Designation: D7168 – 21

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

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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.

[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, $u_r(\bar{\varepsilon} \cdot Y) > 10\,\%$), 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 Amberchrom[4] 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[5] 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]

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

[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.

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.

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) 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 WCSs. 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 WCSs 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) for each of the three vials:

$$\varepsilon = \frac{R_{G} - R_{CB}}{A_{C}} \quad (1)$$

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 and the standard deviation, $s(\varepsilon)$, for the three efficiency values. The standard deviation is used to estimate the relative standard uncertainty of the average efficiency, $u_r(\bar{\varepsilon})$, as follows:

$$u_r(\bar{\varepsilon}) = \sqrt{\frac{s^2(\varepsilon)\ /\ \bar{\varepsilon}^2}{3} + u_r^2(A_C)} \quad (2)$$

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 QC 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 0.5 M 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 0.5 M 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 measurement quality objectives (such as a required detection limit or quantitation limit).

14. Calculations

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

$$AC = \frac{R_A - R_B}{\bar{\varepsilon} \cdot V_A \cdot Y} \quad (3)$$

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,
$\bar{\varepsilon}$ = average fractional detection efficiency,
V_A = volume of the sample aliquant in litres, and
Y = fractional chemical yield from Eq 4.

14.2 Fractional Chemical Yield (Y):

$$Y = \frac{R_S - R_A}{\bar{\varepsilon} \cdot A_S} \quad (4)$$

where:

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

NOTE 5—Eq 4 above and the uncertainty equations below assume the volumes of the spiked and unspiked sample aliquants are the same.

14.3 The component of the combined standard uncertainty of the ^{99}Tc activity concentration due to counting statistics, $u_{cC}(AC)$, is given by:

$$u_{cC}(AC) = \frac{1}{\bar{\varepsilon} \cdot V_A \cdot Y}\left[\left(1 + \frac{AC \cdot V_A}{A_S}\right)^2 \frac{R_A}{t_A} + \frac{R_B}{t_B} + \left(\frac{AC \cdot V_A}{A_S}\right)^2 \frac{R_S}{t_A}\right]^{1/2} \quad (5)$$

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, neglecting the uncertainty of the efficiency, $\bar{\varepsilon}$, is given by:

$$u_r(\bar{\varepsilon} \cdot Y) = \sqrt{\frac{\frac{R_S + R_A}{t_A} + 2(R_A - R_B)^2 (u_r^2(V_A) + u_r^2(\ldots))}{(R_S - R_A)^2} + u_r^2(A_S)} \quad (6)$$

Then the total combined standard uncertainty of the yield is given by:

$$u_c(Y) = Y \cdot \sqrt{u_r^2(\bar{\varepsilon} \cdot Y) + u_r^2(\bar{\varepsilon})} \quad (7)$$

where:

$u_r(V_A)$ = relative standard uncertainty of the sample (or spiked sample) aliquant volume,
$u_r(\bar{\varepsilon})$ = relative standard uncertainty of the average efficiency factor,
$u_r(A_S)$ = relative standard uncertainty of spike added activity, and
$u_r(\ldots)$ = additional relative standard uncertainty associated with the preparation and counting of each sample aliquant (recommended 0.01 by default – see 14.4.1).

14.4.1 The additional uncertainty, $u_r(\ldots)$, accounts for the possible variability of the true yield between the spiked and unspiked aliquants as well as for any other small differences that might affect the observed count rates but that are not otherwise accounted for.

14.4.2 If the relative standard uncertainty exceeds 10 %, (that is, $u_r(\bar{\varepsilon} \cdot Y) > 10\ \%$), reduce the uncertainty by increasing the count time or re-preparing the sample with appropriately adjusted aliquant and spike addition levels. See also the discussion of the quantitation range in Appendix X1.

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

$$u_c(AC) = \left[u_{cC}^2(AC) + AC^2 u_r^2(A_S) + \left(\frac{AC^4}{A_S^2} + \left(\frac{AC^2}{A_S} + \frac{AC}{V_A} \right)^2 \right) V_A^2 \left(u_r^2(V_A) + u_r^2(\ldots) \right) \right]^{1/2} \quad (8)$$

where:

$u_c(AC)$ = combined standard uncertainty of the ^{99}Tc activity concentration, Bq/L,
$u_r(V_A)$ = relative standard uncertainty of the aliquant volume measurement, and
$u_r(...)$ = any additional relative standard uncertainty associated with the preparation and counting of each sample aliquant (same as in Eq 6).

14.6 *Detection Decision*—The critical level or decision 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. DLC is given by (see Note 6 and Note 7):

NOTE 6—The factor 1.645 in Eq 9 assumes a Type I error rate of 5 %. Factors corresponding to other error rates may be substituted as appropriate for the measurement quality objectives.

NOTE 7—The radicand of Eq 9 is an estimate of the variance of blank results derived using the principles of uncertainty propagation. Other uncertainty components may also be propagated. Alternatively, the variance may be estimated experimentally from a population of measured blank results and the factor 1.645 replaced by an appropriate percentile of a Student's *t*-distribution. Any modification of Eq 9 may require corresponding modifications of Eq 9.

$$DLC = \frac{1.645\sqrt{R_B \cdot \frac{t_A + t_B}{t_A \cdot t_B}}}{\bar{\varepsilon} \cdot V_A \cdot Y} \quad (9)$$

14.7 The *a priori* Minimum Detectable Concentration (MDC), in becquerels per litre, is given by (see Note 8):

NOTE 8—Eq 10 is valid only for Type I and Type II error rates of 5 %. The formulation must be modified if the measurement quality objectives specify different tolerable decision error rates.

$$MDC = \frac{\frac{2.71}{t_A} + 3.29\sqrt{R_B \cdot \frac{t_A + t_B}{t_A \cdot t_B}}}{\bar{\varepsilon} \cdot V_A \cdot Y} \quad (10)$$

14.8 Appendix X1 describes calculation procedures for the lower and upper limits of the quantitation range, including the *a priori* Minimum Quantifiable Concentration (MQC).

15. Quality Control

NOTE 9—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 established 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 keep the relative standard counting uncertainty less than 1 %. The matrix used for the demonstration should represent a typical water sample 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 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 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.