



Standard Test Method for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride¹

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1. Scope

1.1 This test method 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 samples would be the final elution from an ion exchange separation or the final strip from a solvent extraction separation.²

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 Water³

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 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 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 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 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, 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.

6.2 *Filter*—25-mm Metricel (0.1 μm pore) membrane filter or equivalent.⁴

6.3 *Vacuum Funnel*—Polysulfone twist-lock with stainless steel screen for filter mounting.⁴

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

¹ This test method is under the jurisdiction of ASTM Committee C-26 on the Nuclear Fuel Cycle.

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

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ Available from Gelman Sciences, Ann Arbor, MI.