National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 17f

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" (described below) of SRM 17f 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 SRM 17f consists of one bottle containing 60 g of crystalline sucrose.

Certified Purity and Uncertainty: 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]. The certified chemical purity of sucrose was determined by measuring the mass fractions 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: 99.956 % ± 0.004 %

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 [2]. 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, $k = 2$, is determined from the Student's t-distribution corresponding to the appropriate degrees of freedom and approximately 95 % confidence.

Reference Values and Uncertainties: Reference values are noncertified values that represent a best estimate of the true value; however, the values do not meet the 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 [1]. Reference values for the optical rotation of a "normal sugar solution" of SRM 17f at four wavelengths are provided in Table 2a in both milliradians and degrees (See "Preparation of a Normal Sugar Solution"). Table 2b includes the reference value for the °Z value for a "normal sugar solution" of SRM 17f. The unit, °Z, is the unit of the "International Sugar Scale" defined by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA). The reference value for the specific rotation is provided in Table 2c.

Expiration of Certification: The certification of **SRM 17f** is valid, within the measurement uncertainties specified, until **01 July 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Notice and Warnings to Users"). The certification is nullified if the SRM is damaged, contaminated, or otherwise 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 expiration, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of technical activities leading to certification of this SRM were under the leadership of K.W. Phinney of the NIST Biomolecular Measurement Division.

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Certificate Revision History on Last Page

Analytical measurements were performed by D.W. Bearden, C.A. Rimmer, M.M. Schantz, L.T. Sniegoski, and T.W. Vetter of the NIST Chemical Sciences Division; K.W. Phinney of the NIST Biomolecular Measurement Division; B.E. Lang of the NIST Biosystems and Biomaterials Division; and D.K. Hancock formerly of NIST.

Statistical analyses were provided by N.-F. Zhang of the NIST Statistical Engineering Division.

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

NOTICE AND WARNING TO USERS

Storage and Use: The SRM should be stored in its original bottle at temperatures between approximately 20 °C and 25 °C. With proper storage and handling, this material will not require drying prior to use. It must be tightly re-capped after usage and protected from excessive moisture and light.

SOURCE, PREPARATION, AND ANALYSIS[1]

Source of Material: 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 17f 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 3. 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 2a.

Optical Rotation Analyses: Analyses for value assignment of the optical rotation of SRM 17f provided in Table 2a were performed at JASCO, Inc. (Easton, MD).

The optical rotation of a normal solution of SRM 17f, provided in Table 2b, is based on the International Sugar Scale, which became effective July 1, 1988. The 100 °Z point of the International Sugar Scale corresponds to the optical rotation caused by a normal solution of pure sucrose at the wavelength of 546.2271 nm in a 200.000 mm polariscope tube at 20.00 °C [3].

Additional Analyses: Table 1 provides the elemental analysis results of SRM 17f and the theoretical percentages calculated for $C_{12}H_{22}O_{11}$. Thus, the comparison shows that the theoretical composition is well within the uncertainty of the elemental analysis measurements.

Table 1. Theoretical Composition and Elemental Analysis of[a] $C_{12}H_{22}O_{11}$ for SRM 17f

Element	SRM 17f Results (%)	Theoretical Calculation (%)
C	42.22 ± 0.12	42.10
H	6.47 ± 0.10	6.48

[a] The uncertainties in the elemental analysis results are expressed as expanded uncertainties, *U*, at the 95 % level of confidence, and are calculated according to the method described in the ISO Guide [2]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effects of between-lab variation. The coverage factor, $k = 2$, is determined from the Student's t-distribution corresponding to the appropriate degrees of freedom and approximately 95 % confidence.

Karl Fischer titration yielded a moisture content of approximately 0.097 mg/g. Results of nuclear magnetic resonance (NMR) analysis were consistent with a purity > 99 %, and additional supporting analyses indicated that there was no significant contribution from other saccharides.

[1] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

ICUMSA Equation: Using the rotatory dispersion equation below, given by ICUMSA [4], 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 2a. This equation was also used to calculate a value for the optical rotation at 589.4400 nm. This value agreed to within 0.2 % 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 = –0.075 047 659 000

a_1 = 3.588 221 904 585

a_2 = 0.051 946 178 300

a_3 = –0.006 515 194 377

λ = wavelength (µm)

Table 2a. Reference Values[(a)] for the Optical Rotation of a "Normal Sugar Solution" of SRM 17f at a Temperature of 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	355.68 ± 0.71	20.379 ± 0.041	711.36 ± 1.42	40.758 ± 0.082
589.4400[(b)]	302.03 ± 0.60	17.305 ± 0.035	604.06 ± 1.21	34.610 ± 0.069
632.9914[(b)]	259.51 ± 0.52	14.869 ± 0.030	519.02 ± 1.04	29.737 ± 0.060
882.60[(b)]	129.41 ± 0.26	7.414 ± 0.015	258.81 ± 0.52	14.829 ± 0.030

Table 2b. Reference Value[(a,c)] for °Z at 546.2271 nm of a "Normal Sugar Solution" of SRM 17f at 20.00 °C ± 0.01 °C

°Z = 99.953 ± 0.201

Table 2c. Reference Value[(a)] for the Specific Rotation $[\alpha]_D^{20°}$ of SRM 17f at 589.4400 nm

Specific Rotation, $[\alpha]_D^{20°}$: 66.517 ± 0.134

(a) The uncertainties in the reference values are expressed as expanded uncertainties, *U*, at the 95 % level of confidence, and are calculated according to the method described in the ISO Guide [2]. 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 within-method variation and a Type B uncertainty component. The coverage factor *k* is determined from the Student's *t*-distribution corresponding to the appropriate degrees of freedom and approximately 95 % confidence.

(b) The optical rotation values for 589.4400 nm, 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 [3].

REFERENCES

[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed March 2013).

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed March 2013); 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://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed March 2013).

[3] ICUMSA – Methods Book, Method GS2/3-1; *The Braunschweig Method for the Polarization of White Sugar by Polarimetry*; ICUMSA Publication Department: Norwich Research Park NR4 7UB, England (1994).

[4] ICUMSA Proceedings 1998, 22nd Session; Bartens: Berlin, Germany; p. 209 (1998).

Certificate Revision History: 29 March 2013 (Extension of certification period, editorial changes); 09 June 2010 (This revision includes an updated certified value for purity, updated uncertainties for the elemental analysis, updated reference values for optical rotation, and minor editorial changes.); 26 May 2009 (Editorial revisions); 08 August 2008 (Original certificate issue 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 http://www.nist.gov/srm.