SP3

SAMPLE PREPARATION

1.0 PURPOSE

The purpose of this procedure is to provide a procedure for sample preparation for various radiochemical analyses.

2.0 RESPONSIBILITIES

2.1 Field Survey Personnel

- Create Lab Work Request (LWR) using the IEAV Database
- Include instructions when special sample processing conditions exist, e.g., the sample may have a volatile isotope (H-3, C-14, Tc-99, etc.) present

2.2 Laboratory Manager and Laboratory Personnel

- Create LWR using the IEAV database when samples are not associated with a Field Survey Project
- Include instructions when special sample processing conditions exist, e.g., the sample may have a volatile isotope (H-3, C-14, Tc-99, etc.) present
- Review each LWR to ensure special sample processing conditions are taken into consideration
- Process samples according to the procedure

3.0 SAMPLE PREPARATION

3.1 Soil and Sediment for Gamma Spectroscopy

3.1.1 If a sample requires analysis for volatile nuclides (e.g. H-3, C-14, Tc-99, or iodine) go to 3.1.9.

3.1.2 Dry the entire the sample in an appropriate container at 110°C (±10°C) for 8-16 hours, or until the sample is completely dry. When drying several samples, the samples should be loaded into the drying oven from top to bottom. If the percent moisture of the sample is requested, the wet weight of the sample is recorded on the wet to dry form before the sample is placed in the drying oven. **See step 3.1.2 of SP3 JHA.**

3.1.3 Turn off the drying ovens and allow the samples to cool. After the samples have cooled, remove them from the drying oven starting from the bottom

and ending at the top. If the percent moisture of the sample is requested, the dry weight of the sample is recorded on the wet to dry form before proceeding to the next step. **See step 3.1.3 of SP3 JHA.**

3.1.4 If the samples from a given task do not require complete homogenization, use a steel mallet to break up each sample. Go to step 3.1.8. Clean the mallet thoroughly with NO COUNT, or equivalent, after each use. **See step 3.1.4 of SP3 JHA.**

3.1.5 For samples requiring homogenization, place each sample in a labeled paint can with 3 to 5 steel balls (if necessary), seal the can, and secure the can on the shaker. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.1.6 Shake each sample for 1 hour. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.1.7 With a can opener, open the end of the can without the lid and remove the steel balls. Go to step 3.1.12 for cleaning steps for the steel balls. Check the sample to ensure that there are no large, solid portions remaining. If the sample needs further blending, transfer the sample to a new container and repeat steps 3.1.5 and 3.1.6. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.1.8 If the sample contains rocks, pass it through a 0.25" sieve to remove the larger rocks. Clean the sieve and the bottom pan between samples by vacuuming it with a HEPA filter vacuum and using a foaming surfactant cleaner, such as NO COUNT. **See step 3.1.8 of SP3 JHA.**

3.1.9 Pour the sample into a tared, labeled counting container. Use a counting container that will hold it with a minimum of void space. Tape the lid of the container to prevent spillage. **See step 3.1.9 of SP3 JHA.**

3.1.10 Weigh and record the net weight of the sample on the sample label.

3.1.11 Clean the exterior of the container and all processing equipment.

3.1.12 Place the steel balls, if necessary, in a paint can containing clean sand. Place the paint can on the shaker for one hour.

3.1.13 Remove the steel balls, if necessary, from the paint can.

3.1.14 Depending on the contaminant of interest, the steel balls may be gamma counted. The steel balls are considered clean if there are no radionuclides identified by the gamma spectroscopy software.

3.1.15 The analyst may also survey the steel balls with a HP-260 probe or an AC50 probe. A background should be obtained for either probe before surveying the steel balls. The results of the survey of the steel balls should not exceed

the MDC of the selected probe in order for the steel balls to be used to process the next sample.

3.2 Soil and Sediment for Wet Chemical Analyses

3.2.1 For a sample requiring the analysis of volatile isotopes, the sample is homogenized by hand, if possible. A representative aliquot is taken and the wet chemical analysis is performed.

3.2.2 For a sample not requiring the analysis of volatile isotopes, dry the entire sample in an appropriate container at 110°C (±10°C) for 8-16 hours, or until the sample is completely dry. When drying several samples, the samples should be loaded into the drying oven from top to bottom. **See step 3.1.2 of SP3 JHA.**

3.2.3 Turn off the drying ovens and allow the samples to cool. After the samples have cooled, remove them from the drying oven starting from the bottom and ending at the top. **See step 3.1.3 of SP3 JHA.**

3.2.4 Place each sample in a labeled paint can, add 3 to 5 steel balls (if necessary), seal the can, and secure the can on the shaker. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.2.5 Shake each sample for 1 hour. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.2.6 With a can opener, open the end of the can without the lid and remove the steel balls. Go to step 3.1.12 for cleaning steps for the steel balls.. **See steps 3.1.5 - 3.1.7 of SP3 JHA.**

3.2.7 If there is visible organic material in the sample, place it in an ashing container, such as a Pyrex beaker and record the weight. The dry weight of the sample is recorded on the dry to ash form. The sample is placed in a muffle furnace at 500°C for at least 4 hours.

3.2.8 Place the ashing container in the muffle furnace. Check the "Set Point" on the furnace to make sure the temperature is correct. If the "Set Point" does not read 500°C, change the "Set Point" to 500°C. **See step 3.2.3 of SP3 JHA.**

3.2.9 After the samples have completed ashing, turn off the furnace. Allow a minimum of 2 hours for the furnace to cool before opening the door. **See step 3.2.4 of SP3 JHA.**

CAUTION: NEVER ATTEMPT TO REMOVE A SAMPLE WHILE THE FURNACE IS ON AND/OR HOT. THERE IS A POTENTIAL FOR ELECTRICAL SHOCK.

3.2.10 After cooling, the sample is then re-weighed and the ashed weight is recorded on the dry to ash form. The dry to ash ratio is then calculated.

3.2.11 The ashed sample is used for analysis with reference to dry weight for calculation of final results.

3.3 Vegetation for All Radiochemical Analyses

3.3.1 Wash the vegetation to remove particles, which may contain radionuclide contamination, from the outer surface.

3.3.2 Determine the wet weight of the sample.

3.3.3 Perform analysis of any volatile radionuclides, if required.

3.3.4 If analysis is not to be performed for volatile radionuclides, dry the vegetation at 110°C (±10°C) for 8 to 16 hours. Weigh the dry sample and calculate the wet to dry weight ratio using the wet to dry form at the end of the procedure. See Steps 3.1.8 and 3.1.9 for sample loading. **See step 3.1.2 and step 3.1.8 of SP3 JHA.**

3.3.5 Ash each sample at 500°C for at least 4 hours (samples may be ashed overnight). Refer to Steps 3.2.7 through 3.2.10 for the ashing procedure, precautions, and JHAs. Weigh the ash material and determine the dry and wet to ash weight ratios using the wet to dry form at the end of the procedure. Analysis should be performed on the vegetation ash and the final results calculated against the original wet weight.

3.4 Water for All Radiochemical Analyses Except for Plutonium, and/or Americium-Curium

NOTE: ALL WATER SAMPLES HAVE A MAXIMUM HOLDING TIME OF SIX MONTHS.

3.4.1 Unless suspended and dissolved solid results are required by the client or there are radioisotopes present that could possibly be volatilized by acidification, acidify all water samples to a pH of 1-2 using nitric or hydrochloric acid.

3.4.2 Samples, acidified or non-acidified, requiring filtration are filtered through a 0.45 micron pore size. The filters with the suspended portion of each sample are placed in a labeled Petri dish.

3.4.3 If requested by the client, suspended and dissolved solid fractions are analyzed by the requested analytical procedure for the isotope(s) of interest.

3.4.4 All water samples will be returned to the licensee and/or client or discarded once the six month holding time is exceeded.

3.5 Water for Plutonium and/or Americium-Curium Analysis

NOTE: ALL WATER SAMPLES HAVE A MAXIMUM HOLDING TIME OF SIX MONTHS.

3.5.1 Samples are filtered through a 0.45 micron pore size. The filters with the suspended portion of each sample are placed in a labeled Petri dish.

3.5.2 The sample filtrate is returned to the original sampling containers. Nitric acid or hydrochloric acid is then used to adjust the pH to approximately 0.6 (approximately 10 mL concentrated HNO_3 per liter of sample). After the addition of the nitric or hydrochloric, add 2 to 5 drops of hydrofluoric acid to the sample to help dissolve any potential plutonium adhering to the sample container.

3.5.3 The acidified liquid and if requested by the client, the filter portion of each sample are analyzed by for the isotope(s) of interest.

3.5.4 All water samples will be returned to the licensee and/or client or discarded once the six month holding time is exceeded.

3.6 Smears/Swipes

3.6.1 Remove the smear from its packet using forceps, touching as little of the smear as possible. The smear may contain loose contamination; effort should be taken to minimize loss or movement of material on the smear. Do not handle smears with bare hands.

3.6.2 If tritium or carbon-14 analysis is required, the smear should come to the laboratory in a scintillation vial containing 5 to 10 mL of de-ionized water. Add 10 mL of scintillation cocktail, Ultima Gold or equivalent. Shake well to mix the water and the cocktail. Submit to counting room for liquid scintillation counting.

3.6.3 Each smear requiring gross alpha and beta counting should be secured to the planchette with double-sided tape, placed in a carrier with the same number as the smear, and counted on the low background alpha/beta counter.

3.6.4 After counting, return the smear, or smear and planchette, to its packet for further analysis or archival.

3.7 Miscellaneous Samples

Miscellaneous samples will be processed to meet client requirements. Processing will

be documented in the task folder indicating what actions were taken to put the sample(s) in a form that could be analyzed. Actions may include, but not be limited to, blending, grinding, acid dissolution, etc.

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SOIL PREP ASSIGNMENT SHEET

TASK #: _____ ACTIVITY LEVEL: _____

ASSIGNED TO: _____ DATE: _____

LWR #: _____

SAMPLES & INSTRUCTIONS: _____

OVEN USE LOG

Oven #	Beginning Sample #	End Sample #	Date/Start Time		Date/End Time		Temp in Celsius	Initial when entered in Oven Log

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WET TO DRY CALC

TASK#:			DATE:			INPUT BY:		
Sample Number	CRUCIBLE NUMBER	CRUCIBLE WEIGHT	CRUC + WET SAMPLE WEIGHT	CRUC + DRY SAMPLE WEIGHT	Grams WET/ Gram DRY	Percent Moisture	Percent Solids	Grams water per gram wet soil
		Analyst review:				Date:		
		Reviewed by:				Date:		
		Given to:				Date:		

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DRY-TO-ASH WEIGHT DATA SHEET

TASK# _____ Date: _____ Input by: _____

Check balance before weighing the samples. All weights are in grams.

Sample Number	CRUCIBLE NUMBER	CRUCIBLE WGHT (A)	CRUC + DRY SAMPLE WGHT (B)	CRUC + ASH SAMPLE WGHT (C)	DRY/ASH RATIO

Equation for calculating the dry to ash ratio:

$$\frac{B-A}{C-A}$$ = No. grams of dry sample per gram of ash sample

Analyst: _____ Date: _____

Reviewed by: _____ Date: _____