~~Designation:C1163–03~~ Designation: C 1163 – 08

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

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~~

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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section ~~8.~~ 9.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

C 859 Terminology Relating to Nuclear Materials

D 1193 Specification for Reagent Water

D 3084 Practice for Alpha-Particle Spectrometry of Water

3. ~~Summary of Test Method~~

~~3.1Guidance 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.~~ Terminology

3.1 For definitions of terms in this standard, refer to Terminology C 859.

4. ~~Significance and Use~~

~~4.1The 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.~~

~~4.2The 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.~~ Summary of Test Method

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[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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Current edition approved July 15, 2008. Published August 2008. Originally approved in 1992. Last previous edition approved in 2003 as C 1163 – 03.

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

[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* ~~, Vol 11.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.

5. ~~Interferences~~

~~5.1Calculation 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.~~ Significance and Use

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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 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 requires more time and attention from the analyst.

6. Interferences

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7. Apparatus

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7.1 *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~~7.2 *Filter*—25-mm 0.1 µm pore, polypropylene membrane filter or equivalent.

~~6.3~~[4]

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

~~6.4~~7.4 *Ultrasonic Bath*.

~~7.~~

8. Reagents

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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.[5] 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~~

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

~~7.3~~

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

~~7.4~~

8.4 *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~~

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.

~~7.6~~

~~[4] Annual Book of ASTM Standards, Vol 11.02.~~

[4] Available from Pall Life Sciences, Ann Arbor, MI, catalog number M5PU025.

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

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.