

## NEOFLAVONES. 1. NATURAL DISTRIBUTION AND SPECTRAL AND BIOLOGICAL PROPERTIES

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*Data on neoflavones isolated from 58 plants belonging to the Clusiaceae, Fabaceae, Rubiaceae, Thelypteridaceae, Passifloraceae, Asteraceae, and Rutaceae families from the literature up to 2002 were systematized and reviewed. A list of 131 4-phenylcoumarins with information on the plant sources, structures, physicochemical data, and pharmacological properties with references to the original literature was composed.*

**Keywords:** Clusiaceae, Fabaceae, Rubiaceae, Thelypteridaceae, Passifloraceae, Asteraceae, Rutaceae, flavonoids, neoflavones, 4-phenylcoumarins

Flavonoids are one of the most widely distributed groups of phenolic compounds with the structural formula  $C_6-C_3-C_6$ . The flavonoid molecule consists of two phenyl rings bonded to a 3-carbon aliphatic moiety. Most flavonoids can be viewed as chromone or chromane derivatives containing an aryl group in the 2, 3, or 4 position.

Neoflavonoids represent a group of natural compounds that also belongs to the flavonoid class. They are based on the 4-phenylcoumarin skeleton. Neoflavonoids include 4-arylcoumarins (neoflavones), 4-arylchromanes, dalbergiones, and dalbergiquinolins. A total of 130 representatives of natural 4-arylcoumarins is known (in 2001). The unique structure, broad popularity in traditional plant medicines containing these compounds, and the valuable pharmacological properties make neoflavones highly interesting.

### NATURAL DISTRIBUTION

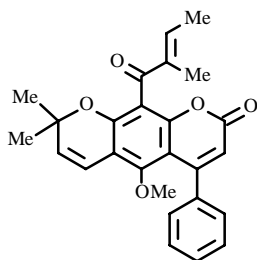
The first neoflavone isolated from natural sources was calophyllolide (**73**). It was isolated in 1951 from extracts of *Calophyllum inophyllum* seeds [1]. Its structure was established in 1957. Neoflavones were recognized as a new group of natural compounds in the mid-60s after several compounds based on 4-phenylcoumarin were isolated from plants of the *Dalbergia* and *Mammea* genera. Neoflavones were isolated from 58 plants of the Clusiaceae, Fabaceae, Rubiaceae, Asteraceae, Thelypteridaceae, Passifloraceae, and Rutaceae families. Dalbergin (**28**) is the most common and widely distributed neoflavone in the plant kingdom.

Data for natural 4-phenylcoumarins have been reviewed [2-4]. We present physicochemical data for neoflavones isolated from plant sources.

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## PHYSICAL CONSTANTS AND SPECTRAL DATA OF NEOFLAVONES



\*refined structure [5]

### 1. Apetatolide\*

*Calophyllum apetalum* [6, 7], *C. inophyllum* [8]

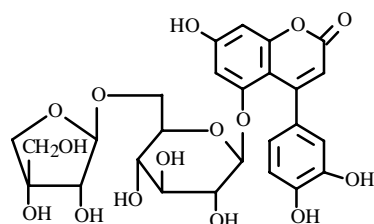
$C_{26}H_{24}O_5$ , mp 203-205°C [7]

UV [7]: 236 (4.55), 275 (4.40)

Mass [7]: 416, 401, 361

PMR (60 MHz,  $CDCl_3$ ) [7]: 1.38 (6H, s), 1.92 (3H, d,  $J = 7$ ), 1.95 (3H, s), 3.03 (3H, s), 5.67 (1H, d,  $J = 10$ ), 6.05 (1H, s), 6.50 (1H, d,  $J = 10$ ), 6.60 (1H, m)

Biol: antitumor activity [9]



### 2. 5-O-[\beta-D-Apiofuranosyl-(1-6)-\beta-D-glucopyranosyl]-7,3',4'-trihydroxy-4-phenylcoumarin

*Coutarea hexandra* [10]

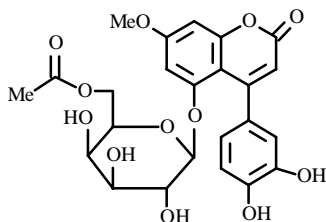
$C_{26}H_{28}O_{15}$

Mass [10]: 579  $[M - H]^+$ , 447, 285

PMR (250 MHz,  $CD_3OD$ ) [10]: 2.62 (1H, dd,  $J = 7.5$ ,  $J = 7.5$ , G-2''), 3.90 (1H, d,  $J = 2$ , A-2''), 4.72 (1H, d,  $J = 7.5$ , G-1''), 4.93 (1H, d,  $J = 2$ , A-1''), 5.84 (1H, s, H-3), 6.45 (1H, d,  $J = 2$ , H-8), 6.50 (1H, d,  $J = 2$ , H-6), 6.69 (1H, dd,  $J = 2$ ,  $J = 7.5$ , H-6'), 6.80 (1H, d,  $J = 7.5$ , H-5'), 6.80 (1H, d,  $J = 2$ , H-2').

$^{13}C$  NMR (69.5 MHz,  $CD_3OD$ ) [10]:

C-2	163.5	C-8a	157.3	G-1''	101.6	A-1''	111.0
3	112.3	1'	133.2	2''	74.6	2''	77.2
4	158.3	2'	116.9	3''	78.1	3''	80.5
4a	105.1	3'	145.5	4''	71.4	4''	75.1
5	158.1	4'	146.9	5''	77.7	5''	66.0
6	98.7	5'	115.9	6''	68.6		
7	163.3	6'	120.7				
8	101.1						



### 3. 5-O-(6''-Acetyl)-\beta-D-galactopyranosyl-3',4'-dihydroxy-7-methoxy-4-phenylcoumarin

*Exostema caribaeum* [11], *Hintonia latiflora* [12]

$C_{24}H_{24}O_{12}$ , mp 215-220°C [11]

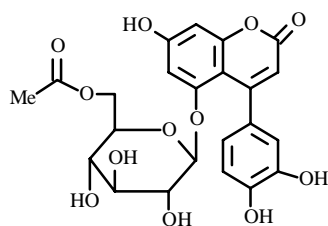
UV (MeOH) [11]: 218, 258, 328

IR (KBr) [11]: 3467, 3409, 3395, 2918, 1730, 1695, 1536, 1614, 1442, 1355, 1258, 1072, 1047.

PMR (80 MHz,  $DMSO-d_6$ ) [11]: 1.96 (3H, s,  $CH_3CO$ ), 2.90-3.6 (6H, m, H-2'', H-3'', H-4'', H-5'', H-6''), 3.75 (3H, s, 7-OMe), 4.63 (1H, d,  $J = 8$ , H-1''), 5.76 (1H, s, H-3), 6.5-6.8 (5H, m, H-6, H-8, H-2', H-5', H-6')

$^{13}\text{C}$  NMR (50 MHz, DMSO- $d_6$ ) [11]:

C-2	159.46	C-7	162.66	C-4'	145.75	C-4''	68.22
3	112.28	8	95.05	5'	115.54	C-5''	72.81
4	156.37	8a	155.67	6'	119.17	C-6''	63.52
4a	103.50	1'	130.44	1''	100.80	OMe-7	55.8
5	155.36	2'	114.76	2''	70.06	CH <sub>3</sub> CO	20.35
6	98.94	3'	144.13	3''	72.66	CH <sub>3</sub> CO	170.12



#### 4. 5-O-(6''-Acetyl- $\beta$ -D-glucopyranosyl)-7,3',4'-trihydroxy-4-phenylcoumarin

*Hintonia latiflora* [13]

$\text{C}_{23}\text{H}_{22}\text{O}_{12}$ , mp 205-208°C [13]

$[\alpha]_{\text{D}} -129^\circ$  (MeOH) [13]

UV (MeOH) [13]: 258, 331

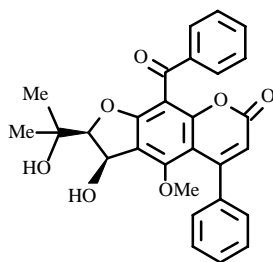
IR (KBr) [13]: 3440, 1701, 1618, 1602, 1384, 1368, 1215, 1083

PMR (300 MHz, DMSO- $d_6$ ) [13]: 2.00 (3H, s, MeCO), 2.60 (1H, dd,  $J = 8$ , H-2''), 3.02 (1H, dd,  $J = 8$ , H-4''), 3.18 (1H, dd,  $J = 8$ , H-3''), 3.41 (1H, dd,  $J = 8.3$ , H-5''), 4.05 (1H, m, H-6''), 4.68 (1H, d,  $J = 8$ , H-1'), 5.58 (1H, s, H-3), 6.25 (1H, d,  $J = 2.3$ , H-8), 6.32 (1H, d,  $J = 2.3$ , H-8), 6.58-6.80 (3H, m, H-2', H-5', H-6')

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ ) [13]:

C-2	160.0	C-7	165.0	C-4'	144.2	C-4''	69.6
3	109.3	8	97.4	5'	114.5	C-5''	73.8
4	155.9	8a	156.1	6'	119.2	C-6''	63.2
4a	100.0	1'	130.8	1''	100.2	OMe-7	55.8
5	156.8	2'	115.6	2''	72.9	Ac	20.6
6	99.8	3'	145.7	3''	76.0	Ac	170.4

#### 5. 9-Benzoyl-6-hydroxy-5-methoxy-4-phenylnodakenetin



*Calophyllum teysmannii* var. *inophyllolide* [14]

$\text{C}_{28}\text{H}_{24}\text{O}_7$ , mp 206-208°C [14]

$[\alpha]_{\text{D}} -8.4^\circ$  ( $\text{CHCl}_3$ ) [14]

UV (EtOH) [14]: 254, 294, 328

IR (KBr) [14]: 3420, 1740, 1609, 1563, 1470, 1382, 1274, 1123, 1015, 965, 706, 656

Mass [14]: 472 (5)  $[\text{M}]^+$ , 454 (4), 439 (4), 397 (100), 368 (50), 319 (40)

HREIMS [14]: 472.1514  $[\text{M}]^+$

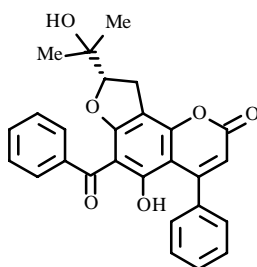
PMR (300 MHz,  $\text{CDCl}_3$ ) [14]: 1.27 (3H, s,  $\text{CH}_3$ -13), 1.48 (3H, s,  $\text{CH}_3$ -12), 3.60 (3H, s,  $\text{CH}_3$ O-5), 4.28 (1H, d,  $J = 6.1$ , H-7), 5.55 (1H, d,  $J = 6.1$ , H-6), 6.00 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 7.47 (2H, t,  $J = 8.0$ , H-3'', H-5''), 7.60 (1H, tt,  $J = 8.0$ ,  $J = 1.2$ , H-4''), 7.89 (2H, dd,  $J = 8.0$ ,  $J = 1.2$ , H-2'', H-6'')

NOE, HMQC, HMBC [14]

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) [14]:

C-2	159.1	C-7	90.3	C-13	28.3	C-1''	137.6
3	113.5	8a	161.6	1'	139.8	2''	129.7
4*	155.2	9**	106.2	2'	126.9	3''	128.6
4a**	106.0	9a*	155.6	3'	128.0	4''	133.8
5	156.6	10	190.9	4'	127.6	5''	128.6
5a	115.9	11	72.8	5'	128.0	6''	129.7
6	71.2	12	24.7	6'	126.9	OMe-7	59.4

\*,\*\*Assignments may be reversed



### 6. (-)-6-Benzoyl-5-hydroxy-4-phenylcolumbianin

*Calophyllum teysmannii* var. *inophyllolide* [14]

$\text{C}_{27}\text{H}_{22}\text{O}_6$

$[\alpha]_{\text{D}} -80.5^\circ$  ( $\text{CHCl}_3$ ) [14]

UV (EtOH) [14]: 238, 296, 348

IR (KBr) [14]: 3446, 1745, 1617, 1540, 1470, 1389, 1123, 918, 698

Mass [14]: 442 (30)  $[\text{M}]^+$ , 424 (20), 409 (24), 383 (100)

HREIMS [14]: 442.1423  $[\text{M}]^+$

PMR (300 MHz,  $\text{CDCl}_3$ ) [14]: 0.94 (3H, s,  $\text{CH}_3$ -13), 1.10 (3H, s,  $\text{CH}_3$ -12), 3.17 (1H, dd,  $J = 9.1$ ,  $J = 15.5$ ,  $\text{CH}_2$ -9b), 3.23 (1H, dd,  $J = 9.4$ ,  $J = 15.5$ ,  $\text{CH}_2$ -9a), 4.57 (1H, dd,  $J = 9.1$ ,  $J = 9.4$ , H-8), 5.99 (1H, s, H-3), 7.37 (2H, m, H-2', H-6'), 7.43 (5H, m, H-3', H-4', H-5', H-3'', H-5''), 7.53 (3H, m, H-2'', H-4'', H-6''), 13.08 (1H, s, OH-5)

NOE, HMQC, HMBC [14]

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) [14]:

C-2	160.0	C-8	92.8	C-13	23.9	C-1''	140.4
3	112.4	9	27.0	1'	139.0	2''	127.6
4*	156.2	9a	105.8	2'	127.3	3''	127.7
4a	102.5	9b*	156.3	3'	128.0	4''	131.7
5**	163.5	10	199.1	4'	128.4	5''	127.7
6	102.9	11	71.3	5'	128.0	6''	127.6
6a**	163.7	12	23.9	6'	127.3		

\*,\*\* Assignments may be reversed

### 7. 9-Benzoyl-5-methoxy-4-phenylnodakenetin

*Calophyllum teysmannii* var. *inophyllolide* [14]

$\text{C}_{28}\text{H}_{24}\text{O}_6$ , mp 208-210°C [14]

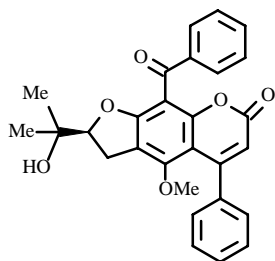
$[\alpha]_{\text{D}} -18.1^\circ$  ( $\text{CHCl}_3$ ) [14]

UV (EtOH) [14]: 224, 252, 292, 336

IR (KBr) [14]: 3450, 1733, 1602, 1563, 1470, 1389, 1266, 1115, 953, 691

Mass [14]: 456 (5)  $[\text{M}]^+$ , 398 (100)

HREIMS [14]: 456.1551  $[\text{M}]^+$



PMR (300 MHz,  $\text{CDCl}_3$ ) [14]: 1.13 (3H, s,  $\text{CH}_3$ -13), 1.14 (3H, s,  $\text{CH}_3$ -12), 3.23 (1H, dd,  $J = 9.4$ ,  $J = 15.4$ ,  $\text{CH}_2$ -6b), 3.30 (3H, s,  $\text{CH}_3$ -O-5), 3.31 (1H, dd,  $J = 8.1$ ,  $J = 15.5$ ,  $\text{CH}_2$ -6a), 4.66 (1H, dd,  $J = 8.1$ ,  $J = 9.4$ , H-7), 5.98 (1H, s, H-3), 7.30 (2H,

m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 7.47 (2H, t, J = 7.9, H-3'', H-5''), 7.59 (1H, tt, J = 7.9, J = 1.2, H-4''), 7.89 (2H, dd, J = 7.9, J = 1.2, H-2'', H-6'')

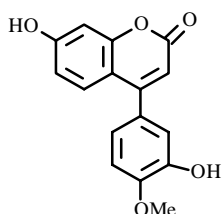
**NOE, HMQC, HMBC** [14]

**X-ray** [14]

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) [14]:

C-2	159.4	C-8a	161.8	C-13	24.5	C-1''	137.4
3	113.4	9	106.8	1'	139.5	2''	129.7
4*	155.2	9a	153.8	2'	127.0	3''	128.6
4a	106.4	10	190.8	3'	127.6	4''	133.8
5*	154.9	11	71.3	4'	128.4	5''	128.6
5a	114.4	11	71.4	5'	127.6	6''	129.7
6	28.3	12	25.6	6'	127.0	CH <sub>3</sub> O-5	59.3
7	91.4						

\*Assignments may be reversed



### 8. Volubolin

*Dalbergia volubilis* [15, 16]

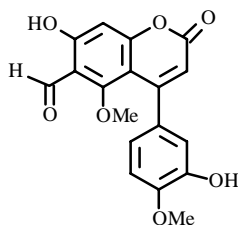
C<sub>16</sub>H<sub>12</sub>O<sub>5</sub>

**UV** (MeOH) [16]: 224 (4.19), 284 (4.40), 340 (3.98)

**IR** (KBr) [16]: 3300, 1715, 1480, 1345, 1140, 1100, 1085, 1005, 935, 810

**Mass** [16]: 284 (21.9), 272 (40.0), 270 (21.2), 269 (26.8), 268 (89.4), 267 (13.6), 256 (25.6), 255 (14.4), 241 (14.1), 240 (41.6), 239 (12.1), 230 (17.8), 229 (17.3), 228 (45.6), 227 (18.1), 226 (18.4), 225 (54.9), 213 (33.5), 212 (41.0), 211 (38.4), 210 (90.2), 209 (24.7), 208 (16.0), 207 (30.5), 205 (13.6), 201 (26.0)

**PMR** (60 MHz, CDCl<sub>3</sub>) [16]: 3.8 (3H, s, OMe), 5.93 (1H, s, H-3), 6.7 (3H, m, H-5, H-6, H-8), 7.2 (3H, m, H-2', H-5', H-6'), 9.85 (1H, s, OH), 10.3 (1H, s, OH)



### 9. Voludal

*Dalbergia volubilis* [16, 17]

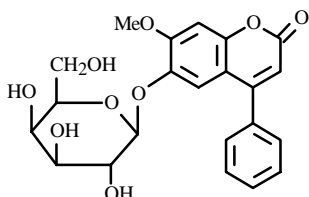
C<sub>18</sub>H<sub>14</sub>O<sub>7</sub>

**UV** (MeOH) [16]: 220 (4.16), 288 (4.8), 343 (3.9)

**IR** (KBr) [16]: 3300, 3010, 2755, 1715, 1690, 1625, 1605, 1585, 1545, 1460, 1390, 1240, 1150

**Mass** [16]: 343 (2.9), 342 (17.9), 314 (3.3), 313 (9.1), 286 (3.0), 285 (4.3), 284 (7.4), 281 (2.7), 270 (8.1), 269 (4.0), 268 (12.1), 267 (2.7), 257 (3.0), 256 (12.1), 255 (3.3), 250 (2.7)

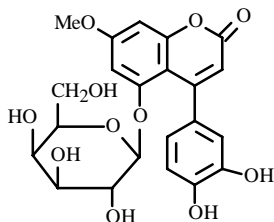
**PMR** (60 MHz, CDCl<sub>3</sub>) [16]: 3.8 (3H, s, OMe), 4.0 (3H, s, OMe), 5.95 (1H, s, H-3), 7.3 (3H, m, H-2', H-5', H-6'), 7.5 (1H, s, H-8), 9.72 (1H, s, CHO), 9.85 (1H, s, OH), 10.2 (1H, s, OH)



### 10. 6-O-β-D-Galactopyranosyldalbergin

*Hesperethusa crenulata* [18]

C<sub>22</sub>H<sub>22</sub>O<sub>9</sub>



**11. 5-O- $\beta$ -D-Galactopyranosyl-3',4'-dihydroxy-7-methoxy-4-phenylcoumarin**

*Exostema caribaeum* [19], *Hintonia latiflora* [12]

$C_{22}H_{22}O_{11}$ , mp 228-231°C [19]

UV (MeOH) [19]: 208, 255, 330

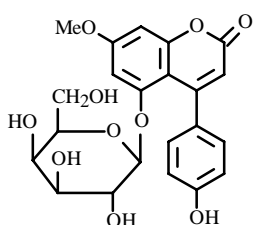
IR (KBr) [19]: 3650, 3403, 1719, 1613, 1160, 1078, 1049

PMR (80 MHz,  $C_5H_5N-d_6$ ) [19]: 3.50 (4H, s, OH), 3.67 (3H, s, 7-OMe), 4.0-4.5 (6H, m, H-2'', H-3'', H-4'', H-5'', H-6''), 5.26 (1H, d, J = 8, H-1''), 6.20 (1H, s, H-3), 6.57 (1H, d, J = 3, H-8), 6.92 (1H, dd, J = 8, J = 3, H-6'), 7.08 (1H, d, J = 3, H-6), 7.20 (1H, d, J = 8, H-5'), 7.38 (1H, d, J = 3, H-2')

$^{13}C$  NMR (50 MHz, DMSO- $d_6$ ) [19]:

C-2	159.52	C-7	162.70	C-4'	145.71	C-3''	73.00
3	112.20	8	95.22	5'	115.51	4''	68.10
4	156.30	8a	156.00	6'	119.30	5''	75.90
4a	103.30	1'	130.44	1''	101.12	6''	60.43
5	155.44	2'	114.70	2''	70.13	OMe-7	55.90
6	98.50	3'	144.10				

**Biol:** antimicrobial activity against *Candida albicans* at MIC 12.5  $\mu$ g/mL [11]



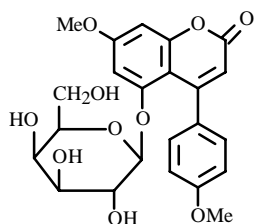
**12. 5-O- $\beta$ -D-Galactopyranosyl-4'-hydroxy-7-methoxy-4-phenylcoumarin**

*Hintonia latiflora* [12]

$C_{22}H_{22}O_{10}$ , mp 218-221°C [12]

IR (KBr) [12]: 3460, 3330, 1720, 1675, 1615, 1515, 1485, 1440, 1280, 970

PMR (90 MHz, DMSO- $d_6$ ) [12]: 3-4.40 (6H, m, H-2''—H-6''), 3.83 (3H, s, 7-OMe), 4.60 (1H, d, J = 7, H-1''), 5.78 (1H, s, H-3), 6.58 (1H, d, J = 3, H-8), 6.65 (1H, d, J = 3, H-6), 6.73 (2H, d, J = 8, H-3', H-5'), 7.16 (2H, d, J = 8, H-2', H-6'), 9.5 (1H, s, 4'-OH)



**13. 5-O- $\beta$ -D-Galactopyranosyl-7,4'-dimethoxy-4-phenylcoumarin**

*Exostema caribaeum* [11]

$C_{23}H_{24}O_{10}$ , mp 217-221°C [11]

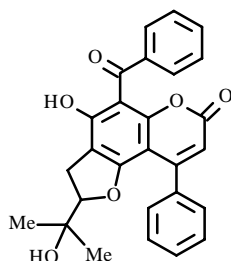
UV (MeOH) [11]: 212, 236

IR (KBr) [11]: 3500, 3200, 1690, 1671, 1614, 1593, 1512, 1444, 1367, 1252, 1205, 1120, 1068, 831

PMR (80 MHz, DMSO- $d_6$ ) [11]: 3.84 (6H, s, OMe-7, OMe-4'), 3.0-4.03 (6H, m, H-2''—H-6''), 4.54 (1H, d, J = 8, H-1''), 5.92 (1H, s, H-3), 6.53 (1H, d, J = 3, H-6), 6.62 (1H, d, J = 3, H-8), 6.87 (2H, d, J = 8, H-3', H-5'), 7.20 (2H, d, J = 8, H-2', H-6')

$^{13}\text{C}$  NMR (50 MHz, DMSO- $d_6$ ) [11]:

C-2	160.00	C-7	162.69	C-4'	157.60	C-3''	73.30
3	112.31	8	95.14	5'	114.20	4''	68.05
4	156.35	8a	155.97	6'	129.37	5''	75.85
4a	103.26	1'	129.80	1''	100.90	6''	60.46
5	155.54	2'	129.37	2''	69.78	OMe-7	55.91
6	98.36	3'	114.20			OMe-4'	55.91



#### 14. Hydrohydroxyisocalanone

*Calophyllum teysmannii* var. *inophylloide* [20]

UV [20]: 236, 256, 318

IR [20]: 3454, 3061, 2925, 2854, 1739, 1603, 1446, 1382, 1283, 1140, 854, 768, 698, 606

Mass [20]: 442 (42)  $[\text{M}]^+$ , 424 (6), 409 (30), 383 (83), 105 (100), 77 (77)

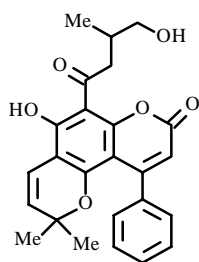
HREIMS [20]: 442.1427  $[\text{M}]^+$

PMR (500 MHz,  $\text{CDCl}_3$ ) [20]: 0.96 (3H, s,  $\text{CH}_3$ -12), 1.05 (3H, s,  $\text{CH}_3$ -13), 3.01 (1H, dd,  $J = 8.8$ ,  $J = 15.5$ , H-7b), 3.14 (1H, dd,  $J = 9.8$ ,  $J = 15.5$ , H-7a), 4.56 (1H, dd,  $J = 8.8$ ,  $J = 9.8$ , H-6), 5.97 (1H, s, H-3), 7.33 (2H, m, H-2', H-6'), 7.44 (3H, m, H-3', H-4', H-5'), 7.49 (2H, t,  $J = 8.0$ , H-3'', H-5''), 7.59 (1H, dd,  $J = 8.0$ ,  $J = 1.5$ , H-4''), 7.66 (2H, dd,  $J = 8.0$ ,  $J = 1.5$ , H-2'', H-6''), 12.18 (1H, s, OH)

HMQC, HMBC [20]

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) [20]:

C-2	158.2	C-7a	110.0	C-13	24.9	C-1''	140.3
3	111.9	8	161.6	1'	137.9	2''	128.2
4	154.2	9	104.8	2'	127.6	3''	128.2
4a	98.9	9a	156.4	3'	128.9	4''	132.4
4b	162.1	10	198.9	4'	127.9	5''	128.2
6	92.8	11	71.6	5'	128.9	6''	128.2
7	26.9	12	23.1	6'	127.6		



#### 15. 7-Hydroxy-8-(4-hydroxy-3-methyl-1-oxobutyl)-4-phenyl-2',2'-dimethyl-2H,6H-benzo[1,2-b:3,4-b']-dipyran-2-one

*Kielmeyera reticulata* [21]

$\text{C}_{25}\text{H}_{24}\text{O}_6$

$[\alpha]_D -1.92^\circ$  ( $\text{CHCl}_3$ ) [21]

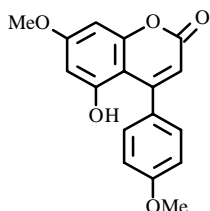
Mass [21]: 402 (30)  $[\text{M} - \text{H}_2\text{O}]^+$ , 388 (28), 387 (100), 359 (6), 331 (16)

PMR (300 MHz,  $\text{CDCl}_3$ ) [21]: 0.95 (3H, s,  $\text{CH}_3$ -6'), 0.97 (3H, s,  $\text{CH}_3$ -5'), 1.01 (3H, d,  $J = 6.7$ ,  $\text{CH}_3$ -5''), 1.83 (1H, s, OH-4'), 2.50 (1H, m, H-3''), 2.71 (1H, dd,  $J = 9.1$ ,  $J = 13.9$ , H-2a''), 3.42 (1H, dd,  $J = 9.0$ ,  $J = 10.5$ , H-4b''), 3.79 (1H, dd,  $J = 5.0$ ,  $J = 10.5$ , H-4a''), 3.82 (1H, dd,  $J = 4.3$ ,  $J = 13.9$ , H-2b''), 5.40 (1H, d,  $J = 10.1$ , H-3'), 6.02 (1H, s, H-3), 6.62 (2H, d,  $J = 10.1$ , H-4'), 7.22 (2H, m, Ph-4), 7.40 (3H, m, Ph-4), 14.67 (1H, s, OH-7)

$^1\text{H}$ - $^{13}\text{C}$  COSY [21]

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) [21]:

C-2	160.1	C-8	104.7	C-6'	28.0	C-1'''	140.4
3	112.2	8a	157.2	1''	206.7	2'''	127.6
4	157.3	2'	79.8	2''	49.4	3'''	128.2
4a	102.8	3'	127.4	3''	34.0	4'''	128.4
5	157.2	4'	115.8	4''	68.7	5'''	128.2
6	106.5	5'	28.0	5''	17.0	6'''	127.6
7	164.6						



### 16. 5-Hydroxy-7,4'-dimethoxy-4-phenylcoumarin

*Exostema acuminatum* [22]

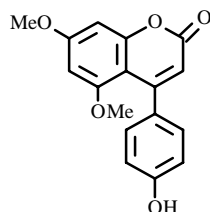
$\text{C}_{17}\text{H}_{14}\text{O}_5$ , mp 210-212°C [22], 212°C [23]

UV (MeOH) [23]: 223, 261, 326

$^{13}\text{C}$  NMR (125.7 MHz,  $\text{CDCl}_3$ ) [22]:

C-2	161.1	C-6	95.9	C-1'	132.4	C-5'	114.3
3	112.6	7	163.3	2'	128.9	6'	128.9
4	157.3	8	96.6	3'	114.3	OMe	55.8
4a	103.7	8a	155.5	4'	158.3	OMe	55.4
5	155.6						

**Biol:** cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V<sup>+</sup>, KB-V<sup>-</sup>, LNCaP, SW626, SKNSH, and M109 [22]



### 17. 4'-Hydroxy-5,7-dimethoxy-4-phenylcoumarin

*Coutarea hexandra* [24, 25], *Exostema mexicanum* [26]

$\text{C}_{17}\text{H}_{14}\text{O}_5$ , mp 214-215°C [24]

UV (MeOH) [27]: 254 (4.09), 322 (4.05); UV (MeOH+MeONa): 254, 328, 372

IR (KBr) [27]: 3300, 1706, 1612, 1159, 1048, 962, 835

Mass [24]: 298 (100)  $[\text{M}]^+$ , 270 (82), 255 (29), 227 (15)

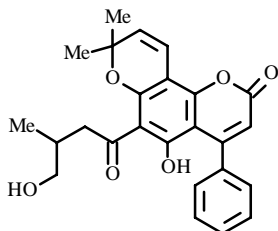
PMR [200 MHz,  $(\text{CD}_3)_2\text{CO}$ ] [27]: 3.56 (3H, s, OMe-5), 3.96 (3H, s, OMe-7), 5.84 (1H, s, H-3), 6.40 (1H, d, J = 2.5, H-6), 6.56 (1H, d, J = 2.5, H-8), 6.92 (2H, d, J = 8.5, H-3', H-5'), 7.16 (2H, d, J = 8.5, H-2', H-6'), 8.56 (1H, s, OH-4')

$^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO-d}_6$ ) [28]:

C-2	159.4	C-6	95.7	C-1'	129.6	C-5'	113.9
3	111.2	7	162.8	2'	128.6	6'	128.6
4	155.1	8	93.6	3'	113.9	OMe	55.5
4a	102.6	8a	156.4	4'	157.3	OMe	55.7
5	157.9						

**Biol:** antiplasmodial activity against *Plasmodium falciparum* [26]





### 18. Hydroxymammeigin

*Kielmeyera elata* [29]

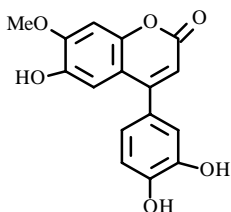
$C_{25}H_{24}O_6$ , mp 174-176°C [29]

UV (MeOH) [29]: 232 (4.53), 284 (4.60), 335 (3.94); (MeOH+NaOH): 252 (4.45), 308 (4.48), 405 (3.78); (MeOH+NaOAc): 232 (4.48), 286 (4.51), 336 (4.07)

IR (KBr) [29]: 3440, 1740, 1720, 1600, 1590, 1380, 1260, 1125, 780, 750, 700

Mass [29]: 420 (2)  $[M]^+$ , 402 (65), 387 (100), 359 (5), 334 (12), 331 (16), 303 (4), 165 (5), 77 (5)

PMR (400 MHz,  $CDCl_3$ ) [29]: 0.98 (3H, d,  $J = 6.8$ , H-5''), 1.57 (6H, s, H-5', H-6'), 2.35 (1H, m, H-3''), 3.05 (1H, dd,  $J = 16.4$ ,  $J = 6.6$ , H-2a''), 3.20 (1H, dd,  $J = 16.4$ ,  $J = 6.9$ , H-2b''), 3.50 (1H, dd,  $J = 10.4$ ,  $J = 6.3$ , H-4a''), 3.57 (1H, dd,  $J = 10.6$ ,  $J = 5.3$ , H-4b''), 5.62 (1H, d,  $J = 10.1$ , H-2'), 5.98 (1H, s, H-3), 6.88 (1H, d,  $J = 10.1$ , H-1'), 7.30-7.38 (5H, m, Ph-4), 14.55 (1H, s, OH-5)



### 19. 3'-Hydroxymelanetin

*Dalbergia odorifera* [30, 31]

$C_{16}H_{12}O_6$ , mp 297-299°C [30]

UV (MeOH) [30]: 235 (4.274), 256 (4.063), 296 (3.934), 346 (4.179)

Mass [30]: 300 (76)  $[M]^+$ , 272 (68), 257 (63), 187 (12), 155 (16), 136 (14), 127 (15), 115 (36), 114 (18), 113 (27), 102 (14), 87 (16), 77 (34), 69 (100), 63 (41), 53 (36), 51 (35)

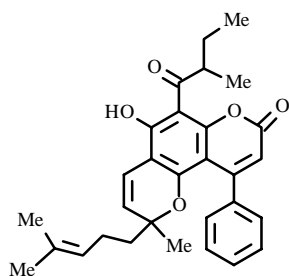
HRMS [30]: 300.0633

PMR (300 MHz,  $DMSO-d_6$ ) [30]: 3.87 (3H, s, OMe-7), 6.05 (1H, s, H-3), 6.79 (1H, dd,  $J = 8$ ,  $J = 1.8$ , H-6'), 6.86 (1H, d,  $J = 1.8$ , H-2'), 6.88 (1H, d,  $J = 8$ , H-5'), 6.98 (1H, s, H-5), 7.07 (1H, s, H-8), 9.39 (1H, s, OH-3'), 9.43 (1H, s, OH-6), 9.52 (1H, s, OH-4')

NOESY, HMQC, HMBC [30]

$^{13}C$  NMR (75.4 MHz,  $DMSO-d_6$ ) [30]:

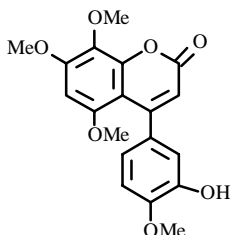
C-2	160.99	C-6	143.79	C-10	111.49	C-4'	147.41
3	110.59	7	152.12	1'	126.68	5'	120.26
4	155.72	8	100.89	2'	145.81	6'	116.08
5	111.11	9	148.83	3'	116.26	7-OMe	56.55



### 20. 5-Hydroxy-2-methyl-6-(2-methylbutanoyl)-2-(4-methyl-3-pentenyl)-10-phenyl-2H,8H-pyrano[2,3-f]chromen-8-one

*Mesua ferrea* [32]

$C_{30}H_{32}O_5$



## 21. 3'-Hydroxy-5,7,8,4'-tetramethoxy-4-phenylcoumarin

*Coutarea hexandra* [33]

$C_{19}H_{18}O_7$ , mp 161-162°C [33]

UV (MeOH) [33]: 263 (4.18), 324 (4.09)

IR (CDCl<sub>3</sub>) [33]: 3540, 1718, 1610, 1595, 1513, 1348, 1280, 1244, 1109, 1060, 862

Mass [33]: 358 (100) [M]<sup>+</sup>, 343 (43), 330 (3), 315 (18), 287 (14), 179 (3), 165 (2)

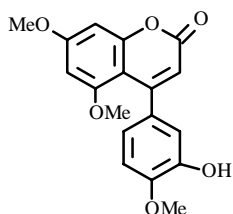
PMR [300 MHz, (CD<sub>3</sub>)<sub>2</sub>CO] [33]: 3.56 (3H, s, 5-OMe), 3.83 (3H, s, 8-OMe), 3.90 (3H, s, 4'-OMe), 3.99 (3H, s, 7-OMe), 5.87 (1H, s, H-3), 6.64 (1H, s, H-6), 6.79 (1H, dd, J = 8.2, J = 2, H-6'), 6.87 (1H, d, J = 2, H-2'), 6.98 (1H, d, J = 8.2, H-5'), 7.75 (1H, s, 3'-OH)

<sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>) [33]:

C-2	159.3	C-6	94.0	C-2'	114.9	8-OMe	60.7
3*	111.5	7**	155.3	3'	145.5	OMe	56.3
4**	155.7	8	129.8	4'	147.7	OMe	56.1
4a	102.8	8a	144.6	5'*	111.2	OMe	55.6
5	153.6	1'	132.0	6'	129.8		

\*,\*\* Assignments may be reversed

Biol: adenosine-3',5'-cyclomonophosphate-phosphodiesterase inhibitor at IC<sub>50</sub> 12.5×10<sup>-5</sup> M [34]



## 22. 3'-Hydroxy-5,7,4'-trimethoxy-4-phenylcoumarin

*Coutarea hexandra* [24], *Exostema acuminatum* [22], *E. mexicanum* [26]

$C_{18}H_{16}O_6$ , mp 153-154°C [24, 27]

UV (MeOH) [24]: 252 (4.13), 329 (4.32)

IR (CHCl<sub>3</sub>) [24]: 3525, 1710, 1615, 1597, 1510, 1158, 1111, 1052, 945, 909, 859, 828

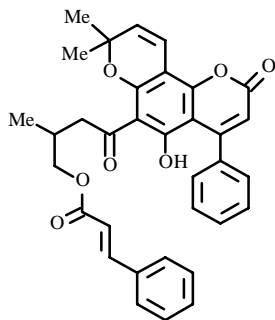
Mass [24]: 328 (100) [M]<sup>+</sup>, 300 (51), 285 (31), 257 (15)

PMR (CDCl<sub>3</sub>) [24]: 3.42 (3H, s), 3.78 (3H, s), 3.86 (3H, s), 5.87 (1H, s), 5.92 (1H, s), 6.6-6.9 (4H, m)

<sup>13</sup>C NMR (125.7 MHz, DMSO-d<sub>6</sub>) [22]:

C-2	161.0	C-6	95.9	C-2'	114.2	C-6'	119.1
3	109.6	7	163.6	3'	144.6	OMe	56.0
4	157.2	8	93.6	4'	146.6	OMe	55.7
4a	103.6	8a	155.4	5'	112.6	OMe	55.6
5	160.0	1'	133.1				

Biol: cytotoxic activity against cell lines BC1, Lu1, Col2, KB-V<sup>+</sup>, KB-V<sup>-</sup>, SW626, SKNSH, and M109 [2]; antiplasmodial activity against *Plasmodium falciparum* [26]



**23. 5-Hydroxy-6-(4-cinnamoyl-3-methyl-1-oxobutyl)-4-phenyl-2',2'-dimethyl-2H,6H-benzo[1,2-b:3,4-b']-dipyran-2-one**

*Kiameyera reticulata* [21], *K. argentea* [35]

$C_{34}H_{30}O_7$

$[\alpha]_D -6.6^\circ$  (CHCl<sub>3</sub>) [21]

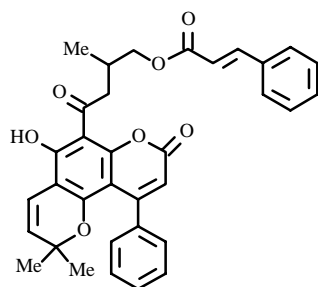
**Mass** [21]: 550 (7) [M]<sup>+</sup>, 402 (31), 388 (19), 387 (82), 347 (10), 331 (28), 147 (52), 131 (100), 103 (56)

**PMR** (300 MHz, CDCl<sub>3</sub>) [21]: 1.06 (3H, d, J = 6.7, CH<sub>3</sub>-5''), 1.55 (3H, s, CH<sub>3</sub>-6'), 1.57 (3H, s, CH<sub>3</sub>-5'), 2.65 (1H, m, H-3''), 3.05 (1H, dd, J = 7.3, H = 17.0, H-2a''), 3.25 (1H, dd, J = 6.1, J = 17.0, H-2b''), 4.16 (2H, d, J = 6.1, CH<sub>2</sub>-4''), 5.64 (1H, d, J = 10.1, H-3'), 5.96 (1H, s, H-3), 6.38 (1H, d, J = 16.0, H-2C), 6.88 (1H, d, J = 10.1, H-4'), 7.26 (2H, m, Ph-4), 7.38 (6H, m, Ph-4, cinnamoyl), 7.46 (2H, m, cinnamoyl), 7.62 (1H, d, J = 16.0, H-3C), 14.65 (1H, s, OH-5)

**<sup>1</sup>H-<sup>13</sup>C COSY** [21]

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) [21]:

C-2	159.9	C-2'	80.7	C-5''	17.8	C-2C	118.4
3	113.4	3'	127.0	1'''	139.8	3C	145.5
4	156.8	4'	116.1	2'''	127.6	4C	134.9
4a	102.8	5'	28.8	3'''	128.2	5C	128.6
5	164.8	6'	28.7	4'''	128.8	6C	129.5
6	107.9	1''	206.0	5'''	128.2	7C	130.9
7	155.5	2''	49.1	6'''	127.6	8C	129.5
8	102.2	3''	30.0	1C	167.3	9C	128.6
8a	158.7	4''	69.3				



**24. 7-Hydroxy-8-(4-cinnamoyl-3-methyl-1-oxobutyl)-4-phenyl-2',2'-dimethyl-2H,6H-benzo[1,2-b:3,4-b']-dipyran-2-one**

*Kiameyera reticulata* [21], *K. argentea* [35]

$C_{34}H_{30}O_7$

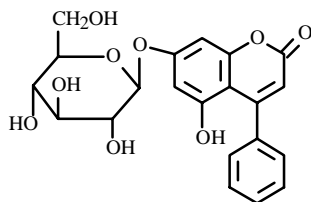
$[\alpha]_D -7.14^\circ$  (CHCl<sub>3</sub>) [21]

**Mass** [21]: 550 (3) [M]<sup>+</sup>, 535 (13), 402 (19), 388 (26), 387 (100), 331 (20), 148 (28), 147 (43), 131 (36), 103 (33)

**PMR** (300 MHz, CDCl<sub>3</sub>) [21]: 0.92 (3H, s, CH<sub>3</sub>-6'), 0.93 (3H, s, CH<sub>3</sub>-5'), 1.18 (3H, d, J = 6.8, CH<sub>3</sub>-5''), 2.71 (1H, m, H-3''), 3.28-3.48 (2H, m, CH<sub>2</sub>-2''), 4.17-4.29 (2H, m, CH<sub>2</sub>-4''), 5.37 (1H, d, J = 10.0, H-3'), 6.01 (1H, s, H-3), 6.41 (1H, d, J = 16.0, H-2C), 6.60 (1H, d, J = 10.0, H-4'), 7.22 (2H, m, Ph-4), 7.37 (6H, m, Ph-4, cinnamoyl), 7.52 (2H, m, cinnamoyl), 7.64 (1H, d, J = 16.0, H-3C), 14.45 (1H, s, OH-7)

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) [21]:

C-2	159.2	C-2'	79.7	C-5''	17.9	C-2C	118.6
3	112.6	3'	127.3	1'''	140.5	3C	145.2
4	156.5	4'	115.8	2'''	127.7	4C	135.1
4a	102.9	5'	28.0	3'''	128.1	5C	128.7
5	157.0	6'	28.0	4'''	128.3	6C	129.3
6	106.4	1''	205.4	5'''	128.1	7C	130.7
7	164.0	2''	49.3	6'''	127.7	8C	129.3
8	104.8	3''	30.8	1C	167.4	9C	128.7
8a	157.0	4''	69.4				



## 25. Serratrin 7- $\beta$ -glucoside

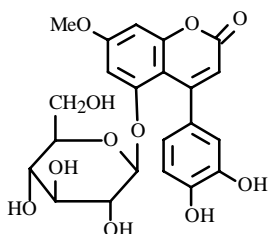
*Passiflora serratodigitata* [36]

$C_{21}H_{20}O_9$ , mp 166-168°C [36]

UV (MeOH) [36]: 208 (5.0), 248, 258 (4.1), 328 (3.9)

IR (KBr) [36]: 3400, 1700, 1615, 1600, 1585, 1550, 1495, 1450, 1355, 1280, 1220, 1170, 1090, 1065, 1030, 940, 820, 700

PMR (90 MHz, DMSO- $d_6$ ) [36]: 3.1-3.7 (6H, m, Glu), 5.0 (1H, d, J = 6, H-1 Glu), 5.8 (1H, s, H-3), 6.25 (1H, d, J = 2, H-8), 6.3 (1H, d, J = 2, H-6), 7.52 (5H, s, Ph-4)



## 26. 5-O-( $\beta$ -D-Glucopyranosyl)-3',4'-dihydroxy-7-methoxy-4-phenylcoumarin

*Hintonia latiflora* [12], *Coutarea hexandra* [10], *Exostema caribaeum* [11],

*E. mexicanum* [37]

$C_{22}H_{22}O_{11}$ , mp 237-238°C [11]

UV (MeOH) [11]: 218, 259, 329

IR (KBr) [11]: 3500, 2900, 1700, 1618, 1430, 1360, 1310, 1230, 1090, 1040

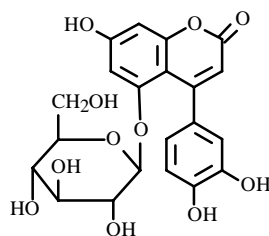
Mass [10]: 461 [M - H]<sup>-</sup>, 299

PMR (250 MHz, CD<sub>3</sub>OD) [10]: 2.62 (1H, dd, J = 7.5, J = 7.5, G-2''), 3.85 (3H, s, OMe), 5.90 (1H, s, H-3), 6.60 (1H, d, J = 2, H-8), 6.65 (1H, d, J = 2, H-6), 6.70 (1H, dd, J = 2, J = 7.5, H-6'), 6.81 (1H, d, J = 7.5, H-5'), 6.81 (1H, d, J = 2, H-2')

<sup>13</sup>C NMR (69.5 MHz, CD<sub>3</sub>OH) [10]:

C-2	163.0	C-7	165.0	C-4'	147.0	C-3''	78.3
3	113.2	8	100.0	5'	115.9	4''	71.2
4	158.0	8a	157.2	6'	120.6	5''	77.7
4a	105.5	1'	132.9	1''	101.5	6''	62.5
5	158.0	2'	117.0	2''	74.6	OMe-7	56.5
6	96.7	3'	145.5				

Biol: inhibits ATP synthesis, proton absorption, and energy transfer in chloroplasts [38, 39]



## 27. 5-O-( $\beta$ -D-Glucopyranosyl)-7,3',4'-trihydroxy-4-phenylcoumarin

*Hintonia latiflora* [12], *Coutarea hexandra* [10]

$C_{21}H_{20}O_{11}$ , mp 250-252°C [12]

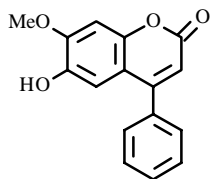
Mass [10]: 447 [M - H]<sup>-</sup>, 285

PMR (250 MHz, CD<sub>3</sub>OD) [10]: 2.62 (1H, dd, J = 7.5, J = 7.5, G-2''), 4.80 (1H, d, J = 7.5, G-1''), 5.83 (1H, s, H-3), 6.43 (1H, d, J = 2, H-8), 6.51 (1H, d, J = 2,

H-6), 6.69 (1H, dd, J = 2, J = 7.5, H-6'), 6.79 (1H, d, J = 7.5, H-5'), 6.80 (1H, d, J = 2, H-2')

<sup>13</sup>C NMR (69.5 MHz, CD<sub>3</sub>OD) [10]:

C-2	163.6	C-7	163.2	C-3'	145.5	G-1''	101.4
3	112.3	8	101.1	4'	146.9	2''	74.6
4	158.3	8a	157.3	5'	115.9	3''	78.2
4a	104.5	1'	133.1	6'	120.5	4''	71.1
5	158.1	2'	117.1			5''	77.7
6	98.1					6''	62.5



## 28. Dalbergin

*Dalbergia sissoo* [40, 41], *D. latifolia* [42], *D. melanoxylon* [43], *D. nigra* [44], *D. miscolobium* [45], *D. sissooides* [46], *D. cearensis* [47], *D. nitidula* [48], *D. riparia* [49], *D. spinosa* [50], *D. coromandeliana* [51], *D. baroni* [52, 53], *D. volubilis* [15, 16], *D. stevensonii* [54], *D. odorifera* [30, 31], *D. cultrata*

[55], *Machaerium kuhlmannii* [56], *M. nictitans* [56], *M. scleroxylon* [57], *M. pedicellatum* [58], *Piptadenia macrocarpa* [59], *Prosopis kuntzei* [60], *Goniorrhachis marginata* [61], *Cassia alata* [62]

$C_{16}H_{12}O_4$ , mp 209°C [63], 209-210°C [40], 210-211°C [30], 211-212°C [16], 214-216°C [54]

UV (MeOH) [16]: 235, 280, 380; (EtOH+NaOH) [63]: 260, 318

IR (KBr) [16]: 3200, 1700, 1610, 1500, 1450

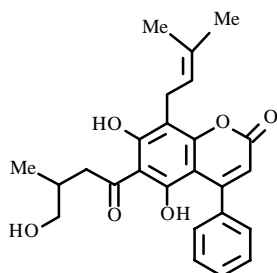
Mass [63]: 268 (100) [M]<sup>+</sup>, 240, 225, 197, 168, 155, 141, 127

PMR (200 MHz, DMSO-d<sub>6</sub>) [30]: 3.86 (3H, s, 7-OMe), 6.16 (1H, s, H-3), 6.79 (1H, s, H-5), 7.08 (1H, s, H-8), 7.52 (5H, m, Ph-4), 9.43 (1H, s, OH-6)

NOESY, HMQC, HMBC [30]

<sup>13</sup>C NMR (75.4 MHz, DMSO-d<sub>6</sub>) [30]:

C-2	160.85	C-6	143.96	C-10	111.32	C-4'	129.95
3	111.70	7	152.35	1'	135.72	5'	128.24
4	155.55	8	100.91	2'	128.71	6'	128.71
5	110.75	9	148.83	3'	128.24	OMe	56.57



## 29. 5,7-Dihydroxy-6-(4-hydroxy-3-methyl-1-oxobutyl)-8-(3-methyl-2-butenyl)-4-phenyl-2H-1-benzopyran-2-one

*Kielmeyera reticulata* [21]

$C_{25}H_{26}O_6$

[α]<sub>D</sub> +1.58° (CHCl<sub>3</sub>) [21]

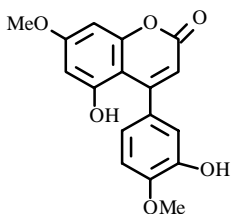
Mass [21]: 404 (20) [M - H<sub>2</sub>O]<sup>+</sup>, 389 (15), 361 (100), 349 (30), 327 (6)

PMR (300 MHz, CDCl<sub>3</sub>) [21]: 0.99 (H, d, J = 6.6, CH<sub>3</sub>-5''), 1.63 (3H, s, CH<sub>3</sub>-6'), 1.69 (3H, s, CH<sub>3</sub>-5'), 2.48 (1H, m, H-3''), 2.73 (1H, dd, J = 8.8, J = 14.0, H-2a''), 3.28 (2H, d, J = 7.6, CH<sub>2</sub>-4'), 3.41 (1H, dd, J = 8.8, J = 11.3, H-2b''), 3.75 (2H, m, CH<sub>2</sub>-4''), 5.07 (1H, d, J = 6.0, H-3'), 5.90 (1H, s, H-3), 7.40 (2H, m, Ph-4), 7.54 (5H, m, Ph-4), 14.61 (1H, s, OH-7)

<sup>1</sup>H-<sup>13</sup>C COSY [21]

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) [21]:

C-2	160.0	C-8	113.4	C-1''	206.6	C-1'''	137.5
3	112.4	8a	158.0	2''	49.4	2'''	128.0
4	155.5	2'	134.6	3''	33.7	3'''	130.0
4a	101.3	3'	121.3	4''	68.5	4'''	130.7
5	156.4	4'	22.1	5''	17.0	5'''	130.0
6	105.6	5'	17.0			6'''	128.0
7	167.6	6'	26.2				



### 30. 5,3'-Dihydroxy-7,4'-dimethoxy-4-phenylcoumarin

*Exostema caribaeum* [11], *Ex. acuminatum* [22]

$C_{17}H_{14}O_6$ , mp 225-226°C [11, 22]

UV (MeOH) [11]: 196, 252, 328

IR (KBr) [11]: 3460, 1690, 1620, 1515, 1440, 1370, 1350, 1335, 1285, 1200, 1050, 835, 810

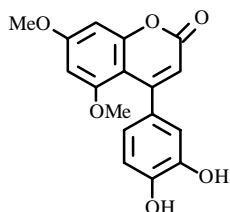
Mass [11]: 314 (100), 286 (64.8), 271 (37.7)

PMR (80 MHz,  $CDCl_3$ ) [11]: 3.83 (3H, s, 7-OMe), 3.98 (3H, s, 4'-OMe), 5.95 (1H, s, H-3), 6.28 (1H, d, J = 3, H-6), 6.51 (1H, d, J = 3, H-8), 6.97-6.99 (3H, m, H-2', H-5', H-6')

$^{13}C$  NMR (125.7 MHz,  $DMSO-d_6$ ) [22]:

C-2	163.0	C-6	94.9	C-1'	132.3	C-5'	115.3
3	104.8	7	152.9	2'	115.3	6'	130.0
4	159.0	8	129.4	3'	143.9	OMe	56.9
4a	112.5	8a	145.1	4'	152.3	OMe	56.7
5	158.8						

Biol: cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V<sup>+</sup>, KB-V<sup>-</sup>, LNCaP, SW626, SKNSH, and M109 [22]



### 31. 3',4'-Dihydroxy-5,7-dimethoxy-4-phenylcoumarin

*Coutarea hexandra* [24, 64], *Exostema mexicanum* [26]

$C_{17}H_{14}O_6$ , mp 211-212°C [24, 64]

UV (MeOH) [24]: 253, 326, 438

IR ( $CDCl_3$ ) [24]: 3600, 3540, 3260, 1707, 1611, 1595, 1513, 1158, 1111, 1052, 945, 910, 872, 858, 828

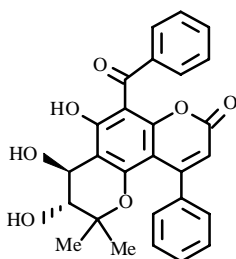
Mass [24]: 314 (100) [M]<sup>+</sup>, 296 (98), 271 (20), 243 (4)

PMR [ $(CD_3)_2CO$ ] [24]: 3.55 (3H, s), 3.92 (3H, s), 5.85 (1H, s), 6.38 (1H, d, J = 2.5), 6.53 (1H, d, J = 2.5), 6.68-6.98 (3H, m)

$^{13}C$  NMR (100 MHz,  $DMSO-d_6$ ) [28]:

C-2	159.7	C-6	95.8	C-1'	129.6	C-5'	114.6
3	111.3	7	163.0	2'	115.1	6'	118.8
4	155.5	8	93.7	3'	144.2	OMe	55.6
4a	103.0	8a	156.7	4'	145.6	OMe	55.7
5	158.2						

Biol: inhibitor of adenosine-3',5'-cyclomonophosphate-phosphodiesterase at  $IC_{50}$   $11.7 \times 10^{-5}$  M [34], antiplasmodial activity against *Plasmodium falciparum* [26]



### 32. trans-7,8-Dihydroxycalanone

*Calophyllum teysmannii* var. *inophyllolide* [14]

$C_{27}H_{22}O_7$

$[\alpha]_D -31.2^\circ$  ( $CHCl_3$ ) [14]

UV (EtOH) [14]: 252, 324

IR (KBr) [14]: 3431, 1722, 1652, 1540, 1455, 1131

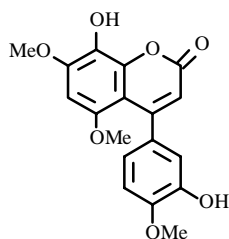
**Mass** [14]: 440 (100) [M - 18]<sup>+</sup>, 412 (20), 397 (30), 368 (50), 319 (40)

**PMR** [300 MHz, (CD<sub>3</sub>)<sub>2</sub>CO] [14]: 0.84 (3H, s, CH<sub>3</sub>-13), 1.02 (3H, s, CH<sub>3</sub>-12), 3.57 (1H, d, J = 7.7, H-7), 4.84 (1H, d, J = 7.7, H-8), 5.78 (1H, s, H-3), 7.33 (2H, m, H-2', H-6'), 7.43 (3H, m, H-3', H-4', H-5'), 7.51 (2H, t, J = 7.4, H-3'', H-5''), 7.63 (1H, tt, J = 7.4, J = 1.0, H-4''), 7.88 (2H, dd, J = 7.4, J = 1.0, H-2'', H-6''), 12.88 (1H, s, OH-9)

**NOE, HMQC, HMBC** [14]

<sup>13</sup>C NMR [75 MHz, (CD<sub>3</sub>)<sub>2</sub>CO] [14]:

C-2	158.6	C-8a	106.4	C-1'	140.6	C-1''	138.5
3	111.7	9	156.6	2'	127.2	2''	129.2
4	152.3	10	108.6	3'	127.6	3''	128.6
4a	102.5	10a	154.1	4'	127.2	4''	133.2
4b	155.9	11	193.1	5'	127.6	5''	128.6
6	80.2	12	18.6	6'	127.2	6''	129.2
7	74.0	13	24.8				
8	68.5						



### 33. 8,3'-Dihydroxy-5,7,4'-trimethoxy-4-phenylcoumarin

*Coutarea hexandra* [33], *Exostema acuminatum* [22]

C<sub>18</sub>H<sub>16</sub>O<sub>7</sub>, mp 191-192°C [33]

**UV** (MeOH) [33]: 271 (4.19), 324 (4.10); (MeOH+MeONa): 286, 332, 384

**IR** (CDCl<sub>3</sub>) [33]: 3545, 1712, 1610, 1590, 1512, 1176, 1112, 1050, 860

**Mass** [33]: 344 (100) [M]<sup>+</sup>, 329 (10), 316 (25), 301 (25), 297 (25), 273 (13), 269 (23)

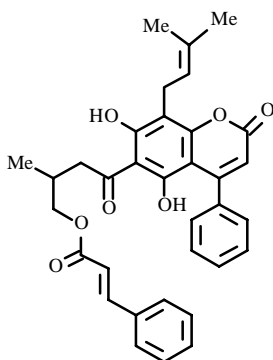
**PMR** [300 MHz, (CD<sub>3</sub>)<sub>2</sub>CO] [33]: 3.51 (3H, s, 5-OMe), 3.90 (3H, s, 4'-OMe), 3.97 (3H, s, 7-OMe), 5.85 (1H, s, H-3), 6.65 (1H, s, H-6), 6.79 (1H, dd, J = 8.2, J = 2, H-6'), 6.83 (1H, d, J = 2, H-2'), 6.98 (1H, d, J = 8.2, H-5'), 7.73 (1H, s, OH), 7.82 (1H, s, OH)

<sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>) [33]:

C-2	160.6	C-6	94.5	C-1'	133.6	C-5'*	111.4
3*	111.7	7**	151.9	2'	115.3	6'	119.1
4	145.6	8	128.1	3'	143.9	OMe	58.6
4a	103.3	8a**	150.8	4'	148.2	OMe	56.1
5	156.2						

\*,\*\*Assignments may be reversed

**Biol:** cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V<sup>+</sup>, KB-V<sup>-</sup>, LNCaP, SW626, SKNSH, and M109 [22]



### 34. 5,7-Dihydroxy-6-(4-cinnamoyl-3-methyl-1-oxobutyl)-8-(3-methyl-2-butenyl)-4-phenyl-2H-1-benzopyran-2-one

*Kielmeyera reticulata* [21]

C<sub>34</sub>H<sub>32</sub>O<sub>7</sub>

[α]<sub>D</sub> -12.17° (CHCl<sub>3</sub>) [21]

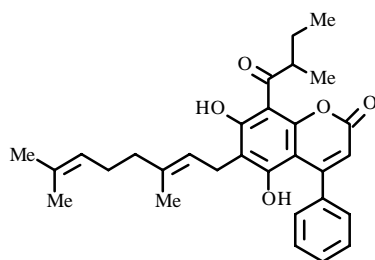
**Mass** [21]: 404 (38) [M - C<sub>6</sub>H<sub>5</sub>C<sub>2</sub>H<sub>2</sub>COOH]<sup>+</sup>, 389 (11), 361 (38), 349 (21), 321 (9), 131 (100)

**PMR** (300 MHz, CDCl<sub>3</sub>) [21]: 1.18 (1H, d, J = 6.0, CH<sub>3</sub>-5''), 1.64 (3H, s, CH<sub>3</sub>-6'), 1.69 (3H, s, CH<sub>3</sub>-5'), 2.71 (1H, m, H-3''), 3.27 (2H, d, J = 8.0,

CH<sub>2</sub>-4'), 3.31 (1H, dd, J = 6.0, J = 15.0, H-2a''), 3.45 (1H, dd, J = 6.0, J = 15.0, H-2b''), 4.24 (2H, m, CH<sub>2</sub>-4''), 5.06 (1H, d, J = 6.0, H-3'), 5.97 (1H, s, OH-5), 6.00 (1H, s, H-3), 6.41 (1H, d, J = 16.0, H-2C), 7.39 (5H, m, Ph-4, cinnamoyl), 7.52 (5H, m, Ph-4, cinnamoyl), 7.66 (1H, d, J = 16.0, H-3C), 14.39 (1H, s, OH-7)

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) [21]:

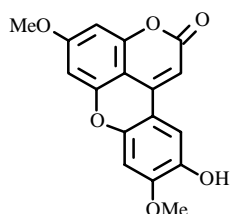
C-2	158.9	C-2'	134.7	C-1'''	137.4	C-1C	167.1
3	112.8	3'	121.3	2'''	128.0	2C	118.7
4	154.6	4'	22.1	3'''	130.1	3C	145.2
4a	101.1	5'	17.9	4'''	130.7	4C	135.1
5	156.5	6'	26.2	5'''	130.1	5C	128.6
6	105.2	1''	205.5	6'''	128.0	6C	129.4
7	167.4	2''	49.3			7C	130.8
8	113.2	3''	30.7			8C	129.4
8a	157.9	4''	69.4			9C	128.6
		5''	18.4				



**35. 6-(3,7-Dimethyl-2,6-octadienyl)-5,7-dihydroxy-8-(2-methylbutanoyl)-4-phenylcoumarin**

*Mesua ferrea* [32]

C<sub>30</sub>H<sub>34</sub>O<sub>5</sub>



**36. 7,4'-Dimethoxy-5'-hydroxy-4-phenyl-5,2'-oxidocoumarin**

*Exostema caribaeum* [19]

C<sub>17</sub>H<sub>12</sub>O<sub>7</sub>, mp 273-274°C [19]

UV (MeOH) [19]: 262, 308, 372, 388

IR (KBr) [19]: 3400, 3330, 3175, 3160, 1677, 1618, 1523, 1458, 1289, 1205, 1169, 1120, 817

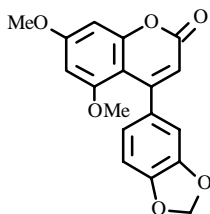
Mass [19]: 312 (100), 284 (30.1), 269 (10), 256 (51.1)

PMR (80 MHz, C<sub>5</sub>H<sub>5</sub>N-d<sub>6</sub>) [19]: 3.86 (3H, s, 7-OMe), 3.90 (3H, s, 4'-OMe), 5.91 (1H, s, H-3), 6.60 (2H, s, H-6, H-8), 6.90 (1H, s, H-3'), 7.20 (1H, s, H-6')

<sup>13</sup>C NMR (50 MHz, DMSO-d<sub>6</sub>) [19]:

C-2	160.90	C-6	96.41	C-1'	107.14	C-5'	141.00
3	100.51	7	163.02	2'	146.32	6'	108.70
4	153.70	8	93.12	3'	96.00	7-OMe	56.14
4a	99.80	8a	154.50	4'	144.31	4'-MeO	56.14
5	150.49						





### 37. 5,7-Dimethoxy-3',4'-methylenedioxy-4-phenylcoumarin

*Countarea hexandra* [65]

$C_{18}H_{14}O_6$ , mp 194-195°C [65]

UV (MeOH) [65]: 248 (4.07), 327 (4.42)

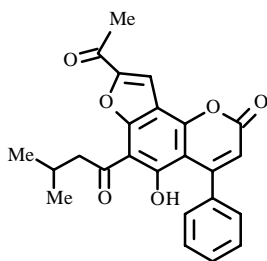
IR (CDCl<sub>3</sub>) [65]: 1710, 1613, 1595, 1505, 1157, 1110, 1052, 1039, 938, 904, 858, 810

Mass [65]: 326 (81) [M]<sup>+</sup>, 298 (100), 289 (13), 255 (4)

PMR (60 MHz, CDCl<sub>3</sub>) [65]: 3.47 (s, 3H), 3.83 (s, 3H), 5.93 (s, 1H), 5.95 (s, 2H), 6.19 (d, J = 2.5, 1H), 6.46 (d, J = 2.5, 1H), 6.75-6.85 (m, 3H)

<sup>13</sup>C NMR (100 MHz, DMSO-d<sub>6</sub>) [28]:

C-2	159.2	C-6	95.9	C-1'	132.8	C-5'	107.3
3	111.6	7	163.0	2'	107.8	6'	120.5
4	154.4	8	93.8	3'	146.8	OMe	55.6
4a	102.8	8a	156.3	4'	146.2	OCH <sub>2</sub> O	100.8
5	157.9						



### 38. Disparacetylfuran A

*Calophyllum dispar* [66]

$C_{24}H_{20}O_6$

UV (EtOH) [66]: 289 (3.87), 363 (3.34), 434 (2.72); (EtOH+NaOH): 252 (3.73), 286 (3.66), 374 (3.71), 432 (3.56)

IR (CHCl<sub>3</sub>) [66]: 3460, 1717, 1684, 1625, 758, 701

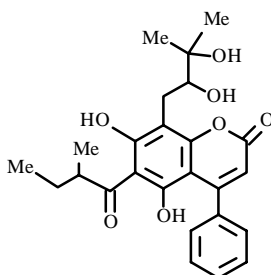
HRFABMS [66]: 403.1186 [M - H]<sup>-</sup>

PMR (270 MHz, CDCl<sub>3</sub>) [66]: 1.07 (6H, d, J = 6.5, CH<sub>3</sub>-4'', CH<sub>3</sub>-5''), 2.29 (1H, m, CH-3''), 2.63 (1H, s, CH<sub>3</sub>-5'''), 3.18 (2H, d, J = 6.5, CH<sub>2</sub>-2''), 6.22 (1H, s, 1H, H-3), 7.37 (2H, m, H-2', H-6'), 7.45 (3H, m, H-3', H-4', H-5'), 7.83 (1H, s, H-3'''), 15.05 (1H, s, OH-5)

HMBC, HMQC [66]

<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [66]:

C-2	158.5	C-8	110.0	C-4'	128.6	C-4''	22.6
3	115.0	9	156.5	5'	127.8	5''	22.6
4	156.4	10	103.4	6'	127.2	2'''	152.3
5	165.9	1'	138.4	1''	204.4	3'''	111.2
6	119.0	2'	127.2	2''	51.8	4'''	186.1
7	147.0	3'	127.8	3''	25.5	5'''	26.4



### 39. Disparidiol B

*Calophyllum dispar* [67]

$C_{25}H_{28}O_7$

UV (EtOH) [67]: 234 (4.22), 284 (4.43), 355 (3.93), 426 (3.59); (EtOH+NaOH): 238 (4.29), 279 (4.31), 381 (4.06), 410 (4.05)

IR (CHCl<sub>3</sub>) [67]: 3460, 1717, 1706, 1617, 768, 702

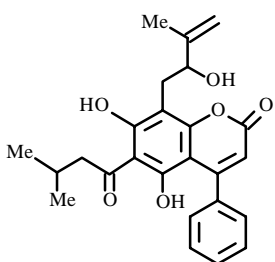
HRLSIMS [67]: 441.1927 [M + H]<sup>+</sup>

HMBC [67]

**PMR** (500 MHz, CDCl<sub>3</sub>) [67]: 0.90 (3H, t, J = 7.5, CH<sub>3</sub>-5''), 1.15 (3H, d, J = 6.5, CH<sub>3</sub>-3''), 1.36 (1H, s, CH<sub>3</sub>-4'''), 1.40 (1H, m, CH<sub>2</sub>-4a''), 1.49 (1H, s, CH<sub>3</sub>-5'''), 1.80 (1H, m, CH<sub>2</sub>-4b''), 2.61 (1H, dd, J = 10.0, J = 15.0, CH<sub>2</sub>-1a'''), 3.37 (1H, d, J = 15.0, CH<sub>2</sub>-1b'''), 3.74 (1H, dd, J = 7.0, J = 8.0, H-2'''), 3.84 (1H, m, H-2''), 5.97 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 10.26 (1H, s, OH-7), 14.29 (1H, s, OH-5)

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>) [67]:

C-2	159.7	C-8	105.3	C-5'	127.6	C-5''	11.8
3	111.9	8a	157.3	6'	127.1	1'''	26.0
4	156.6	1'	139.1	1''	212.1	2'''	80.6
4a	101.9	2'	127.1	2''	46.5	3'''	73.2
5	163.5	3'	127.6	3''	16.5	4'''	26.7
6	107.6	4'	128.2	4''	26.7	5'''	22.2
7	162.4						



#### 40. Disparinol A

*Calophyllum dispar* [68]

C<sub>25</sub>H<sub>26</sub>O<sub>6</sub>, mp 115-116°C [68]

**UV** (EtOH+HCl) [68]: 242 (4.09), 284 (4.42), 341 (4.00); (EtOH+NaOH): 244 (4.21), 302 (4.21), 381 (4.05), 412 (4.07)

**IR** [68]: 1700, 1622, 1580, 1559, 756, 697

**Mass** [68]: 422 (4), 404 (8), 389 (5), 352 (84), 351 (100), 347 (9), 333 (21), 295 (14)

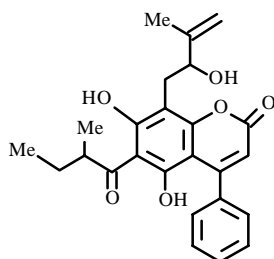
**HREIMS** [68]: 422.1726 [M]<sup>+</sup>

**PMR** (270 MHz, CDCl<sub>3</sub>) [68]: 0.94 (6H, dd, J = 2.0, J = 6.5, CH<sub>3</sub>-5'''), 1.93 (3H, s, CH<sub>3</sub>-5''), 2.23 (1H, m, CH-4''), 3.02 (2H, d, J = 6.5, CH<sub>2</sub>-2''), 3.04 (1H, dd, J = 8.0, J = 15.5, CH<sub>2</sub>-1a'''), 3.32 (1H, dd, J = 2.0, J = 15.0, CH<sub>2</sub>-1b'''), 4.50 (1H, d, J = 7.5, H-2'''), 4.94 (1H, s, CH<sub>2</sub>-4a'''), 5.03 (1H, s, CH<sub>2</sub>-4b'''), 5.96 (1H, s, H-3), 7.33 (2H, m, H-2', H-6'), 7.42 (3H, m, H-3', H-4', H-5'), 10.18 (1H, s, OH-7), 14.43 (1H, s, OH-5)

**DQF-COSY, HMBC** [68]

**<sup>13</sup>C NMR** (67.5 MHz, CDCl<sub>3</sub>) [68]:

C-2	160.0	C-7	162.5	C-4'	128.2	C-5''	22.7
3	112.1	8	104.7	5'	127.7	1'''	28.8
4	156.7	8a	157.7	6'	127.1	2'''	77.5
4a	102.0	1'	139.3	1''	207.5	3'''	146.0
5	163.3	2'	127.1	2''	53.5	4'''	111.1
6	107.8	3'	127.7	4''	25.1	5'''	18.7



#### 41. Disparinol B

*Calophyllum dispar* [68]

C<sub>25</sub>H<sub>26</sub>O<sub>6</sub>

**UV** (EtOH+HCl) [68]: 235 (4.00), 284 (4.24), 340 (3.82); (EtOH+NaOH): 243 (4.09), 302 (4.07), 382 (3.92), 409 (3.93)

**IR** [68]: 3420, 1718, 1618, 1584, 758, 702

**Mass** [68]: 422 (6), 404 (17), 378 (53), 363 (37), 352 (53), 351 (82), 347 (58), 333 (32), 321 (100), 295 (30)

**HREIMS** [68]: 422.1747 [M]<sup>+</sup>

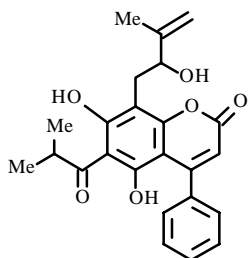
**PMR** (270 MHz, CDCl<sub>3</sub>) [68]: 0.90 (3H, t, J = 7.0, CH<sub>3</sub>-5''), 1.14 (3H, d, J = 6.5, CH<sub>3</sub>-3''), 1.39 (1H, m, CH<sub>2</sub>-4a''), 1.79 (1H, m, CH<sub>2</sub>-4b''), 1.94 (3H, s, CH<sub>3</sub>-5'''), 3.06 (1H, dd, J = 7.5, J = 15.0, CH<sub>2</sub>-1a'''), 3.30 (1H, dd, J = 2.0, J = 15.0, CH<sub>2</sub>-1b'''), 3.84 (1H, m, H-2''), 4.51 (1H, d, J = 6.0, H-2'''), 4.93 (1H, s, CH<sub>2</sub>-4a'''), 5.02 (1H, s, CH<sub>2</sub>-4b'''), 5.97 (1H, s, H-3), 7.33 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 10.20 (1H, s, OH-7), 14.33 (1H, s, OH-5)

**DQF-COSY, HMQC, HMBC** [68]

<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [68]:

C-2	159.9	C-8	104.7	C-5'	127.7	C-5''	11.9
3	112.2	8a	157.7	6'	127.2	1'''	28.7
4	156.7	1'	139.4	1''	212.2	2'''	77.7
4a	102.1	2'	127.2	2''	46.6	3'''	146.0
5	163.4	3'	127.7	3''	16.4	4'''	111.1
6	107.4	4'	128.2	4''	26.8	5'''	18.8
7	162.1						

**Biol:** cytotoxic activity against cell line KB at ED<sub>50</sub> 7 µg/mL [67]



## 42. Disparinol D

*Calophyllum dispar* [67]

C<sub>24</sub>H<sub>24</sub>O<sub>6</sub>

**UV** (EtOH) [67]: 235 (3.75), 283 (3.89), 331 (3.46); (EtOH+NaOH): 258 (3.82), 306 (3.75), 391 (3.42), 409 (3.41)

**IR** (CHCl<sub>3</sub>) [67]: 3420, 1732, 1716, 1622, 1595, 756, 702

**HRFABMS** [67]: 407.1530 [M - H]<sup>-</sup>

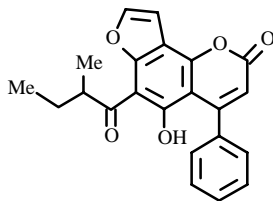
**PMR** (500 MHz, CDCl<sub>3</sub>) [67]: 1.16 (6H, d, J = 7.0, CH<sub>3</sub>-3'', CH<sub>3</sub>-4''), 1.95 (3H, s, CH<sub>3</sub>-5'''), 3.05 (1H, dd, J = 8.0, J = 15.5, CH<sub>2</sub>-1a'''), 3.13 (1H, d, J = 7.0, H-2''), 3.32 (1H, dd, J = 2.0, J = 15.0, CH<sub>2</sub>-1b'''), 4.51 (1H, d, J = 7.5, H-2'''), 4.94 (1H, s, CH<sub>2</sub>-4b'''), 5.03 (1H, s, CH<sub>2</sub>-4a'''), 5.97 (1H, s, H-3), 7.33 (2H, m, H-2', H-6'), 7.42 (3H, m, H-3', H-4', H-5'), 10.20 (1H, s, OH-7), 14.29 (1H, s, OH-5)

**HMBC** [67]

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) [67]:

C-2	159.9	C-7	162.1	C-4'	128.2	C-4''	19.2
3	112.2	8	104.7	5'	127.7	1'''	28.7
4	156.6	8a	157.7	6'	127.1	2'''	77.7
4a	102.1	1'	139.4	1''	212.3	3'''	146.0
5	163.4	2'	127.1	2''	39.9	4'''	111.1
6	106.9	3'	127.7	3''	19.2	5'''	18.7

**Biol:** cytotoxic activity against cell line KB at ED<sub>50</sub> 21 µg/mL [67]



### 43. Disparfuran B

*Calophyllum dispar* [66]

$C_{22}H_{18}O_5$

**UV** (EtOH) [66]: 238 (3.78), 283 (4.00), 330 (3.54); (EtOH+NaOH): 255 (3.78), 282 (3.76), 314 (3.70), 419 (3.46)

**IR** ( $CHCl_3$ ) [66]: 3440, 1732, 1717, 1622, 1559, 756, 700

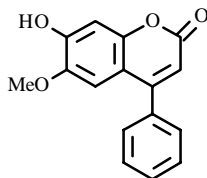
**HRAPCIMS** [66]: 363.1248 [M + H]<sup>+</sup>

**PMR** (270 MHz,  $CDCl_3$ ) [66]: 0.97 (3H, t, J = 7.5,  $CH_3-5''$ ), 1.24 (3H, d, J = 6.5,  $CH_3-3''$ ), 1.55 (1H, m,  $CH_2-4''\beta$ ), 1.86 (1H, m,  $CH_2-4''\alpha$ ), 3.83 (1H, m, H-2''), 6.17 (1H, s, H-3), 7.17 (1H, d, J = 2.0, H-3'''), 7.37 (2H, m, H-2', H-6'), 7.43 (3H, m, H-3', H-4', H-5'), 7.66 (1H, d, J = 2.0, H-2'''), 14.79 (1H, s, OH-5)

**DQF-COSY, HMBC, NOESY** [66]

**$^{13}C$  NMR** (67.5 MHz,  $CDCl_3$ ) [66]:

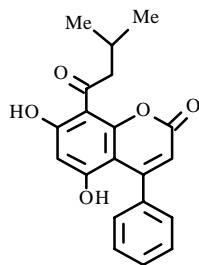
C-2	159.4	C-8	109.8	C-4'	128.4	C-3''	16.3
3	114.3	9	153.4	5'	127.7	4''	26.5
4	156.8	10	103.3	6'	127.1	5''	11.8
5	163.5	1'	138.9	1''	208.6	2'''	143.8
6	104.9	2'	127.1	2''	45.7	3'''	104.7
7	155.8	3'	127.7				



### 44. Isodalbergin

*Dalbergia sissoo* [69]

$C_{16}H_{12}O_4$ , mp 195-196°C [69]



### 45. Isodispar B

*Calophyllum dispar* [67]

$C_{20}H_{17}O_5$

**UV** (EtOH) [67]: 222 (4.40), 292 (4.32), 329 (4.25); (EtOH+NaOH): 225 (4.34), 253 (4.21), 333 (4.45), 381 (3.93), 428 (4.01)

**IR** ( $CHCl_3$ ) [67]: 3500, 1684, 1622, 1594, 774, 700

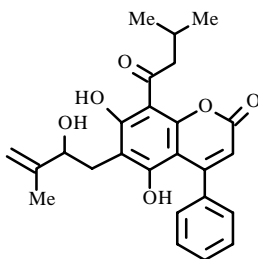
**HRFABMS** [67]: 337.1174 [M - H]<sup>-</sup>

**PMR** (500 MHz,  $CDCl_3$ ) [67]: 1.06 (6H, d, J = 6.5,  $CH_3-4''$ ,  $CH_3-5''$ ), 2.31 (1H, m, H-3''), 3.19 (2H, d, J = 6.5,  $CH_2-2''$ ), 6.02 (1H, s, H-3), 6.26 (1H, s, H-6), 7.43 (m, 2H, H-2', H-6'), 7.56 (m, 3H, H-3', H-4', H-5'), 14.77 (s, 1H, OH-7)

**$^{13}C$  NMR** (125 MHz,  $CDCl_3$ ) [67]:

C-2	158.5	C-6	101.4	C-2'	127.4	C-1''	205.8
3	111.7	7	168.8	3'	129.6	2''	53.5
4	154.0	8	104.9	4'	130.4	3''	25.5
4a	100.8	8a	155.7	5'	129.6	4''	22.6
5	159.6	1'	136.3	6'	127.4	5''	22.6

**Biol:** cytotoxic activity against cell line KB at  $ED_{50}$  8  $\mu$ g/mL [67]



#### 46. Isodisparinol A

*Calophyllum dispar* [68]

$C_{25}H_{26}O_6$

**UV** (EtOH+HCl) [68]: 226 (4.38), 292 (4.32), 328 (4.10); (EtOH+NaOH): 230 (4.29), 263 (4.08), 336 (4.39)

**IR** [68]: 3407, 1717, 1620, 1597, 1559, 758, 702

**Mass** [68]: 422 (6), 404 (12), 389 (6), 352 (57), 351 (100), 347 (9), 333 (30), 305 (10), 295 (15)

**HREIMS** [68]: 422.1726 [M]<sup>+</sup>

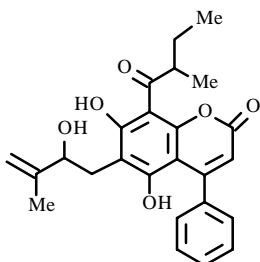
**PMR** (270 MHz, CDCl<sub>3</sub>) [68]: 1.06 (6H, d, J = 7.0, CH<sub>3</sub>-5'''), 1.81 (3H, s, CH<sub>3</sub>-5''), 2.32 (1H, m, CH-4'''), 2.77 (1H, dd, J = 8.0, J = 15.0, CH<sub>2</sub>-1a''), 3.13 (1H, dd, J = 2.0, J = 15.0, CH<sub>2</sub>-1b''), 3.20 (1H, dd, J = 1.5, J = 6.5, H-2'''), 4.32 (1H, d, J = 8.0, H-2''), 4.86 (1H, s, CH<sub>2</sub>-4a''), 4.93 (1H, s, CH<sub>2</sub>-4b''), 6.03 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 9.29 (1H, s, OH-5), 14.80 (1H, s, OH-7)

**HMBC** [68]

<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [68]:

C-2	159.1	C-7	166.9	C-4'	128.2	C-4''	110.6
3	112.2	8	104.1	5'	127.8	1'''	18.4
4*	156.4	8a*	156.5	6'	127.1	2'''	206.1
4a	102.2	1'	139.8	1''	28.6	3'''	53.5
5	160.3	2'	127.1	2''	76.8	4'''	25.7
6	109.8	3'	127.8	3''	146.4	5'''	22.7

\*Assignments may be reversed



#### 47. Isodisparinol B

*Calophyllum dispar* [68]

$C_{25}H_{26}O_6$

**UV** (EtOH+HCl) [68]: 228 (4.21), 289 (4.04), 328 (3.71); (EtOH+NaOH): 229 (3.96), 262 (3.69), 335 (4.03)

**IR** [68]: 3431, 1700, 1605, 771, 694

**HREIMS** [68]: 423.1808 [M + H]<sup>+</sup>

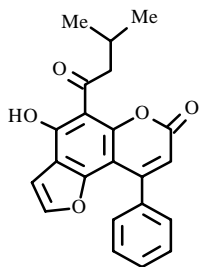
**PMR** (270 MHz, CDCl<sub>3</sub>) [68]: 1.03 (3H, t, J = 7.5, CH<sub>3</sub>-5'''), 1.29 (3H, dd, J = 8.0, J = 6.5, CH<sub>3</sub>-3'''), 1.53 (1H, m, CH<sub>2</sub>-4a'''), 1.82 (3H, s, CH<sub>3</sub>-5''), 1.95 (1H, m, CH<sub>2</sub>-4b'''), 2.78 (1H, dd, J = 8.5, J = 15.0, CH<sub>2</sub>-1a''), 3.12 (1H, dd, J = 2.0, J = 15.0, CH<sub>2</sub>-1b''), 3.99 (1H, m, CH<sub>2</sub>-2'''), 4.32 (1H, d, J = 8.0, H-2''), 4.87 (1H, s, CH<sub>2</sub>-4a''), 4.94 (1H, s, CH<sub>2</sub>-4b''), 6.04 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 9.22 (1H, s, OH-5), 14.80 (1H, s, OH-7)

**HMBC** [68]

<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [68]:

C-2	159.2	C-8	103.7	C-5'	127.8	C-5''	18.4
3	112.2	8a*	156.3	6'	127.1	1'''	210.6
4*	156.4	1'	139.8	1''	28.6	2'''	46.8
4a	102.2	2'	127.1	2''	76.8	3'''	27.2
5	160.2	3'	127.8	3''	146.4	4'''	22.7
6	109.9	4'	128.2	4''	110.6	5'''	11.8
7	167.0						

\*Assignments may be reversed



#### 48. Isodisparfuran A

*Calophyllum dispar* [66]

$C_{22}H_{18}O_5$

**UV** (EtOH) [66]: 230 (3.96), 253 (3.97), 287 (3.84), 337 (3.58);

(EtOH+NaOH): 241 (4.01), 259 (3.90), 280 (3.79), 308 (3.58)

**UR** ( $CHCl_3$ ) [66]: 3450, 2930, 1744, 1609, 756, 702

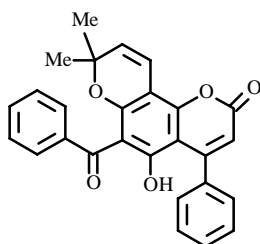
**HREIMS** [66]: 362.1170  $[M]^+$

**PMR** (270 MHz,  $CDCl_3$ ) [66]: 1.09 (6H, d,  $J = 7.0$ ,  $CH_3-4'''$ ,  $CH_3-5'''$ ), 2.37 (1H, m, H-3'''), 3.30 (2H, d,  $J = 7.0$ ,  $CH_2-2'''$ ), 6.22 (1H, s, H-3), 6.96 (1H, d,  $J = 2.0$ , H-3'''), 7.31 (1H, d,  $J = 2.0$ , H-2'''), 7.42 (2H, m, H-2', H-6'), 7.50 (3H, m, H-3', H-4', H-5'), 14.84 (1H, s, OH-5)

**DQF-COSY, HMBC, HMQC** [66]

$^{13}C$  NMR (67.5 MHz,  $CDCl_3$ ) [66]:

C-2	159.2	C-8	106.1	C-4'	129.3	C-1'''	207.3
3	112.0	9	154.3	5'	128.1	2'''	53.6
4	153.7	10	100.0	6'	127.9	3'''	25.6
5	154.2	1'	137.1	2''	146.6	4'''	22.7
6	115.0	2'	127.9	3''	104.9	5'''	22.7
7	162.7	3'	128.1				



#### 49. Isocalanone

*Calophyllum teysmanii* var. *inophyllolide* [14, 70]

$C_{27}H_{20}O_5$ , mp 188-190°C [70]

**UV** (EtOH) [70]: 240 (3.70), 296 (3.67), 386 (2.90)

**IR** (KBr) [70]: 3430, 3068, 2363, 1729, 1597, 1420, 1260, 1188, 1145, 856, 748, 689, 575

**Mass** [70]: 424 (52), 409 (100), 331 (52), 303 (13), 105 (76), 77 (74)

**HREIMS** [70]: 424.13237

**PMR** (500 MHz,  $CDCl_3$ ) [70]: 1.06 (6H, s, Me-12, Me-13), 5.48 (1H, d,  $J = 10.2$ , H-9), 6.02 (1H, s, H-3), 6.83 (1H, d,  $J = 10.2$ , H-10), 7.35-7.38 (2H, m), 7.39-7.42 (5H, m), 7.48-7.53 (3H, m, H-1'—H-6', H-1''—H-6''), 12.54 (1H, s, OH-5)

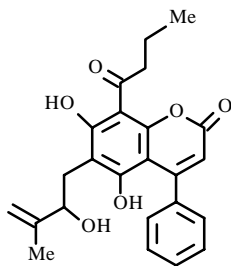
**HMBC** [70]

**X-ray** [70, 71]

$^{13}C$  NMR (125 MHz,  $CDCl_3$ ) [70]:

C-2	159.6	C-8	79.5	C-13	27.9	C-1''	141.4
3	112.7	9	126.9	1'	139.0	2''	127.8
4	156.1	10	114.9	2'	127.3	3''	127.9
4a	102.0	10a	101.6	3'	127.6	4''	131.2
5	162.2	10b*	157.3	4'	128.5	5''	127.9
6	107.1	11	200.4	5'	127.6	6''	127.8
6a*	154.9	12	27.9	6'	127.3		

\*Assignments may be reversed



## 50. Isoracemosol

*Mesua racemosa* [72]

$C_{24}H_{24}O_6$

**UV** (EtOH+HCl) [72]: 225 (3.89), 292 (3.89), 326 (3.66); (EtOH+NaOH): 229 (4.03), 257 (3.88), 335 (4.03)

**IR** [72]: 3447, 1734, 1707, 1597, 1387, 1134, 771, 702

**Mass** [72]: 409 (55), 391 (66), 337 (72)

**HRLSIMS** [72]: 409.1647 [M]<sup>+</sup>

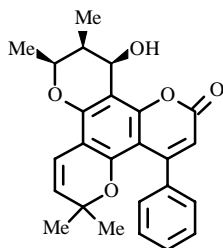
**PMR** (270 MHz,  $CDCl_3$ ) [72]: 1.07 (3H, t,  $J = 7.5$ ,  $CH_3-4'''$ ), 1.78 (2H, m,  $CH_2-3'''$ ), 1.81 (3H, s,  $CH_3-5''$ ), 2.78 (1H, dd,  $J = 2$ ,  $J = 15$ ,  $CH_2-1a''$ ), 3.10 (1H, dd,  $J = 2$ ,  $J = 15$ ,  $CH_2-1b''$ ), 3.34 (2H, t,  $J = 7.0$ ,  $CH_2-2'''$ ), 4.32 (1H, d,  $J = 7.5$ , H-2''), 4.86 (1H, s,  $CH_2-4b''$ ), 4.93 (1H, s,  $CH_2-4a''$ ), 6.03 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 14.77 (1H, s, OH-7)

**HMBC** [72]

<sup>13</sup>C NMR (67.5 MHz,  $CDCl_3$ ) [72]:

C-2	159.3	C-7**	166.8	C-4'	128.2	C-4''	110.6
3	112.1	8	104.0	5'	127.8	5''	18.5
4*	156.6	8a*	156.4	6'	127.1	1'''	206.3
4a	102.2	1'	139.8	1''	28.5	2'''	46.6
5**	160.3	2'	127.1	2''	77.4	3'''	18.1
6	109.8	3'	127.8	3''	146.4	4'''	13.8

\*,\*\*Assignments may be reversed



## 51. Inophyllum A [(+)-cis-dihydroinophyllolide]

*Calophyllum inophyllum* [73, 74], *C. moonii* [75]

$C_{25}H_{24}O_5$ , **mp** 193-195°C [75], 200-201°C [73]

$[\alpha]_D^{25} +43^\circ$  (acetone) [73, 76],  $+68.8^\circ$  ( $CHCl_3$ ) [75]

**UV** (EtOH) [75]: 235 (4.18), 280 (4.15), 286 (4.18), 337 (3.95)

**IR** (KBr) [75]: 3400, 2680-2820, 1710, 1635, 1590, 1445, 1380, 1350, 1150, 850, 770, 700

**Mass** [75]: 404 (25) [M]<sup>+</sup>, 389 (100), 386 (1), 371 (4), 333 (50)

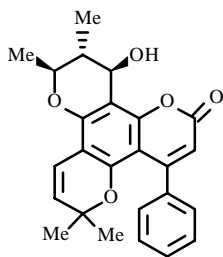
**PMR** ( $CDCl_3$ ) [75]: 0.97 (3H, s,  $CH_3-10$ ), 0.98 (3H, s,  $CH_3-10$ ), 1.16 (3H, d,  $J = 7.2$ ,  $CH_3-3$ ), 1.43 (3H, d,  $J = 6.92$ ,  $CH_3-2$ ), 2.27 (1H, m,  $J = 5.19$ ,  $J = 3.46$ , H-3), 4.43 (1H, m,  $J = 6.92$ ,  $J = 3.46$ , H-2), 5.17 (1H, d,  $J = 5.19$ , H-4), 5.33 (1H, d,  $J = 9.9$ , H-11), 5.96 (1H, s, H-7), 6.51 (1H, d,  $J = 9.9$ , H-12), 7.30 (5H, m, Ph-8)

**X-ray** [76]

<sup>13</sup>C NMR (100 MHz,  $CDCl_3$ ) [74]:

C-2	160.3	C-8	115.9	C-12a	105.5	C-17	127.2
3	111.4	8a	106.3	12b	154.0	18	127.2
4	156.2	8b	152.5	13	139.9	19	26.7
4a	103.1	10	75.7	14	127.2	20	26.7
4b	150.9	11	35.5	15	127.2	21	16.0
6	76.8	12	62.6	16	127.5	22	9.7
7	127.1						

**Biol**: inhibitor of HIV-1 reverse transcriptase (RT) at  $IC_{50}$  30  $\mu$ M [74], antitumor activity [9]



## 52. Inophyllum B [(+)-*trans*-dihydroinophyllolide]

*Calophyllum inophyllum* [73, 74, 77]

$C_{25}H_{24}O_5$ , mp 200-202°C [73]

$[\alpha]_D^{25} +36^\circ$  (acetone) [76]

UV (EtOH) [73]: 232 (4.23), 277 (4.15), 286 (4.2), 334 (3.96)

IR (mineral oil) [73]: 3430, 1717, 767, 703

Mass [73]: 404  $[M]^+$ , 386, 371

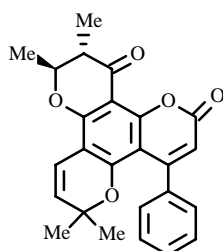
PMR (400 MHz,  $CDCl_3$ ) [74]: 0.91 (3H, s,  $CH_3$ -20), 0.97 (3H, s,  $CH_3$ -19), 1.18 (3H, d,  $J = 6.8$ ,  $CH_3$ -22), 1.47 (3H, d,  $J = 6.4$ ,  $CH_3$ -21), 1.97 (1H, ddq,  $J = 6.8$ ,  $J = 9.1$ ,  $J = 7.8$ , H-11), 3.96 (1H, dq,  $J = 6.4$ ,  $J = 9.1$ , H-10), 4.79 (1H, d,  $J = 7.8$ , H-12), 5.37 (1H, d,  $J = 10.0$ , H-7), 5.97 (1H, s, H-3), 6.53 (1H, d,  $J = 10.0$ , H-8), 7.41, 7.31 (5H, m, Ph-4)

X-ray [76]

$^{13}C$  NMR (100 MHz,  $CDCl_3$ ) [74]:

C-2	160.6	C-8	116.0	C-12a	106.1	C-17	127.3
3	111.7	8a	106.0	12b	153.6	18	127.3
4	156.3	8b	153.7	13	140.0	19	26.9
4a	103.1	10	73.0	14	127.3	20	26.8
4b	151.1	11	38.2	15	127.3	21	18.8
6	76.7	12	61.8	16	127.6	22	12.5
7	127.1						

Biol: inhibitor of HIV-1 RT at  $IC_{50}$  0.038  $\mu M$  [74], activity against HIV-1 in cell culture at  $IC_{50}$  1.4  $\mu M$  [74]



## 53. Inophyllum C [(+)-*trans*-inophyllolide]

*Calophyllum inophyllum* [73, 74, 77, 78], *C. teysmannii* var. *inophyllolide* [14, 79, 80]

$C_{25}H_{22}O_5$ , mp 186-188°C [78], 188-191°C [73]

$[\alpha]_D^{25} +13^\circ$  ( $CHCl_3$ ) [73]

UV (EtOH) [73]: 240 (4.32), 257 (4.46), 266 (4.49), 302 (4.39)

IR (mineral oil) [73]: 1727, 1688, 767, 703

Mass [73]: 402  $[M]^+$ , 387 (15), 331 (56), 303 (28)

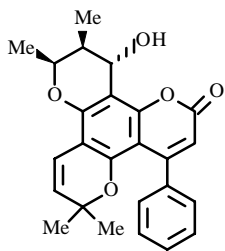
PMR (60 MHz,  $CDCl_3$ ) [73]: 0.95 (3H, s,  $CH_3$ -6), 0.98 (3H, s,  $CH_3$ -6), 1.24 (3H, d,  $J = 7.2$ ,  $CH_3$ -11), 1.56 (3H, d,  $J = 6.6$ ,  $CH_3$ -10), 2.59 (1H, m,  $J = 7.2$ ,  $J = 11.5$ , H-11), 4.32 (1H, m,  $J = 6.6$ ,  $J = 11.5$ , H-10), 5.42 (1H, d,  $J = 10.0$ , H-7), 6.04 (1H, s, H-3), 6.56 (1H, d,  $J = 10.0$ , H-8), 7.3 (5H, m, Ph-4)

$^{13}C$  NMR (100 MHz,  $CDCl_3$ ) [74]:

C-2	159.6	C-8	115.2	C-12a	103.9	C-17	127.5
3	113.5	8a	105.4	12b	154.9	18	127.5
4	159.4	8b	155.1	13	139.9	19	27.2
4a	103.4	10	79.7	14	127.5	20	27.4
4b	155.6	11	47.3	15	127.5	21	19.6
6	78.6	12	190.0	16	127.7	22	10.6
7	127.2						

Biol: inhibitor of HIV-1 RT at  $IC_{50}$  10  $\mu M$  [74], antitumor activity [9], cytotoxic activity against cell lines P388, WEHI164, THP-1, and MOLT4 [14]





#### 54. Inophyllum D

*Calophyllum inophyllum* [73, 74]

$C_{25}H_{24}O_5$ , mp 200-202°C [73]

$[\alpha]_D^{25} +35^\circ$  (CDCl<sub>3</sub>) [76]

UV (EtOH) [73]: 232 (4.23), 277 (4.15), 286 (4.2), 334 (3.96)

IR (mineral oil) [73]: 3430, 1717, 767, 703

Mass [73]: 404 [M]<sup>+</sup>, 386, 371

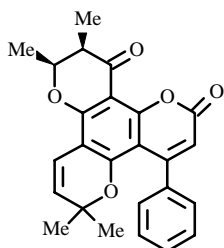
PMR (60 MHz, CDCl<sub>3</sub>) [73]: 0.94 (6H, s, CH<sub>3</sub>-6), 1.17 (3H, d, J = 7.2, CH<sub>3</sub>-11), 1.43 (3H, d, J = 7.0, CH<sub>3</sub>-10), 2.27 (1H, m, H-11), 4.43 (1H, m, J = 3.3, J = 7.0, H-10), 5.17 (1H, d, J = 5.4, H-12), 5.36 (1H, d, J = 10, H-7), 5.96 (1H, s, H-3), 6.55 (1H, d, J = 10, H-8), 7.3 (5H, m, Ph-4)

X-ray [76]

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) [74]:

C-2	160.7	C-8	115.9	C-12a	103.9	C-17	127.3
3	111.7	8a	106.0	12b	154.6	18	127.3
4	156.4	8b	153.9	13	139.9	19	26.9
4a	103.3	10	71.1	14	127.3	20	26.8
4b	151.0	11	37.2	15	127.3	21	17.5
6	77.1	12	64.5	16	127.5	22	9.1
7	127.1						

Biol: inhibitor of HIV-1 RT at IC<sub>50</sub> 11 μM [9], antitumor activity [9]



#### 55. Inophyllum E [(+)-cis-inophyllolide]

*Calophyllum inophyllum* [73, 74, 77],

*C. teysmannii* var. *inophyllolide* [80]

$[\alpha]_D^{25} +70^\circ$  (CHCl<sub>3</sub>) [73]

UV (EtOH) [73]: 240 (4.32), 257 (4.46), 266 (4.49), 302 (4.39)

IR (mineral oil) [73]: 1727, 1688, 767, 703

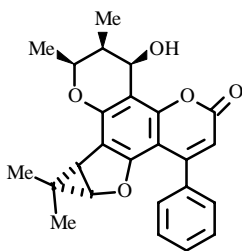
Mass [73]: 402 [M]<sup>+</sup>, 387 (15), 331 (56), 303 (28)

PMR (60 MHz, CDCl<sub>3</sub>) [73]: 0.97 (6H, s, CH<sub>3</sub>-6, CH<sub>3</sub>-6), 1.18 (3H, d, J = 7.2, CH<sub>3</sub>-11), 1.44 (3H, d, J = 6.8, CH<sub>3</sub>-10), 2.67 (1H, m, J = 7.2, J = 3.7, H-11), 4.73 (1H, m, J = 6.8, J = 3.7, H-10), 5.42 (1H, d, J = 10.0, H-7), 6.05 (1H, s, H-3), 6.56 (1H, d, J = 10.0, H-8), 7.3 (5H, m, Ph)

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) [74]:

C-2	159.4	C-8	115.2	C-12a	103.8	C-17	127.6
3	113.3	8a	105.4	12b	154.8	18	127.6
4	159.3	8b	155.3	13	139.8	19	27.3
4a	102.7	10	77.2	14	127.6	20	27.2
4b	155.6	11	45.9	15	127.6	21	16.0
6	78.6	12	191.5	16	127.3	22	9.1
7	127.4						

Biol: inhibitor of HIV-1 RT at IC<sub>50</sub> 100 μM [74], antitumor activity [9]



### 56. Inophyllum G-1

*Calophyllum inophyllum* [74]

$C_{26}H_{26}O_5$

$[\alpha]_D +174.2^\circ$  (CDCl<sub>3</sub>) [74]

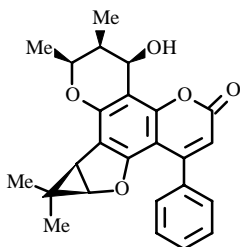
UV (MeOH) [74]: 216, 219, 315, 333

IR (KBr) [74]: 3600-3200, 3100-3000, 2850, 1719, 1700, 1142, 1097, 853, 766, 736, 700

HRCIMS [74]: 404.1610

PMR (400 MHz, CDCl<sub>3</sub>) [74]: 0.71 (3H, s, CH<sub>3</sub>-20), 1.05 (3H, s, CH<sub>3</sub>-19), 1.20 (3H, d, J = 7.1, CH<sub>3</sub>-22), 1.45 (3H, d, J = 6.8, CH<sub>3</sub>-21), 2.34 (1H, ddq, J = 3.8, J = 4.8, J = 7.1, H-11), 2.46 (1H, d, J = 5.7, H-8), 4.23 (1H, d, J = 5.7, H-6), 4.46 (1H, ddq, J = 0.8, J = 3.8, J = 6.8, H-10), 5.12 (1H, dd, J = 4.8, J = 0.8, H-12), 6.01 (1H, s, H-3), 7.41, 7.31 (5H, m, Ph-4)  
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) [74]:

C-2	160.5	C-8	29.4	C-12a	106.1	C-17	127.7
3	111.2	8a	112.0	12b	153.2	18	127.7
4	155.0	8b	153.4	13	137.6	19	22.5
4a	99.0	10	75.9	14	127.7	20	12.7
4b	158.4	11	35.6	15	127.7	21	15.9
6	72.9	12	62.7	16	127.8	22	10.5
7	13.0						



### 57. Inophyllum G-2

*Calophyllum inophyllum* [74]

$C_{26}H_{26}O_5$

$[\alpha]_D -49.1^\circ$  (CDCl<sub>3</sub>) [74]

UV (MeOH) [74]: 215, 217, 224, 335

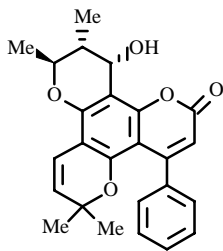
IR (KBr) [74]: 3600-3200, 3100-3000, 3000-2800, 1718, 1699, 1142, 1095, 855, 770, 741, 701

HRCIMS [74]: 404.1610

PMR (400 MHz, CDCl<sub>3</sub>) [74]: 0.73 (s, 3H, CH<sub>3</sub>-20), 1.05 (s, 3H, CH<sub>3</sub>-19), 1.18 (d, J = 7.1, 3H, CH<sub>3</sub>-22), 1.45 (d, J = 6.8, 3H, CH<sub>3</sub>-21), 2.33 (ddq, J = 3.3, J = 5.3, J = 7.1, 1H, H-11), 2.42 (d, J = 5.7, 1H, H-8), 4.21 (d, J = 5.7, 1H, H-6), 4.40 (ddq, J = 0.8, J = 3.3, J = 6.8, 1H, H-10), 5.18 (dd, J = 5.3, J = 0.8, 1H, H-12), 6.02 (s, 1H, H-3), 7.42, 7.32 (m, 5H, Ph-4)  
<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) [74]:

C-2	160.5	C-8	29.3	C-12a	106.0	C-17	127.7
3	111.2	8a	111.9	12b	153.2	18	127.7
4	154.9	8b	153.8	13	137.6	19	22.6
4a	98.9	10	75.7	14	127.7	20	12.6
4b	158.4	11	35.8	15	127.7	21	16.0
6	72.8	12	63.4	16	127.8	22	9.6
7	13.0						

**Biol:** inhibitor of HIV-1 RT at IC<sub>50</sub> 70 μM [74]



### 58. Inophyllum P

*Calophyllum inophyllum* [74, 77]

$C_{25}H_{24}O_5$

$[\alpha]_D^{25} +19.8^\circ$  (CDCl<sub>3</sub>) [74],  $+31.4^\circ$  [76]

UV (MeOH) [74]: 207, 235, 280, 286, 334

IR (KBr) [74]: 3435, 2930, 2860, 1719, 1640, 1590, 1565, 765, 703

HRCIMS [74]: 405.1732 [M + H]<sup>+</sup>

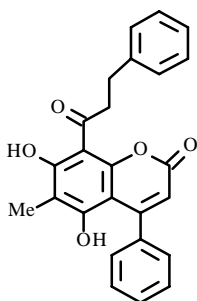
PMR (400 MHz, CDCl<sub>3</sub>) [74]: 0.93 (6H, s, CH<sub>3</sub>-19, CH<sub>3</sub>-20), 1.17 (3H, d, J = 7.0, CH<sub>3</sub>-22), 1.44 (3H, d, J = 6.3, CH<sub>3</sub>-21), 1.79 (1H, ddq, J = 3.3, J = 7.0, J = 10.6, H-11), 4.29 (1H, dq, J = 6.3, J = 10.6, H-10), 5.04 (1H, d, J = 3.3, H-12), 5.37 (1H, d, J = 10.0, H-7), 5.97 (1H, s, H-3), 6.54 (1H, d, J = 10.0, H-8), 7.42, 7.30 (5H, m, Ph-4)

X-ray [76]

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) [74]:

C-2	160.1	C-8	115.9	C-12a	106.1	C-17	127.3
3	111.5	8a	106.3	12b	154.1	18	127.3
4	156.4	8b	153.7	13	139.9	19	26.5
4a	103.6	10	76.9	14	127.3	20	26.9
4b	150.9	11	40.4	15	127.3	21	18.9
6	77.2	12	67.1	16	127.4	22	15.0
7	127.6						

Biol: inhibitor of HIV-1 RT at IC<sub>50</sub> 0.130 μM [74], activity against HIV-1 in cell culture at IC<sub>50</sub> 1.6 μM [74]



### 59. Interruptin A

*Cyclosorus interruptus* [81]

$C_{25}H_{20}O_5$ , mp 143-145°C [81]

UV (MeOH) [81]: 214, 283, 417

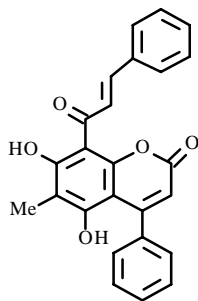
Mass [81]: 400 (36) [M]<sup>+</sup>, 382 (7), 295 (100), 91 (24)

PMR (300 MHz, CDCl<sub>3</sub>) [81]: 2.28 (3H, s, Me-6), 2.96 (2H, t, J = 7.6, CH<sub>2</sub>-3'), 3.29 (2H, t, J = 7.6, CH<sub>2</sub>-2'), 5.95 (1H, s, H-3), 7.16-7.29 (5H, m, H-5', H-6', H-8', H-9', H-4''), 7.43-7.47 (2H, m, H-2'', H-6''), 7.53-7.61 (3H, m, H-7', H-3'', H-5''), 12.89 (1H, s, OH-7)

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) [81]:

C-2	159.4	C-8	107.0	C-5'	128.4	C-2''	127.5
3	112.8	8a	157.1	6'	128.4	3''	130.0
4	153.4	1'	206.1	7'	130.6	4''	126.0
4a	99.9	2'	46.8	8'	128.4	5''	130.0
5	156.9	3'	30.3	9'	128.4	6''	127.5
6	106.0	4'	141.2	1''	136.1	Me-6	7.5
7	164.5						

Biol: antibacterial activity against *Bacillus cereus*, *Staphylococcus epidermidis*, and *M. luteus* [81], cytotoxic activity against cell line KB [81]



## 60. Interruptin B

*Cyclosorus interruptus* [81]

$C_{25}H_{18}O_5$ , mp 222-224°C [81]

UV (MeOH) [81]: 223, 328, 331

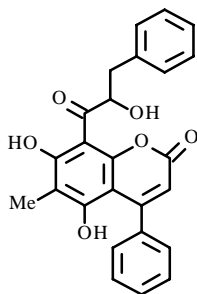
Mass [81]: 398 (11)  $[M]^+$ , 321 (8), 294 (4), 266 (3), 91 (4), 44 (100)

PMR (300 MHz,  $CDCl_3$ ) [81]: 2.05 (3H, s, Me-6), 5.84 (1H, s, OH-5), 6.04 (1H, s, H-3), 7.43-7.50 (6H, m, H-5', H-7', H-9', H-2'', H-4'', H-6''), 7.59-7.62 (2H, m, H-3'', H-5''), 7.74-7.77 (2H, m, H-6', H-8'), 7.95 (1H, d,  $J = 15.5$ , H-3'), 8.28 (1H, d,  $J = 15.5$ , H-2'), 14.72 (1H, s, OH-7)

$^{13}C$  NMR (75 MHz,  $CDCl_3$ ) [81]:

C-2	158.5	C-8	104.7	C-5'	128.9	C-2''	127.6
3	112.1	8a	155.0	6'	129.0	3''	130.0
4	153.7	1'	192.8	7'	130.6	4''	130.7
4a	100.3	2'	126.4	8'	129.0	5''	130.0
5	156.9	3'	145.0	9'	128.9	6''	127.6
6	109.4	4'	135.0	1''	136.0	Me-6	7.6
7	167.5						

**Biol:** cytotoxic activity against cell line KB [81]



## 61. Interruptin C

*Cyclosorus interruptus* [81]

$C_{25}H_{20}O_6$

UV (MeOH) [81]: 290, 360

Mass [81]: 441 (25)  $[M + 2H + Na]^+$ , 417 (54)  $[M + H]^+$ , 295 (80)

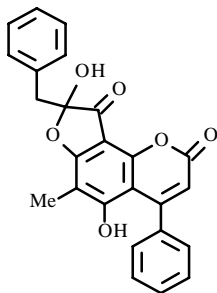
PMR (300 MHz,  $CD_3OD$ ) [81]: 2.27 (3H, s, Me-6), 2.77 (1H, dd,  $J = 8.7$ ,  $J = 13.8$ , H-3'b), 3.18 (1H, dd,  $J = 3.8$ ,  $J = 13.8$ , H-3'a), 5.31 (1H, dd,  $J = 3.8$ ,  $J = 8.7$ , H-2'), 5.91 (1H, s, H-3), 7.07-7.09 (2H, m, H-6', H-8'), 7.18-7.23 (1H, m, H-4''), 7.24-7.26 (3H, m, H-5', H-7', H-9'), 7.28-7.36 (2H, m, H-3'', H-5''), 7.39-7.48 (2H, m, H-2'', H-6'')

HMBC [81]

$^{13}C$  NMR (75 MHz,  $CD_3OD$ ) [81]:

C-2	162.0	C-8	*	C-5'	130.6	C-2''	129.2
3	112.6	8a	150.0	6'	128.8	3''	128.3
4	*	1'	207.9	7'	127.5	4''	129.3
4a	103.5	2'	79.0	8'	128.8	5''	128.3
5	163.2	3'	41.3	9'	130.6	6''	129.2
6	105.1	4'	139.0	1''	134.8	Me-6	8.1
7	159.8						

\*Signals cannot be accurately defined owing to low intensities



## 62. Interruptin D

*Cyclosorus interruptus* [81]

$C_{25}H_{18}O_6$ , mp 181-183°C [81]

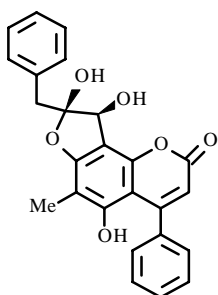
UV (MeOH) [81]: 207, 288, 372, 400

Mass [81]: 415 (100) [M + H]<sup>+</sup>, 397 (5), 295 (20)

PMR (300 MHz, CDCl<sub>3</sub>) [81]: 2.25 (3H, s, Me-6), 3.00 (2H, s, CH<sub>2</sub>-3'), 6.08 (1H, s, H-3), 6.80 (2H, d, J = 6.9, H-5', H-9'), 7.15-7.31 (3H, m, H-6', H-7', H-8'), 7.38-7.49 (5H, m, H-2'', H-3'', H-4'', H-5'', H-6'')

<sup>13</sup>C NMR (75 MHz, CD<sub>3</sub>OD) [81]:

C-2	159.5	C-8	103.8	C-5'	130.5	C-2''	128.0
3	112.5	8a	165.2	6'	128.7	3''	128.0
4	154.1	1'	197.1	7'	127.8	4''	129.3
4a	100.1	2'	105.7	8'	128.7	5''	128.0
5	157.0	3'	41.1	9'	130.5	6''	129.0
6	107.0	4'	131.0	1''	136.8	Me-6	7.1
7	161.7						



## 63. Interruptin E

*Cyclosorus interruptus* [81]

$C_{25}H_{20}O_6$ , mp 118-120°C [81]

UV (MeOH) [81]: 210, 268, 331

Mass [81]: 398 (100) [M - H<sub>2</sub>O]<sup>+</sup>, 321 (82), 294 (36)

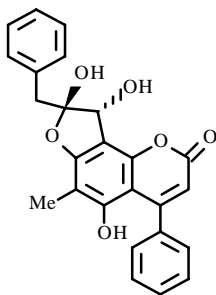
PMR (300 MHz, DMSO-d<sub>6</sub>) [81]: 2.16 (3H, s, Me-6), 2.79 (1H, d, J = 14.0, H-3'a), 3.09 (1H, d, J = 14.2, H-3'b), 4.71 (1H, d, J = 8.1, H-1'), 5.34 (1H, d, J = 8.6, OH-1'), 5.90 (1H, s, H-3), 6.66 (1H, s, OH-2'), 7.05-7.12 (2H, m, H-5', H-9'), 7.07-7.47 (4H, m, H-2'', H-3'', H-5'', H-6''), 7.10-7.16 (1H, m, H-7'), 7.37-7.51 (3H, m, H-6', H-8', H-4'')

<sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>) [81]:

C-2	160.6	C-8	112.5	C-5'	130.5	C-2''	127.4
3	110.1	8a	*	6'	128.3	3''	127.6
4	*	1'	73.9	7'	125.7	4''	128.8
4a	97.7	2'	113.0	8'	128.3	5''	127.6
5	156.2	3'	39.4	9'	130.5	6''	127.4
6	104.1	4'	136.2	1''	137.3	Me-6	8.1
7	154.9						

\*Signals cannot be accurately defined owing to low intensities

**Biol:** antibacterial activity against *Bacillus cereus*, *Staphylococcus epidermidis*, and *M. luteus* [81]



#### 64. Interruptin F

*Cyclosorus interruptus* [81]

$C_{25}H_{20}O_6$ , mp 118-120°C [81]

UV (MeOH) [81]: 210, 268, 331

Mass [81]: 398 (100)  $[M - H_2O]^+$ , 321 (82), 294 (36)

PMR (300 MHz, DMSO- $d_6$ ) [81]: 2.14 (3H, s, Me-6), 2.74 (1H, d, J = 13.8, H-3'a), 2.91 (1H, dd, J = 14.5, J = 0.6, H-3'b), 4.82 (1H, d, J = 5.0, H-1'), 5.44 (1H, d, J = 6.0, OH-1'), 5.87 (1H, s, H-3), 6.40 (1H, s, OH-2'), 7.05-7.12 (2H,

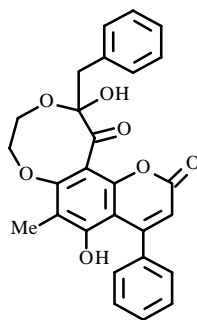
m, H-5', H-9'), 7.07-7.47 (4H, m, H-2'', H-3'', H-5'', H-6''), 7.10-7.22 (1H, m, H-7'), 7.37-7.51 (3H, m, H-6', H-8', H-4'')

$^{13}C$  NMR (75 MHz, DMSO- $d_6$ ) [81]:

C-2	159.0	C-8	*	C-5'	130.4	C-2''	127.4
3	109.9	8a	*	6'	128.2	3''	127.7
4	*	1'	68.3	7'	126.3	4''	128.7
4a	102.5	2'	111.8	8'	128.2	5''	127.7
5	156.6	3'	42.4	9'	130.4	6''	127.4
6	97.7	4'	135.4	1''	137.4	Me-6	8.1
7	153.9						

\*Signals cannot be accurately defined owing to low intensities

**Biol:** antibacterial activity against *Bacillus cereus*, *Staphylococcus epidermidis*, and *M. luteus* [81]



#### 65. Interruptin G

*Cyclosorus interruptus* [81]

$C_{27}H_{22}O_7$ , mp 162-164°C [81]

UV (MeOH) [81]: 208, 290, 375, 407

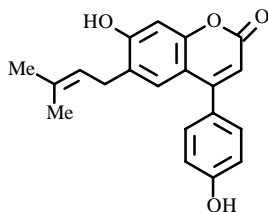
Mass [81]: 460 (6)  $[M + 2H]^+$ , 398 (66), 395 (100)

PMR (300 MHz, DMSO- $d_6$ ) [81]: 2.08 (3H, s, Me-6), 2.76 (1H, d, J = 14.1, H-3'a), 2.94 (1H, d, J = 14.1, H-3'b), 3.41 (2H, t, J = 5.3, CH<sub>2</sub>-11'), 3.49 (2H, t, J = 5.2, CH<sub>2</sub>-10'), 6.00 (1H, s, H-3), 6.81-6.83 (2H, m, H-5', H-9'), 7.11-7.13 (3H, m, H-7', H-3'', H-5''), 7.38-7.39 (2H, m, H-2'', H-6''), 7.48-7.51 (3H, m, H-6', H-8', H-4''), 7.82 (1H, s, OH-2')

$^{13}C$  NMR (75 MHz, DMSO- $d_6$ ) [81]:

C-2	158.9	C-8	105.0	C-6'	127.7	C-2''	128.0
3	111.2	8a	166.1	7'	126.6	3''	127.7
4	154.0	1'	195.2	8'	127.7	4''	129.0
4a	98.4	2'	106.4	9'	130.1	5''	127.7
5	156.5	3'	40.8	10'	60.2	6''	128.0
6	105.4	4'	133.4	11'	72.3	Me-6	7.7
7	159.8	5'	130.1	1''	136.9		

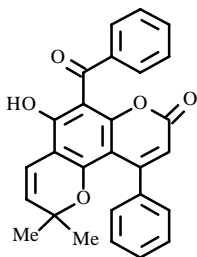
**Biol:** antibacterial activity against *Staphylococcus epidermidis* and *M. luteus* [81]



### 66. Inflacoumarin A

*Glycyrrhiza inflata* [82, 83]

$C_{20}H_{18}O_4$



### 67. Calanone

*Calophyllum teysmannii* var. *inophyllolide* [14, 20, 79, 80]

$C_{27}H_{20}O_5$

**UV** (EtOH) [79]: 242 (4.17), 274 (4.32), 320 (4.01)

**IR** [79]: 3455, 3065, 2979, 2921, 1738, 1607, 1597, 1571, 1442, 1360, 1270, 1135

**HRFABMS** [79]: 425.1366 [MH]<sup>+</sup>

**PMR** (500 MHz,  $CDCl_3$ ) [79]: 0.98 (6H, s,  $CH_3$ -12,  $CH_3$ -13), 5.42 (1H, d,  $J = 10.5$ , H-7), 5.88 (1H, s, H-3), 6.64 (1H, d,  $J = 10.5$ , H-8), 7.23 (2H, m, H-2', H-6'), 7.37 (3H, m, H-3', H-4', H-5'), 7.46 (2H, t,  $J = 8.0$ , H-3'', H-5''), 7.57 (1H, tt,  $J = 8.0$ ,  $J = 1.7$ , H-4''), 7.65 (2H, dd,  $J = 8.0$ ,  $J = 1.7$ , H-2'', H-6''), 12.46 (s, 1H, OH-7)

**HMBC, HMQC** [79]

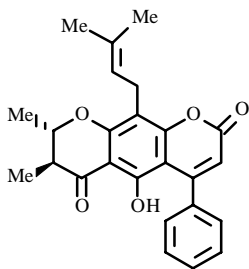
**X-ray** [71]

<sup>13</sup>C NMR (125 MHz,  $CDCl_3$ ) [79]:

C-2	158.0	C-8	115.2	C-13	27.4	C-1''	140.3
3	112.6	8a	105.7	1'	139.7	2''	128.2
4	155.3	9	161.5	2'	127.2	3''	128.2
4a	102.4	10	103.8	3'	127.5	4''	132.3
4b	156.4	10a*	155.9	4'	127.8	5''	128.2
6	79.1	11	198.8	5'	127.5	6''	128.2
7	127.0	12	27.4	6'	127.2		

\*Assignments may be reversed

**Biol:** cytotoxic activity against cell lines P388, WEHI164, THP-1, and MOLT4 [14]



### 68. Calaustralin

*Calophyllum australicum* [84]

$C_{25}H_{24}O_5$ , **mp** 192-193.5°C [84]

**UV** (EtOH-OH<sup>-</sup>) [84]: 286, 315, 425

**IR** [84]: 1718, 1645, 1613

**Mass** [14]: 404 (85) [M]<sup>+</sup>, 389 (80), 361 (100), 349 (45), 333 (65), 305 (75)

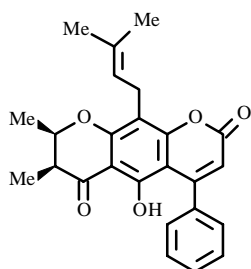
**HREIMS** [14]: 404.1629 [M]<sup>+</sup>

**PMR** (300 MHz,  $CDCl_3$ ) [14]: 1.21 (3H, d,  $J = 7.1$ ,  $CH_3$ -16), 1.56 (3H, d,  $J = 6.2$ ,  $CH_3$ -17), 1.69 (3H, s,  $CH_3$ -15), 1.85 (3H, s,  $CH_3$ -14), 2.64 (1H, dq,  $J = 12.2$ ,  $J = 7.1$ , H-7), 3.46 (2H, d,  $J = 7.5$ ,  $CH_2$ -11), 4.27 (1H, dq,  $J = 12.2$ ,  $J = 6.2$ , H-8), 5.25 (1H, m,  $J = 7.5$ , H-12), 6.00 (1H, s, H-3), 7.30-7.41 (5H, m, Ph), 13.12 (1H, s, OH)

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) [14]:

C-2*	160.6	C-7	45.9	C-11	21.6	C-1'	138.9
3	113.0	8	79.1	12	121.2	2'	127.3
4**	155.8	8a	105.7	13	132.7	3'	128.4
4a***	102.3	9	161.5	14	25.8	4'	127.7
5*	159.8	9a*	160.5	15	18.0	5'	128.4
5a***	108.8	10***	103.5	16	10.0	6'	127.3
6	200.1	10a**	159.1	17	19.6		

\*, \*\*, \*\*\* Assignments may be reversed



### 69. (-)-Calaustralin

*Calophyllum inophyllum* [85]

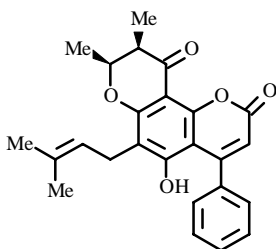
$\text{C}_{25}\text{H}_{24}\text{O}_5$ , mp 197-198°C [85]

UV (EtOH) [85]: 285, 335; (EtOH+NaOH): 285, 313, 424

IR (mineral oil) [85]: 1728, 1625, 1597, 1375, 1160, 1110, 900, 835, 703

Mass [85]: 404 (76)  $[\text{M}]^+$ , 389 (56), 361 (100), 349 (20), 336 (16), 333 (24), 305 (36), 293 (16), 280 (12), 277 (14)

PMR (60 MHz,  $\text{CDCl}_3$ ) [85]: 1.15 (3H, d,  $J = 6$ ), 1.55 (3H, d,  $J = 6$ ), 1.65 (3H, s), 1.85 (3H, s), 2.59 (1H, m), 3.45 (2H, d,  $J = 7$ ), 4.25 (1H, m), 5.30 (1H, d,  $J = 7$ ), 6.00 (1H, s), 7.40 (5H, s), 12.75 (1H, OH)

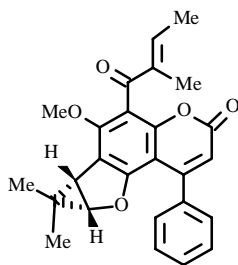


### 70. Calocoumarin A

*Calophyllum inophyllum* [86]

$\text{C}_{25}\text{H}_{24}\text{O}_5$

Biol: antitumor activity [86]

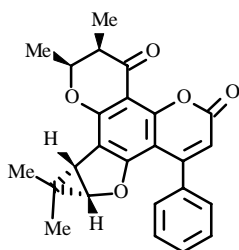


### 71. Calocoumarin B

*Calophyllum inophyllum* [86]

$\text{C}_{26}\text{H}_{25}\text{O}_5$

Biol: antitumor activity [86]



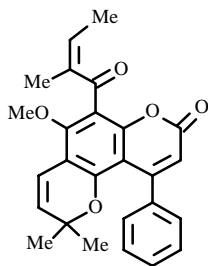
### 72. Calocoumarin C

*Calophyllum inophyllum* [86]

$\text{C}_{25}\text{H}_{22}\text{O}_5$

Biol: antitumor activity [86]





### 73. Calophyllolide

*Calophyllum inophyllum* [1, 77, 87, 88, 89], *C. bracteatum* [90],

*C. aff. pervillei* [91]

$C_{26}H_{24}O_5$ , **mp** 152-154°C [90], 156°C [5], 158-160°C [1], 159-160°C [87]

**UV** (EtOH) [1]: 235 (4.47), 270 (4.27), 295 (4.22), 325 (4.27)

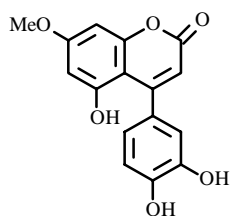
**Mass** [5]: 416 (31)  $[M]^+$ , 401 (100), 361 (12), 331 (13)

**PMR** (300 MHz,  $CDCl_3$ ) [5]: 0.97 (6H, s,  $Me_2CH$ ), 1.90 (3H, dd,  $J = 7.0$ ,  $J = 1.0$ ,  $MeCH=C$ ), 2.01 (3H, m,  $MeCCO$ ), 3.75 (3H, s, MeO), 5.48 (1H, d,  $J = 10.0$ ,  $ArCH=CH$ ), 6.02 (s, 1H,  $C=CHCO$ ), 6.45 (1H, d,  $J = 10.0$ , 1H,  $ArCH=CH$ ), 6.53-6.61 (1H, dq,  $J = 1.3$ ,  $J = 7.0$ ,  $MeCH=C$ ), 7.23-7.27 and 7.37-7.43 (5H, two m, Ph)

$^{13}C$  NMR (75 MHz,  $CDCl_3$ ) [5]:

C-2	159.5	C-7	129.0	C-12	194.3	C-15	127.8
3	114.2	8	116.0	12a	115.0	16	26.9
4	155.0	8a	110.7	12b	152.0	17	26.9
4a	105.6	8b	155.8	13	127.3	18	15.2
4b	151.7	10	144.2	13a	139.5	19	10.7
6	77.4	11	139.9	14	127.5	OMe	63.0

**Biol:** antitumor activity [86]



### 74. Coutareagenin (5,3',4'-trihydroxy-7-methoxyneoflavone)\*

\*structure corrected after re-examination [93]

*Coutarea hexandra* [92, 93]

$C_{16}H_{12}O_6$ , **mp** 193-194°C [93], 208-210°C [92]

**UV** (MeOH) [93]: 261 (4.22), 329 (4.26); (MeOH+NaOMe): 278, 336, 405;

(MeOH+ $AlCl_3$ ): 260, 329; (MeOH+ $AlCl_3+HCl$ ): 260, 329; (MeOH+NaOAc):

274, 330, 362, 400; (MeOH+NaOAc+ $H_3BO_3$ ): 260, 330, 400

**IR** (KBr) [92]: 3350, 1675, 1630, 1175, 1165, 1085

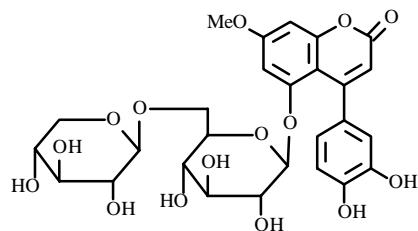
**Mass** [93]: 300 (100)  $[M]^+$ , 299 (13.4), 273 (16.2), 272 (93.6), 257 (22.5), 243 (7.3)

**PMR** (270 MHz,  $DMSO-d_6$ ) [93]: 3.79 (3H, s, 7-OMe), 5.76 (1H, s, H-3), 6.24 (1H, d,  $J = 2.19$ , H-6), 6.49 (1H, d,  $J = 2.19$ , H-8), 6.61 (1H, dd,  $J = 2.20$ ,  $J = 7.20$ , H-6'), 6.71 (1H, d,  $J = 2.20$ , H-2'), 6.72 (1H, d,  $J = 7.20$ , H-5'), 8.96 (1H, s, OH), 9.07 (1H, s, OH), 10.23 (1H, s, OH)

$^{13}C$  NMR [100.6 MHz,  $(CD_3)_2CO$ ] [92]:

C-2	164.1	C-6	94.4	C-10	103.1	C-4'	116.0
3	112.5	7	160.6	1'	131.7	5'	146.6
4	158.2	8	99.4	2'	145.3	6'	120.3
5	157.3	9	156.3	3'	115.6	OMe	56.1

**Biol:** inhibits ATP synthesis and proton absorption in chloroplasts [38], antidiabetic agent [94]



**75. 5-O-[\beta-D-Xylopyranosyl-(1-6)-\beta-D-glucopyranosyl]-7-methoxy-3',4'-dihydroxy-4-phenylcoumarin**

*Coutarea hexandra* [10]

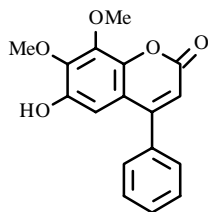
$C_{27}H_{30}O_{15}$

Mass [10]: 593 [M - H]<sup>-</sup>, 461, 299

PMR (250 MHz, CD<sub>3</sub>OD) [10]: 2.62 (1H, dd, J = 7.5, J = 7.5, G-2''), 3.88 (3H, s, OMe), 4.25 (1H, d, J = 7.5, X-1''), 4.80 (1H, d, J = 7.5, G-1''), 5.91 (1H, s, H-3), 6.65 (1H, d, J = 2, H-8), 6.70 (1H, d, J = 2, H-6), 6.71 (1H, dd, J = 2, J = 7.5, H-6'), 6.80 (1H, d, J = 7.5, H-5'), 6.80 (1H, d, J = 2, H-2')

<sup>13</sup>C NMR (69.5 MHz, CD<sub>3</sub>OD) [10]:

C-2	162.9	C-1'	133.0	G-1''	101.6	X-1''	105.5
3	113.3	2'	117.0	2''	74.6	2''	74.9
4	158.1	3'	145.5	3''	77.7	3''	77.3
4a	105.8	4'	147.0	4''	71.1	4''	71.4
5	158.1	5'	115.9	5''	77.7	5''	66.8
6	96.6	6'	120.7	6''	70.3	OMe	56.6
7	165.0						
8	100.6						
8a	157.1						



**76. Kuhlmannin**

*Machaerium kuhlmannii* [56, 95], *M. nictitans* [56, 95],

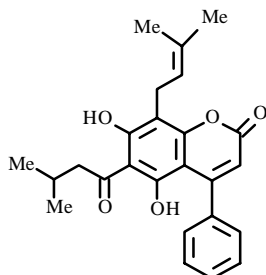
*M. pedicellatum* [58], *Piptadenia macrocarpa* [59]

$C_{17}H_{14}O_5$ , mp 211°C [56]

UV (EtOH) [56]: 214 (4.39), 300 (3.81)

IR (CHCl<sub>3</sub>) [56]: 3500, 1710, 1555, 1395

PMR (60 MHz, CDCl<sub>3</sub>) [56]: 4.08 (3H, s), 4.11 (3H, s), 4.27 (1H, s), 6.3 (1H, s), 6.77 (1H, s), 7.49 (5H, s)



**77. Mammea A/AA (mammeisin)**

*Mammea americana* [96, 97], *M. africana* [98, 99], *Mesua ferrea* [100],

*M. racemosa* [101], *M. thwaitesii* [102], *Kayea assamica* [103], *Kielmeyera elata* [29]

$C_{25}H_{26}O_5$ , mp 83°C [99], 92-112°C [104], 97°C [103], 98-100°C [100], 98-109°C [97]

$[\alpha]_D^{22} +22^\circ$  [103]

UV (EtOH) [97]: 281 (4.41), 338 (4.00); (EtOH+HCl): 249 (3.96), 317 (3.94); (EtOH+NaOH): 243 (4.10), 301 (4.36), 427 (4.10)

IR (KBr) [97]: 1745, 1620, 1597, 766, 698; (CDCl<sub>3</sub>): 3436, 3289, 1730, 1621, 1587

Mass [105]: 406 (85) [M]<sup>+</sup>, 363 (10), 351 (100), 349 (19), 293 (50)

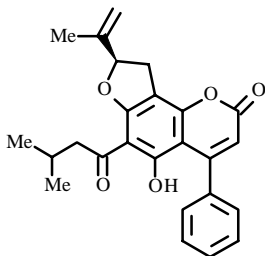
HRMS [105]: 406.1179 [M]<sup>+</sup>

PMR (250 MHz, CDCl<sub>3</sub>) [105]: 0.9 (6H, d, J = 7, Me<sub>2</sub>CH), 1.74 and 1.88 (6H, two s, Me<sub>2</sub>C=CH), 2.16 (1H, m, J = 7, Me<sub>2</sub>CHCH<sub>2</sub>), 2.84 (2H, d, J = 7, CHCH<sub>2</sub>CO), 3.52 (2H, d, J = 7, ArCH<sub>2</sub>CH), 5.2 (1H, t, J = 7, Me<sub>2</sub>C=CHCH<sub>2</sub>), 5.88 (1H, s, PhC=CHCO), 7.3-7.6 (5H, m, ArH), 9.8 (1H, s, OH), 11.2 (1H, s, OH)

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	159.2	C-7	163.3	C-4'	129.7	C-4''	22.6
3	112.7	8	108.0	5'	129.1	1'''	21.7
4	154.4	8a	156.6	6'	127.4	2'''	120.6
4a	100.8	1'	137.2	1''	207.1	3'''	135.5
5	159.5	2'	127.4	2''	53.6	4a'''	18.0
6	107.3	3'	129.1	3''	24.7	4b'''	25.8

**Biol:** inhibitor of oxidative phosphorylation at 0.5  $\mu\text{g}/\text{mL}$  [106]



### 78. Mammea A/AA dehydrocyclo F

*Calophyllum dispar* [66]

$\text{C}_{25}\text{H}_{24}\text{O}_5$

**UV** (EtOH) [66]: 230 (3.79), 283 (4.04), 346 (3.61); (EtOH-NaOH): 250 (3.93), 284 (3.79), 315 (3.74), 428 (3.64)

**IR** ( $\text{CHCl}_3$ ) [66]: 3430, 3060, 2960, 1746, 1617, 768, 700

**HREIMS** [66]: 404.1637  $[\text{M}]^+$

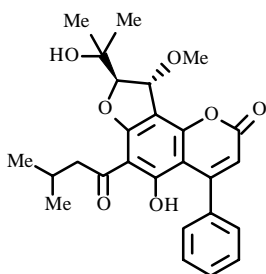
**PMR** (270 MHz,  $\text{CDCl}_3$ ) [66]: 0.96 (6H, d,  $J = 6.5$ ,  $\text{CH}_3\text{-}4''$ ,  $\text{CH}_3\text{-}5''$ ), 1.83 (3H, s,  $\text{CH}_3\text{-}6'''$ ), 2.18 (1H, m, H-3''), 2.90 (1H, dd,  $J = 3.0$ ,  $J = 7.0$ ,  $\text{CH}_2\text{-}2''$ ), 3.15 (1H, dd,  $J = 8.0$ ,  $J = 15.0$ ,  $\text{CH}_2\text{-}3''' \alpha$ ), 3.50 (1H, dd,  $J = 10.0$ ,  $J = 15.0$ ,  $\text{CH}_2\text{-}3''' \beta$ ), 5.03 (1H, s, H-5'''b), 5.15 (1H, s, H-5'''a), 5.48 (1H, t,  $J = 9.0$ , H-2'''), 5.94 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 14.55 (1H, s, OH-5)

**DQF-COSY, HMQC, HMBC, NOESY** [66]

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [66]:

C-2	159.7	C-9	155.6	C-5'	127.5	C-5''	22.5
3	111.9	10	102.2	6'	127.1	2'''	89.1
4	156.5	1'	139.0	1''	205.3	3'''	30.5
5	164.5	2'	127.1	2''	51.7	4'''	142.2
6	103.2	3'	127.5	3''	25.3	5'''	113.2
7	164.5	4'	128.1	4''	22.5	6'''	17.1
8	104.5						

**Biol:** cytotoxic activity against cell line KB at  $\text{ED}_{50}$  40  $\mu\text{g}/\text{mL}$  [66]



### 79. Mammea A/AA methoxycyclo F

*Calophyllum dispar* [66]

$\text{C}_{26}\text{H}_{28}\text{O}_7$

**UV** (EtOH) [66]: 245 (4.01), 282 (4.04), 349 (3.65); (EtOH+NaOH): 241 (3.89), 282 (4.09), 328 (3.75), 414 (3.12)

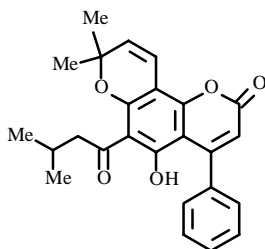
**IR** ( $\text{CHCl}_3$ ) [66]: 3410, 1734, 1719, 1617, 768, 699

**HRLSIMS** [66]: 453.1920  $[\text{M} + \text{H}]^+$

**PMR** (270 MHz,  $\text{CDCl}_3$ ) [101]: 0.96 (6H, d,  $J = 7.0$ ,  $\text{CH}_3\text{-}4''$ ,  $\text{CH}_3\text{-}5''$ ), 1.35 (1H, s,  $\text{CH}_3\text{-}5'''$ ), 1.39 (1H, s,  $\text{CH}_3\text{-}6'''$ ), 2.21 (1H, m, H-3''), 2.81 (1H, dd,  $J = 7.0$ ,  $J = 15.0$ ,  $\text{CH}_2\text{-}2'' \alpha$ ), 3.00 (1H, dd,  $J = 7.0$ ,  $J = 15.0$ ,  $\text{CH}_2\text{-}2'' \beta$ ), 3.64 (3H, s, OMe-3'''), 4.65 (1H, d,  $J = 3.0$ , H-2'''), 5.23 (1H, d,  $J = 3.0$ , H-3'''), 5.99 (1H, s, H-3), 7.31 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 14.75 (1H, s, OH-5)

**HMBC** [66]<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [101]:

C-2	159.8	C-9	156.8	C-6'	127.2	C-2'''	97.7
3	112.5	10	102.8	1''	205.1	3'''	78.6
4	156.5	1'	139.0	2''	52.0	4'''	71.2
5	166.5	2'	127.2	3''	25.0	5'''	25.5
6	103.3	3'	127.7	4''	22.6	6'''	25.9
7	164.4	4'	128.3	5''	22.6	OMe-3'''	57.7
8	105.9	5'	127.7				

**80. Mammea A/AA cyclo D (mammeigin)**

*Mammea americana* [107, 108], *Mesua ferrea* [104, 109],  
*M. thwaitesii* [102], *Kielmeyera pumila* [110], *K. elata* [29],  
*K. lathrophyton* [111]

C<sub>25</sub>H<sub>24</sub>O<sub>5</sub>, **mp** 144-146°C [108], 148-150°C [110], 149-150°C [109],  
 150-151°C [112]

**UV** (EtOH) [110]: 235 (4.43), 288 (4.49), 370 (3.82); (EtOH+NaOH):  
 230 (4.25), 252 (4.36), 312 (4.41); (EtOH+NaOAc): 240 (4.29), 305 (4.32)

**IR** (KBr) [110]: 3440, 1745, 1645, 1615, 1380, 1125, 775, 750, 710

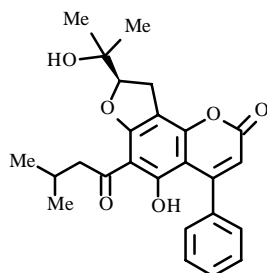
**Mass** [110]: 404 (34) [M]<sup>+</sup>, 389 (100), 371 (27), 347 (21), 105 (11), 77 (11), 42 (20)

**PMR** (300 MHz, CDCl<sub>3</sub>) [111]: 0.94 (6H, d, J = 6.7, Me-4'', Me-5''), 1.60 (6H, s, Me-7', Me-8'), 2.19 (1H, m, H-3''), 2.90 (2H, d, J = 6.9, CH<sub>2</sub>-2''), 5.60 (1H, d, J = 10.0, H-5'), 5.99 (1H, s, H-3), 6.90 (1H, d, J = 10.0, H-4'), 7.30 (1H, m, Ph-4), 7.40 (4H, m, Ph-4), 14.73 (1H, s, OH-5)

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) [111]:

C-2	159.6	C-8	101.4	C-1''	206.7	C-1'''	139.2
3	112.6	8a	157.8	2''	53.5	2'''	127.1
4	154.7	4'	115.5	3''	25.0	3'''	127.5
4a	102.2	5'	126.2	4''	22.6	4'''	128.1
5	164.5	6'	79.8	5''	22.6	5'''	127.5
6	106.9	7'	28.1			6'''	127.1
7	156.4	8'	28.1				

**Biol**: inhibitor of oxidative phosphorylation at 0.5 μg/mL [106]

**81. Mammea A/AA cyclo F (cyclomammeisin)**

*Mammea americana* [113], *Mesua thwaitesii* [102], *Kayea assamica* [103],  
*Kielmeyera elata* [29], *Calophyllum dispar* [66]

C<sub>25</sub>H<sub>26</sub>O<sub>6</sub>, **mp** 105°C [103], 115-117°C [114], 131-132°C [105],  
 148-150°C [113]

**UV** (EtOH) [105]: 280, 347; (EtOH+KOH): 247, 313

**IR** (CDCl<sub>3</sub>) [105]: 3050, 1720, 1610

**Mass** [105]: 422 (22) [M]<sup>+</sup>, 365 (38), 364 (78), 363 (62), 347 (28), 307 (70), 293 (100)

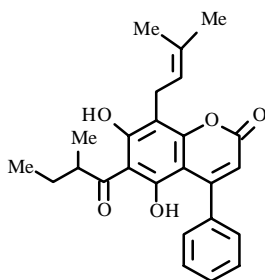
**PMR** (250 MHz, CDCl<sub>3</sub>) [105]: 0.94 (6H, d, J = 7, Me<sub>2</sub>CH), 1.28 and 1.48 (6H, two s, Me<sub>2</sub>CCH), 2.04 (1H, s, Me<sub>2</sub>COH), 2.14 (1H, m, Me<sub>2</sub>CHCH<sub>2</sub>), 2.84 (2H, dd, J = 7, J = 5, CHCH<sub>2</sub>CO), 3.24 (2H, d, J = 9, ArCH<sub>2</sub>CH), 4.80 (1H, t, J = 9,

Me<sub>2</sub>CCHCH<sub>2</sub>, 5.72 (1H, s, PhC=CHCO), 7.0-7.2 (5H, m, ArH), 15.0 (1H, s, OH)

<sup>13</sup>C NMR (67.5 MHz, CDCl<sub>3</sub>) [66]:

C-2	159.8	C-8	92.9	C-3'	127.5	C-3''	25.0
3	111.8	9	26.6	4'	128.2	4''	22.6
4	156.6	9a	105.1	5'	127.5	5''	22.6
4a	102.3	9b	155.5	6'	127.2	1'''	71.4
5	164.3	1'	138.9	1''	205.2	2'''	26.3
6	103.3	2'	127.2	2''	51.9	3'''	24.9
6a	164.2						

**Biol:** cytotoxic activity against cell line KB at ED<sub>50</sub> 6 µg/mL [66]



## 82. Mammae A/AB (MAB 1)

*Mammea africana* [98, 99], *M. americana* [115, 116]

C<sub>25</sub>H<sub>26</sub>O<sub>5</sub>, **mp** 75°C [98], 105-106°C [99], 107-108°C [115], 109-110°C [105]

**UV** (MeOH+HCl) [98]: 232 (4.24), 282 (4.47), 336 (4.01); (MeOH+KOH):

237 (4.35), 300 (4.27), 390 (3.97), 422 (4.03)

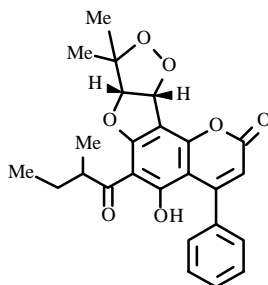
**IR** (CDCl<sub>3</sub>) [105]: 3500, 2950, 1730, 1630

**Mass** [105]: 406 (52) [M]<sup>+</sup>, 351 (29), 349 (100), 294 (28)

**HRMS** [105]: 406.1776 [M]<sup>+</sup>

**PMR** (250 MHz, CDCl<sub>3</sub>) [105]: 0.88 (3H, t, J = 7, MeCH<sub>2</sub>), 1.12 (3H, d, J = 7, MeCH), 1.2-1.8 (2H, m, MeCH<sub>2</sub>CH), 1.80 and 1.94 (6H, two s, Me<sub>2</sub>C=CH), 3.52-3.84 (3H, m, ArCH<sub>2</sub>CH and CH<sub>2</sub>CHCO), 5.4 (1H, t, J = 7, Me<sub>2</sub>C=CHCH<sub>2</sub>), 6.08 (1H, s, PhC=CHCO), 7.44-7.8 (5H, m, ArH), 10.1 (1H, s, OH), 11.2 (1H, s, OH)

**Biol:** inhibitor of oxidative phosphorylation at 0.5 µg/mL [106]



## 83. Mammae A/AB dioxalanocyclo F

*Calophyllum dispar* [67]

C<sub>25</sub>H<sub>24</sub>O<sub>7</sub>

**UV** (EtOH) [67]: 230 (3.83), 281 (4.08), 346 (3.46); (EtOH+NaOH):

256 (4.00), 281 (3.98), 338 (3.34), 416 (3.56)

**IR** (CHCl<sub>3</sub>) [67]: 3440, 1717, 1617, 1559, 768, 702

**Mass** [67]: 436 (41), 403 (25), 389 (57), 379 (61), 378 (41), 363 (20), 347 (37), 321 (90), 305 (45), 171 (30), 43 (100)

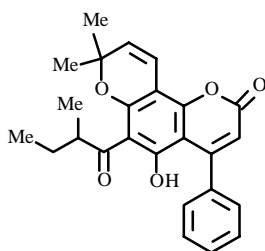
**HREIMS** [67]: 436.1528 [M]<sup>+</sup>

**PMR** (500 MHz, CDCl<sub>3</sub>) [67]: 0.91 (3H, t, J = 7.0, CH<sub>3</sub>-5''), 1.17 (3H, d, J = 7.0, CH<sub>3</sub>-3''), 1.43 (1H, m, CH<sub>2</sub>-4a''), 1.48 (3H, s, CH<sub>3</sub>-4'''), 1.50 (3H, s, CH<sub>3</sub>-5'''), 1.79 (1H, m, CH<sub>2</sub>-4b''), 3.58 (1H, m, H-2''), 5.48 (1H, d, J = 6.0, H-2'''), 6.01 (1H, s, H-3), 6.22 (1H, d, J = 6.0, H-1'''), 7.30 (2H, m, H-2', H-6'), 7.42 (3H, m, H-3', H-4', H-5'), 14.96 (1H, s, OH-5)

**HMBC** [67]

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) [67]:

C-2	158.8	C-8	102.9	C-5'	127.7	C-5''	11.9
3	113.1	8a	156.2	6'	127.2	1'''	81.8
4	157.1	1'	139.0	1''	209.3	2'''	99.4
4a	102.4	2'	127.2	2''	45.7	3'''	85.7
5	166.5	3'	127.7	3''	16.7	4'''	18.4
6	103.2	4'	128.3	4''	26.5	5'''	23.6
7	164.6						



#### 84. Mammae A/AB cyclo D (MAB 5)

*Mammea africana* [98, 99], *Kielmeyera lathrophyton* [111]

$\text{C}_{25}\text{H}_{24}\text{O}_5$ , mp 78-80°C [99], 84-87°C [5], 89.5-92°C [112]

UV (MeOH) [111]: 232, 285, 345; (MeOH+0.1 N HCl) [99]: 233 (4.36), 286 (4.46), 335 (4.76); (MeOH+0.1 N NaOH): 250 (4.30), 310 (4.31), 430 (3.69)

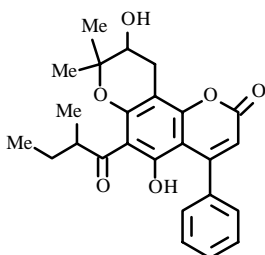
IR ( $\text{CHCl}_3$ ) [111]: 3448, 1748, 1650, 1582, 1132, 1115, 699

Mass [111]: 404 (44)  $[\text{M}]^+$ , 389 (100), 347 (86)

PMR (300 MHz,  $\text{CDCl}_3$ ) [5]: 0.91 (3H, t,  $J = 7.4$ ,  $\text{MeCH}_2\text{CH}$ ), 1.17 (3H, d,  $J = 7.0$ ,  $\text{MeCHCO}$ ), 1.34-1.50 (1H, m,  $\text{MeCH}_2\text{CH}$ ), 1.58 (3H, s,  $\text{Me}_2\text{CH}$ ), 1.59 (3H, s,  $\text{Me}_2\text{CH}$ ), 1.77-1.92 (1H, m,  $\text{MeCH}_2\text{CH}$ ), 3.66-3.78 (1H, m,  $J = 7.0$ ,  $\text{MeCHCO}$ ), 5.64 (1H, d,  $J = 10.1$ ,  $\text{ArCH}=\text{CH}$ ), 6.00 (1H, s,  $\text{C}=\text{CHCO}$ ), 6.91 (1H, d,  $J = 10.1$ ,  $\text{ArCH}=\text{CH}$ ), 7.30-7.45 (5H, m, Ph), 14.70 (1H, s, OH)

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) [111]:

C-2	159.5	C-8	101.4	C-1''	211.3	C-1'''	139.0
3	112.5	8a	157.7	2''	46.5	2'''	127.0
4	154.6	4'	115.4	3''	26.5	3'''	127.5
4a	102.1	5'	126.2	4''	11.7	4'''	128.1
5	164.3	6'	79.7	5''	16.5	5'''	127.5
6	106.8	7'	28.0			6'''	127.0
7	156.3	8'	28.0				



#### 85. Mammae A/AB cyclo E

*Calophyllum dispar* [67]

$\text{C}_{25}\text{H}_{26}\text{O}_6$

UV (EtOH) [67]: 235 (4.11), 282 (4.37), 344 (3.94); (EtOH+NaOH): 254 (4.22), 282 (4.09), 313 (4.01), 426 (3.92)

IR ( $\text{CHCl}_3$ ) [67]: 3400, 1717, 1617, 1561, 768, 704

Mass [67]: 422 (75), 420 (20), 405 (10), 389 (13), 365 (94), 363 (50), 347 (40),

335 (12), 321 (14), 307 (33), 305 (20), 293 (100)

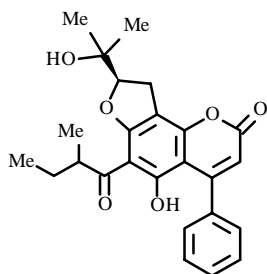
HREIMS [67]: 422.1747  $[\text{M}]^+$

PMR (500 MHz,  $\text{CDCl}_3$ ) [67]: 0.93 (3H, t,  $J = 7.0$ ,  $\text{CH}_3\text{-5''}$ ), 1.16 (3H, d,  $J = 6.5$ ,  $\text{CH}_3\text{-3''}$ ), 1.36 (6H, s,  $\text{CH}_3\text{-4''}$ ,  $\text{CH}_3\text{-5''}$ ), 1.41 (1H, m,  $\text{CH}_2\text{-4a''}$ ), 1.81 (1H, m,  $\text{CH}_2\text{-4b''}$ ), 3.35 (2H, m,  $\text{CH}_2\text{-1''}$ ), 3.64 (1H, m, H-2''), 5.25 (1H, d,  $J = 8.5$ , H-2'''), 5.96 (1H, s, H-3), 7.33 (m, 2H, H-2', H-6'), 7.41 (m, 3H, H-3', H-4', H-5'), 14.67 (s, 1H, OH-7)

HMBC [67]

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) [67]:

C-2	159.8	C-8	104.6	C-5'	127.6	C-5''	11.7
3	112.0	8a	155.5	6'	127.2	1'''	27.1
4	156.7	1'	139.1	1''	209.6	2'''	89.0
4a	102.5	2'	127.2	2''	45.6	3'''	83.0
5	165.0	3'	127.6	3''	16.5	4'''	19.9
6	102.8	4'	128.2	4''	26.4	5'''	20.5
7	163.9						



### 86. Mammaea A/AB cyclo F (MAB 3)

*Mammea americana* [113, 114], *M. africana* [98, 99],

*Kielmeyera pumila* [110], *Calophyllum dispar* [66]

$\text{C}_{25}\text{H}_{26}\text{O}_6$ , mp 115-117°C [114], 134°C [99], 140-142°C [105]

UV (EtOH) [105]: 281, 350; (EtOH+KOH): 246, 315

IR ( $\text{CDCl}_3$ ) [105]: 2950, 1720, 1600

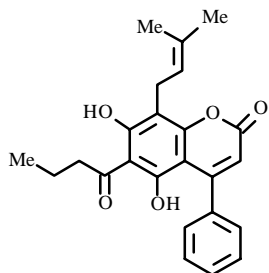
Mass [105]: 422 (100)  $[\text{M}]^+$ , 365 (72), 364 (12), 363 (14), 347 (23), 307 (10), 293 (72)

PMR (250 MHz,  $\text{CDCl}_3$ ) [105]: 0.88 (3H, t,  $J = 7$ ,  $\text{MeCH}_2$ ), 1.12 (3H, dd,  $J = 7$ ,  $J = 2$ ,  $\text{MeCH}$ ), 1.28 and 1.38 (6H, two s,  $\text{Me}_2\text{CCH}$ ), 1.2-1.9 (2H, m,  $\text{MeCH}_2\text{CH}$ ), 1.80 (1H, s,  $\text{Me}_2\text{COH}$ ), 3.24 (2H, d,  $J = 9$ ,  $\text{ArCH}_2\text{CH}$ ), 3.54 (1H, m,  $\text{CH}_2\text{CHCO}$ ), 4.82 (1H, t,  $J = 9$ ,  $\text{Me}_2\text{CCHCH}_2$ ), 5.80 (1H, s,  $\text{PhC}=\text{CHCO}$ ), 7.0-7.4 (5H, m,  $\text{ArH}$ ), 14.28 (1H, s, OH)

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [66]:

C-2	159.8	C-8	92.9	C-3'	127.5	C-3''	16.1
3	111.5	9	26.5	4'	128.1	4''	26.0
4	156.7	9a	105.2	5'	127.5	5''	11.7
4a	102.1	9b	155.3	6'	127.1	1'''	71.3
5	164.7	1'	138.8	1''	209.5	2'''	26.2
6	102.7	2'	127.1	2''	45.5	3'''	24.9
6a	163.9						

Biol: cytotoxic activity against cell line KB at  $\text{ED}_{50}$  5  $\mu\text{g}/\text{mL}$  [66]



### 87. Mammaea A/AC

*Ochrocarpus siamensis* [117], *Mesua racemosa* [101]

$\text{C}_{24}\text{H}_{24}\text{O}_5$ , mp 116-117°C [117]

UV (95% EtOH) [117]: 235 (4.11), 280 (4.21), 335 (3.71)

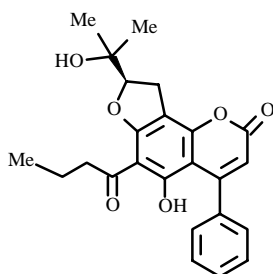
IR (mineral oil) [117]: 3340, 1700, 1620, 1580

Mass [117]: 392 (90)  $[\text{M}]^+$ , 377 (16), 349 (66), 337 (100)

PMR (60 MHz,  $\text{CDCl}_3$ ) [117]: 0.93 (t,  $J = 7$ , 3H), 1.38-1.95 (2H, m), 1.76 (d,  $J = 0.5$ , 3H), 1.90 (d,  $J = 0.5$ , 3H), 2.98 (t,  $J = 7$ , 2H), 3.57 (t,  $J = 7.5$ , 2H), 5.31 (t,  $J = 7.5$ , 1H), 5.96 (s, 1H), 7.52 (s, 5H), 9.72 (s, 1H), 11.15 (s, 1H)

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	159.2	C-7	156.6	C-4'	129.7	C-4''	13.7
3	112.7	8	107.5	5'	129.1	1'''	21.7
4	159.5	8a	163.3	6'	127.4	2'''	120.6
4a	101.3	1'	137.2	1''	207.4	3'''	135.4
5	154.4	2'	127.4	2''	46.8	4'''	18.0
6	107.9	3'	129.1	3''	17.6	5'''	25.8



### 88. Mammaea A/AC cyclo F

*Mammea americana* [114], *Mesua racemosa* [101],

*Calophyllum dispar* [66]

$\text{C}_{24}\text{H}_{24}\text{O}_6$ , mp 119°C [101]

$[\alpha]_{\text{D}} -2.6^\circ$  ( $\text{CHCl}_3$ ) [101]

UV (EtOH) [101]: 230 (3.76), 279 (4.03), 349 (3.64)

IR (KBr) [101]: 3566, 1734, 1704, 1614

Mass [101]: 409 (23), 408 (100)  $[\text{M}]^+$ , 375 (14), 365 (40), 351 (12), 350 (54), 349 (48), 337 (19), 322 (25), 321 (25), 307 (50), 305 (12), 294 (14), 293 (50), 279 (12), 105 (11), 59 (22), 43 (21)

HREIMS [101]: 408.1591

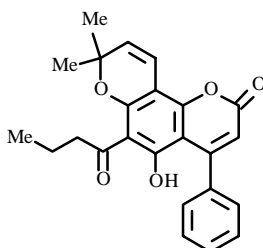
PMR (270 MHz,  $\text{CDCl}_3$ ) [101]: 0.99 (3H, t,  $J = 7.5$ ,  $\text{CH}_3$ -4''), 1.32 (3H, s,  $\text{CH}_3$ -2'''), 1.44 (3H, s,  $\text{CH}_3$ -3'''), 1.68 (2H, m,  $\text{CH}_2$ -3''), 3.00 (2H, m,  $\text{CH}_2$ -2''), 3.32 (2H, dd,  $J = 6.0$ ,  $J = 3.0$ ,  $\text{CH}_2$ -9), 4.92 (1H, t,  $J = 9.0$ , H-8), 5.95 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 14.39 (1H, s, OH-5)

HMBC, HMQC [101]

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	159.7	C-6a	164.3	C-2'	127.2	C-2''	45.2
3	112.1	8	92.8	3'	127.6	3''	17.9
4	156.5	9	26.8	4'	128.3	4''	13.8
4a	102.2	9a	105.1	5'	127.6	1'''	71.6
5	163.8	9b	155.3	6'	127.2	2'''	24.8
6	103.0	1'	139.1	1''	205.4	3'''	26.2

Biol: cytotoxic activity against cell line KB at  $\text{ED}_{50}$  6  $\mu\text{g}/\text{mL}$  [66]



### 89. Mammaea A/AC cyclo D (6-butyryl-5-hydroxy-4-phenylseselin)

*Ochrocarpus siamensis* [117], *Mesua racemosa* [101],

*Mammea siamensis* [118]

$\text{C}_{24}\text{H}_{22}\text{O}_5$ , mp 138-139°C [117]

UV (EtOH) [117]: 232 (4.60), 282 (4.68), 350 (3.97)

IR (mineral oil) [117]: 1725, 1630, 1600, 1575

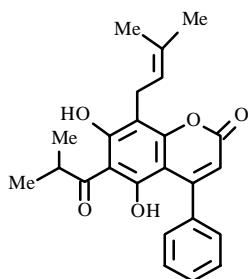
Mass [117]: 390 (44)  $[\text{M}]^+$ , 375 (100), 357 (16), 347 (14)

PMR (60 MHz,  $\text{CDCl}_3$ ) [117]: 0.98 (3H, t,  $J = 7.0$ ), 1.43-1.90 (2H, m), 1.65 (6H, s), 3.98 (2H, t,  $J = 7.0$ ), 5.65 (1H, d,  $J = 10.0$ ), 6.01 (1H, s), 6.94 (1H, d,  $J = 10.0$ ), 7.40 (5H, s), 14.73 (1H, s)



$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	160.1	C-6a	155.2	C-1'	139.7	C-1''	207.5
3	113.1	8	80.3	2'	127.6	2''	47.3
4	156.8	9	116.0	3'	128.1	3''	18.7
4a	102.6	10	126.8	4'	128.7	4''	14.3
5	164.8	10a	101.9	5'	128.1	1'''	28.7
6	107.4	10b	158.7	6'	127.6	2'''	28.7



### 90. Mammaea A/AD (mesuol)

*Mesua ferrea* [104], *M. thwaitesii* [102], *Mammea americana* [119],  
*M. africana* [6, 104]

$\text{C}_{24}\text{H}_{24}\text{O}_5$ , mp 146-148°C [105], 154°C [119], 183-184°C [104]

$[\alpha]_D^{25} +66^\circ$  [104]

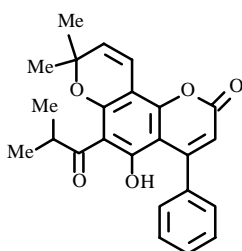
UV (EtOH+KOH) [119]: 234 (4.33), 303 (4.21), 394 (3.84), 426 (4.03)

IR ( $\text{CDCl}_3$ ) [105]: 3450, 1730, 1630

Mass [105]: 392 (35)  $[\text{M}]^+$ , 349 (75), 337 (18), 293 (100)

HRMS [105]: 392.1614  $[\text{M}]^+$

PMR (250 MHz,  $\text{CDCl}_3$ ) [105]: 1.10 (6H, d,  $J = 7$ ,  $\text{Me}_2\text{CH}$ ), 1.74 and 1.88 (6H, two s,  $\text{Me}_2\text{C}=\text{CH}$ ), 3.54 (2H, d,  $J = 7$ ,  $\text{ArCH}_2\text{CH}$ ), 3.72 (1H, m,  $J = 7$ ,  $\text{Me}_2\text{CHCO}$ ), 5.22 (1H, t,  $J = 7$ ,  $\text{Me}_2\text{C}=\text{CHCH}_2$ ), 5.90 (1H, s,  $\text{PhC}=\text{CHCO}$ ), 7.2-7.5 (5H, m,  $\text{ArH}$ ), 9.84 (1H, s, OH), 10.90 (1H, s, OH)



### 91. Mammaea A/AD cyclo D (mesuagin)

*Mesua ferrea* [109], *M. thwaitesii* [102], *M. racemosa* [101],  
*Mammea americana* [6]

$\text{C}_{24}\text{H}_{22}\text{O}_5$ , mp 151-153°C [104], 152-153°C [109]

UV [109]: 235 (4.31), 285-286 (4.40), 362 (3.79)

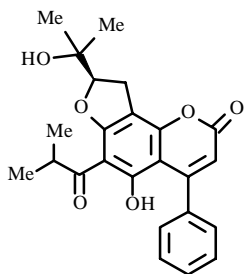
IR (KBr) [109]: 3400, 1739, 1653, 1613, 1378, 709

PMR ( $\text{CDCl}_3$ ) [109]: 1.25 (6H, d), 1.58 (6H, s), 3.73 (1H, m), 5.62 (1H, d),  
5.97 (1H, s), 6.88 (1H, d), 7.33 (5H, m), 14.63 (1H, s)

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	159.7	C-6a	154.8	C-1'	139.3	C-1''	211.5
3	112.7	8	79.9	2'	127.2	2''	39.9
4	156.5	9	115.5	3'	127.7	3a''	19.3
4a	102.6	10	126.3	4'	128.3	3b''	19.3
5	164.6	10a	101.5	5'	127.7	1'''	28.2
6	106.4	10b	157.8	6'	127.2	2'''	28.2

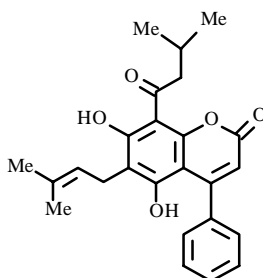
**Biol:** antibacterial activity against *Staphylococcus aureus* [109]



### 92. Mammea A/AD cyclo F

*Mammea americana* [114]

$C_{24}H_{24}O_6$



### 93. Mammea A/BA

*Mammea americana* [115, 116], *M. africana* [116]

$C_{25}H_{26}O_5$ , **mp** 125-126°C [115]

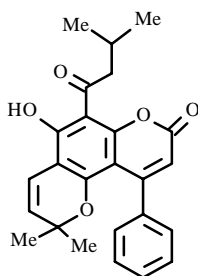
**UV** (EtOH) [116]: 227 (4.43), 234 (4.43), 270 (4.17), 297 (4.24), 335 (4.37); (EtOH+HCl): 225 (4.46), 294 (4.36), 332 (4.25); (EtOH+KOH): 233 (4.40), 261 (4.19), 337 (4.60)

**IR** [116]: 3480, 1723, 1610, 1595

**Mass** [116]: 406 [M]<sup>+</sup>, 363, 351, 349, 293, 265

**PMR** [116]: 1.04 (6H, d, J = 7.0, two CH<sub>3</sub>-3'), 1.63 (3H, s, CH<sub>3</sub>-3''), 1.68 (3H, s, CH<sub>3</sub>-3''), 2.2 (1H, m, H-3'), 3.08 (2H, d, J = 7.0, CH<sub>2</sub>-2'), 3.19 (2H, d, J = 7.0, CH<sub>2</sub>-1''), 5.03 (1H, m, H-2'), 5.79 (1H, s, H-3), 5.90 (1H, s, OH-5), 7.43 (5H, s, Ph-4), 14.53 (1H, s, OH-7)

**Biol**: inhibitor of oxidative phosphorylation at 0.5 µg/mL [106]



### 94. Mammea A/BA cyclo D (isomammeigin)

*Kielmeyera pumila* [110]

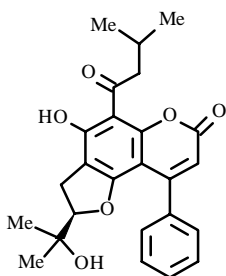
$C_{25}H_{24}O_5$ , **mp** 173-175°C [110]

**UV** (EtOH) [110]: 265 (3.39), 273 (4.39), 314 (4.35), 373 (3.66); (EtOH+NaOH): 230 (4.25), 252 (4.36), 312 (4.41); (EtOH+NaOAc): 240 (4.29), 305 (4.32)

**IR** (KBr) [110]: 3400, 2920, 1745, 1635, 1605, 1580, 1550, 1380, 1190, 1175, 1150, 1135, 910, 870, 790, 755, 700

**Mass** [110]: 404 (22) [M]<sup>+</sup>, 389 (100), 371 (11), 347 (10)

**PMR** (100 MHz, CDCl<sub>3</sub>) [110]: 0.96 (6H, s, two Me-2), 1.07 (6H, d, J = 7.0, two Me-3'), 2.29 (1H, m, CH-3'), 3.18 (2H, d, J = 7.0, CH<sub>2</sub>-2'), 5.37 (1H, d, J = 10.0, H-3), 5.98 (1H, s, H-9), 6.60 (1H, d, J = 10.0, H-4), 7.28 (5H, s, Ph-10), 12.27 (1H, s, OH-5)



### 95. Mammea A/BA cyclo f

*Calophyllum dispar* [66]

$C_{25}H_{24}O_5$ , **mp** 113-115°C [66]

**UV** (EtOH) [66]: 226 (4.10), 234 (4.10), 300 (4.11), 327 (3.87); (EtOH+NaOH): 252 (4.10), 283 (3.7), 389 (3.97)

**IR** (CHCl<sub>3</sub>) [66]: 3470, 1725, 1605, 1561, 756, 702

**HRFABMS** [66]: 421.1674 [M - H]<sup>-</sup>

**PMR** (270 MHz, CDCl<sub>3</sub>) [66]: 0.94 (s, 3H, CH<sub>3</sub>-6''), 1.02 (s, 3H, CH<sub>3</sub>-5''), 1.08 (d, J = 6.5, 6H, CH<sub>3</sub>-4''', CH<sub>3</sub>-5'''), 2.32 (m, 1H, H-3'''), 2.93 (dd, J = 8.5, J = 15.5, 1H, CH<sub>2</sub>-3''β), 3.08 (dd, J = 10.0, J = 15.5, 1H, CH<sub>2</sub>-3''α), 3.19 (d, J = 7.0, 2H, CH<sub>2</sub>-2'''), 4.52 (t, J = 9.0, 1H, H-2''), 6.08 (s, 1H, H-3), 7.32 (m, 2H, H-2',

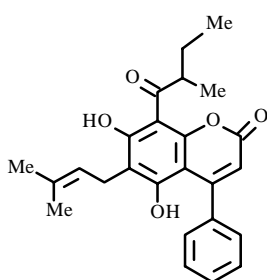
H-6'), 7.44 (m, 3H, H-3', H-4', H-5'), 14.33 (s, 1H, OH-7)

**DQF-COSY, HMQC, HMBC, NOESY** [66]

**<sup>13</sup>C NMR** (67.5 MHz, CDCl<sub>3</sub>) [66]:

C-2	159.1	C-9	157.3	C-5'	127.9	C-6''	24.8
3	111.0	10	98.6	6'	127.4	1'''	206.1
4	154.9	1'	138.0	2''	92.7	2'''	53.4
5	161.9	2'	127.4	3''	26.8	3'''	25.6
6	110.0	3'	127.9	4''	71.6	4'''	22.7
7	163.7	4'	128.8	5''	23.2	5'''	22.7
8	105.0						

**Biol:** cytotoxic activity against cell line KB at ED<sub>50</sub> 9 µg/mL [66]



### 96. Mammea A/BB

*Mammea americana* [115, 116], *Mesua racemosa* [101]

C<sub>25</sub>H<sub>26</sub>O<sub>5</sub>, **mp** 124-125°C [116]

**UV** (EtOH) [116]: 235 (4.43), 295 (4.28), 335 (4.31); (EtOH+HCl): 227 (4.45), 294 (4.38), 333 (4.24); (EtOH+KOH): 234 (4.39), 263 (4.19), 337 (4.58)

**IR** [116]: 3485, 1725, 1615, 1600, 1555

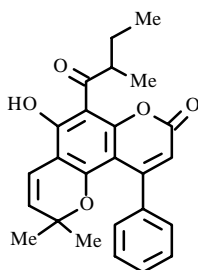
**Mass** [116]: 406 [M]<sup>+</sup>, 363, 351, 349, 293

**PMR** [116]: 1.02 (t, J = 7.0, 3H, CH<sub>3</sub>-4'), 1.26 (d, J = 7.0, 3H, CH<sub>3</sub>-2'), 1.60 (m, 2H, CH<sub>2</sub>-3'), 1.64 (s, 3H, CH<sub>3</sub>-3''), 1.70 (s, 3H, CH<sub>3</sub>-3'''), 3.20 (d, J = 7.0, 2H, CH<sub>2</sub>-1''), 3.87 (m, 1H, H-2'), 5.10 (m, 1H, H-2''), 5.82 (s, 2H, OH-5, H-3), 7.46 (s, 5H, Ph-4), 14.51 (s, 1H, OH-7)

**<sup>13</sup>C NMR** (67.5 MHz, CDCl<sub>3</sub>) [101]:

C-2	159.2	C-8	104.1	C-5'	129.5	C-5''	25.6
3	112.1	8a	155.7	6'	127.5	1'''	210.1
4	154.1	1'	136.8	1''	21.6	2'''	47.0
4a	100.5	2'	127.5	2''	120.8	2a'''	16.6
5	157.0	3'	129.5	3''	134.1	3'''	27.2
6	112.6	4'	130.2	4''	17.9	4'''	11.8
7	166.9						

**Biol:** inhibitor of oxidative phosphorylation at 0.5 µg/mL [106]



### 97. Mammea A/BB cyclo D (ponnalide)

*Mammea americana* [105], *Calophyllum inophyllum* [87]

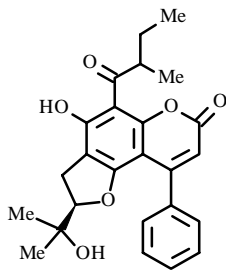
C<sub>25</sub>H<sub>24</sub>O<sub>5</sub>, **mp** 126-127°C [5], 127-129°C [105]

**UV** (EtOH) [105]: 263, 271, 311, 385; (EtOH+KOH): 263, 323, 402

**IR** (CDCl<sub>3</sub>) [105]: 1730, 1640, 1600

**PMR** (250 MHz, CDCl<sub>3</sub>) [105]: 1.0 (6H, s, Me<sub>2</sub>C), 1.04 (3H, t, J = 7, MeCH<sub>2</sub>), 1.32 (3H, d, J = 7, MeCH), 1.3-2.1 (2H, m, MeCH<sub>2</sub>CH), 4.0 (1H, m,

CH<sub>2</sub>CHCO), 5.52 (1H, d, J = 11, ArCH=CH), 6.16 (1H, s, PhC=CHCO), 6.76 (1H, d, J = 11, ArCH=CH), 7.3-7.6 (5H, m, ArH), 14.90 (1H, s, OH)



### 98. Mammea A/BB cyclo F

*Calophyllum dispar* [66]

$C_{25}H_{24}O_5$

**UV** (EtOH) [66]: 227 (4.21), 235 (4.19), 299 (4.22), 325 (4.02);  
(EtOH+NaOH): 252 (4.24), 283 (3.90), 389 (4.07)

**IR** ( $CHCl_3$ ) [66]: 3460, 1732, 1607, 1559, 758, 704

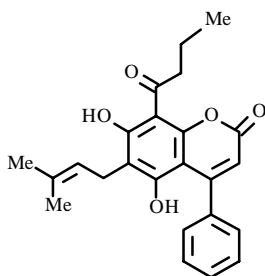
**HRFABMS** [66]: 421.1639 [M - H]<sup>-</sup>

**PMR** (270 MHz,  $CDCl_3$ ) [66]: 0.94 (3H, s,  $CH_3-6''$ ), 1.01 (3H, t,  $J = 7.0$ ,  $CH_3-5'''$ ), 1.02 (3H, s,  $CH_3-5''$ ), 1.28 (3H, d,  $J = 7.0$ ,  $CH_3-3'''$ ), 1.50 (1H, m,  $CH_2-4'''a$ ), 1.94 (1H, m,  $CH_2-4'''b$ ), 2.93 (1H, dd,  $J = 8.0$ ,  $J = 15.0$ ,  $CH_2-3''\beta$ ), 3.07 (1H, dd,  $J = 10.0$ ,  $J = 15.0$ ,  $CH_2-3''\alpha$ ), 3.96 (1H, m, H-2'''), 4.52 (1H, t,  $J = 9.0$ , H-2''), 6.07 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.44 (3H, m, H-3', H-4', H-5'), 14.32 (1H, s, OH-7)

**$^{13}C$  NMR** (67.5 MHz,  $CDCl_3$ ) [66]:

C-2	159.1	C-9	157.1	C-5'	127.9	C-6''	24.8
3	111.0	10	98.7	6'	127.4	1'''	210.4
4	154.9	1'	138.1	2''	92.6	2'''	46.7
5	161.8	2'	127.4	3''	26.6	3'''	16.5
6	110.1	3'	127.9	4''	71.6	4'''	27.1
7	163.9	4'	128.8	5''	23.2	5'''	11.8
8	104.5						

**Biol:** cytotoxic activity against cell line KB at  $ED_{50}$  15  $\mu g/mL$  [64]



### 99. Mammea A/BC

*Mesua racemosa* [72]

$C_{24}H_{24}O_5$ , mp 123-124°C [72]

**UV** (EtOH+HCl) [72]: 225 (4.09), 294 (4.03), 331 (3.85); (EtOH+NaOH):  
235 (4.21), 334 (4.17), 384 (3.73)

**IR** [72]: 3486, 1742, 1705, 1616, 1595, 1389, 1132, 769, 704

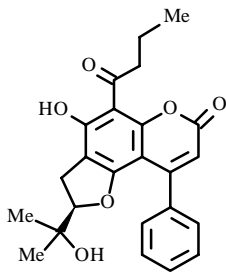
**HRLSIMS** [72]: 391.1526 [M - H]<sup>+</sup>

**PMR** (270 MHz,  $CDCl_3$ ) [72]: 1.03 (3H, t,  $J = 7.0$ ,  $CH_3-4'''$ ), 1.66 (3H, s,  $CH_3-4''$ ), 1.71 (3H, s,  $CH_3-5''$ ), 1.83 (2H, m,  $CH_2-3'''$ ), 3.30 (2H, m,  $CH_2-2''$ ), 3.33 (2H, m,  $CH_2-1''$ ), 5.09 (1H, t,  $J = 1.0$ , H-2''), 5.96 (1H, s, OH-5), 6.01 (1H, s, H-3), 7.44 (2H, m, H-2', H-6'), 7.57 (3H, m, H-3', H-4', H-5'), 14.58 (1H, s, OH-7).

**HMBC** [72]

**$^{13}C$  NMR** (67.5 MHz,  $CDCl_3$ ) [72]:

C-2	158.7	C-7	166.7	C-4'	130.2	C-4''	25.7
3	112.5	8	104.5	5'	129.6	5''	17.9
4	154.1	8a	156.0	6'	127.5	1'''	206.3
4a	100.4	1'	136.8	1''	21.5	2'''	46.7
5	157.0	2'	127.5	2''	120.8	3'''	18.1
6	112.1	3'	129.6	3''	134.1	4'''	13.8



### 100. Mammaea A/BC cyclo F

*Calophyllum dispar* [66]

$C_{24}H_{24}O_6$

**UV** (EtOH) [66]: 227 (4.30), 235 (4.28), 299 (4.31), 331 (4.03);

(EtOH+NaOH): 253 (4.32), 282 (3.92), 389 (4.18)

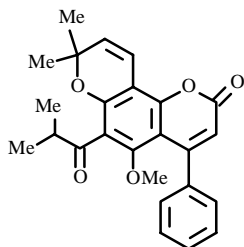
**IR** ( $CHCl_3$ ) [66]: 3460, 1732, 1607, 1559, 758, 704

**HRFABMS** [66]: 407.525  $[M - H]^-$

**PMR** (270 MHz,  $CDCl_3$ ) [66]: 0.94 (3H, s,  $CH_3-6''$ ), 1.01 (3H, t,  $J = 7.0$ ,  $CH_3-5''$ ), 1.02 (3H, s,  $CH_3-5''$ ), 1.28 (3H, d,  $J = 7.0$ ,  $CH_3-3''$ ), 1.50 (1H, m,  $CH_2-4''a$ ), 1.94 (1H, m,  $CH_2-4''b$ ), 2.93 (1H, dd,  $J = 8.0$ ,  $J = 15.0$ ,  $CH_2-3''\beta$ ), 3.08 (1H, dd,  $J = 10.0$ ,  $J = 15.0$ ,  $CH_2-3''\alpha$ ), 3.96 (1H, m, H-2''), 4.52 (1H, t,  $J = 9.0$ , H-2''), 6.07 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.44 (3H, m, H-3', H-4', H-5'), 14.33 (1H, s, OH-7)

**$^{13}C$  NMR** (67.5 MHz,  $CDCl_3$ ) [66]:

C-2	159.1	C-9	157.1	C-5'	127.9	C-6''	24.8
3	111.0	10	98.7	6'	127.4	1'''	210.4
4	154.9	1'	138.1	2''	92.6	2'''	46.7
5	161.8	2'	127.4	3''	26.6	3'''	16.5
6	110.1	3'	127.9	4''	71.6	4'''	27.1
7	163.9	4'	128.8	5''	23.2	5'''	11.8
8	104.5						



### 101. Mesuarin

*Mesua ferrea* [120]

$C_{25}H_{24}O_5$ , **mp** 130°C [120]

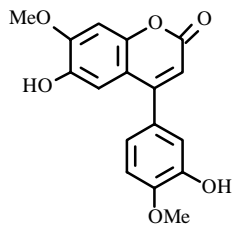
**UV** (EtOH) [120]: 233 (4.63), 282 (4.35), 360 (4.1)

**IR** (KBr) [120]: 1740, 1710, 1600, 1500, 1200, 700

**Mass** [120]: 420  $[M]^+$ , 405, 377

**PMR** (90 MHz,  $DMSO-d_6$ ) [120]: 1.35 (6H, d,  $J = 10.0$ , two Me-2'), 1.55 (s, 6H, two Me-8), 3.01 (s, 3H, OMe-5), 3.72 (1H, m, H-2'), 5.65 (1H, d,  $J = 10.0$ , H-9), 6.0 (s, 1H, H-3), 6.50 (1H, d,  $J = 10.0$ , H-10), 7.30 (m, 5H, Ph-4)

**Biol**: antibacterial activity against *Bacillus firmis* [120]



### 102. Melanein

*Dalbergia baroni* [52, 53], *D. melanoxylon* [43, 121]

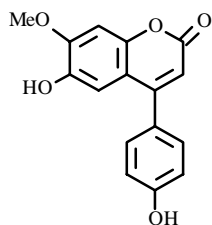
$C_{17}H_{14}O_6$ , **mp** 220.5-222°C [43], 221-223°C [52, 53]

**UV** (EtOH) [53]: 234 (4.38), 256 (4.17), 308 (4.0), 344 (4.09)

**IR** (KBr) [53]: 3289, 2950, 1664, 1616, 1274, 1244, 1174, 1138, 1026, 817

**Mass** [53]: 299, 286, 271, 243

**PMR** (60 MHz,  $DMSO-d_6$ ) [53]: 2.5 (1H, s, OH), 3.2 (1H, s, OH), 3.85 (3H, s, OMe), 3.88 (3H, s, OMe), 6.1 (1H, s, H-3), 6.9-7.18 (5H, m)



### 103. Melanetin

*Dalbergia melanoxyton* [122], *D. odorifera* [30, 31]

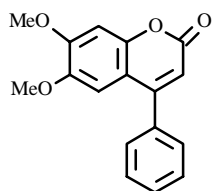
$C_{16}H_{12}O_5$ , mp 233-234°C [123], 236-237°C [124]

UV (EtOH) [123]: 230 (4.50), 263 (4.09), 312 (4.27), 350 (4.21)

IR (KBr) [123]: 3430, 1660

$^{13}C$  NMR (75.4 MHz, DMSO- $d_6$ ) [30]:

C-2	160.39	C-6	143.34	C-10	111.06	C-4'	158.67
3	110.28	7	151.67	1'	125.76	5'	115.48
4	155.03	8	100.43	2'	129.76	6'	129.76
5	110.62	9	148.33	3'	115.48	7-OMe	56.04



### 104. O-Methyldalbergin

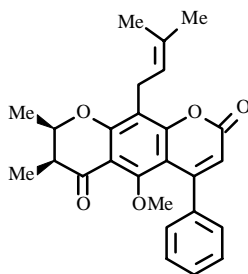
*Dalbergia latifolia* [42], *D. nigra* [44], *D. sissoo* [125],

*Machaerium scleroxylon* [57], *M. pedicellatum* [58], *Prosopis kuntzei* [60],  
*Goniorrhachis marginata* [61]

$C_{17}H_{14}O_4$ , mp 145-146°C [40], 146-147°C [16]

IR (KBr) [54]: 1727; (CHCl<sub>3</sub>): 1709

Mass [126]: 282 (100) [M]<sup>+</sup>, 254 (29.5), 239 (21.5), 224 (12.5), 211 (4.2), 152 (6.6)



### 105. O-methylisocalaustralin

*Calophyllum teysmanii* var. *inophyllolide* [14]

$C_{26}H_{26}O_5$

$[\alpha]_D -10.8^\circ$  (CHCl<sub>3</sub>) [14]

UV (EtOH) [14]: 232, 272, 324

IR (KBr) [14]: 1743, 1695, 1578, 1384, 1327, 1156, 1108, 864, 768, 698

Mass [14]: 418 (86) [M]<sup>+</sup>, 403 (80), 363 (70), 349 (80), 319 (90), 307 (80)

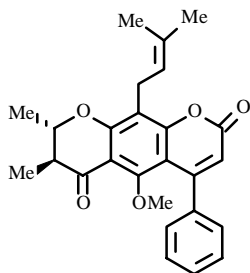
HREIMS [14]: 418.1763 [M]<sup>+</sup>

PMR (300 MHz, CDCl<sub>3</sub>) [14]: 1.13 (3H, d, J = 7.2, CH<sub>3</sub>-16), 1.41 (3H, d, J = 6.6, CH<sub>3</sub>-17), 1.71 (3H, s, CH<sub>3</sub>-15), 1.87 (3H, s, CH<sub>3</sub>-14), 2.67 (1H, dq, J = 3.5, J = 7.2, H-7), 3.10 (3H, s, OMe-5), 3.54 (2H, d, J = 7.5, CH<sub>2</sub>-11), 4.69 (1H, dq, J = 3.5, J = 6.6, H-8), 5.26 (1H, m, J = 7.5, H-12), 6.06 (1H, s, H-3), 7.32-7.39 (5H, m, Ph)

NOE, HMQC, HMBC [14]

$^{13}C$  NMR (75 MHz, CDCl<sub>3</sub>) [14]:

C-2	159.5	C-7	46.1	C-13	133.1	C-2'	127.5
3	114.2	8	76.5	14	25.9	3'	127.6
4	156.6	9	158.6	15	18.1	4'	128.1
4a	108.5	10	115.1	16	9.2	5'	127.6
5	161.5	10a	155.6	17	16.0	6'	127.5
5a	111.0	11	22.2	1'	139.0	OMe	62.5
6	193.6	12	120.8				



### 106. O-Methylcalaustralin

*Calophyllum teysmannii* var. *inophyllolide* [14]

$C_{26}H_{26}O_5$

$[\alpha]_D -18^\circ$  ( $CHCl_3$ ) [14]

UV (EtOH) [14]: 232, 268, 324, 346

IR (KBr) [14]: 1741, 1691, 1577, 1460, 1409, 1384, 1324, 1260, 1124, 1039, 904, 860, 769, 702

Mass [14]: 418 (70)  $[M]^+$ , 403 (70), 363 (40), 349 (65), 319 (90), 307 (60)

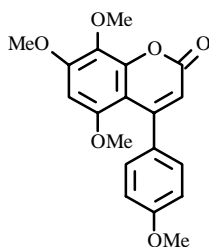
HREIMS [14]: 418.1807  $[M]^+$

PMR (300 MHz,  $CDCl_3$ ) [14]: 1.18 (3H, d,  $J = 7.1$ ,  $CH_3$ -16), 1.55 (3H, d,  $J = 6.3$ ,  $CH_3$ -17), 1.71 (3H, s,  $CH_3$ -15), 1.88 (3H, s,  $CH_3$ -14), 2.55 (1H, dq,  $J = 12.0$ ,  $J = 7.1$ , H-7), 3.10 (3H, s, OMe-5), 3.54 (2H, d,  $J = 7.3$ ,  $CH_2$ -11), 4.27 (1H, dq,  $J = 12.0$ ,  $J = 6.3$ , H-8), 5.27 (1H, m,  $J = 7.3$ , H-12), 6.07 (1H, s, H-3), 7.30-7.39 (5H, m, Ph)

NOE, HMQC, HMBC [14]

$^{13}C$  NMR (75 MHz,  $CDCl_3$ ) [14]:

C-2	159.4	C-7	47.4	C-13	132.9	C-2'	127.4
3	114.0	8	78.7	14	25.7	3'	127.4
4	156.4	9	158.2	15	17.9	4'	128.1
4a	108.3	10	115.0	16	10.4	5'	127.4
5	161.6	10a	155.5	17	19.5	6'	127.4
5a	111.5	11	22.0	1'	138.8	OMe	62.5
6	192.1	12	120.7				



### 107. 8-O-Methylexostemin

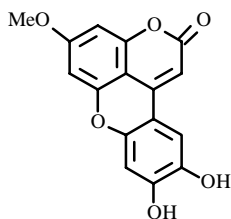
*Coutarea hexandra* [25], *Exostema mexicanum* [26]

$C_{19}H_{18}O_6$ , mp 143-145°C [127]

IR (KBr) [127]: 1722

PMR (200 MHz,  $CDCl_3$ ) [127]: 3.35 (3H, s, OMe-5), 3.91 (3H, s, OMe-4'), 3.95 (3H, s, OMe-8), 3.97 (3H, s, OMe-7), 5.80 (1H, s, H-3), 6.34 (1H, s, H-6), 6.92 (2H, d,  $J = 8.5$ , H-3', H-5'), 7.24 (2H, d,  $J = 8.5$ , H-2', H-6')

Biol: inhibitor of adenosine-3',5'-cyclomonophosphate-phosphodiesterase at  $IC_{50} = 13.3 \times 10^{-5}$  M [34], antiplasmodial activity against *Plasmodium falciparum* [26]



### 108. 7-Methoxy-4',5'-dihydroxy-4-phenyl-5,2'-oxidocoumarin

*Exostema caribaeum* [11], *Hintonia latiflora* [128]

$C_{16}H_{10}O_6$ , mp 300°C (dec.) [11], 342°C (dec.) [19], 342-345°C [128]

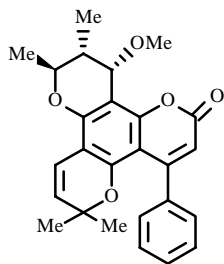
UV (MeOH) [19]: 260, 310, 375, 390; (MeOH+ $AlCl_3$ ) [19]: 275, 325, 406; (MeOH+ $AlCl_3$ +HCl) [19]: 260, 310, 375, 390

IR (KBr) [19]: 3450, 1700, 1640, 1525, 1480, 1313, 1250, 1180

Mass [128]: 298 (100)  $[M]^+$ , 270 (49), 242 (70)

PMR ( $DMSO-d_6$ ) [128]: 3.86 (3H, s, 7-OMe), 6.07 (1H, s, H-3), 6.71 (1H, d,  $J = 2.2$ , H-8), 6.74 (1H, d,  $J = 2.2$ , H-6), 6.76 (1H, s, H-3'), 7.32 (1H, s, H-6')

$^{13}C$  NMR ( $DMSO-d_6$ ) [128]: 56.02, 92.37, 95.97, 96.41, 99.72, 103.22, 106.13, 109.03, 141.17, 143.69, 146.44, 150.59, 152.25, 154.46, 160.91, 162.99



### 109. 12-Methoxyinophyllum P

*Calophyllum teysmannii* var. *inophylloide* [20]

$C_{26}H_{26}O_5$

$[\alpha]_D^{25} +25.5^\circ$  ( $CHCl_3$ ) [20]

UV [20]: 232, 278, 286, 334

IR [20]: 2983, 2914, 1725, 1630, 1570, 1560, 1455, 1382, 1127, 857, 750, 705, 683

Mass [20]: 418 (42)  $[M]^+$ , 403 (100), 388 (32), 372 (50), 347 (82)

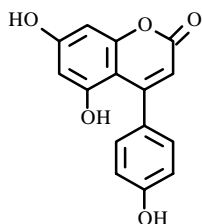
HREIMS [20]: 418.1783  $[M]^+$

PMR (500 MHz,  $CDCl_3$ ) [20]: 0.92 (3H, s,  $CH_3$ -14), 0.94 (3H, s,  $CH_3$ -13), 1.17 (3H, d,  $J = 6.7$ , H-16), 1.41 (3H, d,  $J = 6.3$ , H-15), 1.74 (1H, ddq,  $J = 2.8$ ,  $J = 6.7$ ,  $J = 11.0$ , H-11), 3.65 (3H, s, MeO), 4.32 (1H, dq,  $J = 6.3$ ,  $J = 11.0$ , H-10), 4.62 (1H, d,  $J = 2.8$ , H-12), 5.34 (1H, d,  $J = 10.2$ , H-7), 5.95 (1H, s, H-3), 6.53 (1H, d,  $J = 10.2$ , H-8), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5')

$^{13}C$  NMR (125 MHz,  $CDCl_3$ ) [20]:

C-2	160.5	C-8	116.1	C-12b*	153.8	C-1'	140.2
3	111.7	8a	106.1	13	26.88	2'	127.5
4	156.2	8b*	153.5	14	26.93	3'	127.4
4a	102.7	10	73.5	15	19.1	4'	127.4
4b	151.2	11	38.6	16	13.3	5'	127.4
6	77.1	12	70.8			6'	127.5
7	127.4	12a	104.5			OMe	59.4

\*Assignments may be reversed



### 110. Nivegin

*Echinops niveus* [129, 130]

$C_{15}H_{10}O_5$ , mp 260°C [131], 262-264°C [130], 267-269°C [132], 293-294°C [27], 294-295°C [64]

UV (EtOH) [130]: 267 (4.14), 338 (4.20); (EtOH+NaOH): 278 (4.09), 400 (4.24)

IR (KBr) [130]: 3300, 1660, 1610, 1550, 1500, 1445, 1355, 1249, 1175, 825

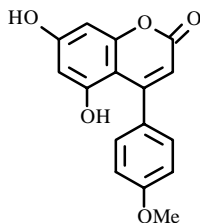
HRMS [130]: 270.0528 (100)  $[M]^+$ , 242.0576 (18), 153 (22), 152 (15), 149 (24), 121 (15), 97 (10), 71 (15), 57 (28)

PMR (90 MHz,  $DMSO-d_6$ ) [130]: 6.16 (1H, d,  $J = 2.12$ ), 6.44 (1H, d,  $J = 2.12$ ), 6.65 (1H, s), 6.89 (2H, d,  $J = 8.76$ ), 7.86 (2H, d,  $J = 8.76$ )

$^{13}C$  NMR (22.5 MHz,  $DMSO-d_6$ ) [130]:

C-2	168.7	C-5	156.3	C-8a	162.1	C-4'	161.8
3	103.6	6	99.5	1'	122.2	5'	116.6
4	164.7	7	164.8	2'	129.0	6'	129.0
4a	104.5	8	94.6	3'	116.6		





### 111. Nivetin

*Echinops niveus* [130]

$C_{16}H_{12}O_5$ , **mp** 256-258°C [27], 263-265°C [64], 314-316°C (dec.) [130]

**UV** (MeOH) [130]: 270 (4.14), 335 (4.15); (MeOH+NaOH): 282 (4.08), 392 (4.16)

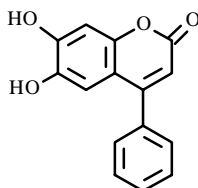
**IR** (KBr) [130]: 3100-3400, 2870, 1665, 1615, 1560, 1510, 1370, 1250, 1200, 848

**Mass** [130]: 284 (100)  $[M]^+$ , 270 (20), 269 (16), 256 (18), 153 (20), 152 (15), 149 (15), 121 (12), 48 (25)

**PMR** (90 MHz, DMSO- $d_6$ ) [130]: 3.85 (s, 3H), 6.17 (d,  $J = 2$ , 1H), 6.44 (d,  $J = 2$ , 1H), 6.66 (s, 1H), 6.96 (d,  $J = 8.7$ , 2H), 7.85 (d,  $J = 8.7$ , 2H)

$^{13}C$  NMR (22.5 MHz, DMSO- $d_6$ ) [130]:

C-2	168.7	C-5	156.3	C-8a	162.1	C-4'	166.5
3	103.6	6	99.5	1'	122.3	5'	113.8
4	164.7	7	164.8	2'	128.5	6'	128.5
4a	104.5	8	94.6	3'	113.8	7-OMe	63.5



### 112. Nordalbergin

*Dalbergia sissoo* [125]

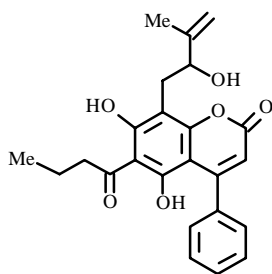
$C_{15}H_{10}O_4$ , **mp** 260-261°C [133], 265°C [63], 268-269°C [40, 125], 274-276°C [134]

**UV** (EtOH) [63]: 262, 356; (EtOH+NaOH): 270, 360

**IR** (mineral oil) [63]: 3450, 3150, 1700, 1680

**Mass** [63]: 254, 226, 197, 181, 169, 152, 141, 127, 115, 102

**PMR** (60 MHz) [63]: 6.1 (1H, s), 6.82 (2H, d,  $J = 2$ ), 7.53 (5H, m), 9.42 (2H, s)



### 113. Racemosol

*Mesua racemosa* [101]

$C_{24}H_{24}O_6$ , **mp** 140.8°C [101]

**UV** (EtOH) [101]: 237 (4.17), 284 (4.38), 339 (3.96)

**IR** (KBr) [101]: 3290, 1734, 1622, 1582

**Mass** [101]: 408 (14)  $[M]^+$ , 390 (13), 375 (15), 364 (21), 349 (16), 347 (17), 339 (11), 338 (41), 337 (100), 321 (24), 319 (24), 309 (14), 295 (22), 105 (13), 43 (25)

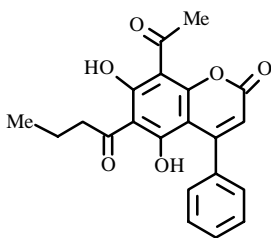
**HREIMS** [101]: 408.1591  $[M]^+$

**PMR** (270 MHz,  $CDCl_3$ ) [101]: 0.98 (3H, t,  $J = 7.5$ ,  $CH_3-4''$ ), 1.70 (2H, m,  $CH_2-3''$ ), 1.93 (3H, s,  $CH_3-5'''$ ), 3.04 (1H, dd,  $J = 7.5$ ,  $J = 15.0$ ,  $CH_2-1b'''$ ), 3.13 (2H, t,  $J = 7.5$ ,  $CH_2-2''$ ), 3.30 (1H, d,  $J = 13.5$ ,  $CH_2-1a'''$ ), 4.50 (1H, d,  $J = 7.5$ , H-2'''), 4.92 (1H, s,  $CH_2-4b'''$ ), 5.03 (1H, s,  $CH_2-4a'''$ ), 5.96 (1H, s, H-3), 7.32 (2H, m, H-2', H-6'), 7.41 (3H, m, H-3', H-4', H-5'), 10.20 (1H, s, OH-7), 14.38 (1H, s, OH-5)

**DQF-COSY, HMQC, HMBC** [101]

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [101]:

C-2	160.0	C-7	162.6	C-4'	128.3	C-4''	13.9
3	112.1	8	104.6	5'	127.7	1'''	28.7
4	156.7	8a	157.7	6'	127.2	2'''	77.2
4a	101.9	1'	139.3	1''	208.0	3'''	145.9
5	163.4	2'	127.2	2''	46.7	4'''	111.1
6	107.6	3'	127.7	3''	18.0	5'''	18.7



#### 114. Racemosone

*Mesua racemosa* [72]

$\text{C}_{21}\text{H}_{18}\text{O}_6$ , mp 149-150°C [72]

UV (EtOH+HCl) [72]: 216 (3.82), 269 (4.12), 325 (3.88); (EtOH+NaOH): 254 (4.00), 291 (4.01), 335 (3.94), 398 (3.76)

IR [72]: 3447, 1753, 1618, 1589, 1153, 769, 700

Mass [72]: 367 (9), 366 (40), 351 (6), 338 (20), 324 (21), 323 (100)

HREIMS [72]: 366.1100 [M]<sup>+</sup>

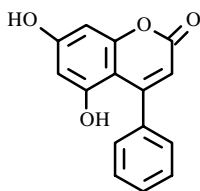
PMR (270 MHz,  $\text{CDCl}_3$ ) [72]: 0.98 (2H, t, J = 7.5,  $\text{CH}_3$ -4''), 1.72 (2H, m,  $\text{CH}_2$ -3''), 2.98 (3H, s,  $\text{CH}_3$ -2'''), 3.15 (2H, t, J = 7.5,  $\text{CH}_2$ -2''), 6.05 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 16.08 (1H, s, OH-5), 16.59 (1H, s, OH-7)

HMBC, DQF-COSY [72]:

C-2	158.1	C-7	172.1	C-3'	127.8	C-2''	46.6
3	112.3	8	102.7	4'	128.5	3''	17.7
4	156.6	8a	161.3	5'	127.8	4''	13.8
4a	101.5	1'	139.0	6'	126.9	1'''	204.0
5	170.4	2'	126.9	1''	208.2	2'''	33.6
6	106.1						

$^{13}\text{C}$  NMR (67.5 MHz,  $\text{CDCl}_3$ ) [72]:

C-2	160.0	C-7	162.6	C-4'	128.3	C-4''	13.9
3	112.1	8	104.6	5'	127.7	1'''	28.7
4	156.7	8a	157.7	6'	127.2	2'''	77.2
4a	101.9	1'	139.3	1''	208.0	3'''	145.9
5	163.4	2'	127.2	2''	46.7	4'''	111.1
6	107.6	3'	127.7	3''	18.0	5'''	18.7



#### 115. Serratatin

*Passiflora serratodigitata* [36]

$\text{C}_{15}\text{H}_{10}\text{O}_4$ , mp 213°C [36]

UV (MeOH) [36]: 208 (5.1), 254, 266 (3.9), 333 (3.91)

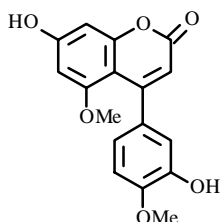
IR (KBr) [36]: 3170, 1660, 1600, 1585, 1545, 1450, 1370, 1155, 1070, 940, 850, 820, 770, 690

Mass [36]: 254 (100) [M]<sup>+</sup>, 226 (95), 197 (47)

**PMR** (90 MHz, DMSO-d<sub>6</sub>) [36]: 5.8 (1H, s, H-3), 6.25 (1H, d, J = 2, H-8), 6.3 (1H, d, J = 2, H-6), 7.4 (5H, s, Ph-4)  
**<sup>13</sup>C NMR** (22.61 MHz, DMSO-d<sub>6</sub>) [36]:

C-2	163.00	C-6	100.40	C-10	158.03	C-4'	128.18
3	96.05	7	158.87	1'	158.00	5'	128.18
4	140.00	8	111.00	2'	128.80	6'	128.80
5	163.88	9	102.40	3'	128.18		

**Biol:** inhibitor of adenosine-3',5'-cyclomonophosphate-phosphodiesterase at IC<sub>50</sub> 7.0×10<sup>-5</sup> M [34]



### 116. Seshadrin

*Dalbergia volubilis* [16, 135], *Exostema mexicanum* [26]

C<sub>17</sub>H<sub>14</sub>O<sub>6</sub>, mp 256-257°C [136]

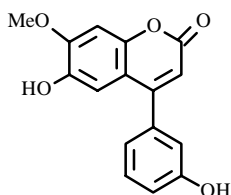
**UV** (MeOH) [16]: 232 (4.2), 256 (4.0), 308 (3.8), 344 (4.1)

**IR** (KBr) [16]: 3340, 3050, 1710, 1615, 1600, 1590, 1450, 1330, 1105, 1080

**Mass** [16]: 314 (3.9), 313 (15.9), 312 (5.1), 300 (4.9), 299 (27.7), 298 (16.1), 293 (5.3), 284 (16.9), 283 (12.0), 282 (5.2), 281 (8.6), 280 (5.0), 271 (6.7), 270 (5.9), 268 (7.9), 267 (8.0), 258 (3.0), 257 (5.9), 256 (10.1), 258 (3.0)

**PMR** (60 MHz, CDCl<sub>3</sub>) [16]: 3.8 (6H, s, OMe), 5.94 (1H, s, H-3), 6.8 (3H, m, H-5, H-6, H-8), 7.3 (1H, d, J = 1.6, H-8), 7.4 (1H, d, J = 1.6, H-6), 9.90 (2H, s, OH)

**Biol:** antiplasmodial activity against *plasmodium falciparum* [26]



### 117. Stevenin

*Dalbergia stevensonii* [54, 137], *D. odorifera* [30, 31], *D. cultrata* [55]

C<sub>16</sub>H<sub>12</sub>O<sub>5</sub>, mp 250-254°C [123], 254°C [54]

**UV** (MeOH) [54]: 222 (4.74), 284 (4.14), 340 (4.05); (MeOH+NaOMe):

252 (4.49), 312 (3.97), 397 (4.08); (MeOH+NaOAc): 301 (4.09), 356 (3.98)

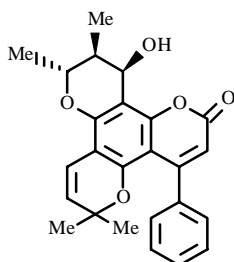
**IR** (CDCl<sub>3</sub>) [54]: 3289, 1707, 1667, 1621

**Mass** [54]: 284 (100) [M]<sup>+</sup>, 256, 241

**PMR** (60 MHz, DMSO-d<sub>6</sub>) [54]: 3.92 (3H, s, OMe), 6.5 (1H, s, H-3), 6.9-7.53 (6H, m), 9.52 (2H, s)

**<sup>13</sup>C NMR** (75.4 MHz, DMSO-d<sub>6</sub>) [30]:

C-2	160.67	C-6	110.61	C-10	111.12	C-4'	116.67
3	111.22	7	152.14	1'	136.81	5'	119.12
4	155.45	8	100.76	2'	115.21	6'	128.80
5	110.61	9	148.62	3'	157.74	7-OMe	56.40



### 118. Soulattrolide [(-)-trans-dihydroinophyllolide]

*Calophyllum soulattri* [138], *C. moonii* [75],

*C. teysmannii* var. *inophyllolide* [79, 139]

C<sub>25</sub>H<sub>24</sub>O<sub>5</sub>, mp 198-200°C [75], 201-202°C [138]

[α]<sub>D</sub> -21.8° (CHCl<sub>3</sub>) [75], -29.6° (CHCl<sub>3</sub>) [138], -30.2° (CHCl<sub>3</sub>) [76]

**UV** (EtOH) [138]: 233 (3.43), 287 (3.37), 335 (3.08)

**IR** (mineral oil) [138]: 3479, 2930, 2860, 1705, 1640, 1590, 1565, 1470, 1415, 1372, 1349, 1310, 1255, 1230, 1200, 1180,

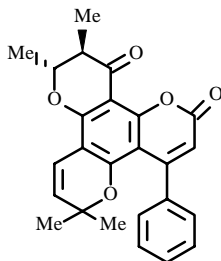
1150, 1140, 1130, 1115, 1062, 1022, 995, 965, 930, 880, 850, 795, 772, 760, 710, 700

**Mass** [138]: 404 (20), 389 (62), 386 (46), 371 (100), 333 (46), 317 (3), 305 (3), 202 (3), 193 (4), 178 (16), 164 (5), 115 (5), 105 (5), 78 (6), 42 (6)

**PMR** (CDCl<sub>3</sub>) [75]: 0.93 (6H, s, CH<sub>3</sub>-10, CH<sub>3</sub>-10), 1.18 (3H, d, J = 6.77, CH<sub>3</sub>-3), 1.45 (3H, d, J = 6.30, CH<sub>3</sub>-2), 1.80 (1H, m, J = 6.77, J = 3.46, J = 10.30, H-3), 2.80 (1H, OH), 4.31 (1H, m, J = 6.29, J = 10.30, H-2), 5.10 (1H, d, J = 3.46, H-4), 5.38 (1H, d, J = 10.0, H-11), 5.96 (1H, s, H-7), 6.55 (1H, d, J = 10.0, H-12), 7.40 (5H, m, Ph-8)

**X-ray** [76]

**Biol:** specific inhibitor of HIV-1 RT at IC<sub>50</sub> 0.34 μM [140]



### 119. Soulattrolone [(-)-*trans*-inophyllolide]

*Calophyllum teysmannii* var. *inophyllolide* [79]

C<sub>25</sub>H<sub>22</sub>O<sub>5</sub>, **mp** 187-190°C [138]

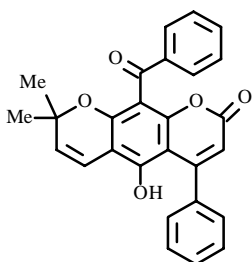
[α]<sub>D</sub> -45.9° (CHCl<sub>3</sub>) [138]

**UV** (EtOH) [138]: 257 (4.46), 266 (4.49), 302 (4.39)

**IR** (mineral oil) [138]: 2930, 2860, 1740, 1690, 1640, 1600, 1580, 1555, 1405, 1380, 1340, 1315, 1240, 1195, 1190, 1145, 1130, 1089, 1000, 955, 920, 880, 860, 835, 775, 735, 705

**Mass** [138]: 402 (33) [M]<sup>+</sup>, 387 (100), 353 (2), 346 (3), 331 (70), 263 (3), 229 (2), 219 (2), 189 (2), 105 (2), 77 (2)

**PMR** (100 MHz, CDCl<sub>3</sub>) [138]: 0.98 (6H, s, CH<sub>3</sub>-6), 1.24 (3H, d, J = 7.2, CH<sub>3</sub>-11), 1.54 (3H, d, J = 6.4, CH<sub>3</sub>-10), 2.58 (1H, m, J = 7.2, J = 10.8, H-11), 4.31 (1H, m, J = 6.4, J = 10.8, H-10), 5.41 (1H, d, J = 10, H-7), 6.03 (1H, s, H-3), 6.54 (1H, d, J = 10, H-8), 7.3 (5H, m, Ph-4)



### 120. Teysmanone A

*Calophyllum teysmannii* var. *inophyllolide* [80]

C<sub>27</sub>H<sub>20</sub>O<sub>5</sub>, **mp** 238-240°C [80]

**UV** (EtOH) [80]: 238 (4.15), 280 (4.13), 334 (3.86)

**IR** (KBr) [80]: 3423, 1700, 1671, 1598, 1447, 1366, 1247, 1193, 1130, 865, 722, 691

**Mass** [80]: 424 (40) [M]<sup>+</sup>, 409 (100), 331 (42), 105 (70), 87 (50), 43 (10)

**HREIMS** [80]: 424.1340 [M]<sup>+</sup>

**PMR** (500 MHz, CDCl<sub>3</sub>) [80]: 1.27 (6H, s, CH<sub>3</sub>-12, CH<sub>3</sub>-13), 5.55 (1H, d, J = 10.2, H-7), 5.93 (1H, s, H-3), 6.52 (1H, d, J = 10.2, H-6), 7.45 (2H, m, H-3'', H-5''), 7.48 (2H, m, H-2', H-6'), 7.58 (1H, dd, J = 7.4, J = 1.2, H-4''), 7.61 (3H, m, H-3', H-4', H-5'), 7.91 (2H, dd, J = 8.4, J = 1.2, H-2'', H-6'')

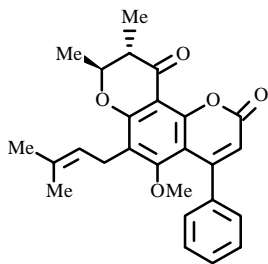
**HMQC, HMBC** [80]

**X-ray** [71]

<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) [80]:

C-2	158.9	C-7	129.1	C-12	28.0	C-6'	127.5
3	113.1	8	78.3	13	28.0	1''	137.6
4*	152.5	8a	157.7	1'	135.9	2''	129.6
4a	101.1	9**	155.0	2'	127.5	3''	128.5
5**	149.9	10	110.4	3'	130.1	4''	133.5
5a	106.3	10a*	152.7	4'	130.6	5''	128.5
6	115.4	11	191.9	5'	130.1	6''	129.6

\*, \*\*Assignments may be reversed



### 121. Teysmanone B

*Calophyllum teysmannii* var. *inophyllolide* [80]

$C_{26}H_{26}O_5$

$[\alpha]_D^{25} +40.5^\circ$  ( $CHCl_3$ ) [80]

**UV** (EtOH) [80]: 226 (3.73), 286 (3.40), 342 (3.22)

**IR** (KBr) [80]: 1718, 1700, 1611, 1577, 1463, 1384, 1114, 696

**PMR** (500 MHz,  $CDCl_3$ ) [80]: 1.24 (3H, d,  $J = 7.1$ ,  $CH_3$ -12), 1.55 (3H, d,  $J = 6.5$ ,  $CH_3$ -11), 1.67 (3H, s,  $CH_3$ -5''), 1.71 (3H, s,  $CH_3$ -5''), 2.59 (1H, dq,  $J = 11.1$ ,  $J = 7.1$ , H-9), 3.00 (3H, s, OMe-5), 3.29 (2H, d,  $J = 6.9$ ,  $CH_2$ -1''), 4.33 (1H, dq,  $J = 11.1$ ,  $J = 6.5$ , H-8), 5.08 (1H, m, H-2''), 6.15 (1H, s, H-3), 7.25-7.43 (5H, m, H-2', H-3', H-4', H-5', H-6')

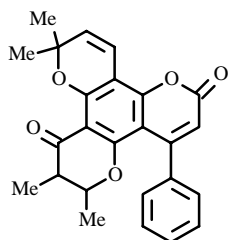
**Mass** [80]: 418 (100)  $[M]^+$ , 403 (70), 387 (10), 363 (25), 349 (60), 335 (35), 319 (80), 307 (40), 105 (42), 55 (45), 43 (70)

**HREIMS** [80]: 418.1806  $[M]^+$

$^{13}C$  NMR (125 MHz,  $CDCl_3$ ) [80]:

C-2*	159.5	C-8	79.5	C-1'	138.4	C-1''	22.6
3	114.7	9	47.2	2'	127.4	2''	121.7
4**	154.4	10	190.7	3'	127.7	3''	132.2
4a***	107.2	10a***	106.4	4'	128.5	4''	25.6
5*	163.2	10b**	153.7	5'	127.7	5''	17.8
6	120.8	11	19.5	6'	127.4	OMe-5	62.0
6a*	160.4	12	10.4				

\*, \*\*, \*\*\*Assignments may be reversed



### 122. Tomentolide A

*Calophyllum tomentosum* [7]

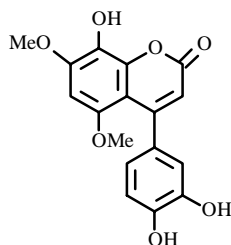
$C_{25}H_{22}O_5$ , mp 201-205°C [7]

**UV** [7]: 237 (4.63), 275-280 (4.59), 350 (3.9)

**IR** [7]: 1740, 1690, 753, 699

**Mass** [7]: 402, 387, 331, 303

**PMR** ( $CDCl_3$ ) [7]: 0.71 (3H, d,  $J = 6$ ), 1.0 (3H, d,  $J = 6$ ), 1.49 (3H, s), 1.52 (3H, s), 2.18 (1H, m), 3.78 (1H, m), 5.58 (1H, d,  $J = 10$ ), 5.85 (1H, s), 6.78 (1H, d,  $J = 10$ )



### 123. 8,3',4'-Trihydroxy-5,7-dimethoxy-4-phenylcoumarin

*Couterea hexandra* [33, 141], *Exostema mexicanum* [26]

$C_{17}H_{14}O_7$ , mp 253-255°C [33]

**UV** (MeOH) [141]: 265 (2.81), 280 (2.98), 327 (3.6), 427 (2.48);

(MeOH+NaOMe): 287, 330, 386

**Mass** [141]: 330 (100)  $[M]^+$ , 302 (13), 287 (21), 259 (7)

**HREIMS** [141]: 330.0824

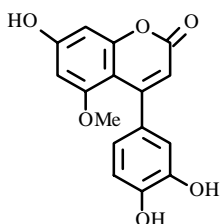
**PMR** (200 MHz,  $CD_3OD$ ) [141]: 3.50 (3H, s, OMe-5), 3.98 (3H, s, OMe-7), 5.90 (1H, s, H-3), 6.57 (1H, s, H-6), 6.66 (1H, dd,  $J = 2$ ,  $J = 7.5$ , H-6'), 6.75 (1H, d,  $J = 2$ , H-2'), 6.81 (1H, d,  $J = 7.5$ , H-5')

<sup>13</sup>C NMR (69.5 MHz, CD<sub>3</sub>OD) [141]:

C-2	162.8	C-6	95.9	C-1'	133.1	C-5'	115.6
3	112.6	7*	152.5	2'	116.2	6'	120.4
4	158.7	8	129.6	3'	145.5	OMe	57.1
4a	105.4	8a	145.2	4'	146.8	OMe	57.2
5*	152.9						

\*Assignments may be reversed

**Biol:** inhibitor of adenosine-3',5'-cyclomonophosphate-phosphodiesterase at IC<sub>50</sub> 3.7×10<sup>-5</sup> M [34], antiplasmodial activity against *Plasmodium falciparum* [26]



#### 124. 7,3',4'-Trihydroxy-5-methoxyneoflavone

*Coutarea hexandra* [142]

C<sub>16</sub>H<sub>12</sub>O<sub>6</sub>, mp 269-270°C [142]

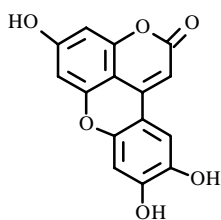
UV (MeOH) [142]: 259 (4.43), 332 (4.77); (MeOH+NaOAc): 257, 272, 332, 378; (MeOH+NaOMe): 245, 271, 381

Mass [142]: 300 (100) [M]<sup>+</sup>, 272 (100), 257 (12), 239 (30), 229 (8), 211 (12), 150 (6), 136 (4)

PMR [(CD<sub>3</sub>)<sub>2</sub>CO] [142]: 3.53 (3H, s, 5-OMe), 5.80 (1H, s, H-3), 6.36 (1H, d, J = 2.5, H-6), 6.43 (1H, d, J = 2.5, H-8), 6.68 (1H, dd, J = 2, J = 8, H-6'), 6.83 (1H, d, J = 2, H-2'), 6.85 (1H, d, J = 8, H-5')

<sup>13</sup>C NMR [(CD<sub>3</sub>)<sub>2</sub>CO] [142]:

C-2	160.7	C-5	159.9	C-8a	158.2	C-4'	146.1
3	112.1	6	97.0	1'	132.7	5'	115.1
4	156.7	7	162.6	2'	115.8	6'	120.1
4a	103.4	8	96.7	3'	145.0	OMe	56.0



#### 125. 7,4',5'-Trihydroxy-4-phenyl-5,2'-oxidocoumarin

*Exostema caribaeum* [19]

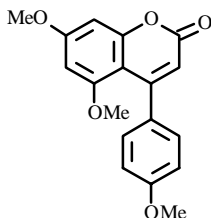
C<sub>15</sub>H<sub>8</sub>O<sub>6</sub>, mp 350°C (dec.) [19]

UV (MeOH) [19]: 262, 310, 350, 372, 390; (MeOH+AlCl<sub>3</sub>): 325, 404; (MeOH+AlCl<sub>3</sub>+HCl): 262, 310, 350, 372, 390

IR (KBr) [19]: 3425, 3250, 1690, 1630, 1560, 1460, 1298, 1170, 830

Mass [19]: 284 (100), 256 (35), 241 (10), 228 (50)

PMR (80 MHz, CDCl<sub>3</sub>/DMSO-d<sub>6</sub>) [19]: 5.91 (1H, s, H-3), 6.44 (2H, s, H-6, H-8), 6.73 (1H, s, H-3'), 7.21 (1H, s, H-6'), 10.30 (3H, s, OH)



### 126. 5,7,4'-Trimethoxy-4-phenylcoumarin

*Coutarea hexandra* [24], *Chiococca alba* [143], *Exostema caribaeum* [11],  
*E. acuminatum* [22], *Indigofera oblongifolia* [144]

$C_{18}H_{16}O_5$ , **mp** 150-152°C [22], 151-152°C [24], 152°C [143]

**UV** ( $CHCl_3$ ) [143]: 260 (4.06), 315 (4.25)

**IR** (KBr) [143]: 1710, 1625, 1590, 1510, 1160, 1110, 1060, 950, 872, 860, 840

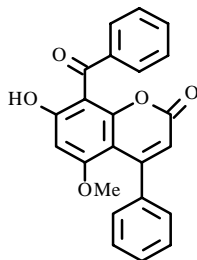
**Mass** [143]: 312 (100)  $[M]^+$ , 284 (51), 269 (48), 241 (2)

**PMR** (200 MHz,  $CDCl_3$ ) [143]: 3.48 (3H, s, OMe-5), 3.87 (6H, s, OMe-7, OMe-4'), 5.90 (1H, s, H-3), 6.24 (1H, d,  $J = 2.5$ , H-6), 6.53 (1H, d,  $J = 2.5$ , H-8), 6.93 (2H, d,  $J = 8.5$ , H-3', H-5'), 7.25 (2H, d,  $J = 8.5$ , H-2', H-6')

$^{13}C$  NMR (125.7 MHz,  $CDCl_3$ ) [22]:

C-2	161.2	C-6	96.0	C-2'	129.1	C-6'	129.1
3	112.8	7	163.6	3'	113.1	OMe-7	56.1
4	157.6	8	94.0	4'	160.0	OMe-7	55.8
4a	103.9	8a	158.6	5'	113.1	OMe-4'	55.7
5	158.6	1'	132.4				

**Biol:** cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V<sup>+</sup>, KB-V<sup>-</sup>, LNCaP, SW626, SKNSH, and M109 [22]



### 127. 4-Phenyl-5-methoxy-7-hydroxy-8-benzoylcoumarin

*Calophyllum teysmannii* var. *inophylloide* [20]

$C_{23}H_{16}O_5$ , **mp** 220-222°C [20]

**UV** [20]: 252, 290, 328

**IR** [20]: 3427, 3082, 2927, 1723, 1627, 1595, 1575, 1360, 1263, 1106, 861, 777, 739, 701

**Mass** [20]: 372 (62)  $[M]^+$ , 371 (100), 343 (40), 149 (6), 105 (54), 77 (57)

**HR-EIMS** [20]: 372.1002

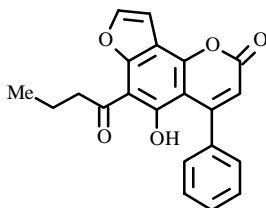
**PMR** (500 MHz,  $CDCl_3$ ) [20]: 3.50 (3H, s, MeO-5), 5.93 (1H, s, H-3), 6.33 (1H, s, H-6), 7.24 (2H, m, H-2', H-6'), 7.38 (3H, m, H-3', H-4', H-5'), 7.48 (2H, t,  $J = 8.0$ , H-3'', H-5''), 7.60 (1H, tt,  $J = 8.0$ ,  $J = 1.6$ , H-4''), 7.65 (2H, dd,  $J = 8.0$ ,  $J = 1.6$ , H-2'', H-6''), 12.30 (s, 1H, OH-7)

**HMBC, NOE** [20]

$^{13}C$  NMR (125 MHz,  $CDCl_3$ ) [20]:

C-2*	158.1	C-7***	167.0	C-3'	127.4	C-3''	128.1
3	112.8	8**	104.3	4'	127.9	4''	132.3
4*	155.5	8a*	156.8	5'	127.4	5''	128.1
4a**	102.6	9	198.5	6'	126.8	6''	128.1
5***	162.4	1'	139.5	1''	140.1	OMe	55.6
6	96.1	2'	126.8	2''	128.1		

\*, \*\*, \*\*\* Assignments may be reversed



### 128. Furanoracemosone

*Mesua racemosa* [72]

$C_{21}H_{16}O_5$

**UV** (EtOH+HCl) [72]: 289 (3.66), 238 (3.54); (EtOH+NaOH): 279 (3.64), 377 (3.17)

**IR** [72]: 3447, 2958, 1742, 1612, 1161, 1136, 762, 698

**Mass** [72]: 349 (13), 348 (53), 333 (6), 306 (12), 305 (100), 277 (10), 221 (7), 71 (6), 69 (10), 57 (10), 55 (6), 43 (9), 28 (12), 18 (8)

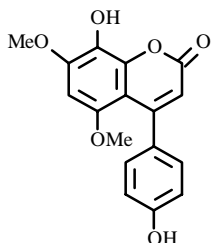
**HREIMS** [72]: 348.0973 [M]<sup>+</sup>

**PMR** (270 MHz,  $CDCl_3$ ) [72]: 1.05 (3H, t, J = 7.0,  $CH_3-4''$ ), 1.79 (2H, m,  $CH_2-3''$ ), 3.25 (2H, t, J = 7.0,  $CH_2-2''$ ), 6.17 (1H, s, H-3), 7.16 (1H, d, J = 2.0, H-9), 7.36 (2H, m, H-2', H-6'), 7.43 (3H, m, H-3', H-4', H-5'), 7.68 (1H, d, J = 2.0, H-8), 14.53 (1H, s, OH-5)

**HMBC** [72]

<sup>13</sup>C NMR (67.5 MHz,  $CDCl_3$ ) [72]:

C-2	159.3	C-6a	156.0	C-1'	138.9	C-6'	127.2
3	114.3	8	143.9	2'	127.2	1''	204.5
4	156.8	9	104.7	3'	127.7	2''	44.9
4a	104.7	9a	109.7	4'	128.4	3''	17.5
5	162.7	9b	153.3	5'	127.7	4''	13.8
6	103.7						



### 129. Exomexin A

*Exostema mexiacnum* [26]

$C_{17}H_{14}O_6$

**Mass** [26]: 314 [M]<sup>+</sup>, 299, 286, 271, 267, 243, 228

**HRMS** [26]: 314.07916

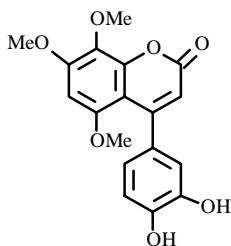
**PMR** (400 MHz,  $CD_3COCD_3$ ) [26]: 3.50 (3H, s, OMe), 3.97 (3H, s, OMe), 5.84 (1H, s, H-3), 6.64 (1H, s, H-6), 6.87 (2H, d, J = 8.6, H-3', H-5'), 7.20 (2H, d, J = 8.6, H-2', H-6'), 7.74 (1H, s, OH), 8.51 (1H, s, OH)

<sup>13</sup>C NMR (100 MHz,  $CD_3OD$ ) [26]:

C-2	163.0	C-6	95.0	C-1'	132.4	C-5'	115.3
3	112.5	7	152.9	2'	130.0	6'	130.0
4	145.1	8	127.4	3'	115.3	OMe-7	57.0
4a	104.9	8a	158.8	4'	159.0	OMe-5	56.7
5	152.4						

**Biol:** antiplasmodial activity against *Plasmodium falciparum* [26]





### 130. Exomexin B

*Exostema mexicanum* [26]

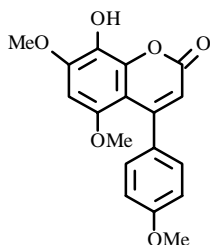
$C_{18}H_{16}O_6$

**Mass** [26]: 344 [M]<sup>+</sup>, 329, 316, 301, 273

**HRMS** [26]: 344.08948

**PMR** (400 MHz, CD<sub>3</sub>COCD<sub>3</sub>) [26]: 3.57 (3H, s, OMe), 3.83 (3H, s, OMe), 3.99 (3H, s, OMe), 5.86 (1H, s, H-3), 6.63 (1H, s, H-6), 6.68 (1H, dd, J = 8.1, J = 2.1, H-6'), 6.83 (1H, d, J = 2.1, H-2'), 6.85 (1H, d, J = 8.1, H-5'), 8.09 (1H, s, OH), 8.11 (1H, s, OH)

**Biol**: antiplasmodial activity against *Plasmodium falciparum* [26]



### 131. Exostemin

*Exostema caribaeum* [145, 146], *E. acuminatum* [22], *Coutarea hexandra* [25], *Chiococca alba* [25]

$C_{18}H_{16}O_6$ , **mp** 158-160°C [22], 173-174°C [145, 146], 195-196°C [147]

**UV** (MeOH) [143]: 260 (4.06), 315 (4.26)

**IR** (KBr) [143]: 3450, 1710, 1590, 1515, 1170, 1110, 1070, 950, 875, 865, 845

**Mass** [143]: 328 (100), 312 (40), 299 (24), 284 (38), 269 (6), 257 (10), 201 (7)

**PMR** (200 MHz, CDCl<sub>3</sub>) [143]: 3.46 (3H, s, OMe-5), 3.87 (3H, s, OMe-4'), 3.98 (3H, s, OMe-7), 5.75 (1H, s, OH-8), 5.80 (1H, s, H-3), 6.34 (1H, s, H-6), 6.92 (2H, d, J = 8.5, H-3', H-5'), 7.24 (2H, d, J = 8.5, H-2', H-6')

**<sup>13</sup>C NMR** (125.7 MHz, DMSO-d<sub>6</sub>) [22]:

C-2	160.1	C-6	93.6	C-2'	129.2	C-6'	129.2
3	113.0	7	151.3	3'	113.2	OMe	56.9
4	156.2	8	151.3	4'	160.4	OMe	56.7
4a	104.9	8a	143.4	5'	113.2	OMe	55.7
5	150.3	1'	127.9				

**Biol**: inhibitor of adenosine-3',5'-cyclomonophosphate-phosphodiesterase at IC<sub>50</sub> 10.4×10<sup>-5</sup> M [34], cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V<sup>+</sup>, KB-V<sup>-</sup>, LNCaP, SW626, SKNSH, and M109 [22]

It should especially be noted that inophyllum A (**51**), B (**52**), C (**53**), D (**54**), E (**55**), and calophyllolide (**73**), which were previously isolated from *Calophyllum inophyllum* [74], were isolated from the acetone extract of the giant African snail *Achatina fulica*, whose principal food source are leaves of *Calophyllum* and which was collected in the Seychelles islands. This is the single described instance of neoflavones isolated from a non-plant source.

Table 1 lists plants that produce neoflavones and 4-phenylcoumarins.

Definite structural trends are observed among natural 4-phenylcoumarins. Neoflavones isolated from plants of the Clusiaceae and Thelypteridaceae families have the phloroglucinol orientation of the hydroxyls in benzene ring A and unsubstituted benzene ring B. Neoflavones produced by plants of the Rubiaceae family contain a mono- or dioxygenated 4-phenyl substituent. Both unsubstituted and substituted benzene rings B are characteristic of neoflavones found in plants of the Fabaceae family. All coumarins obtained from plants of the Thelypteridaceae family contain a methyl substituent in the 6-position of the coumarin system.

TABLE 1. Plant Sources of Neoflavones

Plant	Isolated neoflavones
<u>Clusiaceae family</u>	
<i>Calophyllum apetalum</i>	Apetatolide ( <b>1</b> ) [6, 7]
<i>C. australianum</i>	Calaustralin ( <b>68</b> ) [84]
<i>C. bracteatum</i>	Calophyllolide ( <b>73</b> ) [90]
<i>C. dispar</i>	Disparacetylfuran A ( <b>38</b> ) [66], dispartiol B ( <b>39</b> ) [67], disparinol A ( <b>40</b> ) [68], disparinol B ( <b>41</b> ) [68], disparinol D ( <b>42</b> ) [67], disparturan B ( <b>43</b> ) [66], isodispar B ( <b>45</b> ) [67], isodisparinol A ( <b>46</b> ) [68], isodisparinol B ( <b>47</b> ) [68], isodisparturan A ( <b>48</b> ) [66], mammea A/AA dehydrocyclo F ( <b>78</b> ) [66], mammea A/AA methoxycyclo F ( <b>79</b> ) [66], mammea A/AA cyclo F ( <b>81</b> ) [66], mammea A/AB dioxalanocyclo F ( <b>83</b> ) [67], mammea A/AB cyclo E ( <b>85</b> ) [67], mammea A/AB cyclo F ( <b>86</b> ) [66], mammea A/AC cyclo F ( <b>88</b> ) [66], mammea A/BA cyclo F ( <b>95</b> ) [66], mammea A/BB cyclo F ( <b>98</b> ) [66], mammea A/BC cyclo F ( <b>100</b> ) [66]
<i>C. inophyllum</i>	Acetatolide ( <b>1</b> ) [8], inophyllum A ( <b>51</b> ) [73, 74], inophyllum B ( <b>52</b> ) [73, 74, 77], inophyllum C ( <b>53</b> ) [73, 74, 77, 78], inophyllum D ( <b>54</b> ) [73, 74], inophyllum E ( <b>55</b> ) [73, 74, 77], inophyllum G-1 ( <b>56</b> ) [74], inophyllum G-2 ( <b>57</b> ) [74], inophyllum P ( <b>58</b> ) [58], (-)-calaustralin ( <b>69</b> ) [85], calocoumarin A ( <b>70</b> ) [86], calocoumarin B ( <b>71</b> ) [86], calocoumarin C ( <b>72</b> ) [86], calophyllolide ( <b>73</b> ) [1, 77, 87, 88, 89], mammea A/BB cyclo D ( <b>97</b> ) [87]
<i>C. moonii</i>	Inophyllum A ( <b>51</b> ) [75], soulatrolide ( <b>118</b> ) [75]
<i>C. aff. Pervillei</i>	Calophyllolide ( <b>73</b> ) [91]
<i>C. soulattri</i>	Soulatrolide ( <b>118</b> ) [138]
<i>C. teysmannii</i> var <i>inophylloide</i>	9-Benzoyl-6-hydroxy-5-methoxy-4-phenylnodakenetin ( <b>5</b> ) [14], (-)-6-benzoyl-5-hydroxy-4-phenylcolumbianelin ( <b>6</b> ) [14], 9-benzoyl-5-methoxy-4-phenylnodakenetin ( <b>7</b> ) [14], hydrohydroxyisocalanone ( <b>14</b> ) [20], <i>trans</i> -7,8-dihydroxycalanone ( <b>32</b> ) [14], isocalanone ( <b>49</b> ) [14, 70], inophyllum C ( <b>53</b> ) [14, 79, 80], inophyllum E ( <b>55</b> ) [80], calanone ( <b>67</b> ) [14, 20, 79, 80], O-methylisocalaustralin ( <b>105</b> ) [14], O-methylcalaustralin ( <b>106</b> ) [14], 12-methoxyinophyllum P ( <b>109</b> ) [20], soulatrolide ( <b>118</b> ) [79, 139], soulatrolone ( <b>119</b> ) [79], teysmanone A ( <b>120</b> ) [80], teysmanone B ( <b>121</b> ) [80], 4-phenyl-5-methoxy-7-hydroxy-8-benzoylcoumarin ( <b>127</b> ) [20]
<i>C. tomentosum</i>	Tomentolide A ( <b>122</b> ) [7]
<i>Kayea assamica</i>	Mammea A/AA ( <b>77</b> ) [103], mammea A/AA cyclo F ( <b>81</b> ) [103]
<i>Kielmeyera argentea</i>	Coumarins <b>23</b> [35] and <b>24</b> [35]
<i>K. elata</i>	Hydroxymammeigin ( <b>18</b> ) [29], mammea A/AA ( <b>77</b> ) [29], mammea A/AA cyclo D ( <b>80</b> ) [29], mammea A/AA cyclo F ( <b>81</b> ) [29]
<i>K. lathrophyton</i>	Mammea A/AA cyclo D ( <b>80</b> ) [111], mammea A/AB cyclo D ( <b>84</b> ) [111]
<i>K. pumila</i>	Mammea A/AA cyclo D ( <b>80</b> ) [110], mammea A/AB cyclo F ( <b>86</b> ) [110], mammea A/BA cyclo D ( <b>94</b> ) [110]
<i>K. reticulata</i>	Coumarins <b>15</b> [21], <b>23</b> [21], <b>24</b> [21], and <b>29</b> [21]
<i>Mammea africana</i>	Mammea A/AA ( <b>77</b> ) [98, 99], mammea A/AB ( <b>82</b> ) [98, 99], mammea A/AB cyclo D ( <b>84</b> ) [98, 99], mammea A/AB cyclo F ( <b>86</b> ) [98, 99], mammea A/AD ( <b>90</b> ) [6, 104], mammea A/BA ( <b>93</b> ) [116]
<i>M. americana</i>	Mammea A/AA ( <b>77</b> ) [96, 97], mammea A/AA cyclo D ( <b>80</b> ) [107, 108], mammea A/AA cyclo F ( <b>81</b> ) [113], mammea A/AB ( <b>82</b> ) [115, 116], mammea A/AB cyclo F ( <b>86</b> ) [113, 114], mammea A/AC cyclo F ( <b>88</b> ) [114], mammea A/AD ( <b>90</b> ) [119], mammea A/AD cyclo D ( <b>91</b> ) [6], mammea A/AD cyclo F ( <b>92</b> ) [114], mammea A/BA ( <b>93</b> ) [115, 116], mammea A/BB ( <b>96</b> ) [115, 116], mammea A/BB cyclo D ( <b>97</b> ) [105]
<i>M. siamensis</i>	Mammea A/AC cyclo D ( <b>89</b> ) [118]
<i>Mesua ferrea</i>	Coumarins <b>20</b> and <b>35</b> [32], mammea A/AA ( <b>77</b> ) [100], mammea A/AA cyclo D ( <b>80</b> ) [104, 109], mammea A/AD ( <b>90</b> ) [104], mammea A/AD cyclo D ( <b>91</b> ) [109], mesuarin ( <b>101</b> ) [120]
<i>M. racemosa</i>	Isoracemosol ( <b>50</b> ) [72], mammea A/AA ( <b>77</b> ) [101], mammea A/AC ( <b>87</b> ) [101], mammea A/AC cyclo F ( <b>88</b> ) [101], mammea A/AC cyclo D ( <b>89</b> ) [101], mammea A/AD cyclo D ( <b>91</b> ) [101], mammea A/BB ( <b>96</b> ) [101], mammea A/BC ( <b>99</b> ) [72], racemosol ( <b>113</b> ) [101], racemosone ( <b>114</b> ) [72], furanoracemosol ( <b>128</b> ) [72]
<i>M. thwaitesii</i>	Mammea A/AA ( <b>77</b> ) [102], mammea A/AA cyclo D ( <b>80</b> ) [102], mammea A/AA cyclo F ( <b>81</b> ) [102], mammea A/AD ( <b>90</b> ) [102], mammea A/AD cyclo D ( <b>91</b> ) [102]
<i>Ochrocarpus siamensis</i>	Mammea A/AC ( <b>87</b> ) [117], mammea A/AC cyclo D ( <b>89</b> ) [117]

TABLE 1. (continued)

Plant	Isolated neoflavones
<u>Rubiaceae family</u>	
<i>Chiococca alba</i>	Coumarin <b>126</b> [143], exostemin ( <b>131</b> ) [143]
<i>Coutarea hexandra</i>	Coutareagenin ( <b>74</b> ) [92, 93], coumarins <b>2</b> [10], <b>17</b> [24, 25], <b>21</b> [33], <b>22</b> [24], <b>26</b> [10], <b>27</b> [10], <b>31</b> [24, 64], <b>33</b> [33], <b>37</b> [65], <b>75</b> [10], <b>123</b> [33, 141], <b>124</b> [142], and <b>126</b> [24], 8-O-methylexostemin ( <b>107</b> ) [25], exostemin ( <b>131</b> ) [25]
<i>Exostema acuminatum</i>	Coumarins <b>16</b> [22], <b>22</b> [22], <b>30</b> [22], <b>33</b> [22], and <b>126</b> [22], exostemin ( <b>131</b> ) [22]
<i>E. caribaeum</i>	Coumarins <b>3</b> [11], <b>11</b> [19], <b>13</b> [11], <b>26</b> [11], <b>30</b> [11], and <b>126</b> [11], oxidocoumarins <b>36</b> [19], <b>108</b> [11], and <b>125</b> [19], exostemin ( <b>131</b> ) [145, 146]
<i>E. mexicanum</i>	Coumarins <b>17</b> [26], <b>22</b> [26], <b>26</b> [37], <b>31</b> [26], and <b>123</b> [26], 8-O-methylexostemin ( <b>107</b> ) [26], seshadrin ( <b>116</b> ) [26], exomexin A ( <b>129</b> ) [26], exomexin B ( <b>130</b> ) [26]
<i>Hintonia latiflora</i>	Coumarins <b>3</b> [12], <b>4</b> [13], <b>11</b> [12], <b>12</b> [12], <b>26</b> [12], and <b>27</b> [12], oxidocoumarin <b>108</b> [128]
<u>Leguminosae (Fabaceae) family</u>	
<i>Cassia alata</i>	Dalbergin ( <b>28</b> ) [62]
<i>Dalbergia baroni</i>	Dalbergin ( <b>28</b> ) [52, 53], melanein ( <b>102</b> ) [52, 53]
<i>D. cearensis</i>	Dalbergin ( <b>28</b> ) [47]
<i>D. coromandeliana</i>	Dalbergin ( <b>28</b> ) [51]
<i>D. cultrata</i>	Dalbergin ( <b>28</b> ) [55], stevenin ( <b>117</b> ) [55]
<i>D. latifolia</i>	Dalbergin ( <b>28</b> ) [42], O-methyldalbergin ( <b>104</b> ) [42]
<i>D. melanoxylon</i>	Dalbergin ( <b>28</b> ) [43], melanein ( <b>102</b> ) [52, 53], melanetin ( <b>103</b> ) [122]
<i>D. miscolobium</i>	Dalbergin ( <b>28</b> ) [45]
<i>D. nigra</i>	Dalbergin ( <b>28</b> ) [44], O-methyldalbergin ( <b>104</b> ) [44]
<i>D. nitidula</i>	Dalbergin ( <b>28</b> ) [48]
<i>D. odorifera</i>	3'-Hydroxymelanetin ( <b>19</b> ) [30, 31], dalbergin ( <b>28</b> ) [30, 31], melanetin ( <b>103</b> ) [30, 31], stevenin ( <b>117</b> ) [30, 31]
<i>D. riparia</i>	Dalbergin ( <b>28</b> ) [49]
<i>D. sissoides</i>	Dalbergin ( <b>28</b> ) [46]
<i>D. sissoo</i>	Dalbergin ( <b>28</b> ) [40, 41], isodalbergin ( <b>44</b> ) [69], O-methyldalbergin ( <b>104</b> ) [125], nordalbergin ( <b>112</b> ) [125]
<i>D. spinosa</i>	Dalbergin ( <b>28</b> ) [50]
<i>D. stevensonii</i>	Dalbergin ( <b>28</b> ) [54], stevenin ( <b>117</b> ) [54, 137]
<i>D. volubilis</i>	Volubolin ( <b>8</b> ) [15, 16], voludal ( <b>9</b> ) [16, 17], dalbergin ( <b>28</b> ) [15, 16], seshadrin ( <b>116</b> ) [16, 135]
<i>Glycyrrhiza inflata</i>	Inflacoumarin A ( <b>66</b> ) [82, 83]
<i>Goniorrhachis marginata</i>	Dalbergin ( <b>28</b> ) [61], O-methyldalbergin ( <b>104</b> ) [61]
<i>Indigofera oblongifolia</i>	Coumarin <b>126</b> [144]
<i>Machaerium kuhlmannii</i>	Dalbergin ( <b>28</b> ) [56], kuhlmannin ( <b>76</b> ) [56, 95]
<i>M. nictitans</i>	Dalbergin ( <b>28</b> ) [56], kuhlmannin ( <b>76</b> ) [56, 95]
<i>M. pedicellatum</i>	Dalbergin ( <b>28</b> ) [58], kuhlmannin ( <b>76</b> ) [58], O-methyldalbergin ( <b>104</b> ) [58]
<i>M. scleroxylon</i>	Dalbergin ( <b>28</b> ) [57], O-methyldalbergin ( <b>104</b> ) [57]
<i>Piptadenia macrocarpa</i>	Dalbergin ( <b>28</b> ) [59], kuhlmannin ( <b>76</b> ) [59]
<i>Prosopis kuntzei</i>	Dalbergin ( <b>28</b> ) [60], O-methyldalbergin ( <b>104</b> ) [60]
<u>Thelypteridaceae family</u>	
<i>Cyclosorus interruptus</i>	Interruptins A ( <b>59</b> ) [81], B ( <b>60</b> ) [81], C ( <b>61</b> ) [81], D ( <b>62</b> ) [81], E ( <b>63</b> ) [81], F ( <b>64</b> ) [81], and G ( <b>65</b> ) [81]
<u>Passifloraceae family</u>	
<i>Passiflora serratodigitata</i>	Serratin ( <b>115</b> ) [36], serratin 7- $\beta$ -glucoside ( <b>25</b> ) [36]
<u>Asteraceae family</u>	
<i>Echinops niveus</i>	Nivegin ( <b>110</b> ) [129, 130], nivetin ( <b>111</b> ) [130]
<u>Rutaceae family</u>	
<i>Hesperethusa crenulata</i>	Coumarin <b>10</b> [18]

There are several classifications for neoflavones isolated from plants of the Clusiaceae family [105, 148]. Considering the various structural features of the side chains and the structures of the fused rings in addition to their positions in the molecules, neoflavones can be divided into the following groups:

1. Hydroxy- and methoxycoumarins:
  - a) with hydroxyls or methoxyls (compounds **8, 16, 17, 19, 21, 22, 28, 30, 31, 33, 37, 44, 74, 76, 102-104, 107, 110-112, 115-117, 123, 124, 126, 129-131**);
  - b) alkylated hydroxycoumarin (**66**);
  - c) 6-formylcoumarin (**9**);
  - d) acylated hydroxyneoflavones (**45, 59-61, 114, 127**).
2. O-Glycosides with the carbohydrate in the coumarin 5- (**2-4, 11-13, 26, 27, 75**), 6- (**10**), and 7-positions (**25**).
3. Neoflavones containing both alkyl and acyl substituents in benzene ring A:
  - a) 6-acyl-8-alkylcoumarins (**29, 34, 39, 40-42, 77, 82, 87, 90, 113**);
  - b) 8-acyl-6-alkylcoumarins (**35, 46, 47, 50, 93, 96, 99**).
4. Furocoumarins (**38, 43, 48, 128**).
5. Dihydrofurocoumarins containing a dihydrofuran fused at the C-5—C-6 (**14, 95, 98, 100**), C-6—C-7 (**5, 7**), and C-7—C-8 positions (**6, 62, 63, 64, 78, 79, 81, 83, 86, 88, 92**) of a 4-phenylcoumarin and substituents in the dihydrofuran and benzene rings.
6. Pyranocoumarins (chromeno- $\alpha$ -pyrones) containing 2,2-dimethylchromene condensed with 4-phenylcoumarin at the C-5—C-6 (**15, 20, 24, 67, 73, 94, 97**), C-6—C-7 (**1, 120**), and C-7—C-8 positions (**18, 23, 49, 80, 84, 89, 91, 101, 22-29**) and acyl substituents in the neoflavone benzene ring A and pyran (**32, 85**).
7. Chromanone-coumarins containing a fused 2,3-dimethylchromanone at the C-6—C-7 (**68, 69, 105, 106**) and C-7—C-8 positions (**70, 121**) of the neoflavone skeleton.
8. Tetracyclic 4-phenylcoumarins:
  - a) containing a 2,3-dimethylchromanone ring in the C-7—C-8 position and 2,2-dimethylchromene moiety at the C-5—C-6 position (**53, 55, 119**);
  - b) containing a 2,3-dimethylchromanone ring at the C-5—C-6 position and a 2,2-dimethylchromene moiety at the C-7—C-8 position (**122**);
  - c) containing a 2,3-dimethylchromanol ring at the C-7—C-8 position and a 2,2-dimethylchromene moiety at the C-5—C-6 position (**51, 52, 54, 58, 109, 118**);
  - d) containing a 2,3-dimethylchromanol ring at the C-7—C-8 position and dimethylcyclopropanedihydrofuran and analogous fragments at the C-5—C-6 position (**56, 57, 71, 72**).
9. 4-Phenyl-5,2'-oxidocoumarins (**36, 108, 125**).
10. 7,10-Dioxocoumarin (**65**).

Neoflavones often contain one or several free hydroxyls that are most frequently found in benzene ring A and much more rarely in ring B. O-Alkyl derivatives are represented exclusively as methoxyls. The C-alkyl substituents include primarily the prenyl group (**29, 34, 66, 68-70, 77, 82, 87, 90, 93, 96, 99, 105, 106, 121**), more rarely 2-hydroxy-3-methylbut-3-enyl (**40-42, 46, 47, 50, 113**), and in unique instances 2,3-dihydroxy-3-methylbutyl (**39**) and geranyl (**35**) substituents. The glycoside fragment is most often  $\beta$ -D-galactopyranose (**10-13**) and  $\beta$ -D-glucopyranose (**25-27**). In single instances 6-acetyl- $\beta$ -D-glucopyranose (**4**) and 6-acetyl- $\beta$ -D-galactopyranose (**3**) in addition to disaccharides D-apiofuranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranose (**2**) and  $\beta$ -D-xylopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranose (**75**) are observed. Among the C-acyl substituents are 3-methylbutane (**38, 40, 45, 46, 48, 77-81, 93-95**), 2-methylbutane (**20, 35, 39, 41, 43, 47, 82-86, 96-98**), butane (**50, 87, 88, 89, 99, 100, 113, 114, 128**), 2-methylpropionic (**42, 90-92, 101**), acetic (**38, 114**), benzoic (**5-7, 14, 32, 49, 67, 120, 127**), and 2-methylbut-2-enoic (**1, 71, 73**) acids in addition to 4-hydroxy-3-methyl-1-oxobutanoic acid (**15, 18, 29**) and its 4-cinnamoyl derivatives (**23, 24, 34**). 3-Phenylpropionic (**59**), 3-phenyl-*trans*-acrylic (**60**), and 2-hydroxy-3-phenylpropionic (**61**) acids are found anecdotally. The neoflavone voludal (**9**) has an aldehyde in the 6-position of the coumarin.

## PHYSICAL RESEARCH METHODS

The application of modern physical methods to research on the structure determination of neoflavones reduced significantly the amount of study by chemical methods that was necessary previously, accelerated the elucidation of structures, and also made it possible to work with microquantities of compounds.

### INFRARED SPECTROSCOPY

The IR spectra of neoflavones, like for all compounds based on the coumarin skeleton, have a band very characteristic of C=O stretching at 1750-1690  $\text{cm}^{-1}$ . The IR spectra also exhibit absorption bands in the ranges 1520-1570 and 1600-1630  $\text{cm}^{-1}$  due to vibrations of the  $\alpha$ -pyrone ring in addition to bands at 690-700 and 740-750  $\text{cm}^{-1}$  due to vibrations of the monosubstituted phenyl ring.

### ULTRAVIOLET SPECTROSCOPY

UV spectroscopy is used to establish the structure of 4-phenylcoumarins mainly for determining the orientation of the acyl substituents. All 6-acyl coumarin derivatives have a characteristic absorption maximum at 614 nm in the Gibbs test whereas the 8-acylisomers do not absorb in this region [116]. The UV spectra of 6-acylcoumarins typically have an absorption maximum at 280 nm whereas 8-acylcoumarins exhibit an absorption maximum at 290 nm [105, 115, 116]. It should be noted that the long-wavelength absorption band of 6-acylcoumarins undergoes a large bathochromic shift upon the addition of base (290 to 330 nm) whereas a small bathochromic shift of the long-wavelength band is observed (280 to 300 nm) for 8-acylcoumarins [105, 115, 116].

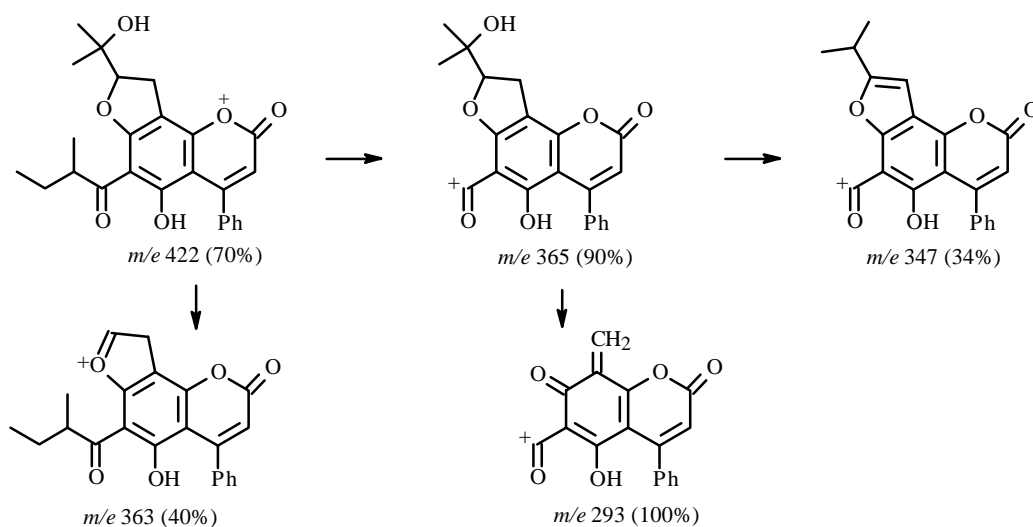
### MASS SPECTROSCOPY

Most reports contain data for the mass numbers and relative intensities of the molecular ions or the base and principal ions.

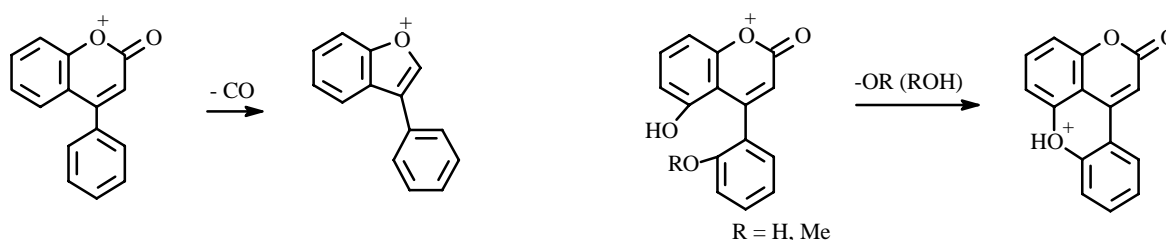
Mass spectra of neoflavones isolated from *Mammea americana*, *M. africana*, and *Calophyllum inophyllum* have been studied in the most detail [8, 98, 99, 116]. Simultaneous application of gas chromatography and mass spectroscopy to identify natural 4-phenylcoumarins and the overall fragmentation pathways of the molecular ions has been described [8]. Mass spectra of mammea A/AA cyclo D (**80**) and mammea A/AB cyclo D (**84**) have base peaks formed by loss of a methyl from the molecular ion, which indicates that the molecule contains a 2,2-dimethylchromene. Fragmentation of the molecular ion by loss of 2-methylbutyryl or 3-methylbutyryl forms the [M - 57] ion.

Mammea A/AA (**77**) and mammea A/AB (**82**) have similar mass spectra [116]. The spectra of these compounds contain [M - 57] peaks due to cleavage of the butyl group of the acyl substituent and [M - 57 - 56] indicative of further elimination of butene from the 3-methylbut-3-enyl substituent. The situation is analogous for the mass spectrum of mammea A/AD (**90**). Loss of a propyl radical from the acyl substituent gives the [M - 43] peak. Further cleavage of butene from the 3-methylbut-2-ene substituent gives the [M - 43 - 56] peak. Mass spectra of these 4-phenylcoumarins contain common characteristic ions at  $m/e$  169 and 171 [116].

Common decomposition pathways are observed in the mass spectra of mammea A/AB (**82**), mammea A/AB cyclo D (**84**), and mammea A/AB cyclo F (**86**) [98]. The spectra of these compounds contain [M - 57] peaks due to elimination of a butyl radical of the acyl moiety on C-6. The spectrum of compound **82** contains additional peaks for [M - 57 - 55] and [M - 55] due to cleavage of butene or butenyl radicals during fragmentation of the 3,3-dimethylallyl substituent. The mass spectrum of compound **84** has the base peak [M - 15] that is typical of the exceedingly stable benzopyryl ion. The fragmentation pattern of mammea A/AB cyclo F (**86**) has been reported [99]. The [M - 75] ion is formed by loss of water from the [M - 57] ion; the [M - 129] ion, by elimination of isobutenoxide from the [M - 57] ion (Scheme 1).



Scheme 1



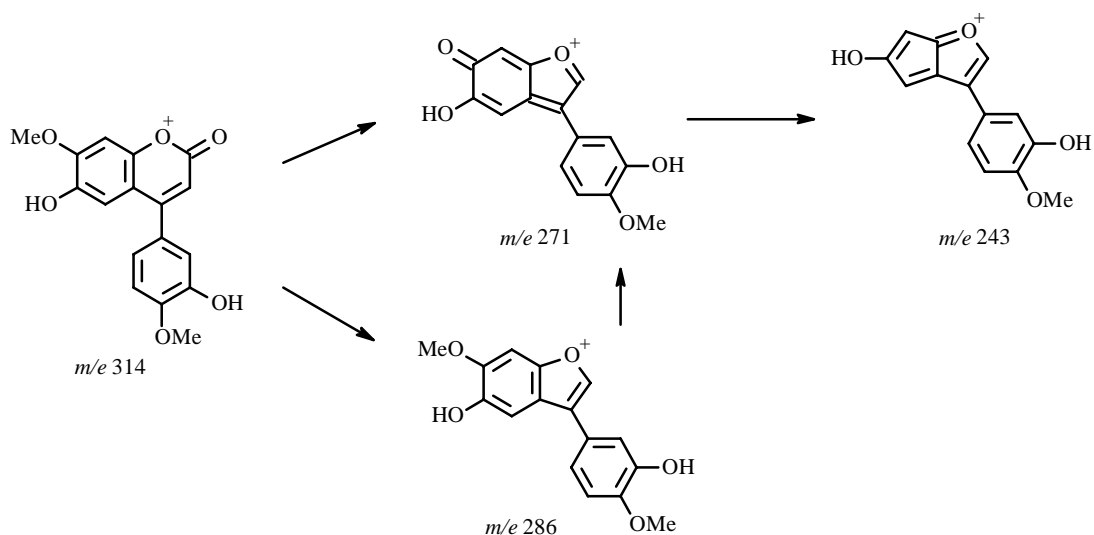
Scheme 2

The base peak  $[M]^+$  ( $C_4H_7O$ ) in the mass spectra of disparinols A (**40**) and B (**41**) and racemosol (**113**) corresponds to fragmentation of the 2-hydroxy-3-methylbutyl-3-ene substituent [68, 101]. The presence in mammea A/AC cyclo F (**88**) of an oxobutyryl substituent and a hydroxyisopropylidihydrofuran ring is confirmed by the presence in the mass spectrum of two base peaks for  $[M - 43]^+$   $[M - CH_2CH_2CH_3]^+$  and  $[M - 115]^+$   $[M - 43 - 72]^+$ , which are formed by elimination of isobutene oxide from the  $[M - 43]^+$  fragment [101].

The mass spectrum of tomentolide A (**122**) [7] has a peak for the molecular ion at  $m/e$  402. A strong peak at  $m/e$  387  $[M - 15]$  corresponds to the stable benzopyryl ion 2,2-dimethylchromene. Other important peaks with  $m/e$  331  $[M - 15 - 56]$  and  $m/e$  303  $[M - 15 - 56 - 28]$  are formed by successive loss of butene and CO from the ion with  $m/e$  387 [7]. The spectrum of apetatolide (**1**) is characterized by a strong peak with  $m/e$  401  $[M - 15]$  and a peak with  $m/e$  361  $[M - 55]$  that correspond to loss of  $C_4H_7$  from the acyl substituent [7]. A strong base peak for  $[M - 15]$  in the mass spectrum of isomammeigin (**94**) indicates the presence in it of a 2,2-dimethylpyran ring [110].

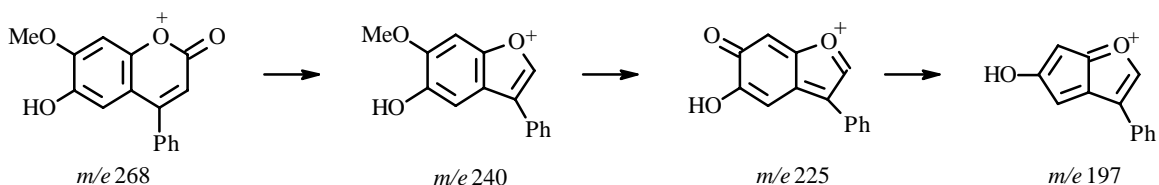
Two important conclusions were made about the common fragmentation pathways of 4-phenylcoumarins after a detailed investigation of the mass spectra of several natural neoflavones and their synthetic analogs [131]. Neoflavones without an O-substituent in the C-2'-position give stable  $[M - 28]^+$  fragments that are formed by decarboxylation. For 2'-oxygenated neoflavones, dehydroxylation or demethoxylation between the OH-5 and C-2' substituent forms the  $[M - 17]^+$  or  $[M - 59]^+$  ions. These characteristic fragmentations prompted a re-examination of the structure of coutareagenin [93, 131] (Scheme 2).

The fragmentation pattern of melanein (**102**) has been reported [53]. It was assumed that the ion with  $m/e$  271 is formed by loss of  $C_2H_3O$  from the molecular ion or loss of methyl from the ion with  $m/e$  286 (Scheme 3).



Scheme 3

An analogous fragmentation pattern was reported for dalbergin (**28**) [63]. The molecular ion with  $m/e$  268 loses the stable molecule  $\text{CO}_2$  to form the benzofuran cation with  $m/e$  240. Loss from this ion of a methyl forms a fragment with  $m/e$  225. Subsequent loss of  $\text{CO}_2$  forms a cation with  $m/e$  197.



The mass spectrum of stevenin (**117**) contains an ion with  $m/e$  241 that may be formed by loss of methyl from the cation with  $m/e$  256 or cleavage of a  $\text{C}_2\text{H}_3\text{O}$  fragment from the molecular ion with  $m/e$  284 [54].

The mass spectrum of serratin (**115**) has a characteristic molecular ion with  $m/z$  254 (100%) and two strong peaks with  $m/z$  226  $[\text{M} - \text{CO}]^+$  (95%) and  $m/z$  197  $[\text{M} - \text{CO} - \text{COH}]^+$  (47%) [36].

A strong peak for the molecular ion with  $m/z$  342  $[\text{M}]^+$  is observed in the mass spectrum of voludal (**9**). The decomposition of this ion by successive loss of two CO molecules gives fragments with  $m/z$  314  $[\text{M} - 28]^+$  and  $m/z$  286  $[\text{M} - 56]^+$  [17]. Ions with  $m/z$  313  $[\text{M} - 29]^+$  and  $m/z$  285  $[\text{M} - 57]^+$  are formed via loss of a  $-\text{CHO}$  radical and subsequent elimination of a CO molecule. A peak with  $m/z$  284  $[\text{M} - 58]^+$  is formed by loss of  $(\text{CHO} + \text{CO} + \text{H})$  from the molecular ion. A strong fragment with  $m/z$  256 corresponds to loss of two methyls from the ion with  $m/z$  286. Loss of a methyl and a water from the ion with  $m/z$  285  $[\text{M} - 57]^+$  forms cations with  $m/z$  270  $[\text{M} - 72]^+$  and  $m/z$  267  $[\text{M} - 75]^+$ , respectively. Fragments with  $m/z$  257  $[\text{M} - 87]^+$  and  $m/z$  255  $[\text{M} - 89]^+$  arise via loss of CO and two methyl radicals [17].

The FABMS spectrum of glucoside **27** contains a quasi-molecular ion with  $m/z$  447  $[\text{M} - \text{H}]^-$  and a base peak with  $m/z$  285  $[\text{M} - \text{H} - 162]^-$  that is formed by loss of the glucoside [10]. The spectrum of glucoside **26** exhibits an analogous quasi-molecular anion with  $m/z$  461  $[\text{M} - \text{H}]^-$  and a base peak with  $m/z$  299  $[\text{M} - \text{H} - 162]^-$ . The difference of 14 amu is consistent with the presence in compound **26** of an additional methyl group [10]. The FABMS of glycoside **2** has a quasi-molecular anion with  $m/z$  579  $[\text{M} - \text{H}]^-$ , a fragment with  $m/z$  447  $[\text{M} - \text{H} - 132]^-$  formed via loss of an apiofuranosyl unit, and a peak for the aglycon with  $m/z$  285  $[447 - 162]$  due to loss of the glucoside [10].

## NMR SPECTROSCOPY

Proton and carbon NMR methods can unambiguously establish the structures of neoflavones. Special investigations and analysis of  $^{13}\text{C}$  NMR spectra of natural and synthetic neoflavones have been reviewed [28]. 4-Phenylcoumarins exhibit signals for C-2, C-3, and C-4 at 159-162, 109-113, and 155-157 ppm, respectively.

Proton NMR spectra of neoflavones typically have a 1H singlet for H-3 in the range 5.70-6.15 ppm owing to the presence of an aryl substituent in the coumarin 4-position. A distinguishing feature of neoflavones that have unsubstituted 4-phenyls is a broad 5H singlet in the range 7.4-7.5 ppm or two multiplets at 7.3-7.4 (2H, H-2', H-6') and 7.4-7.5 ppm (3H, H-3', H-4', H-5').

The 4-(4-methoxy)phenyl substituent is characterized by a 3H singlet for the methoxyl at 3.84-3.87 ppm and two 2H doublets ( $J = 8-8.7$  Hz) in the ranges 6.87-6.96 (H-3', H-5') and 7.20-7.25 (H-2', H-6') ppm [11, 127]. An analogous pattern is observed with a hydroxyl in the 4'-position. The aromatic doublets resonate as doublets ( $J = 8-8.6$  Hz) at 6.73-6.92 (H-3', H-5') and 7.16-7.20 (H-2', H-6') ppm; the hydroxyl proton, at 8.51-9.50 ppm [12, 26, 27]. For a 3,4-dioxygenated 4-phenyl substituent, splitting occurs that is typical of a three-proton ABX system. There are three signals at 6.61-6.70 ppm (dd,  $J = 1.8-2.2$  Hz,  $J = 7.2-8.2$  Hz, 1H, H-6'), 6.71-6.86 ppm (d,  $J = 1.8-2.2$  Hz, 1H, H-2'), and 6.72-6.98 ppm (d,  $J = 7.2-8.2$  Hz, 1H, H-5') [10, 26, 30, 33, 93, 141, 142].

For 5,7-dioxygenated neoflavones, protons H-6 and H-8 resonate as two doublets with a *meta*-constant ( $J = 2.19-3.0$  Hz) at 6.19-6.40 and 6.43-6.56 ppm, respectively [11, 24, 27, 65, 93, 127, 142]. It should be mentioned that a 7-methoxyl appears as a singlet at 3.79-3.96 ppm [11, 24, 27, 65, 93, 127] whereas a 5-methoxyl is found at 3.47-3.56 ppm [24, 27, 65, 127, 142]. This is due to the diamagnetic effect of the 4-aryl substituent. Protons H-5 and H-6 of 6,7-disubstituted 4-arylcoumarins appear as singlets at 6.79-6.98 and 7.07-7.10 ppm, respectively [30]. A singlet for H-6 in spectra of 5,7,8-trisubstituted neoflavones is observed at 6.57-6.65 ppm (DMSO- $d_6$ ) [26, 33, 141] or 6.30-6.35 ppm ( $\text{CDCl}_3$ ) [143].

A distinguishing feature of 4-phenylcoumarins containing 3-methylbutanoic acid is the presence of a 6H doublet ( $J = 6.5-7.0$  Hz) for the methyls at 0.9-1.08 ppm, a multiplet for the CH at 2.14-2.37 ppm, and a doublet ( $J = 6.5-7.0$  Hz) for the methylene at 2.84-3.30 ppm [66, 67, 68, 101, 105, 110, 111, 116]. Signals at 0.88-1.04 ppm (3H, t,  $J = 7.0-7.5$  Hz,  $\text{CH}_3-5$ ), 1.12-1.32 ppm (3H, d,  $J = 6.5-7.0$  Hz,  $\text{CH}_3-3$ ), 1.30-1.50 ppm (1H, m,  $\text{CH}_2-4a$ ), 1.77-1.92 ppm (1H, m,  $\text{CH}_2-4b$ ), and 3.54-4.00 ppm (1H, m, H-2) are typical of a 2-methylbutanoyl moiety [5, 66, 67, 68, 101, 105, 116]. The presence of an *n*-butanoyl substituent in neoflavones gives rise to PMR signals at 0.93-1.07 ppm (3H, t,  $J = 7.0-7.5$  Hz,  $\text{CH}_3-4$ ), 1.68-1.83 ppm (2H, m,  $\text{CH}_2-3$ ), and 2.98-3.34 ppm (2H, t,  $J = 7.0-7.5$  Hz,  $\text{CH}_2-2$ ) [72, 101, 117]. The 2-methylpropionyl group has a characteristic 6H doublet ( $J = 7.0-10.0$  Hz) for the two methyls at 1.10-1.35 ppm and a multiplet for the CH groups at 3.70-3.73 ppm [67, 105, 109, 120]. PMR spectra of neoflavones with a benzoyl group in the coumarin 8-position exhibit characteristic signals at 7.45-7.51 ppm (2H, t,  $J = 7.4-8.0$  Hz, H-3, H-5), 7.57-7.63 ppm (1H, tt,  $J = 7.4-8.4$  Hz,  $J = 1.0-1.7$  Hz, H-4''), and 7.65-7.89 ppm (2H, dd,  $J = 7.4-8.4$  Hz,  $J = 1.0-1.7$  Hz, H-2, H-6) [14, 20, 79, 80]; 6-benzoyl-4-phenylcoumarins, at 7.39-7.43 ppm (2H, m, H-3, H-5) and 7.48-7.53 ppm (3H, m, H-2, H-4, H-6) [14, 70].

Two 3H singlets at 1.63-1.94 ppm, a 2H doublet ( $J = 6.9-8.0$  ppm) at 3.08-3.57 ppm, and a 1H doublet ( $J = 6.0-7.5$  Hz) or triplet ( $J = 7.0-7.5$  Hz) at 5.03-5.40 ppm indicate the presence of a prenyl group in the neoflavone [14, 21, 72, 80, 85, 105, 116, 117]. Signals at 1.81-1.95 ppm (3H, s,  $\text{CH}_3-5$ ), 2.77-3.06 ppm (1H, dd,  $J = 7.5-8.0$  Hz,  $J = 15.0-15.5$  Hz,  $\text{CH}_2-1a$ ), 3.10-3.32 ppm (1H, dd,  $J = 2.0$  Hz,  $J = 15.0$  Hz,  $\text{CH}_2-1b$ ), 4.32-4.51 ppm (1H, d,  $J = 6.0-8.0$  Hz, H-2), 4.86-4.93 ppm (1H, s,  $\text{CH}_2-4a$ ), and 4.94-5.03 ppm (1H, s,  $\text{CH}_2-4b$ ) are found in PMR spectra of 4-phenylcoumarins with a 2-hydroxy-3-methylbut-3-enyl substituent [67, 68, 72, 101].

Two 1H doublets ( $J = 2.0$ ) at 7.16-7.17 ppm and 7.66-7.68 ppm in the PMR spectra of compounds **43** and **128** belong to H-3 and H-2, respectively, of the furan ring fused at the 7- and 8-positions to the coumarin [66, 72]. The diamagnetic effect of the 4-phenyl group in **48** shifts the furan signals to strong field at 6.96 and 7.31 ppm, respectively [66].

Two methyl singlets at 0.94-1.48 ppm, two 1H doublets of doublets at 2.93-3.31 ppm ( $J = 8.1-10.0$  Hz,  $J = 15.0-15.5$  Hz), and a 1H triplet or doublet of doublets at 4.52-4.92 ppm ( $J = 8.1-9.8$  Hz) are characteristic of a 2,3-dihydro-2-(2-hydroxy-2-methylethyl)furan ring fused to a 4-phenylcoumarin [14, 20, 66, 101, 105].

A distinguishing feature of PMR of neoflavones containing a fused 2,2-dimethylpyran ring is the presence of two doublets ( $J = 9.9-11.0$  Hz) for the pyran olefinic protons at 5.33-5.67 (H-3) and 6.50-6.94 ppm (H-4) [7, 5, 20, 21, 29, 70, 73-75, 79, 80, 105, 109-111, 117, 120, 138]. A 2,2-dimethyl group for 6,7- and 7,8-pyrano-4-phenylcoumarins usually gives a 6H singlet or two 3H singlets at 1.38-1.65 ppm [7, 5, 21, 29, 109, 117, 120]. These methyls for 5,6-pyrano-coumarins lie in the



space over the plane of the 4-phenyl substituent. This leads to a diamagnetic shift of their signals by about 0.5-0.6 ppm, as a result of which they are observed at 0.91-1.00 ppm as a 6H singlet or two 3H singlets [5, 20, 21, 73-75, 79, 105, 110, 138]. PMR spectra of neoflavones containing fused 2,3-dimethylchromanone and 2,3-dimethylchromanol rings have been reviewed [148].

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