Carbohydrate Research, 53 (1977) 134–135

Note

Preparation of D-mannose from doum-palm kernels

MOHAMMED A. E. SALLAM*
Department of Chemistry, Faculty of Science, Alexandria University, Alexandria (Egypt)
(Received August 20th, 1976, accepted for publication, August 30th, 1976)

D-Mannose is prepared[1–5] by acid hydrolysis of the D-mannan of ivory nut by using basically the procedure developed by Reiss[2]. Like ivory nut, the kernel of the African doum palm (*Hyphaene thebaica*) has been used to manufacture buttons, and contains a D-mannan[6–8].

The present Note describes a procedure for preparing D-mannose by using the doum-palm kernels as an alternative source of this sugar. The kernel shavings were hydrolyzed with sulfuric acid, the hydrolyzate was decolorized with charcoal, made neutral, and evaporated, and the product dissolved in methanol. Isopropyl alcohol was added to precipitate some gum, and the D-mannose was crystallized from acetic acid in 20% overall yield.

EXPERIMENTAL

The shavings of doum-palm kernel (200 g), freed of the brown seed-coat, were added in small portions to stirred, 75% sulfuric acid (140 ml). The suspension was thoroughly mixed, and the temperature was not allowed to rise above 40°. An irritating odor was noticeable during the treatment, and the meal changed color, first to a reddish-brown, and then to purple. The mixture was allowed to stand for 12 h at room temperature with occasional stirring and then diluted with water (1 liter); it was boiled under reflux for 4 h, cooled, and filtered through cheesecloth to remove debris and insoluble matter (8–10 g). The filtrate was boiled under reflux with animal charcoal (25 g) for 1 h, the suspension filtered on a bed of charcoal and the filtrate made neutral with barium hydroxide and barium carbonate. The precipitated barium sulfate was removed by centrifugation, and washed with hot water (1 liter), and the solution and washings were combined, and evaporated to a syrup. This was treated with boiling methanol (250 ml) under reflux, with occasional shaking, until it dissolved, and the solution was diluted with hot isopropyl alcohol (500 ml). Some gummy, amorphous material was precipitated, and the clear solution

*Present address: Department of Medicinal Chemistry and Pharmacognosy, School of Pharmacy, Purdue University, West Lafayette, Indiana 47907, U. S. A.

was separated by decantation. To extract any D-mannose remaining in the gummy residue, this was triturated with methanol (100 ml): the extract was treated with isopropyl alcohol (200 ml), the insoluble matter was allowed to settle, and the clear supernatant liquor was decanted. After two similar extractions, all of the clear, methanol–isopropyl alcohol extracts were combined, and boiled for 30 min under reflux with charcoal (10 g), and the suspension was filtered hot. The filtrate was evaporated to a syrup which was dissolved in hot, glacial acetic acid (60 ml). The solution was cooled, nucleated with a few crystals of D-mannose, and kept for 3 days at room temperature with occasional shaking. Glacial acetic acid (10 ml) was added during the crystallization, and the mixture was then kept for one day in a refrigerator. The crystalline D-mannose was filtered off, washed consecutively with cold 3:1 ethanol–methanol, cold absolute ethanol, and ether, and dried; yield 40 g; m.p. 125–127°.

The crude D-mannose was recrystallized by dissolving it in an equal weight of water, acidifying with a few drops of glacial acetic acid, boiling with charcoal (0.5 g), and filtering. The filtrate was evaporated under diminished pressure to a syrup which was mixed with hot methanol and isopropyl alcohol. The supernatant liquor was decanted from some gummy material, filtered, nucleated with a few crystals of D-mannose, and allowed to crystallize at room temperature with occasional shaking. D-Mannose (30 g) crystallized in needles, m.p. 129–131°, $[\alpha]_D^{20}$ +14.1° (equil., water) {lit.[3] $[\alpha]_D^{20}$ +14.2° (equil., water)}.

ACKNOWLEDGMENT

The author thanks Professor H. S. El Khadem, Michigan Technological University, for his keen interest

REFERENCES

1 H. S. Isbell and H. L. Frush, *Methods Carbohydr. Chem.*, 1 (1962) 145–147.
2 R. Reiss, *Ber.*, 22 (1889) 609–613.
3 F. J. Bates and Associates, *Polarimetry, Saccharimetry and the Sugars*, National Bureau of Standards Circular, 440 (1942) 471–472.
4 C. S. Hudson and H. L. Sawyer, *J. Am. Chem. Soc.* 39 (1917) 470–478.
5 B. N. Stepanenko, N. K. Shcherbukhina, R. A. Baksova, V. D. Shcherbukhin, and K. Dovletmuradov, *Prikl. Biokhim. Mikrobiol.*, 2 (1966) 39–44.
6 B. Maymone, A. Battaglini, and M. Tiberio, *Ann. Sper. Agrar. (Rome)*, 4 (1950) 603–624.
7 H. El Khadem and M. A. E. Sallam, *Carbohydr. Res.*, 4 (1967) 387–391.
8 El Sayed Amin and A. M. Palfologou, *Carbohydr. Res.* 27 (1973) 447–450