National Institute of Standards and Technology
U.S. Department of Commerce

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Standard Reference Material® 17g

Sucrose

Optical Rotation

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended for use as a saccharimetry standard in calibrating polarimetric systems.

Description: This SRM comprises crystalline sucrose that is certified as a chemical substance of known purity. A unit of SRM 17g consists of one bottle containing approximately 60 g of sucrose.

Certified Value: 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 measurand is the mass fraction of sucrose (expressed in percent) in the material [2].

The certified chemical purity value was determined using a quantitative ^{1}H-nuclear magnetic resonance spectroscopy (q^{1}H-NMR) primary ratio measurement procedure [3,4]. This value is metrologically traceable to the International System of Units (SI) unit of mass, expressed as the mass percentage of sucrose in the material, through use of the NIST PS1 Primary Standard for quantitative NMR (Benzoic Acid) as a calibrant in assigning the certified value [5].

Table 1. Certified Value for Chemical Purity of SRM 17g

Constituent	Purity as Mass Fraction[a,b] (%)	95 % Coverage Interval[a] (%)
Sucrose	99.941	[99.761, 100.000]

[a] The certified value is expressed as the median and the 2.5th and 97.5th percentiles of the result of the purity analysis. The percentiles define an uncertainty interval about the median that specifies a range of values attributable to the measurand with a confidence level of approximately 95 % [6]. For guidance on using and propagating uncertainties described by asymmetrical coverage intervals, see section 7.3 of reference 7.

[b] For some purposes, the sucrose mass fraction can be treated as following the symmetric distribution (99.9 ± 0.1) %, where the ± term is the expanded uncertainty indicating the range of values in which the true value of the analyte is expected to lie with a confidence level of approximately 95 %. For guidance on using and propagating this uncertainty, see reference 7.

Non-Certified Values: Non-certified values for elemental composition and properties associated with the optical activity of SRM 17g are provided in Appendix A.

Period of Validity: The certified value delivered by **SRM 17g** is valid within the measurement uncertainty specified until **31 December 2031.** The certified value is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Value: NIST will monitor this SRM to the end of the period of validity. 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Safety: SRM 17g IS INTENDED FOR RESEARCH USE; NOT FOR HUMAN CONSUMPTION.

Storage: The SRM should be stored in its original container, tightly closed, at temperatures between approximately 20 °C and 25 °C. The SRM should always be protected from moisture, light, and heat. The container should be tightly reclosed immediately after sampling sucrose from the bottle. The previously opened bottle can be reused until the material reaches its expiration date, provided that the opened bottle is immediately reclosed and resealed after sampling, and stored at room temperature.

Use: SRM 17g, stored as described above, should be used without preliminary drying. The minimum sample size is 20 mg.

For use in calibrating polarimetric systems, solutions of the SRM should be freshly prepared under sterile conditions with 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. To prepare an effectively "normal sugar solution", weigh out 23.7018 g of SRM 17g 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 references 8-9. 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 to values for optical rotation of "normal sugar solutions".

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

Analysis: Analyses for chemical identity, purity, and assessment of homogeneity were performed by NIST using twenty-two units of SRM 17g Sucrose Optical Rotation, sampled at regular intervals across the entire production lot. A q^1H-NMR measurement procedure using an internal standard approach was implemented for the determination of sucrose mass fraction. The uncertainty in the certified purity value reflects the 95 % coverage interval of the q^1H-NMR measurement result, calculated using a Bayesian statistical procedure [10]. No trend in mass fraction of sucrose was observed amongst the SRM production lot with respect to filling order and there is no significant heterogeneity at the 95 % level of confidence.

Additional Information: The SRM contains less than 0.04 % moisture. The mass fraction of non-volatile inorganic impurity content, measured as ash residual, was determined to be less than 0.02 %.

Details of the production of this SRM are described in NIST Special Publication 260-217, reference 11.

REFERENCES

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[8] International Commission for Uniform Methods of Sugar Analysis (ICUMSA) – Methods Book, Method GS2/3-1; *The Braunschweig Method for the Polarization of White Sugar by Polarimetry, Official (Reference) Method*; ICUMSA Publication Department: Norwich Research Park NR4 7UB, England (2011).

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If you use this SRM in published work, please reference:

Nelson MA, Lang BE, Mulloor J, Ishikawa M, Kondo Y (2021) Certification of Standard Reference Material® 17g Sucrose Optical Rotation. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-217. https://doi.org/10.6028/NIST.SP.260-217

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: Non-certified values are best estimates based on currently available information. However, they do not meet NIST's criteria for certification [1]. Non-certified values should not be used to establish metrological traceability to the International System of Units (SI) or other higher-order reference system.

Non-certified values for mass fractions of carbon and hydrogen and for properties associated with the optical activity of SRM 17g Sucrose Optical Rotation are provided below.

Table A1. Non-Certified Values for Mass Fraction of Carbon and Hydrogen in SRM 17g[(a)]

Type of Value	Carbon mass fraction[(b)]			Hydrogen mass fraction[(b)]		
	x (%)	s (%)	%Δ[(c)]	x (%)	s (%)	%Δ[(c)]
Observed	42.065	± 0.066	–0.10	6.489	± 0.078	0.17
Theoretical	42.106	± 0.004		6.478	± 0.002	

(a) These values are metrologically traceable to the values for purity and elemental composition of NIST SRM 141e Acetanilide and SRM 350b Benzoic Acid (Acidimetric).

(b) These values are expressed as $x \pm 2s$, where x is the mean value and s is the standard deviation of the analysis results, expressed as % of total mass. While the best estimates of the values lie within the intervals $x \pm 2s$, these intervals might not include the true values. For guidance on using and propagating this uncertainty, see [7].

(c) %Δ is the percent bias of x(Measured) from the theoretical composition, $100\times[x(\text{Measured})/x(\text{Theoretical}) - 1]$

Table A2 Non-Certified Values for Optical Rotation, $\alpha_\lambda^{20.00\ °C}$, of Light Having Wavelength of λ Through 100 mm and 200 mm Pathlengths of Aqueous Solutions at 20.00 °C, Containing Pure Water and 26.0160 g of SRM 17g per 100 cm^3 of Solution[(a)]

λ (nm)	$\alpha_\lambda^{20.00\ °C}$, 100 mm pathlength[(b)] (°)	$\alpha_\lambda^{20.00\ °C}$, 100 mm pathlength[(b)] (mrad)	$\alpha_\lambda^{20.00\ °C}$, 200 mm pathlength[(b)] (°)	$\alpha_\lambda^{20.00\ °C}$, 200 mm pathlength[(b)] (mrad)
546.2271	20.381 ± 0.042	355.71 ± 0.74	40.761 ± 0.084	711.42 ± 1.48
589.4400[(c)]	17.306 ± 0.036	302.05 ± 0.63	34.61 ± 0.072	604.10 ± 1.25
632.9914[(c)]	14.870 ± 0.031	259.53 ± 0.54	29.740 ± 0.062	519.07 ± 1.08
882.60[(c)]	7.415 ± 0.015	129.42 ± 0.27	14.830 ± 0.031	258.84 ± 0.54

(a) These values are metrologically traceable to the angle of rotation of light, having wavelength of 546.2271 nm, passing through 200 mm of a "normal sugar solution" of pure sucrose in pure water, specified as having a mass concentration, c = 26.0160 g/100 cm^3.

(b) These values are expressed as $x \pm 2u(x)$, where x is the value and $u(x)$ is the standard uncertainty of x. The standard uncertainty represents, at the level of one standard deviation, the combined effects on the polarimetric experiments attributed to Type A and Type B uncertainty components. While the best estimates of the values lie within the intervals $x \pm 2u(x)$, these intervals might not include the true values. For guidance on using and propagating this uncertainty, see [7].

(c) The optical rotation values for 589.4400 nm, 632.9914 nm, and 882.60 nm are derived from the measured value for $\alpha_{\lambda=546.2271\ \text{nm}}^{20.00\ °C}$ via the Equation [A1].

Table A3. Non-Certified Value for Specific Rotation of SRM 17g at λ = 589.4400 nm, $[\alpha]^{20.00\ °C}_{\lambda=589.4400\ nm}$[a]

$[\alpha]^{20.00\ °C}_{\lambda=589.4400\ nm}$[b,c] $\left(\frac{° \cdot cm^3}{g \cdot 100\ mm}\right)$
66.522 ± 0.138

[a] This value is metrologically traceable to units of angle (rad), length (m), and mass (g) through gravimetric procedures for the preparation of sample aqueous sucrose solutions, calibration of the polarimetric measurement apparatus and balance, and adequate control of measurement conditions, including temperature, light source frequency and atmospheric pressure.

[b] This value is expressed as $x \pm 2u(x)$, where x is the value and $u(x)$ is the standard uncertainty of x. The standard uncertainty represents, at the level of one standard deviation, the combined effects of Type A and Type B uncertainty components on the polarimetric experiments. While the best estimate of the value lies within the interval $x \pm 2u(x)$, this interval might not include the true value. For guidance on using and propagating this uncertainty, see [7].

[c] This value is based on the calculated value of $\alpha^{20.00\ °C}_{\lambda=589.4400\ nm}$ in Table A2, derived from the value for $\alpha^{20.00\ °C}_{\lambda=546.2271\ nm}$ via Equation [A1].

Table A4. Non-Certified °Z Value on the International Sugar of an Aqueous Solution Containing Pure Water and 26.0160 g of SRM 17g per 100 cm^3 of solution[a]

°Z[b,c]
99.961 ± 0.208

[a] This value is metrologically traceable to the angle of rotation of light, having wavelength of 546.2271 nm, passing through 200 mm of a "normal sugar solution" of pure sucrose in pure water, specified as having a mass concentration, c = 26.0160 g/100 cm^3.

[b] This value is expressed as $x \pm 2u(x)$, where x is the value and $u(x)$ is the standard uncertainty of x. The standard uncertainty represents, at the level of one standard deviation, the combined effects on the polarimetric experiments attributed to Type A and Type B uncertainty components, as well as the uncertainty in the 100 °Z value. While the best estimate of the value lies within the interval $x \pm 2u(x)$, this interval might not include the true value. For guidance on using and propagating this uncertainty, see [7].

[c] This value was derived from the measured value of $\alpha^{20.00\ °C}_{\lambda=546.2271\ nm}$ via the Equation [A2].

Analysis: Microchemical carbon and hydrogen analyses were conducted at NIST and confirmed by measurements performed by an experienced commercial laboratory. The measured non-certified values for mass fractions of carbon and hydrogen in SRM 17g are provided in Table A1.

Polarimetric analyses of SRM 17g were performed at JASCO Corporation (Tokyo, JP) using seven units sampled at regular intervals across the entire production lot. Measurement of values for optical rotation of light at 546.2271 nm (green line of the mercury isotope 198Hg) and 589.4400 nm (sodium D-line) through nominally "normal sugar solutions" prepared from SRM 17g was conducted to establish the non-certified values provided in Tables A2–A4.

Optical rotation values in Table A2 for light of wavelengths other than the green line of mercury isotope ^{198}Hg (546.2271 nm) were calculated using the rotary dispersion equation provided by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) [9,12]:

$$\frac{\alpha^{20.00\ °C}_{\lambda}}{\alpha^{20.00\ °C}_{\lambda=546.2271\ nm}} = \frac{1}{a_0 + a_1 \cdot \lambda^2 + a_2 \cdot \lambda^4 + a_3 \cdot \lambda^6}, \quad [A1]$$

where a_0 is –0.075 047 659 000, a_1 is 3.588 221 904 585, a_2 is 0.051 946 178 300, and a_3 is –0.006 515 194 377.

The ICUMSA International Sugar Scale °Z value for an aqueous solution containing pure water and 26.0160 g of SRM 17g per 100 cm^3 of solution was calculated:

$$°Z = 100\left(\alpha^{20.00\,°C}_{\lambda=546.2271\,nm}/\alpha_{100\,°Z}\right), \quad [A2]$$

where $\alpha_{100\,°Z}$, the value of optical rotation corresponding to the 100 °Z value on the ICUMSA International Sugar Scale, is (40.777 ± 0.001) °, and where the 0.001 value is a 95 % level of confidence expanded uncertainty.
Additional information about the polarimetric analysis and derivation of non-certified values of SRM 17g Sucrose Optical Rotation is provided in [11].

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **31 December 2031**. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix. Before making use of any of the values delivered by this material, users should obtain the most recent version of this documentation, available free of charge through the https://www.nist.gov/srm website.

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