

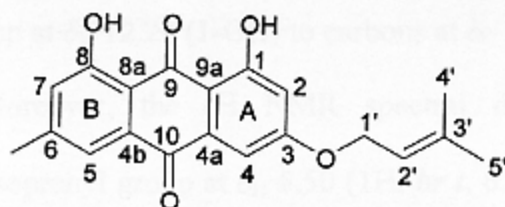
3. RESULTS AND DISCUSSION

3.1 *Structural elucidation of the isolated compounds from the barks of C. formosum ssp. pruniflorum*

The ground dried barks of *C. formosum ssp. pruniflorum* (4.0 kg) were extracted with CH_2Cl_2 and acetone successively at room temperature (each 2×20 L, for 5 days). The crude extracts were evaporated under reduced pressure to afford brownish crude CH_2Cl_2 (76.0 g) and acetone (21.0 g) extracts. The crude CH_2Cl_2 extract was subjected to chromatography and/or recrystallization to yield a new anthraquinone: CPH4 and a new bianthrone: CPH11, together with four known anthraquinones: CPH1-3 and CPH4, two known vismones: CPH8 and CPH9, a known bianthrone: CPH10, four known xanthones: CPH12-15 and a known triterpenoid: CPH18. The crude acetone extract was subjected to chromatography and/or recrystallization to give two known anthraquinones: CPH6 and CPH7, two known xanthones: CPH16-17, two known triterpenoids: CPH19-20 and a mixture of stereroids: CPH21 and CPH22.

Their structures were determined using 1D and 2D NMR spectroscopic data except for the structures of CPH3, CPH13-15 and CPH20 which were determined by single crystal X-ray structure determination.

3.1.1 Compound CPH1



Compound **CPH1** was isolated as a red-orange solid. The UV-Vis spectrum exhibited the absorption bands at 221, 253, 265, 286 and 480 nm characteristic of a conjugated quinone system, which was confirmed by the presence of FT-IR absorption maxima indicating the presence of hydroxyl (3338 cm^{-1}) and chelated carbonyl (1628 cm^{-1}) groups.

The ^1H NMR spectral data of **CPH1** (Table 2) showed two chelated hydroxyl groups at δ 12.28 and 12.11, which were assigned to carbons at C-1 and C-8 from HMBC experiment (Table 2). The appearance of two broad singlet aromatic protons at δ_{H} 7.63 and 7.08 were attributed to *meta* splitting of H-5 and H-7 and long range coupling with an aromatic methyl protons at δ_{H} 2.46 (3H, *s*, Me-6). The COSY cross-peaks were show between H-5/H-7 and Me-6 (Table 1). The lowest-field aromatic proton at δ_{H} 7.63 was assigned to H-5 due to the deshielding region of carbonyl functionality. The position of aromatic proton at C-7 was assigned by HMBC correlations (Figure 2) of the chelated hydroxyl group at δ_{H} 12.11 (8-OH) to the carbons at δ_{C} 113.7 (C-8a), 124.4 (C-7) and 162.5 (C-8) and aromatic methyl protons at δ_{H} 2.46 (6-Me) to the carbons at δ_{C} 121.2 (C-5), 124.4 (C-7) and 148.3 (C-6). The ^1H NMR spectra also showed two signals of *meta*-coupled aromatic protons at δ_{H} 7.38 (1H, *d*, 2.4 Hz) and 6.69 (1H, *d*, 2.4 Hz), and the lowest-field aromatic proton was

assigned to H-4 due to the anisotropic effect from a carbonyl group. An aromatic proton at δ_{H} 6.69 was assigned to H-2 by HMBC correlations (Figure 2) from chelated hydroxyl group at δ_{H} 12.28 (1-OH) to carbons at δ_{C} 107.5 (C-2), 110.1 (C-9a) and 165.1 (C-1). Moreover, the ^1H NMR spectral data (Table 2) showed characteristic of oxy-isoprenyl group at δ_{H} 5.50 (1H, *br t*, 6.9 Hz, H-2'), 4.66 (2H, *d*, 6.9 Hz, H-1'), 1.84 (3H, *s*, H-5') and 1.81 (3H, *s*, H-4'). The position of an oxy-isoprenyl group at C-3 was assigned by HMBC correlations (Figure 2) of an aromatic proton at δ_{H} 7.38 to carbons at δ_{C} 107.5 (C-2), 110.1 (C-9a), 165.9 (C-3) and 182.0 (C-10) and oxy-methylene protons at δ_{H} 4.66 to carbons at δ_{C} 18.3 (C-4'), 118.2 (C-2'), 139.7 (C-3') and 165.9 (C-3), respectively. The complete HMBC correlations were summarized in Table 2. Therefore, compound CPH1 was assigned as madagascin (Ritchie and Taylor, 1964).

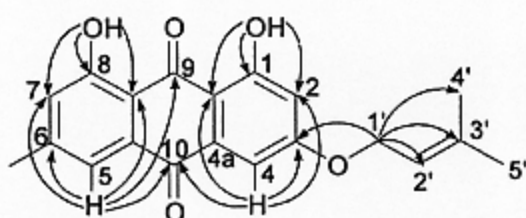
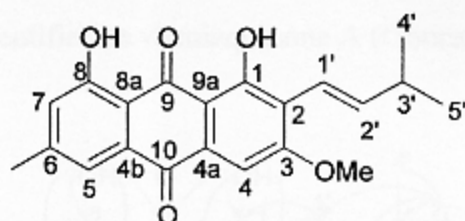


Figure 2 Selected HMBC correlations of CPH1

Table 2 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH1

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)	COSY ($^1\text{H} \rightarrow ^1\text{H}$)
1-OH	165.1	C	12.28, <i>s</i>	1, 2, 9a	
2	107.5	CH	6.69, <i>d</i> , 2.4 Hz	1, 4	H-4
3	165.9	C			
4	108.7	CH	7.38, <i>d</i> , 2.4 Hz	2, 3, 10, 9a	H-2
5	121.2	CH	7.63, <i>br s</i>	6, 7, 9, 10, 8a	H-7, Me-6
6	148.3	C			
7	124.4	CH	7.08, <i>br s</i>	5, 8, 9, 8a	H-5, Me-6
8-OH	162.5	C	12.11, <i>s</i>	6, 7, 8, 8a	
9	190.7	C			
10	182.0	C			
4a	135.2	C			
4b	133.2	C			
8a	113.7	C			
9a	110.1	C			
1'	65.8	CH ₂	4.66, <i>d</i> , 6.9 Hz	3, 2', 3', 4', 5'	H-2', Me-4'
2'	118.2	CH	5.50, <i>br t</i> , 6.9 Hz	4', 5'	H-1', Me-5'
3'	139.7	C			
4'	18.3	CH ₃	1.81, <i>s</i>	2', 3', 5'	H-2'
5'	25.8	CH ₃	1.84, <i>s</i>	2', 3', 4'	H-1'
6-Me	22.1	CH ₃	2.46, <i>s</i>	5, 6, 7	H-5, H-7

3.1.2 Compound CPH2



Compound **CPH2** was isolated as a red-orange solid. The UV-Vis spectrum exhibited the absorption bands at 220, 278 and 425 nm, characteristic of a conjugated quinone system, which was confirmed by the presence of FT-IR absorption maxima indicating the presence of hydroxyl (3425 cm^{-1}) and chelated carbonyl (1624 cm^{-1}) groups.

The ^1H NMR spectral data (Table 3) of **CPH2** were similar to those of **CPH1** except for the presence of a singlet aromatic proton at δ 7.40 (1H, *s*, H-4) and *trans*-3,3-dimethylprop-1-enyl group at δ 6.92 (1H, *dd*, 6.9, 16.2 Hz, H-2'), 6.66 (1H, *dd*, 1.2, 16.2 Hz, H-1'), 2.50 (1H, *m*, H-3'), 1.14 (6H, *d*, 6.9 Hz, H-4' and H-5') instead of the *meta*-coupled aromatic protons at δ 7.38 (1H, *d*, 2.4 Hz, H-4) and 6.69 (1H, *d*, 2.4 Hz, H-2). The location of *trans*-3,3-dimethylprop-1-enyl group was assigned to C-2 by HMBC correlations (Figure 3) from chelated hydroxyl group at δ_{H} 12.84 (1-OH) to carbons at δ_{C} 110.5 (C-9a), 120.0 (C-2), 162.5 (C-1) and 191.4 (C-9) and the olefinic proton of *trans*-3,3-dimethylprop-1-enyl group at δ_{H} 6.66 (H-1') to carbons at δ_{C} 120.0 (C-2), 162.5 (C-1) and 163.0 (C-3). The ^1H NMR spectrum also showed a singlet signal of a methoxyl group at δ_{H} 4.05 (3H, *s*, 3-OMe). The attachment of a methoxyl group at C-3 was assigned by HMBC correlations of the olefinic proton at

δ_{H} 6.66 (H-1') to carbons at δ_{C} 120.0 (C-2), 162.5 (C-1) and 163.0 (C-3). The complete HMBC data in Table 3 confirmed the structure of CPH2. Therefore, compound CPH2 was identified as vismiaquinone A (Goncalves and Mors, 1981).

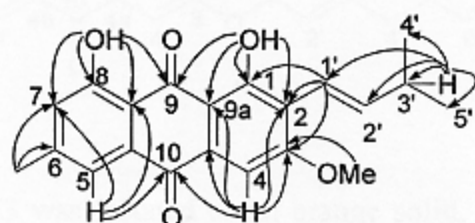
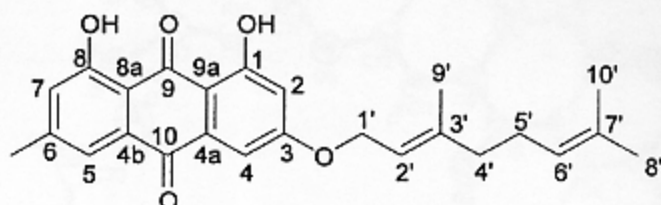


Figure 3 Selected HMBC correlations of CPH2

Table 3 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH2

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	162.5	C	12.84, <i>s</i>	1, 2, 9, 9a
2	120.0	C		
3	163.0	C		
4	103.4	CH	7.40, <i>s</i>	2, 3, 9, 10, 4a, 9a
5	121.1	CH	7.61, <i>br s</i>	6, 7, 8, 9, 10, 8a, 6-Me
6	148.4	C		
7	124.4	CH	7.07, <i>br s</i>	5, 8, 6-Me
8-OH	162.1	C	12.02, <i>s</i>	6, 7, 8, 8a
9	191.4	C		
10	181.9	C		
4a	132.1	C		
4b	133.2	C		
8a	113.7	C		
9a	110.5	C		
1'	115.8	CH	6.66, <i>dd</i> , 1.2, 16.2 Hz	1, 2, 3
2'	146.8	CH	6.92, <i>dd</i> , 6.9, 16.2 Hz	2
3'	33.4	CH	2.50, <i>m</i>	1', 2', 4', 5'
4'	22.5	CH ₃	1.14, <i>d</i> , 6.9 Hz	1', 3'
5'	22.5	CH ₃	1.14, <i>d</i> , 6.9 Hz	1', 3'
3-OMe	56.3	CH ₃	4.05, <i>s</i>	3
6-Me	22.2	CH ₃	2.45, <i>s</i>	6, 7, 4b

3.1.3 Compound CPH3



Compound **CPH3** was isolated as an orange solid, which was recrystallized from CHCl_3 -MeOH (9:1, w/v) to yield orange single crystals in needle shape. The UV-Vis and FT-IR spectra were similar to those of **CPH1**, indicating an anthraquinone skeleton.

The ^1H and ^{13}C NMR spectral data (**Table 4**) were similar to those of **CPH1**, except for the presence of the signal of an oxy-geranyl group at δ_{H} 5.47 (1H, *br t*, 6.6 Hz, H-2'), 5.08 (1H, *br t*, 6.6 Hz, H-6'), 4.67 (2H, *d*, 6.6 Hz, H-1'), 2.12 (4H, *m*, H-4' and H-5'), 1.78 (3H, *s*, H-9'), 1.68 (3H, *s*, H-8') and 1.61 (3H, *s*, H-10') instead of an oxy-isoprenyl group at C-3 of **CPH1**. The X-ray structure of **CPH1** which was shown in **Figure 4** confirmed the structure. In addition, the complete HMBC data were summarized in **Table 4**. Therefore, compound **CPH3** was identified as 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (Botta *et al.*, 1983; Boonnak *et al.*, 2005).

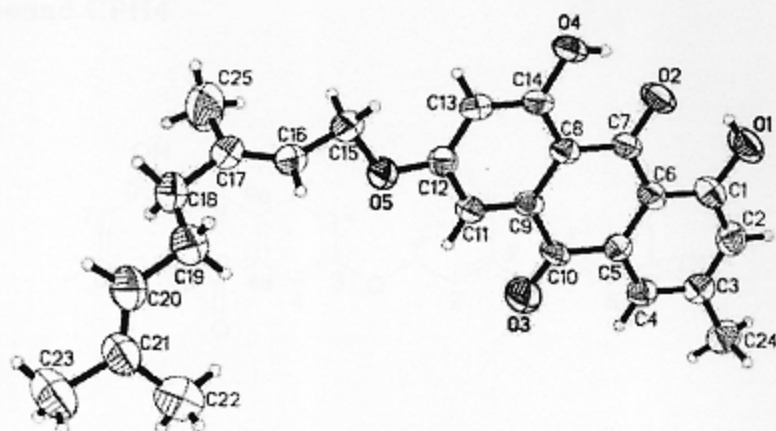
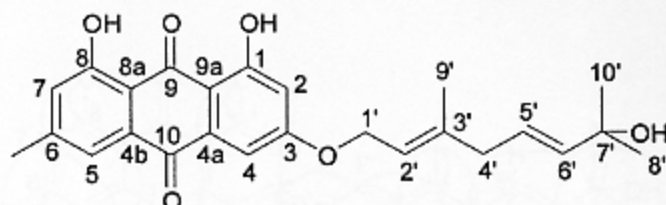


Figure 4 The X-ray structure of CPH3

Table 4 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH3

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	165.1	C	12.29, <i>s</i>	1, 2, 9a
2	107.4	CH	6.67, <i>br d</i> , 2.4 Hz	1, 4
3	165.9	C		
4	108.8	CH	7.36, <i>br d</i> , 2.4 Hz	2, 3, 9, 10, 9a
5	121.2	CH	7.61, <i>br s</i>	6, 7, 8, 9, 10, 8a, 6-Me
6	148.3	C		
7	124.4	CH	7.07, <i>br s</i>	5, 8, 8a, 6-Me
8-OH	162.4	C	12.13, <i>s</i>	6, 7, 8, 8a
9	190.6	C		
10	181.9	C		
4a	135.1	C		
4b	133.2	C		
8a	113.6	C		
9a	110.1	C		
1'	65.8	CH ₂	4.67, <i>d</i> , 6.6 Hz	3, 2', 3'
2'	118.2	CH	5.47, <i>br t</i> , 6.6 Hz	3', 4', 9'
3'	142.8	C		1', 2', 4', 5'
4'	39.5	CH ₂	2.12, <i>m</i>	3', 5', 6'
5'	26.2	CH ₂	2.12, <i>m</i>	3', 4', 7'
6'	123.6	CH	5.08, <i>br t</i> , 6.6 Hz	
7'	132.0	C		
8'	25.7	CH ₃	1.68, <i>s</i>	6', 7', 8'
9'	16.8	CH ₃	1.78, <i>s</i>	2', 3'
10'	17.7	CH ₃	1.61, <i>s</i>	6', 7', 10'
6-Me	22.1	CH ₃	2.45, <i>s</i>	6, 7, 4b

3.1.4 Compound CPH4



Compound **CPH4** was isolated as an orange viscous oil, which was assigned as $C_{25}H_{26}O_6$ from an exact mass measurement. The UV-Vis spectrum exhibited absorption maxima at 269, 283, 366 and 440 nm, suggesting an anthraquinone as a basic structure. FT-IR absorption bands at 1673 and 1625 cm^{-1} and ^{13}C NMR chemical shifts at δ 190.8 and 182.0 also indicated the presence of carbonyl and chelated carbonyl groups, respectively. Chelated hydroxyl protons were shown at δ_H 12.30 (1H, s, 1-OH) and 12.13 (1H, s, 8-OH).

The 1H and ^{13}C NMR spectral data of **CPH4** (Table 5) showed characteristics similar to those of **CPH3** (Table 4), except for the appearance of *trans*-olefinic protons at δ_H 5.62 (1H, *dd*, 6.5, 15.5 Hz, H-5') and 5.69 (1H, *d*, 15.5 Hz, H-6') in **CPH4** instead of methylene protons at C-5' and an olefin at C-6' of an oxy-geranyl group of **CPH3**. The chemical shift of the methylene protons at C-4' were shifted downfield (δ_H 2.79 (2H, *d*, 6.5 Hz, H-4')) compared to δ_H 2.12 of **CPH3** due to double allylic status of these protons. The location of H-4' at C-4' was supported by HMBC correlations. The chemical shift of C-7' (δ_C 70.8) suggested an oxy-quarternary carbon, whose position was confirmed by HMBC correlations with H-5' and H-6'.

Thus, CPH4 was designated as pruniflorone J, a new compound (Boonnak *et al.*, 2006).

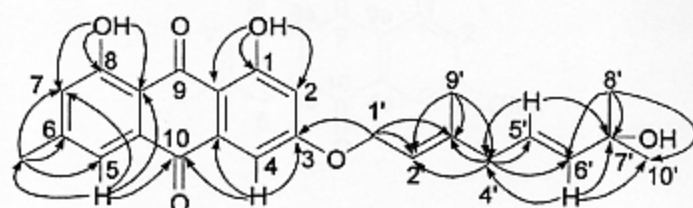
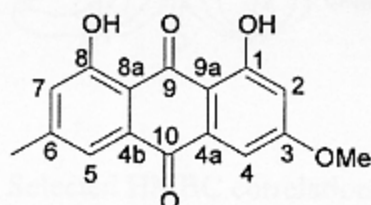


Figure 5 Selected HMBC correlations of CPH4

Table 5 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH4

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	165.1	C	12.30, <i>s</i>	1, 2, 9a
2	107.6	CH	6.68, <i>d</i> , 2.5 Hz	1, 4
3	165.9	C		
4	108.7	CH	7.37, <i>d</i> , 2.5 Hz	3, 10, 9a
5	121.3	CH	7.62, <i>s</i>	7, 10, 8a, 6-Me
6	148.4	C		
7	124.5	CH	7.08, <i>s</i>	5, 8, 8a, 6-Me
8-OH	163.0	C	12.13, <i>s</i>	7, 8, 8a
9	190.8	C		
10	181.9	C		
4a	135.2	C		
4b	133.2	C		
8a	113.6	C		
9a	110.1	C		
1'	65.8	CH ₂	4.68, <i>d</i> , 6.5 Hz	3, 2', 3'
2'	119.0	CH	5.50, <i>br t</i> , 6.6 Hz	4'
3'	142.8	C		
4'	24.1	CH ₂	2.79, <i>d</i> , 6.5 Hz	2', 3', 5', 6'
5'	123.9	CH	5.62, <i>dd</i> , 6.5, 15.5 Hz	4', 7'
6'	140.5	CH	5.69, <i>d</i> , 15.5 Hz	4', 7', 8'
7'	70.8	C		
8'	29.8	CH ₃	1.25, <i>s</i>	8'
9'	16.8	CH ₃	1.77, <i>s</i>	2', 3', 4'
10'	29.7	CH ₃	1.33, <i>s</i>	6', 7', 10'
6-Me	22.1	CH ₃	2.45, <i>s</i>	5, 6, 7

3.1.5 Compound CPH5



Compound **CPH5** was isolated as an orange solid. The UV-Vis spectrum exhibited the absorption bands at 222, 250, 267, 284 and 437 nm, characteristic of a conjugated quinone system, which was confirmed by the presence of FT-IR absorption maxima indicating the presence of hydroxyl (3350 cm^{-1}) and chelated carbonyl (1646 cm^{-1}) groups.

The ^1H and ^{13}C NMR spectral data of **CPH5** (**Table 6**) were similar to those of **CPH1** (**Table 2**), except for the presence of the signal of the methoxyl group at δ_{H} 3.88 (3H, *s*) instead of an oxy-isoprenyl group at C-3 of **CPH1**. The location of a methoxyl group at C-3 was assigned by HMBC correlations (**Figure 6**) of the aromatic proton at δ_{H} 6.67 (H-2) to the carbons at δ_{C} 108.2 (C-4), 110.3 (C-9a), 165.2 (C-1) and 166.6 (C-3) and of an aromatic proton at δ_{H} 7.35 (H-4) to the carbons at δ_{C} 106.8 (C-2), 110.3 (C-9a), 135.3 (C-4a) and 166.6 (C-3). The complete HMBC data were summarized in **Table 6**. Therefore, compound **CPH5** was identified as physcion (Kalidhar, 1989).

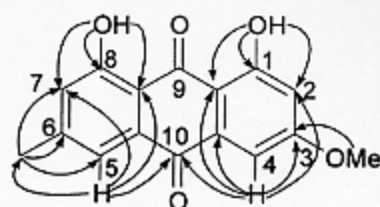
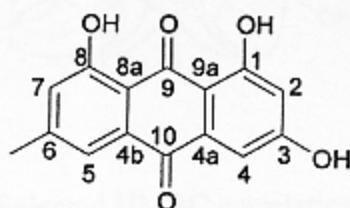


Figure 6 Selected HMBC correlations of CPH5

Table 6 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH5

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	165.2	C	12.31, <i>s</i>	1, 2, 9a
2	106.8	CH	6.67, <i>d</i> , 2.4 Hz	1, 3, 4, 9a
3	166.6	C		
4	108.2	CH	7.35, <i>d</i> , 2.4 Hz	2, 3, 9, 10, 4a, 9a
5	121.3	CH	7.60, <i>br d</i> , 1.5 Hz	7, 9, 10, 8a, 6-Me
6	148.4	C		
7	124.5	CH	7.07, <i>br s</i>	5, 8, 8a, 6-Me
8-OH	162.5	C	12.10, <i>s</i>	6, 7, 8, 8a
9	190.8	C		
10	182.0	C		
4a	135.3	C		
4b	133.2	C		
8a	113.7	C		
9a	110.3	C		
3-OMe	56.1	CH ₃	3.88, <i>s</i>	3
6-Me	22.2	CH ₃	2.45, <i>s</i>	5, 6, 7

3.1.6 Compound CPH6



Compound **CPH6** was isolated as an orange solid. The UV-Vis spectrum exhibited the absorption bands at 220, 251, 266, 285 and 441 nm, characteristic of a conjugated quinone system, which was confirmed by the presence of FT-IR absorption maxima indicating the presence of hydroxyl (3400 cm^{-1}) and chelated carbonyl (1642 cm^{-1}) groups.

The ^1H and ^{13}C NMR spectral data of **CPH6** (Table 7) were similar to those of **CPH5** (Table 6), except for the disappearance of the signal of the methoxyl group at δ_{H} 3.88 (3H, s), indicating that this methoxyl group was replaced by a hydroxyl group. The location of the hydroxyl group at C-3 was assigned by HMBC correlations (Figure 7) of the aromatic proton at δ_{H} 7.24 (H-4) to the carbons at δ_{C} 113.2 (C-2), 170.7 (C-3) and 186.7 (C-10). The complete HMBC data were summarized in Table 7. Therefore, compound **CPH6** was identified as emodin (Cohen and Towers, 1995).

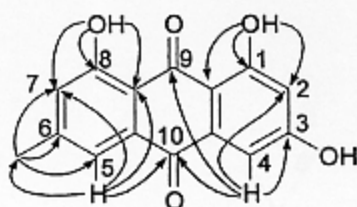
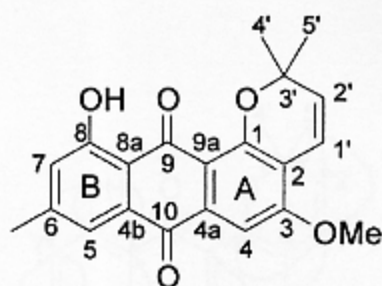


Figure 7 Selected HMBC correlations of CPH6

Table 7 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH6

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	170.0	C	12.20, <i>s</i>	1, 2, 9a
2	113.2	CH	6.62, <i>d</i> , 2.4 Hz	1, 4
3	170.7	C		
4	114.5	CH	7.24, <i>d</i> , 2.4 Hz	2, 3, 10
5	125.6	CH	7.55, <i>br d</i> , 2.4 Hz	7, 9, 10, 8a, 6-Me
6	152.8	C		
7	129.0	CH	7.07, <i>br d</i> , 2.4 Hz	5, 8, 8a, 6-Me
8-OH	167.0	C	12.12, <i>s</i>	7, 8, 8a
9	195.1	C		
10	186.7	C		
4a	138.0	C		
4b	140.1	C		
8a	118.4	C		
9a	113.2	C		
6-Me	20.8	CH ₃	2.45, <i>s</i>	5, 6, 7

3.1.7 Compound CPH7



Compound **CPH7** was isolated as an orange solid. The UV-Vis spectrum exhibited the absorption bands at 208, 224, 265, 285 and 424 nm, characteristic of a conjugated quinone system, which was confirmed by the presence of FT-IR absorption maxima indicating the presence of hydroxyl (3446 cm^{-1}) and chelated carbonyl (1646 cm^{-1}) groups.

The ^1H and ^{13}C NMR spectral data of **CPH7** (Table 8) were similar to those of **CPH2** (Table 3), except for the presence of the signal of the chromane ring at δ_{H} 6.73 (1H, *d*, 10.2 Hz, H-1'), 5.84 (1H, *d*, 10.2 Hz, H-2') and 1.57 (6H, *s*, H-4' and H-5') instead of chelated hydroxyl and *trans*-3,3-dimethylprop-1-enyl groups at δ_{H} 12.84 (1-OH, *s*) and 6.92 (1H, *dd*, 6.9, 16.2 Hz, H-2'), 6.66 (1H, *dd*, 1.2, 16.2 Hz, H-1'), 2.50 (1H, *m*, H-3') and 1.14 (6H, *d*, 6.9 Hz, H-4' and H-5'). The position of the chromane ring on ring A of **CPH7** was confirmed by HMBC correlations of an *olefinic* proton of chromane ring at δ_{H} 6.73 (H-1') to carbons at δ_{C} 77.8 (C-3'), 156.3 (C-1) and 158.8 (C-3). The selected HMBC correlations were shown in Figure 8 for confirmation of the structure of **CPH7**. Therefore, compound **CPH7** was identified as

11-hydroxy-5-methoxy-2,2,9-trimethyl-2*H*-anthra-[1,2-*b*]pyran-7,12-dione (Delle Monache *et al.*, 1979).

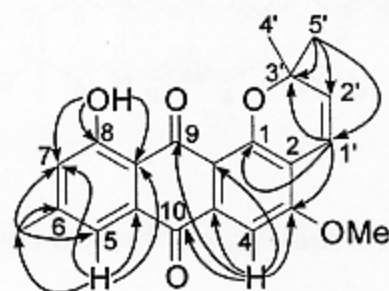
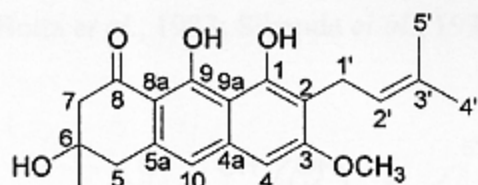


Figure 8 Selected HMBC correlations of CPH7

Table 8 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH7

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	156.3	C		
2	114.9	C		
3	158.8	C		
4	102.8	CH	7.43, <i>s</i>	3, 9, 10, 4a, 9a
5	119.8	CH	7.56, <i>dd</i> , 0.6, 1.5 Hz	7, 10, 8a, 6-Me
6	146.7	C		
7	124.5	CH	7.07, <i>dd</i> , 0.6, 1.5 Hz	5, 8, 8a, 6-Me
8-OH	162.6	C	13.18, <i>s</i>	7, 8, 8a
9	187.2	C		
10	182.8	C		
4a	135.4	C		
4b	132.6	C		
8a	115.4	C		
9a	116.3	C		
1'	116.1	CH	6.73, <i>d</i> , 10.2 Hz	1, 3, 3'
2'	132.2	CH	5.84, <i>d</i> , 10.2 Hz	1', 3', 4', 5'
3'	77.8	C		
4'	28.0	CH ₃	1.57, <i>s</i>	1', 2', 3'
5'	28.0	CH ₃	1.57, <i>s</i>	1', 2', 3'
3-OMe	56.2	CH ₃	4.03, <i>s</i>	3
6-Me	22.0	CH ₃	2.42, <i>s</i>	5, 6, 7

3.1.8 Compound CPH8



Compound **CPH8** was isolated as a greenish brown viscous oil. The UV-Vis spectrum showed maximum absorption bands at 242, 274 and 405 nm, indicating typical features of vismone (Sibanda *et al.*, 1993). The FT-IR spectrum showed the presence of the chelated hydroxyl and chelated carbonyl groups at 3414 and 1632 cm^{-1} , respectively.

The ^1H NMR spectral data of **CPH8** (Table 9) showed the singlet signal of chelated hydroxyl group at δ_{H} 16.14, which was assigned to the hydroxyl at C-9, *peri* to the hydroxyl (δ_{H} 9.95) and carbonyl groups. The singlet signal of a hydroxyl group at δ_{H} 9.95 was assigned to position C-1. The ^1H NMR spectra showed the signal of the isoprenyl and methoxyl groups at δ_{H} 5.24 (1H, *br t*, 6.9 Hz, H-2'), 3.44 (2H, *d*, 6.9 Hz, H-1'), 1.81 (3H, *s*, H-4') and 1.68 (3H, *s*, H-5') and 3.92 (3H, *s*). The positions of an isoprenyl group at C-2 and a methoxyl group at C-3, were assigned by HMBC correlations of a hydroxyl group at δ_{H} 9.95 (1-OH) to carbons at δ_{C} 108.5 (C-9a), 114.8 (C-2), 156.0 (C-1) and 163.9 (C-3), of a methoxyl group at δ_{H} 3.92 with 163.9 (C-3). The ^1H NMR spectra also showed two aromatic protons at δ_{H} 6.86 (1H, *s*, H-10) and 6.54 (1H, *s*, H-4). In addition, the presence of two sets of non-equivalent methylene protons were shown as doublets at δ_{H} 3.07 (1H, *d*, 16.5 Hz, H-5), 3.01 (1H, *d*, 16.5 Hz, H-5) and 2.87 (1H, *d*, 17.7 Hz, H-7), 2.80 (1H, *d*, 17.7 Hz, H-7) and an

aliphatic methyl signal at δ_H 1.44 (3H, *s*, 6-Me). The structure of CPH8 was confirmed by HMBC correlations in Figure 9. Therefore, compound CPH8 was identified as vismone E (Botta *et al.*, 1983; Sibanda *et al.*, 1993).

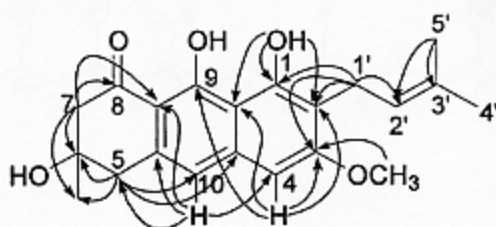
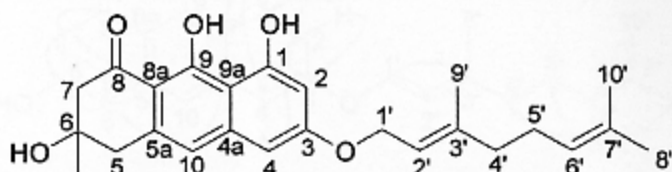


Figure 9 Selected HMBC correlations of CPH8

Table 9 1H , ^{13}C , HMQC and HMBC spectral data of CPH8

Position	^{13}C	DEPT	HMQC	HMBC ($^1H \rightarrow ^{13}C$)
1-OH	156.0	C	9.95, <i>s</i>	1, 2, 3, 9a
2	114.8	C		
3	163.9	C		
4	97.8	CH	6.54, <i>s</i>	2, 3, 9, 9a
5	43.4	CH ₂	3.07, <i>d</i> , 16.5 Hz 3.01, <i>d</i> , 16.5 Hz	6, 10, 4a, 6-Me
6	71.0	C		
7	51.1	CH ₂	2.87, <i>d</i> , 17.7 Hz 2.80, <i>d</i> , 17.7 Hz	6, 8, 8a, 6-Me
8	201.6	C		
9-OH	167.1	C	16.14, <i>s</i>	
10	117.6	CH	6.86, <i>s</i>	4, 5, 4a, 5a
4a	134.1	C		
5a	138.9	C		
8a	108.5	C		
9a	108.5	C		
1'	22.0	CH ₂	3.44, <i>d</i> , 6.9 Hz	1, 2, 3, 2'
2'	122.3	CH	5.24, <i>br t</i> , 6.9 Hz	
3'	131.7	C		
4'	17.8	CH ₃	1.81, <i>s</i>	2', 3'
5'	25.8	CH ₃	1.68, <i>s</i>	2', 3'
3-OMe	55.6	CH ₃	3.92, <i>s</i>	3
6-Me	28.8	CH ₃	1.44, <i>s</i>	5, 6, 7

3.1.9 Compound CPH9



Compound **CPH9** was isolated as a greenish brown viscous oil. The UV-Vis spectrum showed maximum absorption bands at 241, 278, 318, 335 and 405 nm, indicating typical features of vismone (Sibanda *et al.*, 1993; Botta *et al.*, 1983). The FT-IR spectrum showed the presence of a chelated hydroxyl and chelated carbonyl groups at 3400 and 1632 cm^{-1} , respectively.

The ^1H NMR spectral data of **CPH9** (Table 10) were similar to those of **CPH8**, except for the appearance of *meta*-coupled aromatic protons at δ_{H} 6.55 (1H, *d*, 2.4 Hz, H-4) and 6.50 (1H, *d*, 2.4 Hz, H-2) instead of a singlet aromatic proton and isoprenyl group at δ_{H} 6.54 (1H, *s*, H-4) and 5.24 (1H, *br t*, 6.9 Hz, H-2'), 3.44 (2H, *d*, 6.9 Hz, H-1'), 1.81 (3H, *s*, H-4') and 1.68 (3H, *s*, H-5') of **CPH8**. The ^1H NMR spectra also showed the signals of the oxy-geranyl group at δ 5.50 (1H, *br t*, 6.6 Hz, H-2'), 5.10 (1H, *br t*, 6.6 Hz, H-6'), 4.62 (2H, *d*, 6.6 Hz, H-1'), 2.12 (4H, *m*, H-4' and H-5'), 1.76 (3H, *s*, H-9'), 1.61 (3H, *s*, H-10') and 1.44 (3H, *s*, H-8'). The position of the oxy-geranyl group at C-3 was assigned by HMBC correlations from chelated hydroxyl group at δ_{H} 9.78 (1-OH) to carbons at δ_{C} 101.4 (C-4), 108.0 (C-9a), 159.7 (C-3) and 163.0 (C-1). The structure of **CPH9** was confirmed by HMBC correlations

in Figure 10. Therefore, compound CPH9 was identified as vismone D (Botta *et al.*, 1983).

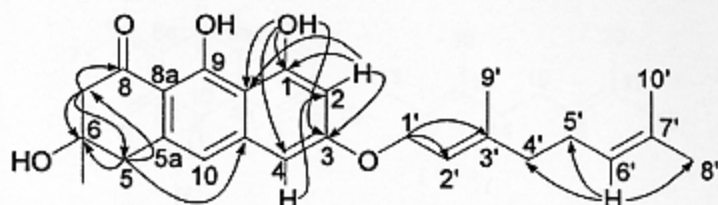
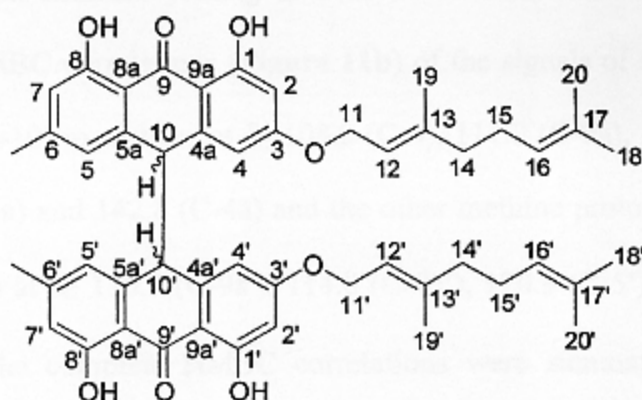


Figure 10 Selected HMBC correlations of CPH9

Table 10 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH9

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	163.0	C	9.78, <i>s</i>	1, 2, 3, 4, 9a
2	100.7	CH	6.50, <i>d</i> , 2.4 Hz	1, 3, 9a
3	159.7	C		
4	101.4	CH	6.55, <i>d</i> , 2.4 Hz	2
5	43.2	CH ₂	3.06, <i>d</i> , 16.5 Hz 3.01, <i>d</i> , 16.5 Hz	6, 4a
6	70.9	C		
7	50.9	CH ₂	2.86, <i>d</i> , 17.4 Hz 2.80, <i>d</i> , 17.4 Hz	6, 8
8	201.5	C		
9-OH	166.2	C	-	-
10	118.9	CH	6.84, <i>s</i>	-
4a	135.2	C		
5a	141.0	C		
8a	107.1	C		
9a	108.0	C		
1'	65.1	CH ₂	4.62, <i>d</i> , 6.6 Hz	2', 3'
2'	117.6	CH	5.50, <i>br t</i> , 6.6 Hz	
3'	141.7	C		
4'	39.5	CH ₂	2.12, <i>m</i>	
5'	26.3	CH ₂	2.12, <i>m</i>	
6'	123.7	CH	5.10, <i>br t</i> , 6.6 Hz	4', 5', 8'
7'	131.9	C		
8'	25.7	CH ₃	1.44, <i>s</i>	
9'	17.7	CH ₃	1.76, <i>s</i>	
10'	16.7	CH ₃	1.61, <i>s</i>	
6-Me	28.9	CH ₃	1.67, <i>s</i>	

3.1.10 Compound CPH10



Compound **CPH10** was isolated as a yellow-green solid. The UV-Vis spectrum showed the absorption bands at 207, 279 and 361 nm. The FT-IR spectrum exhibited absorption bands at 1613 and 3446 cm^{-1} indicating the presence of a chelated carbonyl and hydroxyl groups, respectively. It showed a molecular ion peak at m/z 782 $[\text{M}]^+$ in the EIMS (calcd for m/z 782) corresponding to a molecular formula of $\text{C}_{50}\text{H}_{54}\text{O}_8$. Moreover compound **CPH10** displayed a peak at m/z 392 $[\text{M}-390]^+$ corresponding to the molecular formula of $\text{C}_{25}\text{H}_{28}\text{O}_4$ which was the same as that of 3-*O*-geranylemodin anthrone (Abou-Shoer *et al.*, 1993; Sibanda *et al.*, 1993), a half structure of compound **CPH10**. The structure of 3-*O*-geranylemodin anthrone was shown in **Figure 11a**. These results suggested that compound **CPH10** was a bianthrone type.

The ^1H and ^{13}C NMR spectral data (**Table 11**) showed two sets of the signals, from which each set was similar to that of **CPH3** (**Table 4**). The difference was shown in the presence of the singlet methine protons at δ_{H} 4.33 (1H, *s*, H-10) and 4.32 (1H, *s*, H-10') instead of the carbonyl group at C-10 of **CPH3**. From these results, it

can be concluded that the structure of **CPH10** was produced from two units of 3-*O*-geranylemodin anthrone joining at C-10 of each monomer. The structure was confirmed by HMBC correlations (**Figure 11b**) of the signals of a methine proton at δ_{H} 4.33 (1H, *s*, H-10) to carbons at δ_{C} 108.2 (C-4), 111.0 (C-9a), 114.3 (C-8a), 120.7 (C-5), 139.9 (C-5a) and 142.8 (C-4a) and the other methine proton at δ_{H} 4.32 (1H, *s*, H-10') to carbons at δ_{C} 110.7 (C-9a'), 114.0 (C-8a'), 120.8 (C-5'), 140.2 (C-5a') and 143.5 (C-4a'). The complete HMBC correlations were summarized in **Table 11**. Compound **CPH10** was optically inactive and identified as bianthrone **A**₁ with a *meso* or (\pm)-form at C-10 and C-10'. Therefore, compound **CPH10** was assigned as bianthrone **A**₁ (Botta *et al.*, 1985).

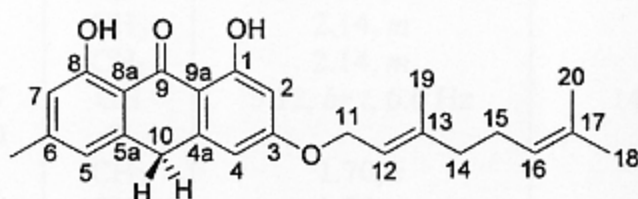


Figure 11a The structure of 3-*O*-geranylemodin anthrone

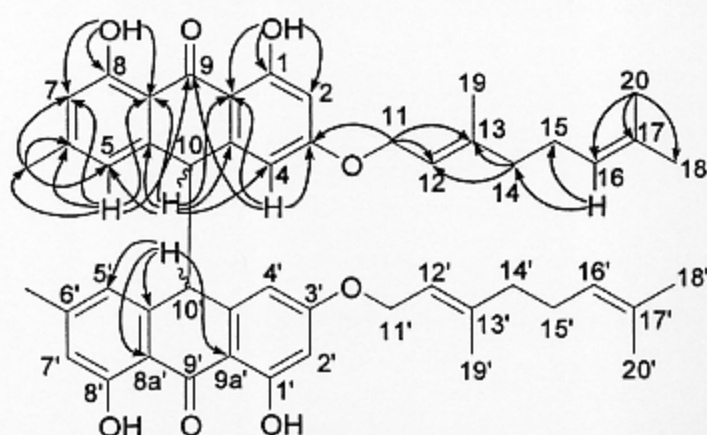


Figure 11b Selected HMBC correlations of **CPH10**

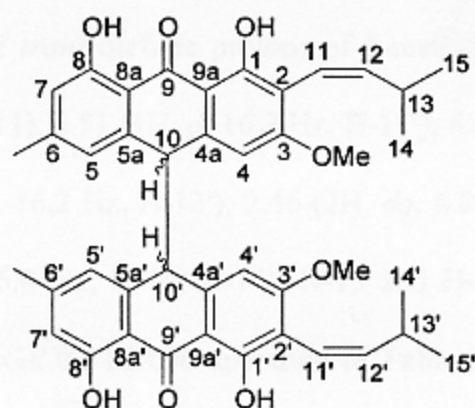
Table 11 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH10

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	164.6	C	12.20, <i>s</i>	1, 2, 9a
2	100.8	CH	6.40, <i>d</i> , 2.4 Hz	1, 2, 3, 9a
3	164.7	C		
4	108.2	CH	6.14, <i>d</i> , 2.4 Hz	3, 9, 9a
5	120.7	CH	6.11, <i>br s</i>	6, 7, 9, 8a, 6-Me
6	146.8	C		
7	117.0	CH	6.69, <i>br s</i>	5, 8a, 6-Me
8-OH	162.0	C	11.91, <i>s</i>	7, 8, 8a
9	190.3	C		
10	56.6	CH	4.33, <i>s</i>	4, 5, 4a, 5a, 8a, 9a
4a	142.8	C		
5a	139.9	C		
8a	114.3	C		
9a	111.0	C		
11	65.4	CH ₂	4.58, <i>d</i> , 6.6 Hz	3, 12, 13
12	118.3	CH	5.49, <i>br t</i> , 6.6 Hz	11, 14
13	142.5	C		
14	39.6	CH ₂	2.14, <i>m</i>	12, 13
15	26.3	CH ₂	2.14, <i>m</i>	16
16	123.7	CH	5.12, <i>br t</i> , 6.6 Hz	14, 15, 18, 20
17	132.0	C		
18	25.7	CH ₃	1.70, <i>s</i>	16, 17, 20
19	16.8	CH ₃	1.79, <i>s</i>	12, 13
20	17.7	CH ₃	1.63, <i>s</i>	16, 17, 18
6-Me	22.0	CH ₃	2.30, <i>s</i>	5, 6, 7

Table 11 (continued)

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1'-OH	164.6	C	12.12, <i>s</i>	1', 2', 9a'
2'	100.7	CH	6.35, <i>d</i> , 2.4 Hz	1', 2', 3'
3'	164.7	C		
4'	108.4	CH	5.99, <i>d</i> , 2.4 Hz	3', 9', 9a'
5'	120.8	CH	5.97, <i>br s</i>	6', 9', 8a', 6'-Me
6'	146.6	C		
7'	116.9	CH	6.69, <i>br s</i>	5', 8a', 6'-Me
8'-OH	162.0	C	11.81, <i>s</i>	7, 8, 8a'
9'	190.3	C		
10'	56.6	CH	4.32, <i>s</i>	5', 4a', 5a', 8a', 9a'
4a'	143.5	C		
5a'	140.2	C		
8a'	114.0	C		
9a'	110.7	C		
11'	65.3	CH ₂	4.53, <i>d</i> , 6.6 Hz	3', 12', 13'
12'	118.3	CH	5.47, <i>br t</i> , 6.6 Hz	11', 14'
13'	142.5	C		
14'	39.6	CH ₂	2.14, <i>m</i>	12', 13'
15'	26.3	CH ₂	2.14, <i>m</i>	16'
16'	123.7	CH	5.12, <i>br t</i> , 6.6 Hz	14', 15', 18', 20'
17'	132.0	C		
18'	25.7	CH ₃	1.70, <i>s</i>	16', 17'
19'	16.7	CH ₃	1.78, <i>s</i>	12', 13'
20'	17.7	CH ₃	1.63, <i>s</i>	16', 17'
6'-Me	22.0	CH ₃	2.28, <i>s</i>	5', 6', 7'

3.1.11 Compound CPH11



Compound **CPH11** was isolated as a yellow-green solid. The UV-Vis spectrum showed the absorption bands at 210, 219, 273 and 360 nm. The FT-IR spectrum exhibited absorption bands at 1631 and 3446 cm^{-1} indicating the presence of a chelated carbonyl and hydroxyl groups, respectively. It showed a molecular ion peak at m/z 674.2864 $[\text{M}]^+$ in the HREIMS (calcd for m/z 674.2880) corresponding to a molecular formula of $\text{C}_{42}\text{H}_{42}\text{O}_8$. Moreover compound **CPH11** displayed a peak at m/z 338 $[\text{M}-336]^+$ corresponding to the molecular formula of $\text{C}_{21}\text{H}_{22}\text{O}_4$ which was the same as that of 2-(3-methyl-1-butenyl)-1,8-dihydroxy-3-methoxy-6-methyl-anthrone (Iinuma *et al.*, 1995), a half structure of compound **CPH11**. The structure of 2-(3-methyl-1-butenyl)-1,8-dihydroxy-3-methoxy-6-methyl-anthrone was shown in the **Figure 12a**. These results suggested that compound **CPH11** was a bianthrone type.

The ^1H and ^{13}C NMR spectral data (**Table 12**) showed two sets of the signals which were similar to those of bivismiaquinone (**Figure 12b**) except for the appearance of the *cis*-olefinic protons of 3-methyl-1-butenyl groups at δ_{H} 6.63 (1H, *t*,

6.6 Hz, H-12 and H-12'), 6.53 (1H, *dd*, 0.7, 6.6 Hz, H-11,) and 6.47 (1H, *dd*, 0.7, 6.6 Hz, H-11'), 2.48 (2H, *m*, H-13 and H-13') and 1.20 (12H, *d*, 6.6 Hz, H-14, H-14', H-15 and H-15') instead of *trans*-olefinic protons of 3-methyl-1-butenyl groups at δ_{H} 6.45 (1H, *d*, 16.2 Hz, H-11), 6.51 (1H, *d*, 16.2 Hz, H-11'), 6.60 (1H, *dd*, 6.8, 16.2 Hz, H-12), 6.66 (1H, *dd*, 6.8, 16.2 Hz, H-12'), 2.46 (2H, *dq*, 6.8, 6.8 Hz, H-13 and H-13') and 1.09 (12H, *d*, 6.8 Hz, H-14, H-14', H-15 and H-15') of bivismiaquinone (Hussein *et al.*, 2003). From the HMBC spectrum in **Table 12**, the signals at δ_{H} 6.53 and 6.47 were assigned to H-11 and H-11' respectively as they exhibited correlations with δ_{C} 143.6 (C-12/12') and 113.4 (C-2/2') while the signals of 1-OH and 1'-OH were correlated with δ_{C} 161.7 (C-1), 113.4 (C-2), 110.6 (C-9a) and 161.3 (C-1'), 113.4 (C-2'), 111.1 (C-9a'), which confirmed the attachment of the 3-methyl-1-butenyl moiety at C-2 and C-2'. The selected HMBC correlations were shown in **Figure 12c**. In this case compound **CPH11** was optically inactive and designated as bianthrone **J**, a new compound with a *meso* or (\pm)-form at C-10 and C-10'. Compound **CPH11** was therefore a geometrical isomer of bivismiaquinone.

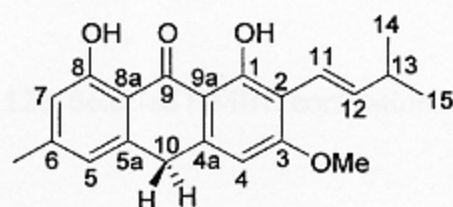


Figure 12a The structure of 2-(3-methyl-1-butenyl)-1,8-dihydroxy-3-methoxy-6-methyl-anthrone

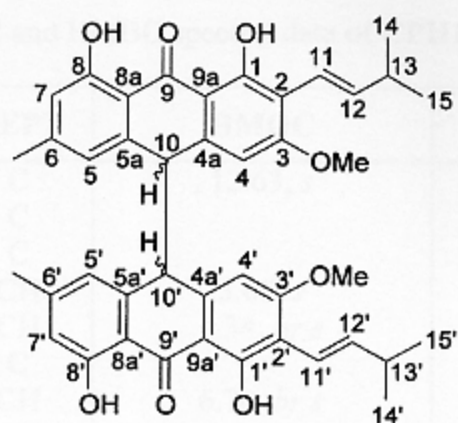


Figure 12b The structure of **bivismiaquinone**

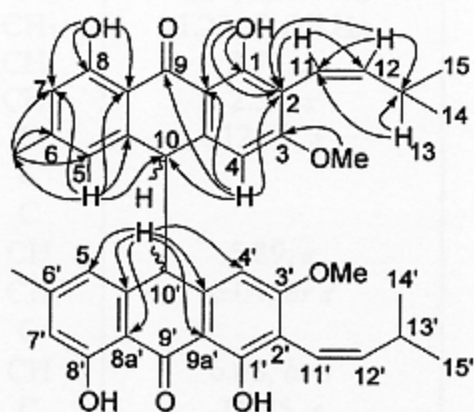
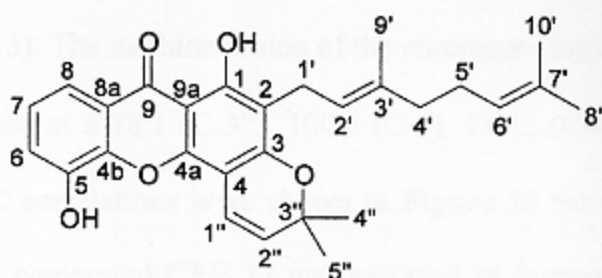


Figure 12c Selected HMBC correlations of **CPH11**

Table 12 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH11

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	161.7	C	12.63, <i>s</i>	1, 2, 9a
2	113.4	C		
3	162.0	C		
4	103.1	CH	5.65, <i>s</i>	2, 9, 9a
5	120.7	CH	6.34, <i>br s</i>	7, 10, 5a, 8a, 6-Me
6	146.9	C		
7	117.0	CH	6.70, <i>br s</i>	5
8-OH	161.7	C	11.91, <i>s</i>	7, 8, 8a
9	190.7	C		
10	56.8	CH	4.26, <i>s</i>	4, 5, 4a, 5a, 8a, 9a
4a	140.1	C		
5a	141.5	C		
8a	114.7	C		
9a	110.6	C		
11	115.6	CH	6.53, <i>dd</i> , 0.7, 6.6 Hz	2, 12
12	143.6	CH	6.63, <i>t</i> , 6.6 Hz	2, 11, 13
13	33.1	CH	2.48, <i>m</i>	11, 12
14	22.6	CH ₃	1.20, <i>d</i> , 6.6 Hz	12, 13
15	22.6	CH ₃	1.20, <i>d</i> , 6.6 Hz	12, 13
3-OMe	55.5	CH ₃	3.71, <i>s</i>	3
6-Me	22.0	CH ₃	2.34, <i>s</i>	5, 6, 7
1'-OH	161.3	C	12.68, <i>s</i>	1', 2', 9a'
2'	113.4	C		
3'	162.0	C		
4'	103.0	CH	5.89, <i>s</i>	2', 9', 10', 9a'
5'	120.7	CH	6.04, <i>br s</i>	7', 10', 5a', 8a', 6'-Me
6'	146.6	C		
7'	117.0	CH	6.68, <i>br s</i>	5'
8'-OH	161.9	C	11.85, <i>s</i>	7', 8', 8a'
9'	190.6	C		
4a'	140.6	C		
5a'	140.6	C		
8a'	114.4	C		
9a'	111.2	C		
10'	57.0	CH	4.25, <i>s</i>	4', 5', 4a', 5a'
11'	115.6	CH	6.47, <i>dd</i> , 0.7, 6.6 Hz	2', 12'
12'	143.6	CH	6.63, <i>t</i> , 6.6 Hz	2', 11', 13'
13'	33.1	CH	2.48, <i>m</i>	11', 12'
14'	22.6	CH ₃	1.20, <i>d</i> , 6.6 Hz	12', 13'
15'	22.6	CH ₃	1.20, <i>d</i> , 6.6 Hz	12', 13'
3'-OMe	55.6	CH ₃	3.80, <i>s</i>	3'
6'-Me	22.0	CH ₃	2.29, <i>s</i>	5', 6'

3.1.12 Compound CPH12



Compound **CPH12** was isolated as a yellow solid, mp. 144-146 °C. The UV-Vis spectrum showed absorption bands at 205, 223, 253, 327 and 369 nm, which indicated a typical xanthone chromophore (Seo *et al.*, 2002). The FT-IR spectrum exhibited conjugated carbonyl group at 1642 cm^{-1} and hydroxyl group at 3424 cm^{-1} .

The ^1H NMR spectrum (Table 13) exhibited signals of a chelated hydroxyl group at δ 13.20 (s, 1-OH) and 1,2,3-trisubstituted benzene ring at δ 7.25 (1H, *t*, 7.8 Hz, H-7), 7.31 (1H, *dd*, 1.8, 7.8 Hz, H-6) and 7.79 (1H, *dd*, 1.8, 7.8 Hz, H-8). The lowest-field aromatic-proton (δ 7.79) was assigned to H-8 due to the deshielding of carbonyl functionality. The presence of geranyl moiety was suggested by the following ^1H NMR spectral data at δ 5.26 (1H, *br t*, 7.2 Hz, H-2'), 5.09 (1H, *br t*, 7.2 Hz, H-6'), 3.38 (2H, *d*, 7.2 Hz, H-1'), 2.02 (4H, *m*, H-4' and H-5'), 1.82 (3H, *s*, H-9'), 1.64 (3H, *s*, H-8') and 1.58 (3H, *s*, H-10'). The location of a geranyl group at C-2 was confirmed by HMBC correlations of a chelated hydroxyl group at δ 13.20 (1-OH) to carbon at δ 103.2 (C-9a), 112.3 (C-2) and 160.6 (C-1) and the methylene protons at δ 3.38 (2H, H-1') to carbon at δ 112.3 (C-2), 121.7 (C-2'), 135.2 (C-3'), 158.7 (C-3) and 160.6 (C-1). Moreover, the presence of chromene ring signal were shown in

^1H NMR spectral data at δ 6.80 (1H, *d*, 9.9 Hz, H-1''), 5.65 (1H, *d*, 9.9 Hz, H-2'') and 1.50 (6H, *s*, H-4'' and H-5'') whose attachment positions were confirmed by HMBC experiment (**Table 13**). The methine proton of the chromene ring at δ 6.80 (H-1'') was correlated with carbon at δ 78.1 (C-3''), 100.6 (C-4), 149.2 (C-4a) and 158.7 (C-3). The selected HMBC correlations were shown in **Figure 13** for confirmation of this structure. Therefore, compound **CPH 12** was assigned as formoxanthone B (Boonsri *et al.*, 2006).

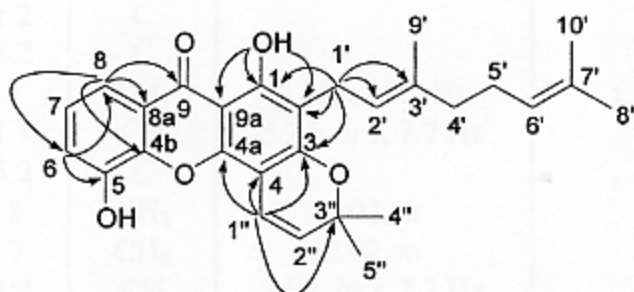
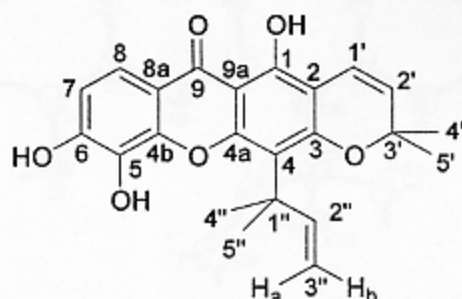


Figure 13 Selected HMBC correlations of **CPH12**

Table 13 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH12

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	160.6	C	13.20, <i>s</i>	1, 2, 9a
2	112.3	C		-
3	158.7	C		-
4	100.6	C		-
5	144.2	C		-
6	120.1	CH	7.31, <i>dd</i> , 1.8, 7.8 Hz	5, 8
7	124.0	CH	7.25, <i>t</i> , 7.8 Hz	5, 8a
8	117.2	CH	7.79, <i>dd</i> , 1.8, 7.8 Hz	6, 9, 4b, 8a
9	180.8	C		-
4a	149.2	C		-
4b	144.1	C		-
8a	121.2	C		-
9a	103.2	C		-
1'	21.1	CH ₂	3.38, <i>d</i> , 7.2 Hz	1, 2, 3, 2', 3'
2'	121.7	CH	5.26, <i>br t</i> , 7.2 Hz	1', 4', 9'
3'	135.2	C		-
4'	39.8	CH ₂	2.02, <i>m</i>	5', 9'
5'	26.7	CH ₂	2.02, <i>m</i>	4'
6'	124.4	CH	5.09, <i>br t</i> , 7.2 Hz	-
7'	131.3	C		-
8'	25.7	CH ₃	1.64, <i>s</i>	6', 7', 10'
9'	16.3	CH ₃	1.82, <i>s</i>	2', 3', 4'
10'	17.7	CH ₃	1.58, <i>s</i>	6', 7', 8'
1''	115.0	CH	6.80, <i>d</i> , 9.9 Hz	3, 4, 4a, 3''
2''	127.4	CH	5.65, <i>d</i> , 9.9 Hz	4, 3'', 4'', 5''
3''	78.1	C		-
4''	28.2	CH ₃	1.50, <i>s</i>	2'', 3''
5''	28.2	CH ₃	1.50, <i>s</i>	2'', 3''

3.1.13 Compound CPH13



Compound **CPH13** was isolated as a brown-yellow solid, which was recrystallized from CHCl_3 -MeOH (4:1, v/v) to yield brown-yellow crystals, mp. 183-184 °C. The X-ray structure of **CPH13** (Figure 14) confirmed a structure with a xanthone skeleton.

Its structure was supported by ^1H and ^{13}C NMR spectral data in Table 14. The ^1H NMR spectral data (Table 14) showed the presence of a chelated hydroxyl group at δ 13.53 (s, 1-OH), two *ortho*-coupled aromatic protons at δ 7.68 (1H, *d*, 9.0 Hz, H-8), and 6.94 (1H, *d*, 9.0 Hz, H-7) and 1,1-dimethylprop-2-enyl at δ 6.76 (1H, *dd*, 10.5, 17.7 Hz, H-2''), 5.22 (1H, *dd*, 1.5, 17.7 Hz, H-3a''), 5.05 (1H, *dd*, 1.5, 10.5 Hz, H-3b'') and 1.65 (6H, *s*, H-4'' and H-5''). Moreover, the presence of the signals of the chromene ring were shown at δ_{H} 6.76 (1H, *d*, 9.9 Hz, H-1'), 5.61 (1H, *d*, 9.9 Hz, H-2') and 1.52 (6H, *s*, H-4' and H-5'). The HMBC data of **CPH13** were summarized in Table 14. Therefore, compound **CPH13** was identified as macluraxanthone by comparison of its spectral data with those reported in the literature (Delle Monache *et al.*, 1981; Fun *et al.*, 2006).

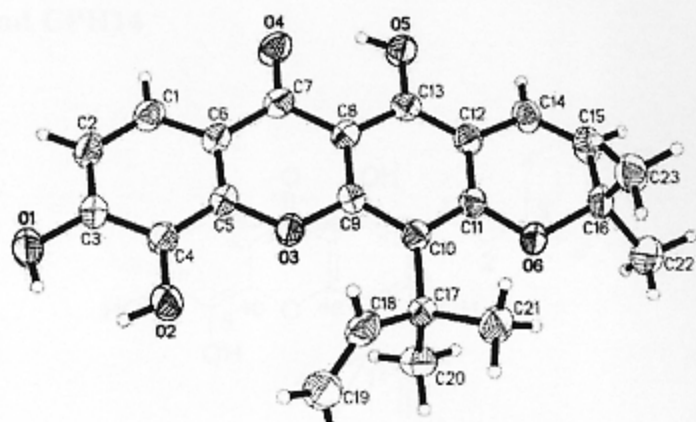
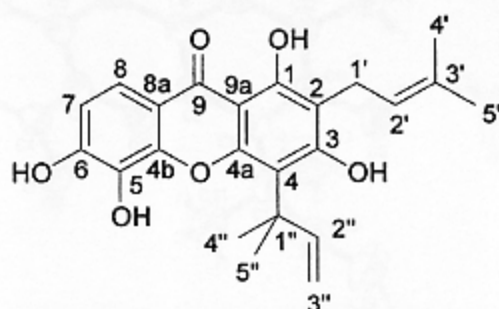


Figure 14 The X-ray structure of CPH13

Table 14 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH13

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	156.8	C	13.53, <i>s</i>	1, 2, 9a
2	105.5	C		
3	158.9	C		
4	113.1	C		
5	131.0	C		
6	149.0	C		
7	112.8	CH	6.94, <i>d</i> , 9.0 Hz	5, 6, 8a
8	117.5	CH	7.68, <i>d</i> , 9.0 Hz	6, 9, 4b
9	180.8	C		
4a	154.1	C		
4b	144.5	C		
8a	113.7	C		
9a	103.0	C		
1'	116.1	CH	6.76, <i>d</i> , 9.9 Hz	1, 2, 3, 3'
2'	127.2	CH	5.61, <i>d</i> , 9.9 Hz	2, 3', 4', 5'
3'	78.3	C		
4'	27.9	CH ₃	1.52, <i>s</i>	2', 3'
5'	27.9	CH ₃	1.52, <i>s</i>	2', 3'
1''	41.4	C		
2''	156.8	CH	6.76, <i>dd</i> , 10.5, 17.7 Hz	1'', 3'', 4'', 5''
3''	103.3	CH ₂	5.22, <i>dd</i> , 1.5, 17.7 Hz 5.05, <i>dd</i> , 1.5, 10.5 Hz	1'', 2''
4''	28.2	CH ₃	1.65, <i>s</i>	4, 1'', 2''
5''	28.2	CH ₃	1.65, <i>s</i>	4, 1'', 2''

3.1.14 Compound CPH14



Compound **CPH14** was isolated as a yellow solid, which was recrystallized from CHCl_3 -MeOH (4:1, v/v) to give brown-yellow crystals, mp. 180-181 °C. Its UV-Vis and FT-IR spectra were similar to those of **CPH13**, indicating the xanthone skeleton.

The X-ray structure of **CPH14** (**Figure 15**) confirmed a structure with a xanthone skeleton. The ^1H and ^{13}C NMR spectral data (**Table 15**) were similar to those of **CPH13** (**Table 14**) except for the appearance of the signal of an isoprenyl group at δ_{H} 5.24 (1H, *br t*, 6.9 Hz, H-2'), 3.47 (2H, *d*, 6.9 Hz, H-1'), 1.86 (3H, *s*, H-4') and 1.79 (3H, *s*, H-5') instead of the chromene ring at δ_{H} 6.76 (1H, *d*, 9.9 Hz, H-1'), 5.61 (1H, *d*, 9.9 Hz, H-2') and 1.52 (6H, *s*, H-4' and H-5'). The attachment of the isoprenyl side chain at C-2 was confirmed by X-ray structure in **Figure 15**. The HMBC correlations data were summarized in **Table 15**. Therefore, compound **CPH14** was identified as gerontaxanthone I by comparison of the spectral data with those reported data (Botta *et al.*, 1986; Boonnak, Chantrapromma and Fun 2006).

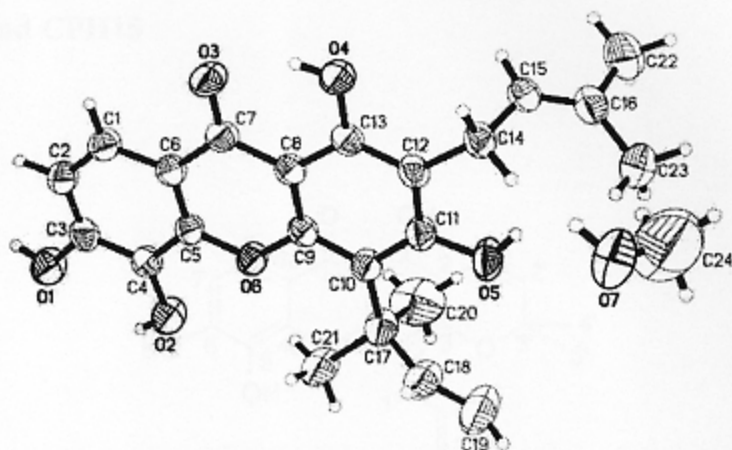
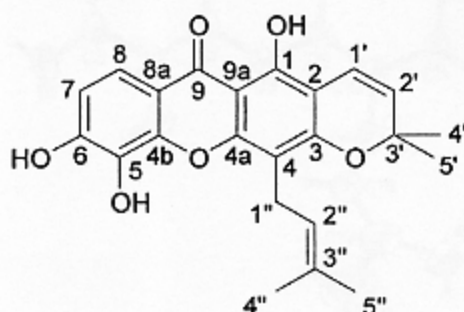


Figure 15 The X-ray structure of CPH14

Table 15 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH14

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	158.9	C	13.61, <i>s</i>	1, 2, 9a
2	110.2	C		
3	161.4	C		
4	111.1	C		
5	131.0	C		
6	149.0	C		
7	112.60	CH	6.94, <i>d</i> , 8.7 Hz	5, 6, 8a
8	117.6	CH	7.70, <i>d</i> , 8.7 Hz	6, 9, 4b
9	180.8	C		
4a	153.3	C		
4b	144.8	C		
8a	113.8	C		
9a	103.0	C		
1'	21.6	CH ₂	3.47, <i>d</i> , 6.9 Hz	1, 2, 3, 2', 3'
2'	121.2	CH	5.24, <i>br t</i> , 6.9 Hz	1', 4', 5'
3'	135.9	C		
4'	17.9	CH ₃	1.86, <i>s</i>	2', 3'
5'	25.9	CH ₃	1.79, <i>s</i>	2', 3'
1''	41.6	C		
2''	154.6	CH	6.68, <i>dd</i> , 10.5, 17.7 Hz	1'', 4'', 5''
3''	106.6	CH ₂	5.30, <i>dd</i> , 0.9, 17.7 Hz 5.15, <i>dd</i> , 0.9, 10.5 Hz	1'', 2''
4''	28.0	CH ₃	1.69, <i>s</i>	4, 1'', 2''
5''	28.0	CH ₃	1.69, <i>s</i>	4, 1'', 2''

3.1.15 compound CPH15



Compound **CPH15** was isolated as a yellow solid, which was recrystallized from CHCl_3 -MeOH (4:1, v/v) to give yellow needle crystals, mp. 218-219 °C. Its UV-Vis and FT-IR spectra were similar to those of **CPH13**, thus indicating the xanthone skeleton.

The X-ray structure of **CPH15** (Figure 16) confirmed a structure with a xanthone skeleton. The ^1H and ^{13}C NMR spectral data (Table 16) were similar to those of **CPH13** (Table 14) except for the presence of the signal of the isoprenyl moiety at δ_{H} 5.23 (1H, *br t*, 7.2 Hz, H-2''), 3.50 (2H, *d*, 7.2 Hz, H-1''), 1.88 (3H, *s*, H-4'') and 1.72 (3H, *s*, H-5'') instead of the 1,1-dimethylprop-2-enyl at δ 6.76 (1H, *dd*, 10.5, 17.7 Hz, H-2''), 5.22 (1H, *dd*, 1.5, 17.7 Hz, H-3a''), 5.05 (1H, *dd*, 1.5, 10.5 Hz, H-3b'') and 1.65 (6H, *s*, H-4'' and H-5'') at C-4 of **CPH13**. The HMBC correlations data of **CPH15** were summarized in Table 16. Therefore, compound **CPH15** was identified as xanthone V_1 by comparison of the spectral data with those reported data (Botta *et al.*, 1986; Chantrapromma *et al.*, 2005).

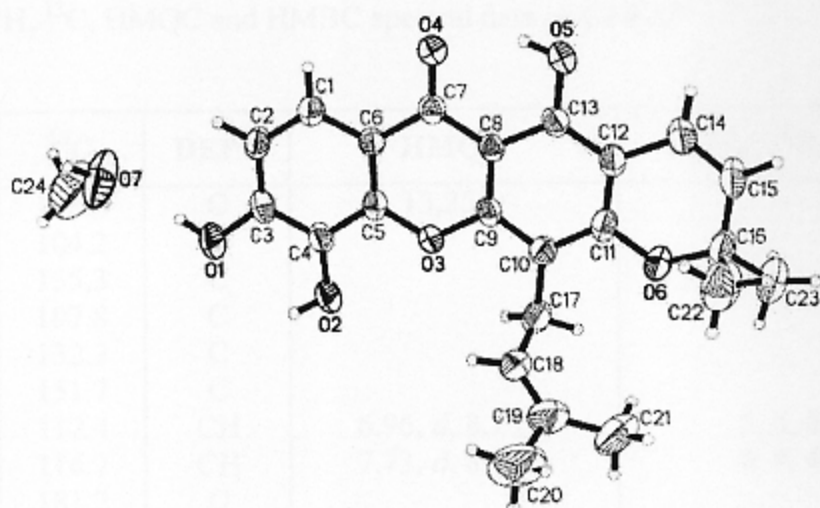
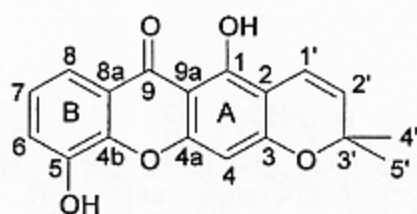


Figure 16 The X-ray structure of CPH15

Table 16 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH15

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	158.0	C	13.25, <i>s</i>	-
2	104.2	C		
3	155.3	C		
4	107.8	C		
5	132.3	C		
6	151.7	C		
7	112.4	CH	6.96, <i>d</i> , 8.7 Hz	5, 6, 8a
8	116.7	CH	7.73, <i>d</i> , 8.7 Hz	6, 9, 4b
9	181.2	C		
4a	154.3	C		
4b	146.4	C		
8a	113.8	C		
9a	102.6	C		
1'	115.6	CH	6.75, <i>d</i> , 9.9 Hz	1, 2, 3, 1', 2'
2'	127.3	CH	5.61, <i>d</i> , 9.9 Hz	2, 2', 3'
3'	78.2	C		
4'	28.0	CH ₃	1.49, <i>s</i>	1', 2', 3'
5'	28.0	CH ₃	1.49, <i>s</i>	1', 2', 3'
1''	21.3	CH ₂	3.50, <i>d</i> , 7.2 Hz	4, 4a, 2'', 3''
2''	122.3	CH	5.23, <i>br t</i> , 7.2 Hz	-
3''	131.4	C		
4''	17.6	CH ₃	1.88, <i>s</i>	2'', 3''
5''	25.5	CH ₃	1.72, <i>s</i>	2'', 3''

3.1.16 Compound CPH16



Compound **CPH16** was obtained as a yellow powder, mp. 222-224 °C. The UV-Vis spectrum of **CPH16** exhibited maxima at 260, 309 and 381 nm, suggesting the presence of a xanthone chromophore. Its FT-IR spectrum showed the absorption bands at 3400 and 1640 cm^{-1} , indicating the hydroxyl and conjugated carbonyl groups, respectively.

The ^1H and ^{13}C NMR spectral data (**Table 17**) showed the presence of 1,2,3-trisubstituted aromatic protons at δ_{H} 7.78 (1H, *dd*, 1.8, 7.8 Hz, H-8), 7.33 (1H, *dd*, 1.8, 7.8 Hz, H-6) and 7.26 (1H, *t*, 7.8 Hz, H-7) on ring B, which were similar to those of **CPH12** (**Table 13**). The signals of the chelated hydroxyl group and the singlet aromatic proton appeared at δ_{H} 12.98 (*s*, 1-OH) and 6.30 (*s*, H-4), respectively. Moreover, the ^1H NMR spectral data (**Table 17**) also showed characteristic of the chromene ring at δ 6.79 (1H, *d*, 9.9 Hz, H-1'), 5.65 (1H, *d*, 9.9 Hz, H-2') and 1.50 (6H, *s*, H-4' and H-5'). The attachment of a chromene ring on ring A was assigned by HMBC correlations (**Figure 17**) of chelated hydroxyl group at δ 12.98 (1-OH) to carbon at δ 101.0 (C-9a), 103.6 (C-2) and 164.6 (C-1) and the methine proton at δ 5.65 (H-2') to carbon at δ 78.3 (C-3') and 103.6 (C-2). By comparison of the

spectroscopic data with published data (Rocha *et al.*, 1994), therefore, compound CPH16 was deduced as deoxyjacareubin.

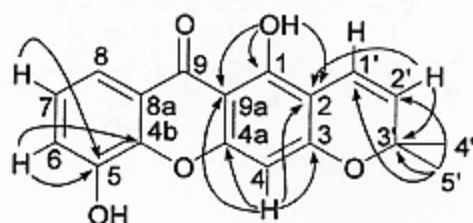
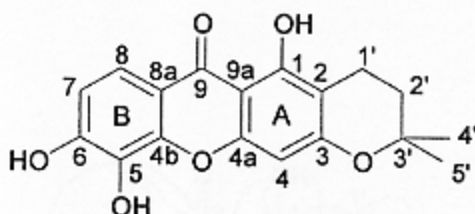


Figure 17 Selected HMBC correlations of CPH16

Table 17 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH16

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	164.6	C	12.98, <i>s</i>	1, 2, 9a
2	103.6	C		
3	161.0	C		
4	99.8	CH	6.30, <i>s</i>	2, 3, 4a, 9a
5	144.3	C		
6	120.4	CH	7.33, <i>dd</i> , 1.8, 7.8 Hz	5, 4a
7	124.2	CH	7.26, <i>t</i> , 7.8 Hz	5
8	117.2	CH	7.78, <i>dd</i> , 1.8, 7.8 Hz	-
9	180.7	C		
4a	163.3	C		
4b	144.1	C		
8a	121.1	C		
9a	101.0	C		
1'	114.6	CH	6.79, <i>d</i> , 9.9 Hz	2, 3, 3'
2'	127.7	CH	5.65, <i>d</i> , 9.9 Hz	2, 3'
3'	78.3	C		
4'	28.2	CH ₃	1.50, <i>s</i>	1', 2', 3'
5'	28.2	CH ₃	1.50, <i>s</i>	1', 2', 3'

3.1.17 Compound CPH17



Compound **CPH17** was isolated as a brown-yellow powder, mp. 122-124 °C. The UV-Vis spectrum exhibited maxima at 282, 338 and 380 nm, suggesting the presence of a xanthone chromophore. Its FT-IR spectrum showed the absorption bands at 3415 and 1646 cm^{-1} , indicating the hydroxyl and conjugated carbonyl groups, respectively.

The ^1H and ^{13}C NMR spectral data (**Table 18**) showed chelated hydroxyl group at δ_{H} 13.38 (*s*, 1-OH), two *ortho*-coupled aromatic protons at δ_{H} 7.67 (1H, *d*, 8.7 Hz, H-8) and 6.89 (1H, *d*, 8.7 Hz, H-7) and one singlet aromatic proton at δ_{H} 6.43 (1H, *s*). Moreover, the ^1H NMR spectral data (**Table 18**) showed the characteristic of the chromane ring at δ_{H} 2.73 (2H, *br t*, 6.9 Hz, H-1'), 1.87 (2H, *br t*, 6.9 Hz, H-2') and 1.39 (6H, *s*, H-4' and H-5'). The HMBC experiment (**Table 18**) was used to confirm the attachment of the chromane ring on ring A. The ^1H NMR signal of the hydroxyl group (1-OH) at δ 13.38 showed correlations to carbon at δ 102.0 (C-9a), 104.0 (C-2) and 160.1 (C-1), while the methylene protons (2H, H-1') at δ 2.73 showed correlations to carbon at δ 31.7 (C-2'), 76.3 (C-3'), 104.0 (C-2) and 161.2 (C-3). The

selected HMBC correlations were displayed in **Figure 18** to confirm this structure. By comparison of the spectroscopic values with previous report (King *et al.*, 1953), compound **CPH17** was identified as 3,4-dihydroxyjacareubin.

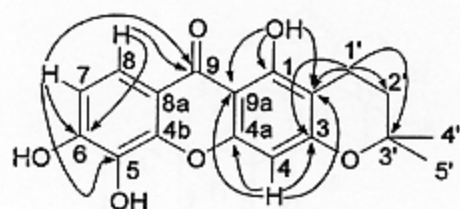
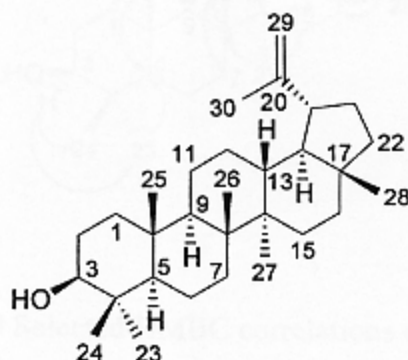


Figure 18 Selected HMBC correlations of CPH17

Table 18 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH17

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1-OH	160.1	C	13.38, <i>s</i>	1, 2, 9a
2	104.0	C		-
3	161.2	C		-
4	95.0	CH	6.43, <i>s</i>	2, 3, 4a, 9a
5	131.8	C		-
6	151.0	C		-
7	112.4	CH	6.89, <i>d</i> , 8.7 Hz	5, 6, 8a
8	116.9	CH	7.67, <i>d</i> , 8.7 Hz	6, 9
9	180.7	C		-
4a	155.8	C		-
4b	146.1	C		-
8a	114.0	C		-
9a	102.0	C		-
1'	15.9	CH ₂	2.73, <i>br t</i> , 6.9 Hz	2, 3, 2', 3'
2'	31.7	CH ₂	1.87, <i>br t</i> , 6.9 Hz	2, 1', 3', 4', 5'
3'	76.3	C		-
4'	26.6	CH ₃	1.39, <i>s</i>	1', 2', 3'
5'	26.6	CH ₃	1.39, <i>s</i>	1', 2', 3'

3.1.18 Compound CPH18



Compound **CPH18** was obtained as a white solid, mp. 193-194 °C. The FT-IR spectrum of this compound showed the absorption band at 3416 cm^{-1} (hydroxyl group). It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^1H and ^{13}C NMR spectra (**Table 19**) showed characteristic of lupane triterpenoids as seven methyl singlet signals at δ 0.76 (15.4), 0.79 (18.0), 0.83 (16.1), 0.94 (14.6), 0.97 (28.0) and 1.03 (16.0) including one vinylic methyl at δ 1.68 (19.3), two protons of an isopropenyl moiety at δ 4.68 (1H, *d*, 2.1 Hz), 4.56 (1H, *m*) and a typical lupane H_{β} -19 proton at δ 2.38 (*dt*, 5.7, 11.1 Hz). An oxymethine proton was shown at δ 3.19 (1H, *dd*, 5.1, 10.8 Hz, H-3). The doublet of doublet splitting pattern together with a large coupling constant of H-3 with $J_{\text{ax-ax}} = 10.8$ Hz and $J_{\text{ax-eq}} = 5.1$ Hz indicated an axial (α) orientation of H-3. The HMBC correlations were summarized in **Table 19** and the key HMBC correlations were shown in **Figure 19**. Therefore, compound **CPH18** was assigned as lupeol (Reynolds *et al.*, 1986; Thongdeeying 2005).

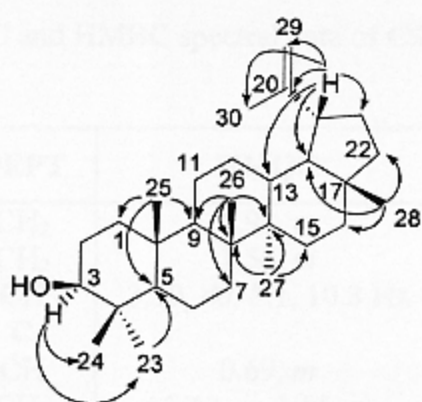
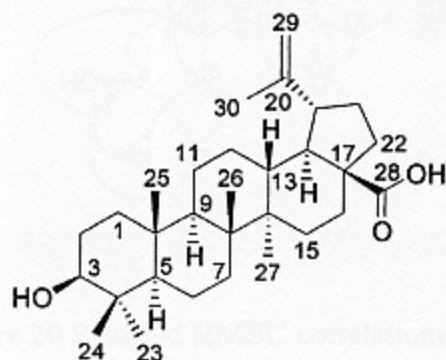


Figure 19 Selected HMBC correlations of CPH18

Table 19 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH18

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1	38.7	CH ₂	0.91, <i>m</i>	1, 4, 23, 24
2	27.4	CH ₂	1.56, <i>m</i>	
3	79.0	CH	3.19, <i>dd</i> , 5.1, 10.8 Hz	
4	38.9	C		
5	55.3	CH	0.69, <i>m</i>	
6	18.3	CH ₂	1.40, <i>m</i> , 1.55, <i>m</i>	
7	34.3	CH ₂	1.40, <i>m</i>	
8	40.8	C		
9	50.5	CH	1.28, <i>m</i>	
10	37.2	C		
11	20.9	CH ₂	1.22, <i>m</i> , 1.45, <i>m</i>	13, 18, 20, 21, 29, 30
12	25.2	CH ₂	1.08, <i>m</i>	
13	38.1	CH	1.67, <i>m</i>	
14	42.8	C		
15	27.5	CH ₂	1.56, <i>m</i>	
16	35.6	CH ₂	1.51, <i>m</i>	
17	43.0	C		
18	48.3	CH	1.38, <i>m</i>	
19	48.0	CH	2.38, <i>dt</i> , 5.7, 11.1 Hz	
20	151.0	C		
21	29.9	CH ₂	1.94, <i>m</i>	3, 4, 5, 24
22	40.0	CH ₂	1.20, <i>m</i> , 1.40, <i>m</i>	
23	28.0	CH ₃	0.97, <i>s</i>	
24	15.4	CH ₃	0.76, <i>s</i>	
25	16.1	CH ₃	0.83, <i>s</i>	
26	16.0	CH ₃	1.03, <i>s</i>	
27	14.6	CH ₃	0.94, <i>s</i>	
28	18.0	CH ₃	0.79, <i>s</i>	
29	109.3	CH ₂	4.56, <i>m</i> , 4.68, <i>d</i> , 2.1 Hz	
30	19.3	CH ₃	1.68, <i>s</i>	

3.1.19 Compound CPH19



Compound **CPH19** was obtained as a colorless crystal, mp. 279-280 °C. The FT-IR spectrum of this compound showed the absorption band at 3415 cm^{-1} (hydroxyl group) and 1686 cm^{-1} (carbonyl group). It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^1H and ^{13}C NMR spectra data of **CPH19** (Table 20 and Figure 20) were similar to those of **CPH18** (Table 19 and Figure 19) except for the appearance of the ^{13}C NMR signal of a carbonyl carbon at δ 179.1 instead of a methyl at δ_{C} 18.0, thus suggesting a carboxylic moiety at C-28. The location of a carboxylic moiety was confirmed by HMBC correlations (Table 20 and Figure 20), in which the methylene protons at δ_{H} 1.93 (2H-22) showed correlations with carbon at δ 56.1 (C-17) and 179.1 (C-28). Its spectroscopic data were comparable with the previous report (Macias *et al.*, 1994; Thongdeeying 2005), therefore, compound **CPH19** was assigned as betulinic acid.

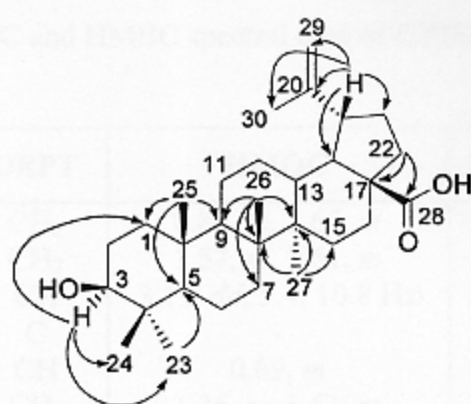


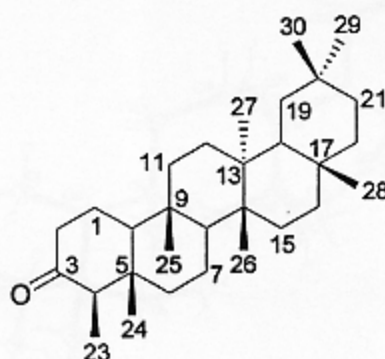
Figure 20 Selected HMBC correlations of **CPH19**

28.8	CH ₂	1.23, w, 1.17, w	
25.4	CH ₂	1.06, w	
24.2	CH	2.32, w	
42.3	C		
26.6	CH ₂	1.15, w, 1.21, w	
32.2	CH ₂	1.40, w, 2.25, w	
35.1	C		
49.1	CH	1.58, w	
46.9	CH	1.01, w	1H, 2H, 2H, 2H, 2H
130.7	C		
26.3	CH ₂	1.42, w, 1.91, w	
27.1	CH ₂	1.11, w, 1.93, w	
27.6	CH ₂	0.97, w	
15.2	CH ₂	0.75, w	
15.3	CH ₂	0.82, w	
15.6	CH ₂	0.74, w	
14.3	CH ₂	0.96, w	
179.1	C		
109.9	CH ₂	4.72, w, 4.81, w, w	2H, 2H
18.1	CH ₂	1.05, w	

Table 20 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH19

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1	38.7	CH ₂	0.88, <i>m</i> , 1.65, <i>m</i>	-
2	26.9	CH ₂	1.57, <i>m</i> , 1.61, <i>m</i>	-
3	78.7	CH	3.19, <i>dd</i> , 5.4, 10.8 Hz	1, 23, 24
4	38.7	C	-	-
5	55.3	CH	0.69, <i>m</i>	4, 6, 7, 9
6	18.2	CH ₂	1.36, <i>m</i> , 1.51, <i>m</i>	-
7	34.2	CH ₂	1.38, <i>m</i>	-
8	40.6	C	-	-
9	50.5	CH	1.26, <i>m</i>	-
10	37.1	C	-	-
11	20.8	CH ₂	1.23, <i>m</i> , 1.43, <i>m</i>	-
12	25.4	CH ₂	1.69, <i>m</i>	-
13	38.2	CH	2.22, <i>m</i>	-
14	42.3	C	-	-
15	29.6	CH ₂	1.15, <i>m</i> , 1.51, <i>m</i>	-
16	32.2	CH ₂	1.40, <i>m</i> , 2.25, <i>m</i>	-
17	56.1	C	-	-
18	49.1	CH	1.58, <i>m</i>	-
19	46.9	CH	3.01, <i>m</i>	18, 20, 21, 29, 30
20	150.7	C	-	-
21	30.5	CH ₂	1.42, <i>m</i> , 1.91, <i>m</i>	-
22	37.1	CH ₂	1.41, <i>m</i> , 1.93, <i>m</i>	17, 18, 28
23	27.6	CH ₃	0.97, <i>s</i>	3, 4, 5, 24
24	15.2	CH ₃	0.75, <i>s</i>	3, 4, 5, 23
25	15.9	CH ₃	0.82, <i>s</i>	1, 5, 9, 10
26	15.6	CH ₃	0.94, <i>s</i>	7, 8, 9, 14
27	14.5	CH ₃	0.98, <i>s</i>	8, 13, 14, 15
28	179.1	C	-	-
29	109.3	CH ₂	4.74, <i>br s</i> , 4.61, <i>br s</i>	19, 30
30	19.1	CH ₃	1.69, <i>s</i>	19, 20, 29

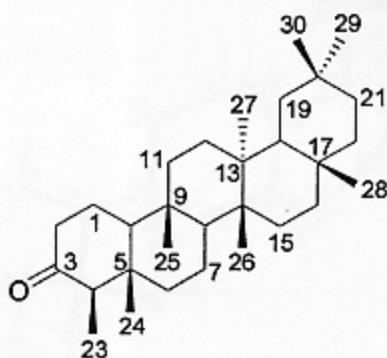
3.1.20 Compound CPH20



Compound **CPH20** was obtained as a white crystal, mp 245-247 °C. The FT-IR spectrum of this compound showed the absorption band at 1715 cm^{-1} (carbonyl group). It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^1H and ^{13}C NMR spectra (**Table 21**) showed characteristic of friedelan triterpenoids as seven methyl singlets at δ 0.72 (14.7), 0.87 (17.9), 0.95 (35.0), 1.00 (31.8), 1.01 (20.3), 1.05 (18.5), 1.18 (32.1) and one methyl doublet at δ 0.89 (3H, *d*, 6.3 Hz, H-23). The HMBC experiment (**Table 21**), in which methyl protons at δ 0.89 (H-23) were correlated with carbons at δ 42.2 (C-5), 58.2 (C-4) and 213.3 (C-3) confirmed the position of carbonyl group at C-3. The X-ray structure of **CPH20** was shown in **Figure 21**. Therefore, compound **CPH20** was assigned as friedelin (Ahad *et al.*, 1991).

3.1.20 Compound CPH20



Compound **CPH20** was obtained as a white crystal, mp 245-247 °C. The FT-IR spectrum of this compound showed the absorption band at 1715 cm^{-1} (carbonyl group). It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^1H and ^{13}C NMR spectra (**Table 21**) showed characteristic of friedelan triterpenoids as seven methyl singlets at δ 0.72 (14.7), 0.87 (17.9), 0.95 (35.0), 1.00 (31.8), 1.01 (20.3), 1.05 (18.5), 1.18 (32.1) and one methyl doublet at δ 0.89 (3H, *d*, 6.3 Hz, H-23). The HMBC experiment (**Table 21**), in which methyl protons at δ 0.89 (H-23) were correlated with carbons at δ 42.2 (C-5), 58.2 (C-4) and 213.3 (C-3) confirmed the position of carbonyl group at C-3. The X-ray structure of **CPH20** was shown in **Figure 21**. Therefore, compound **CPH20** was assigned as friedelin (Ahad *et al.*, 1991).

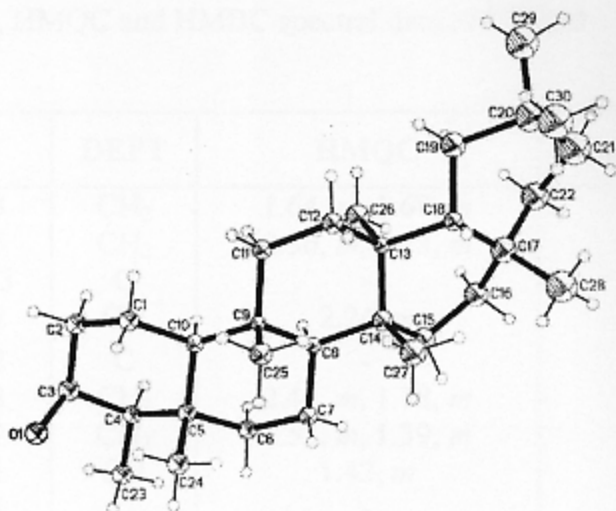
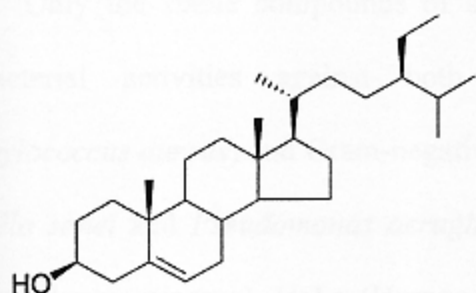


Figure 21 The X-ray structure of CPH20

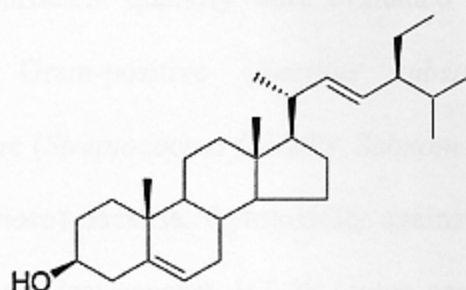
Table 21 ^1H , ^{13}C , HMQC and HMBC spectral data of CPH20

Position	^{13}C	DEPT	HMQC	HMBC ($^1\text{H} \rightarrow ^{13}\text{C}$)
1	22.3	CH ₂	1.64, <i>m</i> , 1.69, <i>m</i>	-
2	41.5	CH ₂	2.36, <i>m</i> , 2.23, <i>m</i>	-
3	213.3	C	-	-
4	58.2	CH	2.24, <i>m</i>	-
5	42.2	C	-	-
6	41.3	CH ₂	2.44, <i>m</i> , 1.78, <i>m</i>	-
7	18.2	CH ₂	1.52, <i>m</i> , 1.39, <i>m</i>	-
8	53.1	CH	1.42, <i>m</i>	-
9	37.4	C	-	-
10	35.6	CH ₂	1.61, <i>m</i> , 1.43, <i>m</i>	-
11	30.5	CH ₂	1.46, <i>m</i> , 1.34, <i>m</i>	-
12	39.7	C	-	-
13	38.3	C	-	-
14	32.4	CH ₂	1.51, <i>m</i> , 1.29, <i>m</i>	-
15	36.0	CH ₂	1.61, <i>m</i> , 1.36, <i>m</i>	-
16	30.0	C	-	-
17	42.8	CH	1.53, <i>m</i>	-
18	22.3	CH ₂	1.64, <i>m</i> , 1.69, <i>m</i>	-
19	35.3	CH ₂	1.62, <i>m</i> , 1.49, <i>m</i>	-
20	28.2	C	-	-
21	39.3	CH ₂	1.48, <i>m</i> , 0.93, <i>m</i>	-
22	32.8	CH ₂	1.50, <i>m</i> , 1.26, <i>m</i>	-
23	6.8	CH ₃	0.89, <i>d</i> , 6.3 Hz	3, 4, 5
24	14.7	CH ₃	0.72, <i>s</i>	4, 5, 6, 10
25	17.9	CH ₃	0.87, <i>s</i>	8, 9, 10, 11
26	20.3	CH ₃	1.01, <i>s</i>	8, 13, 14, 15
27	18.5	CH ₃	1.05, <i>s</i>	12, 13, 14, 18
28	32.1	CH ₃	1.18, <i>s</i>	16, 17, 18, 22
29	31.8	CH ₃	1.00, <i>s</i>	19, 20, 21
30	35.0	CH ₃	0.95, <i>s</i>	19, 20, 21

3.1.21 Compounds CPH21 and CPH22



CPH21



CPH22

The mixture of **CPH21** and **CPH22** was obtained as colorless crystals. The ^1H NMR spectra showed an oxymethine proton at δ 3.57-3.47 (*m*), three olefinic protons at δ 5.36-5.34 (*d*, 5.1 Hz), 5.16 (*dd*, 8.4, 15.1 Hz) and 5.01 (*dd*, 8.4, 15.1 Hz). The ^1H NMR spectral data of this compound corresponded to previous reported data (Thongdeeying 2005). Thus, the mixture was identified as β -sitosterol (**CPH21**) and stigmasterol (**CPH22**).

3.2 *Biological activities of the isolated compounds from the barks of C. formosum ssp. pruniflorum*

Only the stable compounds of sufficient quantity were evaluated for their antibacterial activities against both Gram-positive (*Bacillus subtilis* and *Staphylococcus aureus*) and Gram-negative (*Streptococcus faecalis*, *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*) bacteria. Cytotoxicity against MCF-7 (breast adenocarcinoma), HeLa (Human cervical cancer), HT-29 (colon cancer) and KB (human oral cancer) cell lines were also evaluated. The results of antibacterial activities of the tested compounds were given in **Table 22**. Compound **CPH8** exhibited potent antibacterial activities against *S. faecalis*, *S. typhi* and *S. sonnei*, whereas compound **CPH9** exhibited strong activity against *S. faecalis* and *S. sonnei*. Compounds **CPH14** and **CPH15** showed strong and broad spectrum of antibacterial activities compared to vancomycin. Compound **CPH17** showed inhibition against *B. subtilis* and *S. aureus*, whereas compound **CPH16** was highly active specifically against *S. aureus*. Compounds **CPH1-CPH7**, **CPH10** and **CPH11** showed no antibacterial activity.

From cytotoxicity result show in **Table 23**, compounds **CPH9** and **CPH14** strongly inhibited all cancer cell lines used in this investigation compared to camptothecin, whereas compounds **CPH8**, **CPH15** and **CPH17** showed less inhibitory activity. Compounds **CPH1-CPH7**, **CPH10**, **CPH11** and **CPH13** were found to be inactive for cytotoxic activity (**Table 23**).

Table 22 Antibacterial activities of the compounds isolated from the barks of

C. formosum ssp. *pruniflorum*

Compound	Minimum Inhibitive Concentration ($\mu\text{g/mL}$)					
	<i>B. subtilis</i>	<i>S. aureus</i>	<i>S. faecalis</i>	<i>S. typhi</i>	<i>S. sonnei</i>	<i>P. aeruginosa</i>
CPH1	-	-	-	-	-	-
CPH2	-	-	-	-	-	-
CPH3	-	-	-	-	-	-
CPH4	-	-	-	-	-	-
CPH5	-	-	-	-	-	-
CPH6	-	-	-	-	-	-
CPH7	-	-	-	-	-	-
CPH8	300	75	<1.1	<1.1	2.3	150
CPH9	300	150	<1.1	9.4	2.3	75
CPH10	300	300	300	300	300	300
CPH11	300	300	300	300	300	300
CPH13	4.6	4.6	2.3	9.6	-	-
CPH14	<1.1	<1.1	4.6	37.5	<1.1	<1.1
CPH15	<1.1	<1.1	<1.1	<1.1	-	9.3
CPH16	4.6	<1.1	75	-	150	150
CPH17	<1.1	<1.1	37.5	-	-	37.5
vancomycin	75	75	75	75	75	75

- = Inactive at $> 50.1 \mu\text{g/mL}$ MIC $< 1.1 \mu\text{g/ml}$ highly activeMIC = 1.2-5.0 $\mu\text{g/ml}$ very activeMIC = 5.1-10.0 $\mu\text{g/ml}$ activeMIC = 10.1-50.0 $\mu\text{g/ml}$ moderately activeMIC $> 50.1 \mu\text{g/ml}$ inactive

Table 23 *In vitro* cytotoxic activity of the compounds isolated from the barks of
C. formosum ssp. *pruniflorum*

Compound	Cell lines			
	IC ₅₀ (µg/mL)			
	MCF-7	HeLa	HT-29	KB
CPH1	-	-	-	-
CPH2	-	-	-	-
CPH3	-	-	-	-
CPH4	-	-	-	-
CPH5	-	-	-	-
CPH6	-	-	-	-
CPH7	-	-	-	-
CPH8	>5	>5	>5	4
CPH9	0.7	0.6	>5	0.22
CPH10	>300	>300	>300	>300
CPH11	>300	>300	>300	>300
CPH13	-	-	-	-
CPH14	0.6	0.7	0.7	0.6
CPH15	>25.0	4.7	6.0	2.7
CPH17	>5.0	3.4	>5.0	>5.0
camptothecin	0.2-2.0	0.2-2.0	0.2-2.0	0.2-2.0

- = Inactive at > 50.1 µg/mL

IC₅₀ < 1.0 µg/mL strongly active

IC₅₀ = 1.1-5.0 µg/mL very active

IC₅₀ = 5.1-10.0 µg/mL active

IC₅₀ = 10.1-50.0 µg/mL moderately active

IC₅₀ > 50.1 µg/mL inactive