

NMR and MS). Characterization of other two compounds is based on the data given below.

Z-3-(2,4,5-Trimethoxyphenyl)-2-propenal (4). Rigorous CC of C_6H_6 fractions on crystallization from Me_2CO gave fine, colourless needles, mp 85° . They gave a positive test with 2,4-DNP. IR ν_{max}^{KBr} cm^{-1} : 2830, 1650, 1495, 1460, 1270, 1205, 1155, 870, 820, 740, 660. 1H NMR ($CDCl_3$): δ 3.87, 3.90, 4.00 (9H, sss, 3 \times OMe), 6.5, 7.3 (2H, ss, H-3 and H-6), 6.50 (1H, m, H-2 α), 6.80 (1H, d, J = 6.0 Hz, H-3 β), 10.70 (1H, s, CHO). MS m/z (rel. int.): 222.0 [M]⁺ (50.47), 195 [M - CO + H]⁺ (100), 191.0 (68.30), 152 (27.73), 137 (27.60), 122 (12.62). Found: C, 64.97; H, 6.28. $C_{12}H_{14}O_4$ requires: C, 64.86; H, 6.30.

2,3-Dihydro-4,5,7-trimethoxy-1-ethyl-2-methyl-3-(2,4,5-trimethoxyphenyl)indene (5). Fractions, eluted with 5% MeOH in $CHCl_3$, gave an amorphous powder which could not be crystallized, mp 84° . IR ν_{max}^{KBr} cm^{-1} : 1605, 1575, 1505, 1480, 1390, 1335, 1330, 1200, 1170, 1070, 1035, 975, 880, 800, 770. 1H NMR ($CDCl_3$): 0.87 (3H, t, J = 6.5 Hz, H-9), 1.20 (3H, d, J = 6.5 Hz, H-10), 1.63 (2H, m, H-8), 2.07 (1H, m, H-1), 2.73 (1H, m, H-2), 3.40 (3H, s, OMe), 3.70 (3H, s, OMe), 3.84 (12H, s, 4 \times OMe), 4.37

(1H, d, J = 5.0 Hz, H-3), 6.41 (2H, d, J = 3.0 Hz, H-3' and H-6'), 6.57 (1H, s, H-6). MS m/z (rel. int.): 416.2214 [M]⁺ (100), 387.1724 (3.8), 385.2059 (6.1), 356.0760 (3.0), 341.1260 (2.6), 247.1284 (2.8), 233.1044 (2.7), 220.0921 (13.6), 219.0781 (90.1), 217.0377 (2.8). Found: C, 69.40; H, 7.79. $C_{24}H_{32}O_6$ requires C, 69.23; H, 7.70.

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SURANGIN C, A COUMARIN FROM *MAMMEA LONGIFOLIA*

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Key Word Index—*Mammea longifolia*; Guttiferae; bark; 4-alkylated coumarins; surangin B; surangin C.

Abstract—The isolation and characterization of a new 4-alkylated coumarin is described.

INTRODUCTION

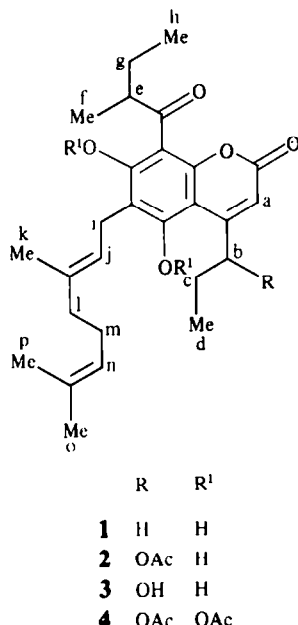
The roots of *Mammea longifolia* (Wight) Planch and Triana have been shown [1] to contain the coumarins surangin A (1) and B (2) and taraxerol. In the course of our search for the presence of new pesticides in Indian plants we have found that the bark of *M. longifolia* contains surangin B and its deacetyl analogue, which we have named surangin C (3). Unlike the roots, the bark contained neither surangin A nor taraxerol.

RESULTS AND DISCUSSION

Surangin C (3), $C_{27}H_{36}O_6$, gave a green colouration with methanolic ferric chloride. The presence of three

hydroxyl functions was established by the formation of a triacetate derivative. Its IR and UV properties [ν_{CHCl_3} cm^{-1} : 3500–3100 (br), 1720, 1610, 1595, 1380 and 1200; UV λ_{max}^{MeOH} nm (log ϵ): 227 (4.01), 256 (4.75) and 333 (4.51)] showed it to be a coumarin closely related to surangins A and B [1]. Its 1H NMR spectrum was very similar to that of surangin B with the exception that it lacked an acetate singlet at δ 2.2 and a one proton multiplet at δ 6.5. However, an additional one proton multiplet was present at δ 4.68 which suggested that surangin C possibly contained a hydroxyl function at methylene b instead of the acetoxyl present in surangin B. This was confirmed from the 1H NMR spectrum of surangin C triacetate in which the multiplet at δ 4.68 was replaced by multiplet of the same magnitude at δ 6.25. In order to establish the total structure, decoupling experiments at several sites in the 1H NMR spectrum of

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surangin C were conducted and this gave patterns compatible with the assigned structure (3).

The final proof of the structure was obtained when surangin B was converted into its triacetate derivative and its spectroscopic data compared with those of the triacetate prepared from surangin C. Both acetates showed identical features (IR, ^1H NMR, TLC) and this, therefore, unambiguously confirmed the structure 3 for surangin C.

EXPERIMENTAL

Mammea longifolia bark was collected from Sawantwadi in June, 1984 and a voucher specimen has been deposited in our own herbarium.

Extraction and isolation. Air dried powdered plant material

(530 g) was stirred at room temp. for 24 hr with petrol (3 l; 60–80°). Filtration followed by removal of petrol gave an oil (18.8 g), an aliquot (8 g) of which was fractionated by CC on silica gel using petrol, petrol containing increasing amounts of CHCl_3 and CHCl_3 . The fractions collected were monitored by TLC (petrol– CHCl_3 , 2:3) and identical fractions were pooled together. Further purification of these fractions by prep. TLC gave the following compounds.

Surangin B (2), colourless crystals (from *n*-hexane– CH_2Cl_2) (0.8 g), mp 98–99° (lit. mp 98–100°) [1]. Its IR and ^1H NMR spectral data were identical with those reported.

Surangin C (3) was obtained as a colourless oil (5.2 g), $[\alpha]_D^{25} + 16.6^\circ$ (CHCl_3 ; c 1%), $[\alpha]_D^{25} - 12.0^\circ$ (MeOH , c 1%) R_f 0.13 using CHCl_3 –petrol (3:2); ^1H NMR (200 MHz, $\text{CDCl}_3 + \text{D}_2\text{O}$) δ 6.1 (1H, s, H-a), 5.23 (1H, t, H-j, $J_{ij} = 7$ Hz), 5.1 (1H, t, H-n, $J_{m,n} = 7$ Hz), 4.68 (1H, m, H-b, $J_{b,c} = 7$ Hz), 3.7 (1H, m, H-e, $J_{e,f} = 7$ Hz), 3.43 (2H, d, H-i), 2.04 (4H, m, H-l–H-m), 1.8 (3H, s, Me-k), 1.8–1.2 (4H, m, H-c–H-g), 1.61 and 1.58 (6H, $2 \times s$, Me-o–Me-p), 1.21 (3H, d, Me-f, $J_{e,f} = 7$ Hz), 1.02 and 0.94 (6H, $2 \times t$, Me-d–Me-h); MS m/z : 456 $[M]^+$.

Surangin C triacetate (4). To a soln of surangin C (50 mg) in Ac_2O (3 ml) was added $\text{C}_5\text{H}_5\text{N}$ (3 drops) and the soln kept at room temp. for 24 hr. Usual work up gave 4 as an oil (48 mg) which failed to crystallize. R_f 0.4 using CHCl_3 –petrol (3:2); IR $\nu_{\text{CHCl}_3}^{\text{max}}$, cm^{-1} : 1783, 1740, 1705, 1615, 1593, 1670, 1370, 1190, 1165, 1098 and 910; ^1H NMR (80 MHz, CDCl_3): δ 6.55 (1H, s, H-a), 6.25 (1H, m, H-b, $J_{b,c} = 7$ Hz), 4.97 (1H, t, H-j, $J_{ij} = 7$ Hz), 4.87 (1H, m, H-n), 3.13 (3H, m, H-e–H-i), 2.42, 2.23 and 2.18 (9H, $3 \times s$, Ac), 2.0 (4H, m, H-l and H-m), 1.8–1.2 (4H, m, H-c and H-g), 1.67 (6H, br s, Me), 1.58 (3H, s, Me), 1.15 (3H, d, Me-f, $J_{e,f} = 7$ Hz), and 0.98 (6H, $2 \times t$, Me-o–Me-p); MS m/z : 582 $[M]^+$.

In a similar manner surangin B was acetylated and the product was identical with the triacetate derived from surangin C.

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