Designation: D7168 – 16

Standard Test Method for ^{99}Tc in Water by Solid Phase Extraction Disk[1]

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 ^{99}Tc 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 ^{99}Tc 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
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 Closed Conduits
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 Set-up, 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.

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 Disks are addressed in Variations A and B of the test method.

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.

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[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.

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. Samples for which the unspiked sample count rate exceeds 50 % of the spiked sample count rate should be reprepared with an appropriately adjusted aliquant and spike addition levels to minimize contributions to uncertainty in the determination of the chemical yield.

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 Amberchrom[3] 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 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 Discs[4] or PTFE Membrane Disks[4, 5]).

8.5 *Hydrochloric Acid, 0.5M*—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 a national standards body (such as NIST in the U.S.).

8.9 *Liquid Scintillation Cocktail*—Commercially prepared LSC cocktail or equivalent.[4, 6]

9. Hazards

9.1 Use extreme caution when handling all acids. They are extremely corrosive, and skin contact could result in severe burns.

9.2 When diluting concentrated acids, always use safety glasses and protective clothing, and add the acid to the water.

10. Sampling

10.1 Collect a sample in accordance with Practices D3370 or Guides D4448 or D6001.

11. Preservation

11.1 Preservation of samples being analyzed for ^{99}Tc is not required.

[3] Amberchrom is a trademark of the Dow Chemical Company, Midland, MI.

[4] 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.

[5] 3M Empore (a trademark of 3M Company, St. Paul, MN) Tc Rad Disks have been found satisfactory for this purpose.

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

11.1.1 Samples may be preserved by freezing. Allow samples to come to ambient temperature prior to processing.

11.1.2 Samples may be processed if they have been previously preserved to pH less than 2 with nitric or hydrochloric acid. It is noted that high concentrations of nitric acid will adversely affect chemical yield. Although yield corrections will correct for losses, better results may be obtained by using unpreserved samples.

12. Calibration

NOTE 1—See Practice D7282 for additional details on set-up, calibration and quality control of liquid scintillation counters.

12.1 The fractional detection efficiency (ε_{Tc}) is determined as outlined in subsequent steps.

12.1.1 Prepare triplicate working calibration source (WCS) adding at least 20 Bq (~540 pCi) of traceable ^{99}Tc in the pertechnetate form to each of three 100 mL portions of reagent water. Each of the three samples is processed using either test method variation (A or B), as appropriate.

12.1.2 Collect the effluents from the three WCS. Process the composited solution according to the test method to verify that greater than 99 % of the technetium was retained by the SPE material in the calibration runs.

12.1.2.1 If analysis of the combined effluent indicates greater than 1% breakthrough of Tc, the concentration of the WCS activity should be corrected for the losses. If the breakthrough of ^{99}Tc is greater than 5%, the cause for the losses should be identified and new WCS prepared.

12.1.3 An analyte-free aliquant of 100 mL reagent water is also processed as a background subtraction count (BSC).

12.1.4 Count the three vials containing the WCS and the BSC in a liquid scintillation spectrometer for a time sufficient to amass greater than 10 000 counts for each of the WCS.

12.1.5 Calculate the ^{99}Tc Detection Efficiency (ε_{Tc}) for each of the three vials:

$$\varepsilon_{Tc} = \frac{R_g - R_{cb}}{A_c} \quad (1)$$

where:

R_g = gross count rate of the vial in the ^{99}Tc count window in counts per second,
R_{cb} = count rate of the BSC associated with the efficiency measurement in the ^{99}Tc count window in counts per second, and
A_c = activity of standard ^{99}Tc added to each vial (Bq).

12.2 Calculate the average, $\bar{\varepsilon}_{Tc}$, and the relative standard deviation, $s_r(\varepsilon_{Tc})$, for the three efficiency values. The relative standard deviation of these parameters is used to estimate the relative standard uncertainty of the average efficiency, $u_r(\bar{\varepsilon}_{Tc})$, as follows:

$$u_r(\bar{\varepsilon}_{Tc}) = \sqrt{\frac{s_r^2(\varepsilon_{Tc})}{3} + u_r^2(A_c)} \quad (2)$$

where:

$u_r(A_c)$ = relative standard uncertainty of the activity of standard ^{99}Tc added to each vial.

12.3 A background subtraction count (BSC) vial consisting of reagent water shall be processed and analyzed with each batch of samples to determine the background count rate in counts per second (R_b) to be used for the calculation of sample results.

13. Procedure

NOTE 2—To minimize the risk of cross-contamination while ensuring reproducibility between the sample and its spiked duplicate, each aliquant-spiked aliquant pair should be run simultaneously and in parallel, using separate dedicated filtration apparatus.

NOTE 3—The sample aliquant is typically 1 L but depending on the activity present and the required detection limit for the analysis, this may vary from 0.1 to several litres.

NOTE 4—A background subtraction count (BSC) consisting of a vial, cocktail and blank disk is performed with each batch to determine the background count rate to be subtracted from each measurement (R_b in Eq 3). If the BSC is to be reused, the user should determine its stability and shelf-life.

13.1 *Test Method Variation A*—For use with Extraction Chromatographic Resin Discs:

13.1.1 For each sample and OC sample to be processed, transfer duplicate 1 L aliquants of sample to each of two beakers.

13.1.2 Acidify samples to pH 2 with nitric acid, if not done previously.

13.1.3 Add a known quantity (~20 Bq) of a traceable ^{99}Tc solution to the second aliquant of the sample which is labeled as the spiked sample. (See 6.2 for comment on appropriate spiking level.)

13.1.4 Add 10 mL of 30 % H_2O_2 to each sample while stirring.

13.1.5 If the presence of organic interferences is suspected, heat the sample on a hotplate at approximately 80°C for about 1 hour or until any visible reaction has subsided. Allow the sample to cool to ambient temperature before proceeding with subsequent steps.

13.1.6 Using forceps, carefully position a disc on the filter stand. Secure the funnel reservoir over the disc.

13.1.7 Precondition the disc by allowing 25 mL of water to pass through the disc by gravity. Check the filter funnel for leaks.

13.1.8 Add the sample to the funnel reservoir and allow to pass through the disc by gravity flow (nominal flow rate should not exceed ~100 mL/min). If needed, vacuum may be used to maintain adequate flow.

13.1.9 Rinse the disc with 25 mL of 0.5M HCl.

13.1.10 Rinse the disc with 100 mL of water.

13.1.11 Apply vacuum to the filtration apparatus to remove residual liquid from the disc.

13.1.12 Detach the reservoir from the filter apparatus.

13.1.13 Using forceps, remove and carefully roll the disc and transfer to a scintillation vial.

13.1.14 Add 15 mL of liquid scintillation cocktail.

13.1.15 Cap and shake the contents of the vial, to allow the disc to disintegrate. A vortex mixer may be used.

13.1.16 Count the sample test source (STS) in a liquid scintillation spectrometer using an optimized energy window within the range of 20 to 292 keV for a period of time adequate to achieve the required detection limit.

13.2 *Test Method Variation B*—For use with PTFE Extraction Membranes:

13.2.1 For each sample and QC sample to be processed, transfer duplicate 1 L aliquants of sample to each of two beakers.

13.2.2 Add a known quantity (~20 Bq) of traceable ^{99}Tc solution to the second aliquant of the sample which is labeled as the spiked sample. (See 6.2 for comment on appropriate spiking level.)

13.2.3 Add 10 mL of 30 % H_2O_2 to each sample while stirring.

13.2.4 If the presence of organic interferences is suspected, heat the sample on a hotplate at approximately 80°C for approximately 1 hour or until any visible reaction has subsided. Allow the sample to cool to ambient temperature before proceeding with subsequent steps.

13.2.5 Using forceps, carefully position a disk on the filter stand. Secure the funnel reservoir over the disk.

13.2.6 Connect the filtering apparatus to a vacuum source.

13.2.7 Pass the sample through the disk at a nominal flow rate of ~100 mL/min.

13.2.8 Rinse the disk with 25 mL of 0.5M HCl.

13.2.9 Rinse the disk with 100 mL of water.

13.2.10 Detach the reservoir from the filter apparatus.

13.2.11 Using forceps, remove and gently roll the disk and transfer to a scintillation vial.

13.2.12 Add 15 mL of liquid scintillation cocktail.

13.2.13 Cap and shake the contents of the vial to mix well. Inspect vial to ensure that the disk is completely immersed in cocktail.

13.2.14 Count the sample test source (STS) in a liquid scintillation spectrometer using an optimized energy window within the range of 20 to 292 keV for a period of time adequate to achieve the required detection limit.

14. Calculations

14.1 ^{99}Tc Activity Concentration(AC_{Tc}) in Bq/L:

$$AC_{\mathrm{Tc}} = \frac{R_a - R_b}{\bar{\varepsilon}_{\mathrm{Tc}} \times V_a \times Y_{\mathrm{Tc}}} \quad (3)$$

where:

R_a = count rate of sample test source (STS) in counts per second,
R_b = count rate of the background subtraction count (BSC) in counts per second,
$\bar{\varepsilon}_{\mathrm{Tc}}$ = average fractional detection efficiency,
V_a = volume of the sample aliquant in litres, and
Y_{Tc} = fractional chemical yield from Eq 4.

14.2 Fractional Chemical Yield (Y_{Tc}):

$$Y_{\mathrm{Tc}} = \frac{(R_{spk} - R_a)}{A_c \times \bar{\varepsilon}_{\mathrm{Tc}}} \quad (4)$$

where:

R_{spk} = gross count rate of the spiked sample aliquant in counts per second, and
A_c = activity of ^{99}Tc added to the spiked sample aliquant in becquerels (Bq).

14.3 The standard uncertainty of the ^{99}Tc activity concentration of the sample attributable to counting uncertainty, $u_{cC}(AC_{\mathrm{Tc}})$, is given by:

$$u_{cC}(AC_{\mathrm{Tc}}) = \frac{\sqrt{\frac{R_a}{t_a} + \frac{R_b}{t_b}}}{\bar{\varepsilon}_{\mathrm{Tc}} \times V_a \times Y_{\mathrm{Tc}}} \quad (5)$$

where:

t_a = count duration of the STS in seconds, and
t_b = BSC count duration in seconds.

14.4 The relative standard uncertainty of the chemical yield is given by:

$$u_r(Y_{\mathrm{Tc}}) = \sqrt{\frac{(R_{spk} + R_a)/t_a}{(R_{spk} - R_a)^2} + u_r^2(\bar{\varepsilon}_{\mathrm{Tc}}) + u_r^2(A_c) + u_r^2(\ldots)} \quad (6)$$

where:

$u_r(\bar{\varepsilon}_{\mathrm{Tc}})$ = relative standard uncertainty of the average efficiency factor,
$u_r(A_c)$ = relative standard uncertainty of spike added activity, and
$u_{r,\ldots}$ = additional relative standard uncertainty associated with the chemical yield determination that has been determined (for example, replicate reproducibility).

14.5 The combined standard uncertainty of the ^{99}Tc activity concentration, in becquerels per litre, is given by:

$$u_r(AC_{\mathrm{Tc}}) = \quad (7)$$

$$\sqrt{u_{cC}^2(AC_{\mathrm{Tc}}) + AC_{\mathrm{Tc}}^2 \times \left(u_r^2(Y_{\mathrm{Tc}}) - \left| u_r^2\left(\bar{E}_{Tc}\right) \right| + u_r^2(\ldots) \right) + 2 + 2\,\frac{AC_{\mathrm{Tc}}\, R_a/t_a}{V_a\, Y_{\mathrm{Tc}}^2\, \bar{\varepsilon}_{\mathrm{Tc}}^2\, A_c}}$$

where:

$u_r(Y_{\mathrm{Tc}})$ = relative standard uncertainty of the chemical yield from Eq 6,
$u_r(V_a)$ = relative standard uncertainty of the aliquant volume measurement, and
$u_{r,\,(\ldots)}$ = any additional relative standard uncertainty that has been determined or as estimated.

14.6 *Detection Decision*—The decision level or critical level concentration is defined as the minimum measured value (that is, analyte concentration) required to give confidence (95 % in this case) that a positive (nonzero) amount of analyte is present in the material analyzed. L_c is given by:

$$L_c = \frac{1.645 \times \sqrt{R_b \times \left(\frac{t_a + t_b}{t_a \times t_b}\right)}}{\bar{\varepsilon}_{\mathrm{Tc}} \times V_a \times Y_{\mathrm{Tc}}} \quad (8)$$

14.7 The *a priori* Minimum Detectable Concentration (MDC), in becquerels per litre, is given by:

$$MDC = \frac{\frac{2.71}{t_a} + 3.29 \times \sqrt{R_b \times \left(\frac{t_a + t_b}{t_a \times t_b}\right)}}{\bar{\varepsilon}_{\mathrm{Tc}} \times V_a \times Y_{\mathrm{Tc}}} \quad (9)$$

15. Quality Control

NOTE 5—In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test. These requirements are based on the Practice D5847.

15.1 *Chemical Yield:*

15.1.1 As indicated in 13.1.3, a known amount of ^{99}Tc is added to a duplicate aliquant of each field and QC sample. As noted in 8.8 the activity of the ^{99}Tc solution used shall be traceable.

15.1.2 The yield of the ^{99}Tc spike will be calculated for each sample and associated QC sample. This yield, typically expressed in percent, may be reported to the client or data user along with the reported results if required.

15.1.3 The relative standard uncertainty of the yield should be less than 5 % or as directed by the client or data user.

15.2 *Detection Efficiency:*

15.2.1 The calibration for this test method is determined by standard addition. The detection efficiency is only used to determine the ^{99}Tc chemical yield. The efficiency of the detector used for the determination may be determined in advance as long as the continued response of each detector used is verified daily or prior to use.

15.3 *Initial Demonstration of Laboratory Capability:*

15.3.1 If the laboratory or analyst has not previously performed this test method, a precision and bias study must be performed to demonstrate laboratory capability.

15.3.2 Analyze seven replicates of a standard solution prepared from an independent reference material containing ^{99}Tc activities sufficient to minimize the relative standard counting uncertainty to less than 1 %. The matrix used for the demonstration should represent a water sample typical for which the test method will be used, (for example, a surface water). The total dissolved solids of the matrix should approximate the levels expected in normal use. In addition uranium should be included in the matrix because ^{234}Th may interfere in the determination of ^{99}Tc. The uranium should be included at a level of approximately ten times the *a priori* MDC of the analysis.

15.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of precision and mean bias of 10 % and ±10 %, respectively, based on a review of the collaborative study data (see Section 16). Practice D5847 should be consulted on the manner by which precision and mean bias are determined from the initial demonstration study. The study should be repeated until precision and bias meet the given limits.

15.3.4 Analyze three replicates of a blank solution matrix. The matrix used for the demonstration should represent a water sample typical for which the test method will be used (for example, a surface water). The total dissolved solids of the matrix should approximate that which may be encountered in normal use. In addition uranium should be included in the matrix because ^{234}Th may interfere in the determination of ^{99}Tc. The uranium should be included at a level of approximately ten times the a priori MDC of the analysis.

15.3.5 Calculate the ^{99}Tc activity for each of these three blank solutions. The study should be repeated until the ^{99}Tc result of each of the three blank solutions is below one-half the associated MDC.

15.4 *Laboratory Control Sample (LCS):*

15.4.1 To ensure that the test method is in control, analyze an LCS with each batch of no more than 20 samples. The activity added to reagent water should be appropriate for the type of samples analyzed and should produce results of sufficient precision to ensure meaningful assessment of accuracy. The LCS must be taken through all the steps of the analytical method including sample preservation and pretreatment. The result obtained for the LCS shall fall within the limit of ±25 % of the expected value.

15.4.2 If the result is not within these limits reporting of the results is halted until the problem is resolved. An indication of the occurrence should accompany the reported results.

TABLE 1 Observed Bias and Precision for ^{99}Tc

Variation A	YP1		YP2		YP3	
	A1	A2	A3	A4	A5	A6
Number of Retained Values	9	10	10	10	9	8
Average True Concentration[A]	0.3948	0.4811	5.892	5.863	19.71	17.86
Average Measured Concentration	0.3966	0.4804	5.851	5.780	19.91	17.99
Relative Bias	0.5 %	−0.1 %	−0.7 %	−1.4 %	1.0 %	0.7 %
Overall Standard Deviation[B]	0.039	0.039	0.309	0.194	0.546	0.485
Overall Relative Standard Deviation	9.9 %	8.1 %	5.3 %	3.4 %	2.7 %	2.7 %
Number of Retained Pairs	9		10		8	
Single Standard Deviation (S_o)[C]	0.021		0.270		0.533	
Analyst Relative Standard Deviation	4.7 %		4.6 %		2.8 %	
Variation B	YP1		YP2		YP3	
	B1	B2	B3	B4	B5	B6
Number of Retained Values	8	8	8	9	7	8
Average True Concentration[A]	0.3996	0.4801	5.868	5.898	19.67	17.83
Average Measured Concentration	0.3985	0.4636	5.489	5.922	19.90	17.12
Relative Bias	−0.3 %	−3.4 %	−6.5 %	0.4 %	1.2 %	−4.0 %
Overall Standard Deviation[B]	0.014	0.036	0.368	0.397	2.560	1.325
Overall Relative Standard Deviation	3.6 %	7.7 %	6.7 %	6.7 %	12.9 %	7.7 %
Number of Retained Pairs	8		8		7	
Single Standard Deviation (S_o)[C]	0.027		0.470		2.385	
Analyst Relative Standard Deviation	6.2 %		8.0 %		12.7 %	

[A] Known concentration for each lab differs by <1 % from average true concentration.
[B] As calculated by mean square residual from linear least squares regression of known concentration on the measured concentration.
[C] As calculated by mean square residual from mixed model linear least squares on measured concentration with linear spike and random lab effects.

15.5 *Method Blank (Blank):*

15.5.1 Analyze a reagent water test blank with each batch of no more than 20 samples. The concentration of analytes found in the blank should be less than the critical level concentration.

15.5.2 If the result is not within these limits, reporting of the results is halted until the problem is investigated and resolved. An indication of the occurrence should accompany the reported results.

15.6 *Matrix Spike (MS):*

15.6.1 The performance of a matrix spike analysis for quality control purposes is not required as each sample is spiked to generate chemical yield corrections. The chemical yield will indicate problems with interferences in a specific sample matrix. Section 15.1 addresses the use of the tracer chemical yield as a measure of result quality.

15.7 *Duplicate:*

15.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch of no more than 20 samples. Calculate the statistical agreement between the two results to ensure they agree at the 99.7 % confidence level (DER ≤ 3.0). This calculation is performed using the determined combined standard uncertainty associated with each result.

15.7.2 In those cases where there is insufficient sample volume to allow performance of a duplicate sample analysis, a duplicate LCS shall be performed.

15.7.3 If the result is not within these limits reporting of the results is halted until the problem is resolved. An indication of the occurrence should accompany the reported results.

15.8 *Independent Reference Material (IRM):*

15.8.1 In order to verify the quantitative value produced by the test method, analyze an IRM submitted on at least a single-blind basis (if practical) to the laboratory at least once per quarter. The concentration of analyte in the traceable reference material should be appropriate to the typical purpose for which the test method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

16. Precision and Bias[7, 8]

16.1 A collaborative study of this test method was conducted for ^{99}Tc. Twelve laboratories participated by analyzing a series of samples that were spiked with ^{99}Tc. The samples were drinking water acidified to pH 2 with HNO_3 with natural uranium added (a known interference) at a nominal concentration of 10 µg/L (ppb). Both variations of this test method were studied. It is the user's responsibility to ensure the validity of this test method for water of untested matrices.

16.2 Each variation tested used a series of blind samples consisting of three sets of "Youden Pairs" (YP) spiked at three levels with mean concentration averaging approximately 0.44, 5.86, and 18.7 Bq L^{-1} (11.8, 158, 504 pCi L^{-1}). The test samples were prepared individually by gravimetric dilution with the levels shown in Table 1 as target values. The concentrations for results evaluated were precisely known and deviated from one another by less than 1 %. Since values used for the evaluation were not identical as envisioned by Practice D2777, single operator and overall standard deviations were estimated as described in the footnote to Table 1. Outlier results were rejected in accordance with the statistical tests outlined in Practice D2777.

16.3 The collaborative study of this test method resulted in the observed bias and precision values presented in Table 1.

17. Keywords

17.1 disc; disk; liquid scintillation counting; radioactivity; radiochemistry; solid phase extraction; technetium; water

[7] Special exemption from this requirement of Practice D2777 was granted by ASTM Committee D19 upon recommendation from the Results Advisor and Technical Operations Section of the Executive Subcommittee on 15 January, 2004.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1184. Contact ASTM Customer Service at service@astm.org.

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