Designation: D7535 – 09 (Reapproved 2015)

Standard Test Method for Lead-210 in Water[1]

This standard is issued under the fixed designation D7535; 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 determination of radioactive ^{210}Pb in environmental water samples (for example, drinking, non-process and effluent waters) in the range of 37 mBq/L (1.0 pCi/L) or greater.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information purposes only.

1.3 This method has been used successfully with tap water. It is the user's responsibility to ensure the validity of this test method for samples larger than 500 mL and for waters of untested matrices.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazards statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 Closed Conduits

D4448 Guide for Sampling Ground-Water Monitoring Wells

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

4. Summary of Test Method

4.1 This test method is based on the utilization of solid phase extraction of lead from water samples with detection of the radioactive lead by beta gas flow proportional counting. An aliquot of the sample is measured into a beaker; iron carrier and lead carrier are added. Lead is scavenged by an iron hydroxide precipitation. Lead is then selectively sorbed on a solid phase extraction column and eluted with water. Lead is precipitated as lead sulfate and is collected on a filter paper. The lead sulfate precipitate is covered with aluminum foil and held for 5 days or longer for ^{210}Bi ingrowth. It is then counted for beta radiation on a gas flow proportional counter.

5. Significance and Use

5.1 This test method was developed to measure the concentration of ^{210}Pb in nonprocess water samples. This test method may be used to determine the concentration of ^{210}Pb in environmental samples.

6. Interferences

6.1 Significant amounts of stable lead (>0.3 mg/L) present in the sample will interfere with the chemical yield determination, leading to positive bias in the yield. If it is known or suspected that natural lead is present in the sample, blank sample aliquots to which no lead carrier content is added should be analyzed. The amount of natural lead contained in the sample shall be used to correct the yield.

6.2 In most cases measurable amounts of ^{210}Pb will be present in the Pb carrier used in 12.1. This additional contribution to the measured sample activity must be determined and treated as additional background activity. For each new carrier solution, the inherent ^{210}Pb in the carrier must be measured. A brief description of this additional background calibration is given in 11.2. Accurate determination of the combined standard uncertainty and the minimum detectable concentration should include this additional activity in the method background count rate.

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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.

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[2] 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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6.3 Previous experimental data suggests that barium concentrations of approximately 30 ppm can be a source of interference, leading to chemical yield determinations that may be non-representative and outside normal acceptance criteria.

7. Apparatus

7.1 *Analytical Balance, 0.0001 g.*

NOTE 1—A thickness of aluminum of approximately 0.003 in. (areal density of approximately 0.02 mg/cm^2) is needed to effectively eliminate the 60 keV beta particles from ^{210}Pb.[3] The thickness of aluminum foil used during calibration of the detector and measurement samples must be held constant to maintain self-absorption of ^{210}Pb and ^{210}Bi beta particles at a constant level.

7.2 *Aluminum foil.*

7.3 *Centrifuge.*

7.4 *Centrifuge tubes (50 mL plastic).*

7.5 *Filters, 25 mm polypropylene,* 0.1 µm, with polycarbonate base and metal screen.

7.6 *Filter apparatus,* polysulfone funnel and 100 mL polypropylene flask.

7.7 *Beta Gas Flow Proportional Counting System,* (<1.0 cpm beta), low background.

7.8 *Glass stir rods.*

7.9 *Glass beakers.*

7.10 *Hot plate.*

7.11 *Petri dish.*

7.12 *Planchets,* stainless steel, flat, with diameter large enough to hold the 25 mm filter.

7.13 *Tweezers.*

7.14 *Watch glass.*

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. Other grades may be used, provided that the reagent is of sufficiently high purity to permit its use without increasing the background of the measurement. Some reagents, even those of high purity, may contain naturally occurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths and potassium compounds and/or artificially produced radionuclides. Consequently, when such reagents are used in the analysis of low-radioactivity samples, the activity of the reagents shall be determined under analytical conditions that are identical to those used for the sample. The activity contributed by the reagents may be considered to be a component of background and applied as a correction when calculating the test sample result. This increased background reduces the sensitivity of the measurement.

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

8.3 *Ammonium hydroxide*—15 M NH_4OH, (concentrated reagent).

8.4 *Iron Carrier (20 mg/mL)*—Dissolve 9.6 g of ferric chloride ($FeCl_3 \cdot 6H_2O$) in 70 mL of 0.5 M HCl and dilute to 100 mL with 0.5 M HCl.

8.5 *Nitric Acid, 16 M HNO_3*—(concentrated reagent) (sp gr 1.42).

8.5.1 *Nitric Acid, 8 M HNO_3*—Add 500 mL of concentrated nitric acid to 400 mL water. Dilute to 1 L with water and mix well.

8.5.2 *Nitric Acid, 1 M HNO_3*—Add 63 mL of concentrated nitric acid to 800 mL water. Dilute to 1 L with water and mix well.

8.5.3 *Nitric Acid, 0.1 M HNO_3*—Add 6.4 mL of concentrated nitric acid to 600 mL water. Dilute to 1 L with water and mix well.

8.6 *Lead Carrier (10 grams Pb/L)*—Dissolve 1.60 g of lead nitrate in water and dilute to 100 mL with water.

8.7 *Lead Extraction Chromatography Column,* 2-mL bed volume, 100–150 µm particle size.[4]

8.8 *Pb-210 Standardizing Solution*—Traceable to a national standardizing laboratory such as National Institute of Standards and Technology, Gaithersburg, MD, USA (NIST) or National Physical Laboratory, Teddington, Middlesex, UK, (NPL) with less than 0.1 mg of stable lead per mL of final solution with a typical ^{210}Pb concentration range from 85 to 125 Bq/mL. The ^{210}Pb calibration source should be in equilibrium with its progeny ^{210}Bi.

8.9 *Sulfuric Acid, 18 M H_2SO_4*—concentrated reagent (sp gr 1.84).

9. Hazards

9.1 Use extreme caution when handling all acids. They are extremely corrosive, and skin contact could result in severe burns.

9.2 When diluting concentrated acids, always use safety glasses and protective clothing, and add the acid to the water.

10. Sampling

10.1 Collect a sample in accordance with Guides D4448 and D6001 and Practice D3370, or other documented procedure as appropriate.

11. Calibration

11.1 *Detection Efficiency Calibration (see also Practice D7282):*

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[3] Radiological Health Handbook, Revised Editions January 1970, Compiled and Edited by the Bureau of Radiological Health and the Training Institute Environmental Control Administration, US Department of Health, Education and Welfare.

[4] Resin available in bulk form and in prepacked columns or cartridges from Eichrom Technologies LLC, Lisle, IL 60532. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

11.1.1 Prepare a set of three working calibration sources according to the calibration procedure outlined in the subsequent steps.

11.1.2 Pipet 1.0 mL of lead carrier into a small beaker.

11.1.3 Add 1.0 mL of ^{210}Pb standardizing solution to the beaker and evaporate to near dryness on a hot plate at a low setting.

11.1.4 Redissolve the residue in 10 mL of 1 M HNO_3.

11.1.5 Follow the steps described in 12.9 through 12.28.

11.1.6 Count to amass at least 10 000 counts. Record the time and date of the midpoint of this counting period as t_m.

11.1.7 *Detection Efficiency Calculation*—Calculate the detection efficiency for ^{210}Bi for each calibration source and each detector using Eq 1.

$$\varepsilon_{Bi} = \frac{R_a - R_b}{c_s \times V_s \times DF \times IF \times Y_{Pb}} \tag{1}$$

where:

ε_{Bi} = detection efficiency for ^{210}Bi,
R_a = total count rate (s^{-1}) for calibration source, (counts divided by count time (s))
R_b = count rate (s^{-1}) for blank or background source, (counts divided by count time (s))
IF = correction factor for ingrowth of ^{210}Bi from ^{210}Pb between the time of the bismuth separation to the midpoint of counting, $1 - e^{-\lambda_{Bi}(t_m - t_s)}$, where:
  λ_{Bi} = decay constant for ^{210}Bi, (1.60×10^{-6} s^{-1}),
  t_s = date and time of ^{210}Bi separation, and
  t_m = midpoint of count of calibration mount (date and time).
DF = correction factor for decay of ^{210}Pb in the carrier solution from the carrier activity reference date to the time of the bismuth separation, $e^{-\lambda_{Pb}(t_s - t_r)}$, where:
  λ_{Pb} = decay constant for ^{210}Pb, (9.85×10^{-10} s^{-1}),
  t_r = reference date and time of ^{210}Pb calibration of lead carrier, and
  t_s = date and time of ^{210}Bi separation.
c_s = activity concentration (or massic activity) of the calibration standard solution (Bq/unit),
V_s = volume (or mass) of standard solution used (mL or g),
Y_{Pb} = chemical yield of lead (see 13.1 and 13.2); $Y_{Pb} = m_s/(c_c \times V_c)$, where:
  m_s = net mass (mg) of lead sulfate found in the source, equal to $m_{F+P} - m_F$, where m_{F+P} is the total mass of the filter plus precipitate and m_F is the tare mass (filter only),
  c_c = mass concentration (mg/mL) of lead (as sulfate) in the carrier solution (see Eq 5), and
  V_c = volume of carrier solution added (mL).

Eq 1 assumes that any ^{210}Pb activity contamination in the Pb carrier is insignificant compared to the amount of ^{210}Pb activity in the calibration source spike.

11.1.8 The variance in the ^{210}Bi detector efficiency, ε_{Bi}, (not the average) is calculated by Eq 2:

$$u_c^2(\varepsilon_{Bi}) = \left(\frac{u^2(R_a) + u^2(R_b)}{m_s^2 \times IF^2} + \left(\frac{R_a - R_b}{m_s \times IF} \right)^2 \times \frac{u^2(m_s)}{m_s^2} + u^2(\hat{F}_c) \times DF_c^2 \right) \times \frac{c_c^2 \times V_c^2}{c_s^2 \times V_s^2 \times DF^2} + \varepsilon_{Bi}^2 \times \left(\frac{u^2(c_s)}{c_s^2} + \frac{u^2(V_s)}{V_s^2} + \frac{u^2(c_c)}{c_c^2} + \frac{u^2(V_c)}{V_c^2} \right) \tag{2}$$

where u(·) denotes standard uncertainty; for example, $u(V_s)$ is the standard uncertainty of V_s.

11.1.8.1 Eq 2 omits the uncertainties due to the activity concentration of the calibration standard solution, c_s, and the concentration of the carrier solution, c_c. These uncertainty components are accounted for when the average efficiency is determined (below).

11.1.8.2 The procedure requires an average of (at least) three efficiencies ($N = 3$) measured using three calibration sources on each detector. For this purpose, calculate ε_1, ε_2, ε_3 for each source, and use Eq 3 to calculate the weighted average:

$$\bar{\varepsilon}_{Bi} = \frac{1}{N} \sum_{i=1}^{N} \varepsilon_i \tag{3}$$

11.1.8.3 To calculate the uncertainty of the average, including uncertainty components due to systematic errors, Eq 4 is used:

$$u(\bar{\varepsilon}_{Bi}) = \sqrt{\frac{1}{N^2} \sum_{i=1}^{N} u_c^2(\varepsilon_i) + \bar{\varepsilon}_{Bi}^2 \times \left(\frac{u^2(c_s)}{c_s^2} + \frac{u^2(c_c)}{c_c^2} \right)} \tag{4}$$

11.2 *Determination of Inherent ^{210}Pb in Pb Carrier:*

11.2.1 As noted in 6.2, in cases where the Pb carrier used in this procedure has not been determined to be free of ^{210}Pb, the ^{210}Pb content inherent in the lead carrier used in this method must be evaluated. Each new carrier solution must be measured for ^{210}Pb prior to use. Once the ^{210}Pb content of the lead carrier is known, the ^{210}Pb count rate from the recovered lead carrier can be calculated for each sample analyzed. The following step provides a protocol to determine the inherent 210Pb in the lead carrier and Section 13 provides the equations needed to correct for the ^{210}Pb inherent in the lead carrier. The specific number of samples, as well as acceptance criteria for the data and statistical tests to determine detectable ^{210}Pb in the lead carrier, should be specified in the laboratory's Quality Systems Manual.

11.2.2 To a minimum of five 50 mL centrifuge tubes, add 1 mL of the Pb carrier solution.

11.2.3 Add 20 mL of water and 4 mL concentrated sulfuric acid to each centrifuge tube.

11.2.4 Continue with the normal sample preparation, beginning at 12.16.

11.2.5 After counting, calculate for each sample the ^{210}Pb concentration and associated uncertainty according to Section 13. Calculate the weighted average $\hat{a}_c$ and standard uncertainty $u(\hat{a}_c)$ of the ^{210}Pb concentration using the calculated sample results from the data set using the equations in 13.3.

12. Procedure

NOTE 2—See Fig. 1 for a diagram of the procedure.

12.1 Add 1.0 mL of lead carrier and 1.0 mL of iron carrier to a maximum 500 mL of sample. Acidify sample with nitric acid to pH 2.

12.2 Cover beaker with a watch glass and heat at near boiling for an hour. Reduce heat.

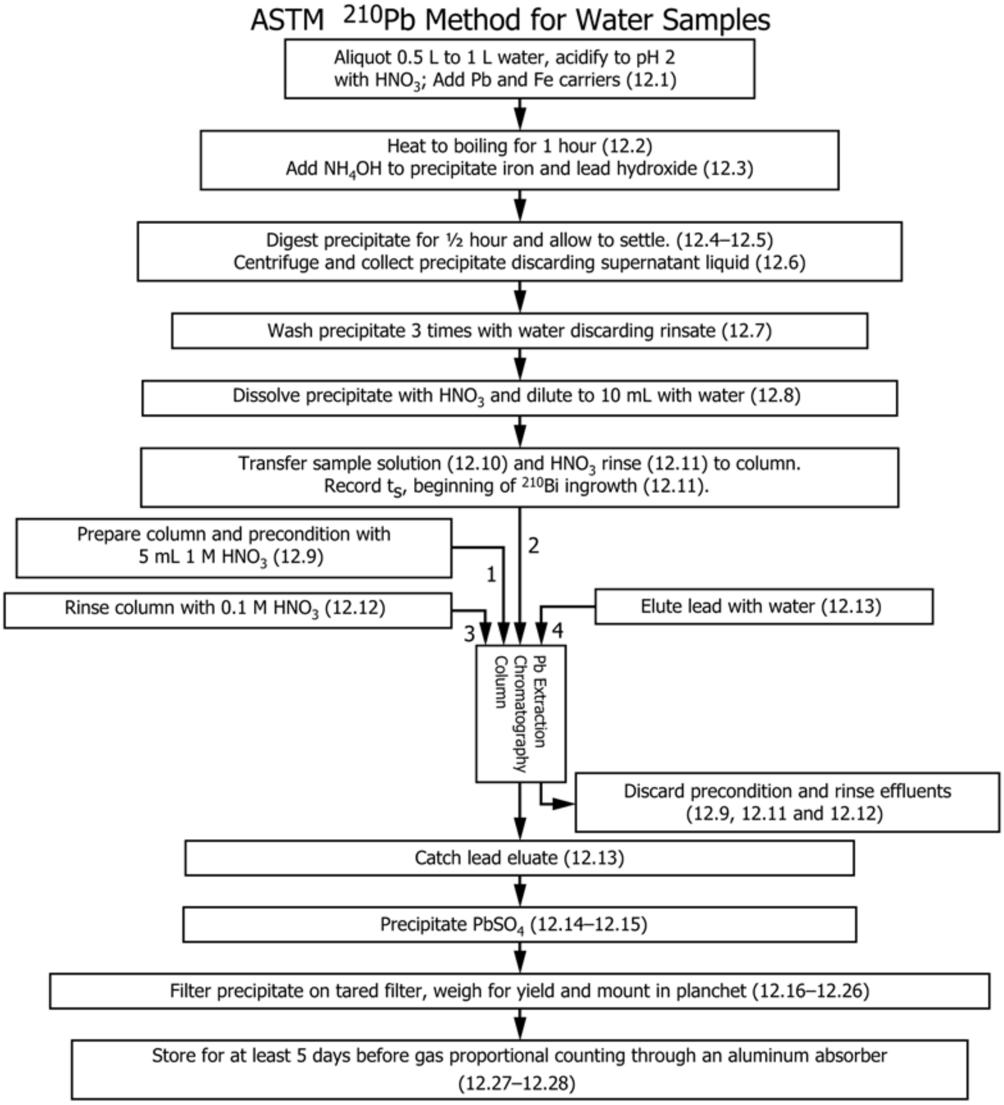

FIG. 1 ^{210}Pb Method for Water Samples

12.3 Remove watch glass. Carefully, with stirring, add approximately 12 mL of concentrated ammonium hydroxide to precipitate iron hydroxide. Add more if necessary to form a precipitate.

12.4 Heat the sample without boiling for another 30 minutes.

12.5 Let the precipitate settle for at least 2 hours (preferably overnight). Decant the supernatant. Transfer the remaining sample along with the precipitate into a 50 mL plastic centrifuge tube.

12.6 Centrifuge at approximately 3500 RPM for approximately 15 minutes and discard the supernatant.

12.7 Wash the precipitate with 10 mL of water. Centrifuge and discard the supernatant. Repeat this step two more times to ensure removal of ammonia prior to the next step.

12.8 Dissolve residue in 1.2 mL of 8 M HNO_3, then dilute to a final volume of 10 mL with water, bringing the final solution to a concentration of 1 M HNO_3.

12.9 Prepare a lead extraction chromatography column by removing the bottom plug and the cap. Press the top frit down snugly to the resin surface using a glass rod (or equivalent) and let the water drain out. Add 5 mL of 1 M HNO_3 and allow the solution to drain by gravity.

12.10 Carefully transfer the sample solution to the reservoir of the column.

12.11 Rinse centrifuge tube with 10 mL of 1 M HNO_3 and add to the column after the feed has passed through. Record the time and date of this rinse. This will be the start of ^{210}Bi ingrowth, t_s.

12.12 Rinse column with 10 mL of 0.1 M HNO_3.

12.13 Elute the lead with 20 mL of water into a plastic centrifuge tube.

12.14 Very carefully add 4 mL of sulfuric acid to each lead eluant. Allow the solution to cool for 10–15 minutes.

NOTE 3—Bismuth-210 may not be quantitatively precipitated with lead in the subsequent step. Minimizing the time elapsed between the elution and precipitation of lead sulfate to less than 60 min will ensure that potential bias imparted due to incomplete precipitation of ^{210}Bi will be less than 1 %.

12.15 Cap the tubes and mix the solution well. A white precipitate is formed. Allow samples to cool for 30 minutes.

12.16 Weigh the polypropylene filters on an analytical balance to the nearest 0.1 mg and record the weight.

12.17 Place the tared filter on a filter apparatus with a 100 mL polysulfone funnel and Erlenmeyer flask.

12.18 Add 3–5 mL of 80 % ethanol to each filter, applying vacuum and ensuring there is no leak along the sides. Add 2–3 mL of water to each filter.

12.19 Gently shake the tube containing the sample and transfer the solution into the filtering funnel.

12.20 Rinse sample tube with 5 mL of water and transfer into the filtering funnel.

12.21 Rinse sides of the funnel thoroughly with water.

12.22 Rinse the funnel with 4–5 mL of 80 % ethanol.

12.23 Remove filter, place it in a plastic Petri dish and dry under a heating lamp for few minutes.

12.24 Reweigh the filter and record the weight.

12.25 Clean a planchet with paper towel moistened with alcohol. Wipe the planchet and let it dry.

12.26 Mount the filter on the center of the planchet using a glue stick or equivalent.

12.27 Cover the planchet with a piece of aluminum foil and wait at least five days for ^{210}Bi ingrowth.

12.28 Count the sample for 12 000 seconds (200 minutes) on a calibrated low background beta gas flow proportional counting system.

13. Calculations

13.1 *Mass concentration of lead (as sulfate) in the carrier solution*—The stoichiometric model for calculating c_c and the associated uncertainty $u(c_c)$, is provided below:

$$c_c = \frac{m_{Pb(NO_3)_2}}{V} \times \frac{M_r(PbSO_4)}{M_r(Pb(NO_3)_2)} = \frac{m_{Pb(NO_3)_2}}{V} \times 0.9156 \qquad (5)$$

where:

c_c = mass concentration (mg/mL) of lead (as sulfate) in the carrier solution,

$m_{Pb(NO_3)_2}$ = mass (mg) of lead nitrate used to prepare the lead carrier solution,

V = total volume (mL) of carrier solution prepared,

$M_r(Pb(NO_3)_2)$ = formula weight of lead nitrate (331.2),

$M_r(PbSO_4)$ = formula weight of lead sulfate (303.3).

and

$$u(c_c) = c_c \times \sqrt{\frac{u^2(m_{Pb(NO_3)_2})}{m^2_{Pb(NO_3)_2}} + \frac{u^2(V)}{V^2}} \qquad (6)$$

where:

$u(m_{Pb(NO_3)_2})$ = standard uncertainty of the $Pb(NO_3)_2$ mass, and

$u(V)$ = standard uncertainty of the volume of carrier.

13.2 *Chemical yield of lead*—Assuming there is little or no lead in the sample, the chemical yield is given by:

$$Y_{Pb} = \frac{m_s}{c_c \times V_c} \qquad (7)$$

where:

Y_{Pb} = chemical yield of lead,

m_s = net mass (mg) of lead sulfate found in the source, equal to $m_{F+P} - m_F$, where m_{F+P} is the total mass of the filter plus precipitate and m_F is the tare mass (filter only),

c_c = mass concentration (mg/mL) of lead (as sulfate) in the carrier solution, and

V_c = volume of carrier solution added (mL).

13.3 *^{210}Pb Contamination in the Lead Carrier*—The ^{210}Pb contamination in the lead carrier (a_c) on the activity reference date (t_r) is defined as:

$$a_c = \frac{R_a - R_b}{m_s \times \bar{\varepsilon}_{Bi} \times DF_c \times IF} \qquad (8)$$

where:

a_c = ^{210}Bi activity per unit mass of lead sulfate (Bq/mg) obtained from the lead carrier, as of the carrier activity reference date,

R_a = total count rate (s^{-1}) for source (counts divided by count time, t_a (s)),

R_b = count rate (s^{-1}) for blank or background source (counts divided by count time, t_b (s)),

m_s = net mass (mg) of lead sulfate found in the source,

DF_c = correction factor for decay of ^{210}Pb in the carrier solution from the carrier activity reference date, t_r, to the time of the bismuth separation, and

IF = correction factor for ingrowth of ^{210}Bi from ^{210}Pb between the time of the bismuth separation to the midpoint of counting, $1-e^{-\lambda_{Bi}(t_m-t_s)}$, where:

λ_{Bi} = decay constant for ^{210}Bi, (1.60×10^{-6} s^{-1}),

t_s = date and time of ^{210}Bi separation, and

t_m = midpoint of count of calibration mount (date and time).

NOTE 4—The lead carrier activity reference date (t_r) is typically chosen as the assay date and time of the first of the five lead carrier samples assayed.

13.3.1 The standard uncertainty $u(a_c)$ of each a_c is calculated using:

$$u(a_c) = \frac{\sqrt{R_a/t_a + R_b/t_b}}{m_s \times \bar{\varepsilon}_{Bi} \times DF_c \times IF} \qquad (9)$$

13.3.2 The method requires preparing at least 5 sources and calculating a weighted average:

$$\hat{a}_c = \frac{\sum_{i=1}^{n} \frac{a_{c,i}}{u^2(a_{c,i})}}{\sum_{i=1}^{n} \frac{1}{u^2(a_{c,i})}} \qquad (10)$$

13.3.3 The standard uncertainty of the weighted average $u(\hat{a}_c)$ is calculated using:

$$u(\hat{a}_c) = \left(\sum_{i=1}^{n} \frac{1}{u^2(a_{c,i})} \right)^{-½} \tag{11}$$

13.3.4 The major uncertainty components should be those due to R_a and R_b (counting statistics), unless the concentration of ^{210}Pb in the lead nitrate is inhomogeneous. If there is inhomogeneity, the uncertainty may be larger. The uncertainty of m_s is considered to be negligible.

NOTE 5—The value of â must be measured for each lot of $Pb(NO_3)_2$ used. When a new batch of lead carrier solution is prepared from the same lot of lead nitrate, continue to use the same carrier assay reference date (t_r) for subsequent decay corrections (use the DF_c factor).

13.4 *Count Rate from Sample Source Due to ^{210}Pb Contamination in the Carrier Added*—The count rate (R_c) from sample source due to ^{210}Pb contamination in the carrier added is calculated by Eq 12.

$$R_c = \hat{a}_c \times m_s \times \bar{\varepsilon}_{Bi} \times DF_c \times IF \tag{12}$$

where:

R_c = count rate (s^{-1}) due to ^{210}Pb contamination in the carrier,

$\hat{a}_c$ = ^{210}Bi activity per unit mass of lead sulfate (Bq/mg) obtained from the lead carrier, as of the carrier activity reference date (weighted average),

m_s = net mass (mg) of lead sulfate found in the source (due to carrier),

$\bar{\varepsilon}_{Bi}$ = average ^{210}Bi detector efficiency,

DF_c = correction factor for decay of ^{210} Pb in the carrier solution from the carrier activity reference date to the time of the bismuth separation, $1 - e^{-\lambda_{Pb}(t_s - t_r)}$, where:

λ_{Pb} = decay constant for ^{210}Pb, (9.85×10^{-10} s^{-1}),

t_r = reference date and time of ^{210}Pb assay of lead carrier, and

t_s = date and time of ^{210}Bi separation.

IF = correction factor for ingrowth of ^{210}Bi from ^{210}Pb between the time of the bismuth separation to the midpoint of counting, $1 - e^{-\lambda_{Bi}(t_m - t_s)}$

13.5 *Calculation of Lead-210 Activity Concentration (AC_{Pb}) in Sample*—The ^{210}Pb activity concentration (Bq/L) in a sample is calculated by either Eq 13 or Eq 14 (these two equations are equivalent).

$$AC_{Pb} = \left(\frac{R_a - R_b}{m_s \times \bar{\varepsilon}_{Bi} \times IF} - \hat{a}_c \times DF_c \right) \times \frac{c_c \times V_c}{V_a \times DF} \tag{13}$$

$$AC_{Pb} = \frac{R_a - R_b - R_c}{\bar{\varepsilon}_{Bi} \times V_a \times DF \times IF \times Y_{Pb}} \tag{14}$$

where:

AC_{Pb} = activity concentration (Bq/L) of ^{210}Pb in the sample,

R_a = total count rate (s^{-1}) for sample (counts divided by count time, t_a (s)),

R_b = count rate (s^{-1}) for blank or background source (counts divided by count time, t_b (s)),

m_s = net mass (mg) of lead sulfate found in the source,

IF = correction factor for ingrowth of ^{210}Bi from ^{210}Pb between the time of the bismuth separation (t_s) to the midpoint of counting (t_m); $1 - e^{-\lambda_{Bi}(t_m - t_s)}$

$\hat{a}_c$ = ^{210}Bi activity per unit mass of lead sulfate (Bq/mg) obtained from the lead carrier, as of the carrier activity reference date,

DF_c = correction factor for decay of ^{210}Pb in the carrier solution from the carrier activity reference date (t_r) to the time of the bismuth separation (t_s), $e^{-\lambda_{Pb}(t_s - t_r)}$

$\bar{\varepsilon}_{Bi}$ = average detection efficiency for ^{210}Bi,

V_a = volume of the sample aliquant analyzed (L),

DF = correction factor for decay of ^{210}Pb in the sample from the collection date (t_c) to the time of the bismuth separation, $e^{-\lambda_{Pb}(t_s - t_c)}$

c_c = mass concentration (mg/mL) of lead (as sulfate) in the carrier solution,

V_c = volume of carrier solution added (mL),

R_c = count rate (s^{-1}) due to ^{210}Pb contamination in the carrier (Eq 12), and

Y_{Pb} = chemical yield of lead (Eq 7).

13.6 *Sample Aliquant Count Rate Uncertainty*—To calculate the standard uncertainty of AC_{Pb} due to counting statistics only, use Eq 15.

$$u_{cC}(AC_{Pb}) = \frac{\sqrt{R_a/t_a + R_b/t_b}}{\bar{\varepsilon}_{Bi} \times V_a \times DF \times IF \times Y_{Pb}} \tag{15}$$

where:

$u_{cC}(AC_{Pb})$ = standard uncertainty (Bq/L) in AC_{Pb} due to counting statistics only,

t_a = counting time for sample (s),

t_b = counting time for blank or background (s).

13.7 *Combined Standard Uncertainty of AC_{Pb}*—The combined standard uncertainty of AC_{Pb} is calculated using Eq 16.

$$u_{cC}(AC_{Pb}) = \Bigg[u_{cC}^2(AC_{Pb}) + \left(\left(\frac{R_a - R_b}{m_s \times \bar{\varepsilon}_{Bi} \times IF} \right)^2 \times \frac{u^2(\bar{\varepsilon}_{Bi})}{\bar{\varepsilon}_{Bi}^2} + u^2(\hat{a}_c) \times DF_c^2 \right) \times \frac{c_c^2 \times V_c^2}{V_a^2 \times DF^2} + AC_{Pb}^2 \times \left(\frac{u^2(V_a)}{V_a^2} + \frac{u^2(c_c)}{c_c^2} + \frac{u^2(V_c)}{V_c^2} \right) \Bigg]^{1/2} \tag{16}$$

The use of this equation assumes that the uncertainty of m_s is negligible and the effect of the correlation between $\hat{a}_c$ and $\bar{\varepsilon}_{Bi}$ on the combined standard uncertainty is negligible.

13.8 *Detection Decision*[5]—The decision level or critical level is defined as the minimum measured value (for example, of the instrument signal or the analyte concentration) required to give confidence (95 % in this case) that a positive (nonzero) amount of analyte is present in the material analyzed. The detection decision may be based on the measured net count rate (corrected for background and carrier contamination):

$$S = R_a - R_b - R_c \tag{17}$$

where R_c is as described earlier. The critical value of S is given by:

$$S_C = 1.645 \sqrt{R_b \times \left(\frac{1}{t_a} + \frac{1}{t_b} \right) + \frac{R_c}{t_a} + u^2(R_c)} \tag{18}$$

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[5] If R_c is measured with good precision so that $u^2(R_c) < R_c / t_a$, the $u(R_c)$ term in the equations for the critical value and MDC may be ignored. The $u^2(R_c)$ value and nominal MDC should be calculated every time R_c is measured for a new carrier.

13.9 *"A Priori" Minimum Detectable Concentration (MDC)*[5]—The nominal MDC (Bq/L) for ^{210}Pb, taking into account the ^{210}Pb in the carrier, is calculated using Eq 19.

$$MDC = \frac{\frac{2.71}{t_a} + 3.29\sqrt{R_b \times \left(\frac{1}{t_a} + \frac{1}{t_b}\right) + \frac{R_c}{t_a} + u^2(R_c)}}{\bar{\varepsilon}_{Bi} \times V_a \times DF \times IF \times Y_{Pb}} \quad (19)$$

14. Quality Control

14.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test. The batch size should not exceed 20 samples, not including QC samples.

14.2 *Calibration and Calibration Verification (see also Practice D7282)*—The standards used in this method shall be traceable to a national standards laboratory (such as NIST in the USA).

14.3 The chemical yield of the lead carrier shall be calculated for each sample and associated QC sample. The chemical yield should be reported along with the reported analytical data.

14.4 *Initial Demonstration of Laboratory/Instrument Capability:*

14.4.1 If a laboratory or analyst has not performed this test before or if there has been a major change in the measurement system, for example, significant instrument change, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory/instrument capability.

14.4.2 Analyze seven replicates of a standard solution prepared from an IRM (independent reference material) containing ^{210}Pb at concentrations sufficient to minimize counting uncertainty to less than 2.5 % relative standard uncertainty. Each replicate must be taken through the complete analytical test method including any sample pretreatment steps. The matrix used for the demonstration should represent a water sample typical for which the method will be used, for example, surface water.

14.4.3 Calculate the mean and standard deviation of the seven replicate values and compare to the acceptable ranges of precision and mean bias of 15 % and ±15 % respectively, at the spiking level suggested in 14.4.2. Test Method D5847 should be consulted on the manner by which the precision and mean bias are determined from the initial demonstration study. This method shall not be used for official samples until precision and bias requirements are met.

14.4.4 Analyze three replicates of a blank (in ^{210}Pb) solution matrix. The matrix used for the demonstration should represent a water sample typical for which the method will be used, for example, surface water. The total dissolved solids (TDS) of the matrix should approximate that which may be encountered in normal use.

14.4.5 Calculate the ^{210}Pb activity for each of these three blank solutions. This method shall not be used for official samples until the results of each of three blank solutions are below the decision level (critical level).

14.5 *Laboratory Control Sample (LCS):*

14.5.1 To ensure that the test method is in control, analyze an LCS with each batch of no more than 20 samples. The activity added to reagent water should be appropriate for the type of samples analyzed and allow sufficient precision to ensure a meaningful assessment of accuracy. The LCS must be taken through all steps of the analytical method including sample preservation and pretreatment. The result obtained for the LCS shall fall within the limit of ±25 % of the expected value.

14.5.2 If the result is not within these limits reporting of the results is halted until the problem is resolved. An indication of the occurrence should accompany the reported results.

14.6 *Method Blank (Blank)*—Analyze a reagent water test blank with each batch of no more than 20 samples. The concentration of ^{210}Pb analyte found in the blank should be less than the decision level (critical level). If the concentration of the ^{210}Pb analyte is above the limit, provide an explanation in the case narrative.

14.7 *Matrix Spike (MS):*

14.7.1 The performance of a matrix spike analysis with every batch is not required given the use of a carrier with each sample. The carrier chemical yield would indicate any problems with interferences in a specific sample matrix. 14.2 addresses the use of the carrier chemical yield as a measure of result quality.

14.8 *Duplicate:*

14.8.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch of no more than 20 samples. Calculate the statistical agreement between the two results to ensure they agree with a 99 % confidence level. This calculation is performed using the determined standard uncertainty associated with each result.

14.8.2 In those cases where there is insufficient sample volume to allow performance of a duplicate sample analysis, a duplicate LCS (LCS-D) shall be performed.

14.8.3 If the result is not within these limits, all samples in the batch must be reanalyzed or the results must be explained in the case narrative.

14.9 *Independent Reference Material (IRM):*

14.9.1 In every quarter that the method is performed, verify the quantitative value produced by the test method by analyzing an IRM submitted on at least single-blind basis (if practical) to the laboratory. The concentration of analyte in the national standards laboratory traceable reference material should be appropriate to the typical purpose for which the method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

15. Precision and Bias[6]

15.1 A collaborative study was conducted with 10 participating labs. The labs analyzed water samples prepared at four activity levels, including blanks and three positive levels. See Table 1.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1181. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Observed ^{210}Pb Precision and Bias Data

Added (Bq/L)	Average Recovery	% Bias	Single operator standard deviation		Total standard deviation	
c	$\bar{X}$		s_o	s_o %	s_T	s_T %
0	0.004	—	0.027	—	0.028	—
0.111	0.086	–22.5	0.019	17.1	0.034	30.3
0.259	0.223	–13.7	0.024	9.2	0.047	18.1
0.370	0.330	–10.8	0.018	4.7	0.055	14.9
0.592	0.544	–8.1	0.043	7.2	0.068	11.5

NOTE 6—Results of this collaborative study may not be typical of results for matrices other than those studied.

15.2 All results from one laboratory were excluded because it deviated significantly from the method.

15.3 With the approval of ASTM Committee D19, the study design used duplicate samples to assess precision, and not Youden pairs as specified by Practice D2777. Consequently, calculations of bias and precision were done in accordance with Practice E691 rather than Practice D2777.

NOTE 7—The precision and bias data offered in this method are aggregate results from ten laboratories. Performance at several laboratories was better, while performance at one laboratory was poorer than the aggregate (although not determined to be a statistical outlier). It is noted that laboratories participating in the round-robin study may not have had prior experience with the method, nor did they have opportunity to perform an Initial Demonstration of Capability prior to their participation in round-robin testing. Review of the method performance data from the round robin study indicates that this method is capable of precision and bias characteristics that exceed the performance indicated in the method precision and bias statement. The precision and bias study performed by a laboratory during the Initial Demonstration of Laboratory / Instrument Capability (14.4) will provide the best indication of the performance of this method at a laboratory.

16. Keywords

16.1 extraction chromatography; lead-210; radioactive lead; radioactivity; radiochemistry

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