



Certificate of Analysis

Standard Reference Material[®] 2702

Inorganics in Marine Sediment

This Standard Reference Material (SRM) is marine sediment collected at the mouth of the Baltimore Harbor, Baltimore, MD and is intended for use in evaluating analytical methods for the determination of selected elements in marine or fresh water sediment and similar matrices. All of the constituents in SRM 2702, for which certified, reference, and information values are provided, were naturally present in the sediment material before processing. A unit of SRM 2702 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment material.

Certified Mass Fraction Values: Certified values, expressed as mass fractions, for elements are provided in Table 1. The certified values are based on the agreement of results from two or more independent analytical techniques performed at NIST and collaborating laboratories [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account.

Reference Mass Fraction Values: Reference values, expressed as mass fractions, are provided for selected elements in Table 2. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

Information Mass Fraction Values: Information values, expressed as mass fractions, are provided in Table 3 for selected elements. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or only a limited number of analyses were performed [1]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2702** is valid, within the measurement uncertainty specified, until **01 October 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage and Use"). However, 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by R.L. Zeisler of the NIST Chemical Sciences Division.

Preparation and collection of the sediment material was completed by M.P. Cronise and C.N. Fales of the NIST Office of Reference Materials and B.J. Porter and M.M. Schantz of the NIST Chemical Sciences Division. Assistance in collecting the sediment material was provided by G.G. Lauenstein, J. Collier, and J. Lewis of the National Oceanic and Atmospheric Administration (NOAA).

Statistical analysis was provided by M. Vangel and W.S. Liggett, Jr., formerly of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Gaithersburg, MD 20899
Certificate Issue Date: 01 May 2019
Certificate Revision History on Last Page

Analytical measurements at NIST were performed by C.M. Beck II, T.A. Butler, R. Demiralp Oflaz, M.E. Howard, R.M. Lindstrom, S.E. Long, E.A. Mackey, J.L. Mann, K.E. Murphy, R.L. Paul, J.R. Sieber, R.D. Vocke, Jr., L.J. Wood, L.L. Yu, and R.L. Zeisler of the NIST Chemical Sciences Division.

Certification of SRM 2702 was done in collaboration with S. Willie, J.W.H. Lam, C. Scriver, and L. Yang of the National Research Council Canada, Institute for National Measurement Standards, Chemical Metrology and S.A. Wilson, M. Adams, P.H. Briggs, P. Lamothe, D. Siems, and J. Taggart of the U.S. Geological Survey, Branch of Geochemistry (Denver, CO).

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

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Handling: This material is a naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicity; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 2702 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2702 are reported on a dry-mass basis. The SRM, as received, contains approximately 2.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis or a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate subsample. The drying procedures described below, or equivalent, are recommended; the temperature of 90 °C shall **NOT** be exceeded.

PREPARATION AND ANALYSIS⁽¹⁾

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore Harbor, Baltimore, MD near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is near the site where SRMs 1941 and 1941a *Organics in Marine Sediment* were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 70 µm (100 % passing), homogenized in a cone blender, radiation sterilized at 33 kGy to 45 kGy (⁶⁰Co) dose, and then packaged in screw-capped amber glass bottles, each containing approximately 50 g.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 2702 are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 2702 was determined by measuring the mass loss after freeze drying subsamples of 1.1 g to 1.3 g for four days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 2702 was 2.39 % ± 0.08 % (95 % confidence level). Alternatively, drying in a conventional oven was studied. One gram portions were dried at 90 °C for 12 h and 18 h; a stable weight was achieved after 18 h. The measured moisture content was 2.27 % ± 0.09 % (95 % confidence level).

Homogeneity Assessment: The homogeneity of SRM 2702 was assessed by analyzing duplicate samples of approximately 0.1 g from fifteen bottles selected by stratified random sampling. Results from direct determinations by instrumental neutron activation analysis (INAA) and energy-dispersive X-ray fluorescence analysis (ED-XRF) showed observed standard deviations for all reported elements within the precision of the analytical techniques. No statistically significant differences among bottles were observed for the elements at this sample size.

⁽¹⁾ Certain commercial equipment, instruments or materials are 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.

Analytical Approach: NIST has a number of analytical techniques available for value assignment. The ones applied to this SRM include dissolution based techniques such as thermal or inductively coupled plasma isotope dilution mass spectrometry (TID-MS or ICP-IDMS) and direct analysis techniques such as INAA, neutron capture prompt gamma activation analysis (PGAA), and radiochemical neutron activation analysis (RNAA). Several of these techniques were also used by collaborating laboratories and complemented by additional dissolution techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma atomic emission spectrometry (ICP-AES) and mass spectrometry (ICP-MS) and direct analysis techniques such as wavelength dispersive X-ray fluorescence (WD-XRF) and ED-XRF. Care has been taken to obtain results from both chemically destructive techniques and non-destructive or direct analysis techniques.

Certified Values: Certified values, derived from the results of several analytical methods performed by NIST and collaborating laboratories, are provided in Table 1. A complete description of the modes of value assignment is given in reference 1. For consistency across elements, a Bayesian statistical model was chosen for computation of certified values and uncertainties [2]. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence). The reporting follows the ISO/JCGM Guide [3].

For each element, there is a NIST result with an uncertainty that is complete in coverage of recognized sources of uncertainty, complemented by results from collaborating laboratories with similarly complete uncertainties, and usually several results without complete uncertainties. The uncertainties of the latter results were augmented on the basis of the differences among the results obtained by different methods [4]. The Bayesian methods combine results by different methods from different laboratories according to the results provided and the uncertainties provided with them. Thus, a consistent methodology was used for all elements. The total mass fraction for each element is listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram or as a percent).

Table 1. Certified Mass Fraction Values for Elements in SRM 2702

	Mass Fraction mg/kg (unless noted as %)		Mass Fraction mg/kg (unless noted as %)
Aluminum (Al) ^(a,b,c,d)	8.41 % ± 0.22 %	Nickel (Ni) ^(a,b,e,f,g,h)	75.4 ± 1.5
Antimony (Sb) ^(a,d,e,f)	5.60 ± 0.24	Phosphorus (P) ^(a,b,d,j)	0.1552 % ± 0.0066 %
Arsenic (As) ^(a,b,d,g)	45.3 ± 1.8	Potassium (K) ^(a,b,c,d)	2.054 % ± 0.072 %
Barium (Ba) ^(a,e,h)	397.4 ± 3.2	Rubidium (Rb) ^(a,d,e)	127.7 ± 8.8
Cadmium (Cd) ^(a,f,g,h,i)	0.817 ± 0.011	Scandium (Sc) ^(a,b,d)	25.9 ± 1.1
Cerium (Ce) ^(a,b,d,e)	123.4 ± 5.8	Sodium (Na) ^(a,b,c,d)	0.681 % ± 0.020 %
Chromium (Cr) ^(a,b,d,e,f)	352 ± 22	Strontium (Sr) ^(a,b,e,h)	119.7 ± 3.0
Cobalt (Co) ^(a,b,d)	27.76 ± 0.58	Thallium (Tl) ^(f,h)	0.8267 ± 0.0060
Lanthanum (La) ^(a,b,d,e)	73.5 ± 4.2	Thorium (Th) ^(a,b,d,e)	20.51 ± 0.96
Lead (Pb) ^(a,b,e,f,h)	132.8 ± 1.1	Titanium (Ti) ^(a,b,c,d)	0.884 % ± 0.082 %
Manganese (Mn) ^(a,b,d)	1757 ± 58	Vanadium (V) ^(a,b,d)	357.6 ± 9.2
Mercury (Hg) ^(g,j,k)	0.4474 ± 0.0069	Zinc (Zn) ^(a,b,d,e,f)	485.3 ± 4.2

- (a) ICP-MS
- (b) ICP-AES
- (c) WD-XRF
- (d) NIST method - INAA
- (e) ED-XRF
- (f) ICP-IDMS
- (g) AAS
- (h) NIST method - ICP-IDMS
- (i) PGAA
- (j) NIST method - RNAA
- (k) NIST method - Cold vapor ICP-IDMS

Reference Values and Uncertainties: Reference values are based on results from one method carried out in several laboratories or from two or more analytical methods without NIST results. The Bayesian methods of combining the results by different methods from different laboratories were applied as above. These results do not fulfill the criteria for certification, because a full estimate of method bias or results from NIST methods have not been available. The reporting follows the ISO/JCGM Guide [3]. The total mass fraction for each selected element is listed in Table 2 as determined by the methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram or as a percent).

Table 2. Reference Mass Fraction Values for Selected Elements in SRM 2702

	Mass Fraction mg/kg (unless noted as %)		Mass Fraction mg/kg (unless noted as %)
Calcium (Ca) ^(a,b,c)	0.343 % ± 0.024 %	Molybdenum (Mo) ^(a,b,f)	10.8 ± 1.6
Copper (Cu) ^(a,b,d,e,f)	117.7 ± 5.6	Selenium (Se) ^(a,b,f)	4.95 ± 0.46
Gallium (Ga) ^(a,b,f)	24.3 ± 1.9	Silver (Ag) ^(d,e)	0.622 ± 0.078
Magnesium (Mg) ^(a,b,c)	0.990 % ± 0.074 %	Tin (Sn) ^(b,d,f)	31.6 ± 2.4

(a) ICP-MS

(b) ICP-AES

(c) WD-XRF

(d) ICP-IDMS

(e) AAS

(f) ED-XRF

Information Values: Information values are given in SRM 2702 to assist users in the assays of non-certified elements. Information values are based on results that did not allow complete assessment of all sources of uncertainty; hence, only estimated means without uncertainties are given.

Table 3. Information Mass Fraction Values for Selected Elements in SRM 2702

	Mass Fraction mg/kg (unless noted as %)		Mass Fraction mg/kg (unless noted as %)
Beryllium (Be) ^(a,b)	3.0	Neodymium (Nd) ^(d)	56
Carbon (C total) ^(c)	3.36 %	Niobium (Nb) ^(b,e)	63
Carbon (C organic) ^(c)	3.27 %	Samarium (Sm) ^(d)	10.8
Cesium (Cs) ^(a,d)	7.1	Sulfur (S) ^(c)	1.5 %
Hafnium (Hf) ^(d)	12.6	Tungsten (W) ^(d)	6.2
Iron (Fe) ^(d)	7.4 %	Uranium (U) ^(a,e)	10.4
Lithium (Li) ^(a,b)	78.2		

(a) ICP-MS

(b) ICP-AES

(c) Combustion techniques

(d) NIST method - INAA

(e) ED-XRF

REFERENCES

- [1] 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.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF> (accessed May 2019).
- [2] Gelman, A.; Carlin, J.B.; Stern, H.S.; Rubin, D.R.; *Bayesian Data Analysis*; Chapman & Hall: London (1995).
- [3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (ISO 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2019); see also 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 May 2019).
- [4] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-k.; Vangel, M.G.; Yen, J.H.; Zhang N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

Certificate Revision History: 01 May 2019 (Fe reinserted as an information value in Table 3; editorial changes); 17 May 2016 (Fe value in Table 1 temporarily removed while being revised; editorial changes); 16 July 2012 (Extension of certification period; editorial changes); 07 January 2004 (This revision reflects a corrected Cu reference value in Table 2, an updated Hg certified value and uncertainty in Table 1, and editorial revisions.); 03 December 2002 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis 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 <https://www.nist.gov/srm>.