Designation: D 1890 – 96

An American National Standard

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

This standard is issued under the fixed designation D 1890; 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 Department of Defense.

1. Scope

1.1 This test method covers the measurement of beta particle activity of water, as referenced to the beta energy of ^{137}Cs, not corrected for conversion electrons. It is applicable to beta emitters having maximum energies above 0.1 MeV and at activity levels above 0.02 Bq/mL of radioactive homogeneous water for most counting systems. This test method is not applicable to samples containing 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 which is defined to be 100 %. For radioassay, data may be expressed in terms of a known radionuclide standard if the radionuclides of concern are known and no fractionation occurred during processing, or may be expressed arbitrarily in terms of some other standard such as cesium-137. General information on radioactivity and measurement of radiation may be found in the literature [2] and Practice D 3648.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water [3]

D 1193 Specification for Reagent Water [3]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water [3]

D 3370 Practices for Sampling Water from Closed Conduits [3]

D 3648 Practice for the Measurement of Radioactivity [4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Becquerel*—a unit of radioactivity equivalent to 1 nuclear transformation per second.

3.1.2 *beta energy, maximum*—the maximum energy of the beta-particle energy spectrum produced during beta decay of a given radioactive species.

NOTE 1—Since a given beta-particle emitter may decay to several different quantum states of the product nucleus, more than one maximum energy may be listed for a given radioactive species.

3.1.3 *counter background—in the measurement of radioactivity*, the counting rate resulting from factors other than the radioactivity of the sample and reagents used.

NOTE 2—Counter background varies with the location, shielding of the detector, and the electronics; it includes cosmic rays, contaminating radioactivity and electrical noise.

3.1.4 *counter beta-particle efficiency—in the measurement of radioactivity*, that fraction of beta particles emitted by a source which is detected by the counter.

3.1.5 *counter efficiency—in the measurement of radioactivity*, that fraction of the disintegrations occurring in a source which is detected by the counter.

3.1.6 *radioactive homogeneous water*—water in which the radioactive material is uniformly dispersed throughout the volume of water sample and remains so until the measurement is completed or until the sample is evaporated or precipitating reagents are added to the sample.

3.1.7 *reagent background—in the measurement of radioactivity of water samples*, the counting rate observed when a sample is replaced by mock sample salts or by reagent chemicals used for chemical separations that contain no analyte.

NOTE 3—Reagent background varies with the reagent chemicals and analytical methods used and may vary with reagents from different

[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.

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[2] Friedlander, G., et al., *Nuclear and Radiochemistry*, 3rd Ed., John Wiley and Sons, Inc., New York, NY, 1981.

Price, W. J., *Nuclear Radiation Detection*, 2nd Ed., McGraw-Hill Book Co., Inc., New York, NY, 1964.

Lapp, R. E., and Andrews, H. L., *Nuclear Radiation Physics*, 4th Ed., Prentice-Hall Inc., New York, NY, 1972.

Overman, R. T., and Clark, H. M., *Radioisotope Techniques*, McGraw-Hill Book Co., Inc., New York, NY, 1960.

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

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

manufacturers and from different processing lots.

3.2 *Definitions*—For terms not defined in this test method or in Terminology D 1129, reference may be made to other published glossaries. [5]

4. Summary of Test Method

4.1 Beta radioactivity may be measured by one of several types of instruments composed of a detecting device and combined amplifier, power supply, and scaler—the most widely used being proportional or Geiger-Müller counters. Where a wide range of counting rates is encountered (0.1 to 1300 counts per seconds), the proportional-type counter is preferable due to a shorter resolving time and greater stability of the instrument. The test sample is reduced to the minimum weight of solid material having measurable beta activity by precipitation, ion exchange resin, or evaporation techniques. Beta particles entering the sensitive region of the detector produce ionization of the counting gas. The negative ion of the original ion pair is accelerated towards the anode, producing additional ionization of the counting gas and developing a voltage pulse at the anode. By use of suitable electronic apparatus, the pulse is amplified to a voltage sufficient for operation of the counter scaler. The number of pulses per unit of time is related to the disintegration rate of the test sample. The beta-particle efficiency of the system can be determined by use of prepared standards having the same radionuclide composition as the test specimen and equivalent residual plated solids. An arbitrary efficiency factor can be defined in terms of some other standard such as cesium-137.

5. Significance and Use

5.1 This test method was developed for the purpose of measuring the gross beta radioactivity in water. It is used for the analysis of both process and environmental water to determine gross beta activity.

6. Measurement Variables

6.1 The relatively high absorption of beta particles in the sample media and any material interposed between source and sensitive volume of the counter results in an interplay of many variables which affect the counting rate of the measurement. Thus, for reliable relative measurements, hold all variables constant while counting all test samples and standards. For absolute measurements, appropriate correction factors are applied. The effects of geometry, backscatter radiation, source diameter, self-scatter and self-absorption, absorption in air and detector window for external counters, and counting coincidence losses have been discussed [2] and may be described by the following relation:

$$cps = Bq_b\,(G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c) \quad (1)$$

where:

cps = recorded counts per second corrected for background,

Bq_b = disintegrations per second yielding beta particles,

G_p = point source geometry (defined by the solid angle subtended by the sensitive area of the detector),

f_{bs} = backscatter factor or ratio of *cps* with backing to *cps* without backing,

f_{aw} = factor to correct for losses due to absorption in the air and window of external detectors. It is equal to the ratio of the actual counting rate to that which would be obtained if there were no absorption by the air and window between the source and sensitive volume of the detector. Expressed in terms of absorption coefficient and density of absorber, $f_{aw} = e^{-\mu x}$, where μ = absorption coefficient, in square centimetres per milligram, and x = absorber density in milligrams per square centimetre.

f_d = factor to correct a spread source counting rate to the counting rate of the same activity as a point source on the same axis of the system,

f_{ssa} = factor to correct for the absorption and scatter of beta particles within the material accompanying the radioactive element, and

f_c = factor for coincident events to correct the counting rate for instrument resolving time losses and defined by the simplified equation, $f_c = 1 - nr$, where, n = the observed counts per second, and r = instrument resolving time in seconds. Generally, the sample size or source to detector distance is varied to obtain a counting rate that precludes coincident losses. Information on the effect of random disintegration and instrument resolving time on the sample count rate as well as methods for determining the resolving time of the counting system may be found in the literature.

For most applications, a detector system is calibrated using a single beta emitting radionuclide and an efficiency of detection, f_o, response curve generated for various sample residue weights. The efficiency of detection for each sample residual weight incorporates all the factors mentioned above so that:

$$f_o = cps/Bq = (G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c) \quad (2)$$

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, density of mounting dish, weight of residue in milligrams per square centimetre, and radionuclide composition, 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 the equivalent of a defined standard. Expressing the data in either of these two ways minimizes the differences in counters and other equipment and in techniques used by the laboratories conducting the tests.

6.2 The limit of sensitivity for both Geiger-Muller and proportional counters is a function of the background counting rate. Massive shielding or anti-coincidence detectors and

[5] American National Standard Glossary of Terms in Nuclear Science and Technology (ANSI N1.1).

circuitry, or both, are generally used to reduce the background counting rate to increase the sensitivity.

7. Interferences

7.1 Material interposed between the test sample and the instrument detector, as well as increasing density in the sample containing the beta emitter, produces significant losses in sample counting rates. Liquid samples are evaporated to dryness in dishes that allow the sample to be counted directly by the detector. Since the absorption of beta particles in the sample solids increases with increasing density and varies inversely with the maximum beta energy, plated solids shall remain constant between related test samples and should duplicate the density of the solids of the plated standard.

7.2 Most beta radiation counters are sensitive to alpha, gamma, and X-ray radiations, with the degree of efficiency dependent upon the type of detector. [2] The effect of interfering radiations on the beta counting rate is more easily evaluated with external-type counters where appropriate absorbers can be used to evaluate the effects of interfering radiation.

8. Apparatus

8.1 *Beta Particle Counter*, consisting of the following components:

8.1.1 *Detector*—The end-window Geiger-Muller tube and the internal or external sample gas-flow proportional chambers are the two most prevalent commercially available detector types. The material used in the construction of the detector should be free from detectable radioactivity. When detectors contain windows, the manufacturer shall supply the window density expressed in milligrams per square centimetre. 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. Detectors requiring external positioning of the test sample are mounted on a tube support of low-density material (aluminum or plastic) and positioned so the center of the window is directly above the center of the test sample. The distance between the detector window and test sample plays an important part in determining the geometry of the system and can be varied for external counters to correspond more favorably with such factors as activity level, source size, sensitivity requirements, energy of beta particles, etc. A convenient arrangement is to combine the tube mount with a sample holder containing slots for positioning the sample at three or four distances from the detector window, varying from approximately 5 to 100 mm from tube flange.

8.1.2 *Detector Shield*—The detector assembly is surrounded by an external radiation shield of massive metal equivalent to approximately 51 mm of lead and lined with 3.2-mm thick aluminum. The material of construction should be free from detectable radioactivity. The shield has a door or port for inserting or removing specimens. Detectors having other than completely opaque windows are light sensitive. The design of the shield and its openings shall eliminate direct light paths to the detector window; beveling of door and opening is generally satisfactory. The percentage of the beta particles scattered from the walls of the shield into the detector can be reduced by increasing the internal diameter of the shield. The use of a detector without a shield will significantly increase the background and the detection capability.

8.1.3 *Scaler*—Normally the scaler, mechanical register, power supply, and amplifier are contained in a single chassis, generally termed the scaler. The power supply and amplifier sections are matched by the manufacturer 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 and with a resolving time no greater than 250μ s for use with Geiger Muller detectors or 5 μs for use with proportional detectors. The instrument shall have an adjustable input sensitivity matched and set by the manufacturer to that of the detector, and a variable high-voltage power supply with indicating meter.

8.2 *Sample Mounting*—Sample mounting shall utilize dishes having a flat bottom of a diameter no greater than that of the detector window preferably having 3.2-mm high side walls with the angle between dish bottom and side equal to or greater than 120° to reduce side-wall scattering (Note 4). Dishes shall be of a material that will not corrode under the plating conditions and should be of uniform surface density preferably great enough to reach backscatter saturation. [2]

NOTE 4—Sample dishes with vertical side walls may be used but the exact positioning of these dishes relative to the detector is very important. This factor becomes critical for dishes having the same diameter as the detector. Dishes having side walls more than 3.2 mm in height are not recommended. Stainless steel has been found to be satisfactory for this purpose.

8.3 *Alpha Particle Absorber*—Aluminum or plastic, having a uniform density such that total absorbing medium (air plus window plus absorber) between sample and sensitive volume of detector is approximately equal to 7 mg/cm ^{2} of aluminum. The absorber diameter shall be equal to or greater than the detector window and should be placed against the window to minimize scattering of the beta particles by the absorber. This absorber is not used when counting beta particles with maximum energies below 0.35 MeV due to the high-count rate loss by absorption (about 48 % at 0.35 MeV in 7 mg/cm ^{2} of aluminum). The alpha particle absorber is not recommended for use with internal beta particle detectors, especially when either the composition or activity ratios of the radionuclides or radioactivity level might vary significantly between samples. Chemical separation of the alpha and beta particle emitters produces a higher degree of accuracy for internal detector measurements. Use published information [2] on beta particle absorption as a guide.

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,