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Note

Isolation of *O*-methyl-*scyllo*-inositol from mung bean seeds

YOSHIMITSU UENO, AKIRA HASEGAWA, AND TSUNEKI TSUCHIYA
Department of Agricultural Chemistry, Gifu University Kakamigahara, Gifu 504 (Japan)
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In the previous paper[1], the isolation and identification of the raffinose family of oligosaccharides in mung bean (*Phaselous vidissimus*) seeds was reported. We have now isolated *O*-methyl-*scyllo*-inositol (1), a sweet and nonreductive, crystalline compound so far unreported in Nature.

A methanolic extract of mung bean seeds was fractionated by carbon-column chromatography and **1** was isolated crystalline from the water eluate. Compound **1** did not reduce Fehling solution, was stable on heating with 0.1M hydrochloric acid for 4 h at 110°, and its i.r. spectrum showed strong absorption at 3600-3200 cm^{-1}. It consumed four molecular equivalents of sodium metaperiodate in water solution.

The n.m.r. data[2–4] for the peracetate (**2**) of **1** were: δ 2.1 (s, 6 protons, two equatorial Ac), 2.0 (s, 9 protons, three equatorial Ac), 3.4 (s, 3 protons, OMe), 5.16–5.22 (6 protons on the ring). Hydrolysis of **1** with 57% hydrogen iodide, and acetylation of the product gave a hexaacetate **3**, m.p. 272°, to which the structure hexa-*O*-acetyl-*scyllo*-inositol (**3**) was assigned on the basis of its n.m.r. data: δ 2.0 (s, 18 protons, six equatorial Ac), 5.25 (s, 6 protons on the ring). These spectral data are compatible only with the inositol hexaacetate having the *scyllo* (all-*trans*) configuration. Therefore, the structure of **1** is *O*-methyl-*scyllo*-inositol. The i.r. and n.m.r. spectra, and R_F values by t.l.c., of **2** were all identical with those of an authentic synthetic sample[5].

EXPERIMENTAL

General. — Evaporations were performed under diminished pressure at 40°. Melting points are uncorrected. N.m.r. spectra were recorded at 60 MHz in chloroform-*d* with a Hitachi R-20A spectrometer (Hitachi, Ltd., Japan), and chemical shifts are given on the δ scale. Paper chromatography was performed on Toyo filter paper No. 2 with 6:4:3, *v/v* butyl alcohol–pyridine–water as solvent and (*a*) aniline hydrogen phthalate[6], (*b*) silver nitrate–sodium hydroxide[7] as spray reagents. T.l.c. was performed on 0.25-mm layers of silica Gel G (0.25 mm thick) with 9:1, *v/v* benzene–methanol as developer.

Isolation of **1**. — Defatted powder (500 g) of mung bean seeds was extracted 4 times with hot 80% methanol (3 liters). The extracts from 4.5 kg of mung beans were concentrated to about 2 liters, and a saturated solution of basic lead acetate (200 ml) was added in order to remove protein. The precipitated lead complex was filtered off and washed with water. The combined filtrate and washings were treated with hydrogen sulfide, and then filtered. The filtrate was deionized with Amberlite IR-120 (H^+) and IRA-410 (OH^-) resins and concentrated to a syrup, yield 290 g. This syrup was chromatographed on a 13-cm (diameter) charcoal–Celite column (Takeda Shirasagi charcoal, 3 kg; Celite No. 545, 3 kg), with water as eluant. The eluate was concentrated to a syrup, which gave 4.3 g of **1** from 60% aqueous ethanol. Recrystallization from the same solvent gave needles, m.p. 243°, $[\alpha]_D$ 0° (lit.[5] m.p. 243°).

Decomposition of **1** *with acid*. — Compound **1**, on heating for 4 h at 110° in an autoclave with 0.1M hydrochloric acid, did not decompose. When it was treated with 6M hydrochloric acid for 24 h at 110°, a spot showing a lower R_F value, together with unchanged **1**, was detected on a paper chromatogram.

Acetylation of **1**. — A mixture of **1** (50 mg), anhydrous sodium acetate (25 mg), and acetic anhydride (4 ml) was heated for 1 h at 120°. The reaction mixture was poured into ice–water to afford the crude acetate; yield 70 mg (60%). Recrystallization from ethanol gave **2**, m.p. 192–193° (lit.[5] m.p. 192–193°).

Demethylation[5] *of* **1**. — Compound **1** (100 mg) in 57% aqueous hydrogen iodide (2 ml) was refluxed for 2 h. Evaporation gave a crystalline mass, which was washed with carbon tetrachloride. The residue was acetylated with pyridine (5 ml) and acetic anhydride (5 ml) overnight at room temperature. The product was recrystallized from ethanol to give **3** (160 mg, 63%), m.p. 284–292°.

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