

(source: Pará), *Vatairea macrocarpa* (Benth.) Ducke (Minas Gerais), *Vatairea paraensis* Ducke (Pará), *Vatairea sericea* Ducke (Pará), *Vataireopsis araroba* (Aguiar) Ducke (Espírito Santo), *Vataireopsis speciosa* Ducke (Amazonas) and *Vataireopsis pallidiflora* Rizz. (Goiás). Small, mostly heartwood, fragments were secured by courtesy of the botanist A. de Mattos. TLC (SiO_2 , C_6H_6 -AcOEt-MeOH 72:25:3) examination of their C_6H_6 extracts failed to reveal the presence of chrysophanol only in the extract from *V. pallidiflora*, prepared from a softwood sample.

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SITOSTEROL- β -D-GALACTOSIDE FROM *HIBISCUS SABDARIFFA**

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Key Word Index—*Hibiscus sabdariffa*; Malvaceae; karkade; sitosterol- β -D-galactoside.

Hibiscus sabdariffa (Malvaceae) or karkade is widely used in Egypt for curing heart and nerve diseases [1,2]. Previously [3] we reported the isolation of a steroidal compound of unknown structure from the leaves of this plant.

The neutral part of the non-volatile fraction of the Et_2O extract of defatted leaves deposited colourless rosettes which were recrystallised ($3 \times \text{CHCl}_3$) to yield sitosterol- β -D-galactoside, m.p. $275\text{--}277^\circ$ (decomp.), $[\alpha]_D -63^\circ$ (in pyridine), ν_{\max} 3450 cm^{-1} (b, OH), NMR (in deuterated pyridine) showed peaks at τ 4.68 (t, one olefinic H), τ at 6.29 (t, H α - to OH). Tetraacetate (needles, MeOH-CHCl_3), m.p. $149\text{--}151^\circ$, $[\alpha]_D -33.3^\circ$; MS; parent ion m/e 744 (agreeing with $\text{C}_{43}\text{H}_{68}\text{O}_{10}$); 331, 211, 229, 169 and 109 (characteristic for glycoside tetraacetates [4]); and at m/e 414 and m/e 396 (base peak), NMR showed sharp peaks at τ 7.9–8.0 (four

acetate groups), τ at 4.72 (t, one olefinic H). Tetra-benzoate (MeOH-CHCl_3), mp $196\text{--}198^\circ$, $[\alpha]_D +10.5^\circ$.

Acid hydrolysis [5] of the glycoside gave sitosterol (mp, mmp 132° , $[\alpha]_D -34^\circ$, IR, M^+ 414) plus galactose (R_f PC 0.12; $\text{BuOH-AcOH-H}_2\text{O}$, 4:1:2.2).

Sitosterol- β -D-galactoside was synthesized [6] from acetobromogalactose pentaacetate [7] and HBr gas in Ac_2O , to obtain a green gum which was dissolved in dry dioxane and added dropwise over 1 hr to a slurry in dioxane of sitosterol, Ag_2CO_3 and MgSO_4 . After 20 hr at room temp., the product was chromatographed over alumina to give sitosterol- β -D-galactoside tetraacetate identical in all respects (IR, mp, mmp and specific rotations) with the natural compound tetraacetate.

It is interesting to note that whereas sitosterol- β -D-glucoside has been isolated from several plant sources, this is the first report of the galactoside.

* Part XIII in a series. For Part XII see Ref. [8].

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ALKALOIDS OF *SIDA CORDIFOLIA**

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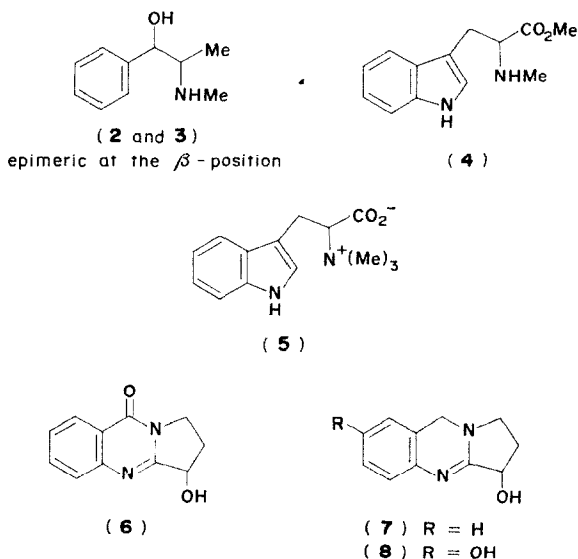
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Key Word Index—*Sida cordifolia*; Malvaceae; β -phenethylamines; carboxylated tryptamines; quinazoline alkaloids; β -phenethylamine; ephedrine; ψ -ephedrine; *S*-(+)-*N*_b-methyltryptophan methyl ester; hypaphorine; vasicinone; vasicine; vasicinol; sympathomimetic amines; bronchodilator principle.

Sida cordifolia L. (Malvaceae) is distributed along with other species of this genus throughout the tropical and sub-tropical plains of India. Extracts of different parts of the plant are reported to be used in the Ayurvedic system of medicine for a variety of purposes [1]. Ghosh and Dutta previously isolated [2] ephedrine and ψ -ephedrine from the aerial parts of this plant, while Dutta reported [3] the presence of appreciable quantities of H₂O-soluble alkaloids but could not isolate them. Reports on nitrogenous constituents of this family of over 700 species are very few. *Gossypium* is the only other genus where the presence of two biogenic amines, viz. 5-hydroxytryptamine [4], and histamine [5] has been reported. No true alkaloids have been previously described in the malvaceae.

From the roots of this plant, we have now isolated three β -phenethylamines, viz. β -phenethylamine (1), ephedrine (2), and ψ -ephedrine (3), 2 carboxylated tryptamines, *S*-(+)-*N*_b-methyltrypto-

phan methyl ester (4) and hypaphorine (5), and three quinazoline alkaloids, vasicinone (6), vasicine (7) and vasicinol (8). In addition, liberal amounts of choline and betaine have been obtained from the H₂O-soluble alkaloid fraction.



* Part I in the projected series "Chemical Constituents of Malvaceae".