

Standard Reference Material[®] 2983

Inorganics in Geoduck Clam Tissue (*Panopea generosa*)

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for validating methods for determining total arsenic (As) and inorganic arsenic (iAs) in geoduck clam tissue and similar materials.

Description: A unit of SRM 2983 consists of one jar sealed inside an aluminized pouch. The jar contains approximately 12 g of cryogenically homogenized frozen tissue material.

Certified Values: The certified values and uncertainties for total As and iAs in SRM 2983 are listed in Table 1. A 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 accounted for [1]. The certified mass fractions in Table 1 are metrologically traceable to the International System of Units (SI) unit of mass fraction expressed as milligrams per kilogram on a wet-mass basis [1,2].

Table 1. Certified Mass Fraction Values for Total Arsenic and Inorganic Arsenic in SRM 2983

Analyte	Mass Fraction ^(a) (mg/kg)
Total Arsenic (As)	3.677 ± 0.063
Inorganic Arsenic (iAs)	0.193 ± 0.011

^(a) Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value, $U_{95\%}(x)$ is the expanded uncertainty of the certified value, and coverage factor is $k = 2$ [3–6].

Non-Certified Values: Non-certified values for elements in SRM 2983 are provided in Appendix A.

Additional Information: Methods used for the analysis of SRM 2983 and additional information are provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 2983** are valid within the measurement uncertainty specified until **31 December 2028**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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>).

Safety: SRM 2983 is intended for research use; not for human consumption.

Storage: SRM 2983 is packaged as a cryogenically homogenized frozen material in a glass jar sealed in an aluminized pouch. The tissue homogenate should not be allowed to thaw prior to subsampling for analysis. This material has been stored at NIST at $-80\text{ }^{\circ}\text{C}$ (or lower) since it was prepared and should be stored by the user at this temperature for the certified values to be valid within the stated uncertainties. An open jar can be reused until the material reaches its expiration date, provided that the open bottle is resealed and stored at $-80\text{ }^{\circ}\text{C}$ (or lower).

Use: It is recommended to use a cold workspace (e.g., an insulated container with dry ice, ice packs, or liquid nitrogen coolant on the bottom and pre-cooled implements, such as spatulas, for transferring the frozen powder) when preparing subsamples of this SRM for analysis. Avoid heavy frost buildup by handling the jar quickly and wiping prior to removing material. Subsamples of this SRM for analysis should be withdrawn from the jar immediately after opening and used without delay for the certified values to be valid within the stated uncertainties. To relate analytical determinations to the certified values in this certificate, test portions of the material equal to or greater than 0.35 g for total As and equal to or greater than 0.5 g for iAs should be used. Test portions should be analyzed on a wet-mass basis.

Full details on the production and evaluation of SRM 2983 are provided free of charge in reference 2.

REFERENCES

- [1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Diewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf> (accessed Mar 2024).
- [2] Bryan Sallee, C.E.; Bachman, M.J.; Christopher, S.J.; Ellisor, D.L.; Ellisor, M.B.; Hoguet, J.C.; Huntington, S.L.; Luvonga, C.; Moors, A.J.; Nadarajan, D.; Narukawa, T.; Ness, J.M.; Pugh, R.S.; Yen, J.H.; Yu, L.L.; *Certification of Standard Reference Material[®] 2983 Inorganics in Geoduck Clam Tissue (*Panopea generosa*)*; NIST Special Publication 260-246; National Institute of Standards and Technology, Gaithersburg, MD (2024); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-246.pdf> (accessed Mar 2024).
- [3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (2008); available at <https://www.bipm.org/en/committees/jc/jcgm/publications> (accessed Mar 2024); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; National Institute of Standards and Technology, Gaithersburg, MD (1994); available at <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Mar 2024).
- [4] JCGM 101:2008 *Evaluation of Measurement Data—Supplement 1 to the “Guide to the Expression of Uncertainty in Measurement”—Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (2008); available at <https://www.bipm.org/en/committees/jc/jcgm/publications> (accessed Mar 2024)
- [5] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall, UK (1993).
- [6] Searle, S.; Casella, G.; McCulloch, C.; *Variance Components*; John Wiley, Hoboken, NJ (1992).

If you use this SRM in published work, please reference:

Bryan Sallee CE, Bachman MJ, Christopher SJ, Ellisor DL, Ellisor MB, Hoguet JC, Huntington SL, Luvonga C, Moors AJ, Nadarajan D, Narukawa T, Ness JM, Pugh RS, Yen JH, Yu LL (2024) Certification of Standard Reference Material[®] 2983 Inorganics in Geoduck Clam Tissue (*Panopea generosa*). (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-246. <https://doi.org/10.6028/NIST.SP.260-246>

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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 of Analysis 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 of Analysis * * * * ***

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 SI or other higher-order reference systems [1]. Non-certified mass fraction values are provided below.

Non-certified mass fraction values for elements in SRM 2983, reported on a wet-mass basis, are provided in Table A1.

Table A1. Non-Certified Mass Fraction Values for Elements in SRM 2983

Element	Mass Fraction ^(a) ($\mu\text{g}/\text{kg}$)	
Cadmium (Cd)	650.3	\pm 7.6
Lead (Pb)	104.9	\pm 1.8
Mercury (Hg)	12.4	\pm 0.2
Selenium (Se)	977.3	\pm 41.6

^(a) Values are expressed as $x \pm U_{95\%}(x)$, where x is the non-certified value and $U_{95\%}(x)$ is the expanded uncertainty of the non-certified value [3].

Non-certified values for proximates and calories in SRM 2983, reported on a wet-mass basis, are provided in Tables A2.

Table A2. Non-Certified Values for Proximates and Calories in SRM 2983

Analyte	Mass Fraction ^(b) (%)	
Ash	1.94	\pm 0.08
Protein	14.38	\pm 0.15
Carbohydrates	2.92	\pm 0.33
Total Fat	1.45	\pm 0.21
Moisture	79.35	\pm 0.09

	Energy ^(b) (kcal/100 g)	
Calories	82.15	\pm 0.94

^(b) Values are expressed as $x \pm U$, where x is the non-certified value and U is the expanded uncertainty based on the standard error for that non-certified value mean [3]. While the best estimate of each measurand lies within the interval $x \pm U$, not all components of uncertainty have been considered.

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **31 December 2028**. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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 Appendix 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

Source and Preparation: SRM 2983 was prepared from geoduck clams (*Panopea generosa*) that were harvested in southeastern Alaska and shipped to NIST whole on wet ice overnight for processing and cryogenic homogenization. Prior to cryohomogenization, geoduck clams were rinsed with water to remove any extraneous debris, shucked, the tissue (skin, viscera, and meat) was rinsed a second time, cut into small pieces, and frozen in liquid nitrogen (LN2) vapor phase freezers (at or below $-150\text{ }^{\circ}\text{C}$) until cryomilling. The material was cryomilled multiple times prior to bottling to generate a fresh, frozen powder homogenate and to ensure complete blending. Subsamples (approximately 12 g) were aliquoted into pre-cooled glass jars, sealed in aluminized pouches, and stored at $-80\text{ }^{\circ}\text{C}$ (or lower). Full details of material source and preparation can be found in reference 2.

Analysis: Value assignment of the mass fractions of trace elements, inorganic arsenic, and proximates in SRM 2983 were based on the combination of measurements made by NIST and collaborating laboratories [2]. The analytical methods for each analyte are outlined in Table B1.

Table B1. Methods Used for Analyte Determinations

Analyte ^(a)	Analytical Method	Lab
Total Arsenic (As)	ICP-MS/MS	NIST
Inorganic Arsenic (iAs)	LC-ICP-MS	NIST; NMIJ
Cadmium (Cd)	ICP-MS/MS	NIST
Lead (Pb)	ICP-MS/MS	NIST
Mercury (Hg)	DC AAS	NIST
Selenium (Se)	ICP-MS/MS	NIST
Ash	AOAC 923.03	Covance Laboratories, Madison, WI
Protein	AOAC 968.06 and AOAC 992.15	Covance Laboratories, Madison, WI
Carbohydrates	Calculation	Covance Laboratories, Madison, WI
Total Fat	AOAC 922.06 and AOAC 954.02	Covance Laboratories, Madison, WI
Moisture	AOAC 925.09	Covance Laboratories, Madison, WI
Calories	Calculation	Covance Laboratories, Madison, WI

^(a) Certified analytes shown in **Bold**.

DC AAS Direct combustion atomic absorption spectrometry
 ICP-MS/MS Inductively coupled plasma tandem mass spectrometry
 LC-ICP-MS Liquid chromatography inductively coupled plasma mass spectrometry

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