

N-CH<sub>3</sub>, 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- $\beta$ -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 CHCl<sub>3</sub> and H<sub>2</sub>O. The CHCl<sub>3</sub> solubles (66.18 g) were chromatographed over a 2 kg column of silica gel PF<sub>254</sub> and eluted with CHCl<sub>3</sub>-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 PF<sub>254</sub> (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 CHCl<sub>3</sub>). The compound gave the following  $R_f$  values on silica gel G TLC-CHCl<sub>3</sub> (0.37), CHCl<sub>3</sub>-MeOH (9:1) (0.86). IR spectrum (KBr) at  $\nu_{\max}$ : 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<sup>-1</sup>  $[-(\text{CH}_2)_n-]$ ,  $n > 4$ ). A PMR spectrum in CDCl<sub>3</sub>, (TMS), showed signals at 5.2  $\delta$  (1H, broad, C<sub>12</sub>-H), 4.5  $\delta$  (1H, m, C<sub>3 $\alpha$</sub> -H), 3.4  $\delta$  (2H, dd, C<sub>28</sub>-H<sub>2</sub>). 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 CHCl<sub>3</sub>) (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

on Gas Chrom Q, 80–100 mesh, isothermal at 260°) to be a mixture of stigmasterol (78.5%) and sitosterol (21.5%).

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