Designation: C1387 – 14

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

This standard is issued under the fixed designation C1387; 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 guide is intended to serve as a reference for laboratories wishing to perform ^{99}Tc 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 ^{99}Tc 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 ^{99}Tc 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.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials
C998 Practice for Sampling Surface Soil for Radionuclides
C999 Practice for Soil Sample Preparation for the Determination of Radionuclides
D1193 Specification for Reagent Water
D7168 Test Method for ^{99}Tc in Water by Solid Phase Extraction Disk
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 For definitions of terms in this guide, refer to Terminology C859.

4. Summary of Guide

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 of 2.1 E 5 years), weak beta (maximum beta energy of 293 keV) emitting radioisotope.

4.2 For the analysis of ^{99}Tc 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.

5. Significance and Use

5.1 This guide offers several options for the determination of ^{99}Tc 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.

6. Interferences

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

6.2 Any elements with a mass-to-charge ratio (m/z) of 99 (that is, naturally occurring isotope of ^{99}Ru or other artificially produced elements of sufficient half-life with similar m/z) can interfere when using mass spectrometry for quantification of

[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.
Current edition approved Jan. 1, 2014. Published February 2014. Originally approved in 1998. Last previous edition approved in 2008 as C1387 – 08. DOI: 10.1520/C1387-14.

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

the ^{99}Tc mass 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.

6.3 Additional 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.

7. Apparatus

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

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

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

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

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

7.3 *Apparatus for the Quantification of ^{99}Tc:*

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

8. Reagents

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.

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 D1193.

8.3 *Tracer:*

8.3.1 *Isotope Dilution Yield Determination:*

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

8.3.1.2 *Mass Spectrometric Yield Determination*—^{97}Tc 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 ^{99}Tc (**1**).[7] [Example: Add 1 ng of ^{97}Tc as a yield tracer for mass spectrometry.]

8.3.1.3 *Rhenium as a Mass Spectrometric Yield Monitor*—The chemical behavior of Re and Tc are similar enough on the extraction resin that Re may be used as a tracer for Tc when using ICP-MS as a detection method (**2**).

8.3.2 *Duplicate Sample Analysis to Monitor Chemical Yield:*

8.3.2.1 Duplicate samples may be analyzed, one spiked with a known amount of ^{99}Tc and one unspiked. The chemical recovery of the spiked sample is then used to correct the unspiked sample to obtain the original sample activity. (See Test Method D7168 for an example of this method.)

8.4 *Reagents for the Extraction of ^{99}Tc from Sample Matrix:*

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

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

8.5.1 *Extraction Chromatrography Resin*—TEVA Resin.[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.[9]

8.5.3 *Hydrogen Peroxide*—30 %.

8.5.4 *Nitric Acid*—(16 M HNO_3) concentrated, specific gravity 1.42.

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

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

8.6 *Reagents for the Quantification of ^{99}Tc:*

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

9. Procedure

9.1 Collect samples in accordance with Specification C998.

9.2 *Soil or Sediment Preparation:*

9.2.1 Oven dry samples at a temperature not to exceed 105°C and homogenize in accordance with Specification C999.

9.2.2 *Optional*—Samples may be placed in a muffle oven to decompose organic matter prior to the extraction of Tc. The

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

[4] *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, see *Analar 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.

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

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

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

[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,[1] which you may attend.

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