National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 920

D-Mannitol

This Standard Reference Material (SRM) is intended primarily for use in the calibration and standardization of procedures for triglyceride determinations in chemical analysis and for the critical evaluation of routine working or secondary reference materials used in these procedures. It is certified as a chemical of known purity. A unit of SRM 920 consists of 50 g of material.

The values of D-mannitol, D-glucitol, and total alditol purities are given below with the associated estimated uncertainty of 0.1 %. Values for loss on drying, ash, and insoluble matter are not certified and are reported below without the estimated uncertainty.

	Weight Percent (%)
D-Mannitol	99.8 ± 0.1
D-Glucitol	0.1 ± 0.1
Total Alditol	99.9 ± 0.1
Loss on drying	<0.02
Ash	<0.001
Insoluble matter	<0.001

Expiration of SRM Certificate: The certification of **SRM 920** is valid, within the measurement uncertainty specified, for five years from date of purchase, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of technical measurements leading to the certification was under the chairmanship of R. Schaffer.

Analyses were performed by NIST in the former NBS Analytical Chemistry Division by R.F. Brady, Jr., B. Coxon, B.A. Johnson, W.H. McCurdy, and W.P. Schmidt.

Technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated by T.W. Mears, of the former NBS Office of Standard Reference Materials.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 February 2016
Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

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

SOURCE, PREPARATION AND ANALYSIS[(1)]

The D-mannitol was obtained from Pfanstiehl Laboratories, Inc. (Waukegan, IL).

The homogeneity of the SRM was monitored by differential scanning calorimetry of 20 samples taken at random from the bulk material, and by polarimetry, gas-liquid chromatography, and thin-layer chromatography. Elemental microanalysis (± 2 SD) showed carbon 39.51 % ± 0.10 % and hydrogen 7.86 % ± 0.12 %; the calculated values are 39.56 % and 7.75 %, respectively. The SRM melted at 167.0 °C to 168.0 °C (corr.) when heated in an open capillary at 0.5 °C•min^{-1}.

The content of D-glucitol in the material was determined by gas-liquid chromatography of hexakis-*o*-(trifluoroacetyl) derivatives on a 10-ft. column of Gas Chrom Q (100 to 120 mesh) coated with 3 % of GE-XE-60 liquid phase, using a flame-ionization detector. The derivatives were prepared by treating 200 mg of the D-mannitol 2 mL of trifluoroacetic anhydride and 10 mg of sodium trifluoroacetate in 2 mL of nitromethane for 2 hours at 25 °C to 30 °C. The solution resulting was diluted to 5.0 mL with nitromethane, and 2-mL aliquots were injected into the gas chromatography (inlet temperature, 135 °C; column 145 °C; detector 150 °C).

The purity of the SRM, as determined by the differential scanning calorimetry, is 99.9 mole percent, and by phase solubility analysis, 99.8 weight percent. For differential scanning calorimetry, the D-mannitol (~0.5 mg) was heated from 380 K to 436 K at 80 K•min^{-1} and then 436 K to 442 K at 0.625 K•min^{-1}. Phase solubility analysis was conducted by equilibrating the material with either ethanol–water (1:1, v/v), or methanol-water (1:1, v/v) at 25 °C for six days.

No impurities were revealed by paper and thin-layer chromatography, infrared spectroscopy (KCl pellet), ultraviolet spectrophotometry (20 % solution of the material in water), or by proton and carbon-13 magnetic resonance spectroscopy (NMR).

The proton NMR spectrum obtained at 90 MHz by accumulating 324 frequency sweeps of a solution of 0.1 g of the material in 0.5 mL of methyl sulfoxide-d_6 showed, as expected for D-mannitol, only a complex multiplet of a doublet at 4.4 ppm and 4.2 ppm, respectively, from tetramethylsilane, due to six hydroxyl protons, a complex multiplet at 3.5 ppm due to eight CH protons.

The proton-decoupled carbon-13 NMR spectrum, accumulated at 22.6 MHz from 1024 scans of a saturated solution of the material in water-hexafluoracetone sesquihydrate (5:1 v/v) with heteronuclear, field-frequency stabilization on fluorine, showed only three singlets, at 122 ppm, 123 ppm, and 129 ppm from external, ^{13}C-enriched carbon disulfide, owing to the two-fold axis of symmetry of D-mannitol.

In the absence of proton decoupling, 5650 scans of a similar solution gave a carbon-13 NMR spectrum that showed only two overlapping doublets ($J13_{C\text{-}H}$ 146 Hz and 141 Hz), each overlapped by a triplet ($J13_{C\text{-}H}$ 142 Hz).

Optical rotations were measured by means of an automatic polarimeter and a high-precision manual polarimeter, by using solutions of the material either in water, or in aqueous 4.7 % ammonium molybdate solution (20.0 mL) diluted to 25.0 mL with 0.5 M sulfuric acid at 20 °C.

$$[\alpha]_{\mathrm{D}}^{20} = 139.0^{\circ} \quad (c\ 1.6 \text{ in acidified 3.8 \% ammonium molybdate soltuion})$$

$$[\alpha]_{\mathrm{D}}^{20} = -0.3^{\circ} \quad (c\ 16 \text{ in water})$$

On being heated at 70 °C under vacuum for 24 h, 7 g samples of the SRM lost 0.001 % to 0.002 % of their weight; however, at 110 °C and 30 torr, constant weight was not reached during 80 h, at which time 50 g samples of the material had decreased in weight by 0.02 %, corresponding mainly to sublimation of the samples.

The residue insoluble in water and the ash content of the SRM were each determined on 25 g samples.

(1) Certain commercial equipment, instruments or materials are identified in this certificate 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.

NOTICE AND WARNING TO USERS

WARNING: This SRM is for research use.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in a well-closed container at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage.

Preparation of Stock Standard Solution: This material is for use as a standard in the determination of triglycerides. Since mannitol solutions require no preliminary handling prior to the automated part of the analysis they are useful in checking automated systems. A stock solution may be prepared by transfer of 182.2 mg of SRM 920 to a 1 L volumetric flask, dissolving it in 0.2 N sulfuric acid and diluting to the mark with 0.2 N sulfuric acid. This solution contains 1 µmol/mL of D-mannitol. Working solutions of 0.050 µmol/mL, 0.100 µmol/mL, and 0.150 µmol/mL may be prepared by dilution of appropriate aliquots with 0.2 N sulfuric acid [1,2]. The stock standard solution (1 µmol/mL) and the working solutions should be stored in glass-stoppered, brown bottle at 4 °C. Under such conditions, these standards should be stable for 6 months. All constituted solutions of D-mannitol should be clear and display no turbidity.

REFERENCES

[1] Kanter, S.L.; *Mannitol as a Primary Standard in the Determinations of Triglycerides*; Clin. Chim. Acta., Vol. 16, pp. 177–178 (1967).

[2] Communication from Lipids Laboratory, Center for Disease Control, Atlanta, GA, January 8, 1974.

Certificate Revision History: 22 February 2016 (Editorial changes); 08 January 2016 (Editorial changes); 23 November 1973 (Revision); 17 January 1972 (Original certificate 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 mailto:srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.