

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 41c

D-Glucose

(Dextrose)

This Standard Reference Material (SRM) consists of high-purity D-Glucose (dextrose) certified as a chemical of known purity. SRM 41c is intended primarily for use in calibrating polarimetric systems and as an oxidation-reduction standard in the identification of sugar derivatives. This SRM is provided in a unit of 70 g.

The specific optical rotation measurements were made using the NIST high-precision polarimeter [1], which required samples that were both chemically and thermally stable. The measurements were made at 20 °C and a 2 dm polariscope cell was used. Unlike previous certifications of SRMs 41, 41a, and 41b, measurements for this certification were performed without spiking with ammonia.

Specific Optical Rotation Certified Values:

$$[\alpha] \frac{20 \,^{\circ}\text{C}}{520} = 931.8 \,\text{mrad} \,(53.39 \,^{\circ}) \,\pm 0.5 \,\text{mrad}$$

$$[\alpha] \frac{20 \, ^{\circ}\text{C}}{546} = 1101.1 \, \text{mrad} \, (63.09 \, ^{\circ}) \, \pm 0.7 \, \text{mrad}$$

[
$$\alpha$$
] $\frac{20 \, ^{\circ}\text{C}}{633} = 798.6 \, \text{mrad} \, (45.76 \, ^{\circ}) \, \pm \, 0.4 \, \text{mrad}$

The uncertainties are two standard deviations of the certified values. No sample heterogeneity was observed.

The certified values for specific optical rotation in vacuo are based on a minimum of 12 measurements at each wavelength of 20.000 g of dried SRM 41c, weighed in air, diluted to 100.00 mL at 20.0 °C with "pure" water, and equilibrated overnight. The wavelengths 589, 546, and 633 nm were obtained from a sodium lamp with a line filter, a mercury lamp with a filter pack, and a helium-neon laser, respectively.

The analytical measurements leading to certification were performed in the NIST Organic Analytical Research Division by D.K. Hancock (polarimetry), B. Coxon (muclear magnetic resonance), and S.A. Margolis (Karl Fischer).

The statistical analysis of the data was made by R. Paule, NIST National Measurement Laboratory.

Expiration of Certification: This certification is valid for five years from date of shipment from NIST.

The overall direction and coordination of the technical measurements leading to the certification were under the chairmanship of B. Coxon.

The technical and support aspects concerning 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.

Gaithersburg, MD 20899 May 27, 1993 (Revision of certificate dated 7-23-86) Thomas E. Gills, Acting Chief Standard Reference Materials Program

(over)

Source of Material: This dextrose was obtained from Pfanstiehl Laboratories, Inc., Waukegan, IL.

Drying Instructions: As this material is hygroscopic, all measurements should be made on material that has been dried under vacuum at 60 °C for 24 h.

WARNING: Measurements on SRMs 41b and 41c indicate that aqueous solutions of D-Glucose, which have been spiked with ammonia to achieve rapid-equilibrium, give higher initial specific optical rotation values than those that have not been spiked. Also, the values for spiked solutions decrease with time, eventually falling below the values for non-spiked solutions.

PURITY

The certified purity of SRM 41c is certified based on the levels of α -D-Glucopyranose and β -D-Glucopyranose determined. The α -D-Glucopyranose and β -D-Glucopyranose were measured in freshly prepared solutions of dried D-Glucose in dimethyl sulfoxide-d, by proton magnetic resonance spectroscopy at 400 MHz and by carbon-13 magnetic resonance spectroscopy at 100.6 MHz. The moisture content was measured using the Karl Fischer method; it is not certified, but provided for information only. No organic impurities were revealed by thin-layer chromatography of this material.

Certified Values: For Information Only:

Total D-Glucose 99.9 ± 0.1% Moisture (0.060)%

as:

 α -D-Glucopyranose 96.2 \pm 0.3%

β-D-Glucopyranose 3.7 \pm 0.3%

The uncertainties are two standard deviations of the certified values.

REFERENCE

[1] Cummings, A.L., and Hocken, R.J., An Accurate Temperature-Controlled Polarimeter, Precision Engineering, 4, pp. 33-38, (1982).