Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

NMR Spectra

for

Synthesis of Hydrazinoheterocycles from Morita-Baylis-Hillman Adducts of Nitroalkenes with Azodicarboxylates

Vaijinath Mane, Jyoti Pandey, Narasimham Ayyagari, Chandan Dey, Raju Kale and Irishi N. N. Namboothiri*

Department of Chemistry, Indian Institute of Technology Bombay, Mumbai 400 076, India

Table of Contents

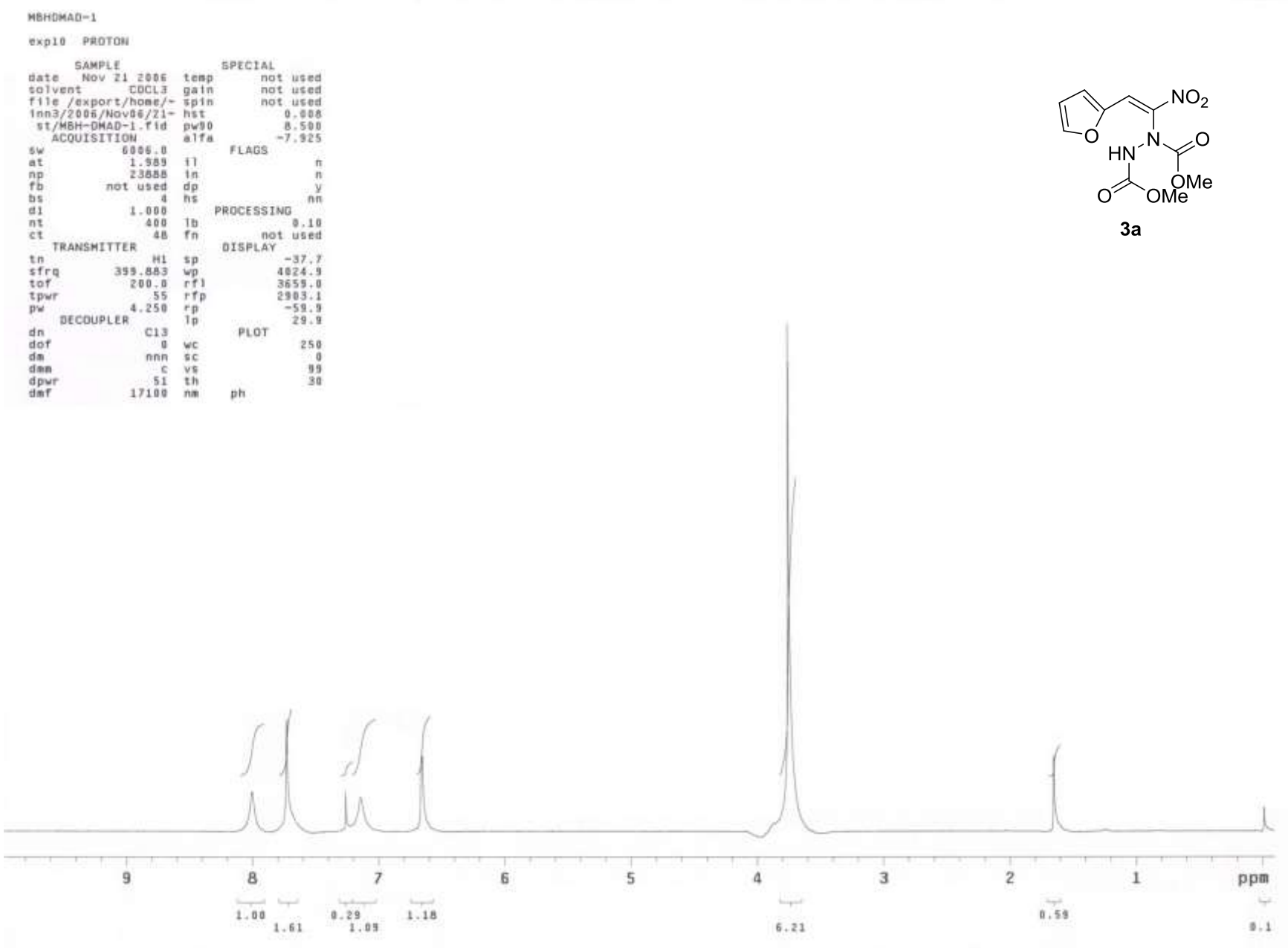
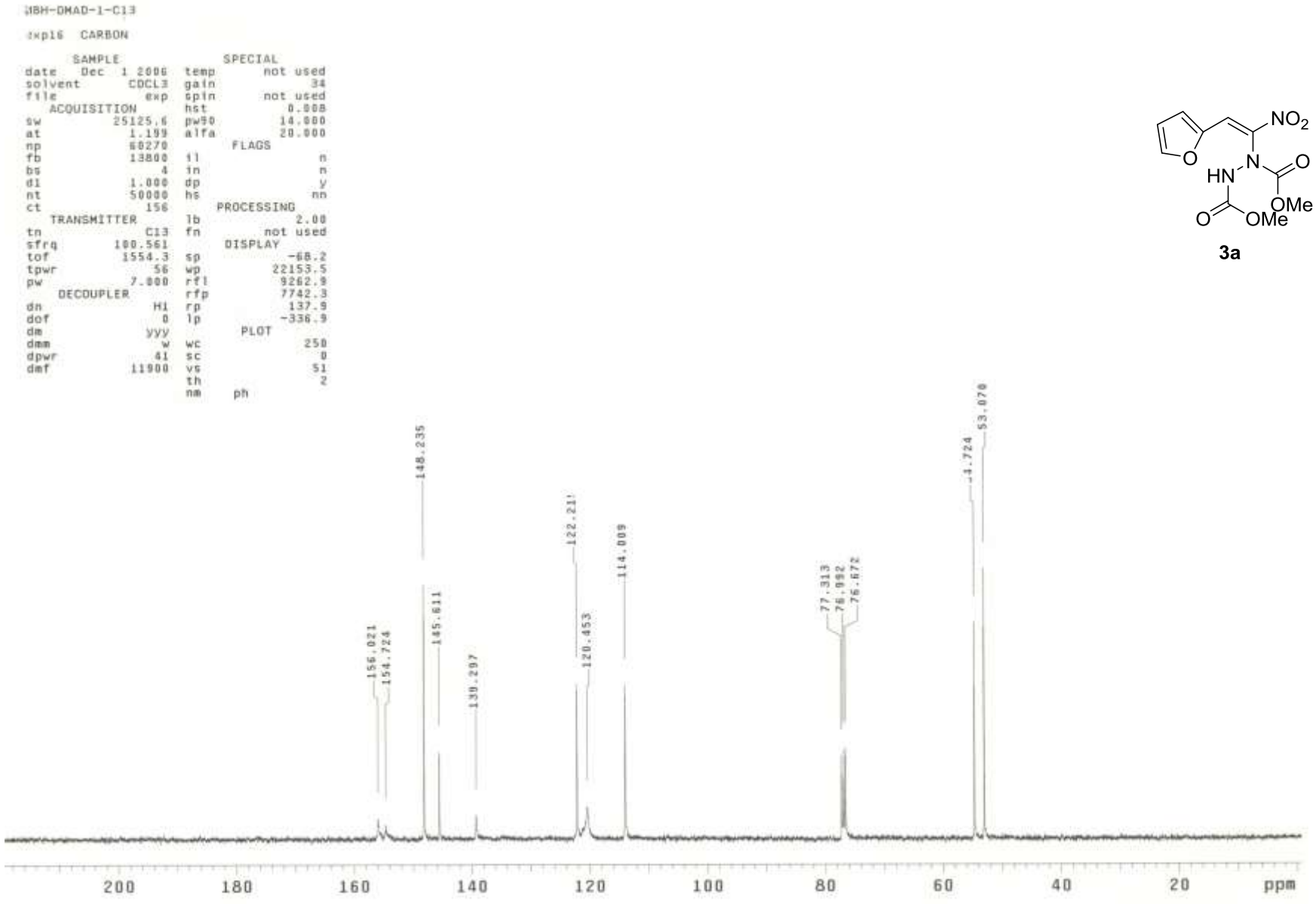


Figure S1. ^{1}H NMR Spectrum of **3a**



3a

Figure S2. ^{13}C NMR Spectrum of **3a**

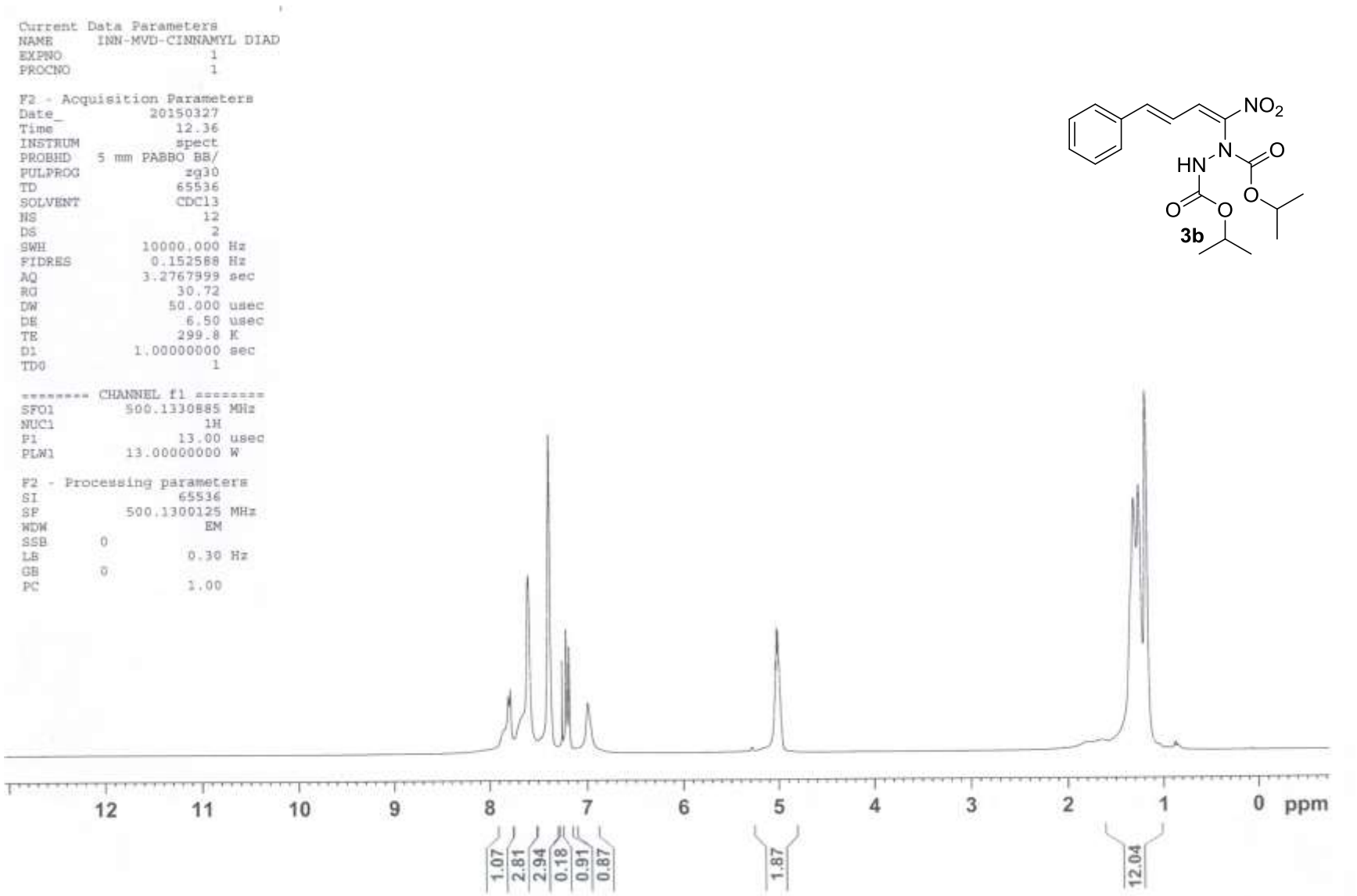


Figure S3. ^{1}H NMR Spectrum of **3b**

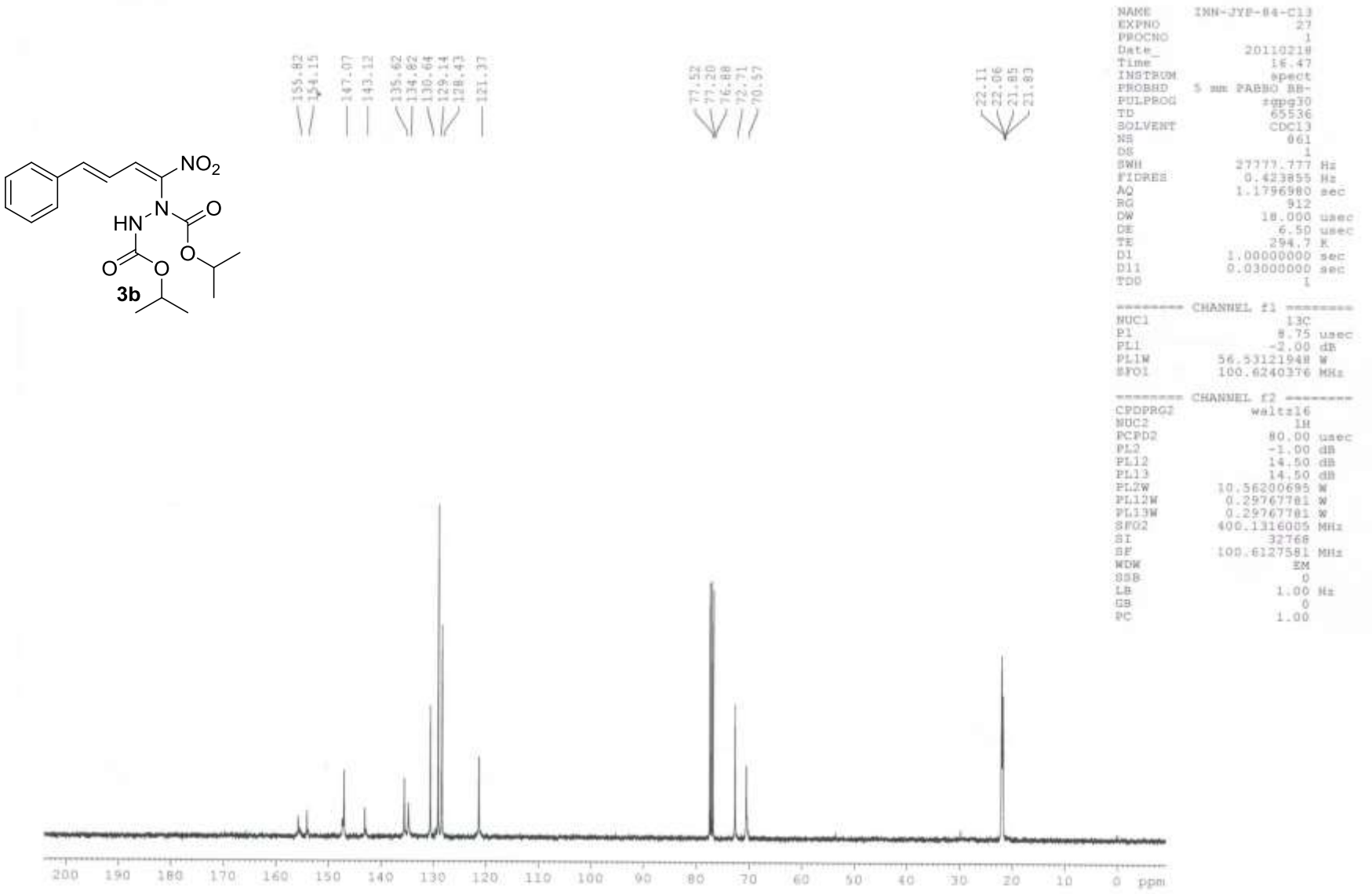


Figure S4. ^{13}C NMR Spectrum of **3b**

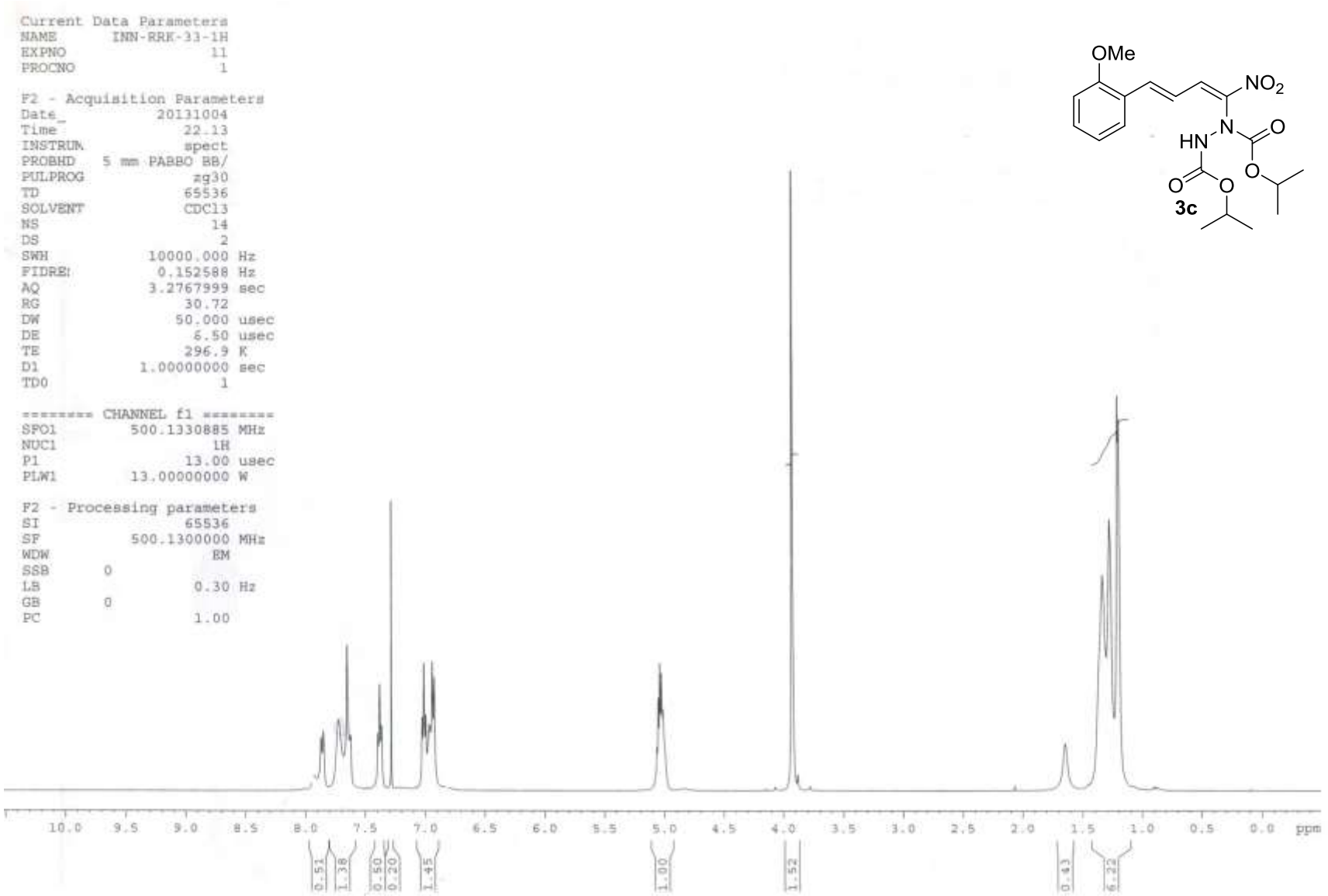


Figure S5. ^{1}H NMR Spectrum of **3c**

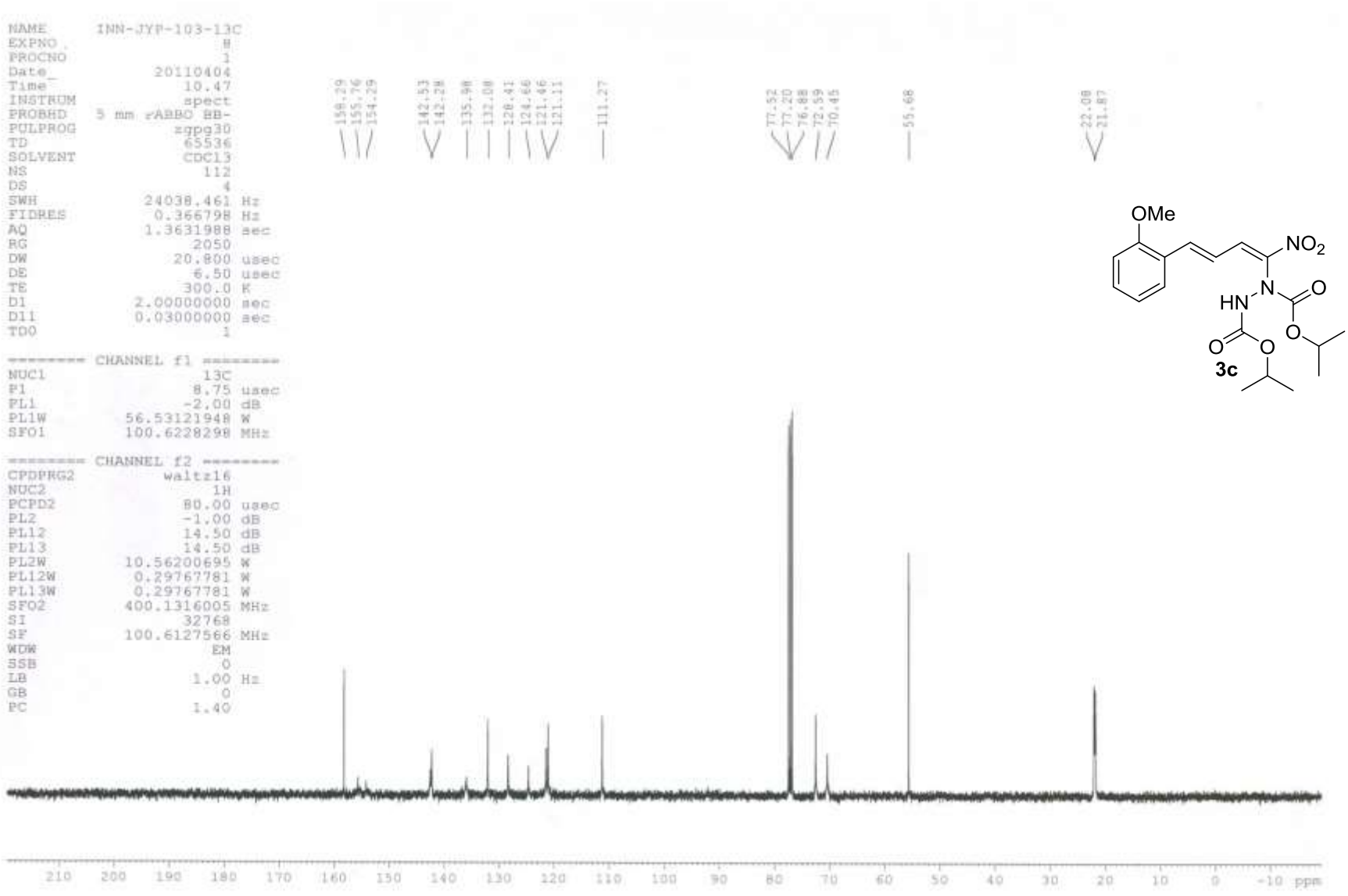


Figure S6. ^{13}C NMR Spectrum of **3c**

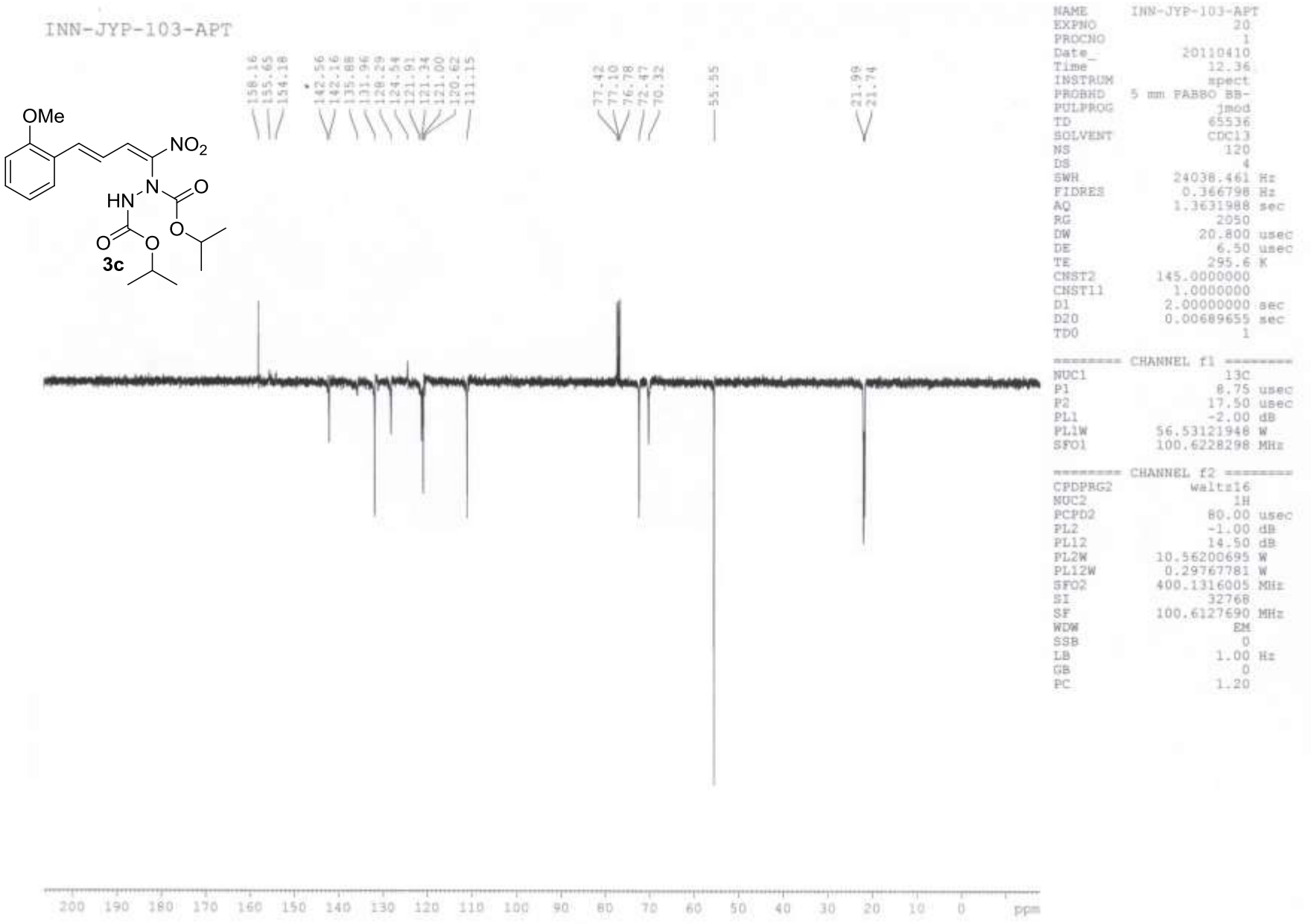


Figure S7. ^{13}C-APT Spectrum of **3c**

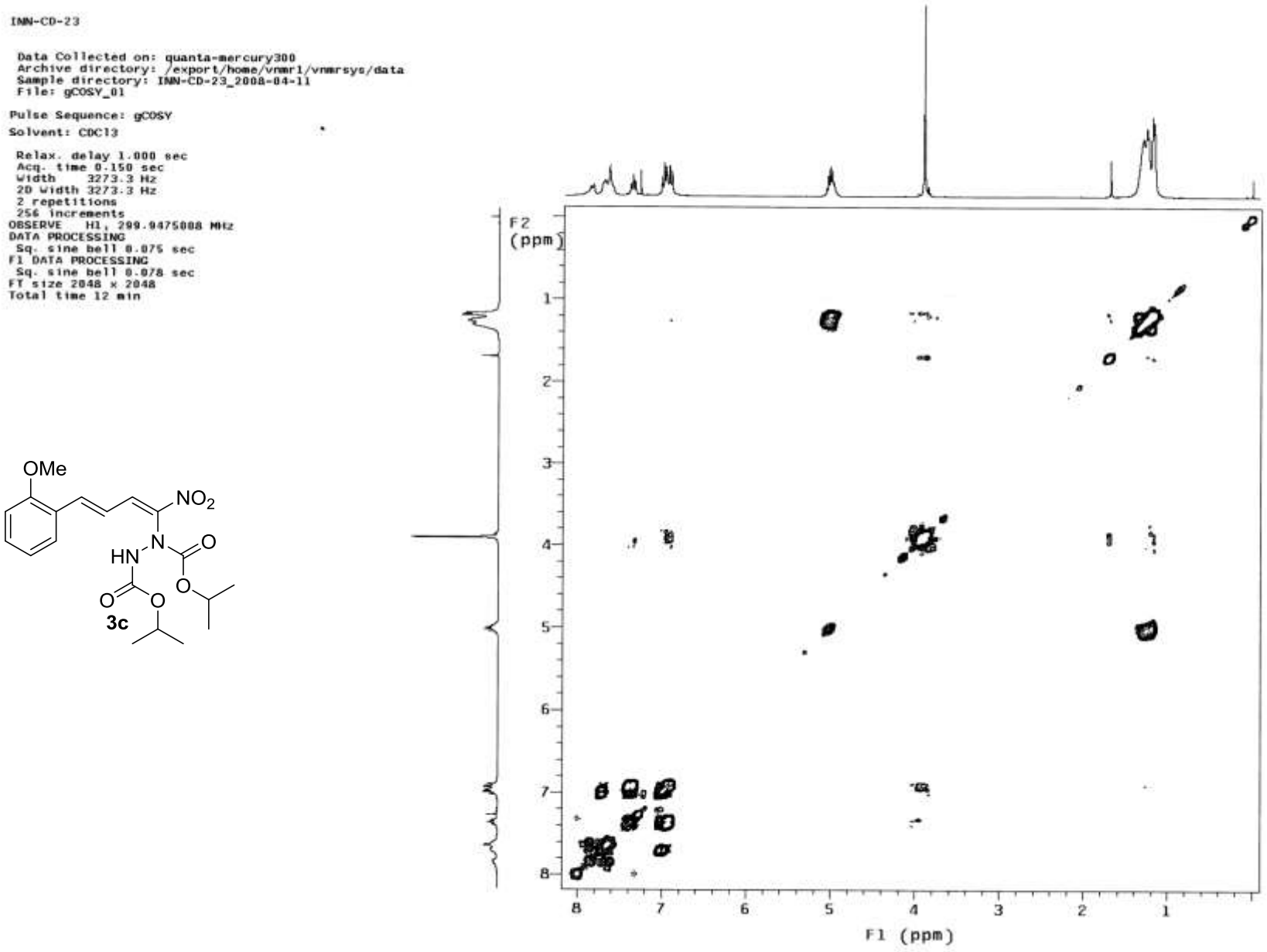


Figure S8. ^{1}H-^{1}H-COSY Spectrum of **3c**

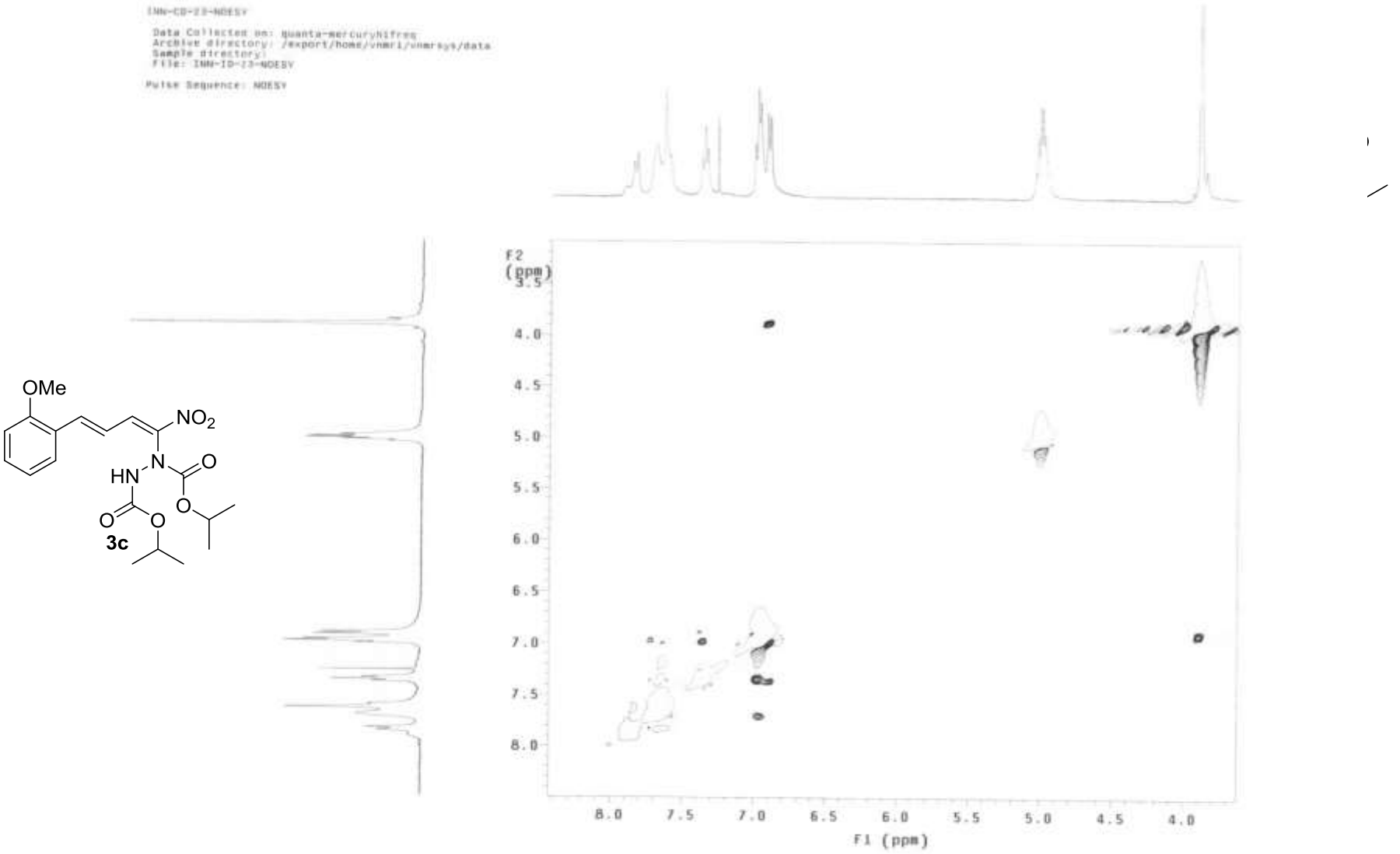


Figure S9. ^{1}H-^{1}H-NOESY Spectrum of **3c**Figure S10. ^{1}H NMR Spectrum of **3d**

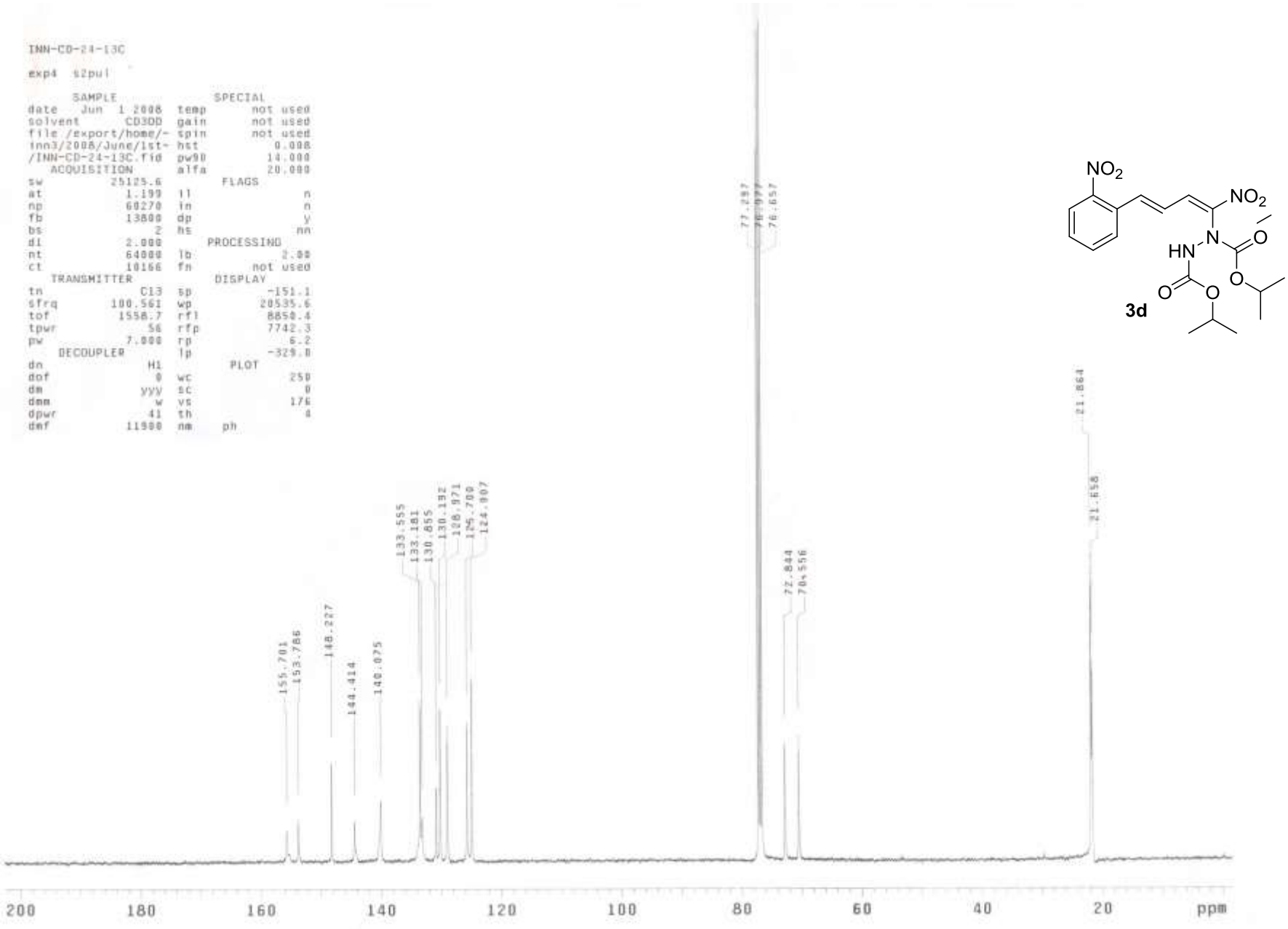


Figure S11. ^{13}C NMR Spectrum of **3d**

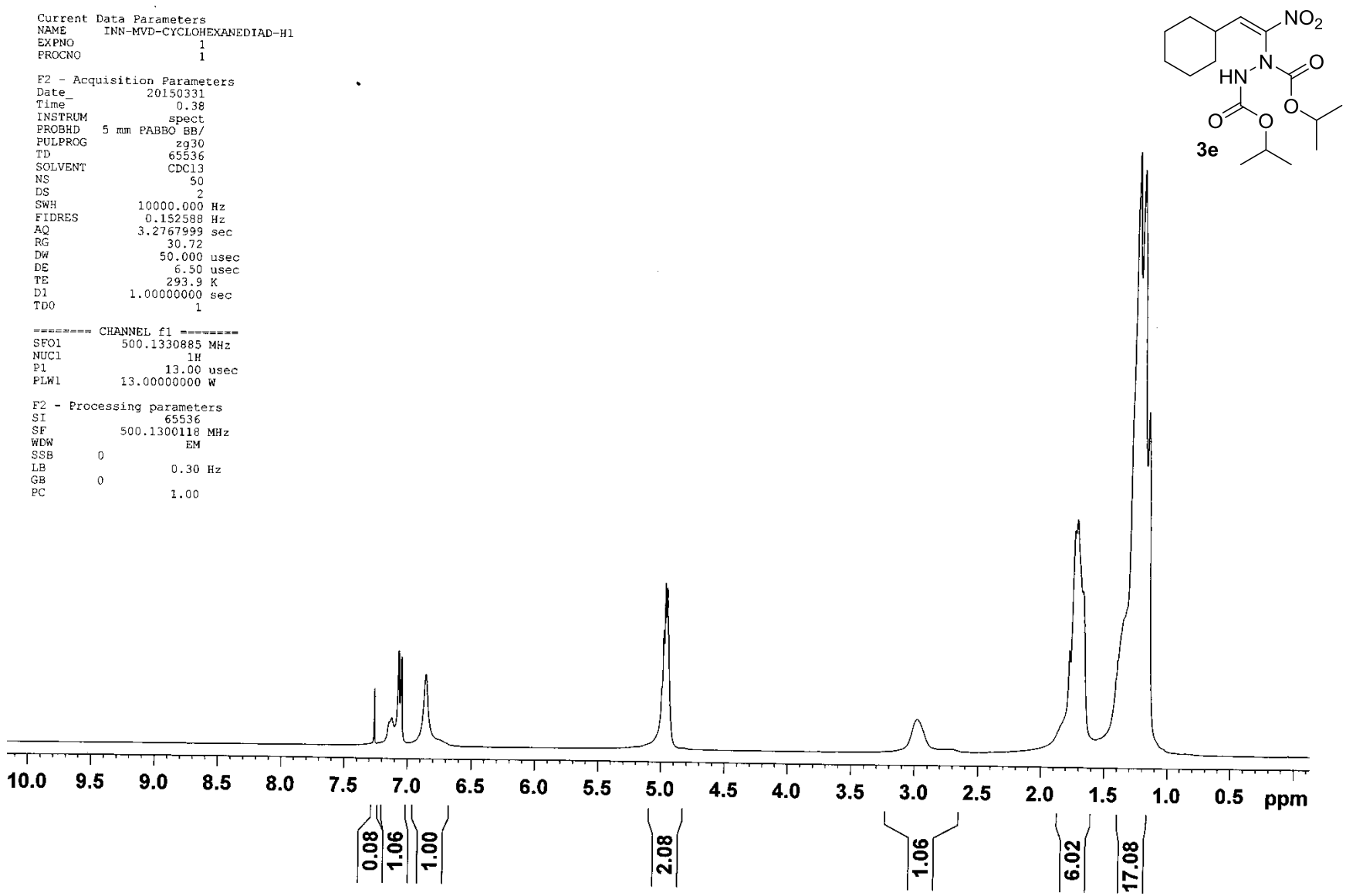


Figure S12. ^{1}H NMR Spectrum of **3e**

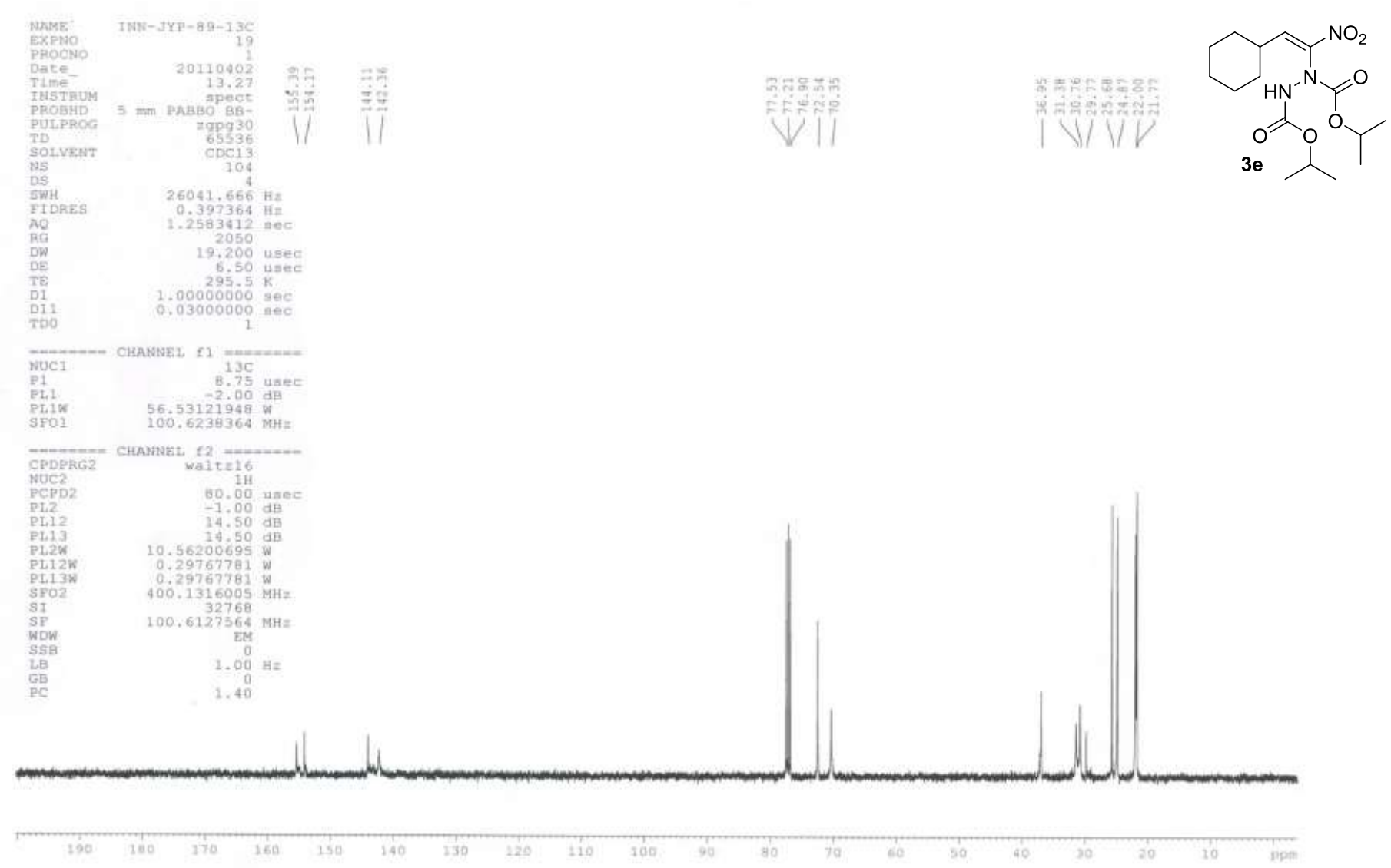


Figure S13. ^{13}C NMR Spectrum of **3e**

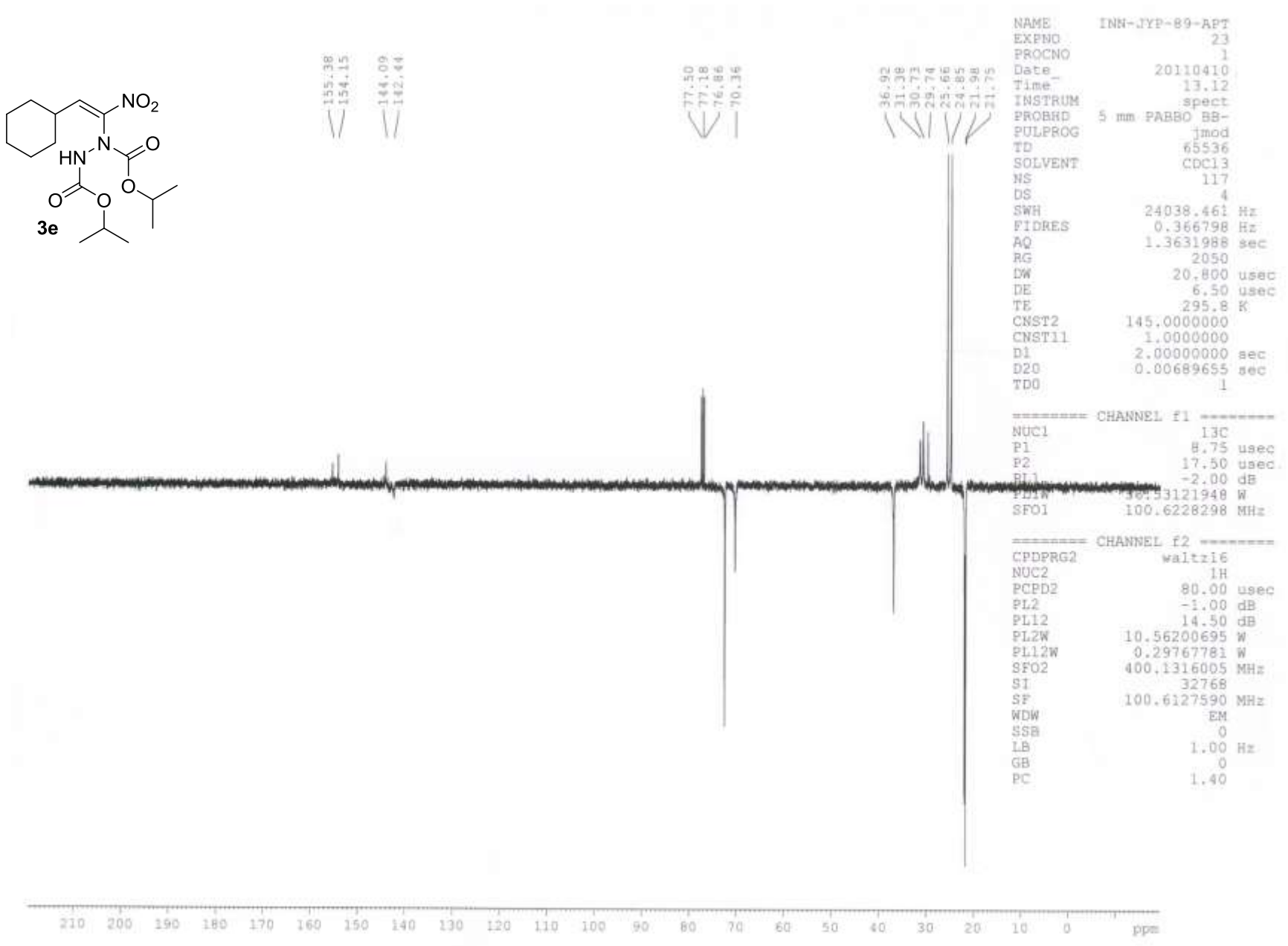
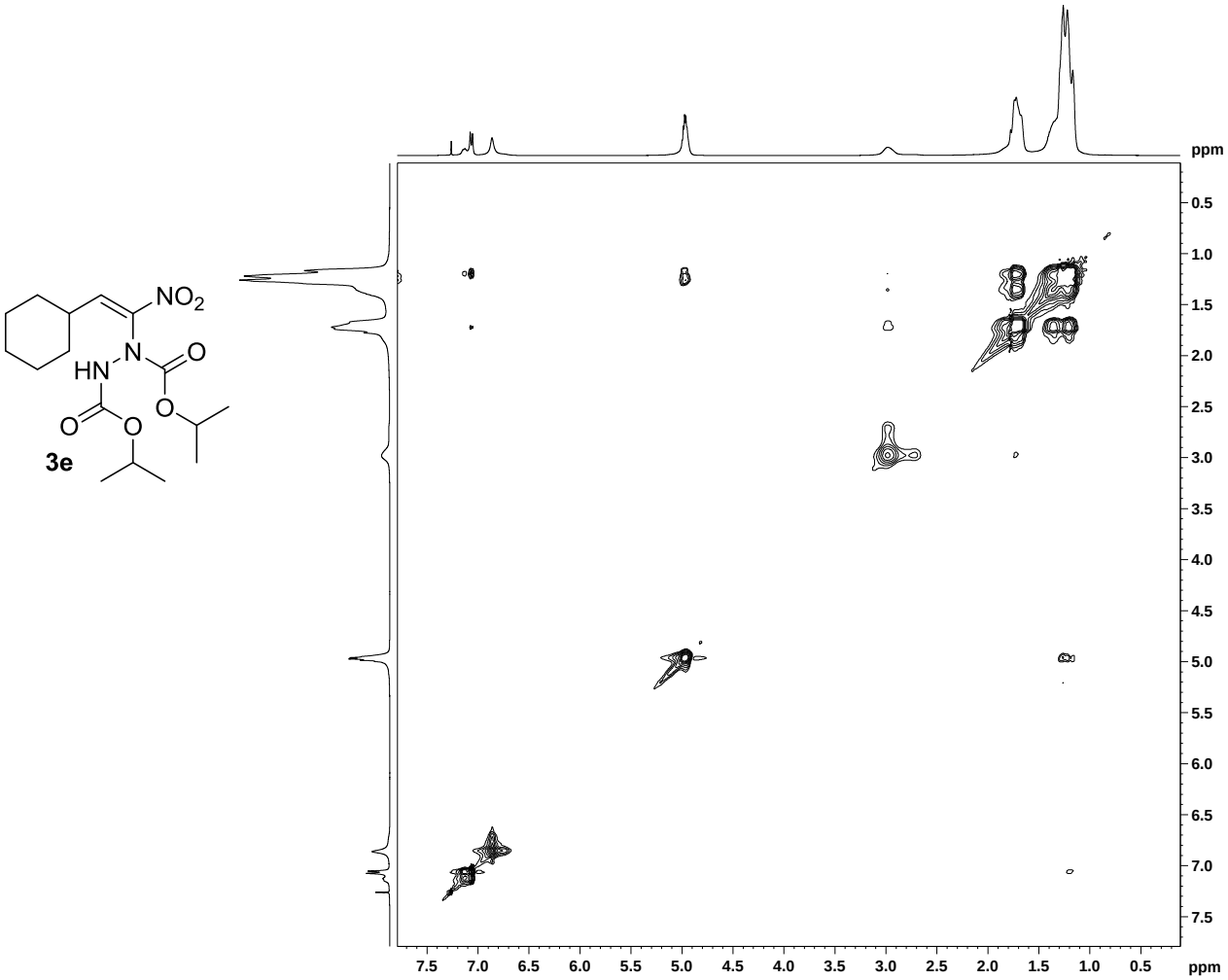


Figure S14. ^{13}C-APT Spectrum of **3e**




```
Current Data Parameters
NAME      INN-MVD-CYCLOHEXANEDIAD-NOESY
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150331
Time              0.43
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG       noesygpph
TD                2048
SOLVENT           CDCl3
NS                  16
DS                  32
SWH            3840.246 Hz
FIDRES         1.875120 Hz
AQ            0.2666496 sec
RG                85.91
DW              130.200 usec
DE                6.50 usec
TE               293.9 K
D0           0.00011365 sec
D1           1.00000000 sec
D8           0.60000002 sec
D16          0.00020000 sec
IN0          0.00026040 sec

======== CHANNEL f1 ========
SFO1         500.1319879 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                 915
SFO1            500.132 MHz
FIDRES         4.196990 Hz
SW               7.678 ppm
FnMODE            TPPI

F2 - Processing parameters
SI                1024
SF          500.1300116 MHz
WDW               QSINE
SSB                  2
LB        0 Hz
GB        0
PC                1.00

F1 - Processing parameters
SI                1024
MC2               TPPI
SF          500.1300116 MHz
WDW               QSINE
SSB                  2
LB        0 Hz
GB        0
```

Figure S15. ^{1}H-^{1}H NOESY Spectrum of **3e**

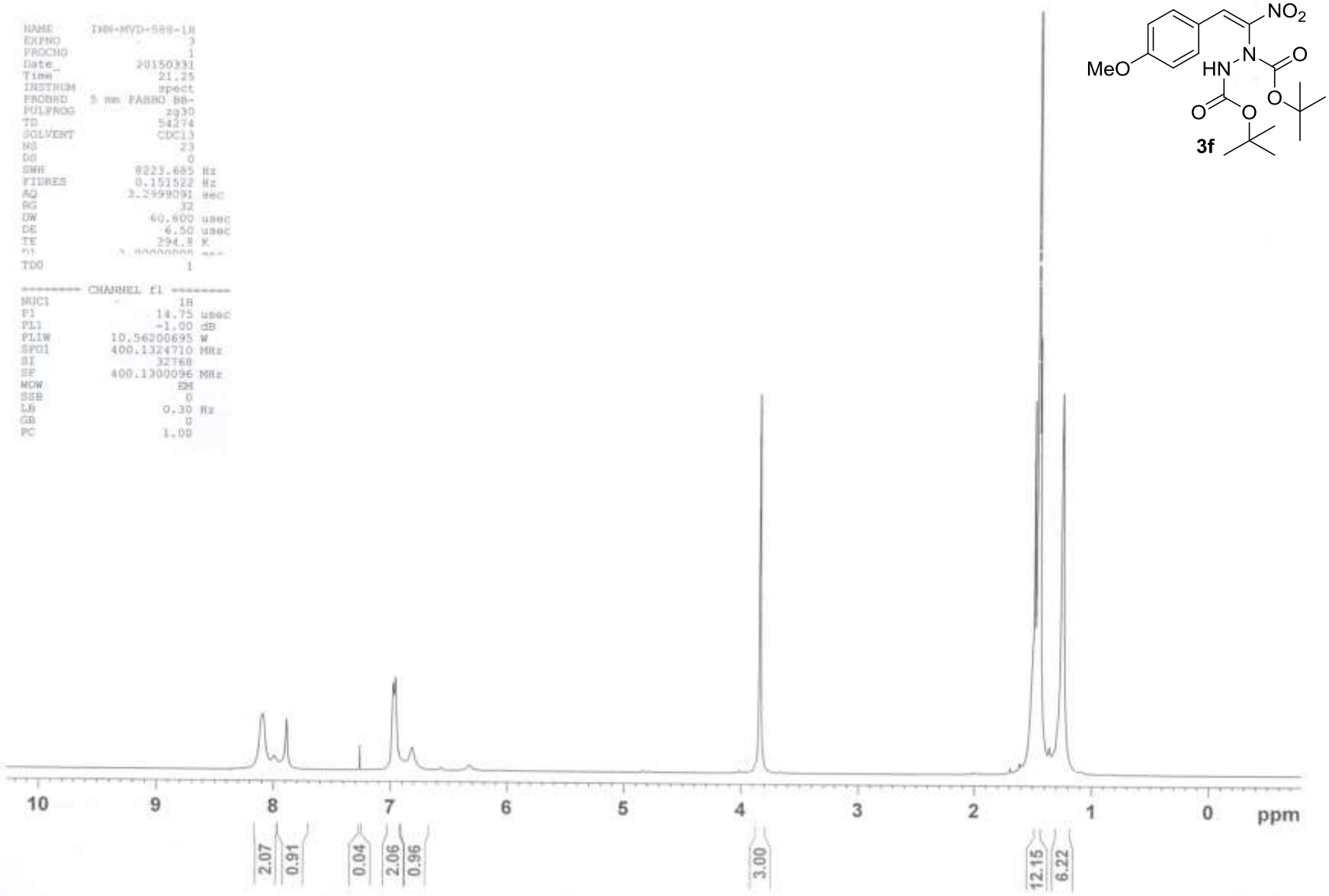


Figure S16. ^{1}H NMR Spectrum of **3f**

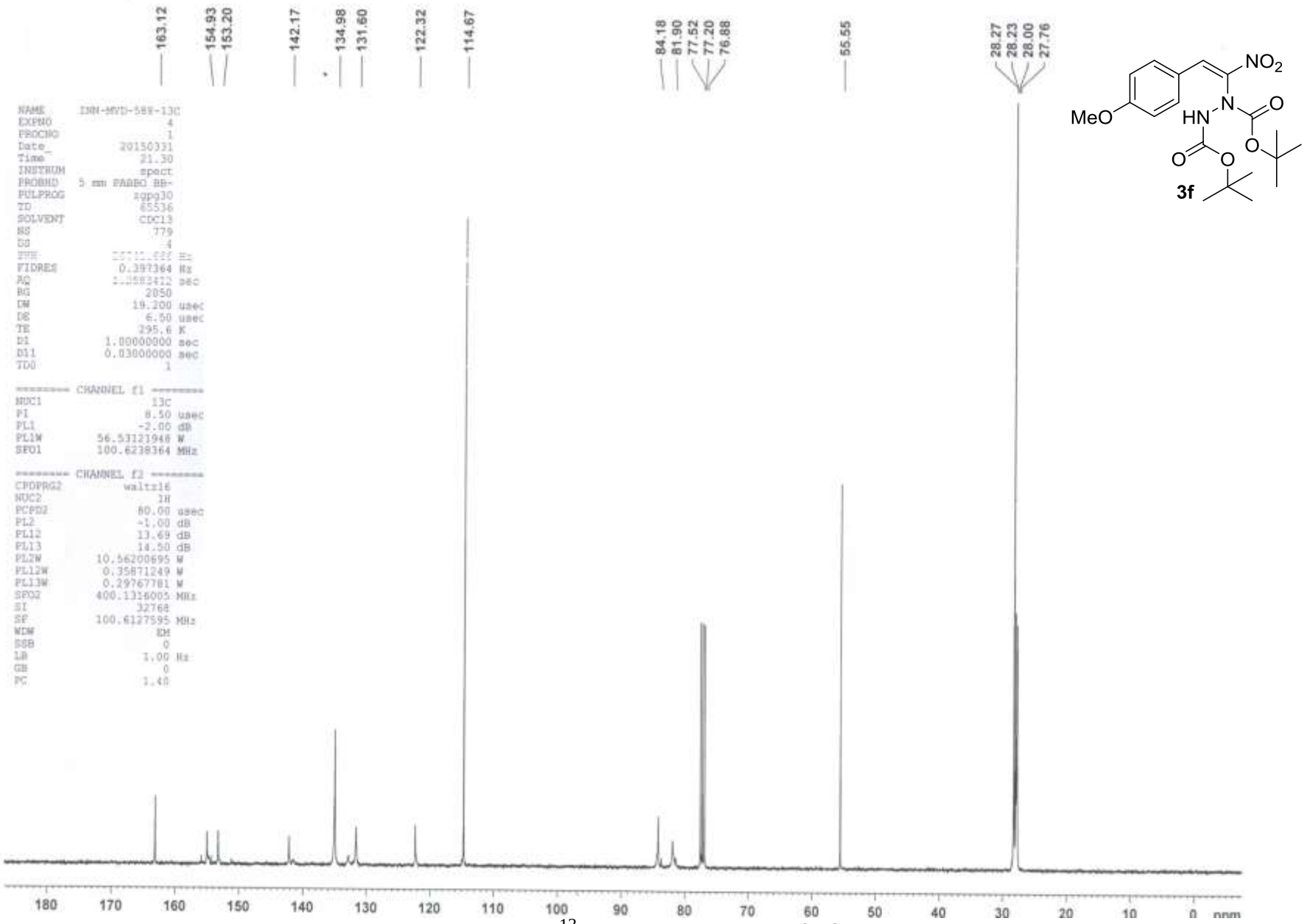


Figure S17. ^{13}C NMR Spectrum of **3f**

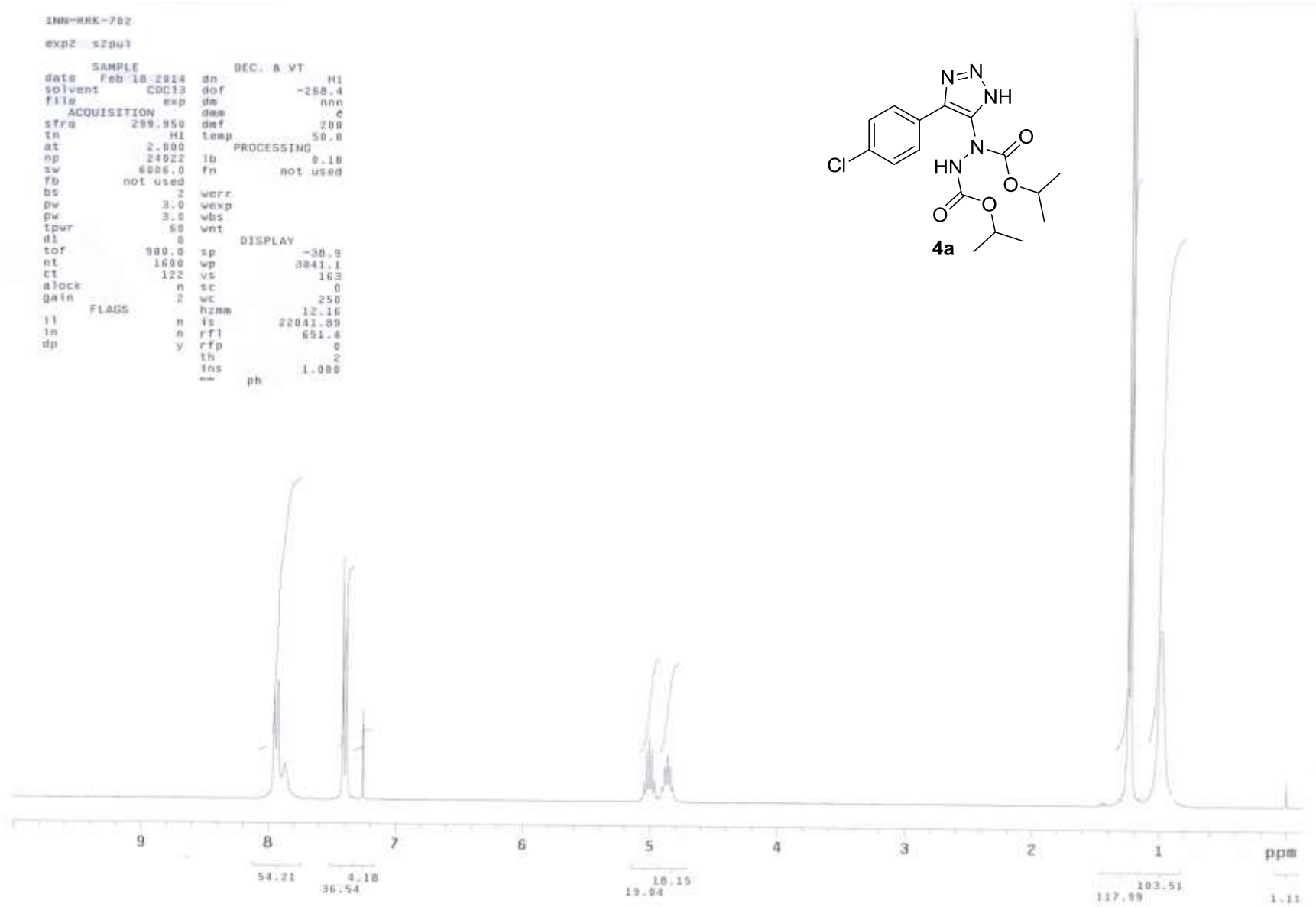


Figure S18. ^{1}H NMR Spectrum of **4a**

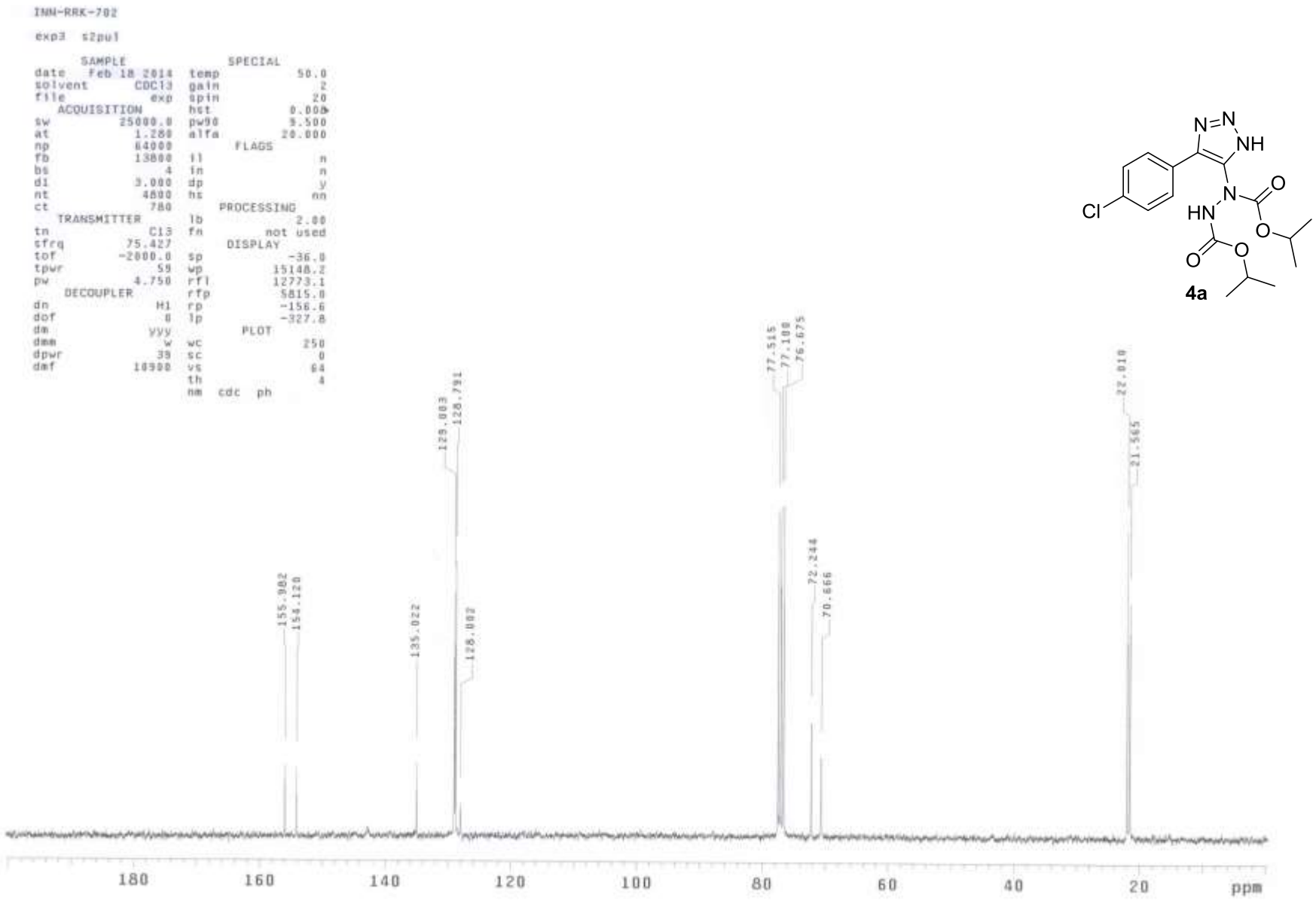


Figure S19. ^{13}C NMR Spectrum of **4a**

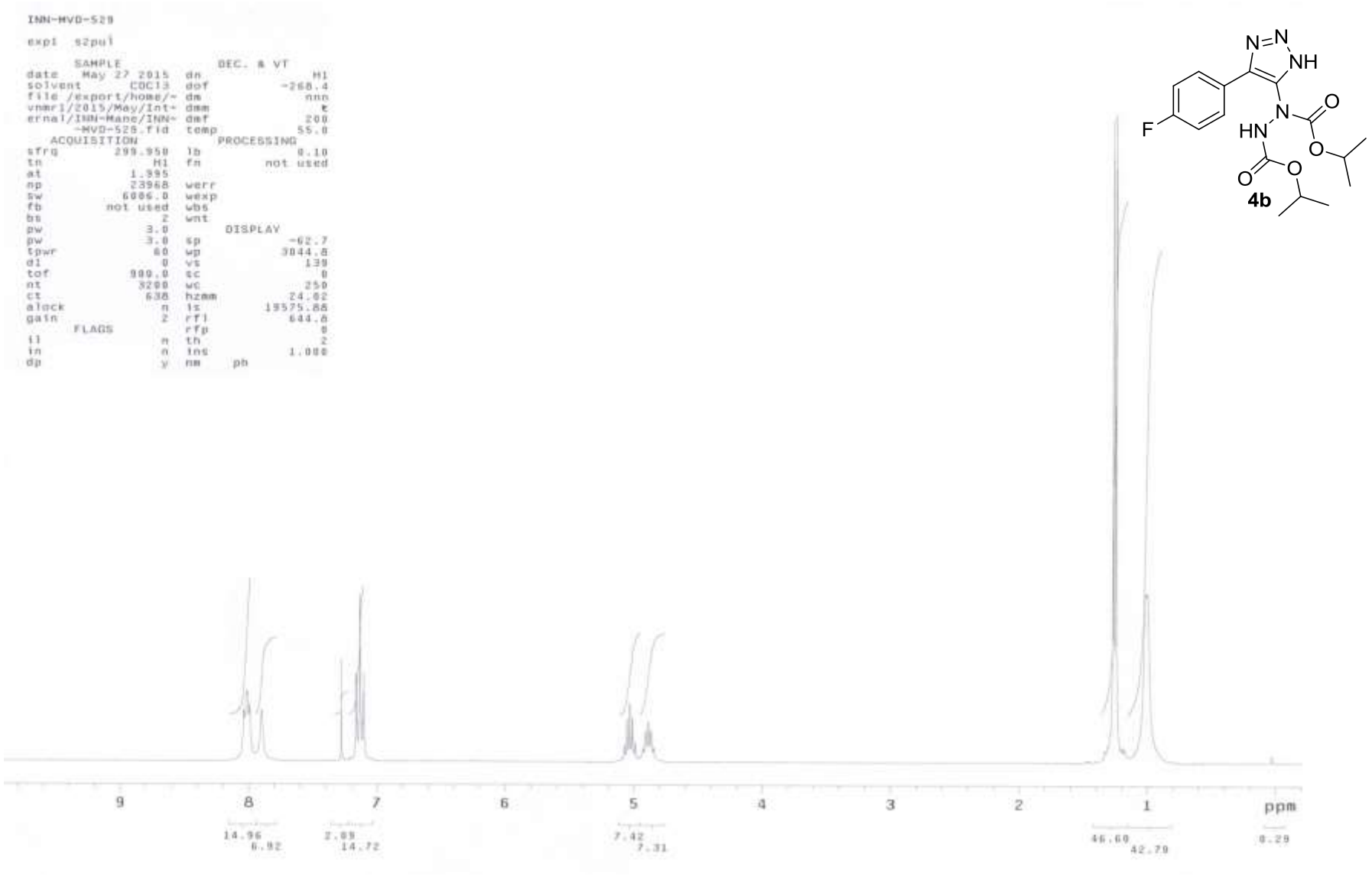


Figure S20. ^{1}H NMR Spectrum of **4b**

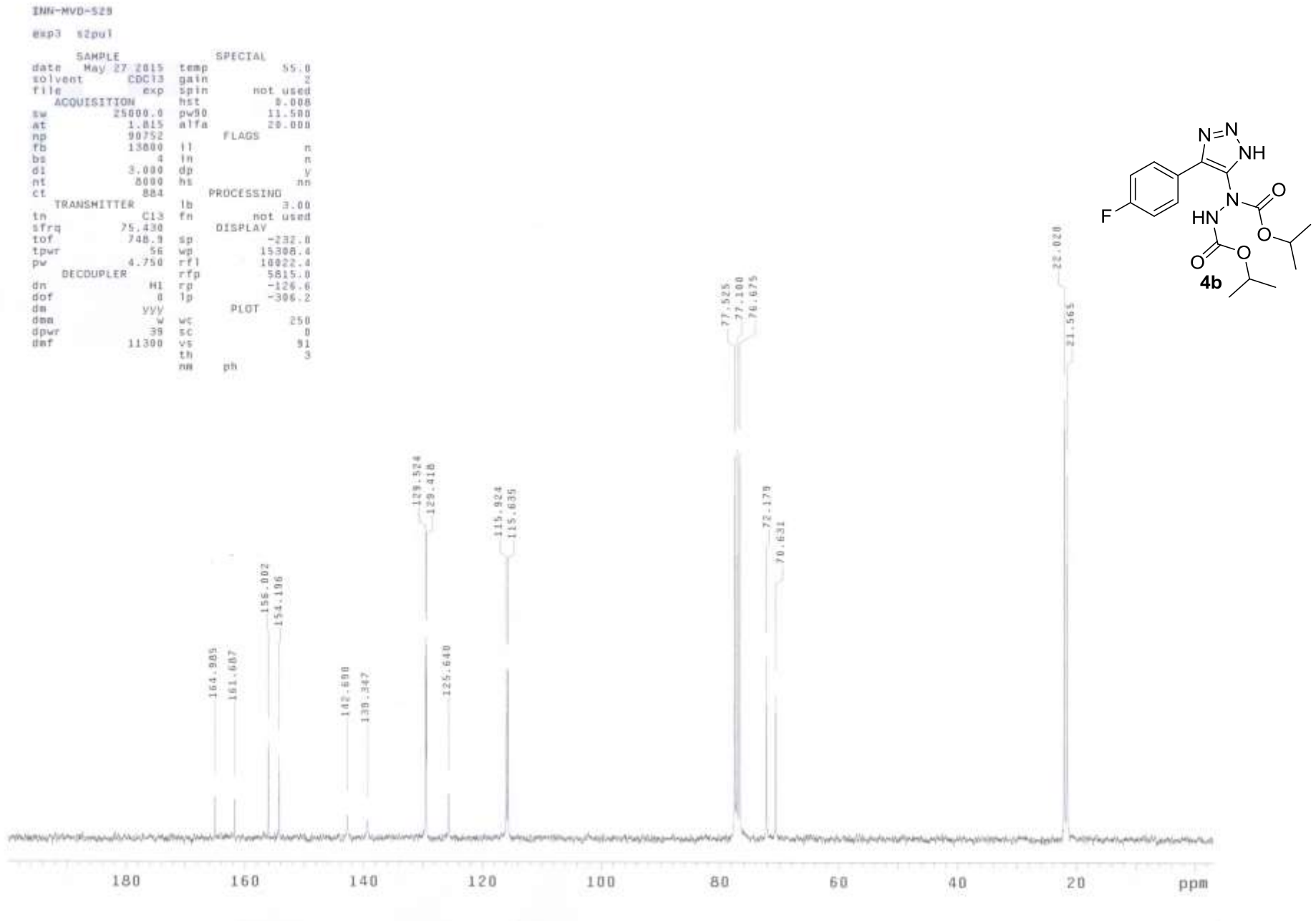


Figure S21. ^{13}C NMR Spectrum of **4b**

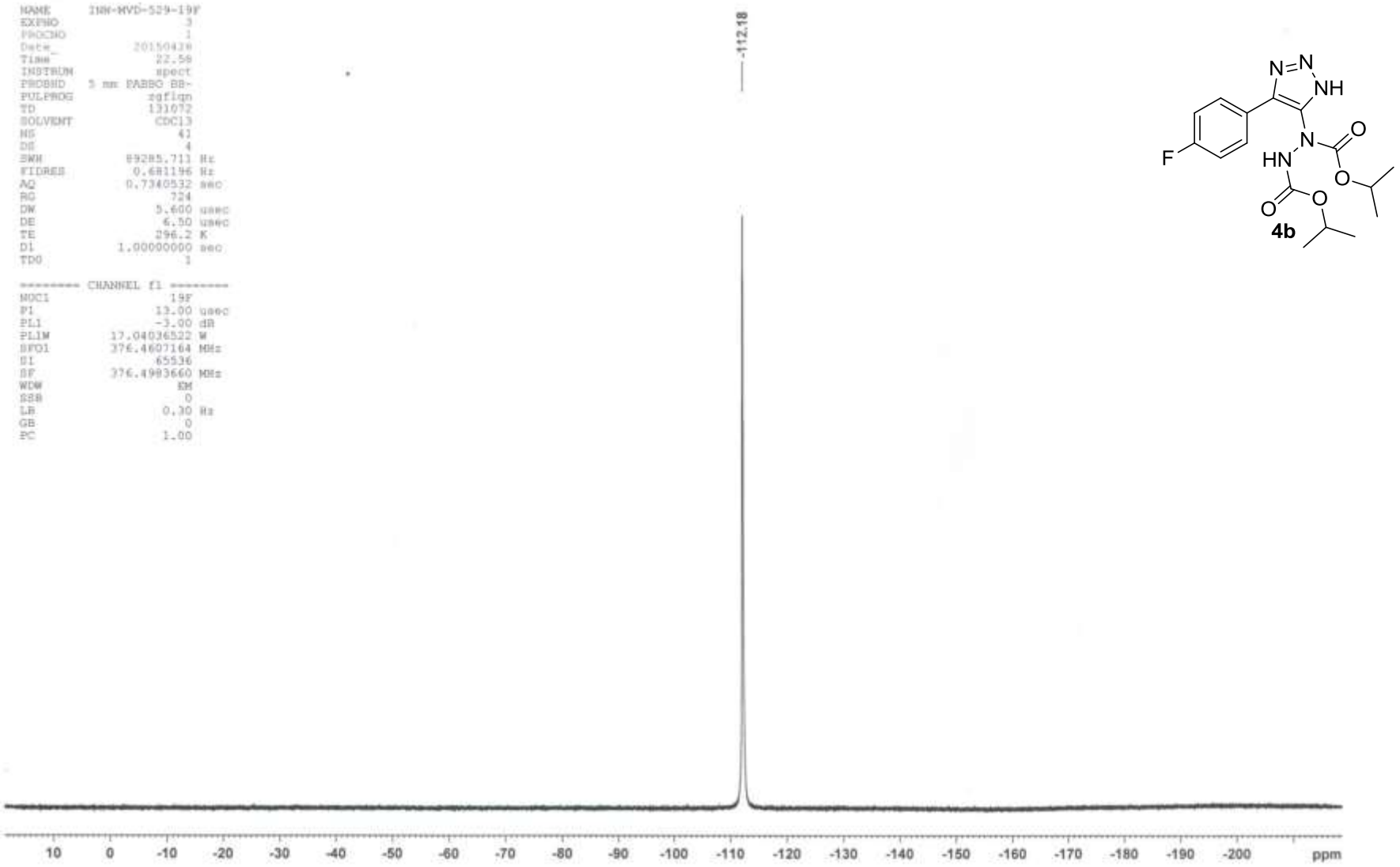


Figure S22. ^{19}F NMR Spectrum of **4b**

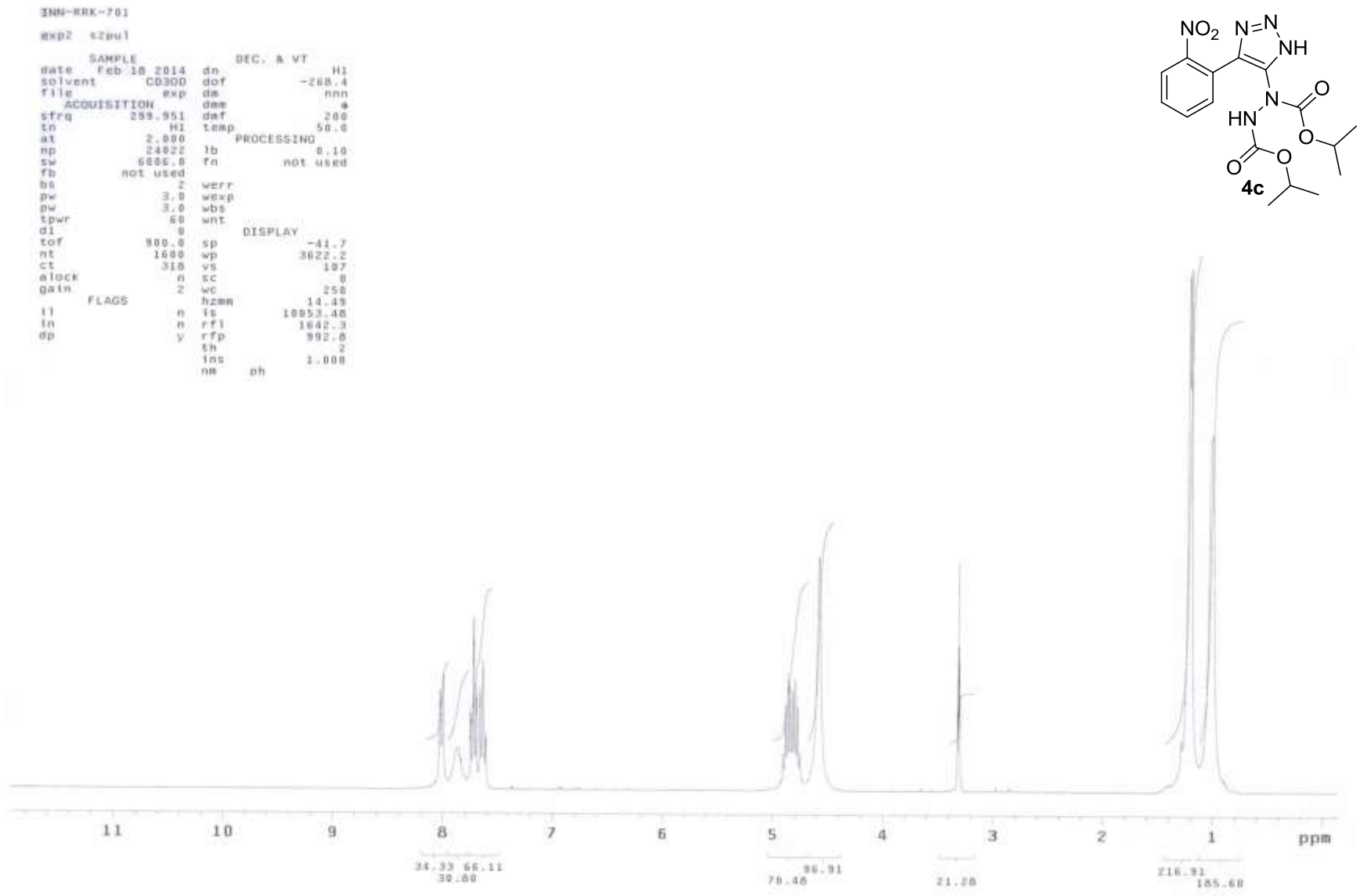


Figure S23. ^{1}H NMR Spectrum of **4c**

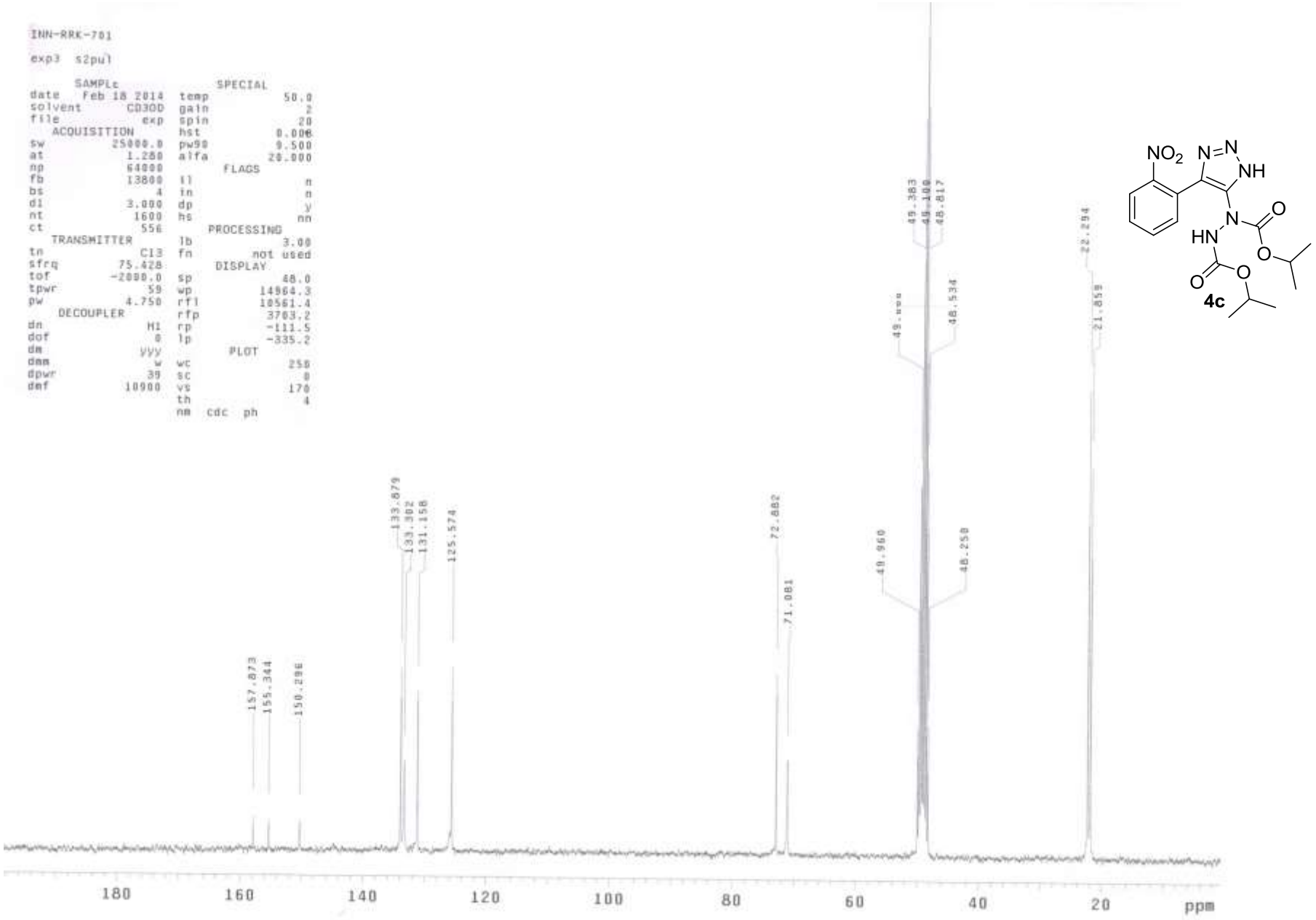


Figure S24. ^{13}C NMR Spectrum of **4c**

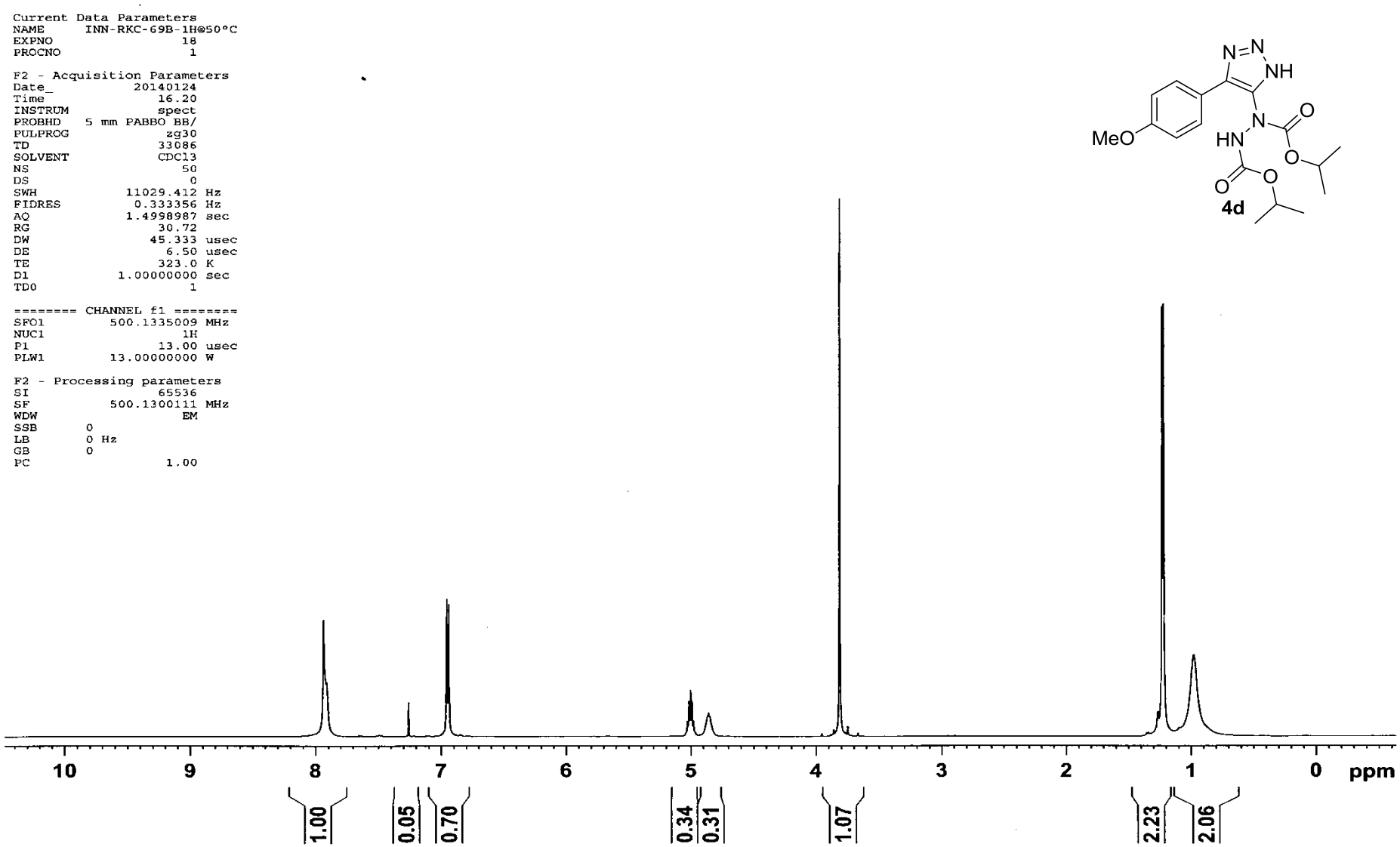


Figure S25. ^{1}H NMR Spectrum of **4d**

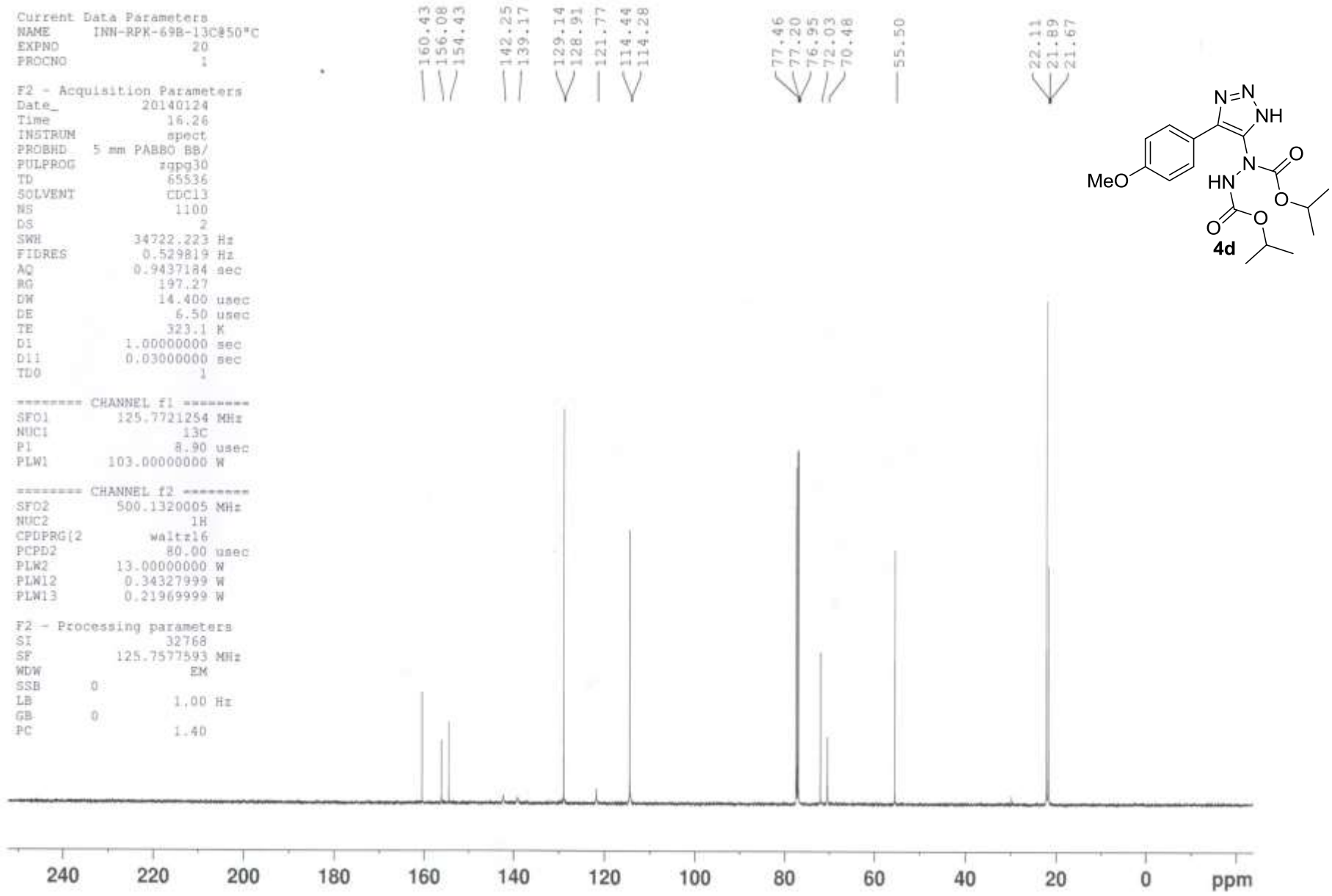


Figure 26.^{13}C NMR Spectrum of **4d**

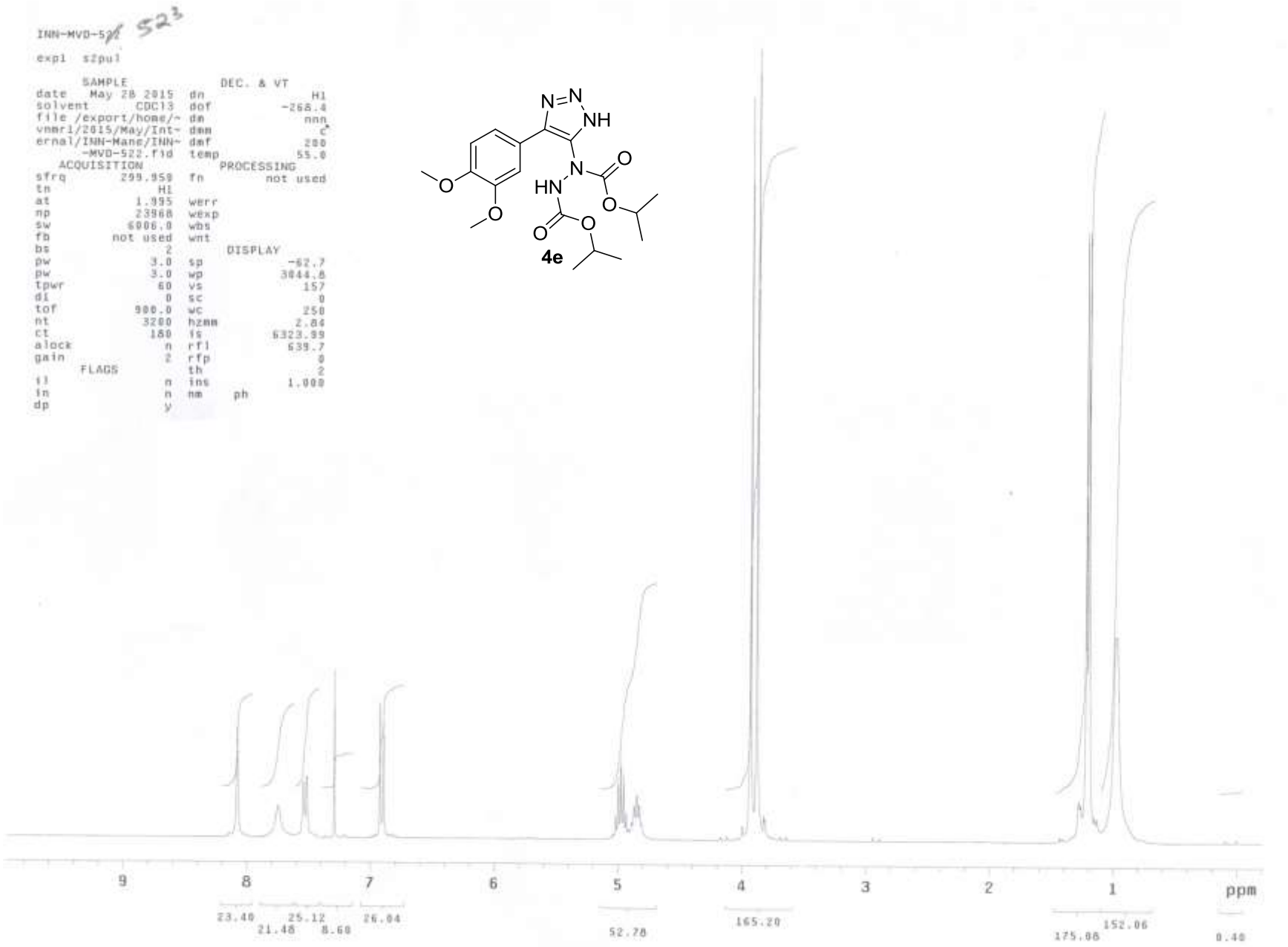


Figure S27. ^{1}H NMR Spectrum of **4e**

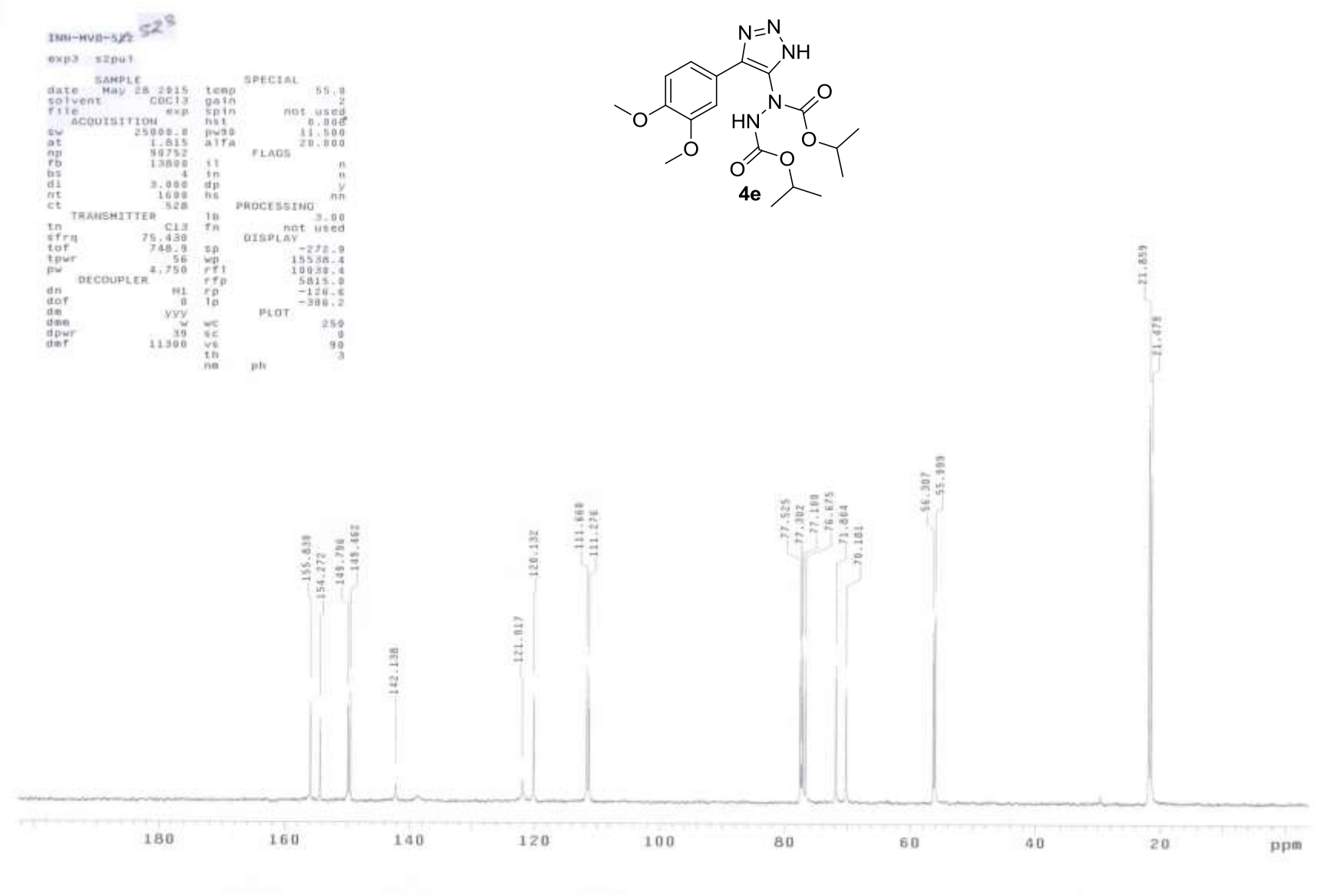


Figure S28. ^{13}C NMR Spectrum of **4e**

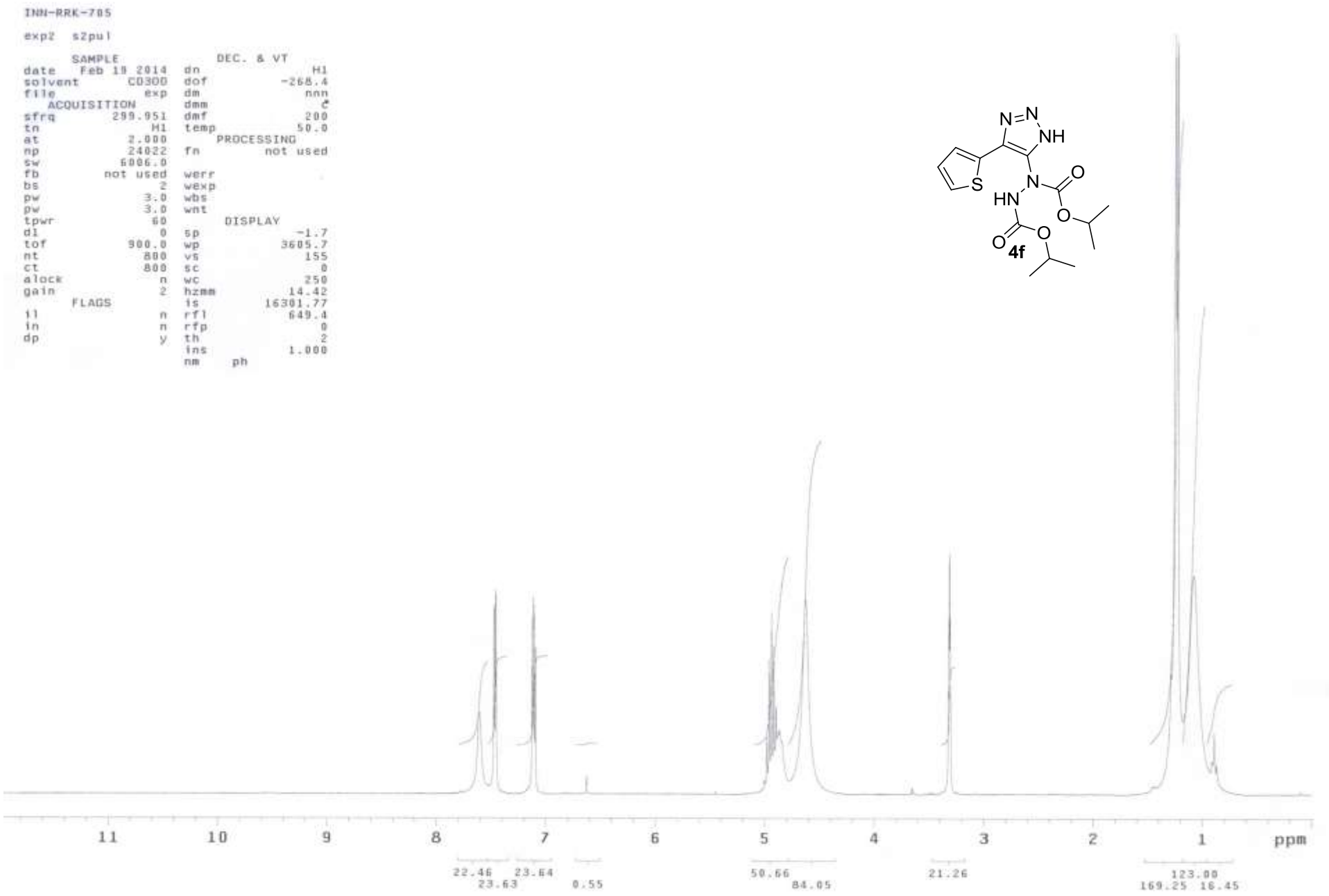


Figure S29. ^{1}H NMR Spectrum of **4f**

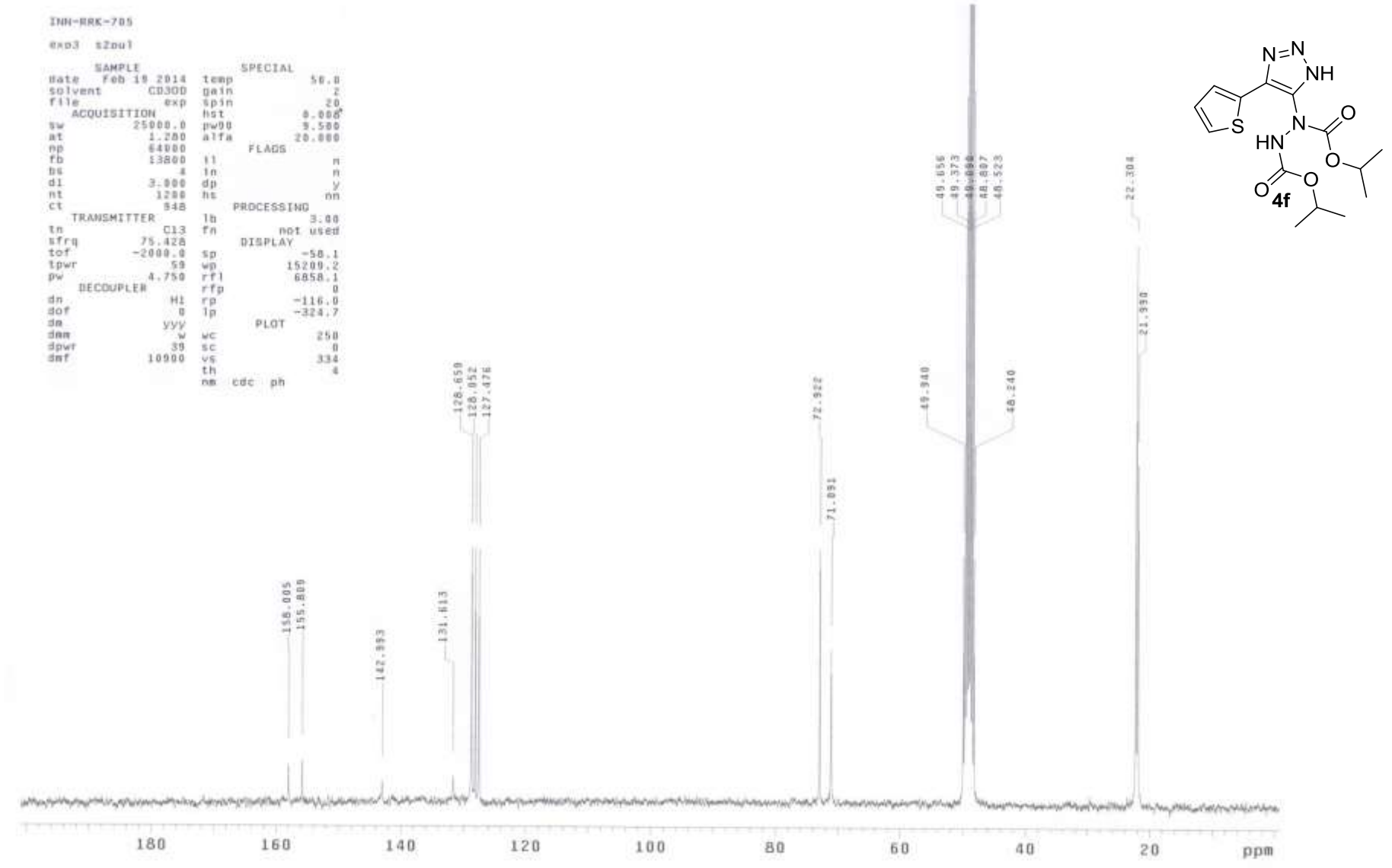


Figure S30. ^{13}C NMR Spectrum of **4f**

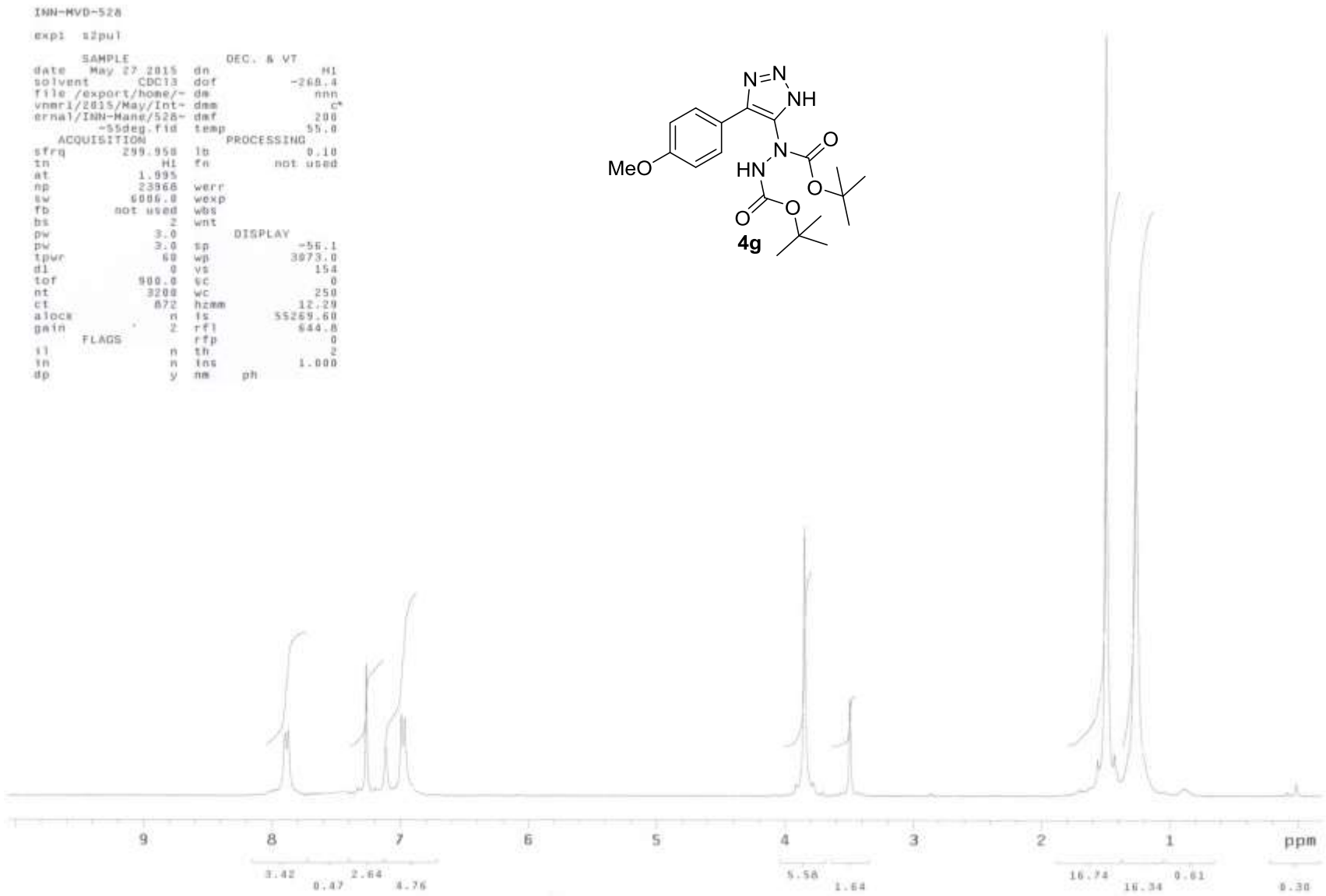


Figure S31. ^{1}H NMR Spectrum of **4g**

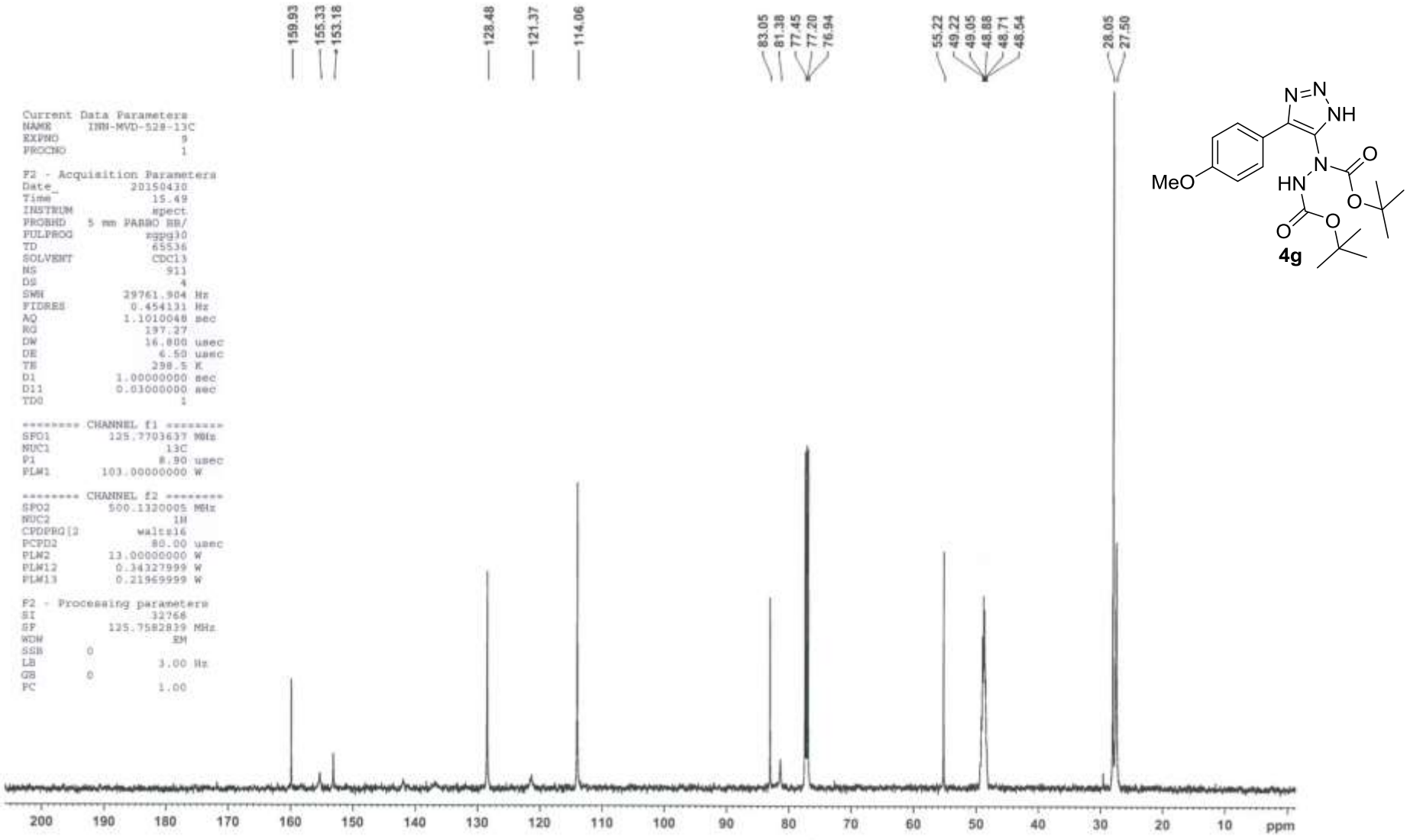


Figure S32. ^{13}C NMR Spectrum of **4g**

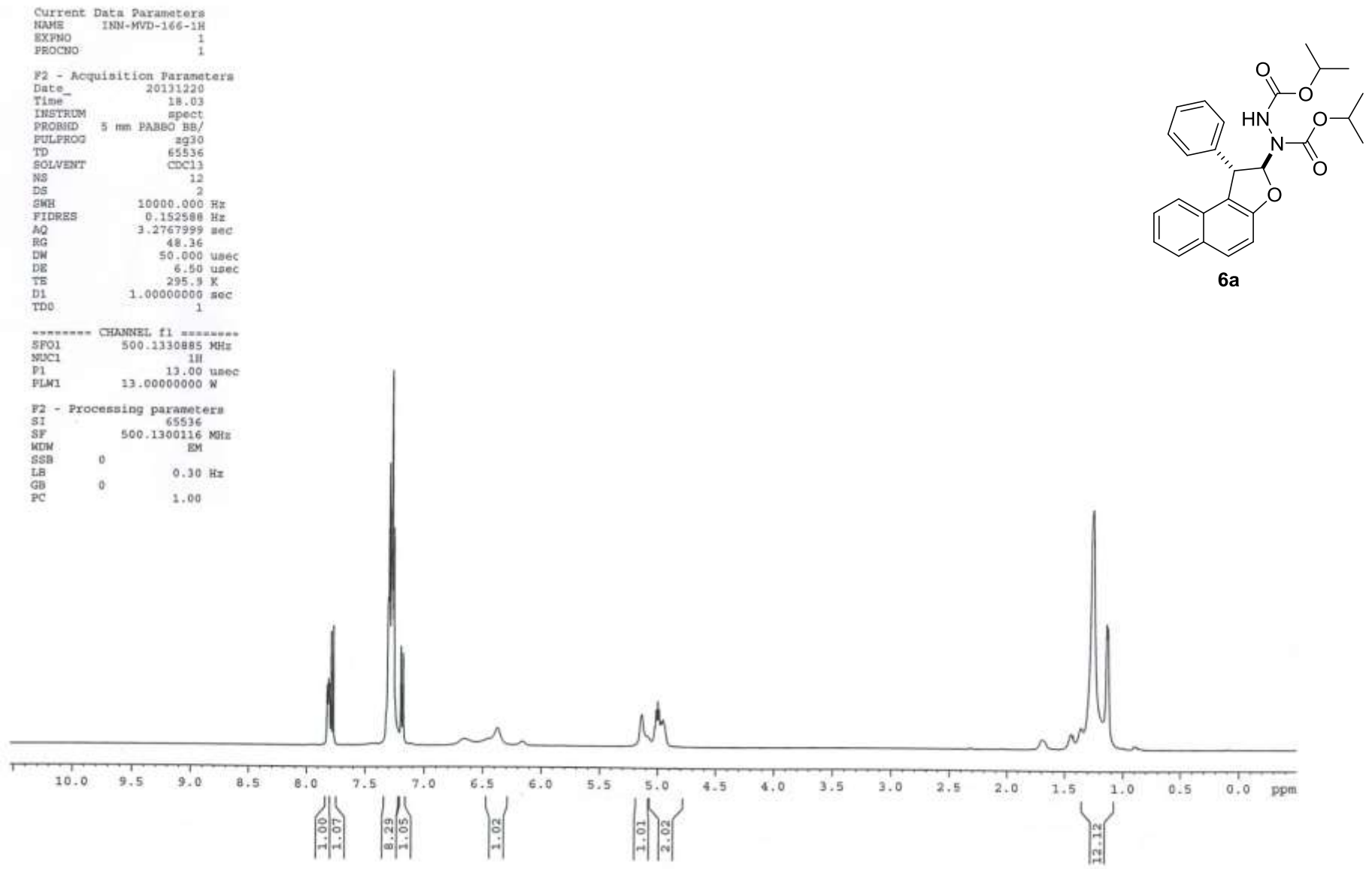


Figure S33. ^{1}H NMR Spectrum of **6a**

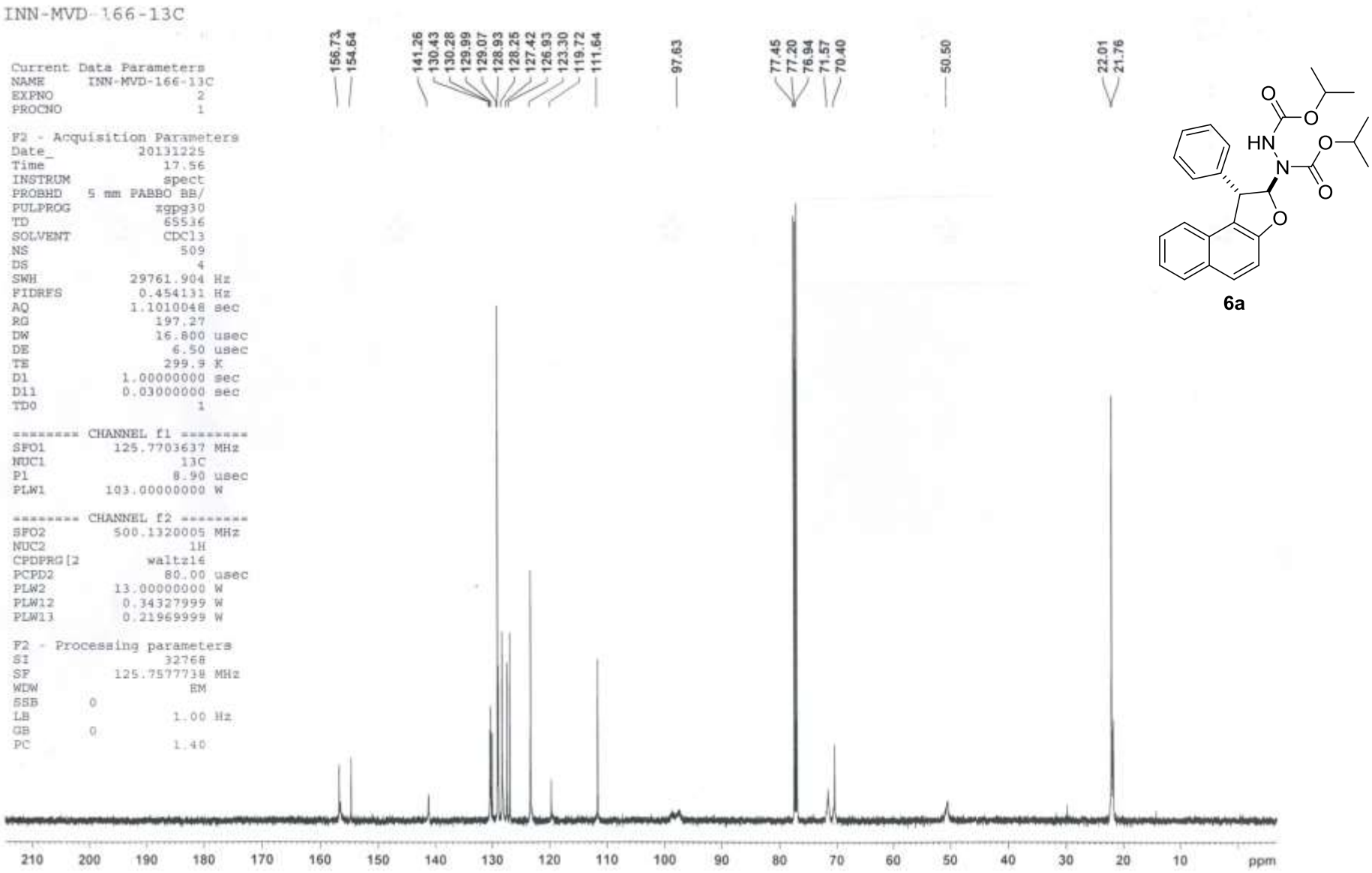


Figure S34. ^{13}C NMR Spectrum of **6a**

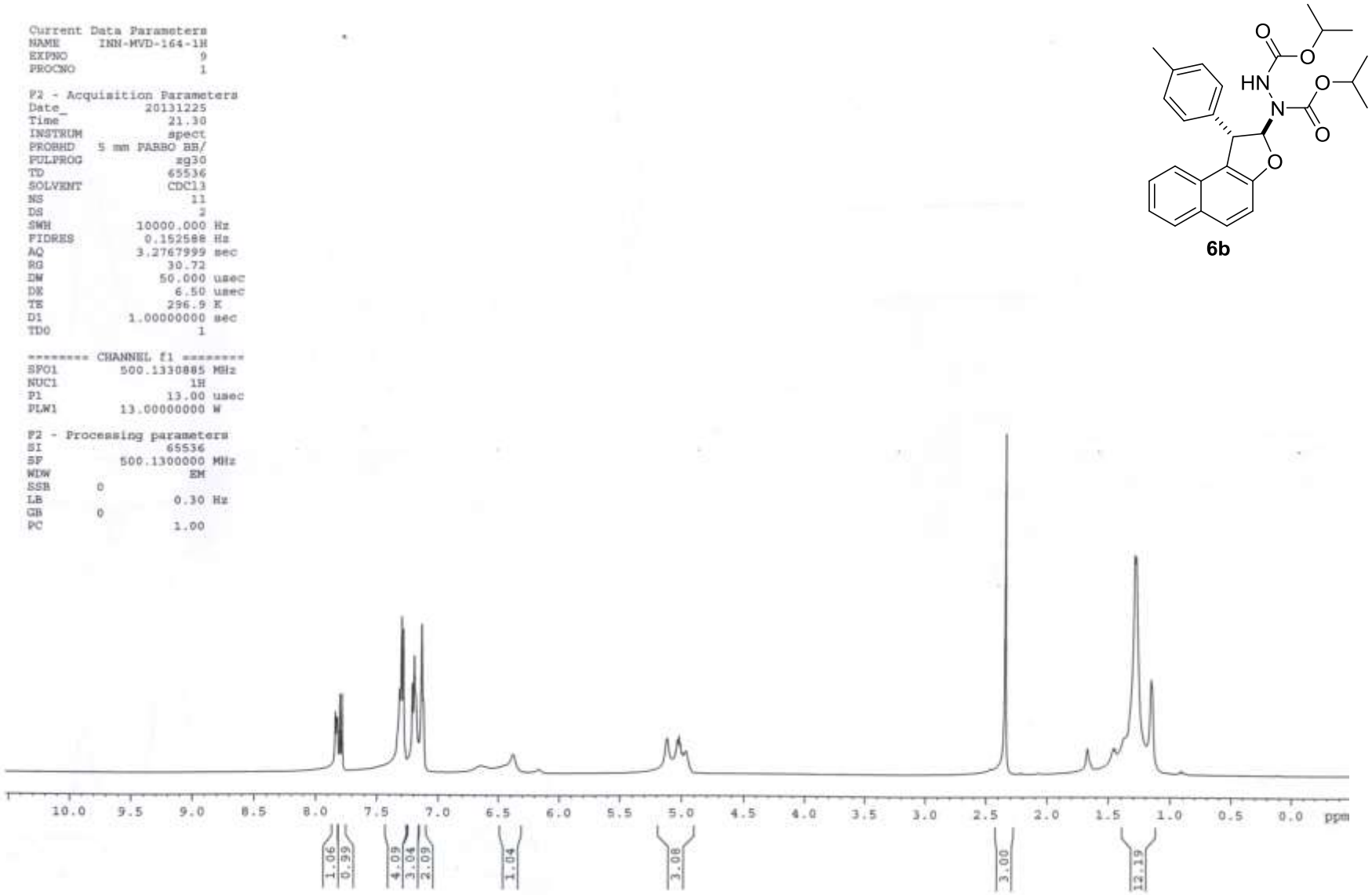


Figure S35. ^{1}H NMR Spectrum of **6b**

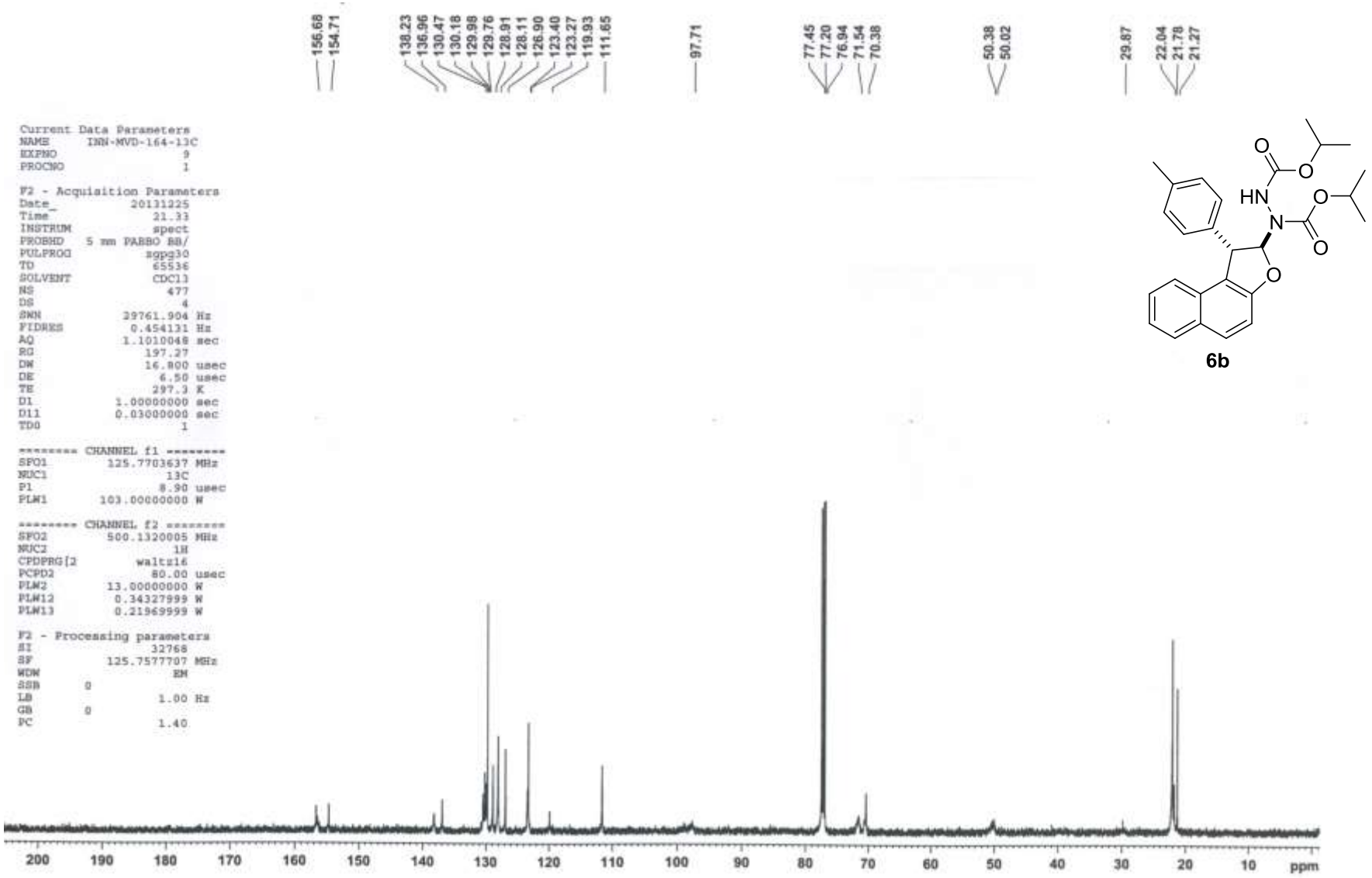


Figure S36. ^{13}C NMR Spectrum of **6b**

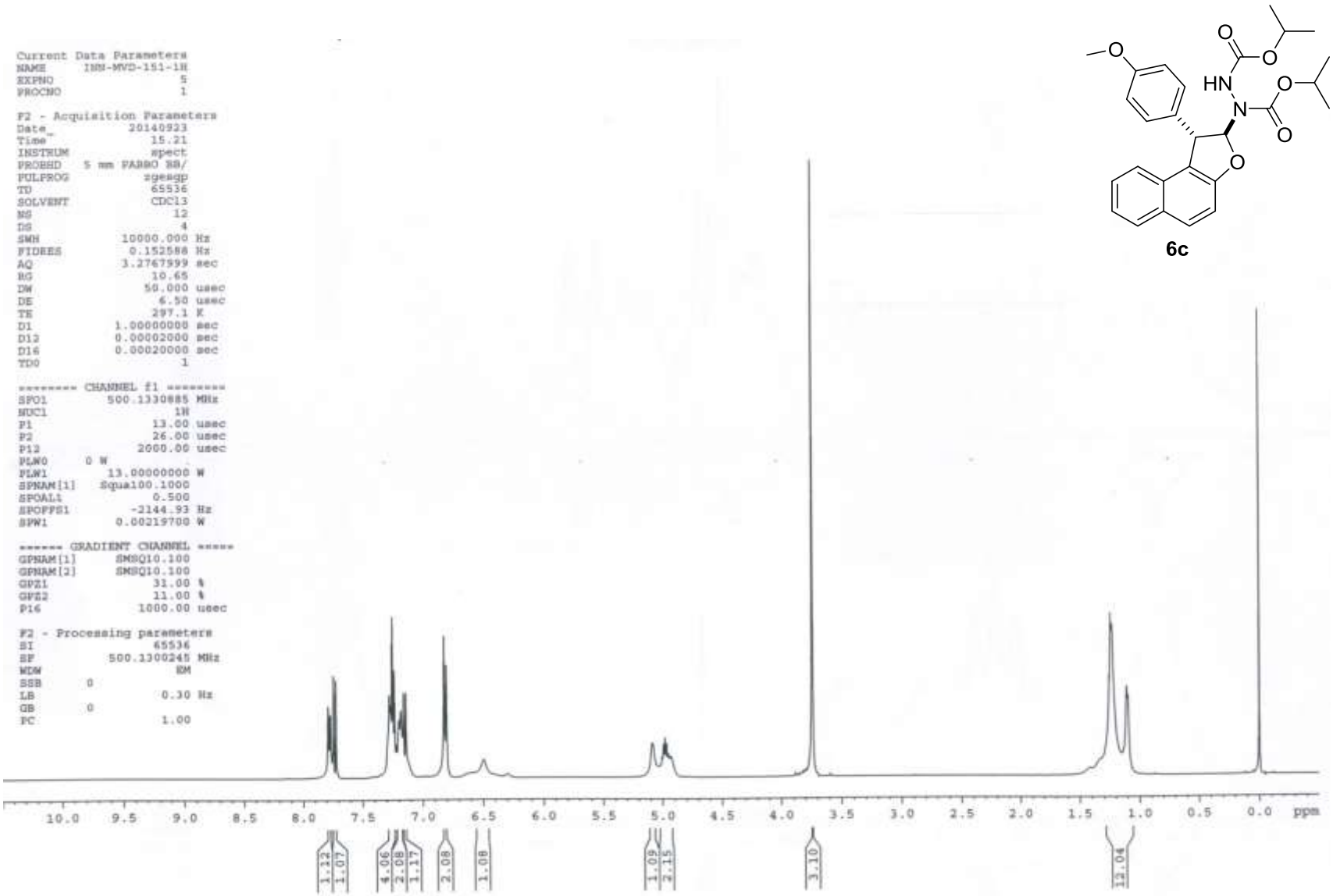


Figure S37. ^{1}H NMR Spectrum of **6c**

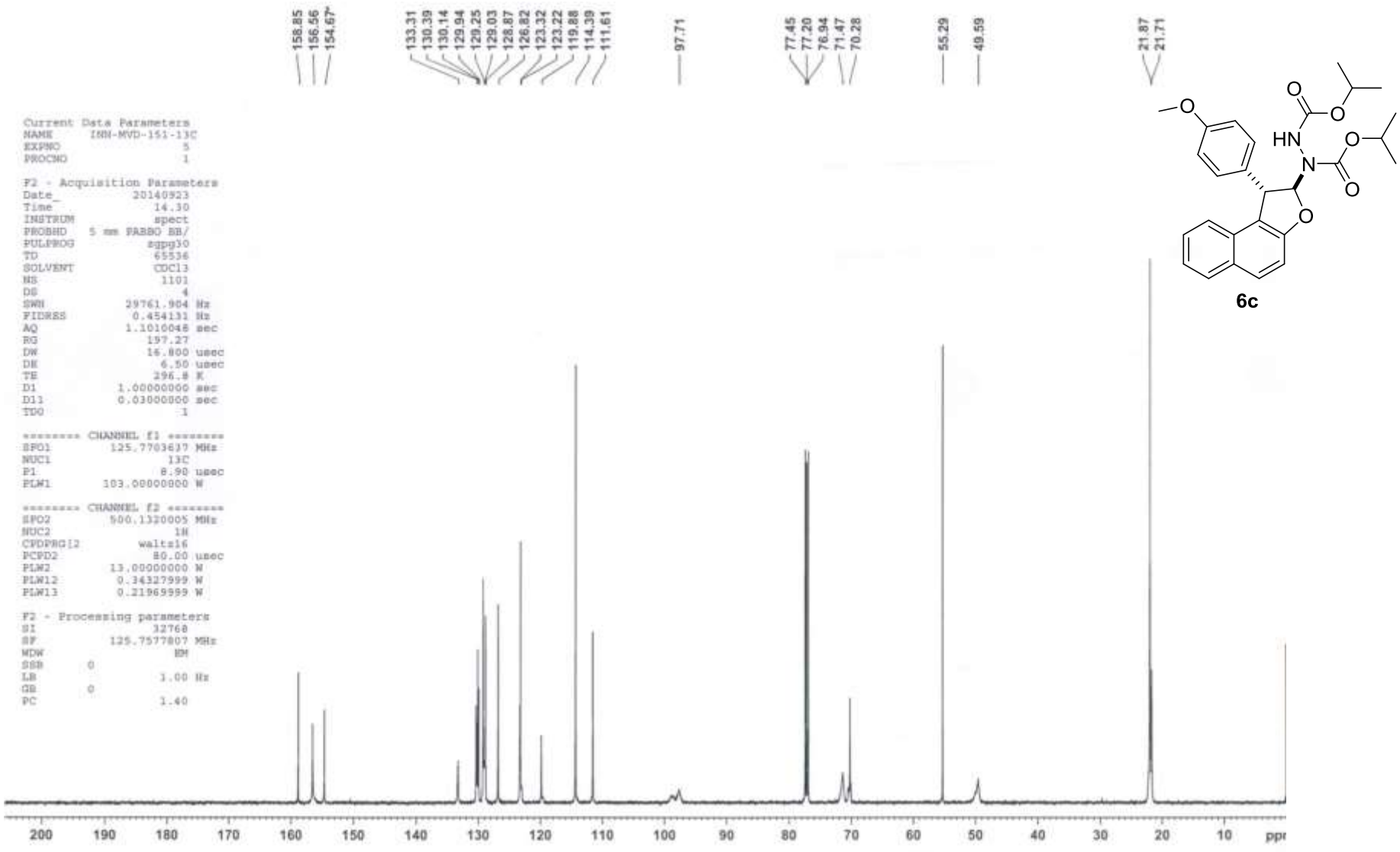


Figure S38. ^{13}C NMR Spectrum of **6c**

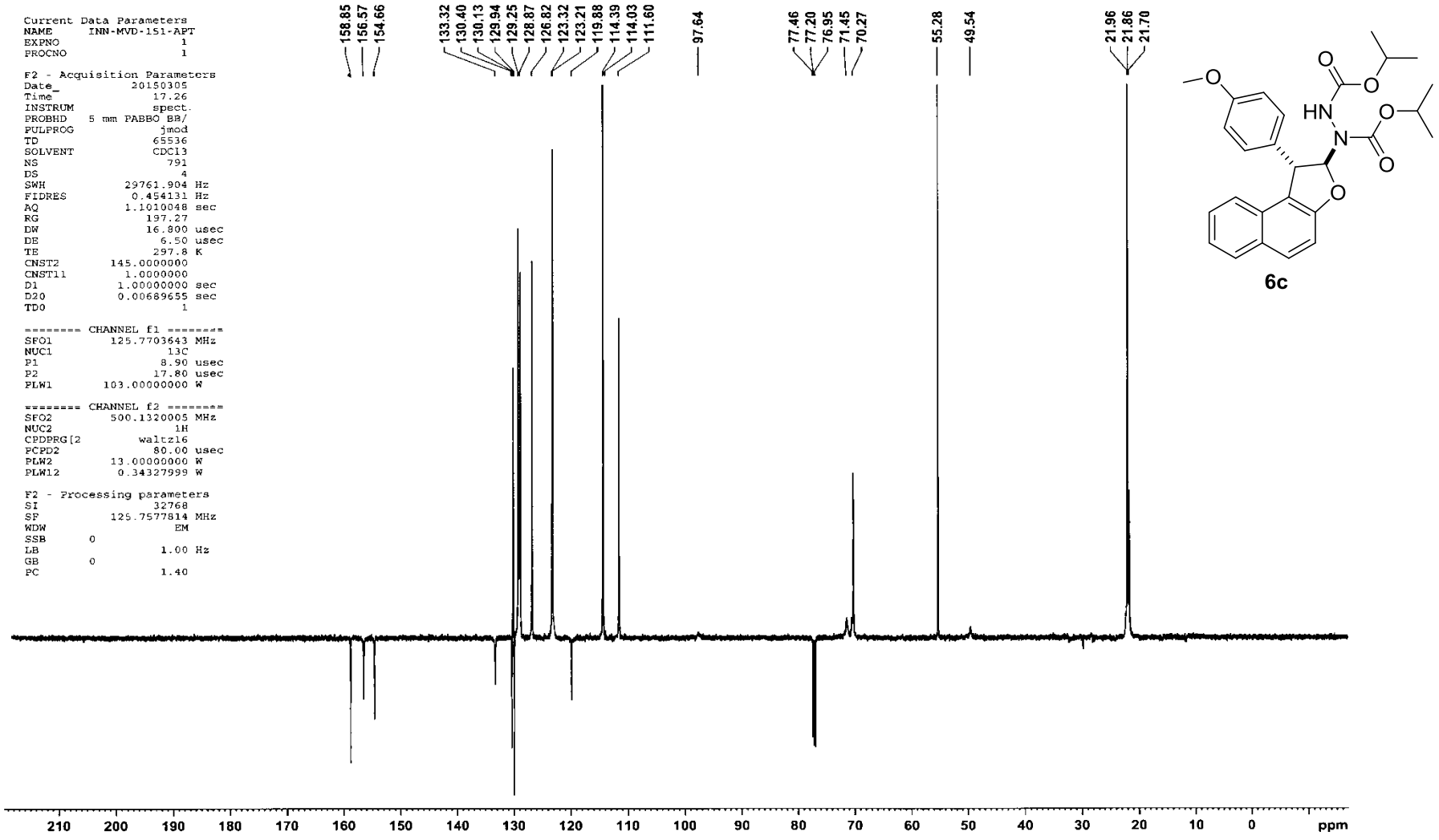
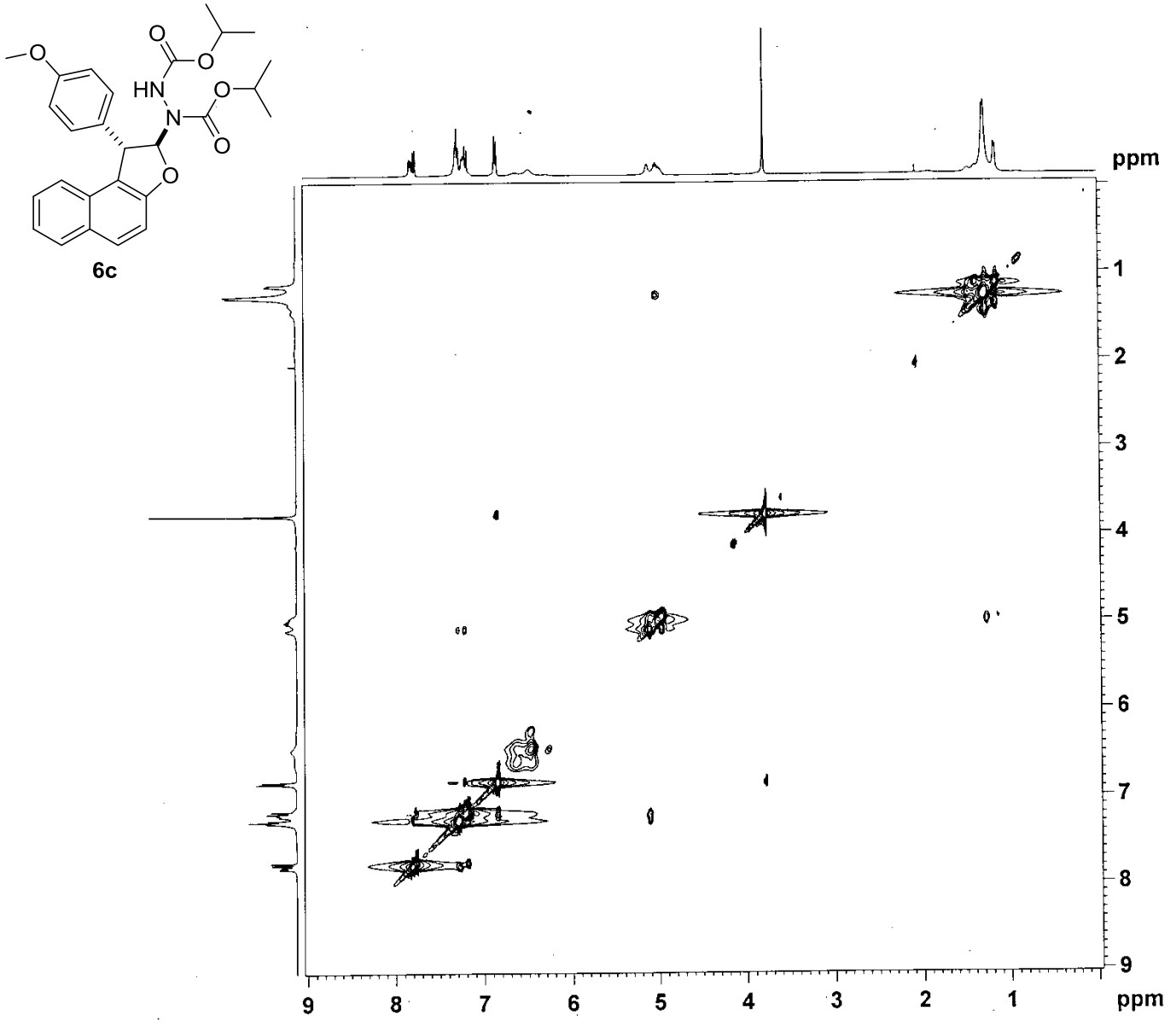


Figure S39. ^{13}C-APT NMR Spectrum of **6c**




```
Current Data Parameters
NAME      INN-MVD-151-NOESY
EXPNO                     7
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151203
Time                  17.45
INSTRUM               spect
PROBHD   5 mm SEI 1H/D-
PULPROG             noesyph
TD                     2048
SOLVENT               CDCl3
NS                       32
DS                       16
SWH                3633.721 Hz
FIDRES             1.774278 Hz
AQ                 0.2818048 sec
RG                      50.8
DW                  137.600 usec
DE                    6.50 usec
TE                   295.6 K
D0                 0.00012901 sec
D1                 1.90374398 sec
D8                 0.60000002 sec
IN0                0.00027520 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.75 usec
PL1                   -3.00 dB
PL1W              16.73965454 W
SFO1             400.1318090 MHz

F1 - Acquisition parameters
TD                       324
SFO1              400.1318 MHz
FIDRES           11.215176 Hz
SW                    9.081 ppm
FnMODE          States-TPPI

F2 - Processing parameters
SI                      2048
SF               400.1300095 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
PC                      1.00

F1 - Processing parameters
SI                       512
MC2             States-TPPI
SF               400.1300095 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
```

Figure S40. ^{1}H-^{1}HNOESY NMRSpectrum of **6c**

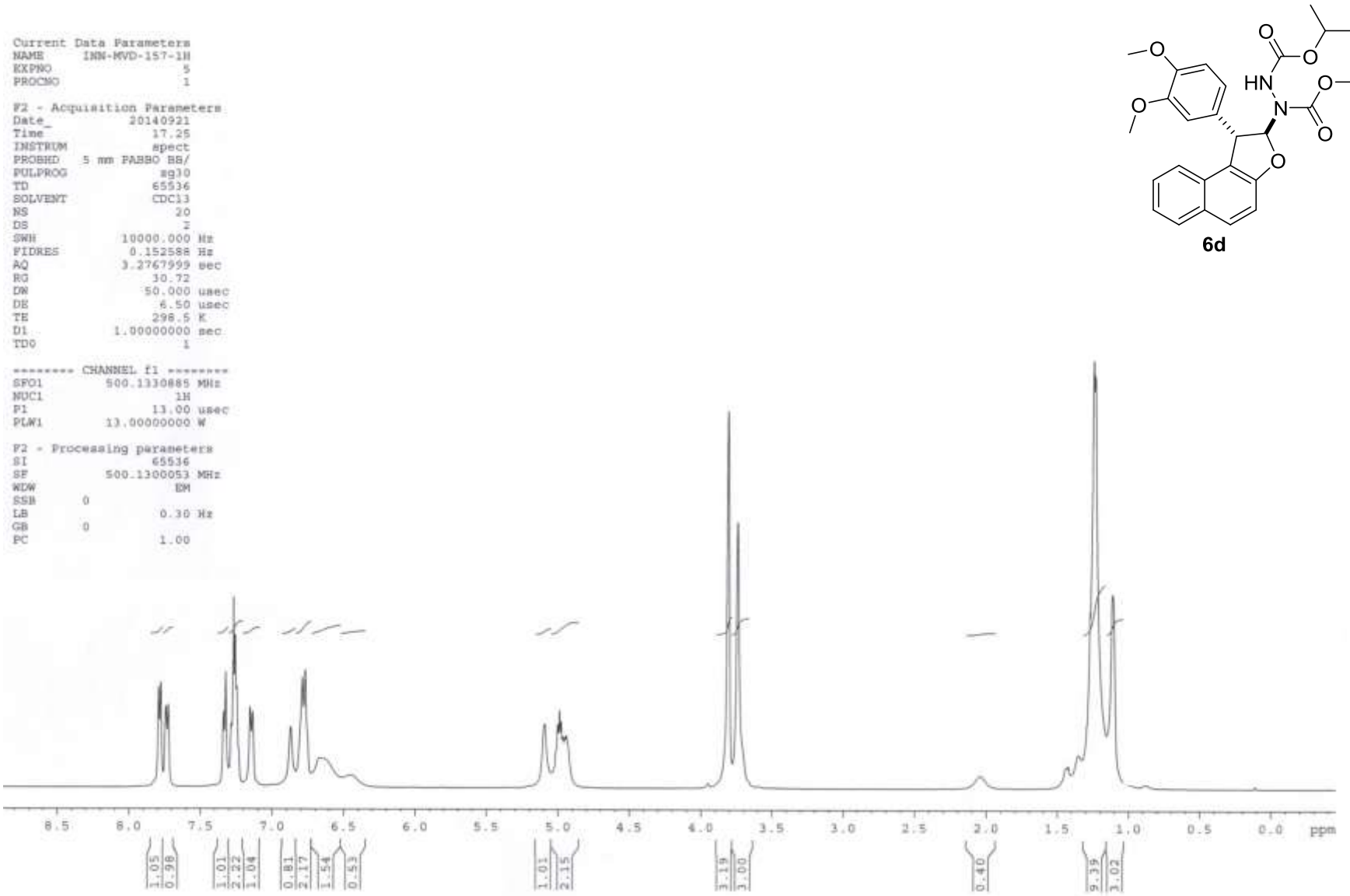


Figure S41. ^{1}H NMR Spectrum of **6d**

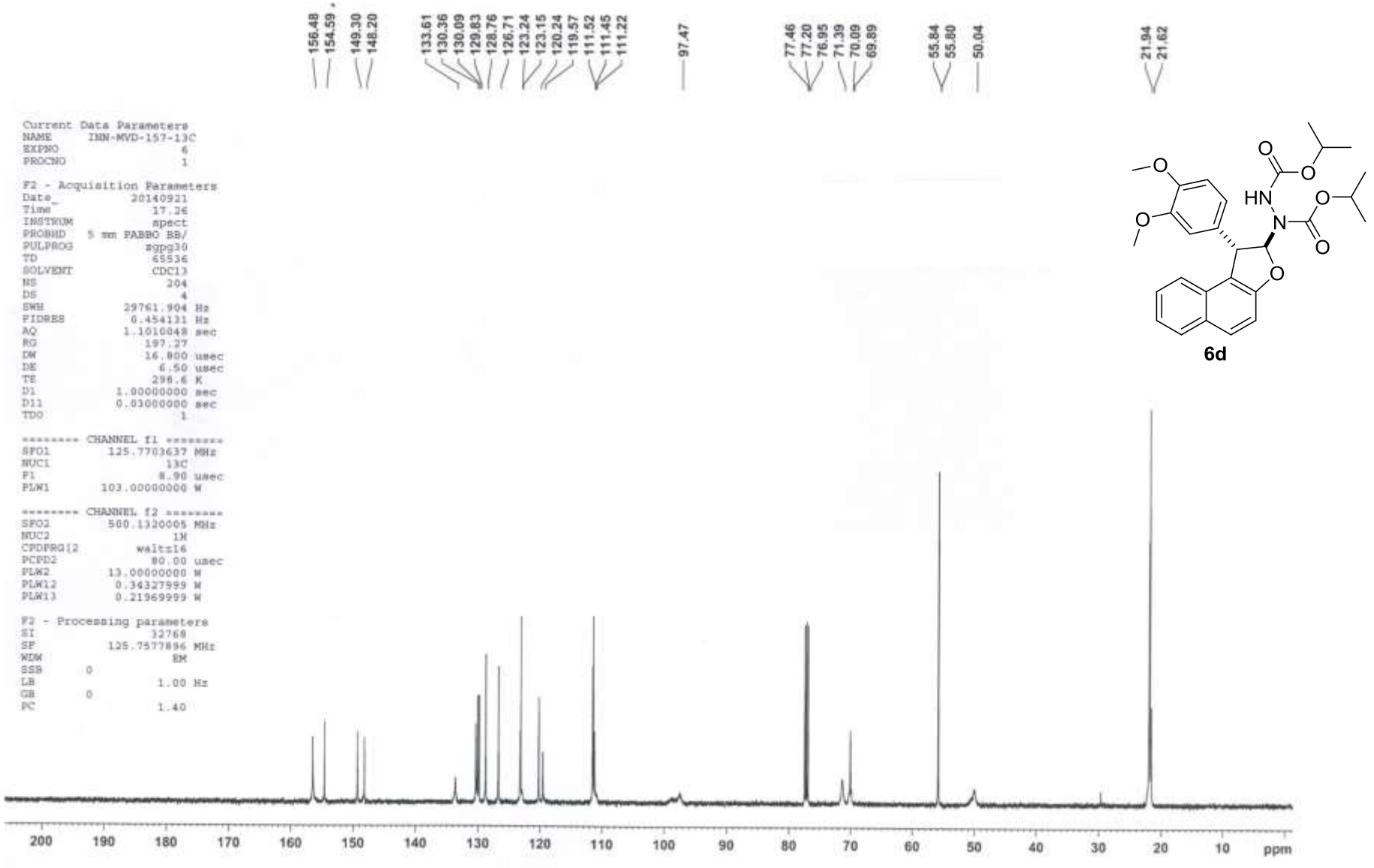
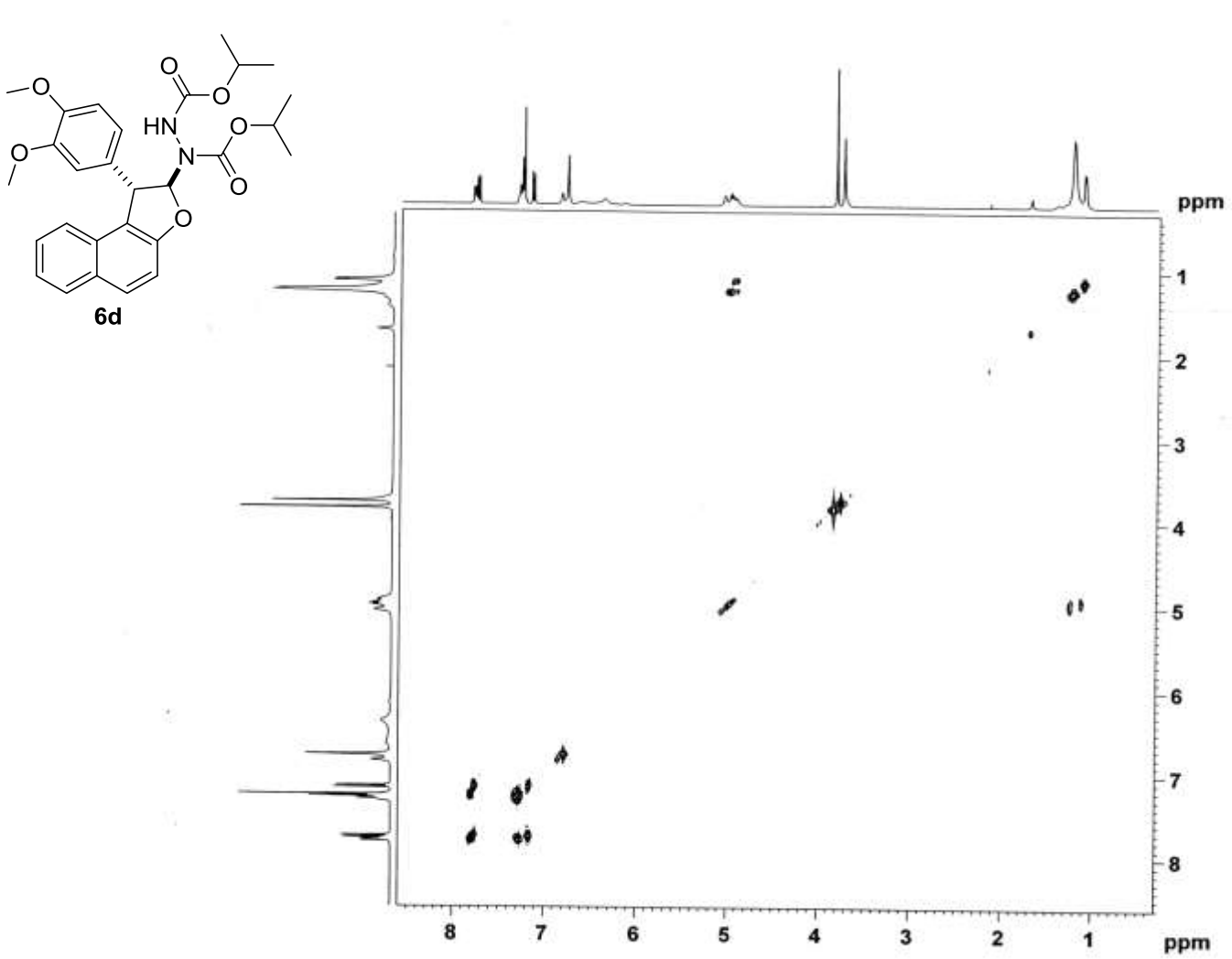


Figure S42. ^{13}C NMR Spectrum of **6d**



NAME	INN-MVD-157-cosy
EXPNO	2
PROCNO	1
Date_	20140101
Time_	16.45
INSTRUM	spect
PROBHD	5 mm SEI 1H/D-
PULPROG	cosygpqf
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	3318.584 Hz
FIDRES	1.620402 Hz
AQ	0.3086153 sec
RG	32
DW	150.667 usec
DE	6.50 usec
TE	295.5 K
D0	0.00000300 sec
D1	1.00000000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00030110 sec
======== CHANNEL f1 ========	
NUC1	1H
P0	6.75 usec
P1	6.75 usec
PL1	-3.00 dB
PL1W	16.73965454 W
SFO1	400.1317914 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ1	10.00 %
P16	1000.00 usec
ND0	1
TD	129
SFO1	400.1318 MHz
FIDRES	25.744913 Hz
SW	8.300 ppm
FnMODE	QF
SI	2048
SF	400.1300098 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	400.1300098 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

Figure S43. ^{1}H-^{1}H COSY NMR Spectrum of **6d**

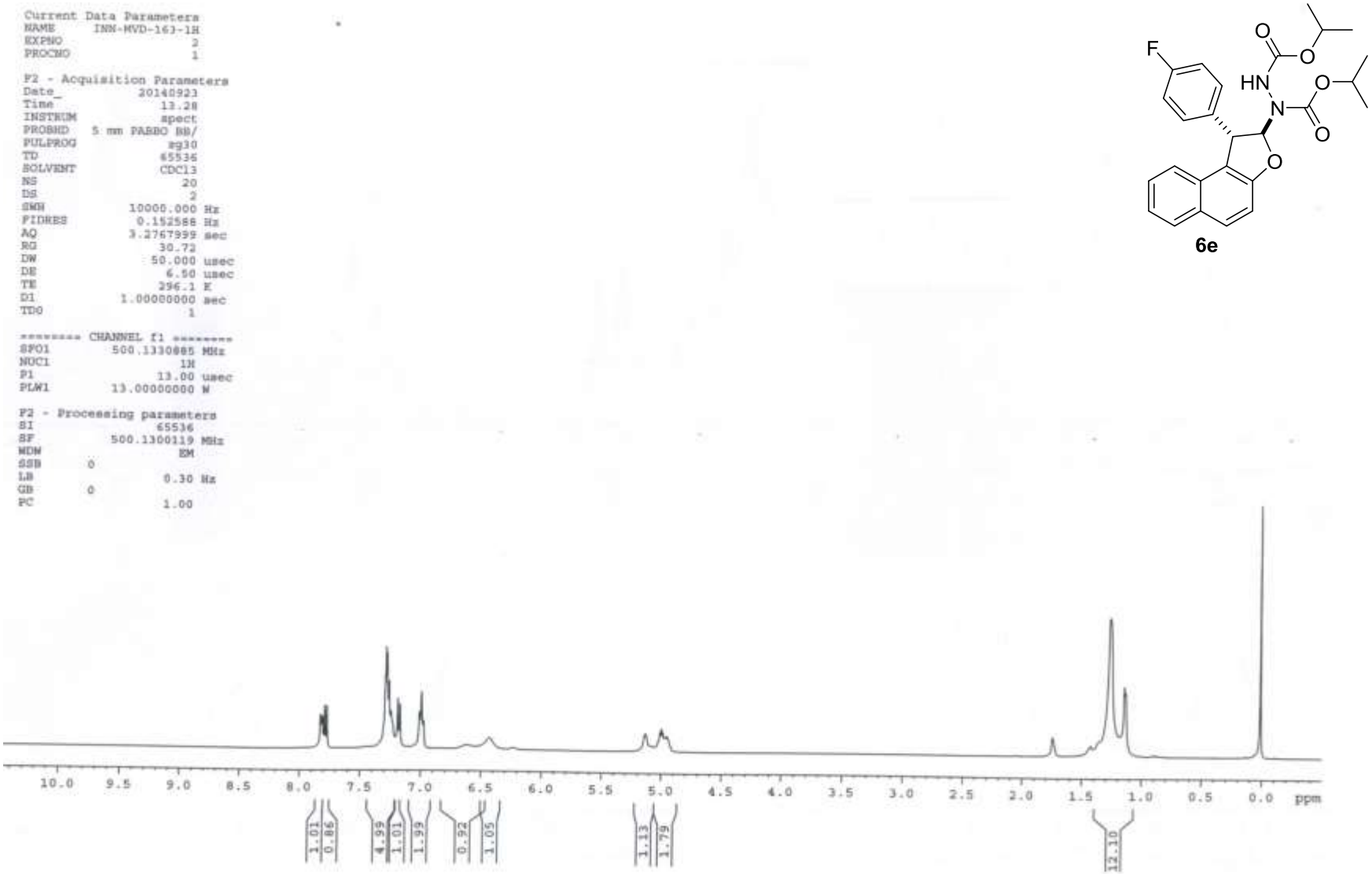


Figure S44. ^{1}H NMR Spectrum of **6e**

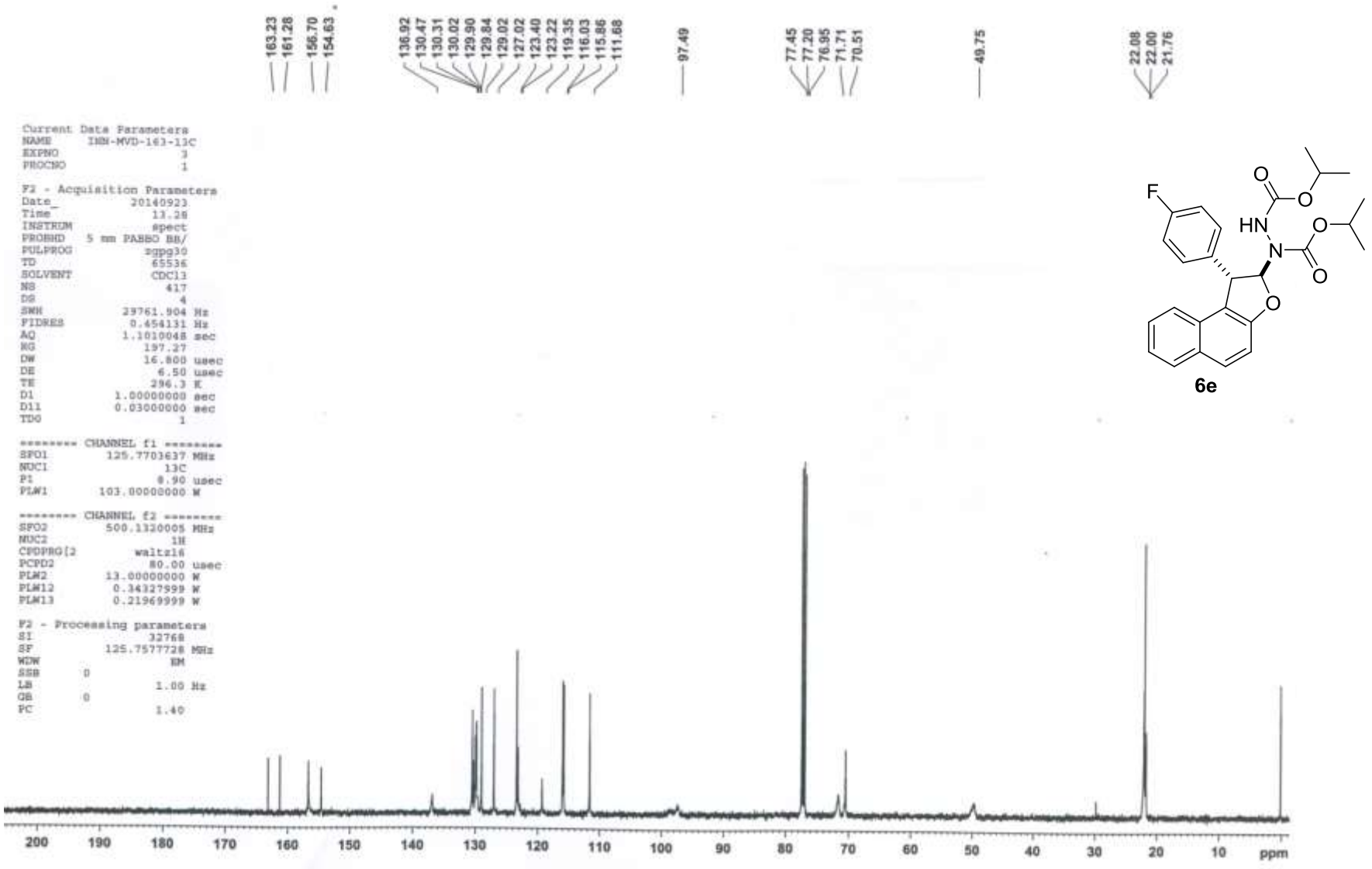


Figure S45. ^{13}C NMR Spectrum of **6e**

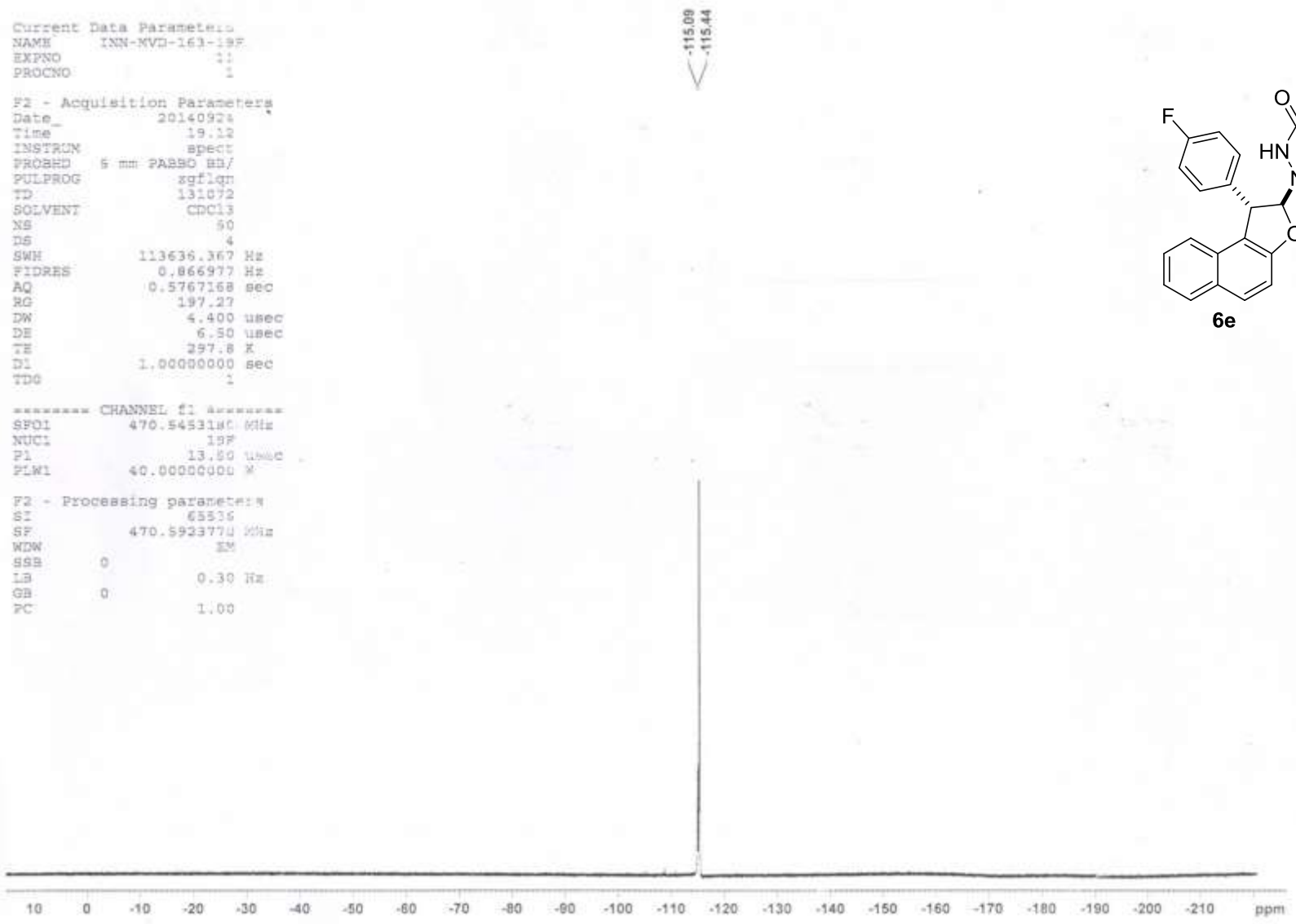


Figure S46. ^{19}F NMR Spectrum of **6e**

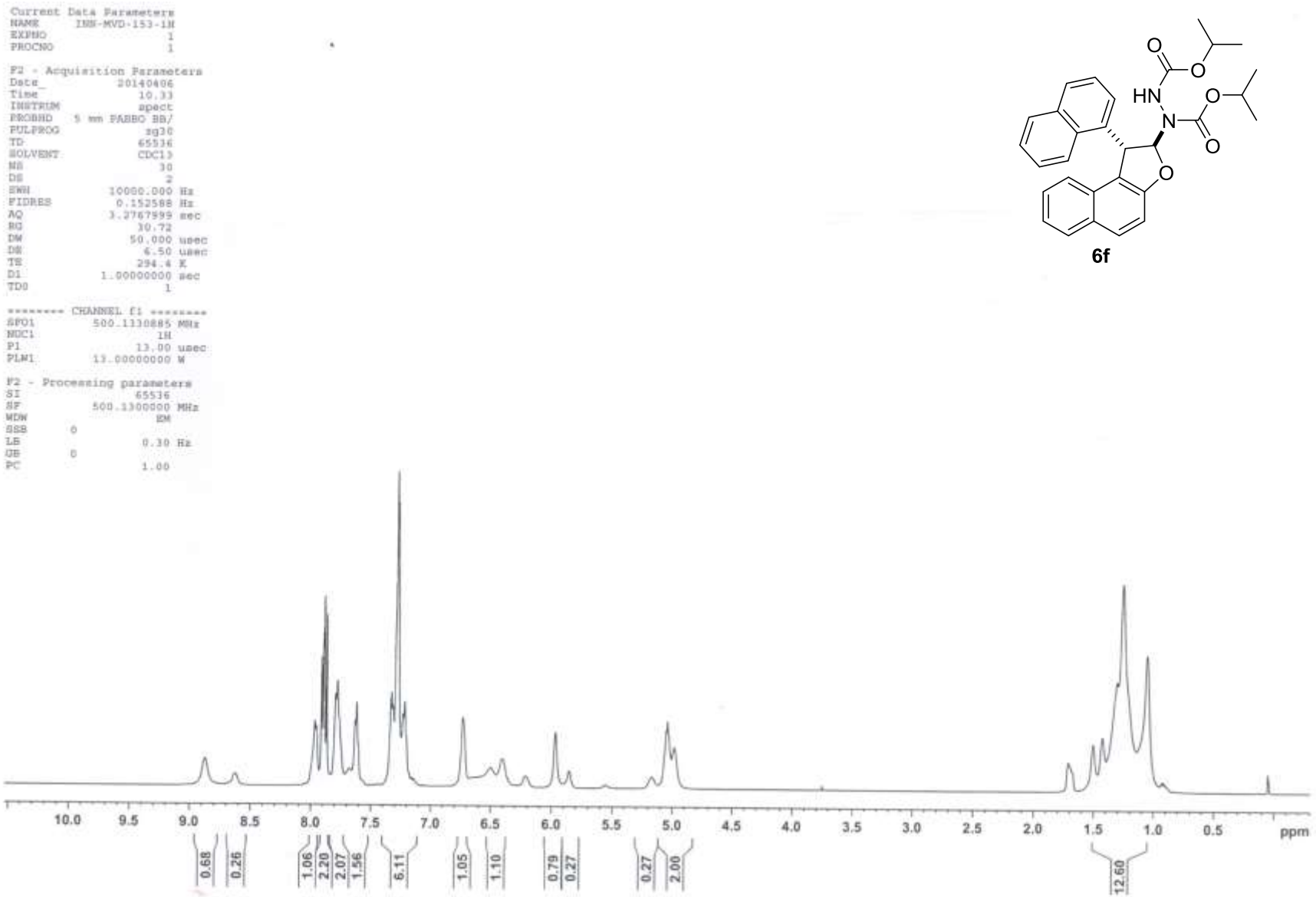


Figure S47. ^{1}H NMR Spectrum of **6f**

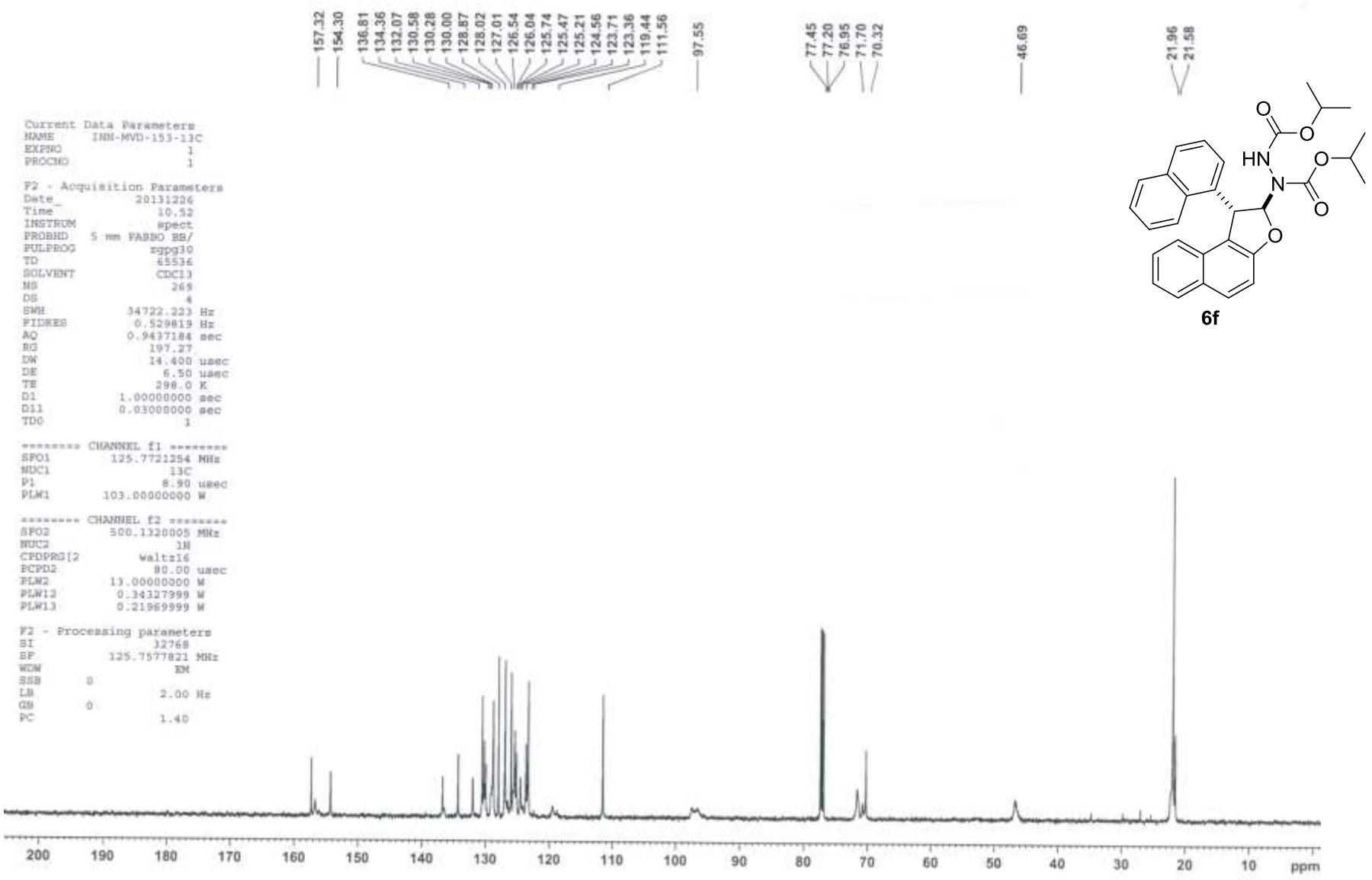


Figure S48. ^{13}C NMR Spectrum of **6f**

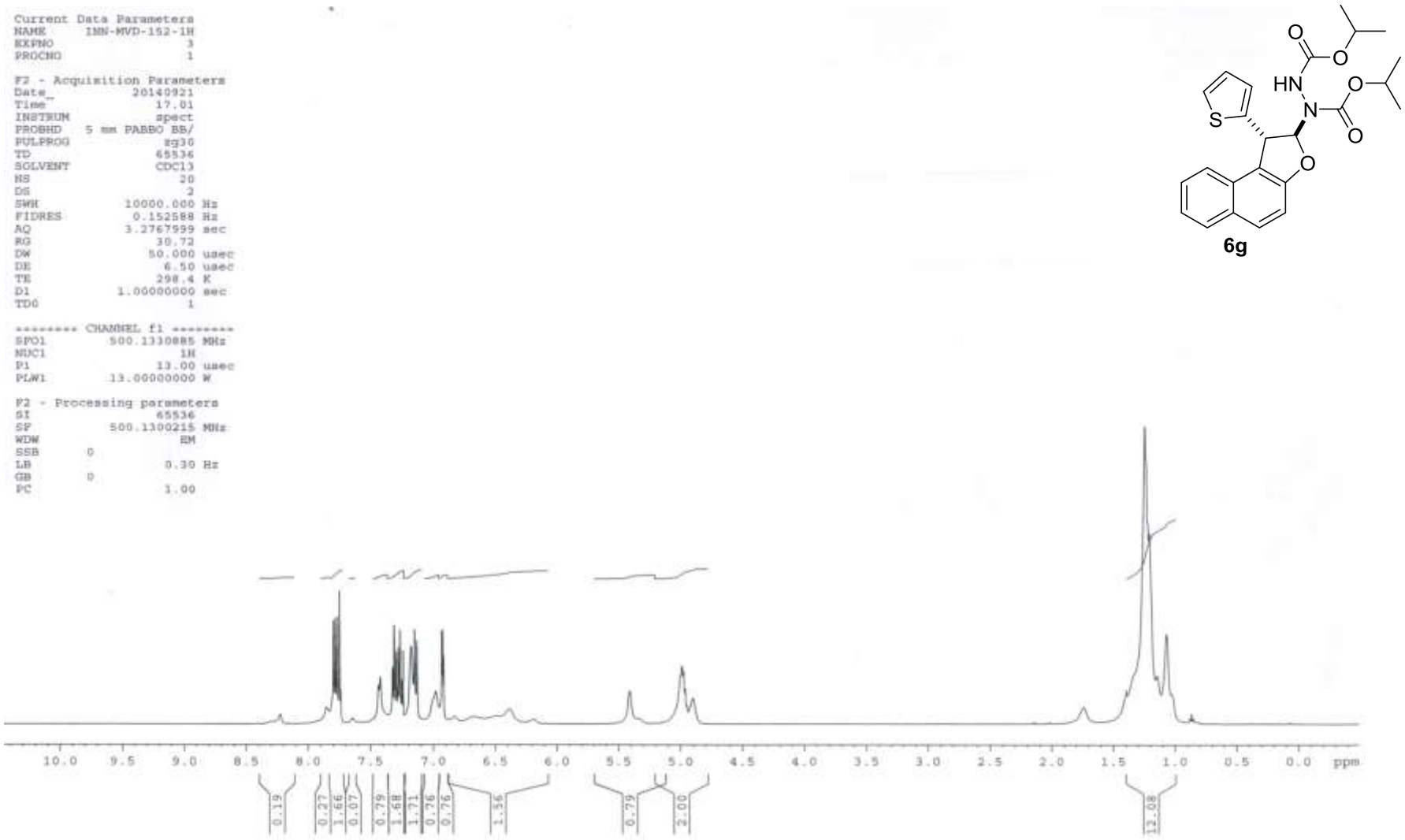


Figure S49. ^{1}H NMR Spectrum of **6g**

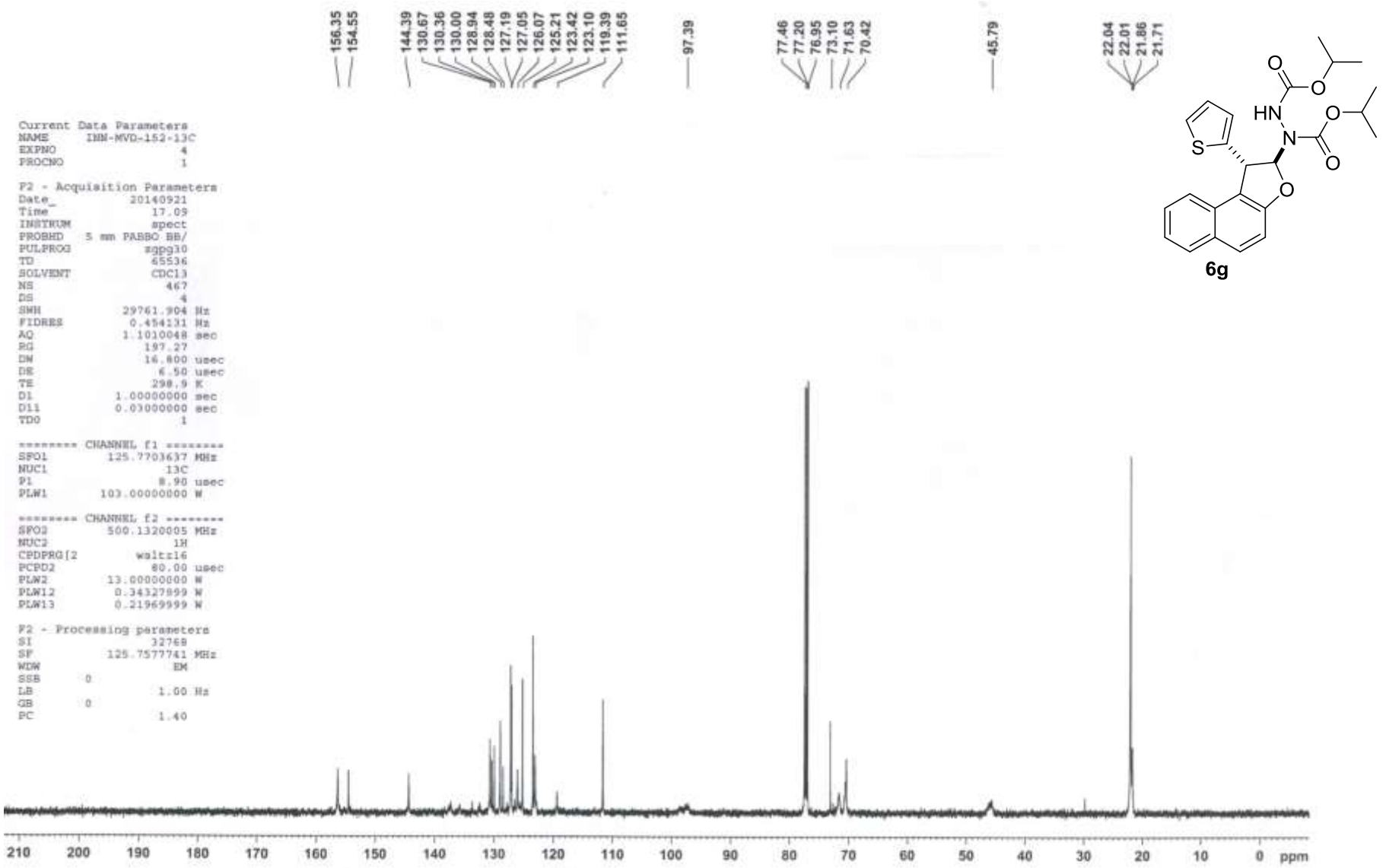


Figure S50. ^{13}C NMR Spectrum of **6g**

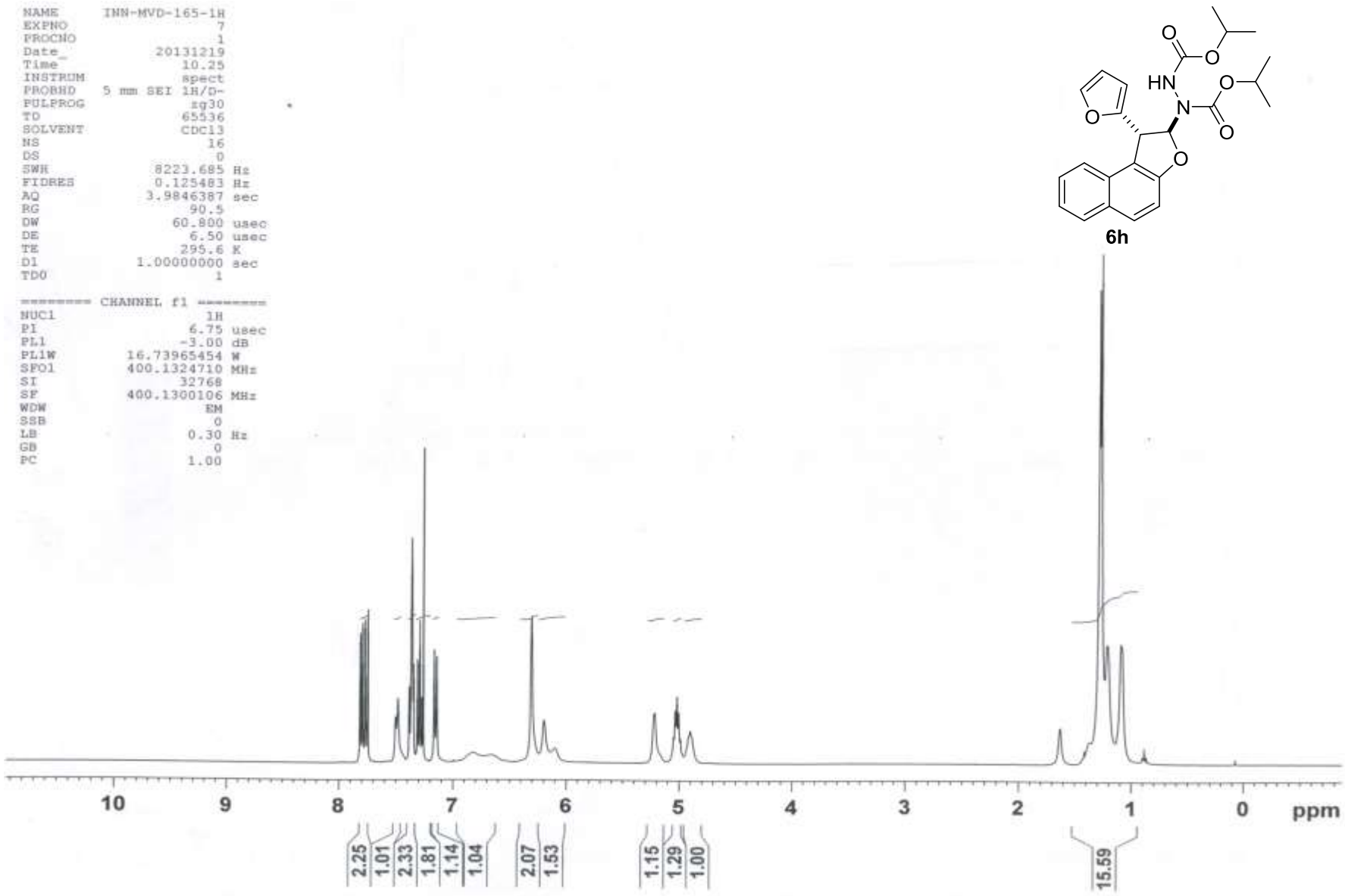


Figure S51. ^{1}H NMR Spectrum of **6h**

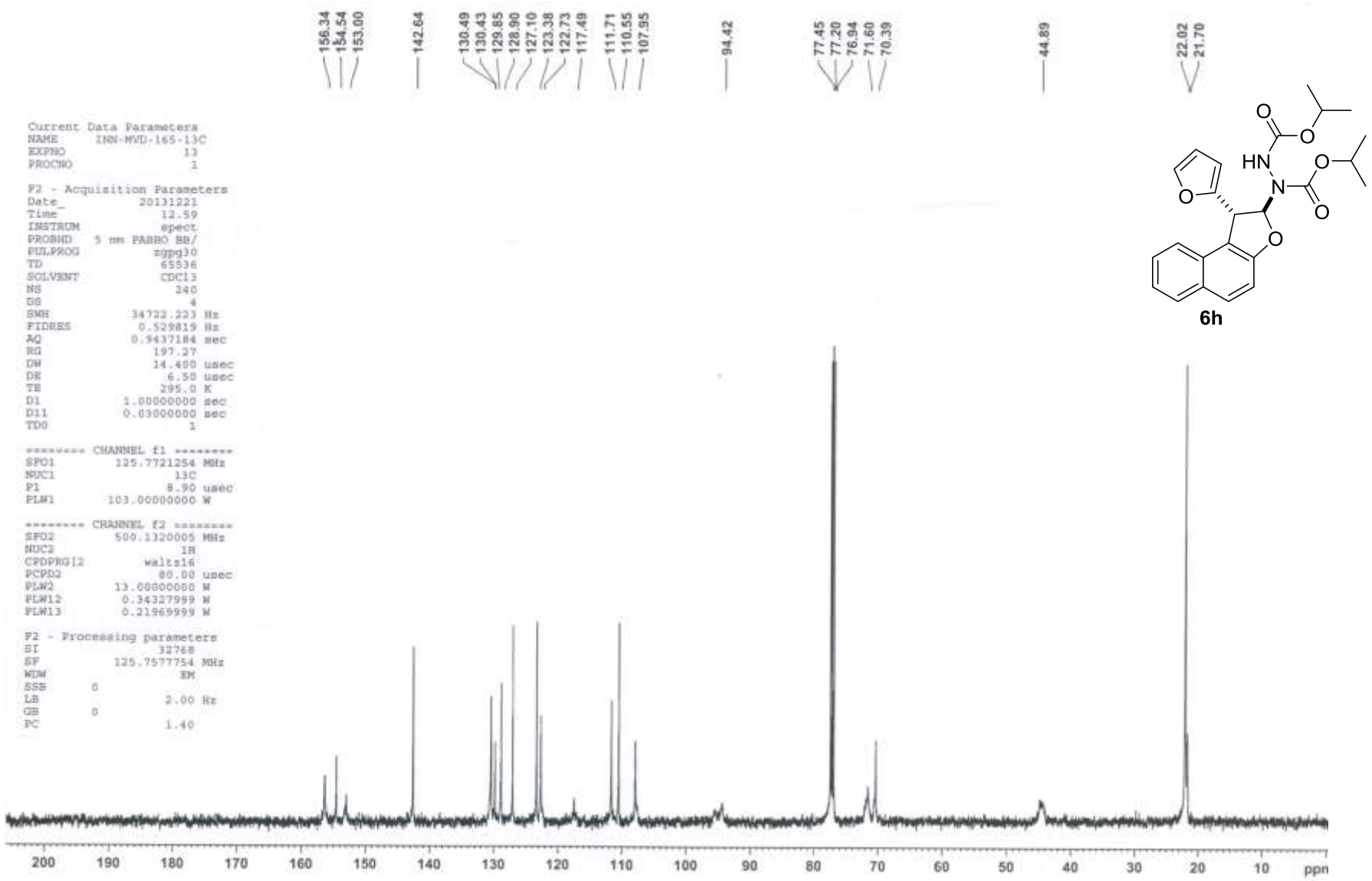
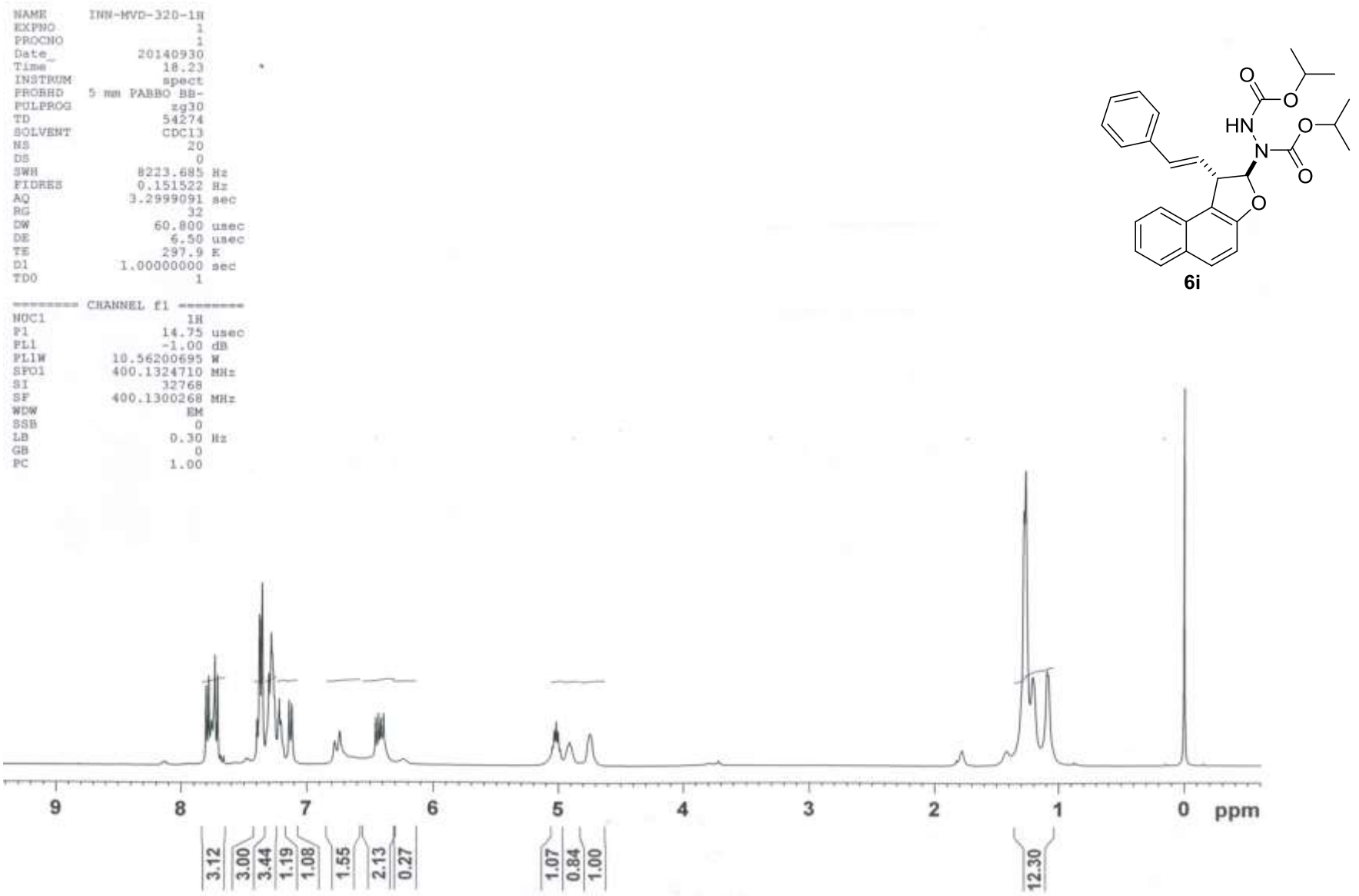


Figure S52. ^{13}C NMR Spectrum of **6h**



6i

Figure S53. ^{1}H NMR Spectrum of **6i**

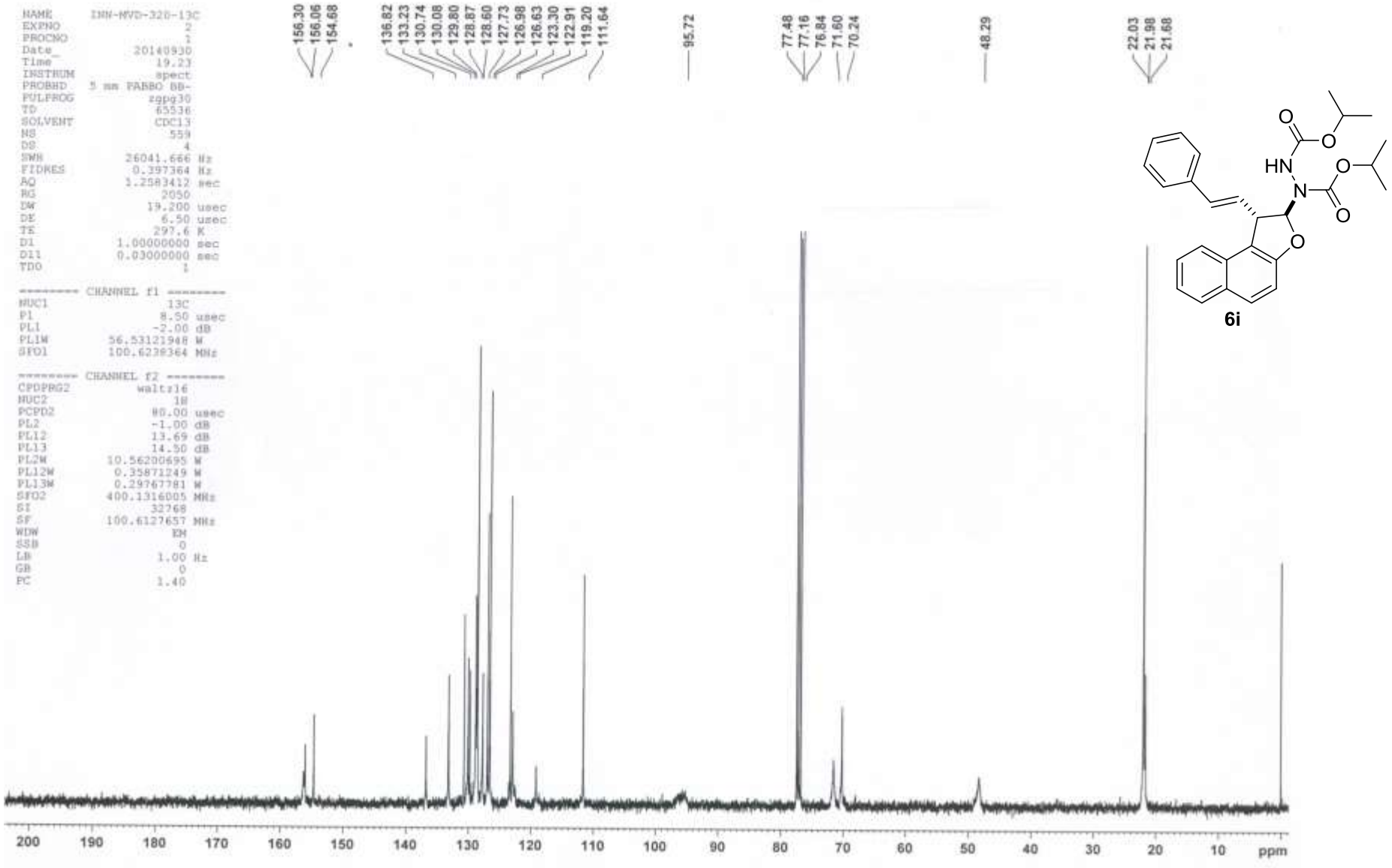
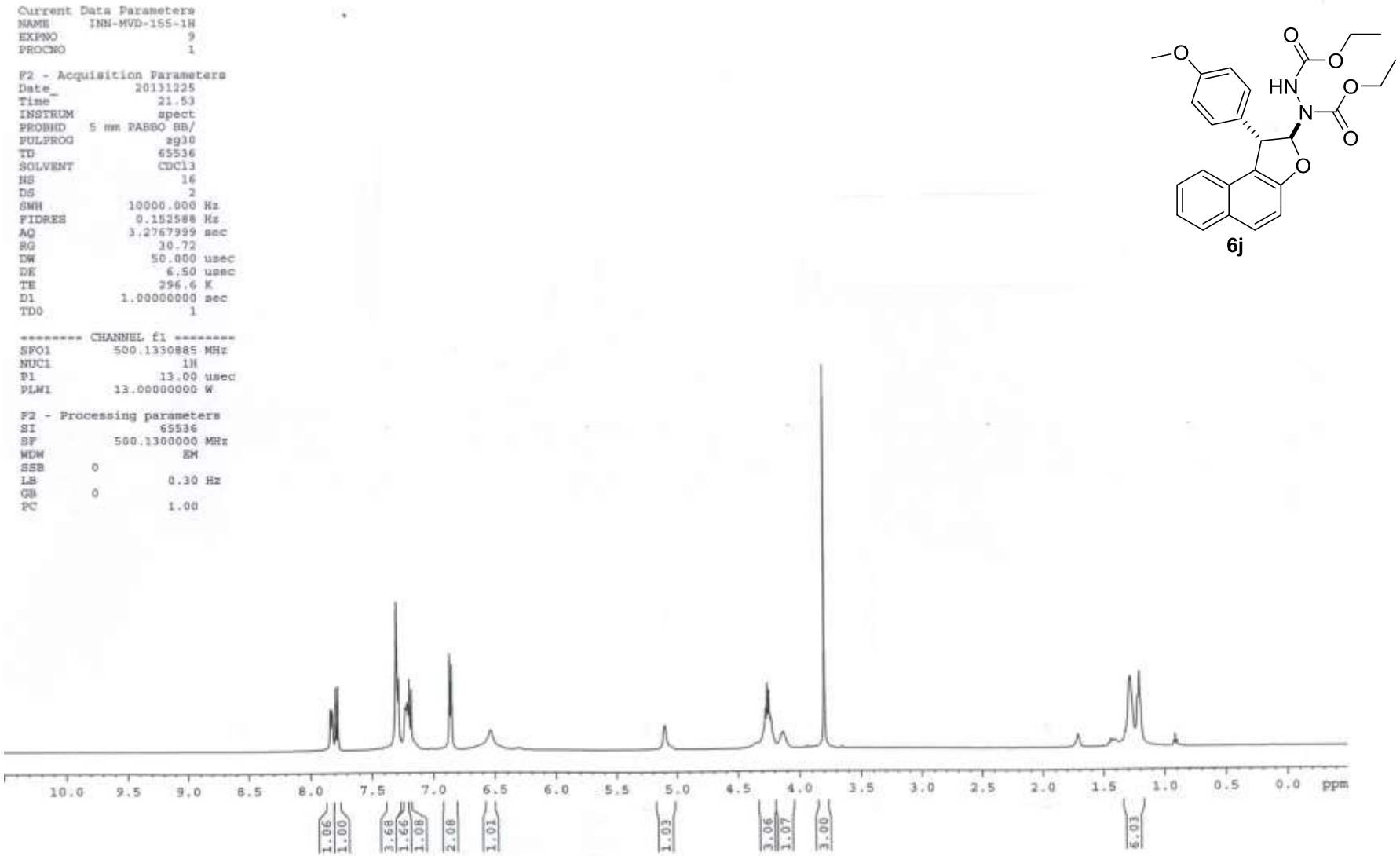


Figure S54. ^{13}C NMR Spectrum of **6i**



6j

Figure S55. ^{1}H NMR Spectrum of **6j**

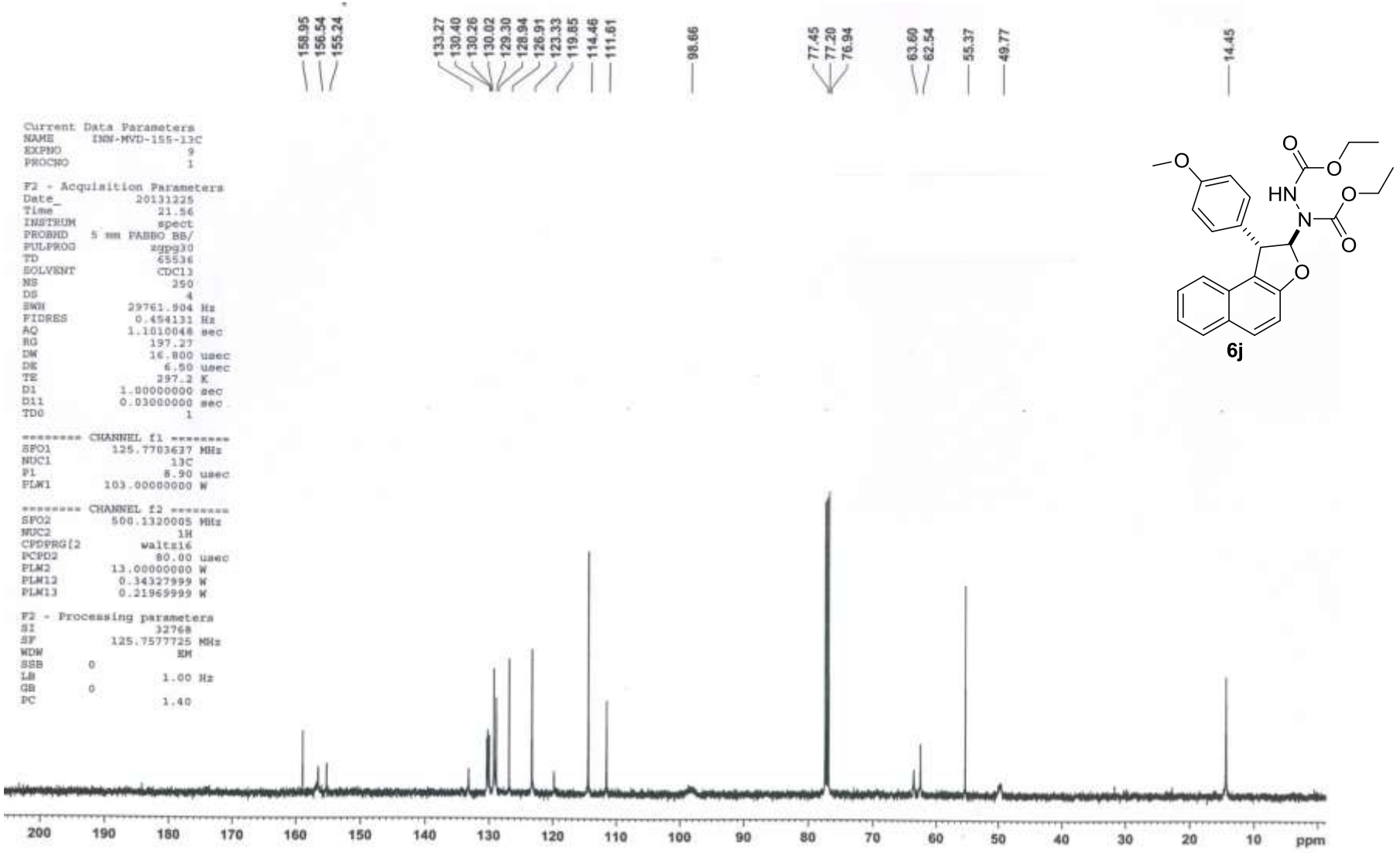


Figure S56. ^{13}C NMR Spectrum of **6j**

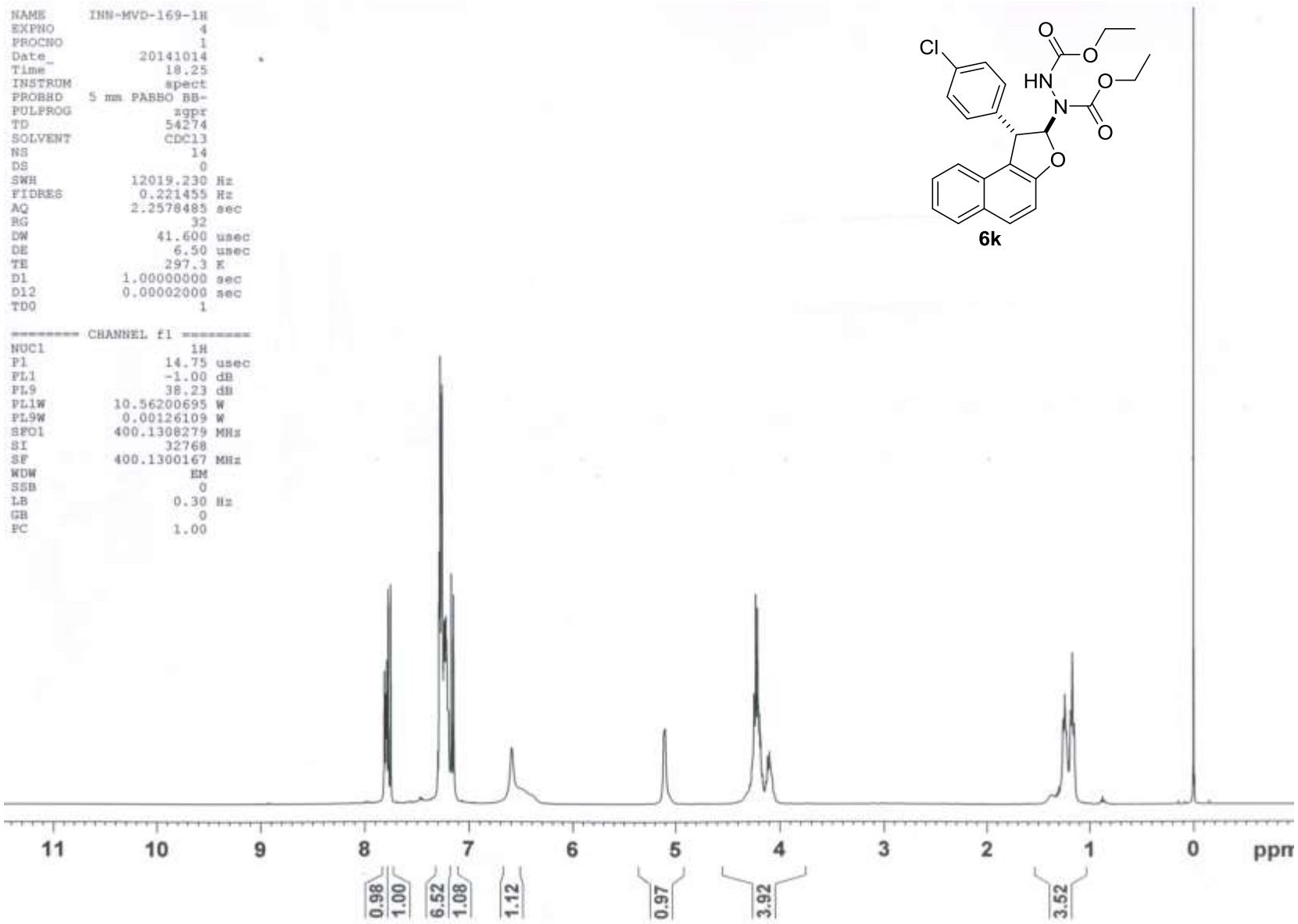


Figure S57. ^{1}H NMR Spectrum of **6k**

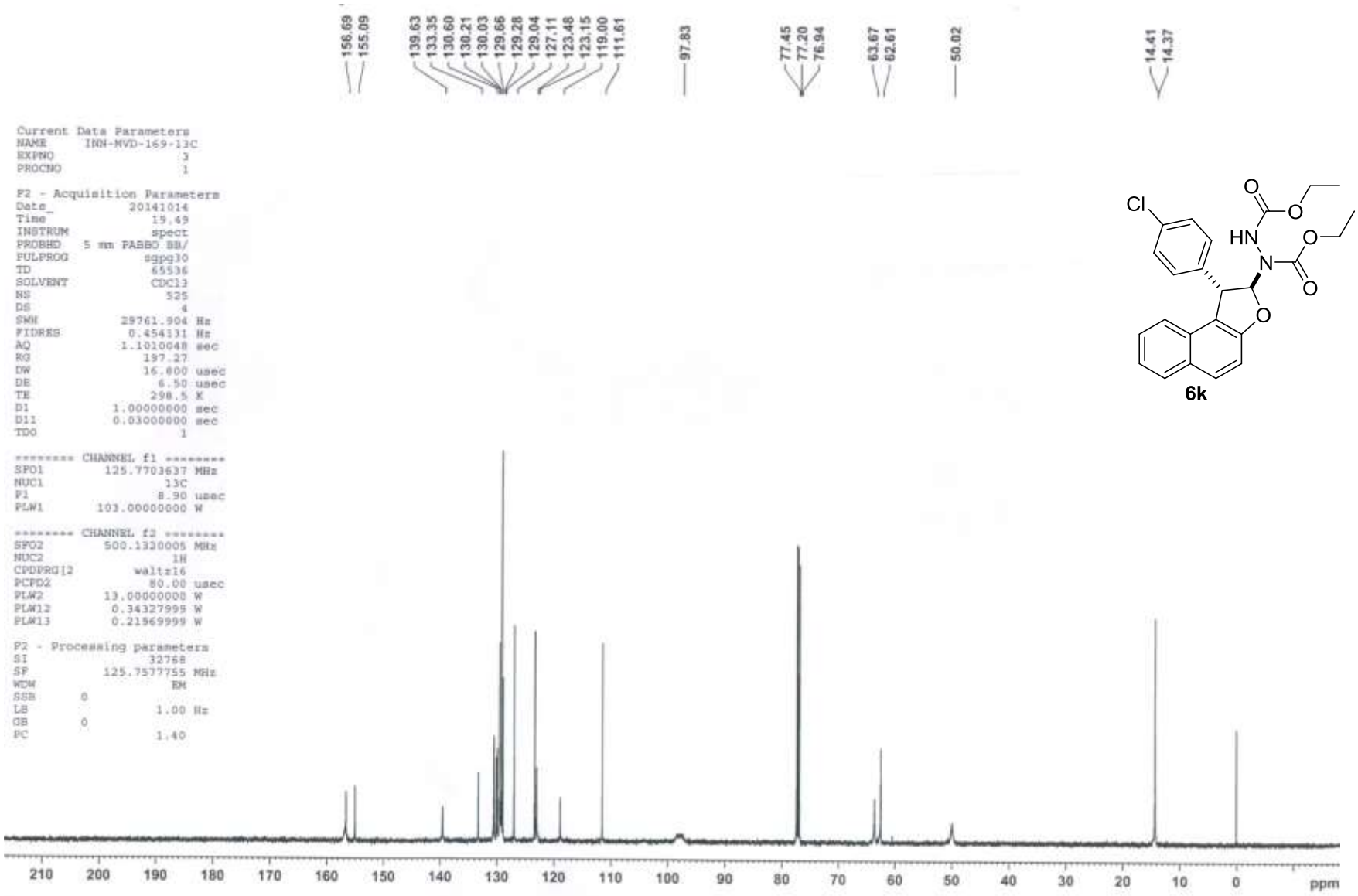


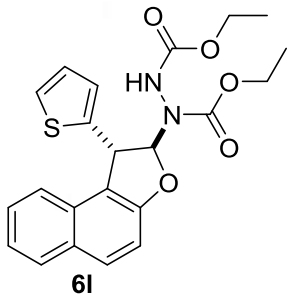
Figure S58. ^{13}C NMR Spectrum of **6k**

```
Current Data Parameters
NAME      INN-MVD-162-1H
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131210
Time               19.33
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            3.2767999 sec
RG                30.72
DW               50.000 usec
DE                6.50 usec
TE                297.6 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
```



6l

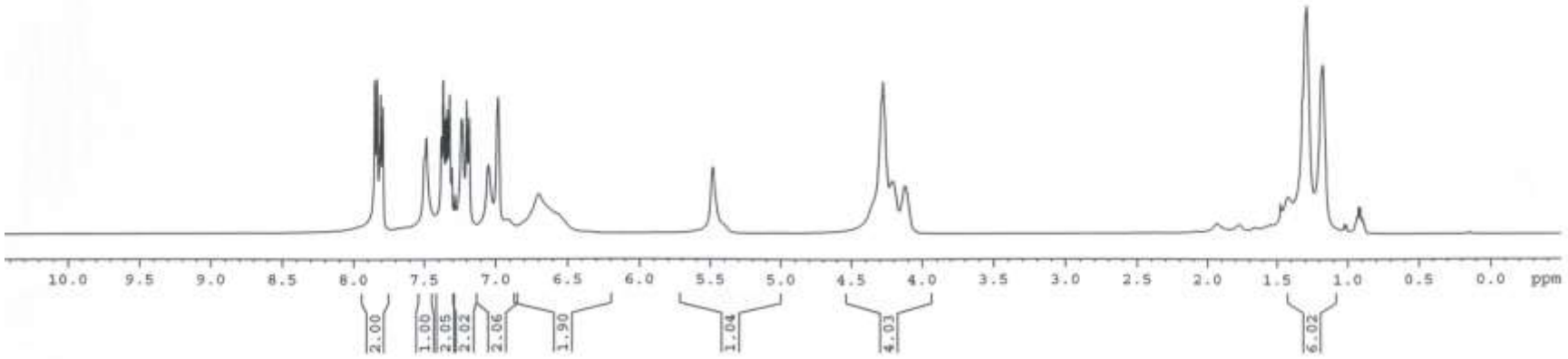


Figure S59. ^{1}H NMR Spectrum of **6l**

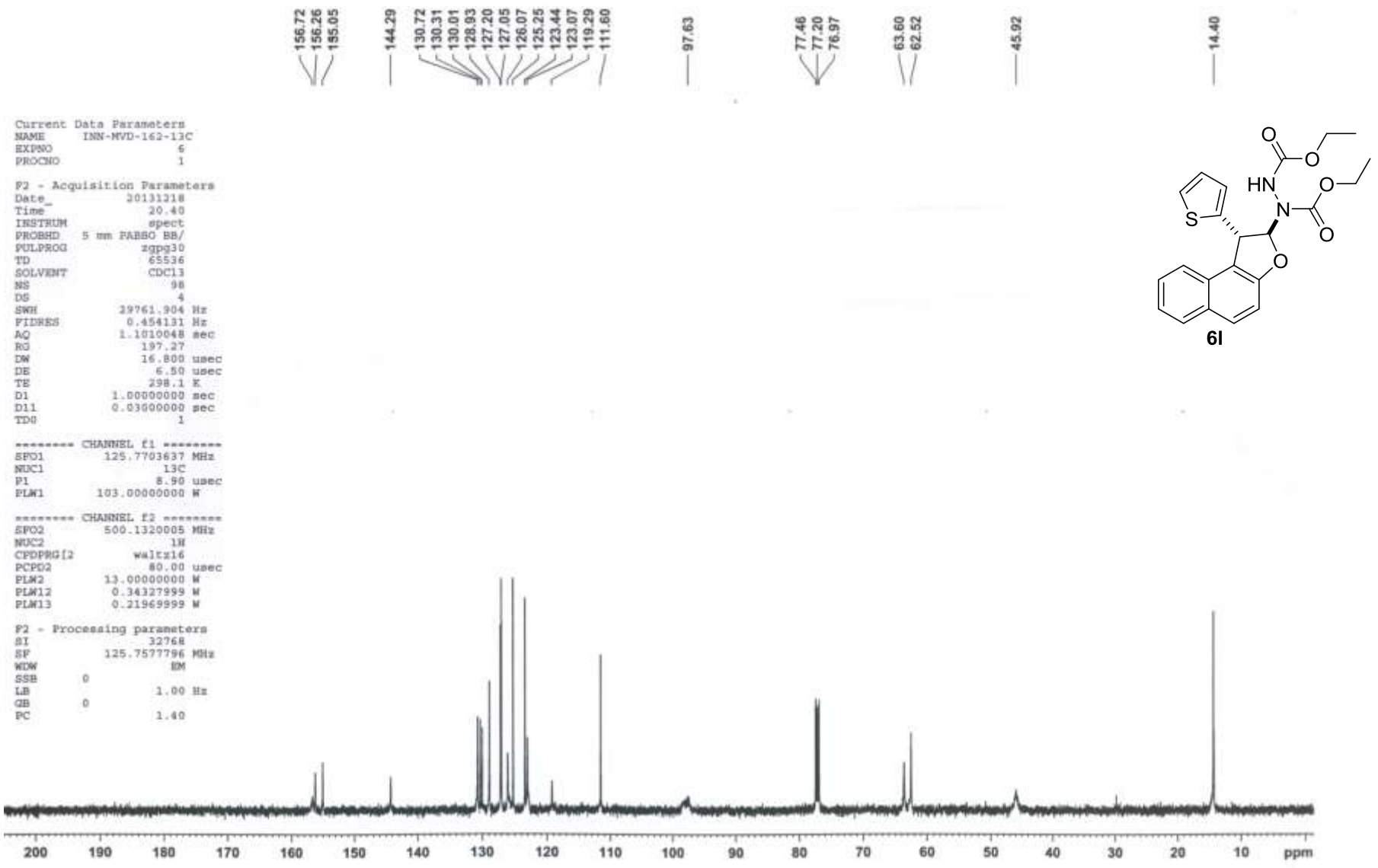


Figure S60. ^{13}C NMR Spectrum of **6l**

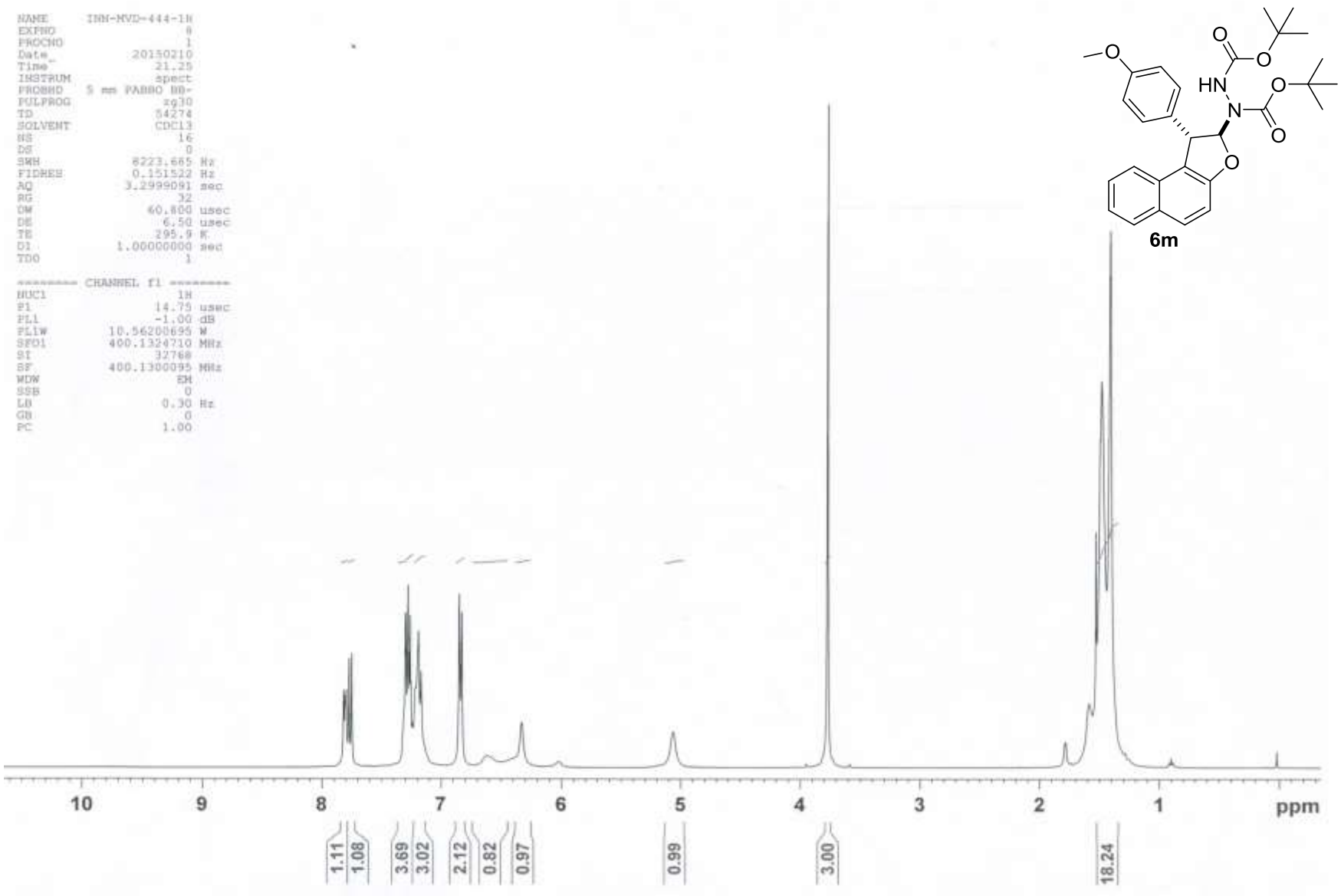


Figure S61. ^{1}H NMR Spectrum of **6m**

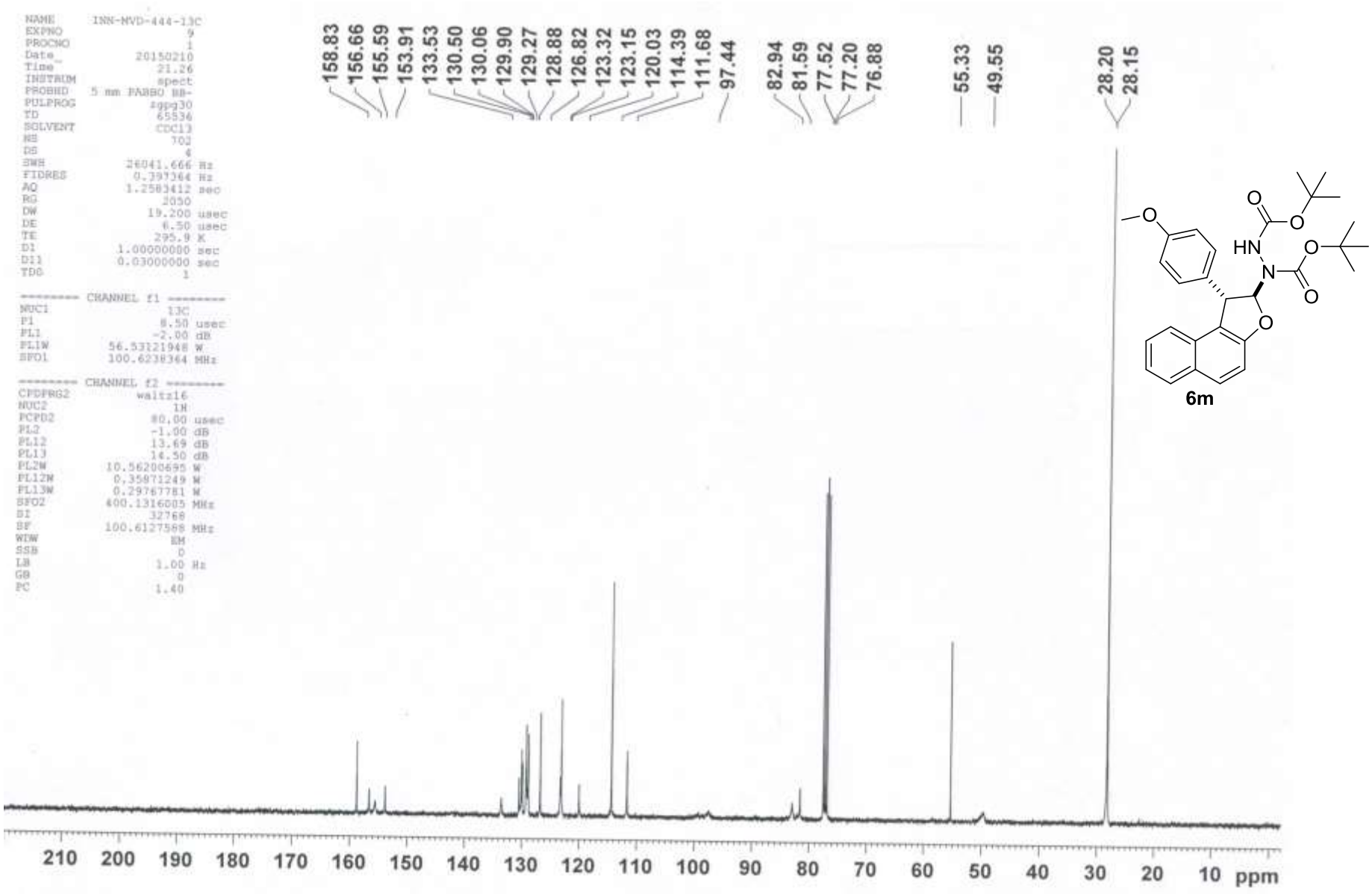


Figure S62. ^{13}C NMR Spectrum of **6m**

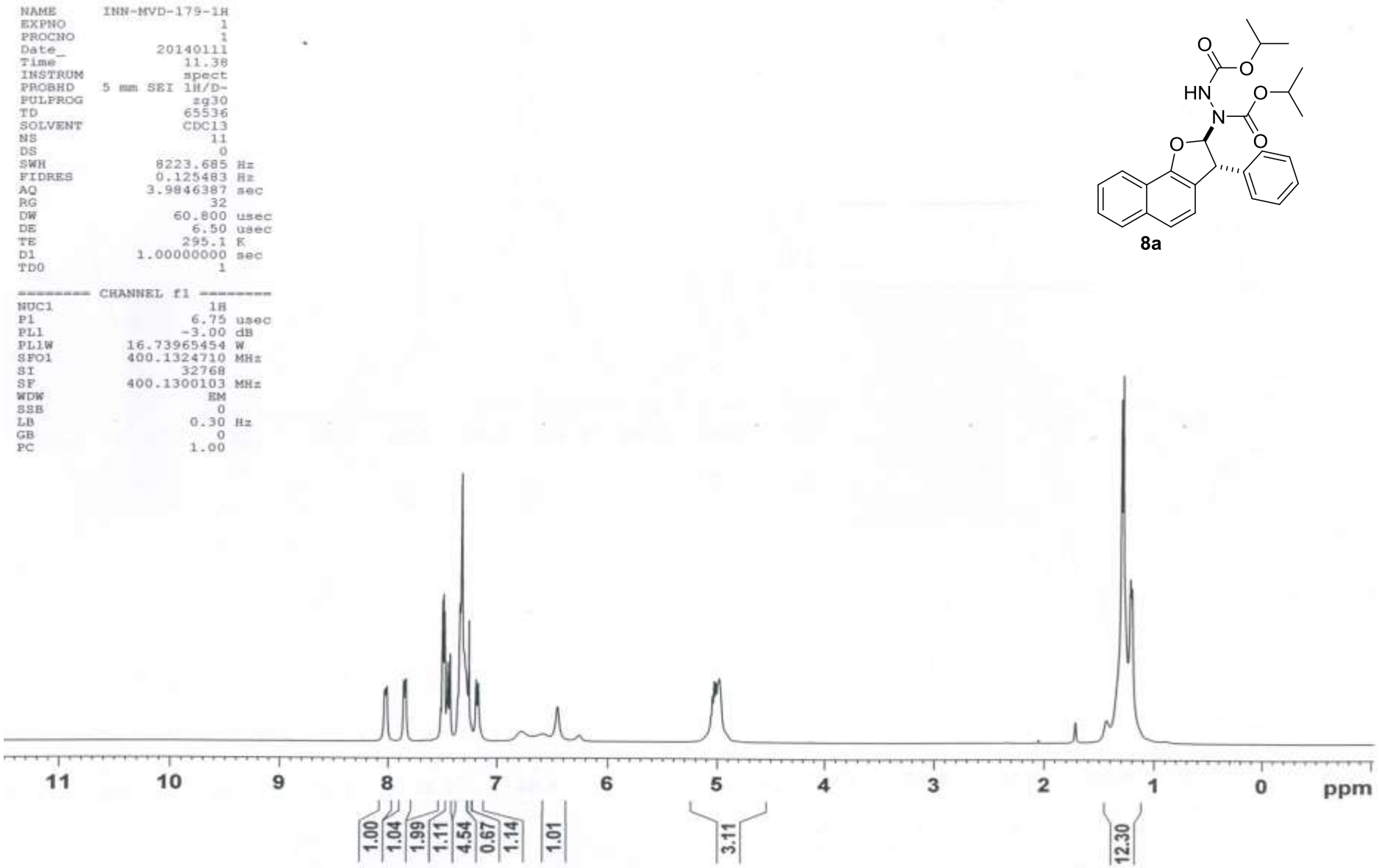


Figure S63. ^{1}H NMR Spectrum of **8a**

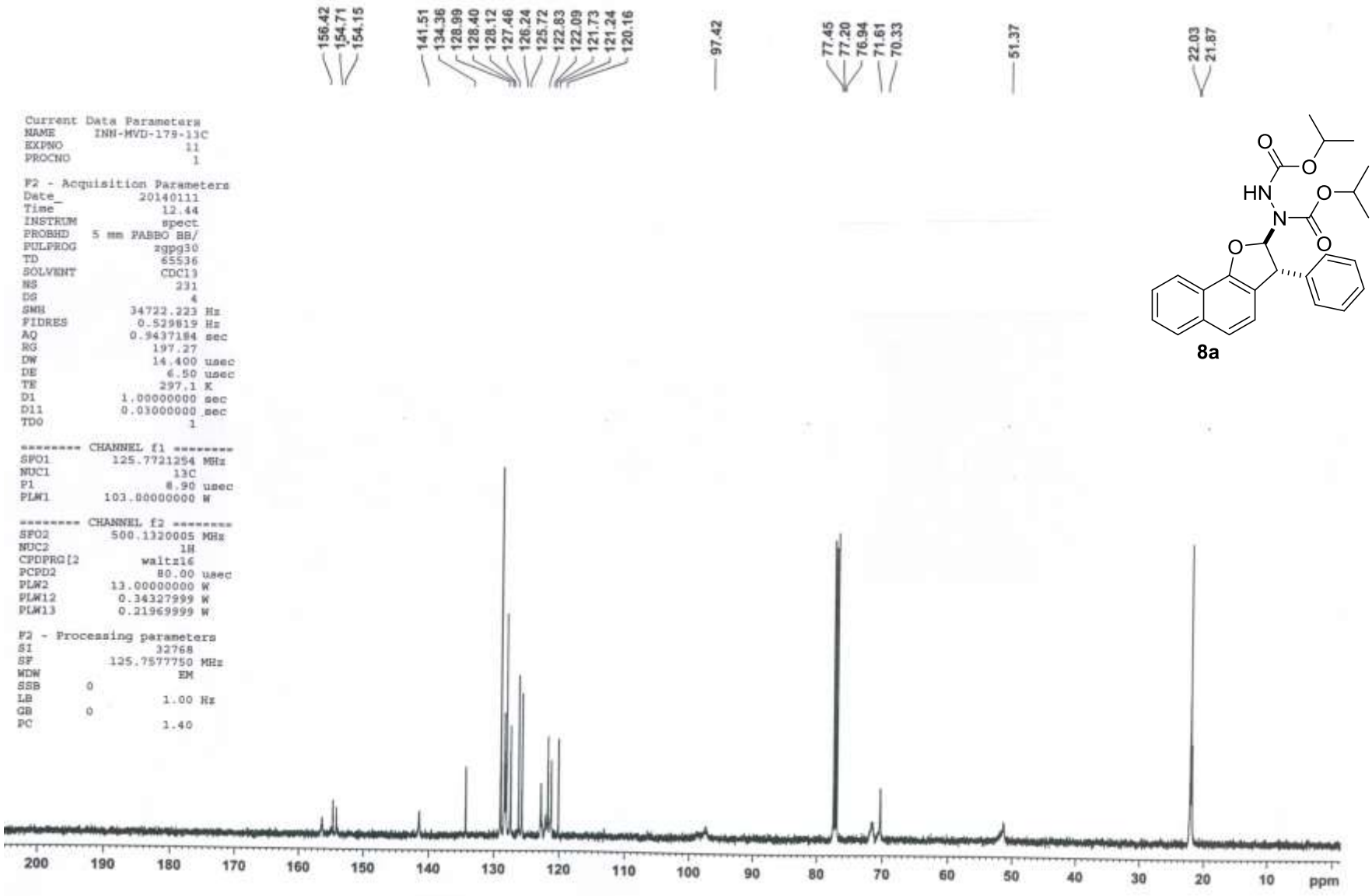


Figure S64. ^{13}C NMR Spectrum of **8a**

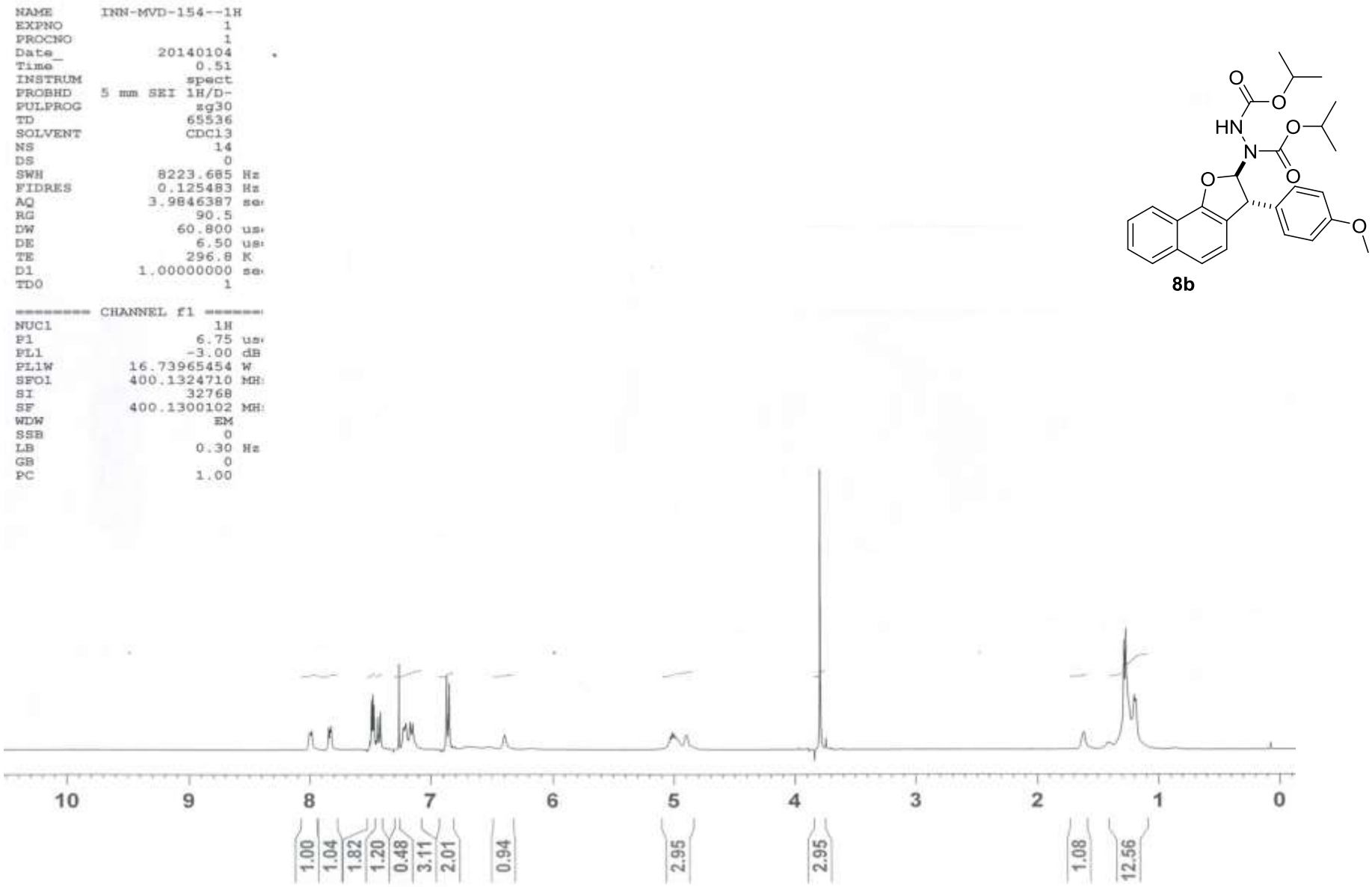


Figure S65. ^{1}H NMR Spectrum of **8b**

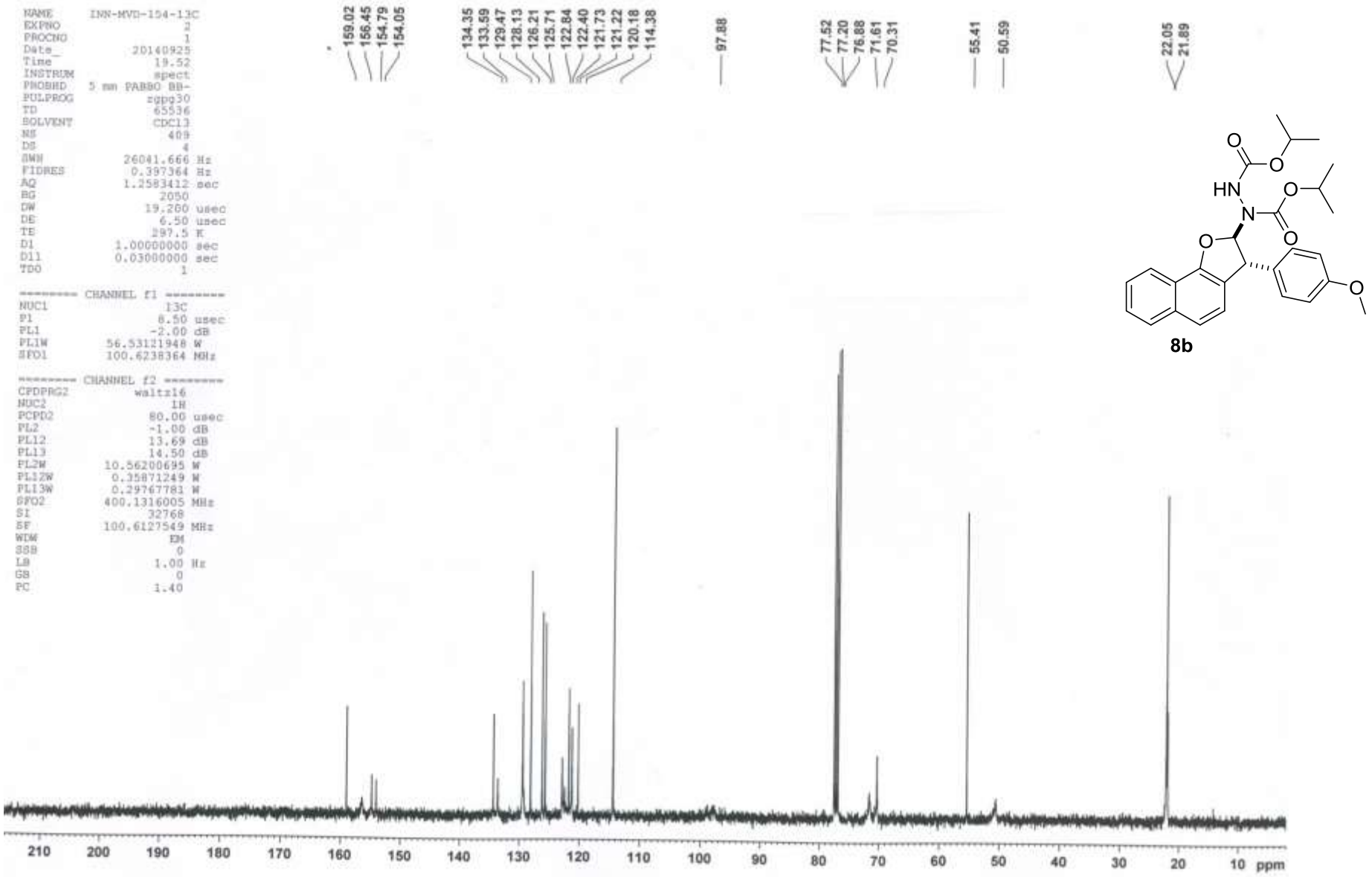


Figure S66. ^{13}C NMR Spectrum of **8b**

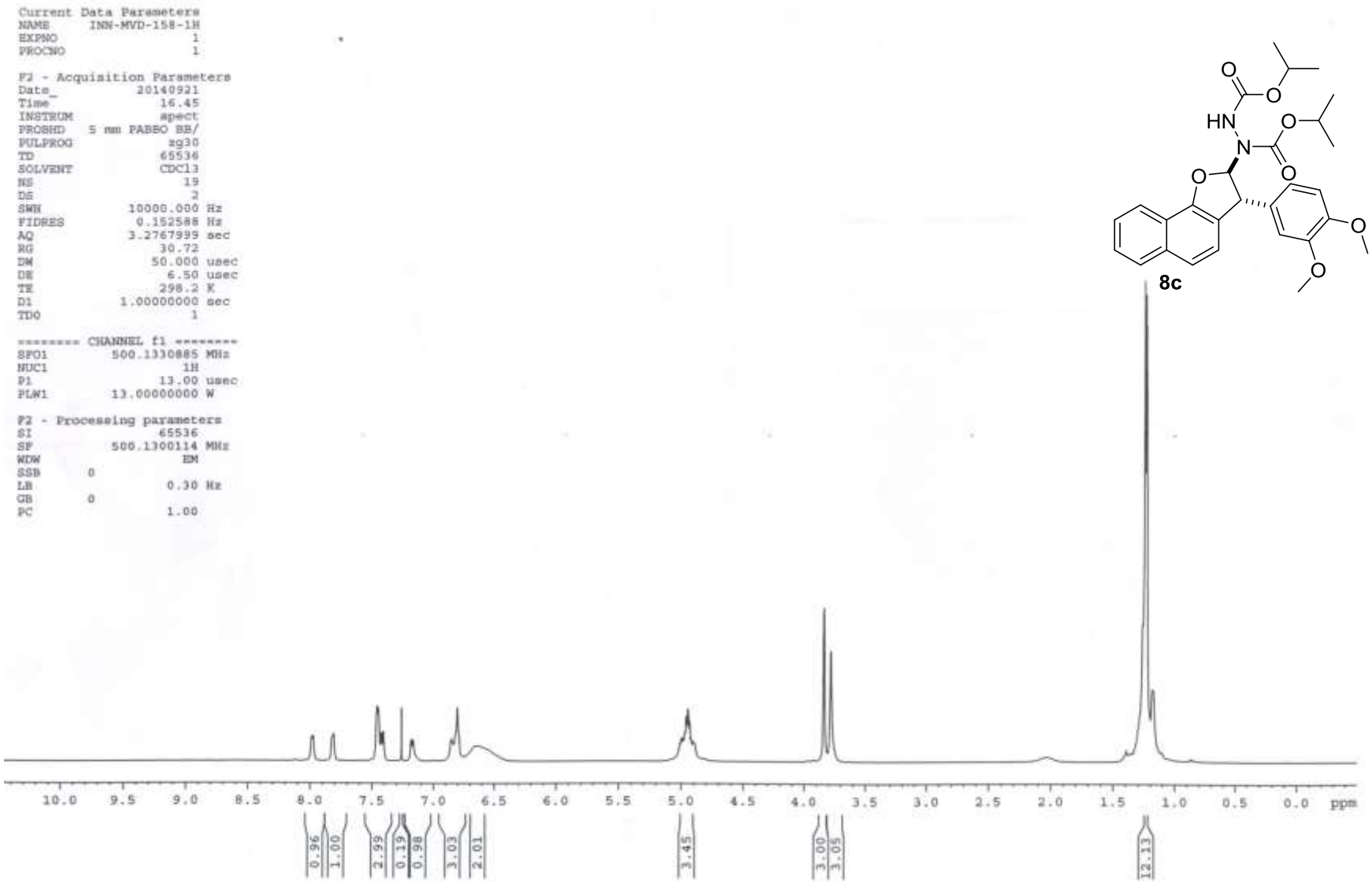


Figure S67. ^{1}H NMR Spectrum of **8c**

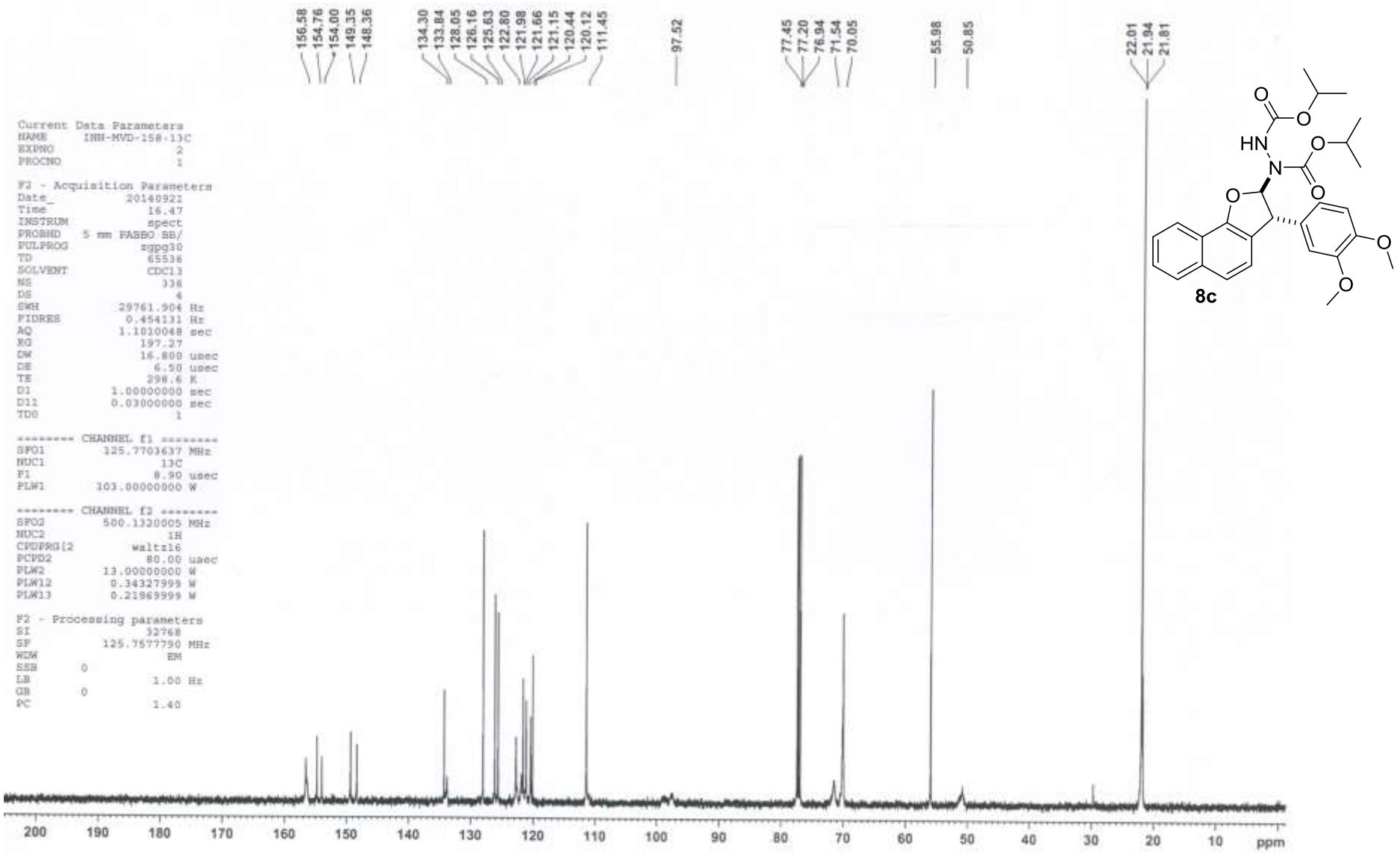


Figure S68. ^{13}C NMR Spectrum of **8c**

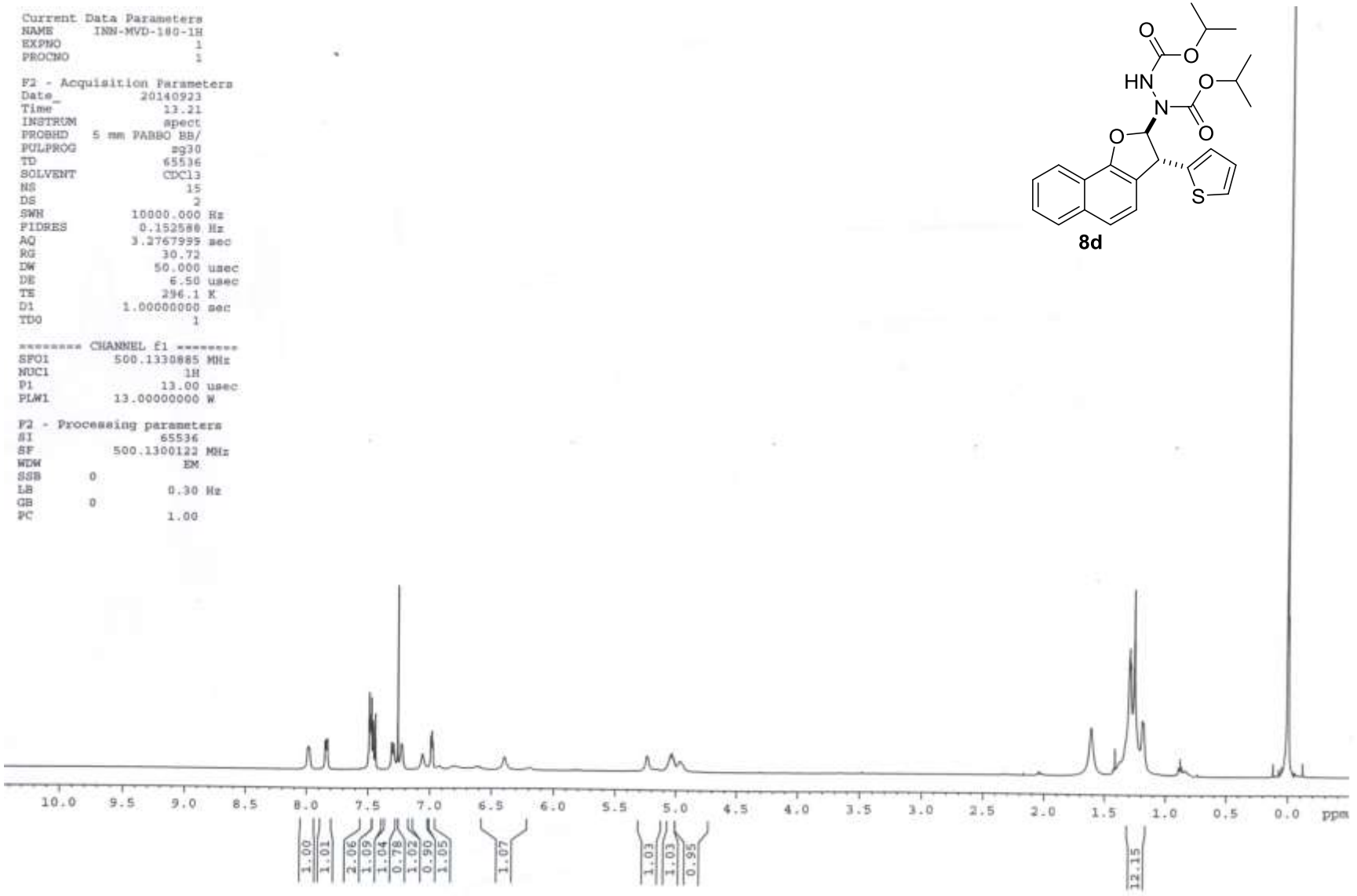


Figure S69. ^{1}H NMR Spectrum of **8d**

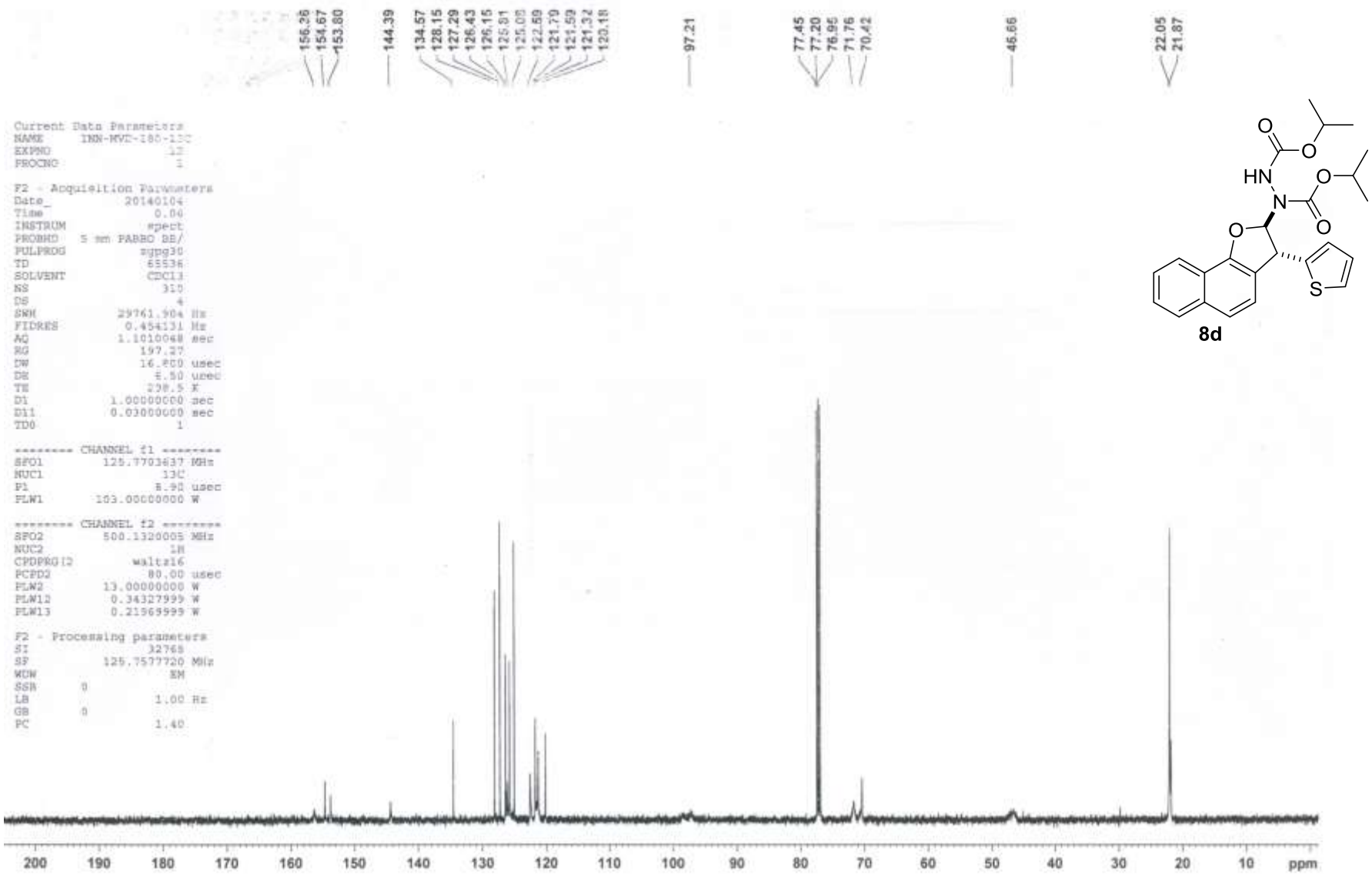


Figure S70. ^{13}C NMR Spectrum of **8d**

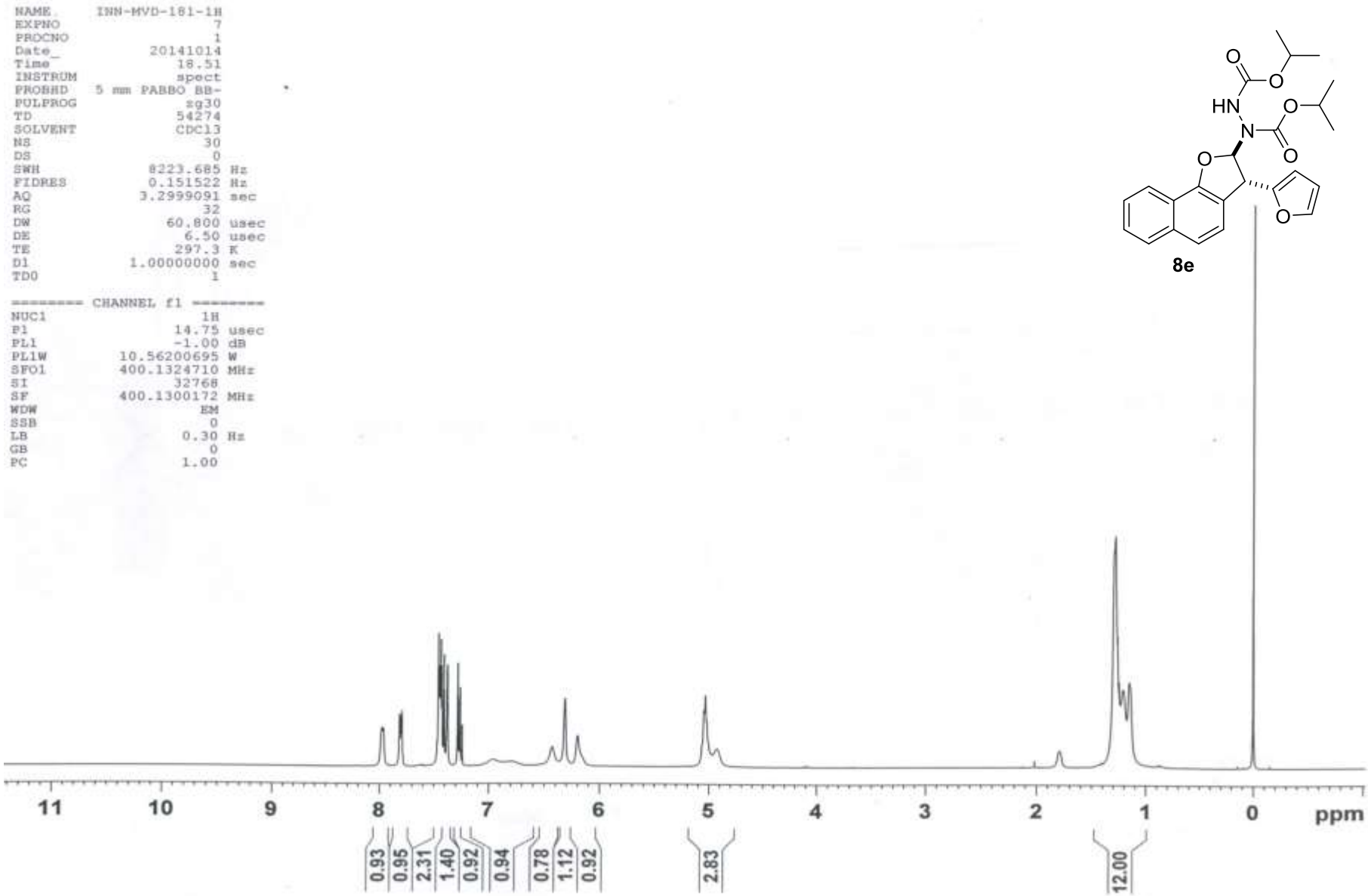


Figure S71. ^{1}H NMR Spectrum of **8e**

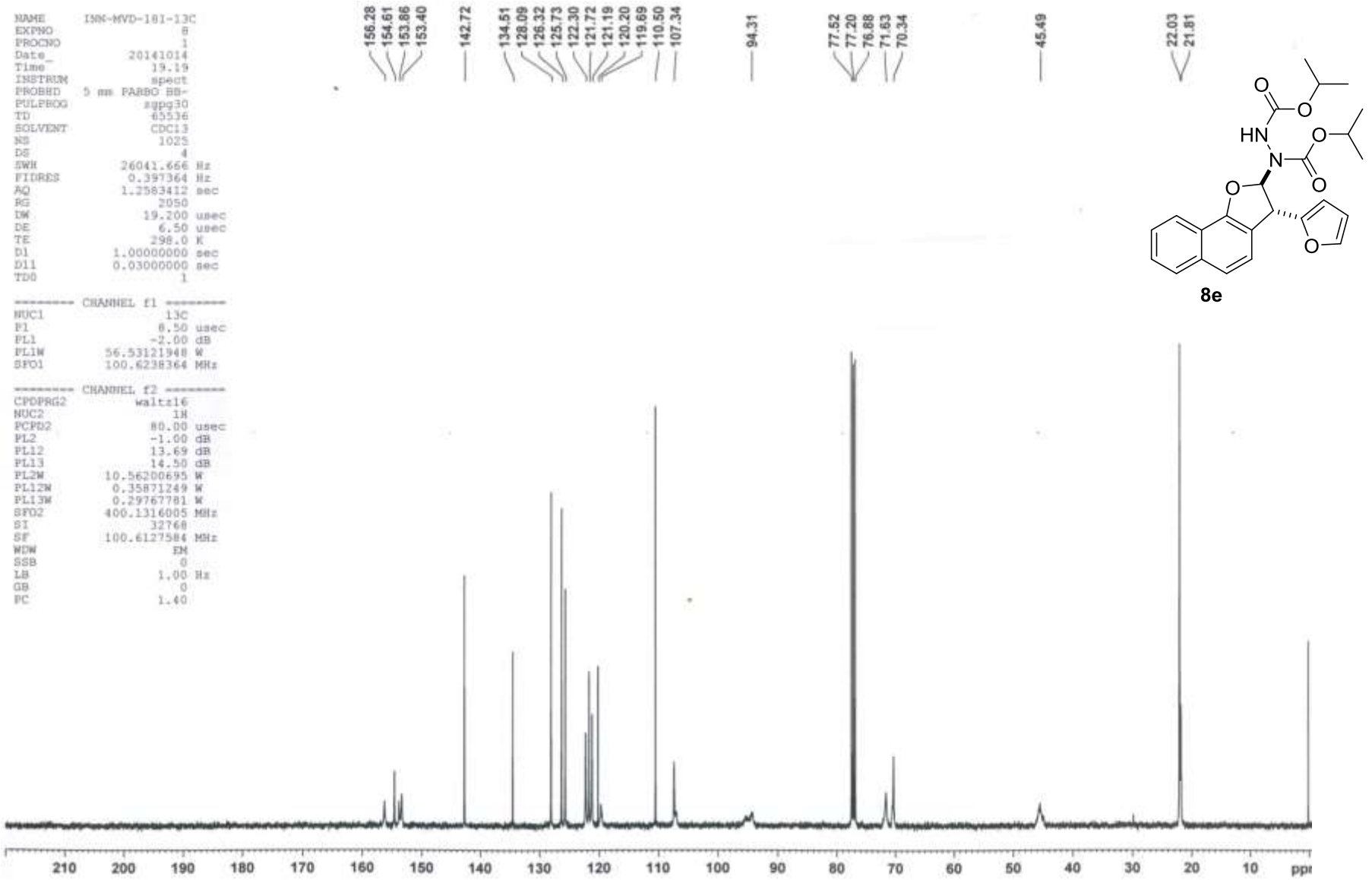


Figure S72. ^{13}C NMR Spectrum of **8e**

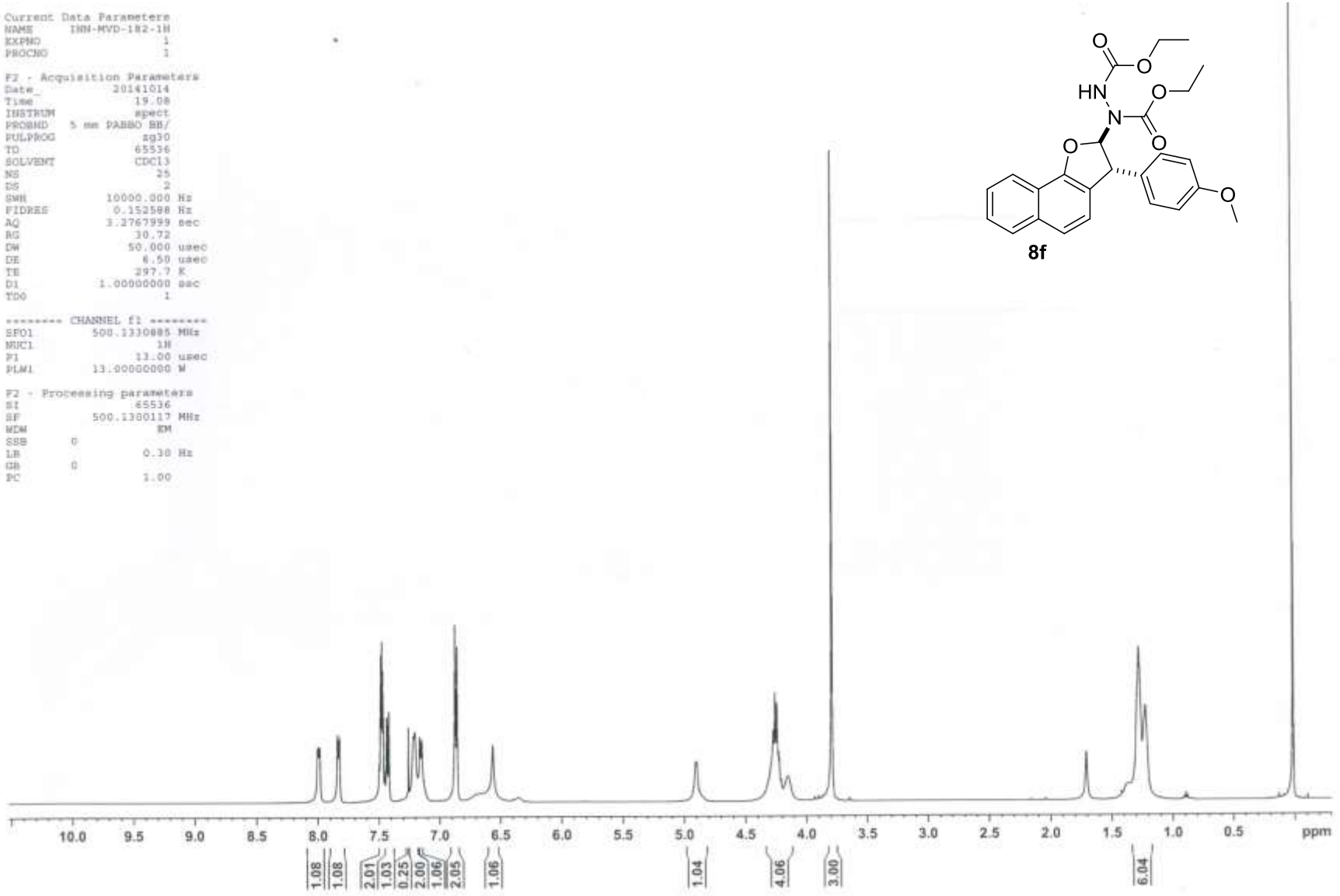


Figure S73. ^{1}H NMR Spectrum of **8f**

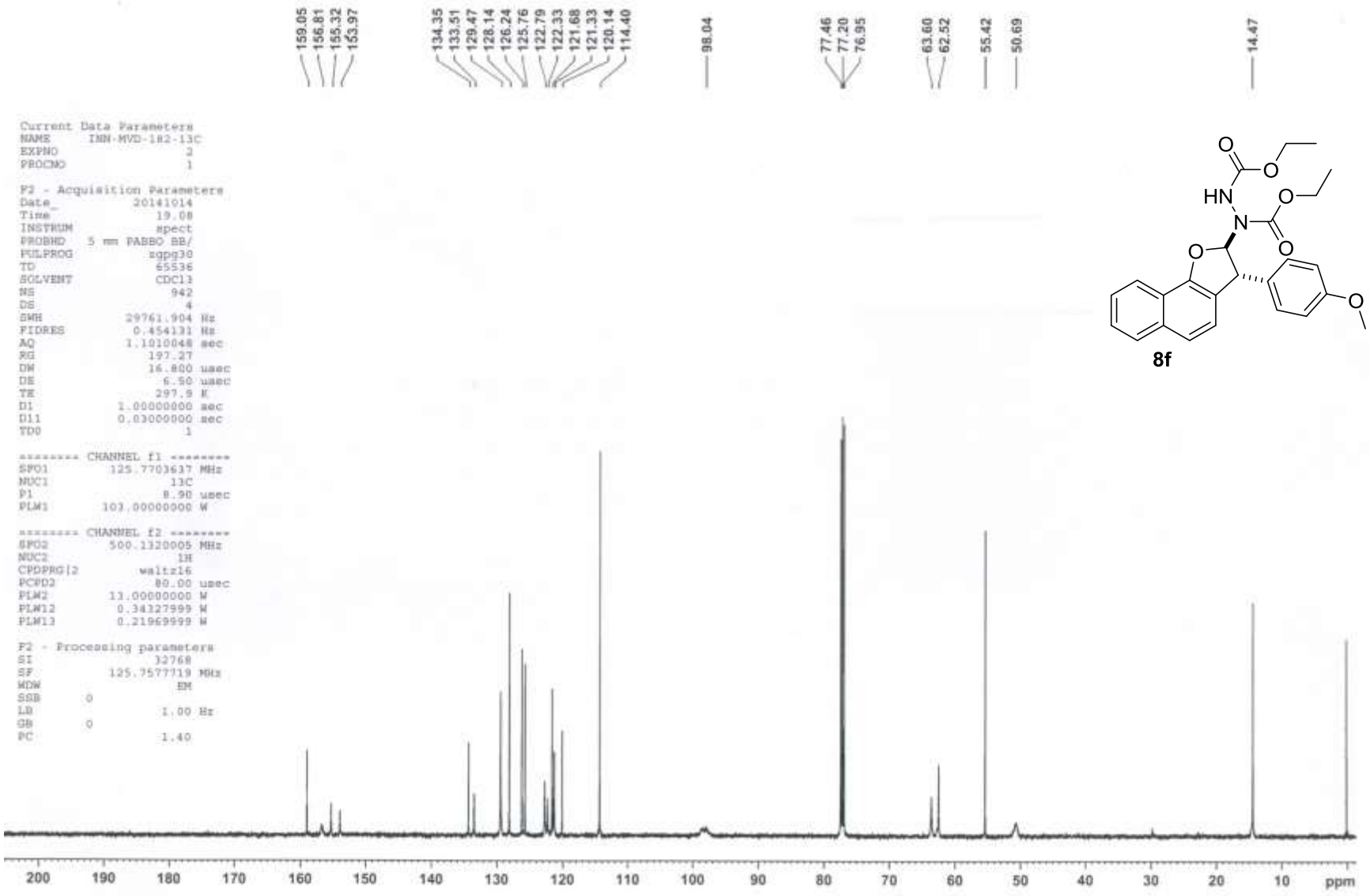


Figure S74. ^{13}C NMR Spectrum of **8f**

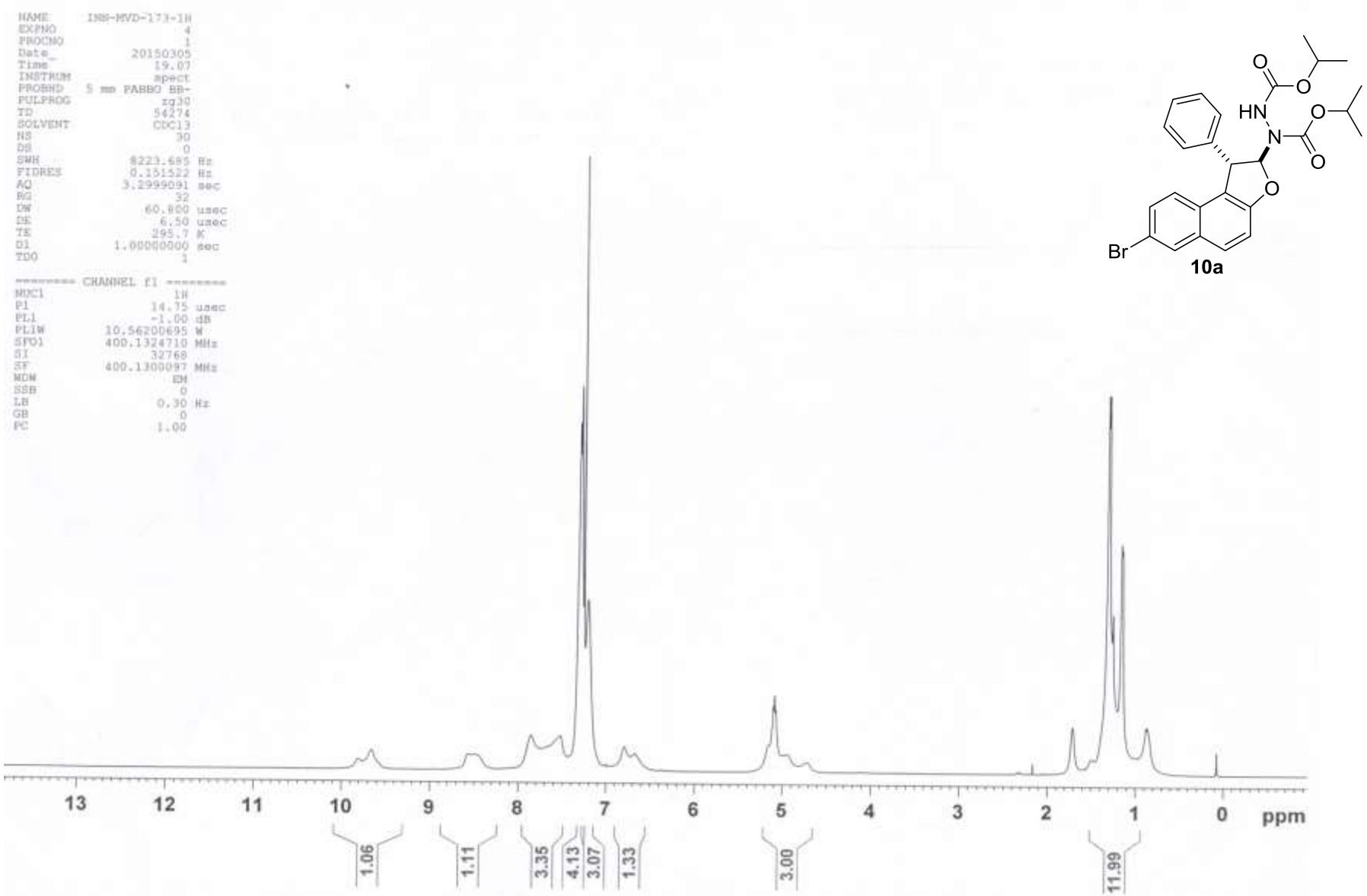


Figure S75. ^{1}H NMR Spectrum of **10a**

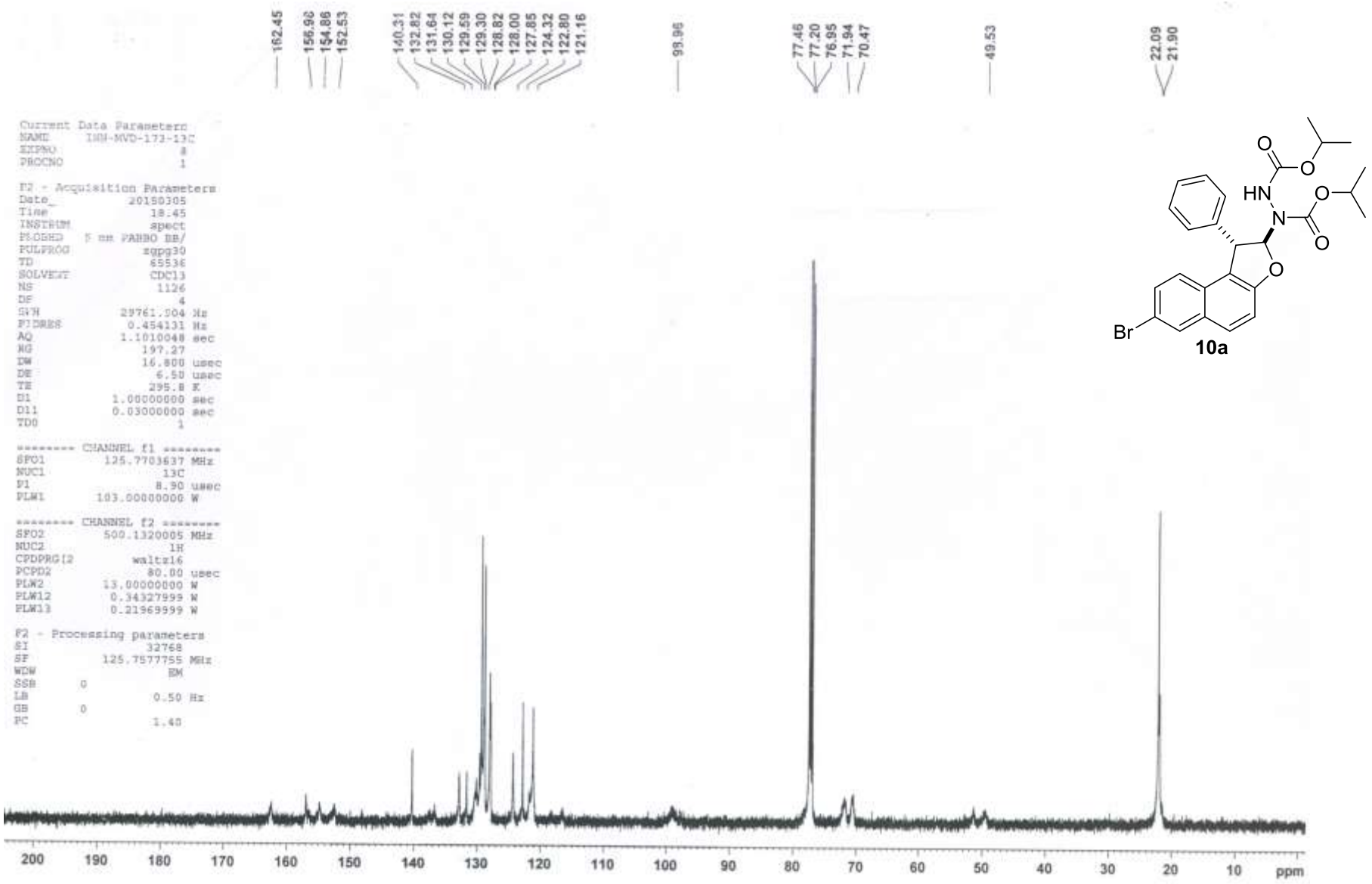


Figure S76. ^{13}C NMR Spectrum of **10a**

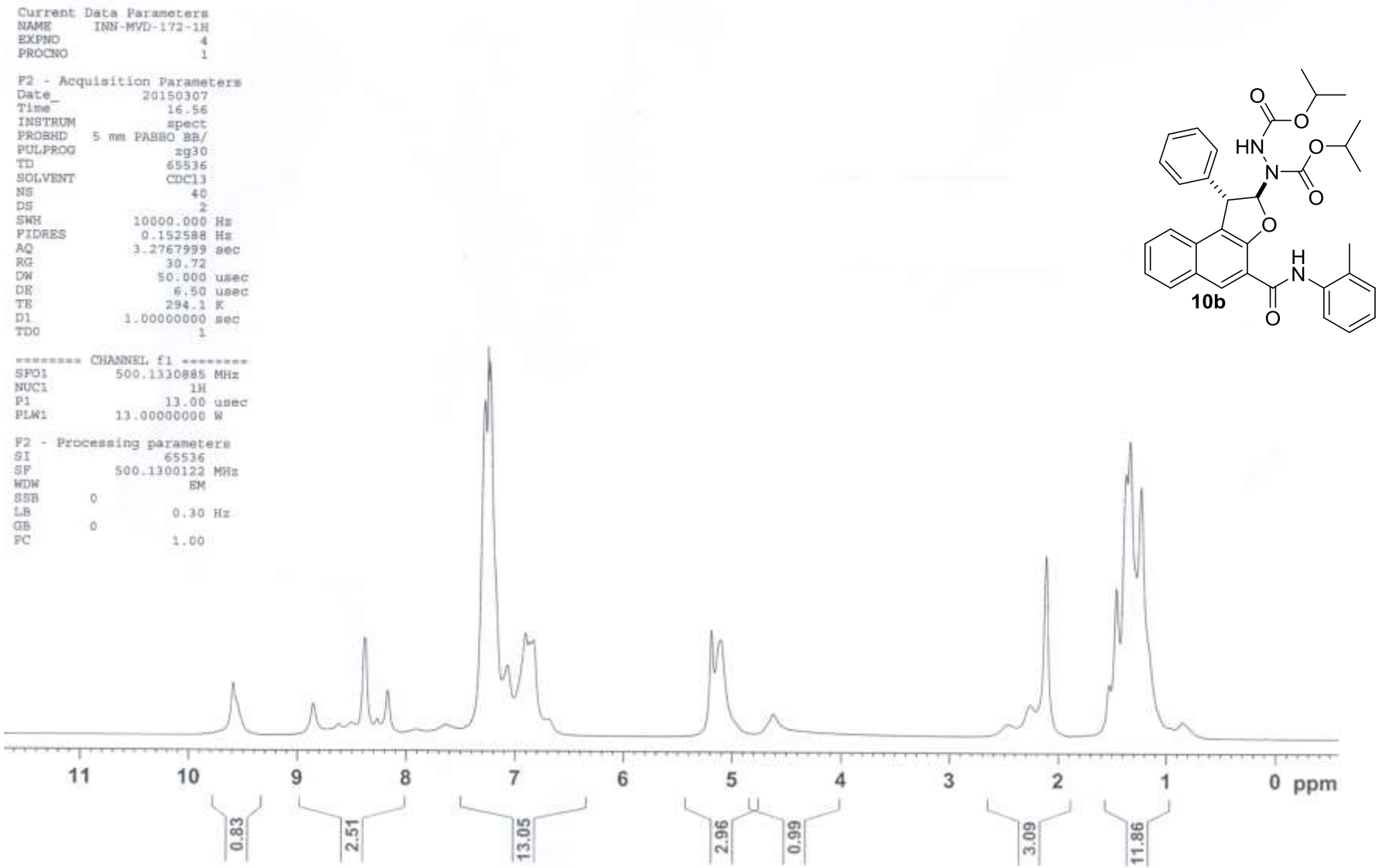


Figure S77. ^{1}H NMR Spectrum of **10b**

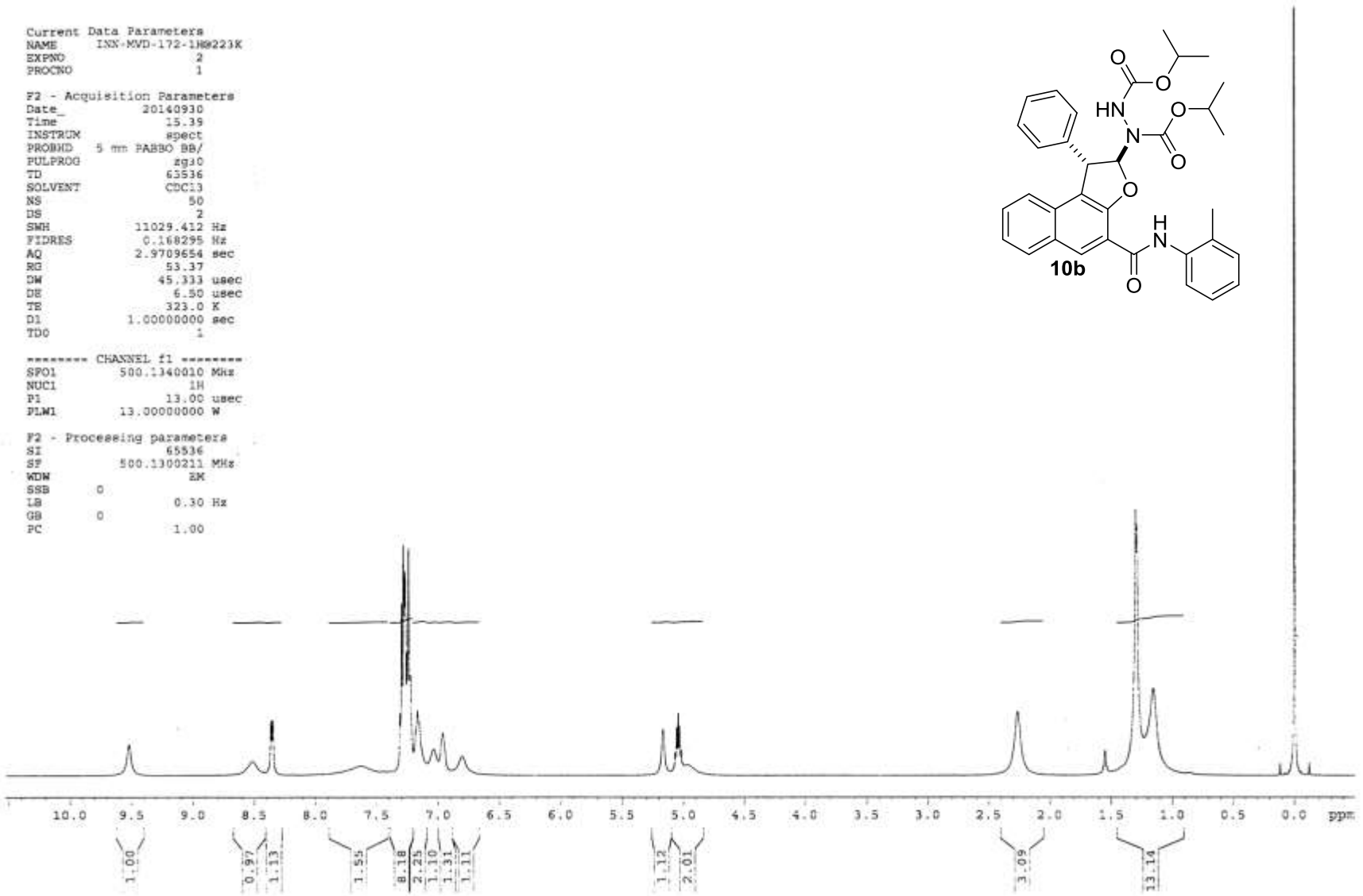


Figure S78. ^{1}H NMR Spectrum of **10b** at 323 k

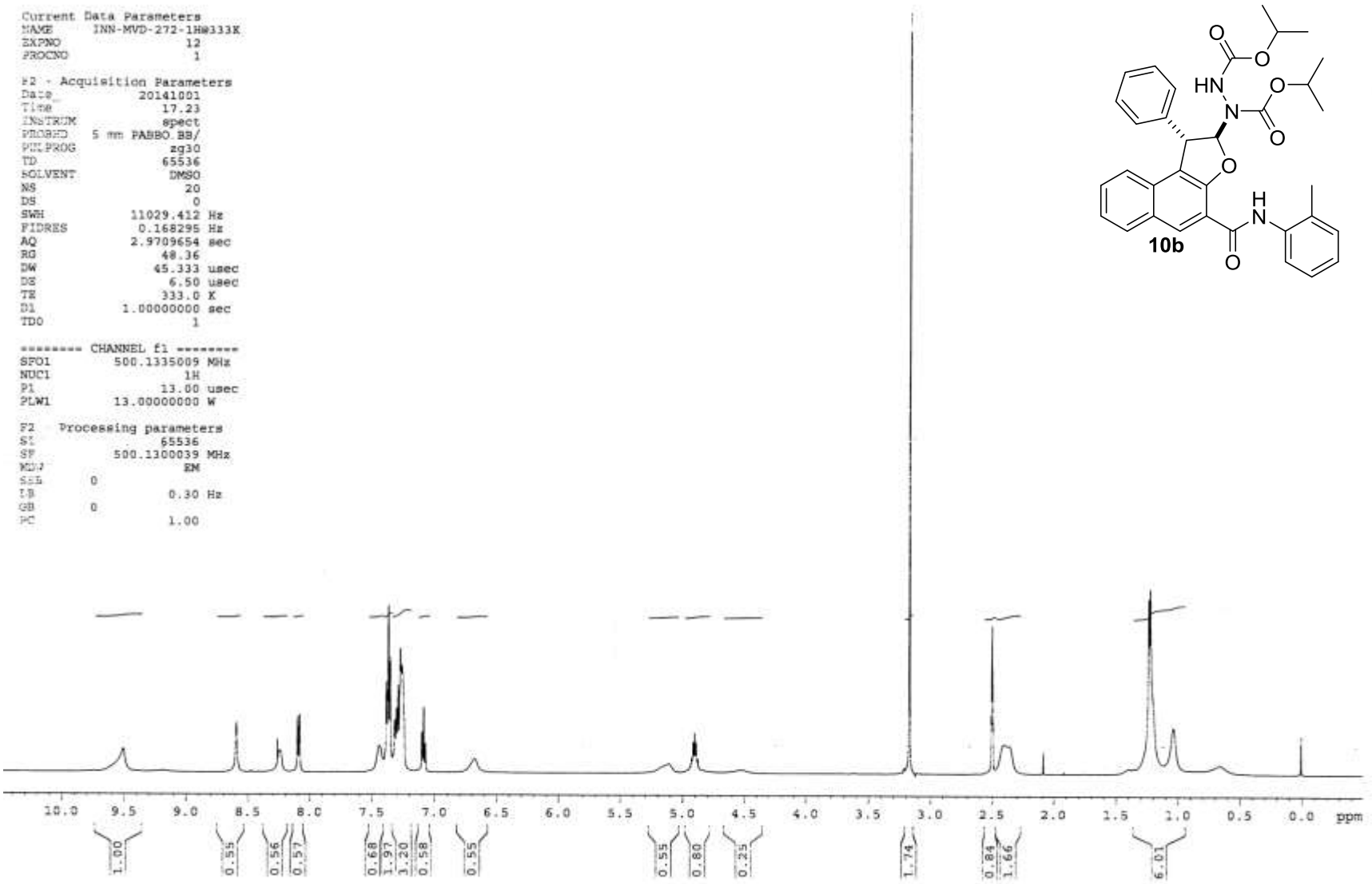


Figure S79. ^{1}H NMR Spectrum of **10b** at 333 k

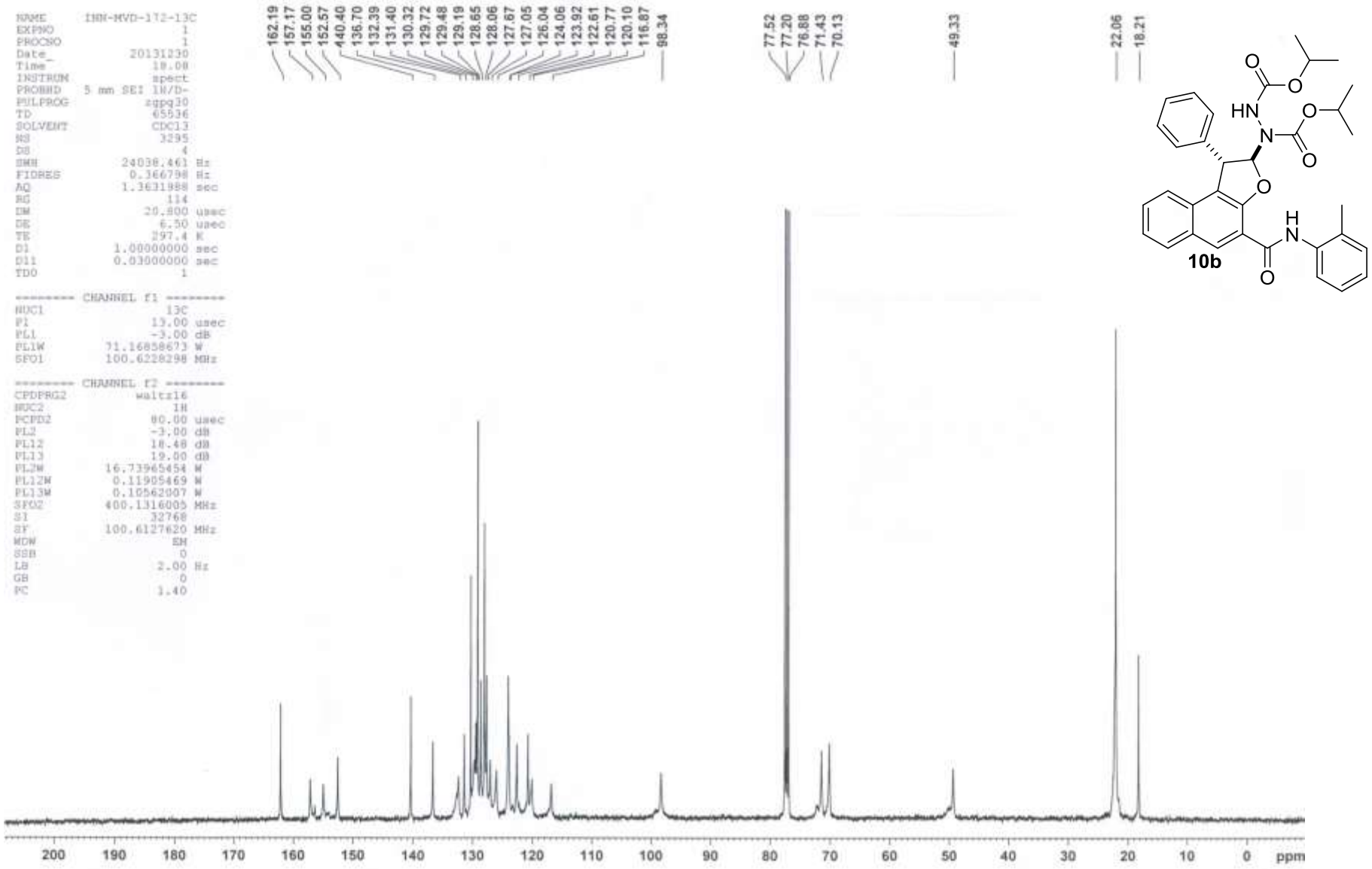


Figure S80. ^{13}C NMR Spectrum of **10b**

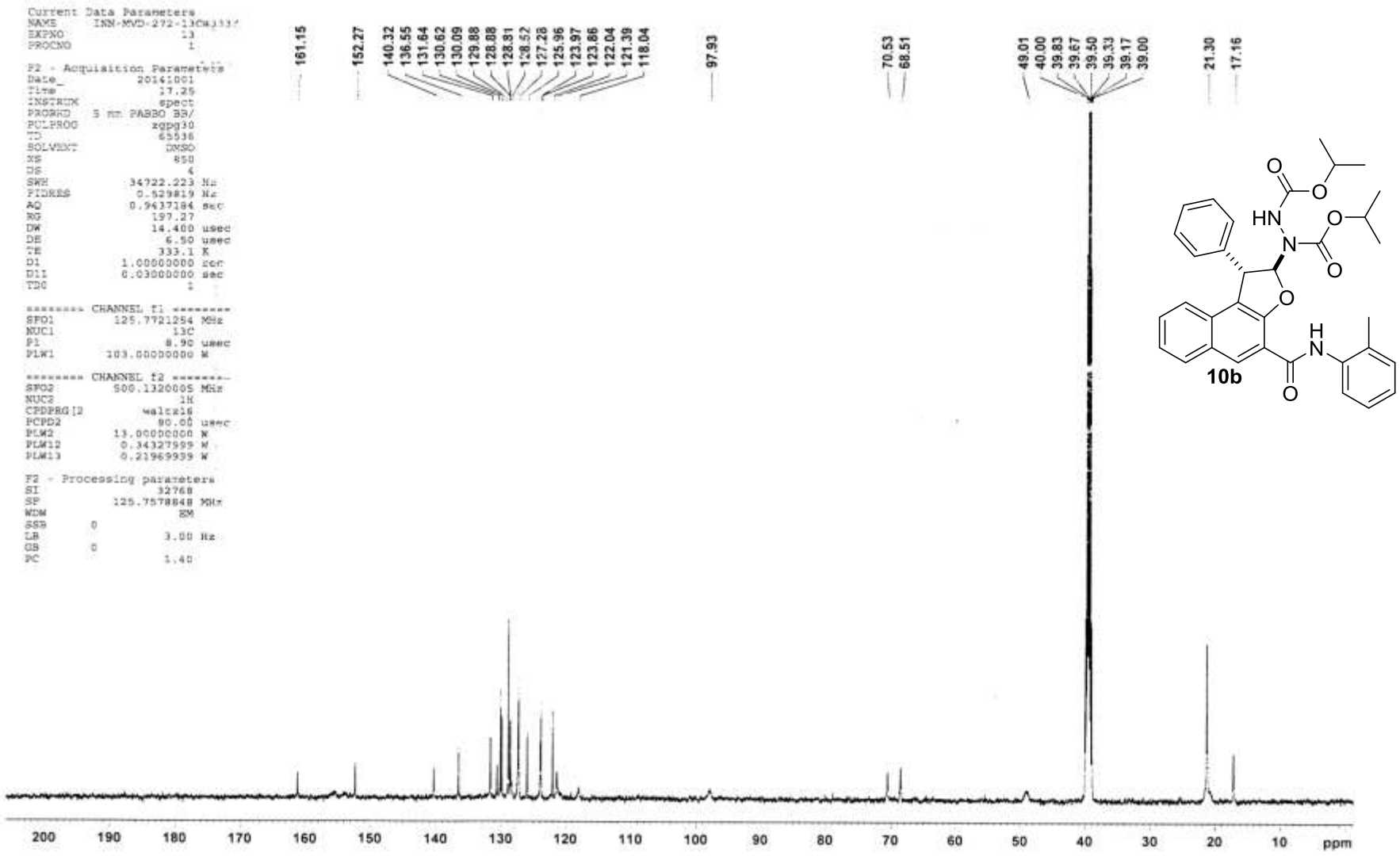


Figure S81. ^{13}C NMR Spectrum of **10b** at 333 k

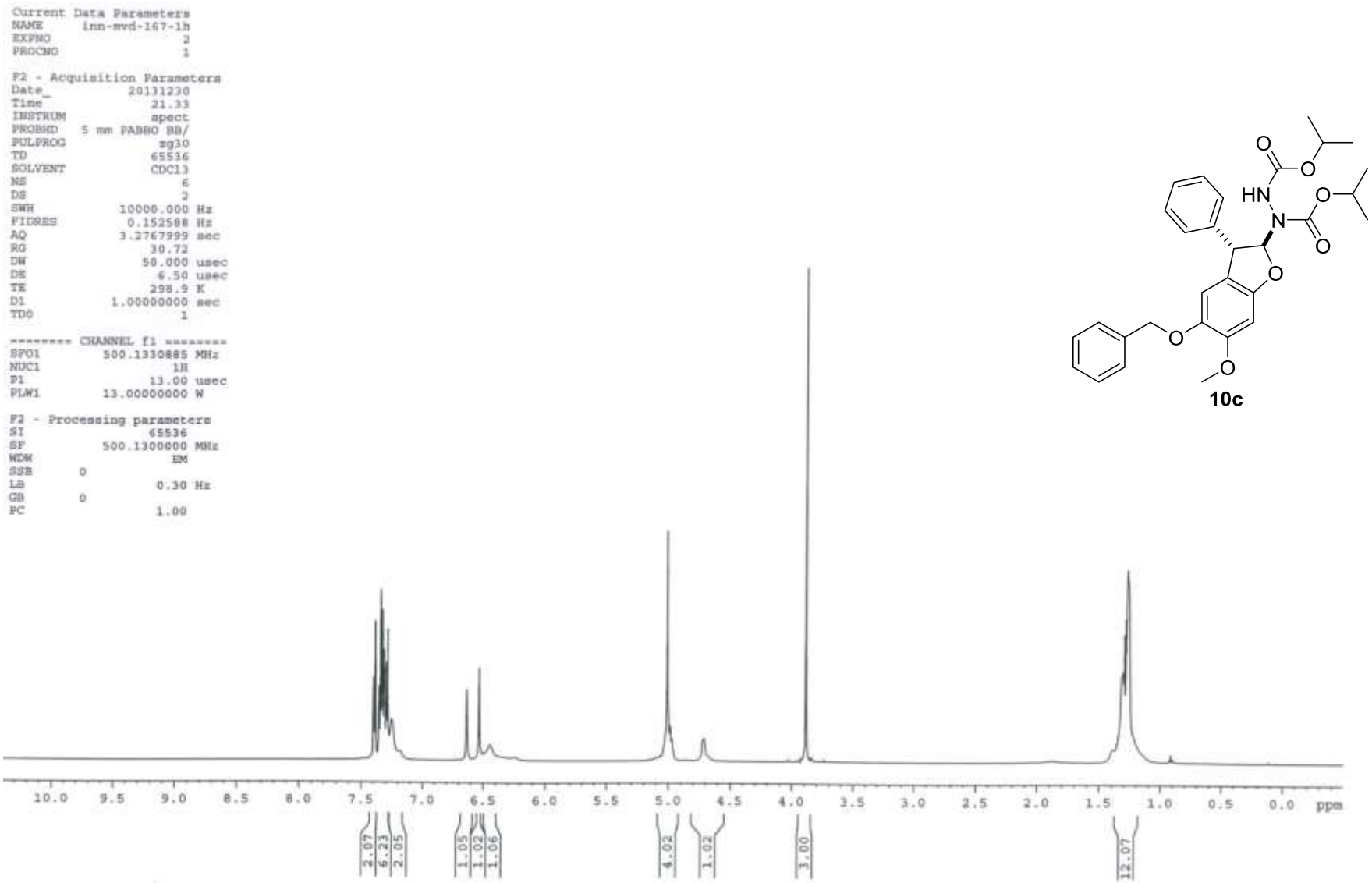


Figure S82. ^{1}H NMR Spectrum of **10c**

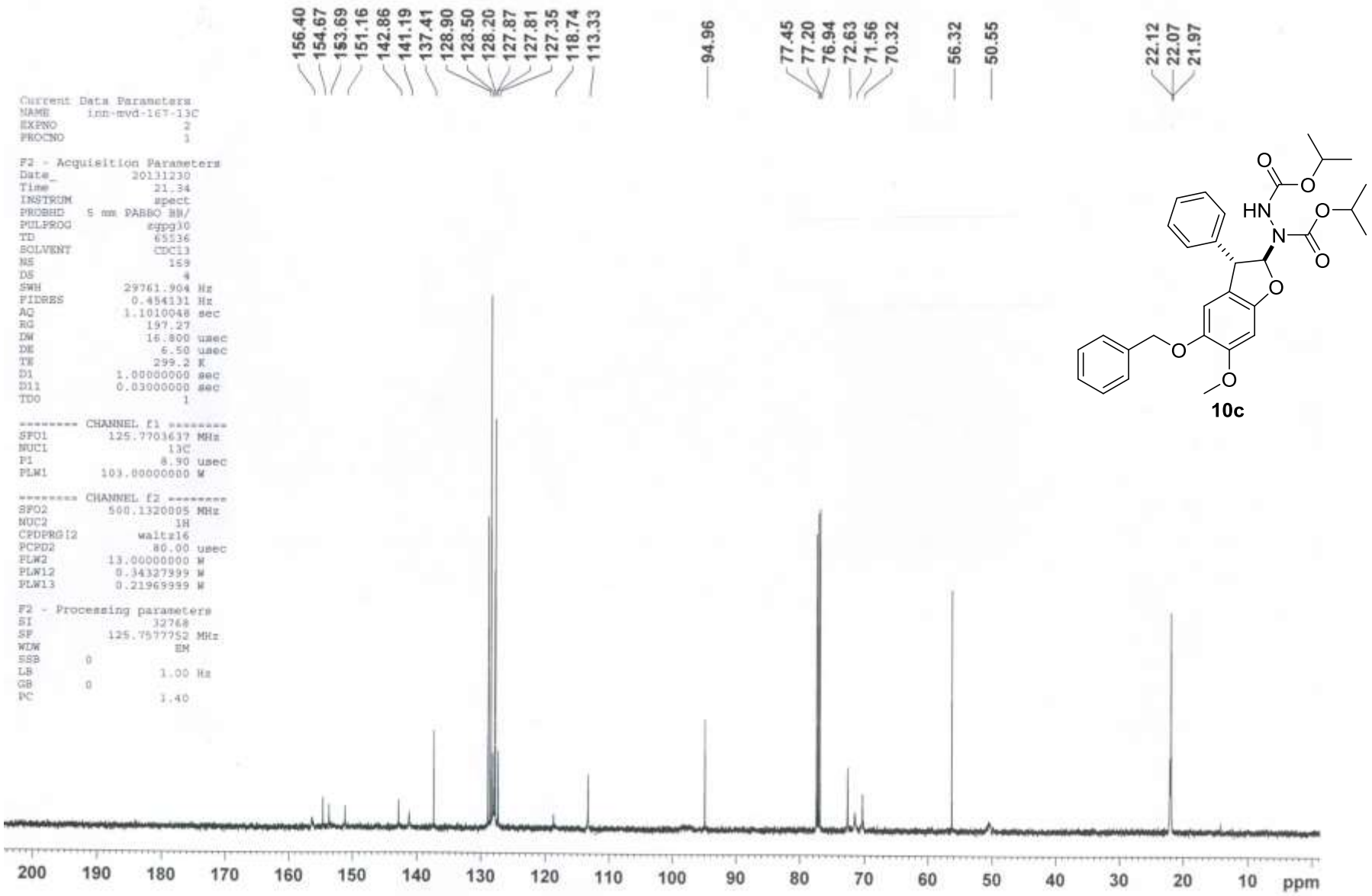


Figure S83. ^{13}C NMR Spectrum of **10c**