National Institute of Standards & Technology

Certificate

Standard Reference Material® 4358

Ocean Shellfish Radionuclide Standard

This Standard Reference Material (SRM) has been developed in cooperation with member laboratories of the International Committee for Radionuclide Metrology and other experienced metrology laboratories. The SRM consists of approximately 150 grams shellfish powder that was freeze-dried, pulverized, bottled, and sterilized. The SRM is intended for use in tests of measurements of radioactivity contained in matrices similar to the sample, for evaluating analytical methods, and as a generally available calibrated "real" sample matrix for laboratory intercomparison.

Certified Values: The certified properties for the ocean shellfish radionuclide standard are presented in Table 1. 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, becquerel (Bq).

Expiration of Certification: The certification of **SRM 4358** 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, Storage, and Use"). 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 material was prepared under the leadership of M. Unterweger of the NIST Radiation Physics Division. The coordination of the technical measurements leading to certification was performed by S. Nour and K.G.W. Inn of the NIST Radiation Physics Division.

Statistical analysis was performed by J. Filliben and H.K. Iyer of the NIST Statistical Engineering Division.

Semi-quantitative composition of the material was determined using x-ray fluorescence analysis by J.R. Sieber of the NIST Chemical Sciences Division.

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

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INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Storage: The SRM should be stored in a dry (humidity less than 80 %) location at a temperature between 5 °C and 60 °C (41-140 °F).

Handling: The SRM should be used only by qualified persons. The bottle should be shaken before opening in a chemical hood and 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.

Use: A sample size of at least 30 grams is recommended for radiochemical analysis.

Instructions for Drying: When nonvolatile radionuclides are to be determined, working samples of this SRM should be dried at 40 °C for 24 hours prior to weighing. Volatile radionuclides (e.g., ^{210}Po, ^{137}Cs, ^{210}Pb, ^{212}Pb and ^{214}Pb) should be determined on samples as received. Separate samples should be dried as described above to obtain a correction factor for moisture. Correction for moisture content must be made to the data for volatile radionuclides before comparing with the values given in this certificate. This procedure ensures that these radionuclides are not lost during drying [1]. The mass loss on drying is typically less than 4 percent.

Details of the SRM preparation: This SRM is a mixture of Irish Sea mussel (0.1 % mass fraction), White Sea mussel (12 % mass fraction), and Japan Sea oyster (87.9 % mass fraction). The raw material was dried, blade milled, and pulverized. The SRM was "V-cone" blended to optimize homogeneity, and bottled in polyethylene bottles. The final bottled SRM was sterilized with > 50 kGy of ^{60}Co radiation to satisfy export regulations and to increase shelf-life.

Heterogeneity: Ten bottles of the SRM were randomly selected and examined for gamma-ray heterogeneity by measuring their emission rates with a 5-inch (12.7 cm) NaI(Tl) detector coupled to a multichannel analyzer. The count rates from each measurement were analyzed for statistical difference for ten selected energy regions, and no detectable heterogeneity was observed. Statistical tests for heterogeneity for transuranium radionuclides were performed based on evaluation of 15 results (3 replicates from 5 different bottles) by alpha spectrometry analyses. The tests showed a between-bottle heterogeneity for the actinides. The contribution from this source of uncertainty has been incorporated into the evaluation of uncertainty. It is recommended that a sample size of 30 grams or larger be used for radiochemical actinide analysis.

Calculation of Certified Massic Activity Values: The certified massic activity values for each nuclide or their ratio (Tables 2 and 3) were determined from the evaluated median of the individual laboratories' means.

Calculation of Uncertainties for the Certified Values: The expanded uncertainties (U) for each of the certified values (Table 2) and ratios (Table 3) include contributions from several sources: 1) bottle-to-bottle variation; 2) within-bottle variation; 3) laboratory-to-laboratory variation; 4) radiochemical tracer SRM uncertainty; and 5) uncertainty due to the use of estimator as median for the certified value. Tables 2 and 3 do not explicitly estimate each uncertainty component; however the reported uncertainty is an overall combination of them all. These uncertainties express the dispersion of values observed both within and between laboratories, combined consistently with the GUM [2,3]. The reported uncertainty is calculated based on the assumption that all participating laboratories are equally competent. Combined standard uncertainty of the certified value is estimated using a nonparametric smoothed bootstrap method based on the contributed measurement results for this particular SRM [4].

Elemental Composition: Semi-quantitative elemental analysis of the shellfish matrix is provided in Table 8.

Table 1. Certified Properties of SRM 4358

Radionuclides	**See Table 2 and 3**
Reference time	**16 February 1998**
Certified massic activities	**See Table 2**
Certified activity ratios	**See Table 3**
Uncertainties[a]	**See Table 2 and 3**

[a] For further information on the expression of uncertainties, see references 2, 3, 4, and 5.

Table 2. Certified Massic Activities[a]

Radionuclide	Massic Activity and expanded uncertainty (k = 2) ($mBq \cdot g^{-1}$)
^{241}Am	0.101 ± 0.014
$^{239,240}Pu$	0.055 ± 0.009
^{238}Pu	0.009 ± 0.001
^{238}U	1.46 ± 0.12
^{234}U	1.56 ± 0.09
^{235}U	0.061 ± 0.013
^{232}Th	0.642 ± 0.052
^{230}Th	0.413 ± 0.044
^{228}Th	1.34 ± 0.25
^{228}Ra[b]	1.33 ± 0.41
^{137}Cs	0.254 ± 0.049
^{210}Pb	6.9 ± 1.1
^{40}K	160 ± 16

[a] A sample size of at least 30 grams is recommended for radiochemical analysis. Refer to Table 6 for associated information
[b] Radium-228 activity values are based on measurements of its ^{228}Ac daughter.

Table 3. Certified Activity Ratios

Radionuclides Ratio	**Ratio and expanded uncertainty (*k* = 2)**
^{234}U / ^{238}U	**1.142 ± 0.048**
^{235}U / ^{238}U	**0.041 ± 0.008**
^{238}Pu / (^{239}Pu+^{240}Pu)	**0.177 ± 0.016**
^{228}Th / ^{232}Th	**2.00 ± 0.61**
^{230}Th / ^{232}Th	**0.654 ± 0.055**

Massic Activities and Mass Ratios for Uncertified Radionuclides: The massic activities and mass ratios for the radionuclides given in Table 5 are not certified at this time, but may be certified at some future time if additional data become available. Users are invited to submit measurement data to contribute to the certification process. The data should be sent to one of the technical contacts listed on page 1.

Table 4. Informational Properties of SRM 4358

Source description	Freeze-dried radiation sterilized shellfish powder, approximately 150 g in a polyethylene bottle
Radionuclide Massic Activity and Radionuclide Ratio Minima and Maxima	See Table 5
Half-lives used	See Table 6
Radiochemical and detection methods	See Table 6 and 7
Elemental composition	See Table 8
Participating laboratories and personnel	See Table 6 and 9

Table 5. Uncertified Radionuclides[(a)] and Range of Reported Values

Radionuclide	Number of Participants	Number of Subsamples	Minimum Value ($mBq \cdot g^{-1}$)	Maximum Value ($mBq \cdot g^{-1}$)
^{244}Cm	2	6	0.001	0.014
^{242}Cm	1	5	0.014	0.015
^{241}Pu	2	10	0.47	0.61
^{226}Ra	2	8	0.08	0.56
^{214}Pb	1	1	0.70	0.70
^{214}Bi	1	1	0.32	0.32
^{212}Pb	2	6	1.2	1.8
^{208}Tl	2	6	0.30	1.5
^{90}Sr	3	10	0.031	0.180
Radionuclide Ratio	Number of Participants	Number of Subsamples	Minimum Activity Ratio Value	Maximum Activity Ratio Value
^{241}Pu / ^{238}Pu	1	5	50.5	65.5
^{241}Pu / $^{239,240}Pu$	1	5	8.5	11.1

(a) Radionuclides (or ratios) for which insufficient numbers of laboratories data sets or for which unresolved discrepant data sets were obtained. As with all information tables, the presented values are to be interpreted cautiously. In particular, the minimum and maximum values presented in the right-most columns of Table 5 are the extremal values obtained by the participating expert laboratories, and due to the small number of data points, such extremal values are known to be notoriously noisy. The reported extremal values are informational and customer labs should not expect their own values to necessarily fall within the reported minima and maxima values.

Table 6. Supporting Information

Radionuclides	Number of Laboratories (total assays)	Half Life[a]	Methods (Table 7)	Contributing Laboratories Acronym (Table 9)
^{241}Am	5 (23)	(432.6 ± 0.6) a	1a, 2b, 3b	IAEA, IPSN, IRMM, NIST, TRMC
^{239,240}Pu	9 (38)	(24100 ± 11) a (6561 ± 7) a	2b, 3b	IAEA, IPSN, JCAC, NAREL, NIRS, NIST, STUK, TRMC, Typhoon
^{238}Pu	6 (28)	(87.7 ± 0.1) a	2b, 3b	IAEA, IPSN, JCAC, NIRS, NIST, TRMC
^{238}Pu / (^{239}Pu+^{240}Pu)	6 (28)	(87.7 ± 0.1) a (24100 ± 11) a (6561± 7) a	2b, 3b	IAEA, IPSN, JCAC, NIRS, NIST, TRMC
^{238}U	10 (46)	(4.468 ± 0.005) 10^9 a	2b, 3b, 3e	ANSTO, IAEA, INER, IRMM, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{234}U	8 (36)	(2.455±0.006) 10^5 a	2b, 3b	IAEA, INER, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{235}U	8 (34)	(7.04 ± 0.01) 10^8 a	2b, 3b	IAEA, INER, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{234}U / ^{238}U	8 (36)	(2.455 ± 0.006) 10^5 a (4.468 ± 0.006) 10^9 a	2b, 3b, 3e	IAEA, INER, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{235}U / ^{238}U	8 (34)	(7.04 ± 0.01) 10^8 a (4.468 ± 0.006) 10^9 a	2b, 3b	IAEA, INER, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{232}Th	8 (34)	(1.402 ± 0.006) 10^{10} a	2b, 3b, 3 e	ANSTO, IAEA, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{230}Th	7 (29)	(7.538 ± 0.030) 10^4 a	2b, 3b	IAEA, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{228}Th	8 (34)	(698.60 ± 0.23) d	2b, 3b	IAEA, IRMM, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{228}Th / ^{232}Th	7 (29)	(698.60 ± 0.23) d (1.402 ± 0.006) 10^{10} a	2b, 3b, 3c	IAEA, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{230}Th / ^{232}Th	7 (29)	(7.538 ± 0.030) 10^4 a (1.402 ± 0.006) 10^{10} a	2b, 3b, 2c	IAEA, JCAC, NAREL, NIRS, NIST, STUK, TRMC
^{228}Ra[b]	3 (15)	(5.75 ± 0.04) a	1a	IRMM, JCAC, Typhoon
^{210}Pb	4 (12)	(22.23 ± 0.12) a	1a	IPSN, IRMM, JCAC, STUK
^{137}Cs	7 (27)	(30.05 ± 0.08) a	1a	IPSN, IRMM, JCAC, NIRS, TRMC, STUK, Typhoon
^{90}Sr	3 (10)	(28.80 ± 0.07) a	2c, 2d, 3c	JCAC, TRMC, Typhoon
^{40}K	4 (12)	(1.2504 ± 0.0030) 10^9 a	1a	IPSN, IRMM, STUK, Typhoon
^{244}Cm	2 (6)	(18.11 ± 0.03) a	2b	INER, IPSN
^{242}Cm	1 (5)	(162.86 ± 0.08) d	2b	INER
^{241}Pu	2 (10)	(14.33 ± 0.04) a	2d	INER, JCAC
^{226}Ra	2 (8)	(1600 ± 7) a	1a	IRMM, TRMC
^{214}Pb	1 (1)	(26.8 ± 0.9) min	1a	IPSN
^{214}Bi	1 (1)	(19.9 ± 0.4) min	1 a	IPSN
^{212}Pb	2 (6)	(10.64 ± 0.01) h	1a	IPSN, Typhoon
^{208}Tl	2 (6)	(3.060 ± 0.008) min	1a	IPSN, Typhoon
Radionuclide Ratio	Number of Laboratories (total assays)	Half Life[a]	Methods (Table 7)	Contributing Laboratories Acronym (Table 9)
^{241}Pu / ^{239,240}Pu	1 (5)	(14.33 ± 0.04) a (24100 ± 11) a (6561 ± 7) a	2d	JCAC
^{241}Pu / ^{238}Pu	1 (5)	(14.33 ± 0.04) a (87.7 ± 0.1) a	2d	JCAC

[a] The stated uncertainty of the half-life is the standard (i.e., k = 1) uncertainty. See references 6 and 7.

[b] Radium-228 activity values are based on measurements of its ^{228}Ac daughter.

Table 7. Radiochemical and Detection Methods

1	Non-destructive
2	Total decomposition
3	Acid leach (any combination of the following HNO_3, HCl, HF, $HClO_4$)
a	Germanium gamma-ray spectrometry
b	Silicon alpha-particle spectrometry
c	Beta-particle counter
d	Liquid scintillation counting
e	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), Atomic Mass Spectrometry (AMS)

Table 8. Semi-quantitative composition of SRM 4358 determined by X-ray fluorescence analysis from ashed shellfish material.

Element	Percent by mass (%)
F	<0.01
Na_2O	20
MgO	6.3
Al_2O_3	2.3
SiO_2	8.2
P_2O_5	32
SO_3	8.8
Cl	6.0
K_2O	7.5
CaO	7.8
TiO_2	0.11
Cr_2O_3	<0.01
MnO	0.097
Fe_2O_3	1.2
NiO	0.010
CuO	0.013
ZnO	0.24
Br	0.15
SrO	0.053
Y_2O_3	0.002
I	0.018
BaO	0.022
Total	101

Table 9. Participating Laboratories and Technical Points of Contact

Laboratory Acronym	Laboratory	Country	Technical Contact
ANSTO	Australian Nuclear Science and Technology Organization	Australia	Dr. Henk van der Gaast
IAEA	International Atomic Energy Agency Marine Environmental Laboratory	Monaco	Dr. P.P. Povinec
INER	Institute of Nuclear Energy Research	Taiwan	Dr. Lee Chung Men
IPSN	lnstitut de Protection et de Surete Nucleaire	France	Dr. M.D. Calmet
IRMM	Institute for Reference Material and Measurements	Belgium	Dr. T. Altzitzoglou
JCAC	Japan Chemical Analysis Center	Japan	Dr. Yoshinori Takata
NAREL	National Air and Radiation Environmental Laboratory	United States of America	Dr. Mary Wisdom
NIRS	National Institute of Radiological Sciences	Japan	Dr. Kiyoshi Nakamura
NIST	National Institute of Standards and Technology	United States of America	S. Nour, Dr. J. LaRosa
STUK	Research and Environmental Surveillance	Finland	Dr. Pia Vesterbacka
TRMC	Taiwan Radiation Monitoring Center	Taiwan	Mr. Ching-Chung Huang
Typhoon	Scientific Production Association Typhoon	Russia	Dr. S.M. Vakulovsky

REFERENCES

[1] Bock, R.; *A Handbook of Decomposition Methods in Analytical Chemistry*; International Textbook Company, Limited. T. & A. Constable Ltd.: Great Britain (1979).

[2] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement; Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed April 2015).

[3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed April 2015).

[4] Efron, B.; Tibshirani, R.J; *An Introduction to the Bootstrap*; Monographs on Statistics & Applied Probability 57, Chapman & Hall: New York (1993).

[5] 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://physics.nist.gov/Pubs/guidelines/TN1297/tn1297s.pdf (accessed April 2015).

[6] 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 April 2015).

[7] National Nuclear Data Center, Brookhaven Laboratory; *Evaluated Nuclear Structure Data File (ENSDF);* Upton, NY (2006); available at http://www.nndc.bnl.gov/ensdf/ (accessed April 2015).

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.