

Supporting Information

Synthesis of Functionalized Thieno[2,3-*b*]indoles via One-pot Reaction of Indoline-2-thiones with Morita–Baylis–Hillman and Rauhut–Currier Adducts of Nitroalkenes

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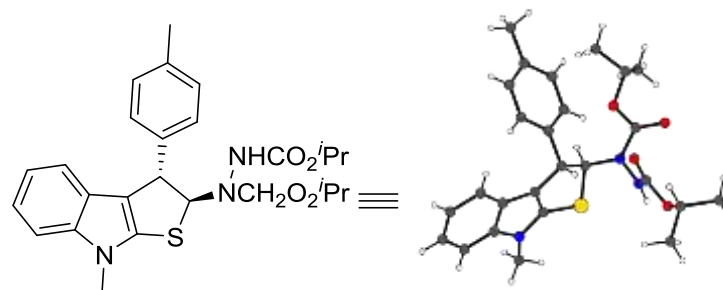
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Entry	Table of Contents	Page
1	Table S1. Crystal data and structure refinement for 3a	S5
2	Table S2. Crystal data and structure refinement for 4h	S7
3	Table S3. Crystal data and structure refinement for 6e	S9
4	Figure S1. ¹ H NMR Spectrum of 3a	S11
5	Figure S2. ¹³ C NMR Spectrum of 3a	S12
6	Figure S3. ¹ H NMR Spectrum of 4a	S13
7	Figure S4. ¹³ C NMR Spectrum of 4a	S14
8	Figure S5. ¹ H NMR Spectrum of 4b	S15
9	Figure S6. ¹³ C NMR Spectrum of 4b	S16
10	Figure S7. ¹ H NMR Spectrum of 4c	S17
11	Figure S8. ¹³ C NMR Spectrum of 4c	S18
12	Figure S9. ¹ H NMR Spectrum of 4d	S19
13	Figure S10. ¹³ C NMR Spectrum of 4d	S20
14	Figure S11. ¹ H NMR Spectrum of 4e	S21
15	Figure S12. ¹³ C NMR Spectrum of 4e	S22
16	Figure S13. ¹ H NMR Spectrum of 4f	S23
17	Figure S14. ¹³ C NMR Spectrum of 4f	S24
18	Figure S15. ¹ H NMR Spectrum of 4g	S25
19	Figure S16. ¹³ C NMR Spectrum of 4g	S26
20	Figure S17. ¹ H NMR Spectrum of 4h	S27

21	Figure S18. ^{13}C NMR Spectrum of 4h	S28
22	Figure S19. ^1H NMR Spectrum of 4i	S29
23	Figure S20. ^{13}C NMR Spectrum of 4i	S30
24	Figure S21. ^1H NMR Spectrum of 6a	S31
25	Figure S22. ^{13}C NMR Spectrum of 6a	S32
26	Figure S23. ^1H NMR Spectrum of 6b	S33
27	Figure S24. ^{13}C NMR Spectrum of 6b	S34
28	Figure S25. ^1H NMR Spectrum of 6c	S35
29	Figure S26. ^{13}C NMR Spectrum of 6c	S36
30	Figure S27. ^1H NMR Spectrum of 6d	S37
31	Figure S28. ^{13}C NMR Spectrum of 6d	S38
32	Figure S29. ^1H NMR Spectrum of 6e	S39
33	Figure S30. ^{13}C NMR Spectrum of 6e	S40
34	Figure S31. ^1H NMR Spectrum of 6f	S41
35	Figure S32. ^{13}C NMR Spectrum of 6f	S42
36	Figure S33. ^1H NMR Spectrum of 6g	S43
37	Figure S34. ^{13}C NMR Spectrum of 6g	S44
38	Figure S35. ^1H NMR Spectrum of 6h	S45
39	Figure S36. ^{13}C NMR Spectrum of 6h	S46
40	Figure S37. ^1H NMR Spectrum of 6i	S47
41	Figure S38. ^{13}C NMR Spectrum of 6i	S48

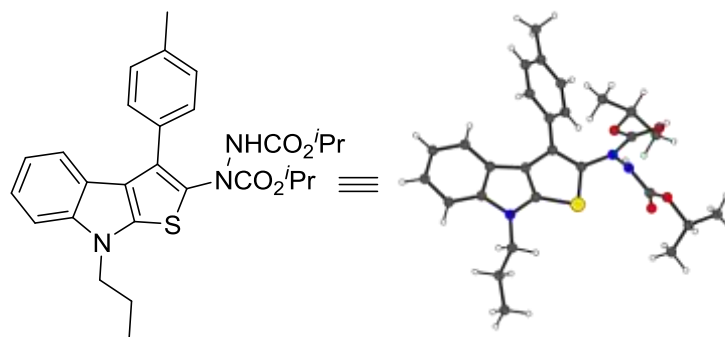
42	Figure S39. ^1H NMR Spectrum of 6j	S49
43	Figure S40. ^{13}C NMR Spectrum of 6j	S50
44	Figure S41. ^{13}C -APT Spectrum of 6j	S51
45	Figure S42. ^{19}F NMR Spectrum of 6j	S52
46	Figure S43. ^1H NMR Spectrum of 6k	S53
47	Figure S44. ^{13}C NMR Spectrum of 6k	S54
48	Figure S45. ^1H - ^1H -COSY Spectrum of 6k	S55
49	Figure S46. ^1H NMR Spectrum of 6l	S56
50	Figure S47. ^{13}C NMR Spectrum of 6l	S57
51	Figure S48. ^1H NMR Spectrum of 6m	S58
52	Figure S49. ^{13}C NMR Spectrum of 6m	S59
53	Figure S50. ^1H NMR Spectrum of 6n	S60
54	Figure S51. ^{13}C NMR Spectrum of 6n	S61
55	Figure S52. ^1H NMR Spectrum of 6o	S62
56	Figure S53. ^{13}C NMR Spectrum of 6o	S63
57	Figure S54. ^1H NMR Spectrum of 6p	S64
58	Figure S55. ^{13}C NMR Spectrum of 6p	S65
59	Figure S56. ^1H NMR Spectrum of 6q	S66
60	Figure S57. ^{13}C NMR Spectrum of 6q	S67
61	Figure S58. ^1H NMR Spectrum of 8	S68
62	Figure S59. ^{13}C NMR Spectrum of 8	S69

Table S1. Crystal data and structure refinement for compound **3a**

Identification code	INN-MVD-798
Empirical formula	C ₂₆ H ₃₁ N ₃ O ₄ S
Formula weight	482.60
Temperature	150 K
Wavelength	0.71070 Å
Crystal system, space group	Triclinic P -1
Unit cell dimensions	$a = 11.08(2)$ Å $\alpha = 63.18(17)^\circ$ $b = 11.755(18)$ Å $\beta = 66.29(17)^\circ$ $c = 11.961(14)$ Å $\gamma = 71.29(18)^\circ$
Volume	1254(4) Å ³
Z	2
Density (Calculated)	1.278 Mg/m ³
Absorption coefficient	0.166 mm ⁻¹

F (000)	514
Crystal size	<u>0.16</u> × <u>0.16</u> × <u>0.04</u> mm
Theta range for data collection	<u>3.1</u> to <u>25.0</u> °
Index ranges	-13<=h<=13, -13<=k<=13, -14<=l<=14
Reflections collected	<u>15311</u>
Independent reflections	4396 [R(int) = <u>0.144</u>]
Completeness to theta = 25 ⁰	99.7 %
Absorption correction	Numerical
Max. and min. transmission	0.989 and 0.997
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4396 / 0 / 313
Goodness-of-fit on F ²	0.870
Final R indices [I>2sigma (I)]	R1 = 0.0621, wR2 = 0.1244
R indices (all data)	R1 = 0.1060, wR2 = 0.1519
Largest diff. peak and hole	<u>0.35</u> and <u>-0.31</u> e.Å ⁻³

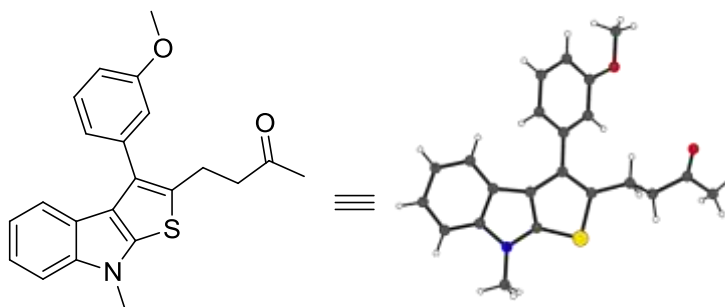
Table S2. Crystal data and structure refinement for compound **4h**



Identification code	INN-MVD-796
Empirical formula	C ₂₈ H ₃₃ N ₃ O ₄ S
Formula weight	507.63
Temperature	150 K
Wavelength	0.71070 Å
Crystal system, space group	Monoclinic P 21/c
Unit cell dimensions	$a = 15.959(5) \text{ \AA}$ $\alpha = 90^\circ$ $b = 8.470(2) \text{ \AA}$ $\beta = 108.034(4)^\circ$ $c = 21.482(6) \text{ \AA}$ $\gamma = 90^\circ$
Volume	2761.1(13) Å ³
Z	4

Density (Calculated)	1.221 Mg/m ³
Absorption coefficient	0.154 mm ⁻¹
F (000)	1080.0
Crystal size	<u>0.35</u> × <u>0.24</u> × <u>0.07</u> mm
Theta range for data collection	<u>3.1</u> to <u>29.2</u> °
Index ranges	-18<=h<=21, -9<=k<=11, -29<=l<=29
Reflections collected	<u>37563</u>
Independent reflections	<u>7432</u> [R(int) = <u>0.064</u>]
Completeness to theta = 29.165 ⁰	99.6 %
Absorption correction	Numerical
Max. and min. transmission	<u>0.989</u> , <u>0.963</u>
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7432 / 0 / 331
Goodness-of-fit on F ²	1.102
Final R indices [I>2sigma (I)]	R1 = 0.0545, wR2 = 0.1192
R indices (all data)	R1 = 0.0701, wR2 = 0.1290
Largest diff. peak and hole	<u>0.40</u> and <u>-0.39</u> e.Å ⁻³

Table S3. Crystal data and structure refinement for compound **6e**



Identification code	INN-MVD-611
Empirical formula	C ₂₂ H ₂₁ NO ₂ S
Formula weight	363.46
Temperature	150 K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic P 21/n
Unit cell dimensions	$a = 11.8883(3) \text{ \AA}$ $\alpha = 90^\circ$ $b = 7.4114(3) \text{ \AA}$ $\beta = 95.281(3)^\circ$ $c = 20.6224(7) \text{ \AA}$ $\gamma = 90^\circ$
Volume	1809.31(11) Å ³
Z	4

Density (Calculated)	1.334 Mg/m ³
Absorption coefficient	0.195 mm ⁻¹
F (000)	768
Crystal size	<u>0.33</u> × <u>0.22</u> × <u>0.15</u> mm
Theta range for data collection	<u>2.0</u> to <u>25.0</u> °
Index ranges	-14<=h<=14, -8<=k<=8, -14<=l<=24
Reflections collected	<u>8097</u>
Independent reflections	3169 [R(int) = <u>0.032</u>]
Completeness to theta = 25.0 ⁰	99.6 %
Absorption correction	Numerical
Max. and min. transmission	0.960, 1.000
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3169 / 0 / 238
Goodness-of-fit on F ²	1.061
Final R indices [I>2sigma (I)]	R1 = 0.0354, wR2 = 0.0940
R indices (all data)	R1 = 0.0431, wR2 = 0.0986
Largest diff. peak and hole	<u>0.23</u> and <u>-0.38</u> e.Å ⁻³

Current Data Parameters
NAME INN-MVD-798-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160403
Time 11.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 294.7 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 500.1298708 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

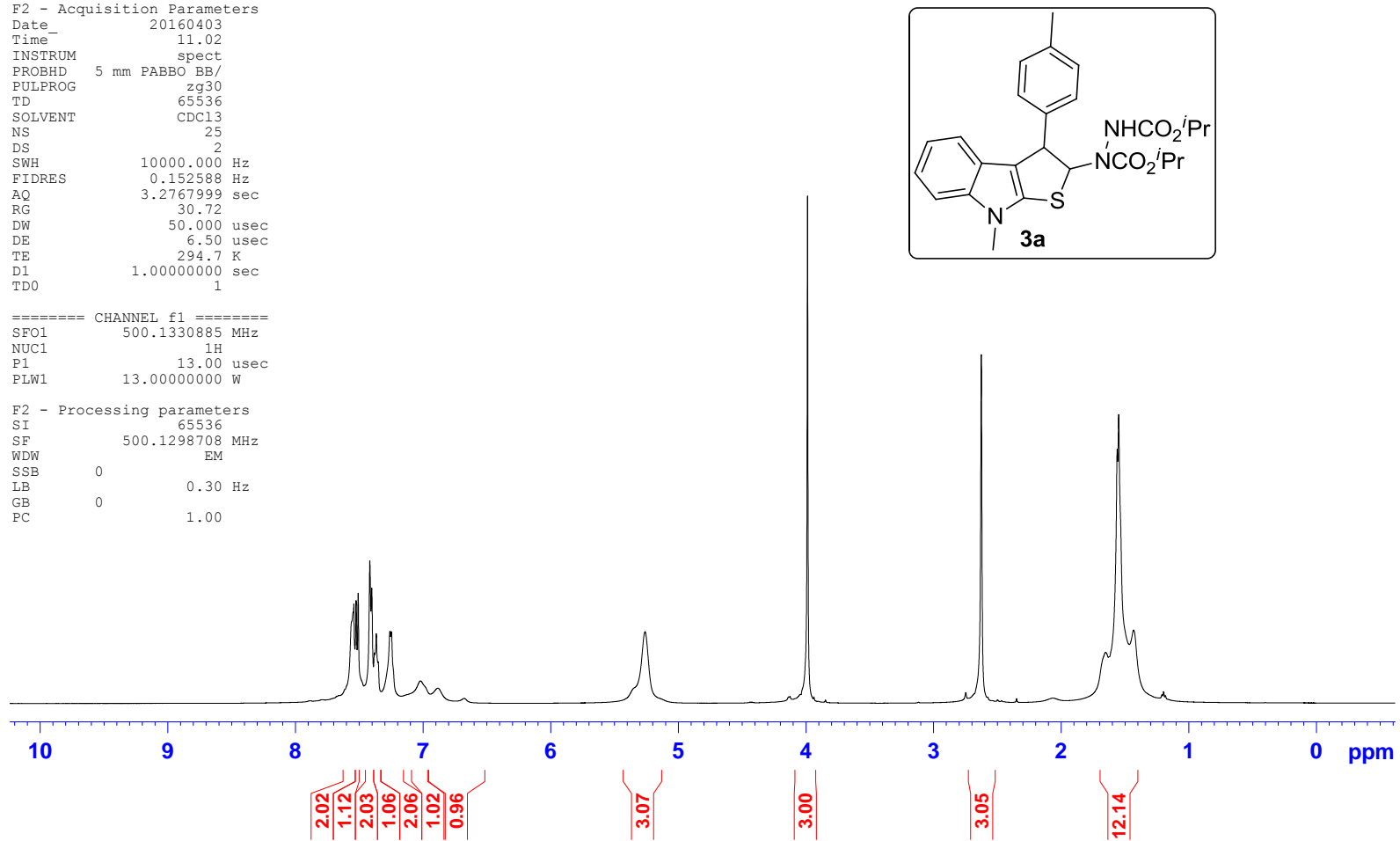


Figure S1. ^1H NMR Spectrum of 3a

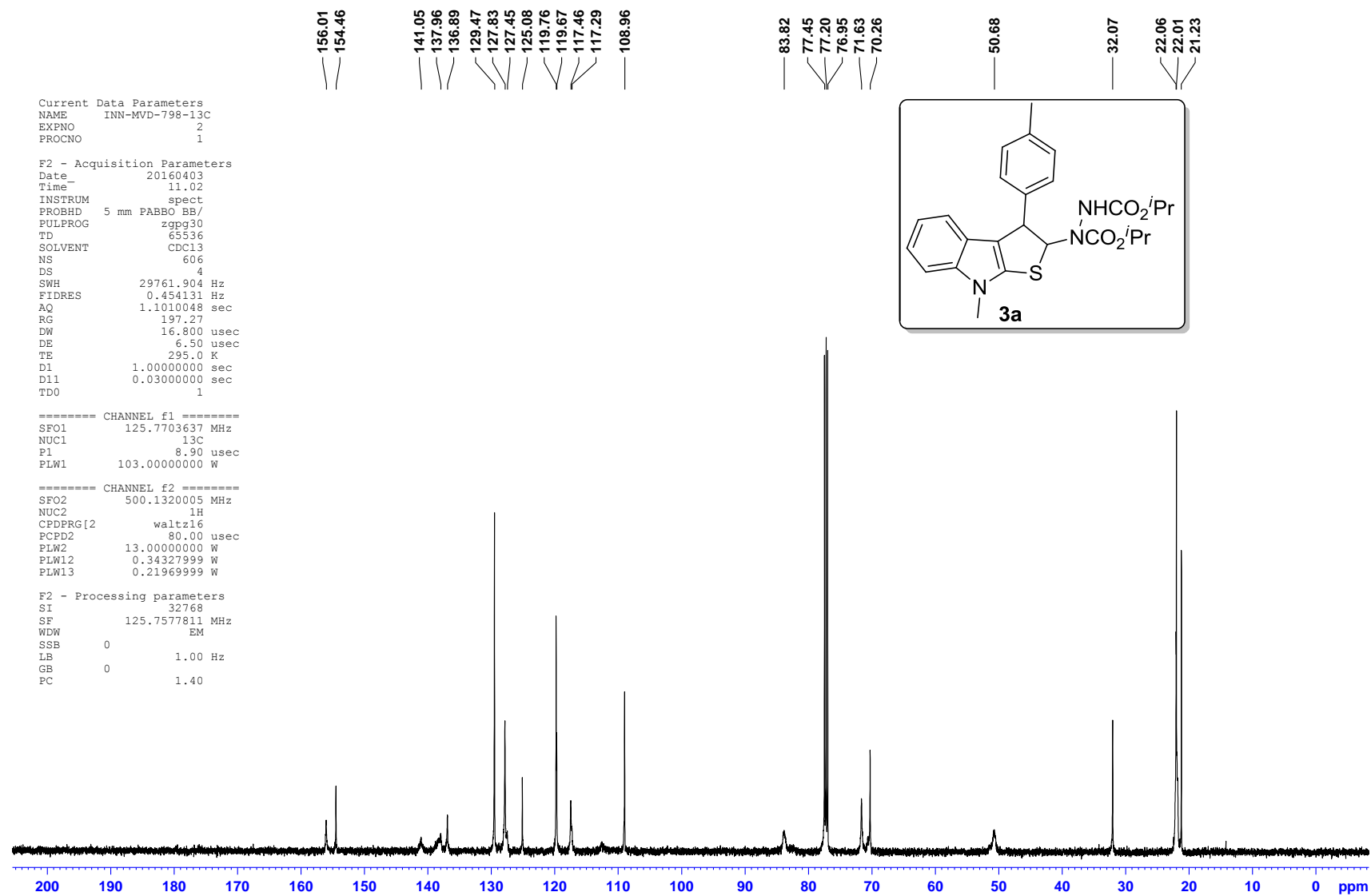


Figure S2. ¹³C NMR Spectrum of 3a

Current Data Parameters
NAME INN-MVD-770-1H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160308
Time_ 15.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TDO 1

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

F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

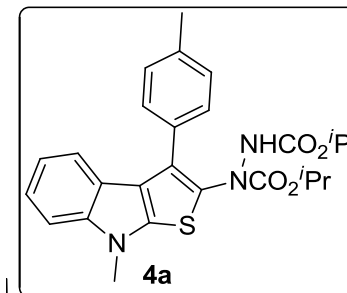
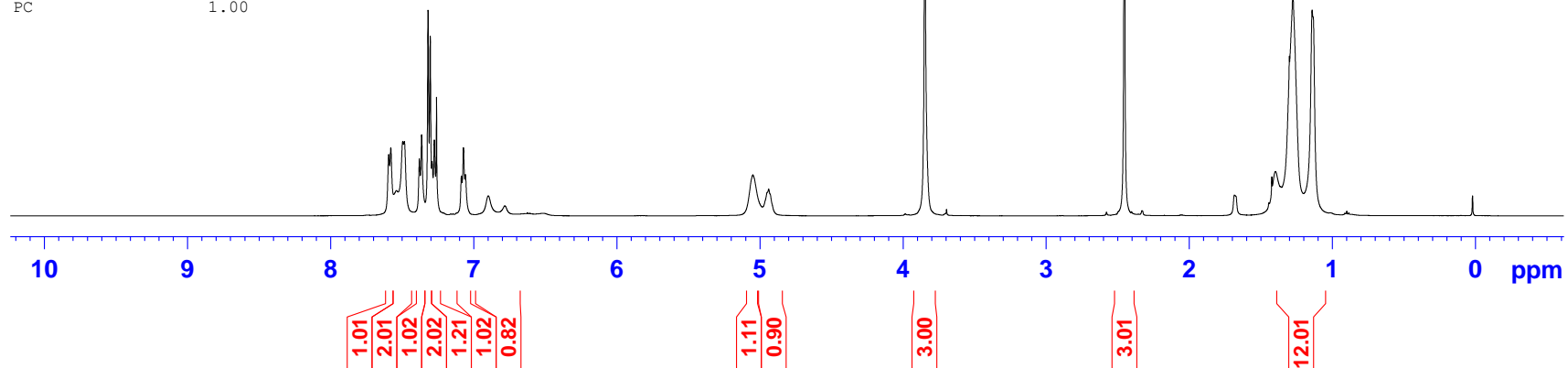


Figure S3. ¹H NMR Spectrum of **4a**

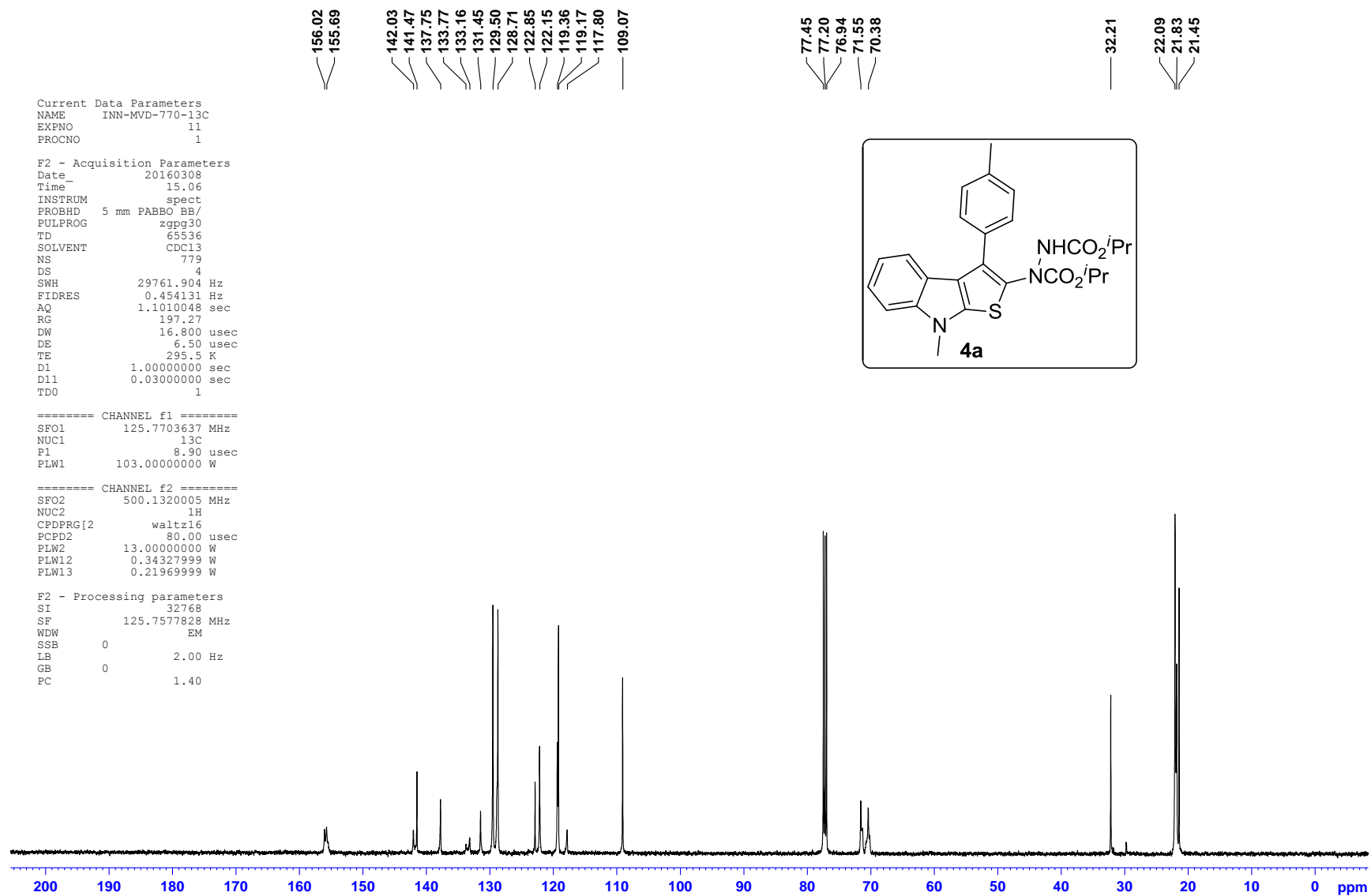


Figure S4. ¹³C NMR Spectrum of 4a

Current Data Parameters
NAME INN-MVD-800-1H
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160408
Time_ 14.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 13.43
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.0000000 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 500.1300117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

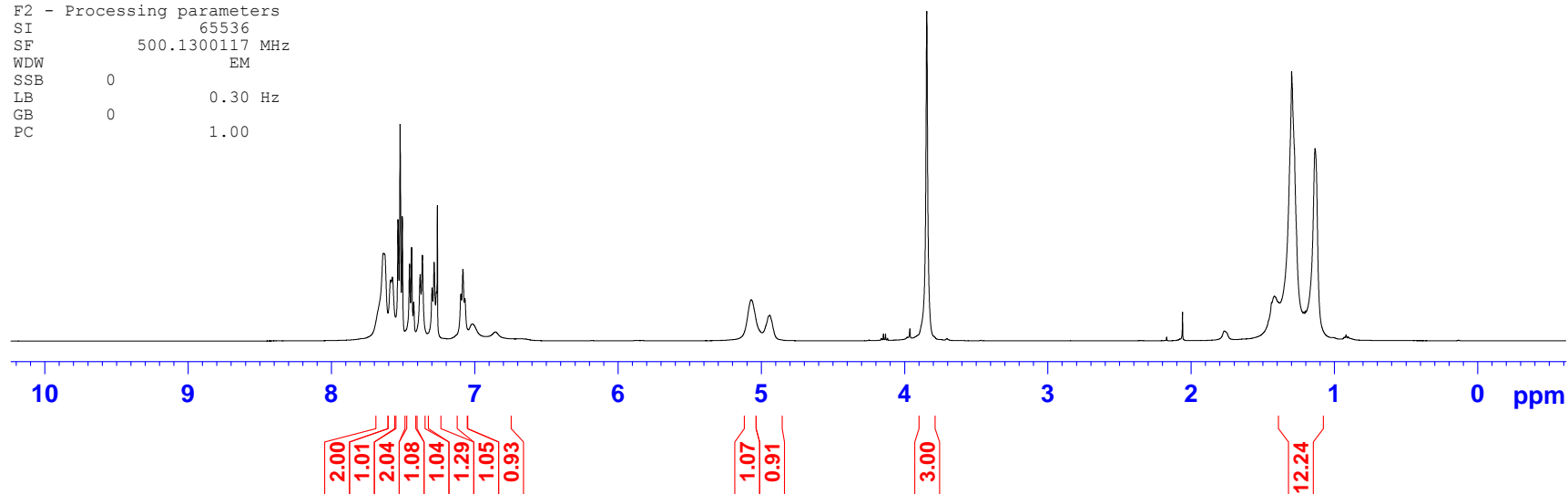
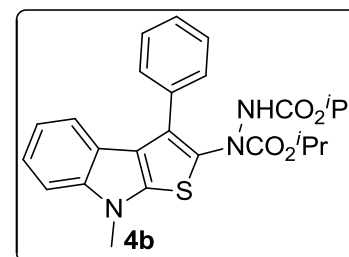


Figure S5. ^1H NMR Spectrum of **4b**

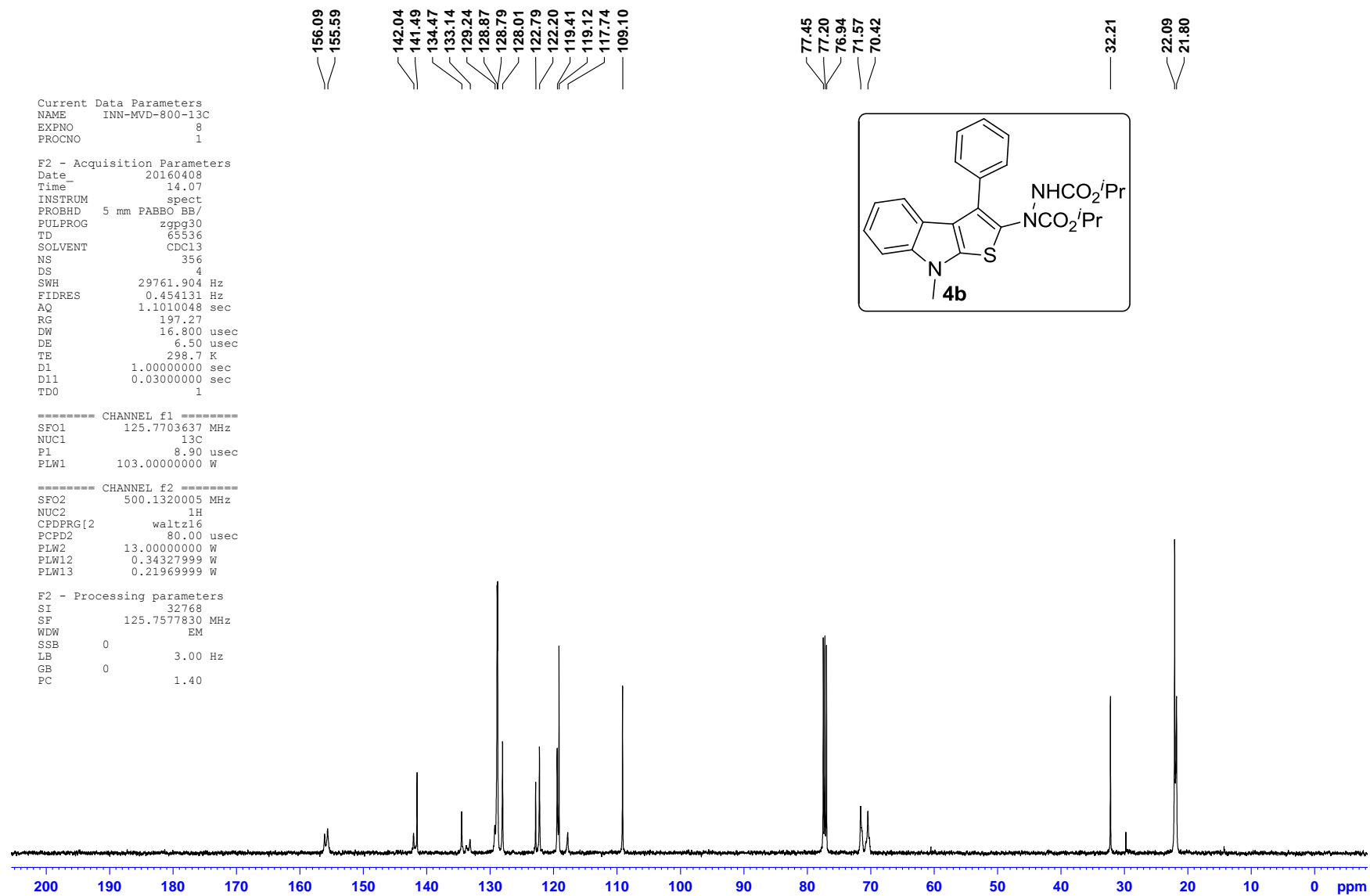


Figure S6. ¹³C NMR Spectrum of **4b**

Current Data Parameters
NAME INN-MVD-789-1H
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160414
Time_ 19.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 24
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 69.35
DW 50.000 usec
DE 6.50 usec
TE 299.9 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 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

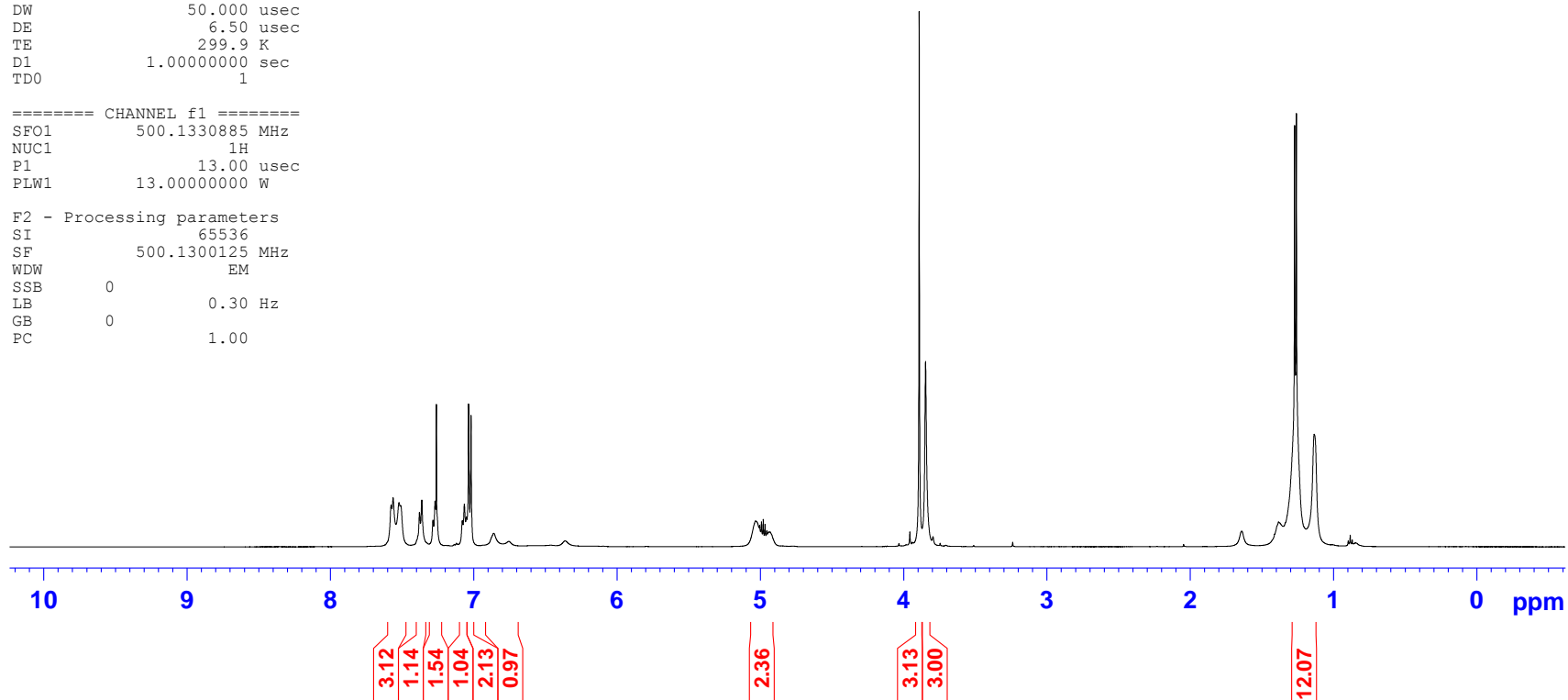
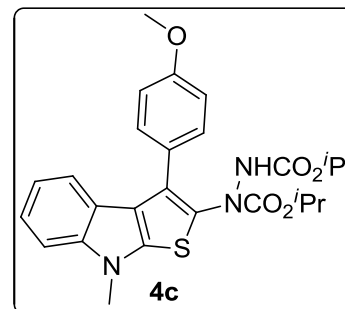


Figure S7. ¹H NMR Spectrum of 4c

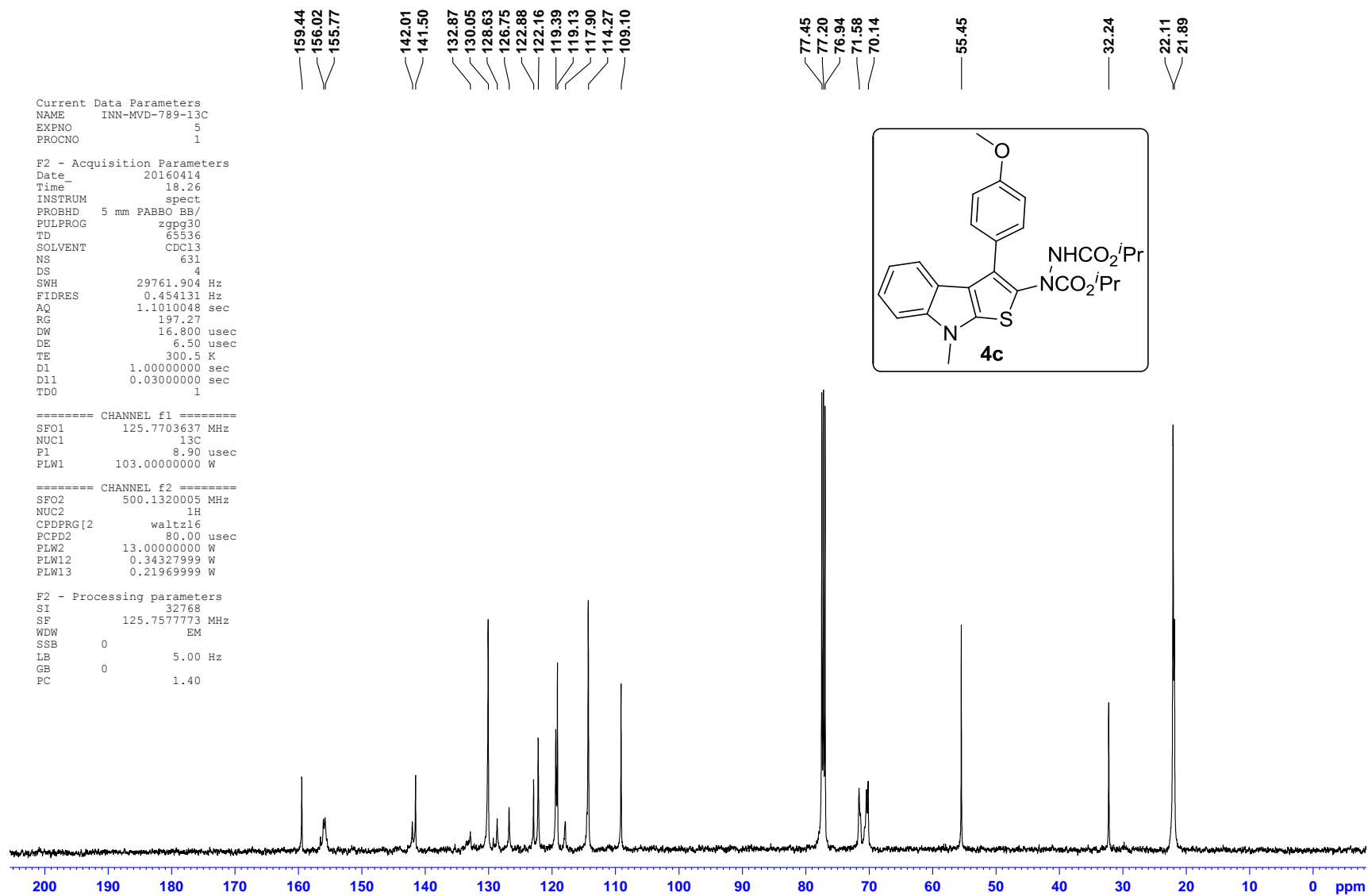


Figure S8. ^{13}C NMR Spectrum of **4c**

Current Data Parameters
NAME INN-MVD-791-1H
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160322
Time_ 12.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.52
DW 50.000 usec
DE 6.50 usec
TE 294.8 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 500.1300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

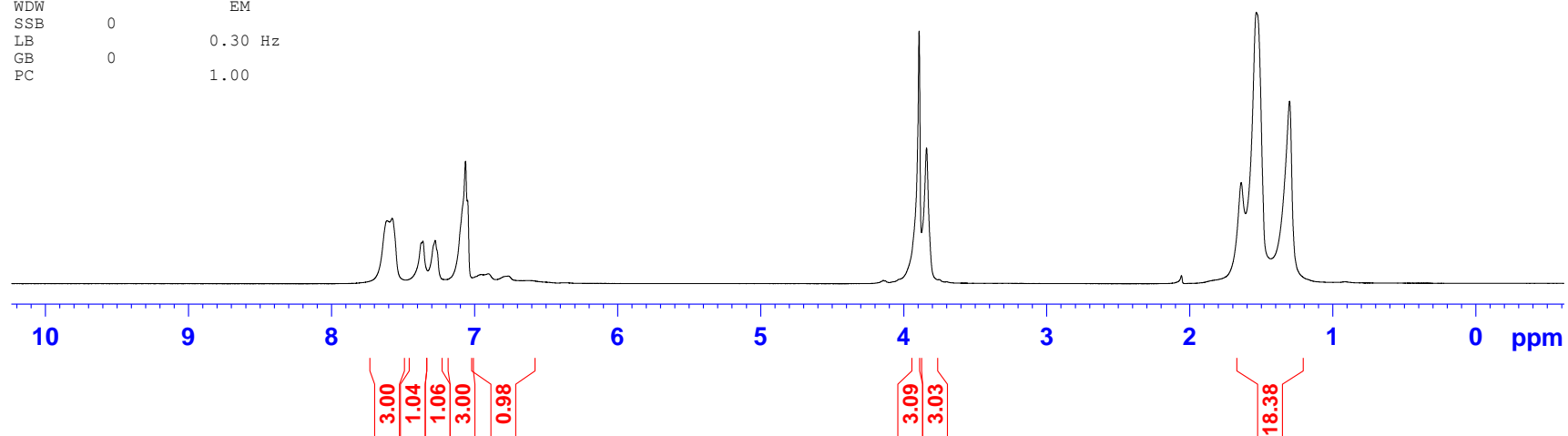
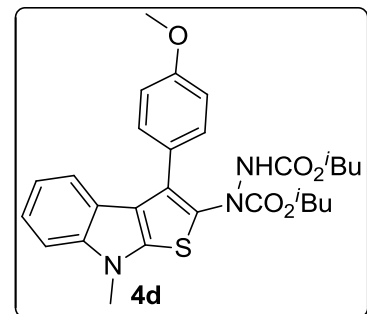


Figure S9. ¹H NMR Spectrum of 4d

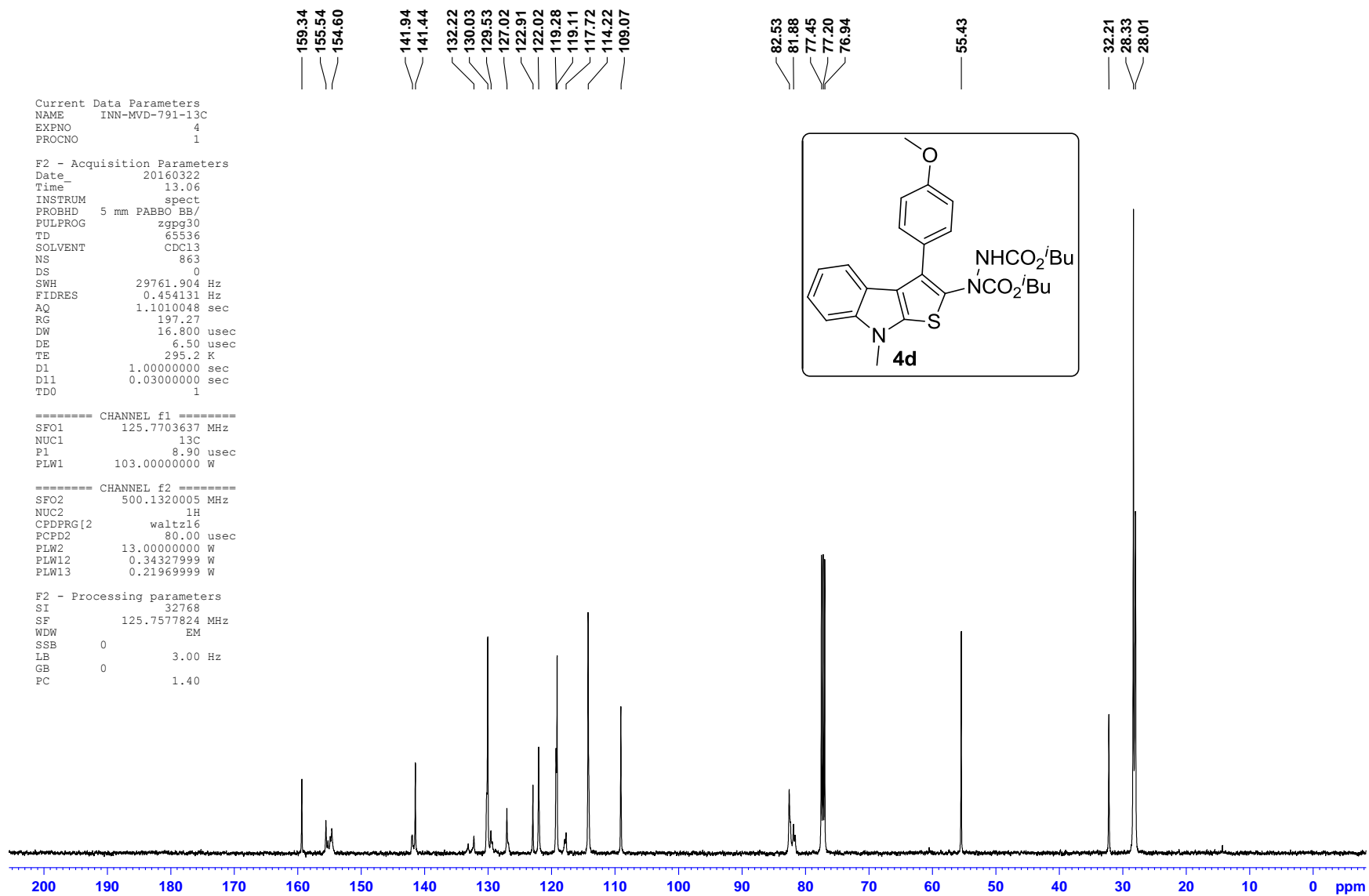


Figure S10. ^{13}C NMR Spectrum of **4d**

Current Data Parameters
NAME INN-MVD-801-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160408
Time_ 13.09
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 3.2767999 sec
RG 53.37
DW 50.000 usec
DE 6.50 usec
TE 297.9 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

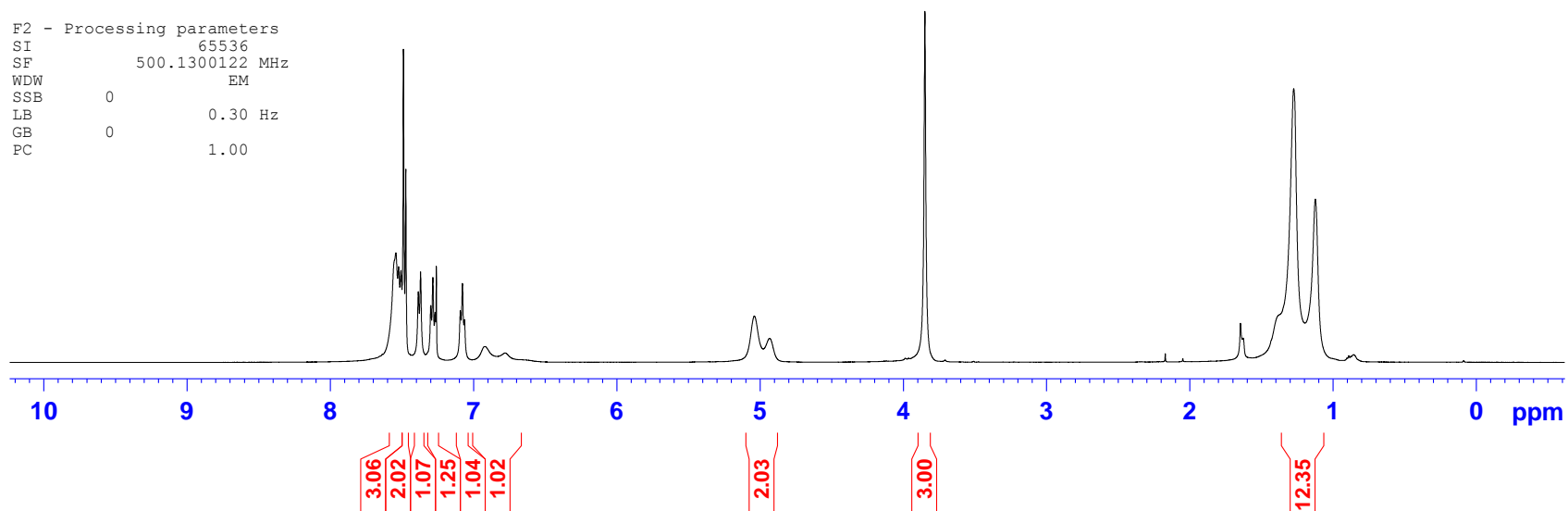
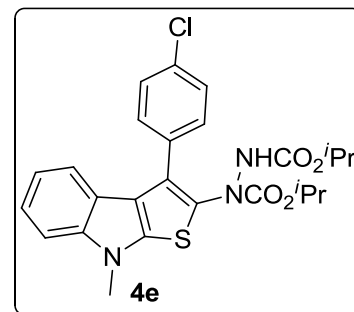


Figure S11. ¹H NMR Spectrum of **4e**

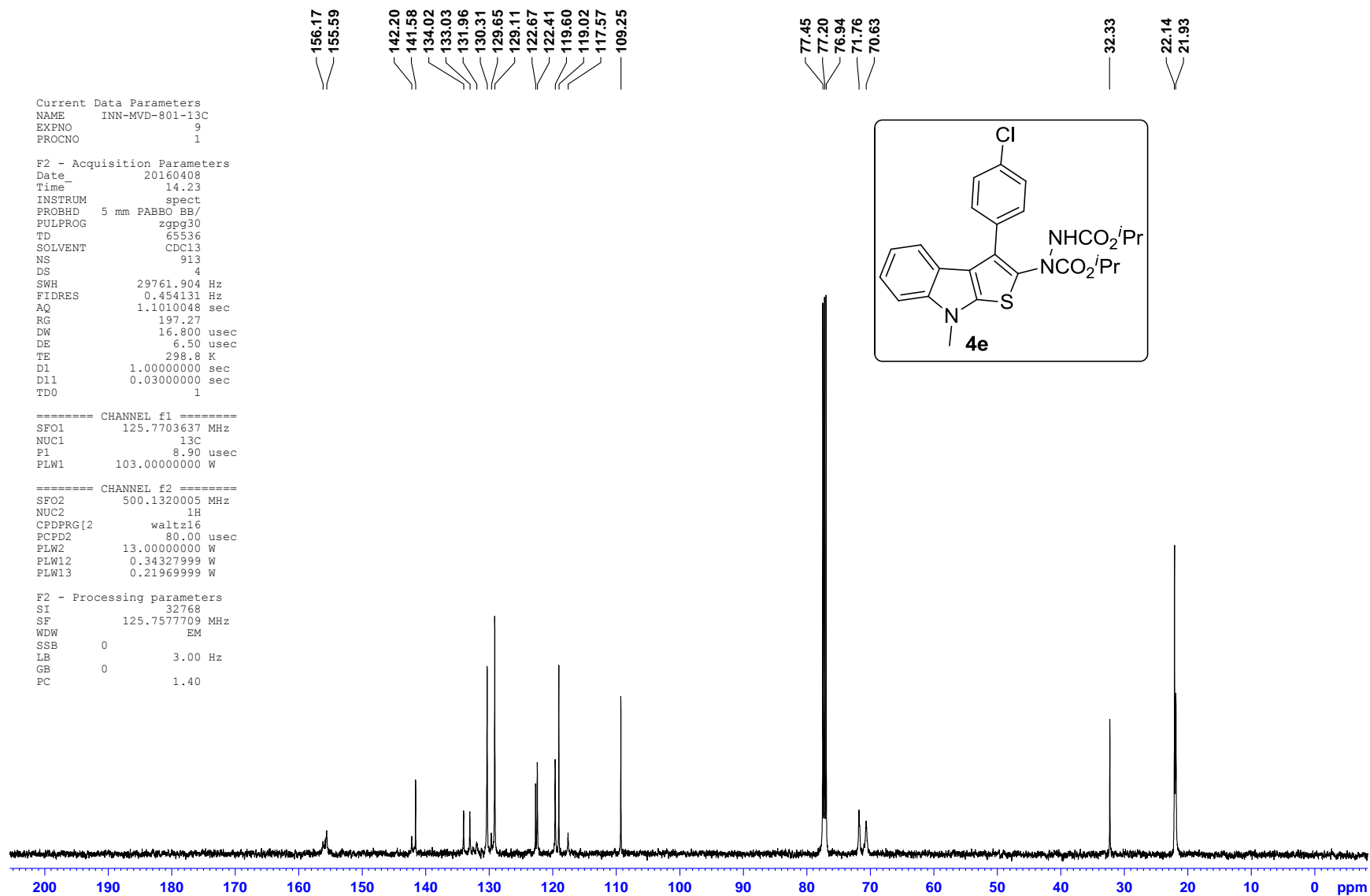


Figure S12. ^{13}C NMR Spectrum of **4e**

Current Data Parameters
NAME INN-MVD-794-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160322
Time_ 13.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 294.3 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 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

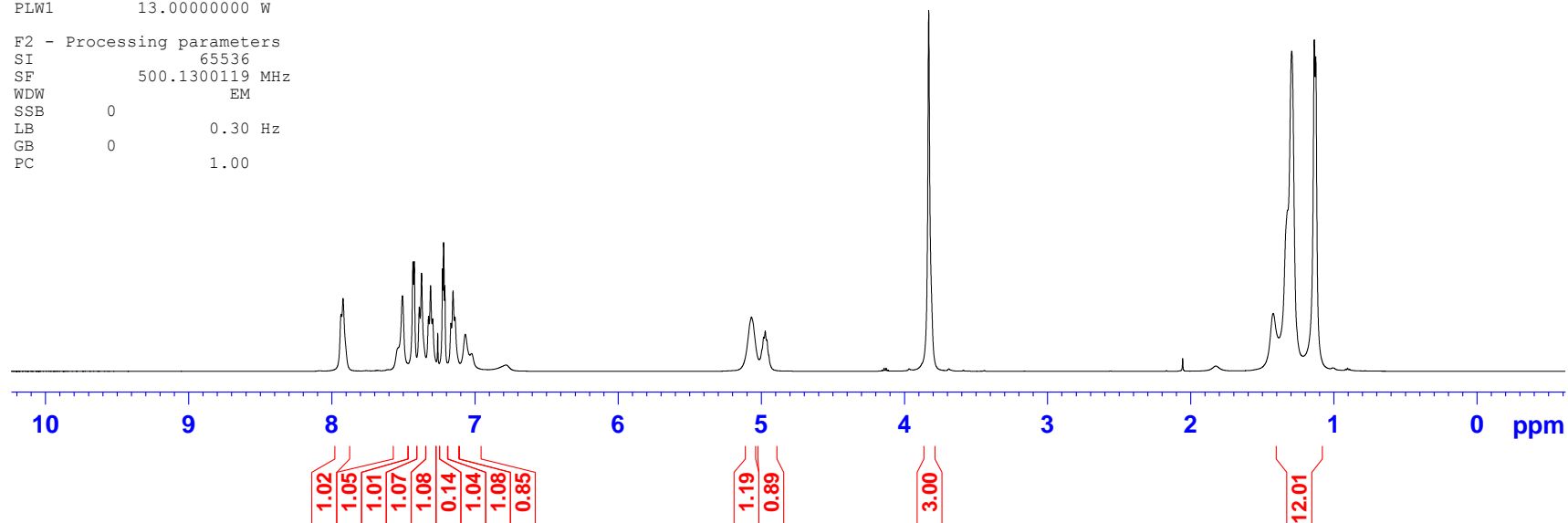
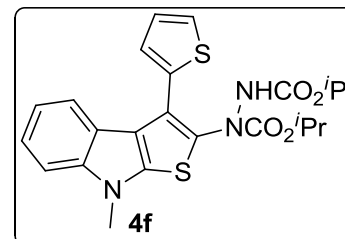


Figure S13. ¹H NMR Spectrum of 4f

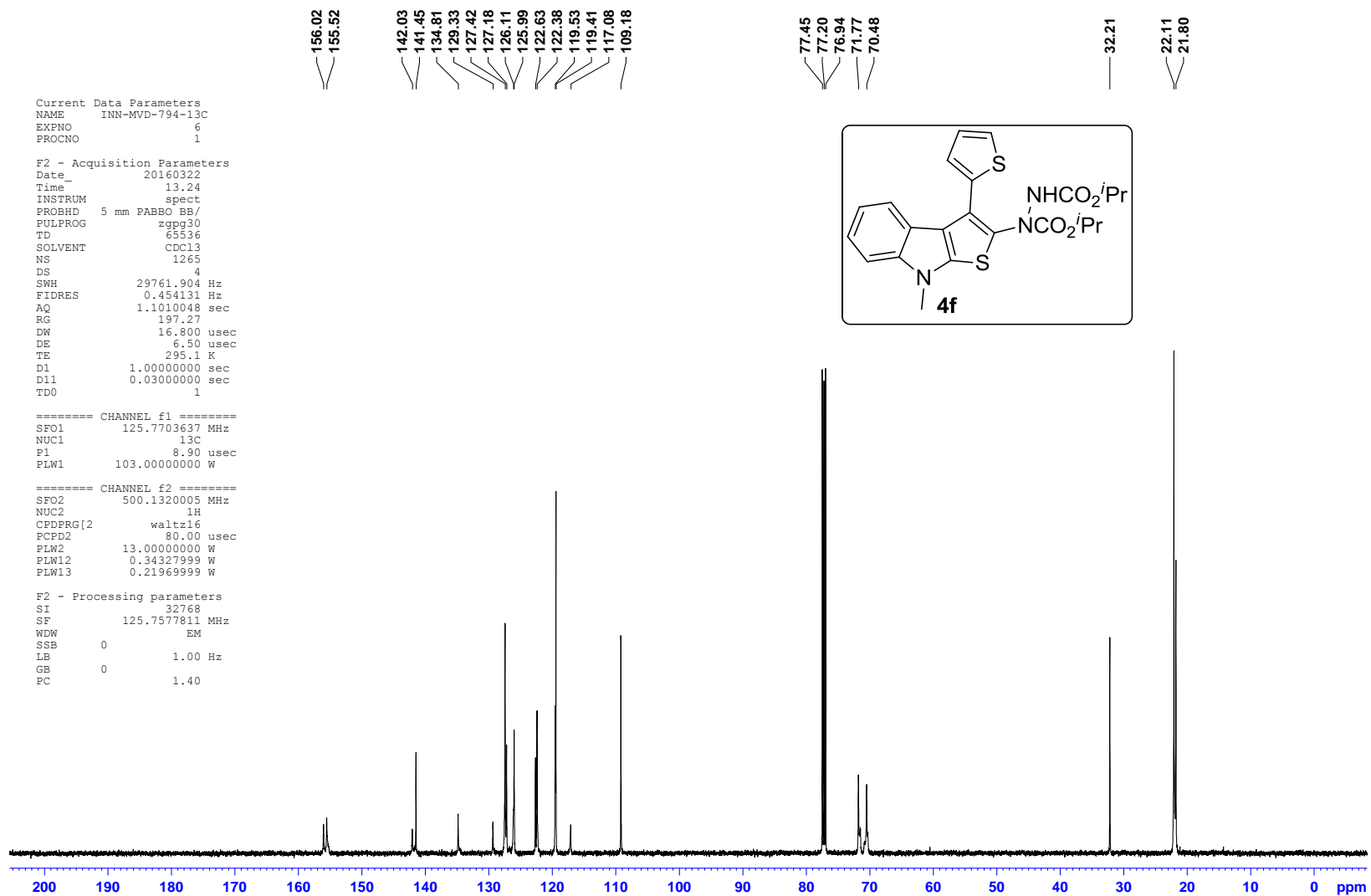


Figure S14. ¹³C NMR Spectrum of 4f

Current Data Parameters
NAME INN-MVD-802-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160414
Time_ 10.30
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 60
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 294.6 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

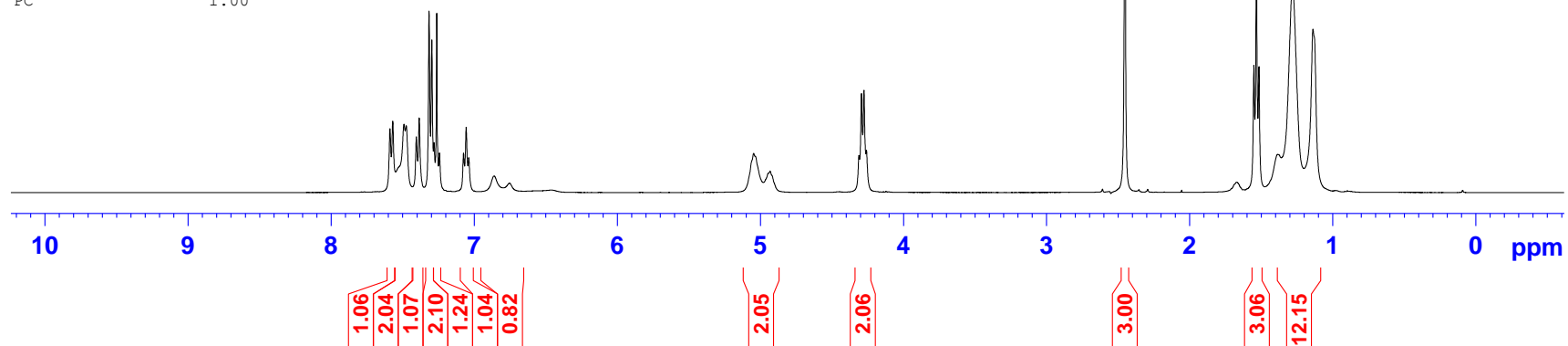


Figure S15. ¹H NMR Spectrum of 4g

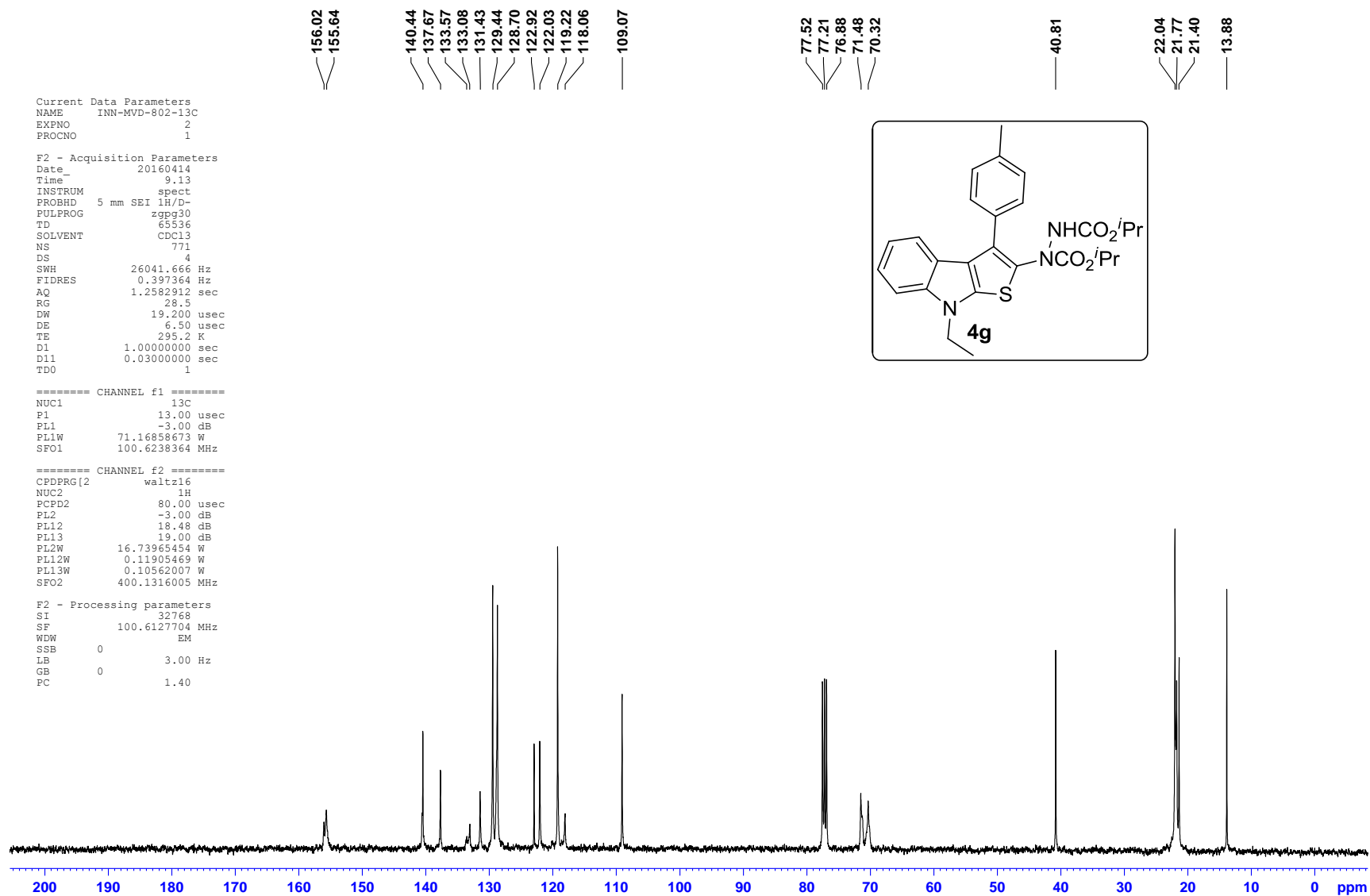


Figure S16. ¹³C NMR Spectrum of 4g

Current Data Parameters
NAME INN-MVD-796-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160403
Time_ 11.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 19
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 15.73
DW 50.000 usec
DE 6.50 usec
TE 294.8 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 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

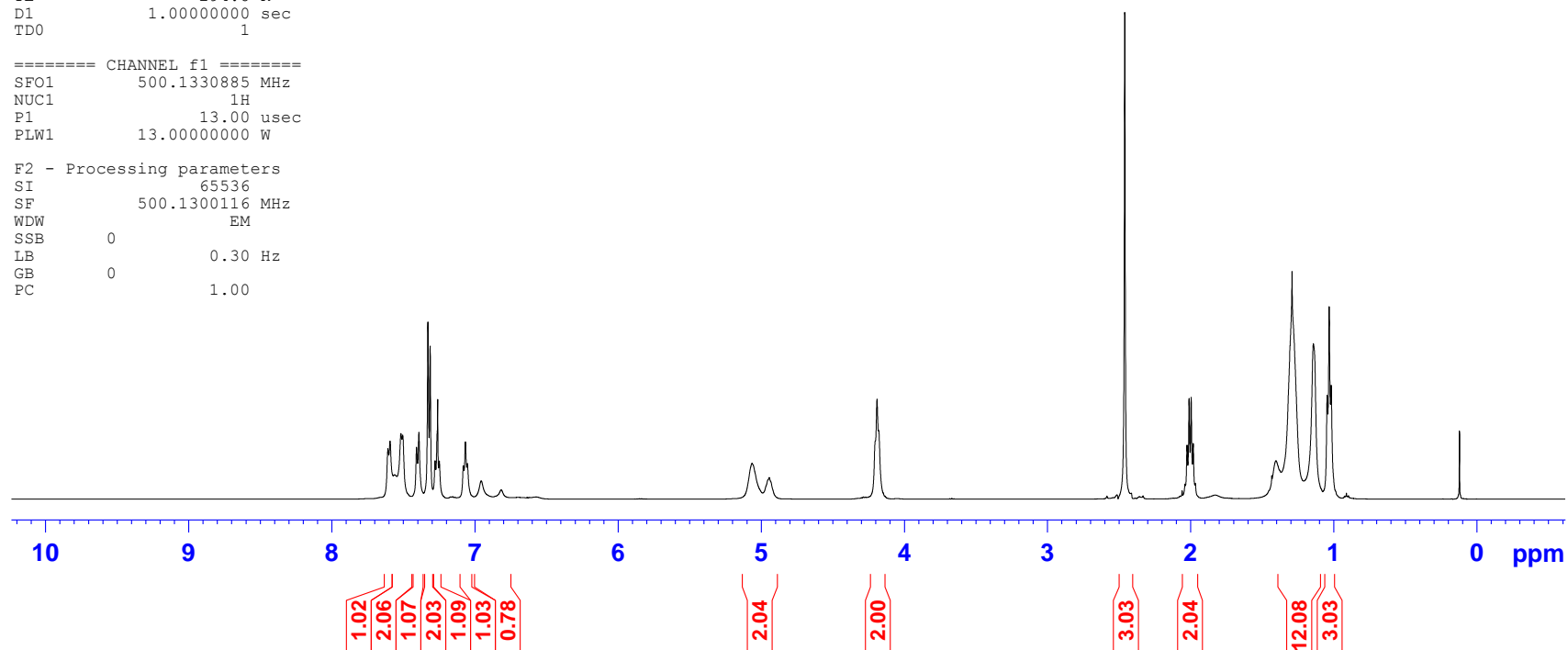
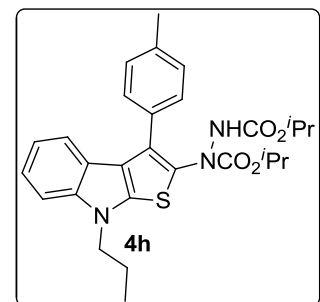


Figure S17. ¹H NMR Spectrum of 4h

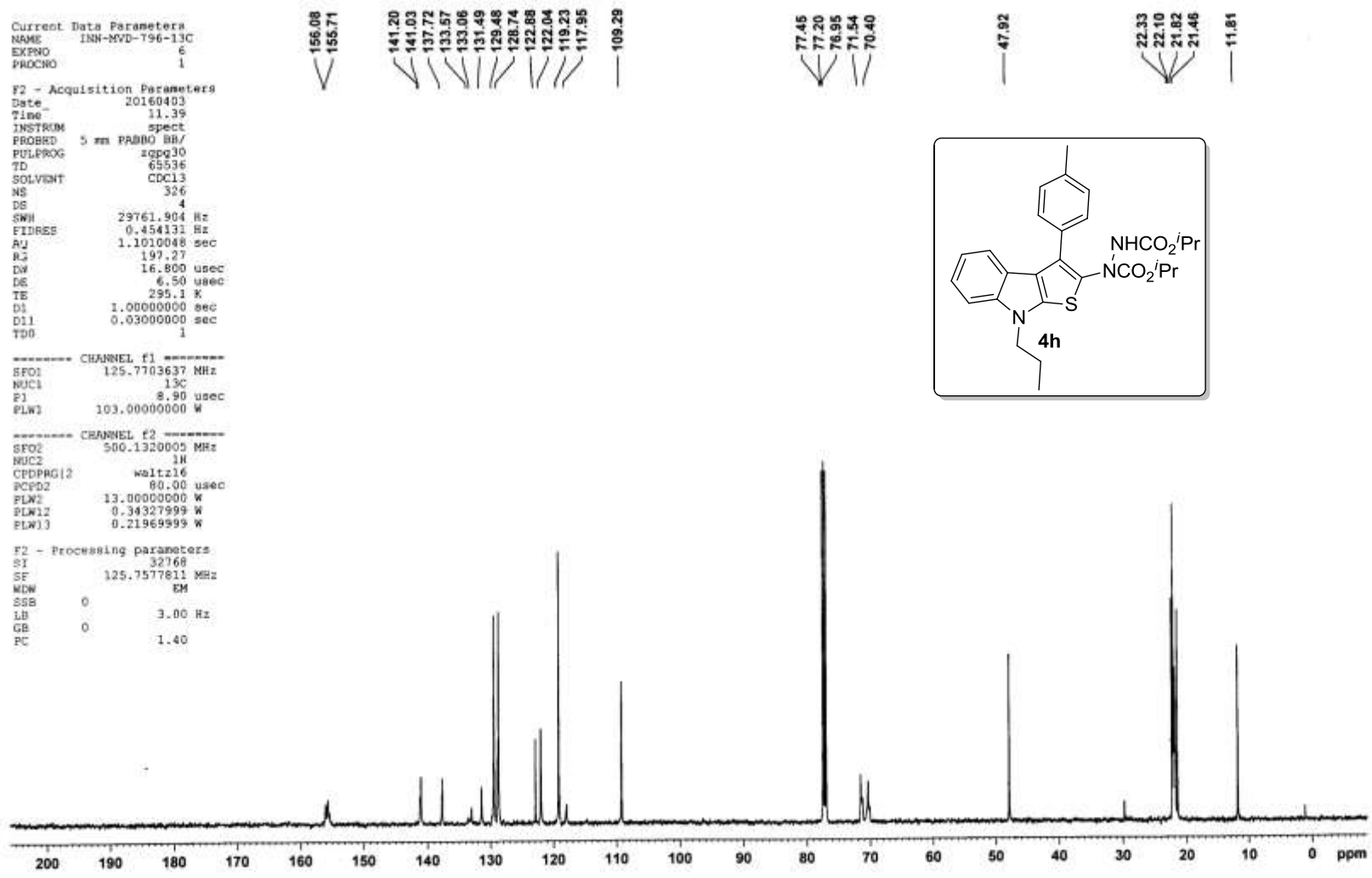


Figure S18. ¹³C NMR Spectrum of 4h

Current Data Parameters
NAME INN-MVD-803-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160417
Time_ 8.39
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 18
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 12.7
DW 60.800 usec
DE 6.50 usec
TE 293.9 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 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

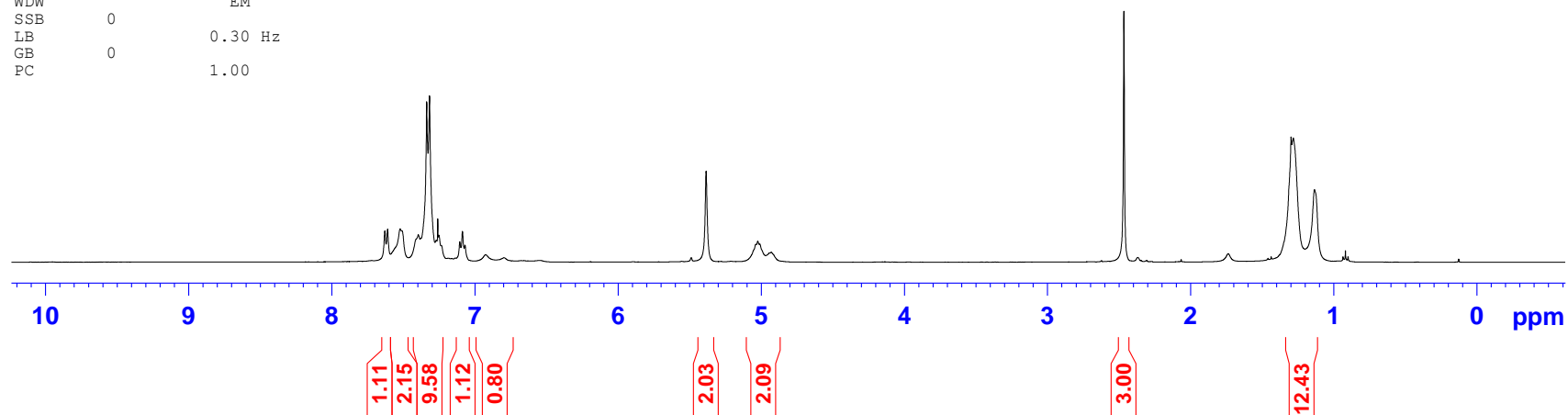
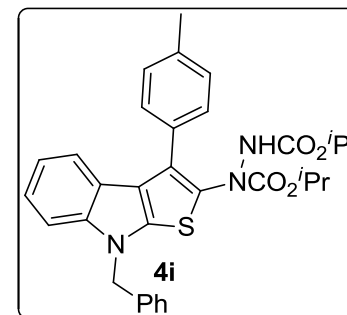


Figure S19. ¹H NMR Spectrum of 4i

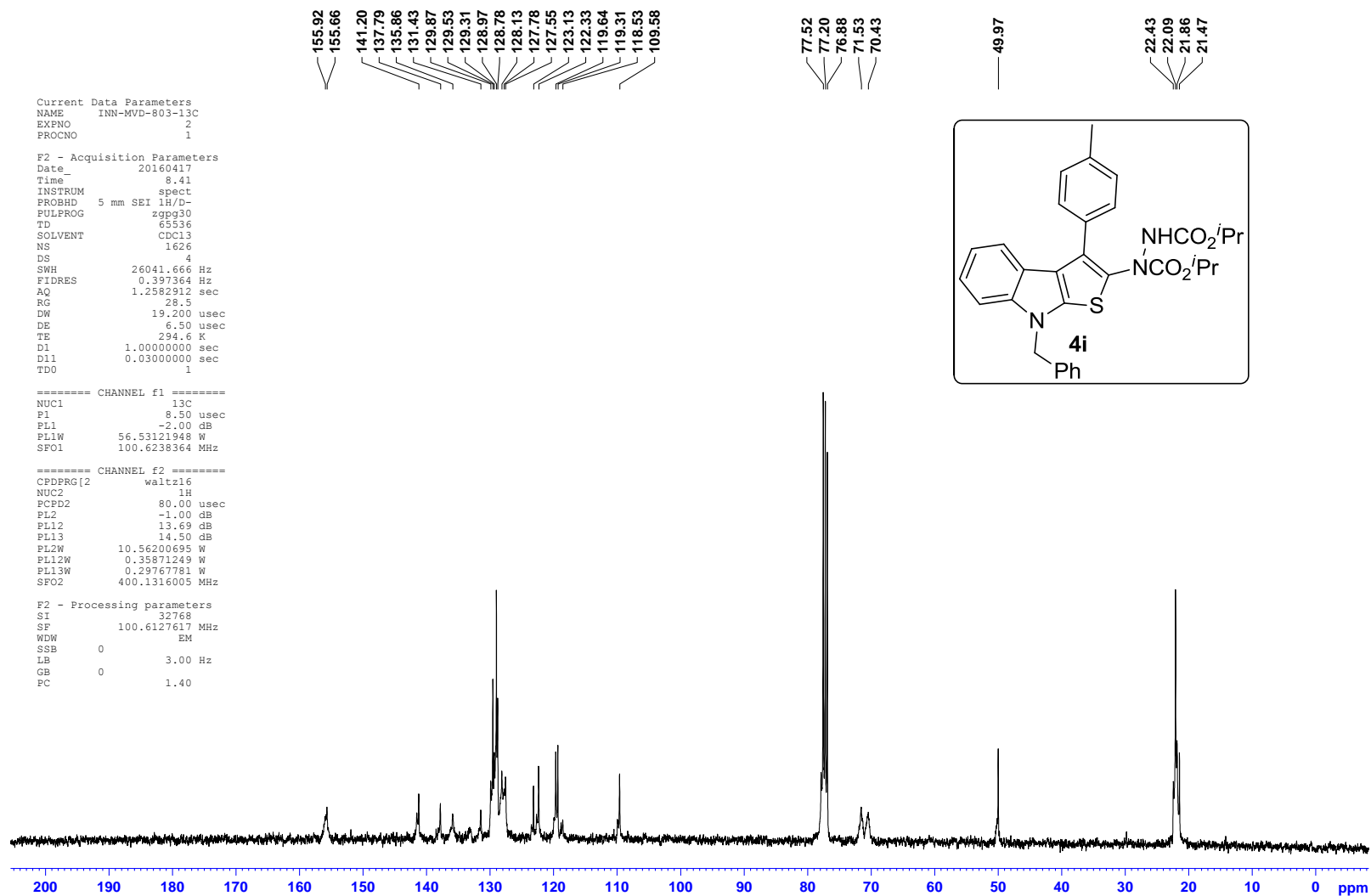


Figure S20. ¹³C NMR Spectrum of 4i

Current Data Parameters
NAME INN-MVD-805-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160712
Time_ 10.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 69.35
DW 50.000 usec
DE 6.50 usec
TE 294.9 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 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

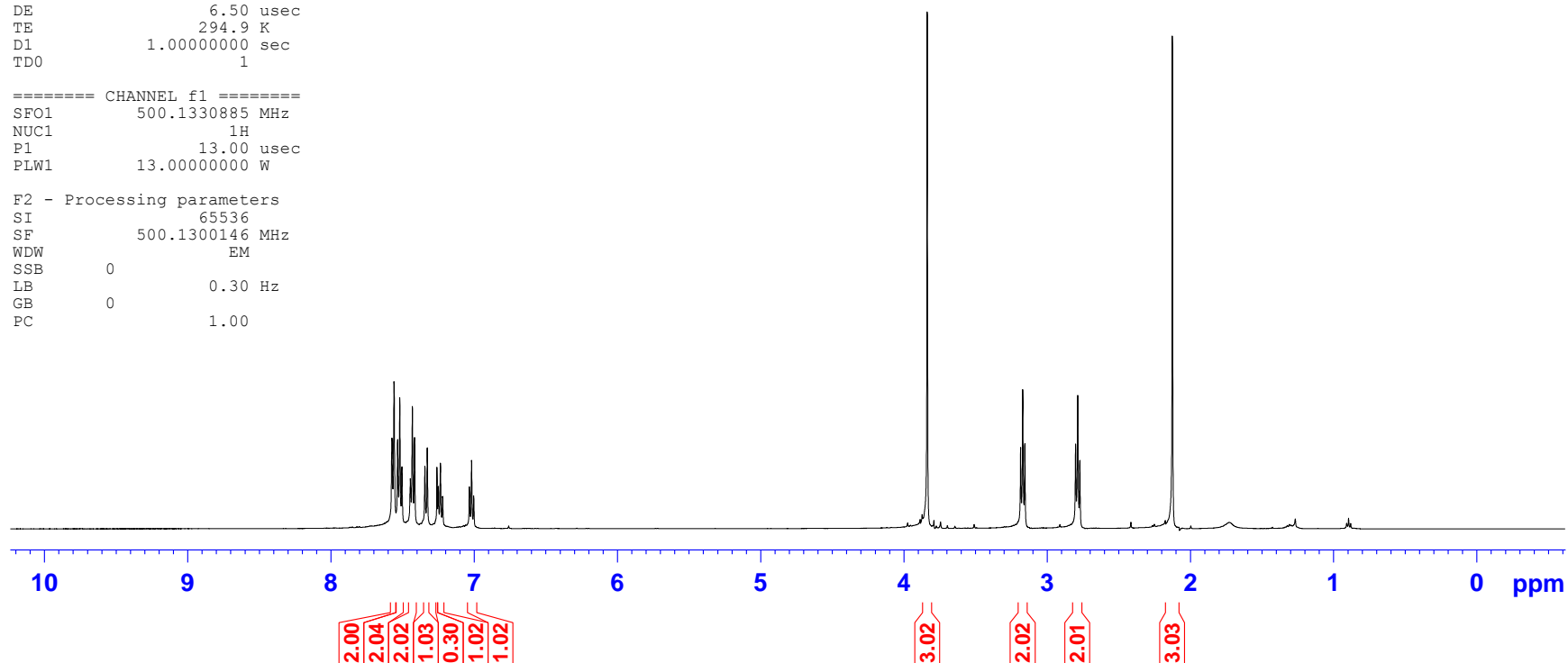
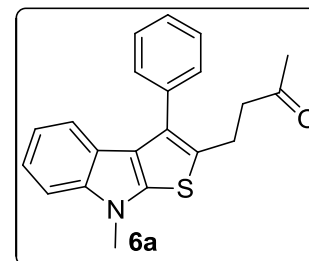


Figure S21. ¹H NMR Spectrum of 6a

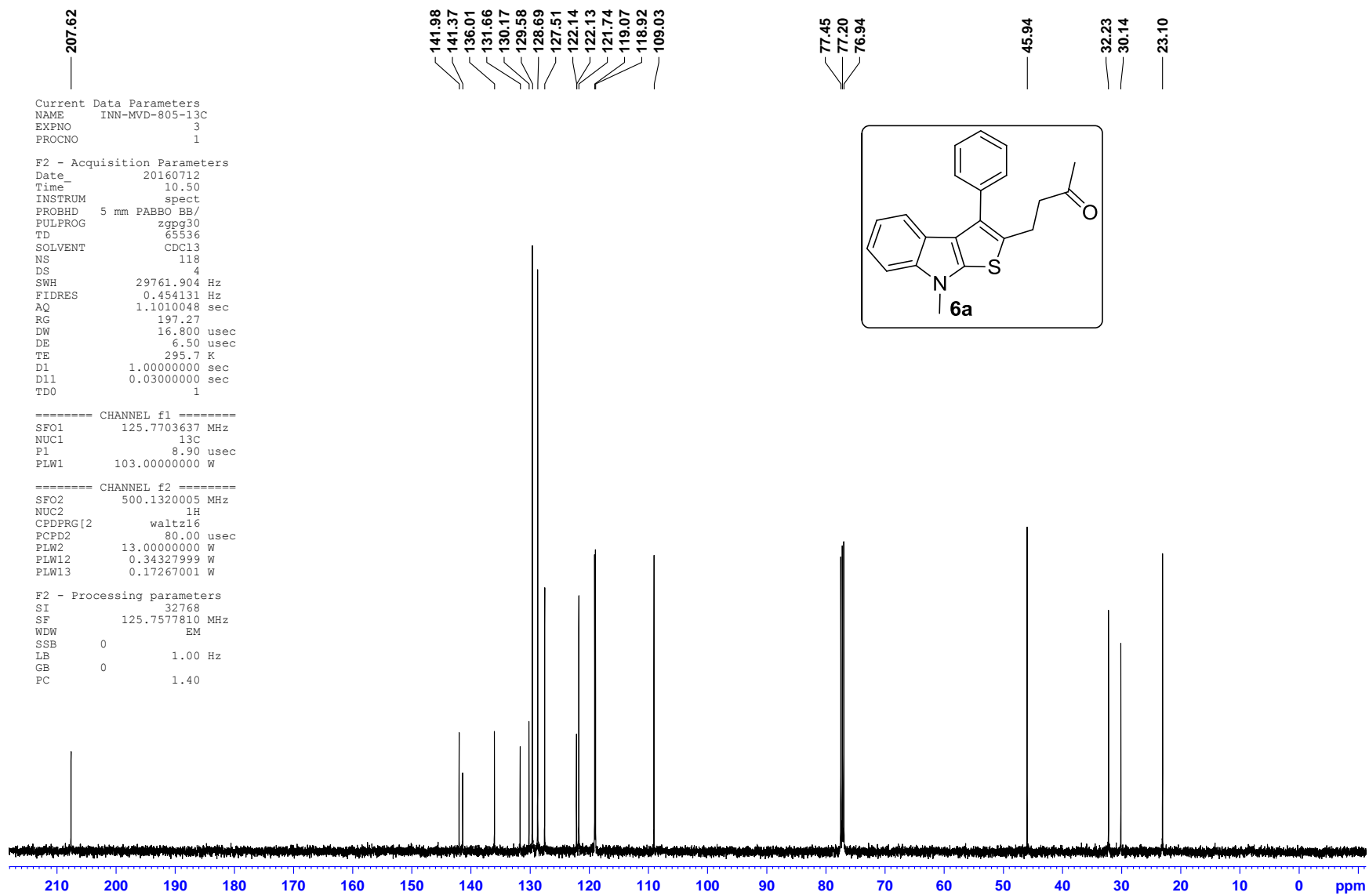


Figure S22. ^{13}C NMR Spectrum of **6a**


```

Current Data Parameters
NAME      INN-MVD-596-1H
EXPNO    3
PROCNO    1

F2 - Acquisition Parameters
Date_     20150813
Time      16.26
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        3.2767999 sec
RG        80.35
DW        50.000 usec
DE        6.50 usec
TE        298.6 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        500.1300123 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

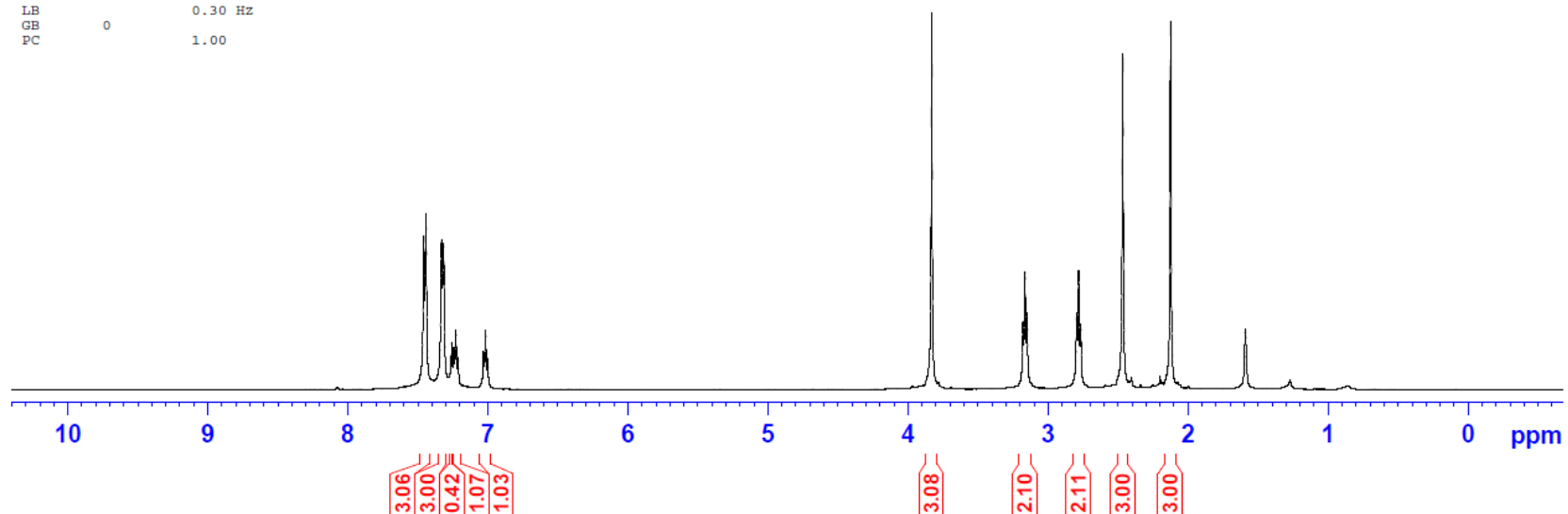
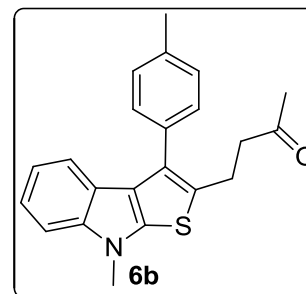


Figure S23. ¹H NMR Spectrum of **6b**

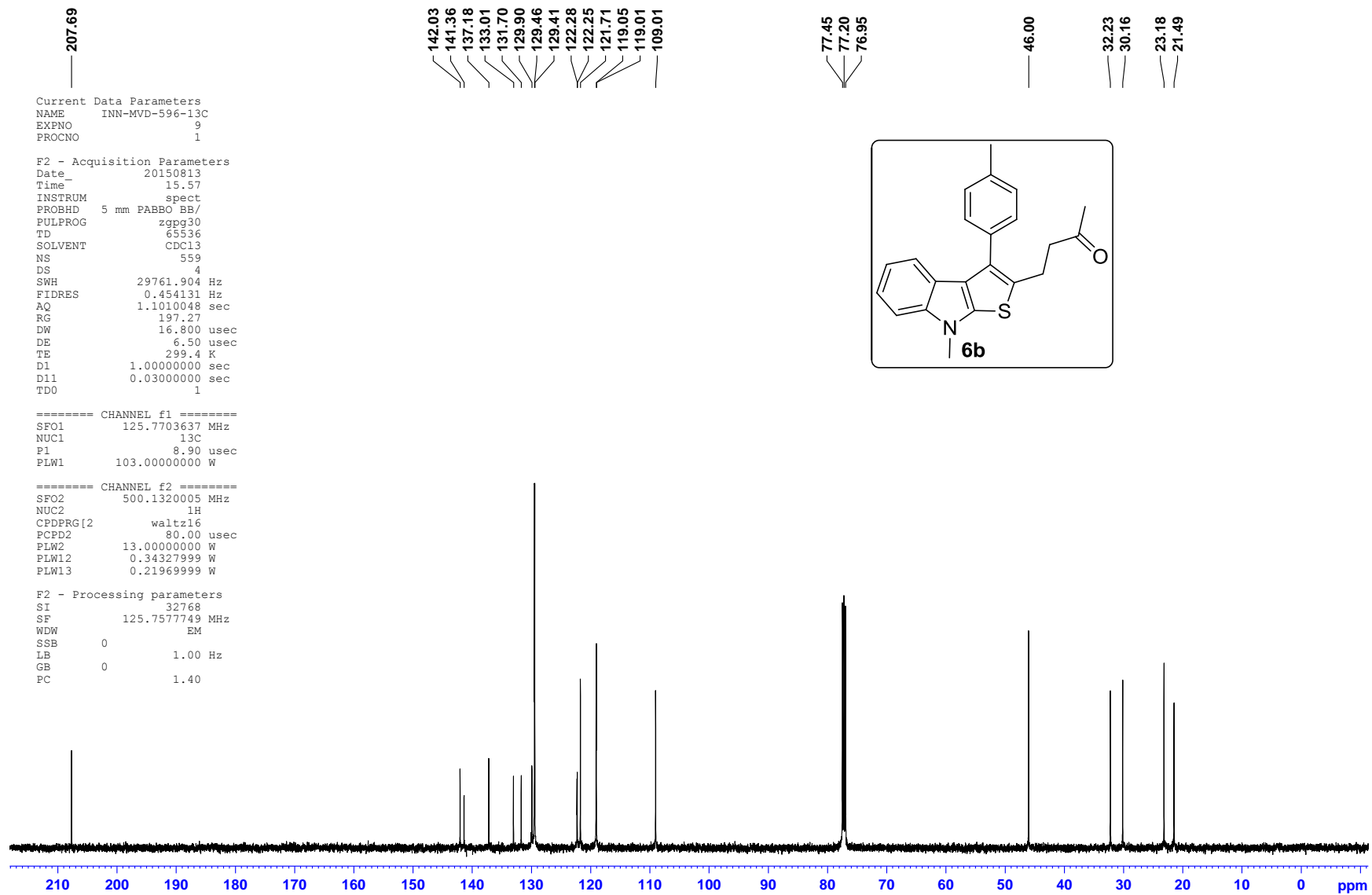


Figure S24. ^{13}C NMR Spectrum of **6b**

Current Data Parameters
NAME INN-MVD-614-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150823
Time_ 4.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 25
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 296.2 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 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

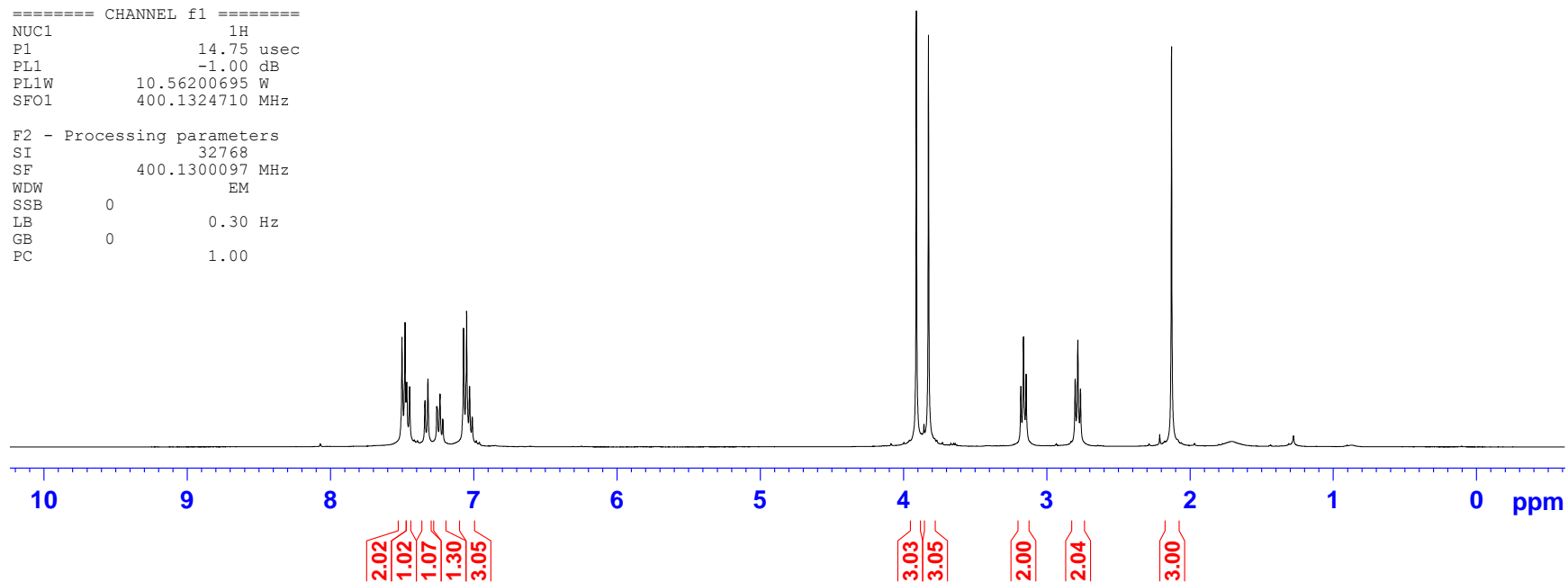
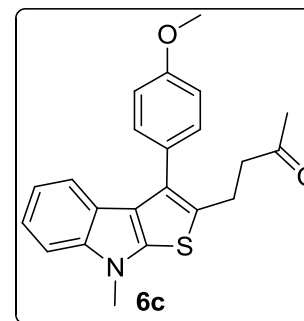


Figure S25. ¹H NMR Spectrum of **6c**

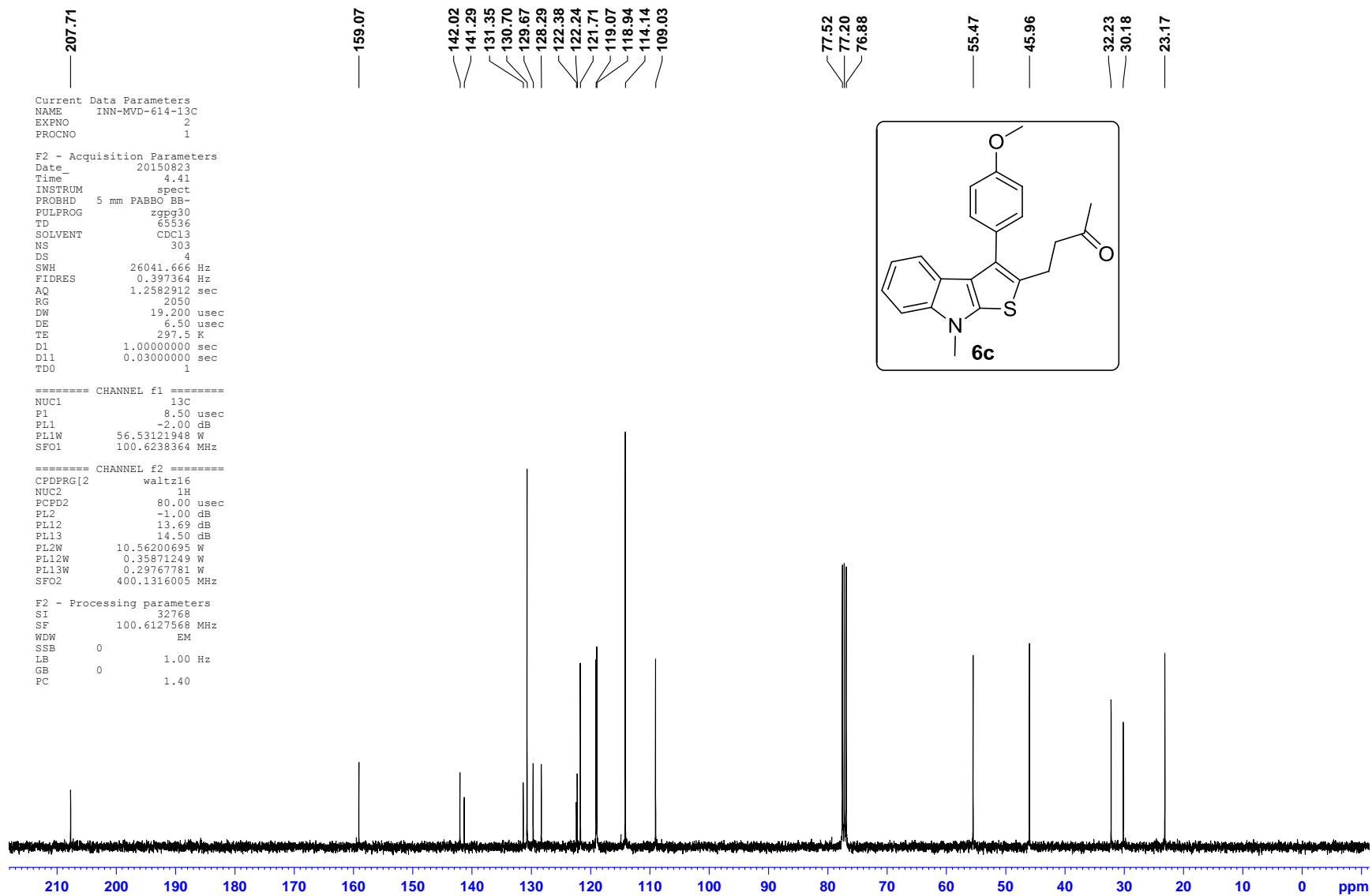


Figure S26. ¹³C NMR Spectrum of **6c**

Current Data Parameters
NAME INN-MVD-641-1H
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150917
Time_ 19.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 293.9 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 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

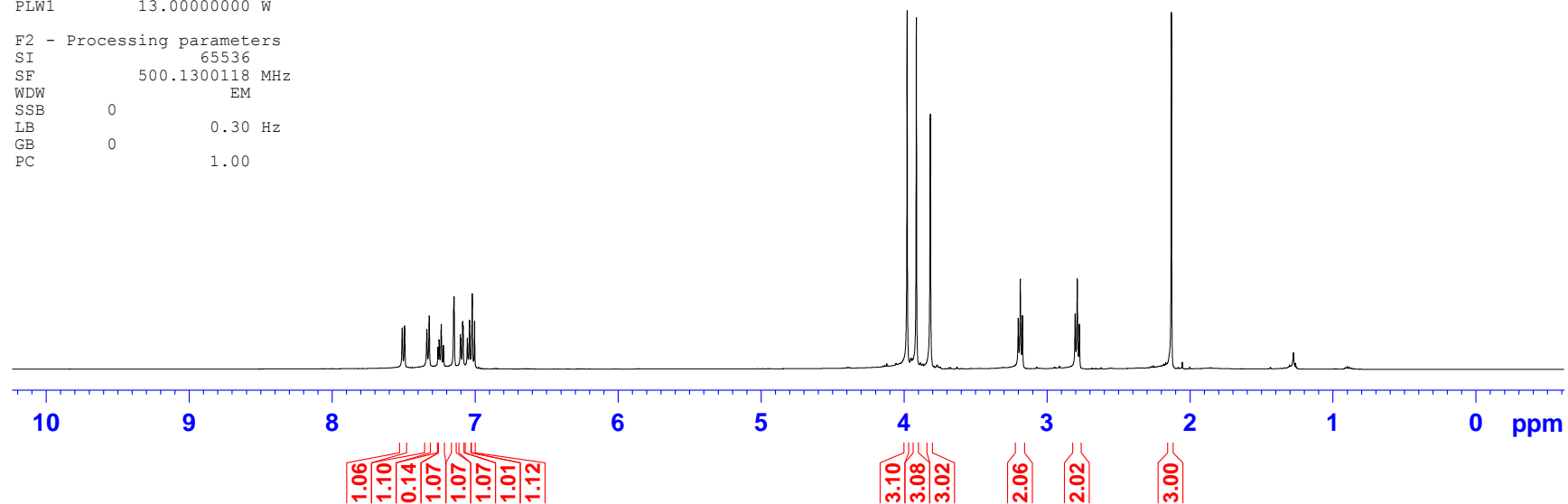
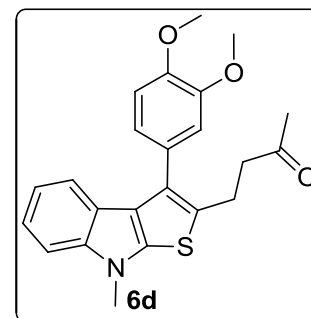


Figure S27. ¹H NMR Spectrum of **6d**

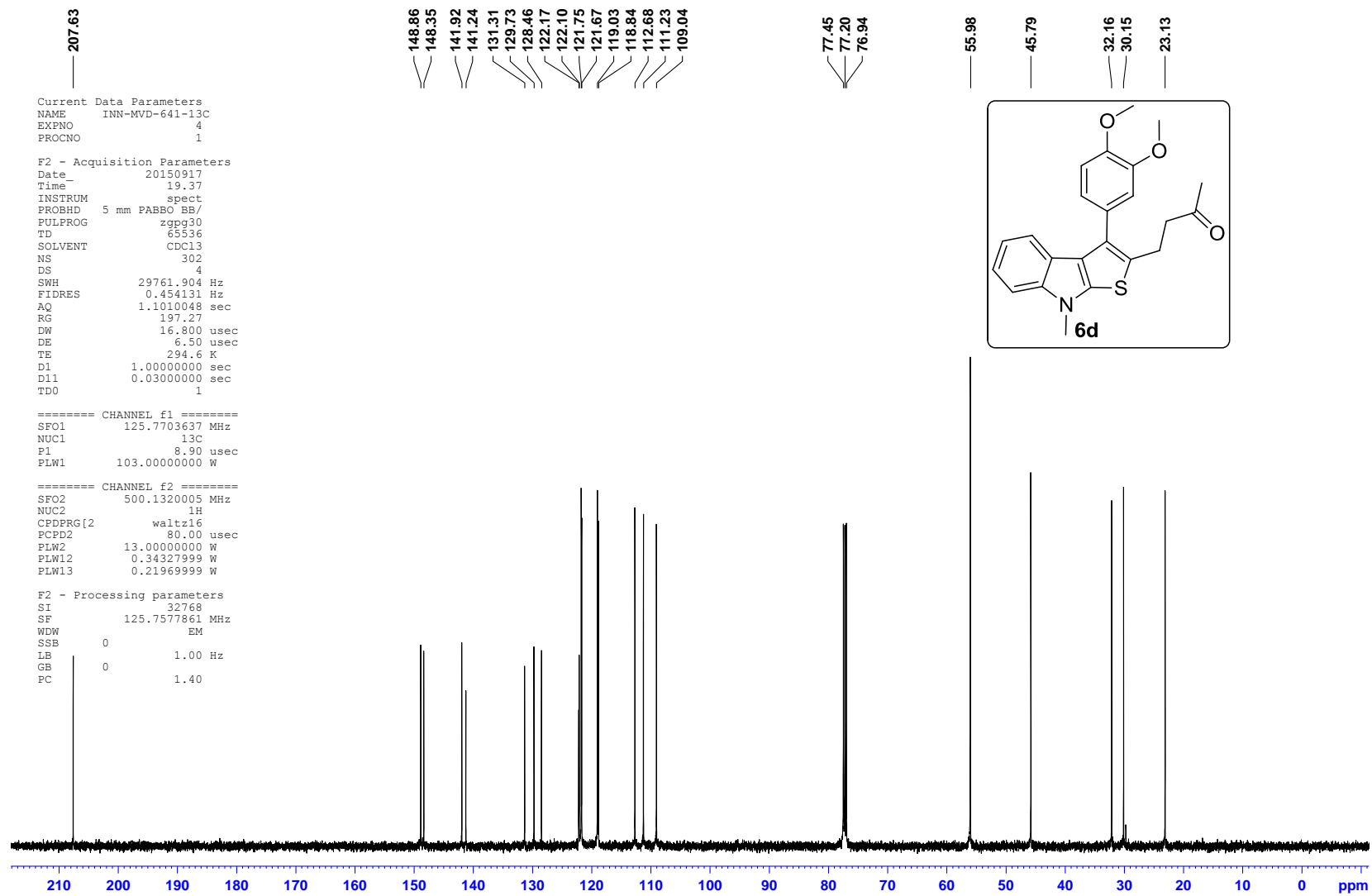


Figure S28. ^{13}C NMR Spectrum of 6d

Current Data Parameters
NAME INN-MVD--612-1H
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161109
Time 14.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 9
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 69.35
DW 50.000 usec
DE 6.50 usec
TE 296.8 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 500.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

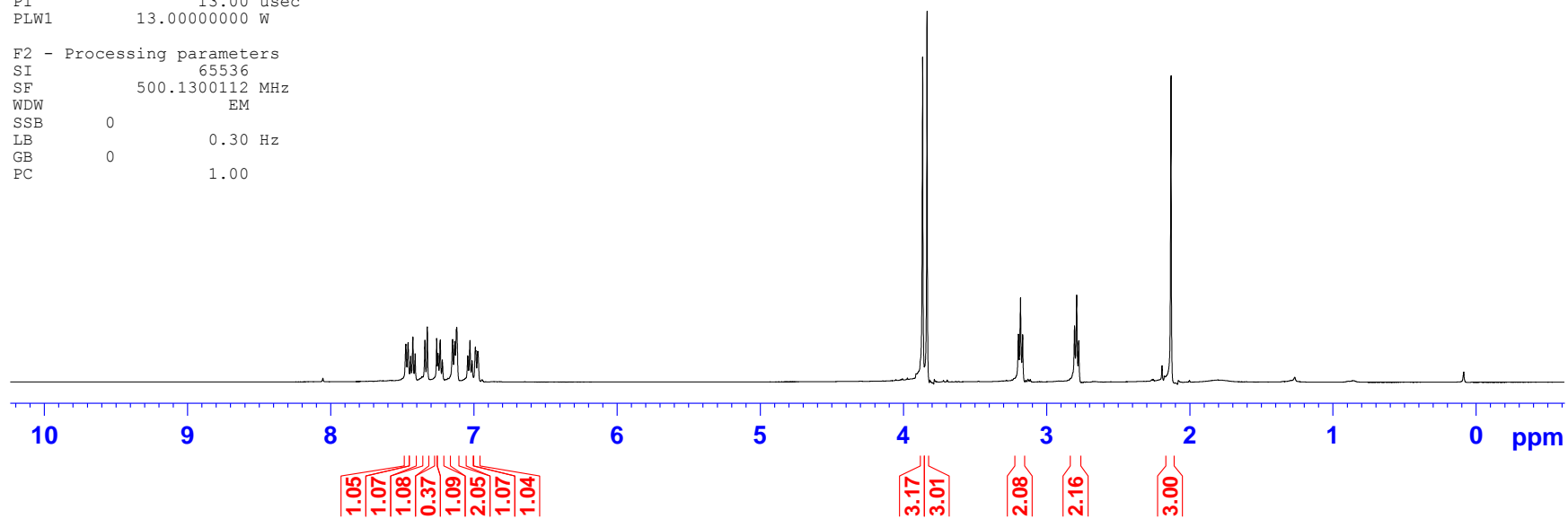
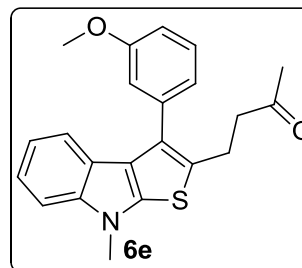


Figure S29. ¹H NMR Spectrum of 6e

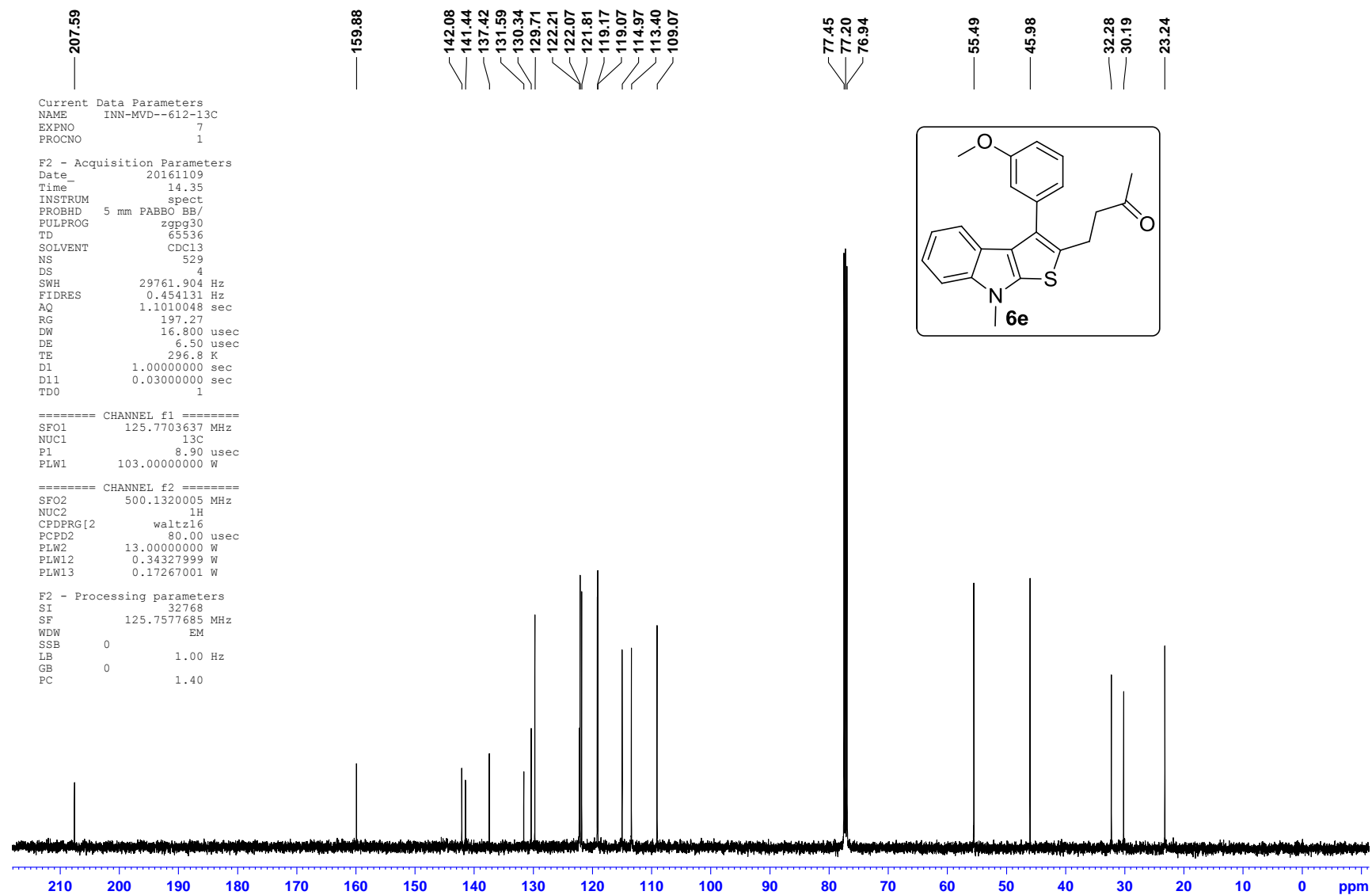


Figure S30. ^{13}C NMR Spectrum of **6e**

Current Data Parameters
NAME INN-MVD-638-1H
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150918
Time_ 18.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 15.73
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 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

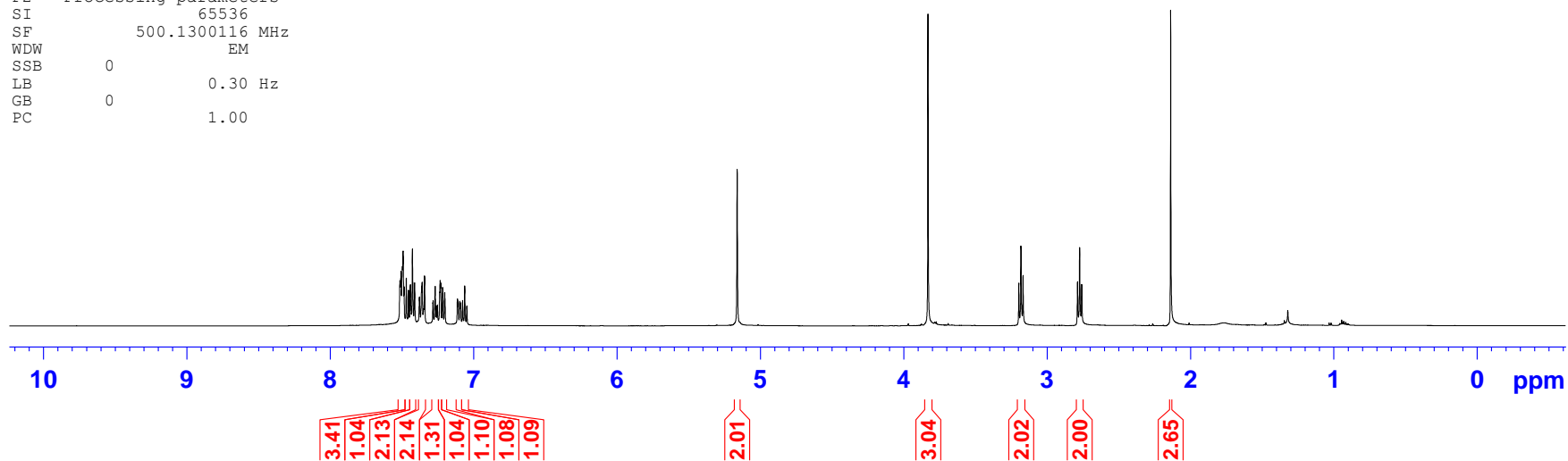
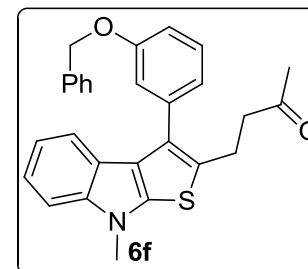


Figure S31. ¹H NMR Spectrum of **6f**

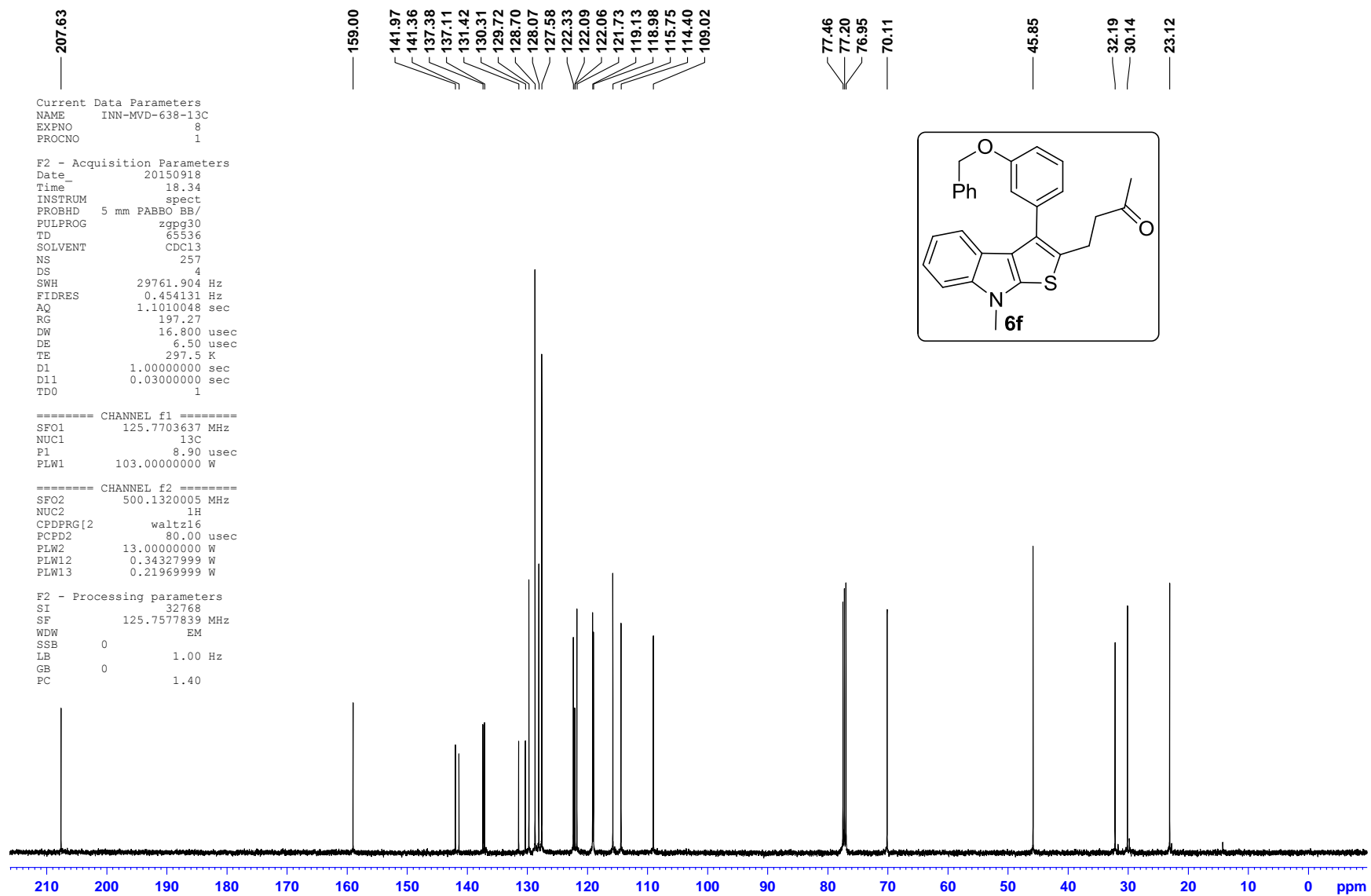


Figure S32. ^{13}C NMR Spectrum of **6f**

Current Data Parameters
NAME INN-MVD-639-1H
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150917
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 54274
SOLVENT CDC13
NS 21
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 301.8 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 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

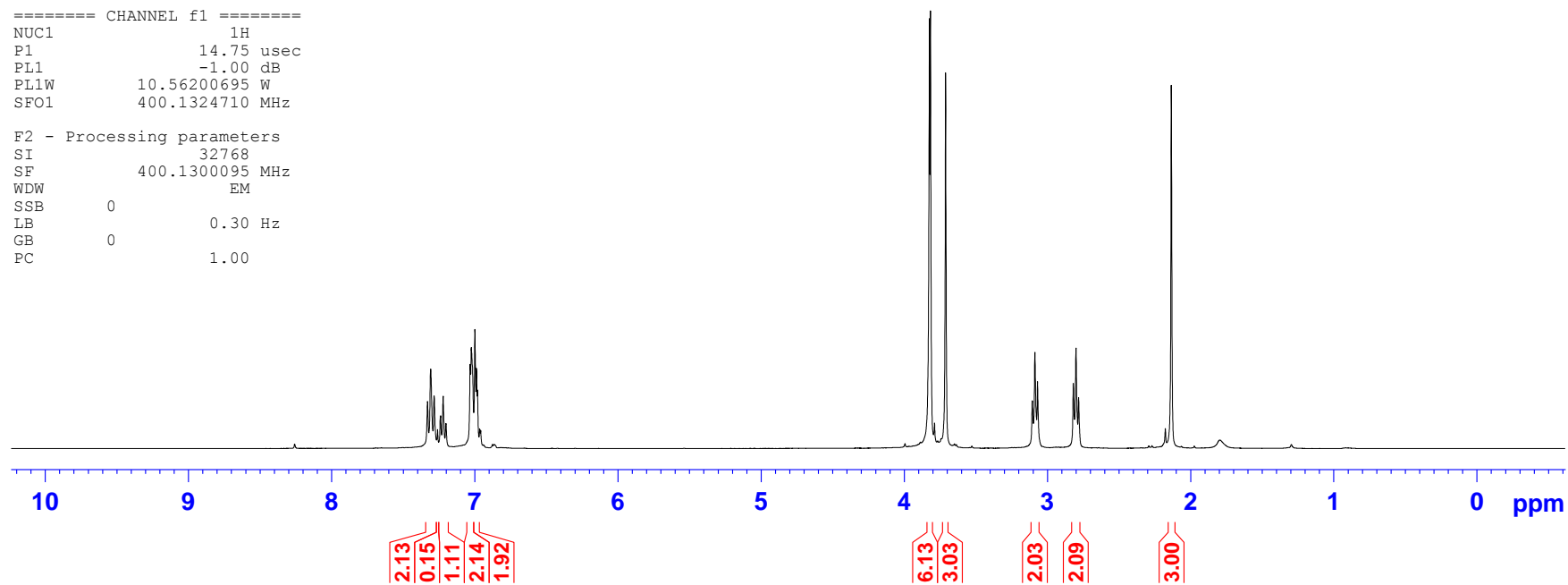
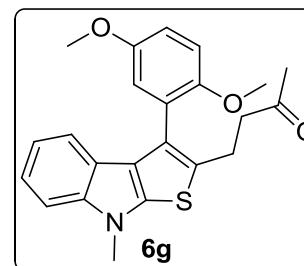


Figure S33. ¹H NMR Spectrum of **6g**

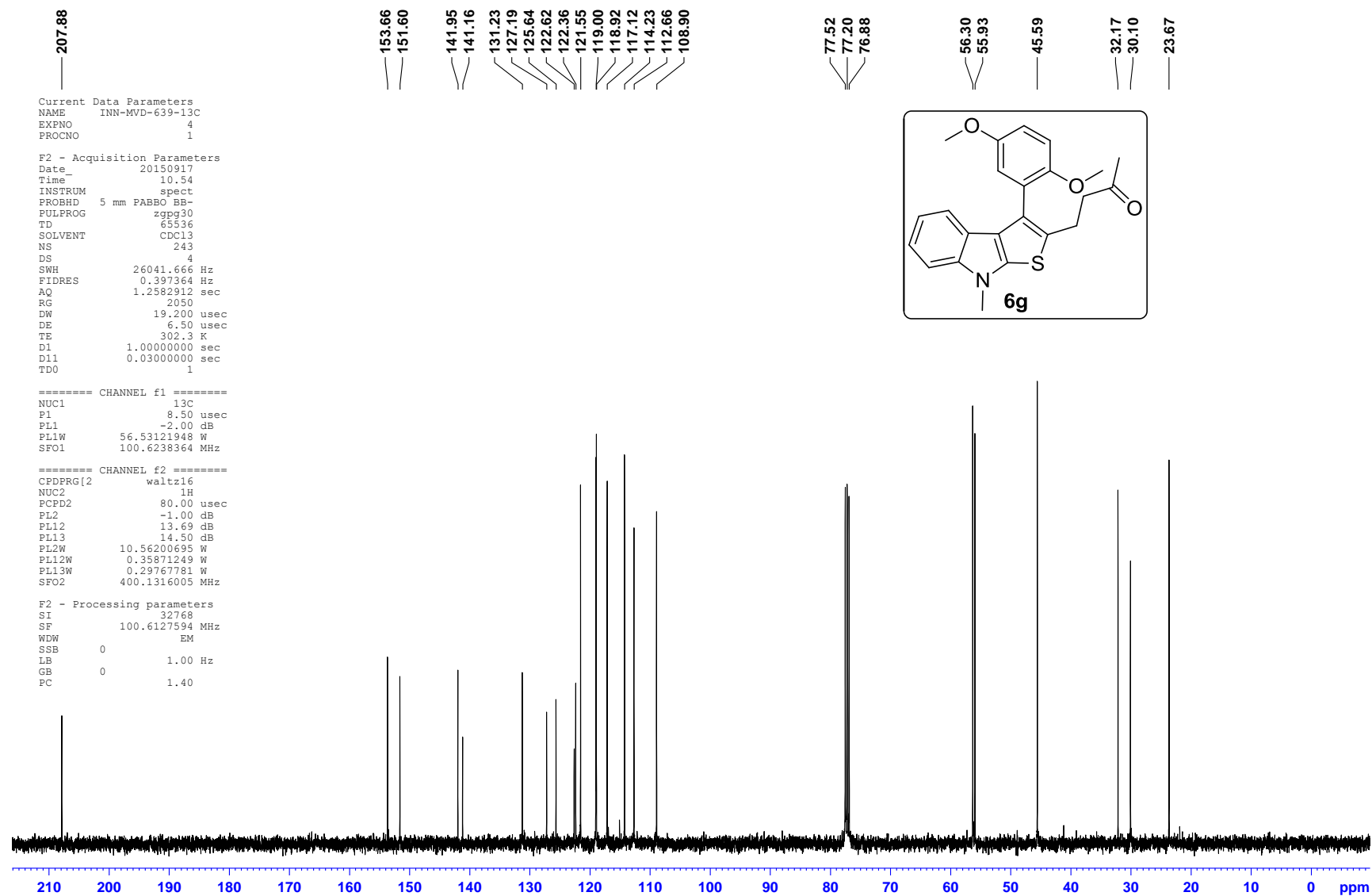


Figure S34. ^{13}C NMR Spectrum of **6g**

Current Data Parameters
NAME INN-MVD-611-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161109
Time_ 14.23
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 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 297.5 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 500.1300208 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

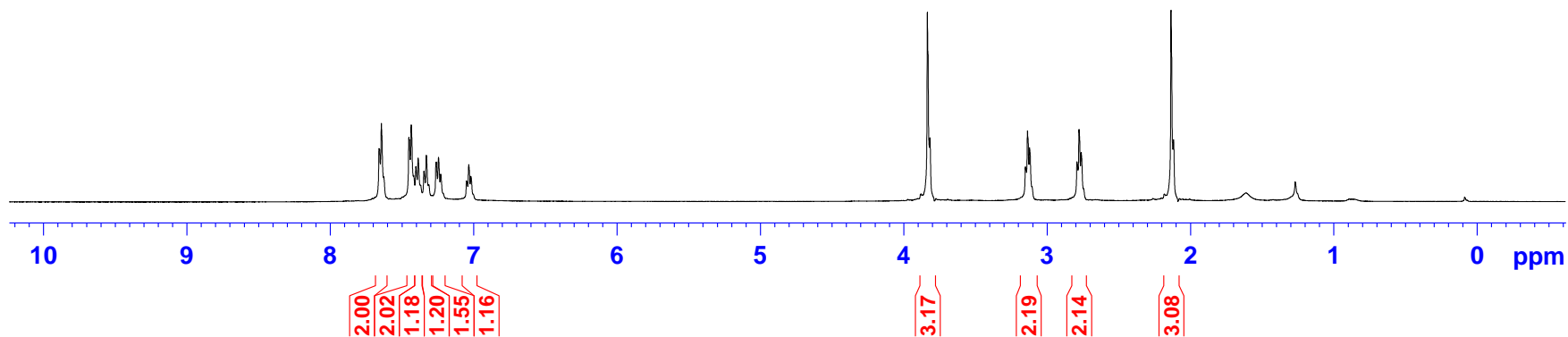
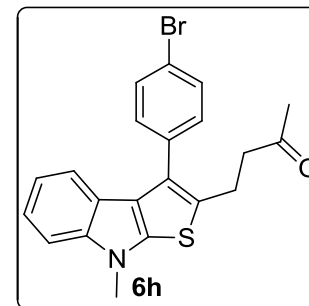


Figure S35. ¹H NMR Spectrum of **6h**

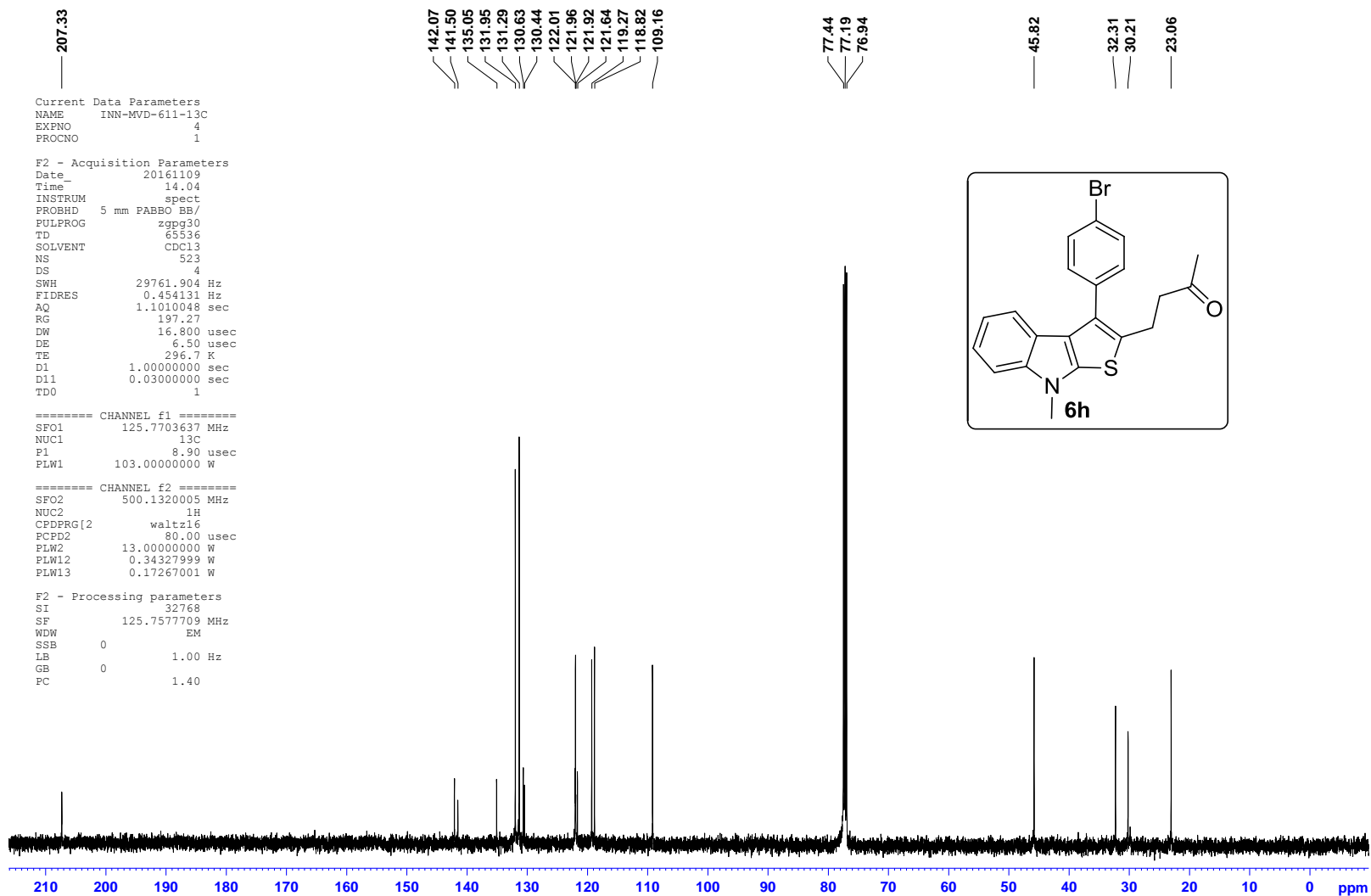


Figure S36. ^{13}C NMR Spectrum of **6h**

Current Data Parameters
NAME INN-MVD-597-1H
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters

Date_ 20150813
Time_ 15.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 30
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 299.2 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 500.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

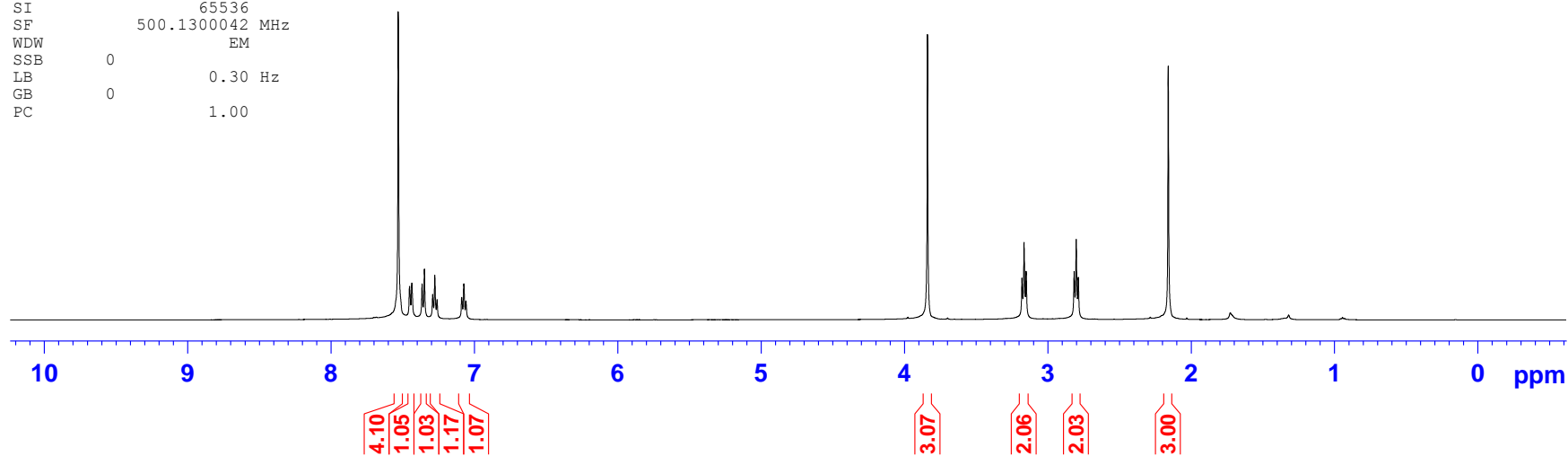
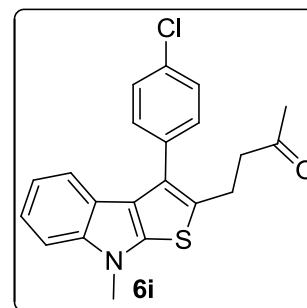


Figure S37. ¹H NMR Spectrum of **6i**

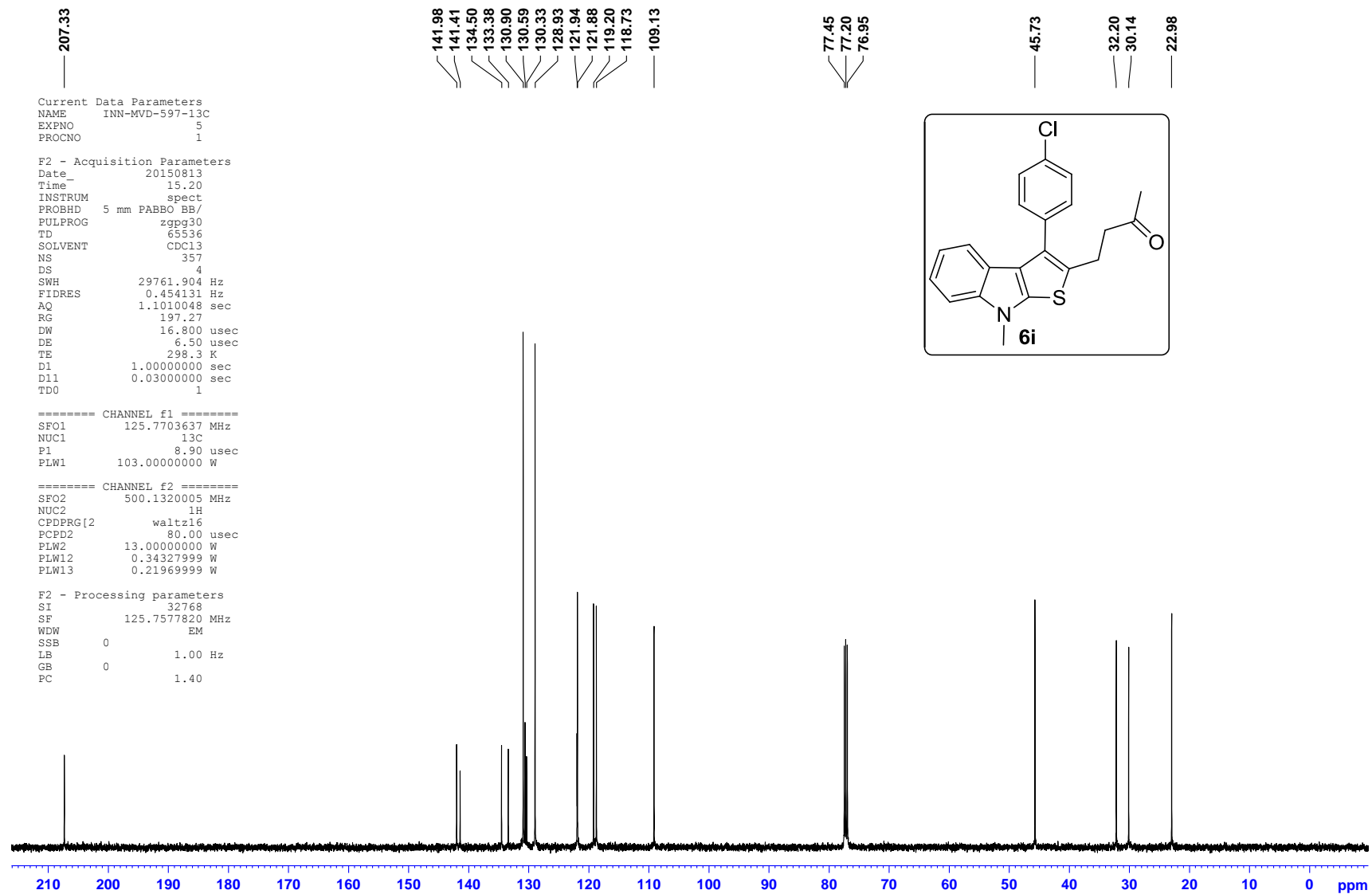


Figure S38. ^{13}C NMR Spectrum of **6i**

Current Data Parameters
NAME INN-MVD-606-1H
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150814
Time 20.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 297.1 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 500.1300016 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

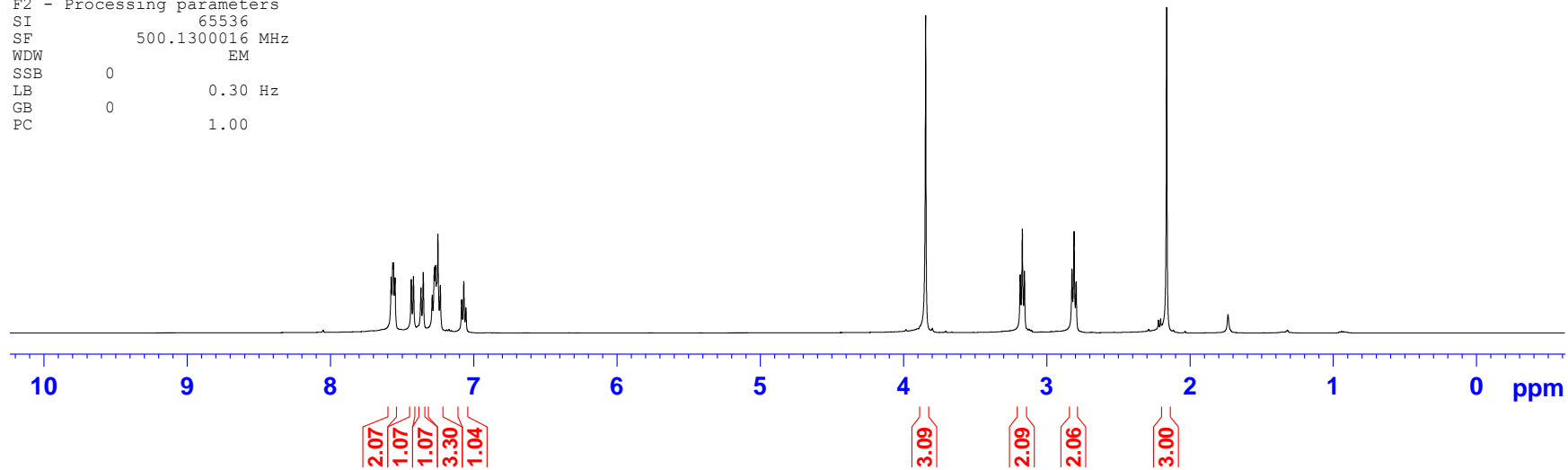
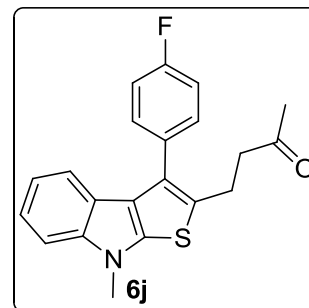


Figure S39. ¹H NMR Spectrum of **6j**

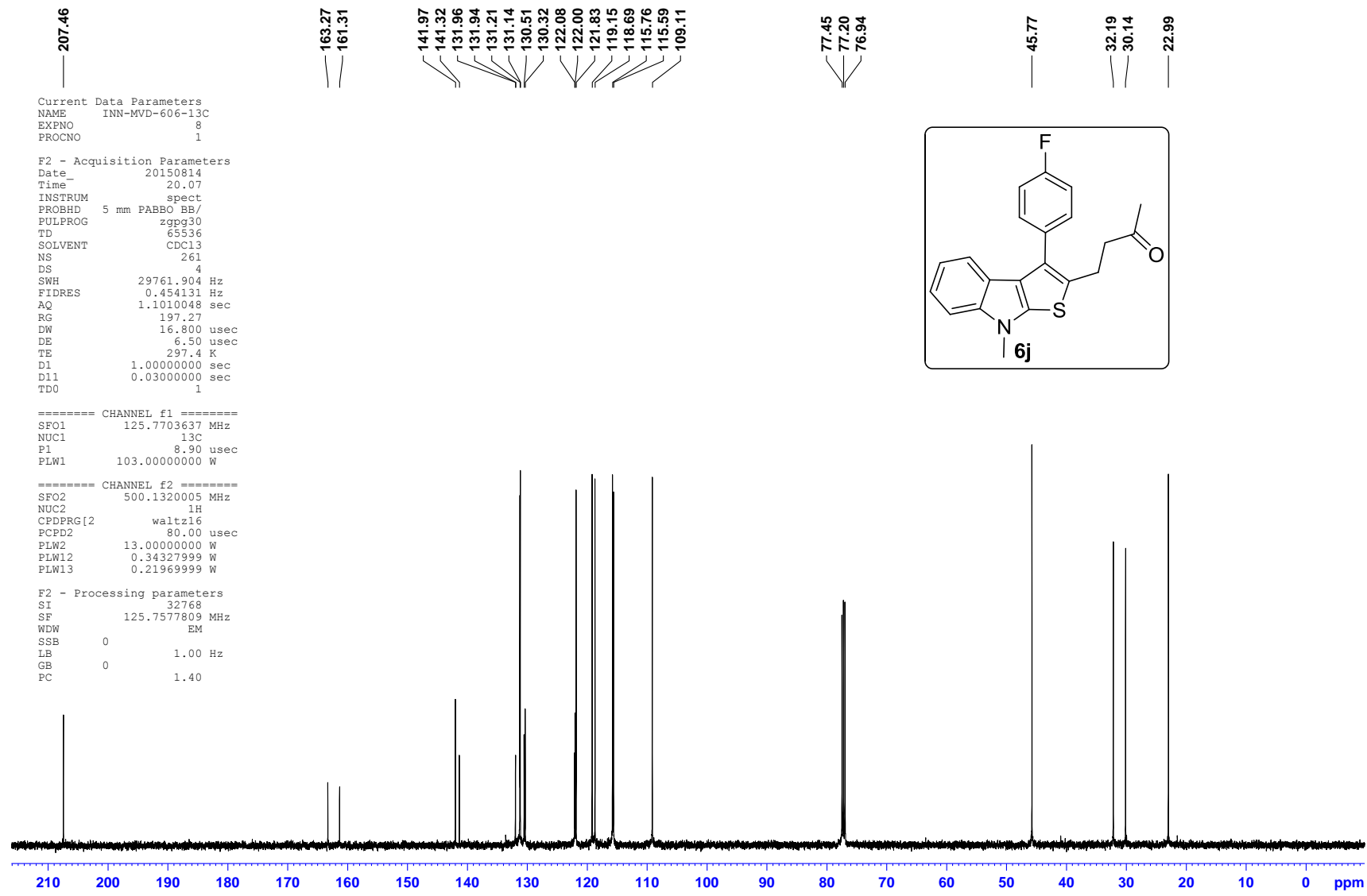


Figure S40. ¹³C NMR Spectrum of **6j**

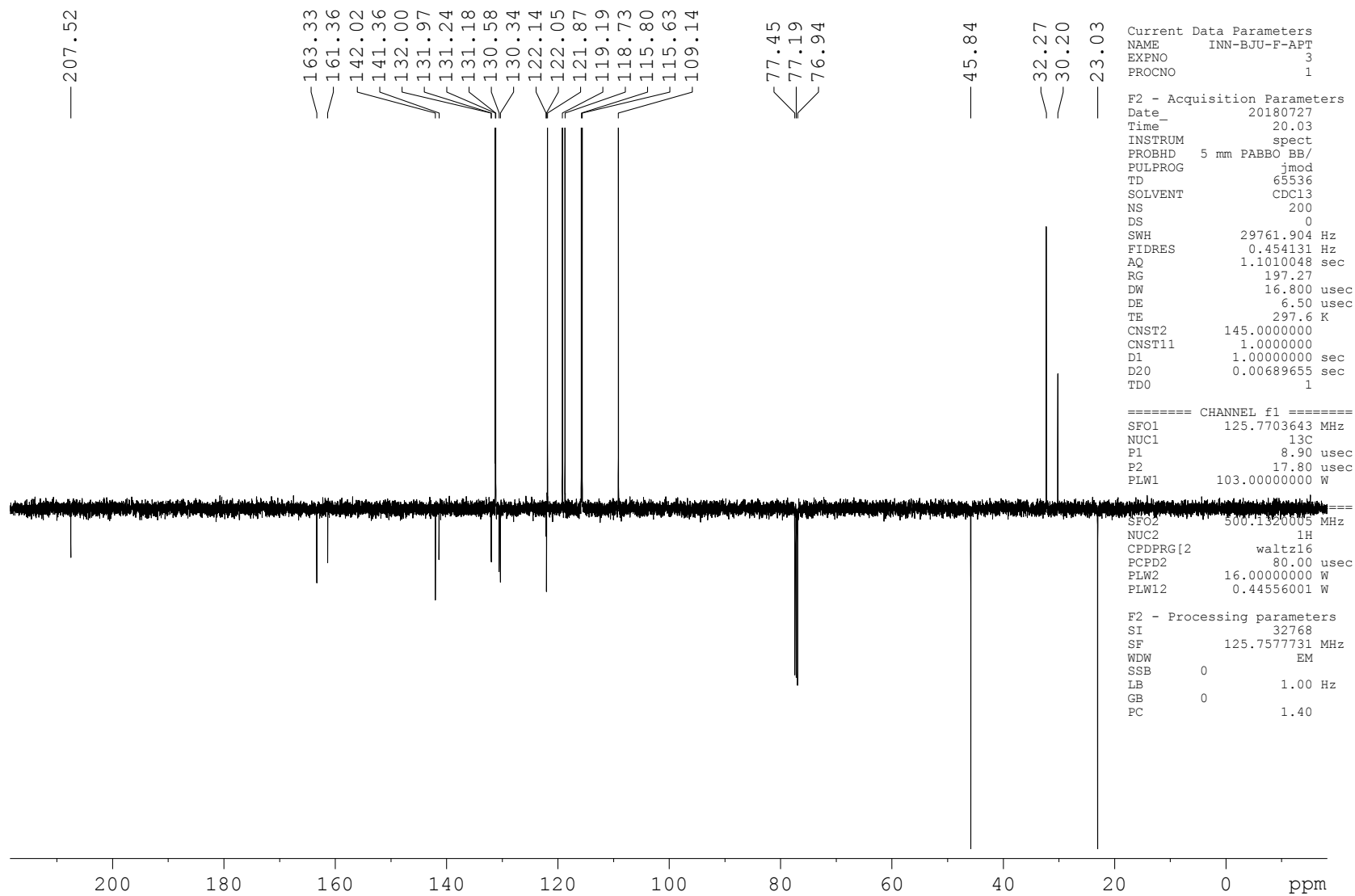


Figure S41. ¹³C-APT Spectrum of 6j

NAME INN-MVD-606-19F
EXPNO 4
PROCNO 1
Date_ 20150822
Time 21.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340532 sec
RG 724
DW 5.600 usec
DE 6.50 usec
TE 296.9 K
D1 1.0000000 sec
TDO 1

----- CHANNEL f1 -----
NUC1 19F
P1 13.00 usec
PL1 -3.00 dB
PL1W 17.04036522 W
SFO1 376.4607164 MHz
SI 65536
SF 376.4983660 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

-114.65

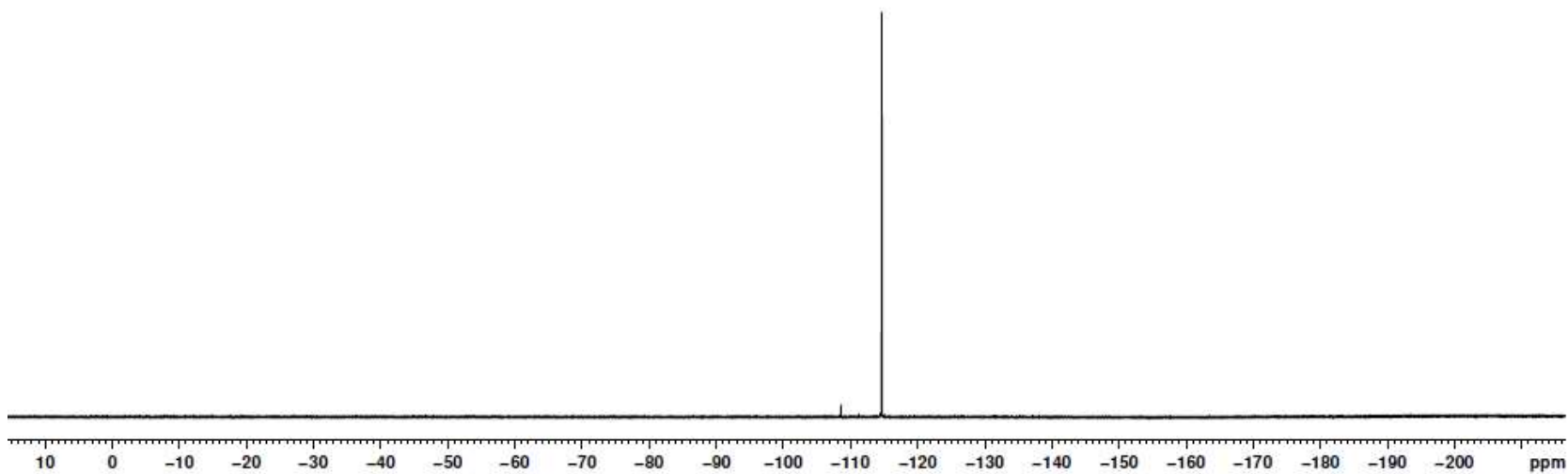
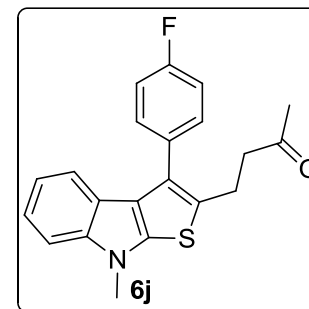


Figure S42. ¹⁹F NMR Spectrum of 6j

Current Data Parameters
NAME INN-MVD-609-1H
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150821
Time_ 18.57
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 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 296.8 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 500.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

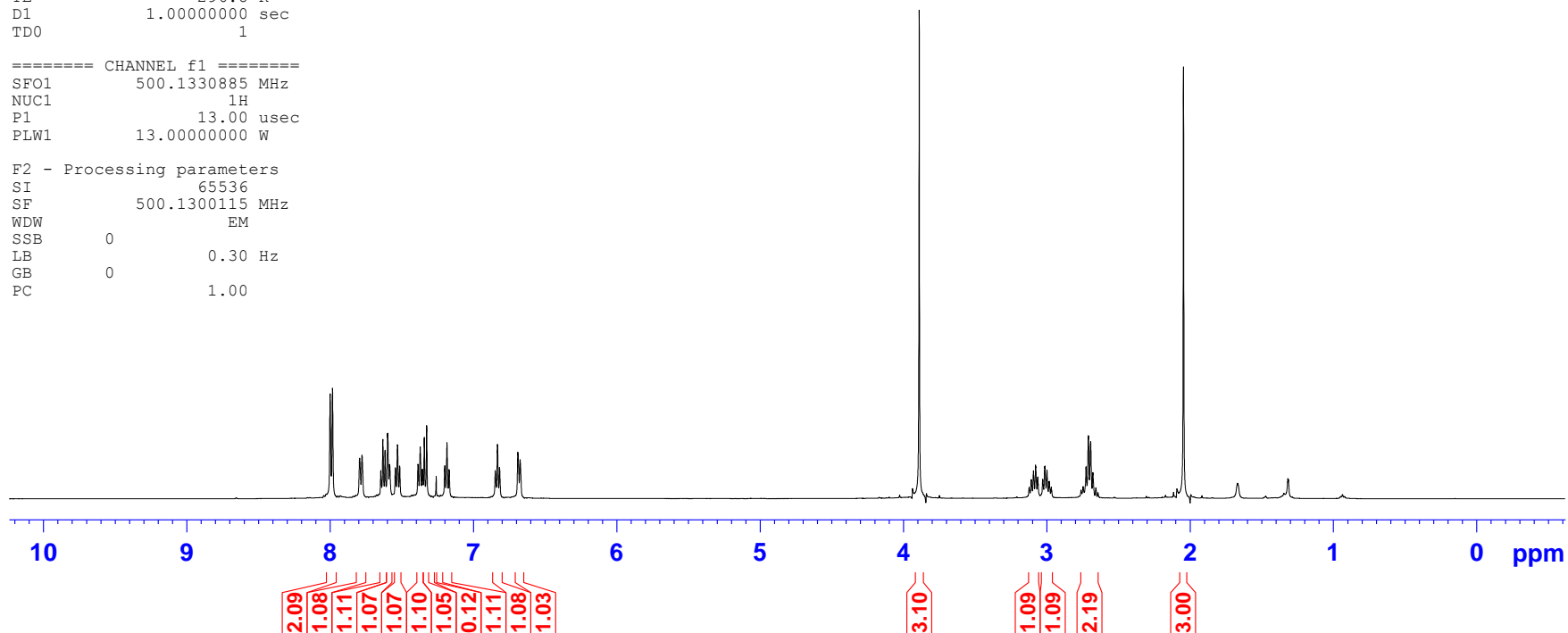
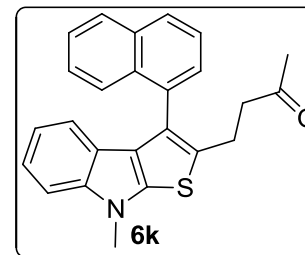


Figure S43. ¹H NMR Spectrum of **6k**

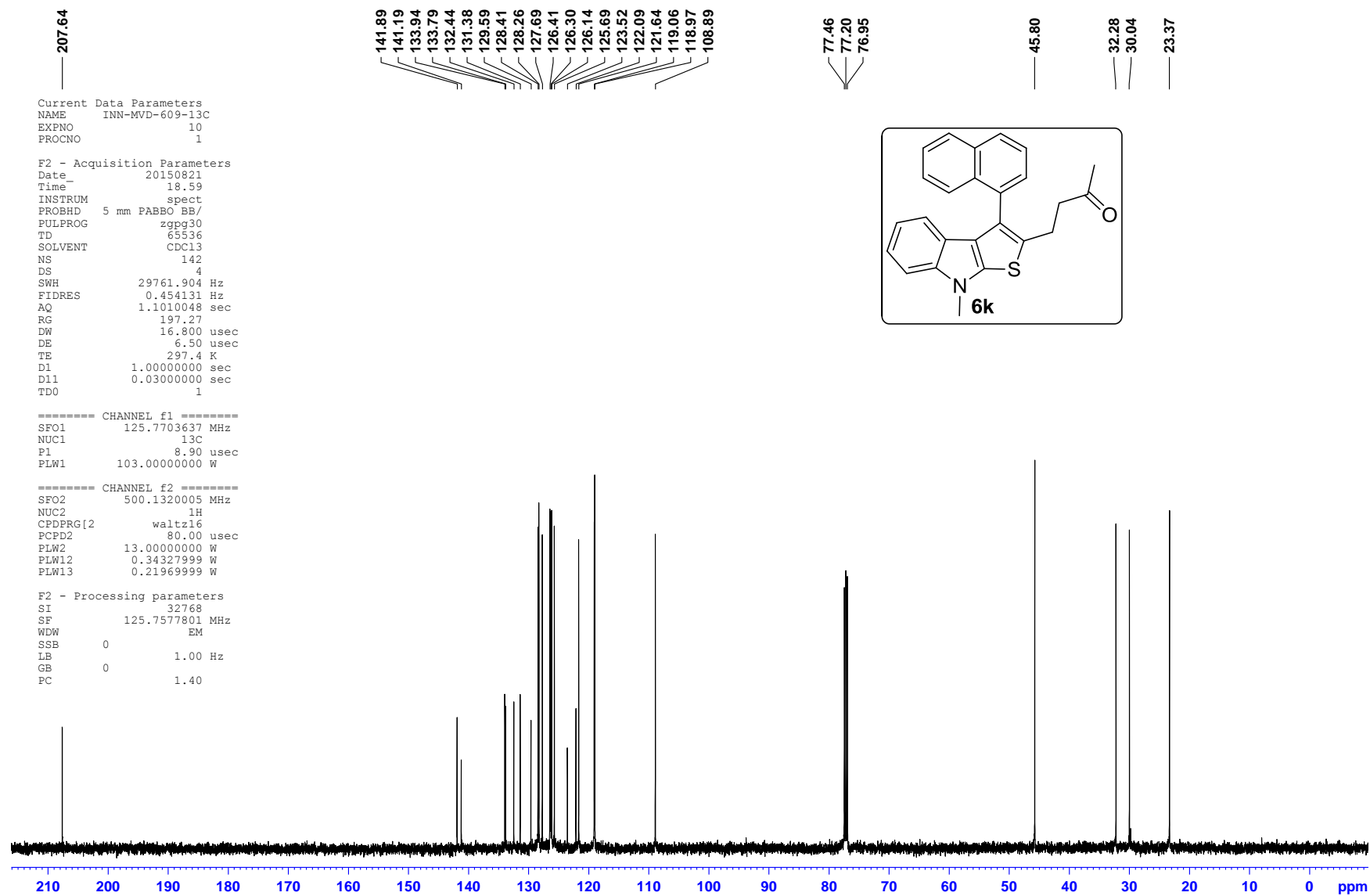


Figure S44. ^{13}C NMR Spectrum of **6k**

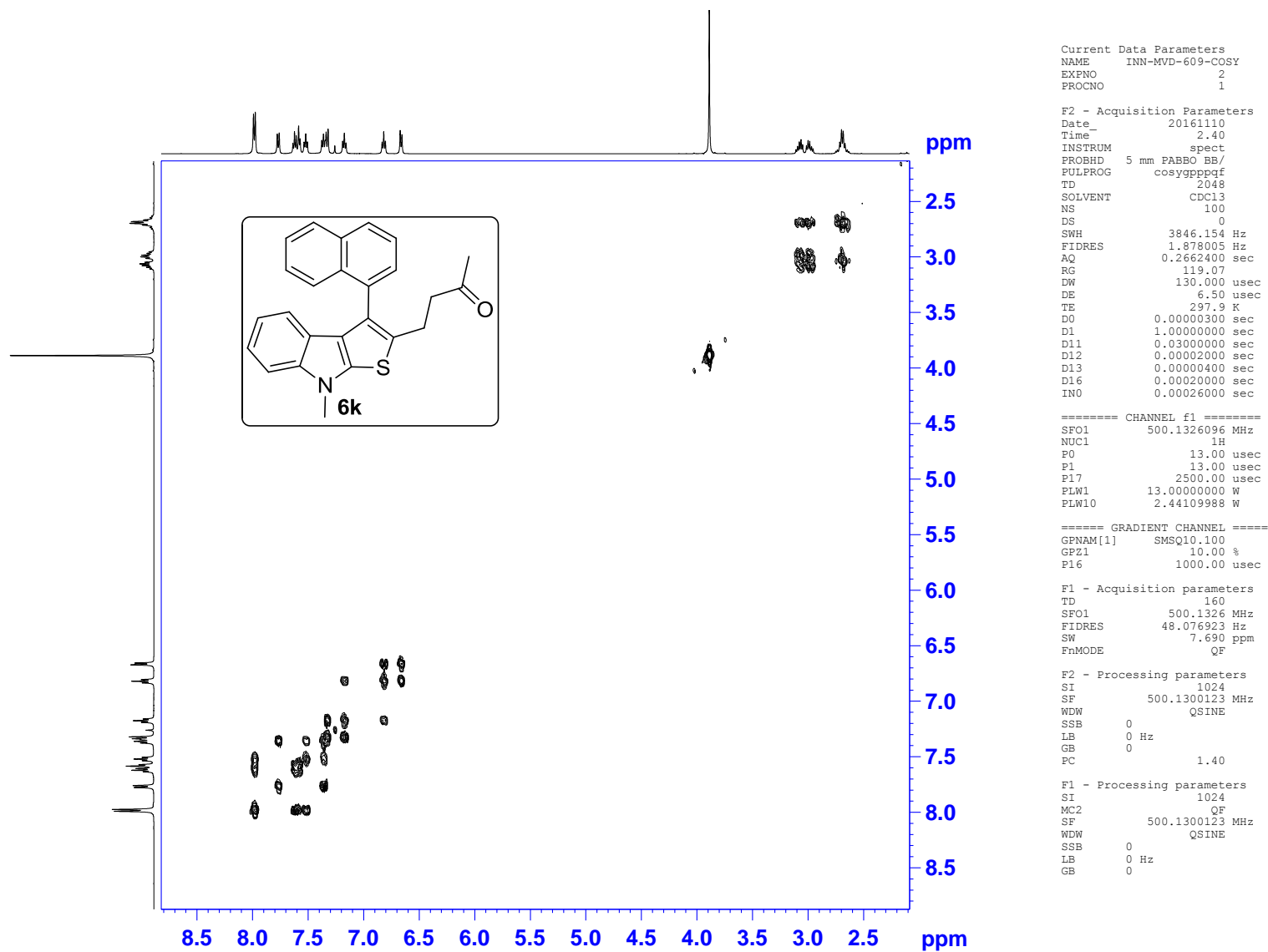


Figure S45. ^1H - ^1H -COSY Spectrum of **6k**

Current Data Parameters
NAME INN-MVD-640-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150917
Time_ 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 53.37
DW 50.000 usec
DE 6.50 usec
TE 293.7 K
D1 1.0000000 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 500.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

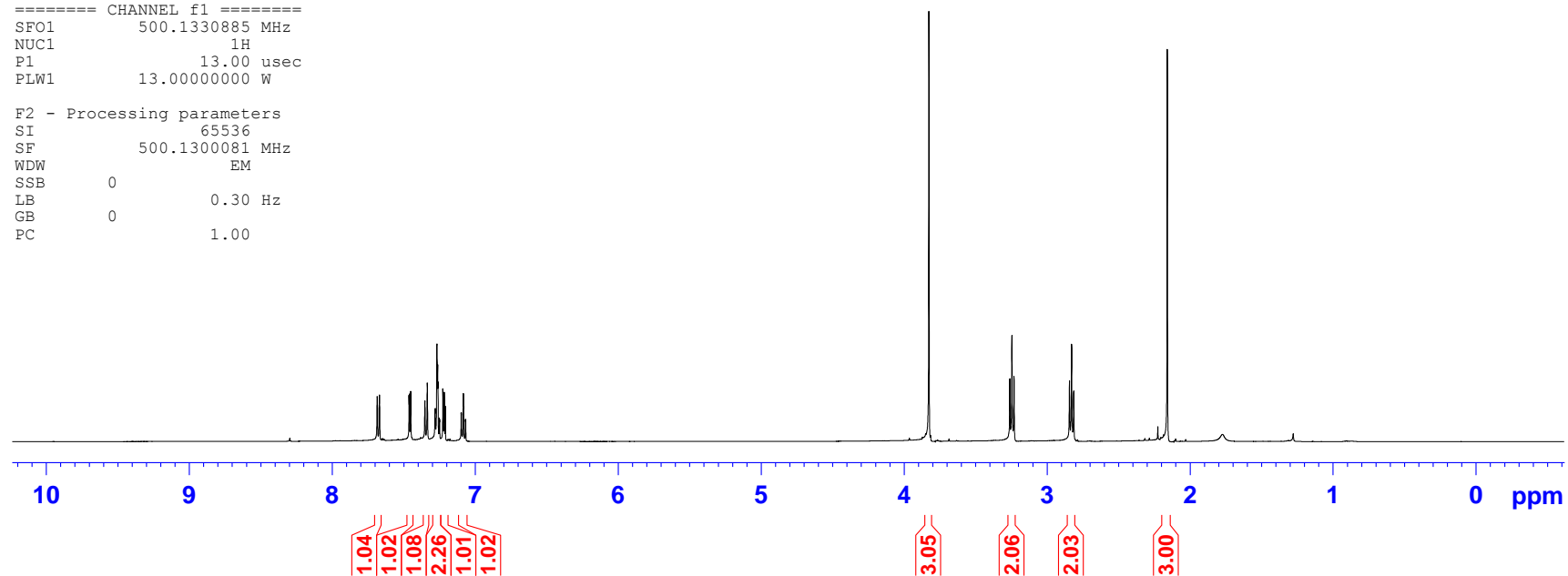
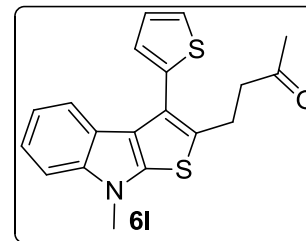


Figure S46. ¹H NMR Spectrum of **61**

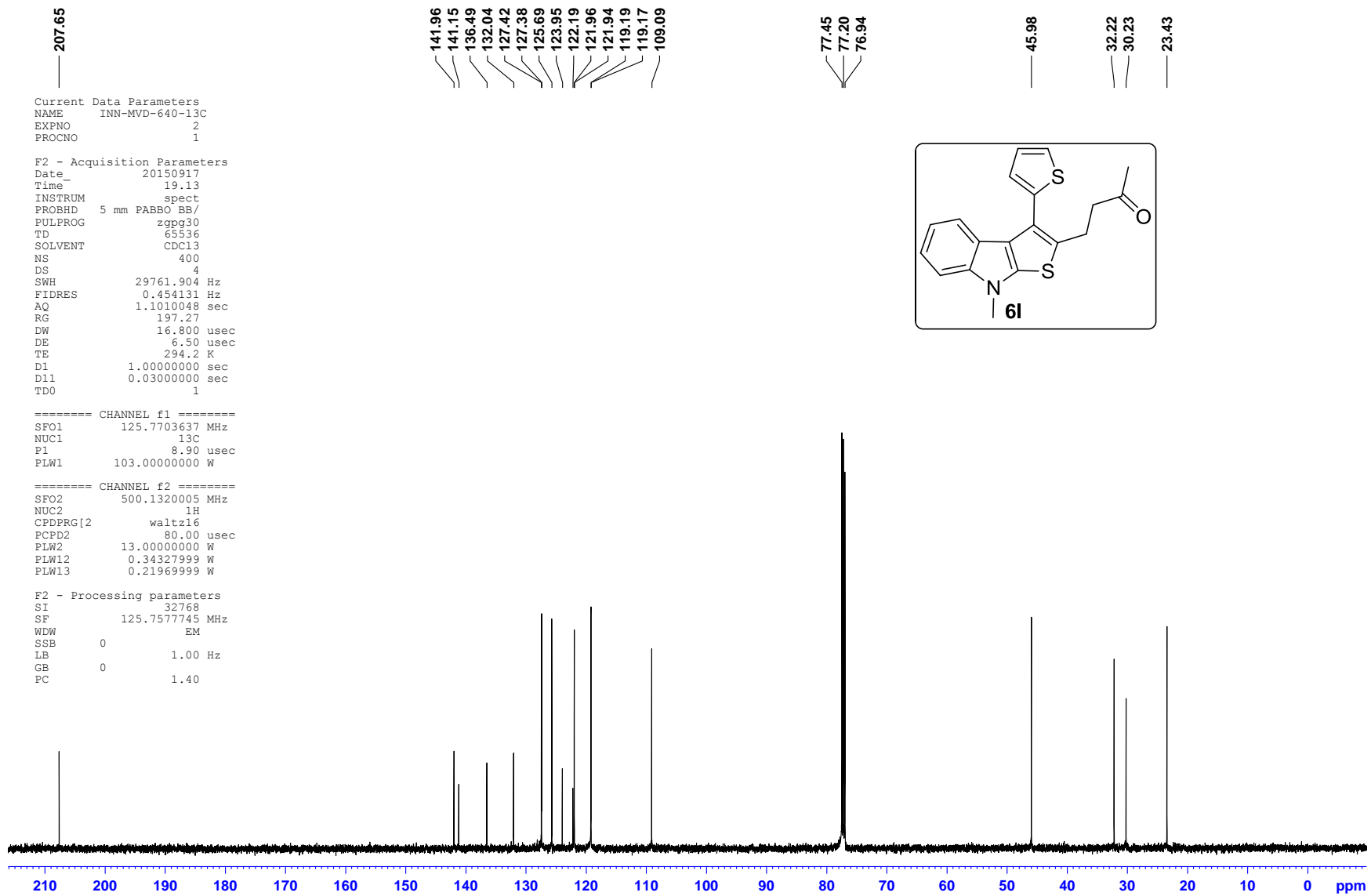


Figure S47. ^{13}C NMR Spectrum of **6l**

Current Data Parameters
NAME INN-MVD-661-1H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151002
Time_ 19.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 24
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 80.35
DW 50.000 usec
DE 6.50 usec
TE 295.6 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 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

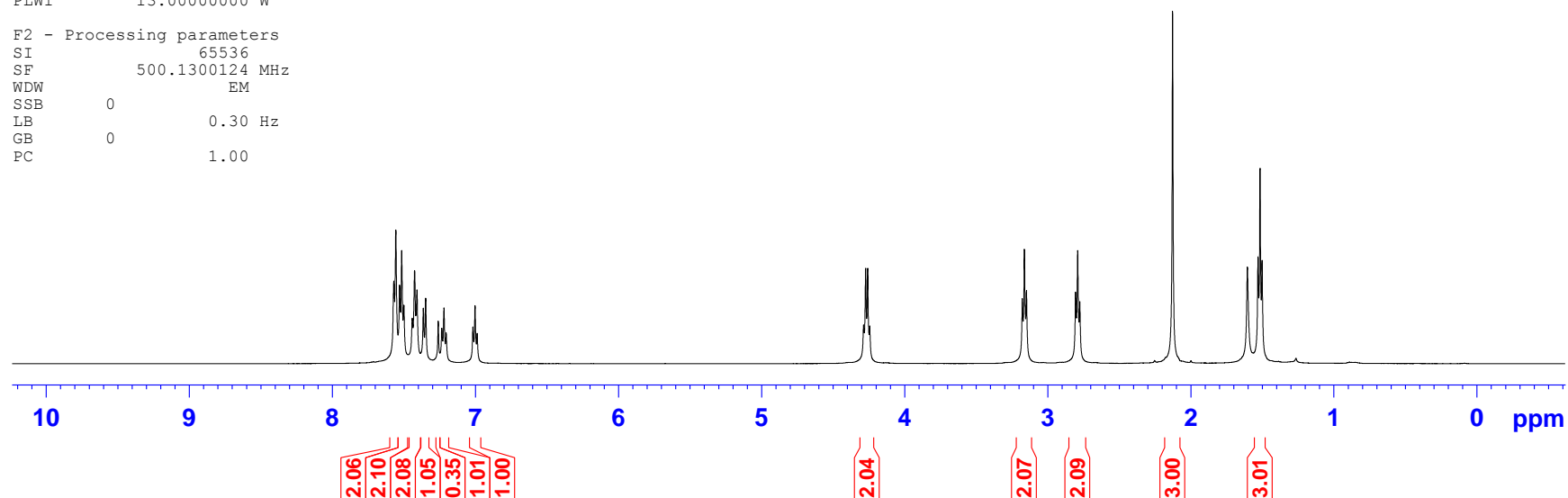
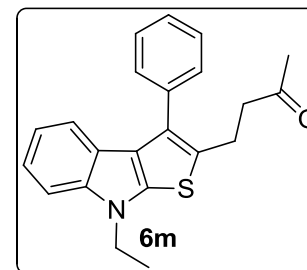


Figure S48. ¹H NMR Spectrum of 6m

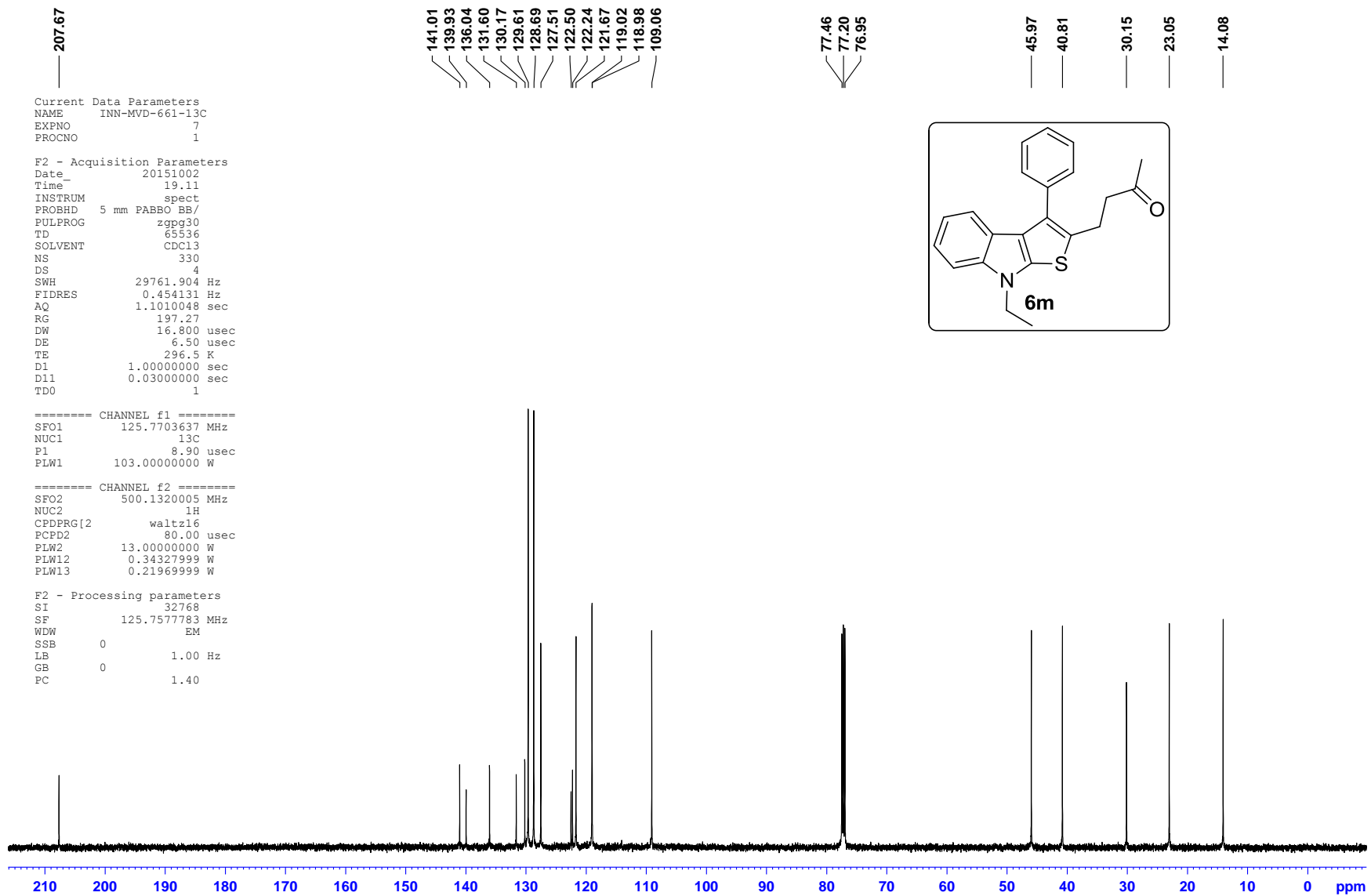


Figure S49. ^{13}C NMR Spectrum of **6m**

Current Data Parameters
NAME INN-MVD-662-1H
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151002
Time_ 18.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 18
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.6 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 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

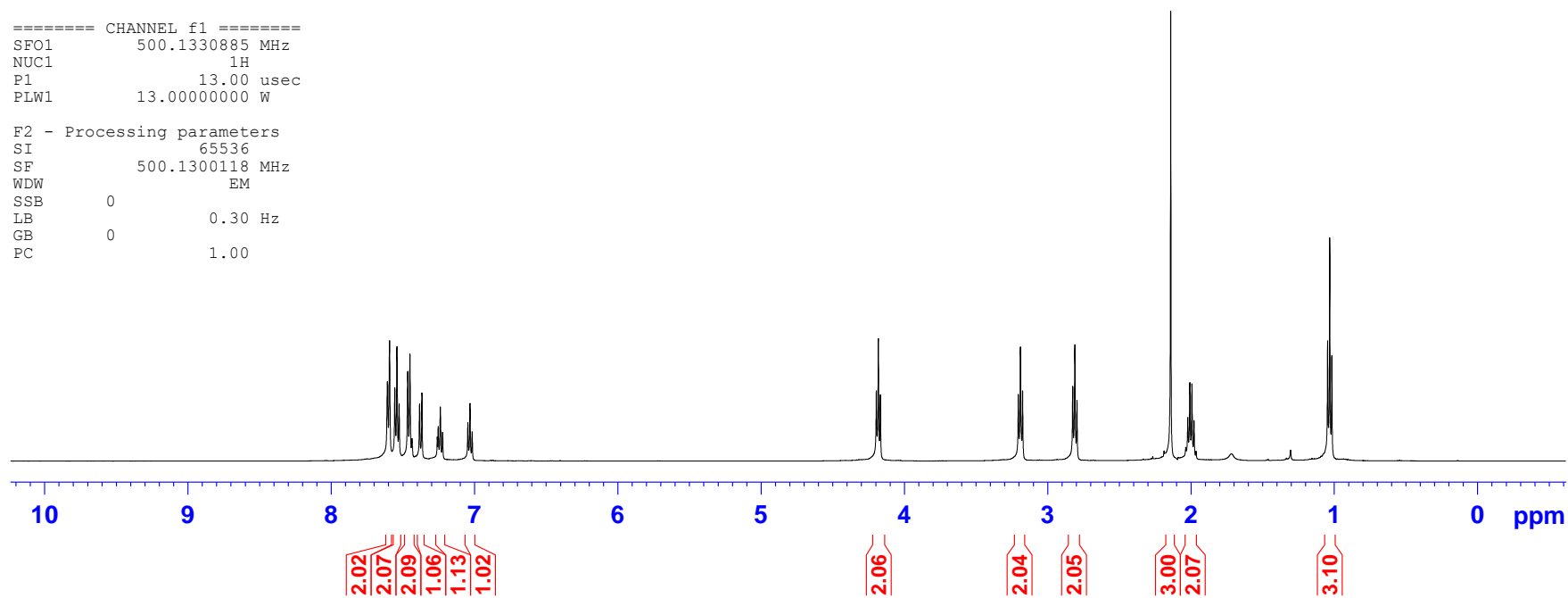
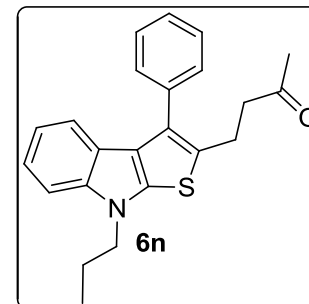


Figure S50. ¹H NMR Spectrum of **6n**

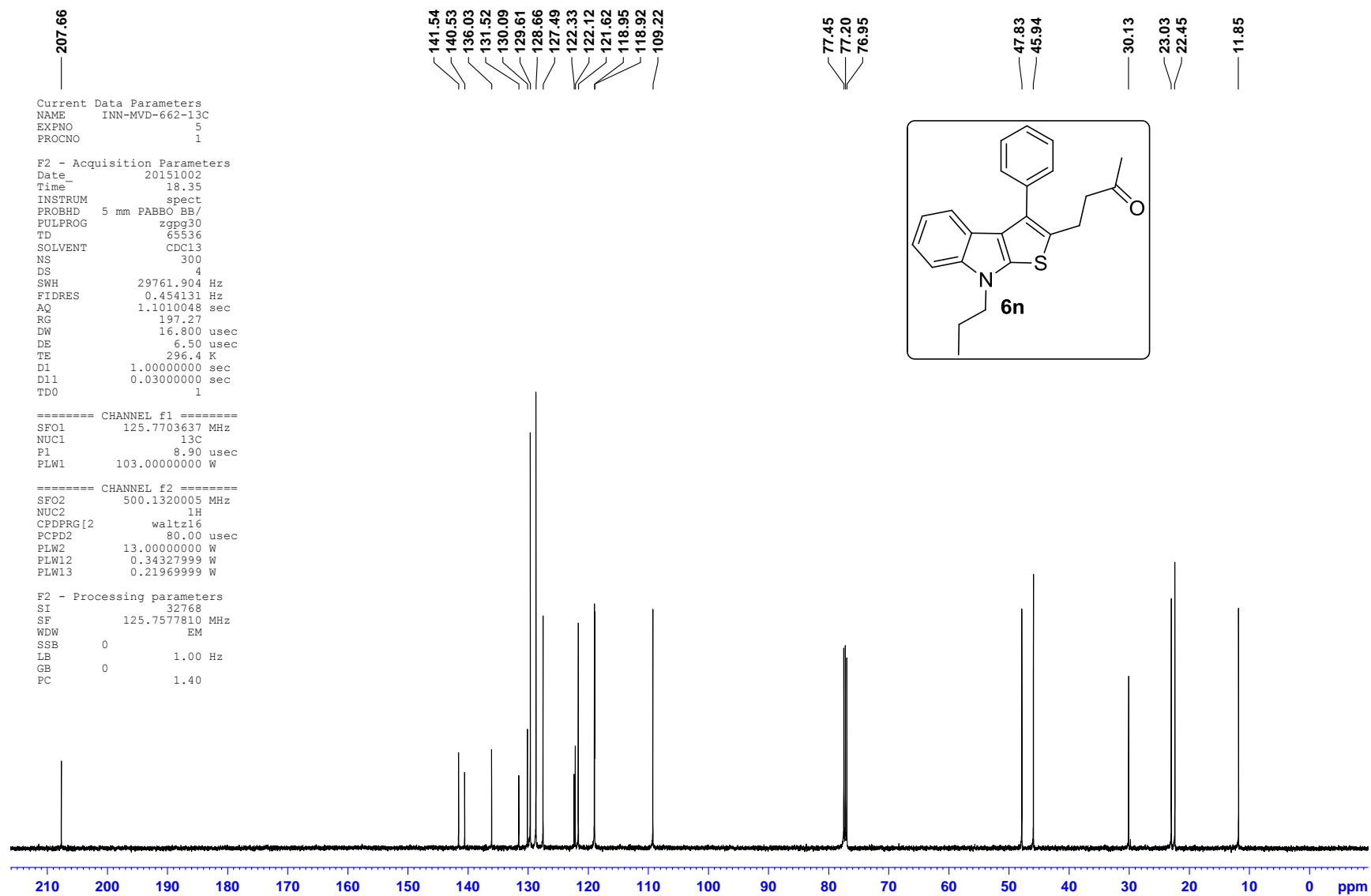


Figure S51. ^{13}C NMR Spectrum of **6n**

Current Data Parameters
NAME INN-MVD-660-1H
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151002
Time_ 18.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 23
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.6 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 500.1300117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

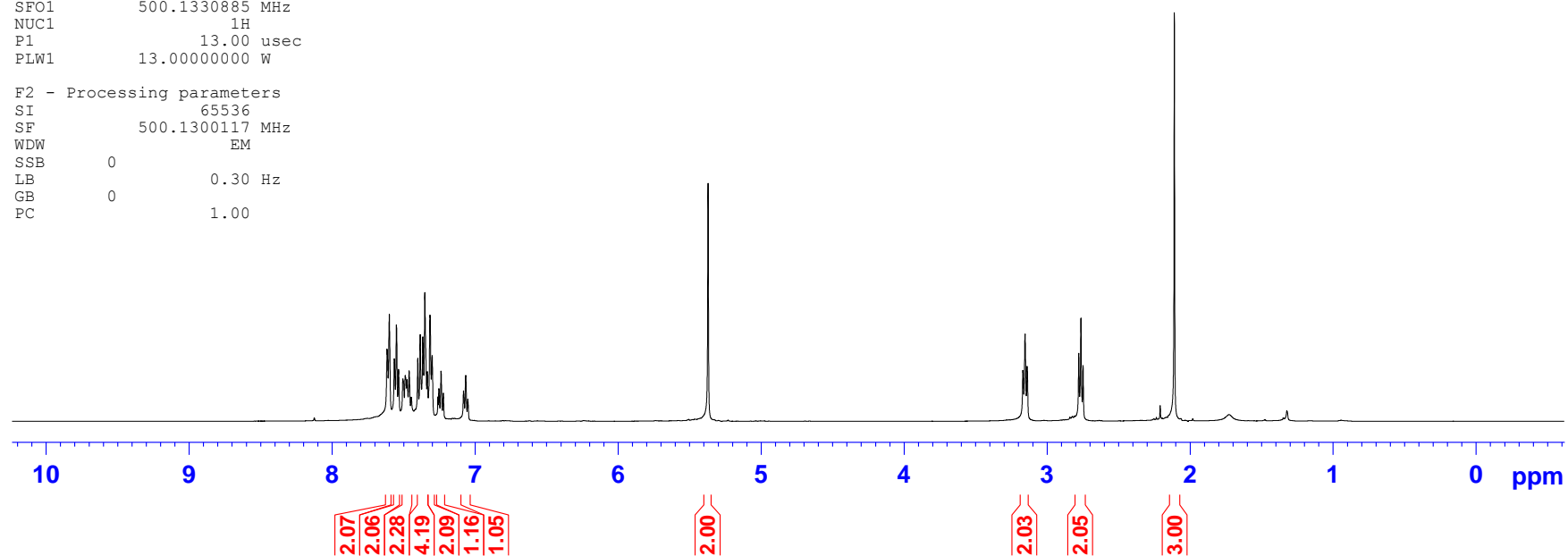
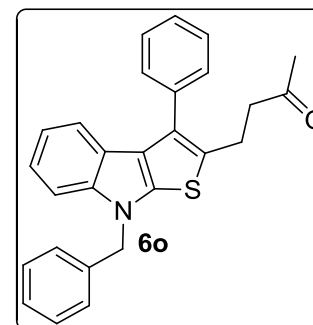


Figure S52. ¹H NMR Spectrum of **60**

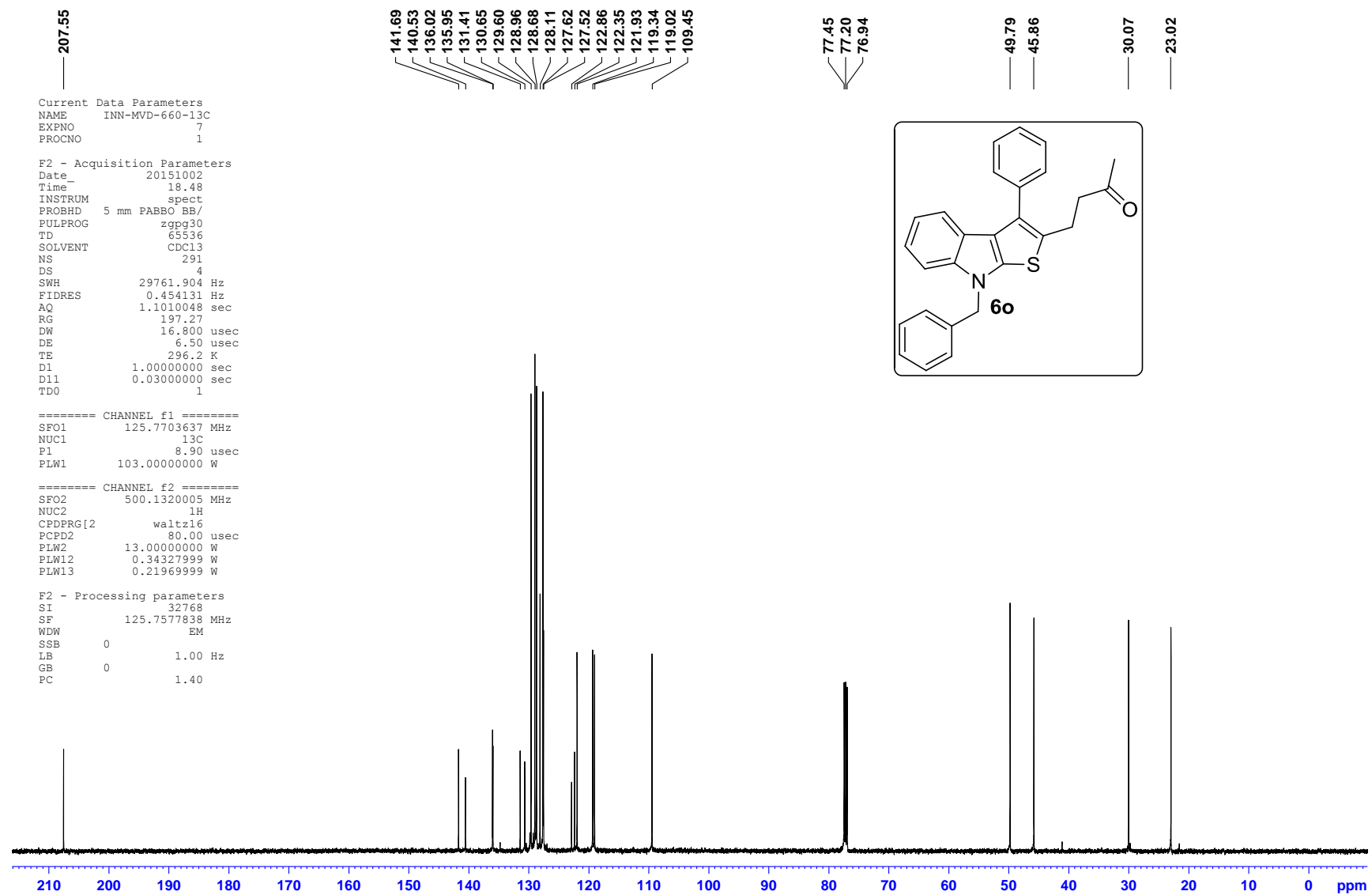


Figure S53. ^{13}C NMR Spectrum of **6o**

Current Data Parameters
NAME INN-MVD-675-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151106
Time_ 13.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 48.36
DW 50.000 usec
DE 6.50 usec
TE 295.6 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 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

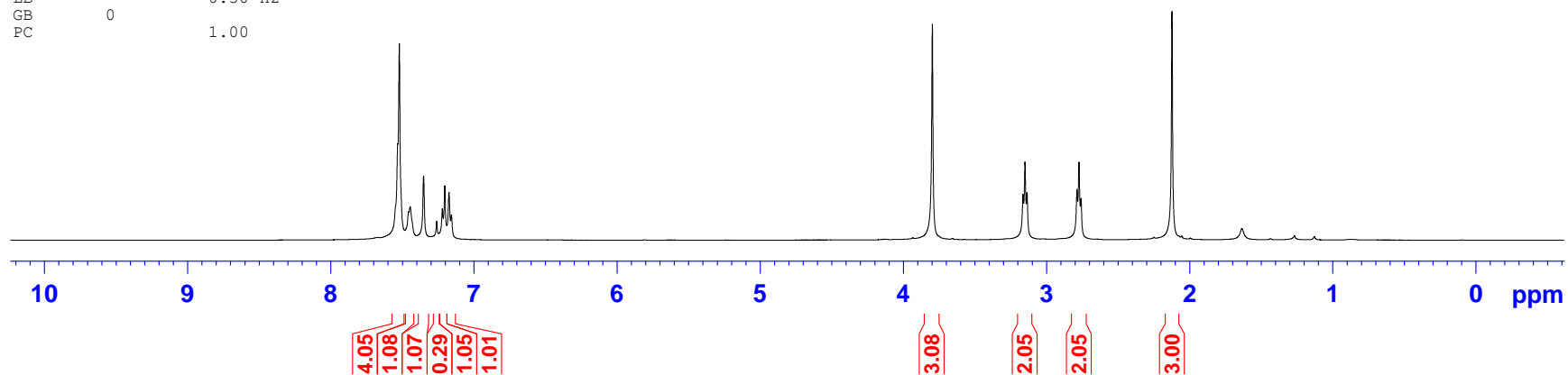
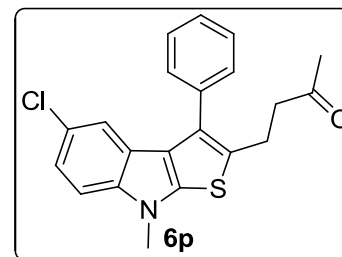


Figure S54. ¹H NMR Spectrum of 6p

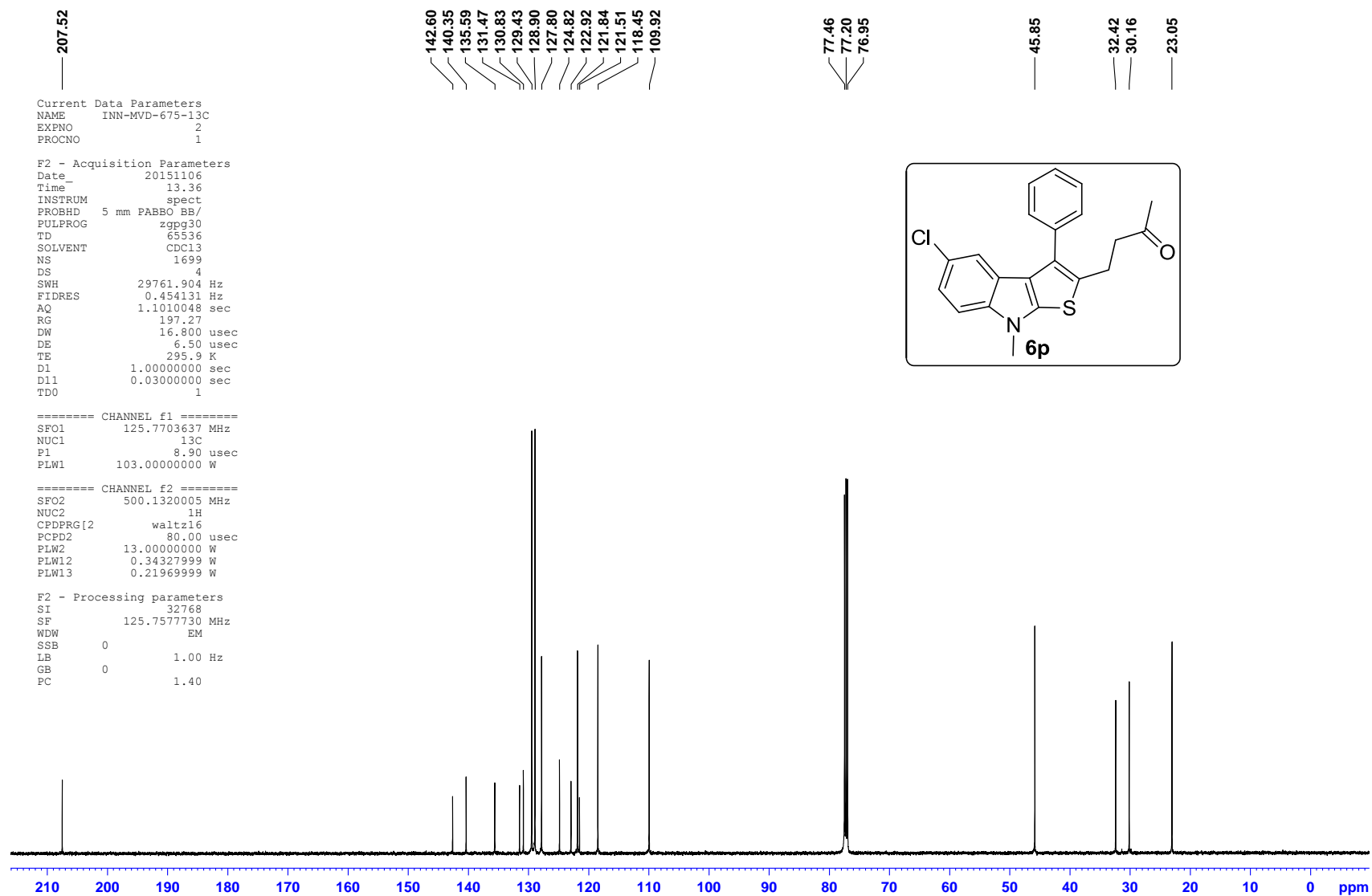


Figure S55. ^{13}C NMR Spectrum of **6p**

Current Data Parameters
NAME INN-MVD-903-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161015
Time 3.40
INSTRUM spect
PROBHD 5 mm SEI 1H/D-
PULPROG zg30
TD 54274
SOLVENT CDCl3
NS 13
DS 0
SWH 8223.685 Hz
FIDRES 0.151522 Hz
AQ 3.2998593 sec
RG 22.6
DW 60.800 usec
DE 6.50 usec
TE 295.3 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 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

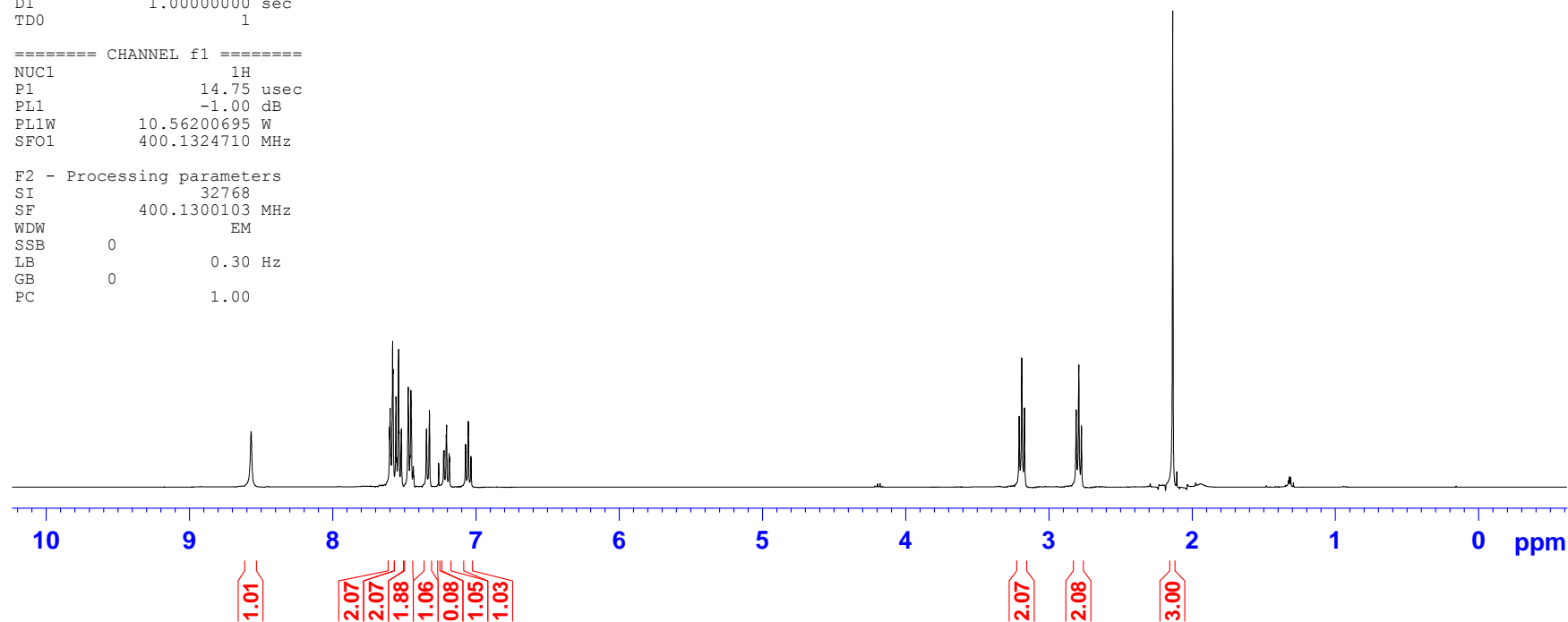
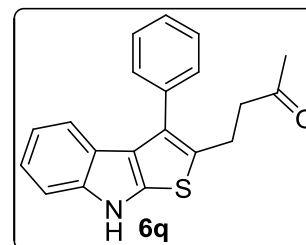


Figure S56. ¹H NMR Spectrum of **6q**

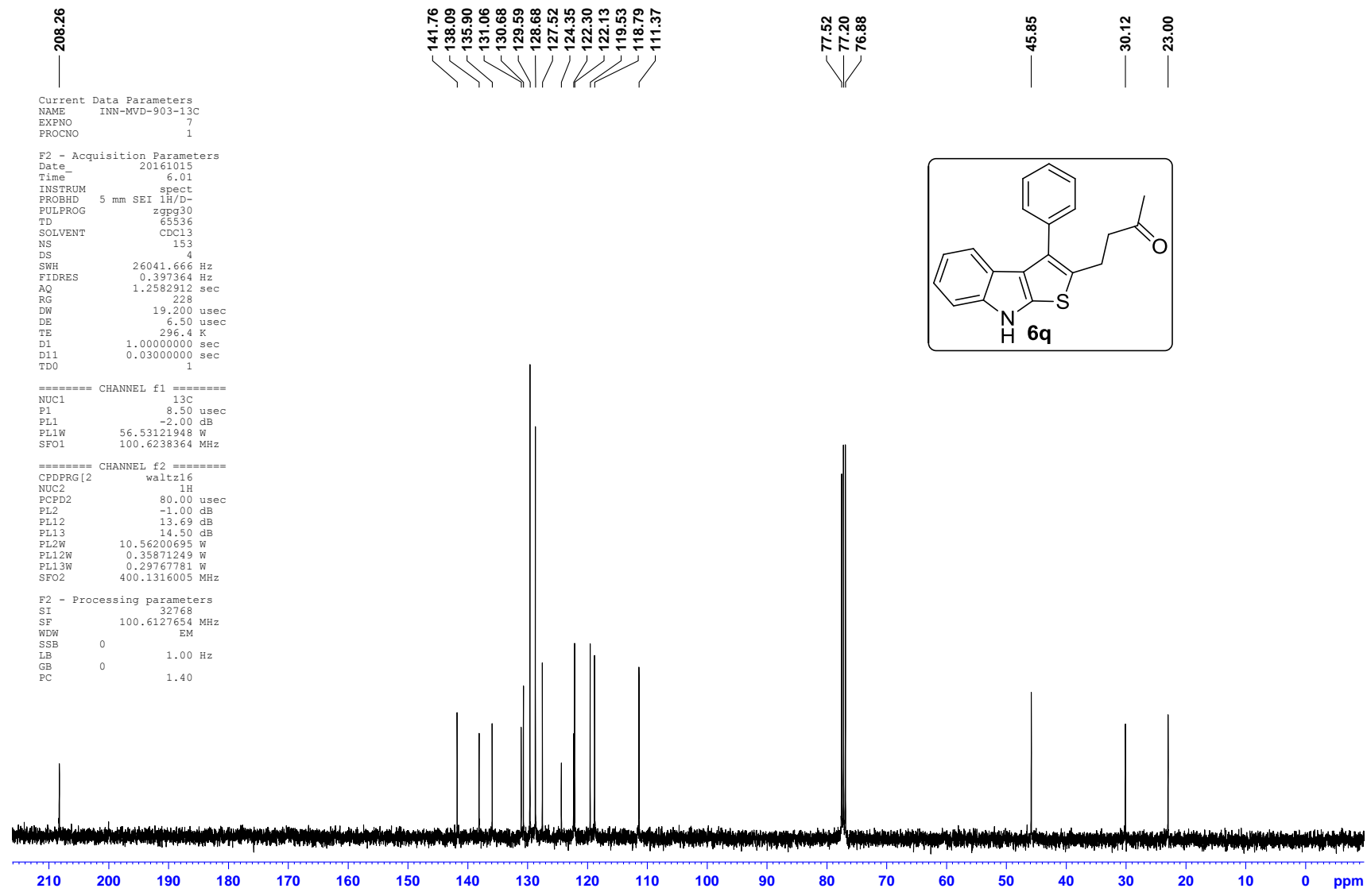


Figure S57. ¹³C NMR Spectrum of **6q**

Current Data Parameters
NAME INN-MVD-889-1H
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161003
Time 16.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 11
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 106.54
DW 50.000 usec
DE 6.50 usec
TE 297.5 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 500.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

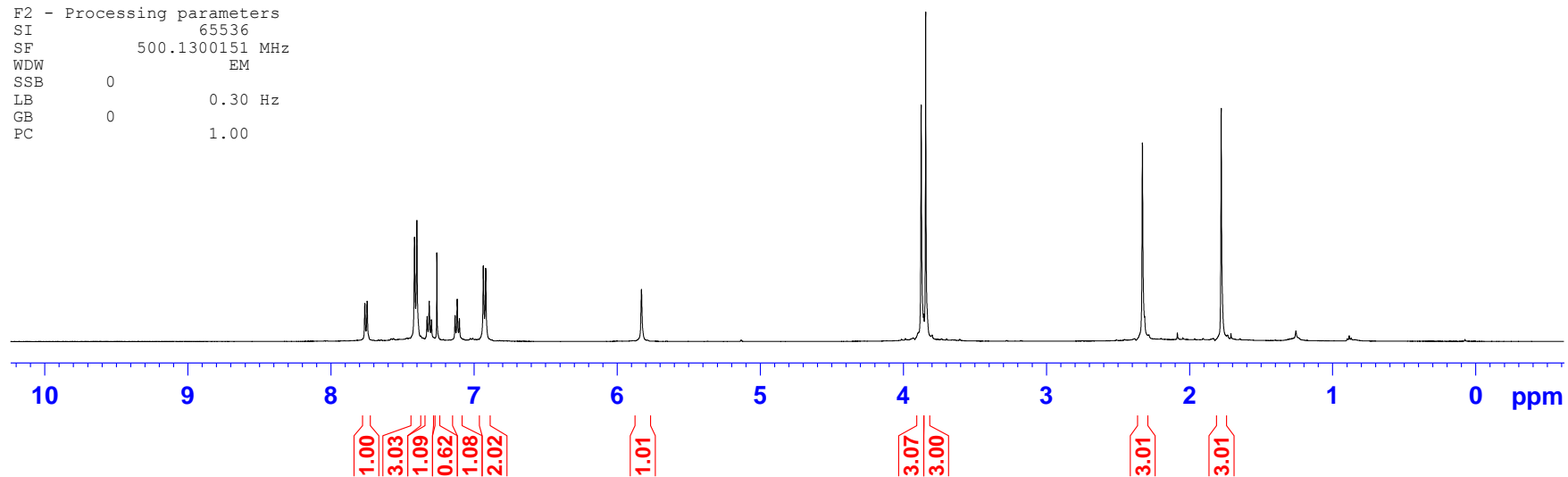
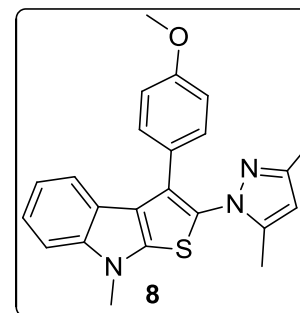


Figure S58. ¹H NMR Spectrum of 8

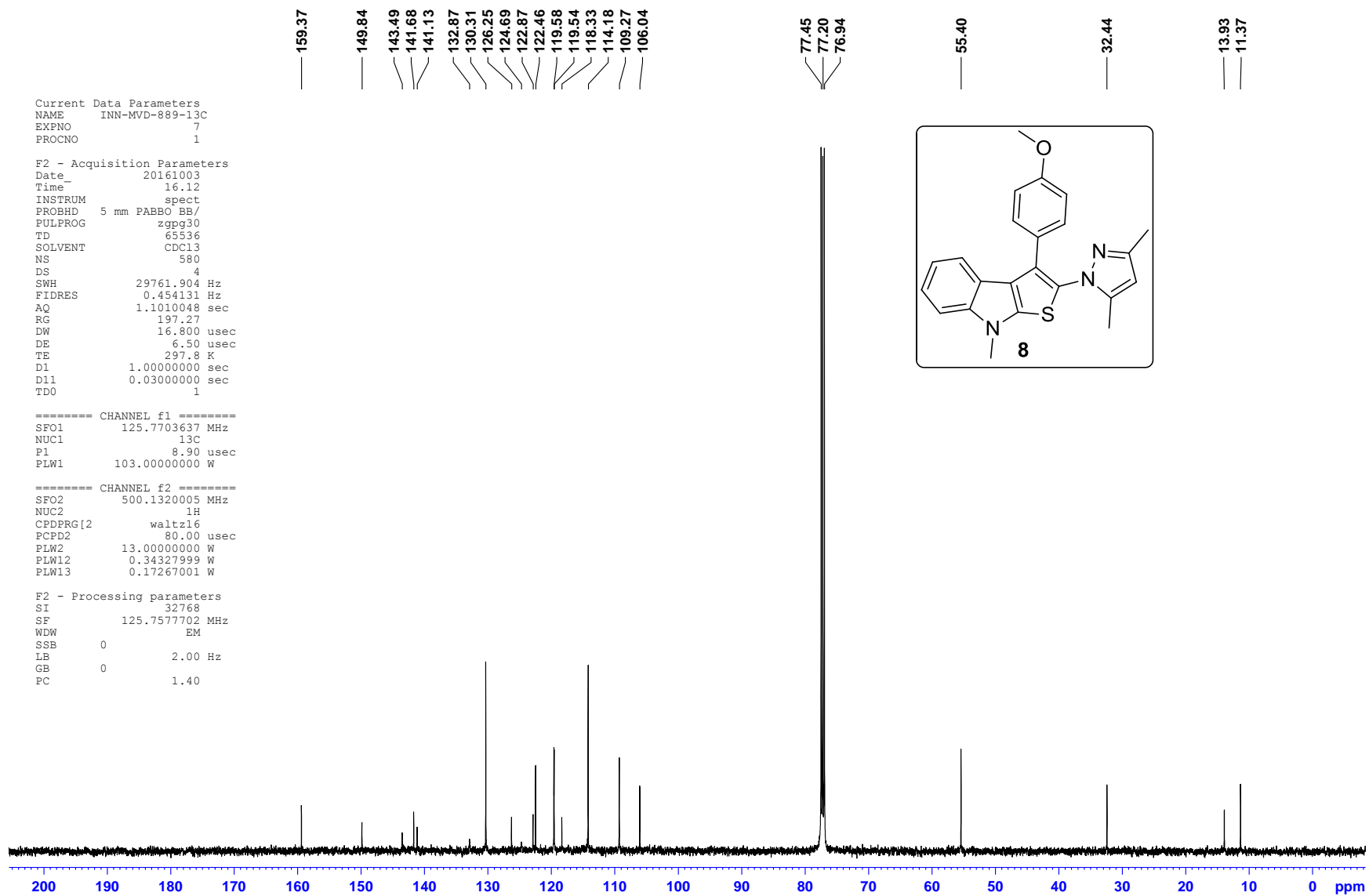


Figure S59. ¹³C NMR Spectrum of **8**