

SUPPLEMENTARY MATERIAL

Rare acetogenins with anti-inflammatory effect from the red alga *Laurencia obtusa*

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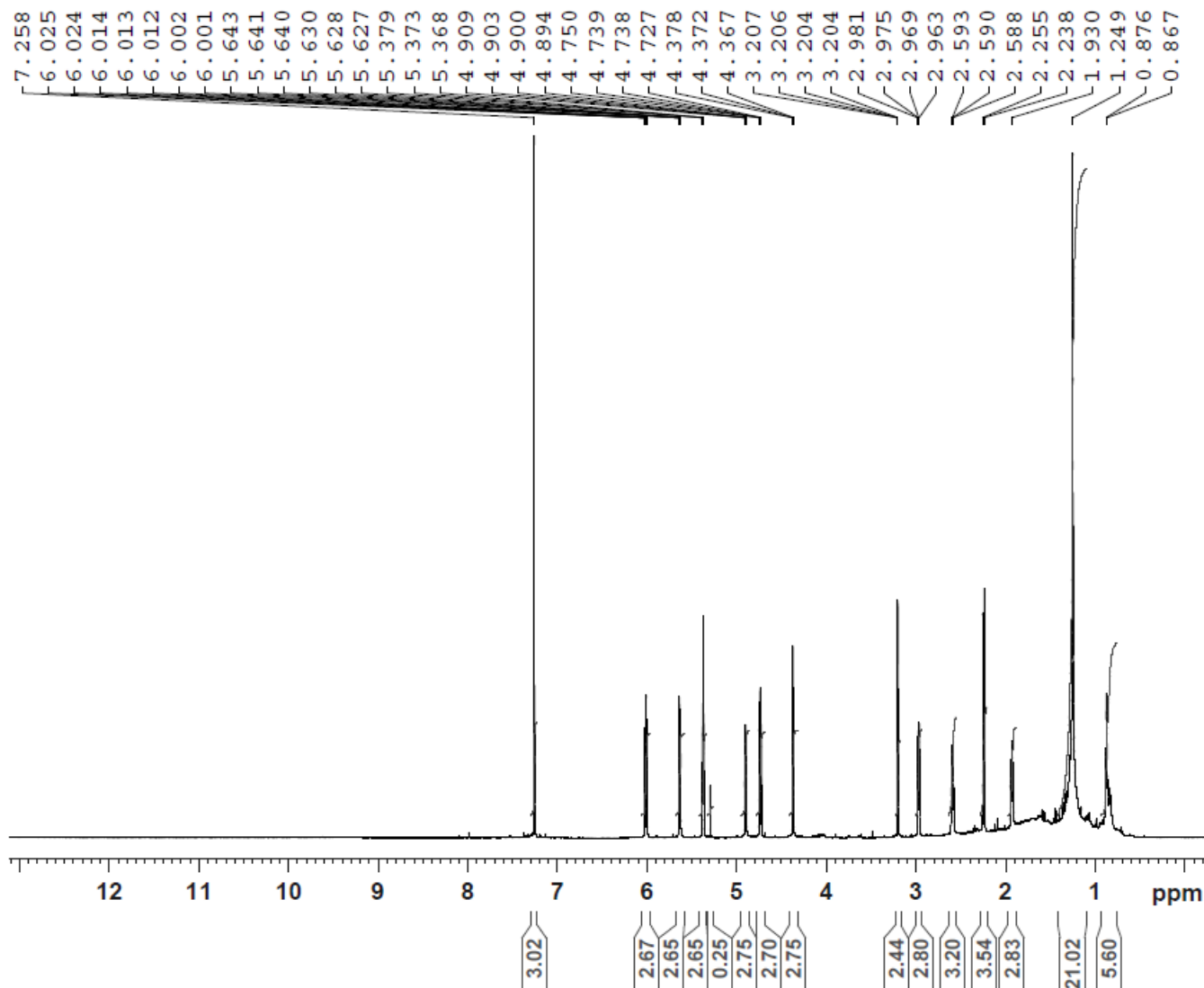
Abstract

Three new rare C12 acetogenins en-yne derivatives (**1-3**) were isolated from the organic extract obtained from the red alga *Laurencia obtusa*, collected from the Red Sea. The chemical structures of the isolated compounds were established by spectroscopical data analyses. Potent anti-inflammatory effect of the isolated metabolites was evidenced by inhibition of releasing the inflammatory mediators (e.g. TNF- α , IL-1 β and IL-6) by employing Human Peripheral Blood Mononuclear Cells (PBMC).

Keywords: Anti-inflammatory; *Laurencia obtusa*; Acetogenins; Spectroscopy; Red Sea

Dr. Walied

Sample : NL-235-1 CDCL3



Current Data Parameters
NAME WALIED NL-235-1 21-02-2017
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170221
Time_ 10.42
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCL3
NS 32
DS 2
SWH 17006.803 Hz
FIDRES 0.259503 Hz
AQ 1.9267584 sec
RG 9.04
DW 29.400 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 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 EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

Figure S1a: ¹H NMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL₃

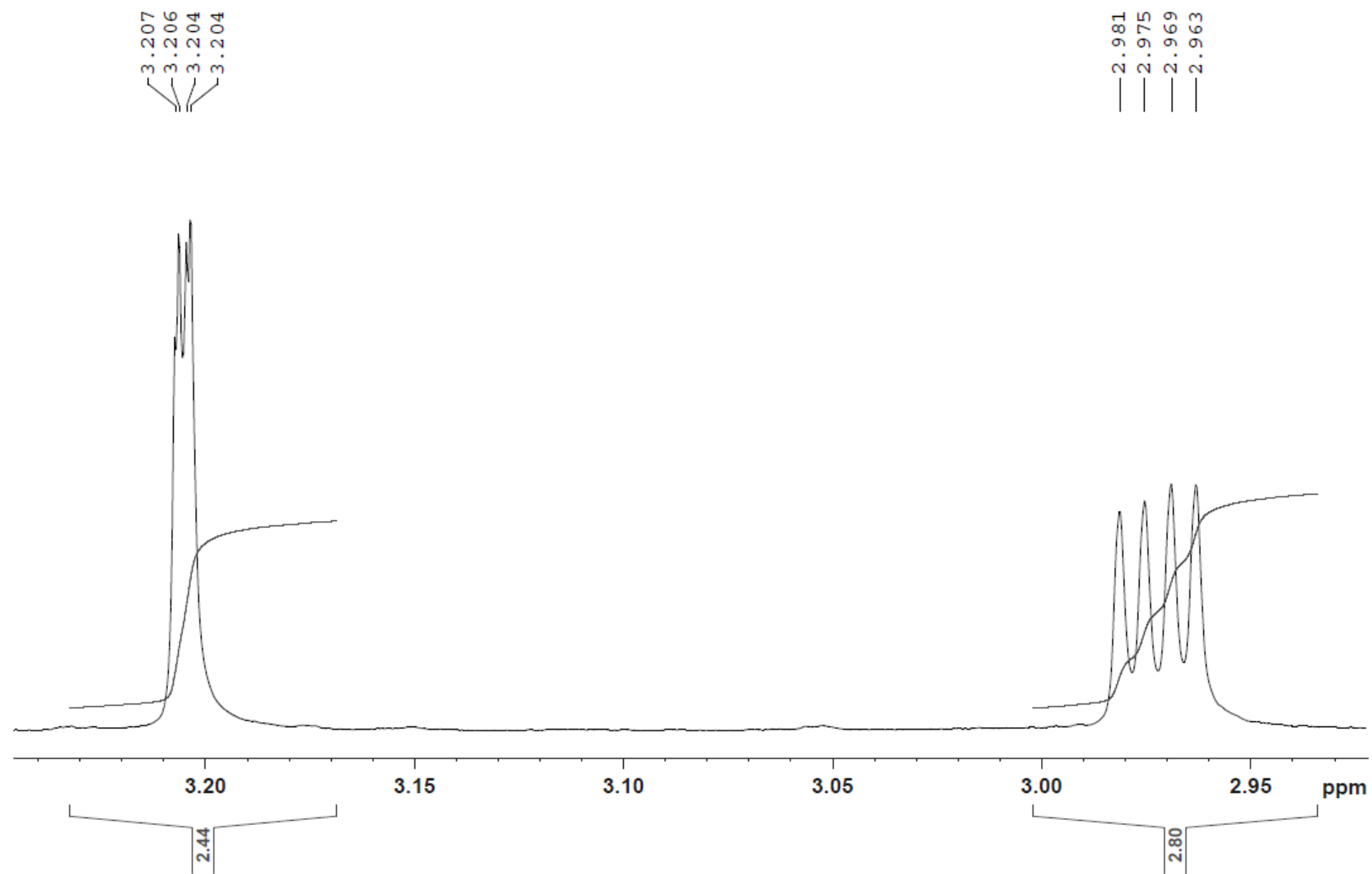


Figure S1b: ¹H NMR of compound 1

Dr. Walied

Sample : NL-235-1 CDCL₃

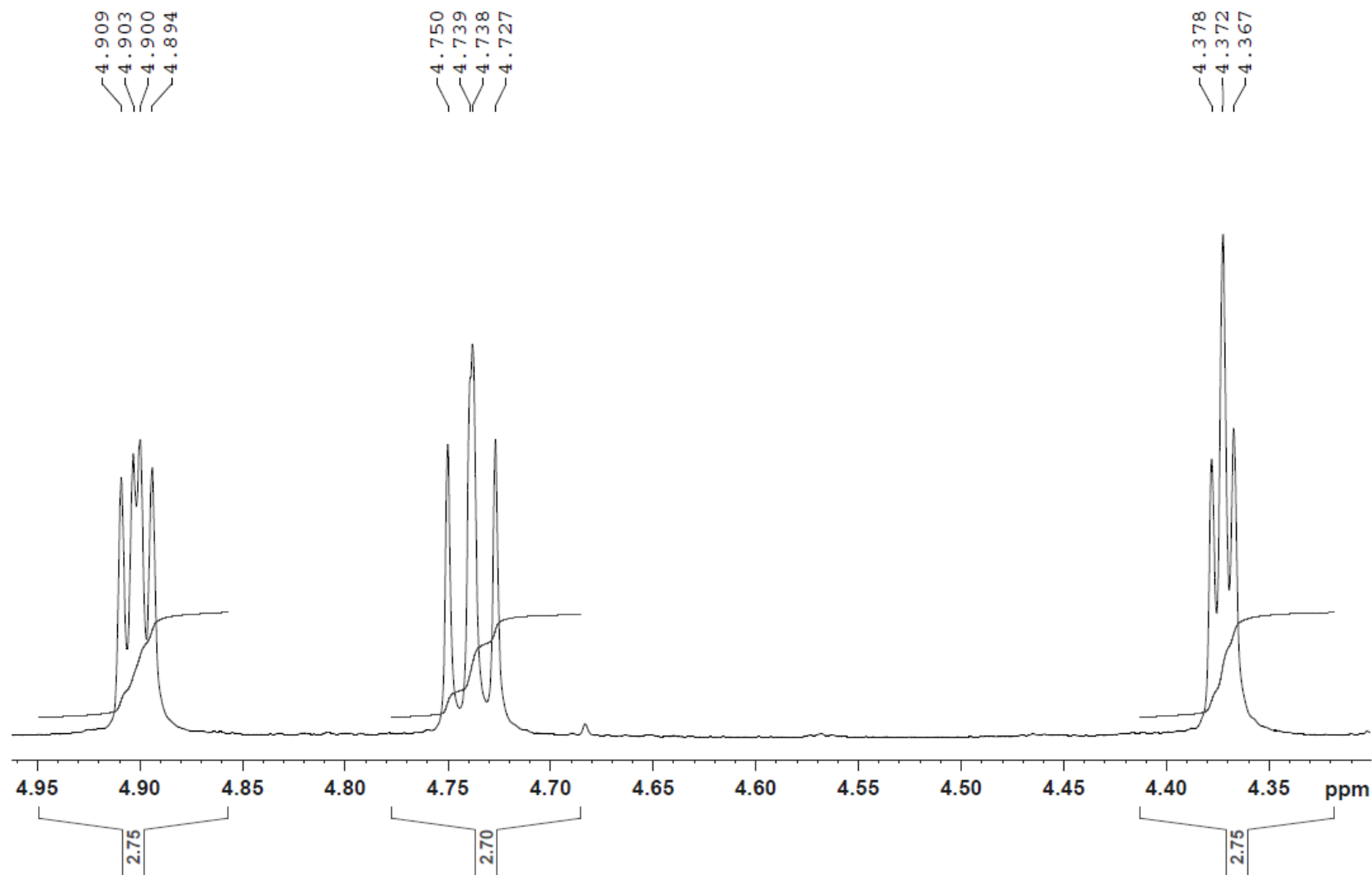


Figure S1c: ¹H NMR of compound 1

Dr. Walled
Sample : NL-235-1 CDCL3

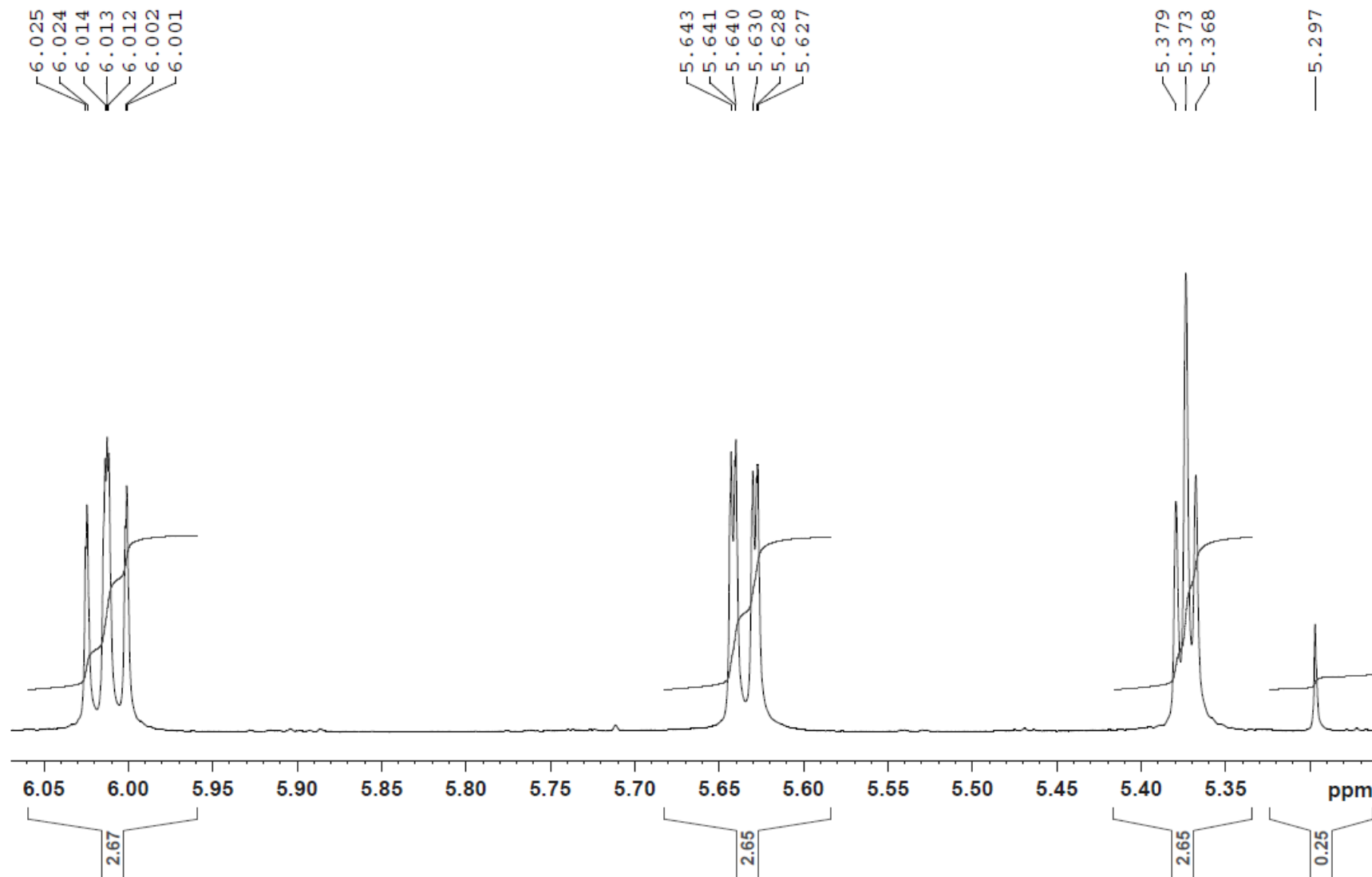


Figure S1d: ^1H NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

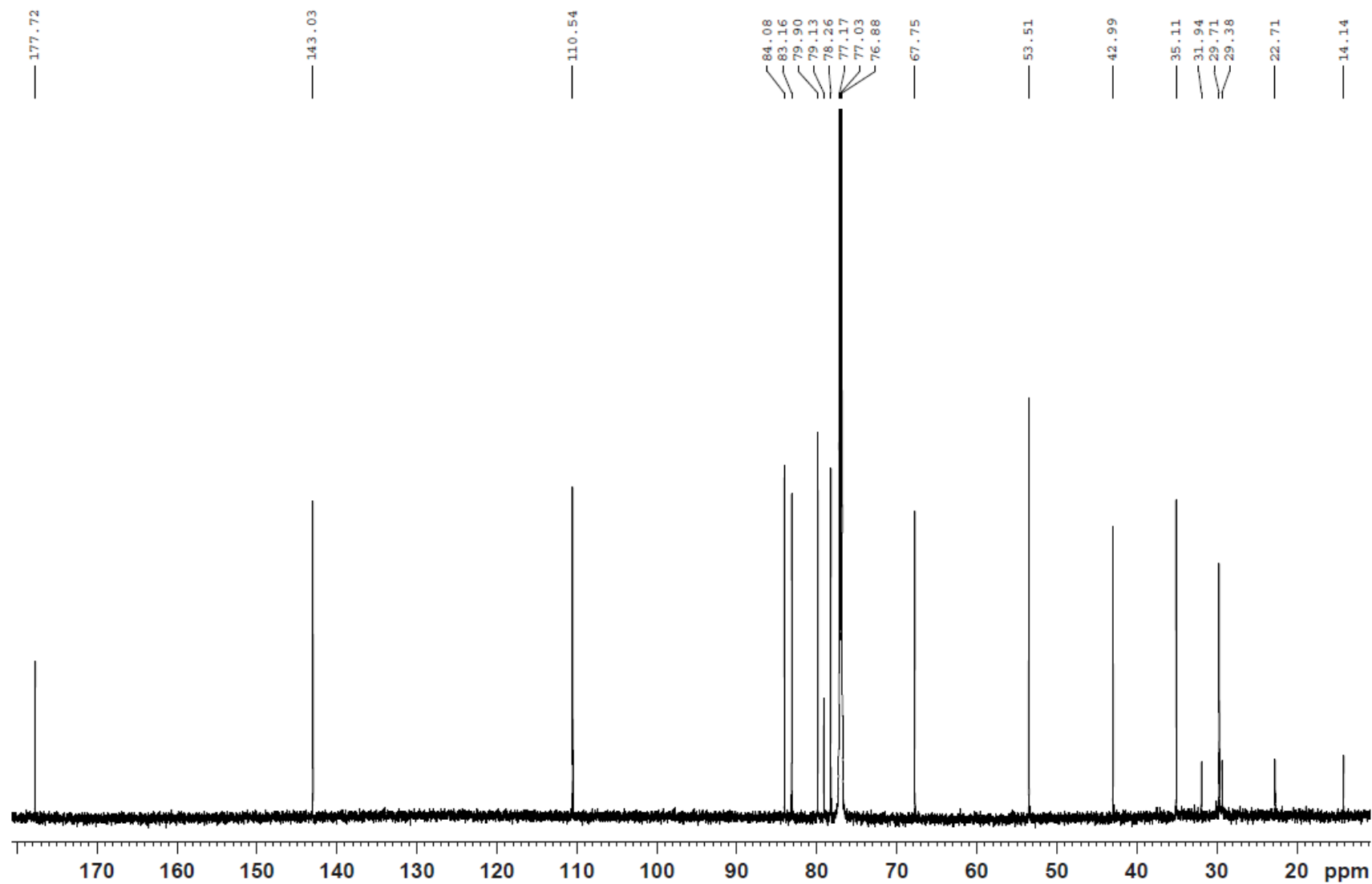


Figure S1e: ¹³CNMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

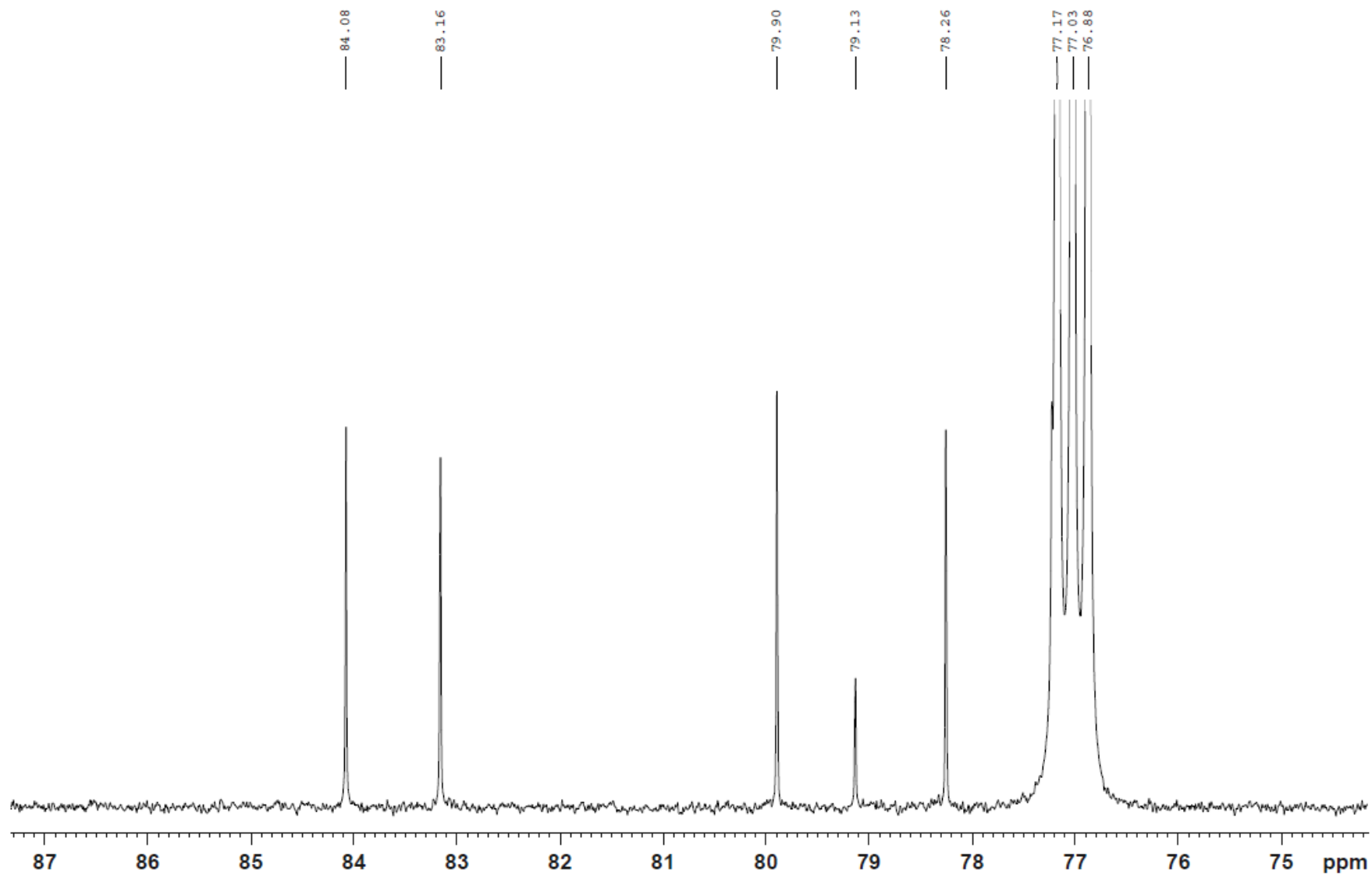


Figure S1f: ¹³CNMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL3



Current Data Parameters
NAME WALIED NL-2351 23-02-2017
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date 20170223
Time 23.22
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG hsqcaddgpp
TD 1024
SOLVENT CDCL3
NS 32
DS 16
SWH 7812.500 Hz
FIDRES 7.629395 Hz
AQ 0.0655360 sec
RG 186.93
DW 64.000 usec
DE 10.00 usec
TE 298.0 K
CNST2 145.0000000
D0 0.0000380 sec
D1 1.48088503 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000480 sec
D16 0.00020000 sec
D21 0.00345000 sec
INO 0.00001410 sec
ZDOPUS

----- CHANNEL f1 -----
SFO1 850.1530380 MHz
NUC1 1H
P1 8.00 usec
P2 16.00 usec
P28 0 usec
PLM1 15.30000019 W

----- CHANNEL f2 -----
SFO2 213.7863316 MHz
NUC2 13C
CPDPRG[2] garp
P3 12.00 usec
P4 24.00 usec
PCPD2 45.00 usec
PLM2 130.00000000 W
PLM12 9.24440002 W

----- GRADIENT CHANNEL -----
GPMAM[1] SMSQ10.100
GPMAM[2] SMSQ10.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 213.7863 MHz
FIDRES 138.919501 Hz
SW 165.871 ppm
PnMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 850.1500200 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
WC2 echo-antiecho
SF 213.7703875 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

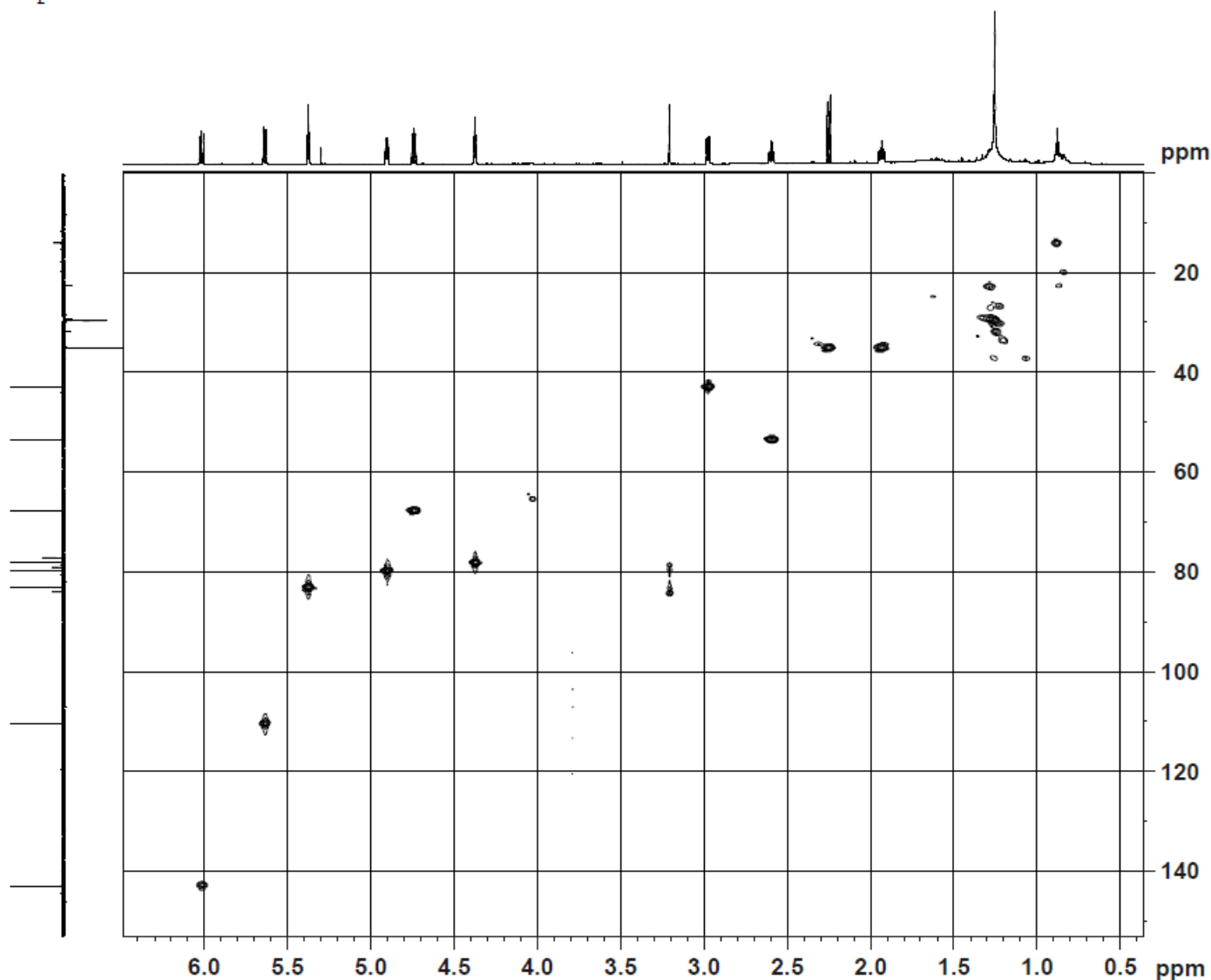


Figure S1g: HSQC NMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL3

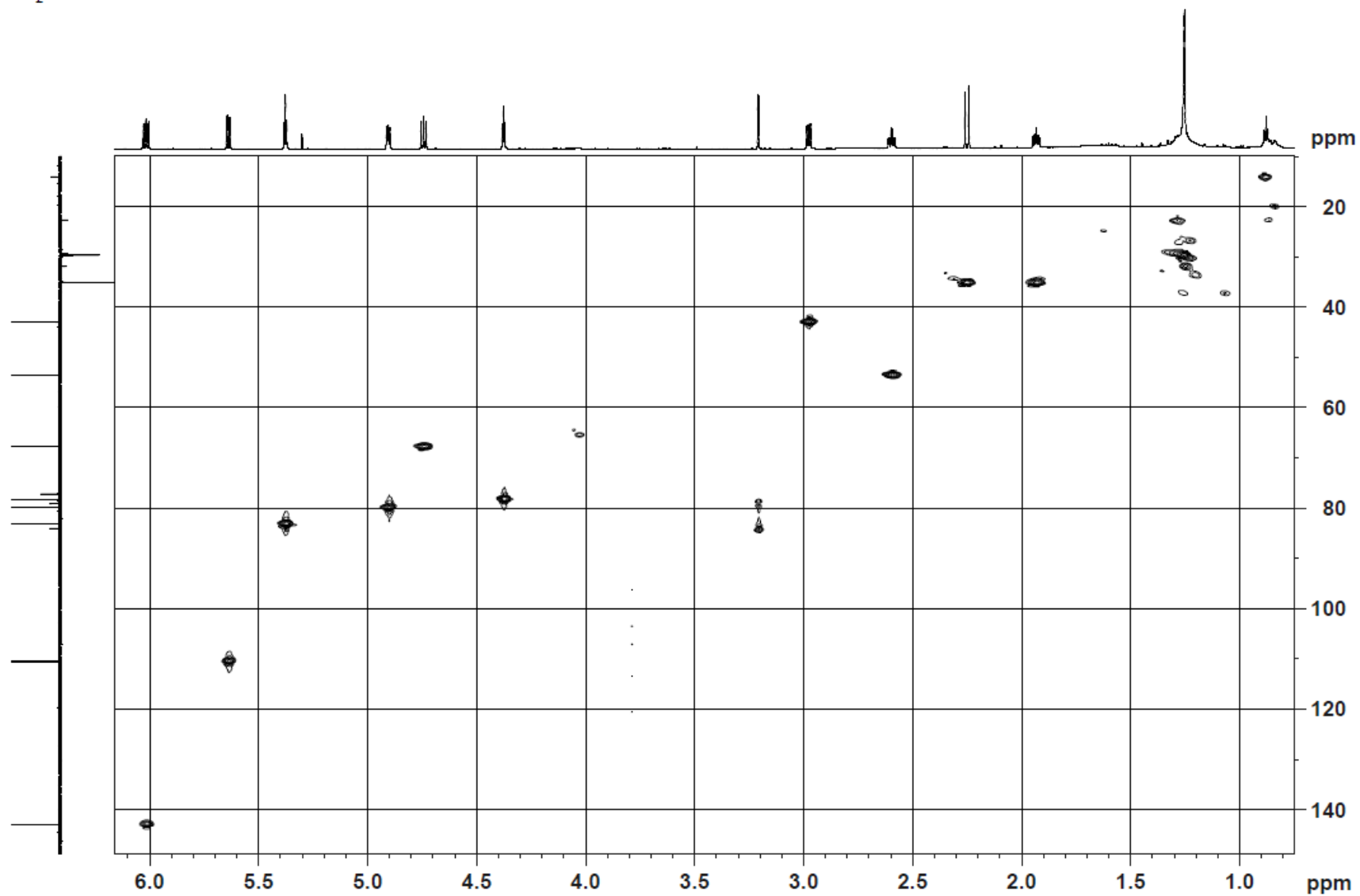
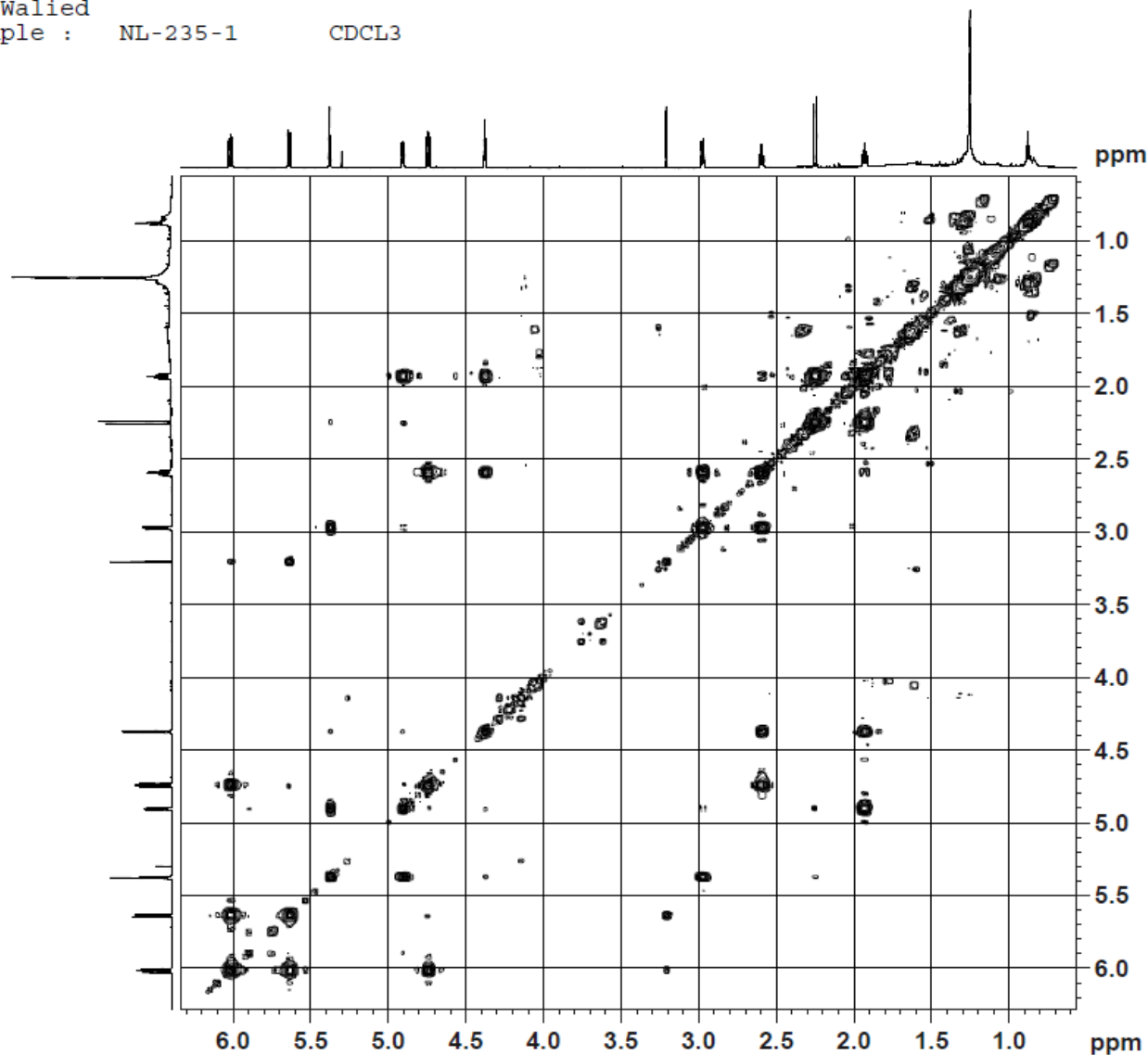


Figure S1h: HSQC NMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL3



Current Data Parameters
NAME WALIED NL-2351 23-02-2017
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170223
Time 16.28
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG cosygpmfzf
TD 2048
SOLVENT CDCL3
NS 16
DS 8
SWH 7812.500 Hz
FIDRES 3.814697 Hz
AQ 0.1310720 sec
RG 186.93
DW 64.000 usec
DE 10.00 usec
TE 298.0 K
DO 0.00000300 sec
D1 1.95904005 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00012800 sec

----- CHANNEL f1 -----
SFO1 850.1530380 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
GPZ1 16.00 %
GPZ2 12.00 %
GPZ3 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 850.153 MHz
FIDRES 61.035156 Hz
SW 9.190 ppm
PnMODE QF

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

F1 - Processing parameters
SI 1024
MC2 QF
SF 850.1500200 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

Figure S1i: COSY NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

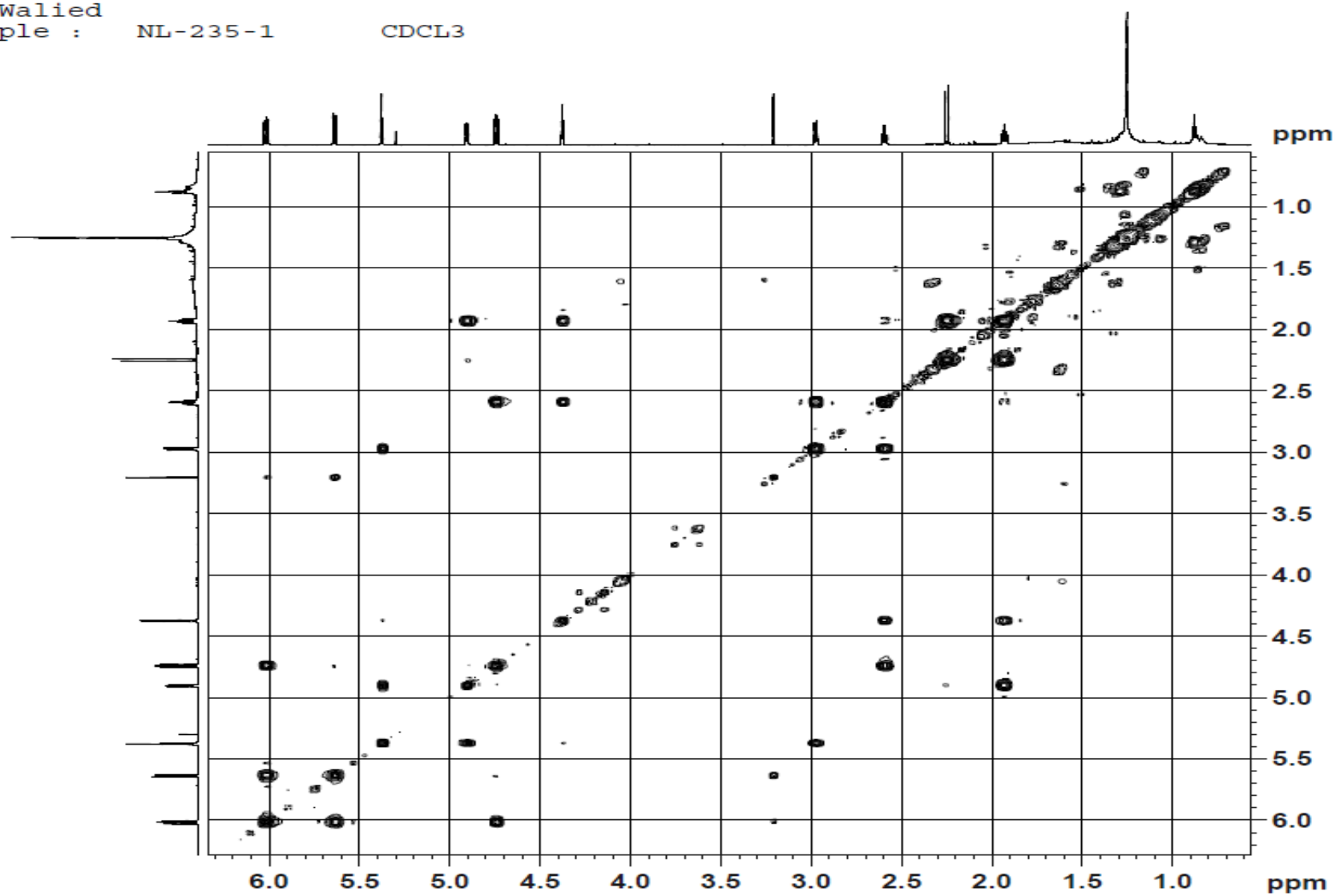


Figure S1j: COSY NMR of compound 1

Dr.Walied

Sample : NL-235-1 CDCL3



Current Data Parameters
NAME WALIED NL-2351 23-02-2017
EXPTNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170224
Time 2.59
INSTRUM spect
PROBHD 5 mm CDQCI 1H-
PULPROG hmbcgpndqf
TD 4096
SOLVENT CDCL3
NS 64
DS 16
SWH 7812.500 Hz
FIDRES 1.907349 Hz
AQ 0.2621440 sec
RG 186.93
DW 64.000 usec
DE 10.00 usec
TE 298.0 K
CNST13 8.0000000
D0 0.00000300 sec
D1 1.41889906 sec
D6 0.06250000 sec
D16 0.00020000 sec
IN0 0.00001050 sec

----- CHANNEL f1 -----
SFO1 850.1530380 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 -----
GPNAM[1] SMO10.100
GPNAM[2] SMO10.100
GPNAM[3] SMO10.100
GPE1 50.00 %
GPE2 30.00 %
GPE3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 213.7917 MHz
FIDRES 372.023804 Hz
SW 222.736 ppm
F2MODE QF

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

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

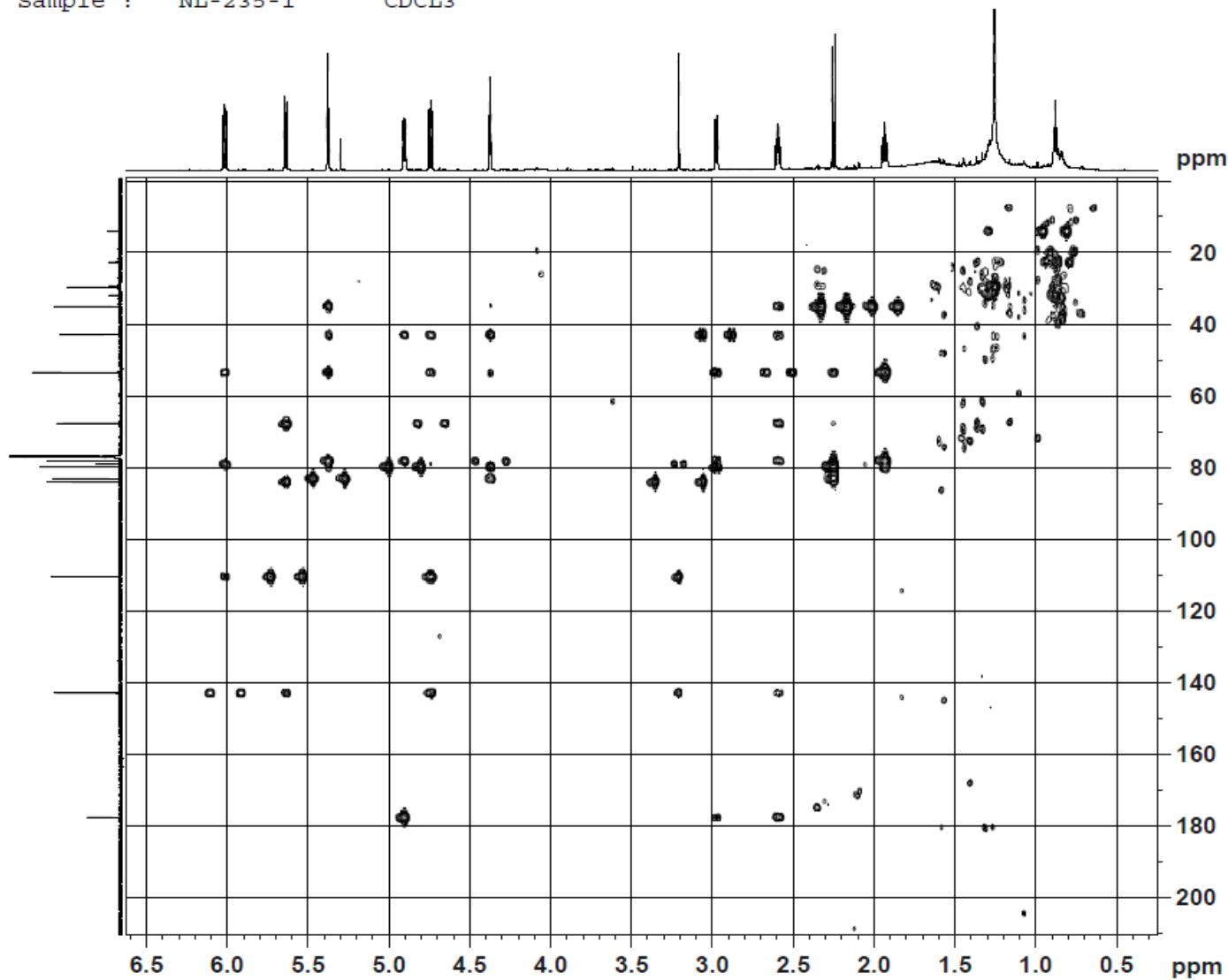


Figure S1k: HMBC NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

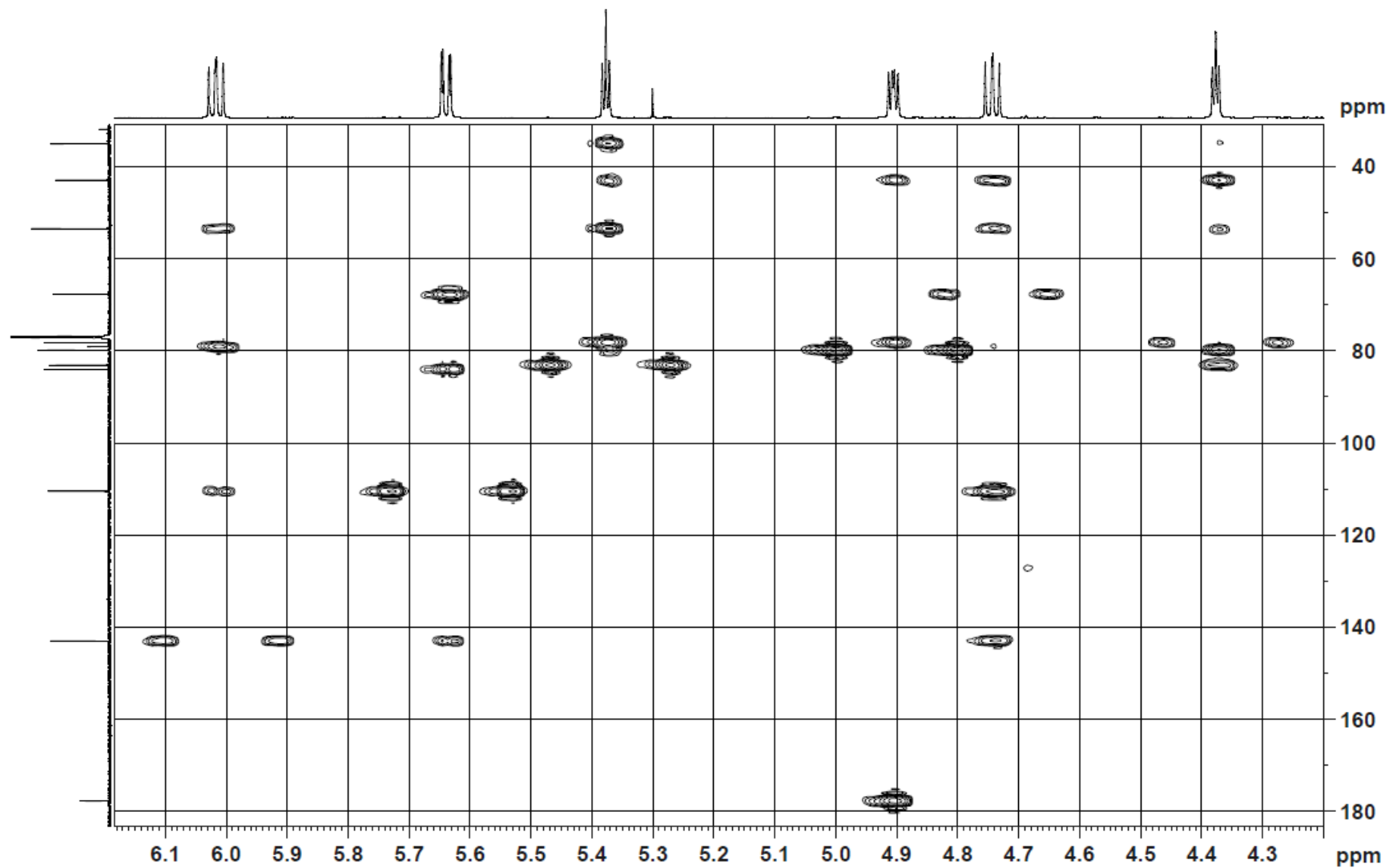


Figure S11: HMBC NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

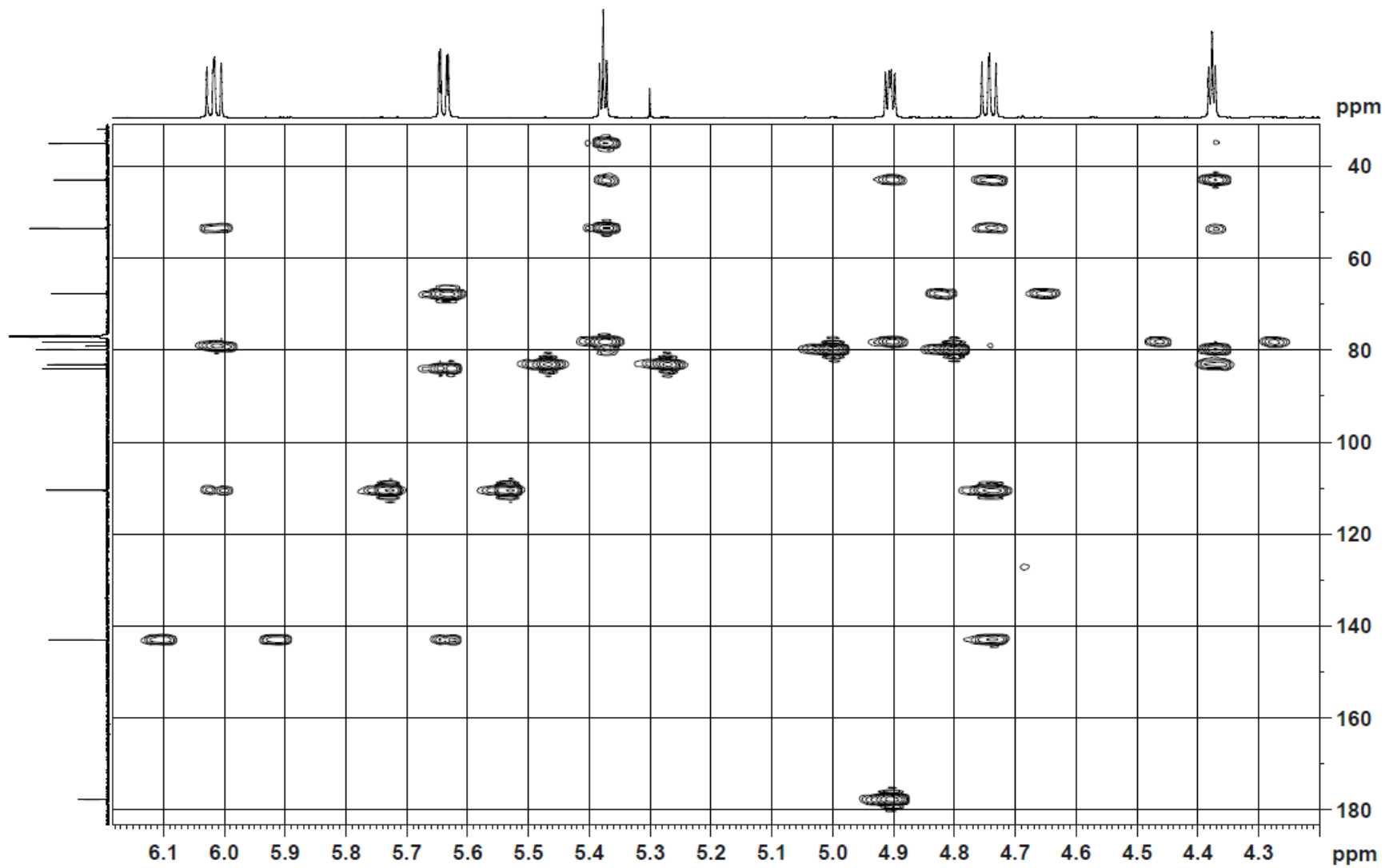


Figure S1m: HMBC NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL₃

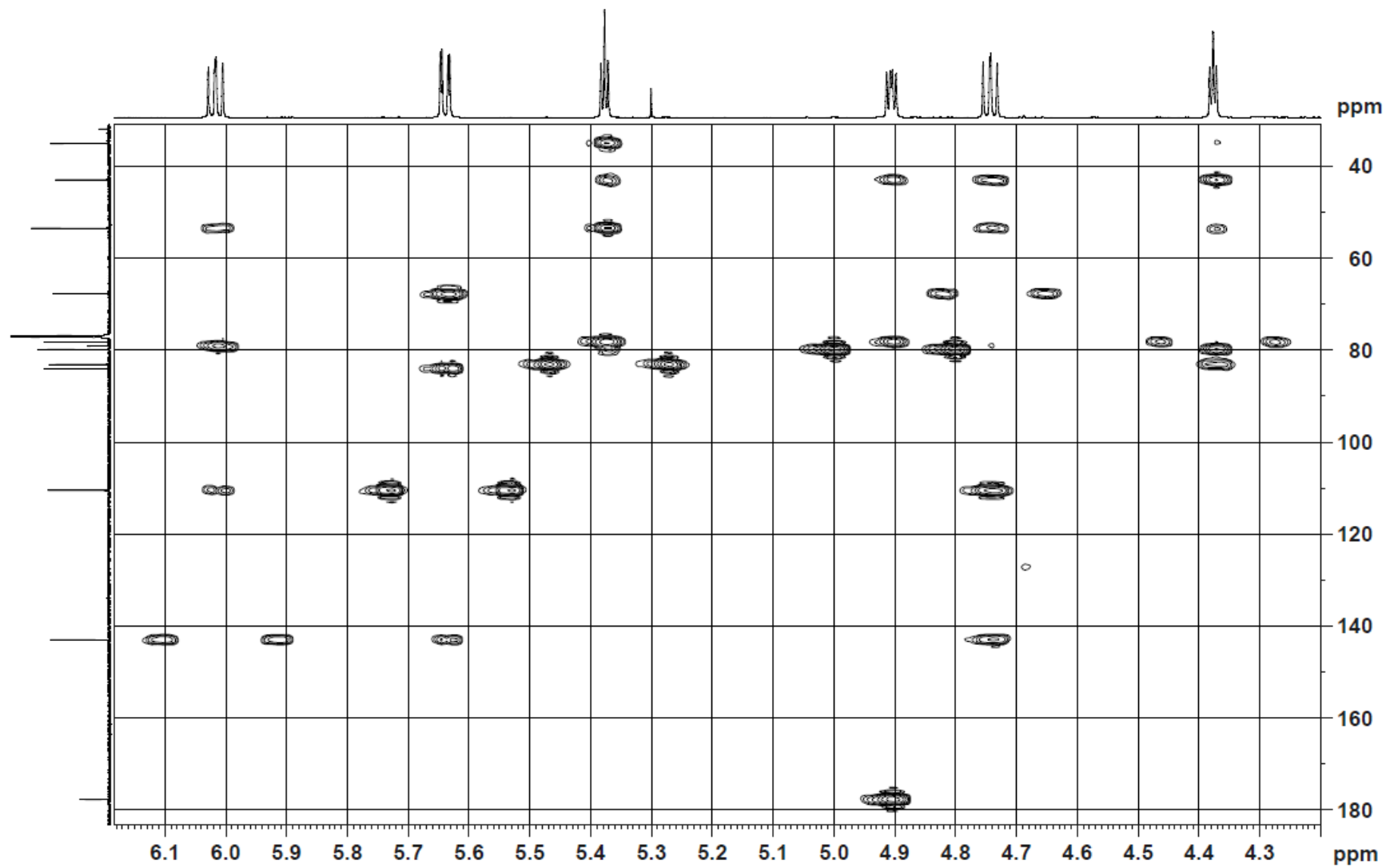
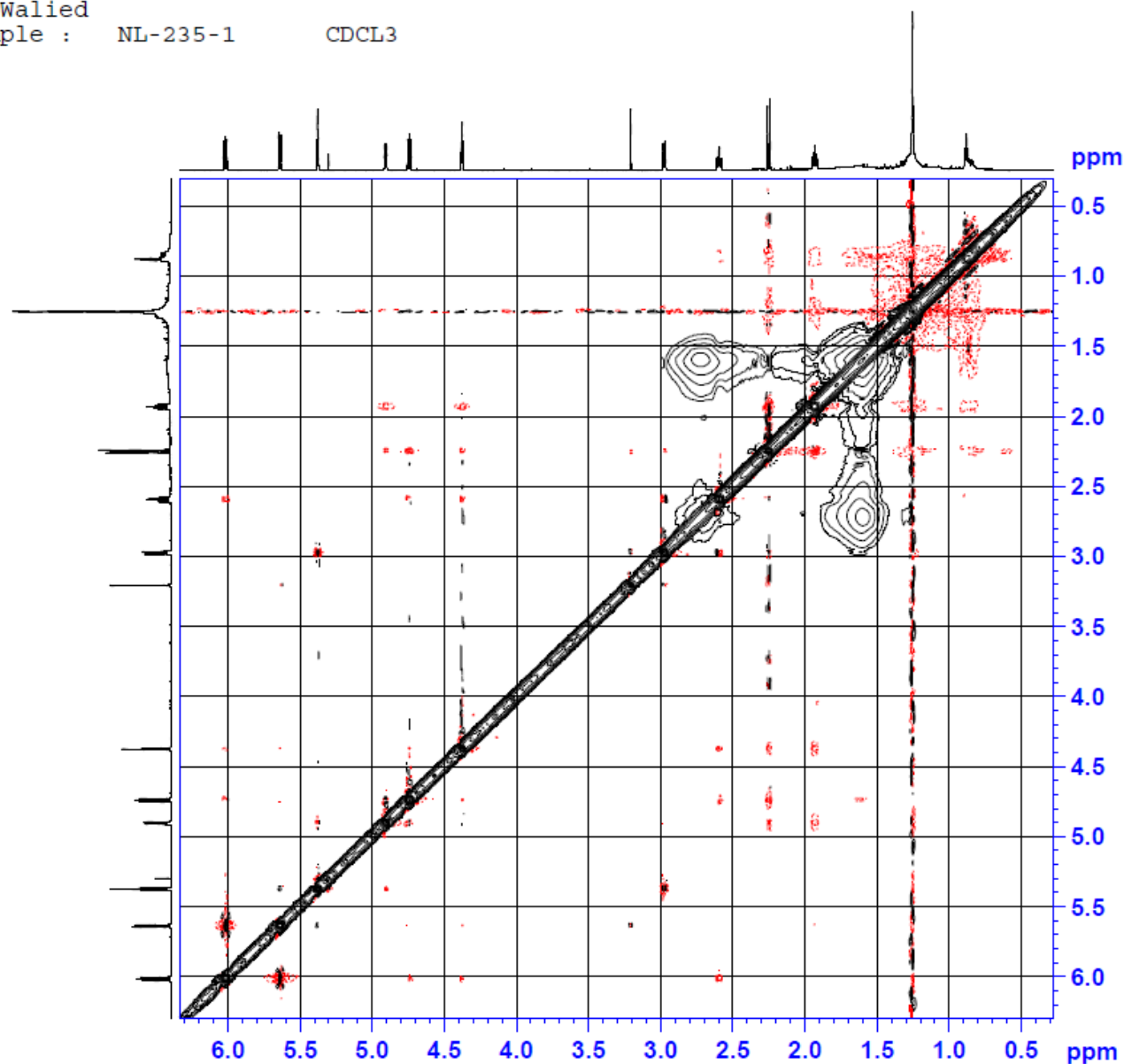


Figure S1n: HMBC NMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL3



Current Data Parameters
NAME WALIED NL-2351 23-02-2017
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170223
Time_ 17.43
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG noesyggpphpg
TD 2048
SOLVENT CDCL3
NS 32
DS 32
SWH 7812.500 Hz
FIDRES 3.814697 Hz
AQ 0.1310720 sec
RG 15.7
DW 64.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00005381 sec
D1 1.98934996 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00012800 sec

----- CHANNEL f1 -----
SFO1 850.1530380 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 -----
GPNAM[1] SMCQ10.100
GPZ1 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 850.153 MHz
FIDRES 30.517578 Hz
SW 9.190 ppm
FnMODE States-TPPI

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

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 850.1500200 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

Figure S10: NOESY NMR of compound 1

Dr. Walied
Sample : NL-235-1 CDCL3

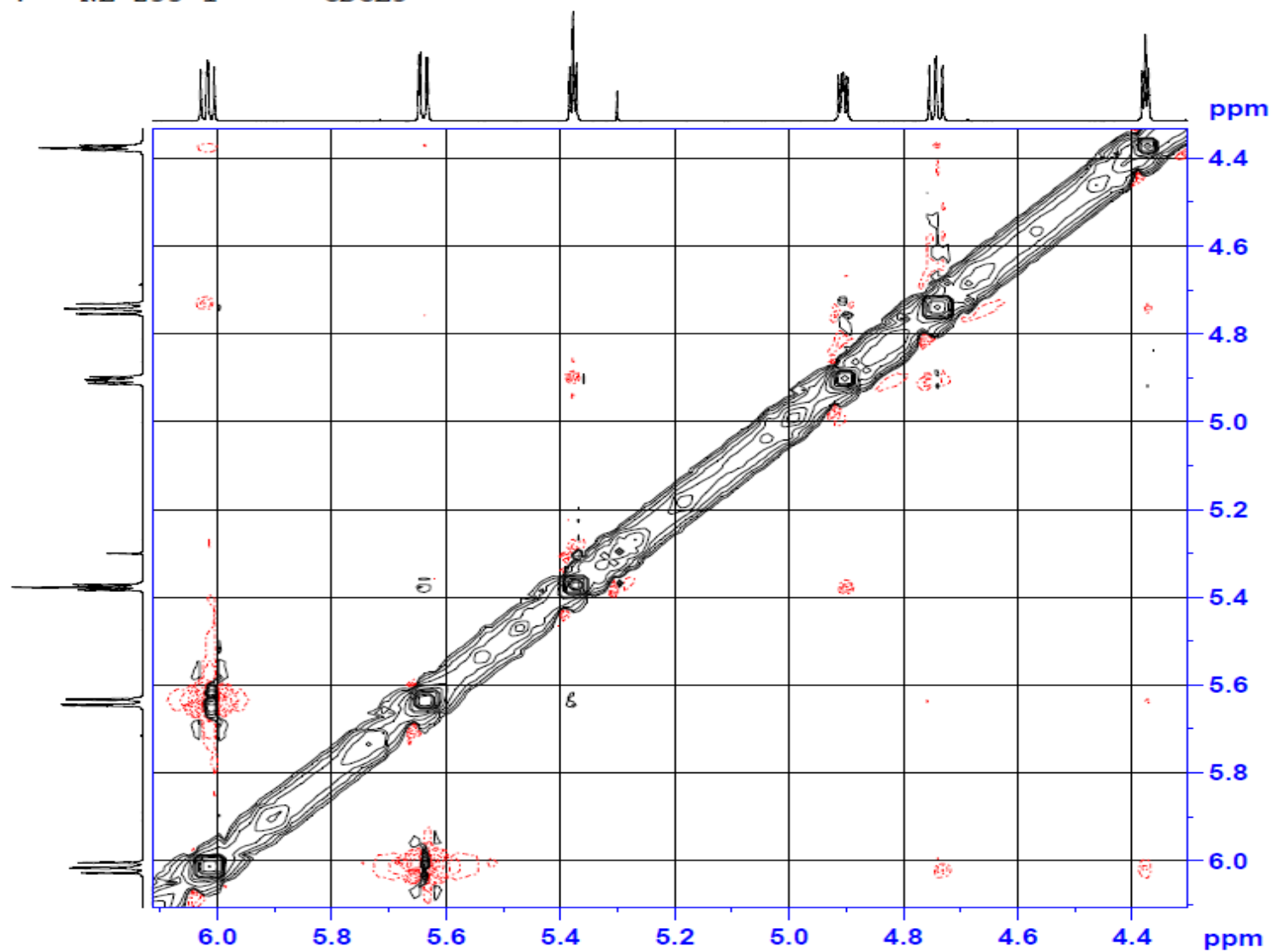


Figure S1p: NOESY NMR of compound 1

Dr. Walied

Sample : NL-235-1

CDCL3

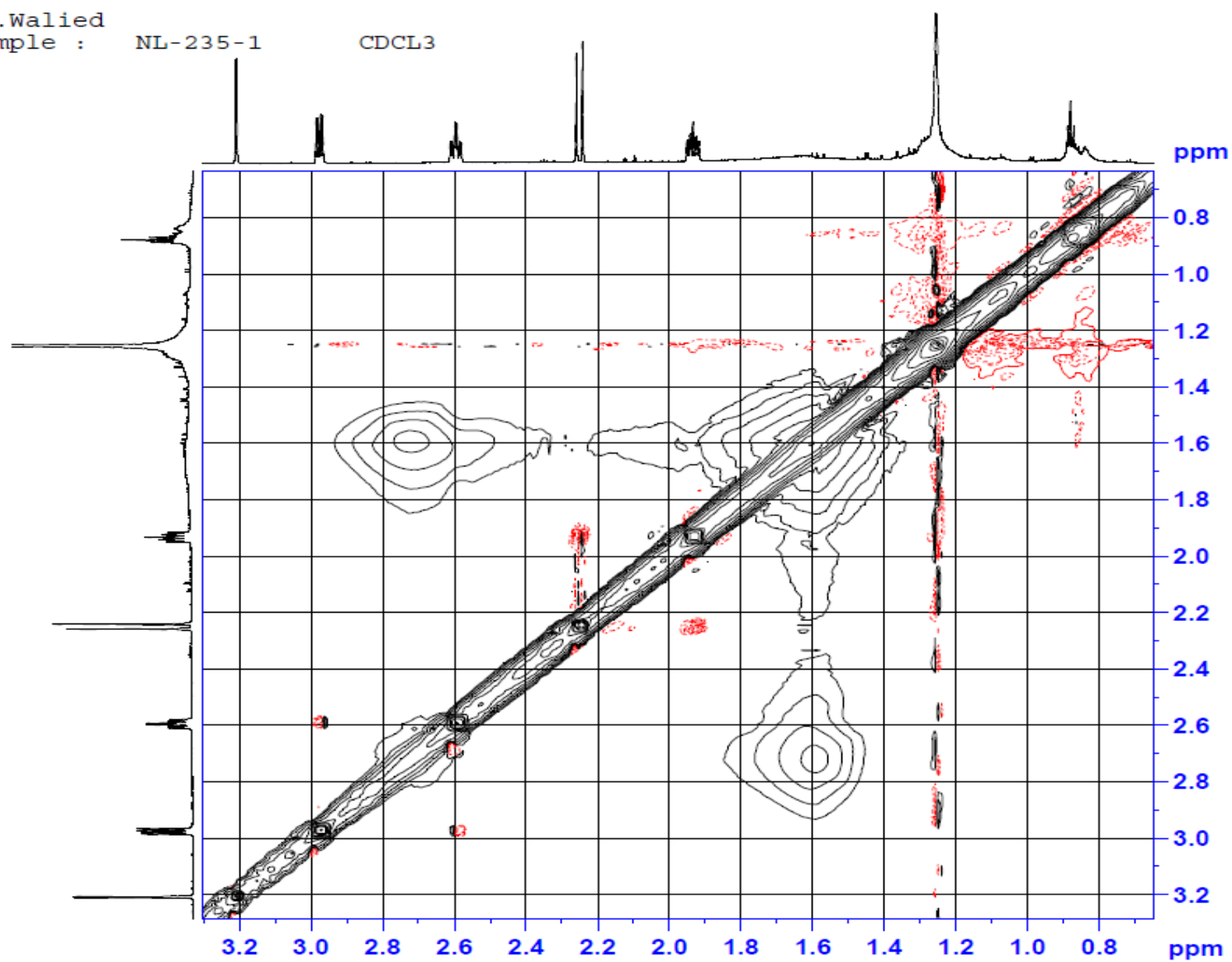
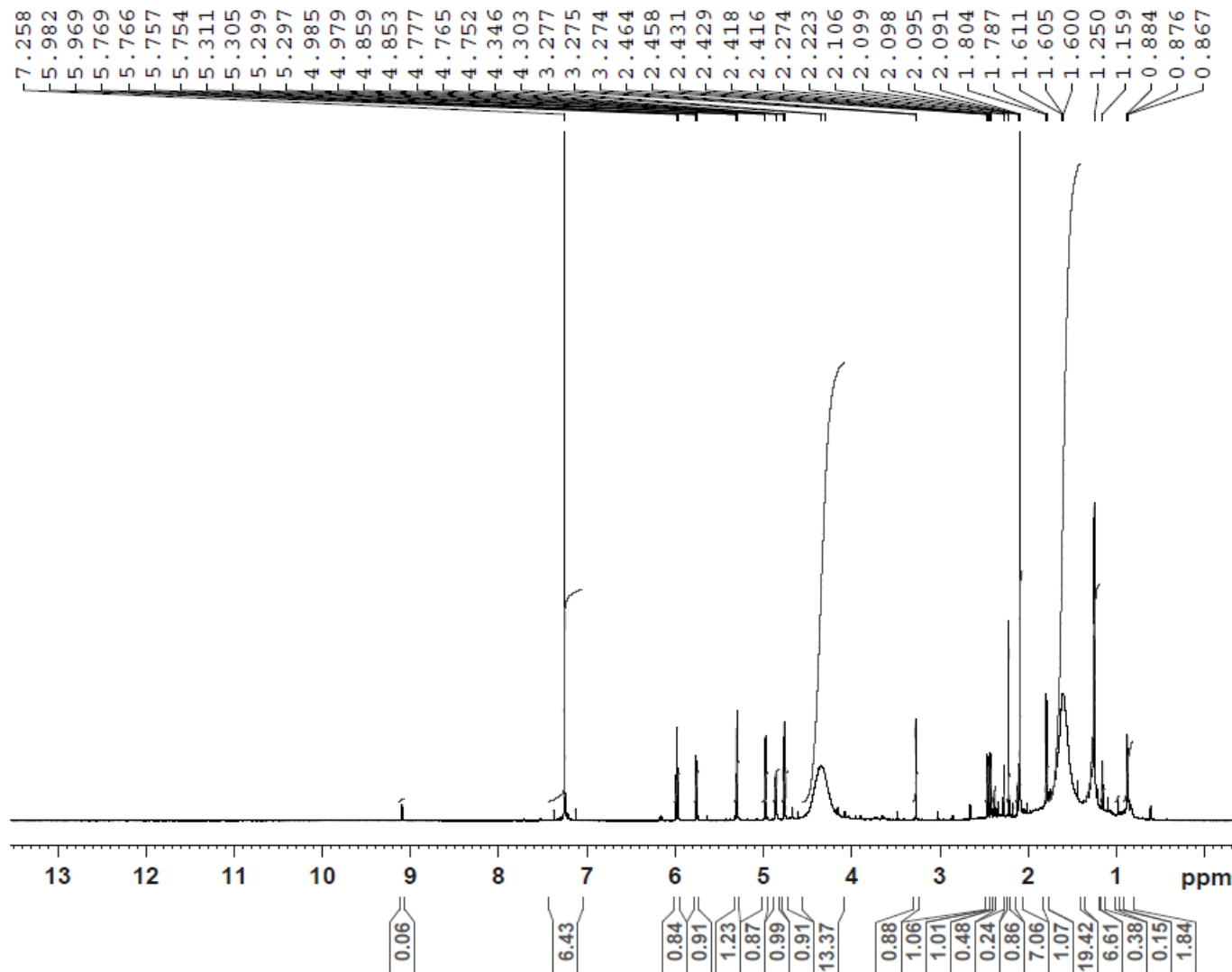


Figure S1q: NOESY NMR of compound 1

Dr. Walied

Sample : NL-229-2 CDCL3



Current Data Parameters
NAME WALIED NL-229-2 21-02-2017
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170221
Time_ 10.14
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 17006.803 Hz
FIDRES 0.259503 Hz
AQ 1.9267584 sec
RG 10.55
DM 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 850.1500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

Figure S2a: ¹H NMR of compound 2

Dr. Walied

Sample : NL-229-2 CDCL₃

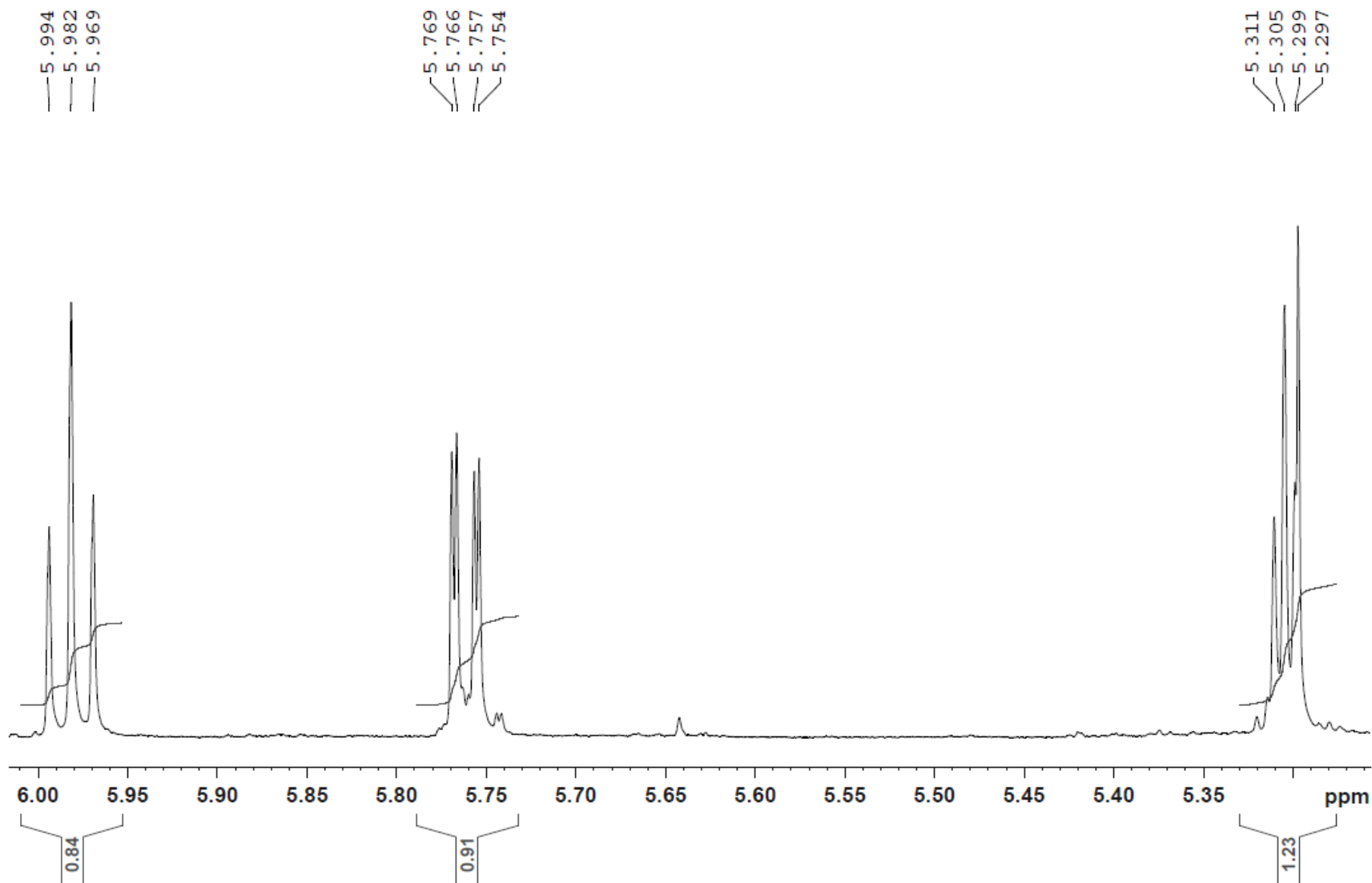


Figure S2a: ¹H NMR of compound 2

Dr. Walied

Sample : NL-229-2 CDCL₃

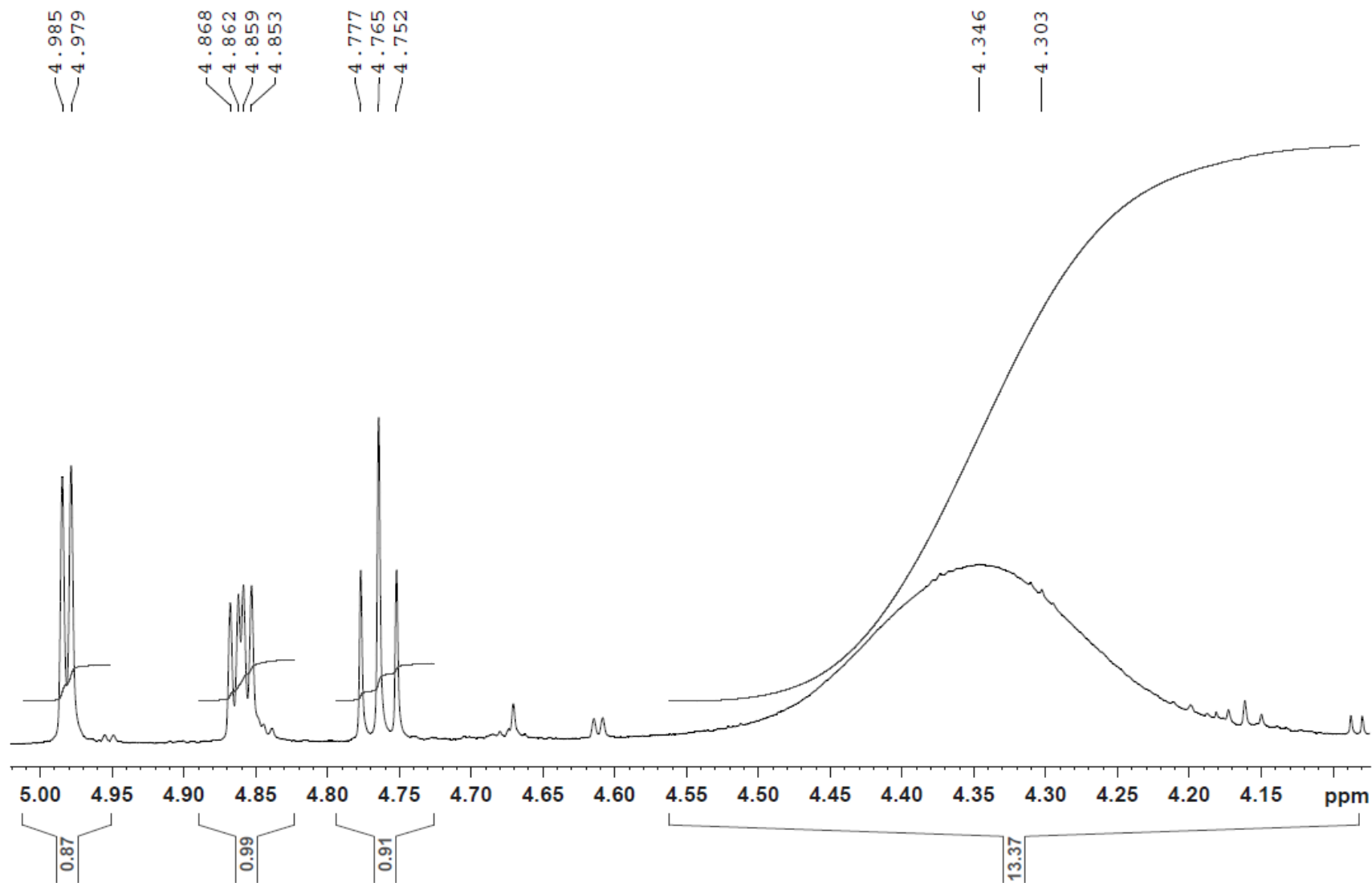


Figure S2b: ¹H NMR of compound 2

Dr. Walled

Sample : NL-229-2

CDCL₃

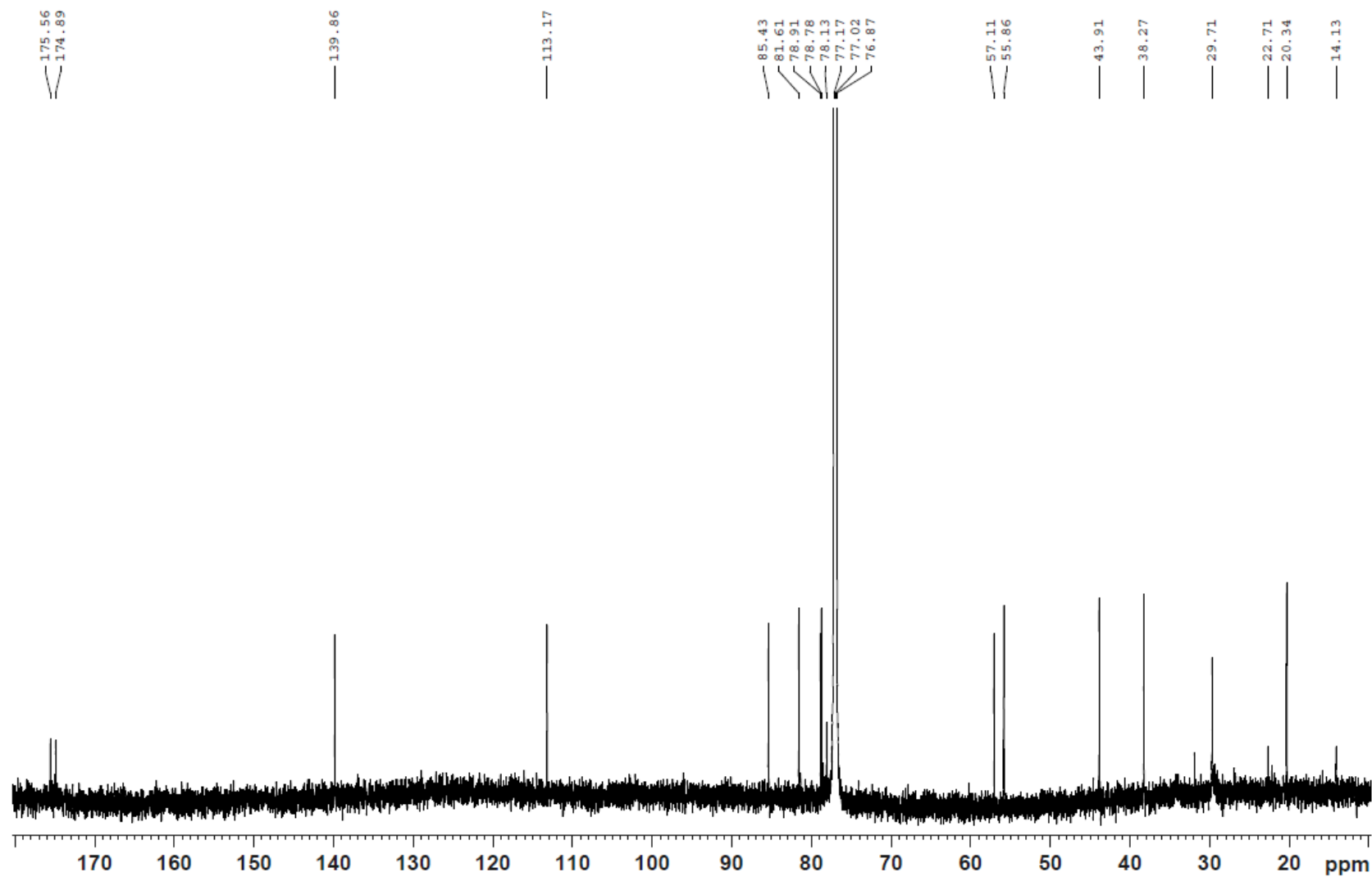
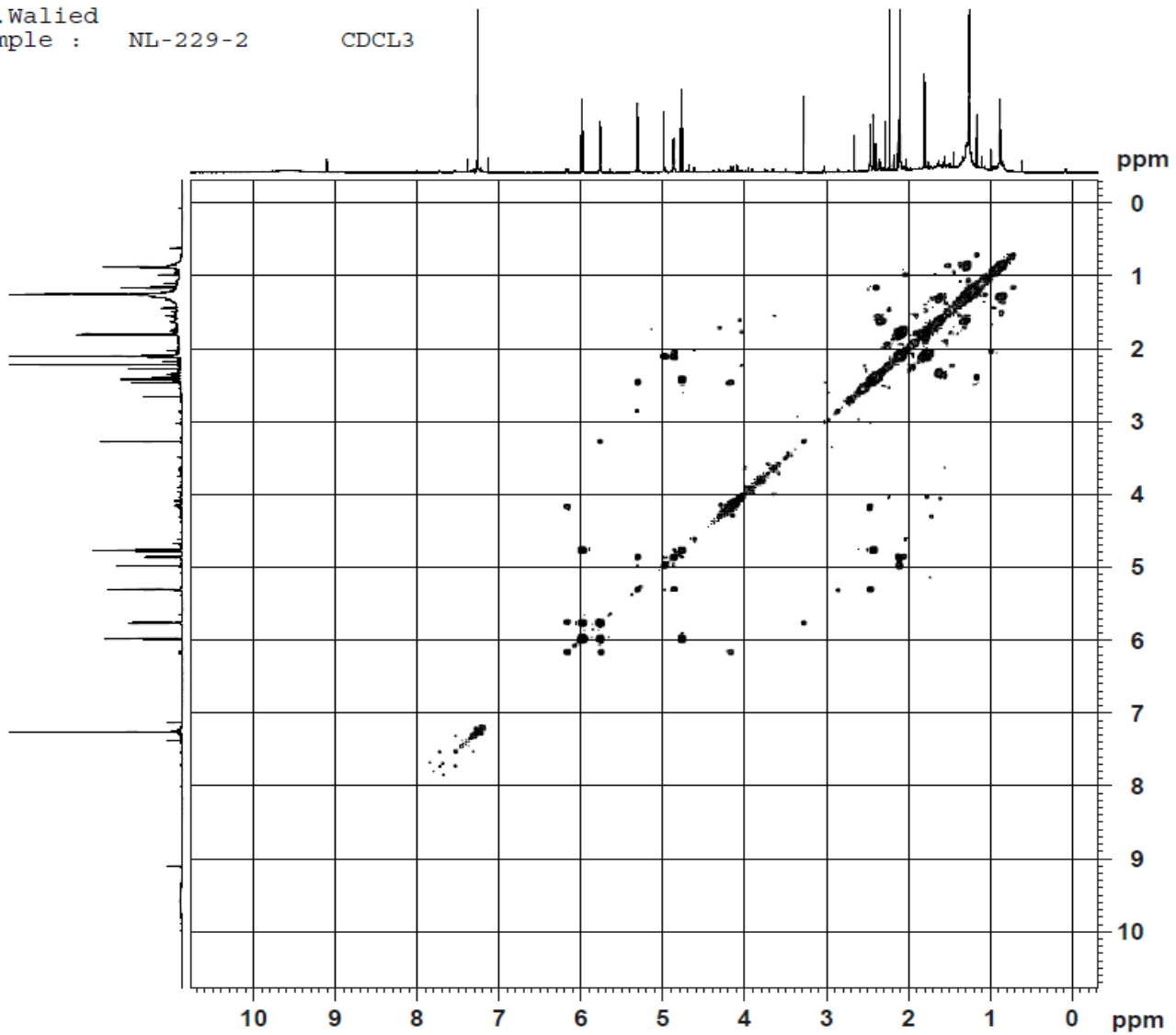


Figure S2c: ¹³C NMR of compound 2

Dr. Walied
Sample : NL-229-2 CDCL3



```
Current Data Parameters
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EXPNO  43
PROCNO  1

F2 - Acquisition Parameters
Date_   20170226
Time    11.17
INSTRUM spect
PROBHD  5 mm CPQCI 1H-
PULPROG cosygpmEqf
TD      2048
SOLVENT CDCL3
NS      16
DS      8
SWH     9433.962 Hz
FIDRES  4.606427 Hz
AQ      0.1085440 sec
RG      186.93
DW      53.000 usec
DE      10.00 usec
TE      298.0 K
DO      0.00000300 sec
D1      1.98156798 sec
D13     0.00000400 sec
D16     0.00020000 sec
IN0     0.00010600 sec

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SFO1    850.1544606 MHz
NUC1    1H
P1      8.00 usec
PLW1    15.30000019 W

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GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPZ1    16.00 %
GPZ2    12.00 %
GPZ3    40.00 %
P16     1000.00 usec

F1 - Acquisition parameters
TD      128
SFO1    850.1545 MHz
FIDRES  73.702827 Hz
SW      11.097 ppm
PrMODE  QF

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

F1 - Processing parameters
SI      1024
MC2     QF
SF      850.1500200 MHz
WDW     SINE
SSB     0
LB      0 Hz
GB      0
```

Figure S2d: COSY NMR of compound 2

Dr. Walied

Sample : NL-229-2

CDCL₃

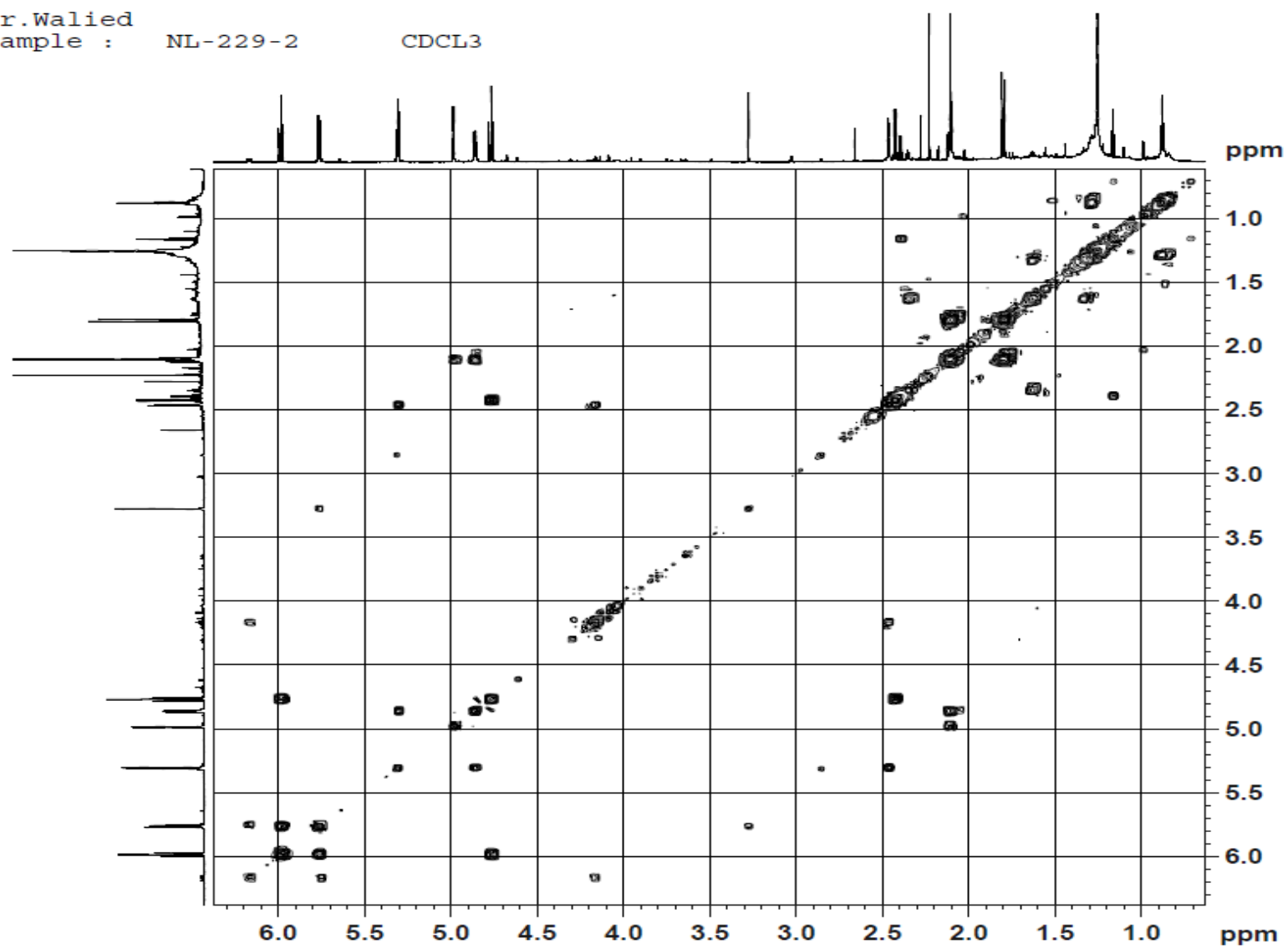


Figure S2e: COSY NMR of compound 2

Dr. Walied
Sample : NL-229-2 CDCL₃

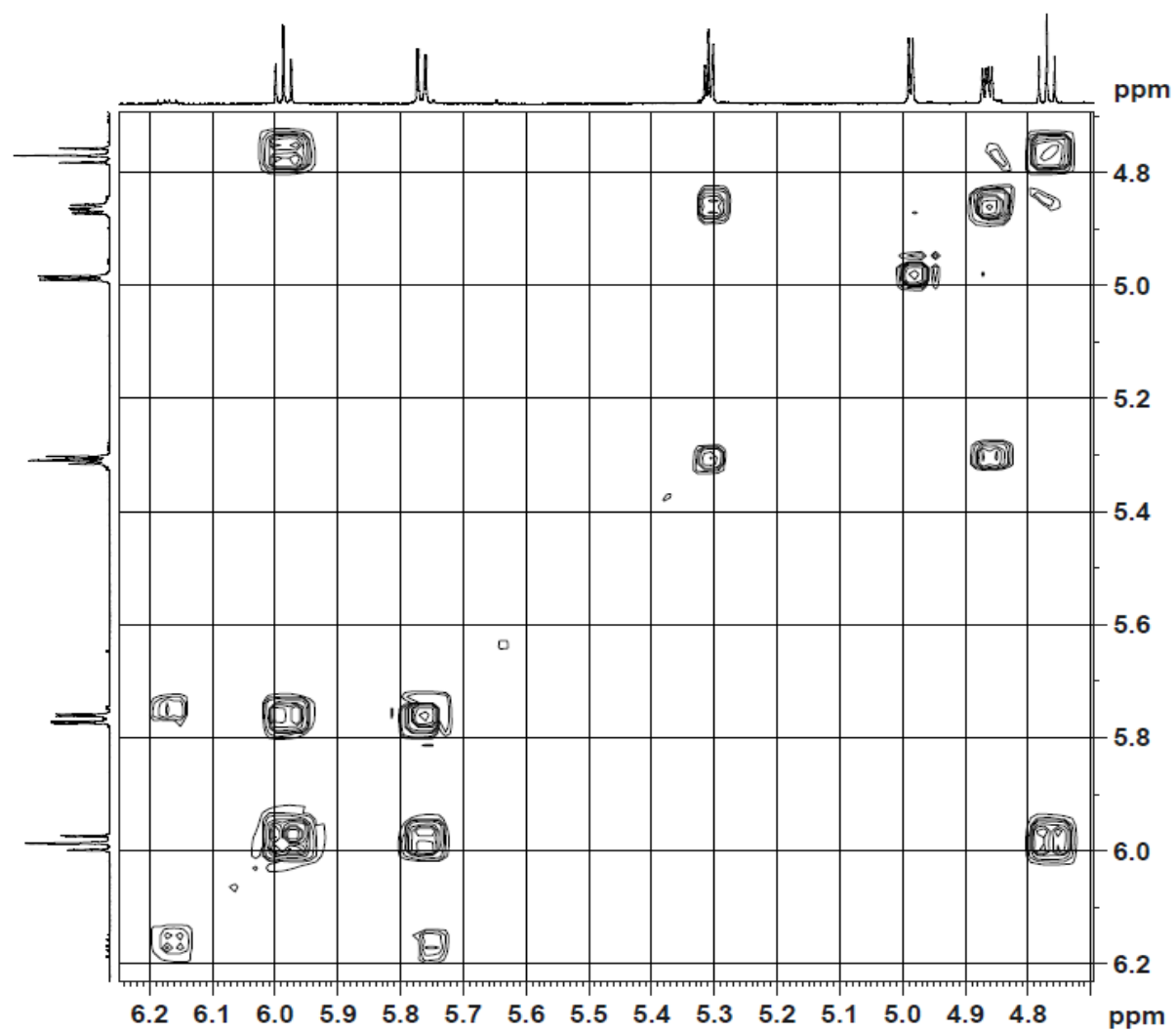
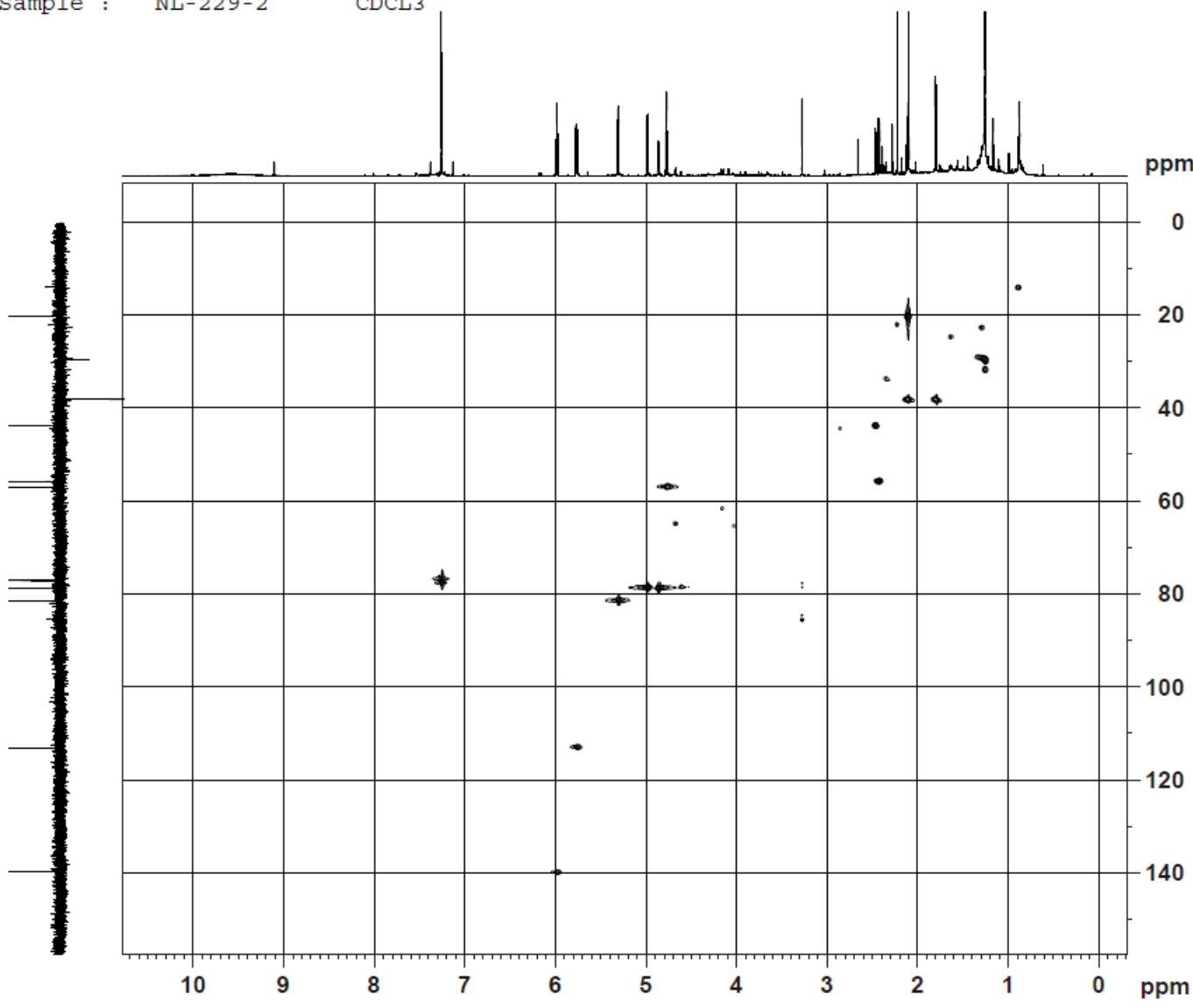


Figure S2f: COSY NMR of compound 2

Dr. Walied
Sample : NL-229-2 CDCL3



Current Data Parameters
NAME WALIED NL-2292 23-02-2017
EXPNO 45
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170226
Time 18.10
INSTRUM spect
PROBHD 5 mm CPQCI 1H
PULPROG hsqcadtgpp
TD 1024
SOLVENT CDCL3
NS 32
DS 16
SMH 9433.962 Hz
FIDRES 9.212853 Hz
AQ 0.0542720 sec
RG 186.93
DM 53.000 usec
DE 10.00 usec
TE 298.0 K
CNS2 145.0000000
D0 0.00000300 sec
D1 1.49214900 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.00000400 sec
D16 0.00020000 sec
D21 0.00345000 sec
INO 0.00001410 sec
ZGPPPM

----- CHANNEL f1 -----
SP01 850.1544606 MHz
NUC1 1H
P1 8.00 usec
P2 16.00 usec
P2B 0 usec
PLW1 15.30000019 W
----- CHANNEL f2 -----
SP02 213.7863316 MHz
NUC2 13C
CPDPRG2 garp
P3 12.00 usec
P4 24.00 usec
PCPD2 45.00 usec
PLW2 130.00000000 W
PLW12 9.24440002 W

----- GRADIENT CHANNEL -----
GPMAM[1] SMSQ10.100
GPMAM[2] SMSQ10.100
GPZ1 80.00 %
GPZ2 20.10 %
PLG 1000.00 usec

F1 - Acquisition parameters
TD 256
SP01 213.7863 MHz
FIDRES 138.519501 Hz
SW 165.871 ppm
PnMODE Echo-Antiacho

F2 - Processing parameters
SI 1024
SF 850.1500200 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiacho
SF 213.7703875 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

Figure S2g: HSQC NMR of compound 2

Dr. Walied

Sample : NL-229-2

CDCL₃



Current Data Parameters
NAME WALIED NL-2292 23-02-2017
EXPNO 46
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170226
Time 21.47
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG hmcgpgndqf
TD 4096
SOLVENT CDCl₃
NS 64
DS 16
SMH 9433.962 Hz
FIDRES 2.303213 Hz
AQ 0.2170880 sec
RG 186.93
DM 53.000 usec
DE 10.00 usec
TE 298.0 K
CNST13 8.0000000
D0 0.00000300 sec
D1 1.46395504 sec
D6 0.06250000 sec
D16 0.00020000 sec
INO 0.00001050 sec

----- CHANNEL f1 -----
SFO1 850.1544606 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 -----
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPE1 50.00 %
GPE2 30.00 %
GPE3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 213.7917 MHz
FIDRES 372.023804 Hz
SW 222.736 ppm
F1MODE QF

F2 - Processing parameters
SI 1024
SF 850.1500200 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

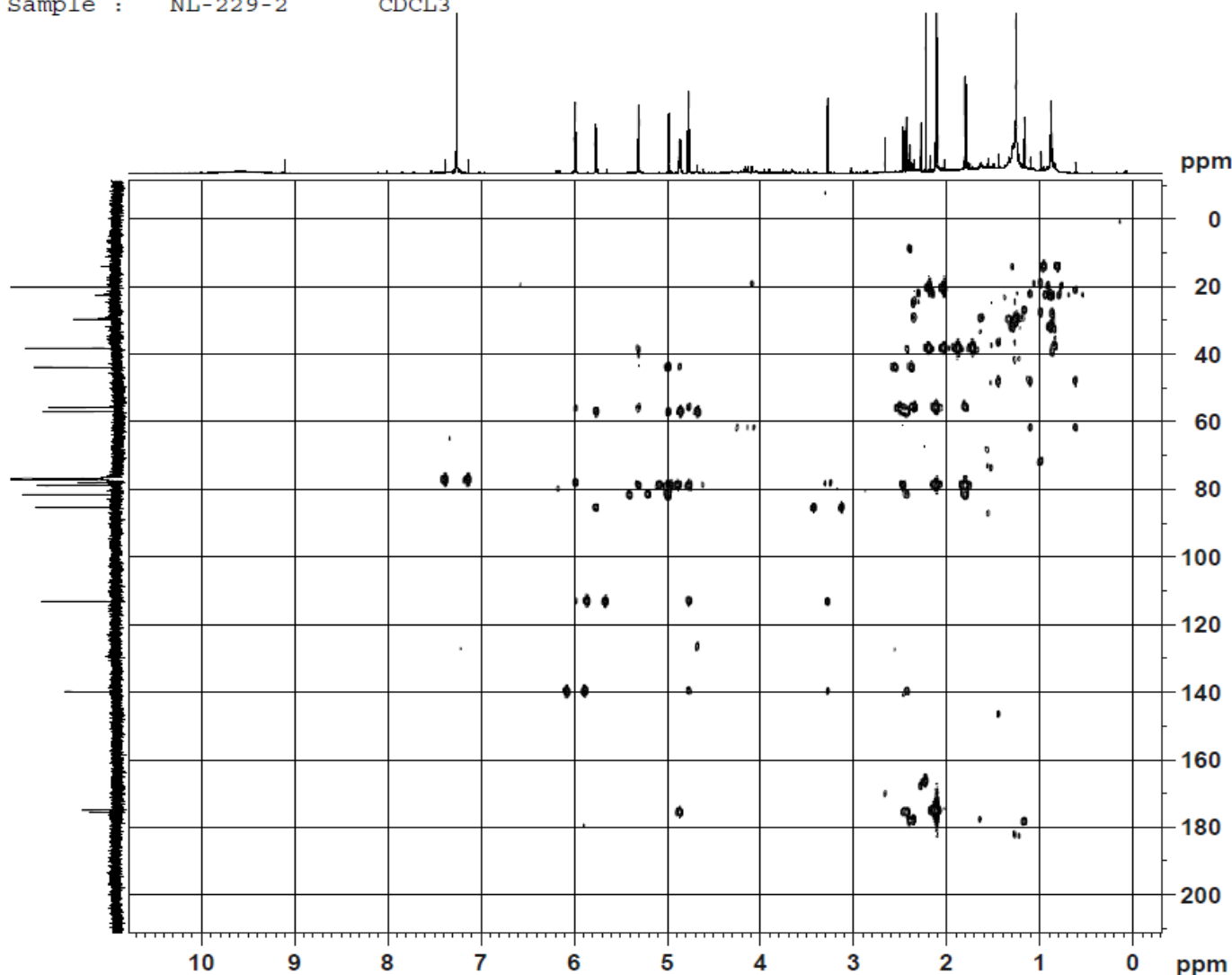


Figure S2h: HMBC NMR of compound 2

Dr. Walied

Sample : NL-229-2

CDCL₃

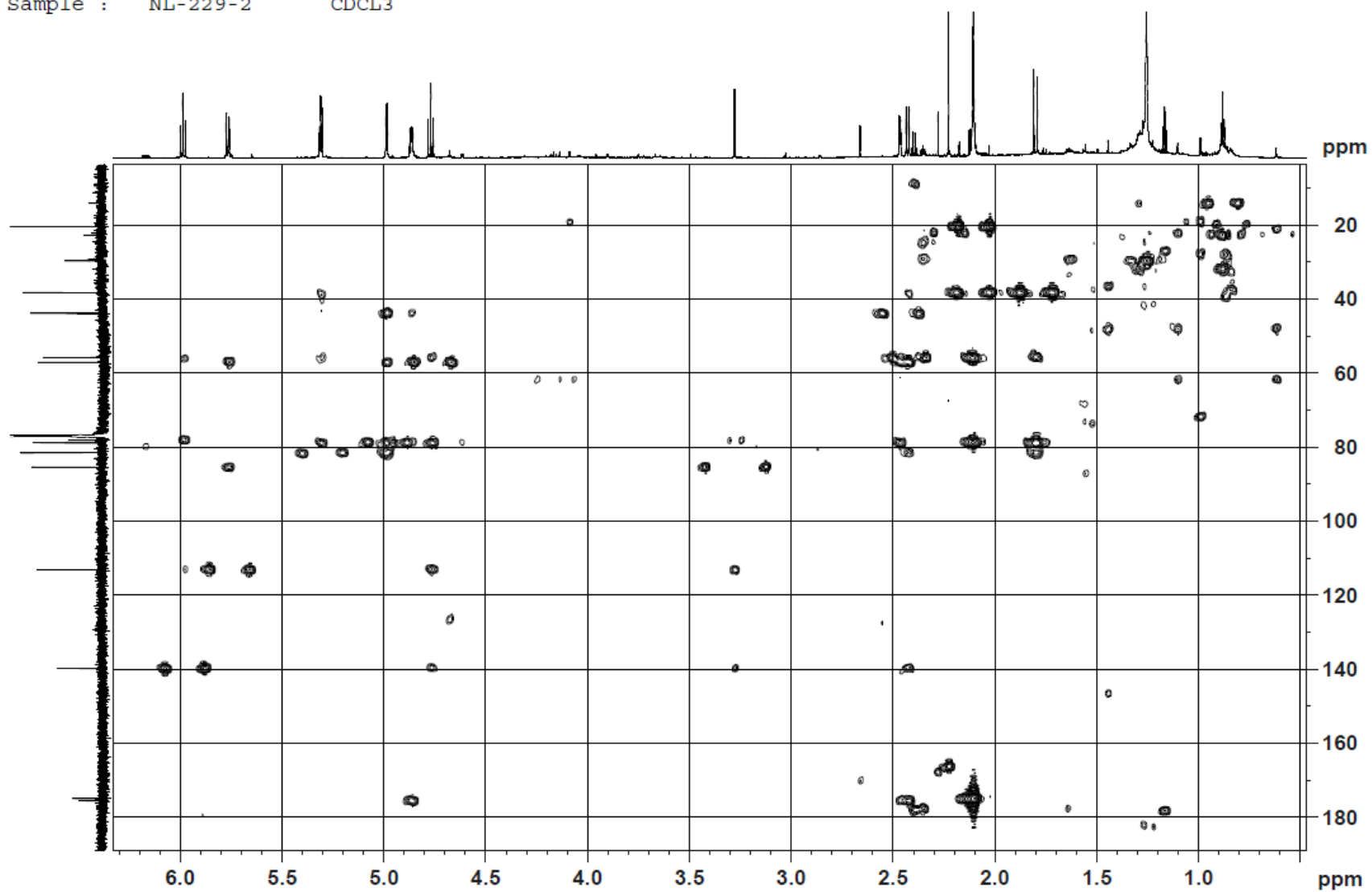


Figure S2i: HMBC NMR of compound 2

Dr. Walied

Sample : NL-229-2 CDCL3

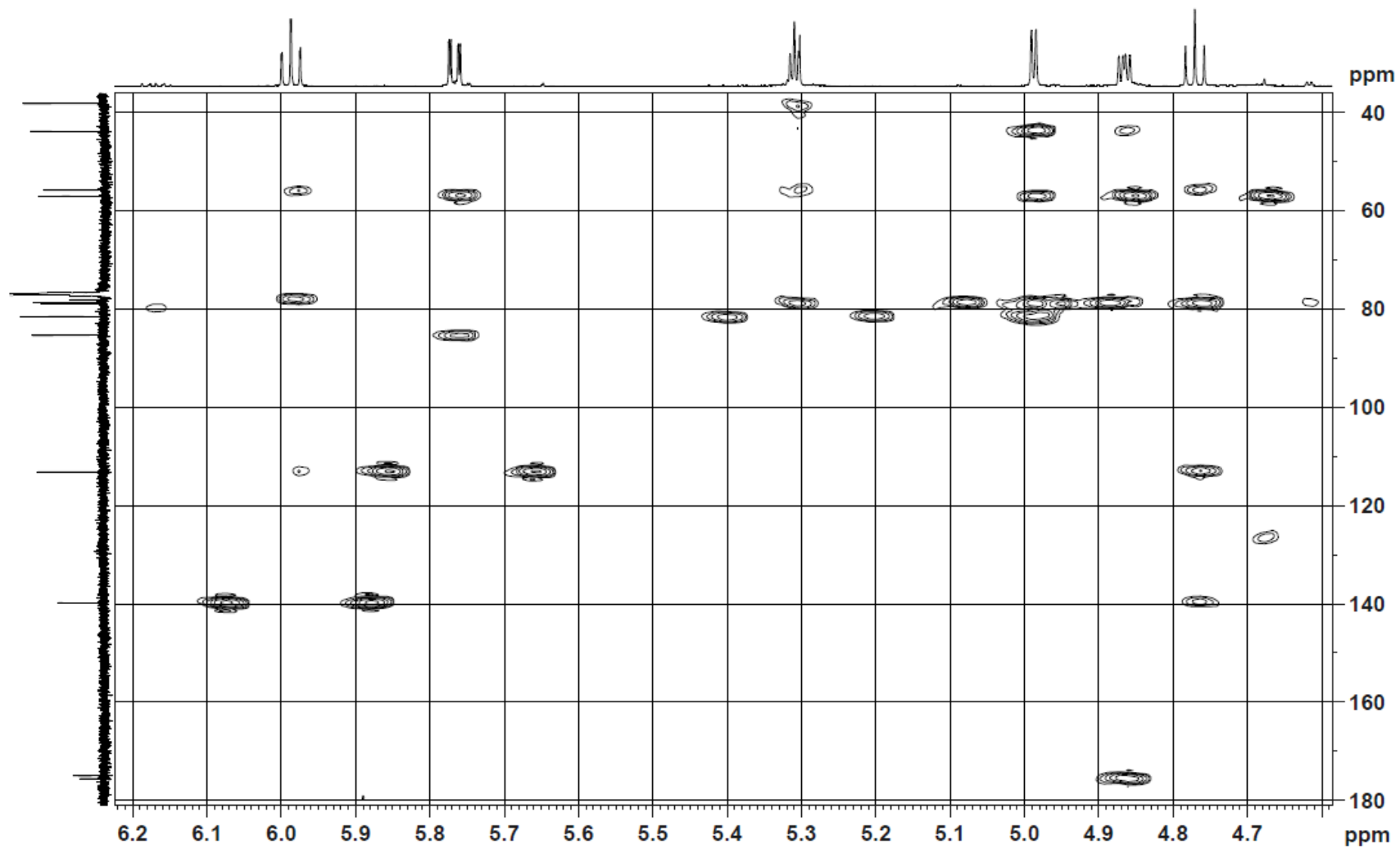


Figure S2j: HMBC NMR of compound 2

Dr. Walied

Sample : NL-229-2 CDCL3

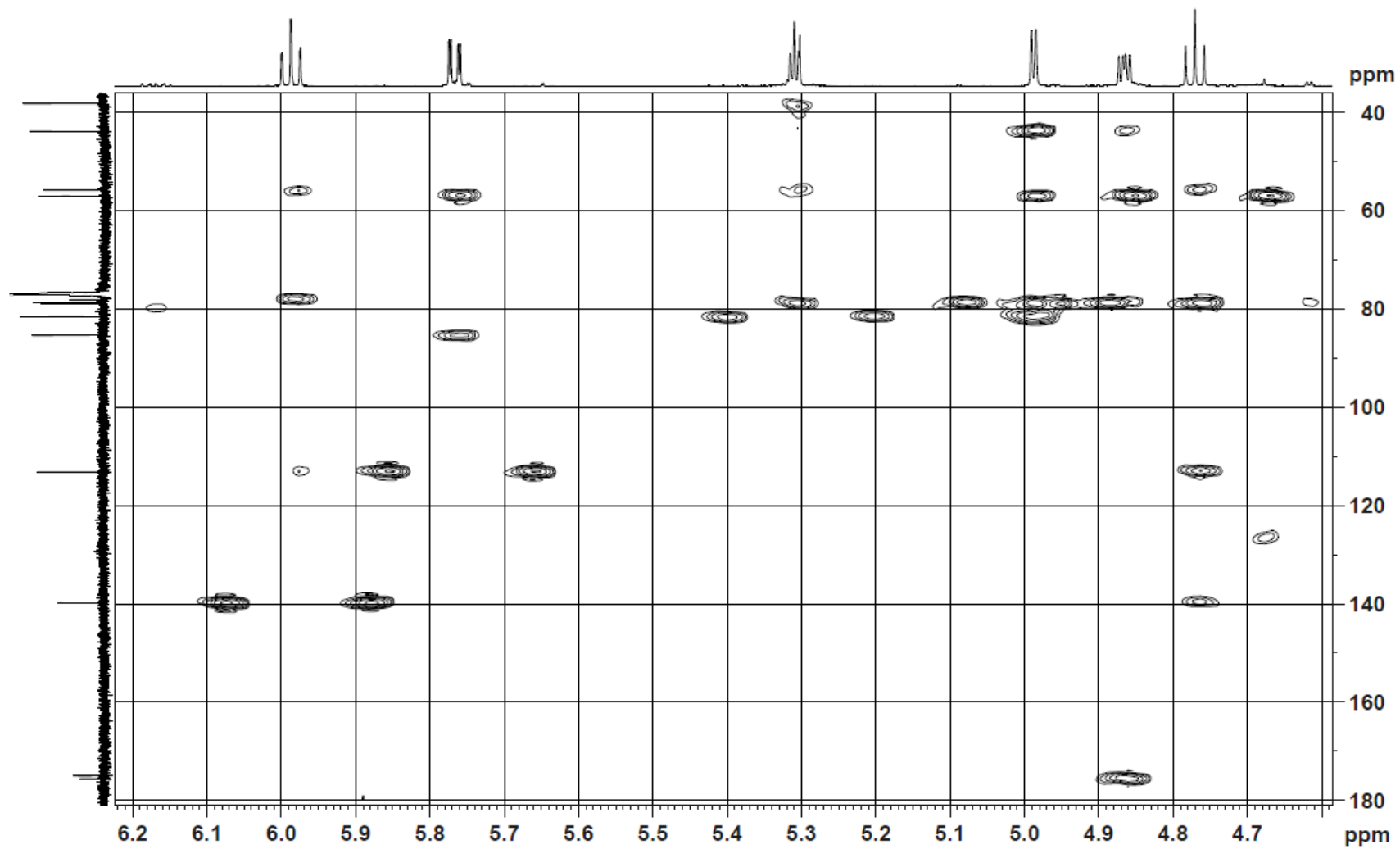


Figure S2k: HMBC NMR of compound 2

Dr. Walied

Sample : NL-229-2

CDCL₃

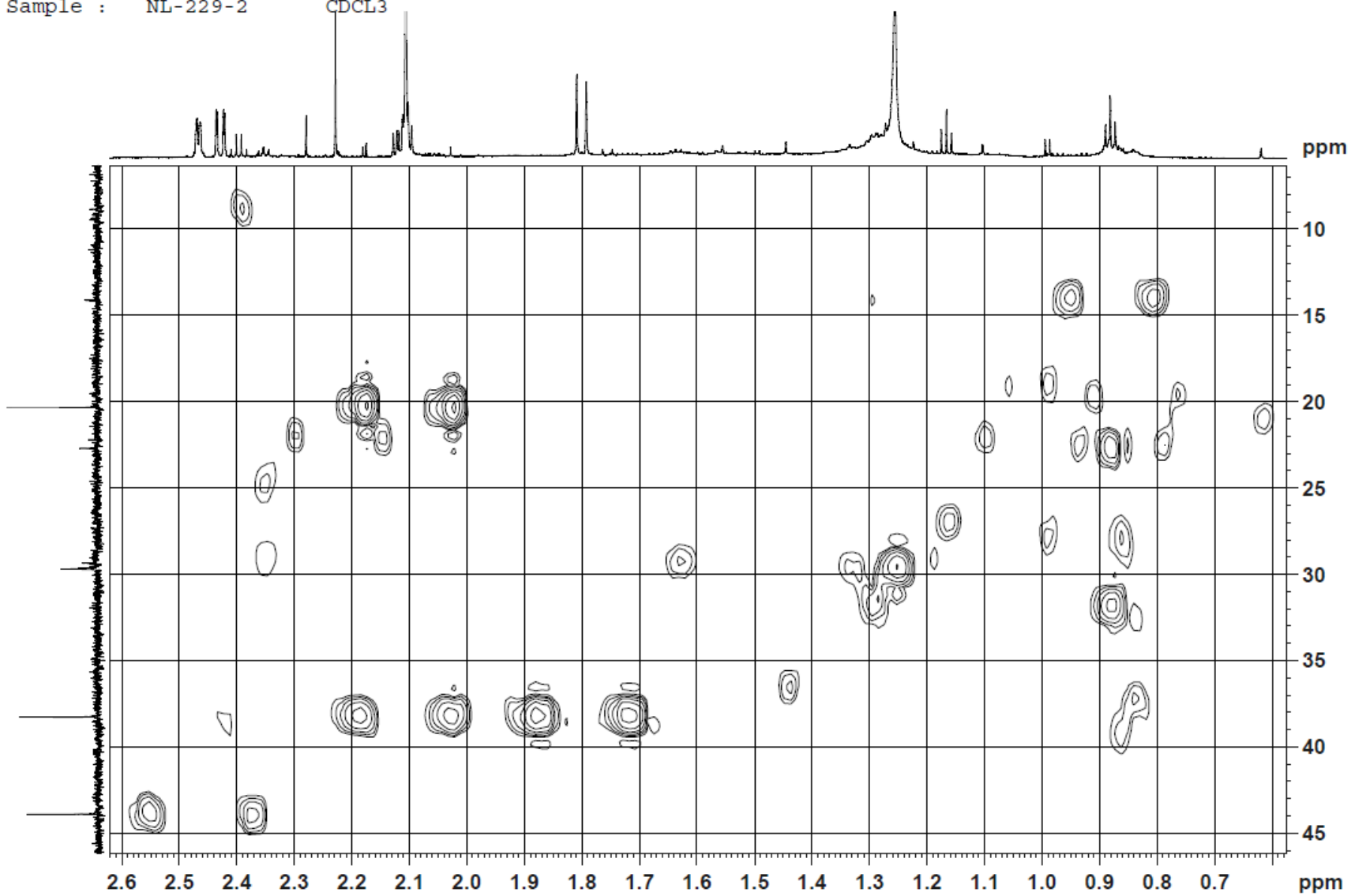
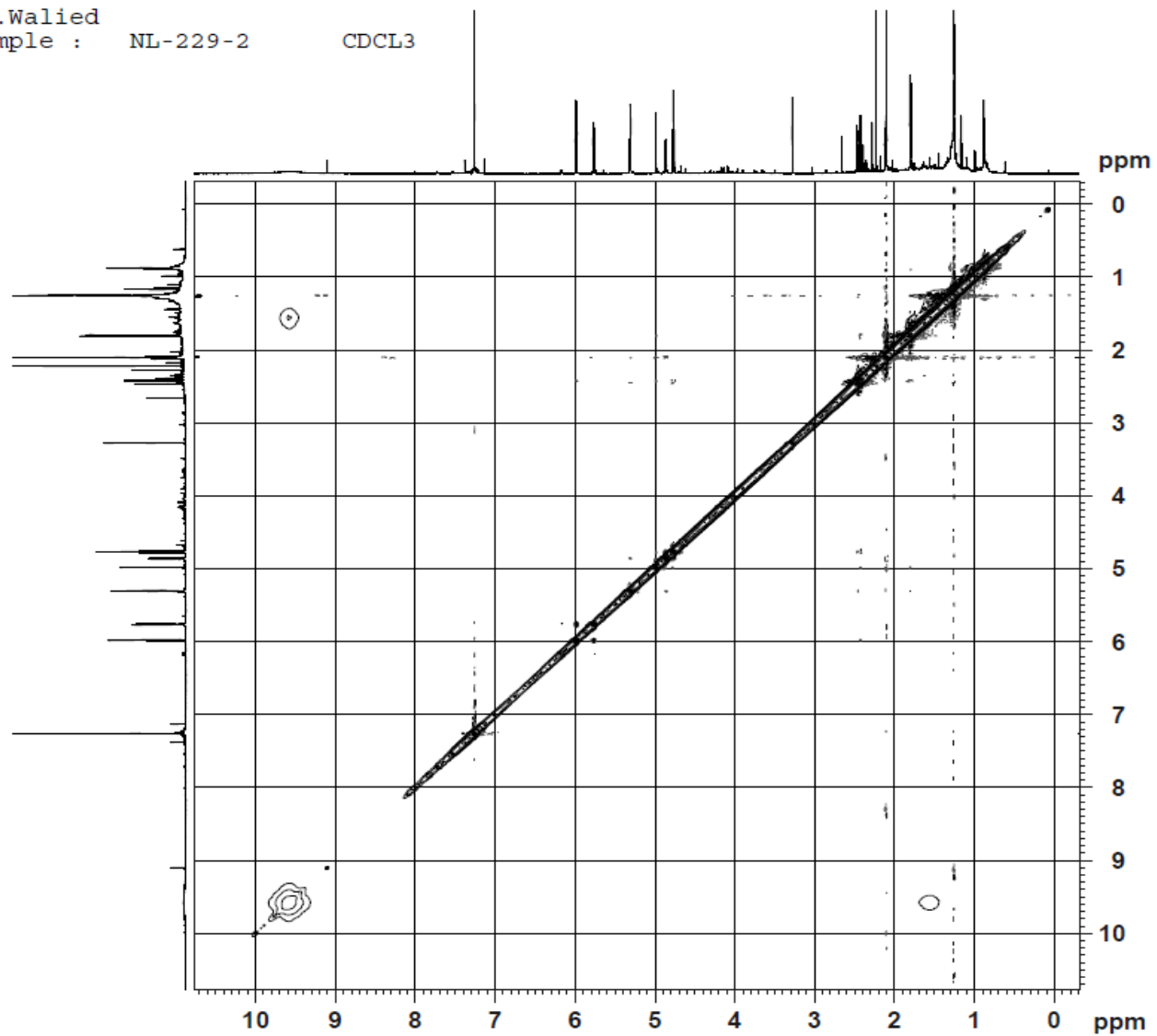


Figure S21: HMBC NMR of compound 2

Dr. Walied
Sample : NL-229-2 CDCL3



```
Current Data Parameters
NAME      WALIED  NL-2292  23-02-2017
EXPNO     44
PROCNO    1

F2 - Acquisition Parameters
Date_     20170226
Time_     12.32
INSTRUM   spect
PROBHD    5 mm CPQCI 1H-
PULPROG   noesygpphpp
TD         2048
SOLVENT   CDCl3
NS         32
DS         32
SWH        9433.962 Hz
FIDRES     4.606427 Hz
AQ         0.1085440 sec
RG         21.28
DW         53.000 usec
DE         10.00 usec
TE         298.0 K
DO         0.00004281 sec
D1         2.01187801 sec
D8         0.30000001 sec
D11        0.03000000 sec
D12        0.00002000 sec
D16        0.00020000 sec
IN0        0.00010600 sec

----- CHANNEL f1 -----
SFO1      850.1544606 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 -----
GPNAM[1]  SMEQ10.100
GPZ1      40.00 %
P16       1000.00 usec

F1 - Acquisition parameters
TD         256
SFO1      850.1545 MHz
FIDRES     36.951414 Hz
SW         11.097 ppm
FnMODE     States-TPPI

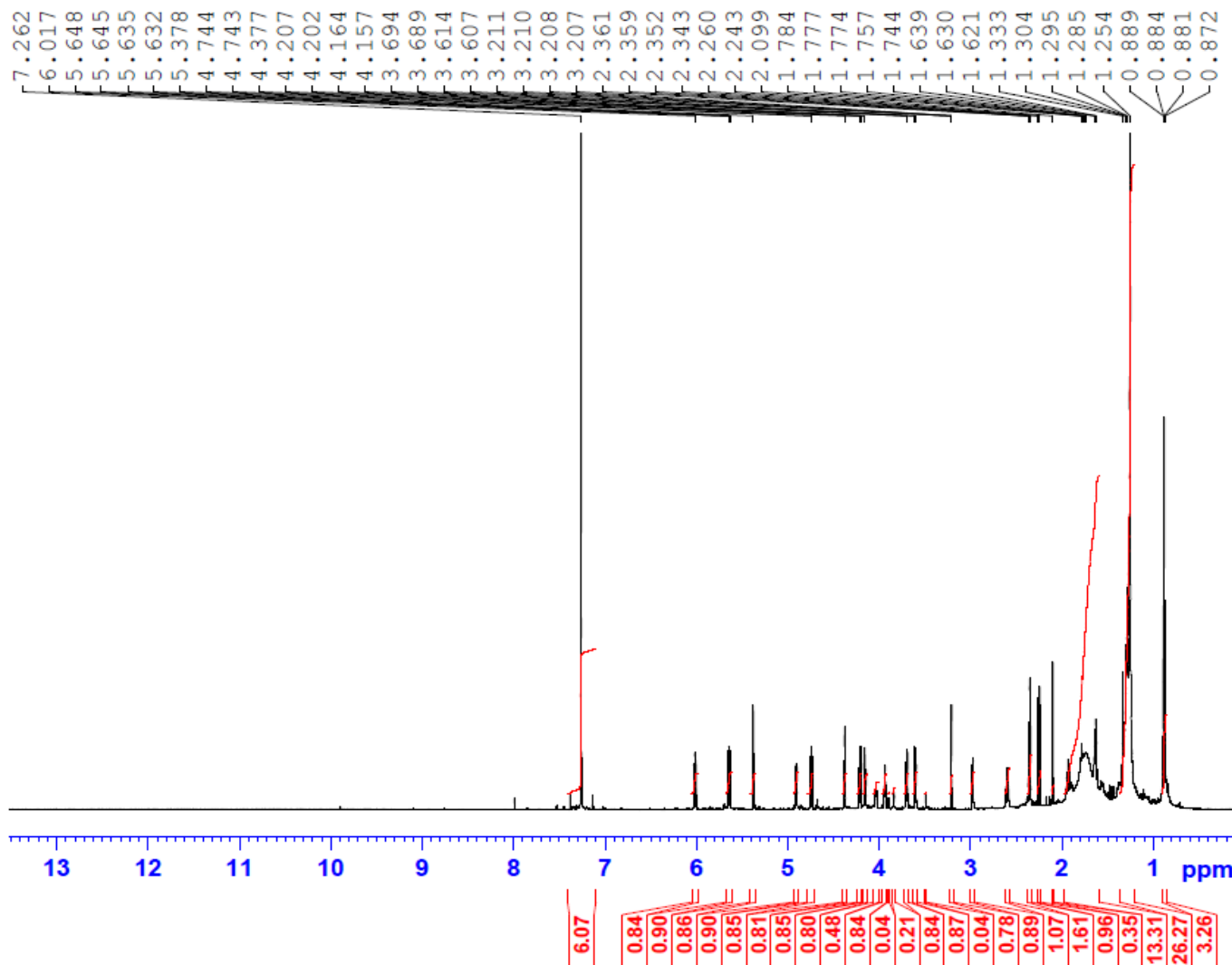
F2 - Processing parameters
SI         1024
SF         850.1500200 MHz
WDW        QSINE
SSB        2
LB         0 Hz
GB         0
PC         1.00

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

Figure S2l: NOSY NMR of compound 2

Dr.Walied

Sample : NL-244-4 CDCL3



Current Data Parameters
NAME WALIED NL-244-4 15-03-2017
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170315
Time 11.27
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCL3
NS 32
DS 2
SMH 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 850.1502000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

Figure S3a: ¹H NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCl₃

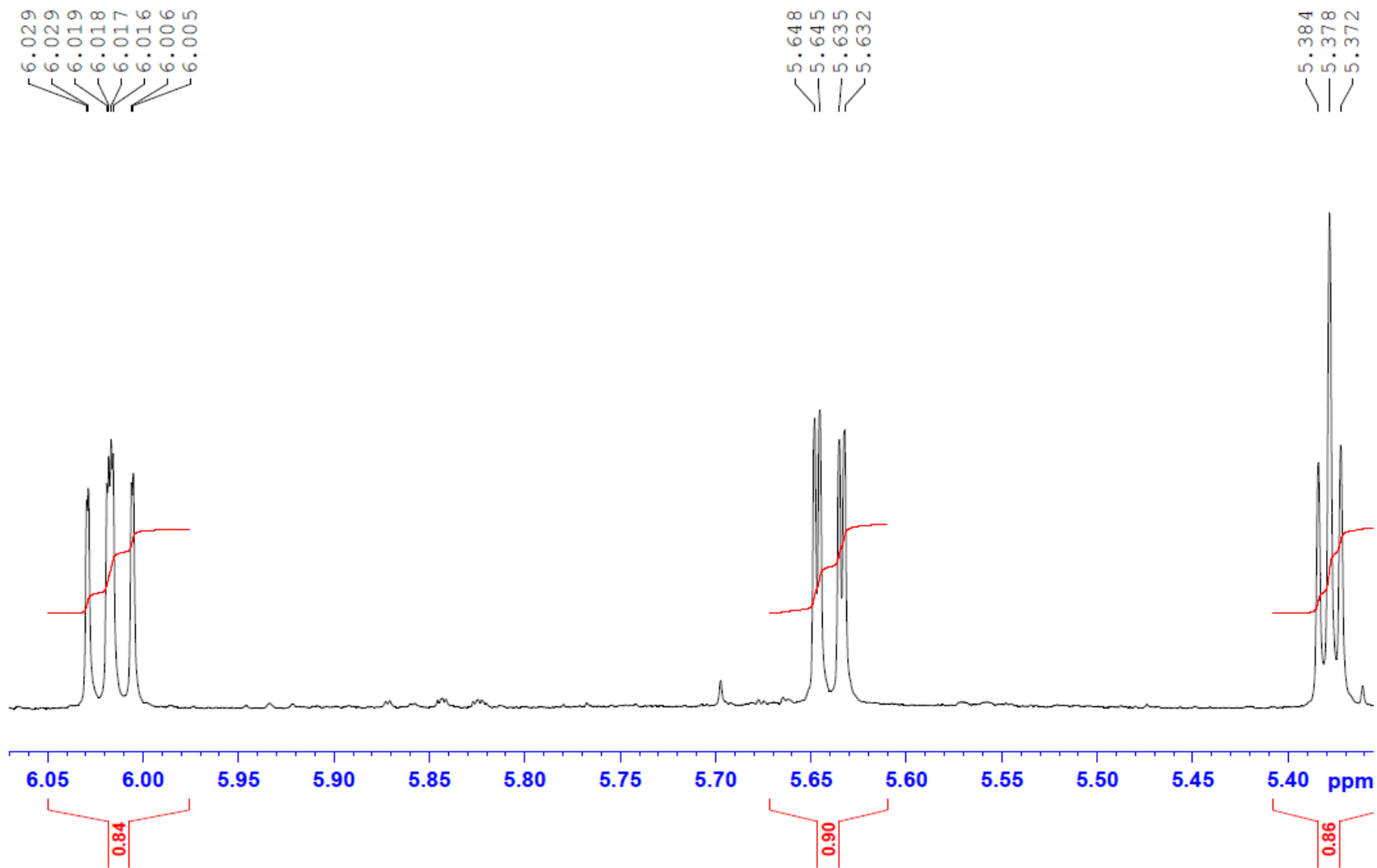


Figure S3b: ¹H NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

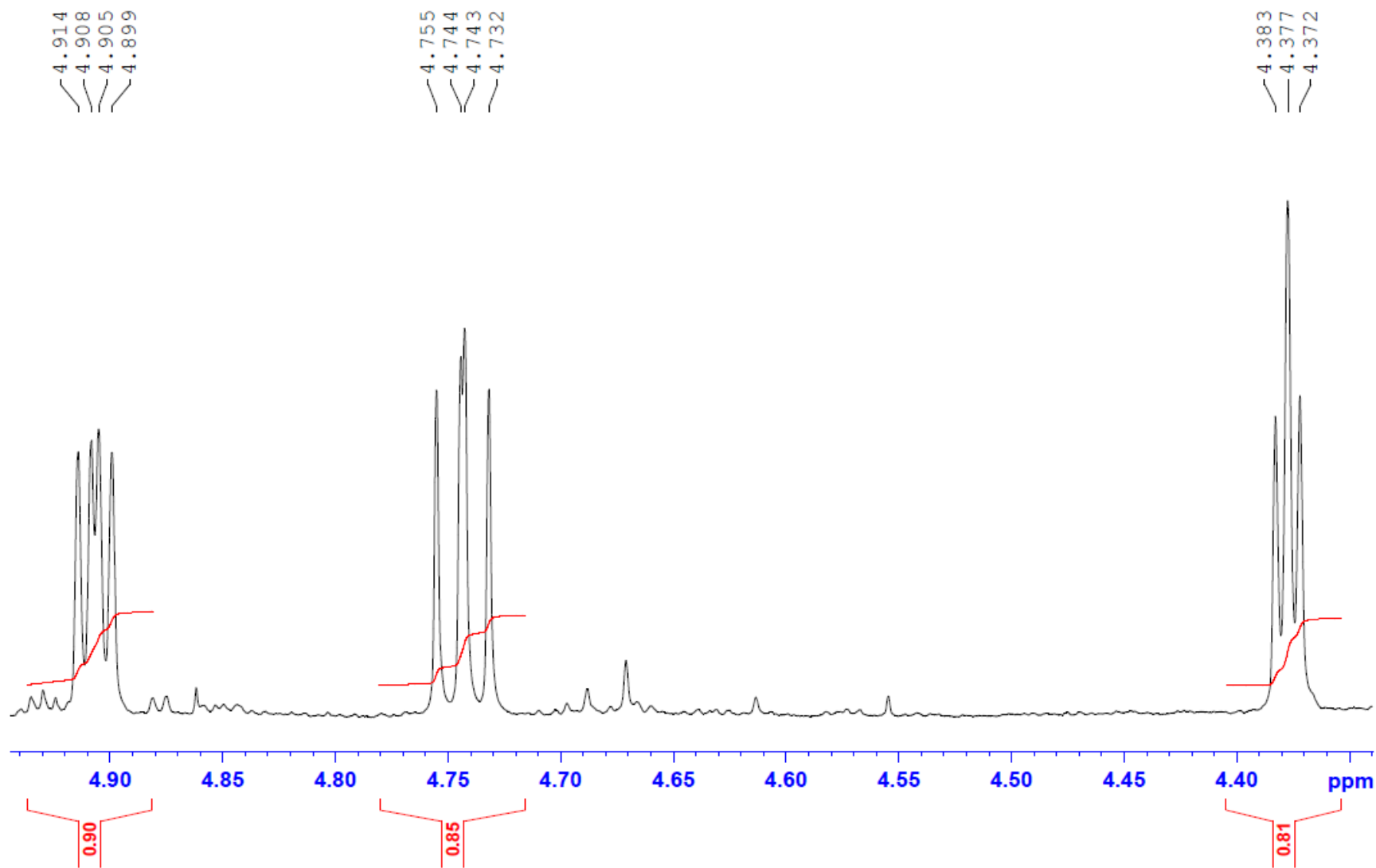


Figure S3c: ^1H NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

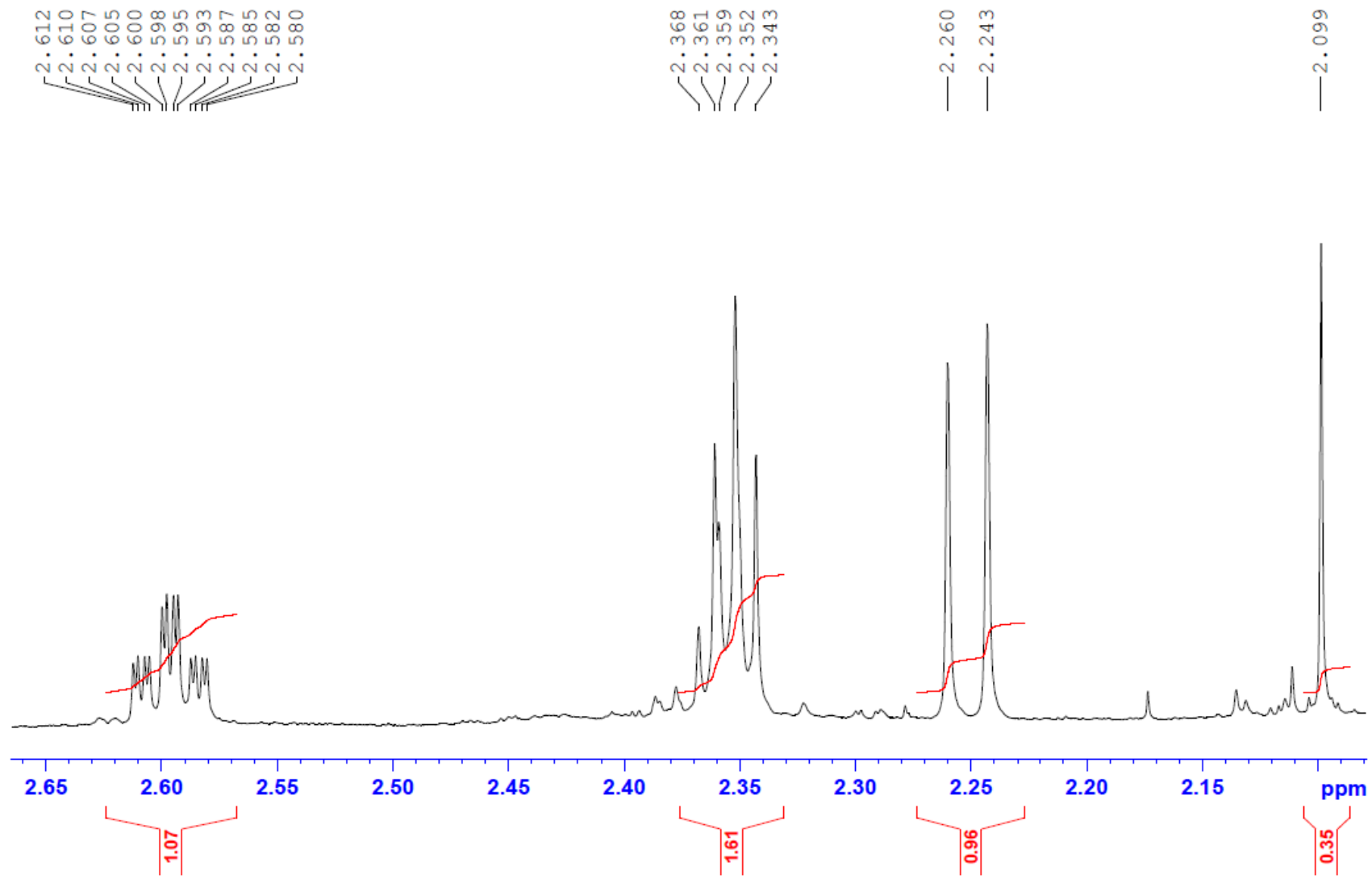
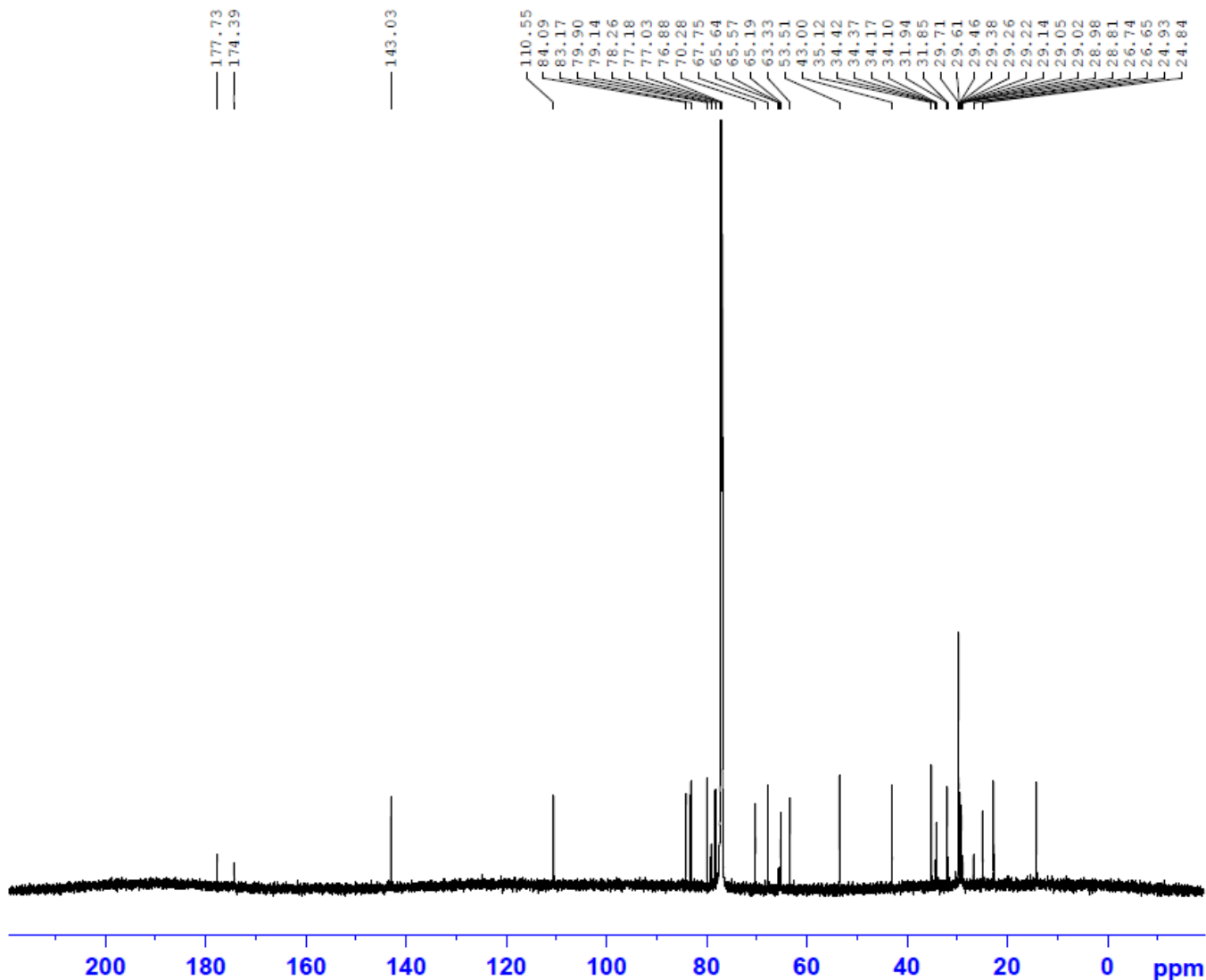


Figure S3d: ¹H NMR of compound 3

Dr.Walied

Sample : NL-244-4

CDCL3



Current Data Parameters
NAME WALIED NL-244-4 16-08-2017
EXNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170317
Time_ 20.35
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 4
SWH 51020.406 Hz
FIDRES 0.778510 Hz
AQ 0.6422528 sec
RG 186.93
DW 9.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL #1 =====
SFO1 213.7917636 MHz
NUC1 13C
P1 12.00 usec
PLW1 130.00000000 W

===== CHANNEL #2 =====
SFO2 850.1534006 MHz
NUC2 1H
PCPD2 waltz16
PCPD2 80.00 usec
PLW2 13.80000019 W
PLW12 0.13800000 W
PLW13 0.08892000 W

F2 - Processing parameters
SI 32768
SF 213.7703875 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
FC 2.00

Figure S3e: ¹³C NMR of compound 3

Dr. Walled
Sample : NL-244-4 CDCL3

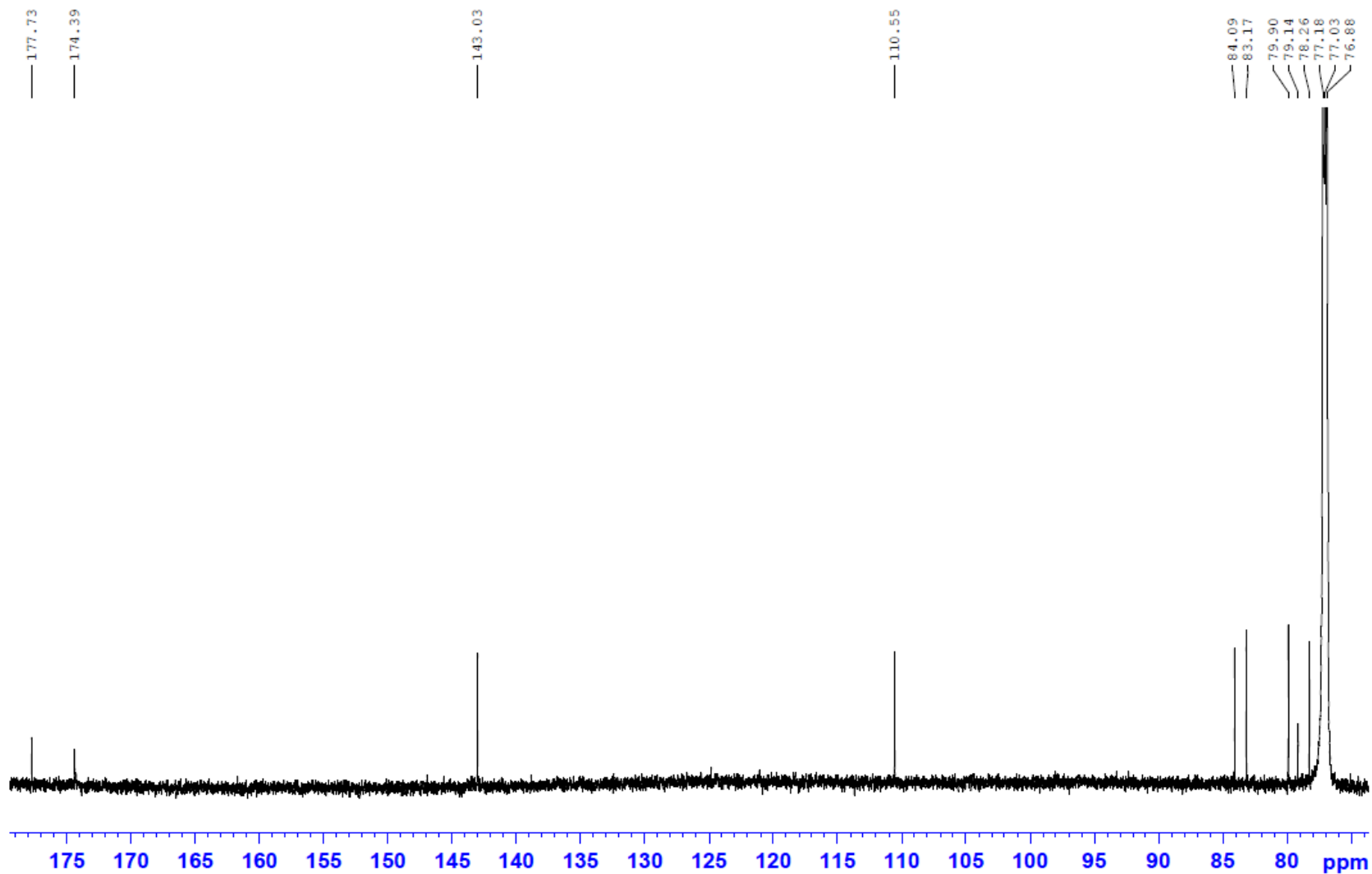


Figure S3f: ^{13}C NMR of compound 3

Dr. Walied

Sample : NL-244-4 CDCL3

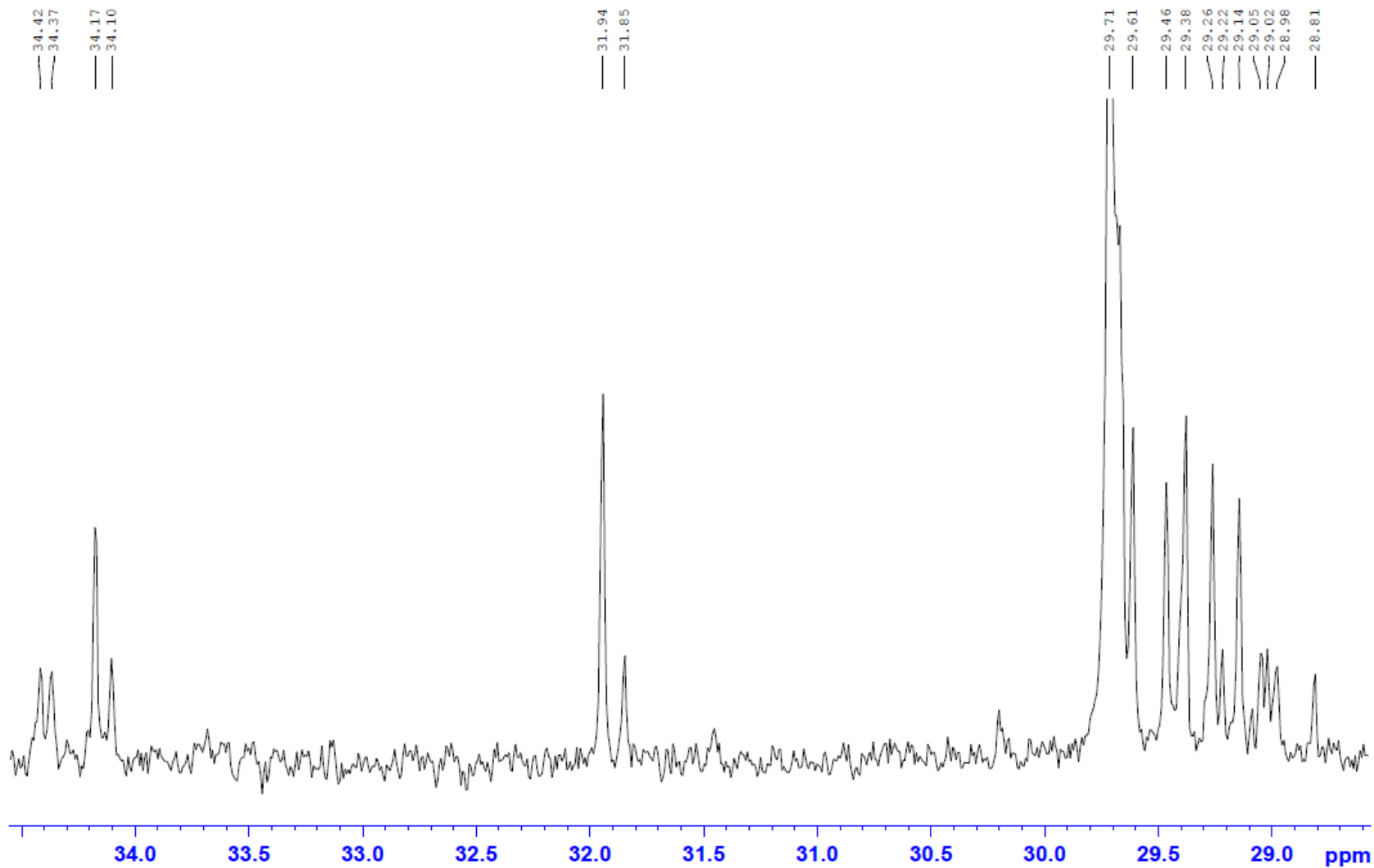
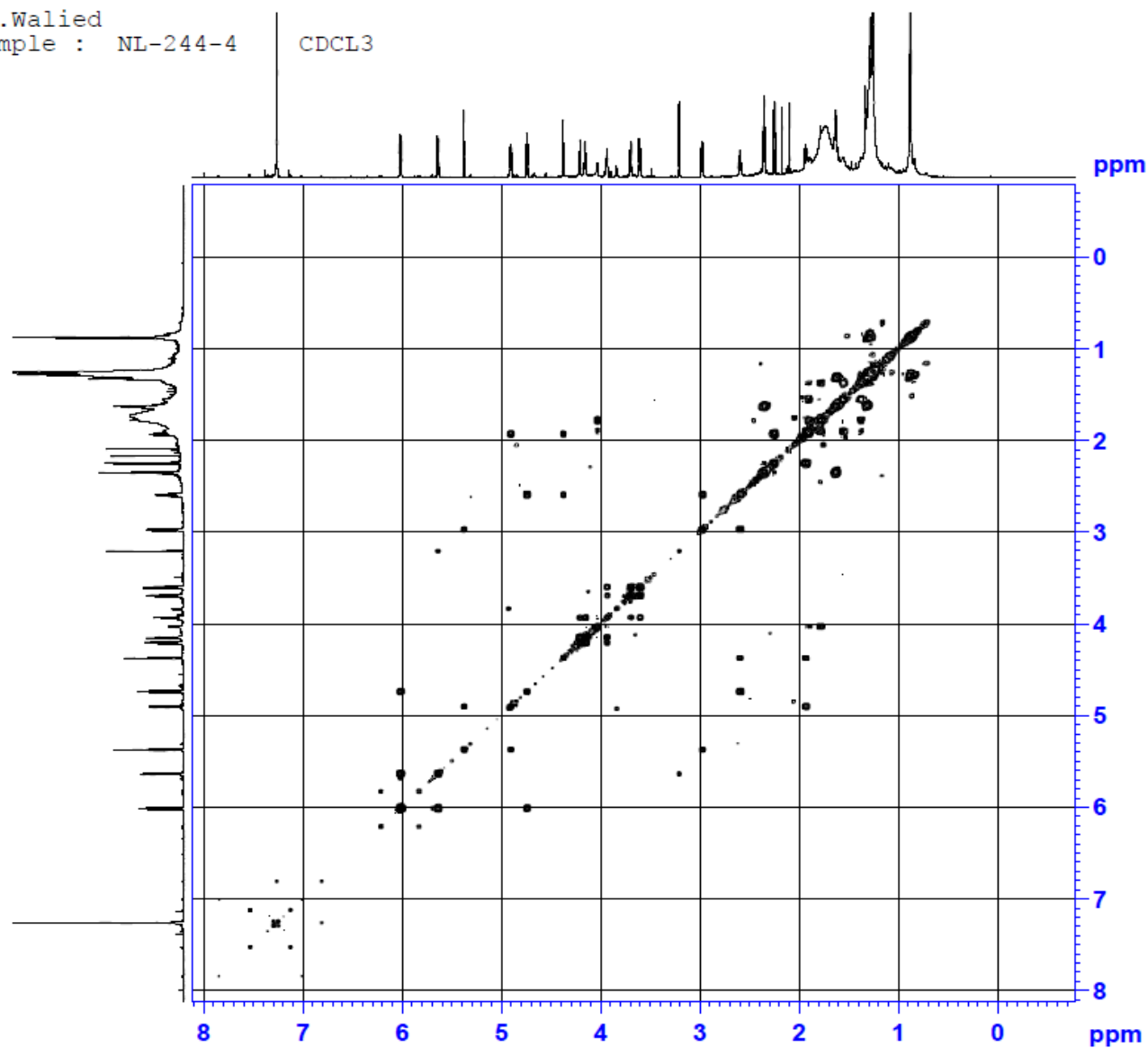


Figure S3g: ¹³C NMR of compound 3

Dr.Walied
Sample : NL-244-4 CDCL3



Current Data Parameters
NAME WALIED NL-244-4 21-02-2017
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170221
Time 14.57
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG cosygpmfcr
TD 2048
SOLVENT CDCL3
NS 16
DS 8
SWH 7575.758 Hz
FIDRES 3.699100 Hz
AQ 0.1351680 sec
RG 126.92
DW 66.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.00000200 sec
D1 1.95494401 sec
D13 0.00000400 sec
D16 0.00020000 sec
INO 0.00013200 sec

===== CHANNEL f1 =====
SF01 850.1331342 MHz
NUCL1 1H
P1 8.00 usec
PLW1 15.30000019 W

===== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GP21 16.00 %
GP22 12.00 %
GP23 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SF01 850.1331 MHz
FIDRES 59.185608 Hz
SF 8.911 ppm
PRMODE QF

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

F1 - Processing parameters
SI 1024
MC2 QF
SF 850.1500200 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

Figure S3h: COSY NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

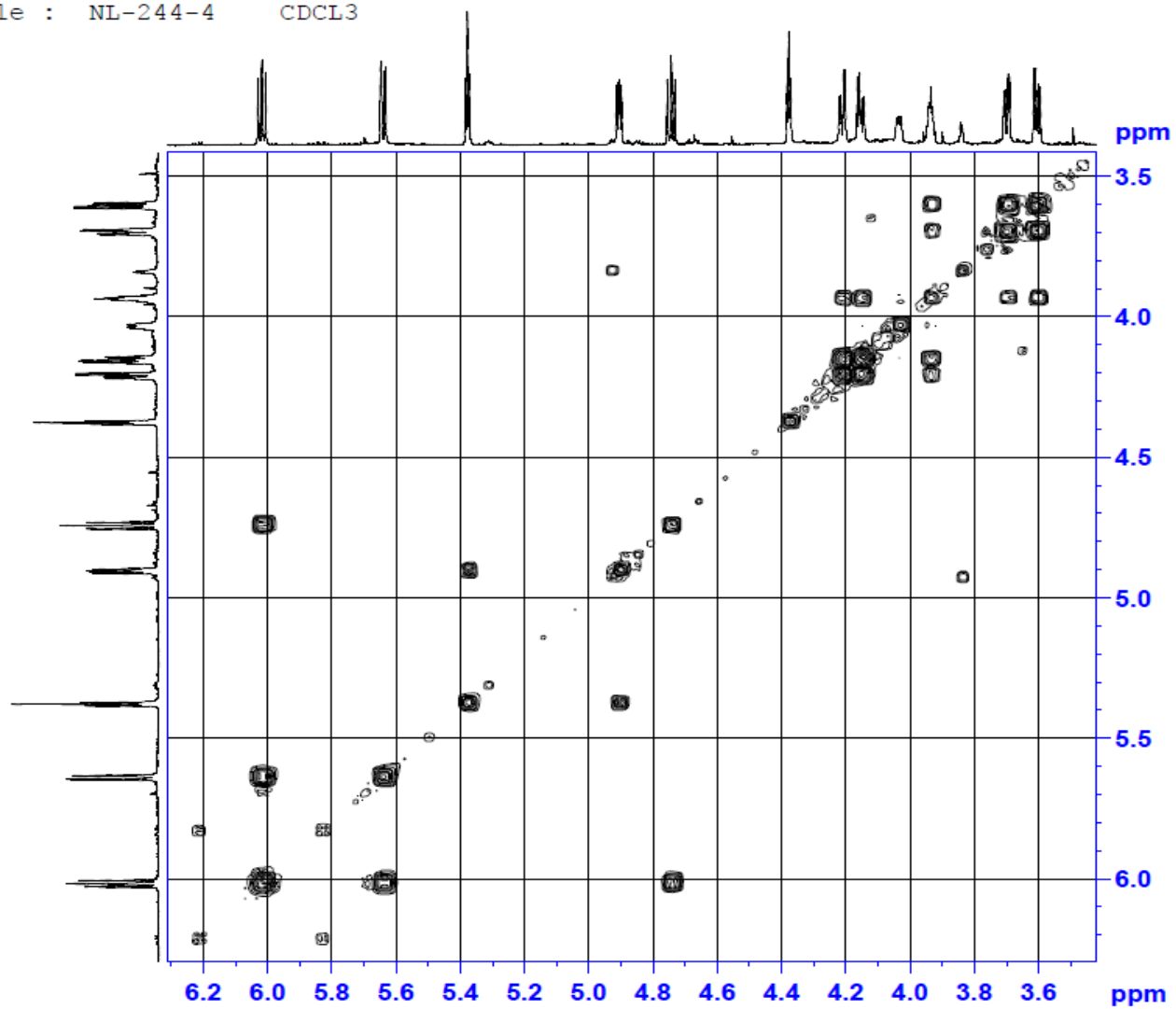


Figure S3i: COSY NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

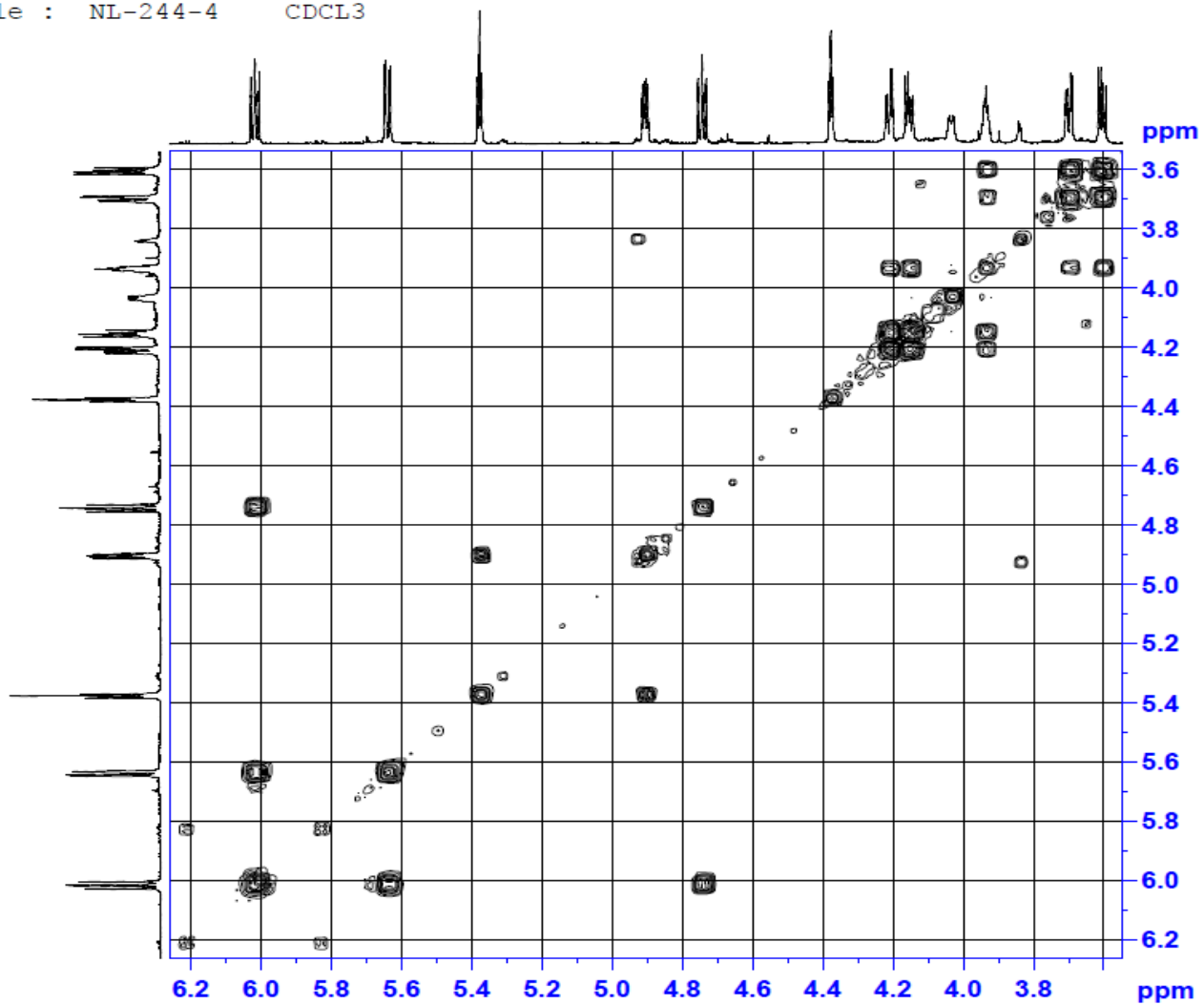


Figure S3j: COSY NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

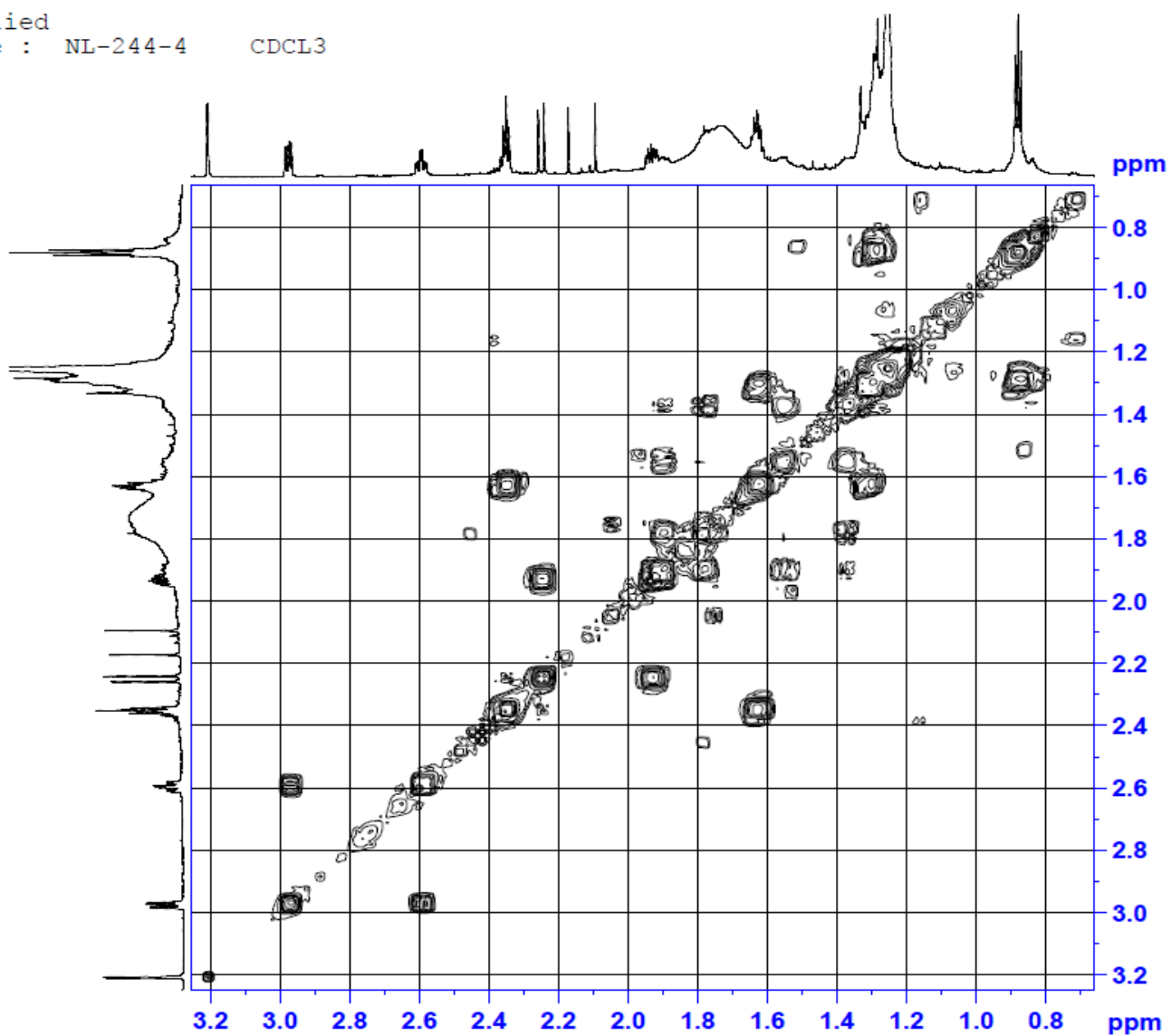


Figure S3k: COSY NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

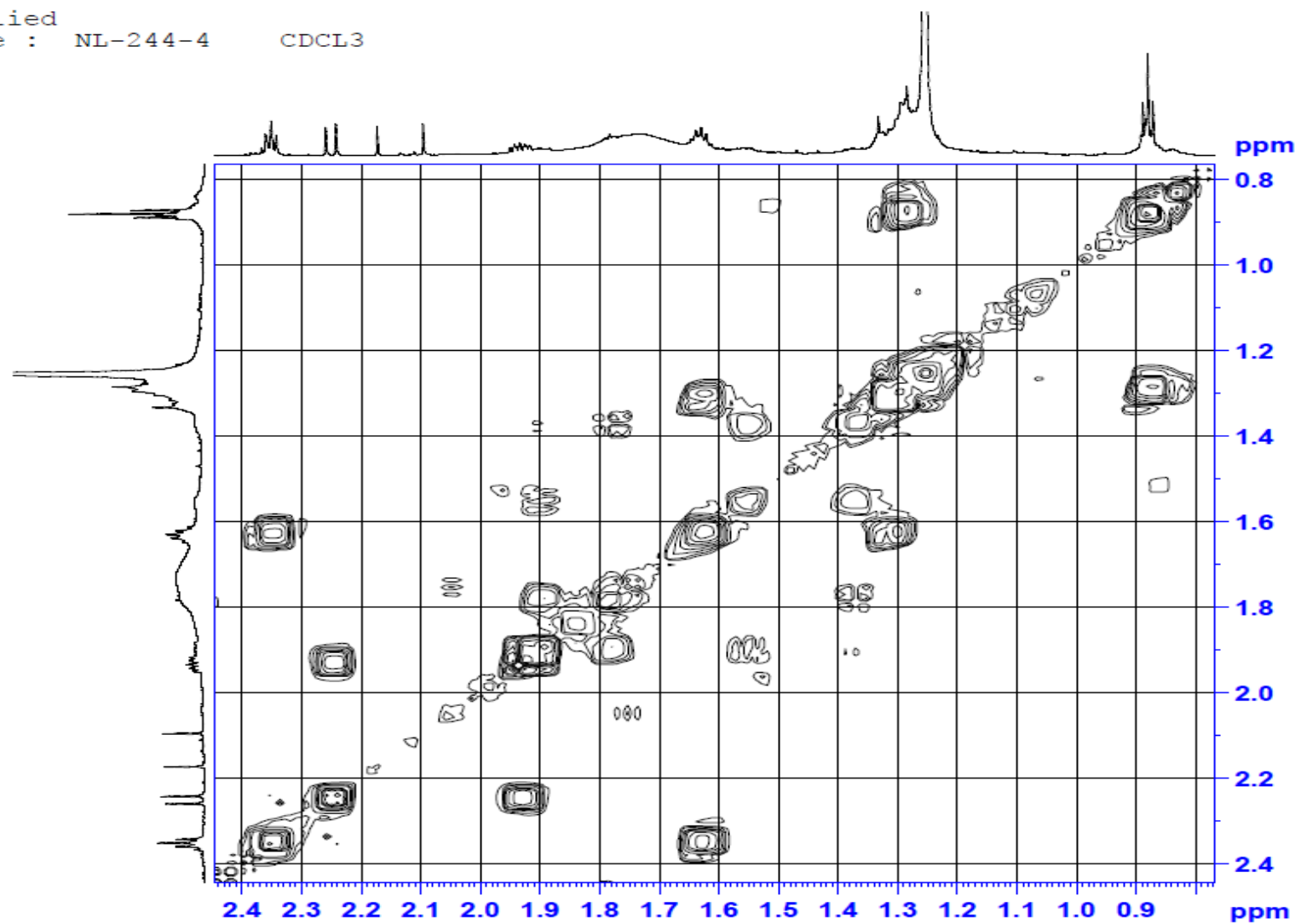
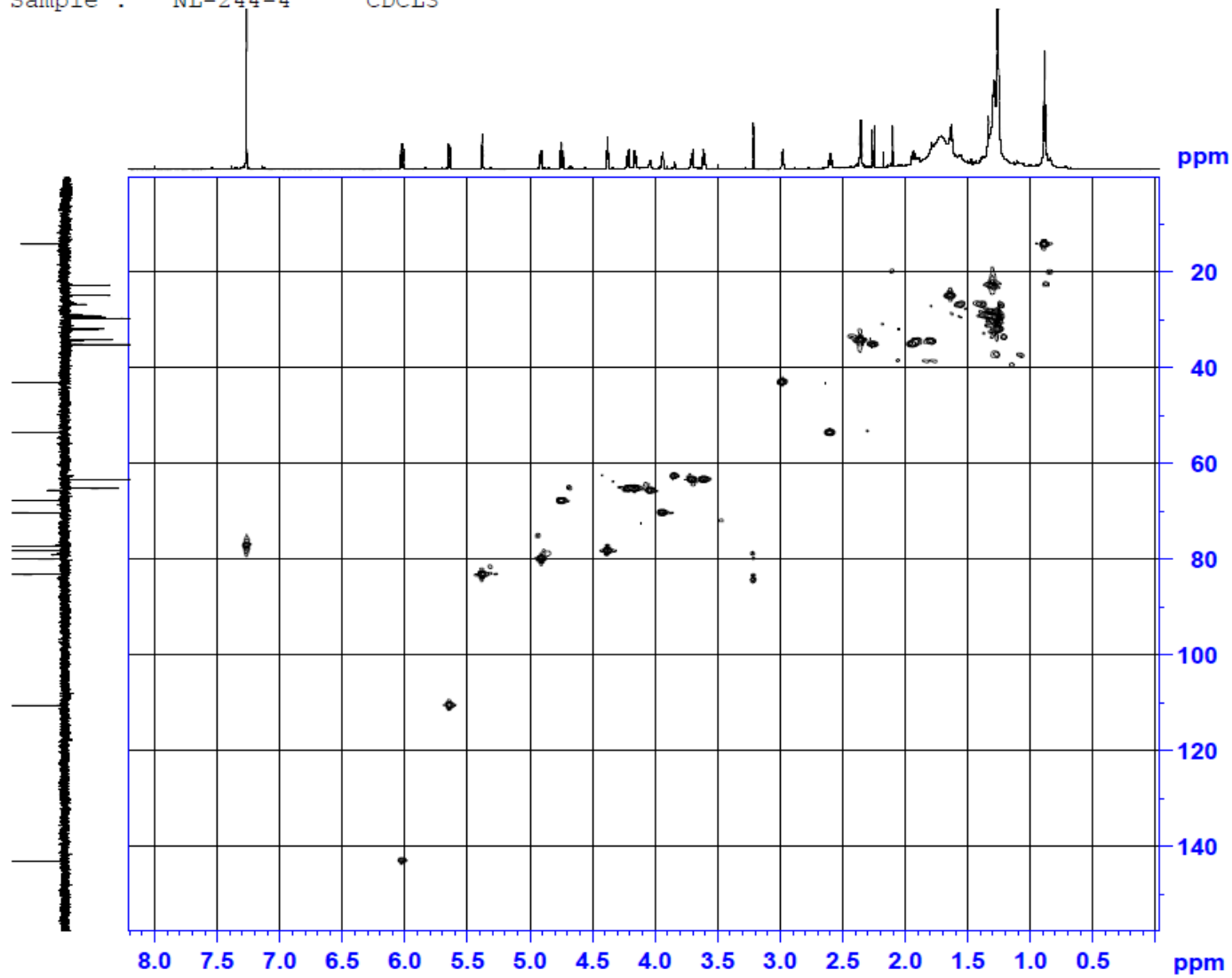


Figure S31: COSY NMR of compound 3

Dr. Walied

Sample : NL-244-4 CDCL3



```
Current Data Parameters
NAME      WALIED  NL-244-4   16-03-2017
EXPNO    24
PROCNO   1

F2 - Acquisition Parameters
Date_    20170318
Time     3.10
INSTRUM  spect
PROBHD   5 mm CPQCI 1H-
PULPROG  hsqcetdtp
TD        1024
SOLVENT  CDCL3
NS        32
DS        16
SWH       8064.516 Hz
FIDRES   7.875504 Hz
AQ        0.0634860 sec
RG        186.93
LW        62.000 usec
DE        10.00 usec
TE        298.0 K
CHST2    145.0000000
DO        0.0000300 sec
D1        1.48293304 sec
D4        0.00172414 sec
D11       0.03000000 sec
D13       0.00000400 sec
D16       0.00020000 sec
D21       0.00345000 sec
TM0       0.00001410 sec
ZGPGTNS

----- CHANNEL f1 -----
SFO1     850.1529628 MHz
NUC1      1H
P1        8.00 usec
P2        16.00 usec
P2S       0 usec
PLW1     15.30000019 W

----- CHANNEL f2 -----
SFO2     213.7863316 MHz
NUC2      13C
CPDPRG2  garp
P3        12.00 usec
P4        24.00 usec
PCPD2    45.00 usec
PLW2     130.00000000 W
PLW12    9.24440002 W

----- GRADIENT CHANNEL -----
GPMAM[1] SMSQ10.100
GPMAM[2] SMSQ10.100
GPP1     80.00 %
GPP2     20.10 %
P16      1000.00 usec

F1 - Acquisition parameters
TD        256
SFO1     213.7863 MHz
FIDRES   138.519501 Hz
SW        165.871 ppm
FMODE    Echo-Antiecho

F2 - Processing parameters
SI        1024
SF        850.1500200 MHz
WDW       QSINE
SSB       2
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
WC2      echo-antiecho
SF        213.7703875 MHz
WDW       QSINE
SSB       2
LB        0 Hz
GB        0
```

Figure S3m: HSQC NMR of compound 3

Dr. Walied

Sample : NL-244-4

CDCL₃

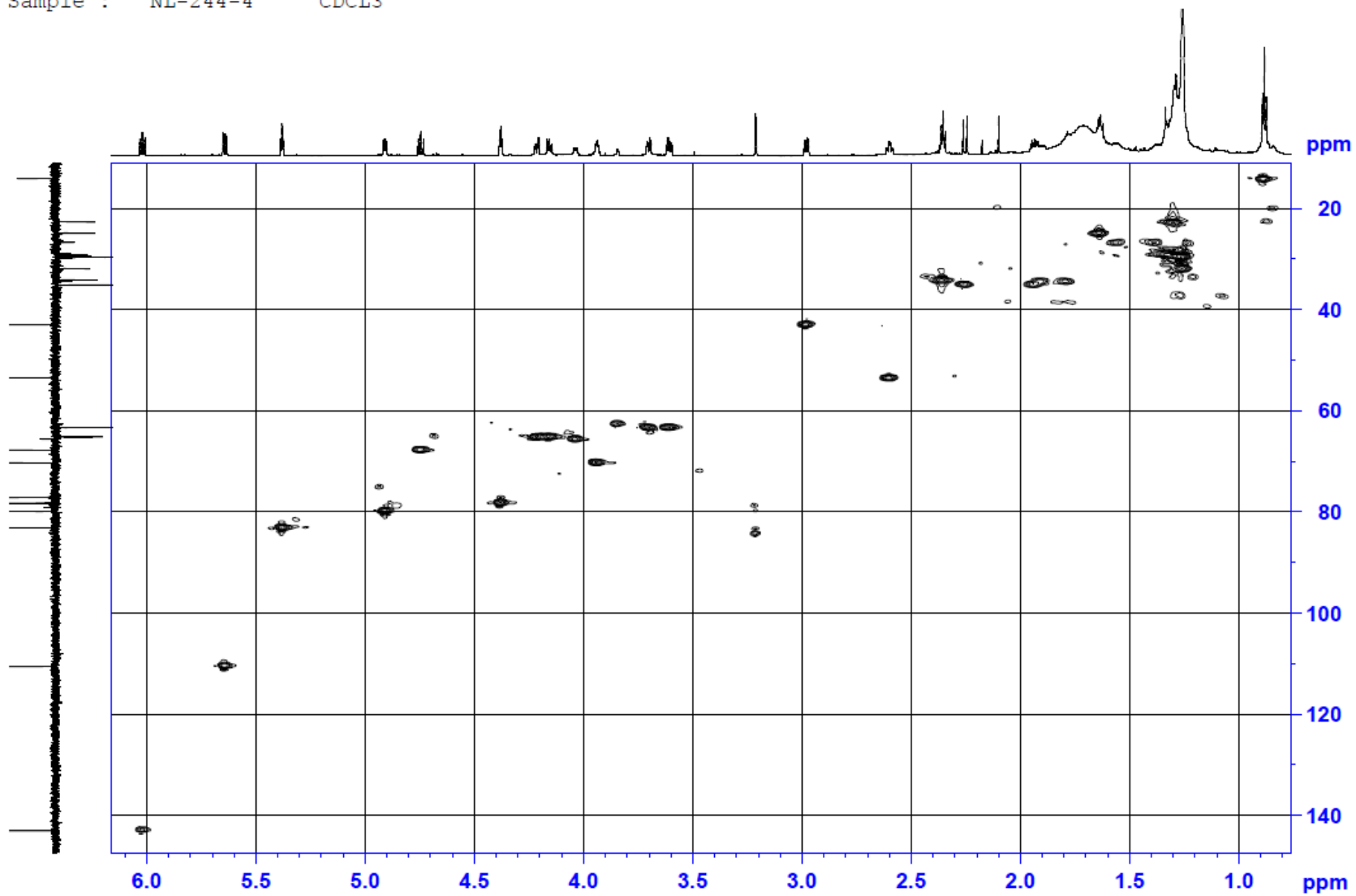
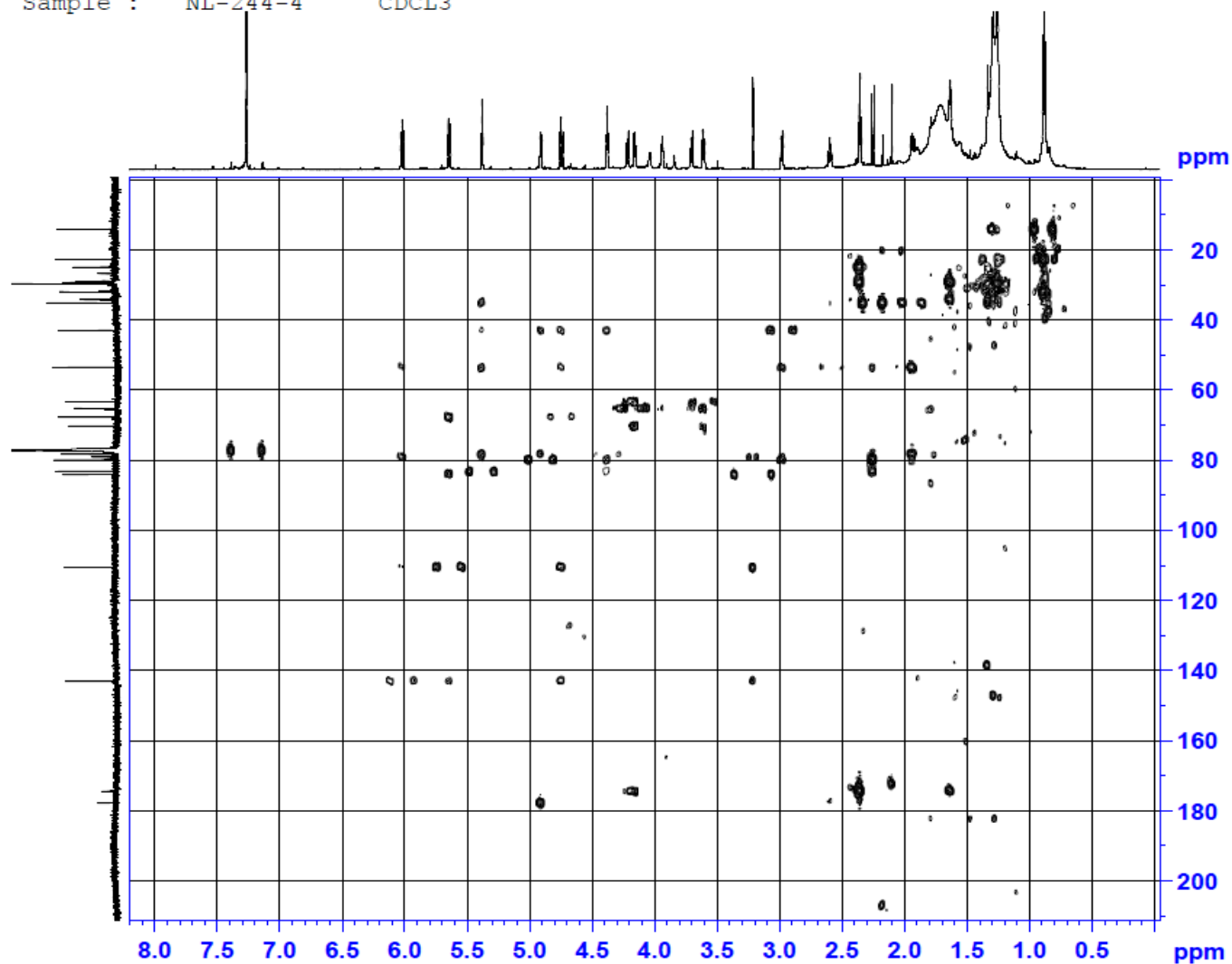


Figure S3n: HSQC NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3



Current Data Parameters
NAME WALIED NL-244-4 16-03-2017
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170318
Time_ 6.48
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
FULPROG hmcgppndgr
TD 4096
SOLVENT CDCL3
NS 128
DS 16
SWH 8064.516 Hz
FIDRES 1.968876 Hz
AQ 0.2539520 sec
RG 186.93
DN 62.000 usec
DE 10.00 usec
TE 298.0 K
CNST13 8.0000000
DO 0.00000300 sec
D1 1.42709100 sec
D5 0.06250000 sec
D16 0.00020000 sec
IN0 0.0001050 sec

===== CHANNEL f1 =====
SFO1 850.1529628 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 =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 213.7917 MHz
FIDRES 372.023804 Hz
SW 222.736 ppm
FMODE QF

F2 - Processing parameters
SI 1024
SF 850.1500200 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 S3o: HMBC NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

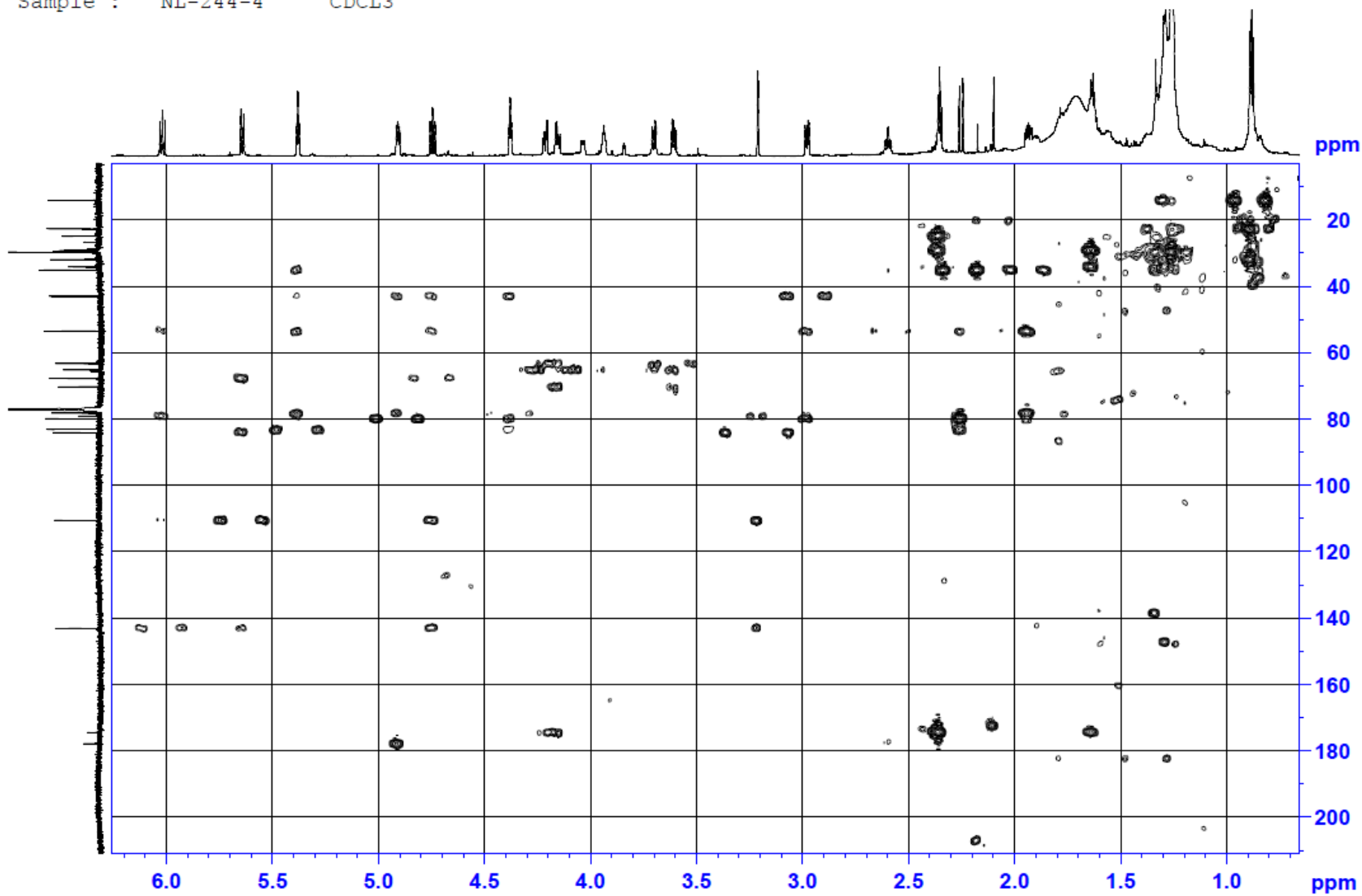


Figure S3p: HMBC NMR of compound 3

Dr.Walied
Sample : NL-244-4 CDCL3

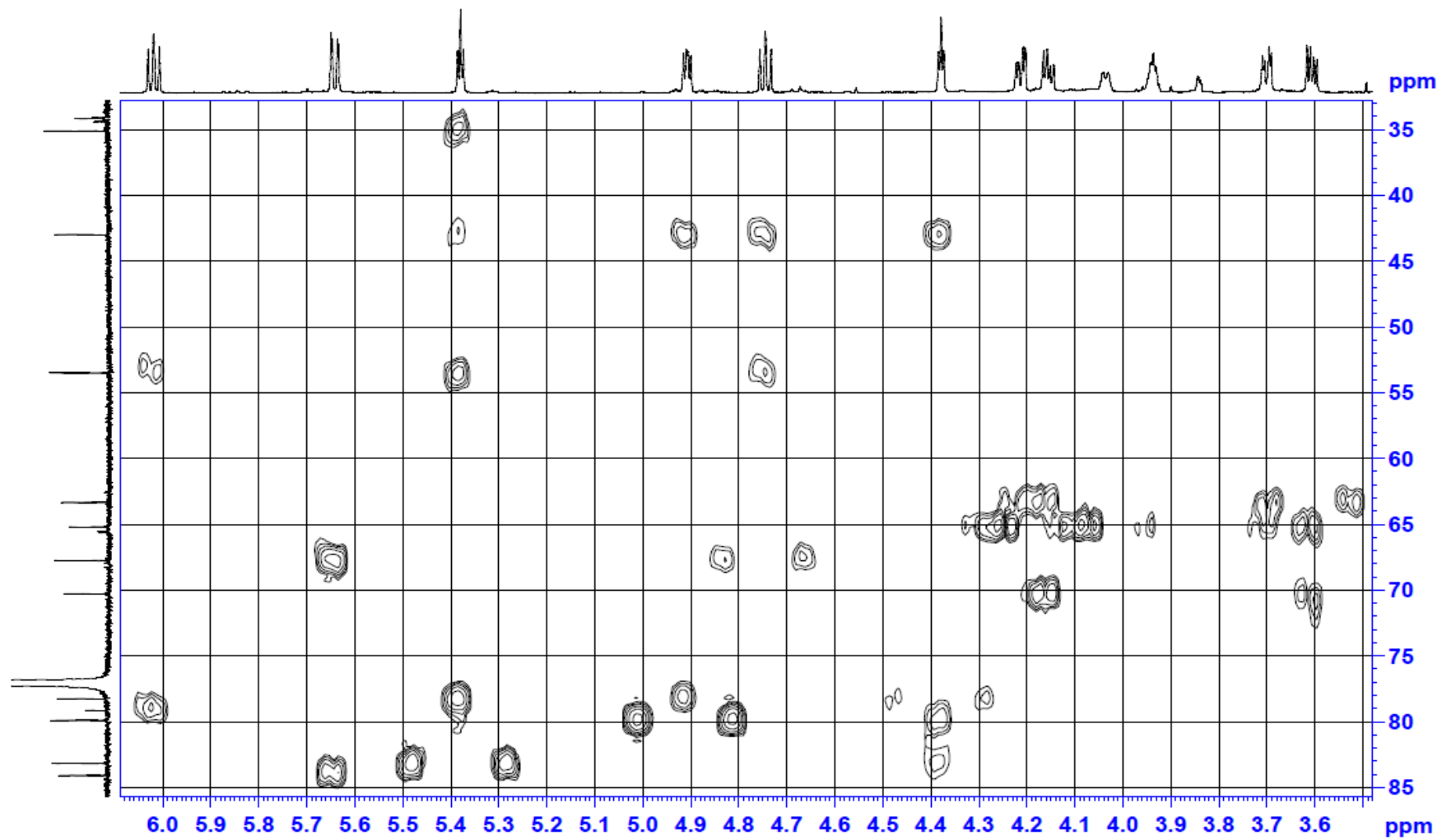


Figure S3q: HMBC NMR of compound 3

Dr. Walied
Sample : NL-244-4 CDCL3

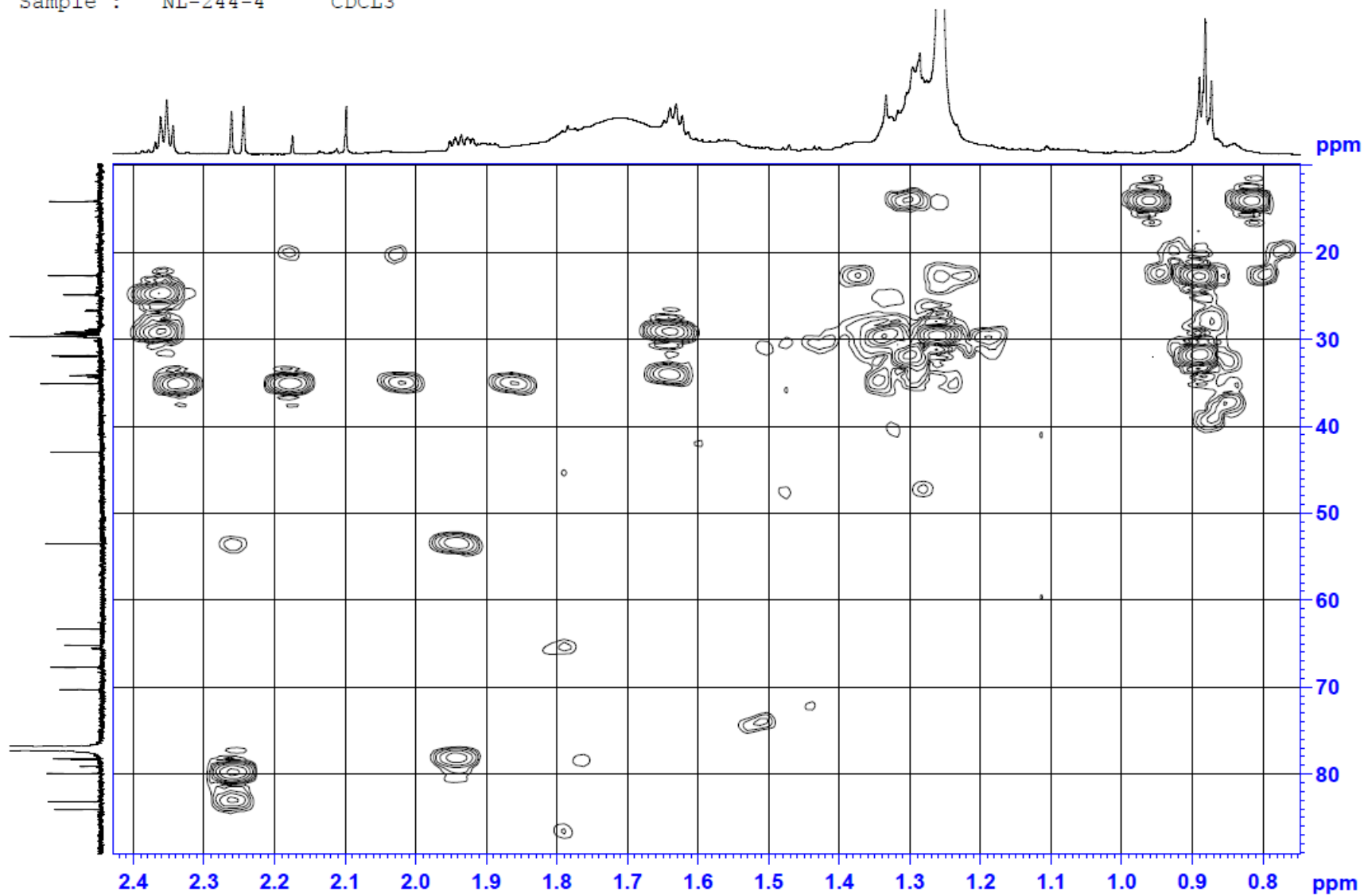


Figure S3r: HMBC NMR of compound 3

Dr.Walied

Sample : NL-244-4

CDCL3

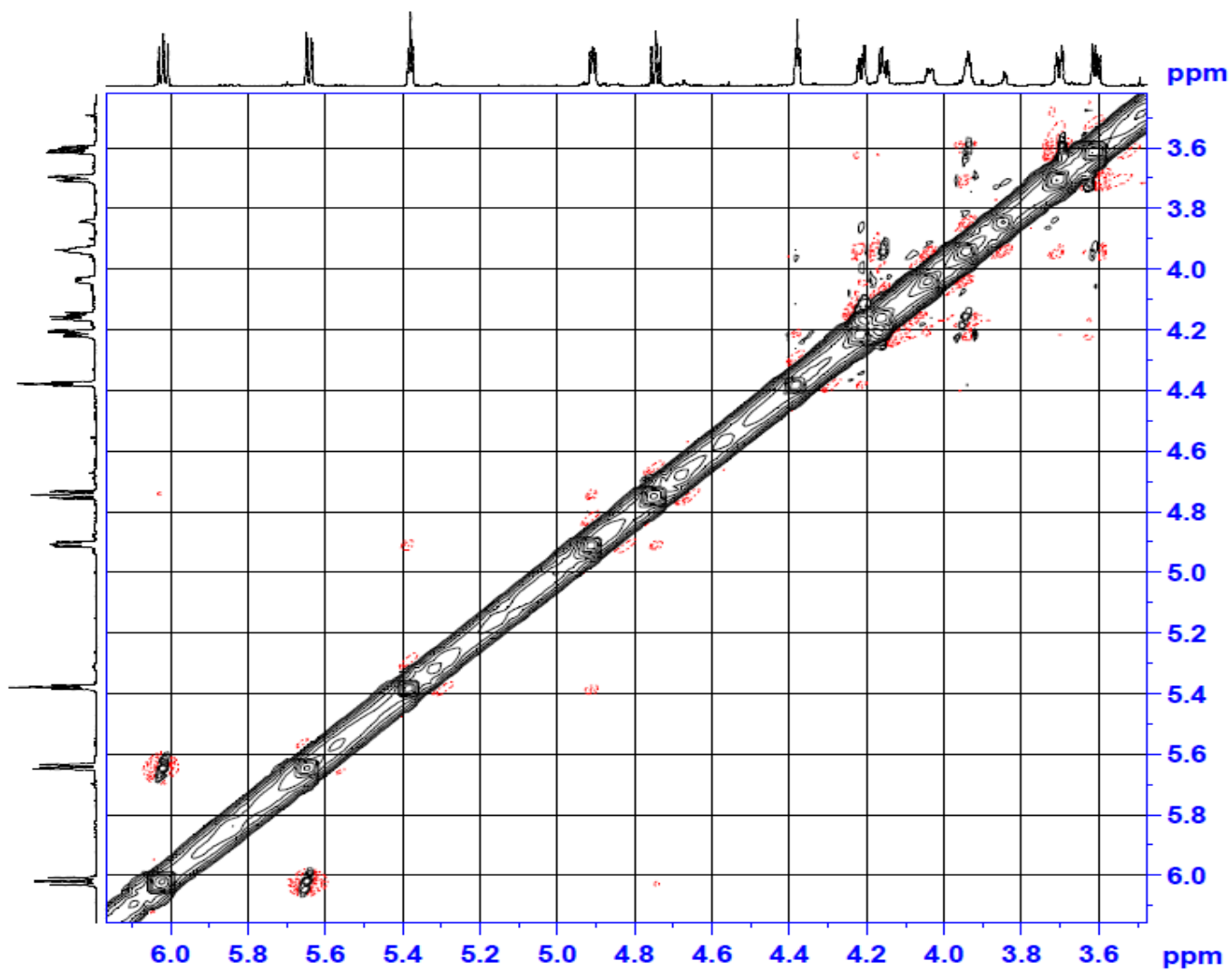


Figure S3s: NOSY NMR of compound 3

Dr.Walied
Sample : NL-244-4 CDCL3

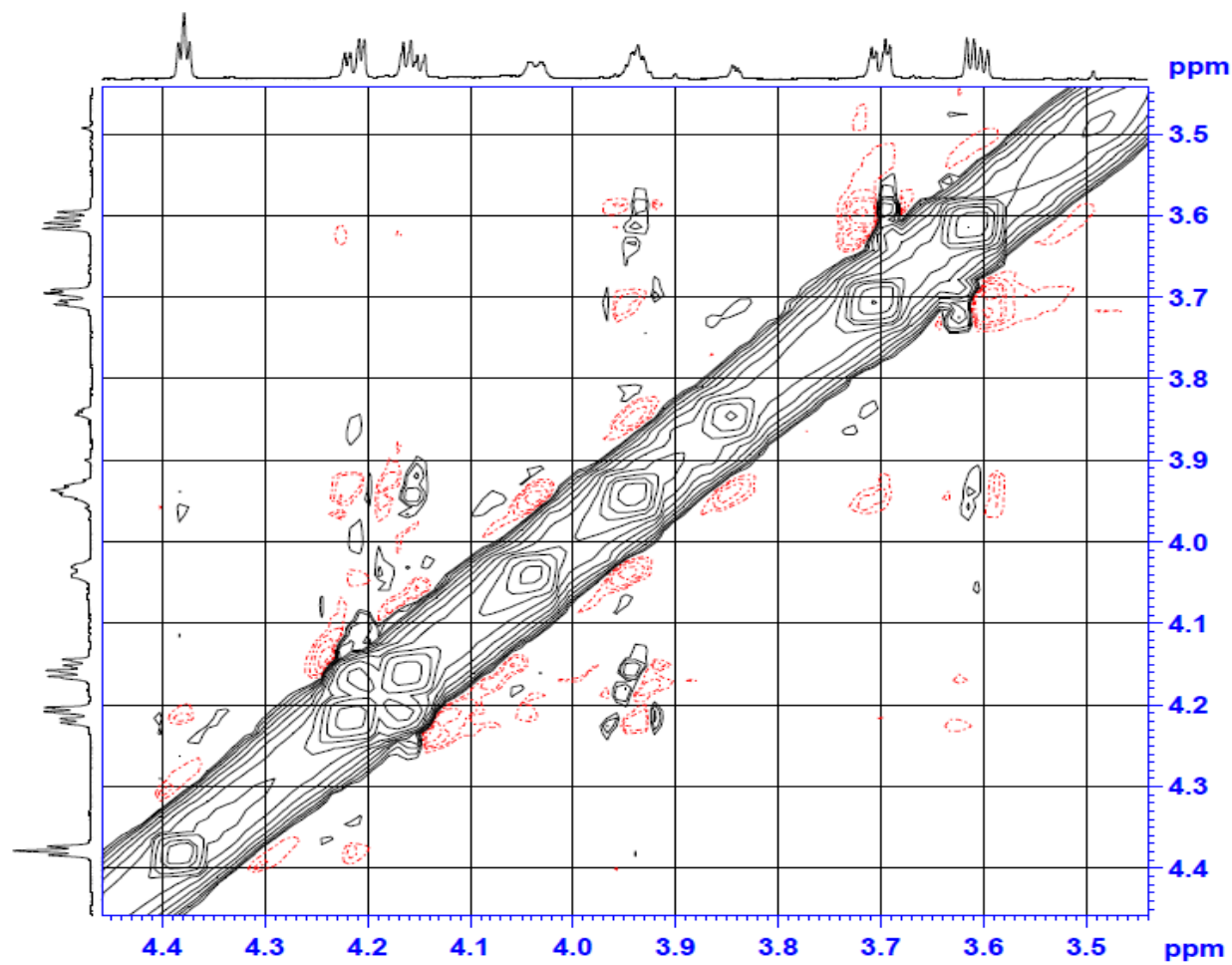


Figure S3t: NOSY NMR of compound 3