



Designation: D7168 – 21

Standard Test Method for ⁹⁹Tc in Water by Solid Phase Extraction Disk¹

This standard is issued under the fixed designation D7168; 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 test method describes a solid phase extraction (SPE) procedure to separate ⁹⁹Tc from environmental water (non-process-related or effluent water samples). Technetium-99 beta activity is measured by liquid scintillation spectrometry.

1.2 This test method is designed to measure ⁹⁹Tc in the range of approximately 0.037 Bq/L (1.0 pCi/L) or greater for a one litre sample.

1.3 This test method has been used successfully with tap water. It is the user's responsibility to ensure the validity of this test method for samples larger than 1 L and for waters of untested matrices.

1.4 Technetium-99 alternatively can be determined in water samples using Practice [D8026](#).

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information only and are not considered standard.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

[D1129](#) Terminology Relating to Water

¹ This test method is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.04](#) on Methods of Radiochemical Analysis.

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

[D1193](#) Specification for Reagent Water

[D2777](#) Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

[D3370](#) Practices for Sampling Water from Flowing Process Streams

[D4448](#) Guide for Sampling Ground-Water Monitoring Wells

[D5847](#) Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

[D6001](#) Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization

[D7282](#) Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

[D7902](#) Terminology for Radiochemical Analyses

[D8026](#) Practice for Determination of Tc-99 in Water by Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminologies [D7902](#) and [D1129](#). For terms not defined in this test method or in Terminologies [D1129](#) or [D7902](#), refer to other published glossaries.³

4. Summary of Test Method

4.1 A measured aliquant of sample is transferred to a beaker. Hydrogen peroxide is added to facilitate the formation of the extractable pertechnetate ion. The sample may be heated to oxidize organics if such are suspected to be present. The entire sample is passed through a technetium-selective SPE disk onto which the pertechnetate is adsorbed. The disk is transferred to a liquid scintillation vial, cocktail added, and the contents well mixed. The beta emission rate of the sample is determined by liquid scintillation spectrometry. Chemical yield corrections are determined by the method of standard additions.

4.2 Minor differences in processing between Extraction Chromatographic Resin Discs and PTFE Membrane Discs are addressed in Variations A and B of the test method.

³ American National Standard Glossary of Terms," *Nuclear Science and Technology (ANSI N1.1)*, American National Standards Institute, 1430 Broadway, New York, NY 10018.

5. Significance and Use

5.1 This test method has not been evaluated for all possible matrices. Test method suitability should be determined on specific waters of interest.

6. Interferences

6.1 Suspended materials must be removed by filtration or centrifuging prior to processing the sample. Suspended particulate matter in the sample will be physically trapped, in part or in whole, on or in the SPE extraction material. This may lead to potential inclusion of radionuclide bearing solids or to signal quenching in the liquid scintillation measurement.

6.2 Technetium-99 activity in the sample may overwhelm the signal from the ^{99}Tc spike addition and interfere with accurate determination of chemical yield. If the relative uncertainty of the chemical yield, neglecting the uncertainty of the counting efficiency, exceeds 10 % (that is, $u_r(\bar{e} \cdot Y) > 10\%$), it can be reduced by increasing the count time or by re-preparing the sample with appropriately adjusted aliquant and spike addition levels. See also the discussion of the quantitation range in [Appendix X1](#).

6.3 Organic compounds present in significant quantities in the sample may degrade the extraction performance of the SPE disk or may lead to elevated levels of quench during liquid scintillation analysis. After the addition of hydrogen peroxide, the sample may be heated to destroy trace organic matter in the sample. If organic components are present in the sample which may survive the peroxide digestion, these may be removed with an appropriate organic removal resin or disk (such as Amberchrom⁴ resin or disk) prior to passing the sample through the extraction chromatographic resin disc.

6.4 The disk may retain tritium-labeled compounds. Setting the ^{99}Tc counting window above the maximum energy for the tritium beta particle will eliminate potential tritium interference.

6.5 Elevated levels of nitrates ($>10\,000\text{ mg L}^{-1}$) will interfere with uptake of ^{99}Tc .

6.6 The higher energy region above the maximum energy for ^{99}Tc should be monitored to help identify cases of significant actinide interference.

6.7 Elevated levels of radionuclides present in anionic form such as iodate, iron (III) and antimony may interfere with measurement of technetium and lead to a positive bias in sample results. Significantly elevated levels of actinides (esp. ^{234}Th decay progeny of uranium) when present in the sample may cause a high bias in the reported ^{99}Tc activity. Manufacturer-specific recommendations about interferences should be taken into consideration when determining the applicability of this test method for a given matrix.

7. Apparatus

7.1 *Filtering Apparatus*, 47 mm diameter filter apparatus as recommended by the SPE manufacturer.

7.2 *Liquid Scintillation Spectrometer*, with multiple energy region of interest (ROI) capabilities.

7.3 *Scintillation Vials*, 20 mL vials, low-potassium glass or plastic, exhibiting suitable optical reproducibility so as not to cause erratic results between samples.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided that the reagent is of sufficiently high purity to permit its use without increasing the background of the measurement. Some reagents, even those of high purity, may contain naturally-occurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths and potassium compounds, or artificially produced radionuclides, or combination thereof. Consequently, when such reagents are used in the analysis of low radioactivity samples, the activity of the reagents shall be determined under analytical conditions that are identical to those used for the sample. The activity contributed by the reagents may be considered to be a component of background and applied as a correction when calculating the test sample result. This increased background reduces the sensitivity of the measurement.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification [D1193](#), Type III.

8.3 *Radioactive Purity*—Radioactive purity shall be such that the measured radioactivity of blank samples does not exceed the calculated probable error of the measurement.

8.4 *Technetium-Specific Solid Phase Extraction (SPE) Disks or Membranes*—(Extraction Chromatographic Resin Discs⁵ or PTFE Membrane Disks^{5, 6}).

8.5 *Hydrochloric Acid, 0.5 M*—Add 42 mL concentrated HCl to 400 mL of reagent water. Dilute to 1 L with water.

8.6 *Nitric Acid*, concentrated.

8.7 *Hydrogen Peroxide*, 30 %.

8.8 *Technetium-99*—as pertechnetate in water or dilute base solution, traceable to the SI through a national metrology institute such as National Institute of Standards and Technology (NIST) or UK National Physical Laboratory (NPL)

8.9 *Liquid Scintillation Cocktail*—Commercially prepared LSC cocktail or equivalent.^{5, 7}

⁵ The sole source of supply of the Eichrom TEVA (a trademark of Eichrom Industries) Discs known to the committee at this time is Eichrom Industries, Inc., Lisle, IL. 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.

⁶ 3M CDS Empore (a trademark of 3M Company, St. Paul, MN) Tc Rad Disks have been found satisfactory for this purpose.

⁷ Ultima Gold (a trademark of Perkin Elmer Life and Analytical Sciences, Shelton, CT) LLT has been found satisfactory for this purpose.

⁴ Amberchrom is a trademark of the Dow Chemical Company, Midland, MI.