

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 143d

Cystine

This Standard Reference Material (SRM) is certified as a neat chemical material of known purity and is intended for use in validating microchemical procedures for the determination of carbon, hydrogen, nitrogen, and sulfur in organic matter. A unit of SRM 143d consists of 2 g of high-purity crystalline cystine.

Certified Chemical Identity: The chemical identity of cystine is certified with highest confidence. 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 [1]. Metrological traceability of the composition of this SRM is realized through the unambiguous determination of cystine chemical structure and purity.

The theoretical elemental composition of pure cystine can be used for validation of microchemical procedures with this high-purity material. The theoretical elemental composition of pure cystine is shown in Table 1. The values and uncertainties were derived from the molecular formula of the certified cystine structure ($C_6H_{12}N_2O_4S_2$) using the corresponding IUPAC atomic weights [2,3] and are expressed as fractions (in percent) of total relative molecular mass.

Table 1. Theoretical Elemental Composition of Cystine (C₆H₁₂N₂O₄S₂)

	Mass Fraction	95 % Confidence ^(a)
Element	(%)	(%)
Carbon (C)	29.988	29.984 - 29.992
Hydrogen (H)	5.033	5.033 - 5.034
Nitrogen (N)	11.657	11.656 - 11.659
Oxygen (O)	26.631	26.629 - 26.634
Sulfur (S)	26.689	26.681 - 26.696

^(a) With about 95 % confidence, the true value of the mass fraction of the element in completely pure cystine is expected to be within the stated Interval. These uncertainties arise from naturally occurring isotopic variability as documented in [2].

Certified Purity Value: The certified purity value was determined using a quantitative ¹H nuclear magnetic resonance spectroscopy (¹H-qNMR) primary ratio measurement procedure [4,5]. The measurand is the mass fraction (expressed as percentage) of cystine [6] and the uncertainty is expressed as the 95 % confidence interval ($U_{95\%}$) [7,8]. Metrological traceability is to the derived SI measurement unit for chemical mass fraction [9].

Table 2. Certified Mass Fraction of Cystine in SRM 143d

	Mass Fraction	Confidence Interval (U95%)
	(%)	(%)
Cystine	99.9	99.7 - 100.0

Expiration of Certification: The certification of **SRM 143d** is valid, within the measurement uncertainty specified, until **31 December 2028**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Overall direction and coordination of the technical activities were under the chairmanship of M.A. Nelson of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief Chemical Sciences Division

Steven J. Choquette, Director

Office of Reference Materials

Gaithersburg, MD 20899 Certificate Issue Date: 30 September 2020 *Certificate Revision History on Last Page* **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.

Analytical measurements at NIST were performed by M.A Nelson, S.A. Margolis, and M.J. Welch of the NIST Chemical Sciences Division.

Statistical analysis was provided by B. Toman of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in its original container at temperatures between 20 °C and 25 °C. It should be tightly closed after use and protected from moisture, heat, and direct light.

Use: SRM 143d stored as described above, should be used without preliminary drying. The recommended minimum sample size is 5 mg.

SOURCE AND ANALYSIS

Source of Material: The SRM source material was obtained from a commercial supplier.

Analytical Approach: Analyses for chemical identity and purity were performed by NIST using twenty-two units, selected at regular intervals across the entire production lot. A ¹H-qNMR primary ratio measurement procedure using an internal standard approach was implemented for the determination of cystine mass fraction with metrological traceability to SI units through the NIST PS1 Primary Standard for quantitative NMR (Benzoic Acid). Calculation of the 95 % coverage interval was performed under the Bayesian paradigm using a hierarchical measurement model for the ¹H-qNMR procedure [10,11]. No trend in mass fraction of cystine was observed with respect to filling order and there is no significant heterogeneity at the 95 % confidence level. Confirmatory assessments of impurity components were made via liquid chromatography with ultraviolet detection (LC-UV) and water analysis by Karl Fischer titration.

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Certificate Revision History: 30 September 2020 (Corrected the transposed values of oxygen and sulfur in Table 1; editorial changes); 10 May 2019 (Title updated; change of expiration date; theoretical chemical composition was replaced with calculated elemental composition; cystine mass fraction and confidence interval were added; supplemental information was removed; editorial changes); 24 February 2017 (Editorial changes); 31 August 2010 (Extension of the certification period; editorial changes); 24 July 1998 (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.