

Supplementary Information
For
Synthesis of Fused Cyanopyrroles and Spirocyclopropanes Via Addition of N-Ylide to
Chalconimines

Siba Prasad Midya, Elumalai Gopi, Nishikant Satam and Irishi N. N. Namboothiri*

Department of Chemistry and Indian Institute of Technology, Bombay, Mumbai 400 076
irishi@iitb.ac.in

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EXPNO 6
PROCNO 1

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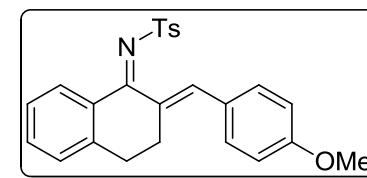
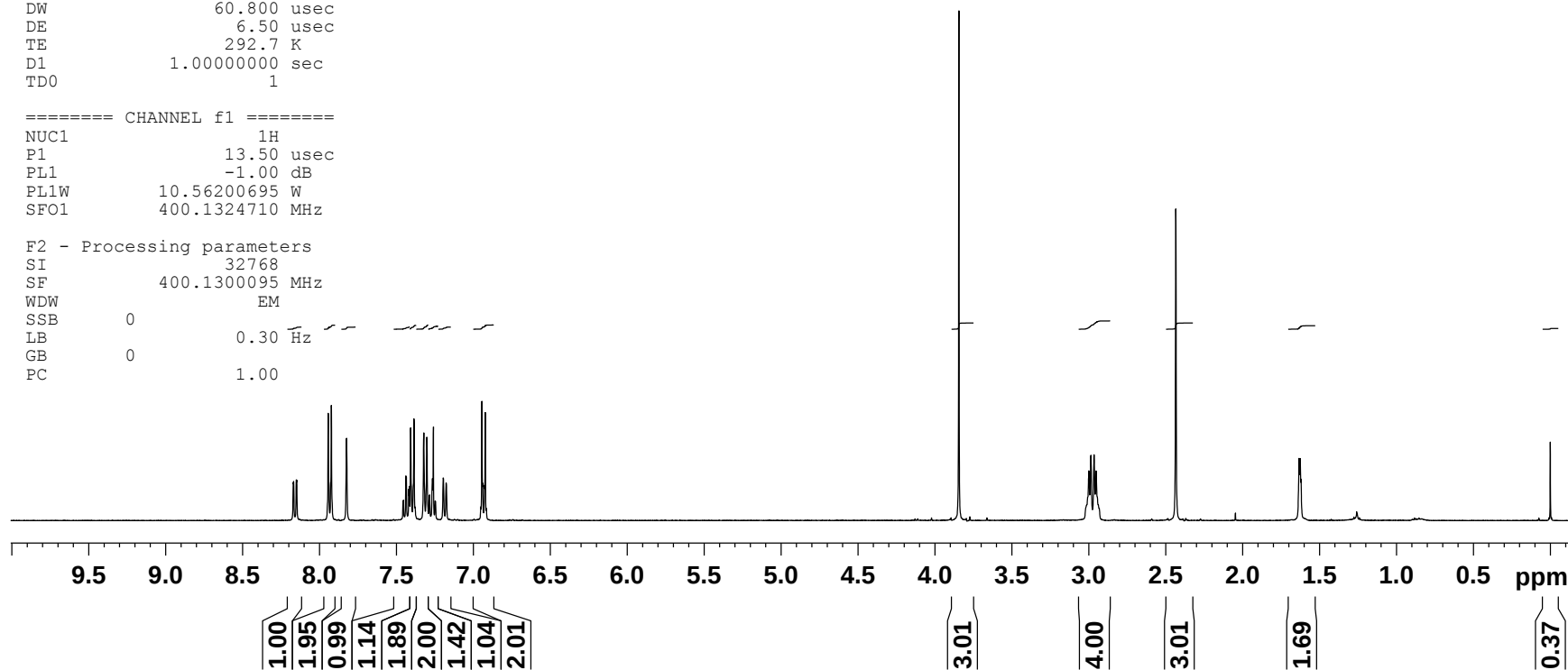
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D1 1.00000000 sec
TD0 1

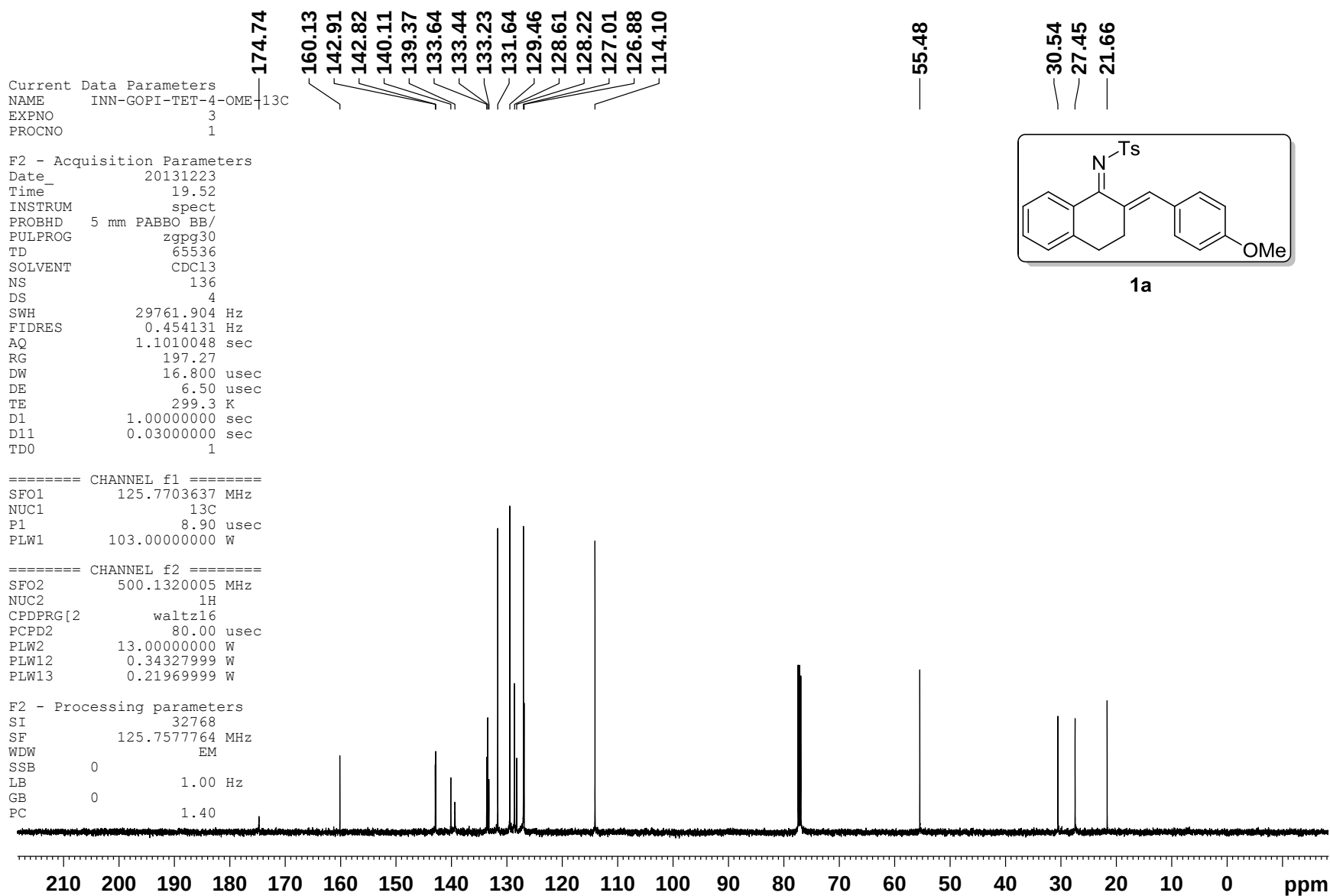
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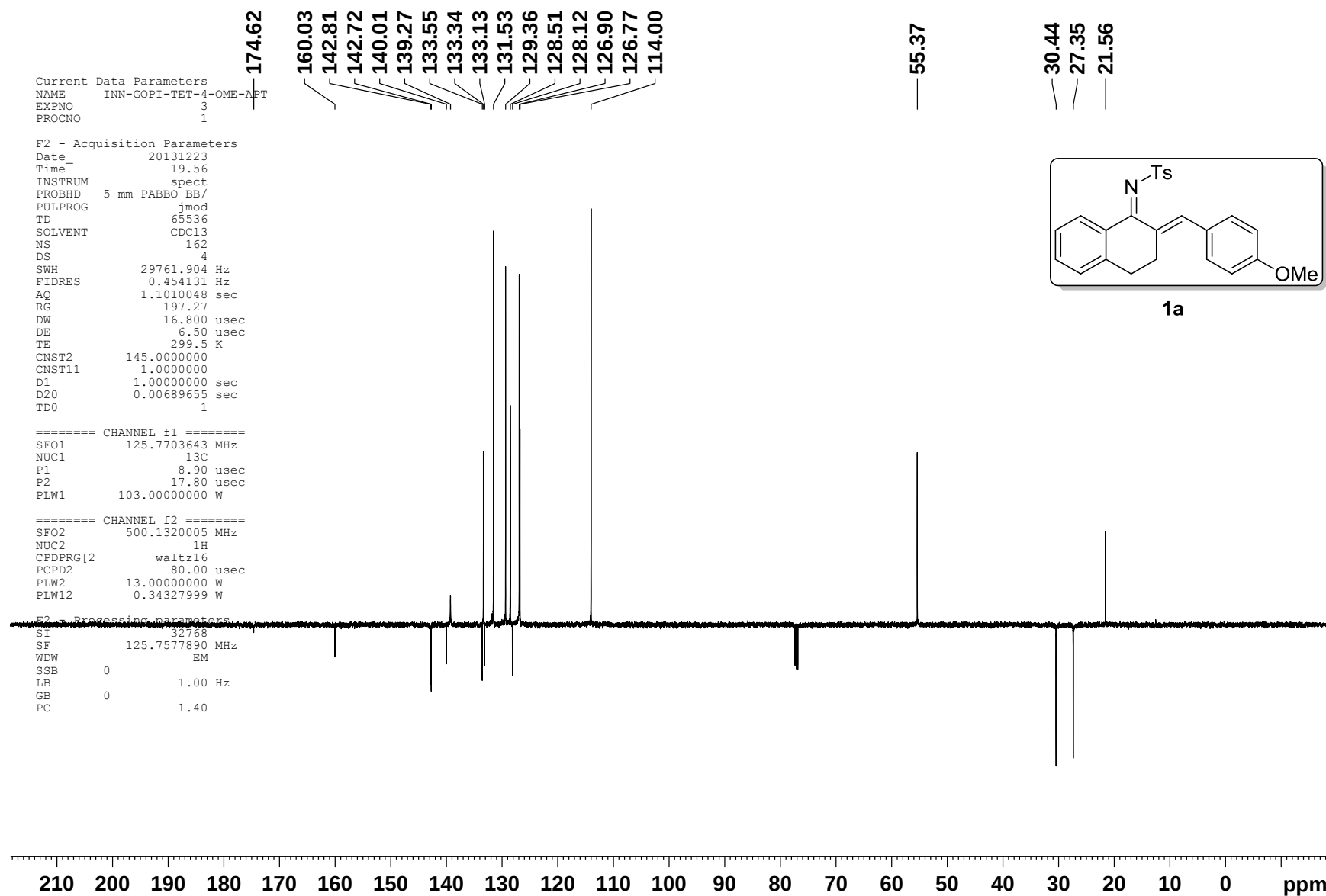
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PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters

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

**1a**Figure S01. ¹H NMR Spectrum of 1a



Figure S03. ¹³C-APT NMR Spectrum of 1a

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EXPNO 1
PROCNO 1

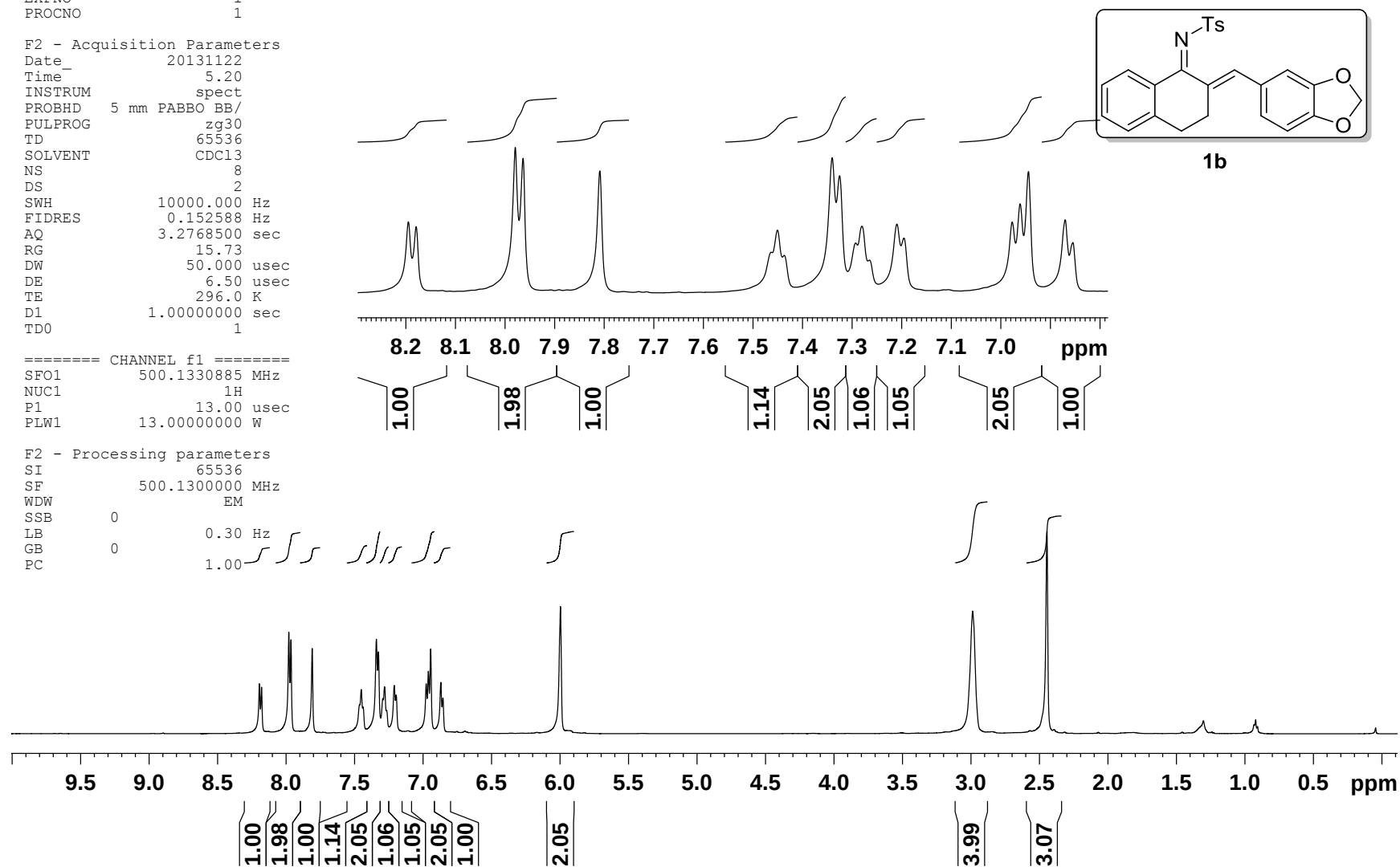
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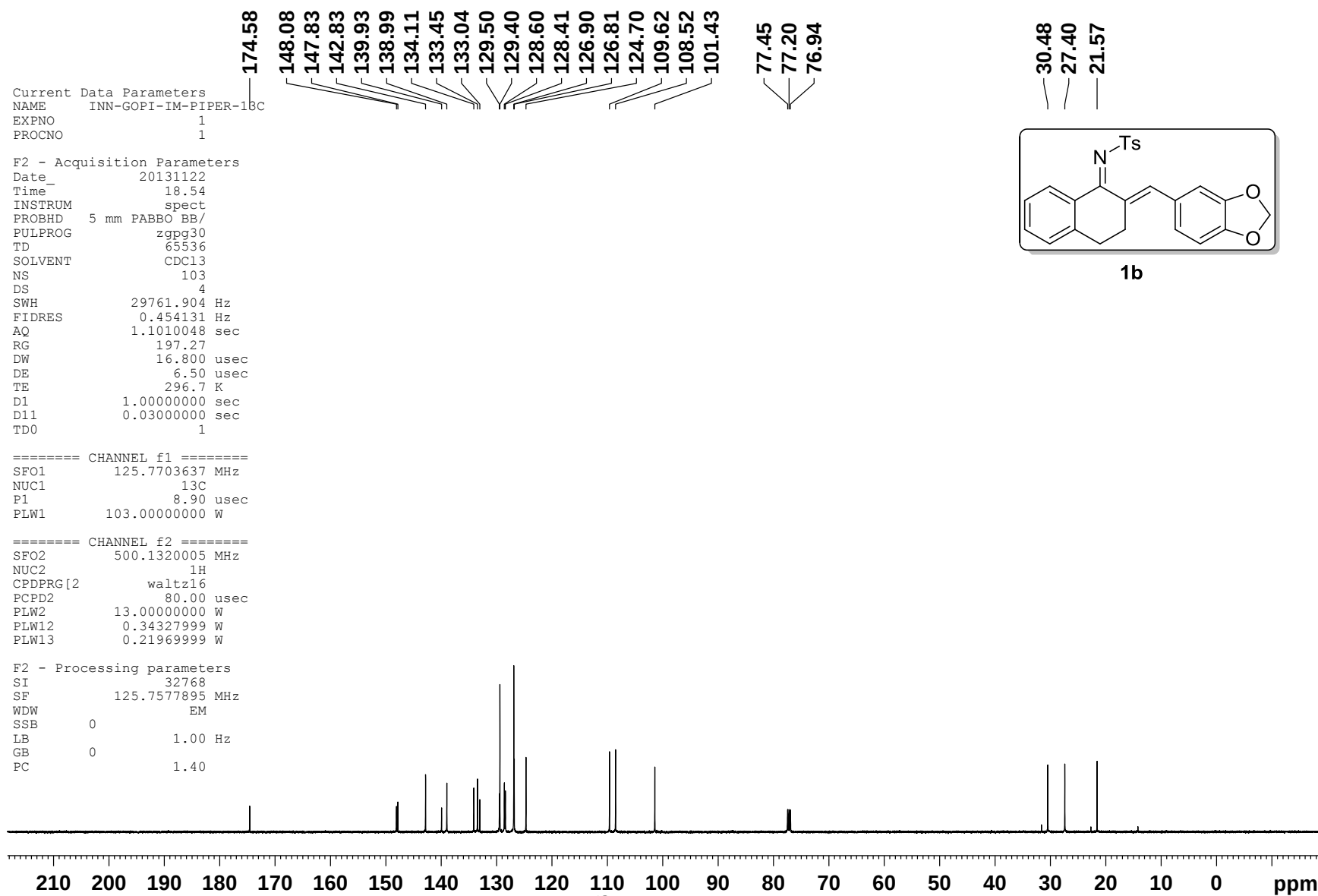
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PULPROG zg30
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SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 15.73
DW 50.000 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
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NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters

SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S04. ¹H NMR Spectrum of 1b

Figure S05. ¹³C NMR Spectrum of 1b

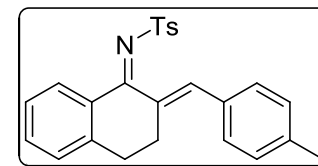


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 EXPNO 1
 PROCNO 1

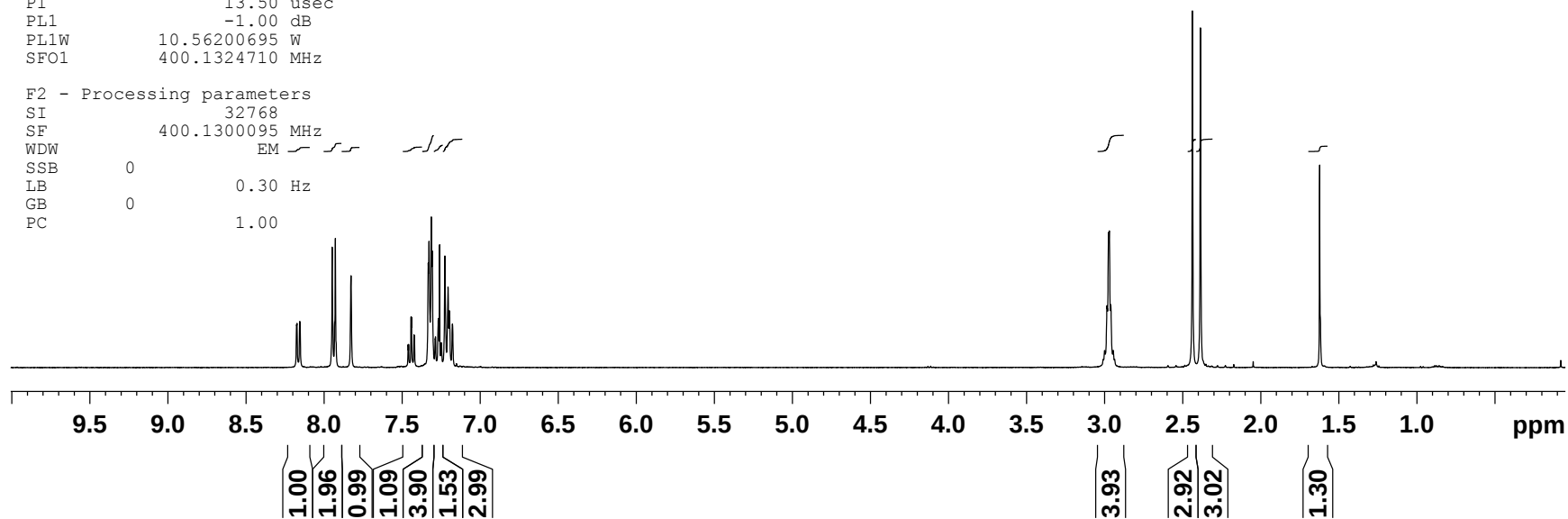
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 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 7
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 293.8 K
 D1 1.00000000 sec
 TD0 1

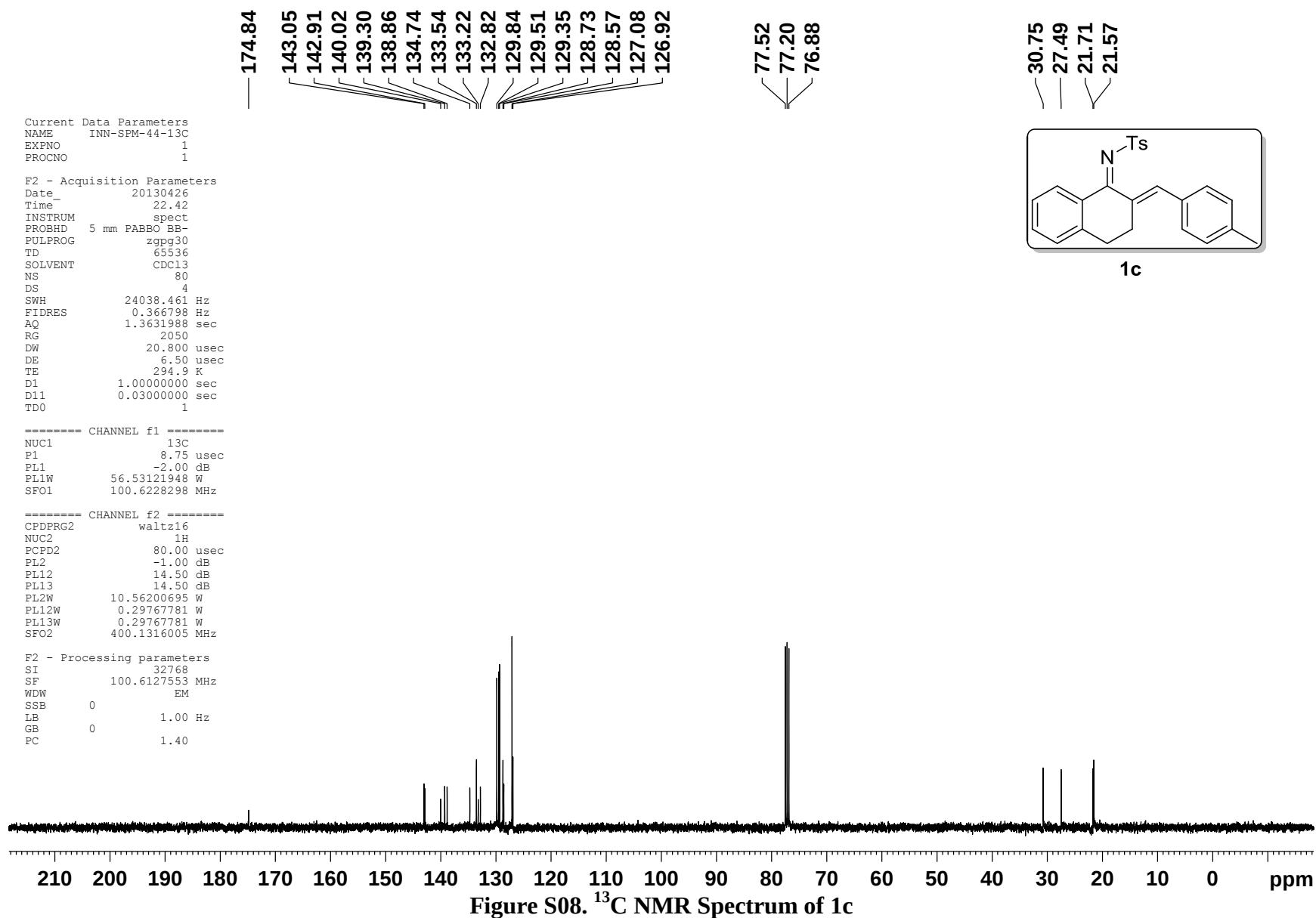
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 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

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



1c

Figure S07. ¹H NMR Spectrum of 1c

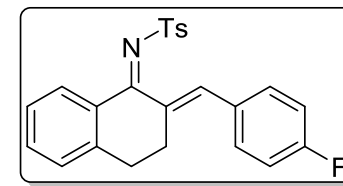
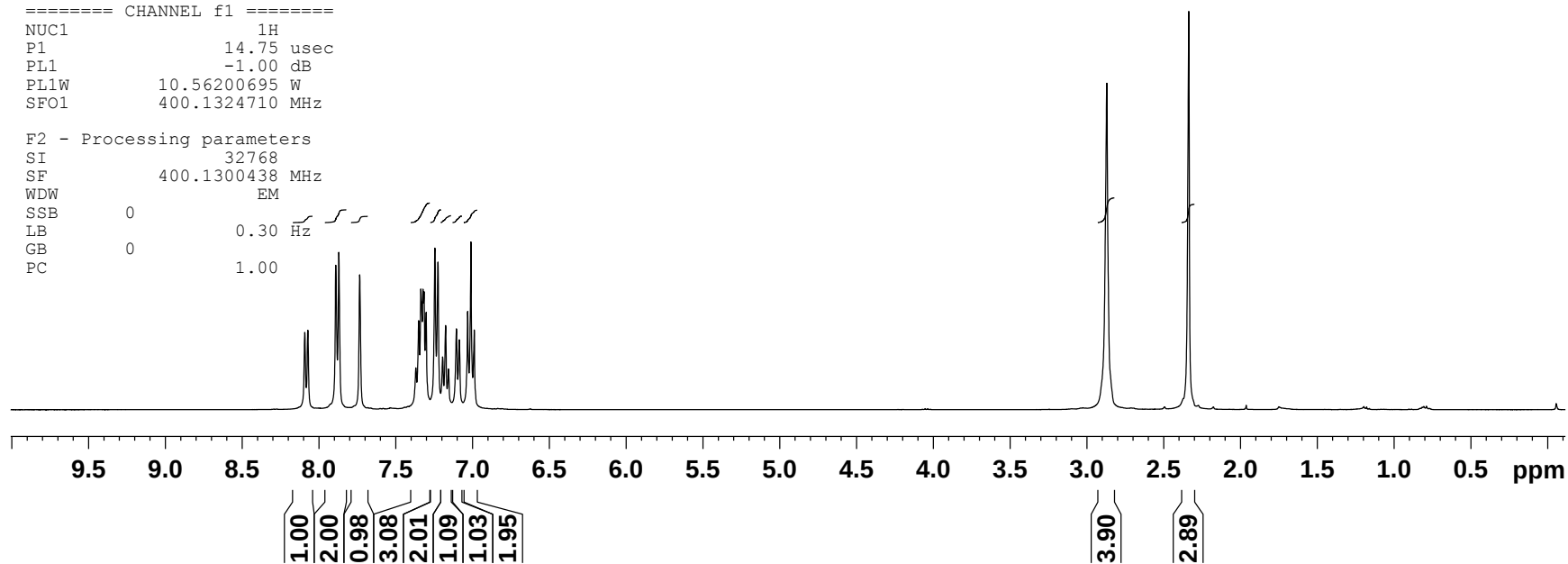


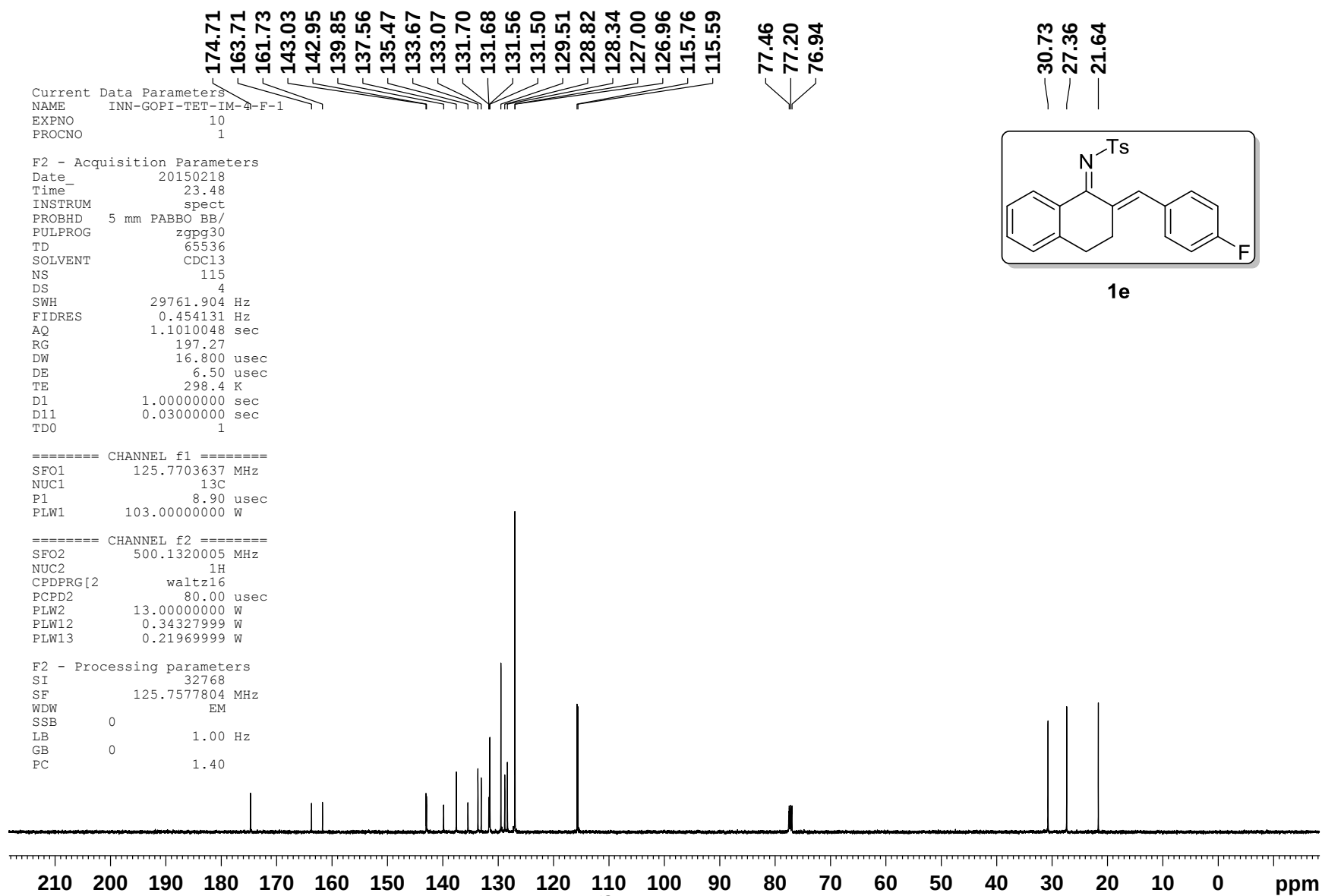
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PROCNO 1

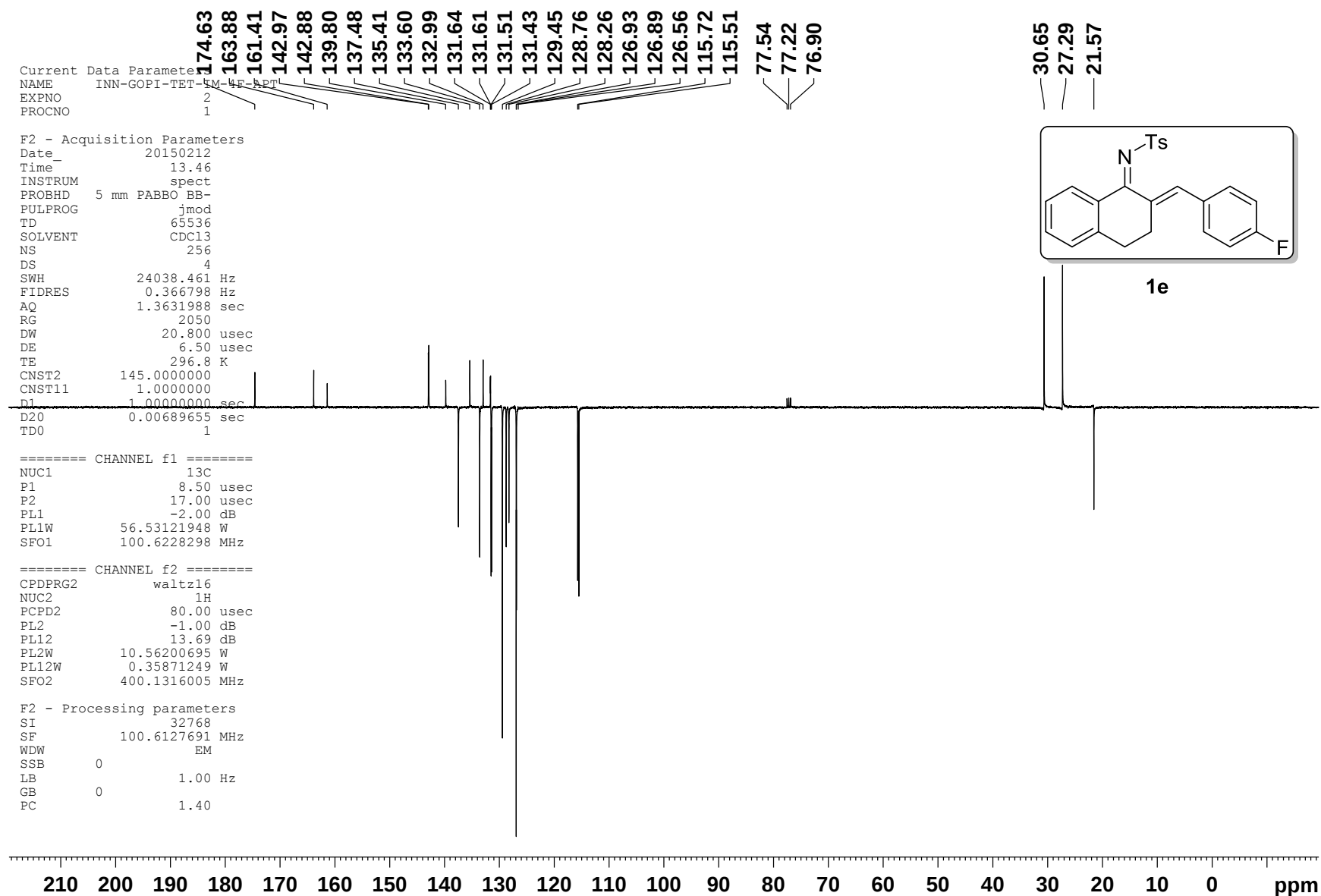
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TD 54274
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2999091 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.75 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300438 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**1e****Figure S09. ¹H NMR Spectrum of 1e**

Figure S10. ¹³C NMR Spectrum of 1e

Figure S11. ¹³C-APT NMR Spectrum of 1e

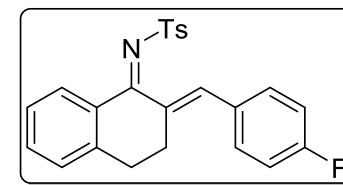
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EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
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Time_ 10.20
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PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 40
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 119.07
DW 4.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 470.5453180 MHz
NUC1 19F
P1 13.50 usec
PLW1 40.00000000 W

F2 - Processing parameters
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SF 470.5923770 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

-111.95



1e

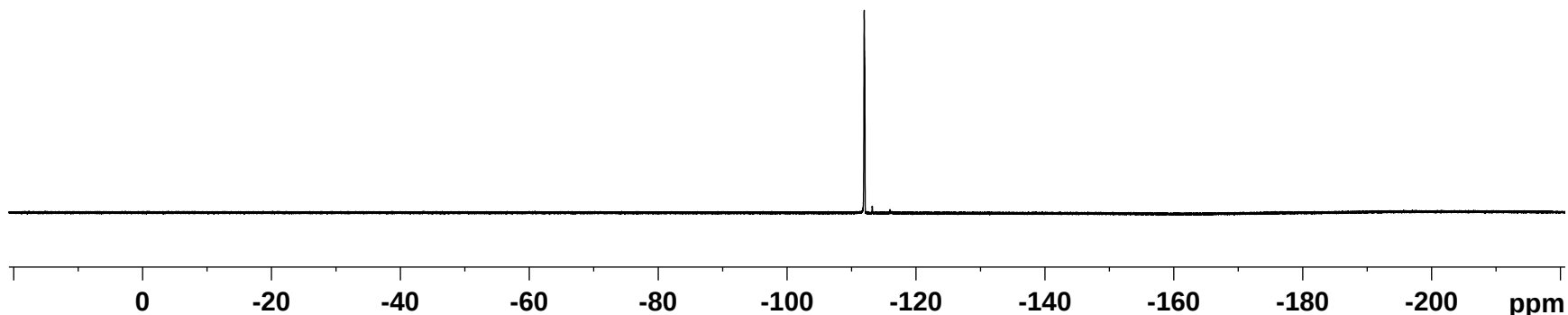


Figure S12. ^{19}F NMR Spectrum of 1e

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EXPNO 2
PROCNO 1

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Time_ 23.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

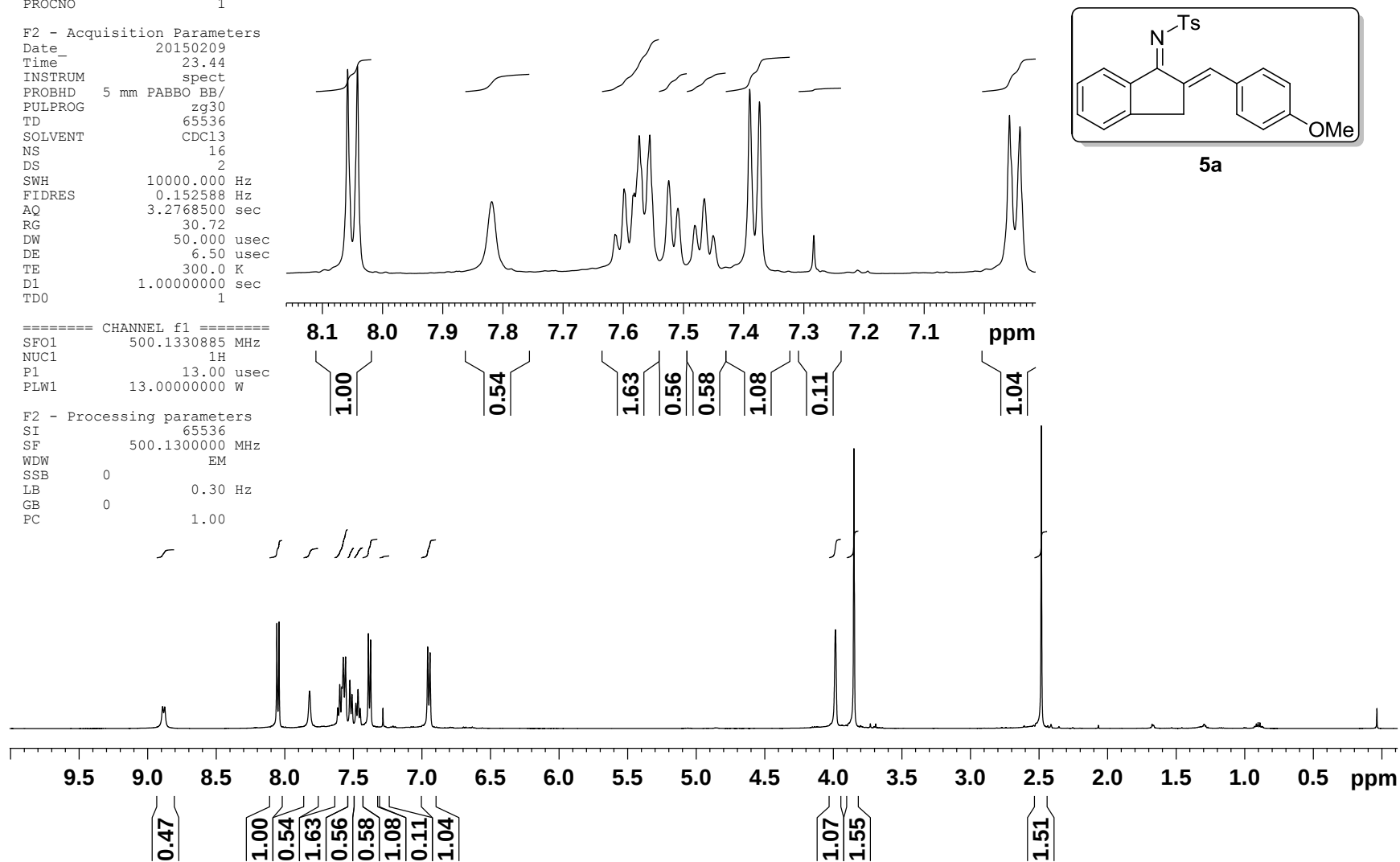
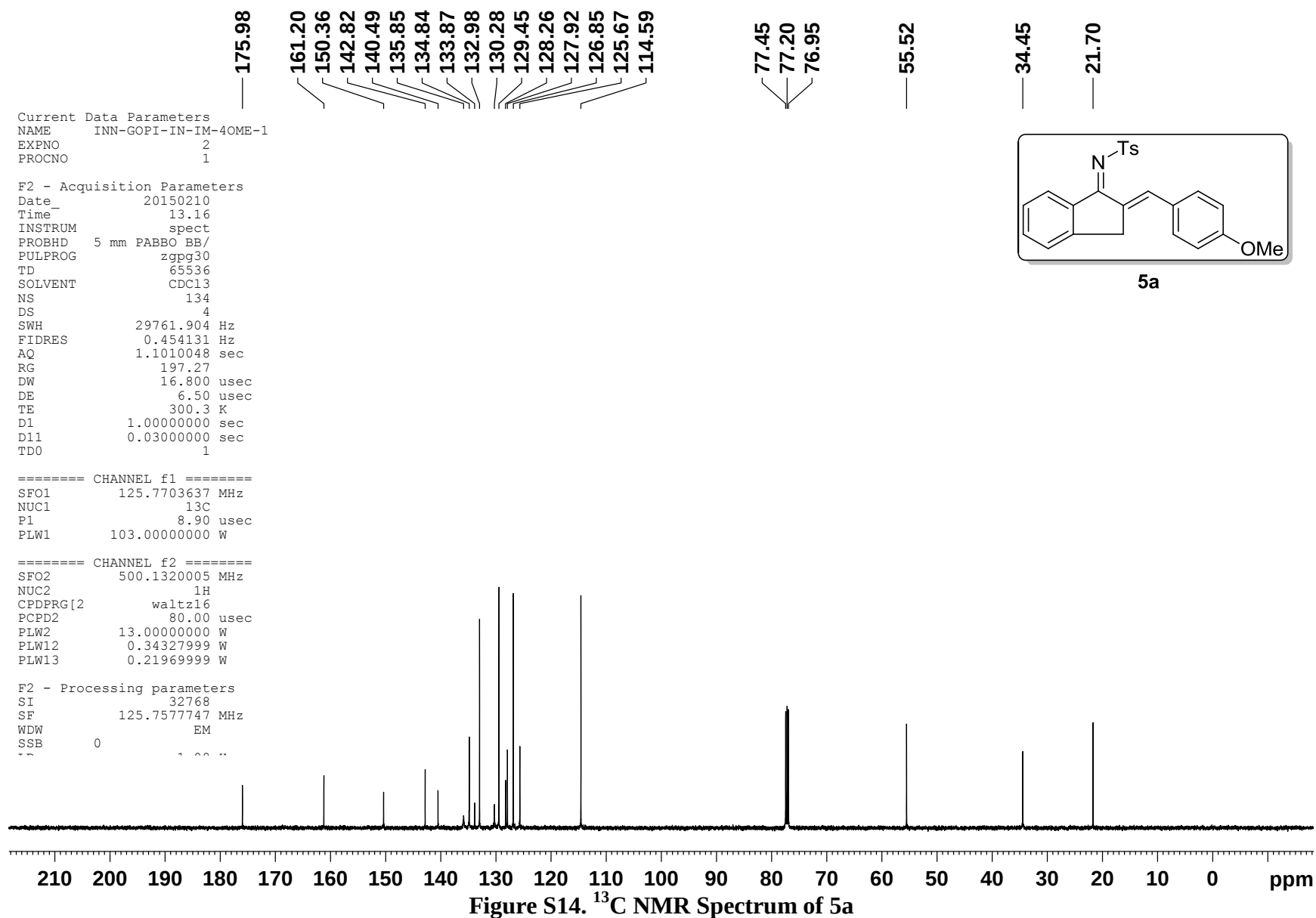
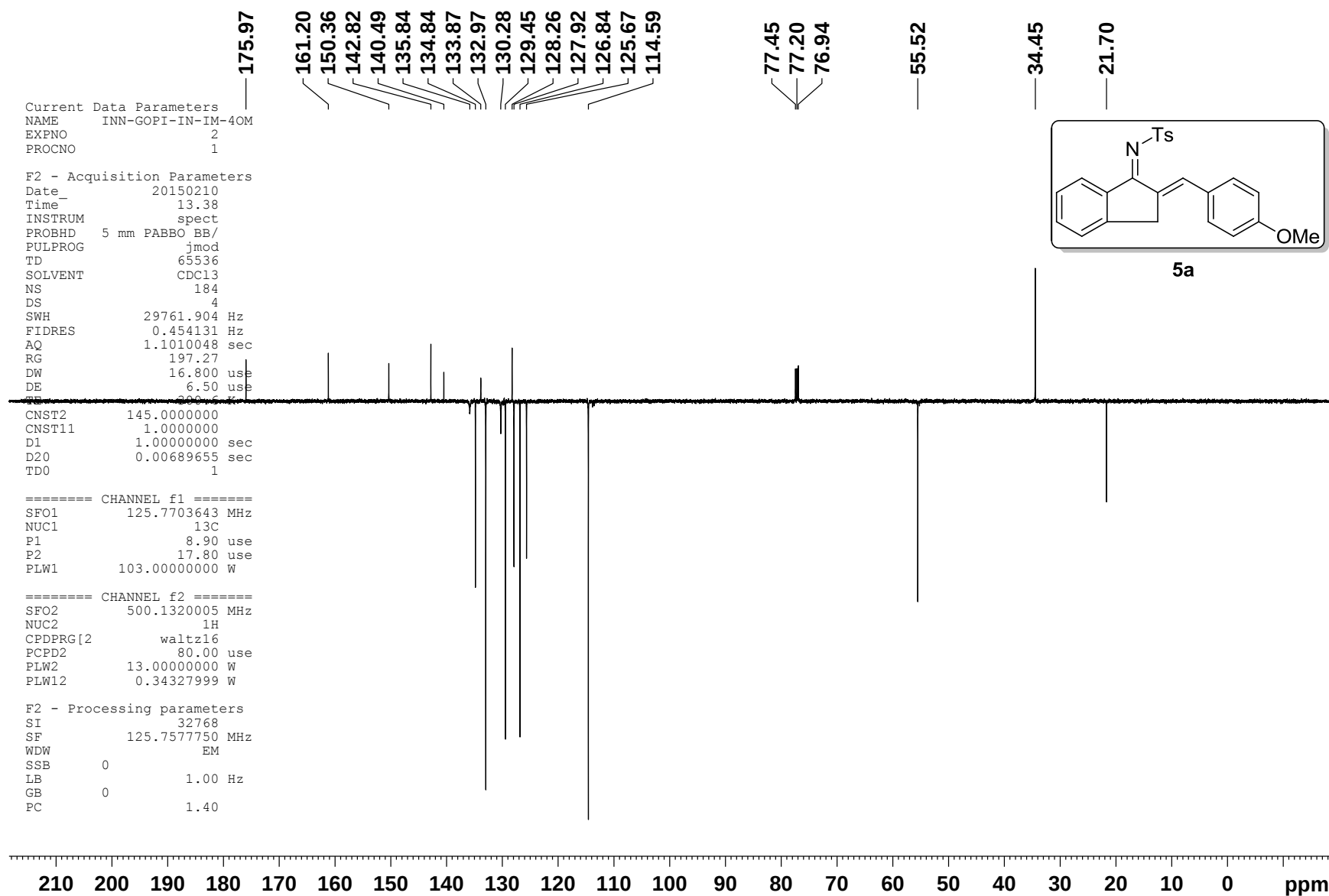


Figure S13. ¹H NMR Spectrum of 5a



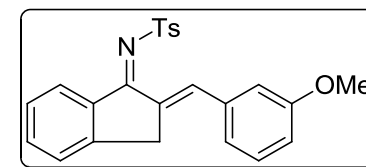
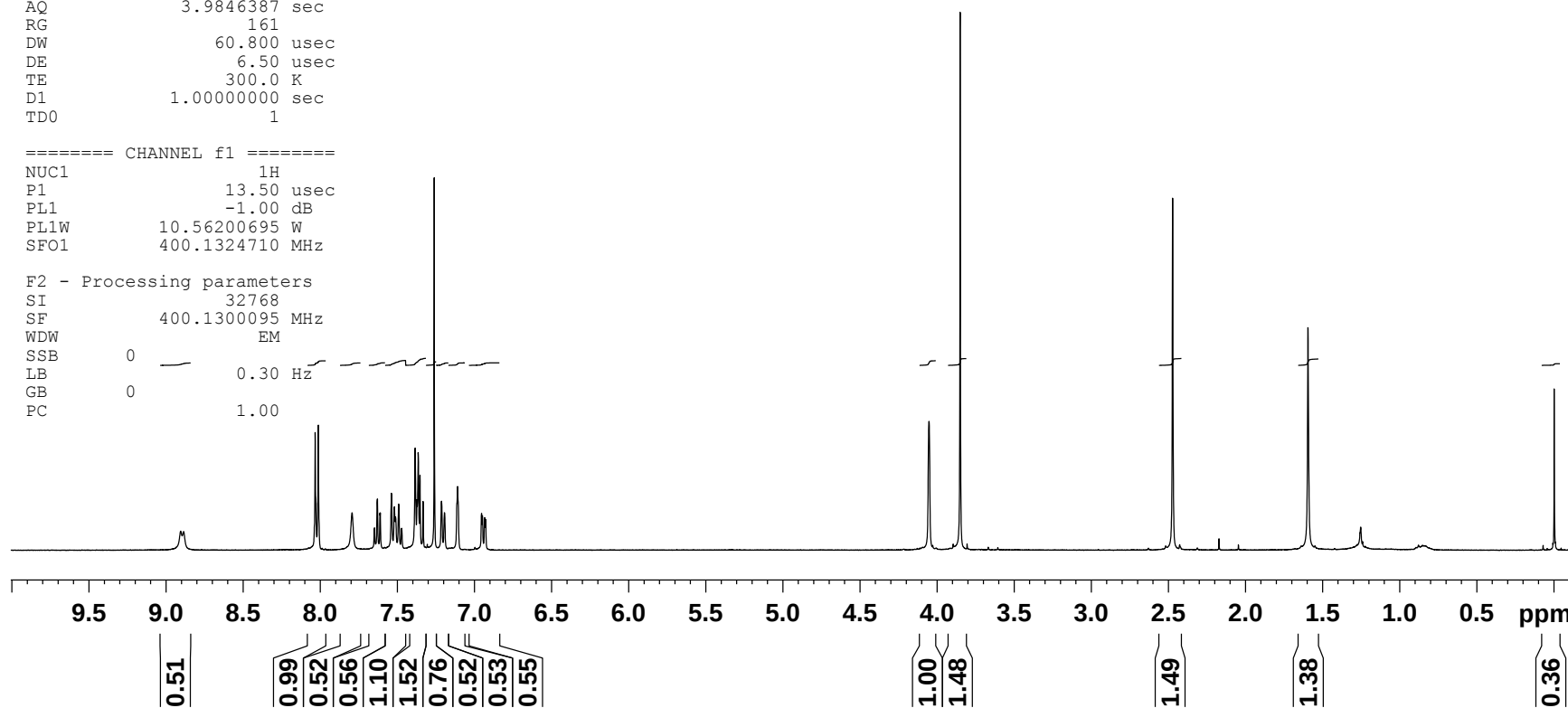
Figure S15. ¹³C-APT NMR Spectrum of 5a

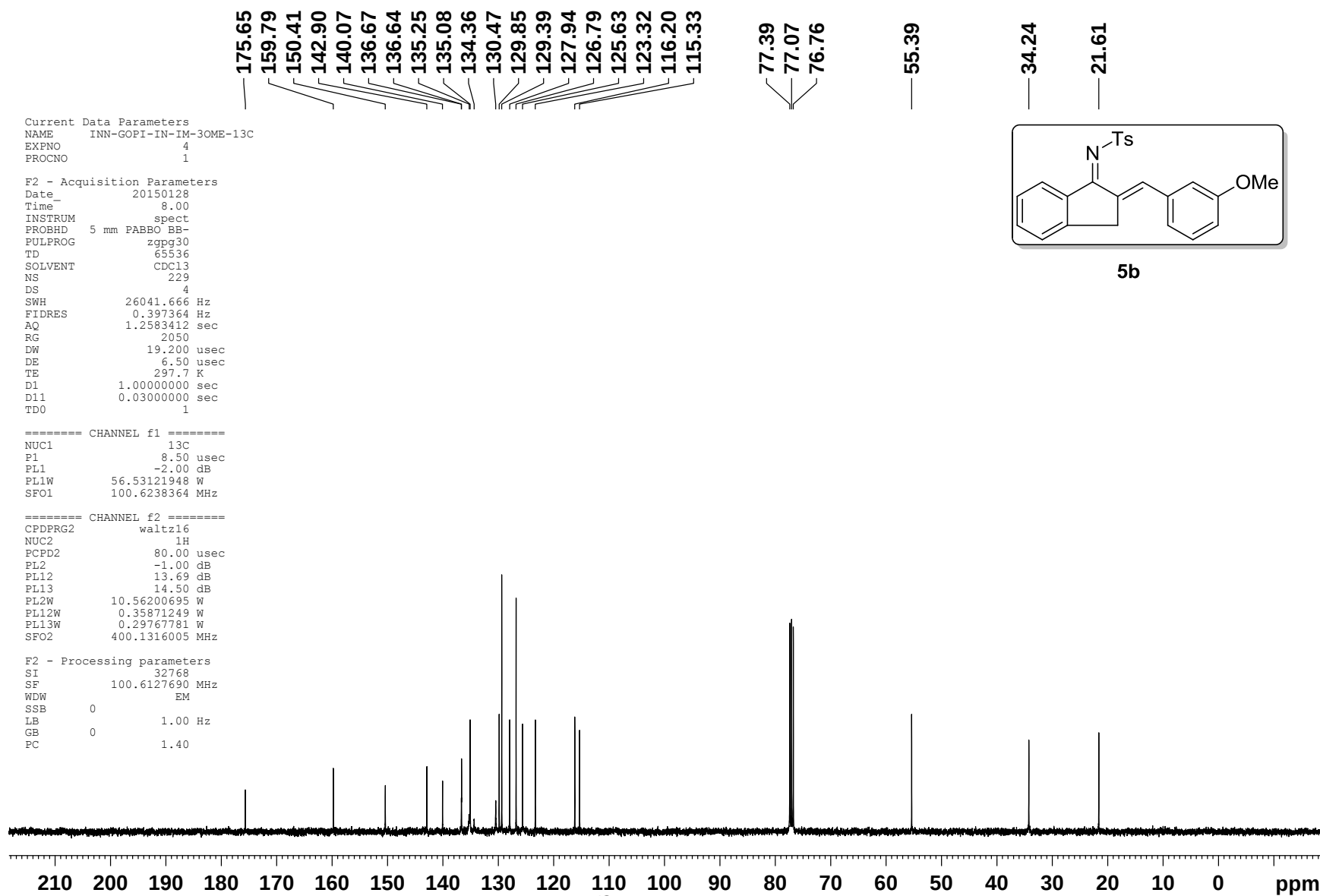
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EXPNO 3
PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.130095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

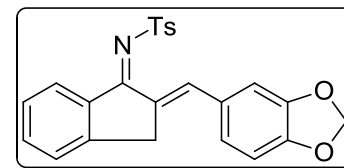
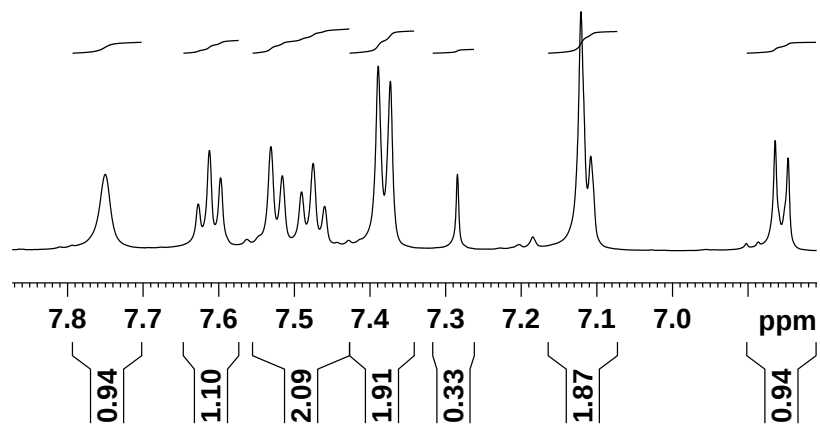
**5b****Figure S16. ¹H NMR Spectrum of 5b**

Figure S17. ¹³C NMR Spectrum of 5b

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 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters

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 Time_ 4.38
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 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 11
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 30.72
 DW 50.000 usec
 DE 6.50 usec
 TE 295.0 K
 D1 1.00000000 sec
 TDO 1

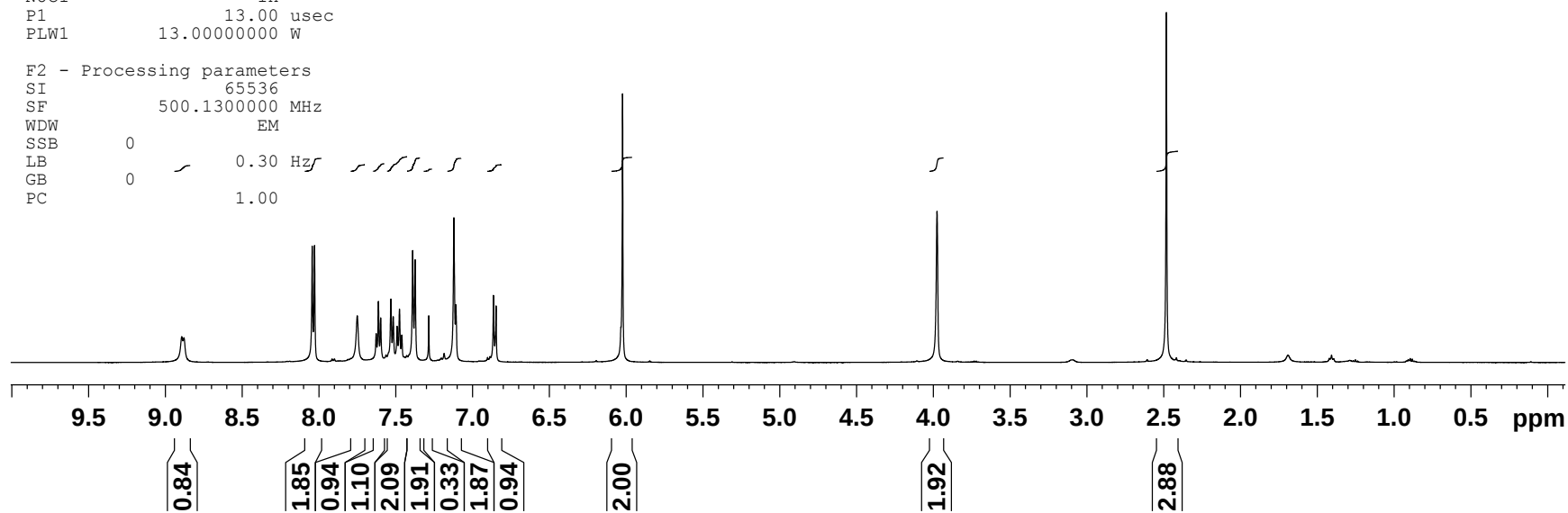


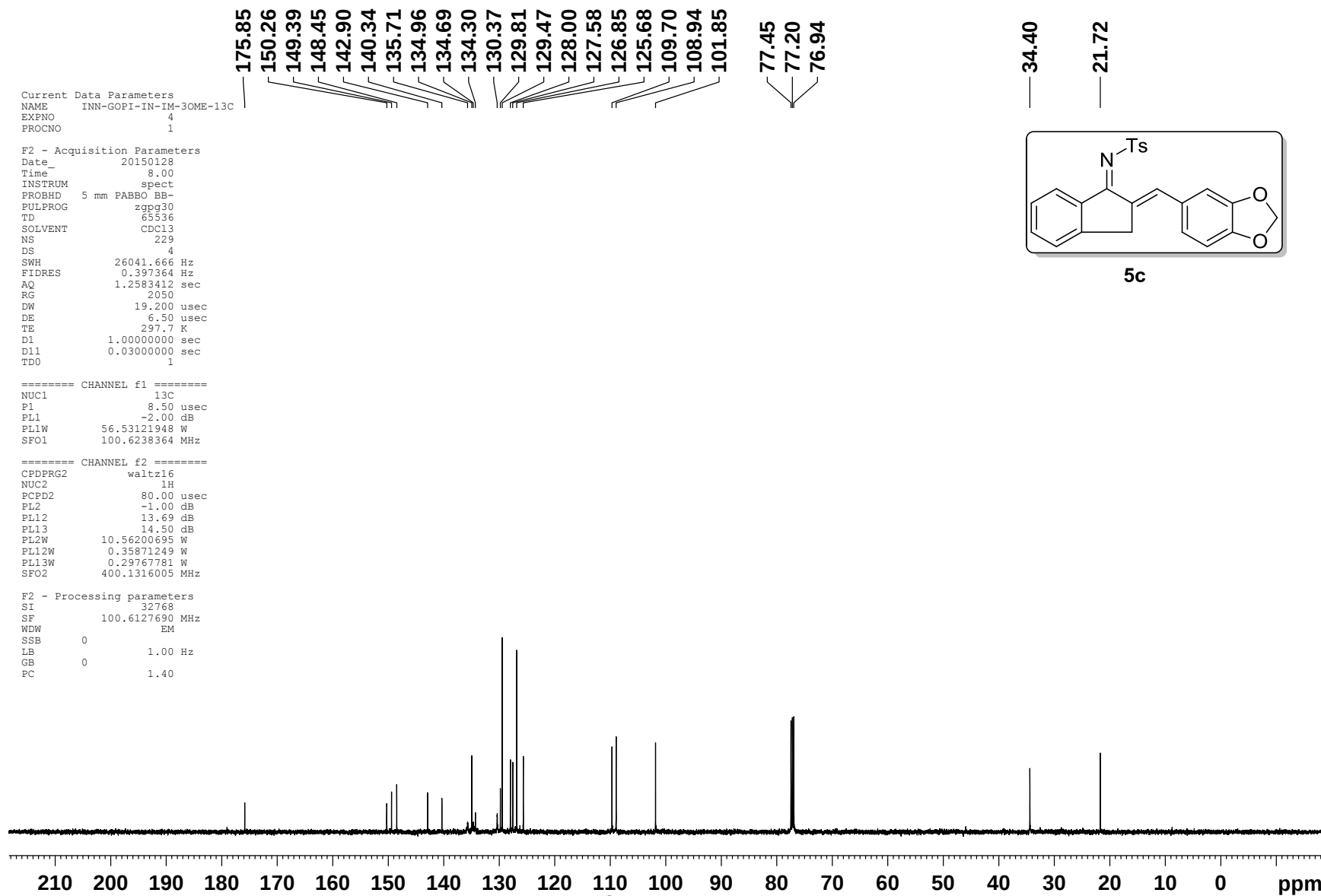
5c

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 NUC1 1H
 P1 13.00 usec
 PLW1 13.00000000 W

F2 - Processing parameters

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 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S18. ¹H NMR Spectrum of 5c

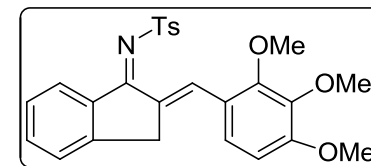
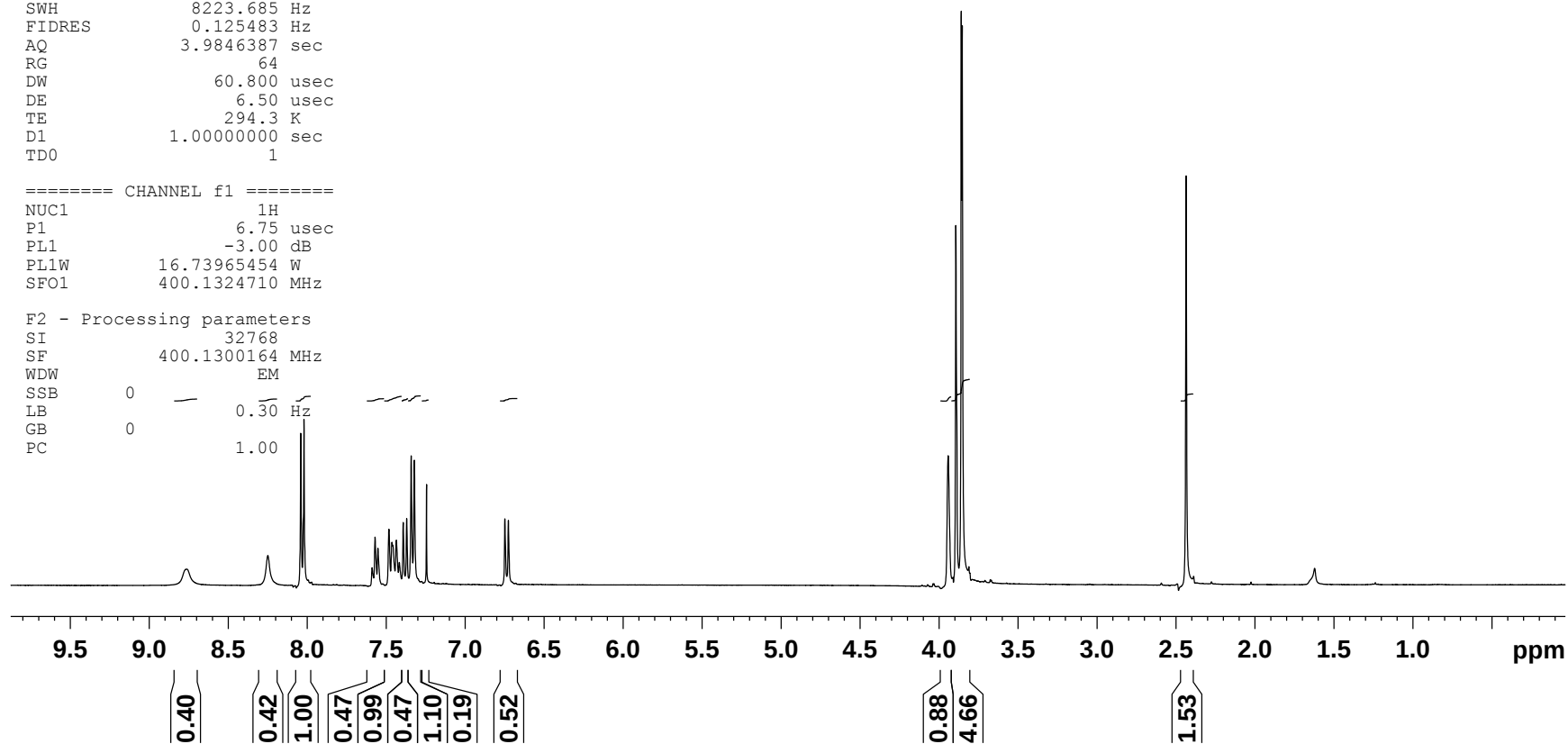
Figure S19. ¹³C NMR Spectrum of 5c

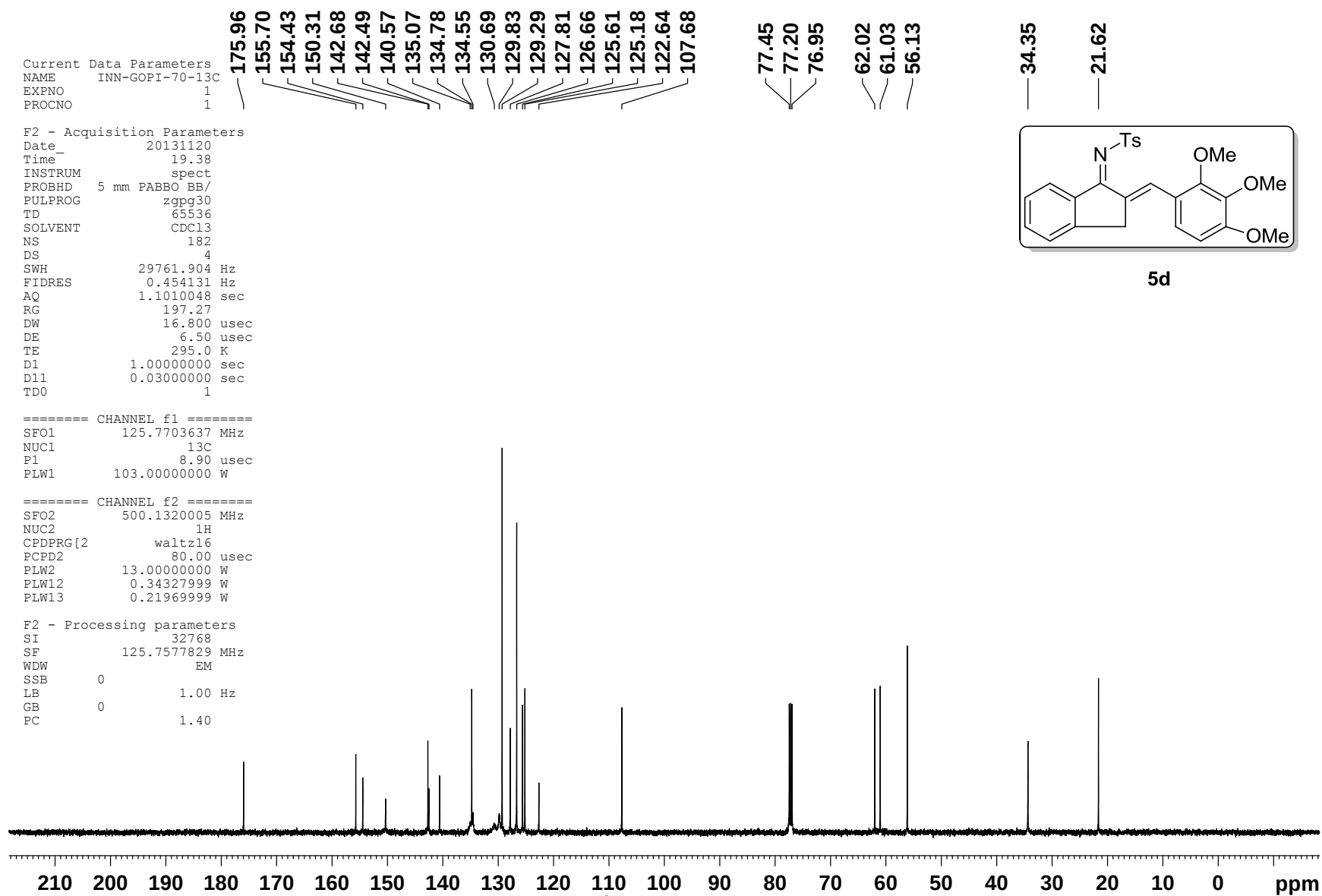
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EXPNO 1
PROCNO 1

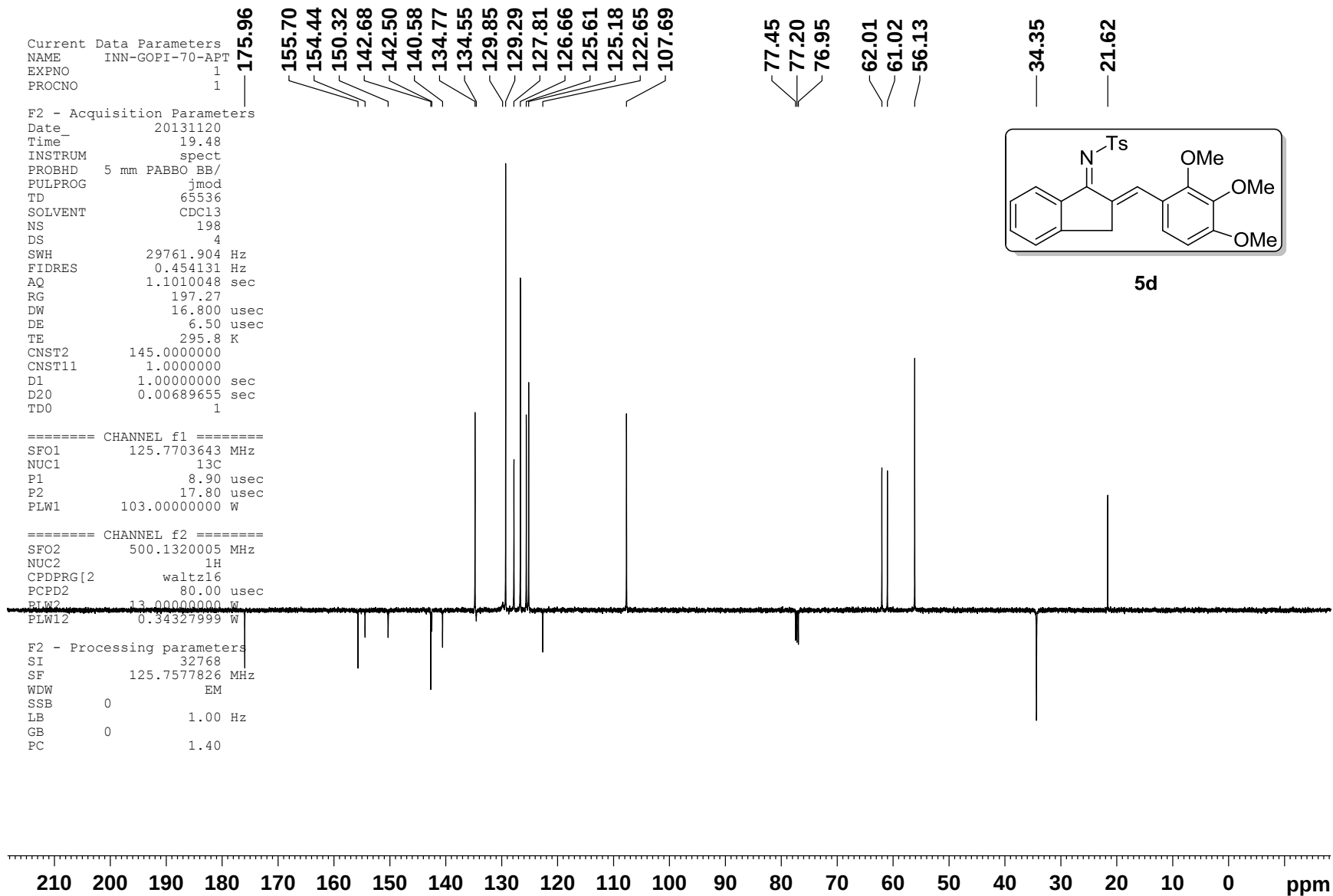
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.75 usec
PL1 -3.00 dB
PL1W 16.73965454 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**5d****Figure S20. ¹H NMR Spectrum of 5d**

Figure S21. ¹³C NMR Spectrum of 5d

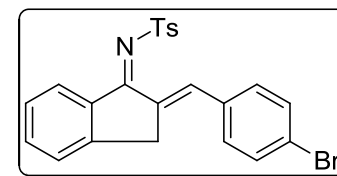
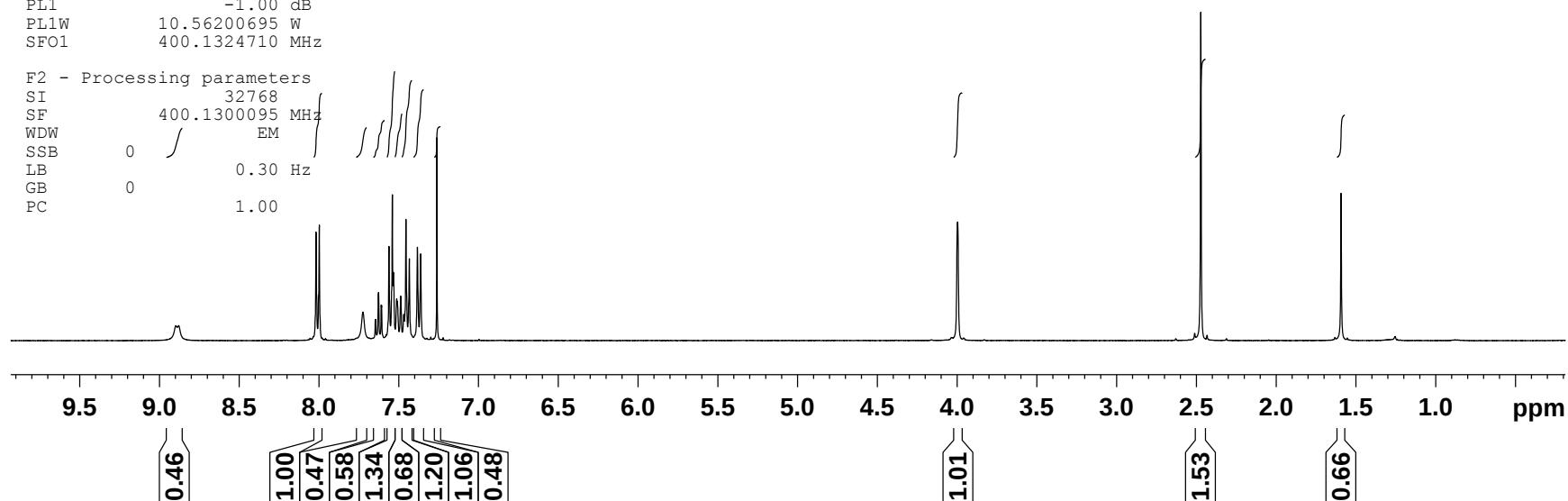
Figure S22. ¹³C-APT NMR Spectrum of 5d

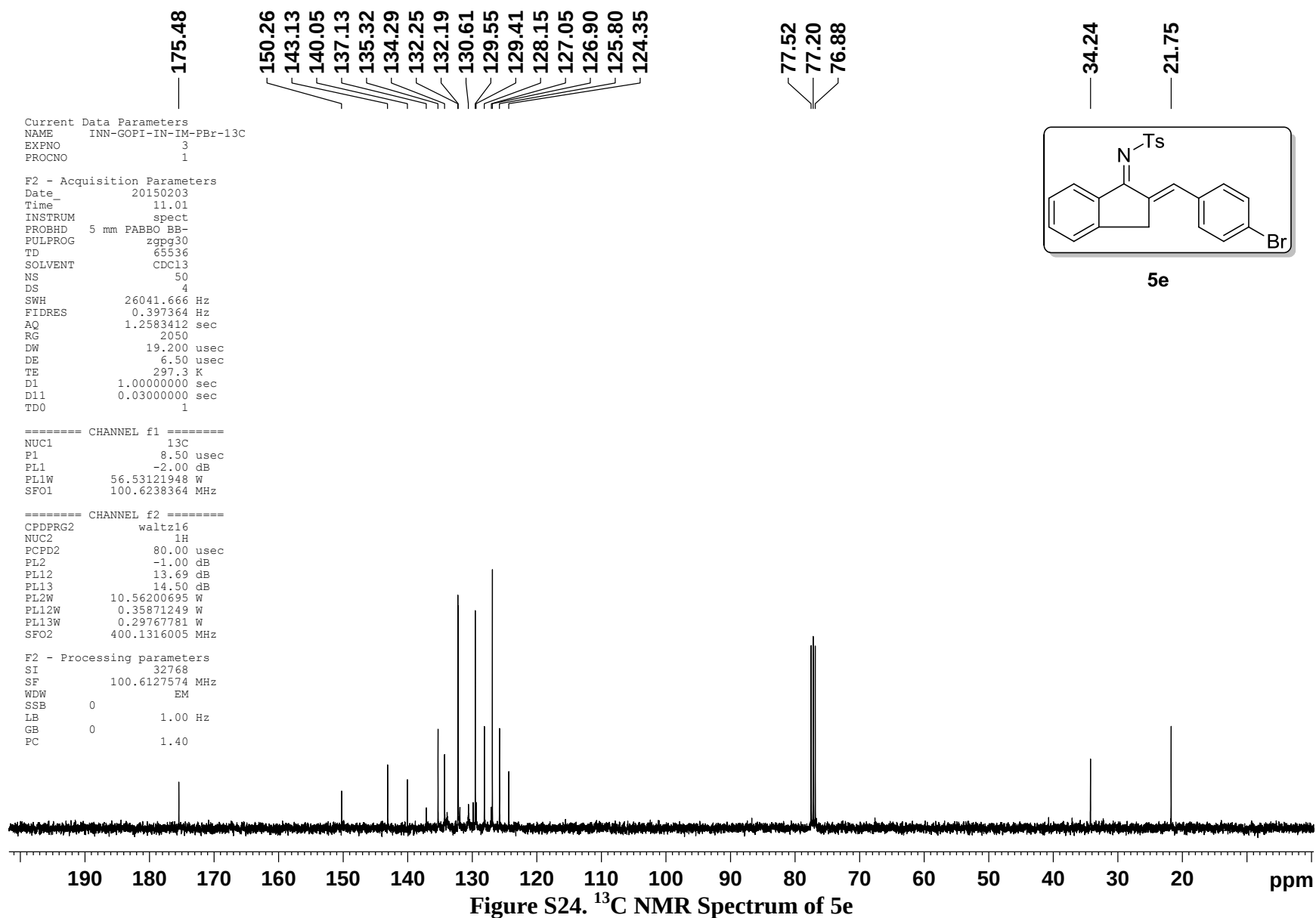
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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
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GB 0
PC 1.00

**5e****Figure S23. ¹H NMR Spectrum of 5e**

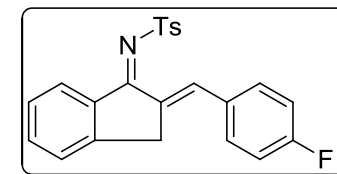
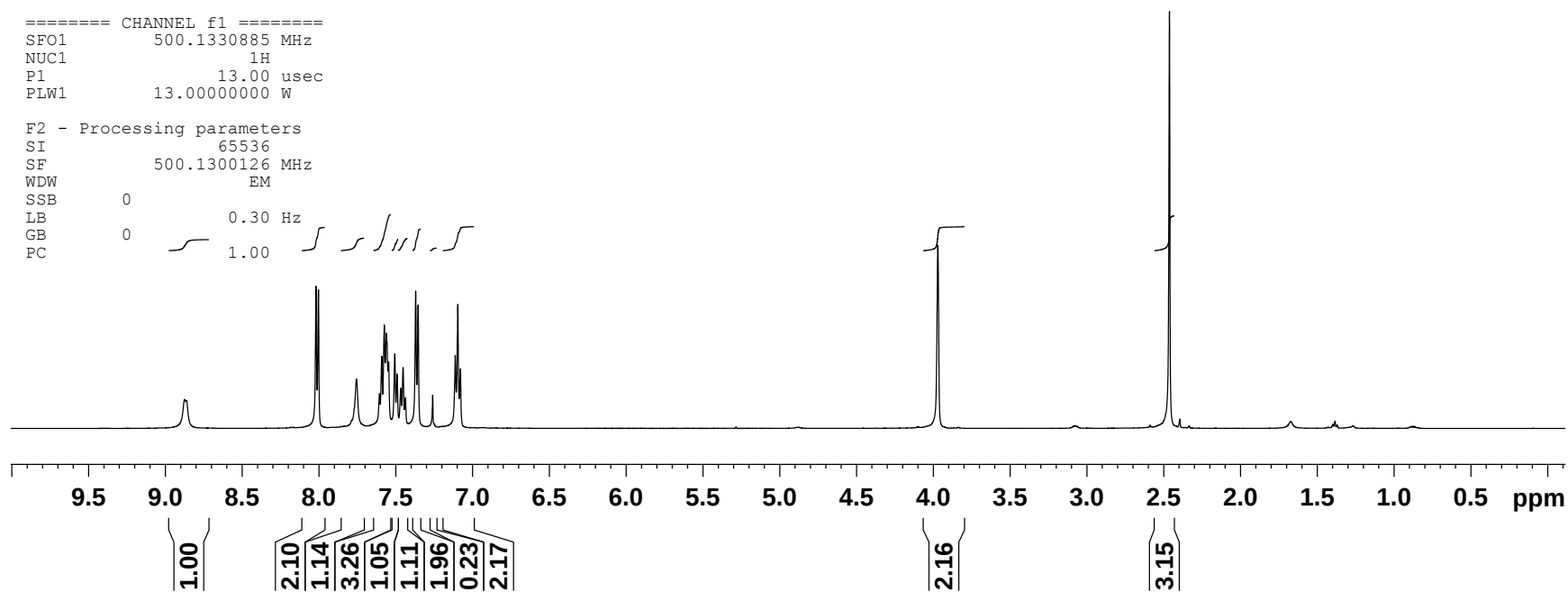


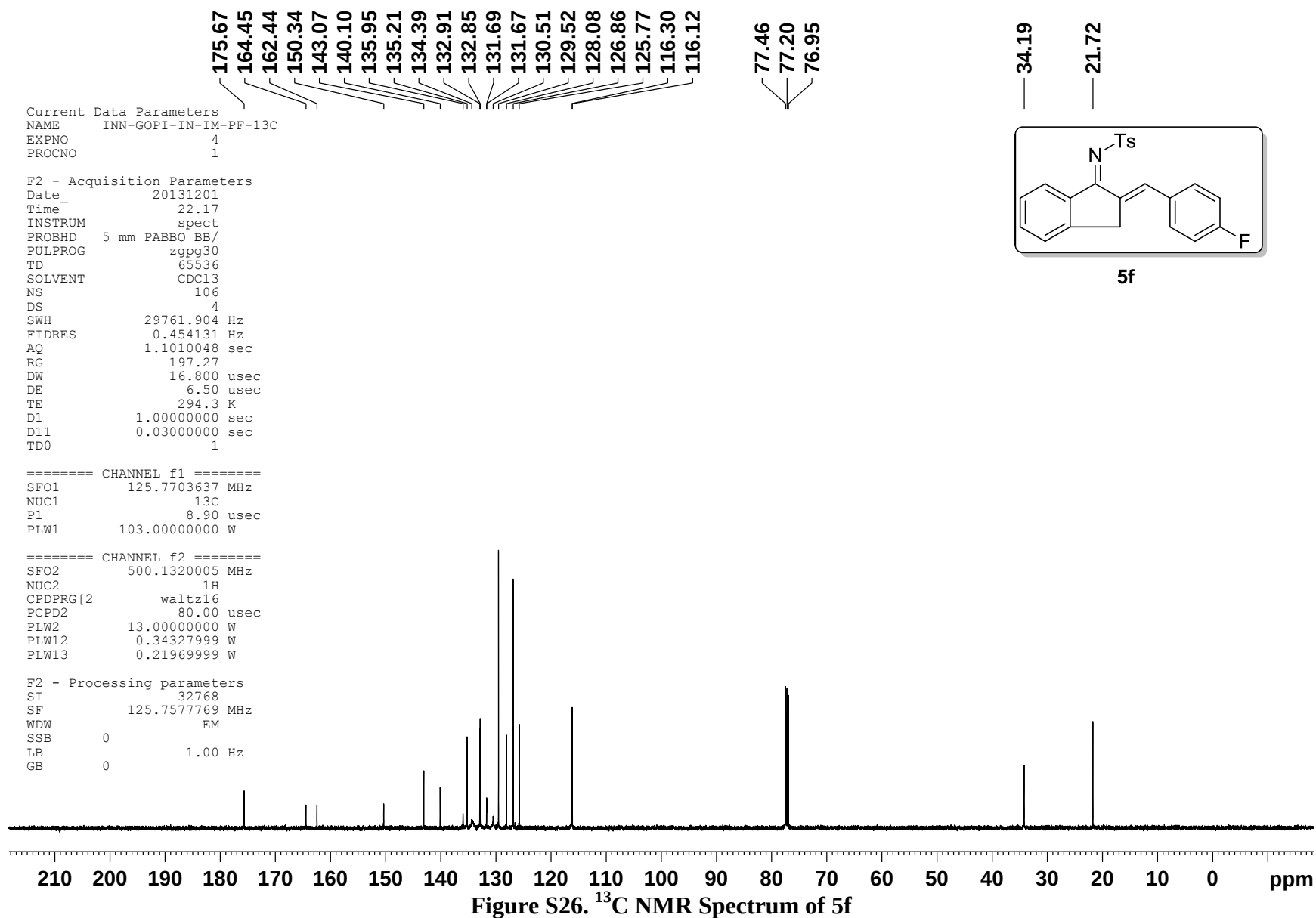
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EXPNO 1
PROCNO 1

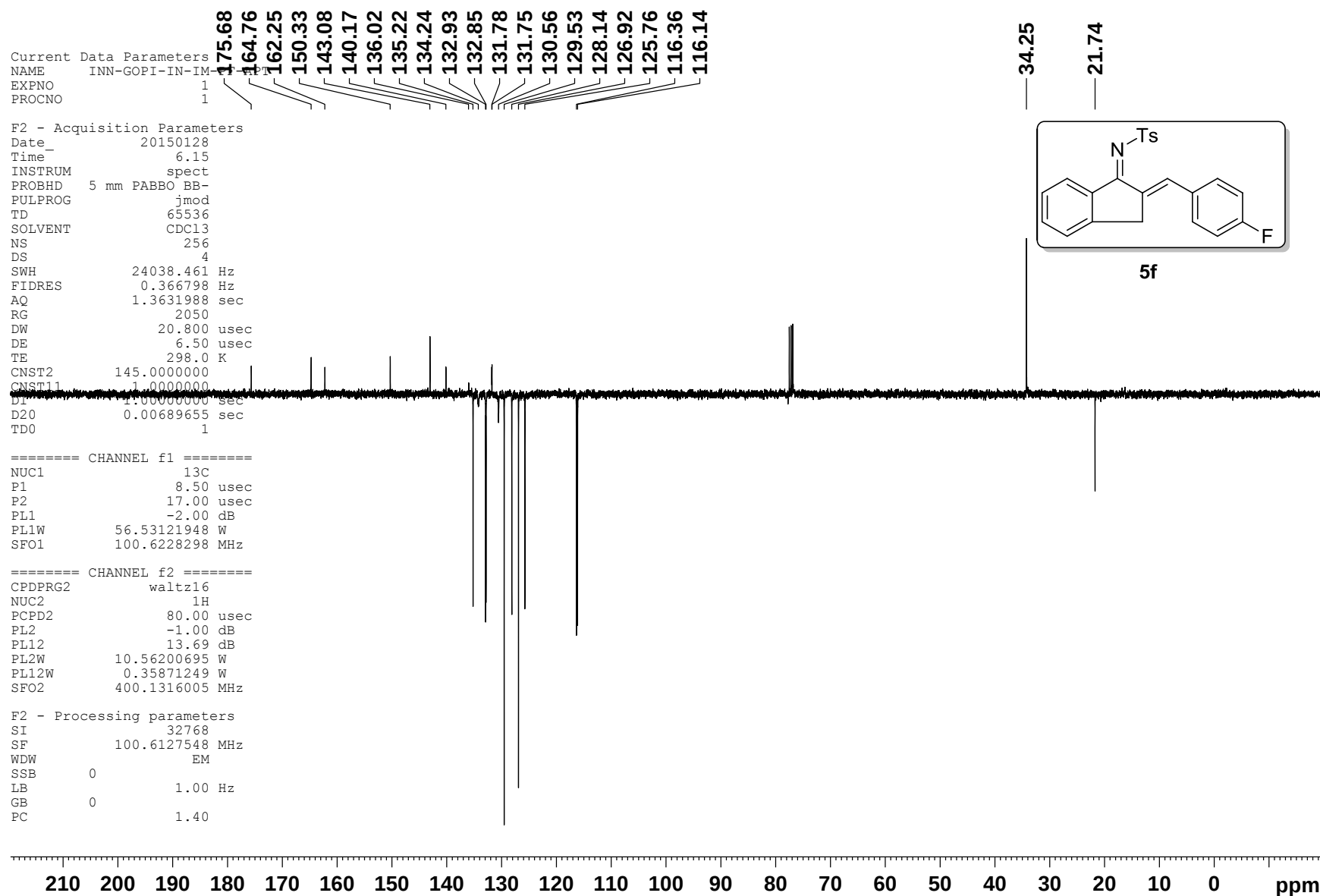
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Time 20.15
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 19
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 53.37
DW 50.000 usec
DE 6.50 usec
TE 300.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**5f****Figure S25. ¹H NMR Spectrum of 5f**



Figure S27. ¹³C-APT NMR Spectrum of 5f

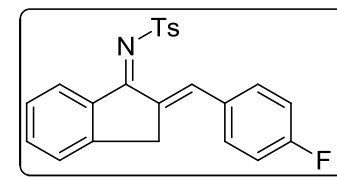
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EXPNO 4
PROCNO 1

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Time_ 8.22
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PULPROG zgfglqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 119.07
DW 4.400 usec
DE 6.50 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 470.5453180 MHz
NUC1 19F
P1 13.50 usec
PLW1 40.00000000 W

F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

— -109.22



5f

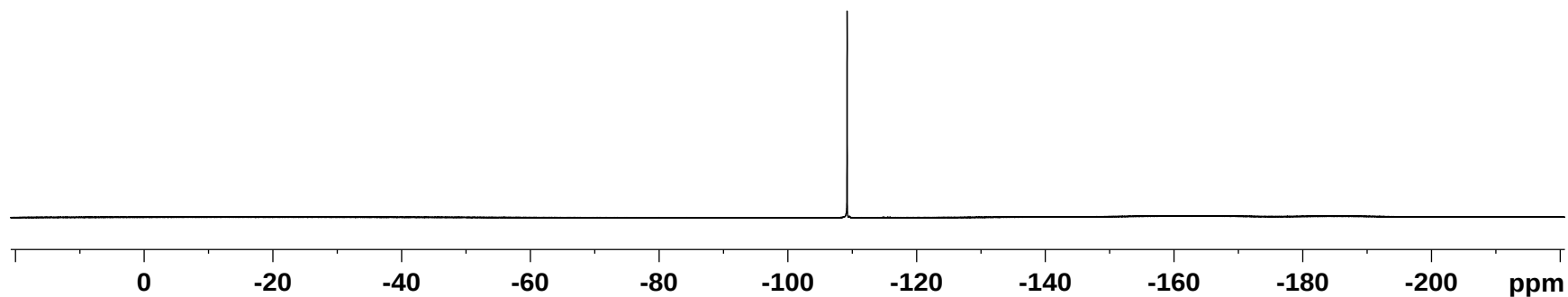


Figure S28. ¹⁹F NMR Spectrum of 5f

Current Data Parameters
 NAME INN-GOPI-TET-4-OMe-1H
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141116
 Time_ 14.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2999091 sec
 RG 144
 DW 60.800 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

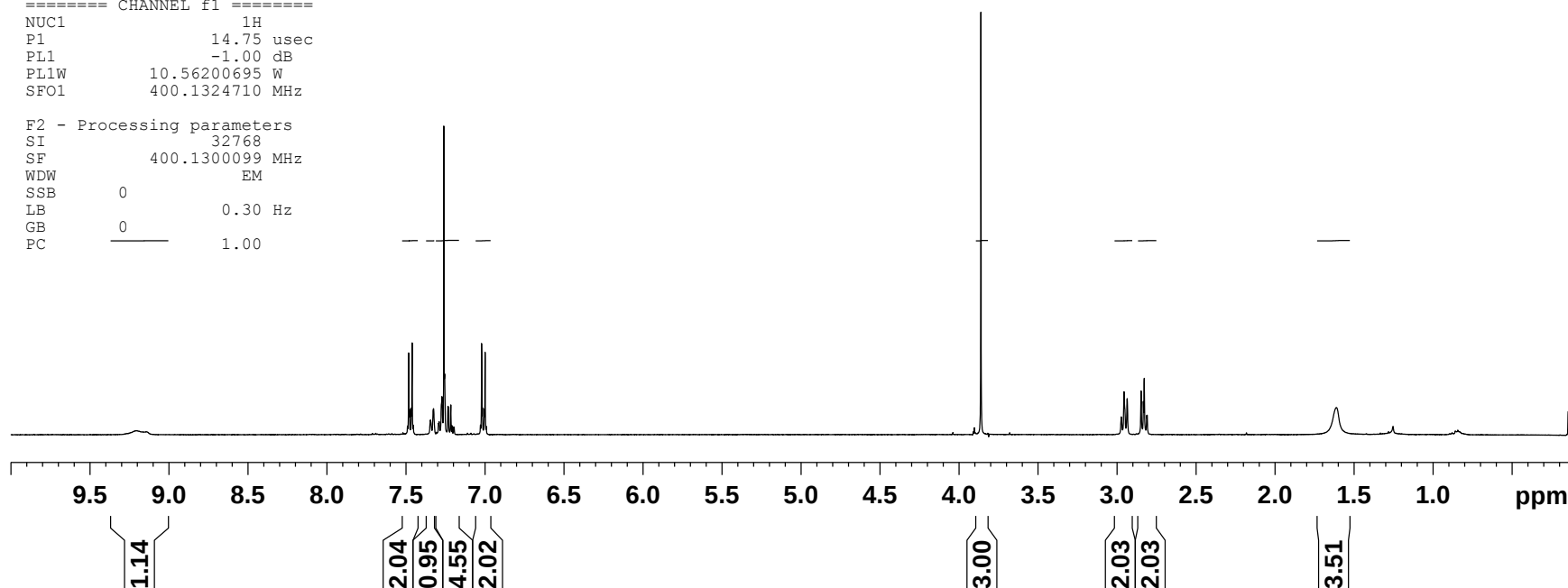
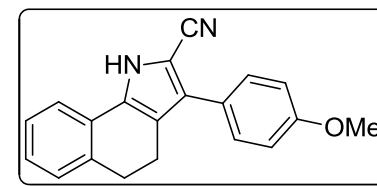
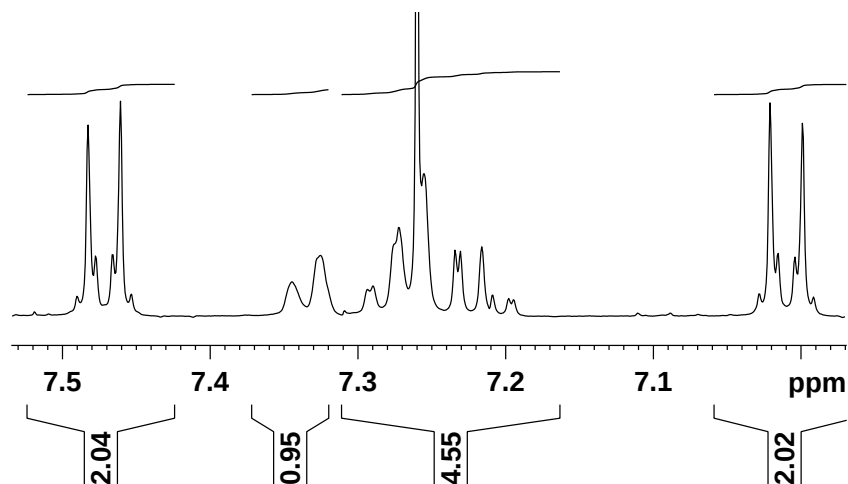
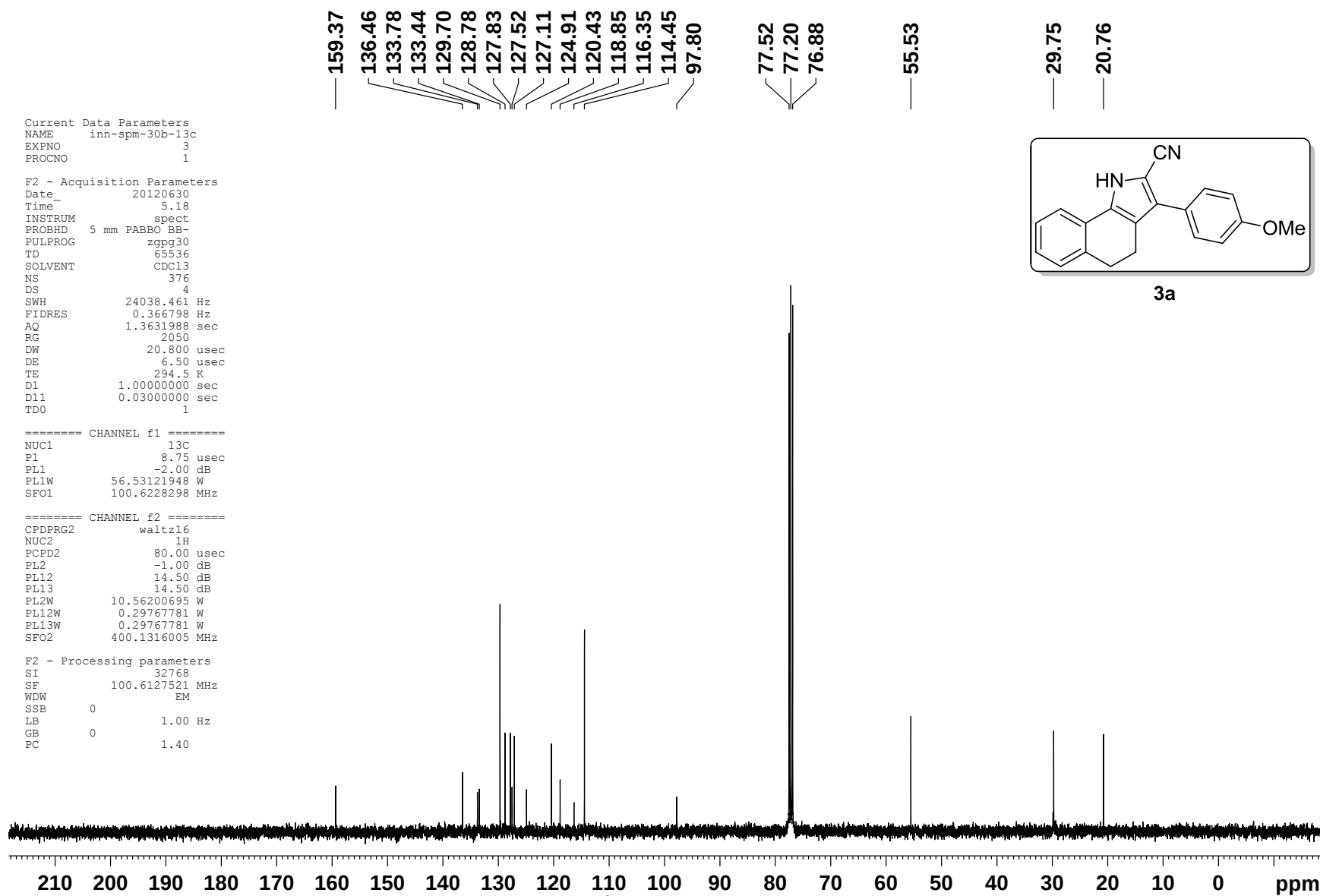
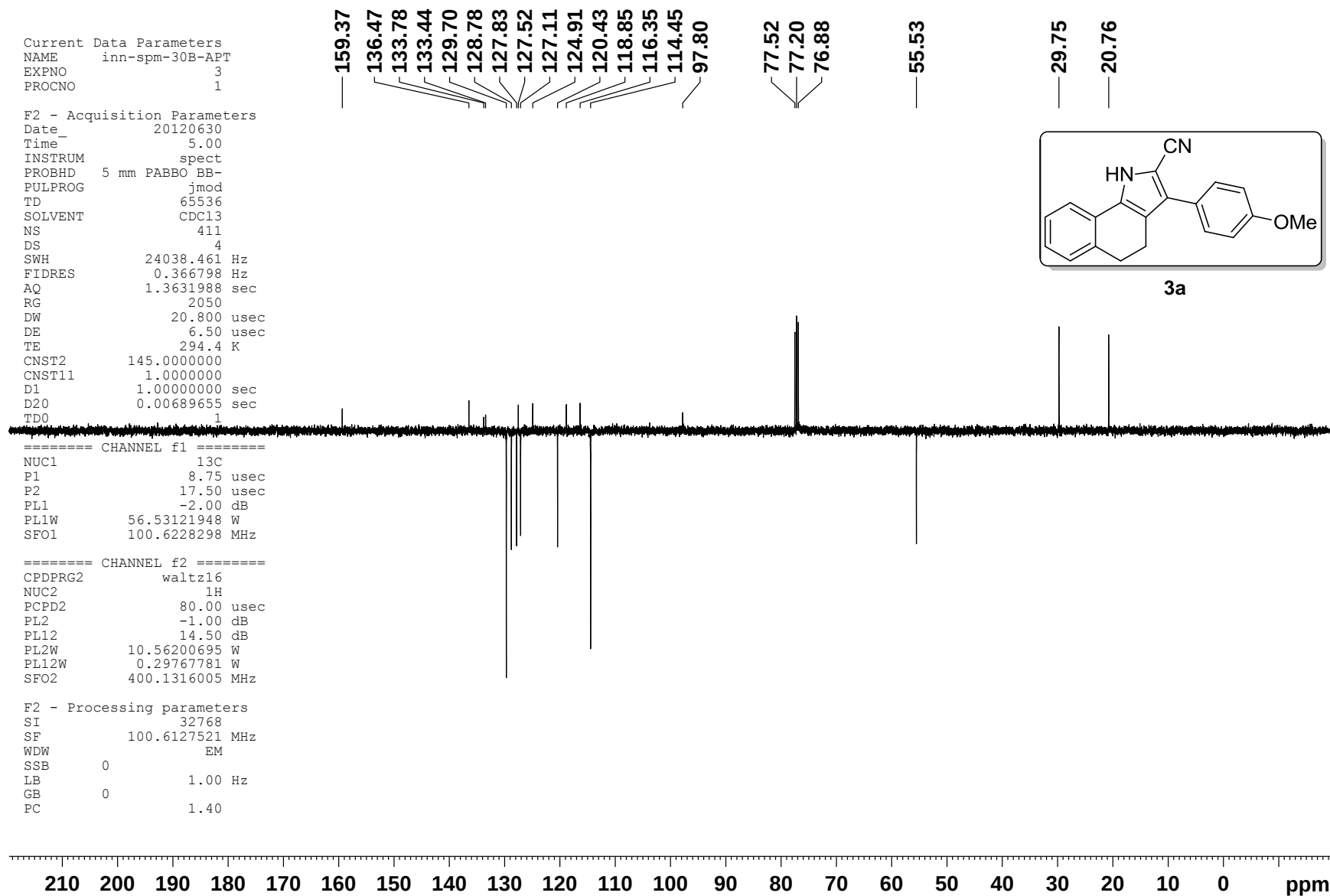
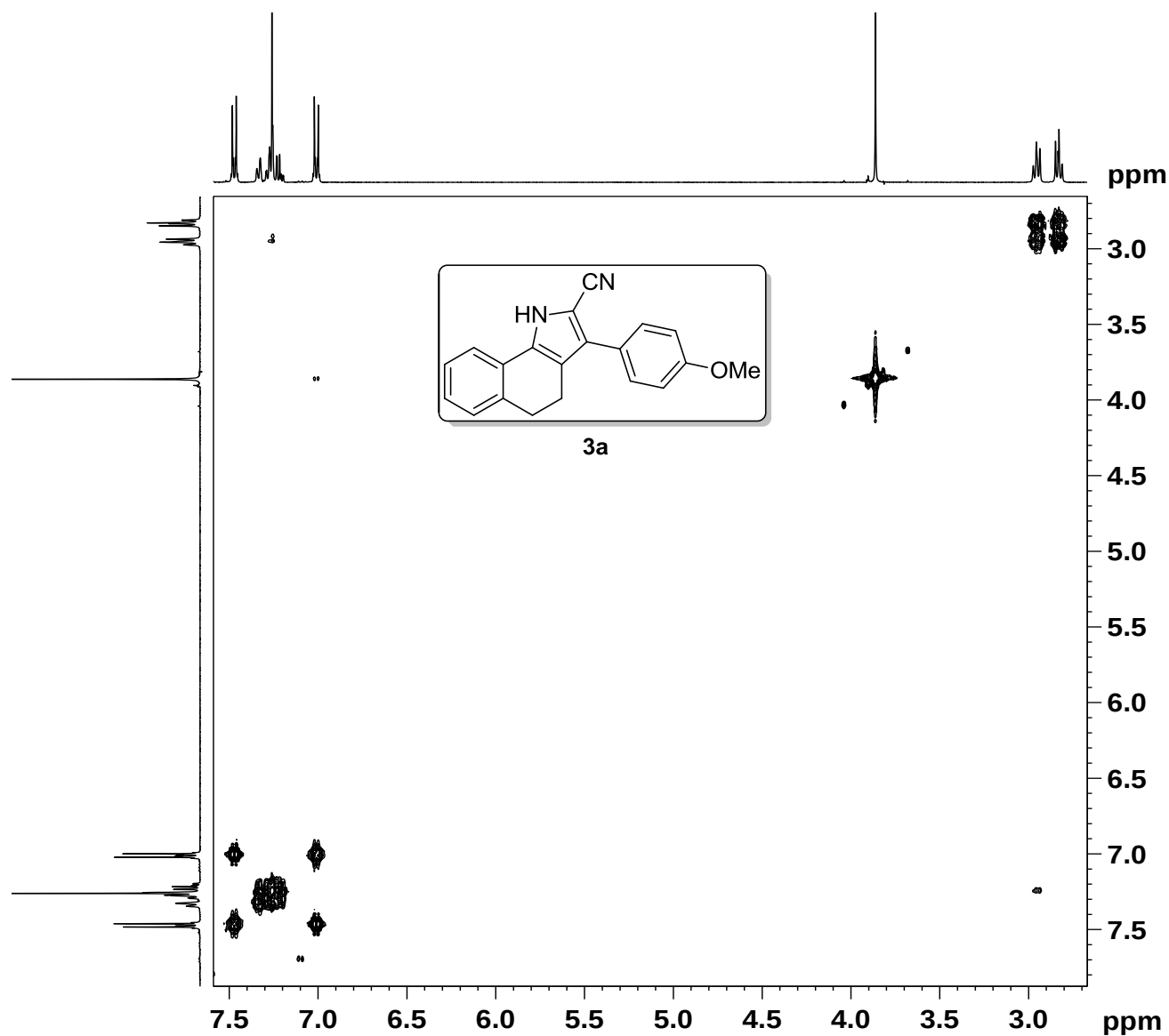


Figure S29. ^1H NMR Spectrum of 3a





Figure S32. ^1H - ^1H -COSY NMR Spectrum of 3a

Current Data Parameters
 NAME INN-GOPI-TET-4-OME-COSY
 EXPNO 23
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141116
 Time 19.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG cosygpgf
 TD 2048
 SOLVENT CDCl3
 NS 16
 DS 16
 SWH 3048.781 Hz
 FIDRES 1.488662 Hz
 AQ 0.3359220 sec
 RG 812
 DW 164.000 usec
 DE 6.50 usec
 TE 295.2 K
 D0 0.00000300 sec
 D1 1.00000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 INO 0.00032800 sec

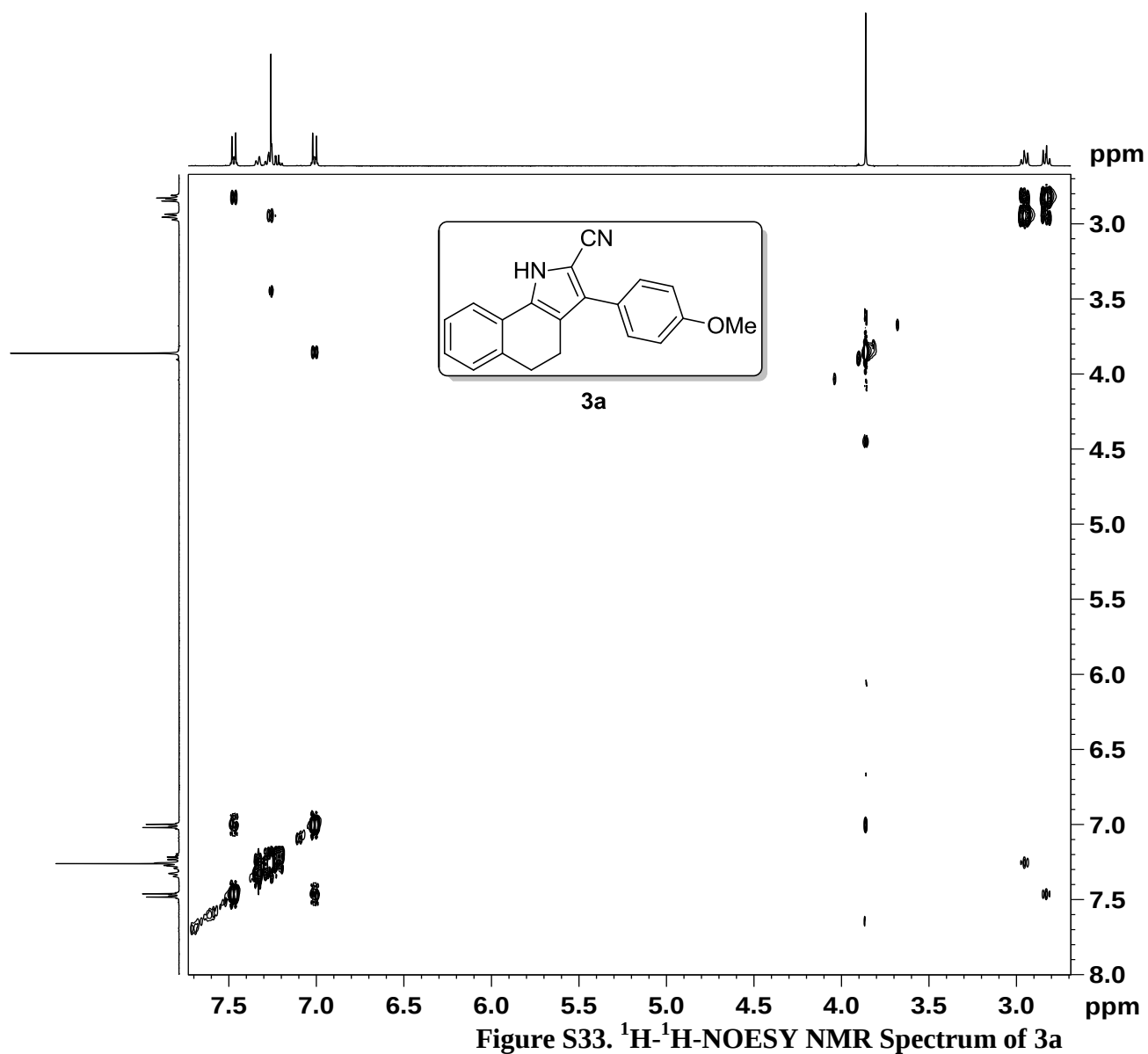
===== CHANNEL f1 =====
 NUC1 1H
 P0 14.75 usec
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324370 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SINE.100
 GPZ1 10.00 %
 P16 1000.00 usec

F1 - Acquisition parameters
 TD 256
 SFO1 400.1324 MHz
 FIDRES 11.909254 Hz
 SW 7.619 ppm
 FnmODE QF

F2 - Processing parameters
 SI 2048
 SF 400.1300099 MHz
 WDW SINE
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
 SI 512
 MC2 QF
 SF 400.1300099 MHz
 WDW GM
 SSB 0
 LB 0 Hz
 GB 0



Current Data Parameters
 NAME INN-GOPI-TET-4-OME-NOESY
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20141116
 Time_ 15.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG noesyph
 TD 2048
 SOLVENT CDCl3
 NS 32
 DS 16
 SWH 3048.781 Hz
 FIDRES 1.488662 Hz
 AQ 0.3359220 sec
 RG 128
 DW 164.000 usec
 DE 6.50 usec
 TE 296.2 K
 D0 0.00014522 sec
 D1 1.00000000 sec
 D8 0.60000002 sec
 IN0 0.00032800 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324370 MHz

F1 - Acquisition parameters
 TD 256
 SFO1 400.1324 MHz
 FIDRES 11.909254 Hz
 SW 7.619 ppm
 FnmODE States-TPPI

F2 - Processing parameters
 SI 2048
 SF 400.1300099 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 512
 MC2 States-TPPI
 SF 400.1300099 MHz
 WDW
 SSB 2
 LB 0 Hz
 GB 0

Current Data Parameters
NAME INN-GOPI-PIPERNAL-1H
EXPNO 4
PROCNO 1

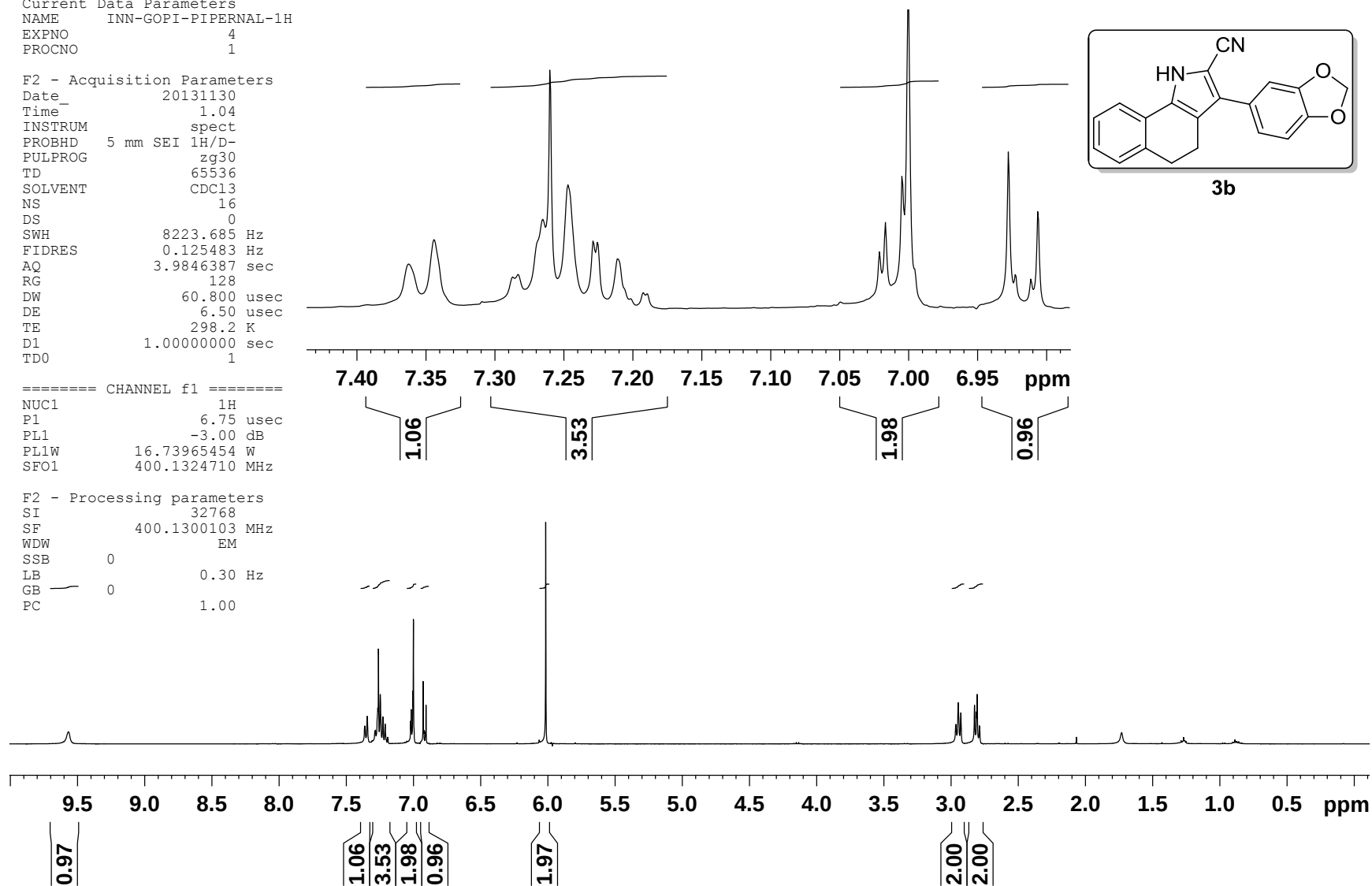
F2 - Acquisition Parameters

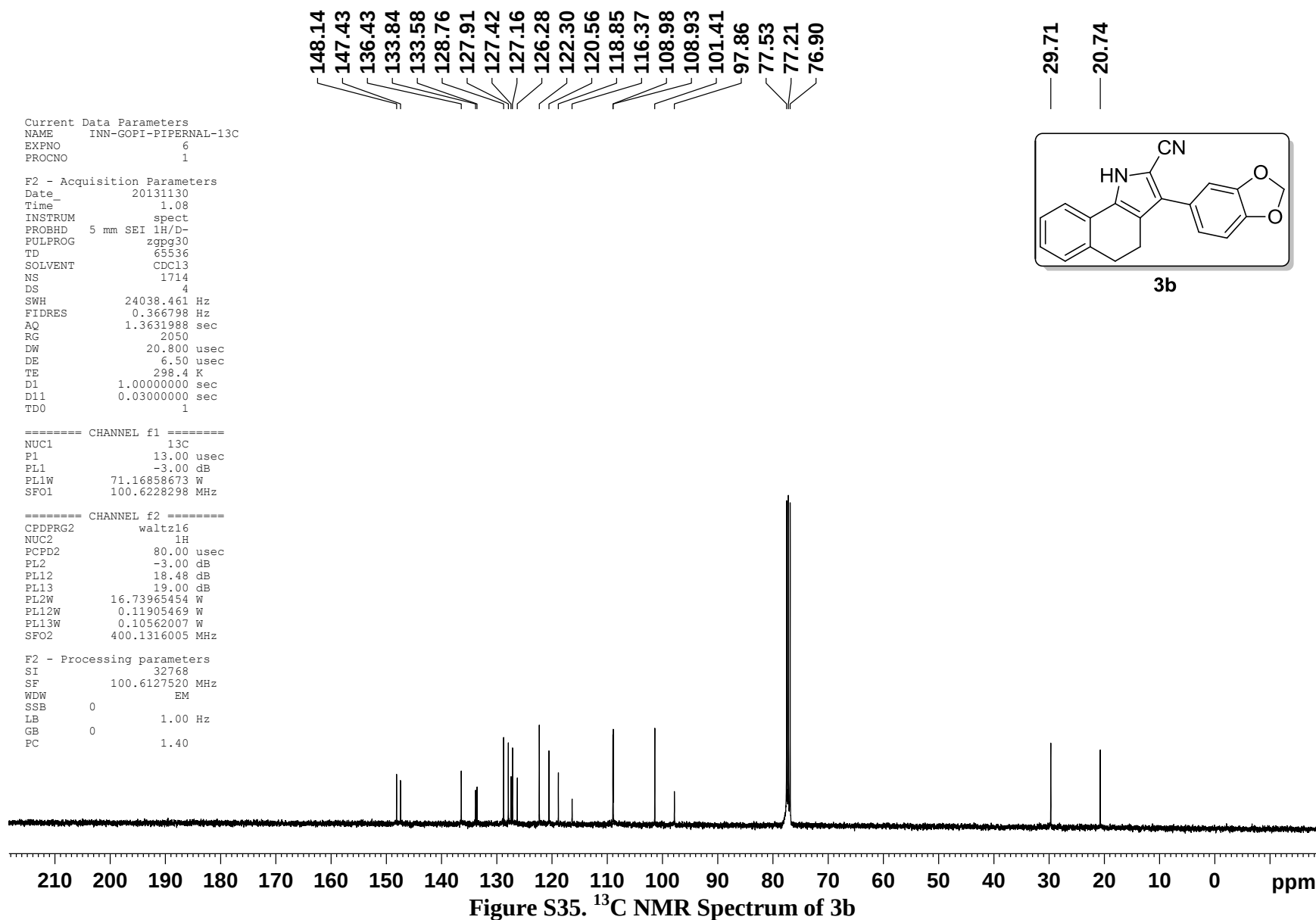
Date_ 20131130
Time 1.04
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.75 usec
PL1 -3.00 dB
PL1W 16.73965454 W
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S34. ¹H NMR Spectrum of 3b



Current Data Parameters
NAME IN-SPM-50B-1H
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120821
Time_ 10.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

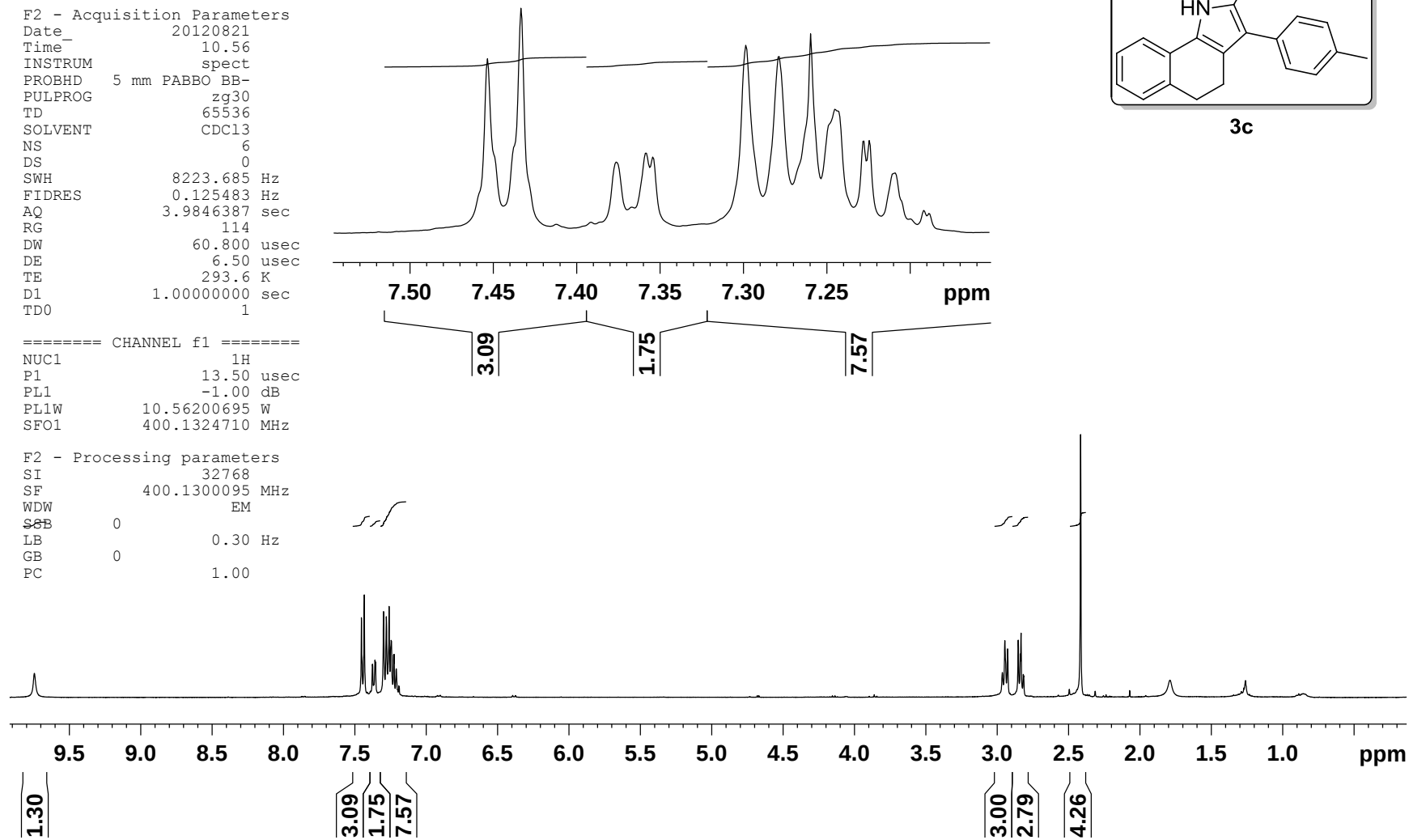
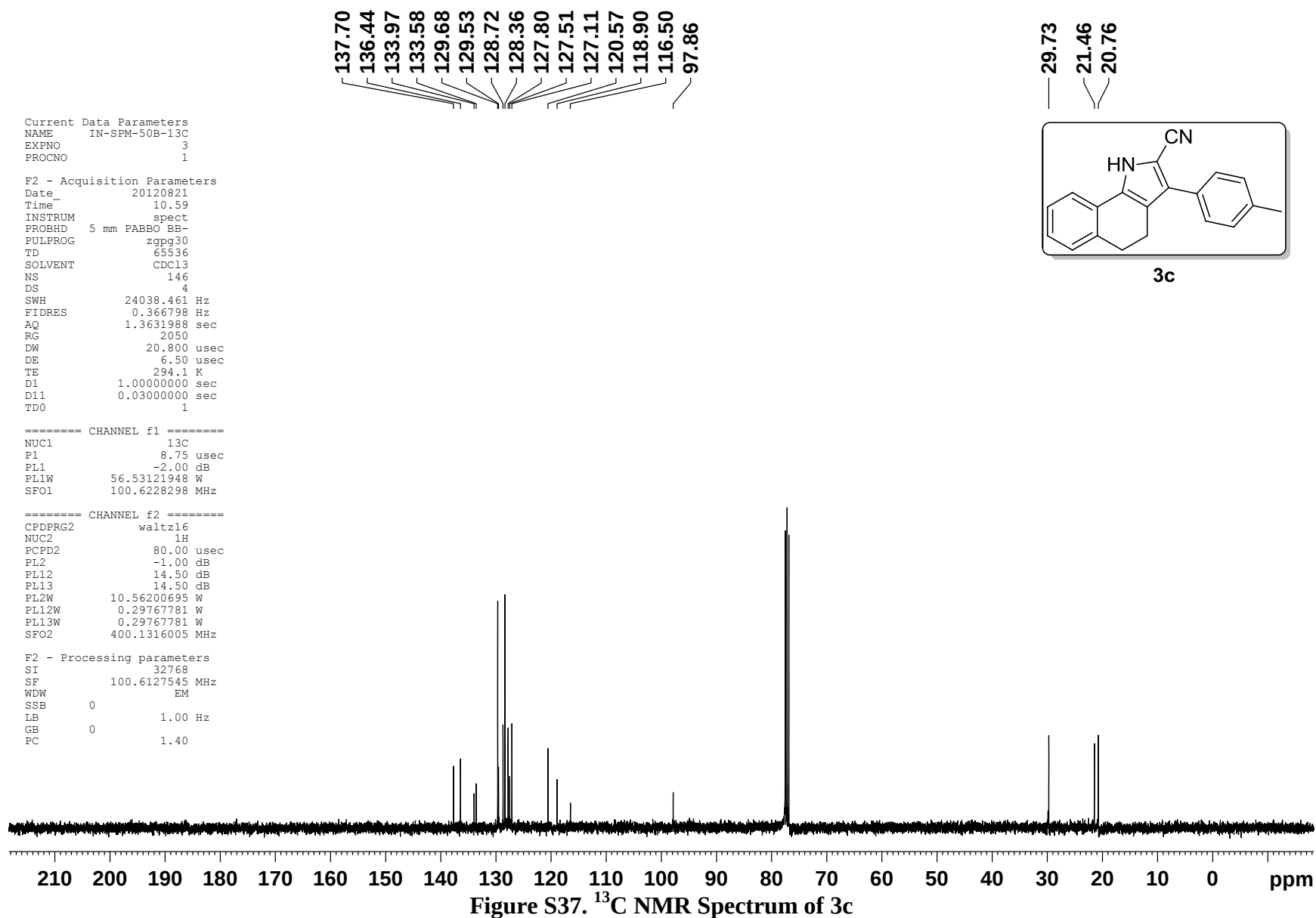


Figure S36. ^1H NMR Spectrum of 3c



Current Data Parameters
NAME INN-GOPI-PH-PYRRO-1H
EXPNO 4
PROCNO 1

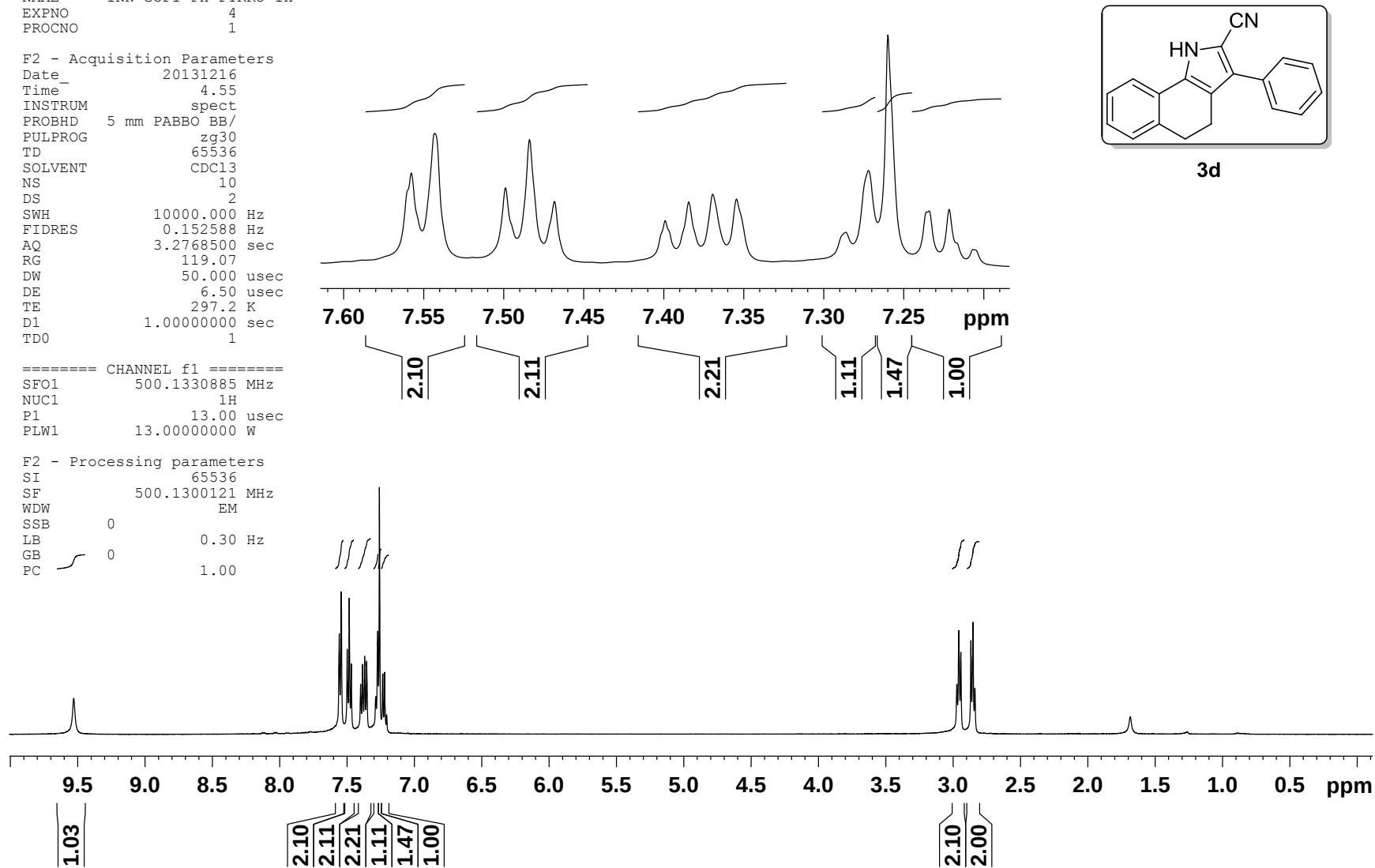
F2 - Acquisition Parameters

Date_ 20131216
Time 4.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 119.07
DW 50.000 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters

SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S38. ¹H NMR Spectrum of 3d

Current Data Parameters
 NAME INN-GOPI-PH-PYRF
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131218
 Time_ 19.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 120
 DS 4
 SWH 29761.904 H
 FIDRES 0.454131 H
 AQ 1.1010048 s
 RG 197.27
 DW 16.800 u
 DE 6.50 u
 TE 297.8 K
 D1 1.00000000 s
 D11 0.03000000 s
 TD0 1

===== CHANNEL f1 =====
 SFO1 125.7703637 M
 NUC1 13C
 P1 8.90 u
 PLW1 103.00000000 W

===== CHANNEL f2 =====
 SFO2 500.1320005 M
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 u
 PLW2 13.00000000 W
 PLW12 0.34327999 W
 PLW13 0.21969999 W

F2 - Processing parameters
 SI 32768
 SF 125.7577737 M
 WDW EM
 SSB 0
 LB 1.00 H
 GB 0
 PC 1.40

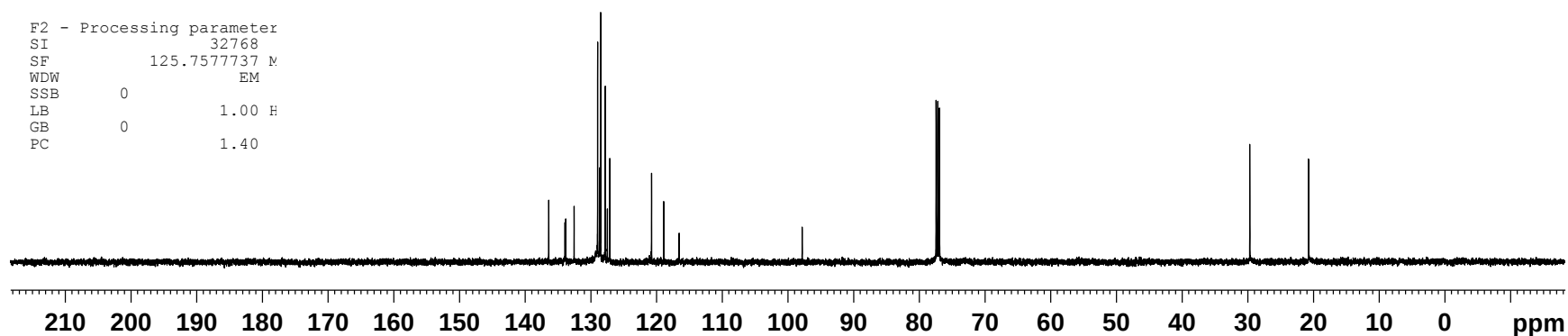
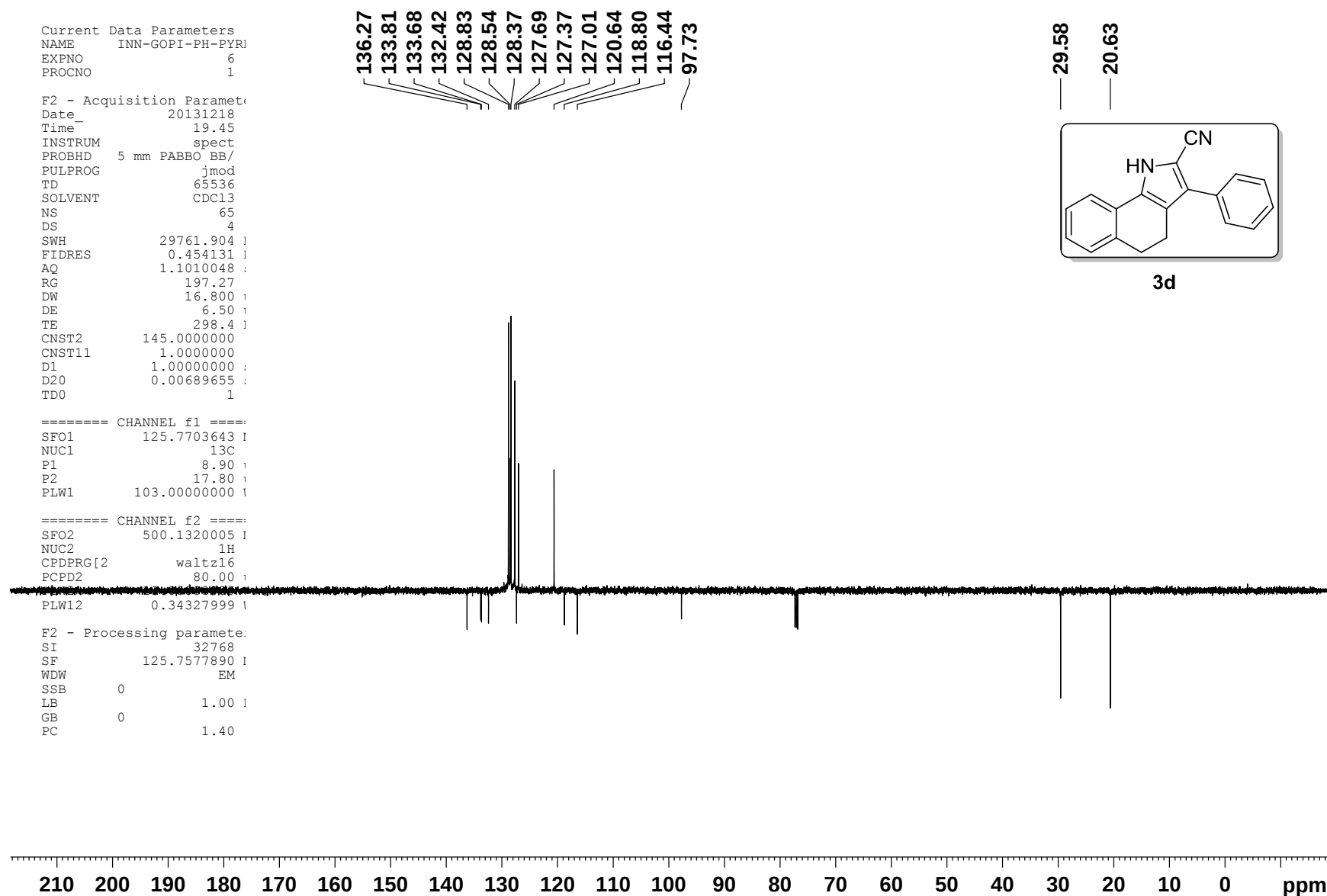


Figure S39. ¹³C NMR Spectrum of 3d

Figure S40. ¹³C-APT NMR Spectrum of 3d

Current Data Parameters
NAME INN-SPM-DDQ-1H
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

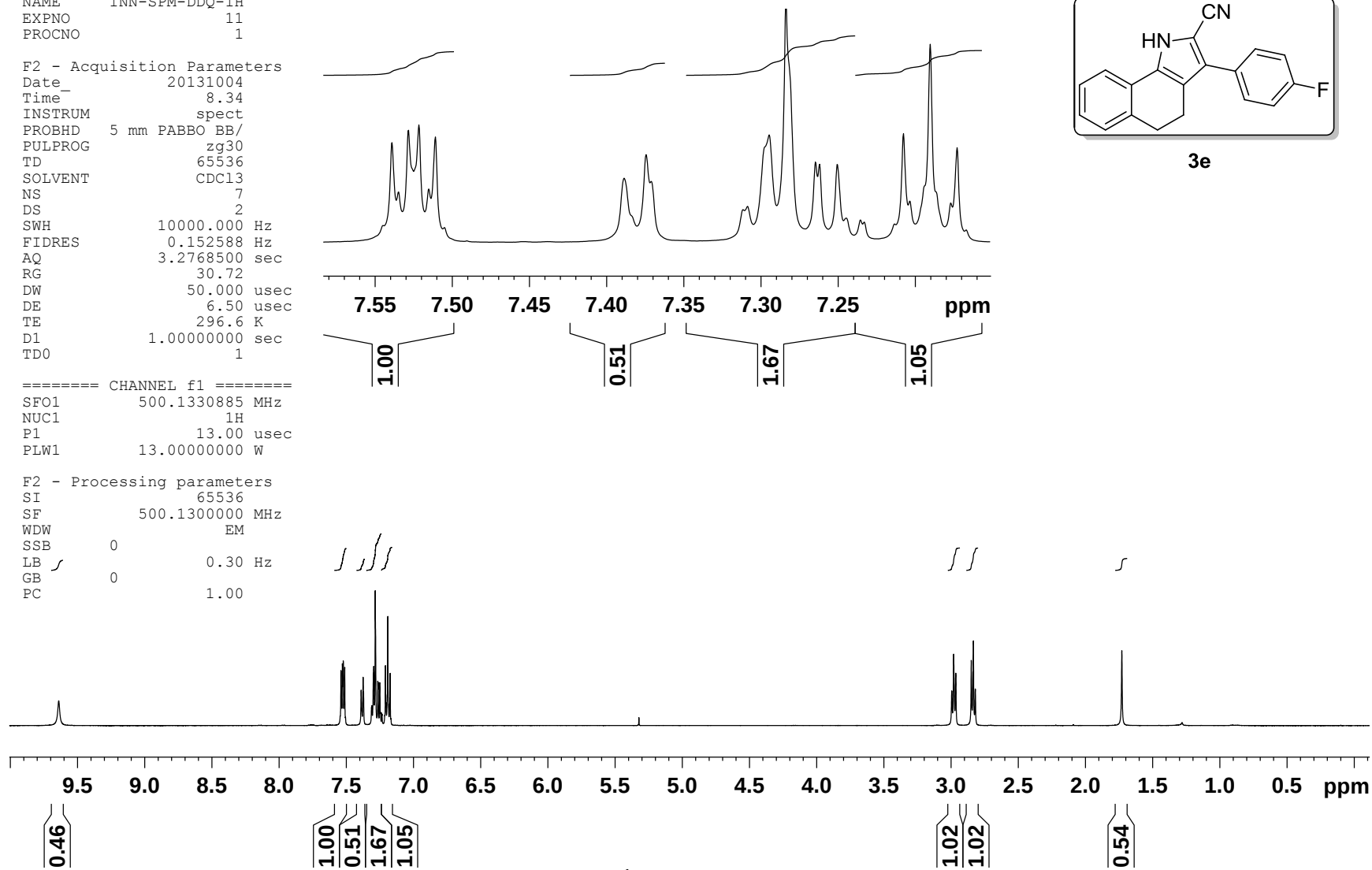
Date_ 20131004
Time_ 8.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

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

SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters

SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S41. ¹H NMR Spectrum of 3e

Current Data Parameters
 NAME INN-SPM-DDQ-13C
 EXPNO 11
 PROCNO 1

F2 - Acquisition Paramet
 Date_ 20131004
 Time_ 21.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 200
 DS 4
 SWH 29761.904
 FIDRES 0.454131
 AQ 1.1010048
 RG 197.27
 DW 16.800
 DE 6.50
 TE 296.9
 D1 1.00000000
 D11 0.03000000
 TD0 1

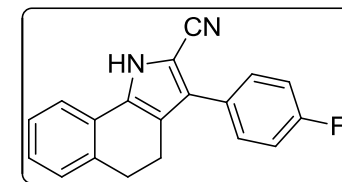
===== CHANNEL f1 =====
 SFO1 125.7703637
 NUC1 13C
 P1 8.90
 PLW1 103.0000000

===== CHANNEL f2 =====
 SFO2 500.1320005
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00
 PLW2 13.00000000
 PLW12 0.34327999
 PLW13 0.21969999

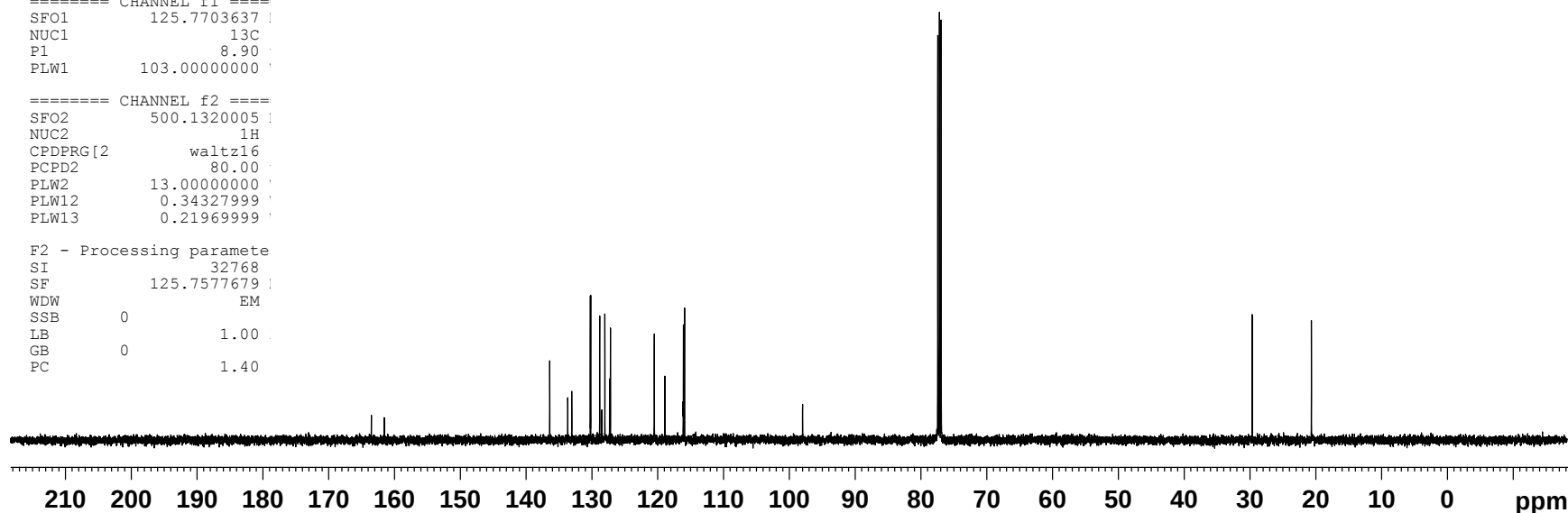
F2 - Processing paramete
 SI 32768
 SF 125.7577679
 WDW EM
 SSB 0
 LB 1.00
 GB 0
 PC 1.40

163.52
 161.55
 136.41
 133.67
 133.07
 130.25
 130.19
 128.82
 128.50
 128.03
 127.32
 127.19
 120.52
 118.93
 116.14
 116.10
 115.93
 98.00
 77.45
 77.20
 76.94

29.67
 20.67



3e

Figure S42. ¹³C NMR Spectrum of 3e

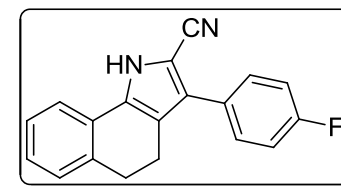
Current Data Parameters
NAME INN-GOPI-TET-PYRROLE-19F
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131211
Time_ 6.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 30
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 119.07
DW 4.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 470.5453180 MHz
NUC1 19F
P1 13.50 usec
PLW1 40.00000000 W

F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

— -113.96



3e

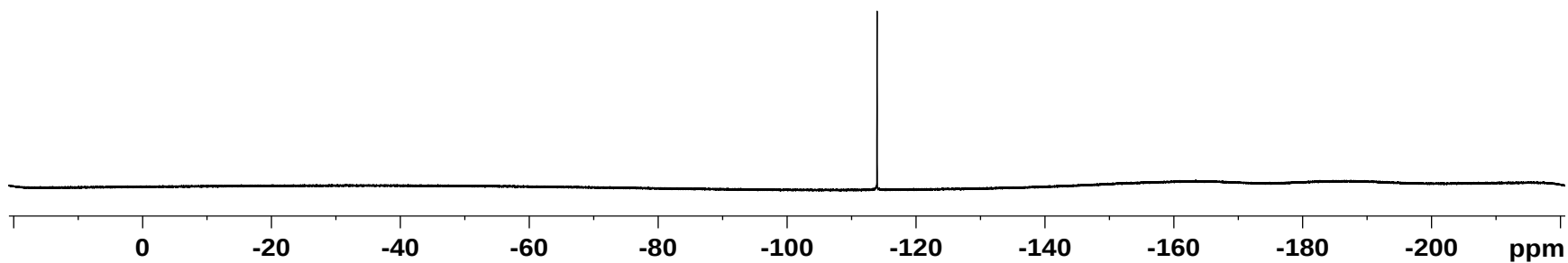


Figure S43. ¹⁹F NMR Spectrum of 3e

Current Data Parameters
NAME inn-spm-30a-1h
EXPNO 101
PROCNO 1

F2 - Acquisition Parameters

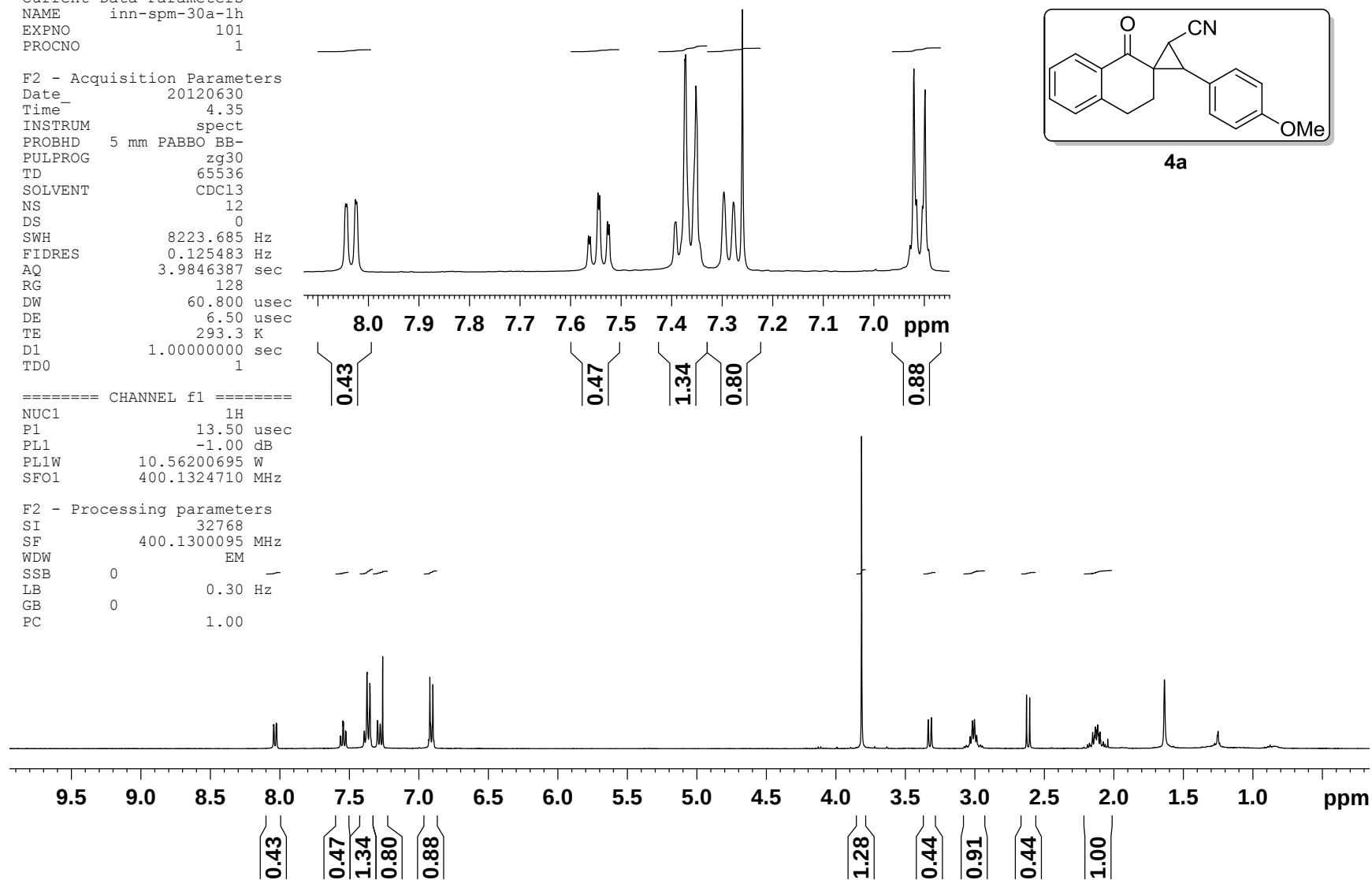
Date_ 20120630
Time_ 4.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1

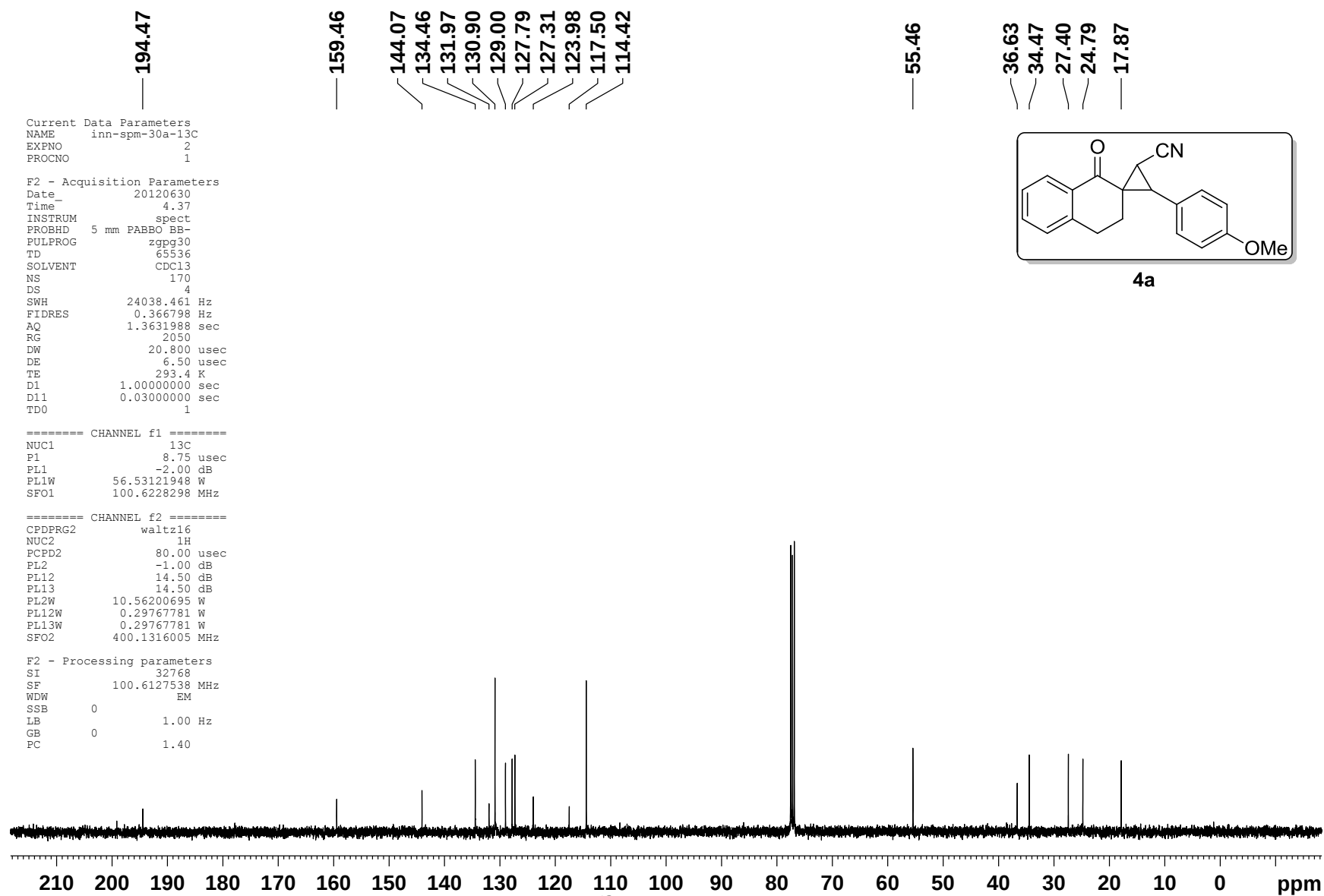
===== CHANNEL f1 =====

NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.130095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S44. ¹H NMR Spectrum of 4a

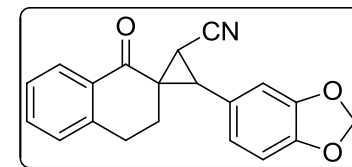
Figure S45. ¹³C NMR Spectrum of 4a

Current Data Parameters
 NAME INN-SPM-126-1-1H
 EXPNO 8
 PROCNO 1

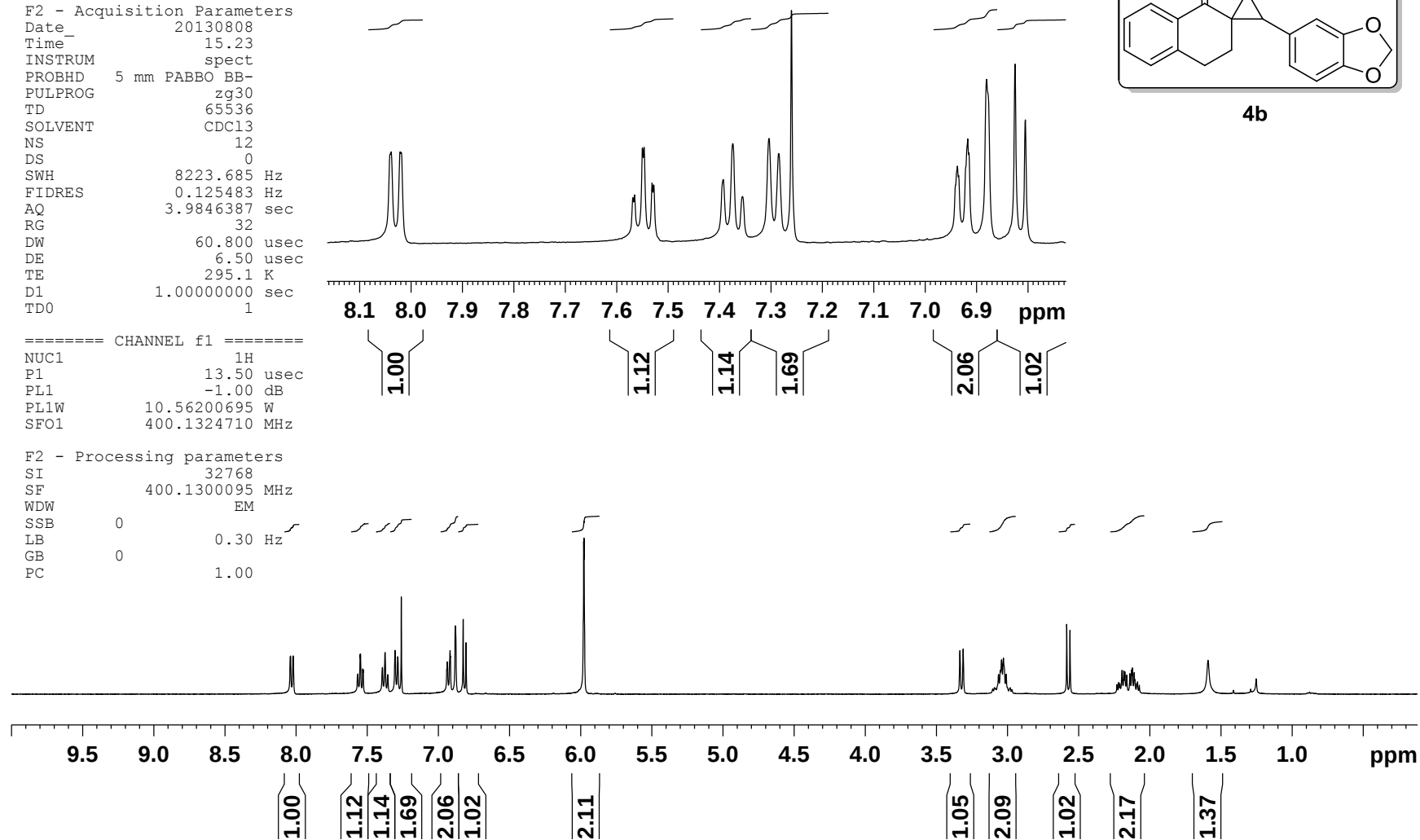
F2 - Acquisition Parameters
 Date_ 20130808
 Time_ 15.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 12
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 295.1 K
 D1 1.00000000 sec
 TDO 1

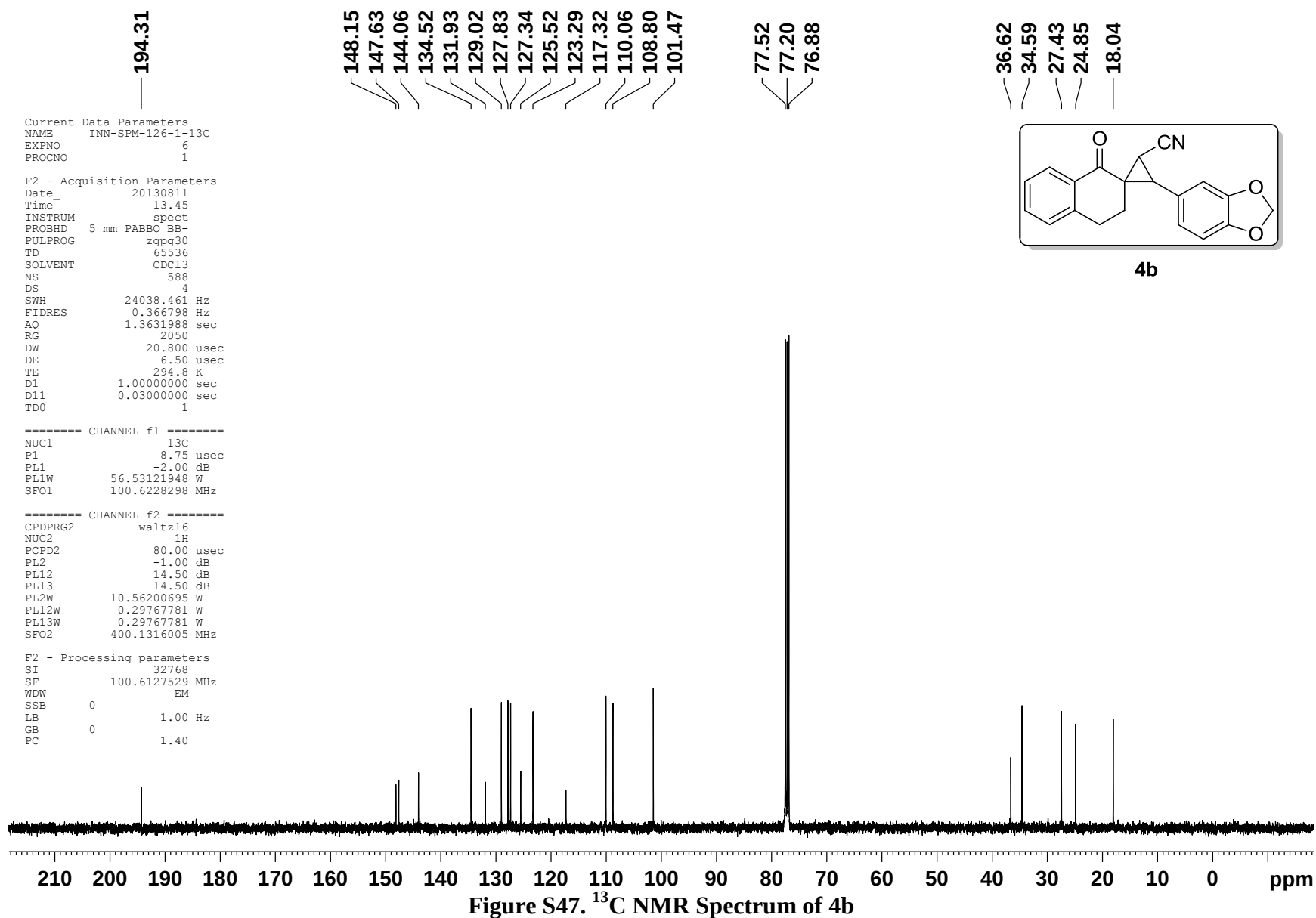
===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4b

Figure S46. ¹H NMR Spectrum of 4b

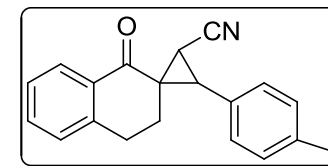
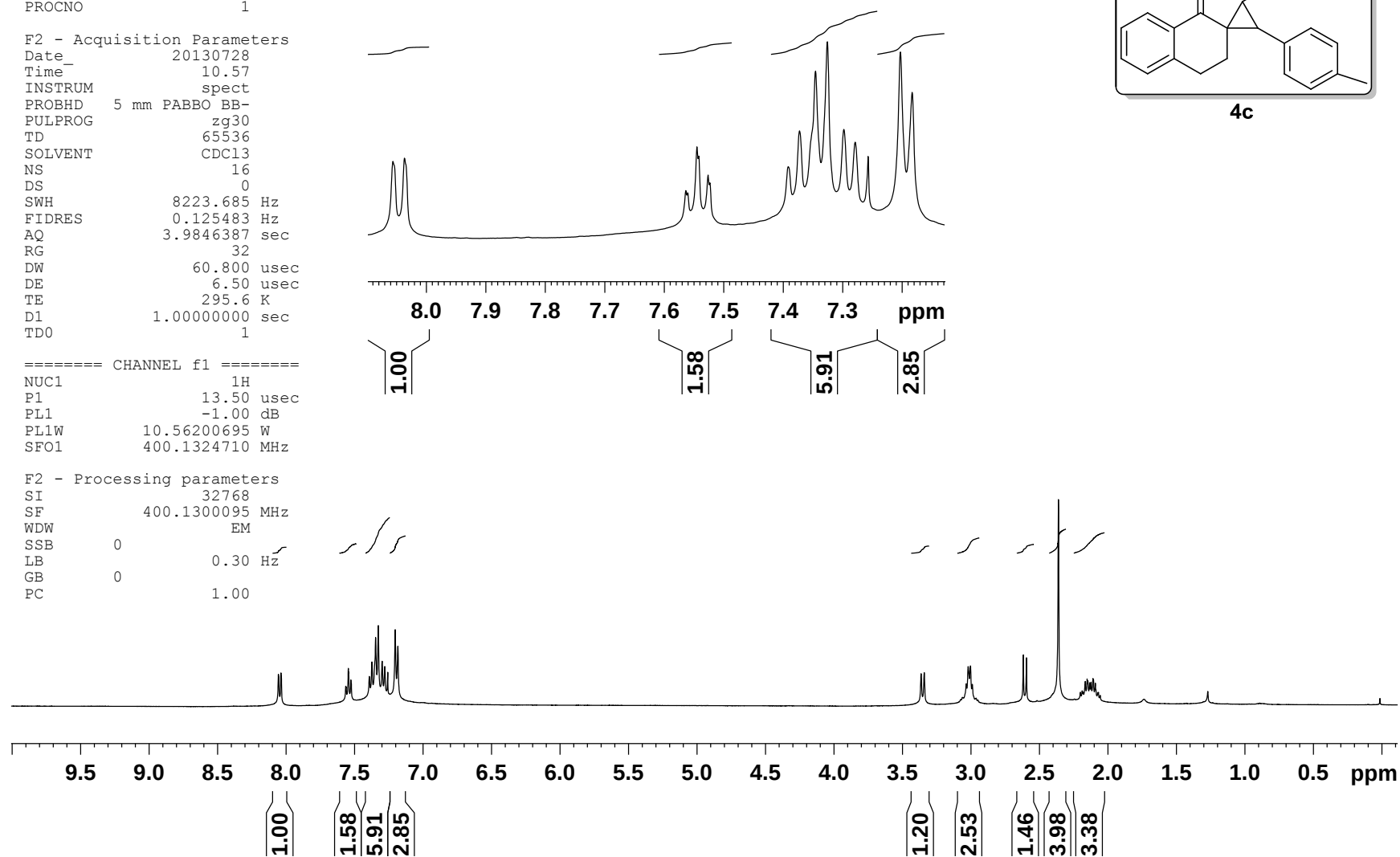


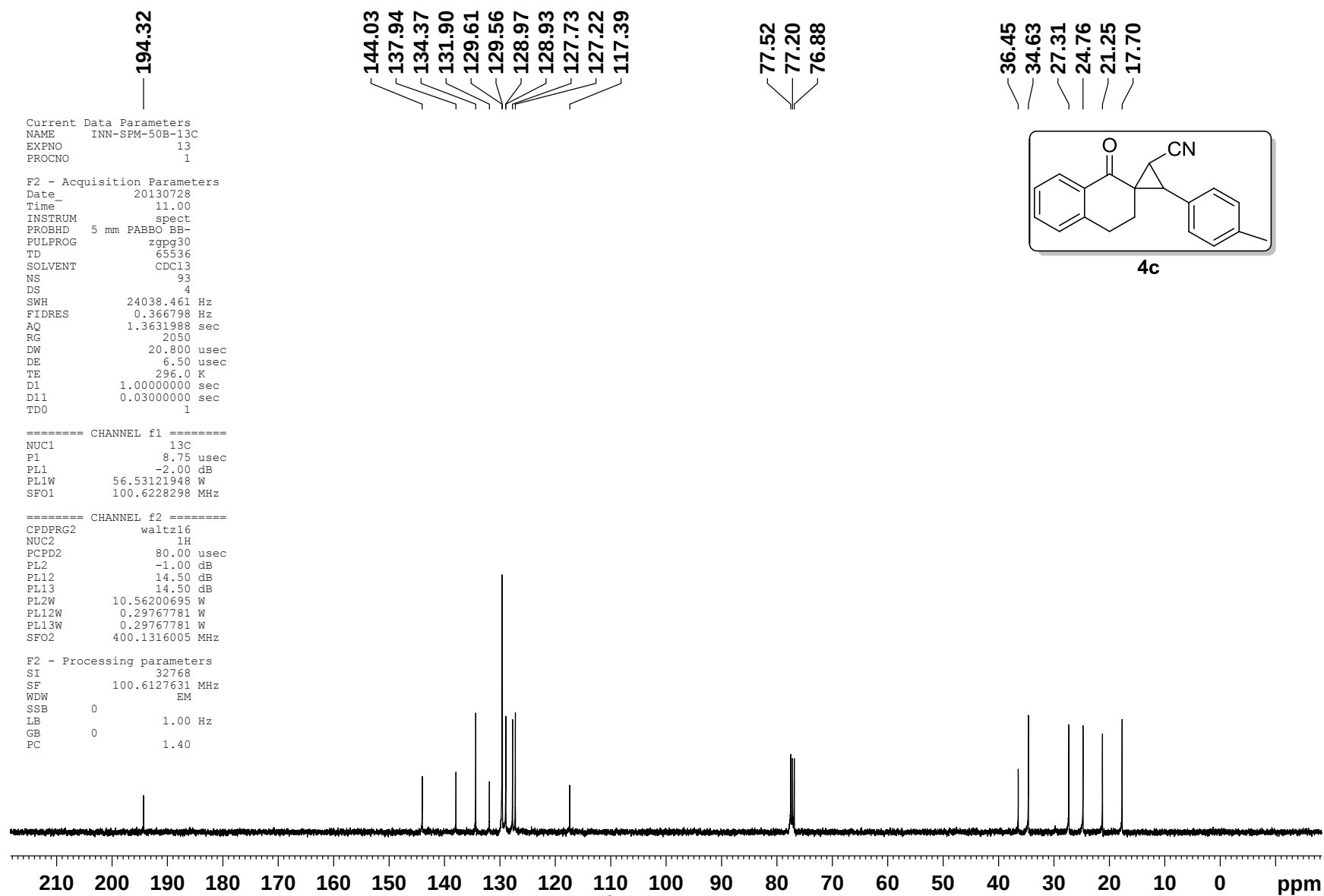
Current Data Parameters
NAME inn-spm-50b-1h
EXPNO 12
PROCNO 1

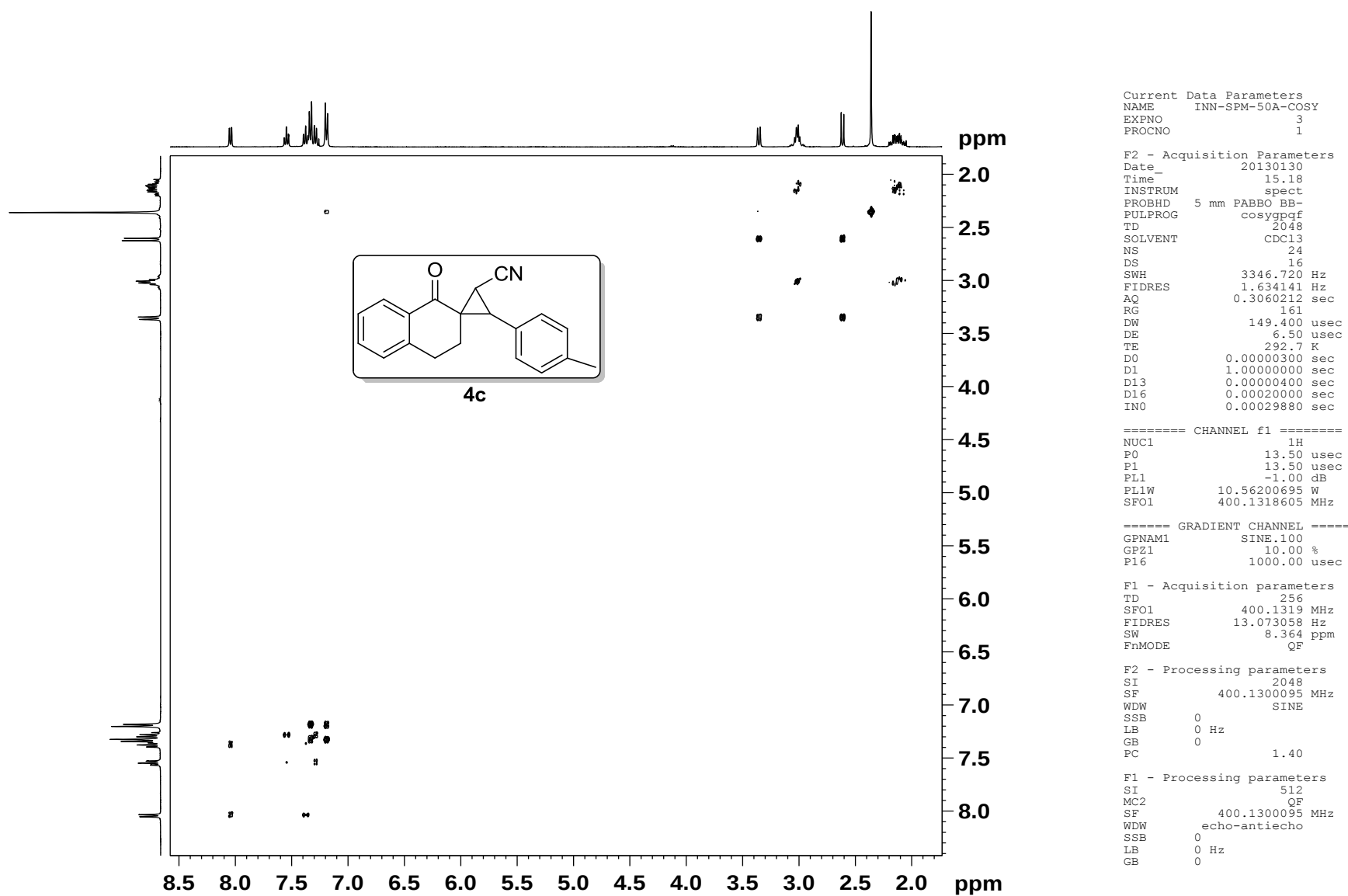
F2 - Acquisition Parameters
Date_ 20130728
Time_ 10.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

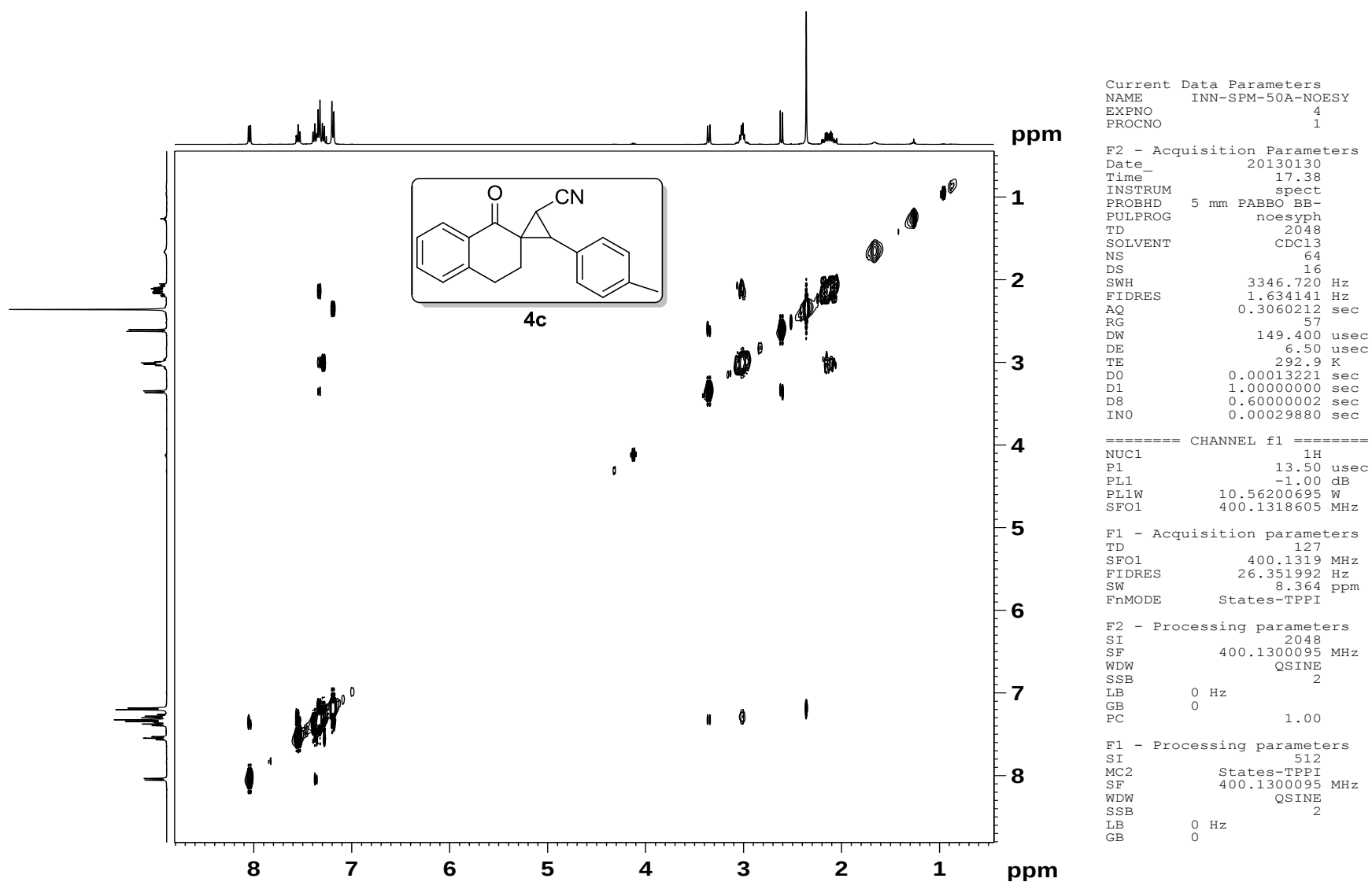
===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.130095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**4c****Figure S48. ¹H NMR Spectrum of 4c**

Figure S49. ¹³C NMR Spectrum of 4c

Figure S50. ^1H - ^1H -COSY NMR Spectrum of 4c

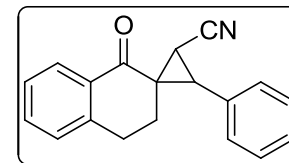
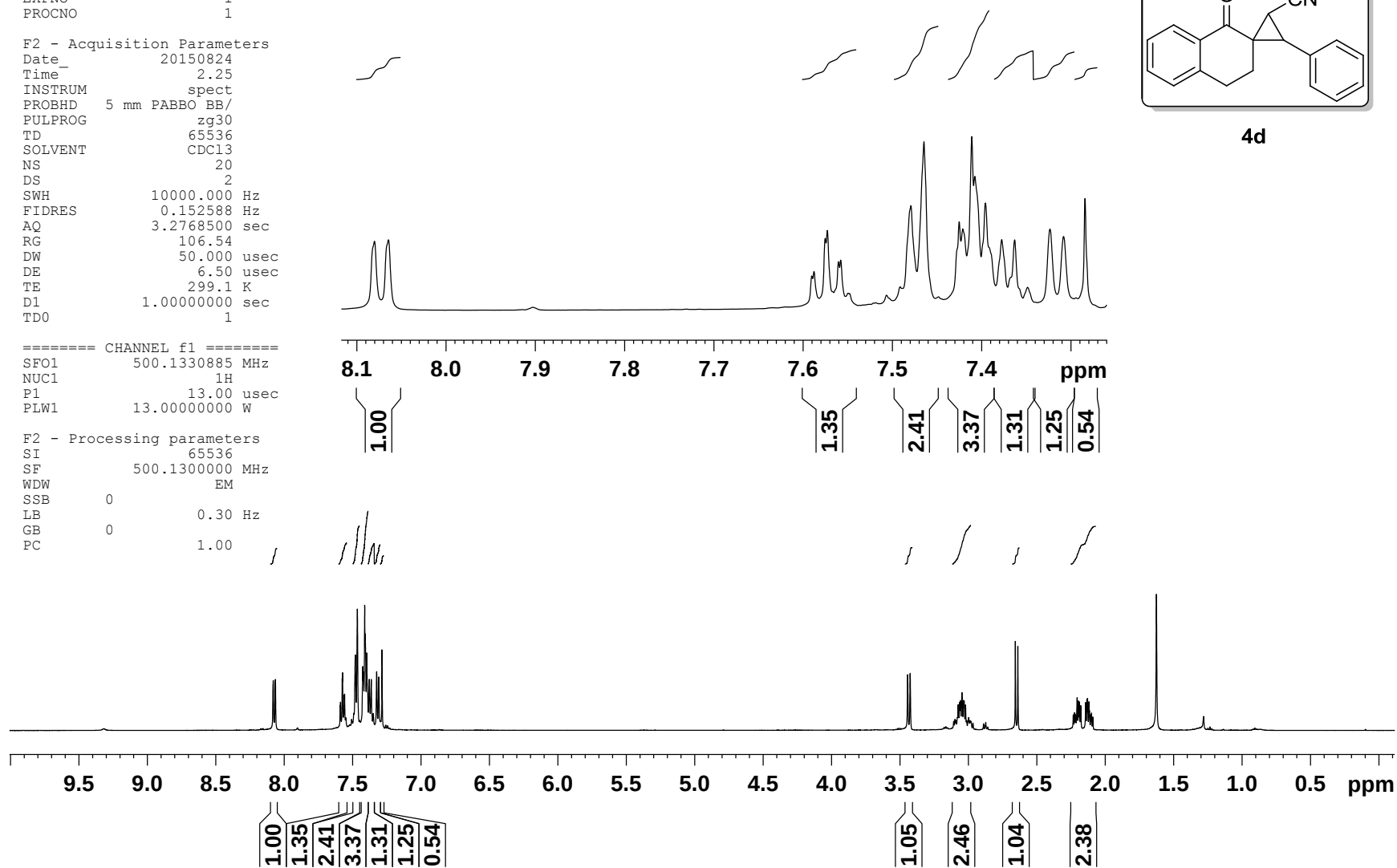
Figure S51. ^1H - ^1H -NOESY NMR Spectrum of 4c

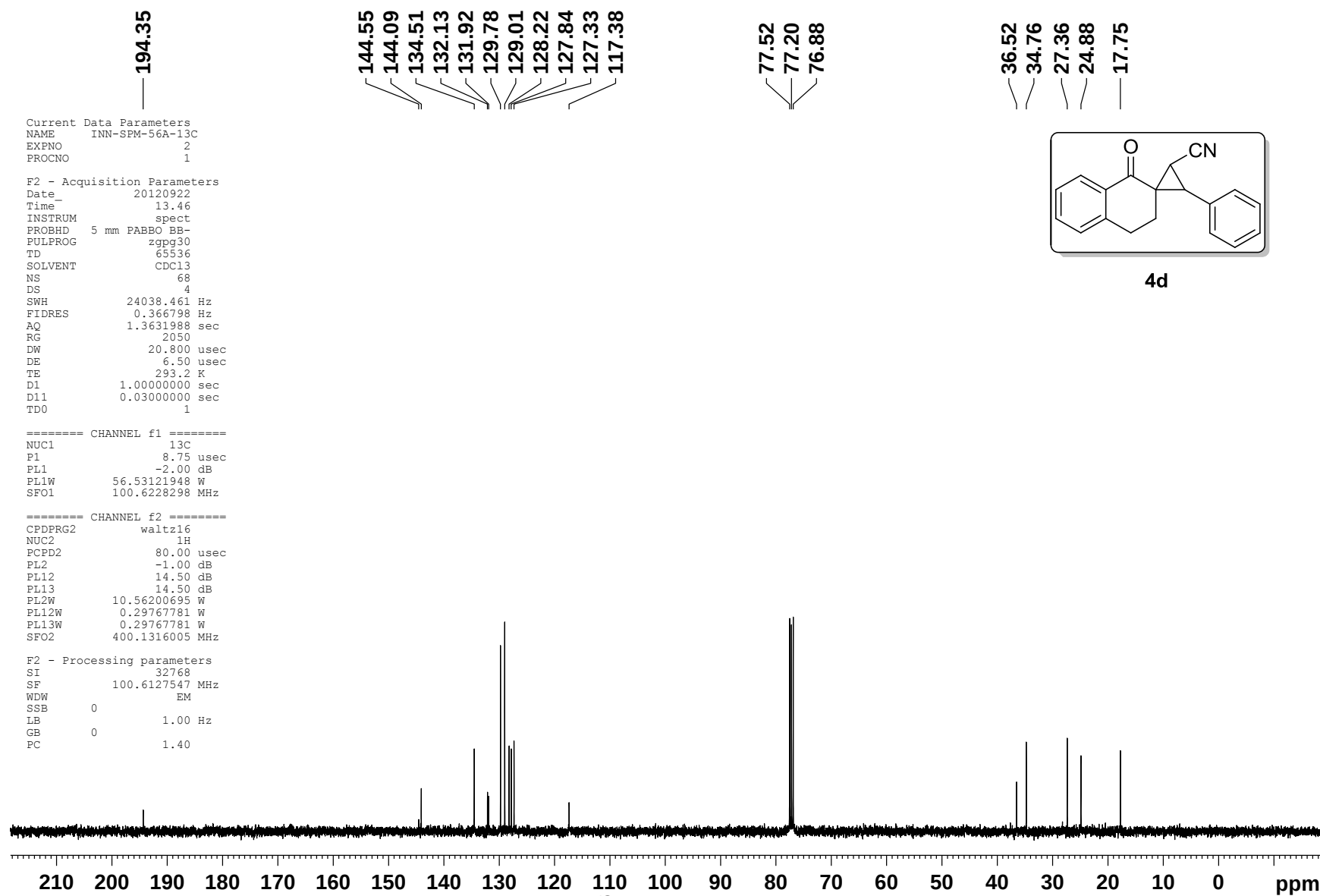
Current Data Parameters
NAME inn-gopi-tet-py-cy-1h
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150824
Time_ 2.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 20
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 106.54
DW 50.000 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**4d****Figure S52. ¹H NMR Spectrum of 4d**

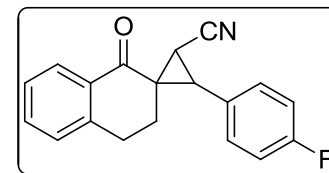
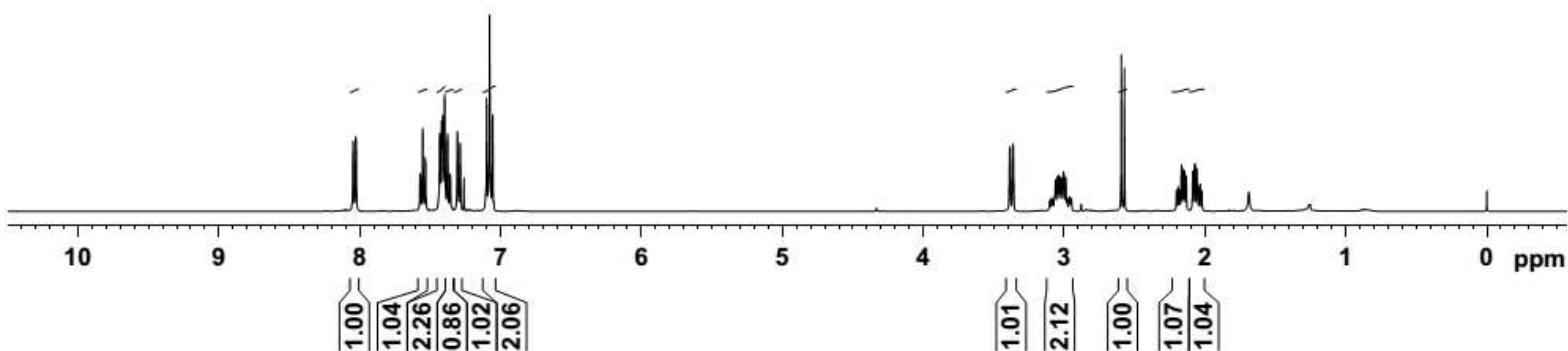
Figure S53. ¹³C NMR Spectrum of 4d

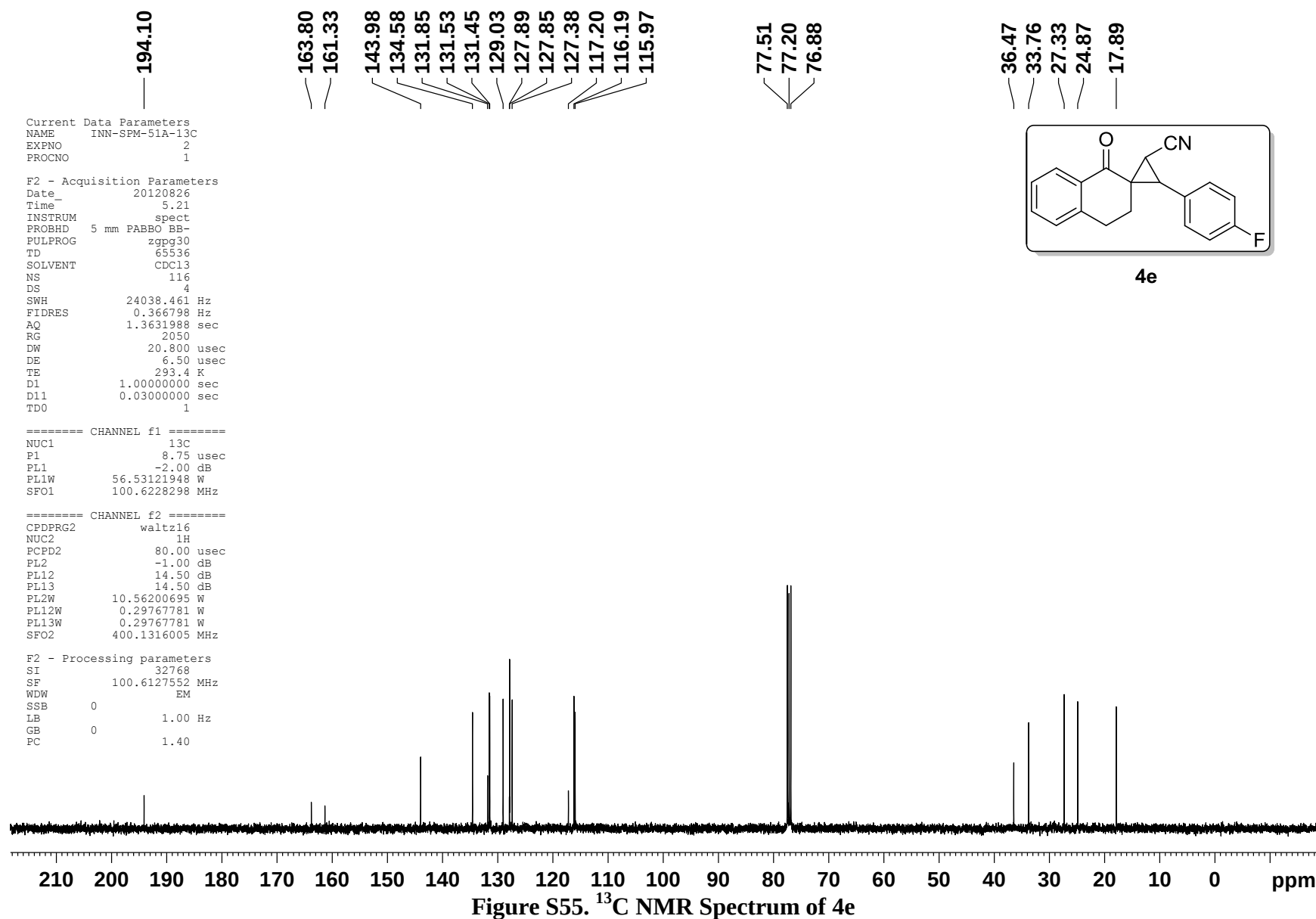
Current Data Parameters
NAME inn-gopi-tetra-4f-1h
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160218
Time_ 17.22
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 14
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 6.75 usec
PL1 -3.00 dB
PL1W 16.73965454 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**4e****Figure S54. ¹H NMR Spectrum of 4e**



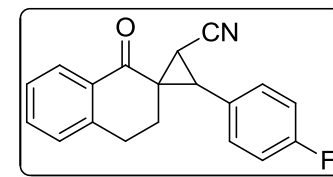
Current Data Parameters
NAME INN-GOPI-TET-CYCLO-19F
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131211
Time_ 6.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 32
DS 4
SWH 113636.367 Hz
FIDRES 0.866977 Hz
AQ 0.5767668 sec
RG 119.07
DW 4.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 470.5453180 MHz
NUC1 19F
P1 13.50 usec
PLW1 40.00000000 W

F2 - Processing parameters
SI 65536
SF 470.5923770 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

— -113.40



4e

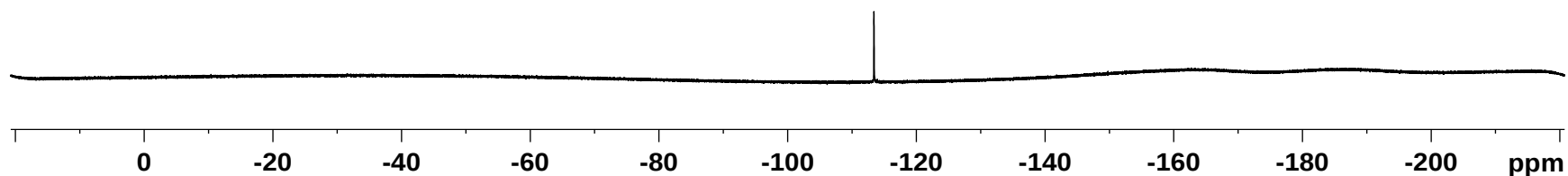


Figure S56. ¹⁹F NMR Spectrum of 4e

Current Data Parameters
NAME inn-gopi-qn-1h-1-ome-1h
EXPNO 6
PROCNO 1

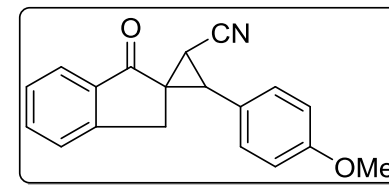
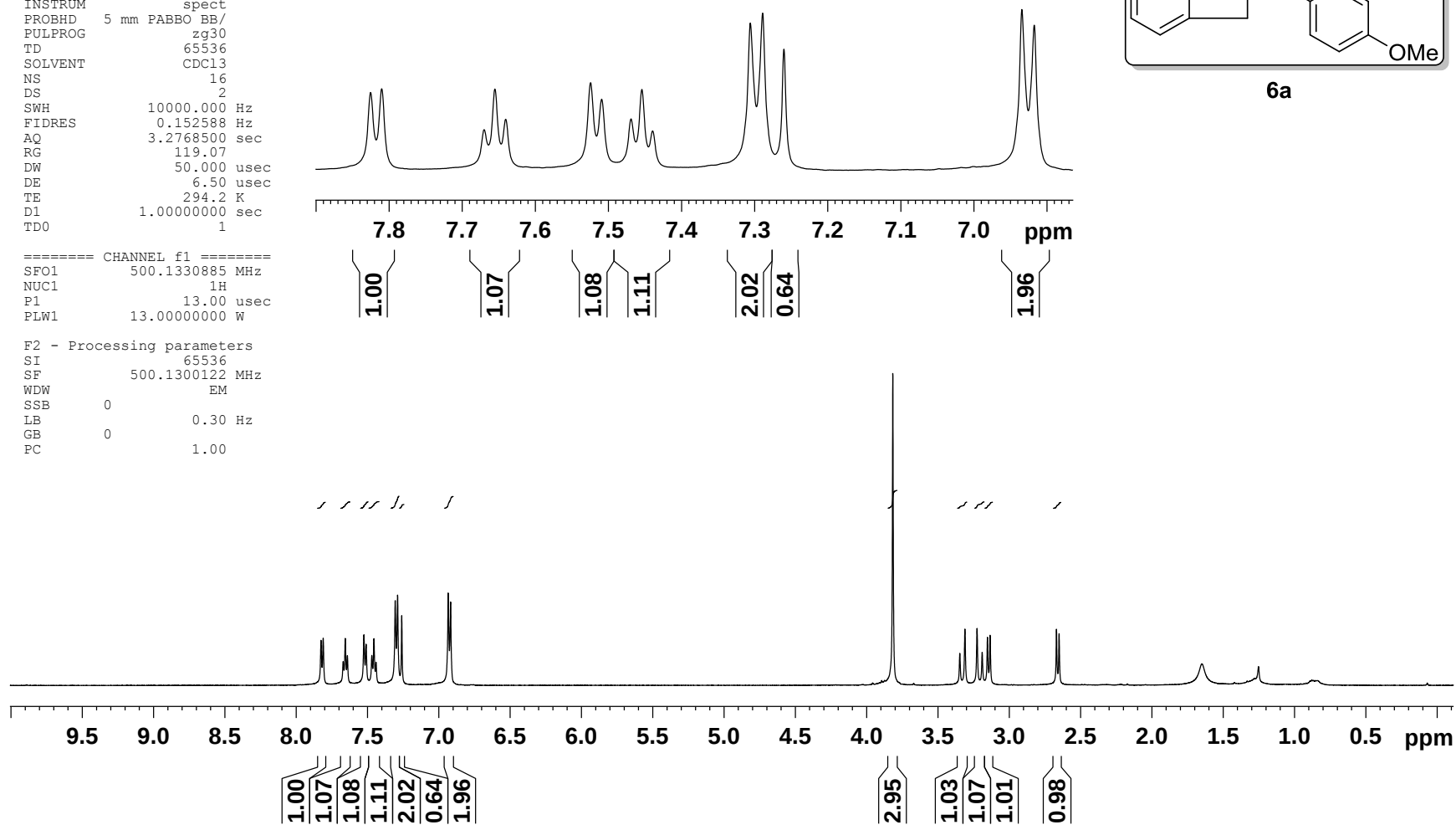
F2 - Acquisition Parameters

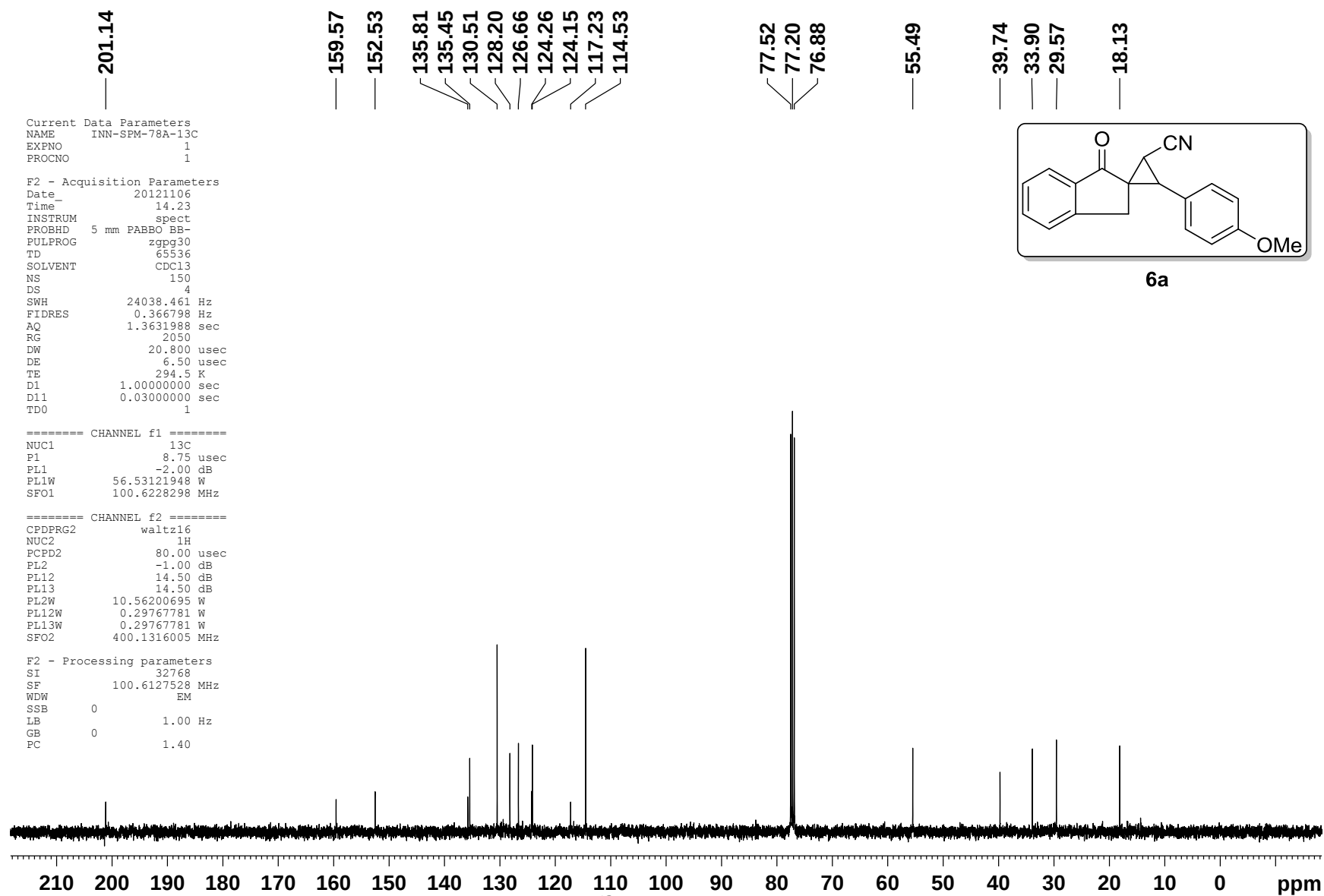
Date_ 20150311
Time 4.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 119.07
DW 50.000 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

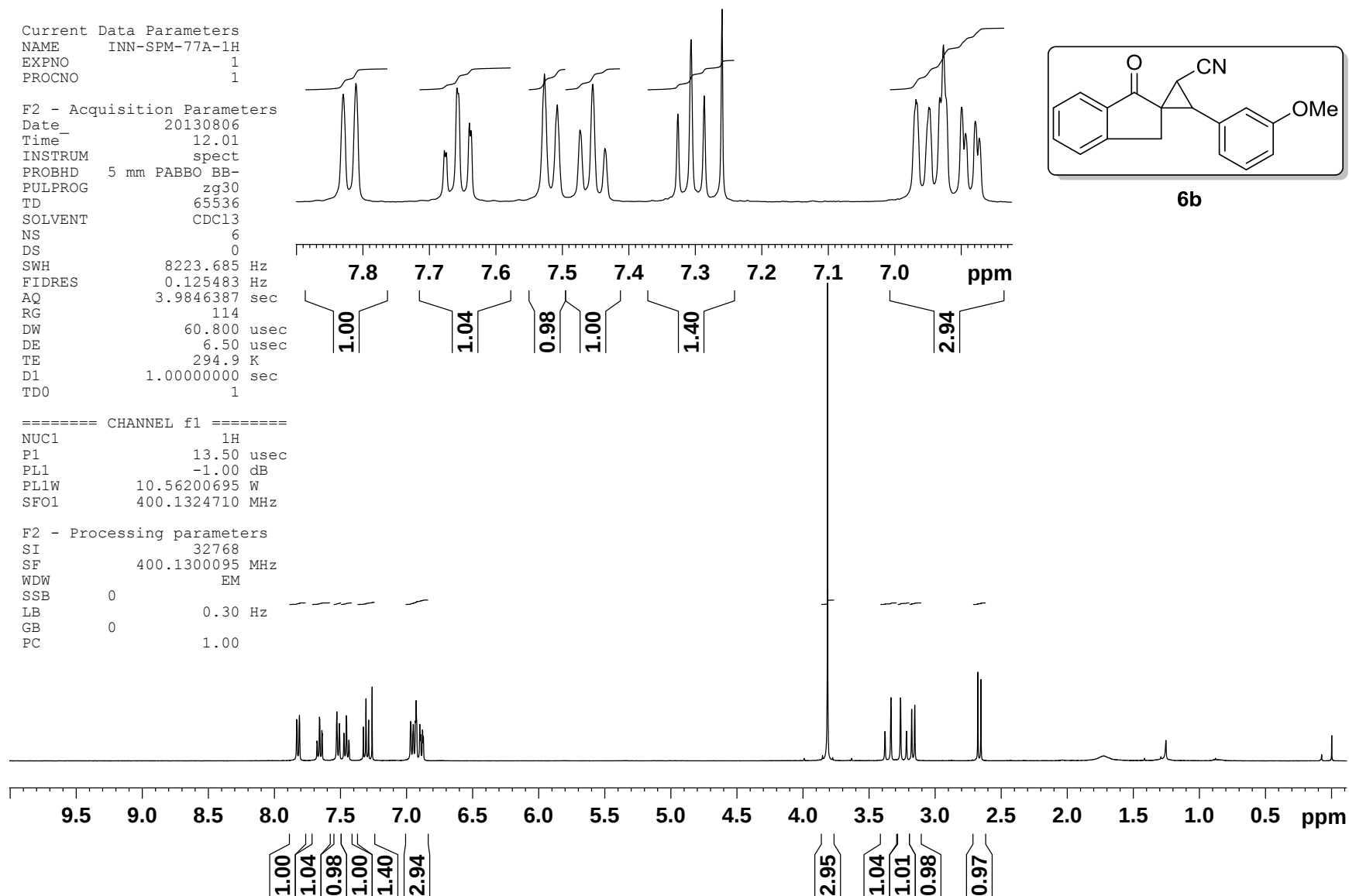
===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

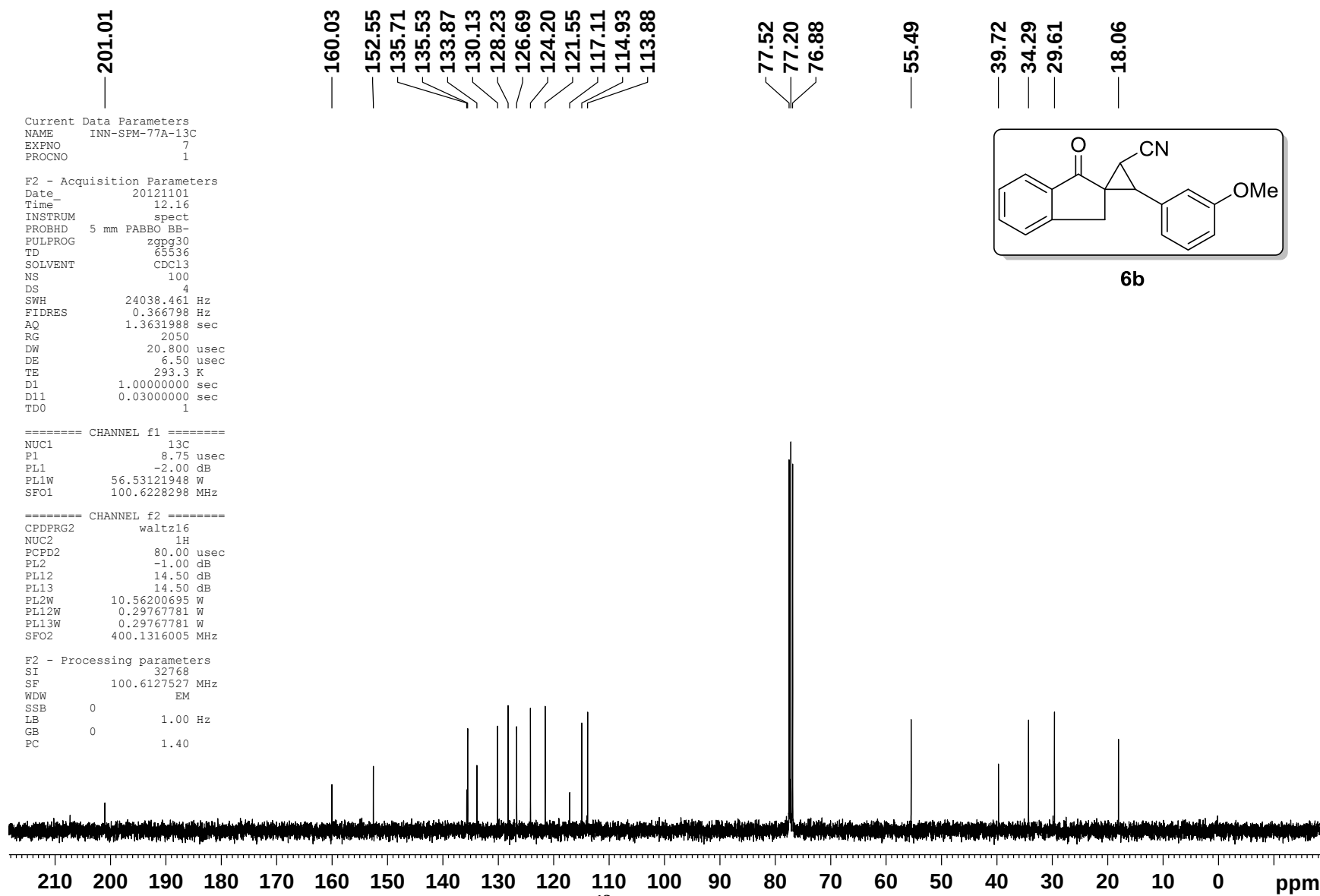
F2 - Processing parameters

SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**6a****Figure S57. ^1H NMR Spectrum of 6a**

Figure S58. ¹³C NMR Spectrum of 6a

Figure S59. ¹H NMR Spectrum of 6b



Current Data Parameters
NAME INN-GOPI-127A-1H
EXPNO 1
PROCNO 1

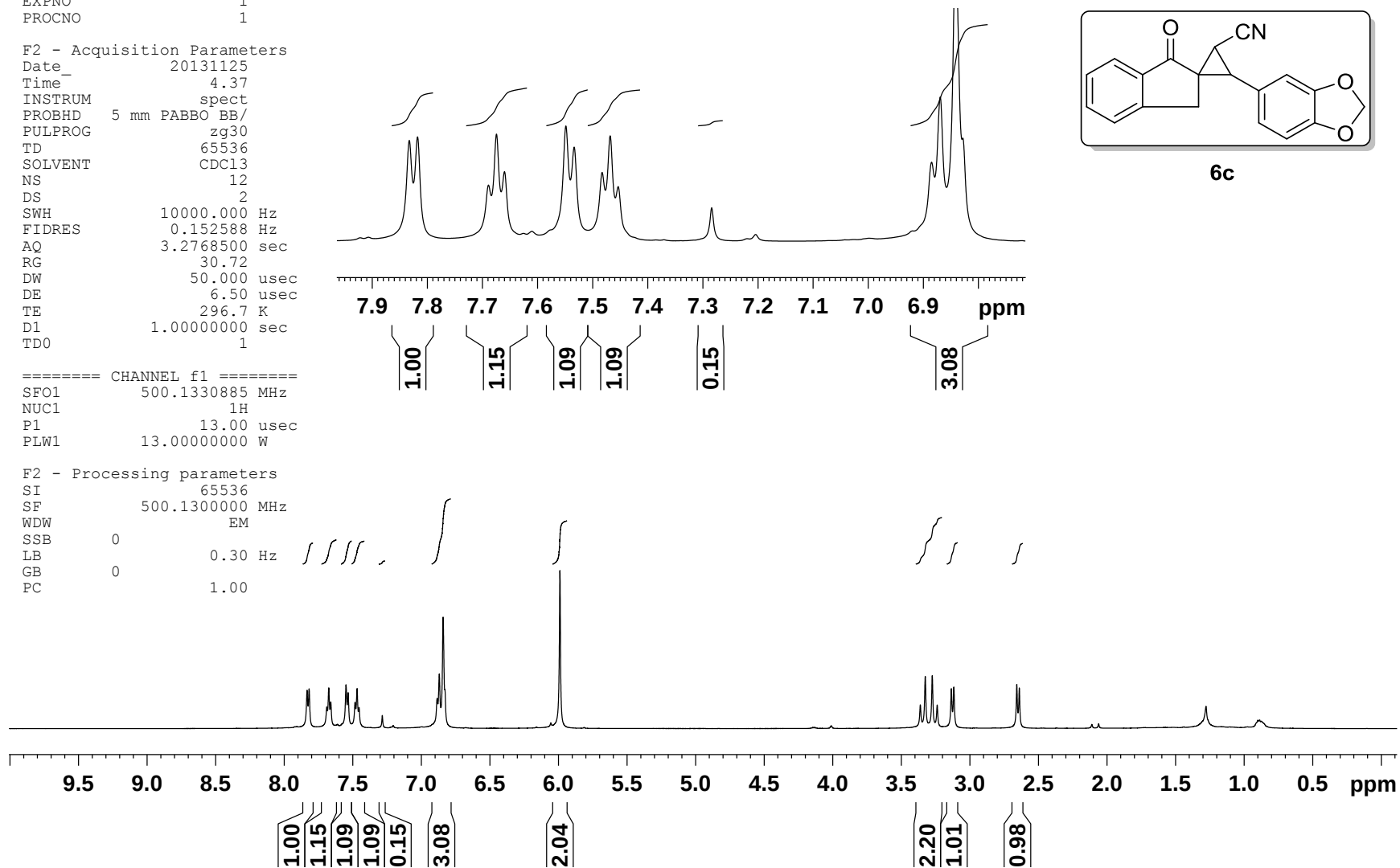
F2 - Acquisition Parameters

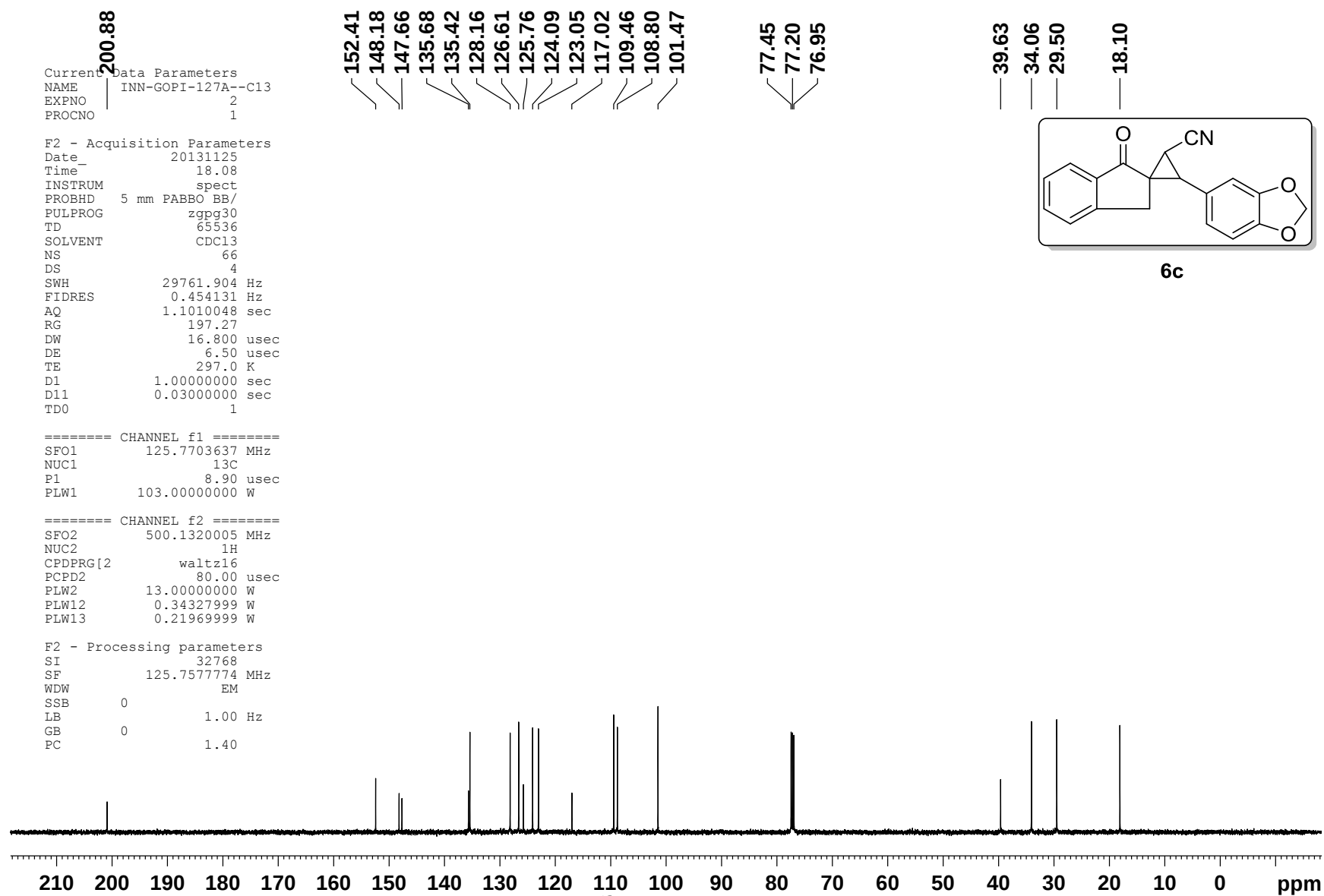
Date_ 20131125
Time_ 4.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 12
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters

SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S61. ¹H NMR Spectrum of 6c

Figure S62. ¹³C NMR Spectrum of 6c

Current Data Parameters
NAME INN-SPM-76A-1H
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters

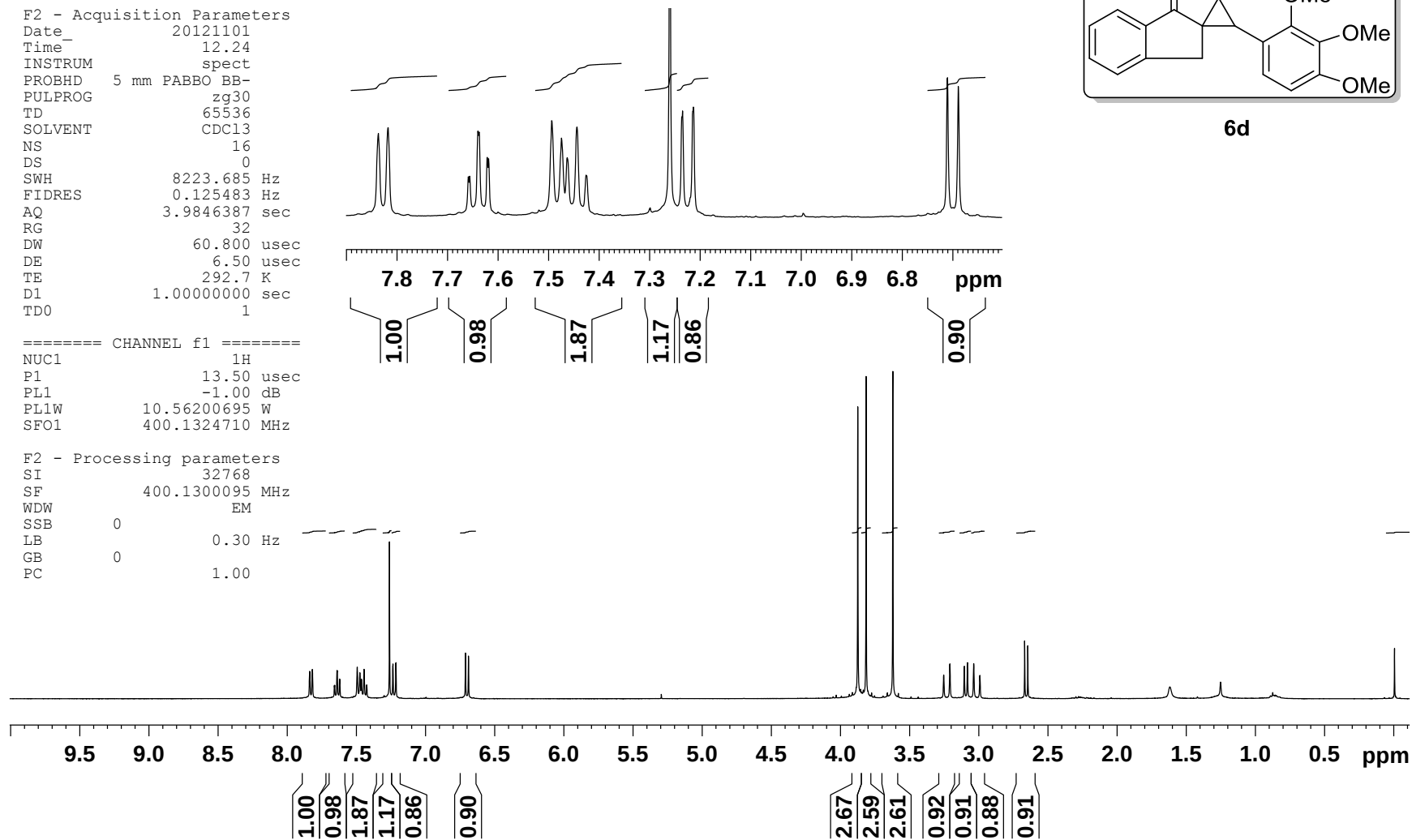
Date_ 20121101
Time_ 12.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TDO 1

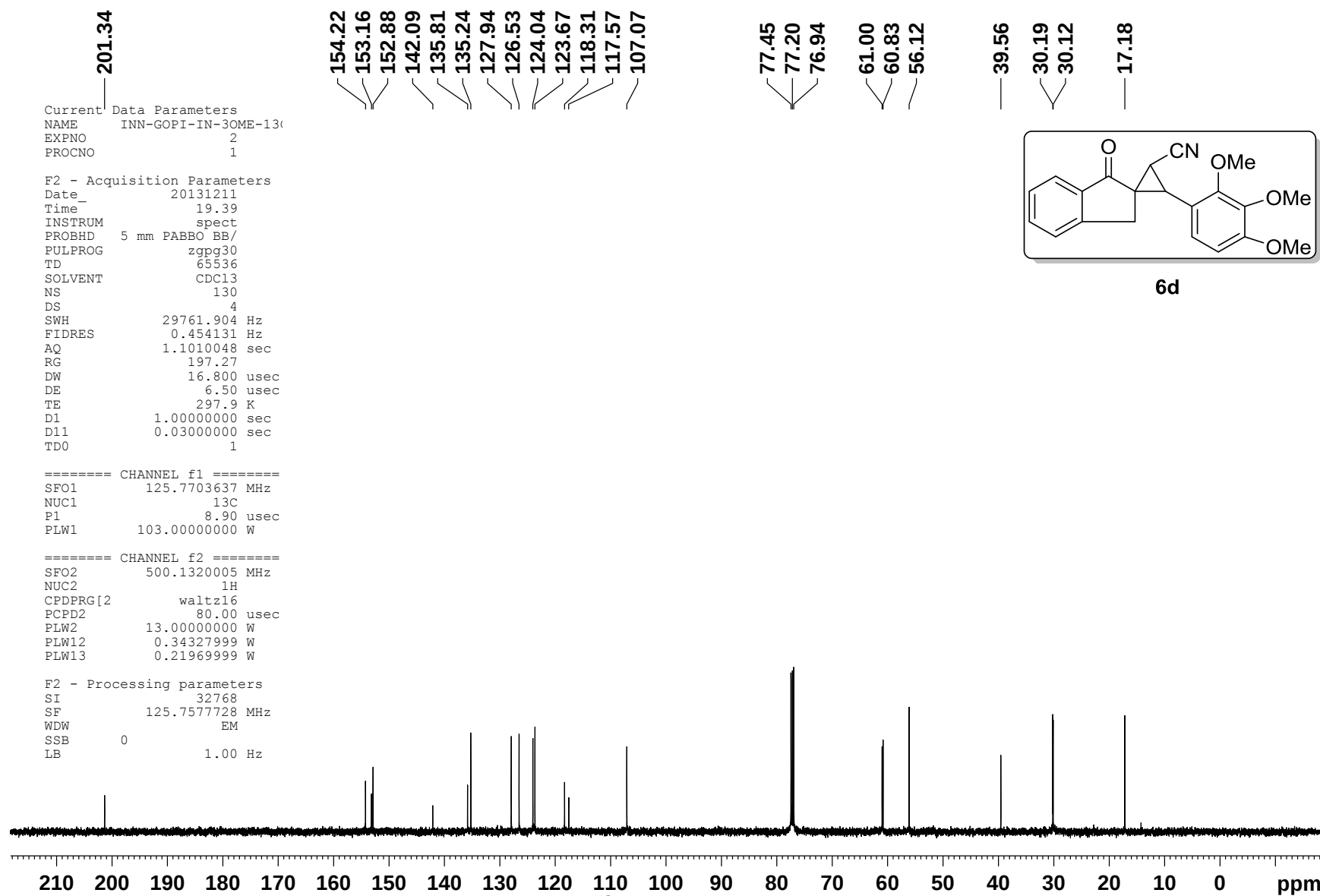
===== CHANNEL f1 =====

NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S63. ¹H NMR Spectrum of 6d

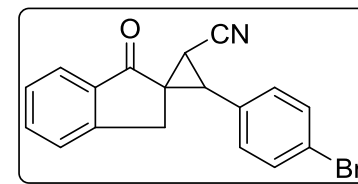
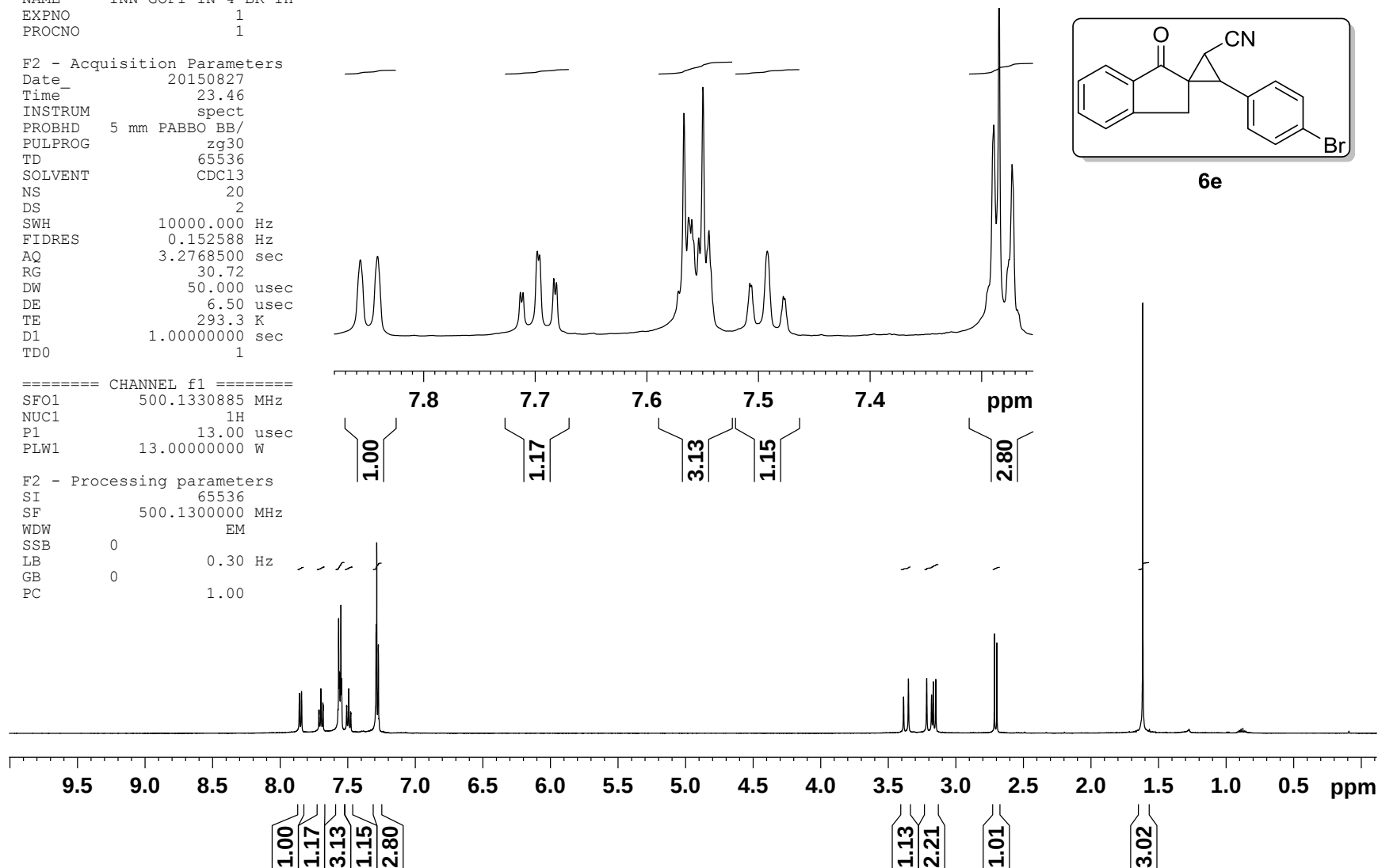
Figure S64. ¹³C NMR Spectrum of 6d

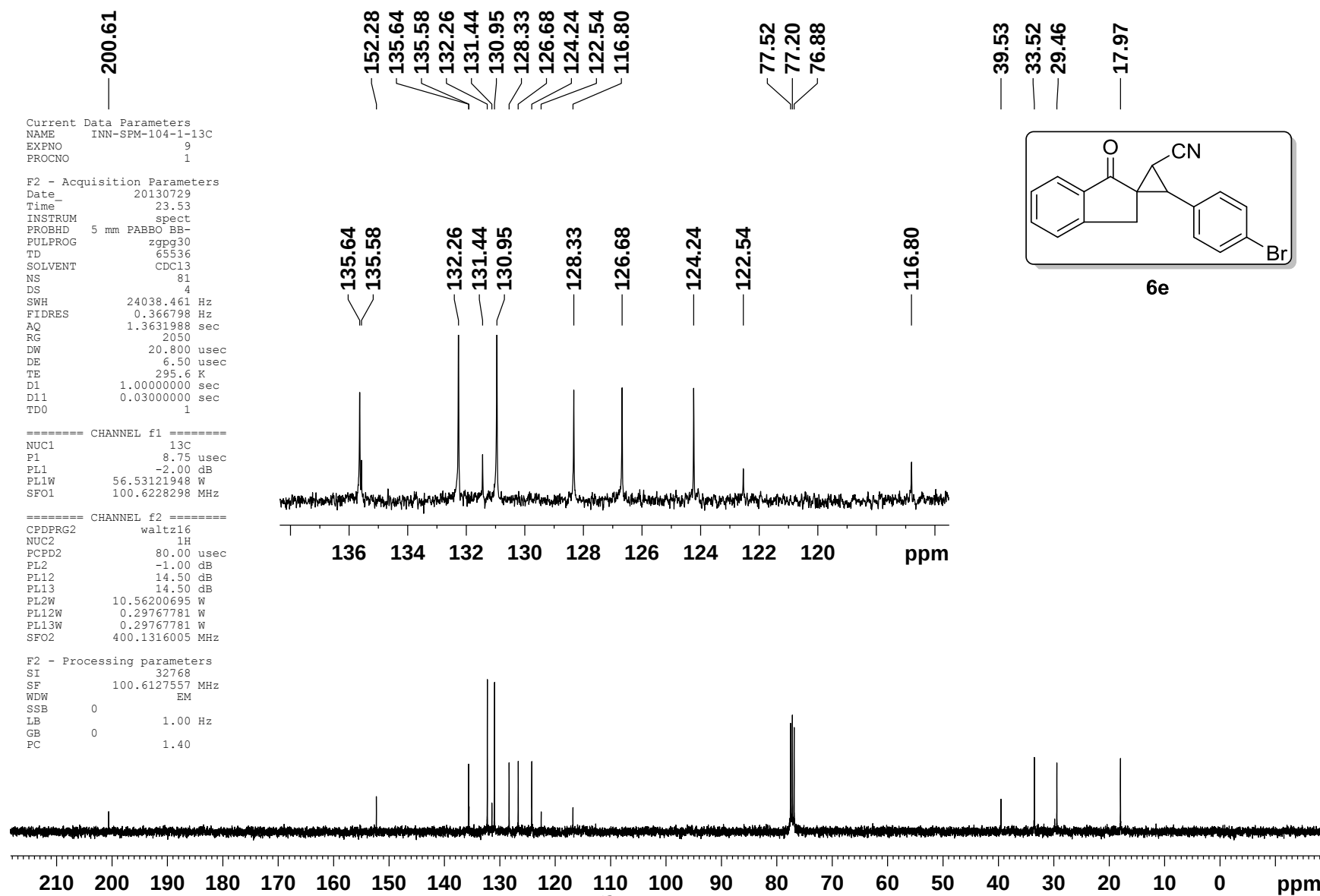
Current Data Parameters
NAME INN-GOPI-IN-4-BR-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150827
Time_ 23.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 13.00 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**6e****Figure S65. ¹H NMR Spectrum of 6e**



Current Data Parameters
 NAME INN-GOPI-IN-PF-1H
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters

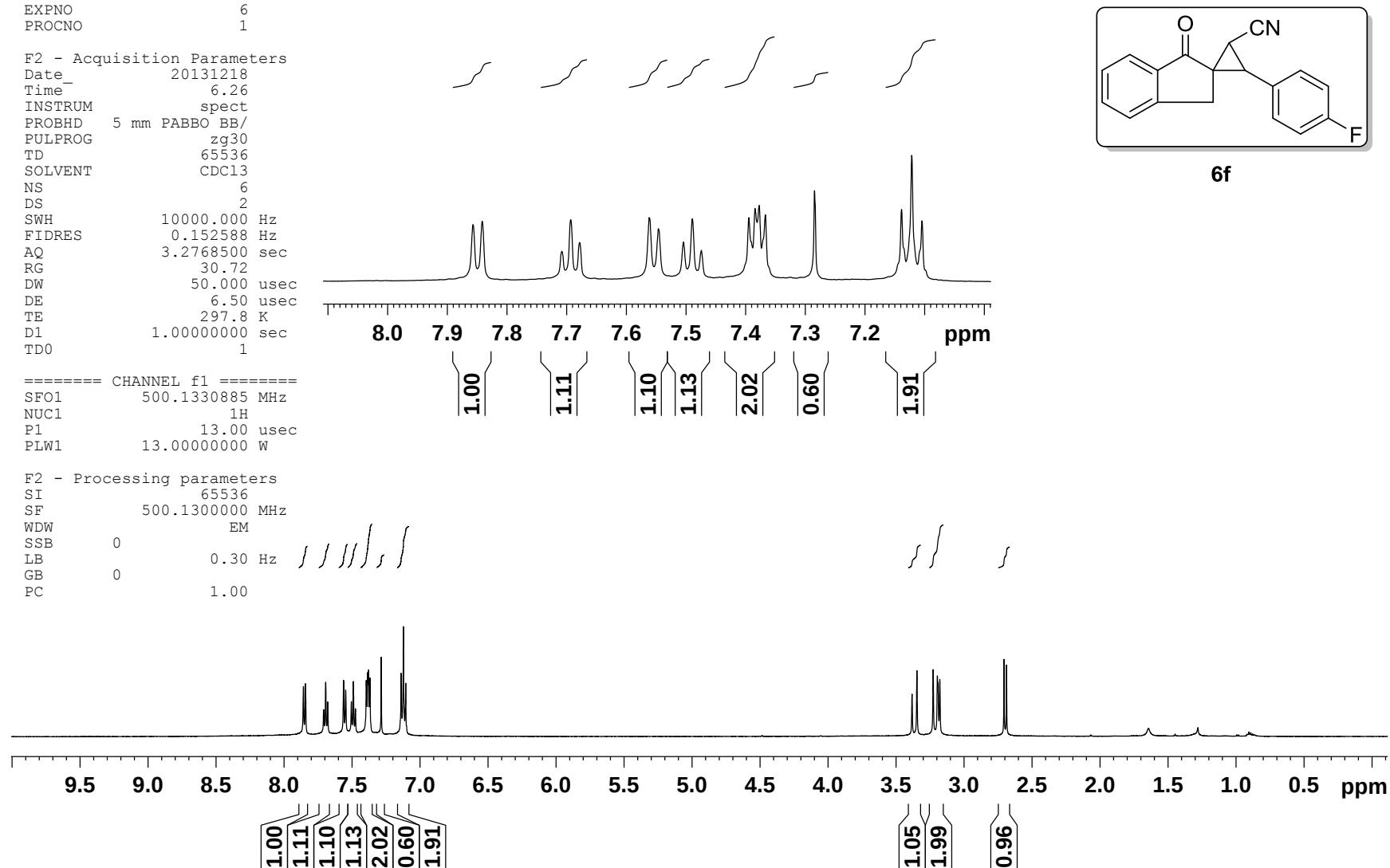
Date_ 20131218
 Time_ 6.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 6
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 30.72
 DW 50.000 usec
 DE 6.50 usec
 TE 297.8 K
 D1 1.00000000 sec
 TDO 1

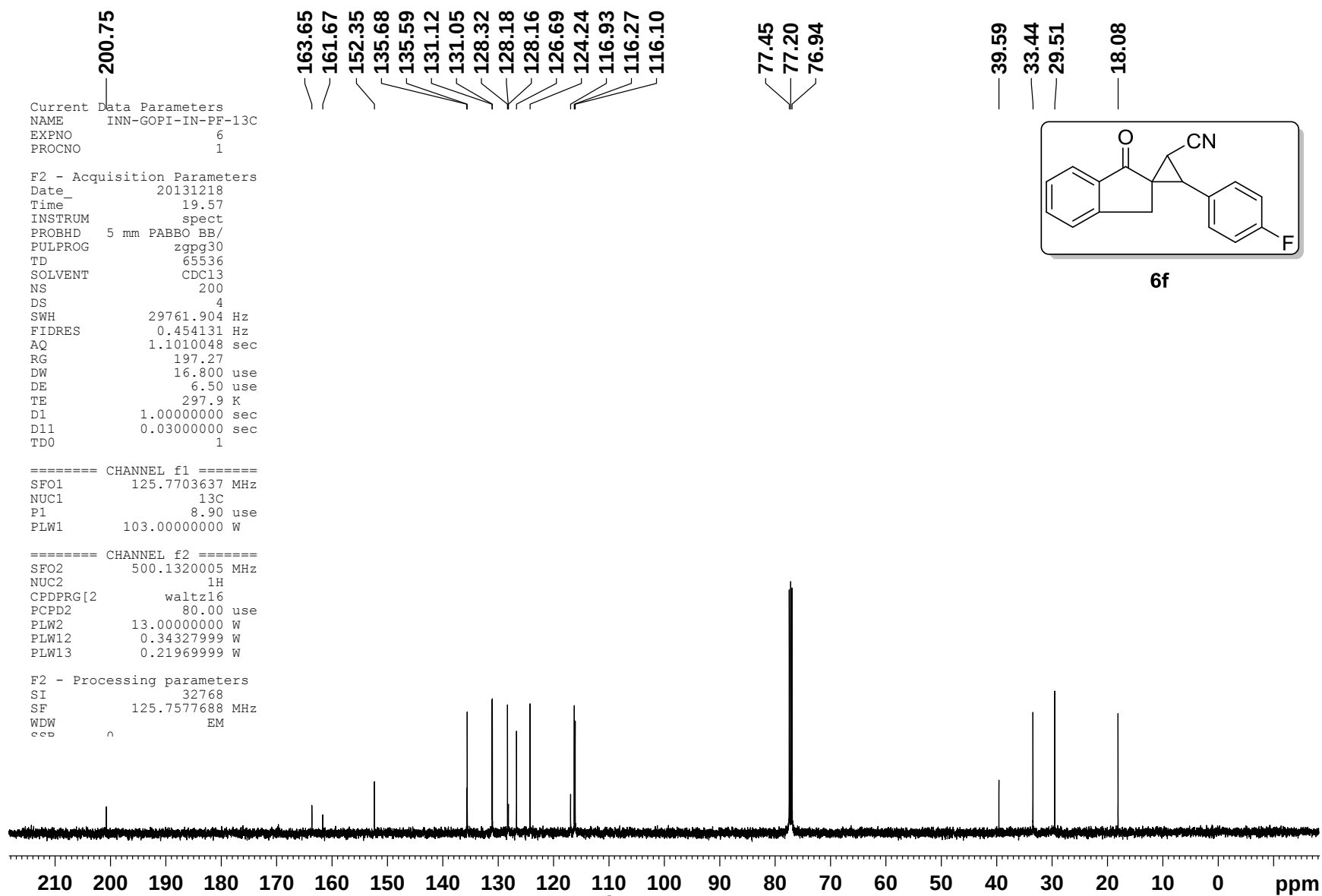
===== CHANNEL f1 =====

SFO1 500.1330885 MHz
 NUC1 1H
 P1 13.00 usec
 PLW1 13.00000000 W

F2 - Processing parameters

SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S67. ¹H NMR Spectrum of 6f

Figure S68. ¹³C NMR Spectrum of **6f**

```

F2 - Acquisition Parameters
Date_      20170105
Time       16.51
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD          54274
SOLVENT     CDCl3
NS          16
DS          0
SWH         8223.685 Hz
FIDRES      0.151522 Hz
AQ          3.2998593 sec
RG          90.5
DW          60.800 usec
DE          6.50 usec
TE          295.8 K
D1          1.00000000 sec
TD0         1

```

```
===== CHANNEL f1 =====
NUC1                      1H
P1                        14.75 usec
PL1                       -1.00 dB
PL1W                     10.56200695 W
SFO1                     400.1324710 MHz
```

```
F2 - Processing parameters
SI              32768
SF              400.1300099 MHz
WDW              EM
SSB             0
LB              0.30 Hz
GB             0
PC              1.00
```

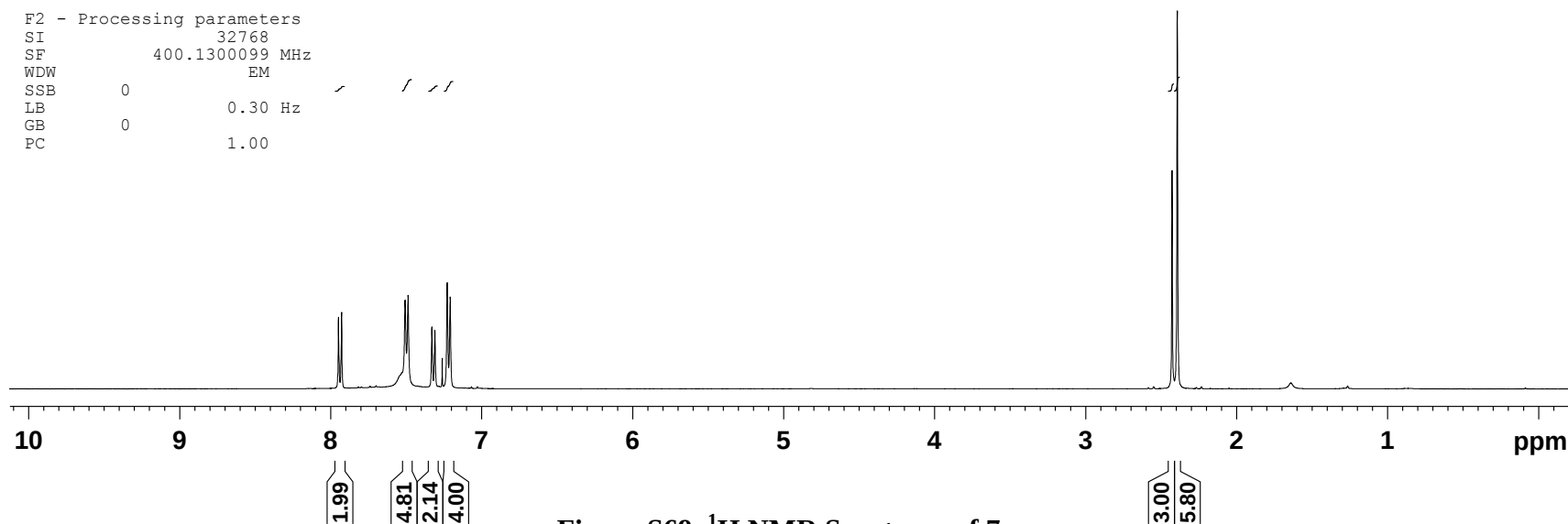
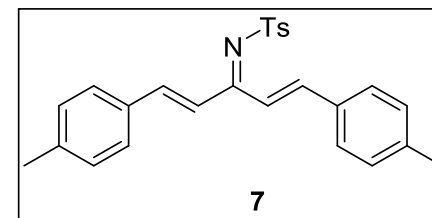
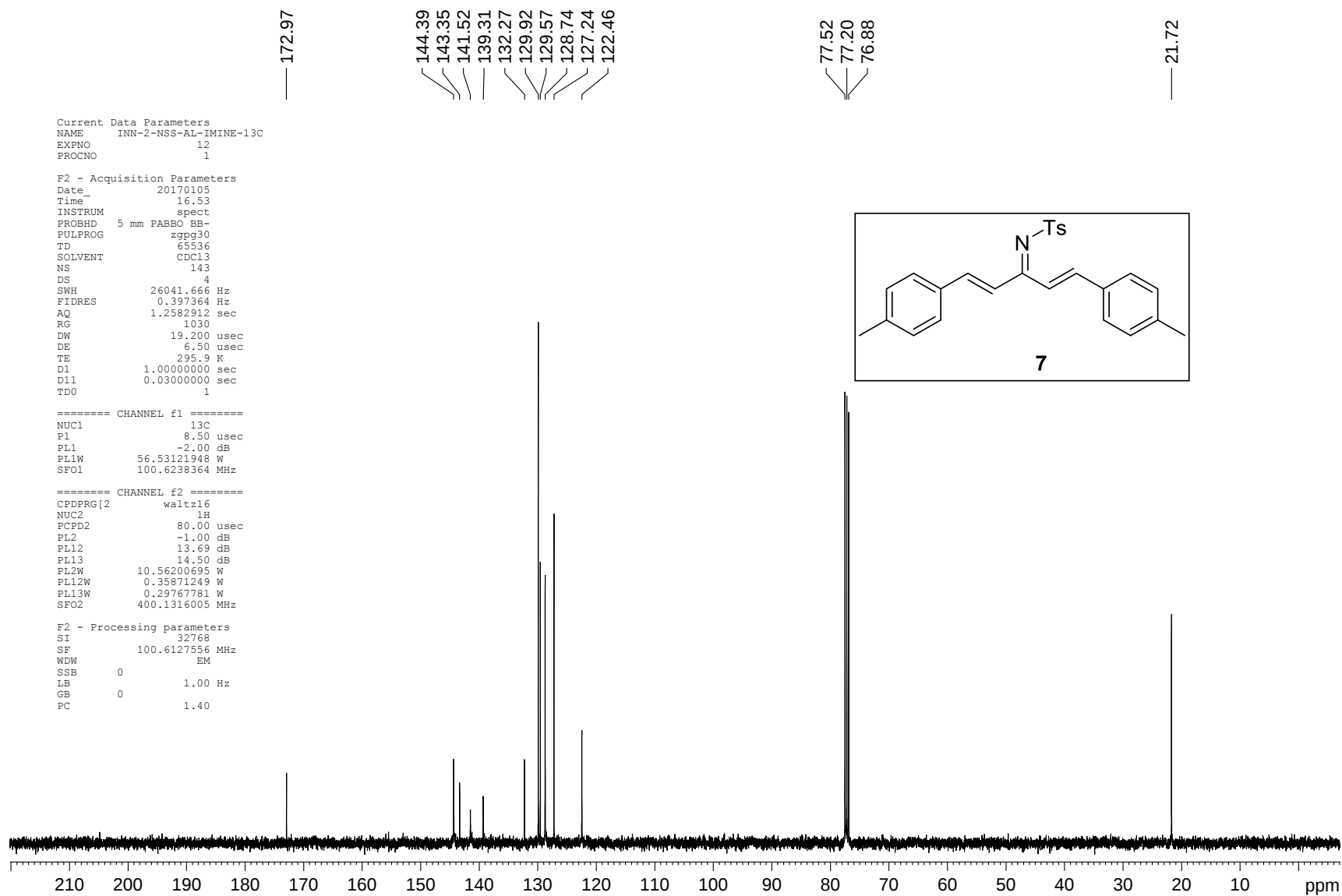


Figure S69. ^1H NMR Spectrum of 7

Figure S70. ¹³C NMR Spectrum of 7

Current Data Parameters
 NAME INN-SPM-110(11)-1H
 EXPNO 16
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130812
 Time_ 6.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 57690
 SOLVENT CDC13
 NS 29
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.166673 Hz
 AQ 2.9999299 sec
 RG 181
 DW 52.000 usec
 DE 6.50 usec
 TE 295.4 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.50 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1299942 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

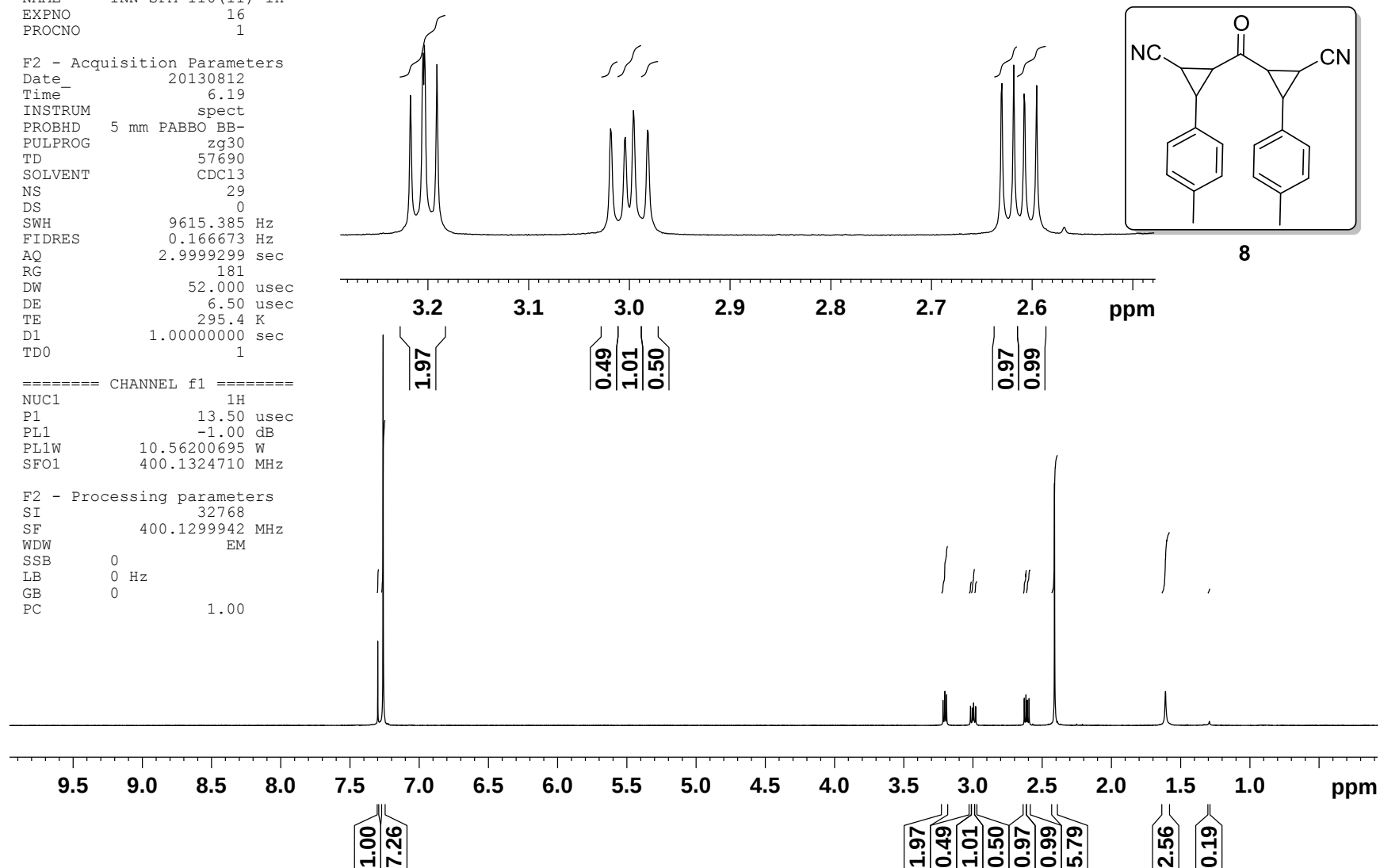
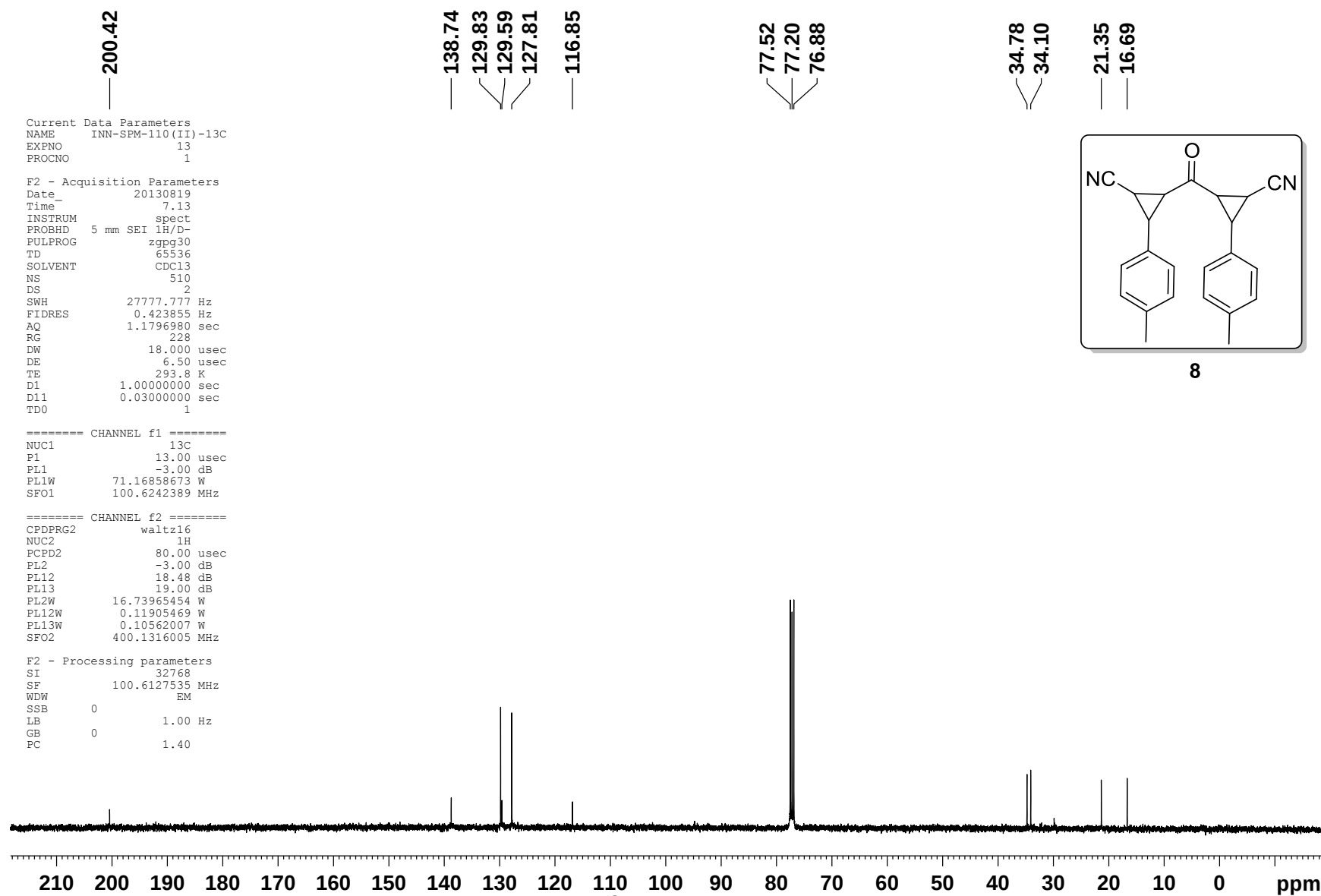
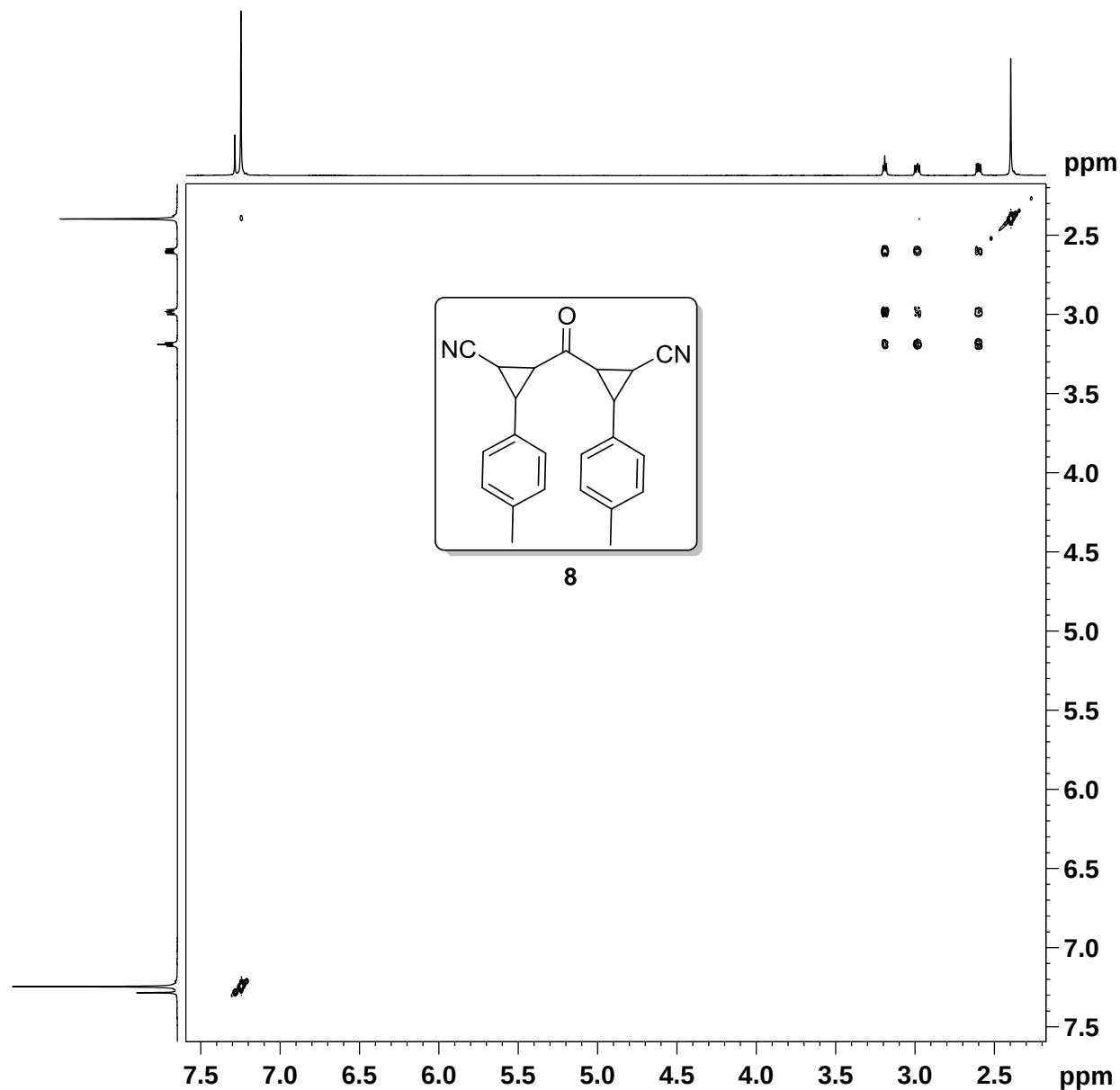


Figure S71. ¹H NMR Spectrum of 8

Figure S72. ¹³C NMR Spectrum of 8

Figure S73. ^1H - ^1H COSY NMR Spectrum of 8

Current Data Parameters
 NAME inn-spm-110-2-1cosy
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131013
 Time_ 21.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG cosygpph
 TD 2048
 SOLVENT CDCl3
 NS 24
 DS 32
 SWH 2685.285 Hz
 FIDRES 1.311174 Hz
 AQ 0.3813376 sec
 RG 106.54
 DW 186.200 usec
 DE 6.50 usec
 TE 297.0 K
 D0 0.00016965 sec
 D1 1.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 IN0 0.00037240 sec

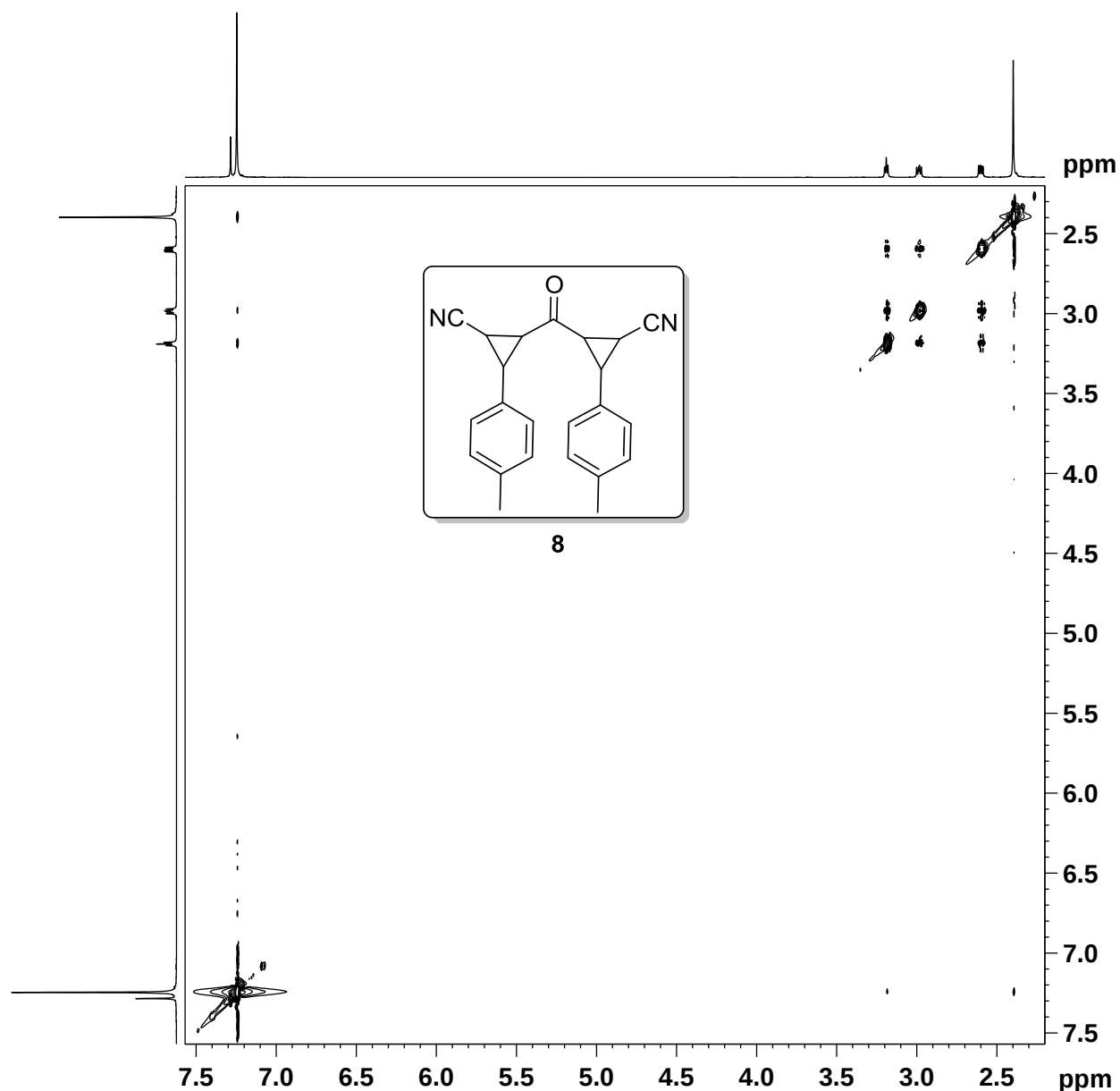
===== CHANNEL f1 =====
 SFO1 500.1324431 MHz
 NUC1 1H
 P1 13.00 usec
 P2 26.00 usec
 PLW1 13.00000000 W

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

F1 - Acquisition parameters
 TD 256
 SFO1 500.1324 MHz
 FIDRES 10.489393 Hz
 SW 5.369 ppm
 FnmODE TPPI

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.00

F1 - Processing parameters

Figure S74. ^1H - ^1H NOESY NMR Spectrum of 8

Current Data Parameters
 NAME inn-spm-110-2-1noesy
 EXPNO 1
 FPROCNO 1

F2 - Acquisition Parameters
 Date_ 20131013
 Time_ 21.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG noesygpph
 TD 2048
 SOLVENT CDCl3
 NS 24
 DS 32
 SWH 2685.285 Hz
 FIDRES 1.311174 Hz
 AQ 0.3813376 sec
 RG 106.54
 DW 186.200 usec
 DE 6.50 usec
 TE 297.0 K
 D0 0.00016965 sec
 D1 1.00000000 sec
 D8 0.30000001 sec
 D16 0.00020000 sec
 IN0 0.00037240 sec

===== CHANNEL f1 =====
 SFO1 500.1324431 MHz
 NUC1 1H
 P1 13.00 usec
 P2 26.00 usec
 PLW1 13.00000000 W

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

F1 - Acquisition parameters
 TD 256
 SFO1 500.1324 MHz
 FIDRES 10.489393 Hz
 SW 5.369 ppm
 FhMODE TPPI

F2 - Processing parameters
 SI 1024
 SF 500.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.00

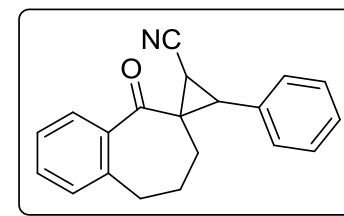
F1 - Processing parameters
 SI 1024
 MC2 TPPI
 SF 500.1300000 MHz
 WDW QSINE
 SSB 2
 LB 0 Hz
 GB 0

Current Data Parameters
 NAME INN-NSS-SUB-FINAL-1H
 EXPNO 3
 PROCNO 1

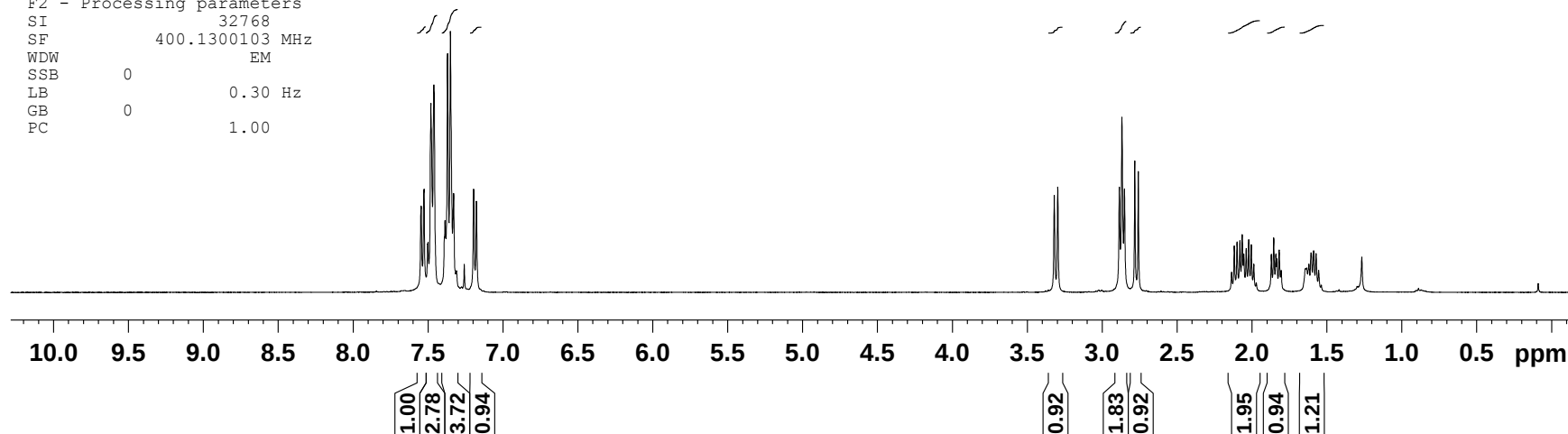
F2 - Acquisition Parameters
 Date_ 20170325
 Time 4.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 54274
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.151522 Hz
 AQ 3.2998593 sec
 RG 71.8
 DW 60.800 usec
 DE 6.50 usec
 TE 299.2 K
 D1 1.00000000 sec
 TD0 1

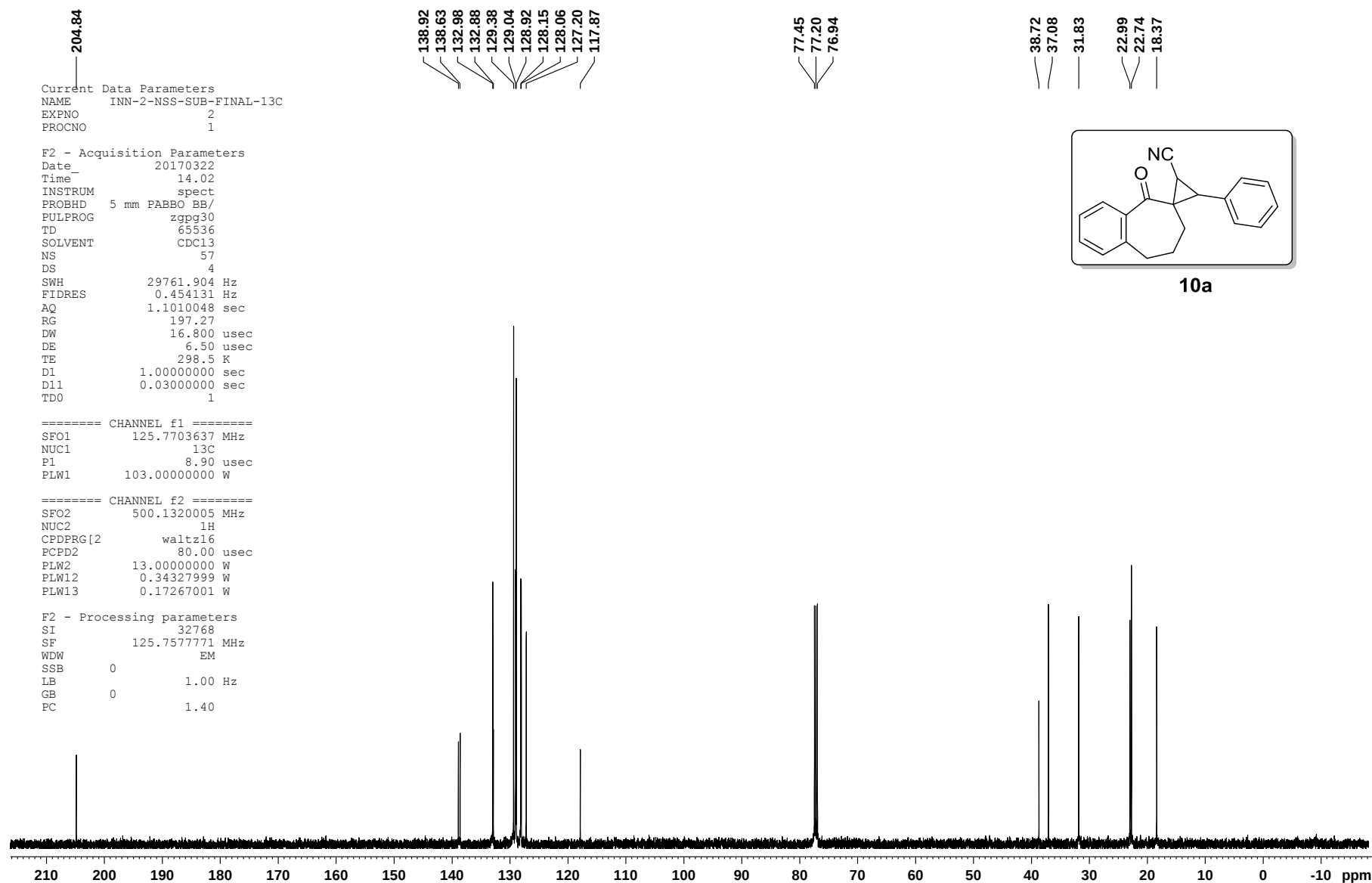
===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300103 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



10a

Figure S75. ¹H NMR Spectrum of 10a

Figure S76. ¹³C NMR Spectrum of 10a

```
Current Data Parameters
NAME      INN-GOPI-PY-PH-DDQ-1H
EXPNO      1
PROCNO     1
```

F2 - Acquisition Parameters

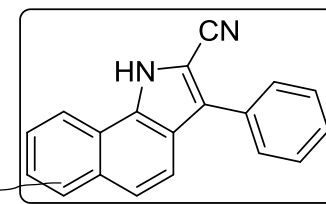
Date	20131220	
Time	9.04	
INSTRUM	spect	
PROBHD	5 mm SEI	1H/D-
PULPROG		zg30
TD		65536
SOLVENT		CDC13
NS		16
DS		0
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG		32
DW	60.800	usec
DE	6.50	usec
TE	294.4	K
D1	1.00000000	sec
TD0		1

```
===== CHANNEL f1 =====
NUC1                1H
P1                   6.75 usec
PL1                  -3.00 dB
PL1W                 16.73965454 W
SFO1                 400.1324710 MHz
```

```

F2 - Processing parameters
SI              32768
SF              400.1300095 MHz
WDW             EM
SSB             0
LB              0.30 Hz
GB              0
PC              1.00

```



11d

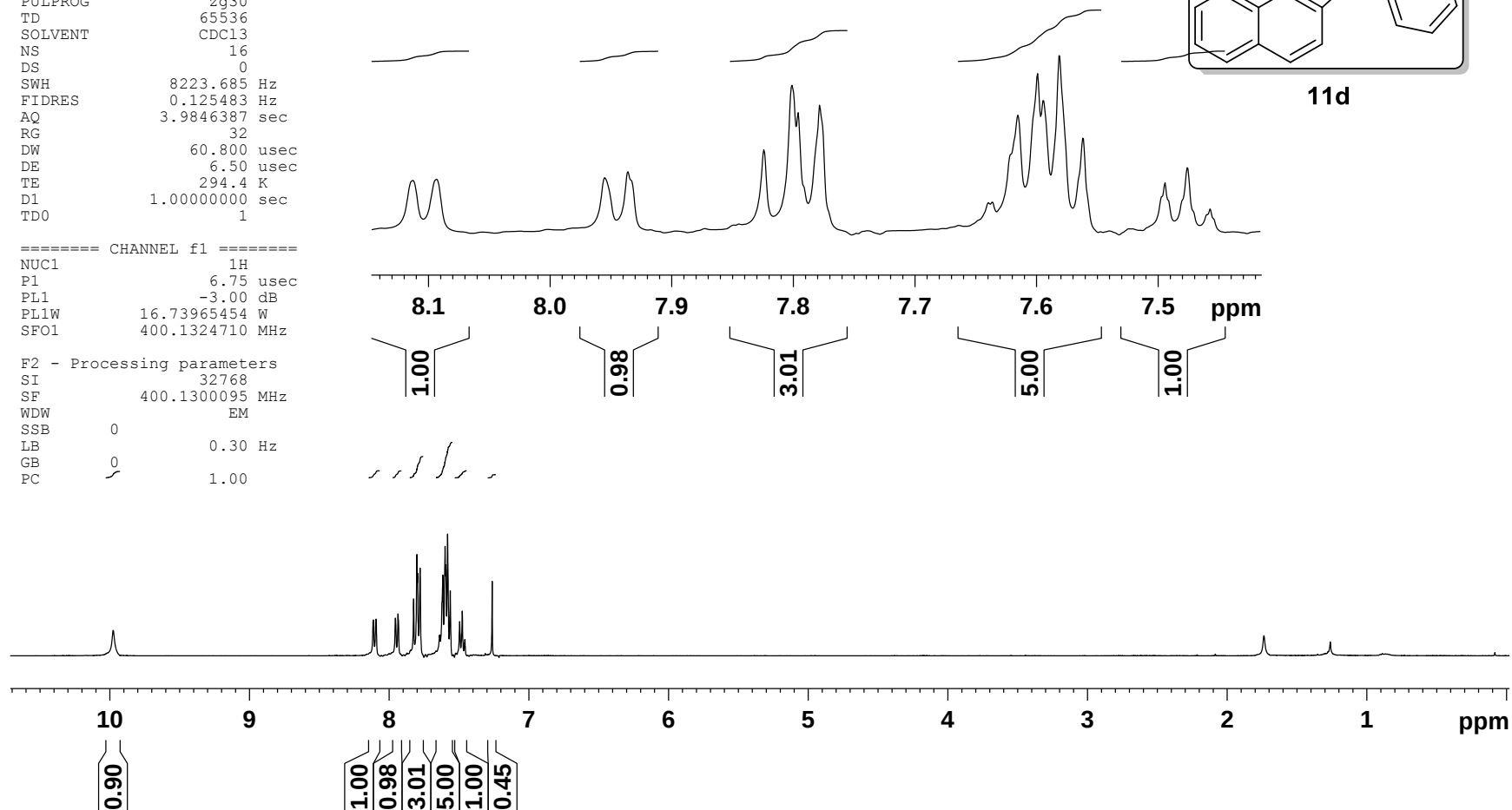


Figure S77. ^1H NMR Spectrum of 11d

