USTUR 125: Sample Fusion using Potassium Fluoride

Purpose	Sample Fusion using Potassium Fluoride	**Method Number**	USTUR 125
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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 sample containing undissolved material is filtered using a Gelman Supor membrane filter in a vacuum filtration apparatus.

1.2. The filter is ashed using a blast burner, then wet ashed with HNO_3 and HF.

1.3. The residue is fused with KF over a blast burner.

1.4. The fusion cake is treated with boric acid to remove any remaining fluoride ions and wet ashed with HNO_3.

1.5. The fusion cake is dissolved in HNO_3 and transferred to a beaker.

2. Apparatus

2.1. Beakers: borosilicate glass, various sizes.

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

2.3. Watch glasses: sizes to fit beakers in use.

2.4. Fume hood.

2.5. Hot plates: adjustable to 250° C.

2.6. Hot plates: magnetic stirring, adjustable to 140° C.

2.7. Stir bars: Teflon-coated.

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

2.9. Platinum dish or crucible.

2.10. Platinum-tipped tongs.

2.11. Blast burner.

2.12. Vacuum Filtration Apparatus

2.13. Glass funnel, no stem

2.14. Gelman Supor membrane filters

2.15. Flashlight

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.

3.4. Hydrofluoric acid (concentrated, 48.0-51.0%, reagent grade).

3.5. Boric acid (reagent grade).

3.6. Potassium Fluoride, anhydrous (reagent grade).

4. Sample Fusion using Potassium Fluoride (KF).

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

NOTE 1: Only use platinum-tipped tongs to handle the platinum dish.

NOTE 2: Never add HCl or HCl/HNO_3 to the platinum dish.

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

4.2. Filter sample containing undissolved material into a vacuum filtration apparatus. Gelman Supor membrane filters are typically used.

4.3. Rinse the sample beaker twice with 6M HCl; pour the rinse through the filter. Check to ensure that all particulates are rinsed out of the beaker. Then rinse the filtration funnel twice with 6M HCl. Set the sample beaker and the watch glasses aside for later use.

4.4. Pour the sample from the vacuum flask into the tared bottle using a glass funnel. Rinse the flask twice with 6M HCl and add to the sample in the bottle. Rinse the funnel one time with 6M HCl. Cap the bottle until fusion is complete.

4.5. Ash the membrane filter containing undissolved material in a 50-mL platinum dish with a blast burner. Be careful to heat the membrane sample slowly, to prevent the membrane from igniting. If the sample does ignite, immediately extinguish by covering the dish with a watch glass.

4.6. Allow the platinum dish to cool. Rinse the sides of the platinum dish, containing the ashed membrane residue, with approximately 5-10 mL of concentrated nitric acid (16 M HNO_3) to wet-ash; heat to dryness on a hot plate set up to 250° C.

CAUTION: Do not expose any unprotected skin to the HF vapors while drying.

4.7. Add approximately 5 mL of concentrated hydrofluoric acid (48% HF) to the dish and heat to dryness on a hot plate set up to 250° C to remove siliceous material.

4.8. Pu/Am recoveries are significantly reduced by the presence of fluorides therefore for Pu/Am cases add approximately 0.5 g anhydrous KF (or less depending on the amount of residue and the size of the platinum dish), 1-5 drops each of 16 M HNO_3 and 48% HF. For U/Th cases approximately 1 g anhydrous KF (more or less depending on the amount of residue and the size of the platinum dish) can be used if Pu/Am analyses are not being conducted, 1-5 drops each of 16 M HNO_3 and 48% HF.

4.9. Fuse the residue with the KF over a blast burner, swirling the liquid fusion across the bottom and lower sides of the dish to ensure complete sample fusion. The residue should be adequately fused when the liquid fusion is clear.

4.10. Once the fusion is cool, rinse the sides of the platinum dish by adding approximately 5-10 mL of 16 M HNO_3, and dry the fusion cake, repeat once. Add another 5-10 mL of 16 M HNO_3 and approximately 50 mg of boric acid to the fused solution, swirl, and heat to dryness at 140° C.

4.11. Rinse the sides of the platinum dish by adding approximately 5-10 mL of 16 M HNO_3, whirl, and heat to dryness at 140° C.

NOTE: The volume of the fusion solution should not be more than 25 mL, if 16 M HNO_3 is used for dissolution.

4.12. Add enough concentrated (16 M) HNO_3 to dissolve the fusion cake and heat to 120°C until dissolved. Transfer the dissolved cake solution to a beaker and rinse the dish twice with 16 M HNO_3. Place fusion solution on a stirring hot plate and

examine the sample with a flashlight in a darkened room to ensure complete dissolution of the fusion cake.

4.13. The fused solution may then be added to the original dissolved sample solution, contained in the labeled I-Chem bottle.

4.14. Using 6-8 M HCl, bring the solution level up to the nearest 5 g of weight. Record the solution weight on the control sheet and on the bottle. Mix solution thoroughly.

5. Source Material

5.1. Adapted from the INEL Radiological and Environmental Sciences Laboratory Analytical Chemistry Branch Procedures Manual, 1982.

5.2. 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.

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