

Supplementary Material

Reduction Chemistry of Natural Pyrethrins and Preliminary Insecticidal Activity of Reduced Pyrethrins

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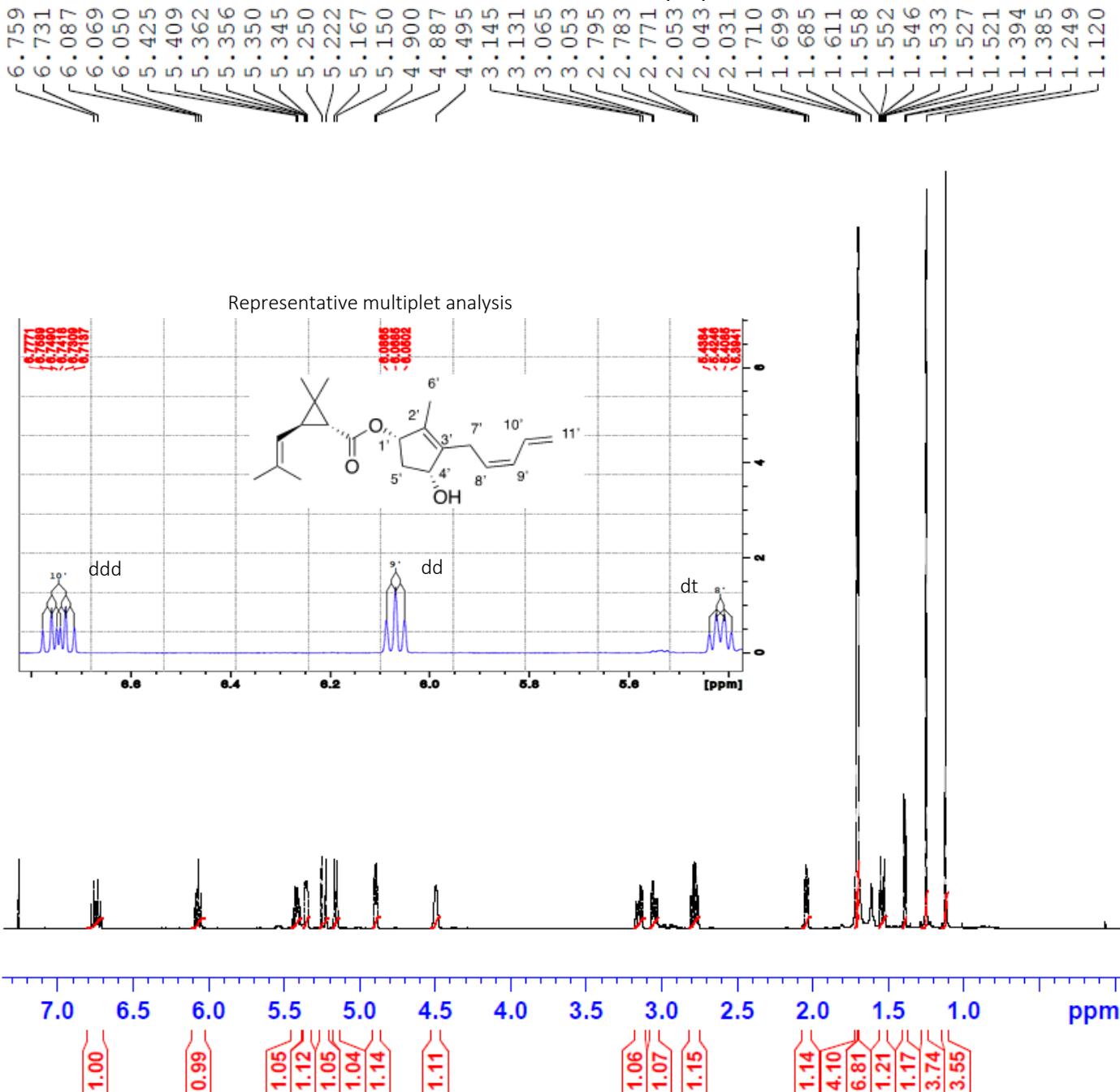
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4R-(4a) NMR Characterisation



Current Data Parameters
 NAME 20180914 Pyrethrin I Alcohol
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

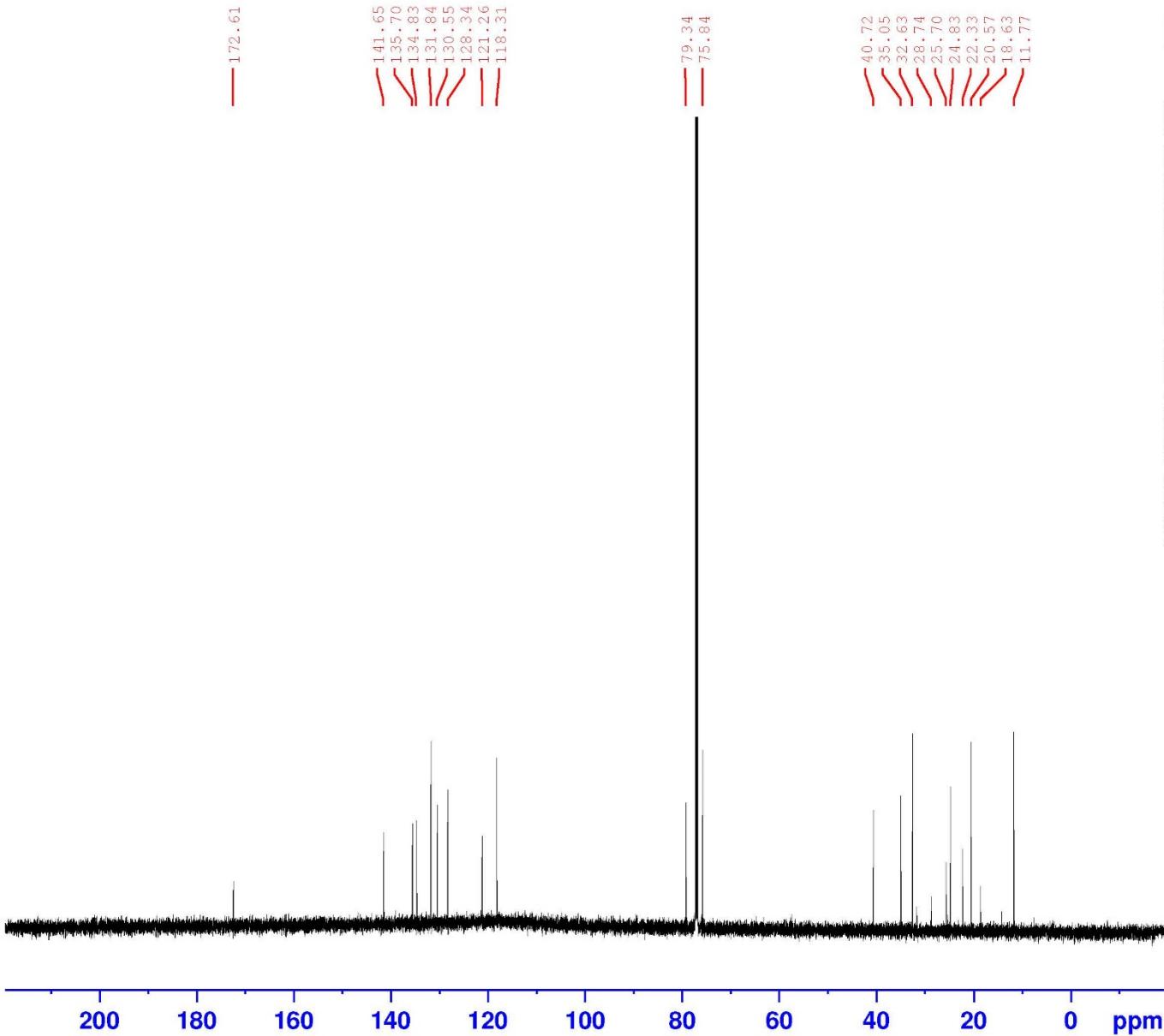
Date 20180914
 Time 18.43
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183389 Hz
 AQ 2.7262976 sec
 RG 80.6
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====

SFO1 600.1337060 MHz
 NUC1 1H
 P1 8.40 usec
 PLW1 12.55000019 W

F2 - Processing parameters

SI 65536
 SF 600.1200266 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME: 20190118_Pyrothrin_I_Alcohol_Major_dia stereomer
EXPNO: 12
PROCNO: 1

P2 - Acquisition Parameters

Date: 20190118
Time: 23:30
DURATION: 30 sec
PROBHD: 5 mm PABBY 1H/
PULPROG: zgpp30
TD: 65536
SOLVENT: CCl4
NS: 2048
DS: 2
SWH: 36037.691 Hz
ETRIPES: 0.51013 Hz
AQ: 0.9087659 sec
RG: 1620
DW: 13.867 usec
DE: 6.5 usec
TB: 298.2 K
D1: 2.0000000 sec
D11: 0.0300000 sec
TD0: 1

===== CHANNEL f1 =====

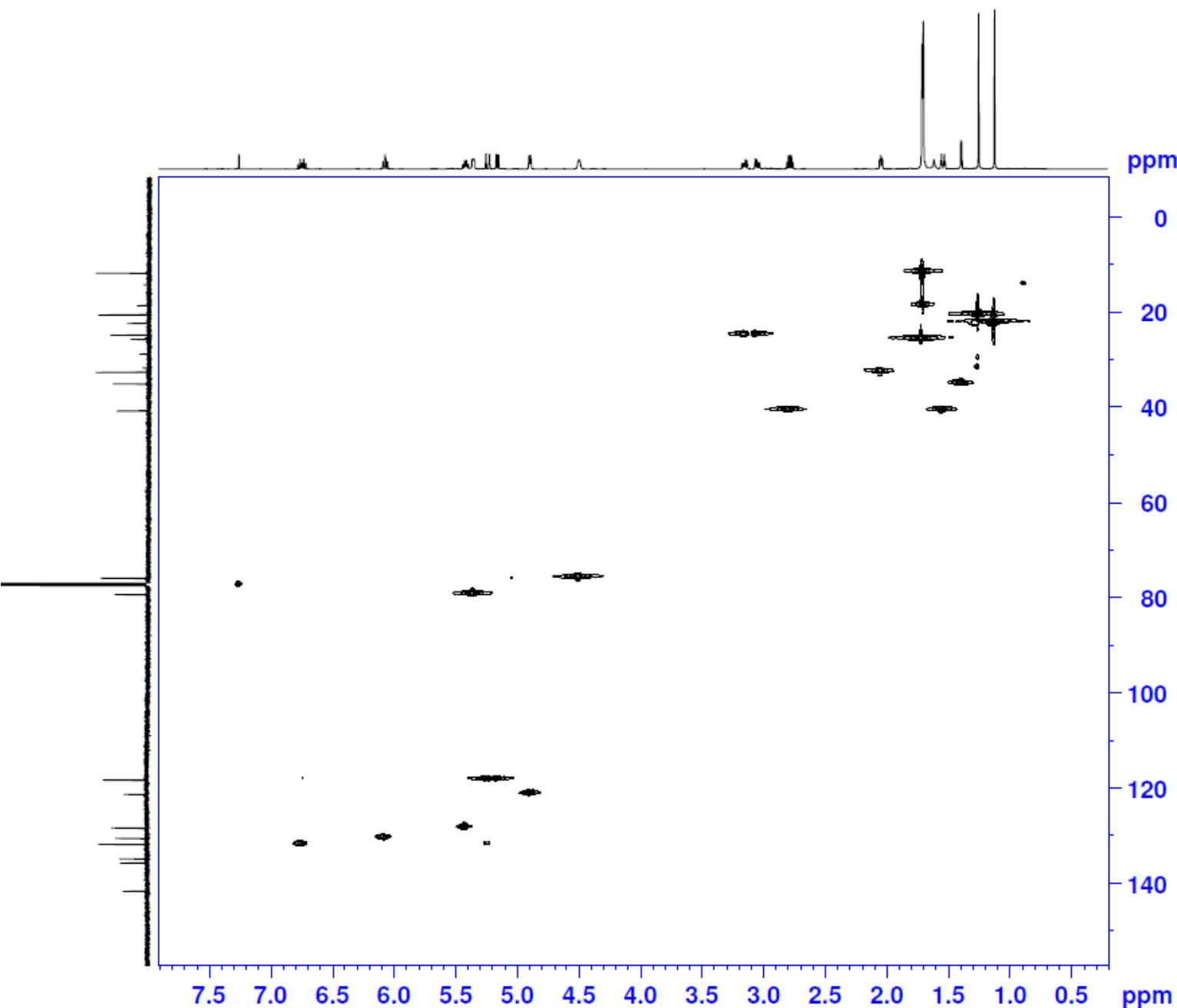
SFO1: 150.9178988 MHz
NUC1: 1H
P1: 16.00 usec
PLW1: 110.76000314 W

===== CHANNEL f2 =====

SFO2: 600.1324005 MHz
NUC2: 1H
CPDPFG12: waltz16
RF1: 1.00 usec
PLW2: 12.55000019 W
PLW12: 0.18072000 W
PLW13: 0.08855400 W

P2 - Processing parameters

S1: 32768
SF: 150.9027905 MHz
NMW: no
SSB: 0
LB: 0 Hz
GB: 0
PC: 1.40





Current Data Parameters
 NAME 20190122 Pyrethrin I Alcohol MAJOR
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20190122
 Time 9.11
 INSTRUM spect
 PROBHD 5 mm PARBI 1H/
 PULPROG noesypph
 TD 4096
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 5411.255 Hz
 FIDRES 1.321107 Hz
 AQ 0.3784704 sec
 RG 203
 DW 92,400 ussec
 DE 6.50 ussec
 TE 298.2 K
 D0 0.00008170 sec
 D1 3.0000000 sec
 D8 0.60000002 sec
 D16 0.00020000 sec
 IN0 0.00018480 sec

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 NUC1 1H
 P1 8.40 ussec
 P2 16.80 ussec
 PIW1 12.55000019 W

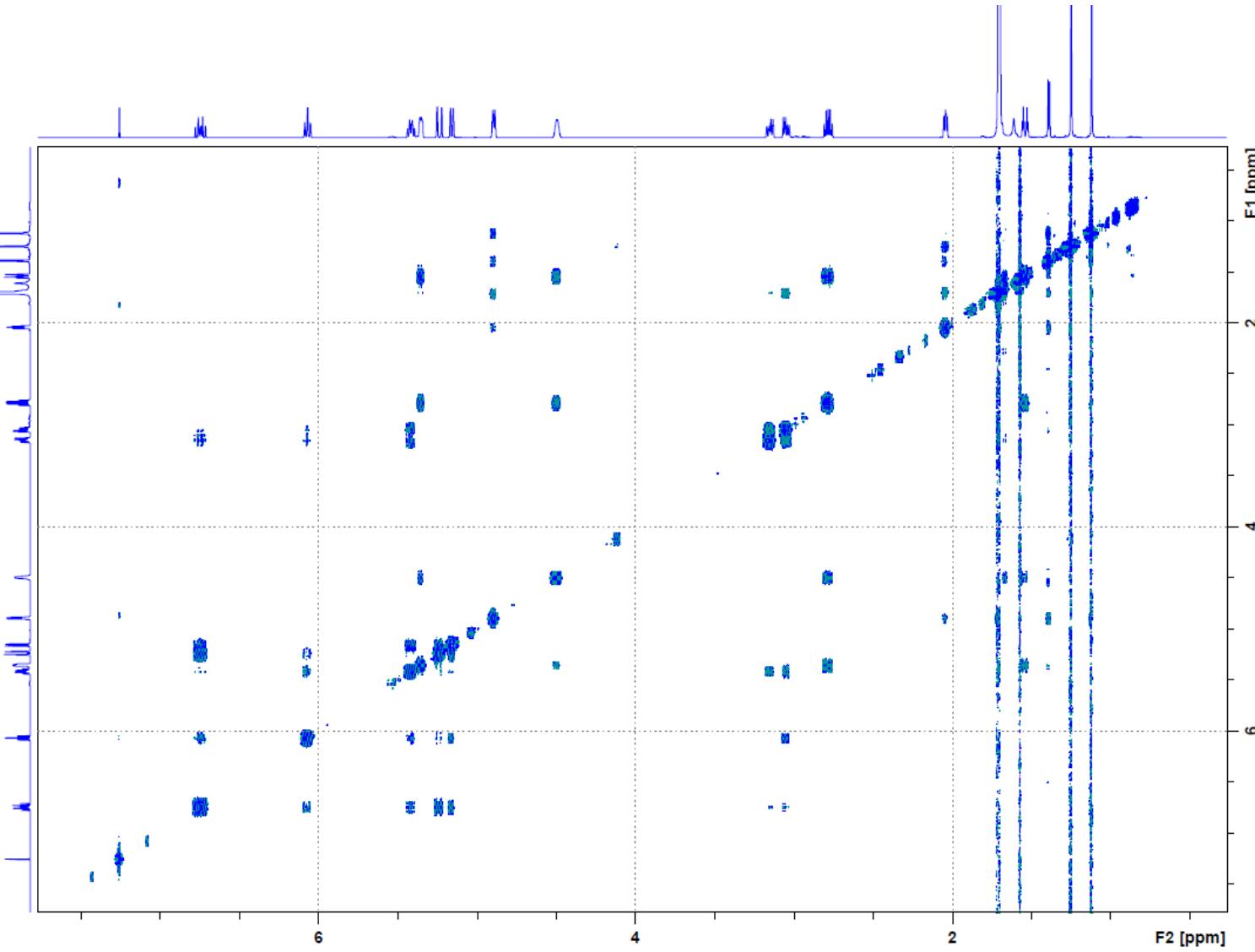
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 GPZ1 40.00 %
 P16 1000.00 ussec

F1 - Acquisition parameters

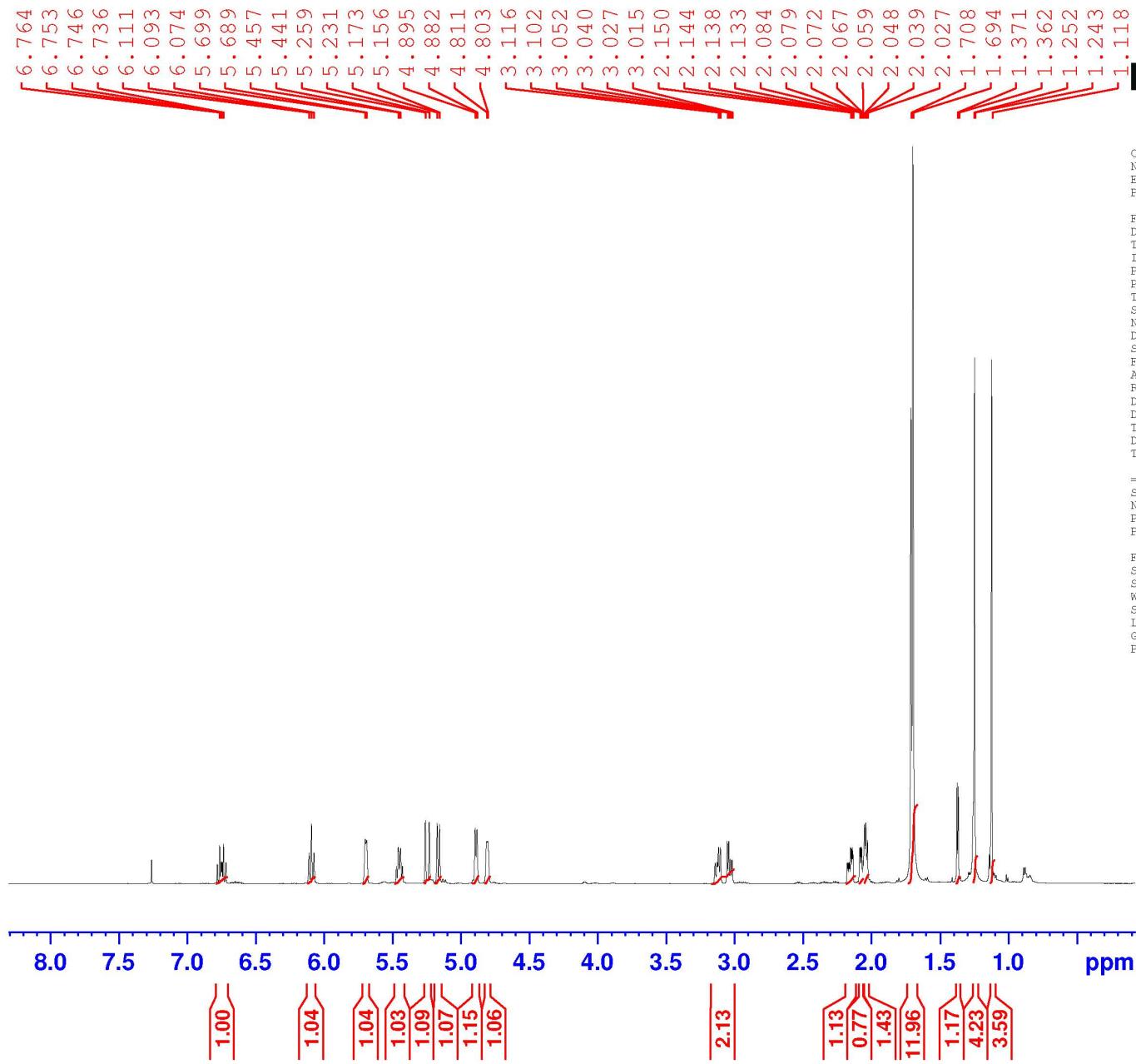
ID 472
 SF01 600.1324 MHz
 FIDRES 11.464524 Hz
 SW 9.017 ppm
 FnMODE States-TPII

F2 - Processing parameters
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 SF 600.1300256 MHz
 WDW QSINK
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
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 MC2 States-TPII
 SF 600.1300234 MHz
 WDW QSINK
 SSB 0
 LB 0 Hz
 GB 0



4S-(4a) NMR Characterisation

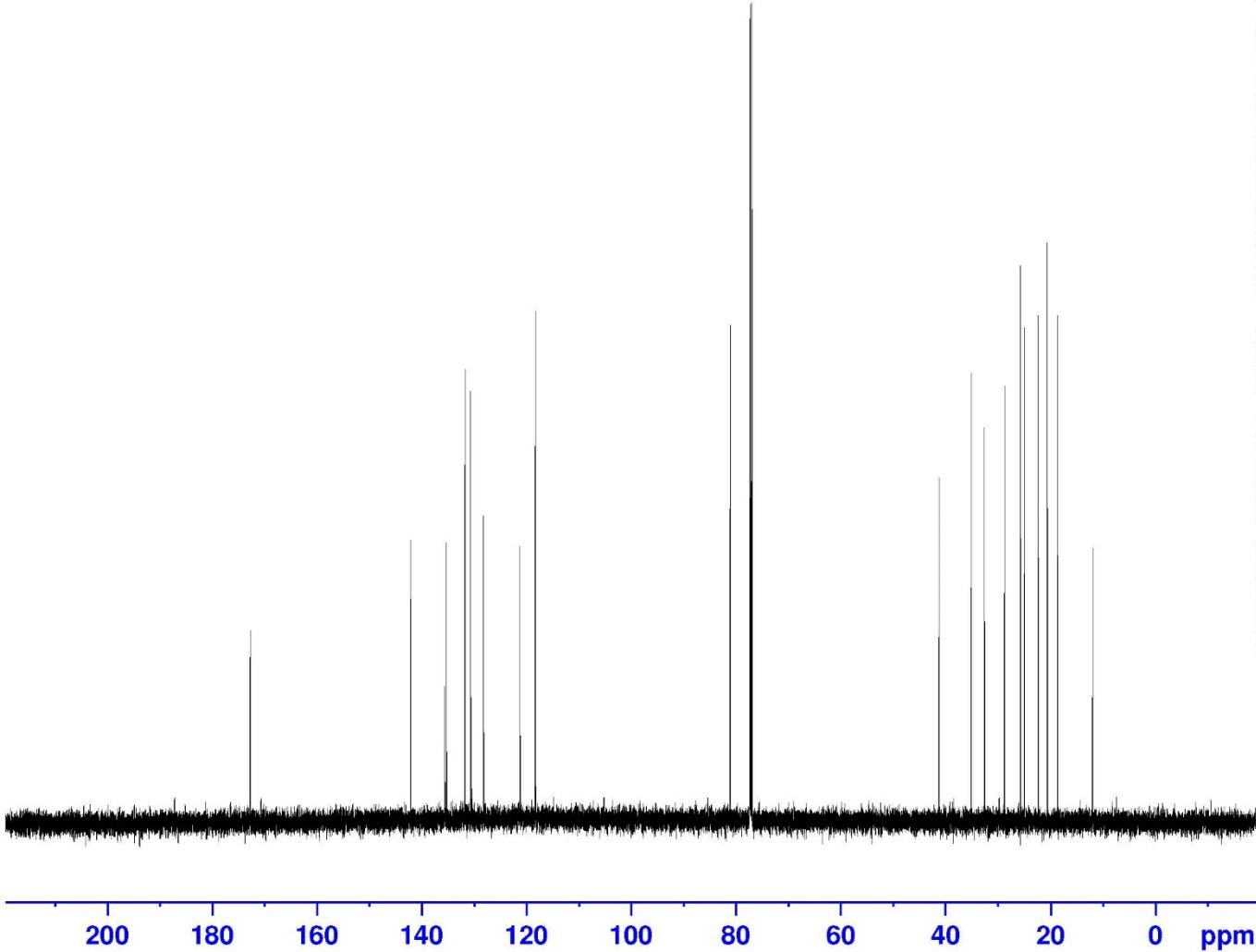


Current Data Parameters
 NAME 20190123 Pyrethrin I OH MINOR
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190123
 Time 18.09
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 32
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 ======
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 8.40 usec
 PLW1 12.55000019 W

P2 - Processing parameters
 SI 65536
 SF 600.1300240 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



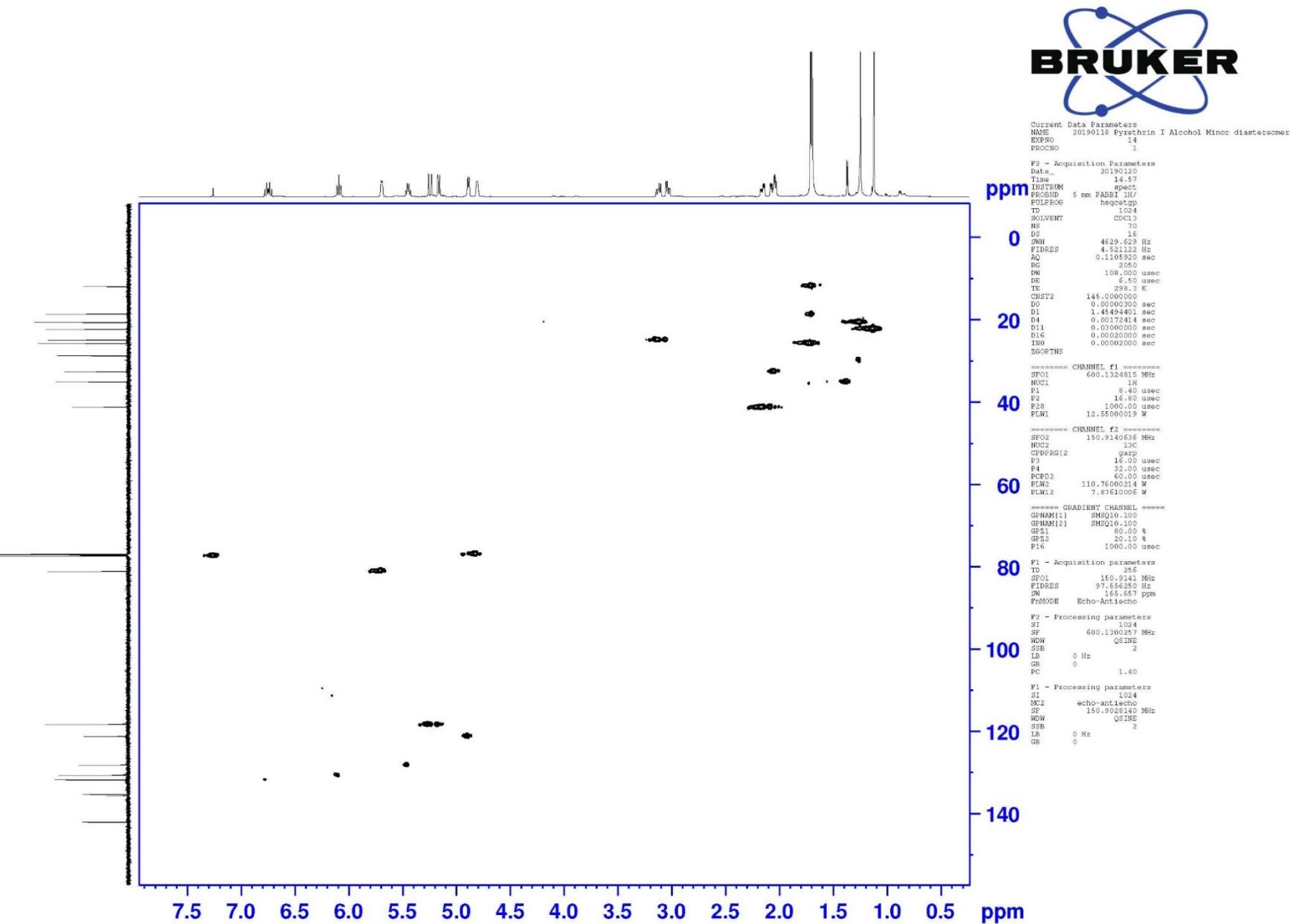
Current Data Parameters
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 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
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 Time 16.43
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 215
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 1440
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

----- CHANNEL f1 -----
 SFO1 150.917898 MHz
 NUC1 ¹³C
 P1 16.00 usec
 PLW1 110.76000214 W

----- CHANNEL f2 -----
 SFO2 600.1324005 MHz
 NUC2 ¹H
 CPDRG[2] waltz16
 FCPD2 70.00 usec
 PLW2 12.55000019 W
 PLW12 0.18072000 W
 PLW13 0.08855400 W

F2 - Processing parameters
 SI 32768
 SF 150.9027917 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40





Current Data Parameters
 NAME 20190122 Pyrethrin I OH MINOR
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date 20190122
 Time 16.54
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG noesypph
 TD 4096
 SOLVENT CDC13
 NS 2
 DS 16
 SWH 5411.255 Hz
 FIDRES 1.321107 Hz
 AQ 0.3784704 sec
 RG 50.8
 DW 92.400 usec
 DE 6.50 usec
 TE 298.2 K
 DO 0.00000170 sec
 D1 3.0000000 sec
 D8 0.60000002 sec
 D16 0.00020000 sec
 INU 0.00018480 sec

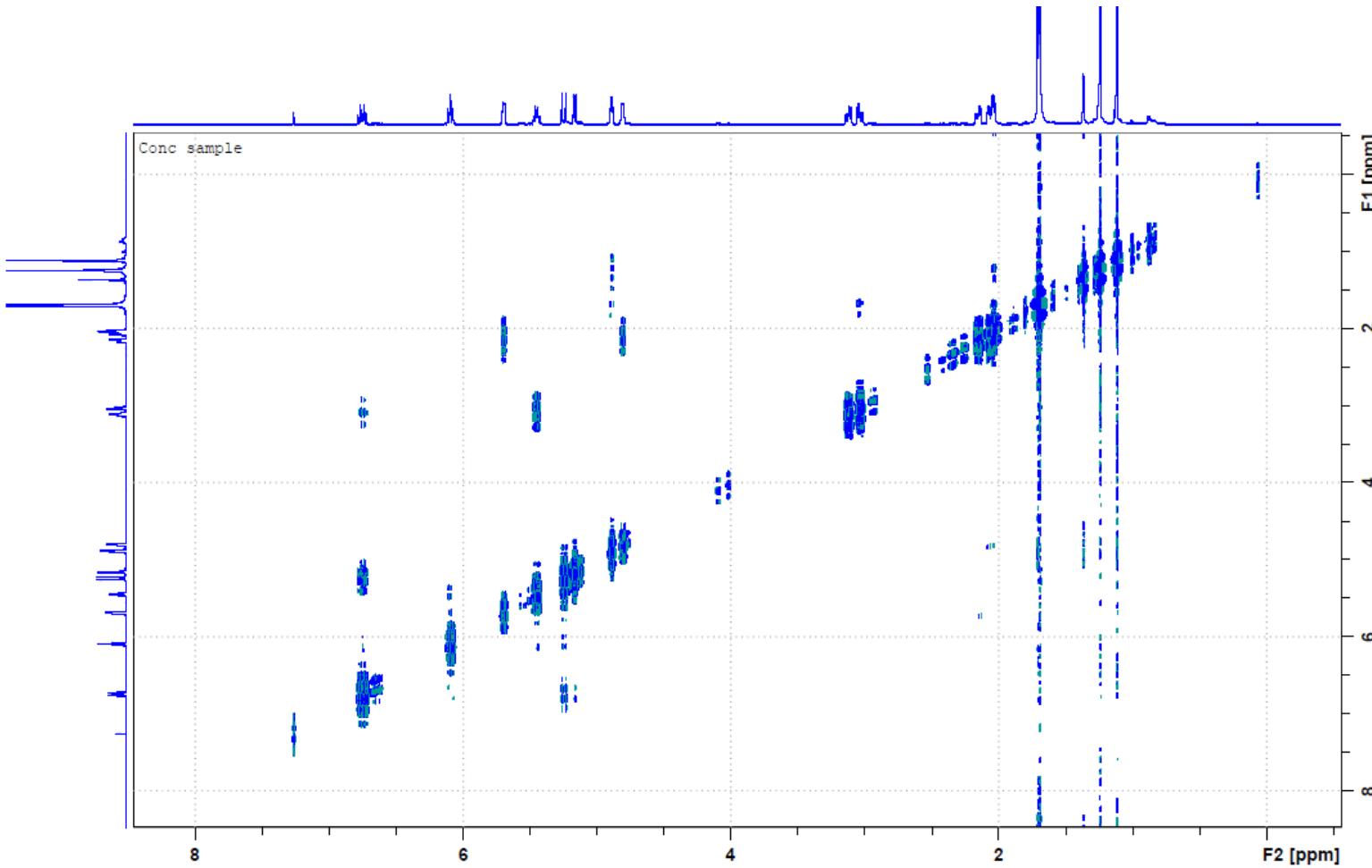
----- CHANNEL f1 -----
 SFO1 600.1323960 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 PLW1 12.55000019 W

----- GRADIENT CHANNEL -----
 GPNAM[1] SMSQ10.100
 GPZ1 40.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 ID 127
 SFO1 600.1324 MHz
 FIDRES 42.608311 Hz
 SW 9.017 ppm
 FnMODE States-TPPI

F2 - Processing parameters
 SI 4096
 SF 600.1300253 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 States-TPPI
 SF 600.1300135 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



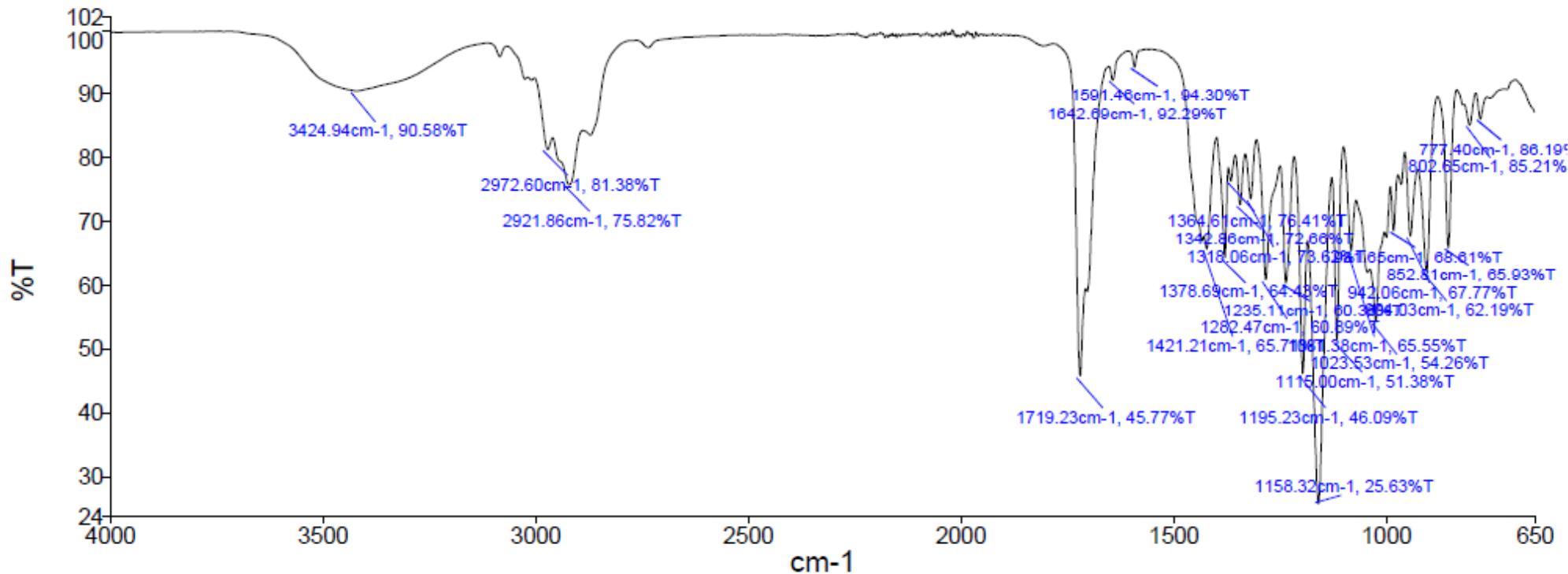
(4a) IR Spectrum

14

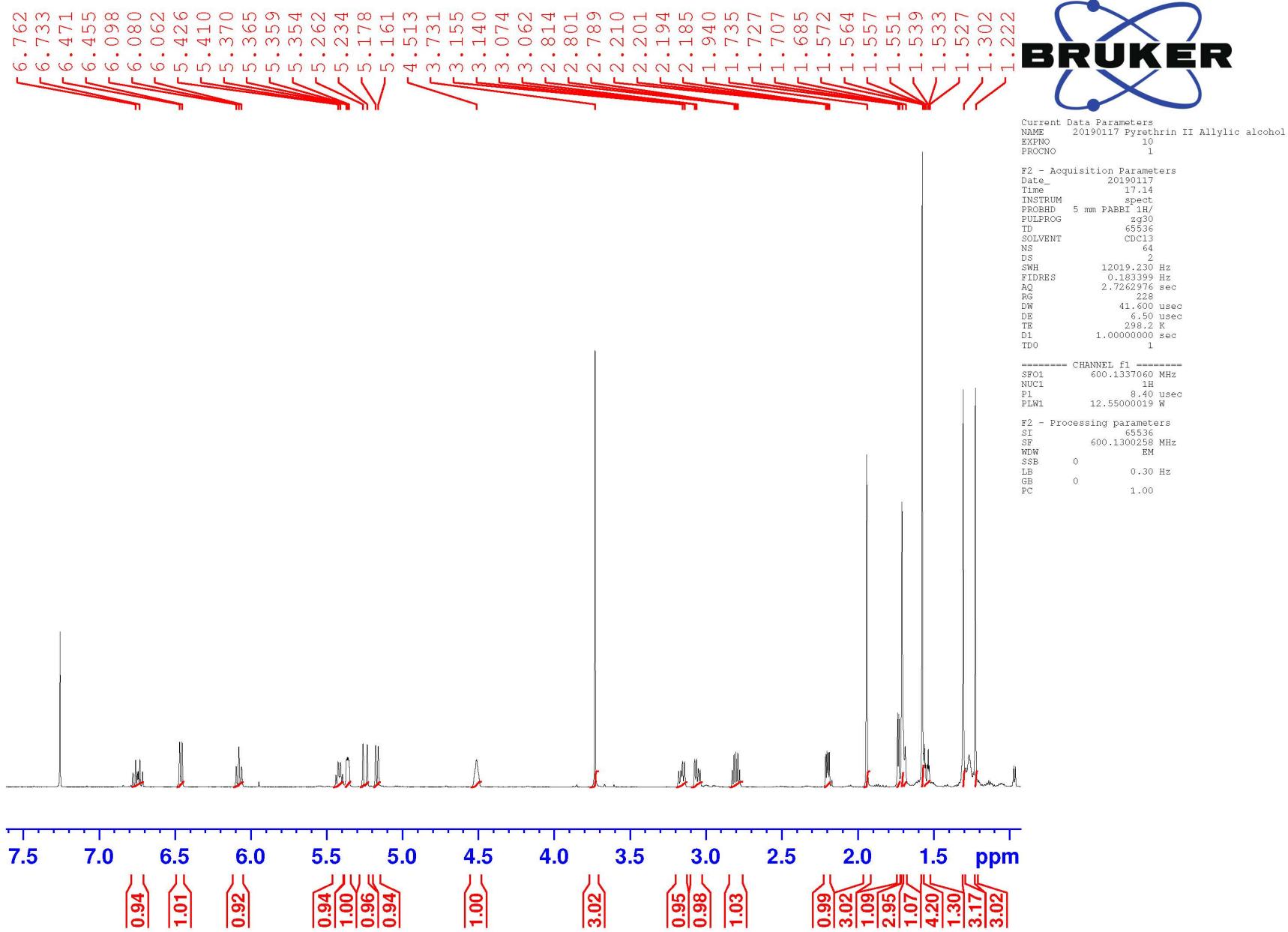
Analyst
Date

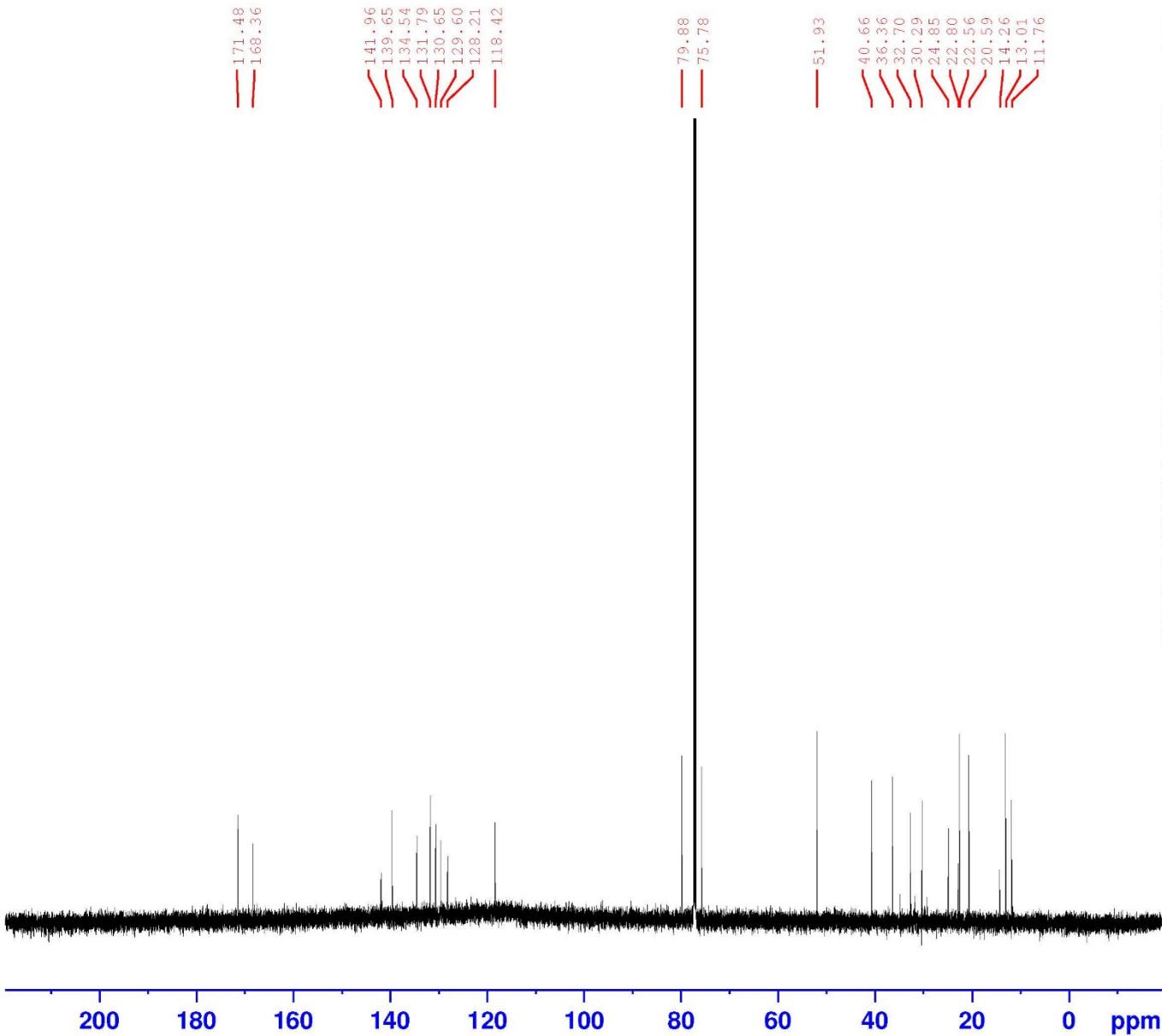
Analyst
Monday, 5 November 2018 1:51 PM

PerkinElmer Spectrum Version 10.4.2
Monday, 5 November 2018 1:51 PM



4R-(4b) NMR Characterisation





Current Data Parameters
NAME 20190117_Pyrethrin II Allylic alcohol
EXPNO 12
PROCNO 1

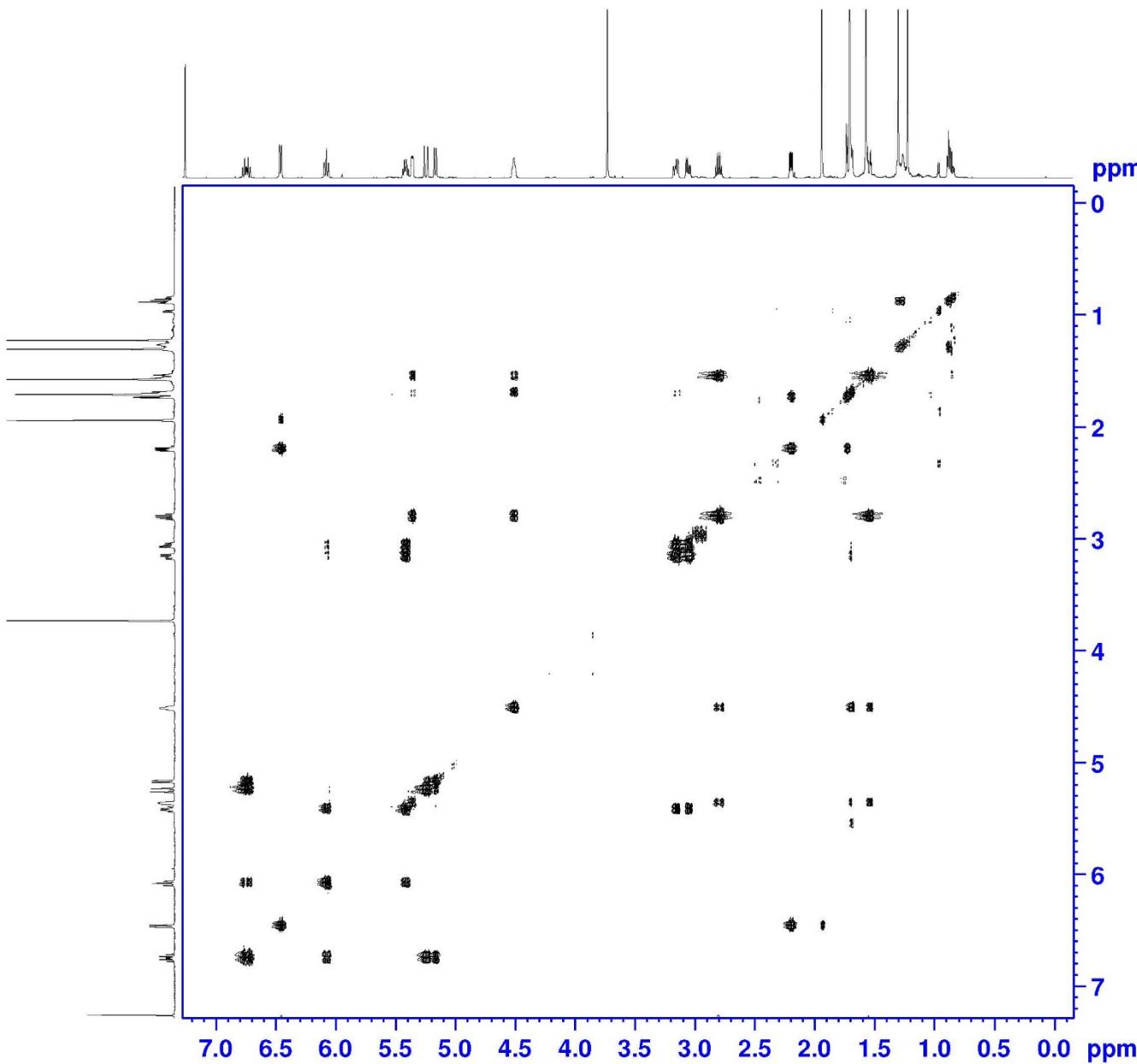
P2 - Acquisition Parameters

Date_ 20190117
Time_ 19.45
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 1440
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TDO 1

----- CHANNEL f1 -----
SF01 150.9178988 MHz
NUC1 13C
P1 16.00 usec
PLW1 110.76000214 W

----- CHANNEL f2 -----
SF02 600.1324005 MHz
NUC2 1H
CPDPFG[2] waltz16
CPDPRG2 70.00 usec
PLM2 12.55000019 W
PLM12 0.18072000 W
PLM13 0.08859400 W

P2 - Processing parameters
SI 32768
SF 150.9027891 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40



Current Data Parameters
NAME 20190117_Pyrethrin II Allylic alcohol
EXPNO 11
PROCNO 1

ppm

E2 - Acquisition Parameters

Date 20190117
Time 17:16
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG cosyqppmfpfpp
TD 2048
SOLVENT CDCl3
NS 5
DS 4
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2293760 sec
RG 2050
DW 112.000 usec
DE 6.50 usec
TE 298.2 K
DO 0.00010130 sec
D1 1.9090694 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
D10 0.00022400 sec

CHANNEL f1

SFO1 600.1321673 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P17 2500.00 usec
PLM1 12.55000019 W
PLM10 1.30999994 W

GRADIENT CHANNEL

GPNAME[1] SINE210.100

GPNAME[2] SINE210.100

GPZ1 10.00 %

GPZ2 20.00 %

P16 1000.00 usec

E1 - Acquisition parameters

TD 256

SFO1 600.1322 MHz

FIDRES 17.438616 Hz

SW 7.439 ppm

PRMODE States-TFFI

E2 - Processing parameters

SI 1024

SF 600.1300296 MHz

WDW QSINE

SSB 2

LB 0 Hz

GB 0

PC 1.40

E1 - Processing parameters

SI 1024

MC2 States-TFFI

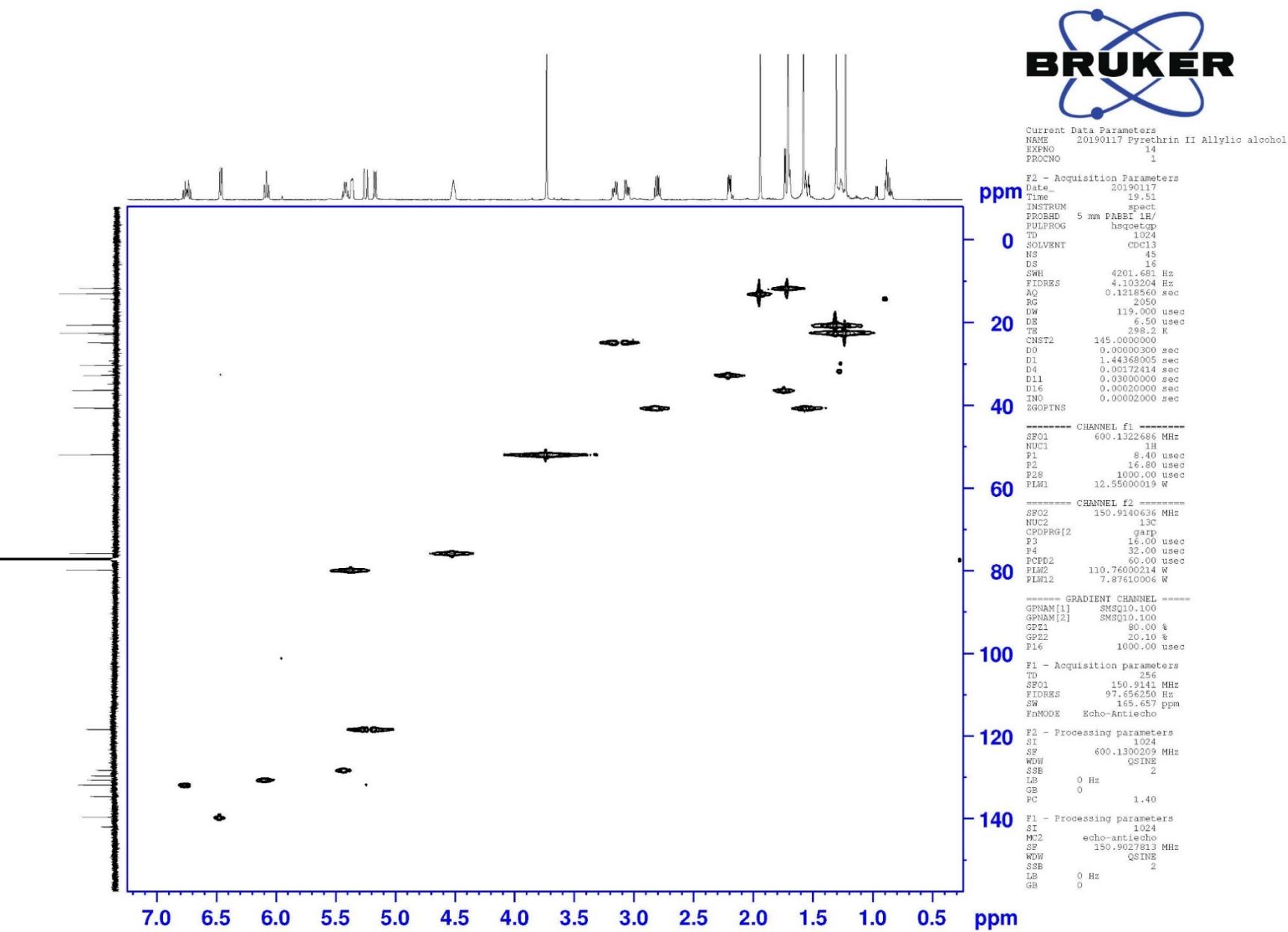
SF 600.1300274 MHz

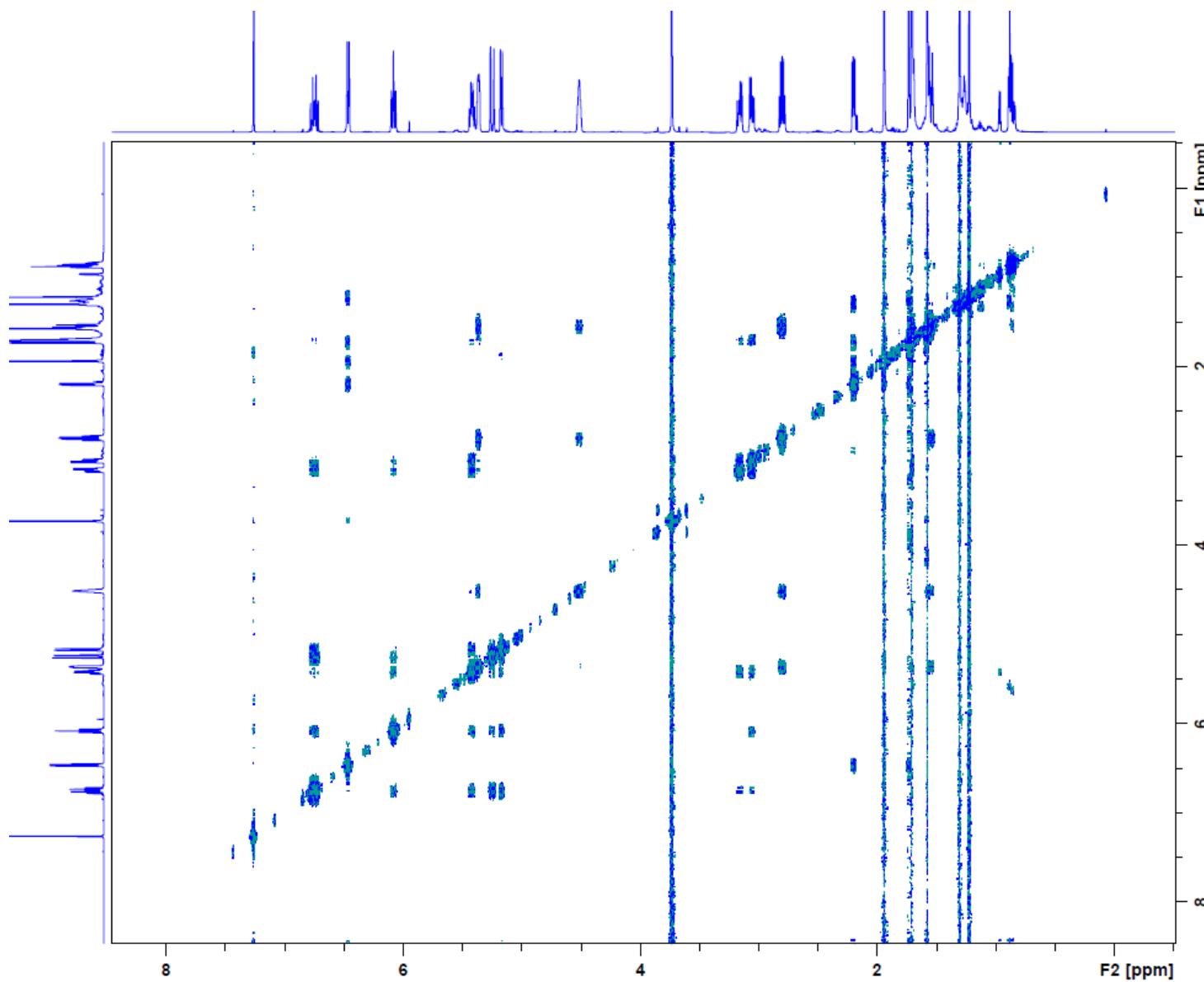
WDW QSINE

SSB 2

LB 0 Hz

GB 0





Current Data Parameters
 NAME 20190121 Pyrethrin II OH MAJOR
 EXPNO 10
 PROCN 1

F2 - Acquisition Parameters

Date 20190121
 Time 8.41
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG noesypph
 TD 4096
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 5411.255 Hz
 FIDRES 1.321107 Hz
 AQ 0.3784704 sec
 RG 203
 DW 92.400 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00008170 sec
 D1 3.0000000 sec
 D8 0.6000002 sec
 D16 0.00020000 sec
 INO 0.00018480 sec

CHANNEL f1 =
 SFO1 600.1323960 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 PLW1 12.55000019 W

GRADIENT CHANNEL
 GPNAM[1] SMSQ10.100
 GPZ1 40.00 %
 P16 1000.00 usec

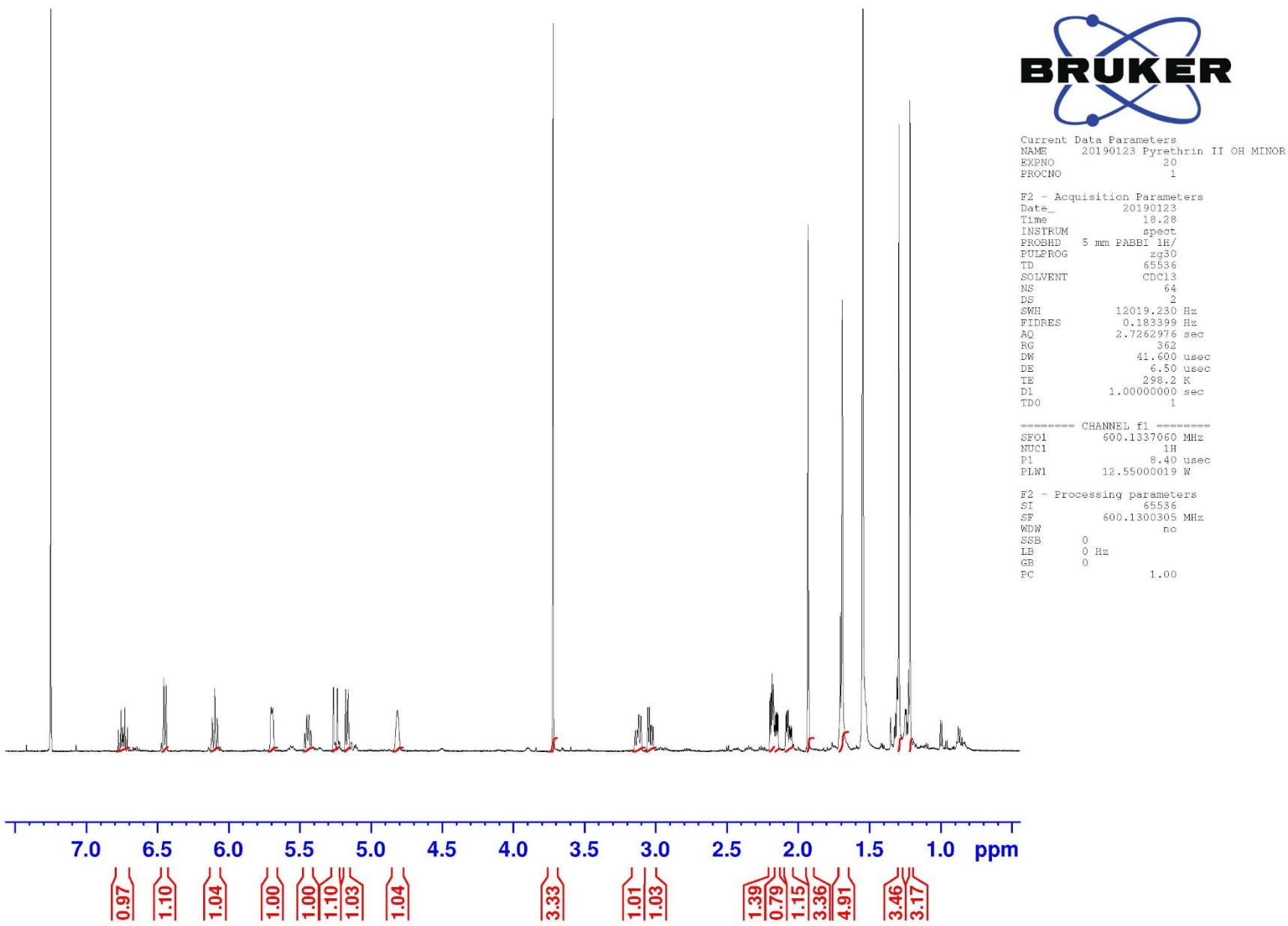
F1 - Acquisition parameters
 TD 397
 SFO1 600.1324 MHz
 FIDRES 13.630366 Hz
 SW 9.017 ppm
 FnMODE States-TPPI

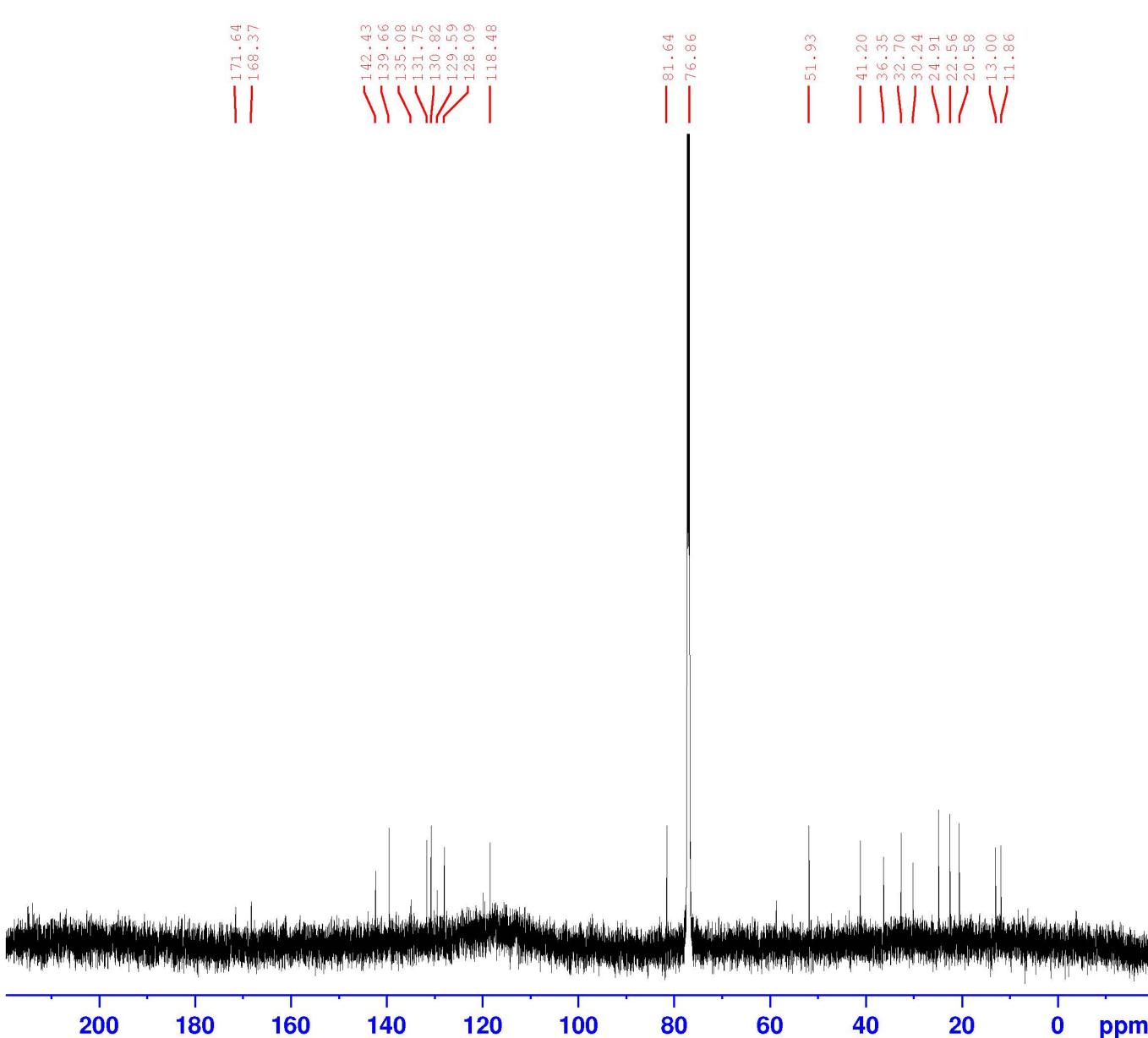
F2 - Processing parameters
 SI 4096
 SF 600.1300252 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 States-TPPI
 SF 600.1300210 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0

4S-(4b) NMR Characterisation

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Current Data Parameters
NAME 20190121_Pyrethrin II Minor OH
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190121
Time 22.33
INSTRUM spect
PROBHD 5 mm PABBI 1H
PULPROG zgppg30
TD 65536
SOLVENT CDCl₃
NS 6144
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 1440
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====

SFO1 150.9178988 MHz
NUC1 13C
P1 16.00 usec
PLW1 110.76000214 W

===== CHANNEL f2 =====

SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 usec
PLW2 12.55000019 W
PLW12 0.18072000 W
PLW13 0.08855400 W

F2 - Processing parameters

SI 32768
SF 150.9027895 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME 20190125 Pyrethrin II OH MINOR
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190125
 Time 10.54
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG cosygppmfpfpp
 TD 2048
 SOLVENT CDCl3
 NS 6
 DS 4
 SWH 4201.681 Hz
 FIDRES 2.051602 Hz
 AQ 0.2437120 sec
 RG 2050
 DW 119.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00010830 sec
 D1 1.89473295 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D16 0.00020000 sec
 IN0 0.00023800 sec

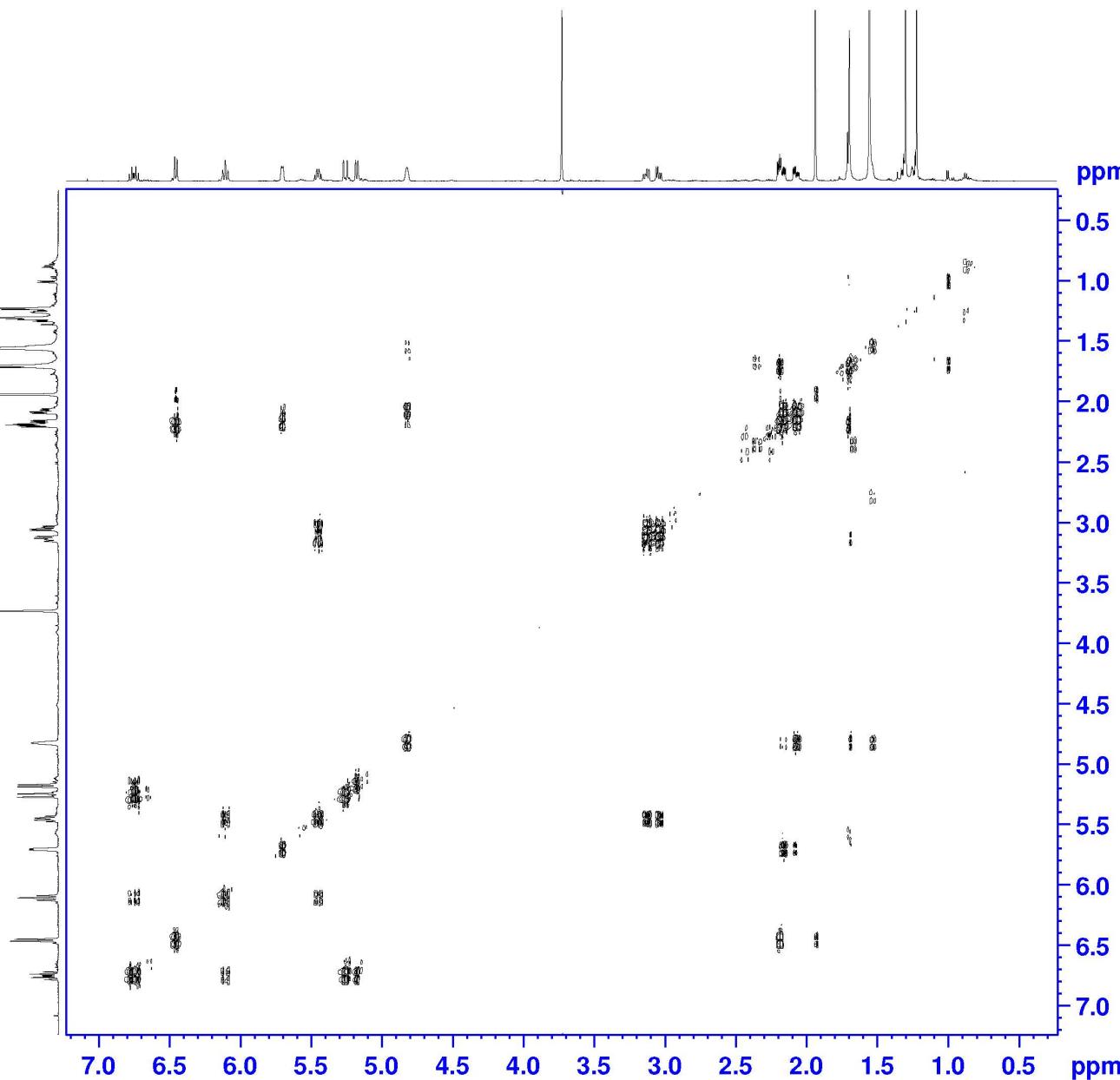
===== CHANNEL f1 =====
 SFO1 600.1322665 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 P17 2500.00 usec
 PLW1 12.55000019 W
 PLW10 1.30999994 W

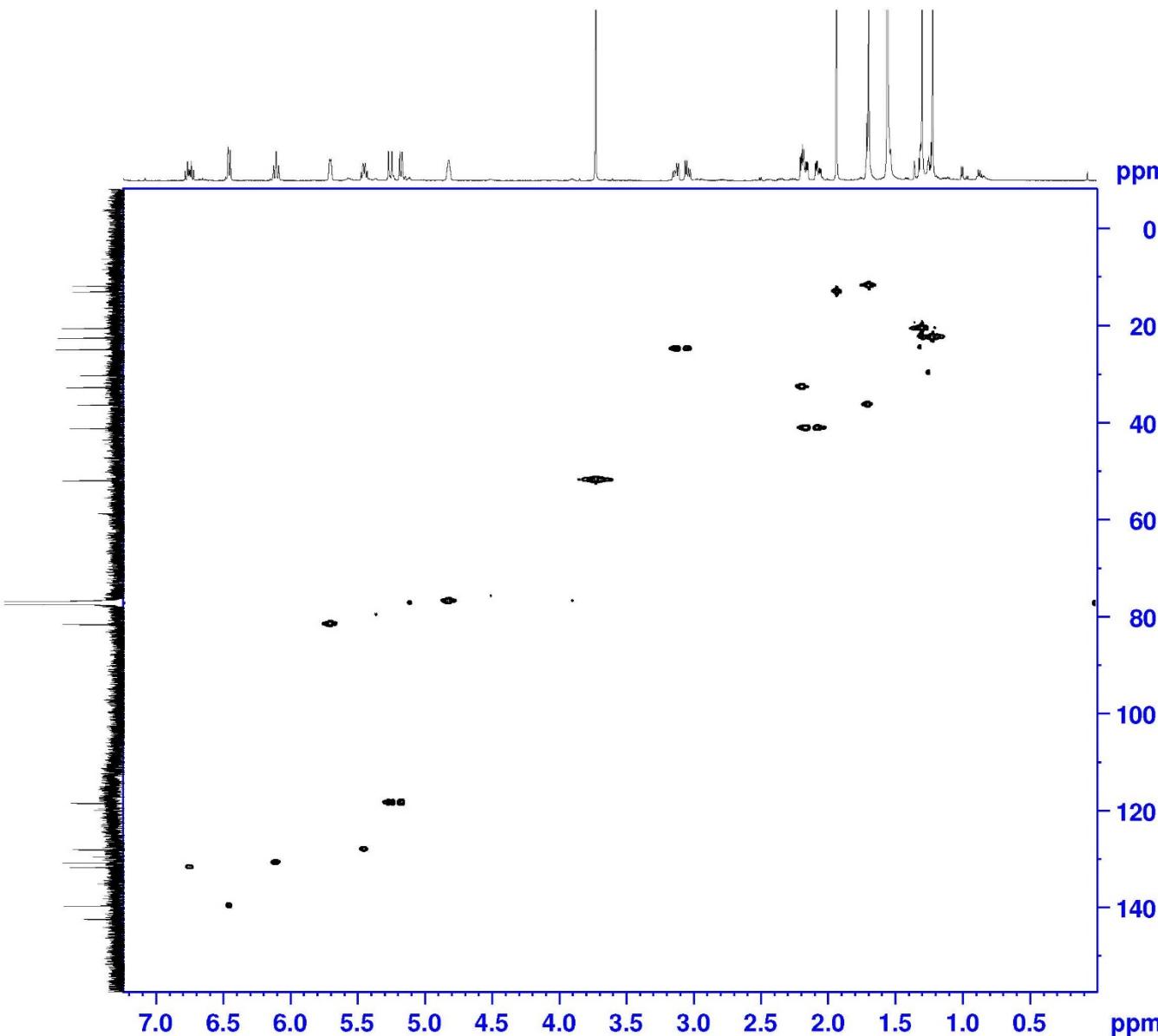
===== GRADIENT CHANNEL =====
 GENAM[1] SMS10.100
 GENAM[2] SMSQ10.100
 GP21 10.00 %
 GPZ2 20.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 124
 SFO1 600.1323 MHz
 FIDRES 33.884521 Hz
 SW 7.001 ppm
 FnMODE States-TPPI

F2 - Processing parameters
 SI 1024
 SF 600.1300272 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 States-TPPI
 SF 600.1300209 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0





Current Data Parameters
NAME: 20190118_Pyrothrin II Alcohol Minor diastereomer
EXPNO: 12
PROCNO: 1

F2 - Acquisition Parameters

Date: 20190119
Time: 12:28
INSTRUM: spect
PROBHD: 5 mm PABET 1H/
PULPROG: hexacetatepp
TD: 65536
SOLVENT: CDCl3
NS: 70
DS: 16
SWH: 4.2473824 Hz
FIDRES: 4.245924 Hz
AQ: 0.1177600 sec
RG: 2050
DW: 115.000 usec
DE: 6.50 usec
TE: 298.3 K
CPDGT2: 145.000000000 sec
D0: 0.00000200 sec
D1: 1.44717596 sec
D4: 0.00172414 sec
D11: 0.00000000 sec
D16: 0.00020000 sec
IM: 0.00002000 sec
ZGOPTNS:

===== CHANNEL f1 =====

SP01 600.1322032 MHz
NUC1 1H
D1 8.00 usec
P2 16.00 usec
P28 1000.00 usec
PLW1 12.5500019 W

===== CHANNEL f2 =====

SP02 150.9140336 MHz
NUC2 13C
CPDPRG[2] 64TP
P3 16.00 usec
P4 32.00 usec
PCPD2 50.00 usec
PLX1 110.7600014 W
PLW12 7.87610006 W

===== GRADIENT CHANNEL =====

GRNM1[1] SM3010.100

GRNM2[2] SM3010.100

GFZ1 80.00 %

GFZ2 20.10 %

P16 1000.00 usec

F1 - Acquisition parameters

TD 65536
SPV01 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PMLMODE Echo-Antiecho

F2 - Processing parameters

SI 1024
SF 600.133600000 MHz
WDW QSBK
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters

SI 1024
MC2 echo-antiecho
SF 150.9028095 MHz
WDW QSBK
SSB 2
LB 0 Hz
GB 0



Current Data Parameters
 NAME 20190121_Pyrethrin II OH MINOR
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20190121
 Time 13.10
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG noesypph
 TD 4096
 SOLVENT CDCl3
 NS 2
 DS 16
 SWH 5411.255 Hz
 FIDRES 1.321107 Hz
 AQ 0.3784704 sec
 RG 575
 DW 92.400 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00008170 sec
 D1 3.0000000 sec
 D8 0.6000002 sec
 D16 0.00020000 sec
 IN0 0.00018480 sec

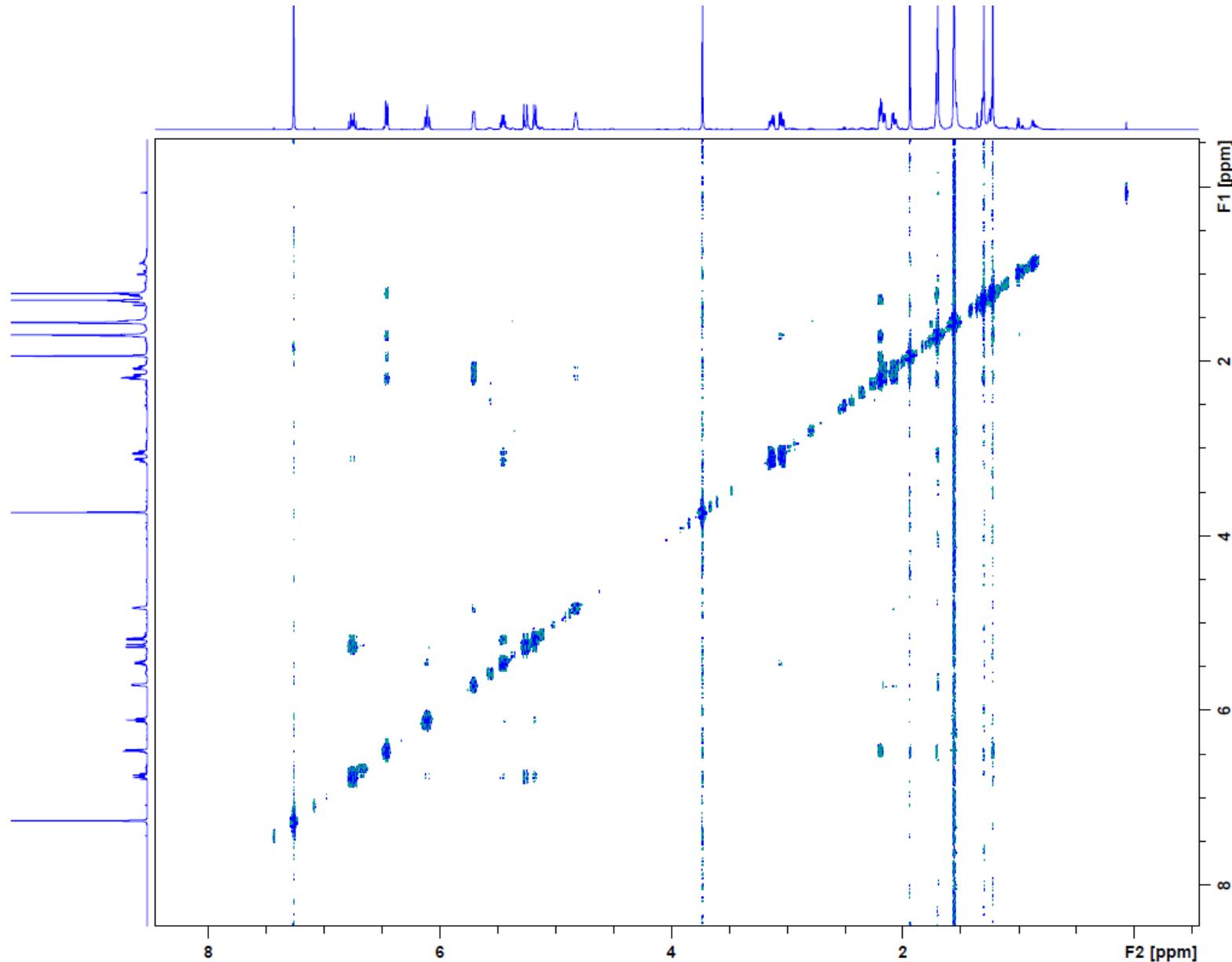
----- CHANNEL f1 -----
 SFO1 600.1323960 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 PLW1 12.55000019 W

----- GRADIENT CHANNEL -----
 GPNAM[1] SMSQ10.100
 GPZ1 40.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 ID 384
 SFO1 600.1324 MHz
 FIDRES 14.091811 Hz
 SW 9.017 ppm
 FmMODE States-TIPPI

F2 - Processing parameters
 SI 4096
 SF 600.1300256 MHz
 NDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 1024
 MC2 States-TIPPI
 SF 600.1300184 MHz
 NDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



(4b) IR Spectrum

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Analyst
Date

Analyst
Monday, 5 November 2018 1:52 PM

PerkinElmer Spectrum Version 10.4.2
Monday, 5 November 2018 1:52 PM

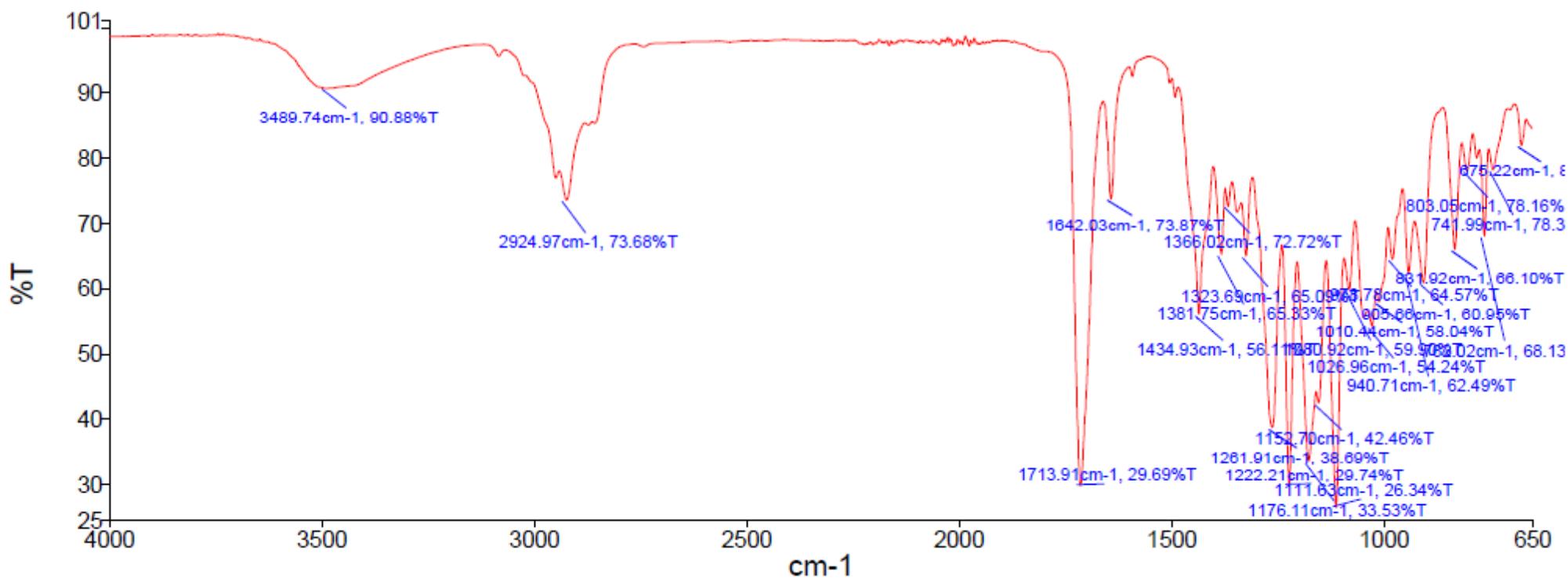
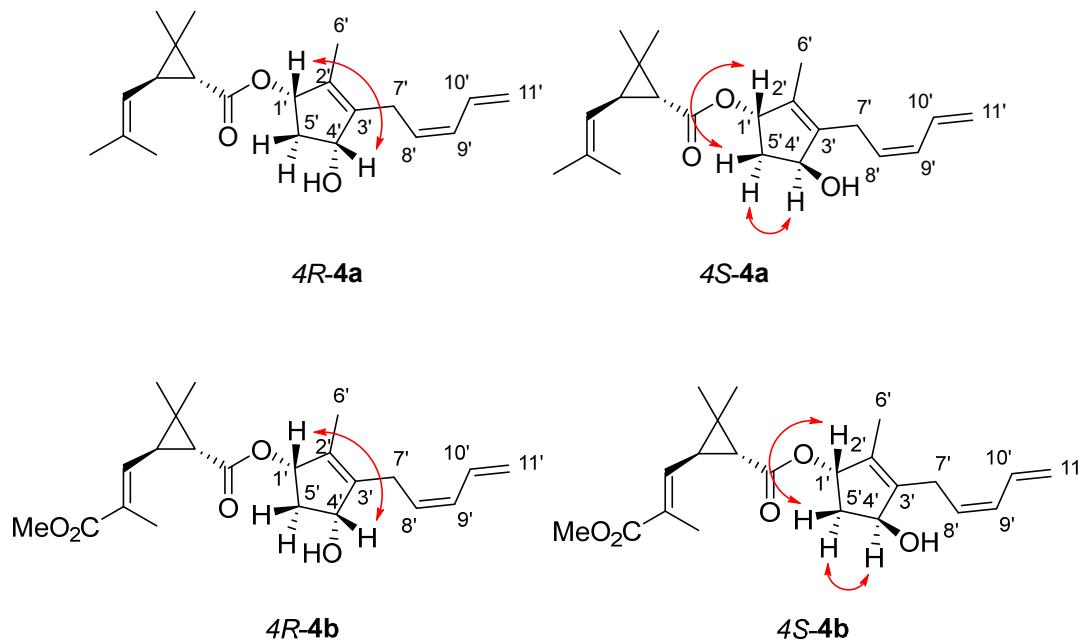
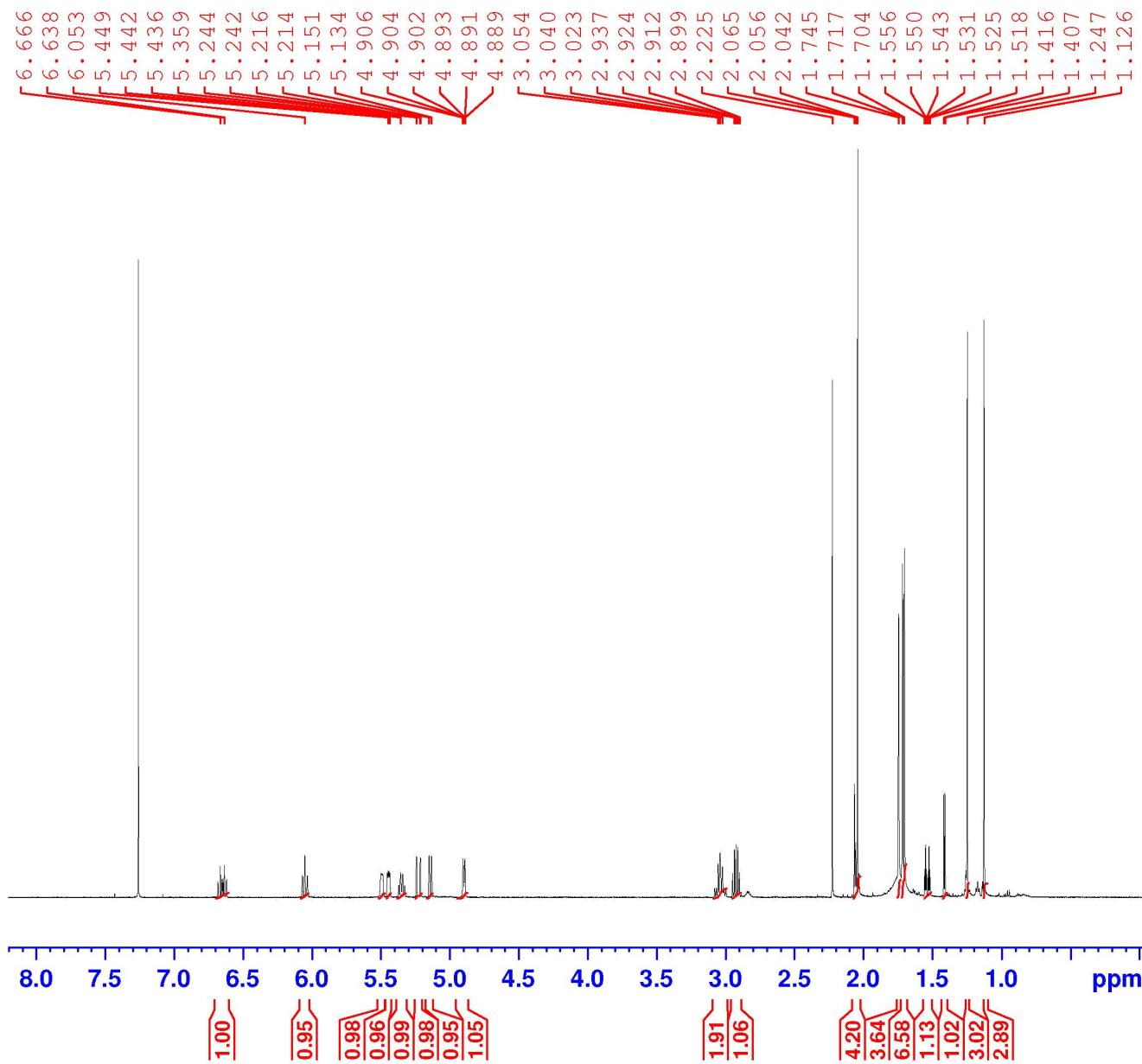


Figure showing nOe correlations for **4a** and **4b** epimers



4R-(5a) NMR Characterisation

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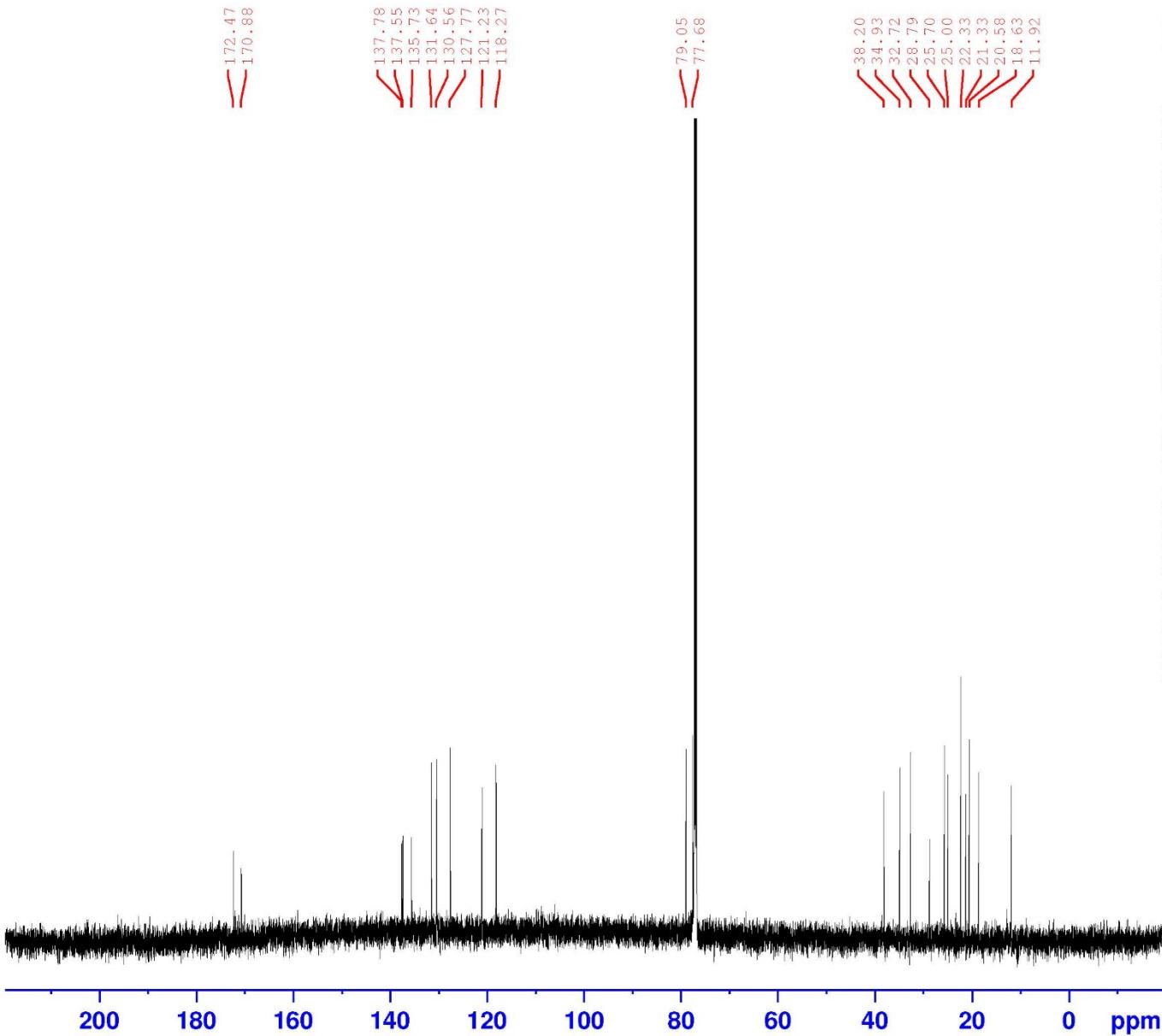


Current Data Parameters
NAME 20190218 Pyrethrin I Acetate MAJOR
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190218
Time 18.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 322
DW 41.600 usec
DE 6.50 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 600.1337061 MHz
NUC1 1H
P1 13.50 usec
PLW1 17.0000000 W

F2 - Processing parameters
SI 65536
SF 600.1300256 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



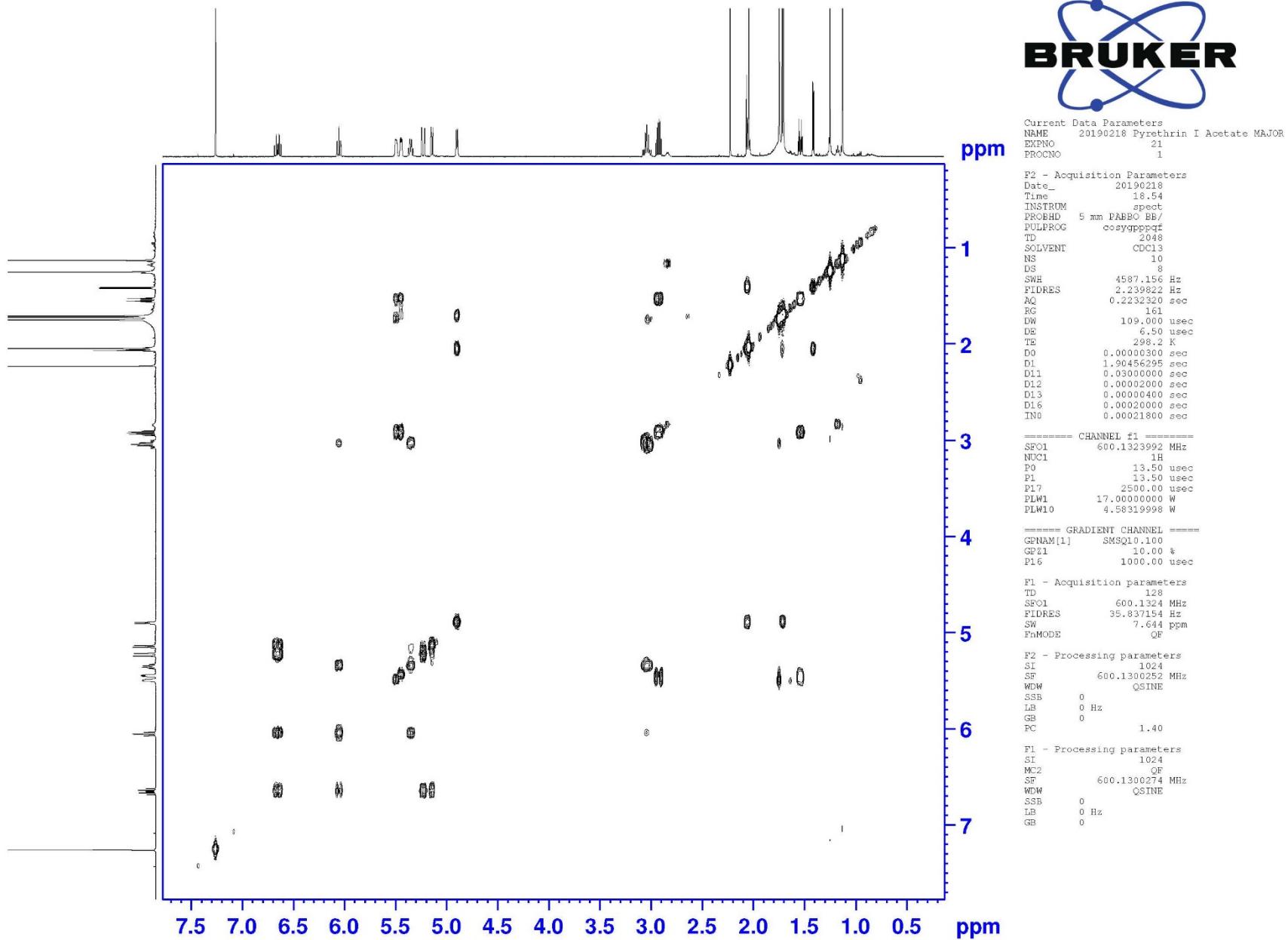
Current Data Parameters
 NAME 20190218_Pyrethrin_I_Acetate.MAJOR
 EXPNO 22
 PROCNO 1

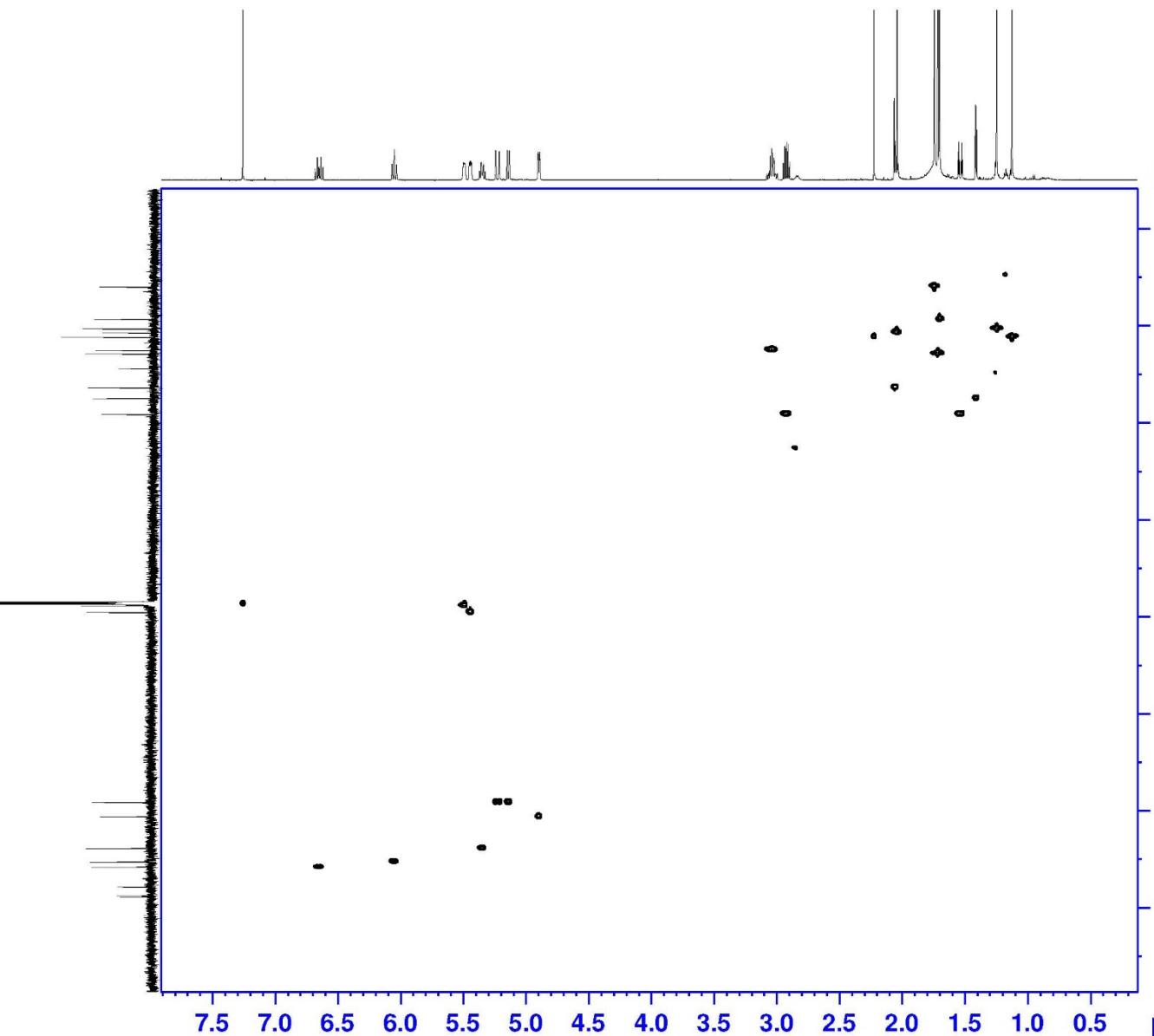
P2 - Acquisition Parameters
 Date_ 20190218
 Time 21:23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PLPROG zgppg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TR 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 ¹³C
 P1 12.00 usec
 PLW1 80.0000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.0000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

P2 - Processing parameters
 SI 32768
 SF 150.9027902 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
NAME 20190218 Pyrethrin I Acetate MAJOR
EXPNO 24
PROCNO 1

P2 - Acquisition Parameters

Date_ 20190218
Time_ 21.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 50
DS 16
SWH 4672.897 Hz
FIDRES 4.563376 Hz
AQ 0.1095680 sec
RG 2050
DW 107.000 usec
DE 6.50 usec
TE 298.3 K
CNST2 145.000000
D0 0.0000300 sec
D1 1.45596802 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002000 sec
ZGOPTNS

===== CHANNEL f1 =====
SF01 600.1324365 MHz
NUC1 1H
P1 13.50 usec
P2 27.00 usec
P28 1000.00 usec
PLW1 17.0000000 W

===== CHANNEL f2 =====
SF02 150.9140636 MHz
NUC2 13C
CPDPFRG[2] garp
P3 12.00 usec
P4 24.00 usec
PCPD2 60.00 usec
PLM2 80.0000000 W
PLW12 3.2000005 W

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

P1 - Acquisition parameters
TD 256
SF01 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PwMode Echo-Antiecho

P2 - Processing parameters
SI 1024
SF 600.1300252 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

P1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 150.9028159 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

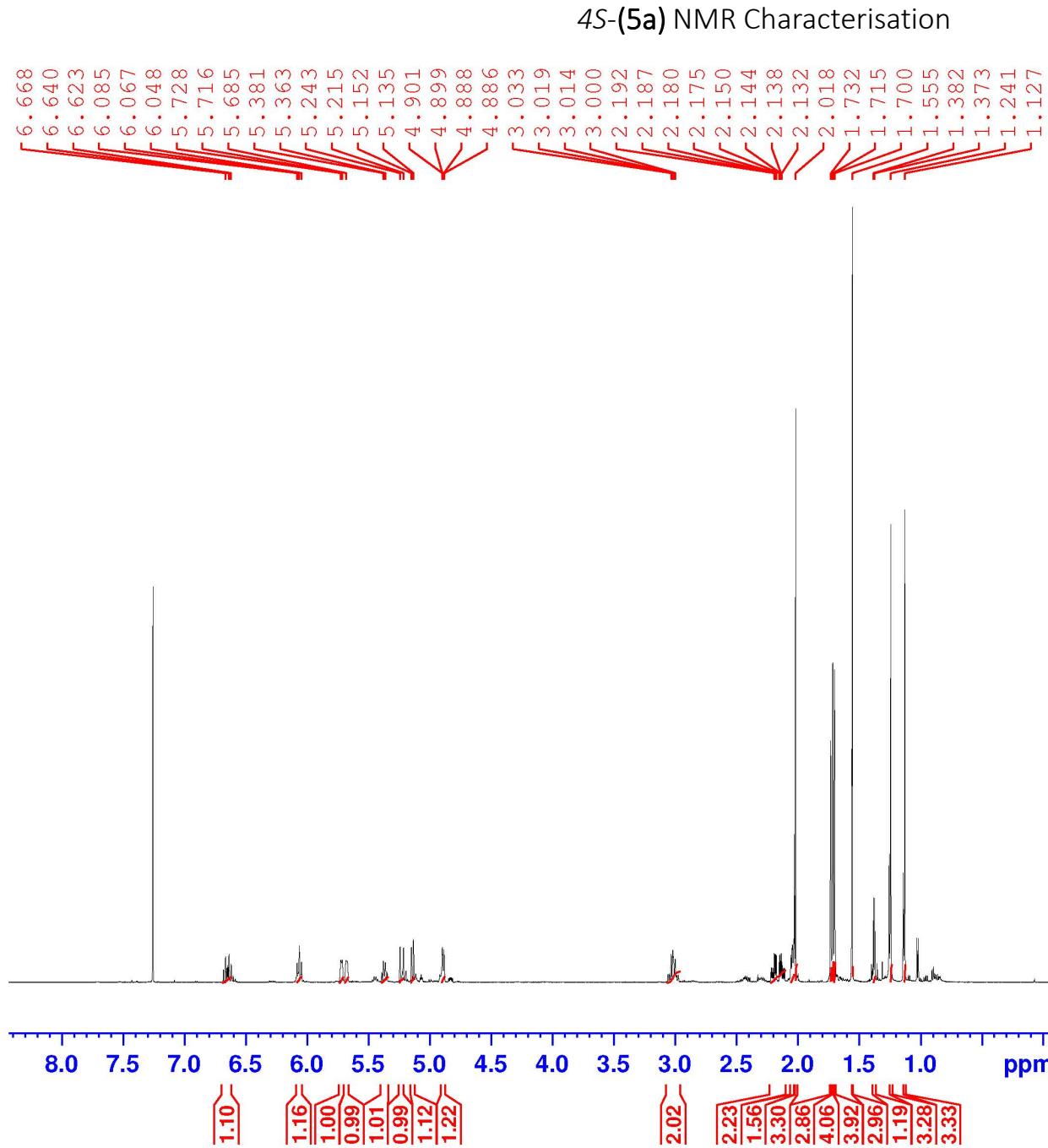


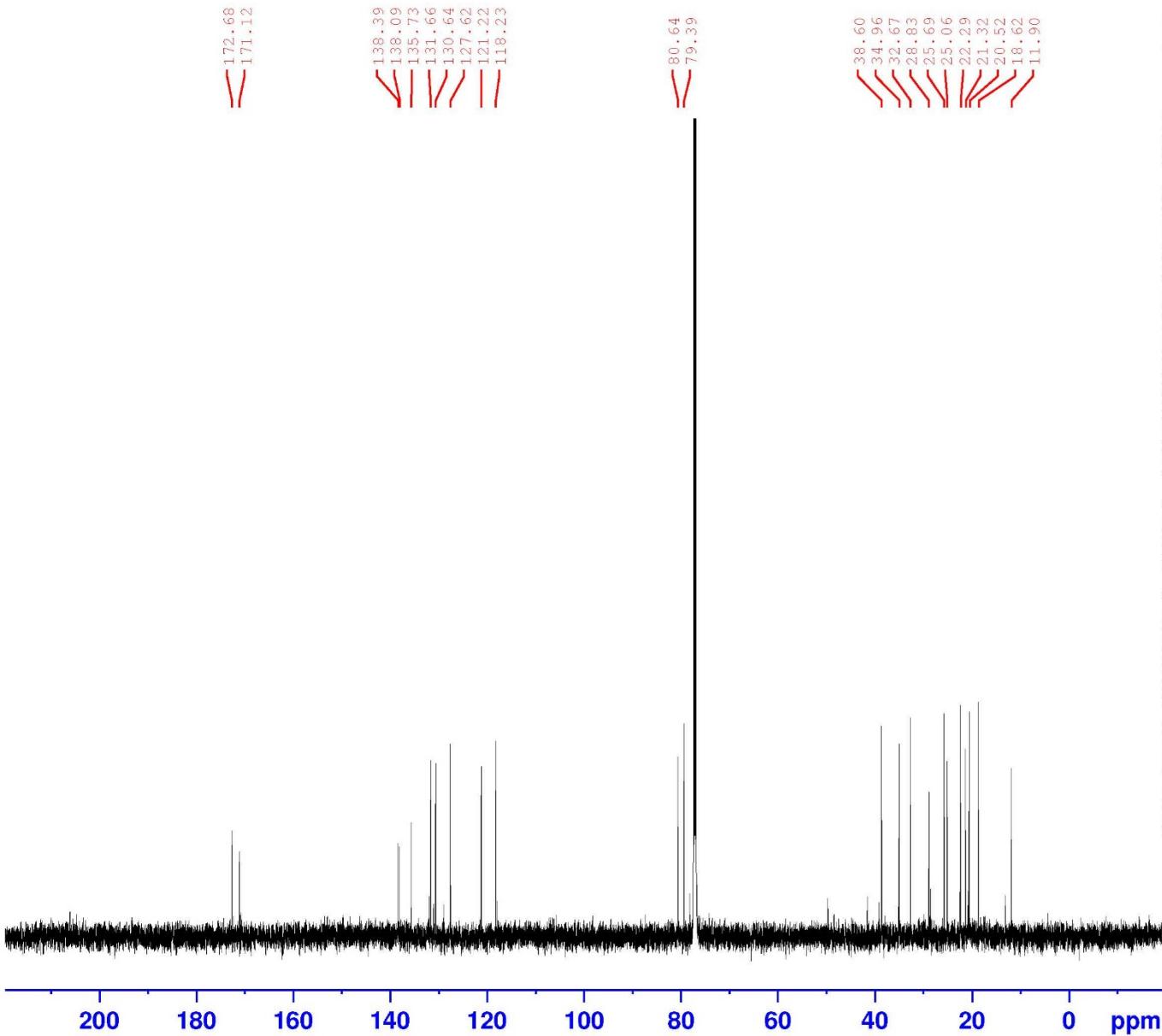
Current Data Parameters
 NAME 20190225 PI Acetate Minor
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190225
 Time 18.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 256
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 600.1337061 MHz
 NUC1 1H
 P1 13.50 usec
 PLW1 17.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300258 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00





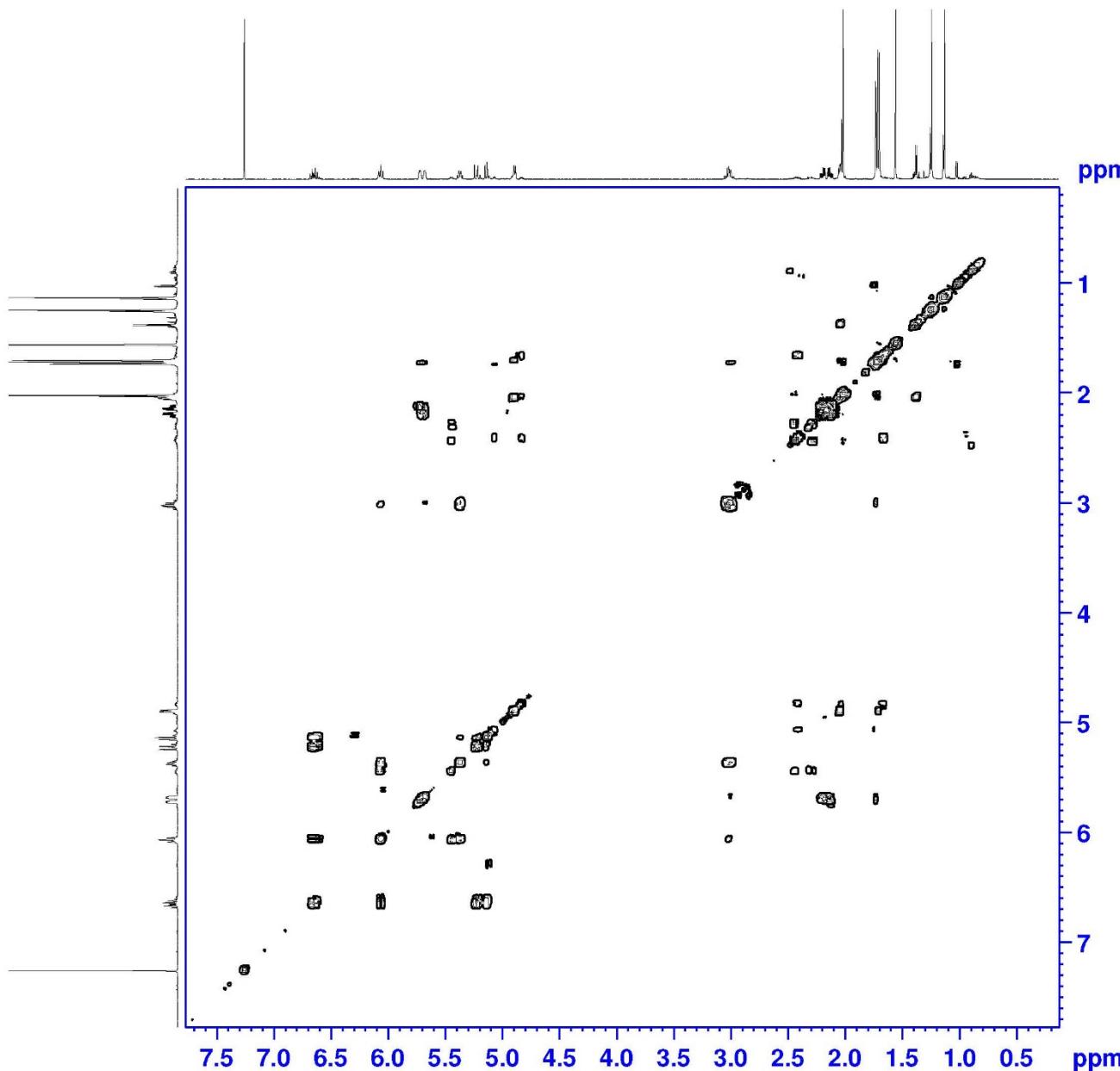
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 NAME 20190225 PI Acetate Minor
 EXPNO 22
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190225
 Time 21.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 80.0000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPMGR[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.0000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9027908 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 20190225 PI Acetate Minor
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190225
 Time 18.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppcf
 TD 2048
 SOLVENT CDCl3
 NS 10
 DS 8
 SWH 4587.156 Hz
 FIDRES 2.239822 Hz
 AQ 0.2232320 sec
 RC 144
 DW 109.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00000300 sec
 D1 1.90456295 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00021800 sec

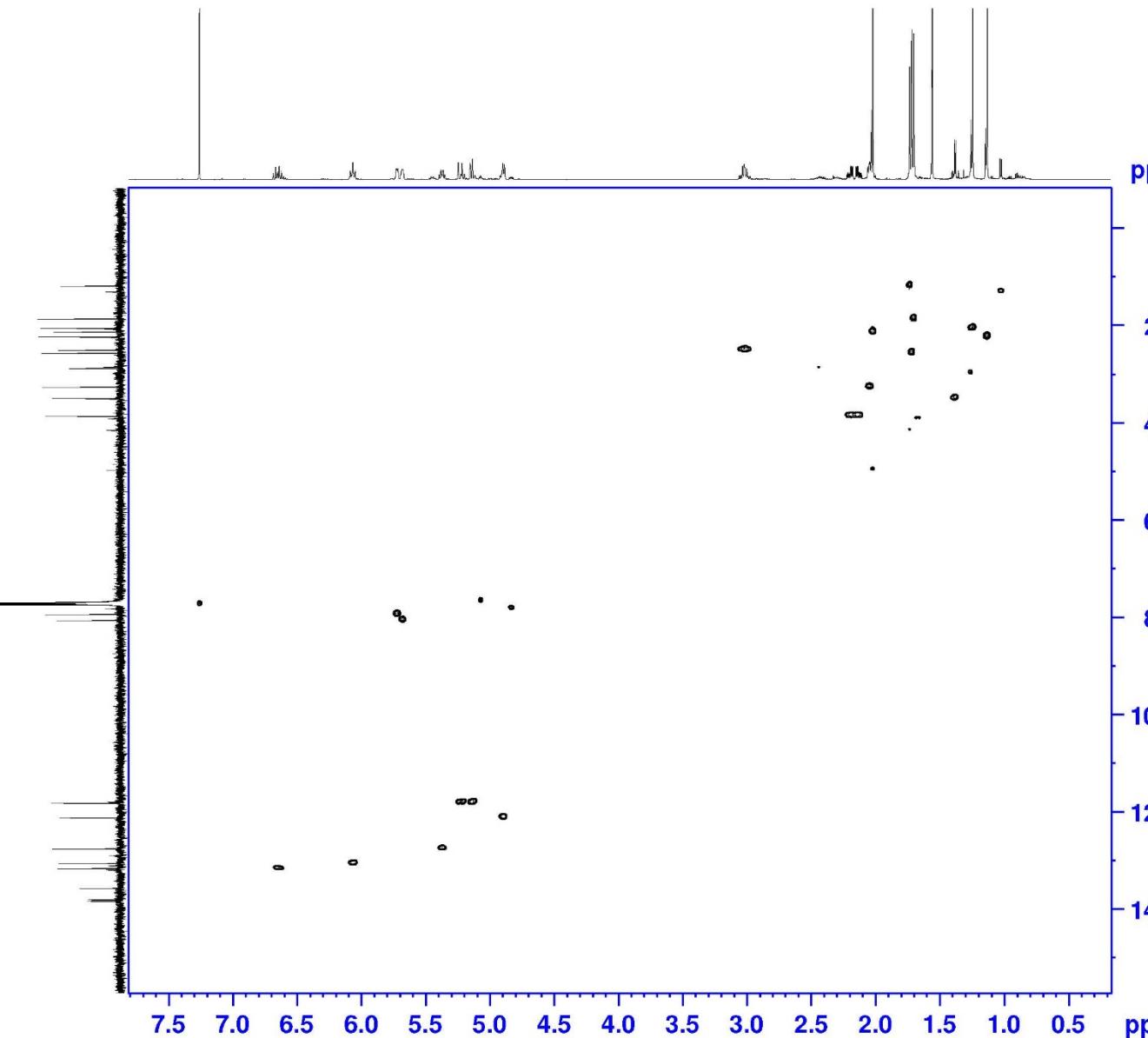
----- CHANNEL f1 -----
 SFO1 600.1323978 MHz
 NUC1 1H
 P0 13.50 usec
 P1 13.50 usec
 P17 2500.00 usec
 PLW1 17.00000000 W
 PLW10 4.58319998 W

----- GRADIENT CHANNEL -----
 GPNAM[1] SMSQ10.100
 GPZ1 10.00 %
 F16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 600.1324 MHz
 FIDRES 35.837154 Hz
 SW 7.644 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SP 600.1300256 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SP 600.1300261 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



Current Data Parameters
NAME 20190225 PI Acetate Minor
EXPNO 24
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190225
Time_ 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 50
DS 16
SWH 4587.156 Hz
FIDRES 4.479644 Hz
AQ 0.1116160 sec
RG 2050
DW 109.000 usec
DE 6.50 usec
TE 298.3 K
CNST2 145.000000
D0 0.00000300 sec
D1 1.45392001 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002000 sec
ZGOPTNS

===== CHANNEL f1 =====
SF01 600.1324175 MHz
NUC1 1H
P1 13.50 usec
P2 27.00 usec
P28 1000.00 usec
PLW1 17.0000000 W

===== CHANNEL f2 =====
SF02 150.9140636 MHz
NUC2 13C
CPDPFRG[2] garp
P3 12.00 usec
P4 24.00 usec
PCPD2 60.00 usec
PLM2 80.0000000 W
PLW2 3.2000005 W

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

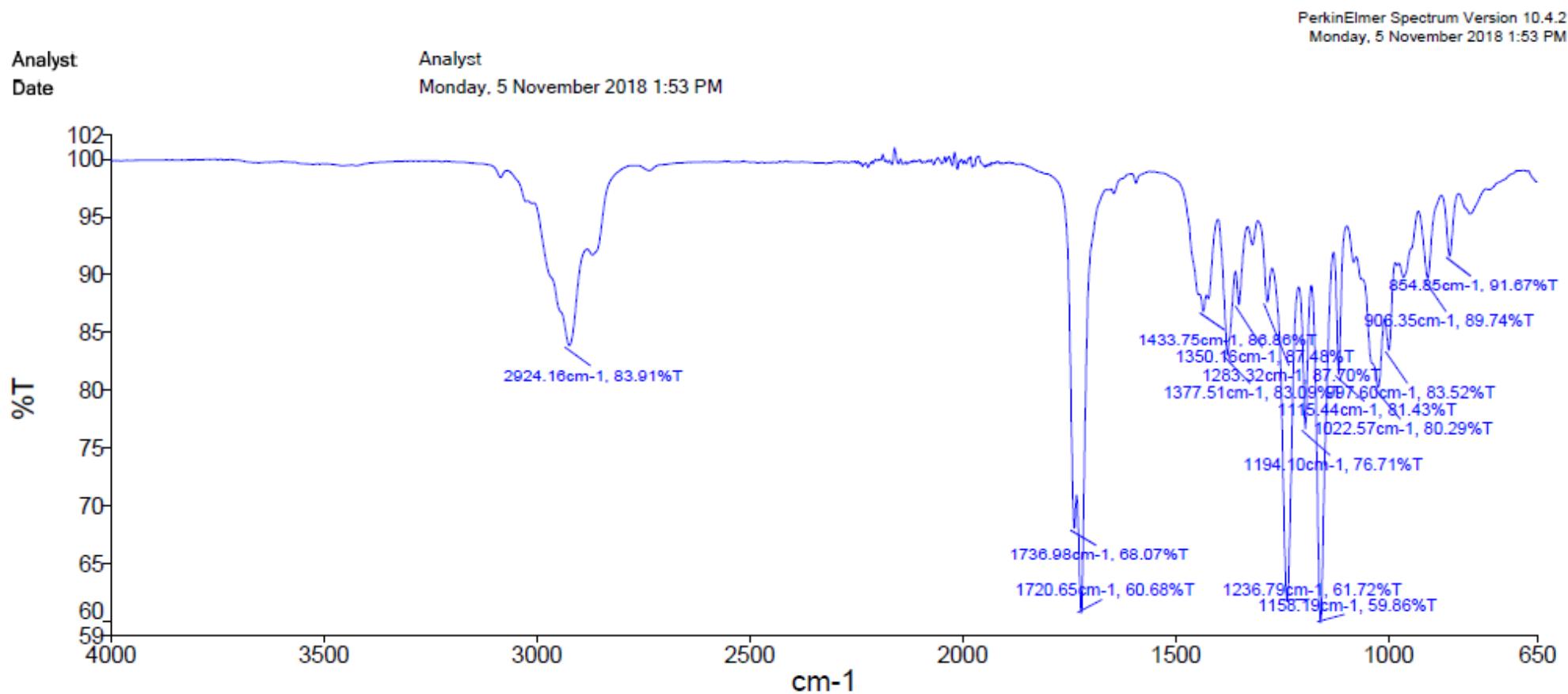
P1 - Acquisition parameters
TD 256
SF01 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PwMode Echo-Antiecho

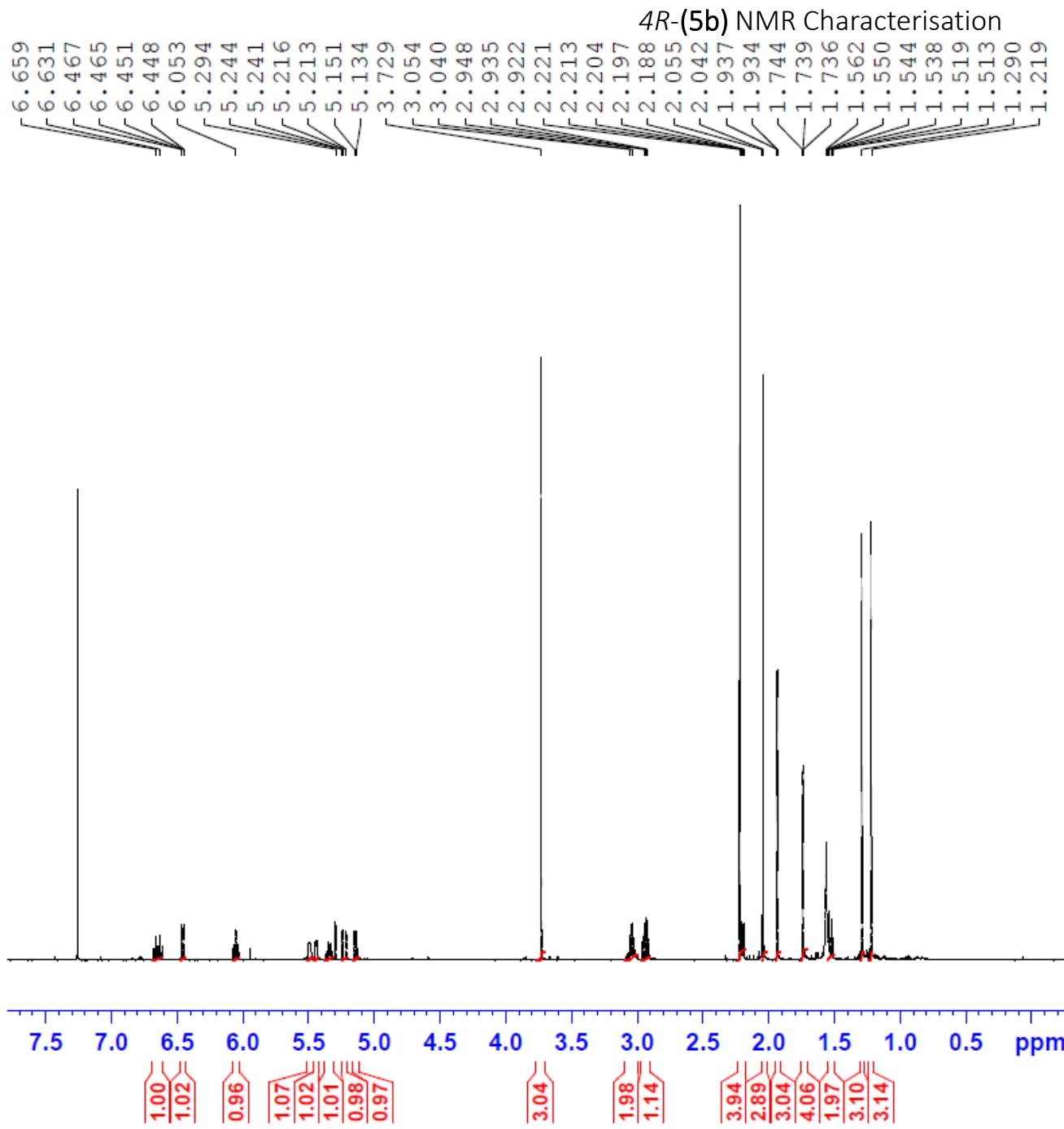
P2 - Processing parameters
SI 1024
SF 600.1300234 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

P1 - Processing parameters
SI 1024
NUC2 echo-antiecho
SF 150.9028227 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

(5a) IR Spectrum

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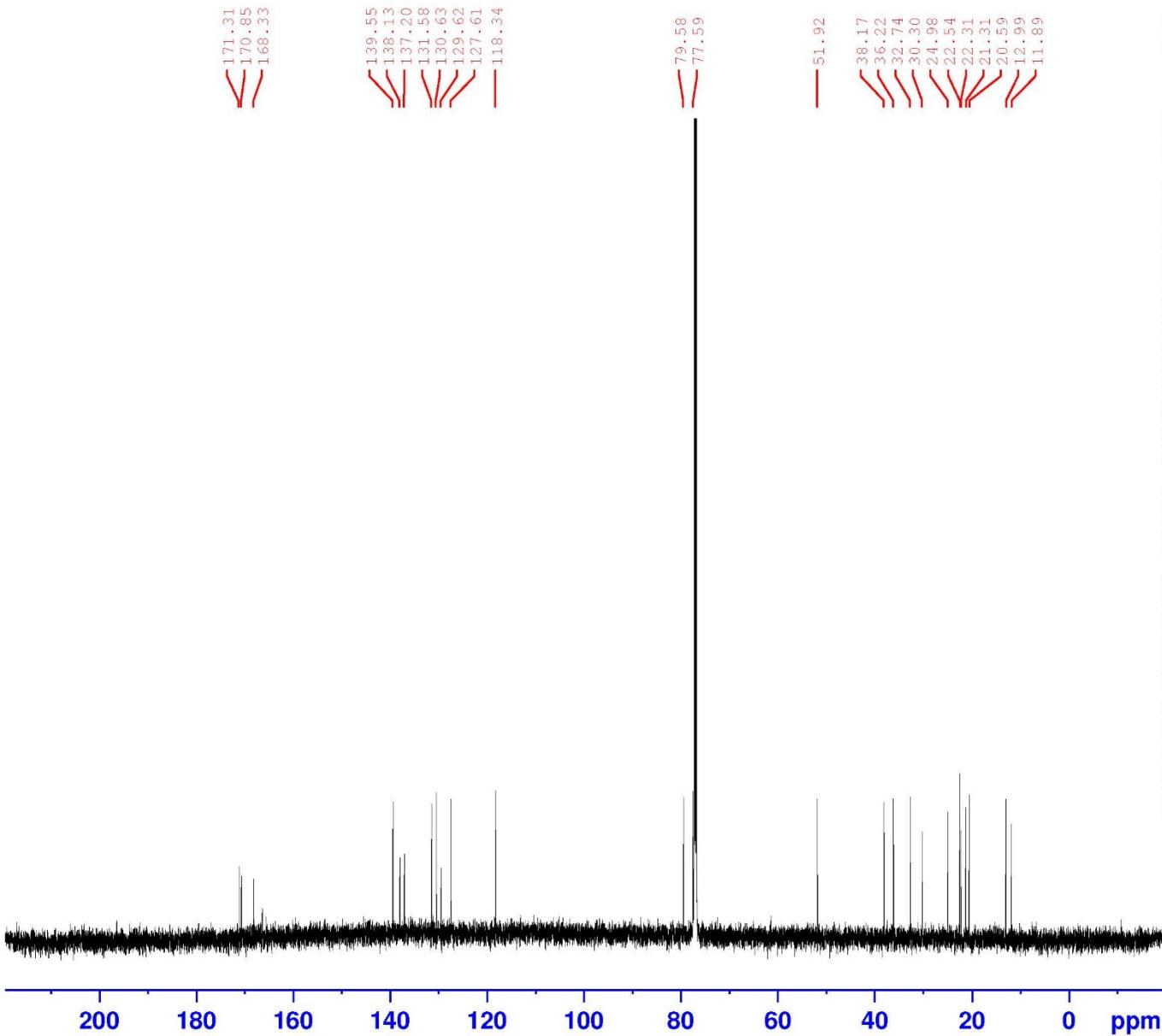


Current Data Parameters
 NAME 20190220 PII Acetate MAJOR
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190220
 Time_ 17.35
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 322
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 600.1337061 MHz
 NUC1 1H
 P1 13.50 usec
 PLW1 17.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300283 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



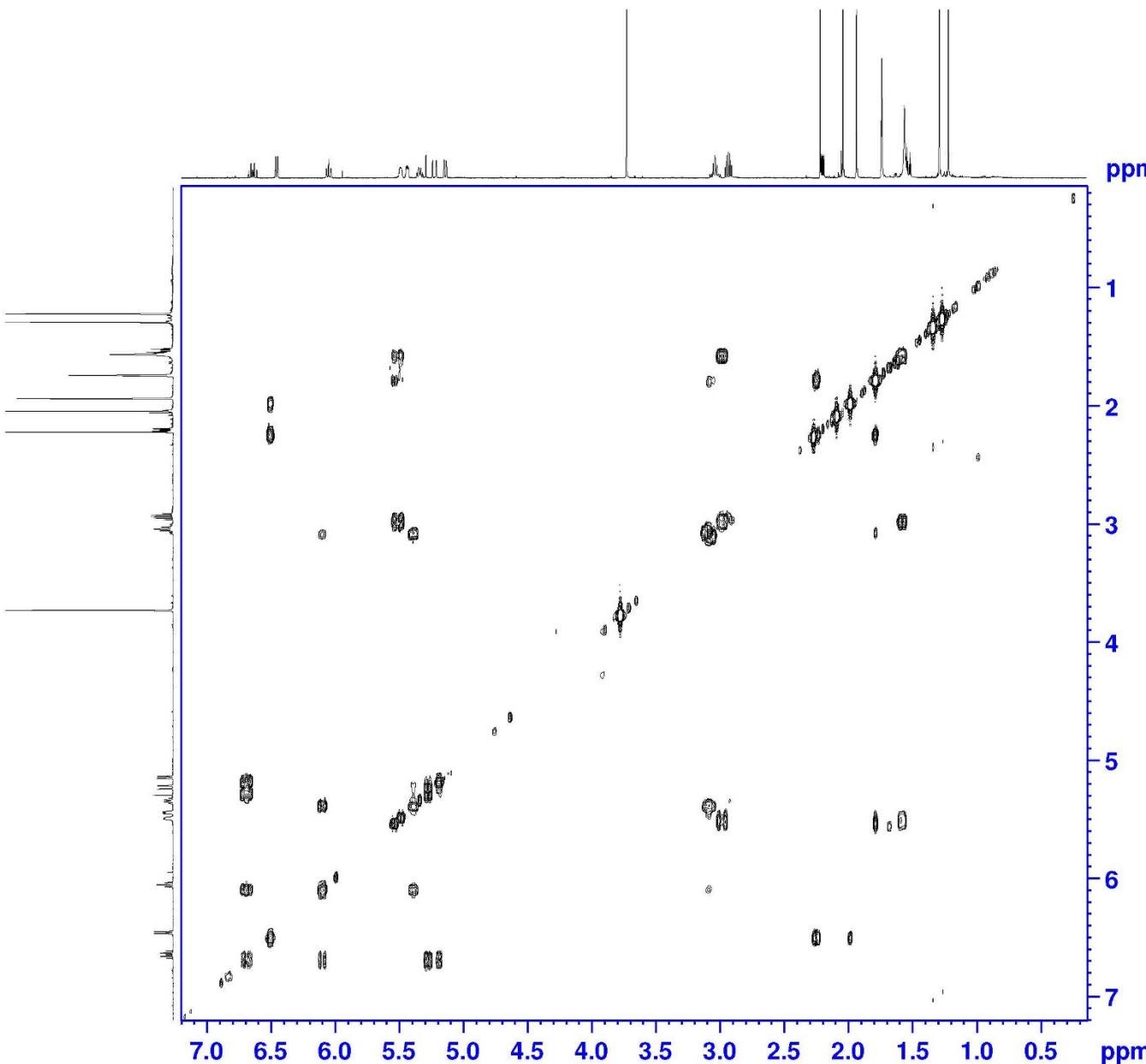
Current Data Parameters
NAME 20190220_PII_Acetate_MAJOR
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190220
Time 20.14
INSTRUM spect
PROBID 5 mm PABBO BB/
PULPROG zgppg30
TD 65536
SOLVENT CDCl₃
NS 2048
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 2050
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 150.9178988 MHz
NUC1 13C
PL 12.00 usec
PLW1 80.00000000 W

===== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPBG[2] waltz16
PCPD2 70.00 usec
PLW2 17.00000000 W
PLW12 0.63230002 W
PLW13 0.30983001 W

F2 - Processing parameters
SI 32768
SF 150.9027925 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME 20190220 PII Acetate MAJOR
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190220
 Time 17.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppcf
 TD 2048
 SOLVENT CDCl3
 NS 10
 DS 8
 SWH 4237.288 Hz
 FIDRES 2.068988 Hz
 AQ 0.2416640 sec
 RC 161
 DW 118.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00000300 sec
 D1 1.88613105 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00023600 sec

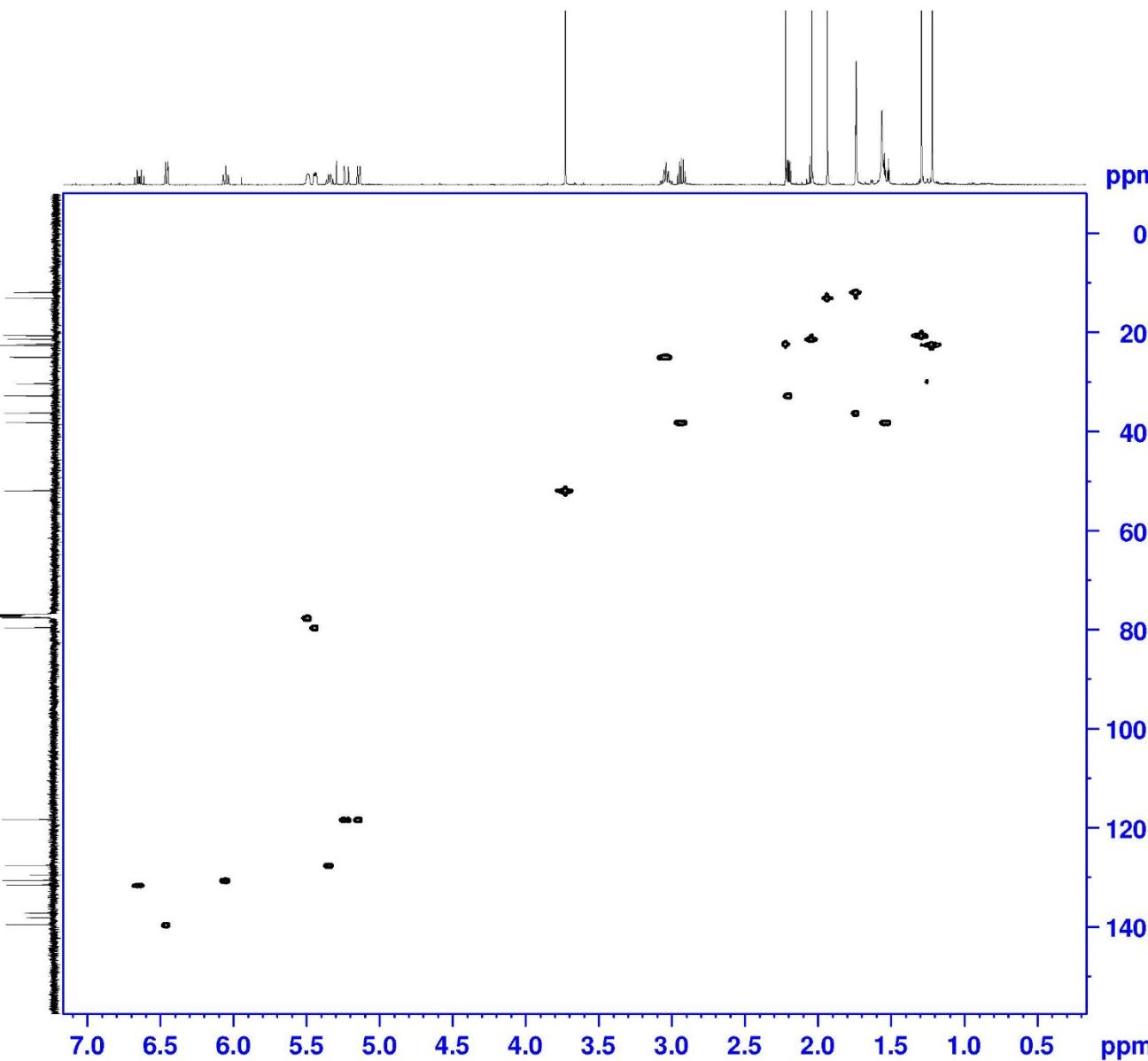
----- CHANNEL f1 -----
 SFO1 600.1322008 MHz
 NUC1 1H
 P0 13.50 usec
 P1 13.50 usec
 P17 2500.00 usec
 PLW1 17.00000000 W
 PLW10 4.58319998 W

----- GRADIENT CHANNEL -----
 GPNAME[1] SMSQ10.100
 GPZ1 10.00 %
 F16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 600.1322 MHz
 FIDRES 33.103813 Hz
 SW 7.061 ppm
 FnMODE QP

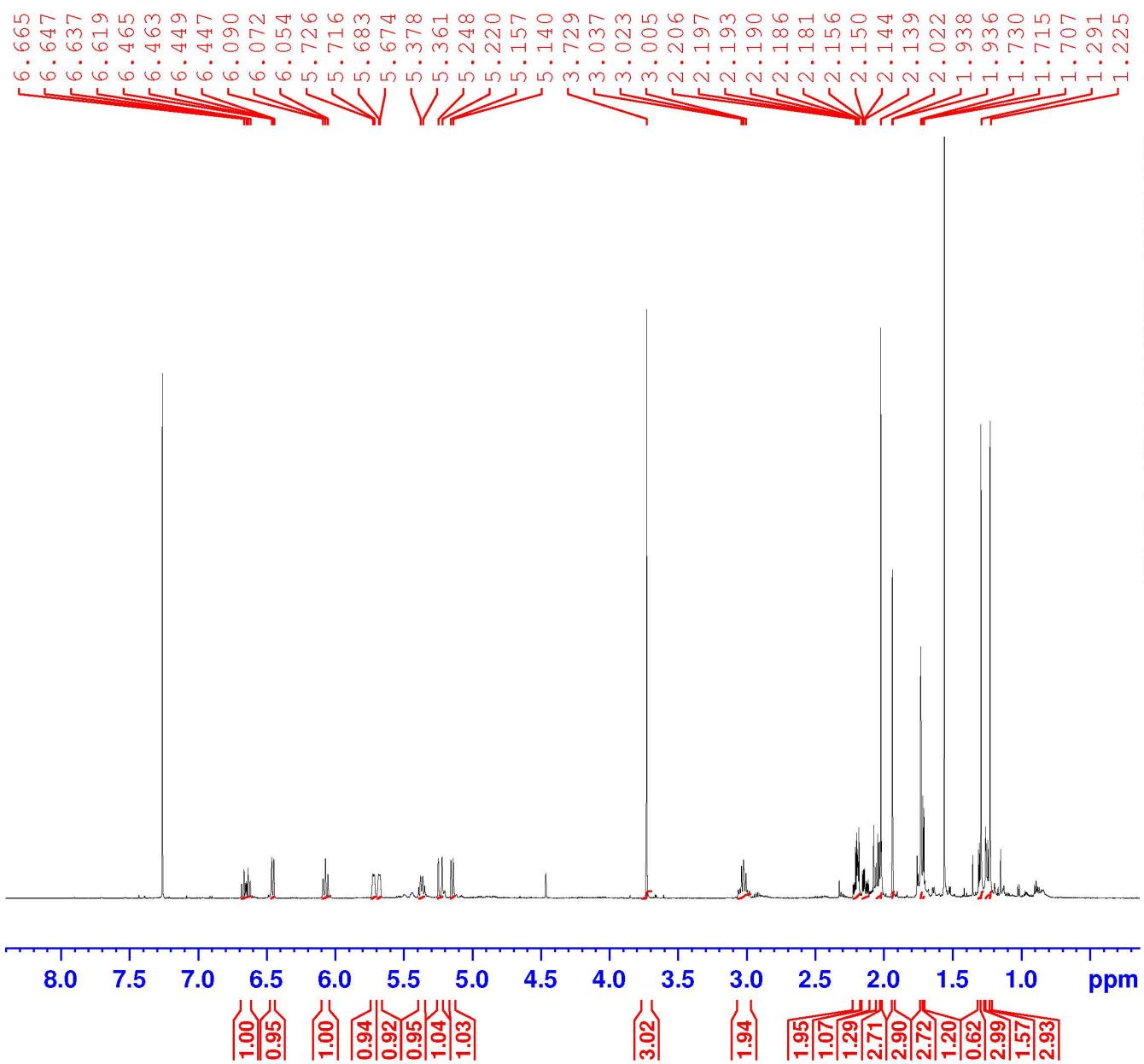
F2 - Processing parameters
 SI 1024
 SP 600.1299971 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QP
 SP 600.1299966 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



4S-(5b) NMR Characterisation

40

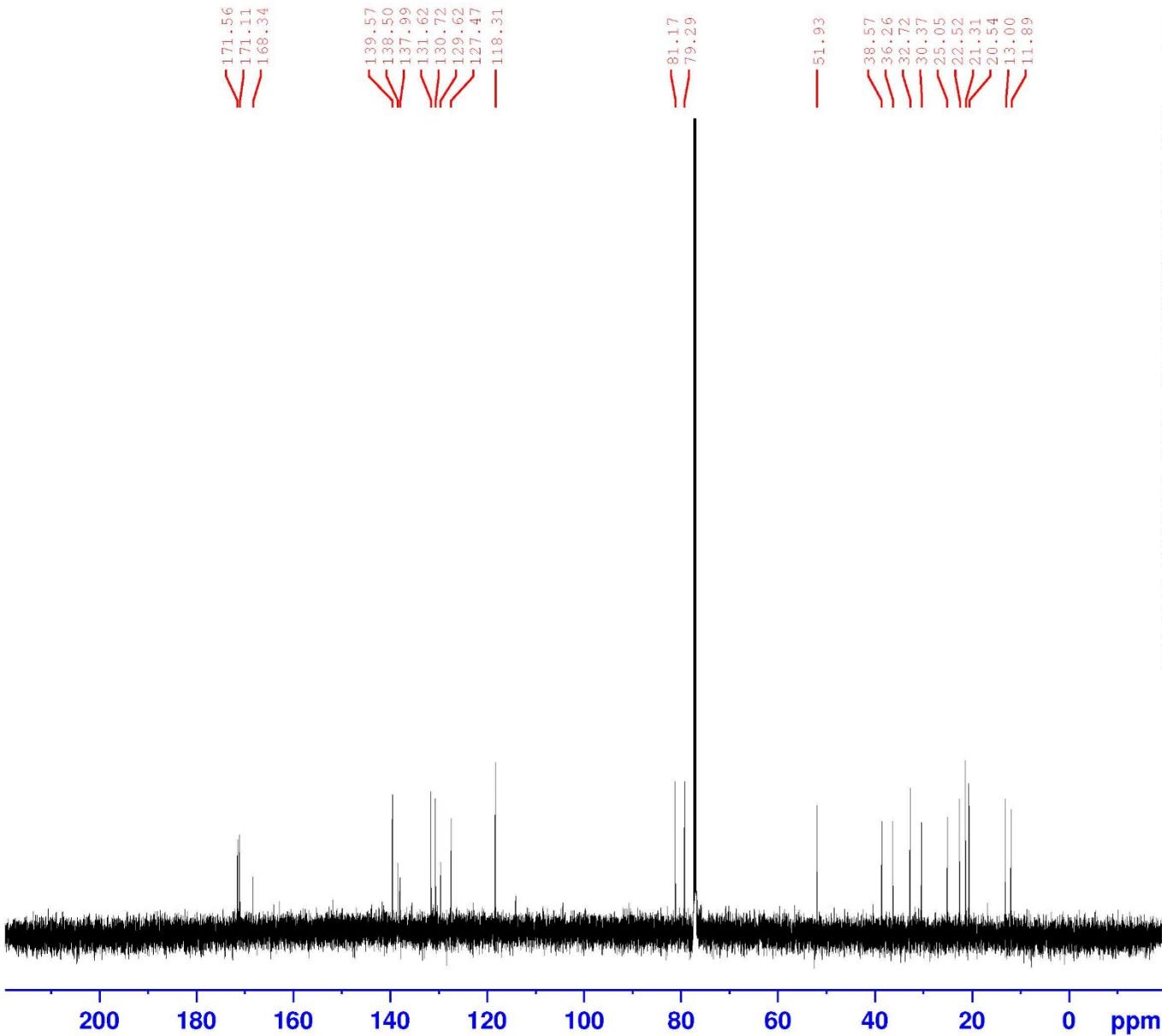


Current Data Parameters
NAME 20190226 Pyrethrin II Acetate Minor
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190226
Time 17.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 287
DW 41.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.1337061 MHz
NUC1 1H
P1 13.50 usec
PEW1 17.0000000 W

F2 - Processing parameters
SI 65536
SF 600.1300261 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME 20190226_Pyrethrin II Acetate Minor
 EXPNO 22
 PROCNO 1

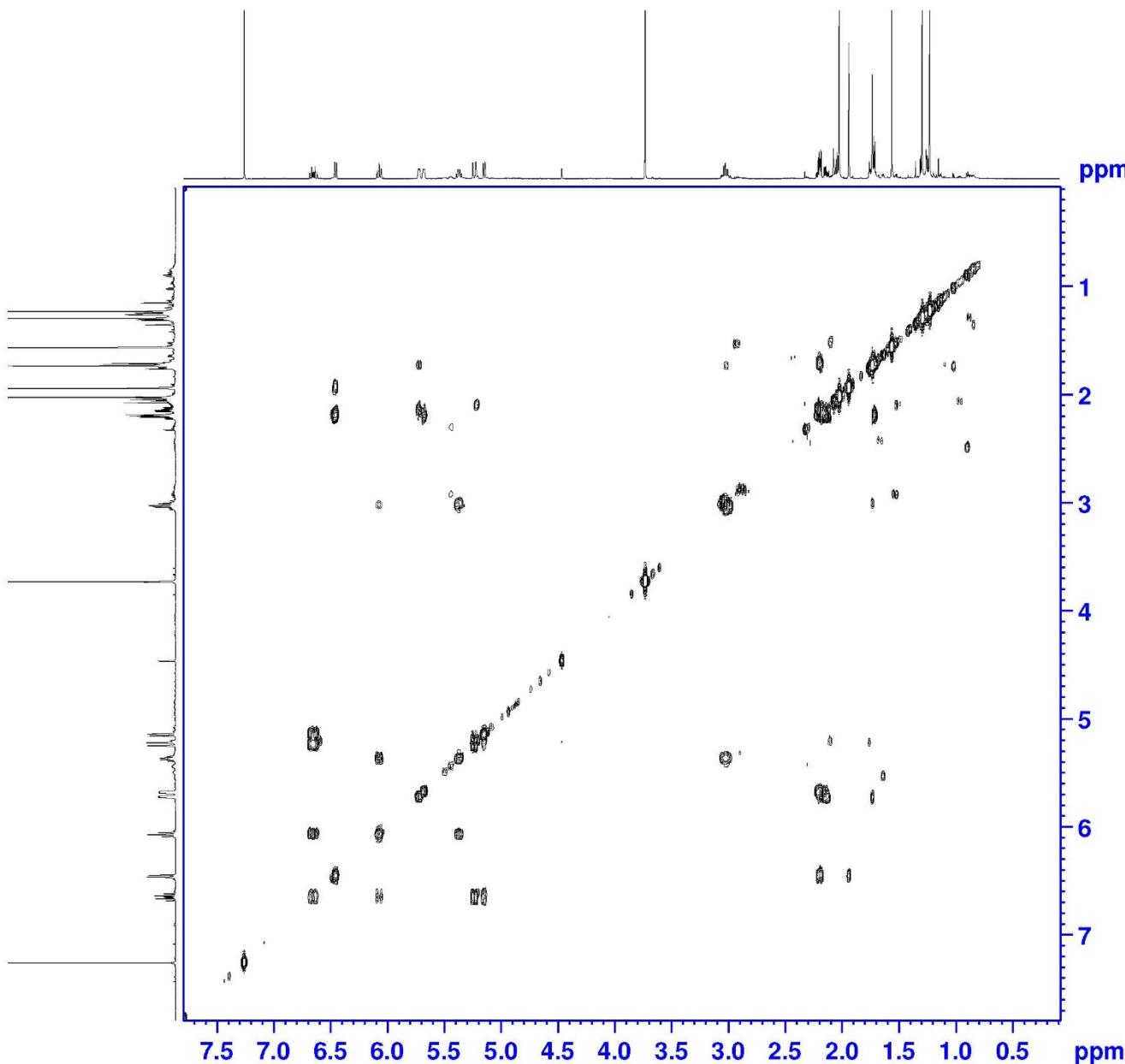
F2 - Acquisition Parameters

Date_ 20190226
 Time_ 21.20
 INSTRUM spect
 PROBHD 5 mm PARBO BB/
 DULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 3096
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDR 1

----- CHANNEL f1 -----
 SFO1 150.9178986 MHz
 NUC1 1H
 PI 12.00 usec
 PLW1 80.0000000 W

----- CHANNEL f2 -----
 SFO2 600.1324005 MHz
 NUC2 1H
 CDPFRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.0000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9027906 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40



Current Data Parameters
NAME 20190226 Pyrethrin II Acetate Minor
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190226
Time_ 17.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
DULPROG cosygpppr1f
TD 2048
SOLVENT CDCl3
NS 10
DS 8
SWH 4629.629 Hz
FIDRES 2.260561 Hz
AQ 0.2211840 sec
RG 144
DW 108.000 usec
DE 6.50 usec
TE 298.2 K
D0 0.00000300 sec
D1 1.9061097 sec
D11 0.0300000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00021600 sec

CHANNEL f1

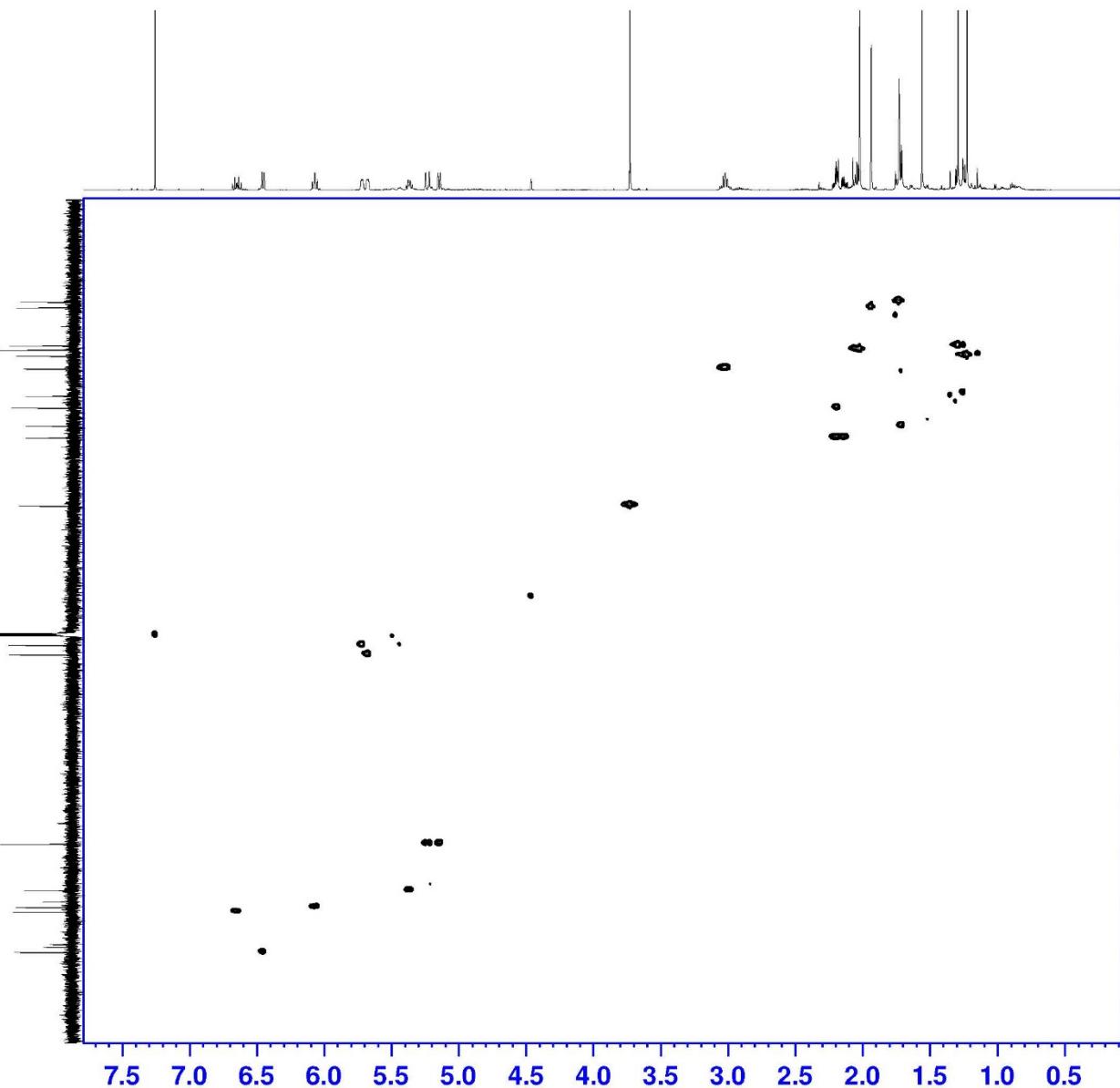
SFO1 600.1323879 MHz
NUC1 1H
P0 13.50 usec
P1 13.50 usec
P17 2500.00 usec
PLW1 17.00000000 W
PLW10 4.58319998 W

===== GRADIENT CHANNEL =====
GPNAME[1] SNSQ10.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 600.1324 MHz
FIDRES 36.168980 Hz
SW 7.714 ppm
PwMODE QF

F2 - Processing parameters
SI 1024
SF 600.1300249 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 0F
SF 600.1300256 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0



Current Data Parameters
 NAME 20190226 Pyrethrin II Acetate Minor
 EXPNO 24
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20190226
 Time_ 21:28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 DULPROG hsqcetgps

TD 1024

SOLVENT CDCl3

NS 50

DS 16

SWH 4629.629 Hz

FIDRES 4.521122 Hz

AQ 0.1105920 sec

RG 2050

DW 108.000 usec

DE 6.50 usec

TE 298.3 K

CNST2 145.0000000

D0 0.00000300 sec

D1 1.45494401 sec

D4 0.00172414 sec

D11 0.03000000 sec

D16 0.00020000 sec

D10 0.00002000 sec

ZGOPTNS

===== CHANNEL f1 =====

SFO1 600.1323855 MHz

NUC1 1H

P1 13.50 usec

P2 27.00 usec

P28 1000.00 usec

PLW1 17.0000000 W

===== CHANNEL f2 =====

SFO2 150.9140636 MHz

NUC2 13C

CPDPRG[2] garp

P3 12.00 usec

P4 24.00 usec

PCPD2 60.00 usec

PLW2 80.00000000 W

PLW12 3.20000005 W

===== GRADIENT CHANNEL =====

GP[NAM[1]] SMSQ10.100

GP[NAM[2]] SMSQ10.100

GP11 80.00 %

GP12 20.10 %

P16 1000.00 usec

P1 - Acquisition parameters

TD 256

SFO1 150.9141 MHz

FIDRES 97.656250 Hz

SW 165.657 ppm

FnMODE Echo-Antiecho

F2 - Processing parameters

SI 1024

SF 600.1300250 MHz

WDW QSIMINE

SSB 2

LB 0 Hz

GB 0

PC 1.40

P1 - Processing parameters

SI 1024

M2 echo-antiecho

SF 150.9028175 MHz

WDW QSIMINE

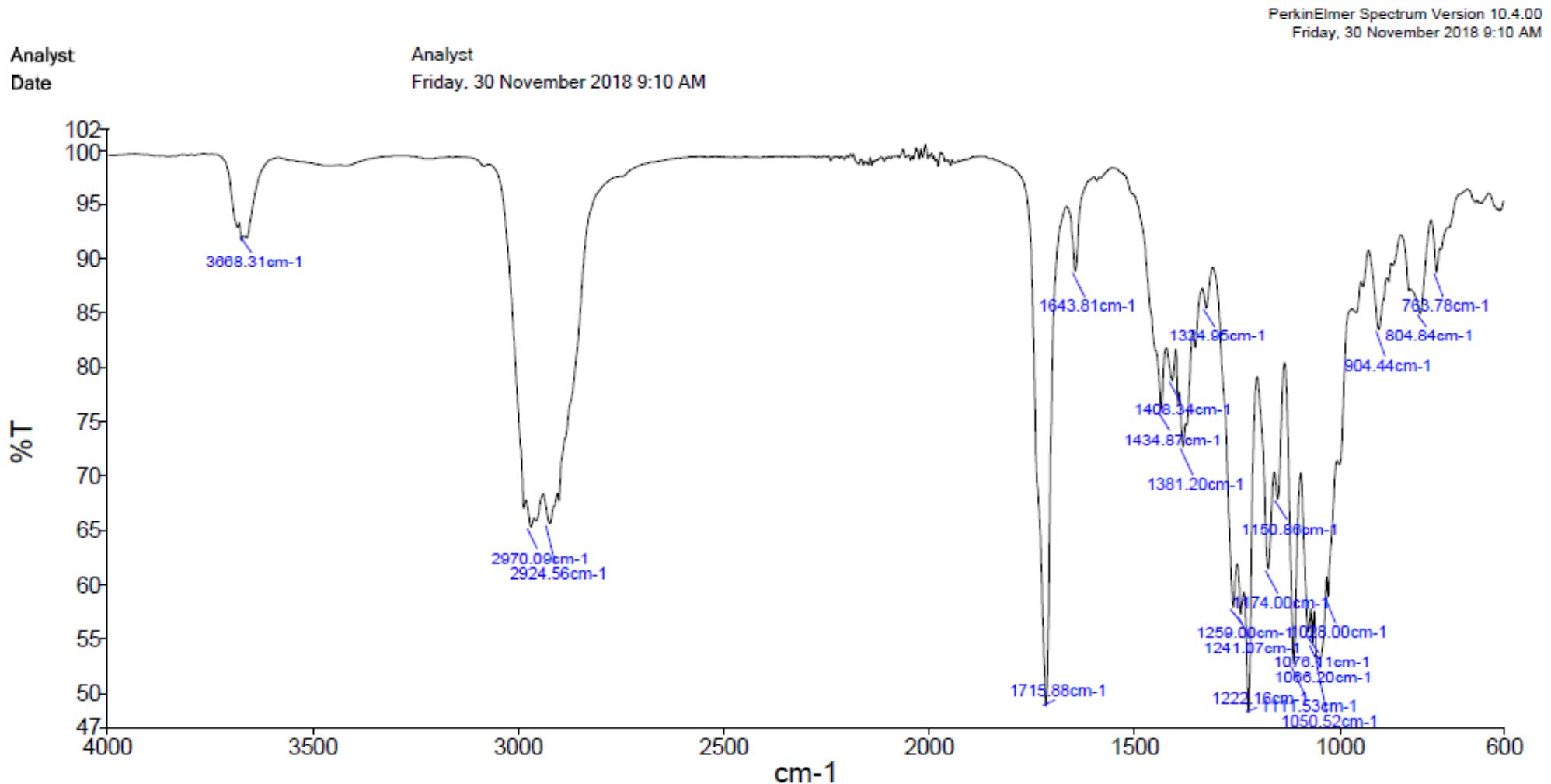
SSB 2

LB 0 Hz

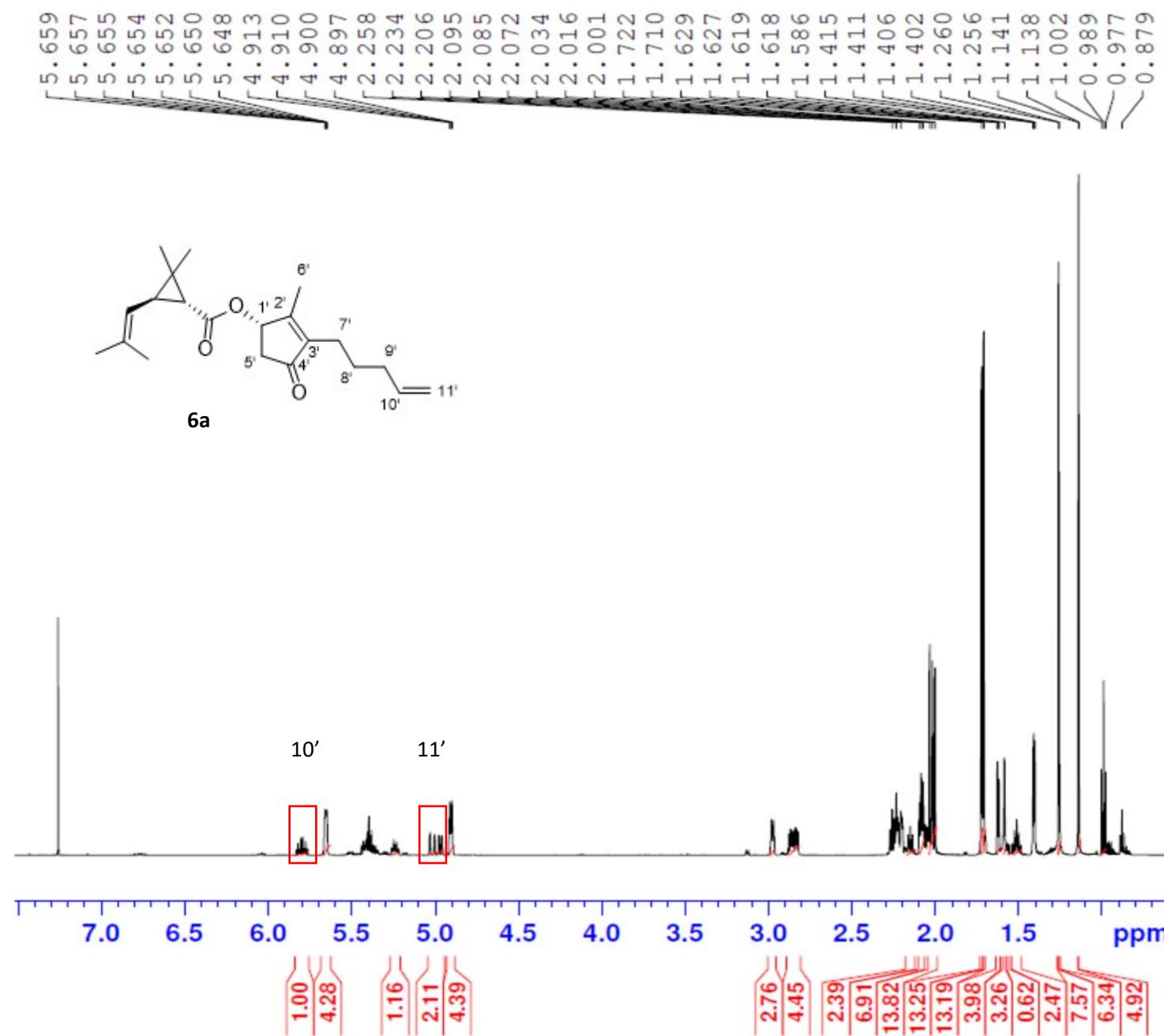
GB 0

(5b) IR Spectrum

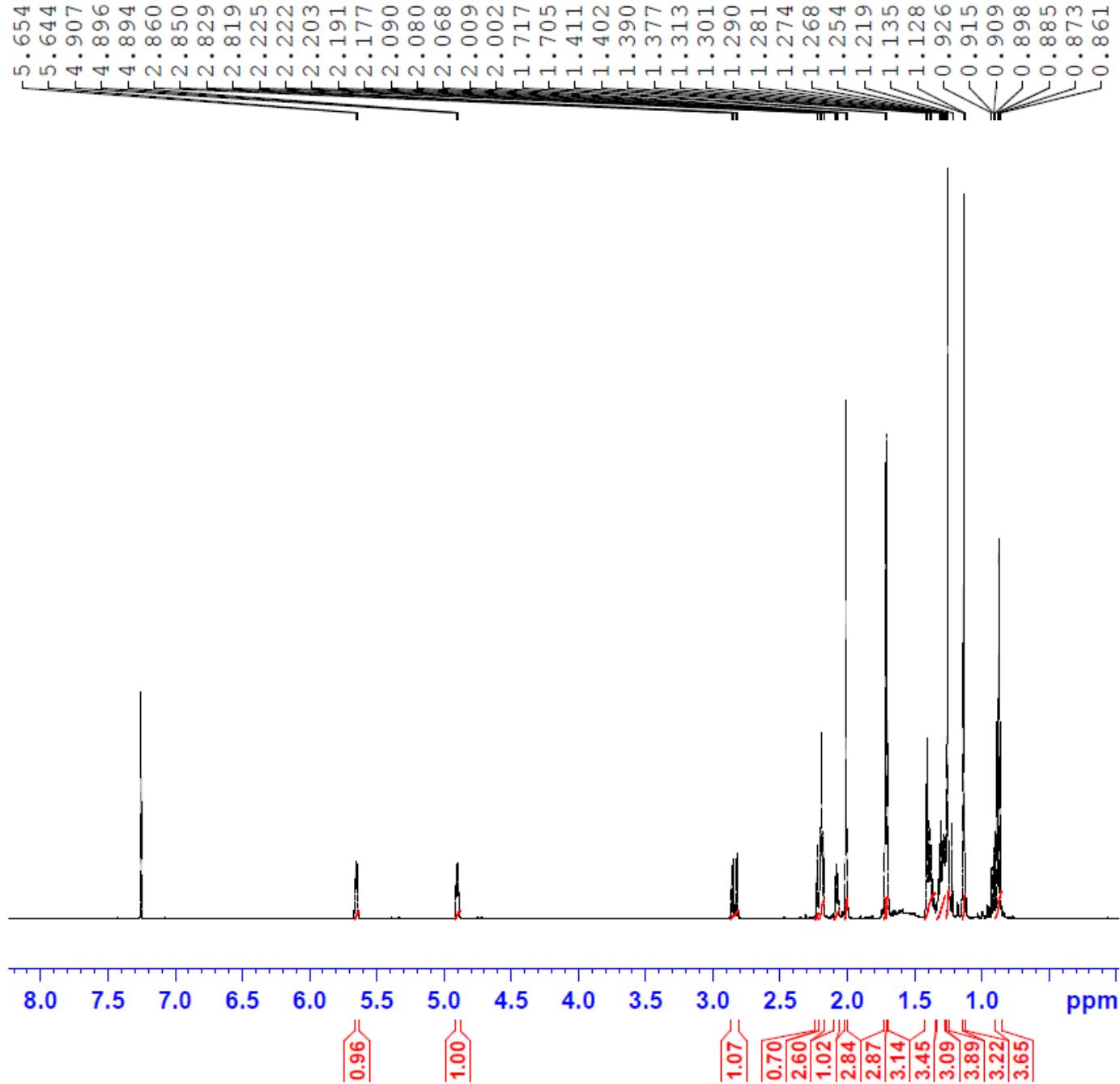
44



Representative ^1H NMR spectrum of partially hydrogenated pyrethrin I (**1a**) showing characteristic peaks for compound **6a**



(7a) NMR Characterisation

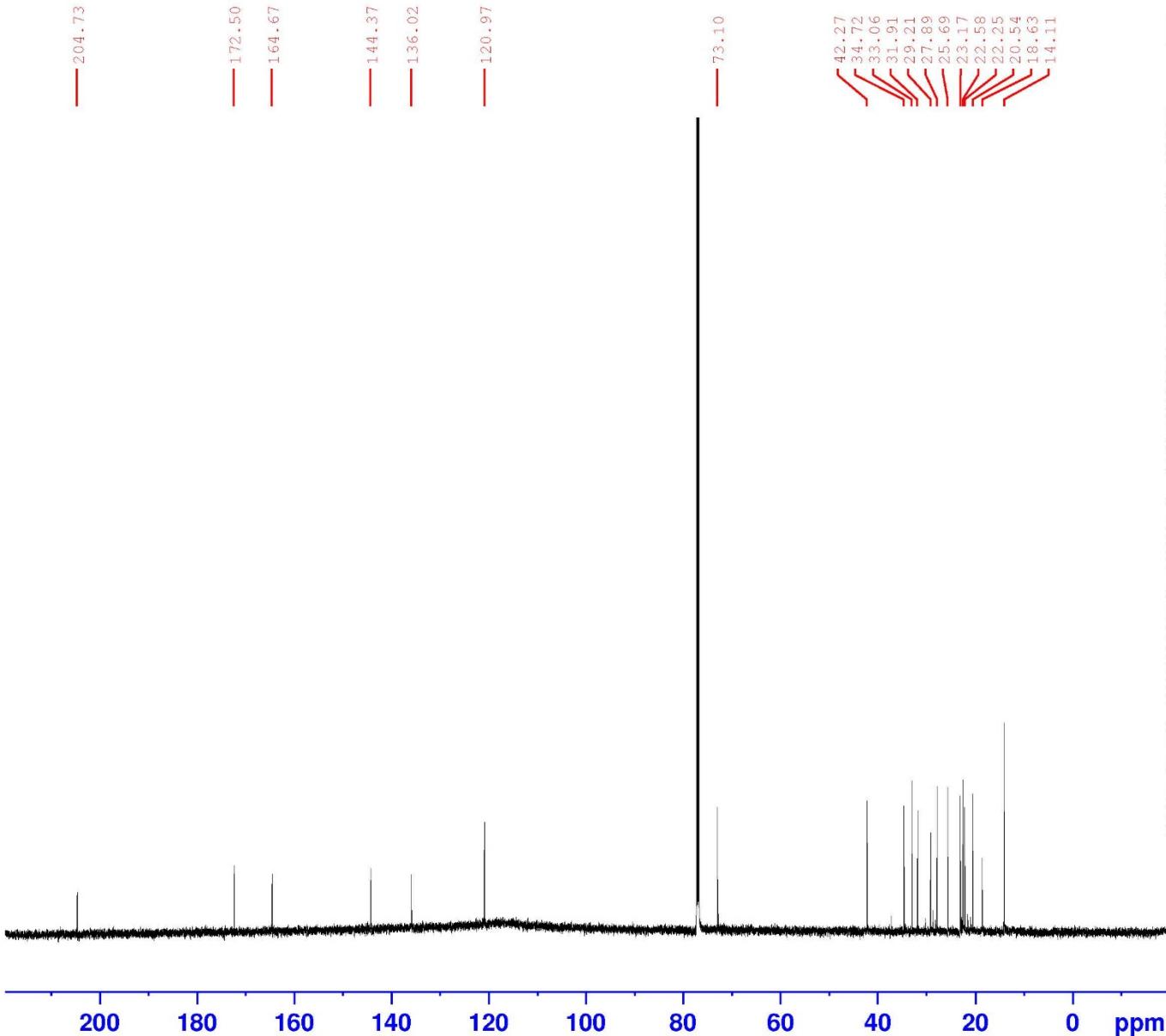


Current Data Parameters
 NAME 20180904 Hydrogenation PI
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180904
 Time 18.28
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 161
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 8.40 usec
 PLW1 12.55000019 W

F2 - Processing parameters
 SI 65536
 SF 600.1300257 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



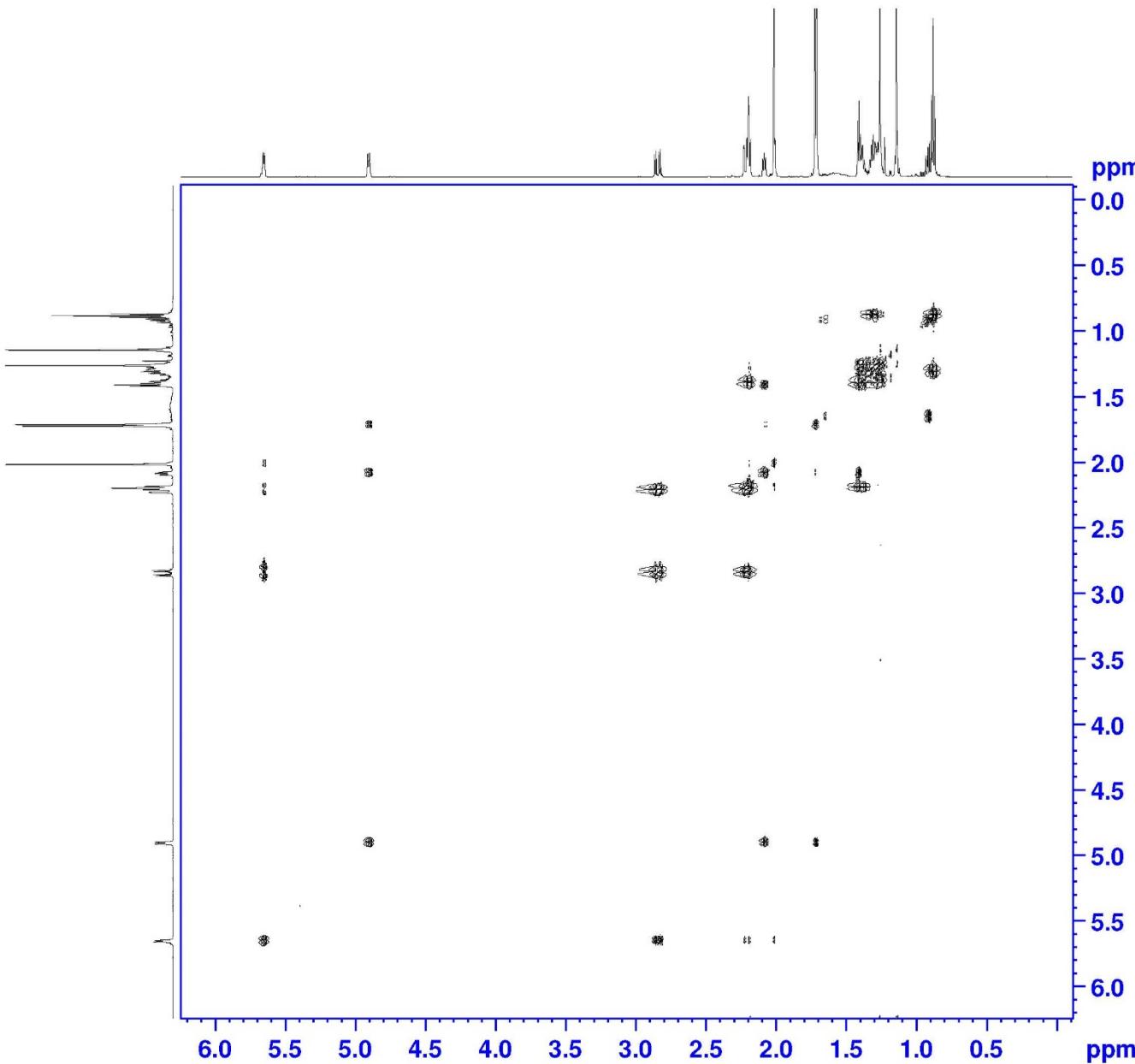
Current Data Parameters
 NAME 20180904 Hydrogenation PI
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180904
 Time 20.13
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 1820
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 16.00 usec
 PLW1 110.76000214 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPMGR[2] waltz16
 PCPD2 70.00 usec
 PLW2 12.55000019 W
 PLW12 0.18072000 W
 PLW13 0.08855400 W

F2 - Processing parameters
 SI 32768
 SF 150.9027920 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 20180904 Hydrogenation PI
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date 20180904
 Time 20.20
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG cosygpmfhhpp
 TD 2048
 SOLVENT CDCl3
 NS 5
 DS 4
 SWH 3816.794 Hz
 FIDRES 1.863669 Hz
 AQ 0.2682880 sec
 RG 2050
 DW 131.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00012030 sec
 D1 1.87015700 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D16 0.00020000 sec
 IN0 0.00026200 sec

===== CHANNEL f1 =====
 SF01 600.1318677 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 P17 2500.00 usec
 PLW1 12.55000019 W
 PLW10 1.30999994 W

===== GRADIENT CHANNEL =====
 GPNAME[1] SMSQ10.100
 GPNAME[2] SMSQ10.100
 GPZ1 10.00 %
 GPZ2 20.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 256
 SF01 600.1319 MHz
 FIDRES 14.909351 Hz
 SW 6.360 ppm
 F1MODE States-TPPI

F2 - Processing parameters
 SI 1024
 SF 600.1300271 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 States-TPPI
 SF 600.1300277 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0



Current Data Parameters
NAME 20180904 Hydrogenation PI
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180904
Time_ 21.10
INSTRUM spect
PROBHD 5 mm PABBT1H/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 50
DS 16
SWH 3816.794 Hz
FIDRES 3.727338 Hz
AQ 0.1341440 sec
RG 2050
DW 131.000 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.000000
D0 0.00000300 sec
D1 1.43139195 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002000 sec
ZGOPTNS

===== CHANNEL f1 =====
SFO1 600.1318677 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P2B 1000.00 usec
PLW1 12.55000019 W

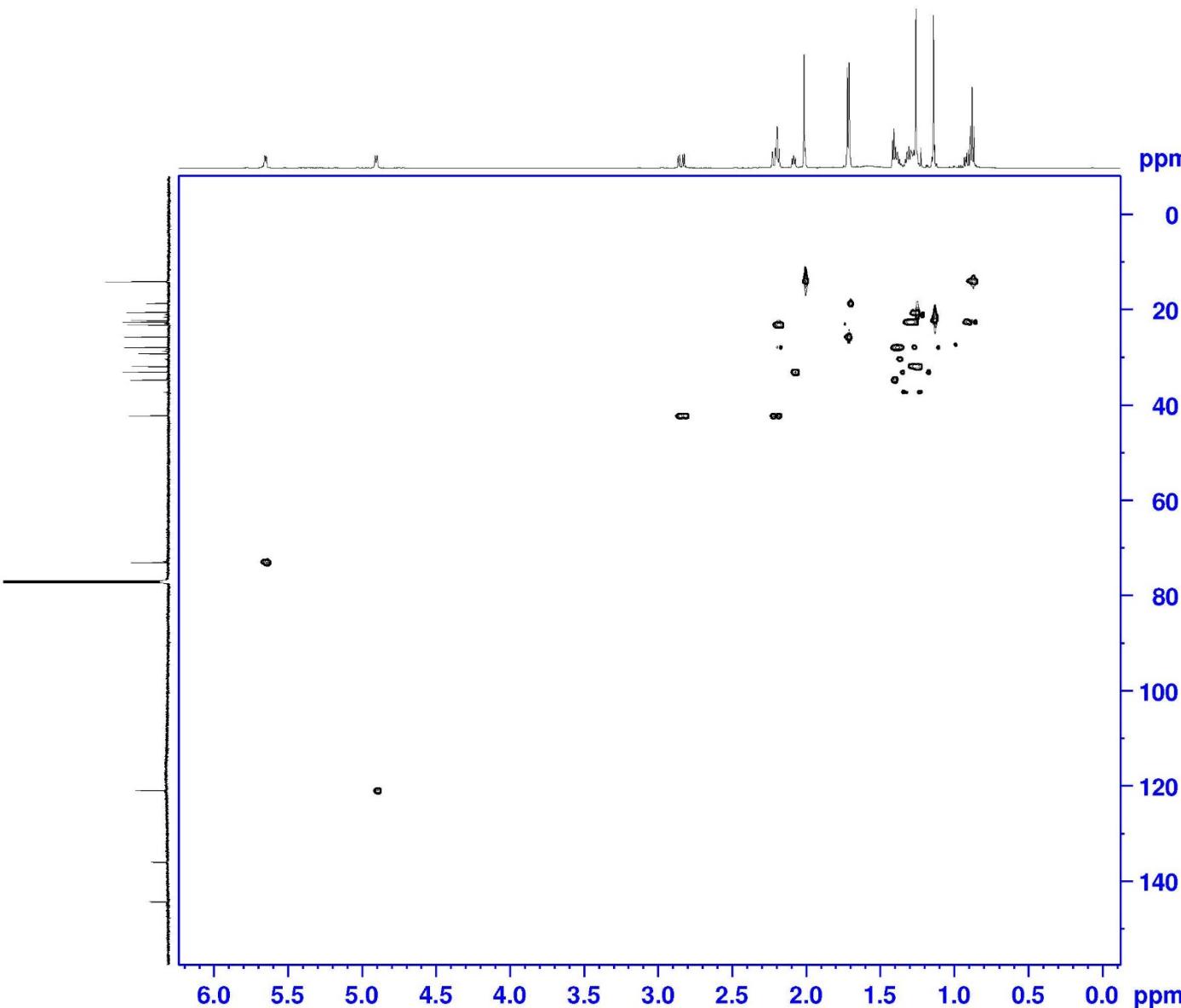
===== CHANNEL f2 =====
SFO2 150.9140636 MHz
NUC2 13C
CPDPFRG[2] garp
P3 16.00 usec
P4 32.00 usec
PCPD2 60.00 usec
PLM2 110.76000214 W
PLW2 7.87610006 W

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

F1 - Acquisition parameters
TD 256
SFO1 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PwMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 600.1300315 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 150.9027851 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0



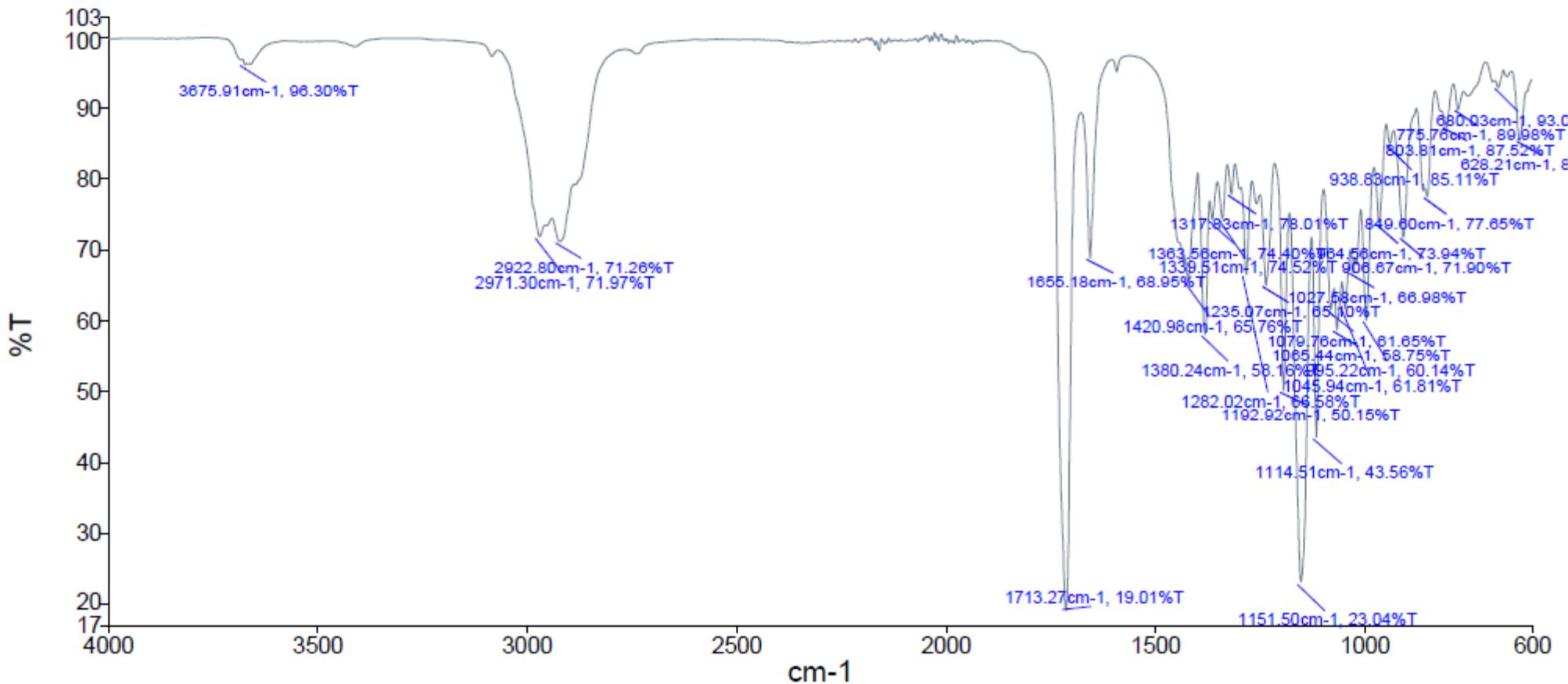
(7a) IR Spectrum

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PerkinElmer Spectrum Version 10.4.2
Wednesday, 17 October 2018 3:00 PM

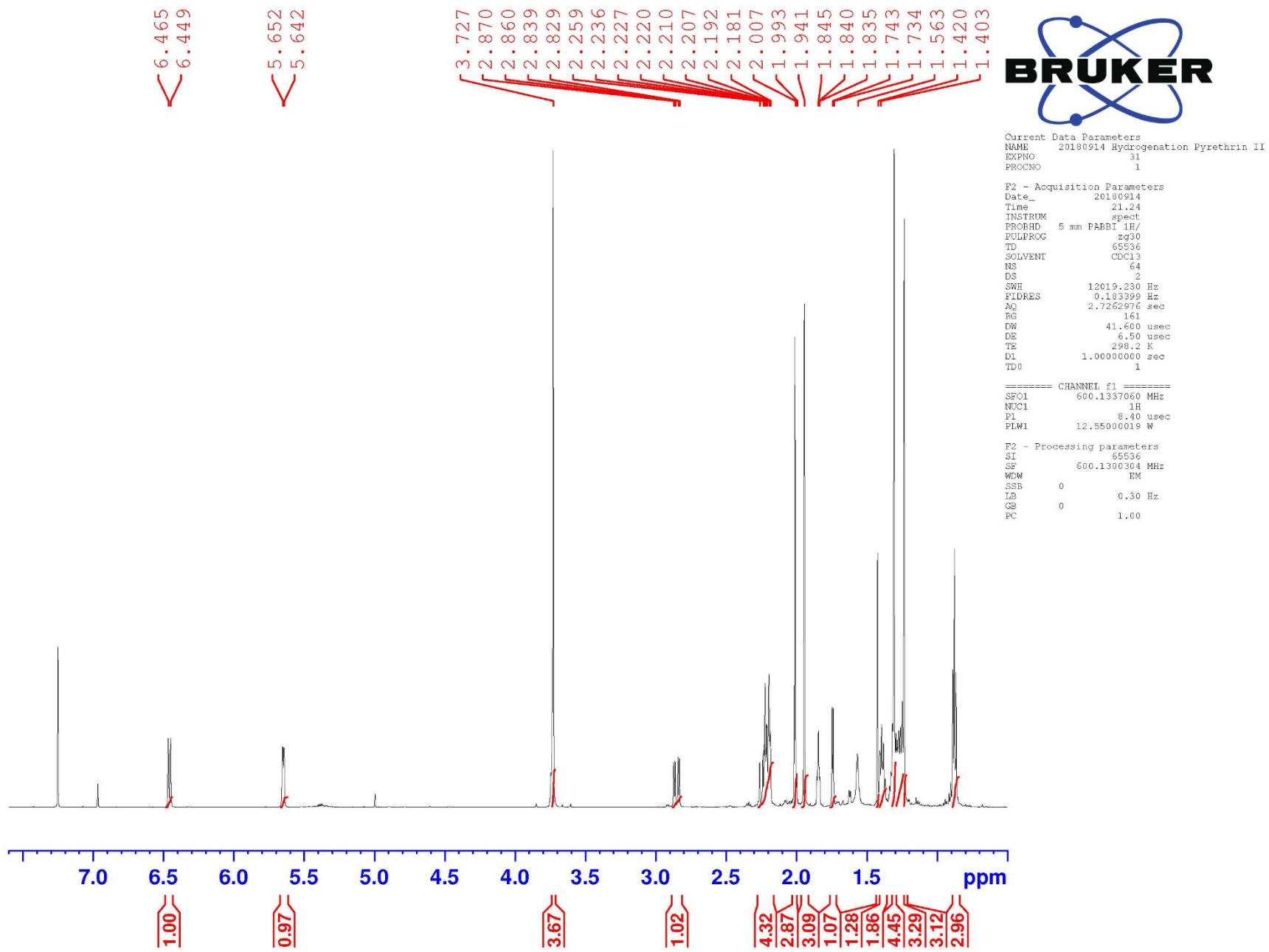
Analyst:
Date:

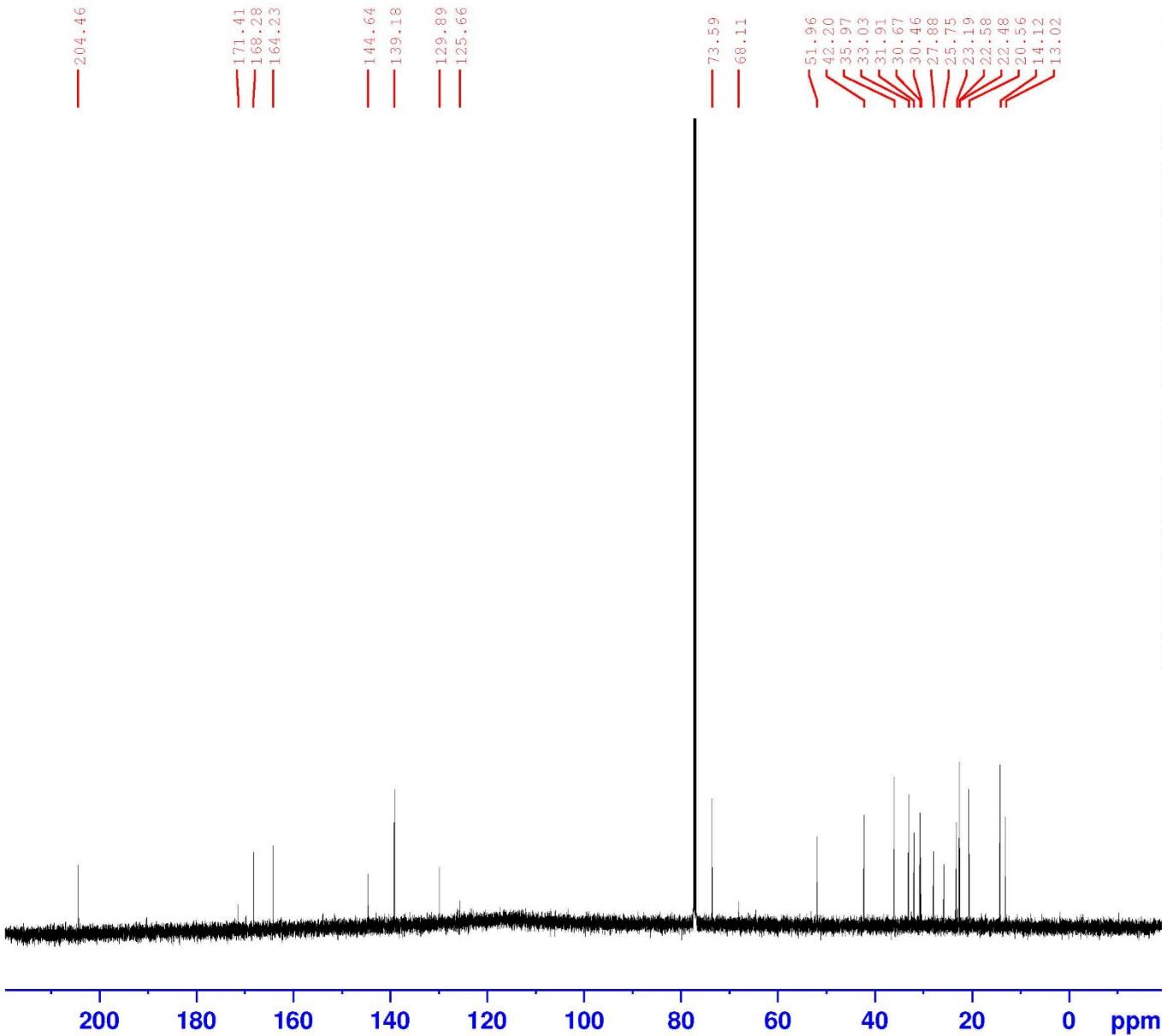
Analyst:
Wednesday, 17 October 2018 3:00 PM



(7b) NMR Characterisation

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Current Data Parameters
 NAME 20180914 Hydrogenation Pyrethrin II
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20180914
 Time_ 21.08
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 DULPROG zgppg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 1440
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

----- CHANNEL f1 -----

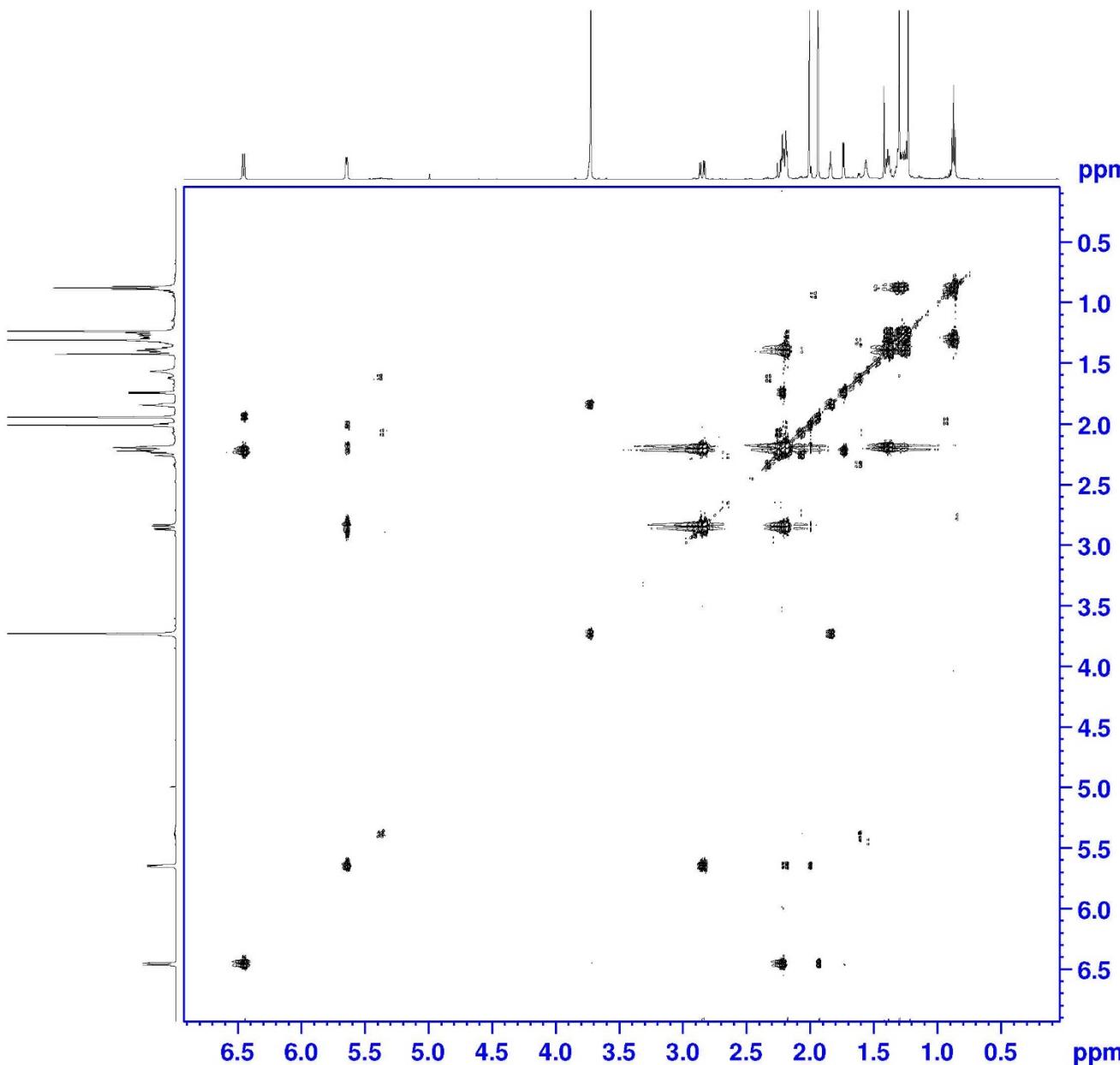
SFO1 150.9178985 MHz
 NUC1 1H
 PC 16.00 usec
 PLW1 110.76000214 W

----- CHANNEL f2 -----

SFO2 600.1324005 MHz
 NUC2 1H
 CDPFRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 12.55000019 W
 PLW12 0.18072000 W
 PLW13 0.02855400 W

F2 - Processing parameters

SI 32768
 SF 150.9027904 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40



Current Data Parameters
NAME 20180914 Hydrogenation Pyrethrin II
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180914
Time_ 21:35
INSTRUM spect
PROBHD 5 mm PABBI 1H/
DULPROG cosygppmfpchpp
TD 2048
SOLVENT CDCl3
NS 10
DS 4
SWH 4132.231 Hz
FIDRES 2.017691 Hz
AQ 0.2478080 sec
RG 2050
DW 121.000 usec
DE 6.50 usec
TE 298.2 K
D0 0.00011030 sec
D1 1.89063704 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00024200 sec

===== CHANNEL f1 =====

SFO1 600.1321235 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P17 2500.00 usec
PLW1 12.55000019 W
PLW10 1.30999994 W

===== GRADIENT CHANNEL =====

GPNAME[1] SMSQ10.100

GPNAME[2] SMSQ10.100

GPZ1 10.00 %

GPZ2 20.00 %

P16 1000.00 usec

F1 - Acquisition parameters

TD 256
SFO1 600.1321 MHz

FIDRES 16.141529 Hz

SW 6.886 ppm

PRMODE States-TPPI

F2 - Processing parameters

SI 1024

SF 600.1300327 MHz

WDW QSINE

SSB 2

LB 0 Hz

GB 0

PC 1.40

F1 - Processing parameters

SI 1024

NC2 States-TPPI

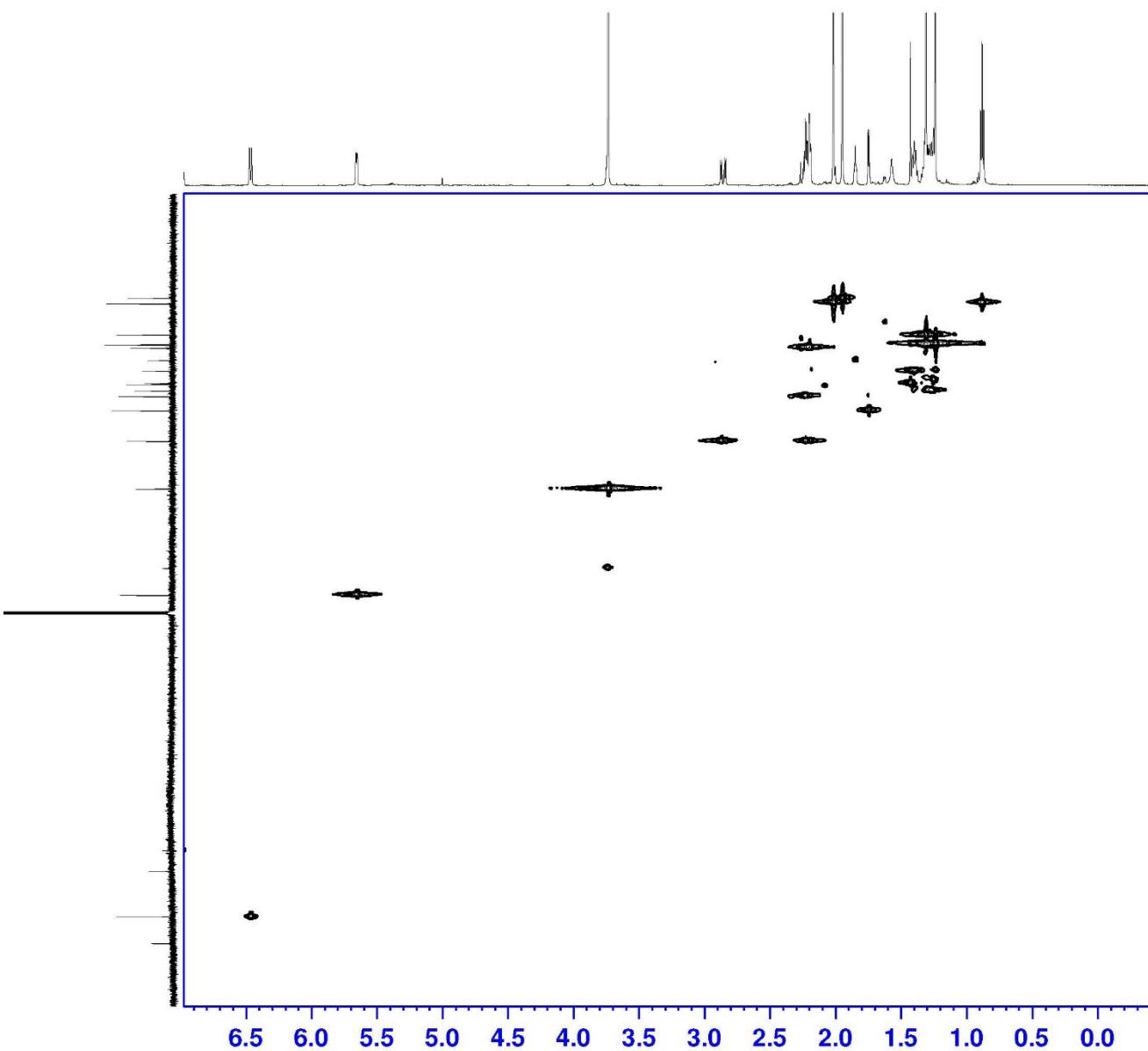
SF 600.1300295 MHz

WDW QSINE

SSB 2

LB 0 Hz

GB 0



Current Data Parameters
NAME 20180914 Hydrogenation Pyrethrin II
EXPNO 34
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180914
Time_ 23.37
INSTRUM spect
PROBHD 5 mm PABBI 1H/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 50
DS 16
SWH 4464.286 Hz
FIDRES 4.359654 Hz
AQ 0.1146880 sec
RG 2050
DW 112.000 usec
DE 6.50 usec
TE 298.0 K
CNSST2 145.000000
D0 0.00000300 sec
D1 1.45084798 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002000 sec
ZGOPTNS

----- CHANNEL f1 -----

SFO1 600.1319837 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P28 1000.00 usec
PLW1 12.55000019 W

----- CHANNEL f2 -----
SFO2 150.9140636 MHz
NUC2 13C
CPDPGRG[2] garp
P3 16.00 usec
P4 32.00 usec
PCPD2 60.00 usec
PLW2 110.76000214 W
PLW12 7.87610006 W

----- GRADIENT CHANNEL -----
GP[NAM[1]] SMSQ10.100
GP[NAM[2]] SMSQ10.100
GP11 80.00 %
GP12 20.10 %
P16 1000.00 usec

P1 - Acquisition parameters
TD 256
SFO1 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PmODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 600.1300288 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

P1 - Processing parameters
SI 1024
M2 echo-antiecho
SF 150.9028085 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

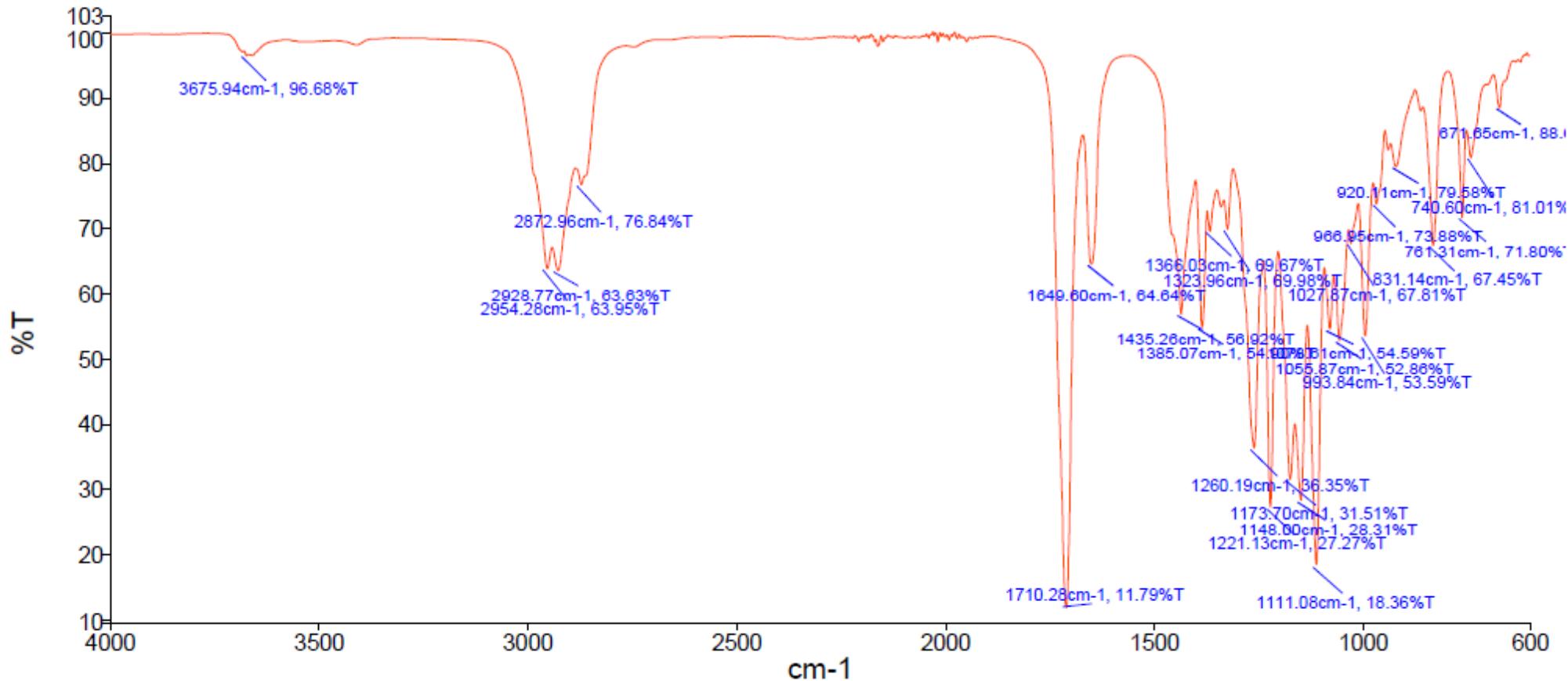
(7b) IR Spectrum

55

PerkinElmer Spectrum Version 10.4.2
Wednesday, 17 October 2018 3:01 PM

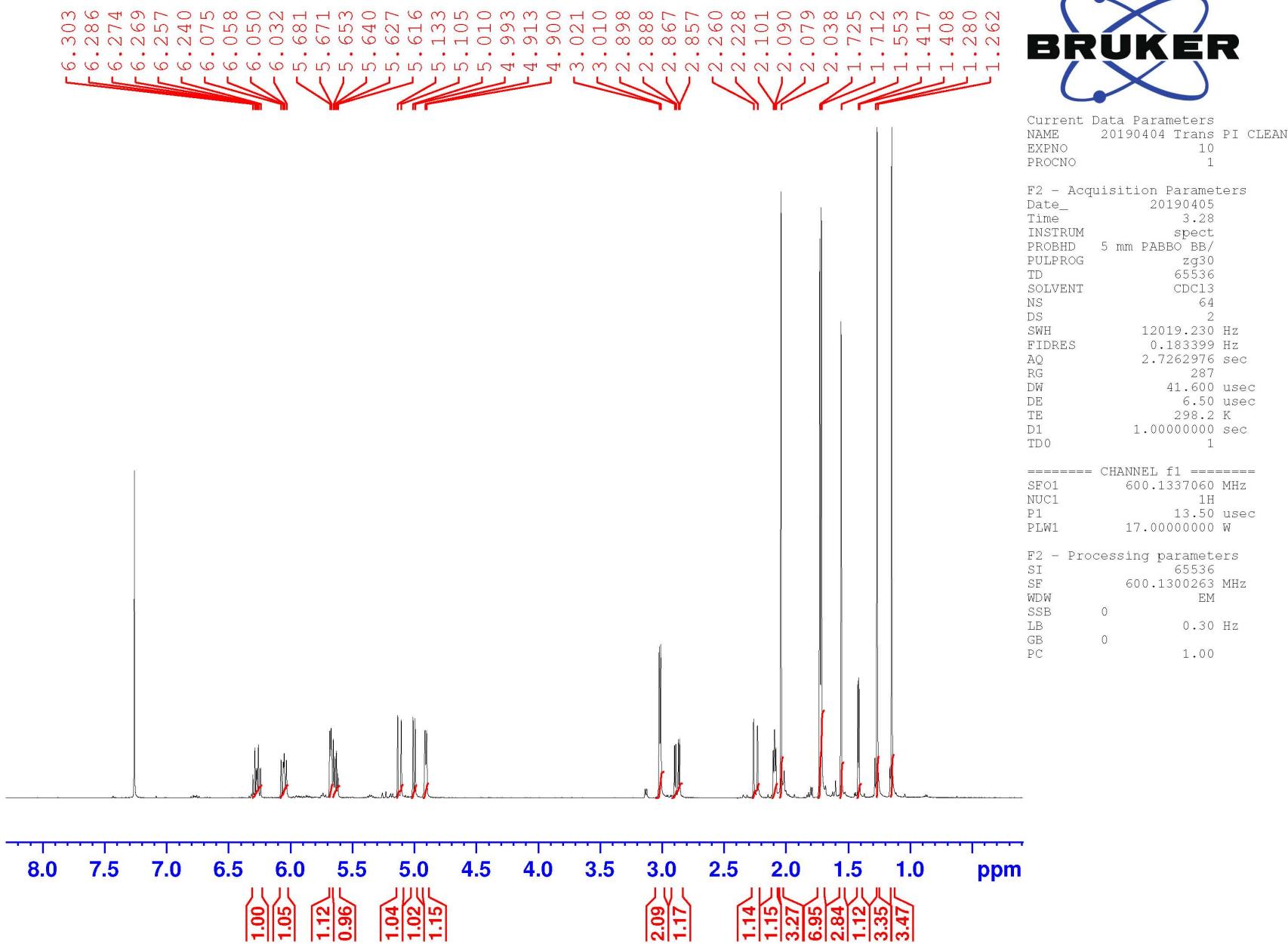
Analyst:
Date:

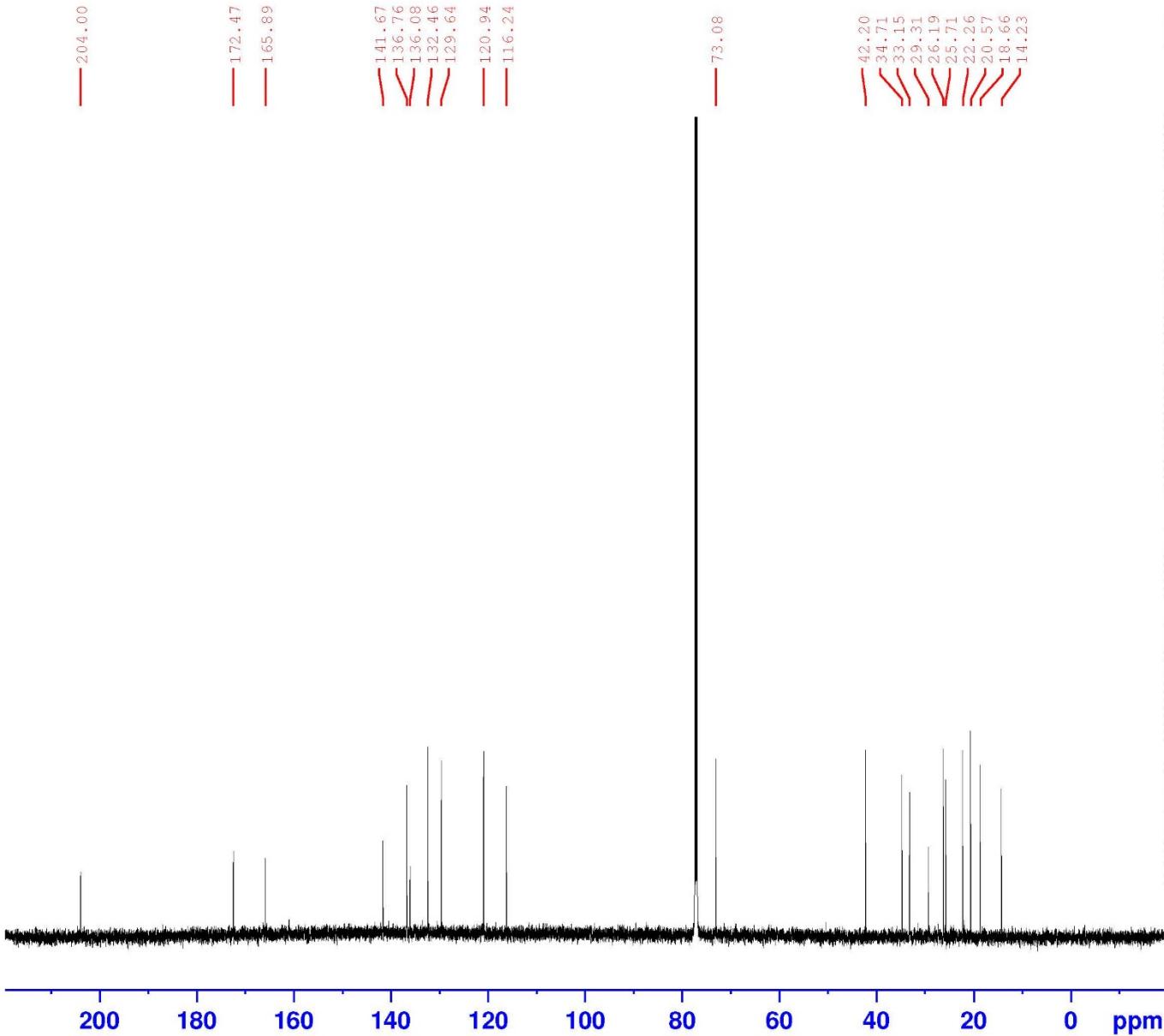
Analyst:
Wednesday, 17 October 2018 3:01 PM



(8a) NMR Characterisation

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Current Data Parameters
 NAME 20190404 Trans PI CLEAN
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190405
 Time 5.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 80.0000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.00000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9027893 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 20190404 Trans PI CLEAN
 EXPNO 11
 PROBNO 1

F2 - Acquisition Parameters
 Date_ 20190405
 Time 3.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppcf
 TD 2048
 SOLVENT CDCl3
 NS 10
 DS 8
 SWH 4545.455 Hz
 FIDRES 2.219460 Hz
 AQ 0.2252800 sec
 RC 144
 DW 110.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00000300 sec
 D1 1.90251505 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00022000 sec

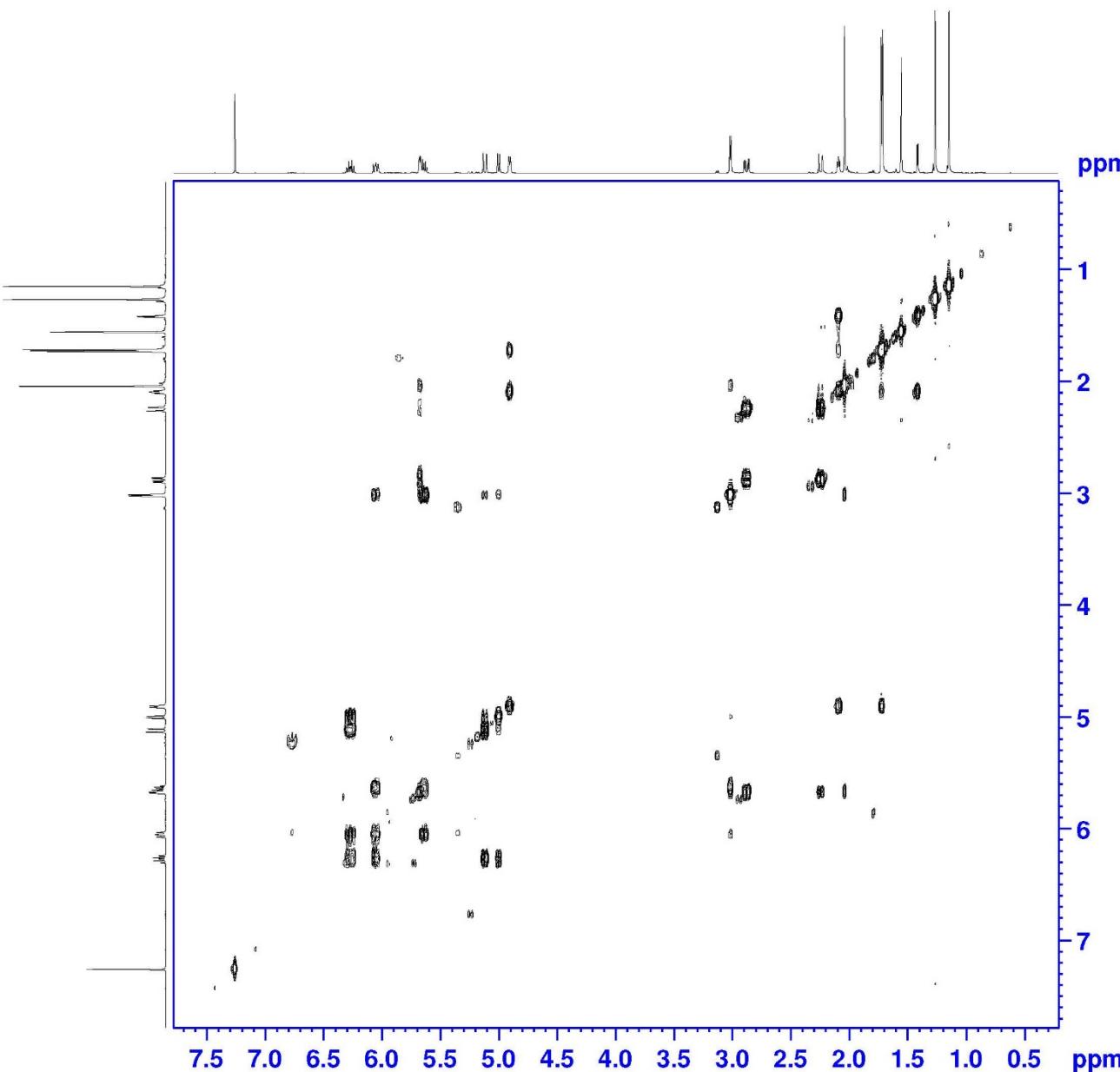
----- CHANNEL f1 -----
 SFO1 600.1324241 MHz
 NUC1 1H
 P0 13.50 usec
 P1 13.50 usec
 P17 2500.00 usec
 PLW1 17.00000000 W
 PLW10 4.58319998 W

----- GRADIENT CHANNEL -----
 GPNAME[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 600.1324 MHz
 FIDRES 35.511364 Hz
 SW 7.574 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SP 600.1300250 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SP 600.1300264 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0





Current Data Parameters
 NAME 20190404 Trans PI CLEAN
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20190405
 Time_ 6.14
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 DULPROG hsqcetgps
 TD 1024
 SOLVENT CDCl3
 NS 50
 DS 16
 SWH 4464.286 Hz
 FIDRES 4.359654 Hz
 AQ 0.1146880 sec
 RG 2050
 DW 112.000 usec
 DE 6.50 usec
 TE 298.2 K
 CNST2 145.000000
 D0 0.0000300 sec
 D1 1.45084798 sec
 D4 0.00172414 sec
 D11 0.0300000 sec
 D16 0.0002000 sec
 IN0 0.00002000 sec
 ZGOPTNS

===== CHANNEL f1 =====
 SFO1 600.1324484 MHz
 NUC1 1H
 P1 13.50 usec
 P2 27.00 usec
 P2B 1000.00 usec
 PLW1 17.0000000 W

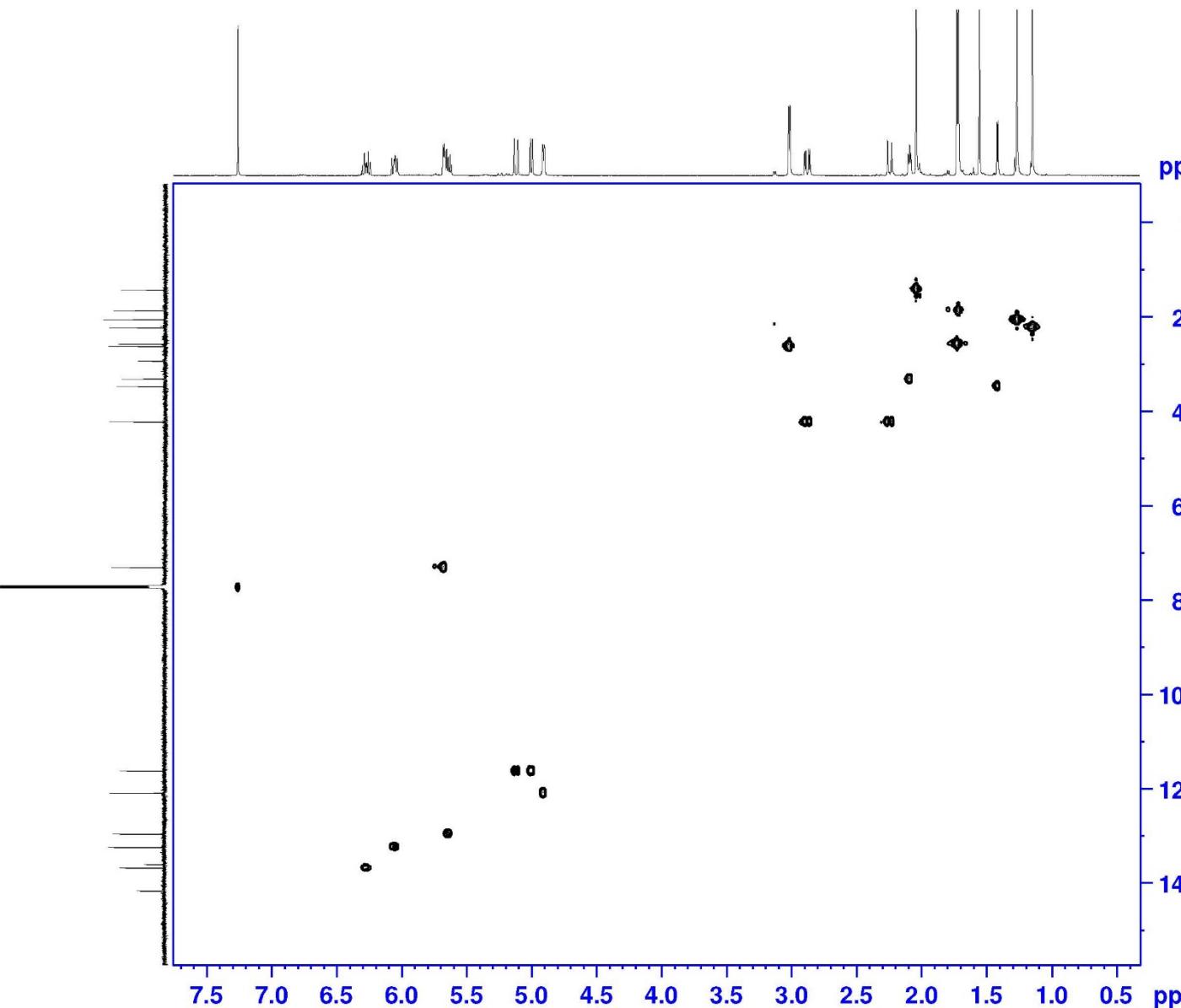
===== CHANNEL f2 =====
 SFO2 150.9140636 MHz
 NUC2 13C
 GDEPRFG[2] garp
 P3 12.00 usec
 P4 24.00 usec
 PCPD2 60.00 usec
 PLM2 80.0000000 W
 PLW2 3.2000005 W

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

P1 - Acquisition parameters
 TD 137
 SFO1 150.9141 MHz
 FIDRES 182.481750 Hz
 SW 165.657 ppm
 PA MODE Echo-Antiecho

F2 - Processing parameters
 SI 1024
 SF 600.1300246 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

P1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 SF 150.9028116 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0



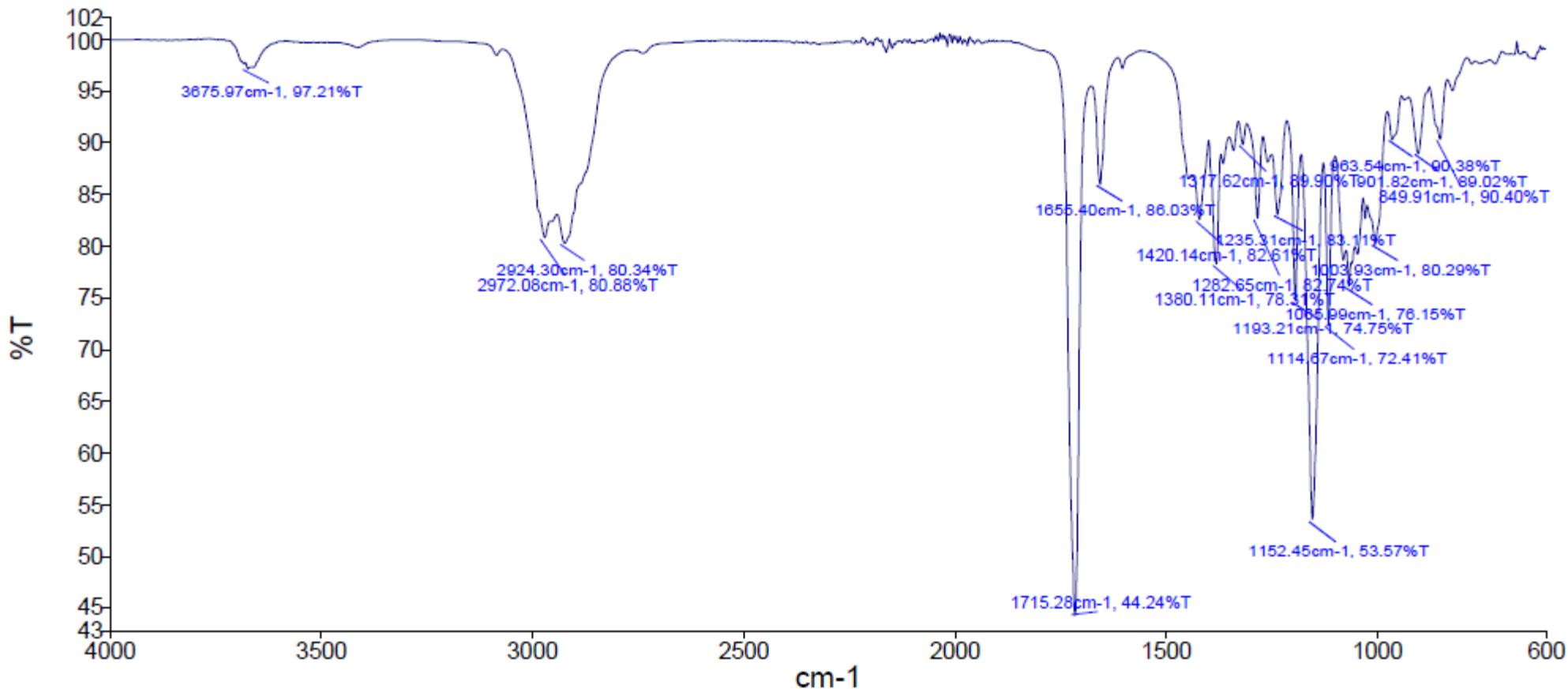
(8a) IR Spectrum

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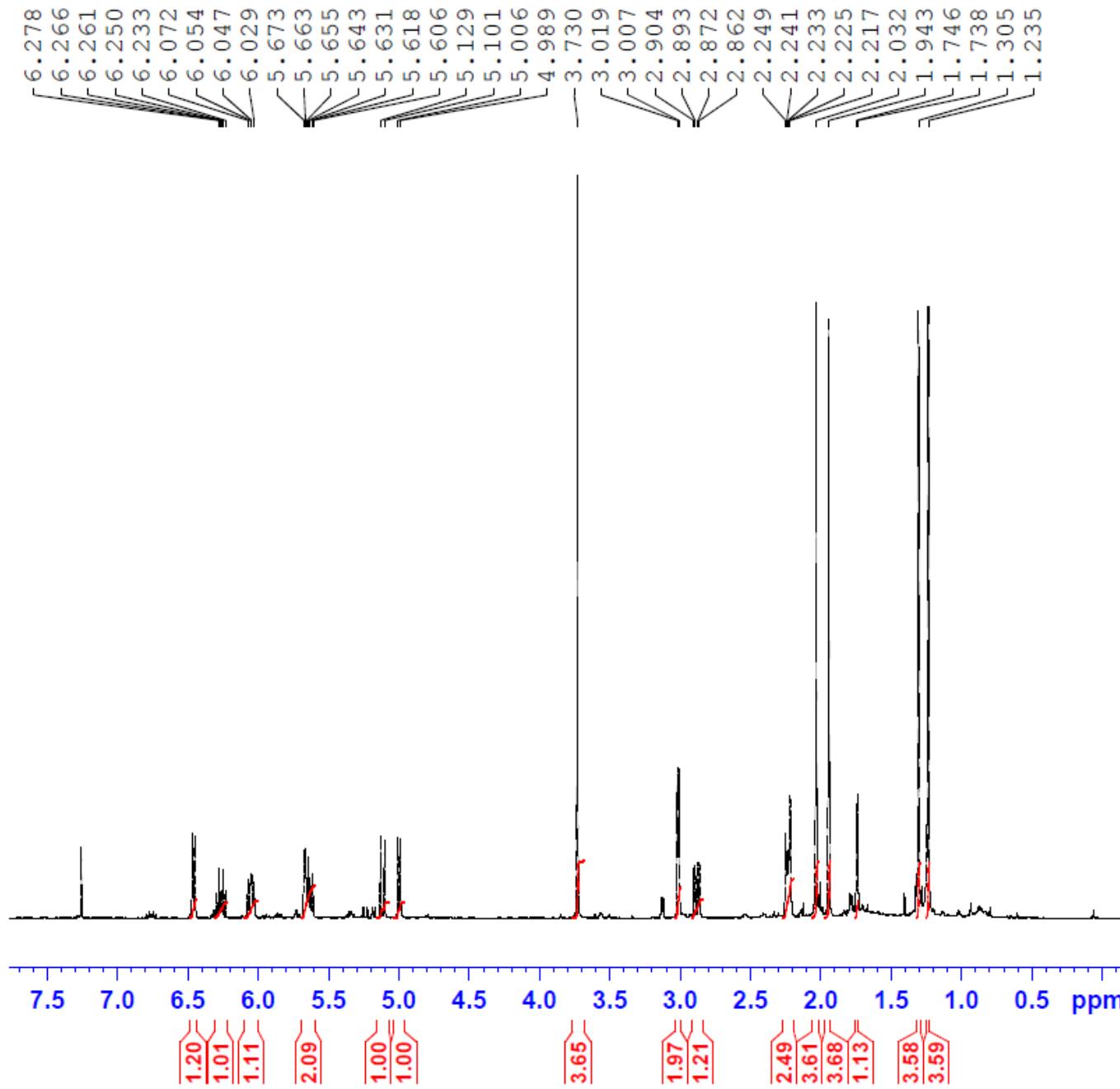
Analyst
Date

Analyst
Wednesday, 17 October 2018 3:01 PM



(8b) NMR Characterisation

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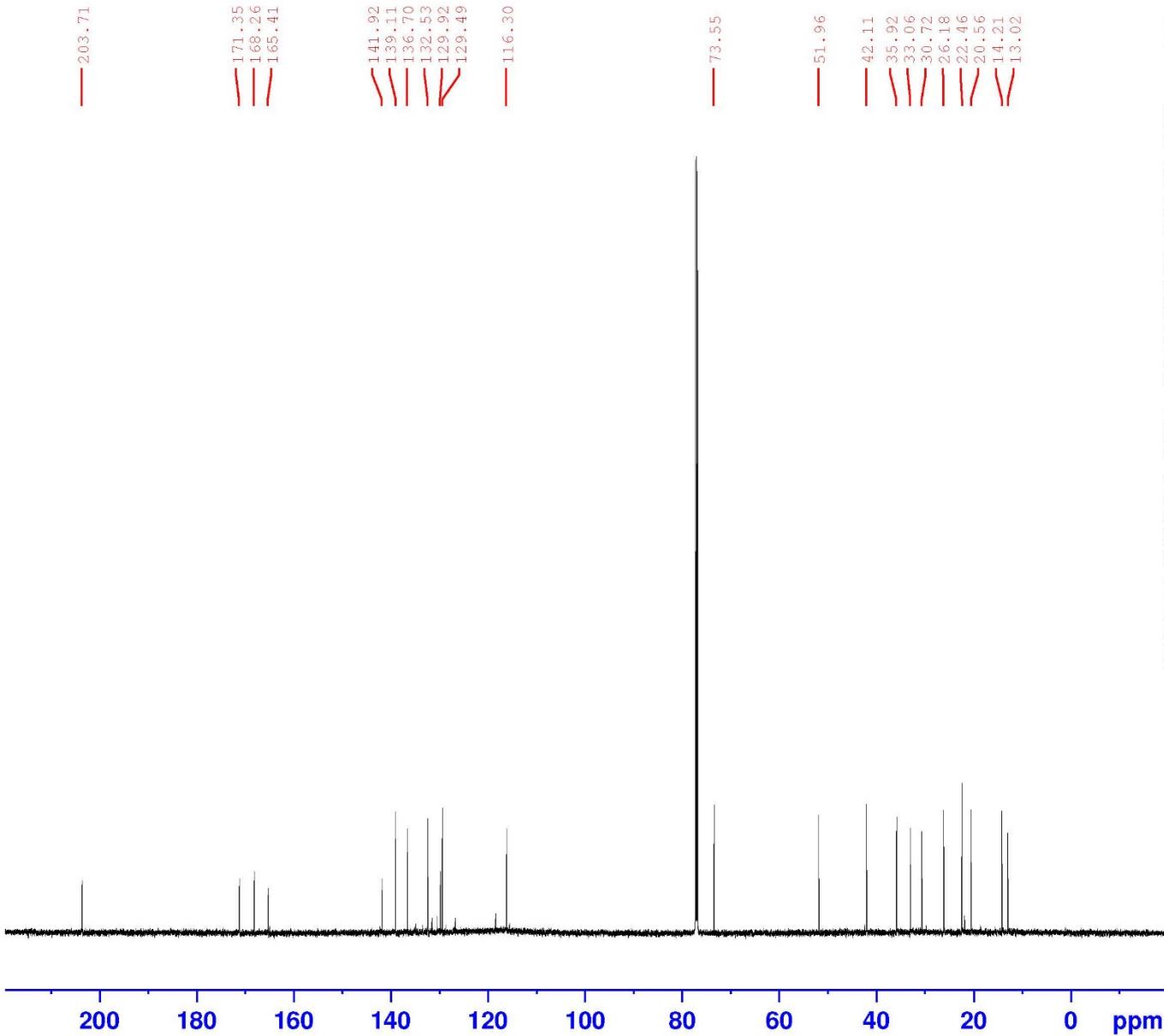


Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date 20181008
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 90.5
DW 41.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 600.1337060 MHz
NUC1 1H
P1 8.40 usec
PLW1 12.55000019 W

F2 - Processing parameters
SI 65536
SF 600.1300270 MHz
WDW EM
SSB 0 0.30 Hz
LB 0
GB 0
PC 1.00



Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters

Date_ 20181008
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBB 1H/
DULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 1620
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----

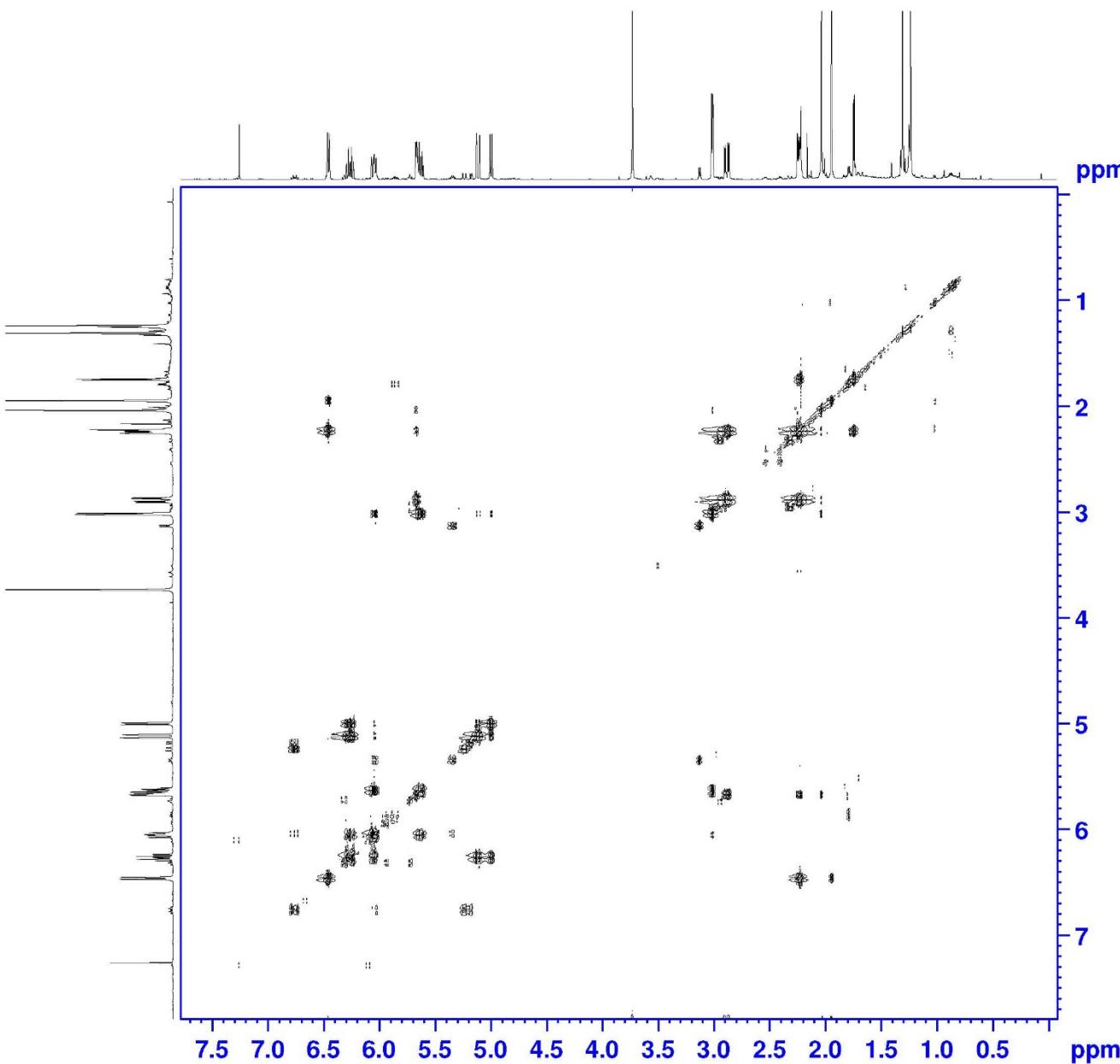
SFO1 150.9178988 MHz
NUC1 1H
PC 16.00 usec
PLW1 110.76000214 W

----- CHANNEL f2 -----

SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 usec
PLW2 12.55000019 W
PLW12 0.18072000 W
PLW13 0.08855400 W

F2 - Processing parameters

SI 32768
SF 150.9027908 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 23
PROCNO 1

P2 - Acquisition Parameters
Date_ 20181008
Time 18.23
INSTRUM spect
PROBHD 5 mm PABBT1H/
PULPROG cosyggmiphpp
TD 2048
SOLVENT CDCl3
NS 5
DS 4
SWH 4716.981 Hz
FIDRES 2.303213 Hz
AQ 0.2170880 sec
RG 2050
DW 106.000 usec
DE 6.50 usec
TE 298.2 K
D0 0.00009530 sec
D1 1.92135704 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00002000 sec
IN0 0.00021200 sec

===== CHANNEL f1 =====
SFO1 600.1323382 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P17 2500.00 usec
PLW1 12.55000019 W
PLW10 1.30999994 W

===== GRADIENT CHANNEL =====
GPNAME[1] SMSQ10.100
GPNAME[2] SMSQ10.100
GP21 10.00 %
GP22 20.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 600.13233 MHz
FIDRES 18.425707 Hz
SW 7.660 ppm
PRMODE States-TPII

F2 - Processing parameters
SI 1024
SF 600.1300250 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 States-TPII
SF 600.1300203 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0



Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 24
PROCNO 1

F2 - Acquisition Parameters

Date_ 20181008
Time 19.12
INSTRUM spect
PROBHD 5 mm PABBI 1H/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 35
DS 16
SWH 4716.981 Hz
FIDRES 4.606427 Hz
AQ 0.1085440 sec
RG 2050
DW 106.000 usec
DE 6.50 usec
TE 298.1 K
CNSST2 145.0000000
D0 0.00000300 sec
D1 1.45699203 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
IN0 0.00002000 sec
ZGOPTNS

----- CHANNEL f1 -----

SFO1 600.1323302 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P28 1000.00 usec
PLW1 12.5500019 W

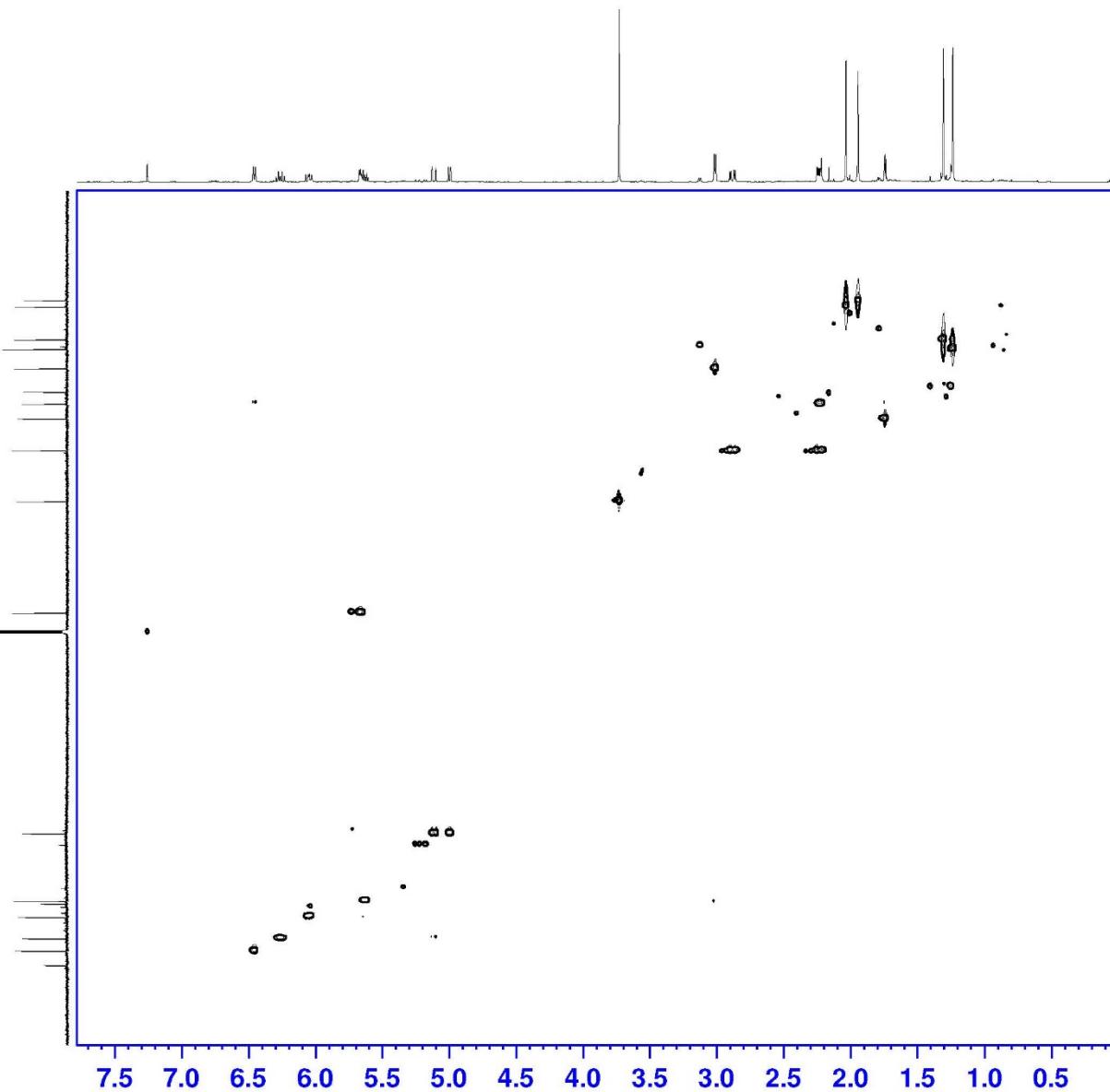
----- CHANNEL f2 -----
SFO2 150.9140636 MHz
NUC2 13C
CPDPGRG[2] garp
P3 16.00 usec
P4 32.00 usec
PCPD2 60.00 usec
PLW2 110.76000214 W
PLW12 7.87610006 W

----- GRADIENT CHANNEL -----
GPNAME[1] SMSQ10.100
GPNAME[2] SMSQ10.100
GP11 80.00 %
GP12 20.10 %
P16 1000.00 usec

P1 - Acquisition parameters
TD 256
SFO1 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PmODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 600.1300239 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

P1 - Processing parameters
SI 1024
M2 echo-antiecho
SF 150.9028240 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0



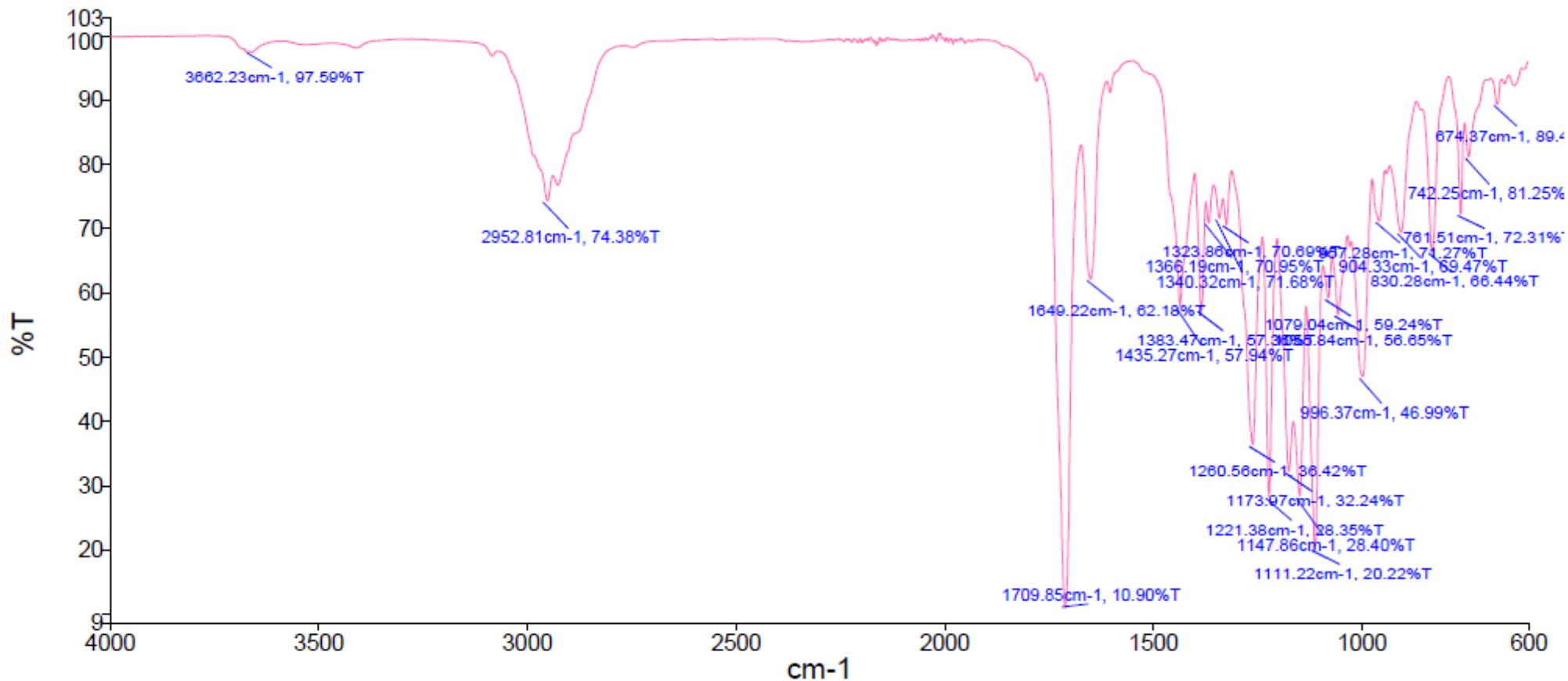
(8b) IR Spectrum

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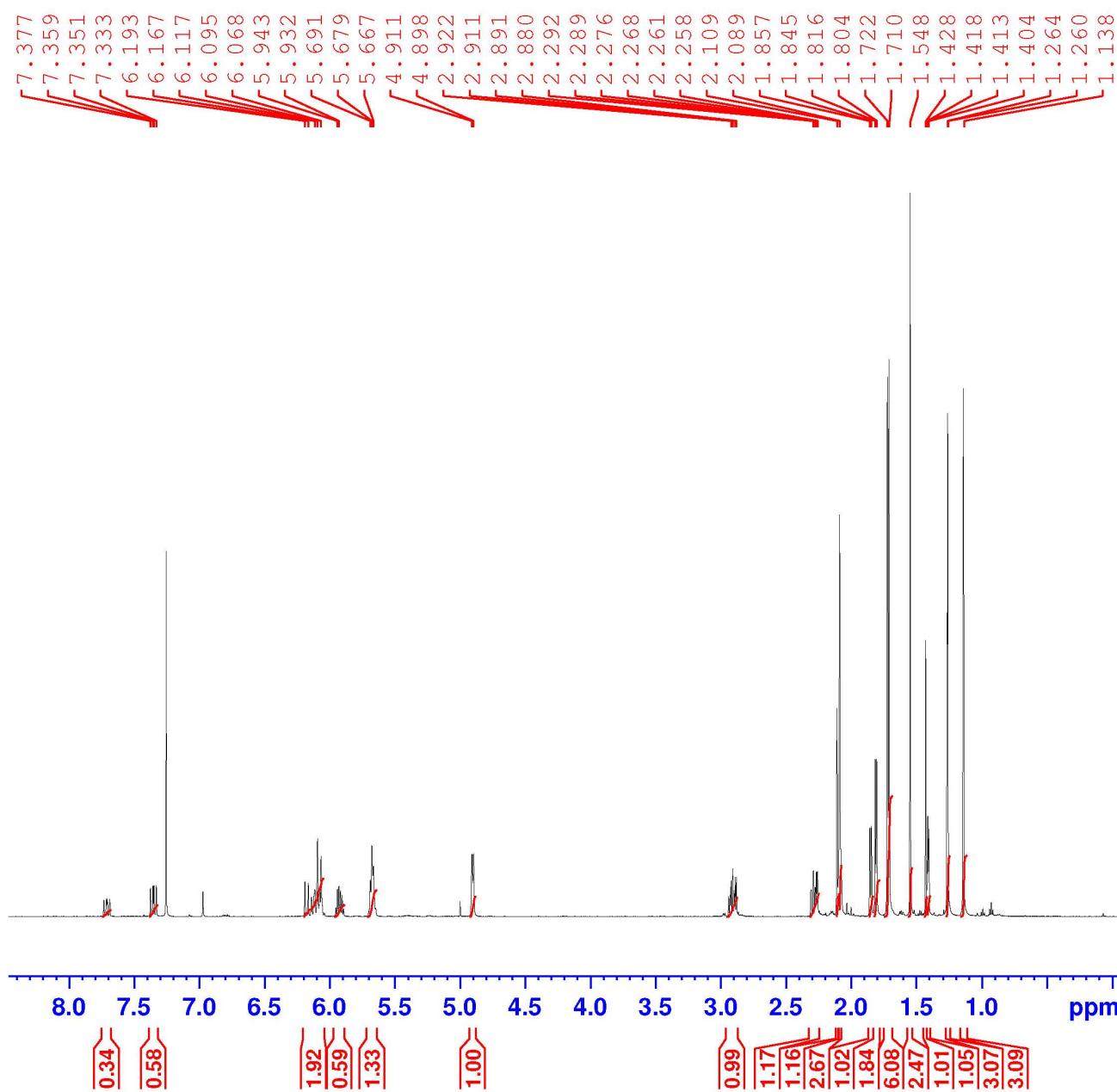
Analyst
Date

Analyst
Wednesday, 17 October 2018 3:02 PM

PerkinElmer Spectrum Version 10.4.2
Wednesday, 17 October 2018 3:02 PM



(9a) NMR Characterisation

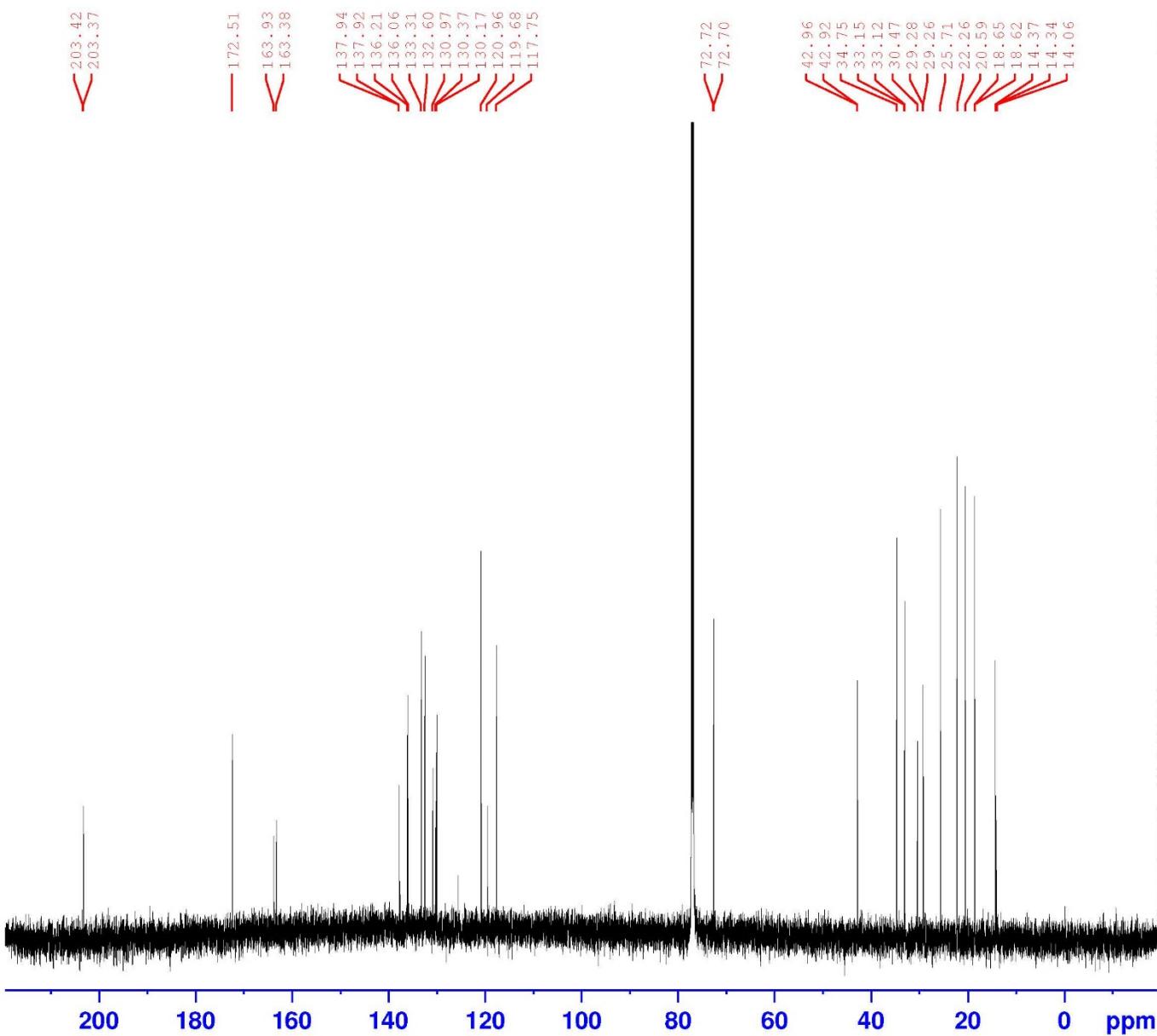


Current Data Parameters
 NAME 20190404 Iso PI CLEAN
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190404
 Time 19.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 64
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 287
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 13.50 usec
 PLW1 17.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300277 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



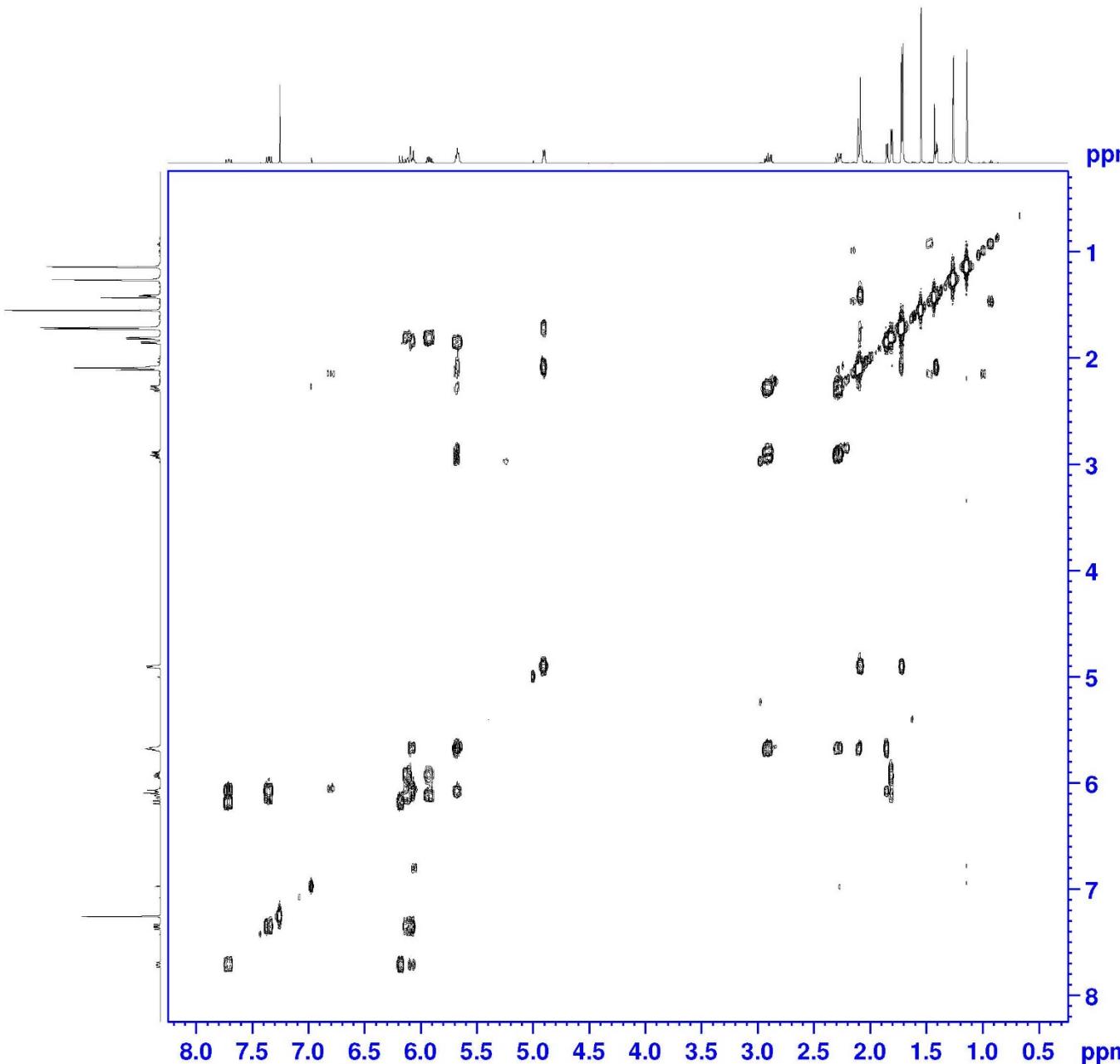
Current Data Parameters
 NAME 20190404 Iso PI CLEAN
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190404
 Tme 21.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 80.00000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.00000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9027898 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 20190404 Iso PI CLEAN
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190404
 Time 19.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpppcf
 TD 2048
 SOLVENT CDCl3
 NS 10
 DS 8
 SWH 4807.692 Hz
 FIDRES 2.347506 Hz
 AQ 0.2129920 sec
 RC 144
 DW 104.000 usec
 DE 6.50 usec
 TE 298.2 K
 D0 0.00000300 sec
 D1 1.91480303 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00020800 sec

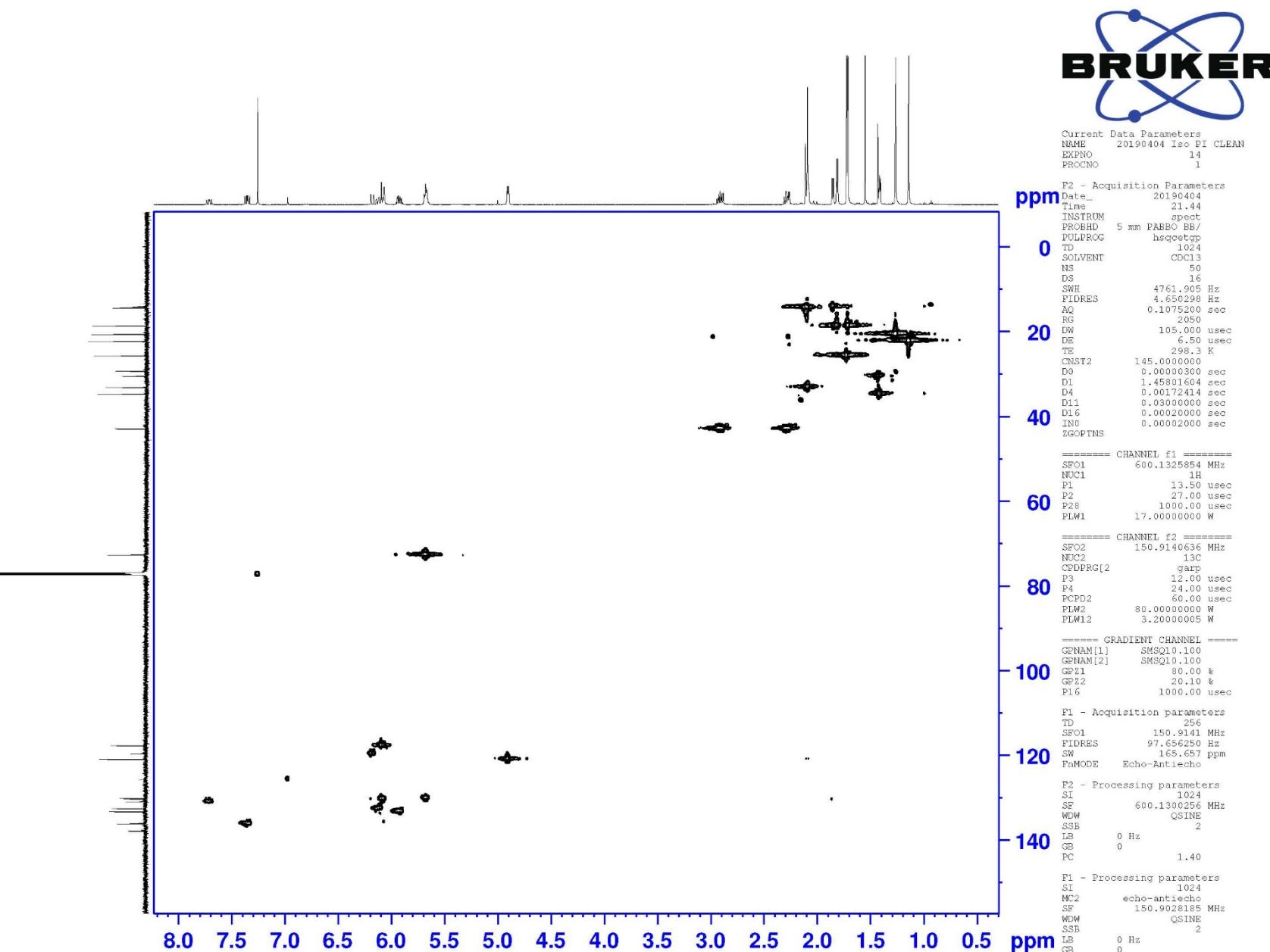
----- CHANNEL f1 -----
 SFO1 600.1325742 MHz
 NUC1 1H
 P0 13.50 usec
 P1 13.50 usec
 P17 2500.00 usec
 PLW1 17.00000000 W
 PLW10 4.58319998 W

----- GRADIENT CHANNEL -----
 GPNAM[1] SMSQ10.100
 GPZ1 10.00 %
 F16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 600.1326 MHz
 FIDRES 37.560097 Hz
 SW 8.011 ppm
 FnMODE QF

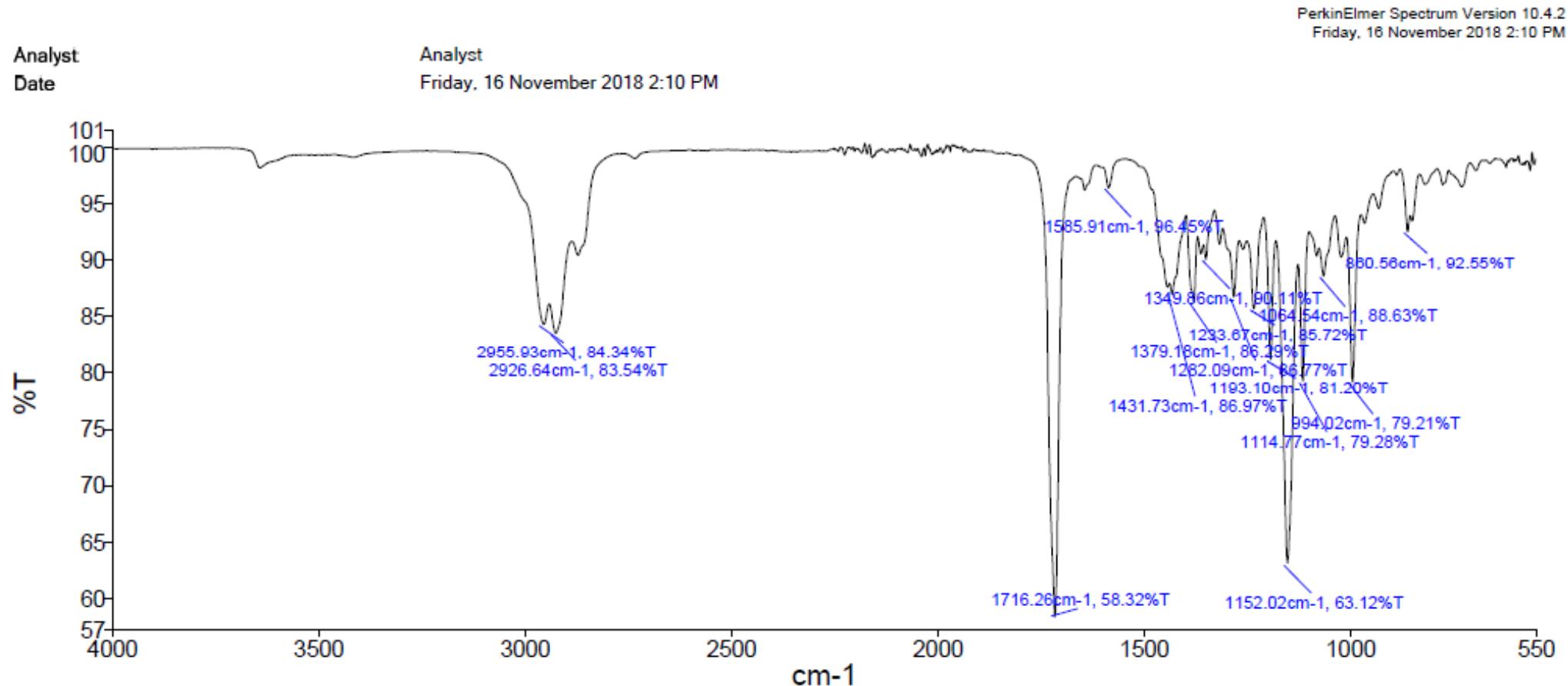
F2 - Processing parameters
 SI 1024
 SF 600.1300254 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

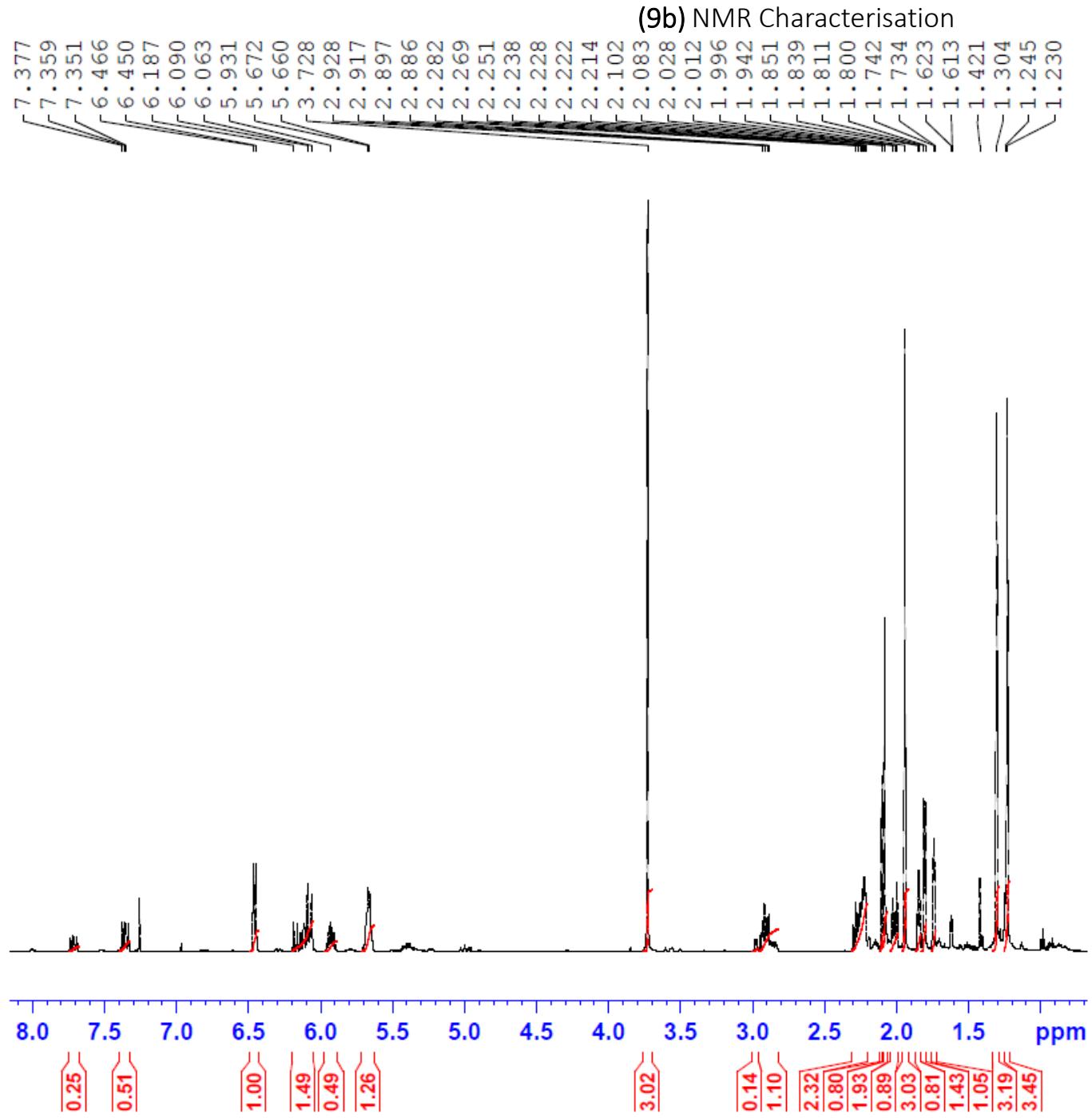
F1 - Processing parameters
 SI 1024
 MC2 QF
 SP 600.1300271 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



(9a) IR Spectrum

70



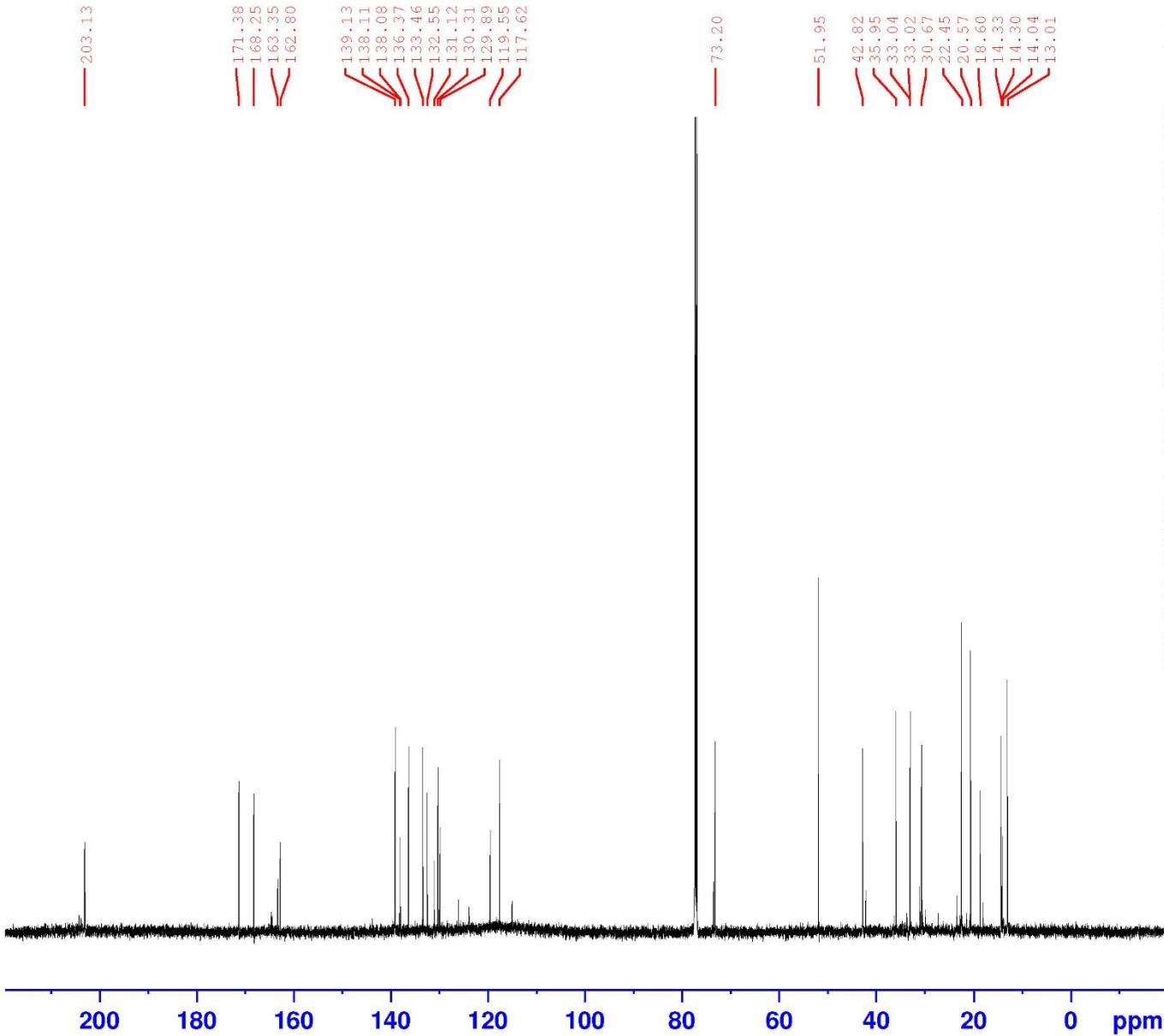


Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date 20181008
Time 17.03
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 12019.230 Hz
FIDRES 0.193399 Hz
AQ 2.7262976 sec
RG 71.8
DW 41.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
SP01 600.1337060 MHz
NUC1 1H
P1 8.40 usec
PLW1 12.55000019 W

F2 - Processing parameters
SI 65536
SF 600.1300267 MHz
WM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_ 20181008
Time_ 3.59
INSTRUM spect
PROBHD 5 mm PARBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 1820
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----

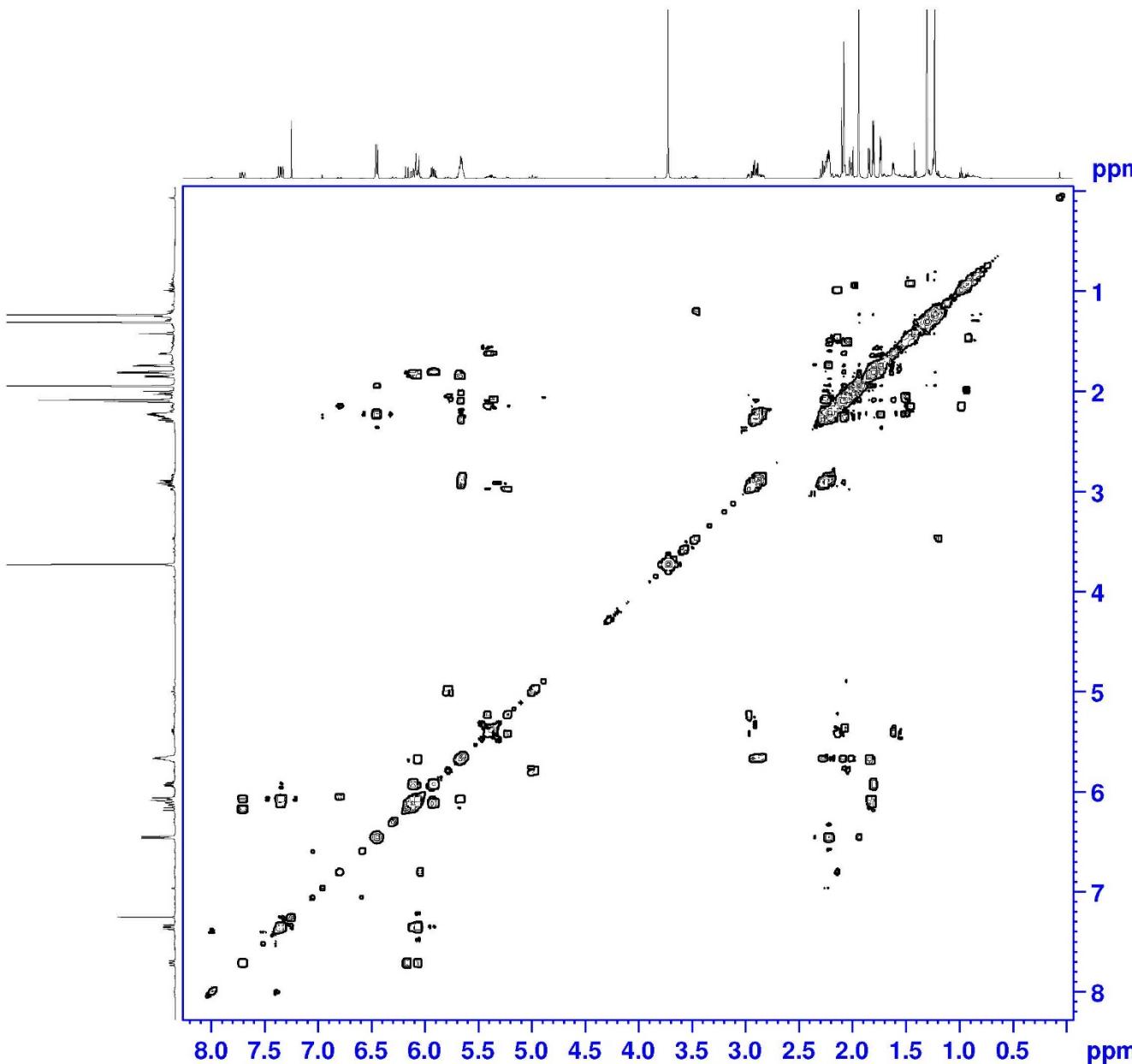
SFO1 150.9178985 MHz
NUC1 1H
PC 16.00 usec
PLW1 110.76000214 W

----- CHANNEL f2 -----

SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 usec
PLW2 12.55000019 W
PLW12 0.18072000 W
PLW13 0.08855400 W

F2 - Processing parameters

SI 32768
SF 150.9027924 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME 20181107 Isopyrethrin II
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181107
Time 9.58
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG cosygpppcf
TD 2048
SOLVENT CDCl3
NS 5
DS 8
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048000 sec
RG 64
DW 100.000 usec
DE 6.50 usec
TE 298.2 K
D0 0.00000300 sec
D1 1.92299497 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00020000 sec

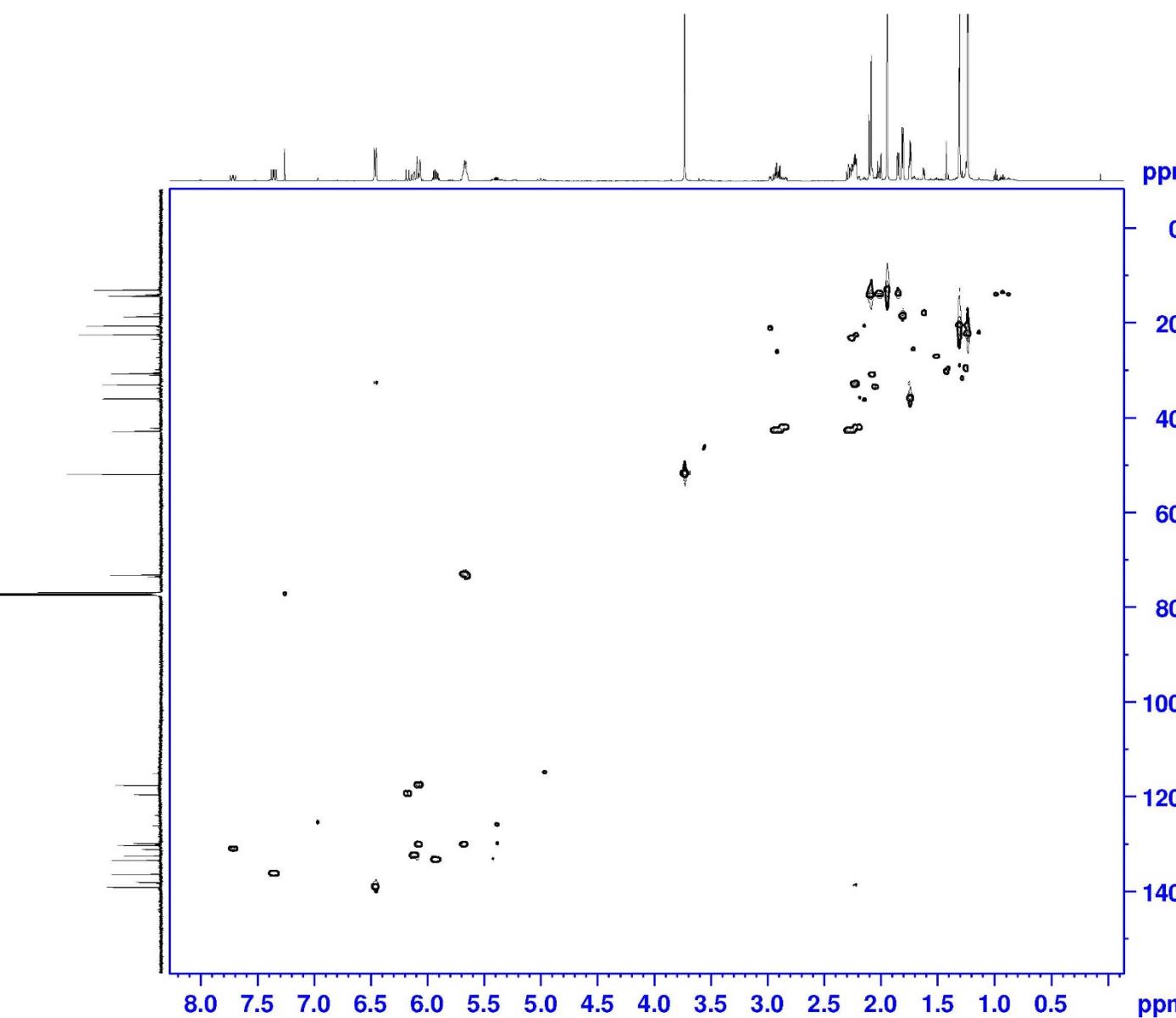
----- CHANNEL f1 -----
SFO1 600.1324960 MHz
NUC1 1H
P0 8.40 usec
P1 8.40 usec
P17 2500.00 usec
PLW1 12.55000019 W
PLW10 1.30999994 W

----- GRADIENT CHANNEL -----
GPNAME[1] SMSQ10.100
GPZ1 10.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 600.1325 MHz
FIDRES 39.062500 Hz
SW 8.331 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 600.1300342 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SP 600.1300265 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0



Current Data Parameters
NAME 20181008 Transfer Hydrogenation PII
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters

Date_ 20181008
Time_ 4:55
INSTRUM spect
PROBHD 5 mm PABBI 1H/
DULPROG hsqcetgps
TD 1024
SOLVENT CDCl3
NS 35
DS 16
SWH 5050.505 Hz
FIDRES 4.932134 Hz
AQ 0.1013760 sec
RG 2050
DW 99.000 usec
DE 6.50 usec
TE 298.1 K
CNSST2 145.0000000
D0 0.00000300 sec
D1 1.46415997 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00020000 sec
D10 0.00002000 sec
ZGOPTNS

----- CHANNEL f1 -----
SFO1 600.1324643 MHz
NUC1 1H
P1 8.40 usec
P2 16.80 usec
P28 1000.00 usec
PLW1 12.55000019 W

----- CHANNEL f2 -----
SFO2 150.9140636 MHz
NUC2 13C
CPDPGRG[2] garp
P3 16.00 usec
P4 32.00 usec
PCPD2 60.00 usec
PLW2 110.76000214 W
PLW12 7.87610006 W

----- GRADIENT CHANNEL -----
GP[NAM[1]] SMSQ10.100
GP[NAM[2]] SMSQ10.100
GP11 80.00 %
GP12 20.10 %
P16 1000.00 usec

P1 - Acquisition parameters
TD 256
SFO1 150.9141 MHz
FIDRES 97.656250 Hz
SW 165.657 ppm
PwMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 600.1300237 MHz
WDW QSIMINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

P1 - Processing parameters
SI 1024
M2 echo-antiecho
SF 150.9028198 MHz
WDW QSIMINE
SSB 2
LB 0 Hz
GB 0

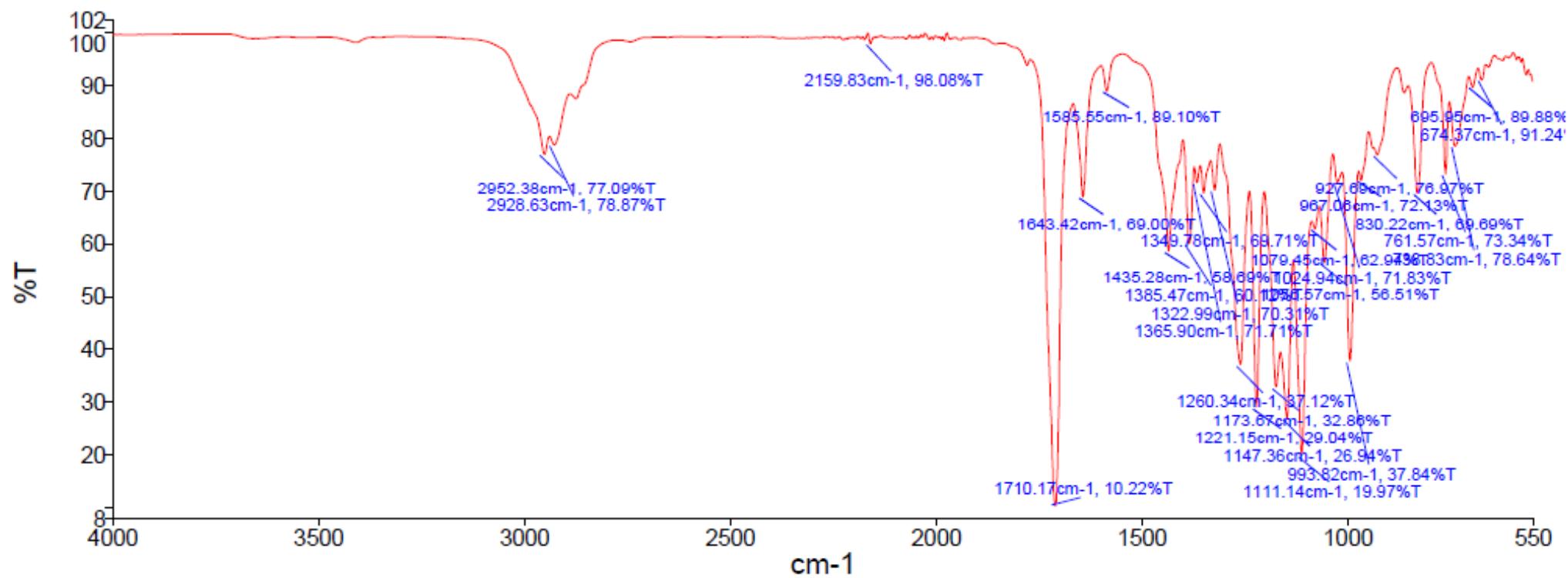
(9b) IR Spectrum

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Analyst
Date

Analyst
Friday, 16 November 2018 2:11 PM

PerkinElmer Spectrum Version 10.4.2
Friday, 16 November 2018 2:11 PM



Semi-synthetic (2a) + (7a) mixture NMR Characterisation

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Current Data Parameters
NAME 20180704 Diimide Reduction PI
EXPNO 10
PROCNO 1

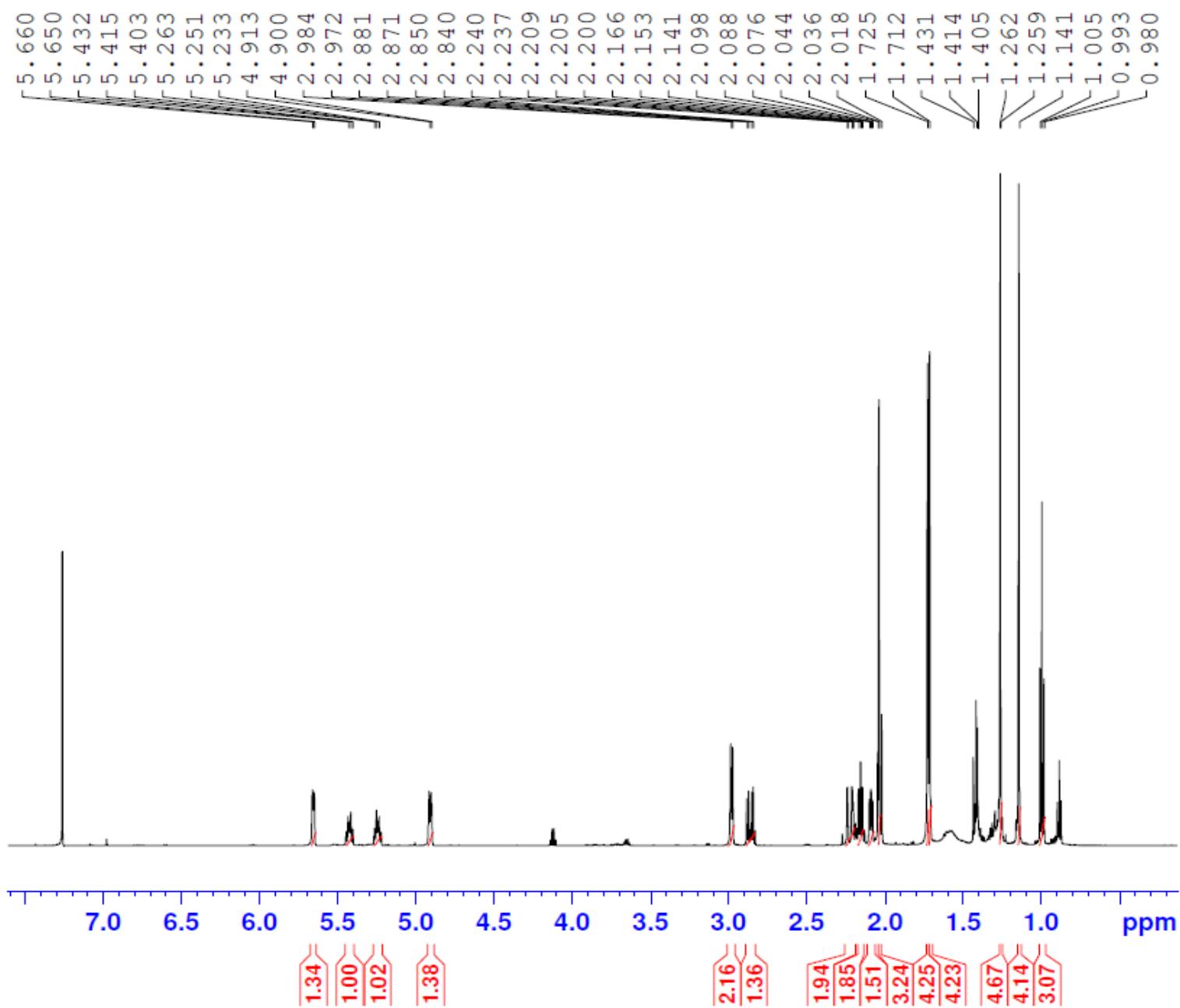
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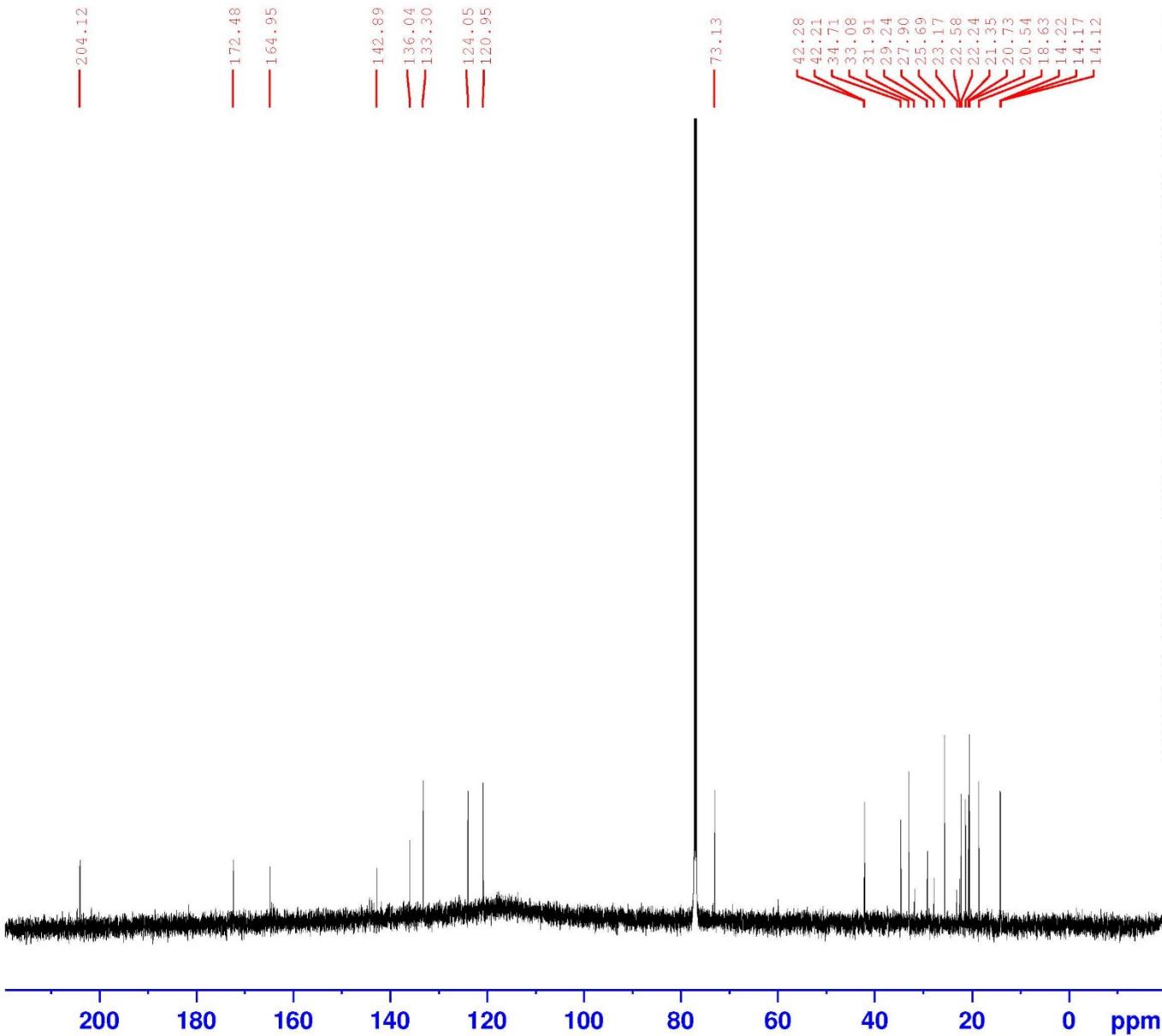
F2 - Acquisition Parameters
Date_          20100704
Time           18.13
INSTRUM        spect
PROBHD        5 mm PABBI 1H/
PULPROG       zg30
TD             65536
SOLVENT        CDC13
NS              64
DS              2
SWH             12019.230 Hz
FIDRES        0.183399 Hz
AQ              2.7262976 sec
RG              181
DW              41.600 usec
DE              6.50 usec
TE              298.2 K
D1             1.0000000 sec
TDO              1

```

----- CHANNEL f1 -----
SFO1 600.1337060 MHz
NUC1 1H
P1 8.40 usec
PLW1 12.55000019 W

```
F2 - Processing parameters  
SI          65536  
SF          600.1300237 MHz  
WDW         no  
SSB         0  
LB          0 Hz  
GB          0  
PC          1.00
```





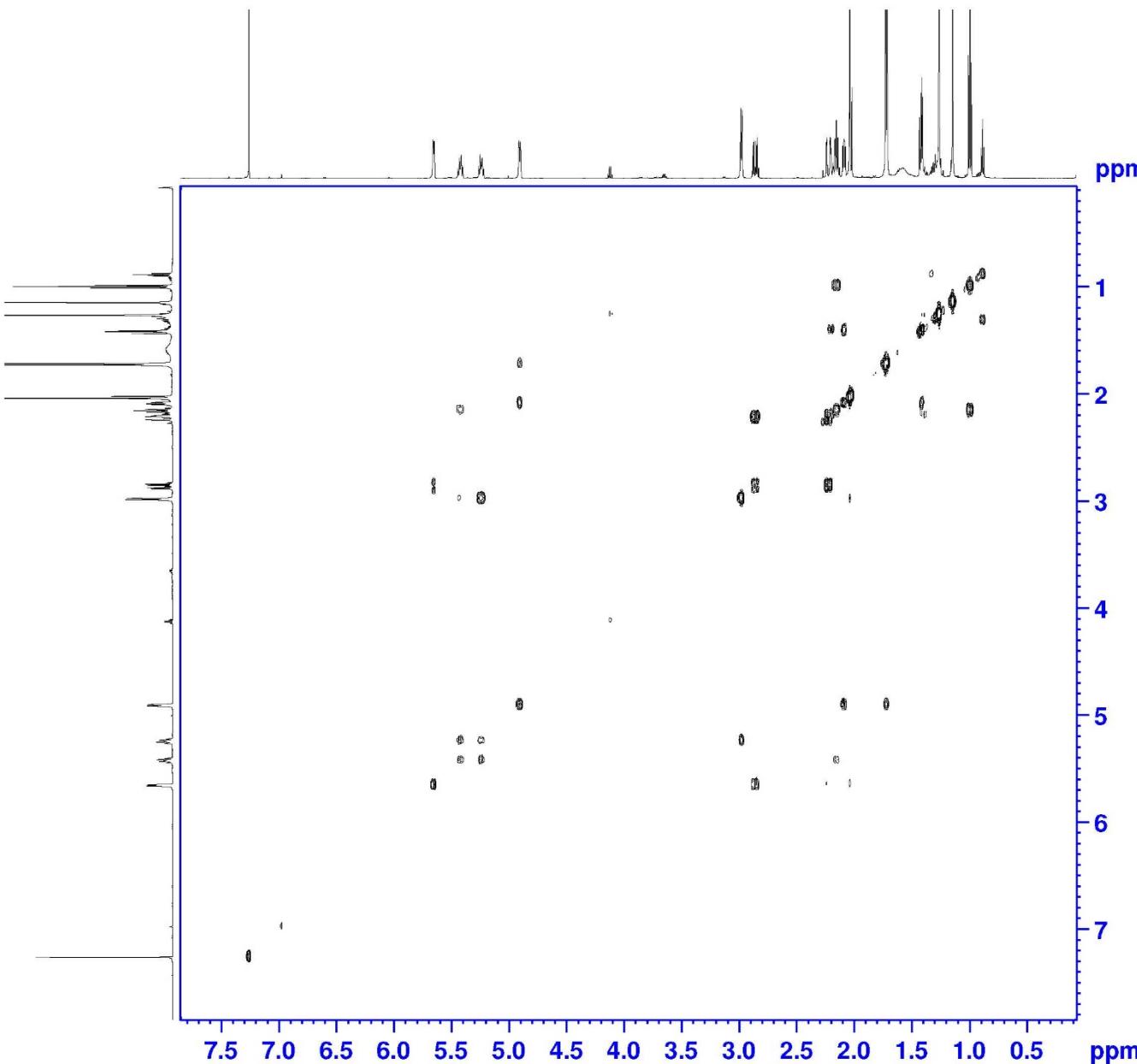
Current Data Parameters
NAME 20180704 Diimide Reduction PI
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180704
Time 19:12
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 1820
DW 13.867 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 150.9178988 MHz
NUC1 13C
P1 16.00 usec
PLW1 110.76000214 W

----- CHANNEL f2 -----
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
FCPD2 70.00 usec
PLW2 12.55000019 W
PLW12 0.18072000 W
PLW13 0.08855400 W

F2 - Processing parameters
SI 32768
SF 150.9027915 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME 20180704 Diimide Reduction PI
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180704
 Time 18:15
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG cosyppppqf
 TD 2048
 SOLVENT CDCl3
 NS 1
 DS 8
 SWH 4672.897 Hz
 FIDRES 2.281688 Hz
 AQ 0.2191360 sec
 RG 64
 DW 107.000 usec
 DE 6.50 usec
 TB 298.2 K
 DO 0.00000300 sec
 D1 1.9086598 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 INO 0.00021400 sec

===== CHANNEL f1 =====
 SFO1 600.1323996 MHz
 NUC1 1H
 P0 8.40 usec
 P1 8.40 usec
 P17 2500.00 usec
 PLW1 12.55000019 W
 PLW10 1.30999994 W

===== GRADIENT CHANNEL =====
 GRBNM[1] SMSQ10.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 128
 SFO1 600.1324 MHz
 FIDRES 36.507008 Hz
 SW 7.786 ppm
 FnMODE QF

F2 - Processing parameters
 SI 1024
 SF 600.1300204 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

P1 - Processing parameters
 SI 1024
 MC2 QF
 SF 600.1300260 MHz
 WDW QSINE
 SSB 0
 LB 0 Hz
 GB 0



Current Data Parameters
 NAME 20180704_Dimide Reduction PI
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters

Date 20180704
 Time 19.18
 INSTRUM spect
 PROBHD 5 mm PABBT1H
 DULPROG hsqcetgps
 TD 1024
 SOLVENT CDCl3
 NS 50
 DS 16
 SWH 4807.692 Hz
 FIDRES 4.695012 Hz
 AQ 0.1064960 sec
 RG 2050
 DW 104.000 usec
 DE 6.50 usec
 TE 298.0 K
 CNST2 145.000000
 D0 0.00000300 sec
 D1 1.45904005 sec
 D4 0.00172414 sec
 D11 0.03000000 sec
 D16 0.00020000 sec
 IN0 0.00002000 sec
 ZGOPTNS

===== CHANNEL f1 =====
 SFO1 600.1323536 MHz
 NUC1 1H
 P1 8.40 usec
 P2 16.80 usec
 P2B 1000.00 usec
 PLW1 12.55000019 W

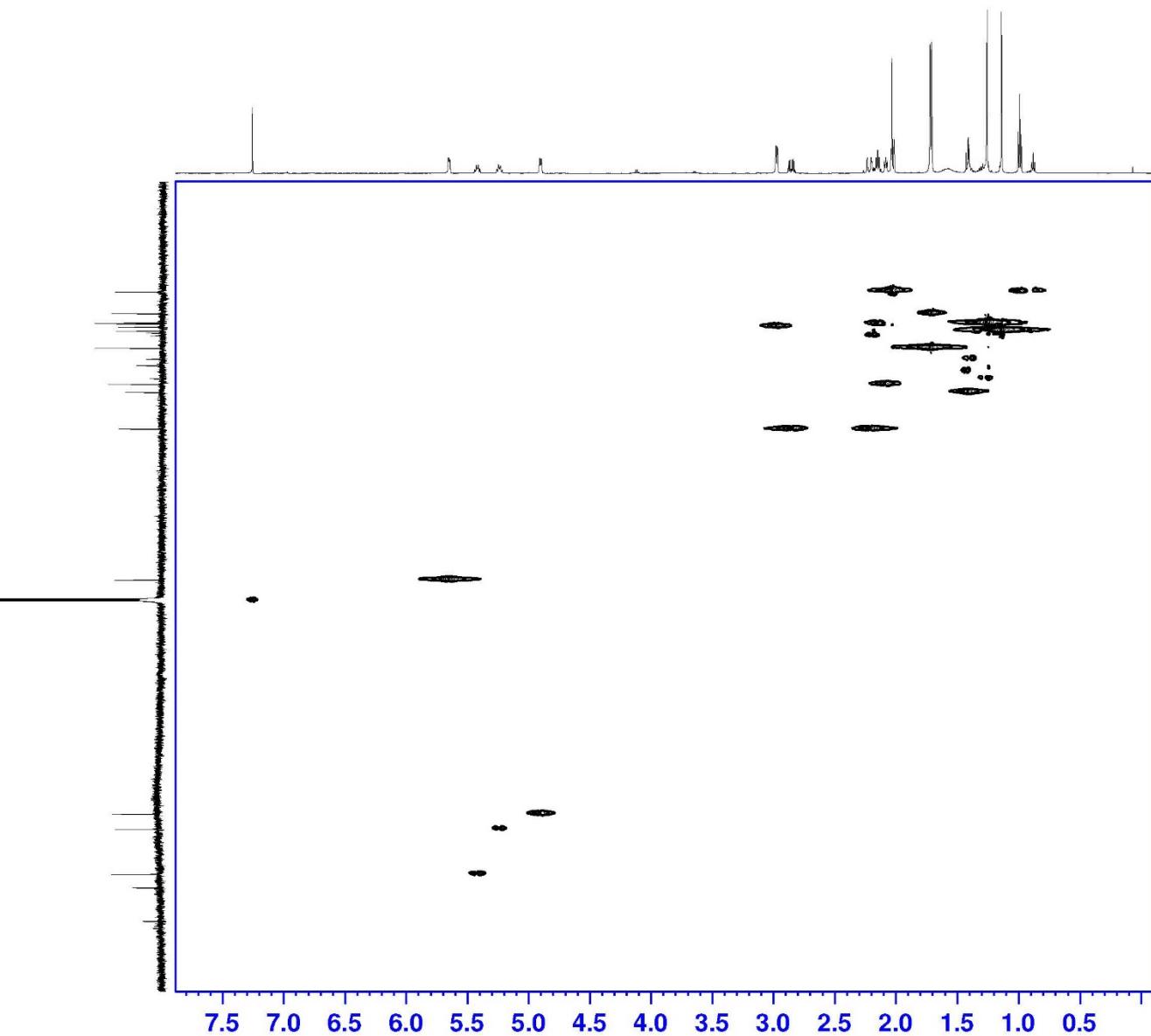
===== CHANNEL f2 =====
 SFO2 150.9140636 MHz
 NUC2 13C
 GDEPRFG[2] garp
 P3 16.00 usec
 P4 32.00 usec
 PCPD2 60.00 usec
 PLM2 110.76000214 W
 PLW2 7.87610006 W

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

P1 - Acquisition parameters
 TD 256
 SFO1 150.9141 MHz
 FIDRES 97.656250 Hz
 SW 165.657 ppm
 PRMODE Echo-Antiecho

P2 - Processing parameters
 SI 1024
 SF 600.1300242 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

P1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 SF 150.9028278 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0



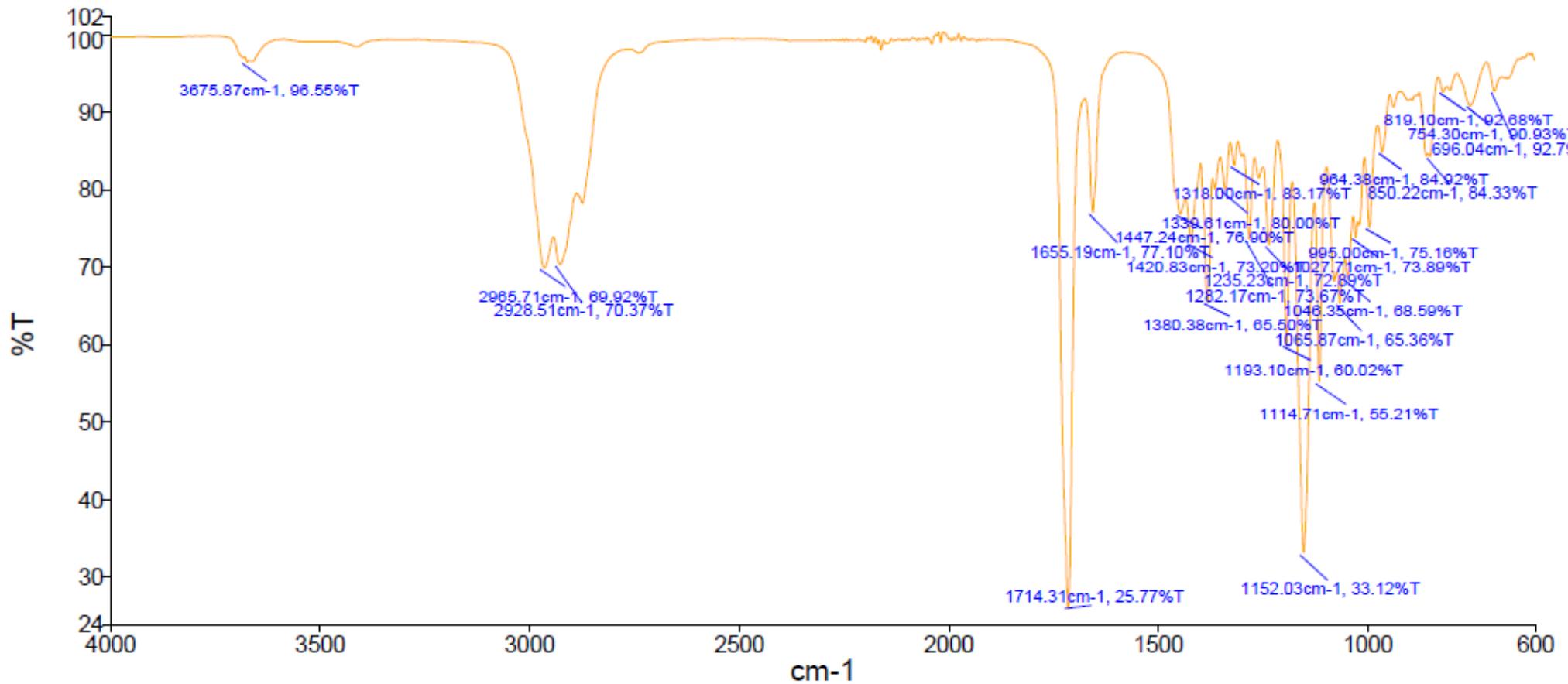
Semi-synthetic (2a) + (7a) mixture IR Spectrum

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PerkinElmer Spectrum Version 10.4.2
Wednesday, 17 October 2018 2:58 PM

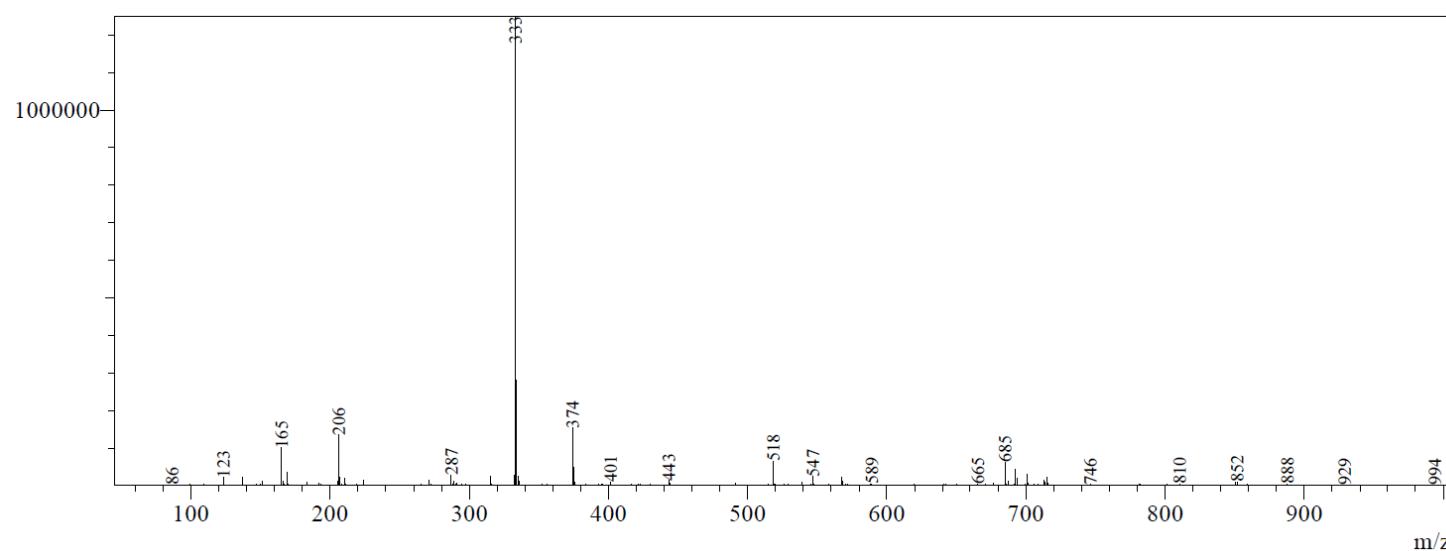
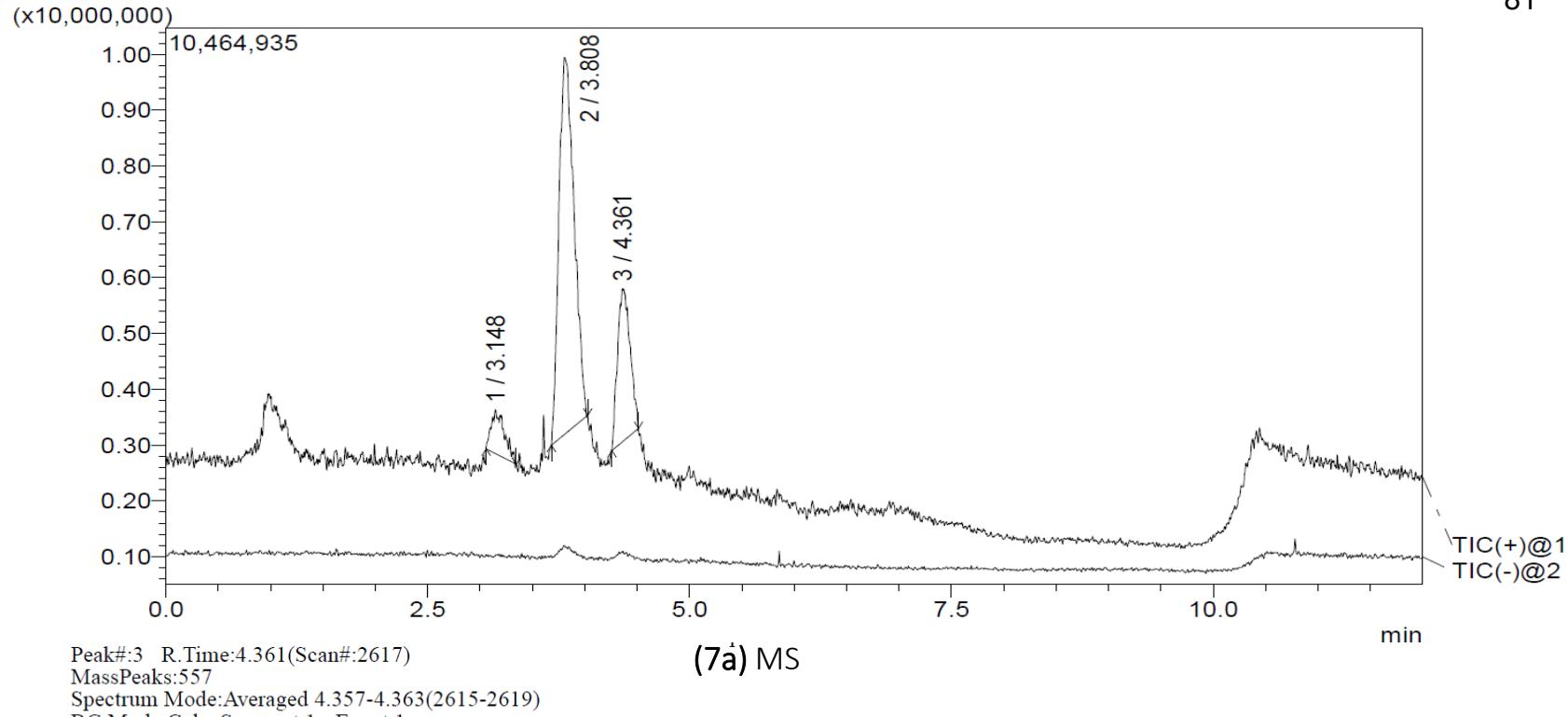
Analyst
Date

Analyst
Wednesday, 17 October 2018 2:58 PM



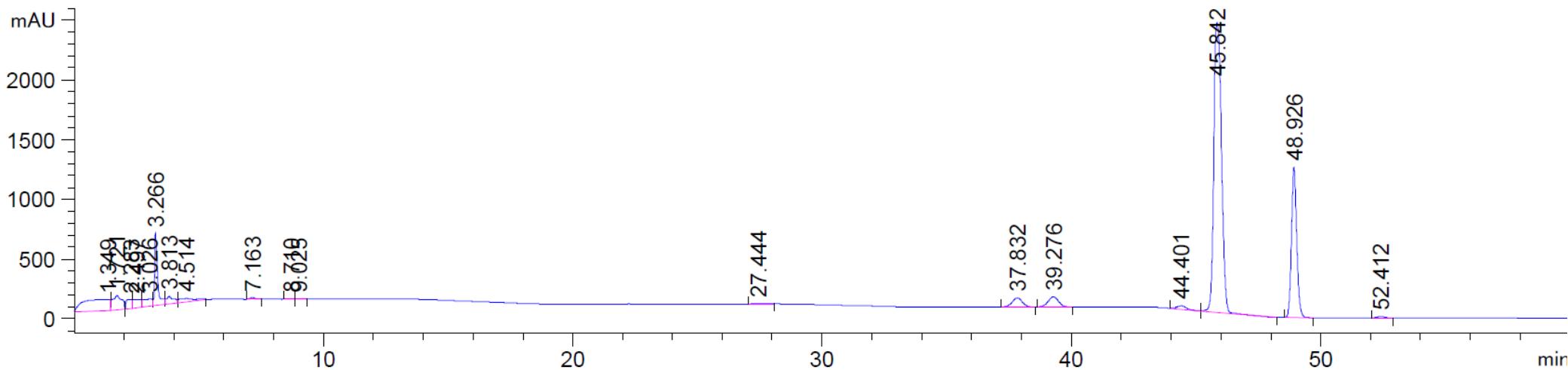
Semi-synthetic (2a) + (7a) mixture LC-MS

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HPLC analysis Semi-synthetic (2a) + (7a) mixture

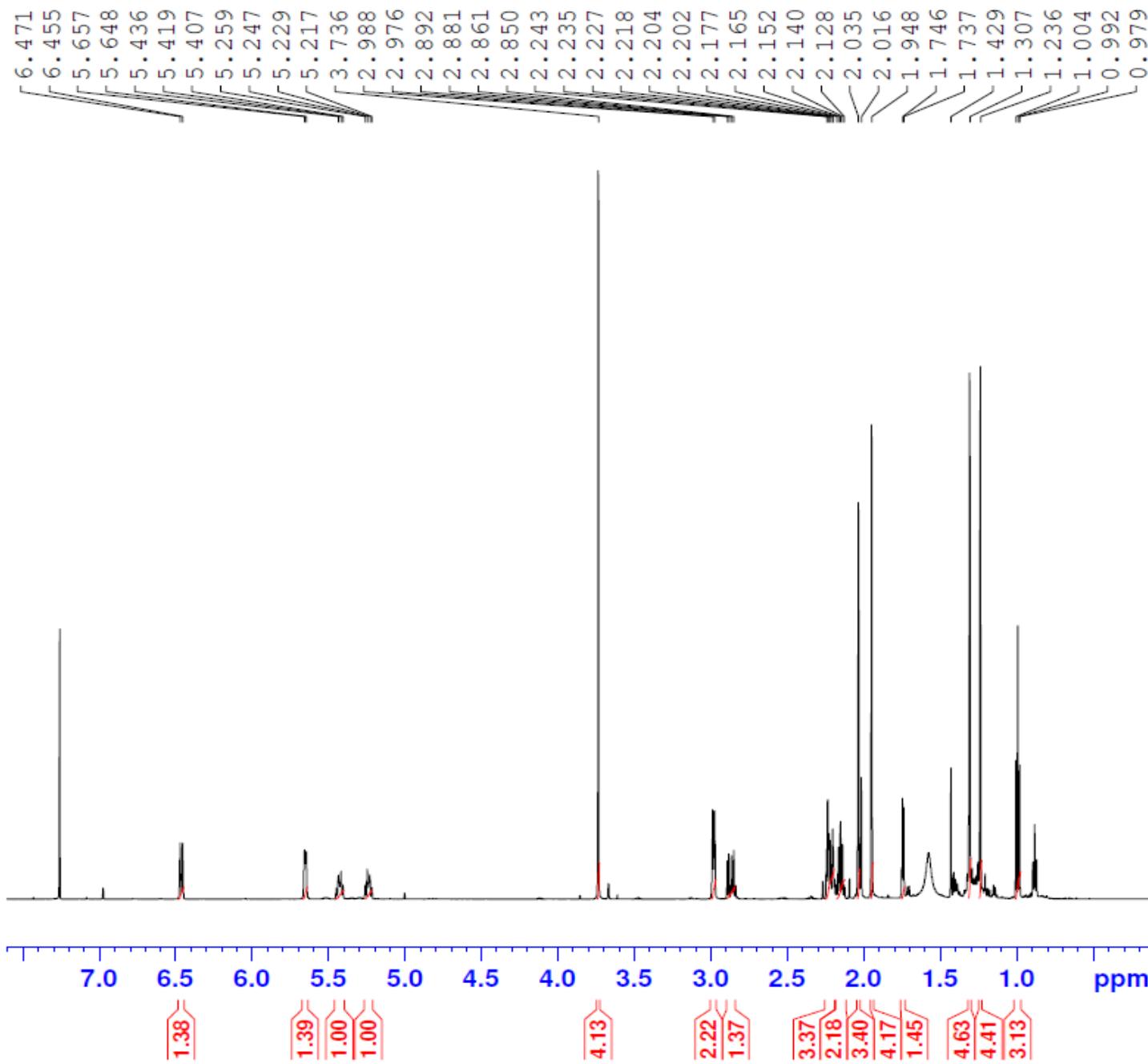
82



Retention time (min)	Compound (#)	Area (mAU)	% Area
45.842	2a	51507.4	68.0
48.926	7a	18624.9	24.7

Semi-synthetic (2b) + (7b) mixture NMR Characterisation

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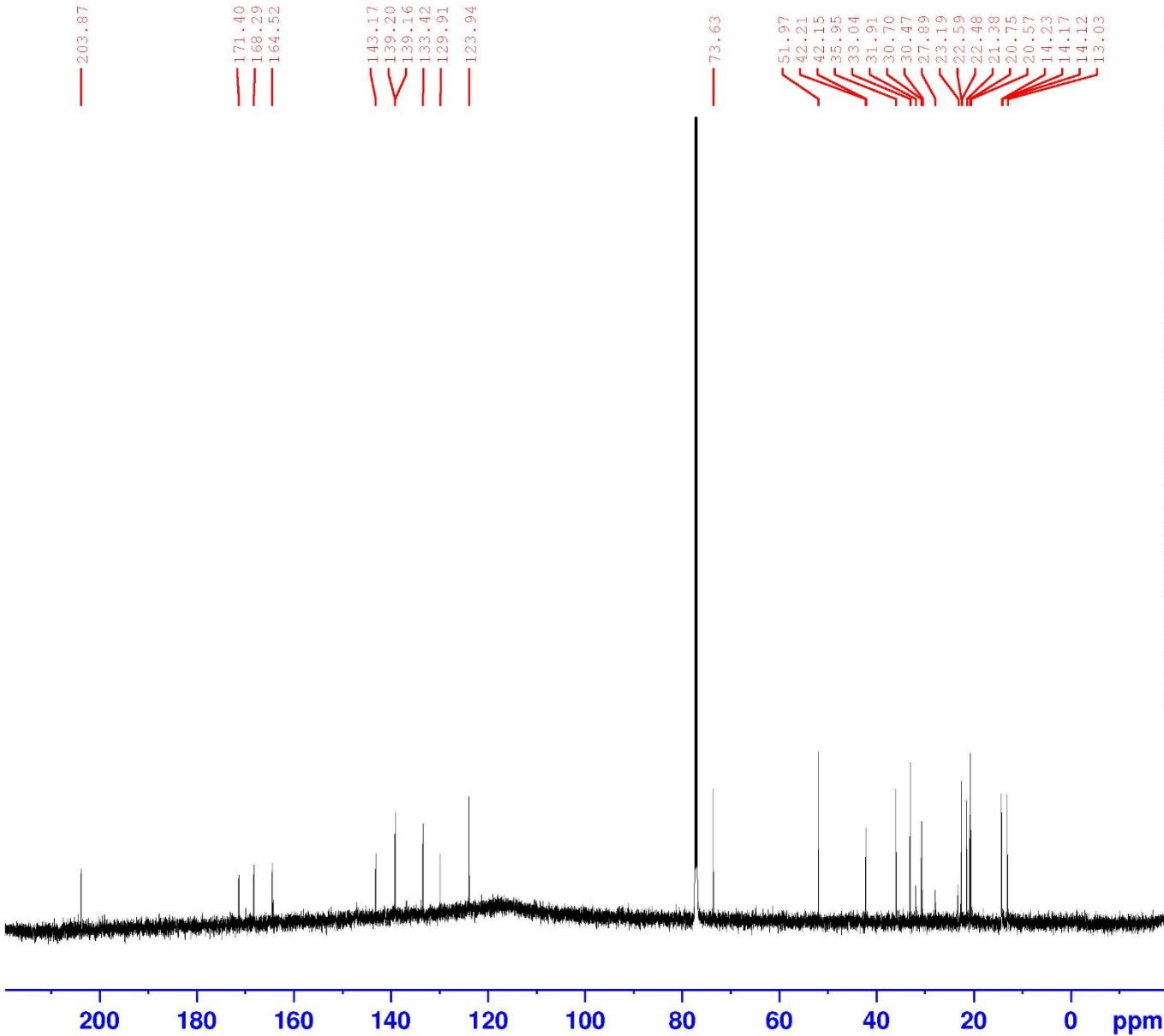
Current Data Parameters
NAME 20180831_JII (Diimide reduction)
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Data_ 20180831
Time 18.02
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 64
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 203
DW 41.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 600.1337060 MHz
NUC1 1H
P1 8.40 usec
PLW1 12.55000019 W

F2 - Processing parameters
SI 65536
SF 600.1300252 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



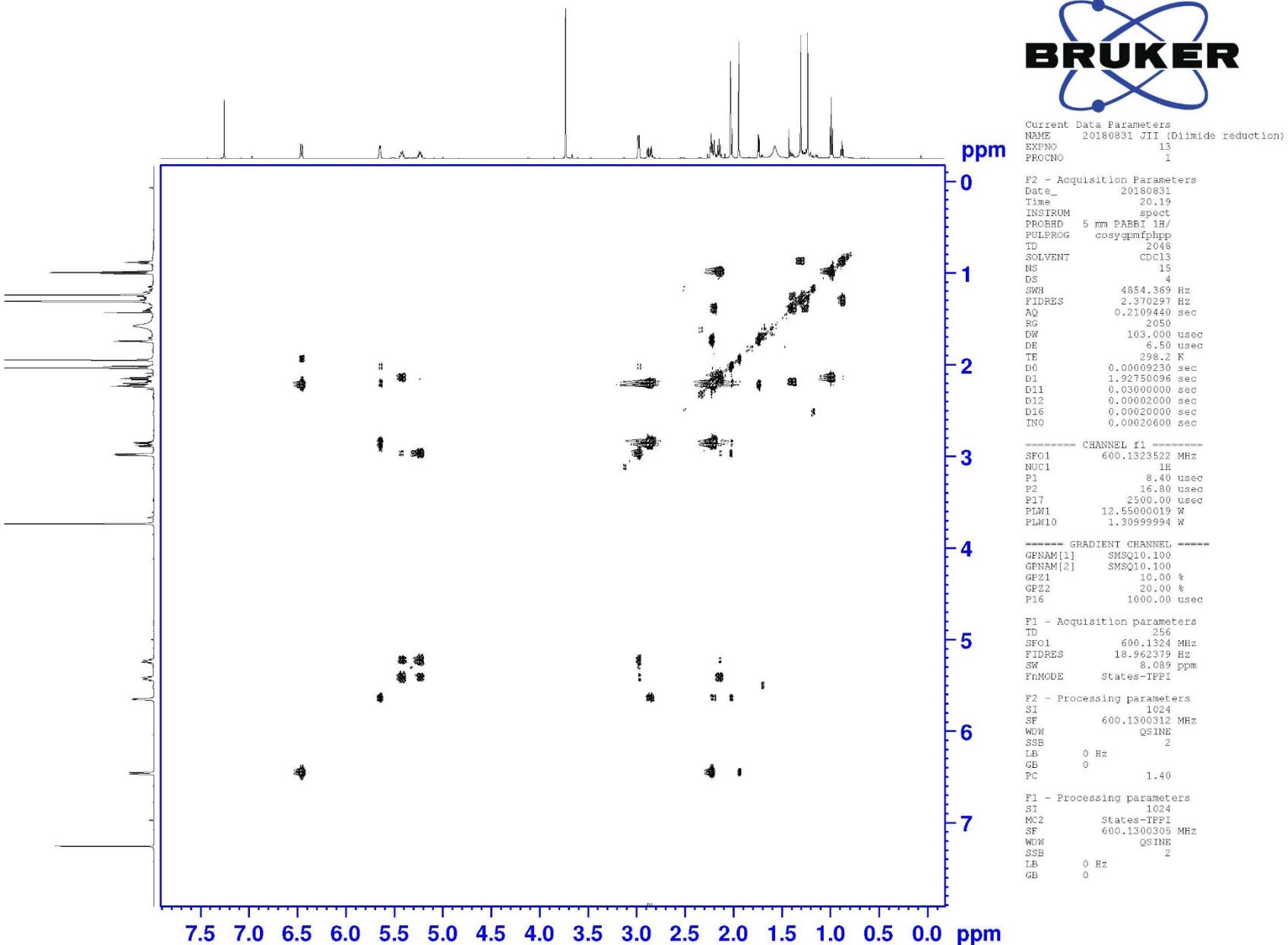
Current Data Parameters
 NAME_ 20180831_JII (Diimide reduction)
 EXPNO 11
 PROCNO 1

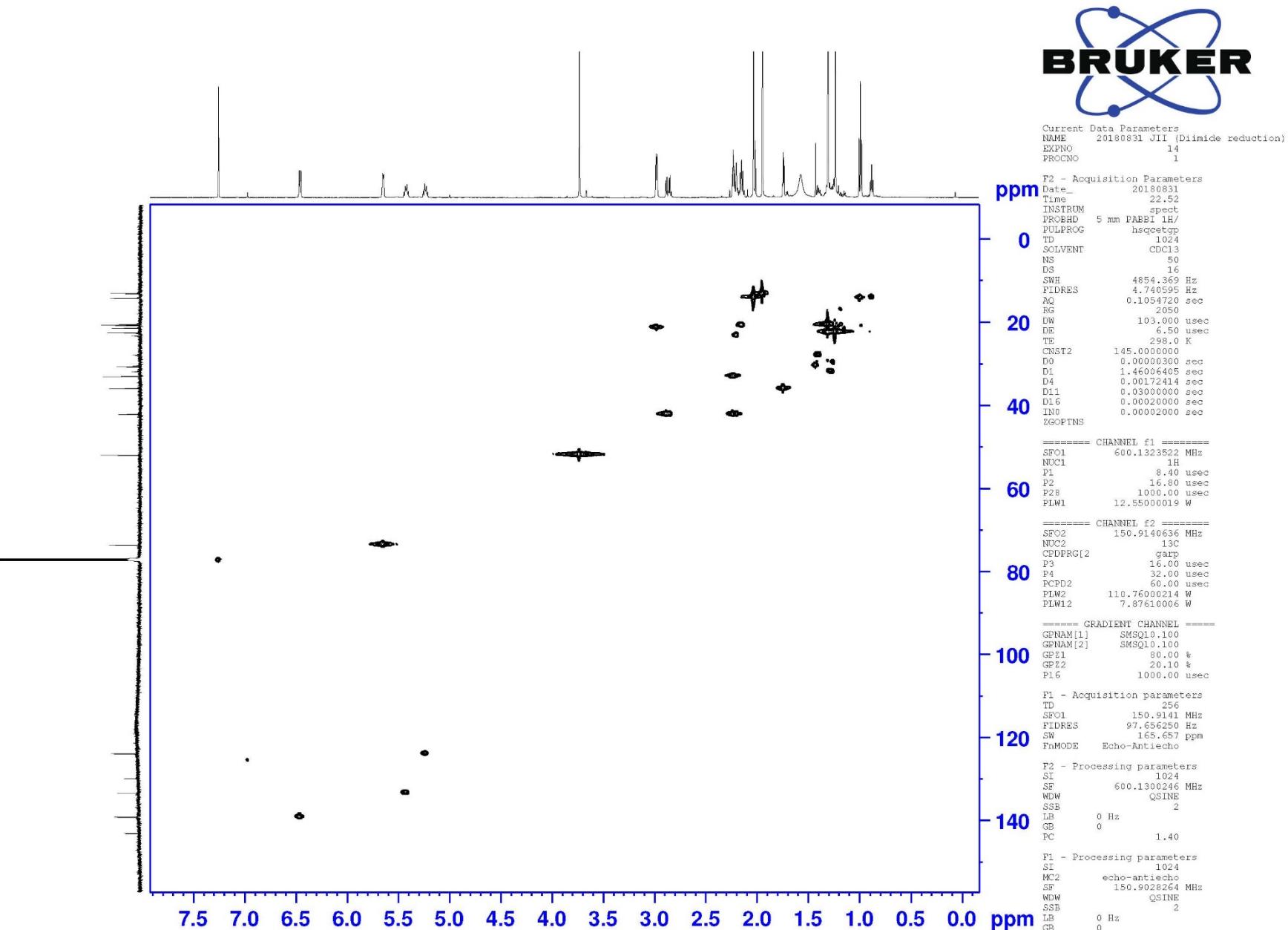
F2 - Acquisition Parameters
 Date_ 20180831
 Time_ 19.55
 INSTRUM spect
 PROBHD 5 mm PABBI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 1820
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

----- CHANNEL f1 -----
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 16.00 usec
 PLW1 110.76000214 W

----- CHANNEL f2 -----
 SFO2 600.1524005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 12.55000019 W
 PLW12 0.18072000 W
 PLW13 0.08655400 W

F2 - Processing parameters
 SI 32768
 SF 150.9027891 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





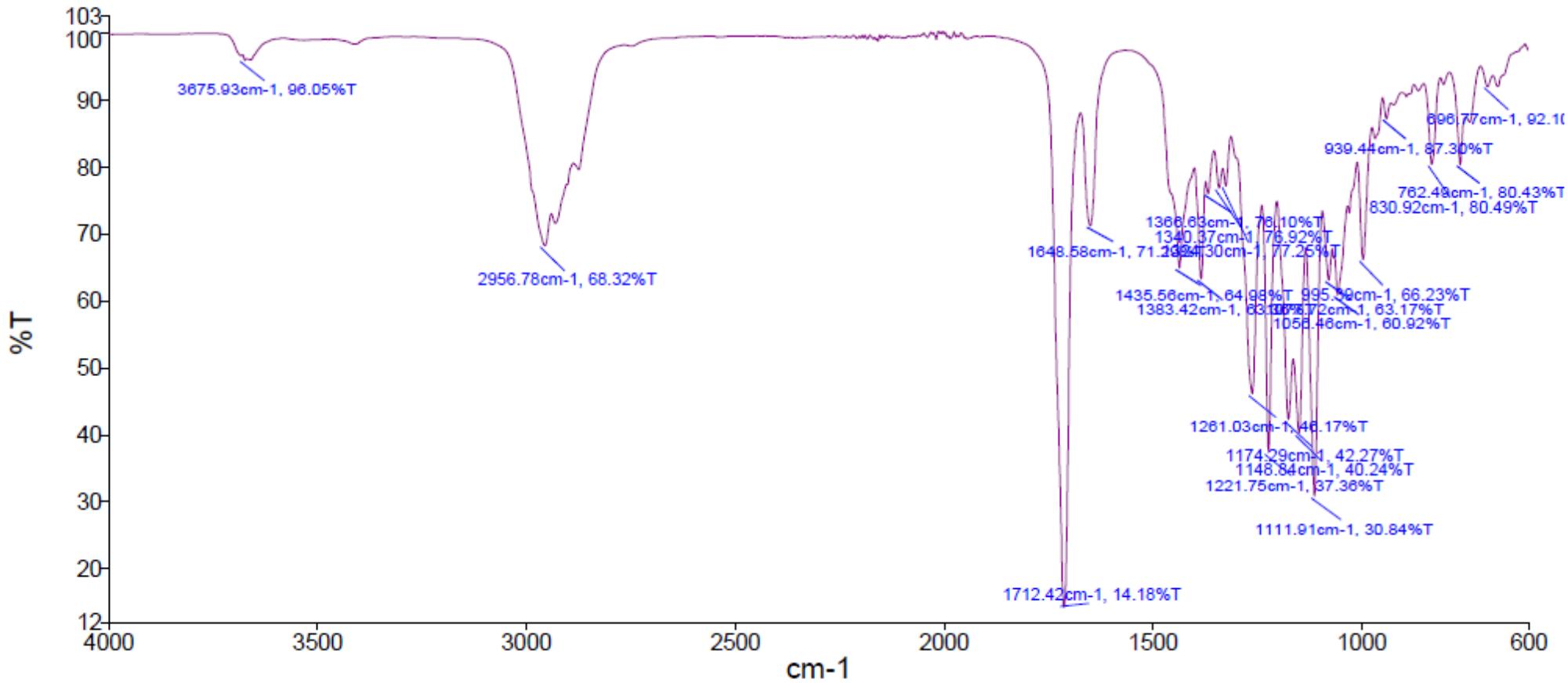
Semi-synthetic (2b) + (7b) mixture IR Spectrum

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PerkinElmer Spectrum Version 10.4.2
Wednesday, 17 October 2018 2:59 PM

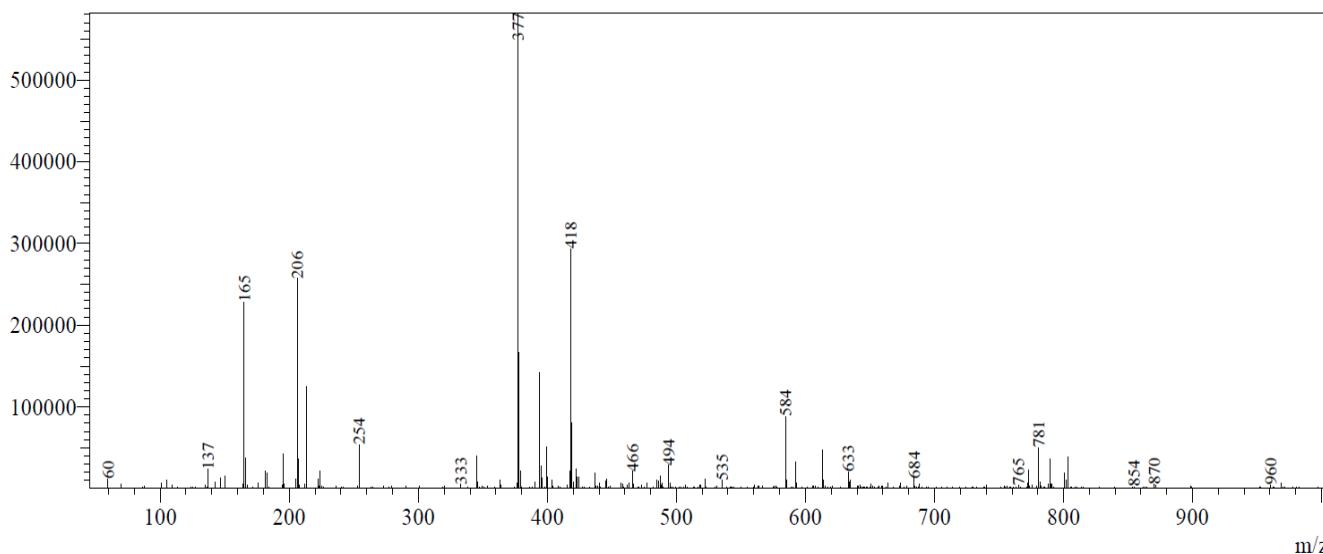
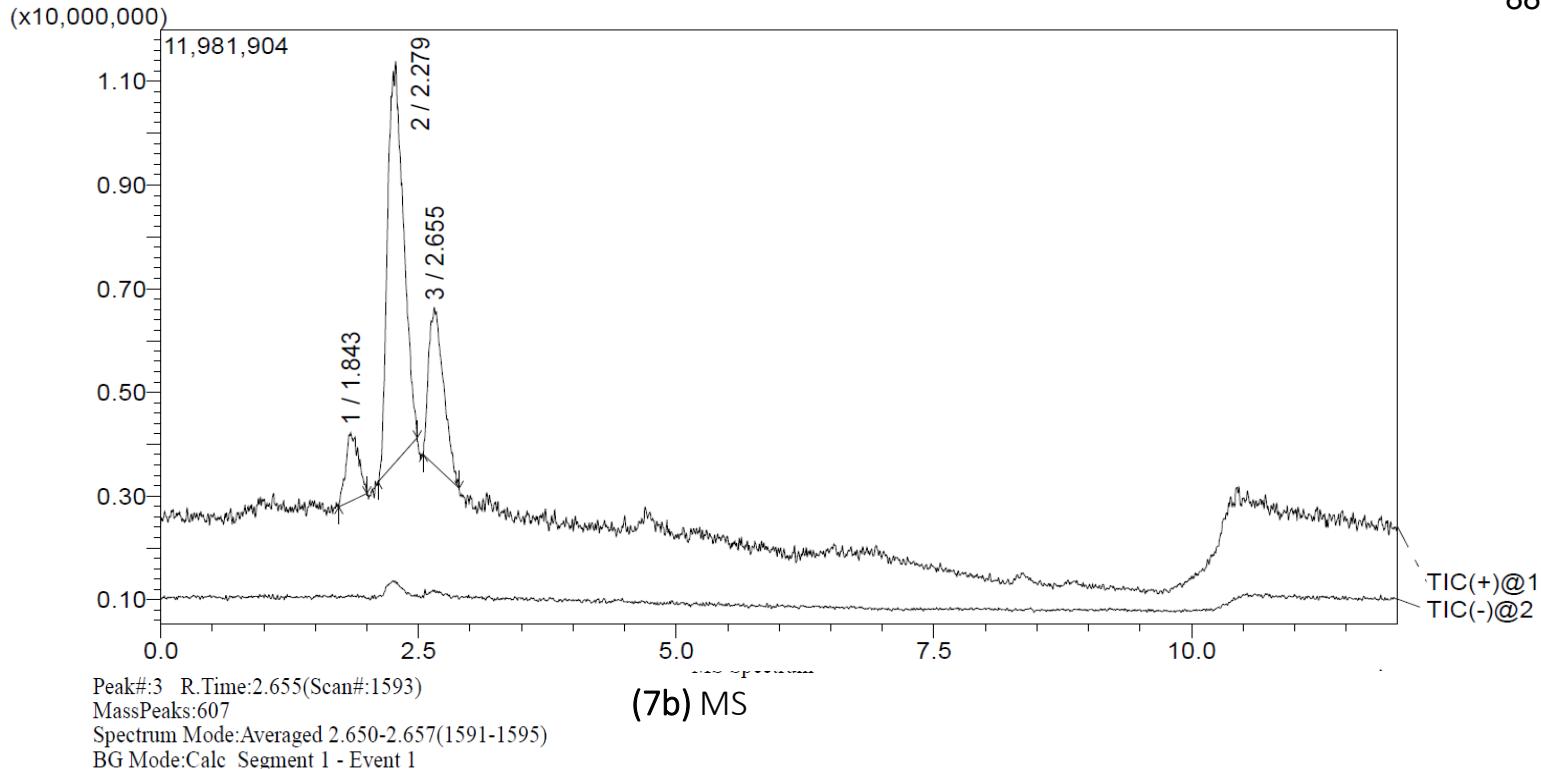
Analyst
Date

Analyst
Wednesday, 17 October 2018 2:59 PM



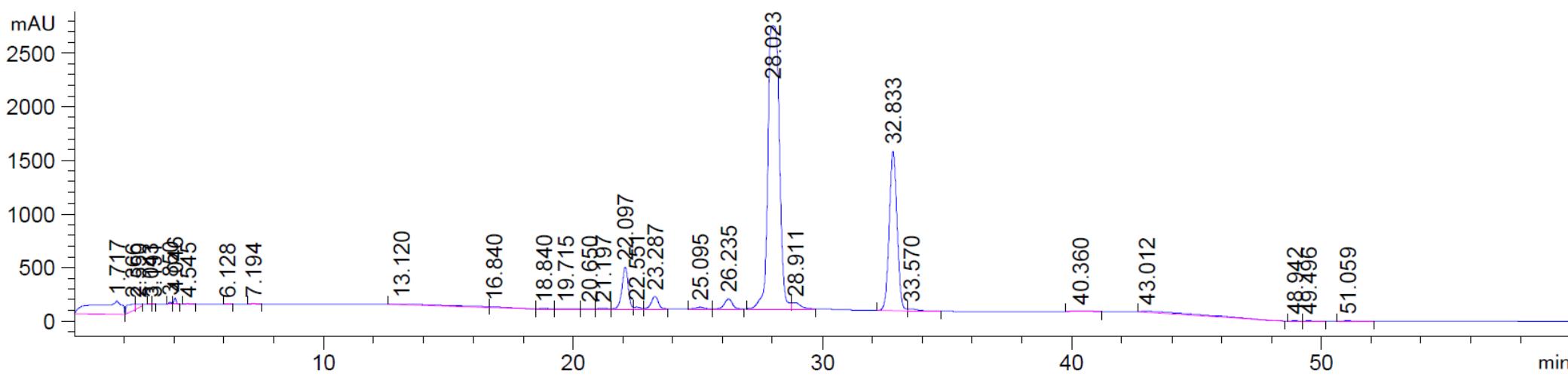
Semi-synthetic (2b) + (7b) mixture LC-MS

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HPLC analysis Semi-synthetic (2b) + (7b) mixture

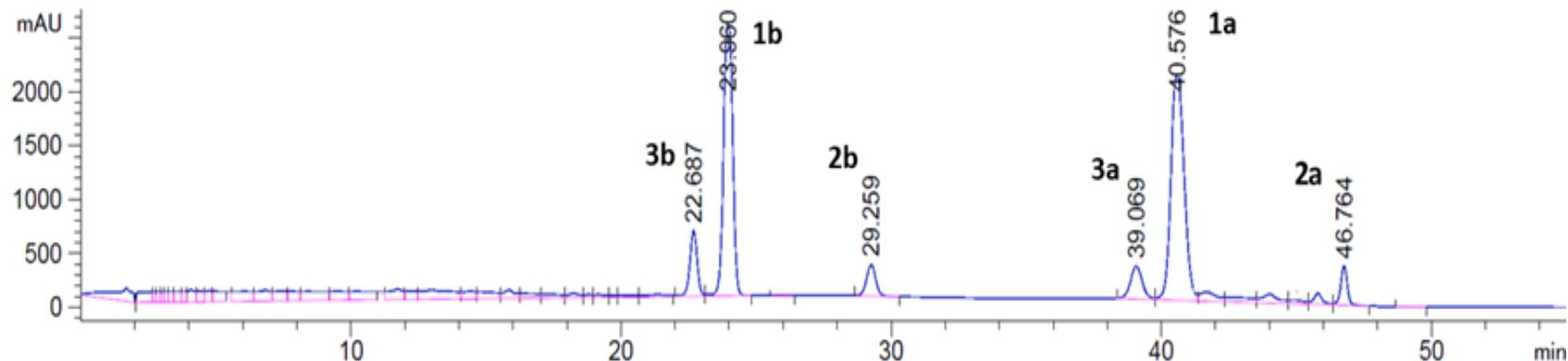
89



Retention time (min)	Compound (#)	Area (mAU)	% Area
28.023	2b	83793.2	65.8
32.833	7b	32642.4	25.6

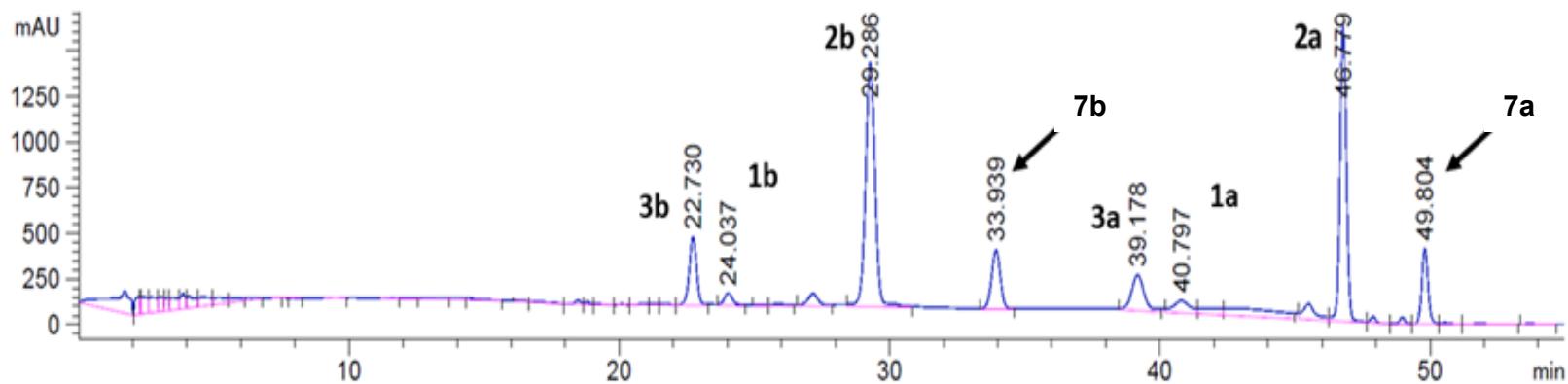
HPLC analysis Pyrethrum

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Retention time (min)	Compound (#)	Area (mAU)	% Area
23.960	1b	60141.1	36.2
29.259	2b	7049.85	4.3
40.576	1a	71498.3	43.1
46.764	2a	6303.67	3.8

Constituents	Peaks (min)	% Concentrate
Pyrethrins 1	23.960, 40.576	79.3
Jasmolins 2	29.259, 46.764	8.1

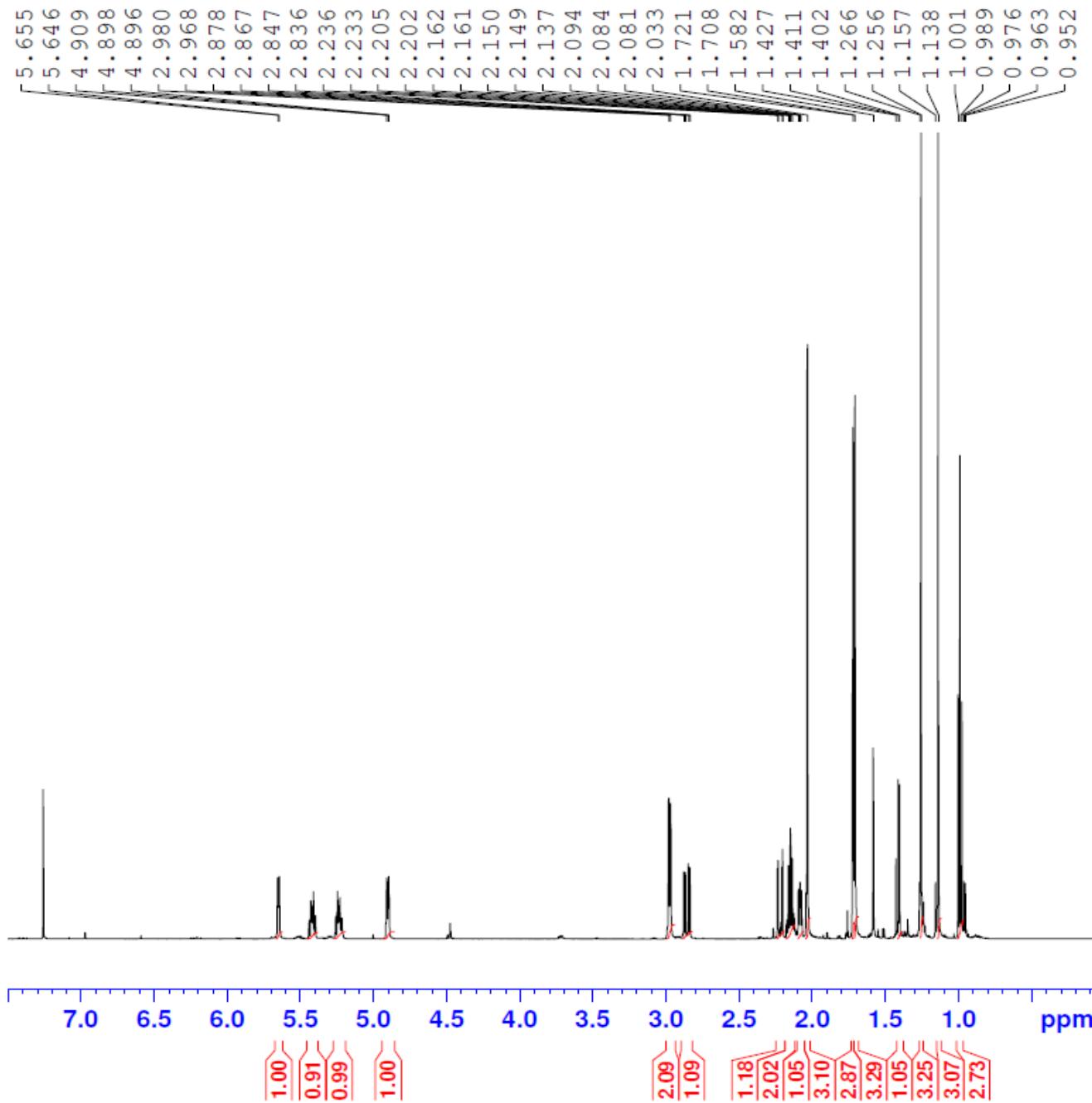


Retention time (min)	Compound (#)	Area (mAU)	% Area
24.037	1b	1587.85	1.8
29.286	2b	32595.5	35.7
40.797	1a	2928.08	3.2
46.779	2a	26629.8	29.2
33.939	7b	7251.37	8.0
49.804	7a	6641.69	7.3

Constituents	Peaks (min)	% Concentrate
Pyrethrins 1	24.037, 40.797	5.0
Jasmolins 2	29.286, 46.779	64.9
Tetrahydropyrethrins 7	33.939, 49.804	15.3

Natural (2a) NMR Characterisation

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Current Data Parameters
 NAME Natural Jasmolin I
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20191022
 Time 17.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 161
 DW 41.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 13.50 usec
 PLW1 17.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300262 MHz
 WDW EM
 SSB 0
 LB 0 0.30 Hz
 GB 0
 PC 1.00



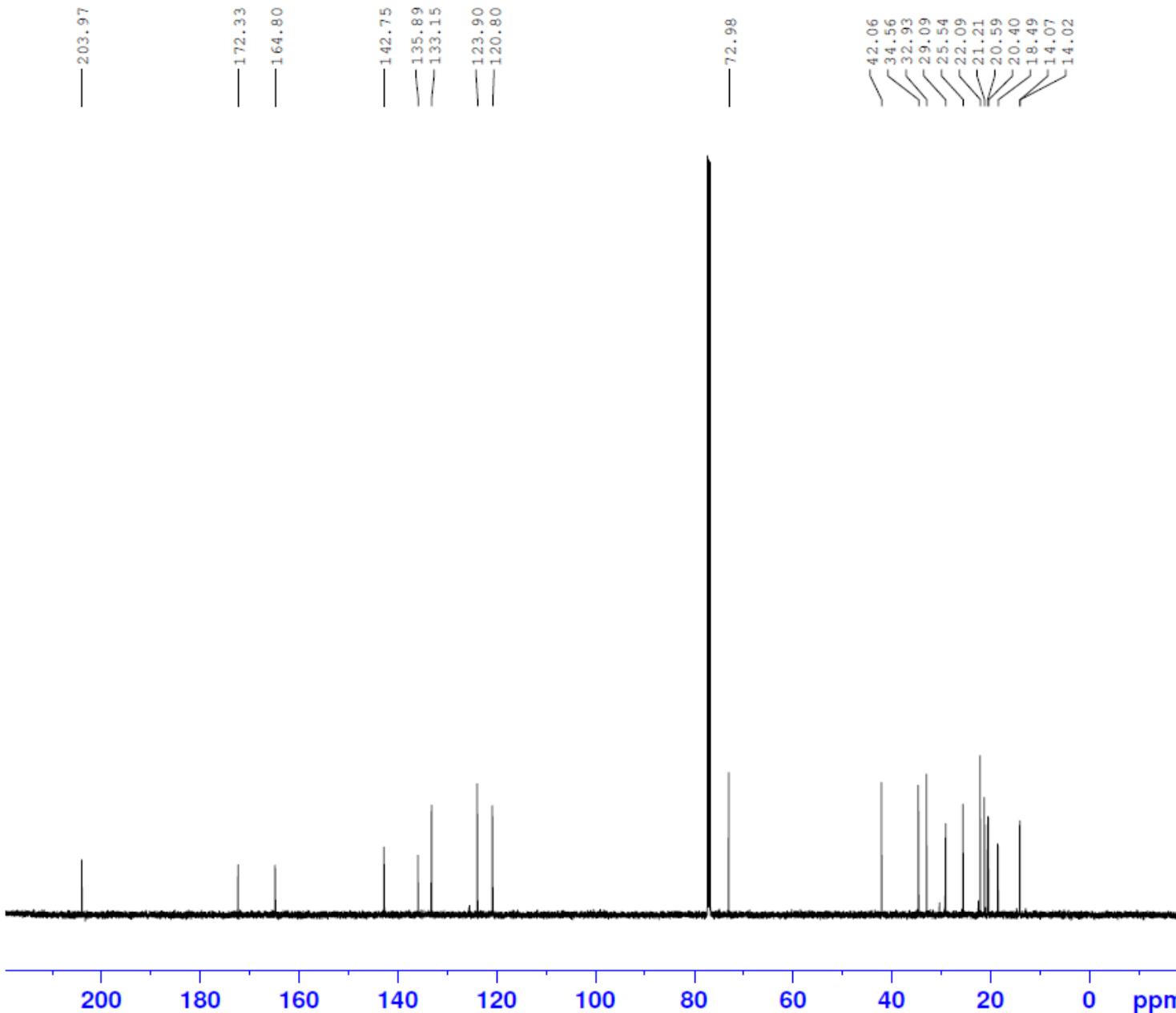
Current Data Parameters
 NAME Natural Jasmolin I
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20191022
 Time 17.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 603
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 80.00000000 W

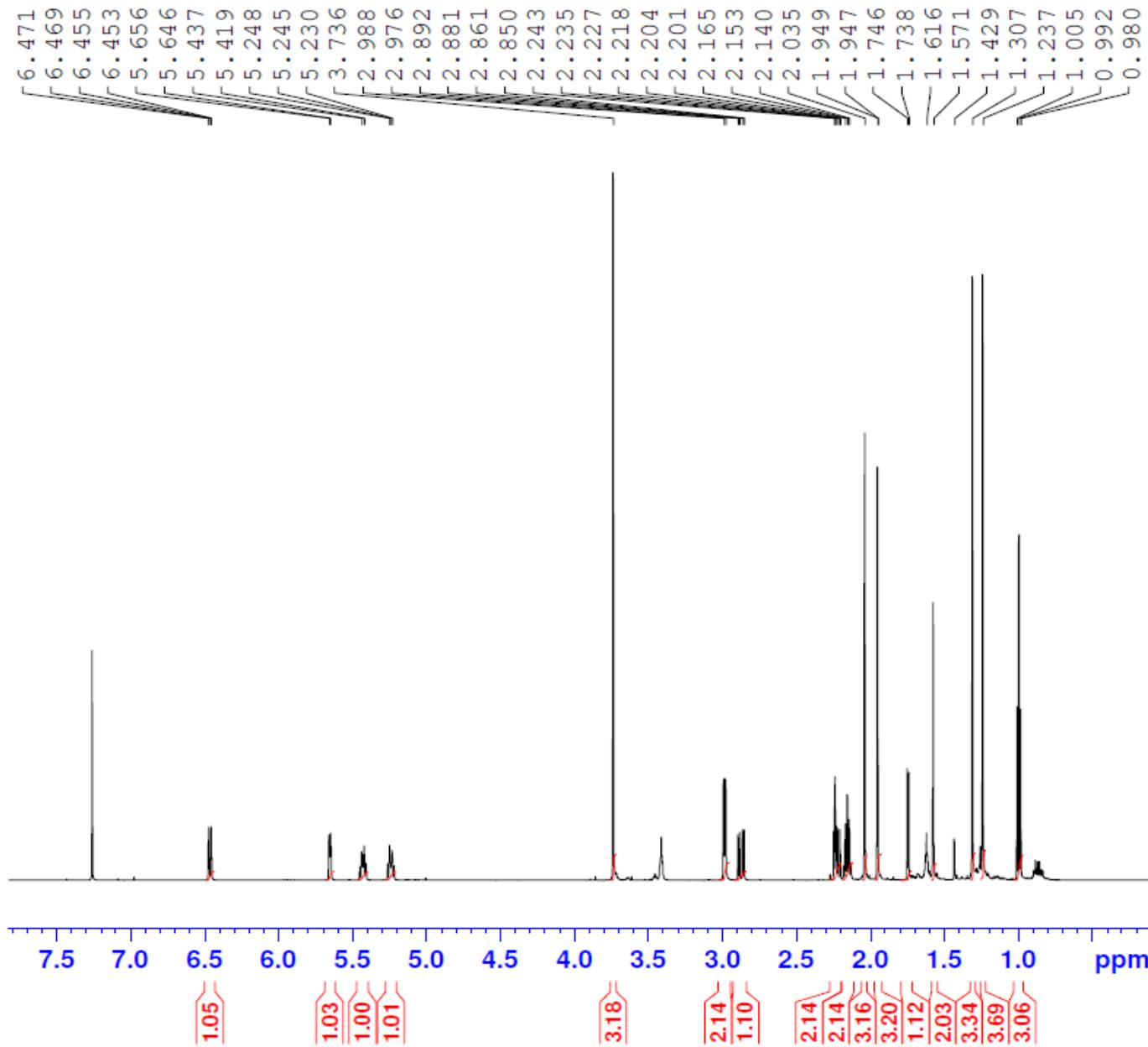
===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.00000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9028135 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Natural (2b) NMR Characterisation

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Current Data Parameters
NAME Natural Jasmolin II
EXPNO 10
PROCNO 1

F2 – Acquisition Parameters

```

Date_          20191022
Time          17.55
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG      zg30
TD            65536
SOLVENT       CDC13
NS            32
DS            2
SWH           12019.230 Hz
FIDRES       0.183399 Hz
AQ            2.7262976 sec
RG            228
DW            41.600 usec
DE            6.50  usec
TE            298.3 K
D1            1.00000000 sec
TD0           1

```

```
===== CHANNEL f1 =====  
SFO1      600.1337060 MHz  
NUC1          1H  
P1           13.50 usec  
PLW1      17.00000000 W
```

F2 - Processing parameters
SI 65536
SF 600.1300260 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME Natural Jasmin II
 EXPNO 11
 PROCNO 1

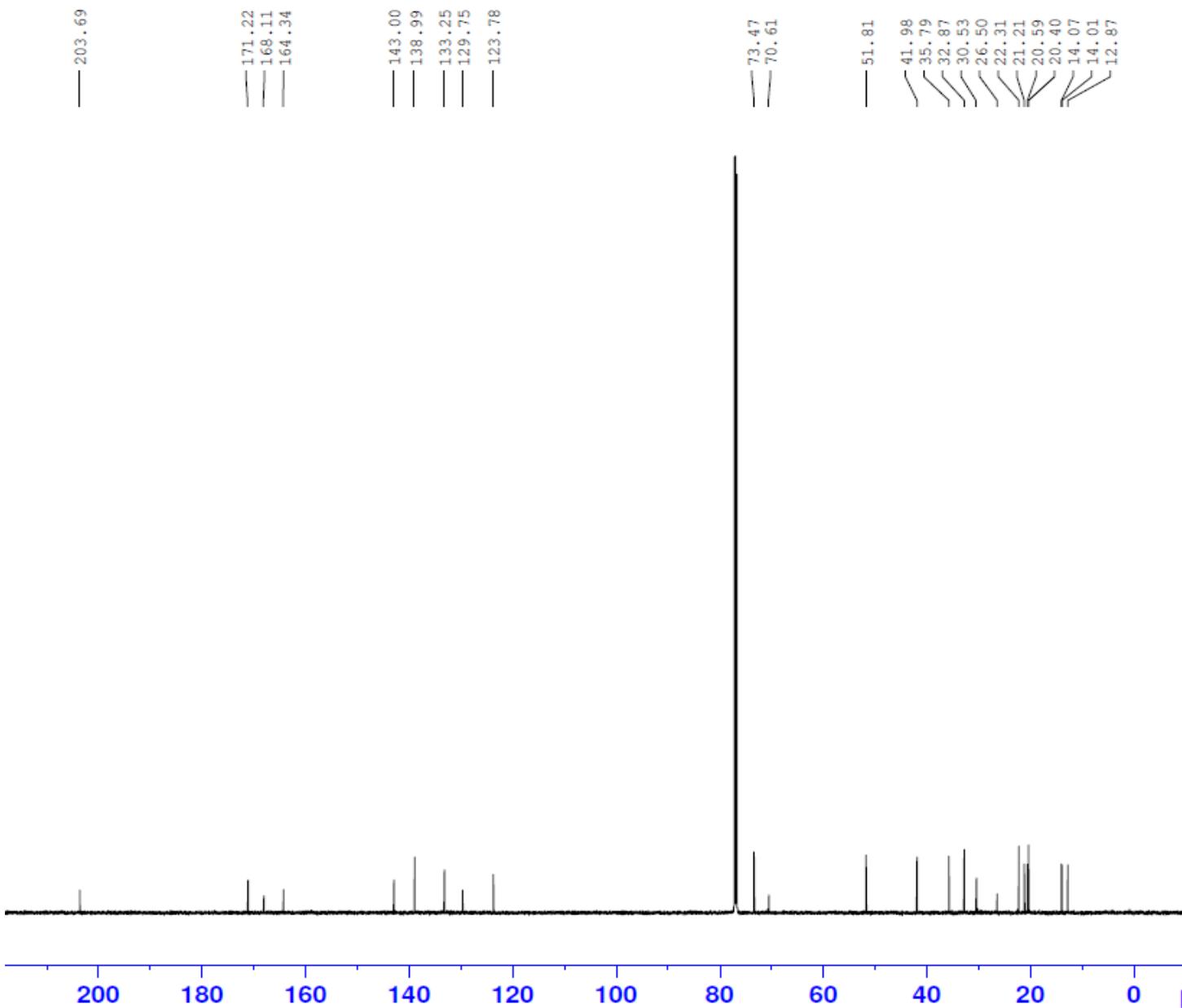
F2 - Acquisition Parameters
 Date_ 20191022
 Time 19.38
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zpgpg30
 TD 65536
 SOLVENT CDC13
 NS 2048
 DS 2
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

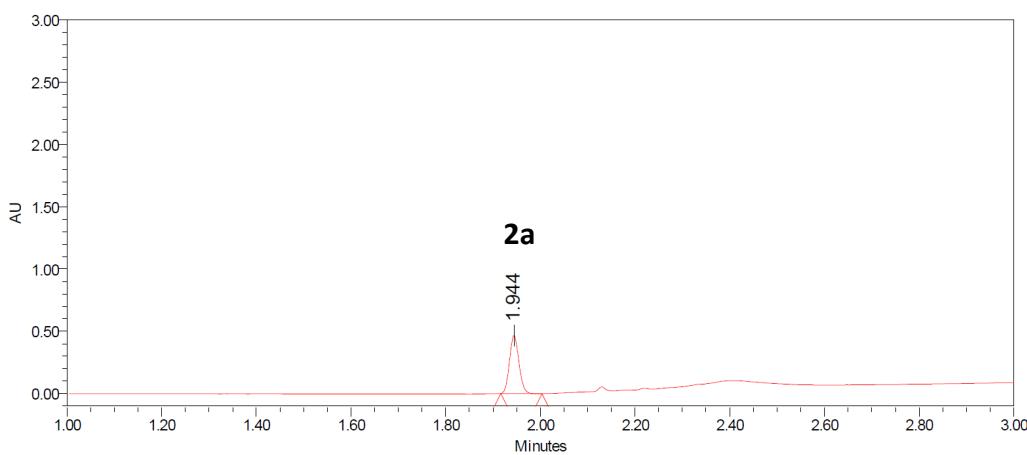
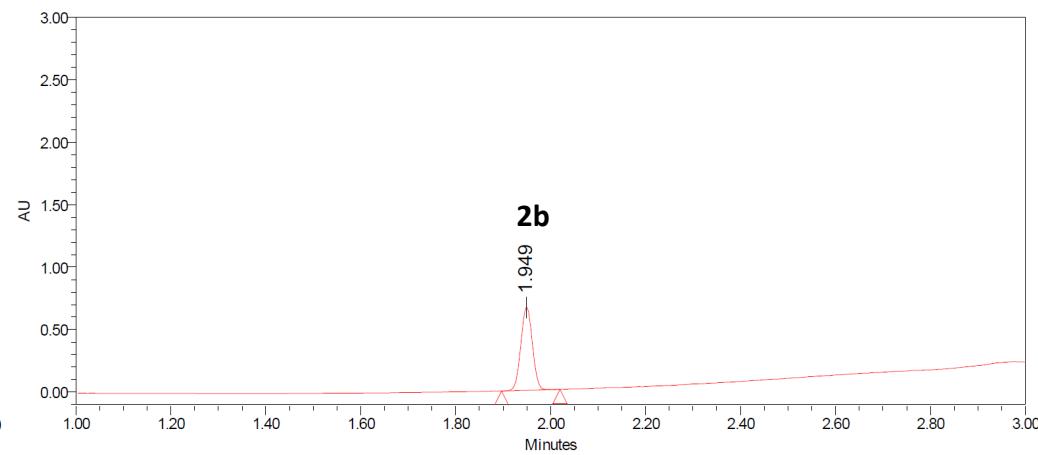
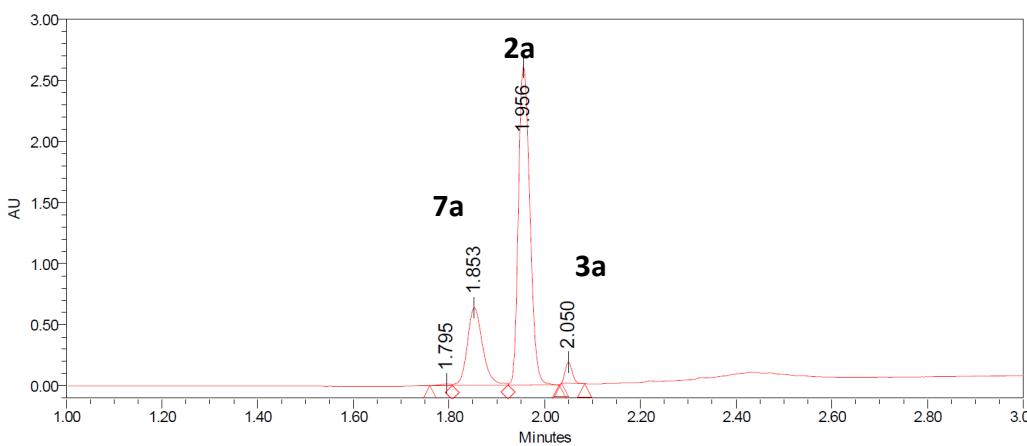
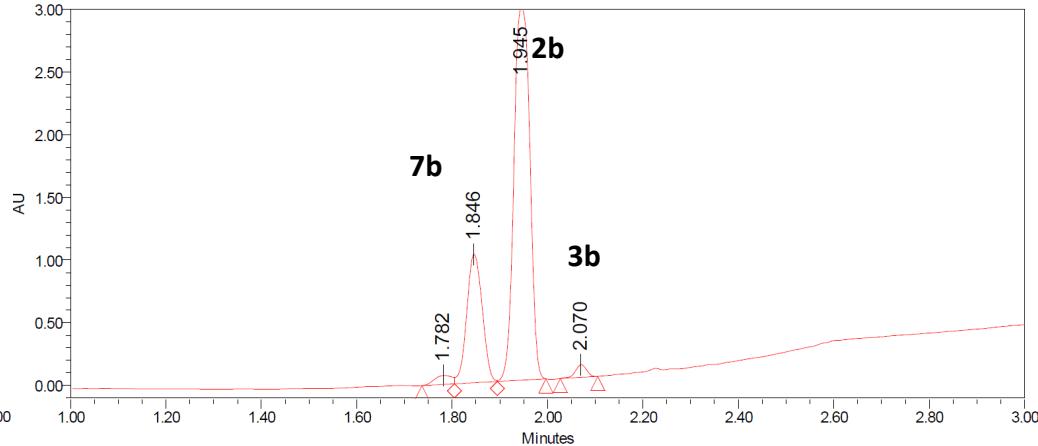
===== CHANNEL f1 =====
 SFO1 150.9178988 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 80.00000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 usec
 PLW2 17.00000000 W
 PLW12 0.63230002 W
 PLW13 0.30983001 W

F2 - Processing parameters
 SI 32768
 SF 150.9028144 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

203.69



Representative chromatogram natural jasmolin I **2a**Representative chromatogram natural jasmolin II **2b**Representative chromatogram of diimide-reduced **1a**Representative chromatogram of diimide-reduced **1b**