USTUR 115: Dissolution of Sample Ash

Purpose	Dissolution of Sample Ash	**Method Number**	USTUR 115
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

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. The amount of acid required to dissolve the sample ash will vary between samples. It will vary due to sample mass, ash content and tissue type. The amount of concentrated HCl and HNO_3 used should be limited to the quantity required for complete ash dissolution. In general, bone samples will require more acid than soft tissues, and large samples will require more acid than small samples. The final concentration of the sample solution should not exceed 8M HCl. The final sample weight will be adjusted to between 100 and 1000g.

2. Apparatus

2.1. Beakers: borosilicate glass, various sizes.

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

2.3. Fume hood.

2.4. Hot plates: adjustable to 150°C.

2.5. Hot plates: magnetic stirring, adjustable to 150°C.

2.6. Stir bars: Teflon-coated.

2.7. Transfer pipettes.

2.8. Flashlight.

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

2.10. Bottles: I-chem; 8, 16, and 32 oz, with teflon-lined caps.

3. Reagents

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

3.2. Hydrochloric acid (concentrated, 36.5-38%, reagent grade).

3.3. Hydrochloric acid (6 M). Add 500 mL of concentrated HCl to 300 mL nanopure H_2O. Dilute to 1000 mL with nanopure H_2O.

4. Sample Ash Dissolution

NOTE: These volumes correspond to a wet sample weight of 100g. Scale volumes up, if the sample weight is larger than this.

4.1. Add 5-10 mL of concentrated HNO_3 to the beaker containing the ashed tissue sample. Place the beaker on a hot plate set to 140°C. Once the sample is warm, add 5-10 mL of concentrated HCl. Heat until the major part of the ash has gone into solution.

NOTE: Experience has shown that the addition of small amounts of HNO_3 at this step will aid in the dissolution of the ash in the following step.

4.2. Add 50-600 mL of 6-8 M HCl to the sample beaker. Place a plain watch glass on the beaker and reflux at 140°C until the ash is dissolved.

4.3. To check the solution for undissolved particles, shine the light from a flashlight through the bottom of the beaker (in a darkened room). If light is reflected from particles of ash, additional treatment may be necessary.

4.4. Place the sample on a stirring hot plate, add a Teflon-coated stir bar, and stir-heat for one hour. If the sample remains undissolved, add additional amounts of 6-8 M HCl and stir-heat for several hours.

4.5. If light is still reflected from particles of ash, additional treatment will be necessary. If the sample solution is not clear:

4.5.1. For soft tissues, proceed to USTUR120 HF acid digestion of soft tissues (unless HF acid digestion has already been performed on the sample).

4.5.2. If the bone sample has not dissolved;

4.5.2.1. Evaporate ash to dryness on a hot plate set at 140°C.

4.5.2.2. Add enough HNO_3 to cover the salts. Cover the sample with a watch glass and reflux for a minimum of 1 hour.

4.5.2.3. Add 5-10 mL concentrated HCl and repeat from Step 4.2 above.

4.5.3. For all other samples, which require filtering, proceed to USTUR125, Sample fusion using potassium fluoride.

4.6. If the sample is completely dissolved, proceed to step 5.

5. Sample Storage

5.1. Choose an I-Chem bottle of the appropriate size, label with the sample name and tare weight of the bottle.

5.2. Cool the sample to room temperature and transfer the sample to the labeled bottle. Rinse the beaker with 6-8 M HCl and pour the rinse into the bottle. Repeat twice.

5.3. Using an analytical balance, bring the solution weight of the sample to the nearest 5g with 6-8 M HCl. Record the gross weight on the bottle and the solution weight on the control sheet. Mix the solution thoroughly.

6. Source Material

6.1. 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.

6.2. LANL Procedures manual. RESL Procedure. Claude Sill's Method.