



# Standard Test Method for Analysis of Urine for Technetium-99 by Inductively Coupled Plasma-Mass Spectrometry<sup>1</sup>

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## 1. Scope

1.1 This test method covers the determination of the concentration of technetium-99 in urine using inductively coupled plasma-mass spectrometry (ICP-MS). This test method can be used to support uranium enrichment and reclamation facility bioassay programs.

1.2 The minimum detectable concentration for this test method, using a quadrupole ICP-MS, is approximately 1.0 ng/L (0.62 Bq/L).

## 2. Referenced Documents

### 2.1 ASTM Standards:

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry<sup>2</sup>

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry<sup>2</sup>

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials<sup>2</sup>

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials<sup>2</sup>

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry<sup>2</sup>

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

### 2.2 Other Standards:

ANSI N13.20 Radiological Measurement Quality<sup>4</sup>

Code of Federal Regulations, Title 10, 835.402<sup>5</sup>

HPS N13.30 Performance Criteria for Radiobioassay<sup>6</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *instrument check standard, n*—standard solutions evaluated at given intervals during batch analysis to evaluate instrument stability during analysis.

3.1.2 *internal reference standard, n*—standard solutions, containing an element with similar chemical properties to the analyte of interest, added to each calibration standard, check standard, and sample for the purpose of monitoring and correcting for fluctuations in matrix, instrument drift, nebulizer and sample orifice blockages, and aerosol transport effects.

3.1.3 *isobar, n*—any nuclide that has the same atomic mass number as another atom, but a different atomic number.

### 3.2 Acronyms:

3.2.1 *ICP-MS, n*—inductively coupled plasma-mass spectrometry.

3.2.2 *LOD, n*—limit of detection.

3.2.3 *LCS, n*—laboratory control standard.

3.2.4 *MDC, n*—minimum detectable concentration.

3.2.5 *%RSD, n*—percent relative standard deviation (1 standard deviation/Mean) \* 100.

3.2.6 *% Bias, n*—((mean – true value)/true value) \* 100.

## 4. Summary of Test Method

4.1 A urine sample is digested in the presence of hydrogen peroxide to decompose the organic matrix. The pertechnetate ion is selectively separated from Ruthenium, actinides, alkali, and alkaline earth metals in the sample matrix using anion exchange chromatography. The <sup>99</sup>Tc is eluted with warmed 3.0 M nitric acid. The <sup>99</sup>Tc isotope is analyzed by ICP-MS using <sup>115</sup>In as the internal reference standard.<sup>7</sup> The chemical recovery is determined by the sample, sample spike method. Recoveries obtained from submitted samples over a one-year period averaged 89.9 %.

## 5. Significance and Use

5.1 Code of Federal Regulations, Title 10, 835.402 states that radiological workers who are likely to receive 100 mrem from intakes are required to be monitored for exposure. For the indirect bioassay for radiological workers exposed to nuclear

<sup>1</sup> This test method 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

<sup>5</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

<sup>6</sup> Health Physics Society, "Performance Criteria for Radiobioassay," HPS N13.30, McLean, VA, 1996.

<sup>7</sup> Crain, J., and Gallimore, D., "Inductively Coupled Plasma-Mass Spectrometry of Synthetic Elements: <sup>99</sup>Tc," *Applied Spectroscopy*, Vol 46, 1992, pp. 547-549.

material containing fission products,  $^{99}\text{Tc}$  must be measured in urine samples.

## 6. Interferences

6.1 Elements or complexes having a mass-to-charge ratio ( $m/z$ ) of 99 will interfere with the  $^{99}\text{Tc}$  analysis. Interfering nuclides of mass 99 (molybdenum and ruthenium) are chemically separated using anion exchange chromatography. The presence of an interfering nuclide may be monitored by evaluating the  $m/z$  value of another isotope of the interfering element.

6.2 Alkali and alkaline earth salts can lead to unstable signals at low levels and signal attenuation at high levels. The  $^{99}\text{Tc}$  is chemically separated from the salts using anion exchange chromatography.

## 7. Apparatus

7.1 *Inductively Coupled Plasma-Mass Spectrometer*, computer-controlled, multichannel peristaltic pump, and an auto-sampler.<sup>8</sup>

7.2 *Centrifuge Tube With Cap*, 15-mL, disposable, graduated, that will accommodate the auto-sampler for sample analysis.

7.3 *Erlenmeyer Flasks and Distillation Columns*, appropriately sized.

7.4 *Plastic Cups*, disposable, appropriately sized.

7.5 *Polyethylene Ion Exchange Column* 12-mL, disposable, or suitable size with a 30- $\mu\text{m}$  porosity frit.

7.6 *Polyethylene Ion Exchange Column Funnels*.

7.7 *Centrifuge Cone*, 50 mL, disposable, graduated.

## 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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>9</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high 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 D 1193.

8.3 *Anion Exchange Resin*—100-200 mesh (chloride form).<sup>10</sup>

8.4 *Argon Gas*, purity 99.99 % or better recommended.

8.5 *Calibration Stock Solution*—Prepare a calibration stock solution containing approximately 2000 ng/L of  $^{99}\text{Tc}$  from a

certified traceable NIST or equivalent certified standard.<sup>11</sup>

8.6 *Hydrochloric Acid (sp gr 1.18)*, concentrated, high-purity Baker Ultrex or equivalent, hydrochloric acid (HCl).

8.7 *Hydrochloric Acid (0.5 M)*—Add 41.7 mL of concentrated HCl to 900 mL of water, dilute to a final volume of 1000 mL, and mix.

8.8 *Hydrogen Peroxide (30 %)*, ( $\text{H}_2\text{O}_2$ ).

8.9 *Nitric Acid (sp gr 1.42)*, concentrated, high-purity Baker Ultrex or equivalent, nitric acid ( $\text{HNO}_3$ ).

8.10 *Nitric Acid (0.5 M)*—Add 31.3 mL of concentrated  $\text{HNO}_3$  to 900 mL of water, dilute to a final volume of 1000 mL, and mix.

8.11 *Nitric Acid (2.5 M)*—Add 156 mL of concentrated  $\text{HNO}_3$  to 800 mL of water, dilute to a final volume of 1000 mL, and mix.

8.12 *Nitric Acid (3.0 M)*—Add 188 mL of concentrated  $\text{HNO}_3$  to 700 mL of water, dilute to a final volume of 1000 mL, and mix.

8.13 *Standard Metals Stock Solution*—Prepare or purchase solutions of beryllium, cobalt, indium, lead, and uranium, or equivalent combination of elements to cover the mass range, to be used as a tuning solution, detector and mass calibration standard, and stability check solution.

8.14  *$^{99}\text{Tc}$  Spike Solution*—Prepare a  $^{99}\text{Tc}$  spike solution containing approximately 8000 ng/L of  $^{99}\text{Tc}$  from a certified traceable NIST or equivalent certified standard.<sup>11</sup>

## 9. Hazards

9.1 Since  $^{99}\text{Tc}$  is radioactive, adequate laboratory facilities along with safe handling techniques must be used. A detailed discussion of all safety precautions needed is beyond the scope of this test method. Follow site- and facility-specific radiation protection and chemical hygiene plans.

## 10. Sampling, Test Specimens, and Test Units

10.1 Urine samples are to be refrigerated at 4°C until analysis. Preservatives may be used if deemed necessary to ensure stability.

10.2 All chain of custody requirements described in laboratory-specific operating procedures must be followed.

## 11. Calibration and Standardization<sup>12</sup>

11.1 Follow the instrument manufacturer's operating manual and laboratory-specific operating procedures for initial start-up and optimization of the ICP-MS and the associated computer control system and peripheral equipment.

11.2 Set up the necessary instrument software files for data acquisition, calculation, quality assurance (QA) and quality control (QC) data requirements, archival data storage, analytical report preparation, and report verification.

11.3 The instrument, data acquisition, and reporting parameters shall be determined to meet customer statement of work requirements.

<sup>8</sup> The VG Elemental PlasmaQuad PQ2+ ICP-MS was used to develop this test method.

<sup>9</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. 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. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

<sup>10</sup> AG MP-1 Macroporous Anion Exchange Resin available from Bio-Rad Co., 300 Reggata Blvd., Richmond, CA 94804, has been found to perform satisfactorily. The feasibility of comparable resins for use in this test method should be demonstrated prior to utilization.

<sup>11</sup> The  $^{99}\text{Tc}$  standard solution is available from NIST, Building 245, Room C114, Gaithersburg, MD 20899.

<sup>12</sup> For quality assurance guides for Nuclear Industry Analytical Laboratories see C 1009, C 1068, C 1128, C 1156, C 1210, and C 1297. For a quality assurance guide for radiological measurements see ANSI Standard N13.20.

11.4 Instrument tuning, detector and mass calibration, and stability check functions for the ICP-MS will require a tuning solution that follows the instrument manufacturer's recommendations. Introduce the daily tuning solution and tune the instrument for optimum response on the selected peaks.

11.4.1 A stock tuning solution can be prepared by adding an aliquot of the standard metal stock solution (see 8.13) or solution as specified by the ICP-MS instrument manufacturer to water and 2 parts volume concentrated  $\text{HNO}_3$  per 100 parts water.

11.4.2 The daily tuning, mass calibration, detector calibration, and stability check solutions should be prepared by diluting an aliquot of the stock tuning solution (see 11.4.1) and 2 parts volume concentrated  $\text{HNO}_3$  per 100 parts water. These solutions should be prepared at analyte concentrations suggested by the instrument manufacturer.

11.5 Check the mass calibration and resolution with the daily tuning solution and elements recommended in accordance with the manufacturer's instrument specifications.

11.6 Make necessary adjustments in the instrument controls to ensure that all of the preceding operating parameters (mass calibration, mass resolution, and baseline) are within previously established laboratory limits. Use the appropriate concentrations (see 11.4.2) for each of the calibration functions suggested by the instrument manufacturer.

11.7 Determine the instrument stability before analyzing any samples. The stability is determined by analyzing the daily tuning solution (see 11.4.2) at least 10 times with a relative standard deviation of less than 5 % on the selected peaks.

11.8 If the relative standard deviation for these isotopes during instrument stability testing was greater than 5 %, determine the cause of the instability, correct the problem, and rerun the stability check.

11.9 Prior to the ICP-MS analysis for  $^{99}\text{Tc}$ , the following QC standards, calibration standards, internal standard, and rinse solution are recommended and should be included in the analytical run:

11.9.1 *Rinse Solution*—Add 2 parts volume concentrated  $\text{HNO}_3$  per 100 parts water. Prepare a sufficient quantity to flush the ICP-MS and auto sampler between standards and samples.

11.9.2  *$^{99}\text{Tc}$  Calibration Standards*—A minimum of four calibration standards should be prepared ranging from 2 to 80 ng/L  $^{99}\text{Tc}$  by diluting the calibration stock solution (see 8.14). The calibration standards should be prepared in 2.5 M  $\text{HNO}_3$  (see 8.11) to match the final acid concentration of the prepared samples.

11.9.3 *Calibration Blank*—The calibration blank should be prepared at the same acid concentration as the calibration standards, 2.5 M  $\text{HNO}_3$  (see 8.11).

11.9.4 *Instrument Check Standard*—Prepare in accordance with 11.9.2. Analyze at minimum a low- and high-level standard throughout the analytical run at a minimum frequency of 10 %

11.9.5 *Artificial Urine*<sup>13</sup>—Prepare an artificial urine solution in accordance with the following instructions. Mix 16.0

parts urea, 2.32 parts NaCl, 3.43 parts KCl, 1.10 parts creatinine, 4.31 parts  $\text{Na}_2\text{SO}_4$  (anhydrous), 0.63 part hippuric acid, 1.06 parts  $\text{NH}_4\text{Cl}$ , 0.54 part citric acid, 0.46 part  $\text{MgSO}_4$  (anhydrous), 2.73 parts  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ , 0.63 part  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ , 0.02 part oxalic acid, 0.094 part lactic acid, 0.48 part glucose (or dextrose), 0.071 part  $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ , 0.029 part pepsin, 5.0 parts concentrated  $\text{HNO}_3$ , and 961 parts water. Mix dry chemicals thoroughly before adding liquids. Stir the mixture thoroughly for approximately 2 h using a magnetic stirrer.

11.9.6 *Method Reagent Blank*—An aliquot of artificial urine that is carried through each step of the procedure. The method reagent blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. The method reagent blank is used to determine the test method MDC.

11.9.7 *Laboratory Control Sample (LCS)*—Prepare the control by adding an appropriate aliquot of the calibration stock solution (see 8.14) to an aliquot of artificial urine to give a  $^{99}\text{Tc}$  concentration within the calibration range. The LCS is carried through each step of the procedure. The LCS result is used to determine if the test method performance is within acceptable control limits.

11.9.8 *Internal Reference Standard*—The recommended concentration for the working In internal reference solution is 20 000 ng/L. The recommended concentration of In within each calibration standard and sample is 200 ng/L. To prepare the working solution, add the required volume of In standard solution to approximately 100 mL of water containing 2 parts concentrated  $\text{HNO}_3$ .

## 12. Procedure

### 12.1 Sample Preparation:

12.1.1 Transfer 30 mL of the urine sample into an Erlenmeyer flask, and add 2 mL of  $\text{H}_2\text{O}_2$ . Add an additional 1 mL if the sample appears dark. Attach a distillation column to the flask.

12.1.2 Transfer 30 mL of the same urine sample into an additional Erlenmeyer flask, and add the same amount of  $\text{H}_2\text{O}_2$  as was added to the unspiked sample (see 12.1.1). Add an appropriate amount (approximately 8000 ng/L  $^{99}\text{Tc}$ ) of a known concentration of  $^{99}\text{Tc}$  spike solution (see 8.14). Attach a distillation column to the flask.

12.1.3 Place the Erlenmeyer flask on a hot plate set at a surface temperature between 150 and 200°C for approximately 1 h or until the sample becomes nearly colorless. Allow the samples to cool to room temperature.

12.1.4 Thoroughly rinse the condensate from the distillation columns attached to the flask with water. Remove the columns and add 1.5 mL of concentrated  $\text{HNO}_3$  to each flask. Add water to a volume of approximately 50 mL.

12.1.5 To assist the slightly acid soluble precipitants to go into solution, place the Erlenmeyer flask back on a hot plate set at a surface temperature between 150 and 200°C for approximately 15 min. Allow the sample to cool to room temperature.

### 12.2 Column Preparation:

12.2.1 Prepare the anion exchange resin by equilibrating with water overnight.

12.2.2 Place one polyethylene disposable column for each sample on a column holder rack.

<sup>13</sup> Robinson, A. V., Fisher, D. R., and Hadley, R. T., "Technical Evaluation of Draft ANSI Standard N13.30, Performance Criteria for Radioassay," Report No. PNL-5107 (draft), Pacific Northwest Laboratory, Richland, WA, 1984.

12.2.3 Place an appropriately sized disposable plastic cup under the column to collect the column waste.

12.2.4 Pour approximately 0.8 mL of prepared resin into the column.

12.2.5 Attach a polyethylene disposable funnel to the ion exchange column.

12.2.6 Condition the column with 30 mL of 0.5 M HNO<sub>3</sub>.

12.2.7 Discard the waste in accordance with laboratory-specific operating procedures.

12.3 *Ion Exchange Separation of <sup>99</sup>Tc:*

12.3.1 Transfer prepared samples and controls (see 12.1.5) to an individual conditioned column. Rinse the beaker with approximately 10 mL of 0.5 M HNO<sub>3</sub> and add to the appropriate sample column.

12.3.2 Allow the sample solution to flow through the column.

12.3.3 Rinse the column with 30 mL of 0.5 M HNO<sub>3</sub> and allow to drain before proceeding to the next step.

12.3.4 Rinse the column with 30 mL of 0.5 M HCl. Once the rinse has eluted through the column, dispose of the collected waste in accordance with the laboratory-specific operating procedures.

12.3.5 Warm an appropriate volume of 3.0 M HNO<sub>3</sub> on a hot plate set at a surface temperature between 150 and 200°C for approximately 30 min, until the solution reaches 95°C.

12.3.6 Place a 50-mL disposable graduated centrifuge cone under each ion exchange column.

12.3.7 Elute the <sup>99</sup>Tc from each column with 25 mL of warmed 3.0 M HNO<sub>3</sub>.

NOTE 1—**Caution:** When adding the warmed acid to each ion exchange column, an automatic pipette is recommended for the acid transfer.

12.3.8 Collect the <sup>99</sup>Tc in the centrifuge cone. Dilute the sample to a final volume of 30 mL with water.

12.3.9 Pour 10 mL of the calibration standards, samples, LCS, and instrument check standards into 15-mL graduated centrifuge cones. Pipette 100 µL of the internal reference standard (see 11.9.8) into each cone. The recommended final internal reference standard concentration is approximately 200 ng/L In per sample.

NOTE 2—Prepare two test method blanks containing the internal standard. Pour 5 mL from one cone into the other to obtain a combined volume of 15 mL. This step is needed to prepare a test method blank of sufficient volume to obtain seven replicate analysis for the MDC calculation (see 13.3.3).

12.3.10 Cap and mix each sample and standard thoroughly.

12.4 *Analysis of <sup>99</sup>Tc:*

12.4.1 Ensure that all instrument set up, calibration and standardization (see Section 11), and required laboratory-specific QC protocol have been followed. A suggested QC sample protocol is given in Appendix X1.

12.4.2 Analyze the standards, prepared samples, and prepared LCS in accordance with the ICP-MS and data system operations described in the site-specific laboratory operating procedures.

## 13. Calculation

13.1 Subtract the calibration blank (see 11.9.3) from the calibration standards (see 11.9.2) and instrument check standards. Subtract the method reagent blank (see 11.9.6) from the samples, sample spikes, and laboratory control samples.

13.2 Apply the appropriate dilution factor for any samples that require additional dilution.

13.3 *Result Calculations:*

13.3.1 Determine the chemical recovery fraction for each sample and control in accordance with the following equation:

$$\text{Recovery} = (Sp - T)/X \quad (1)$$

where:

$Sp$  = measured <sup>99</sup>Tc concentration in the spiked sample, ng/L,

$T$  = measured <sup>99</sup>Tc concentration in the sample, ng/L,

$X$  = known concentration of the <sup>99</sup>Tc spike, ng/L, and

Recovery = sample recovery fraction.

13.3.2 Determine the final result in accordance with the following equation:

$$\text{Final Result} = T/\text{Recovery} \quad (2)$$

13.3.3 Determine the MDC for each sample in accordance with the following equation (see N13.30):<sup>6</sup>

$$\text{MDC} = [4.65(S_b)]/\text{Recovery} \quad (3)$$

where:

$S_b$  = standard deviation obtained from a minimum of seven replicate analyses of the method blank.

13.4 The data package for <sup>99</sup>Tc concentrations should be prepared in accordance with the program statement of work.

NOTE 3—Results may be reported in activity units, such as becquerel per litre, using the specific activity 0.6245 Bq/ng.

## 14. Precision and Bias

14.1 The precision and bias were determined using a NIST traceable <sup>99</sup>Tc standard solution at a level of 6.7 ng/L. The precision and bias were determined from 77 LCSs prepared using an artificial urine matrix, detected in a single laboratory, and analyzed over a period of nine months. Using a low-resolution instrument, the relative standard deviation was found to be 8.2 % and the bias was 0.29 %. Refer to Appendix X2 for additional precision and bias information.

14.2 The bias was determined through an external control program using real urine samples spiked with <sup>99</sup>Tc at levels varying anywhere between 12.4–44.5 ng/L. Over a period of 15 months, the bias was found to be –6.1 % on a total of 57 double-blind samples.

## 15. Keywords

15.1 bioassay; inductively coupled plasma-mass spectrometry; mass; technetium; urine



APPENDIXES

(Nonmandatory Information)

X1. QUALITY ASSURANCE AND QUALITY CONTROL

X1.1 The following quality control information is recommended for the determination of <sup>99</sup>Tc.

X1.2 The instrument should be calibrated using a minimum of four calibration standards and a calibration blank. The calibration correlation coefficient should be equal to or greater than 0.990. In addition to the initial calibration blank, a calibration blank should be analyzed at the end of the analytical run to ensure contamination was not a problem during the analysis.

X1.3 An instrument check standard should be analyzed at a minimum frequency of 10 % throughout the analytical run. The value of the instrument check standard should fall between 80 and 120 % of the true value.

X1.4 Two method blanks should be prepared, mixed, and 14.5 mL poured back into one of the centrifuge cones to ensure

that an adequate method blank volume is present for a minimum of seven repetitive analysis. The standard deviation of the method blank is used to determine the minimum detectable concentration of each sample and control in the batch.

X1.5 A LCS should be analyzed with each batch of sample at a minimum frequency of 10 %.

X1.6 If the QC for the sample batch is not within the established control limits, reanalyze the samples or qualify the results with the appropriate flags, or both.

X1.7 Blind control samples should be submitted by an outside agency in order to determine the laboratory performance capabilities.

X2. METHOD PERFORMANCE COMPARISON OF LIQUID SCINTILLATION COUNTING AND ICP-MS ANALYSIS

X2.1 A method performance study was conducted to compare the LOD (3.29 S<sub>b</sub>) and MDC (4.65 S<sub>b</sub>) values for water, artificial urine, and real urine matrices by liquid scintillation counting and ICP-MS (Refer to Section 13).<sup>14</sup> Fifteen individually prepared method blanks for each matrix were prepared and split into two samples. Of the 15 split sets, one split was analyzed by each test method. The liquid scintillation samples were counted on a Packard 2500TR analyzer with low-level counting capabilities using the internal quench curve correction method. The ICP-MS samples were analyzed on a VG Elemental PlasmaQuad PQ 2+. The final results are given in Table X2.1.

X2.2 Bias and precision for water, artificial urine, and real urine matrices were also evaluated in the comparison study. For each matrix, three different levels between approximately 19 to 57 ng/L <sup>99</sup>Tc, 10 controls per level (n=10), were prepared. Each level was prepared as a single batch by weight. The total uncertainty reported for the <sup>99</sup>Tc standard reference material used (SRM-4288) was 1.62 %. The results of this study are given in Table X2.2. The results listed in Table X2.2 were not corrected for recovery so that a direct comparison, without

TABLE X2.1 Comparison of Liquid Scintillation and ICP-MS Detection Limits


Matrix	LOD, ng/L		MDC, ng/L	
	Liquid Scintillation	ICP-MS	Liquid Scintillation	ICP-MS
Water	4.3	1.6	5.1	2.2
Artificial urine	4.2	0.75	5.4	1.1
Real urine	5.4	0.39	7.0	0.55

TABLE X2.2 Liquid Scintillation and ICP-MS Performance Comparison of <sup>99</sup>Tc analysis in Water, Artificial Urine, and Real Urine Matrices

Matrix	True Value, ng/L	Liquid Scintillation			ICP-MS		
		Mean, ng/L	%RSD	%Bias	Mean, ng/L	%RSD	%Bias
Water	19.0	16.2	7.4	-15	16.9	4.2	-11
Artificial urine	19.0	12.2	13	-36	15.7	5.7	-17
Real urine	18.8	15.4	15	-18	16.3	2.1	-13
Water	38.0	31.6	4.1	-17	33.0	2.8	-13
Artificial urine	38.0	30.9	8.6	-18	34.9	3.5	-8.1
Real urine	37.9	34.2	4.5	-9.7	34.2	4.0	-9.8
Water	57.0	53.1	6.4	-6.8	50.8	5.7	-11
Artificial urine	57.0	50.7	3.6	-11	51.6	2.6	-9.4
Real urine	56.7	50.3	5.5	-11	52.2	4.2	-7.9

<sup>14</sup> Lewis, L. A., and Schweitzer, G. K., "<sup>99</sup>Tc Bioassay: A Direct Comparison of Liquid Scintillation Radiation Detection and ICP-MS Mass Detection of the <sup>99</sup>Tc Isotope," *Applications of Inductively Coupled Plasma-Mass Spectrometry to Radio-nuclide Determinations: Second Volume, ASTM STP 1344*, R. W. Morrow and J. S. Crain, eds., American Society for Testing and Materials, 1998.

interferences, could be made. Since the results reported in Table X2.2 were not corrected for recovery, the reported bias values do not represent the test method bias.

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