

2 EXPERIMENTAL

2.1 General Method

Melting points were recorded in °C and were determined on a digital Electrothermal 9100 Melting Point Apparatus. Ultraviolet spectra were measured with UV-160A spectrophotometer (SHIMADZU). Principle bands (λ_{\max}) were recorded as wavelengths (nm) and $\log \epsilon$ in methanol solution. Infrared spectra were obtained on a FTS165 FT-IR spectrophotometer and were recorded in wave number (cm^{-1}). ^1H and ^{13}C -Nuclear magnetic resonance spectra were recorded on a FT-NMR Bruker Ultra Shield™ 300 MHz at Department of Chemistry, Faculty of Science, Prince of Songkla University or Varian UNITY INOVA spectrometer 500 MHz at Central Instrument Facilities, Prince of Songkla University. Spectrum were recorded in deuteriochloroform, hexadeutero-benzene or hexadeutero-dimethyl sulphoxide and were recorded as δ value in ppm down field from TMS (internal standard δ 0.00). High resolution mass spectra were recorded on a MAT 95 XL at Central Instrument Facilities, Prince of Songkla University. Solvent for extraction and chromatography were distilled at their boiling ranges prior to use. For thin layer chromatography, aluminum sheets of silica gel 60 GF254 (20×20 cm, layer thickness 0.2 mm) were used for analytical purposes and the compounds were visualized under ultraviolet light. Column chromatography was performed by using silica gel 100 (70-230 Mesh ASTM, Merck).

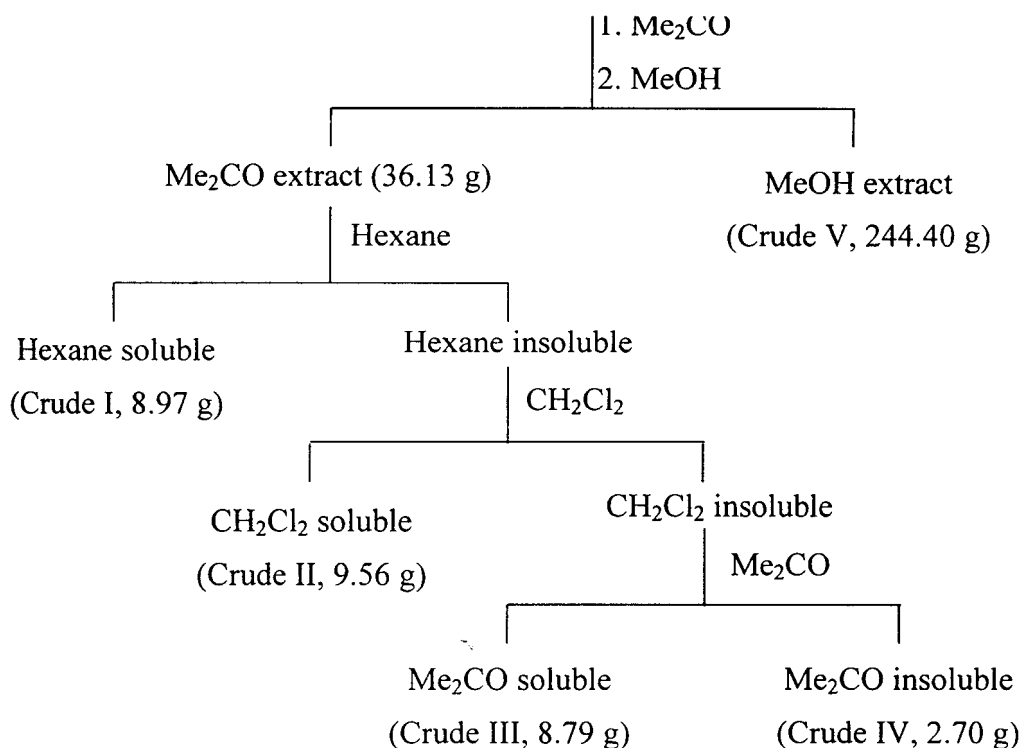
2.2 Plant material

The stems of *M. elliptica* were collected from Pattani province in the Southern part of Thailand. The voucher specimen was identified by Dr. Kitichate Sridith and has been deposited at Prince of Songkla University Herbarium, Biology Department, Faculty of Science, Prince of Songkla University, Thailand.

2.3 Extraction and Isolation

Chopped-dried stems of *M. elliptica* (5.5 kg) were immersed at room temperature in acetone (7 days) and methanol (5 days), successively. After evaporation, the brown solid of acetone extract (36.13 g) and the viscous liquid of methanolic extract (244.40 g) were obtained. The acetone extract (36.13 g) was dissolved in hexane, methylene chloride and acetone to give hexane soluble- (Crude I, 8.97 g), methylene chloride soluble- (Crude II, 9.56 g), acetone soluble- (Crude III, 8.79 g) and acetone insoluble- (Crude IV, 2.70 g) fractions. The process of extraction was shown in Scheme 1.

Chopped-dried stems of *M. elliptica* (5.5 Kg)



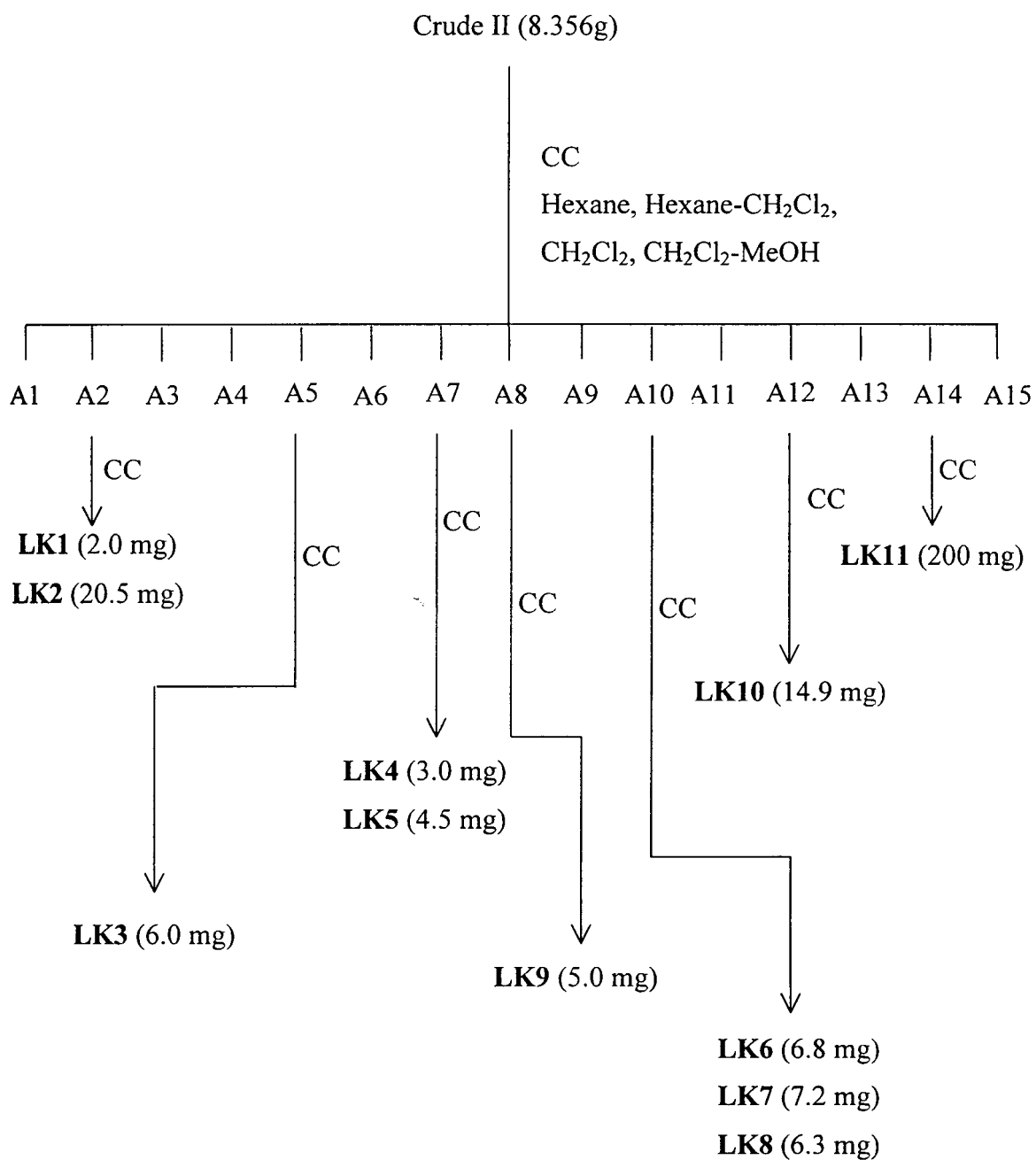
Scheme 1 Extraction of Crude I-V from stems of *M. elliptica*

2.3.1 Purification of Crude II

Crude II (8.536 g) was subjected to column chromatography using silica gel as the stationary phase and eluted with hexane, hexane-methylene chloride, methylene chloride and methylene chloride-methanol. On the basis of their TLC characteristic, the fractions which contained the same major components were combined to give fractions A1-A15 (Table 2). The selected fractions were further purified to give eleven pure compounds as shown in Scheme 2.

Table 2 Physical characteristic and weight of the fractions obtained from CC

Fraction	Weight	Physical characteristic
A1	1.512	Yellow liquid
A2	0.181	Yellow solid mixed with yellow liquid
A3	0.221	Yellow solid mixed with yellow liquid
A4	0.352	Yellow solid mixed with yellow liquid
A5	0.055	Yellow solid
A6	0.104	Yellow solid
A7	0.137	Yellow solid mixed with orange solid
A8	0.222	Yellow solid mixed with orange solid
A9	0.178	Orange viscous liquid
A10	0.100	Yellow solid mixed with orange solid
A11	0.446	Orange viscous liquid
A12	0.194	Yellow solid mixed with orange viscous liquid
A13	0.299	Yellow solid mixed with orange viscous liquid
A14	0.355	Yellow solid mixed with orange viscous liquid
A15	2.351	Brown viscous liquid



Scheme 2 Isolation of **LK1-LK11** from Crude II

Isolation of LK1 and LK2

Fraction A2 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (1:9). The yellow solid of **LK1** (2.0 mg) and the yellow solid of **LK2** (20.5 mg) were obtained.

LK1

Melting point : 185-186 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 225 (4.06), 245 (4.38), 252 (4.41), 280 (3.95), 329 (3.26), 406 (3.75)

IR (KBr) ν (cm⁻¹) : 3448 (O-H stretching), 1672, 1636 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 12.99 (1H, *s*), 8.32 (1H, *m*), 8.30 (1H, *m*), 7.80 (2H, *m*), 7.77 (1H, *d*, *J* = 8.0 Hz), 7.55 (1H, *td*, *J* = 0.5, 8.0 Hz), 2.40 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 189.01 (C=O), 182.46 (C=O), 161.09 (C), 137.27 (CH), 135.00 (C), 134.56 (CH), 134.00 (CH), 133.81 (C), 133.29 (C), 131.34 (C), 127.32 (CH), 126.88 (CH), 119.26 (CH), 115.25 (C), 16.19 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 16.19

CH : 137.27, 134.56, 134.00, 127.32, 126.88, 119.26

LK2

Melting point : 186-187 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 239 (4.40), 244 (4.39), 274 (4.38), 406 (3.81)

IR (KBr) ν (cm⁻¹) : 3441 (O-H stretching), 1673, 1630 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.08 (1H, *s*), 8.28 (1H, *dd*, *J* = 2.0, 6.0 Hz), 8.26 (1H, *dd*, *J* = 2.0, 6.0 Hz), 7.77 (1H, *dt*, *J* = 2.0, 6.0 Hz), 7.79 (1H, *dt*, *J* = 2.0, 6.0 Hz), 7.28 (1H, *s*), 4.91 (2H, *s*), 1.60 (6H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 186.90 (C=O), 181.99 (C=O), 160.24 (C), 158.21 (C), 134.10 (CH), 134.05 (CH), 133.43 (C), 133.35 (C), 133.22 (C), 133.10 (C), 127.30 (CH), 126.61 (CH), 109.56 (C), 109.12 (CH), 101.21 (C), 57.53 (CH₂), 24.63 (2x CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 24.63

CH₂ : 57.53

CH : 134.10, 134.05, 127.30, 126.61, 109.12

EIMS m/z (% relative intensity) : 310 ([M]⁺, 3), 254 (5), 253 (6), 252 (100), 224 (5),
196 (20), 168 (13), 139 (23), 114 (5), 104 (3), 61 (11)

HR-MS m/z 310.0827 [M]⁺ for C₁₈H₁₄O₅ (calcd. 310.0841)

Isolation of LK3

Fraction A5 was rechromatographed on silica gel column using methylene chloride-hexane (3:7) as the eluent to give **LK3** as a yellow solid (6.0 mg).

Melting point : 212-213 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 247 (4.33), 278 (4.30), 314 (3.97), 375 (3.64)

IR (KBr) ν (cm⁻¹) : 3446 (O-H stretching), 1673, 1642 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 12.29 (1H, *s*), 10.47 (1H, *s*), 8.31 (1H, *ddd*, $J = 0.5, 1.5, 7.5$ Hz), 8.26 (1H, *ddd*, $J = 0.5, 1.5, 7.5$ Hz), 7.84 (1H, *dt*, $J = 1.5, 7.5$ Hz), 7.78 (1H, *dt*, $J = 1.5, 7.5$ Hz), 7.68 (1H, *s*), 4.13 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 195.48 (C=O), 181.92 (C=O), 180.17 (C=O), 166.62 (2xC), 141.63 (CH), 134.74 (CH), 133.69 (CH), 133.67 (C), 132.46 (C), 127.39 (CH), 127.11 (CH), 118.05 (C), 117.66 (C), 113.11 (CH), 64.74 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 64.74

CH : 195.48, 134.74, 133.69, 127.39, 127.11, 113.11

Isolation of LK4 and LK5

Fraction A7 was rechromatographed on column chromatography and eluted with mixed solvent of methylene chloride-hexane to give six fractions (A7.1-A7.6).

Fraction A7.4 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (2:8). The yellow solid of **LK4** (3.0 mg) were collected.

Fraction A7.5 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (3:7) to afford **LK5** as a yellow solid (4.5 mg).

LK4

Melting point : 166-168 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 217 (3.38), 257 (3.77), 278 (3.97), 307 (3.65), 429 (3.65)

IR (KBr) ν (cm⁻¹) : 3423 (O-H stretching), 1618, 1582 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.30 (1H, *s*), 9.39 (1H, *s*), 8.29 (1H, *dd*, *J* = 2.0, 6.0 Hz), 8.27 (1H, *dd*, *J* = 2.0, 6.0 Hz), 7.79 (1H, *dt*, *J* = 2.0, 6.0 Hz), 7.77 (1H, *dt*, *J* = 2.0, 6.0 Hz), 7.35 (1H, *s*), 4.94 (2H, *s*), 3.57 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 186.92 (C=O), 182.24 (C=O), 164.07 (C), 161.86 (C), 134.13 (CH), 134.11 (CH), 133.54 (C), 133.49 (C), 127.34 (CH), 126.71 (CH), 114.33 (2xC), 109.74 (CH), 68.98 (CH₂), 59.35 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 59.35

CH₂ : 68.98

CH : 134.13, 134.11, 127.34, 126.71, 109.74

LK5

Melting point : 198-199 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 242 (4.16), 278 (4.12), 407 (3.57)

IR (KBr) ν (cm⁻¹) : 3447 (O-H stretching), 1671, 1630 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.45 (1H, *s*), 8.35 (1H, *m*), 8.32 (1H, *m*), 7.94 (1H, *d*, *J* = 8.0 Hz), 7.91 (1H, *d*, *J* = 16.5 Hz), 7.86 (1H, *d*, *J* = 8.0 Hz), 7.85 (2H, *m*), 6.97 (1H, *d*, *J* = 16.5 Hz), 2.45 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 198.59 (C=O), 189.03 (C=O), 181.90 (C=O), 161.40 (C), 135.69 (CH), 135.04 (CH), 133.52 (CH), 132.90 (C), 130.52 (CH), 129.66 (C), 127.52 (CH), 127.09 (CH), 119.17 (CH), 116.38 (C), 27.58 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 27.58

CH : 135.69, 135.04, 135.01, 134.39, 130.53, 127.52,
127.09, 119.17

EIMS m/z (% relative intensity) : 293 ([M+1]⁺, 1), 292 ([M]⁺, 4), 277 (7), 252 (1),
250 (39), 249 (100), 222 (1), 221 (4), 220 (2), 194 (1), 193 (4), 166 (2), 165 (11),
139 (2), 110 (2), 105 (4), 96 (2), 82 (4) 61 (9)

HR-MS m/z : 292.0785 for C₁₈H₄O₁₂ (calcd. 292.0735)

Isolation of LK6, LK7 and LK8

Fraction A10 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (4:1) to afford compounds **LK6** (6.8 mg), **LK7** (7.2 mg) and **LK8** (6.3 mg) as yellow solid.

LK6

Melting point : 218-219 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 224 (4.46), 250 (4.34), 266 (4.36), 291 (4.21), 318
(3.78), 413 (4.00)

IR (KBr) ν (cm⁻¹) : 3387 (O-H stretching), 1668, 1632 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.04 (1H, *s*), 8.15 (1H, *d*, $J = 8.5$ Hz), 7.80 (2H, *br s*),
7.37 (1H, *d*, $J = 8.5$ Hz), 6.78 (1H, *s*), 4.64 (2H, *s*), 4.03 (3H, *s*), 3.51 (3H, *s*)

¹H NMR (CDCl₃ + C₆D₆) (δ ppm) : 13.03 (1H, *s*), 8.11 (1H, *d*, $J = 8.5$ Hz), 7.78 (1H,
d, $J = 8.0$ Hz), 7.76 (1H, *d*, $J = 8.0$ Hz), 7.31 (1H, *d*, $J = 8.5$ Hz), 6.70 (1H, *s*), 4.61
(2H, *s*), 3.97 (3H, *s*), 3.48 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 187.78 (C=O), 181.81 (C=O), 159.46 (C), 156.40 (C),
146.86 (CH), 134.44 (CH), 133.94 (C), 133.24 (C), 126.95 (C), 125.95 (CH),
125.57 (CH), 120.07 (CH), 118.98 (CH), 114.87 (C), 68.57 (CH₂), 62.36 (CH₃)
58.90 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 62.36, 58.90

CH₂ : 68.57

CH : 134.44, 125.57, 120.07, 118.98

EIMS m/z (% relative intensity) : 315 ($[M+1]^+$,15), 314 ($[M]^+$,70), 299 (100),
284 (90), 266 (41), 238 (27), 225 (26), 197 (14), 128 (15), 69 (16), 61 (76)

HR-MS m/z : 314.0819 for $C_{17}H_{14}O_6$ (calcd. 314.0790)

LK7

Melting point : 197-198 °C

UV (CH_3OH) λ_{max} nm (log ϵ) : 248 (5.11), 268 (5.07), 355 (4.52)

IR (KBr) ν (cm^{-1}) : 3483 (O-H stretching), 1668 (C=O stretching)

1H NMR ($CDCl_3$) (δ ppm) : 8.11(1H, d , $J = 8.0$ Hz), 8.09 (1H, d , $J = 8.5$ Hz), 7.86
(1H, dt , $J = 0.5, 8.0$ Hz), 7.35 (1H, d , $J = 8.5$ Hz), 6.66 (1H, s), 4.65 (2H, s), 4.02
(3H, s), 3.94 (3H, s), 3.50 (3H, s),

^{13}C NMR ($CDCl_3$) (δ ppm) : 182.52 (C=O), 181.51 (C=O), 157.92 (C), 154.83 (C),
145.94 (C), 140.31 (C), 135.75 (C), 133.53 (CH), 128.73 (C), 125.53 (CH), 124.99
(C), 124.84 (C), 123.41 (CH), 120.40 (CH), 68.99 (CH_2), 62.27 (CH_3), 62.15
(CH_3), 58.83 (CH_3)

DEPT 135° ($CDCl_3$) (δ ppm) CH_3 : 62.27, 62.15, 58.83

CH_2 : 68.99

CH : 133.53, 125.53, 123.41, 120.40

EIMS m/z (% relative intensity) : 330 ($[M+2]^+$, 2), 329 ($[M+1]^+$,10), 328 ($[M]^+$, 49),
313 (100), 295 (24), 283 (31), 265 (15), 222 (19), 221 (6), 139 (7), 127 (2), 61 (13)

HR-MS m/z : 328.0948 for $C_{18}H_{16}O_6$ (calcd. 328.0947)

LK8

Melting point : 280-282 °C

UV (CH_3OH) λ_{max} nm (log ϵ) : 233 (4.01), 252 (4.36), 280 (3.89), 326 (3.14), 406
(3.72)

IR (KBr) ν (cm^{-1}) : 3446 (O-H stretching), 1671, 1635 (C=O stretching)

^1H NMR (CDCl_3) (δ ppm) : 13.06 (1H, *s*), 8.34 (1H, *dd*, $J = 1.5, 8.5$ Hz), 8.33 (1H, *dd*, $J = 1.5, 8.5$ Hz), 7.88 (1H, *d*, $J = 8.0$ Hz), 7.84 (2H, *m*), 7.79 (1H, *dd*, $J = 1.0, 8.0$ Hz), 4.87 (2H, *d*, $J = 4.5$ Hz), 2.36 (1H, *t*, $J = 4.5$ Hz)

^{13}C NMR (CDCl_3) (δ ppm) : 189.08 (C=O), 182.25 (C=O), 160.22 (C), 136.36 (C), 134.81 (CH), 134.75 (CH), 134.17 (CH), 133.72 (C), 133.15 (C), 132.46 (C), 127.48 (CH), 126.94 (CH), 119.50 (CH), 115.00 (C), 60.86 (CH_2)

DEPT 135° (CDCl_3) (δ ppm) CH_2 : 60.86

CH : 134.81, 134.75, 134.17, 127.48, 126.94, 119.50

Isolation of LK9

Fraction A8 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (1:1). The major component **LK9** was obtained. The yellow solid of **LK9** was collected (5.0 mg).

Melting point : 280-282 °C

UV (CH_3OH) λ_{max} nm ($\log \epsilon$) : 223 (4.31), 251 (4.18), 266 (4.28), 293 (4.06), 412 (3.88)

IR (KBr) ν (cm^{-1}) : 3339 (O-H stretching), 1671, 1631 (C=O stretching)

^1H NMR (CDCl_3) (δ ppm) : 13.04 (1H, *s*), 8.15 (1H, *d*, $J = 8.5$ Hz), 7.52 (1H, *td*, $J = 0.5, 8.0$ Hz), 7.71 (1H, *d*, $J = 8.0$ Hz), 7.36 (1H, *d*, $J = 8.5$ Hz), 6.76 (1H, *s*), 4.03 (3H, *s*), 2.37 (3H, *s*)

^{13}C NMR (CDCl_3) (δ ppm) : 187.82 (C=O), 182.00 (C=O), 160.62 (C), 155.92 (C), 146.78 (C), 136.93 (CH), 134.45 (C), 132.32 (C), 128.34 (C), 127.78 (C), 125.52 (CH), 119.97 (CH), 118.93 (CH), 114.66 (C), 68.57 (C H_3), 62.36 (CH_3)

DEPT 135° (CDCl_3) (δ ppm) CH_3 : 68.57, 62.36

CH : 136.93, 125.52, 119.97, 118.93

Isolation of LK10

Fraction A12 was separated on column chromatography and eluted with methylene chloride-hexane (8:2). The major component, yellow solid of **LK10** was collected (14.9 mg).

Melting point : 271-273 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 216 (4.62), 268 (4.53), 293 (4.28), 411 (3.94)

IR (KBr) ν (cm⁻¹) : 3405 (O-H stretching), 1663, 1636 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.20 (1H, *s*), 8.16 (1H, *d*, *J* = 8.7 Hz), 7.68 (1H, *d*, *J* = 7.5 Hz), 7.65 (1H, *d*, *J* = 2.4 Hz), 7.48 (1H, *d*, *J* = 7.5 Hz), 7.20 (1H, *dd*, *J* = 2.4, 8.7 Hz), 5.80 (1H, *br s*), 2.35 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 188.12 (C=O), 182.65 (C=O), 163.78 (C), 160.79 (C), 136.37 (CH), 135.94 (C), 134.74 (C), 129.67 (CH), 121.36 (CH), 134.74 (C), 129.67 (CH), 121.36 (CH), 118.79 (C), 113.28 (CH), 16.06 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 16.06

CH : 136.37, 129.67, 121.36, 118.79, 113.28

Isolation of LK11

Fraction A14 was chromatographed on column chromatography and eluted with methylene chloride followed by increasing small amount of methanol in methylene chloride. **LK11** was obtained as a yellow solid (200 mg).

Melting point : 178-180 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 240 (4.51), 244 (4.51), 279 (4.47), 416 (3.87)

IR (KBr) ν (cm⁻¹) : 3445 (O-H stretching), 1660, 1623 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.20 (1H, *s*), 8.22 (1H, *dd*, *J* = 2.5, 7.5 Hz), 8.15 (1H, *dd*, *J* = 2.5, 7.5 Hz), 7.93 (1H, *dt*, *J* = 2.5, 7.5 Hz), 7.89 (1H, *dt*, *J* = 2.5, 7.5 Hz), 7.25 (1H, *s*), 4.54 (2H, *s*)

^{13}C NMR (CDCl_3) (δ ppm) : 186.49 (C=O), 182.17 (C), 164.05 (C), 163.51 (C), 135.05 (CH), 134.88 (CH), 133.65 (C), 133.32 (C), 133.13 (C), 127.14 (CH), 126.76 (CH), 120.59 (C), 109.38 (C), 108.29 (CH), 51.71 (CH_2)

DEPT 135° (CDCl_3) (δ ppm) CH_2 : 51.71

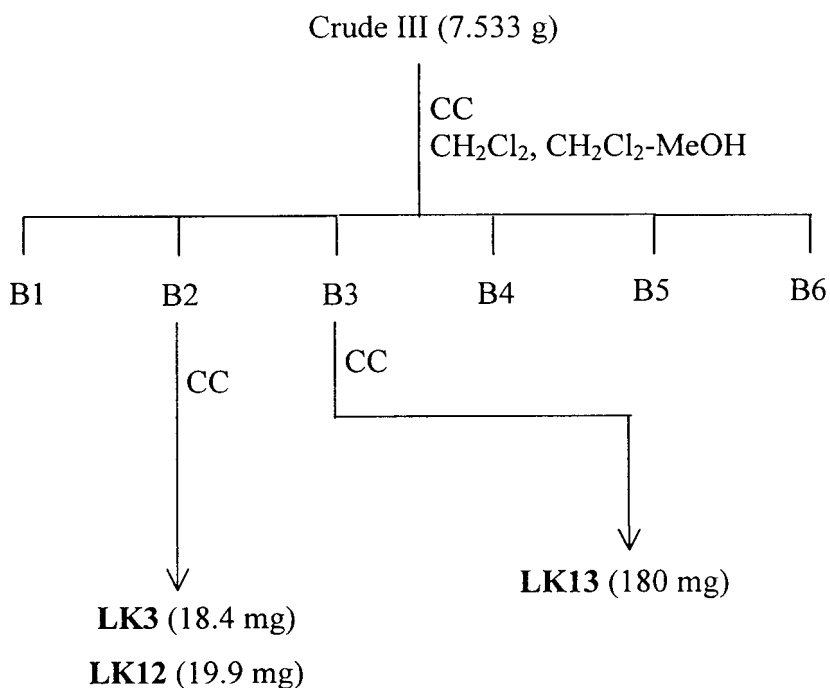
CH : 135.05, 134.88, 127.14, 126.76, 108.29

2.3.2 Purification of Crude III

Crude III (7.533 g) was subjected to column chromatography using silica gel as the stationary phase and eluted with methylene chloride and methylene chloride-methanol. On the basis of their TLC characteristic, the fractions which contained the same major components were combined to give fractions B1-B6 (Table 3). The selected fractions were further purified to afford three pure compounds as shown in Scheme 3.

Table 3 Physical characteristic and weight of fractions obtained from CC

Fraction	Weight (g)	Physical characteristic
B1	1.437	Yellow solid mixed with yellow liquid
B2	0.190	Yellow solid
B3	0.524	Yellow solid
B4	0.150	Yellow solid
B5	1.207	Yellow solid mixed with brown viscous liquid
B6	1.857	Yellow solid mixed with brown viscous liquid



Scheme 3 Isolation of **LK3**, **LK12**, **LK13** from Crude III

Isolation of compound **LK3** and **LK12**

Fraction B2 was rechromatographed on column chromatography and eluted with methylene chloride-hexane (1:1). The yellow solid of **LK3** (18.4 mg) and **LK12** (19.9 mg) were obtained.

LK12

Melting point : 288-289 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 239 (4.40), 243 (4.41), 277 (4.45), 408 (3.82)

IR (KBr) ν (cm⁻¹) : 3396 (O-H stretching), 1660, 1625 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 13.10 (1H, *s*), 8.18 (1H, *dd*, *J* = 2.0, 7.5 Hz), 8.11 (1H, *dd*, *J* = 2.0, 7.5 Hz), 7.89 (1H, *dt*, *J* = 2.0, 7.5 Hz), 7.86 (1H, *dt*, *J* = 2.0, 7.5 Hz), 7.22 (1H, *s*), 2.40 (3H, *s*),

¹³C NMR (CDCl₃) (δ ppm) : 186.59 (C=O), 182.28 (C=O), 163.28 (C), 162.81 (C), 134.96 (CH), 134.84 (CH), 133.35 (C), 133.22 (C), 132.09 (C), 127.08 (CH), 126.75 (CH), 117.81 (CH), 109.29 (C), 107.76 (CH), 8.41 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 8.41

CH : 134.96, 134.84, 127.08, 126.75, 107.76

Isolation of compound LK13

Fraction B3 was further purified by column chromatography and eluted with methylene chloride-hexane (1:1) to give **LK13** as a yellow solid (0.18 g).

Melting point : 290-292 °C

UV (CH₃OH) λ_{\max} nm (log ϵ) : 236 (4.52), 243 (4.48), 277 (4.72), 365 (3.86)

IR (KBr) ν (cm⁻¹) : 3431 (O-H stretching), 1672, 1650 (C=O stretching)

¹H NMR (CDCl₃) (δ ppm) : 8.15 (1H, *dd*, J = 1.5, 7.5 Hz), 8.10 (1H, *dd*, J = 1.5, 7.5 Hz), 7.89 (1H, *dt*, J = 1.5, 7.5 Hz), 7.83 (1H, *dt*, J = 1.5, 7.5 Hz), 7.50 (1H, *s*), 3.56 (1H, *s*), 2.15 (3H, *s*)

¹³C NMR (CDCl₃) (δ ppm) : 183.07 (C=O), 180.65 (C=O), 162.04 (C), 161.06 (C), 134.99 (CH), 134.17 (C), 133.82 (CH), 132.49 (C), 127.09 (CH), 126.65 (C), 126.49 (CH), 118.37 (C), 109.44 (CH), 61.05 (CH₃), 9.47 (CH₃)

DEPT 135° (CDCl₃) (δ ppm) CH₃ : 61.05, 9.47

CH : 134.99, 133.82, 127.09, 126.49, 109.44