Designation: D 3454 – 97

An American National Standard

Standard Test Method for Radium-226 in Water [1]

This standard is issued under the fixed designation D 3454; 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 measurement of soluble, suspended, and total radium-226 in water in concentrations above 3.7×10^{-3} Bq/L. This test method is not applicable to the measurement of other radium isotopes.

1.2 This test method may be used for quantitative measurements by calibrating with a radium-226 standard, or for relative measurements by comparing the measurements made with each other.

1.3 This test method does not meet the current requirements of Practice D 2777.

1.4 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.5 Hydrofluoric acid (HF) is very hazardous and should be used in a well-ventilated hood. Wear rubber gloves, safety glasses or goggles, and a laboratory coat. Avoid breathing any HF fumes. Clean up all spills promptly and wash thoroughly after using HF.

1.6 *This standard does not purport to address all of the other 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias Applicable Methods of Committee D-19 on Water[2]

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129, and to other published glossaries.[4]

4. Summary of Test Method

4.1 This test method[5] is based on the emanation and scintillation counting of radon-222, a gaseous daughter product of radium-226, from a solution.

4.2 Radium-226 is collected from water by coprecipitation on a relatively large amount of barium sulfate. The barium-radium sulfate is decomposed by fuming with phosphoric acid, and the resulting glassy melt is dissolved by evaporation with dilute hydrochloric acid to form soluble barium-radium phosphates and chlorides. These salts are dissolved and the solution is stored for ingrowth of radon-222. After a suitable ingrowth period, the radon gas is removed from the solution by purging with gas and transferred to a scintillation counting chamber. About 4 h after radon-222 collection, the scintillation chamber is counted for alpha activity. The radium-226 concentration is calculated from the alpha count rate of radon-222 and its immediate daughters. The radioactive decay characteristics of radium-226 and its immediate decay progeny are listed in Table 1.

5. Significance and Use

5.1 The most prevalent of the five radium isotopes in ground water, having a half life greater than one day, are radium-226 and radium-228. These two isotopes also present the greatest health risk compared to the other naturally occurring nuclides of equal concentrations if ingested via the water pathway.

5.2 Although primarily utilized on a water medium, this technique may be applicable for the measurement of the radium-226 content of any media once the medium has been completely decomposed and put into an aqueous solution.

---

[1] This test method is under the jurisdiction of ASTM Committee D 19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved Aug. 10, 1997. Published February 1998. Originally published as D 3454 – 75T. Last previous edition D 3454 – 91.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] American National Standard Glossary of Terms in Nuclear Science and Technology, N1.1-1967.

[5] This test method is based on a previously published method by Rushing, D.E., Garcia, W.J., and Clark, D.A. "The Analysis of Effluents and Environmental Samples from Uranium Mills and of Biological Samples for Radium, Polonium and Uranium," Radiological Health and Safety in Mining and Milling of Nuclear Materials, Vol. II, IAEA, Vienna, Austria, 1964), p. 187.

TABLE 1 Radioactive Decay Characteristics of Radium-226 and Its Daughters

Radionuclide	Half-life	Mode of Decay
Radium-226	1602 years	α
Radon-222	3.82 days	α
Polonium-218	3.05 min	α
Lead-214	26.8 min	β, γ
Bismuth-214	19.7 min	β, γ
Polonium-214	164 μ s	α
Lead-210	22.3 years	β, γ

5.3 The general methodology and basis of this technique are similar to the methodology "Radium-226 in Drinking Water (Radon Emanation Technique)" as described in the document *EPA-600//4-80-032*.[6]

6. Interferences

6.1 Only the gaseous alpha-emitting radionuclides interfere, namely, radon-219 and radon-220. Their half lives are 3.9 and 54.5 s respectively; their presence indicates the presence of their parents, radium-223 and radium-224. These short-lived radon isotopes decay before the radon-222 is counted; it is their alpha-emitting decay products that would interfere. These interferences are very rare in water samples but are frequently observed in certain uranium mill effluents.

7. Apparatus

7.1 *Radon Bubbler*[7] (Fig. 1).

7.2 *Radon Scintillation Chamber*[8] (also known as Lucas Cell) (Fig. 2).

7.3 *Manometer*, open-end capillary tube or vacuum gage having a volume which is small compared to the volume of the scintillation chamber, 0, − 760 mm Hg (Fig. 3).

7.4 *Gas Purification Tube*, 7 to 8 mm outside diameter standard wall glass tubing, 100 mm long, constricted at lower end to hold a glass wool plug (Fig. 3). The upper half of the tube is filled with magnesium perchlorate and the lower half with a sodium hydrate-asbestos absorbent.

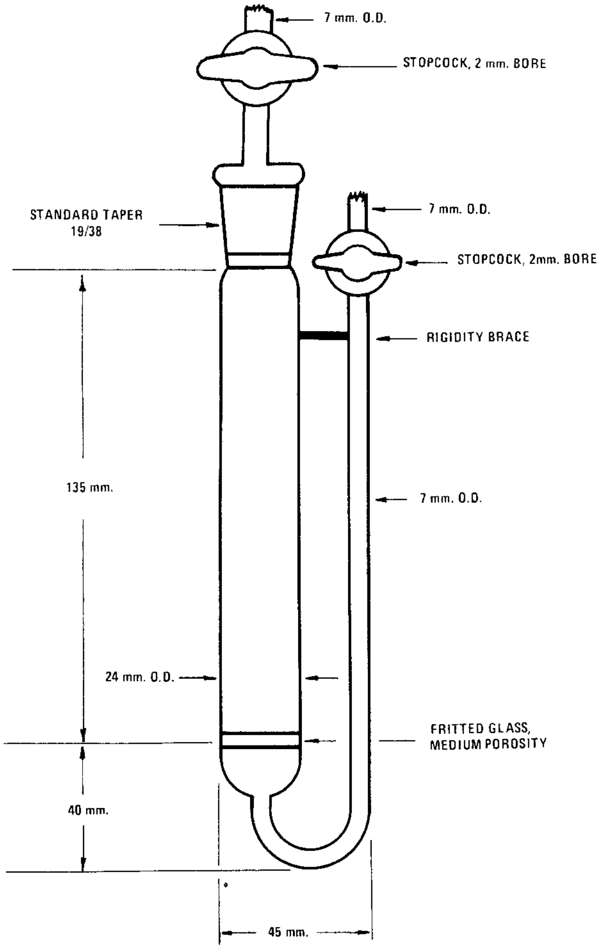

FIG. 1 Radon Bubbler

7.5 *Scintillation Counter Assembly*, consisting of a 50 mm (2 in.) or more in diameter photomultiplier tube mounted in a light-tight housing and coupled to the appropriate preamplifier, high-voltage supply, and scaler. A high-voltage safety switch should open automatically when the light cover is removed to avoid damage to the photomultiplier tube. The preamplifier should incorporate a variable gain adjustment. The counter should be equipped with a flexible ground wire which is attached to the chassis photomultiplier tube by means of an alligator clip or similar device. The operating voltage is ascertained by determining a plateau using radon-222 in the scintillation chamber as the alpha source. The slope of the plateau should not exceed 2 %/100 V. The counter and the scintillation chamber should be calibrated and used as a unit when more than one counter is available. The background counting rate for the counter assembly without the scintillation chamber should range from 0.00 to 0.03 cpm.

7.6 *Membrane Filters*, 0.45-μm pore size.[9]

7.7 *Silicone Grease*, high-vacuum, for bubbler stopcocks.

7.8 *Platinum Ware*, crucibles, 20 to 30 mL, and one 500-mL capacity dish. Platinum ware is cleaned by immersing and rotating in a molten bath of potassium pyrosulfate, removing, cooling, and rinsing in hot tap water, digesting in hot HCl (1+1), rinsing in water, and finally flaming over a burner.

7.9 *Laboratory Glassware*—Glassware may be decontaminated before and between uses by heating for 1 h in EDTA-Na_2CO_3 decontaminating solution at 90 to 100°C, then rinsing in water, in (1+11) HCl and again in water.

---

[6] "Radium-226 in Drinking Water (Radon Emanation Technique)," *Prescribed Procedures for Measurement of Radioactivity in Drinking Water,* August 1980.

[7] Available from Corning Glass Works, Special Sales Section, Corning, N.Y. 11830.

[8] Available from W. H. Johnston Laboratories, 3617 Woodland Ave., Baltimore, MD 21215, and Rocky Mountain Scientific Glass Blowing Co., 4990 E. Asbury Ave. Denver, CO 80222.

[9] Type HAWP (Millipore filter Corp., Bedford, MA) has been found satisfactory. An equivalent may be used.

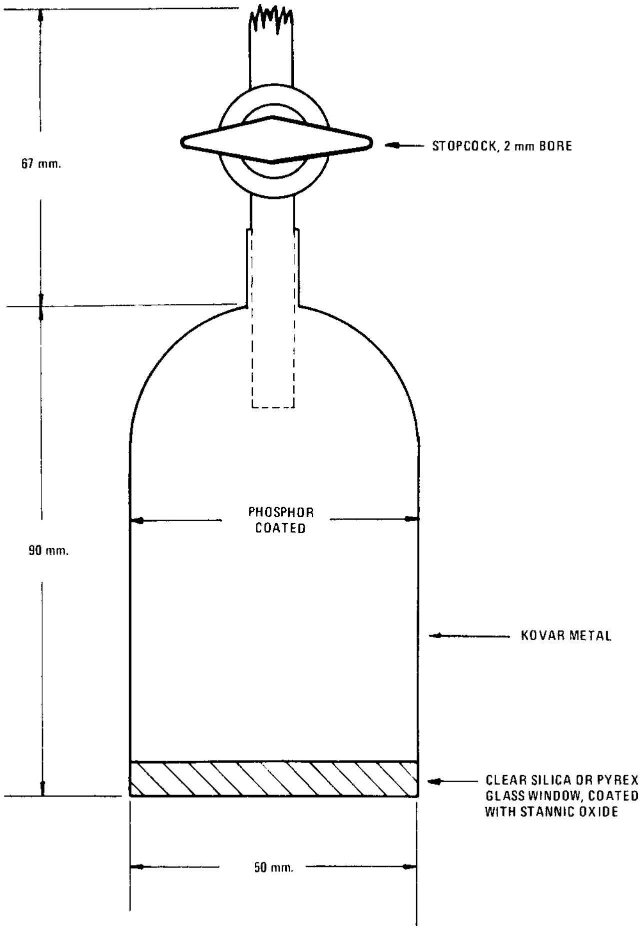

FIG. 2 Radon Scintillation Chamber

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.[10] 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, reference to water shall be understood to mean conforming to Specification D 1193, Type III.

8.3 *Radioactive Purity of Reagents*—Radioactive purity 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 *Ammonium Sulfate Solution* (100 g/L)—Dissolve 10 g of ammonium sulfate ($(NH_4)_2SO_4$) in water and dilute to 100 mL.

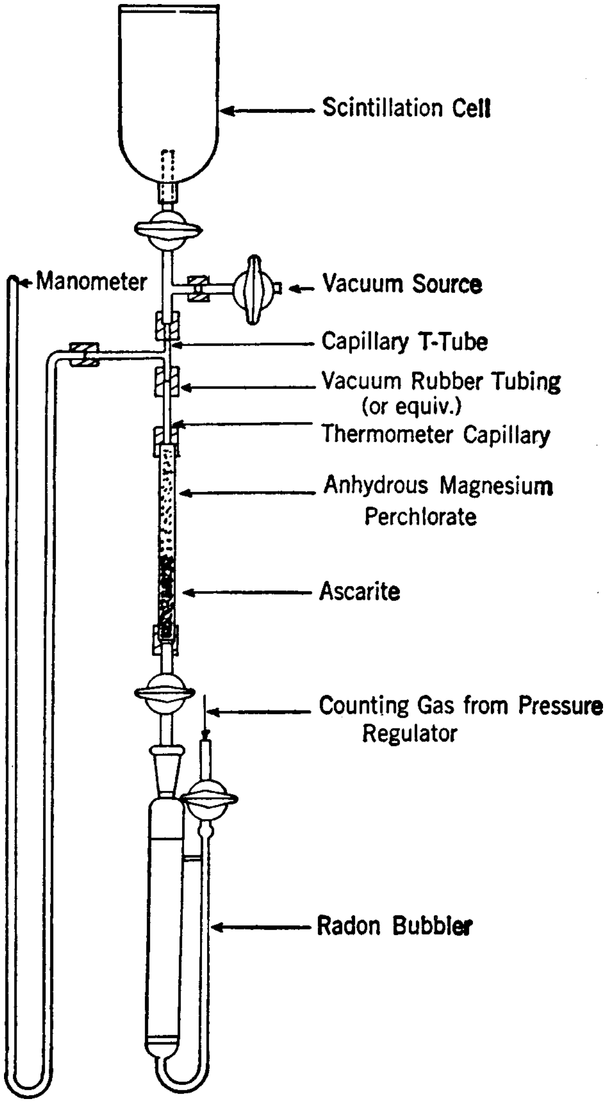

FIG. 3 De-emanation Assembly

8.5 *Barium Chloride Carrier Solution Stock*, (17.8 g/L)—Dissolve 17.8 g of barium chloride ($BaCl_2 \cdot 2H_2O$) in water and dilute to 1 L. This solution will contain 10 mg $Ba^{+\,+}$/mL.

8.6 *Barium-133 Tracer Solution*—(approximately 2.74×10^3 Bq/mL).

8.7 *Barium Chloride Carrier Solution, Working*—Add 100 mL of barium chloride carrier stock solution and 10 mL of barium-133 tracer solution to 890 mL of water and mix thoroughly. This solution will contain approximately 1 g of $Ba^{+\,+}$/L. Allow to stand for 24 h and filter through a membrane filter.

8.8 *EDTA-Sodium Carbonate Decontaminating Solution*—Dissolve 10 g of disodium ethylenediaminetetraacetate and 10 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

8.9 *Flux*—To a large platinum dish (about 500-mL capacity) add 30 mg of $BaSO_4$, 65.8 g of K_2CO_3, 50.5 g of Na_2CO_3, and 33.7 g of $Na_2B_4O_7 \cdot 10\ H_2O$. Mix well and heat cautiously until the water is expelled; fuse and mix thoroughly by swirling. Cool flux, grind it in a porcelain mortar to pass a U. S. Standard No. 10 (2.00-mm) (or finer) sieve. Store in an airtight bottle. (Flux can be prepared in smaller batches.)

---

[10] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.10 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

8.11 *Hydrochloric Acid Solution* (1 + 1)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 1 volume of water.

8.12 *Hydrochloric Acid Solution* (1 + 11)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 11 volumes of water.

8.13 *Hydrochloric Acid Solution* (1 + 49)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 49 volumes of water.

8.14 *Hydrochloric Acid Solution* (1 + 119)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 119 volumes of water.

8.15 *Hydrofluoric Acid* (sp gr 1.15)—Concentrated hydrofluoric acid (HF). Use extreme caution.

8.16 *Hydrogen Peroxide* (1 + 9)—Mix 1 volume of H_2O_2 (30 %) with 9 volumes of water.

8.17 *Magnesium Perchlorate*—Anhydrous magnesium perchlorate $Mg(ClO_4)_2$.

8.18 *Phosphoric Acid* (sp gr 1.69)—Concentrated phosphoric acid (H_3PO_4).

8.19 *Radium Standard Solution* (0.37 Bq/mL).[11]

8.20 *Sodium Hydroxide-Coated Silicate Absorbent, Proprietary*, [12] 8 to 20 mesh.

8.21 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8.22 *Sulfuric Acid Solution* (1 + 359)—Mix 1 volume of concentrated H_2SO_4(sp gr 1.84) with 359 volumes of water. This solution is 0.1 *N*. Slowly add acid to water.

8.23 *Helium*, in a high-pressure cylinder with a two-stage pressure regulator and needle valve.

9. Sampling

9.1 Collect the sample in accordance with the applicable standards as described in Practices D 3370.

10. Calibration and Standardization

10.1 Close the inlet stopcock of a bubbler, (Note 1) add 5 mL of $BaCl_2 \cdot 2H_2O$ carrier solution, 1 mL of concentrated HCl (sp gr 1.19), 3 mL (1.1 Bq) of standard radium solution and fill the bubbler ⅔ to ¾ full with water.

NOTE 1—Before using, test bubblers by placing about 10 mL of water in them and passing air through them at the rate of 3 to 5 mL/min. This should form many fine bubbles rather than a few large ones. Do not use bubblers requiring excessive pressure to initiate bubbling. Reject unsatisfactory bubblers. Corning's“ medium-porosity” fitted glass disks are usually satisfactory.

10.2 Insert the outlet stopcock into the bubbler with the stopcock open. Adjust the helium regulator (diaphragm) valve so that a very slow stream of gas will flow with the needle valve open. Attach the helium supply to the inlet of bubbler and adjust the inlet pressure to produce a froth a few millimetres thick. Establish a zero ingrowth time by purging the liquid with helium for 15 to 20 min.

10.3 In rapid succession, close the inlet stopcock, remove the gas connection, and the close outlet stopcock. Record the date and time and store the bubbler preferably for 2 to 3 weeks before collecting and counting the radon-222.

10.4 Attach a scintillation chamber as shown in Fig. 3; *substitute a glass tube with a stopcock for the bubbler* so that the helium gas can be turned on and off conveniently. Open the stopcock on the scintillation chamber; close the stopcock to the gas and gradually open the stopcock to vacuum source to evacuate the cell. Close the stopcock to the vacuum source and check the manometer reading for 2 min to test the system, especially the scintillation chamber for leaks. If leaks are detected they should be identified and sealed.

10.5 Open the stopcock to the helium gas and allow the gas to enter the chamber slowly until atmospheric pressure is reached. Close all the stopcocks.

10.6 Place the scintillation chamber on the photomultiplier tube (in a light-tight housing), wait 10 min, and obtain a background count rate (preferably over a period of at least 100 min). Phototube must not be exposed to external light with the high voltage applied.

10.7 With the scintillation chamber and bubbler in positions indicated in Fig. 3 and all stopcocks closed, open the stopcock to vacuum and then to the scintillation chamber. Evacuate the scintillation cell and the gas purification system. Close the stopcock to vacuum and check for leaks as in 10.4.

10.8 Adjust the helium regulator (diaphragm) valve so that a very slow stream of gas will flow with the needle valve open. Attach the helium supply to the inlet of the bubbler.

10.9 Very cautiously open the bubbler outlet stopcock to equalize pressure and transfer all or most of the fluid in the inlet side arm to the bubbler chamber.

10.10 Close the outlet stopcock and very cautiously open the inlet stopcock to flush remaining fluid from the side arm and fritted disk. Close the inlet stopcock.

10.11 Repeat steps 10.9 and 10.10 several times to obtain more nearly equal pressure on the two sides of the bubbler.

10.12 With the outlet stopcock fully open, cautiously open the inlet stopcock so that the flow of gas produces a froth a few millimetres thick at the surface of bubbler solution. Maintain the flow rate by adjusting the pressure with the regulator valve and continue de-emanation until the pressure in the scintillation chamber reaches the atmospheric pressure. The total elapsed time for de-emanation should be 15 to 20 min.

10.13 In rapid succession, close the stopcock to the scintillation chamber, close the bubbler inlet and the outlet stopcocks, shut off and disconnect the gas supply. Record the date and time, which is the end of ingrowth and the beginning of decay.

10.14 Store the bubbler for another radon-222 ingrowth in the event a subsequent de-emanation is desired. The standard bubbler containing the standard may be kept and reused indefinitely.

10.15 Four hours after de-emanation, place the scintillation chamber on the photomultiplier tube, wait 10 min, and count with desired statistical accuracy is achieved. Record the date and time the counting was started and finished.

10.16 Calculate the calibration constant *E*, for the scintillation chamber as follows:

$$E = c/A(1 - e^{-\lambda t_1})(e^{-\lambda t_2})$$

---

[11] Standard radium solutions are available from the National Institute of Standards and Technology.

[12] Ascarite II has been found to be suitable and is available from VWR Scientific.

where:

c = net count rate, cpm (cpm standard − cpm background),
A = activity of radium-226 in the bubbler, dpm,
t_1 = ingrowth time of radon-222, h,
t_2 = decay time of radon-222 occurring between de-emanation and counting, h, and
λ = decay constant of radon-222 (0.00755 h^{-1}).

10.17 Carry out the background measurements and calibrations with each scintillation chamber used, and repeat frequently as the calibration constant can change with time.

10.18 To remove radon-222 and prepare the scintillation chamber for reuse, evacuate and cautiously refill with helium. Repeat this evacuation and refilling twice. For chambers containing high activities of radon-222 repeat the procedure more often.

11. Procedure

11.1 *Soluble Radium-226*:

11.1.1 Filter the sample through a membrane filter. Take a 1-L aliquot, or a smaller volume so as not to exceed 1.1 Bq of radium-226, and transfer to a 1500-mL beaker. Acidify with 20 mL of concentrated HCl (sp gr 1.19) per litre of filtrate, heat, and add with vigorous stirring 50 mL of $BaCl_2$ working carrier solution. For sample volumes less than a litre, dilute to 1 L with HCl (1 + 49) prior to the addition of carrier.

11.1.2 Cautiously and with vigorous stirring, add 20 mL of H_2SO_4 (sp gr 1.84). Cover the beaker and allow to stand overnight.

11.1.3 Filter the supernate through a membrane filter, using H_2SO_4 (1 + 359) to transfer the Ba-Ra precipitate to the filter. Wash the precipitate twice with H_2SO_4 (1 + 359).

11.1.4 Place the filter in a platinum crucible, add 0.5 mL of concentrated HF (sp gr 1.15) and 3 drops (0.15 mL) of $(NH_4)_2SO_4$ solution, and evaporate to dryness.

11.1.5 Carefully ignite the filter and residue over a small flame until the carbon is burned off (after charring of filter, a Meeker burner may be used).

11.1.6 Cool, add 1 mL of concentrated H_3PO_4 (sp gr 1.69), and heat on a hot plate to about 200°C. Gradually raise temperature to about 300 to 400°C for 30 min.

11.1.7 Swirl the crucible over a low bunsen flame, adjusted to avoid spattering. Swirl so that the crucible walls are covered with hot concentrated H_3PO_4 (sp gr 1.69). Continue to heat until the $BaSO_4$ dissolves to give a clear melt (just below redness), and then heat for 1 min more to ensure removal of SO_3.

11.1.8 Cool, fill the crucible one-half full with HCl (1 + 1), heat on a steam bath, then gradually add the water to within 2 mm of the top of the crucible.

11.1.9 Evaporate on the steam bath until there are no more vapors of HCl.

11.1.10 Add 6 mL of HCl (1 + 11), swirl, and warm to dissolve the $BaCl_2$ crystals.

11.1.11 Close the inlet stopcock of a greased and tested radon bubbler. Add a drop of water to the fritted disk and transfer the sample from the platinum crucible to the bubbler using a medicine dropper. Rinse the crucible with at least three 2-mL portions of water. Add water until the bubbler is ⅔ to ¾ full.

11.1.12 De-emanate the solution in accordance with 10.2 and 10.3.

11.1.13 After 3 weeks of radon-222 ingrowth, de-emanate and count as described in 10.7 through 10.15.

11.1.14 Transfer the solution in the bubbler to a gamma-counting container. Wash the bubbler thoroughly with HCl (1 + 11) and combine with the sample in a container. Measure the barium-133 gamma activity in a gamma counter. For a discussion of gamma ray counting refer to Practice D 3649. Calculate the sample yield, Y, by dividing the barium-133 activity of the sample by the barium-133 activity of a 50-mL aliquot of $BaCl_2$ carrier working solution counted under identical conditions of volume and geometry as the sample.

11.1.15 The sample may be stored for a second ingrowth or discarded and the bubbler cleaned for reuse. A thorough rinsing with HCl (1 + 49) is a satisfactory cleaning procedure. If however, the radium-226 in a bubbler exceeded 10 pCi, a more rigorous cleaning may be necessary. Remove the stopcock grease, using a cloth and solvent, and then immerse for 1 h in hot (90 to 100°C) EDTA − Na_2CO_3 solution. Heat the bubblers gradually to avoid thermal shock to the fritted glass disks. Remove, cool at room temperature, and rinse with distilled water. Immerse in HCl (1 + 11) and warm for about 30 min. Remove, cool, and rinse with distilled water. Dry and regrease the stopcocks.

11.1.16 Remove radon-222 from the scintillation chamber as described in 10.18.

11.2 *Suspended Radium-226*:

11.2.1 Filter a volume of sample containing up to 30 pCi of radium-226 and 1.0 g of inorganic suspended matter through a membrane filter. If desired the filter and suspended matter from step 10.1.1 may be used.

11.2.2 Place the membrane filter and suspended material into a weighed 30-mL platinum crucible. Carefully ignite over a small flame until the carbon is burned off (after charring of the filter, a Meeker burner may be used).

11.2.3 Cool and weigh the crucible to estimate the residue.

11.2.4 Add 8 g of flux for each gram of residue, but not less than 2 g of flux, and mix with a glass stirring rod.

11.2.5 Heat over a Meeker burner until melting begins, then more carefully to avoid spattering. Continue heating for 20 min after bubbling stops with occasional swirling of the crucible to mix the contents and achieve a uniform melt. A clear melt is usually obtained only when the suspended solids are present in small amounts or have a high silica content.

11.2.6 Remove the crucible from the burner and rotate it as the melt cools to distribute it in a thin layer on the crucible walls.

11.2.7 To a 500 mL beaker containing 120 mL H_2O, slowly add, with stirring, 20 mL of concentrated H_2SO_4 (sp gr 1.84) and 5 mL of H_2O_2(1+9) for each 8 g of flux used.

11.2.8 Place the crucible in a beaker, cover, and swirl the beaker to dissolve the melt.

11.2.9 When the melt is dissolved, lift the crucible with platinum-tipped tongs, and rinse with water, allowing rinse water to go into the beaker.

11.2.10 When the melt is dissolved, rinse and remove the crucible from the beaker and save for reuse in step 11.2.13.

11.2.11 Heat the solution and slowly add 50 mL of $BaCl_2$ working solution with vigorous stirring. Cover the beaker and allow to stand overnight for precipitation.

11.2.12 Add 1 mL of H_2O_2 (1+9) and if the yellow color (from titanium) deepens, add additional H_2O_2 until there is no further color change.

11.2.13 Continue as described in steps 11.1.3 to 11.1.16.

11.3 *Soluble and Suspended Radium–226*:

11.3.1 Take a 1-L aliquot of the thoroughly mixed sample, or a smaller volume so as not to exceed (1.1 Bq) of radium-226 and transfer to a 1500-mL beaker. Acidify with 20 mL of concentrated HCl (sp gr 1.19) per litre of sample, heat, and add with vigorous stirring 50 mL of $BaCl_2$ working carrier solution. For sample volumes less than a litre, dilute to 1 L with HCl (1 + 49) prior to the addition of carrier.

11.3.2 Cautiously and with vigorous stirring add 20 mL of concentrated H_2SO_4 (sp gr 1.84). Cover the beaker and allow to stand overnight.

11.3.3 Filter the supernate through a membrane filter, using H_2SO_4(1 + 359) to transfer the solids to the filter. Wash the solids twice with H_2SO_4 (1 + 359).

11.3.4 Continue as described in steps 11.2.2 to 11.2.10.

11.3.5 Digest the sample for 1 h on a steam bath and add 1 mL of H_2O_2 (1 + 9). If the yellow color (from titanium) deepens, add additional H_2O_2 until there is no further color change.

11.3.6 Continue as described in steps 11.1.3 to 11.1.16.

12. Calculation

12.1 Calculate the concentration of radium-226 in becquerels per litre as follows:

$$D = \frac{c}{EVR} \times \frac{1}{1 - e^{-\lambda t_1}} \times \frac{1}{e^{-\lambda t_2}}$$

where:
D = concentration of Radium-226, Bq/L,
c = calibration constant for the scintillation cell, cpm/disintegrations per minute,
v = volume of sample used, L,
R = recovery factor,
t_1 = elapsed time between the first and second de-emanations; λ is the decay constant of radon-222 (0.181 d^{-1}), days,
t_2 = time interval between the second de-emanation and time of mid count, h; λ is the decay constant of radon-222 (0.00755 h^{-1}),
c = net count rate (sample-background), cps

12.2 The total propagated uncertainty (1 s) for the concentration of Radium-226 is calculated as follows:

$$S_D\,(\mathrm{Bq/L}) = D(\mathrm{Bq/L})*[S_C/C)^2 + (S_E/E)^2 + (S_V/V)^2 + (S_R/R)^2]^{1/2} \tag{1}$$

where:
S_C = one sigma uncertainty of the net sample counting rate,
S_E = one sigma uncertainty of the detection efficiency of the scintillation cell,
S_V = one sigma uncertainty of the sample volume, and
S_R = one sigma uncertainty in the fractional radium recovery.

The one sigma uncertainty (S_C) in the net sample counting rate is calculated from the following:

$$S_C = (G/t_G{}^2 + B/t_B{}^2)^{1/2} \tag{2}$$

where:
G = sample gross counting rate, s^{-1},
B = background counting rate, s^{-1},
t_G = sample counting time, s, and
t_B = background counting time, s.

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

$$MDC(\mathrm{Bq/L}) = \frac{2.71 + 4.65*(t_B*B)^{1/2}}{t_G*E*R*V*F} \tag{3}$$

where:
F = product of the ingrowth and two decay factors,
E = detection efficiency,
R = fractional recovery, and
V = sample volume.

13. Precision and Bias [13]

13.1 The available data do not permit a precision and bias statement to be made in accordance with Practice D 2777.

13.2 A limited collaborative study of this test method was conducted. Seven labs participated by processing one sample at four levels. These collaborative data were obtained on distilled water with reagent grade chemicals added to vary the hardness. The resultant hardness was 125 mg/L for levels A and B and 610 mg/L for levels C and D.

13.3 *Precision*—The overall precision of this test method within its designated range varies with the quantity being tested according to Fig. 4. The relative precision for this test method is approximately 5 %.

13.4 *Bias*—A limited collaborative study of this test method indicated that a negative bias of approximately 3 % was present, based on the average recovery of the known amount of radium-226 added. Recoveries were as follows:

Level	pci/L	Added, Bq/L	Found, Bq/L	Bias, %
A	12.12	0.448	0.4351	−2.9
B	8.96	0.331	0.3221	−2.7
C	25.53	0.944	0.9214	−2.4
D	18.84	0.697	0.6831	−2.0

14. Quality Control

14.1 Whenever possible, the project leader, as part of the external quality control program, should submit quality control samples to the analyst along with routine samples in such a way that the analyst does not know which of the samples are the quality control samples. These external quality control samples which usually include duplicate and blank samples, should test sample collection and preparation as well as sample analysis whenever this is possible. In addition, analysts are expected to run internal quality control samples that will indicate to them whether the analytical procedures are in control. Both the external and internal quality control samples should be prepared in such a way as to duplicate the chemical

---

[13] Supporting data for this test method have been filed at ASTM Headquarters. Request RR:D-19-1120.

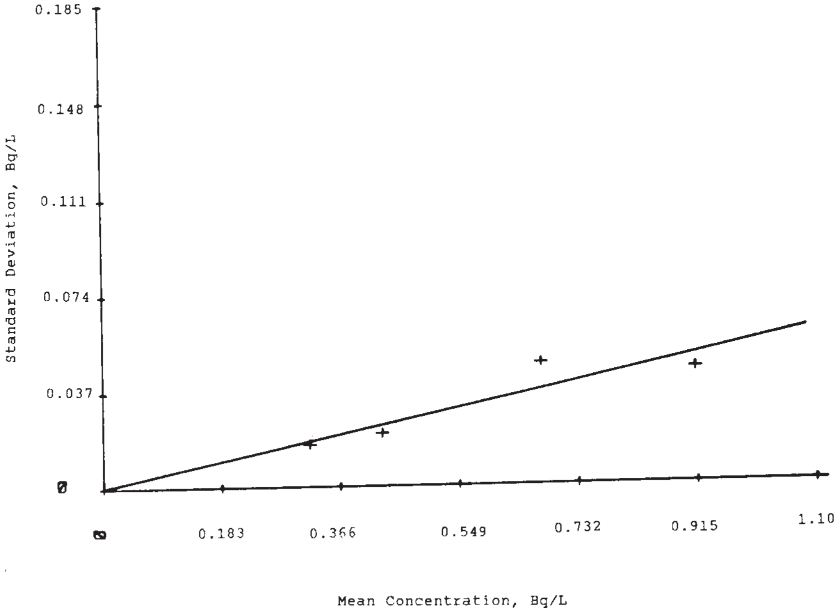

FIG. 4 Overall Standard Deviation versus Mean Concentration

matrix of the routine samples, insofar as this is practical. The quality control samples that are routinely used consist of five basic types: blank samples, replicate samples, reference materials, control samples, and spiked samples.

15. Keywords

15.1 coprecipitation; emanation; radioactivity; radium-226; radon-222; water

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).