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**K-65 SILOS SAMPLING AND ANALYSIS**

**11-15-90**

**DOE/USEPA**

**DOE-277-91**

**2**

**LETTER**



**Department of Energy**

**FMPC Site Office**

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NOV 15 1990

DOE-277-91

Ms. Catherine A. McCord, Remedial Project Director  
U. S. Environmental Protection Agency  
Region V - 5HR-12  
230 South Dearborn Street  
Chicago, IL 60604

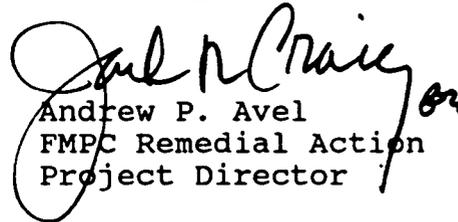
Dear Ms. McCord:

**K-65 SILOS SAMPLING AND ANALYSIS**

Enclosed is a Work Plan Addendum for K-65 Silos Residue Sampling activities. This addendum is required to perform the extractions to meet analysis holding times in support of the K-65 Silos and Operable Unit 4 sampling.

If you have any questions, please contact Jack Craig at FTS 774-6159.

Sincerely,

  
Andrew P. Avel  
FMPC Remedial Action  
Project Director

DP-84:Craig

Enclosure: As stated

cc w/encl.:

R. P. Whitfield, EM-40, FORS  
W. D. Adams, EW-90  
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D. A. Nixon, WMCO  
L. Dolan, IT  
R. L. Glenn, Parsons

# DOCUMENT CHANGE REQUEST

This form is used to initiate permanent changes to controlled distribution project-specific procedures, such as the QAPP, Work Plan, and Sampling Plan.

REQUEST NO. 60

Issue Date:

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Do Not Write In This Block

REQUESTOR: Derryl Stagg PHONE NO.: 738-3100 DATE: 11/6/90  
DOCUMENT TITLE: RI/FS Work Plan Addendum/K-65 Silos Sampling Analysis  
SECTION/PARAGRAPH/PAGE NO.: Work Plan Addendum DOCUMENT NUMBER: Work Plan  
ISSUE DATE: 11/6/90 LATEST REVISION DATE: 11/6/90

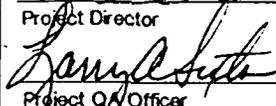
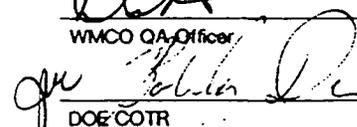
JUSTIFICATION: Customer request to meet EPA CLP holding times for volatile and semi-volatile analysis.

CONTENT OF CHANGE: See attached addendum.

### EFFECTIVE DATE OF CHANGE:

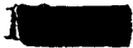
- When all approvals have been obtained: \_\_\_\_\_ Effective Date
- Other (Specify): \_\_\_\_\_

### REQUIRED APPROVALS:

	<u>11/6/90</u>		
Project Director	Date		
	<u>11-6-90</u>		<u>11-13-90</u>
Project QA Officer	Date	WMCO QA Officer	Date
<u>Will G. Hartzel For John Rizzor</u>	<u>11-7-90</u>		<u>11/13/90</u>
Technical Manager	Date	DOE COTR	Date

### TO BE COMPLETED BY DOE

- A. Prior EPA notification required?  Yes  No
- B. Prior EPA approval required?  Yes  No
- C. Immediate implementation?  Yes  No



**EXTRACTION PROCEDURES IN A GLOVE BOX**



## MEDIUM LEVEL VOLATILE EXTRACTION IN GLOVE BOX

### 1.0 Introduction

The purpose of this procedure is to extract the volatile compounds in methanol to meet holding times in support of the K-65 sample analysis.

Place all equipment, supplies and samples in the Glove Box.

#### A. Equipment

1. Balance
2. VOA Vials

#### B. Supplies

1. Premeasured methanol in VOA vials

### 2.0 Procedure

- 2.1 Certify the balance is in calibration by weighing traceable weights.
- 2.2 Note and record the actual weight to 0.1 (call out weights to assistant)
- 2.3 Add premeasured appropriate solvent 9.0 ml to 4 g of sample, and 1.0 ml of surrogate standard mix. Cap the vial and shake for 2 minutes.
- 2.4 Allow the solid material to settle.
- 2.5 Take 1.0 ml of the methanol for analysis and place into a clean labeled GC vial.
- 2.6 Take 4.0 ml for screening by placing extract on a planchette and evaporating.

Reference: Organic CLP SOW 2/88

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## EXTRACTION PROCEDURE FOR SEMI-VOLATILES IN A GLOVE BOX

### 1.0 Introduction

The purpose of this extraction procedure is to extract the semi-volatile organics and meet extraction holding times for higher level radioactive materials in support of K-65 sample analysis.

These extracts shall be screened for radioactive contamination before shipment to the laboratory.

Place extraction equipment supplies and samples in glove box.

- A. Equipment
  - 1. Sonic Disrupter
  - 2. Beakers
  - 3. Flasks
  - 4. Funnels
  - 5. Ring Stands With Clamps
  
- B. Supplies
  - 1. Methylene Chloride: Acetone
  - 2. Anhydrous Sulfate
  - 3. Filter Paper
  - 4. Spiking Solutions

### 2.0 PROCEDURE

#### 2.1 Sample handling:

2.1.1 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.

2.1.2 Waste samples: Samples consisting of multiple phases must be prepared by the phase separation method before extraction. [See Chapter Two of SW-846]. The sonication procedure is for solids only.

2.2 Extraction method for samples expected to contain low concentration of organics (<20 mg/Kg)

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- 2.2.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Place the approximately 30 g of sample into a 400 mL beaker. Record the weight to the nearest 0.1 g. Mix 60 g of anhydrous sodium sulfate into each sample using a spatula. The sample should be freeflowing at this point. Add 500 uL of surrogate standard solution to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spike standard. 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 1 uL injection). Immediately add 100 mL of 1:1 methylene chloride:acetone.
- 2.2.2 Place beaker in ice bath
- 2.2.3 Place the bottom surface of the tip of the 3/4 in. disrupter horn about 1/2 in. below the surface of the solvent, but above the sediment layer.
- 2.2.4 Sonicate for 3 min., with the output control knob set at 10, the mode switch on Pulse and percent-duty cycle knob set at 50%. Do NOT use the microtip probe.
- 2.2.5 Decant and filter extracts through Whatman No. 41 filter paper using gravity filtration or centrifuge and decant extraction solvent.
- 2.2.6 Repeat the extraction two or more times with two additional 100 mL portions of solvent. Decant the extraction solvent after each sonication. On the final sonication, pour the entire sample into the funnel and rinse with extraction solvent.
- 2.2.7 Take 4 mL of extract for screening by placing extract on planchette and evaporate. Make note on preparation sheet.
- 3.0 Cleaning the horns
- 3.1 Rinse the horns with solvent.
- 3.2 Place 75 to 100 mL of solvent in a clean beaker and follow 1.2.2 and 1.2.3 except sonicate for 5 minutes.
- 4.0 Clean up and removal from glove box.
- 4.1 Place the Sodium Sulfate and extracted sample into solid residue containers.
- 4.2 Place the cleaning solvent into the non-RAD rinsate container.

Reference: Organic CLP SOW 2/88