National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 17e

Sucrose
Optical Rotation

This Standard Reference Material (SRM) is intended primarily for use as a saccharimetry standard in calibrating polarimetric systems. The certified chemical purity and reference values for the optical rotation of a "normal solution" of SRM 17e, Sucrose, at 20.00 °C ± 0.01 °C in a 100.00 mm cell and a 200.00 mm cell are provided. A unit of this SRM consists of one bottle containing 60 g of crystalline sucrose.

Certified Purity and Uncertainty: The certified chemical purity was determined by measuring the mass fraction of impurities, including water, other saccharides, and residue from ashing, summing the impurities, and subtracting this sum from 100 %.

Certified Purity of Sucrose as a Mass Fraction (in %): 99.950 ± 0.016

The uncertainty in the certified value is expressed as an expanded uncertainty, U, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [1]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the uncertainty in the measurement of the impurities. The coverage factor, $k = 2.57$, is determined from the Student's t-distribution corresponding to 5 degrees of freedom and 95 % confidence.

Reference Values and Uncertainties: Reference values for the optical rotation of SRM 17e at four wavelengths are provided in Table 1a in both milliradians and degrees. Analyses for value assignment were performed by collaborating laboratories. Reference values are noncertified values that do not meet 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 methods.

Expiration of SRM Certification: The certification of this SRM is valid until **31 August 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 overall direction and coordination of the technical activities were under the chairmanship of M.J. Welch of the NIST Analytical Chemistry Division.

Willie E. May, Chief
Analytical Chemistry Division

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Analytical measurements were performed by L.T. Sniegoski and S.A. Margolis of the NIST Analytical Chemistry Division, Y. Tewari of the NIST Biotechnology Division, and A.M. Lora-Sanchez (CENAM Guest Scientist).

Statistical analyses were provided by L.M. Gill of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert and B.S. MacDonald of the NIST Measurement Services Division.

NOTICE AND WARNING TO USERS

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.

Drying Instruction: This material does not require drying before use if it is not exposed to excessive moisture.

SOURCE AND PREPARATION

Source of Material[1]**:** The material used for this SRM was provided by the California and Hawaiian Sugar Company, Crockett, CA.

Preparation of a Normal Sugar Solution: For very accurate measurements, solutions of the SRM should be freshly prepared under sterile conditions using pure sterilized water. A "normal sugar solution" is defined as 26.0160 g of "pure" sucrose weighed in vacuum, dissolved in pure water, and diluted to 100.000 cm^3 at 20.00 °C. This is equivalent to 23.7018 g of sucrose per 100.000 g of aqueous solution. Therefore, weigh out 23.7018 g of SRM 17e, and add pure sterile water to a total mass of 100.000 g. Additional information on the preparation of a normal sugar solution can be found in reference [2]. In practice, one can accurately weigh an amount that differs from 23.7018 g and multiply the observed rotation by the ratio of 23.7018 to the actual mass to compare results with the reference values in Table 1a.

The optical rotation of a normal solution of SRM 17e at 546.2271 nm corresponds to 99.965 ± 0.054 °Z of the International Sugar Scale which became effective July 1, 1988. The International Sugar Scale (100 °Z) is based on the optical rotation caused by the normal sugar solution in a 200 mm polariscope tube at 20.00 °C [2].

Additional Analyses: Elemental analysis gave the following results: C, 42.14 % ± 0.21 %; H, 6.49 % ± 0.13 %, compared to the theoretical percentages calculated for $C_{12}H_{22}O_{11}$: C, 42.10; H, 6.48 %. Thus the theoretical composition is well within the uncertainty of the elemental analysis measurements. Karl Fischer titration found a moisture content of approximately 0.18 mg/g.

ICUMSA Equation: Using the rotatory dispersion equation below given by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) [3], values for the optical rotation at 632.9914 nm and 882.60 nm were calculated from the measured values at 546.2271 nm. These values are shown in Table 1a. This equation was also used to calculate a value for the optical rotation at 589.4400 nm. This value agreed to within 0.0007 % of the average measured rotation for this SRM at 589.4400 nm.

$$\frac{\alpha_\lambda}{\alpha_{0.5462271}} = \frac{1}{a_0 + a_1 \cdot \lambda^2 + a_2 \cdot \lambda^4 + a_3 \cdot \lambda^6}$$

a_0 = -0.075047659000

a_1 = 3.588221904585

a_2 = 0.051946178300

a_3 = -0.006515194377

λ = wavelength in µm

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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.

Table 1a. Reference Values for the Optical Rotation and Associated Uncertainties[a] of a "Normal Sugar Solution" of SRM 17e at 20.00 °C ± 0.01 °C

Wavelength	100.00 mm cell Optical Rotation		200.00 mm cell Optical Rotation	
in vacuo, nm	mrad	degrees	mrad	degrees
546.2271	355.72 ± 0.19	20.381 ± 0.011	711.45 ± 0.38	40.763 ± 0.022
589.4400	302.06 ± 0.25	17.307 ± 0.014	604.13 ± 0.49	34.614 ± 0.028
632.9914[b]	259.54 ± 0.14	14.870 ± 0.008	519.08 ± 0.28	29.741 ± 0.016
882.60[b]	129.42 ± 0.07	7.415 ± 0.004	258.84 ± 0.14	14.831 ± 0.008

The optical rotation measurements at 546.2271 nm and 589.4400 nm were made using state-of-the-art polarimeters at JASCO, Inc., Easton, MD, and at Bodenswerk Perkin-Elmer GmbH, Uberlingen, Germany. The reference values for 546.2271 nm and 589.4400 nm are the equally weighted means of results obtained from four instruments.

Table 1b. Reference Value and Associated Uncertainty[a,c] for °Z at 546.2271 nm of a "Normal Sugar Solution" of SRM 17e at 20.00 °C ± 0.01 °C

°Z = 99.965 ± 0.054

The International Sugar Scale (100 °Z) is based on the optical rotation caused by the normal sugar solution in a 200 mm polariscope tube at 20.00 °C, effective July 1, 1988 [2].

Table 1c. Reference Value for the Specific Rotation $[\alpha]_{D}^{20\,°}$ and Associated Uncertainty[a] at 589.4400 nm

Specific Rotation, $[\alpha]_{D}^{20\,°}$: 66.524 ± 0.054

[a] The uncertainties in the reference values are expressed as expanded uncertainties, U, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [1]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effects of method differences, within-method variation, and material inhomogeneity. The coverage factor, k = 3.18, is determined from the Student's *t*-distribution corresponding to three degrees of freedom and 95 % confidence for each wavelength.

[b] The optical rotation values for 632.9914 nm and 882.60 nm are calculated, based upon the measurements at 546.2271 nm - (see section entitled ICUMSA Equation).

[c] The uncertainty for the °Z value also includes a component for uncertainty in the 100 °Z value [2].

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland (1993); 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 http://physics.nist.gov/Pubs/.

[2] ICUMSA Methods Book, Method GS2/3-1 (1994), Conley, Norwich, England NR4 7UB (1994).

[3] Proceedings 22nd Session ICUMSA, p. 209 (1998).

Certificate Revision History: 16 September 2003 (Added a wavelength and updated ICUMSA equation); 04 November 1998 (Original certificate date).

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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.