

## Supporting Information for

### Quorumolides A-C, Three Cembranoids from *Euphorbia antiquorum*

Wei-Yan Qi,<sup>†</sup> Jin-Xin Zhao,<sup>‡</sup> Wen-Jun Wei,<sup>†</sup> Kun Gao,<sup>\*,†</sup> Jian-Min Yue<sup>\*,†,‡</sup>

<sup>†</sup>State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China

<sup>‡</sup>State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Chinese Academy of Sciences, 555 Zuchongzhi Road, Shanghai 201203, People's Republic of China

**Table S1.** X-ray crystal data for quorumolide A (**1**).....S3

**Table S2.** X-ray crystal data for quorumolide B (**2**).....S4

**Figure S1.** Key (a) <sup>1</sup>H-<sup>1</sup>H COSY (—), HMBC (H→C), and (b) ROESY correlations of **2**.....S5

**Figure S2.** Key (a) <sup>1</sup>H-<sup>1</sup>H COSY (—), HMBC (H→C), and (b) ROESY correlations of **3**.....S6

**Figure S3.** CD and UV spectra of compounds **2** and **3**.....S7

**Figure S4.** <sup>1</sup>H NMR spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S8

**Figure S5.** <sup>13</sup>C NMR spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S9

**Figure S6.** HSQC spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S10

**Figure S7.** HMBC spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S11

**Figure S8.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S12

**Figure S9.** ROESY spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.....S13

**Figure S10.** HRMS (ESI-TOF) spectrum of quorumolide A (**1**).....S14

**Figure S11.** IR spectrum of quorumolide A (**1**).....S15

**Figure S12.** <sup>1</sup>H NMR spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.....S16

**Figure S13.** <sup>13</sup>C NMR spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.....S17

**Figure S14.** HSQC spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.....S18

<b>Figure S15.</b> HMBC spectrum of quorumolide B ( <b>2</b> ) in CDCl <sub>3</sub> .....	S19
<b>Figure S16.</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of quorumolide B ( <b>2</b> ) in CDCl <sub>3</sub> .....	S20
<b>Figure S17.</b> ROESY spectrum of quorumolide B ( <b>2</b> ) in CDCl <sub>3</sub> .....	S21
<b>Figure S18.</b> HRMS (ESI-TOF) spectrum of quorumolide B ( <b>2</b> ).....	S22
<b>Figure S19.</b> IR spectrum of quorumolide B ( <b>2</b> ).....	S23
<b>Figure S20.</b> <sup>1</sup> H NMR spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S24
<b>Figure S21.</b> <sup>13</sup> C NMR spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S25
<b>Figure S22.</b> HSQC spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S26
<b>Figure S23.</b> HMBC spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S27
<b>Figure S24.</b> <sup>1</sup> H- <sup>1</sup> H COSY spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S28
<b>Figure S25.</b> ROESY spectrum of quorumolide C ( <b>3</b> ) in CDCl <sub>3</sub> .....	S29
<b>Figure S26.</b> HRMS (ESI-TOF) spectrum of quorumolide C ( <b>3</b> ).....	S30
<b>Figure S27.</b> IR spectrum of quorumolide C ( <b>3</b> ).....	S31
<b>Figure S28.</b> ORTEP diagram of <b>1</b> .....	S32
<b>Figure S29.</b> ORTEP diagram of <b>2</b> .....	S33

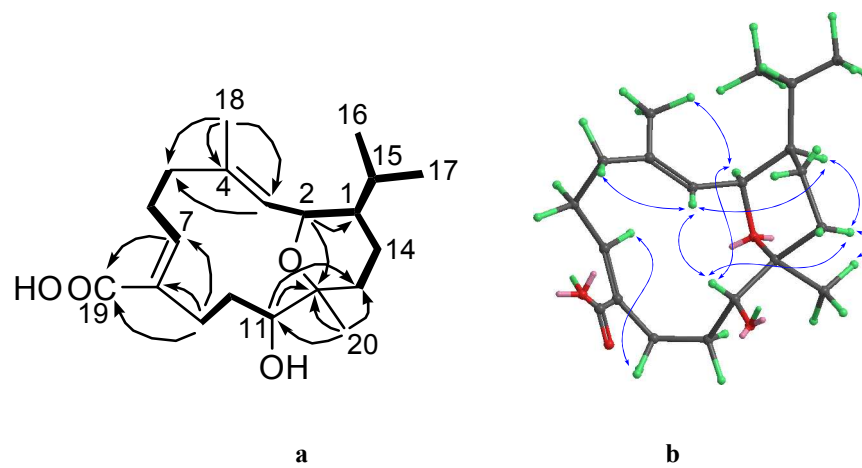
**Table S1. X-ray crystal data for quorumolide A (1) <sup>A</sup>**

Empirical formula	C <sub>20</sub> H <sub>30</sub> O <sub>4</sub>
Formula weight	336.464.44
Temperature	293 (2) K
Wavelength	1.54178 Å
Crystal system, space group	Monoclinic, P2 (1)
Unit cell dimensions	a = 6.14420 (10) Å, α = 90° b = 11.10470 (10) Å, β = 91.6320 (10)° c = 13.24270 (10) Å, γ = 90°
Volume	903.176 (18) Å <sup>3</sup>
Z, calculated density	2, 1.230 Mg/m <sup>3</sup>
Absorption coefficient	0.672 mm <sup>-1</sup>
F (000)	364
Crystal size	0.30 × 0.05 × 0.04 mm
Theta range for data collection	3.34 to 68.98°
Limiting indices	-7 ≤ h ≤ 5, -13 ≤ k ≤ 13, -15 ≤ l ≤ 15
Reflections collected / unique	11695 / 3109 [R (int) = 0.0283]
Completeness to theta = 68.98	96.7%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9736 and 0.8239
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	3109 / 1 / 222
Goodness-of-fit on F <sup>2</sup>	1.064
Final R indices [I > 2σ (I)]	R1 = 0.0339, wR2 = 0.0900
R indices (all data)	R1 = 0.0346, wR2 = 0.0907
Absolute structure parameter	-0.10 (17)
Largest diff. peak and hole	0.127 and -0.163 e. Å <sup>-3</sup>
<sup>A</sup> Colorless crystals of 1 were obtained in the solvent of MeOH	

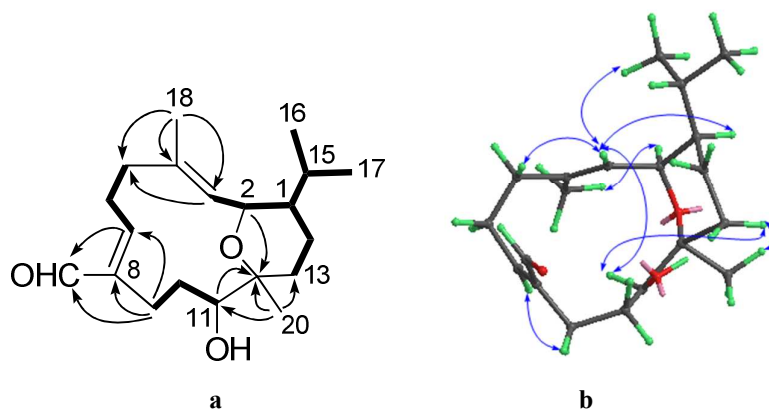
**Table S2. X-ray crystal data for quorumolide B (2)**

Empirical formula	C <sub>20</sub> H <sub>32</sub> O <sub>4</sub>
Formula weight	336.46
Temperature	140 (2) K
Wavelength	1.54178 Å
Crystal system, space group	Monoclinic, P2 (1)
Unit cell dimensions	a = 9.21880 (10) Å, α = 90° b = 10.6982 (2) Å, β = 101.6680 (10)° c = 19.9113 (3) Å, γ = 90°
Volume	1923.16 (5) Å <sup>3</sup>
Z, calculated density	4, 1.162 Mg/m <sup>3</sup>
Absorption coefficient	0.631 mm <sup>-1</sup>
F (000)	736
Crystal size	0.25 × 0.15 × 0.12 mm
Theta range for data collection	2.27 to 69.79°
Limiting indices	-10 ≤ h ≤ 11, -13 ≤ k ≤ 12, -23 ≤ l ≤ 24
Reflections collected / unique	15318 / 6186 [R (int) = 0.0369]
Completeness to theta = 69.79	97.1%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9281 and 0.8581
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	6186 / 1 / 445
Goodness-of-fit on F <sup>2</sup>	1.033
Final R indices [I > 2σ (I)]	R1 = 0.0385, wR2 = 0.1024
R indices (all data)	R1 = 0.0407, wR2 = 0.1048
Absolute structure parameter	-0.15 (14)
Largest diff. peak and hole	0.181 and -0.168 e. Å <sup>-3</sup>
<sup>A</sup> Colorless crystals of 1 were obtained in the solvent of MeOH	

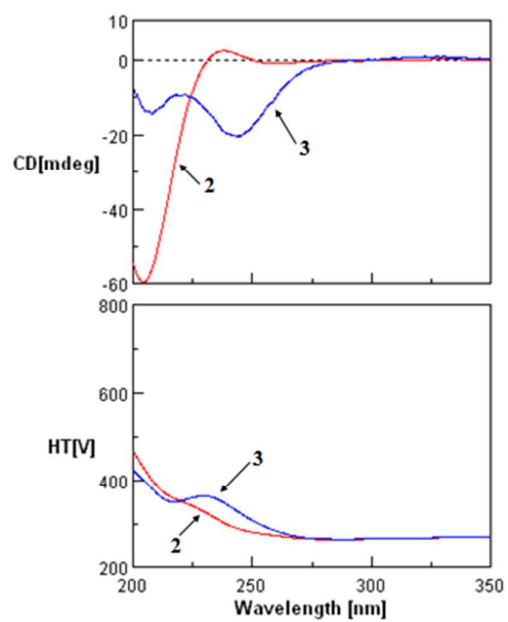
**Figure S1.** Key (a)  $^1\text{H}$ - $^1\text{H}$  COSY (—), HMBC ( $\text{H} \rightarrow \text{C}$ ), and (b) ROESY correlations of **2**



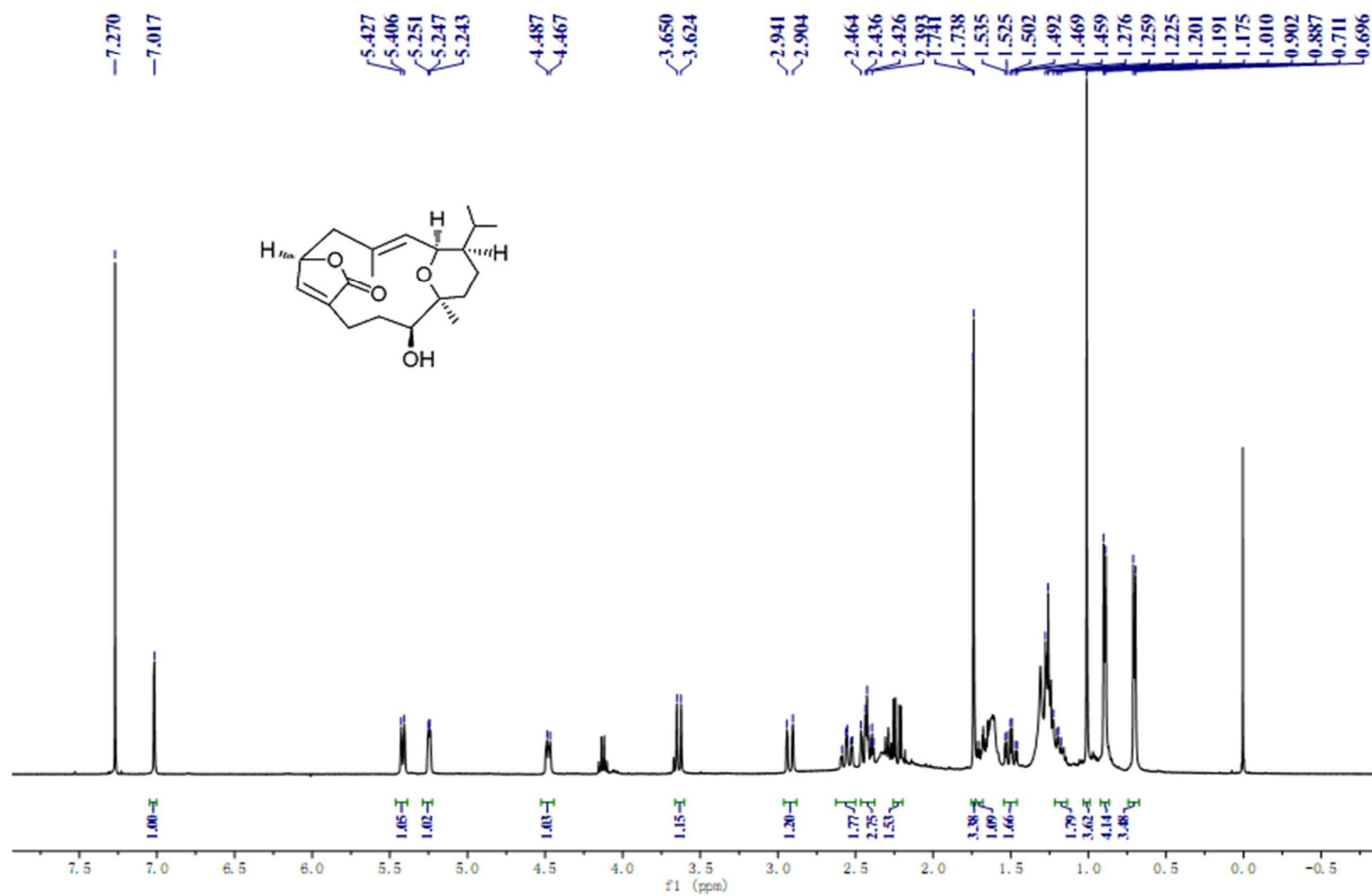
**Figure S2.** Key (a)  $^1\text{H}$ - $^1\text{H}$  COSY (—), HMBC ( $\text{H} \rightarrow \text{C}$ ), and (b) ROESY correlations of **3**



**Figure S3.** CD and UV spectra of compounds **2** and **3**.



**Figure S4.**  $^1\text{H}$  NMR spectrum of quorumolide A (**1**) in  $\text{CDCl}_3$ .

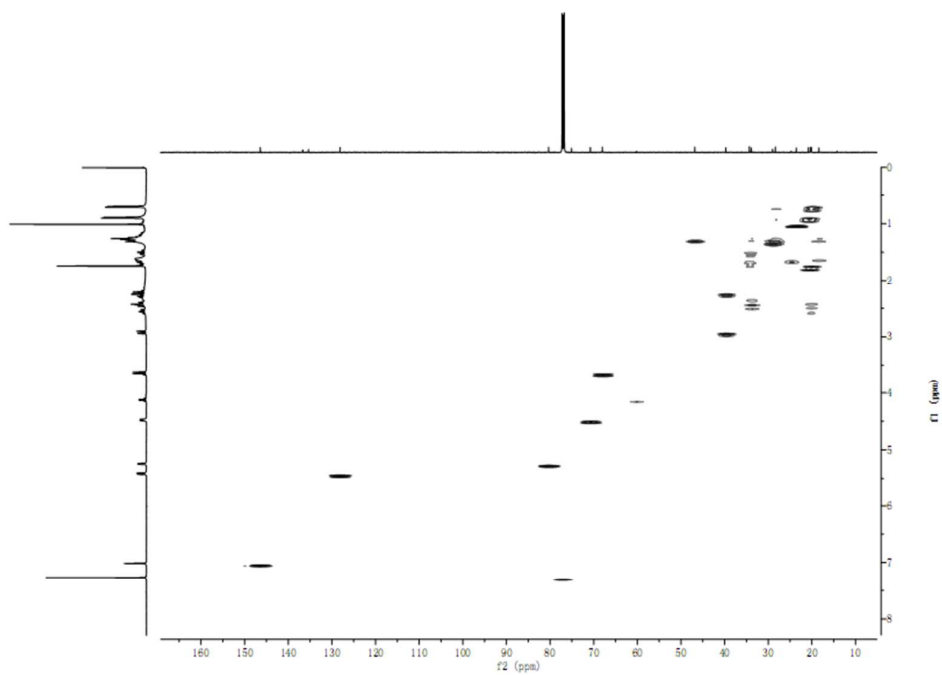




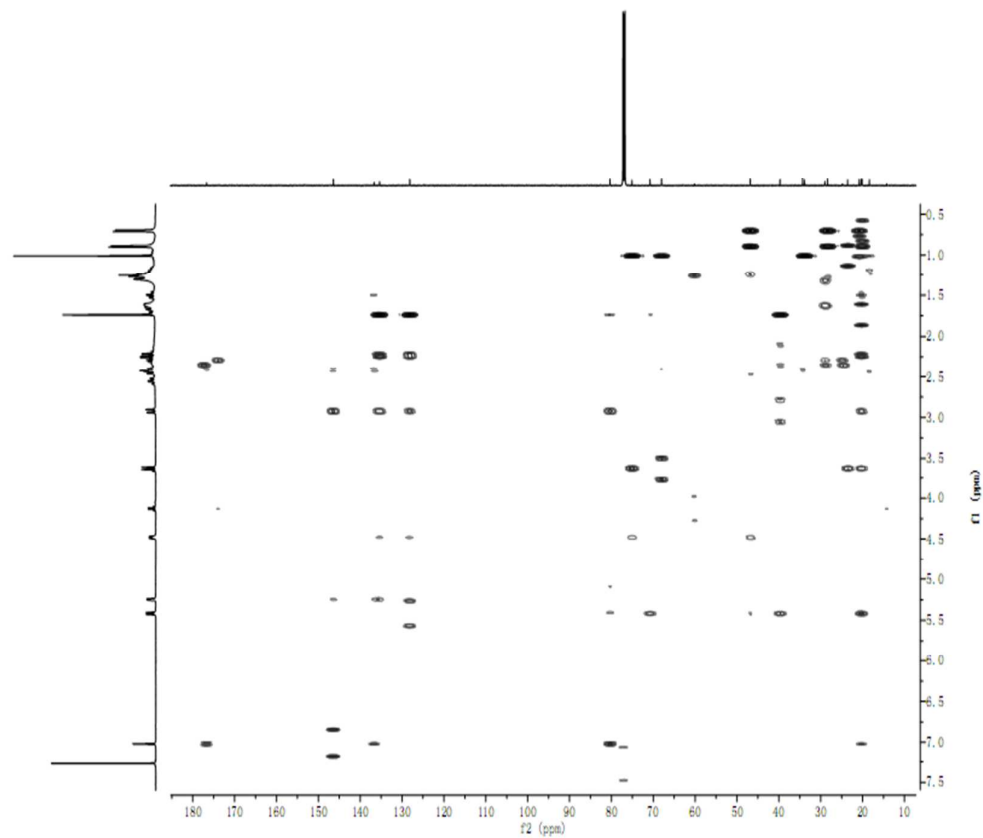
Chemical structure of compound 1 is shown as an inset. The structure is a complex polycyclic molecule with a ketone, an alcohol, and an isopropyl group. The  $^{13}\text{C}$  NMR peaks are labeled with their chemical shifts in ppm:

- 176.674
- 146.402
- 136.681
- 135.361
- 128.146
- 80.335
- 77.254
- 77.000
- 76.746
- 75.086
- 70.772
- 68.027
- 46.844
- 39.727
- 34.334
- 33.898
- 29.009
- 28.380
- 23.586
- 20.818
- 20.348
- 20.313
- 20.138
- 18.398

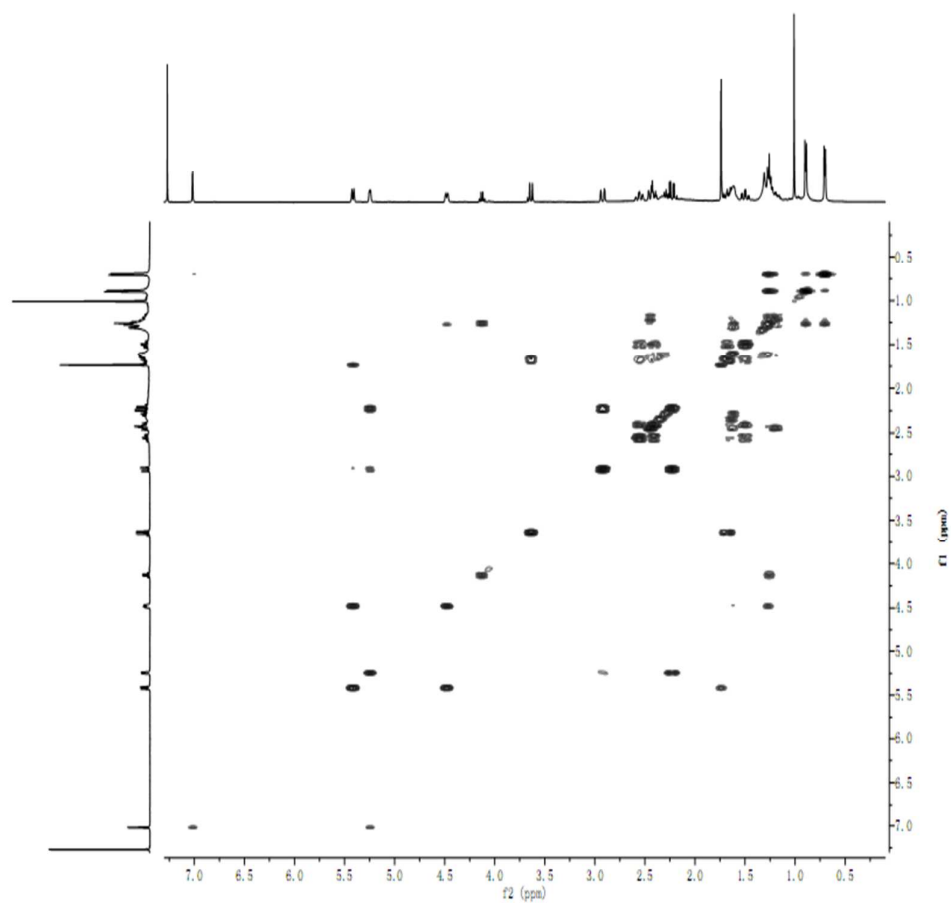
**Figure S6.** HSQC spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.



**Figure S7.** HMBC spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.



**Figure S8.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.



**Figure S9.** ROESY spectrum of quorumolide A (**1**) in CDCl<sub>3</sub>.

Eu-62 CDCL3 ROESY

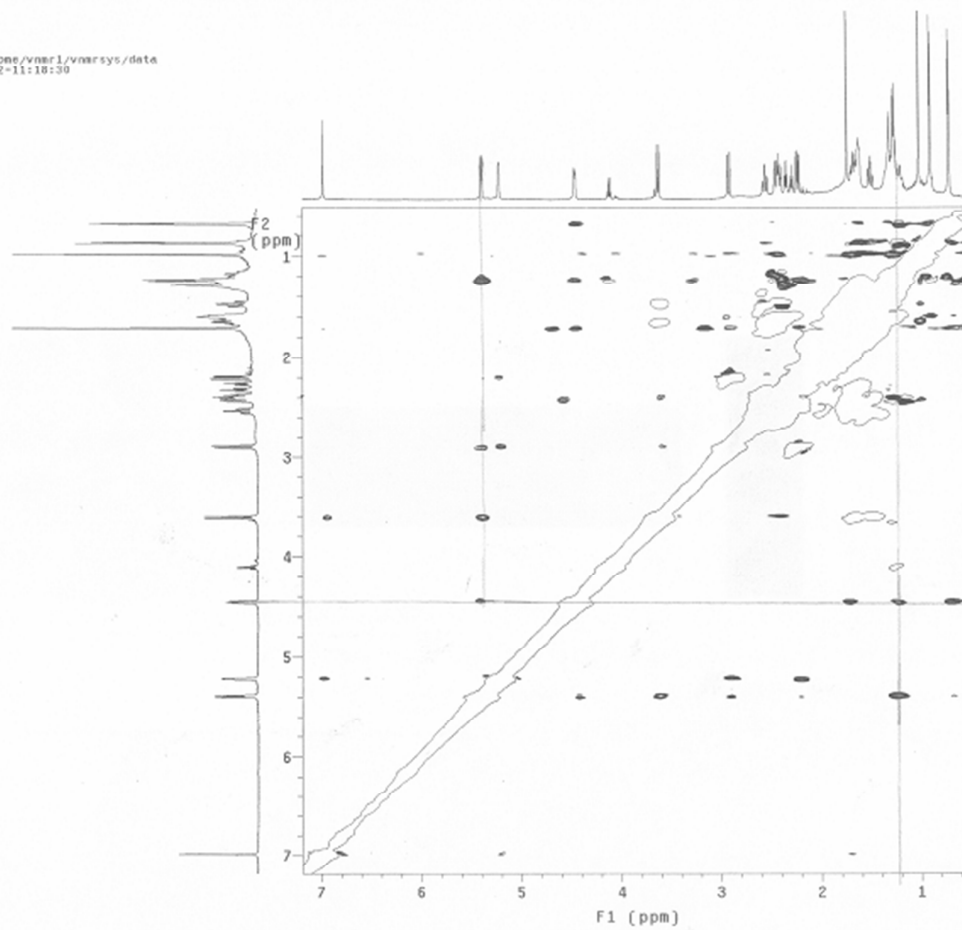
Archive directory: /export/home/vmr1/vnarsys/data  
Sample directory: 1\_30May2012-11:10:30  
File: ROESY

Pulse Sequence: ROESY

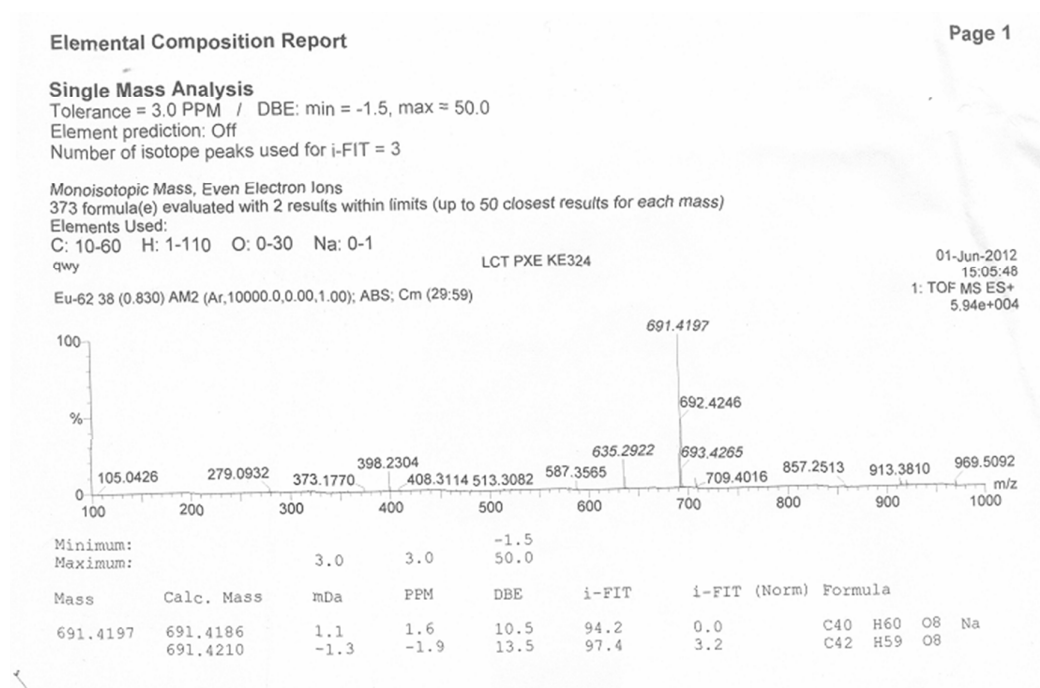
Solvent: CDCl3  
Temp: 25.0 C / 298.1 K  
INNOVA-600 "600MHz"

Relax. delay 1.000 sec  
Mixing 0.200 sec  
Acq. time 0.881 sec  
Width 12780.4 Hz  
2D Width 12780.4 Hz  
4 repetitions  
2 x 270 increments

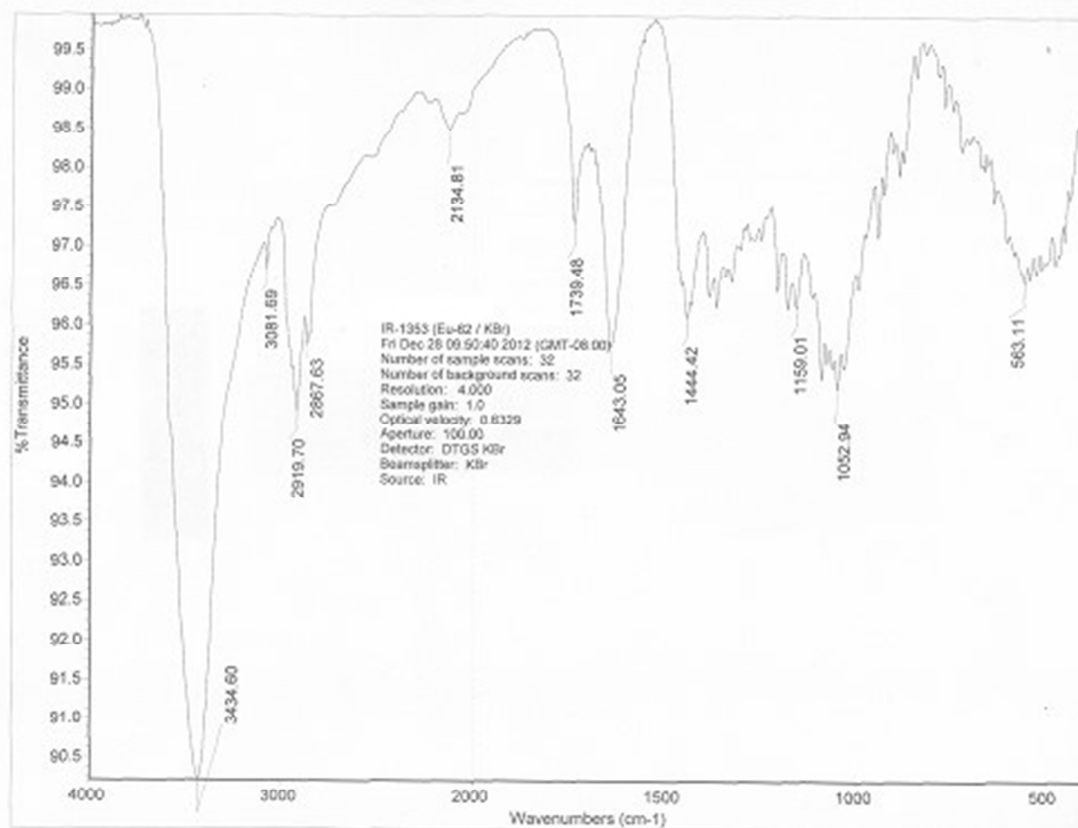
OBSERVE H1, 599.6560146 MHz  
DATA PROCESSING  
Gauss apodization 0.032 sec  
F1 DATA PROCESSING  
Gauss apodization 0.008 sec  
FT size 2048 x 2048  
Total time 47 min, 13 sec



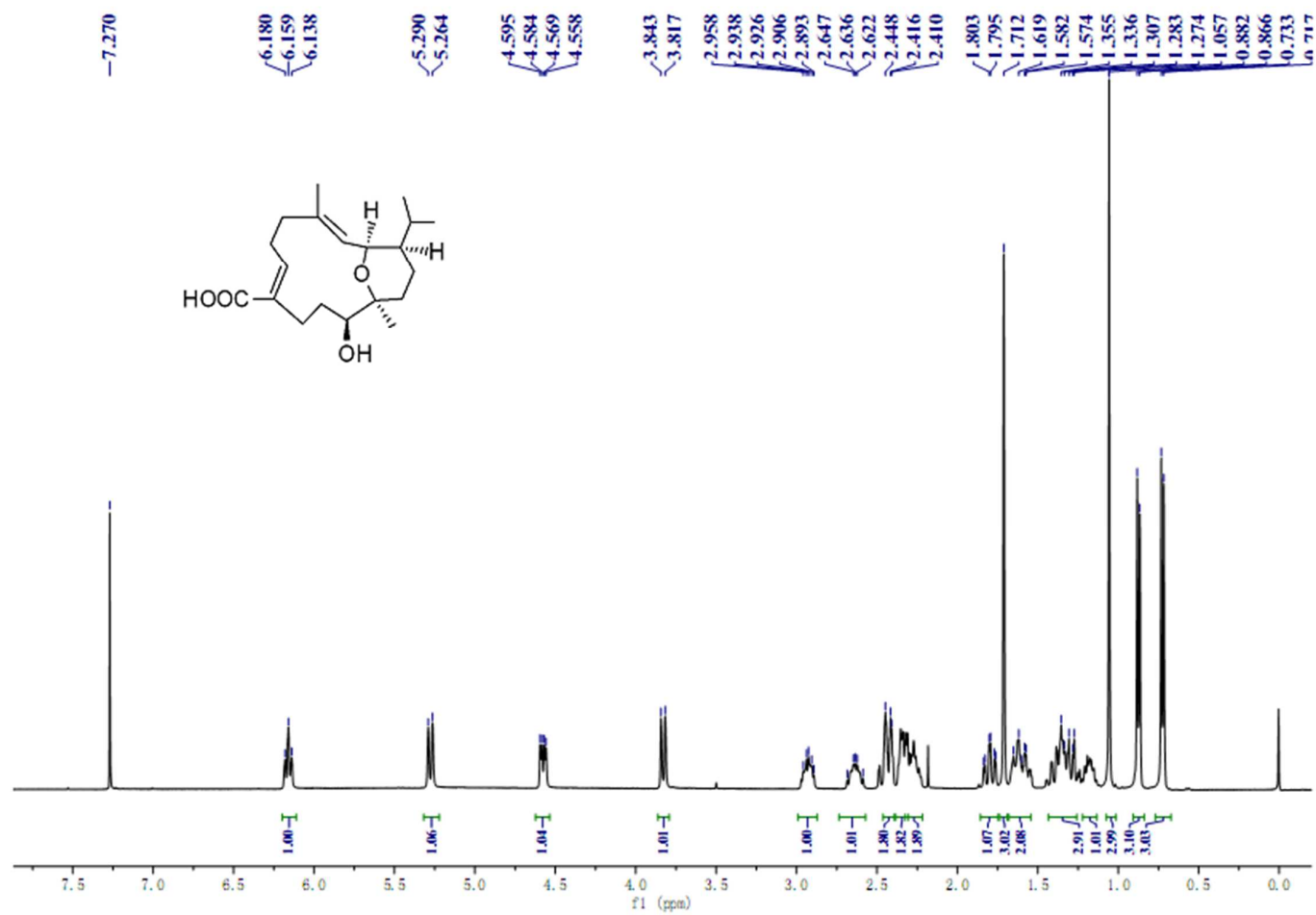
**Figure S10.** HRMS (ESI-TOF) spectrum of quorumolide A (**1**).



**Figure S11.** IR spectrum of quorumolide A (**1**).

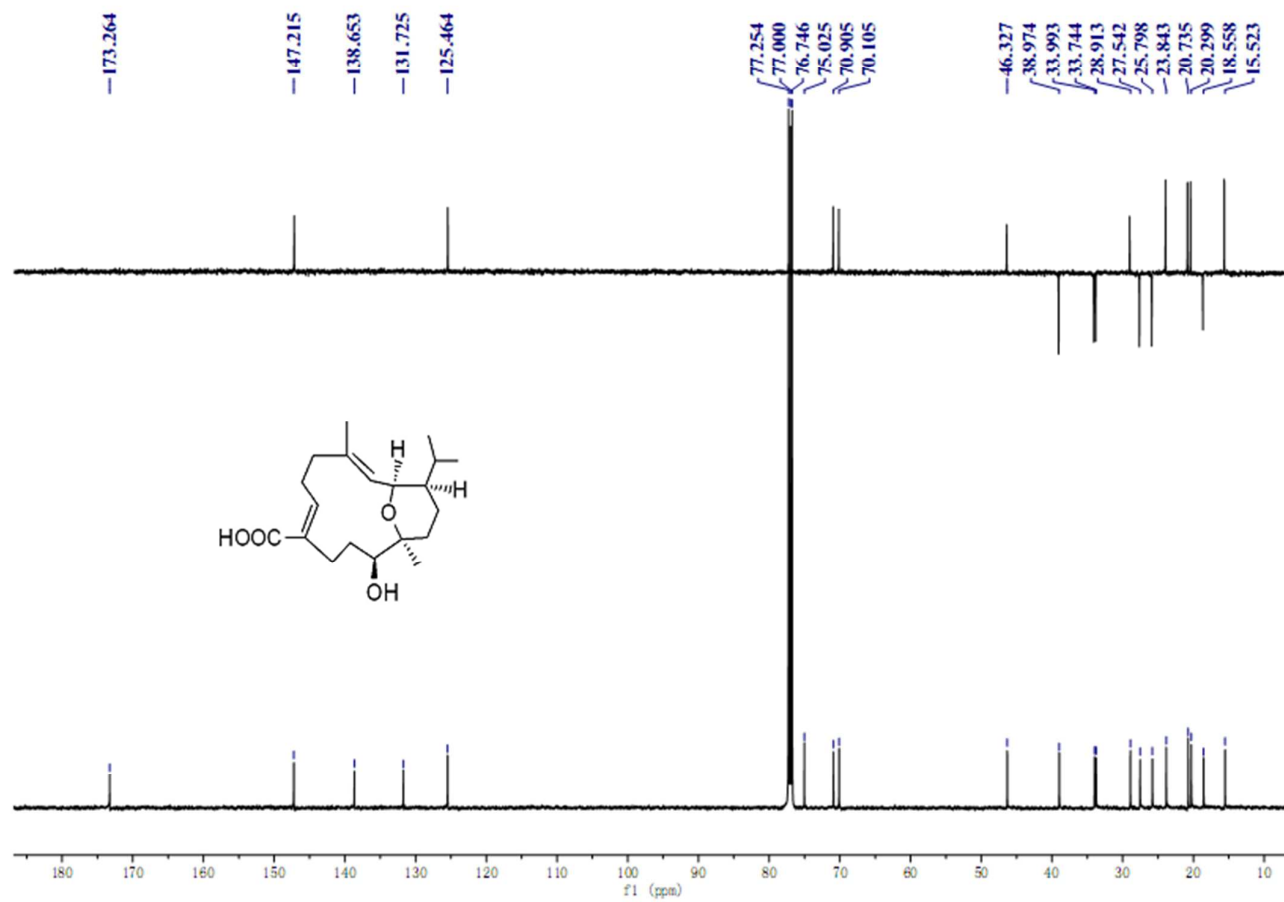


**Figure S12.**  $^1\text{H}$  NMR spectrum of quorumolide B (**2**) in  $\text{CDCl}_3$

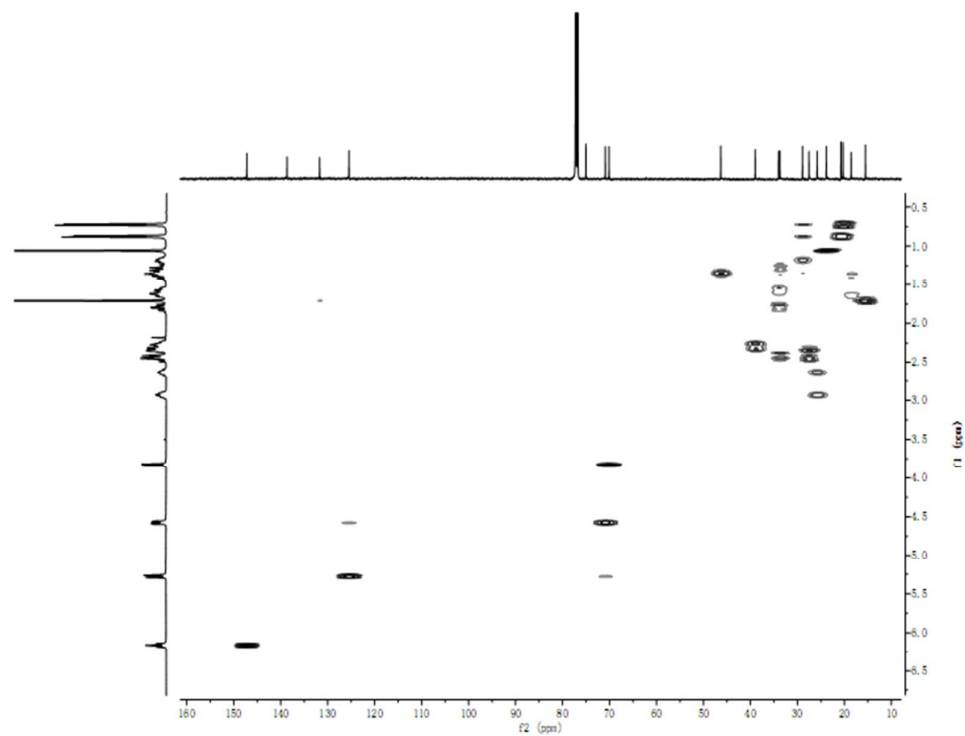




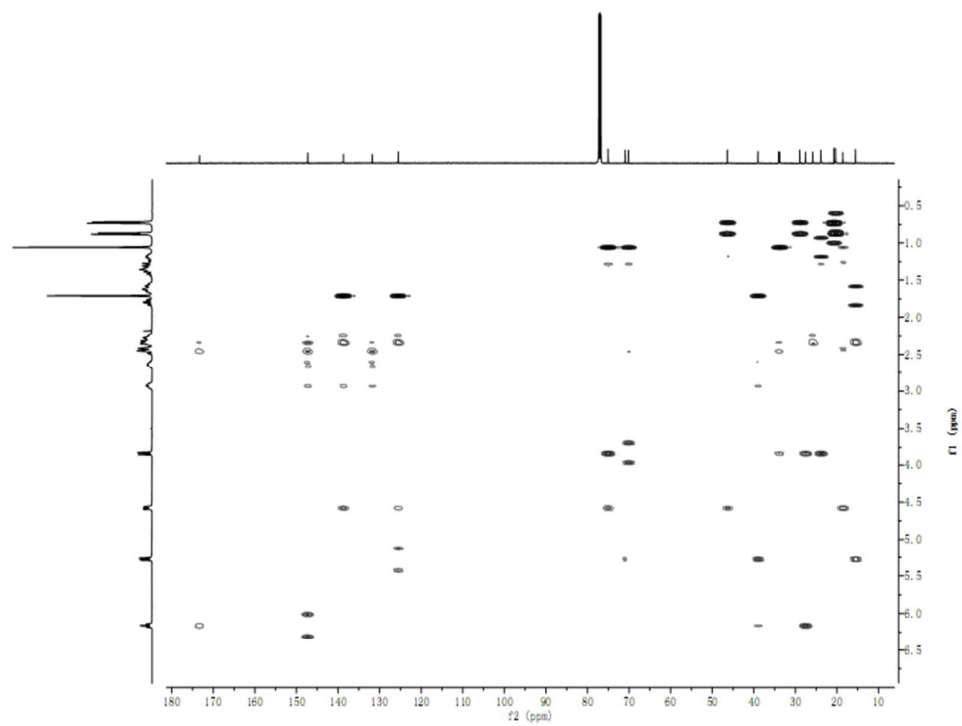
**Figure S13.**  $^{13}\text{C}$  NMR spectrum of quorumolide B (**2**) in  $\text{CDCl}_3$ .



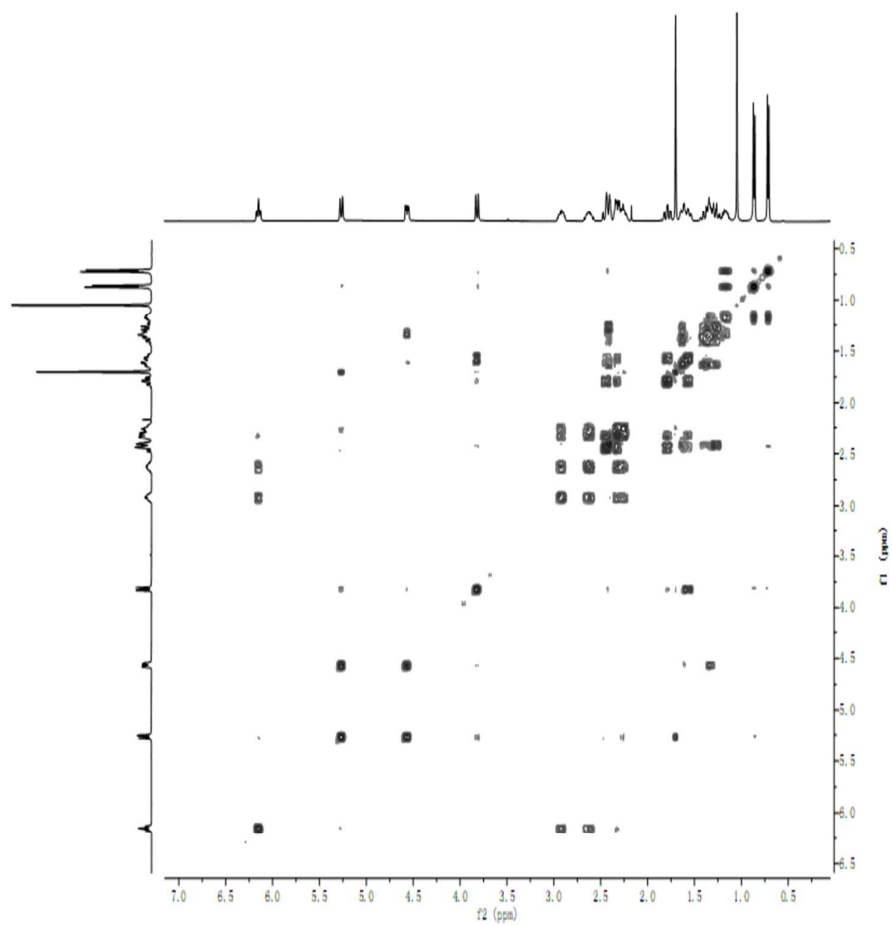
**Figure S14.** HSQC spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.



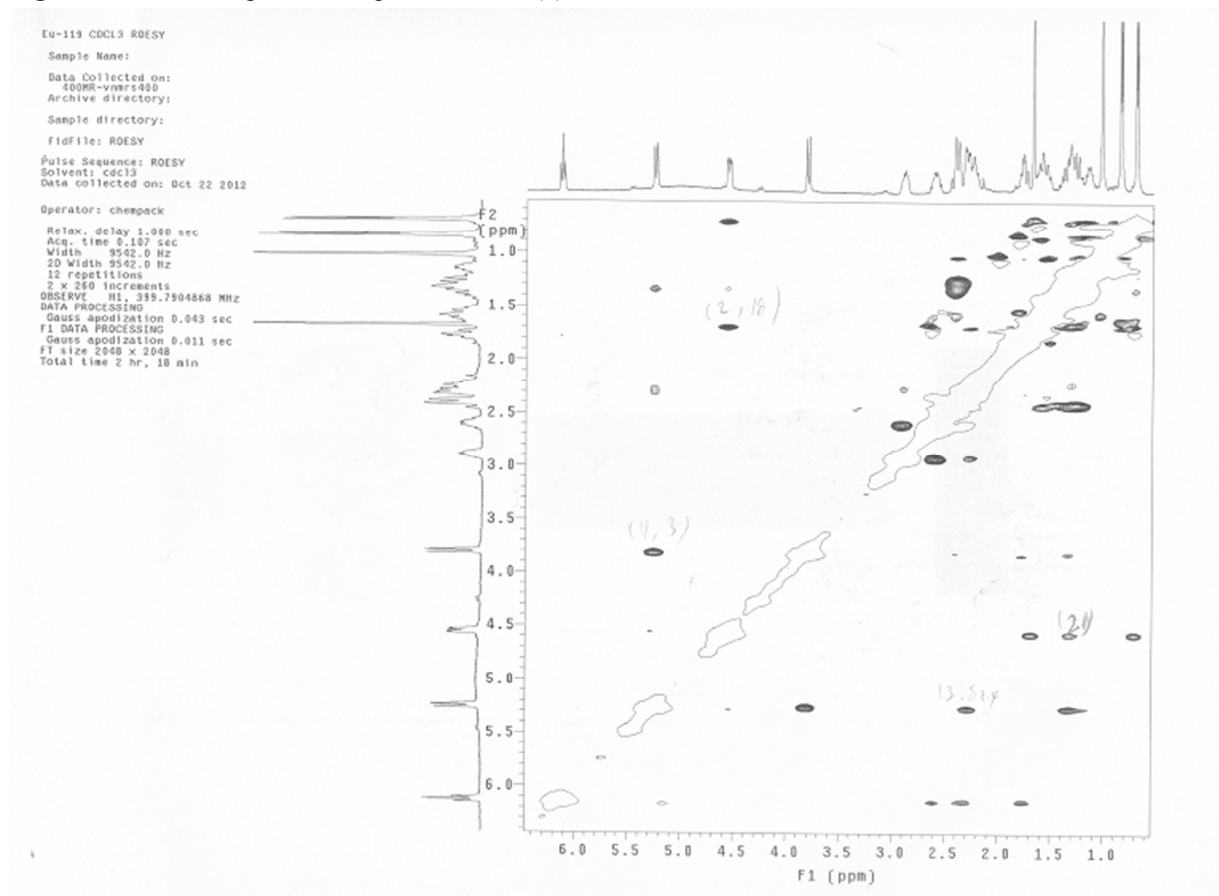
**Figure S15.** HMBC spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.



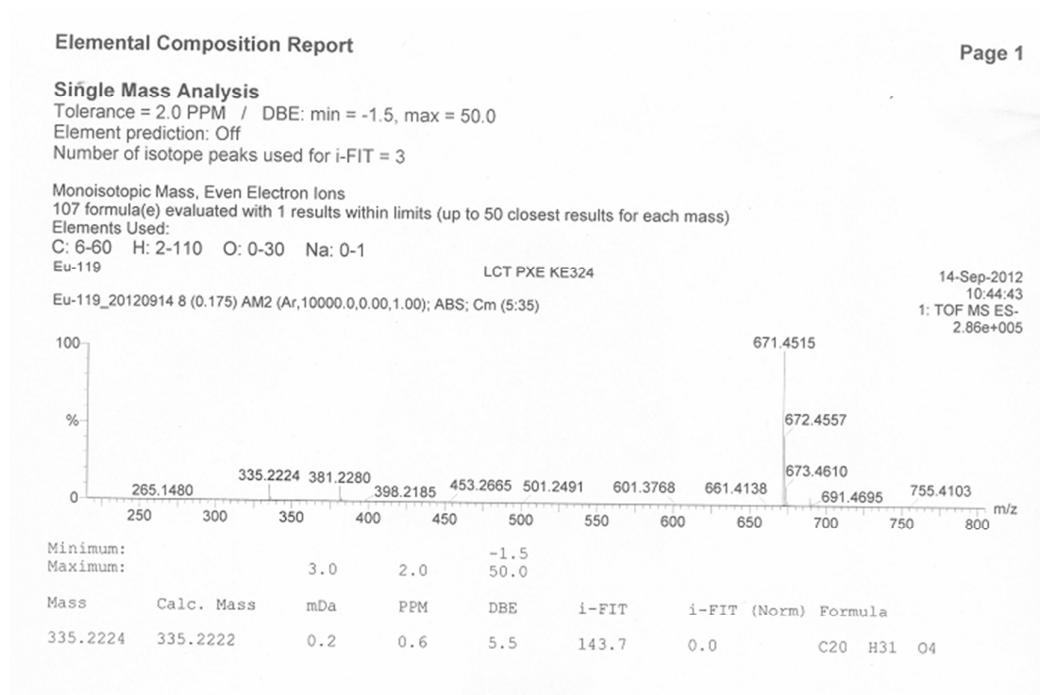
**Figure S16.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of quorumolide B (**2**) in  $\text{CDCl}_3$ .



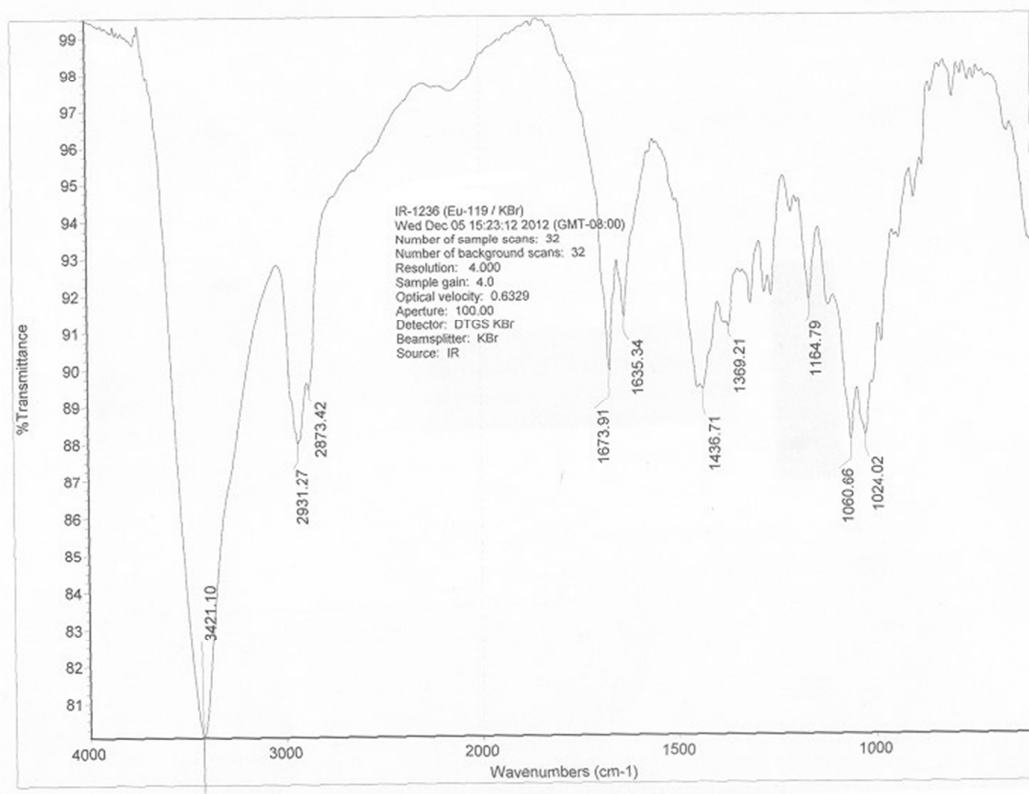
**Figure S17.** ROESY spectrum of quorumolide B (**2**) in CDCl<sub>3</sub>.



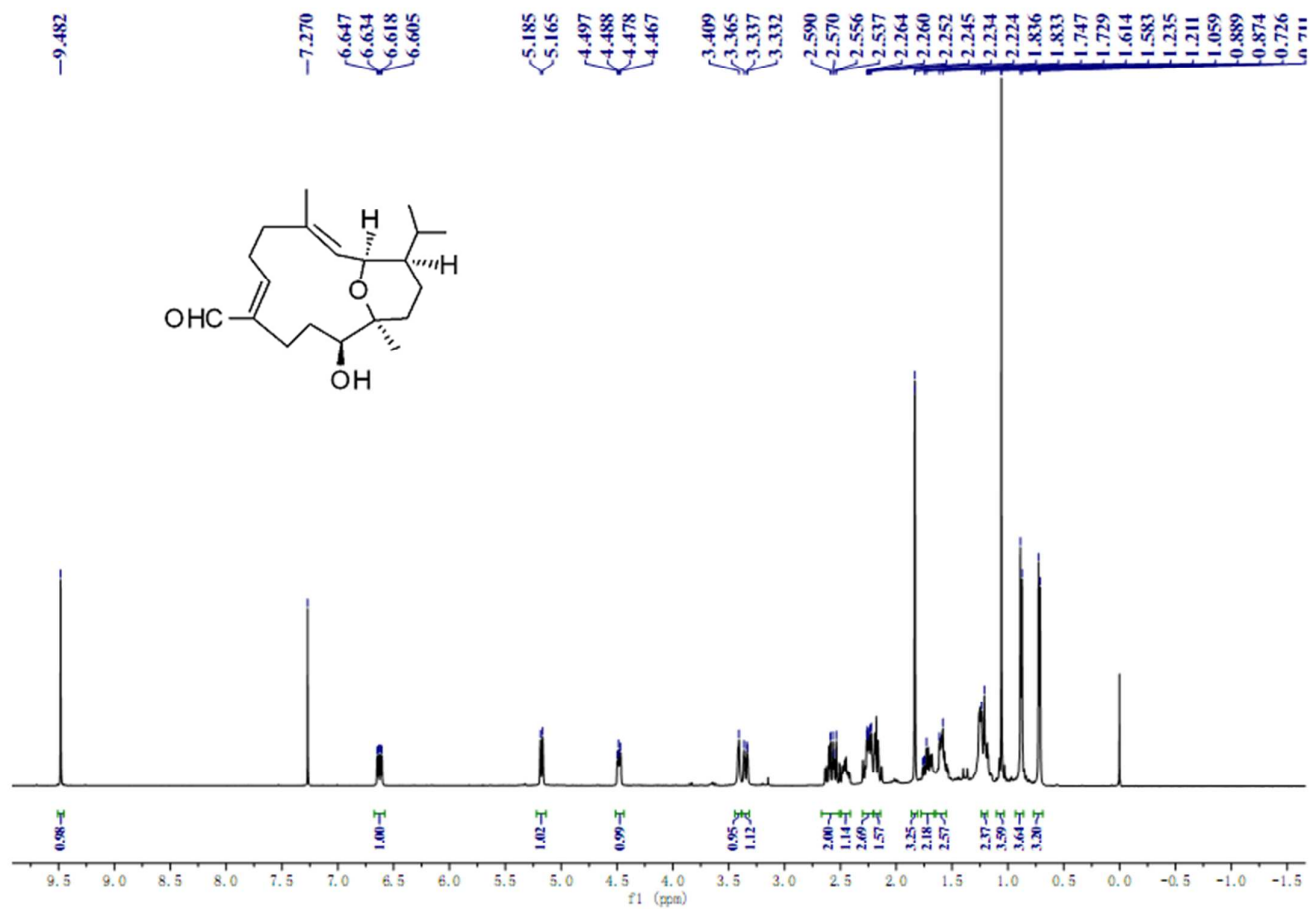
**Figure S18.** HRMS (ESI-TOF) spectrum of quorumolide B (**2**).



**Figure S19.** IR spectrum of quorumolide B (**2**).

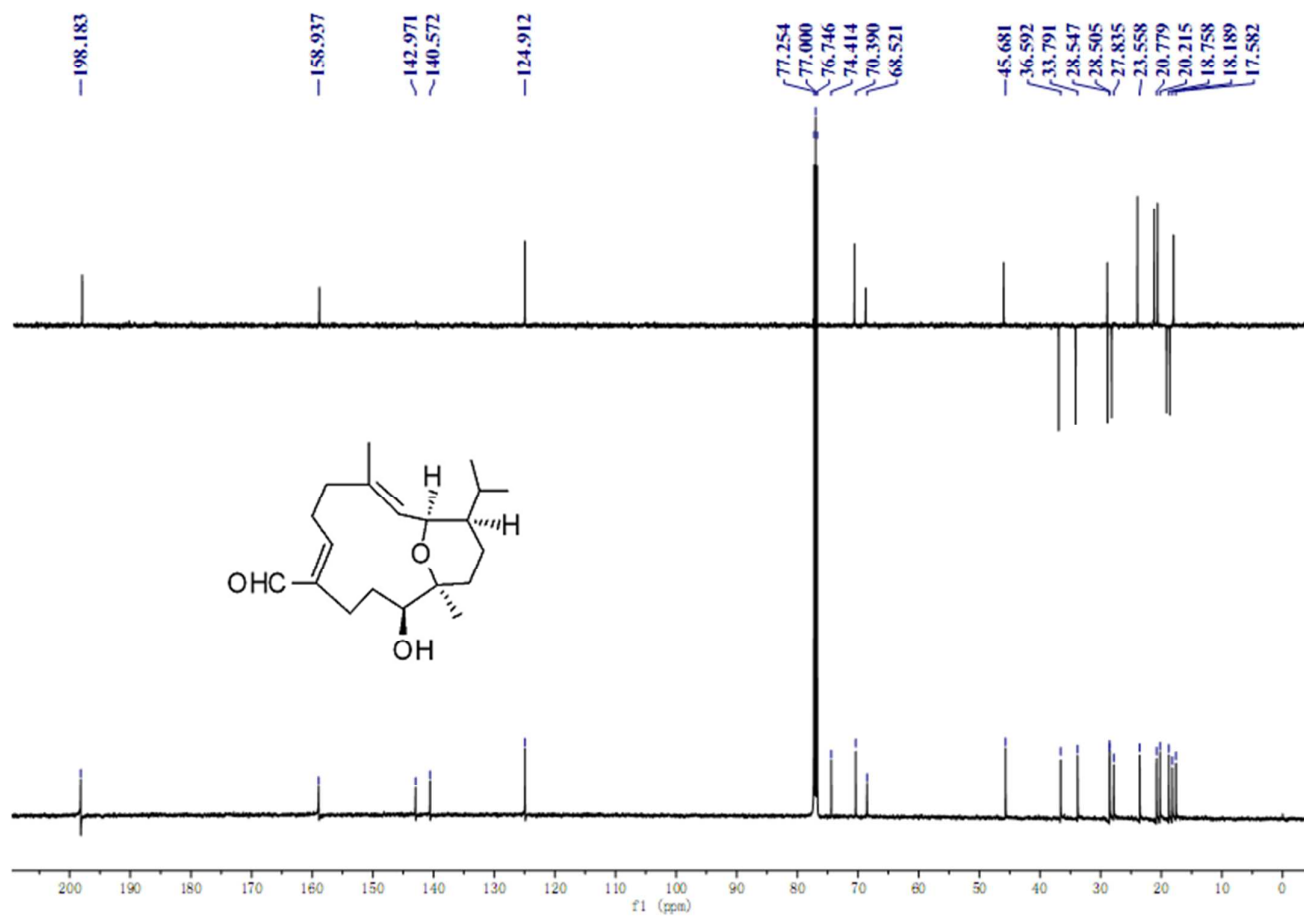


**Figure S20.**  $^1\text{H}$  NMR spectrum of quorumolide C (**3**) in  $\text{CDCl}_3$ .

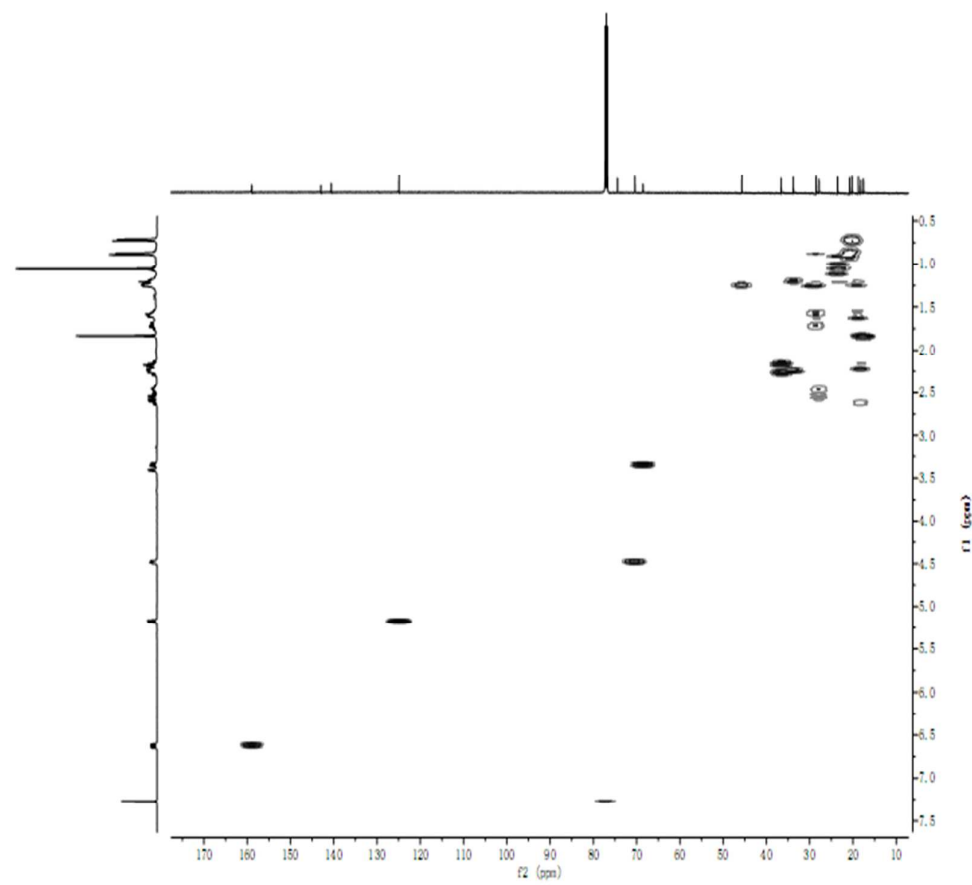




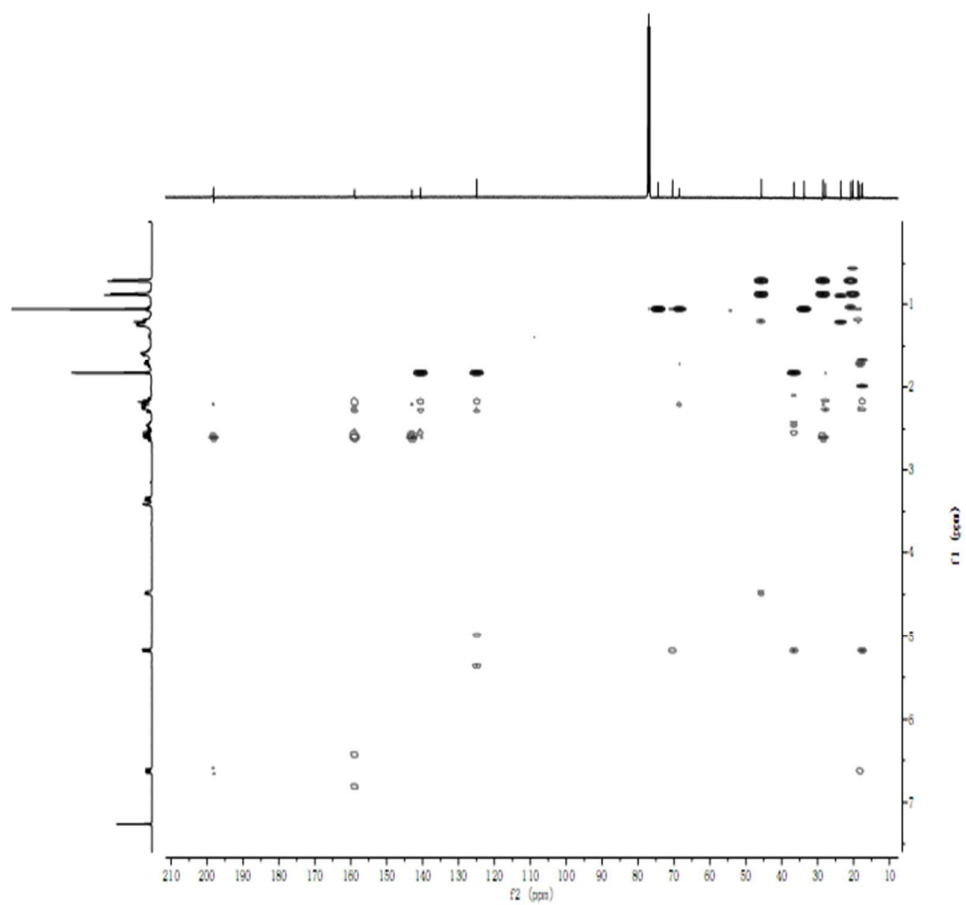
**Figure S21.**  $^{13}\text{C}$  NMR spectrum of quorumolide C (**3**) in  $\text{CDCl}_3$ .



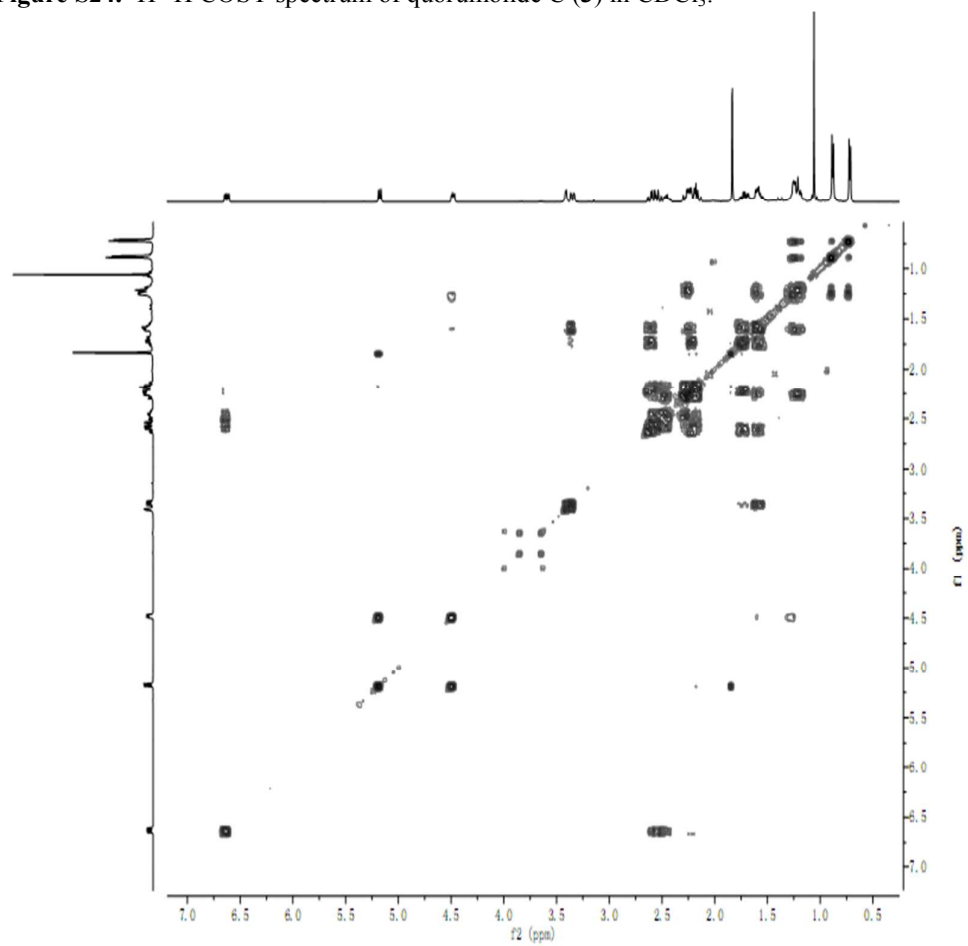
**Figure S22.** HSQC spectrum of quorumolide C (**3**) in CDCl<sub>3</sub>.



**Figure S23.** HMBC spectrum of quorumolide C (**3**) in CDCl<sub>3</sub>



**Figure S24.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of quorumolide C (**3**) in  $\text{CDCl}_3$ .



**Figure S25.** ROESY spectrum of quorumolide C (**3**) in CDCl<sub>3</sub>.

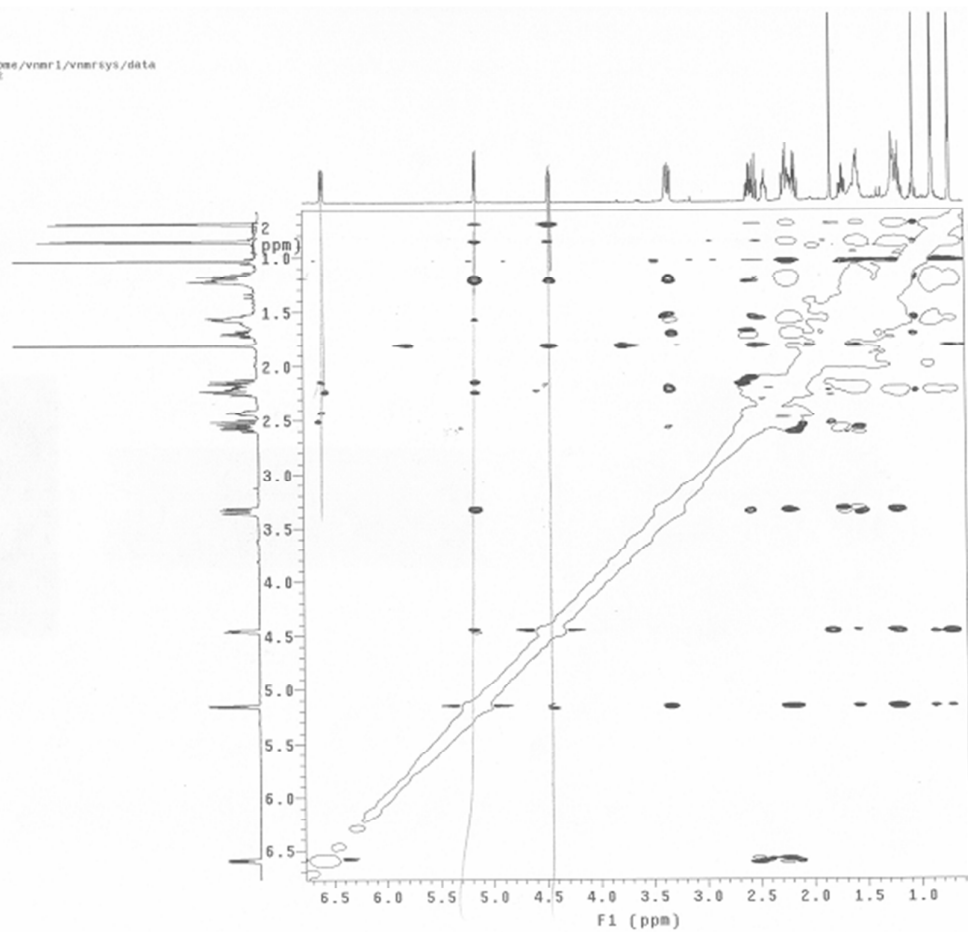
1\_27Mar2012

Archive directory: /export/home/vnmr1/vnmrsys/data  
Sample directory: 1\_27Mar2012  
File: ROESY

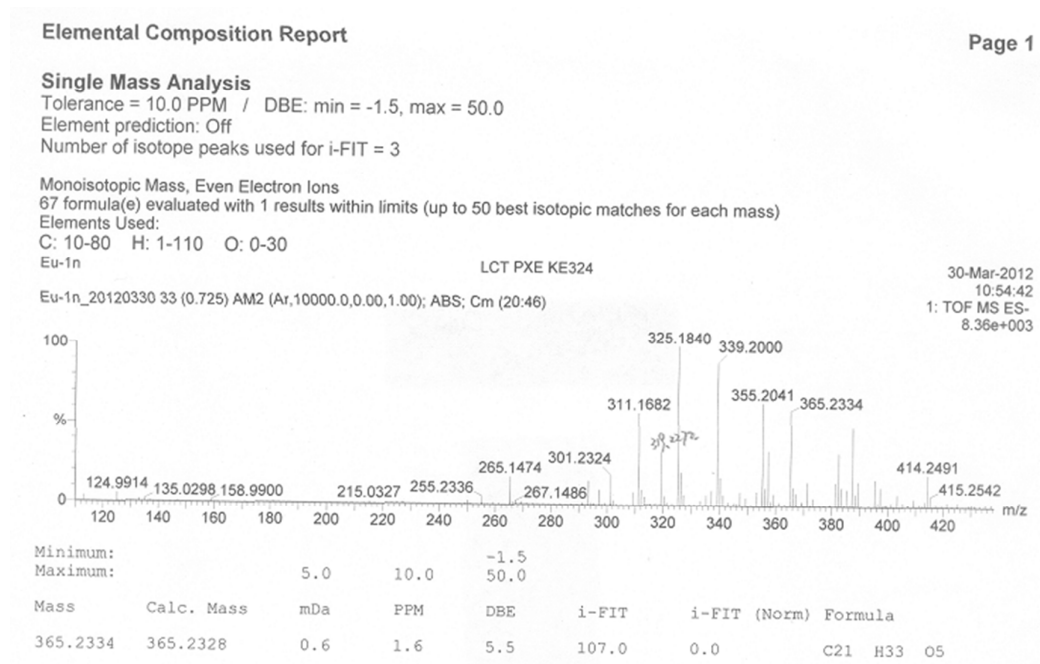
Pulse Sequence: ROESY

Solvent: CDCl<sub>3</sub>  
Temp: 25.0 C / 298.1 K  
INOVA-600 "cinn600"

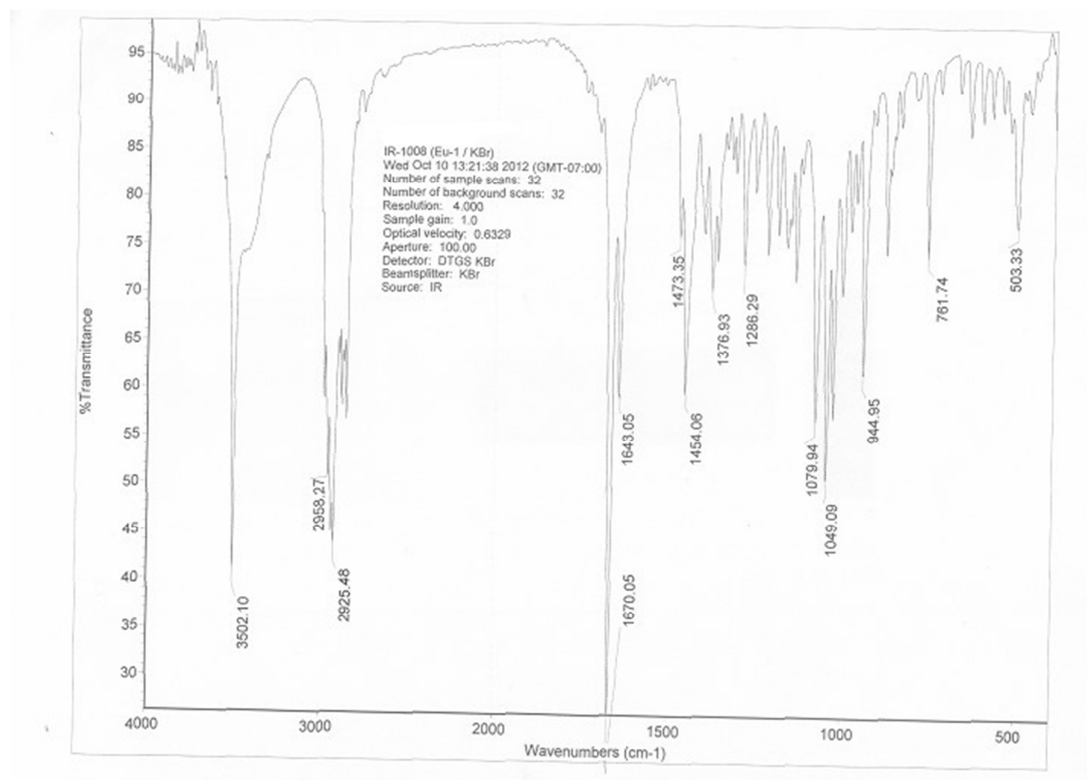
Relax. delay 1.000 sec  
Mixing 0.220 sec  
Acq. time 0.085 sec  
Width 11999.4 Hz  
2D Width 11999.4 Hz  
4 repetitions  
2 x 270 increments  
OBSERVE H1, 599.6560114 MHz  
DATA PROCESSING  
Gauss apodization 0.030 sec  
F1 DATA PROCESSING  
Gauss apodization 0.009 sec  
FT size 2048 x 2048  
Total time 45 min, 8 sec



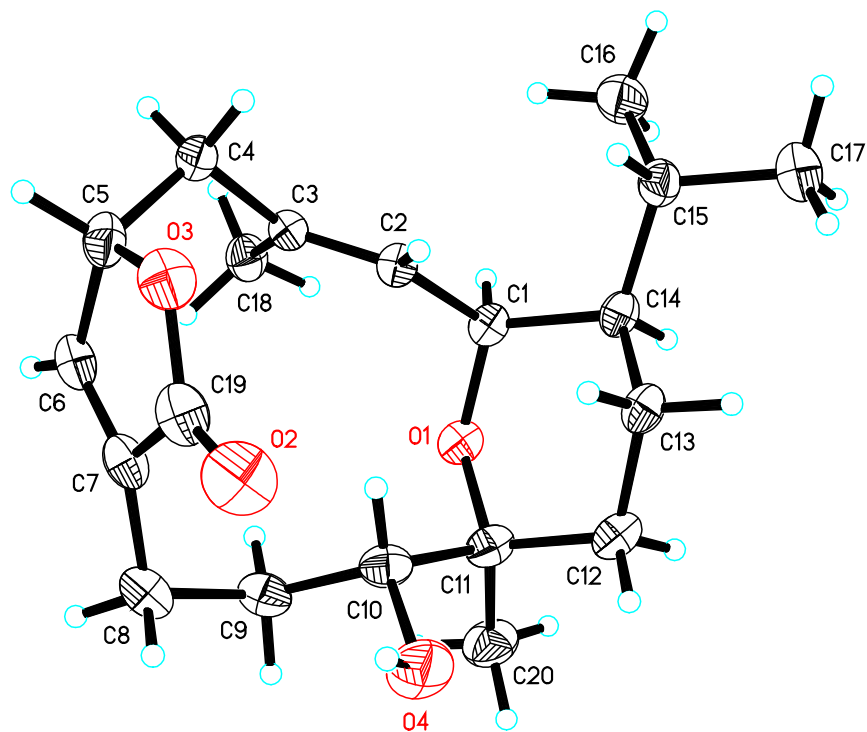
**Figure S26.** HRMS (ESI-TOF) spectrum of quorumolide C (**3**).



**Figure S27.** IR spectrum of quorumolide C (**3**).



**Figure S28.** ORTEP diagram of **1** (thermal ellipsoids at the 50% probability level)





**Figure S29.** ORTEP diagram of **2** (thermal ellipsoids at the 50% probability level)

