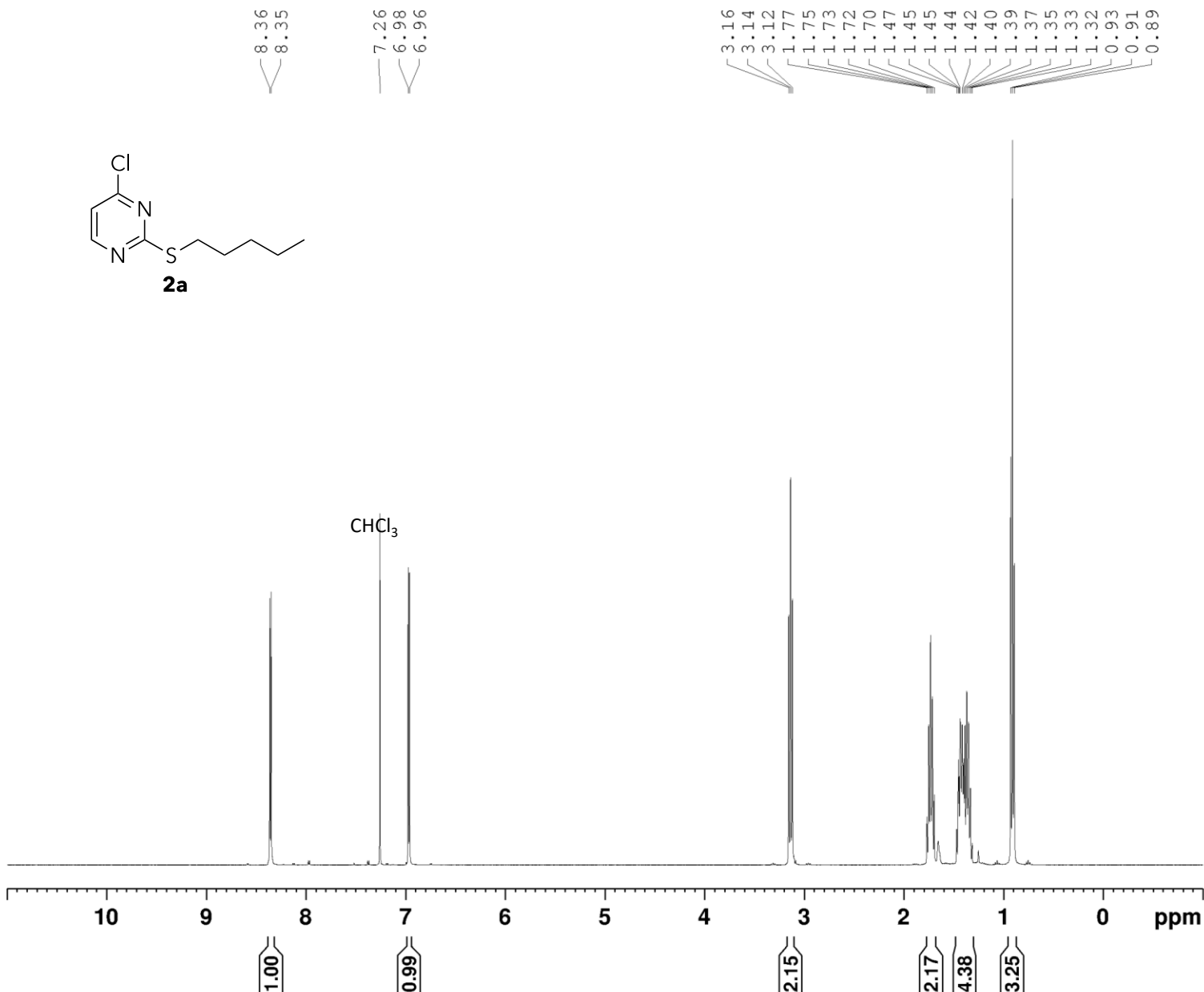


¹H spectrum of 2a

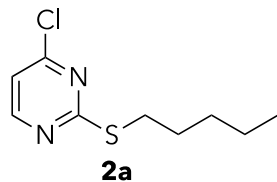


```

PROCNO              1
F2 - Acquisition Parameters
Date_               20230904
Time                13.34 h
INSTRUM             Avance Neo
PROBHD              Z152088_0031 (
PULPROG             zg30
TD                  65536
SOLVENT             CDCl3
NS                   16
DS                   2
SWH                  8196.721 Hz
FIDRES               0.250144 Hz
AQ                   3.9976959 sec
RG                   101
DW                   61.000 usec
DE                   13.89 usec
TE                   300.0 K
D1                   1.00000000 sec
TD0                  1
SFO1                 400.1324708 MHz
NUC1                 1H
P0                   2.67 usec
P1                   8.00 usec
PLW1                 24.03499985 W

F2 - Processing parameters
SI                   65536
SF                   400.1300099 MHz
WDW                  EM
SSB                  0
LB                   0.30 Hz
GB                   0
PC                   1.00
  
```

¹³C spectrum of **2a**



— 174.06

— 161.10
— 158.09

— 116.47

77.47
77.16
76.84

CDCl₃

31.27
31.12
28.71
22.35

— 14.07

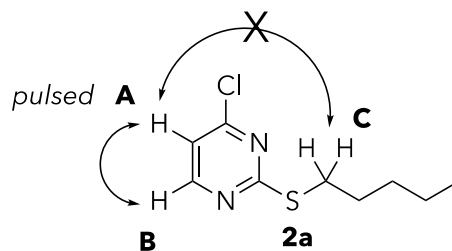
Current Data Parameters
NAME 2a_CS-2-10
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231215
Time 20.04 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 2a

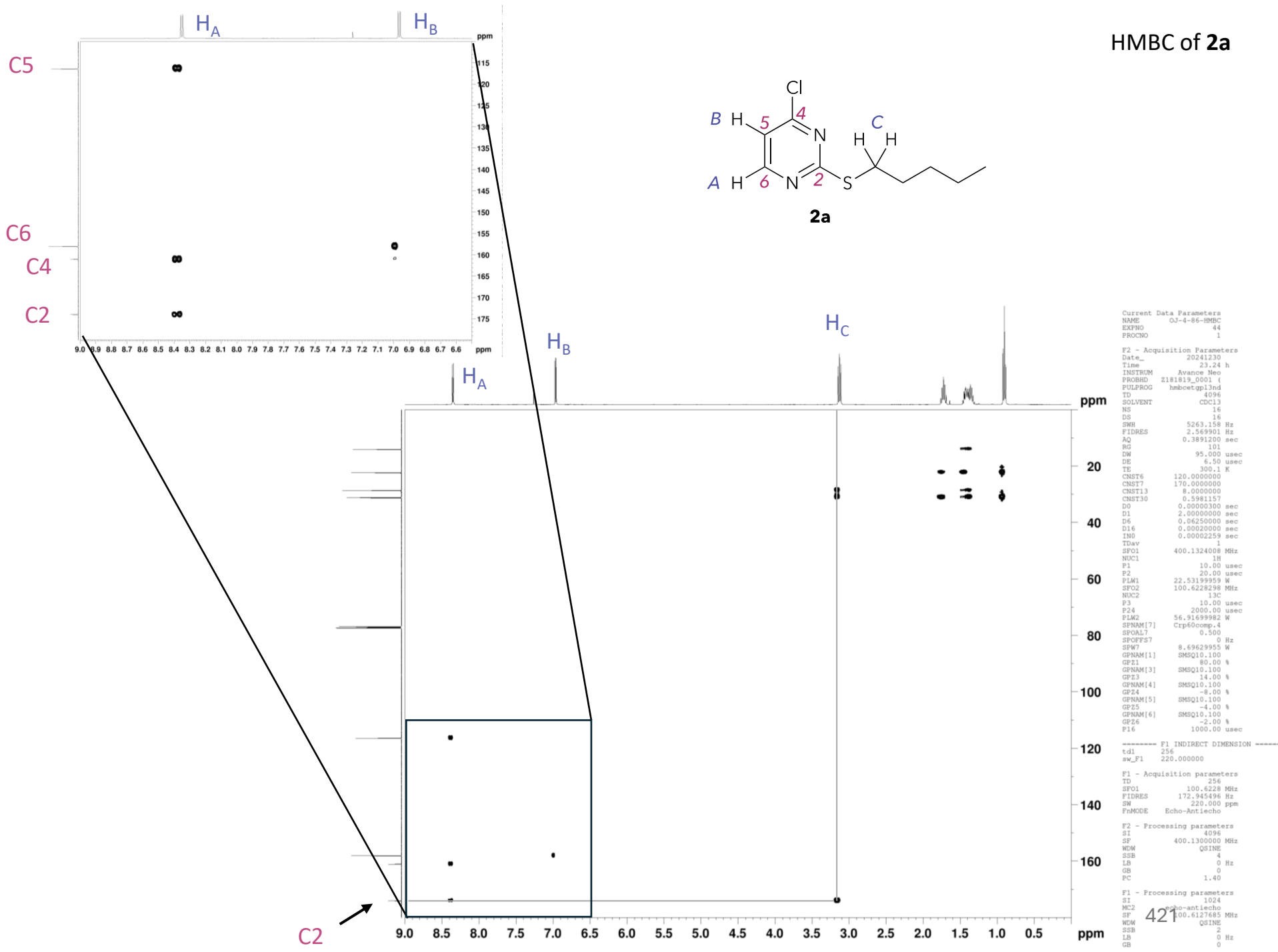
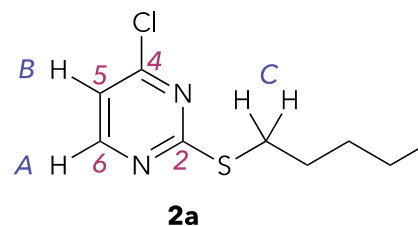


Current Data Parameters
 NAME 2a_CS-2-10
 EXPNO 12
 PROCNO 1

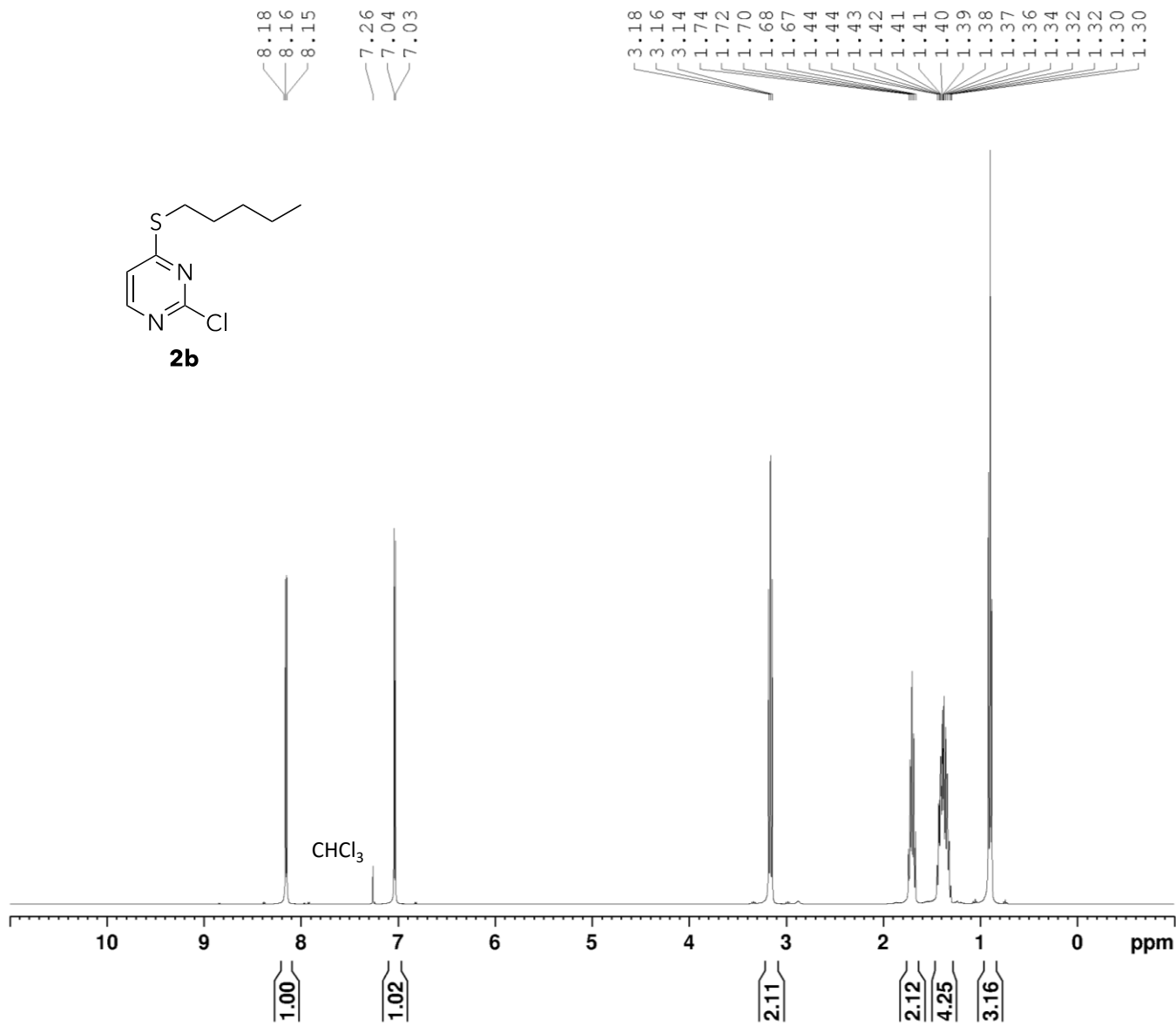
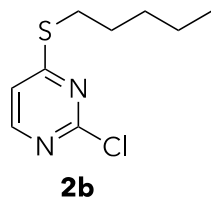
F2 - Acquisition Parameters
 Date_ 20230904
 Time 13.40 h
 INSTRUM Avance Neo
 PROBHD Z152088_0031 (PI HR-BBO400S1-BB
 PULPROG selnoggps.2
 TD 65536
 SOLVENT CDC13
 NS 32
 DS 4
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 10.16 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27266601 sec
 D31 0.30000001 sec
 TD0 1
 ZGPTNS -DCALC_SPOFFS
 SF01 400.1324708 MHz
 NUC1 1H
 CNST21 6.9886608
 P1 8.00 usec
 P12 35612.27 usec
 P32 20000.00 usec
 P44 300.00 usec
 PLW1 24.03499985 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 SPW2 0.00002864 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 SPW29 0.05875600 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 SPW30 1.09389997 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 400.1300138 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

HMBC of 2a



¹H spectrum of **2b**

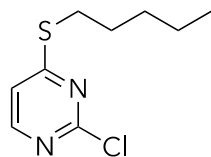


Current Data Parameters
NAME OJ-3-67-H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240511
Time 15.02 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5.0-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 57
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **2b**



2b

174.04
160.95
156.08
117.48
77.47
77.16
76.84
30.98
29.77
28.46
22.23
13.99

CDCl₃

Current Data Parameters

NAME OJ-3-67-C
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters

Date_ 20240511
Time 16.03 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

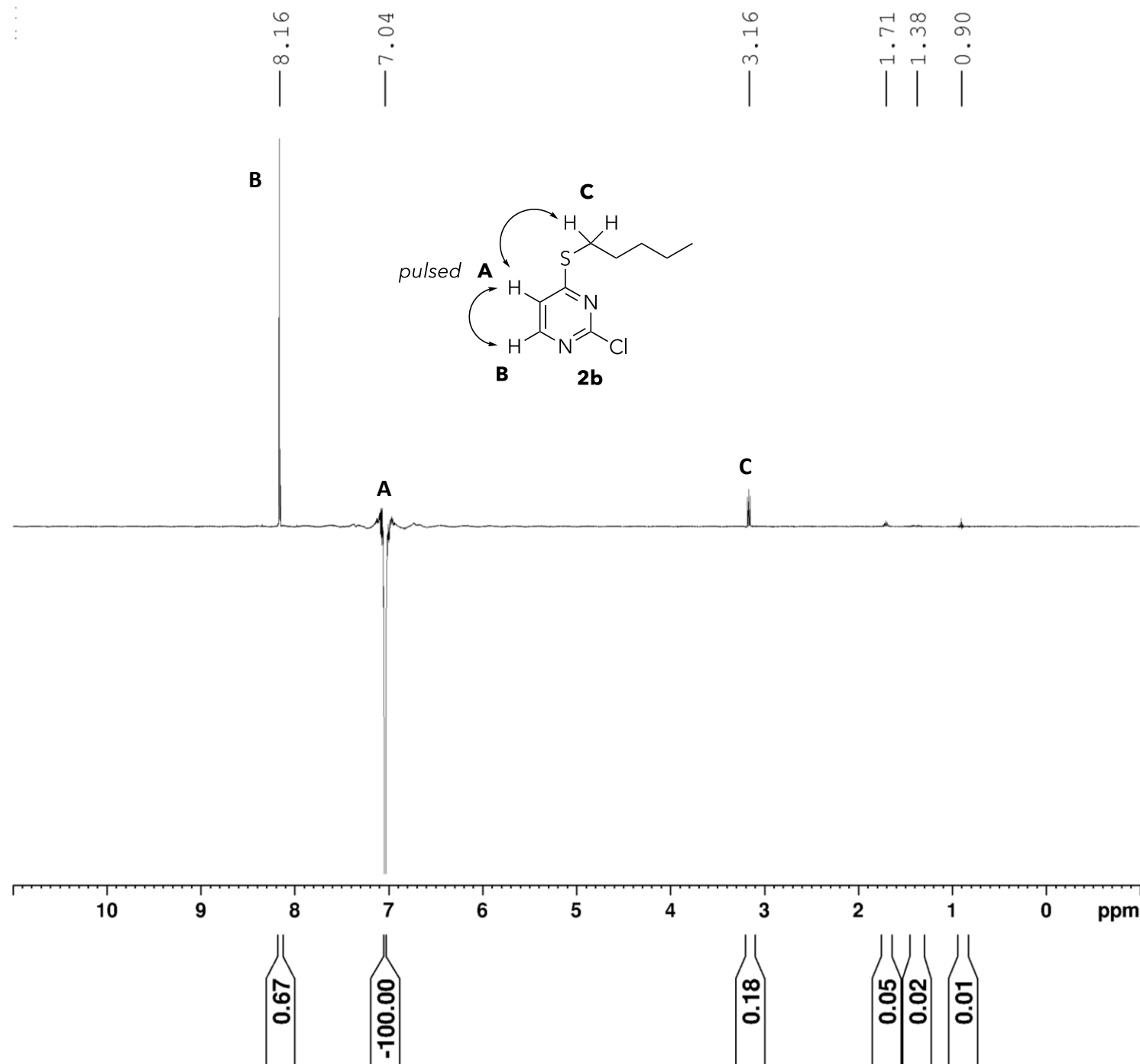
SI 32768
SF 100.6127588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **2b**

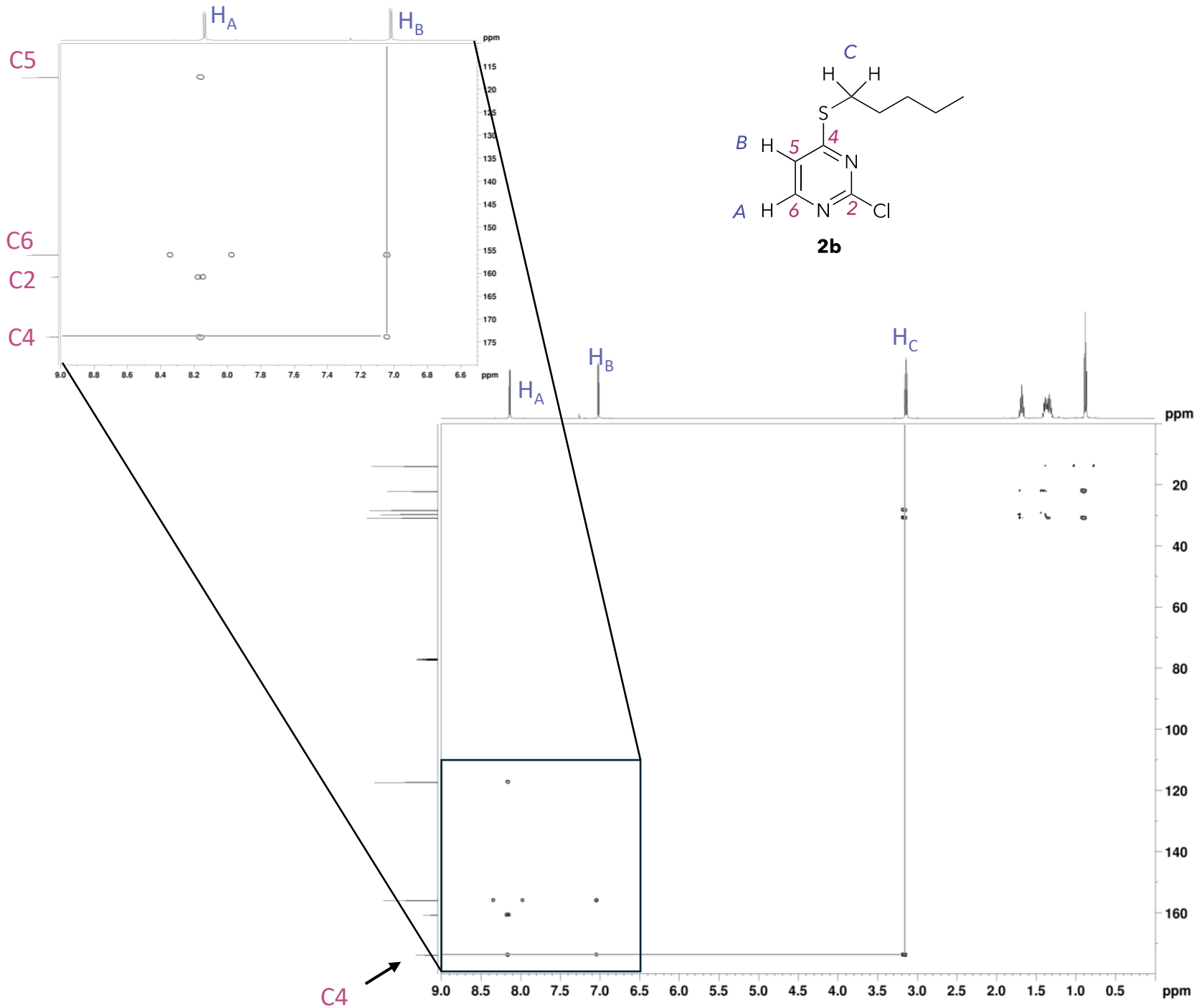
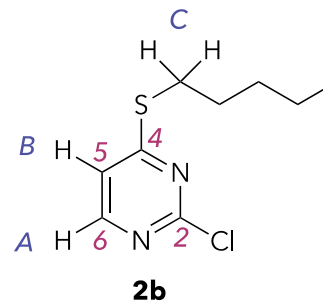
Current Data Parameters
NAME OJ-3-67-C4-NOE-Spin-1
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230218
Time 11.40 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnpgpzs.2
TD 65536
SOLVENT CDC13
NS 64
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 7.0481420
P1 12.00 usec
P12 56514.12 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 436.77 Hz
SPW2 0.00001219 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

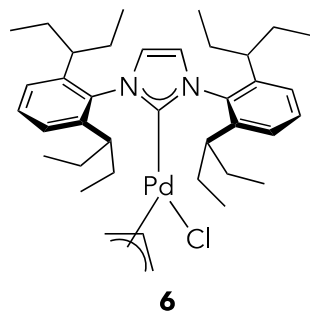
F2 - Processing parameters
SI 32768
SF 500.2300050 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



HMBC of 2b



¹H spectrum of **6**



7.40
7.38
7.36
7.26
7.17
7.15
7.09
4.84
4.82
4.80
4.79
4.78
4.77
4.76
4.74
3.91
3.89
2.90
2.87
2.81
2.56
2.44
2.04
2.01
1.92
1.73
1.61
1.43
1.40
0.99
0.97
0.96
0.79
0.78
0.72
0.71

CHCl₃

acetone

Current Data Parameters
NAME OJ-6-159
EXPNO 15
PROCNO 1

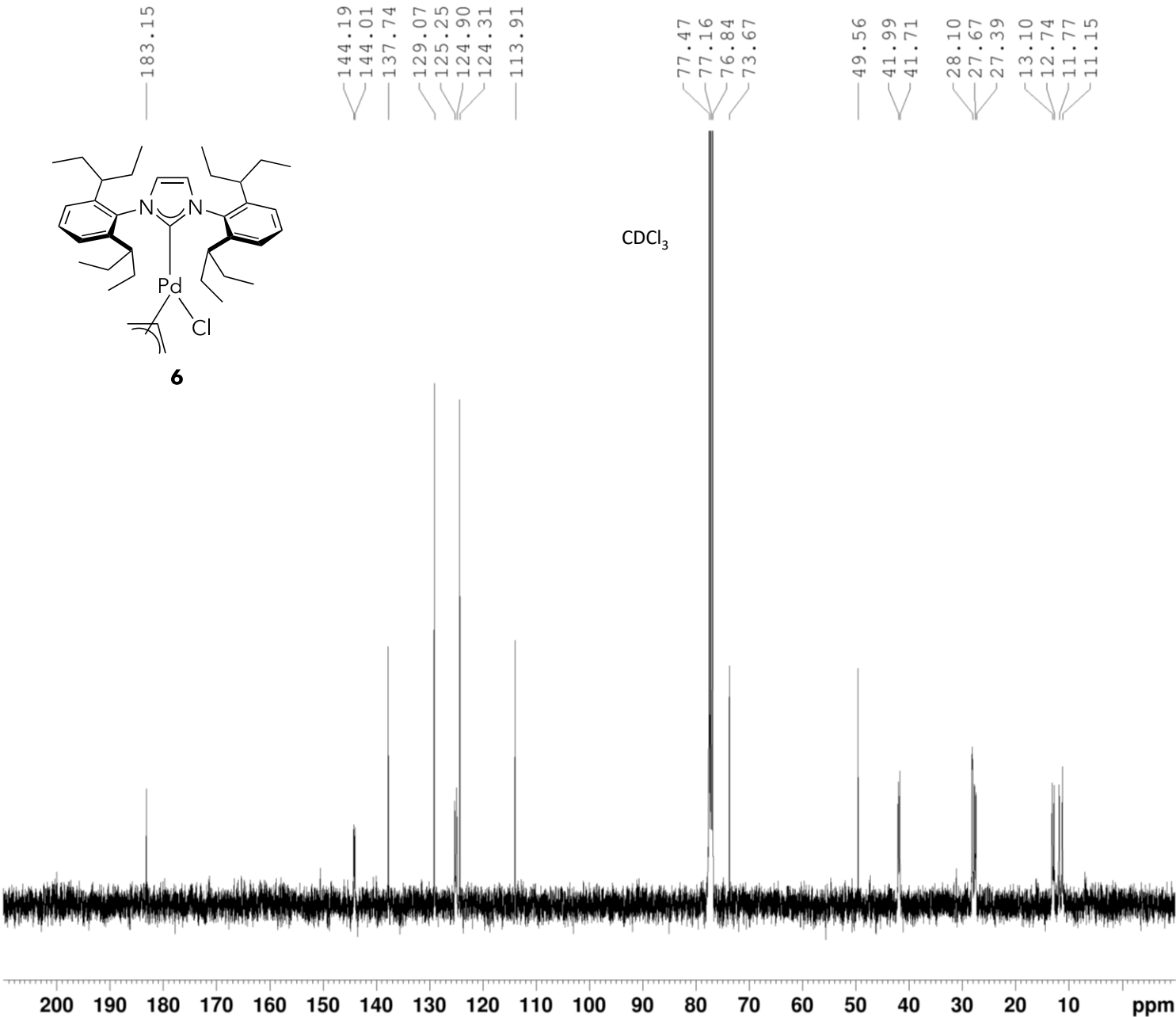
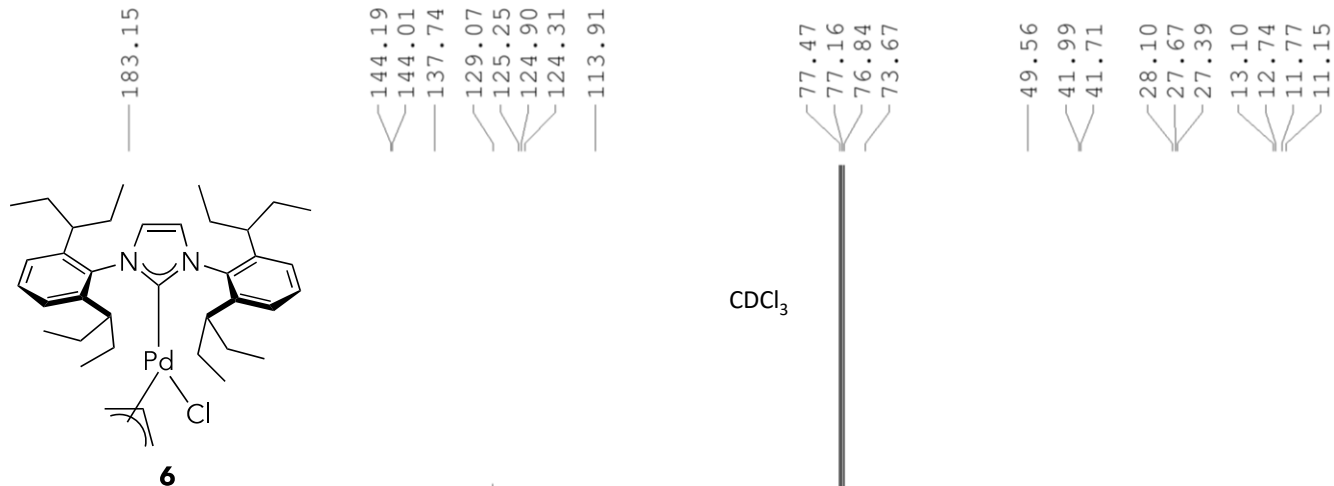
F2 - Acquisition Parameters
Date_ 20240311
Time 21.26 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 9 8 7 6 5 4 3 2 1 0 ppm

1.95
3.85
1.76
1.00
0.96
0.98
0.85
4.12
4.24
9.77
2.79
11.81
11.91

¹³C spectrum of 6

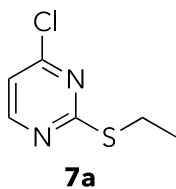


Current Data Parameters
NAME OJ-6-159-C
EXPNO 17
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240312
Time 0.24 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H spectrum of **7a**



8.36
8.35

7.26
6.97
6.96

3.14
3.13

1.38

CHCl₃

0.84

0.98

2.12

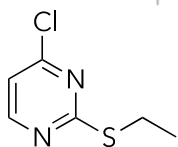
3.22

Current Data Parameters
NAME OJ-5-17-H
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230508
Time 18.41 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 37.93
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 7



7a

— 173.79

— 161.08

— 158.10

— 116.48

77.41

77.16

76.90

— 25.61

— 14.26

CDCl₃

Current Data Parameters
NAME OJ-5-17-C
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230508
Time 20.48 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters
SI 32768
SF 125.7829217 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

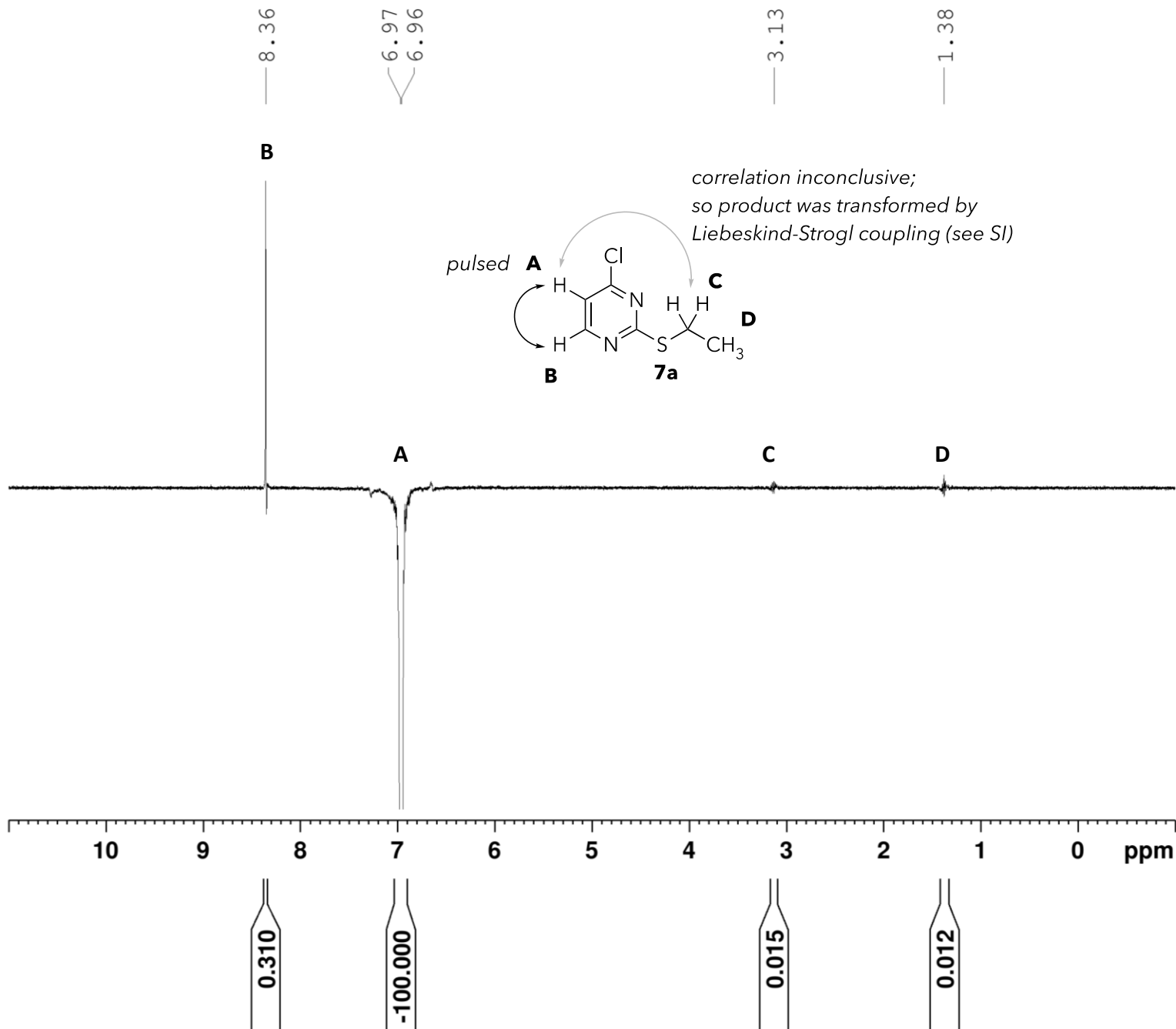
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 7a

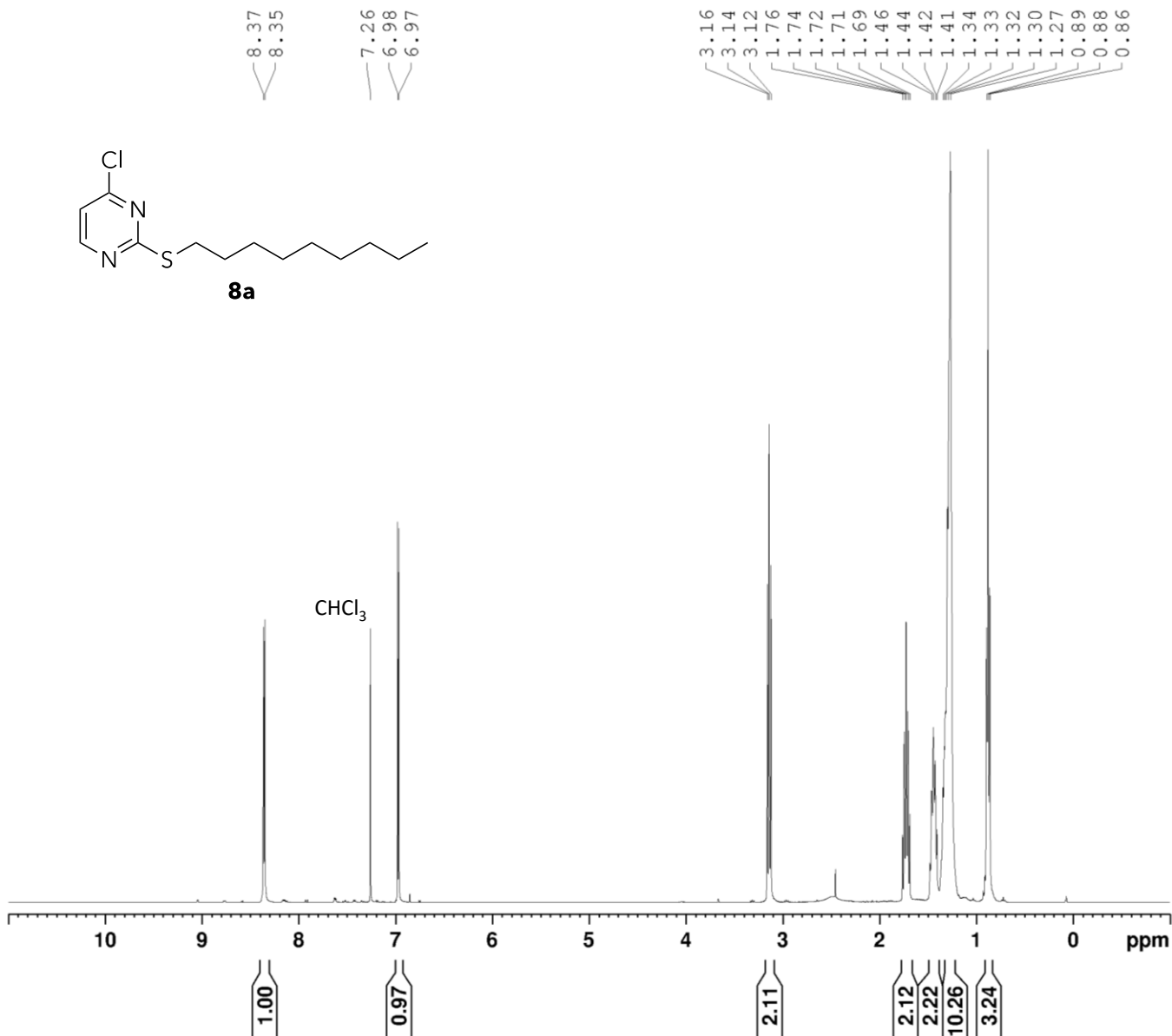
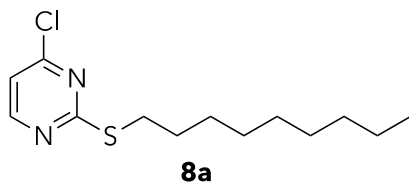
Current Data Parameters
NAME OJ-5-17-NOE
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230509
Time 14.13 h
INSTRUM spect
PROBHD Z125869_0055 ()
PULPROG selnogpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 6.9867911
P1 12.00 usec
P12 33026.49 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 406.08 Hz
SPW2 0.00003568 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300133 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **8a**



Current Data Parameters

NAME OJ-241108-1-H-final
EXPNO 11
PROCNO 1

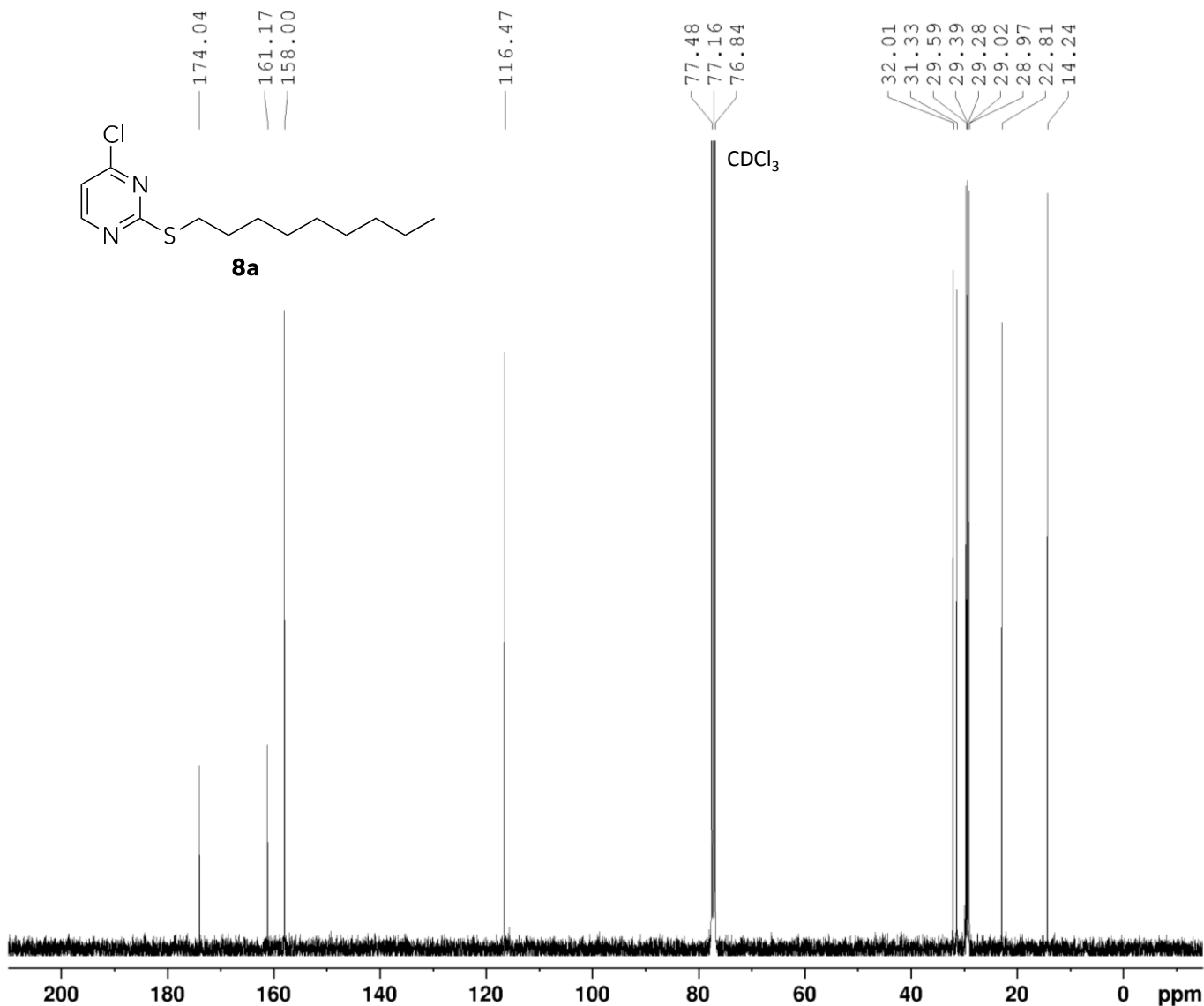
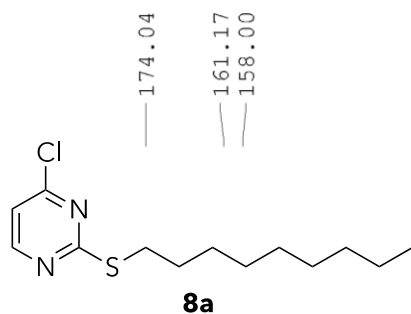
F2 - Acquisition Parameters

Date_ 20241109
Time 19.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5.0
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 8a



Current Data Parameters

NAME OJ-241108-1-C
EXPNO 13
PROCNO 1

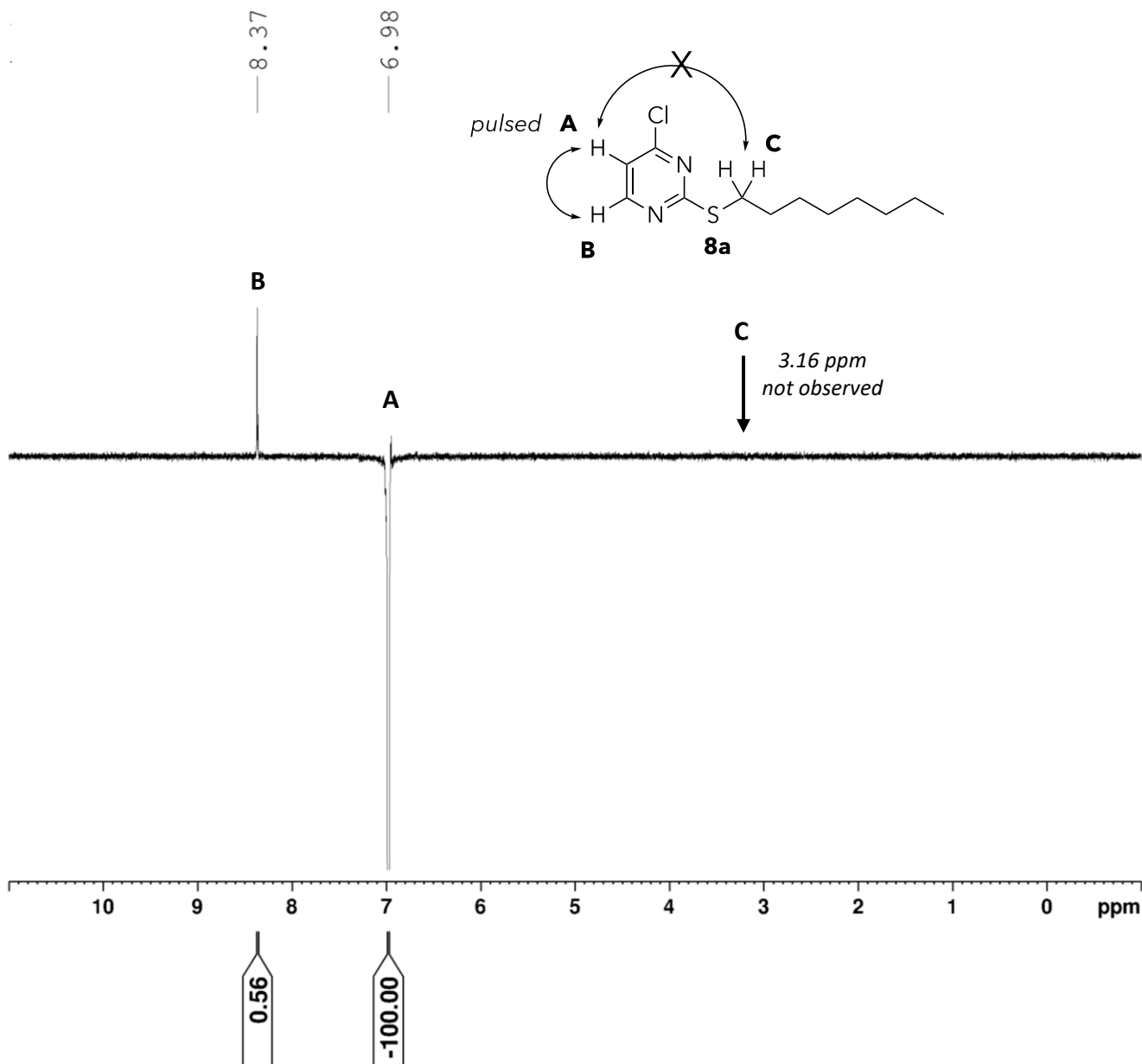
F2 - Acquisition Parameters

Date_ 20241109
Time 20.38 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

SI 32768
SF 100.6127537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 8a

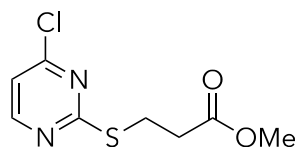


Current Data Parameters
NAME OJ-241108-1-NOE
EXPNO 2
PROCNO 1

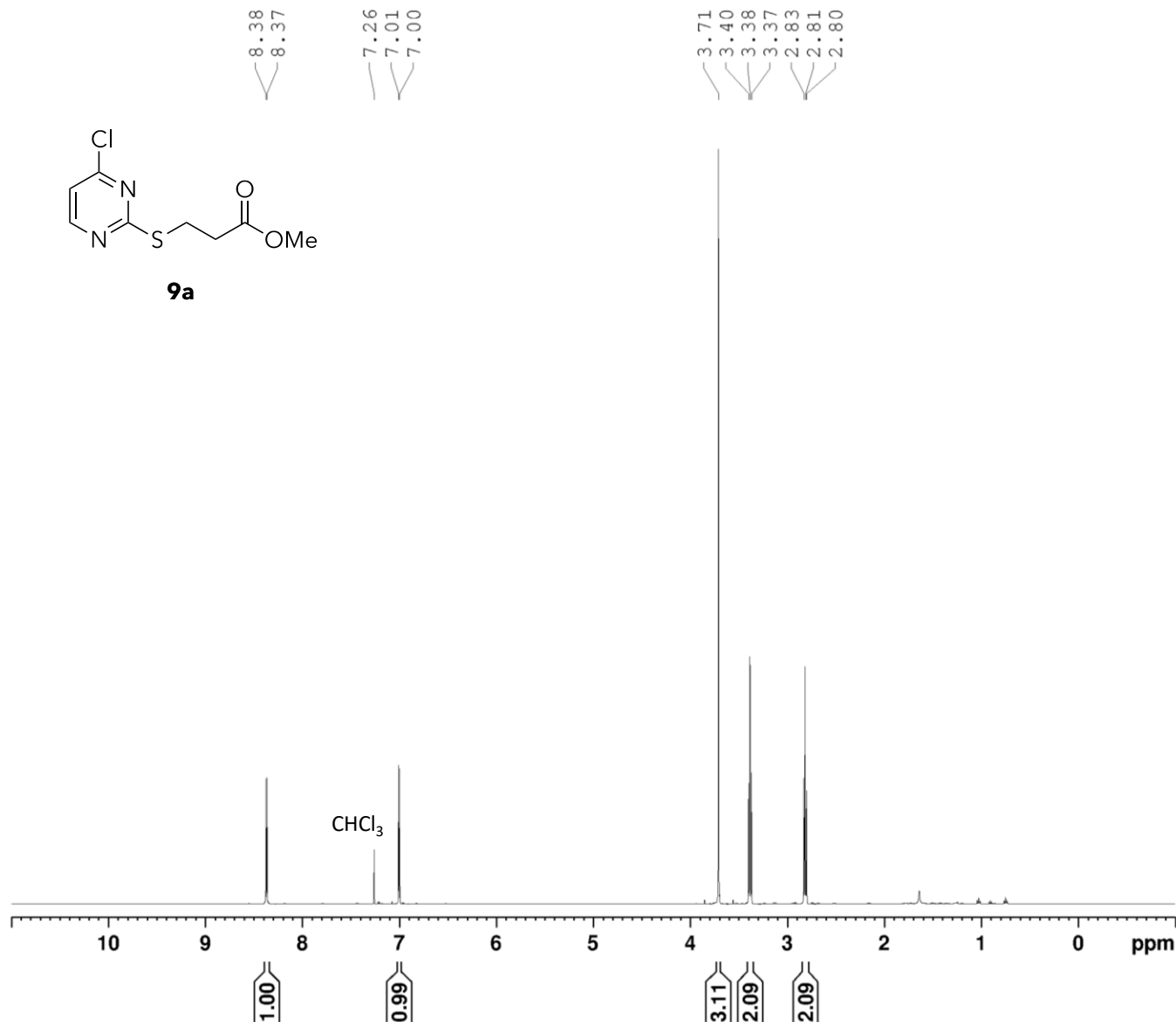
F2 - Acquisition Parameters
Date_ 20241109
Time 21.33 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S1 BB-H&F-05
PULPROG selnpgpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 6.9972119
P1 12.00 usec
P12 57526.85 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 411.30 Hz
SPW2 0.00001176 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300084 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H spectrum of **9a**



9a



Current Data Parameters

NAME OJ-5-5-H
EXPNO 10
PROCNO 1

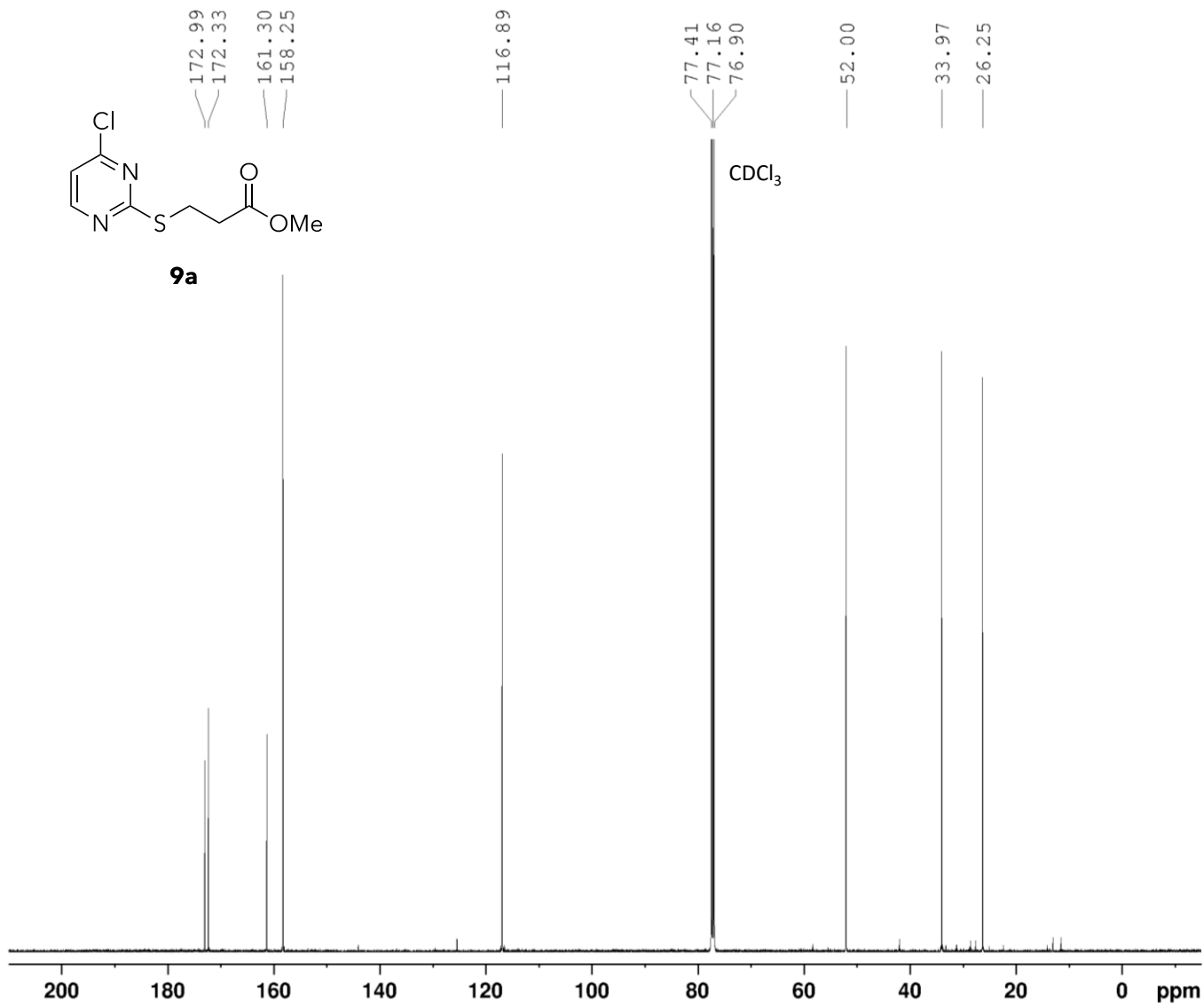
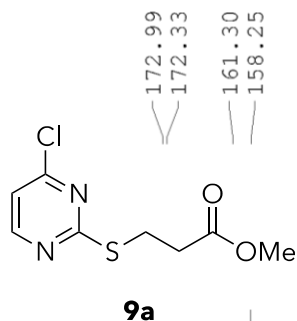
F2 - Acquisition Parameters

Date_ 20230821
Time 12.44 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S1 B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 107.7
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters

SI 65536
SF 500.2300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 9a



Current Data Parameters
NAME OJ-5-5-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230821
Time 13.52 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S1 B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

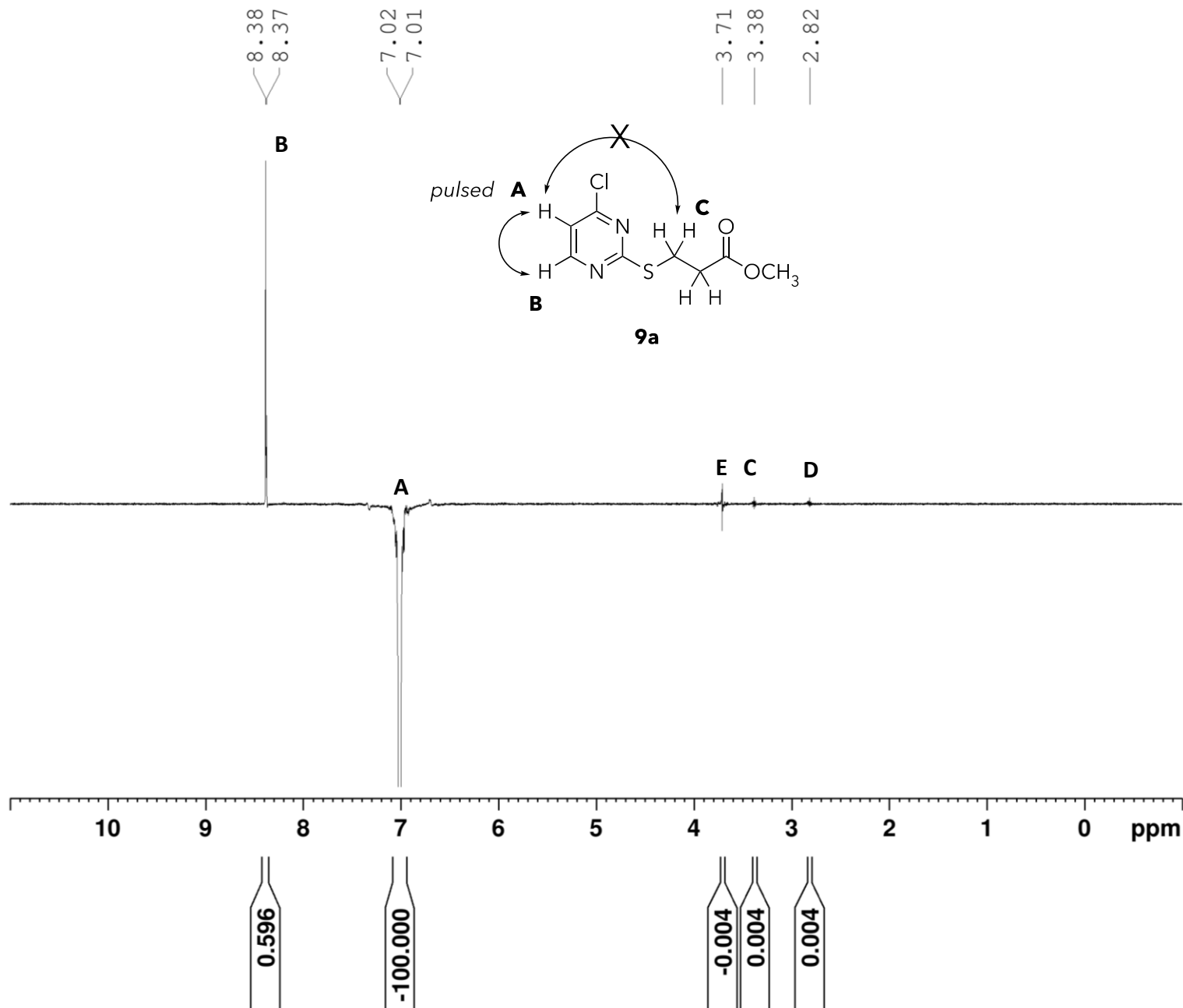
F2 - Processing parameters
SI 32768
SF 125.7829191 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 9a

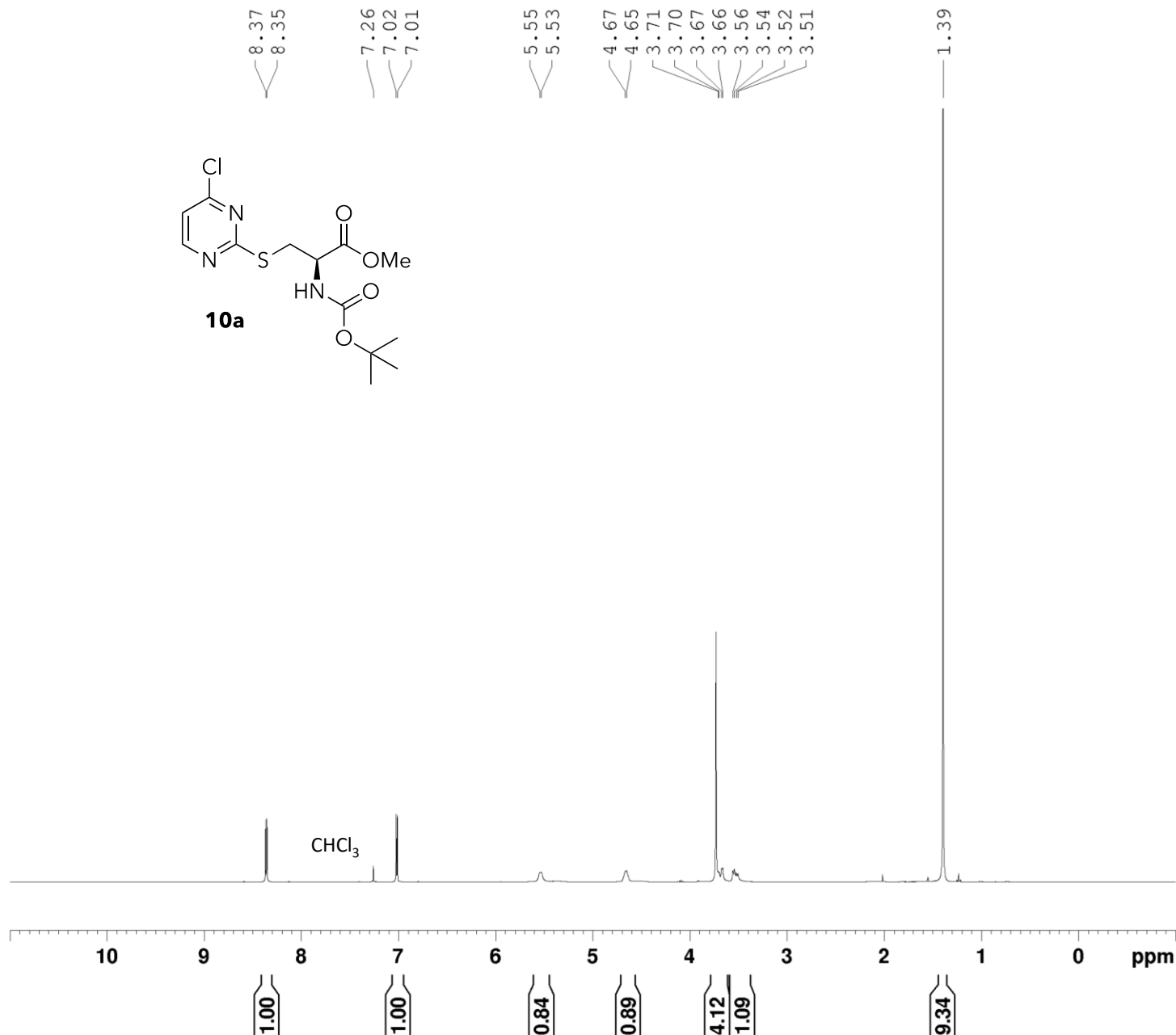
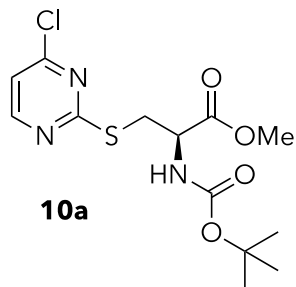
Current Data Parameters
NAME OJ-5-5-NOE
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230506
Time 16.10 h
INSTRUM spect
PROBHD Z125869_0055 (PULPROG selnpgps.2)
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0094762
P1 12.00 usec
P12 54191.82 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 417.43 Hz
SPW2 0.00001325 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of 10a

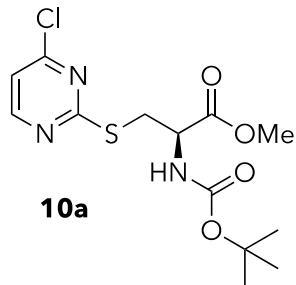


Current Data Parameters
NAME OJ-6-91-H
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231228
Time 21.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (zg30)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 71.8
DW 61.000 usec
DE 13.54 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 10a



172.50
171.28
161.27
158.16
155.24

117.23

80.18
77.48
77.16
76.84

53.36
52.68

33.29
28.36

CDCl₃

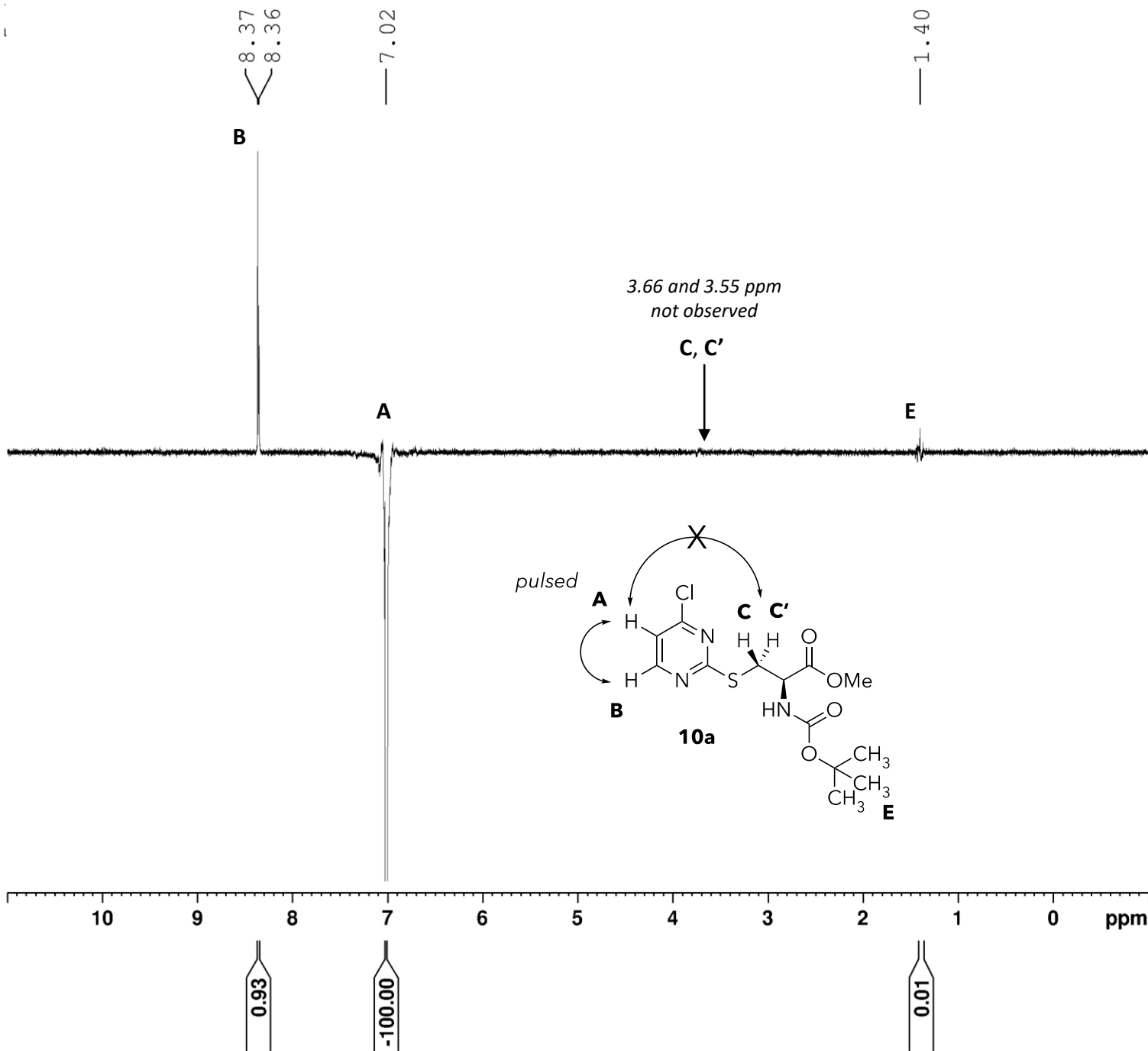
Current Data Parameters
NAME OJ-6-91-C
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231228
Time 23.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.9169982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

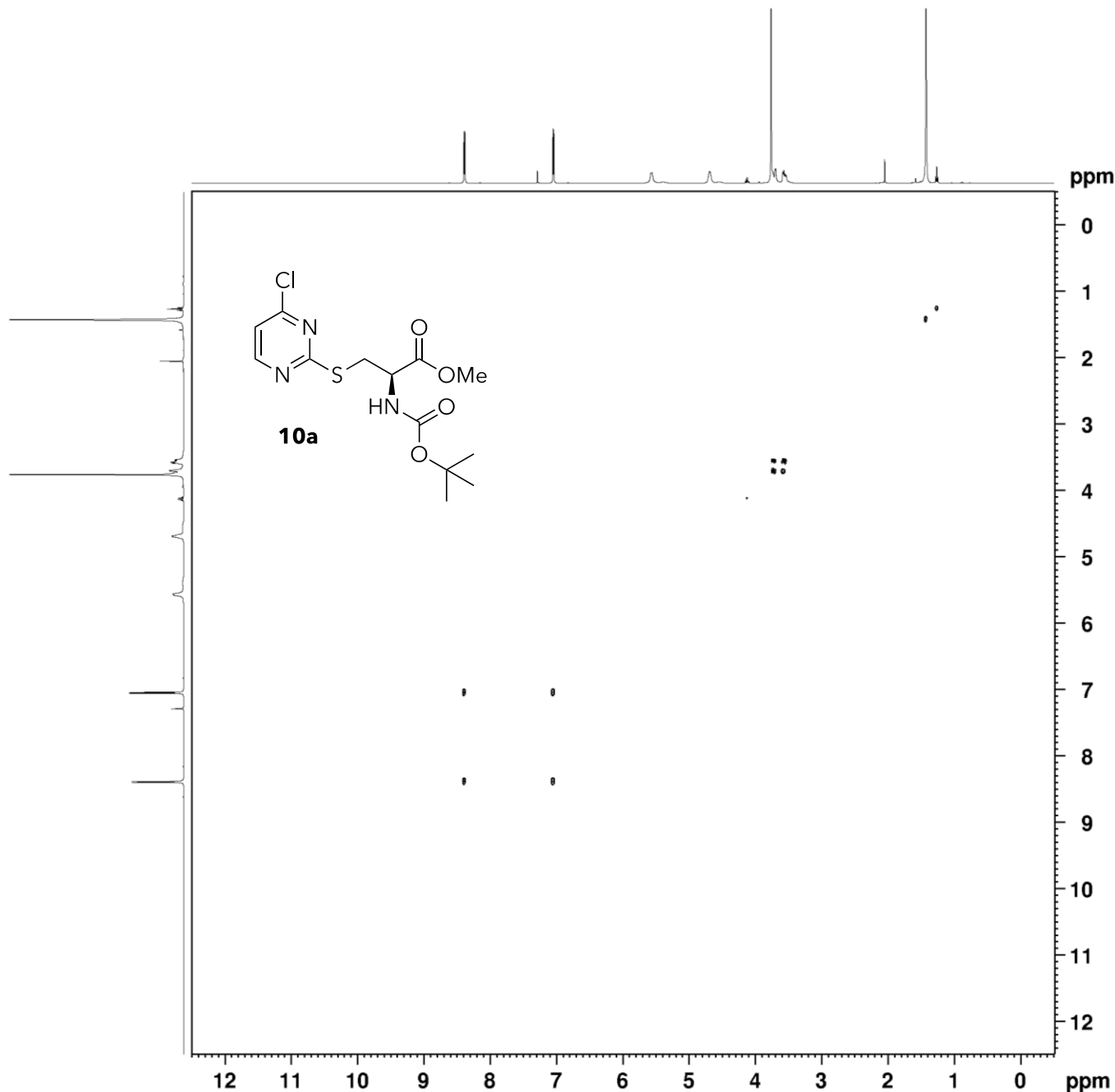
F2 - Processing parameters
SI 32768
SF 100.6127582 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 10a



COSY of 10a



Current Data Parameters
NAME OJ-6-91-COSY
EXPNO 15
PROCNO 1

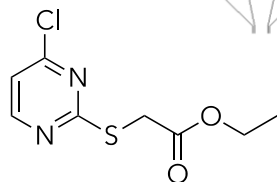
F2 - Acquisition Parameters
Date_ 20240911
Time 17.20 h
INSTRUM spect
PROBHD Z125869_0055 ()
PULPROG cosygpmfppqf
TD 4096
SOLVENT CDCl3
NS 4
DS 16
SWH 6510.417 Hz
FIDRES 3.178914 Hz
AQ 0.3145728 sec
RG 35.18
DW 76.800 usec
DE 16.00 usec
TE 300.0 K
D0 0.00000300 sec
D1 1.12589896 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00015380 sec
TDav 1
SFO1 500.2330014 MHz
NUC1 1H
P1 12.00 usec
P17 2500.00 usec
PLW1 11.44699955 W
PLW10 1.83150005 W
GPNAM[1] SMSQ10.100
GPZ1 16.00 %
GPNAM[2] SMSQ10.100
GPZ2 12.00 %
GPNAM[3] SMSQ10.100
GPZ3 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.233 MHz
FIDRES 50.796490 Hz
SW 12.998 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 500.2300032 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 2048
MC2 QF
SF 500.2300032 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0

¹H spectrum of **11a**

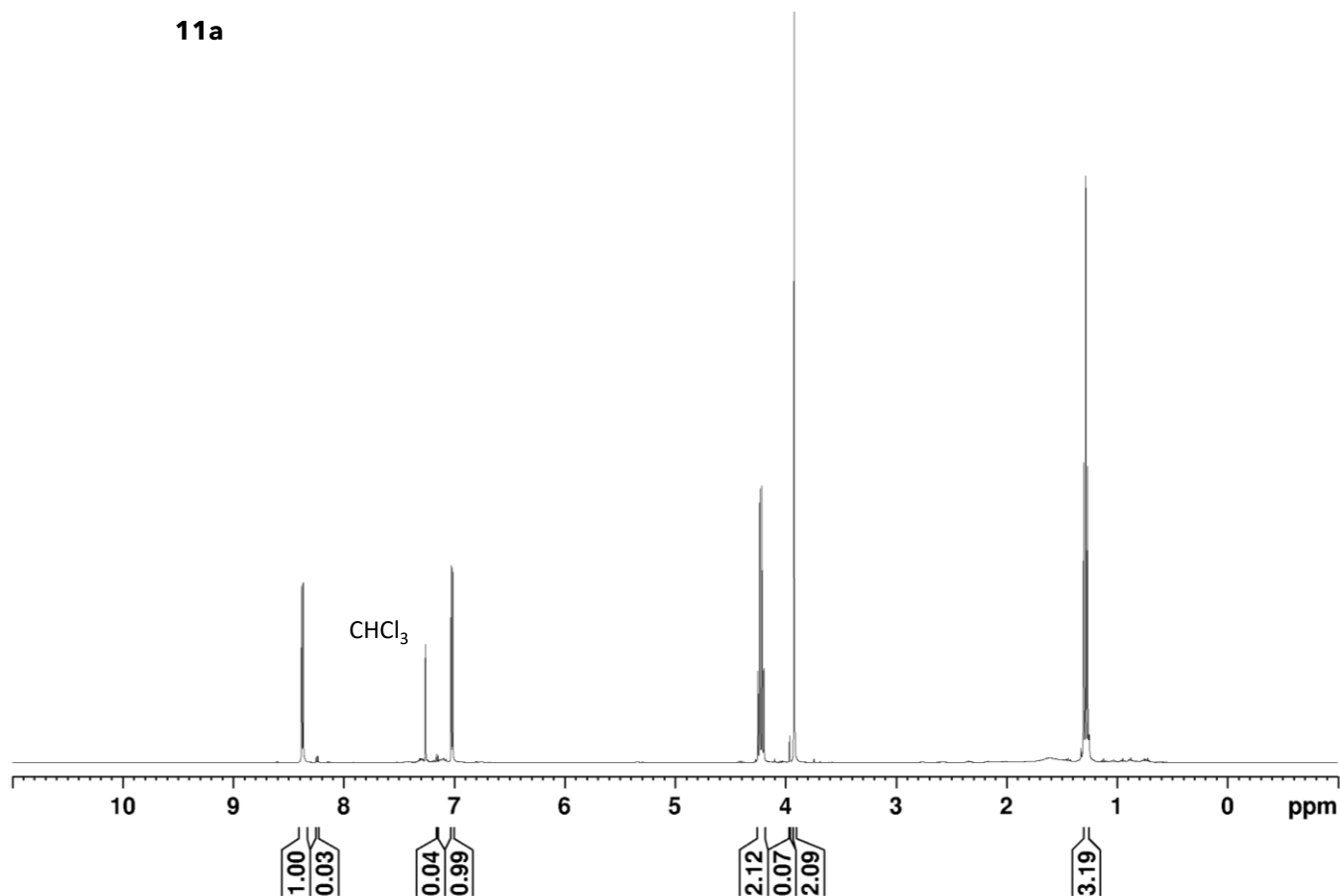


11a

8.381
8.368
8.248
8.234
7.260
7.160
7.146
7.027
7.014

4.250
4.232
4.214
4.196
3.963
3.921

1.324
1.306
1.299
1.282
1.264
1.251



Current Data Parameters

NAME OJ-241014-2-col-1
EXPNO 12
PROCNO 1

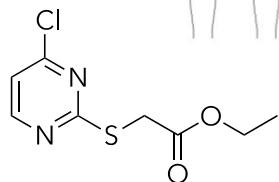
F2 - Acquisition Parameters

Date_ 20241015
Time 21.47 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P...
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **11a**



11a

172.00
168.85
161.31
158.22

117.18

CDCl₃

77.48
77.16
76.84

61.93

33.79

14.30

Current Data Parameters

NAME OJ-241014-2-col-1-C
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters

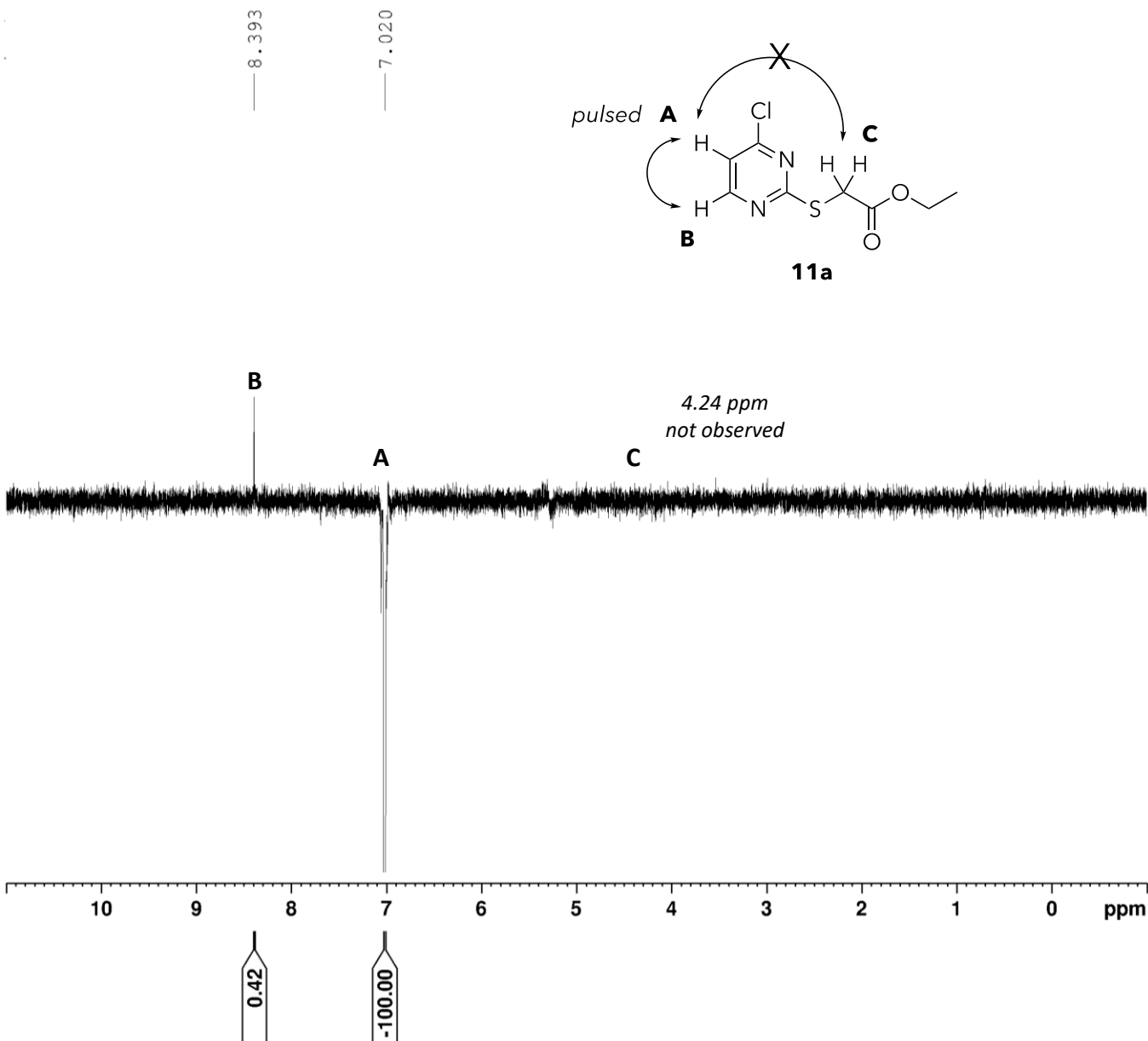
Date_ 20241015
Time 22.53 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P.
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

1D NOE of 11a

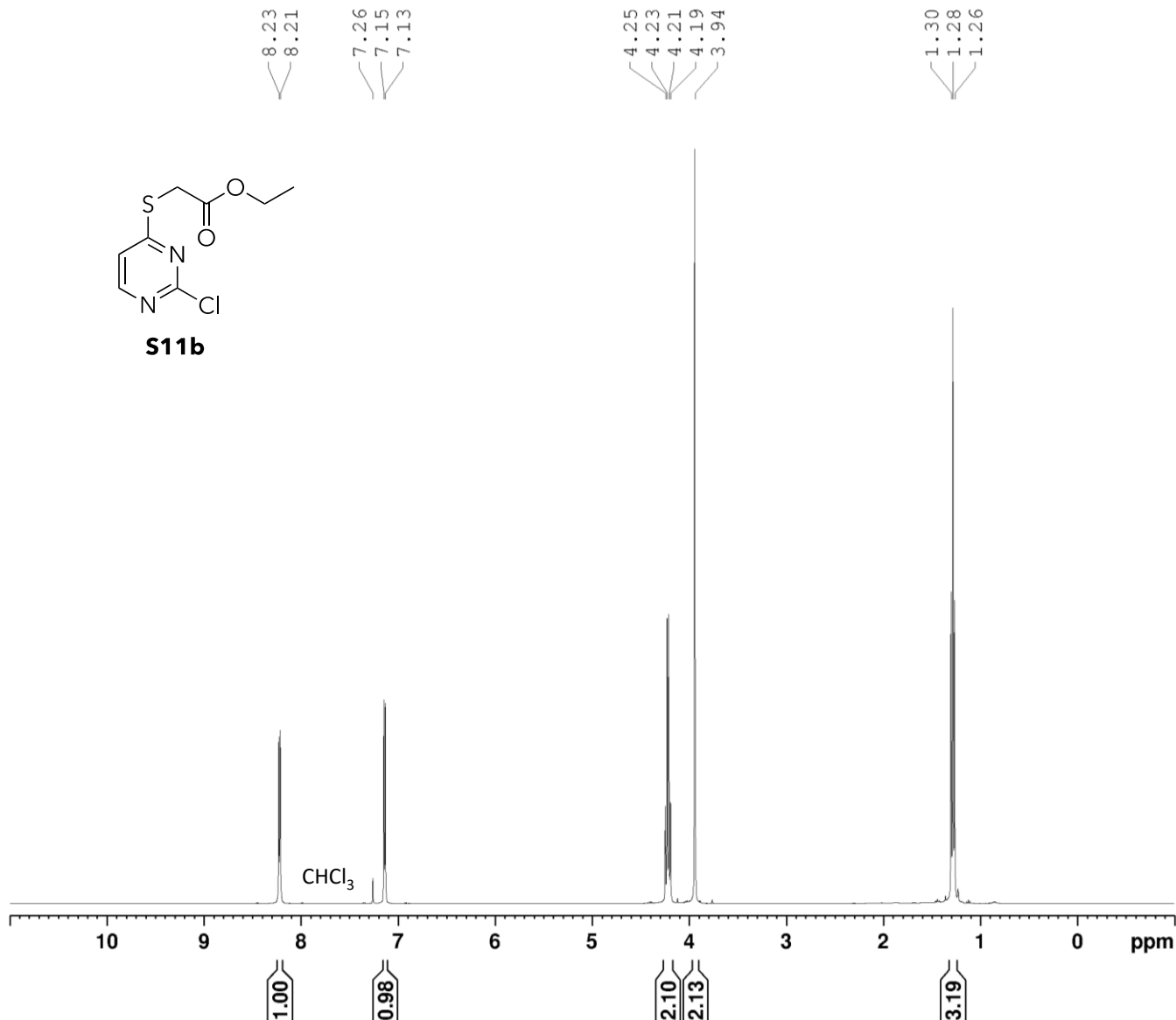
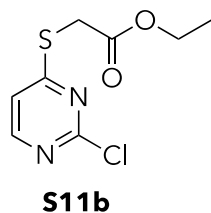


Current Data Parameters
NAME OJ-241014-2-NOE
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241030
Time 14.16 h
INSTRUM spect
PROBHD Z125869_0055 (CPF BBO 500S1 BB-H&F-05)
PULPROG sehnogpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 7.0334611
P1 12.00 usec
P12 47964.46 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 429.43 Hz
SPW2 0.00001692 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300074 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H spectrum of **S11b**

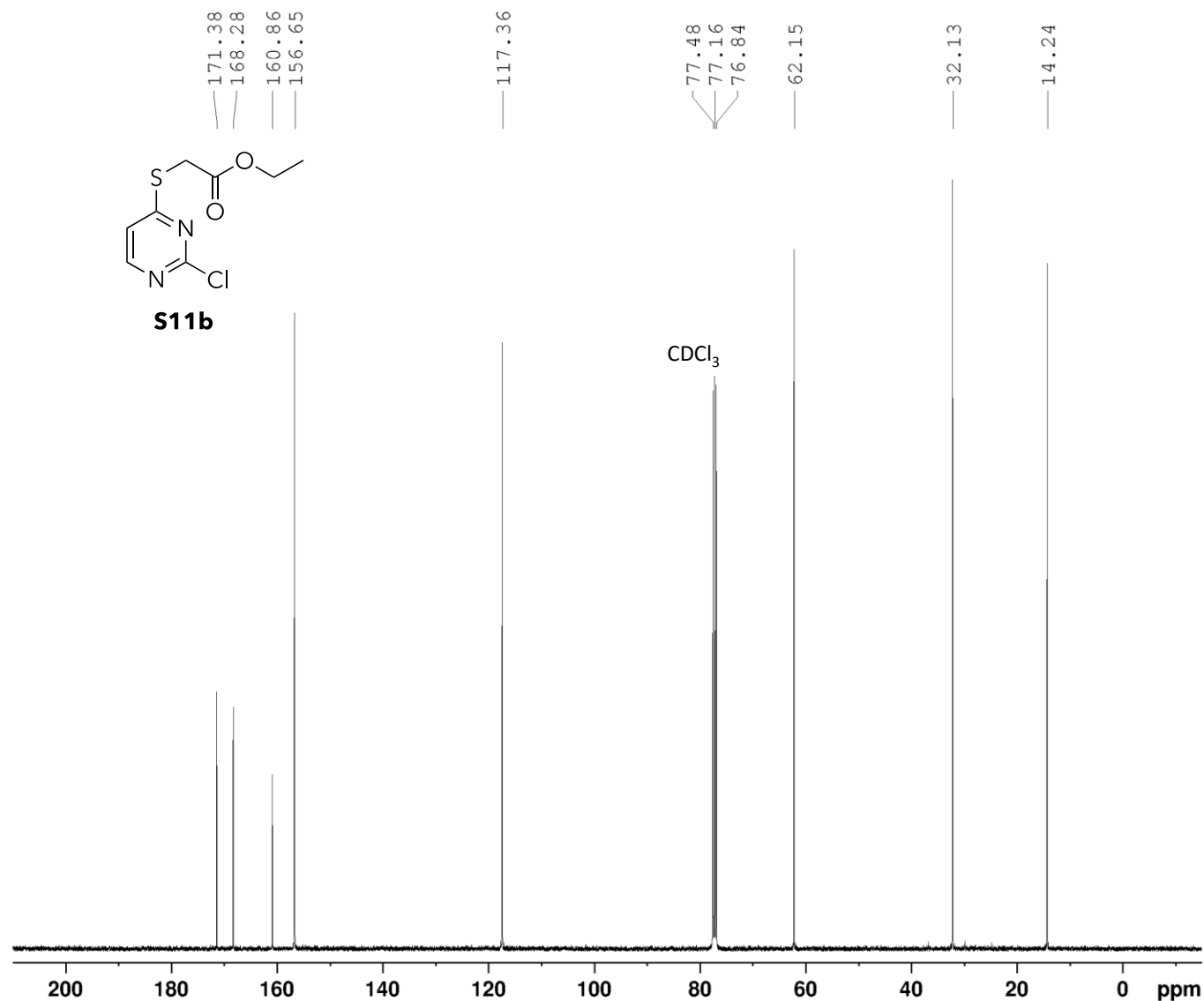
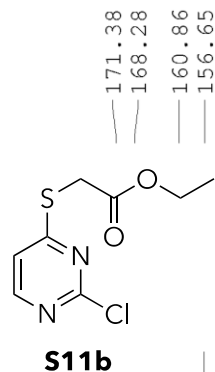


Current Data Parameters
NAME OJ-6-46-H
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241110
Time 18.21 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 64
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **S11b**



Current Data Parameters

NAME OJ-6-46-C
EXPNO 18
PROCNO 1

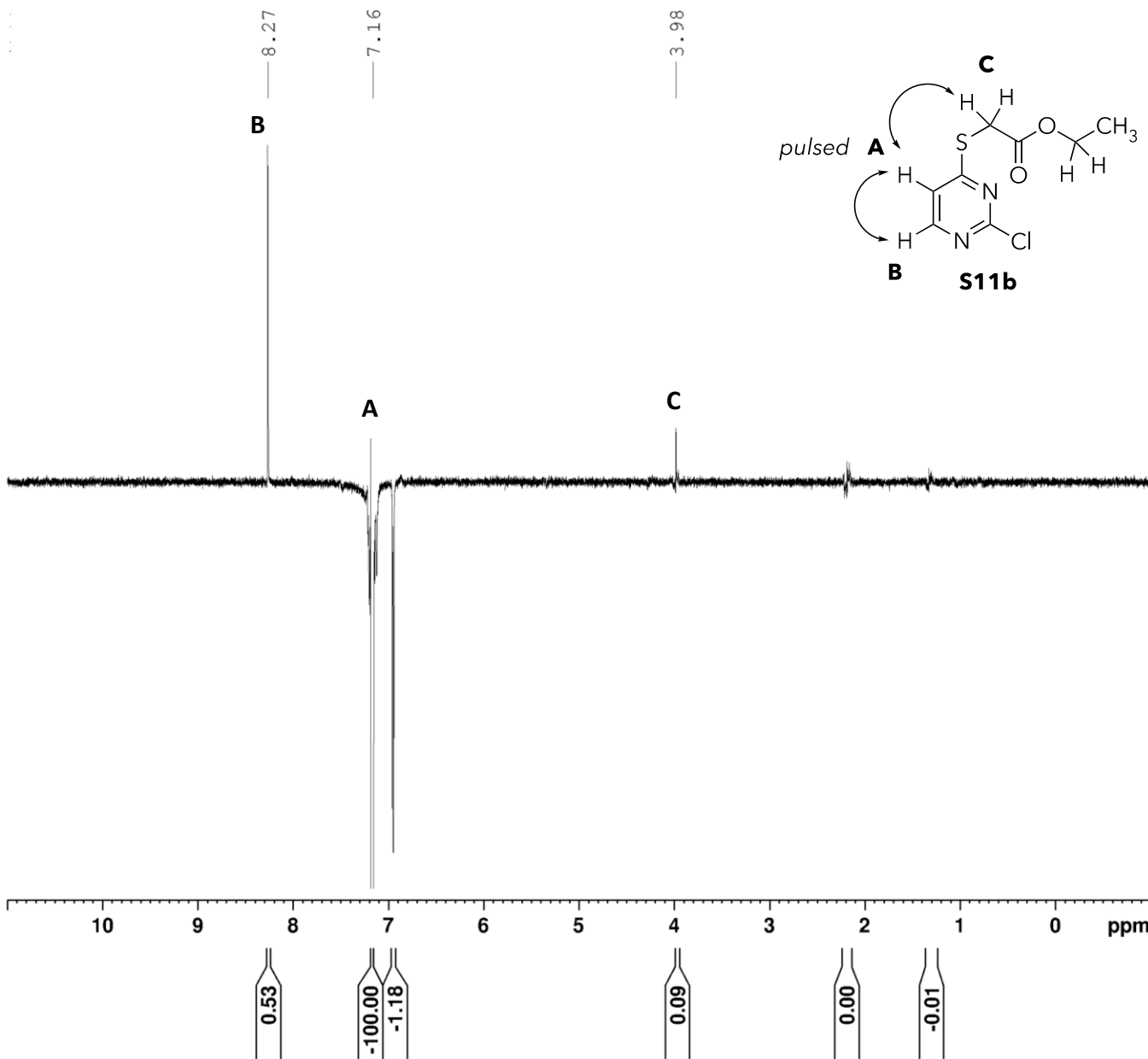
F2 - Acquisition Parameters

Date_ 20241110
Time 21.56 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

SI 32768
SF 100.6127596 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of S11b

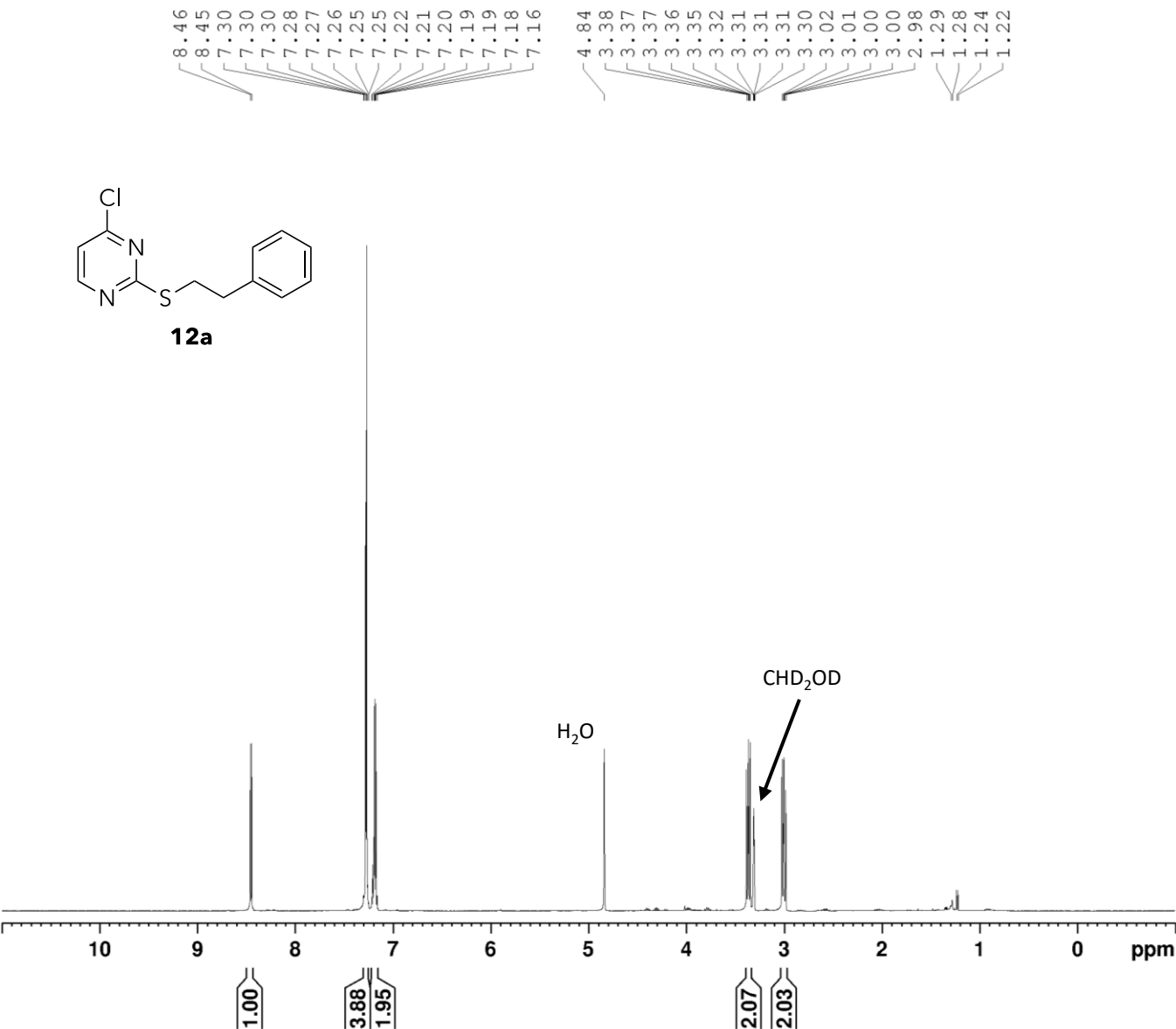


Current Data Parameters
 NAME OJ-6-46-NOE
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231211
 Time 20.47 h
 INSTRUM spect
 PROBHD Z125869_0055 (CPP BBO 500S1 BB-H&F-05 Z)
 PULPROG selnogpzs.2
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767999 sec
 RG 171.72
 DW 50.000 usec
 DE 20.46 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27272600 sec
 D31 0.30000001 sec
 TD0 1
 ZGPTNS -DCALC_SPOFFS
 SFO1 500.2330889 MHz
 NUC1 1H
 CNST21 7.1729078
 P1 12.00 usec
 P12 58810.14 usec
 P32 20000.00 usec
 P44 240.00 usec
 PLW1 11.44699955 W
 SPNAM[2] Gausl_180r.1000
 SPOAL2 0.500
 spoffs2 499.18 Hz
 SPW2 0.00001125 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 spoffs29 0 Hz
 SPW29 0.06296300 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 spoffs30 0 Hz
 SPW30 1.83150005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 500.2300000 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

¹H spectrum of **12a**



Current Data Parameters

NAME OJ-5-27-2-MeOH
EXPNO 13
PROCNO 1

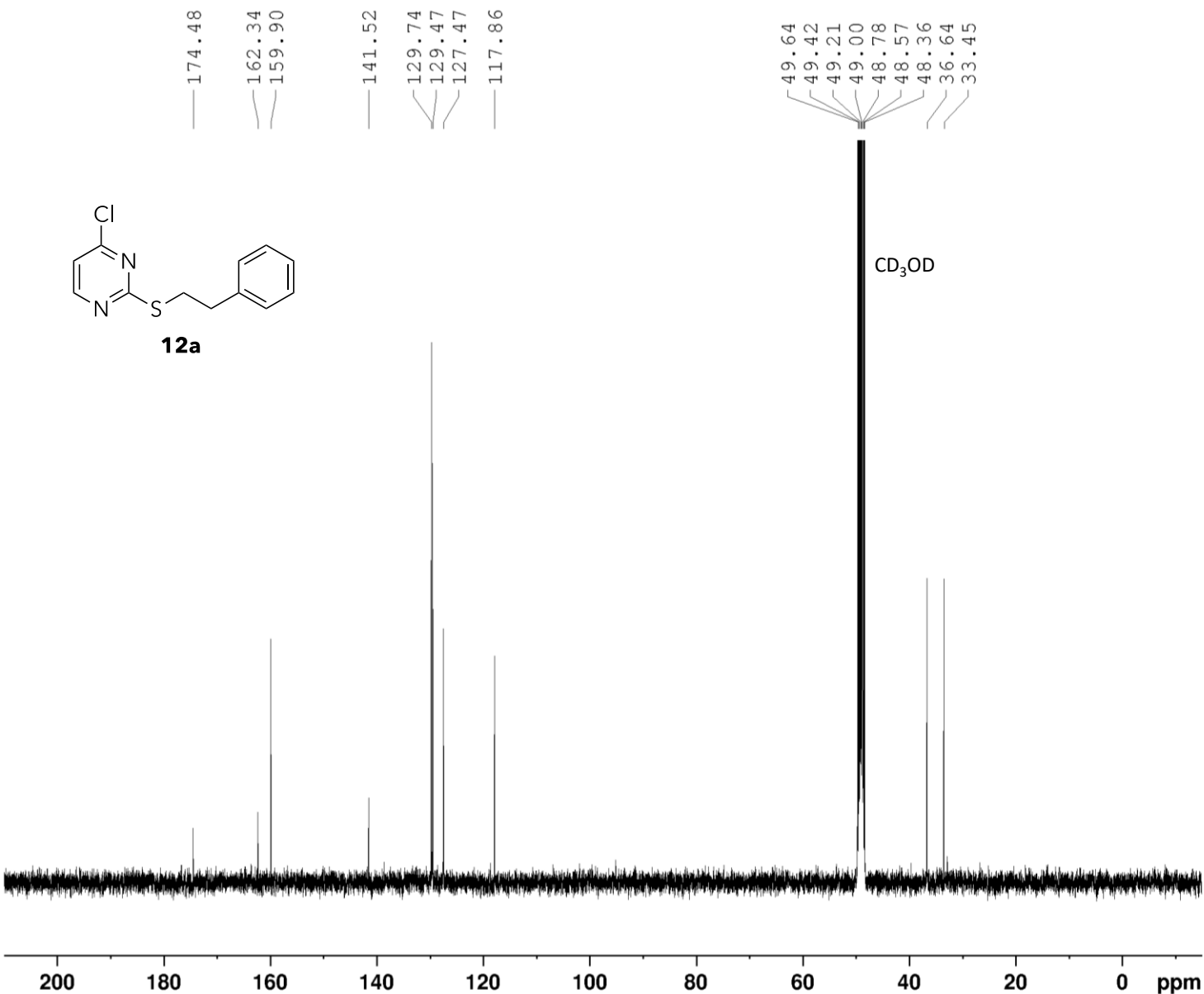
F2 - Acquisition Parameters

Date_ 20240607
Time 10.52 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **12a**



Current Data Parameters

NAME	OJ-5-27-C-MeOH
EXPNO	15
PROCNO	1

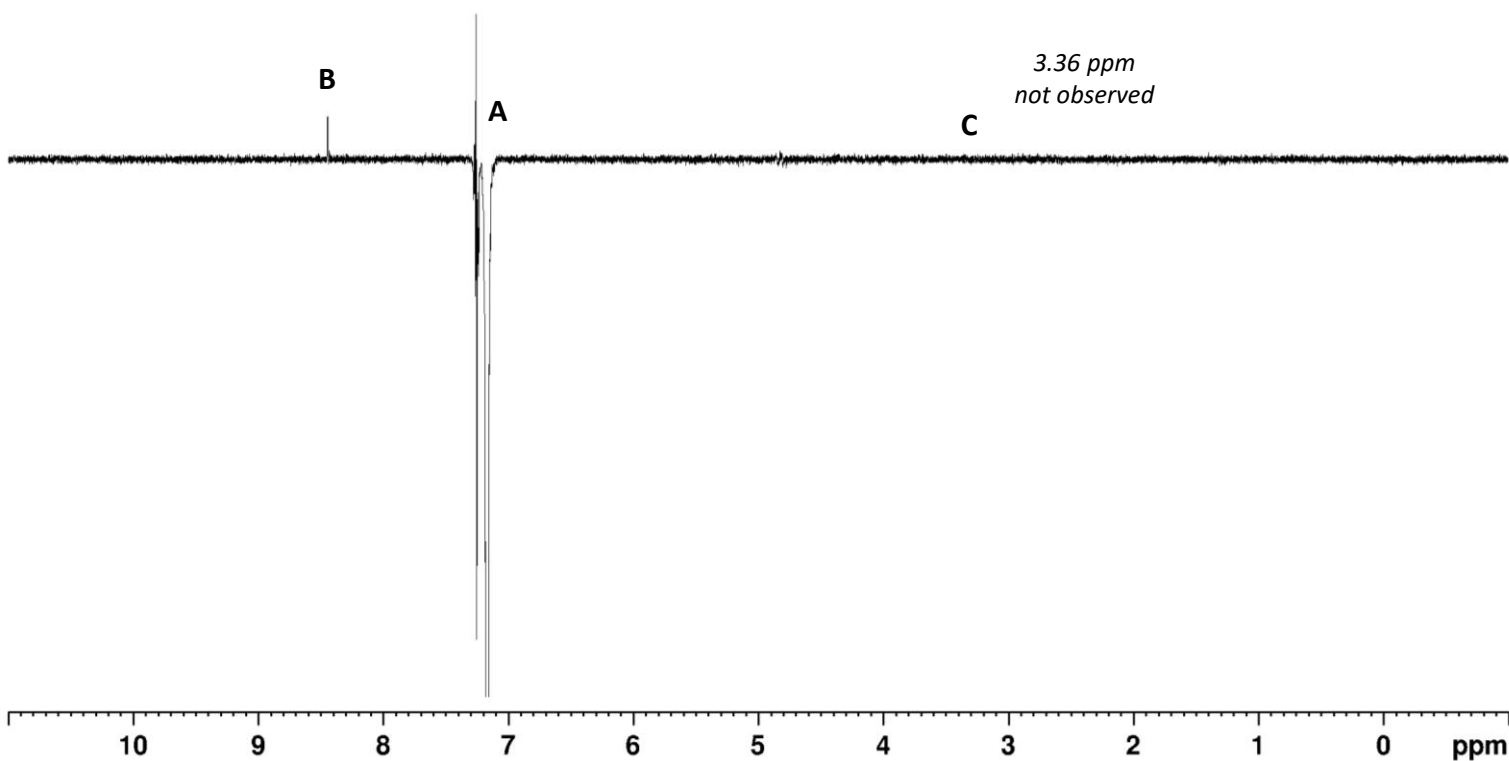
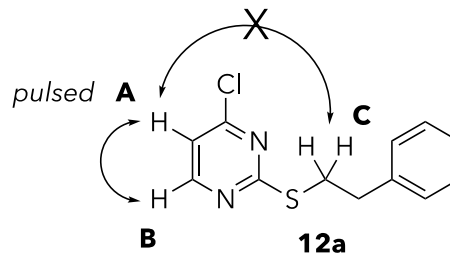
F2 - Acquisition Parameters

Date_	20240610
Time	20.03 h
INSTRUM	Avance Neo
PROBHD	Z181819_0001 (
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	1024
DS	4
SWH	23809.524 Hz
FIDRES	0.726609 Hz
AQ	1.3762560 sec
RG	11.3
DW	21.000 usec
DE	6.50 usec
TE	300.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
PLW1	56.9169982 W
SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2	waltz65
PCPD2	90.00 usec
PLW2	22.53199959 W
PLW12	0.27818000 W
PLW13	0.13992000 W

F2 - Processing parameters

SI	32768
SF	100.6126278 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NOE of **12a**

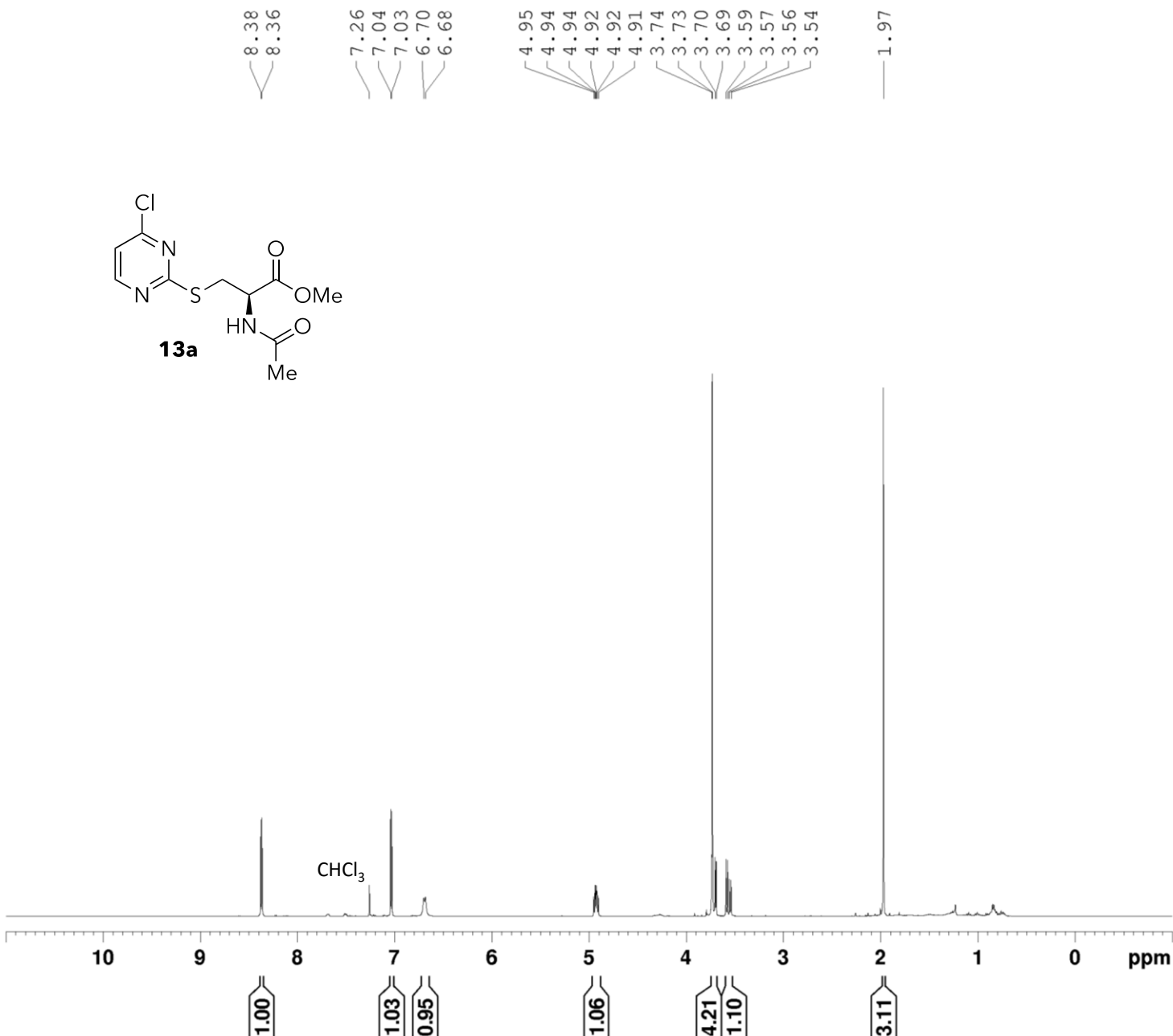
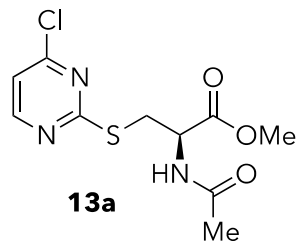


Current Data Parameters
NAME OJ-5-27-MeOH-NOE
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240610
Time 12.18 h
INSTRUM spect
PROBHD Z125869_0055 {
PULPROG selnogpzs.2
TD 65536
SOLVENT MeOD
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.2156410
P1 12.00 usec
P12 88925.23 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPOAL2 0.500
spoffs2 520.56 Hz
SPW2 0.00000492 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300224 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H spectrum of **13a**

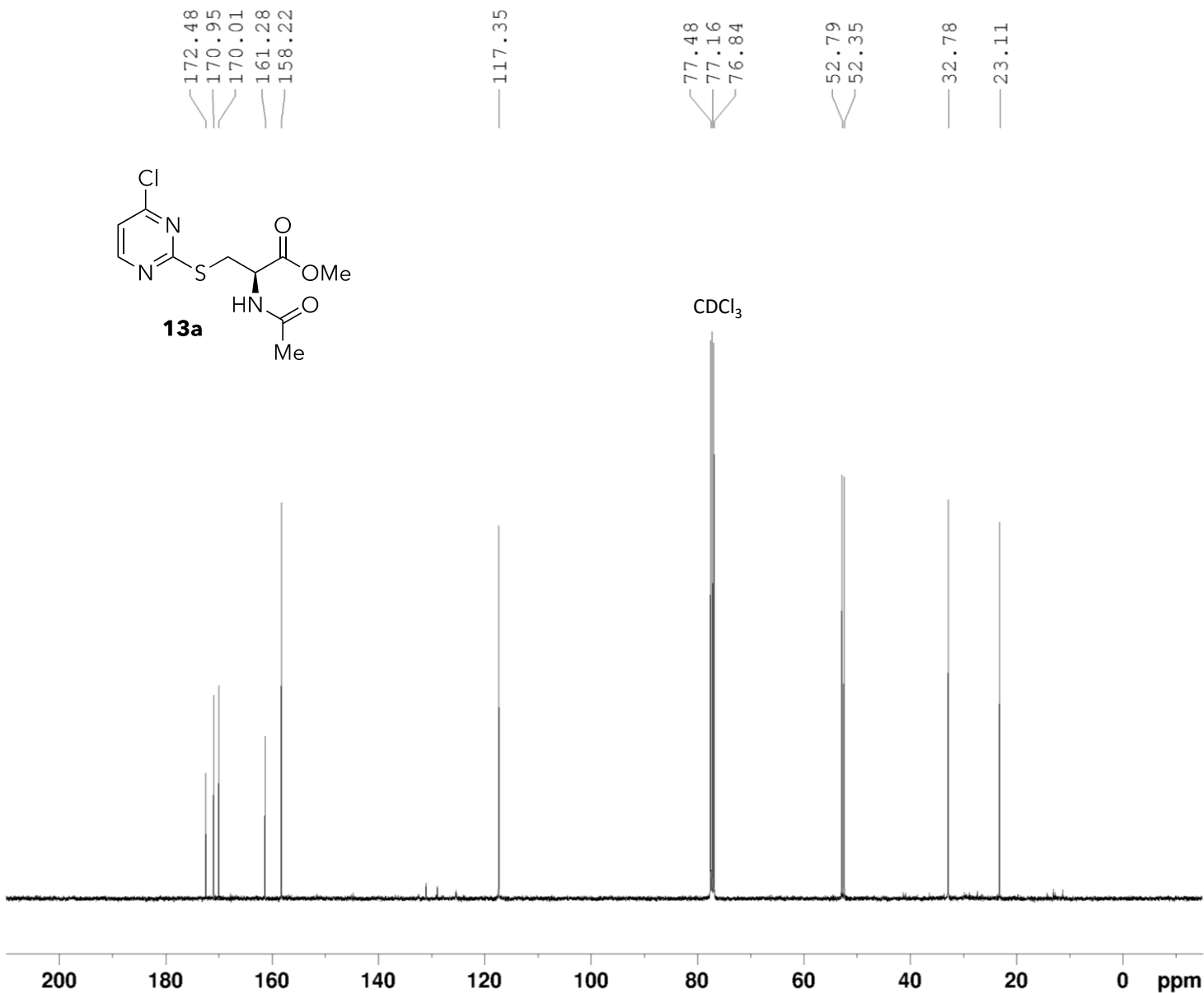
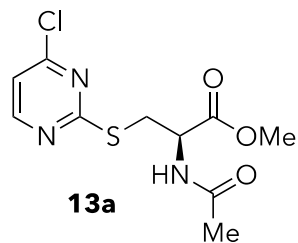


Current Data Parameters
NAME OJ-6-73-2
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231128
Time 20.41 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 300.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **13a**



Current Data Parameters
NAME OJ-6-73-2-C
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231128
Time 23.49 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

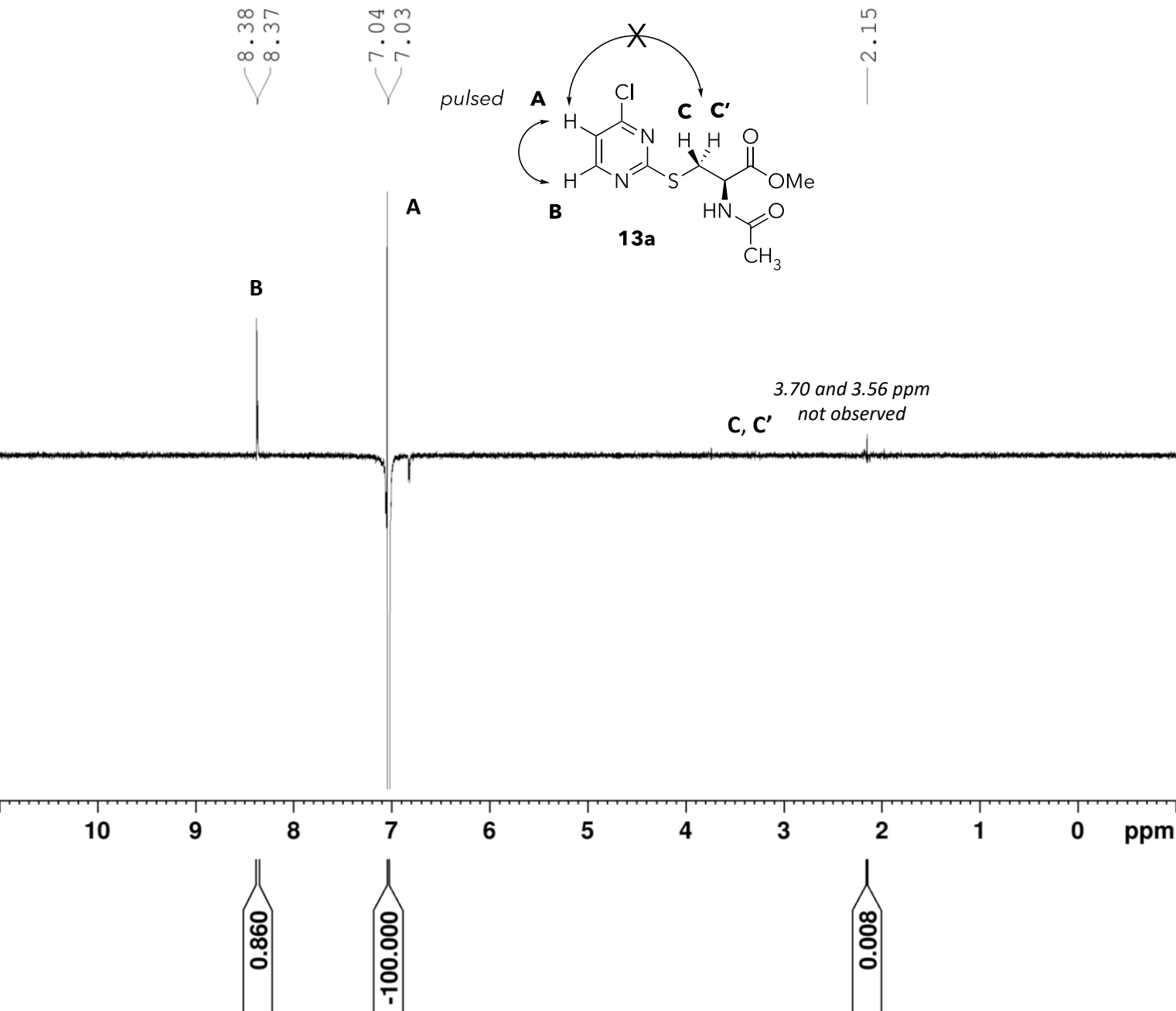
F2 - Processing parameters
SI 32768
SF 100.6127591 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 13a

Current Data Parameters
 NAME OJ-6-73-NOE
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231211
 Time 21.16 h
 INSTRUM spect
 PROBHD Z125869_0055 ()
 PULPROG selnpgps.2
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767999 sec
 RG 171.72
 DW 50.000 usec
 DE 20.46 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27272600 sec
 D31 0.30000001 sec
 TD0 1
 ZGPGTNS -DCALC_SPOFFS
 SF01 500.2330889 MHz
 NUC1 1H
 CNST21 7.0715809
 P1 12.00 usec
 P12 50755.42 usec
 P32 20000.00 usec
 P44 240.00 usec
 PLW1 11.44699955 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 spoffs2 448.50 Hz
 SPW2 0.00001511 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 spoffs29 0 Hz
 SPW29 0.06296300 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 spoffs30 0 Hz
 SPW30 1.83150005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 500.2300193 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



COSY of 13a

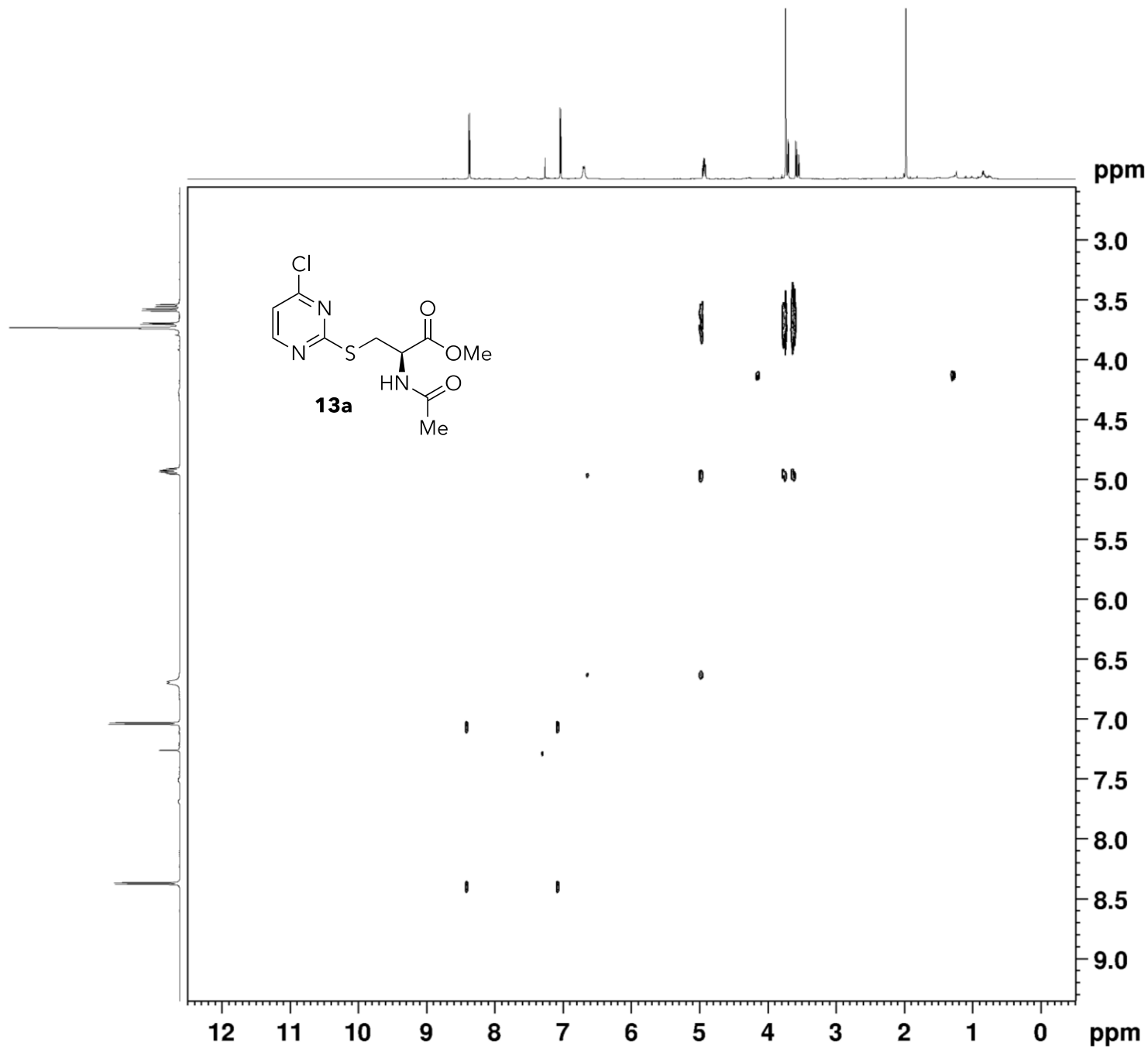
Current Data Parameters
NAME OJ-6-73-COSY
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240911
Time 13.41 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG cosygpmfppqf
TD 4096
SOLVENT CDCl3
NS 4
DS 16
SWH 6510.417 Hz
FIDRES 3.178914 Hz
AQ 0.3145728 sec
RG 69.97
DW 76.800 usec
DE 16.00 usec
TE 300.0 K
D0 0.00000300 sec
D1 1.12589896 sec
D11 0.03000000 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00015380 sec
TDav 1
SFO1 500.2330014 MHz
NUC1 1H
P1 12.00 usec
P17 2500.00 usec
PLW1 11.44699955 W
PLW10 1.83150005 W
GPNAM[1] SMSQ10.100
GPZ1 16.00 %
GPNAM[2] SMSQ10.100
GPZ2 12.00 %
GPNAM[3] SMSQ10.100
GPZ3 40.00 %
P16 1000.00 usec

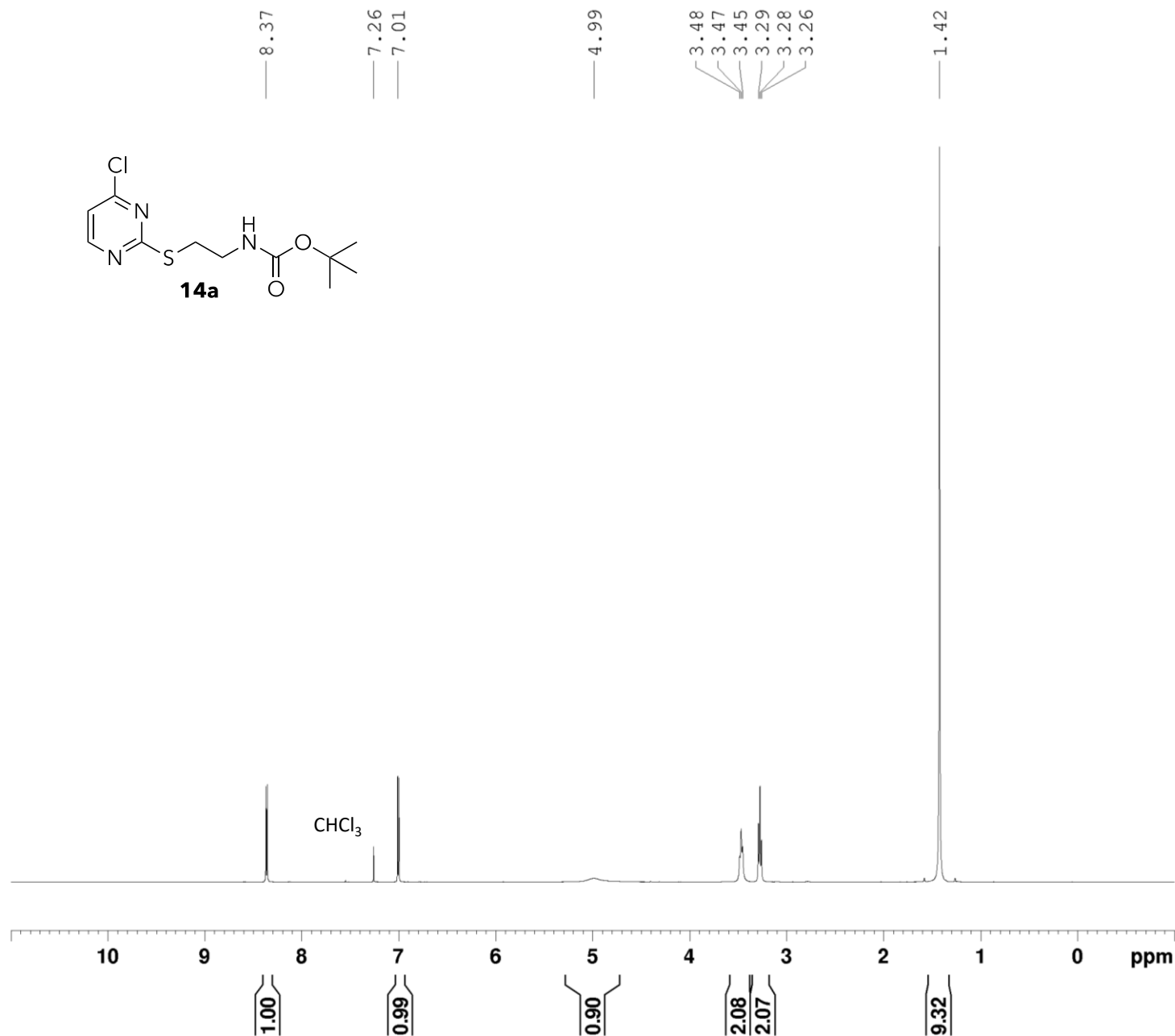
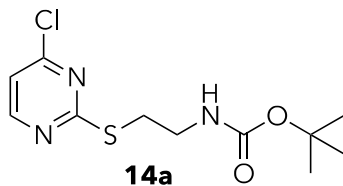
F1 - Acquisition parameters
TD 256
SFO1 500.233 MHz
FIDRES 50.796490 Hz
SW 12.998 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 500.2300032 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 2048
MC2 QF
SF 500.2300032 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0



¹H spectrum of **14a**

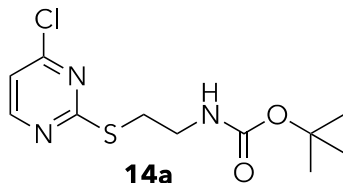


Current Data Parameters
NAME OJ-6-30-H-2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231228
Time 22.40 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **14a**



173.01
161.29
158.19
155.90
116.96
79.59
77.47
77.16
76.84
39.97
31.49
28.50

CDCl₃

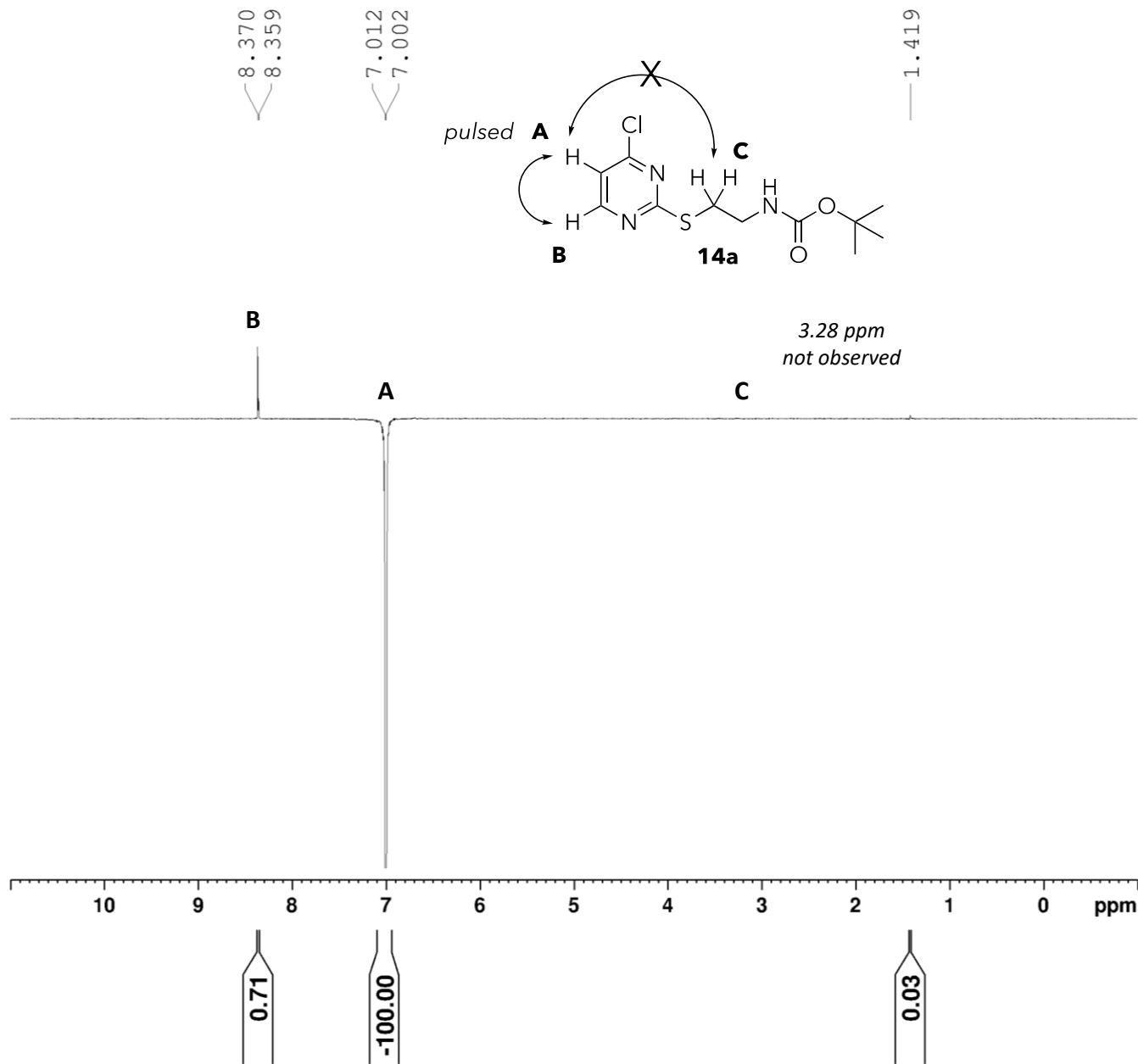
Current Data Parameters
NAME OJ-6-30-C-2
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231229
Time 0.40 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127565 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 14a



Current Data Parameters

NAME	OJ-6-30-NOE
EXPNO	21
PROCNO	1

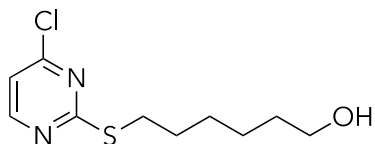
F2 - Acquisition Parameters

Date_	20231211
Time	18.38 h
INSTRUM	spect
PROBHD	Z125869_0055 (CFF BBO 500S1 BB-H
PULPROG	selnpgpzs.2
TD	65536
SOLVENT	CDC13
NS	32
DS	4
SWH	10000.000 Hz
FIDRES	0.305176 Hz
AQ	3.2767999 sec
RG	190.44
DW	50.000 usec
DE	20.46 usec
TE	300.0 K
D1	2.00000000 sec
D8	0.30000001 sec
D16	0.00020000 sec
D20	0.27272600 sec
D31	0.30000001 sec
TD0	1
ZGPTNS	-DCALC_SPOFFS
SFO1	500.2330889 MHz
NUC1	1H
CNST21	7.0198951
P1	12.00 usec
P12	50046.19 usec
P32	20000.00 usec
P44	240.00 usec
PLW1	11.44699955 W
SPNAM[2]	Gaus1_180r.1000
SPOAL2	0.500
spoffs2	422.64 Hz
SPW2	0.00001554 W
SPNAM[29]	Crp60,20,20.10
SPOAL29	0.500
spoffs29	0 Hz
SPW29	0.06296300 W
SPNAM[30]	Bip720,50,20.1
SPOAL30	0.500
spoffs30	0 Hz
SPW30	1.83150005 W
GPZ0	11.00 %
GPNAM[1]	SMSQ10.100
GPZ1	15.00 %
GPNAM[2]	SMSQ10.100
GPZ2	40.00 %
P16	1000.00 usec

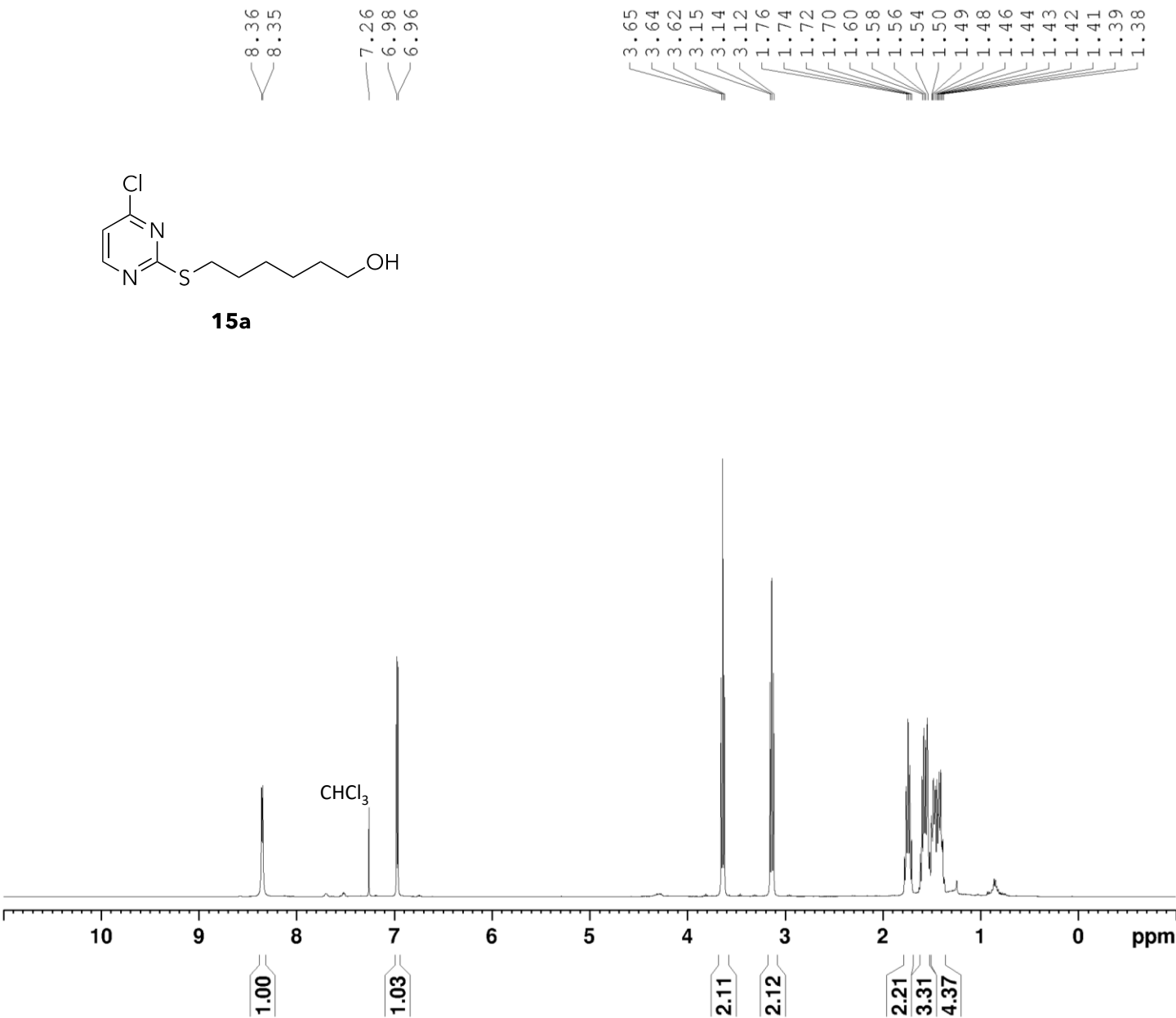
F2 - Processing parameters

SI	32768
SF	500.2300086 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

¹H spectrum of **15a**



15a

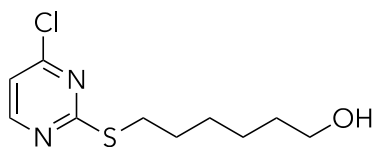


Current Data Parameters
NAME OJ-6-32-H
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231221
Time 18.40 h
INSTRUM Avance Neo
PROBHD z181819_0001 (zg30)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 15a



15a

CDCl₃

173.87

161.25

157.95

116.54

77.47

77.16

76.84

63.01

32.72

31.18

28.97

28.69

25.40

200 180 160 140 120 100 80 60 40 20 0 ppm

Current Data Parameters

NAME OJ-6-32-C
EXPNO 14
PROCNO 1

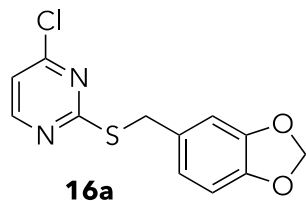
F2 - Acquisition Parameters

Date_ 20240525
Time 21.29 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P...
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H spectrum of **16a**



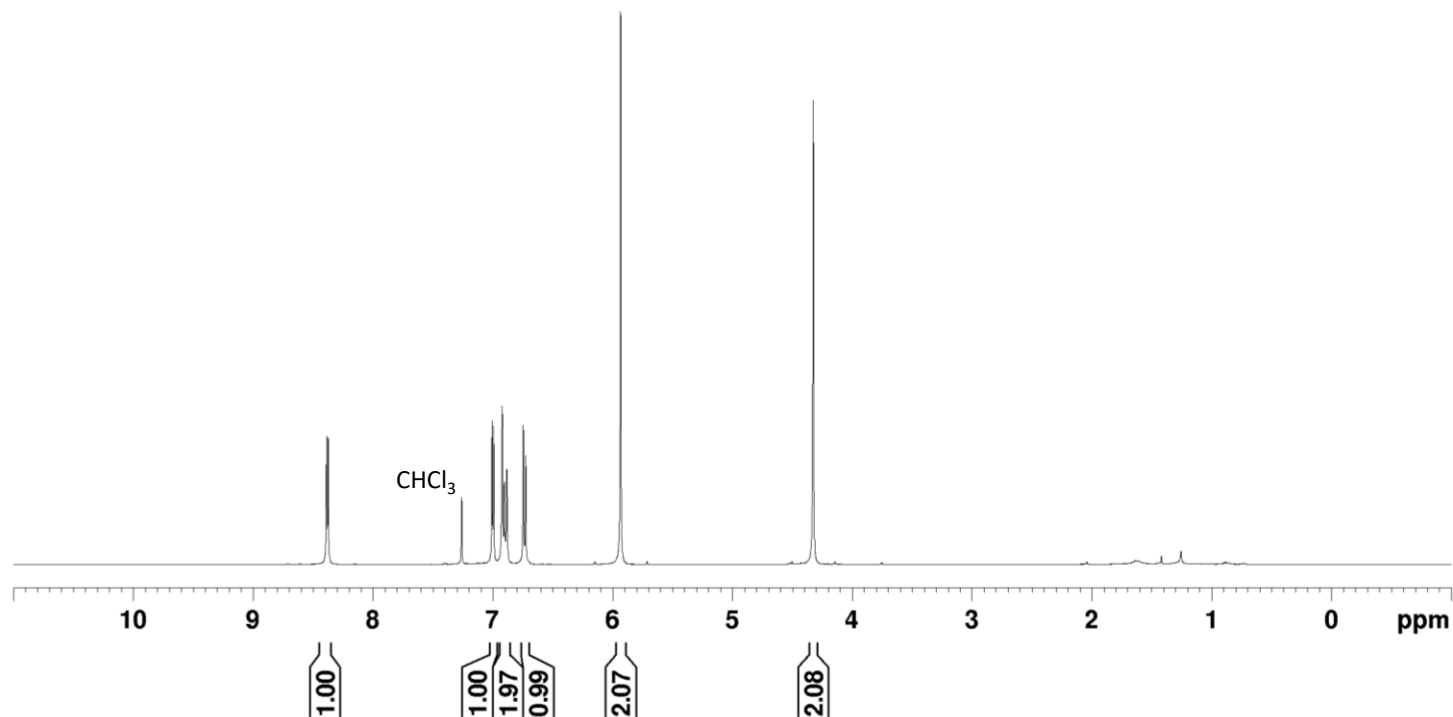
8.39
8.37
7.26
7.01
6.99
6.92
6.92
6.90
6.88
6.75
6.73
5.93

4.33

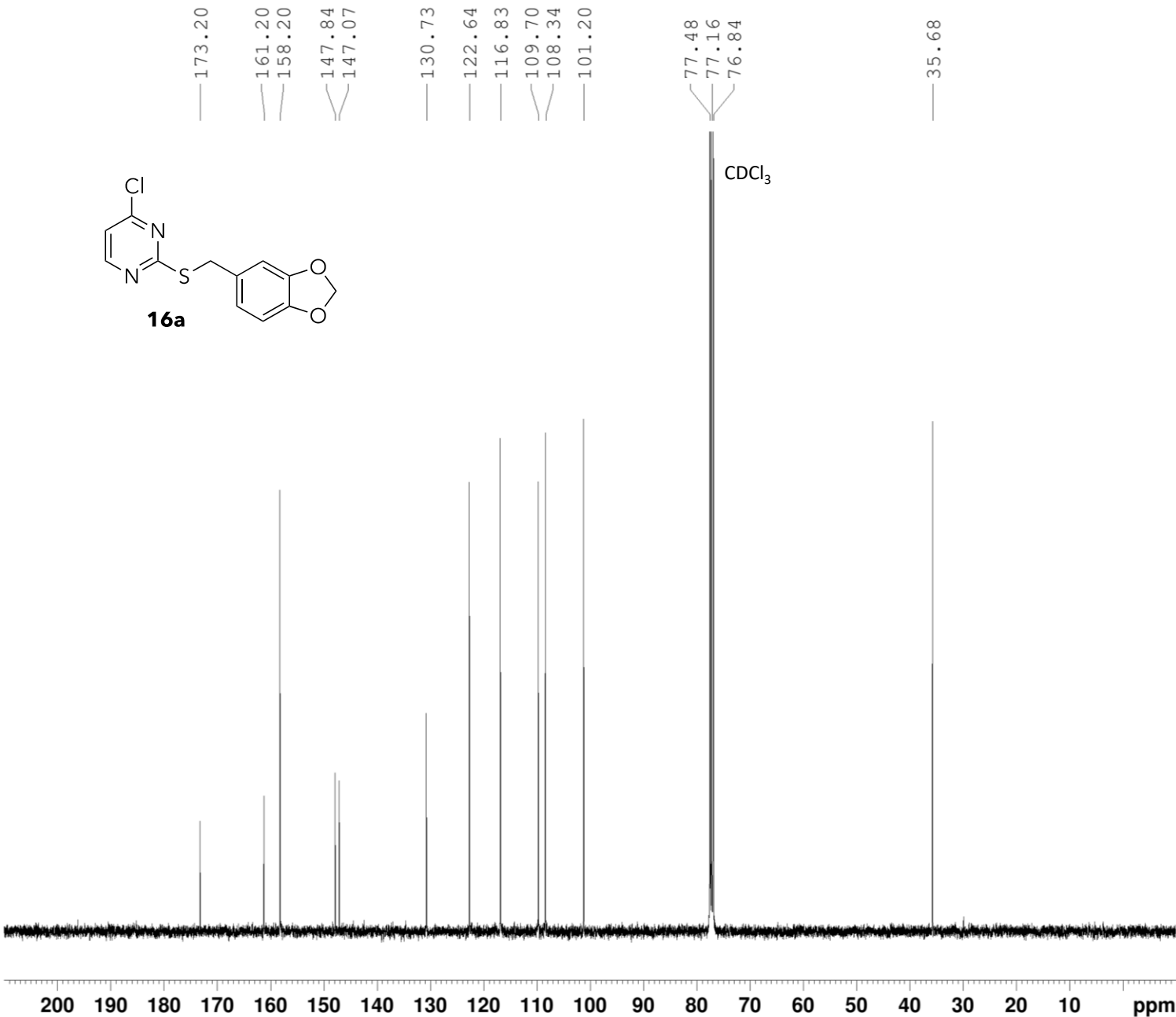
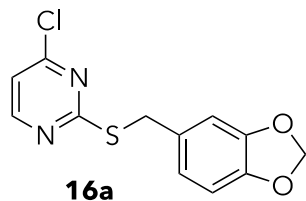
Current Data Parameters
NAME OJ-6-89-H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231228
Time 19.23 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C spectrum of 16a



Current Data Parameters
NAME OJ-6-89-C
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231228
Time 21.24 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

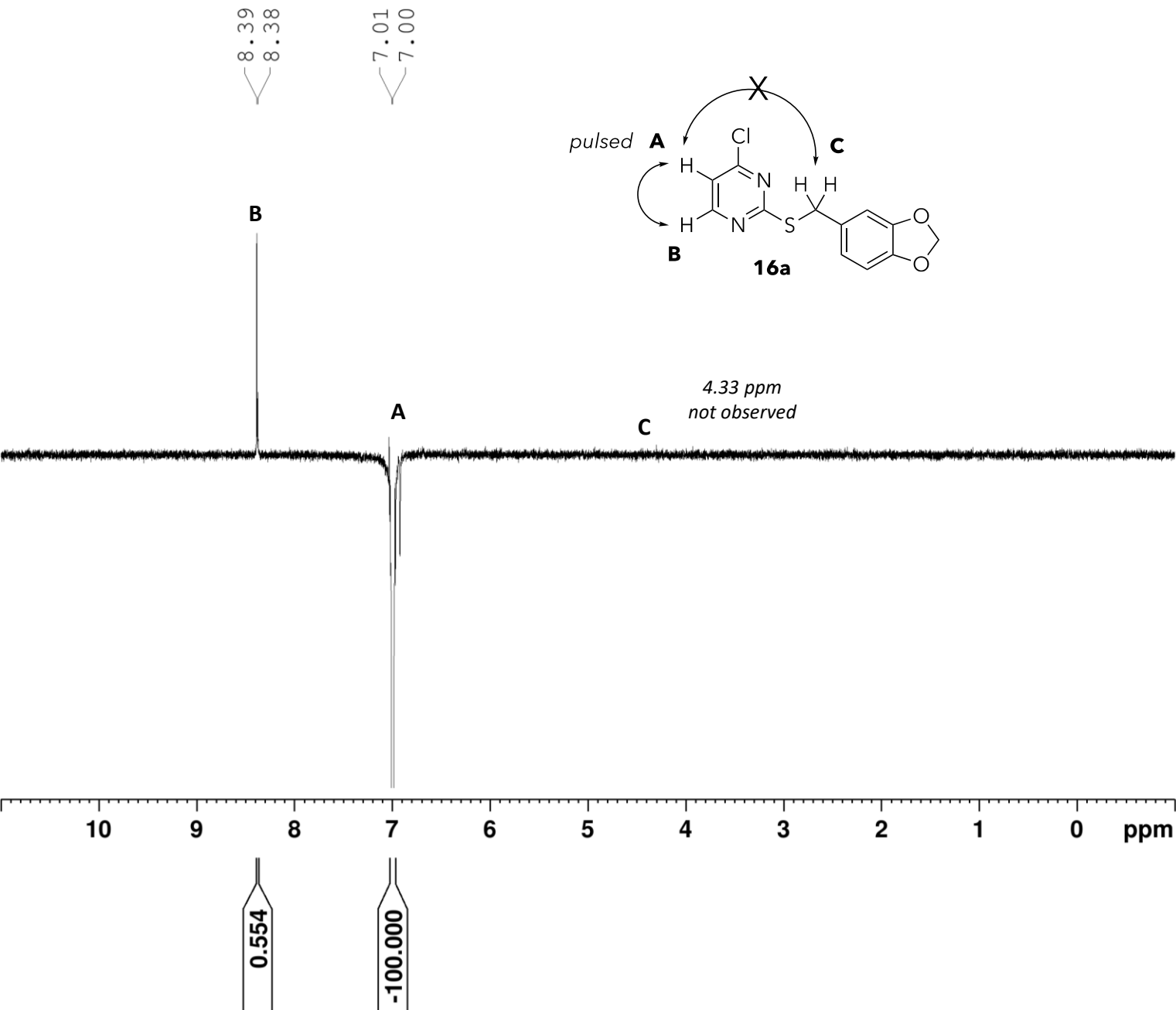
F2 - Processing parameters
SI 32768
SF 100.6127552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 16a

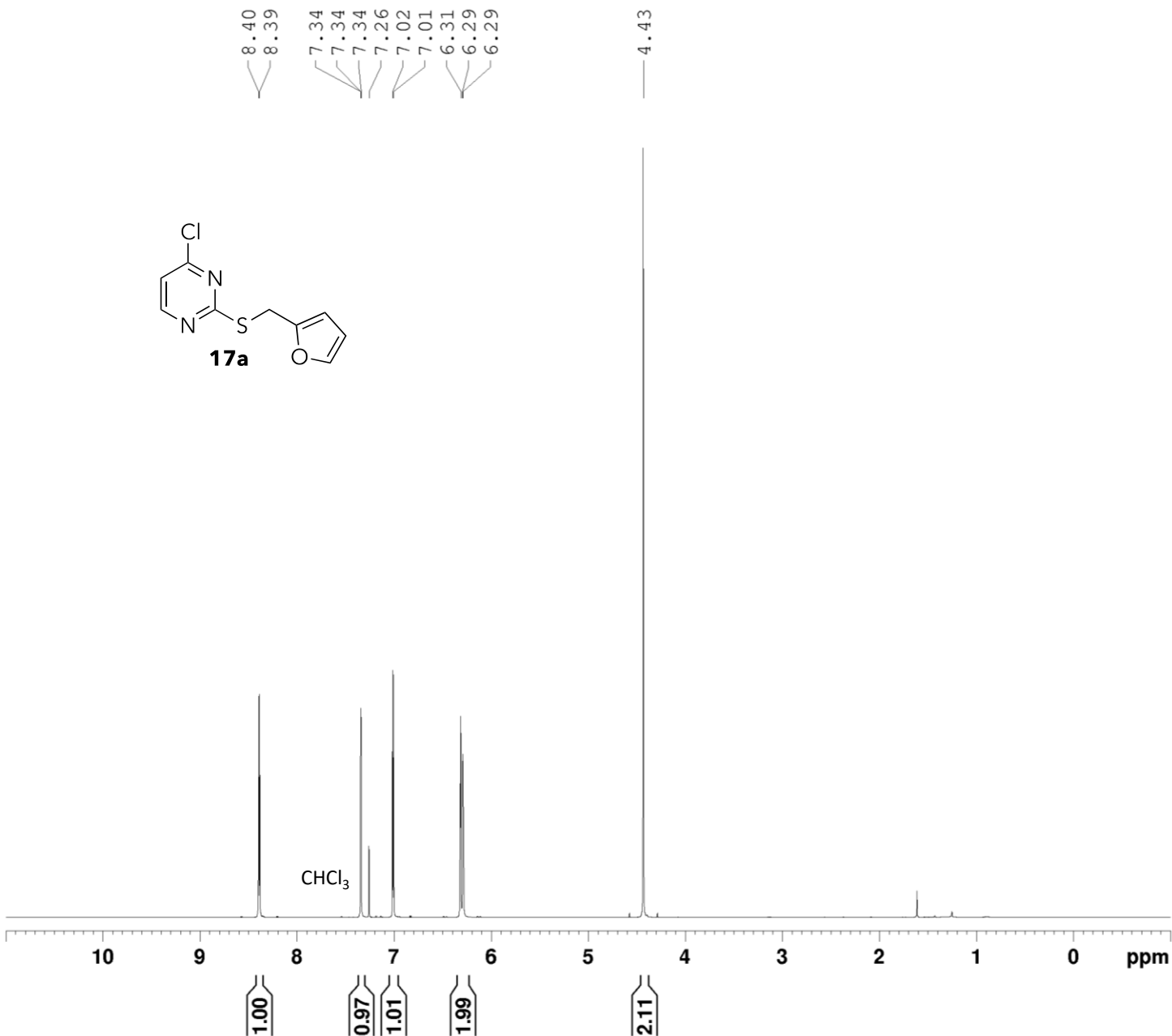
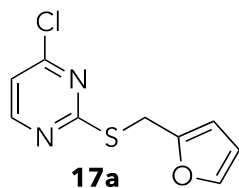
Current Data Parameters
NAME OJ-6-89-NOE
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240107
Time 16.09 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0257978
P1 12.00 usec
P12 52091.11 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 425.59 Hz
SPW2 0.00001434 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300145 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **17a**

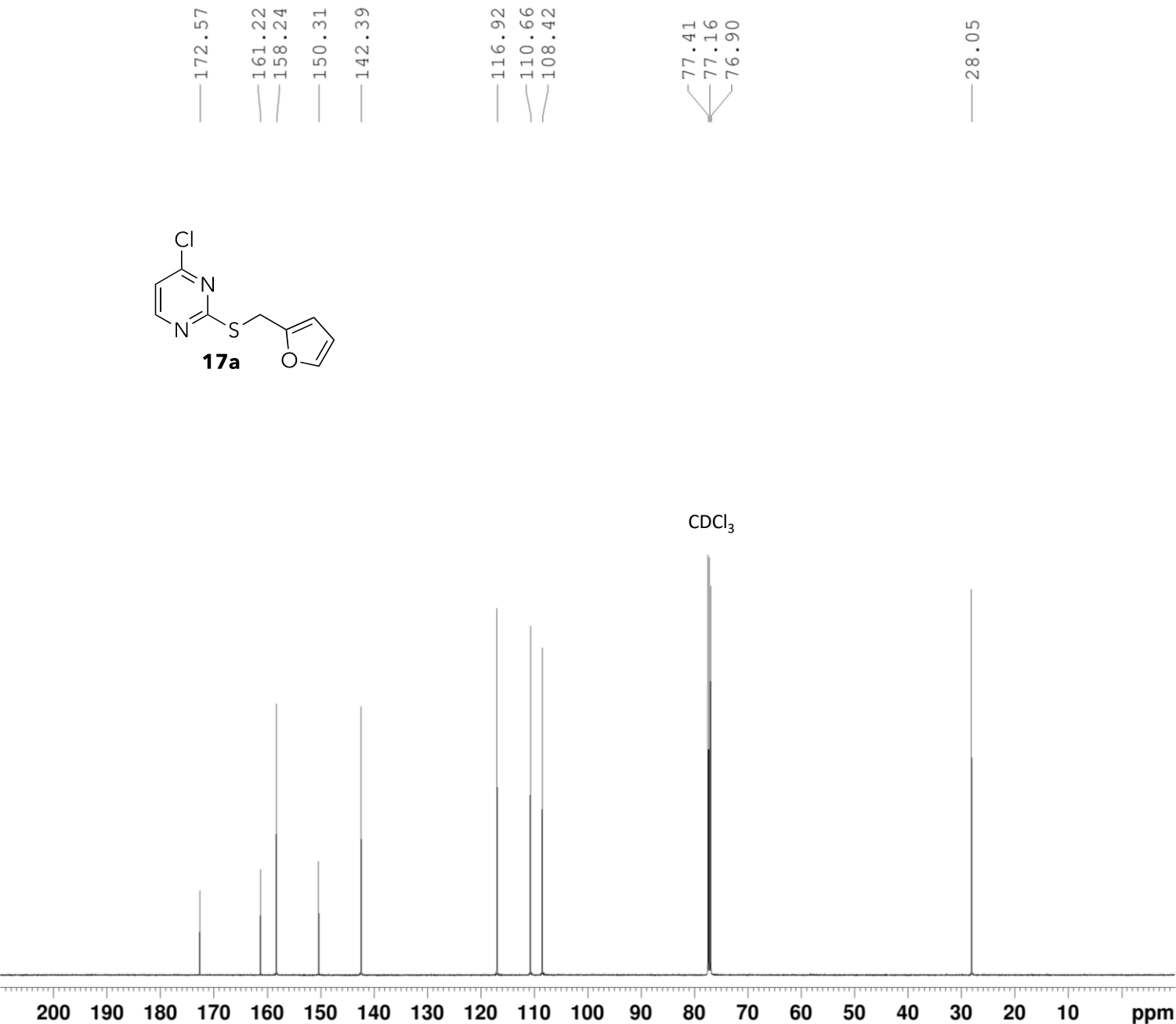
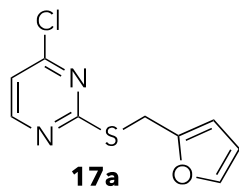


Current Data Parameters
NAME OJ-4-77-NOE
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230506
Time 15.27 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **17a**

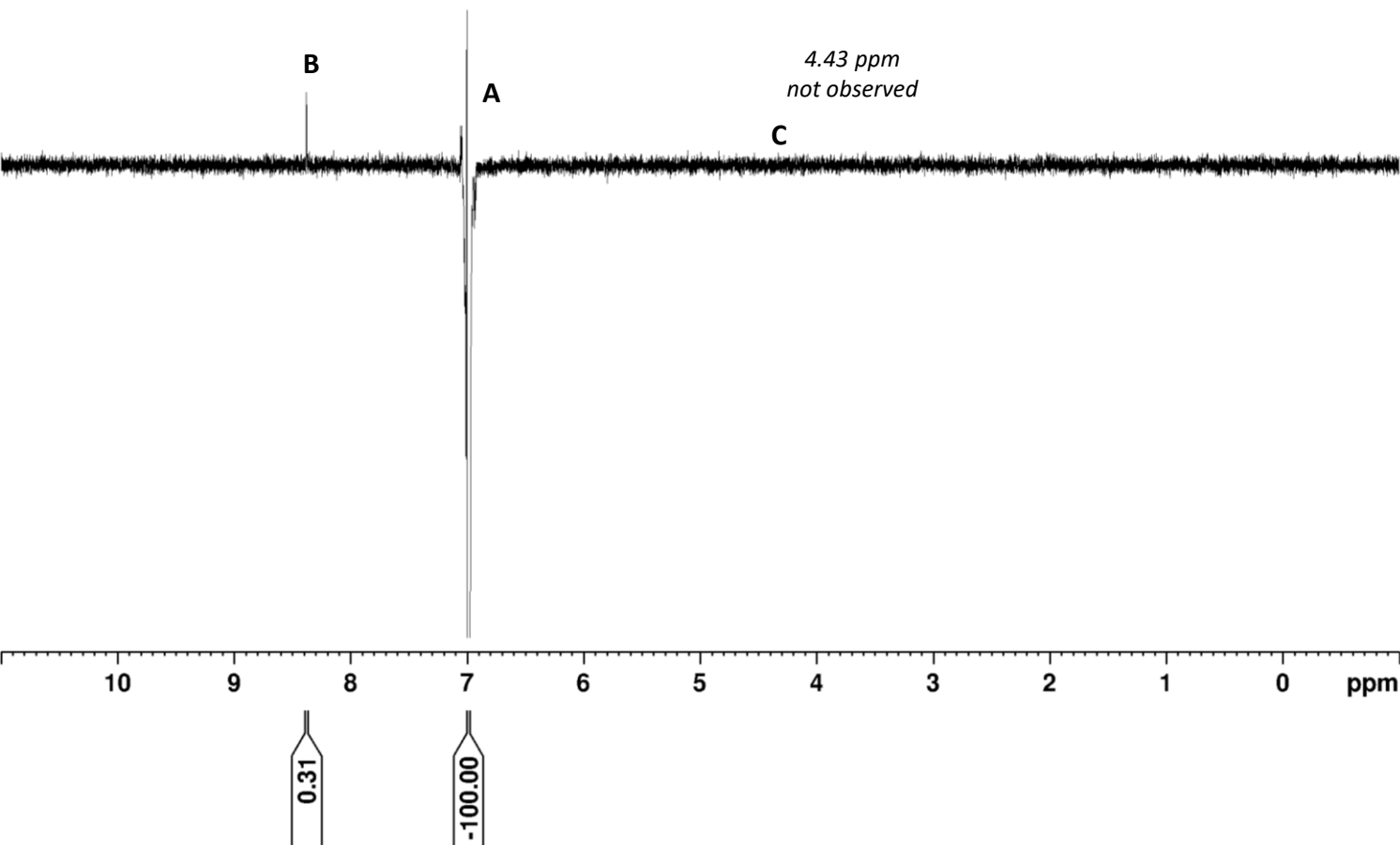
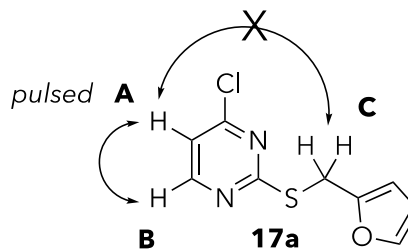


Current Data Parameters
NAME OJ-4-77-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230425
Time 23.04 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters
SI 32768
SF 125.7829207 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **17a**

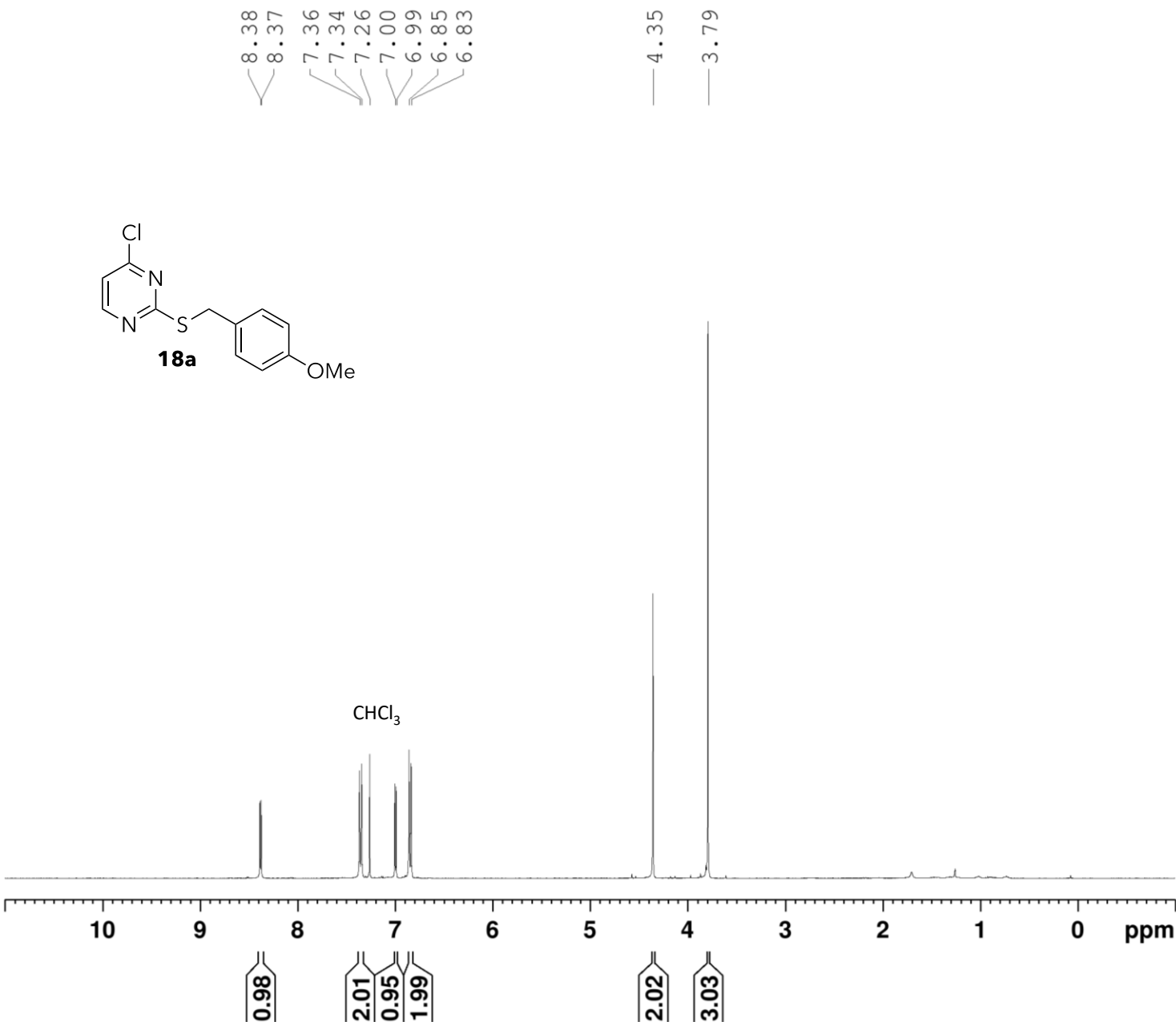


Current Data Parameters
NAME OJ-4-77-NOE-new
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240912
Time 15.20 h
INSTRUM spect
PROBHD Z125869_0055 {
PULPROG selnogpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 171.72
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0406399
P1 12.00 usec
P12 66543.27 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 433.02 Hz
SPW2 0.00000879 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300250 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 465 1.00

¹H spectrum of **18a**

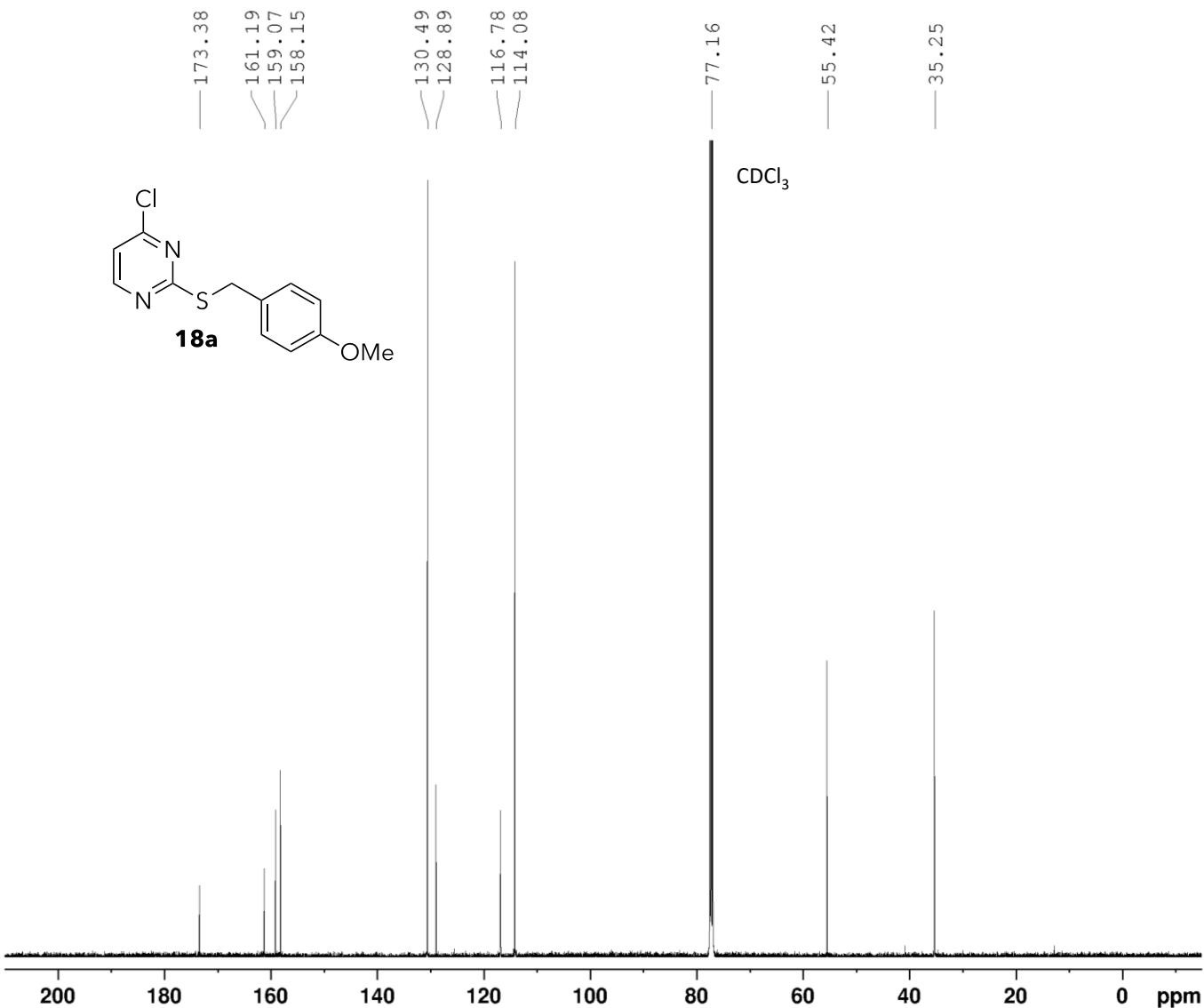


Current Data Parameters
NAME 18a_AR_02_183
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240520
Time 12.01 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SF01 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 18a



Current Data Parameters
NAME 18a_AR_02_183
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240123
Time 19.26 h
INSTRUM spect
PROBHD Z125869_0055 (CFP BBO 500S1 BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

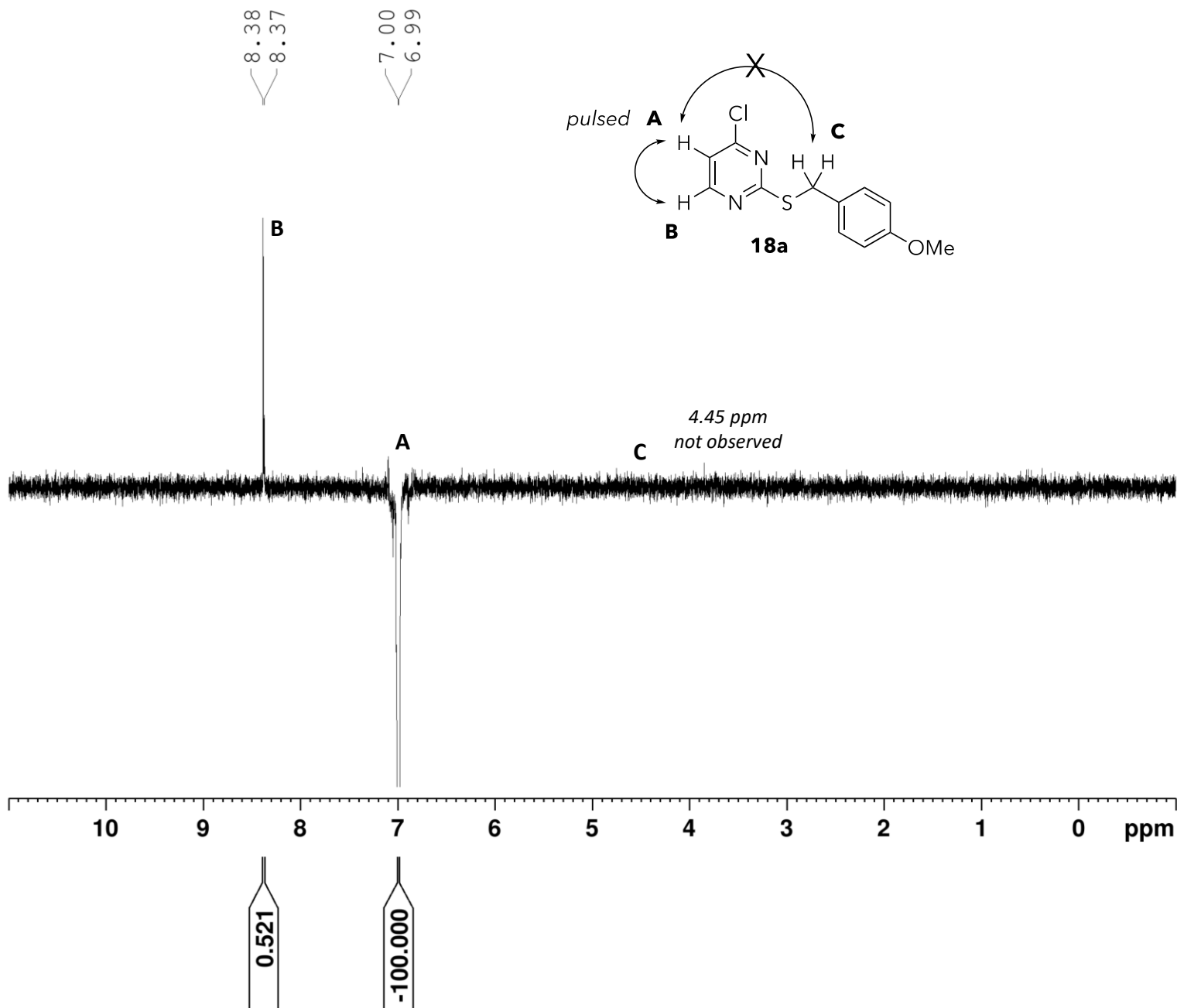
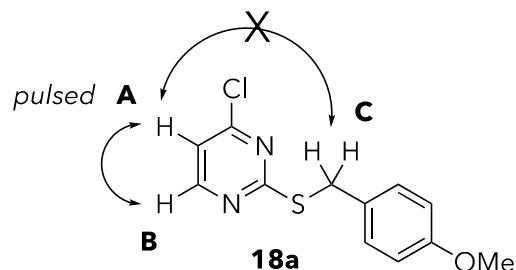
F2 - Processing parameters
SI 32768
SF 125.7829164 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 18a

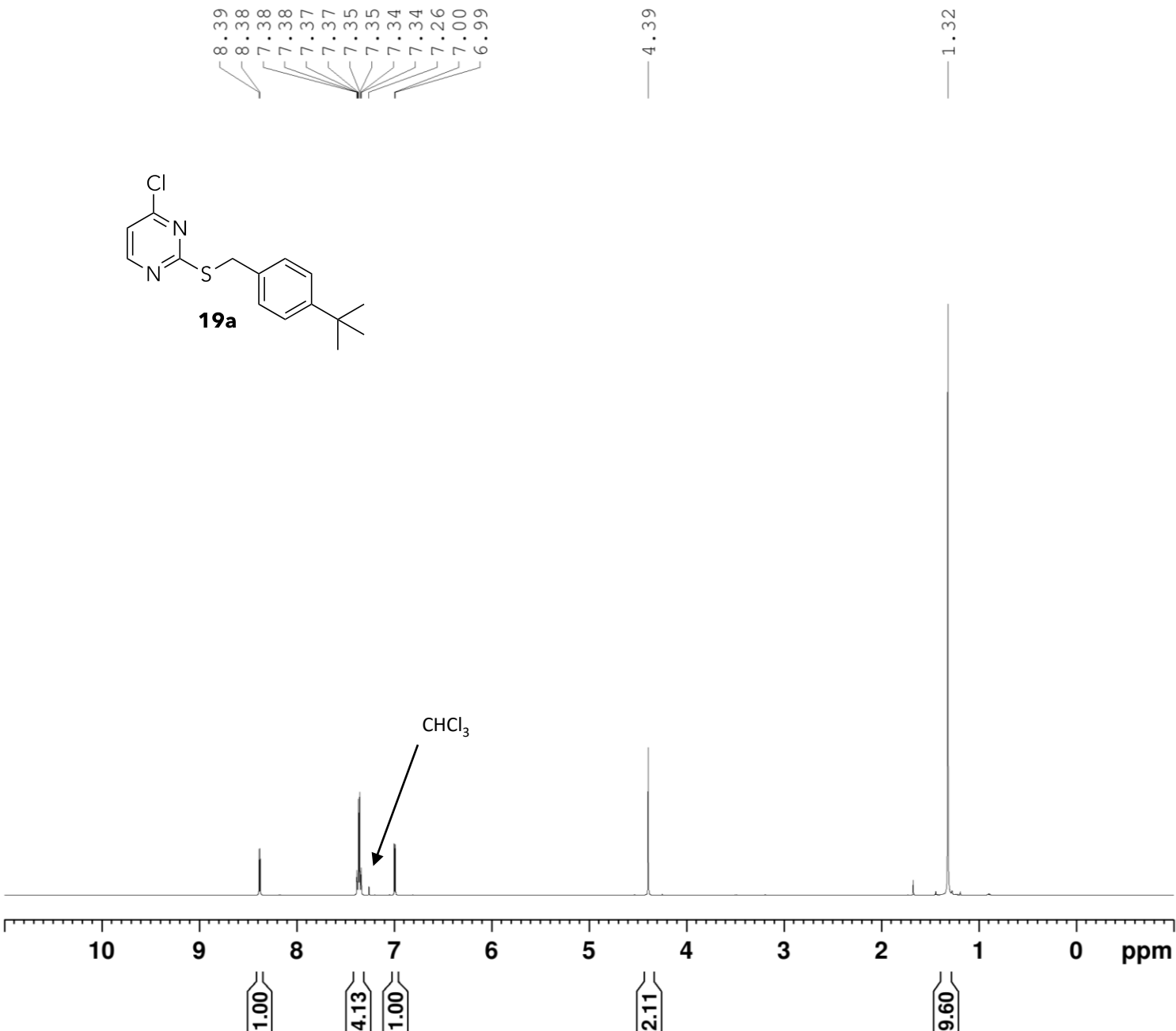
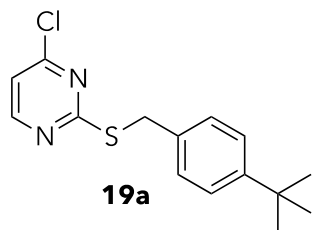
Current Data Parameters
NAME 18a_AR_02_183
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240816
Time 14.33 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnogpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0230908
P1 12.00 usec
P12 39855.84 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 424.24 Hz
SPW2 0.00002450 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300127 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **19a**

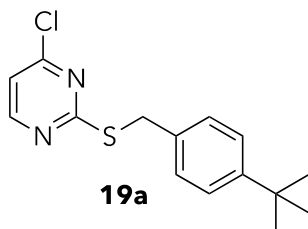


Current Data Parameters
NAME OJ-5-172-H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230818
Time 19.22 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.9
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **19a**



— 173.36
— 161.11
— 158.15
— 150.44

— 133.83
— 128.96
— 125.60
— 116.74

77.42
77.16
76.91

35.30
34.62
31.43

CDCl₃

Current Data Parameters
NAME OJ-5-172-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230818
Time 20.20 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters
SI 32768
SF 125.7829243 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

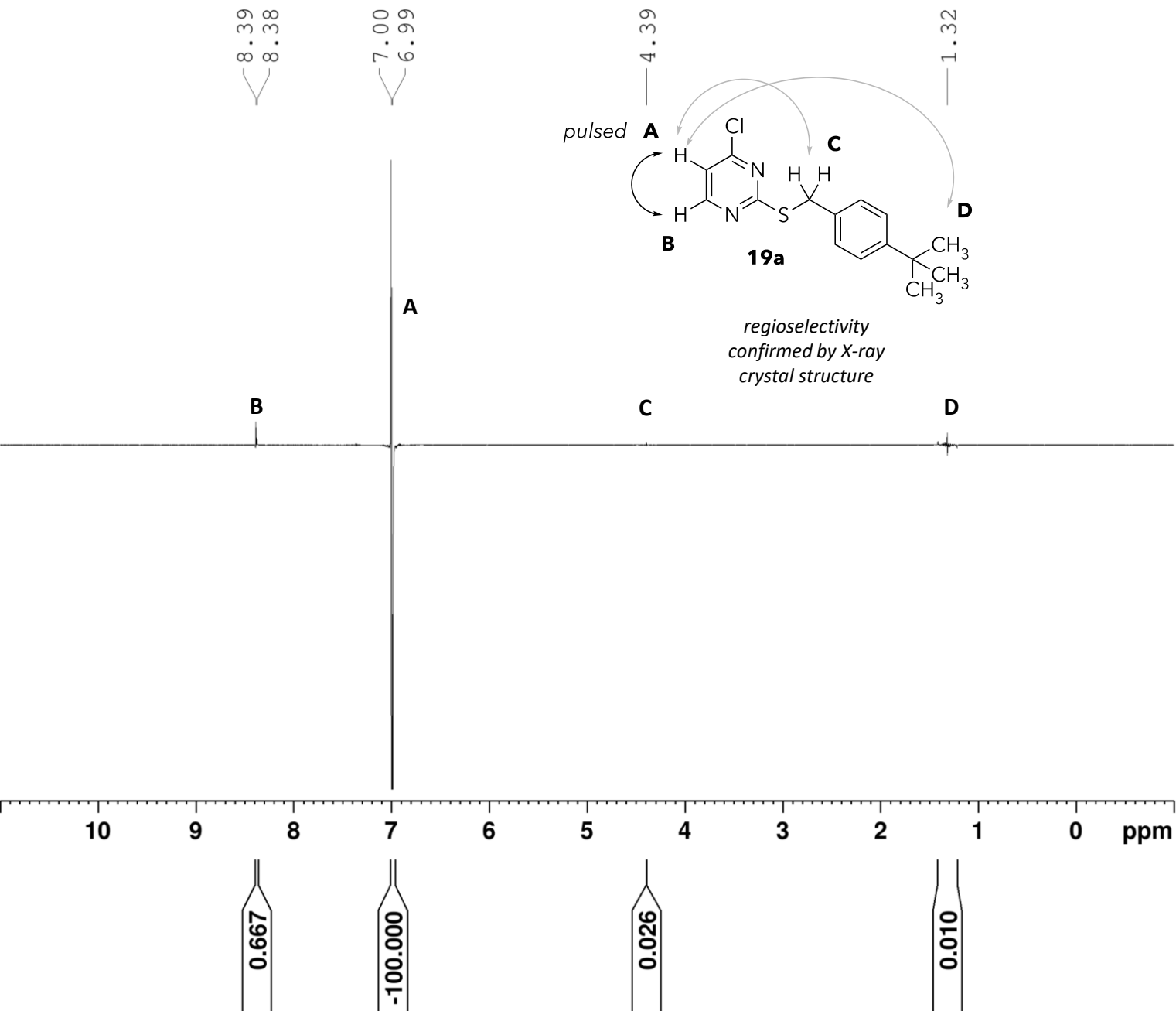
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of **19a**

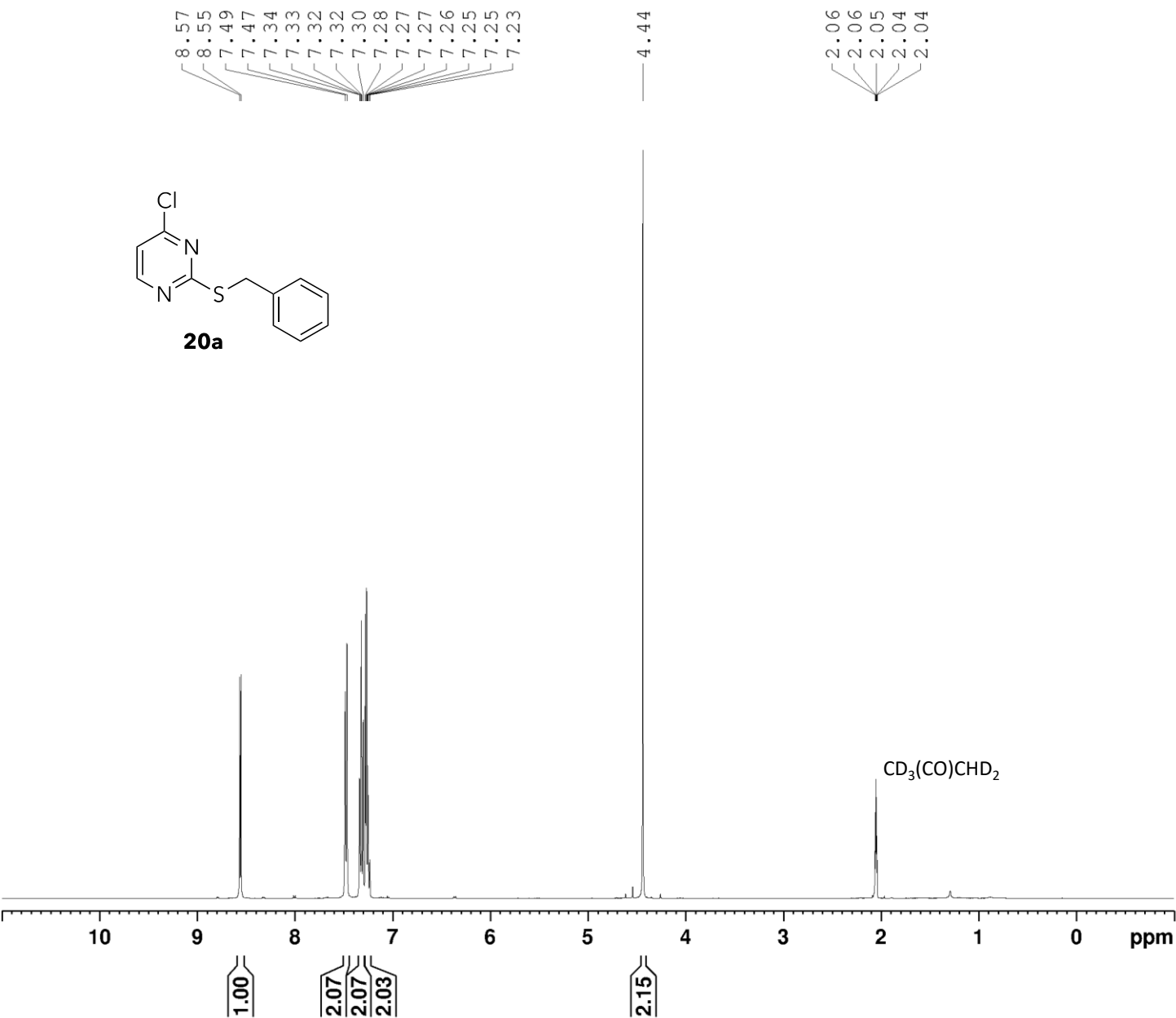
Current Data Parameters
NAME OJ-5-172-NOE
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230818
Time 19.05 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 122.05
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOFINS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0192299
P1 12.00 usec
P12 65262.40 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 422.31 Hz
SPW2 0.00000914 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300146 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of 20a

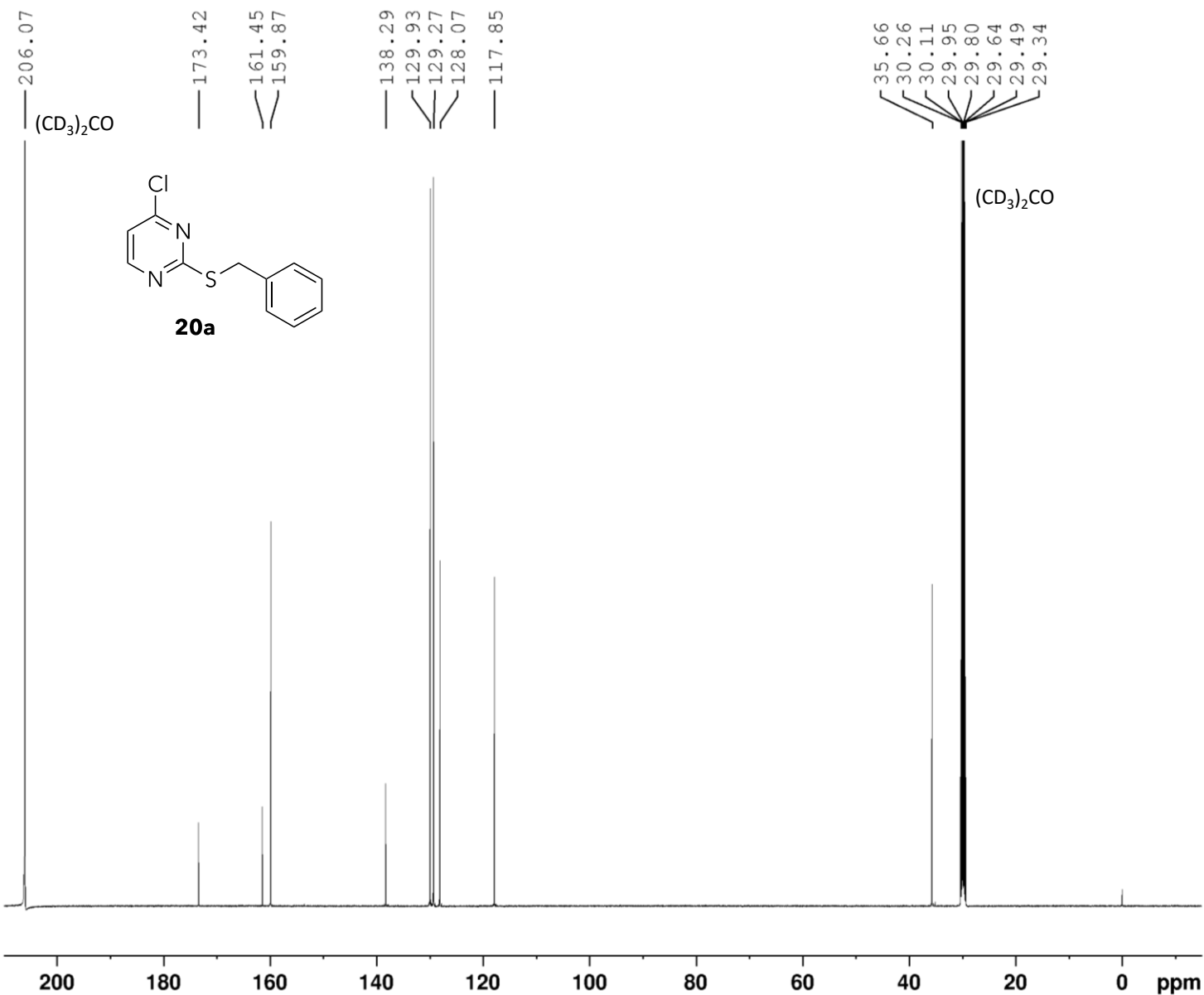


Current Data Parameters
NAME OJ-4-76-H-1-Acetone
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240704
Time 10.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1299628 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 20a



Current Data Parameters

NAME	OJ-4-76-C-Acetone
EXPNO	12
PROCNO	1

F2 - Acquisition Parameters

Date_	20240923
Time	23.50 h
INSTRUM	spect
PROBHD	Z125869_0055 (
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	1.1010048 sec
RG	190.44
DW	16.800 usec
DE	18.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7955118 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
PLW1	56.90299988 W
SFO2	500.2320009 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	11.44699955 W
PLW12	0.25756001 W
PLW13	0.12955000 W

F2 - Processing parameters

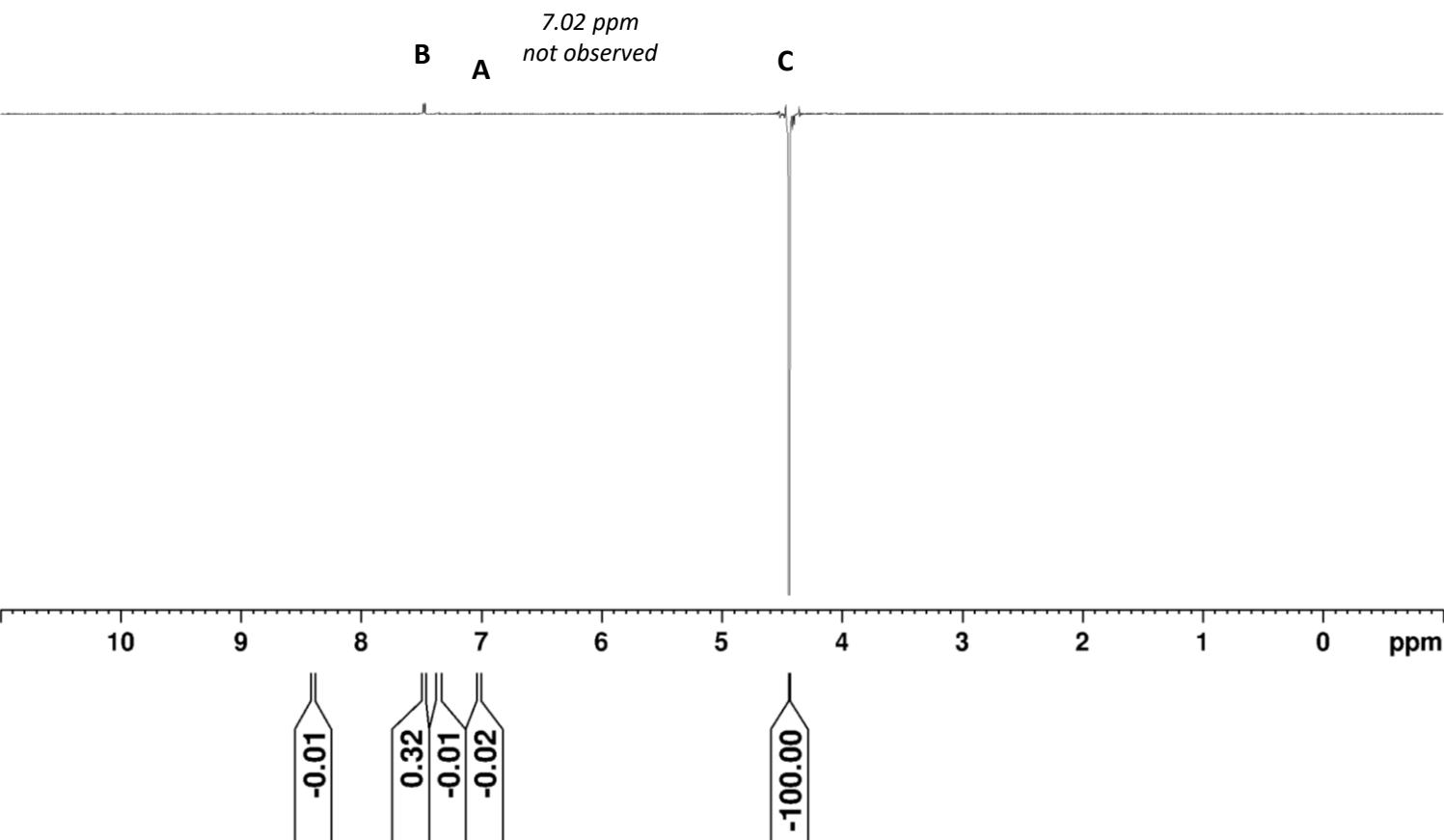
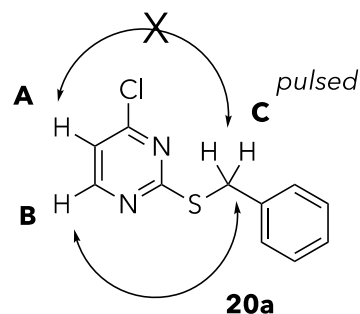
SI	32768
SF	125.7828299 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

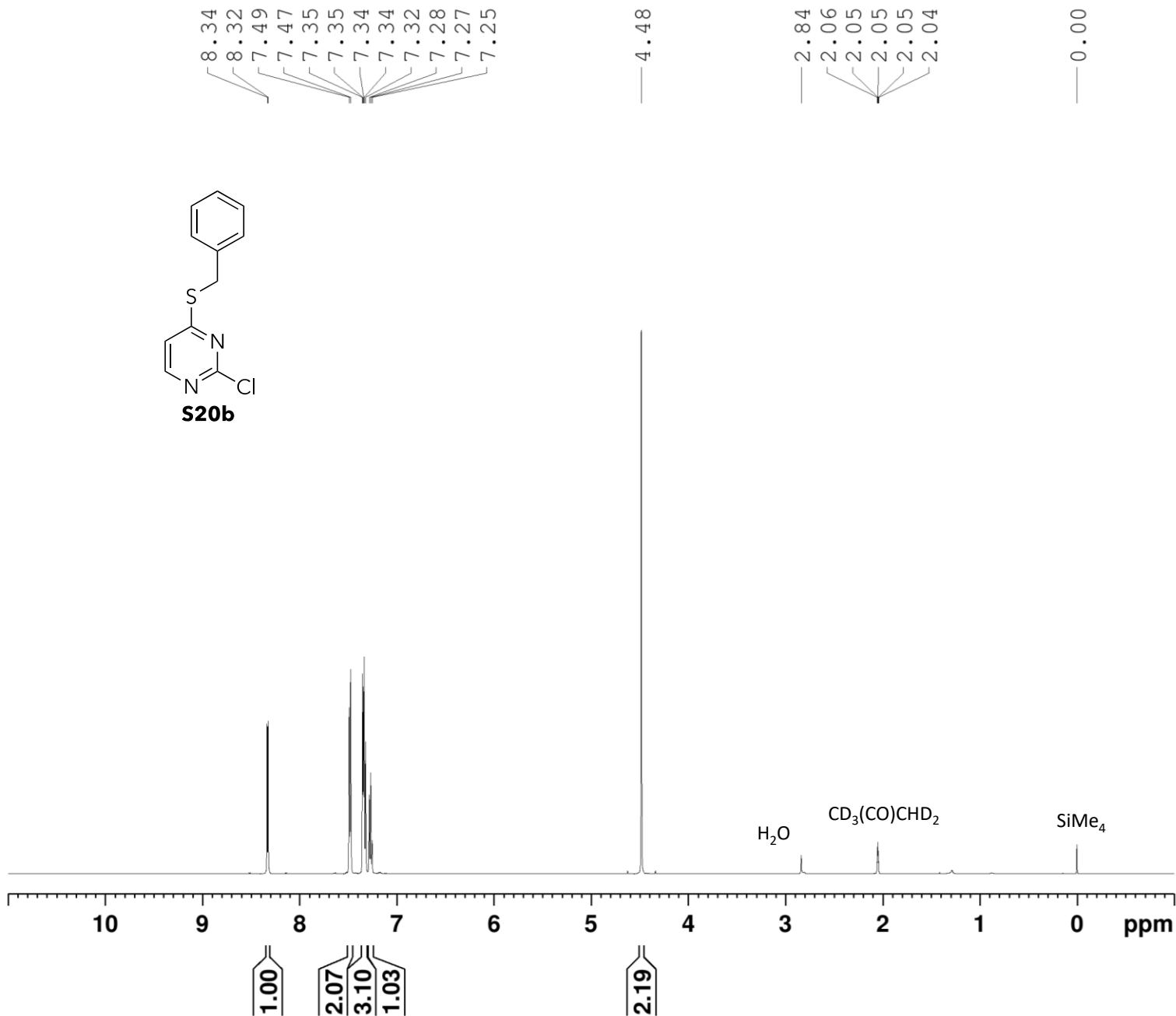
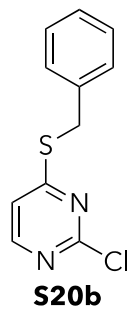
1D NOE of 20a

Current Data Parameters
NAME OJ-4-76-H-NOE
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230425
Time 10.39 h
INSTRUM spect
PROBHD Z125869_0055 {
PULPROG selnpgpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOFINS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 4.4303451
P1 12.00 usec
P12 65260.78 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -872.73 Hz
SPW2 0.00000914 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2299965 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00





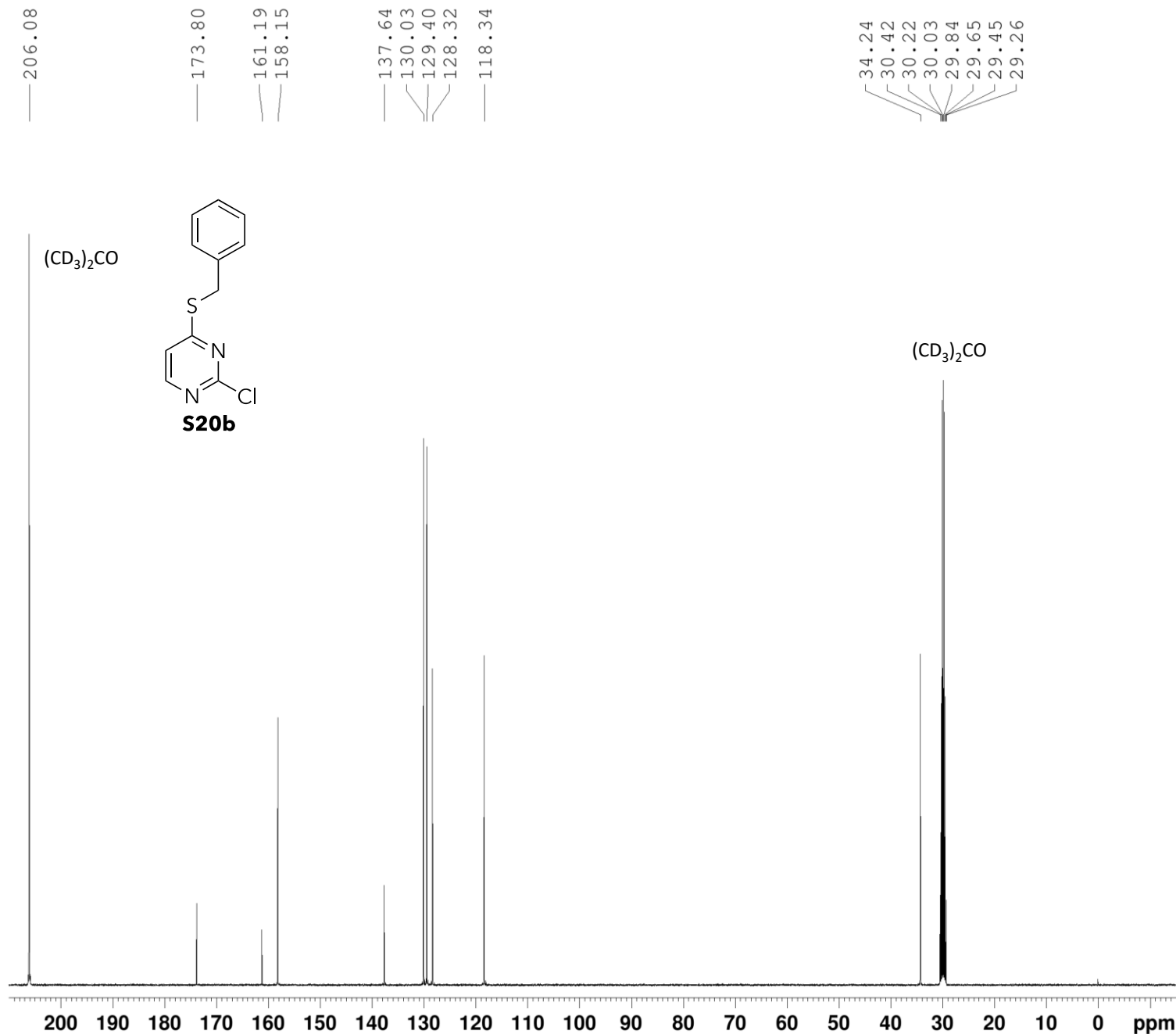
¹H spectrum of S20b

Current Data Parameters
 NAME S20b_AR_240709
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240729
 Time 15.21 h
 INSTRUM spect
 PROBHD Z125869_0055 (
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767999 sec
 RG 35.18
 DW 50.000 usec
 DE 16.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 TD0 1
 SFO1 500.2330889 MHz
 NUC1 1H
 P0 4.00 usec
 P1 12.00 usec
 PLW1 11.44699955 W

F2 - Processing parameters
 SI 65536
 SF 500.2300087 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C spectrum of S20b



Current Data Parameters
NAME S20b_AR_240709
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240729
Time 22.08 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6126836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

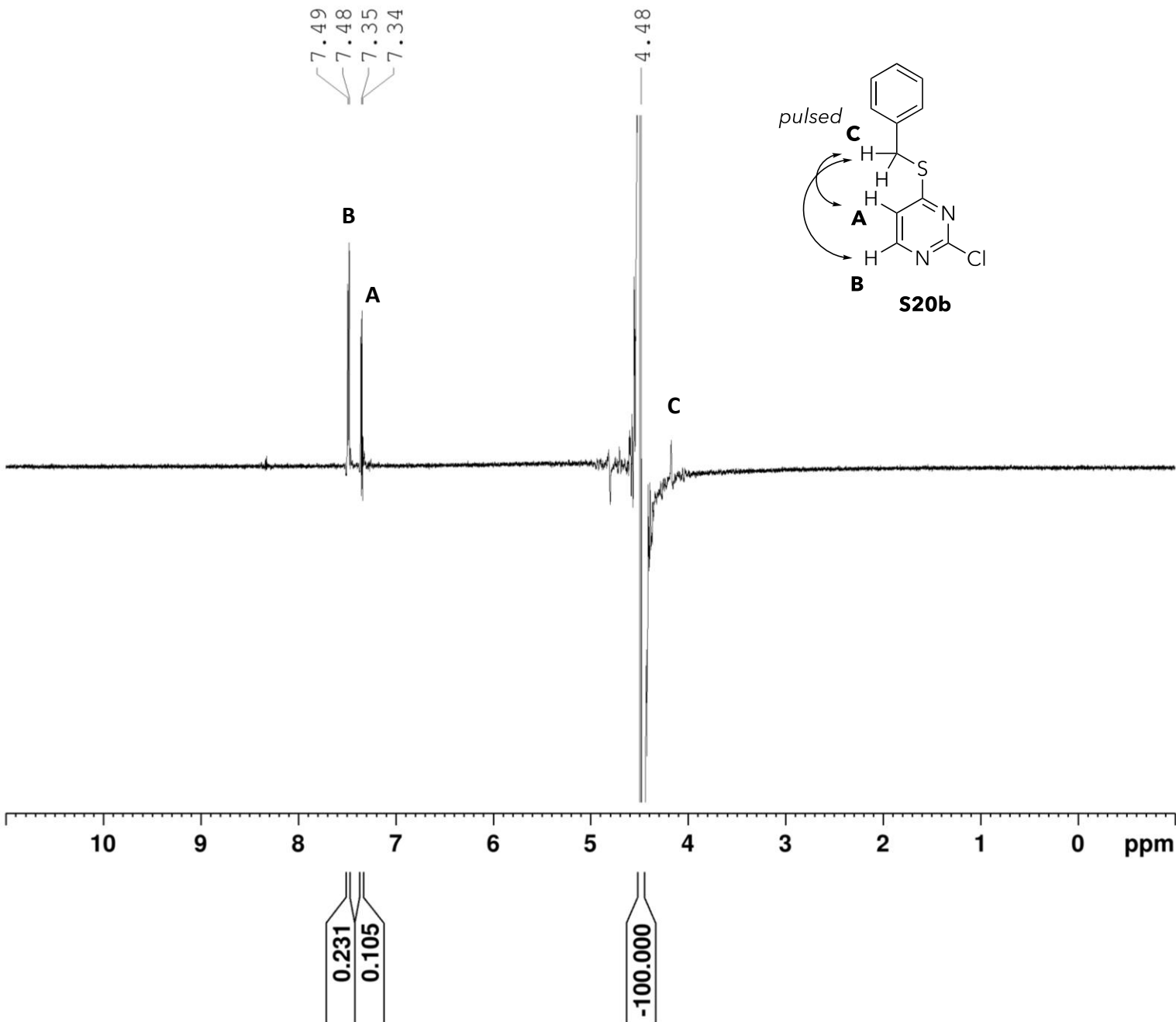
1D NOE of S20b

Current Data Parameters
NAME S20b_AR_240709
EXPNO 4
PROCNO 1

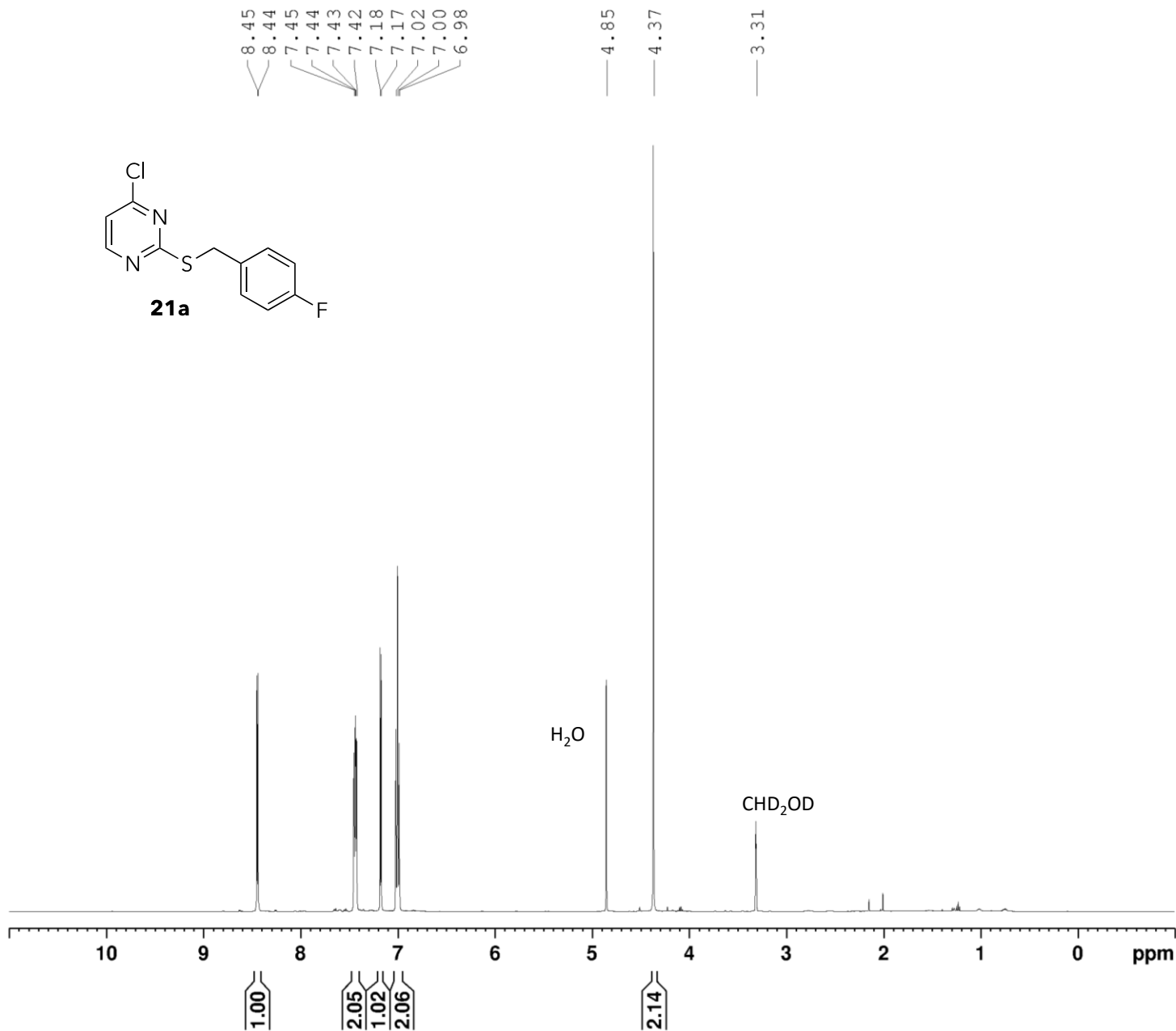
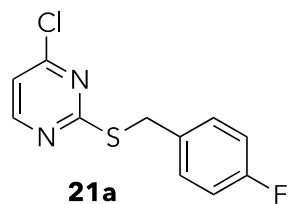
F2 - Acquisition Parameters
Date_ 20240729
Time 15.34 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT Acetone
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1

ZGOPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 4.5018158
P1 12.00 usec
P12 36767.76 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 -836.98 Hz
SPW2 0.00002879 W
SPNAM[29 Crp60,20,20.1
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300099 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **21a**

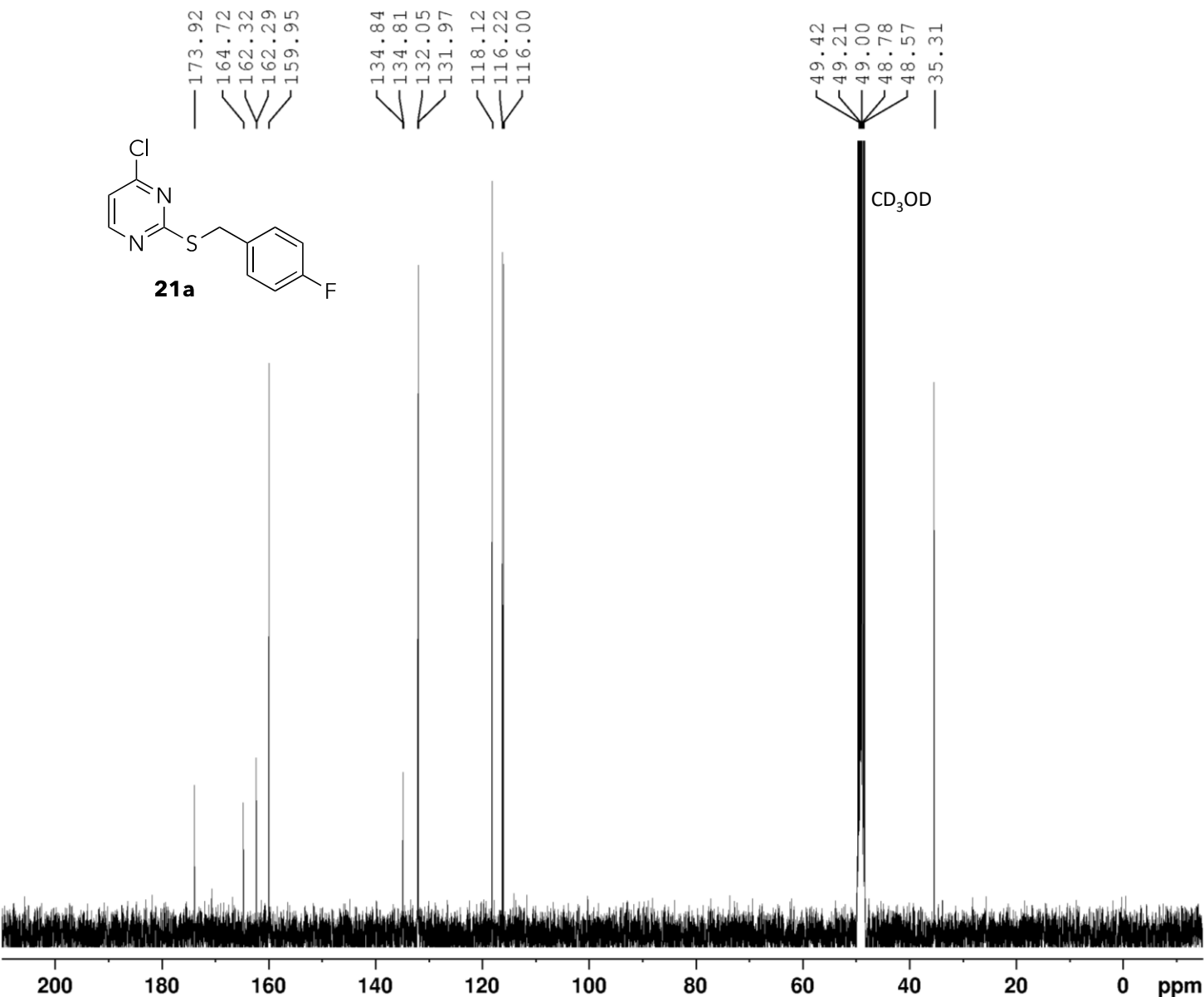


Current Data Parameters
NAME OJ-6-49-H-1
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240611
Time 11.07 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **21a**

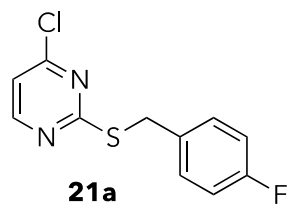


Current Data Parameters
NAME OJ-6-49-C-MeOH
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240925
Time 14.58 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SF01 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.9169982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6126275 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹⁹F spectrum of **21a**



-63.72

C₆H₅CF₃

-116.90

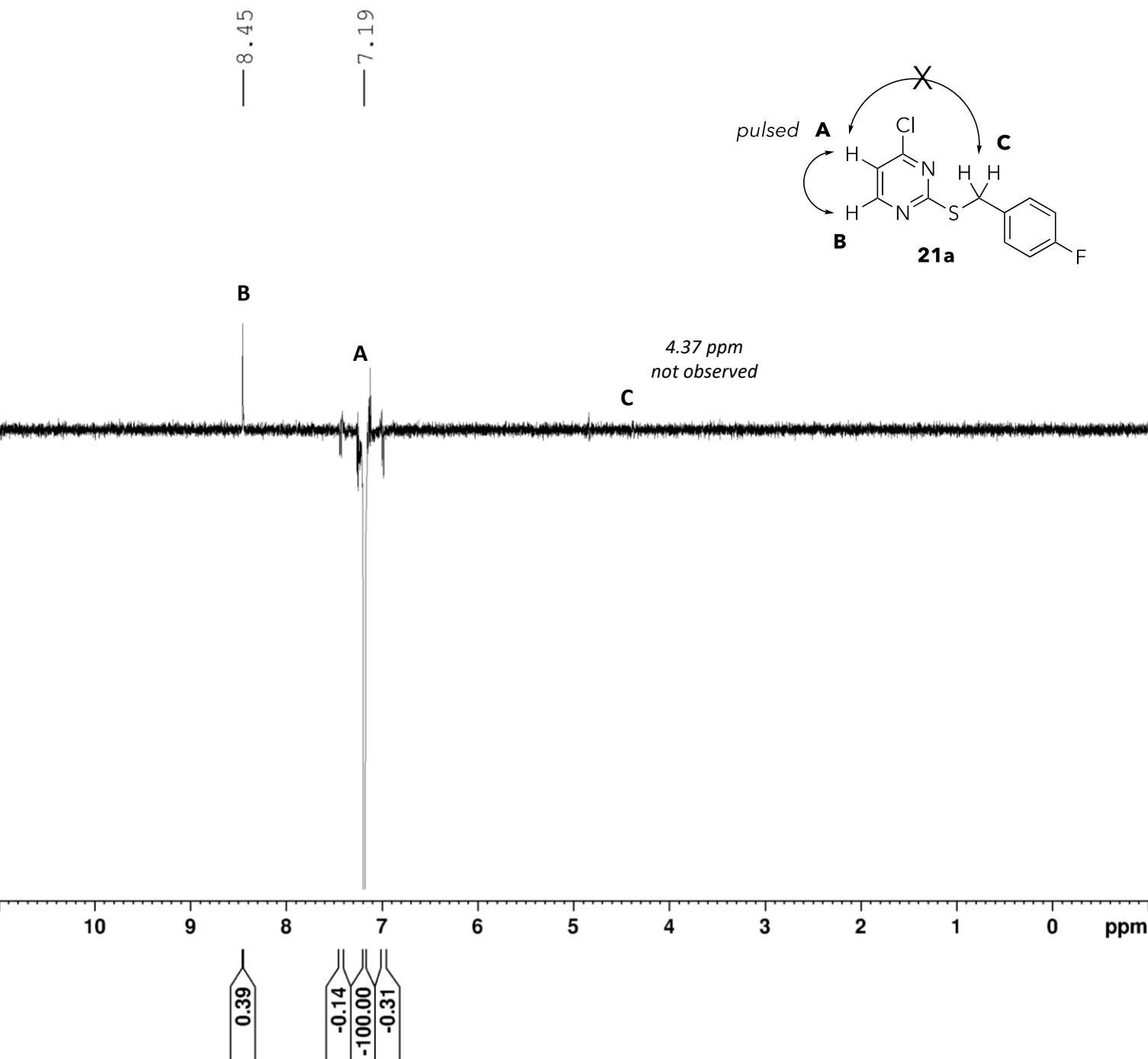
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

Current Data Parameters
NAME OJ-6-49-F-methanol
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240925
Time 15.27 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg
TD 131072
SOLVENT MeOD
NS 16
DS 4
SWH 90909.091 Hz
FIDRES 1.387163 Hz
AQ 0.7208960 sec
RG 101
DW 5.500 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 12.00 usec
PLW1 38.11899948 W

F2 - Processing parameters
SI 65536
SF 376.4981693 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NOE of 21a

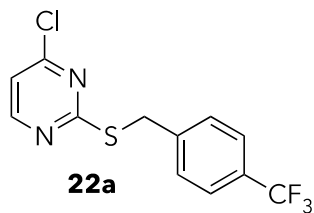


Current Data Parameters
 NAME OJ-6-49-NOE-MeOH-1
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240911
 Time 11.18 h
 INSTRUM spect
 PROBHD Z125869_0055 {
 PULPROG selnogpzs.2
 TD 65536
 SOLVENT MeOD
 NS 32
 DS 4
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767999 sec
 RG 190.44
 DW 50.000 usec
 DE 20.46 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27272600 sec
 D31 0.30000001 sec
 TD0 1
 ZGPTNS -DCALC_SPOFFS
 SFO1 500.2330889 MHz
 NUC1 1H
 CNST21 7.2238698
 P1 12.00 usec
 P12 68290.84 usec
 P32 20000.00 usec
 P44 240.00 usec
 PLW1 11.44699955 W
 SPNAM[2] Gausl_180r.1000
 SPOAL2 0.500
 spoffs2 524.68 Hz
 SPW2 0.00000835 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 spoffs29 0 Hz
 SPW29 0.06296300 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 spoffs30 0 Hz
 SPW30 1.83150005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 500.2300186 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 481 1.00

¹H spectrum of **22a**



8.39
8.38
7.56
7.26
7.02
7.01

4.42

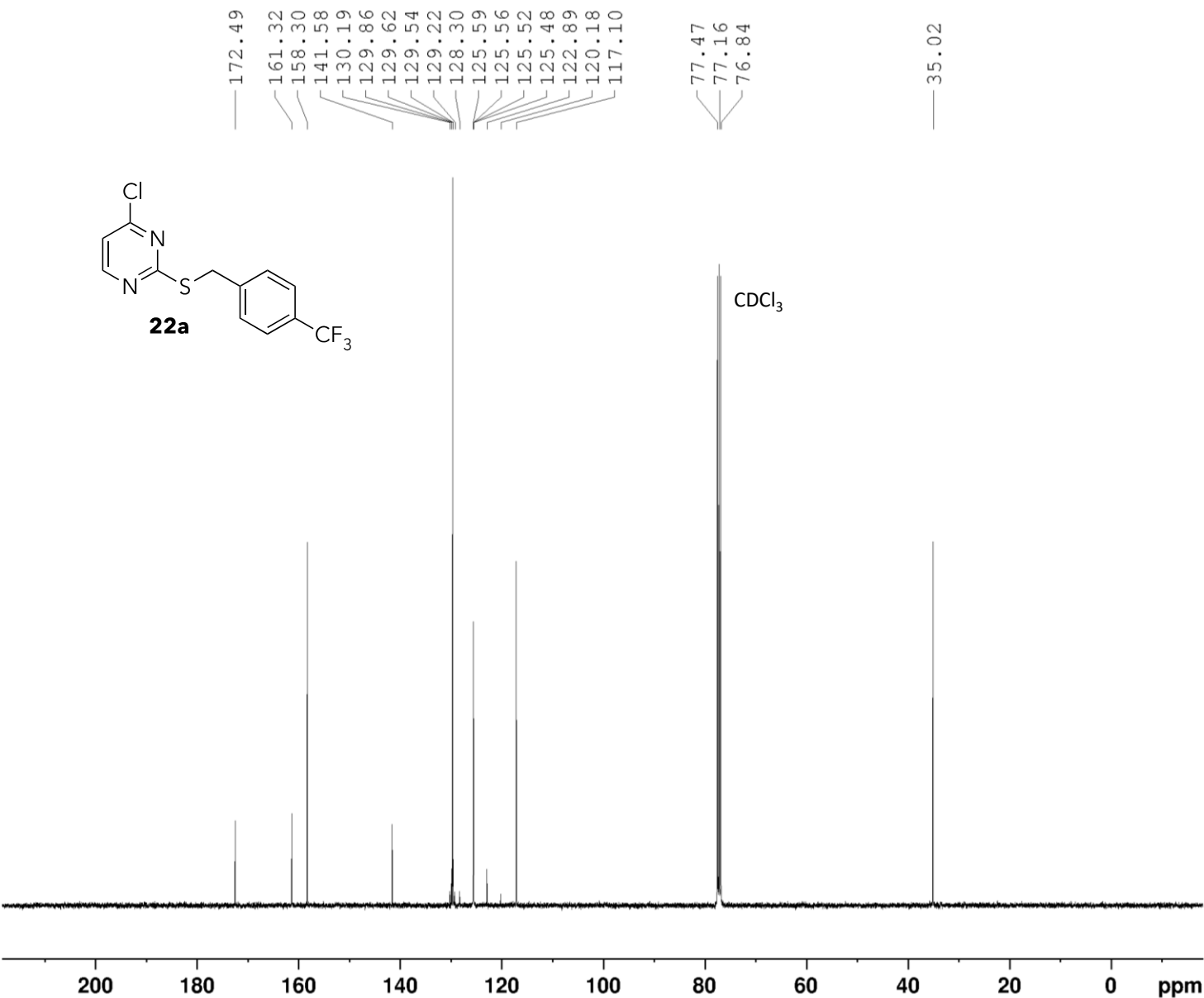
CHCl₃

Current Data Parameters
NAME OJ-8-240624-3-1
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240626
Time 12.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **22a**

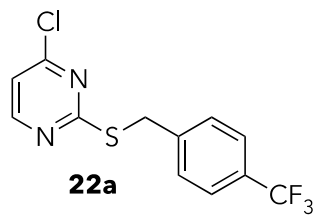


Current Data Parameters
NAME OJ-8-240624-3-C
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240626
Time 13.46 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

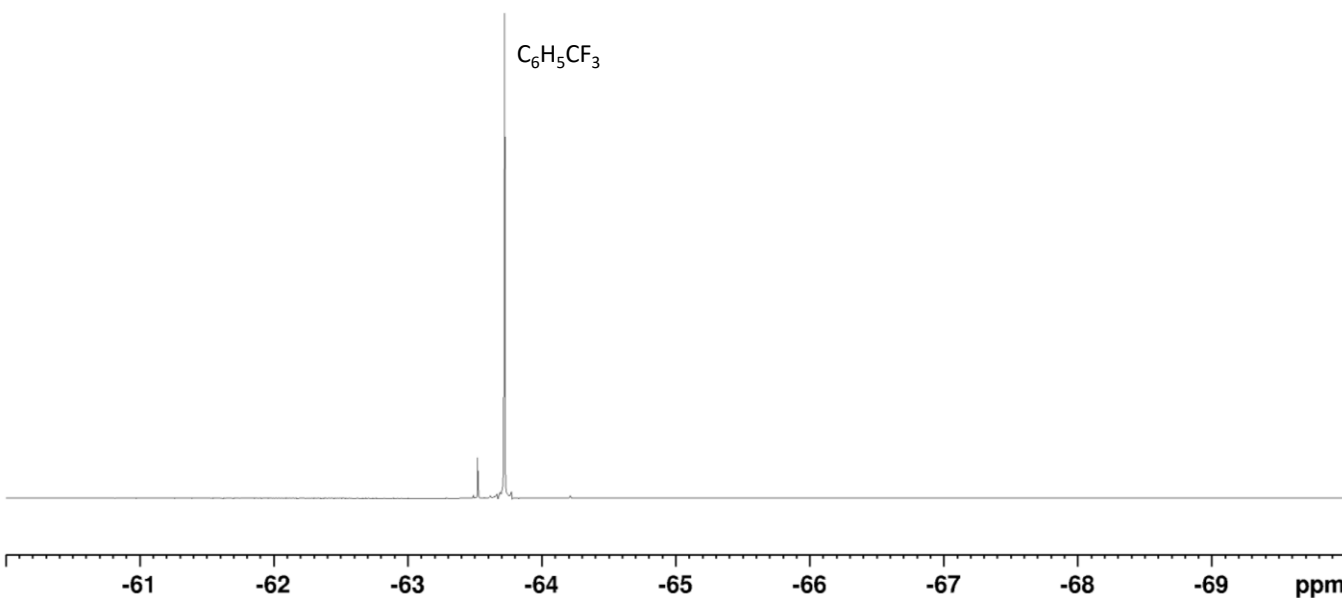
F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹F spectrum of **22a**



— -63.523
— -63.722

C₆H₅CF₃



Current Data Parameters

NAME OJ-8-240624-3-F
EXPNO 10
PROCNO 1

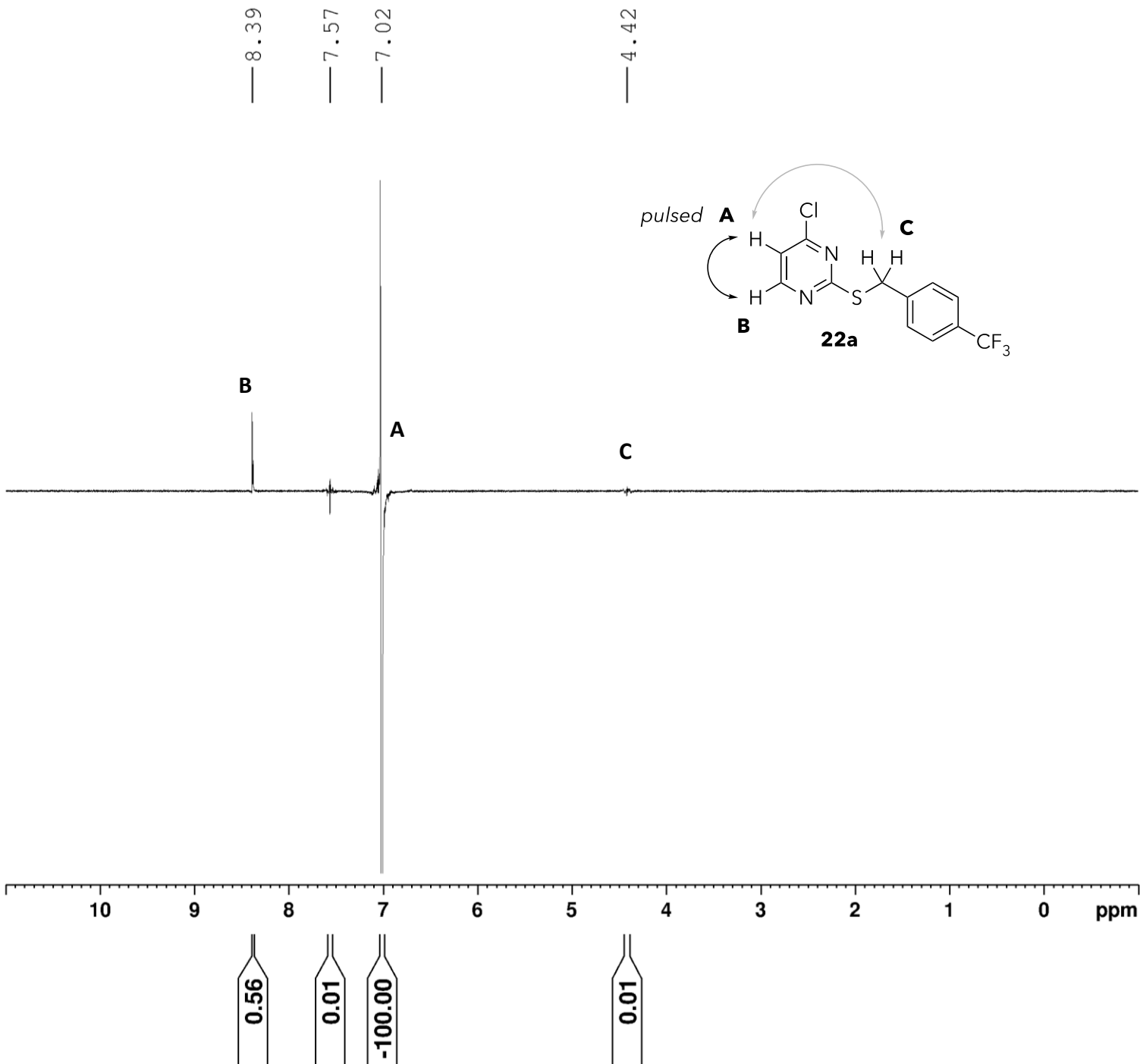
F2 - Acquisition Parameters

Date_ 20240528
Time 12.41 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-
PULPROG zg
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 90909.091 Hz
FIDRES 1.387163 Hz
AQ 0.7208960 sec
RG 101
DW 5.500 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 12.00 usec
PLW1 38.11899948 W

F2 - Processing parameters

SI 65536
SF 376.4987283 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NOE of **22a**

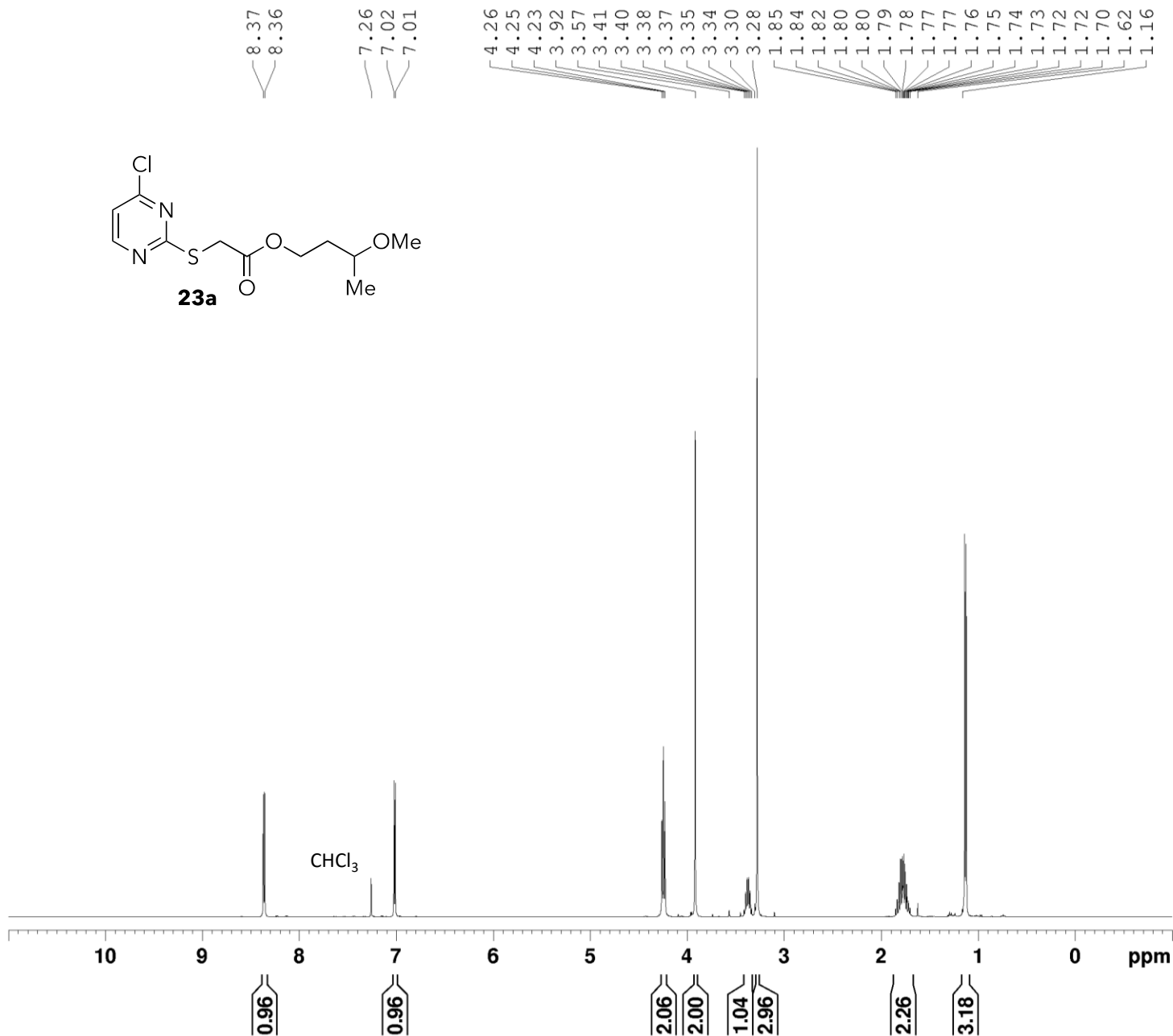
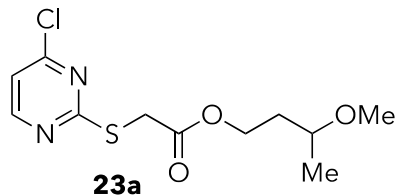


Current Data Parameters
NAME OJ-8-240624-3-NOE
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240627
Time 11.09 h
INSTRUM spect
PROBHD Z125869_0055 {
PULPROG selnpgpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 7.0421801
P1 12.00 usec
P12 55846.15 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 433.79 Hz
SPW2 0.00001248 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 485 1.00

¹H spectrum of **23a**

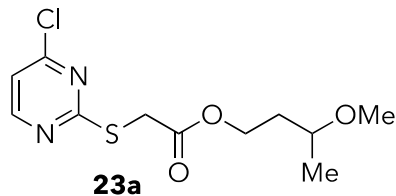


Current Data Parameters
NAME OJ-6-37-H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231030
Time 16.57 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **23a**



171.94
168.78
161.28
158.21

117.17

CDCl₃

77.47
77.16
76.84
73.63
62.91
56.24

35.57
33.70

19.16

Current Data Parameters
NAME OJ-6-37-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231030
Time 18.57 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

F2 - Processing parameters
SI 32768
SF 100.6127574 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

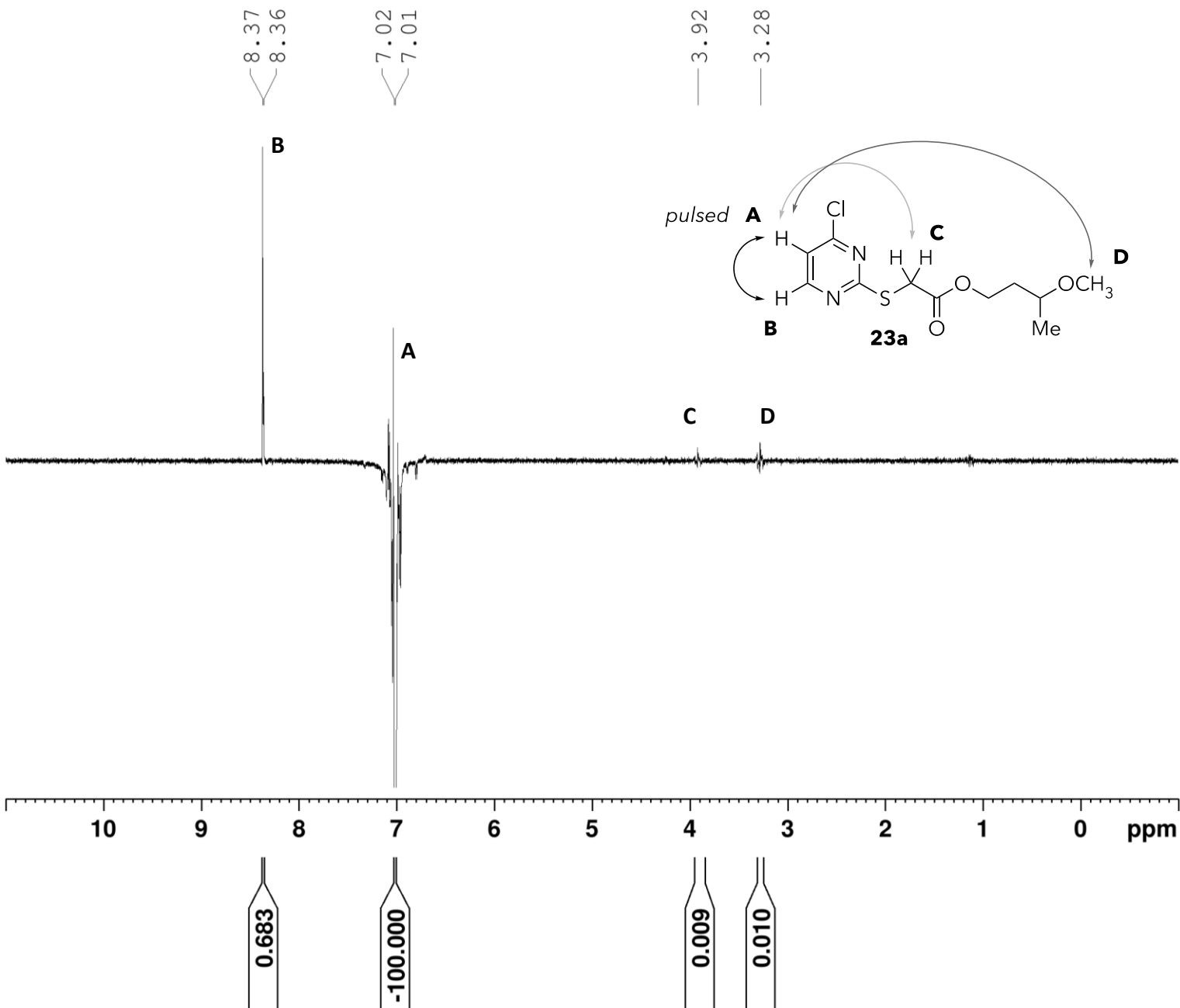
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 23a

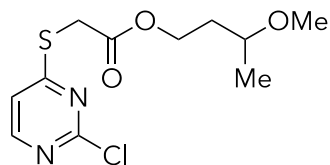
Current Data Parameters
NAME OJ-6-37-NOE
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231211
Time 19.09 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoppzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0359192
P1 12.00 usec
P12 22633.41 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 430.66 Hz
SPW2 0.00007598 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

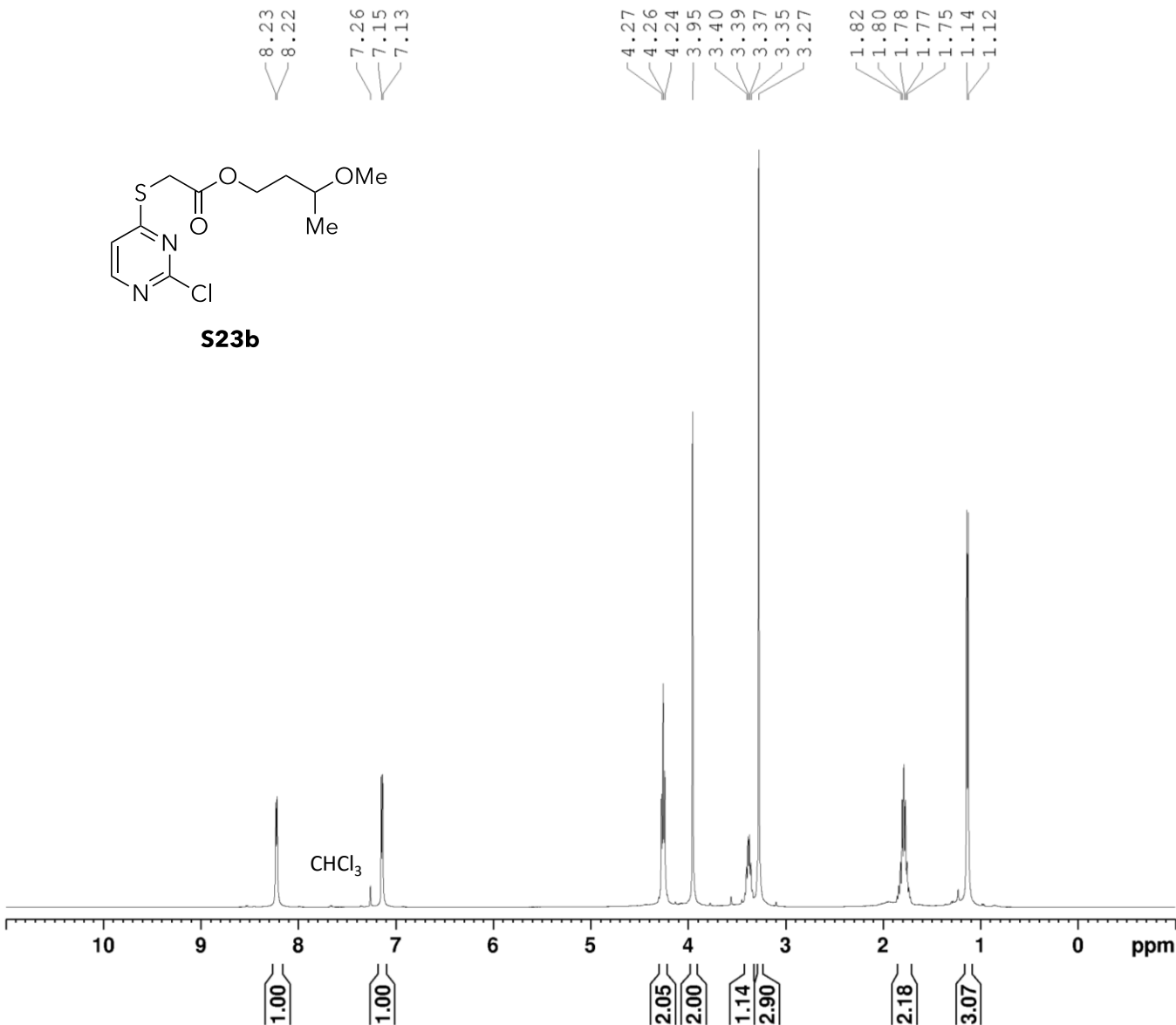
F2 - Processing parameters
SI 32768
SF 500.2300137 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of S23b



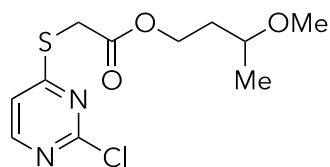
S23b



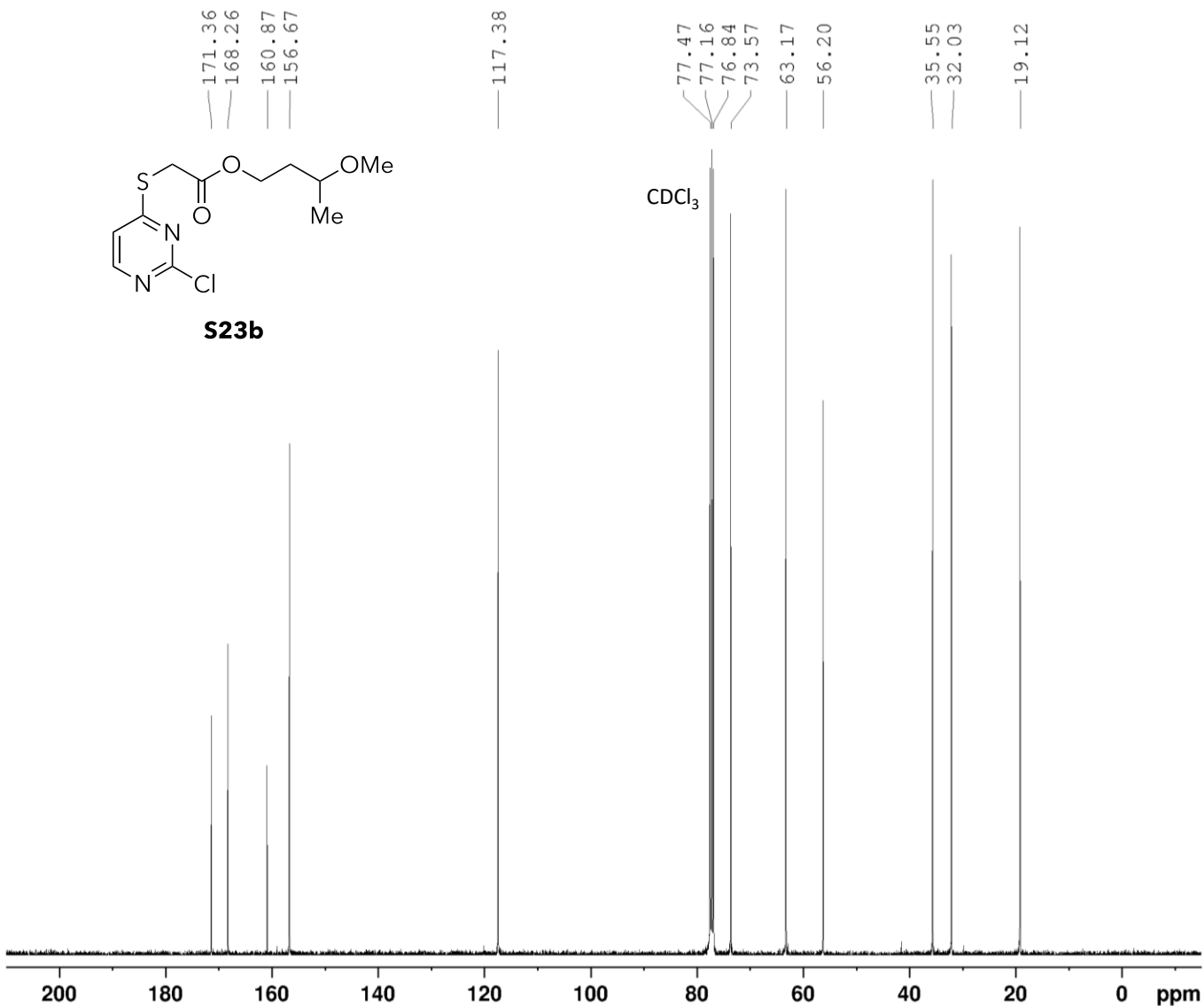
Current Data Parameters
NAME OJ-240730-H-2
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241110
Time 19.48 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 90.5
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



S23b



Current Data Parameters

NAME OJ-240730-C
EXPNO 16
PROCNO 1

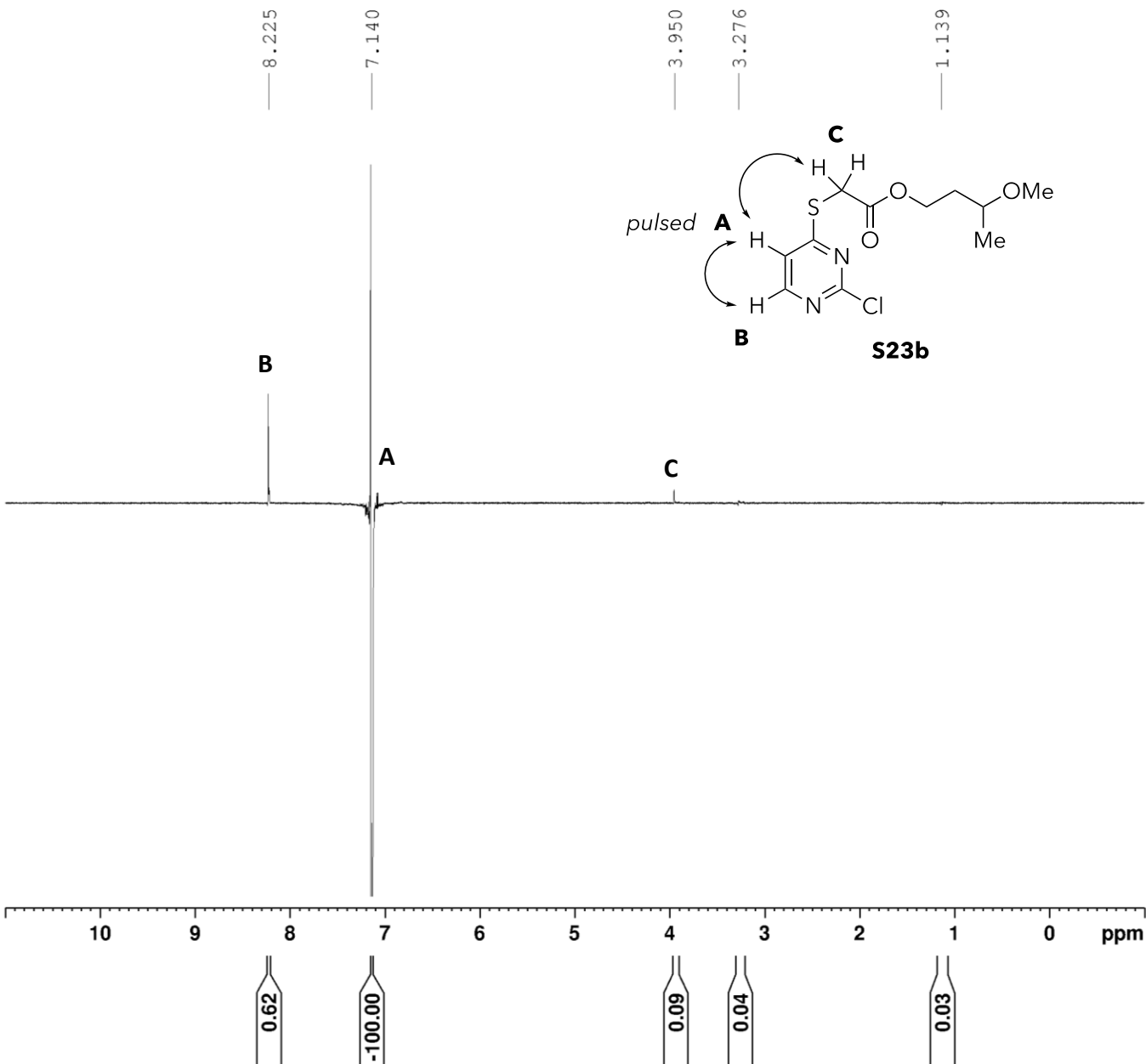
F2 - Acquisition Parameters

Date_ 20241110
Time 20.52 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5.1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters

SI 32768
SF 100.6127595 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **S23b**



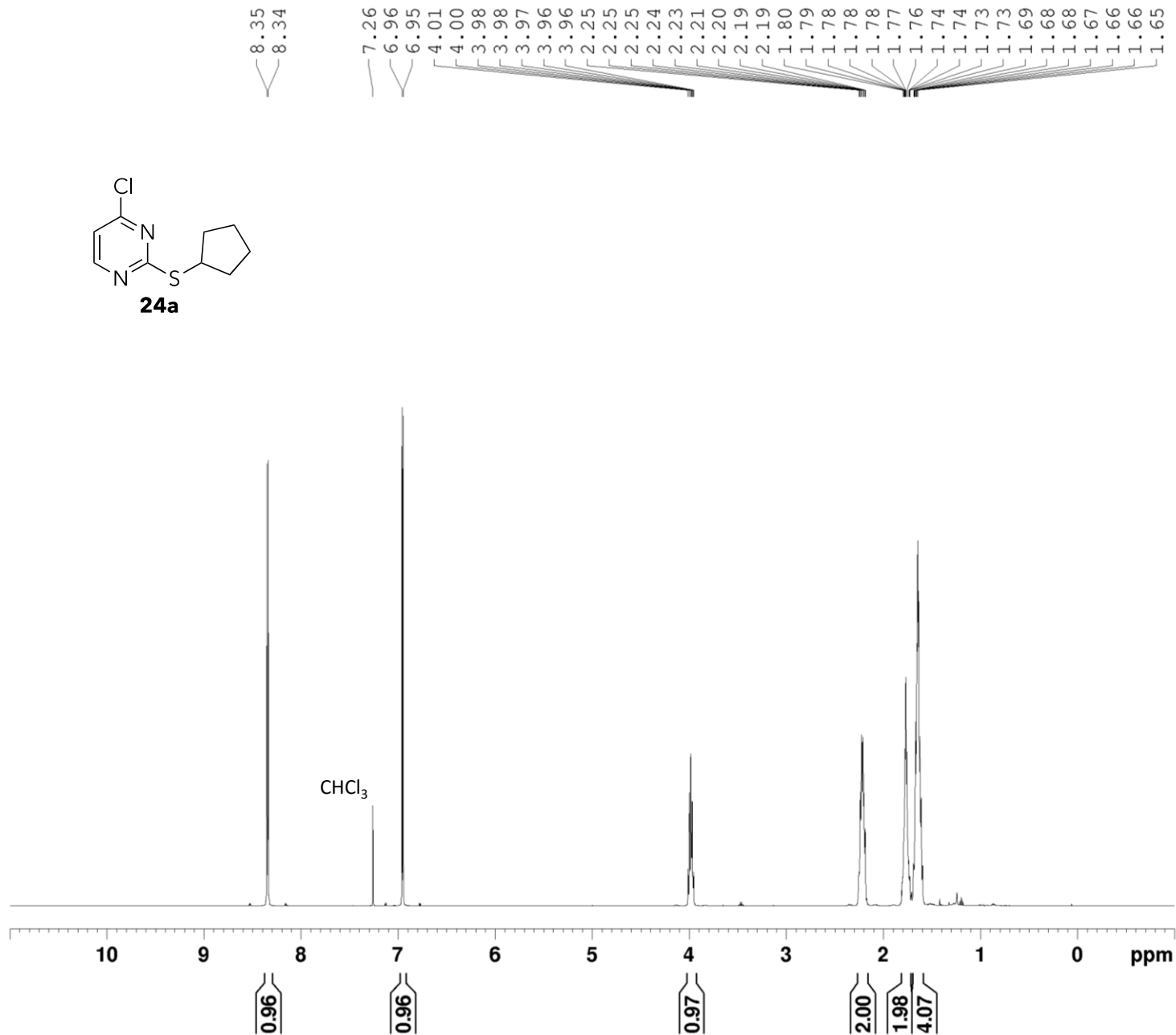
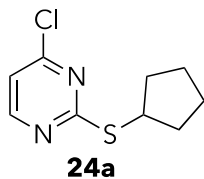
Current Data Parameters

NAME OJ-240730-2-NOE
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20240805
Time 12.07 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S
PULPROG selnogpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 7.1739111
P1 12.00 usec
P12 65269.75 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 499.69 Hz
SPW2 0.00000914 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 491 15.00 %

¹H spectrum of **24a**

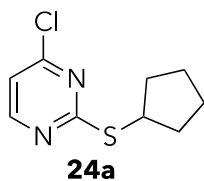


Current Data Parameters
NAME OJ-5-170-H-2
EXPNO 19
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230820
Time 19.46 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 37.93
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 24a



174.50

160.94
158.00

116.34

77.41
77.16
76.90

44.01

33.18

24.98

CDCl₃

Current Data Parameters
NAME OJ-5-170-C-2
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230821
Time 11.22 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters
SI 32768
SF 125.7829200 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

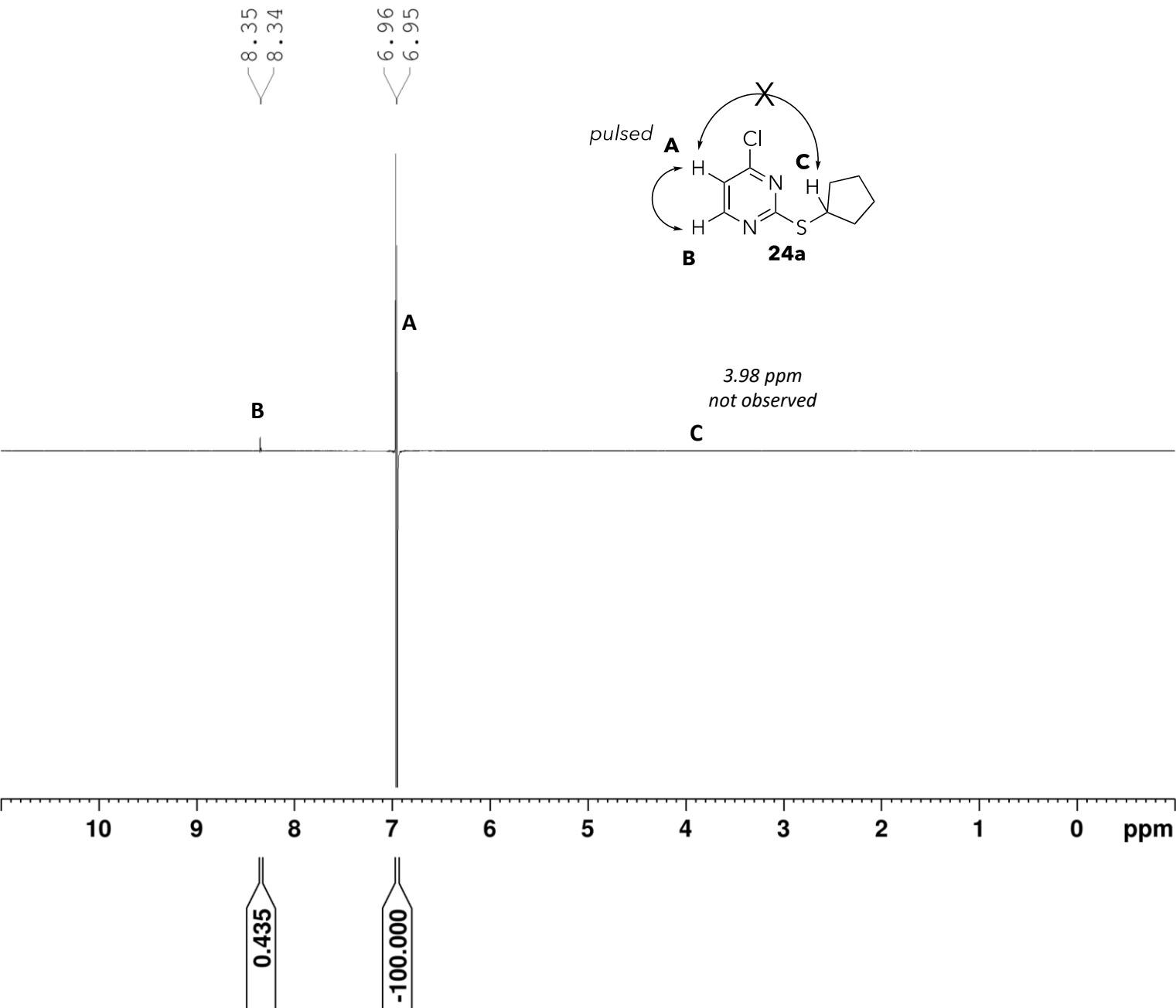
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of **24a**

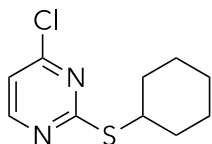
Current Data Parameters
NAME OJ-5-170-NOE
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230820
Time 21.08 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 6.9802170
P1 12.00 usec
P12 62565.61 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 402.79 Hz
SPW2 0.00000994 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

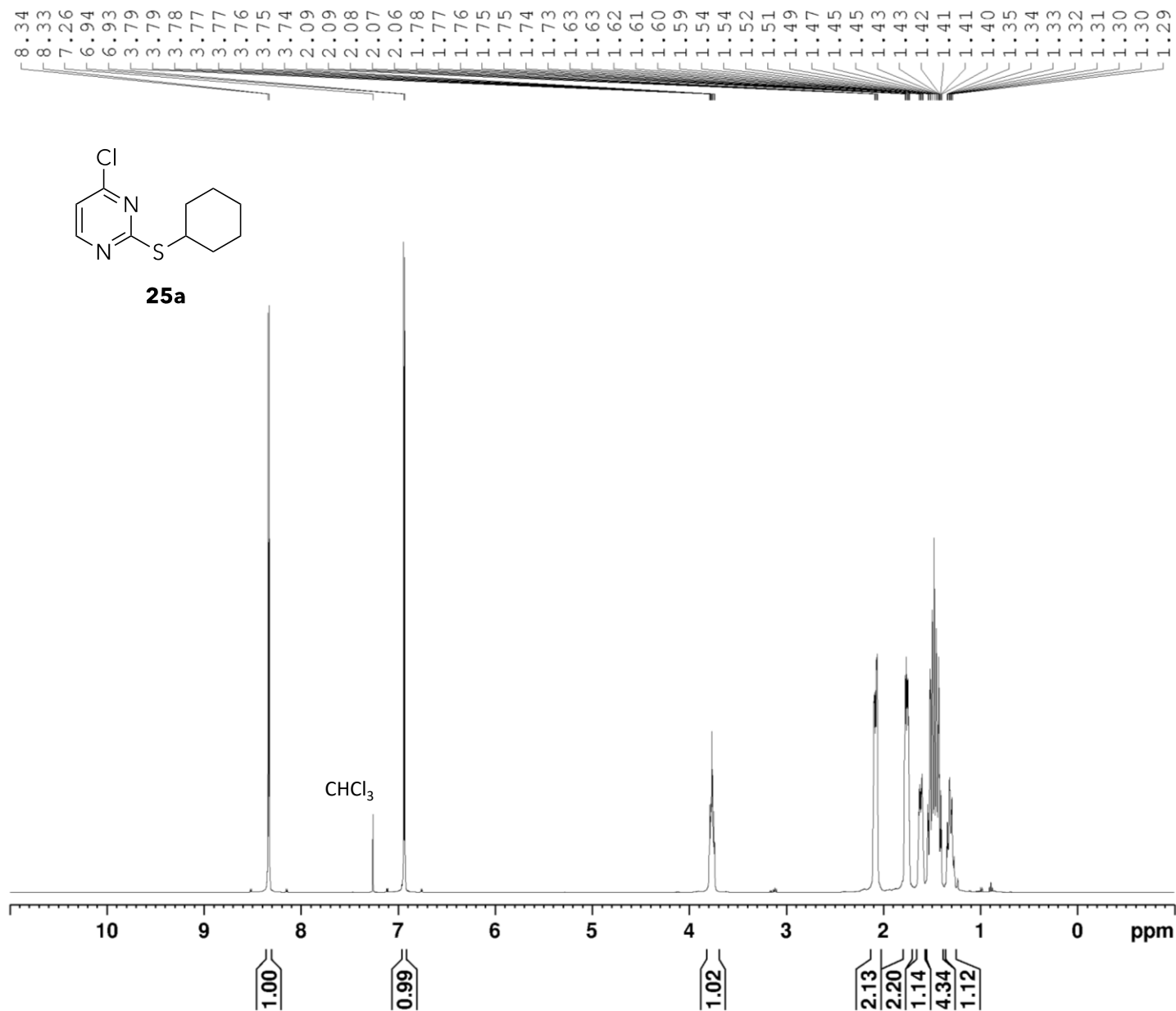
F2 - Processing parameters
SI 32768
SF 500.2300144 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of 25a



25a

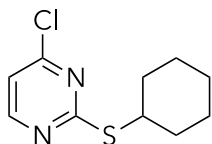


Current Data Parameters
NAME OJ-4-87-H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230425
Time 15.51 h
INSTRUM spect
PROBHD z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 25.21
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 25a



25a

173.80
160.98
158.08
116.34
77.42
77.16
76.91
43.97
32.77
25.98
25.78

CDCl₃

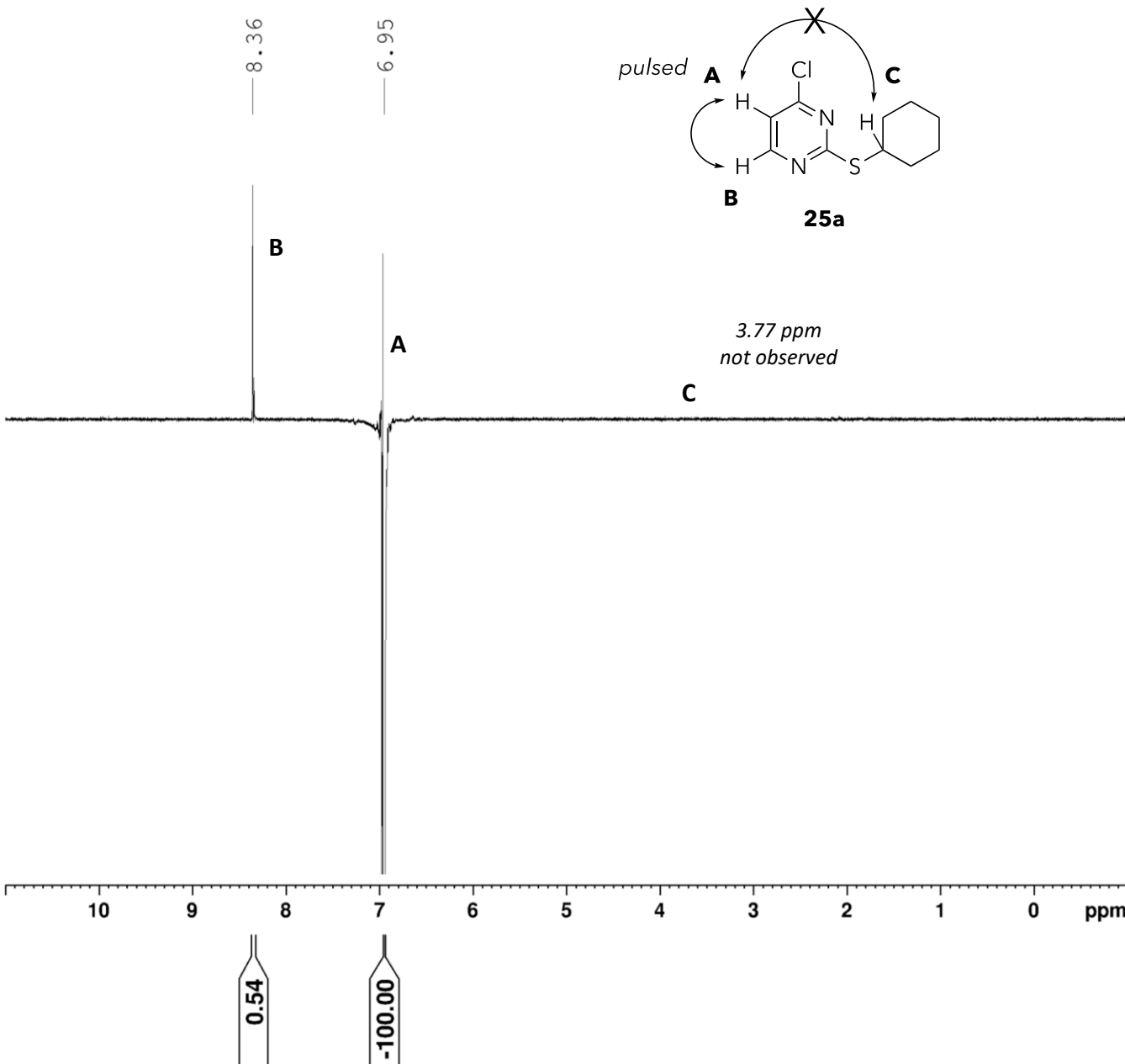
Current Data Parameters
NAME OJ-4-87-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230425
Time 17.24 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

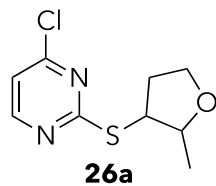
F2 - Processing parameters
SI 32768
SF 125.7829238 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

1D NOE of 25a



¹H spectrum of 26a

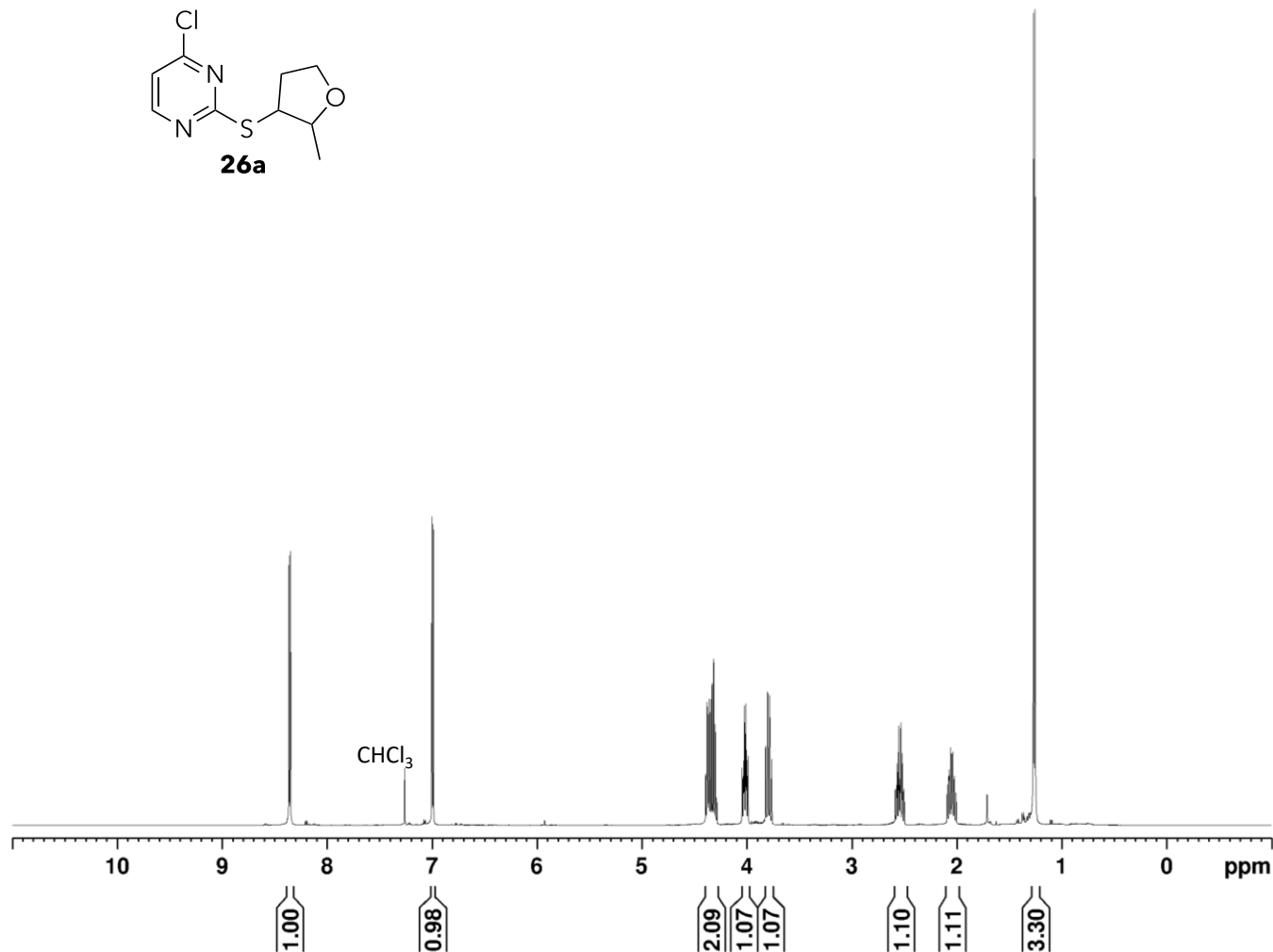


8.36
8.35
7.26
7.00
6.99
4.39
4.38
4.37
4.36
4.36
4.34
4.33
4.32
4.30
4.04
4.03
4.02
4.01
4.00
3.99
3.82
3.80
3.80
3.78
3.78
3.76
2.58
2.57
2.57
2.55
2.55
2.54
2.53
2.52
2.51
2.50
2.09
2.07
2.07
2.06
2.05
2.04
2.02

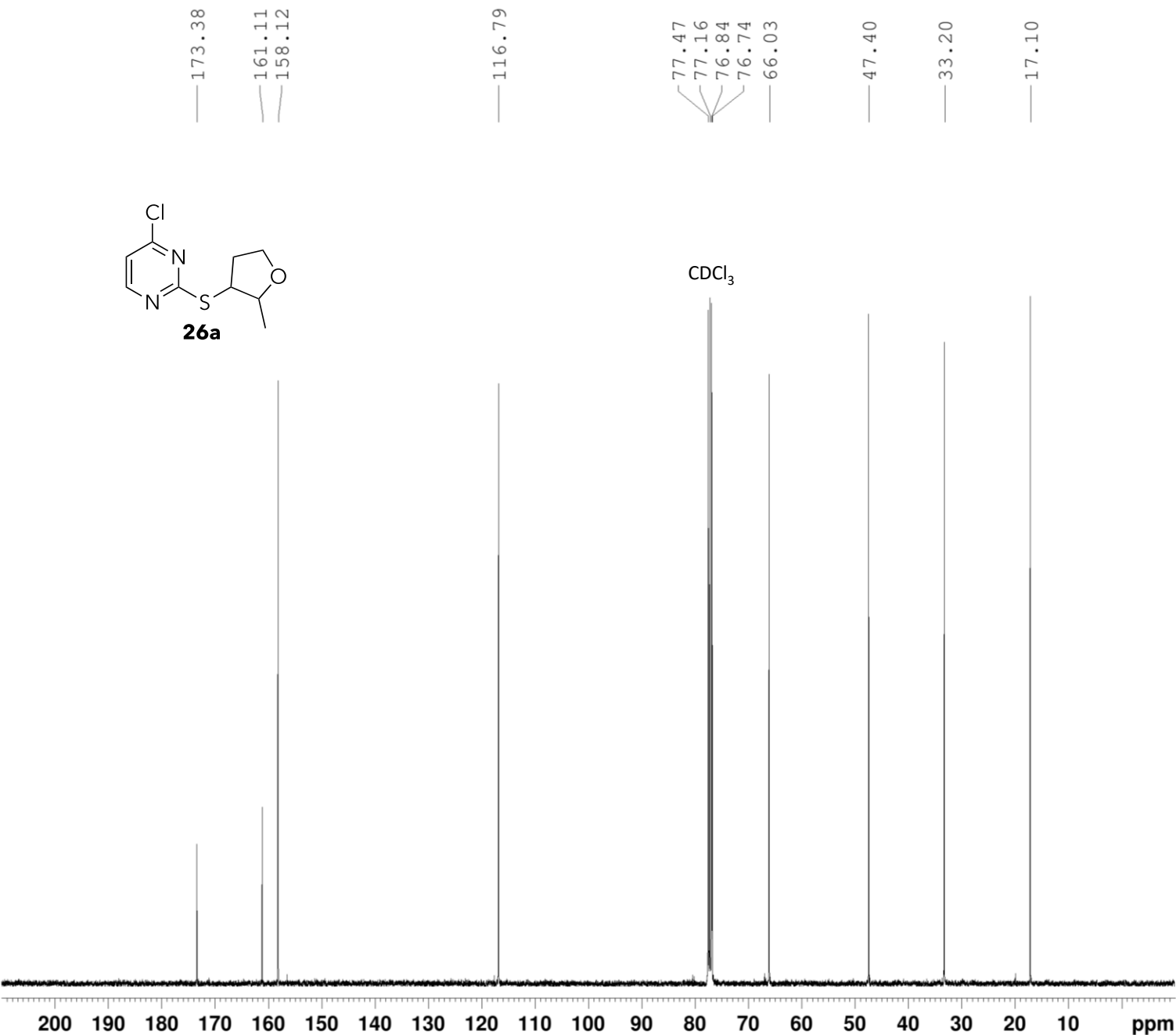
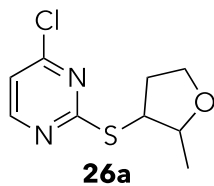
Current Data Parameters
NAME OJ-7-41-H
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240602
Time 21.35 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C spectrum of **26a**



PROCNO 1

F2 - Acquisition Parameters

Date_ 20240602

Time 22.35 h

INSTRUM Avance Neo

PROBHD Z181819_0001 (

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 1024

DS 4

SWH 23809.524 Hz

FIDRES 0.726609 Hz

AQ 1.3762560 sec

RG 8

DW 21.000 usec

DE 6.50 usec

TE 300.1 K

D1 2.00000000 sec

D11 0.03000000 sec

TD0 1

SFO1 100.6228298 MHz

NUC1 13C

P0 3.33 usec

P1 10.00 usec

PLW1 56.91699982 W

SFO2 400.1316005 MHz

NUC2 1H

CPDPRG[2] waltz65

PCPD2 90.00 usec

PLW2 22.53199959 W

PLW12 0.27818000 W

PLW13 0.13992000 W

F2 - Processing parameters

SI 32768

SF 100.6127570 MHz

WDW EM

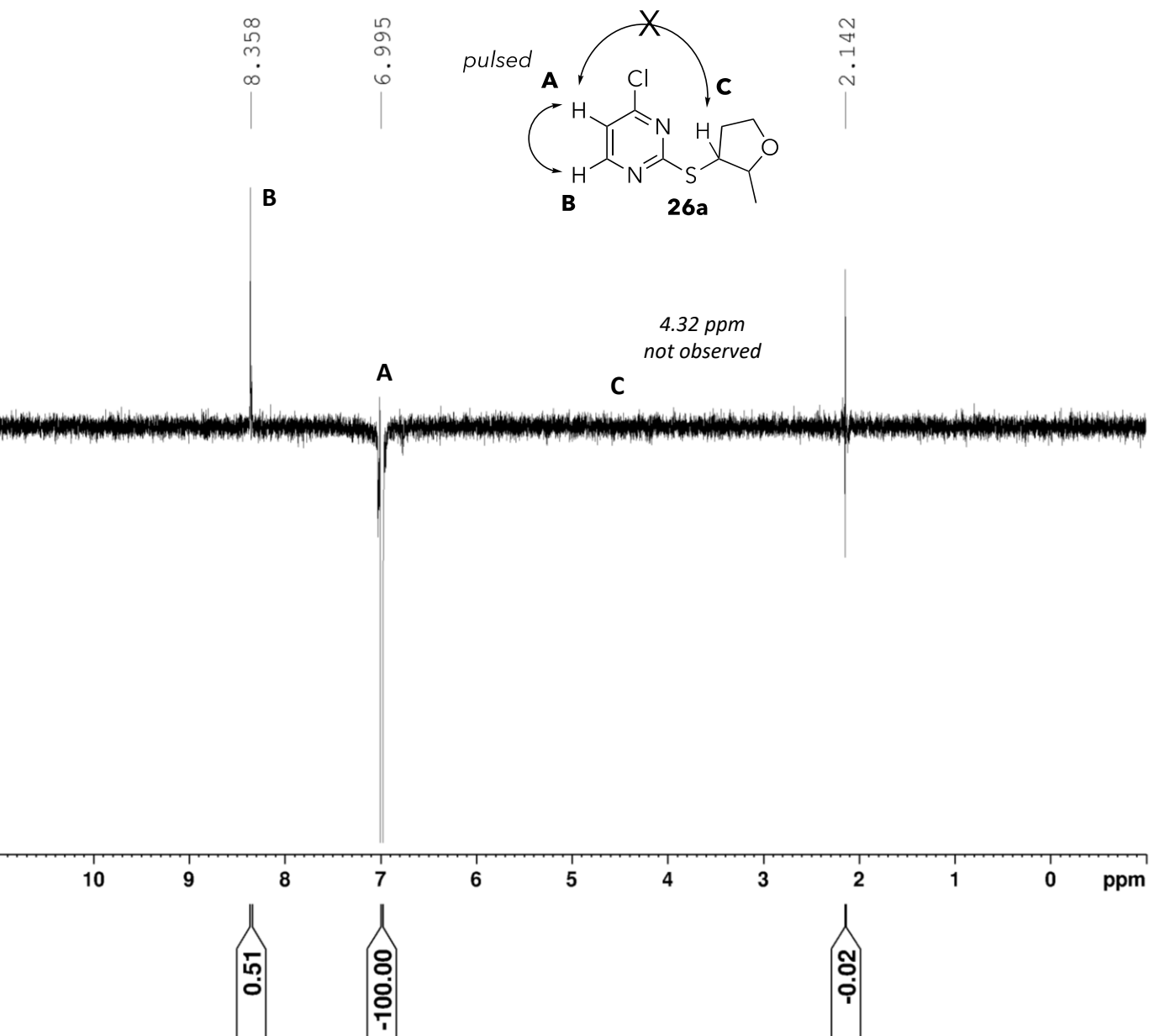
SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NOE of 26a



Current Data Parameters

NAME	OJ-7-41-NOE
EXPNO	20
PROCNO	1

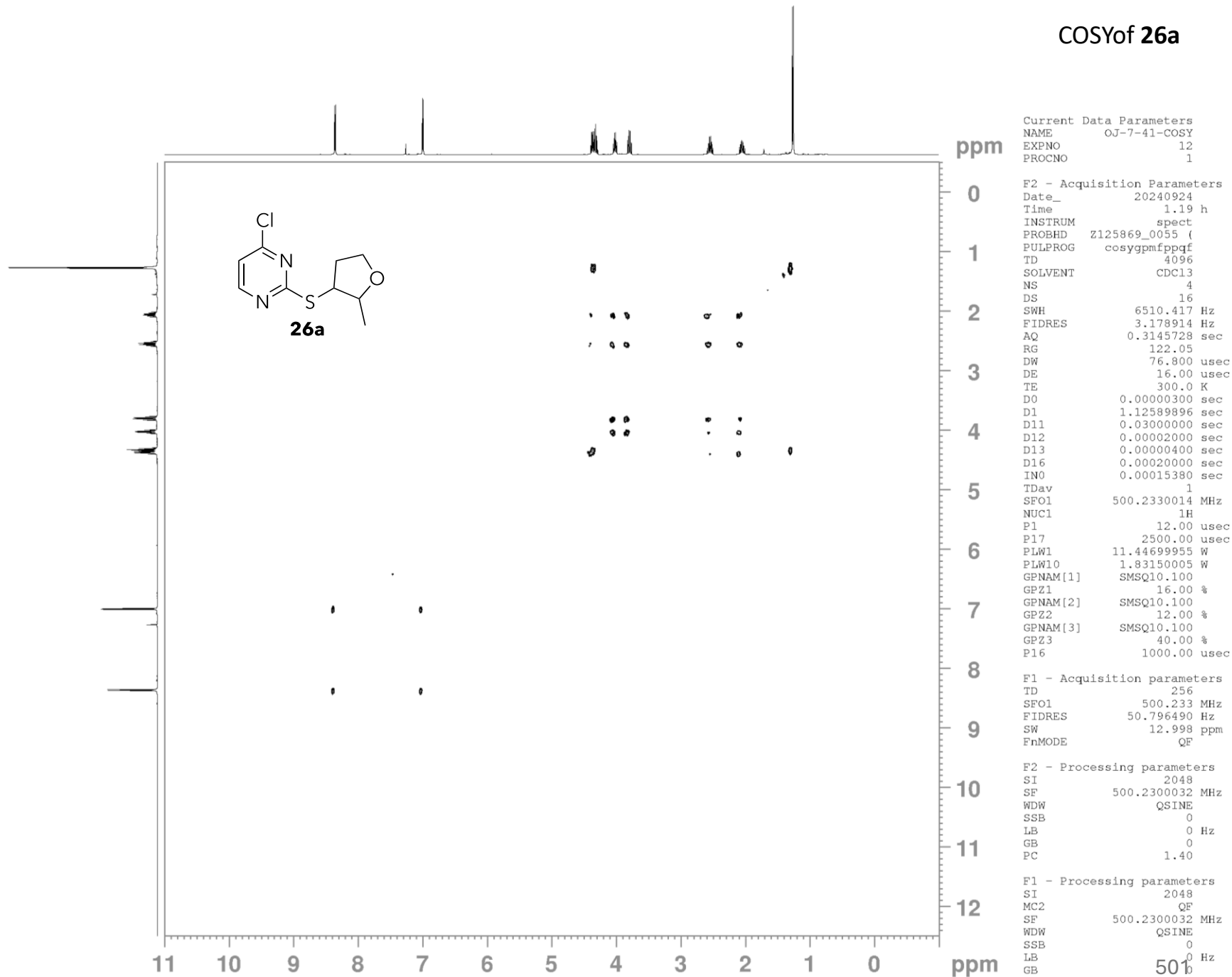
F2 - Acquisition Parameters

Date_	20231211
Time	20.29 h
INSTRUM	spect
PROBHD	Z125869_0055 (CPP BBO 500S1 BB-H&F-05
PULPROG	selnogpzs.2
TD	65536
SOLVENT	CDC13
NS	32
DS	4
SWH	10000.000 Hz
FIDRES	0.305176 Hz
AQ	3.2767999 sec
RG	190.44
DW	50.000 usec
DE	20.46 usec
TE	300.0 K
D1	2.00000000 sec
D8	0.30000001 sec
D16	0.00020000 sec
D20	0.27272600 sec
D31	0.30000001 sec
TD0	1
ZGOTINS	-DCALC_SPOFFS
SFO1	500.2330889 MHz
NUC1	1H
CNST21	7.0369768
P1	12.00 usec
P12	54025.93 usec
P32	20000.00 usec
P44	240.00 usec
PLW1	11.44699955 W
SPNAM[2]	Gaus1_180r.1000
SPOAL2	0.500
spoffs2	431.19 Hz
SPW2	0.00001334 W
SPNAM[29]	Crp60,20,20.10
SPOAL29	0.500
spoffs29	0 Hz
SPW29	0.06296300 W
SPNAM[30]	Bip720,50,20.1
SPOAL30	0.500
spoffs30	0 Hz
SPW30	1.83150005 W
GPZ0	11.00 %
GPNAM[1]	SMSQ10.100
GPZ1	15.00 %
GPNAM[2]	SMSQ10.100
GPZ2	40.00 %
P16	1000.00 usec

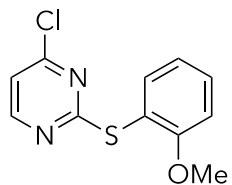
F2 - Processing parameters

SI	32768
SF	500.2300249 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

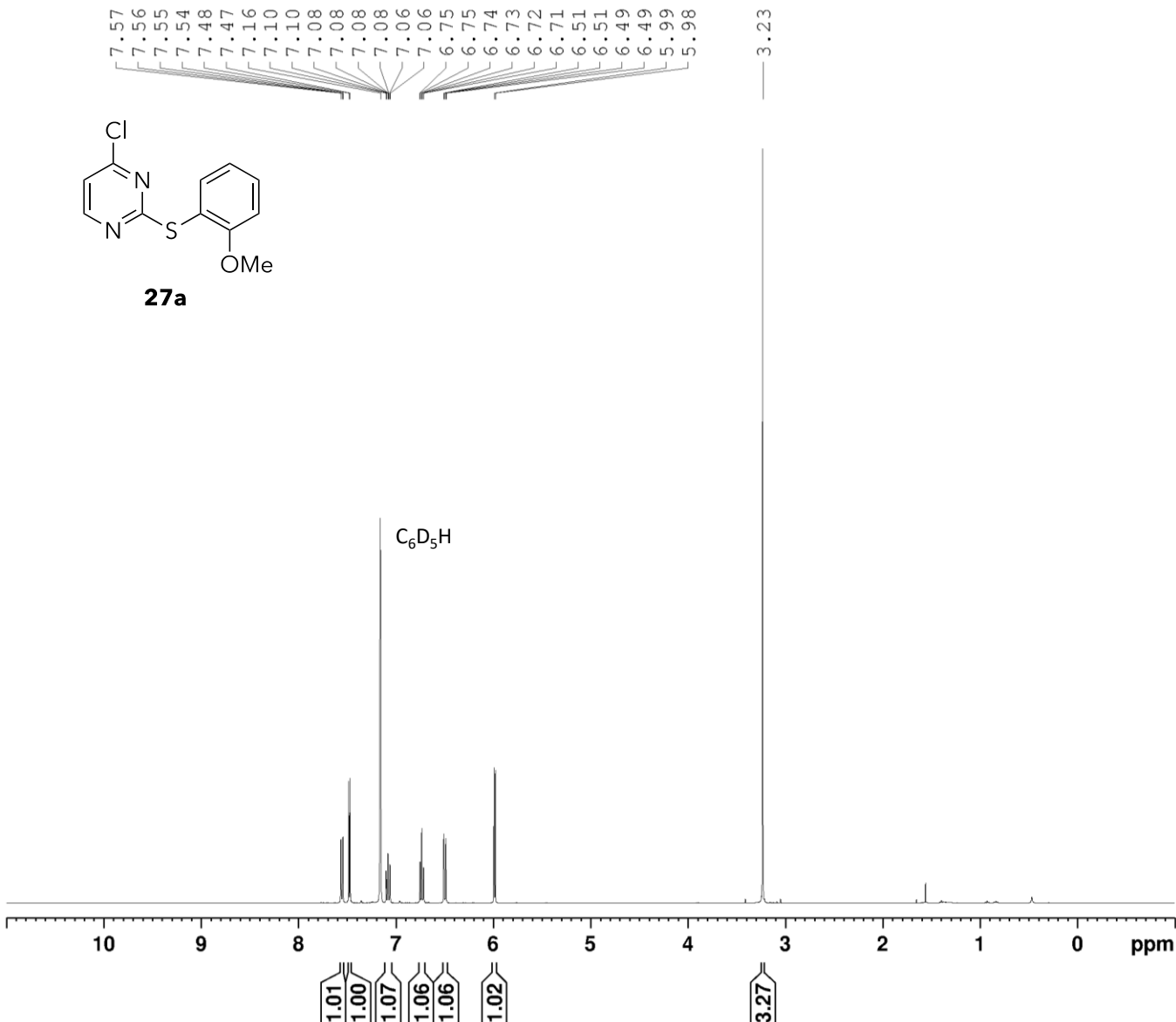
COSYof 26a



¹H spectrum of **27a**



27a

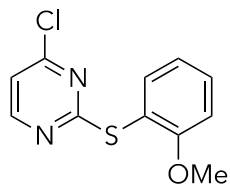


Current Data Parameters
NAME 27a_AR_03_029
EXPNO 11
PROCNO 1

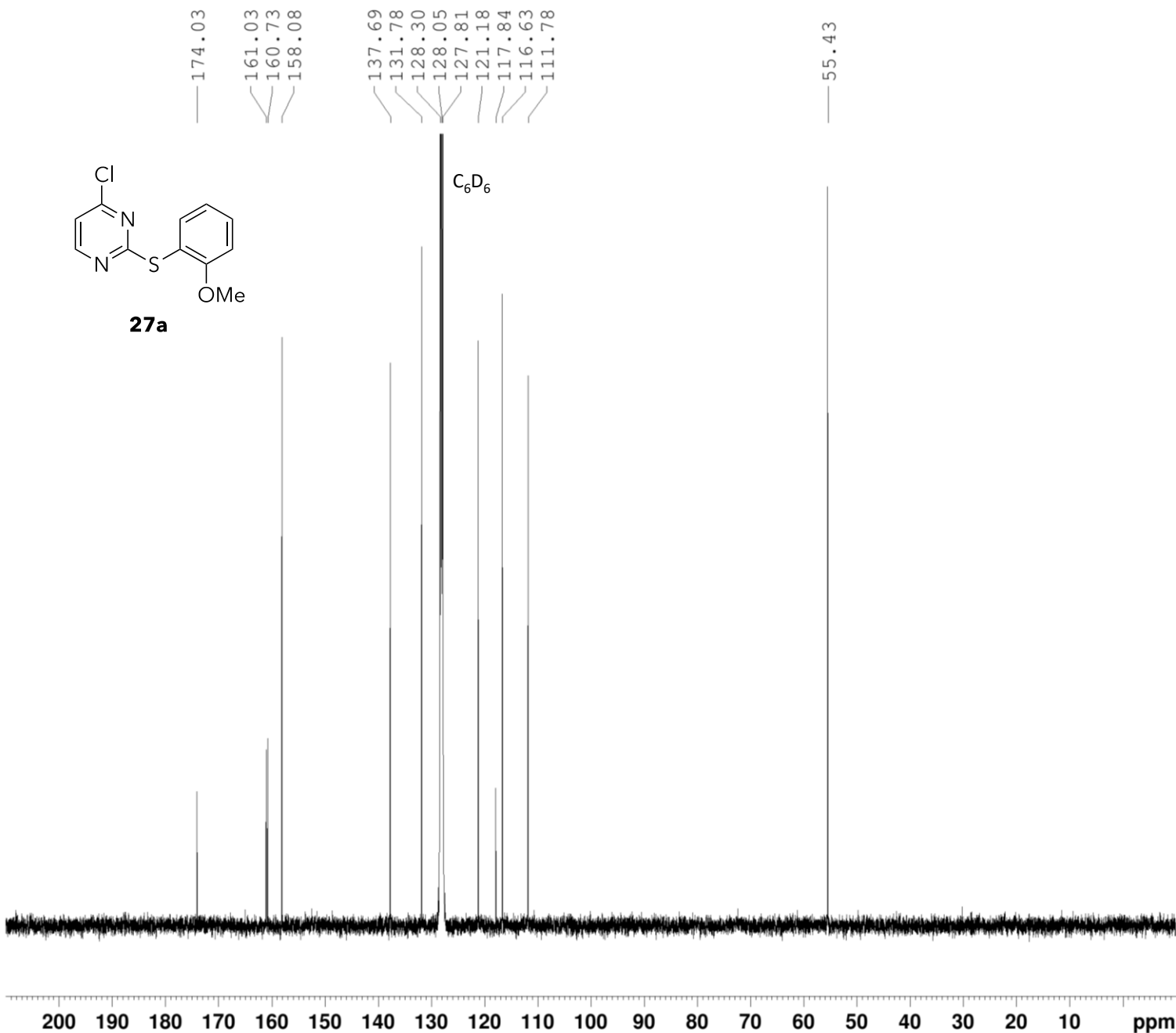
F2 - Acquisition Parameters
Date_ 20240329
Time 16.17 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P...
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300287 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **27a**



27a



DATE_ 20240329
Time_ 23.10 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

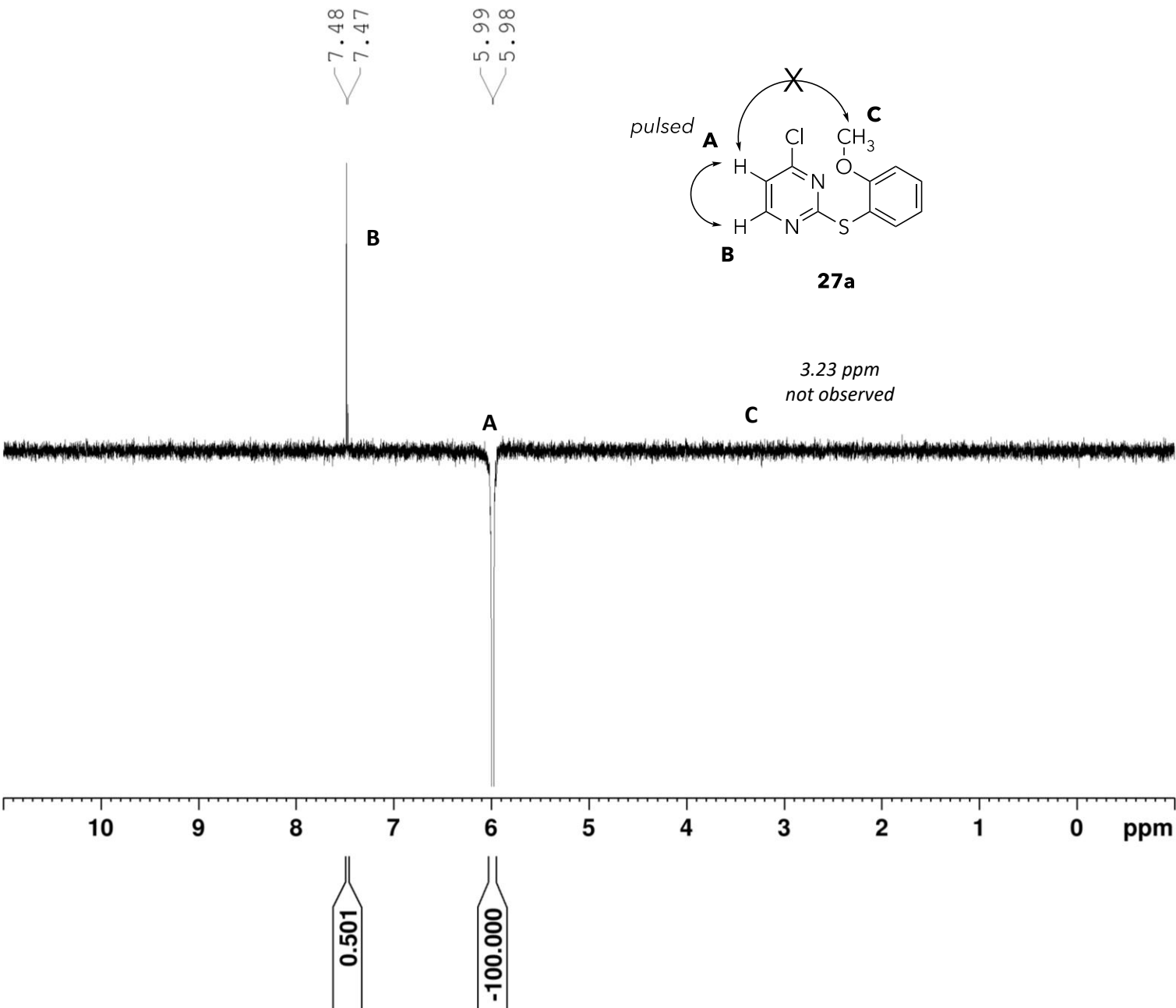
F2 - Processing parameters
SI 32768
SF 100.6127391 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 27a

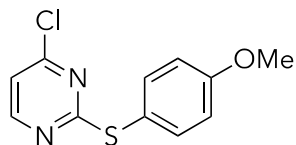
Current Data Parameters
 NAME 27a_AR_03_029
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240329
 Time 16.27 h
 INSTRUM Avance Neo
 PROBHD Z181819_0001 (
 PULPROG selnpgpzs.2
 TD 65536
 SOLVENT CDC13
 NS 32
 DS 4
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 8.88 usec
 TE 300.2 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27266601 sec
 D31 0.30000001 sec
 TD0 1
 ZGOTNS -DCALC_SPOFFS
 SFO1 400.1324708 MHz
 NUC1 1H
 CNST21 6.0634618
 P1 10.00 usec
 P12 55499.87 usec
 P32 20000.00 usec
 P44 300.00 usec
 PLW1 22.53199959 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 SPW2 0.00001727 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 SPW29 0.08606600 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 SPW30 1.60230005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

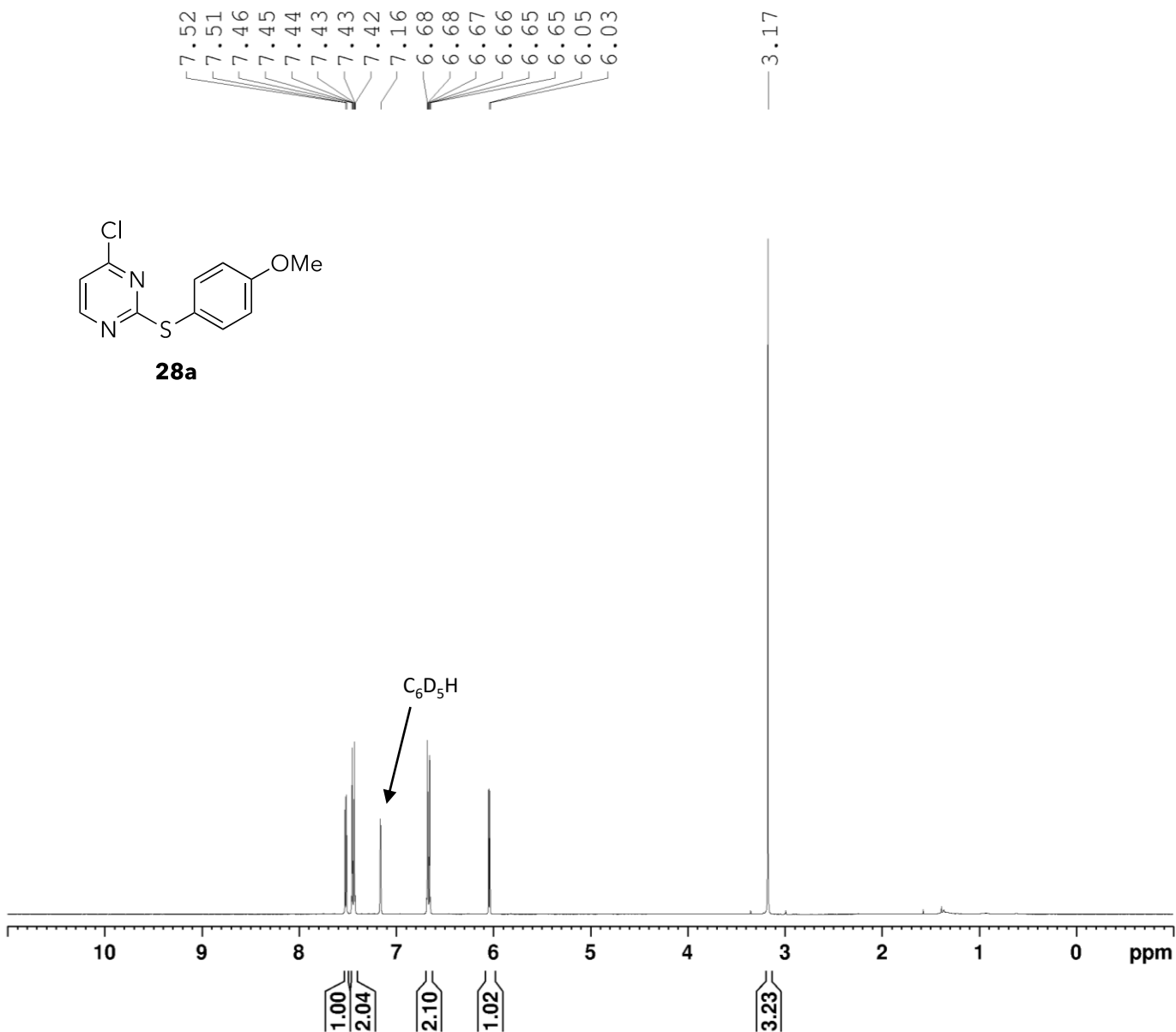
F2 - Processing parameters
 SI 32768
 SF 400.1300287 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



¹H spectrum of 28a



28a

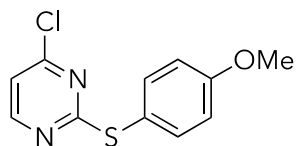


Current Data Parameters
NAME 28a_AR_03_028-benzene-new
EXPNO 17
PROCNO 1

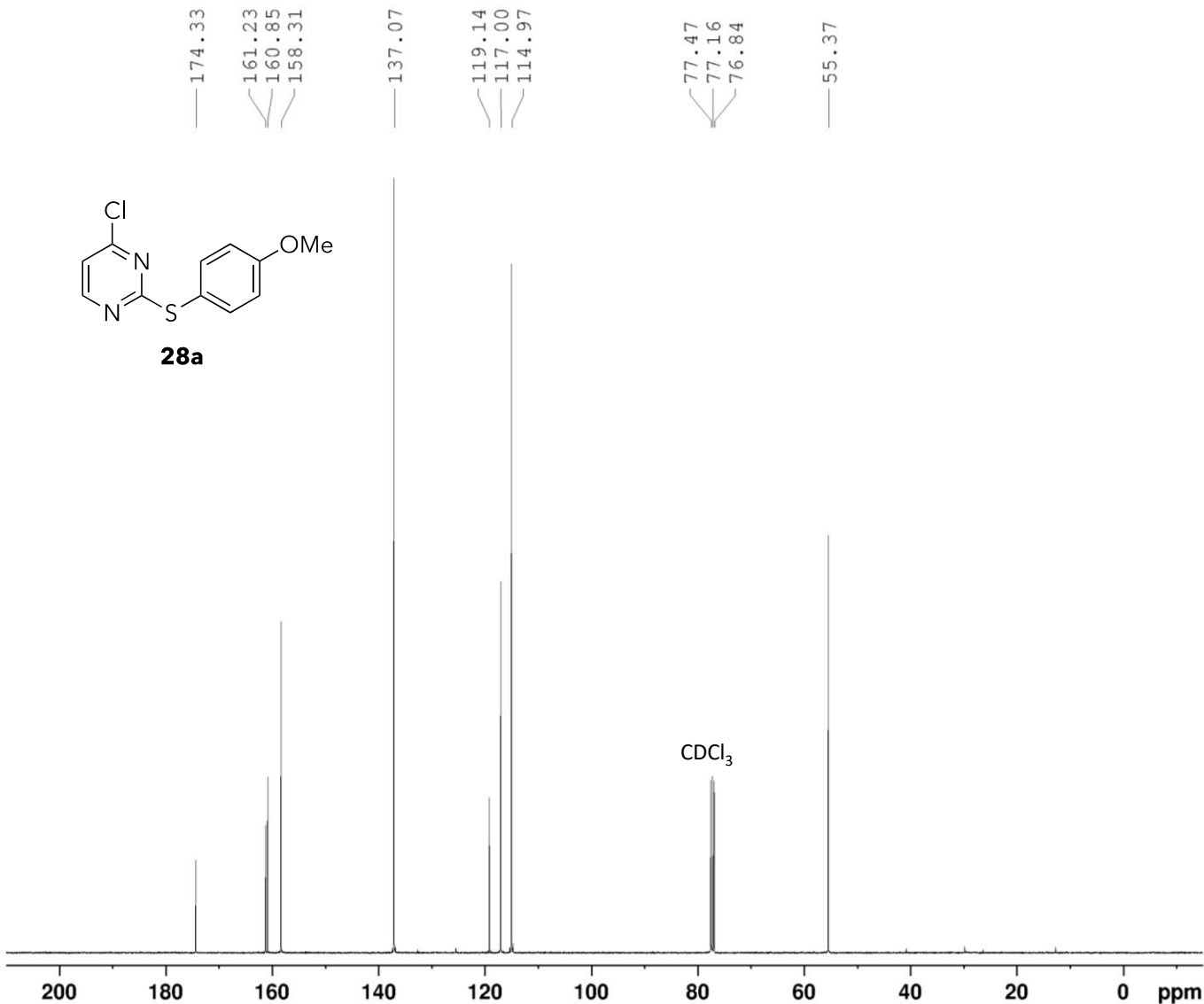
F2 - Acquisition Parameters
Date_ 20241127
Time 19.22 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SF01 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1299967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **28a**



28a

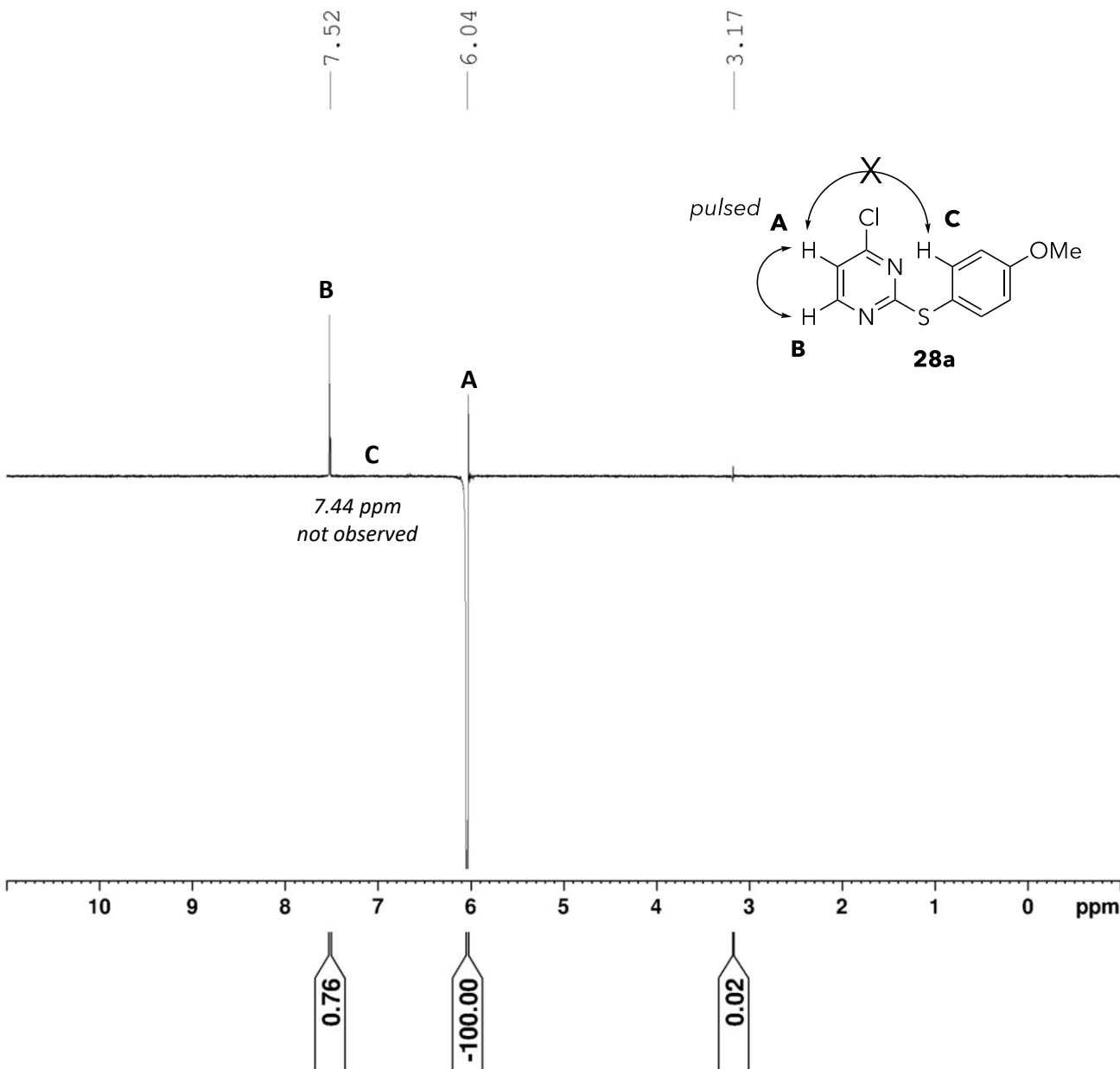


Current Data Parameters
NAME 28a_AR_03_028
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240122
Time 21.12 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127682 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 28a

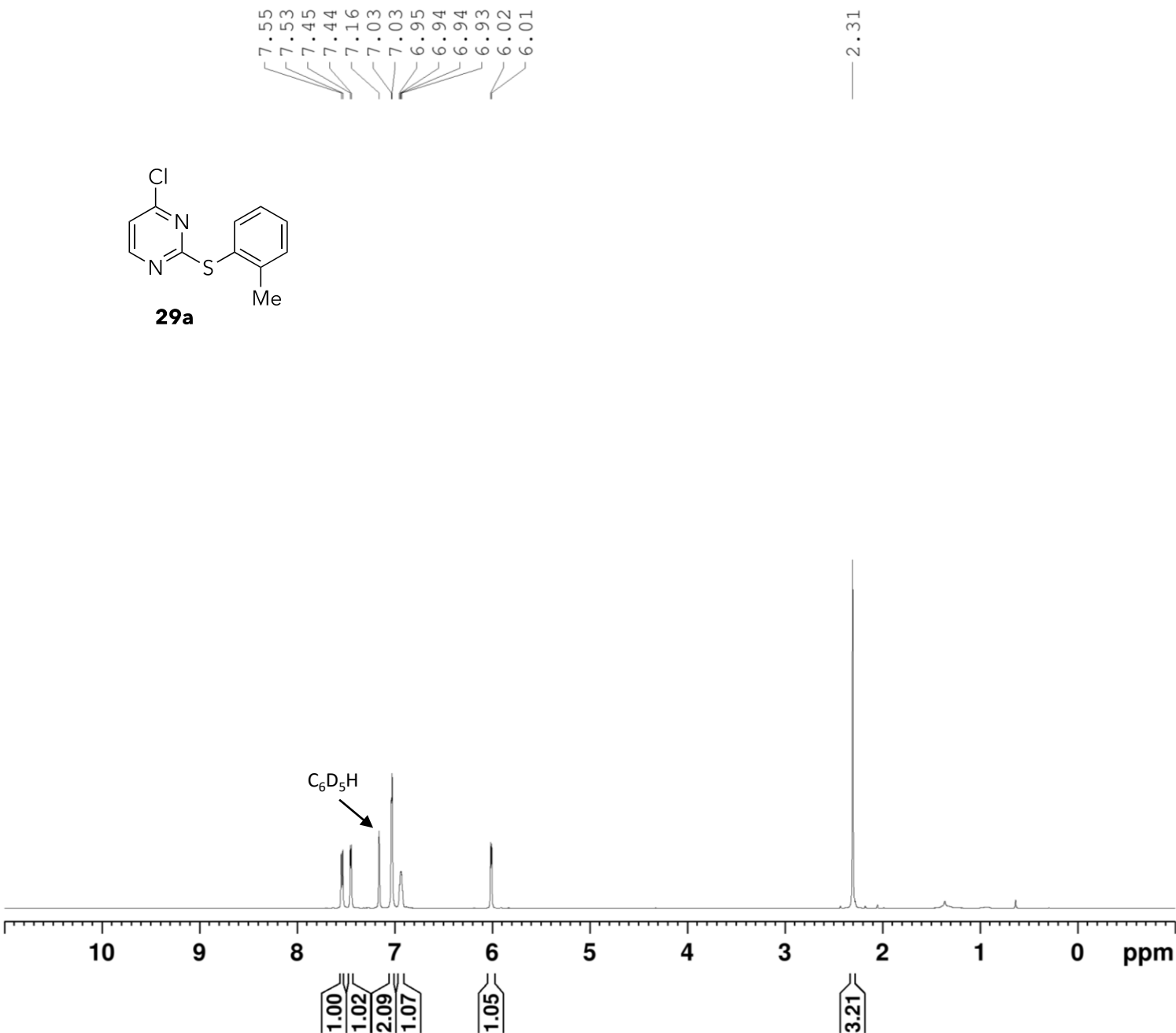
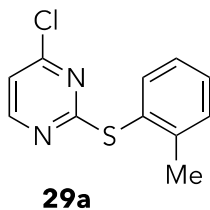


Current Data Parameters
NAME 28a_AR_03_028-benzene-new
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241127
Time 19.27 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5
PULPROG selnpgpzs.2
TD 65536
SOLVENT C6D6
NS 32
DS 4
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 8.88 usec
TE 300.2 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27266601 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SF01 400.1324708 MHz
NUC1 1H
CNST21 6.0300288
P1 10.00 usec
P12 68983.51 usec
P32 20000.00 usec
P44 300.00 usec
PLW1 22.53199959 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
SPW2 0.00001118 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
SPW29 0.08606600 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
SPW30 1.60230005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 400.1299964 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H spectrum of **29a**

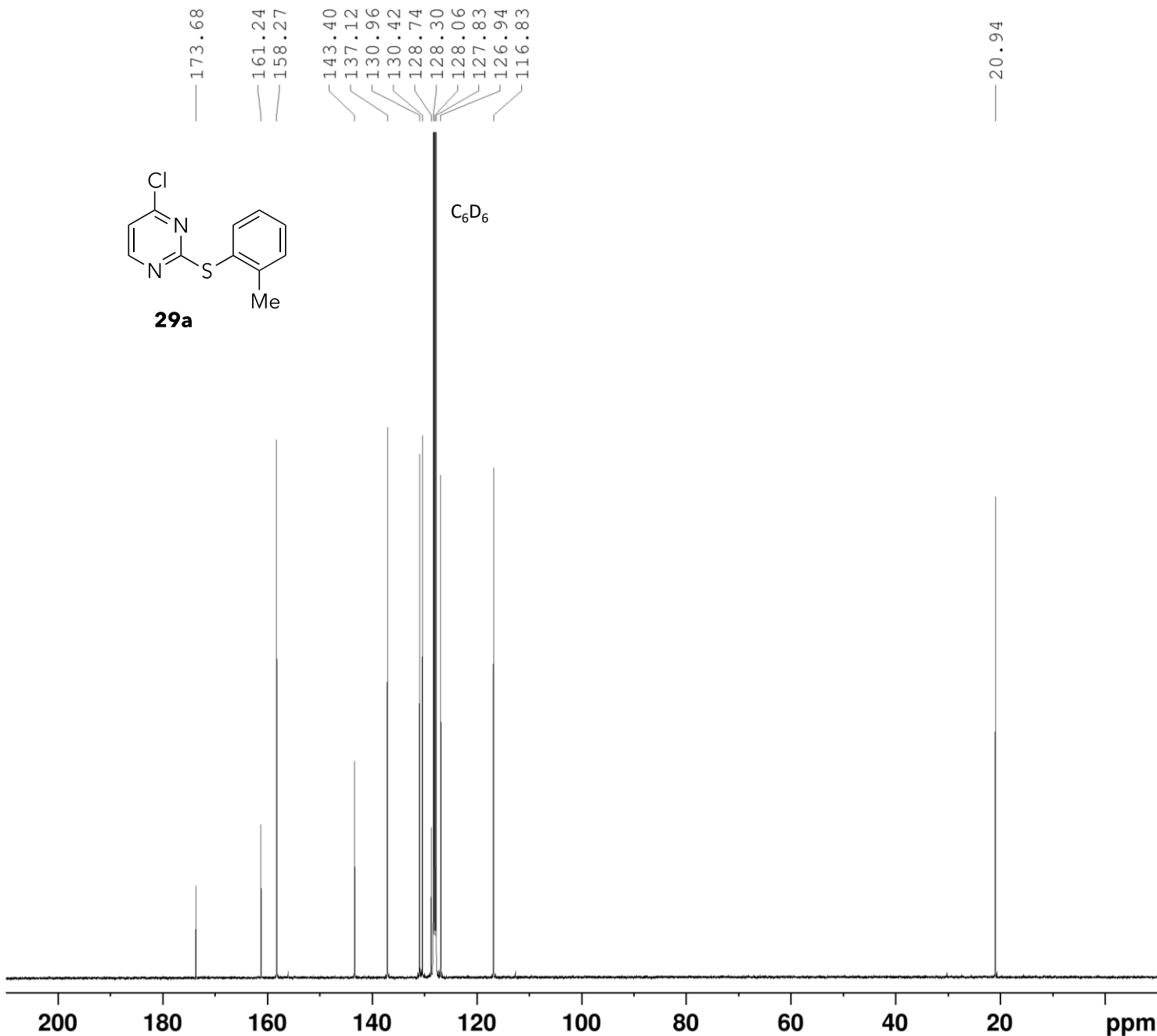
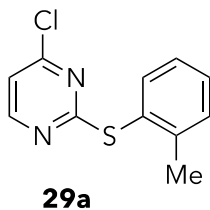


Current Data Parameters
NAME AR_03_030_ahv_p_benzene
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230908
Time 10.29 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 37.93
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2299954 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 29a



Current Data Parameters
NAME 29a_AR_03_30
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231130
Time 0.08 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

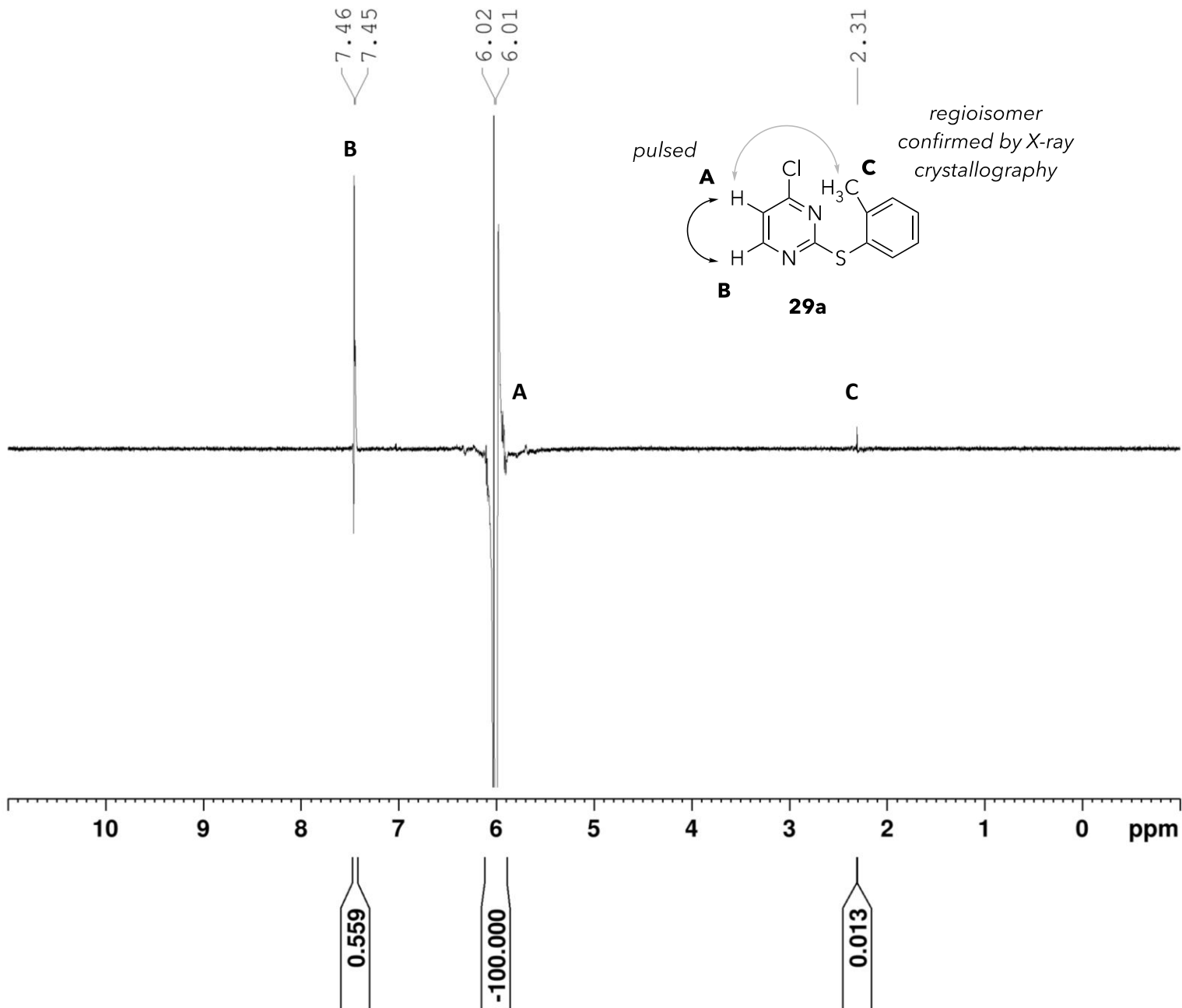
F2 - Processing parameters
SI 32768
SF 100.6127317 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 29a

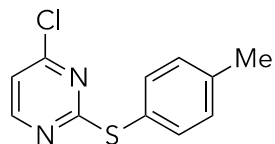
Current Data Parameters
 NAME 29a_AR_03_30
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230908
 Time 10.56 h
 INSTRUM spect
 PROBHD Z125869_0055 (
 PULPROG selnogpzs.2
 TD 65536
 SOLVENT C6D6
 NS 32
 DS 4
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767999 sec
 RG 190.44
 DW 50.000 usec
 DE 20.46 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27272600 sec
 D31 0.30000001 sec
 TD0 1
 ZGPGTNS -DCALC_SPOFFS
 SF01 500.2330889 MHz
 NUC1 1H
 CNST21 6.0071168
 P1 12.00 usec
 P12 29285.79 usec
 P32 20000.00 usec
 P44 240.00 usec
 PLW1 11.44699955 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 spoofs2 -83.98 Hz
 SPW2 0.00004538 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 spoofs29 0 Hz
 SPW29 0.06296300 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 spoofs30 0 Hz
 SPW30 1.83150005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

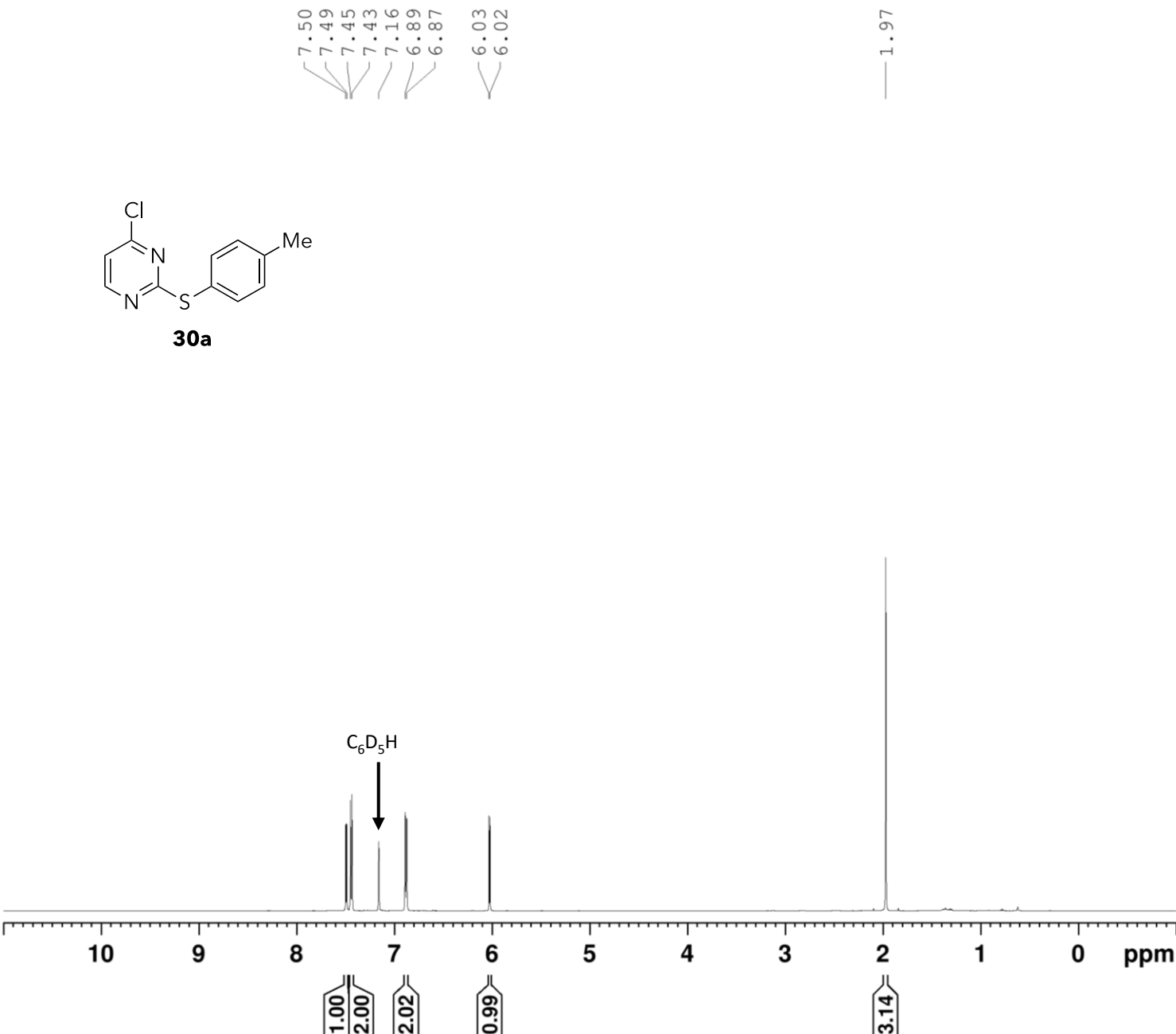
F2 - Processing parameters
 SI 32768
 SF 500.2299941 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



¹H spectrum of **30a**



30a

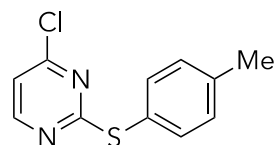


Current Data Parameters
NAME AR_03_031_p
EXPNO 1
PROCNO 1

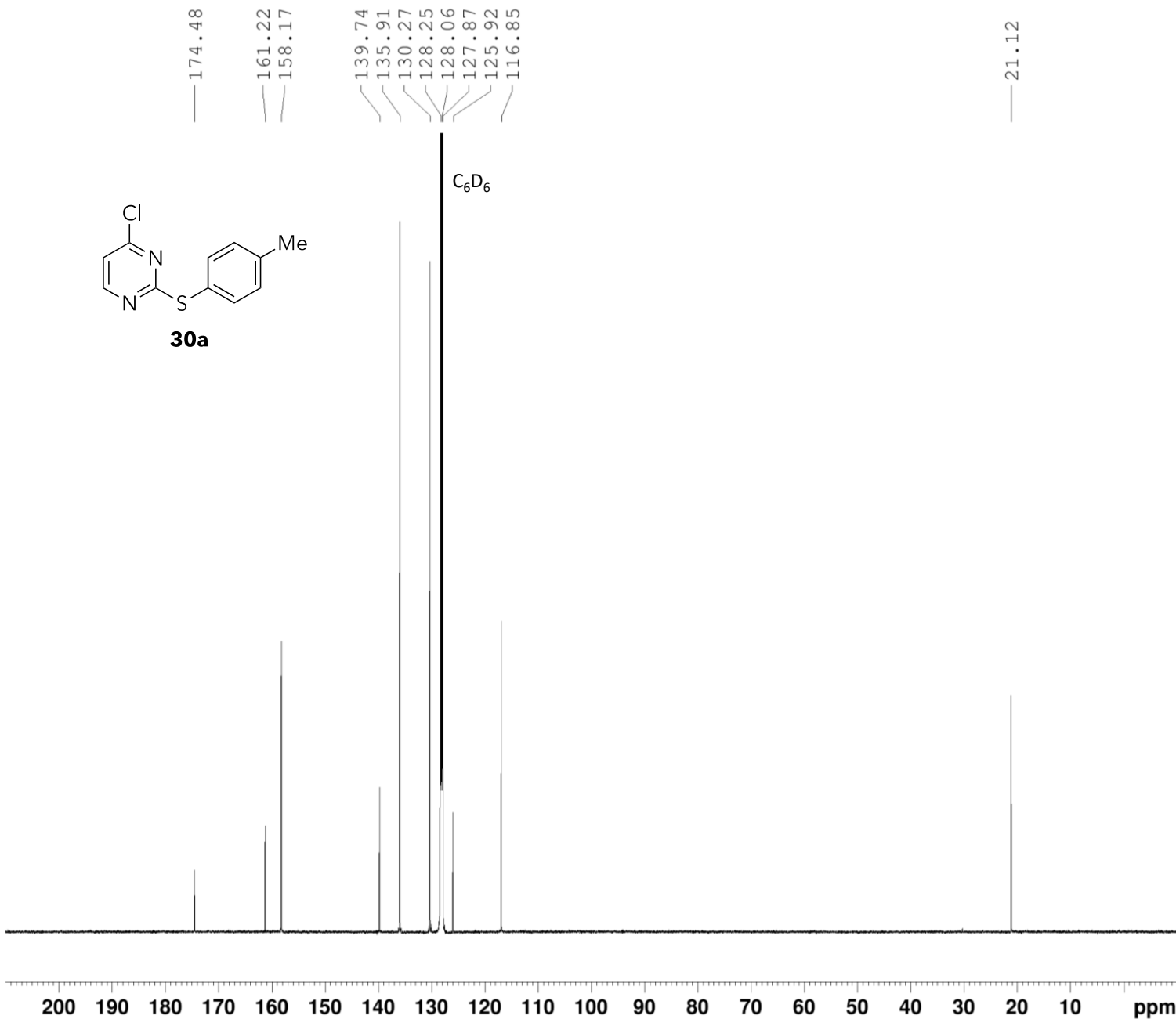
F2 - Acquisition Parameters
Date_ 20230828
Time 9.29 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 43.72
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2299957 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **30a**



30a



Current Data Parameters
NAME 30a_AR_03_031
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240123
Time 17.10 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

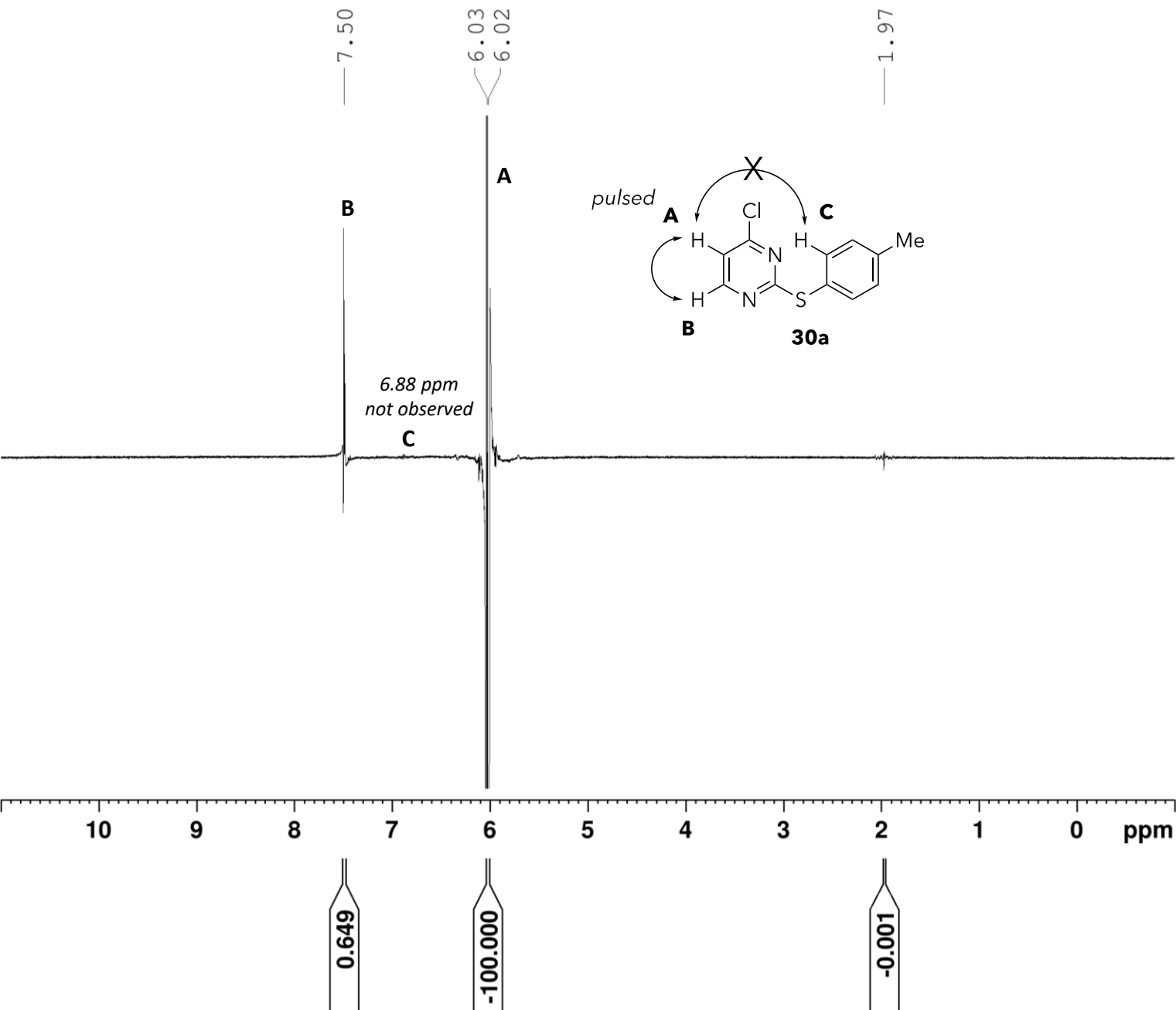
F2 - Processing parameters
SI 32768
SF 125.7828873 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 30a

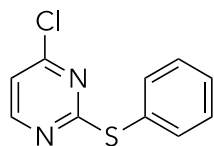
Current Data Parameters
NAME 30a_AR_03_031
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230828
Time 9.40 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT C6D6
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 6.0188208
P1 12.00 usec
P12 71523.89 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -78.13 Hz
SPW2 0.00000761 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

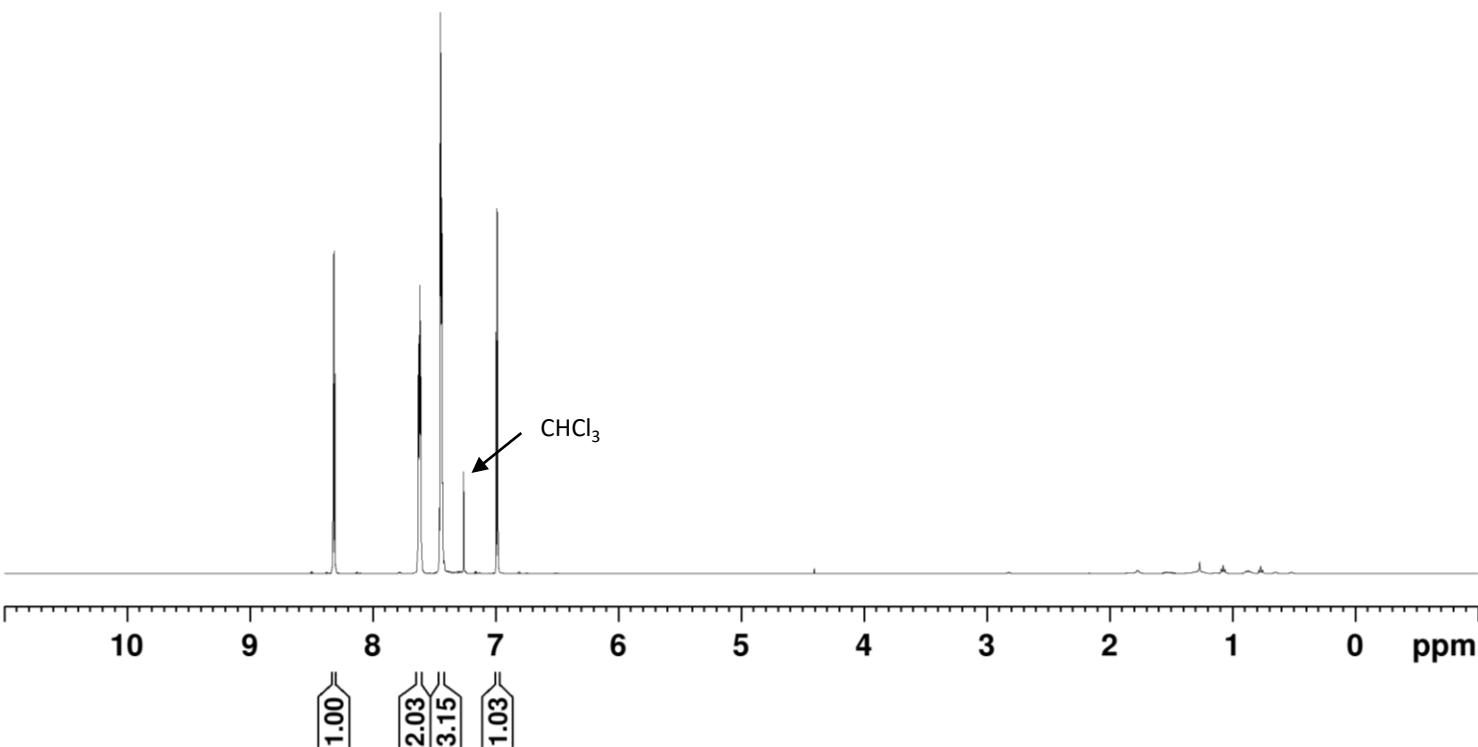
F2 - Processing parameters
SI 32768
SF 500.2299957 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **31a**



31a

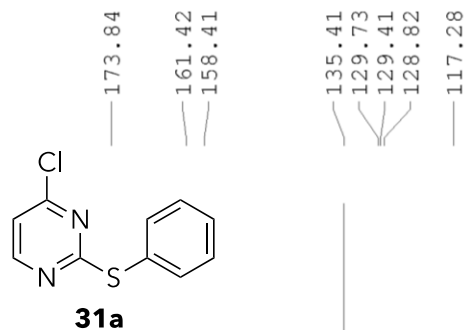


Current Data Parameters
NAME AR_03_026_4_11024
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240110
Time 19.25 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 107.7
DW 50.000 usec
DE 16.00 usec
TE 310.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **31a**



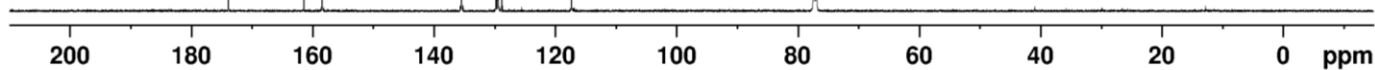
77.41
77.16
76.90

CDCl₃

Current Data Parameters
NAME 31a_AR_03_026
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240110
Time_ 20.22 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 310.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters
SI 32768
SF 125.7829154 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

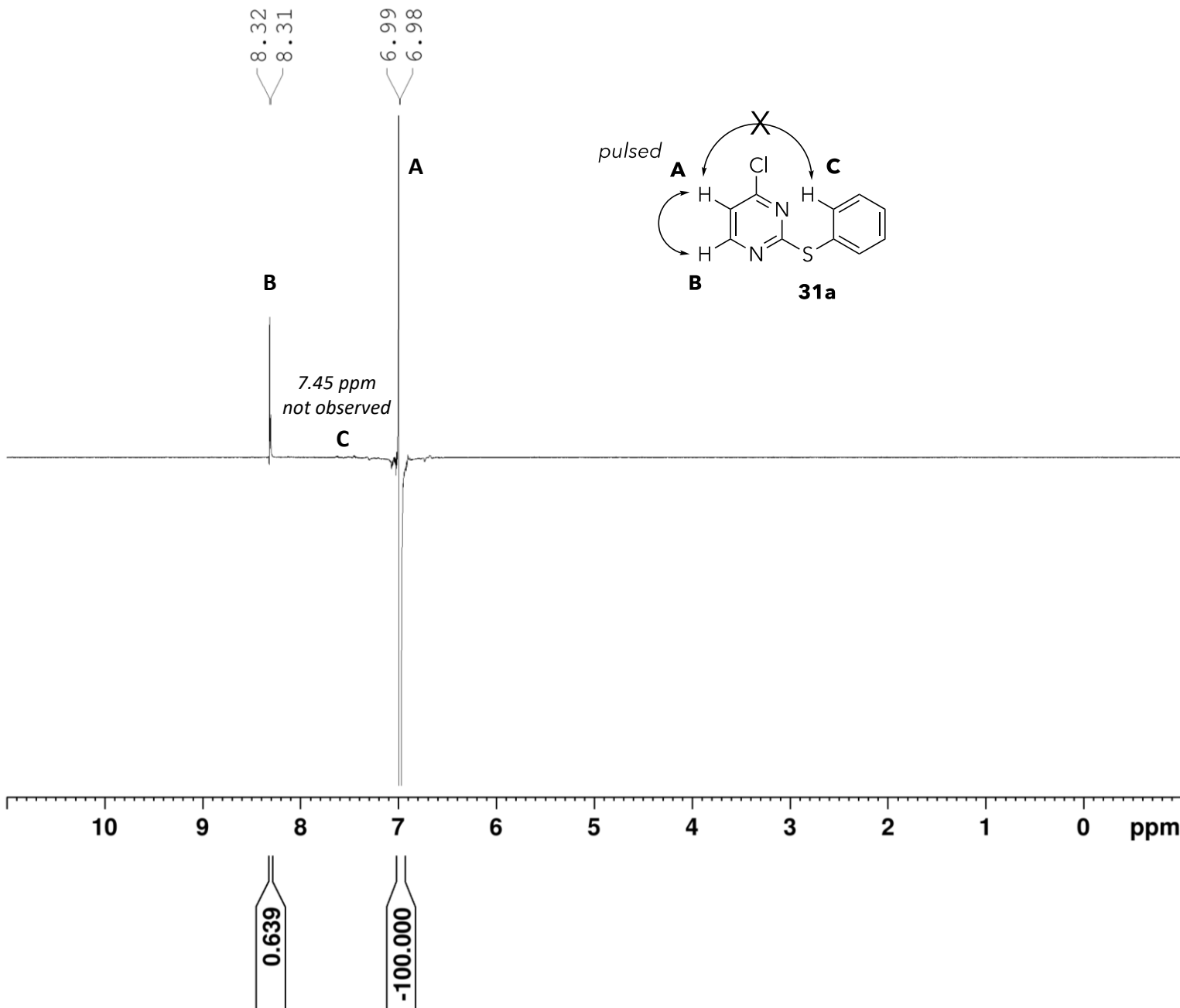


1D NOE of **31a**

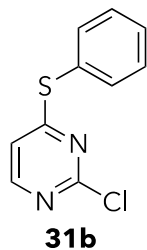
Current Data Parameters
NAME 31a_AR_03_026
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230905
Time 11.43 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0050302
P1 12.00 usec
P12 36662.88 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 415.21 Hz
SPW2 0.00002896 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

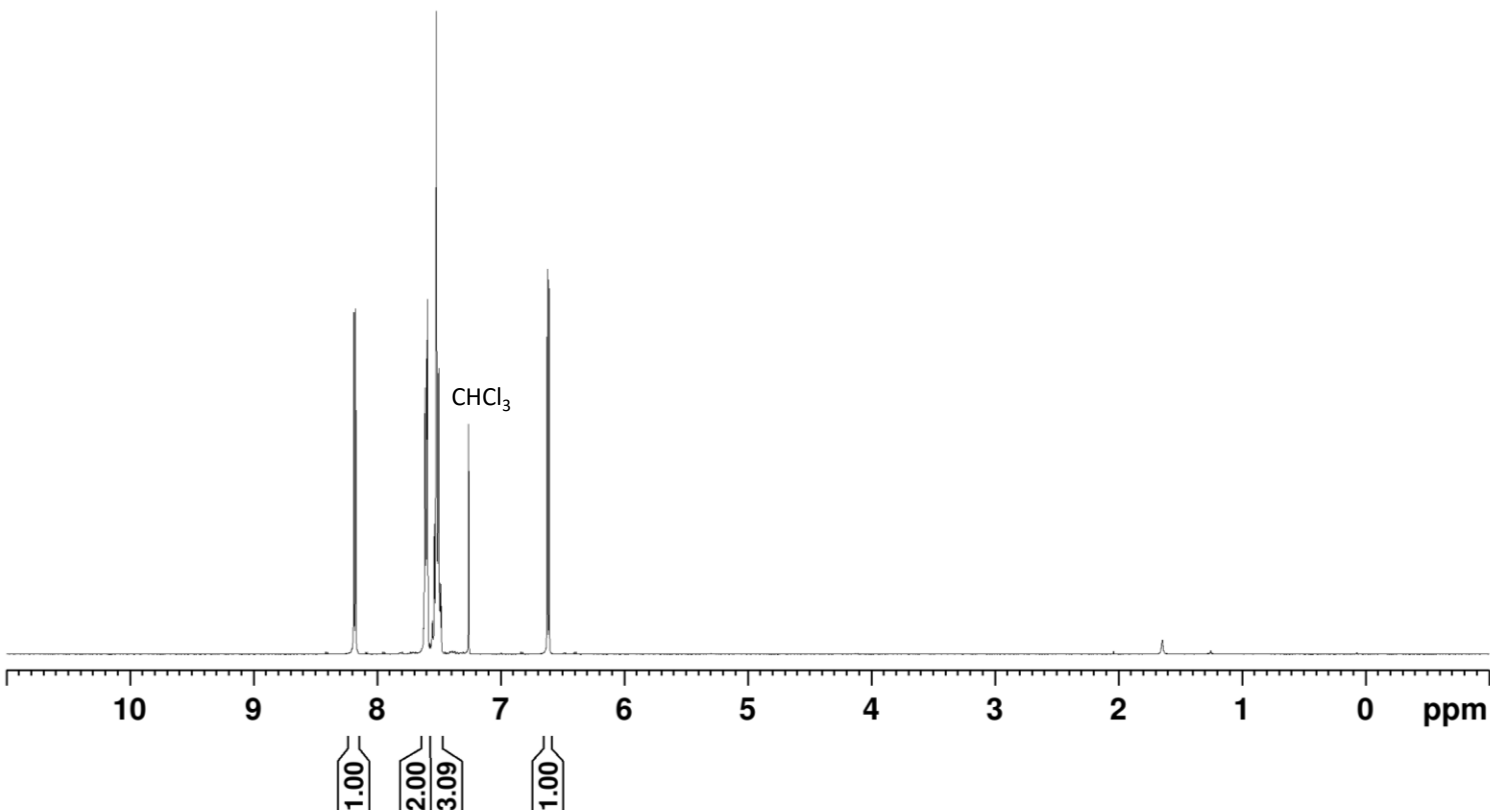
F2 - Processing parameters
SI 32768
SF 500.2300119 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **31b**



8.19
8.17
7.62
7.61
7.60
7.60
7.59
7.54
7.53
7.52
7.52
7.51
7.50
7.26
6.62
6.61



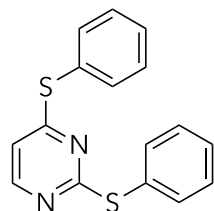
Current Data Parameters
NAME S31b_AR_03_126
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240508
Time 8.56 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

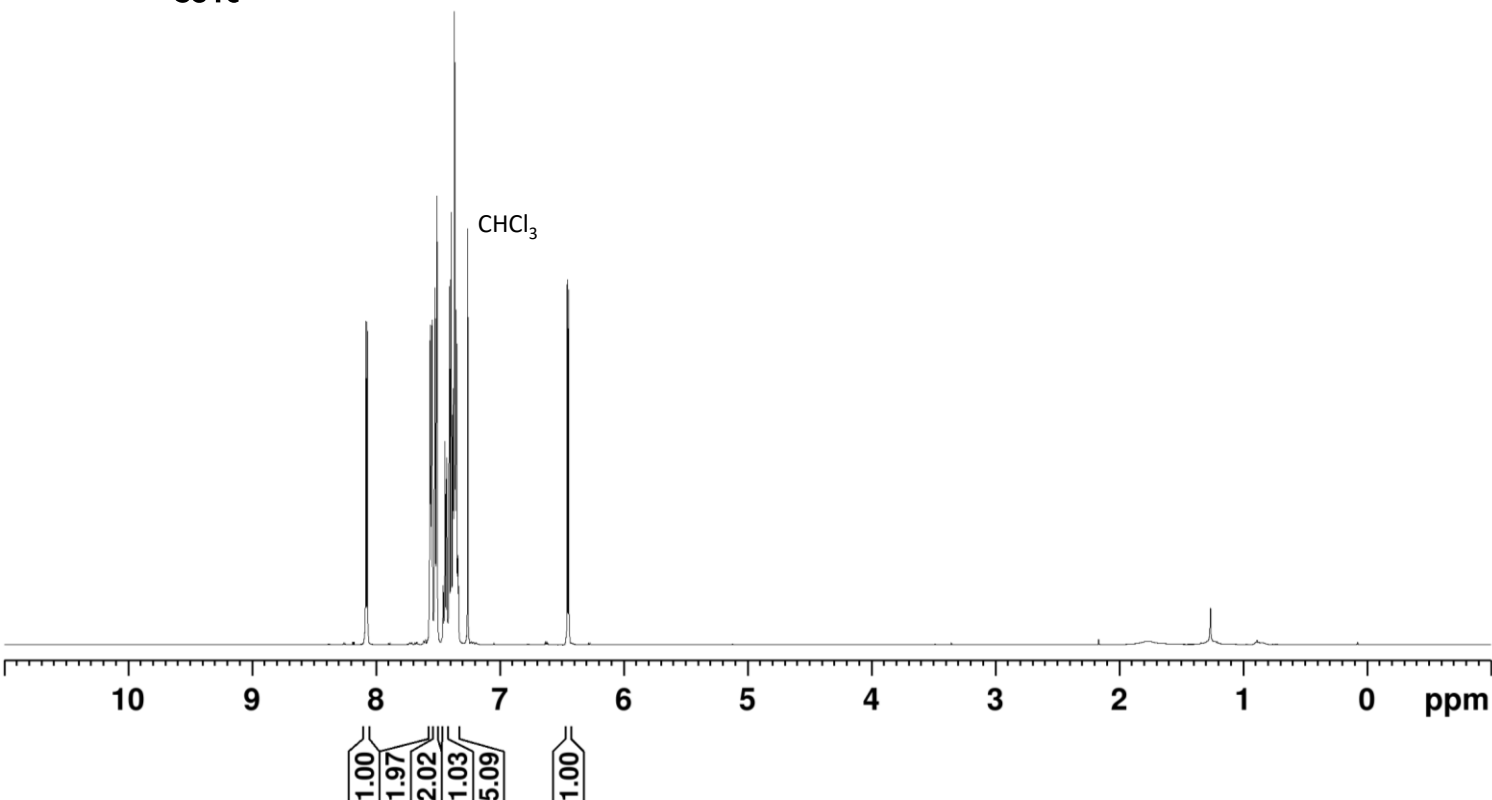
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H spectrum of **S31c**

8.08
8.07
7.57
7.56
7.55
7.55
7.53
7.52
7.51
7.51
7.46
7.44
7.43
7.41
7.39
7.38
7.37
7.35
7.34
7.34
7.33
7.26
6.46
6.45



S31c

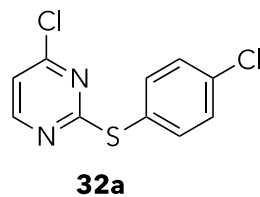


Current Data Parameters
NAME S31c_AR_03_065
EXPNO 10
PROCNO 1

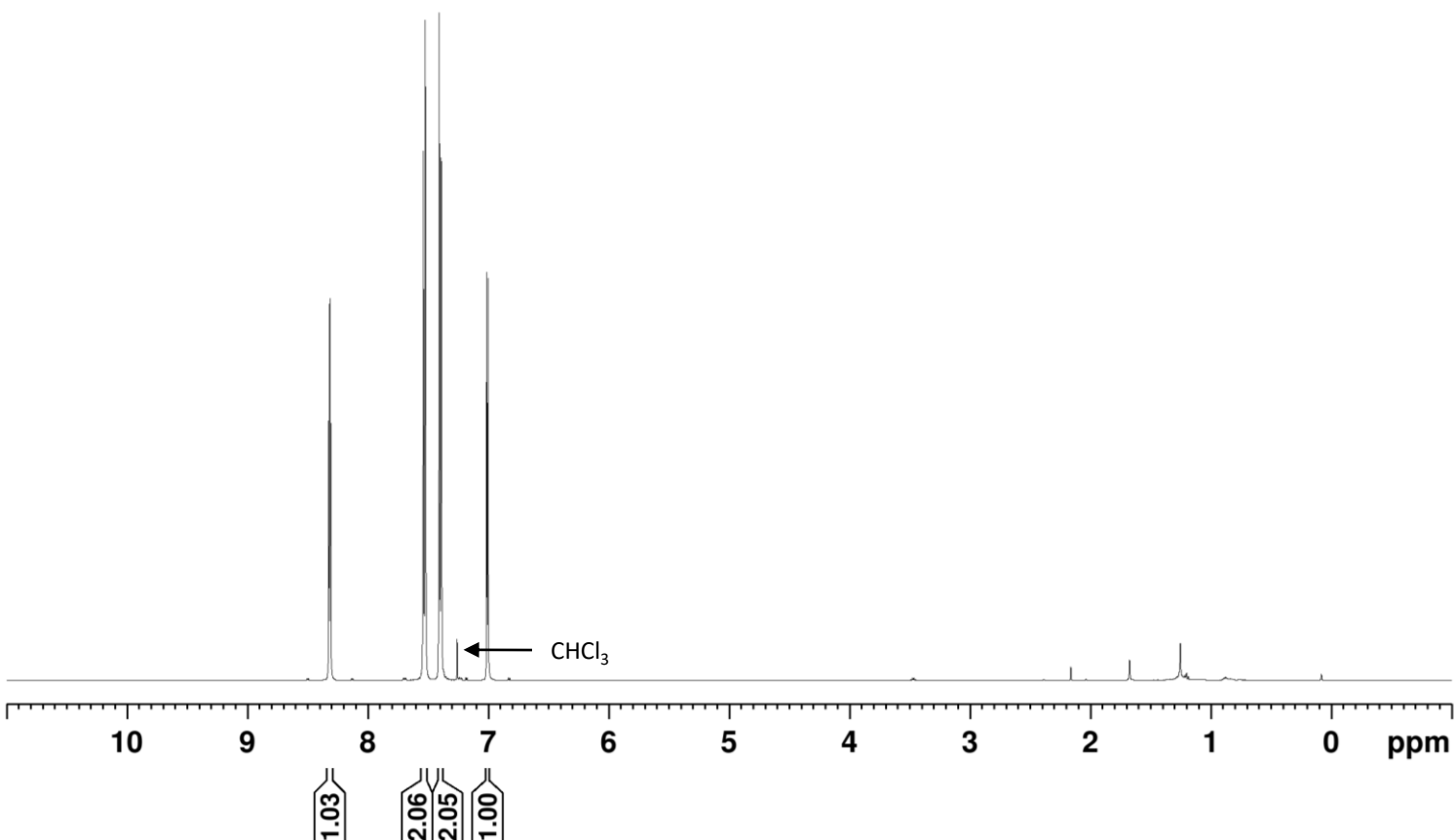
F2 - Acquisition Parameters
Date_ 20240110
Time 17.08 h
INSTRUM spect
PROBHD Z125869_0055 (zg30)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 16.00 usec
TE 310.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H spectrum of **32a**



8.32
8.31
7.54
7.52
7.41
7.39
7.26
7.01
7.00

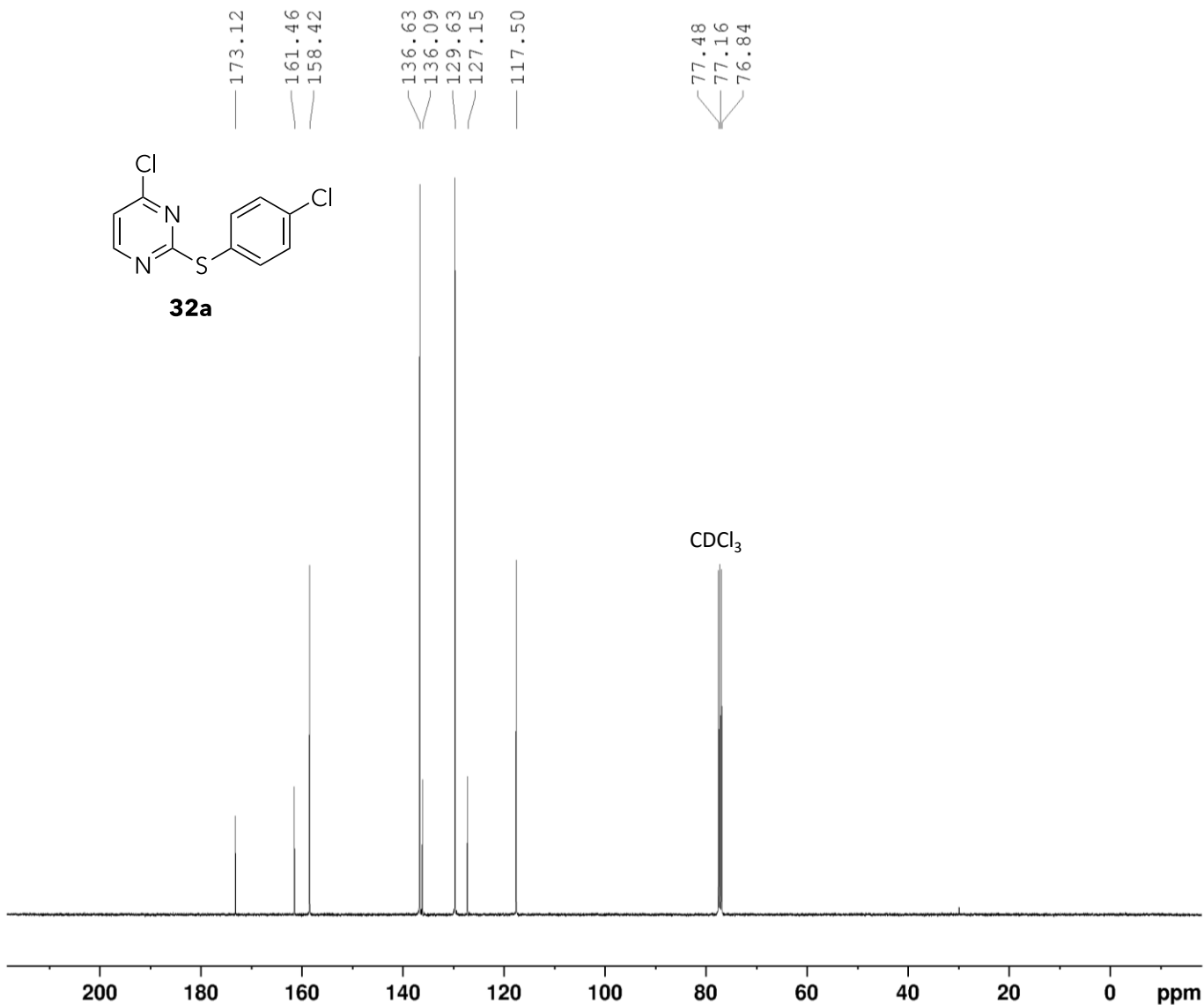
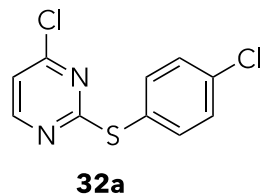


Current Data Parameters
NAME ar_03_35_n
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240304
Time 16.27 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 35.18
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **32a**



Current Data Parameters
NAME 32a_AR_03_035
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231130
Time 0.11 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (PI HR-BBO400S1-BBI
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

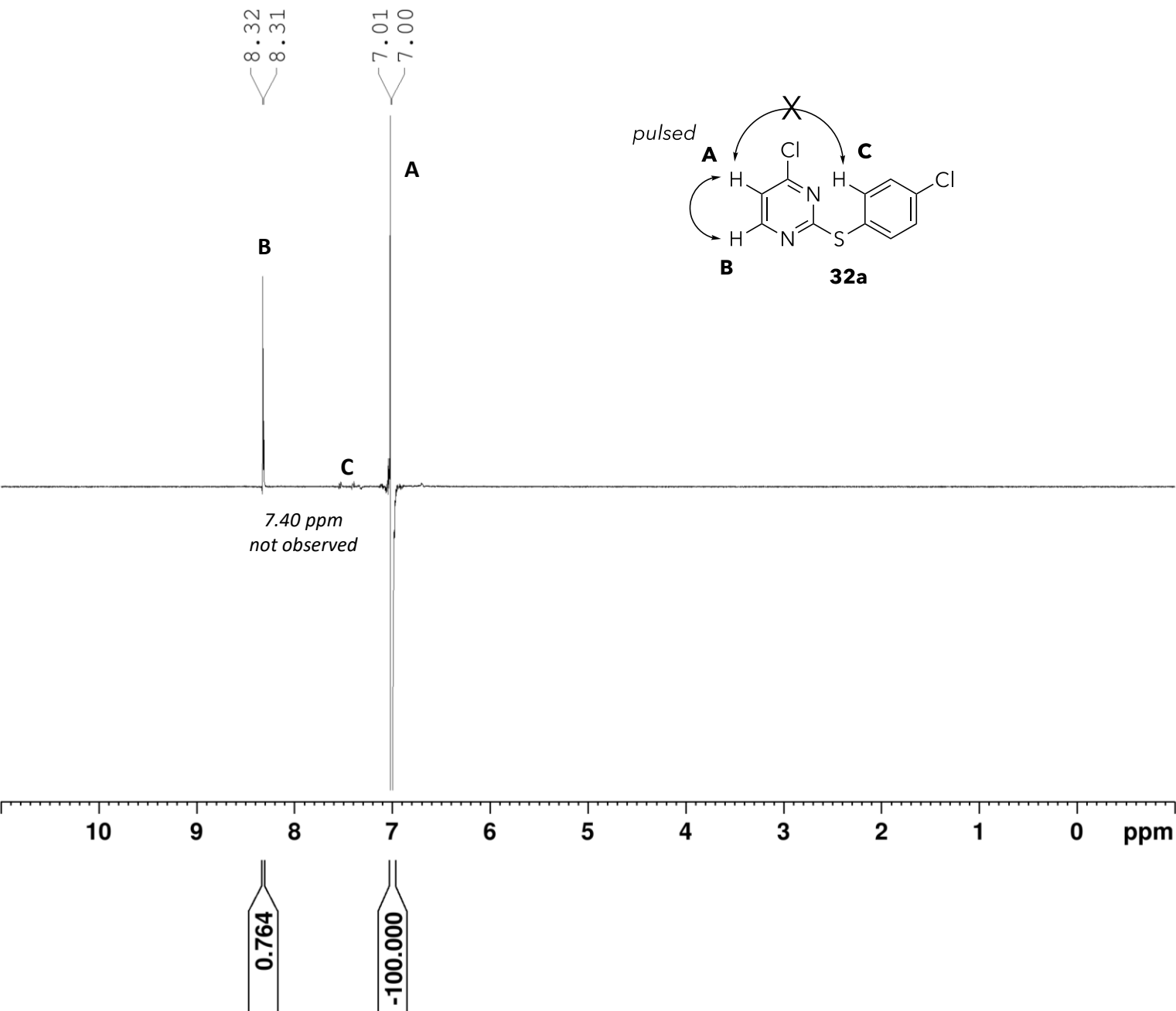
F2 - Processing parameters
SI 32768
SF 100.6127617 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

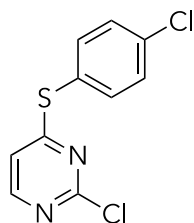
1D NOE of **32a**

Current Data Parameters
NAME 32a_AR_03_035
EXPNO 5
PROCNO 1

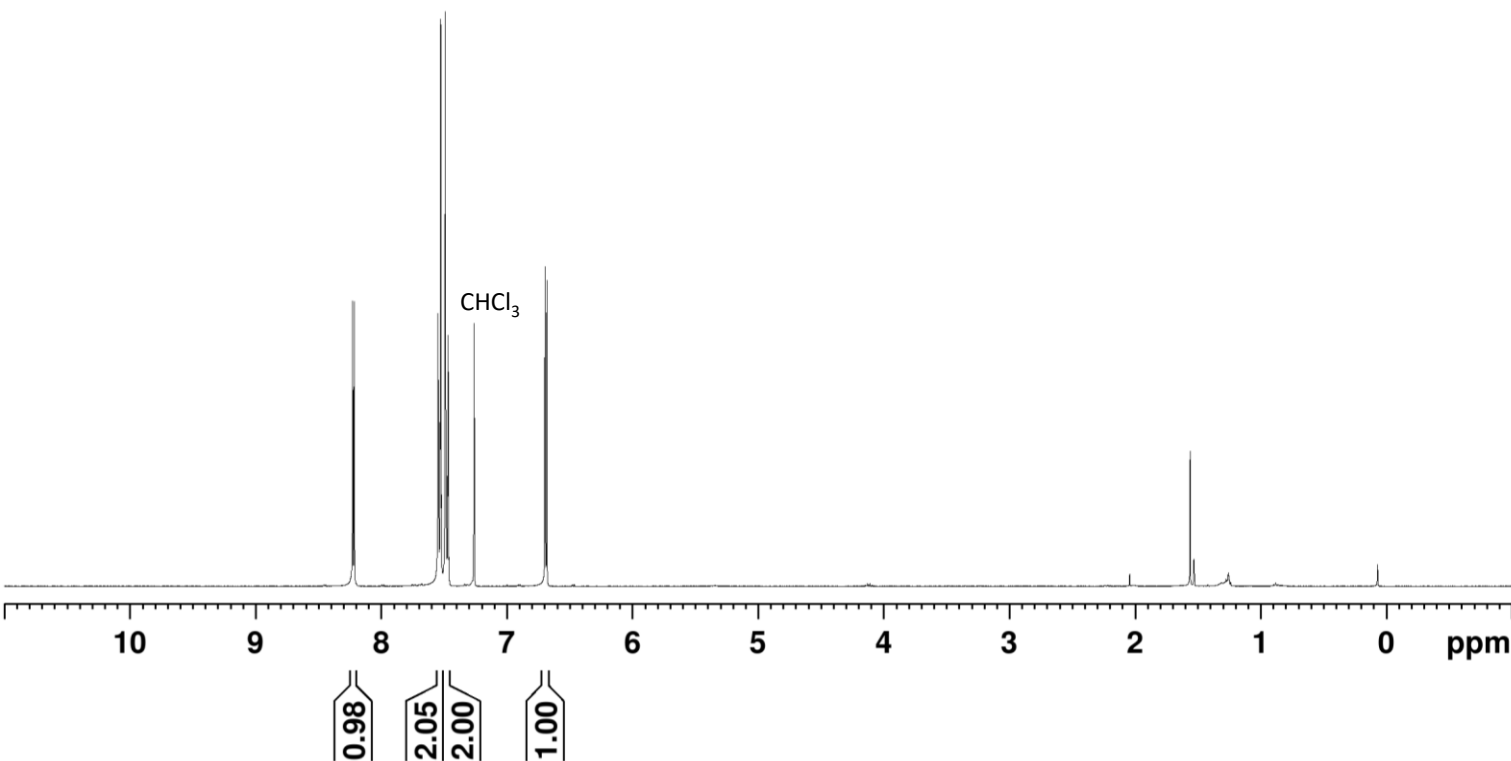
F2 - Acquisition Parameters
Date_ 20240304
Time 16.44 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0326571
P1 12.00 usec
P12 67730.91 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 429.03 Hz
SPW2 0.00000848 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300126 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00





S32b



¹H spectrum of **S32b**

Current Data Parameters
 NAME S32b_AR_240712_3
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240724
 Time 22.30 h
 INSTRUM Avance Neo
 PROBHD Z181819_0001 (
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 13.54 usec
 TE 300.1 K
 D1 1.00000000 sec
 TD0 1
 SFO1 400.1324708 MHz
 NUC1 1H
 P0 3.33 usec
 P1 10.00 usec
 PLW1 22.53199959 W

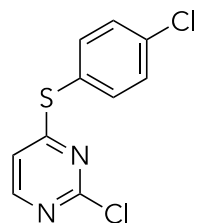
F2 - Processing parameters
 SI 65536
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C spectrum of **S32b**

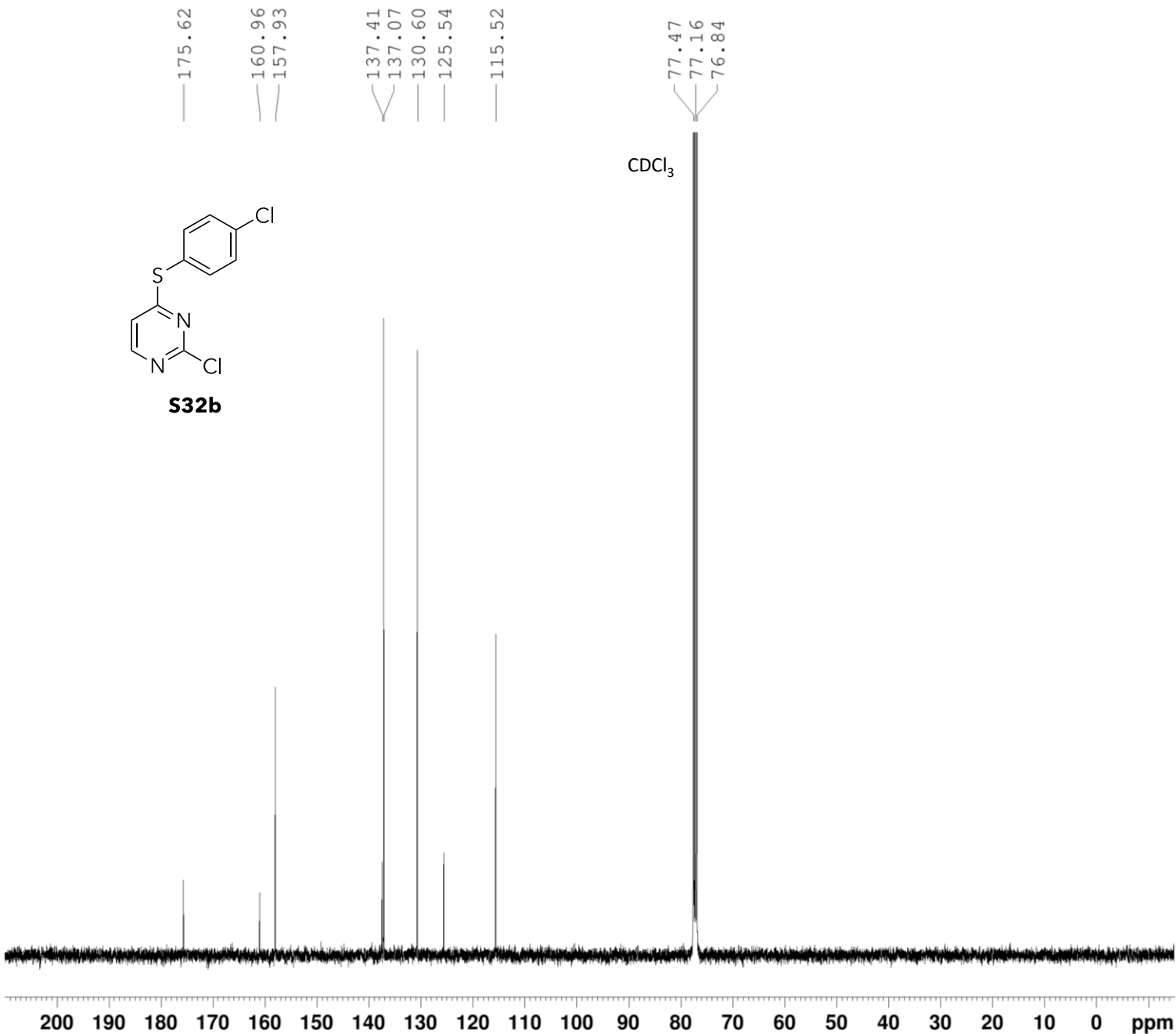
Current Data Parameters
NAME S32b_AR_240712_3
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240724
Time 23.36 h
INSTRUM Avance Neo
PROBHD z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



S32b

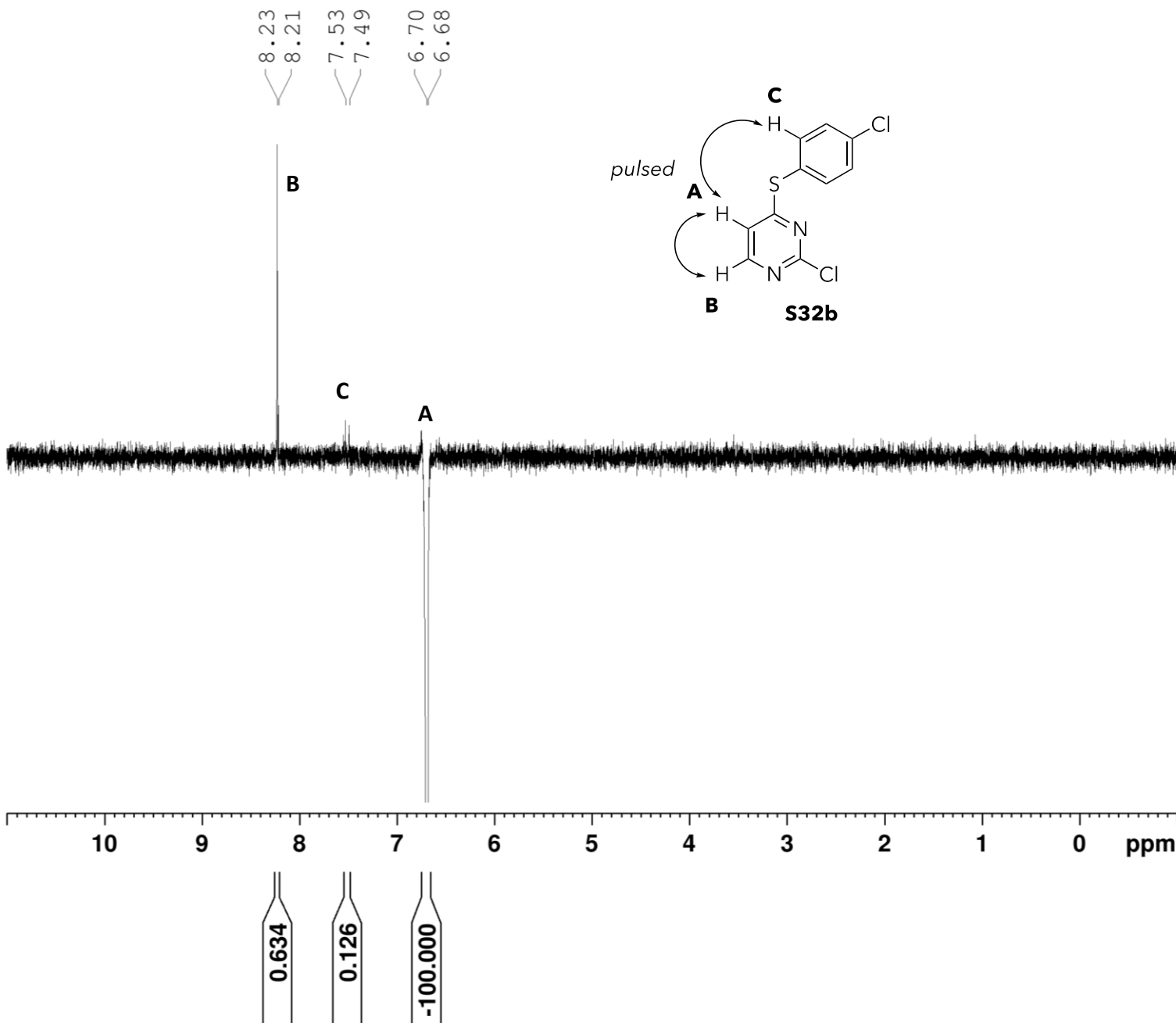


1D NOE of S32b

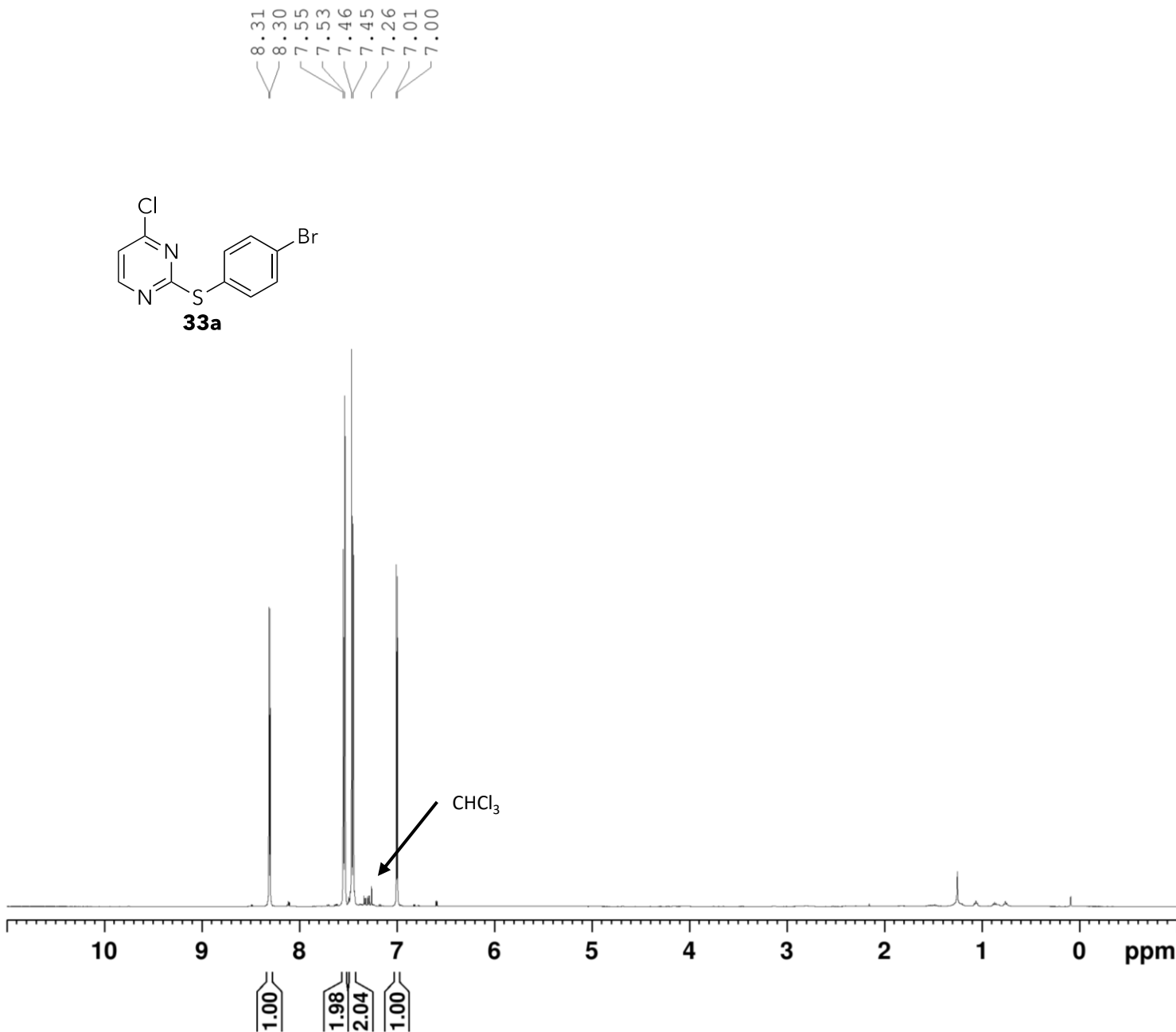
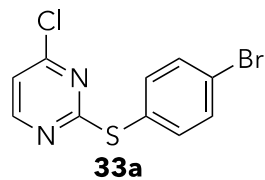
Current Data Parameters
 NAME S32b_AR_240712_3
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240724
 Time 22.35 h
 INSTRUM Avance Neo
 PROBHD Z181819_0001 (
 PULPROG selnpgpzs.2
 TD 65536
 SOLVENT CDC13
 NS 32
 DS 4
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 8.88 usec
 TE 300.1 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27266601 sec
 D31 0.30000001 sec
 TD0 1
 ZGOPTNS -DCALC_SPOFFS
 SFO1 400.1324708 MHz
 NUC1 1H
 CNST21 6.7212062
 P1 10.00 usec
 P12 40216.82 usec
 P32 20000.00 usec
 P44 300.00 usec
 PLW1 22.53199959 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 SPW2 0.00003290 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 SPW29 0.08606600 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 SPW30 1.60230005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



¹H spectrum of **33a**

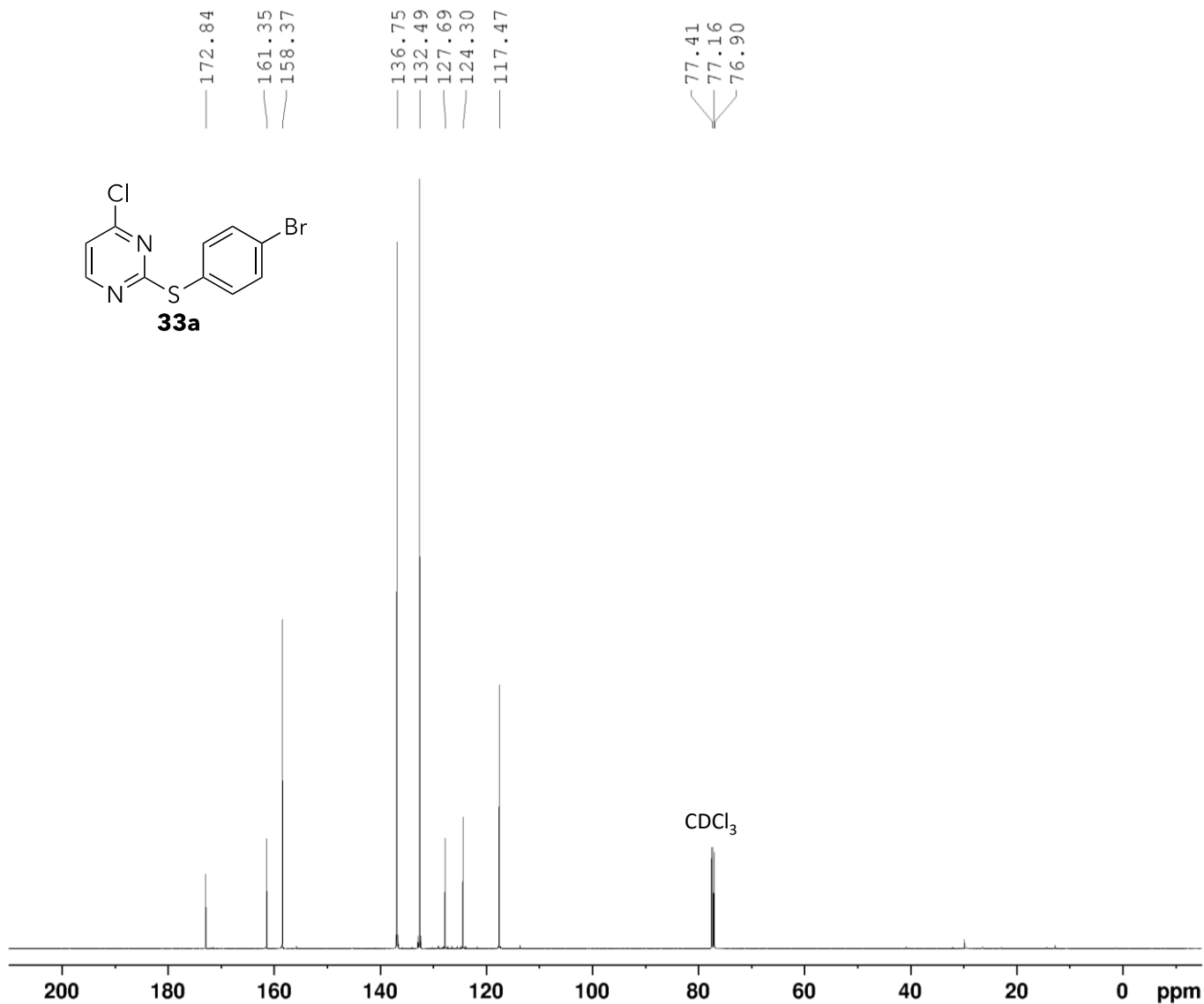
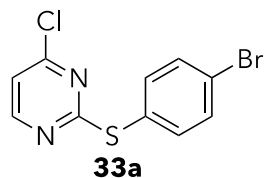


Current Data Parameters
NAME AR_03_034_500
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240119
Time 19.02 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 25.21
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **33a**



Current Data Parameters
NAME AR_03_034_500-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240119
Time 18.55 h
INSTRUM spect
PROBHD Z125869_0055 (CPD BBO 500S1 B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
F1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

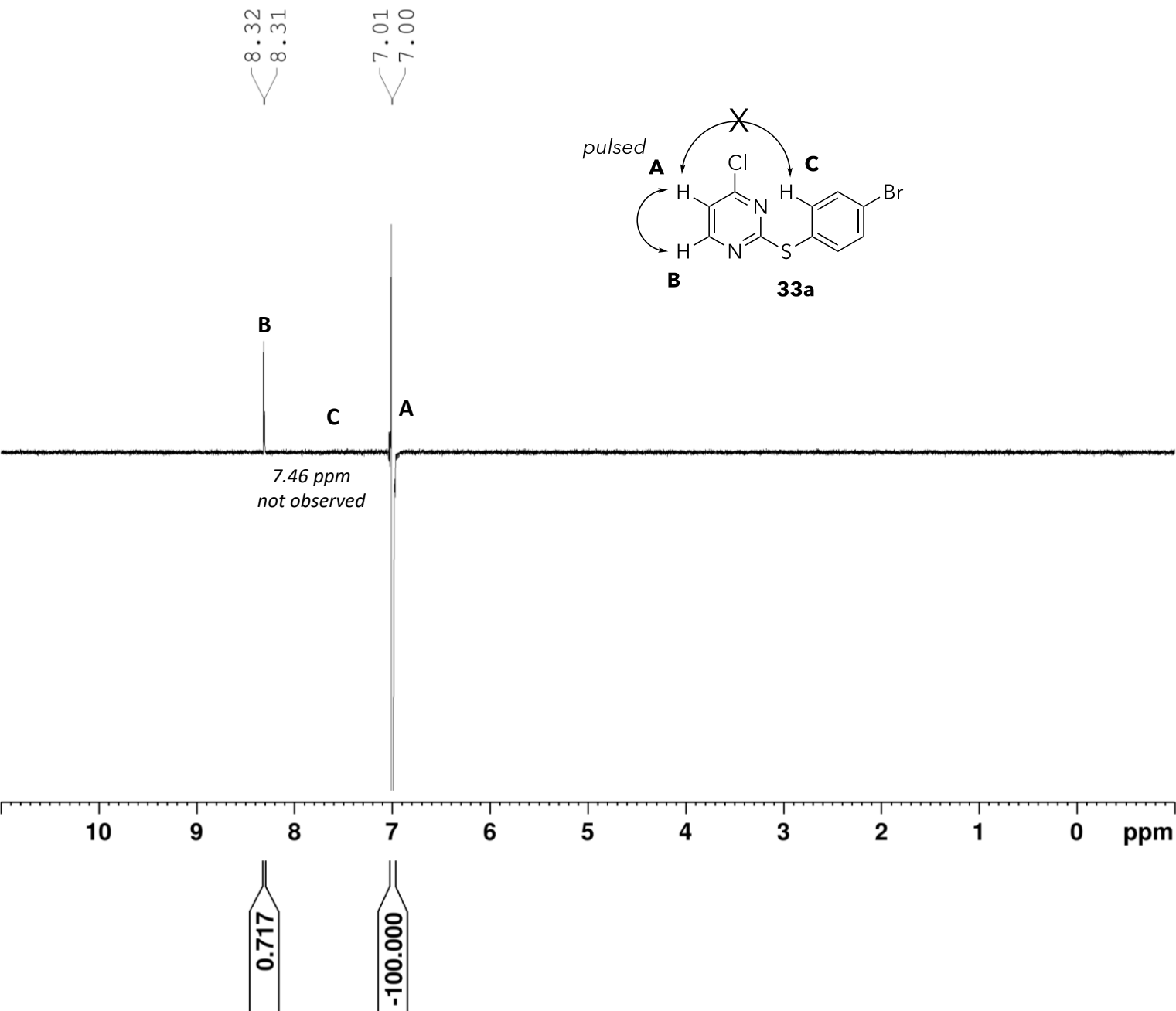
F2 - Processing parameters
SI 32768
SF 125.7829363 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

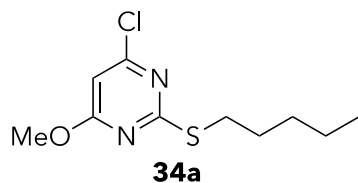
1D NOE of **33a**

Current Data Parameters
NAME 33a_AR_03_034
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240304
Time 16.17 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 171.72
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.0450048
P1 12.00 usec
P12 72443.74 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 435.20 Hz
SPW2 0.00000742 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300226 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00





¹H spectrum of **34a**

Current Data Parameters
 NAME 34a_AR-03-137
 EXPNO 16
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240527
 Time 22.05 h
 INSTRUM Avance Neo
 PROBHD Z181819_0001 (
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 32
 DW 61.000 usec
 DE 13.54 usec
 TE 300.1 K
 D1 1.00000000 sec
 TD0 1
 SFO1 400.1324708 MHz
 NUC1 1H
 P0 3.33 usec
 P1 10.00 usec
 PLW1 22.53199959 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

7.26

6.37

3.94
 3.12
 3.10
 3.08
 3.06
 1.76
 1.74
 1.72
 1.71
 1.69
 1.45
 1.43
 1.42
 1.41
 1.39
 1.38
 1.37
 1.35
 1.33
 1.31
 1.29
 0.91
 0.89
 0.87

CHCl₃

0.84

2.80

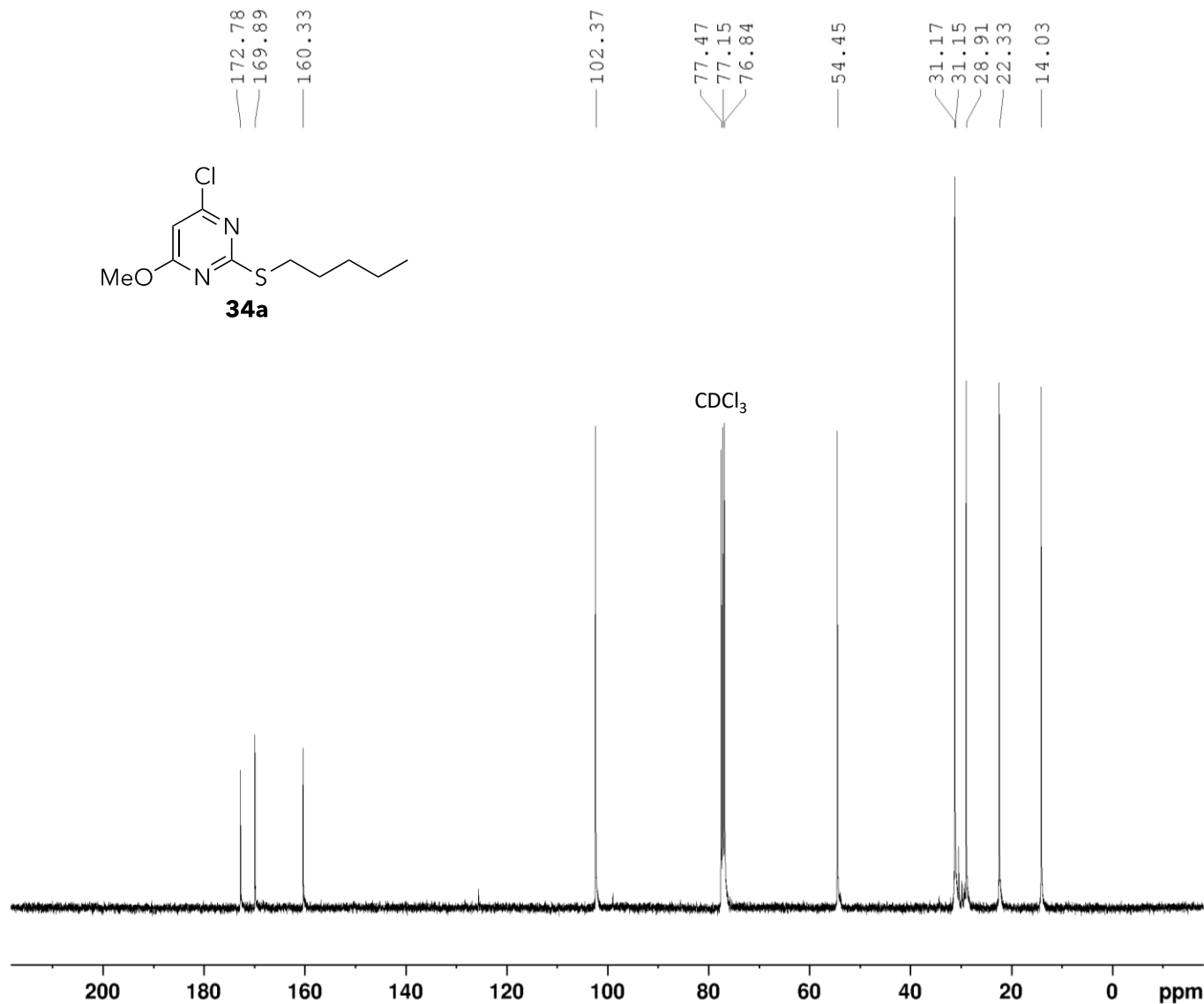
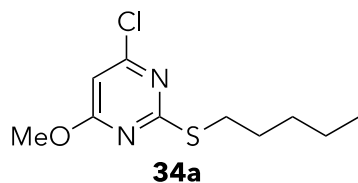
2.05

2.06

4.26

3.00

¹³C spectrum of 34a



Current Data Parameters
NAME 34a_AR-03-137
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240527
Time 22.03 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-S
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

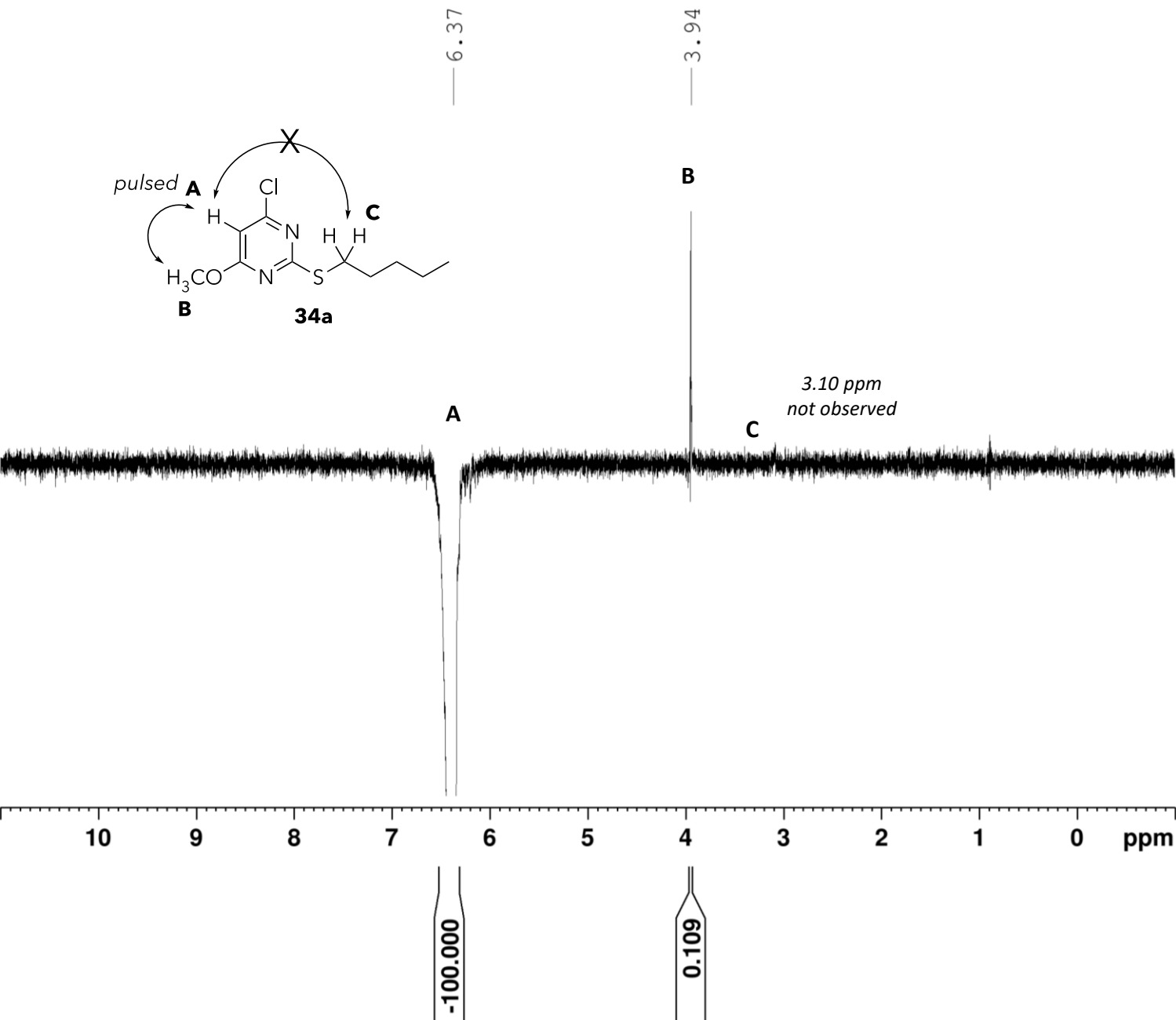
F2 - Processing parameters
SI 32768
SF 100.6127595 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **34a**

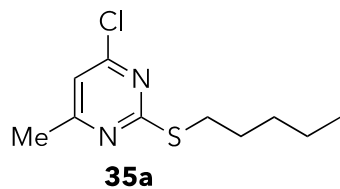
Current Data Parameters
NAME 34a_AR-03-137
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240527
Time 20.57 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG selnogpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 8.88 usec
TE 300.2 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27266601 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 400.1324708 MHz
NUC1 1H
CNST21 6.4120650
P1 10.00 usec
P12 21973.09 usec
P32 20000.00 usec
P44 300.00 usec
PLW1 22.53199959 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
SPW2 0.00011020 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
SPW29 0.08606600 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
SPW30 1.60230005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



¹H spectrum of 35a



— 7.26
— 6.83

3.15
3.13
3.11
2.42
1.76
1.74
1.72
1.71
1.69
1.46
1.45
1.44
1.43
1.41
1.39
1.38
1.37
1.35
1.33
1.31
0.93
0.91
0.89

Current Data Parameters
NAME CS-1-123_puredry
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240526
Time 17.33 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SF01 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

CHCl₃

0.92

2.09

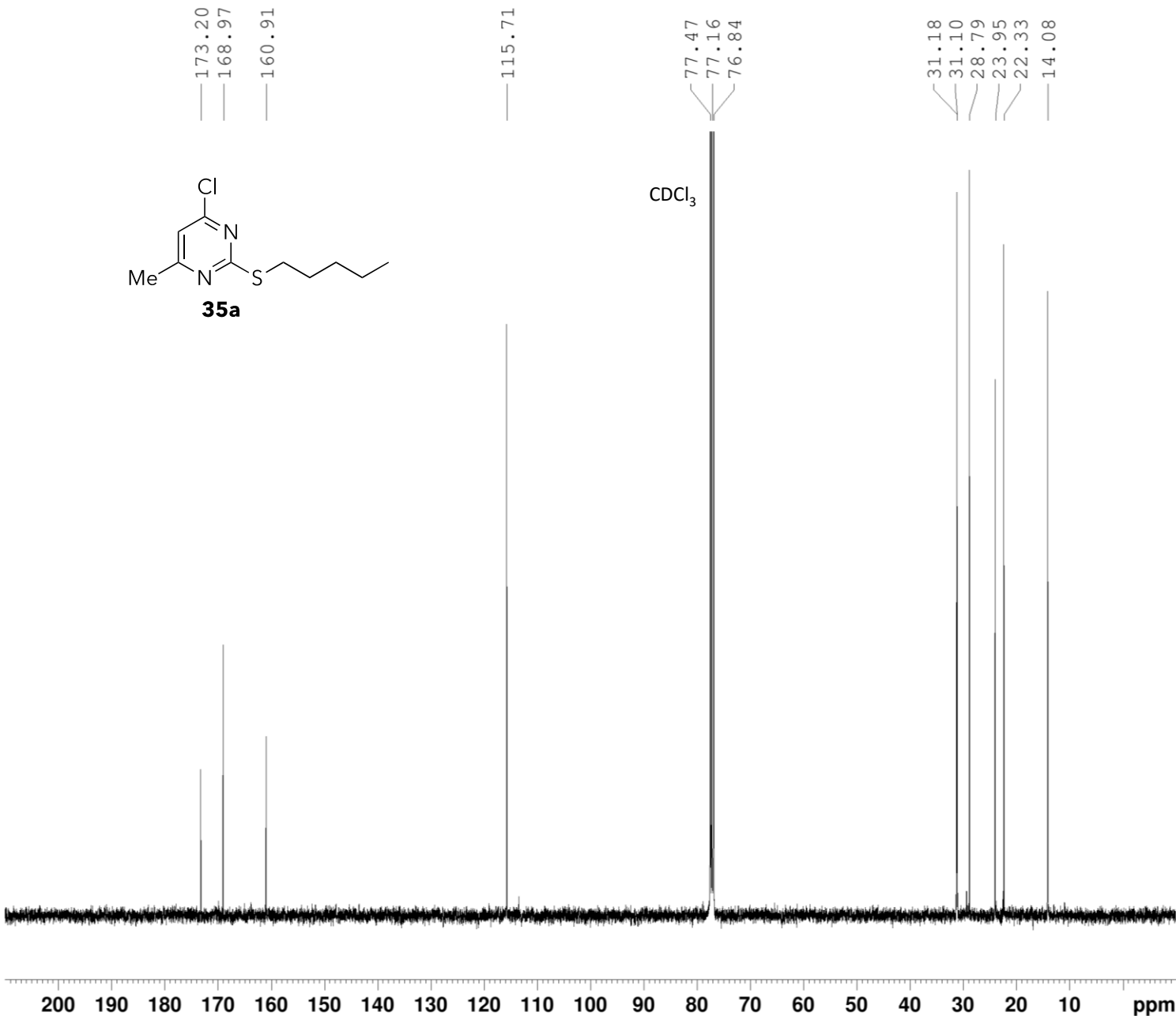
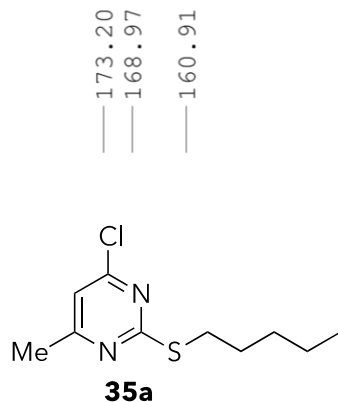
3.00

2.26

4.46

3.27

¹³C spectrum of 35a



Current Data Parameters
NAME 35a_CS-2-49
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231109
Time 21.04 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

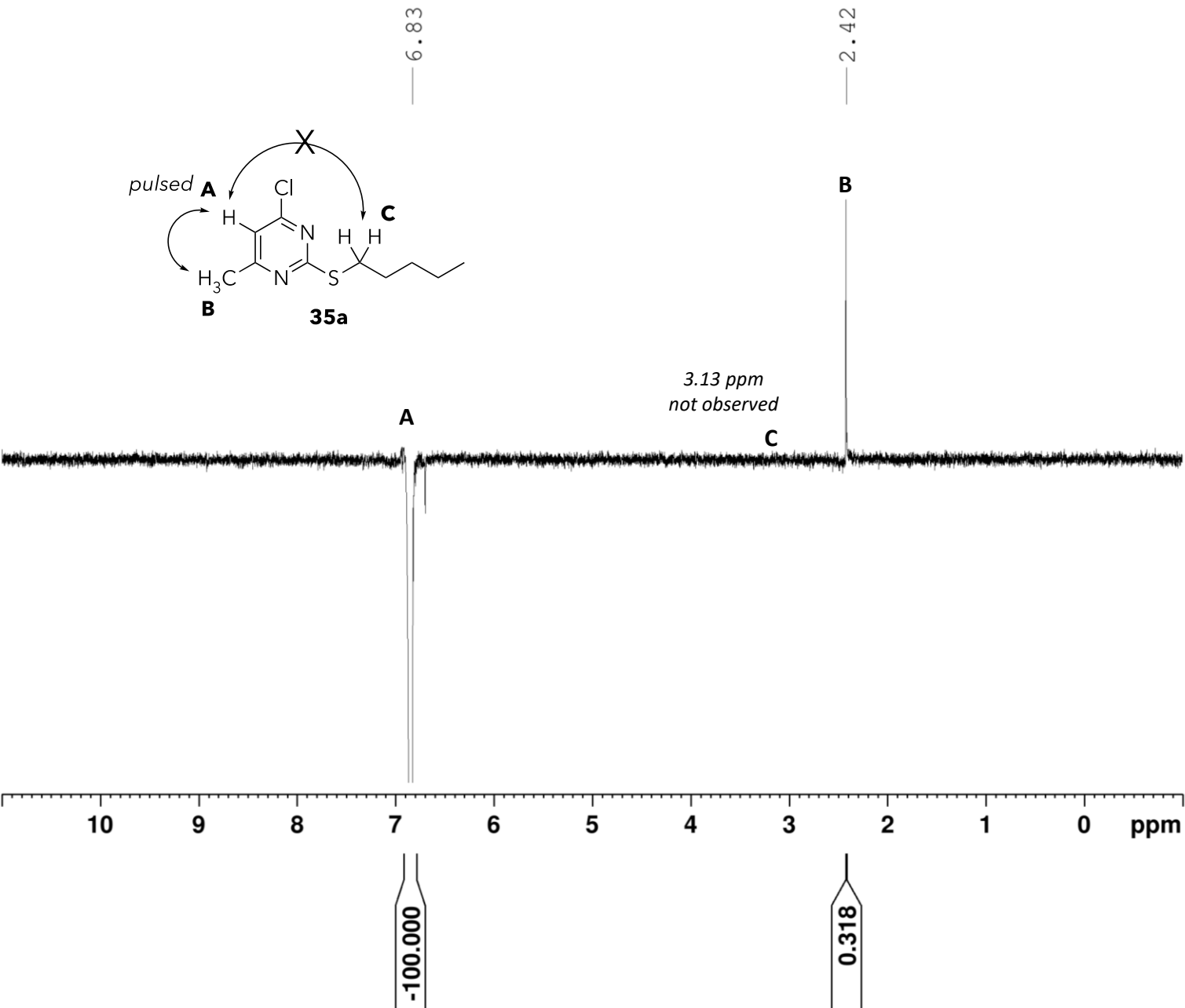
F2 - Processing parameters
SI 32768
SF 100.6127543 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 35a

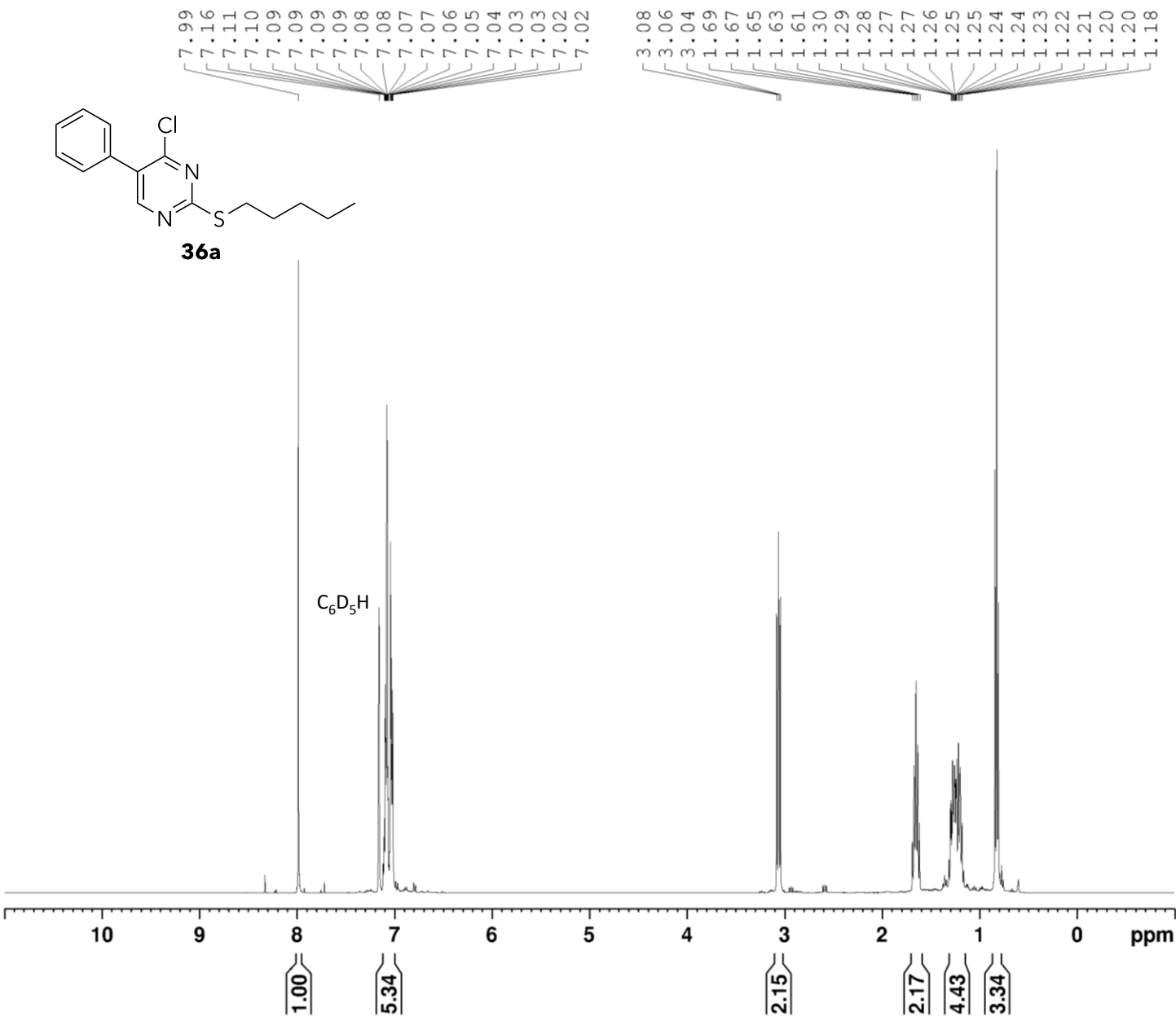
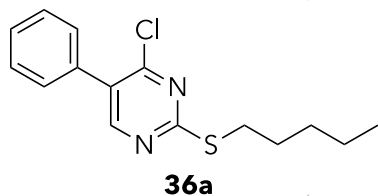
Current Data Parameters
NAME 35a_CS-2-123
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240527
Time 15.32 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 8.88 usec
TE 300.2 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27266601 sec
D31 0.30000001 sec
TD0 1
ZGOTPS -DCALC_SPOFFS
SFO1 400.1324708 MHz
NUC1 1H
CNST21 6.8638892
P1 10.00 usec
P12 28091.77 usec
P32 20000.00 usec
P44 300.00 usec
PLW1 22.53199959 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
SPW2 0.00006742 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
SPW29 0.08606600 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
SPW30 1.60230005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00



¹H spectrum of 36a

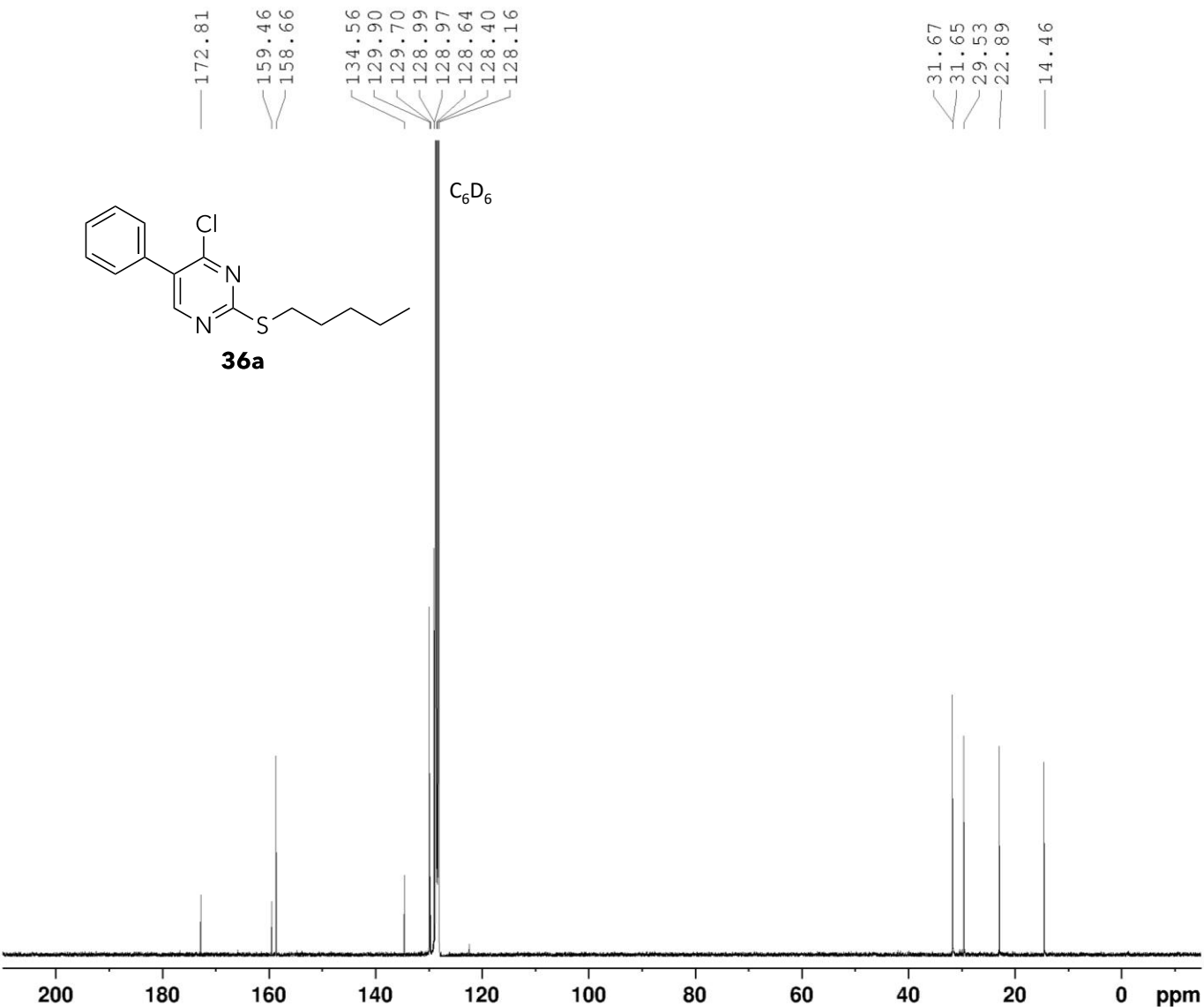


Current Data Parameters
NAME CS-2-14_purebenzene
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231003
Time 12.35 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 71.8
DW 61.000 usec
DE 13.89 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1299968 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 36a



Current Data Parameters

NAME	CS-2-14-C-benzene-1
EXPNO	21
PROCNO	1

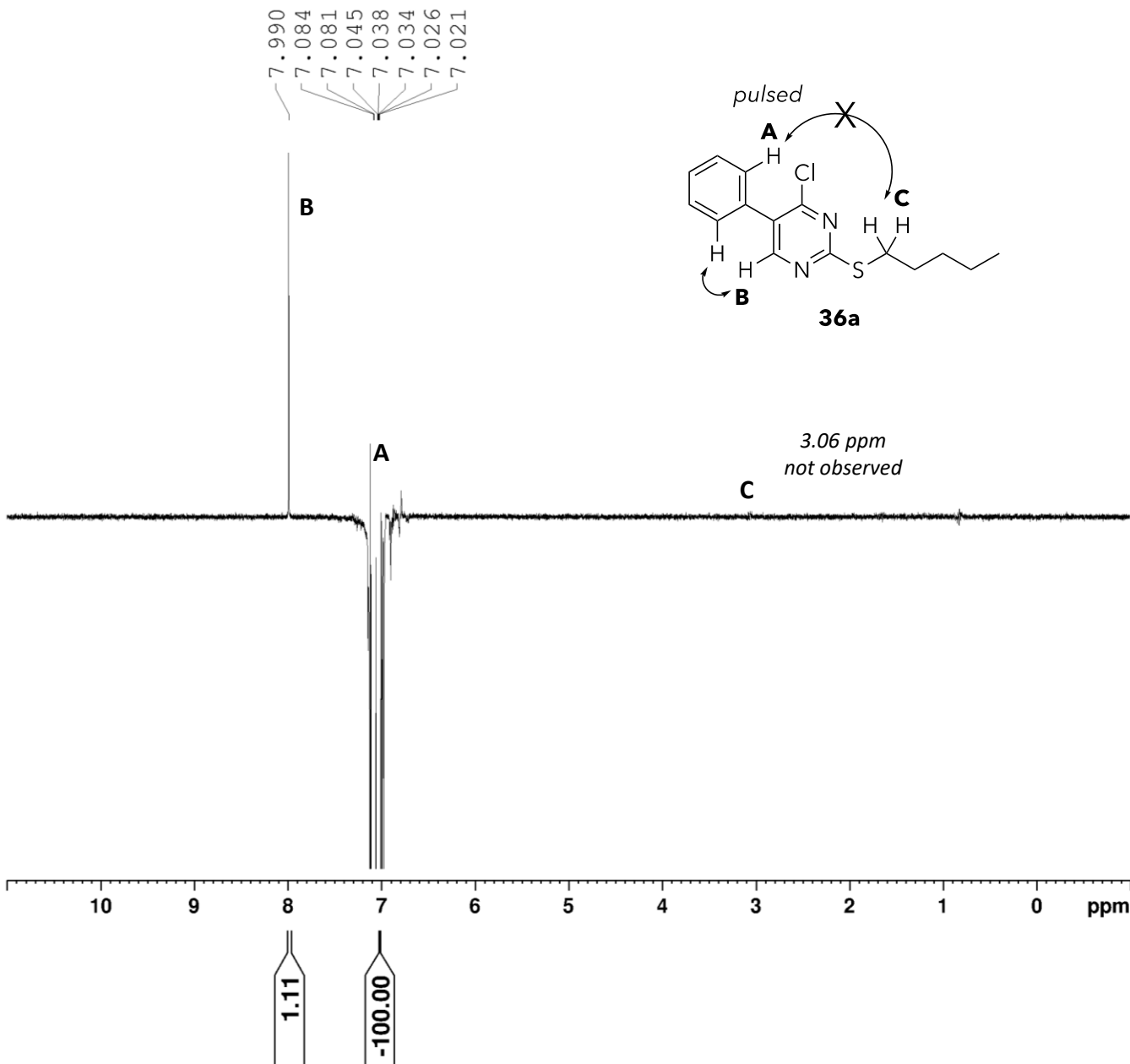
F2 - Acquisition Parameters

Date_	20241020
Time	20.21 h
INSTRUM	Avance Neo
PROBHD	Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG	zgpg30
TD	65536
SOLVENT	C6D6
NS	1024
DS	4
SWH	23809.524 Hz
FIDRES	0.726609 Hz
AQ	1.3762560 sec
RG	8
DW	21.000 usec
DE	6.50 usec
TE	300.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6228298 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
PLW1	56.91699982 W
SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2]	waltz65
PCPD2	90.00 usec
PLW2	22.53199959 W
PLW12	0.27818000 W
PLW13	0.13992000 W

F2 - Processing parameters

SI	32768
SF	100.6126963 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

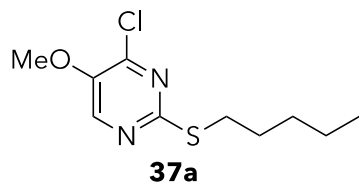
1D NOE of 36a



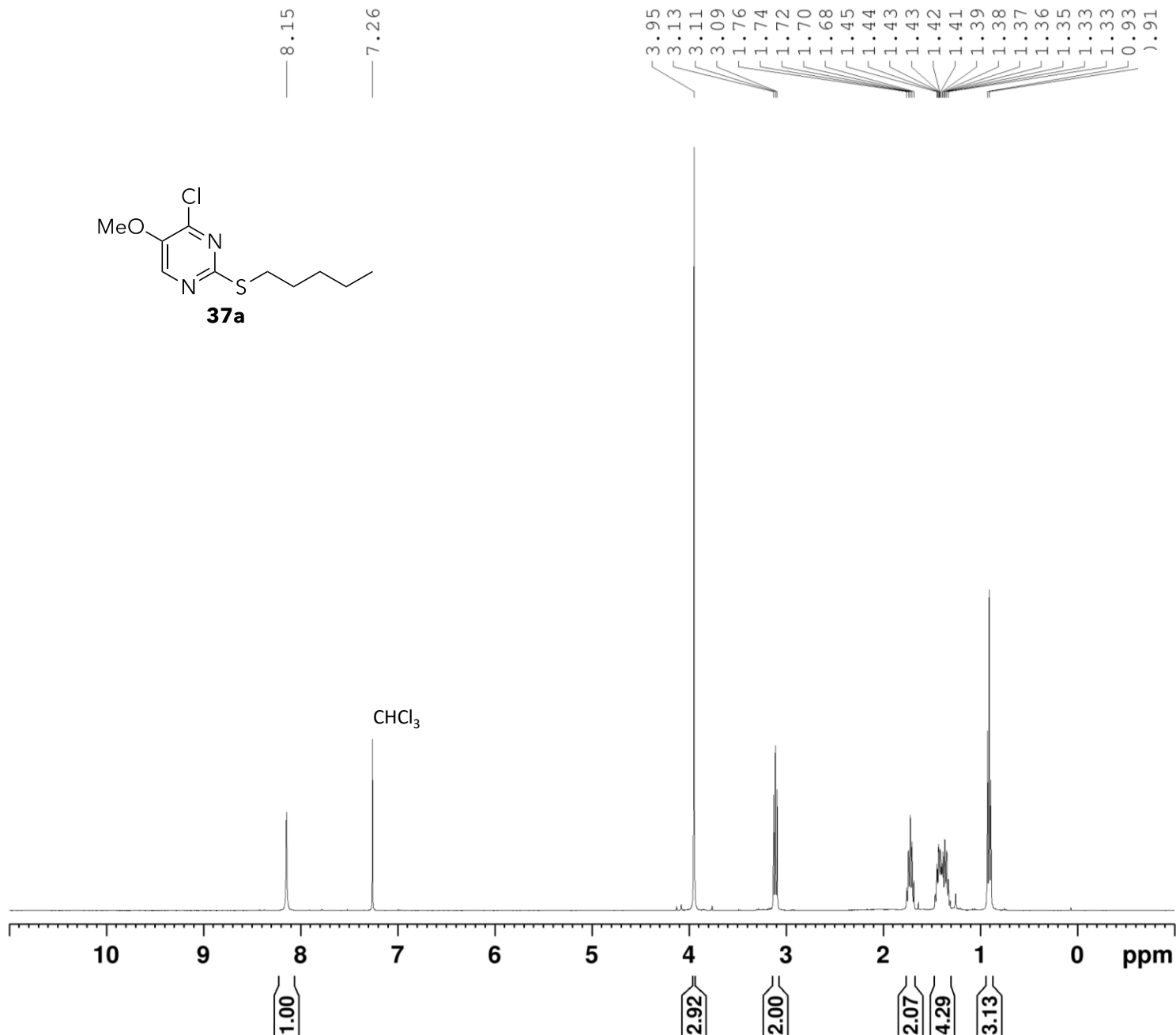
Current Data Parameters
NAME 37a_CS-2-14
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231004
Time 14.35 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (PI HR-BBO400S1-BBF/I
PULPROG selnpgpzs.2
TD 65536
SOLVENT C6D6
NS 32
DS 4
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 10.16 usec
TE 300.1 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27266601 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 400.1324708 MHz
NUC1 1H
CNST21 7.0214391
P1 8.00 usec
P12 50401.93 usec
P32 20000.00 usec
P44 300.00 usec
PLW1 24.03499985 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
SPW2 0.00001430 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
SPW29 0.05875600 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
SPW30 1.09389997 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 400.1299954 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **37a**



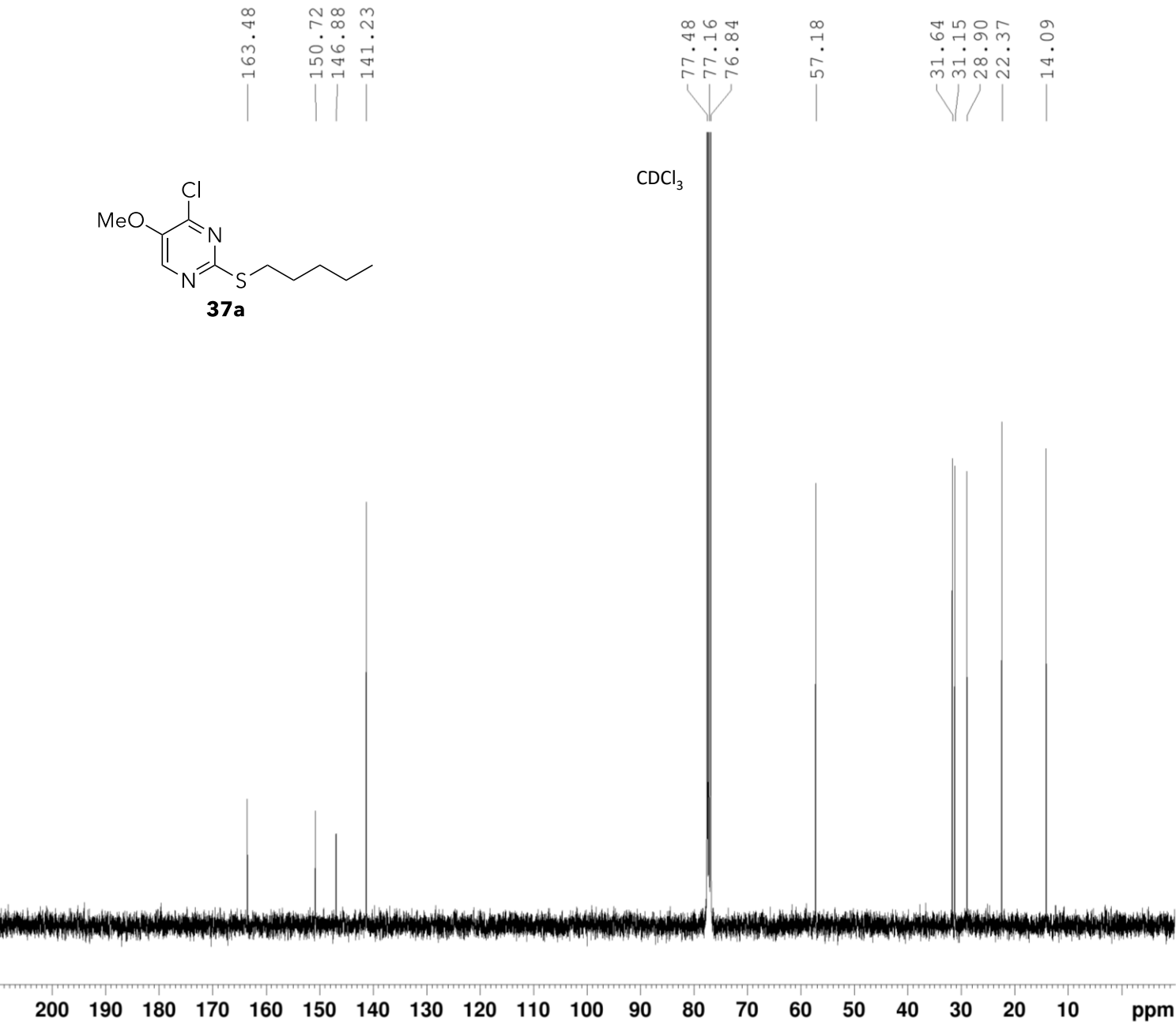
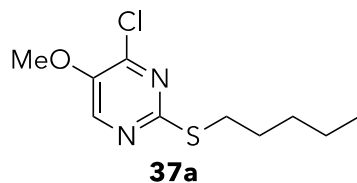
```

NAME           OJ-6-/8-2-H
EXPNO           16
PROCNO          1

F2 - Acquisition Parameters
Date_           20231216
Time            18.29 h
INSTRUM         Avance Neo
PROBHD          Z181819_0001 (
PULPROG         zg30
TD              65536
SOLVENT         CDCl3
NS              16
DS              2
SWH             8196.721 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG              101
DW              61.000 usec
DE              13.54 usec
TE              300.1 K
D1              1.00000000 sec
TD0             1
SFO1            400.1324708 MHz
NUC1            1H
P0              3.33 usec
P1              10.00 usec
PLW1            22.53199959 W

F2 - Processing parameters
SI              65536
SF              400.1300096 MHz
WDW             EM
SSB             0
LB              0.30 Hz
GB              0
PC              1.00
  
```

¹³C spectrum of **37a**



Current Data Parameters
NAME OJ-6-78-2-C
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231216
Time 19.29 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.9169982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

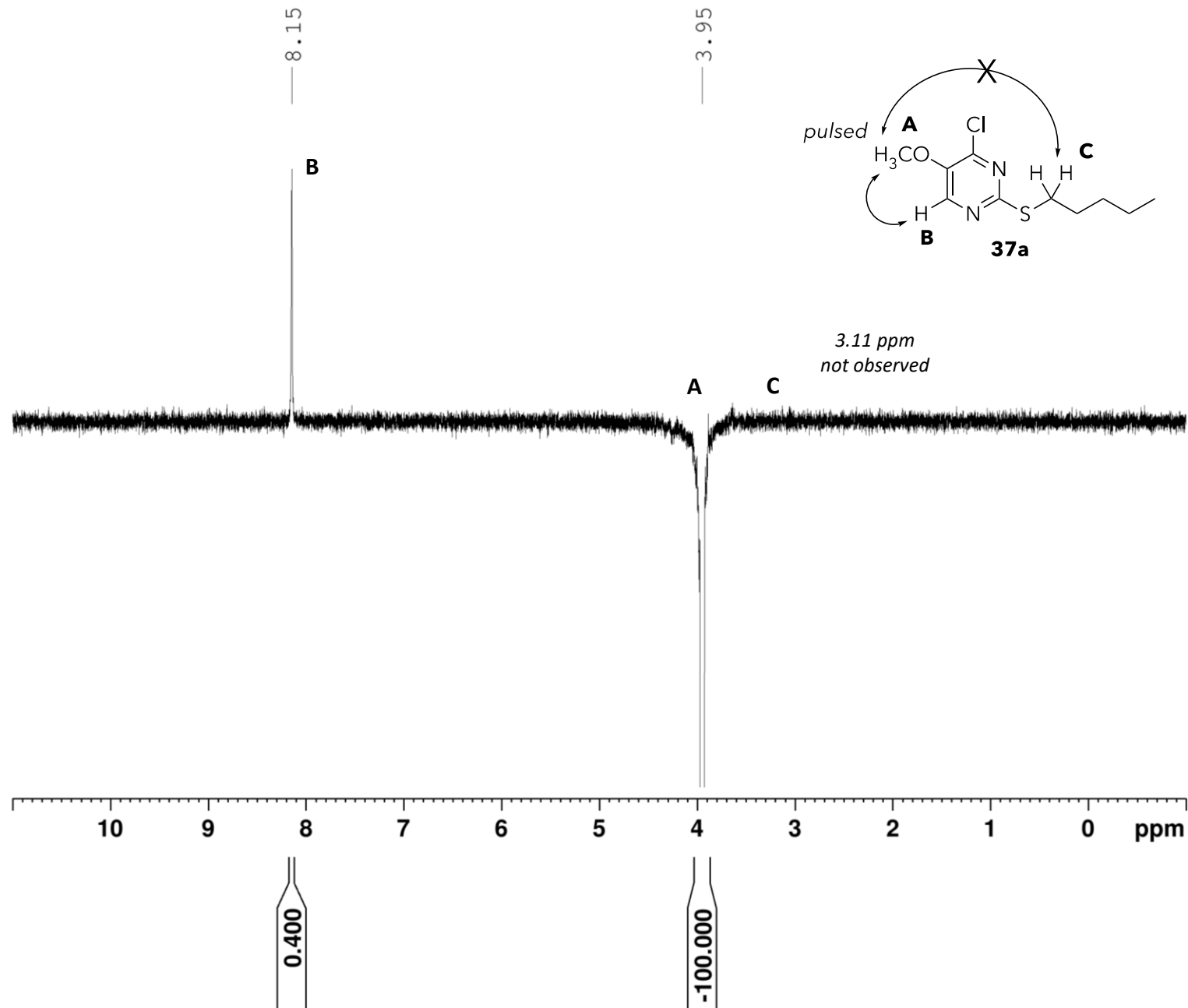
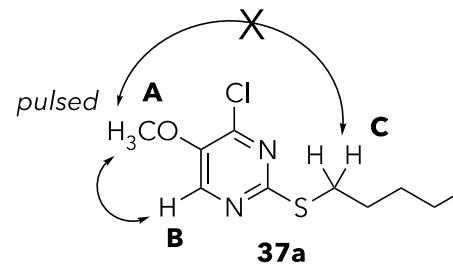
F2 - Processing parameters
SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

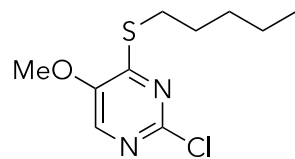
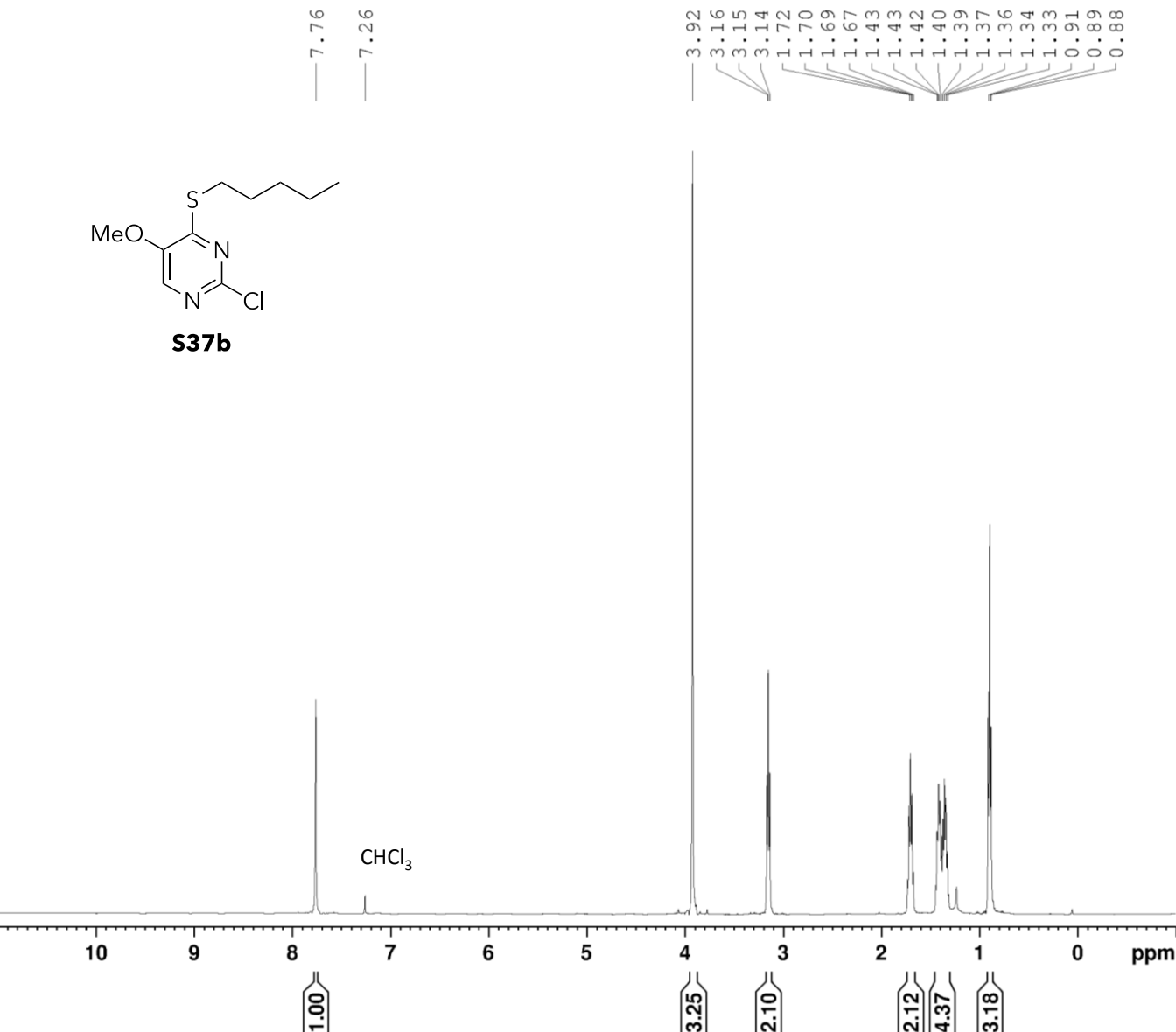
1D NOE of **37a**

Current Data Parameters
NAME OJ-6-78-2-NOE
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231218
Time 16.19 h
INSTRUM spect
PROBHD Z125869_0055 (PULPROG selnoggps.2)
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 3.9711730
P1 12.00 usec
P12 141031.91 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -1102.42 Hz
SPW2 0.00000196 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300123 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



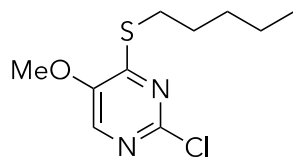
**S37b**

Current Data Parameters
NAME OJ-240923-3-NOE
EXPNO 9
PROCNO 1

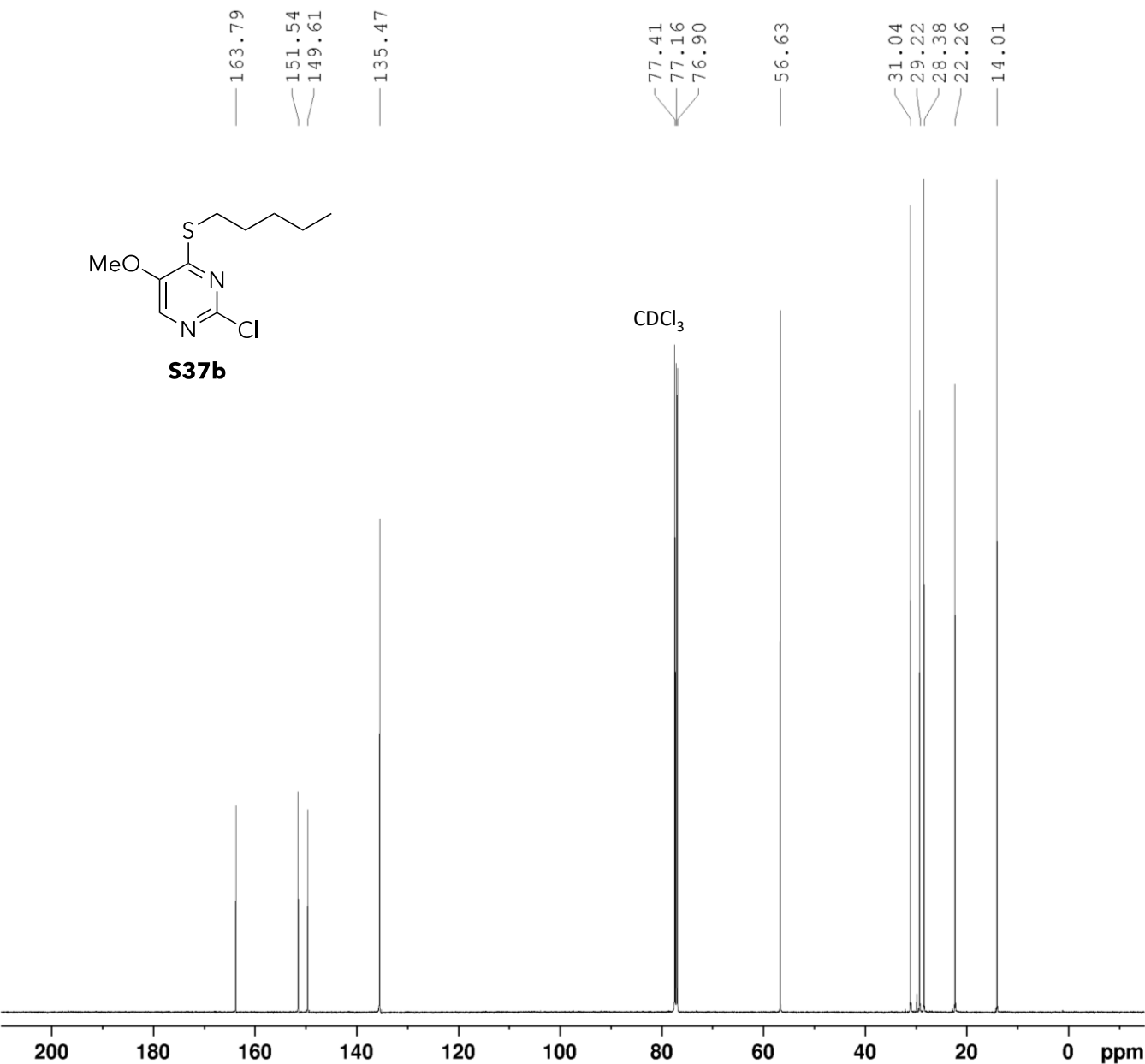
F2 - Acquisition Parameters
Date_ 20240923
Time 22.35 h
INSTRUM spect
PROBHD Z125869_0055 (CPP BBO 500S1 B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **S37b**



S37b



Current Data Parameters

NAME OJ-240923-C
EXPNO 13
PROCNO 1

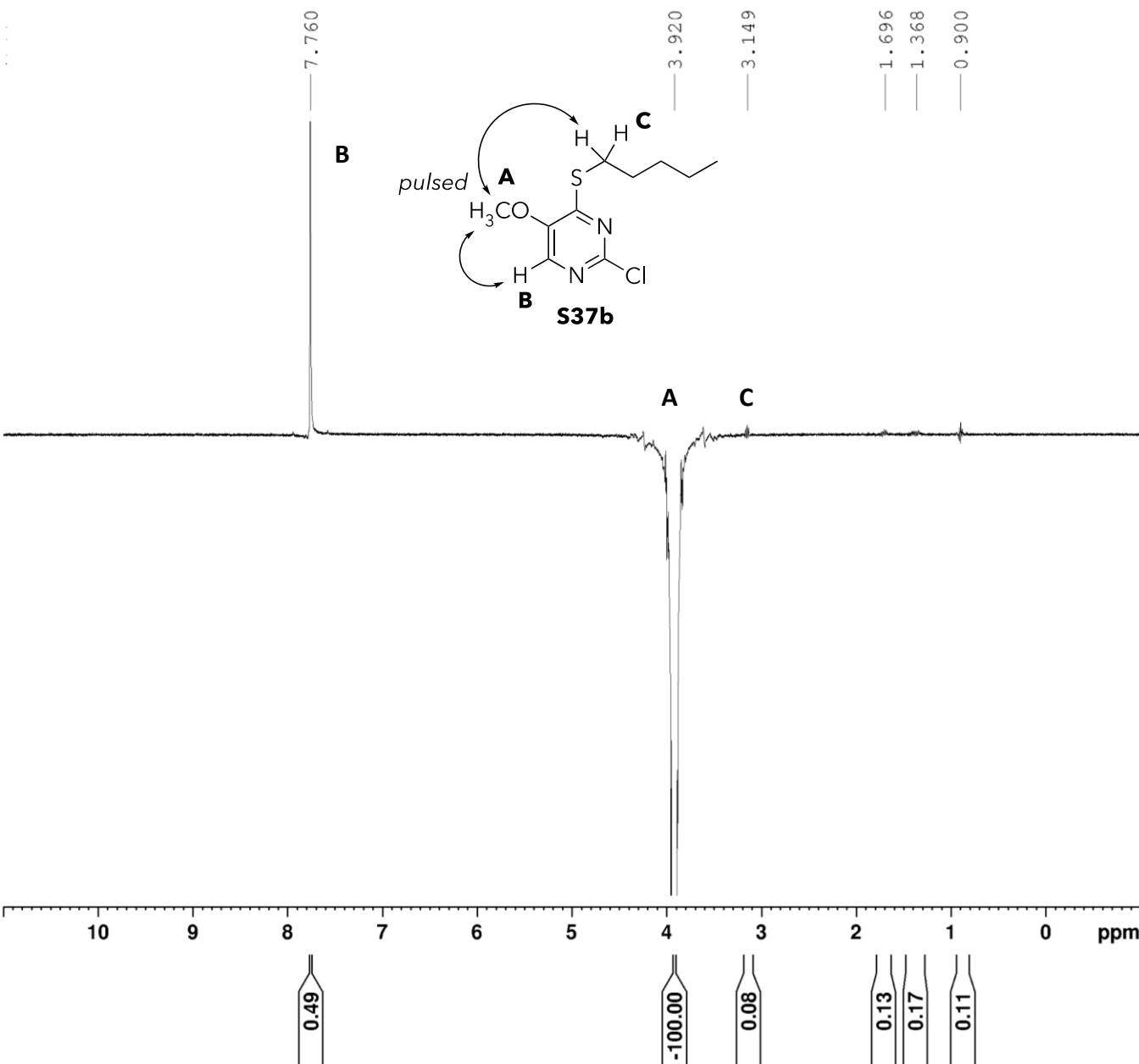
F2 - Acquisition Parameters

Date_ 20240924
Time 2.20 h
INSTRUM spect
PROBHD Z125869_0055 (CPD BBO 500S
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

F2 - Processing parameters

SI 32768
SF 125.7829222 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **S37b**

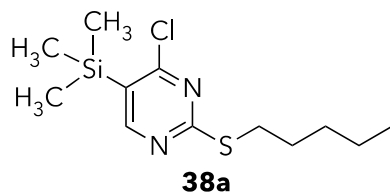


Current Data Parameters
NAME OJ-240923-3-NOE
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240923
Time 22.40 h
INSTRUM spect
PROBHD Z125869_0055 (CPD BBO 500S1 BB-H&
PULPROG selnoggps.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 171.72
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 3.9483471
P1 12.00 usec
P12 33086.13 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -1113.84 Hz
SPW2 0.00003556 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300137 MHz
WDW EM
SSB 0
LB 0.10 Hz
-- ^

¹H spectrum of **38a**



3.15
3.13
3.11
1.77
1.75
1.73
1.71
1.69
1.47
1.47
1.45
1.44
1.43
1.43
1.42
1.41
1.40
1.38
1.37
1.35
1.33
1.31
0.93
0.91
0.89
0.37

Current Data Parameters

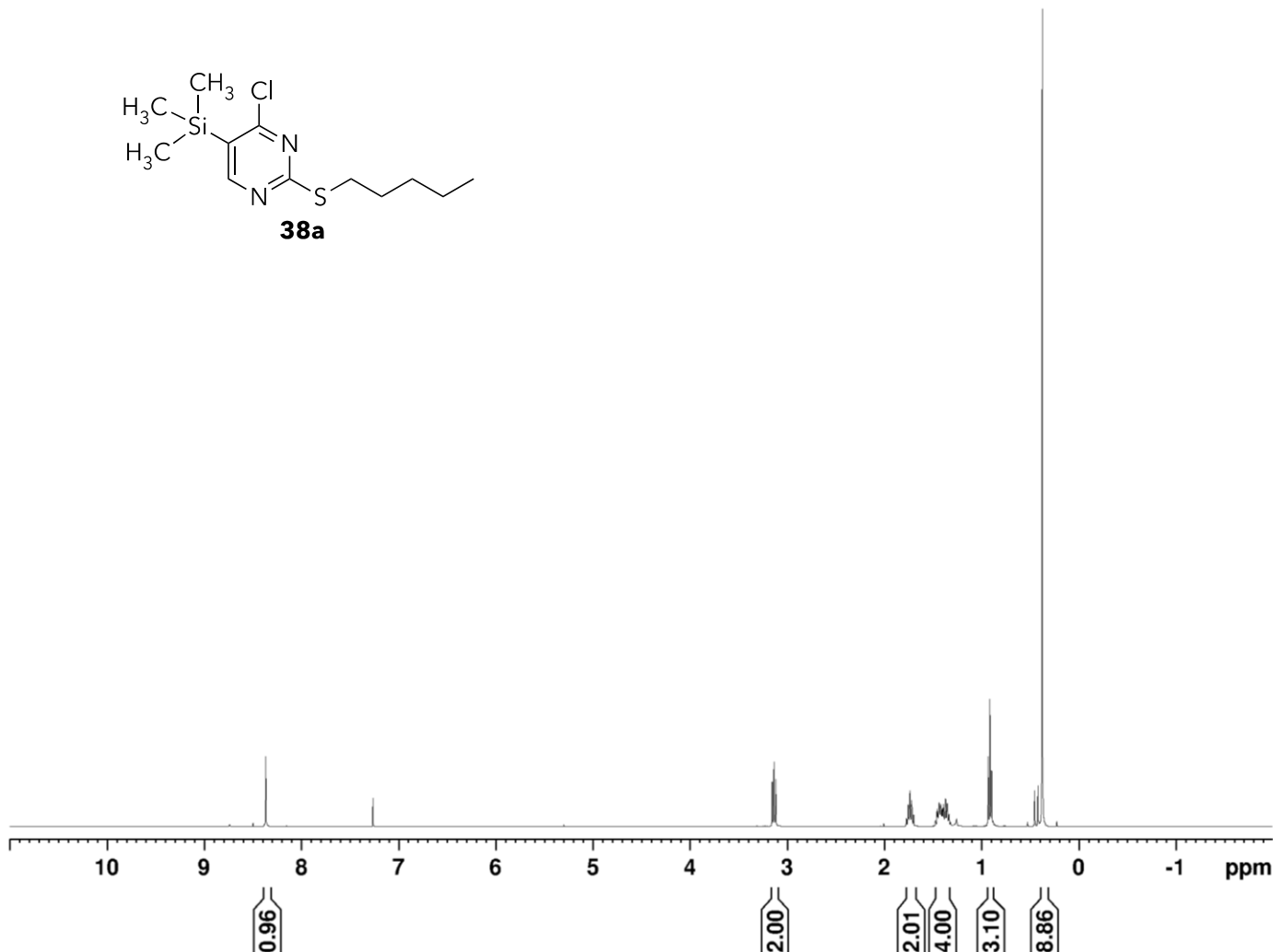
NAME OJ-250102-1e-H-2
EXPNO 27
PROCNO 1

F2 - Acquisition Parameters

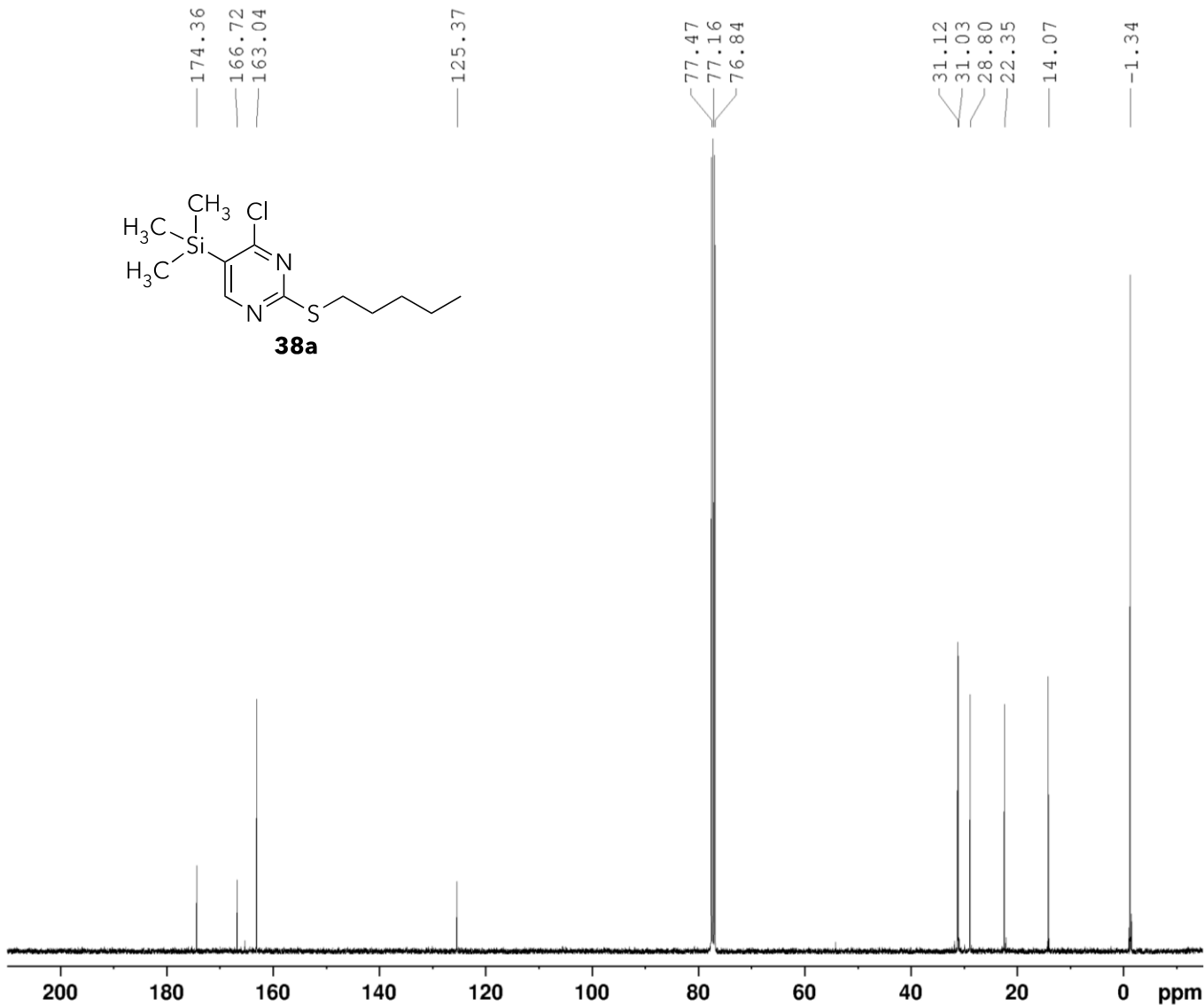
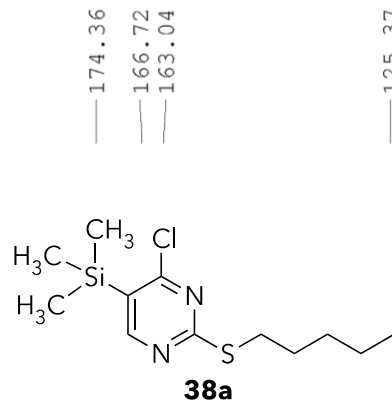
Date_ 20250104
Time 10.36 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C spectrum of 38a

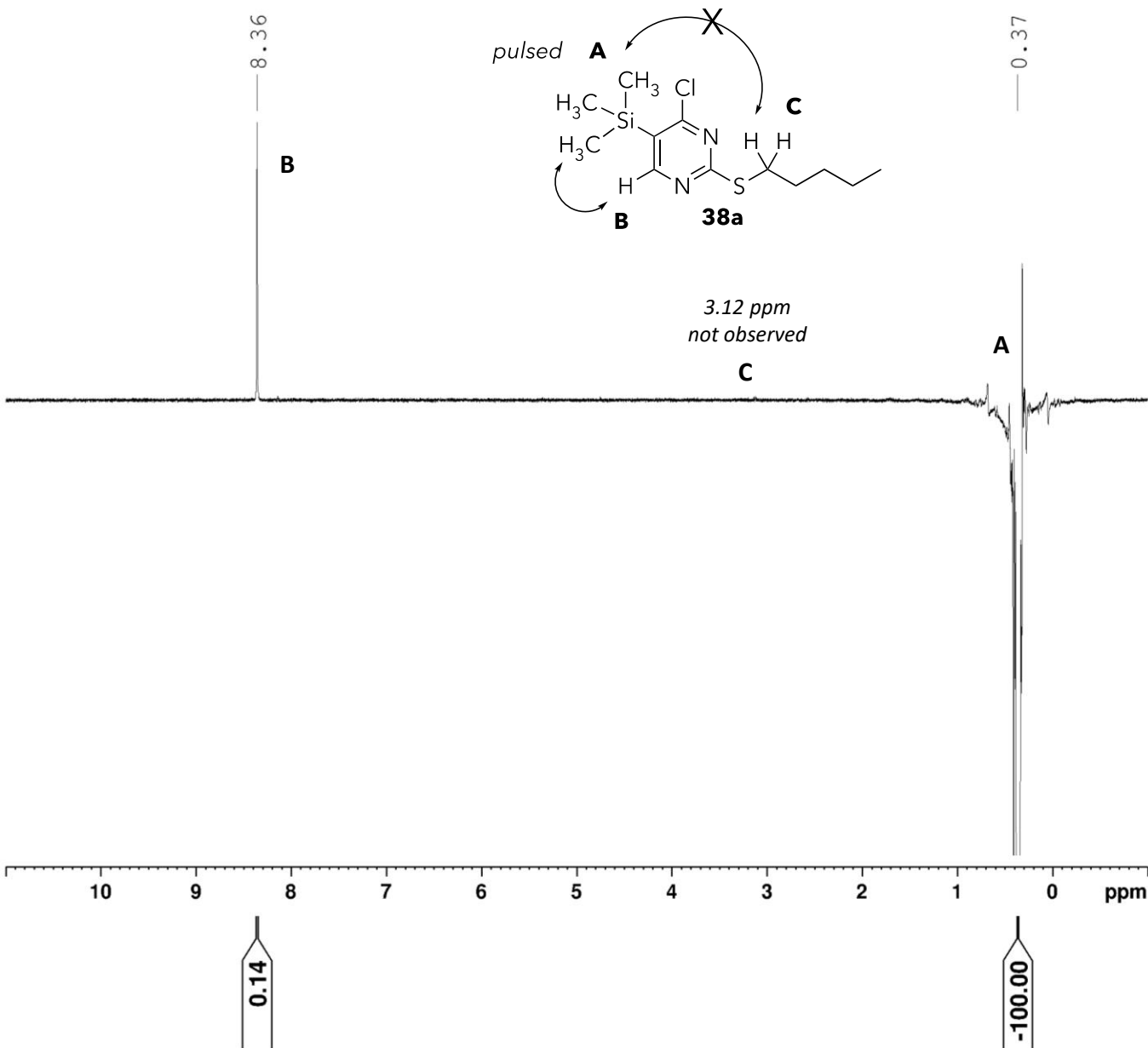


Current Data Parameters
NAME OJ-250102-1e-C
EXPNO 29
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250104
Time 11.39 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 10
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 38a

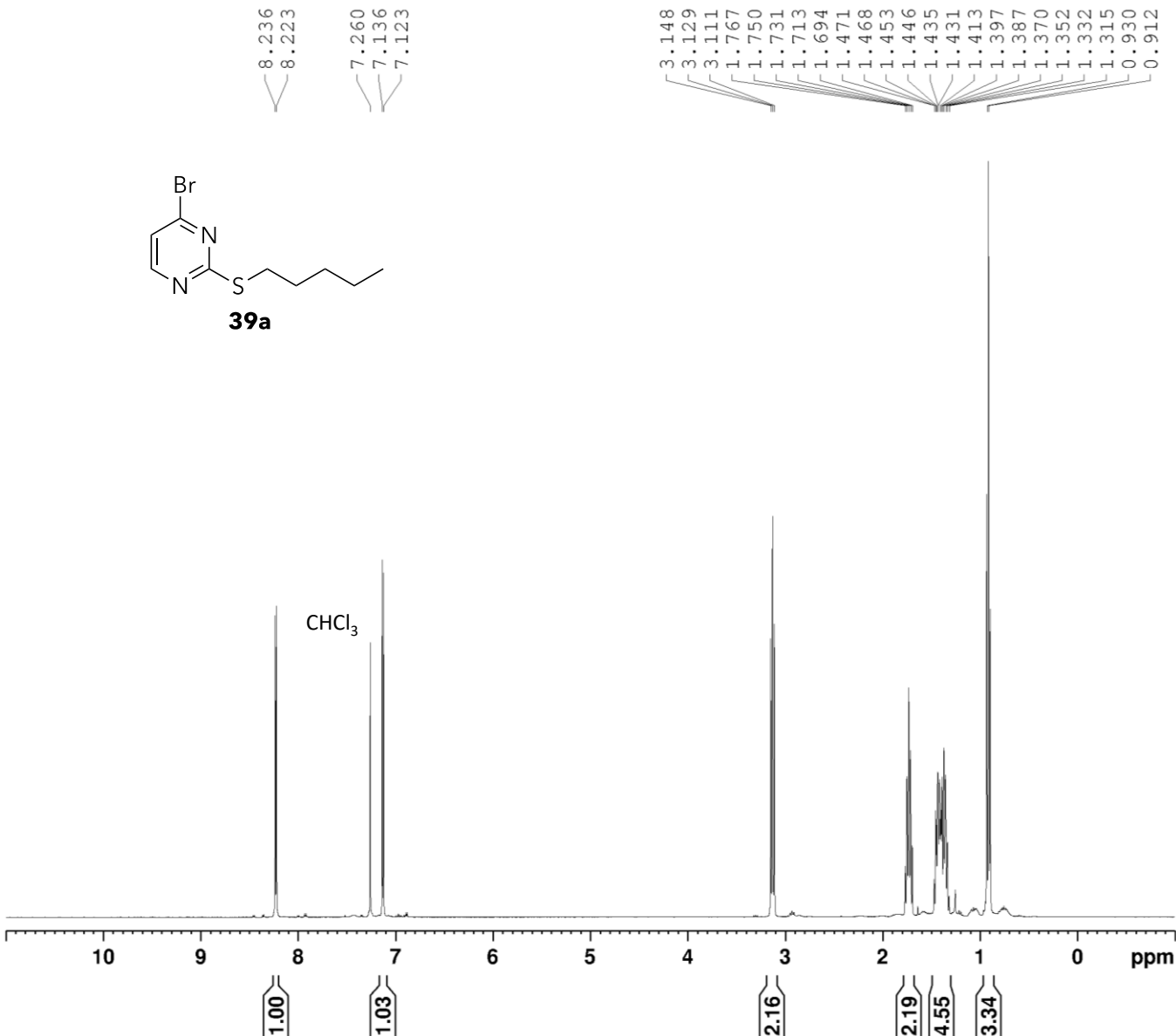
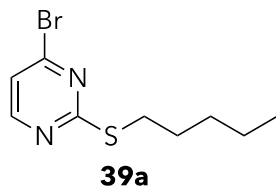


Current Data Parameters
NAME OJ-250102-1e-H
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250104
Time 21.05 h
INSTRUM spect
PROBHD Z125869_0055 (CPD BBO 500S1 BB-H&F-05 Z)
PULPROG selnoggpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 0.3906646
P1 12.00 usec
P12 57511.47 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SFNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -2893.50 Hz
SPW2 0.00001177 W
SFNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SFNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GFNAM[1] SMSQ10.100
GPZ1 15.00 %
GFNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300134 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

¹H spectrum of 39a

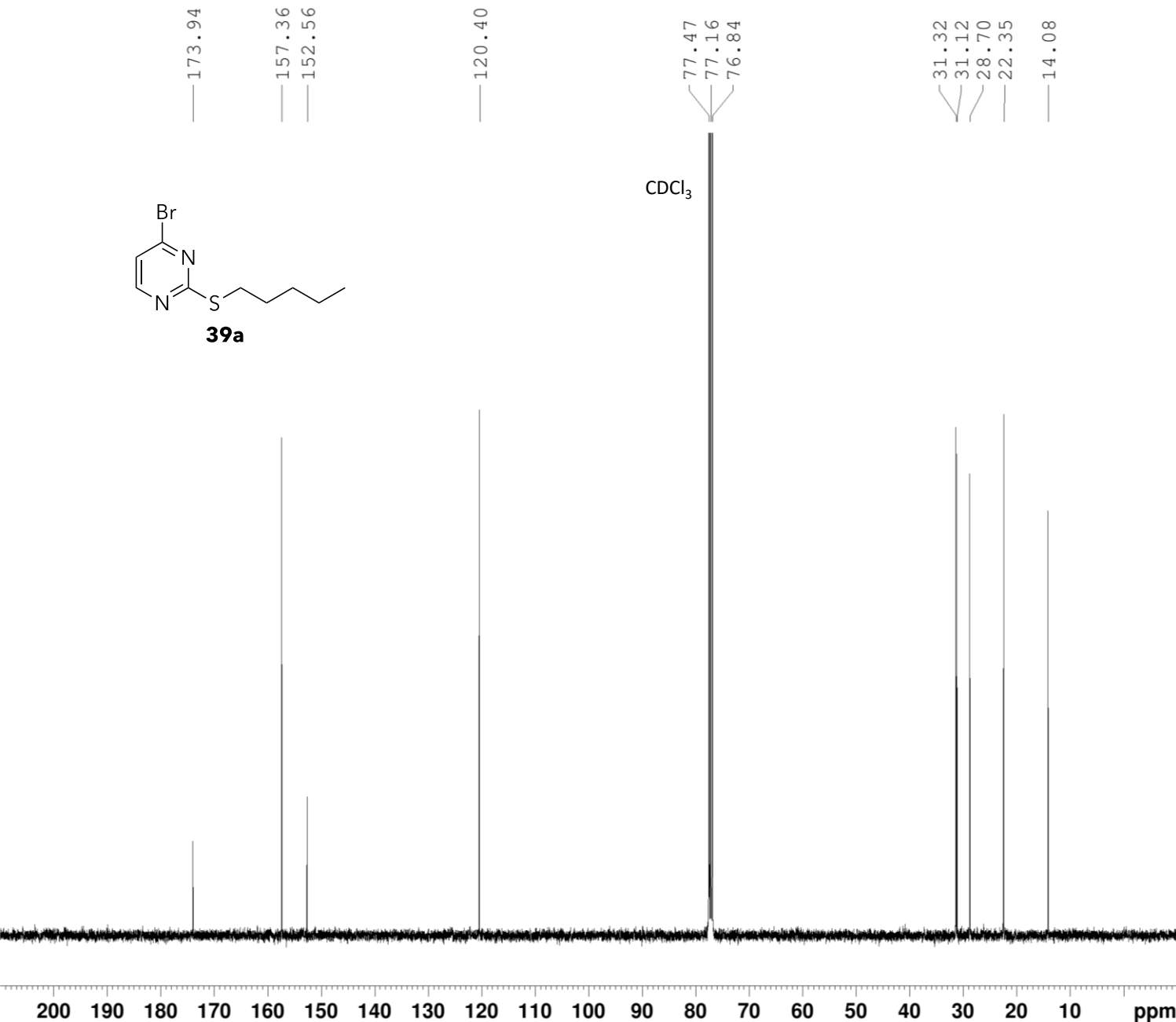
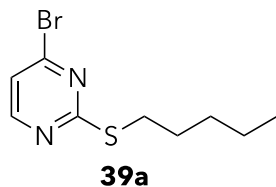


Current Data Parameters
NAME 40a_CS-2-65
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231215
Time 11.07 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P...
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **39a**



Current Data Parameters
NAME 40a_CS-2-65
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231215
Time 21.09 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

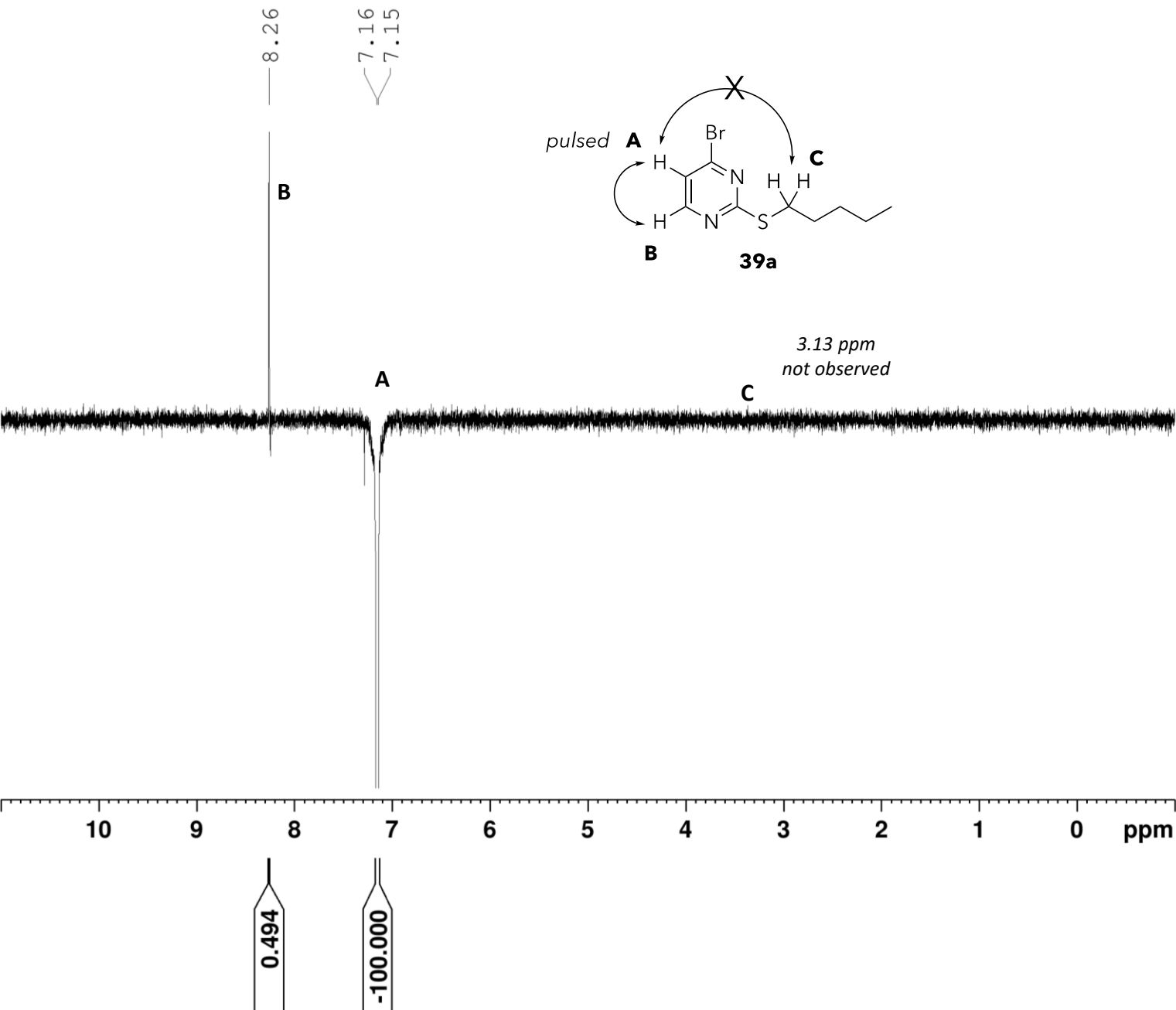
1D NOE of 39a

Current Data Parameters
NAME 40a_CS-2-65
EXPNO 12
PROCNO 1

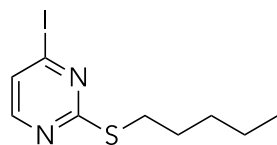
F2 - Acquisition Parameters

Date_ 20231215
Time 11.17 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG selnpgpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 8.88 usec
TE 300.2 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27266601 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 400.1324708 MHz
NUC1 1H
CNST21 7.1555920
P1 10.00 usec
P12 40543.49 usec
P32 20000.00 usec
P44 300.00 usec
PLW1 22.53199959 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
SPW2 0.00003237 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
SPW29 0.08606600 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
SPW30 1.60230005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

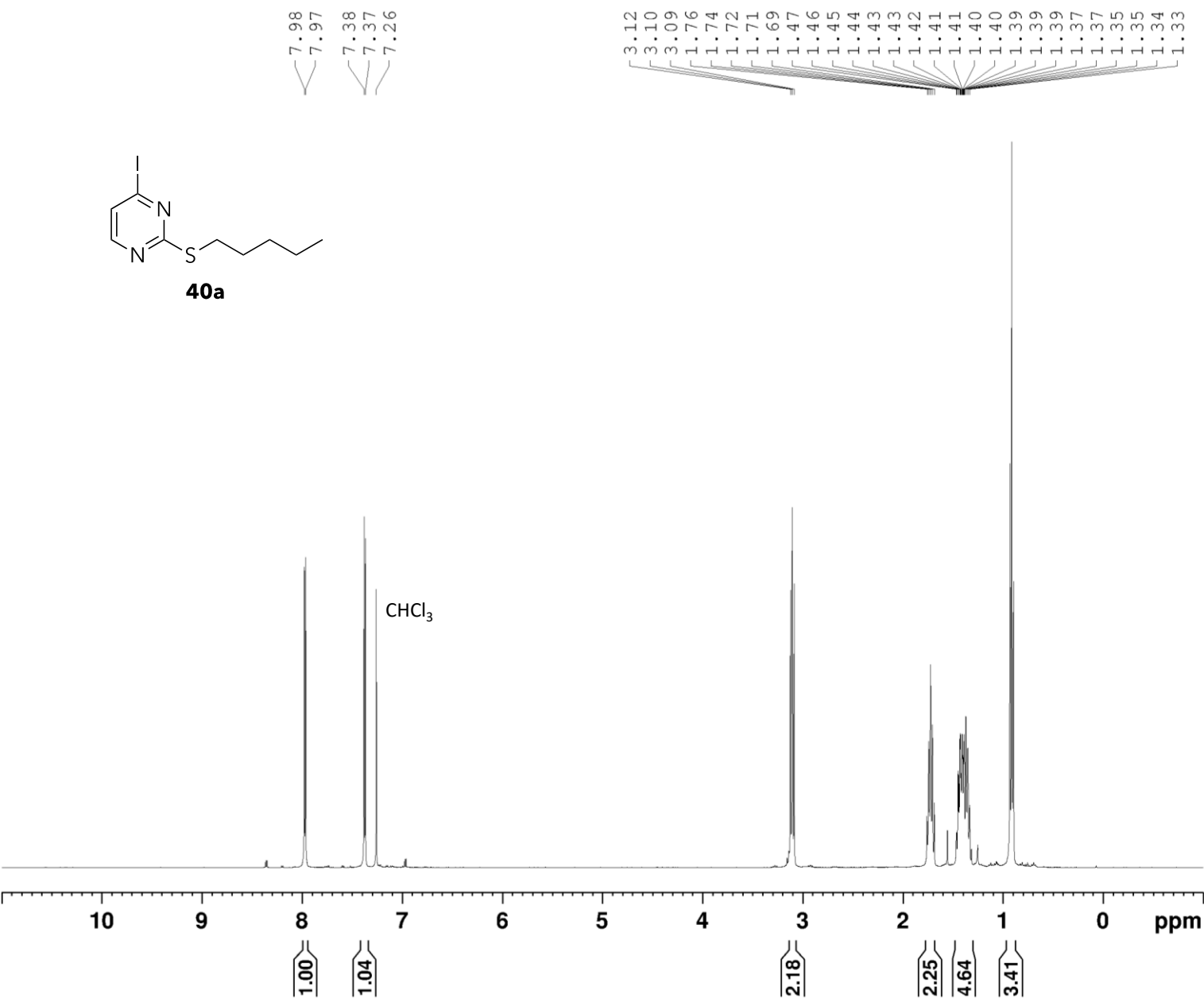
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of 40a



40a

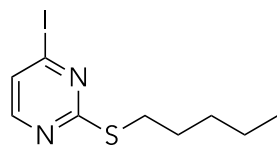


Current Data Parameters
NAME CS-2-39_pure
EXPNO 10
PROCNO 1

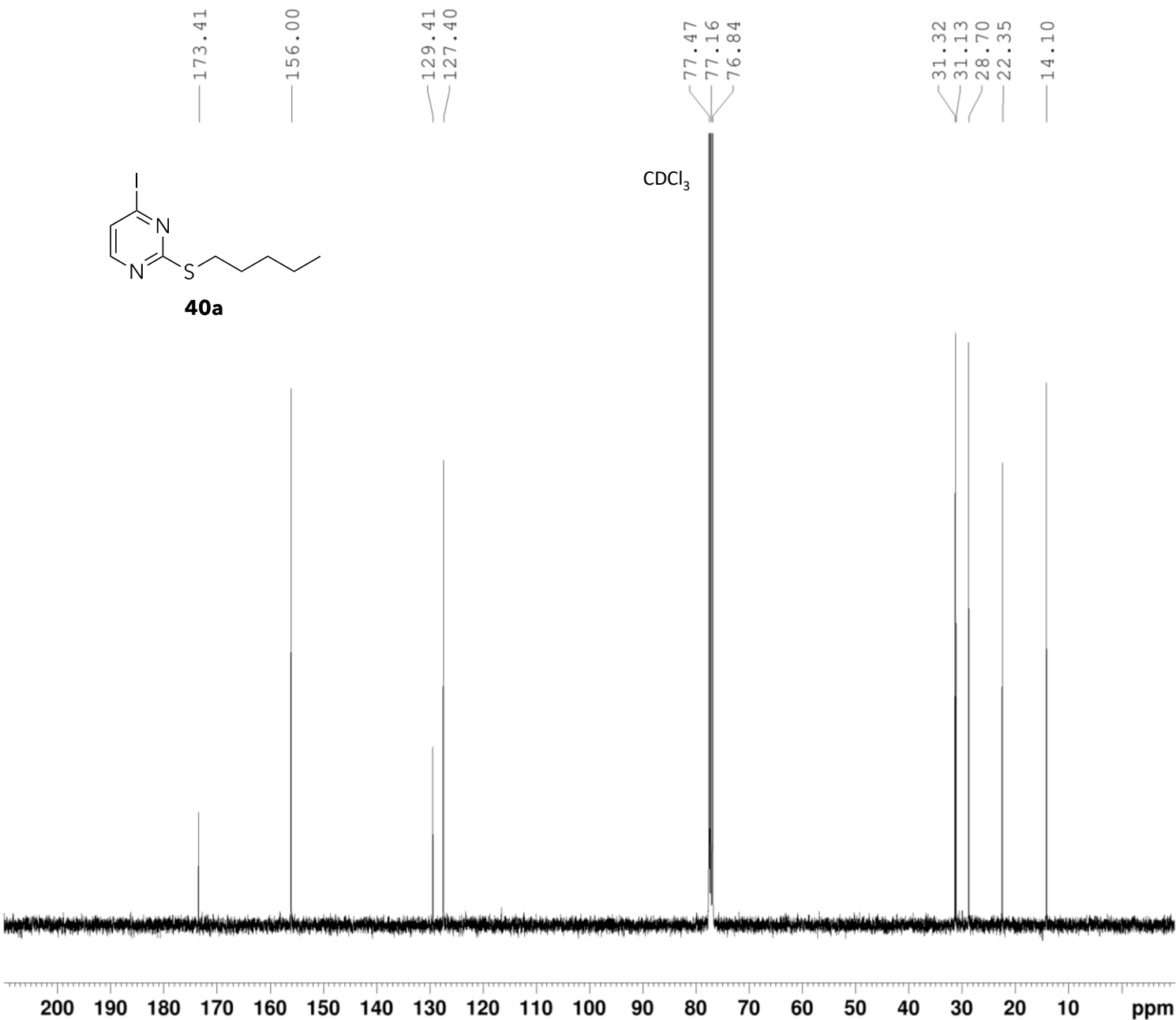
F2 - Acquisition Parameters
Date_ 20231020
Time 16.31 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (PULPROG zg30)
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 40a



40a



Current Data Parameters
NAME 41a_CS-2-39
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231020
Time 20.07 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

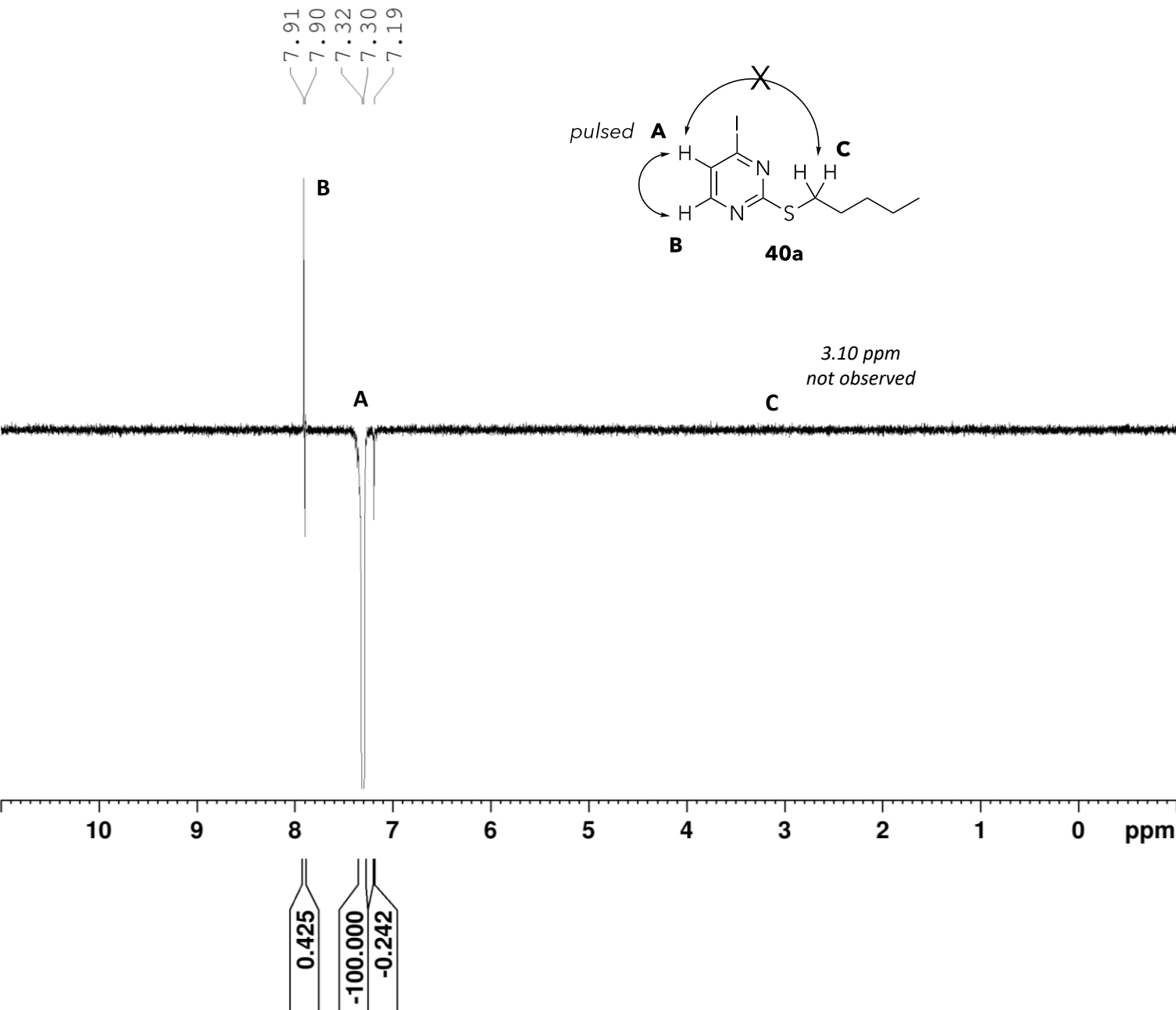
F2 - Processing parameters
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 40a

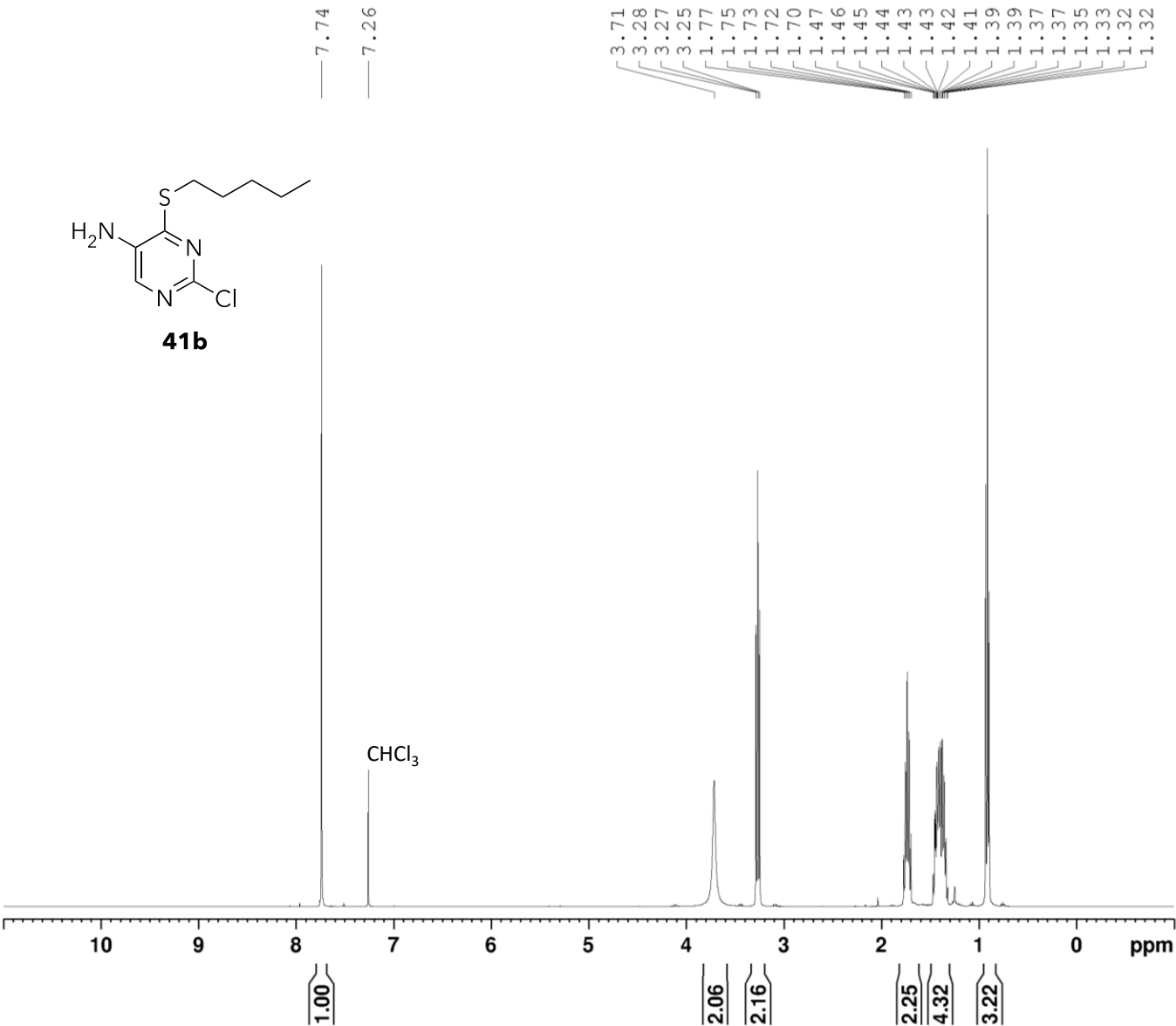
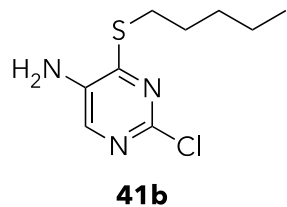
Current Data Parameters
 NAME 41a_CS-2-39
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231024
 Time 11.40 h
 INSTRUM Avance Neo
 PROBHD Z152088_0031 (
 PULPROG selnpgpzs.2
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 10.16 usec
 TE 300.0 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27266601 sec
 D31 0.30000001 sec
 TD0 1
 ZGPTNS -DCALC_SPOFFS
 SFO1 400.1324708 MHz
 NUC1 1H
 CNST21 7.3971410
 P1 8.00 usec
 P12 42599.44 usec
 P32 20000.00 usec
 P44 300.00 usec
 PLW1 24.03499985 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 SPW2 0.00002002 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 SPW29 0.05875600 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 SPW30 1.09389997 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 400.1300368 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



¹H spectrum of **41b**

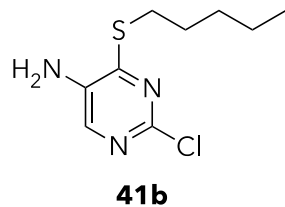


Current Data Parameters
NAME 36a_CS-2-38
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231024
Time 11.23 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (PI HR-BBO400S1-BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SF01 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **41b**



— 157.75
— 149.81
— 140.37
— 136.56

77.48
77.16
76.84

31.04
30.02
28.73
22.28
14.05

CDCl₃

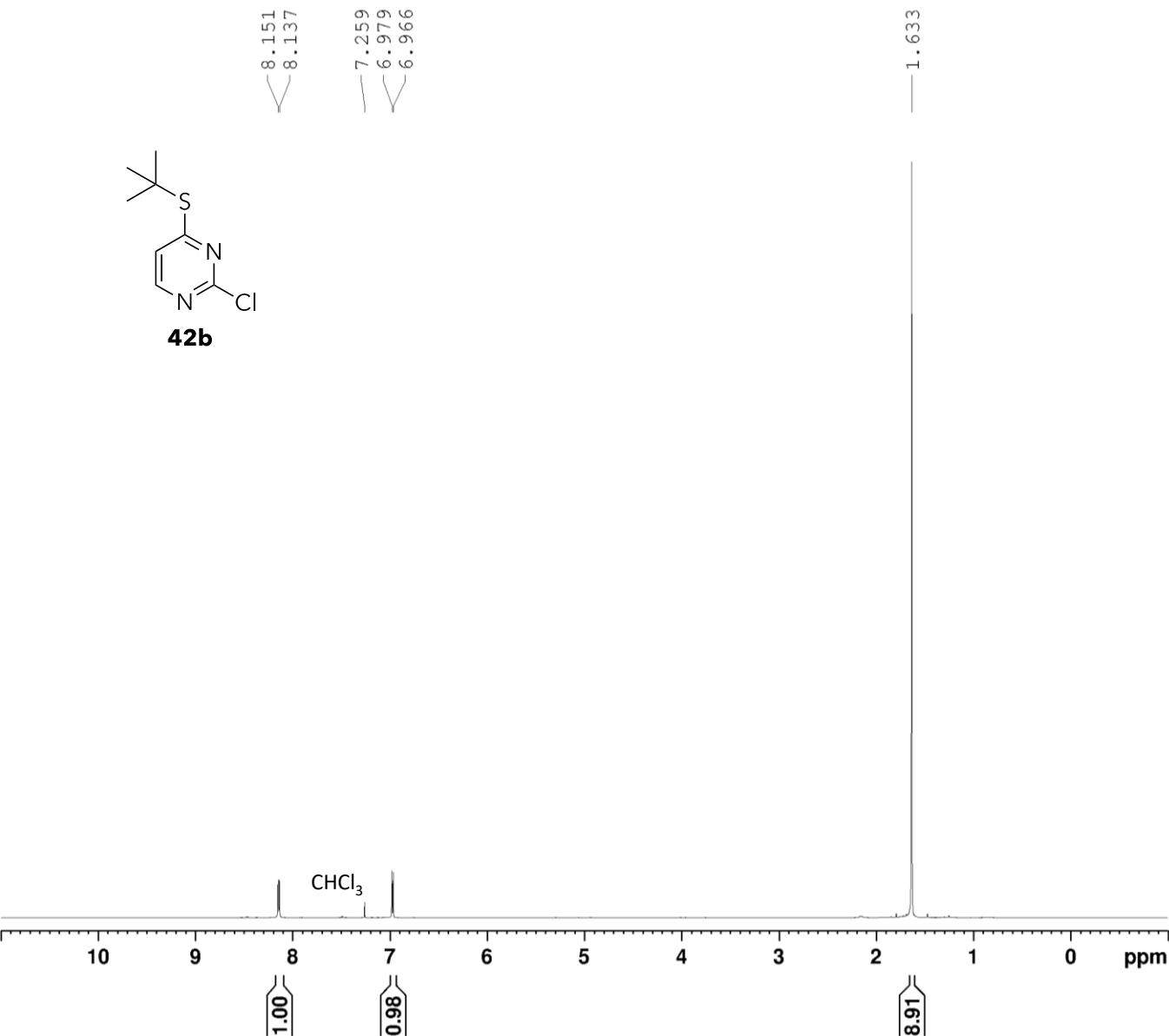
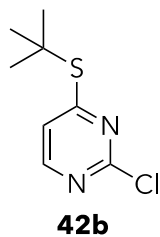
Current Data Parameters
NAME 36a_CS-2-38
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231020
Time 21.15 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (PI HR-BBO400S1-BB)
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

¹H spectrum of **42b**



Current Data Parameters

NAME OJ-7-38-H-2
EXPNO 12
PROCNO 1

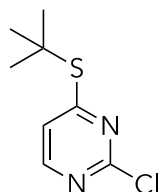
F2 - Acquisition Parameters

Date_ 20240525
Time 16.18 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5.
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

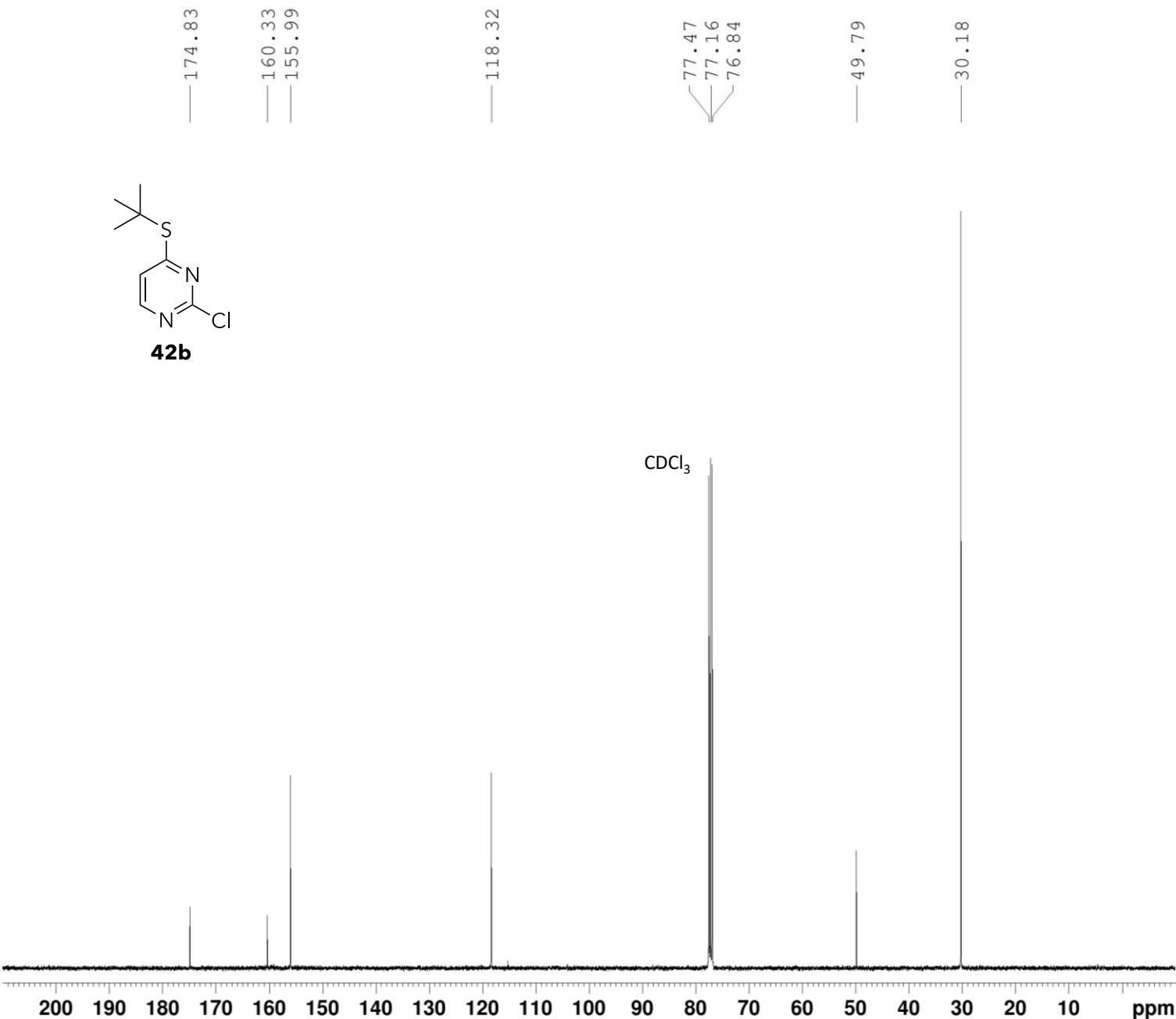
F2 - Processing parameters

SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **42b**



42b



Current Data Parameters
NAME OJ-7-38-C-2
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240525
Time 17.42 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

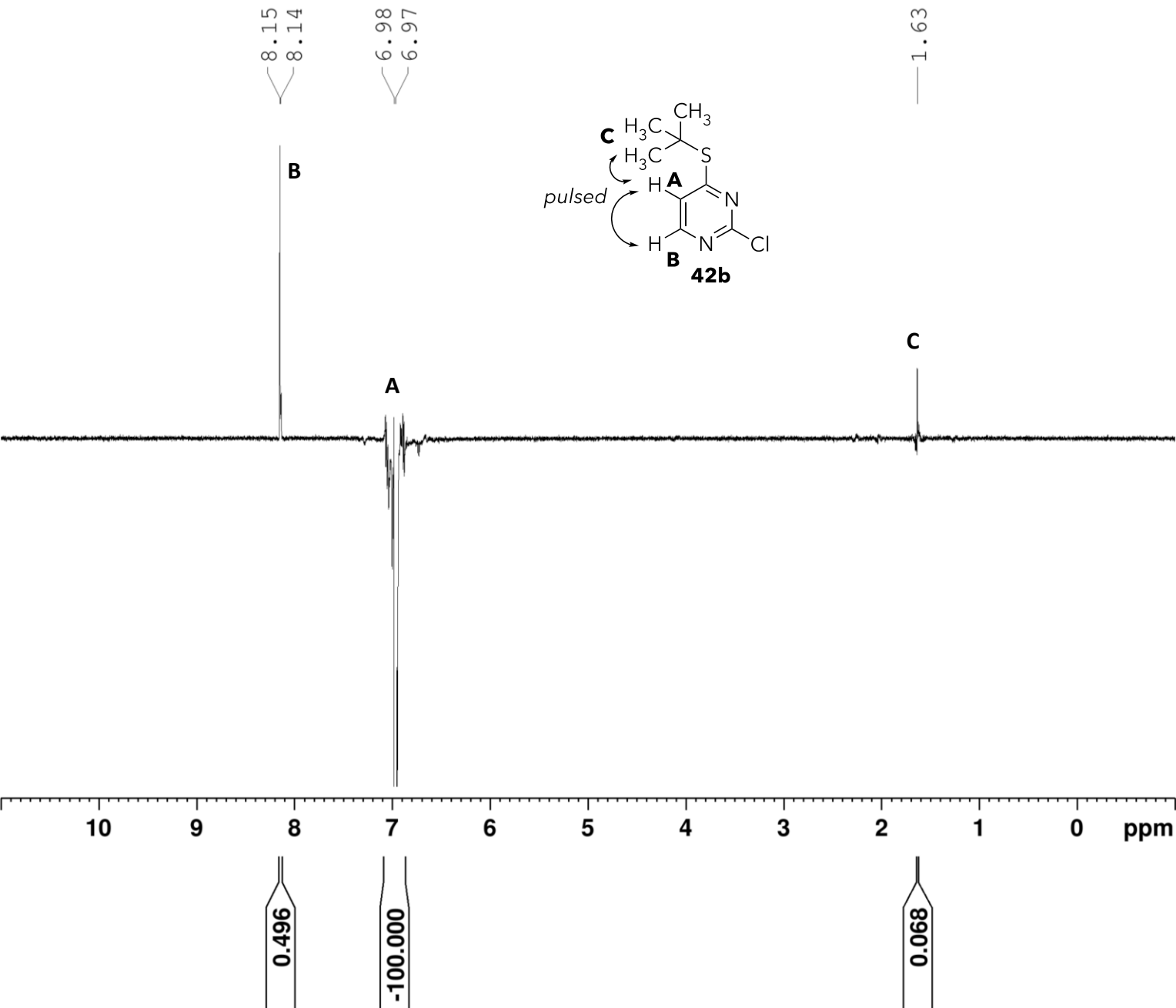
F2 - Processing parameters
SI 32768
SF 100.6127549 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of **42b**

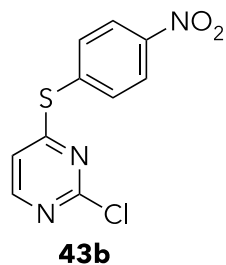
Current Data Parameters
NAME OJ-7-38-NOE
EXPNO 17
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240818
Time 15.08 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnogpzs.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGOTPS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 6.9889188
P1 12.00 usec
P12 83047.54 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 407.15 Hz
SPW2 0.00000564 W
SPNAM[29 Crp60,20,20.1
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

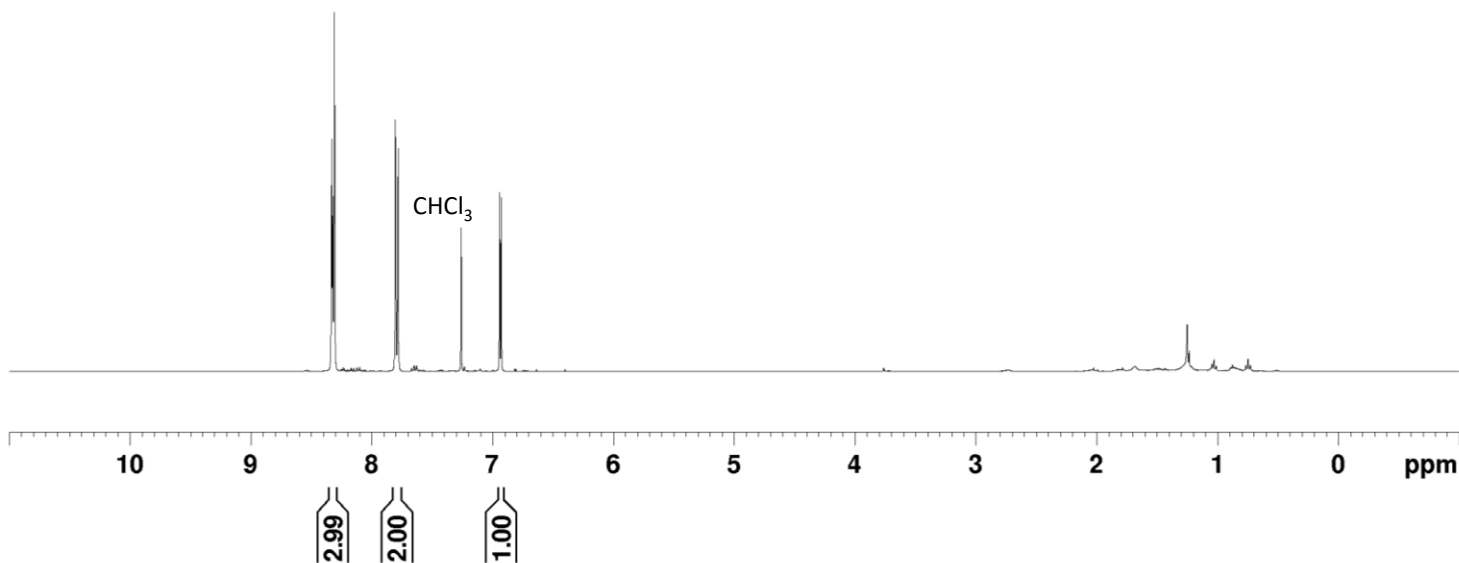
F2 - Processing parameters
SI 32768
SF 500.2300072 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **43b**



8.33
8.31
7.80
7.78
7.26
6.94
6.93

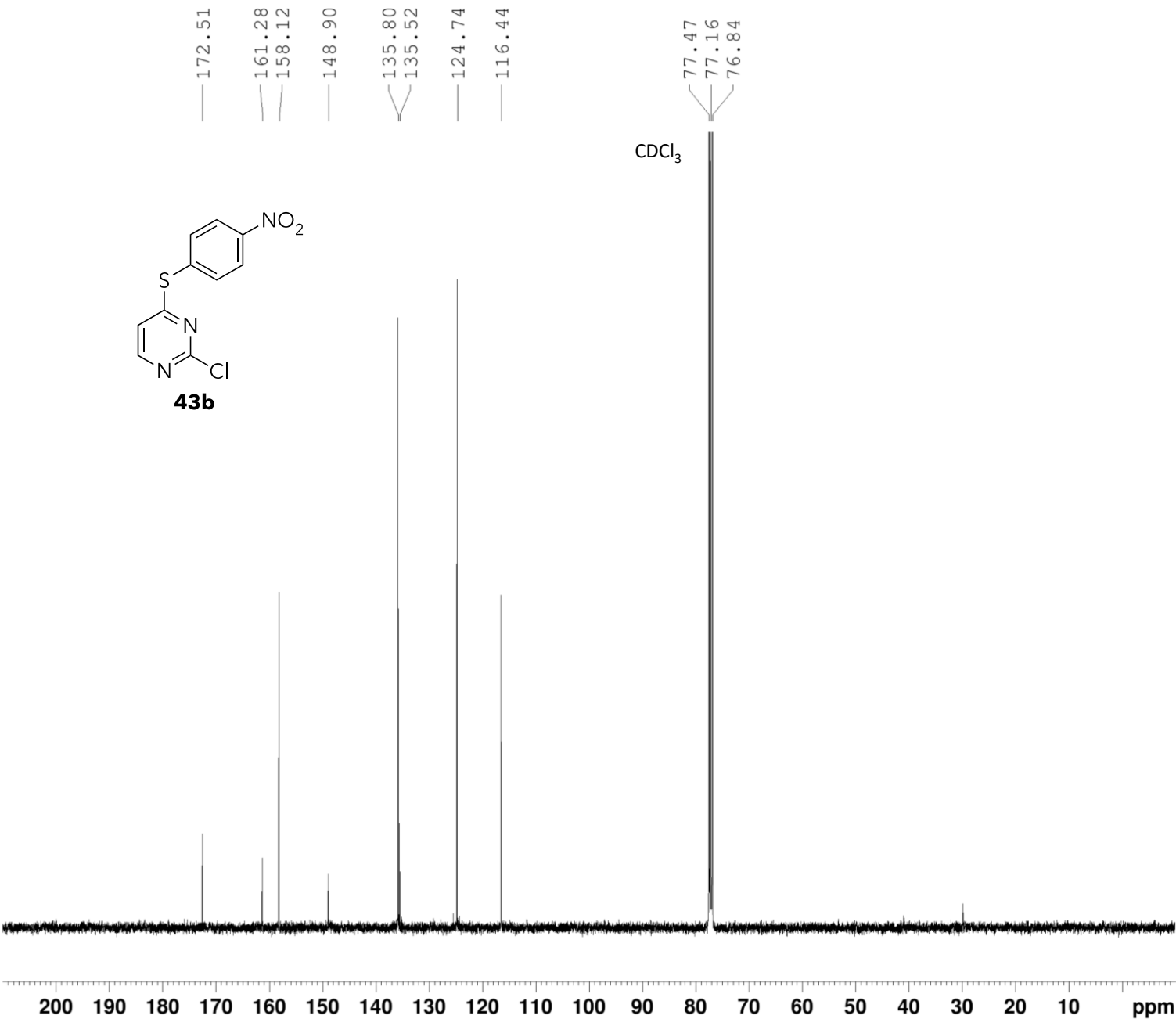
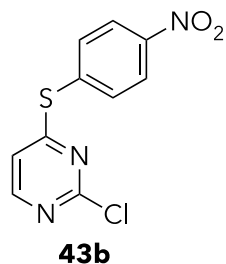


Current Data Parameters
NAME CA-1-34-H
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231229
Time 17.04 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **43b**



Current Data Parameters
NAME CA-1-34-C
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231229
Time 19.04 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

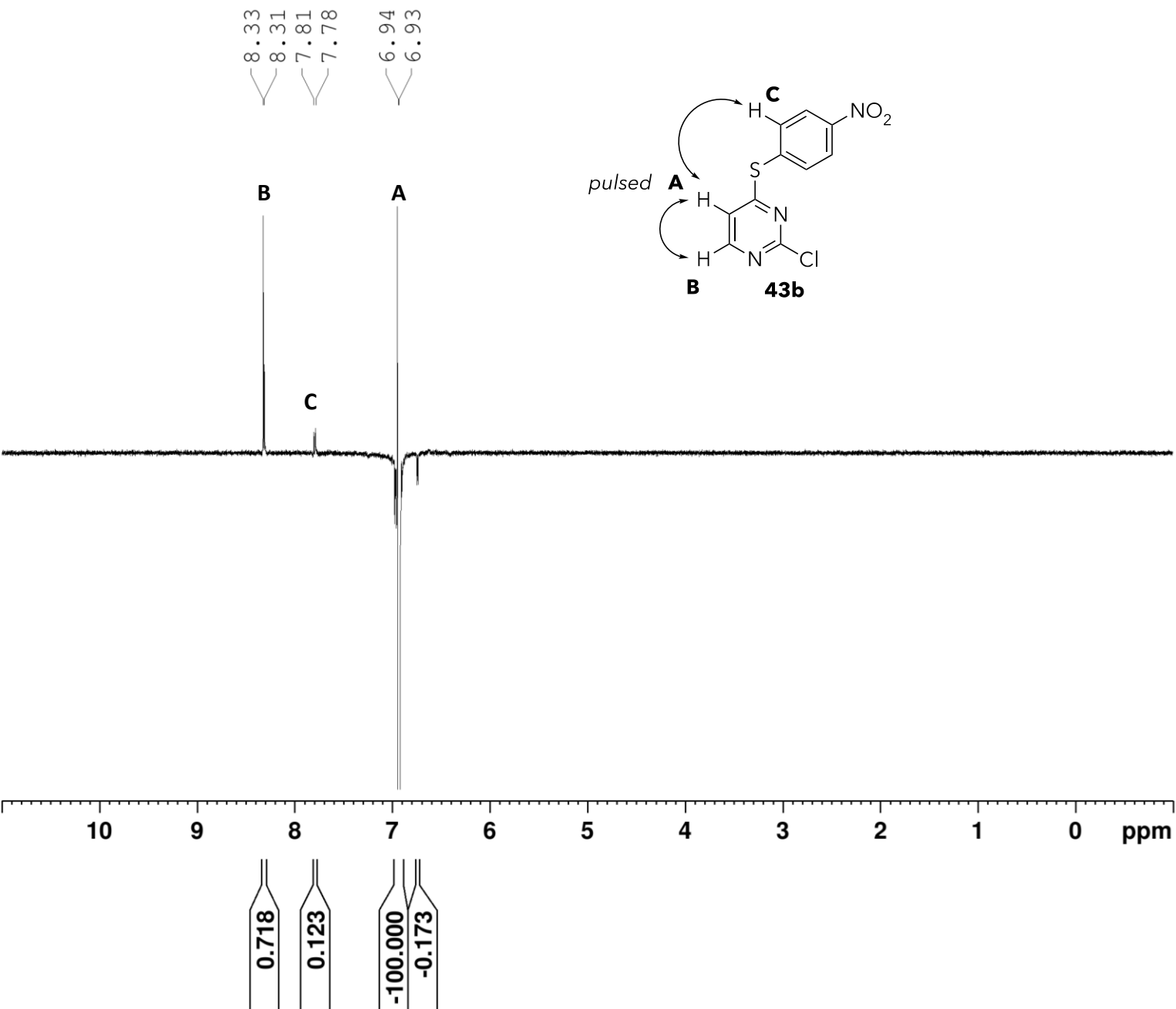
F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 43b

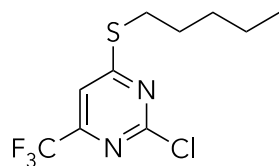
Current Data Parameters
NAME CA-13-34-NOE-1
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231211
Time 19.54 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnpgps.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPGTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 6.9569860
P1 12.00 usec
P12 60252.17 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 391.17 Hz
SPW2 0.00001072 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

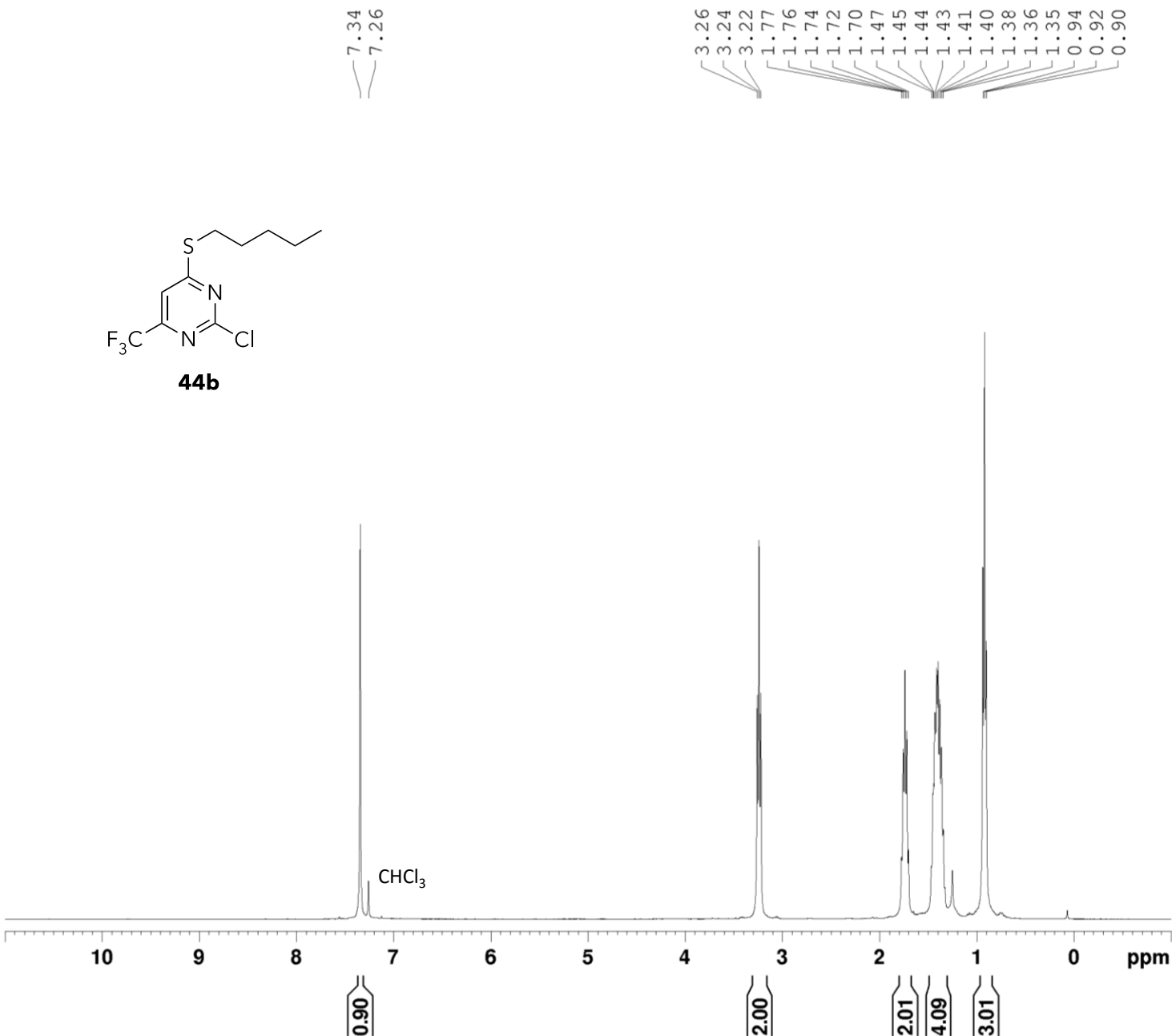
F2 - Processing parameters
SI 32768
SF 500.2300119 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **44b**



44b

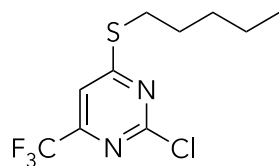


Current Data Parameters
NAME CA-1-32-H
EXPNO 13
PROCNO 1

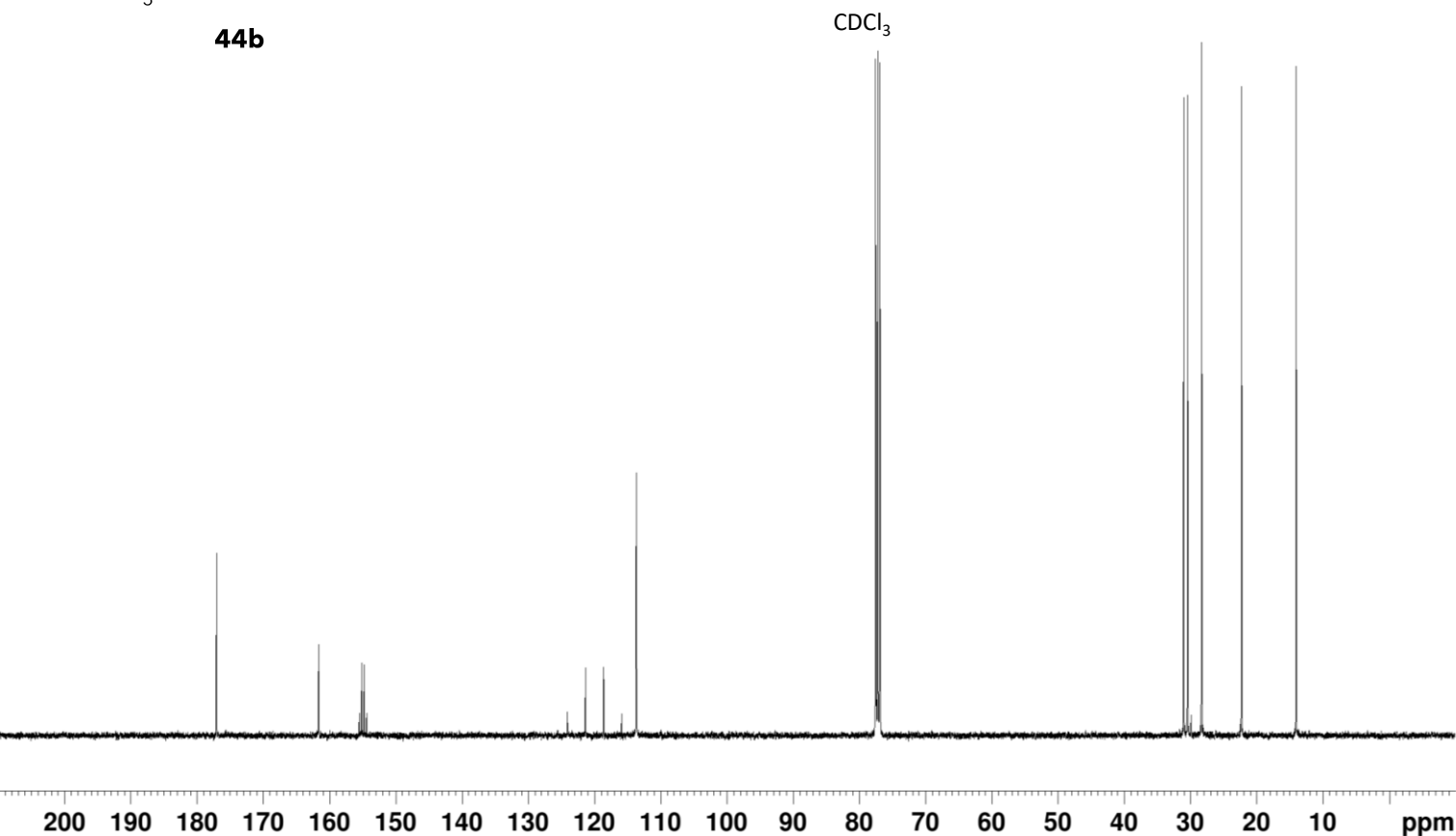
F2 - Acquisition Parameters
Date_ 20231111
Time 19.26 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.89 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 2.67 usec
P1 8.00 usec
PLW1 24.03499985 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of **44b**



44b

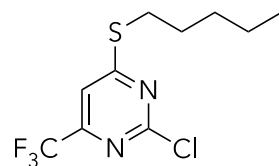


Current Data Parameters
NAME CA-1-32-C
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231111
Time 21.26 h
INSTRUM Avance Neo
PROBHD Z152088_0031 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 2.67 usec
P1 8.00 usec
PLW1 86.55400085 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 24.03499985 W
PLW12 0.18990999 W
PLW13 0.09552100 W

F2 - Processing parameters
SI 32768
SF 100.6127540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹F spectrum of **44b**



44b

C₆H₅F

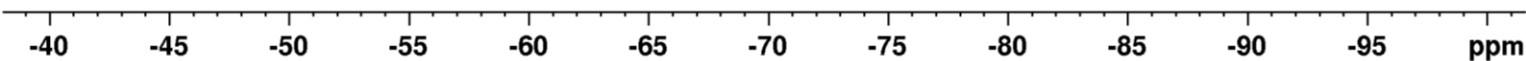
-63.72

-71.08

Current Data Parameters
NAME CA-1-32-F-trifluorotoluene
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240528
Time 11.28 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg
TD 131072
SOLVENT CDC13
NS 16
DS 4
SWH 90909.091 Hz
FIDRES 1.387163 Hz
AQ 0.7208960 sec
RG 101
DW 5.500 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 12.00 usec
PLW1 38.11899948 W

F2 - Processing parameters
SI 65536
SF 376.4987157 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

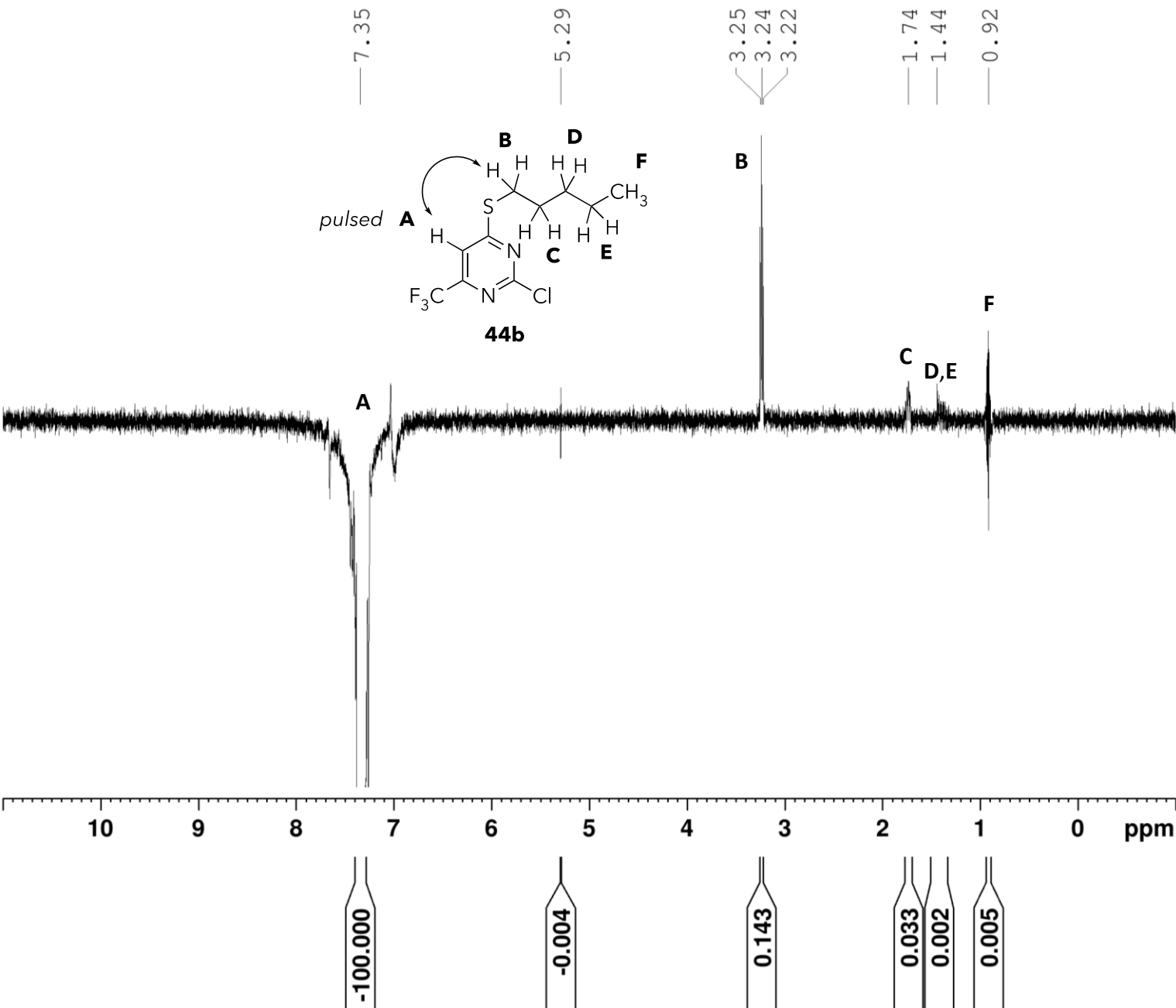


1D NOE of 44b

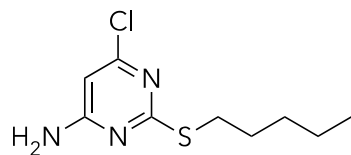
Current Data Parameters
NAME CA-1-32-NOE
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231211
Time 18.14 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnogpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 190.44
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 7.3651209
P1 12.00 usec
P12 43393.07 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 595.33 Hz
SPW2 0.00002067 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

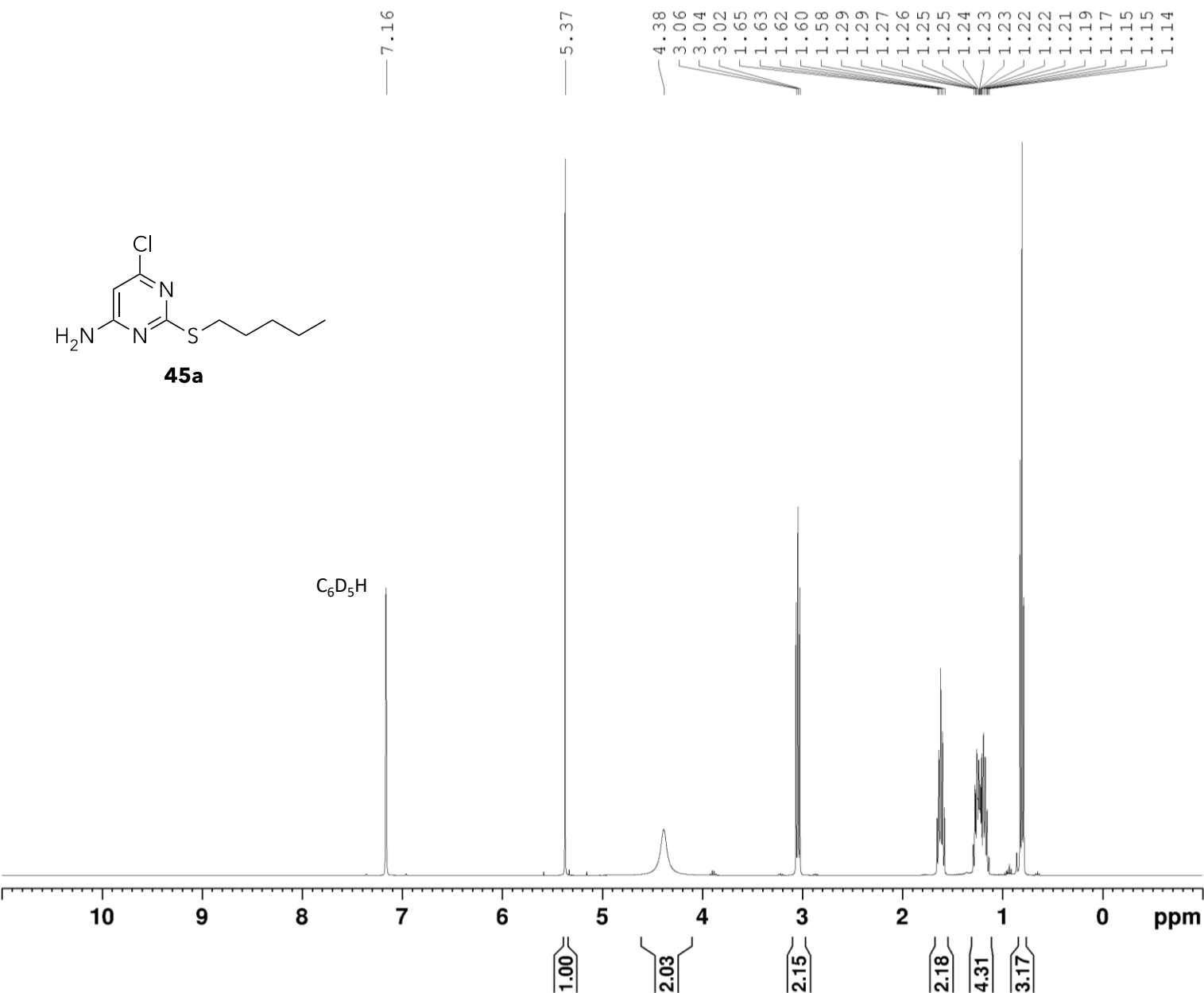
F2 - Processing parameters
SI 32768
SF 500.2300134 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of 45a



45a



NAME CS-1-143_pure_moreconc
EXPNO 10
PROCNO 1

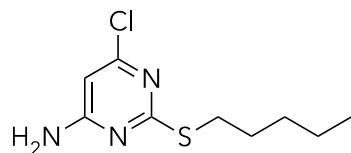
F2 - Acquisition Parameters

Date_ 20230508
Time_ 17.44 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (zgc30)
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 71.8
DW 61.000 usec
DE 13.54 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
SF01 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

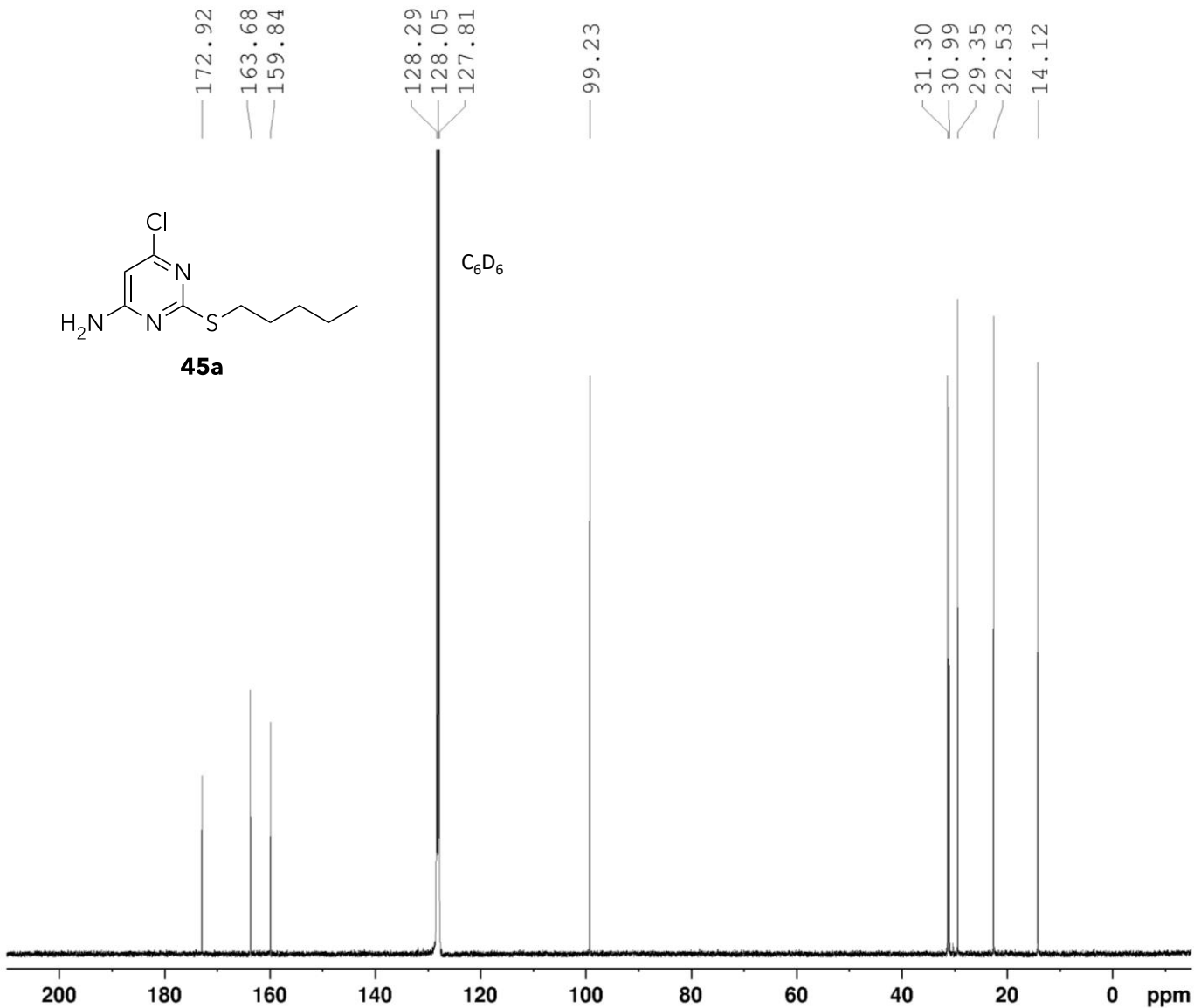
F2 - Processing parameters

SI 65536
SF 400.1299977 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 45a



45a

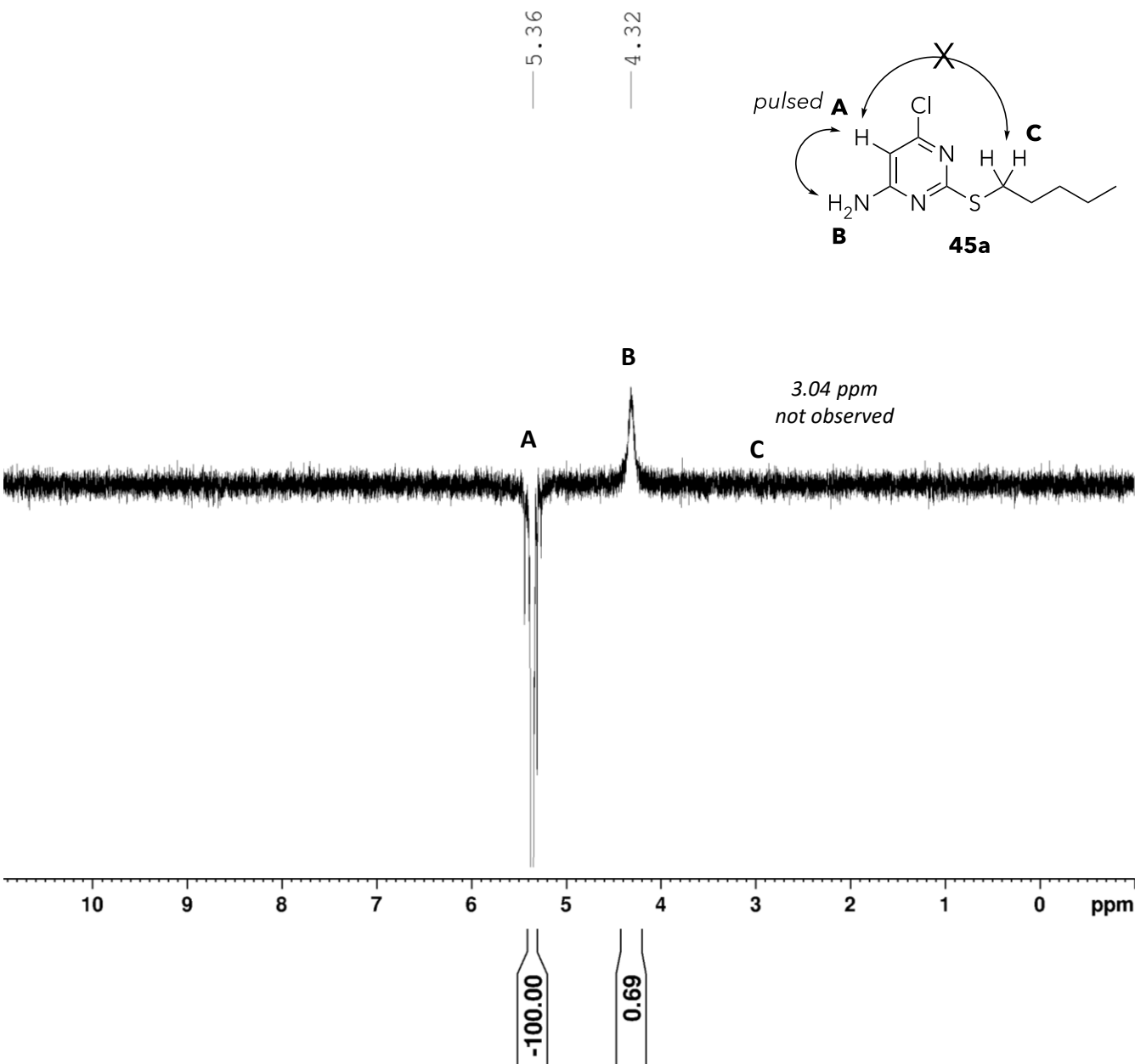


Current Data Parameters
NAME 45a_CS-2-8
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231218
Time 20.03 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

F2 - Processing parameters
SI 32768
SF 100.6127392 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 45a

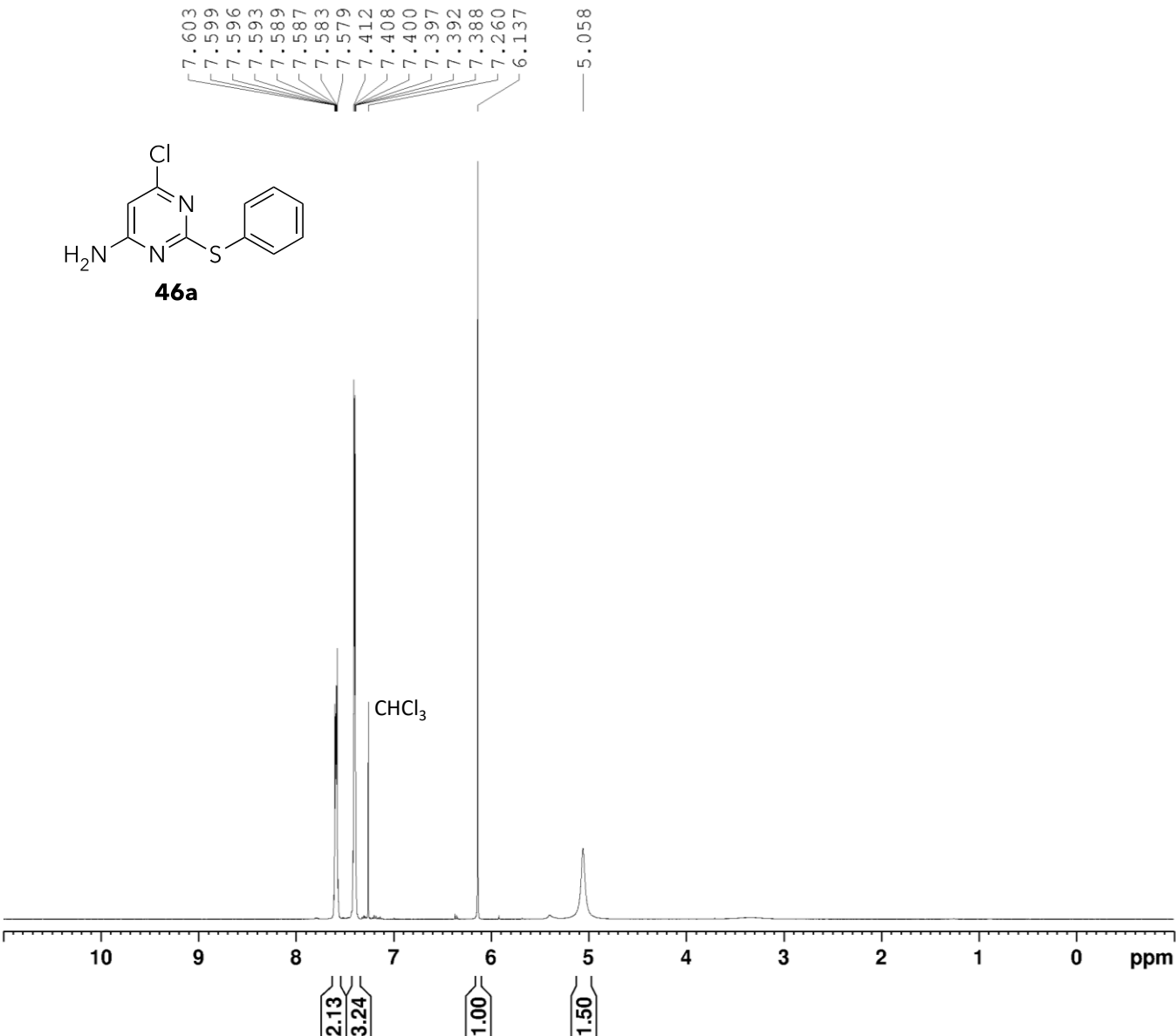
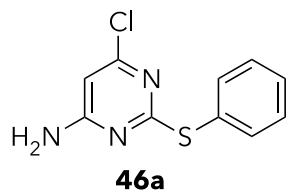


Current Data Parameters
 NAME 45a_CS-2-8
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231215
 Time 11.26 h
 INSTRUM Avance Neo
 PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D)
 PULPROG selnpgps.2
 TD 65536
 SOLVENT C6D6
 NS 32
 DS 4
 SWH 8196.721 Hz
 FIDRES 0.250144 Hz
 AQ 3.9976959 sec
 RG 101
 DW 61.000 usec
 DE 8.88 usec
 TE 300.2 K
 D1 2.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 D20 0.27266601 sec
 D31 0.30000001 sec
 TD0 1
 ZGPGTNS -DCALC_SPOFFS
 SF01 400.1324708 MHz
 NUC1 1H
 CNST21 5.3376250
 P1 10.00 usec
 P12 26479.37 usec
 P32 20000.00 usec
 P44 300.00 usec
 PLW1 22.53199959 W
 SPNAM[2] Gaus1_180r.1000
 SPOAL2 0.500
 SPW2 0.00007588 W
 SPNAM[29] Crp60,20,20.10
 SPOAL29 0.500
 SPW29 0.08606600 W
 SPNAM[30] Bip720,50,20.1
 SPOAL30 0.500
 SPW30 1.60230005 W
 GPZ0 11.00 %
 GPNAM[1] SMSQ10.100
 GPZ1 15.00 %
 GPNAM[2] SMSQ10.100
 GPZ2 40.00 %
 P16 1000.00 usec

F2 - Processing parameters
 SI 32768
 SF 400.1299966 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

¹H spectrum of 46a



Current Data Parameters

NAME OJ-6-118-H-2
EXPNO 13
PROCNO 1

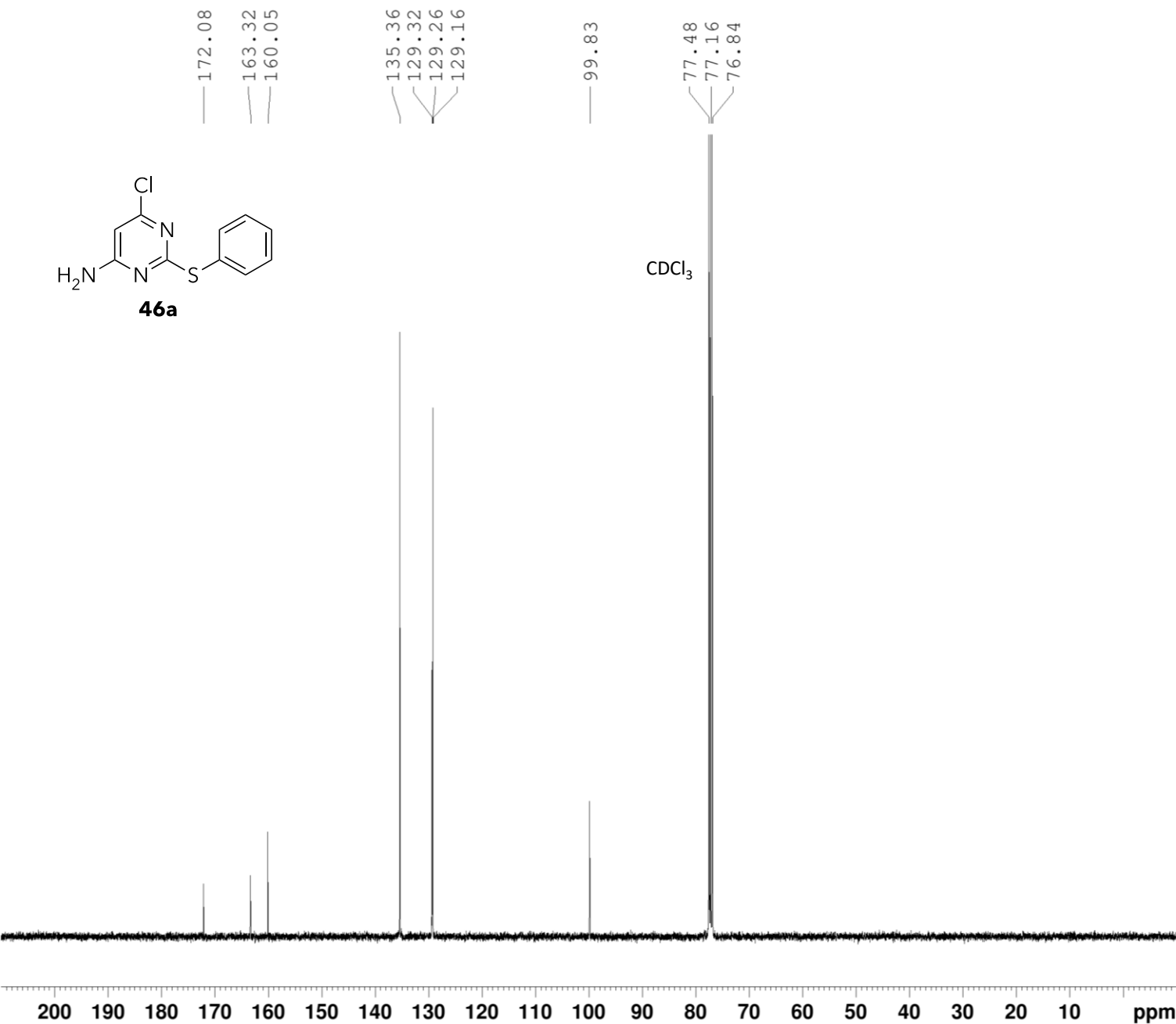
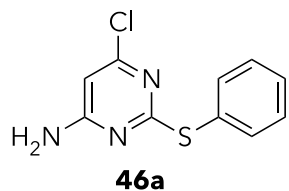
F2 - Acquisition Parameters

Date_ 20240202
Time 14.27 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P/D-5.0
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 46a



Current Data Parameters
NAME OJ-6-118-C
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240202
Time 15.27 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23809.524 Hz
FIDRES 0.726609 Hz
AQ 1.3762560 sec
RG 8
DW 21.000 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.91699982 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 90.00 usec
PLW2 22.53199959 W
PLW12 0.27818000 W
PLW13 0.13992000 W

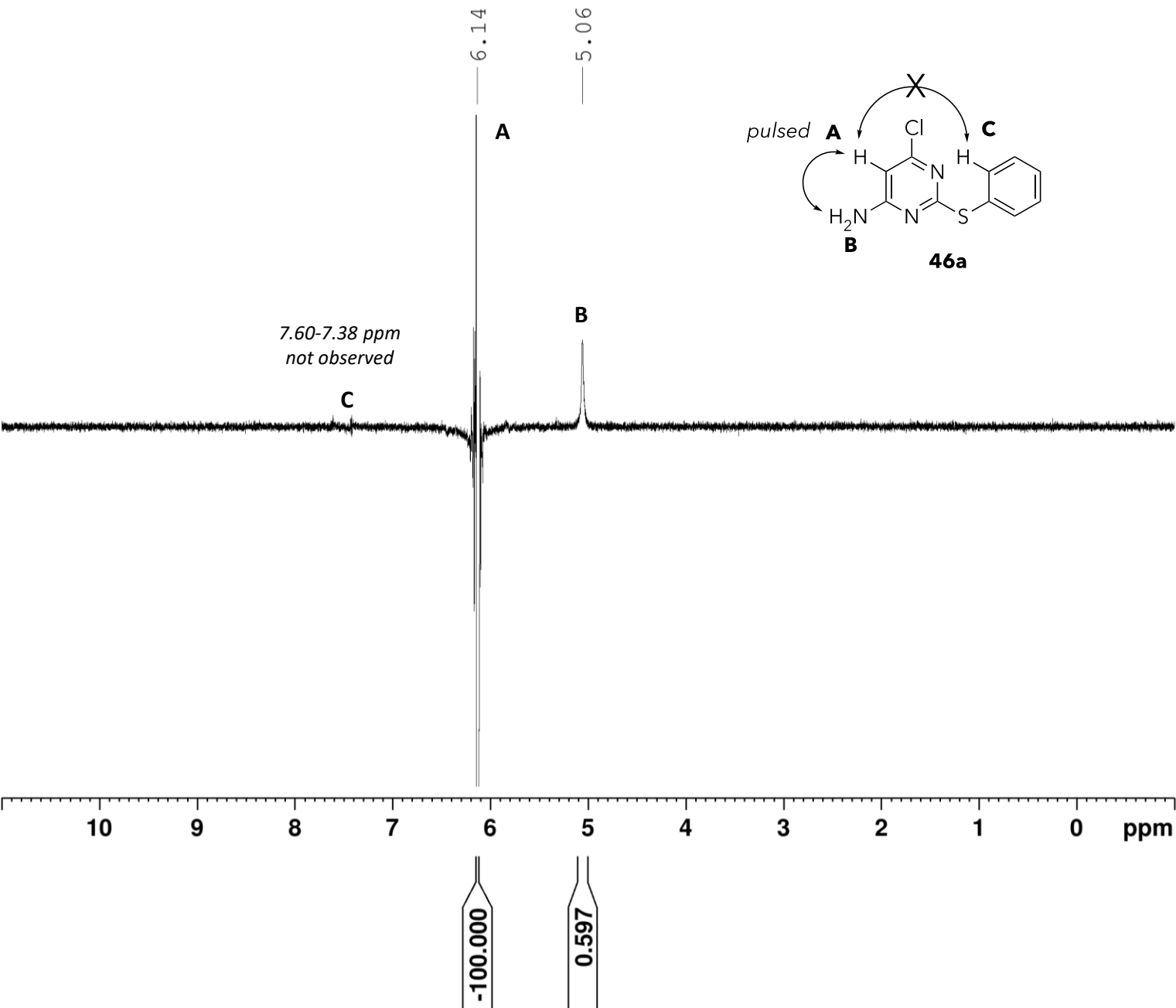
F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 46a

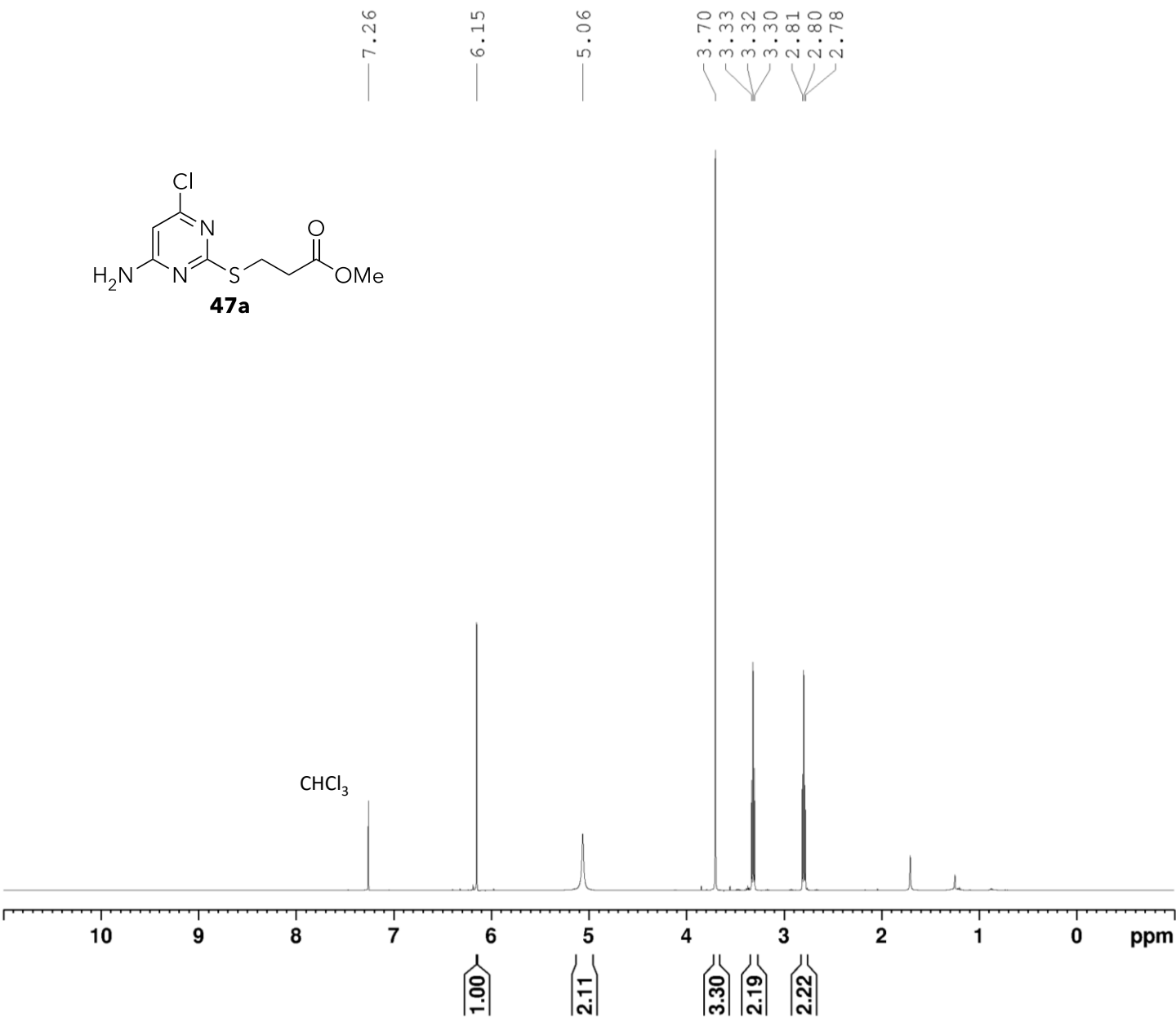
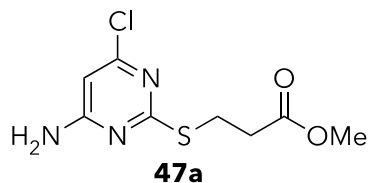
Current Data Parameters
NAME OJ-6-118-1-NOE
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240129
Time 20.01 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggpzs.2
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 171.72
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SF01 500.2330889 MHz
NUC1 1H
CNST21 6.1254139
P1 12.00 usec
P12 89249.50 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gaus1_180r.1000
SPOAL2 0.500
spoffs2 -24.80 Hz
SPW2 0.00000489 W
SPNAM[29 Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30 Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2299953 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of **47a**

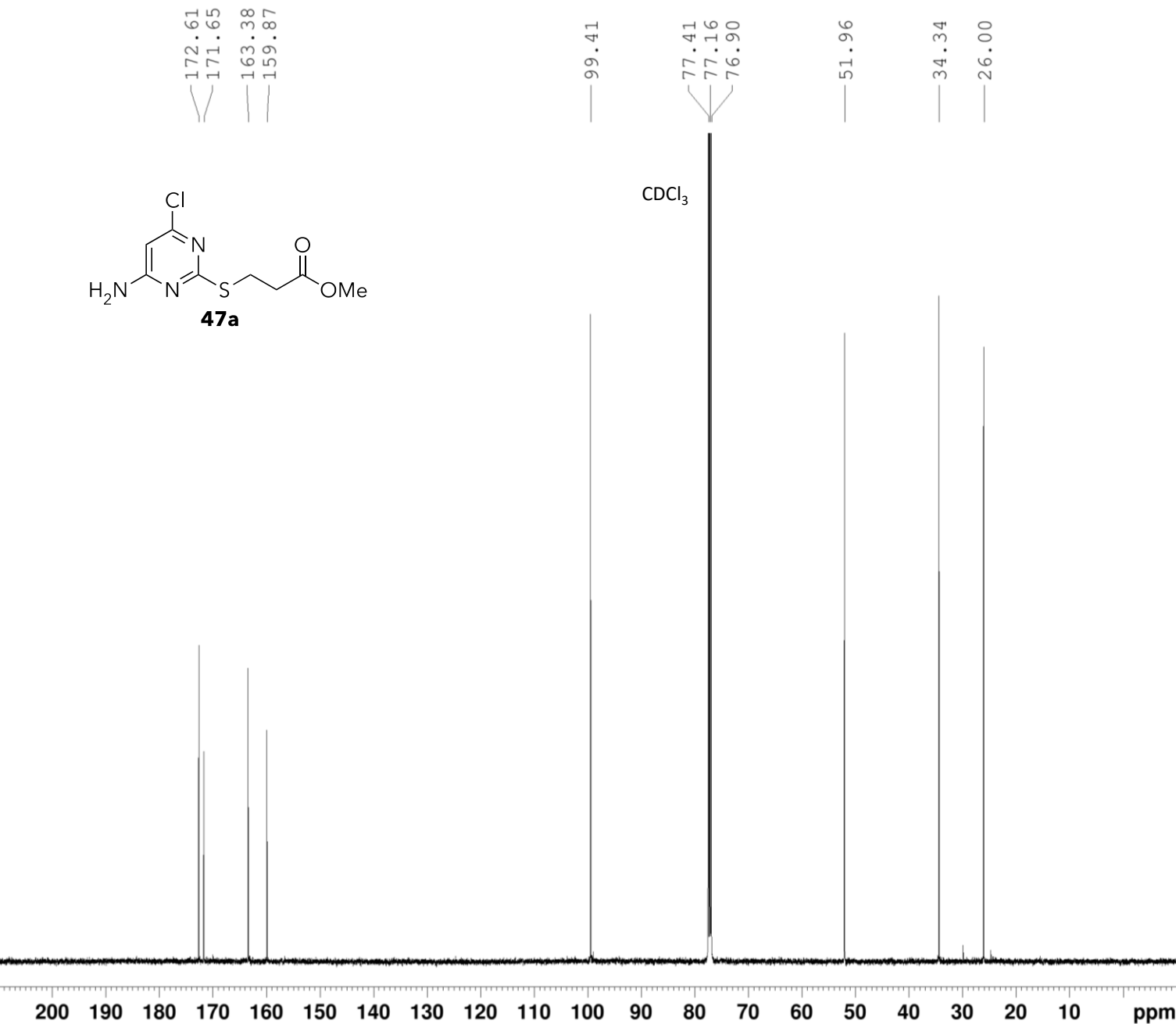
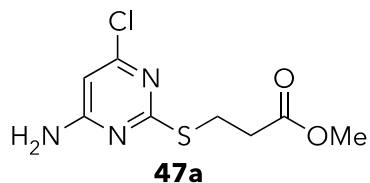


Current Data Parameters
NAME OJ-5-171
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230821
Time 12.53 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 16.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.2330889 MHz
NUC1 1H
P0 4.00 usec
P1 12.00 usec
PLW1 11.44699955 W

F2 - Processing parameters
SI 65536
SF 500.2300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of 47a



Current Data Parameters
NAME OJ-5-171-C
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230821
Time 14.51 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 190.44
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7955118 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 56.90299988 W
SFO2 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 11.44699955 W
PLW12 0.25756001 W
PLW13 0.12955000 W

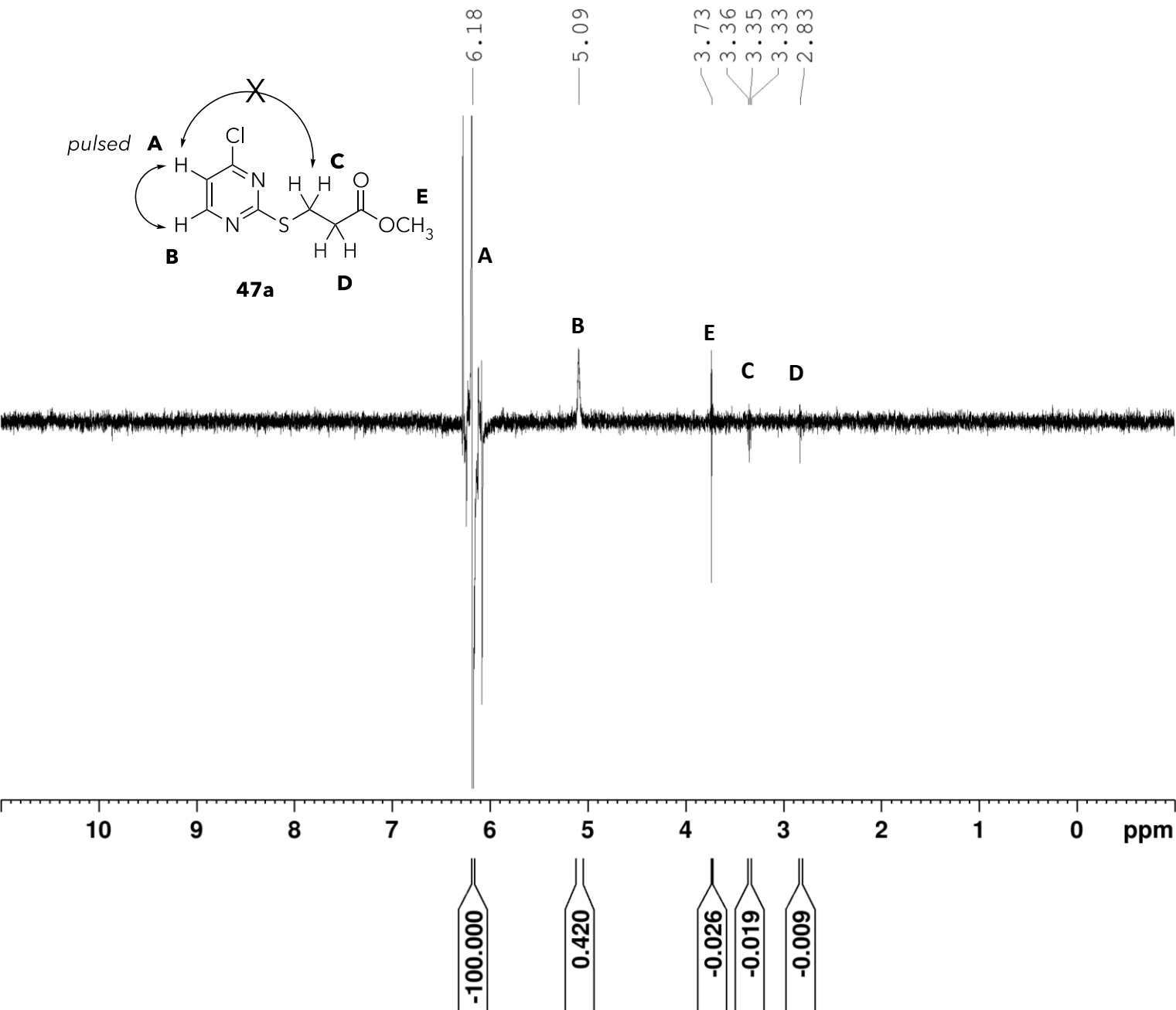
F2 - Processing parameters
SI 32768
SF 125.7829182 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NOE of 47a

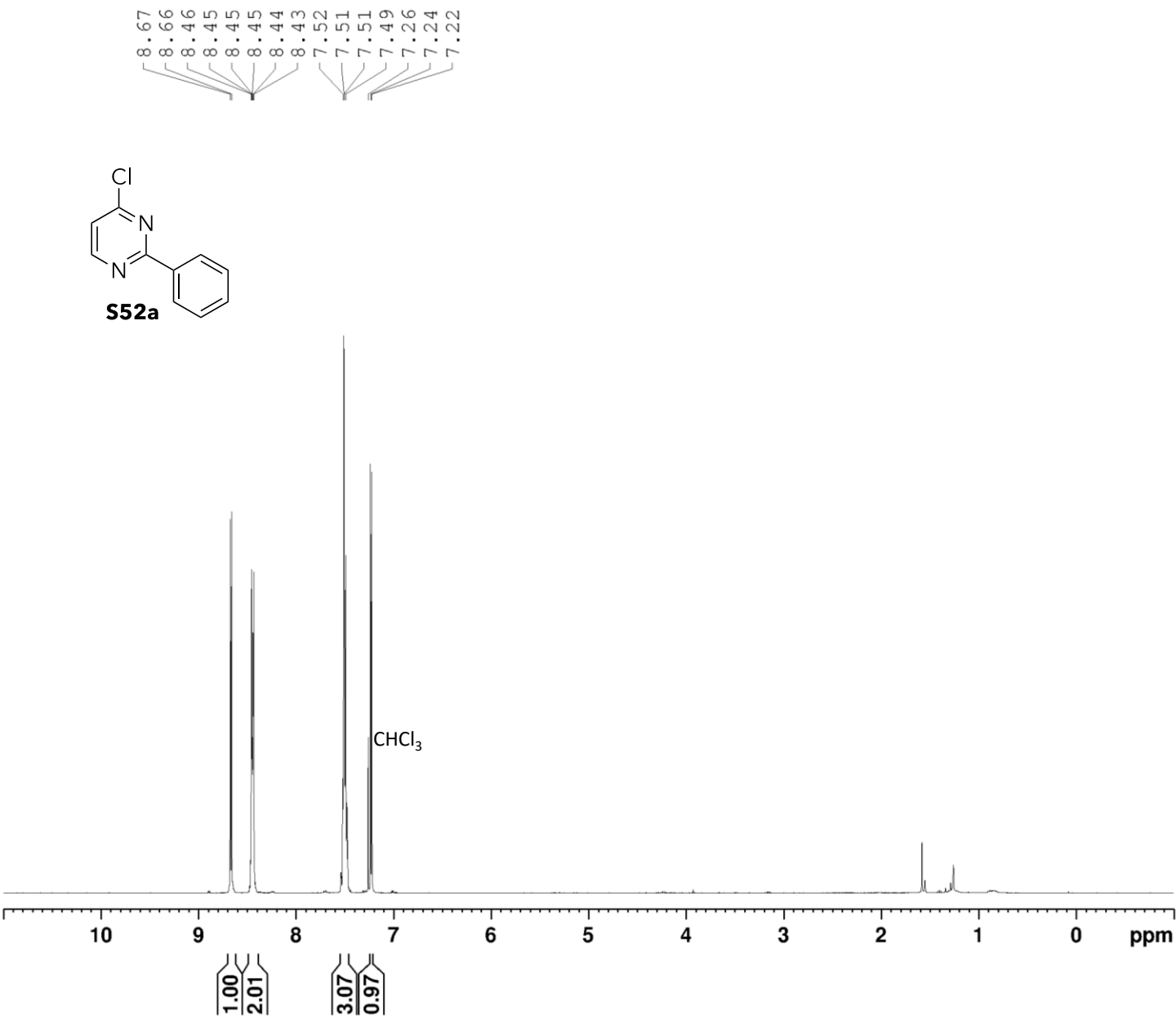
Current Data Parameters
NAME OJ-5-171-NOE-2
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230821
Time 16.44 h
INSTRUM spect
PROBHD Z125869_0055 (
PULPROG selnoggps.2
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 151.18
DW 50.000 usec
DE 20.46 usec
TE 300.0 K
D1 2.00000000 sec
D8 0.30000001 sec
D16 0.00020000 sec
D20 0.27272600 sec
D31 0.30000001 sec
TD0 1
ZGPTNS -DCALC_SPOFFS
SFO1 500.2330889 MHz
NUC1 1H
CNST21 6.1730781
P1 12.00 usec
P12 121103.80 usec
P32 20000.00 usec
P44 240.00 usec
PLW1 11.44699955 W
SPNAM[2] Gausl_180r.1000
SPOAL2 0.500
spoffs2 -0.96 Hz
SPW2 0.00000265 W
SPNAM[29] Crp60,20,20.10
SPOAL29 0.500
spoffs29 0 Hz
SPW29 0.06296300 W
SPNAM[30] Bip720,50,20.1
SPOAL30 0.500
spoffs30 0 Hz
SPW30 1.83150005 W
GPZ0 11.00 %
GPNAM[1] SMSQ10.100
GPZ1 15.00 %
GPNAM[2] SMSQ10.100
GPZ2 40.00 %
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.2300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



¹H spectrum of S52a



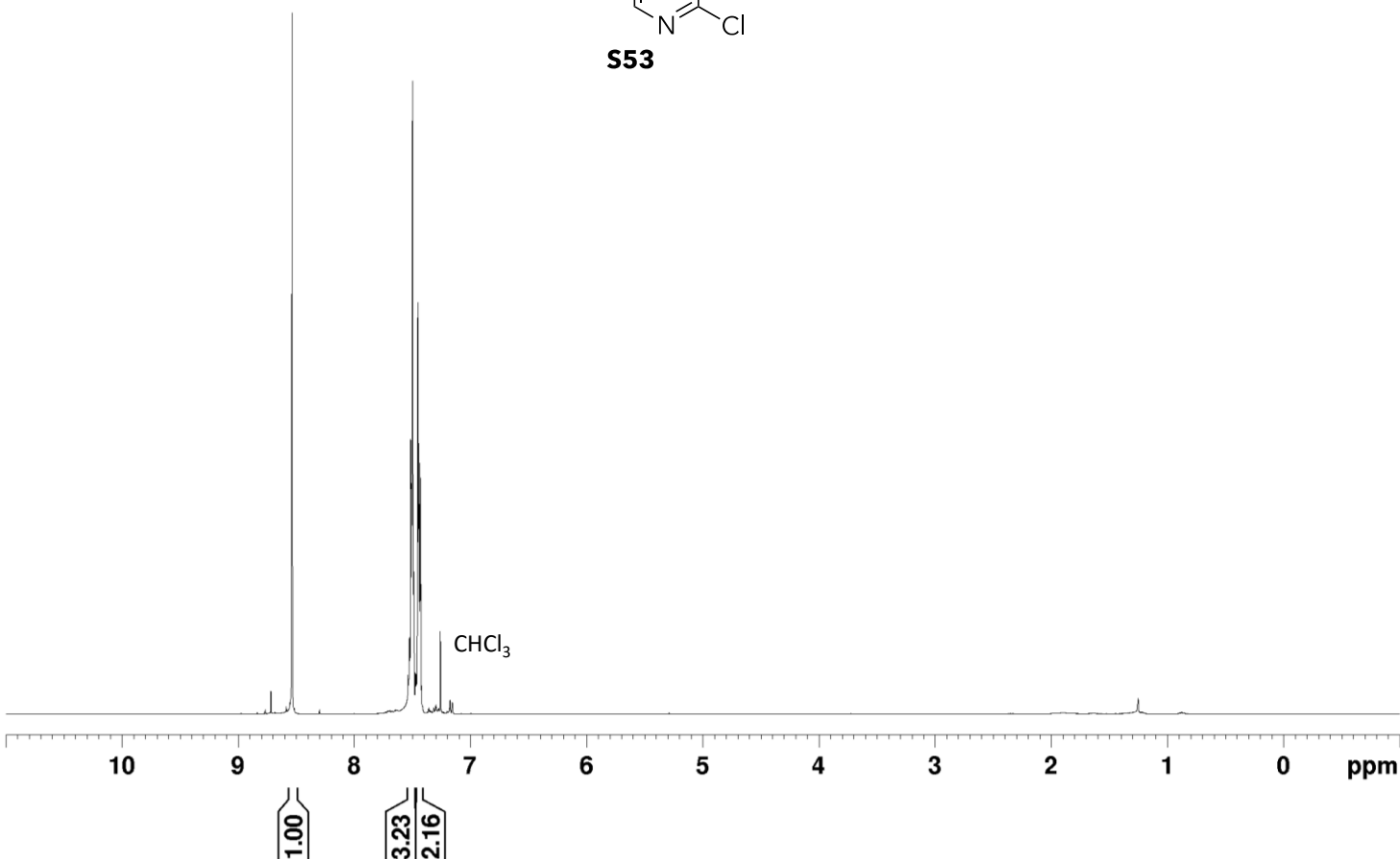
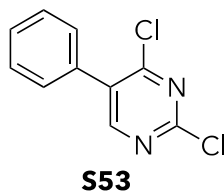
Current Data Parameters
NAME OJ-240821-2-2-H
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240824
Time 20.49 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H spectrum of S53

8.54
7.53
7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.49
7.45
7.45
7.44
7.43
7.43
7.26

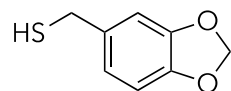


Current Data Parameters
NAME CS-2-21-H
EXPNO 10
PROCNO 1

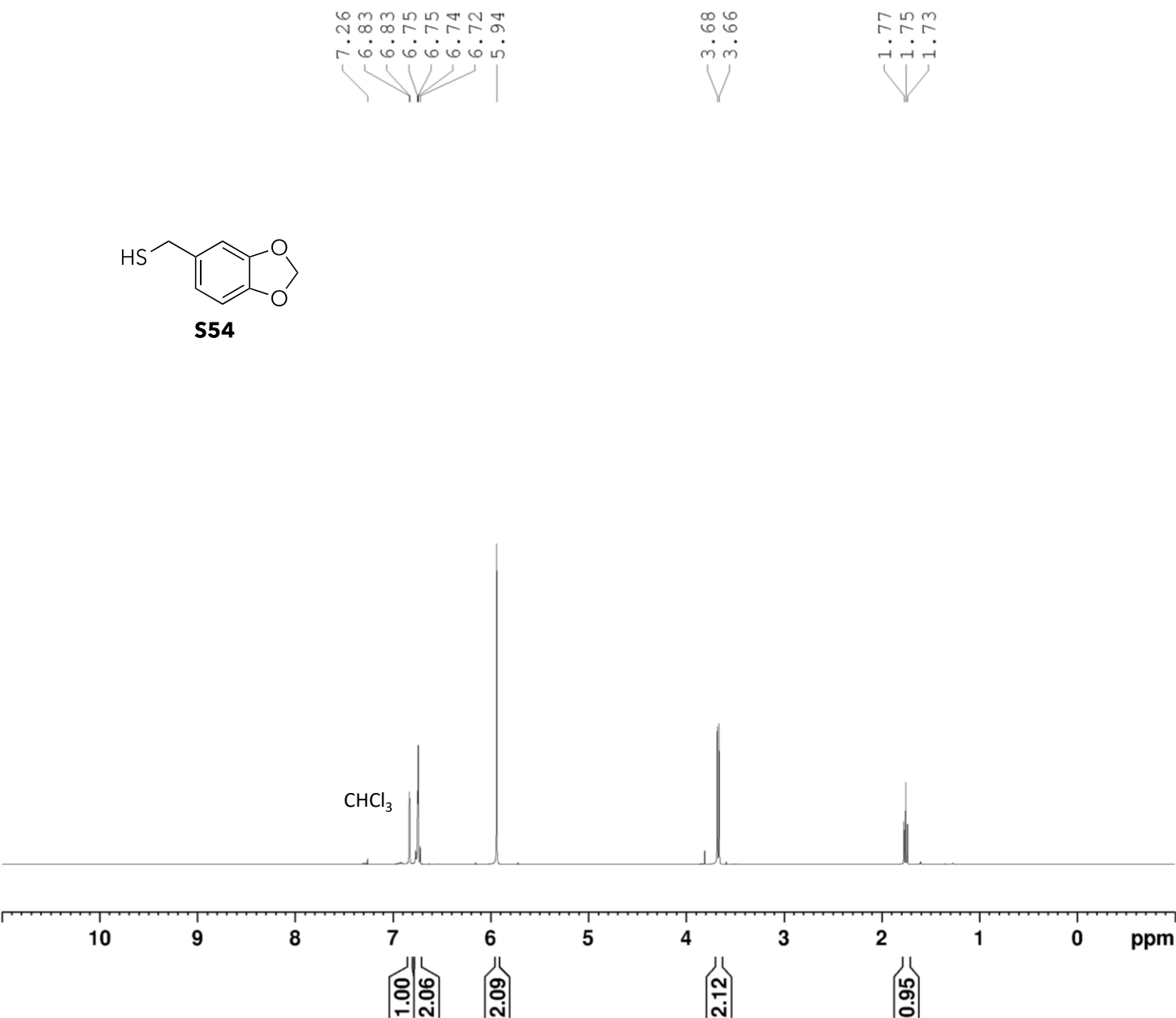
F2 - Acquisition Parameters
Date_ 20240212
Time 16.10 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H spectrum of S54



S54



Current Data Parameters

NAME OJ-7-33-H
EXPNO 13
PROCNO 1

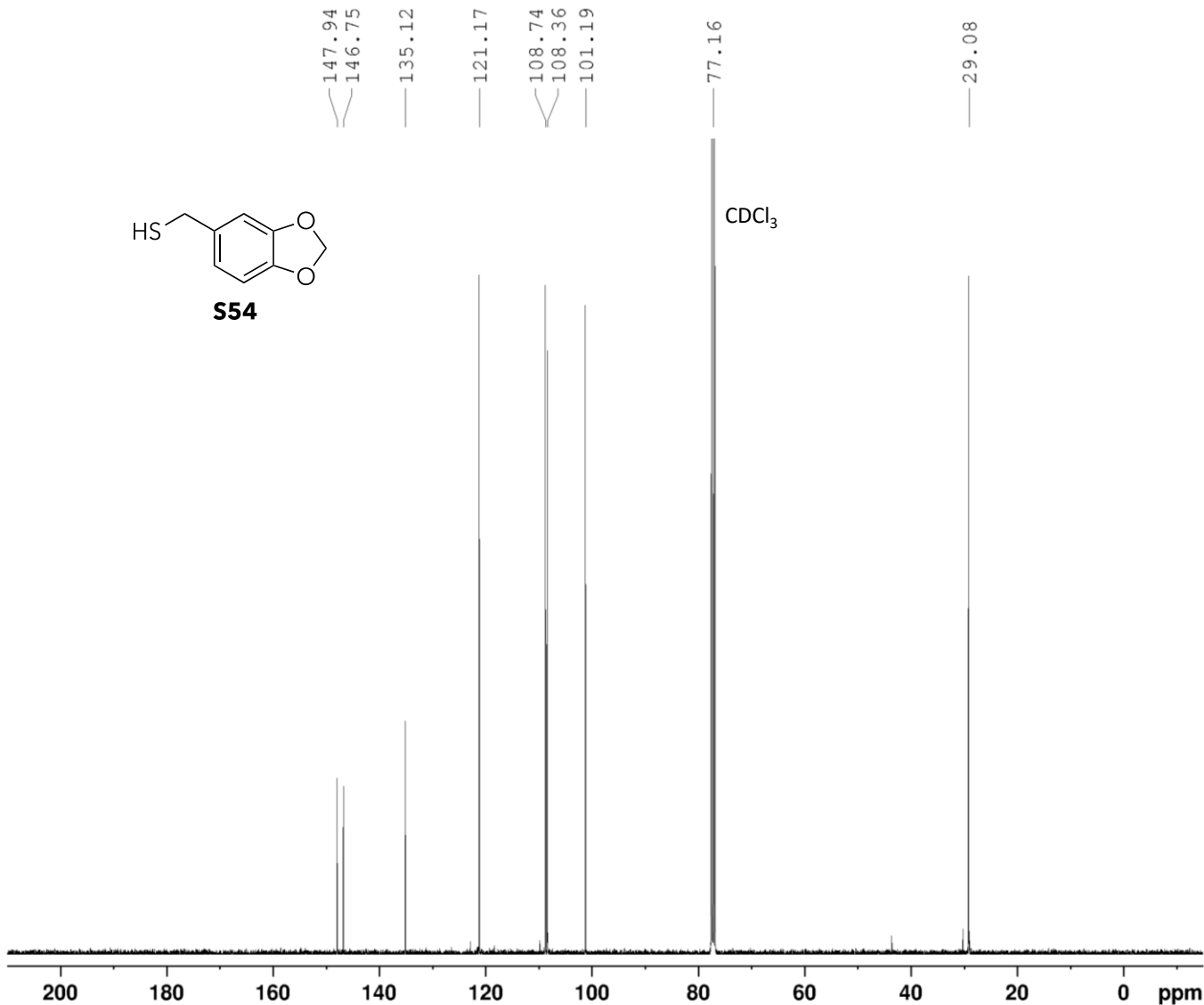
F2 - Acquisition Parameters

Date_ 20240525
Time 17.52 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (zg30)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.721 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 57
DW 61.000 usec
DE 13.54 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.53199959 W

F2 - Processing parameters

SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C spectrum of S54



Current Data Parameters
NAME OJ-7-33-C-2
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240526
Time 0.45 h
INSTRUM Avance Neo
PROBHD Z181819_0001 (PI HR-TBO400S1-BBF/H/P)
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F2 - Processing parameters
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GB 0
PC 1.40

APPENDIX C

CHAPTER FOUR SUPPORTING INFORMATION

Experimental Details

General Materials and Methods

NMR spectra were recorded at 298 K on a Bruker Ascend 400 MHz (400.130 MHz for ^1H NMR, 100.613 for ^{13}C , 376.461 for ^{19}F , 161.967 MHz for ^{31}P) NMR spectrometer. ^1H and ^{13}C NMR chemical shifts are reported in parts per million (ppm) relative to TMS, with the residual solvent peak used as an internal reference [^1H NMR: CHCl_3 (7.26 ppm), $(\text{CD}_3)\text{SO}(\text{CD}_2\text{H})$ (2.50 ppm); ^{13}C NMR: CDCl_3 (77.16 ppm), $(\text{CD}_3)_2\text{SO}$ (39.50 ppm)]. ^{19}F NMR chemical shifts are reported in parts per million (ppm) relative to trifluorotoluene (-63.72 ppm) as an internal reference. ^{31}P NMR chemical shifts are reported in parts per million (ppm) relative to triphenylphosphine (6.00 ppm) as an internal reference. Multiplicities are reported as follows: singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), quartet (q), quintet (quin), and multiplet (m). GC-FID data were collected using a Shimadzu GC-2010 Plus with a flame ionization detector equipped with a SH-Rxi-5ms capillary column (15 m x 0.25 mm ID x 0.25 μm df). GCMS data were collected with a Shimadzu GC-2030 paired with a Shimadzu GCMS-QP2020 NX and equipped with a SH-Rxi-5ms capillary column (30 m x 0.25 mm ID x 0.25 μm df). LC-MS analyses were performed on an Agilent 6538 Q-TOF MS coupled to an Agilent 1290 Infinity UHPLC system. A 50 mm long Zorbax Eclipse Plus C18 column (Agilent Technologies, Santa Barbara, CA; i.d. 2.1 mm, 1.8 μm particle size) was used for liquid chromatography separation at a column temperature of 50 $^\circ\text{C}$, a 0.6 mL/min flow rate, 2–3 μL injection (~ 10 $\mu\text{g/mL}$ concentration), and a column gradient ranging from A:B = 98:2 to A:B = 2:98, where Solvent A = LC-MS-grade water with 0.1% LC-MS-grade formic acid and Solvent B = LC-MS-grade MeCN with 0.1% LC-MS-grade formic acid. Electrospray ionization was operated in positive-ion mode with an m/z range of 50-1700 at 2 Hz. Manual flash column

chromatography was performed on SiliCycle silica gel 60 (40-63 μm particle size), and thin layer chromatography was performed on SiliCycle TLC plates pre-coated with extra hard silica gel 60 F254. Automated flash column chromatography was performed with a Biotage Selekt equipped with Biotage Sfar silica flash cartridges (20 μm particle size; 50 Å pore width) for normal phase separations, or Silica C18 cartridges (30 μm particle size; 100 Å pore width) for reversed phase separations. All reactions that require heating were conducted in aluminum single size vial reaction blocks (Chemglass) fitted to an IKA stirring hot plate equipped with a temperature probe.

When used in the glovebox, THF, toluene, and MeCN were degassed and purified with a JD Meyer solvent system. *p*-Xylene and other solvents used in the glovebox were degassed by the freeze-pump-thaw method. Deuterated solvents (CDCl_3 , C_6D_6 , acetone- d_6 , methanol- d_4) were obtained from Cambridge Isotopes and stored over molecular sieves. Aniline and 2-isopropyl aniline were distilled prior to use as reagents. 2,4-dichloro- (**1**), 2,4-dibromo-, and 2,4-diiodopyrimidine were purified before use by the following method: dihalopyrimidine (5 g) was dissolved in MeCN (25 mL), and the mixture was stirred at room temperature for at least 5 min, then filtered through Celite. The filtrate was then concentrated under vacuum. All other commercially-obtained chemicals were used as received.

Pd-PEPPSI-IPr, Pd-PEPPSI-IPent, dichloro[1,3-bis(2,6-diisopropylphenyl)-1,3-dihydro-2H-imidazol-2-ylidene]palladium(II) dimer, [1,3 bis(diphenylphosphino)propane]palladium(II) chloride, dichlorobis(tri-*o*-tolylphosphine)palladium(II), (η^3 -1-allyl)Pd(IPr)(Cl), (η^3 -1-cinnamyl)Pd(IPr)(Cl), 4-trifluoromethoxyaniline, *o*-anisidine, 4-fluoroaniline, aniline, *m*-xylene, *o*-xylene, and trifluorotoluene (PhCF_3) were obtained from Sigma-Aldrich. (η^3 -1-*t*Bu-indenyl)Pd(IPr)(Cl), (η^3 -1-*t*Bu-indenyl)Pd(IPent)(Cl), and [1,3-bis(2,46-trimethylphenyl)imidazole-2-ylidene][1,3-divinyl-1,1,3,3-tetramethyldisiloxane]palladium(0)

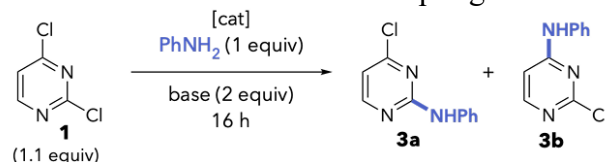
[(IMes)Pd(dvds)] were obtained from Umicore. 2,4-Dichloropyrimidine (**1**), 2,4,5-trichloropyrimidine, 2,4-dibromopyrimidine, 2,4-dichloro-5-methoxypyrimidine, 2,4-diiodopyrimidine, 2,4-dichloro-6-methoxypyrimidine, 5-bromo-2,4-dichloropyrimidine, 2,4-dichloro-6-methoxypyrimidine, 2,4-dichloro-5-methoxypyrimidine, 2,4-dichloro-5-fluoropyrimidine, 2,4-dichloro-5-bromopyrimidine, 4-(2-methoxyethoxy)aniline, 1-amino-2-(isopropylsulphonyl)benzene, 4-fluoro-2-methoxy-3-nitroaniline, 4-morpholinoaniline, 2-isopropylaniline, 3,4,5-trimethoxyaniline, 4-cyanoaniline, 4-fluoro-3-chloroaniline, 4-bromo-2-fluoroaniline, 1-methyl-1H-pyrazol-4-amine, (2-aminophenyl)dimethylphosphine oxide, 4-bromo-2-chloropyrimidine, 2-chloro-4-iodopyrimidine (**39**), cataCXium-A-Pd-G3, XPhos-Pd-G3, Brettphos-Pd-G3, SPhos-Pd-G3, and IMes•HCl were obtained from Ambeed Chemical. Potassium fluoride, tetrakis(triphenylphosphine)palladium(0), and PdCl₂ were obtained from Acros Organics. Potassium carbonate and triphenylphosphine were obtained from Alfa Aesar. tetrahydrofuran (THF), toluene, acetonitrile, benzonitrile, methanol, ethyl acetate, and *p*-xylene were obtained from Fisher Scientific. Tris(dibenzylideneacetone)-dipalladium(0) [Pd₂(dba)₃], 2,4-dichloro-6-methylpyrimidine, 4-trifluoromethylaniline, pyridine-3-boronic acid, 4-aminostyrene, *p*-anisidine, 2-iodoaniline, and Pd(OAc)₂ were obtained from Oakwood Chemical. P^tBu₃-Pd-G4 was obtained from Strem Chemical. Benzene was obtained from Beantown Chemical. Pd-PEPPSI-IMes and dichloro[1,3-bis(2,4,6-trimethylphenyl)-1,3-dihydro-2H-imidazol-2-ylidene]palladium(II) dimer were prepared according to literature procedures.^{1,2} Authentic C4-aminated compound **3b** was prepared by a literature procedure.³

Screening Reaction Conditions for Cross-Coupling of **1** with Aniline

General Procedure. Inside a nitrogen-filled glovebox, the indicated palladium complex (if applicable), additional ligand (if applicable), 2,4-dichloropyrimidine (**1**, 13.0 mg, 0.088 mmol, 1.1 equiv), and base (0.16 mmol, 2.0 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline was added (7.4 μ L, 0.08 mmol, 1 equiv) followed by solvent (0.2 mL). The vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at the indicated temperature for 16 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and mesitylene (4.9 μ L, 0.035 mmol, 0.44 equiv) was added as an internal standard. The mixture was filtered through CeliteTM before GC analysis.

Results

Table C.1. Screening Reaction Conditions for Cross-Coupling of **1** with Aniline



entry	trial	[Pd] (mol %)	solvent	base	temp (°C)	1 (%)	3a (%)	3b (%)	3a : 3b
1	1	$(\eta^3\text{-}t\text{-butylindenyl})\text{-Pd(IPent)Cl}$ (2)	MeCN	KF	80	86.8	n.d.	8.1	1 : >99
2	2					94.4	n.d.	4.2	1 : >99
3	avg					90.6	n.d.	6.2	1 : >99
4	1	None	MeCN	KF	80	84.1	n.d.	12.4	1 : >99
5	2					79.0	n.d.	16.1	1 : >99
6	avg					81.6	n.d.	14.3	1 : >99
7	1	$(\eta^3\text{-}t\text{-butylindenyl})\text{-Pd(IPent)Cl}$ (2)	THF	KF	80	93.0	2.4	2.8	1 : 1.3
8	2					95.7	1.1	1.8	1 : 1.6
9	avg					94.4	1.8	2.3	1 : 1.5
10	1	$(\eta^3\text{-}t\text{-butylindenyl})\text{-Pd(IPent)Cl}$ (2)	THF	KF	50	81.6	3.8	12.2	1 : 3.2
11	2					81.1	4.4	11.3	1 : 2.6
12	avg					81.4	4.1	11.8	1 : 2.9
13	1	$(\eta^3\text{-}t\text{-butylindenyl})\text{-Pd(IPent)Cl}$ (2)	THF	Cs_2CO_3	50	4.7	23.7	49.9	1 : 2.1
14	2					n.d.	26.2	50.5	1 : 1.9
15	avg					2.4	25.0	50.2	1 : 2.0
16	1	$(\eta^3\text{-}t\text{-butylindenyl})\text{-Pd(IPent)Cl}$ (2)	benzene	Cs_2CO_3	50	49.2	25.6	2.7	9.5 : 1
17	2					35.3	33.3	7.4	4.5 : 1
18	avg					42.3	29.5	5.1	7.0 : 1

19	1	(η^3 - <i>t</i> -butylindenyl)-	toluene	Cs ₂ CO ₃	50	n.d.	27.9	63	1 : 2.3
20	2	Pd(IPent)Cl (2)				n.d.	33	59.9	1 : 1.8
21	avg					n.d.	30.5	61.5	1 : 2.1
22	1	(η^3 - <i>t</i> -butylindenyl)-	<i>p</i> -xylene	Cs ₂ CO ₃	50	3.2	35.3	58.7	1 : 1.7
23	2	Pd(IPent)Cl (2)				11.8	37.4	52.3	1 : 1.4
24	avg					7.5	36.4	55.5	1 : 1.6
25	1	(η^3 - <i>t</i> -butylindenyl)-	<i>p</i> -xylene	Cs ₂ CO ₃	50	79.3	20.1	n.d.	>99 : 1
26	2	Pd(IPr)Cl (2)				84.1	14.8	n.d.	>99 : 1
27	avg					81.7	17.5	n.d.	>99 : 1
28	1	Pd-PEPPSI-IPent (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	28.7	60.4	n.d.	>99 : 1
29	2					35.4	55.8	n.d.	>99 : 1
30	avg					32.1	58.1	n.d.	>99 : 1
31	1	Pd-PEPPSI-IPr (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	30.5	65.2	n.d.	>99 : 1
32	2					43.0	51.1	n.d.	>99 : 1
33	avg					36.8	58.2	n.d.	>99 : 1
34	1	[(IPr)PdCl ₂] ₂ (1)	<i>p</i> -xylene	Cs ₂ CO ₃	50	24.0	67.2	n.d.	>99 : 1
35	2					31.9	69.1	n.d.	>99 : 1
36	avg					28.0	68.2	n.d.	>99 : 1
37	1	Pd-PEPPSI-IMes (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	3.6	93.1	n.d.	>99 : 1
38	2					4.2	91.4	n.d.	>99 : 1
39	avg					3.9	92.3	n.d.	>99 : 1
40	1	[(IMes)PdCl ₂] ₂ (1)	<i>p</i> -xylene	Cs ₂ CO ₃	50	3.7	92.3	n.d.	>99 : 1
41	2					1.4	88.2	n.d.	>99 : 1
42	3					2.5	92.6	n.d.	>99 : 1
43	4					2.3	87.4	n.d.	>99 : 1
44	avg					2.5	90.1	n.d.	>99 : 1
45	1	Pd-PEPPSI-IMes (2)	<i>p</i> -xylene	Cs ₂ CO ₃	21	52.8	50.2	n.d.	>99 : 1
46	2					47.7	54.4	n.d.	>99 : 1
47	avg					50.3	52.3	n.d.	>99 : 1
48	1	none	<i>p</i> -xylene	Cs ₂ CO ₃	50	97.0	n.d.	n.d.	N/A
49	2					94.9	n.d.	n.d.	N/A
50	avg					96.0	n.d.	n.d.	N/A
51	1	Pd-PEPPSI-IMes (2)	<i>m</i> -xylene	Cs ₂ CO ₃	50	n.d.	46.2	n.d.	>99 : 1
52	2					n.d.	47.9	n.d.	>99 : 1
53	avg					n.d.	47.1	n.d.	>99 : 1
54	1	Pd-PEPPSI-IMes (2)	<i>o</i> -xylene	Cs ₂ CO ₃	50	n.d.	91.2	n.d.	>99 : 1
55	2					n.d.	92.1	n.d.	>99 : 1
56	avg					n.d.	91.7	n.d.	>99 : 1
57	1	Pd-PEPPSI-IMes (2)	toluene	Cs ₂ CO ₃	50	n.d.	86.1	n.d.	>99 : 1
58	2					n.d.	87.4	n.d.	>99 : 1
59	avg					n.d.	86.8	n.d.	>99 : 1
60	1	Pd-PEPPSI-IMes (2)	benzene	Cs ₂ CO ₃	50	4.5	82.1	n.d.	>99 : 1
61	2					3.7	89.1	n.d.	>99 : 1
62	avg					4.1	85.6	n.d.	>99 : 1
63	1	Pd-PEPPSI-IMes (2)	PhCN	Cs ₂ CO ₃	50	n.d.	63.1	n.d.	>99 : 1
64	2					n.d.	45.8	n.d.	>99 : 1
65	avg					n.d.	54.5	n.d.	>99 : 1

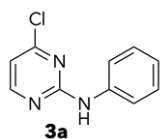
66	1	Pd-PEPPSI-IMes (2)	PhCF ₃	Cs ₂ CO ₃	50	10.0	89.1	n.d.	>99 : 1
67	2					9.2	84.6	n.d.	>99 : 1
68	avg					9.6	86.9	n.d.	>99 : 1
69	1	Pd-PEPPSI-IMes (2)	MeCN	Cs ₂ CO ₃	50	n.d.	21.0	n.d.	>99 : 1
70	2					n.d.	25.5	n.d.	>99 : 1
71	avg					n.d.	23.3	n.d.	>99 : 1
72	1	Pd-PEPPSI-IMes (2)	THF	Cs ₂ CO ₃	50	n.d.	84.4	n.d.	>99 : 1
73	2					n.d.	89.9	n.d.	>99 : 1
74	avg					n.d.	87.2	n.d.	>99 : 1
75	1	Pd-PEPPSI-IMes (2)	EtOAc	Cs ₂ CO ₃	50	11.2	89.9	n.d.	>99 : 1
76	2					14.2	79.1	n.d.	>99 : 1
77	avg					12.7	84.5	n.d.	>99 : 1
78	1	PdCl ₂ (2) + IMes•HCl (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	17.5	75.6	n.d.	>99 : 1
79	2					25.1	62.4	n.d.	>99 : 1
80	3					14.7	83.4	n.d.	>99 : 1
81	4					27.3	65.2	n.d.	>99 : 1
82	avg					21.2	71.1	n.d.	>99 : 1
83	1	[(IPr*)PdCl ₂] ₂ (1)	<i>p</i> -xylene	Cs ₂ CO ₃	50	10.2	3.0	n.d.	>99 : 1
84	2					26.2	2.8	n.d.	>99 : 1
85	avg					18.4	2.9	n.d.	>99 : 1
86	1	CataCXium A Pd G3 (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	60.9	22.6	n.d.	>99 : 1
87	2					64.1	25.0	n.d.	>99 : 1
88	avg					62.5	23.8	n.d.	>99 : 1
89	1	SPhos Pd G3 (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	n.d.	6.6	80.1	1 : 12.1
90	2					3.5	5.9	71.6	1 : 12.1
91	avg					1.8	6.3	75.9	1 : 12.1
92	1	XPhos Pd G3 (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	86.6	9.2	n.d.	>99 : 1
93	2					89.9	3.6	n.d.	>99 : 1
94	avg					88.3	6.4	n.d.	>99 : 1
95	1	BrettPhos Pd G3 (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	77.8	5.9	n.d.	>99 : 1
96	2					75.9	6.3	n.d.	>99 : 1
97	avg					76.9	6.1	n.d.	>99 : 1
98	1	Pd(PPh ₃) ₄ (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	80.9	n.d.	n.d.	>99 : 1
99	2					79.2	n.d.	n.d.	>99 : 1
100	avg					80.1	n.d.	n.d.	>99 : 1
101	1	PdCl ₂ (2)	<i>p</i> -xylene	Cs ₂ CO ₃	50	95.2	2.6	n.d.	>99 : 1
102	2					97.4	2.4	n.d.	>99 : 1
103	avg					96.3	2.5	n.d.	>99 : 1
104	1	Pd(OAc) ₂	<i>p</i> -xylene	Cs ₂ CO ₃	50	96.4	n.d.	n.d.	>99 : 1
105	2					93.3	n.d.	n.d.	>99 : 1
106	avg					94.9	n.d.	n.d.	>99 : 1

^a GC yields calibrated against mesitylene as an internal standard.

Isolation and Characterization of Cross-Coupled Products

General Procedure for Cross-Coupling. Inside a nitrogen filled glovebox, the indicated palladium complex, 2,4-dihalopyrimidine, and Cs_2CO_3 (2.0 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. The indicated amine was added followed by *p*-xylene (volume calculated such that the limiting reagent would have a concentration of 0.4 M). The vessel was sealed with a Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and filtered through Celite™. The filtrate was concentrated under vacuum, the residue was taken up in deuterated solvent, and an aliquot of the solution was analyzed by ^1H NMR analysis to obtain the crude ratio of C2:C4 functionalized products. The NMR sample was recombined with the bulk crude residue, and the solvent was removed under vacuum prior to purification.

General Procedure for Small-Scale Uncatalyzed SNAr Reactions (Used to Facilitate Identification of Regioisomers). Open to air, 2,4-dihalopyrimidine (0.08 mmol, 1.0 equiv), triethylamine (22 μL , 0.16 mmol, 2 equiv) and the indicated amine (0.08 mmol, 1.0 equiv) were combined in a 1-dram vial equipped with a Teflon-coated stir bar. EtOH (volume calculated such that the dihalopyrimidine would have a concentration of 0.4 M) was then added. The vial was sealed with a Teflon-lined cap and the reaction mixture was stirred vigorously at room temperature for 16 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and filtered through Celite™. The filtrate was concentrated under reduced pressure and mesitylene was added as an internal standard (11.1 μL , 0.080 mmol, 1.0 equiv). The residue was taken up in deuterated solvent and analyzed by ^1H NMR.

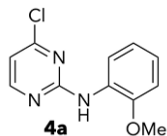


4-Chloro-N-phenylpyrimidin-2-amine (3a). Compound **3a** was prepared

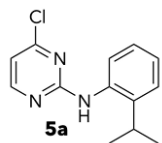
according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), aniline (45.7 μ L, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (*R_f* = 0.16) provided **3a** as a white solid (88.4 mg, 86% yield). ¹H NMR (400 MHz, CDCl₃, δ): 8.28 (d, *J* = 5.2 Hz, 1H), 7.63-7.59 (multiple overlapping signals, 3H), 7.38-7.34 (m, 2H), 7.11-7.07 (m, 1H), 6.74 (d, *J* = 5.2 Hz, 1H). ¹³C {¹H} NMR (101 MHz, CDCl₃, δ): 161.4, 160.0, 159.1, 138.7, 129.1, 123.5, 119.9, 112.2. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₀H₉ClN₃ 206.0480; found 206.0479. X-ray quality crystals were grown from chloroform at 25 °C and the regiochemistry was verified by X-ray diffraction.

On ~1g scale: Inside a nitrogen filled glovebox, [(IMes)PdCl₂]₂ (58.9 mg, 0.061 mmol, 0.01 equiv), 2,4 dichloropyrimidine (**1**, 1 g, 6.71 mmol, 1.1 equiv), and Cs₂CO₃ (3.98 g, 12.2 mmol, 2.0 equiv) were combined in an oven-dried 250 mL round bottom flask equipped with a Teflon-coated stir bar. The reaction vessel was sealed with a rubber septum and removed from the glovebox. Aniline (557 μ L, 6.1 mmol, 1 equiv) and *p*-xylene (14.7 mL) were then added through the septum. The reaction mixture was stirred at 50 °C for 16 h. The reaction mixture was diluted with ethyl acetate (20 mL) and filtered through Celite™. The filtrate was concentrated under vacuum, and a portion of the residue was taken up in CDCl₃ for ¹H NMR analysis, which indicated a >99 : 1 ratio of C2:C4-functionalized products. The NMR sample was recombined with the bulk crude residue, and the solvent was removed under vacuum. Purification by flash column chromatography on

silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.16) provided **3a** as a white solid (990.8 mg, 79% yield).

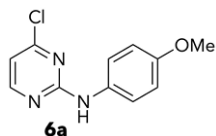


4-Chloro-N-(2-methoxyphenyl)pyrimidin-2-amine (4a). Compound **4a** was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 2-methoxyaniline (56 μ L, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.35) provided **4a** as a colorless oil (70.7 mg, 60% yield). ¹H NMR (400 MHz, CDCl₃, δ): 8.43-8.41 (m, 1H), 8.29 (d, J = 5.2 Hz, 1H), 8.02 (br s, 1H), 7.05-6.98 (m, 2H), 6.92-6.89 (m, 1H), 6.75 (d, J = 5.2 Hz, 1H), 3.90 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃, δ): 161.7, 159.4, 158.3, 148.4, 128.2, 123.0, 121.0, 119.2, 112.0, 110.2, 55.9. HRMS (ESI Q-TOF) m/z : [M+H]⁺ Calcd for C₁₁H₁₁ClN₃O 236.0585; found 236.0599. The regiochemistry of **4a** was confirmed by comparing its ¹H NMR spectrum to the previously reported ¹H NMR data for its regioisomer, 2-chloro-N-(2-methoxyphenyl)pyrimidin-4-amine, corresponding to C4-amination.³ The spectra do not match.



4-Chloro-N-(2-isopropylphenyl)pyrimidin-2-amine (5a). Compound **5a** was prepared according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 2-isopropylaniline (70.8 μ L, 0.50 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using

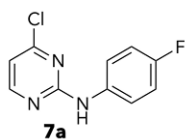
an eluent of hexanes:EtOAc = 70:20 (R_f = 0.35) provided **5a** as a yellow oil. (100 mg, 81% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.21 (d, J = 5.2 Hz, 1H), 7.68-7.65 (m, 1H), 7.35-7.32 (m, 1H), 7.25-7.19 (m, 2H), 7.08 (br s, 1 H), 6.70 (d, J = 5.2 Hz, 1H), 3.15 (septet, J = 6.8 Hz, 1H), 1.25 (d, J = 6.8 Hz, 6H). ^{13}C $\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.9, 161.1, 158.9, 141.9, 134.8, 126.5, 126.2, 126.1, 125.0, 111.8, 28.2, 23.3. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{15}\text{ClN}_3$ 248.0949; found 248.0947. The regiochemistry of **5a** was verified by comparing its ^1H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free $\text{S}_{\text{N}}\text{Ar}$ reaction (following the general $\text{S}_{\text{N}}\text{Ar}$ procedure), which is expected to favor the opposite regioisomer. The $\text{S}_{\text{N}}\text{Ar}$ conditions provided a regioisomer (C2:C4 = <1 : 99) that does not correspond to the one formed under the catalytic conditions.



4-Chloro-N-(4-methoxyphenyl)pyrimidin-2-amine (6a). Compound **6a** was prepared according to the general cross-coupling procedure using Pd-PEPPSI-

IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-methoxyaniline (61.6 mg, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.26) provided **6a** as a white solid (87.2 mg, 74% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.22 (d, J = 5.2 Hz, 1H), 7.46-7.44 (multiple overlapping signals, 3H), 6.90-6.89 (m, 2H), 6.68 (d, J = 5.2 Hz, 1H), 3.80 (s, 3H). ^{13}C $\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.5, 160.4, 159.1, 156.3, 131.6, 122.5, 114.4, 111.7, 55.6. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_{11}\text{ClN}_3\text{O}$ 236.0585; found 236.0588. The regiochemistry of **6a** was confirmed by comparing

its ^1H NMR spectrum to the previously reported ^1H NMR data for its regioisomer, 2-chloro-*N*-(4-methoxyphenyl)pyrimidin-4-amine, corresponding to C4-amination.⁴ The spectra do not match.



4-Chloro-*N*-(4-fluorophenyl)pyrimidin-2-amine (7a). Compound **7a** was

prepared according to the general cross-coupling procedure using Pd-PEPPSI-

IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv),

Cs_2CO_3 (326 mg, 1.0 mmol), 4-fluoroaniline (47 μL , 0.5 mmol, 1 equiv), and xylene (1.25 mL).

Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent

of hexanes:EtOAc = 80:20 (R_f = 0.13) provided **7a** as a white solid (72.5 mg, 65% yield). ^1H NMR

[400 MHz, $(\text{CD}_3)_2\text{SO}$, δ]: 10.1 (br s, 1H), 8.42 (d, J = 5.2 Hz, 1H), 7.72-7.69 (m, 2H), 7.18-7.14

(m, 2H), 6.95 (d, J = 5.2 Hz, 1H). ^{13}C $\{^1\text{H}\}$ NMR [101 MHz, $(\text{CD}_3)_2\text{SO}$, δ]: 160.0, 159.9, 159.7,

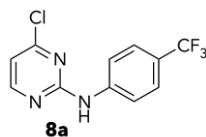
157.6 (d, $^1J_{\text{CF}}$ = 239.2 Hz), 135.9 (q, $^4J_{\text{CF}}$ = 2.2 Hz), 121.1 (d, $^3J_{\text{CF}}$ = 7.5 Hz), 115.1 (d, $^2J_{\text{CF}}$ = 22.0

Hz), 111.5. ^{19}F NMR [376 MHz, $(\text{CD}_3)_2\text{SO}$, δ]: -123.4. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd

for $\text{C}_{10}\text{H}_8\text{ClFN}_3$ 224.0385; found 224.0387. The regiochemistry of **7a** was confirmed by

comparing its ^1H NMR spectrum to the previously reported ^1H NMR data for its regioisomer, 2-chloro-*N*-(4-fluorophenyl)pyrimidin-4-amine, corresponding to C4-amination.⁵ The spectra do not

match.



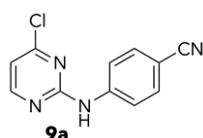
4-Chloro-*N*-[4-(trifluoromethyl)phenyl]pyrimidin-2-amine (8a).

Compound **8a** was prepared according to the general cross-coupling procedure

using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine

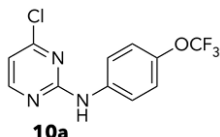
(**1**, 74.5 mg, 0.50 mmol, 1.0 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-(trifluoromethyl)aniline (75

μL , 0.60 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:30 (R_f = 0.37) provided **8a** as a white solid (106.9 mg, 78% yield). ^1H and ^{13}C NMR spectra are consistent with those previously reported.⁶ ^{19}F NMR (376 MHz, CDCl_3 , δ): -62.93. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_8\text{ClF}_3\text{N}_3$ 274.0353; found 274.0362.



4-[(4-Chloropyrimidin-2-yl)amino]benzonitrile (9a). Compound **9a** was

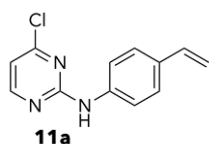
prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 74.5 mg, 0.5 mmol, 1.0 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-aminobenzonitrile (70.9 mg, 0.6 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.16) provided **9a** as a white solid (71.6 mg, 62% yield). NMR spectra are consistent with those previously reported.^{6,7} HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_8\text{ClN}_4$ 231.0432; found 231.0462.



4-Chloro-N-[4-(trifluoromethoxy)phenyl]pyrimidin-2-amine (10a).

Compound **10a** was prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 74.5 mg, 0.5 mmol, 1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-(trifluoromethoxy)aniline (80.5 mg, 0.6 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products.

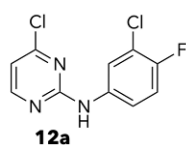
Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.13) provided **10a** as a white solid (102 mg, 71% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.29 (d, J = 5.2 Hz, 1H), 7.62 (d, J = 8.8 Hz, 2H), 7.55 (s, 1H), 7.20 (d, J = 8.8 Hz, 2H), 6.79 (d, J = 5.2 Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.6, 159.8, 159.1, 144.8 (q, $^3J_{\text{CF}}$ = 1.9 Hz), 137.4, 121.9, 121.8 (q, $^1J_{\text{CF}}$ = 256.8 Hz), 120.8, 112.8. ^{19}F NMR (376 MHz, CDCl_3 , δ): – 59.13. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_8\text{ClF}_3\text{N}_3\text{O}$ 290.0303; found 290.0312. The regiochemistry of **10a** was verified by comparing its ^1H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free $\text{S}_{\text{N}}\text{Ar}$ reaction (following the general $\text{S}_{\text{N}}\text{Ar}$ procedure), which is expected to favor the opposite regioisomer. The $\text{S}_{\text{N}}\text{Ar}$ conditions provided a 1 : 20 ratio of regioisomers, with the minor isomer corresponding spectroscopically to the one formed under the catalytic conditions (**10a**).



4-Chloro-N-(4-vinylphenyl)pyrimidin-2-amine (11a). Compound **11a** was prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**,

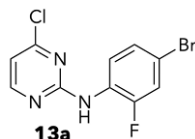
81.9 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-vinylaniline (58.5 μL , 0.50 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.39) provided **11a** as a white solid (103 mg, 89% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.28 (d, J = 5.2 Hz, 1H), 7.56-7.52 (multiple overlapping signals, 3H), 7.40 (d, J = 8.6 Hz, 2H), 6.75 (d, J = 5.2 Hz, 1H), 6.69 (dd, J = 17.6 11.0 Hz, 1H), 5.68 (d, J = 17.6 Hz, 1H), 5.19 (d, J = 11.0 Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.7, 159.7, 158.7, 138.1, 136.3, 133.1, 127.0, 119.8, 112.8, 112.3. HRMS (ESI Q-TOF) m/z :

$[M+H]^+$ Calcd for $C_{12}H_{11}ClN_3$ 232.0636; found 232.0649. The regiochemistry of **11a** was verified by comparing its 1H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free S_NAr reaction (following the general S_NAr procedure), which is expected to favor the opposite regioisomer. The S_NAr conditions provided a 1:17 ratio of regioisomers, with the minor isomer corresponding spectroscopically to the one formed under the catalytic conditions (**11a**).



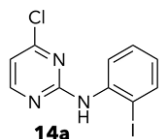
4-Chloro-N-(3-chloro-4-fluorophenyl)pyrimidin-2-amine (12a). Compound

12a was prepared according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 3-chloro-4-fluoroaniline (72.8 mg, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by 1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.13) provided **12a** as a white solid (76.1 mg, 59% yield). 1H NMR (400 MHz, $CDCl_3$, δ): 8.30 (d, J = 5.2 Hz, 1H), 7.81-7.79 (m, 1H), 7.38-7.34 (multiple overlapping signals, 2H), 7.13-7.09 (m, 1H), 6.80 (d, J = 5.2 Hz, 1H). ^{13}C $\{^1H\}$ NMR (101 MHz, $CDCl_3$, δ): 161.7, 159.6, 159.1, 154.4 (d, $^1J_{CF}$ = 245.8 Hz), 135.3 (d, $^3J_{CF}$ = 3.0 Hz), 121.9, 121.2 (d, $^2J_{CF}$ = 18.5 Hz), 119.5 (d, $^3J_{CF}$ = 6.6 Hz), 116.8 (d, $^2J_{CF}$ = 22.5 Hz), 112.9. ^{19}F NMR (376 MHz, $CDCl_3$, δ): -122.6. HRMS (ESI Q-TOF) m/z : $[M+H]^+$ Calcd for $C_{10}H_7Cl_2FN_3$ 257.9996; found 257.9978. X-ray quality crystals were grown from chloroform at 0 °C and the regiochemistry was verified by X-ray diffraction (see pp. SXX–SXX).



***N*-(4-bromo-2-fluorophenyl)-4-chloropyrimidin-2-amine (13a).** Compound

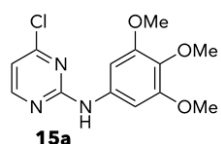
13a was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 74.5 mg, 0.5 mmol, 1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 4-bromo-2-fluoroaniline (114 mg, 0.6 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (*R*_f = 0.42) provided **13a** as a white solid (95.3 mg, 63% yield). ¹H NMR [400 MHz, (CD₃)₂SO, δ]: 9.68 (br s, 1H), 8.38 (d, *J* = 5.3 Hz, 1H), 7.65-7.56 (multiple overlapping signals, 2H), 7.41-7.38 (m, 1H), 6.97 (d, *J* = 5.3 Hz, 1H). ¹³C{¹H} NMR [101 MHz, (CD₃)₂SO, δ]: 160.2, 160.1, 160.0, 155.2 (d, 1JCF = 252.2 Hz), 127.3 (d, 3JCF = 3.5 Hz), 127.2 (d, 4JCF = 2.0 Hz), 126.2 (d, 2JCF = 11.8 Hz), 119.0 (d, 2JCF = 23.4 Hz), 116.2 (d, 3JCF = 9.5 Hz), 112.0.¹⁹F NMR [376 MHz, (CD₃)₂SO, δ]: -120.31. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₀H₇BrClFN₃ 301.9490; found 301.9495. The regiochemistry of **13a** was verified by comparing its ¹H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free S_NAr reaction (following the general S_NAr procedure), which is expected to favor the opposite regioisomer. The S_NAr conditions provided a regioisomer (C2:C4 = <1 : 99) that does not correspond to the one formed under the catalytic conditions.



4-Chloro-*N*-(2-iodophenyl)pyrimidin-2-amine (14a). Compound **14a** was

prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 2-iodoaniline (109.5 mg, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-

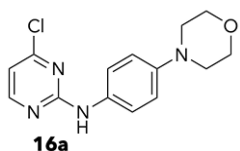
functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.39) provided **14a** as an off-white solid (92 mg, 56% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.31 (d, J = 5.4 Hz, 1H), 8.27-8.24 (m, 1H), 7.84-7.81 (m, 1H), 7.45 (br s, 1H), 7.40-7.36 (m, 1H), 6.85-6.80 (multiple overlapping signals, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.7, 159.8, 159.1, 139.3, 139.1, 129.2, 125.3, 121.7, 113.1, 90.8. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_8\text{ClIN}_3$ 331.9446; found 331.9454. The regiochemistry of **14a** was verified by comparing its ^1H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free $\text{S}_{\text{N}}\text{Ar}$ reaction (following the general $\text{S}_{\text{N}}\text{Ar}$ procedure), which is expected to favor the opposite regioisomer. The $\text{S}_{\text{N}}\text{Ar}$ conditions provided a 1:18 ratio of regioisomers, with the minor isomer corresponding spectroscopically to the one formed under the catalytic conditions (**14a**).



4-Chloro-N-(3,4,5-trimethoxyphenyl)pyrimidin-2-amine (15a). Compound

15a was prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 3,4,5-trimethoxyaniline (91.6 mg, 0.50 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:20 (R_f = 0.11) provided **15a** as a white solid (117 mg, 79% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.27 (d, J = 5.2 Hz, 1H), 7.46 (br s, 1H), 6.89 (s, 2H), 6.74 (d, J = 5.2 Hz, 1H), 3.86 (s, 6H), 3.82 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.4, 160.0, 159.1, 153.5, 134.7, 134.3, 112.0, 97.9, 61.1, 56.2. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{13}\text{H}_{15}\text{ClN}_3\text{O}_3$ 296.0796; found 296.0801. The regiochemistry of **15a** was verified by comparing

its ^1H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free $\text{S}_{\text{N}}\text{Ar}$ reaction (following the general $\text{S}_{\text{N}}\text{Ar}$ procedure), which is expected to favor the opposite regioisomer. The $\text{S}_{\text{N}}\text{Ar}$ conditions provided a regioisomer (C2:C4 = <1 : 99) that does not correspond to the one formed under the catalytic conditions.



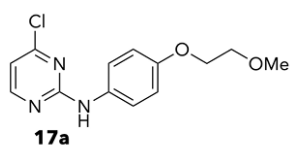
4-Chloro-N-(4-morpholinophenyl)pyrimidin-2-amine (16a). Compound

16a was prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**,

74.5 mg, 0.5 mmol, 1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), 4-morpholinoaniline (106.8 mg, 0.60 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:30 (R_f = 0.10) provided **16a** as a white solid (107.3 mg, 74% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.23 (d, J = 5.2 Hz, 1H), 7.47-7.44 (m, 2H), 7.29 (br s, 1H), 6.94-6.91 (m, 2H), 6.69 (d, J = 5.2 Hz, 1H), 3.88-3.86 (m, 4H), 3.14-3.11 (m, 4H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 161.4, 160.4, 159.1, 147.8, 131.4, 121.9, 116.6, 111.6, 67.0, 50.0. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{14}\text{H}_{16}\text{ClN}_4\text{O}$ 291.1009; found 291.1007. X-ray quality crystals were grown from chloroform at 0 °C and the regiochemistry was verified by X-ray.

On 1 mmol scale: Inside a nitrogen filled glovebox, $[(\text{IMes})\text{PdCl}_2]_2$ (9.65 mg, 0.01 mmol, 0.01 equiv) 2,4 dichloropyrimidine (**1**, 149 mg, 1 mmol, 1 equiv), Cs_2CO_3 (652 mg, 2 mmol, 2.0 equiv), and 4-morpholinoaniline (213.6 mg, 1.2 mmol, 1.2 equiv) were combined in an oven-dried 25 mL round bottom flask equipped with a Teflon-coated stir bar. The reaction vessel was sealed

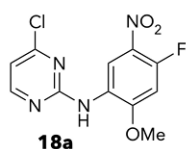
with a rubber septum and removed from the glovebox. *p*-Xylene (2.5 mL) was added through the septum. The reaction mixture was stirred at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate (5 mL) and filtered through Celite™. The filtrate was concentrated under vacuum, and a portion of the residue was taken up in CDCl₃ for ¹H NMR analysis, which indicated a >99 : 1 ratio of C2:C4-functionalized products. The NMR sample was recombined with the bulk crude residue, and the solvent was removed under vacuum. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:30 (*R_f* = 0.10) provided **16a** as a white solid (215.1 mg, 74% yield).



4-Chloro-N-[4-(2-methoxyethoxy)phenyl]pyrimidin-2-amine (17a).

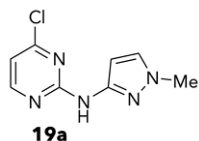
Compound **17a** was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 4-(2-methoxyethoxy)aniline (77.5 μL, 0.500 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:30 (*R_f* = 0.28) provided **17a** as a yellow solid (104.2 mg, 74% yield). ¹H NMR (400 MHz, CDCl₃, δ): 8.19 (d, *J* = 5.2 Hz, 1H), 7.74 (br s, 1H), 7.44-7.42 (m, 2H), 6.91-6.88 (m, 2H), 6.65 (d, *J* = 5.2 Hz, 1H), 4.09 (t, *J* = 4.6 Hz, 2H), 3.73 (t, *J* = 4.6 Hz, 2H), 3.43 (s, 3H). ¹³C {¹H} NMR (101 MHz, CDCl₃, δ): 161.3, 160.3, 159.0, 155.3, 131.8, 122.2, 115.1, 111.5, 71.1, 67.6, 59.2. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₃H₁₅ClF₃N₃O₂ 280.0847; found 280.0863. The regiochemistry of **17a** was verified by comparing its ¹H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free S_NAr reaction (following the

general S_NAr procedure), which is expected to favor the opposite regioisomer. The S_NAr conditions provided a 1:6 ratio of regioisomers, with the minor isomer corresponding spectroscopically to the one formed under the catalytic conditions (**17a**).

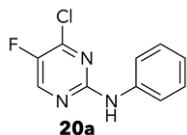


4-Chloro-N-(4-fluoro-2-methoxy-5-nitrophenyl)pyrimidin-2-amine (18a).

Compound **18a** was prepared according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 81.9 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 4-fluoro-2-methoxy-5-nitroaniline (93.1 mg, 0.50 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 70:30 (*R_f* = 0.12) provided **18a** as a white solid (102 mg, 68% yield). ¹H NMR (400 MHz, CDCl₃, δ): 9.36 (d, ⁴*J*_{HF} = 8.3 Hz, 1H), 8.38 (d, *J* = 5.2 Hz, 1H), 7.77 (br s, 1H), 6.87 (d, *J* = 5.2 Hz, 1H), 6.75 (d, ³*J*_{HF} = 12.0 Hz, 1H), 4.01 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃, δ): 161.7, 159.0, 153.1 (d, 2*J*_{CF} = 9.4 Hz), 152.4 (d, 1*J*_{CF} = 262.1 Hz), 130.1, 130.0, 125.0 (d, 3*J*_{CF} = 2.9 Hz), 115.3 (d, 3*J*_{CF} = 3.0 Hz), 113.5, 100.0 (d, 2*J*_{CF} = 26.5 Hz), 57.0. ¹⁹F NMR (376 MHz, CDCl₃, δ): −119.9. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₁H₉ClFN₄O₃ 299.0342; found 299.0361. The regiochemistry of **18a** was verified by comparing its ¹H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free S_NAr reaction (following the general S_NAr procedure), which is expected to favor the opposite regioisomer. The S_NAr conditions provided a regioisomer (C2:C4 = <1 : 99) that does not correspond to the one formed under the catalytic conditions.

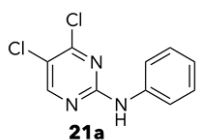


4-Chloro-N-(1-methyl-1H-pyrazol-3-yl)pyrimidin-2-amine (19a). Compound **19a** was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloropyrimidine (**1**, 74.5 mg, 0.5 mmol, 1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), 1-methyl-1H-pyrazol-4-amine (58.2 mg, 0.6 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (*R_f* = 0.28) provided **19a** as an off-white solid (26.2 mg, 25% yield). ¹H NMR (400 MHz, CDCl₃, δ): 8.24 (d, *J* = 5.0 Hz, 1H), 7.85 (app s, 1H), 7.47 (app s, 1H), 7.19 (br s, 1H), 6.69 (d, *J* = 5.0 Hz, 1H), 3.90 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃, δ): 161.8, 159.7, 159.0, 131.1, 122.1, 121.8, 111.4, 39.5. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₈H₉ClN₅ 210.0541; found 210.0548. The regiochemistry of **19a** was verified by comparing its ¹H NMR spectrum to the spectrum of the crude reaction mixture obtained from the corresponding catalyst-free S_NAr reaction (following the general S_NAr procedure), which is expected to favor the opposite regioisomer. The S_NAr conditions provided a regioisomer (C2:C4 = <1 : 99) that does not correspond to the one formed under the catalytic conditions.



4-Chloro-5-fluoro-N-phenylpyrimidin-2-amine (20a). Compound **20a** was prepared according to the general cross-coupling procedure using [(IPr)PdCl₂]₂ (5.7 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloro-5-fluoropyrimidine (83.5 mg, 0.5 mmol, 1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), aniline (55 μL, 0.6 mmol, 1.2 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of

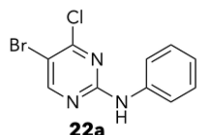
hexanes:EtOAc = 80:20 (R_f = 0.45) provided **20a** as a white solid (72 mg, 64% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.26 (s, 1H), 7.56-7.54 (m, 2H), 7.37-7.34 (m, 2H), 7.27 (broad s, 1H), 7.11-7.07 (m, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 155.8 (d, $^4J_{\text{CF}}$ = 3.7 Hz), 148.3 (d, $^1J_{\text{CF}}$ = 255.2 Hz), 148.2 (d, $^2J_{\text{CF}}$ = 18.1 Hz), 146.0 (d, $^2J_{\text{CF}}$ = 23.2 Hz), 138.7, 129.2, 123.5, 119.4. ^{19}F NMR (376 MHz, CDCl_3 , δ): -148.5. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_8\text{ClFN}_3$ 224.0385; found 224.0384. The regiochemistry of **20a** was confirmed by comparing its ^1H NMR spectrum to the previously reported ^1H NMR data for its regioisomer, 2-chloro-5-fluoro-*N*-phenylpyrimidin-4-amine, corresponding to C4-amination.⁸ The spectra do not match.



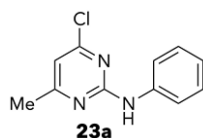
4,5-dichloro-*N*-phenylpyrimidin-2-amine (21a). Compound **21a** was prepared

according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4,5-trichloropyrimidine (63 μL , 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), aniline (46 μL , 0.50 mmol, 1 equiv), and xylene (1.25 mL).

Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.39) provided **21a** as a white solid (70.8 mg, 59% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.34 (s, 1H), 7.57-7.55 (m, 2H), 7.38-7.34 (m, 2H), 7.21 (br s, 1H), 7.12-7.09 (m, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 158.4, 157.7, 138.3, 129.3, 123.9, 119.8, 119.7, 118.8. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_8\text{Cl}_2\text{N}_3$ 240.0090; found 240.0105. The regiochemistry of **21a** was confirmed by comparing its ^1H NMR spectrum to the previously reported ^1H NMR data for its regioisomer, 2,5-chloro-*N*-phenylpyrimidin-4-amine, corresponding to C4-amination.^{7,9} The spectra do not match.

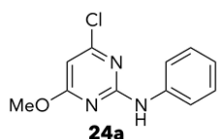


5-Bromo-4-chloro-N-phenylpyrimidin-2-amine (22a). Compound **22a** was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 5-bromo-2,4-dichloropyrimidine (63 μ L, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), aniline (46 μ L, 0.50 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (*R_f* = 0.45) provided **22a** as a white solid (56.9 mg, 40% yield). ¹H NMR (400 MHz, CDCl₃, δ): 8.42 (s, 1H), 7.56-7.54 (m, 2H), 7.38-7.34 (m, 2H), 7.30 (br s, 1H), 7.13-7.09 (m, 1H). ¹³C {¹H} NMR (101 MHz, CDCl₃, δ): 160.4, 159.9, 158.3, 138.3, 129.2, 123.9, 119.8, 107.3. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₀H₈BrClN₃ 283.9585; found 283.9581. The regiochemistry of **22a** was confirmed by comparing its ¹H NMR spectrum to the previously reported ¹H NMR data for its regioisomer, 2-chloro-5-bromo-N-phenylpyrimidin-4-amine, corresponding to C4-amination.⁸ The spectra do not match.



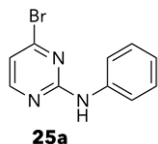
4-Chloro-6-methyl-N-phenylpyrimidin-2-amine (23a). Compound **23a** was prepared according to the general cross-coupling procedure using [(IMes)PdCl₂]₂ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-dichloro-6-methylpyrimidine (120.8 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (326 mg, 1.0 mmol), aniline (46 μ L, 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ¹H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (*R_f* = 0.39) provided **23a** as a white solid (67 mg, 61% yield). ¹H NMR (400 MHz, CDCl₃, δ): 7.63-7.61 (m, 2H), 7.36-7.32 (m, 2H), 7.28 (br s, 1H), 7.09-7.05

(m, 1H), 6.64 (s, 1H), 2.41 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 169.6, 161.4, 159.4, 138.9, 129.1, 123.2, 119.5, 111.5, 23.9. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_{11}\text{ClN}_3$ 220.0636; found 220.0636. The regiochemistry of **23a** was confirmed by comparing its ^1H NMR spectrum to the previously reported ^1H NMR data for its regioisomer, 2-chloro-6-methyl-*N*-phenylpyrimidin-4-amine, corresponding to C4-amination.¹⁰ The spectra do not match.



4-Chloro-6-methoxy-*N*-phenylpyrimidin-2-amine (24a). Compound **24a** was

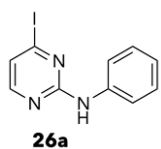
prepared according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (8.2 mg, 0.01 mmol, 0.01 equiv), 2,4-dichloro-6-methoxypyrimidine (197 mg, 1.1 mmol, 1.1 equiv), Cs_2CO_3 (652 mg, 2.0 mmol), aniline (92 μL , 1.0 mmol, 1 equiv), and xylene (2.5 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.45) provided **24a** as a colorless oil (96.6 mg, 41% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 7.60-7.58 (m, 2H), 7.36-7.32 (m, 2H), 7.21 (br s, 1H), 7.09-7.05 (m, 1H), 6.23 (s, 1H), 3.98 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 171.4, 160.4, 159.0, 138.8, 129.1, 123.3, 119.6, 98.1, 54.5. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_{11}\text{ClN}_3\text{O}$ 236.0585; found 236.0588. X-ray quality crystals were grown from chloroform at 0 °C and the regiochemistry was verified by X-ray diffraction.



4-Bromo-*N*-phenylpyrimidin-2-amine (25a). Compound **25a** was prepared

according to the general cross-coupling procedure using Pd-PEPPSI-IMes (5.9 mg, 0.01 mmol, 0.02 equiv), 2,4-dibromopyrimidine (130.9 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1.0 mmol), aniline (45.7 μL , 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis

of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.35) provided **25a** as a white solid (97 mg, 78% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.16 (d, J = 5.2 Hz, 1H), 7.59-7.57 (m, 2H), 7.43 (br s, 1H), 7.37-7.33 (m, 2H), 7.11-7.07 (m, 1H) 6.90 (d, J = 5.2 Hz, 1H). ^{13}C $\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 159.6, 158.3, 152.8, 138.6, 129.1, 123.6, 119.8, 116.2. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_9\text{BrN}_3$ 249.9974; found 249.9987. The regiochemistry of **25a** was verified by comparison to a sample of **25a** that was prepared from 2-chloro-4-bromopyrimidine.

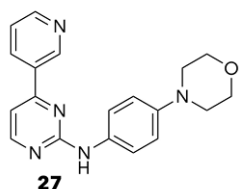


4-Iodo-N-phenylpyrimidin-2-amine (26a). Compound **26a** was prepared

according to the general cross-coupling procedure using $[(\text{IMes})\text{PdCl}_2]_2$ (4.1 mg, 0.005 mmol, 0.01 equiv), 2,4-diiodopyrimidine (165.9 mg, 0.55 mmol, 1.1 equiv),

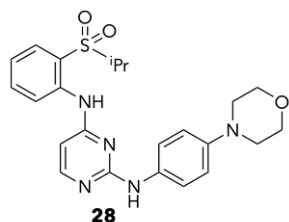
Cs_2CO_3 (326 mg, 1.0 mmol), aniline (46 μL , 0.5 mmol, 1 equiv), and xylene (1.25 mL). Analysis of the crude reaction mixture by ^1H NMR indicated a >99 : 1 ratio of C2:C4-functionalized products. Purification by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 80:20 (R_f = 0.42) provided **26a** as an orange solid (127.7 mg, 77% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 7.89 (d, J = 5.2 Hz, 1H), 7.63 (br s, 1H), 7.58-7.56 (m, 2H), 7.37-7.34 (m, 2H), 7.18 (d, J = 5.2 Hz, 1H), 7.12-7.08 (m, 1H). ^{13}C $\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 157.9, 155.3, 138.2, 131.1, 129.2, 123.9, 123.2, 119.9. HRMS (ESI Q-TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{10}\text{H}_9\text{IN}_3$ 297.9836; found 297.9851. The regiochemistry of **26a** was verified by comparison to a sample of **26a** that was prepared from 2-chloro-4-iodopyrimidine.

Synthesis and Characterization of Momelotinib Analogues



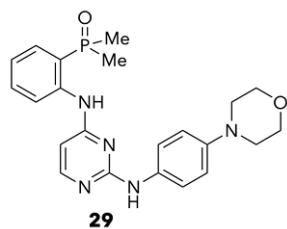
N-(4-morpholinophenyl)-4-(pyridin-3-yl)pyrimidin-2-amine (**27**). Inside a nitrogen filled glovebox, 4-chloro-*N*-(4-morpholinophenyl)pyrimidin-2-amine (**16a**, 72.7 mg, 0.25 mmol, 1 equiv), pyridin-3-ylboronic acid (30.7 mg, 0.25 mmol, 1 equiv), K₂CO₃ (69 mg, 0.5 mmol, 2 equiv) and Pd(PPh₃)₄ (12.4 mg, 0.011 mmol, 4.4 mol %) were combined in an oven dried 1-dram vial equipped with a Teflon coated stir bar. Dioxane (0.6 mL) was added and the vessel was sealed with a Teflon-lined cap and removed from the glovebox. The cap was removed, and de-ionized water (0.6 mL) was added. The headspace was then flushed with N₂ for 30-45 seconds and then sealed again with the Teflon-lined cap. The reaction mixture was left to stir vigorously at 90 °C for 72 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and filtered through Celite™. The filtrate was concentrated under vacuum and purified by flash column chromatography on silica gel using an eluent of CH₂Cl₂:MeOH = 95:5 (*R_f* = 0.38), providing **27** as a yellow solid (46.7 mg, 56% yield). ¹H NMR (400 MHz, CDCl₃, δ): 9.28 (br s, 1H), 8.72 (br s 1H), 8.47 (d, *J* = 5.1 Hz, 1H), 8.34 (d, *J* = 8.4 Hz, 1H), 7.58-7.56 (m, 2H), 7.44-7.41 (m, 1H), 7.32 (br s, 1H), 7.12 (d, *J* = 5.1 Hz, 1H), 6.99–6.97 (multiple overlapping signals, 2H), 3.90-3.88 (m, 4H), 3.16-3.14 (m, 4H). ¹³C {¹H} NMR (101 MHz, CDCl₃, δ): 162.7, 160.7, 159.0, 151.5, 148.7, 147.2, 134.6, 133.0, 132.5, 123.8, 121.5, 116.9, 107.9, 67.0, 50.3. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₁₉H₂₀N₅O 334.1662; found 334.1676.

*N*⁴-(2-(isopropylsulfonyl)phenyl)-*N*²-(4-morpholinophenyl)pyrimidine-2,4-diamine (**28**). Open



to air, 4-chloro-*N*-(4-morpholinophenyl)pyrimidin-2-amine (**16a**, 72.7 mg, 0.25 mmol, 1 equiv), and 2-(isopropylsulfonyl)aniline (49.8 mg, 0.25 mmol, 1 equiv) were combined in a 100 mL round bottom flask

equipped with a Teflon coated stir bar. Isopropyl alcohol (10 mL) was added, followed by aqueous 1 M HCl (0.5 mL). A reflux condenser was attached, and the reaction mixture was stirred at 70 °C for 24 h. The reaction mixture was cooled to 0 °C and triethylamine (1 mL) was added. The resulting solution was extracted with ethyl acetate (3 x 50 mL). The crude mixture was dried under vacuum and purified by flash column chromatography on silica gel using an eluent of CH₂Cl₂:MeOH = 95:5 (*R*_f = 0.31), providing **28** as a white solid containing ~5% of an impurity that may correspond to the protonated form of **28** (52.5 mg, 46% yield). ¹H NMR (400 MHz, CDCl₃, δ): 9.31 (br s, 1H), 8.37 (d, *J* = 8.4 Hz, 1H), 7.98-7.96 (m, 1H), 7.88-7.86 (m, 1H), 7.57-7.53 (m, 1H), 7.44-7.42 (m, 2H), 7.25-7.23 (m, 1H), 7.20-7.16 (m, 1H), 6.89-6.87 (m, 2H), 6.19-6.18 (d, *J* = 8.4 Hz, 1H), 3.88-3.86 (m, 4H), 3.20 (septet, *J* = 6.8 Hz, 1H), 3.15-3.12 (m, 4H), 1.29 (d, *J* = 6.8 Hz, 6H). ¹³C {¹H} NMR (101 MHz, CDCl₃, δ): 160.2, 159.0, 155.0, 147.9, 139.0, 134.8, 131.6, 131.4, 124.3, 123.5, 123.1, 122.4, 116.4, 99.0, 67.0, 56.0, 50.1, 15.4. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₂₃H₂₈N₅O₃S 454.1907; found 454.1909.

Dimethyl(2-[(2-[(4-morpholinophenyl)amino]pyrimidin-4-yl)amino]phenyl)phosphine oxide

(29). Open to air, 4-chloro-*N*-(4-morpholinophenyl)pyrimidin-2-amine (**16a**, 72.7 mg, 0.25 mmol, 1 equiv), and (2-aminophenyl)dimethylphosphine oxide (42.3 mg, 0.25 mmol, 1 equiv)

were combined in a 100 mL round bottom flask equipped with a Teflon coated stir bar. Isopropyl alcohol (10 mL) was added, followed by aqueous 1 *M* HCl (0.5 mL). A reflux condenser was attached, and the reaction mixture was stirred at 70 °C for 24 h. The reaction mixture was cooled to 0 °C and triethylamine (1 mL) was added. The resulting solution was extracted with ethyl acetate (3 x 50 mL). The crude mixture was dried under vacuum and purified by flash column chromatography on silica gel using an eluent of CH₂Cl₂:MeOH = 95:5 (*R_f* = 0.17), providing **29** as a white solid (83.9 mg, 79% yield). ¹H NMR (400 MHz, CDCl₃, δ): 10.91 (br s, 1H), 8.61-8.58 (m, 1H), 7.93 (d, *J* = 5.8 Hz, 1H), 7.80 (br s, 1H), 7.47-7.45 (m, 2H), 7.42-7.38 (m, 1H), 7.23-7.20 (m, 1H), 7.08-7.04 (m, 1H), 6.91-6.89 (m, 2H), 6.12 (d, *J* = 5.8 Hz, 1H), 3.88-3.86 (m, 4H), 3.14-3.12 (m, 4H), 1.83-1.80 (d, *J* = 13.2 Hz, 6H). ¹³C{¹H} NMR (101 MHz, CDCl₃, δ): 160.5, 158.7, 153.2, 147.9, 144.5 (d, 3JCP = 2.9 Hz), 132.8 (d, 3JCP = 2.2 Hz), 131.8, 129.8 (d, 2JCP = 10.9 Hz), 122.8, 122.32, 122.29 (d, 2JCP = 20 Hz), 119.0 (d, 1JCP = 94.7 Hz), 116.5, 100.1, 67.1, 50.1, 19.0 (d, 1JCP = 71.8 Hz) ³¹P NMR (162 MHz, CDCl₃, δ): 45.2. HRMS (ESI Q-TOF) *m/z*: [M+H]⁺ Calcd for C₂₂H₂₇N₅O₂P 424.1897; found 424.1911.

Experiments to Investigate Mechanism

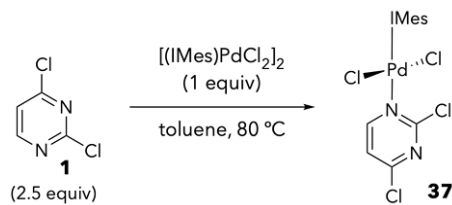
Suzuki Cross-Coupling of 1 Procedure. Inside a nitrogen filled glovebox, Pd-PEPPSI-IMes (1.0 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (**1**, 13.0 mg, 0.088 mmol, 1.1 equiv), phenylboronic acid (9.8 mg, 0.08 mmol, 1 equiv), and Cs₂CO₃ (52 mg, 0.16 mmol, 2.0 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline was then added if indicated in Table S2 (1.9 μ L, 0.02 mmol, 0.25 equiv) followed by *p*-xylene (0.2 mL). The vial was sealed with a Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and filtered through Celite™. The filtrate was concentrated under vacuum and mesitylene was added as an internal standard (11.1 μ L, 0.080 mmol, 1.0 equiv). The residue was taken up in CDCl₃ and analyzed by ¹H NMR. Compounds **31a** and **31b** have been previously reported.¹¹

Results

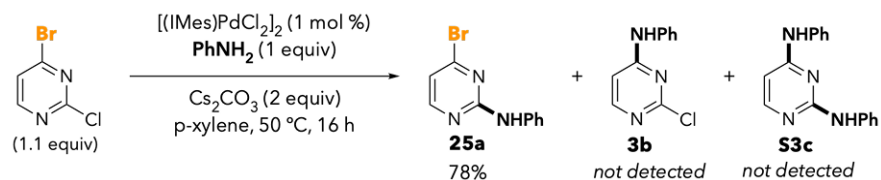
Table C.2. Suzuki-Miyaura Cross-Coupling of **1** Under Conditions Optimized For C2-Amination^a

entry	trial	PhNH ₂ (equiv)	unreacted 1 (%)	31a (%)	31b (%)	3a (%)	3b (%)
1	1	0	30	1	21	--	--
2	2	0	52	2	25	--	--
3	average	0	41	2	23	--	--
4	1	0.25 equiv	30	1	25	20	n.d.
5	2	0.25 equiv	44	n.d.	20	21	n.d.
6	average	0.25 equiv	37	1	23	21	n.d.

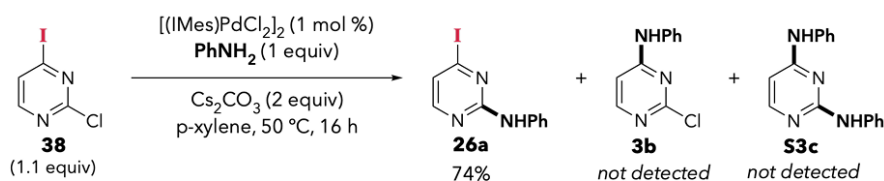
^a "n.d." = not detected. "--" = not applicable.



Synthesis and Characterization of 38 Procedure. Procedure. In a nitrogen-filled glovebox, [(IMes)PdCl₂]₂ (113.2 mg, 0.1 mmol, 1 equiv), 2,4-dichloropyrimidine (1, 37.2 mg, 0.25 mmol, 2.5 equiv), and toluene (10 mL) were combined in an oven-dried 100 mL round bottom flask equipped with a Teflon-coated stir bar. A condenser was attached and the top of the condenser was sealed with a rubber septum. The reaction apparatus was removed from the glovebox and the reaction mixture was stirred at 80 °C for 16 h under a N N Cl Cl Pd-PEPPSI-IMes (2 mol %) PhB(OH)₂ (1 equiv) PhNH₂ (0 or 0.25 equiv) C_s2CO₃ (2 equiv) p-xylene, 50 °C, 16 h N N Ph Cl (1.1 equiv) N N Cl NHPH + 1 30b 3a N N Cl Ph 30a N N Cl NHPH 3b + + Pd Cl Cl N N Cl Cl IMes N N Cl Cl (2.5 equiv) [(IMes)PdCl₂]₂ (1 equiv) toluene, 80 °C 1 37 19 nitrogen atmosphere. The crude reaction mixture was filtered through a pad of silica layered over Celite™. The silica/Celite pad was washed with CH₂Cl₂ (20 mL) and the combined filtrate was concentrated under reduced pressure. Pentane was added to the resulting residue, resulting in a precipitate. The precipitate was separated from the supernatant by filtration and then dried under vacuum to afford 37 as pale yellow crystals contaminated with 43 mol % of unreacted 1 and 20 mol % of [(IMes)PdCl₂]₂ (53.5 mg, 30% yield after adjusting for impurities). ¹H NMR (400 MHz, CDCl₃, δ): 8.35 (d, *J* = 5.8 Hz, 1H), 7.16 (d, *J* = 5.8 Hz, 1H), 7.11 (s, 2H), 7.08 (s, 4H), 2.40 (s, 6H), 2.34 (s, 12 H). ¹³C{¹H} NMR (101 MHz, CDCl₃, δ): 163.3, 161.1, 160.2, 150.0, 139.6, 136.7, 135.0, 129.3, 124.4, 120.3, 21.3, 19.2. Structure was confirmed by XRD analysis (see pp S47 S55).



Pyrimidine Substrates with Mixed Halides Including 39. Inside a nitrogen-filled glovebox, 4-bromo-2-chloropyrimidine (17.0 mg, 0.088 mmol, 1.1 equiv), Cs_2CO_3 (52 mg, 0.16 mmol, 2.0 equiv) and $[(\text{IMes})\text{PdCl}_2]_2$ (0.8 mg, 0.0008 mmol, 0.01 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline (7.3 μL , 0.08 mmol, 1 equiv) and *p*-xylene (0.2 mL) were added and the vial was sealed with a Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate and filtered through Celite™. The filtrate was concentrated and mesitylene was added as an internal standard (11.1 μL , 0.080 mmol, 1.0 equiv). The residue was taken up in CDCl_3 (0.4 mL) for ^1H NMR analysis, which indicated a 78% yield of **26a**, 20% yield of unreacted 4-bromo-2-chloropyrimidine, and none of **3b** or **S3c**.



Inside a nitrogen-filled glovebox, 2-chloro-4-iodopyrimidine (21.1 mg, 0.088 mmol, 1.1 equiv), Cs_2CO_3 (52 mg, 0.16 mmol, 2.0 equiv) and $[(\text{IMes})\text{PdCl}_2]_2$ (0.8 mg, 0.0008 mmol, 0.01 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline (7.3 μL , 0.08 mmol, 1 equiv) and *p*-xylene (0.2 mL) were added and the vial was sealed with a

Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate and filtered through Celite™. The filtrate was concentrated and mesitylene was added as an internal standard (11.1 µL, 0.080 mmol, 1.0 equiv). The residue was taken up in CDCl₃ (0.4 mL) for ¹H NMR analysis, which indicated a 74% yield of **27a**, 27% yield of unreacted **39**, and none of **3b** or **S3c**.

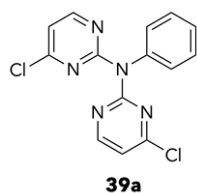
Evaluation of Pd⁰ Sources and Characterization of Diarylation. General Procedure for Evaluation of Pd Sources. Inside a nitrogen-filled glovebox, the indicated palladium complex, additional ligand (if applicable), 2,4-dichloropyrimidine (**1**, 13.0 mg, 0.088 mmol, 1.1 equiv), and Cs₂CO₃ (52.0 mg, 0.16 mmol, 2.0 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline (7.4 µL, 0.08 mmol, 1 equiv) and *p*-xylene (0.2 mL) were added and the vial was sealed with a PTFE-lined cap and removed from the glovebox. The reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate (~3 mL) and filtered through Celite™. Mesitylene (3.7 µL, 0.027 mmol, 0.33 equiv) was added as an internal standard, and the residue was taken up in CDCl₃ for analysis by ¹H NMR spectroscopy, which enabled quantification of **1**, **3a**, **3b**, and the sum of (**40a** + **40b** + **40c**). The three diarylated isomers have overlapping diagnostic signals by ¹H NMR, so NMR analysis provides the total yield of the three isomers but not yields of the individual isomers. The sample was then further analyzed by GC-FID to determine the ratio of **40a:40b:40c**, assuming identical response factors of the three isomers. The yields of **40a**, **40b**, **40c** were then calculated from their ratio and the total yield of (**40a** + **40b** + **40c**). Samples of **40a** and **40b** were isolated and characterized (see below). **40c** was identified on the basis of its appearance as a third compound that can be detected by GCMS that has the same molecular weight as **40a** and **40b**, and whose

diagnostic signal in the ^1H NMR spectrum (corresponding to the proton on carbon 6 of the pyrimidine rings) overlaps with one of the signals of **40b**.

Table C.3. Evaluation of Pd^0 Sources for Amination of **1**^a

entry	trial	[Pd] (mol %)	IMes•HCl (mol %)	1 (%)	3a (%)	3b (%)	40a (%)	40b (%)	40c (%)	C2 : C4
1	1	[(IMes)PdCl ₂] ₂	0	0.9	88.1	n.d.	2.2	<1	n.d.	>99 : 1
2	2			1.9	86.8	n.d.	2.1	<1	n.d.	>99 : 1
3	avg			1.4	87.5	n.d.	2.2	<1	n.d.	>99 : 1
4	1	PdCl ₂ (2)	2	24.8	61.6	<1	0.9	0.8	n.d.	80 : 1
5	2			26.7	64.8	n.d.	1.0	0.9	n.d.	75 : 1
6	avg			25.8	63.2	<1	1.0	0.9	n.d.	78 : 1
7	1	Pd ⁰ (dba) ₃	2	34.2	15.8	n.d.	3.9	5.0	6.1	1.7 : 1
8	2			39.4	14.3	n.d.	2.5	4.4	9.0	1.1 : 1
9	avg			36.8	15.1	n.d.	3.2	4.7	7.6	1.3 : 1
10	1	Pd ⁰ (PPh ₃) ₄	2	46.3	14.9	n.d.	1.6	5.0	6.5	1.3 : 1
11	2			31.2	15.1	n.d.	2.0	6.1	9.9	1.0 : 1
12	avg			38.8	15.0	n.d.	1.8	5.6	8.2	1.1 : 1
13	1	IMesPd ⁰ (dvds)	2	17.8	27.9	1.4	9.1	8.6	2.4	3.7 : 1
14	2			23.7	25.1	2.3	7.4	6.8	2.8	3.2 : 1
15	avg			20.8	26.5	1.9	8.3	7.7	2.6	3.4 : 1

^aNMR yields. C2:C4 ratio = [**3a** + (2 x **40a**) + **40b**] / [**3b** + **40b** + (2 x **40c**)].

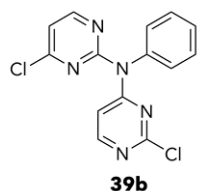


Synthesis and Characterization of 39a. Inside a nitrogen-filled glovebox,

[(IMes)Pd⁰(vs)] (6.0 mg, 0.01 mmol, 0.02 equiv), 2,4 dichloropyrimidine (**1**, 82.0 mg, 0.55 mmol, 1.1 equiv), Cs₂CO₃ (226 mg, 2 mmol, 2.0 equiv), and

aniline (46 μL , 0.5 mmol, 1 equiv) were combined in an oven dried 1-dram vial equipped with a Teflon-coated stir bar. *p*-Xylene (1.25 mL) was added and the vial was sealed with a Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred at 50 °C for 16 h. The reaction mixture was diluted with ethyl acetate and filtered through Celite™. The filtrate was

concentrated under vacuum, and a portion of the residue was taken up in CDCl_3 for ^1H NMR analysis, which indicated a product ratio of 3a:3b:(39a + 39b + 39c) = 42 : 42 : 16. The NMR sample was recombined with the bulk crude residue, solvent was removed under vacuum, and the residue was purified by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 50:50 (R_f = 0.34) to provide 39a as a yellow oil (12.7 mg, 8% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.43 (d, J = 5.2 Hz, 2H), 7.46-7.42 (m, 2H), 7.36-7.32 (m, 1H), 7.25-7.24 (m, 2H), 7.02 (d, J = 5.2 Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 164.2, 162.2, 161.8, 159.2, 129.7, 128.0, 127.5, 116.6. X-ray quality crystals were grown from chloroform at 25 °C and the regiochemistry was verified by X-ray diffraction (see pp. S55-S58).

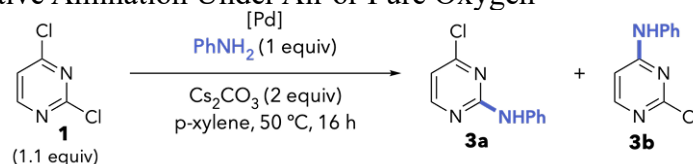


Synthesis and Characterization of 39b. Inside a nitrogen-filled glovebox, Pd2dba3 (4.8 mg, 0.005 mmol, 0.01 equiv), IMes•HCl (3.4 mg, 0.01 mmol, 0.02 equiv), 2,4-dichloropyrimidine (1, 82.0 mg, 0.55 mmol, 1.1 equiv), Cs_2CO_3 (326 mg, 1 mmol, 2.0 equiv), and aniline (46 μL , 0.5 mmol, 1 equiv) were combined in an oven dried 1-dram vial equipped with a Teflon-coated stir bar. *p*-Xylene (1.25 mL) was added and the vial was sealed with a Teflon-lined cap and removed from the glovebox. The reaction mixture was stirred at 50 °C for 16 h. The reaction mixture was diluted with ethyl acetate and filtered through Celite™. The filtrate was concentrated under vacuum, and a portion of the residue was taken up in CDCl_3 for ^1H NMR analysis, which indicated a product ratio of 3a:3b:(39a + 39b + 39c) = 65 : 21 : 14. The NMR sample was recombined with the bulk crude residue, solvent was removed under vacuum, and the residue was purified by flash column chromatography on silica gel using an eluent of hexanes:EtOAc = 50:50 (R_f = 0.31) to provide 39b as a yellow oil (5.8 mg, 4% yield). ^1H NMR (400 MHz, CDCl_3 , δ): 8.43 (d, J = 5.2 Hz, 1H), 8.38 (d, J = 5.8 Hz, 1H), 7.51-7.47 (m, 2H), 7.43-

7.40 (m, 1H), 7.31 (d, $J = 5.8$ Hz, 1H), 7.23-7.21 (m, 2H), 7.06 (d, $J = 5.2$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3 , δ): 163.8, 163.1, 161.7, 161.2, 160.5, 159.3, 158.9, 130.1, 128.7, 128.3, 116.8, 110.8.

Evaluating Sensitivity to Oxygen. Procedure. Inside a nitrogen-filled glovebox, Pd precatalyst (0.0008 mmol or 0.0016 mmol, 1 or 2 mol %), 2,4-dichloropyrimidine (**1**, 13.0 mg, 0.088 mmol, 1.1 equiv), and Cs_2CO_3 (52 mg, 0.16 mmol, 2.0 equiv) were combined in an oven-dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline (7.4 μL , 0.08 mmol, 1 equiv) and *p*-xylene (0.2 mL) were added and the vial was sealed with a PTFE-lined silicone septum cap and removed from the glovebox. For the “open-to-air” trials, a 16G needle was inserted through the septum into the headspace of the vial and left in position for the duration of the reaction time. For the “ O_2 ” reactions, an O_2 -filled balloon was fitted with a 21G needle. This needle and a 23G outgassing needle were inserted through the septum. The 21G (O_2 inlet) needle was submerged into the reaction mixture and the mixture was sparged with O_2 for ~30 seconds. The 21G (O_2 inlet) needle was then positioned in the headspace of the vial and the outgassing needle was removed. The O_2 -filled balloon was kept in place for the duration of the reaction time. The reaction mixture was stirred vigorously at 50 $^\circ\text{C}$ for 16 h. The reaction mixture was diluted with ethyl acetate (~3 mL) and mesitylene (4.9 μL , 0.035 mmol, 0.44 equiv) was added as an internal standard. The mixture was filtered through CeliteTM before GC analysis.

Table C.4. C2-Selective Amination Under Air or Pure Oxygen^a



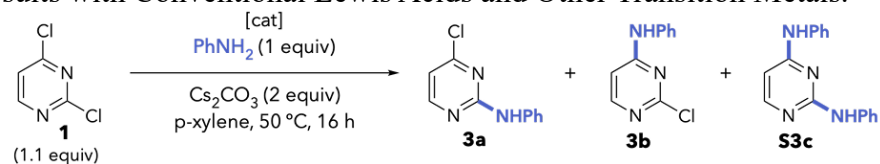
entry	trial	atmosphere	[Pd] (mol %)	unreacted 1 (%)	3a (%)	3b (%)	3a : 3b
1	1	open-to-air	Pd-PEPPSI-IMes (2)	5.1	91.4	n.d.	>99 : 1
2	2			3.4	92.9	n.d.	>99 : 1
3	avg			4.3	92.2	n.d.	>99 : 1
4	1	open-to-air	[(IMesPdCl ₂) ₂]	4.8	92.3	n.d.	>99 : 1
5	2			2.9	91.1	n.d.	>99 : 1
6	avg			3.9	91.7	n.d.	>99 : 1
7	1	O ₂	Pd-PEPPSI-IMes (2)	2.7	94.6	n.d.	>99 : 1
8	2			5.1	91.6	n.d.	>99 : 1
9	avg			3.9	93.1	n.d.	>99 : 1
10	1	O ₂	[(IMesPdCl ₂) ₂]	4.9	91.6	n.d.	>99 : 1
11	2			3.6	90.7	n.d.	>99 : 1
12	avg			4.3	91.2	n.d.	>99 : 1

^a GC yields calibrated against mesitylene.

Evaluation of Conventional Lewis Acids and Other Transition Metals. Procedure

Open to air, catalyst (0.0016 mmol, 0.02 equiv or 0.080 mmol, 1 equiv), 2,4-dichloropyrimidine (**1**, 13.0 mg, 0.088 mmol, 1.1 equiv), and Cs₂CO₃ (52 mg, 0.16 mmol, 2.0 equiv) were combined in an oven dried 1-dram vial equipped with a Teflon-coated stir bar. Aniline (7.3 μ L, 0.08 mmol, 1 equiv) and *p*-xylene (0.2 mL) were added. The vial was immediately fitted with a PTFE-lined silicone septum cap equipped with an N₂-ingass needle and an outgassing needle. The headspace of the reaction was flushed with N₂ for 30–45 seconds and then the reaction mixture was stirred vigorously at 50 °C for 16 h. The reaction mixture was then diluted with ethyl acetate and filtered through Celite™. The filtrate was concentrated under vacuum and mesitylene was added as an internal standard (11.1 μ L, 0.080 mmol, 1.0 equiv). The residue was taken up in CDCl₃ (0.4 mL) and analyzed by ¹H NMR spectroscopy.

Table C.5. Results with Conventional Lewis Acids and Other Transition Metals.^a



entry	trial	catalyst (mol %)	3a (%)	3b (%)	S3c (%)	3a : 3b
1	1	Zn(OTf) ₂ (2)	<1	<1	<1	--
2	2		<1	<1	<1	--
3	avg		<1	<1	<1	--
4	1	Zn(OTf) ₂ (100)	4.8	9.1	<1	1 : 2
5	2		5.3	9.5	<1	1 : 2
6	avg		5.1	9.3	<1	1 : 2
7	1	ZnCl ₂ (2)	<1	<1	<1	--
8	2		<1	<1	<1	--
9	avg		<1	<1	<1	--
10	1	ZnCl ₂ (100)	7.4	2.9	<1	3 : 1
11	2		7.7	3.5	<1	2 : 1
12	avg		7.6	3.2	<1	2 : 1
13	1	BF ₃ .OEt (2)	<1	<1	<1	--
14	2		<1	<1	<1	--
15	avg		<1	<1	<1	--
16	1	BF ₃ .OEt (100)	1.9	4.3	6.2	1 : 2
17	2		2.3	4.7	5.1	1 : 2
18	avg		2.1	4.5	5.7	1 : 2
19	1	Al(OTf) ₂ (2)	<1	<1	<1	--
20	2		<1	<1	<1	--
21	avg		<1	<1	<1	--
22	1	Al(OTf) ₂ (100)	2.5	4.8	<1	1 : 2
23	2		2.9	5.1	<1	1 : 2
24	avg		2.7	5.0	<1	1 : 2
25	1	InCl ₃ (2)	<1	<1	<1	--
26	2		<1	<1	<1	--
27	avg		<1	<1	<1	--
28	1	InCl ₃ (100)	<1	<1	<1	--
29	2		<1	<1	<1	--
30	avg		<1	<1	<1	--
31	1	AgNO ₃ (2)	<1	<1	<1	--
32	2		<1	<1	<1	--
33	avg		<1	<1	<1	--
34	1	AgNO ₃ (100)	12.0	12.6	<1	1 : 1
35	2		12.4	12.2	<1	1 : 1
36	avg		12.2	12.4	<1	1 : 1
37	1	LiCl (2)	<1	<1	<1	--
38	2		<1	<1	<1	--
39	avg		<1	<1	<1	--
40	1	LiCl (100)	<1	<1	<1	--
41	2		<1	<1	<1	--
42	avg		<1	<1	<1	--
43	1	AlCl ₃ (2)	1.6	2.9	3.2	1 : 2
44	2		1.5	3.2	3.5	1 : 2
45	avg		1.6	3.1	3.4	1 : 2
46	1	AlCl ₃ (100)	<1	24.9	<1	<1 : 99
47	2		<1	24.1	<1	<1 : 99
48	avg		<1	24.5	<1	<1 : 99
49	1	CuCl ₂ (2)	<1	<1	<1	--
50	2		<1	<1	<1	--
51	avg		<1	<1	<1	--
52	1	CuCl ₂ (100)	<1	<1	<1	--
53	2		<1	<1	<1	--
54	avg		<1	<1	<1	--
55	1	[(IPr)CuCl (2)]	7.7	6.7	<1	1 : 1

56	2		7.4	6.9	<1	1 : 1
57	avg		7.6	6.8	<1	1 : 1
58	1	[(IMes)AuCl] (2)	8.5	51.9	<1	1 : 6
59	2		8.7	52.3	<1	1 : 6
60	avg		8.6	52.1	<1	1 : 6
61	1	[(IPr)NiCpCl] (2)	1.1	2.1	<1	1 : 2
62	2		1.0	2.3	<1	1 : 2
63	avg		1.1	2.2	<1	1 : 2

^a NMR yields.

Computational Details

General Methods

Calculations were performed with Gaussian 16.¹² An ultrafine integration grid and the keyword 5d were used for all calculations. Geometry optimizations of stationary points were carried out in implicit solvent using the SMD solvation model for *p*-xylene. Unless otherwise indicated, geometry optimizations employed the MN15L¹³ functional, the 6-31G(d) basis set for C and H, the 6-31+G(d) basis set for N and Cl, and the LANL2DZ basis set and effective core potential for Pd and Cs. Frequency analyses were carried out at the same level as the optimizations to evaluate the zero-point vibrational energy and thermal corrections at the default temperature and pressure (298.15 K and 1 atm). The nature of the stationary points was determined in each case according to the appropriate number of negative eigenvalues of the Hessian matrix. Forward and reverse intrinsic reaction coordinate (IRC) calculations were carried out on the optimized transition structures to ensure that the TSs indeed connect the appropriate reactants and products.¹⁴ Multiple conformations/configurations were considered when applicable, and the lowest energy conformations are reported. Unless otherwise indicated, the final reported energies were obtained from single point energy calculations on the optimized geometries using MN15,¹⁵ the SMD solvation model (*p*-xylene), and the def2-TZVPD basis set. Gibbs free energy values are reported

after applying Cramer and Truhlar's quasi-harmonic approximation to vibrational entropy¹⁶ and Head-Gordon's quasi-harmonic approximation to vibrational enthalpy¹⁷ to frequencies that are less than 100 cm⁻¹. Reported free energies are corrected for temperature to 323.15 K and for concentration using the following values representative of those that could hypothetically be present after one catalytic turnover under experimental conditions that use a slight excess of aniline: [**1**] = 0.392 M, [PhNH₂] = 0.472 M, [(NHC)PdCl₂]₂] = 0.004 M, [all other Pd-containing species and **3a**, **3b**] = 0.008 M, [all Cs salts] = 0.001 M, and [**STSxxa** and **STSxxb**] = 0.392 M. All thermodynamic quantities were computed with the GoodVibes code.¹⁸ 3D images of structures were generated with CylView.¹⁹

Additional Versions of Computed Free Energy Diagram for
Proposed Mechanism

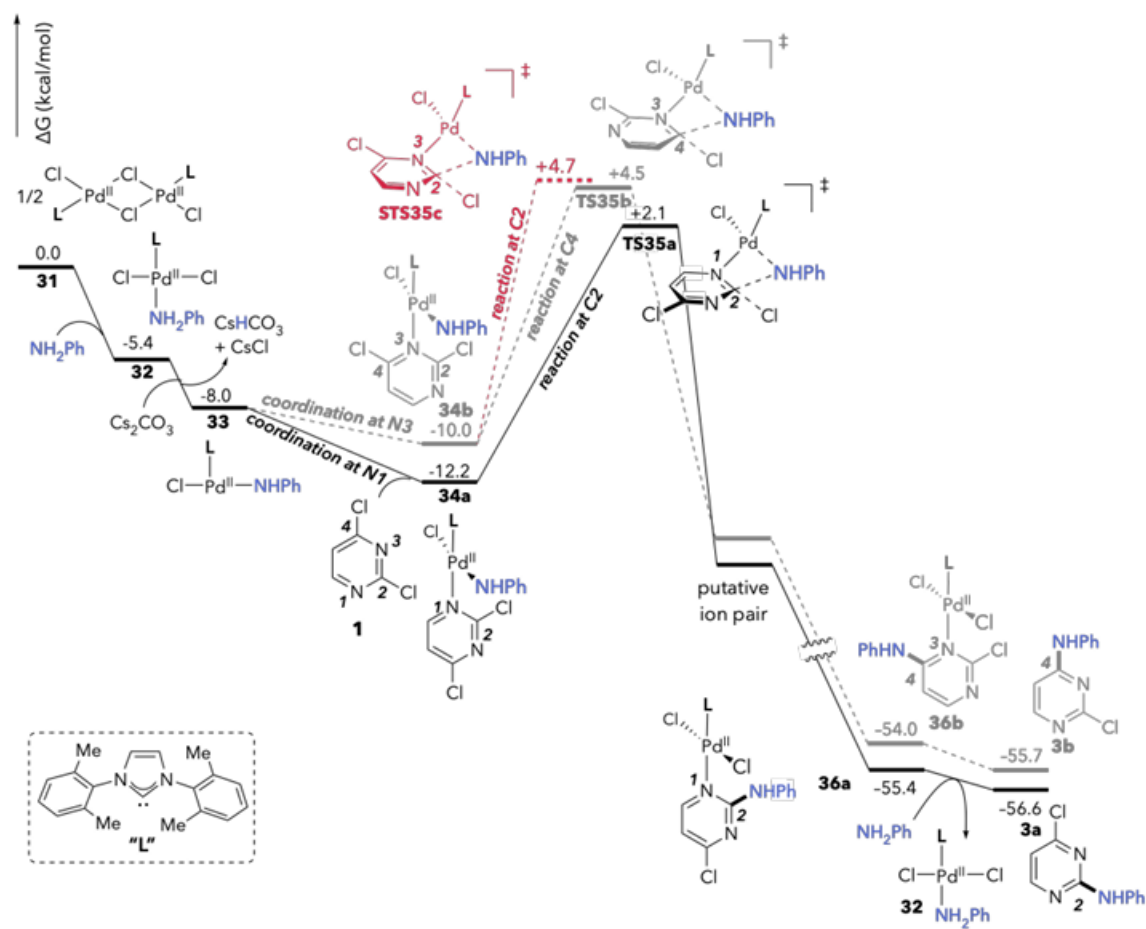


Figure C.1. Version of Fig. 3 with additional details: energetics of reaction at C2 via coordination at N3 (STS36c) and possible intermediacy of an ion pair prior to 37. Computed at the SMD(p-xylene)-MN15/def2TZVPD // SMD(p-xylene)-MN15L/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/LANL2DZ (Pd) level of theory.

Free Energy Diagrams for Oxidative Addition and Ligand Exchange

C. Oxidative Addition and Ligand Exchange

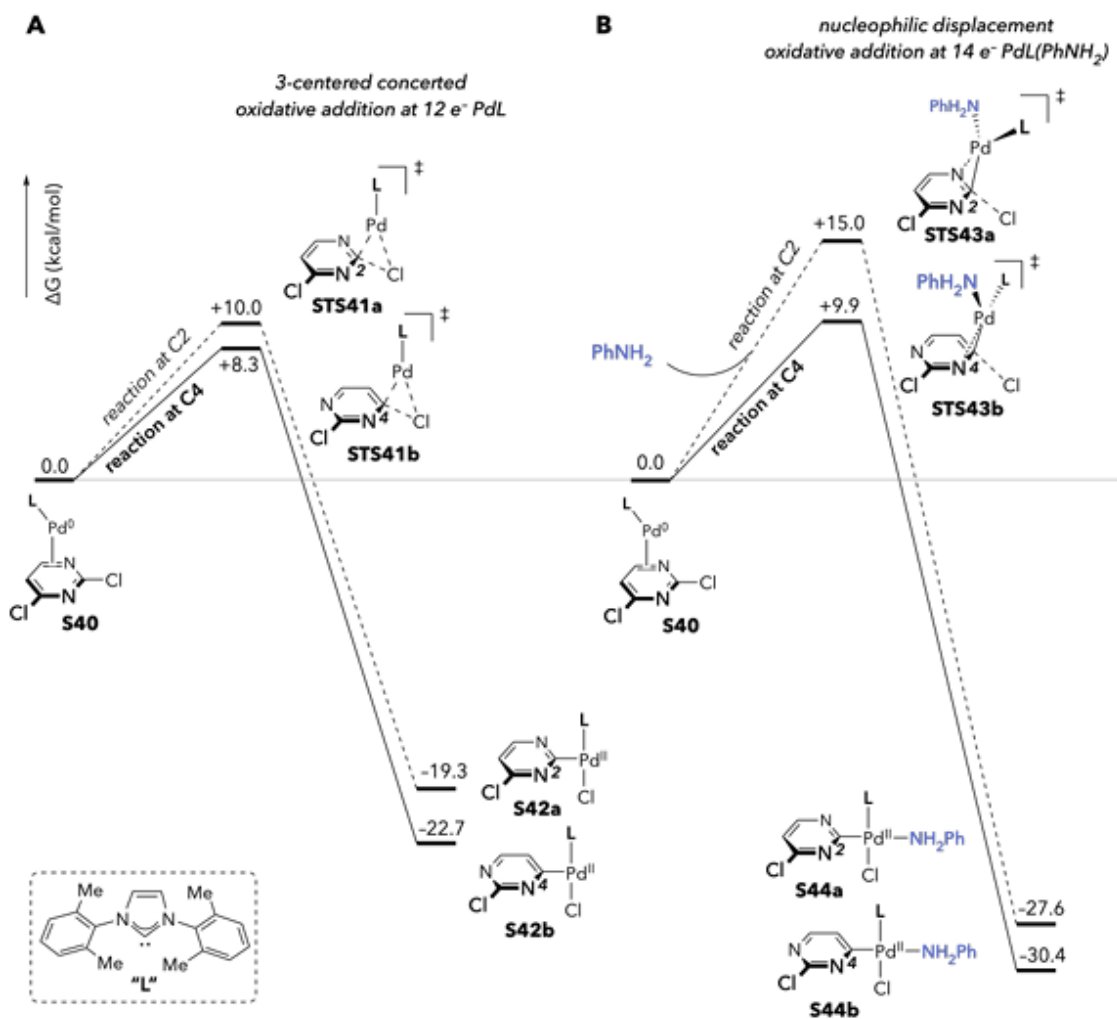


Figure C.2. Oxidative addition at (A) monoligated PdL through a 3-centered concerted mechanism and (B) at bisligated PdL(PhNH₂) through a nucleophilic displacement mechanism are both computed to be C4-selective and to be irreversible. Computed at the SMD(p-xylene)-MN15/6-311++G(2d,p)/SDD(Pd) // SMD(p-xylene)-wB97XD/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/SDD (Pd) level of theory.

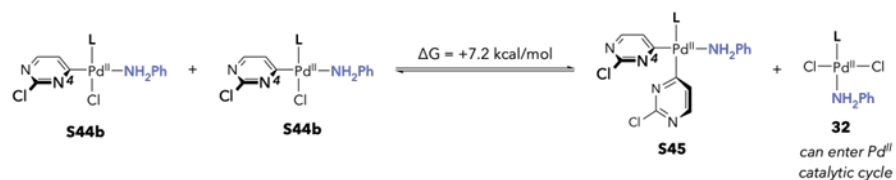


Figure C.3. If oxidative addition at Pd(0) does occur (favoring C4—Cl cleavage), it is energetically feasible for ligand exchange to result in complex 32, which is predicted to be a possible intermediate in the Pd^{II}-catalyzed C2-selective mechanism (see Fig. 3). Computed at the SMD(p-xylene)-MN15/6-311++G(2d,p)/SDD(Pd) // SMD(p-xylene)-wB97XD/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/SDD (Pd) level of theory.

Intrinsic Reaction Coordinate Calculation

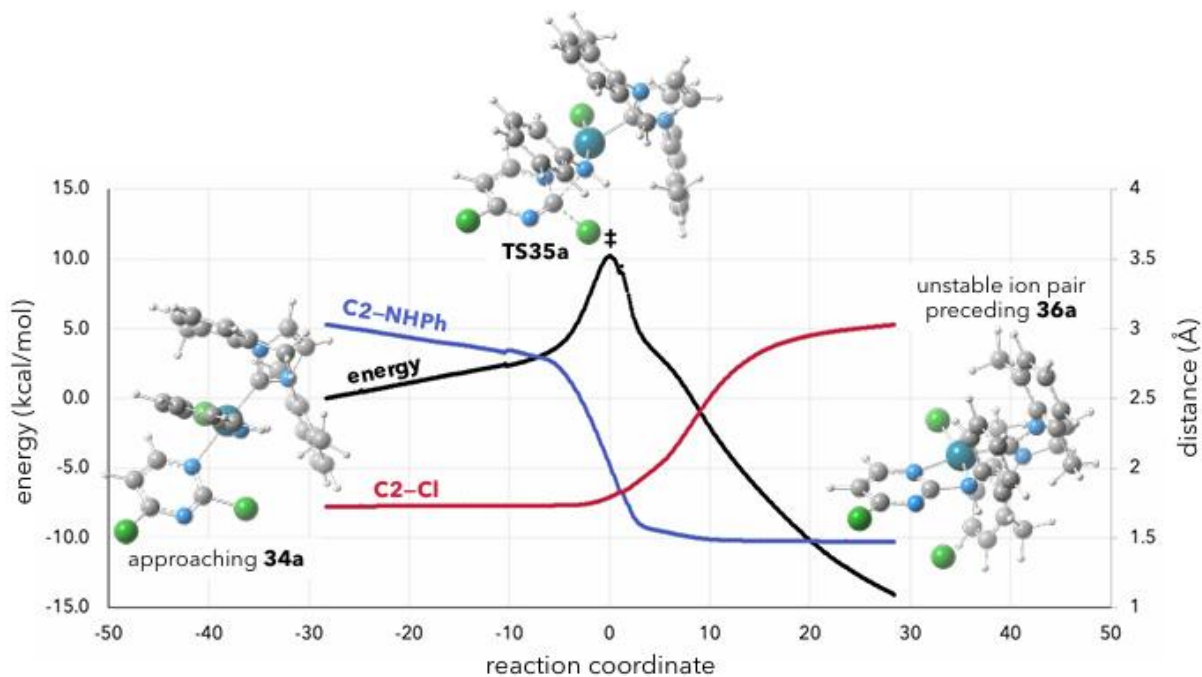


Figure C.4. Energy and bond parameters for IRC calculations on TS36a suggest initial formation of an ion pair prior to collapse to 37a, although the putative ion pair does not converge as a stable intermediate. Computed at the SMD(p-xylene)-MN15L/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/LANL2DZ (Pd) level of theory.

Relative Energies of TS36a and TS36b with other DFT
Methods

Table C.6. Relative free energies of TS36b and TS36b computed at various levels of theory.

entry	optimization method	single point energy method	$\Delta\Delta G_{\text{TS36b-TS36a}}$ (kcal/mol)
1	SMD(p-xylene)-MN15L/6-31G(d) (C, H)/ 6-31+G (N, Cl)/LANL2DZ (Pd)	SMD(p-xylene)-MN15/ def2TZVPD	2.5
2		SMD(p-xylene)-MN15/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	2.4
3		SMD(p-xylene)-MN15L/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	2.1
4		SMD(p-xylene)-M06/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	2.7
5		SMD(p-xylene)-B3LYP/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	4.3
6		SMD(p-xylene)-B3LYP-D3/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	3.0
7		SMD(p-xylene)- ω B97XD/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	3.4
8	SMD(p-xylene)-MN15L/6-31+G(d) (C, H, N, Cl)/SDD (Pd)	SMD(p-xylene)-MN15/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	2.2
9	SMD(p-xylene)- ω B97XD/6-31G(d) (C, H)/ 6-31+G (N, Cl)/LANL2DZ (Pd)	SMD(p-xylene)-MN15/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	3.1
10	SMD(p-xylene)- ω B97XD/6-31G(d) (C, H)/ 6-31+G (N, Cl)/SDD (Pd)	SMD(p-xylene)-MN15/ 6-311++G(2d,p) (C, H, N, Cl)/ SDD (Pd)	3.0

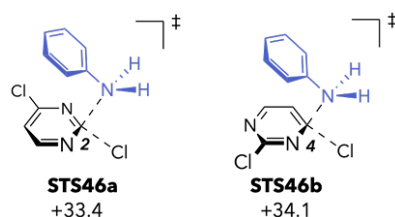
Other S_NAr Mechanisms

Figure C.5. The uncatalyzed S_NAr with neutral aniline is computed to be prohibitively high-energy. Computed free energies of activation in kcal/mol are relative to separated reactants 1 and PhNH₂. Computed at the SMD(p-xylene)-MN15/def2TZVPD // SMD(p-xylene)-MN15L/6-31G(d) (C, H)/6-31+G(d) (N, Cl) level of theory.

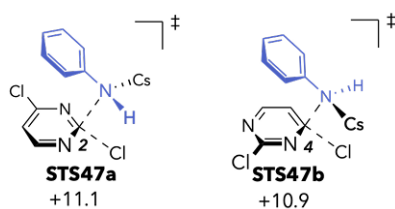


Figure C.6. The uncatalyzed S_NAr using deprotonated aniline (cesium salt) is predicted to give poor selectivity slightly favoring reaction at C4. Computed free energies of activation in kcal/mol are relative to separated reactants 1, PhNH₂, and Cs₂CO₃ (with CsHCO₃ as byproduct). The uncatalyzed S_NAr does not actually occur to a significant extent under the experimental conditions without Pd, suggesting that the computed barriers for this mechanism are artificially low (likely relating to difficulty modeling the energies of salts and their poor solubilities in p-xylene). Computed at the SMD(p-xylene)-MN15/def2TZVPD // SMD(p-xylene)-MN15L/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/LANL2DZ (Cs) level of theory.

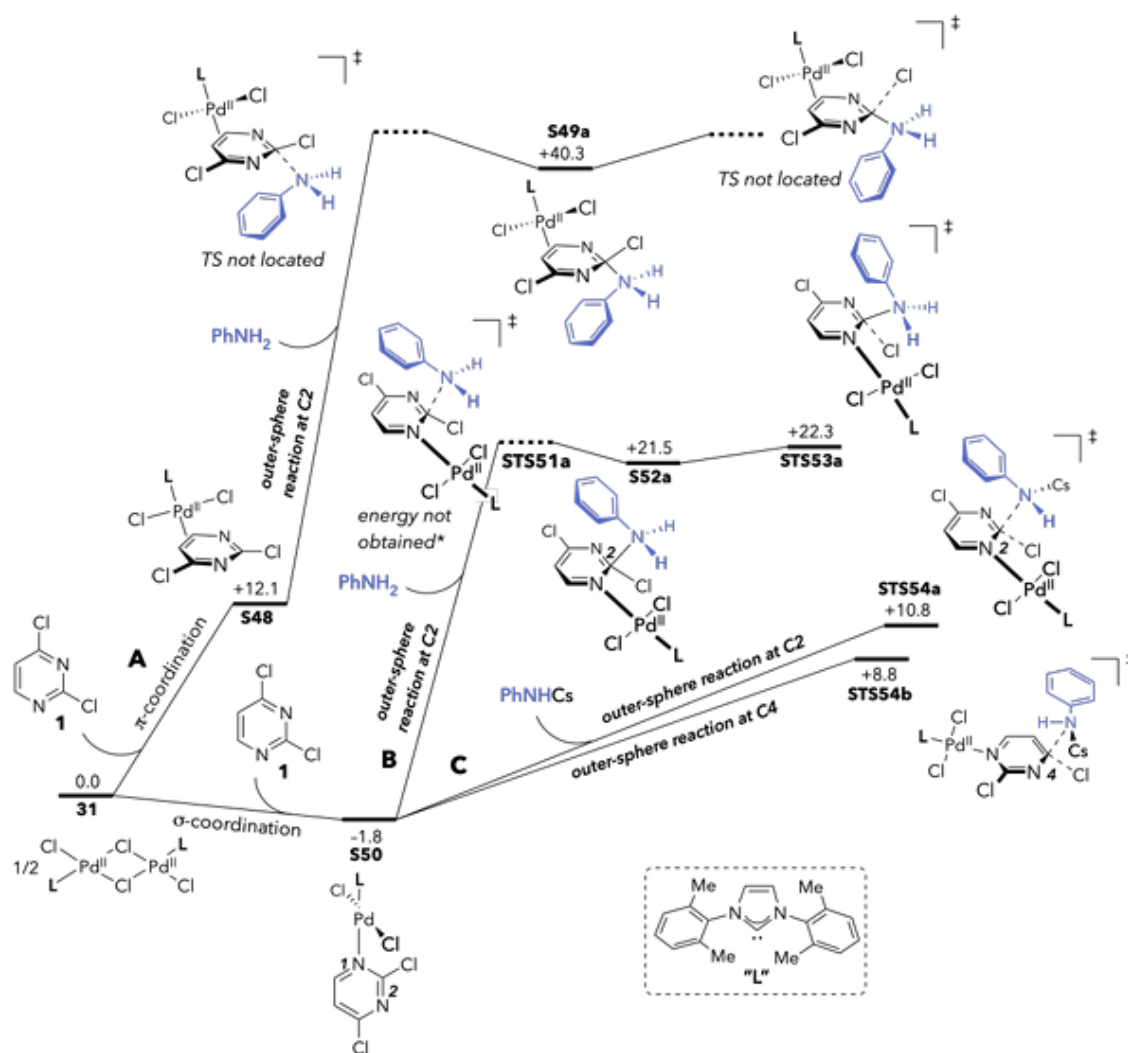


Figure C.7. Alternative outer-sphere Pd-catalyzed S_NAr mechanisms. (A) External attack of aniline on a p-complex is prohibitively high-energy. (B) External attack of aniline on a Lewis acid complex is a much higher-energy pathway than the computed inner-sphere mechanism in Fig. 3. (C) External attack of deprotonated aniline (cesium salt) on a Lewis acid complex would lead to C4-selectivity, contrary to experimental results. Computed free energies of activation in kcal/mol are relative to separated reactants $[LPdCl_2]_2$, **1**, $PhNH_2$, and Cs_2CO_3 (with $CsHCO_3$ as byproduct). Computed at the SMD(p-xylene)-MN15/def2TZVPD // SMD(p-xylene)-MN15L/6-31G(d) (C, H)/6-31+G(d) (N, Cl)/LANL2DZ (Pd, Cs) level of theory.

X-Ray Crystallographic Details

Compound 3a (CCDC Deposition Number: 2521688) Data for **3a** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK α radiation ($\lambda = 0.71073$ Å). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.

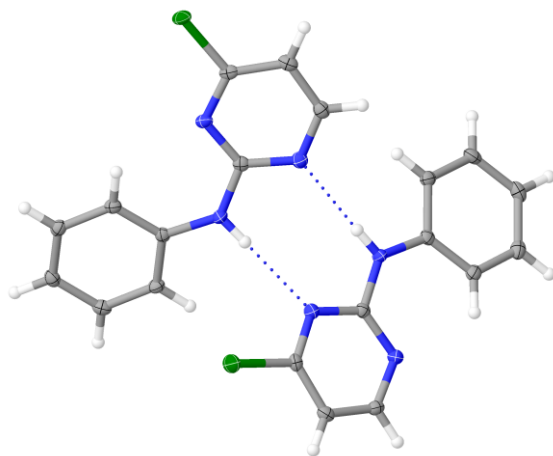


Figure C.8. X-ray crystal structure of **3a**.

Crystallographic Data for **3a**: C₁₀H₈ClN₃ ($M=205.64$, g/mol), monoclinic, $a = 21.5088(5)$ Å, $b = 6.9718(2)$ Å, $c = 12.4992(3)$ Å, $\alpha = 90^\circ$, $\beta = 103.1100(10)^\circ$, $\gamma = 90^\circ$, $V = 1825.47(8)$ Å³, $Z = 8$, $T = 100$ K, $\mu(\text{MoK}\alpha) = 0.375\text{mm}^{-1}$, $\rho_{\text{calc}} = 1.497\text{g/cm}^3$, 57236 reflections measured, 4184 unique ($R_{\text{int}} = 0.0503$, $R_{\text{sigma}} = 0.0221$), $R_1 = 0.0358$ ($I > 2\sigma(I)$) and wR_2 was 0.0884 (all data).

Table C.7. Crystal data and structure refinement for **3a**.

CCDC number	2521688
Empirical formula	C ₁₀ H ₈ ClN ₃
Formula weight	205.64
Temperature [K]	100
Crystal system	monoclinic
Space group (number)	$P2_1/c$ (14)
a [Å]	21.5088(5)
b [Å]	6.9718(2)
c [Å]	12.4992(3)
α [°]	90
β [°]	103.1100(10)
γ [°]	90
Volume [Å ³]	1825.47(8)
Z	8
ρ_{calc} [gcm ⁻³]	1.497
μ [mm ⁻¹]	0.375
$F(000)$	848
Crystal size [mm ³]	0.03×0.12×0.21
Crystal colour	colourless
Crystal shape	plate
Radiation	MoK α ($\lambda=0.71073$ Å)
2θ range [°]	5.83 to 54.98 (0.77 Å)
Index ranges	$-27 \leq h \leq 27$ $-9 \leq k \leq 9$ $-16 \leq l \leq 16$
Reflections collected	57236
Independent reflections	4184 $R_{\text{int}}=0.0503$ $R_{\text{sigma}} = 0.0221$
Completeness to $\theta = 25.242^\circ$	99.9 %
Data / Restraints / Parameters	4184 / 0 / 271
Goodness-of-fit on F^2	1.073
Final R indexes [$I \geq 2\sigma(I)$]	$R_1=0.0358$ $wR_2 = 0.0820$
Final R indexes [all data]	$R_1=0.0491$ $wR_2 = 0.0884$
Largest peak/hole [eÅ ⁻³]	0.71/−0.31

Table C.8. Atomic coordinates and U_{eq} [$\text{\AA}^2$] for **3a**.

Atom	x	y	z	U_{eq}
Cl1A	0.90488(2)	0.71315(7)	0.48627(3)	0.02194(12)
N1A	0.79173(6)	0.5842(2)	0.39758(11)	0.0147(3)
N2A	0.74655(6)	0.5585(2)	0.20401(11)	0.0148(3)
N3A	0.68862(6)	0.4880(2)	0.33790(11)	0.0141(3)
H3A	0.6912(9)	0.487(3)	0.4049(18)	0.017(5)
C1A	0.74341(7)	0.5444(2)	0.30947(13)	0.0134(3)
C2A	0.84397(8)	0.6526(2)	0.37390(13)	0.0153(3)
C3A	0.85312(8)	0.6788(2)	0.26874(13)	0.0157(3)
H3AA	0.891424	0.729778	0.254611	0.019
C4A	0.80190(8)	0.6244(2)	0.18608(13)	0.0154(3)
H4A	0.806124	0.634288	0.112253	0.018
C5A	0.63026(7)	0.4254(2)	0.27245(13)	0.0139(3)
C6A	0.58626(8)	0.3493(2)	0.32826(13)	0.0157(3)
H6A	0.596617	0.345320	0.406261	0.019
C7A	0.52776(8)	0.2796(2)	0.27093(14)	0.0186(3)
H7A	0.498594	0.227150	0.309848	0.022
C8A	0.51160(8)	0.2862(3)	0.15685(15)	0.0200(4)
H8A	0.471567	0.238593	0.117320	0.024
C9A	0.55489(8)	0.3634(2)	0.10170(14)	0.0183(3)
H9A	0.544033	0.368456	0.023717	0.022
C10A	0.61376(8)	0.4335(2)	0.15773(13)	0.0160(3)
H10A	0.642600	0.486504	0.118345	0.019
Cl1B	0.70374(2)	0.48907(6)	0.94234(3)	0.01874(13)
N1B	0.75097(6)	0.4396(2)	0.77116(11)	0.0147(3)
N2B	0.69587(6)	0.5049(2)	0.58503(11)	0.0155(3)
N3B	0.79916(6)	0.4009(2)	0.62276(11)	0.0155(3)
H3B	0.7953(9)	0.424(3)	0.5566(17)	0.019(5)
C1B	0.74741(7)	0.4486(2)	0.66257(13)	0.0139(3)
C2B	0.69927(8)	0.4970(2)	0.80254(13)	0.0155(3)
H2B	0.700139	0.492696	0.878832	0.019
C3B	0.64402(8)	0.5629(2)	0.73281(14)	0.0180(3)
H3BA	0.607977	0.605339	0.758318	0.022
C4B	0.64565(8)	0.5617(2)	0.62294(14)	0.0173(3)
H4B	0.608720	0.603818	0.571192	0.021
C5B	0.85795(7)	0.3186(2)	0.67531(13)	0.0136(3)
C6B	0.89334(8)	0.2340(2)	0.60673(13)	0.0157(3)
H6B	0.877239	0.235740	0.529405	0.019
C7B	0.95156(8)	0.1476(2)	0.65030(14)	0.0175(3)
H7B	0.975210	0.091559	0.602705	0.021
C8B	0.97558(8)	0.1425(2)	0.76327(14)	0.0168(3)
H8B	1.015117	0.081265	0.793439	0.020
C9B	0.94087(8)	0.2285(2)	0.83141(13)	0.0164(3)
H9B	0.957220	0.225933	0.908670	0.020
C10B	0.88272(8)	0.3182(2)	0.78908(13)	0.0155(3)
H10B	0.860053	0.378577	0.836814	0.019

Cl1X	0.5839(5)	0.6302(16)	0.5518(8)	0.031(3)
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Table C.9. Anisotropic displacement parameters ($\text{\AA}^2$) for 3a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1A	0.0159(2)	0.0307(2)	0.0175(2)	−0.00158(16)	0.00026(15)	−0.00527(17)
N1A	0.0137(6)	0.0159(7)	0.0143(6)	0.0001(5)	0.0028(5)	−0.0001(5)
N2A	0.0136(7)	0.0166(7)	0.0146(6)	0.0006(5)	0.0042(5)	0.0002(5)
N3A	0.0129(6)	0.0194(7)	0.0102(6)	0.0005(5)	0.0031(5)	−0.0009(5)
C1A	0.0129(7)	0.0118(7)	0.0156(7)	0.0008(6)	0.0036(6)	0.0011(6)
C2A	0.0130(7)	0.0149(8)	0.0165(8)	0.0000(6)	0.0002(6)	0.0013(6)
C3A	0.0139(8)	0.0140(8)	0.0200(8)	0.0003(6)	0.0055(6)	0.0002(6)
C4A	0.0174(8)	0.0148(8)	0.0154(7)	0.0017(6)	0.0068(6)	0.0016(6)
C5A	0.0124(7)	0.0130(7)	0.0163(8)	0.0005(6)	0.0033(6)	0.0025(6)
C6A	0.0160(8)	0.0157(8)	0.0159(8)	0.0020(6)	0.0048(6)	0.0025(6)
C7A	0.0126(7)	0.0185(8)	0.0260(9)	0.0024(7)	0.0073(6)	0.0009(6)
C8A	0.0130(8)	0.0194(8)	0.0254(9)	−0.0016(7)	−0.0003(6)	0.0006(7)
C9A	0.0194(8)	0.0188(8)	0.0151(8)	−0.0015(6)	0.0008(6)	0.0039(7)
C10A	0.0158(8)	0.0171(8)	0.0151(8)	0.0001(6)	0.0038(6)	0.0023(6)
Cl1B	0.0192(2)	0.0266(2)	0.0117(2)	−0.00269(16)	0.00619(15)	−0.00435(17)
N1B	0.0151(7)	0.0162(7)	0.0137(6)	−0.0008(5)	0.0052(5)	−0.0028(5)
N2B	0.0146(7)	0.0156(7)	0.0160(7)	−0.0005(5)	0.0029(5)	−0.0010(5)
N3B	0.0153(7)	0.0210(7)	0.0105(6)	0.0010(5)	0.0037(5)	0.0016(6)
C1B	0.0143(7)	0.0122(7)	0.0154(7)	−0.0010(6)	0.0037(6)	−0.0025(6)
C2B	0.0184(8)	0.0147(8)	0.0146(7)	−0.0034(6)	0.0063(6)	−0.0051(6)
C3B	0.0163(8)	0.0169(8)	0.0223(8)	−0.0029(7)	0.0075(7)	−0.0013(7)
C4B	0.0150(8)	0.0159(8)	0.0205(8)	−0.0002(6)	0.0032(6)	−0.0003(6)
C5B	0.0127(7)	0.0131(8)	0.0150(7)	0.0014(6)	0.0033(6)	−0.0010(6)
C6B	0.0169(8)	0.0185(8)	0.0124(7)	−0.0007(6)	0.0048(6)	−0.0004(6)
C7B	0.0167(8)	0.0171(8)	0.0206(8)	−0.0020(7)	0.0079(6)	−0.0010(6)
C8B	0.0130(8)	0.0158(8)	0.0212(8)	0.0017(7)	0.0027(6)	−0.0005(6)
C9B	0.0165(8)	0.0179(8)	0.0141(7)	0.0010(6)	0.0022(6)	−0.0026(6)
C10B	0.0157(8)	0.0167(8)	0.0149(8)	−0.0009(6)	0.0054(6)	−0.0010(6)
Cl1X	0.033(6)	0.031(6)	0.025(5)	0.005(4)	0.000(4)	0.008(5)

Compound **12a** (CCDC Deposition Number: 2521691) Data for **12a** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK_α radiation ($\lambda = 0.71073 \text{ \AA}$). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were

refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.



Figure C.9. X-ray crystal structure of **12a**.

Crystallographic Data for **12a**: C₁₀H₆Cl₂FN₃ (*M*=258.08, g/mol), monoclinic, *a* = 3.6909(3) Å, *b* = 22.815(2) Å, *c* = 12.0168(11) Å, α = 90°, β = 93.371(4)°, γ = 90°, *V* = 1010.16(15) Å³, *Z* = 4, *T* = 100 K, $\mu(\text{MoK}\alpha)$ = 0.628 mm⁻¹, ρ_{calc} = 1.697 g/cm³, 14258 reflections measured, 1790 unique (*R*_{int} = 0.0937, *R*_{sigma} = 0.0512), *R*₁ = 0.0515 (*I* > 2σ(*I*)) and *wR*₂ was 0.1120 (all data).

Table C.10. Crystal data and structure refinement for **12a**.

CCDC number	2521691
Empirical formula	C ₁₀ H ₆ Cl ₂ FN ₃
Formula weight	258.08
Temperature [K]	100
Crystal system	monoclinic
Space group (number)	<i>P</i> 2 ₁ / <i>c</i> (14)
<i>a</i> [Å]	3.6909(3)

b [Å]	22.815(2)
c [Å]	12.0168(11)
α [°]	90
β [°]	93.371(4)
γ [°]	90
Volume [Å ³]	1010.16(15)
Z	4
ρ_{calc} [gcm ⁻³]	1.697
μ [mm ⁻¹]	0.628
$F(000)$	520
Crystal size [mm ³]	0.01×0.04×0.21
Crystal colour	colourless
Crystal shape	needle
Radiation	MoK α (λ =0.71073 Å)
2 θ range [°]	4.93 to 50.10 (0.84 Å)
Index ranges	$-4 \leq h \leq 4$ $-27 \leq k \leq 27$ $-14 \leq l \leq 14$
Reflections collected	14258
Independent reflections	1790 $R_{\text{int}} = 0.0937$ $R_{\text{sigma}} = 0.0512$
Completeness to $\theta = 25.052^\circ$	99.6 %
Data / Restraints / Parameters	1790 / 1 / 148
Goodness-of-fit on F^2	1.070
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0515$ $wR_2 = 0.1025$
Final R indexes [all data]	$R_1 = 0.0766$ $wR_2 = 0.1120$
Largest peak/hole [eÅ ⁻³]	0.79/−0.40

Table C.11. Atomic coordinates and U_{eq} [Å²] for **12a**.

Atom	x	y	z	U_{eq}
Cl1	0.3887(3)	0.57807(4)	0.54236(7)	0.0198(3)
Cl2	0.6312(3)	0.25157(4)	0.94413(8)	0.0211(3)
F1	0.3928(7)	0.23745(10)	0.70835(18)	0.0264(6)
N1	0.2739(8)	0.51818(13)	0.7207(2)	0.0133(7)
N2	0.0400(8)	0.55768(13)	0.8875(2)	0.0138(7)
N3	0.1774(9)	0.45994(14)	0.8745(2)	0.0148(7)
C1	0.1640(10)	0.51319(17)	0.8245(3)	0.0141(8)
C2	0.2474(10)	0.57118(17)	0.6774(3)	0.0146(8)
C3	0.1255(11)	0.61991(17)	0.7307(3)	0.0177(9)
H3A	0.109567	0.657353	0.696172	0.021
C4	0.0282(10)	0.61042(16)	0.8378(3)	0.0160(8)

H4	−0.052039	0.642986	0.879062	0.019
C5	0.2466(10)	0.40507(16)	0.8260(3)	0.0135(8)
C6	0.3993(10)	0.36177(17)	0.8960(3)	0.0152(8)
H6	0.469801	0.370847	0.971384	0.018
C7	0.4483(10)	0.30575(17)	0.8561(3)	0.0152(8)
C8	0.3495(11)	0.29285(17)	0.7466(3)	0.0174(9)
C9	0.2030(10)	0.33514(17)	0.6761(3)	0.0178(9)
H9	0.137996	0.325793	0.600551	0.021
H3	0.117(11)	0.4598(18)	0.9430(14)	0.021
C10	0.1499(10)	0.39157(17)	0.7153(3)	0.0145(8)
H10	0.047845	0.420832	0.666747	0.017

Table C.12. Anisotropic displacement parameters ($\text{\AA}^2$) for 12a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C11	0.0248(5)	0.0255(5)	0.0095(4)	0.0035(4)	0.0033(4)	−0.0016(4)
C12	0.0276(5)	0.0199(5)	0.0162(5)	0.0053(4)	0.0043(4)	0.0070(4)
F1	0.0458(15)	0.0156(12)	0.0182(12)	−0.0039(10)	0.0050(11)	0.0016(11)
N1	0.0174(17)	0.0152(17)	0.0071(15)	0.0022(13)	−0.0001(13)	−0.0021(13)
N2	0.0156(17)	0.0155(16)	0.0099(15)	−0.0016(13)	−0.0017(13)	0.0002(14)
N3	0.0247(19)	0.0148(17)	0.0051(14)	−0.0010(13)	0.0041(13)	0.0016(14)
C1	0.0123(19)	0.020(2)	0.0099(17)	0.0026(16)	−0.0037(15)	−0.0031(16)
C2	0.0131(19)	0.025(2)	0.0051(17)	0.0042(16)	−0.0027(15)	−0.0056(17)
C3	0.024(2)	0.015(2)	0.0139(19)	0.0051(16)	−0.0003(17)	−0.0042(17)
C4	0.018(2)	0.013(2)	0.017(2)	−0.0038(16)	−0.0043(16)	−0.0002(16)
C5	0.0140(19)	0.015(2)	0.0119(18)	−0.0003(15)	0.0045(16)	−0.0032(16)
C6	0.016(2)	0.024(2)	0.0060(17)	−0.0002(16)	0.0020(15)	0.0009(17)
C7	0.0134(19)	0.020(2)	0.0124(18)	0.0037(16)	0.0028(15)	0.0024(16)
C8	0.023(2)	0.015(2)	0.0152(19)	−0.0031(16)	0.0073(17)	−0.0007(17)
C9	0.021(2)	0.022(2)	0.0100(18)	−0.0003(17)	0.0004(16)	−0.0037(18)
C10	0.0124(19)	0.021(2)	0.0106(18)	0.0030(16)	0.0020(15)	0.0003(16)

Compound 16a (CCDC Deposition Number: 2521690) Data for **16a** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK_α radiation ($\lambda = 0.71073 \text{ \AA}$). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon

atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.

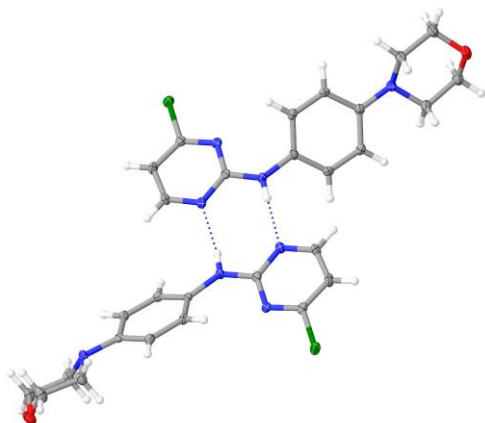


Figure C.10. X-ray crystal structure of **16a**.

Crystallographic Data for 16a: C₁₄H₁₅ClN₄O (*M*=290.75, g/mol), triclinic, *a* = 10.3272(6) Å, *b* = 10.4095(6) Å, *c* = 14.8291(8) Å, α = 78.802(3)°, β = 72.476(3)°, γ = 60.607(3)°, *V* = 1322.41(14) Å³, *Z* = 4, *T* = 100 K, $\mu(\text{MoK}\alpha)$ = 0.290 mm⁻¹, ρ_{calc} = 1.460 g/cm³, 43170 reflections measured, 4690 unique (*R*_{int} = 0.0741, *R*_{sigma} = 0.0374), *R*₁ = 0.0389 (*I* > 2σ(*I*)) and *wR*₂ was 0.0983 (all data).

Table C.13. Crystal data and structure refinement for **16a**.

CCDC number	2521690
Empirical formula	C ₁₄ H ₁₅ ClN ₄ O
Formula weight	290.75
Temperature [K]	100
Crystal system	triclinic
Space group (number)	<i>P</i> $\bar{1}$ (2)
<i>a</i> [Å]	10.3272(6)
<i>b</i> [Å]	10.4095(6)
<i>c</i> [Å]	14.8291(8)

α [°]	78.802(3)
β [°]	72.476(3)
γ [°]	60.607(3)
Volume [Å ³]	1322.41(14)
Z	4
ρ_{calc} [gcm ⁻³]	1.460
μ [mm ⁻¹]	0.290
$F(000)$	608
Crystal size [mm ³]	0.02×0.03×0.14
Crystal colour	colourless
Crystal shape	rod
Radiation	MoK α (λ =0.71073 Å)
2 θ range [°]	4.75 to 50.15 (0.84 Å)
Index ranges	−12 ≤ h ≤ 12 −12 ≤ k ≤ 12 −17 ≤ l ≤ 17
Reflections collected	43170
Independent reflections	4690 $R_{\text{int}} = 0.0741$ $R_{\text{sigma}} = 0.0374$
Completeness to $\theta = 25.075^\circ$	99.3 %
Data / Restraints / Parameters	4690 / 0 / 369
Goodness-of-fit on F^2	1.064
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0389$ $wR_2 = 0.0868$
Final R indexes [all data]	$R_1 = 0.0627$ $wR_2 = 0.0983$
Largest peak/hole [eÅ ⁻³]	0.21/−0.32

Table C.14. Atomic coordinates and U_{eq} [Å²] for **16a**.

Atom	x	y	z	U_{eq}
Cl1A	0.03609(6)	0.51545(6)	0.61629(4)	0.02081(15)
Cl1B	0.95302(7)	0.98003(6)	0.39763(4)	0.02213(15)
O1A	0.23260(19)	0.75520(18)	1.21442(11)	0.0234(4)
O1B	0.81639(19)	0.78724(19)	−0.21698(11)	0.0266(4)
N1A	0.2233(2)	0.61371(19)	0.61899(12)	0.0164(4)
N2A	0.3788(2)	0.6788(2)	0.48039(12)	0.0171(4)
N2B	0.6130(2)	0.8127(2)	0.52858(12)	0.0183(4)
N1B	0.7699(2)	0.87921(19)	0.39298(12)	0.0158(4)
N3B	0.6062(2)	0.7946(2)	0.37883(12)	0.0161(4)
N3A	0.3858(2)	0.7026(2)	0.62927(13)	0.0171(4)
N4B	0.7531(2)	0.7274(2)	−0.01519(12)	0.0172(4)
N4A	0.2120(2)	0.7409(2)	1.02807(12)	0.0174(4)

C1B	0.6671(2)	0.8292(2)	0.43434(15)	0.0156(5)
C1A	0.3270(3)	0.6629(2)	0.57537(15)	0.0155(5)
C8B	0.7188(2)	0.7527(2)	0.08236(15)	0.0151(5)
C5B	0.6477(2)	0.7873(2)	0.27904(14)	0.0143(5)
C5A	0.3414(2)	0.7097(2)	0.72918(15)	0.0155(5)
C3A	0.2147(3)	0.5876(3)	0.46436(16)	0.0209(5)
H3AA	0.175025	0.560049	0.425540	0.025
C6B	0.5391(3)	0.7978(2)	0.23677(15)	0.0160(5)
H6B	0.439233	0.818417	0.274285	0.019
C4A	0.3208(3)	0.6402(2)	0.42736(15)	0.0201(5)
H4A	0.354303	0.649349	0.360444	0.024
C9A	0.2853(3)	0.6062(2)	0.88784(15)	0.0173(5)
H9A	0.274606	0.527552	0.928443	0.021
C8A	0.2587(2)	0.7320(2)	0.92787(15)	0.0158(5)
C10B	0.7909(3)	0.7626(2)	0.22154(15)	0.0172(5)
H10B	0.866369	0.756356	0.248706	0.021
C7A	0.2768(3)	0.8443(2)	0.86553(15)	0.0184(5)
H7A	0.261956	0.929613	0.890099	0.022
C9B	0.8256(3)	0.7468(2)	0.12473(15)	0.0169(5)
H9B	0.923675	0.731738	0.086819	0.020
C7B	0.5741(3)	0.7788(2)	0.14114(15)	0.0165(5)
H7B	0.498740	0.783405	0.114592	0.020
C10A	0.3264(3)	0.5951(2)	0.79079(15)	0.0174(5)
H10A	0.344700	0.508720	0.765759	0.021
C2A	0.1707(3)	0.5783(2)	0.56203(15)	0.0166(5)
C2B	0.8233(2)	0.9114(2)	0.45153(16)	0.0174(5)
C12A	0.1278(3)	0.8795(3)	1.16905(16)	0.0222(5)
H12A	0.113521	0.971196	1.190123	0.027
H12B	0.027260	0.879654	1.187982	0.027
C3B	0.7812(3)	0.8958(3)	0.54889(16)	0.0221(5)
H3BA	0.822989	0.918024	0.589088	0.027
C14A	0.3125(3)	0.6115(3)	1.07944(15)	0.0208(5)
H14A	0.416312	0.603885	1.061183	0.025
H14B	0.320656	0.520694	1.061198	0.025
C12B	0.6708(3)	0.8018(3)	−0.16523(16)	0.0236(5)
H12C	0.667715	0.708315	−0.165480	0.028
H12D	0.591496	0.880810	−0.196416	0.028
C4B	0.6732(3)	0.8451(3)	0.58337(16)	0.0217(5)
H4B	0.639959	0.832715	0.650078	0.026
C6A	0.3162(3)	0.8334(2)	0.76811(15)	0.0189(5)
H6A	0.326240	0.912202	0.727259	0.023
C11B	0.6353(3)	0.8385(3)	−0.06410(15)	0.0225(5)
H11C	0.628684	0.936565	−0.063686	0.027
H11D	0.534609	0.843919	−0.029556	0.027
C11A	0.1843(3)	0.8775(3)	1.06208(15)	0.0194(5)
H11A	0.107132	0.963364	1.032844	0.023

H11B	0.280347	0.886157	1.042627	0.023
C13A	0.2514(3)	0.6228(3)	1.18544(15)	0.0231(5)
H13A	0.151634	0.621368	1.204470	0.028
H13B	0.323277	0.536354	1.217726	0.028
C14B	0.9050(3)	0.7049(3)	−0.07110(15)	0.0225(5)
H14C	0.984132	0.621771	−0.041696	0.027
H14D	0.915466	0.794724	−0.071881	0.027
C13B	0.9295(3)	0.6714(3)	−0.17188(16)	0.0276(6)
H13C	1.032360	0.657784	−0.208834	0.033
H13D	0.925603	0.578050	−0.171036	0.033
H3A	0.448(3)	0.735(3)	0.6005(17)	0.019(7)
H3B	0.536(3)	0.768(3)	0.4130(19)	0.031(7)

Table C.15. Anisotropic displacement parameters ($\text{\AA}^2$) for 16a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1A	0.0259(3)	0.0243(3)	0.0193(3)	−0.0015(2)	−0.0040(2)	−0.0175(3)
Cl1B	0.0264(3)	0.0280(3)	0.0201(3)	−0.0048(2)	−0.0038(2)	−0.0183(3)
O1A	0.0302(9)	0.0265(9)	0.0163(8)	−0.0031(7)	−0.0078(7)	−0.0133(8)
O1B	0.0323(10)	0.0299(10)	0.0143(8)	0.0001(7)	−0.0038(7)	−0.0134(8)
N1A	0.0195(10)	0.0151(10)	0.0156(9)	−0.0019(8)	−0.0038(8)	−0.0087(8)
N2A	0.0219(10)	0.0162(10)	0.0146(9)	−0.0024(8)	−0.0041(8)	−0.0094(9)
N2B	0.0241(11)	0.0213(10)	0.0125(9)	−0.0015(8)	−0.0043(8)	−0.0126(9)
N1B	0.0202(10)	0.0142(9)	0.0153(9)	−0.0017(7)	−0.0038(8)	−0.0096(8)
N3B	0.0205(10)	0.0213(10)	0.0109(9)	−0.0016(8)	−0.0018(8)	−0.0138(9)
N3A	0.0221(11)	0.0225(11)	0.0130(9)	−0.0022(8)	−0.0021(8)	−0.0157(9)
N4B	0.0216(10)	0.0187(10)	0.0123(9)	−0.0028(8)	−0.0040(8)	−0.0094(9)
N4A	0.0216(10)	0.0165(10)	0.0145(9)	−0.0023(7)	−0.0040(8)	−0.0089(9)
C1B	0.0186(12)	0.0121(11)	0.0146(11)	−0.0030(9)	−0.0032(9)	−0.0058(10)
C1A	0.0193(12)	0.0108(11)	0.0148(11)	−0.0020(9)	−0.0027(9)	−0.0062(10)
C8B	0.0202(12)	0.0098(11)	0.0148(11)	−0.0015(9)	−0.0030(9)	−0.0068(10)
C5B	0.0206(12)	0.0104(11)	0.0128(10)	−0.0017(8)	−0.0037(9)	−0.0078(10)
C5A	0.0167(11)	0.0202(12)	0.0129(11)	−0.0011(9)	−0.0049(9)	−0.0102(10)
C3A	0.0281(13)	0.0247(13)	0.0176(12)	−0.0028(10)	−0.0066(10)	−0.0168(11)
C6B	0.0173(11)	0.0144(11)	0.0163(11)	−0.0016(9)	−0.0014(9)	−0.0086(10)
C4A	0.0279(13)	0.0232(13)	0.0116(11)	−0.0016(9)	−0.0050(9)	−0.0132(11)
C9A	0.0219(12)	0.0149(11)	0.0171(11)	0.0013(9)	−0.0044(9)	−0.0110(10)
C8A	0.0158(11)	0.0169(12)	0.0153(11)	−0.0023(9)	−0.0034(9)	−0.0077(10)
C10B	0.0218(12)	0.0193(12)	0.0154(11)	−0.0025(9)	−0.0049(9)	−0.0124(10)
C7A	0.0240(12)	0.0157(11)	0.0180(11)	−0.0028(9)	−0.0054(9)	−0.0103(10)
C9B	0.0187(12)	0.0169(11)	0.0168(11)	−0.0032(9)	−0.0014(9)	−0.0103(10)
C7B	0.0212(12)	0.0160(11)	0.0157(11)	−0.0005(9)	−0.0067(9)	−0.0101(10)
C10A	0.0218(12)	0.0155(11)	0.0174(11)	−0.0039(9)	−0.0047(9)	−0.0094(10)
C2A	0.0194(12)	0.0142(11)	0.0167(11)	−0.0010(9)	−0.0036(9)	−0.0086(10)
C2B	0.0181(12)	0.0134(11)	0.0212(12)	−0.0044(9)	−0.0042(9)	−0.0067(10)

C12A	0.0243(13)	0.0241(13)	0.0188(12)	-0.0041(10)	-0.0040(10)	-0.0112(11)
C3B	0.0278(13)	0.0271(13)	0.0177(12)	-0.0043(10)	-0.0073(10)	-0.0153(11)
C14A	0.0243(12)	0.0200(12)	0.0157(11)	-0.0016(9)	-0.0053(9)	-0.0078(10)
C12B	0.0274(13)	0.0274(13)	0.0160(12)	-0.0042(10)	-0.0042(10)	-0.0123(11)
C4B	0.0290(13)	0.0275(13)	0.0113(11)	-0.0024(9)	-0.0035(9)	-0.0154(11)
C6A	0.0265(13)	0.0182(12)	0.0166(11)	0.0008(9)	-0.0050(10)	-0.0145(11)
C11B	0.0236(13)	0.0266(13)	0.0153(11)	-0.0025(10)	-0.0051(10)	-0.0094(11)
C11A	0.0213(12)	0.0215(12)	0.0181(12)	-0.0043(9)	-0.0046(9)	-0.0108(10)
C13A	0.0293(14)	0.0243(13)	0.0156(12)	0.0000(10)	-0.0070(10)	-0.0118(11)
C14B	0.0241(13)	0.0258(13)	0.0151(11)	-0.0027(10)	-0.0019(10)	-0.0106(11)
C13B	0.0254(13)	0.0349(15)	0.0168(12)	-0.0062(10)	-0.0014(10)	-0.0100(12)

Compound **25a** (CCDC Deposition Number: 2521689) Data for **25a** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK α radiation ($\lambda = 0.71073$ Å). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.

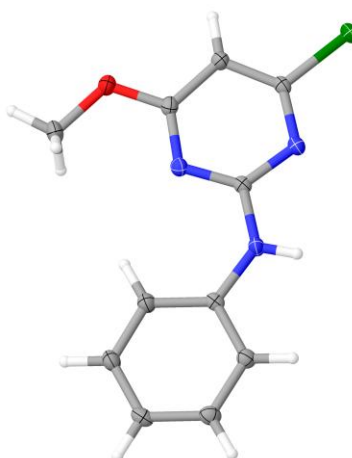


Figure C.11. X-ray crystal structure of **25a**.

Crystallographic Data for 25a: $C_{11}H_{10}ClN_3O$ ($M=235.67$, g/mol), monoclinic, $a = 9.9343(4)$ Å, $b = 5.1815(2)$ Å, $c = 20.7246(7)$ Å, $\alpha = 90^\circ$, $\beta = 91.0790(10)^\circ$, $\gamma = 90^\circ$, $V = 1066.60(7)$ Å³, $Z = 4$, $T = 100$ K, $\mu(\text{MoK}\alpha) = 0.338\text{mm}^{-1}$, $\rho_{\text{calc}} = 1.468\text{g/cm}^3$, 36987 reflections measured, 3253 unique ($R_{\text{int}} = 0.0266$, $R_{\text{sigma}} = 0.0152$), $R_1 = 0.0358$ ($I > 2\sigma(I)$) and wR_2 was 0.0878 (all data).

Table C.16. Crystal data and structure refinement for **25a**.

CCDC number	2521689
Empirical formula	$C_{11}H_{10}ClN_3O$
Formula weight	235.67
Temperature [K]	100
Crystal system	monoclinic
Space group (number)	$P2_1/c$ (14)
a [Å]	9.9343(4)
b [Å]	5.1815(2)
c [Å]	20.7246(7)
α [°]	90
β [°]	91.0790(10)
γ [°]	90
Volume [Å ³]	1066.60(7)
Z	4
ρ_{calc} [gcm ⁻³]	1.468
μ [mm ⁻¹]	0.338
$F(000)$	488
Crystal size [mm ³]	0.08×0.12×0.31
Crystal colour	colourless
Crystal shape	prism
Radiation	MoK α ($\lambda=0.71073$ Å)
2 θ range [°]	5.63 to 61.01 (0.70 Å)
Index ranges	$-14 \leq h \leq 14$ $-7 \leq k \leq 7$ $-27 \leq l \leq 29$
Reflections collected	36987
Independent reflections	3253 $R_{\text{int}} = 0.0266$ $R_{\text{sigma}} = 0.0152$
Completeness to $\theta = 25.242^\circ$	99.9 %
Data / Restraints / Parameters	3253 / 0 / 150
Goodness-of-fit on F^2	1.097
Final R indexes $[I \geq 2\sigma(I)]$	$R_1 = 0.0358$ $wR_2 = 0.0831$

Final <i>R</i> indexes [all data]	$R_1 = 0.0426$ $wR_2 = 0.0878$
Largest peak/hole [$\text{e}\text{\AA}^{-3}$]	0.41/−0.27

Table C.17. Atomic coordinates and U_{eq} [$\text{\AA}^2$] for **25a**.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C11	0.31584(3)	0.10922(6)	0.33652(2)	0.01969(9)
O1	0.67293(10)	0.2883(2)	0.50456(4)	0.0234(2)
N1	0.49804(10)	0.4696(2)	0.33082(5)	0.0151(2)
N2	0.66464(10)	0.5496(2)	0.41439(5)	0.0154(2)
N3	0.65024(10)	0.7964(2)	0.32026(5)	0.0156(2)
H3	0.6057(18)	0.820(4)	0.2859(9)	0.026(4)
C1	0.60456(11)	0.5991(2)	0.35706(6)	0.0136(2)
C2	0.45369(11)	0.2793(2)	0.36729(6)	0.0149(2)
C3	0.50609(13)	0.2041(3)	0.42611(6)	0.0186(2)
H3A	0.471087	0.064337	0.450267	0.022
C4	0.61577(12)	0.3527(2)	0.44702(6)	0.0171(2)
C5	0.78776(14)	0.4357(3)	0.52551(7)	0.0260(3)
H5A	0.860363	0.414460	0.494593	0.039
H5B	0.818347	0.375069	0.568108	0.039
H5C	0.763066	0.618499	0.528154	0.039
C6	0.76207(12)	0.9608(2)	0.32890(6)	0.0145(2)
C7	0.77850(12)	1.1488(2)	0.28119(6)	0.0168(2)
H7	0.716122	1.158211	0.245958	0.020
C8	0.88515(13)	1.3214(2)	0.28492(6)	0.0186(2)
H8	0.895143	1.448271	0.252269	0.022
C9	0.97742(12)	1.3100(2)	0.33606(6)	0.0186(2)
H9	1.050077	1.428948	0.338827	0.022
C10	0.96174(13)	1.1222(3)	0.38301(6)	0.0197(2)
H10	1.025121	1.112541	0.417825	0.024
C11	0.85525(13)	0.9474(3)	0.38031(6)	0.0185(2)
H11	0.845971	0.820389	0.412994	0.022

Table C.18. Anisotropic displacement parameters ($\text{\AA}^2$) for 25a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C11	0.01771(14)	0.02181(15)	0.01939(15)	−0.00006(11)	−0.00354(10)	−0.00618(11)
O1	0.0266(5)	0.0269(5)	0.0165(4)	0.0048(4)	−0.0054(4)	−0.0095(4)
N1	0.0131(4)	0.0160(5)	0.0161(5)	−0.0011(4)	−0.0011(3)	−0.0009(4)
N2	0.0152(4)	0.0167(5)	0.0143(4)	−0.0013(4)	−0.0007(4)	−0.0013(4)
N3	0.0151(4)	0.0160(5)	0.0155(5)	0.0016(4)	−0.0036(4)	−0.0020(4)
C1	0.0126(5)	0.0129(5)	0.0154(5)	−0.0017(4)	0.0011(4)	0.0005(4)
C2	0.0127(5)	0.0155(5)	0.0164(5)	−0.0030(4)	−0.0004(4)	−0.0014(4)
C3	0.0196(6)	0.0204(6)	0.0156(5)	0.0017(5)	−0.0016(4)	−0.0055(5)

C4	0.0170(5)	0.0201(6)	0.0142(5)	−0.0005(4)	−0.0012(4)	−0.0012(4)
C5	0.0234(6)	0.0342(8)	0.0201(6)	0.0002(5)	−0.0074(5)	−0.0093(6)
C6	0.0131(5)	0.0132(5)	0.0173(5)	−0.0018(4)	0.0009(4)	0.0004(4)
C7	0.0158(5)	0.0157(5)	0.0188(5)	0.0010(4)	−0.0009(4)	0.0004(4)
C8	0.0173(5)	0.0155(5)	0.0230(6)	0.0028(5)	0.0007(4)	−0.0003(4)
C9	0.0143(5)	0.0165(5)	0.0251(6)	−0.0009(5)	0.0009(4)	−0.0017(4)
C10	0.0164(5)	0.0212(6)	0.0212(6)	−0.0002(5)	−0.0038(4)	−0.0026(5)
C11	0.0174(5)	0.0193(6)	0.0188(6)	0.0021(5)	−0.0027(4)	−0.0024(5)

Compound 38 (CCDC Deposition Number: 2523200) Data for **38** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK α radiation ($\lambda = 0.71073$ Å). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.

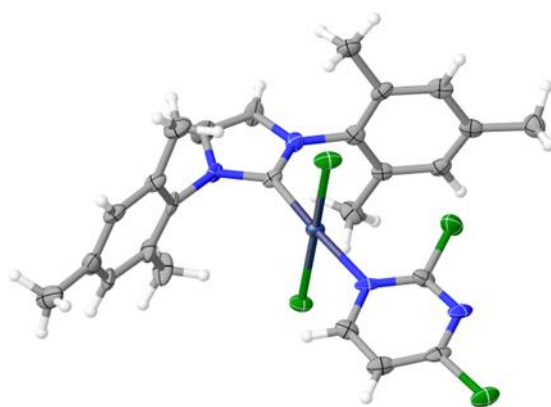


Figure C.12. X-ray crystal structure of **38**.

Crystallographic Data for 38: C₂₅H₂₆Cl₄N₄Pd ($M=630.70$, g/mol), monoclinic, $a = 28.983(3)$ Å, $b = 8.8135(10)$ Å, $c = 20.792(2)$ Å, $\alpha = 90^\circ$, $\beta = 97.375(3)^\circ$, $\gamma = 90^\circ$, $V = 5267.1(10)$ Å³, $Z = 8$, $T = 100.00$ K, $\mu(\text{MoK}\alpha) = 1.132\text{mm}^{-1}$, $\rho_{\text{calc}} = 1.591\text{g/cm}^3$, 9322 reflections measured, 9322 unique ($R_{\text{int}} = ?$, $R_{\text{sigma}} = 0.0314$), $R_1 = 0.0785$ ($I > 2\sigma(I)$) and wR_2 was 0.2096 (all data).

Table C.19. Crystal data and structure refinement for **38**.

CCDC number	2523200
Empirical formula	C ₂₅ H ₂₆ Cl ₄ N ₄ Pd
Formula weight	630.70
Temperature [K]	100.00
Crystal system	monoclinic
Space group (number)	$P2/c$ (13)
a [Å]	28.983(3)
b [Å]	8.8135(10)
c [Å]	20.792(2)
α [°]	90
β [°]	97.375(3)
γ [°]	90
Volume [Å ³]	5267.1(10)
Z	8
ρ_{calc} [gcm ⁻³]	1.591
μ [mm ⁻¹]	1.132
$F(000)$	2544
Crystal size [mm ³]	0.12×0.19×0.21
Crystal colour	yellow
Crystal shape	prism
Radiation	MoK α ($\lambda=0.71073$ Å)
2 θ range [°]	4.83 to 50.10 (0.84 Å)
Index ranges	$-34 \leq h \leq 34$ $0 \leq k \leq 10$ $0 \leq l \leq 24$
Reflections collected	9322
Independent reflections	9322 $R_{\text{int}} = ?$ $R_{\text{sigma}} = 0.0314$
Completeness to $\theta = 25.050^\circ$	99.7 %
Data / Restraints / Parameters	9322 / 590 / 849
Goodness-of-fit on F^2	1.160
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0785$ $wR_2 = 0.2022$
Final R indexes [all data]	$R_1 = 0.0913$ $wR_2 = 0.2096$

Largest peak/hole [$\text{e}\text{\AA}^{-3}$]	2.19/−1.95
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Table C.20. Atomic coordinates and U_{eq} [$\text{\AA}^2$] for **38**.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Pd1	0.37135(3)	0.68742(9)	0.34150(4)	0.0176(2)
Cl1	0.37311(11)	0.5179(3)	0.42619(12)	0.0293(6)
Cl2	0.36449(9)	0.8447(3)	0.25279(12)	0.0253(6)
Cl3	0.45937(10)	0.4747(4)	0.31810(15)	0.0372(7)
Cl4	0.38008(12)	0.1274(3)	0.13716(13)	0.0341(7)
N1	0.3425(3)	0.9536(9)	0.4193(4)	0.0162(17)
N2	0.4170(3)	0.9151(10)	0.4312(4)	0.0215(19)
N3	0.3716(3)	0.5048(10)	0.2756(4)	0.023(2)
N4	0.4137(3)	0.3099(11)	0.2294(4)	0.027(2)
C1	0.3768(3)	0.8590(12)	0.4008(5)	0.019(2)
C2	0.3637(4)	1.0680(13)	0.4607(6)	0.031(3)
H2	0.348312	1.148075	0.479888	0.038
C3	0.4101(4)	1.0416(12)	0.4677(6)	0.025(2)
H3	0.433427	1.099418	0.492921	0.030
C4	0.4623(3)	0.8517(12)	0.4244(5)	0.0216(16)
C5	0.4844(4)	0.8943(12)	0.3721(5)	0.021(2)
C6	0.5256(3)	0.8208(13)	0.3638(5)	0.024(2)
H6	0.541633	0.850054	0.328709	0.028
C7	0.5443(4)	0.7044(13)	0.4056(5)	0.025(2)
C8	0.5224(4)	0.6747(13)	0.4616(5)	0.025(2)
H8	0.536030	0.603874	0.492889	0.030
C9	0.4815(4)	0.7469(12)	0.4716(5)	0.023(2)
C10	0.4669(4)	1.0222(13)	0.3270(6)	0.031(3)
H10A	0.492964	1.066204	0.307744	0.046
H10B	0.452578	1.100388	0.351480	0.046
H10C	0.443729	0.982767	0.292549	0.046
C11	0.5872(4)	0.6194(15)	0.3942(6)	0.033(3)
H11A	0.578431	0.520221	0.375169	0.049
H11B	0.607115	0.605230	0.435559	0.049
H11C	0.604130	0.677211	0.364482	0.049
C12	0.4597(4)	0.7230(13)	0.5324(5)	0.028(3)
H12A	0.456310	0.820944	0.553573	0.043
H12B	0.479571	0.656246	0.561753	0.043
H12C	0.429034	0.676192	0.521534	0.043
C13	0.2941(3)	0.9455(11)	0.3981(5)	0.021(2)
C14	0.2741(4)	1.0335(13)	0.3468(5)	0.027(2)
C15	0.2273(4)	1.0196(15)	0.3282(6)	0.030(3)
H15	0.213191	1.081188	0.293631	0.036
C16	0.1996(4)	0.9197(15)	0.3574(6)	0.034(3)
C17	0.2204(4)	0.8388(13)	0.4117(5)	0.025(2)
H17	0.201491	0.774864	0.434210	0.030

C18	0.2681(4)	0.8492(13)	0.4338(5)	0.024(2)
C19	0.3020(4)	1.1493(15)	0.3142(7)	0.041(3)
H19A	0.313591	1.227761	0.345587	0.061
H19B	0.282108	1.196158	0.277900	0.061
H19C	0.328361	1.098975	0.297959	0.061
C20	0.1488(4)	0.9015(19)	0.3338(6)	0.046(4)
H20A	0.141731	0.793706	0.326262	0.069
H20B	0.141119	0.957736	0.293154	0.069
H20C	0.130446	0.941017	0.366453	0.069
C21	0.2898(4)	0.7684(15)	0.4930(6)	0.036(3)
H21A	0.312250	0.693873	0.480923	0.054
H21B	0.265558	0.716256	0.513407	0.054
H21C	0.305738	0.841768	0.523496	0.054
C22	0.4101(4)	0.4248(12)	0.2701(5)	0.023(2)
C23	0.3758(4)	0.2779(12)	0.1902(5)	0.026(3)
C24	0.3340(4)	0.3549(13)	0.1895(5)	0.029(3)
H24	0.307308	0.329278	0.160101	0.035
C25	0.3336(4)	0.4695(12)	0.2334(5)	0.025(2)
H25	0.305838	0.526162	0.234425	0.030
Pd1B	0.8553(3)	0.1962(11)	0.3771(6)	0.0171(11)
Cl1B	0.7963(4)	0.3661(17)	0.3885(11)	0.035(3)
Cl2B	0.9091(8)	0.016(2)	0.3555(18)	0.026(4)
Cl3B	0.8636(5)	−0.060(2)	0.4934(9)	0.014(3)
Cl4B	0.7073(6)	−0.3326(19)	0.4234(7)	0.046(3)
N1B	0.9366(7)	0.396(2)	0.4110(7)	0.028(3)
N2B	0.9004(8)	0.453(2)	0.3164(8)	0.031(3)
N3B	0.8073(6)	0.027(2)	0.3917(12)	0.0183(17)
N4B	0.7838(9)	−0.182(4)	0.4527(14)	0.022(3)
C1B	0.8992(7)	0.354(2)	0.3676(9)	0.020(4)
C2B	0.9625(11)	0.511(4)	0.3889(12)	0.064(9)
H2B	0.990159	0.554435	0.410620	0.077
C3B	0.9402(10)	0.549(3)	0.3291(11)	0.052(7)
H3B	0.949436	0.625874	0.301214	0.063
C7B	0.8020(5)	0.4395(18)	0.1511(6)	0.023(3)
C6B	0.7969(5)	0.5433(15)	0.2000(7)	0.023(3)
H6B	0.770497	0.608160	0.196445	0.028
C5B	0.8303(6)	0.5522(15)	0.2541(6)	0.026(4)
C4B	0.8689(5)	0.4573(17)	0.2593(6)	0.025(3)
C9B	0.8741(5)	0.3535(18)	0.2104(7)	0.025(3)
C8B	0.8406(6)	0.3446(18)	0.1563(7)	0.021(3)
H8B	0.844173	0.273700	0.122882	0.026
C10B	0.8276(12)	0.677(3)	0.3068(11)	0.047(7)
H10D	0.832579	0.777054	0.288239	0.071
H10E	0.851612	0.658367	0.343615	0.071
H10F	0.796848	0.674313	0.321690	0.071
C11B	0.7650(8)	0.423(3)	0.0927(11)	0.027(4)

H11D	0.734362	0.446290	0.105649	0.041
H11E	0.765075	0.319229	0.076207	0.041
H11F	0.771529	0.494125	0.058697	0.041
C12B	0.9176(9)	0.256(4)	0.2150(17)	0.037(7)
H12D	0.944230	0.319042	0.207594	0.055
H12E	0.913219	0.175786	0.182073	0.055
H12F	0.923326	0.209947	0.258174	0.055
C18B	0.9829(5)	0.2265(18)	0.4856(6)	0.0216(16)
C13B	0.9479(6)	0.3348(18)	0.4746(6)	0.026(3)
C14B	0.9220(6)	0.3742(19)	0.5239(7)	0.032(5)
C15B	0.9312(6)	0.305(2)	0.5844(6)	0.025(4)
H15B	0.913528	0.332299	0.618092	0.030
C16B	0.9662(6)	0.197(2)	0.5954(6)	0.022(4)
C17B	0.9920(5)	0.1576(18)	0.5461(7)	0.023(4)
H17B	1.015925	0.083629	0.553654	0.028
C19B	0.8866(11)	0.495(3)	0.5123(12)	0.042(7)
H19D	0.899905	0.582280	0.492299	0.063
H19E	0.876539	0.525088	0.553640	0.063
H19F	0.859825	0.456323	0.483264	0.063
C20B	0.9749(10)	0.127(3)	0.6636(11)	0.026(5)
H20D	0.964888	0.197630	0.695302	0.040
H20E	1.008139	0.105037	0.674602	0.040
H20F	0.957182	0.031927	0.664307	0.040
C21B	1.0156(9)	0.182(4)	0.4350(11)	0.034(5)
H21D	0.997034	0.141848	0.395999	0.051
H21E	1.037579	0.104127	0.453522	0.051
H21F	1.032829	0.271584	0.423647	0.051
C22B	0.8119(14)	−0.073(6)	0.441(2)	0.019(3)
C23B	0.7446(8)	−0.190(3)	0.4092(12)	0.028(6)
C24B	0.7351(8)	−0.094(3)	0.3586(12)	0.028(5)
H24B	0.706887	−0.101447	0.330027	0.033
C25B	0.7683(8)	0.015(4)	0.3499(14)	0.031(7)
H25B	0.763267	0.081993	0.313913	0.038
Pd1C	0.8447(3)	0.1957(13)	0.3718(7)	0.0174(14)
Cl1C	0.7828(5)	0.3515(19)	0.3853(12)	0.028(3)
Cl2C	0.9021(9)	0.031(2)	0.348(2)	0.021(4)
Cl3C	0.8590(7)	−0.031(3)	0.5019(12)	0.021(4)
Cl4C	0.7098(6)	−0.3536(18)	0.4448(8)	0.034(3)
N1C	0.9177(7)	0.429(2)	0.4020(8)	0.023(3)
N2C	0.8802(8)	0.466(2)	0.3067(9)	0.022(3)
N3C	0.8029(7)	0.015(3)	0.3947(14)	0.0183(17)
N4C	0.7856(10)	−0.188(4)	0.4652(17)	0.022(3)
C1C	0.8834(8)	0.367(2)	0.3584(10)	0.019(4)
C2C	0.9380(10)	0.556(3)	0.3804(11)	0.033(5)
H2C	0.963345	0.612306	0.402161	0.039
C3C	0.9138(9)	0.586(3)	0.3205(11)	0.028(4)

H3C	0.918335	0.669362	0.293298	0.034
C7C	0.7866(6)	0.416(2)	0.1369(8)	0.023(3)
C6C	0.7771(6)	0.520(2)	0.1837(9)	0.028(5)
H6C	0.749088	0.577272	0.177544	0.034
C5C	0.8085(7)	0.540(2)	0.2394(8)	0.027(4)
C4C	0.8494(6)	0.456(2)	0.2484(7)	0.024(3)
C9C	0.8589(6)	0.352(2)	0.2016(9)	0.025(3)
C8C	0.8276(7)	0.332(2)	0.1459(9)	0.037(7)
H8C	0.834093	0.261022	0.113895	0.045
C10C	0.7997(11)	0.667(3)	0.2891(12)	0.033(5)
H10G	0.815275	0.761026	0.278369	0.049
H10H	0.812151	0.634962	0.333037	0.049
H10I	0.766197	0.685887	0.287099	0.049
C11C	0.7509(10)	0.384(4)	0.0775(13)	0.035(6)
H11G	0.720114	0.418897	0.086105	0.052
H11H	0.749780	0.274912	0.068670	0.052
H11I	0.759817	0.438288	0.039878	0.052
C12C	0.9069(11)	0.277(5)	0.2061(19)	0.038(9)
H12G	0.931054	0.353254	0.218637	0.057
H12H	0.911244	0.234174	0.163845	0.057
H12I	0.909050	0.196336	0.238671	0.057
C18C	0.9707(6)	0.273(2)	0.4750(7)	0.0216(16)
C13C	0.9325(6)	0.369(2)	0.4645(7)	0.024(3)
C14C	0.9078(6)	0.405(2)	0.5154(8)	0.026(5)
C15C	0.9213(8)	0.346(3)	0.5768(8)	0.032(6)
H15C	0.904394	0.370946	0.611601	0.038
C16C	0.9595(8)	0.250(3)	0.5874(7)	0.034(6)
C17C	0.9843(6)	0.213(2)	0.5365(8)	0.029(5)
H17C	1.010408	0.147438	0.543715	0.034
C19C	0.8682(11)	0.512(3)	0.5054(15)	0.030(5)
H19G	0.879615	0.613913	0.497887	0.045
H19H	0.852037	0.512274	0.544086	0.045
H19I	0.846557	0.479405	0.467787	0.045
C20C	0.9748(13)	0.179(5)	0.6540(12)	0.037(8)
H20G	0.981830	0.259470	0.686255	0.056
H20H	1.002619	0.116762	0.652040	0.056
H20I	0.949673	0.115040	0.666367	0.056
C21C	1.0033(9)	0.247(3)	0.4227(12)	0.023(4)
H21G	0.984722	0.220629	0.381495	0.035
H21H	1.024938	0.164069	0.436216	0.035
H21I	1.020921	0.340005	0.417289	0.035
C22C	0.8109(16)	−0.078(7)	0.447(3)	0.019(3)
C23C	0.7450(9)	−0.209(3)	0.4258(15)	0.021(5)
C24C	0.7313(8)	−0.121(3)	0.3738(14)	0.022(5)
H24C	0.701664	−0.133280	0.349026	0.026
C25C	0.7622(9)	−0.012(3)	0.3579(14)	0.018(5)

H25C	0.754414	0.045147	0.319425	0.021
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Table C.21. Anisotropic displacement parameters ($\text{\AA}^2$) for **38**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pd1	0.0213(4)	0.0149(4)	0.0160(4)	-0.0007(3)	-0.0003(3)	-0.0030(3)
C11	0.0479(17)	0.0196(13)	0.0192(13)	0.0042(11)	-0.0001(11)	-0.0084(12)
C12	0.0342(14)	0.0210(13)	0.0193(12)	0.0043(10)	-0.0020(10)	-0.0010(11)
C13	0.0349(16)	0.0391(17)	0.0341(16)	-0.0116(13)	-0.0092(12)	0.0097(13)
C14	0.0585(19)	0.0216(14)	0.0222(13)	-0.0048(11)	0.0048(13)	-0.0065(13)
N1	0.021(4)	0.006(4)	0.022(4)	-0.006(3)	0.006(3)	-0.003(3)
N2	0.018(4)	0.024(5)	0.023(5)	0.000(4)	0.006(4)	-0.003(4)
N3	0.042(6)	0.018(5)	0.009(4)	0.003(3)	-0.002(4)	-0.009(4)
N4	0.047(6)	0.021(5)	0.012(4)	0.003(4)	0.000(4)	0.004(4)
C1	0.020(5)	0.019(5)	0.018(5)	-0.003(4)	0.007(4)	-0.009(4)
C2	0.036(7)	0.021(6)	0.035(6)	-0.019(5)	0.001(5)	-0.013(5)
C3	0.022(5)	0.015(5)	0.040(6)	-0.014(5)	0.007(5)	-0.010(4)
C4	0.017(4)	0.021(4)	0.026(3)	-0.007(3)	0.002(3)	-0.011(3)
C5	0.024(5)	0.018(5)	0.020(5)	0.000(4)	0.001(4)	-0.008(4)
C6	0.020(5)	0.031(6)	0.020(5)	-0.009(5)	0.003(4)	-0.010(5)
C7	0.024(5)	0.025(6)	0.024(5)	-0.003(5)	-0.004(4)	-0.011(5)
C8	0.025(6)	0.022(6)	0.028(6)	0.000(5)	-0.002(4)	-0.017(5)
C9	0.025(6)	0.024(6)	0.020(5)	-0.008(4)	0.007(4)	-0.012(5)
C10	0.031(6)	0.025(6)	0.036(6)	0.009(5)	0.001(5)	-0.002(5)
C11	0.026(6)	0.038(7)	0.034(6)	0.003(6)	0.005(5)	0.000(5)
C12	0.034(6)	0.030(6)	0.021(6)	-0.005(5)	0.001(5)	-0.006(5)
C13	0.017(5)	0.016(5)	0.031(6)	-0.006(4)	0.014(4)	-0.001(4)
C14	0.022(6)	0.030(6)	0.031(6)	0.003(5)	0.015(5)	0.004(5)
C15	0.023(6)	0.043(7)	0.027(6)	0.001(5)	0.013(5)	0.013(5)
C16	0.023(6)	0.045(8)	0.033(6)	-0.016(6)	0.003(5)	0.001(5)
C17	0.030(6)	0.027(6)	0.020(5)	-0.004(5)	0.010(4)	-0.008(5)
C18	0.024(5)	0.027(6)	0.021(5)	-0.004(5)	0.010(4)	-0.011(5)
C19	0.036(7)	0.036(7)	0.057(8)	0.017(6)	0.034(6)	0.004(6)
C20	0.025(6)	0.081(11)	0.032(7)	-0.011(7)	0.009(5)	-0.012(7)
C21	0.041(7)	0.044(8)	0.025(6)	0.000(6)	0.013(5)	-0.011(6)
C22	0.029(6)	0.014(5)	0.025(6)	0.000(4)	-0.007(4)	-0.002(4)
C23	0.051(7)	0.016(6)	0.012(5)	0.004(4)	0.004(5)	-0.004(5)
C24	0.045(7)	0.019(6)	0.022(5)	-0.001(5)	0.001(5)	-0.011(5)
C25	0.027(6)	0.019(6)	0.027(6)	0.005(5)	-0.002(5)	-0.005(5)
Pd1B	0.014(3)	0.0183(13)	0.0196(18)	-0.0012(10)	0.004(2)	-0.0005(18)
Cl1B	0.026(7)	0.030(5)	0.051(5)	0.005(4)	0.019(6)	0.010(5)
Cl2B	0.020(6)	0.028(5)	0.030(9)	-0.007(5)	0.005(6)	0.007(3)
Cl3B	0.014(4)	0.013(6)	0.015(5)	-0.005(4)	0.000(4)	0.000(4)
Cl4B	0.040(5)	0.048(7)	0.052(8)	-0.012(5)	0.014(5)	-0.027(5)
N1B	0.028(5)	0.029(7)	0.025(4)	-0.001(4)	0.000(4)	-0.010(4)

N2B	0.035(6)	0.031(6)	0.025(4)	0.002(3)	0.001(4)	−0.013(5)
N3B	0.015(3)	0.022(3)	0.019(4)	−0.004(3)	0.005(3)	−0.001(2)
N4B	0.023(3)	0.025(4)	0.017(8)	−0.005(4)	0.003(3)	−0.007(3)
C1B	0.021(6)	0.022(6)	0.018(5)	−0.006(4)	0.007(4)	−0.003(5)
C2B	0.063(11)	0.075(14)	0.046(7)	0.026(9)	−0.021(8)	−0.050(11)
C3B	0.054(9)	0.057(11)	0.041(7)	0.018(7)	−0.013(7)	−0.036(9)
C7B	0.025(6)	0.021(5)	0.024(4)	0.004(4)	0.009(4)	−0.008(5)
C6B	0.030(6)	0.019(6)	0.023(5)	0.005(5)	0.009(4)	−0.006(5)
C5B	0.033(6)	0.023(6)	0.023(5)	0.002(5)	0.008(4)	−0.006(5)
C4B	0.029(6)	0.023(5)	0.023(4)	0.002(3)	0.006(4)	−0.009(4)
C9B	0.027(6)	0.026(4)	0.023(4)	0.002(3)	0.007(4)	−0.004(5)
C8B	0.025(6)	0.021(6)	0.020(5)	0.004(5)	0.009(4)	−0.007(5)
C10B	0.095(18)	0.027(8)	0.017(8)	0.004(7)	−0.005(8)	0.022(11)
C11B	0.032(8)	0.015(10)	0.032(7)	0.001(6)	0.000(6)	−0.005(7)
C12B	0.034(8)	0.047(13)	0.030(11)	0.001(9)	0.005(7)	0.008(9)
C18B	0.017(4)	0.021(4)	0.026(3)	−0.007(3)	0.002(3)	−0.011(3)
C13B	0.025(6)	0.030(7)	0.023(4)	−0.004(4)	0.002(3)	−0.003(5)
C14B	0.034(9)	0.037(10)	0.026(5)	−0.001(5)	0.006(6)	0.005(7)
C15B	0.028(9)	0.023(9)	0.023(5)	−0.007(5)	0.003(5)	−0.002(7)
C16B	0.022(7)	0.017(9)	0.027(4)	−0.006(5)	0.004(5)	−0.008(6)
C17B	0.023(8)	0.020(9)	0.027(4)	−0.006(4)	0.003(4)	−0.006(6)
C19B	0.053(14)	0.054(12)	0.019(10)	0.000(8)	0.006(9)	0.023(11)
C20B	0.018(10)	0.031(13)	0.031(5)	0.002(6)	0.005(6)	−0.006(8)
C21B	0.028(10)	0.046(17)	0.029(7)	−0.005(7)	0.008(8)	−0.002(8)
C22B	0.018(3)	0.020(3)	0.018(5)	−0.005(3)	0.004(3)	−0.002(3)
C23B	0.024(5)	0.041(9)	0.017(9)	−0.004(7)	0.002(5)	−0.012(6)
C24B	0.022(6)	0.045(10)	0.016(8)	−0.003(7)	0.002(6)	−0.011(6)
C25B	0.020(6)	0.046(12)	0.027(8)	0.005(8)	−0.001(6)	−0.010(7)
Pd1C	0.016(3)	0.0169(15)	0.020(2)	0.0002(13)	0.006(3)	−0.002(2)
C11C	0.020(7)	0.023(4)	0.046(5)	0.004(3)	0.017(6)	0.001(4)
C12C	0.019(7)	0.023(6)	0.023(7)	−0.002(4)	0.006(7)	0.002(4)
C13C	0.023(5)	0.018(8)	0.022(6)	−0.008(4)	0.007(4)	0.005(4)
C14C	0.022(4)	0.023(4)	0.059(10)	0.011(5)	0.009(5)	−0.013(3)
N1C	0.020(6)	0.025(6)	0.026(4)	−0.003(4)	0.010(4)	−0.003(4)
N2C	0.022(6)	0.023(5)	0.025(4)	0.000(3)	0.012(4)	−0.004(4)
N3C	0.015(3)	0.022(3)	0.019(4)	−0.004(3)	0.005(3)	−0.001(2)
N4C	0.023(3)	0.025(4)	0.017(8)	−0.005(4)	0.003(3)	−0.007(3)
C1C	0.017(7)	0.021(5)	0.022(5)	−0.003(4)	0.013(4)	0.000(5)
C2C	0.034(9)	0.032(7)	0.032(6)	0.000(5)	0.006(6)	−0.014(7)
C3C	0.028(8)	0.029(6)	0.030(6)	0.000(5)	0.011(5)	−0.010(6)
C7C	0.025(6)	0.021(5)	0.024(4)	0.004(4)	0.009(4)	−0.008(5)
C6C	0.032(7)	0.028(8)	0.026(5)	0.001(6)	0.007(5)	−0.001(6)
C5C	0.028(6)	0.033(8)	0.023(6)	0.001(6)	0.010(5)	−0.002(6)
C4C	0.025(6)	0.025(6)	0.024(4)	0.003(4)	0.011(4)	−0.007(4)
C9C	0.027(6)	0.026(4)	0.023(4)	0.002(3)	0.007(4)	−0.004(5)
C8C	0.035(8)	0.045(12)	0.030(6)	−0.009(7)	0.000(6)	0.008(9)
C10C	0.053(14)	0.031(9)	0.015(8)	0.007(7)	0.010(7)	0.008(9)
C11C	0.036(9)	0.033(15)	0.034(8)	−0.003(8)	−0.002(7)	0.000(9)
C12C	0.035(8)	0.056(19)	0.024(12)	−0.005(12)	0.005(7)	0.011(10)

C18C	0.017(4)	0.021(4)	0.026(3)	−0.007(3)	0.002(3)	−0.011(3)
C13C	0.023(5)	0.025(7)	0.026(4)	−0.005(4)	0.007(3)	−0.006(5)
C14C	0.024(8)	0.030(11)	0.025(5)	−0.007(5)	0.006(5)	−0.005(7)
C15C	0.030(10)	0.041(15)	0.026(5)	−0.004(7)	0.007(6)	0.001(9)
C16C	0.032(9)	0.046(17)	0.025(4)	−0.006(6)	0.003(6)	0.004(9)
C17C	0.030(9)	0.030(12)	0.026(4)	−0.006(5)	0.004(4)	−0.001(8)
C19C	0.029(10)	0.037(12)	0.025(10)	−0.008(8)	0.006(8)	0.001(8)
C20C	0.023(13)	0.06(2)	0.028(5)	0.001(9)	0.003(7)	−0.001(13)
C21C	0.018(10)	0.023(14)	0.031(7)	−0.002(7)	0.005(7)	−0.009(7)
C22C	0.018(3)	0.020(3)	0.018(5)	−0.005(3)	0.004(3)	−0.002(3)
C23C	0.023(5)	0.021(8)	0.018(9)	−0.006(7)	0.003(5)	−0.005(5)
C24C	0.020(5)	0.026(9)	0.020(9)	−0.003(7)	0.003(6)	−0.006(5)
C25C	0.015(5)	0.019(9)	0.020(7)	−0.005(6)	0.005(5)	0.001(5)

Compound 40a (CCDC Deposition Number: 2521692) Data for **40a** were collected at 100 K on a Bruker D8 VENTURE Duo Fixed Chi Three-Circle Diffractometer using MoK α radiation ($\lambda = 0.71073$ Å). All data were integrated with SAINT V8.40B and corrected for absorption using SADABS 2016/2.^{20,21} The structure was solved by dual methods with SHELXT and refined by full-matrix least-squares methods against F^2 using SHELXL.^{22,23} All non-hydrogen atoms were refined with anisotropic displacement parameters. The locations of hydrogen atoms in the investigated could be identified from difference Fourier maps. The hydrogen atom bound to nitrogen atom of the carbamate group was placed and refined. Hydrogen atoms bound to carbon atoms were placed in geometrically calculated positions and refined using a riding model. Isotropic thermal parameters of the placed hydrogen atoms were fixed to 1.2 times the U value of the atoms they are linked to (1.5 times for methyl groups). Calculations and refinement of the structure was carried out using APEX4²⁴ and Olex2²⁵ software.

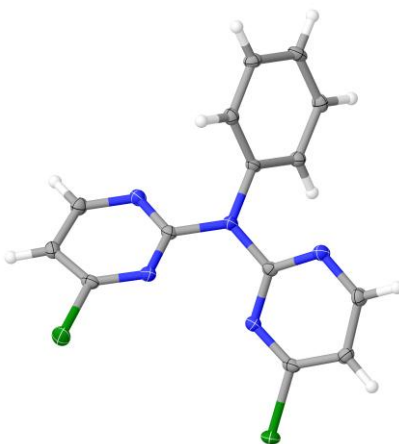


Figure C.13. X-ray crystal structure of **40a**.

Crystallographic Data for **40a**: $\text{C}_{14}\text{H}_9\text{Cl}_2\text{N}_5$ ($M=318.16$, g/mol), monoclinic, $a = 11.0973(2)$ Å, $b = 10.8141(2)$ Å, $c = 11.7483(2)$ Å, $\alpha = 90^\circ$, $\beta = 99.3340(10)^\circ$, $\gamma = 90^\circ$, $V = 1391.21(4)$ Å³, $Z = 4$, $T = 100$ K, $\mu(\text{MoK}\alpha) = 0.466\text{mm}^{-1}$, $\rho_{\text{calc}} = 1.519\text{g/cm}^3$, 46345 reflections measured, 3200 unique ($R_{\text{int}} = 0.0651$, $R_{\text{sigma}} = 0.0272$), $R_1 = 0.0338$ ($I > 2\sigma(I)$) and wR_2 was 0.0791 (all data).

Table C.22. Crystal data and structure refinement for **40a**.

CCDC number	2521692
Empirical formula	$\text{C}_{14}\text{H}_9\text{Cl}_2\text{N}_5$
Formula weight	318.16
Temperature [K]	100
Crystal system	monoclinic
Space group (number)	$P2_1/n$ (13)
a [Å]	11.0973(2)
b [Å]	10.8141(2)
c [Å]	11.7483(2)
α [°]	90
β [°]	99.3340(10)
γ [°]	90
Volume [Å ³]	1391.21(4)
Z	4
ρ_{calc} [gcm ⁻³]	1.519
μ [mm ⁻¹]	0.466
$F(000)$	648
Crystal size [mm ³]	0.04×0.06×0.1

Crystal colour	colourless
Crystal shape	plate
Radiation	MoK α ($\lambda=0.71073$ Å)
2 θ range [°]	5.15 to 54.96 (0.77 Å)
Index ranges	$-14 \leq h \leq 14$ $-14 \leq k \leq 14$ $-15 \leq l \leq 15$
Reflections collected	46345
Independent reflections	3200 $R_{\text{int}} = 0.0651$ $R_{\text{sigma}} = 0.0272$
Completeness to $\theta = 25.242^\circ$	99.9 %
Data / Restraints / Parameters	3200 / 0 / 193
Goodness-of-fit on F^2	1.049
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0338$ $wR_2 = 0.0712$
Final R indexes [all data]	$R_1 = 0.0532$ $wR_2 = 0.0791$
Largest peak/hole [eÅ $^{-3}$]	0.35/−0.32

Table C.23. Atomic coordinates and U_{eq} [Å 2] for **40a**.

Atom	x	y	z	U_{eq}
Cl1	0.50490(4)	0.02233(4)	0.29414(4)	0.02461(12)
N1	0.250000	0.39903(18)	0.250000	0.0155(4)
N2	0.37601(13)	0.22467(13)	0.27576(12)	0.0149(3)
N3	0.40596(13)	0.39439(13)	0.40795(13)	0.0166(3)
C1	0.34810(15)	0.33580(15)	0.31520(14)	0.0136(3)
C2	0.46733(16)	0.16761(15)	0.34135(15)	0.0158(4)
C3	0.53420(16)	0.21586(16)	0.44075(15)	0.0170(4)
H3	0.598450	0.171504	0.486442	0.020
C4	0.50060(16)	0.33332(16)	0.46863(15)	0.0174(4)
H4	0.546500	0.372941	0.533790	0.021
C5	0.250000	0.5324(2)	0.250000	0.0138(5)
C6	0.35181(16)	0.59605(16)	0.22663(15)	0.0155(3)
H6	0.420796	0.552183	0.209404	0.019
C7	0.35195(16)	0.72519(16)	0.22867(15)	0.0167(4)
H7	0.422391	0.769269	0.215351	0.020
C8	0.250000	0.7893(2)	0.250000	0.0182(5)
H8	0.250001	0.877104	0.250001	0.022
Cl2	0.83588(4)	0.04204(4)	0.49339(4)	0.01969(11)
N4	0.750000	0.41233(18)	0.250000	0.0129(4)
N5	0.79888(13)	0.23896(13)	0.36624(12)	0.0137(3)
N6	0.93842(13)	0.40869(13)	0.36909(13)	0.0160(3)

C9	0.83390(15)	0.34974(15)	0.33151(14)	0.0130(3)
C10	0.88027(16)	0.18395(15)	0.44419(15)	0.0152(3)
C11	0.99290(16)	0.23249(16)	0.48994(15)	0.0171(4)
H11	1.049677	0.189755	0.545438	0.020
C12	1.01638(16)	0.34802(16)	0.44862(16)	0.0182(4)
H12	1.091929	0.386500	0.478235	0.022
C13	0.750000	0.5460(2)	0.250000	0.0132(5)
C14	0.70496(16)	0.60913(16)	0.33659(15)	0.0159(4)
H14	0.674104	0.565029	0.395612	0.019
C15	0.70529(16)	0.73786(16)	0.33637(16)	0.0175(4)
H15	0.674747	0.781824	0.395655	0.021
C16	0.750000	0.8022(2)	0.250000	0.0170(5)
H16	0.749999	0.890083	0.249999	0.020

Table C.24. Anisotropic displacement parameters ($\text{\AA}^2$) for 40a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2(a^*)^2U_{11} + k^2(b^*)^2U_{22} + \dots + 2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	0.0232(2)	0.0142(2)	0.0341(3)	-0.00699(19)	-0.00215(19)	0.00535(17)
N1	0.0168(10)	0.0106(10)	0.0174(11)	0.000	-0.0019(8)	0.000
N2	0.0148(7)	0.0125(7)	0.0172(7)	-0.0005(6)	0.0017(6)	-0.0003(6)
N3	0.0189(8)	0.0139(7)	0.0160(7)	0.0000(6)	0.0004(6)	0.0009(6)
C1	0.0149(8)	0.0117(8)	0.0142(8)	0.0010(6)	0.0026(7)	-0.0006(6)
C2	0.0170(9)	0.0109(8)	0.0204(9)	-0.0003(7)	0.0057(7)	0.0001(7)
C3	0.0144(9)	0.0164(8)	0.0196(9)	0.0026(7)	0.0011(7)	0.0014(7)
C4	0.0183(9)	0.0188(9)	0.0142(9)	-0.0009(7)	-0.0003(7)	-0.0016(7)
C5	0.0174(12)	0.0096(11)	0.0134(11)	0.000	-0.0004(9)	0.000
C6	0.0161(9)	0.0148(8)	0.0154(8)	-0.0008(7)	0.0019(7)	0.0016(7)
C7	0.0177(9)	0.0160(8)	0.0164(9)	0.0012(7)	0.0028(7)	-0.0046(7)
C8	0.0272(14)	0.0103(11)	0.0164(12)	0.000	0.0019(10)	0.000
Cl2	0.0231(2)	0.0133(2)	0.0220(2)	0.00480(16)	0.00169(17)	0.00196(16)
N4	0.0133(10)	0.0090(9)	0.0149(10)	0.000	-0.0022(8)	0.000
N5	0.0142(7)	0.0129(7)	0.0139(7)	-0.0002(6)	0.0014(6)	0.0009(5)
N6	0.0131(7)	0.0147(7)	0.0194(8)	-0.0013(6)	0.0000(6)	0.0001(6)
C9	0.0137(8)	0.0128(8)	0.0128(8)	-0.0018(6)	0.0025(6)	0.0024(6)
C10	0.0198(9)	0.0110(8)	0.0156(8)	-0.0007(6)	0.0050(7)	0.0024(7)
C11	0.0156(9)	0.0176(8)	0.0166(9)	-0.0009(7)	-0.0018(7)	0.0051(7)
C12	0.0136(9)	0.0186(8)	0.0212(9)	-0.0031(7)	-0.0008(7)	0.0013(7)
C13	0.0109(11)	0.0096(11)	0.0175(12)	0.000	-0.0020(9)	0.000
C14	0.0149(8)	0.0162(8)	0.0163(9)	-0.0002(7)	0.0018(7)	-0.0012(7)
C15	0.0159(9)	0.0163(8)	0.0201(9)	-0.0042(7)	0.0023(7)	0.0025(7)
C16	0.0140(12)	0.0106(11)	0.0241(13)	0.000	-0.0041(10)	0.000

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