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Supplementary Information

RP TLC-Based Lipophilicity Assessment of Some Natural and Synthetic Coumarins

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General

Optical rotation: Rudolph Research Analytical AUTOPOL IV Automatic Polarimeter.

UV spectra: GBC Cintra 40 UV-Visible spectrometer. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra: at 500 and 125 MHz, respectively (Bruker Avance III 500 spectrometer), with $CDCl_3$ as solvent and TMS as reference; EIMS (75 eV): Agilent 7890A GC/MS system equipped with a 5975 MSD detector; HR ESI MS: Agilent Technologies 6210 Time-of-flight LC/MS system.

Preparative high performance liquid chromatography (HPLC): Agilent Technologies 1100 LC system equipped with a 1200 series fraction collector.

Anomalin (**1**)

White powder; $[\alpha]_D^{20}$ +8.0° (*c* 0.1, MeOH); UV (ACN/H_2O) λ_{max}/nm 204, 218, 254, 296 *sh*, 324; EI MS *m/z* (rel. int.) 327 $[M + H\text{-AngOH}]^+$ (30), 311 (30), 243 (10), 229 (50), 213 (25), 83 (100), 55 (40); HR ESI MS *m/z* 875.3271 $[2M + Na]^+$, 490.1854 $[M + NH_4 + HCOOH]^+$, 449.1594 $[M + Na]^+$, 444.2039 $[M + NH_4]^+$ i 327.1251 $[M + H\text{-AngOH}]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

Isopteryksin (**2**)

$[\alpha]_D^{20}$ +1° (*c* 0.2, MeOH); UV (ACN/H_2O) λ_{max}/nm 204, 218, 254 *sh*, 294 *sh*, 322; EI MS *m/z* (rel. int.) 386 $[M]^+$ (8), 311 (8), 286 (10), 261 (5), 244 (35), 229 (100), 213 (25), 191 (15), 83 (60), 55 (25); HR ESI MS *m/z* 795.2619 $[2M + Na]^+$, 450.1533 $[M + NH_4 + HCOOH]^+$, 409.1269 $[M + Na]^+$, 404.1715 $[M + NH_4]^+$, 327.1239 $[M + H\text{-AngOH}]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

Isolaserpitin (**3**)

$[\alpha]_D^{20}$ –63° (*c* 0.1, MeOH); UV (ACN/H_2O) λ_{max}/nm 204, 220, 248 *sh*, 296 *sh*, 324; EI MS *m/z* (rel. int.) 344 $[M]^+$ (2), 311 (10), 244 (70), 229 (15), 213 (55), 201 (100), 173 (25) 83 (45), 55 (30); HR ESI MS *m/z* 711.2411 $[2M + Na]^+$, 362.1607 $[M + NH_4]^+$, 327.1282 $[M + H\text{-}H_2O]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

Laserpitin (**4**)

$[\alpha]_D^{20}$ +92° (*c* 0.1, MeOH); UV (ACN/H_2O) λ_{max}/nm 204, 218, 246 *sh*, 292 *sh*, 324; EI MS *m/z* (rel. int.) 344 $[M]^+$ (15), 311 (10), 244 (75), 229 (35), 213 (30), 201 (100), 173 (30) 83 (90), 55 (35); HR ESI MS *m/z* 711.2432 $[2M + Na]^+$, 362.1623 $[M + NH_4]^+$, 245.0831 $[M + H\text{-AngOH}]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

Meranzin (**5**)

$[\alpha]_D^{20}$ +34° (*c* 0.06, MeOH); UV (ACN/H_2O) λ_{max}/nm 202, 220 *sh*, 246 *sh*, 322; HR ESI MS *m/z* 278.1401 $[M + NH_4]^+$, 261.1146 $[M + H]^+$, 343.1028 $[M + H\text{-}H_2O]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

Phellopterin (**6**)

UV (MeOH) λ_{max}/nm 222, 248, 268, 312; HR ESI MS *m/z* 301.1190 $[M + H]^+$, 623.1862 $[2M + Na]^+$; ^{1}H NMR in Table S1; ^{13}C NMR in Table S2.

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Table S1. ^{1}H NMR data of compounds **1**-**4** at 500 MHz in $CDCl_3$ (δ in ppm, mult., *J* in Hz)

H	Anomalin **1**	Isopteryksin **2**	Isolaserpitin **3**	Laserpitin **4**	Meranzin **5**	Phellopterin **6**
3	6.22 d (9.4)	6.23 d (9.5)	6.25 d (9.5)	6.22 d (9.4)	6.26 d (9.5)	6.28 d (9.8)
4	7.58 d (9.4)	7.60 d (9.5)	7.64 d (9.5)	7.60 d (9.4)	7.64 d (9.5)	8.12 d (9.8)
5	7.35 d (8.5)	7.35 d (8.6)	7.34 d (8.6)	7.35 d (8.6)	7.36 d (8.6)	–
6	6.81 d (8.5)	6.80 d (8.6)	6.79 d (8.6)	6.80 d (8.6)	6.87 d (8.6)	–
1'a	–	–	–	–	3.21 dd (12.6;4.4)	–
1'b	–	–	–	–	3.02 m	–
2'	–	–	–	–	3.02 m	7.62 d (2.4)
3'	5.45 d (4.8)	5.41 d (4.8)	5.23 d (4.8)	4.09 d (4.8)	–	6.99 d (2.4)
4'	6.70 d (4.8)	6.60 d (4.8)	5.47 d (4.8)	6.49 d (4.8)	1.50 s	–
5'	1.49 s	1.48 s	1.51 s	1.49 s	1.39 s	–
1'	–	–	–	–	–	4.85 br d (7.2)
2'	–	–	–	–	–	5.61 tqq (7.2; 1.4)
6'	1.46 s	1.44 s	1.44 s	1.44 s	–	–
3''	6.12 qq (7.4; 1.4)	6.13 qq (7.3; 1.5)	6.16 qq (7.3; 1.5)	6.16 qq (7.3; 1.5)	–	–
4''	1.96 dq (7.4; 1.4)	1.96 dq (7.4; 1.5)	2.00 dq (7.3; 1.5)	2.00 dq (7.3; 1.4)	–	1.74 br s
5''	1.85 sept (1.4)	1.85 sept (1.5)	1.94 sept (1.5)	1.89 sept (1.5)	–	1.70 br s
2'''	–	2.11 s	–	–	–	–
3'''	6.03 qq (7.2; 1.4)	–	–	–	–	–
4'''	1.98 dq (7.2; 1.4)	–	–	–	–	–
5'''	1.83 sept (1.4)	–	–	–	–	–
–OH	–	–	3.11 br s	2.90 br s	–	–
$-OCH_3$	–	–	–	–	3.94 s	4.18 s

Table S2. ^{13}C NMR data of compounds **1**-**4** at 125 MHz in $CDCl_3$ (δ in ppm)

C	Anomalin **1**	Isopteryksin **2**	Isolaserpitin **3**	Laserpitin **4**	Meranzin **5**	Phellopterin **6**
2	159.7	159.9	160.5	159.9	161.0	160.5
3	113.2	113.2	112.6	113.0	113.2	112.8
4	143.1	143.3	143.3	143.3	143.7	139.4
5	129.1	129.1	128.7	129.3	127.1	144.3
6	114.3	114.3	114.6	114.5	107.4	114.5
7	156.7	156.7	156.0	157.0	160.7	150.8
8	107.5	107.0	110.8	107.1	114.3	126.9
9	154.0	154.0	154.4	154.2	153.4	144.8
10	112.4	112.5	112.4	112.3	113.0	107.6
1'	-	-	-	-	22.5	-
2'	77.4	77.1	77.6	78.6	63.0	145.1
3'	70.1	69.8	72.4	71.6	59.3	105.0
4'	60.1	61.0	60.0	63.4	19.1	-
5'	22.5	23.0	25.7	25.6	24.8	-
6'	25.4	24.9	22.6	20.9	-	-
1''	166.2	166.4	167.0	169.1	-	70.4
2''	127.4	127.0	127.2	127.3	-	119.8
3''	138.4	139.8	139.6	139.1	-	139.6
4''	15.5	15.8	15.8	15.8	-	25.8
5''	20.4	20.5	20.6	20.4	-	18.1
1'''	166.4	169.8	-	-	-	-
2'''	127.0	20.6	-	-	-	-
3'''	139.8	-	-	-	-	-
4'''	15.7	-	-	-	-	-
5'''	20.3	-	-	-	-	-
$-OCH_3$	-	-	-	-	56.2	60.8

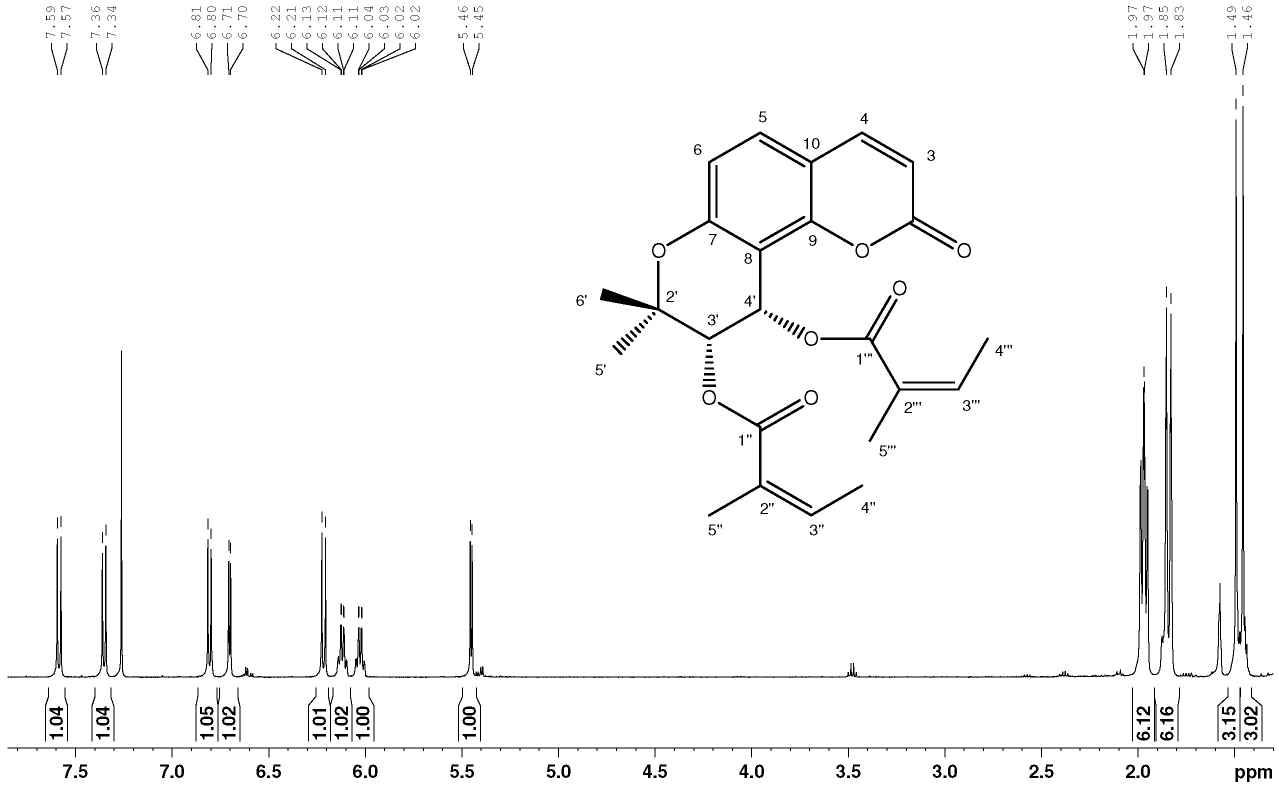

Figure S1. [1]H NMR spectrum of the compound **1** (anomalin).

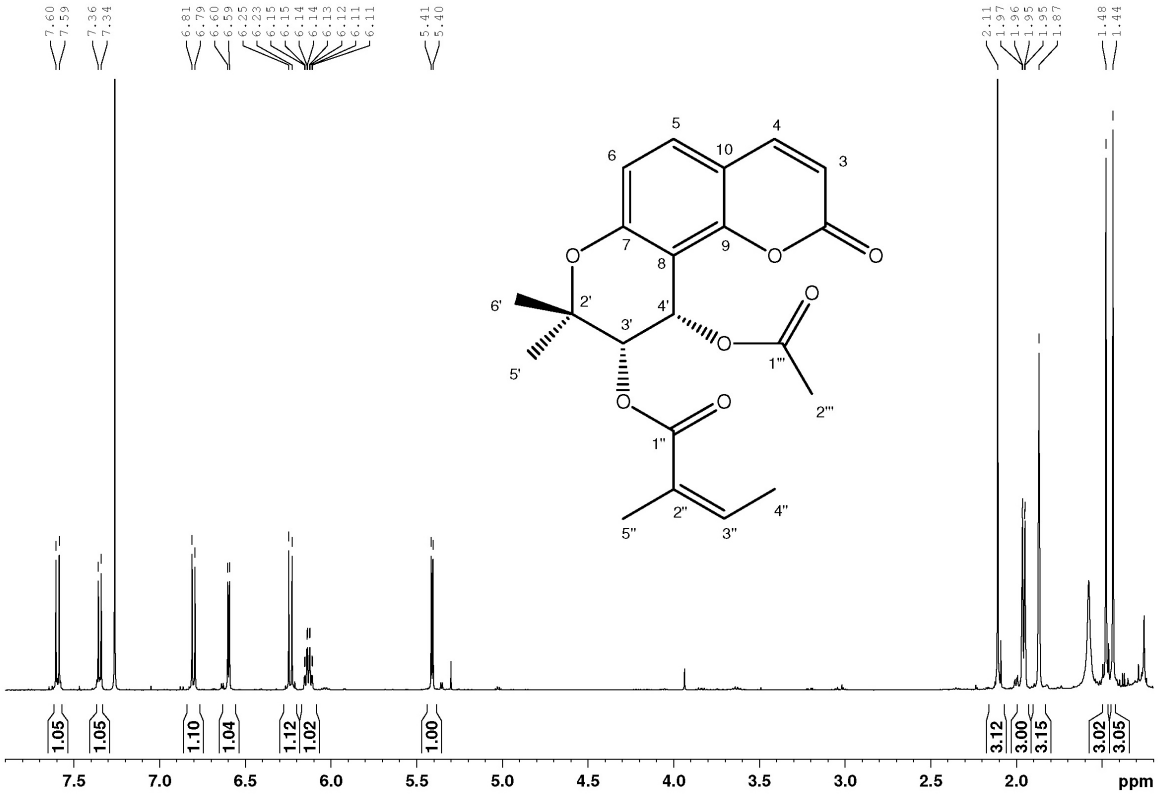

Figure S2. [1]H NMR spectrum of the compound **2** (isopteryxin).

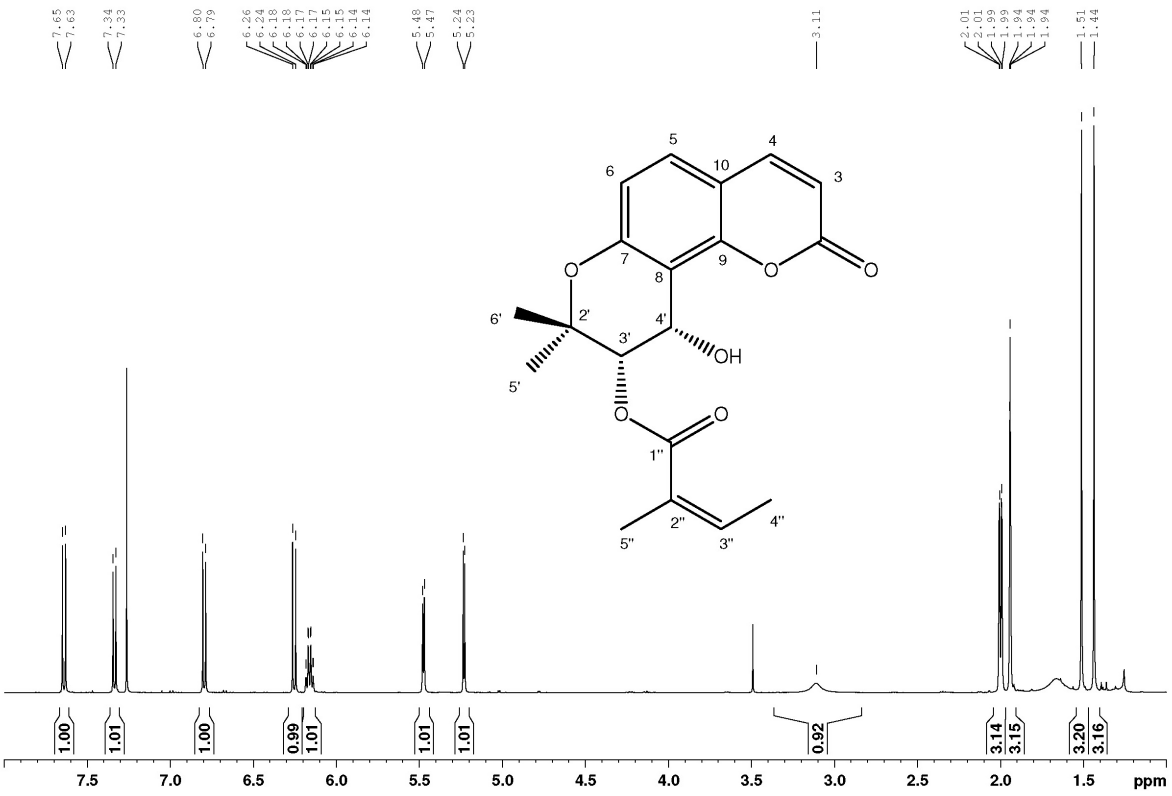

Figure S3. [1]H NMR spectrum of the compound **3** (isolaserpitin).

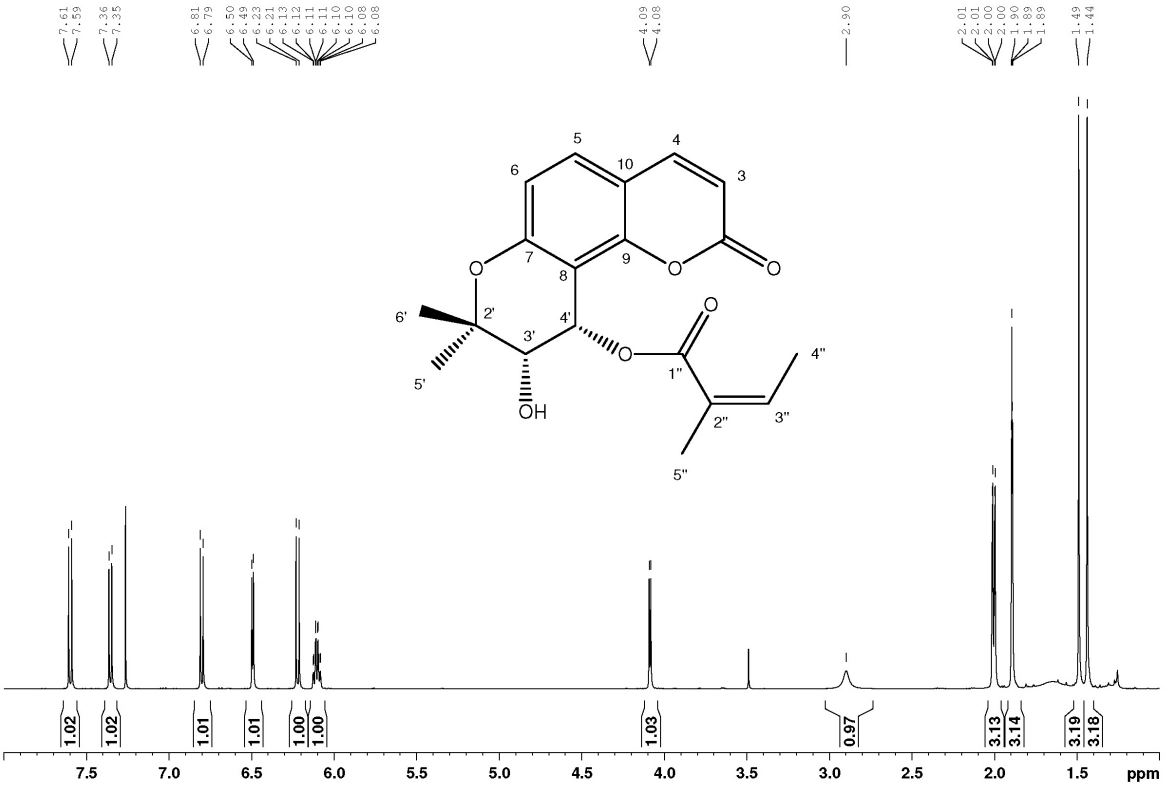

Figure S4. [1]H NMR spectrum of the compound **4** (laserpitin).

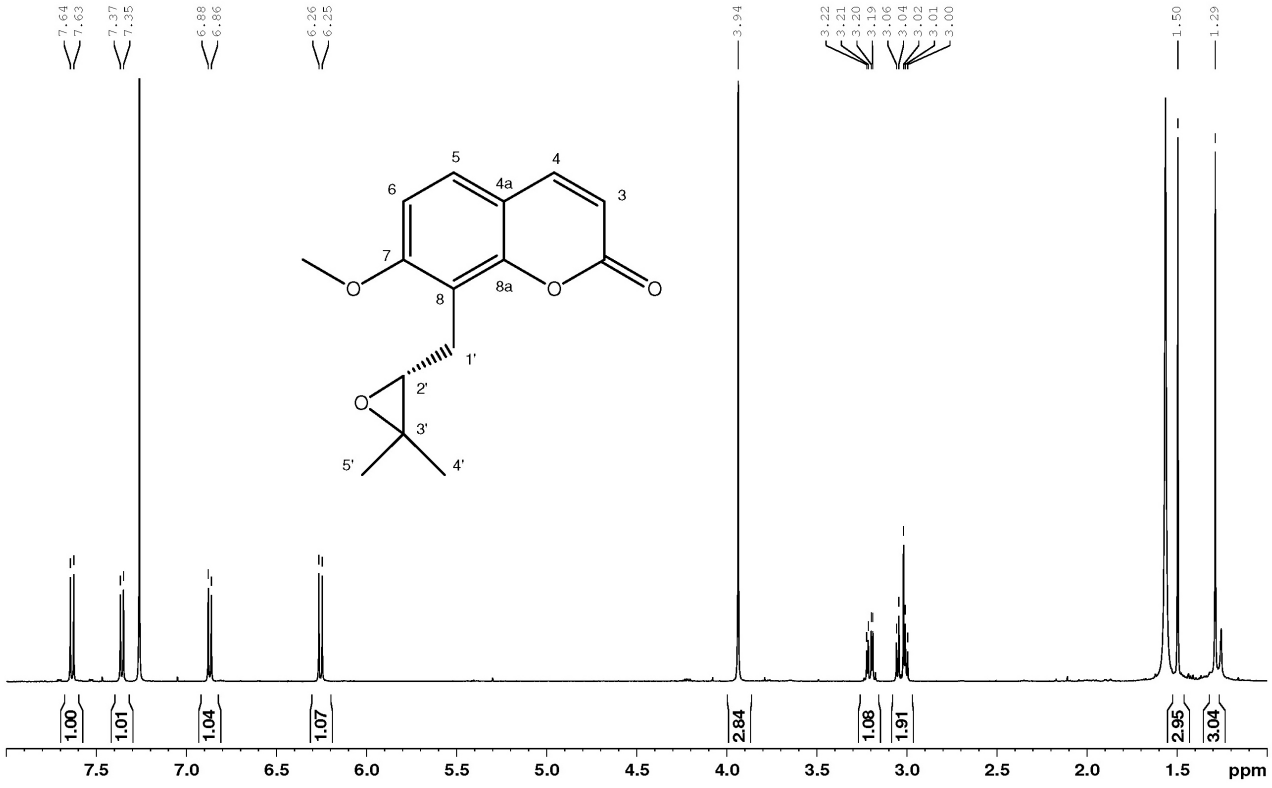

Figure S5. [1]H NMR spectrum of the compound **5** (meranzin).

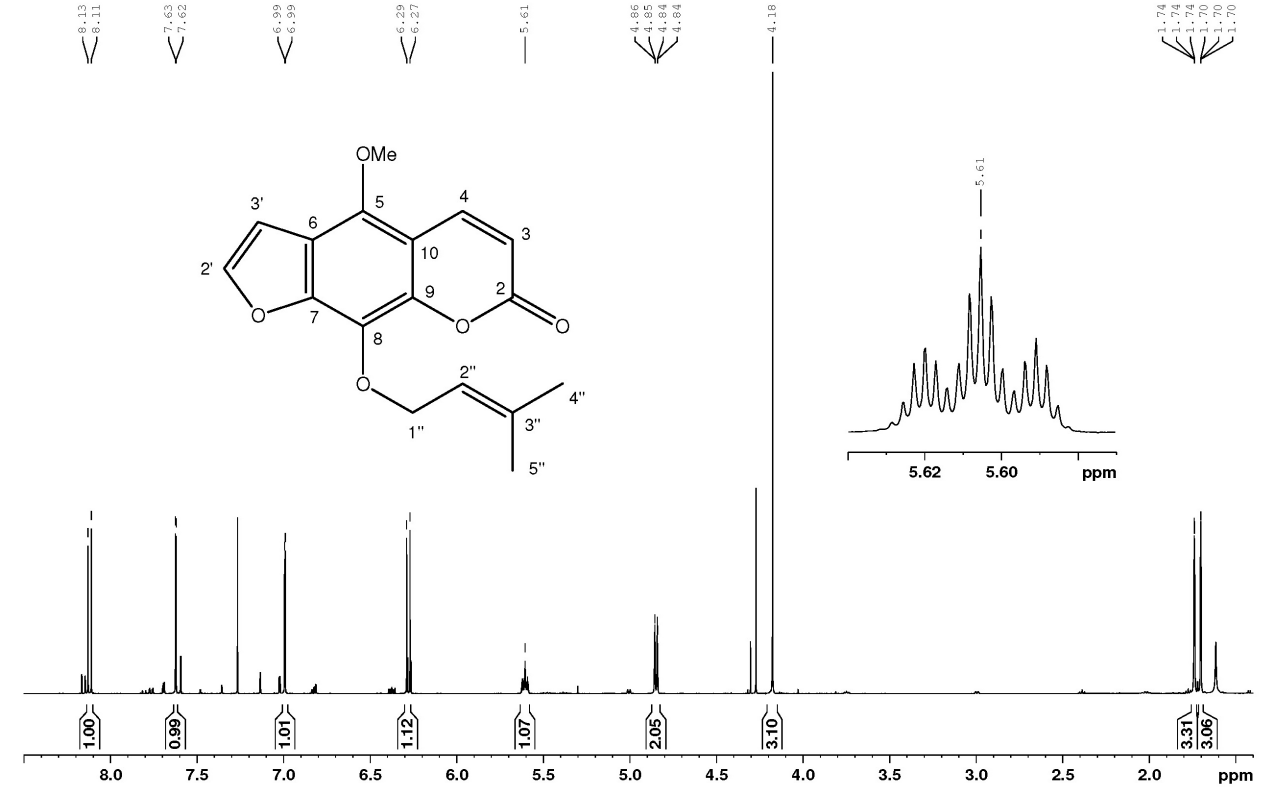

Figure S6. [1]H NMR spectrum of the compound **6** (meranzin).