Chemical Investigation of Ceylonese Plants. Part 21.† Extractives of *Pentadesma butyracea* Sabine (Guttiferae)

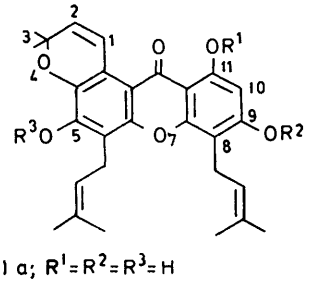
By **Sarath P. Gunasekera, Kanapathipillai Sivapalan,** and **M. Uvais S. Sultanbawa,*** Department of Chemistry, University of Sri Lanka, Peradeniya Campus, Peradeniya, Sri Lanka
W. David Ollis, Department of Chemistry, University of Sheffield, Sheffield S3 7HF

From the bark and timber extracts of *Pentadesma butyracea* Sabine the following compounds have been isolated: β-amyrin acetate, β-amyrin, β-sitosterol, 1,3,5-trihydroxy-2-methoxyxanthone, jacareubin, osajaxanthone, and a new xanthone, pentadesmaxanthone, identified as 5,9,11-trihydroxy-3,3-dimethyl-6,8-bis-(3-methylbut-2-enyl)-3*H*,12*H*-pyrano[3,2-*a*]xanthen-12-one.

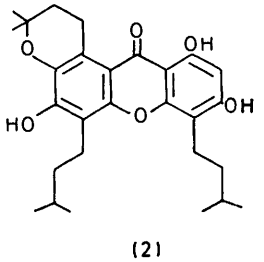
We report the results of studies on the extractives of the bark and timber of *Pentadesma butyracea* Sabine, of the sub-family Moronobeoideae (Guttiferae).[1-5]

Bark Extractives.—From the light petroleum extract of the bark, sodium carbonate- and sodium hydroxide-soluble fractions were first removed, and the neutral fraction was separated on a silica gel column to give β-amyrin acetate, β-amyrin, and β-sitosterol. Separation of the sodium hydroxide-soluble fraction in a similar manner gave two pigments, 1,3,5-trihydroxy-2-methoxyxanthone[1] (3) and a new pigment named pentadesmaxanthone (1a), $C_{28}H_{30}O_6$.

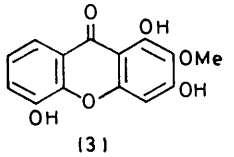
The u.v. spectral pattern (Table 1) and characteristic absorption at 1 655 cm^{-1} in the i.r. spectrum of pentadesmaxanthone indicated that it was a xanthone. The u.v. spectra of hydrogenated pentadesmaxanthone (2) (see Table 1) closely resembled that of hydrogenated tovophyllin[6] (6) and other 1,3,6,7-tetraoxygenated



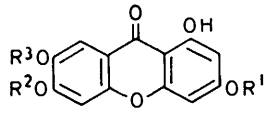
(1) a; $R^1=R^2=R^3=H$
b; $R^1=R^2=R^3=Ac$
c; $R^1=H, R^2=R^3=Me$



(2)

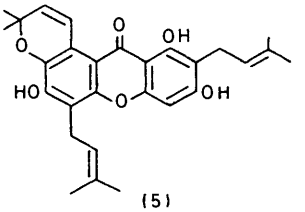


(3)

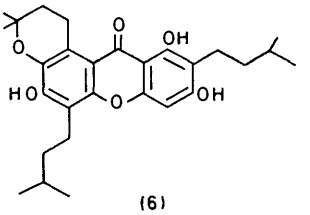


(4) a; $R^1=R^3=H, R^2=Me$
b; $R^1=R^2=R^3=Me$

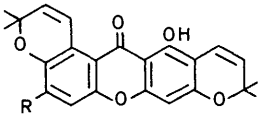
xanthones such as 1,3,7-trihydroxy-6-methoxyxanthone[6] (4a), 1-hydroxy-3,6,7-trimethoxyxanthone[7] (4b), and the 1,3,6,7-tetraoxygenated xanthone[6] (7a). Therefore it was concluded that pentadesmaxanthone has a 1,3,6,7-tetraoxygenated system.



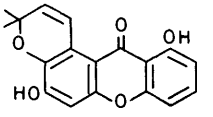
(5)



(6)



(7) a; R = OMe
b; R = H



(8)

The formation of a triacetate (1b) indicated the presence of three hydroxy-groups and the formation of only a dimethyl ether (1c) with an excess of diazomethane indicated that one of these was chelated. The presence of a chelated hydroxy-group was supported by a bathochromic shift in the u.v. on addition of aluminium chloride,[8] and a low field n.m.r. signal at τ −3.2 to −3.3.

The n.m.r. data of pentadesmaxanthone (1a) and its hydrogenation product (2), triacetate (1b), and dimethyl ether (1c) are given in Table 2.

The presence of a 2,2-dimethyl-2*H*-pyran ring attached to the xanthone ring in an angular position was clearly shown by the low τ value for one of the 2*H*-pyran AB doublets, with *J* 10 Hz, due to deshielding by the xanthone carbonyl group, in pentadesmaxanthone and its triacetate and dimethyl ether. Such data have been used in establishing the structures of tovoxanthone[9] (8), tovophyllin[6] (5), and thwaitesixanthone[2] (7b). The n.m.r. data also indicate the presence of two 3,3-dimethylallyl groups,[10] *e.g.* in pentadesmaxanthone (1a) with characteristic signals[1] for two olefinic methyl groups at τ 8.22 (6 H), $ArCH_2$ at τ 6.4 (2 H), and vinylic

† Part 20, ref. 19.

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H at τ 4.7 (1 H). Further, hydrogenation of pentadesmaxanthone gave a hexahydro-compound (2) in the n.m.r. spectrum of which the 2*H*-pyran doublet and 3,3-dimethylallyl signals had disappeared and were replaced by signals corresponding to the appropriate saturated groups (see Table 2).

m/e **447**, $(M - CH_3)^+$, and a further peak at *m/e* 216, $(M - 2CH_3)^{2+}$, as in the case of thwaitesixanthone [2] (7b). The ion $(M - CH_3)^+$ also provided the base peak in the spectra of the triacetate and the dimethyl ether, indicating the stability of the pyrylium ion.

Timber Extractives.—The neutral fraction of the light petroleum extract of the timber gave β-amyrin and β-sitosterol by separation on a silica gel column. Similar separation of acidic fraction gave jacareubin [14] (9a). A chloroform extract of the methanol extract gave four pigments of which jacareubin [14] (9a), osajaxanthone [15] (9b), and 1,3,5-trihydroxy-2-methoxyxanthone [1] (3) were identified by comparison with authentic samples.

TABLE 1
U.v. data

	$\lambda_{max.}$/nm (log ε)						Solvent
Pentadesmaxanthone (1a)	249 (4.42)	261 (4.51)	279sh (4.25)	323 (4.23)	340sh (4.10)	370 (3.89)	*a*
Hexahydropentadesmaxanthone (2)	243 (4.39)	261 (4.52)	278sh (4.13)	321 (4.32)		368 (3.98)	*a*
Hexahydrotovophyllin (6)	245 (4.47)	263 (4.53)	280sh (5.57)	325 (4.30)		380 (3.48)	
1,3,7-Trihydroxy-6-methoxyxanthone (4a)	239 (4.34)	256 (4.50)		310 (4.36)		362 (3.99)	*b*
1-Hydroxy-3,6,7-trimethoxyxanthone (4b)	238 (4.37)	256 (4.59)		309 (4.23)		356 (4.09)	*b*
Tetrahydro-5-methoxythwaitesixanthone (7a)	242.5 (4.42)	261.5 (4.59)		317 (4.36)		366 (3.99)	

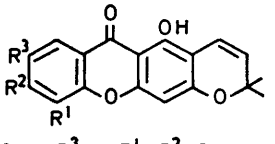
[a] Absolute EtOH. [b] 95% EtOH.

TABLE 2
N.m.r. data [τ values; solvent $(CD_3)_2CO$]

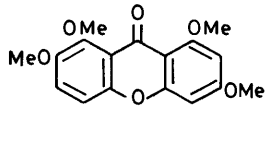
Compound	(1a) (100 MHz)	(2) (60 MHz)	(1b) (60 MHz)	(1a) (60 MHz)
11-OH	−3.2 to −3.3			
1-H	2.00 (1 H, d, *J* 10 Hz)	6.67 (2 H, t, *J* 7 Hz)	2.10 (1 H, d, *J* 10 Hz)	2.00 (1 H, d, *J* 10 Hz)
10-H	3.74 (1 H, s)	3.82 (1 H, s)	3.13 (1 H, s)	3.62 (1 H, s)
2-H	4.16 (1 H, d, *J* 10 Hz)	8.22 (2 H, t, *J* 7 Hz)	4.13 (1 H, d, *J* 10 Hz)	4.14 (1 H, d, *J* 10 Hz)
−CH=C	4.7 (2 H, t, *J* 7.4 Hz)		4.8 (2 H, m)	4.8 (2 H, m)
$ArCH_2$	6.4 (4 H, m)	7.22 (4 H, q, *J* 6.5 Hz)	6.42 (4 H, d, *J* 7.5 Hz)	6.4 (4 H, m)
$:CMe_2$	8.22 (6 H, s) 8.36 (6 H, s)		8.20 (6 H, s) 8.32 (6 H, s)	8.19 (6 H, s) 8.30 (6 H, s)
$-(^-O)CMe_2$	8.56 (3 H, s) 8.66 (3 H, s)	8.67 (6 H, s)	8.60 (3 H, s) 8.70 (3 H, s)	8.30 (3 H, s) 8.49 (3 H, s)
5-, 9-, and 11-OAc			7.65 (9 H s)	
5- and 9-OMe				6.04 (3 H, s) 6.07 (3 H, s)
>CH–CH_2		8.32—8.85 (6 H, m)		
Me_2C		9.06 (12 H, d, *J* 4.8 Hz)		

Only one aromatic proton resonates as a singlet (at τ 3.74) in pentadesmaxanthone, as in the case of tovophyllin [6] (5) (τ 3.57). The high τ value indicates that this proton is in the phloroglucinol ring of the xanthone nucleus. The positions available in this ring are C-2 and -4 (basic xanthone ring numbers). In general in such oxygenated systems the C-2 proton resonates at τ 3.5—3.7 and the C-4 proton at τ 3.2—3.5.[11,12] Hence in pentadesmaxanthone the high τ value (3.74) and a negative Gibbs test [13] enable the assignment of this proton to C-2. This was supported by the chemical shift of the corresponding proton in pentadesmaxanthone triacetate (Table 2). The above assignment leaves xanthone positions 4 and 5 for the 3,3-dimethylallyl groups. Hence pentadesmaxanthone is 5,9,11-trihydroxy-3,3-dimethyl-6,8-bis-(3-methylbut-2-enyl)-3*H*,12*H*-pyrano[3,2-*a*]xanthen-12-one (1a).

In the mass spectrum of pentadesmaxanthone the molecular ion was observed at *m/e* 462 with base peak at



(9) a; R^3 = H, R^1 = R^2 = OH
b; R^1 = R^2 = H, R^3 = OH



(10)

The fourth pigment had a u.v. spectrum which indicated the presence of 1,3,7,8-tetraoxygenated system.[16,17] It showed a bathochromic shift in ethanol with aluminium chloride,[8] indicating the presence of a chelated hydroxy-group. A sodium acetate-induced

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shift [16] in the u.v. indicated the presence of a free hydroxy-group in position 3. The tetramethyl ether, prepared with dimethyl sulphate, was identical with authentic 1,3,7,8-tetramethoxyxanthone [17] (10), confirming the oxygenation pattern. Further studies on this hydroxy-compound were not possible owing to lack of material.

The first triprenylated xanthones to be reported from the Guttiferae were tovophyllin A and B from *Tovomita macrophylla* Pl. et Tr.[6] Pentadesmaxanthone (1a) differs from tovophyllin (5) only in the position of one of the isoprenyl groups; this is the second report of a triprenylated xanthone from the Guttiferae species.

Although Scheinmann *et al.* postulated that jacareubin could be a taxonomic marker for the genus *Calophyllum* owing to its presence in almost all species investigated, it has now been observed also in some species of the genera *Kielmeyera*,[18] *Pentadesma*, and *Mesua*.[19]

EXPERIMENTAL

U.v. and i.r. spectral data were obtained with Unicam SP 8000B and Perkin-Elmer 257 spectrophotometers, respectively. Optical rotations were determined with a Bellingham and Stanley polarimeter. Microanalytical data were obtained from the CSIRO Microanalytical Service, Melbourne. M.p.s were obtained with a Kofler hot-stage apparatus. Merck (30—70 mesh) silica gel was used for column chromatography. Light petroleum used was generally the fraction of b.p. 60—80 °C.

Pentadesma butyracea Sabine was obtained from Peradeniya Botanical Gardens, Sri Lanka, and processed as reported in earlier parts for other species.

Extractives of the Bark.—Isolation of β-amyrin acetate, β-amyrin, and β-sitosterol. The cold light petroleum extract of the bark (7.5 kg) on concentration gave a yellow solid (A) (150 g, 2%). The solid (A) (50 g) was dissolved in ether (1 l) and washed with ice-cold 2% sodium hydroxide. The ether layer after washing with water, drying, and evaporation gave a neutral fraction (B) (35.2 g). The sodium carbonate- and sodium hydroxide-soluble fractions were acidified with dilute hydrochloric acid and extracted with ether. The ethereal extracts on processing as above gave a phenolic fraction (5.32 g) as a yellowish brown semisolid (C), and an acidic fraction (5.22 g) as a yellow gum. The neutral fraction (B) (5 g) was chromatographed on silica gel (125 g). Elution with benzene–light petroleum (1 : 3) gave β-amyrin acetate (0.046 g), needles, m.p. 237—238° (from light petroleum), $[\alpha]_D^{27}$ +78.3° ($CHCl_3$) {lit.,[20] m.p. 238—240°, $[\alpha]_D$ +79.8° ($CHCl_3$)}, identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c. comparison). Elution with benzene–light petroleum (1 : 1) gave β-amyrin (3.12 g) needles, m.p. 196° [from benzene–light petroleum (1 : 3)] $[\alpha]_D^{27}$ +85.0° ($CHCl_3$) [lit.,[20] m.p. 200°, $[\alpha]_D$ +86.1°], identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c. comparison). Elution with chloroform–benzene (1 : 1) mixture gave β-sitosterol (0.210 g), m.p. 136—137°, identified by comparison with an authentic sample.

The acidic fraction showed a streak on t.l.c. and was not further investigated.

Isolation of Pentadesmaxanthone (1a).—The phenolic fraction (C) (5.0 g) was chromatographed on silica gel (125 g). Elution with chloroform–benzene (1 : 1) gave *pentadesmaxanthone* (1a) (0.250 g), yellow needles, m.p. 262—263° (from $CHCl_3$), R_F 0.72 ($CHCl_3$); green colouration with $FeCl_3$; negative Gibbs test (Found: C, 72.4; H, 6.6%; M^+, 462. $C_{28}H_{30}O_6$ requires C, 72.7; H, 6.55%; *M*, 462); $\lambda_{max.}$ (EtOH–$AlCl_3$) 239 (log ε 4.45), 272 (4.51), 289sh (4.25), 349 (4.35), 370 (4.11), and 420nm (3.92); $\lambda_{max.}$ (EtOH–NaOAc) 249 (log ε 4.45), 261 (4.51), 279sh (4.25), 324 (4.18), 340sh (4.10), and 370 nm (4.06); $\nu_{max.}$ (KBr) 3 480, 3 350, 3 000, 2 940, 2 880, 1 655, 1 625, 1 580, 1 505, 1 445, 1 380, 1 300, 1 270, 1 215, 1 180, 1 135, 1 100, 1 085, 1 035, 975, 935, 900, 880, 845, 810, 790, 780, 735, 700, 685, and 630 cm^{-1}; n.m.r. data in Table 2; *m/e* 462(45%), 447(100), 435(11), 433(5), 421(55), 409(35), 394(45), 391(50), 379(20), 375(23), 365(55), 353(45), 337(25), 326(30), 309(40), 297(23), 281(11), 269(11), 216(15), 203(11), 196(11), 188(20), 176(11), and 168(13).

Pentadesmaxanthone Triacetate (1b).—Pentadesmaxanthone (1a) (0.030 g) was heated with acetic anhydride (1 ml) and pyridine (1 ml) for 16 h; work-up gave the *triacetate* (1b) (0.018 g), white needles, m.p. 183—185° [from benzene–chloroform (1 : 1)], R_F 0.50 (benzene), M^+ 588; $\lambda_{max.}$ (EtOH) 242 (log ε 4.27), 263 (4.31), 292 (4.00), 306 (3.91), 374 (3.83), and 395 nm (3.79); $\nu_{max.}$ ($CHCl_3$) 1 760 (OAc) and 1 642 cm^{-1} (xanthone CO); n.m.r. data in Table 2; *m/e* 588(60%), 573(100), 548(30), 547(19), 546(31), 531(68), 505(17), 489(26), 463(10), 461(12), 446(9), 406(11), 404(8), 391(14), and 365(7).

Pentadesmaxanthone Dimethyl Ether (1c).—Pentadesmaxanthone (1a) (0.020 g) in ether (20 ml) was treated with diazomethane in ether and gave a mixture of two products. The mixture was separated on a plate of silica gel with chloroform. The major product on crystallisation from light petroleum gave yellow needles of the *dimethyl ether* (1c) (0.013 5 g), m.p. 189—190°, R_F 0.72 [chloroform–benzene (1 : 1)] (Found: M^+, 490.234 6. $C_{30}H_{34}O_6$ requires *M*, 490.235 5); $\lambda_{max.}$ (EtOH) 247 (log ε 4.30), 267 (4.41), 321 (4.11), 343 (3.97), and 385 nm (3.72); $\lambda_{max.}$ (EtOH–$AlCl_3$) 238 (log ε 4.35), 270 (4.42), 325 (4.16), 343 (4.07) and 395 nm (3.84); $\nu_{max.}$ (KBr) 1 645 cm^{-1} (xanthone CO); n.m.r. data in Table 2; *m/e* 490(32%), 488(40), 475(100), 449(20), 437(8), 469(6), 407(7), 390(6), 381(6), 367(8), 351(6), 294(6), 217(5), 119(95), 117(98), 91(40), 83(50), 55(40), and 44(86).

Hexahydropentadesmaxanthone (2).—Pentadesmaxanthone (1a) (0.030 g) in absolute ethanol (30 ml) was hydrogenated over Adams catalyst [21] at atmospheric pressure and room temperature. Filtration and removal of solvent gave *hexahydropentadesmaxanthone* (2) as light yellow plates (0.020 g), m.p. 273—275° (from ethanol), R_F 0.72 ($CHCl_3$), M^+ 468; $\lambda_{max.}$ (EtOH) in Table 1; $\lambda_{max.}$ (EtOH–$AlCl_3$) 239 (log ε 4.51), 272 (4.53), 290sh (4.31), 343 (4.45), and 415 nm (4.03); $\lambda_{max.}$ (EtOH–NaOAc) 241 (log ε 4.55), 261 (4.60), 278 (4.38), 323.5 (4.40), and 372 nm (4.09); $\nu_{max.}$ (KBr) 3 460 (OH) and 1 650 cm^{-1} (xanthone CO); *m/e* 468(100%), 453(10), 439(16), 426(40), 425(98), 413(35), 412(37), 411(98), 397(10), 369(14), 356(59), 355(40), 339(12), 336(9), 324(8), 311(15), 300(25), 299(50), 271(14), 163(8), 84(6), and 82(9).

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Isolation of 1,3,5-*Trihydroxy-2-methoxyxanthone* (3).—Further elution of the above column with chloroform yielded a yellow semisolid (0.200 g). This was separated on a plate of silica gel with chloroform–acetone (5 : 1) into two yellow solids. The less polar, minor constituent was identified as pentadesmaxanthone (1a) (0.020 g). The major component on crystallisation from ethyl acetate gave yellow plates of 1,3,5-trihydroxy-2-methoxyxanthone [1] (3) (0.041 g), m.p. 278° (lit.,[2] 280°), identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c. comparison).

Extractives of Timber.—The cold light petroleum extract of the timber (9.75 kg) on concentration gave a yellowish brown semisolid (D) (20.8 g). The solid (D) (5 g) in ether was washed with ice-cold 2% sodium carbonate and 2% sodium hydroxide solutions. The ether layer after washing, drying, and evaporation gave a neutral fraction (E) as a white semisolid (2.82 g). The sodium hydroxide- and sodium carbonate-soluble fractions were acidified with dilute hydrochloric acid and extracted with ether. On work-up as usual the phenolic fraction gave a yellow pigment (F) and the acidic fraction a yellow semisolid (G) (1.05 g).

Isolation of Jacareubin.—The yellow pigment (F) on crystallisation from ethyl acetate gave jacareubin [14] (0.050 g), m.p. 254—256° (lit.,[14] 254—256°), identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c. comparison).

Isolation of β-*Amyrin and Sitosterol.*—The semi-solid (E) (2 g) was chromatographed on silica gel. Elution with benzene gave β-amyrin (1.202 g), white flakes, m.p. 195—197°. Elution with benzene–chloroform (1 : 3) gave β-sitosterol, m.p. 136—137°.

Methanol Extract.—Isolation of osajaxanthone [15] (9b) *and jacareubin* (9a). The powdered timber after extraction with cold light petroleum and hot benzene was extracted with hot methanol. Evaporation gave a black tarry solid (H) (175 g). The solid (H) was extracted with hot chloroform (Soxhlet). Concentration gave osajaxanthone (9b) (0.200 g), m.p. 256—258° (from chloroform) (lit.,[15] m.p. 264—265°), identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c.). The mother liquor on evaporation gave a yellowish brown semisolid (J) (4.25 g). The solid (J) (4 g) was chromatographed on silica gel. Elution with chloroform gave jacareubin [14] (0.030 g), m.p. 253—254°, and osajaxanthone (0.086 g), m.p. 256—258°.

Isolation of a tetraoxygenated xanthone. Further elution of the above column with $CHCl_3$ gave a yellow semisolid (0.130 g). This was separated on a plate of silica gel with chloroform–methanol (99 : 1) to give osajaxanthone (10 mg) and a tetraoxygenated xanthone (0.060 g), m.p. 226—228° (from chloroform), R_F 0.45 [$CHCl_3$–MeOH (99 : 1)], $\lambda_{max.}$ (EtOH) 237sh (log ε 4.47), 240 (4.49), 263 (4.43), 314 (4.35), and 372nm (3.89); $\lambda_{max.}$ (EtOH—$AlCl_3$), 230 (log ε 4.54), 248 (4.38), 268 (4.43), 275 (4.39), 320 (4.27), 351 (4.05), and 372nm (3.92); $\lambda_{max.}$ (EtOH–NaOAc) 240 (log ε 4.37), 268 (4.29), 317 (3.98), and 372 nm (3.89).

The tetraoxygenated xanthone (0.010 g) was refluxed with dimethyl sulphate (1 ml), calcined potassium carbonate (1 g), and dry acetone (3 ml) for 5 h; the usual work-up gave 1,3,7,8-tetramethoxyxanthone (10) (0.006 g), white crystals, m.p. 162—163° (from light petroleum) (lit.,[17] 165°), identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c. comparison).

Isolation of 1,3,5-*trihydroxy-2-methoxyxanthone* (3).—Further elution with chloroform–methanol (99 : 1) gave 1,3,5-trihydroxy-2-methoxyxanthone (3) (0.020 g), m.p. 278°, identical with an authentic sample (mixed m.p., i.r. spectra, and t.l.c.).

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