

Standard Reference Material[®] 4357

Ocean Sediment Environmental Radioactivity Standard

CERTIFICATE

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in tests or measurements of environmental radioactivity contained in matrices similar to the sample, for evaluating analytical methods, and as a generally available calibrated “real” sample matrix in interlaboratory comparisons.

Description: SRM 4357 is a blend of ocean sediments collected off the coast of Sellafield, UK, and in the Chesapeake Bay, USA. This SRM has been developed in cooperation with member laboratories of the International Committee for Radionuclide Metrology and expert national laboratories. A unit of SRM 4357 consists of approximately 85 g of material in a powder form contained in a polyethylene bottle.

Certified Values: The certified properties for the ocean sediment radionuclide standard are presented in Table 1 and Table 2. NIST certified values, as used within the context of this certificate, are values for which NIST has the highest confidence in the 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. Massic activity is the preferred name for the quantity activity (Bq) divided by the total mass of the sample [1]. For further information on the expression of uncertainties see references 2 and 3. Each reporting laboratory maintains its own traceability to the derived SI unit, becquerel (Bq).

Additional Information: Non-certified values are provided in Appendix A. Values of potential interest and additional information are provided in Appendices B and C.

Period of Validity: The certified values delivered by **SRM 4357** are 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. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified. Periodic recertification of this SRM is not required.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

James M. Adams, Chief
Radiation Physics Division
Certificate Revision History on Page 3

Steven J. Choquette, Director
Office of Reference Materials

Table 1: Certified Massic Activities for Radionuclides with a Normal Distribution of Measurement Results
REFERENCE TIME is 16 February 1994

Radio-nuclide	Mean $\pm 2s_m$ (mBq•g ⁻¹) ^(a)	2.5 % to 97.5 % Tolerance Limit (mBq•g ⁻¹) ^(b)	Number of Assays	Half-life ^(c) (a)	Analytical Method(s) ^(d,e)	Contributing Laboratories (See Table C1)
⁴⁰ K	225 $\pm$ 5	190 – 259	31	(1.277 $\pm$ 0.008) x 10 ⁹	3a	KU, MAFF, NIR, NPL, YAEL
²²⁶ Ra	12.7 $\pm$ 0.4	10.3 – 15.0	21	1600 $\pm$ 7	3a	EML, IT, KU, NPL
²²⁸ Ra	13.3 $\pm$ 0.8	9.2 – 17.4	20	5.75 $\pm$ 0.03	3a	IT, KU, NPL
²²⁸ Th	12.1 $\pm$ 0.3	9.7 – 14.6	40	1.9131 $\pm$ 0.0009	1c, 3a	EML, KU, LGC, MAFF, NIST, NPL, OSUH, YAEL
²³⁰ Th	12.0 $\pm$ 0.5	9.6 – 14.4	18	75380 $\pm$ 300	1c	LGC, NIST, OSUH
²³² Th	13.0 $\pm$ 0.3	11.6 – 14.3	18	(1.405 $\pm$ 0.006) x 10 ¹⁰	1c	LGC, NIST, OSUH

(a) The stated uncertainty is two times the standard deviation of the mean.

(b) Normal tolerance limit for 95 % confidence and 95 % coverage. See reference 4.

(c) The stated uncertainty is the standard uncertainty. See reference 5.

(d) Radiochemical Methods: (1) HF-HNO₃ or HF-HNO₃-HClO₄ dissolution; (2) NaOH-HCl leach; (3) Non-destructive analysis

(e) Detection Methods: a) Germanium gamma-ray spectrometer; b) Thin-window beta-particle Geiger counter; c) Silicon surface-barrier alpha-particle spectrometer

Table 2: Certified Massic Activities for Radionuclides with a Non-Normal Distribution of Measurement Results
REFERENCE TIME is 16 February 1994

Radio-nuclide	Mean $\pm 2s_m$ (mBq•g ⁻¹) ^(a)	2.5 % to 97.5 % Tolerance Limit (mBq•g ⁻¹) ^(b)	Number of Assays	Half-life ^(c) (a)	Analytical Method(s) ^(d,e)	Contributing Laboratories (See Table C1)
⁹⁰ Sr	4.4 $\pm$ 0.3	2.1 – 8.4	49	28.78 $\pm$ 0.04	1b, 2d	EML, LGC, MAFF, NE, NRPB, YAEL
¹³⁷ Cs	12.7 $\pm$ 0.2	10.8 – 15.9	76	30.07 $\pm$ 0.03	3a	EML, IT, KU, LGC, MAFF, NE, NIR, NPL, ORNL, OSUB, YAEL
²³⁸ Pu	2.29 $\pm$ 0.05	1.96 – 2.98	65	87.7 $\pm$ 0.3	1c, 4e	EML, GAU, LGC, MAFF, NIST, OSUB, RESL
²³⁹⁺²⁴⁰ Pu	10.4 $\pm$ 0.2	9.3 – 13.2	84	24110 $\pm$ 30 6564 $\pm$ 11	1c, 3a, 4e	EML, GAU, IT, LGC, MAFF, NIST, OSUB, OSUH, RESL

(a) The stated uncertainty is two times the standard deviation of the mean.

(b) Weibull tolerance limit for 95 % confidence and 95 % coverage.

(c) The stated uncertainty is the standard uncertainty. See reference 5.

(d) Radiochemical Methods: 1) HF-HNO₃ or HF-HNO₃-HClO₄ dissolution; 2) NaOH-HCl leach; 3) Non-destructive analysis; 4) Fusion

(e) Detection Methods: a) Germanium gamma-ray spectrometer; b) Thin-window beta-particle Geiger counter; c) Silicon surface-barrier alpha-particle spectrometer; d) Plastic-phosphor beta-particle scintillation counter; e) Thermal Ionization mass spectrometer

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

Storage and Handling: The SRM should be stored in a dry location at room temperature. The bottle should be shaken before opening in a chemical hood, and the bottle should be recapped tightly as soon as subsamples are 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 national, international, and carrier regulations.

Heterogeneity: This material has been measured using sample sizes of 10 g to 100 g, for which heterogeneity of gamma-ray-emitting radionuclides has been detectable. Furthermore, heterogeneity has been detected at a sample size of 10 grams for ^{90}Sr and transuranium radionuclides. The expected variation of measurements due to heterogeneity has been incorporated in the certified tolerance limits and in the uncertainty of mean concentration values. The certified values for radionuclides with a normal distribution of analytical measurements are listed in Table 1. Table 2 lists the certified values for radionuclides with a non-normal distribution of analytical measurements. It is recommended that a sample size of 10 grams or larger be used for radiometric and radiochemical analysis.

Instructions for Drying: When nonvolatile radionuclides are to be determined, working samples of this SRM should be dried in air at 40 °C for 24 hours prior to weighing. Volatile radionuclides (e.g., ^{137}Cs , ^{210}Pb and ^{212}Pb) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture content is to be made to the data for volatile radionuclides before comparing to the certified values. This procedure ensures that these volatile radionuclides are not lost during drying. The weight loss on drying is typically less than 2 percent.

Radionuclide Leachability: All actinides and their daughters are approximately 87 % removed from the sample by HNO_3 or $\text{HNO}_3\text{-HCl}$ leaching procedures. Total sample digestion or non-destructive analysis is required to produce results that can be compared to those listed in this certificate.

Application of the Certified Values: When five or more measurements are available, compute the sample mean and ascertain that the mean falls within the certified mean plus uncertainties interval. When four or fewer measurements are available, then ascertain that all of the individual values are within the certified tolerance limits interval.

REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: <https://www.nist.gov/pml/special-publication-811> (accessed Nov 2022).
- [2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at <https://www.bipm.org/en/committees/jc/jcgm/publications> (accessed Nov 2022).
- [3] 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 <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Nov 2022).
- [4] Natrella, M.G.; *Experimental Statistics*, Handbook 91, United States Department of Commerce National Bureau of Standards (1963).
- [5] National Nuclear Data Center, Brookhaven Laboratory; *Evaluated Nuclear Structure Data File (ENSDF)*; Upton, NY (2006); available at <https://www.nndc.bnl.gov/ensdf/> (accessed Nov 2022).
- [6] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Monographs on Statistics & Applied Probability 57, Chapman & Hall: New York (1993).

Certificate Revision History: 01 November 2022 (Updated format; editorial changes); March 1997 (Original certificate issue date).
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Certain commercial equipment, instruments, or materials may be identified in this Certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at <https://www.nist.gov/srm>.

***** End of Certificate *****

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system. Non-certified values are provided in Table A1.

Table A1. Massic Activity Values of Radionuclides for which there are insufficient numbers of data sets or for which discrepant data sets were obtained
REFERENCE TIME is 16 February 1994

Radio-nuclide ^(a)	Mean (mBq•g ⁻¹)	Range of Reported Results (mBq•g ⁻¹)	Number of Assays	Half-life ^(b)	Analytical Method(s) ^(c,d)	Contributing Laboratories (See Table C1)
¹²⁹ I	0.009	0.006 – 0.012	6	$(1.57 \pm 0.04) \times 10^7$ a	5a	NIR
¹⁵⁵ Eu	1.4	1.2 – 1.5	2	(4.68 ± 0.05) a	3a	MAFF
²¹⁰ Po	14	12 – 15	5	(138.376 ± 0.002) d	3a	OSUH
²¹⁰ Pb	24	14 – 35	19	(22.3 ± 0.2) a	3a	IT, ORNL
²¹² Pb	14	13 – 14	5	(10.64 ± 0.01) h	3a	MAFF
²¹⁴ Bi	15	9 – 20	5	(19.9 ± 0.4) m	3a	MAFF
²³⁴ U	12	9 – 15	68	$(2.45 \pm 0.02) \times 10^5$ a	1c, 3a	AWE, IT, LGC, NIST, OSUH, RESL
²³⁵ U	0.6	0.1 – 1.4	63	$(7.038 \pm 0.006) \times 10^8$ a	1c, 3a, 4e	AWE, GAU, IT, NIST, NPL, RESL
²³⁷ Np	0.007	0.004 – 0.009	9	$(2.14 \pm 0.01) \times 10^6$ a	1c, 3a	LRM, KU
²³⁸ U	12	7 – 16	76	$(4.468 \pm 0.003) \times 10^9$ a	1c, 3a, 4e	AWE, GAU, IT, LGC, NIST, OSUH, RESL
²⁴¹ Am	10	7 – 18	97	(432.7 ± 0.6) a	1c, 3a	AWE, BNF, IT, KU, LGC, MAFF, NIST, NPL, ORNL, OSUB, RESL

^(a) Data for these radionuclides are provided for information only.

^(b) The stated uncertainty is the standard uncertainty. See reference 6 on page 3.

^(c) Radiochemical Methods: 1) HF-HNO₃ or HF- HNO₃-HClO₄ dissolution; 2) NaOH-HCl leach; 3) Non-destructive analysis; 4) Fusion; 5) Heat oxidation and charcoal-filter trapping

^(d) Detection Methods: a) Germanium gamma-ray spectrometer; b) Thin-window beta-particle Geiger counter; c) Silicon surface-barrier alpha-particle spectrometer d) Plastic-phosphor beta-particle scintillation counter; e) Thermal Ionization mass spectrometer

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

***** End of Appendix A *****

APPENDIX B

Values of Potential Interest to Users: Table B 1 provides values of potential interest to users.

Table B1. Semi-Quantitative Emission Spectrographic Analysis of SRM 4357
Data provided by United States Department of the Interior, Geological Survey (Reston, VA).

Element	Amount ($\mu\text{g}\cdot\text{g}^{-1}$)	Element	Amount ($\mu\text{g}\cdot\text{g}^{-1}$)	Element	Amount ($\mu\text{g}\cdot\text{g}^{-1}$)
Ag	0.12	Ho	<6.8	Re	<10
Al	24700	I ^(a)	3.1	Rh	<2.2
As	<100	In	<10	Ru	<2.2
Au	<6.8	Ir	<15	Sb	<68
B	34	K	5070	Sc	2.8
Ba	143	La	25	Si	>340000
Be	<0.1	Li	<68	Sm	<10
Bi	<10	Lu	<15	Sn	<4.6
Ca	6267	Mg	3930	Sr	64
Cd	<32	Mn	163	Ta	<320
Ce	<43	Mo	1.8	Tb	<32
Co	2.9	Na	4000	Th	<46
Cr	27	Nb	10	Tl	<10
Cu	82	Nd	<32	Tm	<4.6
Dy	<22	Ni	97	U	<220
Er	<4.6	Os	<15	V	21
Eu	<2.2	P	<680	W	<15
Fe	10700	Pb	12	Y	12
Ga	3.5	Pd	<1.0	Yb	1.8
Gd	<32	Pr	<100	Zn	45
Ge	<4.6	Pt	<2.2	Zr	540
Hf	<150				

^(a) Data provided by Niedersächsisches Institut für Radioökologie (NIR) (Hannover, Germany).

***** End of Appendix B *****

APPENDIX C

Details of the SRM Preparation: This SRM is a blend of material collected off the coast of Sellafield, UK, and in the Chesapeake Bay, USA, in the weight ratio of 1:200, respectively. A. Knight and M. Measures of the National Radiological Protection Board, UK, collected, sieved to –200 mesh, dried and analyzed the Sellafield sediment before sending it to NIST. The Chesapeake Bay sediment was freeze dried, blended with the Sellafield sediment, sterilized with 50 kiloGrays of ^{60}Co radiation, and pulverized with a “pancake-style” air-jet mill. The average particle diameter for the resulting powder is approximately 6 μm and more than 99 %, by weight, of the particles are less than 20 μm in diameter.

Preparation: This Standard Reference Material was prepared in the Radioactivity Group, Physical Measurement Laboratory, Radiation Physics Division, J.M.R. Hutchinson, Group Leader formerly of NIST. The overall technical direction leading to certification was provided by K.G.W. Inn formerly of NIST. Statistical support was provided by J.J. Filliben, W.S. Liggett, N.F. Zhang of the NIST Statistical Engineering Division; and E.S. Lagergren and K.R. Eberhardt formerly of NIST.

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

Technical Contacts: For technical questions regarding this material, please contact SRMs@nist.gov.

Calculation of the Uncertainties: The bootstrap is a computationally-intensive statistical procedure for estimating and computing the uncertainty of a statistic whose form is complicated and/or whose underlying assumptions (e.g., normality) are non-standard. The virtue of this procedure is that it provides a straightforward, rigorous methodology for computing uncertainties that would otherwise be difficult to obtain. See reference 6 on page 3.

The bootstrap was utilized here for calculation of the mean and tolerance limits. The calculations of these statistics are complicated because the normality of some of the data is suspect. The usual underlying assumptions do not hold due to a variety of experimental conditions, including interlaboratory biases, within-laboratory methodology differences, and material heterogeneity.

Procedurally, the bootstrap estimate for the uncertainty of a statistic (e.g., the mean) is obtained as follows:

1. From the original sample of n observations, compute the statistic of interest (e.g., the mean).
2. From the original n data points, extract a random sample (with replacement) of n points (this becomes the bootstrap sample).
3. Compute the statistic of interest (e.g., the mean) from this bootstrap sample (this will be the bootstrap statistic).
4. Repeat steps 2 and 3 a large number of times (e.g., 1000 times); the bootstrap statistic will, of course, change from one bootstrap sample to the next.
5. Compute the standard deviation of the statistic by applying the usual standard deviation formula to the 1000 bootstrap statistics.

Table C1. Participating Laboratories and Personnel

Abbreviation	Laboratory	Location/Country	Personnel Status as of March 1997
AWE	Atomic Weapons Establishment	Aldermaston, UK	B. Warren
BNF	British Nuclear Fuels Ltd	Sellafield, UK	T. Bates
EML	Environmental Measurements Laboratory, US Department of Energy	New York, NY, USA	P. Krey, M. Feiner (Retired)
EPA	Environmental Protection Agency	Las Vegas, NV, USA	C.-K. Liu (Now at the US Department of Energy, Richland, Washington), R. Holloway (Independent Consultant)
GAU	Geosciences Advisory Unit, Southampton Oceanography Center	Southampton, UK	I. Croudace
IT	International Technology Corporation	Oak Ridge, TN, USA	J. Harvey (ElChroM Industries, Inc.)
KU	Kanazawa University	Kanazawa, JP	M. Yamamoto
LGC	Laboratory of the Government Chemist	Teddington, UK	A. Holms
LRM	Laboratoire de Radioecologie Marine, Commissariat a L'Energie Atomique	Centre D'Studies Nucleaires de Fonteney-Aux-Roses, FR	P. Germain
MAFF	Ministry of Agriculture, Fisheries and Food	Lowestoft, UK	B. Harvey
NE	Nuclear Electric	Gravesend, UK	K. Odell
NIR	Niedersächsisches Institut für Radioökologie	Hannover, GE	J. Handl
NIST	National Institute of Standards and Technology	Gaithersburg, MD, USA	K. G.W. Inn , Z. Lin, J. Filliben, F. Schima
NPL	National Physical Laboratory	Teddington, UK	M. Woods, Simon Jerome
NRPB	National Radiological Protection Board	Didcot, Oxfordshire, UK	D. Popplewell (Retired)
ORNL	Oak Ridge National Laboratory, Martin Marietta Energy Systems	Oak Ridge, TN, USA	I.L Larsen
OSUB	Oregon State University	Newport, OR, USA	T. Beasley (Now at EML)
OSUH	Oregon State University	Newport, OR, USA	C.A. Huh
RESL	Radiological and Environmental Sciences Laboratory	Idaho Falls, ID, USA	S. Morton
SSEB	South of Scotland Electricity Board	Hunterston, UK	P. Young
Yael	Yankee Atomic Environmental Laboratory	Bolton, MA, USA	D. McCurdy

***** End of Appendix C *****