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Standard Guide for the Determination of Technetium-99 in Soil[1]

This standard is issued under the fixed designation C 1387; 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 ~~document~~guide is intended to serve as a reference for laboratories wishing to perform Tc-99 analyses in soil. Several options are given for selection of a tracer and for the method of extracting the Tc from the soil matrix. Separation of Tc from the sample matrix is performed using an extraction chromatography resin. Options are then given for the determination of the Tc-99 activity in the original sample. It is up to the user to determine which options are appropriate for use, and to generate acceptance data to support the chosen procedure.

1.2 Due to the various extraction methods available, various tracers used, variable detection methods used, and lack of certified reference materials for Tc-99 in soil, there is insufficient data to support a single method written as a standard method.

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

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 859 Terminology Relating to Nuclear Materials

C~~998 Sampling Surface Soil for Radionuclides~~ 998 Practice for Sampling Surface Soil for Radionuclides

C 999 Practice for Soil Sample Preparation for the Determination of Radionuclides

D~~1193 Standard Specification for Reagent Water~~ 1193 Specification for Reagent Water

E 11 Specification for ~~Wire-cloth~~ Wire Cloth and Sieves for Testing Purposes

3. Terminology

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

4. Summary of Guide

~~3.1There~~4.1 There are no stable isotopes of technetium. Technetium-99 is produced by the fission of uranium and plutonium, and has been released to the environment via nuclear weapons testing and nuclear materials processing. In an oxidizing environment, it exists as the very mobile pertechnetate ion, TcO_4^-. Technetium-99 is a long-lived (half-life 213,000 years), weak beta (beta max of 293 keV) emitting radioisotope.

~~3.2For the analysis of Tc-99 in soil, a tracer is added to the sample matrix, or spiked duplicate samples are prepared, and then the Tc is extracted from the soil matrix by one of several methods, including acid leaching or one of various fusion methods. The resulting solution is passed through an extraction chromatography column. Technetium is known to be retained by the extraction chromatography material while most other elements pass through the column. The column is washed with dilute acid to remove any remaining interferents. The resin may then be counted directly by adding it to a liquid scintillation cocktail and counting by liquid scintillation spectrometry, or the Tc may be eluted from the resin for alternative counting or mass spectrometric techniques.~~

~~**4.Significance and Use**~~

~~4.1This guide offers several options for the determination of Tc-99 in soil samples. Sample sizes of up to 200 g are possible, depending on the method chosen to extract Tc from the soil matrix. It is up to the user to determine if it is appropriate for the intended use of the final data.~~

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

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[2] 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 12.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.

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5. ~~Interferences~~

~~5.1Any radionuclide not completely removed by the extraction chromatography column that has a beta decay energy similar to or higher than Tc-99 will interfere when counting techniques are used for quantification of the Tc-99 activity.~~

~~5.2Any elements with a mass-to-charge ratio (m/z) of 99 (that is, naturally occurring isotope of Ru-99, or other artificially produced elements of sufficient half-life with similar m/z) can interfere when using mass spectrometry for quantification of the Tc-99 activity. Any element with the same m/z as the isotope used as an isotope dilution tracer or internal standard will cause a bias in the yield correction. Corrections should be included in the mass spectrometry data reduction for known interferences.~~

~~5.3Additional interferences may be encountered, depending on the tracer and measurement technique chosen. It is up to the user to determine and correct for any additional interferences.~~ Significance and Use

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7. Apparatus

~~6.1~~

7.1 *Apparatus for the Extraction of Tc from Sample Matrix*:

~~6.1.1See~~7.1.1 See the individual extraction method descriptions to compile a list of the equipment needed for the chosen extraction method.

~~6.2~~7.2 *Apparatus for the Purification of Tc from the Soil Extract*:

~~6.2.1~~

7.2.1 *Extraction column*—with a bed volume of several milliliters for the extraction chromatography resin.

~~6.2.2~~[3]

7.2.2 *Column extension funnels*—that can be added to the extraction column such that a few hundred milliliters of solution can be added to the column at one time.

~~6.2.3~~

7.2.3 *Column rack*— ~~to hold~~ holds columns such that several extractions can be performed simultaneously.

~~6.3~~7.3 *Apparatus for the Quantification of Tc-99*:

~~6.3.1See~~7.3.1 See the individual detection method descriptions to compile a list of the equipment needed for the chosen detection method.

~~7.~~8. Reagents

~~7.1~~

8.1 *Purity of Reagents*—All chemicals should, at a minimum, be of reagent grade and should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] High Purity reagents are suggested if mass spectrometry is chosen as the detection method. Other grades of reagents may be used provided it is first determined that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

~~7.2~~

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, as defined by Type I of Specification D 1193.

~~[3] Annual Book of ASTM Standards, Vol 11.01.~~

[3] Prepacked columns from Eichrom Technologies, LLC, (Darien, IL) or BioRad (Richmond, CA) poly prep columns have been found satisfactory for this purpose.

[4] ~~*Annual Book of ASTM Standards*, Vol 14.02.~~Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, Washington, D. C. 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. Pharmaceutical Convention (USPC), Rockville, MD.

~~7.3~~

8.3 *Tracer*:

~~7.3.1~~

8.3.1 *Isotope Dilution Yield Determination* :

~~7.3.1.1~~

8.3.1.1 *Radiometric Yield Determination*— Tc-95m[5] or Tc-99m[6] have been used to monitor the chemical yield of the extraction and purification of Tc-99 prior to quantification. [Example: Add 10 nCi of Tc-99m as a yield tracer when determining yield by gamma spectrometry.]

~~7.3.1.2~~

8.3.1.2 *Mass Spectrometric Yield Determination*—Tc-97 may be produced in a nuclear reactor in very limited quantities to be used as an isotope dilution tracer for the mass spectrometric determination of Tc-99 **(1)**.[7] [Example: Add 1 ng of Tc-97 as a yield tracer for mass spectrometry.]

~~7.3.2~~

8.3.2 *Duplicate Sample Analysis to Monitor Chemical Yield*:

~~7~~8.3.2.1 Duplicate samples may be analyzed, one spiked with a known amount of Tc-99 and one unspiked. The chemical recovery of the spiked sample is then used to correct the unspiked sample to obtain the original sample activity.

~~7.4~~8.4 *Reagents for the Extraction of Tc-99 from Sample Matrix*:

~~7.4.1 See~~8.4.1 See the individual extraction method descriptions to compile a list of the reagents needed for the chosen extraction method.

~~7.5~~8.5 *Reagents for the Purification of Tc from the Sample Matrix*:

~~7.5.1~~

8.5.1 *Extraction Chromatrography Resin*— TEVA Resin.

~~7.5.2~~[8]

8.5.2 *Prefilter Resin*—a nonionic acrylic ester polymer resin used to remove residual organic matter prior to the extraction chromatography resin column.

~~7.5.3~~[9]

8.5.3 *Hydrogen Peroxide*—30 %.

~~7.5.4~~

8.5.4 *Nitric Acid*—(HNO_3) concentrated, specific gravity 1.42.

~~7.5.5~~

8.5.5 *1M Nitric Acid*—Add 63 mL of high purity HNO_3 to 900 mL of DI water, dilute to a final volume of 1 liter.

~~7.5.6~~

8.5.6 *4M Nitric Acid*—Add 250 mL of high purity HNO_3 to 600 mL of DI water, dilute to a final volume of 1 liter.

~~7.6~~

8.6 *Reagents for the Quantification of Tc-99*:

~~7.6.1 See~~8.6.1 See the individual detection method descriptions to compile a list of the reagents needed for the chosen detection method.

~~8.~~9. Procedure

~~8~~9.1 Collect samples in accordance with Specification C 998.

~~8.2~~

9.2 *Soil or Sediment Preparation* :

~~8~~9.2.1 Oven dry samples at a temperature not to exceed 105°C and homogenized in accordance with Specification C 999.

~~8.2.2~~

9.2.2 *Optional*—Samples may be placed in a muffle oven to decompose organic matter prior to the extraction of Tc. The muffling techniques reported vary significantly **(2-4)** . If desired, ~~it is suggested that~~weigh 5–10 g of the sample be weighed in a

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~~[5] Prepacked columns from EIChroM Technologies (Darien, IL) or BioRad (Richmond, CA) poly prep columns have been found satisfactory for this purpose.~~

[5] Tc-95m may be obtained from Analytics, Inc., Atlanta, GA, or other suitable supplier.

~~[6] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, Washington, D. C. 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. Pharmaceutical Convention (USPC), Rockville, MD.~~

[6] Tc-99m may be obtained from a local medical pharmacy supplier or other suitable supplier.

~~[7] Tc-95m may be obtained from Analytics, Inc., Atlanta, GA, or other suitable supplier.~~

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

~~[8] Tc-99m may be obtained from a local medical pharmacy supplier or other suitable supplier.~~

[8] The sole source of supply of the apparatus known to the committee at this time is TEVA Resin from Eichrom Technologies, LLC. 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, which you may attend.

~~[9] The boldface numbers in parentheses refer to the list of references at the end of this standard.~~

[9] Prefilter columns are available from Eichrom Technologies, LLC or Amberchrom GC-71CD resin has been found satisfactory for this purpose.