2.1 INTRODUCTION

2.1.1 Introduction

***Garcinia cowa* Roxb**. is commonly known as “Cha muang” (ชะมวง). *G. cowa* is a small to medium-sized tree 15-30 m high. The trees are found scattered in lowland, undulating areas and peat swamp forests. The leaves are opposite, 7-15 cm long and 2-3.5 cm wide. The male flower is pink to red in colour and is found in clusters in the leaf axils and also below the leaves. The flower has 4 sepals and 4 petals. The fruit is a subglobose berry, 3 cm in diameter and turns dull orange-yellow when ripe. The seeds are embedded in the edible orange pulp. Both the young shoots and fruit are edible. Young leaves and shoots are used in many Thai soups (Poomipamorn, *et al*., 1997).

Figure 2 *Garcinia cowa*

2.1.2 Review of Literatures

All were the same as those described in part 1 (see **Table 1**).

2.1.3 The Objectives

Based on NAPRALERT database, phytochemical examination on the fruits of *G. cowa* has not yet been reported. These prompted us to investigate its chemical constituents in order to provide additional information of this plant. This research involved isolation, purification and structural elucidation of the chemical constituents isolated from the fruits of *G. cowa*, which was collected from Sathingmoo district, amphur Singhanakhon, Songkhla province.

2.2 EXPERIMENTAL

2.2.1 Chemicals and Instruments

All were the same as those described in part 1.

2.2.2 Plant material

The fruits of *Garcinia cowa* were collected from Sathingmoo district, amphur Singhanakhon, Songkhla province in the Southern part of Thailand. A voucher specimen has been deposited at the herbarium of the Department of Biology, Faculty of Science, Prince of Songkla University, Songkhla, Thailand.

2.2.3 Chemical investigation of the crude hexane extract of the fruits

Chemical investigation of the crude hexane extract was divided into two parts. The first investigation was a preliminary study and carried out in a small scale in order to separate and purify the major components. The second one aimed to isolate additional minor components, which could not be separated in the first investigation.

2.2.3.1 The first investigation

The fresh fruits (44.00 g) of *G. cowa* were sliced into small pieces and extracted with hexane (2.5 L) over the period of 3 days at room temperature for three times. After filtration, the filtrate was evaporated to dryness under reduced pressure to afford a crude hexane extract as a brown viscous liquid in 0.49 g. It was dissolved in acetone. The acetone-soluble part showed many UV-active spots on normal phase TLC with 20% ethyl acetate in hexane as a mobile phase (5 runs). Further separation by column chromatography over silica gel was performed. Elution was conducted initially with pure hexane, gradually enriched with ethyl acetate until pure ethyl acetate, followed by increasing amount of methanol until 70% methanol in ethyl acetate. Fractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford ten fractions, as shown in **Table 21**.

Table 21 Fractions obtained from the crude hexane extract (small scale) by column chromatography over silica gel

Fraction	Eluent	Weight (g)	Physical appearance
1	100% Hexane - 15% EtOAc/Hexane	0.0172	Orange solid
2	15% EtOAc /Hexane	0.0408	Yellow gum mixed with white solid
3	15% EtOAc /Hexane	0.0097	Yellow gum
4	15% EtOAc /Hexane	0.0323	Brown-orange gum
5	15-30% EtOAc /Hexane	0.0532	Yellow gum
6	30-40% EtOAc /Hexane	0.0100	Yellow gum

Table 21 (Continued)

Fraction	Eluent	Weight (g)	Physical appearance
7	40-50% EtOAc /Hexane	0.0129	Brown gum
8	50-60% EtOAc /Hexane	0.1729	Brown-red gum
9	60-80% EtOAc /Hexane	0.0612	Brown-yellow gum
10	100%EtOAc - 70% MeOH/ EtOAc	0.0525	Brown gum

Fraction 1 displayed no distinct spot on normal phase TLC under UV-S using 15% ethyl acetate in hexane as a mobile phase (3 runs). It was not investigated further.

Fraction 2 showed two major adjacent components on normal phase TLC under UV-S using 15% ethyl acetate in hexane as a mobile phase (3 runs) with the R_f values of 0.55 and 0.43. Its chromatogram on normal phase TLC using 30% dichloromethane in hexane as a mobile phase (6 runs) showed two UV-active components with the R_f values of 0.57 and 0.21. It was further purified by column chromatography over silica gel. Elution was performed with 100% hexane, followed by increasing the polarity with dichloromethane until 100% dichloromethane. Subfractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure, to give seven subfractions, as shown in **Table 22**.

Table 22 Subfractions obtained from **fraction 2** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
2-1	100% Hexane - 20% CH_2Cl_2/Hexane	0.0009	White solid
2-2	20-30% CH_2Cl_2/Hexane	0.0076	Yellow solid
2-3	30% CH_2Cl_2/Hexane	0.0049	Yellow solid
2-4	30% CH_2Cl_2/Hexane	0.0019	Orange solid
2-5	30% CH_2Cl_2/Hexane	0.0153	Yellow-red solid
2-6	30-70% CH_2Cl_2/Hexane	0.0037	Yellow gum
2-7	100% CH_2Cl_2	0.0068	Orange gum

Subfraction 2-1 contained many inseparable spots on normal phase TLC under UV-S using 30% dichloromethane in hexane as a mobile phase (5 runs). No further purification was attempted.

Subfraction 2-2 (**W1**) was obtained as a yellow solid (0.0076 g), melting at 100.5-101.2 °C. Its chromatogram showed a single UV-active spot on normal phase TLC with 30% dichloromethane in hexane as a mobile phase (5 runs) with the R_f value of 0.43.

UV(MeOH) λ_{max} nm (log ε)	245 (4.20), 261 (4.18), 314 (4.06), 358 (3.64)
FT-IR (KBr) $\nu_{cm^{-1}}$	3372 (OH stretching), 1646 (C=O stretching), 1598 (C=C aromatic ring)

^{1}H NMR (CDCl$_3$) (δppm) (300 MHz)		13.49 (*s*, 1H), 6.74 (*s*, 1H), 6.32 (*s*, 1H), 5.27 (*m*, 1H), 5.24 (*m*, 1H), 4.13 (*d, J* = 6.9 Hz, 2H), 3.96 (*s*, 3H), 3.90 (*s*, 3H), 3.80 (*s*, 3H), 3.35 (*d, J* = 7.2 Hz, 2H), 1.85 (*brs*, 3H), 1.80 (*brs*, 3H), 1.68 (*brs*, 6H)
^{13}C NMR (CDCl$_3$) (δppm) (75 MHz)		182.00, 163.61, 159.85, 158.04, 155.42, 155.21, 144.04, 137.33, 131.78, 130.87, 123.24, 122.32, 112.16, 111.55, 104.01, 98.20, 88.63, 60.95, 55.99, 55.80, 26.16, 25.91, 25.83, 21.57, 18.19, 17.79
DEPT (135°) (CDCl$_3$)	CH :	123.24, 122.32, 98.20, 88.63
	CH$_2$:	26.16, 21.57
	CH$_3$:	60.95, 55.99, 55.80, 25.91, 25.83, 18.19, 17.79

Subfraction 2-3 showed no definite spot on normal phase TLC under UV-S using 30% dichloromethane in hexane as a mobile phase (5 runs). Thus, it was not further investigated.

Subfraction 2-4 displayed one major yellow spot and many UV-active spots on normal phase TLC using 30% dichloromethane in hexane as a mobile phase (5 runs) with the R$_f$ value of the major spot of 0.13, which was identified as **W2**. No further separation was carried out.

Subfraction 2-5 (**W2**) was a yellow-red solid, melting at 63.2-64.0 °C. Its chromatogram showed only one UV-active spot on normal phase TLC with 30% dichloromethane in hexane as a mobile phase (5 runs) with the R$_f$ value of 0.13.

UV(MeOH) λ_{max} nm (log ε)		244 (4.35), 259 (4.28), 316 (4.17), 357 (3.73)
FT-IR (KBr) $\nu_{cm^{-1}}$		3410 (OH stretching), 1642 (C=O stretching), 1613, 1597 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)		13.86 (*s*, 1H), 6.44 (*brs*, 1H), 6.33 (*s*, 1H), 6.24 (*brs*, 1H), 5.28 (*m*, 3H), 4.08 (*d, J* = 6.3 Hz, 2H), 3.80 (*s*, 3H), 3.56 (*d, J* = 7.2 Hz, 2H), 3.45 (*d, J* = 7.2 Hz, 2H), 1.87 (*brs*, 3H), 1.85 (*brs*, 3H), 1.83 (*brs*, 3H), 1.77 (*brs*, 3H), 1.68 (*brs*, 6H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)		182.44, 161.53, 160.58, 155.06, 153.55, 152.33, 142.27, 135.70, 133.84, 132.66, 131.82, 123.53, 121.53, 121.15, 113.99, 111.94, 108.36, 103.59, 93.24, 62.00, 26.40, 25.86, 25.83, 25.79, 22.63, 21.45, 18.20, 17.95, 17.92
DEPT (135°) ($CDCl_3$)	CH :	123.53, 121.53, 121.15, 93.24
	CH_2:	26.40, 22.63, 21.45
	CH_3 :	62.00, 25.86, 25.83, 25.79, 18.20, 17.95, 17.92

Subfraction 2-6 showed many spots on normal phase TLC using 30% dichloromethane in hexane as a mobile phase (5 runs). No further purification was performed.

Subfraction 2-7 showed many inseparable spots on normal phase TLC using 30% dichloromethane in hexane as a mobile phase (5 runs). Therefore, it was not further investigated.

Fraction 3 demonstrated two overlapping UV-active spots on normal phase TLC using 15% dichloromethane in hexane as a mobile phase (3 runs) with the R_f values of

0.40 and 0.38. When using 10% acetone in hexane as a mobile phase (5 runs). The chromatogram displayed two spots on normal phase TLC under UV-S with the R_f values of 0.34 and 0.24. It was separated by precoated TLC on silica gel plates using 10% acetone in hexane as a mobile phase (7 runs) to give two bands.

Band 1 (**W8)** was an orange solid (0.0021 g), melting at 159.8-160.5 °C. It contained one spot on normal phase TLC using 10% acetone in hexane as a mobile phase (5 runs) with the R_f value of 0.34. Its ^1H NMR spectrum indicated the appearance of a pure compound. Because of low quantity, **W8** was additionally separated in the second investigation.

Band 2 was obtained as a yellow-orange solid (0.0017 g). Its chromatogram on normal phase TLC using 10% acetone in hexane as a mobile phase (5 runs) showed a single spot with the R_f value of 0.24, which was identified to be **W2** as a pure compound.

Fraction 4 contained many inseparable UV-active spots on normal phase TLC with 15% ethyl acetate in hexane as a mobile phase (3 runs). No attempted separation was carried out.

Fraction 5 showed one major yellow spot on normal phase TLC under UV-S with 15% ethyl acetate in hexane as a mobile phase (3 runs) with the R_f value of 0.24. It was further purified by column chromatography over silica gel. Elution was conducted initially with 100% hexane and gradually enrich with ethyl acetate until 100% ethyl acetate. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to give five subfractions, as shown in **Table 23**.

Table 23 Subfractions obtained from **fraction 5** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
5-1	100% Hexane - 5% EtOAc/Hexane	0.0041	White solid
5-2	5-20% EtOAc/Hexane	0.0131	Yellow solid
5-3	20-40% EtOAc/Hexane	0.002	Yellow solid
5-4	40-80% EtOAc/Hexane	0.0093	Yellow gum
5-5	100% EtOAc	0.0103	Yellow gum

Subfraction 5-1 showed no definite spot on normal phase TLC under UV-S using 30% ethyl acetate in hexane as a mobile phase. Thus, it was not further investigated.

Subfraction 5-2, upon standing at room temperature, afforded a yellow solid (**W3**, 0.0097 g), melting at 175.8-176.4 °C. Its chromatogram showed only one spot on normal phase TLC under UV-S with the R_f value of 0.65 using 30% ethyl acetate in hexane as a mobile phase. According to its ^{1}H NMR spectral data, it was assigned to be β-mangostin. The filtrate contained many pale UV-active spots on normal phase TLC. No further purification was performed.

UV(MeOH) λ_{max} nm (log ε)	244 (4.15), 259 (4.07), 315 (3.97), 358 (3.52)
FT-IR (KBr) ν_{cm-1}	3402 (OH stretching), 1646 (C=O stretching), 1594 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)	13.42 (*s*, 1H), 6.82 (*s*, 1H), 6.37 (*brs*, 1H), 6.33 (*s*, 1H), 5.26 (*mt*, J = 6.3 Hz, 1H), 5.23 (*mt*, J = 6.9 Hz, 1H), 4.09 (*d*, J = 6.3 Hz, 2H), 3.90 (*s*, 3H), 3.81 (*s*, 3H), 3.35 (*d*, J = 7.2 Hz, 2H), 1.83 (*brs*, 3H), 1.80 (*brs*, 3H), 1.69 (*brs*, 3H), 1.68 (*brs*, 3H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)	181.94, 163.53, 159.78, 155.72, 155.24, 154.40, 142.58, 137.05, 132.08, 131.68, 123.21, 122.33, 112.42, 111.53, 103.83, 101.46, 88.81, 62.06, 55.83, 26.55, 25.81 (Cx2), 21.36, 18.22, 17.77
DEPT (135°) ($CDCl_3$)	CH : 123.21, 122.33, 101.46, 88.81 CH_2 : 26.55, 21.36 CH_3 : 62.06, 55.83, 25.81 (Cx2), 18.22, 17.77

Subfraction 5-3 contained many inseparable UV-active spots on normal phase TLC with 30% ethyl acetate in hexane as a mobile phase. No attempted separation was carried out.

Subfraction 5-4 appeared many UV-active spots using 30% ethyl acetate in hexane as a mobile phase. Because of low quantity, It was not further separated.

Subfraction 5-5 showed no UV-active spot on normal phase TLC using 30% ethyl acetate in hexane as a mobile phase. Therefore, it was not further investigated.

Fraction 6 showed one major orange spot on normal phase TLC under UV-S with 15% ethyl acetate in hexane as a mobile phase (3 runs) with the R_f value of 0.17. Further chromatographic separation by preparative TLC on silica gel plates with 10% ethyl acetate in hexane as a mobile phase (8 runs) afforded two bands.

Band 1 was obtained as a yellow gum (0.0010 g). Its chromatogram showed one major yellow spot on normal phase TLC under UV-S with the R_f value of 0.33 using 10% ethyl acetate in hexane as a mobile phase (5 runs). Its ^{1}H NMR spectrum indicated that it contained some impurities. Because of low quantity, it was not further purified.

Band 2 (**W4**) was obtained as an orange gum (0.0044 g). Its chromatogram appeared one major spot on normal phase TLC under UV-S with the R_f value of 0.25 using 10% ethyl acetate in hexane as a mobile phase (5 runs).

UV(MeOH) λ_{max} nm (log ε)	244 (4.33), 258 (4.26), 315 (4.15), 355 (3.71)
FT-IR (neat) ν_{cm-1}	3365 (OH stretching), 1639 (C=O stretching), 1606 (C=C aromatic ring)
^{1}H NMR (CDCl$_3$) (δppm) (300 MHz)	13.84 (*s*, 1H), 6.75 (*s*, 1H), 6.29 (*s*, 1H), 6.25 (*brs*, 1H), 5.30 (*mt*, *J* = 7.2 Hz, 1H), 5.25 (*mt*, *J* = 6.3 Hz, 1H), 4.13 (*d, J* = 6.3 Hz, 2H), 3.96 (*s*, 3H), 3.80 (*s*, 3H), 3.45 (*d, J* = 6.9 Hz, 2H), 1.85 (*s*, 6H), 1.77 (*s*, 3H), 1.68 (*s*, 3H)
^{13}C NMR (CDCl$_3$) (δppm) (75 MHz)	182.10, 161.57, 160.63, 158.14, 155.46, 155.04, 144.20, 137.29, 135.78, 131.89, 123.16, 121.48, 111.89, 108.45, 103.75, 98.31, 93.16, 60.69, 56.04, 26.18, 25.92, 25.87, 21.46, 18.19, 17.94

DEPT (135°) ($CDCl_3$) CH : 123.16, 121.48, 98.31, 93.16
CH_2 : 26.18, 21.46
CH_3 : 60.96, 56.04, 25.92, 25.87, 18.19, 17.94
EIMS *m/z* (% relative intensity): 424 (([M$^+$], 30), 423 (94), 380 (100), 368 (41), 352 (86), 336 (40), 324 (40)
HREI-MS: *m/z* found 424.1899 ([M$^+$], calcd. for $C_{25}H_{28}O_6$, 424.1886)

Fraction 7 showed two overlapping UV-active spots on normal phase TLC using 15% ethyl acetate in hexane as a mobile phase (3 runs). When using 50% dichloromethane in hexane as a mobile phase (4 runs), the chromatogram showed three UV-active spots on normal phase TLC with the R_f values of 0.44, 0.35 and 0.23. Further purification by precoated TLC on silica gel plates with 60% dichloromethane in hexane as a mobile phase (2 runs) afforded three bands.

Band 1 was an orange gum (0.0031 g). It was shown to be **W4** as a major component on normal phase TLC with the R_f value of 0.51 using 60% dichloromethane in hexane as a mobile phase (5 runs) and ^{1}H NMR spectral data was identical to those of **W4**.

Band 2 (**W6**) was obtained as a pale yellow solid (0.0023 g). Its chromatogram displayed one major spot on normal phase TLC under UV-S with the R_f value of 0.39 using 60% dichloromethane in hexane as a mobile phase (5 runs). Its ^{1}H NMR spectrum indicated the presence of a pure compound. Because of low quantity, this compound (**W6**) was additionally separated in the second investigation.

Band 3 (**W7**) was obtained as an orange solid (0.0013 g). Its chromatogram showed one UV-active spot on normal phase TLC with the R_f value of 0.27 using 60%

dichloromethane in hexane as a mobile phase (5 runs). Its ^1H NMR spectrum suggested that it was a pure compound. Because it was obtained in low quantity, further investigation was performed in next part.

<u>Fraction 8</u> contained many inseparable UV-active spots on normal phase TLC with 20% ethyl acetate in hexane (8 runs) and 30% ethyl acetate in hexane (4 runs) as mobile phases. No further purification was carried out.

<u>Fraction 9</u> showed one major brown spot on normal phase TLC under UV-S using 20% ethyl acetate in hexane (8 runs) and 30% ethyl acetate in hexane (4 runs) as mobile phases with the R_f value of 0.33. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure hexane, gradually enriched with ethyl acetate until pure ethyl acetate, followed by increasing amount of methanol until 50% methanol in ethyl acetate. Subfractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure to give four subfractions, as shown in **Table 24**.

Table 24 Subfractions obtained from **fraction 9** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
9-1	100% Hexane - 30% EtOAc/Hexane	0.0066	Yellow gum
9-2	30% EtOAc/Hexane	0.0129	Yellow gum

Table 24 (Continued)

Subfraction	Eluent	Weight (g)	Physical appearance
9-3	30% EtOAc/Hexane	0.0218	Brown-yellow solid
9-4	30-50% EtOAc/Hexane - 50% MeOH/EtOAc	0.0175	Brown-yellow gum

Subfraction 9-1 appeared many pale inseparable UV-active spots using 30% ethyl acetate in hexane as a mobile phase (4 runs). Because of low quantity, It was not further investigated.

Subfraction 9-2 displayed one major brown spot with the R_f value of 0.30, which was shown to be identical to **W5,** and many pale UV-active spots on normal phase TLC using 30% ethyl acetate in hexane as a mobile phase (4 runs). Further separation was not performed.

Subfraction 9-3 (**W5**) was obtained as a brown-yellow solid (0.0218 g), melting at 122.2-124.0 °C. Its chromatogram showed only one UV-active spot on normal phase TLC using 30% ethyl acetate in hexane (4 runs) as a mobile phase with the R_f value of 0.30.

UV(MeOH) λ_{max} nm (log ε)	243 (4.48), 258 (4.37), 316 (4.28), 357 (3.88)
FT-IR (KBr) ν_{cm-1}	3387 (OH stretching), 1642 (C=O stretching), 1606 (aromatic ring)

^{1}H NMR (CDCl$_3$) (δ ppm) (300 MHz)	13.88 (*s*, 1H), 6.72 (*s*, 1H), 6.23 (*s*, 1H), 5.48 (*brt*, J = 8.1 Hz, 1H), 5.24 (*brt*, J = 6.0 Hz, 1H), 5.01 (*brt*, J = 6.6 Hz, 1H), 4.35 (*s*, 2H), 4.03 (*d, J* = 6.0 Hz, 2H), 3.79 (*s*, 3H), 3.46 (*d, J* = 6.9 Hz, 2H), 2.02 (*m*, 4H), 1.83 (*s*, 3H), 1.81 (*s*, 3H), 1.58 (*s*, 3H), 1.53 (*s*, 3H)
^{13}C NMR (CDCl$_3$) (δ ppm) (75 MHz)	181.82, 161.41, 160.54, 155.60, 154.94, 154.67, 142.66, 137.12, 135.47, 133.34, 131.26, 126.98, 124.31, 123.32, 111.97, 108.37, 103.29, 101.66, 93.49, 62.51, 61.87, 39.70, 26.57, 26.44, 25.59, 22.75, 21.49, 17.64, 16.45
DEPT (135°) (CDCl$_3$)	CH : 126.98, 124.31, 123.32, 101.66, 93.49
	CH$_2$: 62.51, 39.70, 26.57, 26.44, 21.49
	CH$_3$: 61.87, 25.59, 22.75, 17.64, 16.45

Subfraction 9-4 contained many inseparable UV-active spots using 30% ethyl acetate in hexane as a mobile phase (4 runs). No further purification was performed.

Fraction 10 showed no UV-active spot on normal phase using 30% ethyl acetate in hexane as a mobile phase (4 runs). No further investigation was attempted.

2.2.3.2 The second investigation

The fresh fruits (2,300 g) of *G. cowa* were sliced into small pieces and extracted with hexane (15 L) over the period of 30 days at room temperature for six times. After filtration, the filtrate was evaporated to dryness under reduced pressure to afford a crude hexane extract as a brown viscous liquid in 25.20 g. The crude hexane extract was tested for its solubility in various solvents at room temperature. The results were demonstrated in **Table 25**.

Table 25 Solubility of the crude hexane extract in various solvents at room temperature

Solvent	**Solubility**	**Physical appearance**
Petroleum ether	++++	Brown-yellow solution
$CHCl_3$	++++	Brown-yellow solution
EtOAc	+++	Dark yellow solution
MeOH	++	Dark brown solution
H_2O	+	Yellow solution
10% HCl	+	Brown solid in brown solution
10% NaOH	+++	Black solid in brown solution
10% $NaHCO_3$	++	Black solid in brown solution

Symbol meaning + partially soluble ++ moderately soluble +++ well – soluble ++++ very well – soluble

It was concluded, based on the solubility results, that the major components in the crude hexane extract was soluble in non-polar solvents. The crude extract (25.20 g) was separated by quick column chromatography over silica gel using pure hexane, ethyl acetate-hexane gradient, pure ethyl acetate, methanol-ethyl acetate gradient and finally with pure methanol. All fractions were examined by TLC, combined on the basis of their chromatography characteristics and then evaporated to dryness under reduced pressure to give seven fractions, as shown in **Table 26**.

Table 26 Fractions obtained from the crude hexane extract by quick column chromatography over silica gel

Fraction	Eluent	Weight (g)	Physical appearance
A	100% Hexane - 5% EtOAc/Hexane	1.3312	Orange-yellow viscous liquid
B	5% EtOAc/Hexane	2.5515	Orange-brown viscous liquid
C	5-20% EtOAc/Hexane	6.0440	Red-brown viscous liquid
D	30% EtOAc/Hexane	1.0400	Red-brown viscous liquid
E	30-50% EtOAc/Hexane	0.7049	Red-brown viscous liquid
F	70% EtOAc/Hexane-50% MeOH/EtOAc	3.0508	Dark-brown viscous liquid
G	70% MeOH/EtOAc-100% MeOH	1.6156	Dark-brown viscous liquid

Fraction A showed two spots under UV-S on normal phase TLC with 10% ethyl acetate in hexane as a mobile phase (4 runs) with the R_f values of 0.78 and 0.61, respectively. It was separated by column chromatography over silica gel with 100%

hexane and increasing the polarity with ethyl acetate until 100% ethyl acetate. Fractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure to afford three fractions, as shown in **Table 27**.

Table 27 Fractions obtained from **fraction A** by column chromatography over silica gel

Fraction	**Eluent**	**Weight (g)**	**Physical appearance**
A1	100% Hexane-5% EtOAc/Hexane	0.5752	Yellow viscous liquid
A2	5-7% EtOAc/Hexane	0.1821	Yellow-brown viscous liquid
A3	10% EtOAc/Hexane-100% EtOAc	0.3695	Brown viscous liquid

Fraction A1 showed no major UV-active spot on normal phase TLC with 5% ethyl acetate in hexane as a mobile phase (5 runs). It was not further investigated.

Fraction A2 displayed two major spots under UV-S on normal phase TLC using 5% ethyl acetate in hexane as a mobile phase (5 runs) with the R_f values of 0.48 and 0.38. Further purification by preparative TLC on silica gel plates with 5% ethyl acetate in hexane as a mobile phase (3 runs) gave a yellow-orange viscous liquid in 116.4 mg. Its chromatogram showed two spots on normal phase TLC, which were the same as those before separation. When using 5% acetone in hexane as a mobile phase (3 runs), the chromatogram showed two major spots under UV-S on normal phase TLC with the R_f values of 0.58 and 0.45. It was separated by column chromatography with pure hexane, gradually enriched with acetone and finally with pure acetone. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in **Table 28**.

Table 28 Subfractions obtained from **fraction A2** after purification by preparative TLC followed by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
A2b-1	100% Hexane - 2% Acetone/Hexane	0.0009	White solid
A2b-2	2% Acetone/Hexane	0.0089	Yellow solid
A2b-3	2% Acetone/Hexane	0.0327	Yellow gum mixed with yellow solid
A2b-4	5-30% Acetone/Hexane	0.0620	Yellow-red gum
A2b-5	50% Acetone/Hexane - 100% Acetone	0.0065	Yellow gum

Subfraction A2b-1 which contained no major UV-active spot on normal phase TLC with 2% acetone in hexane as a mobile phase (3 runs). It was not further investigated.

Subfraction A2b-2 (W9) was obtained as a yellow solid (0.0089 g), melting at 177.5-178.2 °C. Its chromatogram displayed a single spot under UV-S on normal phase TLC using 2% acetone in hexane as a mobile phase (3 runs) with the R_f value of 0.30.

UV(MeOH) λ_{max} nm (log ε)		240 (4.22), 279 (4.44), 289 (4.48), 330 (4.05)
FT-IR (KBr) $\nu_{cm^{-1}}$		3320 (OH stretching), 1639 (C=O stretching), 1604 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)		13.77 (*s*, 1H), 6.76 (*s*, 1H), 6.72 (*d, J* = 9.9 Hz, 1H), 6.24 (*s*, 1H), 5.57 (*d, J* = 9.9 Hz, 1H), 5.24 (*brt*, *J* = 6.9 Hz, 1H), 4.12 (*d, J* = 6.3 Hz, 2H), 3.96 (*s*, 3H), 3.80 (*s*, 3H), 1.85 (*s*, 3H), 1.68 (*s*, 3H), 1.47 (*s*, 6H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)		182.07, 159.71, 158.17, 157.99, 156.25, 155.41, 144.08, 137.24, 131.92, 127.10, 123.13, 115.76, 111.91, 104.50, 103.91, 98.40, 93.96, 77.94, 60.95, 56.06, 28.32 (Cx2), 26.16, 25.92, 18.19
DEPT (135°) ($CDCl_3$)	CH :	127.10, 123.13, 115.76, 98.40, 93.96
	CH_2 :	26.16
	CH_3 :	60.95, 56.06, 28.32 (Cx2), 25.92, 18.19

Subfraction A2b-3 showed one major spot under UV-S on normal phase TLC using 2% acetone in hexane as a mobile phase (3 runs) with the R_f value of 0.30 indicated the presence of **W9** as a major component.

Subfraction A2b-4 (W10) displayed a single spot under UV-S on normal phase TLC using 2% acetone in hexane as a mobile phase (3 runs) with the R_f value of 0.18.

UV(MeOH) λ_{max} nm (log ε)		243 (3.95), 279 (4.16), 289 (4.19), 332 (3.90)
FT-IR (neat) ν_{cm-1}		3348 (OH stretching), 1648 (C=O stretching), 1609, 1595 (C=C aromatic ring)
^{1}H NMR (CDCl$_3$) (δ ppm) (300 MHz)		13.77 (*s*, 1H), 6.73 (*d, J* = 9.9 Hz, 1H), 6.50 (*brs*, 1H), 6.28 (*s*, 1H), 5.56 (*d, J* = 9.9 Hz, 1H), 5.25 (*m*, 2H), 4.05 (*d, J* = 6.3 Hz, 2H), 3.80 (*s*, 3H), 3.55 (*d, J* = 7.2 Hz, 2H), 1.87 (*s*, 3H), 1.82 (*s*, 3H), 1.69 (*s*, 6H), 1.47 (*s*, 6H)
^{13}C NMR (CDCl$_3$) (δ ppm) (75 MHz)		182.37, 159.72, 157.90, 156.24, 153.43, 152.40, 142.35, 133.79, 132.67, 131.87, 127.02, 123.50, 121.09, 115.77, 114.11, 111.88, 104.39, 103.68, 94.07, 77.91, 62.00, 28.33 (Cx2), 26.37, 25.83, 25.79, 22.64, 18.21, 18.00
DEPT (135°) (CDCl$_3$)	CH :	127.02, 123.50, 121.09, 115.77, 94.07
	CH$_2$:	26.37, 22.64
	CH$_3$:	62.00, 28.33 (Cx2), 25.83, 25.79, 18.21, 18.00
EIMS *m/z* (% relative intensity):		476 (([M$^+$], 19), 475 (48), 460 (100), 432 (87)
HREI-MS: *m/z* found		476.2191 ([M$^+$], calcd. for $C_{29}H_{32}O_6$, 476.2199)

Subfraction A2b-5 which contained no major UV-active spot on normal phase TLC with 2% acetone in hexane as a mobile phase (3 runs). It was not further investigated.

Fraction A3 showed many inseparable UV-active spot on normal phase TLC using 10% ethyl acetate in hexane as a mobile phase (4 runs). No attempted investigation was performed.

Fraction B displayed three major spots under UV-S on normal phase TLC with the R_f values of 0.50, 0.45 and 0.30 and showed one minor spot with the R_f value of 0.61 using 10% ethyl acetate in hexane as a mobile phase (4 runs). It was separated by column chromatography over silica gel. Elution was conducted initially with pure hexane, gradually enriched with ethyl acetate until pure ethyl acetate, followed by increasing amount of methanol until pure methanol. Fractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to give six fractions, as shown in **Table 29**.

Table 29 Fractions obtained from **fraction B** by column chromatography over silica gel

Fraction	**Eluent**	**Weight (g)**	**Physical appearance**
B1	100% Hexane - 4% EtOAc/Hexane	0.0477	Pale orange solid
B2	4-8% EtOAc/Hexane	0.0969	Yellow gum
B3	8% EtOAc/Hexane	0.1172	Brown-yellow gum mixed with yellow solid
B4	8% EtOAc/Hexane	1.0360	Brown-yellow gum mixed with yellow solid
B5	8-20% EtOAc/Hexane	0.8352	Yellow solid
B6	40% EtOAc/Hexane - 100% MeOH	0.1454	Brown gum

Fraction B1 contained many pale inseparable UV-active spots on normal phase TLC with 5% ethyl acetate in hexane as a mobile phase (4 runs). Further purification was therefore not attempted.

Fraction B2, upon standing at room temperature, afforded a yellow solid (0.0257 g). Its chromatogram showed one UV-active spot on normal phase TLC with the R_f value of 0.38 using 5% ethyl acetate in hexane as a mobile phase (4 runs), which indicated the presence of **W1** as a pure component. In addition, the filtrate showed no UV-active spot. Therefore, it was not further investigated.

Fractions B3 showed no major UV-active spot on normal phase TLC with 5% ethyl acetate in hexane as a mobile phase (4 runs). No attempted investigation was performed.

Fraction B4 showed one UV-active spot on normal phase TLC with 5% ethyl acetate in hexane as a mobile phase (4 runs). When using 10% acetone in hexane as a mobile phase (3 runs), the chromatogram displayed two UV-active components on normal phase TLC with the R_f values of 0.25 and 0.15. Further separation by column chromatography over silica gel using 100% hexane, acetone-hexane gradient, and finally with 100% acetone. All subfractions were examined by TLC, combined on the basis of their chromatography characteristics and then evaporated to dryness under reduced pressure to afford six subfractions, as shown in **Table 30**.

Table 30 Subfractions obtained from **fraction B4** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
B4-1	100% Hexane - 2% Acetone/Hexane	0.0712	Brown viscous liquid
B4-2	5-10% Acetone/Hexane	0.3775	Brown-yellow solid
B4-3	10% acetone/Hexane	0.0688	Brown viscous liquid
B4-4	10-15% Acetone/Hexane	0.4009	Yellow-orange solid
B4-5	15-50% Acetone/Hexane	0.0421	Brown viscous liquid
B4-6	50% Acetone/Hexane - 100% Acetone	0.2652	Brown viscous liquid

Subfraction B4-1 displayed no distinct UV-active component on normal phase TLC with 5% acetone in hexane (2 runs) and 10% acetone in hexane (4 runs) as mobile phases. Thus, further separation was not performed.

Subfraction B4-2 (W8) was obtained as a brown-yellow solid (0.3775 g), melting at 159.8-160.5 °C. Its chromatogram contained a single spot under UV-S on normal phase TLC using 5% acetone in hexane (2 runs) and 10% acetone in hexane (4 runs) as mobile phases with the R_f value of 0.34.

UV(MeOH) $\lambda_{\max}$ nm (log ε)		240 (4.23), 278 (4.52), 289 (4.57), 330 (4.22)
FT-IR (KBr) $\nu_{\text{cm-1}}$		3341 (OH stretching), 1642 (C=O stretching), 1600 (C=C aromatic ring)
^{1}H NMR (CDCl$_3$) (δppm) (300 MHz)		13.70 (*s*, 1H), 6.87 (*s*, 1H), 6.71 (*d, J* = 9.9 Hz, 1H), 6.63 (*brs*, 1H), 6.22 (*s*, 1H), 5.56 (*d, J* = 9.9 Hz, 1H), 5.26 (*brt*, *J* = 6.3 Hz, 1H), 4.06 (*d, J* = 6.0 Hz, 2H), 3.80 (*s*, 3H), 1.83 (*s*, 3H), 1.69 (*s*, 3H), 1.46 (*s*, 6H)
^{13}C NMR (CDCl$_3$) (δppm) (75 MHz)		181.95, 159.83, 157.86, 156.22, 155.67, 154.69, 142.70, 137.00, 132.12, 127.13, 123.17, 115.70, 112.08, 104.45, 103.68, 101.74, 94.16, 77.94, 61.97, 28.31 (Cx2), 26.52, 25.83, 18.23
DEPT (135°) (CDCl$_3$)	CH :	127.13, 123.17, 115.70, 101.74, 94.16,
	CH$_2$:	26.52
	CH$_3$:	61.97, 28.31 (Cx2), 25.83, 18.23

Subfraction B4-3 showed two major UV-active spots on normal phase TLC using 5% acetone in hexane (2 runs) and 10% acetone in hexane (4 runs) as mobile phases with the R$_f$ values of 0.34 and 0.20, which were identified to be **W13** and **W2**, respectively. Therefore, this subfraction was not conducted.

Subfraction B4-4 appeared **W2** as a major component on normal phase TLC using 5% acetone in hexane (2 runs) and 10% acetone in hexane (4 runs) as mobile phases with the R$_f$ value of 0.20. Thus, it was not investigated.

Subfraction B4-5 its chromatogram showed one yellow spot on normal phase TLC using 5% acetone in hexane (2 runs) and 10% acetone in hexane (4 runs) as mobile phases with the R_f value of 0.20, which indicated the appearance of **W2** as a major compound, and further showed many inseparable spots below **W2**. Hence, this subfraction was not purified.

Subfraction B4-6 displayed no definite spot under UV-active on normal phase TLC using 30% acetone in hexane as a mobile phase. No further separation was conducted.

Fraction B5, upon standing at room temperature, to give a yellow solid (0.1030 g). Its chromatogram on normal phase TLC using 10% ethyl acetate in hexane (2 runs) and 20% ethyl acetate in hexane as mobile phases showed one UV-active spot with the R_f value of 0.38 which was assigned as **W3**. In addition, the filtrate showed no spot under UV-S. Therefore, it was not further investigated.

Fraction B6 showed no UV-active spot on normal phase TLC using 10% ethyl acetate in hexane (2 runs) and 20% ethyl acetate in hexane as mobile phases. No attempted investigation was performed.

Fraction C contained three major UV-active components on normal phase TLC with the R_f values of 0.27, 0.16 and 0.05 using 10% ethyl acetate in hexane (4 runs) as a mobile phase. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure hexane, gradually enriched with ethyl acetate until pure ethyl acetate, followed by increasing amount of methanol until pure methanol. Fractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure to give eight fractions, as shown in **Table 31**.

Table 31 Fractions obtained from **fraction C** by column chromatography over silica gel

Fraction	Eluent	Weight (g)	Physical appearance
C1	100% Hexane - 5% EtOAc/Hexane	0.0378	Yellow gum mixed with yellow solid
C2	5-10% EtOAc /Hexane	0.0478	Brown gum mixed with brown solid
C3	10-15% EtOAc/Hexane	0.0305	Brown gum mixed with brown solid
C4	15% EtOAc/Hexane	0.1910	Brown-red gum
C5	15% EtOAc/Hexane	0.7623	Brown-red gum mixed with brown solid
C6	15% EtOAc/Hexane	0.2483	Brown-yellow gum
C7	20-50% EtOAc/Hexane	3.1469	Brown gum
C8	70% EtOAc/Hexane-100% MeOH	1.4537	Brown gum mixed with brown solid

Fraction C1 contained no distinct UV-active spot on normal phase TLC with 6% ethyl acetate in hexane as a mobile phase (4 runs). Thus, further purification was not performed.

Fraction C2 displayed many UV-active components on normal phase TLC with 6% ethyl acetate in hexane as a mobile phase (4 runs). Because it was obtained in low quantity, further investigation was then not conducted.

Fraction C3 contained many inseparable spots under UV-S on normal phase TLC with 6% ethyl acetate in hexane as a mobile phase (4 runs). Therefore, it was not further investigated.

Fraction C4 showed three major UV-active components with the R_f values of 0.63, 0.58 and 0.43, respectively on normal phase TLC using 20% ethyl acetate in hexane (4 runs) as a mobile phase. This fraction was not further separated because of limitation of time.

Fraction C5 appeared two major UV-active spots on normal phase TLC using 20% ethyl acetate in hexane (4 runs) as a mobile phase with the R_f values of 0.40 and 0.25, which were assigned to be **W4** and **W11**, respectively. Therefore, this fraction was not separated.

Fraction C6 displayed three yellow spots under UV-S on normal phase TLC using 20% ethyl acetate in hexane (4 runs) as a mobile phase with the R_f values of 0.35, 0.23 and 0.13. Further chromatographic separation by column chromatography over silica gel. Elution was performed initially with pure hexane, gradually enriched with ethyl acetate until pure ethyl acetate, followed by increasing methanol up to pure methanol. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in **Table 32**.

Table 32 Subfractions obtained from **fraction C6** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
C6-1	100% Hexane - 5% EtOAc/Hexane	0.0058	Yellow solid
C6-2	5-20% EtOAc /Hexane	0.0049	Brown-orange gum
C6-3	10-20% EtOAc/Hexane	0.0137	Brown-orange gum mixed with orange solid
C6-4	20% EtOAc/Hexane	0.0927	Brown-orange gum
C6-5	20-50% EtOAc/Hexane	0.0366	Yellow gum
C6-6	70% EtOAc/Hexane-100% MeOH	0.1738	Brown gum

Subfraction C6-1 displayed one major spot with the R_f value of 0.74 under UV-active on normal phase TLC using 20% ethyl acetate in hexane (2 runs) as a mobile phase. This result indicated the presence of **W9.** Thus, further separation was not performed.

Subfraction C6-2 contained many inseparable spots under UV-S on normal phase TLC with 20% ethyl acetate in hexane (2 runs) as a mobile phase. Therefore, further separation was not conducted.

Subfraction C6-3 showed one UV-active spot on normal phase TLC using 20% ethyl acetate in hexane (2 runs) as a mobile phase with the R_f value of 0.35 which was identified as **W3**. Therefore, it was not further investigated.

Subfraction C6-4 appeared one yellow UV-active spot on normal phase TLC using 10% ethyl acetate in hexane (4 runs) as a mobile phase with the R_f value of 0.26 indicated the presence of **W4** as a major compound.

Subfraction C6-5 showed one yellow spot under UV-S on normal phase TLC with 10% ethyl acetate in hexane as a mobile phase (4 runs) with the R_f value of 0.06. Further purification by preparative TLC on silica gel plates with 10% ethyl acetate in hexane (8 runs) as a mobile phase afforded four bands.

Band 1 (**W12**) was obtained as a yellow solid (0.0124 g), melting at 136.3-137.0 °C. Its chromatogram showed one major yellow spot on normal phase TLC under UV-S with the R_f value of 0.06 using 10% ethyl acetate in hexane as a mobile phase (4 runs).

UV(MeOH) λ_{max} nm (log ε)	243 (4.77), 257 (4.66), 317 (4.57), 358 (4.12)
FT-IR (KBr) ν_{cm-1}	3365 (OH stretching), 1639 (C=O stretching), 1606 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz)	13.78 (*s*, 1H), 6.83 (*s*, 1H), 6.53 (*brs*, 2H), 6.29 (*s*, 1H), 5.29 (*mt*, *J* = 7.5 Hz, 1H), 5.26 (*mt*, *J* = 6.3 Hz, 1H), 5.02 (*mt*, *J* = 6.6 Hz, 1H), 4.08 (*d, J* = 6.0 Hz, 2H), 3.80 (*s*, 3H), 3.43 (*d, J* = 6.9 Hz,

		2H), 2.02 (*m*, 4H), 1.87 (*s*, 3H), 1.83 (*s*, 3H), 1.76 (*s*, 3H), 1.59 (*s*, 3H), 1.54 (*s*, 3H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)		181.96, 161.59, 160.61, 155.71, 155.00, 154.00, 142.63, 137.15, 135.52, 135.25, 131.27, 124.29, 123.27, 121.59, 112.18, 108.67, 103.56, 101.58, 93.22, 61.97, 39.70, 26.56, 26.48, 25.83, 25.60, 21.44, 17.90, 17.66, 16.47
DEPT (135°) ($CDCl_3$)	CH :	124.29, 123.27, 121.59, 101.58, 93.22
	CH_2 :	39.70, 26.56, 26.48, 21.44
	CH_3 :	61.97, 25.83, 25.60, 17.90, 17.66, 16.47

Band 2 was obtained as a yellow gum (0.0014 g) that contained no definite spot under UV-S on normal phase TLC using 20% ethyl acetate in hexane as a mobile phase (6 runs). Therefore, it was not further investigated.

Band 3 was a yellow gum (0.0012 g) which showed a single UV-active spot on normal phase TLC using 20% ethyl acetate in hexane (6 runs) as a mobile phase with the R_f value of 0.23. Its ^{1}H NMR spectrum indicated that it contained some impurities. Because of low quantity, this band was not further purified.

Band 4 was a yellow gum (0.0013 g). Its chromatogram showed one UV-active spot on normal phase TLC using 20% ethyl acetate in hexane (6 runs) as a mobile phase with the R_f value of 0.13. Its ^{1}H NMR spectral data indicated that it contained some impurities. Because it was obtained in low quantity, further investigation was not performed.

Subfraction C6-6 showed no definite spot on normal phase TLC under UV-S using 20% ethyl acetate in hexane as a mobile phase (2 runs). No further purification was conducted.

Fraction C7 contained one major UV-active component on normal phase TLC with the R_f value of 0.08 using 100% chloroform (2 runs) as a mobile phase. It was further separated by column chromatography over silica gel. Elution was conducted initially with 100% chloroform, gradually enriched with methanol and finally with 100% methanol. Subfractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure to give four subfractions, as shown in **Table 33**.

Table 33 Subfractions obtained from **fraction C7** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
C7-1	100% $CHCl_3$	0.0002	White solid
C7-2	2% MeOH/$CHCl_3$	0.0422	Yellow viscous liquid
C7-3	2-4% MeOH/$CHCl_3$	2.9125	Brown viscous liquid
C7-4	20% MeOH/$CHCl_3$- 100% MeOH	0.3732	Orange viscous liquid

Subfraction C7-1 contained no distinct UV-active spot on normal phase TLC with 10% dichloromethane in hexane as a mobile phase (4 runs). Thus, further purification was not performed.

Subfraction C7-2 displayed three yellow spots under UV-S on normal phase TLC using 20% dichloromethane in hexane, 30% dichloromethane in hexane and 40% dichloromethane in hexane (4 runs) as mobile phases with the R_f values of 0.47, 0.21 and 0.08. Further separation by column chromatography over silica gel. Elution was performed initially with 100% hexane, gradually enriched with dichloromethane until 70% dichloromethane. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in **Table 34**.

Table 34 Subfractions obtained from **subfraction C7-2** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
C7-21	100% Hexane - 20% CH_2Cl_2/Hexane	0.0123	Yellow solid
C7-22	20% CH_2Cl_2/Hexane	0.0108	Yellow solid
C7-23	30% CH_2Cl_2/Hexane	0.0190	Brown-orange gum
C7-24	30-40% CH_2Cl_2/Hexane	0.0334	Orange gum
C7-25	40-50% CH_2Cl_2/Hexane	0.0319	Brown-yellow gum
C7-26	60-70% CH_2Cl_2/Hexane	0.0178	Orange gum

Subfraction C7-21 showed no UV-active spot on normal phase TLC using 40% dichloromethane in hexane (7 runs) as a mobile phase. No attempted investigation was carried out.

Subfraction C7-22, upon standing at room temperature, afforded a yellow solid (**W11**, 0.0081 g), melting at 210.0-210.7 °C. Its chromatogram showed a single spot on normal phase TLC under UV-S with the R_f value of 0.64 using 40% dichloromethane in hexane (7 runs) as a mobile phase. The filtrate contained no UV-active spot on normal phase TLC. Thus, further purification was not performed.

UV(MeOH) λ_{max} nm (log ε)		245 (4.44), 265 (4.42), 328 (4.30), 387 (3.81)
FT-IR (KBr) ν_{cm-1}		3266 (OH stretching), 1631 (C=O stretching), 1596 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)		13.34 (*s*, 1H), 8.03 (*d, J* = 10.2 Hz, 1H), 6.82 (*s*, 1H), 6.36 (*s*, 1H), 6.20 (*brs*, 1H), 5.82 (*d, J* = 10.2 Hz, 1H), 5.23 (*m*, 1H), 3.90 (*s*, 3H), 3.35 (*d, J* = 7.2 Hz, 2H), 1.80 (*s*, 3H), 1.68 (*s*, 3H), 1.50 (*s*, 6H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)		182.41, 163.62, 159.62, 155.45, 153.05, 150.76, 136.77, 132.20, 131.71, 122.31, 121.03, 119.72, 111.49, 108.79, 104.00, 102.21, 88.94, 77.21, 55.85, 27.35 (Cx2), 25.81, 21.35, 17.78
DEPT (135°) ($CDCl_3$)	CH :	132.20, 122.31, 121.03, 102.21, 88.94
	CH_2 :	21.35
	CH_3 :	55.85, 27.35 (Cx2), 25.81, 17.78
EIMS *m/z* (% relative intensity):		408 ([M^+], 15), 407 (46), 392 (47), 364 (60), 352 (100), 334 (16), 168 (21), 166 (25), 148 (86), 125 (21), 111 (32), 97 (51), 83 (65), 71 (66), 69 (70), 57 (99)
HREI-MS: *m/z* found		408.1555 ([M^+], calcd. for $C_{24}H_{24}O_6$, 408.1573)

Subfraction C7-23 its chromatogram showed one UV-active spot on normal phase TLC using 40% dichloromethane in hexane (7 runs) as a mobile phase with the R_f value of 0.47, indicating the appearance of **W3** as a major compound, and further showed inseparable spots over **W3**. Hence, it was not further conducted.

Subfraction C7-24 appeared only one UV-active spot on normal phase TLC with the R_f value of 0.37 using 40% dichloromethane in hexane (7 runs) as a mobile phase. It was assigned as **W4**. Therefore, this fraction was not purified.

Subfraction C7-25 demonstrated two distinct spots under UV-S on normal phase TLC using 40% dichloromethane in hexane (7 runs) as a mobile phase with the R_f values of 0.37 and 0.28, respectively. It was separated by column chromatography over silica gel using 100% hexane, dichloromethane-hexane gradient and finally with 100% dichloromethane. All subfractions were examined by TLC, combined on the basis of their chromatography characteristics and then evaporated to dryness under reduced pressure to afford four subfractions. The results were shown in **Table 35**.

Table 35 Subfractions obtained from **subfraction C7-25** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
C7-251	2-30% CH_2Cl_2/Hexane	0.0019	Yellow solid
C7-252	40% CH_2Cl_2/Hexane	0.0050	Orange viscous liquid
C7-253	40% CH_2Cl_2/Hexane	0.0085	Yellow viscous liquid
C7-254	40% CH_2Cl_2/Hexane- 100% CH_2Cl_2	0.0131	Yellow viscous liquid

Subfraction C7-251 showed no definite spot on normal phase TLC under UV-S using 30% dichloromethane in hexane (3 runs) as a mobile phase. No further purification was attempted.

Subfraction C7-252 appeared one major UV-active spot with the R_f value of 0.52 on normal phase TLC using 30% dichloromethane in hexane (3 runs) as a mobile phase. This result indicated the presence of **W4.** Thus, further separation was not performed.

Subfraction C7-253 displayed only one UV-active spot on normal phase TLC with the R_f value of 0.33 using 30% dichloromethane in hexane (3 runs) as a mobile phase and then to test with the other condition using 30% ethyl acetate in hexane (2runs) as a mobile phase which showed a single spot with the R_f value of 0.21. Further purification by precoated TLC on silica gel plates with 10% ethyl acetate in hexane (11 runs) as a mobile phase gave three bands.

Band 1 was a white solid (0.0003 g) which showed a single UV-active spot on normal phase TLC using 12% ethyl acetate in hexane (2 runs) and 20% ethyl acetate in hexane (2 runs) as mobile phases with the R_f value of 0.63. Its ^{1}H NMR spectral data indicated that it contained some impurities. Because of low quantity, it was not further purified.

Band 2 was a white solid (0.0008 g) which contained one UV-active spot on normal phase TLC using 12% ethyl acetate in hexane (2 runs) and 20% ethyl acetate in hexane (2 runs) as mobile phases with the R_f value of 0.33. According to its ^{1}H NMR spectral data, this component contained some impurities. Thus, further purification was not attempted.

Band 3 was obtained as a yellow solid (0.0063 g). Its chromatogram showed one major spot on normal phase TLC under UV-S with the R_f value of 0.13 using 12% ethyl acetate in hexane (2 runs) and 20% ethyl acetate in hexane (2 runs) as mobile phases and its ^1H NMR spectrum suggested that it was a pure compound. Because this compound was kept in a NMR tube in $CDCl_3$ solution at 10 °C and its chromatogram on normal phase TLC showed three spots using 10% ethyl acetate in hexane (3 runs) as a mobile phase with the R_f values of 0.40, 0.18 and 0.05, thus, it was decomposed. Further attempted purification by precoated TLC on silica gel plates with 10% ethyl acetate in hexane (5 runs) as a mobile phase afforded three bands, as shown in **Table 36**.

Table 36 Bands obtained from **band 3 of subfraction C7-253** by precoated TLC on silica gel plates

Band	Weight (g)	Physical appearance	R_f
1	0.0003	White solid	0.45
2	0.0008	White solid	0.23
3	0.0011	Pale yellow solid	0.08

These bands showed one UV-active spot using 20% ethyl acetate in hexane (3 runs) as a mobile phase. The separation was not successful because ^1H NMR spectral data of these bands contained impurities. Thus, it was not investigated further.

Subfraction C7-254 contained no spot on normal phase TLC under UV-S using 30% dichloromethane in hexane (3 runs) as a mobile phase. No attempted purification was carried out.

Subfraction C7-26 displayed no definite spot on normal phase TLC under UV-S using 60% dichloromethane in hexane (3 runs) as a mobile phase. No further separation was conducted.

Subfraction C7-3 showed two spots under UV-S on normal phase TLC using 10% ethyl acetate in hexane (4 runs) as a mobile phase with the R_f values of 0.21 and 0.08. Further separation by column chromatography over silica gel. Elution was performed initially with 100% hexane, gradually enriched with ethyl acetate until 100% ethyl acetate, followed by increasing methanol up to 70% methanol in ethyl acetate. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in **Table 37**.

Table 37 Subfractions obtained from **subfraction C7-3** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
C7-31	100% Hexane - 10% EtOAc/Hexane	0.0031	Yellow viscous liquid
C7-32	15-20% EtOAc/Hexane	0.0563	Yellow viscous liquid
C7-33	20-60% EtOAc/Hexane	2.6022	Brown-red viscous liquid
C7-34	60% EtOAc/Hexane-100% EtOAc	0.1326	Brown-red viscous liquid
C7-35	2-70% MeOH/EtOAc	0.1111	Dark brown viscous liquid

Subfraction C7-31 demonstrated no distinct spot on normal phase TLC under UV-S using 20% ethyl acetate in hexane (3 runs) as a mobile phase. Therefore, it was not further separated.

Subfraction C7-32 displayed many inseparable UV-active spots on normal phase TLC using 20% ethyl acetate in hexane (3 runs) as a mobile phase. No further purification was carried out.

Subfraction C7-33 showed one major UV-active spot on normal phase TLC using 20% ethyl acetate in hexane as a mobile phase (3 runs) with the R_f value of 0.20 and showed many pale UV-active spots over a major spot. When using 50% dichloromethane in hexane as a mobile phase (8 runs) its chromatogram appeared two overlapping UV-active spots on normal phase TLC. It was separated by column chromatography over silica gel using 100% hexane, dichloromethane-hexane gradient, 100% dichloromethane, methanol-dichloromethane gradient and finally with 100% methanol. All subfractions were examined by TLC, combined on the basis of their chromatography characteristics and then evaporated to dryness under reduced pressure to afford three subfractions, as shown in **Table 38**.

Table 38 Subfractions obtained from **subfraction C7-33** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
C7-331	100% Hexane - 100% CH_2Cl_2	0.0349	Yellow viscous liquid
C7-332	2-5% MeOH/CH_2Cl_2	1.9227	Yellow viscous liquid
C7-333	10% MeOH/CH_2Cl_2- 100% MeOH	0.6617	Brown viscous liquid

Subfraction C7-331 contained many inseparable UV-active spots on normal phase TLC using 70% dichloromethane in hexane as a mobile phase (5 runs). Thus, further purification was not attempted.

Subfraction C7-332 showed two major UV-active spots on normal phase TLC using 50% dichloromethane in hexane as a mobile phase (8 runs) with the R_f values of 0.33 and 0.26. It was further separated by column chromatography over silica gel using pure hexane, gradually enriched with dichloromethane until pure dichloromethane, followed by increasing amount of methanol until pure methanol. Subfractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure to give four subfractions, as shown in **Table 39**.

Table 39 Subfractions obtained from **subfraction C7-332** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
C7-332A	100% Hexane - 50% CH_2Cl_2/Hexane	0.0262	Yellow gum mixed with yellow solid
C7-332B	70% CH_2Cl_2/Hexane-100% CH_2Cl_2	1.0801	Orange gum
C7-332C	100% CH_2Cl_2	0.2793	Orange solid
C7-332D	2% MeOH/CH_2Cl_2-100% MeOH	0.5233	Brown gum

Subfraction C7-332A showed no UV-active spot on normal phase TLC using pure dichloromethane as a mobile phase (3 runs). No further separation was conducted.

Subfraction C7-332B showed one major UV-active spot on normal phase TLC using pure dichloromethane as a mobile phase (3 runs) with the R_f value of 0.29. When using pure chloroform as a mobile phase (5 runs), its chromatogram showed one UV-active spot on normal phase TLC with the R_f value of 0.18. Further separation by column chromatography over silica gel was performed. Elution was performed initially with pure chloroform, gradually enriched with methanol until pure methanol. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to give four subfractions, as shown in **Table 40**.

Table 40 Subfractions obtained from **subfraction C7-332B** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
C7-332B-1	100% $CHCl_3$	0.0129	Yellow solid
C7-332B-2	2% MeOH/$CHCl_3$	0.6407	Yellow viscous liquid
C7-332B-3	2-4% MeOH/$CHCl_3$	0.3388	Orange viscous liquid mixed with orange solid
C7-332B-4	6% MeOH/$CHCl_3$-100% MeOH	0.0778	Brown viscous liquid

Subfraction C7-332B-1 showed many pale UV-active spots on normal phase TLC using 100% chloroform (10 runs) as a mobile phase. No further purification was attempted.

Subfraction C7-332B-2 displayed two spots under UV-S on normal phase TLC using 100% chloroform (10 runs) as a mobile phase with the R_f values of 0.43 and 0.38. When using 50% dichloromethane in hexane as a mobile phase, the chromatogram showed three UV-active spots on normal phase TLC with the R_f values of 0.26, 0.18 and 0.08. Further separation by column chromatography over silica gel was conducted. Elution was performed initially with pure hexane, gradually enriched with dichloromethane up to pure dichloromethane, followed by increasing methanol up to pure methanol. Subfractions, with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in **Table 41**.

Table 41 Subfractions obtained from **subfraction C7-332B-2** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
C7-332B-21	100% Hexane - 70% CH_2Cl_2/Hexane	0.0560	Yellow viscous liquid
C7-332B-22	70% CH_2Cl_2/Hexane	0.0021	Yellow solid
C7-332B-23	100% CH_2Cl_2	0.0512	Yellow viscous liquid
C7-332B-24	100% CH_2Cl_2	0.1071	Brown-orange viscous liquid
C7-332B-25	100% CH_2Cl_2- 2% MeOH/ CH_2Cl_2	0.4233	Brown-orange viscous liquid
C7-332B-26	2% MeOH/ CH_2Cl_2- 100% MeOH	0.1261	Brown viscous liquid

Subfraction C7-332B-21 appeared no distinct spot on normal phase TLC under UV-S using 50% dichloromethane in hexane as a mobile phase. Therefore, it was not further separated.

Subfraction C7-332B-22 showed one yellow spot on normal phase TLC under UV-S using 50% dichloromethane in hexane as a mobile phase with the R_f value of 0.26. Its chromatogram showed the same spot with the highest of value as found in **subfraction C7-332B-23**. Therefore, it was further investigated in **subfraction C7-332B-23**.

Subfraction C7-332B-23 contained three definite UV-active spots with the R_f values of 0.30, 0.20 and 0.12, respectively using 50% dichloromethane in hexane (2 runs) as a mobile phase. Further purification by preparative TLC on silica gel plates with 60% dichloromethane in hexane (5 runs) as a mobile phase afforded three bands.

Band 1 was obtained as a pale yellow solid (0.0055 g), melting at 250.1-250.9 °C. Its chromatogram displayed one major spot on normal phase TLC under UV-S with the R_f value of 0.32 using 70% dichloromethane in hexane as a mobile phase (2 runs). Its ^{1}H NMR spectrum indicated the presence of **W6** as a pure compound.

UV(MeOH) λ_{max} nm (log ε)		242 (4.26), 260 (4.24), 293 (3.79), 320 (4.01), 364 (3.91)
FT-IR (KBr) $\nu_{cm^{-1}}$		3301 (OH stretching), 1648 (C=O stretching), 1608, 1578 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)		13.05 (*s*, 1H), 7.61 (*s*, 1H), 6.94 (*s*, 1H), 6.43 (*s*, 1H), 6.38 (*brs*, 1H), 5.24 (*tt*, *J* = 6.9, 1.5 Hz, 1H), 4.02 (*s*, 3H), 3.92 (*s*, 3H), 3.37 (*d, J* = 6.9 Hz, 2H), 1.81 (*brs*, 3H), 1.69 (*brs*, 3H)
^{13}C NMR ($CDCl_3$) (δppm) (75 MHz)		179.85, 163.83, 159.33, 156.23, 152.52, 152.36, 144.31, 132.10, 122.19, 113.61, 111.73, 104.58, 103.34, 102.48, 89.57, 56.52, 55.90, 25.80, 21.35, 17.79
DEPT (135°) ($CDCl_3$)	CH :	122.19, 104.58, 102.48, 89.57
	CH_2 :	21.35
	CH_3 :	56.52, 55.90, 25.80, 17.79

Band 2 was obtained as a yellow solid (0.0192 g), melting at 178.3-179.0 °C. Its chromatogram showed one UV-active spot on normal phase TLC with the R_f value of 0.26 using 70% dichloromethane in hexane as a mobile phase (2 runs). Its ^{1}H NMR spectrum was identified as **W7**.

UV(MeOH) λ_{max} nm (log ε)		246 (4.42), 282 (3.85), 318 (4.13), 361 (3.64)
FT-IR (KBr) ν_{cm-1}		3301 (OH stretching), 1649 (C=O stretching), 1608, 1578 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz)		12.98 (*s*, 1H), 7.93 (*d*, *J* = 8.7 Hz, 1H), 6.99 (*d*, *J* = 8.7 Hz, 1H), 6.58 (*brs*, 1H), 6.48 (*s*, 1H), 5.24 (*mt*, *J* = 6.9 Hz, 1H), 4.12 (*s*, 3H), 3.95 (*s*, 3H), 3.37 (*d, J* = 6.9 Hz, 2H), 1.82 (*s*, 3H), 1.70 (*s*, 3H)
^{13}C NMR ($CDCl_3$) (δ ppm) (75 MHz)		180.06, 164.06, 159.67, 155.72, 154.23, 149.57, 133.63, 131.93, 122.03, 121.92, 115.14, 112.35, 112.27, 103.17, 89.80, 61.94, 55.97, 26.37, 25.79, 21.32, 17.79
DEPT (135°) ($CDCl_3$)	CH :	122.03, 121.92, 112.35, 89.80
	CH_2 :	21.32
	CH_3 :	61.94, 55.97, 25.79, 17.79
EIMS *m/z* (% relative intensity):		356 ([M^+], 10), 355 (45), 340 (25), 312 (84), 300 (100), 297 (25), 177 (29), 148 (83), 140 (27), 97 (32), 95 (38), 85 (60), 83 (97), 69 (65), 57 (84)
HREI-MS: *m/z* found		356.1250 ([M^+], calcd. for $C_{20}H_{20}O_6$, 356.1260)

Band 3 was a yellow gum (0.0104 g). It was shown to be **W12** as a major compound on normal phase TLC with the R_f value of 0.16 using 70%

dichloromethane in hexane as a mobile phase (2 runs) and the ^{1}H NMR spectral data was identical to those **W12**.

Subfraction C7-332B-24 its chromatogram showed a yellow UV-active spot on normal phase TLC with the R_f value of 0.08 using 50% dichloromethane in hexane as a mobile phase. Its ^{1}H NMR spectrum was identical to that of **W12**. Thus, it was not separated.

Subfraction C7-332B-25 its chromatogram on normal phase TLC using 100% dichloromethane as a mobile phase (3 runs) showed one UV-active spot with the R_f value of 0.33, indicating the presence of **W13**. This component was also obtained from **subfraction C7-332B-3**. Therefore, this subfraction was not performed.

Subfraction C7-332B-26 contained many inseparable UV-active spots on normal phase TLC using 100% dichloromethane as a mobile phase (3 runs). Thus, further purification was not attempted.

Subfraction C7-332B-3, upon standing at room temperature, afforded a yellow solid (**W13**, 0.0263 g**)**, melting at 179.3-180.0 °C. Its chromatogram showed a single spot on normal phase TLC under UV-S with the R_f value of 0.24 using 100% dichloromethane (7 runs) as a mobile phase. According to its ^{1}H NMR spectral data, it was assigned to be α-mangostin. The filtrate showed the same chromatogram as that of **W13** on normal phase TLC using 100% dichloromethane (7 runs) as a mobile phase. Thus, further purification was not conducted.

UV(MeOH) λ_{max} nm (log ε)		244 (4.65), 258 (4.58), 316 (4.47), 358 (4.01)
FT-IR (KBr) $\nu_{cm^{-1}}$		3380 (OH stretching), 1646 (C=O stretching), 1594 (C=C aromatic ring)
^{1}H NMR (CDCl$_3$) (δ ppm) (300 MHz)		13.78 (*s*, 1H), 6.82 (*s*, 1H), 6.41 (*s*, 1H), 6.29 (*s*, 1H), 6.28 (*s*, 1H), 5.29 (*mt*, J = 7.2 Hz, 1H), 5.26 (*mt*, J = 6.6 Hz, 1H), 4.08 (*d, J* = 6.3 Hz, 2H), 3.81 (*s*, 3H), 3.45 (*d, J* = 7.2 Hz, 2H), 1.84 (*brs*, 6H), 1.77 (*brs*, 3H), 1.69 (*brs*, 3H)
^{13}C NMR (CDCl$_3$) (δ ppm) (75 MHz)		182.02, 161.62, 160.60, 155.76, 155.05, 154.56, 142.57, 137.04, 135.66, 132.13, 123.16, 121.47, 112.18, 108.50, 103.61, 101.58, 93.30, 62.03, 26.56, 25.84, 25.81, 21.45, 18.22, 17.91
DEPT (135°) (CDCl$_3$)	CH :	123.16, 121.47, 101.58, 93.30
	CH$_2$:	26.56, 21.45
	CH$_3$:	62.03, 25.84, 25.81, 18.22, 17.91

Subfraction C7-332B-4 displayed no UV-active spot on normal phase TLC using pure chloroform (10 runs) as a mobile phase. No further purification was carried out.

Subfraction C7-332C showed one UV-active spot on normal phase TLC using pure dichloromethane as a mobile phase (3 runs) with the R_f value of 0.29, indicating the presence of **W13** as a major compound, further showed many inseparable spots under UV-S on normal phase TLC. Thus, further purification was not attempted.

Subfraction C7-332D contained many pale UV-active spots on normal phase TLC using pure dichloromethane as a mobile phase (3 runs). No further separation was performed.

Subfraction C7-333 contained no UV-active spot on normal phase TLC using 70% dichloromethane in hexane as a mobile phase (4 runs). Thus, further purification was not conducted.

Subfraction C7-34 showed long oval UV-active spot on normal phase TLC using 20% ethyl acetate in hexane as a mobile phase (3 runs). When using pure chloroform as a mobile phase (4 runs), its chromatogram displayed one UV-active spot on normal phase TLC with the R_f value of 0.24. It was separated by column chromatography over silica gel using pure chloroform, methanol-chloroform gradient and finally with pure methanol. Subfractions were examined by TLC, combined on the basis of their chromatography characteristics and evaporated to dryness under reduced pressure to give three subfractions, as shown in **Table 42**.

Table 42 Subfractions obtained from **subfraction C7-34** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
C7-341	100% $CHCl_3$	0.0052	Yellow viscous liquid mixed with yellow solid
C7-342	2% $MeOH/CHCl_3$	0.0013	Yellow viscous liquid
C7-343	5% $MeOH/CHCl_3$- 100% MeOH	0.1250	Brown viscous liquid

Subfraction C7-341 appeared no distinct spot on normal phase TLC under UV-S using pure chloroform (4 runs) and 2% acetone in chloroform (2 runs) as mobile phases. Therefore, it was not further separated.

Subfraction C7-342 (**W14**) showed a single UV-active spot on normal phase TLC using pure chloroform (4 runs) and 2% acetone in chloroform (2 runs) as mobile phases with the R_f value of 0.52.

UV(MeOH) λ_{max} nm (log ε)		240 (3.88), 257 (3.81), 320 (3.56), 365 (3.55)
FT-IR (neat) ν_{cm-1}		3365 (OH stretching), 1638 (C=O stretching), 1603 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δ ppm) (300 MHz)		13.39 (*brs*, 1H), 7.60 (*s*, 1H), 6.94 (*s*, 1H), 6.39 (*s*, 1H), 6.37 (*brs*, 1H), 6.29 (*brs*, 1H), 5.31 (*brt*, *J* = 6.3 Hz, 1H), 5.06 (*m*, 1H), 4.02 (*s*, 3H), 3.49 (*d, J* = 7.2 Hz, 2H), 2.10 (*m*, 4H), 1.84 (*s*, 3H), 1.68 (*s*, 3H), 1.60 (*s*, 3H)
^{13}C NMR ($CDCl_3$) (δ ppm) (125 MHz)		179.96, 162.23, 160.10, 157.61, 156.03, 152.61, 144.25, 139.95, 132.19, 123.63, 121.14, 113.34, 108.48, 104.05, 103.07, 102.58, 94.23, 56.49, 39.70, 26.29, 25.68, 21.37, 17.71, 16.25
DEPT (135°) ($CDCl_3$)	CH :	123.63, 121.14, 104.05, 102.58, 94.23
	CH_2 :	39.70, 26.29, 21.37
	CH_3 :	56.49, 25.68, 17.71, 16.25

Subfraction C7-343 displayed no UV-active spot on normal phase TLC using pure chloroform (4 runs) and 2% acetone in chloroform (2 runs) as mobile phases. Thus, further purification was not conducted.

Subfraction C7-35 showed long tail on normal phase TLC under UV-S using 20% ethyl acetate in hexane as a mobile phase (3 runs). No further investigation was attempted.

Subfraction C7-4 showed no spot under UV-S on normal phase TLC using 30% ethyl acetate in hexane and 50% ethyl acetate in hexane (2 runs) as mobile phases. No attempted investigation was carried out.

Fraction C8 contained many inseparable spots under UV-S on normal phase TLC with 40% ethyl acetate in hexane as a mobile phase (4 runs). Therefore, it was not further investigated.

Fraction D showed one major component on normal phase TLC under UV-S using 40% ethyl acetate in hexane (2 runs) and 50% ethyl acetate in hexane (2 runs) as mobile phases with the R_f value of 0.58. It was further purified by column chromatography over silica gel. Elution was performed with 100% hexane, followed by increasing the polarity with ethyl acetate until 100% ethyl acetate and finally enriched the polarity with methanol until 100% methanol. Fractions, which contained the similar components, were combined and evaporated to dryness under reduced pressure, to give five fractions, as shown in **Table 43**.

Table 43 Fractions obtained from **fraction D** by column chromatography over silica gel

Fraction	**Eluent**	**Weight (g)**	**Physical appearance**
D1	100% Hexane - 40% EtOAc/Hexane	0.0165	Pale yellow gum
D2	40% EtOAc/Hexane	0.0583	Yellow gum
D3	40-50% EtOAc/Hexane	0.1731	Yellow-orange gum mixed with yellow solid
D4	50-80% EtOAc/Hexane	0.3975	Yellow-brown gum mixed with yellow solid
D5	2% MeOH/ EtOAc - 100% MeOH	0.3001	Brown gum

Fraction D1 demonstrated no spot on normal phase TLC under UV-S using 10% ethyl acetate in hexane (6 runs) as a mobile phase. Therefore, it was not further separated.

Fraction D2 displayed two spots under UV-S on normal phase TLC using 10% ethyl acetate in hexane (6 runs) as a mobile phase with the R_f values of 0.26 and 0.05. Further separation by column chromatography over silica gel was performed. Elution was conducted initially with pure hexane and finally enriched with ethyl acetate up to pure ethyl acetate. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in **Table 44**.

Table 44 Subfractions obtained from **fraction D2** by column chromatography over silica gel

Subfraction	Eluent	Weight (g)	Physical appearance
D2-1	100% Hexane - 20% EtOAc/Hexane	0.0065	Yellow viscous liquid
D2-2	20-50% EtOAc/Hexane	0.0297	Yellow viscous liquid
D2-3	60% EtOAc/Hexane-100% EtOAc	0.0209	Brown viscous liquid

Subfraction D2-1 displayed no definite UV-active spot on normal phase TLC using 30% ethyl acetate in hexane (2 runs) as a mobile phase. Therefore, it was not further investigated.

Subfraction D2-2 contained two UV-active spots with the R_f values of 0.25 and 0.08, respectively using 60% dichloromethane in hexane (3 runs) as a mobile phase. Further purification by preparative TLC on silica gel plates with 40% dichloromethane in hexane (5 runs) as a mobile phase afforded three bands.

Band 1 was a yellow solid (0.0007 g), which showed two spots under UV-S on normal phase TLC using 50% dichloromethane in hexane (5 runs) as a mobile phase with the R_f values of 0.80 and 0.45, respectively. Because of low quantity, it was not further purified.

Band 2 was a yellow gum (0.0014 g) its chromatogram showed a single UV-active spot on normal phase TLC using 50% dichloromethane in hexane (5 runs) as a mobile phase with the R_f value of 0.70. Its ^{1}H NMR spectral data indicated that it contained some impurities. No attempted investigation was carried out.

Band 3 (**W15**) was obtained as a yellow gum (0.0023 g). Its chromatogram appeared one major spot on normal phase TLC under UV-S with the R_f value of 0.13 using 50% dichloromethane in hexane (5 runs) as a mobile phase.

UV(MeOH) λ_{max} nm (log ε)		244 (4.33), 268 (4.07), 311 (4.16), 352 (3.83)
FT-IR (neat) ν_{cm-1}		3412 (OH stretching), 1643 (C=O stretching), 1595 (C=C aromatic ring)
^{1}H NMR ($CDCl_3$) (δppm) (300 MHz)		14.52 (*s*, 1H), 13.00 (*s*, 1H), 10.37 (*s*, 1H), 6.95 (*s*, 1H), 6.59 (*brs*, 1H), 5.23 (*m*, 2H), 4.08 (*d, J* = 6.3 Hz, 2H), 3.83 (*s*, 3H), 3.35 (*d, J* = 7.2 Hz, 2H), 1.84 (*s*, 3H), 1.81 (*s*, 3H), 1.69 (*s*, 6H)
^{13}C NMR ($CDCl_3$) (δppm) (125 MHz)		190.08, 181.32, 167.09, 167.00, 157.49, 155.20, 154.89, 143.45, 137.48, 132.63, 132.62, 122.57, 121.08, 112.35, 111.00, 102.30, 102.07, 101.77, 62.15, 26.60, 25.80, 25.79, 20.51, 18.23, 17.81
DEPT (135°) ($CDCl_3$)	CH :	122.57, 121.08, 101.77
	CH_2 :	26.60, 20.51
	CH_3 :	62.15, 25.80, 25.79, 18.23, 17.81
EIMS *m/z* (% relative intensity):		438 ([M^+], 4), 437 (12), 381 (14), 366 (22), 177 (32), 160 (20), 148 (27), 132, (17), 118 (23), 111 (24), 97 (40), 84 (60), 82 (87), 69 (81)
HREI-MS: *m/z* found		438.1691 ([M^+], calcd. for $C_{25}H_{26}O_7$, 438.1679)

Subfraction D2-3 showed no spot on normal phase TLC under UV-S using 30% ethyl acetate in hexane (2 runs) as a mobile phase. No attempted investigation was carried out.

Fraction D3 showed many inseparable spots under UV-S on normal phase TLC with 30% ethyl acetate in hexane (2 runs) and 40% ethyl acetate in hexane (2 runs) as mobile phases. Therefore, it was not further investigated.

Fraction D4 demonstrated one major spot under UV-S on normal phase TLC using 30% ethyl acetate in hexane (2 runs) and 40% ethyl acetate in hexane (2 runs) as mobile phases with the R_f value of 0.32. Further separation by column chromatography over silica gel was conducted. Elution was performed initially with pure hexane, gradually enriched with ethyl acetate up to pure ethyl acetate, followed by increasing methanol up to pure methanol. Subfractions with the similar chromatogram were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in **Table 45**.

Table 45 Subfractions obtained from **fraction D4** by column chromatography over silica gel

Subfraction	**Eluent**	**Weight (g)**	**Physical appearance**
D4-1	100% Hexane - 20% EtOAc/Hexane	0.0261	Orange viscous liquid
D4-3	20-50% EtOAc/Hexane	0.1035	Yellow viscous liquid
D4-4	50% EtOAc/Hexane	0.1034	Orange viscous liquid
D4-5	70% EtOAc/Hexane- 100% MeOH	0.1561	Brown viscous liquid

Subfraction D4-1 contained many inseparable spots under UV-S on normal phase TLC with 20% ethyl acetate in hexane (2 runs) as a mobile phase. Therefore, it was not further separated.

Subfraction D4-2 its chromatogram on normal phase TLC using 20% ethyl acetate in hexane (2 runs) as a mobile phase showed one UV-active spot with the R_f value of 0.18, indicating that it was **W5**. This compound obtained from **subfraction D4-3**. Therefore, this subfraction was not performed.

Subfraction D4-3 showed a single spot on normal phase TLC under UV-S using 20% ethyl acetate in hexane (2 runs) with the R_f value of 0.18. According to its ^{1}H NMR spectral data, it was identified to be **W5**. It was previously investigated from **subfractions 9-2** and **9-3** in the first part.

Subfraction D4-4 displayed no spot under UV-S on normal phase TLC with 20% ethyl acetate in hexane (2 runs) as a mobile phase. Thus, further purification was not conducted.

Fraction D5 showed no distinct spot under UV-S on normal phase TLC with 30% ethyl acetate in hexane (2 runs) and 40% ethyl acetate in hexane (2 runs) as mobile phases. Therefore, it was not further separated.

Fractions E to G showed no UV-active spot on normal phase using 40% ethyl acetate in hexane (2 runs) and 50% ethyl acetate in hexane (2 runs) as mobile phases. No further investigations were attempted.

2.3 RESULTS AND DISCUSSION

The fresh fruits of *Garcinia cowa* collected from Songkhla province in the Southern part of Thailand were extracted with hexane. The crude hexane extract was separated by quick column chromatography. Selected fractions were further purified by column chromatography, precoated TLC or preperative TLC using various mobile phase systems to obtain five new xanthones (**W4, W7, W10, W11** and **W15**), together with ten known xanthones, namely fuscaxanthone C (**W1**), 7-*O*-methylgarcinone E (**W2**), β-mangostin (**W3**), cowanol (**W5**), 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone (**W6**), mangostanin (**W8**), 6-*O*-methylmangostanin (**W9**), cowanin (**W12**), α-mangostin (**W13**) and cowaxanthone (**W14**), one of the new xanthone (**W4**) was previously reported as synthetic xanthone. All structures were elucidated using 1D and 2D NMR spectroscopic data and/or comparison of ^{1}H and / or ^{13}C NMR spectral data from those reported in the literature. The ^{13}C NMR signals were assigned from DEPT, HMQC and HMBC spectra.

2.3.1 Structure determination of compounds isolated from the fruits of *G. cowa*

2.3.1.1 Compound W 13: 1,3,6-trihydroxy-7-methoxy-2,8-*bis*(3-methyl-2-butenyl)-xanthone (α-mangostin)

Compound **W13** was isolated as a yellow solid, melting at 179.3-180.0 °C. The IR spectrum (**Figure 113**) exhibited absorption bands at 3380 cm^{-1} for a hydroxyl group, 1646 cm^{-1} for a conjugated carbonyl group and 1594 cm^{-1} for a conjugated carbon of aromatic ring. The UV spectrum (**Figure 112**) showed maximum absorption bands at λ_{max} 244, 258, 316 and 358 nm, indicating that **W13** was a xanthone derivative. Its ^{1}H NMR spectrum (**Figure 114**) (**Table 46**) showed the presence of three hydroxyl groups [δ_H 13.78 (1H, *s*, chelated OH), 6.41 (1H, *brs*) and 6.28 (1H, *brs*)], two aromatic protons [δ_H 6.82 (1H, *s*) and 6.29 (1H, *s*)] and one methoxyl group [δ_H 3.81 (3H, *s*)]. Other signals could be attributed to two prenyl groups {[δ_H 5.26 (1H, *mt*, J = 6.6 Hz), 4.08 (2H, *d,* J = 6.3 Hz), 1.84 (6H, *brs*), 1.69 (3H, *brs*)] and [5.29 (1H, *mt*, J = 7.2 Hz), 3.45 (2H, *d,* J = 7.2 Hz), 1.84 (6H, *brs*), 1.77 (3H, *brs*)]}. The ^{13}C NMR spectrum (**Figure 115**) (**Table 46**) showed 24 resonances for 24 carbon atoms: thirteen quaternary carbons (δ_C 182.02, 161.62, 160.60, 155.76, 155.05, 154.56, 142.57, 137.04, 135.66, 132.13, 112.18, 108.50 and 103.61), four methine carbons (δ_C 123.16, 121.47, 101.58 and 93.30), two methylene carbons (δ_C 26.56 and 21.45) and five methyl carbons (δ_C 62.03, 25.84, 25.81, 18.22 and 17.91). The chelated hydroxyl group (δ_H 13.78, 1-OH) was placed at C-1 because of its intramolecular hydrogen bond with the carbonyl group. In the HMBC spectrum (**Figure 118**) (**Table 46**), this chelated hydroxy proton and the methylene protons (δ_H 3.45, H-11) of the prenyl unit gave cross peaks with the same quaternary aromatic carbon (δ_C 108.50, C-2), indicating that the prenyl unit was located at C-2. The high-field aromatic proton at δ_H 6.29 was assigned to H-4, based on the HMBC correlations, with C-2, C-3 (δ_C 161.62), C-4a (δ_C 155.05) and C-9a (δ_C 103.61). These data indicated that the C-3 position was attached by a hydroxyl group. The lowest-field methylene protons (δ_H 4.08) of the prenyl unit gave cross peaks, in the HMBC spectrum, with three quaternary aromatic carbons [δ_C 142.57 (C-7), 137.04 (C-8) and 112.18 (C-8a)]. These results indicated that the prenyl group was located at the other *peri* position (C-8) to a carbonyl group. The correlation of the methoxy protons with the quaternary aromatic carbon (C-7), in the HMBC spectrum, established the location of the methoxyl group at C-7. The remaining aromatic proton at δ_H 6.82 showed cross peaks with C-6 (δ_C 155.76), C-7, C-8a and C-10a (δ_C 154.56), suggesting that it was located at C-5 (δ_C 101.58). The correlations of the lowest-field hydroxy proton (δ_H 6.41) with C-5, C-6 and C-7, in the HMBC spectrum, indicating the location of this hydroxyl group at C-6. Thus, compound **W13** was identified as 1,3,6-trihydroxy-7-methoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone, which was isolated from *G. mangostana* (Sen, *et al.*, 1982).

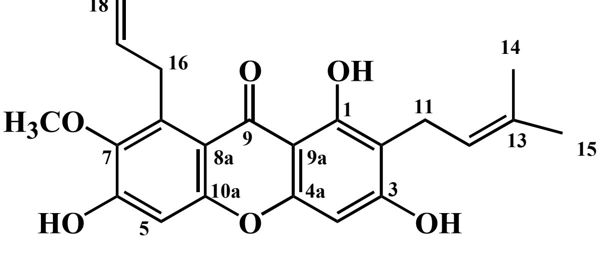

Table 46 The NMR data of compound **W13** and α-mangostin in $CDCl_3$

Position	**W13**		**HMBC Correlation**	**α-mangostin**	
	δ_H (mult., J_{Hz})	**δ_C (C-Type)**		**δ_H (mult., J_{Hz})^c**	**δ_C (C-Type)^d**
1		160.60 (C)			159.9 (C)
1-OH	13.78 (1H, *s*)		C-1, C-2, C-9a	13.80 (1H, *s*)	
2		108.50 (C)			109.7 (C)
3		161.62 (C)			162.3 (C)
3-OH	6.28 (1H, *s*)			6.16 (1H, *s*)	
4	6.29 (1H, *s*)	93.30 (CH)	C-2, C-3, C-4a, C-9, C-9a	6.28 (1H, *s*)	92.3 (CH)
4a		155.05 (C)			154.2 (C)
5	6.82 (1H, *s*)	101.58 (CH)	C-6, C-7, C-8a, C-9, C-10a	6.82 (1H, *s*)	101.8 (CH)
6		154.56 (C)			156.9 (C)
6-OH	6.41 (1H, *s*)		C-5, C-6, C-7	6.31 (1H, *s*)	
7		142.57 (C)			143.4 (C)
7-OCH_3	3.81 (3H, *s*)	63.02 (OCH_3)	C-7	3.81 (3H, *s*)	60.2 (OCH_3)
8		137.04 (C)			136.4 (C)
8a		112.18 (C)			110.1 (C)
9		182.02 (C=O)			181.3 (C=O)
9a		103.61 (C)			101.9 (C)
10a		155.76 (C)			154.6 (C)
11	3.45 (2H, *d*, 7.2)	21.45 (CH_2)	C-1, C-2, C-3, C-12, C-13	3.45 (2H, *d*, 7.0)	21.0 (CH_2)

Table 46 (Continued)

Position	W13		HMBC Correlation	α-mangostin	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})[c]	δ_C (C-Type)[d]
12	5.29 (1H, *mt*, 7.2)	121.47 (CH)		5.28 (1H, *t*, 7.0)	122.7 (CH)
13		135.66 (C)			130.3 (C)
14	1.84 (6H, *brs*)	17.91 (CH_3)	C-12, C-13	1.85 (3H, *s*)	17.7 (CH_3)
15	1.77 (3H, *brs*)	25.84 (CH_3)	C-12, C-13	1.78 (3H, *s*)	25.6 (CH_3)
16	4.08 (2H, *d*, 6.3)	26.56 (CH_2)	C-7, C-8, C-8a, C-17, C-18	4.11 (2H, *d*, 7.0)	25.8 (CH_2)
17	5.26 (1H, *mt*, 6.6)	123.16 (CH)		5.28 (1H, *brt*, 7.0)	123.8 (CH)
18		132.13 (C)			130.3 (C)
19	1.69 (3H, *brs*)	25.81 (CH_3)	C-17, C-18	1.70 (3H, *s*)	25.6 (CH_3)
20	1.84 (6H, *brs*)	18.22 (CH_3)	C-17, C-18	1.82 (3H, *s*)	18.1 (CH_3)

[c] ^{1}H NMR 400 MHz of α-mangostin in $CDCl_3$ (Mahabusarakam, *et al*., 1987)

[d] ^{13}C NMR 22.5 MHz of α-mangostin in DMSO-d_6 (Sen, *et al*., 1982)

2.3.1.2 Compound W3: 1,6-dihydroxy-3,7-dimethoxy-2,8-*bis*(3-methyl-2-butenyl)-xanthone (β-mangostin)

Compound **W3** was obtained as a yellow solid, melting at 175.8-176.4 °C. The UV (**Figure 34**) and the IR (**Figure 35**) spectra were similar to those of **W13**. These supported that **W3** has a xanthone skeleton. Its ^{1}H NMR data (**Figure 36**) (**Table 47**) were also similar to those of **W13** except for an additional signal of a methoxyl group at δ_H 3.90. The ^{13}C NMR spectral data (**Figure 37**) (**Table 47**) showed the same number of quaternary, methine, methylene and methyl carbons as those found in **W13** except for the fact that it contained one more methyl carbon at δ_C 55.83. This carbon corresponded to a methoxy carbon due to its chemical shift value. These data supported the presence of the methoxyl group. In the HMBC spectrum (**Figure 40**) (**Table 47**), the methoxy protons gave a cross peak with a quaternary carbon (δ_C 163.53, C-3), indicating the attachment of the methoxyl group at C-3. The remaining HMBC correlations were almost identical to those of **W13**, suggesting that **W3** contained the chelated hydroxyl group at C-1 (δ_C 159.78), two prenyl groups at C-2 (δ_C 111.53) and C-8 (δ_C 137.05), one free hydroxyl group at C-6 (δ_C 154.40) and two methoxyl groups at C-3 (δ_C 163.53) and C-7 (δ_C 142.58), respectively. Therefore, compound **W3** was assigned as 1,6-dihydroxy-3,7-dimethoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone, the methyl ether of **W13**, which was previously isolated from *G. cowa* (Likhitwitayawuid, *et al.*, 1998).

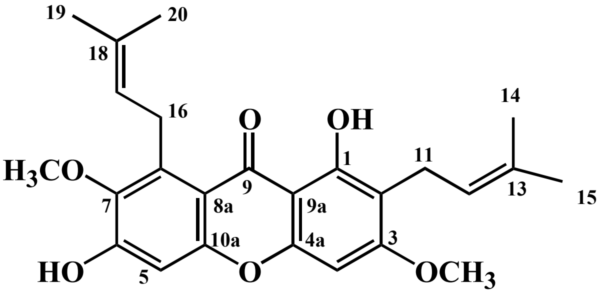

Table 47 The NMR data of compound **W3** and *β*-mangostin in $CDCl_3$

Position	**W3**		**HMBC Correlation**	***β*-mangostin**[c]	
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**		δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**
1		159.78 (C)			159.8 (C)
1-OH	13.42 (1H, *s*)		C-1, C-2, C-9a	13.39 (1H, *s*)	
2		111.53 (C)			111.7 (C)
3		163.53 (C)			163.5 (C)
3-OCH_3	3.90 (3H, *s*)	55.83 (OCH_3)		3.88 (3H, *s*)	56.1 (OCH_3)
4	6.33 (1H, *s*)	88.81 (CH)	C-2, C-3, C-4a, C-9, C-9a	6.32 (1H, *s*)	89.0 (CH)
4a		155.24 (C)			155.7 (C)
5	6.82 (1H, *s*)	101.46 (CH)	C-6, C-7, C-8a, C-9, C-10a	6.81 (1H, *s*)	101.6 (CH)
6		154.40 (C)[a]			155.3 (C)[b]
6-OH	6.37 (1H, *brs*)			6.30 (1H, *brs*)	
7		142.58 (C)	C-7		142.68 (C)
7-OCH_3	3.81 (3H, *s*)	62.06 (OCH_3)		3.79 (3H, *s*)	63.2 (OCH_3)
8		137.05 (C)			137.1 (C)
8a		112.42 (C)			112.6 (C)
9		181.94 (C=O)			181.9 (C=O)
9a		104.83 (C)			104.0 (C)
10a		155.72 (C)[a]			154.4 (C)[b]
11	3.35 (2H, *d*, 7.2)	21.36 (CH_2)	C-1, C-2, C-3, C-12, C-13	3.33 (2H, *d*, 7.0)	21.7 (CH_2)
12	5.23 (1H, *mt*, 6.9)	122.33 (CH)		5.21 (1H, *brt*, 7.0)	122.4 (CH)
13		131.68 (C)			131.7 (C)
14	1.80 (3H, *brs*)	17.77 (CH_3)	C-12, C-13	1.78 (3H, *s*)	18.1 (CH_3)

Table 47 (Continued)

Position	W3		HMBC Correlation	β-mangostin[c]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
15	1.68 (3H, *brs*)	25.81 (CH_3)	C-12, C-13	1.67 (6H, *s*)	26.1 (CH_3)
16	4.09 (2H, *d*, 6.3)	26.55 (CH_2)	C-7, C-8, C-8a, C-17, C-18	4.08 (2H, *d*, 6.4)	26.9 (CH_3)
17	5.26 (1H, *mt*, 6.3)	123.21 (CH)		5.24 (1H, *brt*, 6.4)	123.3 (CH)
18		132.08 (C)			132.1 (C)
19	1.69 (3H, *brs*)	25.81 (CH_3)	C-17, C-18	1.67 (6H, *s*)	26.1 (CH_3)
20	1.83 (3H, *brs*)	18.22 (CH_3)	*C-17, C-18*	1.81 (3H, *s*)	18.6 (CH_3)

[a,b] signals interchangeable

[c] ^{1}H NMR 300 MHz and ^{13}C NMR 75.5 MHz of β-mangostin in $CDCl_3$ (Likhitwitayawuid, *et al.*, 1998)

2.3.1.3 Compound W1: 1-hydroxy-3,6,7-trimethoxy-2,8-*bis*(3-methyl-2-butenyl)-xanthone (fuscaxanthone C)

Compound **W1** was obtained as a pale yellow solid, melting at 100.5-101.2 °C. The UV spectrum (**Figure 20**) showed maximum absorption bands at λ_{max} 245, 261, 314 and 358 nm indicated that **W1** has a xanthone chromophore. This was supported by the IR (**Figure 21**) absorption bands at 3372 and 1646 cm^{-1} for a hydroxyl group and a conjugated carbonyl group, respectively. The ^{1}H NMR spectral data (**Figure 22**) (**Table 48**) were similar to those of **W3** except for the presence of one additional methoxyl group at δ_H 3.96. These evidence suggested that one free hydroxyl group in **W3** was replaced by one methoxyl group in **W1**. The ^{13}C NMR spectrum (**Figure 23**) (**Table 48**) confirmed the above conclusion by the presence of one additional methoxy carbon at δ_C 55.99, which correlated to the methoxy protons (δ_H 3.96, OCH_3-6), in the HMQC spectrum (**Figure 25**). This methoxyl group was attached to C-6 (δ_C 158.04) due to a HMBC correlation (**Figure 26**) (**Table 48**) between the methoxy protons and C-6. In addition, the other substituents were located at the same positions as those found in **W3** by the HMBC data. Therefore, compound **W1** was identified as 1-hydroxy-3,6,7-trimethoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone, which was previously isolated from *G. fusca* (Ito, *et al.*, 2003a).

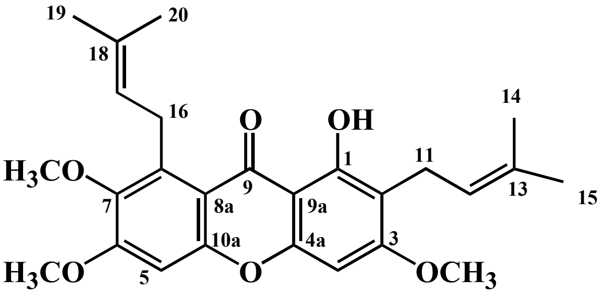

Table 48 The NMR data of compound **W1** and fuscaxanthone C in $CDCl_3$

Position	W1		HMBC Correlation	fuscaxanthone C[a]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
1		159.85 (C)			159.8 (C)
1-OH	13.49 (1H, *s*)		C-1, C-2, C-9a	13.48 (1H, *s*)	
2		111.55 (C)			111.5 (C)
3		163.61 (C)			163.4 (C)
3-OCH_3	3.90 (3H, *s*)	55.80 (OCH_3)		3.91 (3H, *s*)	55.8 (OCH_3)
4	6.32 (1H, *s*)	88.63 (CH)	C-2, C-3, C-4a, C-9, C-9a	6.33 (1H, *s*)	88.6 (CH)
4a		155.21 (C)			155.2 (C)
5	6.74 (1H, *s*)	98.20 (CH)	C-6, C-7, C-8a, C-9, C-10a	6.75 (1H, *s*)	98.2 (CH)

Table 48 (Continued)

Position	W1		HMBC Correlation	fuscaxanthone C[a]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
6		158.04 (C)			158.0 (C)
6-OCH$_3$	3.96 (3H, *s*)	55.99 (OCH$_3$)	C-6	3.96 (3H, *s*)	56.0 (OCH$_3$)
7		144.04 (C)			144.0 (C)
7-OCH$_3$	3.80 (3H, *s*)	60.95 (OCH$_3$)	C-7	3.80 (3H, *s*)	60.9 (OCH$_3$)
8		137.33 (C)			137.0 (C)
8a		112.16 (C)			112.1 (C)
9		182.00 (C=O)			182.0 (C=O)
9a		104.01 (C)			104.0 (C)
10a		155.42 (C)			155.4 (C)
11	3.35 (2H, *d*, 7.2)	21.57 (CH$_2$)	C-1, C-2, C-3, C-12, C-13	3.36 (2H, *d*, 7.0)	21.4 (CH$_2$)
12	5.27 (1H, *m*)	122.32 (CH)		5.25 (1H, *m*)	122.3 (CH)
13		130.87 (C)			131.7 (C)
14	1.80 (3H, *brs*)	17.79 (CH$_3$)	C-12, C-13	1.80 (3H, *s*)	17.8 (CH$_3$)
15	1.68 (6H, *brs*)	25.91 (CH$_3$)	C-12, C-13	1.68 (6H, *s*)	25.9 (CH$_3$)
16	4.13 (2H, *d*, 6.9)	26.16 (CH$_2$)	C-7, C-8, C-8a, C-17, C-18	4.13 (2H, *d*, 6.6)	26.2 (CH$_2$)
17	5.24 (1H, *m*)	123.24 (CH)		5.24 (1H, *m*)	123.2 (CH)
18		131.78 (C)			131.8 (C)
19	1.68 (6H, *brs*)	25.83 (CH$_3$)	C-17, C-18	1.68 (6H, *s*)	25.8 (CH$_3$)
20	1.85 (3H, *brs*)	18.19 (CH$_3$)	C-17, C-18	1.85 (3H, *s*)	18.2 (CH$_3$)

[a] ^{1}H NMR 400 MHz and ^{13}C NMR 100 MHz of fuscaxanthone C in CDCl$_3$ (Ito, *et al.*, 2003a)

2.3.1.4 Compound W4: 1,3-dihydroxy-6,7-dimethoxy-2,8-*bis*(3-methyl-2-butenyl)-xanthone

Compound **W4** was obtained as an orange gum. Its UV spectrum (**Figure 41**) (λ_{max} 244, 258, 315 and 355 nm) showed the presence of a xanthone nucleus. Its IR (**Figure 42**) spectrum showed an absorption band at 3365 (OH), 1639 (α,β-unsaturated C=O) and 1606 (conjugated double bond) cm^{-1}. The high resolution electron ionization mass spectrum (HREI-MS) (**Figure 48**) showed the molecular ion at *m/z* 424.1899 for a molecular formula of $C_{25}H_{28}O_6$. Its ^1H NMR spectrum (**Figure 43**) (**Table 49**) was similar to that of **W1** except that the methoxyl group at δ_H 3.90 of **W1** was replaced, in **W4**, by one free hydroxyl group (δ_H 6.25). The ^{13}C NMR spectrum (**Figure 44**) (**Table 49**) showed the same number and type of quaternary, methine, methylene and methyl carbons as those found in **W1** with less one methoxy carbon. These supported the absence of one methoxy carbon and the presence of one additional oxyquaternary carbon. In the HMBC spectral data (**Figure 47**) (**Table 49**), a high field aromatic proton (δ_H 6.29, H-4) showed cross peaks with C-2 (δ_C 108.45), C-3 (δ_C 161.57), C-4a (δ_C 155.04) and C-9a (δ_C 103.75), suggesting that the C-3 position was attached by a hydroxyl group. In addition, the HMBC correlation confirmed the attachment of all remaining substituents to be at the same location as those found in **W1**. Compound **W4** was assigned as 1,3-dihydroxy-6,7-dimethoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone. It was a new natural xanthone. However, the synthesis of this compound was previously reported in 1998 (Lu, *et al.*, 1998).

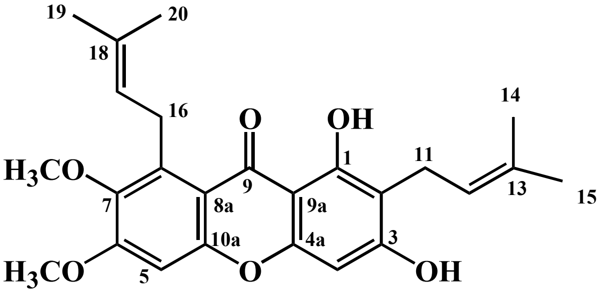

Table 49 The NMR data of compound **W4** in $CDCl_3$

Position	**W4**		**HMBC Correlation**
	δ_H **(mult.,** J_{Hz}**)**	δ_C **(C-Type)**	
1		160.63 (C)	
1-OH	13.84 (1H, *s*)		C-1, C-2, C-9a
2		108.45 (C)	
3		161.57 (C)	
3-OH	6.25 (1H, *brs*)		
4	6.29 (1H, *s*)	93.16 (CH)	C-2, C-3, C-4a, C-9, C-9a
4a		155.04 (C)	
5	6.75 (1H, *s*)	98.31 (CH)	*C-6, C-7, C-8a, C-9, C-10a*
6		158.14 (C)	
6-OCH_3	3.96 (3H, *s*)	56.04 (OCH_3)	C-6
7		144.20 (C)	
7-OCH_3	3.80 (3H, *s*)	60.96 (OCH_3)	C-7
8		137.29 (C)	
8a		111.89 (C)	
9		182.10 (C=O)	
9a		103.75 (C)	
10a		155.46 (C)	
11	3.45 (2H, *d*, 6.9)	21.46 (CH_2)	C-2, C-3, C-12, C-13
12	5.30 (1H, *mt*, 7.2)	121.48 (CH)	
13		135.78 (C)	
14	1.85 (6H, *s*)	17.94 (CH_3)*	C-12, C-13
15	1.77 (3H, *s*)	25.92 (CH_3)	C-12, C-13
16	4.13 (2H, *d*, 6.3)	26.18 (CH_2)	*C-7, C-8, C-8a, C-17, C-18*
17	5.25 (1H, *mt*, 6.3)	123.16 (CH)	

Table 49 (Continued)

Position	W4		HMBC Correlation
	δ_H (mult., J_{Hz})	δ_C (C-Type)	
18		131.89 (C)	
19	1.68 (3H, *s*)	25.87 (CH_3)	*C-17, C-18*
20	1.85 (6H, *s*)	18.19 (CH_3)*	C-17, C-18

* signals interchangeable

2.3.1.5 Compound W2: 1,3,6-trihydroxy-7-methoxy-2,5,8-*tris*(3-methyl-2-butenyl)xanthone (7-*O*-methylgarcinone E)

Compound **W2** was isolated as a yellow solid, melting at 63.2-64.0 °C. Its UV spectrum (**Figure 27**) (λ_{max} 244, 259, 316 and 357 nm) indicated the presence of a xanthone nucleus. The IR spectrum (**Figure 28**) showed absorption bands at 3410 (OH) and 1642 (α,β-unsaturated C=O) cm^{-1}. The ^{1}H NMR spectrum (**Figure 29**) (**Table 50**) and ^{13}C NMR spectral data (**Figure 30**) (**Table 50**) were similar to those of **W13**, suggesting that **W2** was also a 2,8-diprenylated xanthone. Direct comparison of their ^{1}H NMR spectra revealed that a low field aromatic proton (δ_H 6.82) of **W13** was replaced, in **W2**, by a prenyl group [δ_H 5.28 (3H, *m*), 3.56 (2H, *d*, J = 7.2 Hz), 1.87 (3H, *brs*) and 1.68 (6H, *brs*)]. In the HMBC correlation (**Figure 33**) (**Table 50**), the methylene protons (δ_H 3.56, H-21) of the prenyl group gave cross peaks with three quaternary carbons [δ_C 113.99 (C-5), 152.33 (C-6) and 153.55 (C-10a)], indicating that the prenyl group was located at C-5 (δ_C 113.99). The location of all remaining substituents were established by the HMBC correlations to be the same as those found in **W13**. Thus, compound **W2** was assigned as 1,3,6-trihydroxy-7-methoxy-2,5,8-*tris*

(3-methyl-2-butenyl)xanthone. This compound was previously isolated from *G*. cowa (Likhitwitayawuid, *et al*., 1997).

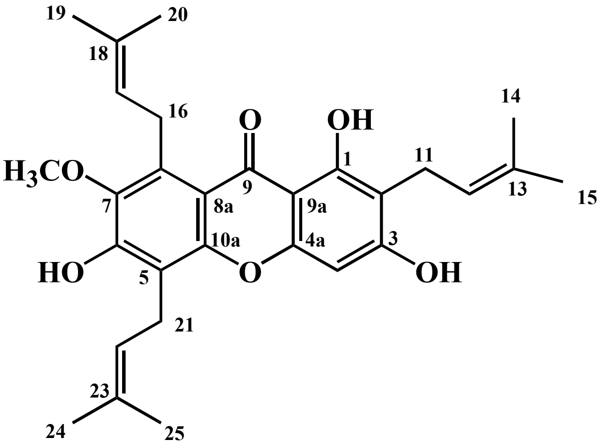

Table 50 The NMR data of compound **W2** and 7-*O*-methylgarcinone E in $CDCl_3$

Position	**W2**		**HMBC Correlation**	**7-*O*-methylgarcinone E**[a]	
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**		δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**
1		160.58 (C)			160.5 (C)
1-OH	13.86 (1H, *s*)		C-1, C-2, C-9a	13.82 (1H, *s*)	
2		108.36 (C)			108.3 (C)
3		161.53 (C)			161.5 (C)
3-OH	6.24 (1H, *brs*)			6.15 (1H, *brs*)	
4	6.33 (1H, *s*)	93.24 (CH)	C-2, C-3, C-4a, C-9a	6.31 (1H, *s*)	93.2 (CH)
4a		155.06 (C)			155.0 (C)
5		113.99 (C)			113.9 (C)
6		152.33 (C)			152.3 (C)
7		142.27 (C)			142.2 (C)
7-OCH_3	3.80 (3H, *s*)	62.00 (OCH_3)		3.79 (3H, *s*)	62.0 (OCH_3)
8		133.84 (C)			131.8 (C)

Table 50 (Continued)

Position	W2		HMBC Correlation	7-*O*-methylgarcinone E[a]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
8a		111.94 (C)			111.9 (C)
9		182.44 (C=O)			182.4 (C=O)
9a		103.59 (C)			103.6 (C)
10a		153.55 (C)			153.5 (C)
11	3.45 (2H, *d*, 7.2)	21.45 (CH_2)	C-1, C-2, C-3, C-12, C-13	3.44 (2H, *d*, 7.4)	21.4 (CH_2)
12	5.28 (3H, *m*)	121.53 (CH)		5.28 (1H, *m*)	121.5 (CH)
13		135.70 (C)			135.7 (C)
14	1.83 (3H, *brs*)	17.92 (CH_3)	C-12, C-13	1.83 (3H, *brs*)	17.9 (CH_3)
15	1.77 (3H, *brs*)	25.86 (CH_3)	C-12, C-13	1.75 (3H, *brs*)	25.8 (CH_3)
16	4.08 (2H, *d*, 6.3)	26.40 (CH_2)	C-7, C-8, C-8a, C-17, C-18	4.05 (2H, *d*, 6.4)	26.4 (CH_2)
17	5.28 (3H, *m*)	123.53 (CH)		5.25 (1H, *m*)	123.5 (CH)
18		131.82 (C)			133.9 (C)
19	1.68 (6H, *brs*)	25.79 (CH_3)	C-17, C-18	1.67 (6H, *brs*)	25.79 (CH_3)
20	1.85 (3H, *brs*)	18.20 (CH_3)	C-17, C-18	1.81 (3H, *brs*)	18.1 (CH_3)
21	3.56 (2H, *d*, 7.2)	22.63 (CH_2)	C-5, C-6, C-10a, C-22, C-23	3.55 (2H, *d*, 7.3)	22.6 (CH_2)
22	5.28 (3H, *m*)	121.15 (CH)		5.27 (1H, *m*)	121.1 (CH)
23		132.66 (C)			132.6 (C)
24	1.68 (6H, *brs*)	25.83 (CH_3)	C-22, C-23	1.67 (6H, *brs*)	25.8 (CH_3)
25	1.87 (3H, *brs*)	17.95 (CH_3)	C-22, C-23	1.85 (3H, *brs*)	17.9 (CH_3)

[a] ^1H NMR 500 MHz and ^{13}C NMR 125.7 MHz of 7-*O*-methylgarcinone E in $CDCl_3$ (Likhitwitayawuid, *et al*., 1997)

2.3.1.6 Compound W6: 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)-xanthone

Compound **W6** was obtained as a pale yellow solid, melting at 250.1-250.9 °C. The xanthone chromophore was evident by its UV spectrum (**Figure 56**) with maximum absorption bands at λ_{max} 242, 260, 293, 320 and 364 nm while the hydroxyl and the carbonyl stretching frequencies were found in the region of 3301 and 1648 cm^{-1}, respectively, in the IR spectrum (**Figure 57**). The ^{1}H NMR spectral data (**Figure 58**) (**Table 51**) were similar to those of **W3** except that a prenyl unit [δ_H 5.26 (1H, *mt*, *J* = 6.3 Hz), 4.09 (2H, *d*, *J* = 6.3 Hz), 1.81 (3H, *brs*) and 1.69 (3H, *brs*)] of **W3** was replaced, in **W6**, by a lowest-field aromatic proton [δ_H 7.61 (1H, *s*)]. The methine aromatic carbon at δ_C 104.58 (C-8) also correlated to the lowest-field aromatic proton, in the HMQC spectrum (**Figure 61**). The lowest-field aromatic proton showed strong cross peaks with C-7 (δ_C 144.31), C-8a (δ_C 113.61), C-9 (δ_C 179.85) and C-10a (δ_C 152.52), in the HMBC spectrum (**Figure 62**) (**Table 51**), suggesting that this aromatic proton was located at C-8 in the deshielding zone of a carbonyl group. The methoxy protons (δ_H 4.02) showed a correlation with C-7, indicating the presence of the methoxyl group at C-7. Irradiation of the methoxy protons (δ_H 4.02), in the NOE spectrum (**Figure 63**), enhanced signal of H-8, supporting the location of the methoxyl group at C-7. In addition, the other substituents were assigned to the same positions as those found in **W3** by the HMBC data. Thus, compound **W6** was identified as 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone, which was previously isolated from *G. mangostana* (Nilar, *et al.*, 2002).

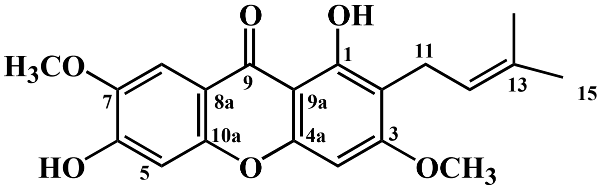

Table 51 The NMR data of compound **W6** and 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone in $CDCl_3$

Position	**W6**		**HMBC Correlation**	**1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone**[c]	
	δ_H (mult., J_{Hz})	**δ_C (C-Type)**		**δ_H (mult., J_{Hz})**	**δ_C (C-Type)**
1		159.33 (C)			159.4 (C)
1-OH	13.05 (1H, *s*)		C-1, C-2, C-9a	13.04 (1H, *s*)	
2		111.73 (C)			111.8 (C)
3		163.83 (C)			163.9 (C)
3-OCH_3	3.92 (3H, *s*)	55.90 (OCH_3)	C-3	3.92 (3H, *s*)	55.9 (OCH_3)
4	6.43 (1H, *s*)	89.57 (CH)	C-2, C-3, C-4a, C-9, C-9a	6.43 (1H, *s*)	89.6 (CH)
4a		156.23 (C)			156.3 (C)
5	6.94 (1H, *s*)	102.48 (CH)	C-6, C-7, C-8a, C-9	6.94 (1H, *s*)	102.5 (CH)
6		152.36 (C)[a]			152.4 (C)[b]
6-OH	6.38 (1H, *brs*)			6.40 (1H, *brs*)	
7		144.31 (C)			144.4 (C)
7-OCH_3	4.02 (3H, *s*)	56.52 (OCH_3)	C-7	4.01 (3H, *s*)	56.6 (OCH_3)
8	7.61 (1H, *s*)	104.58 (CH)	C-7, C-8a C-9, C-10a	7.61 (1H, *s*)	104.6 (CH)
8a		113.61 (C)			113.6 (C)
9		179.85 (C=O)			179.9 (C=O)
9a		103.34 (C)			103.4 (C)
10a		152.52 (C)[a]			152.6 (C)[b]

Table 51 (Continued)

Position	W6		HMBC Correlation	1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl) xanthone[c]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
11	3.37 (2H, *d*, 6.9)	21.35 (CH_2)	C-1, C-2, C-3, C-12, C-13	3.37 (2H, *d*, 7.3)	21.4 (CH_2)
12	5.24 (1H, *tt*, 6.9, 1.5)	122.19 (CH)		5.24 (1H, *tt*, 7.3, 1.4)	122.2 (CH)
13		132.10 (C)			131.9 (C)
14	1.81 (3H, *brs*)	17.79 (CH_3)	C-12, C-13	1.80 (3H, *s*)	17.8 (CH_3)
15	1.69 (3H, *brs*)	25.80 (CH_3)	C-12, C-13	1.69 (3H, *s*)	25.8 (CH_3)

[a,b] signals interchangeable

[c] ^{1}H NMR 500 MHz and ^{13}C NMR 125 MHz of 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone in $CDCl_3$ (Nilar, *et al.*, 2002)

2.3.1.7 Compound W12: 1,3,6-trihydroxy-7-methoxy-2-(3-methyl-2-butenyl)-8-(3,7-dimethyl-2,6-octadienyl)xanthone (cowanin)

Compound **W12** was obtained as a yellow solid, melting at 136.3-137.0 °C. The UV spectrum (**Figure 105**) showed maximum absorption bands at λ_{max} 243, 257, 317 and 358 nm indicated that **W12** has a xanthone skeleton while the IR spectrum (**Figure 106**) showed absorption bands at 3365 and 1639 cm^{-1} for a hydroxyl group and a conjugated carbonyl group. Its ^{1}H NMR data (**Figure 107**) (**Table 52**) were similar to those of **W13** except for the presence of a geranyl group. These suggested that one prenyl group [δ_H 5.26 (1H, *mt*, J = 6.6 Hz), 4.08 (2H, *d, J* = 6.3 Hz), 1.84 (6H, *brd*, J =

1.8 Hz) and 1.69 (3H, *brd*, $J = 0.9$ Hz)] of **W13** was replaced, in **W12**, by one geranyl group [δ_H 5.26 (1H, *mt*, $J = 6.3$ Hz), 5.02 (1H, *mt*, $J = 6.6$ Hz), 4.08 (2H, *d*, $J = 6.0$ Hz), 2.02 (4H, *m*), 1.83 (3H, *s*), 1.59 (3H, *s*) and 1.54 (3H, *s*)]. The highly-deshielded position of the methylene protons (δ_H 4.08, H-16) of a geranyl group indicating that this side chain was located at a *peri* position (δ_C 137.15, C-8), due to an anisotropic effect of a carbonyl group. These methylene protons, H-16, showed cross peaks, in the HMBC spectrum (**Figure 111**) (**Table 52**), with C-7 (δ_C 142.63), C-8 (δ_C 137.15), C-8a (δ_C 112.18) and C-17 (δ_C 123.27), supporting the location of the geranyl group at C-8. Two sets of methylene protons at δ_H 2.02 [4H, *m*, (H-20 and H-21)] belonged to the geranyl group. These were supported by the HMBC correlations (**Figure 111**) (**Table 52**) between the methylene proton (H-20) with C-17 and C-18 (δ_C 135.52) and the other methylene protons (H-21) with C-22 (δ_C 124.29) and C-23 (δ_C 131.27). suggesting that H-21 was located at C-22. The locations of all remaining substituents were established by the HMBC correlations to be the same as those found in **W13**. Therefore, compound **W12** was determined as 1,3,6-trihydroxy-7-methoxy-2-(3-methyl-2-butenyl)-8-(3,7-dimethyl-2,6-octadienyl)xanthone. Its ^{1}H NMR spectral data were in agreement with those of cowanin (Na Pattalung, *et al.*, 1994).

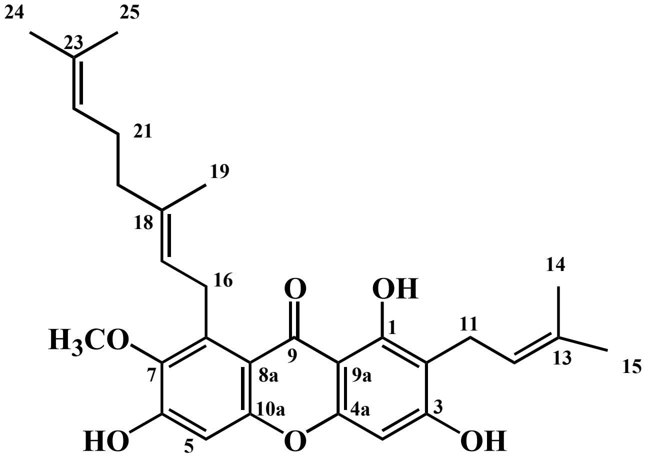

Table 52 The NMR data of compound **W12** and cowanin in $CDCl_3$

Position	**W12**		**HMBC Correlation**	**cowanin**[b]
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**		δ_H **(mult., J_{Hz})**
1		160.61 (C)		
1-OH	13.78 (1H, *s*)		C-1, C-2, C-9a	13.80 (1H, *s*)
2		108.67 (C)		
3		161.59 (C)		
3-OH	6.53 (2H, *brs*)			6.13 (2H, *brs*)
4	6.29 (1H, *s*)	93.22 (CH)	C-2, C-3, C-4a, C-9a	6.30 (1H, *s*)
4a		155.00 (C)		
5	6.83 (1H, *s*)	101.58 (CH)	C-6, C-7, C-8a, C-9, C-10a	6.86 (1H, *s*)
6		154.00 (C)[a]		
6-OH	6.53 (2H, *brs*)			6.13 (2H, *brs*)
7		142.63 (C)		
7-OCH_3	3.80 (3H, *s*)	61.97 (OCH_3)	C-7	3.80 (3H, *s*)
8		137.15 (C)		
8a		112.18 (C)		
9		181.96 (C=O)		
9a		103.56 (C)		
10a		155.71 (C)[a]		
11	3.43 (2H, *d*, 6.9)	21.44 (CH_2)	C-2, C-3, C-12, C-13	3.45 (2H, *d*, 7.0)
12	5.29 (1H, *mt*, 7.5)	121.59 (CH)		5.28 (1H, *m*)
13		135.25 (C)		
14	1.87 (3H, *s*)	16.47 (CH_3)	C-12, C-13	1.84 (3H, *s*)
15	1.76 (3H, *s*)	25.83 (CH_3)	C-12, C-13	1.76 (3H, *s*)
16	4.08 (2H, *d*, 6.0)	26.48 (CH_2)	C-7, C-8, C-8a, C-17	4.09 (2H, *d*, 7.0)
17	5.26 (1H, *mt*, 6.3)	123.27 (CH)		5.28 (1H, *m*)
18		135.52 (C)		

Table 52 (Continued)

Position	W12		HMBC Correlation	cowanin[b]
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})
19	1.83 (3H, *s*)	17.66 (CH_3)	C-17, C-18, C-20	1.82 (3H, *s*)
20	2.02 (4H, *m*)	39.70 (CH_2)	C-17, C-18	2.03 (4H, *m*)
21	2.02 (4H, *m*)	26.56 (CH_2)	C-22, C-23	2.03 (4H, *m*)
22	5.02 (1H, *mt*, 6.6)	124.29 (CH)		5.03 (1H, *mt*, 7.0)
23		131.27 (C)		
24	1.54 (3H, *s*)	17.90 (CH_3)	C-22, C-23	1.54 (3H, *s*)
25	1.59 (3H, *s*)	25.60 (CH_3)	C-22, C-23	1.59 (3H, *s*)

[a] signals interchangeable

[b] ^{1}H NMR 400 MHz of cowanin in $CDCl_3$ (Na Pattalung, *et al*., 1994)

2.3.1.8 Compound W5: 1,3,6-trihydroxy-7-methoxy-2-(3-methyl-4-hydroxy-2-butenyl)-8-(3,7-dimethyl-2,6-octadienyl)xanthone (cowanol)

Compound **W5** was obtained as a brown-yellow solid, melting at 122.2-124.0 ° C. Its UV spectrum (**Figure 49**) (λ_{max} 243, 258, 316 and 357 nm) showed the presence of a xanthone nucleus. Its IR (**Figure 50**) spectrum showed an absorption band at 3387 (OH), 1642 (α,β-unsaturated C=O) and 1606 (conjugated aromatic carbons) cm^{-1}. Its ^{1}H (**Figure 51**) (**Table 53**) and ^{13}C NMR spectra (**Figure 52**) (**Table 53**) were similar to those of **W12**, suggesting that **W5** was also 1,3,6,7-tetraoxygenated xanthone. Direct comparison of their ^{1}H NMR spectra revealed that the methyl protons of a prenyl side chain of **W12** was replaced, in **W5**, by an oxymethylene protons. The presence of an additional oxymethylene carbon at δ_C 62.51 (C-14) supported this conclusion. Thus, the side chain became 3-methyl-4-hydroxy-2-butenyl unit [δ_H 5.48 (1H, *brt*, J = 8.1 Hz), 4.35 (2H, *s*), 3.46 (2H, *d*, J = 7.8 Hz) and 1.81 (3H, *s*)]. In the HMBC spectrum (**Figure 55**) (**Table 53**), these oxymethylene protons (δ_H 4.35, H-14) gave cross peaks with C-12 (δ_C 126.98) and C-13 (δ_C 133.34), indicating that it was located at C-13. In addition, the other substituents were assigned to the same positions as those found in **W12** by the HMBC data. Hence, the structure of compound **W5** was identified as 1,3,6-trihydroxy-7-methoxy-2-(3-methyl-4-hydroxy-2-butenyl)-8-(3,7-dimethyl-2,6-octadienyl)xanthone, which was previously isolated from *G. cowa* (Na Pattalung, *et al.*, 1994).

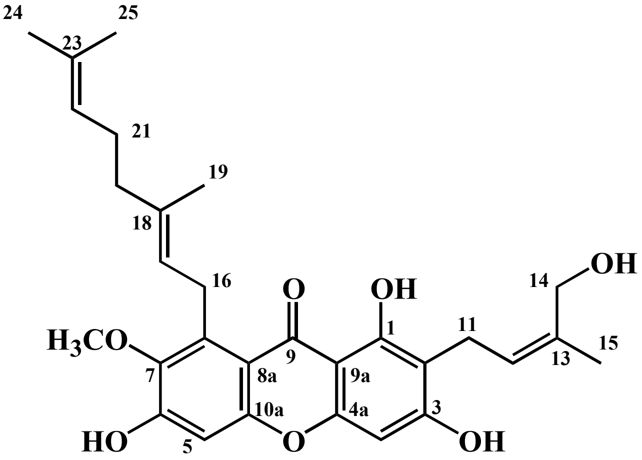

Table 53 The NMR data of compound **W5** and cowanol in $CDCl_3$

Position	**W5**		**HMBC Correlation**	**cowanol**[c]
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})
1		160.54 (C)		
1-OH	13.88 (1H, *s*)		C-1, C-2, C-9a	13.96 (1H, *s*)
2		108.37 (C)		
3		161.41 (C)[a]		
3-OH				
4	6.23 (1H, *s*)	93.49 (CH)	C-2, C-3, C-4a, C-9a	6.28 (1H, *s*)
4a		154.94 (C)[a]		
5	6.72 (1H, *s*)	101.66 (CH)	C-7, C-8a, C-9, C-10a	6.80 (1H, *s*)
6		154.67 (C)[b]		
6-OH				
7		142.66 (C)		
7-OCH_3	3.79 (3H, *s*)	61.87 (OCH_3)	C-7	3.79 (3H, *s*)
8		137.12 (C)		
8a		111.97 (C)		
9		181.82 (C=O)		
9a		103.29 (C)		
10a		155.60 (C)[b]		
11	3.46 (2H, *d*, 6.9)	21.49 (CH_2)	C-1, C-2, C-12, C-13	3.51 (2H, *d*, 7.0)
12	5.48 (1H, *brt*, 8.1)	126.98 (CH)		5.47 (1H, *brt*, 7.0)
13		133.34 (C)		
14	4.35 (2H, *s*)	62.51 (CH_2)	C-12, C-13	4.35 (2H, *brs*)
15	1.81 (3H, *s*)	22.75 (CH_3)	C-12, C-13	1.79 (3H, *s*)
16	4.03 (2H, *d*, 6.0)	26.44 (CH_2)	C-7, C-8, C-8a, C-17, C-18	4.09 (2H, *d*, 7.0)
17	5.24 (1H, *brt*, 6.0)	123.32 (CH)	C-19	5.24 (1H, *brt*, 7.0)

Table 53 (Continued)

Position	W5		HMBC Correlation	cowanol[c]
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})
18		135.47 (C)		
19	1.83 (3H, *s*)	16.45 (CH_3)	C-18, C-20	1.82 (3H, *s*)
20	2.02 (4H, *m*)	39.70 (CH_2)	C-17, C-18	2.03 (4H, *m*)
21	2.02 (4H, *m*)	26.57 (CH_2)	C-22, C-23	2.03 (4H, *m*)
22	5.01 (1H, *brt*, 6.6)	124.31 (CH)	C-19, C-20	5.02 (1H, *brt*, 7.0)
23		131.26 (C)		
24	1.53 (3H, *s*)	17.64 (CH_3)	C-22, C-23	1.54 (3H, *s*)
25	1.58 (3H, *s*)	25.59 (CH_3)	C-22, C-23	1.59 (3H, *s*)

[a,b] signals interchangeable

[c] ^{1}H NMR 400 MHz of cowanol in $CDCl_3$ (Na Pattalung, *et al.*, 1994)

2.3.1.9 Compound W14: 1,3,6-trihydroxy-7-methoxy-2-(3,7-dimethyl-2,6-octadienyl)xanthone (cowaxanthone)

Compound **W14** was obtained as a yellow viscous liquid. The xanthone chromophore was evident by its UV spectrum (**Figure 119**) which showed maximum absorption bands at λ_{max} 240, 257, 320 and 365 nm. The hydroxyl stretching and the carbonyl stretching frequencies were found in the region of 3365 and 1638 cm^{-1}, respectively, in the IR spectrum (**Figure 120**). The ^{1}H NMR spectral data (**Figure 121**) (**Table 54**) were similar to those of **W6** except that one prenyl group [δ_H 3.37 (2H, *d*, 6.9), 5.24 (1H, *tt*, 6.9, 1.5), 1.81 (3H, *brs*) and 1.69 (3H, *brs*)] and one methoxyl group [δ_H 3.92 (3H, *s*)] of **W6** at the right-handed side were replaced, in **W14**, by one geranyl group [δ_H 5.31 (1H, *brt*, J = 6.3 Hz), 5.06 (1H, *m*), 3.49 (2H, *d, J* = 7.2 Hz), 2.10 (4H, *brs*), 1.84 (3H, *s*), 1.68 (3H, *s*) and 1.60 (3H, *s*)] and one hydroxyl group [δ_H 6.29 (1H, *brs*)], respectively. The presence of two sets methylene carbons [δ_C 39.70 (C-15) and 26.29 (C-16)], which were also the characteristic of a geranyl group and an oxyquaternary carbon [δ_C 162.23 (C-3)], confirmed the above conclusion. The location of the geranyl unit was located at C-2 (δ_C 108.48), in the HMBC spectrum (**Figure 125**) (**Table 54**), since the methylene protons (δ_H 3.49, H-11) of this geranyl group gave cross peaks with C-1 (δ_C 160.10), C-2, C-3, C-12 (δ_C 121.14) and C-13 (δ_C 139.95). Thus, C-3 was attached by a hydroxyl group. The locations of all remaining substituents were established by the HMBC correlations to be the same as those found in **W6**. Hence, compound **W14** was identified as 1,3,6-trihydroxy-7-methoxy-2-(3,7-dimethyl-2,6-octadienyl)xanthone. It was previously isolated from *G. cowa* (Na Pattalung, *et al.*, 1994).

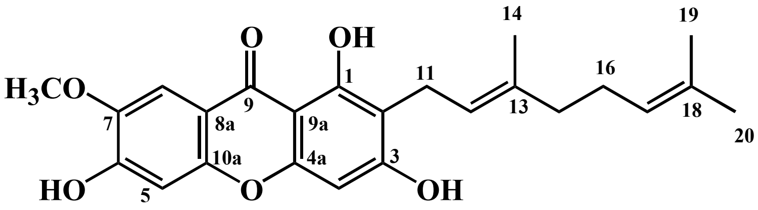

Table 54 The NMR data of compound **W14** and cowaxanthone in $CDCl_3$

Position	**W14**		**HMBC Correlation**	**cowaxanthone**[c]
	δ_H **(mult.,** J_{Hz}**)**	δ_C **(C-Type)**		δ_H **(mult.,** J_{Hz}**)**
1		160.10 (C)		
1-OH	13.39 (1H, *s*)		C-1, C-2, C-9a	14.45 (1H, *s*)
2		108.48 (C)		
3		162.23 (C)		
3-OH	6.29 (1H, *brs*)[a]			-
4	6.39 (1H, *s*)	94.23 (CH)	C-2, C-4a, C-9a	6.37 (1H, *s*)
4a		156.03 (C)		
5	6.94 (1H, *s*)	102.58 (CH)	C-6, C-7, C-8a	6.91 (1H, *s*)
6		152.61 (C)[b]		
6-OH	6.37 (1H, *brs*)[a]			
7		144.25 (C)		
7-OCH_3	4.02 (3H, *s*)	56.49 (OCH_3)	C-7	4.00 (3H, *s*)
8	7.60 (1H, *s*)	104.05 (CH)	C-6, C-7, C-9	
8a		113.34 (C)		
9		179.96 (C=O)		
9a		103.07 (C)		
10a		157.61 (C)[b]		
11	3.49 (2H, *d*, 7.2)	21.37 (CH_2)	C-1, C-2, C-3, C-12, C-13	3.46 (2H, *d*, 6.9)
12	5.31 (1H, *brt*, 6.3)	121.14 (CH)		5.30 (1H, *brt*, 7.0)
13		139.95 (C)		
14	1.84 (3H, *s*)	16.25 (CH_3)	C-12, C-13	1.83 (3H, *s*)
15	2.10 (4H, *m*)	39.70 (CH_2)		2.06 (4H, *m*)
16	2.10 (4H, *m*)	26.29 (CH_2)		2.06 (4H, *m*)

Table 54 (Continued)

Position	W14		HMBC Correlation	cowaxanthone[c]
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})
17	5.06 (*m*, 1H)	123.63(CH)		5.06 (*brt*, 7.0, 1H)
18		132.19 (C)		
19	1.60 (*s*, 3H)	17.71 (CH_3)	C-17, C-18	1.58 (*s*, 3H)
20	1.68 (*s*, 3H)	25.68 (CH_3)	C-17, C-18	1.66 (*s*, 3H)

[a,b] signals interchangeable

[c] ^{1}H NMR 400 MHz of cowaxanthone in $CDCl_3$ (Na Pattalung, *et al*., 1994)

2.3.1.10 Compound W15: 1,3,6-trihydroxy-4-formyl-7-methoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone

Compound **W15** was isolated as a yellow gum. Its UV spectrum (**Figure 126**) (λ_{max} 244, 268, 311 and 352 nm) indicated the presence of a xanthone nucleus. The IR spectrum (**Figure 127**) showed absorption bands at 3412 (OH) and 1643 (*α,β*-unsaturated C=O) cm^{-1}. The high resolution electron ionization mass spectrum (HREI-MS) (**Figure 133**) showed the molecular ion at *m/z* 438.1691 for a molecular formula of $C_{25}H_{26}O_7$. The ^{1}H NMR spectrum (**Figure 128**) (**Table 55**) and ^{13}C NMR spectral data (**Figure 129**) (**Table 55**) were similar to those of **W13** except that a high field aromatic proton [δ_H 6.29, (1H, *s*)] of **W13** was replaced, in **W15**, by a formyl group [δ_H 10.37, (1H, *s*)]. Its ^{13}C NMR spectra (**Figure 129**) (**Table 55**) confirmed the above conclusion by the presence of one additional carbonyl carbon of formyl group at δ_C 190.08. This formyl group was attached to C-4 (δ_C 102.07) due to the HMBC correlations (**Figure 132**) (**Table 55**) with C-3 (δ_C 167.00) and C-4 (δ_C 102.07). In addition, the other substituents were assigned to the same positions as those found in **W13** by the HMBC data. Therefore, compound **W15** was identified as 1,3,6-trihydroxy-4-formyl-7-methoxy-2,8-*bis*(3-methyl-2-butenyl)xanthone. It was a new natural occurring xanthone.

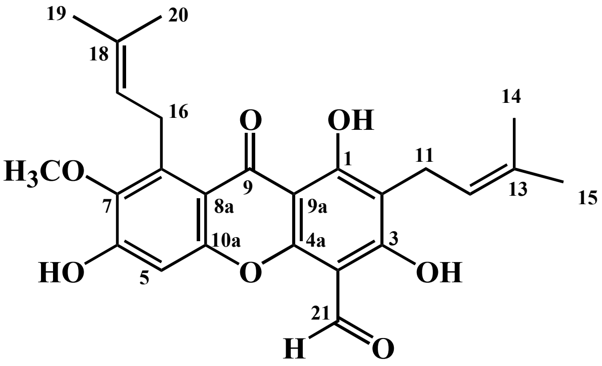

Table 55 The NMR data of compound **W15** in $CDCl_3$

Position	W15		HMBC Correlation
	δ_H (mult., J_{Hz})	δ_C (C-Type)	
1		167.09 (C)	
1-OH	14.52 (1H, *s*)		C-1, C-2, C-9a
2		111.00 (C)	
3		167.00 (C)	
3-OH	13.00 (1H, *s*)		C-2, C-3, C-4
4		102.07 (C)	
4a		157.49 (C)	
5	6.95 (1H, *s*)	101.77 (CH)	C-6, C-7, C-8a, C-9, C-10a
6		154.89 (C)[a]	

Table 55 (Continued)

Position	**W15**		**HMBC Correlation**
	δ_H (mult., J_{Hz})	**δ_C (C-Type)**	
6-OH	6.59 (1H, *brs*)		
7		143.45 (C)	
7- OCH_3	3.83 (3H, *s*)	62.15 (OCH_3)	C-7
8		137.48 (C)	
8a		112.35 (C)	
9		181.32 (C=O)	
9a		102.30 (C)	
10a		155.20 (C)[a]	
11	3.35 (2H, *d*, 7.2)	20.51 (CH_2)	C-2, C-3, C-12, C-13
12	5.23 (2H, *m*)	121.08 (CH)	
13		132.62 (C)	
14	1.81 (3H, *s*)	17.81 (CH_3)	C-12, C-13
15	1.69 (6H, *s*)	25.80 (CH_3)[b]	C-12, C-13
16	4.08 (2H, *d*, 6.3)	26.60 (CH_2)	C-7, C-8, C-8a, C-17, C-18
17	5.23 (2H, *m*)	122.57 (CH)	
18		132.63 (C)	
19	1.69 (6H, *s*)	25.79 (CH_3)[b]	C-17, C-18
20	1.84 (3H, *s*)	18.23 (CH_3)	C-17, C-18
21	10.37 (1H, *s*)	190.08 (CHO)	C-3, C-4

[a,b] signals interchangeable

2.3.1.11 Compound W7: 1,6-dihydroxy-3,5-dimethoxy-2-(3-methyl-2-butenyl)-xanthone

Compound **W7** was obtained as a yellow solid, melting at 178.3-179.0 °C. Its UV spectrum (**Figure 67**) (λ_{max} 246, 282, 318 and 361 nm) showed the presence of a xanthone chromophore. Its IR (**Figure 68**) spectrum showed an absorption band at 3301 (OH), 1649 (α,β-unsaturated C=O) cm^{-1}. The high electron ionization mass spectrum (HREI-MS) (**Figure 74**) showed the molecular ion at *m/z* 356.1250 for a molecular formula of $C_{20}H_{20}O_6$. Its ^1H (**Figure 69**) (**Table 56**) and ^{13}C NMR spectra (**Figure 70**) (**Table 56**) were similar to those of **W6**. Two sets of doublet signals of two-*ortho* aromatic protons [δ_H 7.93 (1H, *d*, J = 8.7 Hz) and 6.99 (1H, *d*, J = 8.7 Hz)] belonged to H-8 and H-7, respectively, according to the HMBC correlation (**Figure 73**) (**Table 56**). The aromatic proton (δ_H 6.99, H-8) showed cross peaks with C-6 (δ_C 154.23), C-9 (δ_C 180.06) and C-10a (δ_C 149.57) while the other one (δ_H 7.93, H-7) showed cross peaks with C-5 (δ_C 133.63), C-6 and C-8a (δ_C 115.14). The signal of the methoxy protons [δ_H 4.02], in the HMBC spectrum (**Figure 73**) (**Table 56**), showed a correlation to C-5, indicating the attachment of the methoxyl group at C-5. In addition, the other substituents were assigned to the same positions as those found in **W6** by the HMBC data. Therefore, compound **W7** was identified as 1,6-dihydroxy-3,5-dimethoxy-2-(3-methyl-2-butenyl)xanthone, which was a new xanthone derivative and differed from **W6** in the location of the methoxyl group at the left-handed aromatic ring.

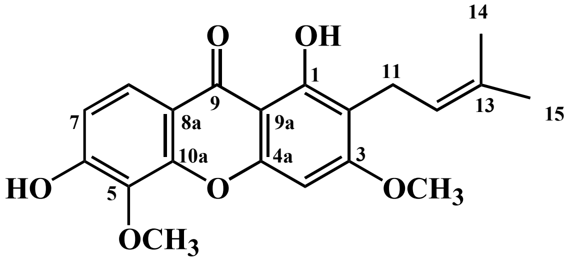

Table 56 The NMR data of compound **W7** in $CDCl_3$

Position	**W7**		**HMBC Correlation**
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**	
1		159.67 (C)	
1-OH	12.98 (1H, *s*)		C-1, C-2, C-9a
2		112.27 (C)	
3		164.06 (C)	
3-OCH_3	3.95 (3H, *s*)	55.97 (OCH_3)	C-3
4	6.48 (1H, *s*)	89.80 (CH)	C-2, C-3, C-4a, C-9, C-9a
4a		155.72 (C)	
5		133.63 (C)	
5-OCH_3	4.12 (3H, *s*)	61.94 (OCH_3)	C-5
6		154.23 (C)[a]	
6-OH	6.58 (1H, *brs*)		
7	6.99 (1H, *d*, 8.7)	112.35 (CH)	C-5, C-6, C-8a
8	7.93 (1H, *d*, 8.7)	121.92 (CH)	C-6, C-9, C-10a
8a		115.14 (C)	
9		180.06 (C=O)	
9a		103.17 (C)	
10a		149.57 (C)[a]	
11	3.37 (2H, *d*, 6.9)	21.32 (CH_2)	C-1, C-3, C-12, C-13
12	5.24 (1H, *mt*, 6.9)	122.03 (CH)	
13		131.93 (C)	
14	1.82 (3H, *s*)	17.79 (CH_3)	C-12, C-13
15	1.70 (3H, *s*)	25.79 (CH_3)	C-12, C-13

[a] signals interchangeable

2.3.1.12 Compound W10: 1,6-dihydroxy-7-methoxy-5,8-*bis*(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone

Compound **W10** was obtained as a yellow-red gum. The UV spectrum (**Figure 89**) showed maximum absorption bands at λ_{max} 243, 279, 289 and 332 nm, indicating that **W10** has a xanthone skeleton, while the IR spectrum (**Figure 90**) showed absorption bands at 3348 and 1648 cm^{-1} for a hydroxyl group and a conjugated carbonyl group. The high resolution electron ionization mass spectrum (HREI-MS) (**Figure 96**) showed the molecular ion at *m*/*z* 476.2191 for a molecular formula of $C_{29}H_{32}O_6$. Its ^{1}H NMR data (**Figure 91**) (**Table 57**) were similar to those of **W2** except for the presence of a dimethylchromene ring [δ_H 6.73 (1H, *d*, *J* = 9.9 Hz), 5.56 (1H, *d, J* = 9.9 Hz) and 1.47 (6H, *s*)]. These suggested that a prenyl group [δ_H 5.28 (2H, *m*), 3.45 (2H, *d*, *J* = 7.2 Hz), 1.83 (3H, *brs*) and 1.77 (3H, *brs*)] and a hydroxyl group [δ_H 6.24 (1H, *brs*)] of the right-handed side in **W2** were replaced, in **W10**, by the dimethylchromene ring. In the HMBC spectrum (**Figure 95**) (**Table 57**), one of olefinic protons in the dimethylchromene ring at δ_H 6.73 (H-11) showed cross peaks with C-1 (δ_C 157.90), C-2 (δ_C 104.39), C-3 (δ_C 159.72) and C-13 (δ_C 77.91) while the other olefinic proton (δ_H 5.56, H-12) showed cross peaks with C-2 and C-13, indicating that the dimethylchromene ring formed an ether linkage with C-3, not C-2. The location of all remaining substituents were established by the HMBC correlations to be the same as those found in **W2**. Thus, compound **W10** was identified as 1,6-dihydroxy-7-methoxy-5,8-*bis*(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone. It was a new naturally occurring xanthone.

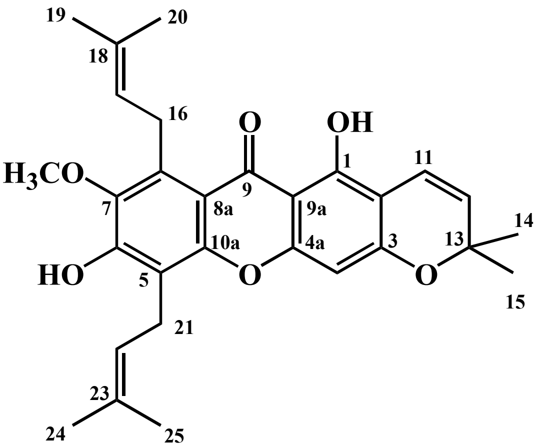

Table 57 The NMR data of compound **W10** in $CDCl_3$

Position	**W10**		**HMBC Correlation**
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**	
1		157.90 (C)	
1-OH	13.77 (1H, *s*)		C-1, C-9a
2		104.39 (C)	
3		159.72 (C)	
4	6.28 (1H, *s*)	94.07 (CH)	C-3, C-4a, C-9, C-9a
4a		156.24 (C)	
5		114.11 (C)	
6		152.40 (C)	
6-OH	6.50 (1H, *brs*)		C-5, C-6, C-7
7		142.35 (C)	
7-OCH_3	3.80 (3H, *s*)	62.00 (OCH_3)	C-7
8		133.79 (C)	
8a		111.88 (C)	
9		182.37 (C=O)	
9a		103.68 (C)	

Table 57 (Continued)

Position	W10		HMBC Correlation
	δ_H (mult., J_{Hz})	δ_C (C-Type)	
10a		153.43 (C)	
11	6.73 (1H, *d*, 9.9)	115.77 (CH)	C-1, C-2, C-3, C-13
12	5.56 (1H, *d*, 9.9)	127.02 (CH)	C-2, C-13
13		77.91 (C)	
14, 15	1.47 (6H, *s*)	28.33 (C)	C-11, C-12, C-13
16	4.05 (2H, *d*, 6.3)	26.37 (CH_2)	C-7, C-8, C-8a, C-17, C-18
17	5.25 (2H, *m*)	123.50 (CH)	
18		131.87 (C)	
19	1.69 (6H, *s*)	25.79 (CH_3)[a]	C-17, C-18
20	1.82 (3H, *s*)	18.21 (CH_3)	C-17, C-18
21	3.55 (2H, *d*, 7.2)	22.64 (CH_2)	C-5, C-6, C-10a, C-22, C-23
22	5.25 (2H, *m*)	121.09 (CH)	
23		132.67 (C)	
24	1.69 (6H, *s*)	25.83 (CH_3)[a]	C-22, C-23
25	1.87 (3H, *s*)	18.00 (CH_3)	C-22, C-23

[a] signals interchangeable

2.3.1.13 Compound W8: 1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone

Compound **W8** was obtained as a brown-yellow solid, melting at 159.8-160.5 ° C. The xanthone chromophore was evident by its UV spectrum (**Figure 75**) with maximum absorption bands at $\lambda_{\max}$ 240, 278, 289 and 330 nm while the hydroxyl and the carbonyl stretching frequencies were found in the region of 3341 and 1642 cm^{-1}, respectively, in the IR spectrum (**Figure 76**). Its ^1H NMR spectral data (**Figure 77**) (**Table 58**) were similar to those of **W10** except that a prenyl unit [δ_H 5.25 (2H, *m*), 3.55 (2H, *d*, J = 7.2 Hz), 1.87 (3H, *s*) and 1.69 (6H, *s*)] of **W10** was replaced, in **W8**, by a lowest-field aromatic proton [δ_H 6.87 (1H, *s*)]. The ^{13}C NMR spectrum (**Figure 78**) (**Table 58**) confirmed the above conclusion by the presence of one additional methine carbon at δ_C 101.74, which correlated to a lowest-field aromatic proton [δ_H 6.87, H-5], in the HMQC spectrum (**Figure 80**). This aromatic proton showed cross peaks with C-7 (δ_C 142.70), C-8a (δ_C 112.08), C-9 (δ_C 181.95) and C-10a (δ_C 155.67), in the HMBC spectrum (**Figure 81**) (**Table 58**), suggesting that this aromatic proton was located at C-5. In addition, the other substituents were located at the same positions as those found in **W10** by the HMBC data. Therefore, compound **W8** was identified as 1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano-(2',3':3,2)xanthone, which was previously isolated from *G. mangostana* (Sen, *et al.*, 1981).

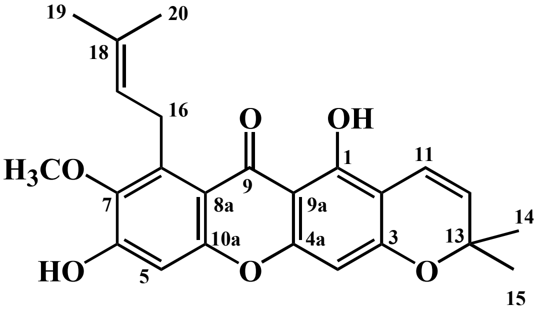

Table 58 The NMR data of compound **W8** and 1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone in $CDCl_3$

Position	**W8**		**HMBC Correlation**	**1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)-xanthone**[b]	
	δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**		δ_H **(mult., J_{Hz})**	δ_C **(C-Type)**
1		157.86 (C)			157.8 (C)
1-OH	13.70 (1H, *s*)		C-1, C-2, C-9a	13.55 (1H, *s*)	
2		104.45 (C)			104.4 (C)
3		159.83 (C)			159.8 (C)
4	6.22 (1H, *s*)	94.16 (CH)	C-2, C-3, C-4a, C-9	6.15 (1H, *s*)	94.0 (CH)
4a		156.22 (C)			156.1 (C)
5	6.87 (1H, *s*)	101.74 (CH)	C-7, C-8a, C-9, C-10a	6.73 (1H, *s*)	101.6 (CH)
6		154.69 (C)[a]			154.5 (C)
6-OH	6.63 (1H, *brs*)			6.27 (1H, *s*)	
7		142.70 (C)			142.7 (C)
7-OCH_3	3.80 (3H, *s*)	61.97 (OCH_3)	C-7	3.73 (3H, *s*)	61.8 (OCH_3)
8		137.00 (C)			136.9 (C)
8a		112.08 (C)			112.1 (C)
9		181.95 (C=O)			181.8 (C=O)
9a		103.68 (C)			103.6 (C)
10a		155.67 (C)[a]			155.6 (C)
11	6.71 (1H, *d*, 9.9)	115.70 (CH)	C-1, C-3, C-13	6.64 (1H, *d*, 10.0)	115.6 (CH)
12	5.56 (1H, *d*, 9.9)	127.13 (CH)	C-2, C-13	5.47 (1H, *d*, 10.0)	126.9 (CH)

Table 58 (Continued)

Position	W8		HMBC Correlation	1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)-xanthone[b]	
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_H (mult., J_{Hz})	δ_C (C-Type)
13		77.94 (C)			77.8 (C)
14, 15	1.46 (6H, *s*)	28.31 (CH_3)	C-12, C-13	1.40 (6H, *s*)	28.3 (CH_3)
16	4.06 (2H, *d*, 6.0)	26.52 (CH_2)	C-7, C-8, C-8a, C-17, C-18	4.01 (2H, *d*, 6.5)	26.5 (CH_2)
17	5.26 (1H, *brt*, 6.3)	123.17 (CH)		5.18 (1H, *t*, 6.5)	123.1 (CH)
18		132.12 (C)			131.8 (C)
19	1.69 (3H, *s*)	25.83 (CH_3)	C-17, C-18	1.62 (3H, *s*)	25.6 (CH_3)
20	1.83 (3H, *s*)	18.23 (CH_3)	C-17, C-18	1.76 (3H, *s*)	18.1 (CH_3)

[a] signals interchangeable

[b] ^{1}H NMR 80 MHz and ^{13}C NMR 15.1 MHz of 1,6-dihydroxy-7-methoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone in $CDCl_3$ (Sen, *et al.*, 1981)

2.3.1.14 Compound W9: 1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone

Compound **W9** was isolated as a yellow solid, melting at 177.5-178.2 °C. Its UV spectrum (**Figure 82**) (λ_{max} 240, 279, 289 and 330 nm) indicated the presence of a xanthone nucleus. The IR spectrum (**Figure 83**) showed absorption bands at 3320 (OH) and 1639 (α,β-unsaturated C=O) cm^{-1}. The ^{1}H NMR data (**Figure 84**) (**Table 59**) were also similar to those of **W8** except for an additional signal of a methoxyl group at δ_H 3.96. The ^{13}C NMR spectral data (**Figure 85**) (**Table 59**) showed the same number and type of quaternary, methine, methylene and methyl carbons as those found in **W8** except for the fact that it contained one more methyl carbon at δ_C 56.06. These data supported the presence of one additional methoxyl group. In the HMBC spectrum (**Figure 88**) (**Table 59**), the methoxy protons gave a cross peak with a quaternary carbon (δ_C 158.17, C-6), indicating the attachment of the methoxyl group at C-6. The remaining HMBC correlations were almost identical to those of **W8**, suggesting that **W9** contained a chelated hydroxyl group at C-1 (δ_C 157.99), the dimethylchromene ring formed an ether linkage with C-3 (δ_C 159.71), two aromatic protons at C-4 (δ_C 93.96) and C-5 (δ_C 98.40), the other methoxyl group at C-7 (δ_C 144.08) and one prenyl group at C-8 (δ_C 137.24). Thus, compound **W9** was assigned as 1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone, the methyl ether of **W8**, which was previously isolated from *G. mangostana* (Sen, *et al.*, 1980).

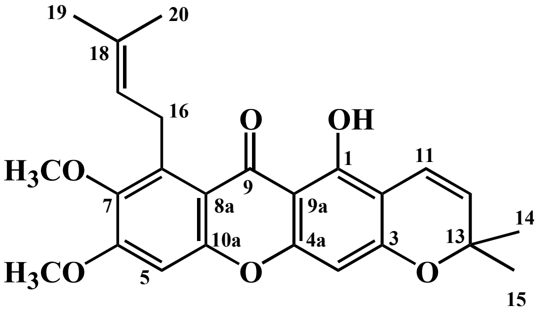

Table 59 The NMR data of compounds **W9** and 1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)xanthone in $CDCl_3$

Position	**W9**		**HMBC Correlation**	**1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)-xanthone** [a]
	δ_H **(mult.,** J_{Hz}**)**	δ_C **(C-Type)**		δ_C **(C-Type)**
1		157.99 (C)		157.8 (C)[b]
1-OH	13.77 (1H, *s*)			
2		104.50 (C)		104.3 (C)
3		159.71 (C)		159.5 (C)[b]
4	6.24 (1H, *s*)	93.96 (CH)	C-3, C-4a, C-9a	93.8 (CH)
4a		156.25 (C)		156.0 (C)[b]
5	6.76 (1H, *s*)	98.40 (CH)	C-6, C-7, C-8a, C-9, C-10a	98.2 (CH)
6		158.17 (C)		158.0 (C)[b]
6-OCH_3	3.96 (3H, *s*)	56.06 (OCH_3)	C-6	55.9 (OCH_3)
7		144.08 (C)		143.9 (C)
7-OCH_3	3.80 (3H, *s*)	60.95 (OCH_3)	C-7	60.8 (OCH_3)
8		137.24 (C)		137.0 (C)
8a		111.91 (C)		111.7 (C)
9		182.07 (C=O)		181.8 (C=O)
9a		103.91 (C)		103.7 (C)
10a		155.41(C)		155.2 (C)[b]
11	6.72 (1H, *d*, 9.9)	115.76 (CH)	C-3, C-13	115.6 (CH)
12	5.57 (1H, *d*, 9.9)	127.10 (CH)	C-2, C-13	126.9 (CH)

Table 59 (Continued)

Position	W9		HMBC Correlation	1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':3,2)-xanthone[a]
	δ_H (mult., J_{Hz})	δ_C (C-Type)		δ_C (C-Type)
13		77.94 (C)		77.8 (C)
14, 15	1.47 (6H, *s*)	28.32 (CH_3)	C-12, C-13	28.2 (CH_3)
16	4.12 (2H, *d*, 6.3)	26.16 (CH_2)	C-7, C-8, C-8a, C-17, C-18	26.0 (CH_2)
17	5.24 (1H, *brt*, 6.9)	123.13 (CH)		123.1 (CH)
18		131.92 (C)		131.5 (C)
19	1.68 (3H, *s*)	25.92 (CH_3)	C-17, C-18	25.8 (CH_3)
20	1.85 (3H, *s*)	18.19 (CH_3)	C-17, C-18	18.1 (CH_3)

[a] ^{13}C NMR 15.1 MHz of 1-hydroxy-6,7-dimethoxy-8-(3-methyl-2-butenyl)-6',6'-dimethylpyrano-(2',3':3,2)xanthone in $CDCl_3$ (Sen, *et al.*, 1980)

[b] tentative assignment

2.3.1.15 Compound W11: 1,6-dihydroxy-3-methoxy-2-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':7,8)xanthone

Compound **W11** was obtained as a yellow solid, melting at 210.0-210.7 °C. The UV spectrum (**Figure 97**) showed maximum absorption bands at λ_{max} 245, 265, 328 and 387 nm, indicating that **W11** has a xanthone chromophore. This was supported by the IR (**Figure 98**) absorption bands at 3266 and 1631 cm^{-1} for a hydroxyl group and a conjugated carbonyl group, respectively. The high resolution electron ionization mass spectrum (HREI-MS) (**Figure 104**) showed the molecular ion at *m*/*z* 408.1555 for a molecular formula of $C_{24}H_{24}O_6$. The ^{1}H NMR spectral data (**Figure 99**) (**Table 60**) were similar to those of **W3** except for the presence of the dimethylchromene ring [δ_H 8.03 (1H, *d*, J = 10.2 Hz), 5.82 (1H, *d,* J = 10.2 Hz) and 1.50 (6H, *s*)]. The lowest-field *cis* olefinic proton [δ_H 8.03, H-16] of the dimethylchromene ring showed cross peaks with C-7 (δ_C 136.77) and C-18 (δ_C 77.21), in the HMBC spectrum (**Figure 103**) (**Table 60**), suggesting that this olefinic proton was located at C-16 in the deshielding zone of a carbonyl group. The other olefinic proton [δ_H 5.82, H-17] of this dimethylchromene ring showed cross peaks with C-8 (δ_C 119.72) and C-18, indicating that the dimethylchromene ring was fused to C-7 and C-8 of the xanthone nucleus with an ether linkage at C-7. In addition, the other substituents were assigned to the same positions as those found in **W3** by the HMBC data. The structure of compound **W11** was elucidated as 1,6-dihydroxy-3-methoxy-2-(3-methyl-2-butenyl)-6',6'-dimethylpyrano(2',3':7,8)xanthone. It was a new natural occurring xanthone.

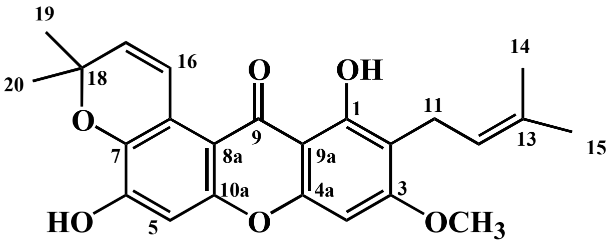

Table 60 The NMR data of compound **W11** in $CDCl_3$

Position	**W11**		**HMBC Correlation**
	δ_H **(mult.,** J_{Hz}**)**	δ_C **(C-Type)**	
1		159.62 (C)	
1-OH	13.34 (1H, *s*)		C-1, C-2, C-9a
2		111.49 (C)	
3		163.62 (C)	
3-OCH_3	3.90 (3H, *s*)	55.85 (OCH_3)	C-3
4	6.36 (1H, *s*)	88.94 (CH)	C-2, C-3, C-4a, C-9a
4a		155.45 (C)	
5	6.82 (1H, *s*)	102.21 (CH)	C-6, C-7, C-8a, C-10a
6		150.76 (C)[a]	
6-OH	6.20 (1H, *brs*)		
7		136.77 (C)	
8		119.72 (C)	
8a		108.79 (C)	
9		182.41 (C=O)	
9a		104.00 (C)	
10a		153.05 (C)[a]	
11	3.35 (2H, *d*, 7.2)	21.35 (CH_2)	C-1, C-2, C-3, C-12, C-13
12	5.23 (1H, *m*)	122.31 (CH)	
13		131.71 (C)	
14	1.80 (3H, *s*)	17.78 (CH_3)	C-12, C-13
15	1.68 (3H, *s*)	25.81 (CH_3)	C-12, C-13
16	8.03 (1H, *d*, 10.2)	121.03 (CH)	C-7, C-18
17	5.82 (1H, *d*, 10.2)	132.20 (CH)	C-8, C-18
18		77.21 (C)	
19, 20	1.50 (6H, *s*)	27.35 (C)	C-17, C-18

[a] signals interchangeable