Designation: D2460 – 07 (Reapproved 2013)

Standard Test Method for Alpha-Particle-Emitting Isotopes of Radium in Water[1]

This standard is issued under the fixed designation D2460; 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 covers the separation of dissolved radium from water for the purpose of measuring its radioactivity. Although all radium isotopes are separated, the test method is limited to alpha-particle-emitting isotopes by choice of radiation detector. The most important of these radioisotopes are ^{223}Ra, ^{224}Ra, and ^{226}Ra. The lower limit of concentration to which this test method is applicable is 3.7×10^{-2} Bq/L (1 pCi/L).

1.2 This test method may be used for absolute measurements by calibrating with a suitable alpha-emitting radioisotope such as ^{226} Ra, or for relative methods by comparing measurements with each other. Mixtures of radium isotopes may be reported as equivalent ^{226}Ra. Information is also provided from which the relative contributions of radium isotopes may be calculated.

1.3 *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 a specific precautionary statement, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
C859 Terminology Relating to Nuclear Materials
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1943 Test Method for Alpha Particle Radioactivity of 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
D3454 Test Method for Radium-226 in Water
D3648 Practices for the Measurement of Radioactivity
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

3. Terminology

3.1 *Definition:*

3.1.1 For definitions of terms used in this standard, see Terminologies C859 and D1129. For terms not included in these, reference may be made to other published glossaries (**1, 2**).[3]

4. Summary of Test Method

4.1 Radium is collected from the water by coprecipitation with mixed barium and lead sulfates. The barium and lead carriers are added to a solution containing alkaline citrate ion which prevents precipitation until interchange has taken place. Sulfuric acid is then used to precipitate the sulfates, which are purified by nitric acid washes. The precipitate is dissolved in ammoniacal EDTA. The barium and radium sulfates are reprecipitated by the addition of acetic acid, thereby separating them from lead and other radionuclides. The precipitate is dried on a planchet, weighed to determine the chemical yield, and alpha-counted to determine the total disintegration rate of alpha-particle-emitting radium isotopes. This procedure is based upon published ones (**3, 4**).

5. Significance and Use

5.1 Radium is one of the most radiotoxic elements. Its isotope of mass 226 is the most hazardous because of its long half-life. The isotopes 223 and 224, although not as hazardous, are of some concern in appraising the quality of water.

5.2 The alpha-particle-emitting isotopes of radium other than that of mass 226 may be determined by difference if radium-226 is measured separately, such as by Test Method D3454. Note that one finds ^{226}Ra and ^{223}Ra together in variable proportions (**5, 6**), but ^{224}Ra does not normally occur with

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

them. Thus, ^{223}Ra often may be determined by simply subtracting the ^{226}Ra content from the total: and if ^{226}Ra and ^{223}Ra are low, ^{224}Ra may be determined directly. The determination of a single isotope in a mixture is less precise than if it occurred alone.

6. Interferences

6.1 A barium content in the sample exceeding 0.2 mg will bias chemical yield high and lead to falsely low sample results.

6.2 The presence of suspended solids or insoluble precipitates which fail to dissolve during step 12.5 will bias chemical yield high and lead to falsely low sample results.

6.3 The total alpha particle emission rate from the prepared sample changes over time. This will influence the radium detection efficiency of the counting system used. Initially, the total emission rate will increase as the short-lived radon progeny ingrow in the processed sample. After reaching a maximum, the alpha emission rate will decline at the half life of the radium isotope of interest. In samples of pure isotope, maximum emission rate after radium separation is reached after a period of 4 hours for ^{223}Ra, 24 hours for ^{224}Ra, and 28 days for ^{226}Ra. (See Fig. 1.)

6.4 The alpha particle detection efficiency decreases with increasing precipitate mass. Controlling the precipitate mass relative to that used for calibration of the test will minimize the introduction of significant bias into sample results.

6.5 The changing alpha emission rate and self-absorption effects noted in 6.3 and 6.4 can be addressed by reproducing these conditions during the calibration of the instrument. A series of standards analyzed per 11.2 may be used to generate a curve describing efficiencies over a range of precipitate masses and a series of time encompassing the ingrowth curve (~30 days) of ^{222}Rn daughters. (See Fig. 2).

7. Apparatus

7.1 For suitable gas-flow proportional or alpha-scintillation counting equipment, refer to Test Method D1943.

8. Reagents

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.[4] 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 precision, or increasing the bias, of the determination.

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 *Radioactivity Purity of Reagents ,* shall be such that the measured results of blank samples do not exceed the calculated probable error of the measurement or are within the desired precision.

8.4 *Acetic Acid, Glacial* (sp gr 1.05).

8.5 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

8.6 *Ammonium Hydroxide* (7 *M*)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) with 1 volume of water.

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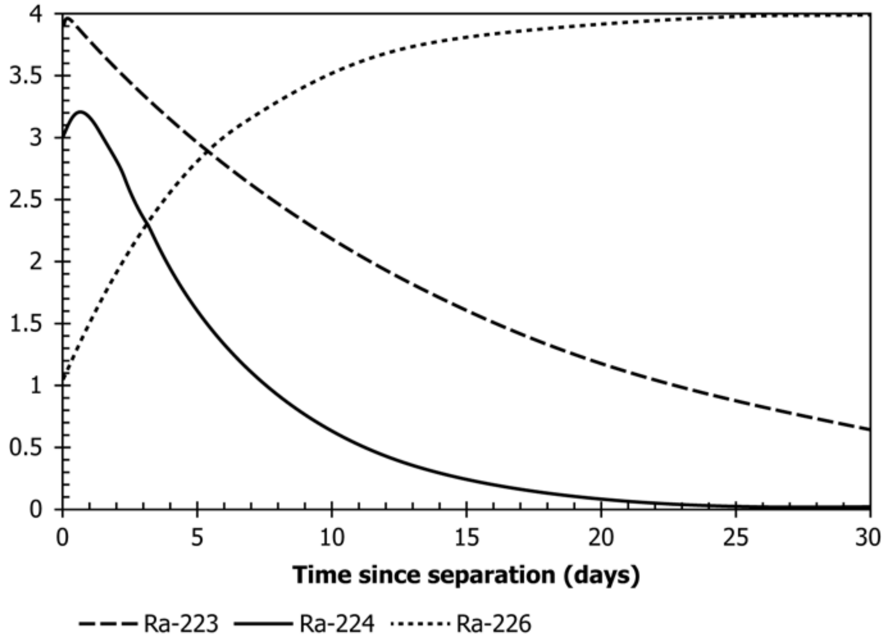
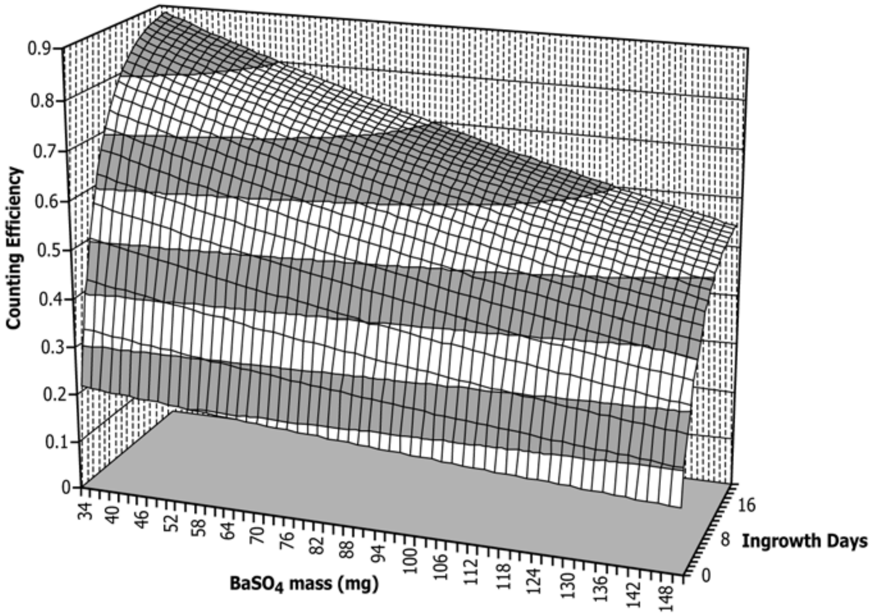
[4] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of Reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDN Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



NOTE 1—Vertical scale is ratio of the total alpha radioactivity at later time, *t*, to radioactivity, A_0, at initial time of separation.

FIG. 1 Growth and Decay of Alpha Activity into Initially Pure Radium Isotopes



Burns, D. C., "Growth and Decay of Alpha Activity into Initially Pure Radium Isotopes," Calibration Plot, Paragon Analytics, Inc., Fort Collins, CO, 2003.

FIG. 2 Typical Alpha Particle Efficiency as Function of Time and Precipitate Mass

8.7 *Barium Nitrate Carrier Solution—Standardized* (10.0 mg Ba^{++}/mL)—Dissolve 1.90 g of barium nitrate ($Ba(NO_3)_2$) in water and dilute to 100 mL.

8.7.1 To perform standardization (in triplicate):

8.7.1.1 Pipette 2.0 mL carrier solution into a centrifuge tube containing 15 mL water.

8.7.1.2 Add 1 mL 18 N H_2SO_4 while stirring and digest precipitate in a water bath for 10 min.

8.7.1.3 Allow to cool. Centrifuge, and decant supernatant.

8.7.1.4 Wash precipitate with 15 mL water. Centrifuge and decant supernatant.

8.7.1.5 Transfer the precipitate to a tared stainless steel planchet with a minimum of water.

8.7.1.6 Dry under infrared lamp, store in desiccator, and weigh as $BaSO_4$.

NOTE 1—0.5884 gram Ba^{++} is equivalent to 1.000 gram $BaSO_4$.

8.8 *Citric Acid Solution* (350 g/L)—Dissolve 350 g of citric acid (anhydrous) in water and dilute to 1 L.

8.9 *Disodium Ethylendiamine Tetraacetate Solution (EDTA)* (93 g/L)—Dissolve 93 g of disodium ethylenediamine tetraacetate dihydrate in water and dilute to 1 L.

8.10 *Lead Nitrate Carrier Solution* (104 mg Pb/mL)—Dissolve 33.2 g of lead nitrate ($Pb(NO_3)_2$) in water and dilute to 200 mL.

8.11 *Methyl Orange Indicator Solution* —Dissolve 1.0 g of methyl orange in water and dilute to 1 L.

8.12 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

8.13 *Sulfuric Acid* (9 *M*)—Cautiously add with stirring 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 1 volume of water.

9. Safety Precautions

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

10. Sampling

10.1 Collect the sample in accordance with Practices D3370, Guide D4448, or Guide D6001, as applicable.

10.2 Sample 1 L, or a smaller volume, provided that it is estimated to contain from 3.7 to 370 Bq (100 to 10 000 pCi) of radium. Add 10 mL of HNO_3/L of sample.

11. Calibration and Standardization

11.1 For absolute counting, the alpha-particle detector must be calibrated to obtain the ratio of count rate to disintegration rate.

TABLE 1 Growth of Alpha Activity into Initially Pure Radium-226

Time, h	Correction, *F*
0	1.0000
1	1.0160
2	1.0362
3	1.0578
4	1.0798
5	1.1017
6	1.1235
24	1.4886
48	1.9043
72	2.2513
96	2.5408
120	2.7823
144	2.9839
192	3.2925
240	3.5073
360	3.8006
480	3.9193
720	3.9867

11.2 Use ^{226}Ra standards traceable to a national standards laboratory (such as NIST or NPL). Analyze two or more portions of such solution, containing known disintegration rates, in accordance with Section 12. After counting, correct the measured activity for chemical yield, and calculate the efficiency, *E* (see Section 13), as the ratio of the observed counting rate to the known disintegration rate.

11.3 The ratio of the net count rate to known ^{226}Ra disintegration rate is a function of precipitate mass and time elapsed between the formation of the final barium sulfate precipitate and counting.

12. Procedure

12.1 Add to a measured volume of sample 5 mL of citric acid solution and make alkaline (pH > 7.0) with 7 *M* NH_4OH. Confirm the alkalinity with pH-indicating paper or strip. Add 2 mL of lead carrier and 1.00 mL of barium carrier, and mix.

12.2 Heat to boiling and add 10 drops of methyl orange pH-indicator solution. With stirring, add 9 *M* H_2SO_4 until the solution becomes pink, then add 5 drops more.

12.3 Digest the precipitate with continued heating for 10 min. Let cool and collect the precipitate in a centrifuge tube. When large volumes are handled, collection will be facilitated by first letting the precipitate settle, and then decanting most of the clear liquid. Centrifuge, then discard the supernatant liquid.

12.4 Wash the precipitate with 10 mL of HNO_3, centrifuge and discard the washings. Repeat this wash of the precipitate.

12.5 Dissolve the precipitate in 10 mL of water, 10 mL of EDTA solution, and 4 mL of 7 *M* NH_4OH. Warm if necessary to effect dissolution.

12.6 Reprecipitate barium sulfate ($BaSO_4$) by the dropwise addition of acetic acid, then add 3 drops more. Record the time. Centrifuge, then discard the supernatant liquid. Add 10 mL of water, mix well, centrifuge, and discard the supernatant liquid.

12.7 Clean, flame, cool, and weigh a stainless steel planchet that fits the alpha-particle counter being used. Transfer the precipitate to the planchet with a minimum of water. Dry, flame, and weigh the precipitate to determine the chemical yield.

12.8 Promptly count the planchet in an appropriate alpha-particle counter, recording the time. Reserve the planchet for additional measurements, if desired (see 13.6).

12.9 Measure the background count rate of the detector by counting an empty, cleaned and flamed planchet for at least as long as the precipitate was counted.

13. Calculation

13.1 Calculate the fractional radium recovery (chemical yield of the carrier) as follows:[5]

TABLE 2 Important Alpha-Particle-Emitting Isotopes of Radium and their Descendants^A

Nuclide		Radiation		Half-Life
Parent	Descendents	Type^B	Energy, MeV^C	
^{226}Ra		α	4.784 (94.5 %) 4.601 (5.6 %)	1.60 × 10^3 years
	^{222}Rn	α	5.490 (99.9 %)	3.83 days
	^{218}Po	α	6.003 (100.0 %)	3.10 min
	^{214}Pb	β (γ)		26.8 min
	^{214}Bi	β (γ)		19.9 min
	^{214}Po	α	7687 (100.0 %)	1.64 × 10^{-4} s
^{224}Ra		α	5.685 (94.9 %) 5.449 (5.1 %)	3.66 days
	^{220}Rn	α	6.288 (99.9 %)	55.6 s
	^{216}Po	α	6.778 (100.0 %)	0.15 s
	^{212}Pb	β (γ)		10.6 h
	^{212}Bi	β (64.1 %) (γ)		1.01 h
		α (35.9 %)	6.090 (9.75 %) 6.051 (25.1 %) Others	
	^{212}Po	α	8.785	0.299 µs
	^{208}Tl	β (γ)		3.05 min
^{223}Ra		α (γ)	5.716 (51.6 %) 5.607 (25.2 %) 5.747 (9.0 %) 5.540 (9.0 %) 5.434 (2.2 %) 5.502 (1.0 %) 5.871 (1.0 %) Others	11.4 days
	^{219}Rn	α (γ)	6.819 (79.4 %) 6.552 (12.9 %) 6.425 (7.5 %)	3.96 s
	^{215}Po	α	7.386 (100.0 %)	1.78 ms
	^{211}Pb	β (γ)		36.1 min
	^{211}Bi	α (γ)	6.623 (83.5 %) 6.278 (16.2 %)	2.14 min
	^{207}Tl	β		4.77 min

^{A} Descendents with half-lives of less than 30 days.

^{B} Gamma ray indicated only when emission probability per decay is more than 5 % and energy is greater than 0.1 MeV.

^{C} Energy indicated for alpha radiation only. Emission probability per decay in parentheses.

$$Y = (M_B - M_P)/0.01699 \quad (1)$$

where:

M_B = mass of planchet with the dried barium sulfate precipitate, g,
M_P = mass of planchet only, g, and
0.01699 = mass of barium sulfate precipitate if all of the added barium carrier (10.0 mg) were recovered, g.

13.2 Calculate the concentration *AC* of alpha-emitting radium radionuclides as ^{226}Ra in Bq of radium per litre as follows:

$$AC = \frac{R_n}{EVYIF} \quad (2)$$

where:

R_n = alpha counting rate, net counts/s (sample counts/s minus background counts/s),
E = detection efficiency of the counter for alpha particles, counts/disintegration,
V = sample volume, L,
Y = fractional chemical yield for the separation, and

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[5] Eq 1 assumes that exactly 10.0 mg Ba^{++} carrier is added. The theoretical mass of $BaSO_4$ precipitate assuming 100 % recovery (0.01699 g) is derived by dividing the mass, in grams, of barium (Ba^{++}) added by 0.5884 g Ba^{++}/ g $BaSO_4$ (for example, 0.01699 = 0.010 g Ba^{++} / 0.5884). If the standardized concentration of the barium carrier is found to differ from 10.0 mg/mL, the denominator of Eq 1 is modified to reflect the actual quantity of barium carrier added.

IF = correction for the ingrowth of descendants between the time of separation (see 12.6 and Table 1) and the time of counting.

13.3 See Section 10 of Practices D3648 concerning the overall uncertainty in a measurement.

13.4 The combined standard uncertainty (CSU) for the concentration of alpha-emitting radium isotopes is calculated as follows:

$$TPU = AC(Bq/L)*\left(\left(\frac{S_N}{R_n}\right)^2+\left(\frac{S_E}{E}\right)^2+\left(\frac{S_V}{V}\right)^2+\left(\frac{S_Y}{Y}\right)^2\right)^{1/2} \quad (3)$$

where:

S_N = one sigma uncertainty of the net sample alpha counting rate,
S_E = one sigma uncertainty of the detection efficiency of the alpha counter,
S_V = one sigma uncertainty of the sample volume, and
S_Y = one sigma uncertainty in the fractional radium recovery.

13.4.1 The one-sigma uncertainty (S_N) in the net sample counting rate is calculated as follows:

$$S_N = (R_S/t_S + R_b/t_b)^{1/2} \quad (4)$$

where:

R_S = the sample gross counting rate, (s^{-1}),
R_b = the background counting rate, (s^{-1}),
t_s = the sample counting time, s, and
t_b = the background counting time, s.

13.5 The *a priori* minimum detectable concentration (MDC) is calculated as follows:

$$MDC\,(Bq/L) = \frac{3.29*\left(R_b t_b*\left(1+\frac{t_s}{t_b}\right)\right)^{1/2}+2.71}{t_s*E*Y*V*IF} \quad (5)$$

where:

t_s = the counting duration, s, and other terms are as defined earlier.

13.6 The relative contribution of various radium isotopes, if desired, may be obtained by alpha-particle spectroscopy (**7**). Otherwise, repeated measurements of the activity permit estimation of the isotopic composition. Table 2 lists radioactive properties of ^{226}Ra, ^{224}Ra, ^{223}Ra, and their descendants (**8**). Fig. 1 shows characteristic growth and decay curves for the three important isotopes, and equations and tables have been published (**9**).

14. Precision and Bias[6]

14.1 A limited collaborative test of this test method was conducted. Seven laboratories participated by processing samples at three levels. The results from one laboratory were rejected as outliers according to the statistical tests outlined in Practice D2777. These collaborative data were obtained on distilled water without chemical interferences. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

14.2 *Precision*—The overall precision of this test method within its designated range varies with the quantity being tested. See Table 4 for the precision data obtained.

14.3 *Bias*—The limited collaborative study of this test method indicated that there was no statistically significant observed bias in the test method for any level. See Table 3 for the bias data obtained.

15. Quality Control

15.1 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. The batch size should not exceed 20 samples, not including QC samples.

15.2 *Detector Efficiency*—Standards used in this method shall be traceable to a national standards laboratory such as NIST or NPL.

15.2.1 Use three standards for each point in the calibration curve.

15.2.2 The efficiency of each detector shall be verified prior to use, using a source traceable to a national standards laboratory.

15.3 *Initial Demonstration of Laboratory/Instrument Capability:*

15.3.1 If a laboratory or analyst has not performed this test before or if there has been a major change in the measurement system, for example, significant instrument change, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory/instrument capability.

15.3.2 Analyze seven replicates of a standard solution prepared from an IRM (independent reference material) containing accurately known concentrations of radium-226 at concentrations sufficient to minimize the counting uncertainty to less than 2 % at two sigma. Each replicate must be taken through the complete analytical test method including any sample preservation and pretreatment steps. The matrix and chemistry of the solution should be equivalent to that of the samples.

15.3.3 Calculate the mean and standard deviation of the replicate 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. Test Method D5847 should be consulted on the manner by which precision and mean bias are determined from the initial demonstration study.

15.3.4 This method shall not be used for official samples until precision and bias requirements are met.

TABLE 3 Determination of Bias

Amount Added Bq/L	Mean	Bias, ±	Bias, %
0.455	0.522	0.067	14.7
4.588	4.67	0.082	1.7
45.51	47.49	1.98	4.3

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1003.

TABLE 4 Precision Data

Bq/L	s(o)	s(t)
0.455	0.057	0.149
4.588	0.303	0.577
45.51	5.996	7.588

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 LCS should contain radium-226 at a concentration exceeding approximately two to five times the client specified MDC or as specified by the laboratory. The LCS must be taken through all the steps of the method. 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.

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 the analyte found in the blank should be less than the customer's MDC, as specified by the laboratory or below the lowest concentration of analyte in the batch.

15.5.2 The Method Blank must be taken through all the steps of the method.

15.5.3 If the concentration of analyte is found above the limit, the results must be flagged.

15.6 *Matrix Spike:*

15.6.1 Analyze at least one matrix spike sample with each batch of no more than 20 samples by spiking an aliquot of a sample within the batch with a known concentration of radium.

15.6.2 The spike should produce a concentration of radium that is 2 to 5 times the anticipated sample concentration or as specified by the laboratory, whichever is greater.

15.6.3 The Matrix Spike must be taken through all the steps of the method.

15.6.4 Calculate the percent recovery of the matrix spike (R) using the following formula:

$$R = \left| \frac{(A_{as} - A_a)*100}{A_s} \right| \quad (6)$$

where:

A_{as} = the concentration AC of alpha-emitting radium radionuclides as ^{226}Ra in becquerels (Bq) of radium per litre measured in the spiked aliquot,

A_a = the concentration AC of alpha-emitting radium radionuclides as ^{226} Ra in becquerels (Bq) of radium per litre in the sample, and

A_s = the spiked concentration AC of alpha-emitting radium radionuclides as ^{226}Ra in becquerels (Bq) of radium per litre.

15.6.5 The percent recovery, R, should fall within the limit of 50 to 150 % of the expected value. If the concentration is not within these limits, provide an explanation in the case narrative.

15.7 *Duplicate:*

15.7.1 Analyze a sample in duplicate with each batch of no more than 20 samples.

15.7.2 In those cases where there is insufficient sample to allow performance of a duplicate sample analysis, a duplicate analysis of a laboratory control sample duplicate (LCS-D) shall be performed.

15.7.3 In the absence of laboratory specified control limits, compare to the single operator precision using an F test.

15.7.4 If the result exceeds the precision limit, all samples in the batch must be reanalyzed or the results must be flagged with an indication that they do not fall within the performance criteria of the method.

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 single-blind basis (if practical) to the laboratory at least once per quarter that samples are analyzed.

15.8.2 The concentration of analyte in the national standards laboratory traceable reference material should be appropriate to the typical purpose for which the method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

16. Keywords

16.1 alpha particles; radioactivity; radium isotopes; water

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