Constituents of an Extract of *Cryptocarya rubra* Housed in a Repository with Cytotoxic and Glucose Transport Inhibitory Effects[#]

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[#]Dedicated to Professor Otto Sticher, of ETH-Zurich, Zurich, Switzerland, for his pioneering work in pharmacognosy and phytochemistry.

Content of Supporting Information

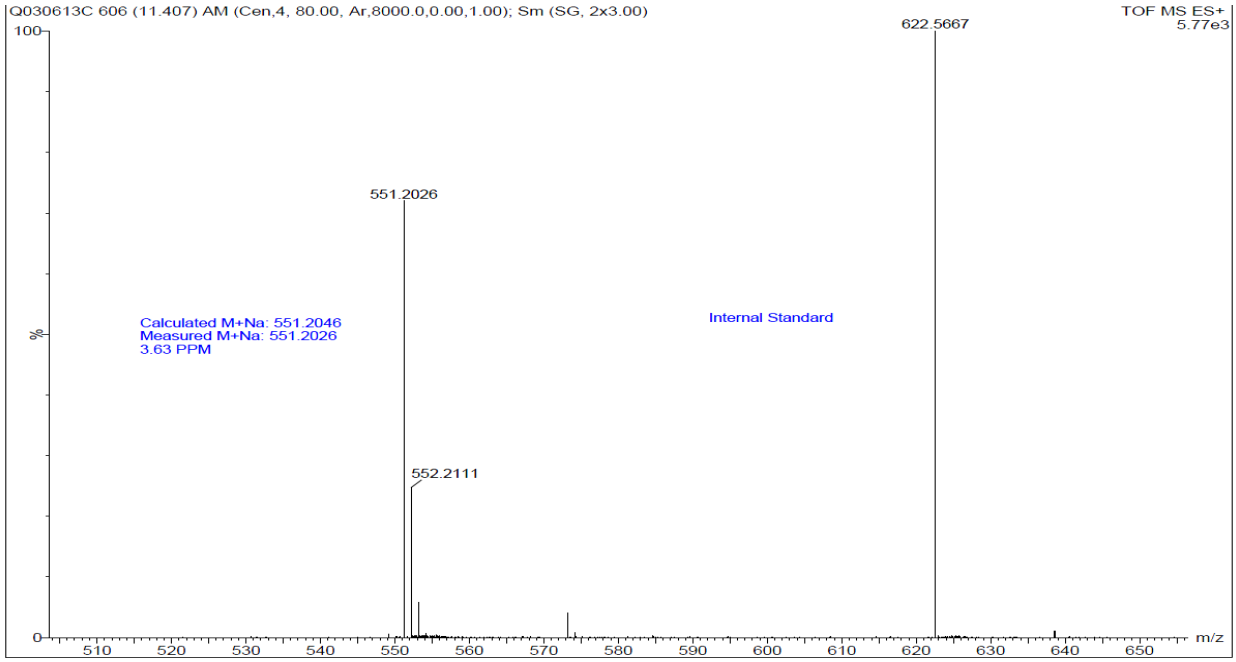
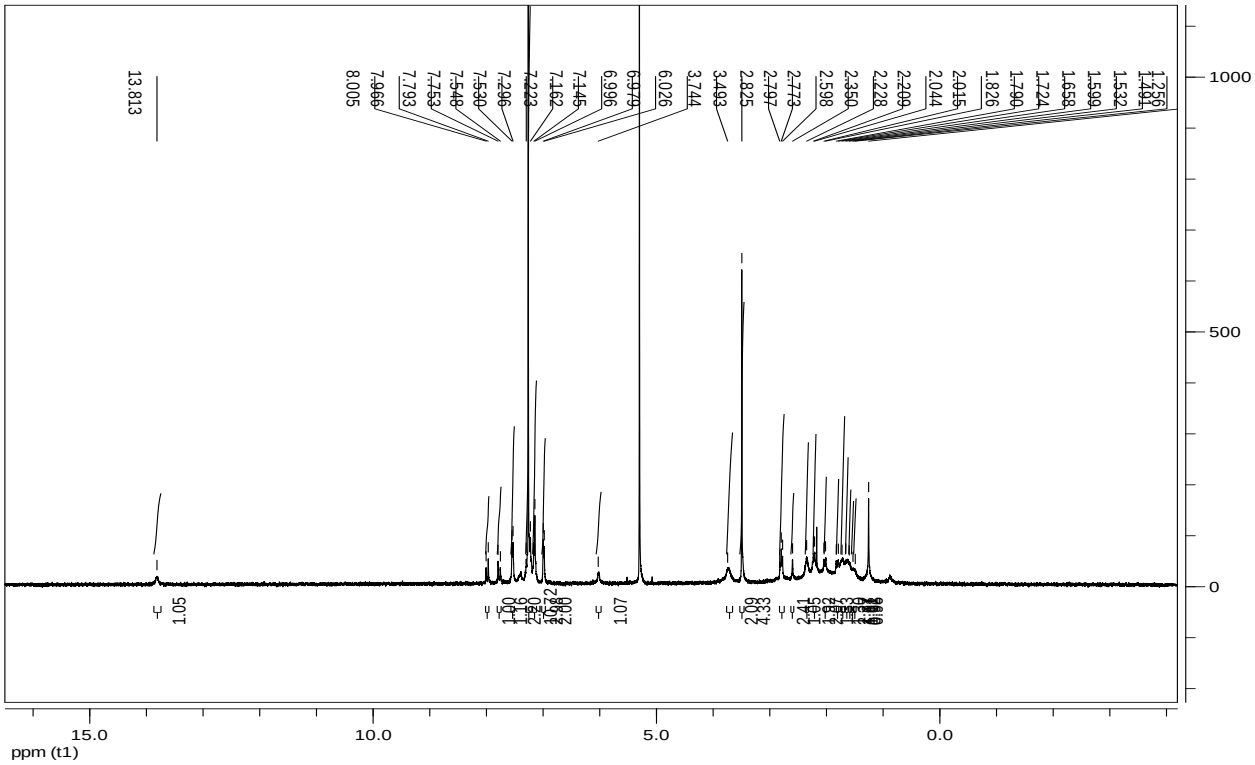


Figure S1. MS spectrum of (–)-rubrichalcolactone (**1**).



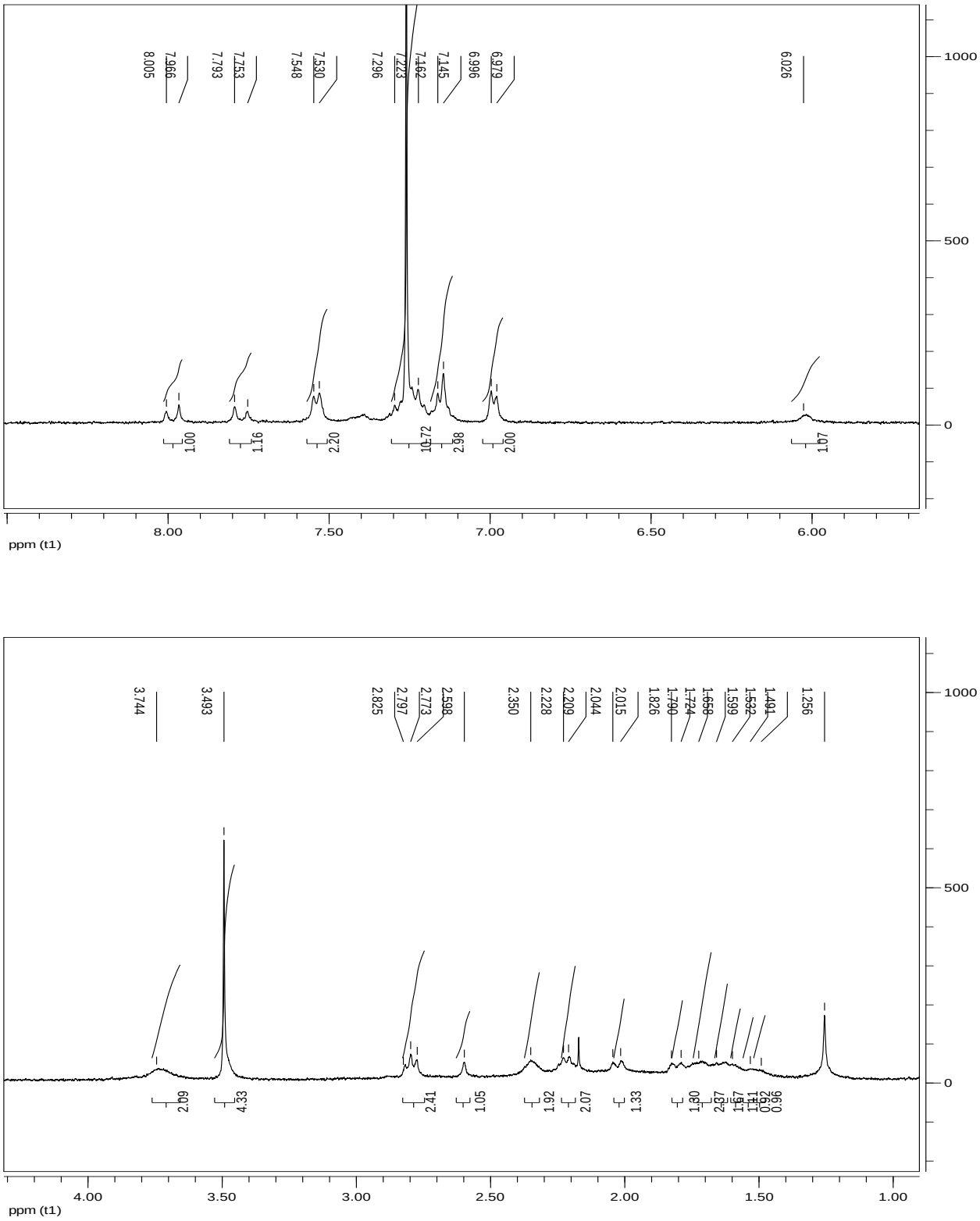
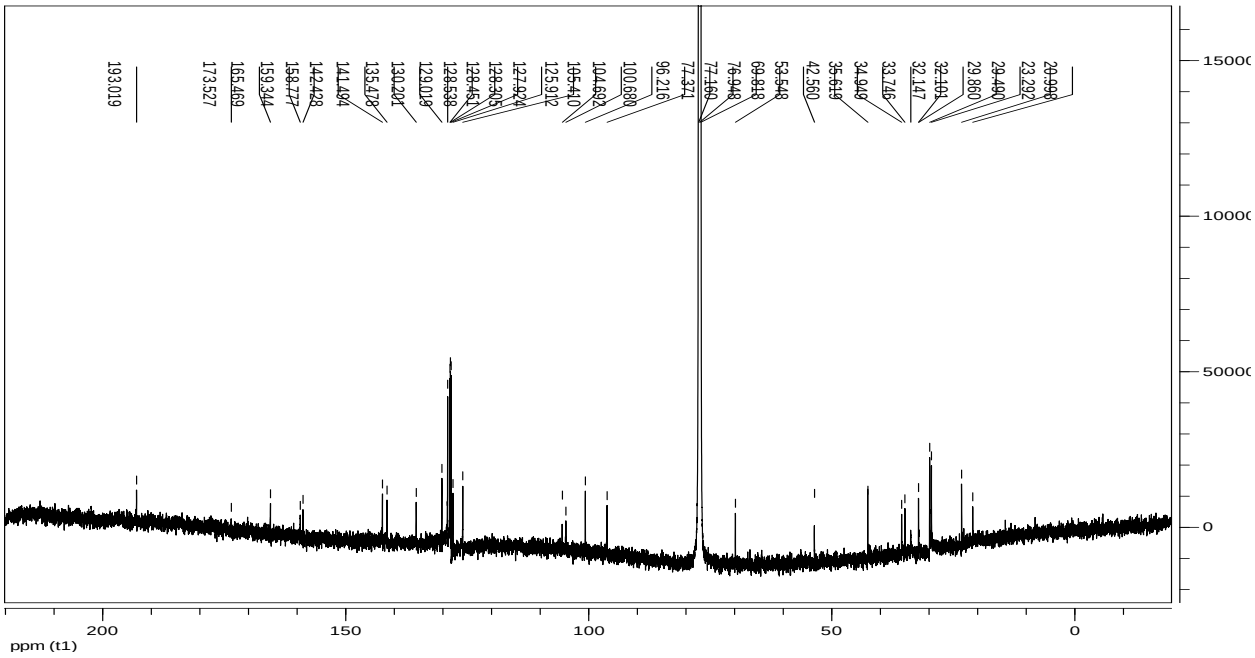


Figure S2a. ^{1}H NMR spectrum of (–)-rubrichalcolactone (**1**).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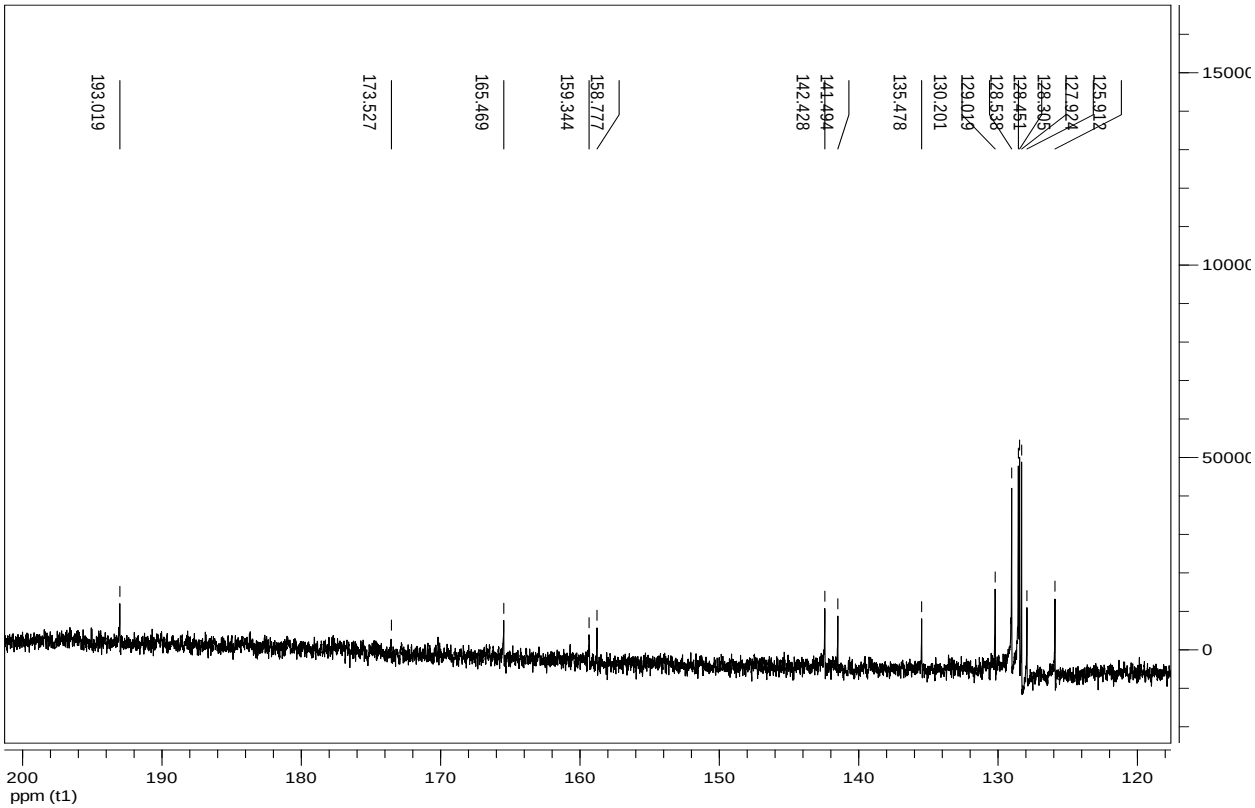
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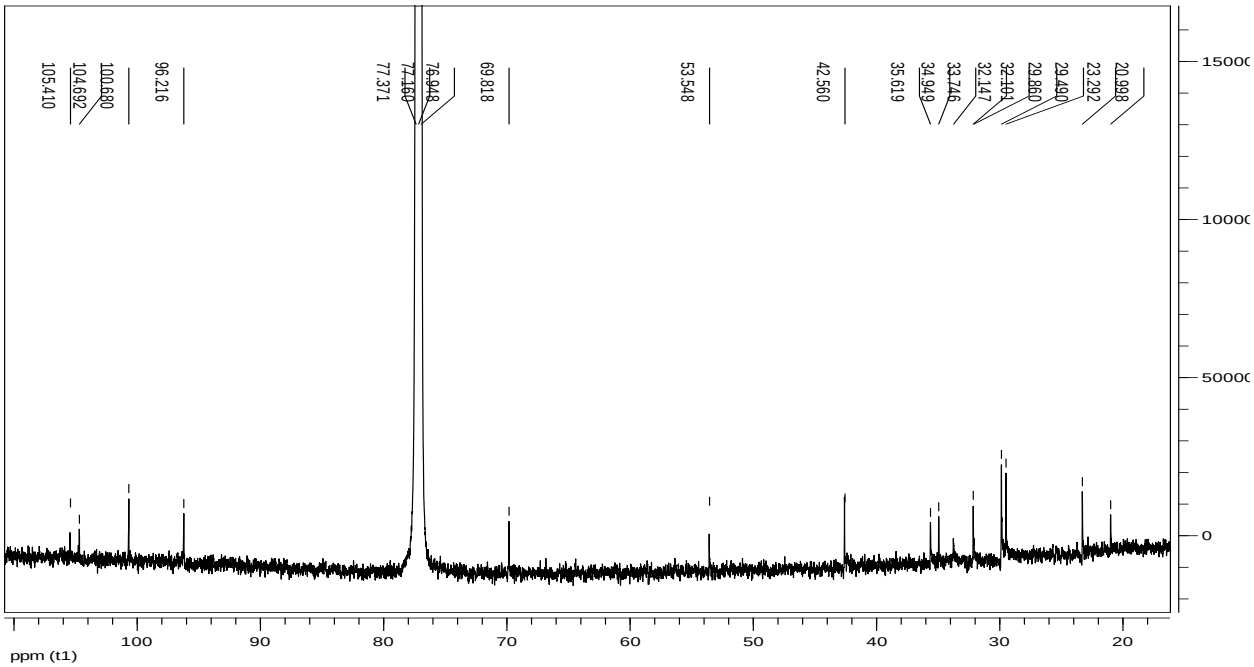


Figure S2b. ^{13}C NMR spectrum of (–)-rubrichalcolactone (**1**).

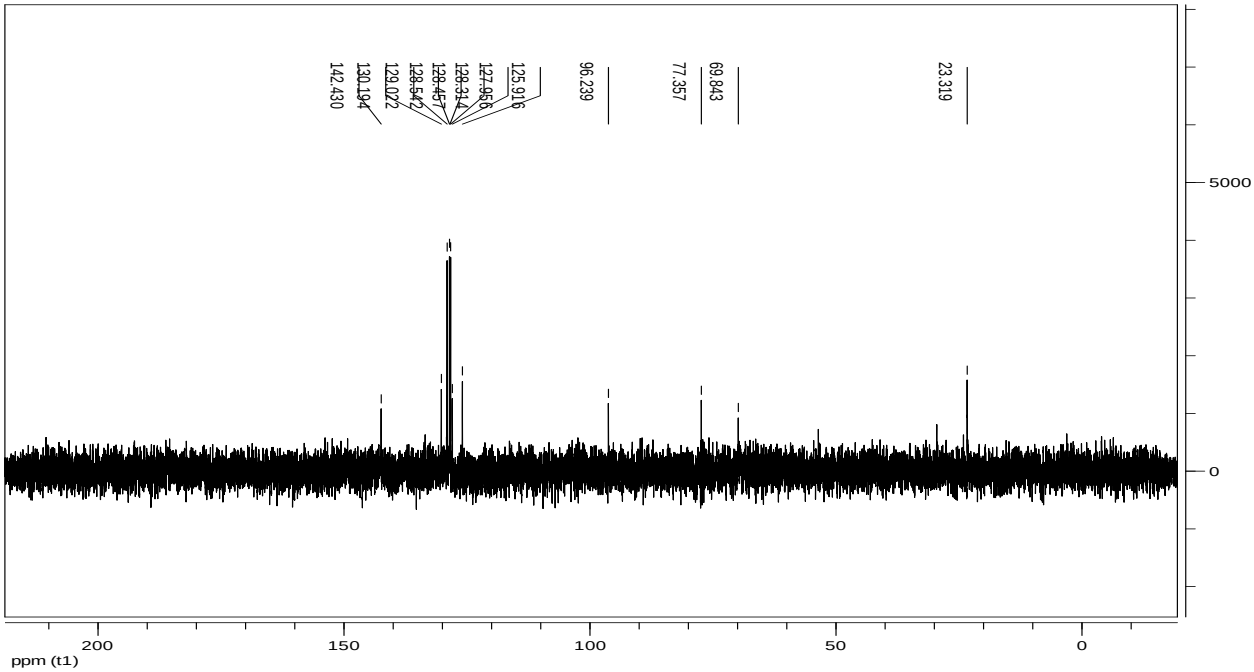


Figure S2c. DEPT 90 NMR spectrum of (–)-rubrichalcolactone (**1**).

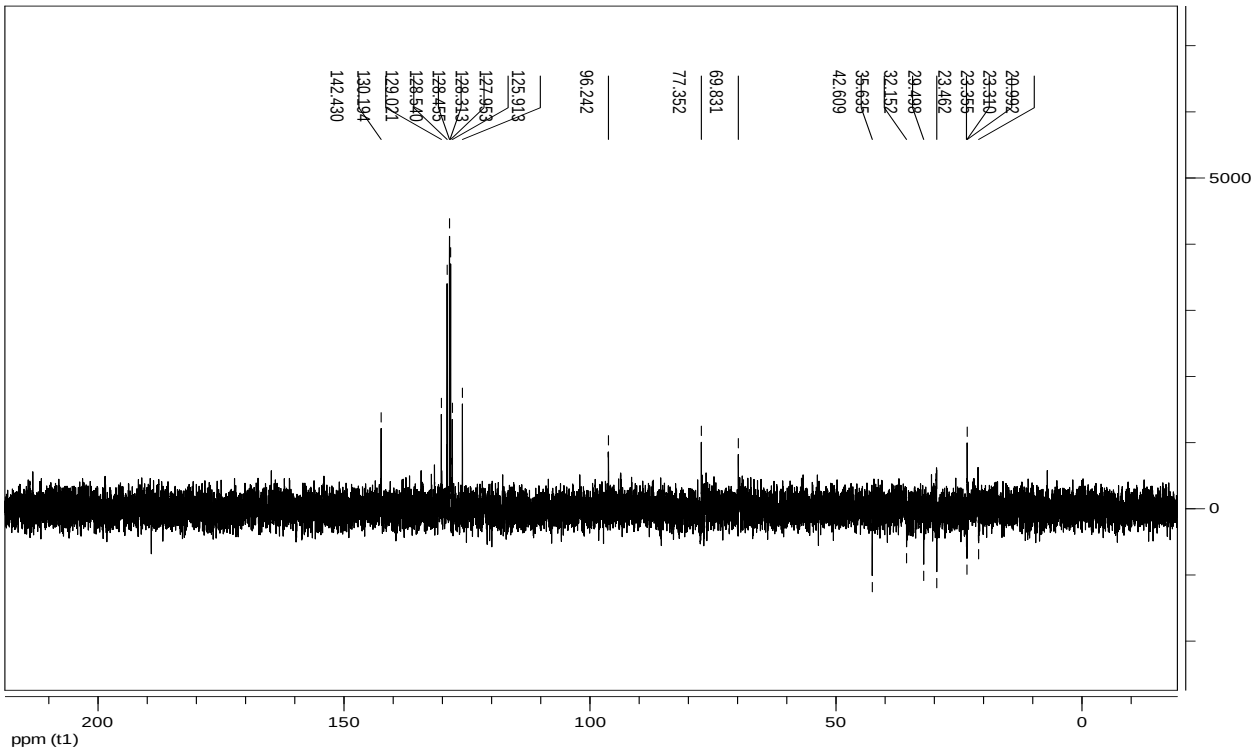


Figure S2d. DEPT 135 NMR spectrum of (–)-rubrichalcolactone (**1**).

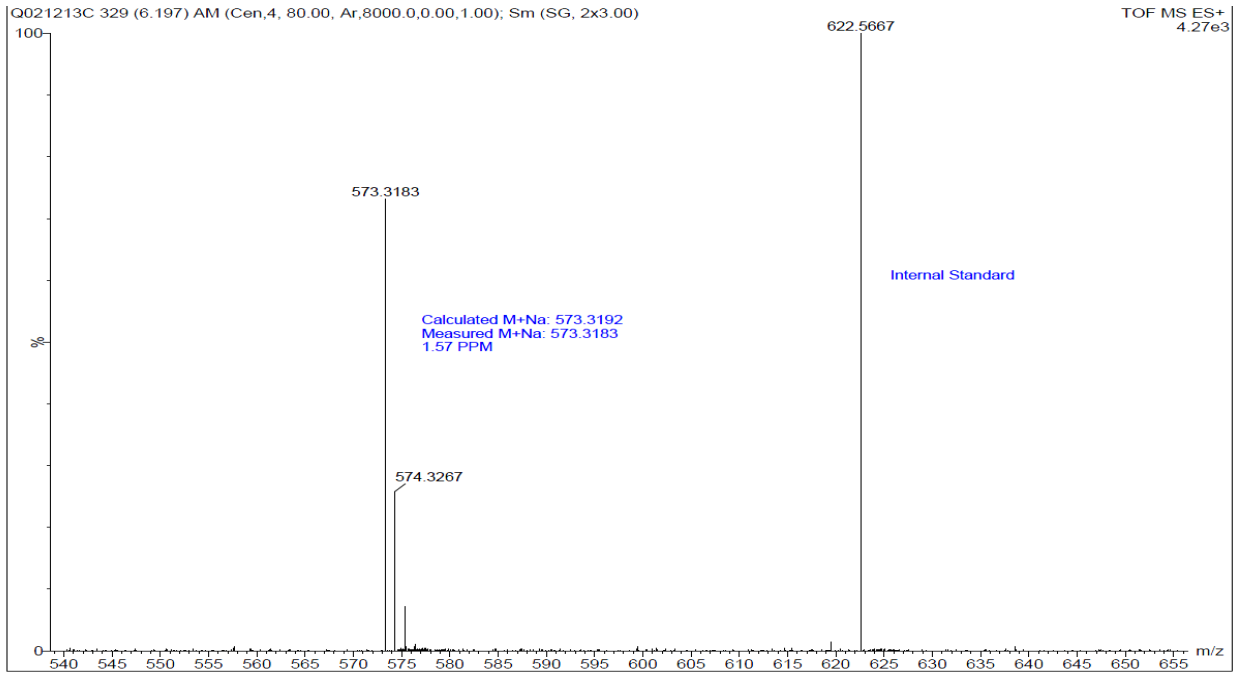
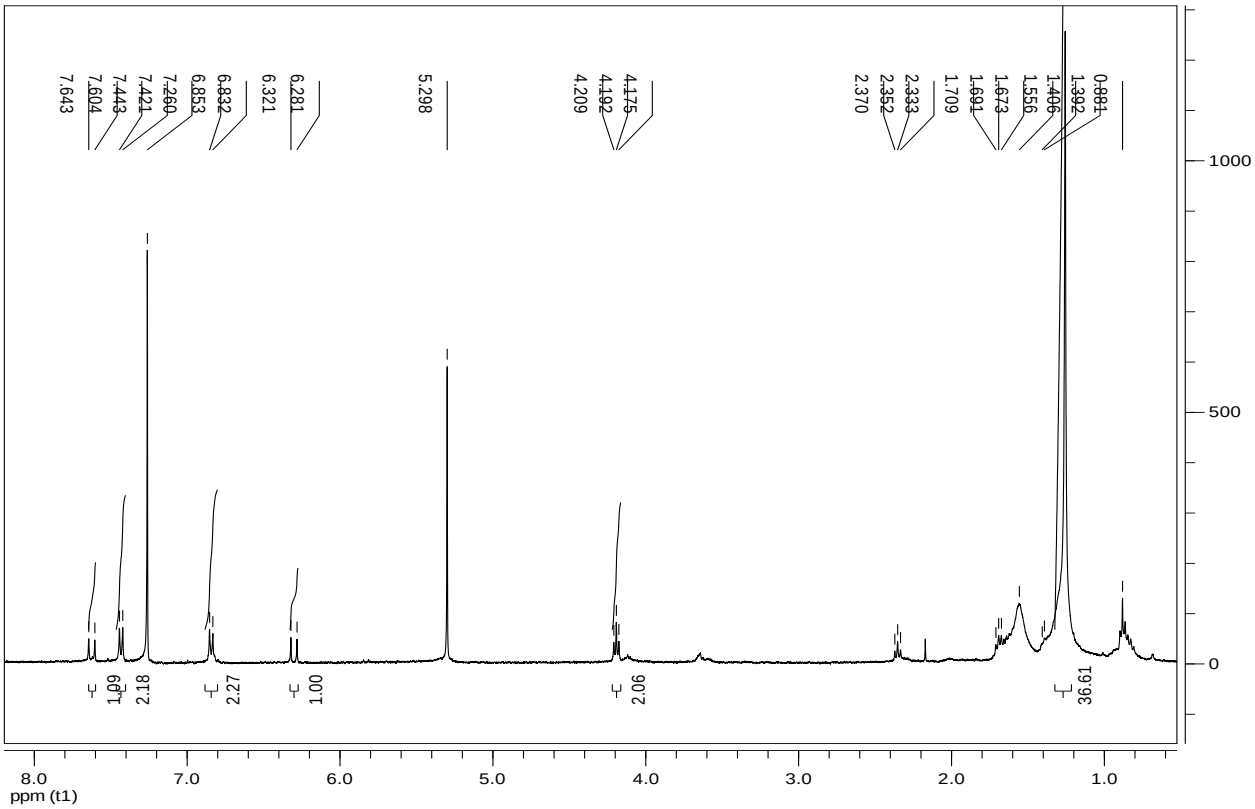


Figure S3. MS spectrum of 1,16-hexadecanediol-di-*p*-coumaroate (**2**).



Figures S4a. ^{1}H NMR spectrum of 1,16-hexadecanediol-di-*p*-coumaroate (**2**).

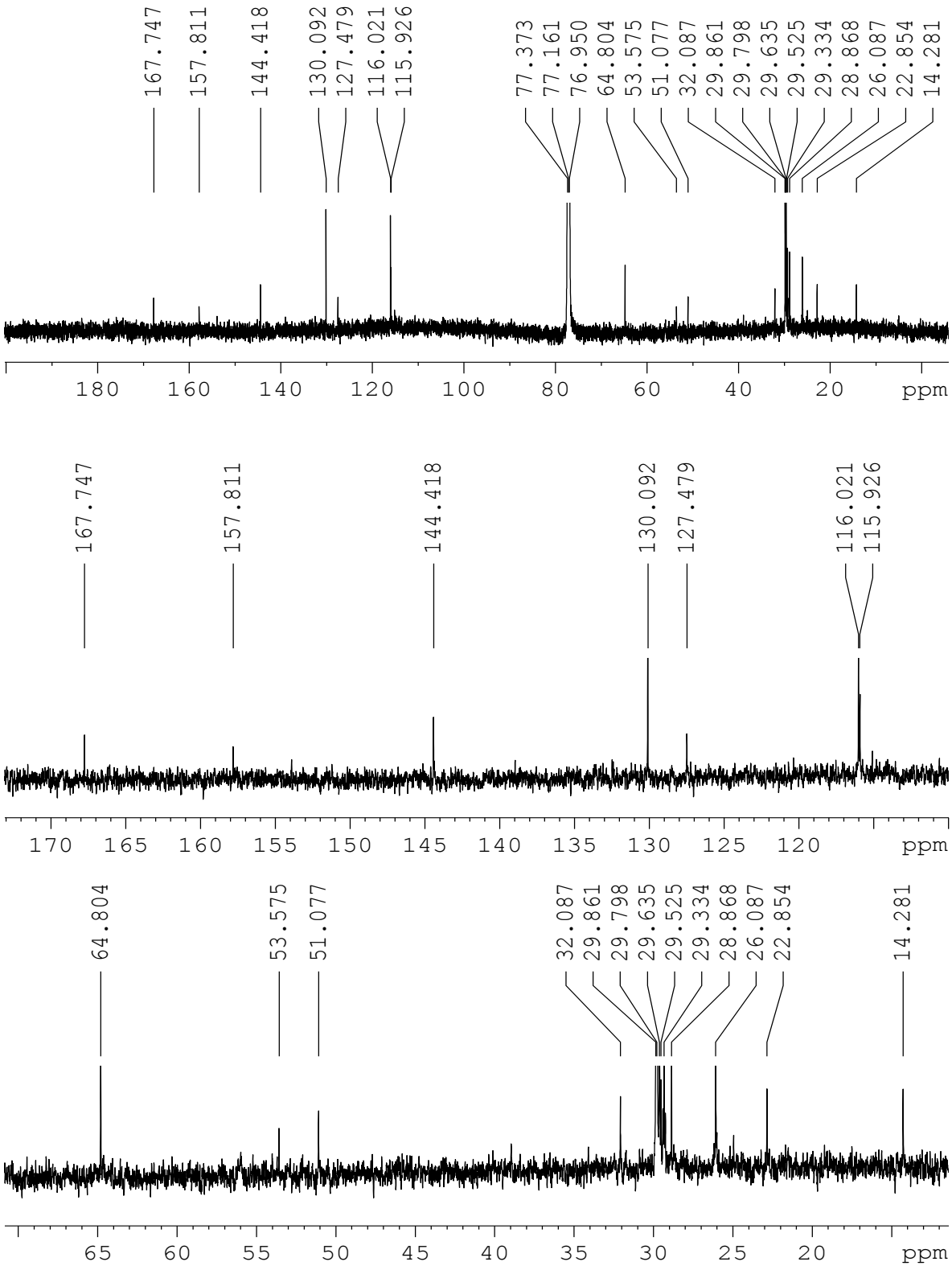


Figure S4b. ^{13}C NMR spectrum of 1,16-hexadecanediol-di-*p*-coumaroate (**2**). Note: The signals at δ_C 32.1, 22.9 and 14.3 are due to *n*-hexane complexed with **2**.[23,24] The signals at δ_C 53.6 is due to the trace solvent component of dichloromethane.[24] The signals at δ_C 51.1 is due to the trace solvent component of MeOH.[24]

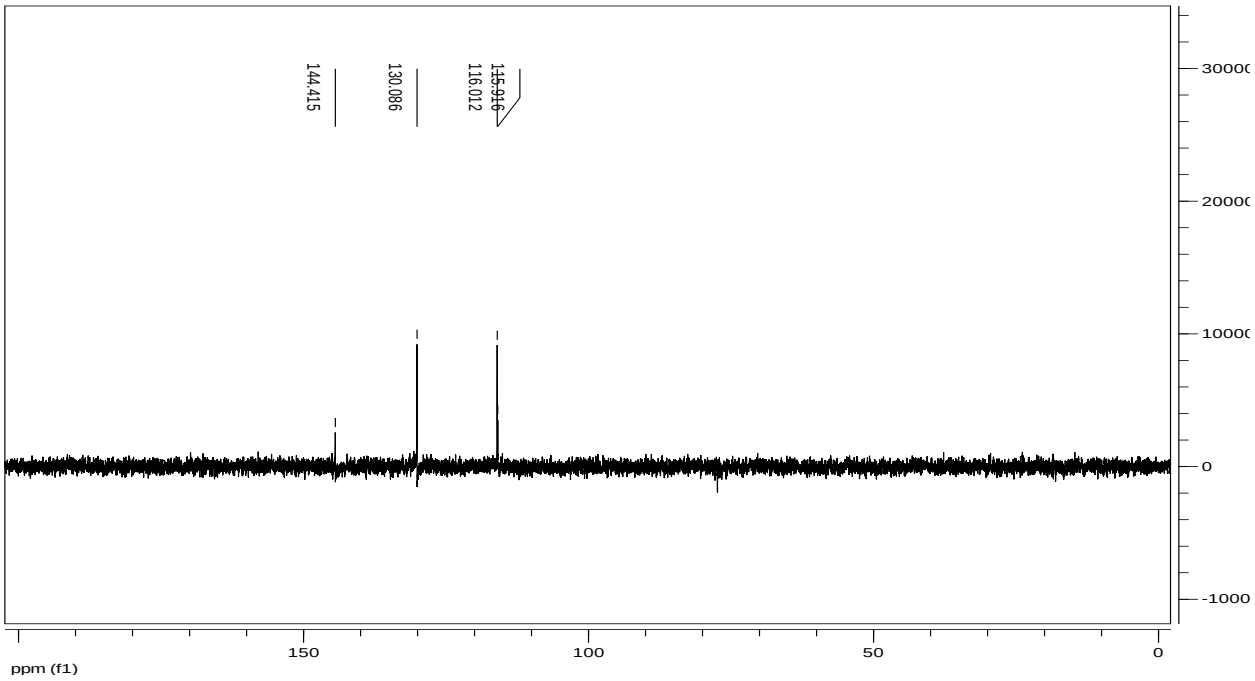


Figure S4c. DEPT 90 NMR spectrum of 1,16-hexadecanediol-di-*p*-coumaroate (**2**).

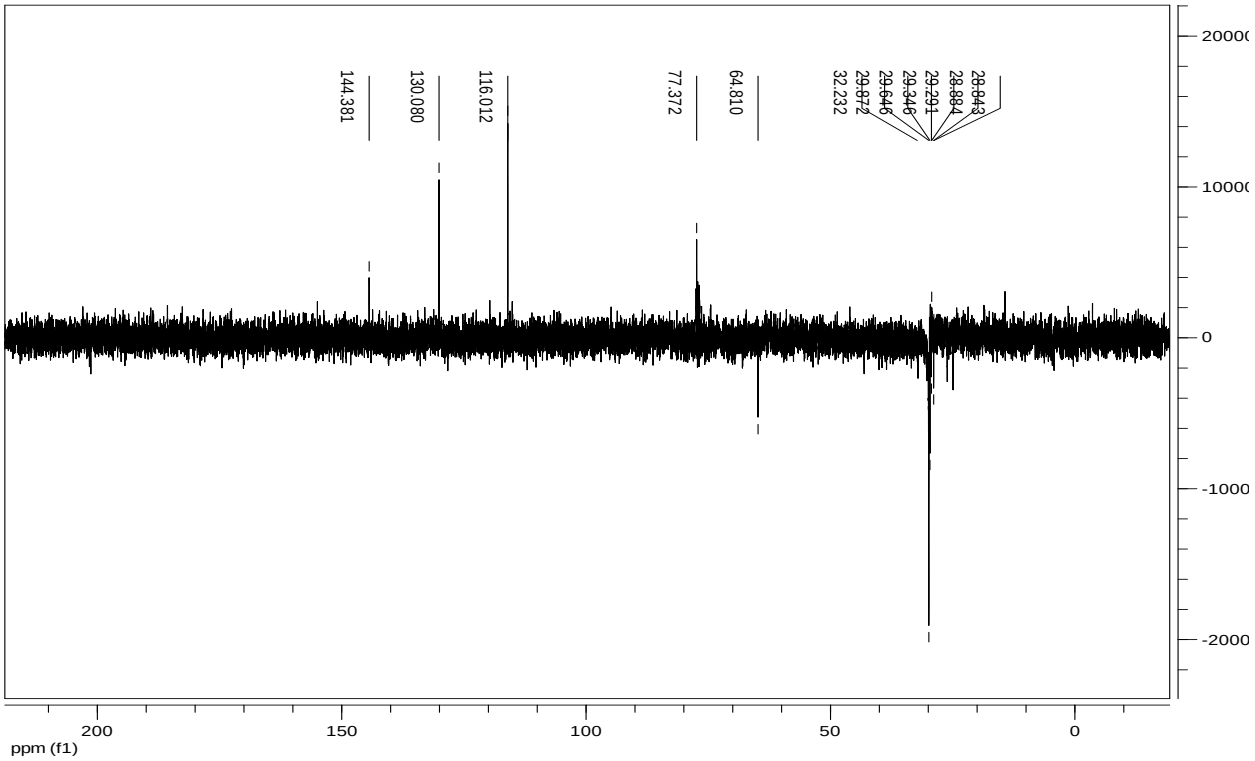
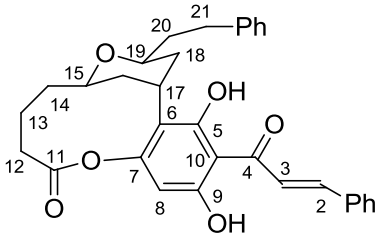


Figure S4d. DEPT 135 NMR spectrum of 1,16-hexadecanediol-di-*p*-coumaroate (**2**).



1

Figure S5. Structure indicating the conformation of the ring systems based on Dreiding model for **1**.

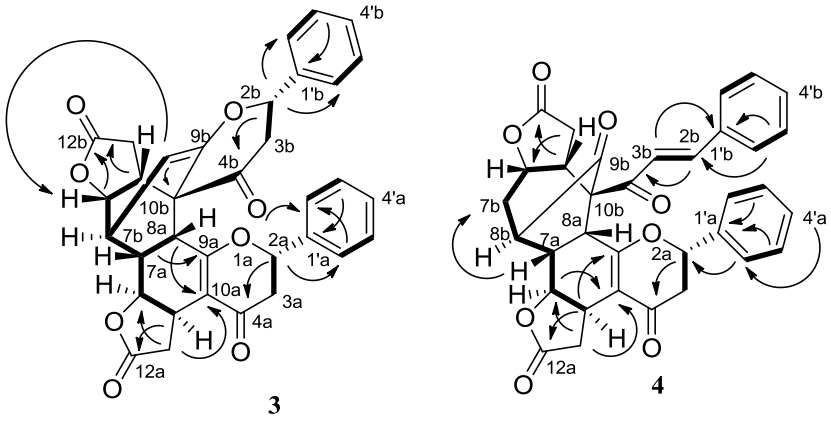


Figure S6. COSY (▬ , $^{1}H \rightarrow {}^{1}H$) and key HMBC (⌒ , $^{1}H \rightarrow {}^{13}C$) correlations of **3** and **4**.

Table S1. ^{1}H- and ^{13}C NMR Spectroscopic Data of Compounds 3 and 4 in $CDCl_3$

Position[a]	3		4	
	δ_C (mult.)[b]	δ_H[c]	δ_C (mult.)[b]	δ_H[d]
2a	79.5 CH	5.38 6 dd (3.6, 14.4)	81.0 CH	5.20 dd (3.0, 15.6)
3a	40.7 CH_2	2.87 d (8.0)	41.8 CH_2	2.56 dd (2.4, 17.4)
		2.93 d (8.8)		2.83 dd (11.4, 16.8)
4a	190.8 C		190.9 C	
5a	33.2 CH	3.28 br t (5.2)	32.9 CH	3.43 br t (5.4)
6a	80.1 CH	4.68 m	79.3 CH	4.80 m
7a	37.0 CH	2.73 br d (8.0)	33.9 CH	2.87 br d (8.4)
8a	45.3 CH	3.35 d (7.6)	38.4 CH	4.07 d (9.0)
9a	168.4 C		169.1 C	
10a	112.6 C		111.0 C	
11a	37.9 CH_2	2.82 m	37.8 CH_2	2.81 overlap
		2.93 d (8.8)		3.08 dd (7.2, 17.4)
12a	175.1 C		174.6 C	
1′a	135.6 C		135.5 C	
2′a	126.3 CH	7.22 m	127.1 CH	7.17 d (7.2)
3′a	129.1 CH	7.34 m	128.5 CH	6.91 m
4′a	129.6 CH	7.34 m	128.9 CH	6.91 m
5′a	129.1 CH	7.34 m	128.5 CH	6.91 m
6′a	126.3 CH	7.22 m	127.1 CH	7.17 d (7.2)
2b	76.1 CH	4.93 m	145.3 CH	7.28 d (15.6)
3b	47.2 CH_2	2.55 m	124.0 CH	7.78 d (15.6)
4b	204.7 C		195.1 C	
5b	40.7 CH	3.20 m	41.8 CH	3.37 m
6b	80.6 CH	4.93 m	78.5 CH	4.92 m
7b	38.9 CH	3.20 m	36.3 CH_2	2.24 br d (18.0)
				2.72 m
8b	100.7 CH	5.29 d (5.6)	34.8 CH	2.72 m
9b	153.7 C		205.9 C	
10b	53.6 C		63.0 C	
11b	32.5 CH_2	2.07 dd (2.4, 18.8)	30.2 CH_2	2.40 dd (3.0, 19.2)
		2.82 m		2.80 dd (1.8, 14.4)
12b	174.5 C		173.9 C	
1′b	138.2 C		134.6 C	
2′b	125.5 CH	7.16 m	129.2 CH	7.46 m
3′b	129.0 CH	7.38 m	129.3 CH	7.46 m
4′b	129.0 CH	7.38 m	131.2 CH	7.46 m
5′b	129.0 CH	7.38 m	129.3 CH	7.46 m
6′b	125.5 CH	7.16 m	129.2 CH	7.46 m

[a]Assigned by analysis of ^{1}H, ^{13}C, DEPT 90, DEPT 135, COSY, HSQC, and HMBC NMR spectra.
[b]Recorded at 150.9 MHz and referenced to the solvent residual peak at δ 77.16.[24]. CH_3, CH_2, CH, and C multiplicities were determined by DEPT 90 and DEPT 135 and HSQC experiments.
[c]Recorded at 400.1 MHz and referenced to the solvent residual peak at δ 7.26.[24].
[d]Recorded at 600.2 MHz and referenced to the solvent residual peak at δ 7.26.[24].

Table S2. ^{1}H NMR Spectroscopic Data of the Known Compounds from *C. rubra*

Position*	**5**[a]	**7**[b]	**S1**[a]	**S2**[c]	**S3**[b]
2	7.79 d (15.2)	5.49 m	5.45 dd (2.8, 12.8)	5.60 dd (2.8, 12.4)	7.55 br s
3	6.79 d (15.6)	2.74 dd (2.4, 12.6) 2.95 dd (10.2, 12.6)	2.85 dd (2.8, 17.2) 3.13 dd (12.8, 16.8)	2.84 dd (3.2, 17.2) 3.21 dd (12.8, 17.2)	7.42 m
4					7.42 m
5	4.03 m	3.89 q (7.8)			7.42 m
6	5.49 br d (8.8)	5.49 m	6.07 d (1.6)	5.97 d (2.0)	7.55 br s
7	6.56 br d (10.4)	6.29 dd (2.4, 7.8)			7.79 d (15.6)
8	6.23 br d (11.6)	6.10 dd (1.8, 7.8)	6.09 d (2.0)	6.01 d (2.0)	6.48 d (16.2)
9					
10					
11	2.66 dd (12.4, 17.6) 2.82 dd (8.8, 17.6)	2.41 dd (7.8, 13.2) 2.98 dd (6.6, 13.2)			
12					
1'					
2'	7.58 m	7.45 m	7.46 m	7.58 d (7.2)	
3'	7.43 m	7.45 m	7.46 m	7.47 m	
4'	7.43 m	7.45 m	7.46 m	7.47 m	
5'	7.43 m	7.45 m	7.46 m	7.47 m	
6'	7.58 m	7.45 m	7.46 m	7.58 d (7.2)	
4-OH	15.99 s				
5-OH			12.02 s	12.15 s	
7-OCH_3			3.82 s		

*Assignments based on analysis of 2D NMR spectra.

[a]Data (δ) measured in $CDCl_3$ at 400.1 MHz and referenced to the solvent residual peak at δ 7.26.[24]

[b]Data (δ) measured in $CDCl_3$ at 600.2 MHz and referenced to the solvent residual peak at δ 7.26.[24].

[c]Data (δ) measured in acetone-d_6 at 400.1 MHz and referenced to the solvent residual peak at δ 2.05[24]

J values are presented in Hz. **S1**: (±)-5-hydroxy-7-methoxyflavanone; **S2**: (±)-5,7-dihydroxyflavanone; **S3**: cinnamic acid.

Table S3. ^{13}C NMR Spectroscopic Data of the Known Compounds from *C. rubra*

Position*	**5**[a]	**7**[a]	**S1**[a]	**S2**[b]	**S3**[c]
1					134.2 C
2	142.5 CH	81.1 CH	79.4 CH	80.0 CH	128.5 CH
3	116.8 CH	42.9 CH_2	43.6 CH_2	43.7 CH_2	129.1 CH
4	174.2 C	190.4 C	195.9 C	196.8 C	129.1 CH
5	34.0 CH	30.7 CH	164.3 C	165.1 C	129.1 CH
6	76.3 CH	76.8 CH	95.3 CH	96.9 CH	128.5 CH
7	139.9 CH	134.8 CH	168.1 C	167.4 C	147.1 CH
8	130.7 CH	124.3 CH	94.4 CH	95.9 CH	117.0 CH
9	186.0 C	162.7 C	162.9 C	164.2 C	170.2 C[d]
10	103.4 C	109.0 C	103.3 C	103.2 C	
11	35.4 CH_2	33.2 CH_2			
12	174.6 C	175.6 C			
1'	134.9 C	137.7 C	138.5 C	140.1 C	
2'	128.3 CH	126.3 CH	126.3 CH	127.3 CH	
3'	129.2 CH	129.0 CH	129.0 CH	129.5 CH	
4'	130.3 CH	129.3 CH	129.0 CH	129.4 CH	
5'	129.2 CH	129.0 CH	129.0 CH	129.5 CH	
6'	128.3 CH	126.3 CH	126.3 CH	127.3 CH	
7-OCH_3			55.9 CH_3		

*Assigned by analysis of DEPT 90, DEPT 135, COSY, HSQC, and HMBC NMR spectra (CH_3, CH_2, CH, and C multiplicities were determined by DEPT 90 and DEPT 135 and HSQC experiments).
[a]Data (δ) measured in $CDCl_3$ at 100.6 MHz and referenced to the solvent residual peak at δ 77.16.[24]
[b]Data (δ) measured in acetone-d_6 at 100.6 MHz and referenced to the solvent residual peak at δ 29.84.[24]
[c]Data (δ) measured in $CDCl_3$ at 150.9 MHz and referenced to the solvent residual peak at δ 77.16.[24]
[d]Observed in the HMBC 2D NMR spectrum.
S1: (±)-5-hydroxy-7-methoxyflavanone; **S2**: (±)-5,7-dihydroxyflavanone; **S3**: cinnamic acid.

Aanalytical data of known compounds isolated from *C. rubra*

(+)-Bicaryanone A (**3**): Amorphous colorless powder; $[\alpha]^{20}_{D}$ +161.4 (*c* 0.07, MeOH); UV (MeOH) λ_{max} (log ε) 270 (3.83) nm; ECD (MeOH, nm) λ_{max} (Δε) 305.5 (–4.48), 267.0 (+14.18), 199.5 (+21.81); IR (dried film) ν_{max} 2924, 2853, 1782, 1717, 1667, 1615, 1455, 1299, 906, 760 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table S1; positive-ion sodiated HRESIMS *m*/*z* 587.1690 (calcd for $C_{34}H_{28}O_8Na$, 587.1682).

(+)-Chalcocaryanone C (**4**): Amorphous colorless powder; $[\alpha]^{20}_{D}$ +220.0 (*c* 0.01, MeOH); UV (MeOH) λ_{max} (log ε) 306 (4.02) nm; ECD (MeOH, nm) λ_{max} (Δε) 320.5 (+1.56), 268.5 (+7.80), 198.5 (+10.0); IR (dried film) ν_{max} 2924, 2853, 1784, 1727, 1669, 1603, 1574, 1450, 978,

759 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table S1; positive-ion sodiated HRESIMS *m*/*z* 587.1686 (calcd for $C_{34}H_{28}O_8Na$, 587.1682).

(+)-Cryptocaryone (**5**): Amorphous yellow powder. $[\alpha]^{20}_{D}$ +183.3 (*c* 0.03, MeOH); UV (MeOH) λ_{max} (log ε) 383 (3.76), 287 (3.55), 235 (3.47) nm; ECD (MeOH, nm) λ_{max} (Δε) 441.5 (+0.24), 384.0 (+2.34), 288.0 (–0.38), 250.0 (+0.50), 236.5 (–0.62), 223.5 (–0.78), 203.0 (+2.61); IR (dried film) ν_{max} 3442, 2926, 1778, 1729, 1629, 1579, 1496, 1283, 1022, 933, 831 cm^{-1}; ^{1}H and ^{13}C NMR data, see Tables S2 and S3; positive-ion sodiated HRESIMS *m*/*z* 305.0780 (calcd for $C_{17}H_{14}O_4Na$, 305.0790).

(+)-Desmethylinfectocaryone (**6**): Amorphous yellow powder; $[\alpha]^{20}_{D}$ +45.0 (*c* 0.02, MeOH); UV (MeOH) λ_{max} (log ε) 380 (3.95), 283 (3.82), 230 (3.82) nm; ECD (MeOH, nm) λ_{max} (Δε) 396.5 (+0.60), 382.0 (+0.68), 272.5 (+0.32), 231.0 (–1.55), 223.5 (–1.66), 201.5 (-0.58); IR (dried film) ν_{max} 2924, 2853, 1711, 1630, 1562, 1449, 1281, 759 cm^{-1}; ^{1}H NMR ($CDCl_3$, 800.1 MHz, referenced to the solvent residual peak at δ 7.26[24]): 7.71 d (J = 15.2 Hz, H-2), 7.57 d (J = 8.0 Hz, H-2′ and H-6′), 7.41 m (H-3′–H-5′), 7.04 d (J = 16.0 Hz, H-3), 6.72 m (H-7), 6.21 dd (J = 10.4, 3.2 Hz, H-8), 2.68 m (H-6 and H-11), 2.48 m (H-6 and H-11); positive-ion sodiated ESIMS *m*/*z* 307.1.

(+)-Cryptocaryanones A (**7**): Amorphous colorless powder. $[\alpha]^{20}_{D}$ +20 (*c* 0.05, MeOH); UV (MeOH) λ_{max} (log ε) 319 (3.38) nm; ECD (MeOH, nm) λ_{max} (Δε) 357.5 (–0.48), 313.5 (+0.71), 240.0 (+0.28), 226.0 (–0.56), 204.5 (+0.98); IR (dried film) ν_{max} 2923, 2852, 1783, 1731, 1659, 1591, 1427, 1172, 761 cm^{-1}; ^{1}H and ^{13}C NMR data, see Tables S2 and S3; positive-ion sodiated HRESIMS *m*/*z* 305.0800 (calcd for $C_{17}H_{14}O_4Na$, 305.0790).

(±)-5-Hydroxy-7-methoxyflavanone (**S1**): Amorphous colorless powder; $[\alpha]^{20}_{D}$ 0 (*c* 0.06, MeOH); UV (MeOH) λ_{max} (log ε) 287 (3.87), 212 (4.04) nm; ECD (MeOH, nm) λ_{max} (Δε) No

obvious CE values observed in CD spectrum; IR (dried film) ν_{max} 1644, 1575, 1499, 1445, 1091, 827, 741 cm^{-1}; ^{1}H and ^{13}C NMR data, see Tables S2 and S3; positive-ion sodiated HRESIMS *m*/*z* 293.0784 (calcd for $C_{16}H_{14}O_4Na$, 293.0790).

(±)-5,7-Dihydroxyflavanone (**S2**): Amorphous colorless powder; $[\alpha]^{20}_{D}$ 0 (*c* 0.1, MeOH); UV (MeOH) λ_{max} (log ε) 288 (3.77), 210 (3.93) nm; ECD (MeOH, nm) λ_{max} (Δε) No obvious CE values observed in CD spectrum; IR (dried film) ν_{max} 3091, 1633, 1586, 1488, 1090, 825, 768 cm^{-1}; ^{1}H and ^{13}C NMR data, see Tables S2 and S3; positive-ion sodiated HRESIMS *m*/*z* 279.0612 (calcd for $C_{15}H_{12}O_4Na$, 279.0633).

Cinnamic acid (**S3**): Amorphous colorless powder; UV (MeOH) λ_{max} (log ε) 272 (3.80), 215 (3.69) nm; IR (dried film) ν_{max} 1687, 1633, 1581, 1552, 1495, 1450, 1287, 873, 772 cm^{-1}; ^{1}H and ^{13}C NMR data, see Tables S2 and S3; positive-ion sodiated HRESIMS *m*/*z* 171.0426 (calcd for $C_9H_8O_2Na$, 171.0422).