USTUR 110: Preparation of Tissue for Actinide Determination: Wet Ashing of Tissues

Purpose	Wet Ashing of Tissues	**Method Number**	USTUR 110
Original Date	3/1/00	**Revisions By**	Michael Aman
Revision Number	0	**Approved By**	Jim Elliston
Revision Date	05/25/05	**Approval Date**	05/31/05

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Section 3 of this procedure.

1. Principle of Method

1.1. Selected tissue samples are wet-ashed.

2. Apparatus

2.1. Beakers: borosilicate glass, various sizes.

2.2. Watch glasses: Pyrex, plain and ribbed, in sizes to fit beakers in use.

2.3. Fume hood.

2.4. Hot plates: adjustable to 140°C.

2.5. Transfer pipettes.

2.6. Spot Check Surface Thermometers

3. Reagents

3.1. Nitric acid (concentrated, 69-71%, reagent grade).

3.2. Hydrogen peroxide (concentrated, 30%, reagent grade).

4. Sample Acid-Ashing

NOTE 1: If any obviously foreign solid material is present at this stage (e.g. surgical staples), it should be removed, placed in a vial and labeled. This material may be analyzed later. Record on sample sheet.

NOTE 2: Do not increase the temperature above 150°C due to the formation of metaphosphate complexes.

4.1. Add concentrated HNO_3 to the muffled sample to completely cover the ash and thoroughly rinse the sides of the beaker. Cover the beaker with a plain watch glass

and reflux on a hot plate set at 140°C until the sample loses its viscosity. If the sample wet ashes completely in HNO_3, skip to USTUR 115.

4.2. The following day, change to a ribbed watch glass and periodically add 1 to 2 mL of 30% H_2O_2 drop wise, allowing foaming to subside between additions. Bring solution back up to temperature.

4.3. Keep the solution volume at a minimum, by only adding concentrated nitric acid to prevent the sample from drying out.

4.4. Wet ashing is complete when the sample loses its brown color. A minimum of 4 hours will be necessary for this step.

4.5. Once the liquid sample loses its brown color, use a ribbed watch glass to prevent contamination and take the sample to dryness. The dried ash material should be white, cream, or purple in color, but many various colors have been observed. Use the solution color as the prominent index of whether the sample has been oxidized completely and note the precipitate color and uniformity.

4.6. Choose one of the following depending on the conditions of the sample:

4.6.1. All lung and lymph node tissues require HF treatment and/or KF fusion and liver and brain samples may require HF treatment or KF fusion; proceed to USTUR120, HF acid digestion of soft tissues.

4.6.2. Otherwise, proceed to USTUR115, Dissolution of sample ash.

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.