ASTM INTERNATIONAL

Designation: D1943 – 20

Standard Test Method for
Alpha Particle Radioactivity of Water [1]

This standard is issued under the fixed designation D1943; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the measurement of alpha particle activity of water. It is applicable to nuclides that emit alpha particles with energies above 3.9 MeV and at activity levels above 0.02 Bq/mL (540 pCi/L) of radioactive homogeneous water. This test method is not applicable to samples containing alpha-emitting radionuclides that are volatile under conditions of the analysis.

1.2 This test method can be used for either absolute or relative determinations. In tracer work, the results may be expressed by comparison with a standard that is defined to be 100 %. For radioassay, data may be expressed in terms of alpha disintegration rates after calibration with a suitable standard. General information on radioactivity and measurement of radiation has been published in Refs **(1-3)**[2] and summarized in Practices D3648.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Flowing Process Streams
D3648 Practices for the Measurement of Radioactivity
D4448 Guide for Sampling Ground-Water Monitoring Wells
D6001 Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization
D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
D7902 Terminology for Radiochemical Analyses

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminologies D1129 and D7902.

3.1.2 For terms not defined in this standard, Terminology D1129, or Terminology D7902, reference may be made to other published glossaries.

4. Summary of Test Method

4.1 The test sample is reduced by evaporation or a suitable chemical method to the minimum weight of material having measurable alpha activity. Alpha radioactivity is measured by an instrument composed of a detecting device, amplifier, power supply, and scaler—the most widely used being proportional and scintillation counters. In the proportional counter, which may be of the windowless or thin window type, alpha particles entering the sensitive region of the detector produce ionization of the counting gas. The negative ion of the original ion pair is

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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.

Current edition approved Oct. 15, 2020. Published November 2020. Originally approved in 1996. Last previous edition approved in 2012 as D1943 – 05 (2012). DOI: 10.1520/D1943-20.

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

[3] 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.

accelerated towards the anode, producing additional ionization of the counting gas and developing a voltage pulse at the anode. In the scintillation detector, alpha particles interact with the material of the phosphor, transferring some of their energy to electrons. These electrons subsequently lose part of their energy by excitation rather than ionization of atoms, and the excited atoms revert to the ground state by re-emitting energy in the form of light quanta. A suitable light-sensitive device, usually a photomultiplier or solid-state diode, transforms the resulting flashes of light into voltage pulses. By use of suitable electronic apparatus, the pulse is amplified to a voltage sufficient for operation of the counting scaler. The number of pulses per unit time is related to the disintegration rate of the test sample. The efficiency of the system can be determined by use of a suitable alpha standard having equivalent residual plated solids.

5. Significance and Use

5.1 This test method was developed for the purpose of measuring gross alpha radioactivity in water. It is used for the analysis of both process and environmental water to determine gross alpha activity which is often a result of natural radioactivity present in minerals.

6. Measurement Variables

6.1 The relatively high absorption of alpha particles in the sample media affects the counting rate of the measurement. Effects of geometry, back-scatter, source diameter, as well as the purity, pressure variation, and type of counting gas used shall also be considered. Thus, for reliable relative measurements, the variables shall be held constant while counting all test samples and standards. For absolute measurements, appropriate efficiency factors shall be applied. If a windowless proportional counter is employed, the sample mount shall be electrically conducting.

6.1.1 In tracer studies or tests requiring only relative measurements, in which the data are expressed as being equivalent to a defined standard, the above correction factors can be simply combined into a counting efficiency factor. The use of a counting efficiency factor requires that sample mounting, material of mounting dish, and weight of residue (milligrams per square centimetre), in addition to conditions affecting the above described factors, remain constant throughout the duration of the test and that the comparative standard be prepared for counting in the same manner as the test samples. The data from comparative studies between independent laboratories when not expressed in absolute units are more meaningful when expressed as percentage relationships or as equivalent of a defined standard.

6.2 The limit of sensitivity for both scintillation and proportional counters is a function of the background counting rate which should be as low as is feasible. Massive shielding is not used for alpha counters. The maximum activity for this test method is 1600 Bq.

7. Interferences

7.1 Solids content in the sample containing the alpha emitter produces significant losses in sample counting rates of about 10 to 15 % loss at 1 mg/cm^2. Liquid samples shall be evaporated to dryness onto dishes that allow the sample to be counted directly by the detector. Solids on the dish shall remain constant in amount between related test samples, and should duplicate the density of the solids of the plated standard.

7.2 Most alpha counters are insensitive to beta, gamma, and X-ray ([1-3]).

8. Apparatus

8.1 *Alpha Particle Counter,* consisting of either a proportional detector or a scintillation detector, and a scaler conforming to the following requirements:

8.1.1 *Proportional Detector*—This may be one of several types commercially available. The material used in the construction of the detector should contain a minimal amount of detectable radioactivity. To establish freedom from undesirable characteristics, the manufacturer shall supply voltage plateau and background counting rate data. Voltage plateau data shall show the threshold voltage, slope, and length of plateau for a particular input sensitivity.

8.1.2 *Scintillation Detector*—This may be one of several types commercially available. It shall consist of an "activated" zinc sulfide phosphor having a minimum effective diameter of 36.5 mm and a superficial density of 10 to 15 mg/cm^2. The phosphor shall be mounted so that it can be attached and optically coupled to a multiplier phototube. Extraneous light shall be excluded from the phosphor either by its being covered with a thin (less than 1 mg/cm^2) opaque window or by enclosing the assembly in a lightproof sample changer. The material used in the construction of the detector shall be free from detectable radioactivity. To establish freedom from undesirable characteristics, the manufacturer shall supply voltage plateau and background counting rate data. Voltage plateau data shall show the threshold voltage, slope, and length of a plateau for a specified scaler sensitivity.

8.1.3 *Scaler*—Often the scaler, power supply, and amplifier are contained in a single chassis, generally termed the scaler. The power supply and amplifier sections shall be matched with the type of detector to produce satisfactory operating characteristics and to provide sufficient range in adjustments to maintain controlled conditions. The manufacturer shall provide resolving time information for the counting system. The scaler shall have capacity for storing and visually displaying at least 10^6 counts with a resolving time no greater than 5 µs. The instrument shall have an adjustable input sensitivity that can be matched to the detector and a variable high voltage power supply with indicating meter.

8.2 *Sample Mounting Dish*—Dishes having a flat bottom of a diameter slightly less than the inside diameter of the detector. Flat dishes are preferred, but dishes may be used that have 3.2 mm high side walls with the angle between dish bottom and side equal to or greater than 120°. Dishes shall be of a material that will not corrode under the plating conditions and shall be of uniform surface density; platinum and stainless steel have been used for this purpose.

9. Reagents

9.1 *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 and free from radioactivity to preclude detrimental effects. Some chemicals, even of high purity, contain naturally occurring radioactive elements, for example, uranium, actinium, and thorium. Consequently, when carrier chemicals are used in the analysis of low-radioactivity samples, the radioactivity of the carriers shall be determined under identical analytical conditions of the sample including residual dish solids. The radioactivity of the reagents shall be considered as background and subtracted from the test sample counting rate.

9.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

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

9.4 *Nitric Acid* (1 + 30)—Mix 1 volume of concentrated HNO_3 (sp gr 1.42) with 30 volumes of water.

9.5 *Alpha-Emitting Radioactive Standard Solution* (~200 Bq/mL), traceable to the SI through a national metrology institute (NMI) such as National Institute of Science and Technology (NIST) in the United States and the National Physical Laboratory (NPL) in the United Kingdom.

10. Sampling

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

10.2 Preserve the sample in a radioactive homogeneous state. A sample shall be made radioactive homogeneous by addition of a reagent in which the radionuclides or compounds of the radionuclides present would be soluble in large concentrations. Addition of acids, complexing agents, or stable chemically similar carriers may be used to obtain homogeneity. Consideration of the chemical nature of the radionuclides and compounds present and the subsequent chemistry of the method shall indicate the action to be taken.

11. Establishing Gas Proportional Counter Operating Plateau

11.1 Put the instrument into operation according to the manufacturer's instructions. Place a quality control (QC) source in the detector, set the sensitivity control near its maximum and turn the "count" switch to "count" position. Slowly increase the high voltage until the first counts are observed and record the "threshold" voltage. Advance the voltage in increments of convenient magnitude (approximately 25 V) and determine the counting rate at four or more settings of the sensitivity control at each voltage setting. Measure the background counting rate at each of the settings using an empty sample mounting dish in place of the standard.

11.1.1 The QC source may be any available alpha-emitting radionuclide having a half-life sufficiently long to eliminate decay corrections. Knowledge of its true disintegration rate is not essential. The radionuclide shall be permanently fixed to the dish and uniformly distributed over an area preferably smaller than the dish bottom; electro-deposition and flaming of a salt-flee solution are the two methods most generally used. Quality control sources are commercially available.

11.2 Plot the gross counting rate of the standard against the voltage. The counting rate should rise initially as the voltage is increased, then, for at least some of the settings of the sensitivity control, reach an approximate constant value, and finally rise again. The" plateau" of the curve should be at least 100 V in length and have a slope less than 2 %/100 V; however, shorter plateaus or one with greater slope shall be acceptable if a well-regulated high voltage power supply is available.

11.3 Plot the ratio of the square of the net counting rate of the standard to the background counting rate against the voltage for each of the settings of the sensitivity control.

11.4 Determine the optimum conditions for operation of the instrument by selecting values for the high-voltage and sensitivity adjustments that correspond to some point lying on the plateau of the counting-rate-versus-voltage plot and near the maximum value of the ratio of the sample-squared-to-background counting rates.

12. Control of Instrument Operation

12.1 Tolerance charts or statistical control charts are used to assure that the instrument is operating to within pre-specified limits of the initial calibration. Repetitive measurements of a quality control source are taken to develop the tolerance chart or statistical control chart. The QC source is then used on a daily or prior-to-use basis to ensure proper operation. Refer to Practices D3648 or Practice D7282 for the preparation of a tolerance chart or control chart.

13. Calibration and Standardization for General Measurements

13.1 Place a known amount of a NMI-traceable alpha standard (approximately 200 Bq) into a volume of water sufficient to dissolve salts (or into a volume of water containing dissolved salts) equivalent to those of the test samples and prepare for counting as directed in Section 15. Throughout the experiment, the evaporation, mounting, counting, and density of plate solids of this reference standard shall be identical with those of the test samples. Count for the length of time required to obtain the desired relative standard counting uncertainty (typically 1 %). The efficiency factor for each dissolved salt weight, ε, is then calculated according to the following equation:

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[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

$$\varepsilon = \frac{R}{A} \qquad (1)$$

where:

R = measured count rate (s^{-1}), and
A = alpha activity added (Bq).

The alpha-emitting standard should have approximately the same alpha-particle energy as the nuclides of interest so that mass attenuation effects can be estimated appropriately.

13.1.1 Purified natural uranium, which has specific activity of 0.02535 Bq/µg, has been found satisfactory for this purpose. Other alpha-emitter preparations of known activity, for example, ^{241}Am or ^{237}Np, may also be used. When available, all calibration solutions shall be traceable to an NMI.

14. Calibration and Standardization for Tracer Experiments

14.1 Add a known quantity of activity from a reference solution of the tracer (approximately 180 Bq) to a radioactivity-free standard test sample and process as directed in Section 15.

15. Procedure

15.1 Place an appropriate volume of the test solution in a glass beaker, add 3 mL of concentrated HNO_3 (sp gr 1.42) for each 100 mL of solution, and evaporate to 1 to 2 mL. Quantitatively transfer to the mounting dish and evaporate to dryness. Adjust the heat carefully to prevent spattering or boiling. A ring heater having a continuously variable voltage control or adjustable infrared heat lamps are the preferable heat sources for the final evaporation and drying. Uniform spreading of the residual salts is necessary for reliable comparative data. After drying, heat the dish to dull redness for a few seconds using a burner. Cool hygroscopic solids in a dry atmosphere and store in a desiccator until the start of counting. Place the sample in the counter and count for a time interval sufficient to attain the desired statistical reliability. Record the reading of the scaler. Transfer of large volume samples to smaller beakers as evaporation nears completion makes for easier transfer of the test specimen to the mounting dish. Make all transfers with HNO_3 (1 + 30). Choose the sample size with consideration for the absorption of alpha particles in the residual solids. The size should be such that the density of the deposit on the plate shall not exceed 5 mg/cm^2.

15.2 Precipitation methods may be used expediently to concentrate the radioactive material into small amounts of precipitate. The precipitate is separated and washed free of precipitant by centrifugation or filtration. Choose the method of separation that will produce a uniform deposit of precipitate after quantitatively transferring to the mounting dish for counting. Calibrate the instrument under counting conditions identical to those used for the samples. More detailed information is published in Refs (1-3) on the techniques and equipment for separation and mounting of the precipitate.

16. Calculation

16.1 Results may be expressed in becquerels per millilitre (Bq/mL). This test method is useful for comparing activities of a group of samples, as in tracer experiments. Results may also be reported in terms of equivalent ^{241}Am activity or other standard radionuclide activity using the empirical efficiency determined by use of a reference standard. If it is known that only one nuclide is present, its disintegration rate may be determined by use of the efficiency factor determined from a reference standard of that nuclide obtained from a NMI. Calculate the results as follows:

$$AC = \frac{R_N}{\varepsilon \cdot V} \qquad (2)$$

where:

AC = alpha activity concentration (Bq/mL),
R_N = net count rate (s^{-1}),
ε = detector efficiency factor, and
V = volume of test specimen (mL).

The combined standard uncertainty of the alpha concentration is calculated as:

$$u_c(AC) = \sqrt{\frac{u_c^2(R_N)}{(\varepsilon \cdot V)^2} + AC^2\left(\frac{u^2(\varepsilon)}{\varepsilon^2} + \frac{u^2(V)}{V^2}\right)} \qquad (3)$$

where:

$u_c(AC)$ = combined standard uncertainty of AC,
$u_c(R_N)$ = combined standard (counting) uncertainty of R_N,
$u(\varepsilon)$ = standard uncertainty of the efficiency ε, and
$u(V)$ = standard uncertainty of the volume V.

The net count rate, R_N, and its combined standard uncertainty, $u_c(R_N)$, are defined as:

$$R_N = R_S - R_B = \frac{C_S}{t_S} - \frac{C_S}{t_B} \qquad (4)$$

$$u_c(R_N) = \sqrt{\frac{C_S}{t_S^2} + \frac{C_B}{t_B^2}} \qquad (5)$$

where:

R_S = sample count rate (s^{-1}),
R_B = background count rate (s^{-1}),
C_S = sample counts,
C_B = background counts,
t_s = counting time of sample (s), and
t_B = counting time of background (s).