National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 17d

Sucrose

This Standard Reference Material (SRM) is intended for use as a saccharimetry standard in calibrating polarimetric systems. Certified values and the associated uncertainties for the optical rotation of a "normal solution" of SRM 17d, Sucrose, at 20.00 ± 0.01 °C in a 200.00 mm cell, are given below. This SRM is supplied in a unit containing 60 g of crystalline sucrose.

Wavelength in vacuo, nm	Optical Rotation mrad	degrees	Uncertainty[a] mrad	degrees
546.2271	711.64	(40.774°)	± 0.17	(0.010°)
589.4400	604.26[b]	(34.622°)	± 0.12	(0.007°)
632.9914	519.17	(29.746°)	± 0.17	(0.010°)

[a]The uncertainties given here are values based on scientific judgment and include a contribution for minor observed sample heterogeneity. The uncertainties should approximate ± two standard deviations of the certified values.

[b]Calculated value - see section on ICUMSA Equation.

The optical rotation at 589.4400 nm corresponds to a specific rotation, $[\alpha]_D^{20^\circ} = 66.540^\circ$

The optical rotation measurements were made using a high-precision polarimeter. The wavelengths 546 and 633 nm were obtained from a mercury lamp with a filter pack and a helium-neon laser, respectively. The certified values are based on a minimum of nine measurements at each wavelength.

NOTICE AND WARNINGS TO USERS

Stability & Expiration of Certification: When the SRM is stored under normal laboratory conditions, this certification is valid within the specified uncertainty limits for 5 years from the date of shipment. In the event that the certification becomes invalid prior to that time, users will be notified by NIST. Please return the enclosed registration form to facilitate notification.

Measurements of optical rotation were made by D.K. Hancock and other analyses were performed by B. Coxon, S.A. Margolis, and L.T. Sniegoski of the NIST Organic Analytical Research Division, and by F.W. Parrish, of the U.S. Department of Agriculture, New Orleans, LA. Elemental analyses were obtained from Galbraith Laboratories, Knoxville, TN.

Gaithersburg, MD 20899
September 24, 1993
(Revision of certificate dated 8-11-86)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

The technical measurements leading to certification were directed by B. Coxon of the NIST Center for Analytical Chemistry.

Statistical analyses were made by R.C. Paule of the NIST National Measurement Laboratory.

The original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.W. Seward. Revision of this certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

Drying Instruction: This material does not require drying before use.

Preparation of a Normal Sugar Solution: For very accurate measurements, solutions of the SRM should be freshly prepared under pure sterile conditions using pure sterilized water. The normal sugar solution is defined as 26.0160 g of "pure" sucrose weighed in vacuum and dissolved in pure water and diluted to 100.000 cm^3 at 20.00 °C. This is equivalent to 23.7017 g of sucrose per 100.000 g of aqueous solution. Therefore, weigh out 23.7017 g of SRM 17d, and add pure sterile water to a total weight of 100.000 g. [2,3]

The optical rotation of a normal solution of SRM 17d at 546.2271 nm corresponds to 99.993 ± 0.010 °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.

Elemental analysis gave the following results: C, 42.44; H, 6.48% compared to percentages calculated for $C_{12}H_{22}O_{11}$: C, 42.10 H, 6.48%.

This SRM has been analyzed for impurities and the results tabulated below are not certified, but are given for information only:

Impurities	Concentration, μg/g
Polysaccharide (98.8% glucan)	(782)
Moisture	(114)
Invert sugar	(49.6)
Ash	(8)
Raffinose	Not detected

No impurities were detected in deuterium oxide solutions of this SRM, either by ^1H NMR spectroscopy at 400 MHz or by ^{13}C NMR spectroscopy at 100.6 MHz.

The impurity content given above should be stable unless the material is exposed to moisture or microbes.

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

ICUMSA Equation: Using the rotatory dispersion equation below given by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) [4], a value for the optical rotation at 633 nm was calculated from the measured value at 546 nm. This calculated value agrees with the measured value within its uncertainty. The value of the optical rotation at 589.4400 nm is also a calculated value; it is the average value calculated with the ICUMSA equation from the measured values at 546.2271 nm and 632.9914 nm.

$$\frac{\alpha_\lambda}{\alpha_{0.5462271}} = a + \frac{b}{\lambda^2} + \frac{c}{\lambda^4} + \frac{d}{\lambda^8}$$

a= -0.0017982
b= +0.2765318
c= +0.00655736
d= +0.0000103825
λ= wavelength in μm

REFERENCES

[1] Proceedings 15th Session ICUMSA, 42 (1970).
[2] Proceedings 16th Session ICUMSA, 52-74 (1974).
[3] Cummings, A.L., Coxon, B., Layer, H.P., and Hocken, R.J., Proceedings of the 1978 Technical Session on Cane Sugar Refining Research, Sept. 17-19, 1978, Washington, DC, U.S. Department of Agriculture, P.O. Box 19687, New Orleans, LA 70129, pp. 191-204 (1979).
[4] Proceedings 16th Session ICUMSA, 56 (1974).