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Designation: D5072-98 Designation: D 5072 - 09

# Standard Test Method for Radon in Drinking Water<sup>1</sup>

This standard is issued under the fixed designation D 5072; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1This test method covers the measurement of radon in drinking water in concentrations above 0.04 Bq/L.

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

1.3

1.1 This test method covers the measurement of radon in drinking water in concentrations above 2 Bq/L.

1.2 This test method may be used for absolute measurements by calibrating with a <sup>226</sup>Ra standard or for relative measurements by comparing the measurements made with each other.

1.3 This test method is used successfully with drinking water samples and Type III reagent water conforming to Specification D 1193. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. 1.5 This standard does not purport to address 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.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D 1129 Terminology Relating to Water D 1193 Specification for Reagent Water S.//Standards.iten.al

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19D19 on Water

D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup> Practices for Sampling Water from Closed Conduits

D 3648 Practices for the Measurement of Radioactivity

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water

D 4448 Guide for Sampling Ground-Water Monitoring Wells

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D 6001 Guide for Direct-Push Ground Water Sampling for Environmental Site Characterization

D 7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements 2.2 Other Documents

Multi-Agency Radiological Laboratory Analytical Protocols Manual EPA402-B-04-001A, NUREG1576, NTIS PB2004-105421, July 2004.

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129 and to other published glossaries. Definitions: For definitions of terms used in this test method, refer to Terminology D 1129. For terms not defined in this test method or in Terminology D 1129, reference may be made to other published glossaries.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 11.01

<sup>2</sup> 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.

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<sup>&</sup>lt;sup>1</sup> 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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# 4. Summary of Test Method

4.1This test method is based on the scintillation counting of radon-222 and its daughters, a gaseous daughter product of radium-226.

4.2An aliquot of unaerated water is drawn into a syringe then gently injected beneath 10 mL of liquid scintillation mix that does not contain an emulsifier. The vials are capped, shaken, and allowed to stand 3 h prior to counting. A commercial liquid scintillation counter is used to count the sample.

4.1 This test method is based on the scintillation counting of <sup>222</sup>Rn and its progeny.

4.2 In a glass liquid scintillation vial, an aliquot of unaerated water is drawn into a syringe then gently injected beneath 10 mL of a suitable liquid scintillation cocktail that does not contain an emulsifier. The vials are capped, shaken, and allowed to stand 3 hours prior to counting to permit dark adaptation and buildup of short-lived radon progeny. Radon-222 contained in the sample is selectively partitioned into the scintillation cocktail. The sample is counted using a liquid scintillation counting system optimized for detection of <sup>222</sup>Rn activity.

# 5. Significance and Use

5.1 The most prevalent of the radon isotopes in ground water is radon-222. is <sup>222</sup>Rn. This isotope presents the greatest health risk compared to the other naturally occurring radon isotopes if ingested via the water pathway.

# 6. Interferences

6.10ther radionuclides soluble in the scintillation mix may interfere. Water that is contaminated with high energy beta/gamma emitters, even though they are not soluble in the scintillation mix, may also interfere. These interferences would be rare in drinking water samples but may be observed in some cases.

<u>6.1</u> Other radionuclides soluble in the scintillation cocktail may interfere. High energy beta/gamma emitters, even though they are not soluble in the scintillation mix, may also interfere. These interferences will cause a high bias if present in a significant quantity. These interferences would be rare in drinking water samples but may be observed in some cases.

6.2 A reduced or increased counting efficiency may result if sample quenching is significantly different than that of the calibration standard.

6.3 Scintillation stock cocktails and sample cocktail mixes must be dark-adapted to prevent artificial excitation of fluors which may lead to falsely elevated count rates which could compromise data quality.

# 7. Apparatus

7.1 Sampling Funnel.

- 7.2 Tube, with standard faucet fitting.
- 7.3 Disposable Syringe, 12 mL capacity, with 20 gauge, 38 mm (1 in.) hypodermic needle.
- 7.4 Glass Liquid Scintillation Vials, 20 mL capacity with polyethylene inner seal cap liners.
- 7.5 Liquid Scintillation Counter alog/standards/sist/63252484-95a0-4301-99ab-ebf721f93e09/astm-d5072-09

# 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.<sup>3</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 conforming to Specification D 1193, Type III.<sup>4</sup>

8.3 *Radioactive Purity* Radioactive Purity of Reagents — Radioactive purity shall be such that the measured radioactivity of blank samples does not exceed the calculated probable error of measurements.

8.4 *Radium-226 Solution Standard*, traceable to the National Institute of Standards and Technology (NIST)., traceable to a national standards laboratory such as the National Institute of Standards and Technology (NIST) or the National Physical Laboratory (NPL).

8.5 Scintillation Cocktail Mix, without emulsifier. Toluene based mix is acceptable.

# 9. <u>Safety</u>

9.1 Some scintillation cocktails can pose a significant health hazard if handled improperly. Refer to manufacturer instructions for the safe use of these materials.

<sup>&</sup>lt;sup>3</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 Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>&</sup>lt;sup>4</sup> If this water is not aerated or degassified prior/subsequent to demineralization, radon background may be substantial. This can happen if the lab uses RO, EDI or only deminerialization resins for purification of their water.

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# 10. Sampling

NOTE 1—Refer to Practices D 3370 for applicable sampling instructions. Also see U.S. Environmental Protection Agency reports EPA 520<sup>5</sup> and EPA 600<sup>6</sup>.

9.1Attach the sampling funnel and tube to a faucet with the standard faucet fitting.

9.2Slowly turn on the water and allow a steady stream to flow out of the funnel for approximately 2 min. This purges the tube and ensures a fresh sample.

9.3Reduce the flow of water and invert the funnel. The flow should be adjusted to a level that does not cause turbulence in the pool of water contained in the funnel. Allow excess water to spill over one edge of the funnel.

9.4Examine the hose connection and tubing for air bubbles or pockets. If these are visible, raise or lower the funnel until they are removed.

### **10.Calibration and Standardization**

10.1Add a known quantity of traceable NIST radium-226 standard solution to a known volume of water.

10.2Combine a 10 mL aliquot of the radium-226 standard solution with 10 mL of scintillation mix in a 20 mL glass scintillation vial.

10.3Allow approximately 30 days for buildup of radon (that is, secular equilibrium with radium-226).

10.4Shake vial to transfer nearly all the radon to the scintillation mix phase (radon is highly soluble in the scintillation mix). The radium-226 remains in the aqueous phase and, therefore, does not contribute significantly to the count rate.

10.5Allow for the buildup of short-lived radon progeny and for dark-adapting by waiting 3 h before counting.

10.6In liquid-scintillation vials, prepare background samples consisting of 10 mL of Type III reagent water and 10 mL of scintillation solution. Cap the vials and shake to mix the contents. Allow approximately 30 days for decay of any residual radon. 10.7Count the standard and background samples for 50 min or longer.

10.1 Attach the sampling funnel and tube to a faucet with the standard faucet fitting.

10.2 Slowly turn on the water and allow a steady stream to flow out of the funnel for approximately 2 min. This purges the tube and ensures a fresh sample.

10.3 Reduce the flow of water and invert the funnel. The flow should be adjusted to a level that does not cause turbulence in the pool of water contained in the funnel. Allow excess water to spill over one edge of the funnel.

10.4 Examine the hose connection and tubing for air bubbles or pockets. If these are visible, raise or lower the funnel until they are removed.

# 11. Calibration and Standardization

11.1 Add a known quantity of traceable <sup>226</sup>Ra standard solution to a known volume of water.

11.2 Prepare three <sup>222</sup>Rn calibration standards by combining a 10 mL aliquot of the <sup>226</sup>Ra standard solution with 10 mL of scintillation cocktail in a 20 mL glass scintillation vial. Securely cap each vial and shake to mix the contents.

<u>11.3</u> In liquid scintillation vials, prepare three background samples containing 10 mL of Type III reagent water and 10 mL of scintillation solution. Cap the vials and shake to mix the contents.

11.4 Allow approximately 30 days for buildup of radon (that is, secular equilibrium with <sup>226</sup>Ra).

 $\frac{11.5}{1.5}$  Shake vial to transfer nearly all the radon to the scintillation mix phase (radon is highly soluble in the scintillation mix). The <sup>226</sup>Ra remains in the aqueous phase and, therefore, does not contribute significantly to the count rate.

11.6 Allow for the buildup of short-lived radon progeny and for dark-adapting by waiting 3 hours before counting.

<u>11.7</u> Count the standard samples for a counting period sufficiently long to obtain a relative counting uncertainty of <1 % (10 000 net counts minimum). Count background samples at least as long as the test samples.

11.8 Calculate the calibration factor (CF) as per Section 13.1.

#### <u>12.</u> Procedure

1+2.1 Clean scintillation vials with alcohol and add 10 mL of scintillation mix.cocktail.

112.2 Collect unaerated sample in accordance with Section 910.

142.3 Place the tip of the hypodermic needle approximately 3 cm under the surface of the water in the funnel and withdraw a few millilitres of water and eject this water. Using this procedure, rinse the syringe and hypodermic needle two or three more times.

1+2.4 Again, place the tip of the needle approximately 3 cm below the surface of the water and withdraw approximately 12 mL.

<sup>&</sup>lt;sup>5</sup> EPA 520/5–83–027 Methods and Results of EPA's Study of Radon in Drinking Water. Published December 1983. For availability, contact the Superintendent of Documents, U.S. Government Printing Office, North Capital and "H" Sts., NW, Washington, DC 20401.

<sup>&</sup>lt;sup>5</sup> EPA 520/5–83–027 Methods and Results of EPA's Study of Radon in Drinking Water. Published December 1983. Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

<sup>&</sup>lt;sup>6</sup> EPA 600/2–87/082 Two Test Procedures for Radon in Drinking Water. Published March 1989. For availability, contact the Superintendent of Documents, U.S. Government Printing Office, North Capital and "H" Sts., NW, Washington, DC 20401.

<sup>&</sup>lt;sup>6</sup> EPA 600/2-87/082 Two Test Procedures for Radon in Drinking Water. Published March 1989. Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

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NOTE 2—The water should be pulled into the syringe slowly to avoid extreme turbulence and collection of air bubbles. If large air bubbles are noticed in the syringe, the sample should be rejected and redrawn.

142.5 Invert the syringe and slowly eject any small air bubbles and extra water. Retain precisely 10 mL of water in the syringe. 142.6 Remove the cap from a vial and carefully place the tip of the needle into the bottom of the liquid scintillation solution. Slowly eject the water from the syringe into the vial.

Note3—The water <u>3</u>—Water is injected under the liquid scintillation solution to prevent loss of radon from the sample. If the water is forced out of the syringe with much pressure, it will cause turbulence in the solution and could result in loss of radon.

 $1\pm 2.7$  Carefully withdraw the hypodermic needle from the vial and replace the cap. The cap should be tightly secured to prevent leakage. Shake the vial to mix the contents.

112.8 Repeat the previous steps to obtain two separate aliquots from each sample.

11.9Load 12.9 Load the samples into the liquid-scintillation counter and, counting system and after waiting for 3 h, count for at least 50 minutes.

1+2.10 After dark-adapting them, count a background sample, consisting of 10 mL of water and 10 mL of scintillation solution, and a standard radium-226 solution sample for 50 min at the beginning of counting and after every ten drinking water samples.

#### 12.13. Calculation

12.1Calculate the becquerels per litre of radon in the sample by using the following equation:

$$A = \frac{(C_{S} - C_{B})(1000\text{mL})}{(CF)(D)(10\text{mL})(1L)}$$

13.1 Calibration Factor, CF:

$$CF = \frac{(C_{CS} - C_B)}{A_{CS}} \tag{1}$$

where:

iTeh Standards

$A\underline{C_{CS}} =$	becquerels of radon per litre of sample, calibration standard count rate, counts per second ( $s^{-1}$ ) (as prepared in Section	
	11.2), (https://standards.iteh.al)	
$C_{c}B =$	sample counts per second (cps), average background sample count rate ( $s^{-1}$ ) (as prepared in Section 11.3), and	

 $C_{\rm B}A_{\rm CS}$  = sample counts per second (eps), <u>average background sample count rate (s ) (as prepared in Section 11.5), and</u>  $C_{\rm B}A_{\rm CS}$  = background eps, calibration standard <sup>226</sup>Ra activity, Bq.

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<u>13.2</u> Sample Activity, AC—Calculate the radon activity concentration in the sample as follows. First calculate the net count rate in counts per second:

$$R_n = R_a - R_b \tag{2}$$

Then calculate the activity concentration of radon in the sample, in becquerels per liter (Bq/L), using the following equation:

$$AC = \frac{R_n}{CF \times D \times V_a} \tag{3}$$

where:

 $\overline{AC}$  = activity concentration of <sup>222</sup>Rn (Bq/L),

 $\underline{R}_a \equiv \underline{\text{Sample count rate } (s^{-1})},$ 

- $\overline{\underline{R}}_{b} = \overline{background \ count \ rate \ (s^{-1})},$
- $\underline{R}_n \equiv \underline{\text{net count rate } (s^{-1})},$
- $\overline{CF}$  = conversion factor calculated as, (cps measured for calibration standard in 10.7) /(dps (disintegrations per second) of radium-226 contained in 10-mL aliquot of 10.2). calibration factor calculated as in Eq 1,

$$D = \frac{1}{\text{decay correction } D = \exp\left(-\frac{0.693(T)}{t_{1/2}}\right)},$$

decay correction factor  $D = e^{-ln(2) \times T/t_{1/2}}$ ,

- $V_a \equiv$  volume of sample analyzed (0.010 L),
- $\overline{T}$  = time in days from collection time to midpoint of counting time, and
- $t_{\frac{1}{2}}$  = radiological half-life of radon, 3.82 days. half-life of <sup>222</sup>Rn, 3.82 d.

### $t_{1/2}$

### 13.Precision and Bias

13.1The collaborative test conducted on this test method included 15 laboratories each with one operator. Three activity levels between 60 Bq/L and 2454 Bq/L were tested with three replicates per level. The determination of the precision and bias statements were made in accordance with Practice D2777.

13.2These collaborative test data were obtained using reagent grade water. For other matrices, these data may not apply. 13.3The overall and single operator precision have been found to vary with level in a manner according to Table 1.