National Institute of Standards & Technology

Certificate

Standard Reference Material® 4350B

River 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 of measurements of environmental radioactivity in river sediment and similar matrices. A unit of SRM 4350B consists of approximately 85 g of freeze-dried, pulverized river sediment in a polyethylene bottle.

Certified Values: The certified properties for the river sediment radionuclide standard are listed in Table 1 with the property values listed in Table 2. A NIST certified value, as used within the context of this certificate, is a value 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 4350B** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored properly and that no change in composition has occurred. This matrix is considered to be stable; however, its stability has not been rigorously assessed. This SRM should be handled in accordance with instructions given in this certificate (see “Instructions for Handling and Storage”). Accordingly, periodic recertification of this SRM is not required. 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.

Statistical consultation was provided by W.S. Liggett, Jr., formerly of NIST.

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

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Certificate Issue Date: 03 November 2015
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
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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. 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 5 g for which it has been found to be homogeneous.

Details of the SRM preparation: The soil was pulverized with a "pancake" style air jet mill. The average particle diameter for the resulting powder is 8 µm. More than 99 %, by weight, of the particles are less than 20 µm in diameter.

Certified Values: Each certified value was determined by two or more methods and/or two or more laboratories. The ^{239}Pu + ^{240}Pu are recoverable by normal HNO_3 or HNO_3-HCl leaching procedures as well as by the more vigorous methods of chemical treatment listed in Table 2. The uncertainties on certified values are expanded uncertainties, $U = ku_c$. The quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [1,2], and where the individual standard uncertainties are evaluated by either type A or type B assessments. The combined standard uncertainty is multiplied by a coverage factor of k = 3 and was chosen to obtain an approximate 99 % level of confidence.

Table 1. Certified Properties of SRM 4350B

Radionuclides	**See Table 2**
Reference time	**09 September 1981**
Certified massic activities	**See Table 2**
Uncertainties	**See Table 2**

Table 2. Certified Massic Activity Values for SRM 4350B

Radionuclide	**Massic Activity ($Bq•g^{-1}$)**	**Relative Expanded Uncertainty (%)**	**Half-life(a,b) (years)**	**Analytical Methods(c,d)**
^{60}Co	**4.64×10^{-3}**	**5.0**	**5.2711 ± 0.0008**	**1a, 1f, 5a**
^{137}Cs	**2.90×10^{-2}**	**6.3**	**30.05 ± 0.08**	**1b, 1f, 3a, 4f, 5a**
^{152}Eu	**3.05×10^{-2}**	**4.0**	**13.522 ± 0.016**	**3a, 5a**
^{154}Eu	**3.78×10^{-3}**	**15**	**8.601 ± 0.004**	**3a, 5a**
^{226}Ra	**3.58×10^{-2}**	**10**	**1600 ± 7**	**2d**
^{238}Pu	**1.3×10^{-5}**	**17**	**87.74 ± 0.03**	**1c, 2c, 3c**
$^{239+240}$Pu	**5.08×10^{-4}**	**5.8**	**---**	**1c, 2c, 3c**
^{239}Pu	**---**	**---**	**24100 ± 11**	**---**
^{240}Pu	**---**	**---**	**6561 ± 7**	**---**
^{241}Am	**1.5×10^{-4}**	**21**	**432.6 ± 0.6**	**1c, 2c, 3c**

(a) Half-life taken from the Table of Radionuclides (accessed Nov 2015) [3].
(b) The uncertainty listed with each value is an expanded uncertainty, U, calculated as $U = ku_c$, with coverage factor, k = 1.
(c) Sample Decomposition Methods:
1 HF-HNO_3 or HF-HNO_3-$HClO_4$ dissolution
2 KF-pyrosulfate fusion [4,5,6]
3 HCl, HNO_3 or HCl-HNO_3 leaching [7,8,9]
4 HCl-NaOH leaching [7]
5 Non-destructive analysis
(d) Instrumental Methods:
a Gamma-ray spectrometry with Ge(Li) detector
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 X-ray photons or beta-particle counting with gas-flow proportional counter

Information Values: Information values for massic activity are provided in Table 3 and mass spectrometry data analysis at the Savannah River National Laboratory is provided in Table 4. 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 [10]. Information values cannot be used to establish metrological traceability.

Table 3. Information Massic Activity Values for SRM 4350B

Radionuclide	Massic Activity ($Bq \bullet g^{-1}$)	Laboratory[a]	Analytical Methods[b,c]
^{40}K	5.6×10^{-1}	RESL	5a
^{55}Fe	1.7×10^{-2}	WHOI	3f
^{90}Sr	5.3×10^{-3}	EML, RESL, WHOI	1b, 2f, 3b, 4e
^{228}Th	3.35×10^{-2}	RESL	2c
^{230}Th	2.95×10^{-2}	RESL	2c
^{232}Th	3.32×10^{-2}	RESL	2c
^{234}U	3.32×10^{-2}	RESL	2c
^{235}U	1.7×10^{-3}	RESL	2c
^{238}U	3.08×10^{-2}	RESL	2c

[a] See Table 7 for participating laboratories and technical points of contact information.
[b] Sample Decomposition Methods:
1 HF-HNO_3 or HF-HNO_3-$HClO_4$ dissolution
2 KF-pyrosulfate fusion [4,5,6]
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e Beta-particle scintillation counting with plastic phosphor
f X-ray photons or beta-particle counting with gas-flow proportional counter

If additional data become available, other radioactivity concentrations 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 method used to the technical contacts on page 1.

Table 4. Mass Spectrometry Data Analysis Performed at Savannah River National Laboratory
By Dr. J Halverson for SRM 4350B

Nuclide	Atom Percent
^{239}Pu	89.91
^{240}Pu	9.43
^{241}Pu	0.318
^{242}Pu	0.336

Homogeneity Assessment: Ten 100 g sample bottles were examined for homogeneity using their gamma-ray-emission rates by counting them in a 5 inch NaI(Tl) well detector coupled to a multichannel analyzer. The count rates from each bottle were compared over each of thirteen selected energy regions and also over the total gamma-ray spectrum (0.04 MeV to 1.85 MeV). The net sample-to-sample inhomogeneities from gamma-ray-emission rates are summarized below in Table 5. Inhomogeneities of ^{90}Sr and ^{137}Cs are less than 2 percent for 10 g samples. Inhomogeneities of alpha-particle emitting radionuclides are less than 3 percent.

Table 5. Net Sample-To-Sample Inhomogeneities for SRM 4350B

Energy Region (keV)	Standard Deviation of the Mean (%)
47 – 107	1.27
109 – 157	1.81
159 – 203	3.15
204 – 271	1.23
273 – 315	2.04
317 – 423	1.51
425 – 540	1.08
542 – 752	0.98
754 – 848	0.94
850 – 1030	0.74
1032 – 1300	0.81
1302 – 1662	0.64
1663 – 1845	3.55
47 – 1845	0.47

Table 6. Semi-Quantitative Emission Spectrographic Analysis for SRM 4350B[a]

Element	Concentration (ppm)	Element	Concentration (%)
Antimony (Sb)	< 32	Aluminum (Al)	8.9
Arsenic (As)	< 150	Calcium (Ca)	2.5
Barium (Ba)	703	Iron (Fe)	6.8
Beryllium (Be)	2.5	Magnesium (Mg)	1.6
Bismuth (Bi)	< 10	Manganese (Mn)	0.066
Boron (B)	27	Phosphorus (P)	< 0.068
Cadmium (Cd)	< 34	Potassium (K)	> 1.5
Cerium (Ce)	90	Silicon (Si)	> 34
Chromium (Cr)	58	Sodium (Na)	2.2
Cobalt (Co)	25	Titanium (Ti)	0.60
Copper (Cu)	40		
Dysprosium (Dy)	< 22		
Erbium (Er)	< 10		
Europium (Eu)	< 2.2		
Gadolinium (Gd)	< 15		
Gallium (Ga)	16		
Germanium (Ge)	< 1.5		
Gold (Au)	< 10	Major Components Recalculated as Oxides	Concentration
Hafnium (Hf)	< 15		(%)
Holmium (Ho)	< 6.8	SiO_2	> 73
Indium (In)	< 6.8	Al_2O_3	17
Iridium (Ir)	< 15	Fe_2O_3	9.5
Lanthanum (La)	57	MgO	2.7
Lead (Pb)	42	CaO	3.5
Lithium (Li)	< 68	Na_2O	2.9
Lutetium (Lu)	< 15	K_2O	> 1.8
Manganese (Mn)	660	TiO_2	1.0
Molybdenum (Mo)	< 1.0	P_2O_5	< 0.16
Neodymium (Nd)	40	MnO	0.085
Nickel (Ni)	33		
Niobium (Nb)	29		
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)	< 10		
Scandium (Sc)	24		
Silver (Ag)	< 0.10		
Strontium (Sr)	583		
Tantalum (Ta)	< 460		
Terbium (Tb)	< 32		
Thallium (Tl)	< 4.6		
Thorium (Th)	< 22		
Thulium (Tm)	< 4.6		
Tin (Sn)	< 1.8		
Tungsten (W)	< 10		
Uranium (U)	< 320		
Vanadium (V)	160		
Ytterbium (Yb)	3.5		
Yttrium (Y)	33		
Zinc (Zn)	203		
Zirconium (Zr)	403		

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

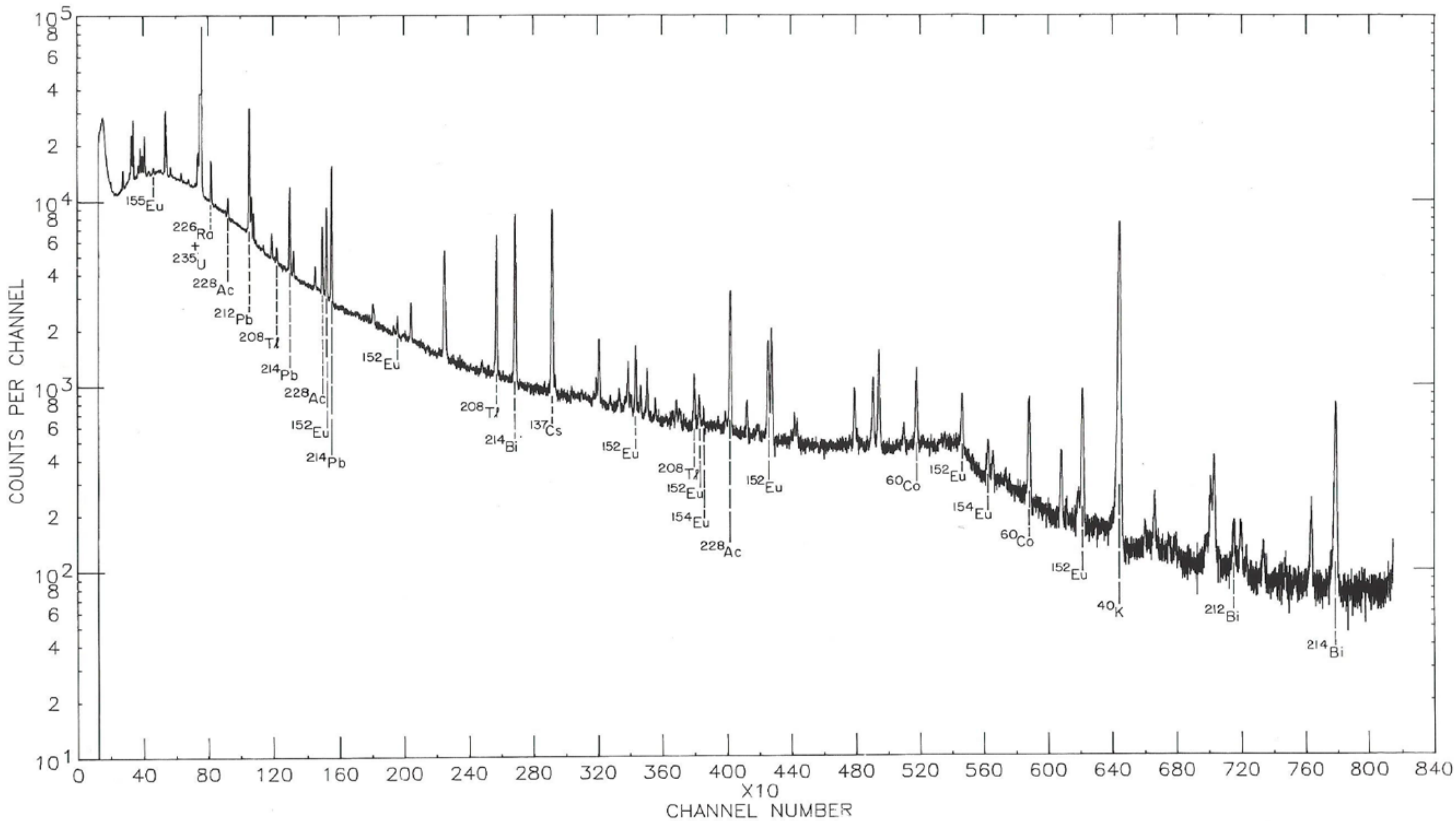

Figure 1. Gamma-ray spectrum of SRM 4350B, with 60 cm^3 Ge(Li) detector. Background has not been subtracted and contributes typically 20 percent to the peaks for many natural radioelements.

Table 7. Participating Laboratories and Technical Points of Contact at Time of Certification

Laboratory Acronym	Laboratory	City/State	Technical Contact
EML	U.S. Department of Energy, Environmental Measurements Laboratory	New York, NY	Dr. H.L. Volchok, Mr. M.S. Feiner
OSU	Oregon State University School of Oceanography	Newport, OR	Dr. T.M. Beasley
RESL	U.S. Department of Energy, Radiation and Environmental Sciences Laboratory (Reference Laboratory for the U.S. Nuclear Regulatory Commission)	Idaho Falls, ID	Mr. D.G. Olson, Mr. D.R. Percival
NIST	U.S. Department of Commerce, National Institute of Standards and Technology (formerly National Bureau of Standards)	Gaithersburg, MD	Dr. J.M.R. Hutchinson, Dr. K.G.W. Inn
WHOI	Woods Hole Oceanographic Institution	Woods Hole, MA	Dr. V.T. Bowen, Dr. H.D. Livingston

REFERENCES

[1] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2015).

[2] Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Nov 2015).

[3] Laboratoire National Henri Becquerel; *Table of Radionuclides, Recommended Data (online)*, Saclay-Gif-sur-Yvette, France (2013); available at http://www.nucleide.org/DDEP_WG/DDEPdata.htm (accessed Nov 2015).

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[10] May, W.; Parris, R.; Beck, 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 Nov 2015).

Certificate Revision History: 03 November 2015 (Certified massic activity half-lives added to Table 2; editorial changes); September 9, 1981 (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.