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PRENYLATED XANTHONES FROM *CUDRANIA COCHINCHINENSIS*

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Key Word Index—*Cudrania cochinchinensis*; Moraceae; prenylated xanthone, ^{13}C NMR; gerontoxanthone.

Abstract—In the course of our studies on the bark of *Cudrania cochinchinensis*, we isolated four new prenylated xanthones, named gerontoxanthones E, G, H and I, along with the known xanthone, cudraniaxanthone. The structures of new xanthones were established by spectroscopic and chemical means.

INTRODUCTION

In a previous paper [1], we described the isolation of four new prenylated xanthones, gerontoxanthones A–D, from *Cudrania cochinchinensis* var. *gerontogea*. As a part of our continuing studies on this plant [1, 2], we now wish to report the isolation and structure elucidation of four new prenylated xanthones.

RESULTS AND DISCUSSION

Methanolic extraction of the fresh bark of *C. cochinchinensis* var. *gerontogea*, followed by solvent partition and CC led to the isolation of four new prenylated xanthones, gerontoxanthones E, G, H and I, together with the known xanthone, cudraniaxanthone, which was identified by comparison of its spectral data with those reported [3].

Gerontoxanthone G (**1**) was assigned the molecular formula $C_{23}H_{24}O_6$ (*m/z* 396.1592). Its UV spectrum was indicative of a 1,3,5,6-tetraoxygenated xanthone chromophore [1, 4]. Compound **1** gave a triacetate (**1a**) on acetylation, suggesting the presence of three hydroxyl groups, in which the one at C-1 was chelated (δ13.54 in ^{1}H NMR). The ^{1}H NMR spectrum of **1** showed the presence of a 2,3-dihydro-2,3,3-trimethylfuran ring (δ4.53, *q*, J=6.8 Hz, methine; δ1.39, *d*, J=6.8 Hz, *sec*-methyl; 1.49, 1.24, *gem*-dimethyl) and a 3-methylbut-2-enyl group (δ5.40, *m*, olefinic proton; δ3.44, *d*, J=7.3 Hz, benzylic methylene; δ1.76, 1.75, two methyls). Furthermore, the singlet aromatic proton signals at δ7.52 and 6.29 were assigned to H-8 and H-4 (or H-2), respectively. Cyclization of **1** with 2,2-dichloro-5,6-dicyanobenzoquinone (DDQ) [5] gave a product which was identified as gerontoxanthone A (**1b**) by direct comparison with an authentic sample [1]. This indicated that the dihydrofuran ring of **1** was closed at C-2 and the 3-methylbut-2-enyl side chain was located at C-7. From the above evidence, the structure of **1** was concluded to be 4′,5′-dihydro-1,5,6-trihydroxy-7-(3-methylbut-2-enyl)-4′,4′,5′-trimethylfurano-(2′,3′:3,2)xanthone.

Gerontoxanthone E (**2**) was assigned the molecular formula $C_{24}H_{26}O_6$ (*m/z* 410.174). Its UV and ^{1}H NMR spectra were very similar to those of **1**. The ^{1}H NMR spectrum indicated the presence of a 3-methylbut-2-enyl group (δ5.40, *m*, olefinic proton; δ3.42, *d*, J=7.3 Hz, benzylic methylene; δ1.77, 1.75, two methyls) and a 2,3-dihydro-2,3,3-trimethylfuran ring (δ4.55, *q*, J=6.1 Hz, methine; δ1.40, *d*, J=6.1 Hz, *sec*-methyl; δ1.50, 1.25, *gem*-dimethyl). In addition, the spectrum contained signals at δ13.46 (chelated 1-OH), 9.28 (5-OH or 6-OH), 7.70 (H-8), 6.43 (H-4) and 4.01 (OMe). In the UV spectrum, the λ_{max} at 313 nm was shifted bathochromically to 364 nm on addition of sodium acetate, indicating that the free hydroxyl group was located at C-6 and the methoxy group had to be placed at C-5. The above evidence led us to conclude that the structure of **2** was 4′,5′-dihydro-1,6-dihydroxy-5-methoxy-7-(3-methylbut-2-enyl)-4′,4′,5′-trimethylfurano (2′,3′:3,2)-xanthone. The ^{13}C NMR spectrum also supported the proposed structure (Table 1).

Gerontoxanthone H (**3**) was assigned the molecular formula $C_{23}H_{24}O_5$ (*m/z* 380.1651). Its UV spectrum was indicative of a 1,3,7-trioxygenated xanthone chromophore [5, 6]. Acetylation of **3** gave a triacetate (**3a**). As one of the three free hydroxyl groups was a chelated hydroxyl group (^{1}H NMR, δ13.35) at C-1, the others were concluded to be located at C-3 and C-7. The ^{1}H NMR spectrum showed the presence of two prenyl groups (δ5.30, 2H, *m*; δ4.19, 2H, *d*, J=6.8 Hz; δ3.50, 2H, *d*, J=7.4 Hz; δ1.88, 1.84, 2× Me; δ1.66, 1.65, 2× Me) and three aromatic protons, two of which were *ortho*-coupled to each other (δ7.41, *d*, J=9.3 Hz, H-6; δ7.34, *d*, J=9.3 Hz, H-5) and the other was a singlet (δ6.32, H-2 or H-4). The low-field benzylic proton signals (δ4.19) suggested that one prenyl group was located at C-8 [5]. Therefore, the possible structure of this compound was either **3** or **4**. The spectral features of gerontoxanthone H, however, differed from the reported data of 6-deoxy-γ-mangostin (**4**) [5]. In the UV spectrum, the λ_{max} was immediately shifted on addition of aluminium trichloride, suggesting the presence of an isolated H-2 proton. Furthermore, the signal at δ98.6 (*d*) in the ^{13}C NMR spectrum was assignable to C-2 rather than to C-4, indicating that another prenyl group was located at C-4.

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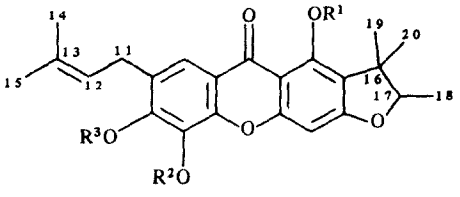
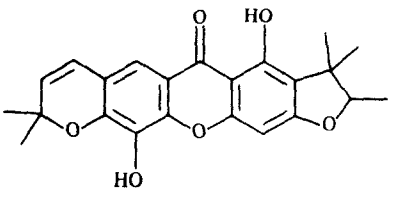
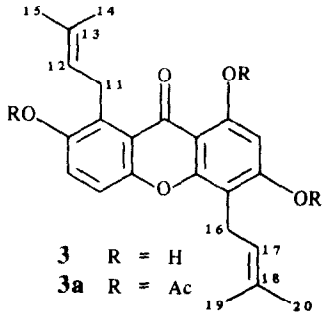


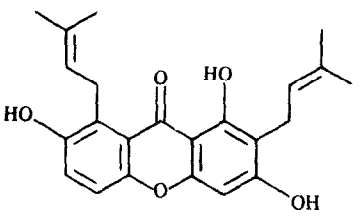
1 $R^1 = R^2 = R^3 = H$
1a $R^1 = R^2 = R^3 = Ac$
2 $R^1 = R^3 = H$, $R^2 = Me$



1b



3 R = H
3a R = Ac



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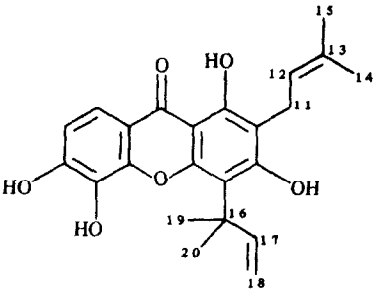

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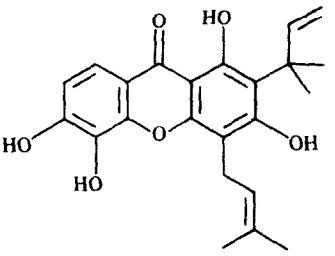

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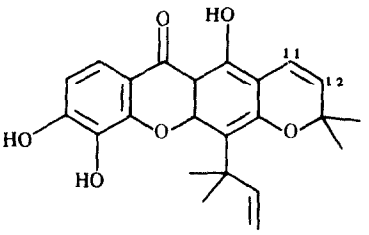

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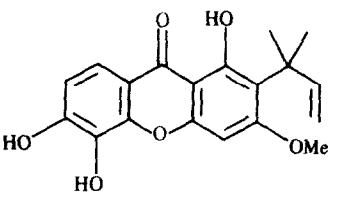

Cudraniaxantone

These findings led us to conclude the structure of **3** to be 1,3,7-trihydroxy-4,8-di(3-methylbut-2-enyl)xanthone.

Gerontoxanthone I (**5**) was assigned the molecular formula $C_{23}H_{24}O_6$ (*m/z* 396.1564). Its UV spectrum showed a characteristic chromophore of a 1,3,5,6-tetraoxygenated xanthone with four free hydroxy groups [4]. One of the free hydroxyl groups was chelated (^{1}H NMR, δ13.86, 1-OH) and two were *ortho*-dihydroxy group (the UV maximum was shifted with $NaOAc/H_3BO_4$ and $AlCl_3/HCl$). The ^{1}H NMR spectrum showed the pressure of two *ortho*-coupled aromatic protons (δ7.63, *d*, $J = 8.8$ Hz, H-8, δ7.01, *d*, $J = 8.8$ Hz, H-7) on ring B, and 1,1-dimethylprop-2-enyl (δ6.60, 1H, *dd*, $J = 17.7$ and 10.4 Hz; δ5.47, *d*, $J = 17.7$ Hz; δ5.35, *d*, $J = 10.4$ Hz; δ1.81, *gem*-dimethyl) and 3-methylbut-2-enyl (δ5.22, 1H, *m*; δ3.37, 2H, *d*, $J = 7.0$ Hz; δ1.78, 1.66, two methyls) side chains. Both side chains had to be located on ring A, two structures (**5** and **6**) being possible. Cyclization of **5** with DDQ gave **7**. The ^{1}H NMR (in acetone-d_6) of **7** showed new signals at δ6.77 (*d*, $J = 9.8$ Hz), 5.62 (*d*, $J = 9.8$ Hz) and 1.52 (*gem*-dimethyl), indicating the presence of a 2H-pyran ring. The 1,1-dimethylprop-2-enyl side chain remained intact during the reaction with DDQ. In the ^{1}H NMR spectrum (in pyridine) of **7**, the solvent-induced shifts, +0.23 for H-11 and −0.01 for H-12, indicated that the 2H-pyran ring was linear [7] Compound **7** was identified as macluraxanthone by comparison of the spectral data with those reported [7,8]. Therefore, the two side chains, 3-methylbut-2-enyl and 1,1-dimethylprop-2-enyl, were located at C-2 and C-4,

Table 1. ^{13}C NMR spectral data for gerontoxanthones G(**1**), E(**2**), H(**3**) and I(**5**)

C	1	2	3	5
1	159.0 *s*	158.6 *s*	163.1 *s*	160.4 *s*
2	114.2 *s*	114.1 *s*	98.6 *d*	112.5 *s*
3	166 8 *s*	166.7 *s*	163 7 *s*	161 9 *s*
4	90.5 *d*	90.5 *d*	106.7 *s*	111.9 *s*
4a	160.1 *s*	159.3 *s*	155.8 *s*	155.4 *s*
4b	146.1 *s*	150 0 *s*	152 8 *s*	147.5 *s*
5	133.9 *s*	134.2 *s*	117.3 *d*	134 2 *s*
6	151 1 *s*	155.3 *s*	124 9 *d*	152 1 *s*
7	127 3 *s*	127 7 *s*	152.7 *s*	113 9 *d*
8	116 8 *d*	120 7 *d*	129.5 *s*	117.7 *d*
8a	117.8 *s*	117.9 *s*	119 8 *s*	115 2 *s*
9	182 0 *s*	181.3 *s*	184.9 *s*	182 3 *s*
9a	104.6 *s*	104.0 *s*	104.8 *s*	104.1 *s*
11	28 8 *t*	26.2 *t*	26.7 *t*	22.8 *t*
12	123.3 *d*	122.6 *d*	124.3 *d*	123.8 *d*
13	132.7 *s*	129 4 *s*	132 0 *s*	132 5 *s*
14	18.3 *q*	18.2 *q*	18.8 *q*	18.4 *q*
15	26 4 *q*	29.0 *q*	26.5 *q*	26.3 *q*
16	44.4 *s*	44.3 *s*	22.5 *t*	42.7 *s*
17	92.1 *d*	92.1 *d*	123.9 *d*	151.8 *d*
18	15.0 *q*	14 9 *q*	131 8 *s*	112 8 *t*
19	21.4 *q*	21.1 *q*	18.5 *q*	29.2 *q*
20	26 0 *q*	25.8 *q*	26.3 *q*	29.2 *q*
5-OMe		62.3 *q*		

Measured at 22.5 MHz in acetone-d_6

respectively. On the basis of the above evidence, the structure of **5** was concluded to be 4-(1,1-dimethylprop-2-enyl)-1,3,5,6-tetrahydroxy-2-(3-methylbut-2-enyl)-xanthone.

EXPERIMENTAL

Mps uncorr; ^1H and ^{13}C NMR: 270 and 22.5 MHz, respectively; MS: 70 eV.

Plant material Fresh root bark of *C. cochinchinensis* var. *gerontogea* was collected at Chai-I, Taiwan. The plant was identified by Muh-Tsuen Kao (National Taiwan University).

Extraction and separation. The fresh root bark of *C. cochinchinensis* var. *gerontogea* (1.5 kg) was chopped and extracted with boiling MeOH (10 l × 4). The MeOH extract (101.8 g) was suspended in H_2O (500 ml) and extracted successively with C_6H_6 (2 l), $CHCl_3$ (2 l), EtOAc (3.5 l) and BuOH (1 5 l) to give the respective extracts in yields of 42.1, 11.0, 13.2 and 12.4 g. A portion of the C_6H_6 extract (*ca* 20 g) was chromatographed on a silica gel column (5 5 × 80 cm) The column was eluted successively with C_6H_6, C_6H_6–EtOAc (8:1, 6.1, 4·1 and 1:1), EtOAc and $CHCl_3$–MeOH to give fractions A–H, which were further subjected to repeated CC on silica gel, eluting with a gradient of C_6H_6 and EtOAc, followed by polyamide CC and prep. TLC. These procedures led to the isolation of the following compounds. gerontoxanthones E (5 mg, **2**) and F (8 mg) from fraction C; gerontoxanthone A (29 mg) from fraction D; gerontoxanthones B (16 mg) and G (65 mg, **1**) and sterols (53 mg) from fraction E; gerontoxanthones C (15 mg), H (25 mg, **3**) and I (35 mg, **5**) from fraction F; gerontoxanthone D (21 mg) and cudraniaxanthone (115 mg) from fraction H.

Gerontoxanthone G (**1**). Yellowish fine needles (MeOH), mp 203–205°, $[\alpha]_D^{25°}$ 0 (Me_2CO, *c* 0.11), HRMS *m/z*: 396.1592, Calcd for $[M]^+$, $C_{23}H_{24}O_6$: 396.1573, TLC: R_f 0.21 [C_6H_6–EtOAc (6:1)], solvent B), orange yellow under UV light, red with Flavone T and greenish brown with $FeCl_3$; UV λ_{max}^{MeOH} nm (log ε): 254 (4.23), 285 (3.63), 328 (3.88), + $AlCl_3$ (after 10 min): 244, 269, 292 (sh), 388; + $AlCl_3$ + HCl: 243 (sh), 257, 264 (sh), 284 (sh), 353; + NaOAc: 270 (sh), 285 (sh), 355; IR ν_{max}^{KBr} cm^{-1}. 3620, 3450, (OH), 1660 (conj C=O), 1618, 1595 EIMS *m/z* (rel int.). 396 $[M]^+$ (26), 381 $[M-Me]^+$ (100), 325 (23); ^1H NMR (270 MHz, Me_2CO-d_6): δ13 54 (1H, *s*, ex. D_2O, 1-OH), 9.08 (1H, *s*, ex. D_2O, OH), 8.74 (1H, *s*, ex. D_2O, OH), 7 52 (1H, *s*, H-8), 6.29 (1H, *s*, H-4), 5 40 (1H, *m*, H-12), 4.53 (1H, *q*, J=6.8 Hz, H-17), 3.44 (2H, *d*, J=7.3 Hz, H-11), 1.76, 1.75 (6H, each *s*, 13-Me_2), 1 39 (3H, *d*, J =6.8 Hz, 17-Me),. 1.49, 1 24 (6H, each *s*, 16-Me_2); ^1H NMR (270 MHz, pyridine-d_5): δ14.2 (1H, *s*, 1-OH), 8.05 (1H, *s*, H-8), 6 09 (1H, *s*, H-4), 5 65 (1H, *m*, H-12), 4.42 (1H, *q*, J=6.1 Hz, H-17), 3.77 (2H, *d*, J=7.3 Hz, H-11), 1.75, 1.69 (6H, each *s*, 13-Me_2), 1.49, 1 24 (6H, each *s*, 16-Me_2), 1.27 (3H, *d*, J=6.1 Hz, 17-Me).

Gerontoxanthone-G triacetate (**1a**). Colourless needles, (MeOH), mp 115–116°; EIMS *m/z* (rel. int.): 522 $[M]^+$ (13), 480 $[M-Ac+H]^+$ (100), 465 $[M-Ac-Me+H]^+$ (52), 438 $[M-2Ac+2H]^+$ (72), 423 $[M-2Ac-Me+2H]^+$ (100), 396 $[M-3Ac+3H]^+$ (24), 381 $[M-3Ac-Me+3H]^+$ (95); ^1H NMR (270 MHz, $CDCl_3$): δ7.98 (1H, *s*, H-8), 6 65 (1H, *s*, H-4), 5.23 (1H, *m*, H-12), 4.52 (1H, *q*, J=6 6 Hz, H-17), 3.30 (2H, *d*, J=7.3 Hz, H-11), 2.51, 2.42, 2.35 (9H, each *s*, 1,5,6-OAc), 1.75, 1.70 (6H, each *s*, 13-Me_2), 1.57 (6H, *s*, 16-Me_2), 1.40 (3H, *d*, J=6.6 Hz, 17-Me)

Cyclization of gerontoxanthone G. Compound **1** (20 mg) was treated with DDQ (20 mg) in dry C_6H_6 (20 ml) and the mixture

was refluxed for 1 hr The product was purified by silica gel CC and prep TLC (C_6H_6–EtOAc 8·1) to give compound **1b** mp 235–237°, EIMS *m/z* 399 $[M]^+$, ^1H NMR (Me_2CO-d_6): δ13 46 (1H, *s*, 1-OH), 8 63 (1H, *s*, OH), 7.44 (1H, *s*, H-8), 6 59 (1H, *d*, J =9.8 Hz, H-11), 6 38 (1H, *s*, H-4), 5 91 (1H, *d*, J=9 8 Hz, H-12), 4.55 (1H, *q*, J=6 4 Hz, H-17), 1.51 (9H, *s*, 13-Me_2, 16-Me), 1 40 (3H, *d*, J=6 4 Hz, 17-Me), 1 24 (3H, *s*, 16-Me)

Gerontoxanthone E (**2**). Pale yellow needles (MeOH); mp 136–138°, $[\alpha]_D^{25}$ 0 (Me_2CO, *c* 0 04); HRMS *m/z* 410 1739, Calcd for $[M]^+$, C_{24} $H_{26}O_8$ 410 1729, TLC R_f 0 69 (solvent B), orange yellow under UV light, red with Flavone T and greenish brown with $FeCl_3$, UV λ_{max}^{MeOH} nm (log ε) 254 (4 7), 280 (sh) (3 85), 313 (4 5); $+AlCl_3$ (after 10 min) 244, 285 (sh), 343, 390 (sh), + NaOAc. 239, 280 (sh), 364, IR ν_{max}^{KBr} cm^{-1} 1650 (conj. C=O), EIMS *m/z* (rel int) 410 $[M]^+$ (26), 395 $[M-Me]^+$ (100), 339 (11), ^1H NMR (270 MHz, Me_2CO-d_6) δ13 46 (1H, *s*, ex D_2O, 1-OH), 9 28 (1H, *s*, ex D_2O, 6-OH), 7 70 (1H, *s*, H-8), 6 43 (1H, *s*, H-4), 5.40 (1H, *m*, H-12), 4 55 (1H, *q*, J=6 1 Hz, H-17), 4 01 (3H, *s*, 5-OMe), 3 42 (2H, *d*, J=7.3 Hz, H-11), 1 77, 1 75 (6H, each *s*, 13-Me_2), 1 40 (3H, *d*, J=6 1 Hz, 17-Me), 1 50, 1 25 (6H each *s*, 16-Me_2).

Gerontoxanthone H (**3**) Yellow needles (MeOH), mp 175–177° HRMS *m/z* 380 1651, Calcd for $[M]^+$, $C_{23}H_{24}O_5$ 380.1644, TLC R_f 0 26 (solvent B), brown colour under UV light, red with Flavone T and dark green with $FeCl_3$. UV λ_{max}^{MeOH} nm (log ε) 238 (4.53), 264 (4.77), 317 (4.32), 382 (3 90); $+AlCl_3$ 232, 280, 335, 440; + NaOAc. 245, 278, 342, 415, IR ν_{max}^{KBr} cm^{-1} 3560, 3460 (OH), 1640 (conj C=O), 1602, 1580, EIMS *m/z* (rel int.) 380 $[M]^+$ (62), 365 $[M-Me]^+$ (13), 337 $[M-C_3H_7]^+$ (100), 281 (40), ^1H NMR (270 MHz, Me_2CO-d_6) δ13 35 (1H, *s*, ex D_2O, 1-OH), 9 77 (1H, *br s*, ex D_2O, OH), 8 72 (1H, *br s*, ex D_2O, OH), 7 41 (1H, *d*, J=9 3 Hz, H-6), 7 34 (1H, *d*, J=9 3 Hz, H-5), 6 32 (1H, *s*, H-2), 5.30 (2H, *m*, H-12, 17), 4 19 (2H, *d*, J =6 8 Hz, H_2-11), 3 50 (2H, *d*, J=7 4 Hz, H_2-16), 1 88, 1 84 (6H, each *s*, 13-Me_2), 1.66, 1 65 (6H, each *s*, 18-Me_2)

Gerontoxanthone-H triacetate (**3a**) EIMS *m/z* (rel. int) 506 $[M]^+$ (46), 464 $[M-Ac+H]^+$ (63), 421 $[M-2Ac+H]^+$ (100), 379 $[M-3Ac+2H]^+$ (78), 337 (25), 323 (31), 281 (31); ^1H NMR (270 MHz, $CDCl_3$): δ7.344, 7.341 (2H, each *s*, H-5, 6), 6 79 (1H, *s*, H-2), 5 13 (2H, *m*, H-12, 17), 3 99 (2H, *d*, J=6.4 Hz, H_2-11), 3 52 (2H, *d*, J=7.3 Hz, H_2-16), 2 44 (3H, *s*, 1-OAc), 2 362, 2 356 (6H, each *s*, 3,5-OAc), 1 86, 1 82 (6H, each *s*, 13-Me_2), 1 69, 1 67 (6H, each *s*, 18-Me_2)

Gerontoxanthone I (**5**). Yellow needles (MeOH), mp 178–180°, HRMS *m/z* 396 1564, Calcd for $[M]^+$, $C_{23}H_{24}O_6$ 396 1572, TLC R_f 0.13 (solvent B), orange red colour under UV light, red colour with Flavone T and greenish brown with $FeCl_3$ UV ν_{max}^{MeOH} nm (log ε) 255 (4 4), 285 (3.76), 329 (4 13), $+AlCl_3$ (after 10 min): 246, 276, 294, 327, 394, $+AlCl_3+HCl$: 254, 272 (sh), 288 (sh), 347, 392 (sh); + NaOAc: 257, 290 (sh), 367, + NaOAc + H_3BO_3 261, 288, 349, IR ν_{max}^{KBr} cm^{-1}· 1625 (conj C=O), EIMS *m/z* (rel int) 396 $[M]^+$ (92), 381 $[M-Me]^+$ (25), 353 $[M-Me-CO]^+$ (50), 340 $[M-C_4H_8]^+$ (90), 325 $[M-C_5H_{11}]^+$ (100), 297 (25), 285 (32), 153 (6), ^1H NMR (270 MHz, Me_2CO-d_6) δ13.86 (1H, *s*, ex. D_2O, 1-OH), 9 72 (1H, *s*, ex D_2O, OH), 7 97 (1H, *s*, ex D_2O, OH), 7 76 (1H, *s*, ex D_2O, OH), 7.63 (1H, *d*, J=8.8 Hz, H-8), 7 01 (1H, *d*, J=8 8 Hz, H-7), 6.60 (1H, *dd*, J=17 7 and 10 4 Hz, H-17), 5 47 (1H, *d*, J=17.7 Hz, Ha-18), 5.35 (1H, *d*, J=10.4 Hz, Hb-18), 5 22 (1H, *m*, H-12), 3 37 (2H, *d*, J =7.0 Hz, H_2-11), 1 81 (6H, *s*, 16-Me_2), 1 78, 1 66 (6H, each *s*, 13-Me_2)

Cyclization of gerontoxanthone I Compound **5** (25 mg) was refluxed with DDQ (25 mg) in dry C_6H_6 (25 ml) for 1 hr. The products were filtered, and the filtrate subjected to silica gel CC with C_6H_6–EtOAc (5 2), followed by polyamide CC with 50% EtOH to yield **7** EIMS *m/z* 394, ^1H NMR (270 MHz, $CDCl_3$): δ13 53 (1H, *s*, 1-OH), 7 69 (1H, *d*, J=8 8 Hz, H-8), 6.96 (1H, *d*, J =8 8 Hz, H-7), 6 77 (1H, *d*, J=9 8 Hz, H-11), 6 74 (1H, *dd*, J =17 6 and 9 3 Hz, H-17), 5 62 (1H, *d*, J=9 8 Hz, H-12), 5 22 (1H, *d*, J=17 6 Hz, Ha-18), 5 05 (1H, *d*, J=9 3 Hz, Hb-18), 1 65 (6H, *s*, 16-Me_2), 1 52 (6H, *s*, 13-Me_2) $\Delta\delta=\delta$ (pyridine)$-\delta(CDCl_3)$ +0 27 (H-8), +0 27 (H-7), +0 23 (H-11), −0 01 (H-12). The compound was identical with macluraxanthone [8]

Cudraniaxanthone Yellowish needles (MeOH), $C_{19}H_{18}O_6$; mp 300°, TLC R_f 0 38 on a polyamide plate (60% EtOH, solvent D) The spectroscopic data (^1H NMR, ^{13}C NMR etc) agreed with those reported [3]

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