Isolation of β-Sitosterol from Chufa (*Cyperus esculentus* L.) Tubers

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The chemical constitution of the tubers of *Cyperus esculentus* L., also known as chufa, tiger nut, edible cyperus, rushnut, and earth almond, has received sporadic attention through many years. The principal component is the oil, sometimes called sedge oil, which forms about 25% of the tubers and which was valued as a food and for lubrication purposes.[1] The detailed analysis of the oil and other constituents of the tubers cultivated in Egypt will be shortly reported elsewhere.[2]

An early publication by Baughman and Jamieson[3] dealing with the fatty acid constituents of the oil mentioned a phytosterol that was isolated by saponification of the unsaturated acid fraction. These authors reported for the crystalline product m.p. 134–135° and for its acetyl derivative m.p. 122–123°. As it appears that the product has not been examined by later workers, we wish now to report on its identity.

Saponification of the oil (about 25% of the tubers, by light petroleum extraction) with alcoholic potassium hydroxide gave a nonsaponifiable fraction as a bright yellow oil in 0.68% yield. By chromatographic fractionation of this material, there has been obtained β-sitosterol, m.p. 136–137°, $[\alpha]^{28}_{D}$ −34°. It afforded an acetate, m.p. 126–127°, $[\alpha]^{28}_{D}$ −41°, and a benzoate, m.p. 142–4°, $[\alpha]^{28}_{D}$ −15°. The properties of the compound, its acetate, and benzoate agree with those reported by Bernstein and Wallis[4] for β-sitosterol isolated from cottonseed oil; moreover, the melting points of the alcohol and the acetate were undepressed by the authentic samples. The infrared absorption spectrum[5] (Nujol) of the alcohol contained bands at 3400 cm.$^{-1}$ (OH), 840 and 803 (trisubstituted olefin), and was identical with that of authentic material. It is likely, therefore, that β-sitosterol is the material which has been isolated by Baughman and Jamieson.[3]

EXPERIMENTAL[6]

β-Sitosterol. Exhaustive extraction of the finely ground tubers of *Cyperus esculentus* L. with light petroleum (b.p. 70–80°) removed about 25% as oil. Saponification of the oil with ethanolic potassium hydroxide followed by work-up in the usual manner gave 0.68% of a bright yellow thick oil. Percolation of a benzene solution of 0.683 g. of the nonsaponifiable matter through an alumina column and prolonged washing with the same solvent removed 0.368 g. of wax and oily unsaturated hydrocarbon material. Final stripping of the column with 3% methanol in benzene removed 0.268 g. of a colorless solid. Recrystallization from methanol gave β-sitosterol as colorless plates, m.p. and mixed m.p. 136–137°, $[\alpha]^{28}_{D}$ −34° ($CHCl_3$); ϵ_{max} = 3,200(204 mμ); in ethanol; (reported[4] m.p. 136–137°, $[\alpha]^{28}_{D}$ −36°). The infrared absorption spectrum (Nujol) was identical with that of an authentic sample.

β-Sitosteryl acetate was prepared by treating a pyridine solution of β-sitosterol with acetic anhydride at 100° for 1 hr. The product, isolated by the usual work-up, was crystallized from methanol to give colorless needles, m.p. and mixed m.p. 126–127°, $[\alpha]^{28}_{D}$ −41° ($CHCl_3$), ϵ_{max} = 3400 (204 mμ, ethanol); (reported[4] m.p. 125–126°).

β-Sitosteryl benzoate was prepared by heating a mixture of β-sitosterol, pyridine, and benzoyl chloride on the water bath for 1 hr. followed by isolation in the usual manner. Crystallization of the product from methanol gave needles, m.p. 142–144°, $[\alpha]^{28}_{D}$ −15° ($CHCl_3$); (reported[4] m.p. 145°).

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(5) We thank Professor F. G. Baddar, Ein-Shams University, for the infrared measurements.

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The Orientation of the Isopropyl Group of Dihydroabietic γ-Lactone

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Treatment of commercial, partially hydrogenated rosin or dihydroabietic acids with strong mineral acid has led to dihydroabietic α-lactone (the lactone of "hydroxytetrahydroabietic acid"[1c]).[2-7] The con-

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