



Designation: C 1163 – 03

Standard Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride¹

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 (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 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

D 3084 Practice for Alpha-Particle Spectrometry of Water⁴

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 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 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 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 Spectrometer*—A system should be assembled that is capable of 60 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 D 3084.

6.2 *Filter*—25-mm 0.1 μm pore, polypropylene membrane filter or equivalent.⁵

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

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.

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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 *Annual Book of ASTM Standards*, Vol 11.02.

⁵ Available from Pall Life Sciences, Ann Arbor, MI, catalog number M5PU025.