the spike and duplicate analyses of Metals, Cyanide and Mercury.

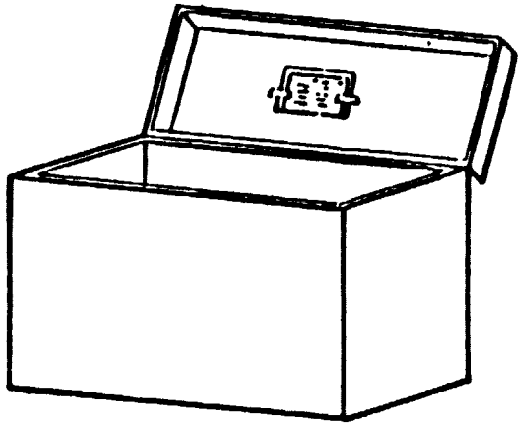
SAMPLE PACKAGING SUMMARY

- ENCLOSE ALL SAMPLE CONTAINERS IN CLEAR PLASTIC BAGS

- COOL ORGANIC LOW WATERS TO 4°C (DO NOT ICE DIOXIN SAMPLES, INORGANIC LOW WATERS, OR MEDIUM/HIGH LEVEL WATERS OR SOILS; ICE IS OPTIONAL FOR LOW LEVEL SOILS)



- PACK ALL MEDIUM AND HIGH LEVEL WATER AND SOIL SAMPLES IN METAL PAINT CANS
- SURROUND CONTENTS OF CAN WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL

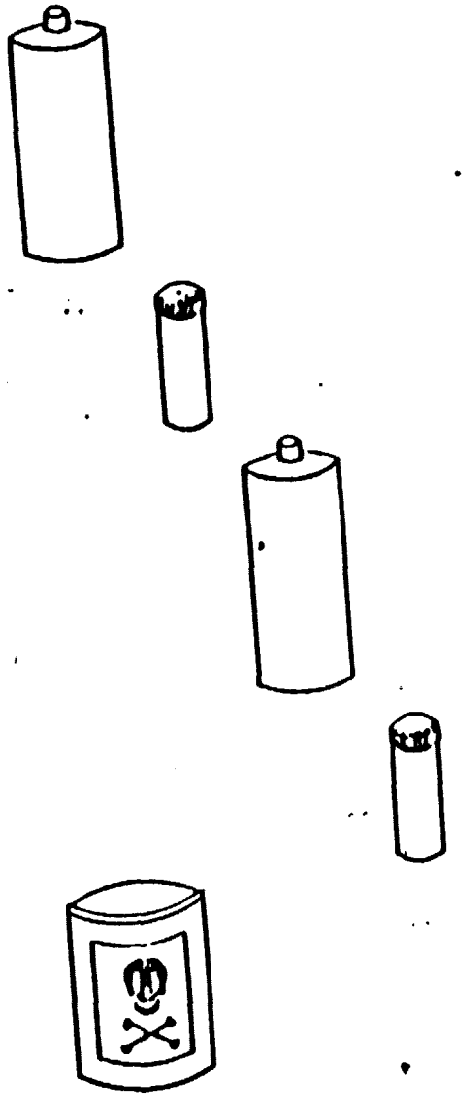


- PACK SEALED PAINT CANS OR PLASTIC-ENCLOSED SAMPLE BOTTLES IN SHIPMENT CONTAINER
- USE A METAL ICE CHEST FOR SHIPMENT
  (DO NOT USE CARDBOARD OR STYROFOAM CONTAINERS TO SHIP SAMPLES)
- SURROUND CONTENTS WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL
  (DO NOT USE EARTH OR ICE PACKING MATERIALS)
- TAPE PAPERWORK IN PLASTIC BAGS UNDER COOLER LID
- CLOSE COOLER AND SEAL WITH CUSTODY SEALS

CLP

INORGANIC SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
METALS ANALYSIS (LOW LEVEL)	1 LITER	1 X 1-LITER POLYETHYLENE BOTTLE
METALS ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 X 16-OZ. WIDE-MOUTH GLASS JAR
CYANIDE (CN^-) ANALYSIS (LOW LEVEL)	1 LITER	1 X 1-LITER POLYETHYLENE BOTTLE
CYANIDE (CN^-) ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 X 16-OZ. WIDE-MOUTH GLASS JAR



*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

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ORGANIC SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW LEVEL)	1 GALLON	2 X 80-OZ. AMBER GLASS BOTTLES OR 4 X 1-LITER AMBER GLASS BOTTLES
EXTRACTABLE ANALYSIS (MEDIUM LEVEL*)	1 GALLON	4 X 32-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	80 ML	2 X 40-ML GLASS VIALS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT *

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INORGANIC SAMPLE COLLECTION REQUIREMENTS

SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
METALS AND CYANIDE (CN^-) ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.	1 X 8-OZ. WIDE-MOUTH GLASS JAR
		OR
		2 X 4-OZ. WIDE-MOUTH GLASS JARS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

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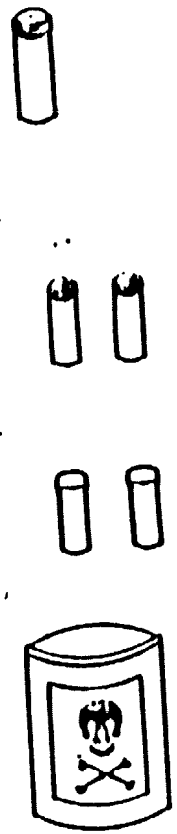
ORGANIC SAMPLE COLLECTION REQUIREMENTS

SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.	1 X 8-OZ. WIDE-MOUTH GLASS JAR OR 2 X 4-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	240 ML	2 X 120-ML WIDE-MOUTH GLASS VIALS



*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

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HIGH HAZARD SAMPLE COLLECTION REQUIREMENTS

	REQUIRED VOLUME		CONTAINER TYPE
LIQUID SAMPLES			
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR
SOLID SAMPLES			
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT