Designation: C1163 – 14 (Reapproved 2023)

Standard Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride[1]

This standard is issued under the fixed designation C1163; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the preparation of separated fractions of actinides for alpha spectrometry. It is applicable to any of the actinides that can be dissolved in dilute hydrochloric acid. Examples of applicable samples would be the final elution from an ion exchange separation or the final strip from a solvent extraction separation.[2]

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C859 Terminology Relating to Nuclear Materials

C1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry

D1193 Specification for Reagent Water

D3084 Practice for Alpha-Particle Spectrometry of Water

3. Terminology

3.1 For definitions of terms in this standard, refer to Terminology C859.

4. Summary of Test Method

4.1 Guidance is provided for the sample mounting of separated actinides using coprecipitation with neodymium fluoride. The purified samples are prepared and mounted on a membrane filter to produce a deposit that yields alpha spectra of sufficient quality for most analytical methodologies. Samples can be prepared more rapidly using coprecipitation than by electrodeposition and have comparable resolution.

5. Significance and Use

5.1 The determination of actinides by alpha spectrometry is an essential function of many environmental and other programs. Alpha spectrometry allows the identification and quantification of most alpha-emitting actinides. Although numerous separation methods are used, the final sample preparation technique has historically been by electrodeposition (Practice C1284). However, electrodeposition may have some drawbacks, such as time required, incompatibility with prior chemistry, thick deposits, and low recoveries. These problems may be minimized by using the neodymium fluoride coprecipitation method whose performance is well documented (**1-6**).[4] To a lesser extent cerium fluoride has been used (**7**) but is not addressed in this practice.

5.2 The sample mounting technique described in this practice is rapid, adds an additional purification step, since only those elements that form insoluble fluorides are mounted, and the sample and filter media can be dissolved and remounted if problems occur. The recoveries are better and resolution approaches normal in electrodeposited samples. Recoveries are sufficiently high that for survey work, if quantitative recoveries are not necessary, tracers can be omitted. Drawbacks to this technique include use of very hazardous hydrofluoric acid and the possibility of a non-reproducible and ill-defined counting geometry from filters that are not flat and may not be suitable for long retention. Also, although the total turn around time for

[1] This practice is under the jurisdiction of ASTM Committee C26 on the Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Jan. 1, 2023. Published January 2023. Originally approved in 1992. Last previous edition approved in 2014 as C1163 – 14. DOI: 10.1520/C1163-14R23.

[2] Hindman, F. D., "Actinide Separations for α Spectrometry Using Neodymium Fluoride Coprecipitation," *Analytical Chemistry*, 58, 1986, pp. 1238–1241.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

coprecipitation may be less than for electrodeposition, coprecipitation requires more time and attention from the analyst.

6. Interferences

6.1 Calculation of a result from a sample that gives poor resolution should not be attempted since it probably implies an error in performing the separation or mounting procedure.

7. Apparatus

7.1 *Alpha Spectrometer*—A system should be assembled that is capable of 60 keV to 70 keV resolution on an actual sample prepared by this practice, have a counting efficiency of greater than 20 %, and a background of less than 0.005 cpm over each designated energy region. Resolution is defined as the full-width at half-maximum (FWHM) in keV, or the distance between those points on either side of the alpha energy peak where the count is equal to one-half the maximum count. Additional information can be found in Practice D3084.

7.2 *Filter*—25 mm 0.1 µm pore, polypropylene membrane filter or equivalent that will provide suitable alpha spectrometry resolution.[5]

7.3 *Vacuum Funnel*—Polysulfone twist-lock with stainless steel screen for filter mounting.[5]

7.4 *Ultrasonic Bath.*

7.5 *Plastic Centrifuge Tube,* 50 mL.

7.6 *Stainless Steel Disk,* 2.54 cm diameter.

7.7 *Infrared Heat Lamp.*

7.8 *Tape,* double-sided.

8. Reagents

8.1 *Purity of Reagents*—Reagent-grade chemicals must be used in all procedures. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, if such specifications are available.[6] Other grades may be used, if it is ascertained that the reagent is of sufficiently high purity to permit its use without reducing the accuracy of the determination. All reagents should be stored in polypropylene bottles.

8.2 *Purity of Water*—Unless otherwise indicated, water means reagent water as defined in Specification D1193, Type III.

8.3 *Reagent Blanks*—Reagent blanks should be analyzed to determine their contribution to the sample result.

8.4 *Neodymium Chloride Stock Solution (10 mg Nd/mL)*—Heat 25 mL of 12*M* hydrochloric acid and 1.17 g of neodymium oxide on a hotplate until the neodymium oxide is in solution. Cool the solution and dilute to 100 mL with water.

8.5 *Neodymium Chloride Carrier Solution (0.5 mg Nd/mL)*—Dilute 5 mL of the 10 mg Nd/mL neodymium chloride stock solution to 100 mL with water.

8.6 *Carbon Suspension*—Fume ten 47 mm cellulose filters[7] for about 10 min in 10 mL of 18*M* sulfuric acid. Cool the suspension and dilute to 500 mL with water. The carbon suspension is used as a visual aid in identifying the presence of the precipitate.

8.7 *Substrate Solution*—Dilute 1 mL of the 10 mg Nd/mL neodymium chloride and 20 mL of 12*M* hydrochloric acid to 400 mL with water. Add, with swirling, 10 mL of 29*M* hydrofluoric acid and 8 mL of the carbon suspension. Dilute the suspension to 500 mL with water. Each day before use, place the substrate suspension in a sonic bath for 15 min.

8.8 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (12*M* HCl).

8.9 *3M Hydrochloric Acid*—Add 250 mL concentrated hydrochloric acid to water and dilute to 1 L with water.

8.10 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (18*M* H_2SO_4).

8.11 *Hydrofluoric Acid (48 %)*—Concentrated hydrofluoric acid (29*M* HF). **Warning—**Severe burns can result from exposure of skin to concentrated hydrofluoric acid.

8.12 *Neodymium Oxide (Nd_2O_3).*

8.13 *80 % Ethanol.*

8.14 *20 % Titanium Trichloride*—Available as a 20 % solution of titanium trichloride from commercial suppliers.

8.15 *Sodium Sulfate Solution*—Dissolve 52 g of anhydrous sodium sulfate in 500 mL of 18*M* sulfuric acid.

8.16 *Safranine-0 Solution, 0.1 %*—Dissolve 0.1 g of safranine-0 in 100 mL of water.

9. Hazards

9.1 **Warning**—Adequate laboratory facilities, such as fume hoods and controlled ventilation, along with safe techniques must be used in this procedure. Extreme care should be exercised in using hydrofluoric and other hot, concentrated acids. Use of rubber gloves is recommended.

9.2 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Familiarization and compliance with the Safety Data Sheet is essential.

[5] The sole source of supply for filter media specifically evaluated for alpha spectrometry coprecipitation (RF-100-25PP01) is Eichrom Technologies, LLC, Lisle, IL. The described vacuum funnel is available from Pall Life Sciences, Ann Arbor, MI, catalog numbers 4203 or 4204 as needed. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[7] Ga-6 Metricel or equivalent has been found suitable for this purpose.

10. Sample Preparation

10.1 Add 2 mL of sodium sulfate solution to the actinide fraction and evaporate to complete dryness in a glass beaker. Cool to room temperature and add 10 mL of 3*M* HCl. Cover the beaker with a watch glass, bring to a boil, and keep at a boiling temperature for 5 min.

10.2 Transfer the solution to a capped 50 mL plastic centrifuge tube using about 2 mL of 3*M* HCl as a rinse. For uranium, follow procedure described in 10.6 – 10.8.

10.3 Add 100 µL of the 0.5 mg/mL Nd carrier solution to the tube. Gently shake the capped tube to mix the solution.

10.4 Add 5 mL of 48 % HF to the solution in the tube and mix well by gently swirling the tube. Let stand at least 5 min.

10.5 Proceed with mounting procedure (Section 11).

10.6 Add 1 drop of 0.1 % safranine-0 and 2 drops titanium trichloride to the uranium solution. Uranium reduction is indicated by a change from a purple or blue to an almost colorless solution. If this color change does not occur or persist, add another drop or two of titanium trichloride.

10.7 Add 100 µL of the 0.5 mg/mL Nd carrier solution to the uranium solution. Gently swirl the tube to mix the solution.

10.8 Add 5 mL of 48 % HF to the uranium solution and mix well by gently swirling the tube. Let stand at least 5 min. A reappearance of color at this step may indicate incomplete uranium reduction and require the addition of more titanium trichloride and additional neodymium chloride carrier solution.

10.9 Proceed with mounting procedure (Section 11).

11. Mounting Procedure

11.1 Mount a 25 mm membrane filter on a stainless steel support in a polysulfone twist-lock funnel.

11.2 With vacuum applied, draw about 2 mL of 80 % ethanol through the filter.

11.3 As the filter becomes dry, add the following solutions, in order, to the center of the filter:

11.3.1 Five mL of the substrate solution which has been freshly treated for 15 min in a sonic bath,

11.3.2 The vigorously stirred sample from a capped tube,

11.3.3 Five mL of 3M HCl is used to rinse the sample container,

11.3.4 Five mL of water is used to rinse the sample container, and

11.3.5 Two mL of 80 % ethanol is used to rinse the filter.

11.4 Dry the filter for 5 min under an infra-red heat lamp at a distance of 30 cm to 40 cm. Excess heating in drying will distort the filter.

11.5 Apply a 2.54 cm wide double-sided tape[8] to one side of a clean, 2.54 cm diameter, stainless steel disk. Trim the tape flush with the edge of the disk using a blade or knife. Center the dried filter on the taped side of the disk. Attach the filter to the tape by gently pressing the edge of the filter in several places with the tip of a forceps or tweezers.

11.6 Submit the sample for alpha spectrometry.

12. Precision and Bias

12.1 This practice addresses an intermediate step in an overall separation and measurement scheme and does not produce a measurement. Hence, a statement of precision and bias is not meaningful.

13. Keywords

13.1 actinides; alpha particle; alpha spectrometry; cerium fluoride; energy resolution; neodymium fluoride

[8] Scotch 665 has been found suitable for this purpose.

REFERENCES

(1) Sill, C. W., "Precipitation of Actinides as Fluorides or Hydroxides for High-Resolution Alpha Spectrometry," *Nuclear and Chemical Waste Management*, Vol 7, 1987, pp. 201–215.

(2) Hindman, F. D., "Actinide Separations for α Spectrometry Using Neodymium Fluoride Coprecipitation," *Analytical Chemistry*, Vol 58, 1986, pp. 1238–1241.

(3) Rao, R. R., and Cooper, E. L., "Separation of Low Levels of Actinides by Selective Oxidation / Reduction and Co-precipitation with Neodymium Fluoride," *Journal of Radioanalytical and Nuclear Chemistry, Articles*, Vol 197, No. 1, 1995, pp. 133–148.

(4) Kaye, J. H., Strebin, R. S., and Orr, R. D., "Rapid, Quantitative Analysis of Americium, Curium, and Plutonium Isotopes in Hanford Samples Using Extraction Chromatography and Precipitation Platin," *Journal of Radioanalytical and Nuclear Chemistry, Articles*, Vol 194, No. 1, 1995, pp. 191–196.

(5) Nilsson, H., Rameback, H., and Skalberg, M., "An Improved Method for α-Source Preparation Using Neodymium Fluoride Coprecipitation," *Nuclear Instruments and Methods in Physics Research A*, Vol 462, 2001, pp. 397–404.

(6) Lieberman, R., and Moghissi, A. A., "Coprecipitation Technique for Alpha Spectroscopic Determination of Uranium, Thorium, and Plutonium," *Health Physics*, Vol 15, 1968, pp. 359–362.

(7) Maxwell, S. L., Culligan, B. K., and Noyes, G. W., "Rapid Separation Method for ^{237}Np and Pu Isotopes in Large Soil Samples," *Applied Radiation and Isotopes*, Vol 69, 2011, pp. 917–923.