

NEOFLAVONES. 1. NATURAL DISTRIBUTION AND SPECTRAL AND BIOLOGICAL PROPERTIES

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UDC 547.814.5

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 C₆—C₃—C₆. 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 dalbergiquinols. 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.

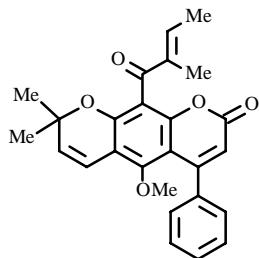
NATURAL DISTRIBUTION

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



1. Apetatolide*

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

$C_{26}H_{24}O_5$, mp 203-205°C [7]

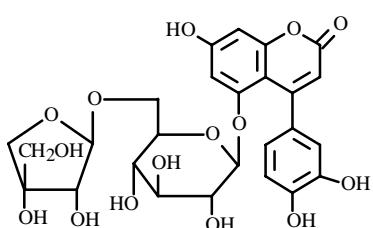
UV [7]: 236 (4.55), 275 (4.40)

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]

*refined structure [5]



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

Coutarea hexandra [10]

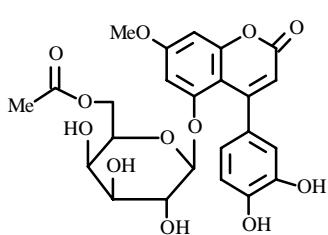
$C_{26}H_{28}O_{15}$

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, $J = 2, H-2'$).

¹³C 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)- β -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]

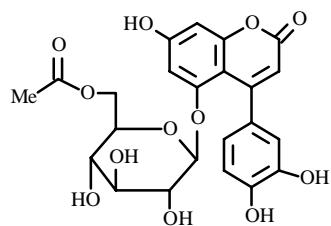
UV (MeOH) [11]: 218, 258, 328

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')

¹³C NMR (50 MHz, DMSO-d₆) [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 ₃ CO	20.35
6	98.94	3'	144.13	3''	72.66	CH ₃ CO	170.12



4. 5-O-(6''-Acetyl-β-D-glucopyranosyl)-7,3',4'-trihydroxy-4-phenylcoumarin

Hintonia latiflora [13]

C₂₃H₂₂O₁₂, mp 205-208°C [13]

[α]_D -129° (MeOH) [13]

UV (MeOH) [13]: 258, 331

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

PMR (300 MHz, DMSO-d₆) [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')

¹³C NMR (75 MHz, DMSO-d₆) [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]

C₂₈H₂₄O₇, mp 206-208°C [14]

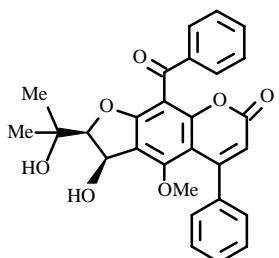
[α]_D -8.4° (CHCl₃) [14]

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) [M]⁺, 454 (4), 439 (4), 397 (100), 368 (50), 319 (40)

HREIMS [14]: 472.1514 [M]⁺



PMR (300 MHz, CDCl₃) [14]: 1.27 (3H, s, CH₃-13), 1.48 (3H, s, CH₃-12), 3.60 (3H, s, CH₃O-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'')

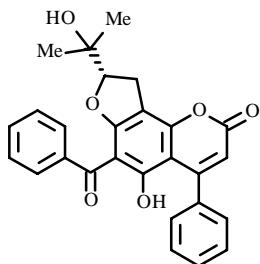
NOE, HMQC, HMBC [14]

¹³C NMR (75 MHz, CDCl₃) [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-phenylcolumbianelin



Calophyllum teysmannii var. *inophyllolide* [14]

C₂₇H₂₂O₆

[α]_D -80.5° (CHCl₃) [14]

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

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

Mass [14]: 442 (30) [M]⁺, 424 (20), 409 (24), 383 (100)

HREIMS [14]: 442.1423 [M]⁺

PMR (300 MHz, CDCl₃) [14]: 0.94 (3H, s, CH₃-13), 1.10 (3H, s, CH₃-12), 3.17 (1H, dd, J = 9.1, J = 15.5, CH₂-9b), 3.23 (1H, dd, J = 9.4, J = 15.5, CH₂-9a), 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)

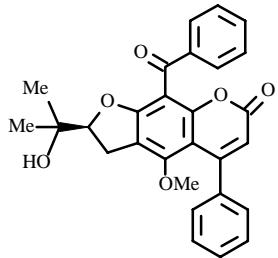
NOE, HMQC, HMBC [14]

¹³C NMR (75 MHz, CDCl₃) [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-phenylnodakenin



Calophyllum teysmannii var. *inophyllolide* [14]

C₂₈H₂₄O₆, mp 208-210°C [14]

[α]_D -18.1° (CHCl₃) [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) [M]⁺, 398 (100)

HREIMS [14]: 456.1551 [M]⁺

PMR (300 MHz, CDCl₃) [14]: 1.13 (3H, s, CH₃-13), 1.14 (3H, s, CH₃-12), 3.23 (1H, dd, J = 9.4, J = 15.4, CH₂-6b), 3.30 (3H, s, CH₃O-5), 3.31 (1H, dd, J = 8.1, J = 15.5, CH₂-6a), 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'')

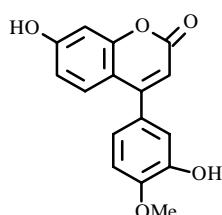
NOE, HMQC, HMBC [14]

X-ray [14]

¹³C NMR (75 MHz, CDCl₃) [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 ₃ O-5	59.3
7	91.4						

*Assignments may be reversed



8. Volubolin

Dalbergia volubilis [15, 16]

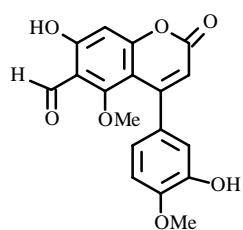
C₁₆H₁₂O₅

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, CDCl₃) [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]

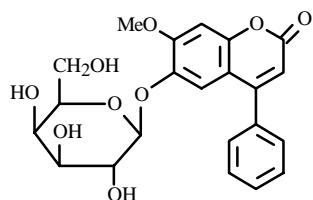
C₁₈H₁₄O₇

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)

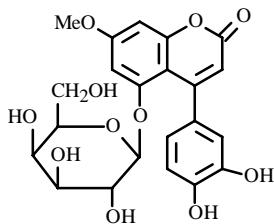
PMR (60 MHz, CDCl₃) [16]: 3.8 (3H, s, OMe), 4.0 (3H, s, OMe), 5.95 (1H, s, H-3), 7.3 (3H, m, H-2', H-5', H-6'), 7.5 (1H, s, H-8), 9.72 (1H, s, CHO), 9.85 (1H, s, OH), 10.2 (1H, s, OH)



10. 6-O-β-D-Galactopyranosyldalbergin

Hesperethusa crenulata [18]

C₂₂H₂₂O₉



11. 5-O- β -D-Galactopyranosyl-3',4'-dihydroxy-7-methoxy-4-phenylcoumarin

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

C₂₂H₂₂O₁₁, mp 228-231°C [19]

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

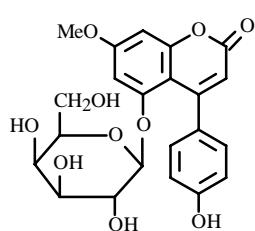
IR (KBr) [19]: 3650, 3403, 1719, 1613, 1160, 1078, 1049

PMR (80 MHz, C₅H₅N-d₆) [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'')

¹³C NMR (50 MHz, DMSO-d₆) [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 µg/mL [11]



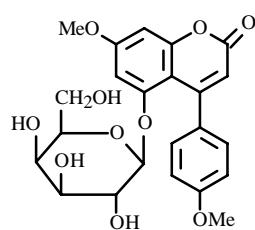
12. 5-O- β -D-Galactopyranosyl-4'-hydroxy-7-methoxy-4-phenylcoumarin

Hintonia latiflora [12]

C₂₂H₂₂O₁₀, mp 218-221°C [12]

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

PMR (90 MHz, DMSO-d₆) [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- β -D-Galactopyranosyl-7,4'-dimethoxy-4-phenylcoumarin

Exostema caribaeum [11]

C₂₃H₂₄O₁₀, mp 217-221°C [11]

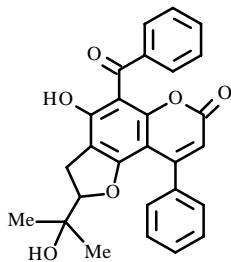
UV (MeOH) [11]: 212, 236

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

PMR (80 MHz, DMSO-d₆) [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')

¹³C NMR (50 MHz, DMSO-d₆) [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) [M]⁺, 424 (6), 409 (30), 383 (83), 105 (100), 77 (77)

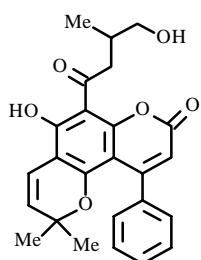
HREIMS [20]: 442.1427 [M]⁺

PMR (500 MHz, CDCl₃) [20]: 0.96 (3H, s, CH₃-12), 1.05 (3H, s, CH₃-13), 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]

¹³C NMR (125 MHz, CDCl₃) [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]

C₂₅H₂₄O₆

[α]_D -1.92° (CHCl₃) [21]

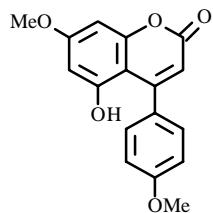
Mass [21]: 402 (30) [M - H₂O]⁺, 388 (28), 387 (100), 359 (6), 331 (16)

PMR (300 MHz, CDCl₃) [21]: 0.95 (3H, s, CH₃-6'), 0.97 (3H, s, CH₃-5'), 1.01 (3H, d, J = 6.7, CH₃-5''), 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)

¹H-¹³C COSY [21]

¹³C NMR (75 MHz, CDCl₃) [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]

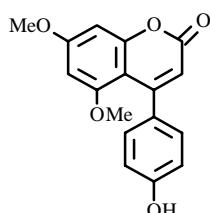
C₁₇H₁₄O₅, mp 210-212°C [22], 212°C [23]

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

¹³C NMR (125.7 MHz, CDCl₃) [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⁺, KB-V⁻, LNCaP, SW626, SKNSH, and M109 [22]



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

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

C₁₇H₁₄O₅, 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

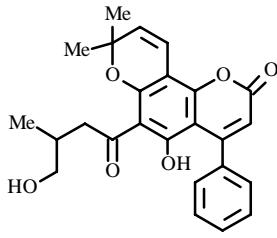
Mass [24]: 298 (100) [M]⁺, 270 (82), 255 (29), 227 (15)

PMR [200 MHz, (CD₃)₂CO] [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')

¹³C NMR (100 MHz, DMSO-d₆) [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]

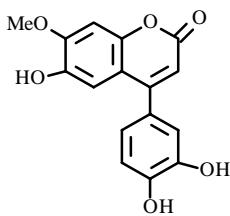
C₂₅H₂₄O₆, mp 174-176°C [29]

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, CDCl₃) [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'-Hydroxymelanatin

Dalbergia odorifera [30, 31]

C₁₆H₁₂O₆, 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)

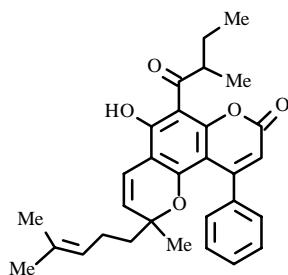
HRMS [30]: 300.0633

PMR (300 MHz, DMSO-d₆) [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]

¹³C NMR (75.4 MHz, DMSO-d₆) [30]:

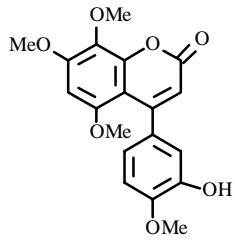
C-2	160.99	C-6	143.79	C-10	111.49	C-4'	147.41
3	110.59	7	152.12	1'	126.68	5'	120.26
4	155.72	8	100.89	2'	145.81	6'	116.08
5	111.11	9	148.83	3'	116.26	7-OMe	56.55



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]

C₃₀H₃₂O₅



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

Coutarea hexandra [33]

PMR [300 MHz, ($\text{CD}_3\text{CO}_2\text{CO}$) [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, $J = 8.2, 2, \text{H}-6'$), 6.87 (1H, d, $J = 2, \text{H}-2'$), 6.98 (1H, d, $J = 8.2, \text{H}-5'$), 7.75 (1H, s, 3'-OH)

IR (CDCl_3) [33]: 3540, 1718, 1610, 1595, 1513, 1348, 1280, 1244, 1109, 1060, 862

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

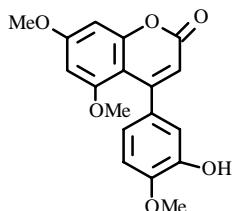
PMR [300 MHz, ($\text{CD}_3\text{CO}_2\text{CO}$) [33]: 3.56 (3H, s, 5-OMe), 3.83 (3H, s, 8-OMe), 3.90 (3H, s, 4'-OMe), 3.99 (3H, s, 7-OMe), 5.87 (1H, s, H-3), 6.64 (1H, s, H-6), 6.79 (1H, dd, $J = 8.2, 2, \text{H}-6'$), 6.87 (1H, d, $J = 2, \text{H}-2'$), 6.98 (1H, d, $J = 8.2, \text{H}-5'$), 7.75 (1H, s, 3'-OH)

$^{13}\text{C NMR}$ (75 MHz, DMSO-d_6) [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 $\text{IC}_{50} 12.5 \times 10^{-5} \text{ M}$ [34]



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

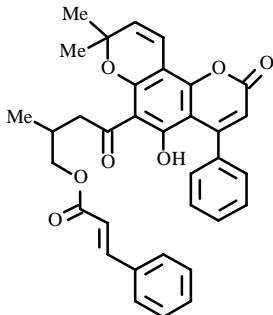
Coutarea hexandra [24], *Exostema acuminatum* [22], *E. mexicanum* [26]

PMR (CDCl_3) [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)

$^{13}\text{C NMR}$ (125.7 MHz, DMSO-d_6) [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⁺, KB-V⁻, 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

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

C₃₄H₃₀O₇

[α]_D -6.6° (CHCl₃) [21]

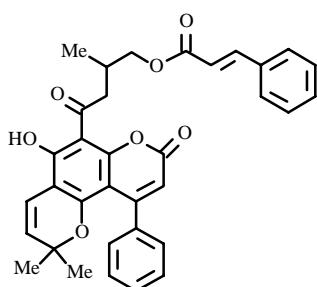
Mass [21]: 550 (7) [M]⁺, 402 (31), 388 (19), 387 (82), 347 (10), 331 (28), 147 (52), 131 (100), 103 (56)

PMR (300 MHz, CDCl₃) [21]: 1.06 (3H, d, J = 6.7, CH₃-5''), 1.55 (3H, s, CH₃-6''), 1.57 (3H, s, CH₃-5''), 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, CH₂-4''), 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)

¹H-¹³C COSY [21]

¹³C NMR (75 MHz, CDCl₃) [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

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

C₃₄H₃₀O₇

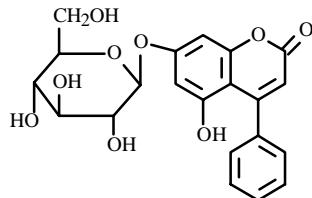
[α]_D -7.14° (CHCl₃) [21]

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

PMR (300 MHz, CDCl₃) [21]: 0.92 (3H, s, CH₃-6''), 0.93 (3H, s, CH₃-5''), 1.18 (3H, d, J = 6.8, CH₃-5''), 2.71 (1H, m, H-3''), 3.28-3.48 (2H, m, CH₂-2''), 4.17-4.29 (2H, m, CH₂-4''), 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)

¹³C NMR (75 MHz, CDCl₃) [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. Serratin 7- β -glucoside

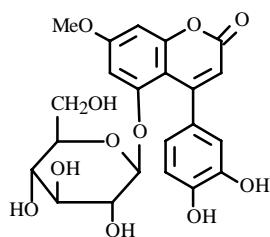
Passiflora serratodigitata [36]

C₂₁H₂₀O₉, 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-d₆) [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-(β -D-Glucopyranosyl)-3',4'-dihydroxy-7-methoxy-4-phenylcoumarin

Hintonia latiflora [12], *Coutarea hexandra* [10], *Exostema caribaeum* [11], *E. mexicanum* [37]

C₂₂H₂₂O₁₁, mp 237-238°C [11]

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

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

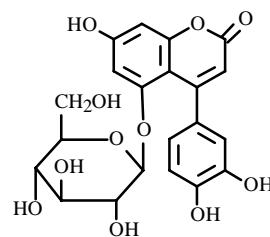
Mass [10]: 461 [M - H]⁻, 299

PMR (250 MHz, CD₃OD) [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')

¹³C NMR (69.5 MHz, CD₃OH) [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-(β -D-Glucopyranosyl)-7,3',4'-trihydroxy-4-phenylcoumarin

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

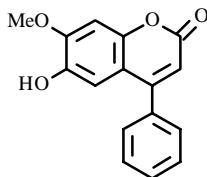
C₂₁H₂₀O₁₁, mp 250-252°C [12]

Mass [10]: 447 [M - H]⁻, 285

PMR (250 MHz, CD₃OD) [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')

¹³C NMR (69.5 MHz, CD₃OD) [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. melanoxyylon* [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

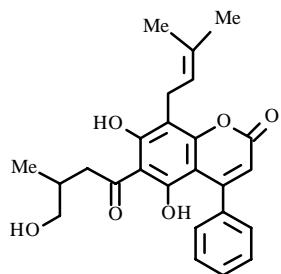
Mass [63]: 268 (100) $[M]^+$, 240, 225, 197, 168, 155, 141, 127

PMR (200 MHz, DMSO-d₆) [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]

¹³C NMR (75.4 MHz, DMSO-d₆) [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-butenoyl)-4-phenyl-2H-1-benzopyran-2-one

Kielmeyera reticulata [21]

$C_{25}H_{26}O_6$

$[\alpha]_D +1.58^\circ$ (CHCl₃) [21]

Mass [21]: 404 (20) $[M - H_2O]^+$, 389 (15), 361 (100), 349 (30), 327 (6)

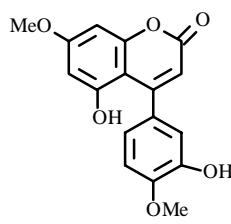
PMR (300 MHz, CDCl₃) [21]: 0.99 (H, d, J = 6.6, CH₃-5''), 1.63 (3H, s,

CH₃-6''), 1.69 (3H, s, CH₃-5''), 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, CH₂-4''), 3.41 (1H, dd, J = 8.8, J = 11.3, H-2b''), 3.75 (2H, m, CH₂-4''), 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)

¹H-¹³C COSY [21]

¹³C NMR (75 MHz, CDCl₃) [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]

C₁₇H₁₄O₆, 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

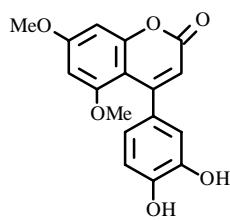
Mass [11]: 314 (100), 286 (64.8), 271 (37.7)

PMR (80 MHz, CDCl₃) [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')

¹³C NMR (125.7 MHz, DMSO-d₆) [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⁺, KB-V⁻, LNCaP, SW626, SKNSH, and M109 [22]



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

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

C₁₇H₁₄O₆, mp 211-212°C [24, 64]

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

IR (CDCl₃) [24]: 3600, 3540, 3260, 1707, 1611, 1595, 1513, 1158, 1111, 1052, 945, 910, 872, 858, 828

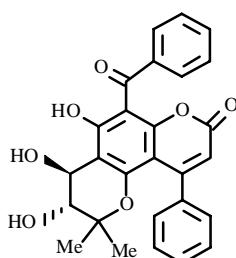
Mass [24]: 314 (100) [M]⁺, 296 (98), 271 (20), 243 (4)

PMR [(CD₃)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)

¹³C NMR (100 MHz, DMSO-d₆) [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 IC₅₀ 11.7×10⁻⁵ M [34], antiplasmodial activity against *Plasmodium falciparum* [26]



32. trans-7,8-Dihydroxycalanone

Calophyllum teysmannii var. *inophyllolide* [14]

C₂₇H₂₂O₇

[α]_D -31.2° (CHCl₃) [14]

UV (EtOH) [14]: 252, 324

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

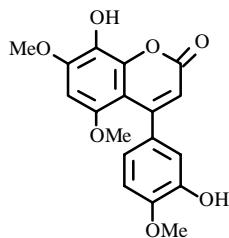
Mass [14]: 440 (100) $[M - 18]^+$, 412 (20), 397 (30), 368 (50), 319 (40)

PMR [300 MHz, $(CD_3)_2CO$] [14]: 0.84 (3H, s, CH₃-13), 1.02 (3H, s, CH₃-12), 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]

¹³C NMR [75 MHz, $(CD_3)_2CO$] [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]

$C_{18}H_{16}O_7$, **mp** 191–192°C [33]

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

IR ($CDCl_3$) [33]: 3545, 1712, 1610, 1590, 1512, 1176, 1112, 1050, 860

Mass [33]: 344 (100) $[M]^+$, 329 (10), 316 (25), 301 (25), 297 (25), 273 (13), 269 (23)

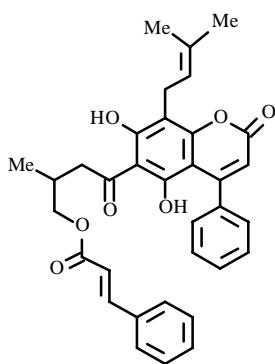
PMR [300 MHz, $(CD_3)_2CO$] [33]: 3.51 (3H, s, 5-OMe), 3.90 (3H, s, 4'-OMe), 3.97 (3H, s, 7-OMe), 5.85 (1H, s, H-3), 6.65 (1H, s, H-6), 6.79 (1H, dd, J = 8.2, J = 2, H-6'), 6.83 (1H, d, J = 2, H-2'), 6.98 (1H, d, J = 8.2, H-5'), 7.73 (1H, s, OH), 7.82 (1H, s, OH)

¹³C NMR (75 MHz, DMSO-d₆) [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⁺, KB-V⁻, LNCaP, SW626, SKNSH, and M109 [22]



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

Kielmeyera reticulata [21]

$C_{34}H_{32}O_7$

$[\alpha]_D$ -12.17° ($CHCl_3$) [21]

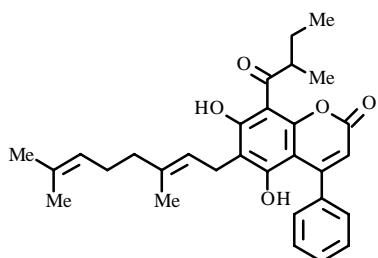
Mass [21]: 404 (38) $[M - C_6H_5C_2H_2COOH]^+$, 389 (11), 361 (38), 349 (21), 321 (9), 131 (100)

PMR (300 MHz, $CDCl_3$) [21]: 1.18 (1H, d, J = 6.0, CH₃-5''), 1.64 (3H, s, CH₃-6'), 1.69 (3H, s, CH₃-5'), 2.71 (1H, m, H-3''), 3.27 (2H, d, J = 8.0,

$\text{CH}_2\text{-}4'$), 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, $\text{CH}_2\text{-}4''$), 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)

^{13}C NMR (75 MHz, CDCl_3) [21]:

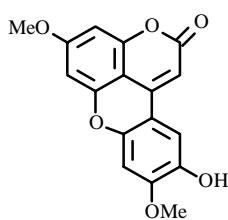
C-2	158.9	C-2'	134.7	C-1'''	137.4	C-1C	167.1
3	112.8	3'	121.3	2''	128.0	2C	118.7
4	154.6	4'	22.1	3'''	130.1	3C	145.2
4a	101.1	5'	17.9	4'''	130.7	4C	135.1
5	156.5	6'	26.2	5'''	130.1	5C	128.6
6	105.2	1''	205.5	6'''	128.0	6C	129.4
7	167.4	2''	49.3			7C	130.8
8	113.2	3''	30.7			8C	129.4
8a	157.9	4'	69.4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

$\text{C}_{30}\text{H}_{34}\text{O}_5$



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

Exostema caribaeum [19]

$\text{C}_{17}\text{H}_{12}\text{O}_7$, mp 273-274°C [19]

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

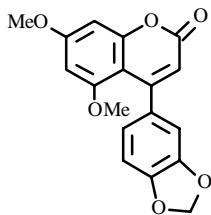
IR (KBr) [19]: 3400, 3330, 3175, 3160, 1677, 1618, 1523, 1458, 1289, 1205, 1169, 1120, 817

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

PMR (80 MHz, $\text{C}_5\text{H}_5\text{N-d}_6$) [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')

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

C₁₈H₁₄O₆, mp 194–195°C [65]

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

IR (CDCl₃) [65]: 1710, 1613, 1595, 1505, 1157, 1110, 1052, 1039, 938, 904, 858, 810

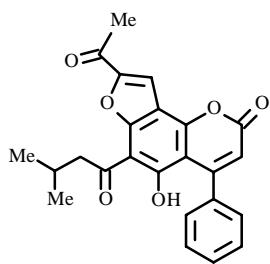
Mass [65]: 326 (81) [M]⁺, 298 (100), 289 (13), 255 (4)

PMR (60 MHz, CDCl₃) [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)

¹³C NMR (100 MHz, DMSO-d₆) [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 ₂ O	100.8
5	157.9						

38. Disparacetyl furan A



Calophyllum dispar [66]

C₂₄H₂₀O₆

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 (CHCl₃) [66]: 3460, 1717, 1684, 1625, 758, 701
HRFABMS [66]: 403.1186 [M - H]⁻

PMR (270 MHz, CDCl₃) [66]: 1.07 (6H, d, J = 6.5, CH₃-4'', CH₃-5''), 2.29 (1H, m, CH-3''), 2.63 (1H, s, CH₃-5'''), 3.18 (2H, d, J = 6.5, CH₂-2''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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. Dispardiol B

Calophyllum dispar [67]

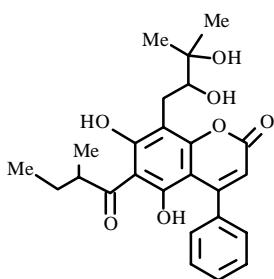
C₂₅H₂₈O₇

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 (CHCl₃) [67]: 3460, 1717, 1706, 1617, 768, 702

HRLSIMS [67]: 441.1927 [M + H]⁺

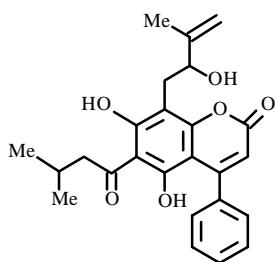
HMBC [67]



PMR (500 MHz, CDCl₃) [67]: 0.90 (3H, t, J = 7.5, CH₃-5''), 1.15 (3H, d, J = 6.5, CH₃-3''), 1.36 (1H, s, CH₃-4''), 1.40 (1H, m, CH₂-4a''), 1.49 (1H, s, CH₃-5''), 1.80 (1H, m, CH₂-4b''), 2.61 (1H, dd, J = 10.0, J = 15.0, CH₂-1a''), 3.37 (1H, d, J = 15.0, CH₂-1b''), 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)

¹³C NMR (125 MHz, CDCl₃) [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]

C₂₅H₂₆O₆, 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)

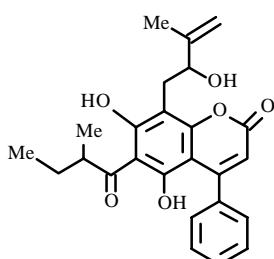
HREIMS [68]: 422.1726 [M]⁺

PMR (270 MHz, CDCl₃) [68]: 0.94 (6H, dd, J = 2.0, J = 6.5, CH₃-5''), 1.93 (3H, s, CH₃-5''), 2.23 (1H, m, CH-4''), 3.02 (2H, d, J = 6.5, CH₂-2''), 3.04 (1H, dd, J = 8.0, J = 15.5, CH₂-1a''), 3.32 (1H, dd, J = 2.0, J = 15.0, CH₂-1b''), 4.50 (1H, d, J = 7.5, H-2''), 4.94 (1H, s, CH₂-4a''), 5.03 (1H, s, CH₂-4b''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₅H₂₆O₆

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]⁺

PMR (270 MHz, CDCl₃) [68]: 0.90 (3H, t, J = 7.0, CH₃-5''), 1.14 (3H, d, J = 6.5, CH₃-3''), 1.39 (1H, m, CH₂-4a''), 1.79 (1H, m, CH₂-4b''), 1.94 (3H, s, CH₃-5'''), 3.06 (1H, dd, J = 7.5, J = 15.0, CH₂-1a''), 3.30 (1H, dd, J = 2.0, J = 15.0, CH₂-1b''), 3.84 (1H, m, H-2''), 4.51 (1H, d, J = 6.0, H-2''), 4.93 (1H, s, CH₂-4a''), 5.02 (1H, s, CH₂-4b''), 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)

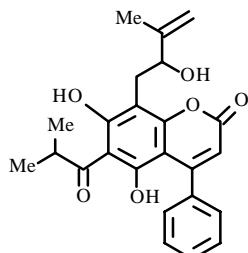
DQF-COSY, HMQC, HMBC [68]

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 7 µg/mL [67]

42. Disparinol D



Calophyllum dispar [67]

C₂₄H₂₄O₆

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 (CHCl₃) [67]: 3420, 1732, 1716, 1622, 1595, 756, 702

HRFABMS [67]: 407.1530 [M - H]⁻

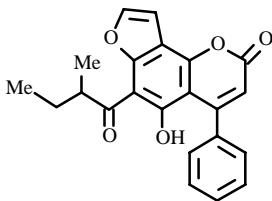
PMR (500 MHz, CDCl₃) [67]: 1.16 (6H, d, J = 7.0, CH₃-3'', CH₃-4''), 1.95 (3H, s, CH₃-5''), 3.05 (1H, dd, J = 8.0, J = 15.5, CH₂-1a''), 3.13 (1H, d, J = 7.0, H-2''), 3.32 (1H, dd, J = 2.0, J = 15.0, CH₂-1b''), 4.51 (1H, d, J = 7.5, H-2''), 4.94 (1H, s, CH₂-4b''), 5.03 (1H, s, CH₂-4a''), 5.97 (1H, s, H-3), 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]

¹³C NMR (125 MHz, CDCl₃) [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 ED₅₀ 21 µg/mL [67]



43. Disparfuran B

Calophyllum dispar [66]

C₂₂H₁₈O₅

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 (CHCl₃) [66]: 3440, 1732, 1717, 1622, 1559, 756, 700

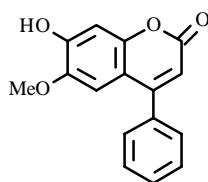
HRAPCIMS [66]: 363.1248 [M + H]⁺

PMR (270 MHz, CDCl₃) [66]: 0.97 (3H, t, J = 7.5, CH₃-5''), 1.24 (3H, d, J = 6.5, CH₃-3''), 1.55 (1H, m, CH₂-4''β), 1.86 (1H, m, CH₂-4''α), 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]

¹³C NMR (67.5 MHz, CDCl₃) [66]:

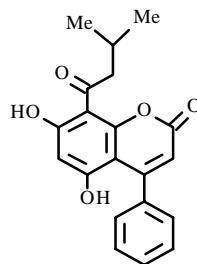
C-2	159.4	C-8	109.8	C-4'	128.4	C-3''	16.3
3	114.3	9	153.4	5'	127.7	4''	26.5
4	156.8	10	103.3	6'	127.1	5''	11.8
5	163.5	1'	138.9	1''	208.6	2'''	143.8
6	104.9	2'	127.1	2''	45.7	3'''	104.7
7	155.8	3'	127.7				



44. Isodalbergin

Dalbergia sissoo [69]

C₁₆H₁₂O₄, mp 195-196°C [69]



45. Isodispar B

Calophyllum dispar [67]

C₂₀H₁₇O₅

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 (CHCl₃) [67]: 3500, 1684, 1622, 1594, 774, 700

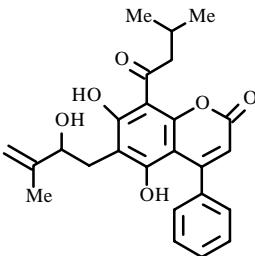
HRFABMS [67]: 337.1174 [M - H]⁻

PMR (500 MHz, CDCl₃) [67]: 1.06 (6H, d, J = 6.5, CH₃-4'', CH₃-5''), 2.31 (1H, m, H-3''), 3.19 (2H, d, J = 6.5, CH₂-2''), 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)

¹³C NMR (125 MHz, CDCl₃) [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 ED₅₀ 8 µg/mL [67]



46. Isodisparinol A

Calophyllum dispar [68]

C₂₅H₂₆O₆

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]⁺

PMR (270 MHz, CDCl₃) [68]: 1.06 (6H, d, J = 7.0, CH₃-5''), 1.81 (3H, s, CH₃-5''), 2.32 (1H, m, CH-4''), 2.77 (1H, dd, J = 8.0, J = 15.0, CH₂-1a''), 3.13 (1H, dd, J = 2.0, J = 15.0, CH₂-1b''), 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, CH₂-4a''), 4.93 (1H, s, CH₂-4b''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₅H₂₆O₆

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

HRLSIMS [68]: 423.1808 [M + H]⁺

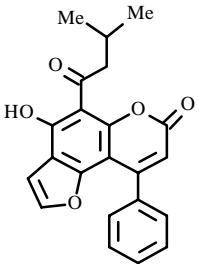
PMR (270 MHz, CDCl₃) [68]: 1.03 (3H, t, J = 7.5, CH₃-5''), 1.29 (3H, dd, J = 8.0, J = 6.5, CH₃-3''), 1.53 (1H, m, CH₂-4a''), 1.82 (3H, s, CH₃-5''), 1.95 (1H, m, CH₂-4b''), 2.78 (1H, dd, J = 8.5, J = 15.0, CH₂-1a''), 3.12 (1H, dd, J = 2.0, J = 15.0, CH₂-1b''), 3.99 (1H, m, CH₂-2''), 4.32 (1H, d, J = 8.0, H-2''), 4.87 (1H, s, CH₂-4a''), 4.94 (1H, s, CH₂-4b''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₂H₁₈O₅

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)

IR (CHCl₃) [66]: 3450, 2930, 1744, 1609, 756, 702

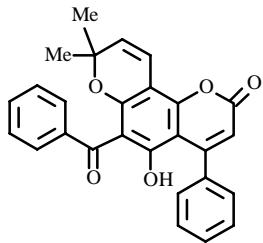
HREIMS [66]: 362.1170 [M]⁺

PMR (270 MHz, CDCl₃) [66]: 1.09 (6H, d, J = 7.0, CH₃-4'', CH₃-5''), 2.37 (1H, m, H-3'''), 3.30 (2H, d, J = 7.0, CH₂-2'''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₇H₂₀O₅, 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, CDCl₃) [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)

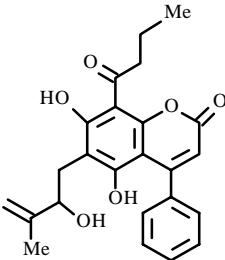
HMBC [70]

X-ray [70, 71]

¹³C NMR (125 MHz, CDCl₃) [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]

C₂₄H₂₄O₆

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]⁺

PMR (270 MHz, CDCl₃) [72]: 1.07 (3H, t, J = 7.5, CH₃-4''), 1.78 (2H, m, CH₂-3'''), 1.81 (3H, s, CH₃-5''), 2.78 (1H, dd, J = 2, J = 15, CH₂-1a''), 3.10 (1H, dd, J = 2, J = 15, CH₂-1b''), 3.34 (2H, t, J = 7.0, CH₂-2''), 4.32 (1H, d, J = 7.5, H-2''), 4.86 (1H, s, CH₂-4b''), 4.93 (1H, s, CH₂-4a''), 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)

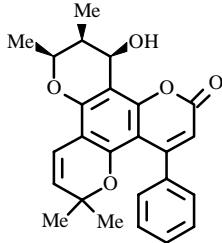
HMBC [72]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₅H₂₄O₅, mp 193-195°C [75], 200-201°C [73]

[α]_D +43° (acetone) [73, 76], +68.8° (CHCl₃) [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]⁺, 389 (100), 386 (1), 371 (4), 333 (50)

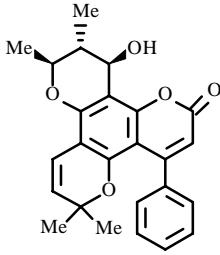
PMR (CDCl₃) [75]: 0.97 (3H, s, CH₃-10), 0.98 (3H, s, CH₃-10), 1.16 (3H, d, J = 7.2, CH₃-3), 1.43 (3H, d, J = 6.92, CH₃-2), 2.27 (1H, m, J = 5.19, J = 3.46, H-3), 4.43 (1H, m, J = 6.92, J = 3.46, H-2), 5.17 (1H, d, J = 5.19, H-4), 5.33 (1H, d, J = 9.9, H-11), 5.96 (1H, s, H-7), 6.51 (1H, d, J = 9.9, H-12), 7.30 (5H, m, Ph-8)

X-ray [76]

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 30 μM [74], antitumor activity [9]



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

Calophyllum inophyllum [73, 74, 77]

C₂₅H₂₄O₅, mp 200-202°C [73]

[α]_D +36° (acetone) [76]

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

IR (mineral oil) [73]: 3430, 1717, 1767, 703

Mass [73]: 404 [M]⁺, 386, 371

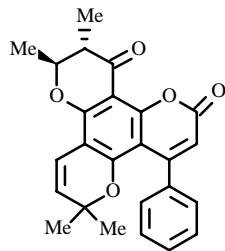
PMR (400 MHz, CDCl₃) [74]: 0.91 (3H, s, CH₃-20), 0.97 (3H, s, CH₃-19), 1.18 (3H, d, J = 6.8, CH₃-22), 1.47 (3H, d, J = 6.4, CH₃-21), 1.97 (1H, ddq, J = 6.8, J = 9.1, J = 7.8, H-11), 3.96 (1H, dq, J = 6.4, J = 9.1, H-10), 4.79 (1H, d, J = 7.8, H-12), 5.37 (1H, d, J = 10.0, H-7), 5.97 (1H, s, H-3), 6.53 (1H, d, J = 10.0, H-8), 7.41, 7.31 (5H, m, Ph-4)

X-ray [76]

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 0.038 μM [74], activity against HIV-1 in cell culture at IC₅₀ 1.4 μM [74]



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

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

C₂₅H₂₂O₅, mp 186-188°C [78], 188-191°C [73]

[α]_D +13° (CHCl₃) [73]

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

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

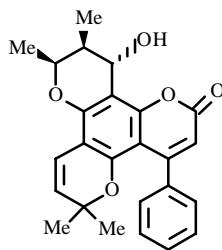
Mass [73]: 402 [M]⁺, 387 (15), 331 (56), 303 (28)

PMR (60 MHz, CDCl₃) [73]: 0.95 (3H, s, CH₃-6), 0.98 (3H, s, CH₃-6), 1.24 (3H, d, J = 7.2, CH₃-11), 1.56 (3H, d, J = 6.6, CH₃-10), 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)

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 10 μM [74], antitumor activity [9], cytotoxic activity against cell lines P388, WEHI164, THP-1, and MOLT4 [14]



54. Inophyllum D

Calophyllum inophyllum [73, 74]

C₂₅H₂₄O₅, mp 200-202°C [73]

[α]_D +35° (CDCl₃) [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

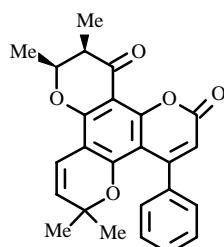
PMR (60 MHz, CDCl₃) [73]: 0.94 (6H, s, CH₃-6), 1.17 (3H, d, J = 7.2, CH₃-11), 1.43 (3H, d, J = 7.0, CH₃-10), 2.27 (1H, m, H-11), 4.43 (1H, m, J = 3.3, J = 7.0, H-10), 5.17 (1H, d, J = 5.4, H-12), 5.36 (1H, d, J = 10, H-7), 5.96 (1H, s, H-3), 6.55 (1H, d, J = 10, H-8), 7.3 (5H, m, Ph-4)

X-ray [76]

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 11 μM [9], antitumor activity [9]



55. Inophyllum E [(+)-cis-inophyllumide]

Calophyllum inophyllum [73, 74, 77],

C. teysmannii var. *inophyllumide* [80]

[α]_D +70° (CHCl₃) [73]

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

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

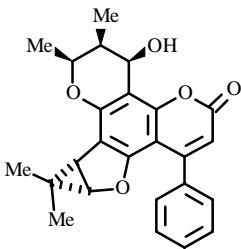
Mass [73]: 402 [M]⁺, 387 (15), 331 (56), 303 (28)

PMR (60 MHz, CDCl₃) [73]: 0.97 (6H, s, CH₃-6, CH₃-6), 1.18 (3H, d, J = 7.2, CH₃-11), 1.44 (3H, d, J = 6.8, CH₃-10), 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)

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 100 μM [74], antitumor activity [9]



56. Inophyllum G-1

Calophyllum inophyllum [74]

C₂₆H₂₆O₅

[α]_D +174.2° (CDCl₃) [74]

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

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

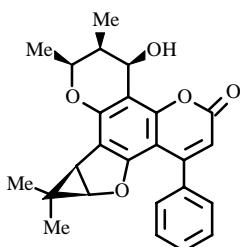
766, 736, 700

HRCIMS [74]: 404.1610

PMR (400 MHz, CDCl₃) [74]: 0.71 (3H, s, CH₃-20), 1.05 (3H, s, CH₃-19), 1.20 (3H, d, J = 7.1, CH₃-22), 1.45 (3H, d, J = 6.8, CH₃-21), 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)

¹³C NMR (100 MHz, CDCl₃) [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]

C₂₆H₂₆O₅

[α]_D -49.1° (CDCl₃) [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

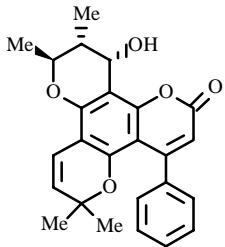
HRCIMS [74]: 404.1610

PMR (400 MHz, CDCl₃) [74]: 0.73 (s, 3H, CH₃-20), 1.05 (s, 3H, CH₃-19), 1.18 (d, J = 7.1, 3H, CH₃-22), 1.45 (d, J = 6.8, 3H, CH₃-21), 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)

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 70 μM [74]



58. Inophyllum P

Calophyllum inophyllum [74, 77]

C₂₅H₂₄O₅

[α]_D +19.8° (CDCl₃) [74], +31.4° [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]⁺

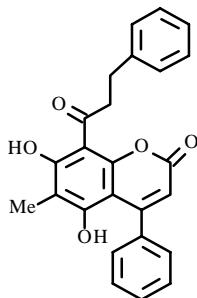
PMR (400 MHz, CDCl₃) [74]: 0.93 (6H, s, CH₃-19, CH₃-20), 1.17 (3H, d, J = 7.0, CH₃-22), 1.44 (3H, d, J = 6.3, CH₃-21), 1.79 (1H, ddq, J = 3.3, J = 7.0, J = 10.6, H-11), 4.29 (1H, dq, J = 6.3, J = 10.6, H-10), 5.04 (1H, d, J = 3.3, H-12), 5.37 (1H, d, J = 10.0, H-7), 5.97 (1H, s, H-3), 6.54 (1H, d, J = 10.0, H-8), 7.42, 7.30 (5H, m, Ph-4)

X-ray [76]

¹³C NMR (100 MHz, CDCl₃) [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 IC₅₀ 0.130 μM [74], activity against HIV-1 in cell culture at IC₅₀ 1.6 μM [74]



59. Interruptin A

Cyclosorus interruptus [81]

C₂₅H₂₀O₅, mp 143-145°C [81]

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

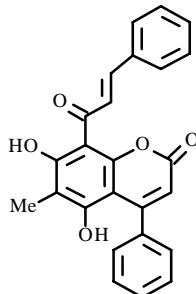
Mass [81]: 400 (36) [M]⁺, 382 (7), 295 (100), 91 (24)

PMR (300 MHz, CDCl₃) [81]: 2.28 (3H, s, Me-6), 2.96 (2H, t, J = 7.6, CH₂-3'), 3.29 (2H, t, J = 7.6, CH₂-2'), 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)

¹³C NMR (75 MHz, CDCl₃) [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

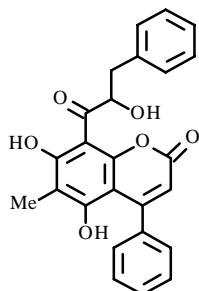
Mass [81]: 398 (11) $[M]^+$, 321 (8), 294 (4), 266 (3), 91 (4), 44 (100)

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)

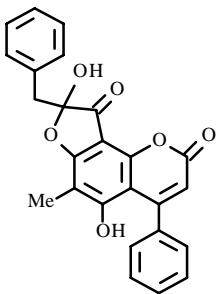
PMR (300 MHz, CD_3OD) [81]: 2.27 (3H, s, Me-6), 2.77 (1H, dd, J = 8.7, J = 13.8, H-3'b), 3.18 (1H, dd, J = 3.8, J = 13.8, H-3'a), 5.31 (1H, dd, J = 3.8, J = 8.7, H-2'), 5.91 (1H, s, H-3), 7.07-7.09 (2H, m, H-6', H-8'), 7.18-7.23 (1H, m, H-4''), 7.24-7.26 (3H, m, H-5', H-7', H-9'), 7.28-7.36 (2H, m, H-3'',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

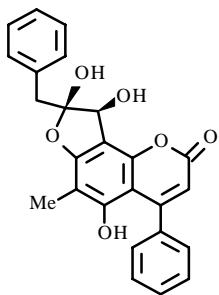
UV (MeOH) [81]: 207, 288, 372, 400

Mass [81]: 415 (100) $[M + H]^+$, 397 (5), 295 (20)

PMR (300 MHz, $CDCl_3$) [81]: 2.25 (3H, s, Me-6), 3.00 (2H, s, CH_2 -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'')

^{13}C NMR (75 MHz, CD_3OD) [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) $[M - H_2O]^+$, 321 (82), 294 (36)

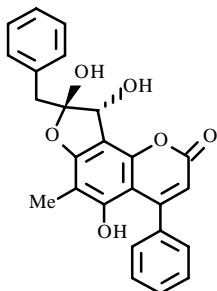
PMR (300 MHz, $DMSO-d_6$) [81]: 2.16 (3H, s, Me-6), 2.79 (1H, d, $J = 14.0$, H-3'a), 3.09 (1H, d, $J = 14.2$, H-3'b), 4.71 (1H, d, $J = 8.1$, H-1'), 5.34 (1H, d, $J = 8.6$, OH-1'), 5.90 (1H, s, H-3), 6.66 (1H, s, OH-2'), 7.05-7.12 (2H, m, H-5', H-9'), 7.07-7.47 (4H, m, H-2'', H-3'', H-5'', H-6''), 7.10-7.16 (1H, m, H-7'), 7.37-7.51 (3H, m, H-6', H-8', H-4'')

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

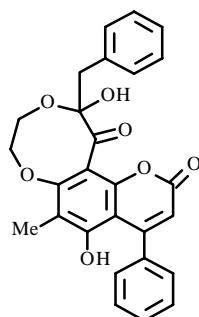
PMR (300 MHz, DMSO-d₆) [81]: 2.14 (3H, s, Me-6), 2.74 (1H, d, J = 13.8, H-3'a), 2.91 (1H, dd, J = 14.5, J = 0.6, H-3'b), 4.82 (1H, d, J = 5.0, H-1'), 5.44 (1H, d, J = 6.0, OH-1'), 5.87 (1H, s, H-3), 6.40 (1H, s, OH-2'), 7.05-7.12 (2H, m, H-5', H-9'), 7.07-7.47 (4H, m, H-2'', H-3'', H-5'', H-6'')

¹³C NMR (75 MHz, DMSO-d₆) [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

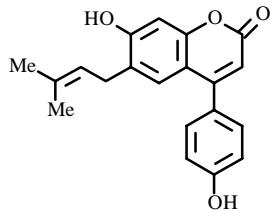
Mass [81]: 460 (6) $[M + 2H]^+$, 398 (66), 395 (100)

PMR (300 MHz, DMSO-d₆) [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, CH₂-11'), 3.49 (2H, t, J = 5.2, CH₂-10'), 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')

¹³C NMR (75 MHz, DMSO-d₆) [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		

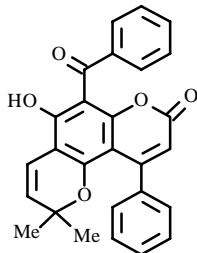
Biol: antibacterial activity against *Staphylococcus epidermidis* and *M. luteus* [81]



66. Inflacoumarin A

Glycyrrhiza inflata [82, 83]

C₂₀H₁₈O₄



67. Calanone

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

C₂₇H₂₀O₅

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]⁺

PMR (500 MHz, CDCl₃) [79]: 0.98 (6H, s, CH₃-12, CH₃-13), 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]

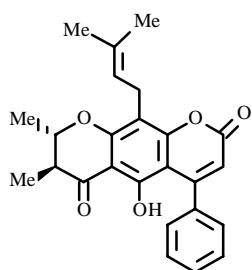
X-ray [71]

¹³C NMR (125 MHz, CDCl₃) [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 australianum [84]

C₂₅H₂₄O₅, mp 192-193.5°C [84]

UV (EtOH-OH) [84]: 286, 315, 425

IR [84]: 1718, 1645, 1613

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

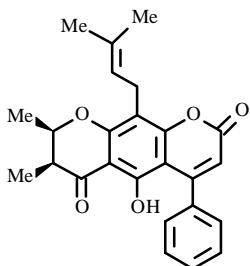
HREIMS [14]: 404.1629 [M]⁺

PMR (300 MHz, CDCl₃) [14]: 1.21 (3H, d, J = 7.1, CH₃-16), 1.56 (3H, d, J = 6.2, CH₃-17), 1.69 (3H, s, CH₃-15), 1.85 (3H, s, CH₃-14), 2.64 (1H, dq, J = 12.2, J = 7.1, H-7), 3.46 (2H, d, J = 7.5, CH₂-11), 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)

¹³C NMR (75 MHz, CDCl₃) [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]

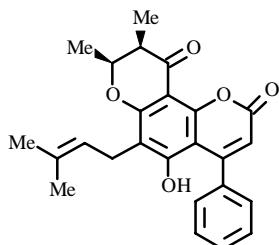
C₂₅H₂₄O₅, mp 197-198°C [85]

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) [M]⁺, 389 (56), 361 (100), 349 (20), 336 (16), 333 (24), 305 (36), 293 (16), 280 (12), 277 (14)

PMR (60 MHz, CDCl₃) [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)

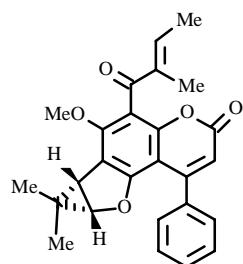


70. Calocoumarin A

Calophyllum inophyllum [86]

C₂₅H₂₄O₅

Biol: antitumor activity [86]

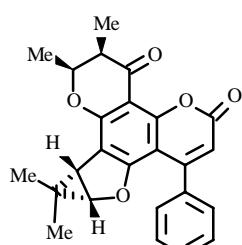


71. Calocoumarin B

Calophyllum inophyllum [86]

C₂₆H₂₅O₅

Biol: antitumor activity [86]

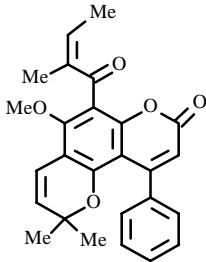


72. Calocoumarin C

Calophyllum inophyllum [86]

C₂₅H₂₂O₅

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)

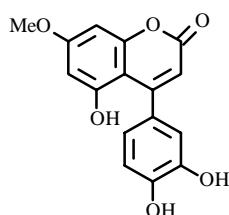
Mass [5]: 416 (31) $[M]^+$, 401 (100), 361 (12), 331 (13)

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+AlCl₃): 260, 329; (MeOH+AlCl₃+HCl): 260, 329; (MeOH+NaOAc):
274, 330, 362, 400; (MeOH+NaOAc+H₃BO₃): 260, 330, 400

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

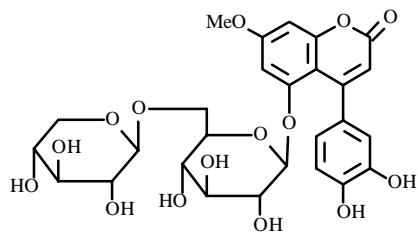
Mass [93]: 300 (100) $[M]^+$, 299 (13.4), 273 (16.2), 272 (93.6), 257 (22.5), 243 (7.3)

PMR (270 MHz, DMSO-d₆) [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, (CD₃)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-[β -D-Xylopyranosyl-(1 \rightarrow 6)- β -D-glucopyranosyl]-7-methoxy-3',4'-dihydroxy-4-phenylcoumarin

Coutarea hexandra [10]

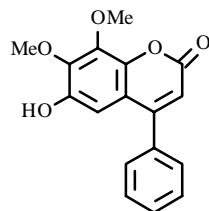
C₂₇H₃₀O₁₅

Mass [10]: 593 [M - H]⁻, 461, 299

PMR (250 MHz, CD₃OD) [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')

¹³C NMR (69.5 MHz, CD₃OD) [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],

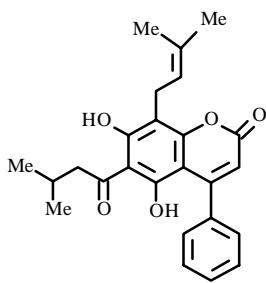
M. pedicellatum [58], *Piptadenia macrocarpa* [59]

C₁₇H₁₄O₅, **mp** 211°C [56]

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

IR (CHCl₃) [56]: 3500, 1710, 1555, 1395

PMR (60 MHz, CDCl₃) [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]

C₂₅H₂₆O₅, **mp** 83°C [99], 92-112°C [104], 97°C [103], 98-100°C [100],

98-109°C [97]

[α]_D +22° [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; (CDCl₃): 3436, 3289, 1730, 1621, 1587

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

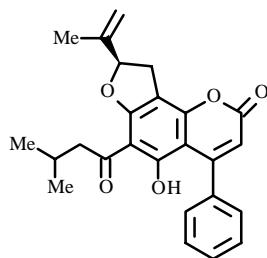
HRMS [105]: 406.1179 [M]⁺

PMR (250 MHz, CDCl₃) [105]: 0.9 (6H, d, J = 7, Me₂CH), 1.74 and 1.88 (6H, two s, Me₂C=CH), 2.16 (1H, m, J = 7, Me₂CHCH₂), 2.84 (2H, d, J = 7, CHCH₂CO), 3.52 (2H, d, J = 7, ArCH₂CH), 5.2 (1H, t, J = 7, Me₂C=CHCH₂), 5.88 (1H, s, PhC=CHCO), 7.3-7.6 (5H, m, ArH), 9.8 (1H, s, OH), 11.2 (1H, s, OH)

¹³C NMR (67.5 MHz, CDCl₃) [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 µg/mL [106]



78. Mammea A/AA dehydrocyclo F

Calophyllum dispar [66]

C₂₅H₂₄O₅

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 (CHCl₃) [66]: 3430, 3060, 2960, 1746, 1617, 768, 700

HREIMS [66]: 404.1637 [M]⁺

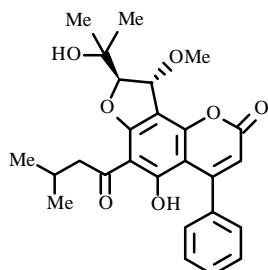
PMR (270 MHz, CDCl₃) [66]: 0.96 (6H, d, J = 6.5, CH₃-4'', CH₃-5''), 1.83 (3H, s, CH₃-6'''), 2.18 (1H, m, H-3''), 2.90 (1H, dd, J = 3.0, J = 7.0, CH₂-2''), 3.15 (1H, dd, J = 8.0, J = 15.0, CH₂-3''' α), 3.50 (1H, dd, J = 10.0, J = 15.0, CH₂-3''' β), 5.03 (1H, s, H-5'''b), 5.15 (1H, s, H-5'''a), 5.48 (1H, 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]

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 40 µg/mL [66]



79. Mammea A/AA methoxycyclo F

Calophyllum dispar [66]

C₂₆H₂₈O₇

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)

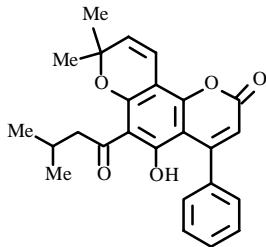
IR (CHCl₃) [66]: 3410, 1734, 1719, 1617, 768, 699

HRLSIMS [66]: 453.1920 [M + H]⁺

PMR (270 MHz, CDCl₃) [101]: 0.96 (6H, d, J = 7.0, CH₃-4'', CH₃-5''), 1.35 (1H, s, CH₃-5'''), 1.39 (1H, s, CH₃-6'''), 2.21 (1H, m, H-3''), 2.81 (1H, dd, J = 7.0, J = 15.0, CH₂-2''' α), 3.00 (1H, dd, J = 7.0, J = 15.0, CH₂-2''' β), 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]**¹³C NMR** (67.5 MHz, CDCl₃) [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]

C₂₅H₂₄O₅, 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

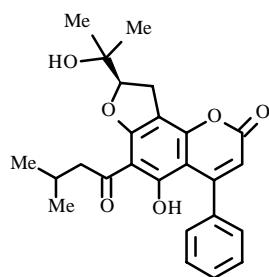
Mass [110]: 404 (34) [M]⁺, 389 (100), 371 (27), 347 (21), 105 (11), 77 (11), 42 (20)

PMR (300 MHz, CDCl₃) [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, CH₂-2''), 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)

¹³C NMR (75 MHz, CDCl₃) [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]

C₂₅H₂₆O₆, 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 (CDCl₃) [105]: 3050, 1720, 1610

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

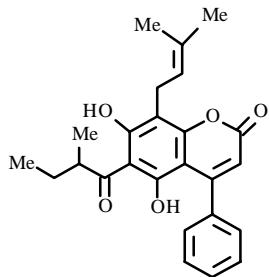
PMR (250 MHz, CDCl₃) [105]: 0.94 (6H, d, J = 7, Me₂CH), 1.28 and 1.48 (6H, two s, Me₂CCH), 2.04 (1H, s, Me₂COH), 2.14 (1H, m, Me₂CHCH₂), 2.84 (2H, dd, J = 7, J = 5, CHCH₂CO), 3.24 (2H, d, J = 9, ArCH₂CH), 4.80 (1H, t, J = 9,

$\text{Me}_2\text{CCHCH}_2$), 5.72 (1H, s, PhC=CHCO), 7.0-7.2 (5H, m, ArH), 15.0 (1H, s, OH)

^{13}C NMR (67.5 MHz, CDCl_3) [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_{50} 6 $\mu\text{g/mL}$ [66]



82. Mammea A/AB (MAB 1)

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

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

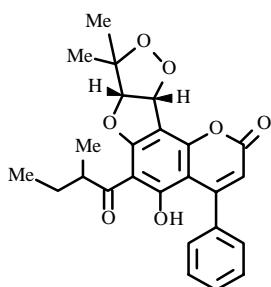
IR (CDCl_3) [105]: 3500, 2950, 1730, 1630

Mass [105]: 406 (52) [M^+], 351 (29), 349 (100), 294 (28)

HRMS [105]: 406.1776 [M^+]

PMR (250 MHz, CDCl_3) [105]: 0.88 (3H, t, $J = 7$, MeCH_2), 1.12 (3H, d, $J = 7$, MeCH), 1.2-1.8 (2H, m, MeCH_2CH), 1.80 and 1.94 (6H, two s, $\text{Me}_2\text{C=CH}$), 3.52-3.84 (3H, m, ArCH_2CH and CH_2CHCO), 5.4 (1H, t, $J = 7$, $\text{Me}_2\text{C=CHCH}_2$), 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 $\mu\text{g/mL}$ [106]



83. Mammea A/AB dioxalanocyclo F

Calophyllum dispar [67]

$\text{C}_{25}\text{H}_{24}\text{O}_7$

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_3) [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^+]

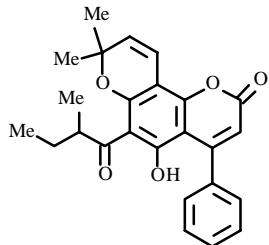
PMR (500 MHz, CDCl_3) [67]: 0.91 (3H, t, $J = 7.0$, $\text{CH}_3\text{-5''}$), 1.17 (3H, d, $J = 7.0$, $\text{CH}_3\text{-3''}$), 1.43 (1H, m, $\text{CH}_2\text{-4a''}$), 1.48 (3H, s, $\text{CH}_3\text{-4''}$), 1.50 (3H, s, $\text{CH}_3\text{-5''}$), 1.79 (1H, m, $\text{CH}_2\text{-4b''}$), 3.58 (1H, 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]

¹³C NMR (125 MHz, CDCl₃) [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. Mammea A/AB cyclo D (MAB 5)



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

C₂₅H₂₄O₅, 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 (CHCl₃) [111]: 3448, 1748, 1650, 1582, 1132, 1115, 699

Mass [111]: 404 (44) [M]⁺, 389 (100), 347 (86)

PMR (300 MHz, CDCl₃) [5]: 0.91 (3H, t, J = 7.4, MeCH₂CH), 1.17 (3H, d, J = 7.0, MeCHCO), 1.34-1.50 (1H, m, MeCH₂CH), 1.58 (3H, s, Me₂CH), 1.59 (3H, s, Me₂CH), 1.77-1.92 (1H, m, MeCH₂CH), 3.66-3.78 (1H, m, J = 7.0, MeCHCO), 5.64 (1H, d, J = 10.1, ArCH=CH), 6.00 (1H, s, C=CHCO), 6.91 (1H, d, J = 10.1, ArCH=CH), 7.30-7.45 (5H, m, Ph), 14.70 (1H, s, OH)

¹³C NMR (75 MHz, CDCl₃) [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. Mammea A/AB cyclo E

Calophyllum dispar [67]

C₂₅H₂₆O₆

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)

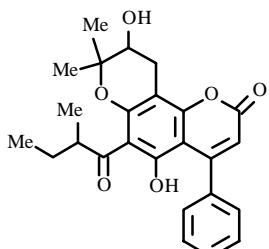
IR (CHCl₃) [67]: 3400, 1717, 1617, 1561, 768, 704

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 [M]⁺

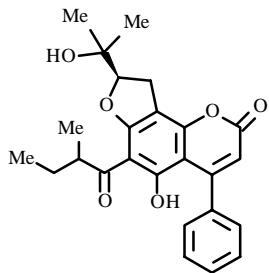
PMR (500 MHz, CDCl₃) [67]: 0.93 (3H, t, J = 7.0, CH₃-5''), 1.16 (3H, d, J = 6.5, CH₃-3''), 1.36 (6H, s, CH₃-4''), CH₃-5''), 1.41 (1H, m, CH₂-4a''), 1.81 (1H, m, CH₂-4b''), 3.35 (2H, m, CH₂-1''), 3.64 (1H, m, H-2''), 5.25 (1H, d, J = 8.5, H-2''), 5.96 (1H, s, H-3), 7.33 (m, 2H, H-2', H-6'), 7.41 (m, 3H, H-3', H-4', H-5'), 14.67 (s, 1H, OH-7)

HMBC [67]



¹³C NMR (125 MHz, CDCl₃) [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. Mammea A/AB cyclo F (MAB 3)

Mammea americana [113, 114], *M. africana* [98, 99],
Kielmeyera pumila [110], *Calophyllum dispar* [66]
C₂₅H₂₆O₆, mp 115-117°C [114], 134°C [99], 140-142°C [105]

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

IR (CDCl₃) [105]: 2950, 1720, 1600

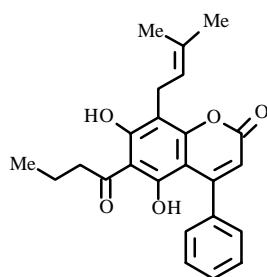
Mass [105]: 422 (100) [M]⁺, 365 (72), 364 (12), 363 (14), 347 (23),
307 (10), 293 (72)

PMR (250 MHz, CDCl₃) [105]: 0.88 (3H, t, J = 7, MeCH₂), 1.12 (3H, dd, J = 7, J = 2, MeCH), 1.28 and 1.38 (6H, two s, Me₂CCH), 1.2-1.9 (2H, m, MeCH₂CH), 1.80 (1H, s, Me₂COH), 3.24 (2H, d, J = 9, ArCH₂CH), 3.54 (1H, m, CH₂CHCO), 4.82 (1H, t, J = 9, Me₂CCHCH₂), 5.80 (1H, s, PhC=CHCO), 7.0-7.4 (5H, m, ArH), 14.28 (1H, s, OH)

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 5 µg/mL [66]



87. Mammea A/AC

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

C₂₄H₂₄O₅, mp 116-117°C [117]

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

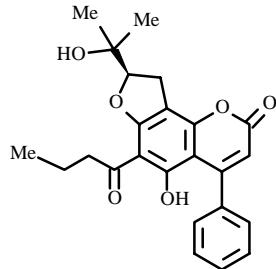
IR (mineral oil) [117]: 3340, 1700, 1620, 1580

Mass [117]: 392 (90) [M]⁺, 377 (16), 349 (66), 337 (100)

PMR (60 MHz, CDCl₃) [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)

¹³C NMR (67.5 MHz, CDCl₃) [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. Mammea A/AC cyclo F

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

Calophyllum dispar [66]

C₂₄H₂₄O₆, mp 119 °C [101]

[α]_D -2.6° (CHCl₃) [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) [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

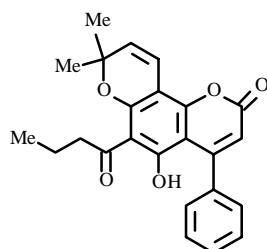
PMR (270 MHz, CDCl₃) [101]: 0.99 (3H, t, J = 7.5, CH₃-4''), 1.32 (3H, s, CH₃-2'''), 1.44 (3H, s, CH₃-3'''), 1.68 (2H, m, CH₂-3''), 3.00 (2H, m, CH₂-2''), 3.32 (2H, dd, J = 6.0, J = 3.0, CH₂-9), 4.92 (1H, t, J = 9.0, H-8), 5.95 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 14.39 (1H, s, OH-5)

HMBC, HMQC [101]

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 6 µg/mL [66]



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

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

Mammea siamensis [118]

C₂₄H₂₂O₅, mp 138-139 °C [117]

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

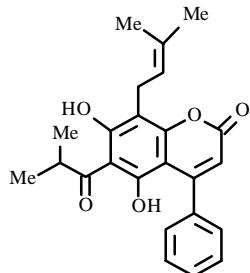
IR (mineral oil) [117]: 1725, 1630, 1600, 1575

Mass [117]: 390 (44) [M]⁺, 375 (100), 357 (16), 347 (14)

PMR (60 MHz, CDCl₃) [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)

¹³C NMR (67.5 MHz, CDCl₃) [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. Mammea A/AD (mesuol)

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

C₂₄H₂₄O₅, mp 146-148°C [105], 154°C [119], 183-184°C [104]
[α]_D +66° [104]

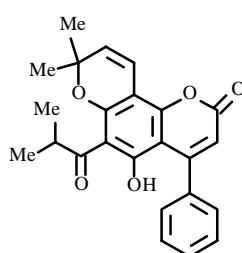
UV (EtOH+KOH) [119]: 234 (4.33), 303 (4.21), 394 (3.84), 426 (4.03)

IR (CDCl₃) [105]: 3450, 1730, 1630

Mass [105]: 392 (35) [M]⁺, 349 (75), 337 (18), 293 (100)

HRMS [105]: 392.1614 [M]⁺

PMR (250 MHz, CDCl₃) [105]: 1.10 (6H, d, J = 7, Me₂CH), 1.74 and 1.88 (6H, two s, Me₂C=CH), 3.54 (2H, d, J = 7, ArCH₂CH), 3.72 (1H, m, J = 7, Me₂CHCO), 5.22 (1H, t, J = 7, Me₂C=CHCH₂), 5.90 (1H, s, PhC=CHCO), 7.2-7.5 (5H, m, ArH), 9.84 (1H, s, OH), 10.90 (1H, s, OH)



91. Mammea A/AD cyclo D (mesuagin)

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

C₂₄H₂₂O₅, mp 151-153°C [104], 152-153°C [109]

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

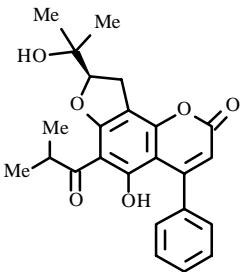
IR (KBr) [109]: 3400, 1739, 1653, 1613, 1378, 709

PMR (CDCl₃) [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)

¹³C NMR (67.5 MHz, CDCl₃) [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

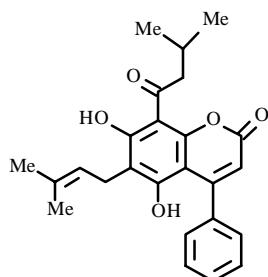
Biol: antibacterial activity against *Staphylococcus aureus* [109]



92. Mammea A/AD cyclo F

Mammea americana [114]

C₂₄H₂₄O₆



93. Mammea A/BA

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

C₂₅H₂₆O₅, mp 125–126°C [115]

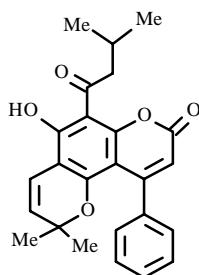
UV (EtOH) [116]: 227 (4.43), 234 (4.43), 270 (4.17), 297 (4.24), 335 (4.37); (EtOH+HCl): 225 (4.46), 294 (4.36), 332 (4.25); (EtOH+KOH): 233 (4.40), 261 (4.19), 337 (4.60)

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

Mass [116]: 406 [M]⁺, 363, 351, 349, 293, 265

PMR [116]: 1.04 (6H, d, J = 7.0, two CH₃-3'), 1.63 (3H, s, CH₃-3''), 1.68 (3H, s, CH₃-3''), 2.2 (1H, m, H-3'), 3.08 (2H, d, J = 7.0, CH₂-2'), 3.19 (2H, d, J = 7.0, CH₂-1''), 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]

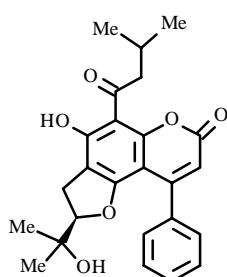
C₂₅H₂₄O₅, mp 173–175°C [110]

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]⁺, 389 (100), 371 (11), 347 (10)

PMR (100 MHz, CDCl₃) [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, CH₂-2'), 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]

C₂₅H₂₄O₅, 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 (CHCl₃) [66]: 3470, 1725, 1605, 1561, 756, 702

HRFABMS [66]: 421.1674 [M - H]⁻

PMR (270 MHz, CDCl₃) [66]: 0.94 (s, 3H, CH₃-6''), 1.02 (s, 3H, CH₃-5''), 1.08 (d, J = 6.5, 6H, CH₃-4'', CH₃-5''), 2.32 (m, 1H, H-3''), 2.93 (dd, J = 8.5, J = 15.5, 1H, CH₂-3''β), 3.08 (dd, J = 10.0, J = 15.5, 1H, CH₂-3''α), 3.19 (d, J = 7.0, 2H, CH₂-2''), 4.52 (t, J = 9.0, 1H, H-2''), 6.08 (s, 1H, H-3), 7.32 (m, 2H, H-2',

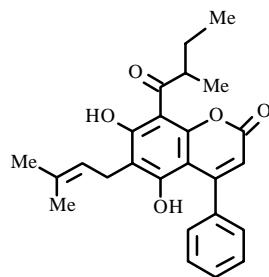
H-6'), 7.44 (m, 3H, H-3', H-4', H-5'), 14.33 (s, 1H, OH-7)

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

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 9 µg/mL [66]



96. Mammea A/BB

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

C₂₅H₂₆O₅, 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

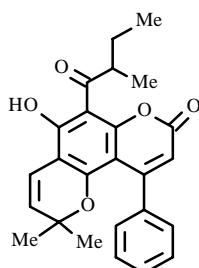
Mass [116]: 406 [M]⁺, 363, 351, 349, 293

PMR [116]: 1.02 (t, J = 7.0, 3H, CH₃-4'), 1.26 (d, J = 7.0, 3H, CH₃-2'), 1.60 (m, 2H, CH₂-3'), 1.64 (s, 3H, CH₃-3''), 1.70 (s, 3H, CH₃-3''), 3.20 (d, J = 7.0, 2H, CH₂-1''), 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)

¹³C NMR (67.5 MHz, CDCl₃) [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)

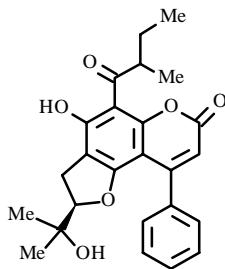
Mammea americana [105], *Calophyllum inophyllum* [87]

C₂₅H₂₄O₅, mp 126-127°C [5], 127-129°C [105]

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

IR (CDCl₃) [105]: 1730, 1640, 1600

PMR (250 MHz, CDCl₃) [105]: 1.0 (6H, s, Me₂C), 1.04 (3H, t, J = 7, MeCH₂), 1.32 (3H, d, J = 7, MeCH), 1.3-2.1 (2H, m, MeCH₂CH), 4.0 (1H, m, CH₂CHCO), 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]

C₂₅H₂₄O₅

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 (CHCl₃) [66]: 3460, 1732, 1607, 1559, 758, 704

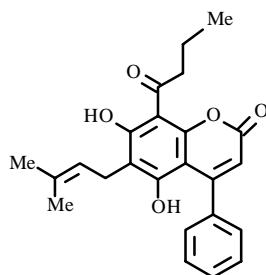
HRFABMS [66]: 421.1639 [M - H]⁻

PMR (270 MHz, CDCl₃) [66]: 0.94 (3H, s, CH₃-6''), 1.01 (3H, t, J = 7.0, CH₃-5'''), 1.02 (3H, s, CH₃-5''), 1.28 (3H, d, J = 7.0, CH₃-3'''), 1.50 (1H, m, CH₂-4''a), 1.94 (1H, m, CH₂-4''b), 2.93 (1H, dd, J = 8.0, J = 15.0, CH₂-3''β), 3.07 (1H, dd, J = 10.0, J = 15.0, CH₂-3''α), 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')

¹³C NMR (67.5 MHz, CDCl₃) [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 ED₅₀ 15 µg/mL [64]



99. Mammea A/BC

Mesua racemosa [72]

C₂₄H₂₄O₅, 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

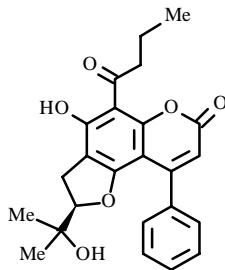
HRLSIMS [72]: 391.1526 [M - H]⁺

PMR (270 MHz, CDCl₃) [72]: 1.03 (3H, t, J = 7.0, CH₃-4''), 1.66 (3H, s, CH₃-4''), 1.71 (3H, s, CH₃-5''), 1.83 (2H, m, CH₂-3''), 3.30 (2H, m, CH₂-2''), 3.33 (2H, m, CH₂-1''), 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')

HMBC [72]

¹³C NMR (67.5 MHz, CDCl₃) [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. Mammea A/BC cyclo F

Calophyllum dispar [66]

C₂₄H₂₄O₆

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)

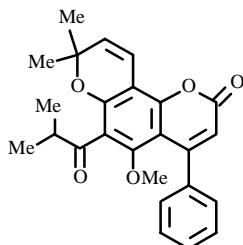
IR (CHCl₃) [66]: 3460, 1732, 1607, 1559, 758, 704

HRFABMS [66]: 407.525 [M - H]⁻

PMR (270 MHz, CDCl₃) [66]: 0.94 (3H, s, CH₃-6''), 1.01 (3H, t, J = 7.0, CH₃-5''), 1.02 (3H, s, CH₃-5''), 1.28 (3H, d, J = 7.0, CH₃-3'''), 1.50 (1H, m, CH₂-4''a), 1.94 (1H, m, CH₂-4''b), 2.93 (1H, dd, J = 8.0, J = 15.0, CH₂-3''β), 3.08 (1H, dd, J = 10.0, J = 15.0, CH₂-3''α), 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')

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₅H₂₄O₅, **mp** 130°C [120]

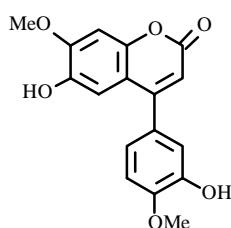
UV (EtOH) [120]: 233 (4.63), 282 (4.35), 360 (4.1)

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

Mass [120]: 420 [M]⁺, 405, 377

PMR (90 MHz, DMSO-d₆) [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]

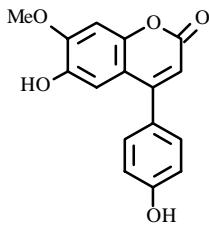
C₁₇H₁₄O₆, **mp** 220.5-222°C [43], 221-223°C [52, 53]

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-d₆) [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 melanoxylon [122], *D. odorifera* [30, 31]

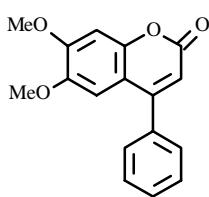
C₁₆H₁₂O₅, mp 233-234°C [123], 236-237°C [124]

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

IR (KBr) [123]: 3430, 1660

¹³C NMR (75.4 MHz, DMSO-d₆) [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],

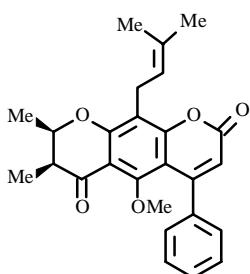
Machaerium scleroxylon [57], *M. pedicellatum* [58], *Prosopis kuntzei* [60],

Goniorrhachis marginata [61]

C₁₇H₁₄O₄, mp 145-146°C [40], 146-147°C [16]

IR (KBr) [54]: 1727; (CHCl₃): 1709

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



105. O-methylisocalaustralin

Calophyllum teysmanii var. *inophyllolide* [14]

C₂₆H₂₆O₅

[α]_D -10.8° (CHCl₃) [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]⁺, 403 (80), 363 (70), 349 (80), 319 (90), 307 (80)

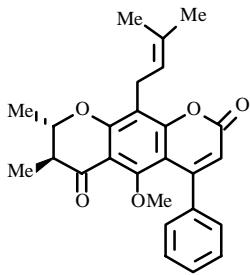
HREIMS [14]: 418.1763 [M]⁺

PMR (300 MHz, CDCl₃) [14]: 1.13 (3H, d, J = 7.2, CH₃-16), 1.41 (3H, d, J = 6.6, CH₃-17), 1.71 (3H, s, CH₃-15), 1.87 (3H, s, CH₃-14), 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, CH₂-11), 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]

¹³C NMR (75 MHz, CDCl₃) [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]

C₂₆H₂₆O₅

[α]_D -18° (CHCl₃) [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)

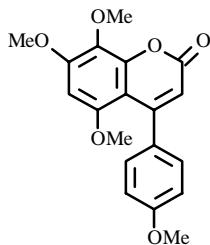
HREIMS [14]: 418.1807 [M]⁺

PMR (300 MHz, CDCl₃) [14]: 1.18 (3H, d, J = 7.1, CH₃-16), 1.55 (3H, d, J = 6.3, CH₃-17), 1.71 (3H, s, CH₃-15), 1.88 (3H, s, CH₃-14), 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, CH₂-11), 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]

¹³C NMR (75 MHz, CDCl₃) [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

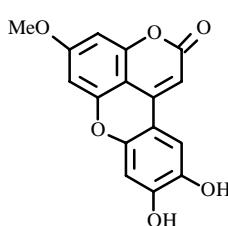
Coutarea hexandra [25], *Exostema mexicanum* [26]

C₁₉H₁₈O₆, mp 143-145°C [127]

IR (KBr) [127]: 1722

PMR (200 MHz, CDCl₃) [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 IC₅₀ = 13.3×10⁻⁵ 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]

C₁₆H₁₀O₆, mp 300°C (dec.) [11], 342°C (dec.) [19], 342-345°C [128]

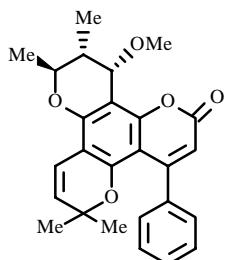
UV (MeOH) [19]: 260, 310, 375, 390; (MeOH+AlCl₃) [19]: 275, 325, 406; (MeOH+AlCl₃+HCl) [19]: 260, 310, 375, 390

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

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

PMR (DMSO-d₆) [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')

¹³C NMR (DMSO-d₆) [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]

C₂₆H₂₆O₅

[α]_D +25.5° (CHCl₃) [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)

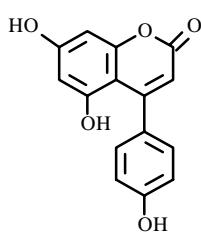
HREIMS [20]: 418.1783 [M]⁺

PMR (500 MHz, CDCl₃) [20]: 0.92 (3H, s, CH₃-14), 0.94 (3H, s, CH₃-13), 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')

¹³C NMR (125 MHz, CDCl₃) [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]

C₁₅H₁₀O₅, 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)

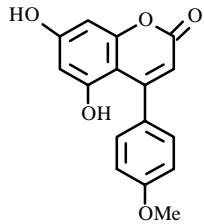
IR (KBr) [130]: 3300, 1660, 1610, 1550, 1500, 1445, 1355, 1249, 1175, 825

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-d₆) [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)

¹³C NMR (22.5 MHz, DMSO-d₆) [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]

C₁₆H₁₂O₅, 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)

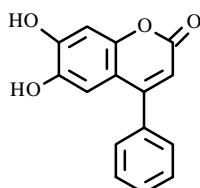
IR (KBr) [130]: 3100-3400, 2870, 1665, 1615, 1560, 1510, 1370, 1250, 1200, 848

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-d₆) [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)

¹³C NMR (22.5 MHz, DMSO-d₆) [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]

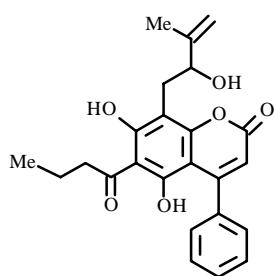
C₁₅H₁₀O₄, mp 260-261°C [133], 265°C [63], 268-269°C [40, 125], 274-276°C [134]

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]

C₂₄H₂₄O₆, 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)

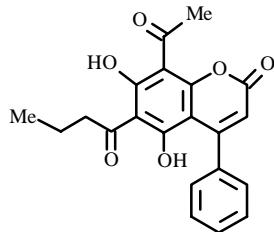
HREIMS [101]: 408.1591 [M]⁺

PMR (270 MHz, CDCl₃) [101]: 0.98 (3H, t, J = 7.5, CH₃-4''), 1.70 (2H, m, CH₂-3''), 1.93 (3H, s, CH₃-5'''), 3.04 (1H, dd, J = 7.5, J = 15.0, CH₂-1b''), 3.13 (2H, t, J = 7.5, CH₂-2''), 3.30 (1H, d, J = 13.5, CH₂-1a''), 4.50 (1H, d, J = 7.5, H-2''), 4.92 (1H, s, CH₂-4b''), 5.03 (1H, s, CH₂-4a''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₂₁H₁₈O₆, 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]⁺

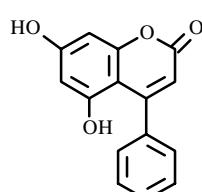
PMR (270 MHz, CDCl₃) [72]: 0.98 (2H, t, J = 7.5, CH₃-4''), 1.72 (2H, m, CH₂-3''), 2.98 (3H, s, CH₃-2''), 3.15 (2H, t, J = 7.5, CH₂-2''), 6.05 (1H, s, H-3), 7.30 (2H, m, H-2', H-6'), 7.40 (3H, m, H-3', H-4', H-5'), 16.08 (1H, s, OH-5), 16.59 (1H, s, OH-7)

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						

¹³C NMR (67.5 MHz, CDCl₃) [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. Serratin

Passiflora serratodigitata [36]

C₁₅H₁₀O₄, 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

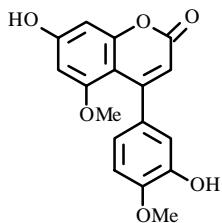
Mass [36]: 254 (100) [M]⁺, 226 (95), 197 (47)

PMR (90 MHz, DMSO-d₆) [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)

¹³C NMR (22.61 MHz, DMSO-d₆) [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 IC₅₀ 7.0×10⁻⁵ M [34]



116. Seshadrin

Dalbergia volubilis [16, 135], *Exostema mexicanum* [26]

C₁₇H₁₄O₆, mp 256-257°C [136]

UV (MeOH) [16]: 232 (4.2), 256 (4.0), 308 (3.8), 344 (4.1)

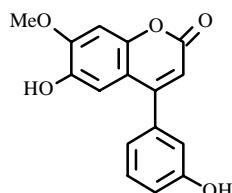
IR (KBr) [16]: 3340, 3050, 1710, 1615, 1600, 1590, 1450, 1330, 1105, 1080

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, CDCl₃) [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]

C₁₆H₁₂O₅, 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)

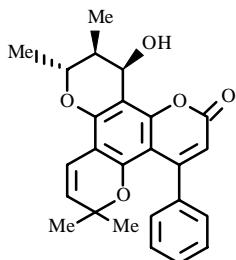
IR (CDCl₃) [54]: 3289, 1707, 1667, 1621

Mass [54]: 284 (100) [M]⁺, 256, 241

PMR (60 MHz, DMSO-d₆) [54]: 3.92 (3H, s, OMe), 6.5 (1H, s, H-3), 6.9-7.53 (6H, m), 9.52 (2H, s)

¹³C NMR (75.4 MHz, DMSO-d₆) [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]

C₂₅H₂₄O₅, mp 198-200°C [75], 201-202°C [138]

[α]_D -21.8° (CHCl₃) [75], -29.6° (CHCl₃) [138], -30.2° (CHCl₃) [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,

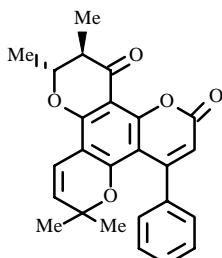
1150, 1140, 1130, 1115, 1062, 1022, 995, 965, 930, 880, 850, 795, 772, 760, 710, 700

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_3) [75]: 0.93 (6H, s, CH_3 -10, CH_3 -10), 1.18 (3H, d, J = 6.77, CH_3 -3), 1.45 (3H, d, J = 6.30, CH_3 -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_{50} 0.34 μM [140]



119. Soulattrolone [(-)-*trans*-inophyllolide]

Calophyllum teysmannii var. *inophyllolide* [79]

$\text{C}_{25}\text{H}_{22}\text{O}_5$, **mp** 187-190°C [138]

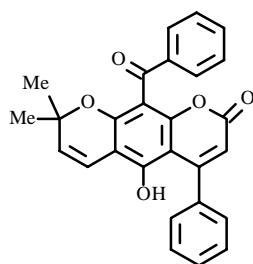
$[\alpha]_D$ -45.9° (CHCl_3) [138]

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^+], 387 (100), 353 (2), 346 (3), 331 (70), 263 (3), 229 (2), 219 (2), 189 (2), 105 (2), 77 (2)

PMR (100 MHz, CDCl_3) [138]: 0.98 (6H, s, CH_3 -6), 1.24 (3H, d, J = 7.2, CH_3 -11), 1.54 (3H, d, J = 6.4, CH_3 -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]

$\text{C}_{27}\text{H}_{20}\text{O}_5$, **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^+], 409 (100), 331 (42), 105 (70), 87 (50), 43 (10)

HREIMS [80]: 424.1340 [M^+]

PMR (500 MHz, CDCl_3) [80]: 1.27 (6H, s, CH_3 -12, CH_3 -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'')

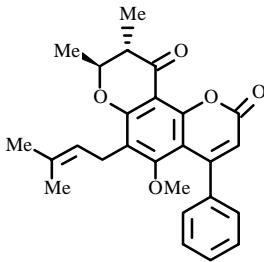
HMQC, HMBC [80]

X-ray [71]

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

C₂₆H₂₆O₅

[α]_D +40.5° (CHCl₃) [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, CDCl₃) [80]: 1.24 (3H, d, J = 7.1, CH₃-12), 1.55 (3H, d,

J = 6.5, CH₃-11), 1.67 (3H, s, CH₃-5''), 1.71 (3H, s, CH₃-5''), 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, CH₂-1''), 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')

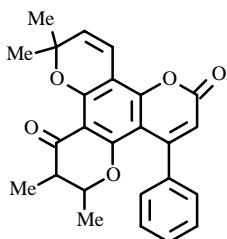
Mass [80]: 418 (100) [M]⁺, 403 (70), 387 (10), 363 (25), 349 (60), 335 (35), 319 (80), 307 (40), 105 (42), 55 (45), 43 (70)

HREIMS [80]: 418.1806 [M]⁺

¹³C NMR (125 MHz, CDCl₃) [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]

C₂₅H₂₂O₅, **mp** 201-205°C [7]

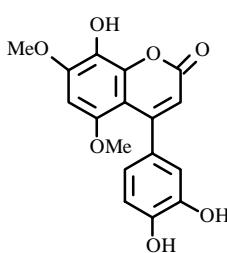
UV [7]: 237 (4.63), 275-280 (4.59), 350 (3.9)

IR [7]: 1740, 1690, 753, 699

Mass [7]: 402, 387, 331, 303

PMR (CDCl₃) [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

Coutarea hexandra [33, 141], *Exostema mexicanum* [26]

C₁₇H₁₄O₇, **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

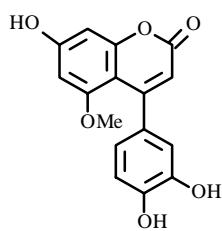
PMR (200 MHz, CD₃OD) [141]: 3.50 (3H, s, OMe-5), 3.98 (3H, s, OMe-7), 5.90 (1H, s, H-3), 6.57 (1H, s, H-6), 6.66 (1H, dd, J = 2, J = 7.5, H-6'), 6.75 (1H, d, J = 2, H-2'), 6.81 (1H, d, J = 7.5, H-5')

¹³C NMR (69.5 MHz, CD₃OD) [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 IC₅₀ 3.7×10⁻⁵ M [34], antiplasmodial activity against *Plasmodium falciparum* [26]



124. 7,3',4'-Trihydroxy-5-methoxyneoflavone

Coutarea hexandra [142]

C₁₆H₁₂O₆, mp 269–270°C [142]

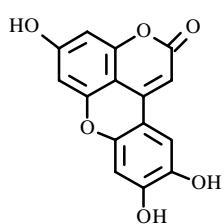
UV (MeOH) [142]: 259 (4.43), 332 (4.77); (MeOH+NaOAc): 257, 272, 332, 378; (MeOH+NaOMe): 245, 271, 381

Mass [142]: 300 (100) [M]⁺, 272 (100), 257 (12), 239 (30), 229 (8), 211 (12), 150 (6), 136 (4)

PMR [(CD₃)₂CO] [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')

¹³C NMR [(CD₃)₂CO] [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 carabaeum [19]

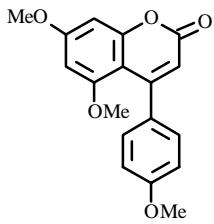
C₁₅H₈O₆, mp 350°C (dec.) [19]

UV (MeOH) [19]: 262, 310, 350, 372, 390; (MeOH+AlCl₃): 325, 404; (MeOH+AlCl₃+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, CDCl₃/DMSO-d₆) [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]

C₁₈H₁₆O₅, mp 150–152°C [22], 151–152°C [24], 152°C [143]

UV (CHCl₃) [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, CDCl₃) [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')

¹³C NMR (125.7 MHz, CDCl₃) [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⁺, KB-V⁻, LNCaP, SW626, SKNSH, and M109 [22]

127. 4-Phenyl-5-methoxy-7-hydroxy-8-benzoylcoumarin

Calophyllum teysmannii var. *inophylloide* [20]

C₂₃H₁₆O₅, 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

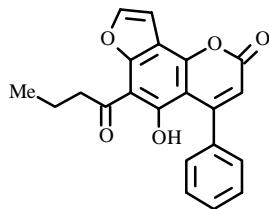
PMR (500 MHz, CDCl₃) [20]: 3.50 (3H, s, MeO-5), 5.93 (1H, s, H-3), 6.33 (1H, s, H-6), 7.24 (2H, m, H-2', H-6'), 7.38 (3H, m, H-3', H-4', H-5'), 7.48 (2H, t, J = 8.0, H-3'', H-5''), 7.60 (1H, tt, J = 8.0, J = 1.6, H-4''), 7.65 (2H, dd, J = 8.0, J = 1.6, H-2'', H-6''), 12.30 (s, 1H, OH-7)

HMBC, NOE [20]

¹³C NMR (125 MHz, CDCl₃) [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]

C₂₁H₁₆O₅

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)

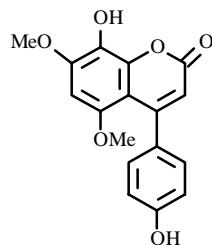
HREIMS [72]: 348.0973 [M]⁺

PMR (270 MHz, CDCl₃) [72]: 1.05 (3H, t, J = 7.0, CH₃-4''), 1.79 (2H, m, CH₂-3''), 3.25 (2H, t, J = 7.0, CH₂-2''), 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]

¹³C NMR (67.5 MHz, CDCl₃) [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]

C₁₇H₁₄O₆

Mass [26]: 314 [M]⁺, 299, 286, 271, 267, 243, 228

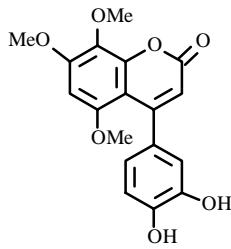
HRMS [26]: 314.07916

PMR (400 MHz, CD₃COCD₃) [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)

¹³C NMR (100 MHz, CD₃OD) [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]

C₁₈H₁₆O₆

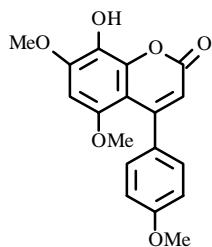
Mass [26]: 344 [M]⁺, 329, 316, 301, 273

HRMS [26]: 344.08948

PMR (400 MHz, CD₃COCD₃) [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]

C₁₈H₁₆O₆, **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, CDCl₃) [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')

¹³C NMR (125.7 MHz, DMSO-d₆) [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 IC₅₀ 10.4×10⁻⁵ M [34], cytotoxic activity against cell lines BC1, Lu1, Col2, KB, KB-V⁺, KB-V⁻, 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 (1) [6, 7]
<i>C. australianum</i>	Calaustralin (68) [84]
<i>C. bracteatum</i>	Calophyllolide (73) [90]
<i>C. dispar</i>	Disparacetylfuran A (38) [66], dispardiol B (39) [67], disparinol A (40) [68], disparinol B (41) [68], disparinol D (42) [67], disparifuran B (43) [66], isodispar B (45) [67], isodisparinol A (46) [68], isodisparinol B (47) [68], isodisparifuran A (48) [66], mammea A/AA dehydrocyclo F (78) [66], mammea A/AA methoxycyclo F (79) [66], mammea A/AA cyclo F (81) [66], mammea A/AB dioxalanocyclo F (83) [67], mammea A/AB cyclo E (85) [67], mammea A/AB cyclo F (86) [66], mammea A/AC cyclo F (88) [66], mammea A/BA cyclo F (95) [66], mammea A/BB cyclo F (98) [66], mammea A/BC cyclo F (100) [66]
<i>C. inophyllum</i>	Acetatolide (1) [8], inophyllum A (51) [73, 74], inophyllum B (52) [73, 74, 77], inophyllum C (53) [73, 74, 77, 78], inophyllum D (54) [73, 74], inophyllum E (55) [73, 74, 77], inophyllum G-1 (56) [74], inophyllum G-2 (57) [74], inophyllum P (58) [58], (-)-calaustralin (69) [85], calocoumarin A (70) [86], calocoumarin B (71) [86], calocoumarin C (72) [86], calophyllolide (73) [1, 77, 87, 88, 89], mammea A/BB cyclo D (97) [87]
<i>C. moonii</i>	Inophyllum A (51) [75], soulatrolide (118) [75]
<i>C. aff. Pervillei</i>	Calophyllolide (73) [91]
<i>C. soulattri</i>	Soulatrolide (118) [138]
<i>C. teysmannii</i> var <i>inophylloide</i>	9-Benzoyl-6-hydroxy-5-methoxy-4-phenylnodakenetin (5) [14], (-)-6-benzoyl-5-hydroxy-4-phenylcolumbianelin (6) [14], 9-benzoyl-5-methoxy-4-phenylnodakenetin (7) [14], hydrohydroxyisocalanone (14) [20], <i>trans</i> -7,8-dihydroxycalanone (32) [14], isocalanone (49) [14, 70], inophyllum C (53) [14, 79, 80], inophyllum E (55) [80], calanone (67) [14, 20, 79, 80], O-methylisocalaustralin (105) [14], O-methylcalaustralin (106) [14], 12-methoxyinophyllum P (109) [20], soulatrolide (118) [79, 139], soulatrolone (119) [79], teysmanone A (120) [80], teysmanone B (121) [80], 4-phenyl-5-methoxy-7-hydroxy-8-benzoylcoumarin (127) [20]
<i>C. tomentosum</i>	Tomentolide A (122) [7]
<i>Kayea assamica</i>	Mammea A/AA (77) [103], mammea A/AA cyclo F (81) [103]
<i>Kielmeyera argentea</i>	Coumarins 23 [35] and 24 [35]
<i>K. elata</i>	Hydroxymammeigin (18) [29], mammea A/AA (77) [29], mammea A/AA cyclo D (80) [29], mammea A/AA cyclo F (81) [29]
<i>K. lathrophyton</i>	Mammea A/AA cyclo D (80) [111], mammea A/AB cyclo D (84) [111]
<i>K. pumila</i>	Mammea A/AA cyclo D (80) [110], mammea A/AB cyclo F (86) [110], mammea A/BA cyclo D (94) [110]
<i>K. reticulata</i>	Coumarins 15 [21], 23 [21], 24 [21], and 29 [21]
<i>Mammea africana</i>	Mammea A/AA (77) [98, 99], mammea A/AB (82) [98, 99], mammea A/AB cyclo D (84) [98, 99], mammea A/AB cyclo F (86) [98, 99], mammea A/AD (90) [6, 104], mammea A/BA (93) [116]
<i>M. americana</i>	Mammea A/AA (77) [96, 97], mammea A/AA cyclo D (80) [107, 108], mammea A/AA cyclo F (81) [113], mammea A/AB (82) [115, 116], mammea A/AB cyclo F (86) [113, 114], mammea A/AC cyclo F (88) [114], mammea A/AD (90) [119], mammea A/AD cyclo D (91) [6], mammea A/AD cyclo F (92) [114], mammea A/BA (93) [115, 116], mammea A/BB (96) [115, 116], mammea A/BB cyclo D (97) [105]
<i>M. siamensis</i>	Mammea A/AC cyclo D (89) [118]
<i>Mesua ferrea</i>	Coumarins 20 and 35 [32], mammea A/AA (77) [100], mammea A/AA cyclo D (80) [104, 109], mammea A/AD (90) [104], mammea A/AD cyclo D (91) [109], mesuarin (101) [120]
<i>M. racemosa</i>	Isoracemosol (50) [72], mammea A/AA (77) [101], mammea A/AC (87) [101], mammea A/AC cyclo F (88) [101], mammea A/AC cyclo D (89) [101], mammea A/AD cyclo D (91) [101], mammea A/BB (96) [101], mammea A/BC (99) [72], racemosol (113) [101], racemosone (114) [72], furanoracemosol (128) [72]
<i>M. thwaitesii</i>	Mammea A/AA (77) [102], mammea A/AA cyclo D (80) [102], mammea A/AA cyclo F (81) [102], mammea A/AD (90) [102], mammea A/AD cyclo D (91) [102]
<i>Ochrocarpus siamensis</i>	Mammea A/AC (87) [117], mammea A/AC cyclo D (89) [117]

TABLE 1. (continued)

Plant	Isolated neoflavones
<u>Rubiaceae family</u>	
<i>Chiococca alba</i>	Coumarin 126 [143], exostemin (131) [143]
<i>Coutarea hexandra</i>	Coutareagenin (74) [92, 93], coumarins 2 [10], 17 [24, 25], 21 [33], 22 [24], 26 [10], 27 [10], 31 [24, 64], 33 [33], 37 [65], 75 [10], 123 [33, 141], 124 [142], and 126 [24], 8-O-methylexostemin (107) [25], exostemin (131) [25]
<i>Exostema acuminatum</i>	Coumarins 16 [22], 22 [22], 30 [22], 33 [22], and 126 [22], exostemin (131) [22]
<i>E. caribaeum</i>	Coumarins 3 [11], 11 [19], 13 [11], 26 [11], 30 [11], and 126 [11], oxidocoumarins 36 [19], 108 [11], and 125 [19], exostemin (131) [145, 146]
<i>E. mexicanum</i>	Coumarins 17 [26], 22 [26], 26 [37], 31 [26], and 123 [26], 8-O-methylexostemin (107) [26], seshadrin (116) [26], exomexin A (129) [26], exomexin B (130) [26]
<i>Hintonia latiflora</i>	Coumarins 3 [12], 4 [13], 11 [12], 12 [12], 26 [12], and 27 [12], oxidocoumarin 108 [128]
<u>Leguminosae (Fabaceae) family</u>	
<i>Cassia alata</i>	Dalbergin (28) [62]
<i>Dalbergia baroni</i>	Dalbergin (28) [52, 53], melanein (102) [52, 53]
<i>D. cearensis</i>	Dalbergin (28) [47]
<i>D. coromandeliana</i>	Dalbergin (28) [51]
<i>D. cultrata</i>	Dalbergin (28) [55], stevenin (117) [55]
<i>D. latifolia</i>	Dalbergin (28) [42], O-methyldalbergin (104) [42]
<i>D. melanoxyton</i>	Dalbergin (28) [43], melanein (102) [52, 53], melanetin (103) [122]
<i>D. miscolobium</i>	Dalbergin (28) [45]
<i>D. nigra</i>	Dalbergin (28) [44], O-methyldalbergin (104) [44]
<i>D. nitidula</i>	Dalbergin (28) [48]
<i>D. odorifera</i>	3'-Hydroxymelanetin (19) [30, 31], dalbergin (28) [30, 31], melanetin (103) [30, 31], stevenin (117) [30, 31]
<i>D. riparia</i>	Dalbergin (28) [49]
<i>D. sissoides</i>	Dalbergin (28) [46]
<i>D. sissoo</i>	Dalbergin (28) [40, 41], isodalbergin (44) [69], O-methyldalbergin (104) [125], nordalbergin (112) [125]
<i>D. spinosa</i>	Dalbergin (28) [50]
<i>D. stevensonii</i>	Dalbergin (28) [54], stevenin (117) [54, 137]
<i>D. volubilis</i>	Volubolin (8) [15, 16], voludal (9) [16, 17], dalbergin (28) [15, 16], seshadrin (116) [16, 135]
<i>Glycyrrhiza inflata</i>	Inflacoumarin A (66) [82, 83]
<i>Goniorrhachis marginata</i>	Dalbergin (28) [61], O-methyldalbergin (104) [61]
<i>Indigofera oblongifolia</i>	Coumarin 126 [144]
<i>Machaerium kuhlmannii</i>	Dalbergin (28) [56], kuhlmannin (76) [56, 95]
<i>M. nictitans</i>	Dalbergin (28) [56], kuhlmannin (76) [56, 95]
<i>M. pedicellatum</i>	Dalbergin (28) [58], kuhlmannin (76) [58], O-methyldalbergin (104) [58]
<i>M. scleroxylon</i>	Dalbergin (28) [57], O-methyldalbergin (104) [57]
<i>Piptadenia macrocarpa</i>	Dalbergin (28) [59], kuhlmannin (76) [59]
<i>Prosopis kuntzei</i>	Dalbergin (28) [60], O-methyldalbergin (104) [60]
<u>Thelypteridaceae family</u>	
<i>Cyclosorus interruptus</i>	Interruptins A (59) [81], B (60) [81], C (61) [81], D (62) [81], E (63) [81], F (64) [81], and G (65) [81]
<u>Passifloraceae family</u>	
<i>Passiflora serratodigitata</i>	Serratin (115) [36], serratin 7- β -glucoside (25) [36]
<u>Asteraceae family</u>	
<i>Echinops niveus</i>	Nivegin (110) [129, 130], nivitin (111) [130]
<u>Rutaceae family</u>	
<i>Hesperehusa crenulata</i>	Coumarin 10 [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- α -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-Dioxocancoumarin (**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 β -D-galactopyranose (**10-13**) and β -D-glucopyranose (**25-27**). In single instances 6-acetyl- β -D-glucopyranose (**4**) and 6-acetyl- β -D-galactopyranose (**3**) in addition to disaccharides D-apofuranosyl-(1 \rightarrow 6)- β -D-glucopyranose (**2**) and β -D-xylopyranosyl-(1 \rightarrow 6)- β -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 cm⁻¹. The IR spectra also exhibit absorption bands in the ranges 1520-1570 and 1600-1630 cm⁻¹ due to vibrations of the α -pyrone ring in addition to bands at 690-700 and 740-750 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].

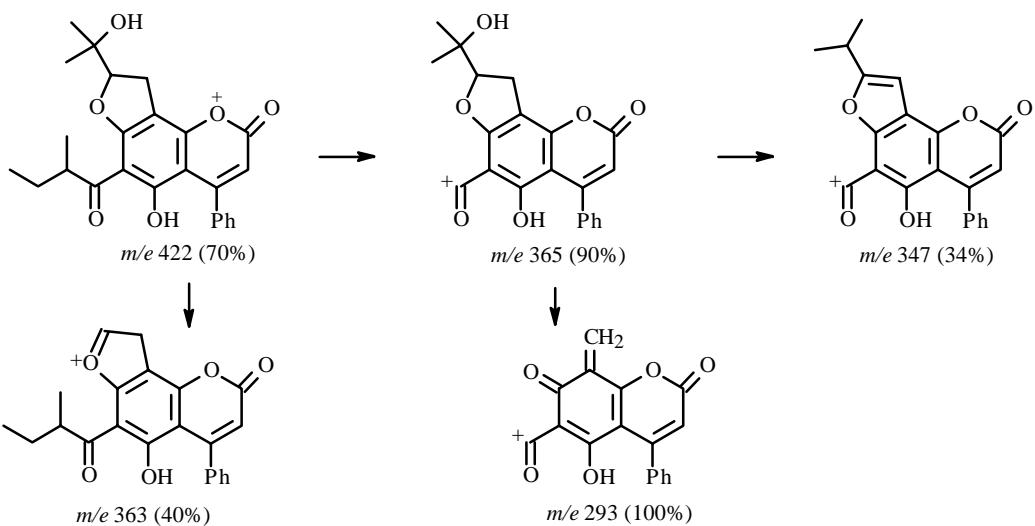
MASS SPECTROSCOPY

Most reports contain data for the mass numbers and relative intensities of the molecular ions or the base and principal ions.

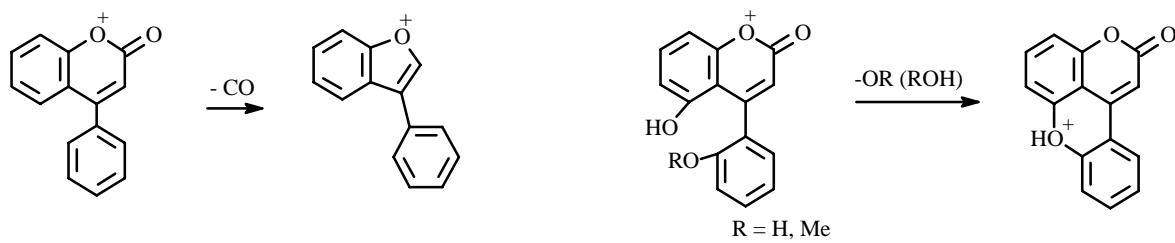
Mass spectra of neoflavones isolated from *Mammea americana*, *M. africana*, and *Calophyllum inophyllum* have been studied in the most detail [8, 98, 99, 116]. Simultaneous application of gas chromatography and mass spectroscopy to identify natural 4-phenylcoumarins and the overall fragmentation pathways 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 benzopyranyl 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 isobutenoide from the [M - 57] ion (Scheme 1).



Scheme 1



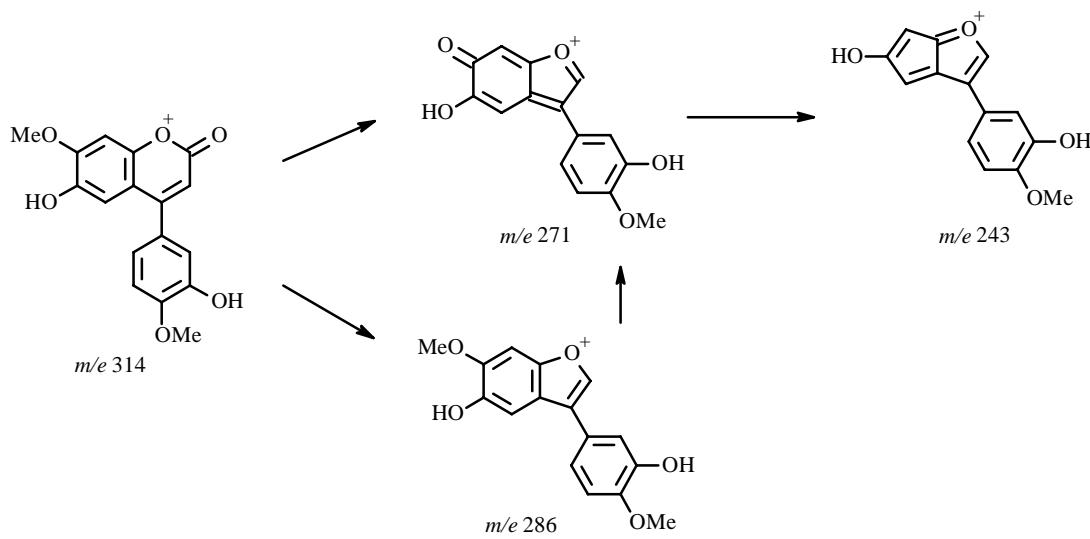
Scheme 2

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 isobuteneoxide from the $[M - 43]^+$ fragment [101].

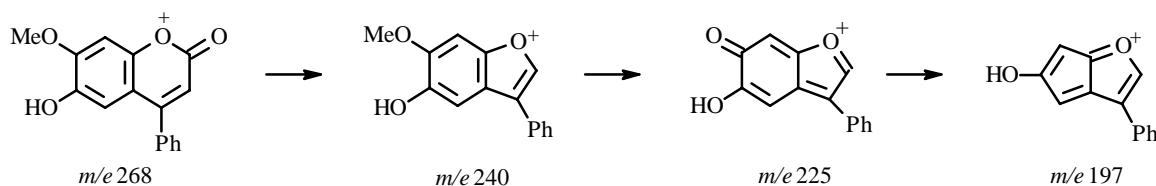
The mass spectrum of tomentolide A (**122**) [7] has a peak for the molecular ion at $m/e 402$. A strong peak at $m/e 387$ [$M - 15$] corresponds to the stable benzopyranyl ion 2,2-dimethylchromene. Other important peaks with $m/e 331$ [$M - 15 - 56$] and $m/e 303$ [$M - 15 - 56 - 28$] are formed by successive loss of butene and CO from the ion with $m/e 387$ [7]. The spectrum of apetatalide (**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).



An analogous fragmentation pattern was reported for dalbergin (**28**) [63]. The molecular ion with *m/e* 268 loses the stable molecule CO₂ 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 CO₂ 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 C₂H₃O 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 [M - CO]⁺ (95%) and *m/z* 197 [M - CO - COH]⁺ (47%) [36].

A strong peak for the molecular ion with *m/z* 342 [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 [M - 28]⁺ and *m/z* 286 [M - 56]⁺ [17]. Ions with *m/z* 313 [M - 29]⁺ and *m/z* 285 [M - 57]⁺ are formed via loss of a -CHO radical and subsequent elimination of a CO molecule. A peak with *m/z* 284 [M - 58]⁺ is formed by loss of (CHO + CO + 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 [M - 57]⁺ forms cations with *m/z* 270 [M - 72]⁺ and *m/z* 267 [M - 75]⁺, respectively. Fragments with *m/z* 257 [M - 87]⁺ and *m/z* 255 [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 [M - H]⁻ and a base peak with *m/z* 285 [M - 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 [M - H]⁻ and a base peak with *m/z* 299 [M - 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 [M - H]⁻, a fragment with *m/z* 447 [M - 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 SPECGTROSCOPY

Proton and carbon NMR methods can unambiguously establish the structures of neoflavones. Special investigations and analysis of ^{13}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 ($J = 8\text{-}8.7 \text{ Hz}$) 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 ($J = 8\text{-}8.6 \text{ Hz}$) 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, $J = 1.8\text{-}2.2 \text{ Hz}$, $J = 7.2\text{-}8.2 \text{ Hz}$, 1H, H-6'), 6.71-6.86 ppm (d, $J = 1.8\text{-}2.2 \text{ Hz}$, 1H, H-2'), and 6.72-6.98 ppm (d, $J = 7.2\text{-}8.2 \text{ Hz}$, 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 ($J = 2.19\text{-}3.0 \text{ Hz}$) 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-d₆) [26, 33, 141] or 6.30-6.35 ppm (CDCl₃) [143].

A distinguishing feature of 4-phenylcoumarins containing 3-methylbutanoic acid is the presence of a 6H doublet ($J = 6.5\text{-}7.0 \text{ Hz}$) for the methyls at 0.9-1.08 ppm, a multiplet for the CH at 2.14-2.37 ppm, and a doublet ($J = 6.5\text{-}7.0 \text{ Hz}$) 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, $J = 7.0\text{-}7.5 \text{ Hz}$, CH₃-5), 1.12-1.32 ppm (3H, d, $J = 6.5\text{-}7.0 \text{ Hz}$, CH₃-3), 1.30-1.50 ppm (1H, m, CH₂-4a), 1.77-1.92 ppm (1H, m, CH₂-4b), 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, $J = 7.0\text{-}7.5 \text{ Hz}$, CH₃-4), 1.68-1.83 ppm (2H, m, CH₂-3), and 2.98-3.34 ppm (2H, t, $J = 7.0\text{-}7.5 \text{ Hz}$, CH₂-2) [72, 101, 117]. The 2-methylpropionyl group has a characteristic 6H doublet ($J = 7.0\text{-}10.0 \text{ Hz}$) 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, $J = 7.4\text{-}8.0 \text{ Hz}$, H-3, H-5), 7.57-7.63 ppm (1H, tt, $J = 7.4\text{-}8.4 \text{ Hz}$, $J = 1.0\text{-}1.7 \text{ Hz}$, H-4''), and 7.65-7.89 ppm (2H, dd, $J = 7.4\text{-}8.4 \text{ Hz}$, $J = 1.0\text{-}1.7 \text{ Hz}$, 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 ($J = 6.9\text{-}8.0 \text{ ppm}$) at 3.08-3.57 ppm, and a 1H doublet ($J = 6.0\text{-}7.5 \text{ Hz}$) or triplet ($J = 7.0\text{-}7.5 \text{ Hz}$) 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, CH₃-5), 2.77-3.06 ppm (1H, dd, $J = 7.5\text{-}8.0 \text{ Hz}$, $J = 15.0\text{-}15.5 \text{ Hz}$, CH₂-1a), 3.10-3.32 ppm (1H, dd, $J = 2.0 \text{ Hz}$, $J = 15.0 \text{ Hz}$, CH₂-1b), 4.32-4.51 ppm (1H, d, $J = 6.0\text{-}8.0 \text{ Hz}$, H-2), 4.86-4.93 ppm (1H, s, CH₂-4a), and 4.94-5.03 ppm (1H, s, CH₂-4b) 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 ($J = 8.1\text{-}10.0 \text{ Hz}$, $J = 15.0\text{-}15.5 \text{ Hz}$), and a 1H triplet or doublet of doublets at 4.52-4.92 ppm ($J = 8.1\text{-}9.8 \text{ Hz}$) 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 ($J = 9.9\text{-}11.0 \text{ Hz}$) 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-pyranocoumarins 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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