National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 143d

Cystine
(L-Cystine)

$[-SCH_2CH(NH_2)COOH]_2$

Standard Reference Material (SRM) 143d is intended primarily for use in validating microchemical procedures for the determination of carbon, hydrogen, nitrogen, and sulfur in organic matter. SRM 143d is pure crystalline cystine supplied in a 2 g unit size.

The overall purity of this SRM was assessed at NIST using a variety of methods and techniques. The total impurity is less than 0.2 % relative.

Theoretical Chemical Composition: Pure cystine has the following theoretical chemical composition expressed as fractions (in %) of its total relative molecular mass [1].

Element	Fractions (in %)
Carbon	29.99
Hydrogen	5.03
Nitrogen	11.66
Sulfur	26.69
Oxygen	26.63

NIST recommends that the theoretical composition be used for this material. The analytical techniques used to generate the supplemental information validated the theoretical composition of this material within their experimental error.

Expiration of Certification: The certification of this SRM lot is valid until **30 September 2008**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is contaminated or 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. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 24 July 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Analytical measurements at NIST were performed by S.A. Margolis and M.J. Welch of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical activities were under the chairmanship of M.J. Welch of the NIST Analytical Chemistry Division.

NOTICE AND WARNINGS TO USER

Storage: The SRM should be stored in its original bottle at temperatures between approximately 20 °C and 25 °C. It must be tightly re-capped after use and protected from excessive moisture and light.

Instructions for Use: SRM 143d is not hygroscopic under ordinary conditions of storage as described above, and can be used without preliminary drying.

SOURCE AND ANALYSIS

Source of Material: The SRM material was obtained from Fluka Chemical Corporation, Ronkonkoma, NY.

Analytical Approach: Tests for impurities were performed using a variety of techniques including: liquid chromatography (LC) after reaction with 9-fluorenylmethyl chloroformate to form amino acid derivatives detectable by UV at 260 nm; gas chromatography/mass spectrometry (GC/MS) after forming trimethylsilyl derivatives; and Karl Fischer titration for moisture. No significant impurities were found by either LC or GC/MS. The Karl Fischer titration found negligible water in the material. Because cystine does not melt, differential scanning calorimetry could not be used as an overall measure of the purity. Based upon the tests that were done, total impurities are less than 0.2 % and should not significantly affect the elemental determinations.

SUPPLEMENTAL INFORMATION

To confirm the theoretical chemical composition for carbon, hydrogen, nitrogen, and sulfur, three collaborating laboratories performed the analyses. Fifty bottles of the entire bottled lot were selected by a stratified random sampling. Each laboratory tested three taken from the group of fifty, chosen such that one bottle came from the first third of the bottling run, one from the second third, and one from the final third. Analyses were performed in duplicate from each of the three bottles for each of the four elements: carbon, hydrogen, nitrogen, and sulfur. The three laboratories were: Atlantic Microlab, Inc., Norcross, GA; Galbraith Laboratories, Inc., Knoxville, TN; and Schwartzkopf Microanalytical Laboratory, Inc., Woodside, NY. The results from the three laboratories were in good agreement and agreed well with the theoretical composition. The data from the three laboratories fell within the ranges presented in Table 1.

Table 1. Range of Values (Fractions) for Measured[a] Relative Atomic Masses for Carbon, Hydrogen, Nitrogen, and Sulfur in SRM 143d [2]

Element	Range Fraction (in %)
Carbon	29.83 - 30.01
Hydrogen	4.94 - 5.19
Nitrogen	11.52 - 11.76
Sulfur	26.60 - 26.96
Oxygen[b]	26.46 - 26.82

[a] These results are provided to demonstrate user experience with this material.

[b] Oxygen content is calculated by assuming that all that is not carbon, hydrogen, nitrogen, or sulfur must be oxygen [% O = 100 - (% C + % H + % N + % S]

REFERENCES

[1] IUPAC Commission on Atomic Weights and Isotopic Abundances, "Atomic Weights of the Elements 1995," Pure & Appl. Chem., **68**, p. 2339, (1996).

[2] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1994).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.