National Institute of Standards & Technology

Certificate

Standard Reference Material® 4354

Freshwater Lake Sediment Environmental Radioactivity Standard

This Standard Reference Material (SRM), which has been developed in cooperation with member laboratories of the International Committee for Radionuclide Metrology, is intended for use in tests or measurements of environmental radioactivity contained in matrices similar to the sample, for evaluating analytical methods, or as a generally available calibrated "real" sample matrix in interlaboratory comparisons. A unit of SRM 4354 consists of approximately 25 g of freeze-dried, pulverized freshwater lake sediment (gyttja) in a polyethylene bottle.

Certified Values: The certified properties for the freshwater lake sediment environmental radioactivity standard are presented in Tables 1 and 2. NIST certified values, as used within the context of this certificate, are values for which NIST has the highest confidence in its uncertainty assessment. They are consensus values, obtained from a thorough statistical evaluation based on different activity measurement methods as obtained by NIST and outside collaborating laboratories. Each reporting laboratory maintains its own traceability to the derived SI unit, the becquerel (Bq).

Expiration of Certification: The certification of **SRM 4354** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored properly and that no change in composition has occurred. Periodic recertification of this SRM is not required. This SRM should be handled in accordance with instructions given in this certificate (see "Instructions for Handling and Storage"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Radiological and Chemical Hazard: Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for radiological and chemical hazard information.

This SRM was prepared in the Center for Radiation Research, Nuclear Radiation Division, Radioactivity Group Coordination of the technical measurements leading to the certification of this SRM was performed by D.D. Hoppes, formerly of NIST.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Technical Contacts: Jerome LaRosa (e-mail jerome.larosa@nist.gov; phone: 1-301-975-8333) and Jacqueline Mann (e-mail: jacqueline.mann@nist.gov; phone: 1-301-975-4472) NIST, Mail Stop 8462, Gaithersburg, MD 20899-8462, fax 1-301-926-7416.

Lisa R. Karam, Chief
Radiation Physics Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 December 2016
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING AND STORAGE

Handling and Storage: The SRM should be stored in a dry location at room temperature. The bottle should be shaken before opening in a chemical hood, and then recapped tightly as soon as any subsamples have been removed. This SRM is a dried, sterilized fresh lake sediment; however, inhalation or ingestion of this material should be avoided. The bottle (or any subsequent container) should always be clearly marked. If the SRM is transported, it should be packed, marked, labeled, and shipped in accordance with applicable local, national, and international regulations.

Working samples of this SRM should be dried in air at 40 °C for at least 24 h prior to weighing. The material has been tested to a minimum sample size of 30 g for which it has been found to be homogeneous. Furthermore, the material has been tested at a sample size of two grams for which actinide radionuclides have been found to be homogeneous.

Details of SRM Preparation: This SRM contains low levels of anthropogenic and natural radioactivity. The sediment was pulverized with a "pancake"-style air-jet mill and radiation sterilized. The average particle size for the resulting powder is 8 µm, and more than 99 %, by weight, of the particles are less than 20 µm in diameter.

Certified Values: Certified values are those measured by two or more methods and/or two or more laboratories. The $^{239+240}$Pu contained is recoverable by normal HNO_3-HCl leaching procedures as well as by the more vigorous methods of chemical treatment listed below. If additional data become available, other radionuclides may be certified and purchasers will be notified. To aid in these certifications, users are requested to send their measurement results for uncertified radioactivities, together with the methods used, to the technical contacts listed on page 1.

Table 1. Certified Properties of SRM 4354

Radionuclides	**See Table 2**
Reference time	**14 February 1986**
Certified massic activities	**See Table 2**
Uncertainties	**See Table 2**

Table 2. Certified Massic Activity Values for SRM 4354

Radionuclide	Massic Activity ($Bq \cdot g^{-1}$)	Tolerance Limits[a] (%)	Standard Deviation (%)	Number of Measurements	Analytical Methods[b,c]
^{60}Co	3.20×10^{-1}	13	4.56	16	5a
^{90}Sr	1.09	23	7.91	17	1b, 3b, 4e
^{137}Cs	5.92×10^{-2}	7	2.37	14	5a
^{228}Th	2.86×10^{-2}	13	6.71	17	1c, 2c
^{232}Th	2.68×10^{-2}	10	7.75	21	1c, 2c
^{235}U	7.5×10^{-4}	19	5.89	12	1c, 5f
^{238}U	1.74×10^{-2}	8	2.51	15	1c, 5f
^{238}Pu	2.6×10^{-4}	+14 to –38	8.31	17	1c
$^{239+240}$Pu	4.00×10^{-3}	+7 to –18	4.12	13	1c
^{241}Am	1.1×10^{-3}	+30 to –81	16.8	13	1c

[a] Because of variations in the determination of radionuclide concentrations, the uncertainties used are the 95 % tolerance limits for coverage of at least 95 % of the measured values for this lot of bottled sediment samples. In other words, if measurements were made on all the samples (with precision and accuracy no worse than that of the measurements used to certify this SRM), then at least 95 percent of these measured values would fall within the indicated tolerance limits with confidence 95 percent. Tolerance limits are based on the standard deviation and number of measurements listed.

[b] Sample Decomposition Methods:

1. HF-HNO_3 or HF-HNO_3-$HClO_4$ dissolution
2. KF-pyrosulfate fusion [1,2]
3. HCl, HNO_3 or HCl-HNO_3 leaching [3]
4. HCl-NaOH leaching [3]
5. Non-destructive analysis

[c] Instrumental Methods:

a. Gamma-ray spectrometry detector [3,4]
b. Beta-particle counting with thin-window Geiger counter
c. Alpha-particle spectrometry with surface-barrier detector
d. Radon emanation counting
e. Beta-particle scintillation counting with plastic-phosphor
f. Delayed neutron activation [5]

Information Values: Information values for massic activity are provided in Table 3, homogeneity assessment in Table 4, and semi-quantitative emission spectrographic analysis is provided in Table 5. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [6]. Information values cannot be used to establish metrological traceability.

Table 3. Information Massic Activity Values for SRM 4354

Radionuclide	Massic Activity ($Bq \cdot g^{-1}$)	Laboratory[(a)]	Method Code[(b,c)]
^{210}Pb	1.2×10^{-1}	NIST, ORNL	1b, 5a
^{226}Ra	3×10^{-2}	EML	2d
^{230}Th	1.3×10^{-2}	EML, NIST	1c
^{234}U	1.9×10^{-2}	EML, NIST	1c

(a) See Table 6 for laboratory acronyms.
(b) Sample Decomposition Methods:
1. $HF\text{-}HNO_3$ or $HF\text{-}HNO_3\text{-}HClO_4$ dissolution
2. KF-pyrosulfate fusion [1,2]
3. HCl, HNO_3 or $HCl\text{-}HNO_3$ leaching [3]
4. HCl-NaOH leaching [3]
5. Non-destructive analysis

(c) Instrumental Methods:
a. Gamma-ray spectrometry detector [3,4]
b. Beta-particle counting with thin-window Geiger counter
c. Alpha-particle spectrometry with surface-barrier detector
d. Radon emanation counting
e. Beta-particle scintillation counting with plastic-phosphor
f. Delayed neutron activation [5]

Homogeneity Assessment: The material has been tested at a sample size of 30 grams, for which gamma-ray-emitting radionuclides have been found to be homogeneous. Furthermore, the material has been tested at a sample size of 2 grams for which actinide radionuclides have been found to be homogeneous. The homogeneity assessment information is provided below.

Gamma-Ray-Emitting Radionuclides: Eight thirty-gram bottled samples were examined for homogeneity in their gamma-ray-emission rates by counting them in a 5-in NaI(Tl) well detector coupled to a multichannel analyzer. The count rates from each bottle were compared over each of ten selected energy regions and also over the total gamma-ray spectrum (0.05 – 1.82 MeV). The sample-to-sample inhomogeneities inferred from these gamma-ray-emission rates are summarized in Table 4.

Table 4. Information Sample-To-Sample Inhomogeneities Values for Gamma-Ray-Emitting Radionuclides in SRM 4354

Energy Region (keV)	Standard Deviation (%)
0.05 – 0.16	1.64
0.16 – 0.27	1.95
0.27 – 0.32	1.53
0.32 – 0.44	1.87
0.44 – 0.54	1.90
0.54 – 0.74	0.57
0.74 – 1.01	0.59
1.01 – 1.23	0.65
1.23 – 1.53	0.45
1.53 – 1.82	0.71
0.05 – 1.82	0.59

Alpha-Particle-Emitting Radionuclides: Non-normal concentration distributions for the U, Th, and Pu isotopes were characterized through statistical tests for consistency between laboratories and also within and between bottles. Estimates of radionuclide inhomogeneity were determined by subtracting in quadrature the counting statistics from the standard deviation of the measurements. The inhomogeneities for uranium and thorium isotopes are estimated to be about one percent (one standard deviation) for eight-gram samples. The inhomogeneity for $^{239+240}$Pu is estimated to be approximately four percent for two-gram samples.

Table 5. Semi-Quantitative Emission Spectrographic Analysis for SRM 4354[a]

Element	Concentration (ppm)	Element	Concentration (%)
Antimony (Sb)	< 32	Aluminum (Al)	1.5
Arsenic (As)	< 150	Calcium (Ca)	1.06
Barium (Ba)	270	Iron (Fe)	2.6
Beryllium (Be)	< 1.0	Magnesium (Mg)	0.18
Bismuth (Bi)	< 10	Manganese (Mn)	0.043
Boron (B)	< 4.7	Phosphorus (P)	0.11
Cadmium (Cd)	< 32	Potassium (K)	0.24
Cerium (Ce)	153	Silicon (Si)	15
Chromium (Cr)	22	Sodium (Na)	0.18
Cobalt (Co)	12	Titanium (Ti)	0.041
Copper (Cu)	27		
Dysprosium (Dy)	< 22		
Erbium (Er)	< 10		
Europium (Eu)	2.8		
Gadolinium (Gd)	< 15		
Gallium (Ga)	< 1.5		
Germanium (Ge)	< 1.5		
Gold (Au)	< 10	Major Components Recalculated as Oxides	Concentration (%)
Hafnium (Hf)	< 15		
Holmium (Ho)	< 6.8	SiO_2	31
Indium (In)	< 6.8	Al_2O_3	2.8
Iridium (Ir)	< 15	Fe_2O_3	3.7
Lanthanum (La)	93	MgO	0.30
Lead (Pb)	11	CaO	1.5
Lithium (Li)	< 68	Na_2O	0.24
Lutetium (Lu)	< 15	K_2O	0.29
Manganese (Mn)	430	TiO_2	0.068
Molybdenum (Mo)	< 1.0	P_2O_5	0.26
Neodymium (Nd)	150	MnO	0.056
Nickel (Ni)	9.2		
Niobium (Nb)	< 3.2		
Osmium (Os)	< 22		
Palladium (Pd)	< 1.0		
Platinum (Pt)	< 4.6		
Praseodymium (Pr)	< 68		
Rhenium (Re)	< 10		
Rhodium (Rh)	< 2.2		
Ruthenium (Ru)	< 2.2		
Samarium (Sm)	11		
Scandium (Sc)	7.2		
Silver (Ag)	< 0.10		
Strontium (Sr)	137		
Tantalum (Ta)	< 460		
Terbium (Tb)	< 32		
Thallium (Tl)	< 4.6		
Thorium (Th)	< 22		
Thulium (Tm)	< 4.6		
Tin (Sn)	2.3		
Tungsten (W)	< 10		
Uranium (U)	< 320		
Vanadium (V)	54		
Ytterbium (Yb)	3.7		
Yttrium (Y)	26		
Zinc (Zn)	88		
Zirconium (Zr)	42		

[a] The relative uncertainty for each reported concentration is plus 50 %, and minus 33 %.

Table 6. Participating Laboratories and Technical Points of Contact at Time of Certification

Laboratory Acronym	Laboratory	City/State	Technical Contact
EML	Environmental Measurements Laboratory U.S. Department of Energy	New York, NY	Dr. H.L. Volchok, Mr. M.S. Feiner
ORNL	Oak Ridge National Laboratory Martin Marietta Energy Systems	Oak Ridge, TN	Dr. I.L. Larsen
LANL	Los Alamos National Laboratory University of California	Los Alamos, NM	Dr. E.S. Gladney
OSU	Oregon State University School of Oceanography	Newport, OR	Dr. T.M. Beasley
RESL	Radiation and Environmental Sciences Laboratory U.S. Nuclear Regulatory Commission	Idaho Falls, ID	Mr. D.G. Olson, Mr. D. Percival
NIST	National Institute of Standards and Technology (formerly National Bureau of Standards) U.S. Department of Commerce	Gaithersburg, MD	Dr. J.M.R. Hutchinson, Dr. K.G.W. Inn

REFERENCES

[1] Bernabee, R.; Percival, D.; Hindman, F.; *Liquid-liquid Extraction Separation and Determination of Plutonium and Americium*; Analytical Chemistry, Vol. 52(14), pp. 2351 (1980).

[2] Sill, C.W.; Hindman, F.C.; Anderson, J.L.; *Simultaneous Determination of Alpha-emitting Nuclides of Radium through Californium in Large Environmental and Biological Samples*; Anal. Chem., Vol. 51(8), p. 1307 (1979).

[3] *Environmental Measurements Laboratory Procedures Manual*; HASL 300 with supplements, Volchock, H.L.; de Planque, G., Eds.; New York: NY (1986).

[4] Cutshall, N.H.; Larsen, I.L.; Olsen, C.R.; *Direct Analysis of ^{210}Pb in Sediment Samples: Self-absorption Corrections*; Nucl. Inst. Methods, Vol. 206, p. 309 (1983).

[5] Gladney, E.S.; Curtis, D.B.; Pervin, D.R.; Owens, J.W.; Goode, W.E.; *Nuclear Techniques for the Chemical Analysis of Environmental Materials*; Report LA-8192-MS, Los Alamos National Laboratory: Los Alamos, NM (1980).

[6] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at: http://www.nist.gov/srm/publications.cfm (accessed Dec 2016).

Certificate Revision History: 08 December 2016 (Editorial changes); November 1986 (Original certificate issue date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov*; or via the Internet at* http://www.nist.gov/srm.