



Designation: C 1163 – 9803

## Standard Test Method Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride<sup>1</sup>

This standard is issued under the fixed designation C 1163; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This ~~test method~~ practice covers the preparation of separated fractions of actinides for alpha spectrometry as an alternate to electrodeposition. It is applicable to any of the actinides that can be dissolved in dilute hydrochloric acid. Examples of applicable

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<sup>1</sup> This ~~test method~~ practice is under the jurisdiction of ASTM Committee C-26 on the Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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samples would be the final elution from an ion exchange separation or the final strip from a solvent extraction separation.<sup>2</sup>

1.2 *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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 8.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>3</sup>

D 3084 Practice for Alpha-Particle Spectrometry of Water<sup>4</sup>

## 3. Summary of Test Method

3.1 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 equal to electrodeposited samples. Samples can be prepared more rapidly than by electrodeposition and have comparable resolution.

## 4. Significance and Use

4.1 The determination of actinides by alpha spectrometry is an essential ~~portion~~ function of many environmental programs. Alpha spectrometry allows the identification and quantification of most alpha-emitting actinides. Although numerous separation methods are used, the final sample preparation technique ~~is usually~~ has historically been by electrodeposition. However, electrodeposition may have some drawbacks, such as time required, incompatibility with prior chemistry, thick deposits, and low recoveries. These problems can be minimized using the neodymium fluoride method.

4.2 The sample mounting technique described in this ~~test method~~ 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 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. Also, although the total turn around time for coprecipitation may be less than for electrodeposition, coprecipitation required more time and attention from the analyst.

## 5. Interferences

5.1 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.

## 6. Apparatus

6.1 *Alpha Spectrometry*—A system should be assembled that is capable of 60 to 70 keV resolution on an actual sample prepared by this ~~test method~~, 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 D 3084.

6.2 *Filter*—25-mm ~~Metricel~~ (0.1 ± 0.1 μm pore), polypropylene membrane filter or equivalent.<sup>5</sup>

6.3 *Vacuum Funnel*—Polysulfone twist-lock with stainless steel screen for filter mounting.<sup>5</sup>

6.4 *Ultrasonic Bath*.

## 7. Reagents

7.1 *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.<sup>6</sup> 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.

7.2 *Purity of Water*— Unless otherwise indicated, water means reagent water as defined in Specification D 1193, Type III.

<sup>2</sup> Hindman, F. D., "Actinide Separations for Alpha Spectrometry Using Neodymium Fluoride Coprecipitation," *Analytical Chemistry*, 58, 1986, pp. 1236–1241.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> Available from Gelman Sciences, Ann Arbor, MI.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>6</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

<sup>5</sup> Available from Pall Life Sciences, Ann Arbor, MI, catalog number M5PU025.

<sup>6</sup> Ga-6 Metricel or equivalent has been found suitable for this purpose.

<sup>6</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

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

7.4 *Neodymium Chloride Stock Solution (10 mg Nd/mL)*—Heat 25 mL of 12*N* 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.

7.5 *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.

7.6 *Carbon Suspension*—Fume ten 47-mm cellulose filters<sup>7</sup> 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.

7.7 *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.

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

7.9 *3*N* Hydrochloric Acid*—Add 250 mL concentrated hydrochloric acid to water and dilute to 1 L with water.

7.10 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (18*M* H<sub>2</sub>SO<sub>4</sub>).

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

7.12 *Neodymium Oxide (Nd<sub>2</sub>O<sub>3</sub>)*.

7.13 *80 % Ethanol*.

7.14 *20 % Titanium Trichloride* —Available as a 20 % solution of titanium trichloride from commercial suppliers.

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

7.16 *Safranin-0 Solution, 0.1 %* —Dissolve 0.1 g of safranin-0 in 100 mL of water.

~~7.17 *20 % Hydrofluoric–10 % Perchloric Acid*—Slowly add 200 mL concentrated hydrofluoric acid and 100 mL of perchloric acid to water and dilute to 1 L.~~

## 8. Hazards

8.1 **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. Sample Preparation

9.1 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 *N* HCl. Cover the beaker with a watch glass, bring to a boil, and keep at a boiling temperature for 5 min.

9.2 Transfer the solution to a capped 50-mL plastic centrifuge tube using about 2 mL of 3*N* HCl as a rinse. For uranium, follow procedure described in 9.6-9.8.

9.3 Add 100  $\mu$ L of the 0.5 mg/mL Nd carrier solution to the tube. Gently shake the capped tube to mix the solution.

9.4 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.

9.5 Proceed with mounting procedure (Section 10).

9.6 Add 1 drop of 0.1 % safranin-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.

9.7 Add 100  $\mu$ L of the 0.5 mg/mL Nd carrier solution to the uranium solution. Gently swirl the tube to mix the solution.

9.8 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.

9.9 Proceed with mounting procedure (Section 10).

## 10. Mounting Procedure

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

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

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

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

10.3.2 The vigorously stirred sample from a capped tube,

10.3.3 Five mL of ~~hot 20 % hydrofluoric–10 % perchloric acid~~ 3*N* HCl is used to ~~rinse~~ rinse of the sample container,

10.3.4 Five mL ~~water~~ rinse of the filter first water is used to rinse the sample container, and

<sup>7</sup>Scotch 665

<sup>7</sup>Ga-6 Metrical or equivalent has been found suitable for this purpose.

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

10.4 Dry the filter for 5 min under an infra-red heat lamp at a distance of 12 to 16 in. Excess heating in drying will distort the filter.

10.5 Apply a 2.54 cm wide double-coated cellophane type tape<sup>8</sup> 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. Place the sample in a small, closed container (such as a 5 cm diameter Petri dish) for transport and storage. tweezers.

10.6 Submit the sample for alpha spectrometry.

## 11. Precision and Bias

11.1 This test method 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.

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<sup>8</sup> Scotch 665 has been found suitable for this purpose.

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