

UNITED STATES TRANSURANIUM AND URANIUM REGISTRIES
ANALYTICAL PROCEDURE MANUAL

USTUR 120: Hydrofluoric Acid Digestion of Soft Tissues

Purpose	Hydrofluoric Acid Digestion of Soft Tissues	Method Number	USTUR 120
Original Date	3/1/00	Revisions By	Gail E. Deckert
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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 selected soft tissue is treated with hydrofluoric acid to dissolve any siliceous material.

2. Apparatus

- 2.1. Beakers: Teflon, with covers.
- 2.2. Fume hood.
- 2.3. Hot plates: adjustable to 250°C.
- 2.4. Transfer pipettes.
- 2.5. Spot check surface thermometers.
- 2.6. Teflon scraper.

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 H₂O. Dilute to 1000 mL with nanopure H₂O.
- 3.4. Hydrofluoric acid (concentrated, 48.0-51.0%, reagent grade).
- 3.5. Boric acid (reagent grade).

4. Hydrofluoric acid digestion of soft tissue

CAUTION: Use extreme care with HF. Double gloves are required. Wash gloves after use.

- 4.1. Transfer the sample into the proper size Teflon beaker using 6-8 M HCl. If necessary, use a Teflon scraper to dislodge ash from the sides of the beaker.
- 4.2. Evaporate the sample to dryness at 120°C.
- 4.3. Add concentrated HNO₃ to completely cover the ash with approximately 2-3 cm height of acid.
- 4.4. Estimate the volume of HNO₃, then add one-fifth that volume of HF.
- 4.5. Cover the Teflon beaker with a Teflon cover and reflux for 2-3 hours on a hot plate with a surface temperature of 250°C. (CAUTION: Do Not Allow beaker contents to go dry.)
- 4.6. Remove the beaker from the hot plate and cool until the acid is no longer fuming. Add the same volume of HF as in step 4.4 and reflux for an additional 2-3 hours.
- 4.7. If the sample still contains undissolved particles repeat the HF addition for a third time and reflux for an additional 2-3 hours.

NOTE: Larger samples may require extra HF additions and longer reflux periods.

- 4.8. Remove the cover and evaporate to dryness at 120-140°C.
- 4.9. Wash the sides of the beaker with concentrated HNO₃. Add ~50 mg boric acid to the beaker, swirl. The beaker should contain at least 1 cm depth of acid, then heat to dryness at 120-140°C.
- 4.10. Wash the sides of the beaker with concentrated HNO₃ a second time and fill to a depth of 1 cm, then bring to dryness at 120-140°C.
- 4.11. 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.