USTUR 105: Preparation of Tissue for Actinide Determination: Dry Ashing of Tissues

Purpose	Preparation of Tissue for Actinide Determination: Dry Ashing of Tissues	**Method Number**	USTUR 105
Original Date	3/1/00	**Revisions By**	Gail E. Deckert
Revision Number	0	**Approved By**	Jim Elliston
Revision Date	3/1/00	**Approval Date**	1/31/01

1. Principle of Method

1.1. Selected tissue samples are oven-dried at 120°C and muffled at 500°C.

1.2. Dry ash weights are determined.

2. Apparatus

2.1. Beakers: borosilicate glass, various sizes.

2.2. Analytical Balance: with at least two-decimal-place accuracy.

2.3. Watch glasses: Pyrex, ribbed, in sizes to fit beakers in use.

2.4. Drying oven, vented.

2.5. Muffle furnace: adjustable to 500°C.

2.6. Biological Safety Cabinet.

2.7. High-Temperature Marker.

3. Sample Drying

NOTE: See USTUR020 regarding all tissue handling, including thawing and weighing of tissue into beakers, which will be performed in the Biological Safety Cabinet.

3.1. Record the sample number using a black marking pen on a clean, tared borosilicate glass beaker. In a Biological Safety Cabinet, unpackage the tissue and place the tissue in the beaker. Record the sample net weight on a sample control sheet (See Figure 1). Analytical balance must be capable of 1% precision.

3.2. Cover the beaker with a ribbed watch glass and place in the drying oven.

3.3. Dry sample for 2-5 days. Drying time is dependent on the size and moisture content of the sample. Samples may be stored in the drying oven when they are completely dry.

4. Sample Muffle-Ashing

NOTE: The very large muffle furnace exhibits temperature gradients. Care must be taken to use the correct temperature for samples on the bottom or in the middle of the furnace.

4.1. Using a high temperature marker, record the sample number on the beaker over that of the marking pen. (NOTE: The beaker must be warm for the marker to work.)

4.2. Transfer dry samples to a muffle furnace and heat samples according to the following schedule. Increase the temperature in increments of 50°C every 4 hours.

NOTE: Liver samples and higher fat content samples need to have temperatures rise at a slower rate, based on the amount of fumes coming off samples. Between 200 and 300°C, the temperature should be increased by 25°C increments, holding at a given temperature until evolution of smoke stops. At 350°C, the temperature may have to be held longer than 4 hours, if samples evolve a lot of smoke.

Day 1 150°C start, four hours later move to 200°C, then four hours later move to 225°C and hold for the night.

Day 2 move to 250°C in the morning, four hours later move to 275°C, then four hours later move to 300°C and hold for the night.

Day 3 move to 325°C in the morning, four hours later move to 350°C, then four hours later move to 400°C and hold for the night.

Days 4-34 move to 450°C in the morning, four hours later move to 500°C, and then maintain the temperature at 500°C for 30 days, shut off furnace, and allow the samples to cool.

4.3. Cool samples to room temperature. Record ash weights of samples. For small samples you must use a balance capable of 1/100 of sample weight (e.g. for a 1 g sample the balance must be capable of 0.01 precision).

4.4. Proceed to USTUR110 Wet Ashing of Tissues.

5. Source Material

5.1. H. A. Boyd, B. C. Eutsler, and J.F. McInroy, “Determination of Americium and Plutonium in Autopsy Tissue: Methods and Problems,” in Actinides in Man and Animals, Proceedings of the Snowbird Actinide Workshop, Oct 15-17, 1979, M. E. Wrenn, scientific editor (R. D. Press, Salt Lake City, Utah, 1981), pp. 43-52.

5.2. LANL Procedures manual. RESL Procedure. Claude Sill’s Method.