METHOD 9310

GROSS ALPHA AND GROSS BETA

1.0 SCOPE AND APPLICATION

     1.1   This method covers the measurement of gross alpha and gross beta particle activities in surface and ground waters.

     1.2   The method is applicable to the measurement of alpha emitters having energies above 3.9 mega electron volts (MeV) and beta emitters having maximum energies above 0.1 MeV.

     1.3   The minimum limit of concentration to which this method is applicable depends on sample size, counting-system characteristics, background, and counting time.

     1.4   Because, in this method for gross alpha and gross beta measurement, the radioactivity of the sample is not separated from the solids of the sample, the solids concentration is very much a limiting factor in the sensitivity of the method for any given water sample. Also, for samples with very low concentrations of radioactivity, it is essential to analyze as large a sample aliquot as is needed to give reasonable times.

     1.5   The largest sample aliquot that should be counted for gross alpha activity is that size aliquot which gives a solids density thickness of 5 mg/cm^2 in the counting planchet. For a 2-in. diameter counting planchet (20 cm^2), an aliquot containing 100 mg of nitrated dissolved solids would be the maximum aliquot size for that sample which should be evaporated and counted for gross alpha activity.

     1.6   When the concentration of total solids (TS) is known for a given water sample and the alpha background and the counting efficiency of a given counting system are known, the counting time that is needed to meet the required sensitivity (3 pCi/L) can be determined by equations given in Appendix C.

     1.7   For the counting of gross beta activity in a water sample, the TS is not as limiting as for gross alpha activity because beta particles are not stopped in solids as easily as are alpha particles. Very often a single sample aliquot is evaporated and counted for both gross alpha and gross beta activity. In that case, the sample aliquot size would be dictated by the solids limitations for alpha particles. For water samples that are to be counted for gross beta activity, equations in Appendix C can also be used to determine the necessary counting time to meet a sensitivity for gross beta activity (4 pCi/L).

     1.8   Radionuclides that are volatile under the sample preparation conditions of this method will not be measured. In some areas of the country the nitrated water solids (sample evaporated with nitric acid present) will

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not remain at a constant weight after being dried at 105°C for 2 hr and then exposed to the atmosphere before and during counting. Other radioactivities (such as some chemical forms of radioiodine) may also be lost during the sample evaporation and drying at 105°C. Those types of water samples need to be heated to a dull red heat for a few minutes to convert the salts to oxides. Sample weights are then usually sufficiently stable to give consistent counting rates, and a correct counting efficiency can then be assigned. Some radioactivities, such as the cesium radioisotopes, may be lost when samples are heated to a dull red color. Such losses are limitations of the test method.

1.9   This method provides a rapid screening measurement to indicate whether specific analyses are required. When the gross alpha particle activity exceeds 5 pCi/L, the same or an equivalent sample shall be analyzed for alpha-emitting radium isotopes (Method 9315) or an alternative measurement of radium-226 alpha emission (Standard Methods for the Examination of Water and Wastewater, 15th edition, Method 705 or 706, respectively). Gross beta particle emissions exceeding 15 pCi/L in a sample shall be analyzed for strontium-89 and cesium-134 (Standard Methods for the Examination of Water and Wastewater, 15th edition, Methods 704 and 709, respectively). If gross beta activity exceeds 50 pCi/L, the identity of the major radioactive constituents must be evaluated and the appropriate organ and total body doses determined.

2.0   SUMMARY OF METHOD

2.1   An aliquot of a preserved water sample is evaporated to a small volume and transferred quantitatively to a tared 2-in. stainless steel counting planchet. The sample residue is dried to constant weight, reweighed to determine dry residue weight, and then counted for alpha and/or beta radioactivity.

2.2   Counting efficiencies for both alpha and beta particle activities are selected according to the amount of sample solids from counting efficiency vs. sample solids standard curves.

3.0   INTERFERENCES

3.1   Moisture absorbed by the sample residue is an interference because it obstructs counting and self-absorption characteristics. If a sample is counted in an internal proportional counter, static charge on the sample residue can cause erratic counting, thereby preventing an accurate count.

3.2   Nonuniformity of the sample residue in counting planchet interferes with the accuracy and precision of the method.

3.3   Sample density on the planchet area should be not more than 10 mg/cm^2 for gross alpha and not more than 20 mg/cm^2 for gross beta.

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3.4   When counting alpha and beta particle activity by a gas-flow proportional counting system, counting at the alpha plateau discriminates against beta particle activity, whereas counting at the beta plateau is sensitive to alpha particle activity present in the sample. This latter effect should be determined and compensated for during the calibration of the specific instrument being used.

4.0   APPARATUS AND MATERIALS

   4.1   Gas-flow proportional counting system, or

   4.2   Scintillation detection system, or

   4.3   Stainless steel counting planchets.

   4.4   Electric hot plate.

   4.5   Drying oven.

   4.6   Drying lamp.

   4.7   Glass desiccator.

   4.8   Glassware.

   4.9   Analytical balance.

5.0   REAGENTS

   5.1   All chemicals should be of "reagent-grade" or equivalent whenever they are commercially available.

   5.2   Distilled or deionized water (Type II) having a resistance value between 0.5 and 2.0 megaohms (2.0 to 0.5 mhos)/cm at 25°C.

   5.3   Nitric acid, 1 N: Mix 6.2 mL 16 N HNO_3 (conc.) with deionized or distilled water and dilute to 100 mL.

6.0   SAMPLE COLLECTION, PRESERVATION, AND HANDLING

   6.1   All samples must have been collected in a manner which addresses the considerations discussed in Chapter Nine of this manual.

   6.2   It is recommended that samples be preserved at the time of collection by adding enough 1 N HNO_3 to the sample to bring it to pH 2 (15 mL 1 N HNO_3 per liter of sample is usually sufficient). If samples are to be collected without preservation, they should be brought to the laboratory within 5 days and then preserved and held in the original container for a minimum of 16 hr before analysis or transfer of the sample.

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6.3   The container choice should be plastic rather than glass to prevent loss due to breakage during transportation and handling.

7.0   PROCEDURE

    7.1   <u>Calibration</u>:

        7.1.1   For absolute gross alpha and gross beta measurement, the detectors must be calibrated to obtain the ratio of count rate to disintegration rate.   Americium-241 (used for alpha activity in the collaborative test of this method) has higher alpha particle energy and radium-226 radionuclides but is close to the energy of the alpha particles emitted by naturally occurring thorium-228 and radium-224.   Standards should be prepared in the geometry and weight ranges to be encountered in these gross analyses.   It is, therefore, the prescribed radionuclide for gross alpha calibration.   NBS or NBS-traceable americium-241 is available from Standard Reference Materials Catalog, NBS Special Publications 260, U.S. Department of Commerce (1976) and from Quality Assurance Branch, EMSL-LV, P.O. Box 15027, Las Vegas, Nevada 89114.

        7.1.2   Strontium-90 and cesium-137 have both been used quite extensively as standards for gross beta activity.   Standard solutions of each of these radionuclides are readily available.   Cesium is volatile at elevated temperatures (above 450°C).   Some water supplies have dissolved solids (salts) that, when converted to nitrate salts, are quite hygroscopic and need to be converted to oxides by heating to red heat to obtain sample aliquots that are weight-stable.   Sample weight stability is essential to gross alpha and gross beta measurements to ensure the accuracy of the self-absorption counting efficiency factor to be used for the samples. Strontium-90 in equilibrium with its daughter yttrium-90 is the prescribed radionuclide for gross beta calibrations.

        7.1.3   For each counting instrument to be used, the analyst should prepare separate alpha and beta particle self-absorption graphs showing water sample residue weight (mg) vs. the efficiency factor (cpm/dpm), using standard alpha and beta emitter solutions and tap water.   For the alpha graph standard, alpha activity is added to varying sizes of aliquots of tap water such that the aliquot residue weight is varied between 0 and 100 mg (for a 2-in. counting planchet).   A similar graph is prepared with standard beta activity and tap-water aliquots, varying the residue weight between 0 and 300 mg (for a 2-in. planchet).   If it is planned to use water-sample aliquot volumes that always contain 100 mg of dried water solids, then only the efficiency factor for that residue weight needs to be established.

        7.1.4   Tap water aliquots, with added americium-241 or strontium-90 standard, should be acidified with a few mL 16 N HNO_3, evaporated to a small volume in a beaker on a hot plate, transferred quantitatively in 5-mL portions or less to a tared counting planchet, evaporated to dryness, and finally dried at 105°C for 1 hr (or flamed to a red heat if dried

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solids appear to be noticeably hygroscopic). Weight-stable aliquot residues should then be alpha and/or beta counted until at least 10,000 total counts have been accumulated. A single set of reference standards prepared in this way can be used for each counting instrument for separate graph preparations and can be stored for reverification whenever needed.

7.2   Transfer to a beaker an aliquot of water sample of a volume that contains no more than 100 mg (for alpha only or alpha and beta determination) or 200 mg (for beta only determination) of total water solids. Evaporate the aliquot to near dryness on a hot plate. If water samples are known or suspected to contain chloride salts, those chloride salts should be converted to nitrate salts before the sample residue is transferred to a stainless steel planchet (chlorides will attack stainless steel and increase the sample solids, and no correction can be made for those added solids). Chloride salts can be converted to nitrate salts by adding 5-mL portions of 16 N HNO_3 to the sample residue and evaporating to near dryness. (Two treatments are usually sufficient.) Add 10 mL 1 N HNO_3 to the beaker and swirl to dissolve the residue. Quantitatively transfer the aliquot concentrate in small portions (not more than 5 mL at a time) to a tared planchet, evaporating each portion to dryness.

7.3   Dry the sample residue in a drying oven at 105°C for at least 1 hr, cool in a desiccator, weigh, and count. Store the sample residue in a desiccator until ready for counting.

7.4   Some types of water-dissolved solids, when converted to nitrate salts, are quite hygroscopic even after being dried at 105°C for 1 hr. When such hygroscopic salts are present with samples that are put into an automatic counting system, those samples gain weight while they are waiting to be counted, and inaccurate counting data result. When there is evidence of hygroscopic salts in sample counting planchets, it is recommended that they be flamed to a dull red heat with a Meeker burner for a few minutes to convert the nitrate salts to oxides before weighing and counting. (It is possible to have a loss of cesium during the flaming of the samples.)

7.5   Count for alpha and beta activity at their respective voltage plateaus. If the sample is to be recounted for reverification, store it in a desiccator.

NOTE: As long as counting chambers are capable of handling the same size planchet, alpha and beta activities can be determined at their respective voltage plateaus in the designated counting instruments. Keep the planchet in the desiccator until ready to count because vapors from moist residue can damage detector and window and can cause erratic measurements. If the gas-flow internal proportional counter does not discriminate for the higher energy alpha pulses at the beta plateau, the alpha activity must be subtracted from the beta plus alpha activity. This is particularly important for samples with high alpha activity.

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7.6   Calculations:

    7.6.1   Calculate the alpha radioactivity by the following equation:

$$\text{Alpha (pCi/liter)} = \frac{A \times 1,000}{2.22 \times C \times V}$$

    where:

        A = net alpha count rate (gross alpha count rate minus the
             background count rate) at the alpha voltage plateau;

        C = alpha efficiency factor, read from the graph (Paragraph
             7.1.3) of efficiency vs. mg of water solids per cm^2 of
             planchet area, cpm/dpm);

        V = volume of sample aliquot (mL); and

      2.22 = conversion factor from dpm/pCi.

    7.6.2   Calculate the beta radioactivity by the following equations:

    7.6.2.1 If there are no significant alpha counts when the sample
is counted at the alpha voltage plateau, the beta activity can be
determined from the following equation:

$$\text{Beta (pCi/liter)} = \frac{B \times 1,000}{2.22 \times D \times V}$$

    where:

        B = net beta count rate (gross alpha count rate minus the
            background count rate at the beta voltage plateau),

        D = beta efficiency factor, read from the graph (Paragraph
            7.1.3) of efficiency vs. mg of water solids per cm^2 of
            planchet area, (cpm/dpm).

        V = volume of sample aliquot (mL).

      2.22 = conversion factor from dpm/pCi.

    7.6.3   When counting beta radioactivity in the presence of alpha
radioactivity by gas-flow proportional counting systems (at the beta
plateau), alpha particles are also counted. Because alpha particles are
more readily absorbed by increasing sample thickness than beta particles,
the alpha/beta count ratios vary with increasing sample thickness.
Therefore, it is necessary to prepare a calibration curve by counting
standards containing americium-241 with increasing thickness of solids on

the alpha plateau and then on the beta plateau, plotting the ratios of the two counts vs. density thickness. The alpha amplification factor (E) from that curve is used to correct the amplified alpha count on the beta plateau. When significant alpha activity is indicated by the sample count at the alpha voltage plateau, the beta activity of the sample can be determined by counting the sample at the beta voltage plateau and calculating the activity from the following equation:

$$\text{Beta (pCi/liter)} = \frac{(B - AE) \times 1{,}000}{2.22 \times D \times V}$$

where:

B = as defined above.

D = as defined above.

A = as defined above.

E = alpha amplification factor, read from the graph of the ratio of alpha counted at the beta voltage/alpha counted at the alpha voltage vs. sample density thickness.

V = volume of sample aliquot (mL).

2.22 = conversion factor from dpm/pCi.

7.7   Errors associated with the results of the analysis should also be reported.

8.0 QUALITY CONTROL

8.1   All quality control data should be maintained and available for easy reference or inspection.

8.2   Employ a minimum of one blank per sample batch to determine if contamination is occurring.

8.3   Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample-preparation and analytical process.

8.4   Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

9.0 METHOD PERFORMANCE

9.1 In a collaborative study of two sets of paired water samples containing known additions of radionuclides, 15 laboratories determined the gross alpha activity and 16 analyzed gross beta activity. The samples contained simulated water minerals of approximately 350 mg fixed solids/L. The alpha results of one laboratory were rejected as outliers.

The average recoveries of added gross alpha activity were 86, 87, 84, and 82%. The precision (random error) at the 95% confidence level was 20 and 24% for the two sets of paired samples. The method was biased low, but not seriously.

The average recoveries of added gross beta activity were 99, 100, 100, and 100%. The precision (random error) at the 95% confidence level was 12 and 18% for the two sets of paired samples. The method showed no bias.

10.0 REFERENCES

10.1 None required.

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                 ( Start )                        (A)                              (B)
                     |                             |                                |
                     v                             v                                v
        +---------------------+        +---------------------+        +---------------------+
        | 7.1 Calibrate using |        |        7.2          |        | 7.6.1 Calculate     |
        | Am-241 for gross    |        |    Does water       |  Yes   | alpha radioactivity.|
        | alpha activity; Sr-90|  <--  |  sample contain     |----> +---------------------+
        | or Cs-137 for gross |        |    chloride         |        | 7.2 Convert to      |
        | beta activity.      |        |     salts?          |        | nitrate salts.      |
        +---------------------+        +---------------------+        +---------------------+
                     |                        | No                              |
                     v                        v                                 v
        +---------------------+        +---------------------+        +---------------------+
        | 7.1.3 Prepare       |        | 7.2 Add HNO3; swirl;|        | 7.6.2 Calculate beta|
        | separate alpha      |        | transfer each aliquot|       | radioactivity.      |
        | and beta particle   |        | to tard planchet;   |        +---------------------+
        | self-absorption     |        | evaporate.          |                 |
        | graphs.             |        +---------------------+                 v
        +---------------------+                 |                          ( Stop )
                     |                          v
                     v                 +---------------------+
        +---------------------+        | 7.3 Dry sample      |
        | 7.1.4 Acidify tap   |        | residue; weigh      |
        | water aliquots with |        | and count.          |
        | HNO3; evaporate;    |        +---------------------+
        | transfer to         |                 |
        | planchet.           |                 v
        +---------------------+        +---------------------+
                     |                 |       7.4           |
                     v                 |       Are           |  Yes   +---------------------+
        +---------------------+        |   hygroscopic       |----> | 7.4 Flame to a      |
        | 7.1.4 Evaporate     |        |     salts           |        | dull red heat.      |
        | and dry: count      |        |    present?         |        +---------------------+
        | alpha, beta         |        +---------------------+                 |
        | residue for reference|               | No                           |
        | standard.           |                v  <--------------------------- 
        +---------------------+        +---------------------+
                     |                 | 7.5 Weigh and       |
                     v                 | count.              |
        +---------------------+        +---------------------+
        | 7.2 Transfer aliquot|                 |
        | of water sample to  |                 v
        | beaker; evaporate.  |                (B)
        +---------------------+
                     |
                     v
                    (A)
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