

Biological significance. The presence of alkaloids in these spineless monstrose plants imparts a disagreeable bitter taste, as well as known pharmacotoxic effects [9], and quite likely has a protective value to the plants in repelling hungry rodents and other animals.

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IDENTIFICATION AND C-13 N.M.R. SPECTRUM OF STACHYDRINE FROM *CADABA FRUTICOSA*

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Key Word Index—*Cadaba fruticosa*; Capparidaceae; cadabine; stachydrine.

Abstract—Cadabine, a compound isolated from the leaves of *Cadaba fruticosa* has been identified as stachydrine and its C-13 NMR studied.

In 1971, Ahmad and Basha [1] isolated hygroscopic crystalline needles, m.p. 98–100 from the leaves of *Cadaba fruticosa*, a straggling shrub found around Karachi. The compound contained nitrogen and from its ionic properties was presumed to be a betaine. It did not show any absorptions in the UV region and did not respond to mass spectrometry because of its low volatility. The IR spectrum afforded peaks at 1625 cm^{-1} (COO^-) and 3450 cm^{-1} ($-\text{OH}$).

NMR spectroscopy of a solution of the picrate in CF_3COOD afforded peaks at 4.56 δ (m, 1H), 3.78 δ (m, 2H), 3.37 δ (s, 3H), 3.54 δ (s, 3H) and

broad multiplets in the region 2.3–3.08 (4H). The multiplet at 3.78 δ collapsed to a singlet on spin-spin decoupling when irradiated at 2.5 δ . The above signals were assigned to the C-2, C-5, N- CH_3 , N- CH_3 , and C-3 and C-4 protons respectively. This spectrum was similar to that reported [2] for stachydrine (1) in D_2O . Since no C-13 NMR spectra of any betaines have previously been reported, such a spectrum was recorded at 25.2 MHz in D_2O using dioxan as internal standard. The fully decoupled spectrum afforded six sharp singlets at 75.3, 68.2, 52.7, 46.8, 24.8 and 19.2 ppm relative to TMS, which were assigned to C-2, C-5,

N-CH₃, N-Me, C-3 and C-4 carbons respectively.

The structure (1) was confirmed by comparison with a synthetic specimen (m.p. NMR, IR).

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ISOLATION OF ERYTHRODIOL MONOPALMITATE FROM *TAGETES* cv. SEN. DIRKSEN

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Key Word Index—*Tagetes* cv. Sen. Dirksen; Compositae; erythrodiol monopalmitate; 3- β -palmitoxy-olea-12en-28-ol; palmitic acid; sterols.

Plant. *Tagetes* cv. Sen. Dirksen. *Source.* The plant material was collected in the United States by Dr. Robert E. Perdue, Jr. [1]. A voucher specimen, identified by Dr. Perdue, has been deposited at the Department of Pharmacognosy and Pharmacology, University of Illinois at the Medical Center (specimen CA-2033). *Uses.* None, but other species of this genus have shown nematocidal [2,3] and antitumor [4] activities.

Previous work. None on this species. From *T. minuta*: thiophenes [5], terpenes [6,7], flavonoids [4, 8-11], carotenoids [12-14]; *T. erecta*: carotenoids [13], sterols [17], thiophenes [18].

Plant part examined. Whole plant. *Isolation and Identification.* The powdered plant material (5 kg) was defatted with light petroleum, followed by extraction with MeOH and concentration *in vacuo* to yield 617 g of residue, which was partitioned between CHCl₃ and H₂O. The CHCl₃ solubles (66.18 g) were chromatographed over a 2 kg column of silica gel PF₂₅₄ and eluted with CHCl₃-MeOH (9:1). Fractions 11-33 (50 ml each) were combined, taken to dryness (12.96 g), and rechromatographed over a second column of silica gel PF₂₅₄ (600 g). This column was developed with benzene and work-up of fractions 76-102 (20 ml each) yielded 71.1 mg of a crystalline material from acetone. The compound was identified as erythrodiol monopalmitate by the following physical

methods. It gave a positive Liebermann-Burchard test for triterpenes; m.p. 112-113°; $[\alpha]_D^{27.5} + 57.5^\circ$ (conc 0.1 in CHCl₃). The compound gave the following R_f values on silica gel G TLC-CHCl₃ (0.37), CHCl₃-MeOH (9:1) (0.86). IR spectrum (KBr) at ν_{\max} : 3475 (s) (OH), 2915 (s), 2840 (s), 1700 (s) (ester C=O), 1465 (s), 1380 (m), 1360 (m), 1260 (m), 1240 (m), 1140 (w), 1070 (w), 1045 (w), 1010 (w), 820 (w), 810 (w), 800 (w) (trisubstituted double bond) and 720 cm⁻¹ $[-(\text{CH}_2)_n-]$, $n > 4$. A PMR spectrum in CDCl₃, (TMS), showed signals at 5.2 δ (1H, broad, C₁₂-H), 4.5 δ (1H, m, C_{3 α} -H), 3.4 δ (2H, dd, C₂₈-H₂). The MS showed a molecular ion at m/e 680 (1.7%), followed by ions at m/e 662 (3.5%), 425 (4.8%), 393 (5.2%), 256 (2.2%), 234 (39.1%), 203 (100%) and 189 (12.6%). All of these data were in agreement with those reported for erythrodiol monopalmitate isolated from *Madhuca butyracea* (Sapotaceae) [19]. *Saponification* with boiling 5% alcoholic KOH (3 ml) for 3 hr gave erythrodiol, m.p. 210-214°; $[\alpha]_D^{27.5} + 95^\circ$ (conc 0.1 in CHCl₃) (MS and PMR spectra and MS of its acetate [20]) and palmitic acid (GLC of its methyl ester 5% OV-101 on Gas Chrom Q, 80-100 mesh at 260°). The isolation of this compound from *Tagetes* cv. Sen. Dirksen represents the first report of its occurrence in the Compositae.

The isolate from fractions 162-165 from the second column was shown by GLC (5% OV-101