

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1566a

Oyster Tissue

This Standard Reference Material (SRM) is intended primarily for use in calibrating instrumentation and validating methodology for the chemical analysis of marine bivalve tissue. A unit of SRM 1566a contains approximately 25 grams of oyster tissue.

Certified Concentrations of Constituent Elements: The certified elemental concentrations are shown in Table 1. Certified values are based on results obtained by reference methods of known accuracy; or alternatively, from results obtained by two or more independent and reliable analytical methods. Noncertified values are given, for information only, in Table 2. All values are based on minimum sample size of 250 mg of the dried material.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification is invalid after 5 years from the date of shipping. Please return the attached registration card to register your SRM.

Storage: The material should be kept in its tightly closed, original bottle and stored in a desiccator over Mg(ClO₄)₂ at temperatures between 10-30 °C. It should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight.

Use: A minimum sample weight of 250 mg of the dried material (see Instructions for Drying) is necessary for any certified value in Table 1 to be valid within the stated uncertainty. The bottle should be shaken well before each use, closed tightly immediately after use, and stored as described above.

The statistical analysis of the data was performed by S.B. Schiller and K.R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the analytical chemistry measurements leading to this certificate were performed in the NIST Center for Analytical Chemistry by R. Zeisler.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Gaithersburg, MD 20899 October 2, 1989

Stanley D. Rasberry, Chief Office of Standard Reference Materials

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Instructions for Drying: Before weighing, samples of SRM 1566a should be dried to constant weight by one of the following procedures:

- 1. Reduced-pressure drying at room temperature for 48 hours over Mg(ClO₄)₂ in a vacuum desiccator at approximately 1.3 x 10⁴ Pa (100 mm Hg).
- 2. Vacuum drying at room temperature for 24 hours at a pressure of approximately 30 Pa (0.2 mm Hg) using a cold trap.
 - 3. Freeze drying for 20 hours at a pressure of approximately 3 Pa (0.02 mm Hg).

Source and Preparation of Material: The oysters for this reference material were obtained from a commercial source. They had been shucked, frozen, and packaged in sealed plastic bags. The oyster material was ground, freeze-dried, and powdered at Leon Laboratories, Fort Lauderdale, FL. At NIST, the oyster tissue was jet-milled to pass a 355-um screen, radiation-sterilized, and bottled.

Homogeneity Assessment: Samples from randomly selected bottles of SRM 1566a were analyzed for homogeneity by x-ray fluorescence and neutron activation methods. In addition, results by other analytical methods were examined for evidence of inhomogeneity. The uncertainties in Table 1 include estimates of inhomogeneity.

Table 1. Certified Concentrations of Constituent Elements

Element ¹	Concentration, ² Wt. Percent	Element ¹	Concentration, ² Wt. Percent
Calcium ^{d,e,o} Chlorine ^{g,k,o} Magnesium ^{d,e,k} Phosphorus ^{d,n}	0.196 ± 0.019 0.829 ± 0.014 0.118 ± 0.017 0.623 ± 0.018	Potassium ^{f,k} Sodium ^{d,k} Sulfur ^{g,j}	0.790 ± 0.047 0.417 ± 0.013 0.862 ± 0.019
Element ¹	Concentration, ² µg/g	Element ¹	Concentration, ² µg/g
Aluminum ^{f,k} Arsenic ^{c,k,l} Cadmium ^{b,h,k,l,m} Chromium ^{h,k,l} Cobalt ^{k,l} Copper ^{b,e,h,k,l,m,o} Iodine ^{i,k,l} Iron ^{d,k,o} *Lead ^h	202.5 ± 12.5 14.0 ± 1.2 4.15 ± 0.38 1.43 ± 0.46 0.57 ± 0.11 66.3 ± 4.3 4.46 ± 0.42 539 ± 15 0.371 ± 0.014	Manganese ^{d,e,k,o} Mercury ^{a,l} Nickel ^{b,h,l,o} Selenium ^{k,l,o} Silver ^{h,k,l} Strontium ^{e,h,o} Uranium ^{i,l} Vanadium ^{i,k,l} Zinc ^{d,e,k,l,m,o}	12.3 ± 1.5 0.0642 ± .0067 2.25 ± 0.44 2.21 ± 0.24 1.68 ± 0.15 11.1 ± 1.0 0.132 ± 0.012 4.68 ± 0.15 830 ± 57

Lead was determined by isotope dilution mass spectrometry, inductively coupled plasma, at NIST and at another laboratory.

1. Analytical Methods:

^aAtomic absorption spectrometry, cold vapor

Atomic absorption spectrometry, electrothermal

^cAtomic absorption spectrometry, hydride generation

^dAtomic emission spectrometry, direct current plasma

Atomic emission spectrometry, inductively coupled plasma Atomic emission spectroscopy, flame

glon chromatography

^bIsotope dilution mass spectrometry, inductively coupled plasma

Isotope dilution mass spectrometry, resonance ionization

Isotope dilution mass spectrometry, thermal ionization

Neutron activation, instrumental

Neutron activation, radiochemical

^mPolarography

ⁿSpectrophotometry

[°]X-ray fluorescence spectrometry

2. Based on dry weight. (For drying instructions, please refer to the section of this certificate on Instructions for Drying.)

The certified concentrations are weighted means of results from two or more analytical techniques. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel [1]. Each uncertainty is obtained from a 95% prediction interval plus an allowance for systematic error among the methods used. The allowance for systematic error is equal to the greatest difference between the weighted mean (certified value) and the component means for the analytical methods used. In the absence of systematic error, the resulting uncertainty limits will cover the concentration of approximately 95% of samples of this SRM having a minimum sample size of 250 mg.

Table 2. Noncertified Concentrations of Constituent Elements

Concentration,
Percent by Weight

Nitrogen (Kjeldahl)

(6.81)

Method Reference. Official Methods of Analysis of the Association of Official Analytical Chemists, Arlington, VA, 14th Ed., 1984, p. 16, Nitrogen (Total) in Fertilizers, Kjeldahl Method (Final Action): Method 2.057, Improved Method for Nitrate Free Samples. Mercuric Oxide was used as a catalyst. Samples were dried as described in procedure 2 under "Instructions for Drying".

Element	Concentration, µg/g	ì	Element	Concentration, µg/g
Antimony	(0.01)		Rubidium	(3)
Cerium	(0.4)		Samarium	(0.06)
Cesium	(0.02)		Scandium	(0.06)
Europium	(0.01)		Tantalum	(0.003)
Fluorine	(240)		Terbium	(0.007)
Gold	(0.01)		Thorium	(0.04)
Hafnium	(0.04)		*Tin	(3)
Lanthanum	(0.3)			(5)

Note: There is evidence that tin is inhomogeneously distributed in the SRM.

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[1] R.C. Paule and J. Mandel, Consensus Values and Weighting Factors, NBS J. Research 87, 377-385 (1982).