

SUPPLEMENTARY MATERIAL

Isolaurenidificin and Bromlaurenidificin, two new C₁₅-acetogenins from the red alga *Laurencia obtusa*

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Isolaurenidificin and Bromlaurenidificin, two new C₁₅-acetogenins from the red alga *Laurencia obtusa*

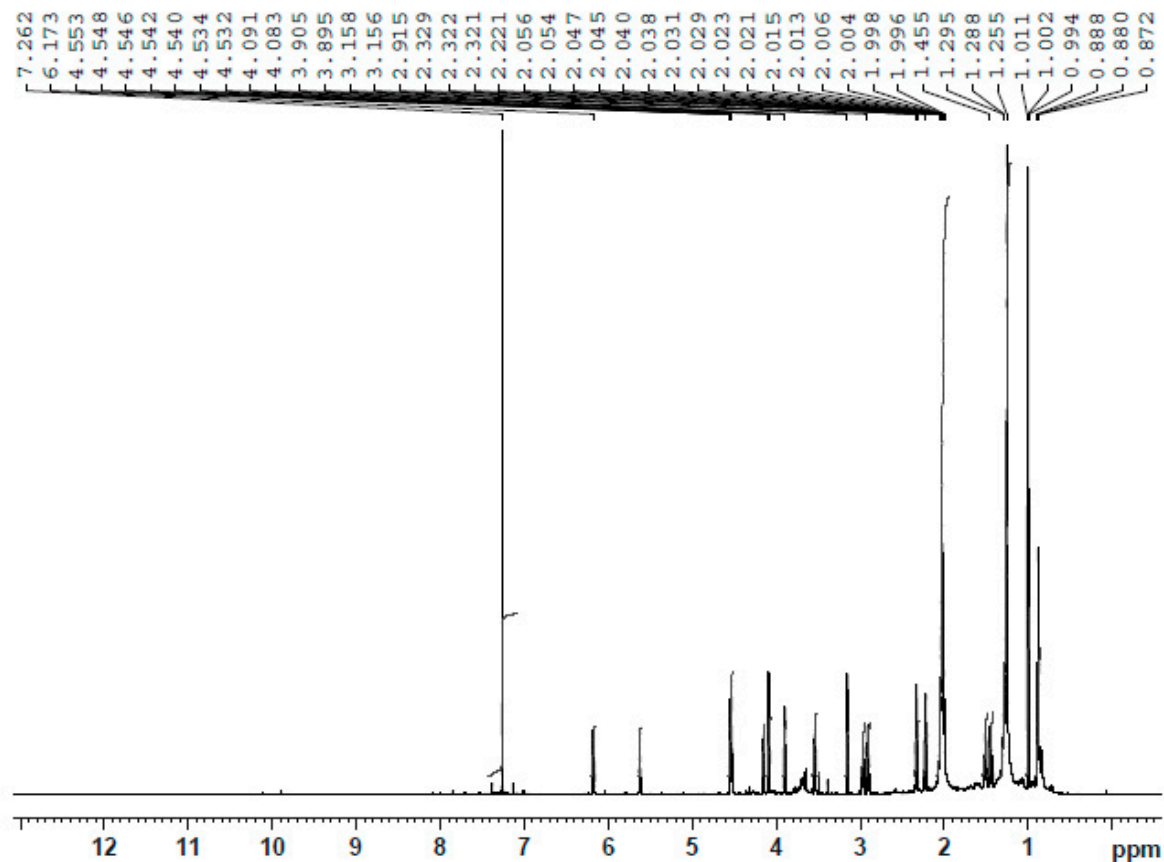
Abstract

Chromatographic investigation of the CH₂Cl₂/MeOH extract of the Red Sea red alga *Laurencia obtusa* gave two new hexahydrofuro[3,2-b]furan-based C₁₅-acetogenin, namely, isolaurenidificin (**1**) and bromlaurenidificin (**2**). Their chemical structures were elucidated based on extensive analyses of their spectral data. The apoptosis-inducing or inhibiting effect of both compounds on apoptosis of peripheral blood neutrophils was studied.

Keywords: Marine algae; Fatty acids; halogenations; spectroscopy; Anti-inflammatory.

Dr.Nahed Bawaked

Sample : BO-183-5 CDCL3



Current Data Parameters
NAME NAHRD BO-1835 07-12-2016
EXPRO 40
PROCNO 1

F2 - Acquisition Parameters
Date 20161207
Time 14.35
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 17006.803 Hz
FIDRES 0.259503 Hz
AQ 1.9267584 sec
RG 10.55
DW 29.400 usec
DR 10.00 usec
TR 298.0 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 850.1552500 MHz
NUC1 1H
P1 8.00 usec
PLW1 15.30000019 W

F2 - Processing parameters
SI 65536
SF 850.1500200 MHz
WDW RM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

Figure S1a: ¹H NMR of compound 1

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Sample : BO-183-5 CDCL3

6.194
6.194
6.186
6.182
6.181
6.178
6.177
6.173
6.165
6.154

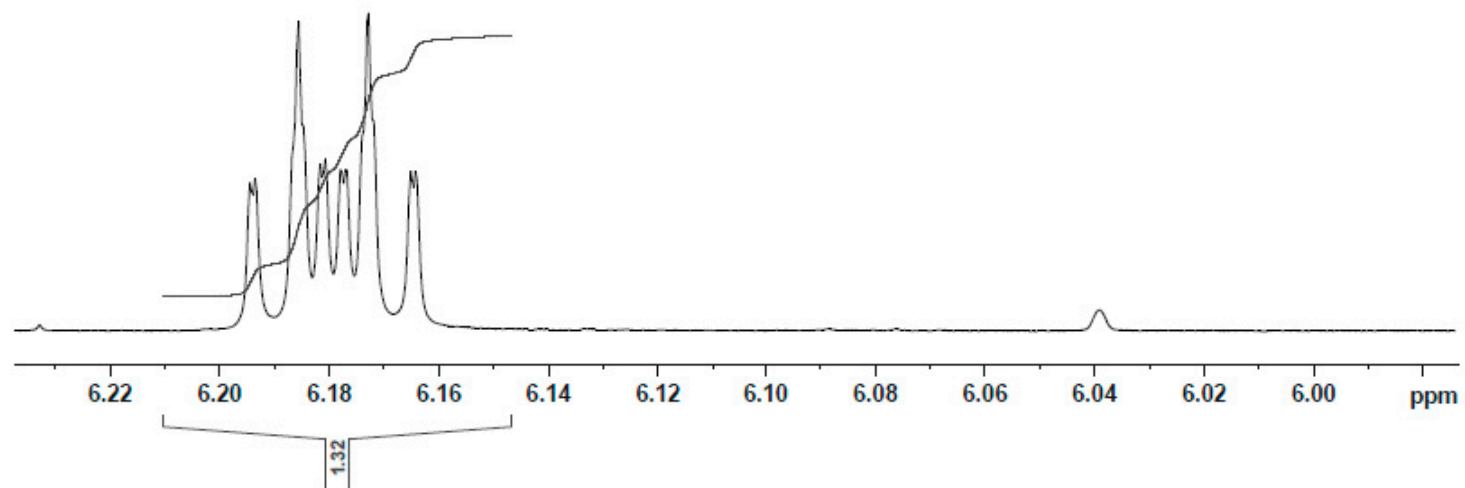


Figure S1b: ¹H NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

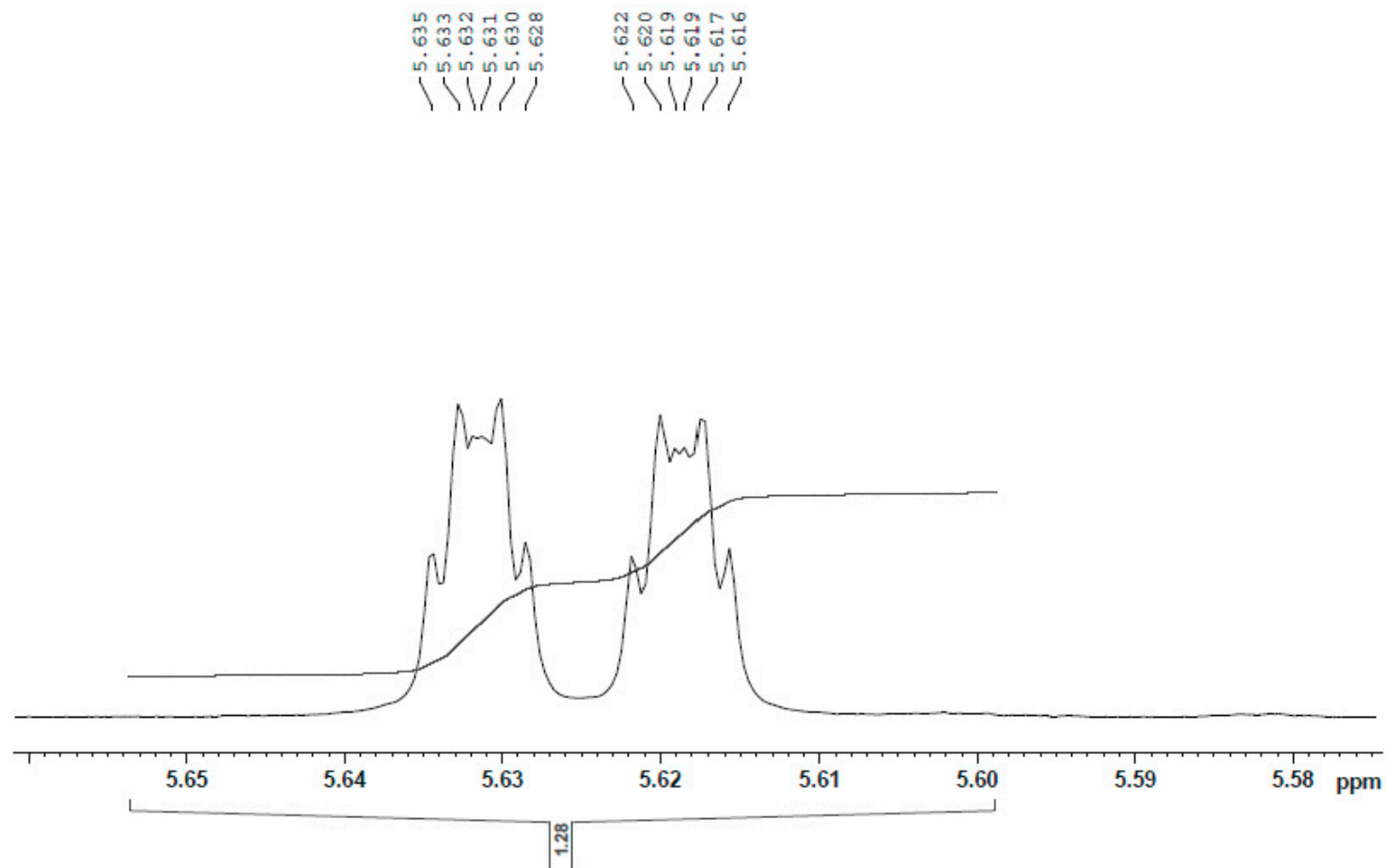


Figure S1c: ¹H NMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCL3

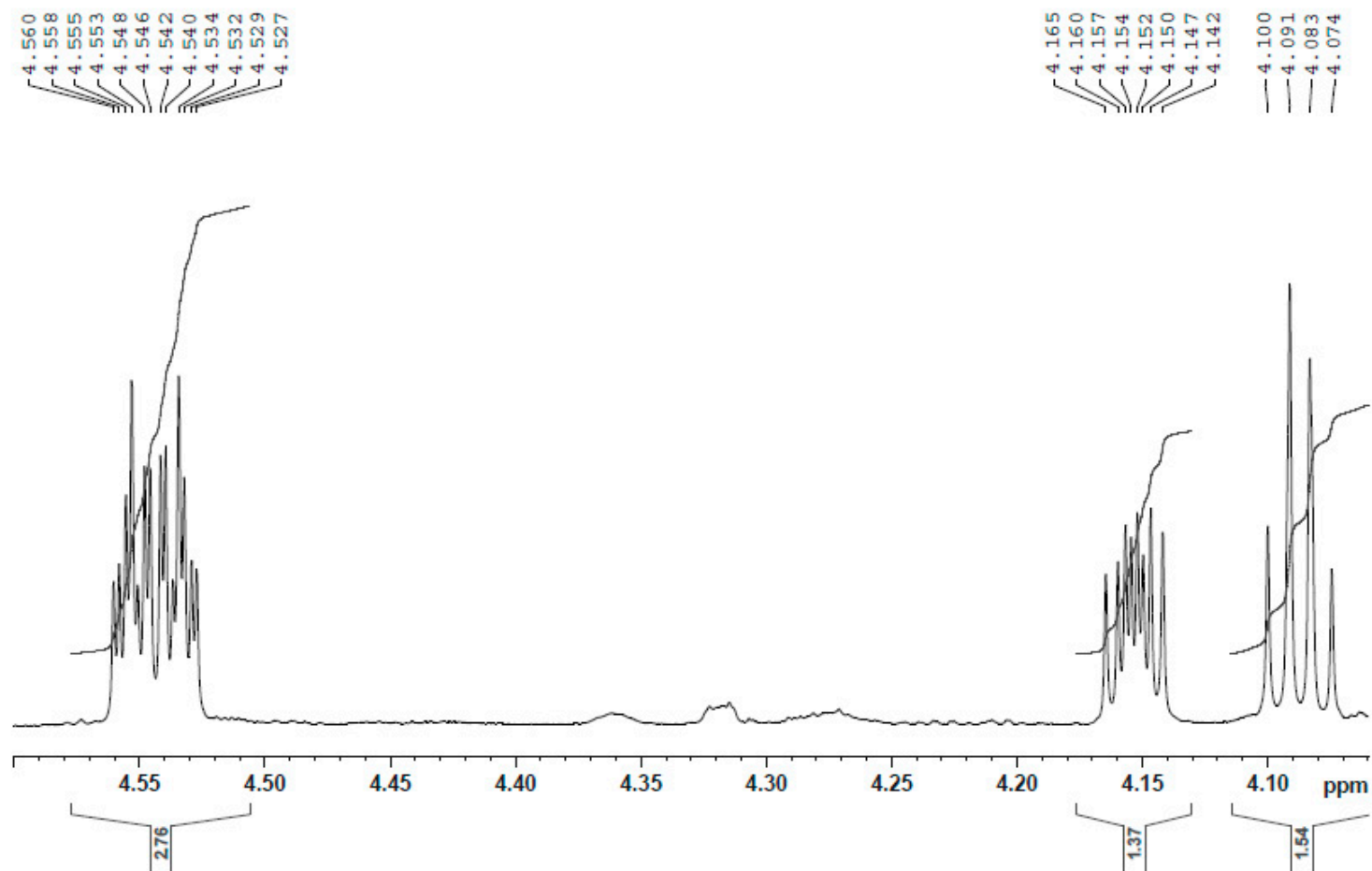


Figure S1d: ^1H NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

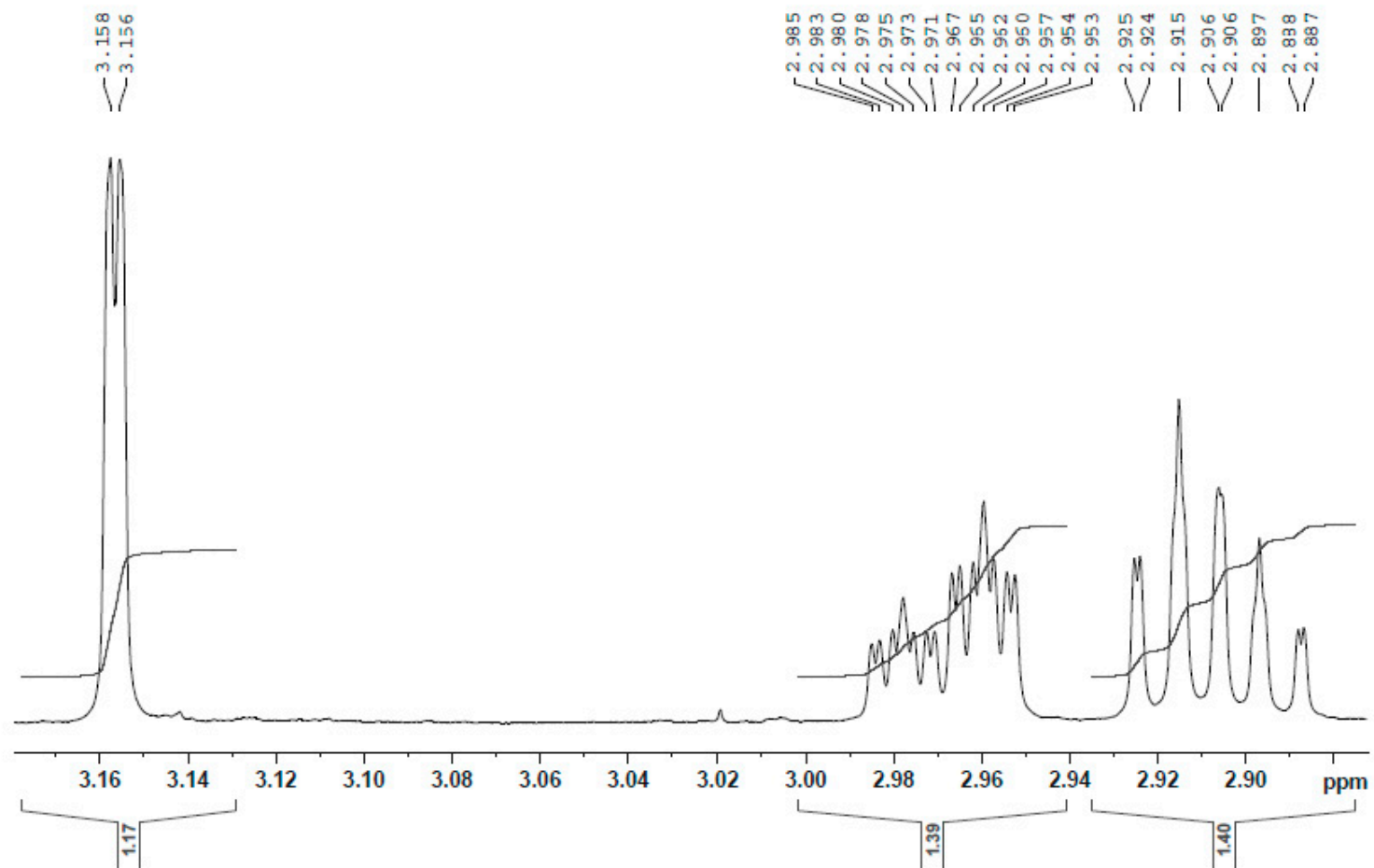
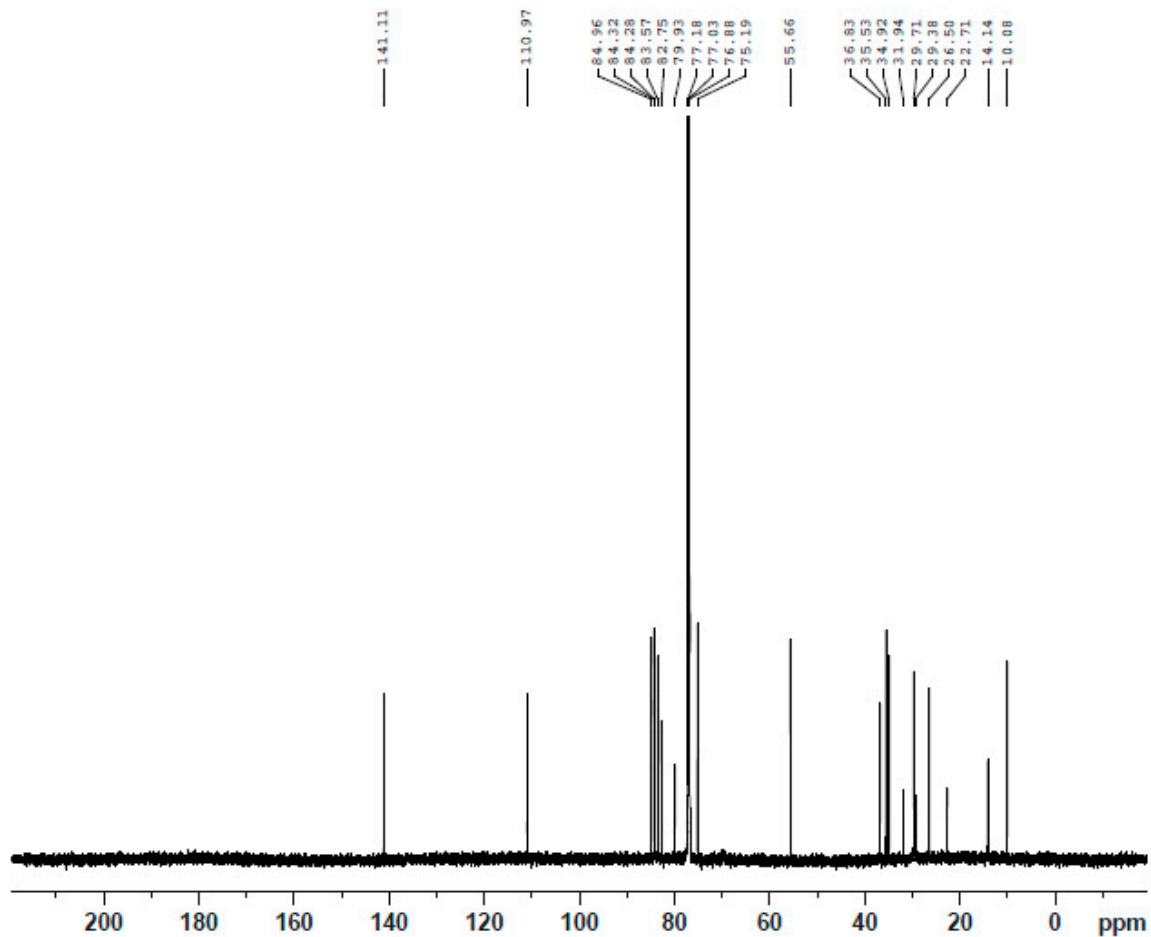


Figure S1e: ¹HNMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCL3



Current Data Parameters
NAME NAMED BO-1835 07-12-2016
EXPNO 41
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161208
Time 20.24
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 51020.406 Hz
FIDRES 0.778510 Hz
AQ 0.6422528 sec
RG 186.93
RW 9.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 213.7917636 MHz
NUC1 13C
P1 12.00 usec
PLW1 130.0000000 W

===== CHANNEL f2 =====
SFO2 850.1534006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 13.8000019 W
PLW12 0.1380000 W
PLW13 0.08832000 W

F2 - Processing parameters
SI 32768
SF 213.7703875 MHz
WDW EM
SCB 0
LB 1.50 Hz
CB 0
PC 2.00

Figure S1f: ¹³CNMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCL3

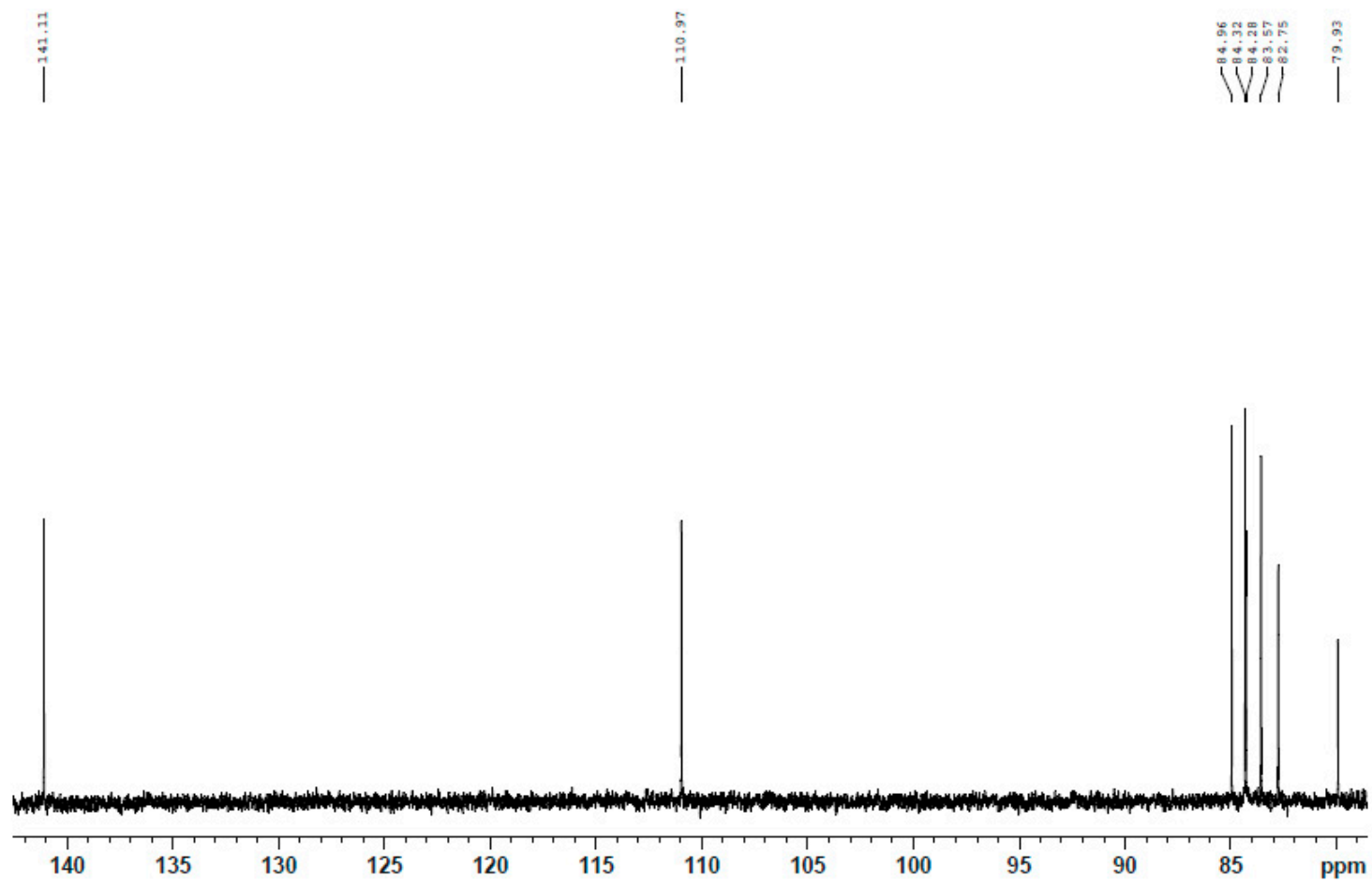
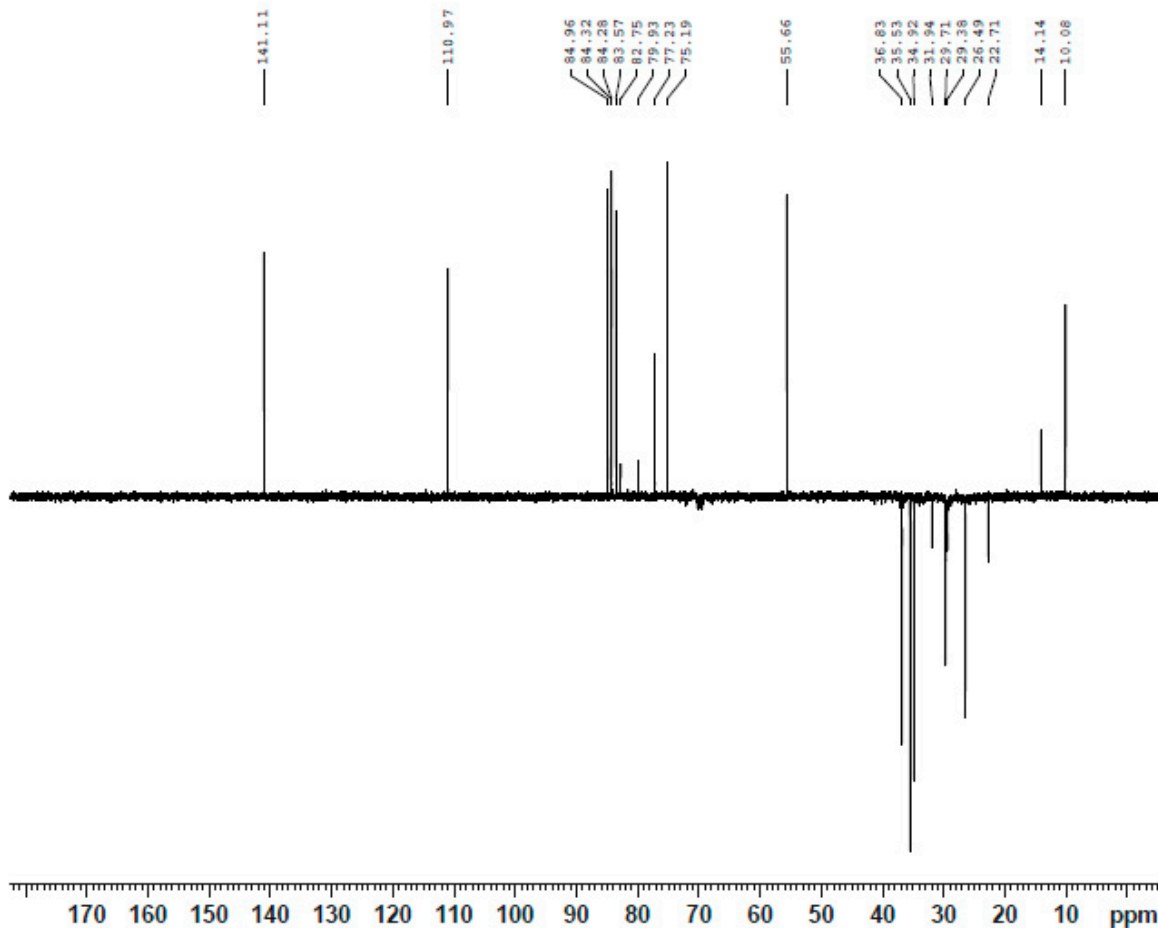


Figure S1g: ^{13}C NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3



Current Data Parameters
NAME NAHD BO-1835 07-12-2016
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161208
Time 21.19
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG deptsol35
TD 65536
SOLVENT CDCL3
NS 1200
DS 4
SWH 51020.406 Hz
FIDRES 0.778510 Hz
AQ 0.6422528 sec
RG 166.86
RW 9.800 usec
DS 18.00 usec
TE 298.0 K
CNET2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

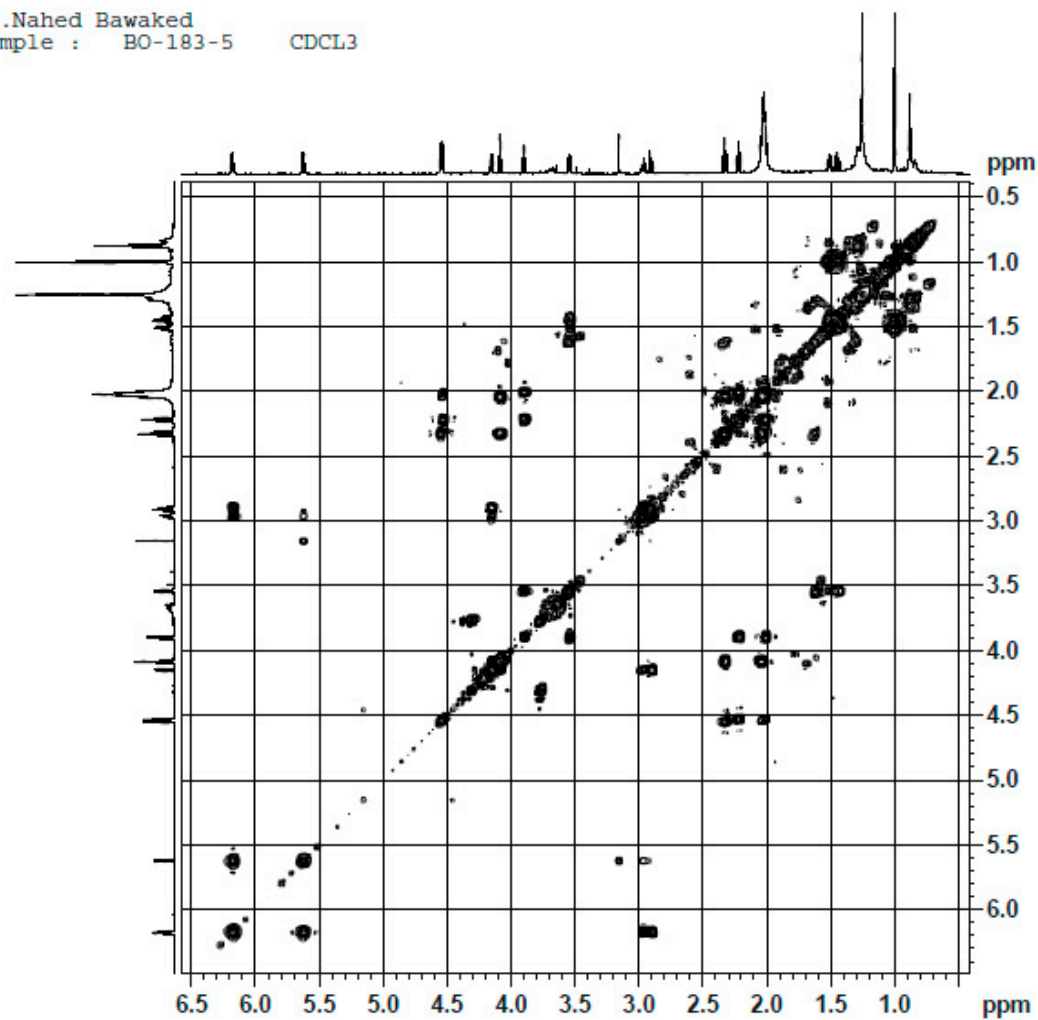
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NUC1 13C
P1 12.00 usec
P13 2000.00 usec
PLM0 0 W
PLM1 130.00000000 W
SFOALS Crp60comp.4
SFOALS 0.500
SFOFSS 0 Hz
SFOFS 28.60199928 W

===== CHANNEL f2 =====
SFO2 850.1534006 MHz
NUC2 1H
CDDPRG2 waltz16
P3 8.00 usec
P4 16.00 usec
PCPD2 80.00 usec
PLM2 13.80000019 W
PLM12 0.13800000 W

F2 - Processing parameters
SI 32768
SF 213.7703875 MHz
WUW IM
SGB 0
LB 1.50 Hz
GB 0
PC 2.00

Figure S1h: DEPT NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3



```
Current Data Parameters
NAME      NAHED   BO-1835   07-12-2016
EXPNO    44
PROCNO   1

F2 - Acquisition Parameters
Date_    20161208
Time     21.22
INSTRUM  spect
PROBHD   5 mm CPQCI 1H-
PULPROG  cosygpmtqf
TD        2048
SOLVENT  CDCl3
NS        32
DS        8
SWH       7812.500 Hz
FIDRES    3.814697 Hz
AQ        0.1310720 sec
RG        186.93
EW        64.000 usec
EK        10.00 usec
TK        298.0 K
DQ        0.00000300 sec
DI        1.95904005 sec
DI3       0.00000400 sec
DI6       0.00020000 sec
IN0       0.00012800 sec

===== CHANNEL f1 =====
SFO1     850.1532536 MHz
NUC1      1H
P1        8.00 usec
PLW1     15.30000019 W

===== GRADIENT CHANNEL =====
GPNAM(1) SMSQ10.100
GPNAM(2) SMSQ10.100
GPNAM(3) SMSQ10.100
CPE1     16.00 %
CPE2     12.00 %
CPE3     40.00 %
P16      1000.00 usec

F1 - Acquisition parameters
TD        128
SFO1     850.1533 MHz
FIDRES    61.035156 Hz
SW        9.190 ppm
PnMODE    QF

F2 - Processing parameters
SI        1024
SF        850.1500200 MHz
WDW       SINC
SSB       0
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
MC2       QF
SF        850.1500200 MHz
WDW       SINC
SSB       0
LB        0 Hz
GB        0
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Figure S1i: COSY NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

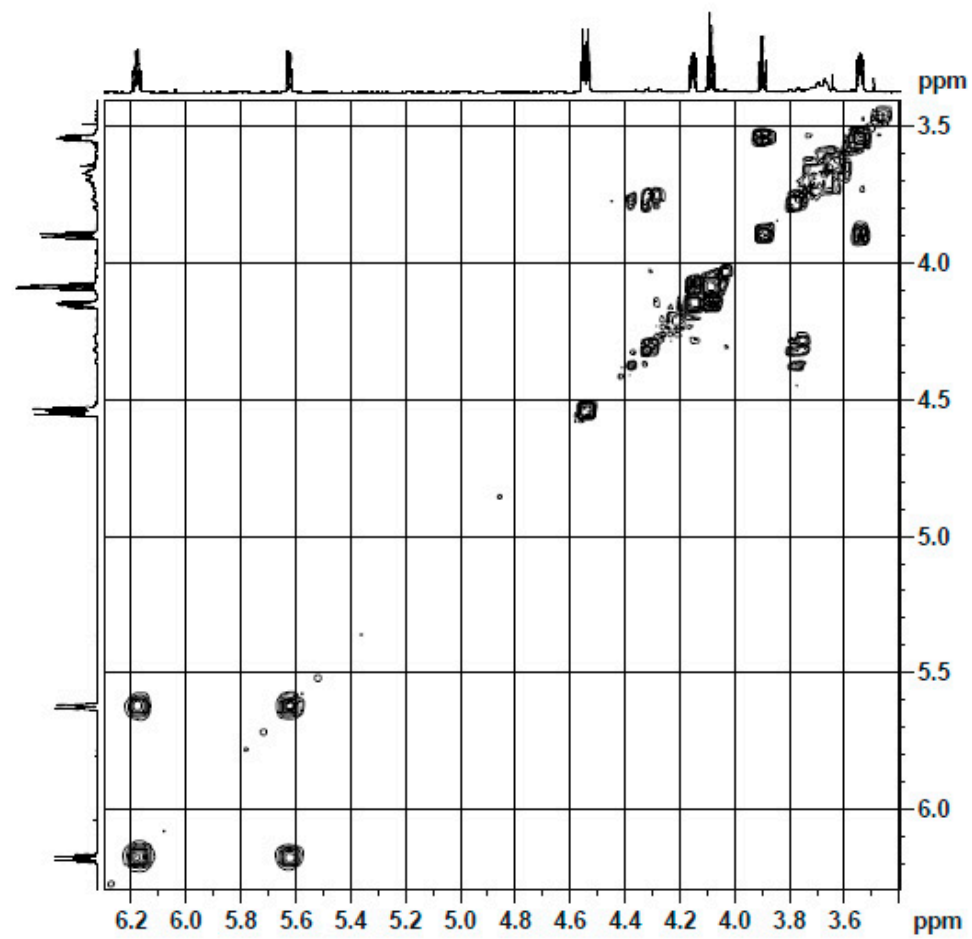


Figure S1j: COSY NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

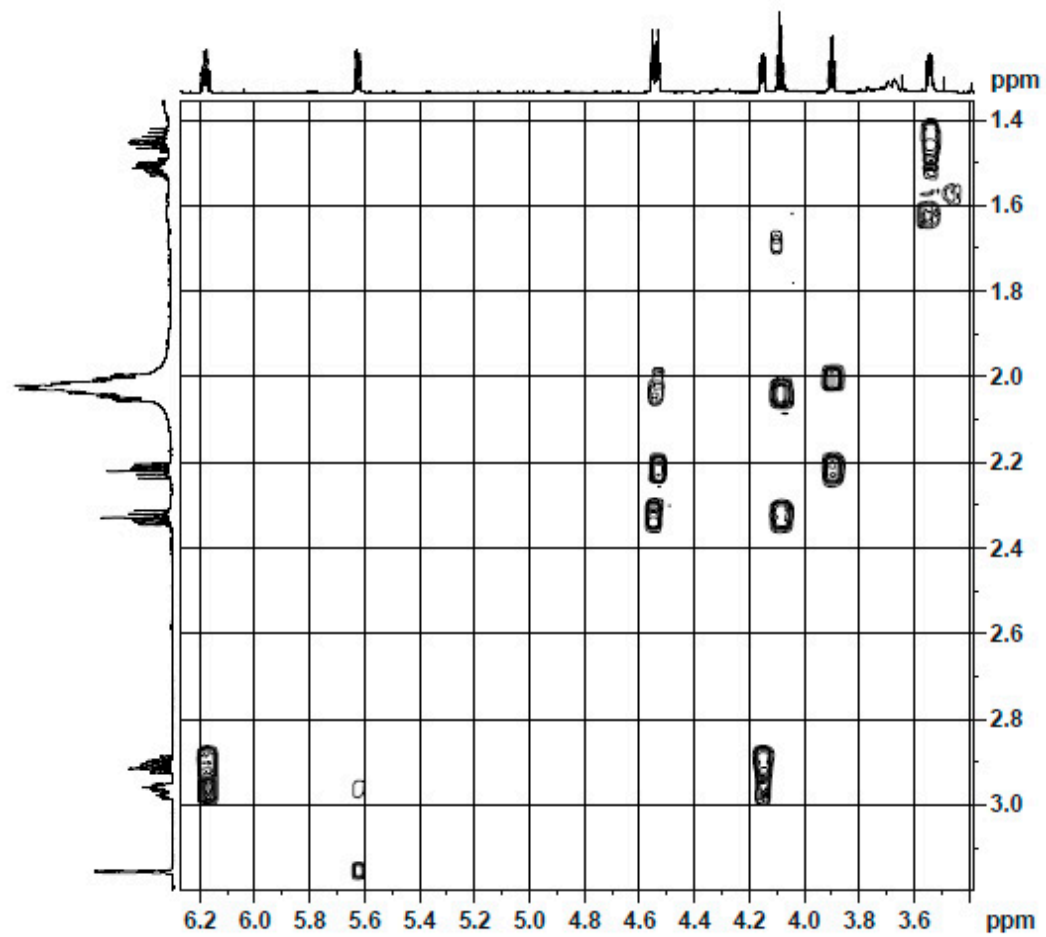


Figure S1k: COSY NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

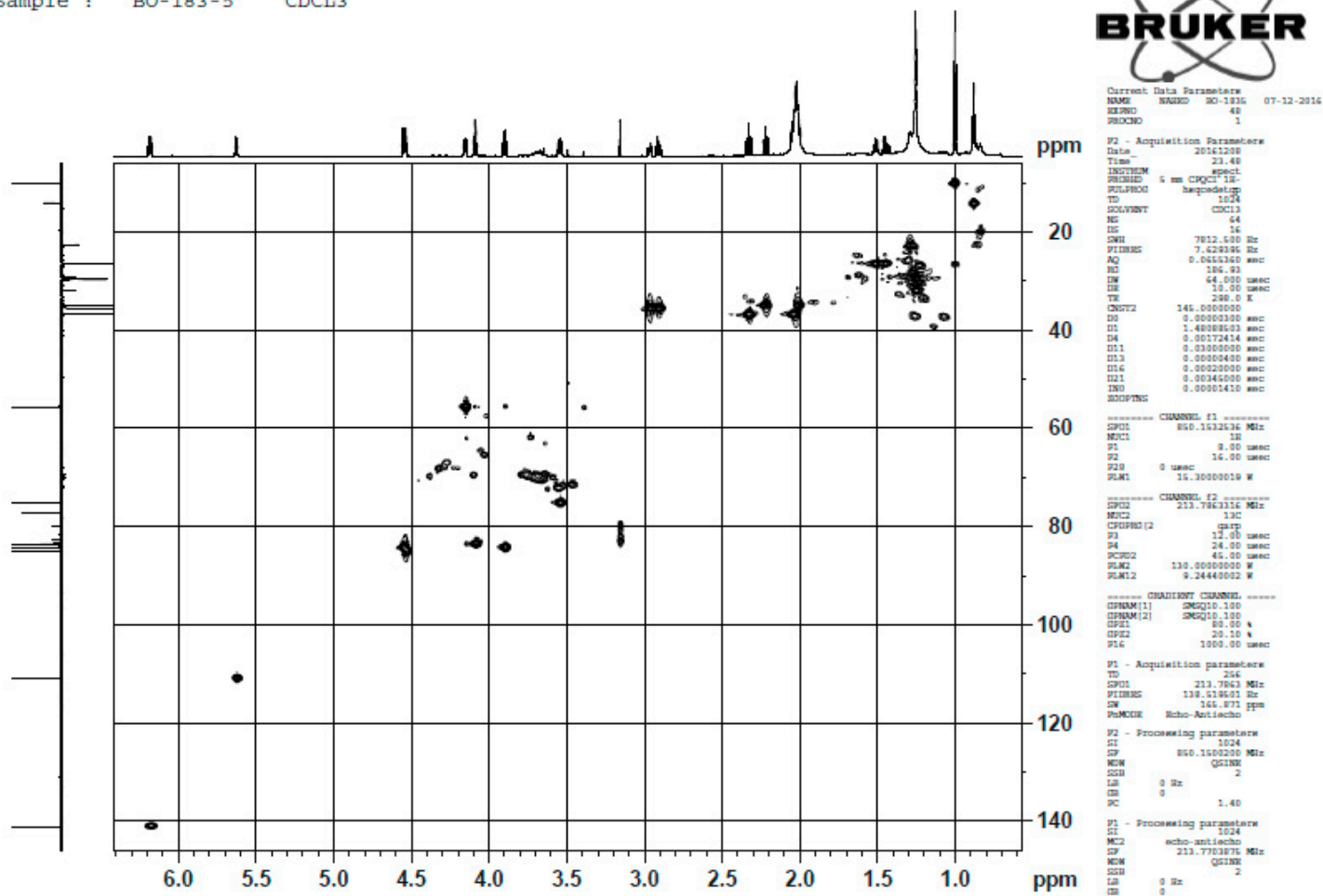


Figure S11: HSQC NMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCl₃

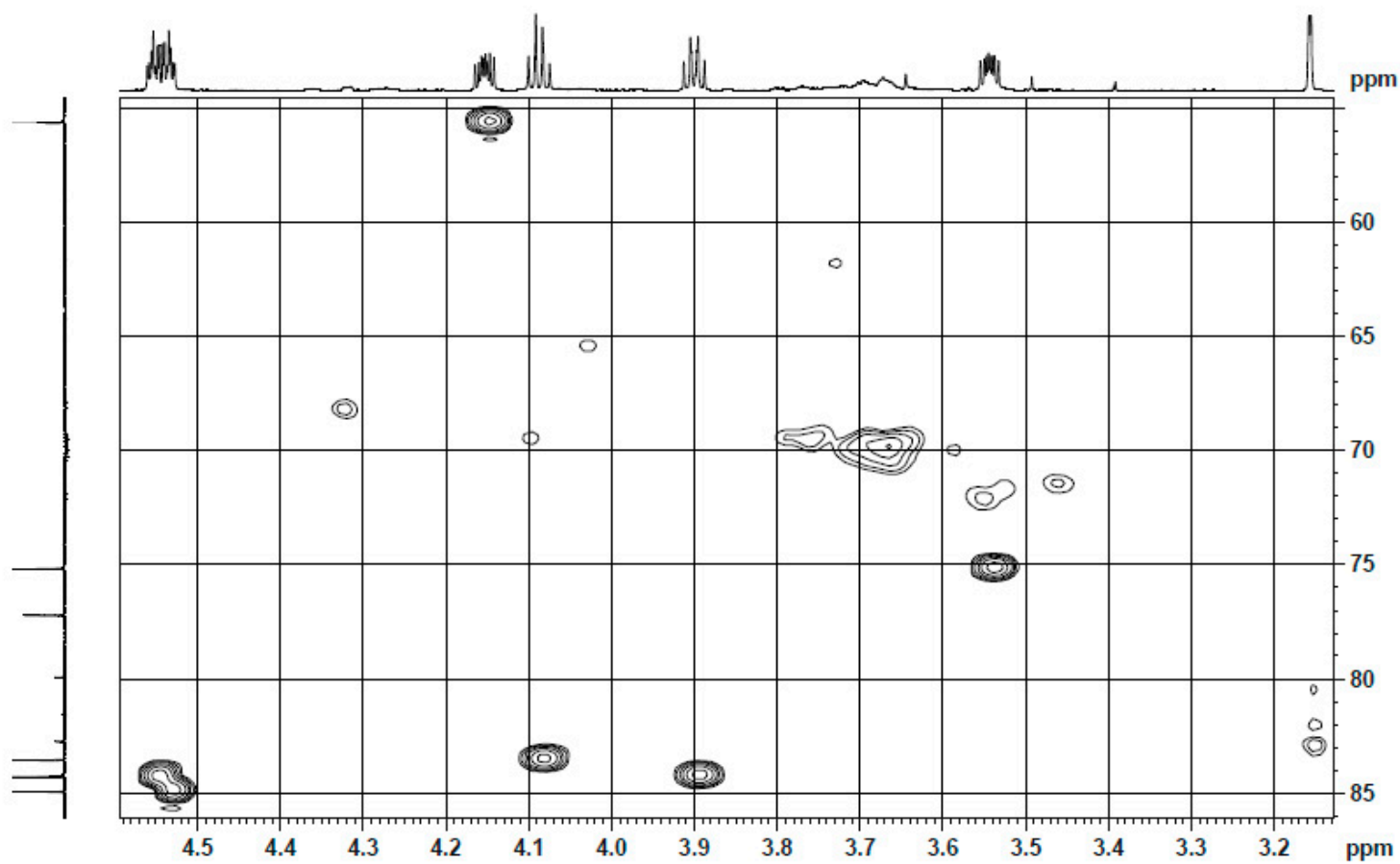


Figure S1m: HSQC NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

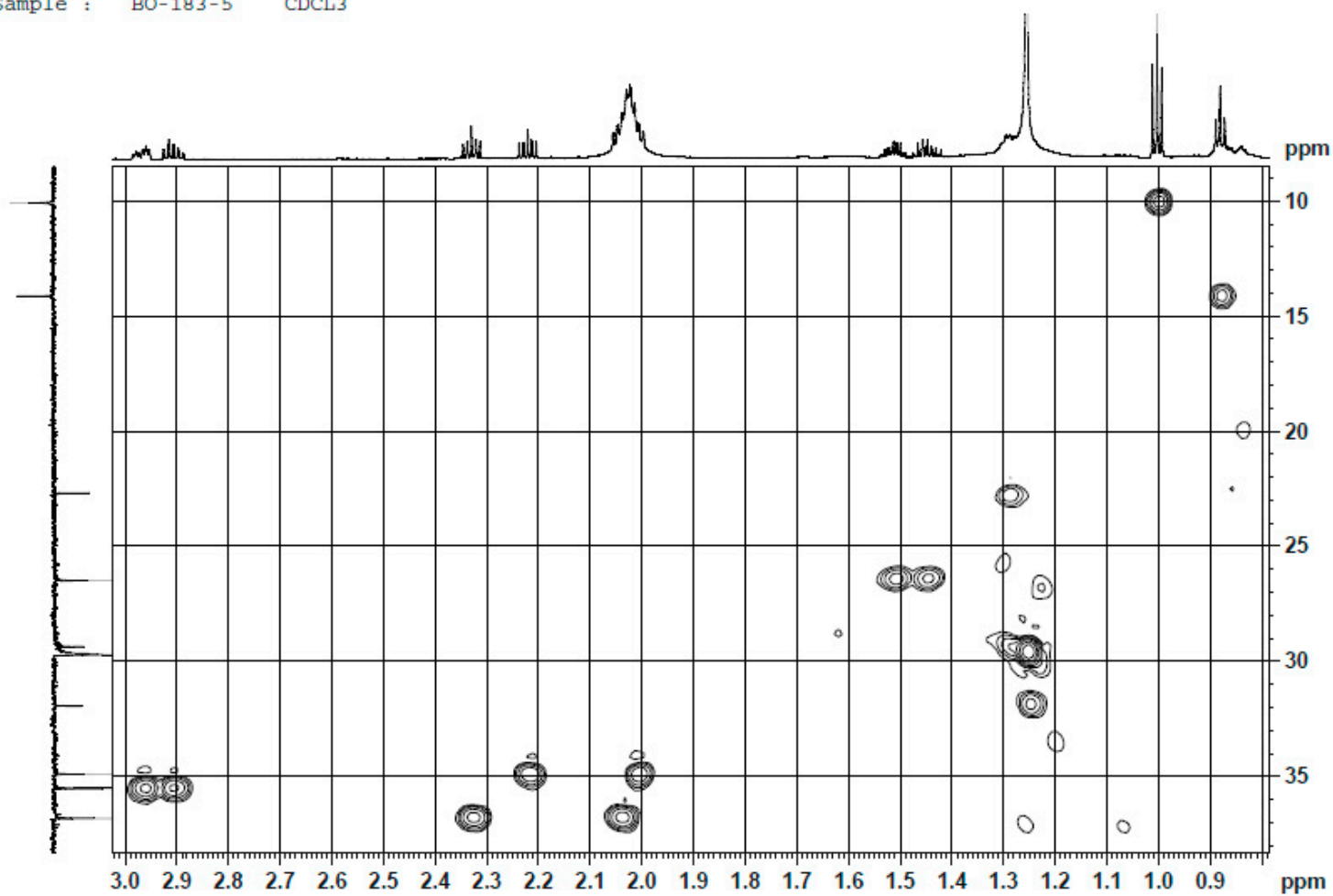


Figure S1n: HSQC NMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCL3

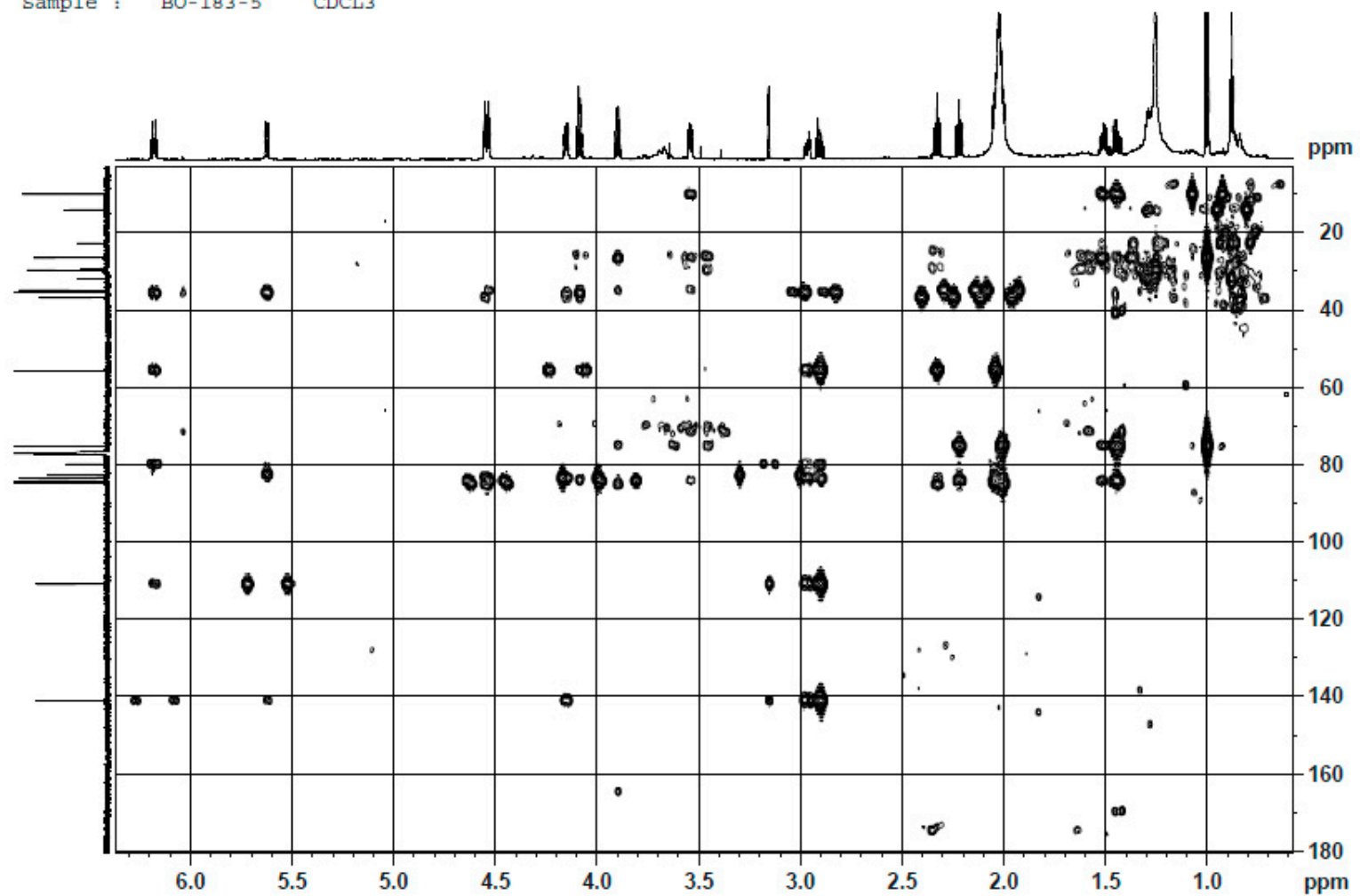


Figure S10: HMBC NMR of compound 1

Dr. Nahed Bawaked
Sample : BO-183-5 CDCL3

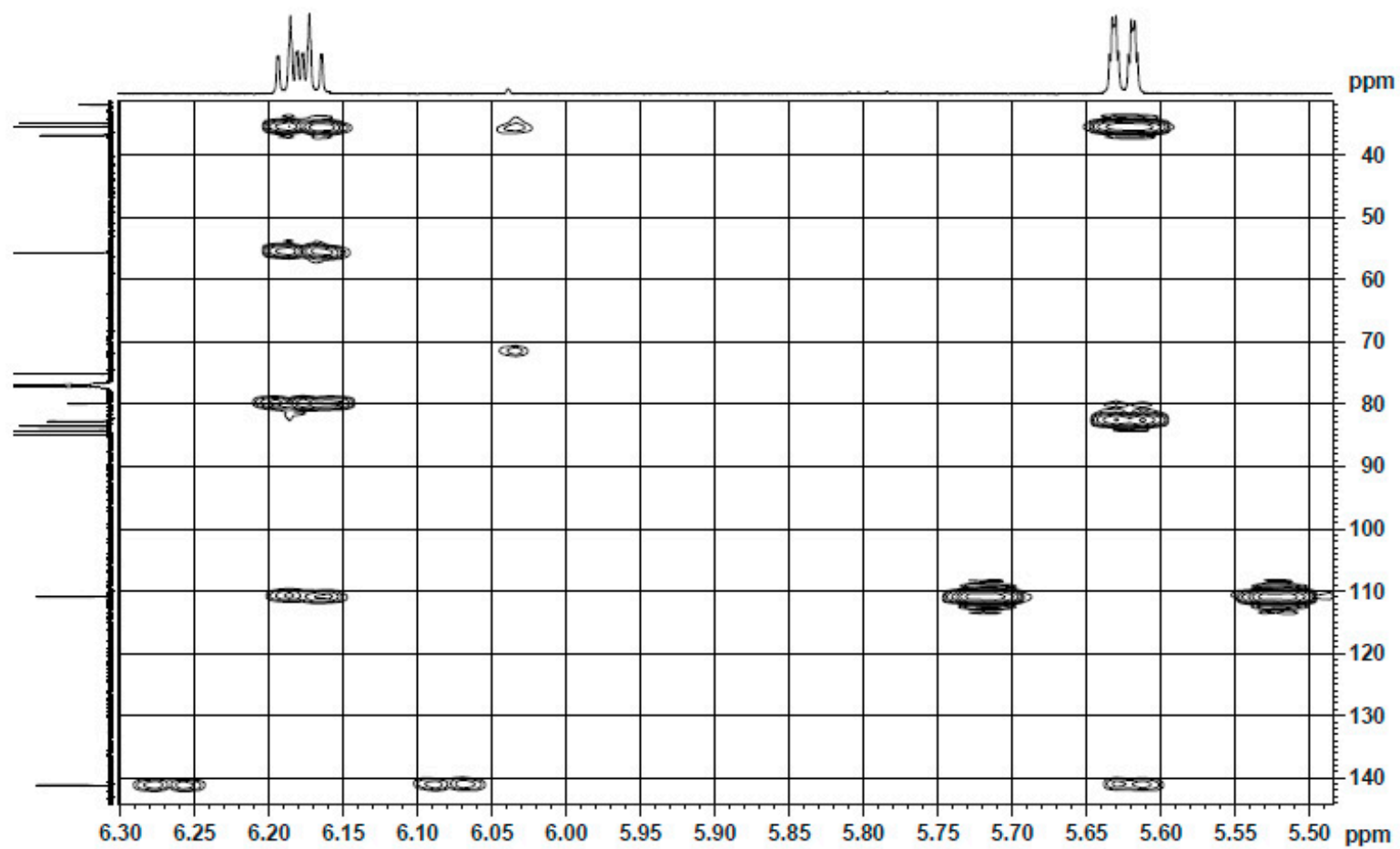


Figure S1p: HMBC NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3

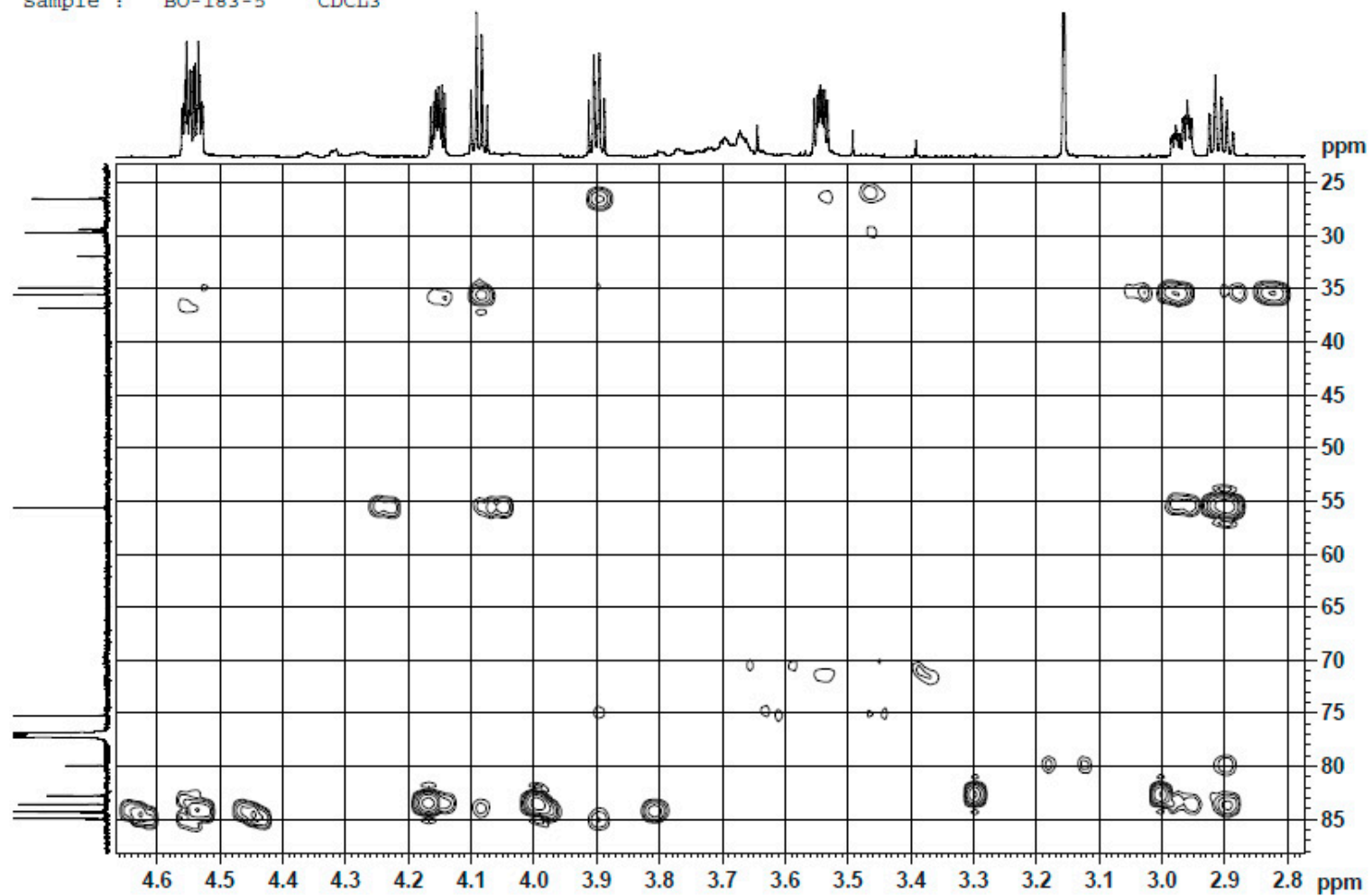
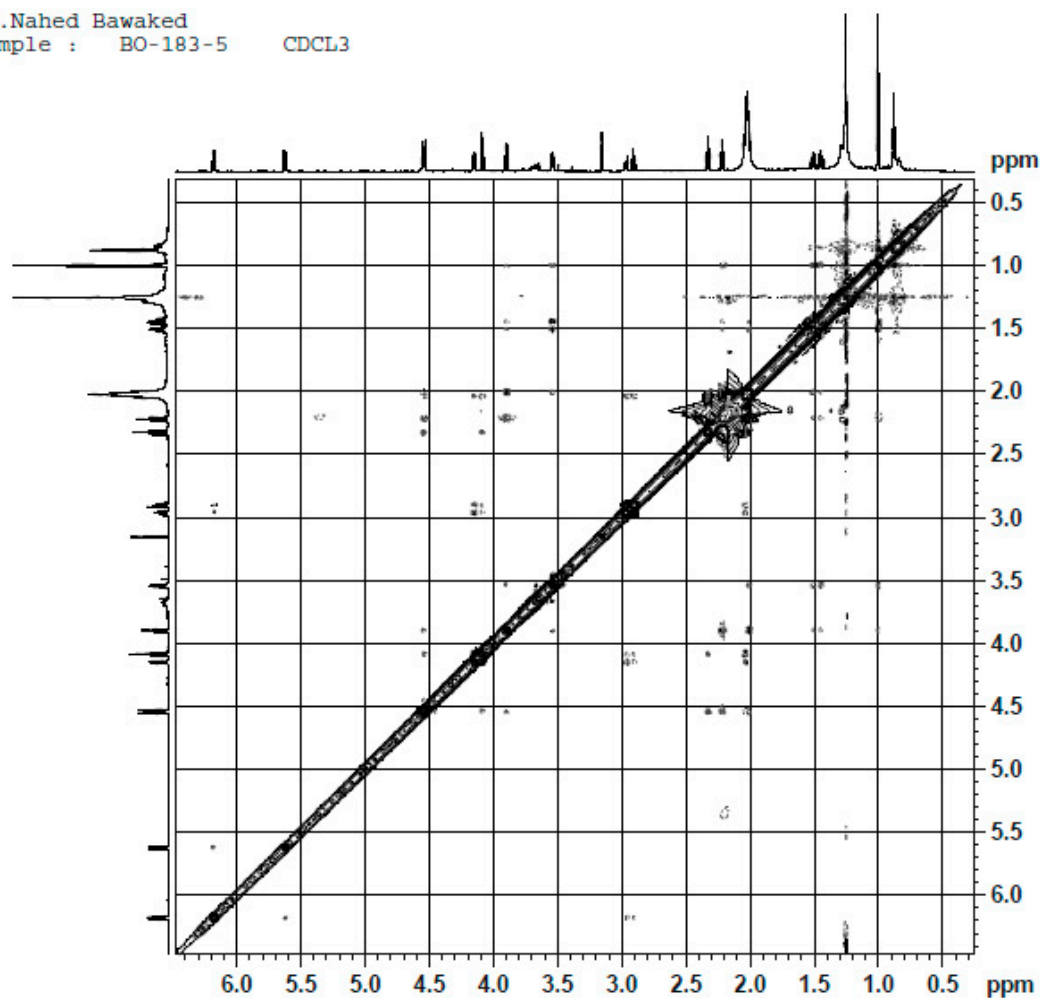


Figure S1q: HMBC NMR of compound 1

Dr.Nahed Bawaked
Sample : BO-183-5 CDCL3



```
Current Data Parameters
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EXPNO     50
PROCNO    1

F2 - Acquisition Parameters
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PULPROG   noesygpppp
TD        2048
SOLVENT   cdcl3
NS        32
DS        32
SWH        7812.500 Hz
FIDRES    3.814697 Hz
AQ         0.1310720 sec
RG         17.28
DSW        64.0000 usec
DE         10.00 usec
TE         298.0 K
D0         0.00002001 sec
D1         1.9934996 sec
D8         0.30000001 sec
D11        0.03000000 sec
D12        0.00002000 sec
D16        0.00020000 sec
RG         0.00012800 sec

===== CHANNEL f1 =====
SFO1      850.132536 MHz
NUC1      1H
P1         8.00 usec
P2         16.00 usec
P17        2500.00 usec
PLW1      15.30000019 W
PLW10     1.70000005 W

===== GRADIENT CHANNEL =====
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GPE1      40.00 %
P16        1000.00 usec

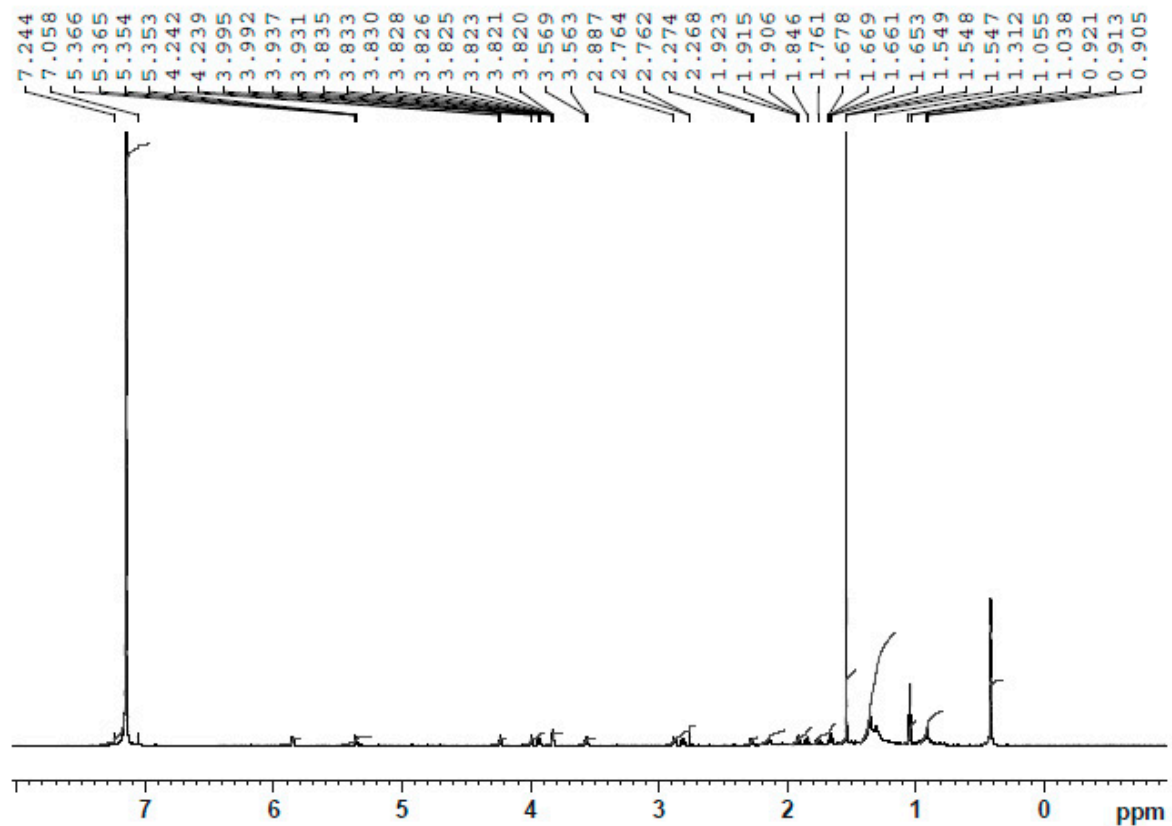
F1 - Acquisition parameters
TD         256
SFO1      850.1333 MHz
FIDRES    30.517578 Hz
SW         9.190 ppm
PnMODE    States-TFPI

F2 - Processing parameters
SI         1024
SF         850.1500200 MHz
WDW        GQ138
SSB        2
LB         0 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TFPI
SF         850.1500200 MHz
WDW        GQ138
SSB        2
LB         0 Hz
GB         0
```

Figure S1r: NOESY NMR of compound 1

Dr.Nahed Bawaked
Sample BO-68-5 C6D6



Current Data Parameters
NAME NAHEB BO-68-5 9-1-2017
EXPNO 130
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170109
Time 13.19
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 32
DS 2
SWH 17006.803 Hz
FIDRES 0.259503 Hz
AQ 1.9267584 sec
RG 10.55
DW 29.400 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 850.1552500 MHz
NUC1 1H
P1 8.00 usec
PLW1 15.30000019 W

F2 - Processing parameters
SI 65536
SF 850.1500000 MHz
WDW RM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

Figure S2a: ¹H NMR of compound 2

Dr. Nahed Bawaked
Sample BO-68-5 C6D6

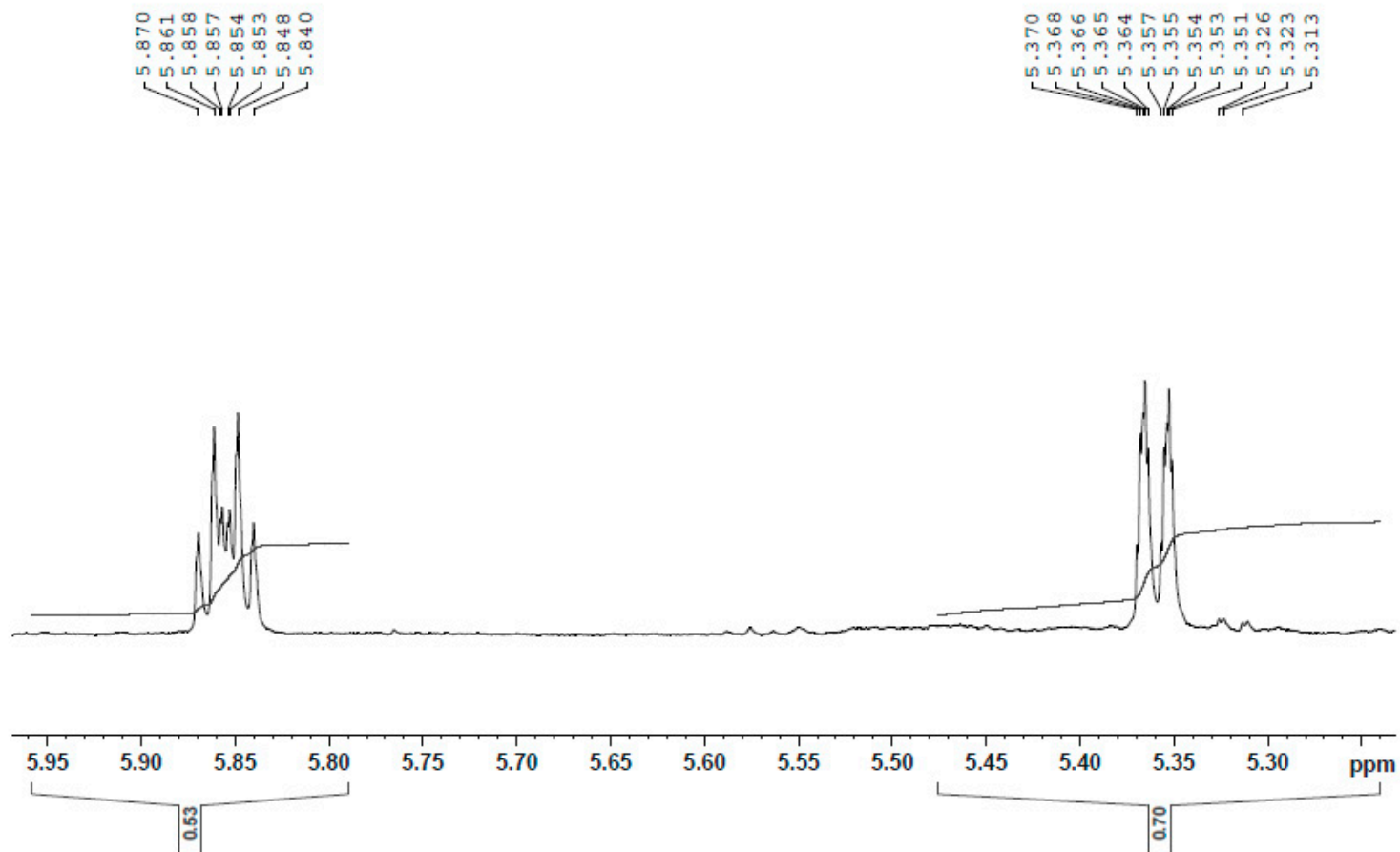


Figure S2b: ^1H NMR of compound 2

Dr. Nahed Bawaked
Sample BO-68-5 C6D6

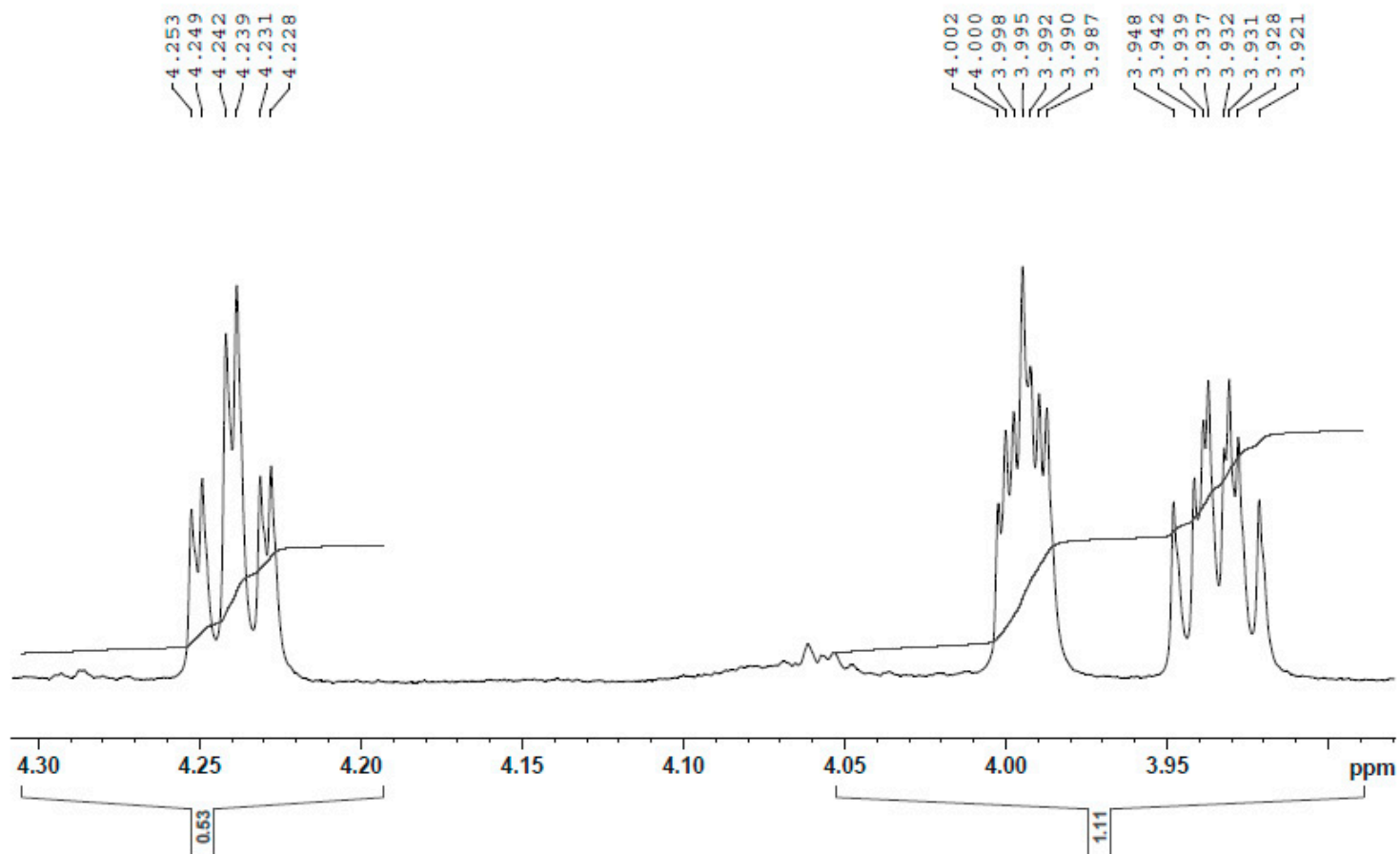


Figure S2c: ^1H NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6

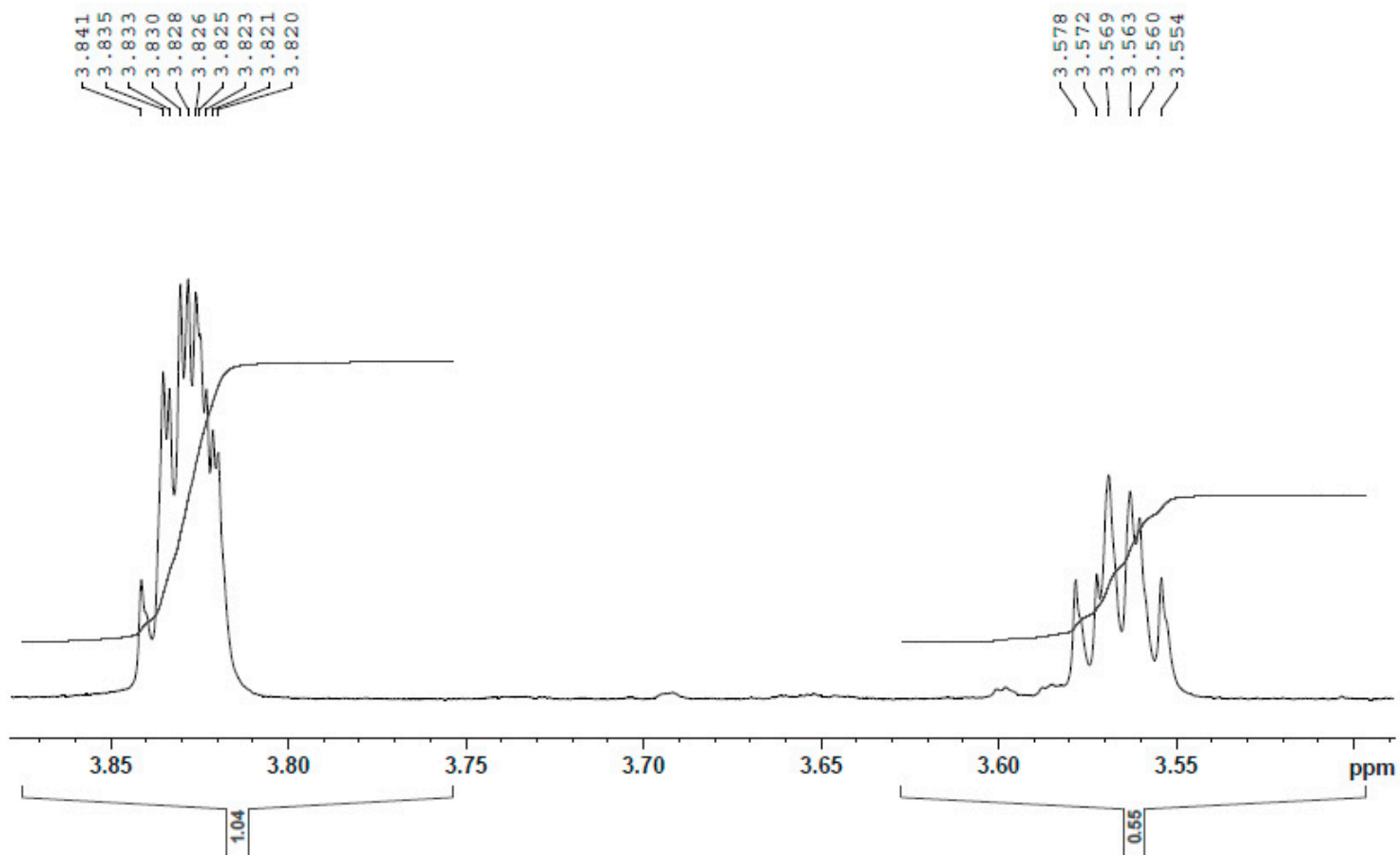


Figure S2d: ^1H NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6

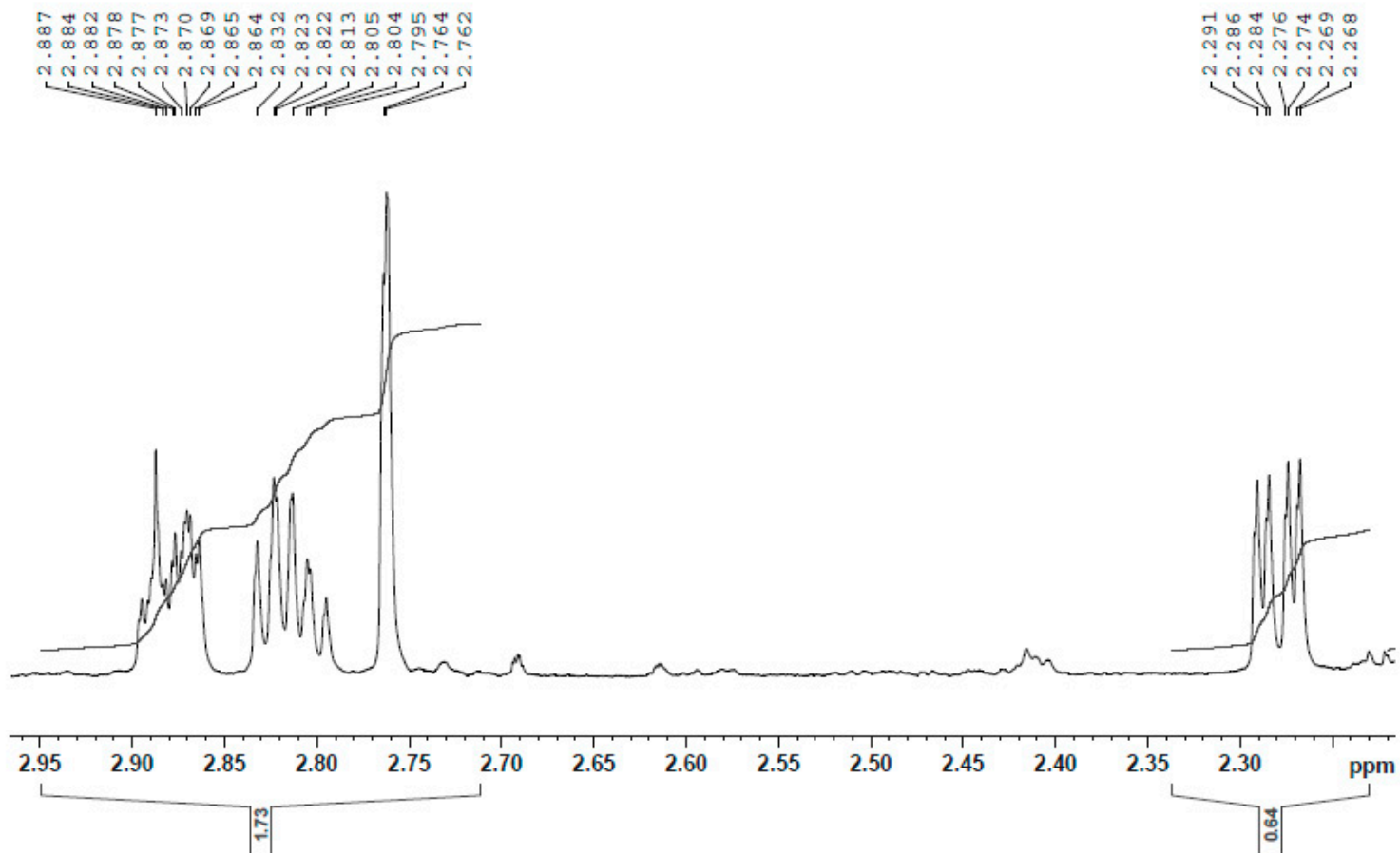
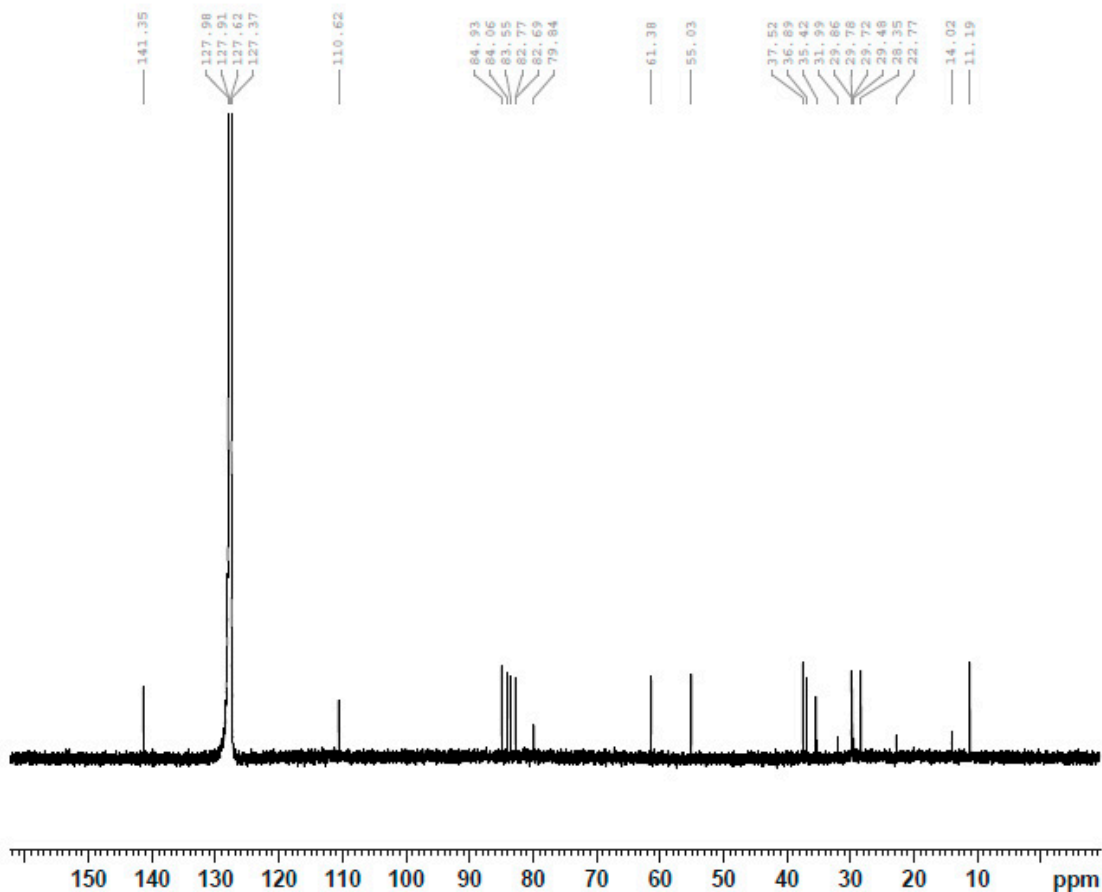


Figure S2e: ¹H NMR of compound 2

Dr. Nahed Bawaked
Sample BO-68-5 C6D6



```
Current Data Parameters
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EXPNO    131
PROCNO    1

F2 - Acquisition Parameters
Date_     20170110
Time      20.56
INSTRUM   spect
PROBHD    5 mm CPQCI 1H-
PULPROG   zgpg30
TD         65536
SOLVENT   C6D6
NS         1500
DS         4
SWH        51020.406 Hz
FIDRES     0.778510 Hz
AQ         0.6422528 sec
RG         186.92
DM         9.800 usec
DE         18.00 usec
TE         298.0 K
D1         2.0000000 sec
d11        0.0100000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      213.7917626 MHz
NUC1       13C
P1         12.00 usec
PLW1       130.0000000 W

===== CHANNEL f2 =====
SFO2      850.1574006 MHz
NUC2       1H
CPCPRG2[2]  waltz16
PCPD2      80.00 usec
PLW2       13.80000019 W
PLW12      0.13800000 W
PLW13      0.08832000 W

F2 - Processing parameters
SI         32768
SF         213.7703875 MHz
WDW        HM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00
```

Figure S2f: ^{13}C NMR of compound 2

Dr. Nahed Bawaked
Sample BO-68-5 C6D6

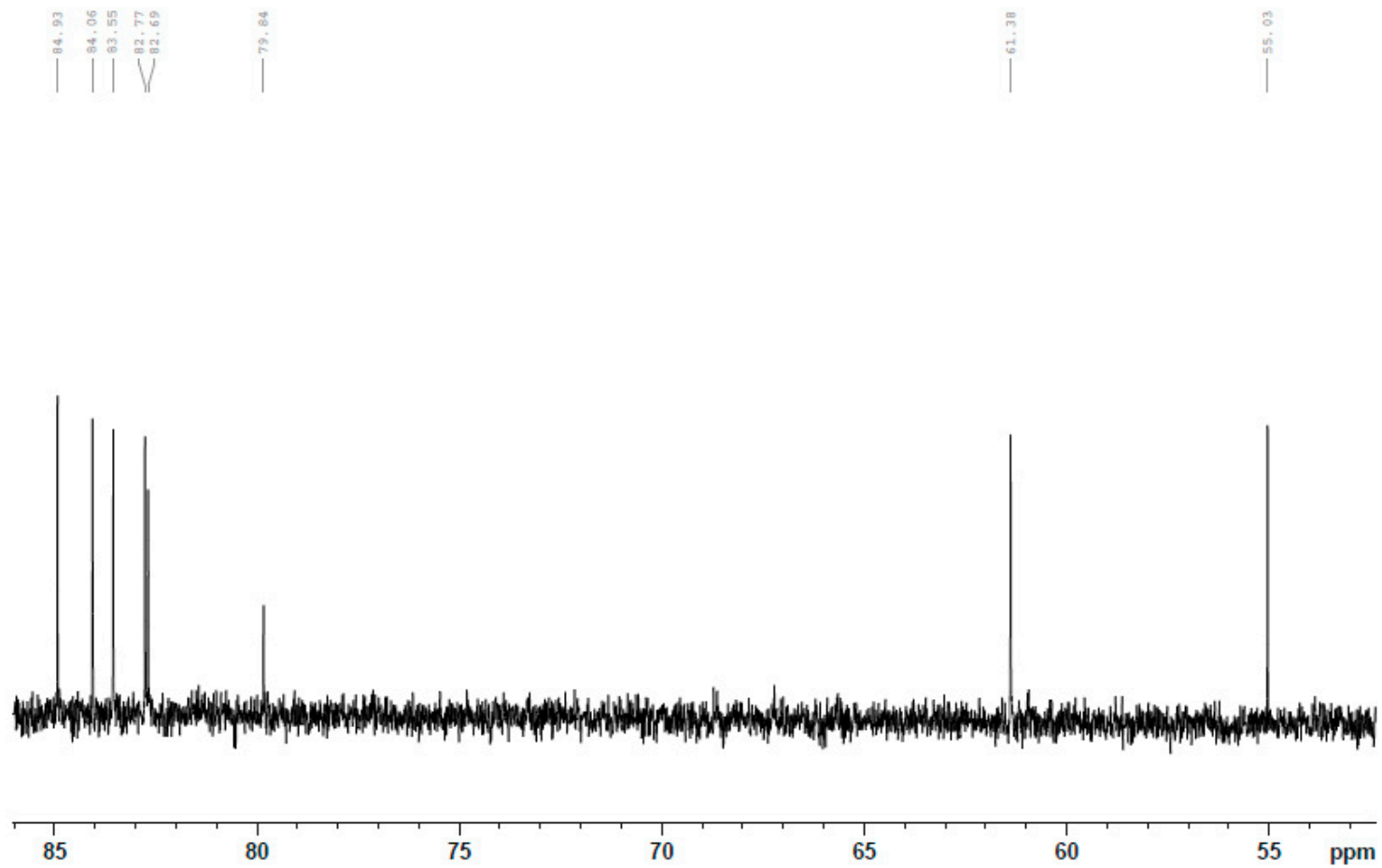


Figure S2g: ^{13}C NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6

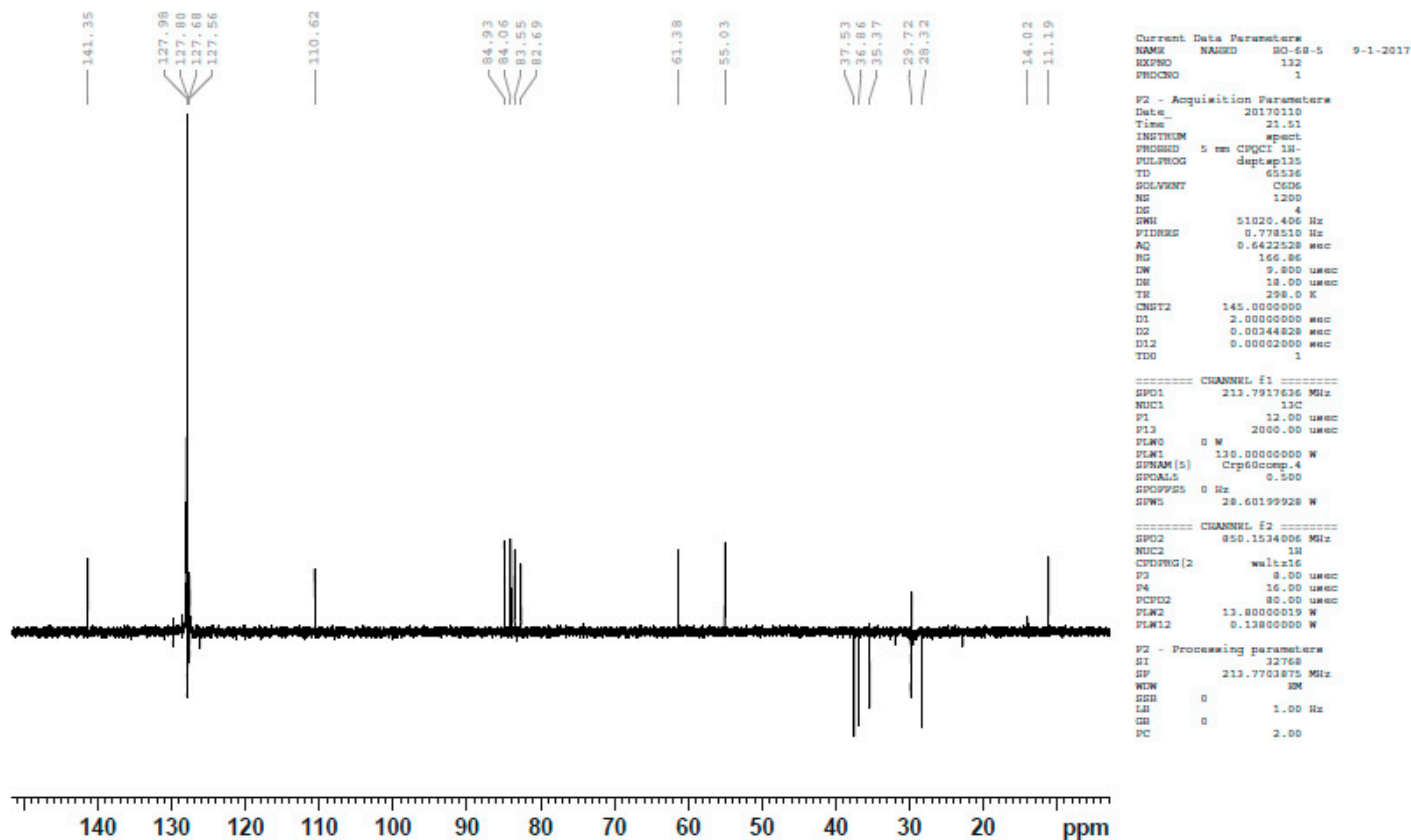
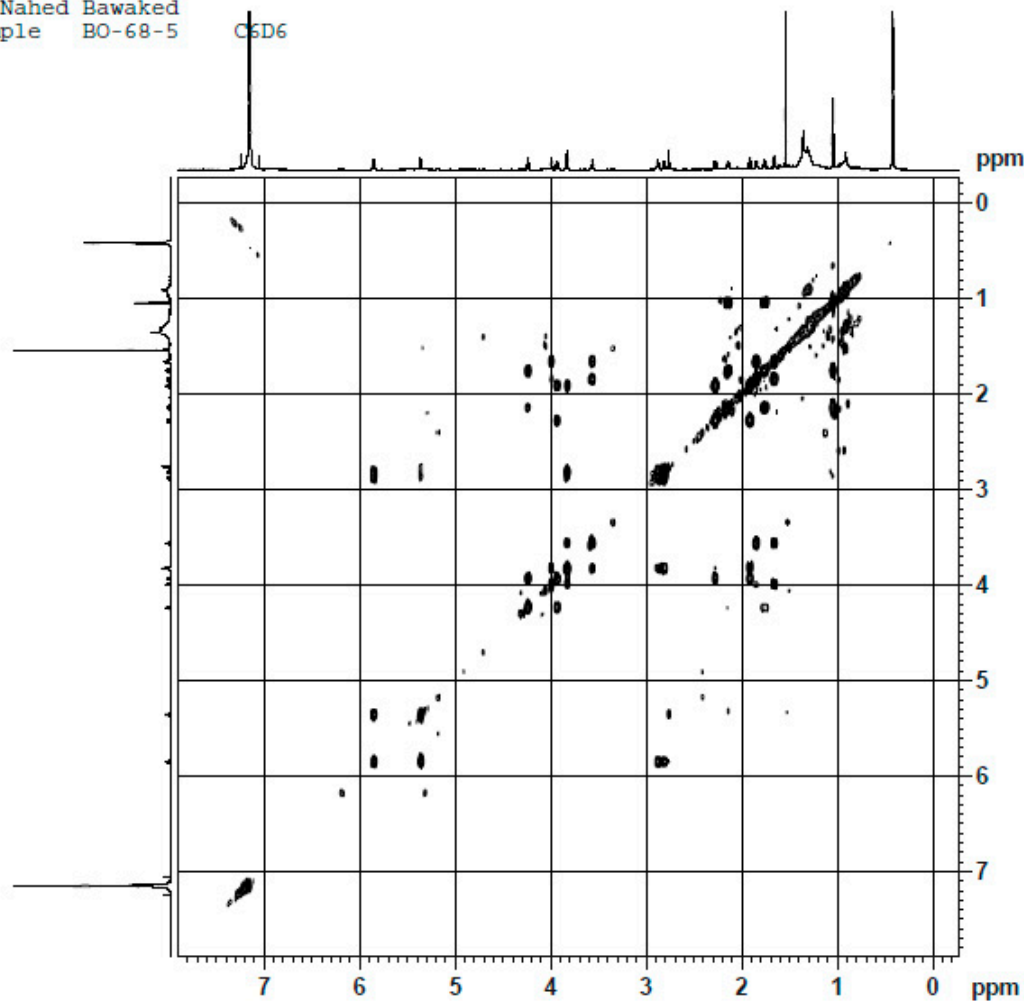


Figure S2h: DEPT NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5



Current Data Parameters
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EXPNO 134
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170110
Time 21.54
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG cwygmrf2
TD 2048
SOLVENT C6D6
NS 32
DS 8
SHE 6544.444 Hz
FIDRES 1.390842 Hz
AQ 0.1474560 sec
RG 186.93
DW 72.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.0000000 sec
D1 1.94265604 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00014400 sec

===== CHANNEL f1 =====
SFO1 850.1532408 MHz
NUC1 1H
P1 8.00 usec
PLW1 15.30000019 W

===== GRADIENT CHANNEL =====
GPRAM[1] SMSQ10.100
GPRAM[2] SMSQ10.100
GPRAM[3] SMSQ10.100
GPE1 16.00 %
GPE2 12.00 %
GPE3 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 850.1532 MHz
FIDRES 54.253471 Hz
SW 8.168 ppm
F2-MODE GP

F2 - Processing parameters
SI 1024
SF 850.1500000 MHz
WDM SINE
SGB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 GP
SF 850.1500000 MHz
WDM SINE
SGB 0
LB 0 Hz
GB 0

Figure S2i: COSY NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6

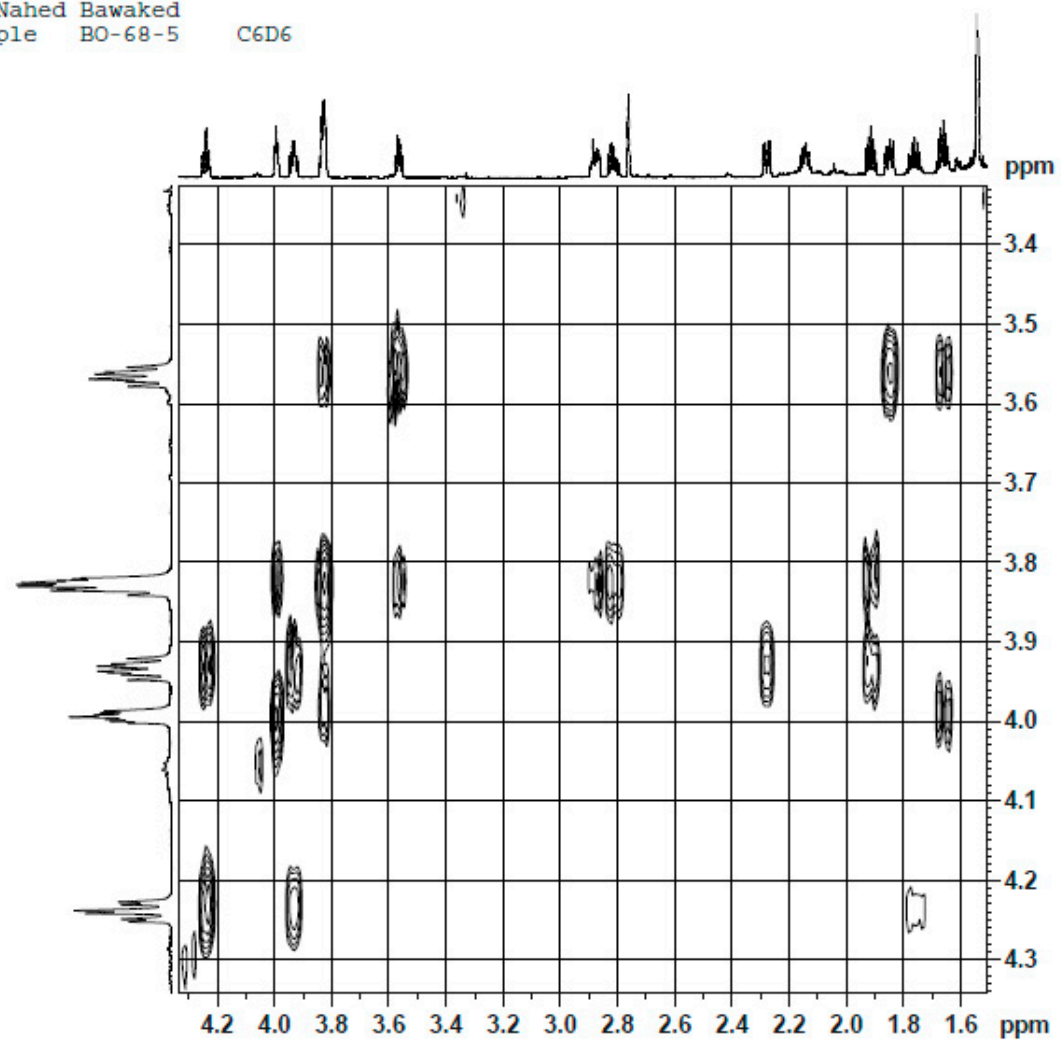


Figure S2j: COSY NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6

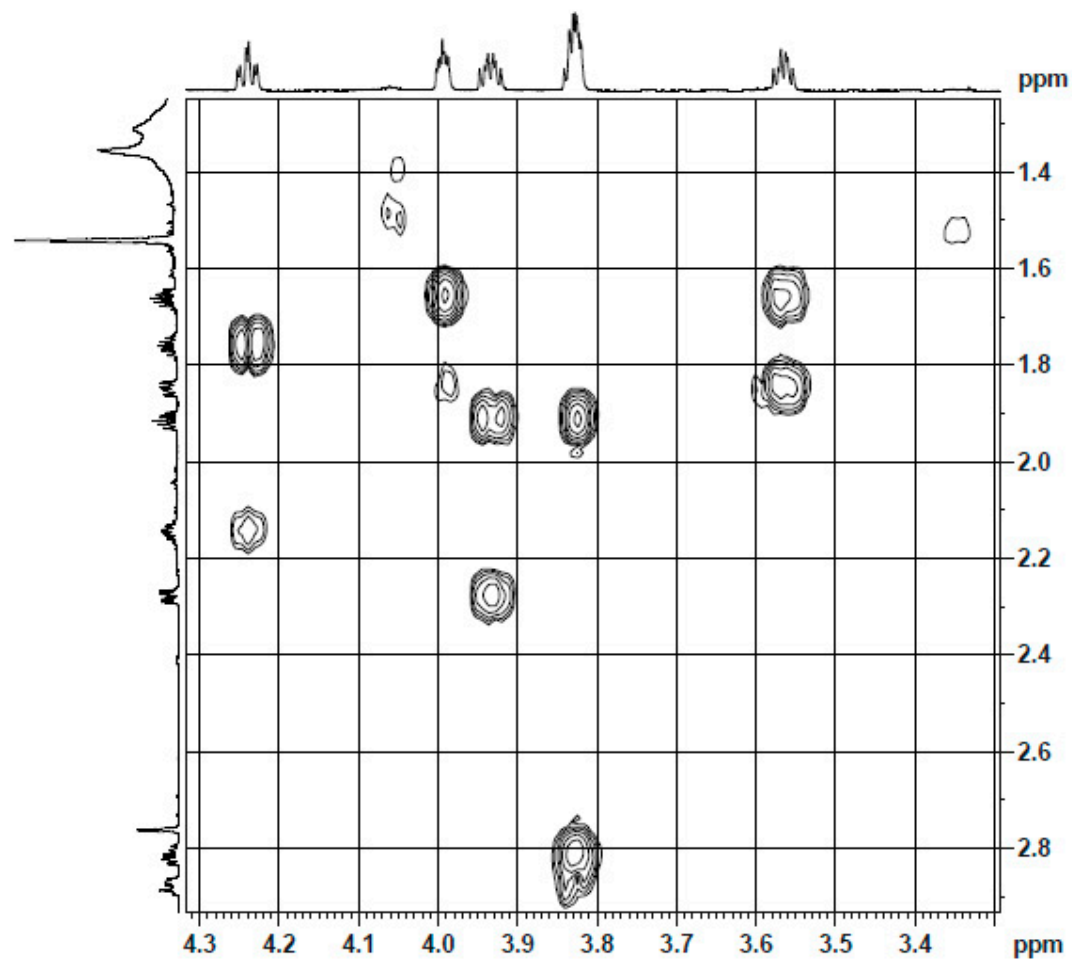
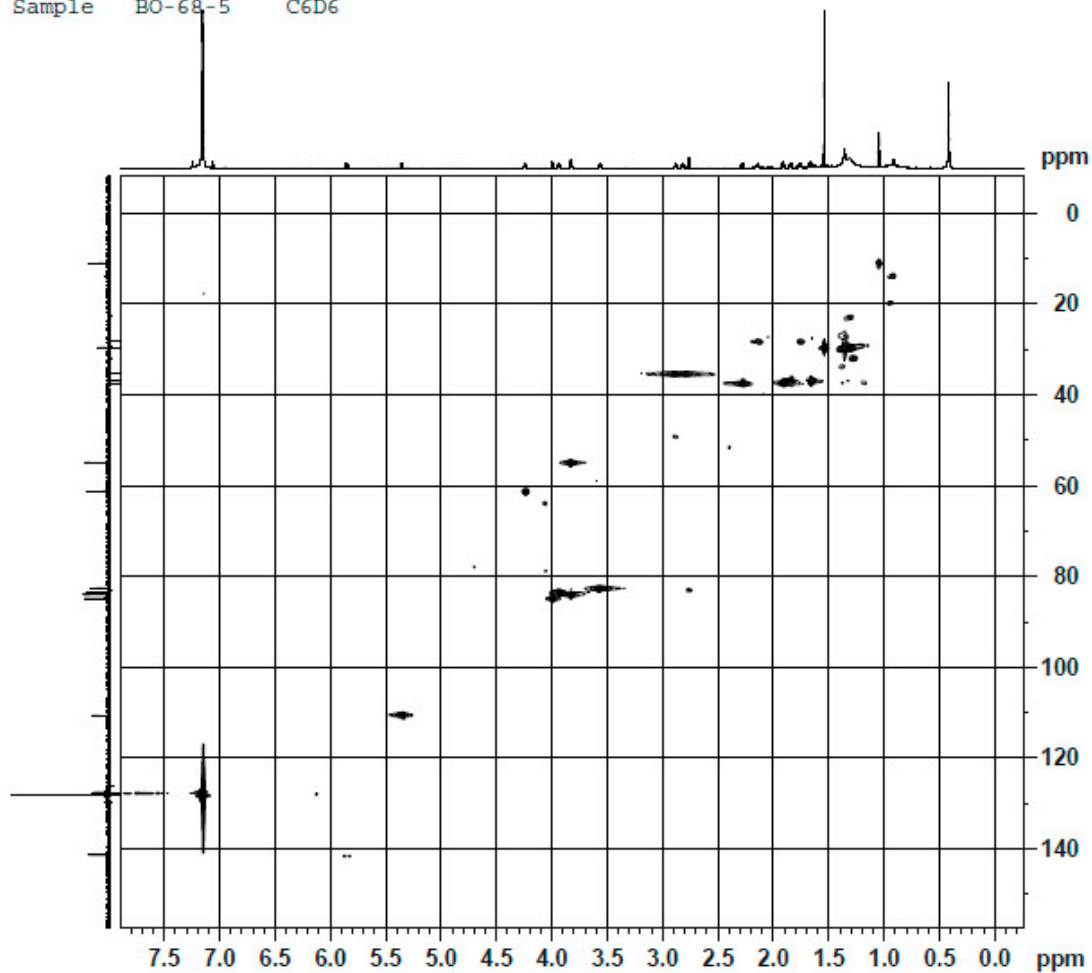


Figure S2k: COSY NMR of compound 2

Dr.Nahed Bawaked
 Sample BO-68-5 C6D6



```

Current Data Parameters
NAME NAHED BO-68-5 9-1-2017
KUNO 119
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170111
Time 3.10
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG hsqcdecoupl
TD 1024
SOLVENT C6D6
NS 40
DS 16
SFO 604.444 Hz
FIDRES 6.781054 Hz
AQ 0.3737280 sec
RG 186.93
DM 72.000 usec
DE 10.00 usec
TE 298.0 K
CHST2 145.000000
D0 0.0000000 sec
D1 1.47269297 sec
D4 0.00172414 sec
D11 0.0000000 sec
D13 0.0000400 sec
D14 0.0000000 sec
D21 0.00340000 sec
IND 0.0001410 sec
SOLPFGS

----- CHANNEL f1 -----
SFO1 500.1312400 MHz
NUC1 13C
P1 8.00 usec
P2 0 usec
P21 16.00 usec
PLW1 15.30000019 W

----- CHANNEL f2 -----
SFO2 213.7863316 MHz
NUC2 13C
CPROG12 garr
P3 12.00 usec
P4 24.00 usec
PCPD2 45.00 usec
PLW2 130.0000000 W
PLW12 9.24440002 W

----- GRADIENT CHANNEL -----
GRANM(1) SMC210.100
GRANM(2) SMC210.100
GPR1 80.00 %
GPR2 20.10 %
PL4 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 213.7863 MHz
FIDRES 138.519601 Hz
DS 160.871 ppm
PULPROG Echo-antiEcho

F2 - Processing parameters
SI 1024
SF 500.1500000 MHz
WDW QDINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC 2 echo-antiEcho
SF 213.7703870 MHz
WDW QDINE
SSB 2
LB 0 Hz
GB 0
  
```

Figure S21: HSQC NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5

C6D6

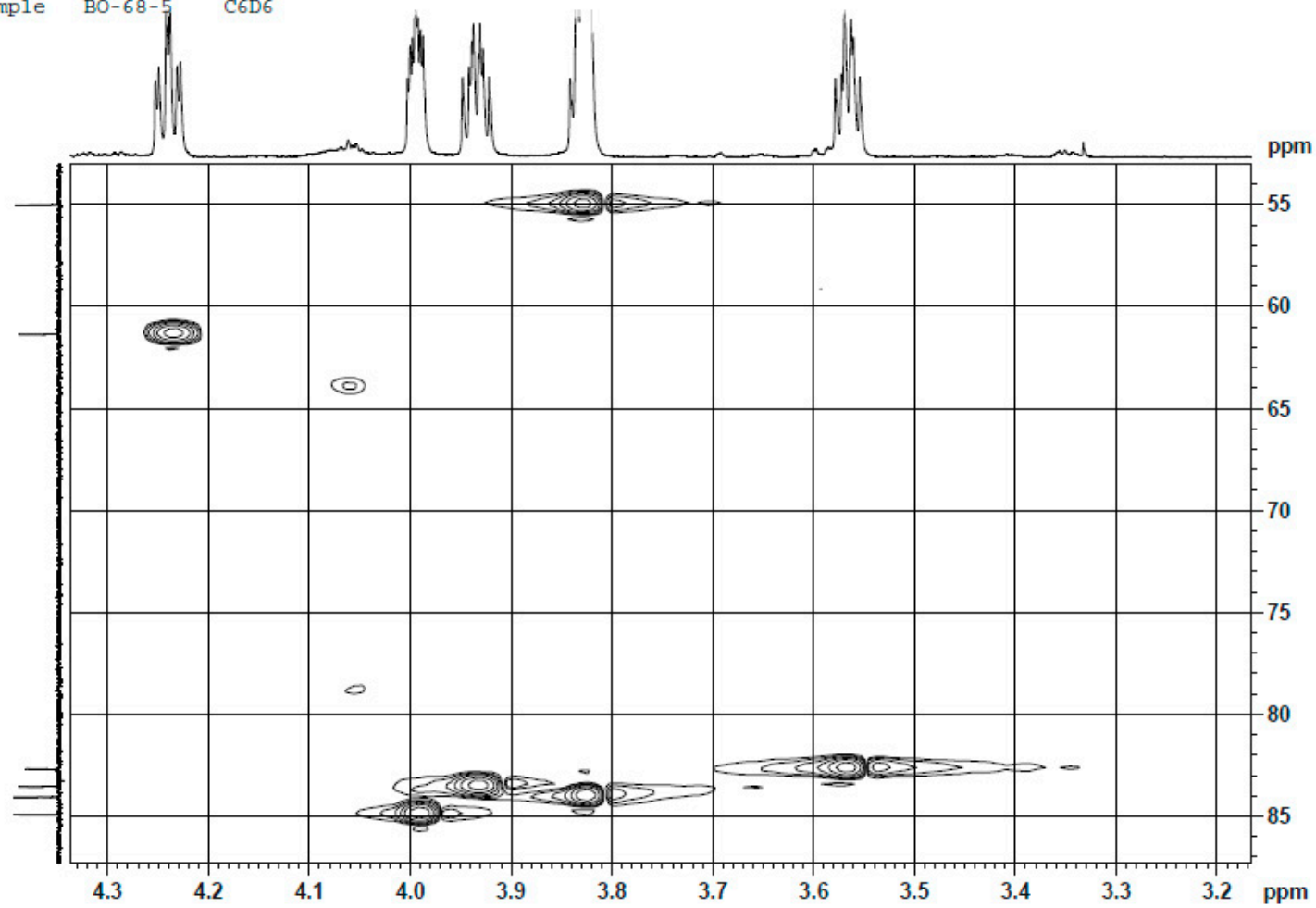
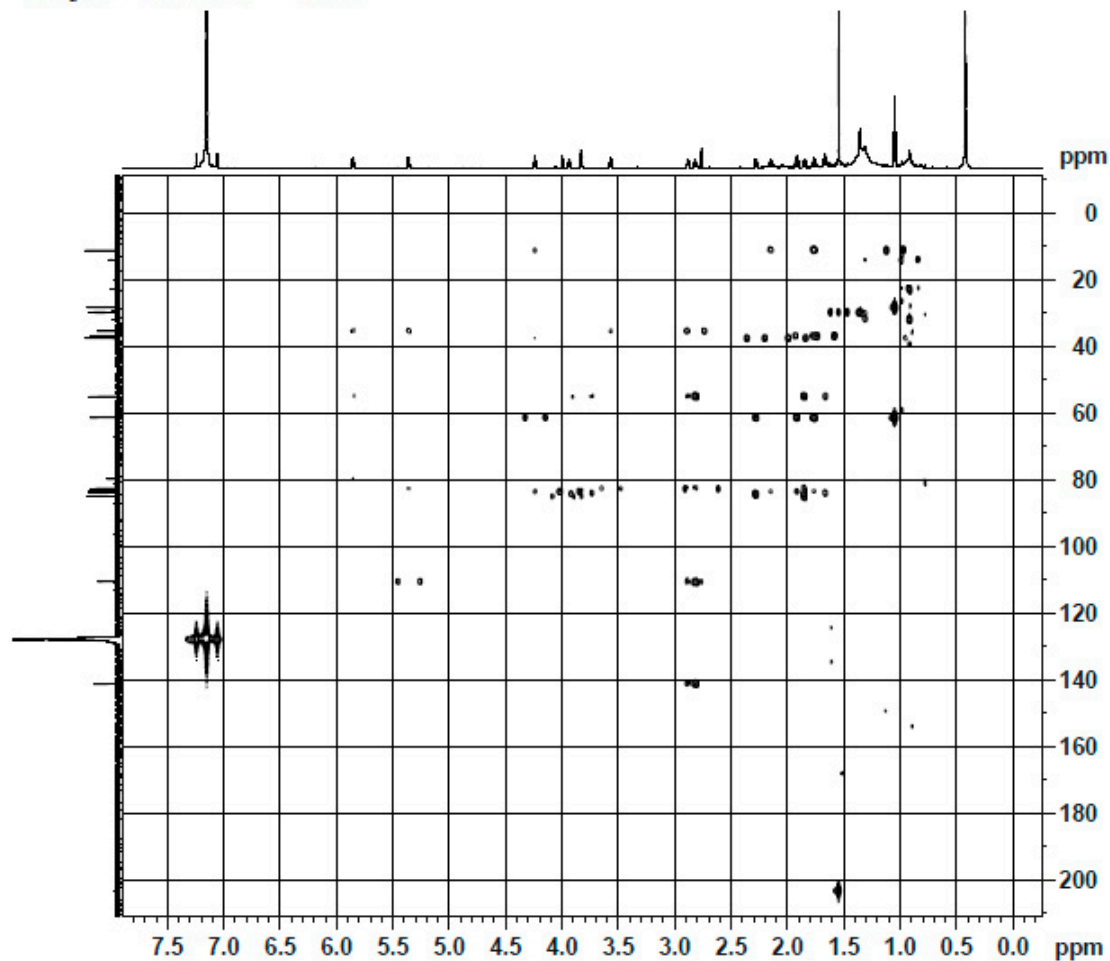


Figure S2m: HSQC NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5 C6D6



```
Current Data Parameters
NAME NAHED BO-68-5 9-1-2017
EXPNO 140
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170111
Time 7.40
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG hbzgpgdgdg
TD 65536
SOLVENT CDCl3
NS 75
DS 16
SWH 6944.444 Hz
FIDRES 1.495421 Hz
AQ 0.2949120 sec
RG 386.93
DQ 72.000 usec
DE 10.00 usec
TE 298.0 K
CNS113 8.0000000
DD 0.0000300 usec
DI 1.3841305 usec
DK 0.0425000 usec
DL1 0.0002000 usec
IR0 0.0001050 usec

----- CHANNEL f1 -----
SFO1 850.1532468 MHz
NUC1 1H
P1 8.00 usec
P2 16.00 usec
PLW1 15.30000019 W

----- CHANNEL f2 -----
SFO2 213.7917305 MHz
NUC2 13C
P3 12.00 usec
PLW2 130.00000000 W

----- GRADIENT CHANNEL -----
GPRAM[1] SMOQ10.100
GPRAM[2] SMOQ10.100
GPRAM[3] SMOQ10.100
GPE1 50.00 k
GPE2 30.00 k
GPE3 40.10 k
P16 1000.00 usec

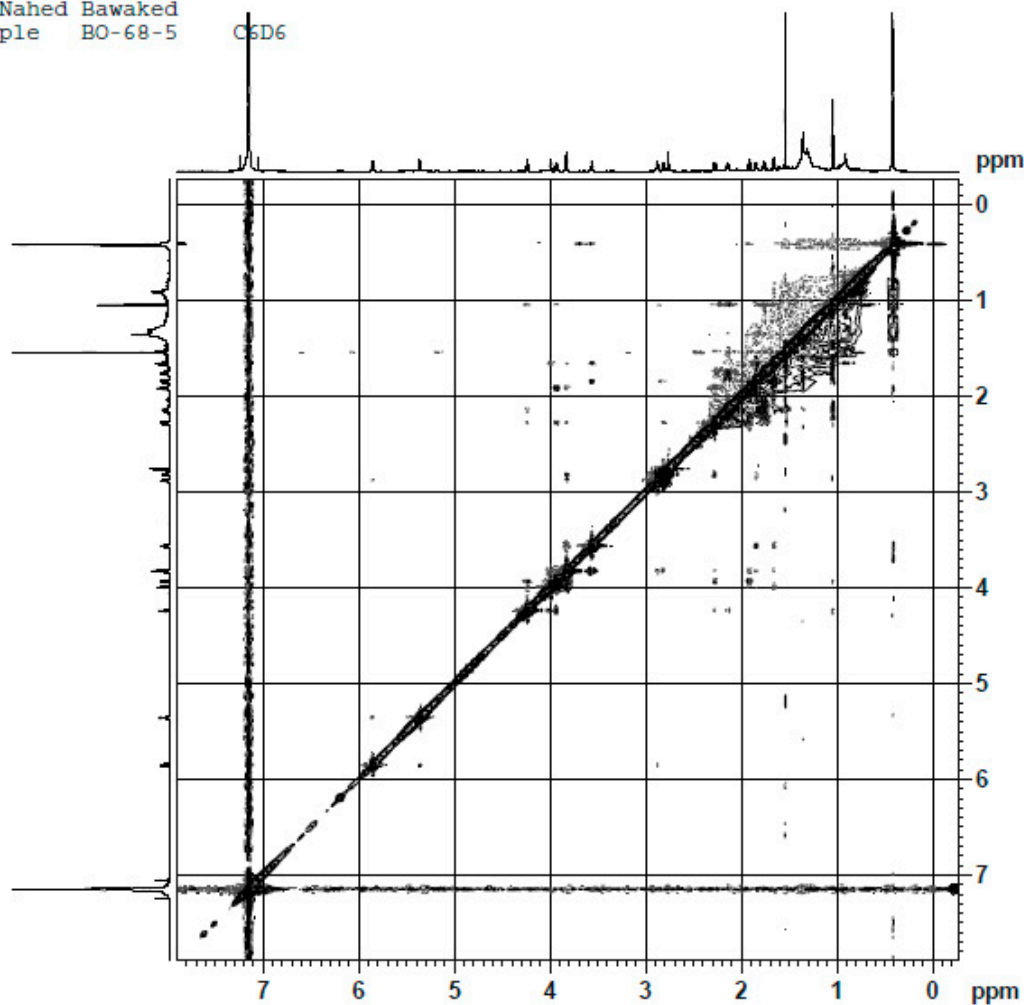
F1 - Acquisition parameters
TD 128
SFO1 213.7917 MHz
FIDRES 372.023804 Hz
DQ 72.000 usec
P16 1000.00 usec

F2 - Processing parameters
SI 1024
SF 850.1500000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 213.7703875 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
```

Figure S2n: HMBC NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5



```
Current Data Parameters
NAME      NAHED   BO-68-5   9-1-2017
EXPNO    136
PROCNO   1

F2 - Acquisition Parameters
Date_    20170111
Time     0.19
INSTRUM  spect
PROBHD   5 mm CPQCI 1H-
PULPROG  noesypphpp
TD       2048
SOLVENT  CD66
NS       16
DS       32
SWH      6944.444 Hz
FIDRES   3.390842 Hz
AQ       0.1474560 sec
RG       35.7
DM       72.000 usec
DE       15.00 usec
TE       299.0 K
DQ       0.00006181 sec
D1       1.97296596 sec
D8       0.30000001 sec
D11      0.03000000 sec
D12      0.00002000 sec
D16      0.00020000 sec
IND      0.00014400 sec

----- CHANNEL f1 -----
SFO1    850.1532408 MHz
NUC1     1H
P1       8.00 usec
P2      15.00 usec
P17     2500.00 usec
PLA1    15.30000019 W
PLA10   1.700000005 W

----- GRADIENT CHANNEL -----
GPRAM[1]  GRG10.100
GP11     40.00 %
P16     1000.00 usec

F1 - Acquisition parameters
TD       256
SFO1    850.1532 MHz
FIDRES   27.126736 Hz
SW       8.168 ppm
P2PROG   States-TPPI

F2 - Processing parameters
SI       1024
SF      850.1500000 MHz
WDW      Q2
SSB      2
LB       0 Hz
GB       0
PC       1.00

F1 - Processing parameters
SI       1024
MC2     States-TPPI
SF      850.1500000 MHz
WDW      Q2
SSB      2
LB       0 Hz
GB       0
```

Figure S2o: NOESY NMR of compound 2

Dr.Nahed Bawaked
Sample BO-68-5

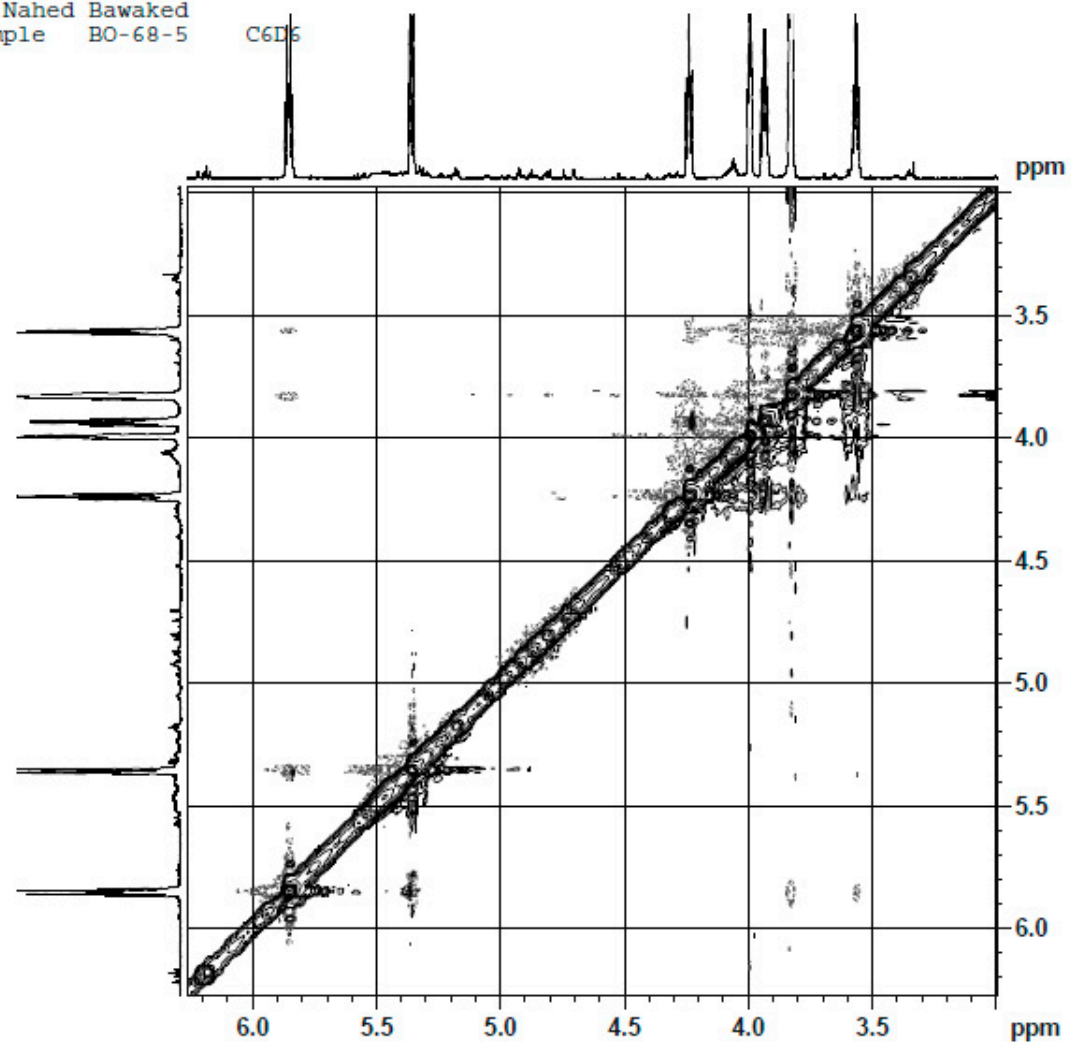


Figure S2p: NOESY NMR of compound 2