

## Supporting Information

for

### **Trogopterins A–C: Three new neolignans from feces of *Trogopterus xanthipes***

Soyoon Baek<sup>§,1</sup>, Xuikui Xia<sup>§,1,2</sup>, Byung Sun Min<sup>3</sup>, Chanil Park<sup>\*,¶,4</sup> and Sang Hee Shim<sup>\*,¶,1</sup>

Address: <sup>1</sup>School of Biotechnology, Yeungnam University, Daedong, Gyeongsan, Gyeongbuk 712-749, South Korea, <sup>2</sup>Key Laboratory for Applied Microbiology of Shandong Province, Biotechnology Center of Shandong Academy of Sciences, Jinan 250014, P. R. China, <sup>3</sup>College of Pharmacy, Catholic University of Daegu, 13-13 Hayang-ro, Gyeongsan, Gyeongbuk 712-702, South Korea and <sup>4</sup>Department of Marine Biology & Aquaculture, Gyeongsang National University, Tongyeong, Gyeongnam 650-160, South Korea

Email: Sang Hee Shim<sup>\*</sup>-shshim29@ynu.ac.kr; Chanil Park<sup>\*</sup>-vinus96@hanmail.net

<sup>\*</sup>Corresponding author

<sup>§</sup>These authors equally contributed to this work.

<sup>¶</sup>Tel. Sang Hee Shim-+82 53 810 3028; Chanil Park-+82 55 772 9153

## Contents

- Figure S1:  $^1\text{H}$  NMR spectrum of compound **1** (DMSO- $d_6$ , 600 MHz)
- Figure S2:  $^{13}\text{C}$  NMR spectrum of compound **1** (DMSO- $d_6$ , 150 MHz)
- Figure S3:  $^1\text{H},^1\text{H}$  COSY spectrum of **1** (DMSO- $d_6$ , 600 MHz)
- Figure S4: HMQC spectrum of compound **1** (DMSO- $d_6$ , 600 MHz)
- Figure S5: HMBC spectrum of compound **1** (DMSO- $d_6$ , 600 MHz)
- Figure S6: NOESY spectrum of compound **1** (DMSO- $d_6$ , 600 MHz)
- Figure S7: HREIMS of compound **1**
- Figure S8:  $^1\text{H}$  NMR spectrum of compound **2** (pyridine- $d_5$ , 600 MHz)
- Figure S9:  $^{13}\text{C}$  NMR spectrum of compound **2** (pyridine- $d_5$ , 150 MHz)
- Figure S10:  $^1\text{H},^1\text{H}$  COSY spectrum of **2** (pyridine- $d_5$ , 600 MHz)
- Figure S11: HMQC spectrum of compound **2** (pyridine- $d_5$ , 600 MHz)
- Figure S12: HMBC spectrum of compound **2** (pyridine- $d_5$ , 600 MHz)
- Figure S13: HREIMS of compound **2**
- Figure S14:  $^1\text{H}$  NMR spectrum of compound **3** (CD<sub>3</sub>OD, 600 MHz)
- Figure S15:  $^{13}\text{C}$  NMR spectrum of compound **3** (CD<sub>3</sub>OD, 150 MHz)
- Figure S16:  $^1\text{H},^1\text{H}$  COSY spectrum of **3** (CD<sub>3</sub>OD, 600 MHz)
- Figure S17: HMQC spectrum of compound **3** (CD<sub>3</sub>OD, 600 MHz)
- Figure S18: HMBC spectrum of compound **3** (CD<sub>3</sub>OD, 600 MHz)
- Figure S19: NOESY spectrum of compound **3** (CD<sub>3</sub>OD, 600 MHz)
- Figure S20: HREIMS of compound **3**

TXE16-4\_wet1d  
File: xp  
Pulse Sequence: wet1D

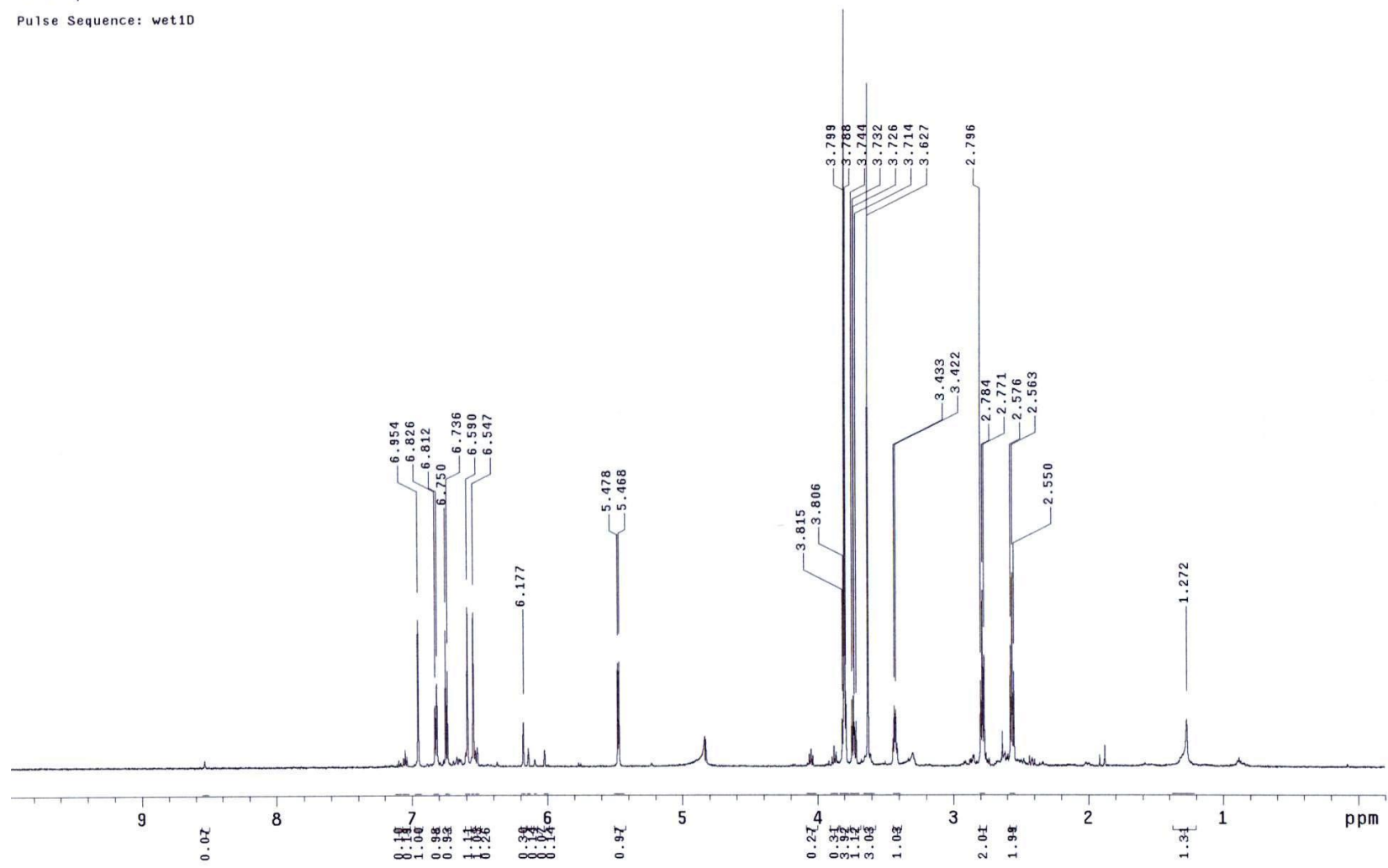


Figure S1: <sup>1</sup>H NMR spectrum of compound 1 (DMSO-*d*<sub>6</sub>, 300 MHz).

TXE16-4  
File: xp  
Pulse Sequence: s2pu1

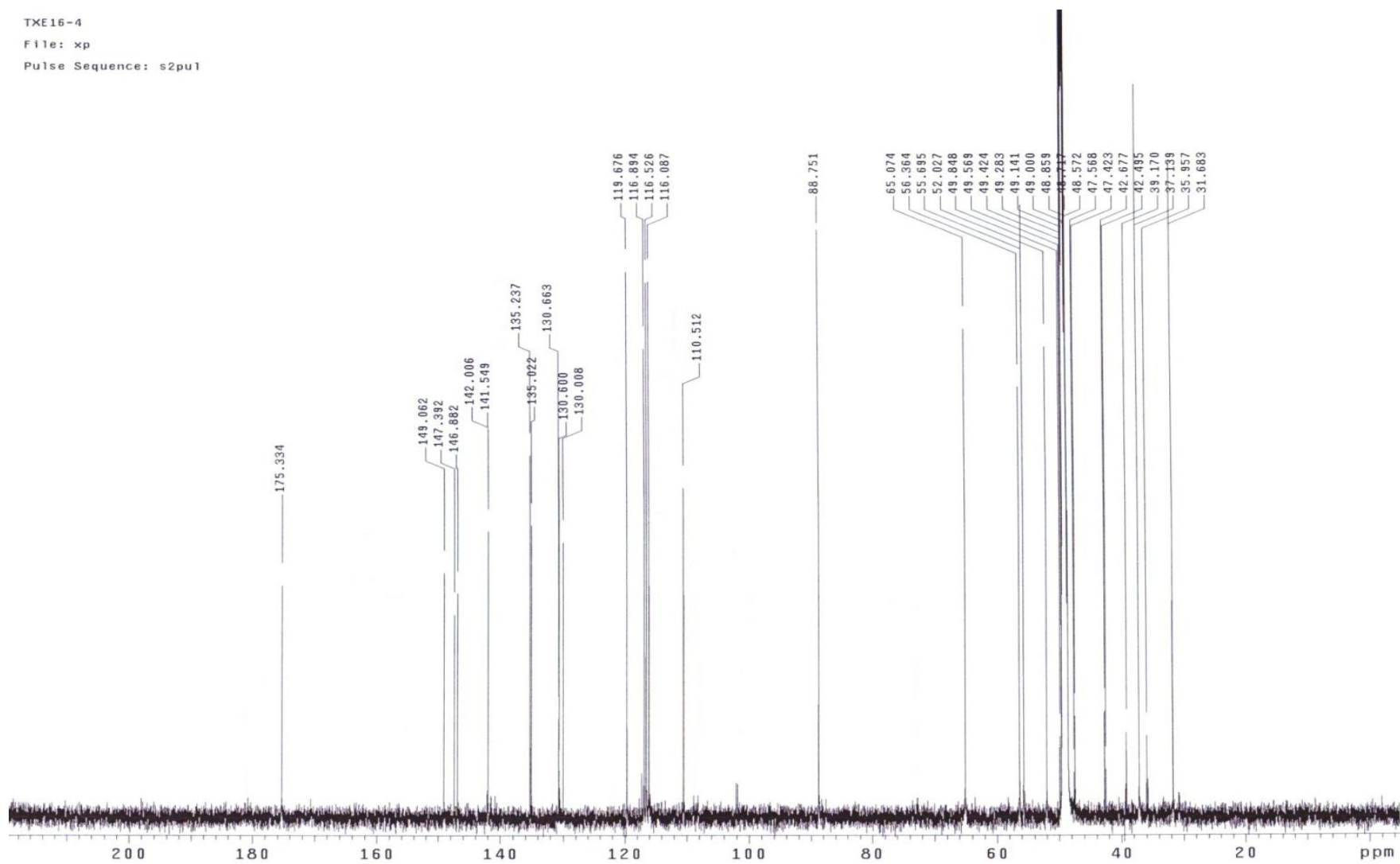


Figure S2:  $^{13}\text{C}$  NMR spectrum of compound 1 (DMSO- $d_6$ , 75 MHz).

TXE16-4  
File: xp  
Pulse Sequence: gCOSY  
Solvent: cd3od  
Temp: 25.0 C / 298.1 K  
Operator: vmmr1  
VNMRS-600 "ynnmr600"  
  
Relax. delay 1.000 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
8 repetitions  
512 increments  
OBSERVE H1, 599.8582915 MHz  
DATA PROCESSING  
Sine bell 0.080 sec  
F1 DATA PROCESSING  
Sine bell 0.160 sec  
FT size 8192 x 8192  
Total time 1 hr, 32 min, 51 sec

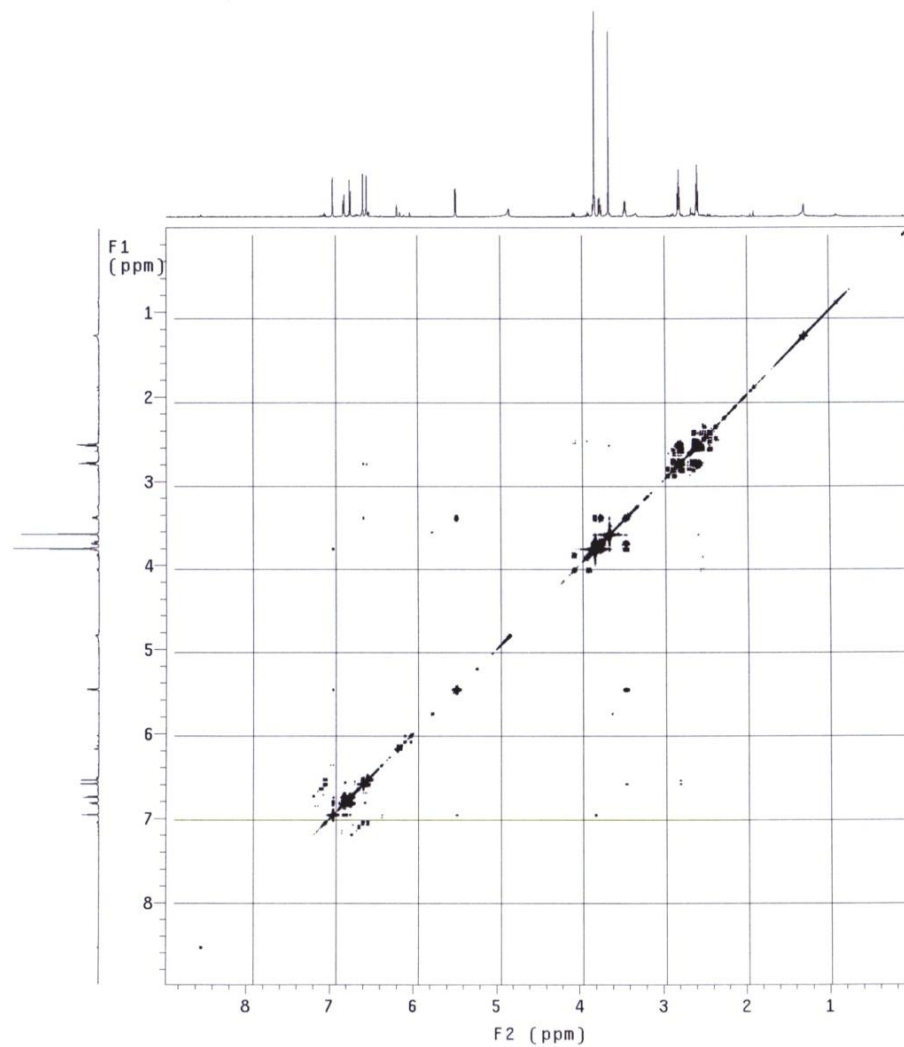


Figure S3:  $^1\text{H}, ^1\text{H}$  COSY spectrum of compound **1** ( $\text{DMSO}-d_6$ , 600 MHz).

TXE16-4

File: xp

Pulse Sequence: gHMOC

Solvent: cd3od  
Temp. 25.0 C / 298.1 K  
Operator: vnmr1  
VNMR-600 "vnmr600"

Relax. delay 1.000 sec  
Acq. time 0.128 sec  
Width 6410.3 Hz  
2D Width 25641.0 Hz  
16 repetitions  
2 x 512 increments  
OBSERVE H1, 599.8582903 MHz  
DECOUPLE C13, 150.8457980 MHz  
Power 37 dB  
on during acquisition  
off during delay  
W40\_swpg modulated  
DATA PROCESSING  
Gauss apodization 0.059 sec  
F1 DATA PROCESSING  
Gauss apodization 0.018 sec  
FT size 4096 x 4096  
Total time 6 hr, 27 sec

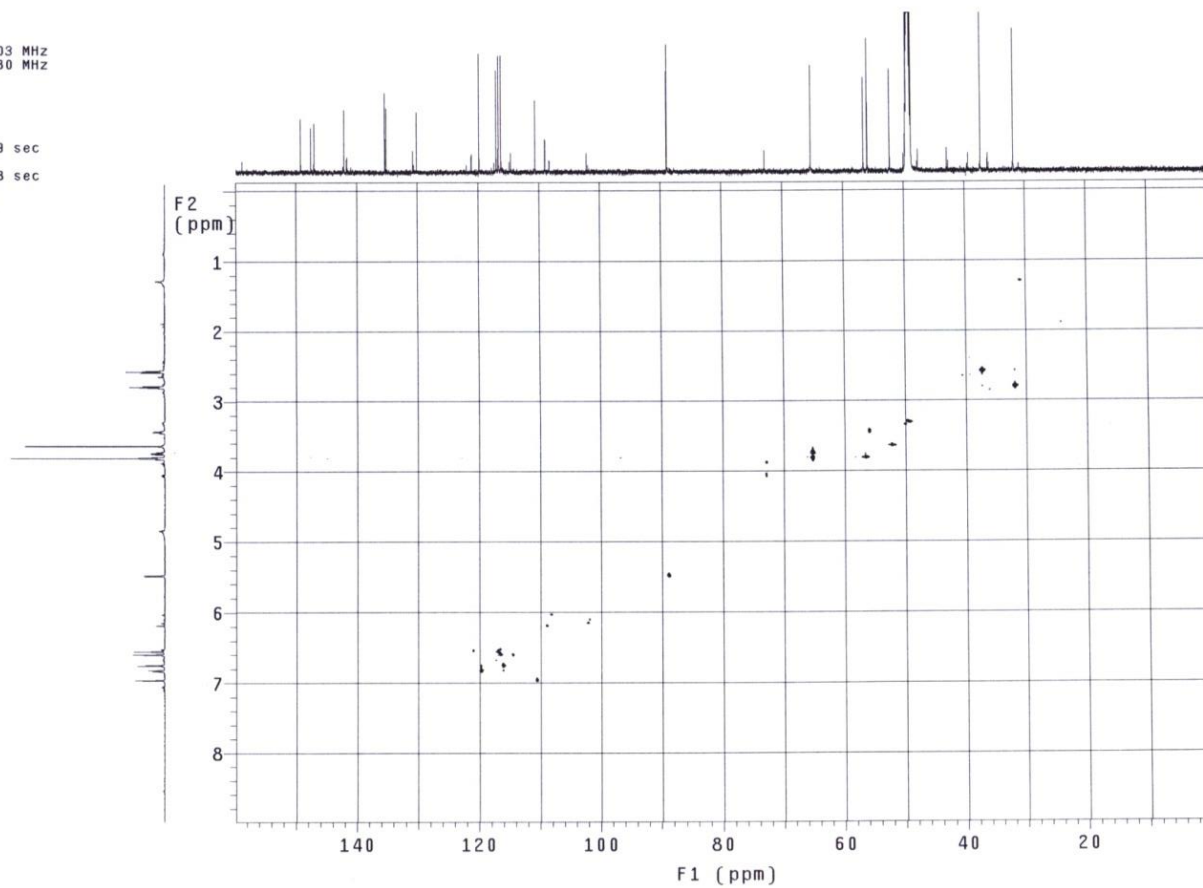


Figure S4: HMQC spectrum of compound 1 (DMSO-*d*<sub>6</sub>, 600 MHz).

TXE16-4

File: xp

Pulse Sequence: gHMBC

Solvent: cd3od  
Temp: 25.0 C / 298.1 K  
Operator: vnmr1  
VNMR5-600 "vnmr600"

Relax. delay 1.000 sec  
Mixing 0.080 sec  
Acq. time 0.128 sec  
Width 6410.3 Hz  
2D Width 30165.9 Hz  
32 repetitions  
512 increments  
OBSERVE H1, 599.8582933 MHz  
DATA PROCESSING  
Sine bell 0.064 sec  
F1 DATA PROCESSING  
Sine bell 0.017 sec  
FT size 4096 x 4096  
Total time 6 hr, 12 min, 14 sec

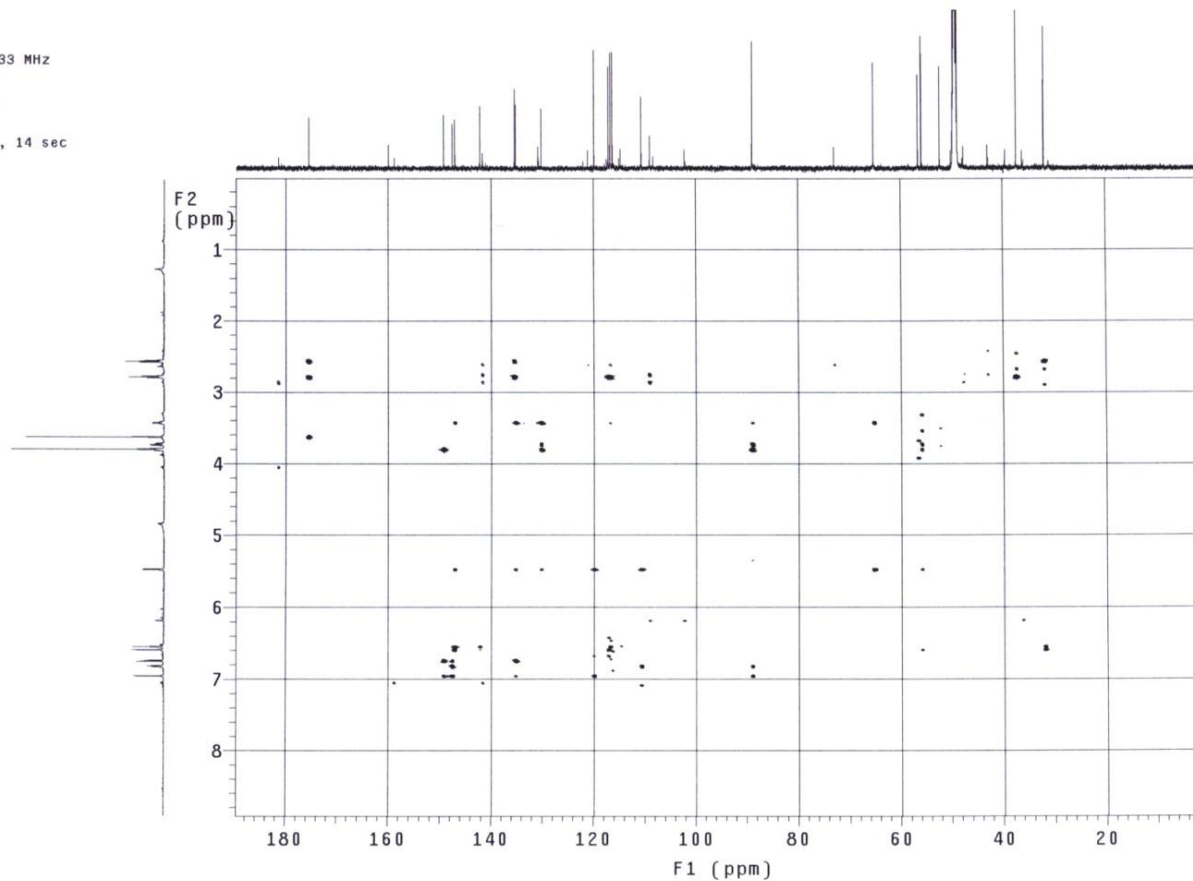


Figure S5: HMBC spectrum of compound 1 (DMSO- $d_6$ , 600 MHz).

TXE16-4

File: xp

Pulse Sequence: NOESY

Solvent: cd3od  
Temp. 25.0 C / 298.1 K  
Operator: vnmr1  
VNMR5-600 "ynmr600"

Relax. delay 1.000 sec  
Mixing 0.400 sec  
Acq. time 0.160 sec  
Width 6410.3 Hz  
2D Width 6410.3 Hz  
16 repetitions  
2 x 512 increments  
OBSERVE H1, 599.8582914 MHz  
DATA PROCESSING  
Gauss apodization 0.074 sec  
F1 DATA PROCESSING  
Gauss apodization 0.148 sec  
FT size 8192 x 8192  
Total time 7 hr, 45 min, 43 sec

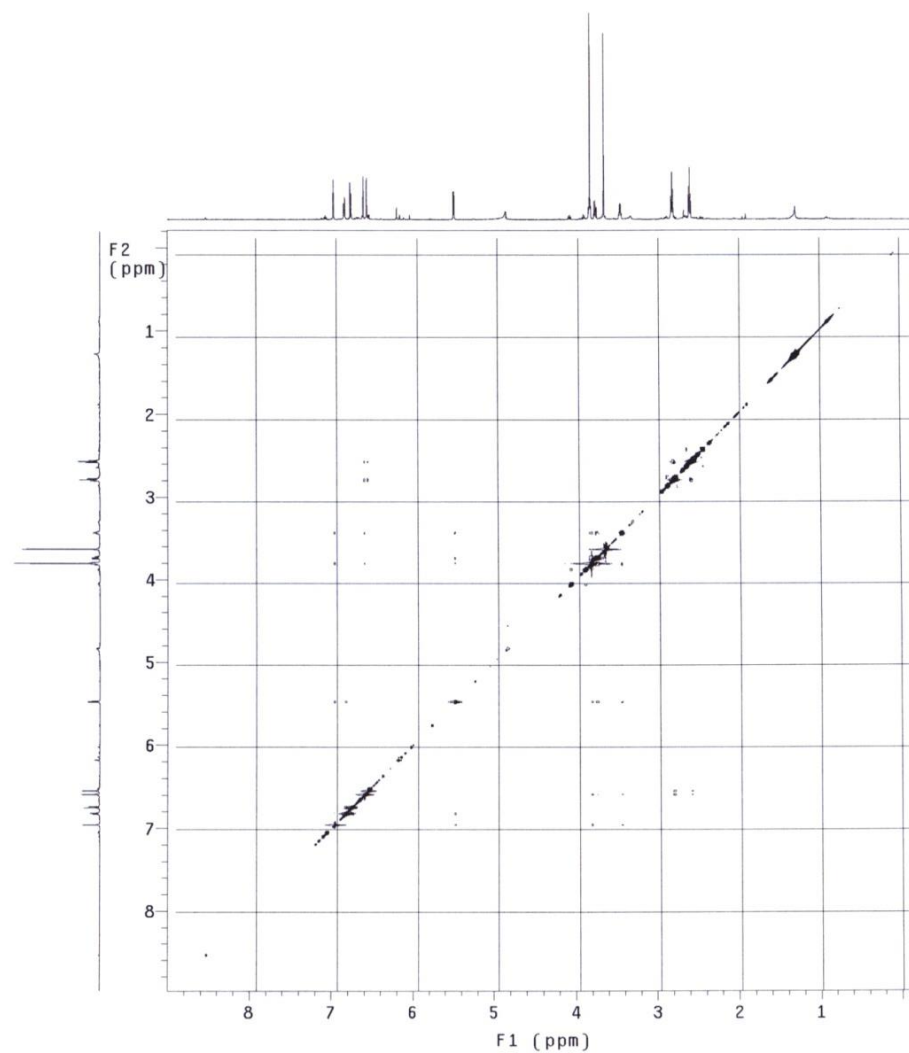


Figure S6: NOESY spectrum of compound 1 (DMSO- $d_6$ , 600 MHz).



File: 10221  
Sample: TXE-1

Date Run: 12-12-2013  
Ionization mode: EI+

Time Run: 11:21:59

Instrument: JEOL JMS600  
Inlet: Direct Probe

R.T.: 1.45.6

Scan: 139-163  
Base: m/z 344; 6.1%FS

TIC: 1084381

Selected Isotopes :  $C_{0-50}H_{0-100}O_{0-10}$

Error Limit : 20 ppm

<u>Measured Mass</u>	<u>% Base</u>	<u>Formula</u>	<u>Calculated Mass</u>	<u>Error</u>
374.1364	39.8%	$C_{27}H_{18}O_2$	374.1307	-15.0
		$C_{20}H_{22}O_7$	374.1365	0.4

Figure S7: HREIMS of compound 1.

TXE17-3  
File: xp  
Pulse Sequence: s2pu1

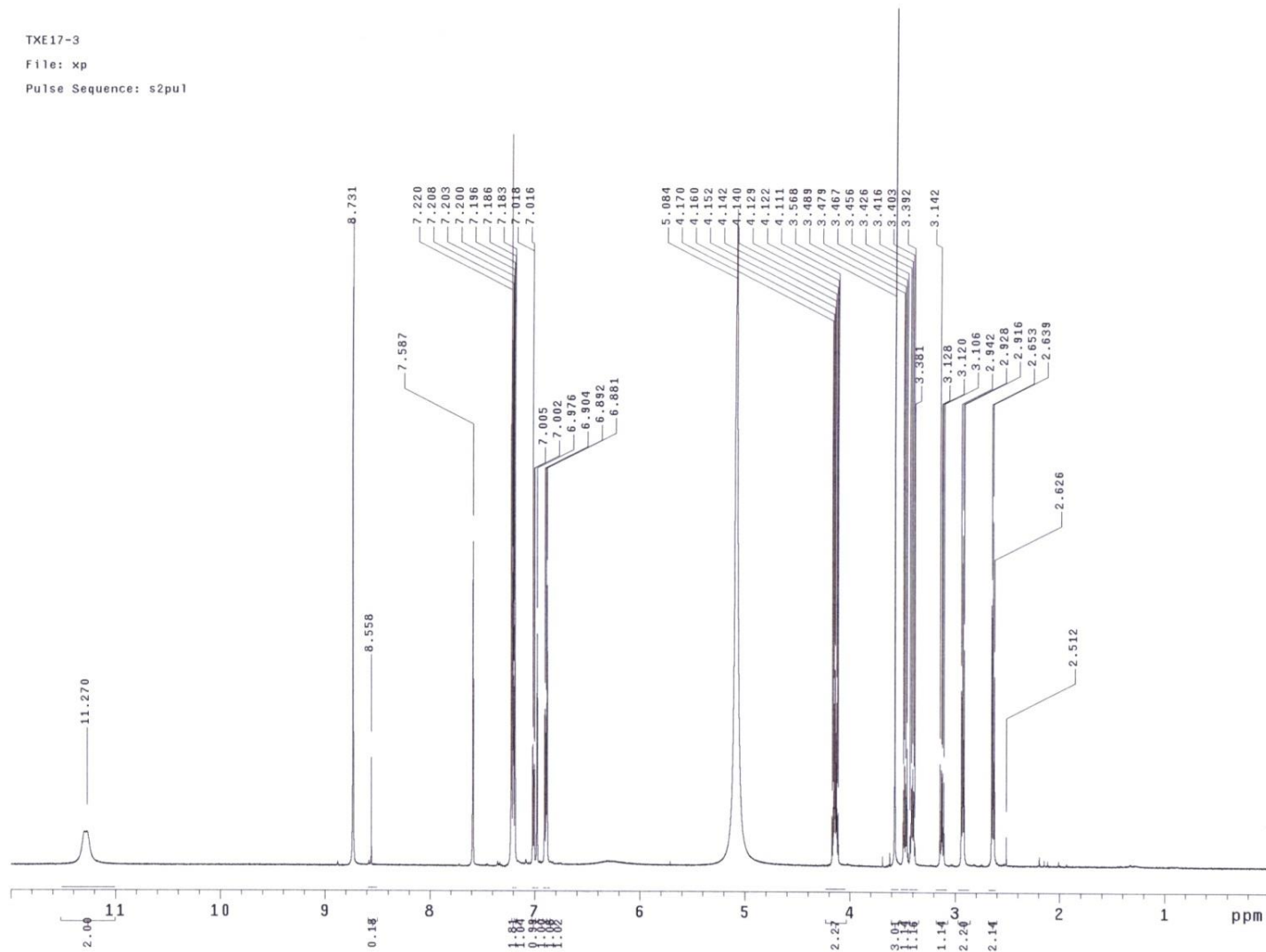


Figure S8:  $^1\text{H}$  NMR spectrum (pyridine- $d_5$ , 600 MHz) of compound 2.

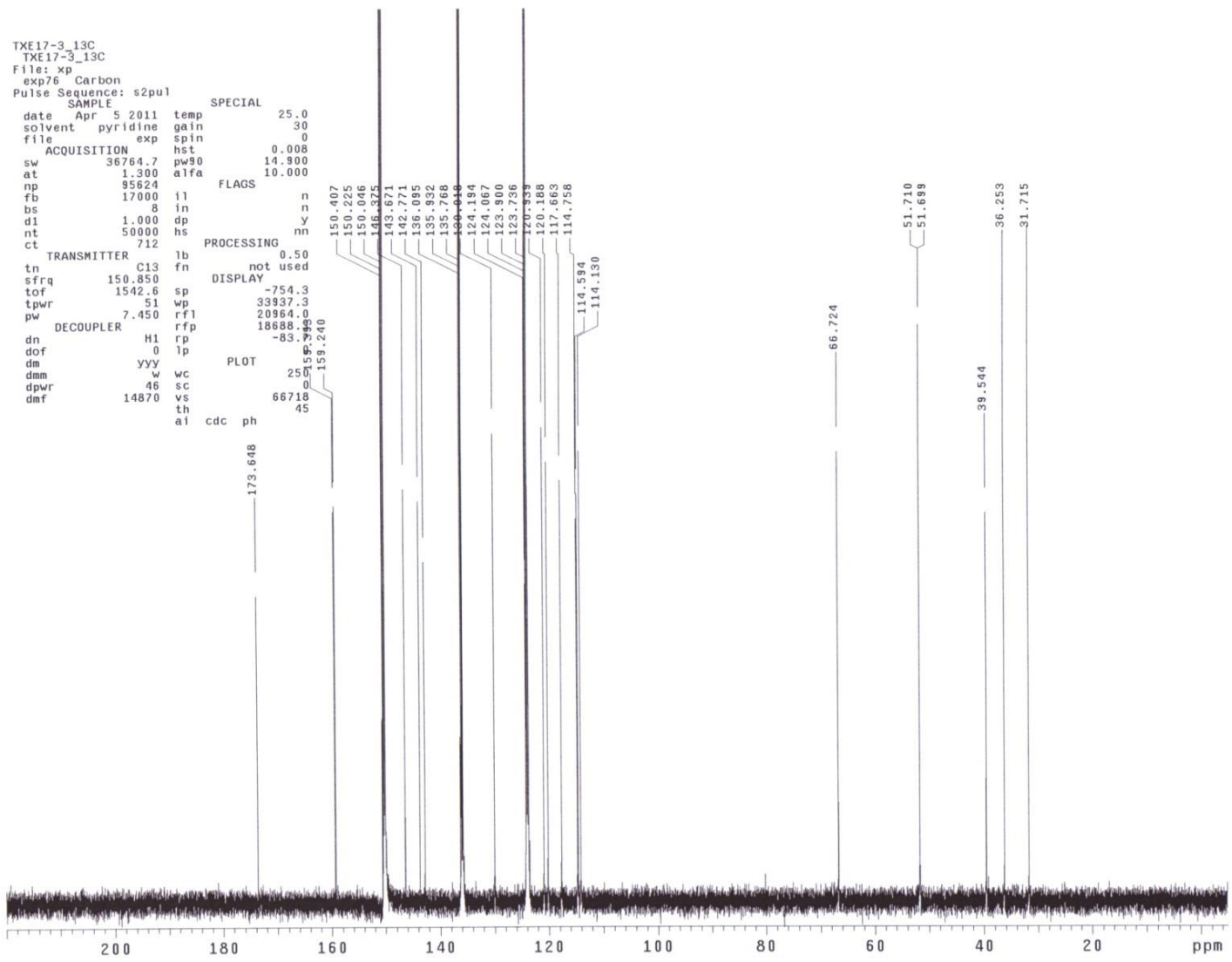


Figure S9:  $^{13}\text{C}$  NMR spectrum (pyridine- $d_5$ , 150 MHz) of compound 2.

TXE17-3

File: xp

Pulse Sequence: gCOSY

Solvent: pyridine  
Temp.: 25.0 C / 298.1 K  
Operator: vnmr1  
VNMR5-600 "ynnmr600"

Relax. delay 1.000 sec  
Acq. time 0.139 sec  
Width 7352.9 Hz  
2D Width 7352.9 Hz  
8 repetitions  
256 increments  
OBSERVE H1, 599.8558984 MHz  
DATA PROCESSING  
Sine bell 0.070 sec  
F1 DATA PROCESSING  
Sine bell 0.070 sec  
FT size 8192 x 8192  
Total time 40 min, 22 sec

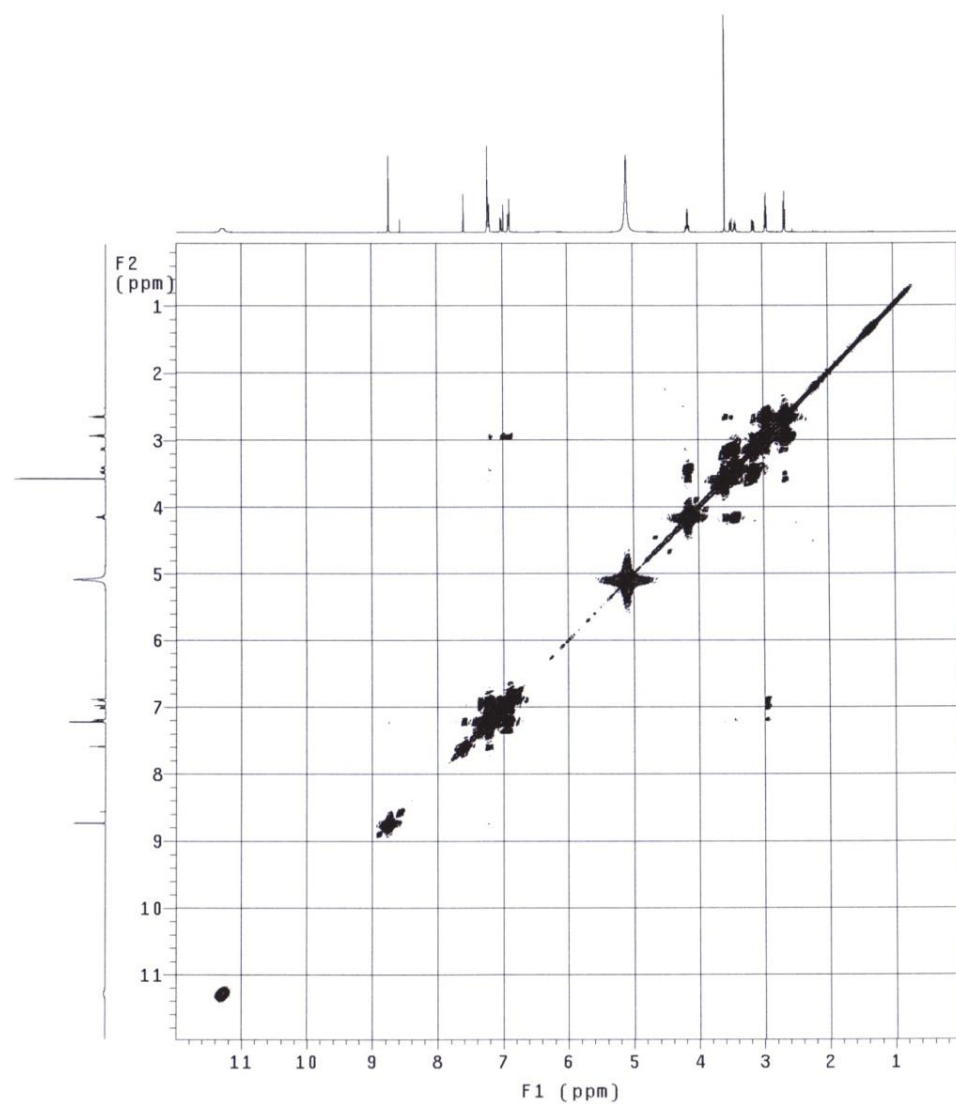


Figure S10:  $^1\text{H},^1\text{H}$  COSY spectrum (pyridine- $d_5$ , 600 MHz) of compound 2.

TXE17-3\_(2)

File: xp

Pulse Sequence: gHMOC

Solvent: pyridine  
Temp. 25.0 C / 298.1 K  
Operator: vnmr1  
VNMRS-600 "ynmr600"

Relax. delay 1.000 sec  
Acq. time 0.128 sec  
Width 7440.5 Hz  
2D Width 30165.9 Hz  
32 repetitions  
2 x 512 increments  
OBSERVE H1, 599.8550044 MHz  
DECOUPLE C13, 150.8474194 MHz  
Power 37 dB  
on during acquisition  
off during delay  
W40\_swpgf modulated  
DATA PROCESSING  
Gauss apodization 0.059 sec  
F1 DATA PROCESSING  
Gauss apodization 0.024 sec  
FT size 8192 x 8192  
Total time 10 hr, 43 min, 55 sec

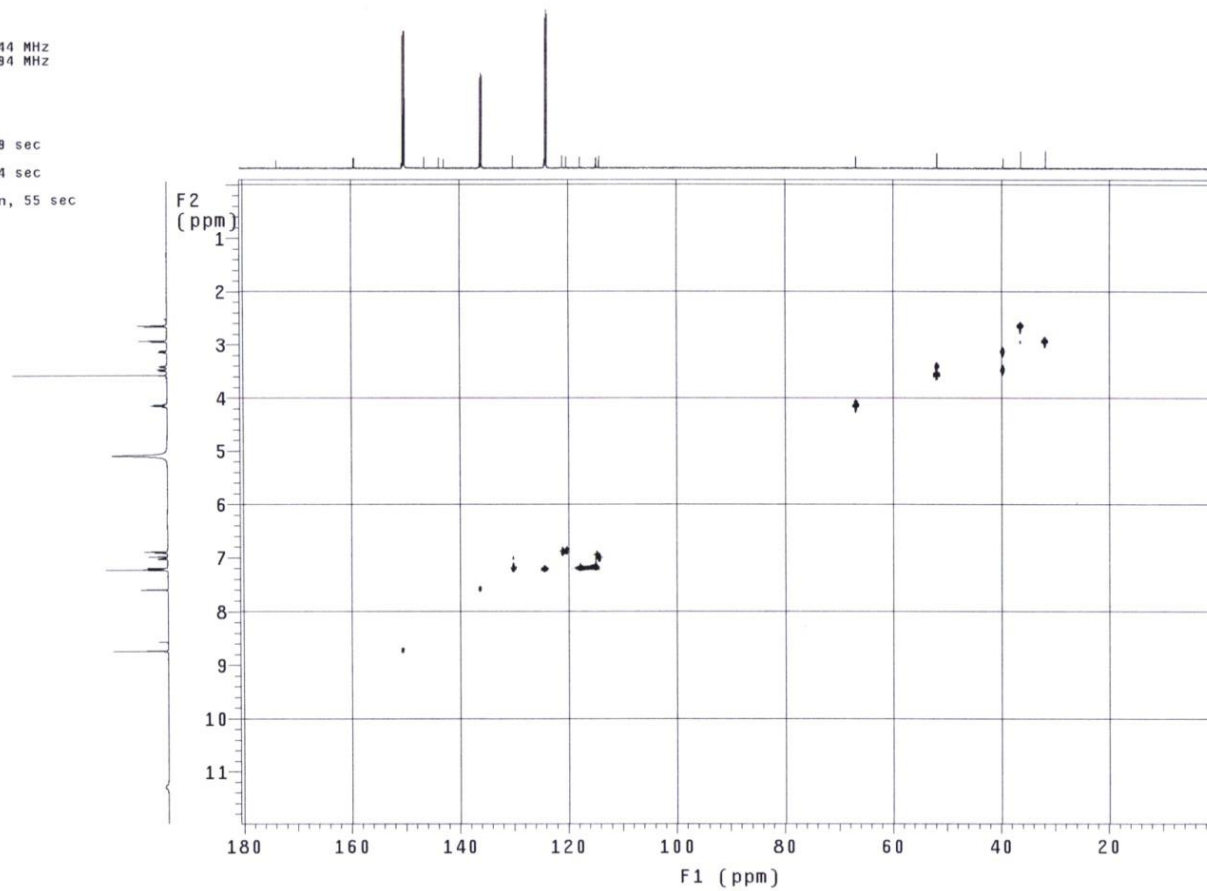


Figure S11: HMQC spectrum (pyridine-*d*<sub>5</sub>, 600 MHz) of compound 2.

TXE17-3

File: xp

Pulse Sequence: gHMBC

Solvent: pyridine

Temp. 25.0 C / 298.1 K

Operator: vnmr1

VNMRS-600 "vnmr600"

Relax. delay 1.000 sec

Mixing 0.080 sec

Acq. time 0.128 sec

Width 7352.9 Hz

2D Width 36199.1 Hz

16 repetitions

512 increments

OBSERVE H1, 599.8558978 MHz

DATA PROCESSING

Sine bell 0.064 sec

F1 DATA PROCESSING

Sine bell 0.014 sec

FT size 4096 x 4096

Total time 2 hr, 47 min, 21 sec

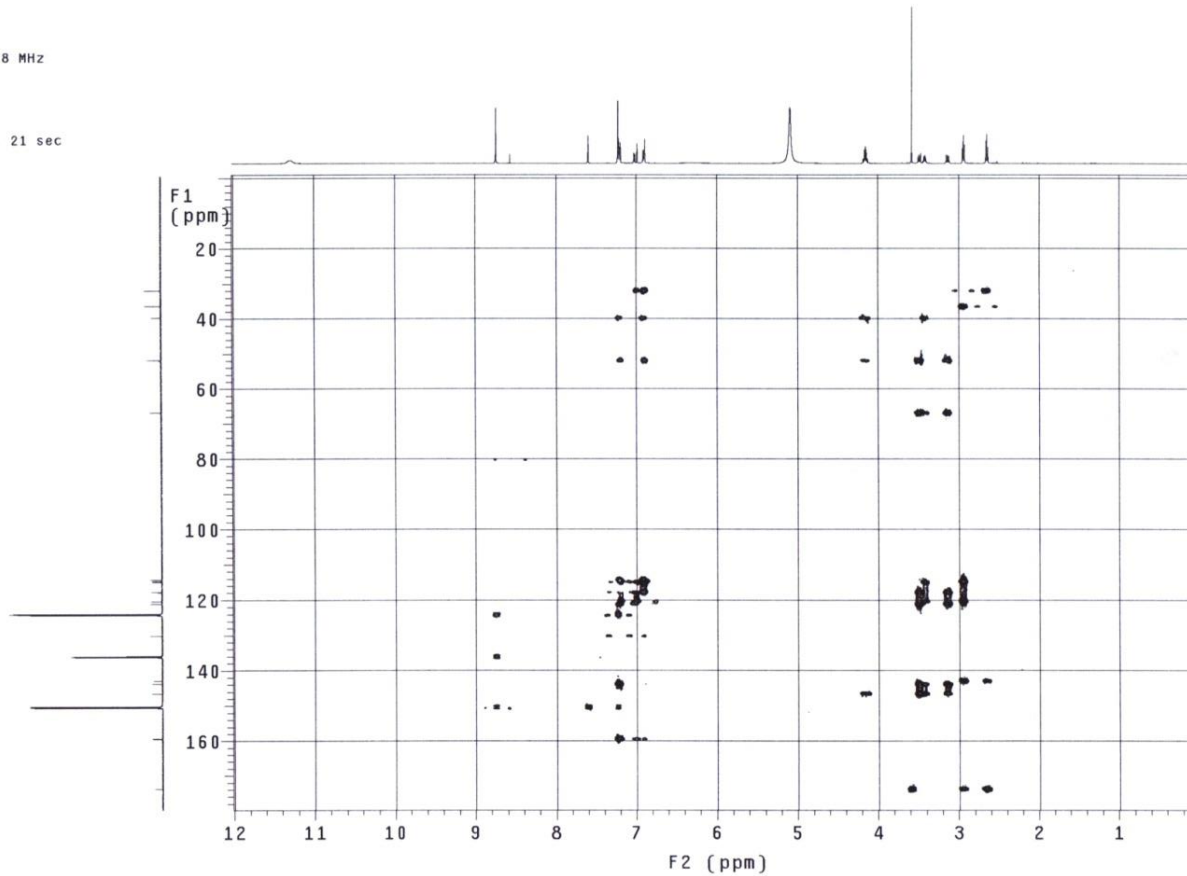


Figure S12: HMBC spectrum (pyridine- $d_5$ , 600 MHz) of compound 2.

File: 10220  
Sample: TXE-2

Date Run: 12-12-2013  
Ionization mode: EI+

Time Run: 09:56:10

Instrument: JEOL JMS600  
Inlet: Direct Probe

R.T.: 2:47.3

Scan: 140  
Base: m/z 330; 8.4%FS

TIC: 684936

Selected Isotopes : C<sub>0-50</sub>H<sub>0-100</sub>O<sub>0-10</sub>

Error Limit : 20 ppm

<u>Measured Mass</u>	<u>% Base</u>	<u>Formula</u>	<u>Calculated Mass</u>	<u>Error</u>
330.1469	100.0%	C <sub>26</sub> H <sub>18</sub>	330.1409	-18.0
		C <sub>19</sub> H <sub>22</sub> O <sub>5</sub>	330.1467	-0.6
		C <sub>12</sub> H <sub>26</sub> O <sub>10</sub>	330.1526	17.0

Figure S13: HREIMS of compound 2.

TXE3233-10-Y-1

File: xp

Pulse Sequence: s2pul

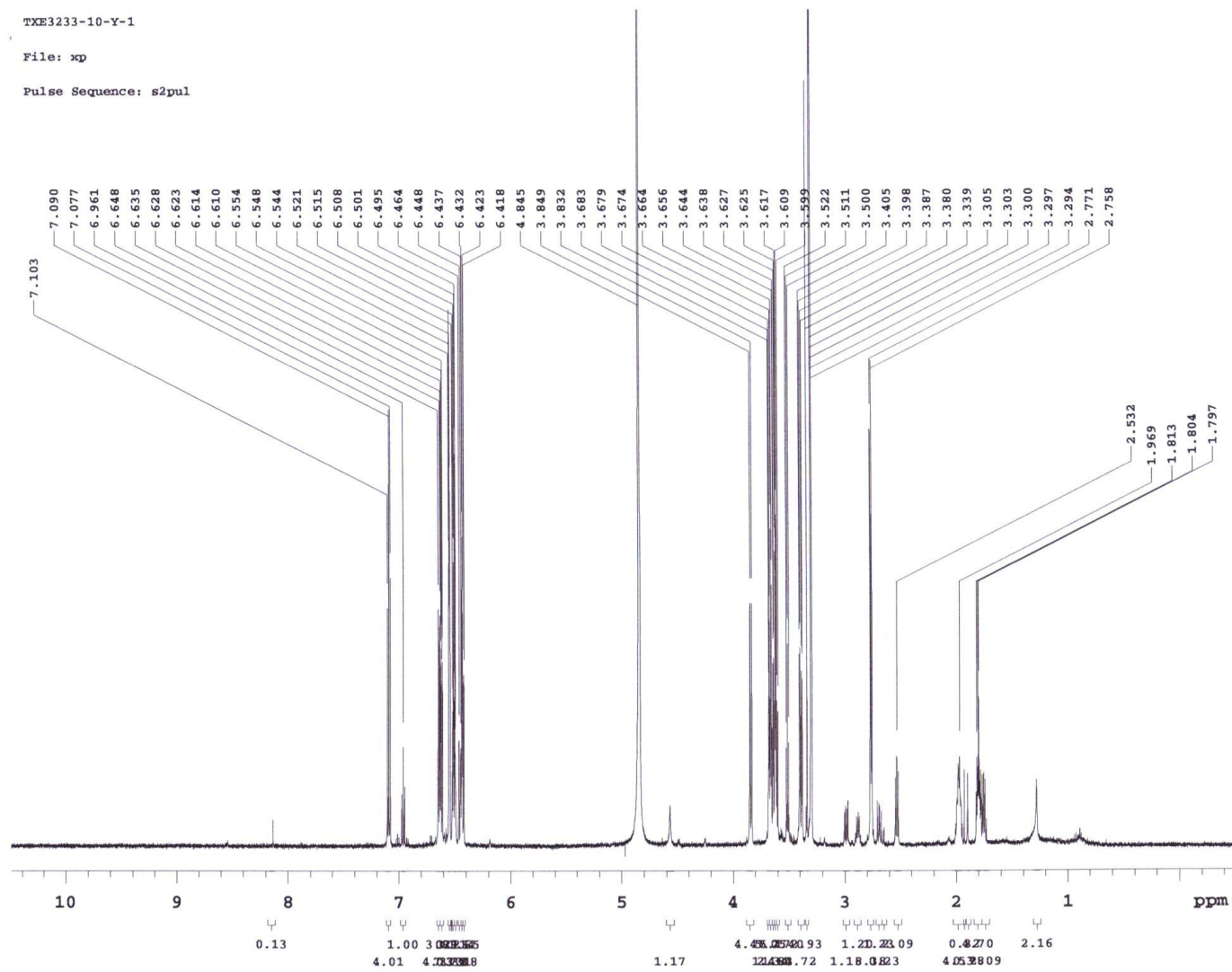


Figure S14:  $^1\text{H}$  NMR spectrum ( $\text{CD}_3\text{OD}$ , 600 MHz) of compound **3**.



TXE3233-10-Y-1

File: xp

Pulse Sequence: s2pul

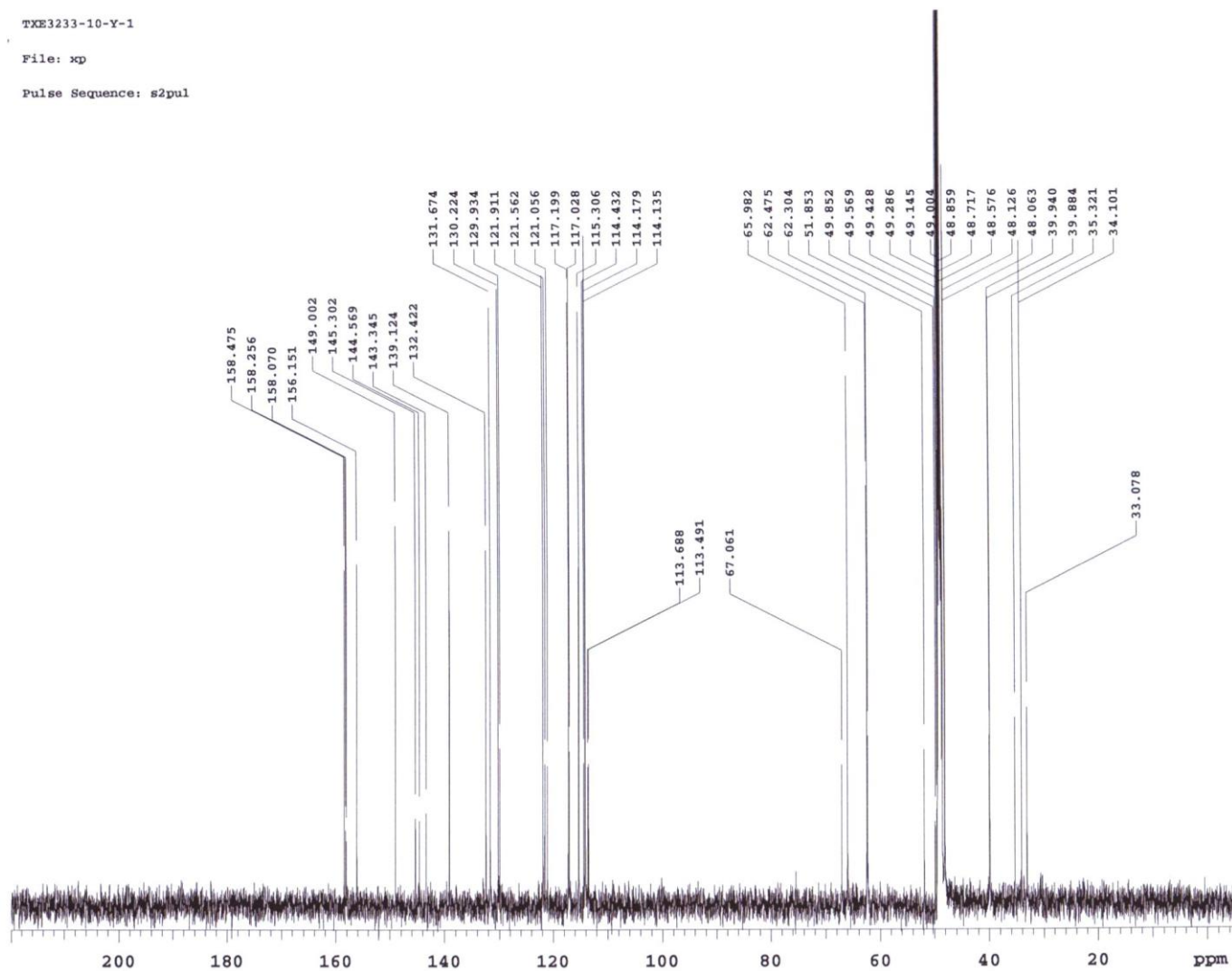


Figure S15:  $^{13}\text{C}$  NMR spectrum ( $\text{CD}_3\text{OD}$ , 600 MHz) of compound **3**.

TXE3233-10-Y-1

File: xp

Pulse Sequence: gCOSY

Solvent: cd3od  
Temp: 25.0 C / 298.1 K  
Operator: vnmr1  
VNMRS-600 "ynnmr600"

Relax. delay 1.000 sec  
Acq. time 0.203 sec  
Width 5040.3 Hz  
2D Width 5040.3 Hz  
8 repetitions  
512 increments  
OBSERVE H1, 599.8582852 MHz  
DATA PROCESSING  
Sine bell 0.102 sec  
F1 DATA PROCESSING  
Sine bell 0.203 sec  
FT size 8192 x 8192  
Total time 1 hr, 36 min, 34 sec

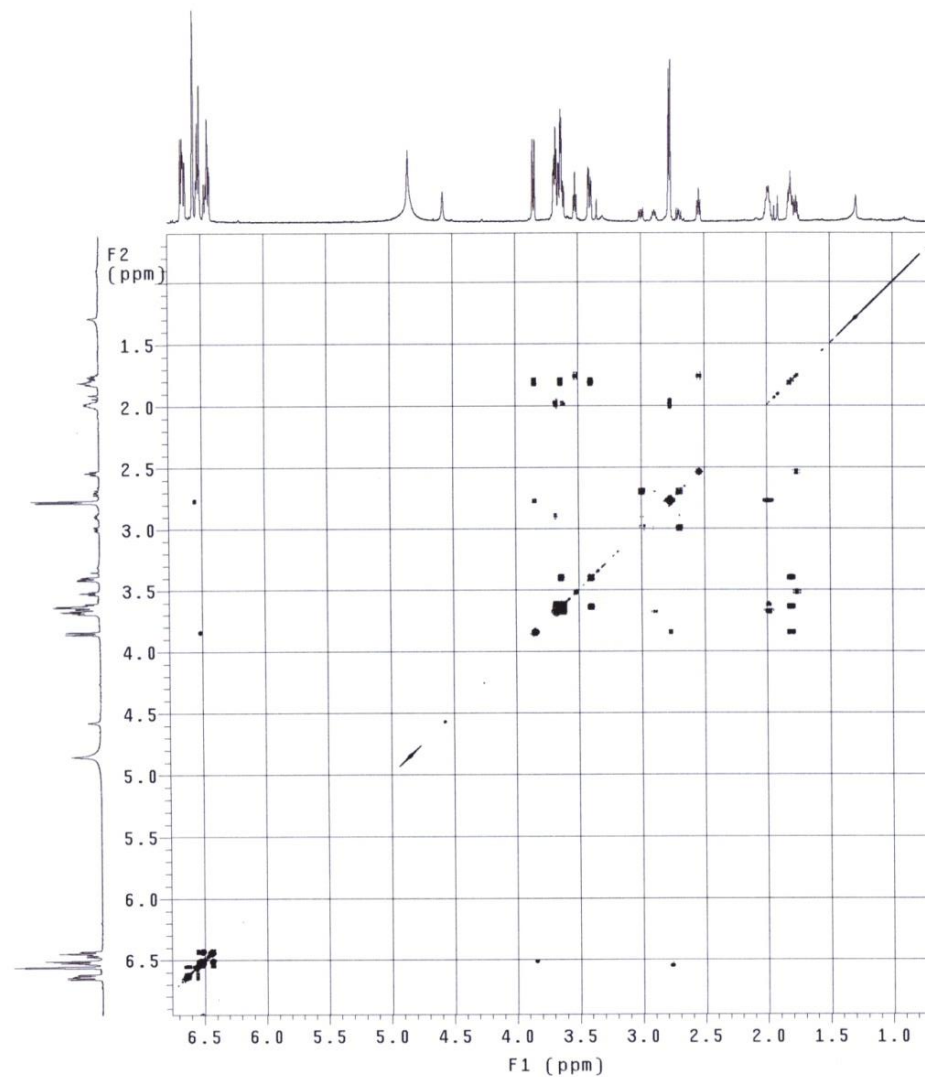


Figure S16: <sup>1</sup>H,<sup>1</sup>H COSY spectrum (CD<sub>3</sub>OD, 600 MHz) of compound **3**.

TXE3233-10-Y-1

File: xp

Pulse Sequence: gHMQC

Solvent: cd3od

Temp. 25.0 C / 298.1 K

Operator: vnmr1

VNMR5-600 "ynnmr600"

Relax. delay 1.000 sec

Acq. time 0.128 sec

Width 5319.1 Hz

20 Width 25641.0 Hz

32 repetitions

2 x 256 increments

OBSERVE H1, 599.8582843 MHz

DECOUPLE C13, 150.8457980 MHz

Power 26 dB

on during acquisition

off during delay

W40\_swpfg modulated

DATA PROCESSING

Gauss apodization 0.059 sec

F1 DATA PROCESSING

Gauss apodization 0.009 sec

FT size 2048 x 4096

Total time 5 hr, 21 min, 18 sec

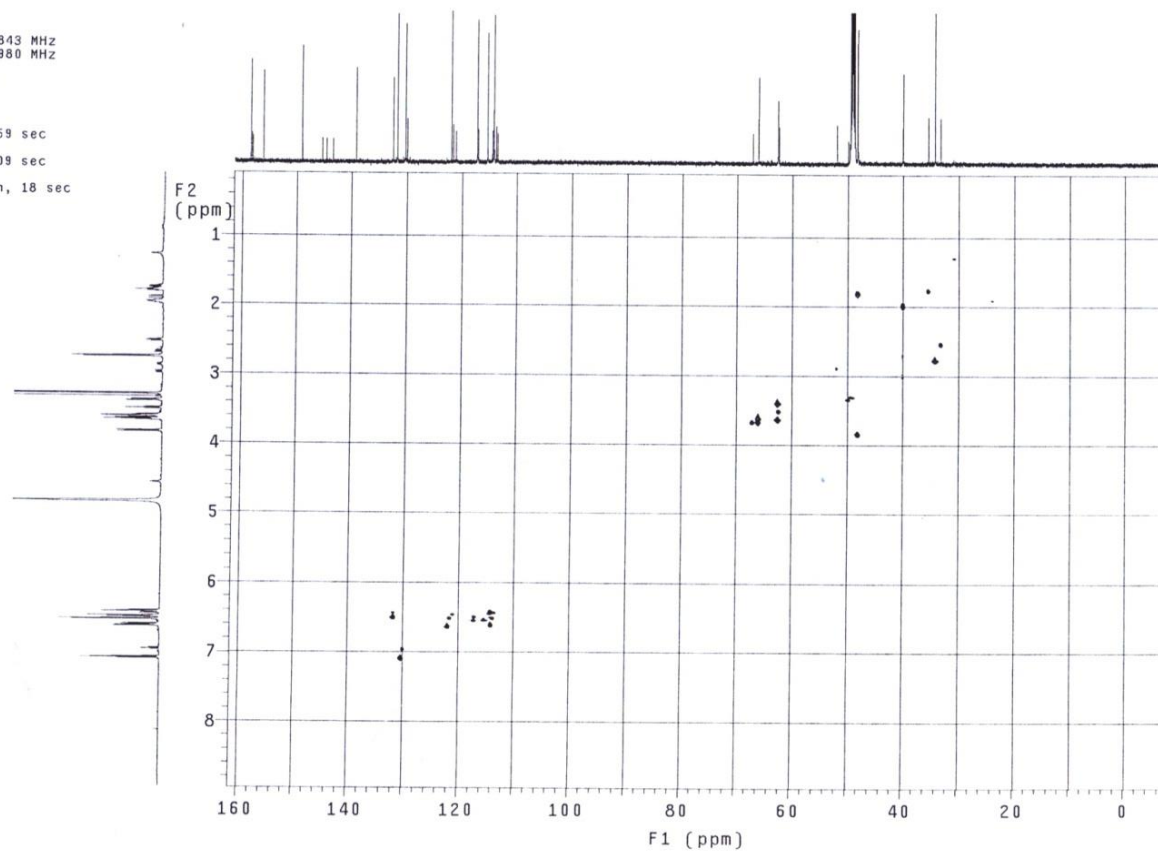


Figure S17: HMQC spectrum (CD<sub>3</sub>OD, 600 MHz) of compound 3.

TXE3233-10-Y-1

File: xp

Pulse Sequence: gHMBC

Solvent: cd3od

Temp. 25.0 C / 298.1 K

Operator: vnmr1

VNMR5-600 "vnmr600"

Relax. delay 1.000 sec

Mixing 0.080 sec

Acq. time 0.128 sec

Width 5319.1 Hz

2D Width 36199.1 Hz

32 repetitions

512 increments

OBSERVE H1, 599.8582872 MHz

DATA PROCESSING

Sine bell 0.084 sec

F1 DATA PROCESSING

Sine bell 0.014 sec

FT size 2048 x 4096

Total time 5 hr, 34 min, 4 sec

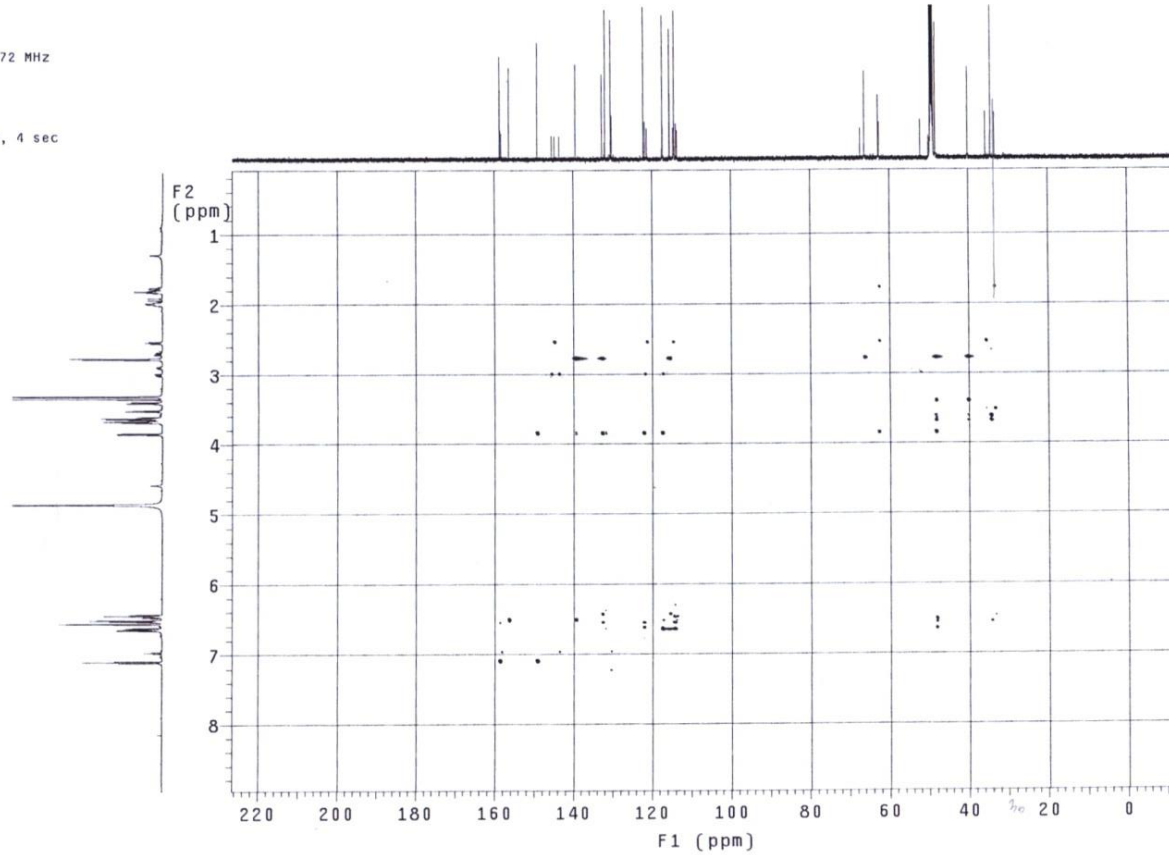


Figure S18: HMBC spectrum (CD<sub>3</sub>OD, 600 MHz) of compound **3**.

TXE3233-10-Y-1

File: xp

Pulse Sequence: NOESY

Solvent: cd3od  
Temp. 25.0 C / 298.1 K  
Operator: vmmr1  
VNMR-600 "ynnmr600"

Relax. delay 1.000 sec  
Mixing 0.700 sec  
Acq. time 0.161 sec  
Width 6345.2 Hz  
2D Width 6345.2 Hz  
16 repetitions  
2 x 512 increments  
OBSERVE H1, 599.8582877 MHz  
DATA PROCESSING  
Gauss apodization 0.075 sec  
F1 DATA PROCESSING  
Gauss apodization 0.074 sec  
FT size 4096 x 4096  
Total time 8 hr, 30 min, 23 sec

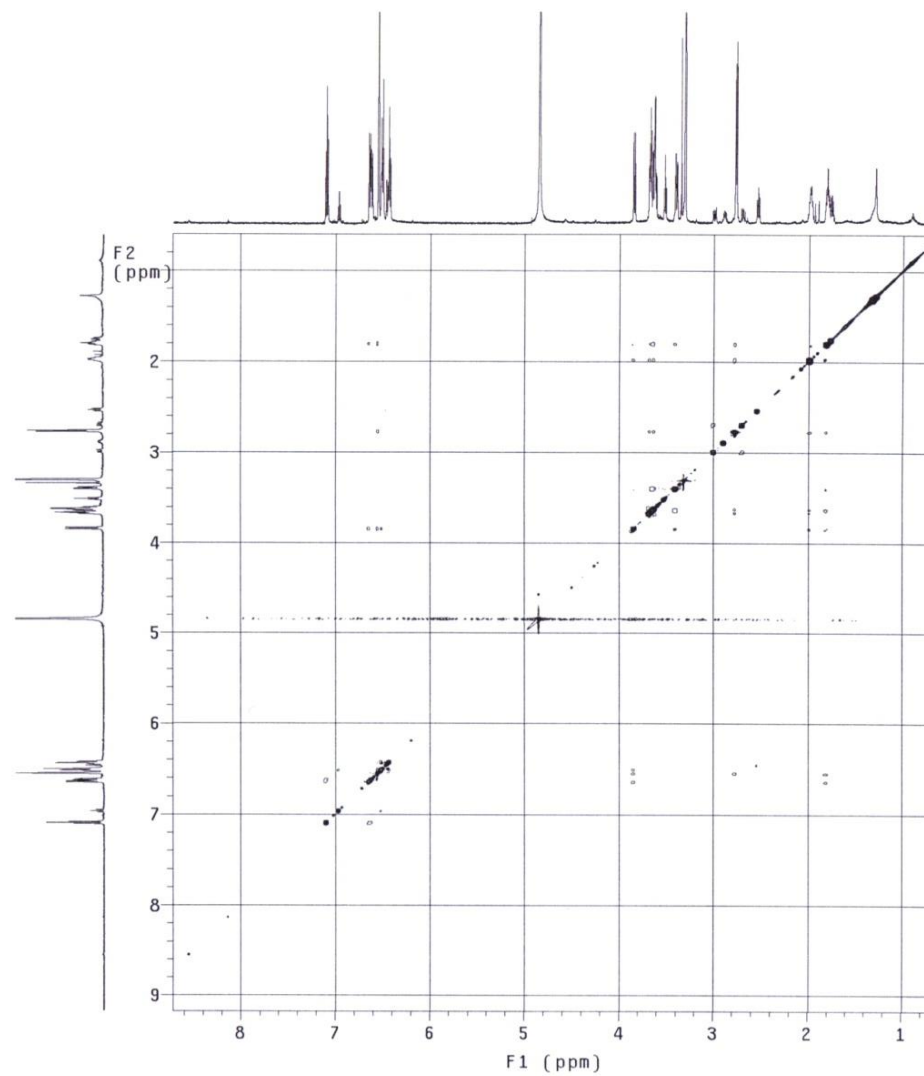


Figure S19: NOESY spectrum (CD<sub>3</sub>OD, 600 MHz) of compound **3**.

[ Elemental Composition ]

Data : EI636  
Sample: TXE 3  
Note : -  
Inlet : Direct  
RT : 0.31 min  
Elements : C 40/0, H 49/0, O 10/0  
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5  
Unsaturation (U.S.) : -0.5 - 10.0

Date : 01-Apr-2014 20:03

Page: 1

Ion Mode : EI+  
Scan#: (14,16)

Observed m/z	Int%	Err [ppm / mmu]	U.S.	Composition
300.1360	100.0	-0.7 / -0.2	9.0	C 18 H 20 O 4

[ Theoretical Ion Distribution ]

Molecular Formula : C18 H20 O4  
(m/z 300.1362, MW 300.3544, U.S. 9.0)  
Base Peak : 300.1362, Averaged MW : 300.3524(a), 300.3532(w)

Page: 1

m/z	INT.	
300.1362	100.0000	*****
301.1395	20.1726	*****
302.1422	2.7252	**
303.1448	0.2766	
304.1474	0.0226	
305.1500	0.0016	

Figure S20: HREIMS of compound 3.