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

NMR Spectra and HPLC Profiles
for

Stereoselective synthesis of 2-hydrazino-2,3-dihydrofurans *via* cascade Michael addition-substitution involving reaction of curcumin and other 1,3-dicarbonyls with α-hydrazinonitroalkenes

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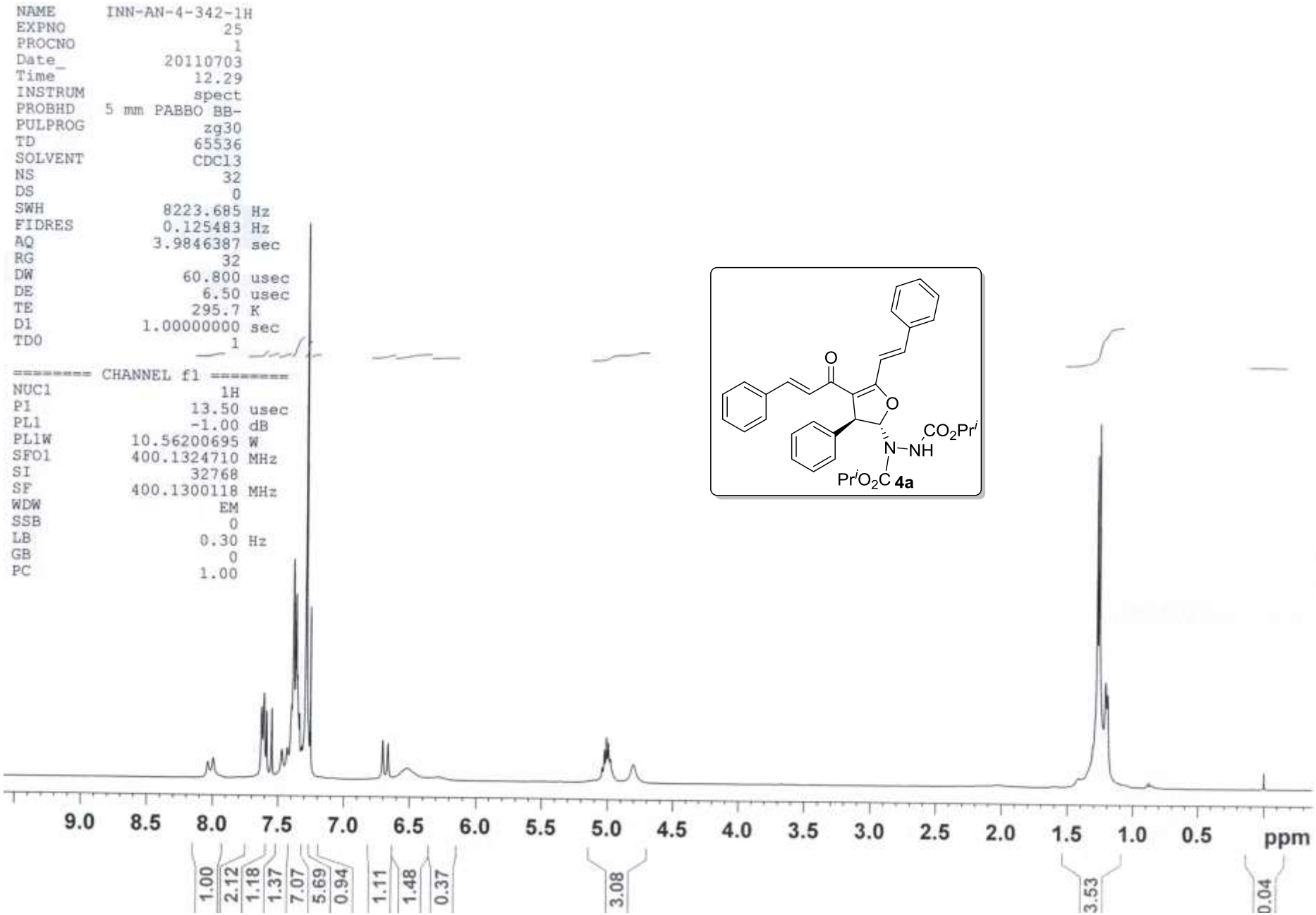


Fig S1. ^{1}H NMR Spectrum of 4a

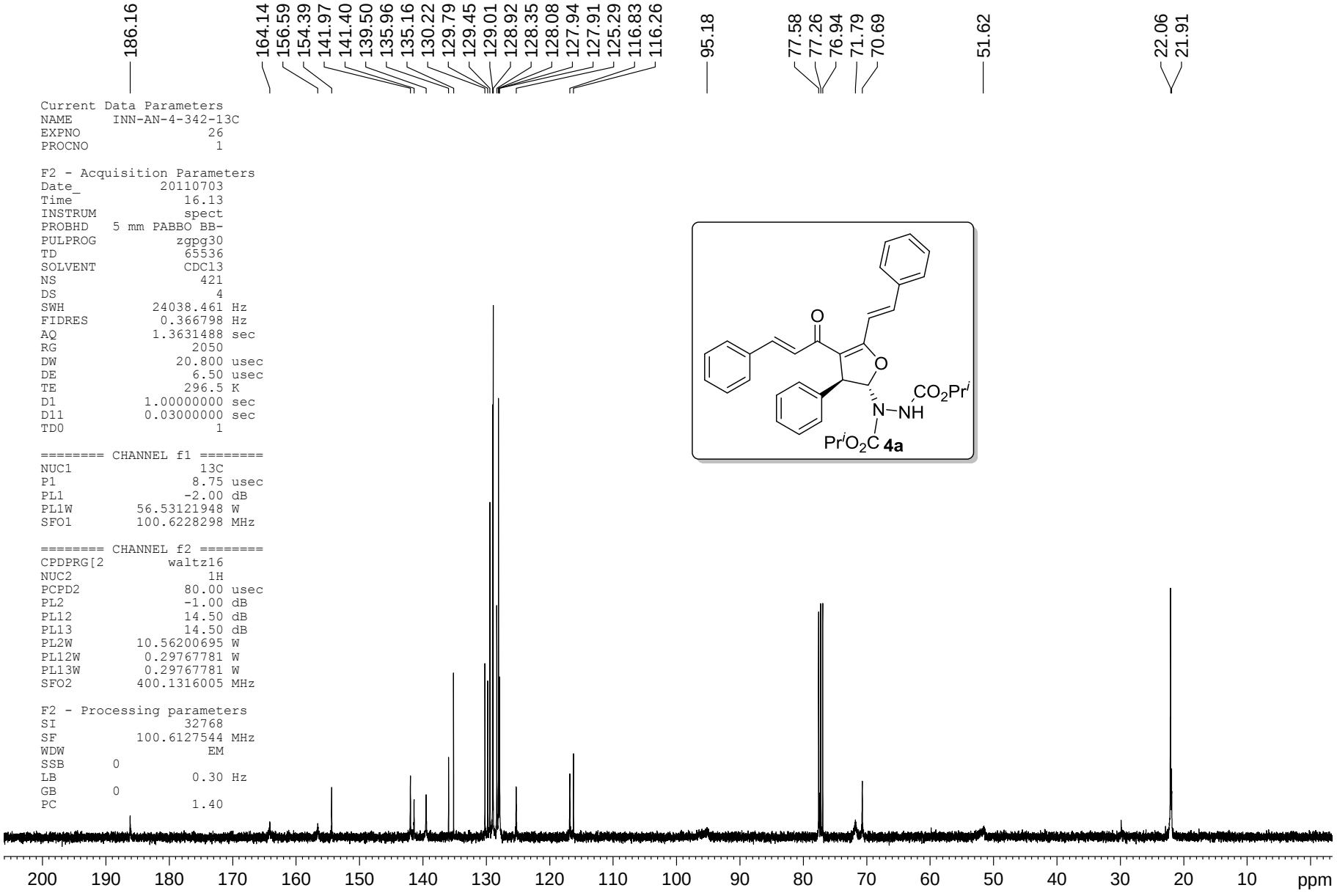


Fig S2. ^{13}C NMR Spectrum of 4a

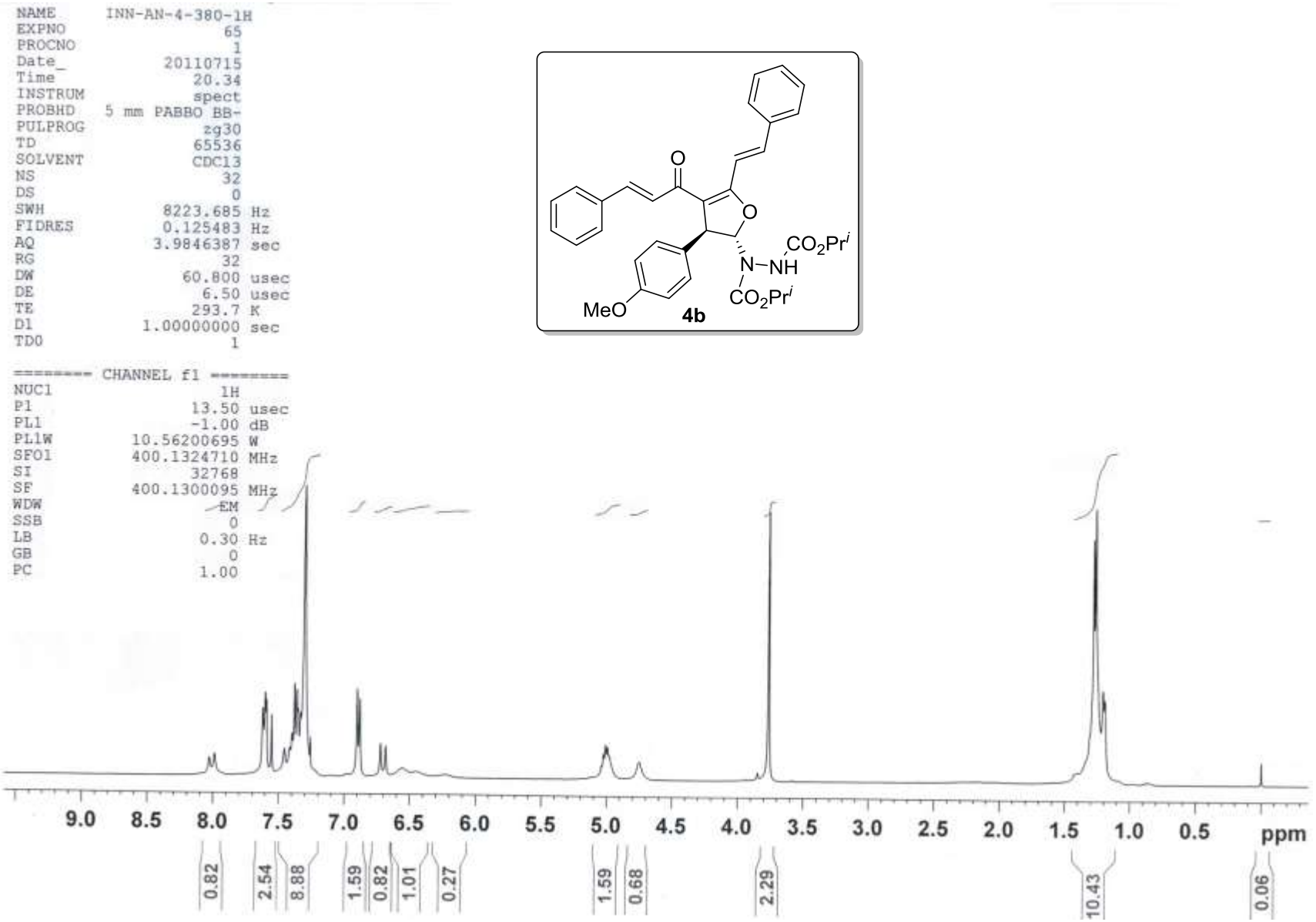


Fig S3. ^{1}H NMR Spectrum of 4b

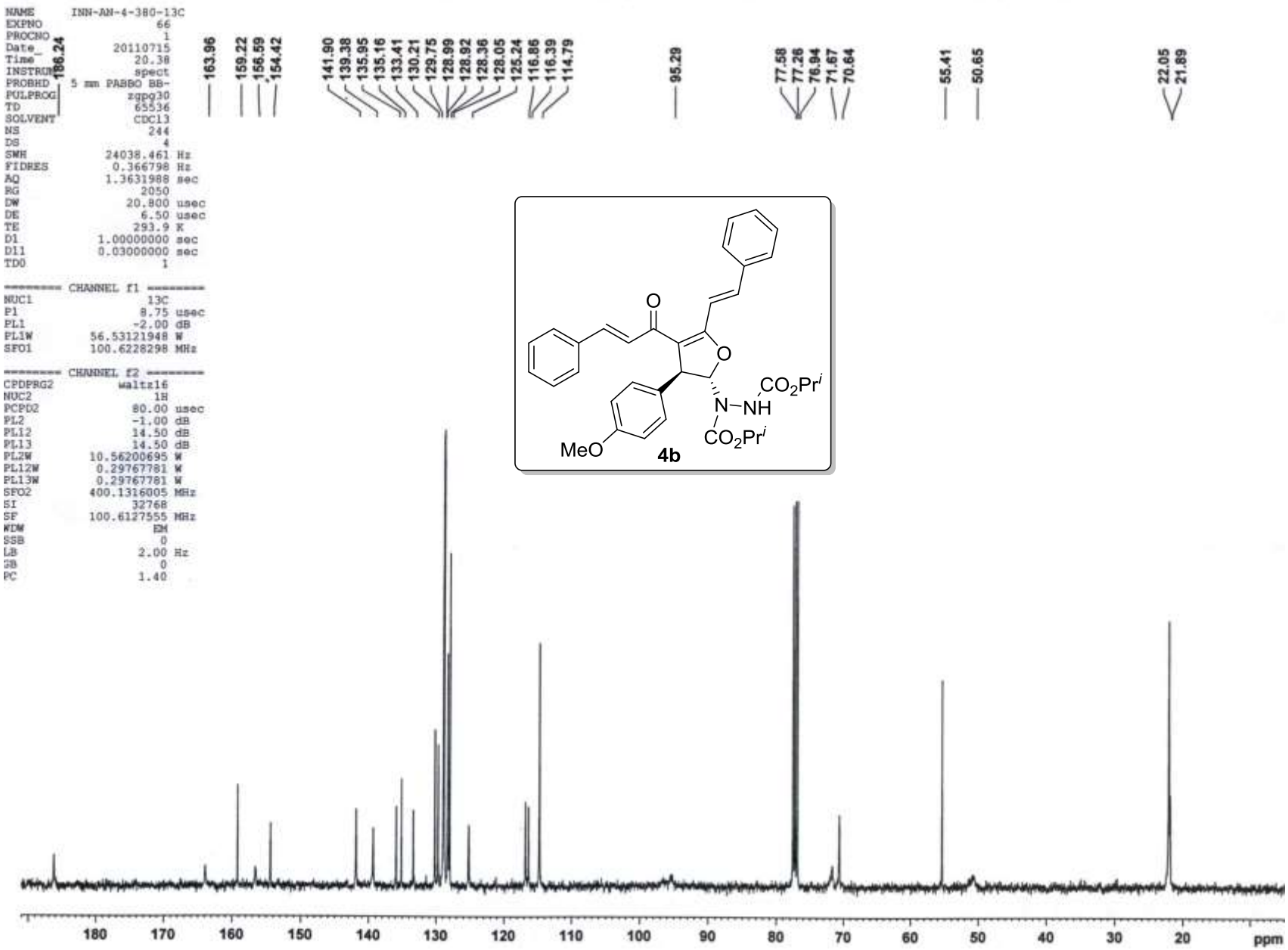


Fig S4. ^{13}C NMR Spectrum of 4b

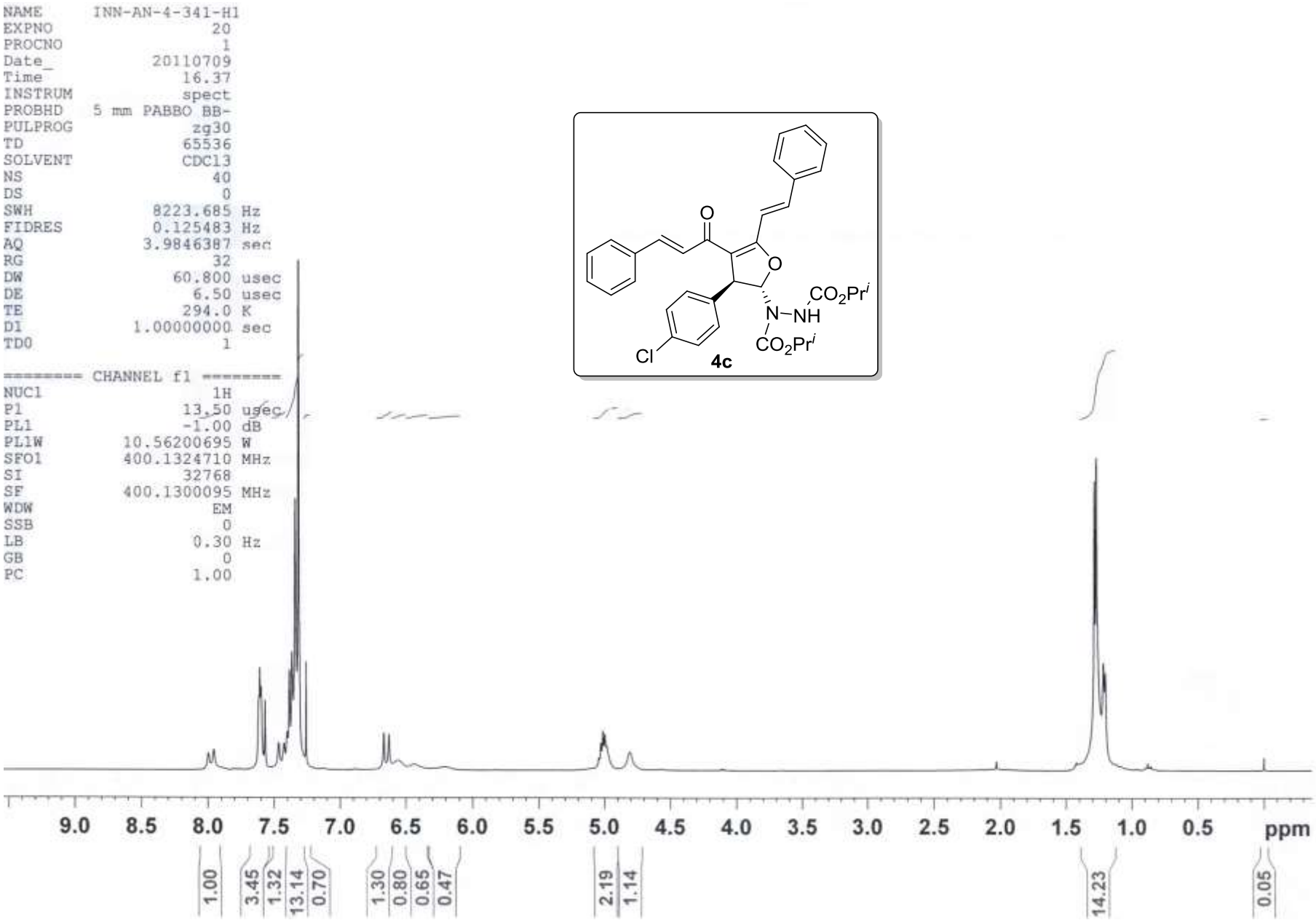


Fig S5. ^{1}H NMR Spectrum of 4c

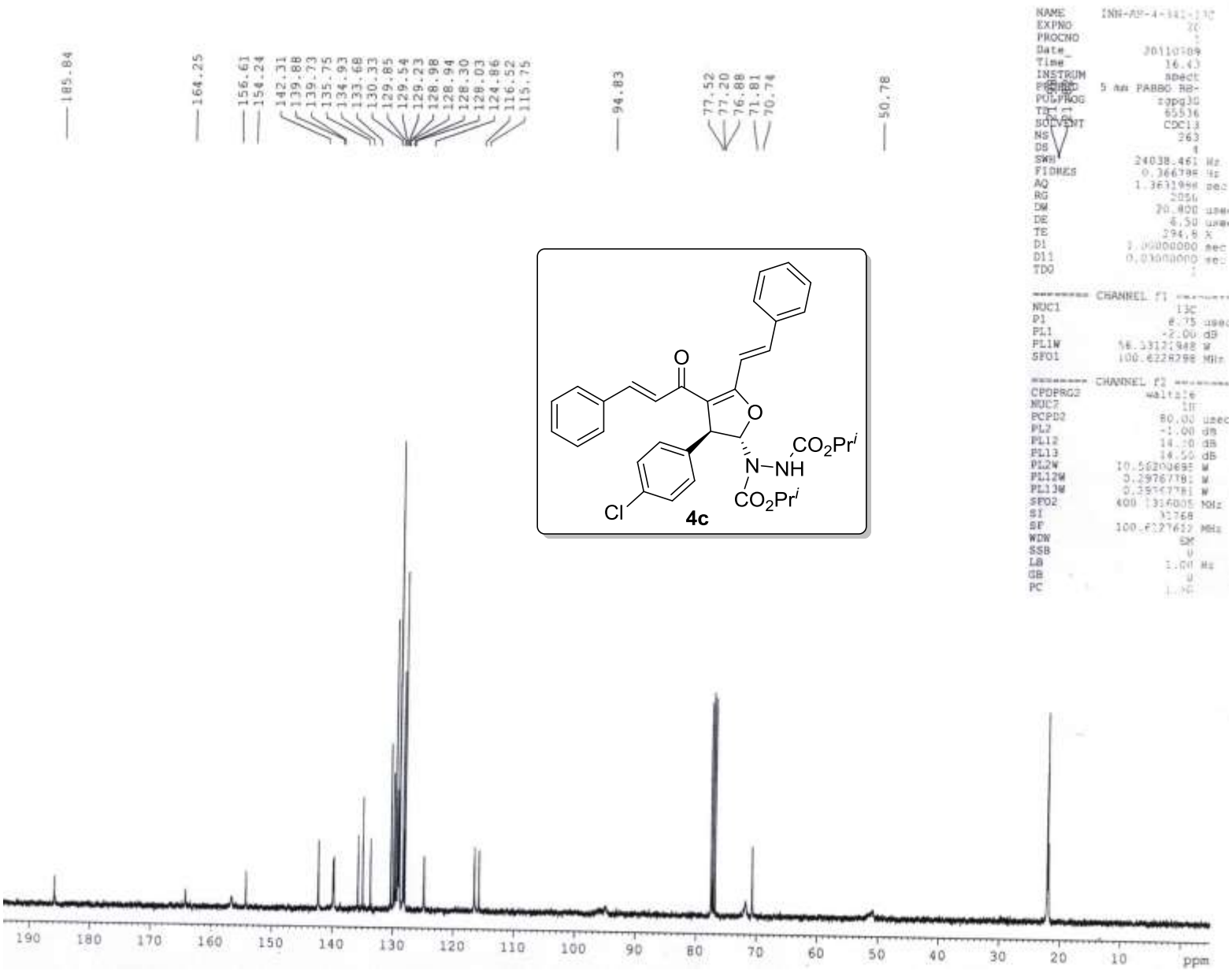


Fig S6. ^{13}C NMR Spectrum of 4c

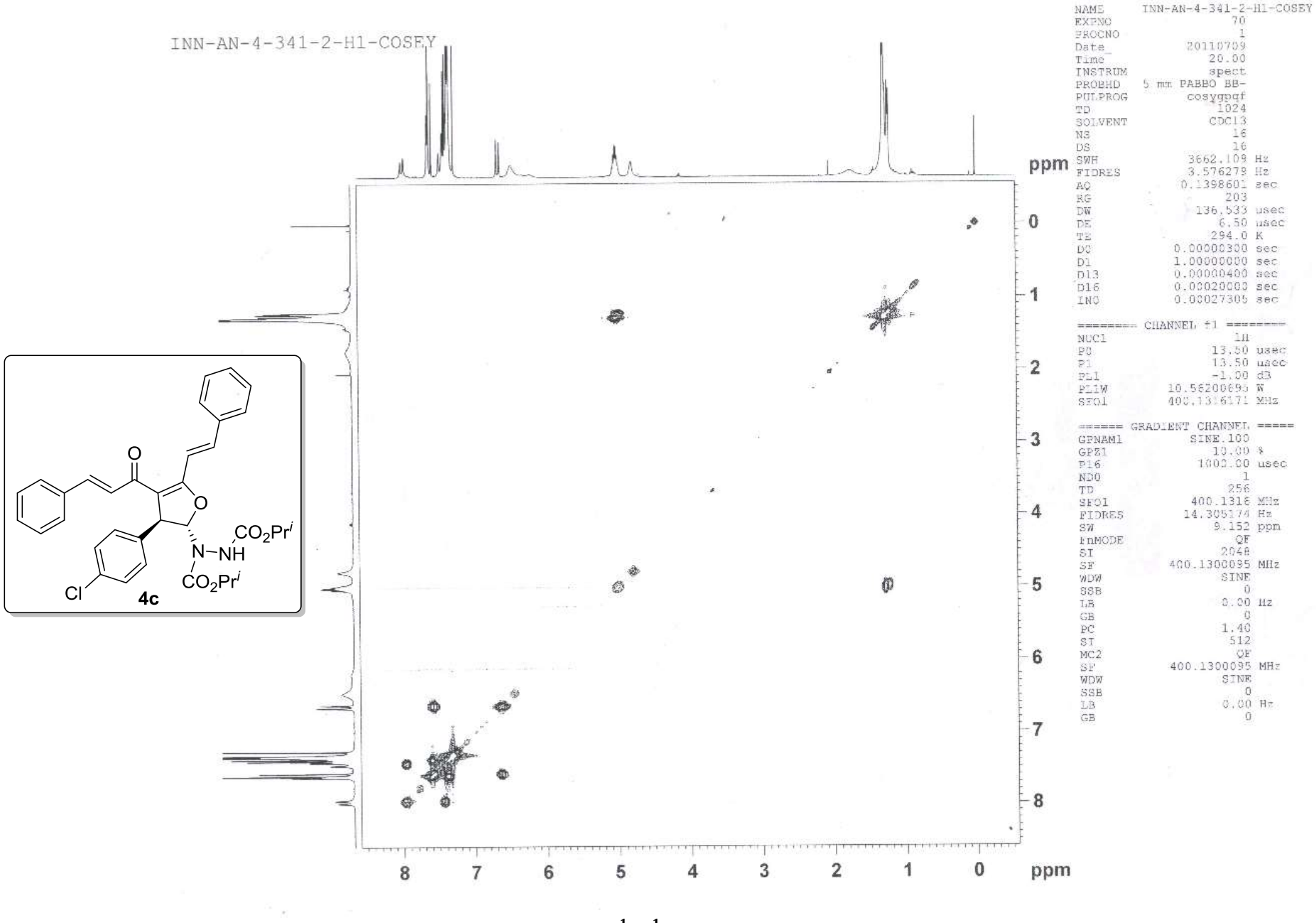
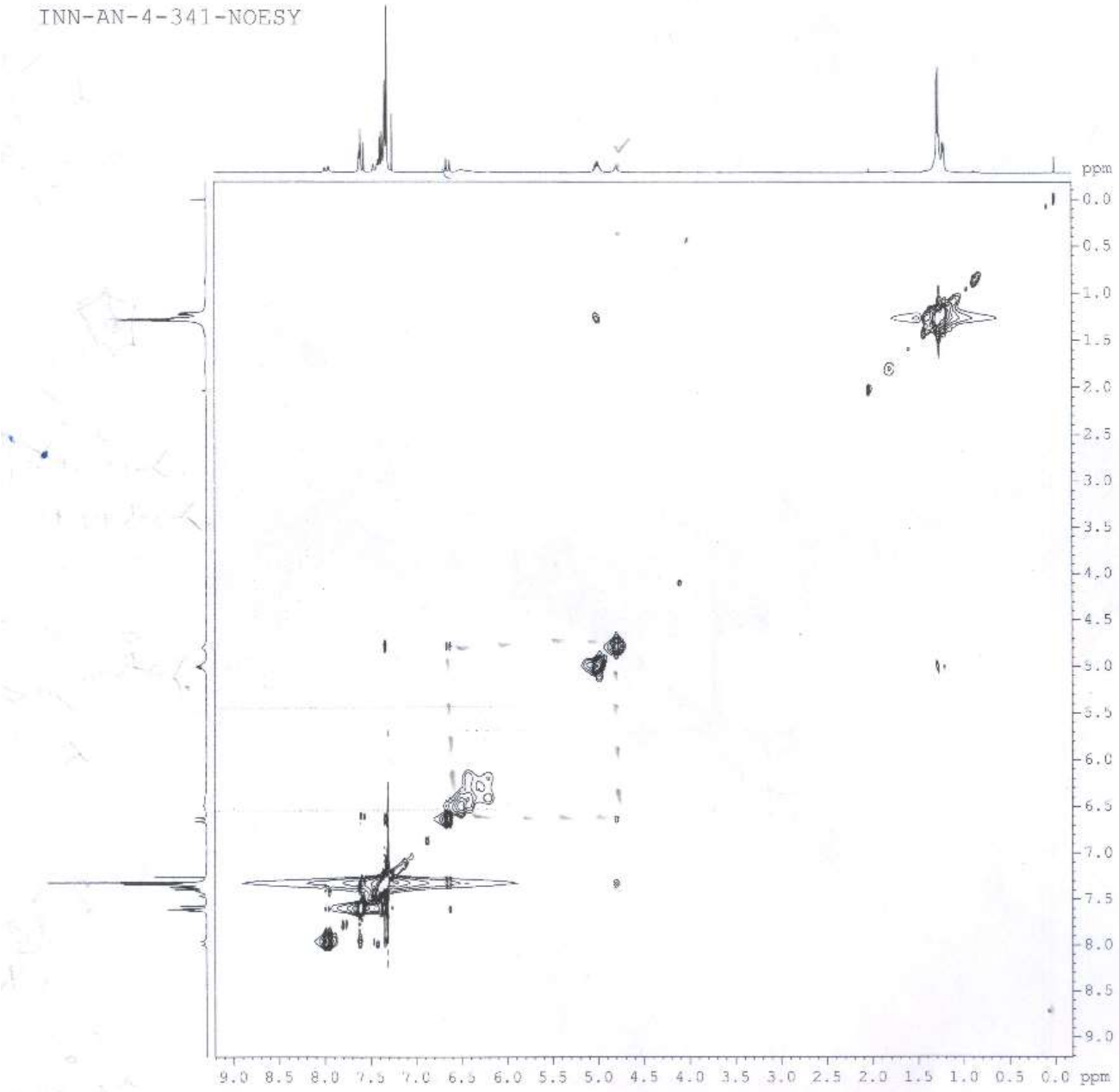


Fig S7. ^{1}H-^{1}H COSY NMR Spectrum of 4c

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PROCNO               1
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PULPROG         noesyph
TD                 2048
SOLVENT            CDCl3
NS                   32
DS                   16
SWH           3759.398 Hz
FIDRES        1.835644 Hz
AQ            0.2724340 sec
RG                2.56
DW              133.000 usec
DE                6.50 usec
TE              300.0 K
D0            0.00001575 sec
D1            1.90274398 sec
D8            0.30000001 sec
IN0           0.00026585 sec

-------- CHANNEL f1 --------
NUC1                1H
P1                13.50 usec
PL1               -1.00 dB
PL1W        10.56300545 W
SFO1        400.1318138 MHz
NDO                 1
TD                256
SFO1          400.1318 MHz
FIDRES       14.692340 Hz
SW            9.400 ppm
FnMODE       States-TPPI
SI                2048
SF          400.1300095 MHz
WDW               QSINE
SSB                 2
LB                0.00 Hz
GB                  0
PC                1.00
SI                 512
MC2          States-TPPI
SF          400.1300095 MHz
WDW               QSINE
SSB                 2
LB                0.00 Hz
GB                  0
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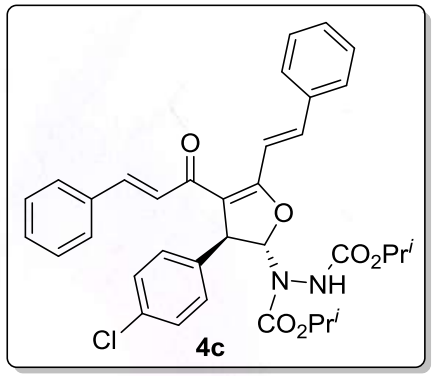
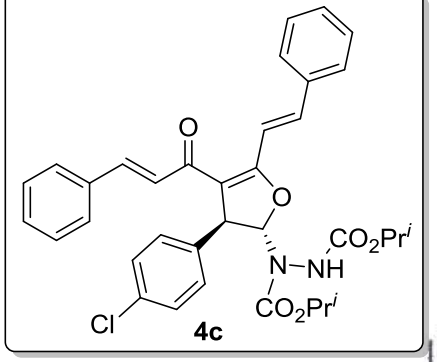
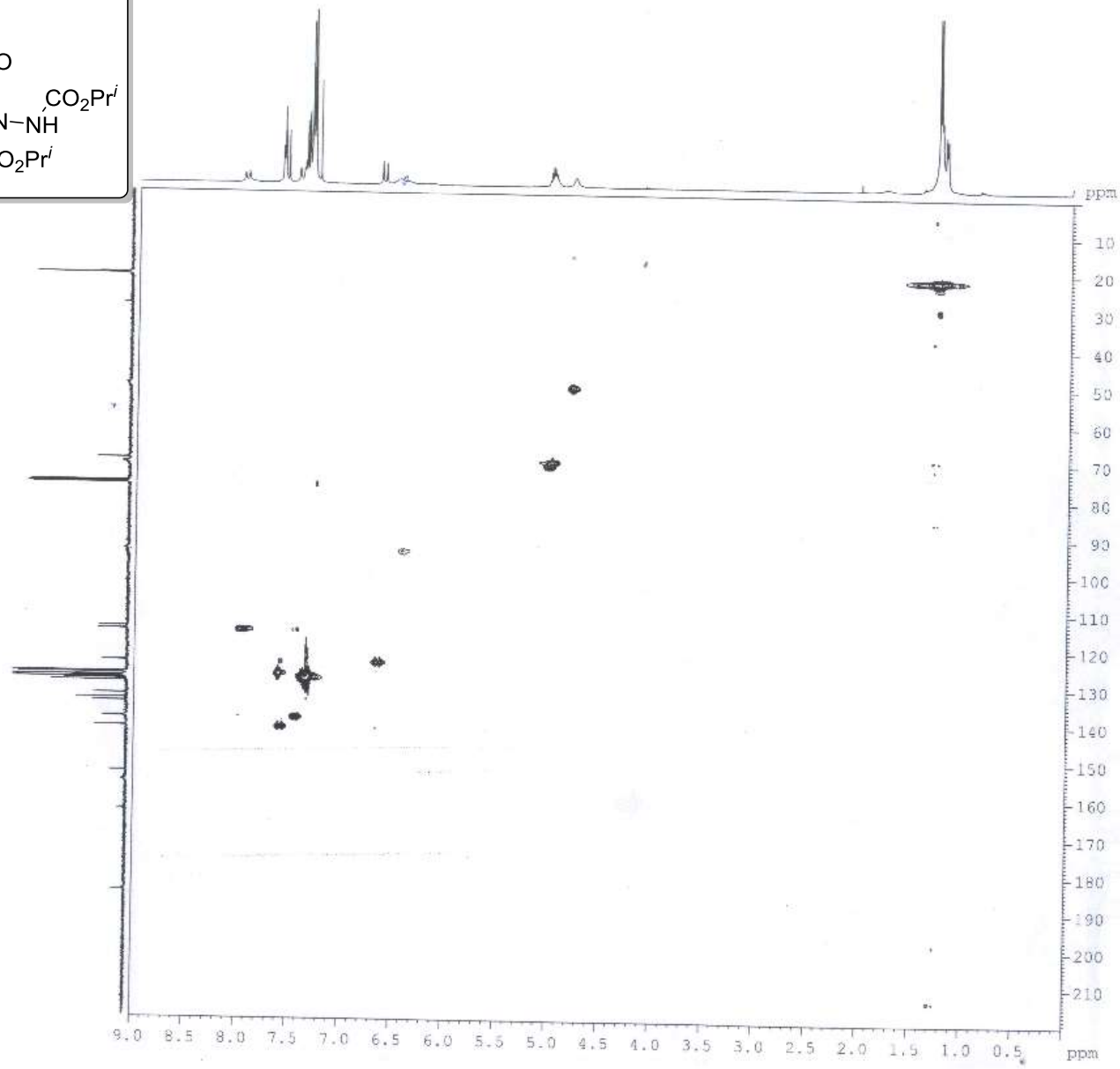


Fig S8. ^{1}H-^{1}H NOESY NMR Spectrum of 4c



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PROCNO                      1
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PULPROG              hsqcetgp
TD                       2048
SOLVENT                CDCl3
NS                          16
DS                          16
SWH                 3759.398 H
FIDRES              1.835644 H
AQ                  0.2724340 s
RG                       1820
DW                    133.000 u
DE                      6.50 u
TE                     295.1 K
CNST2              145.0000000
D0                 0.00000300 s
D1                 1.45187199 s
D4                 0.00172414 s
D11                0.03000000 s
D13                0.00000400 s
D16                0.00020000 s
IN0                0.00002070 s
ZGOPTNS

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.50 u
P2                      27.00 u
P28                      0.10 u
PL1                     -1.00 d
PL1W               10.56200695 W
SFO1              400.1318138 M

======== CHANNEL f2 ========
CPDPRG2                 garp
NUC2                      13C
P3                       8.75 u
P4                      17.50 u
PCPD2                   70.00 u
PL2                     -2.00 d
PL12                    16.00 d
PL2W               56.53121948 W
PL12W               0.89595944 W
SFO2              100.6248425 M

====== GRADIENT CHANNEL =
GPNAM1                SINE.100
GPNAM2                SINE.100
GPZ1                     80.00 %
GPZ2                     20.10 %
P16                    1000.00 u
ND0                          2
TD                         256
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Fig S9. ^{1}H-^{13}C HSQC NMR Spectrum of 4c

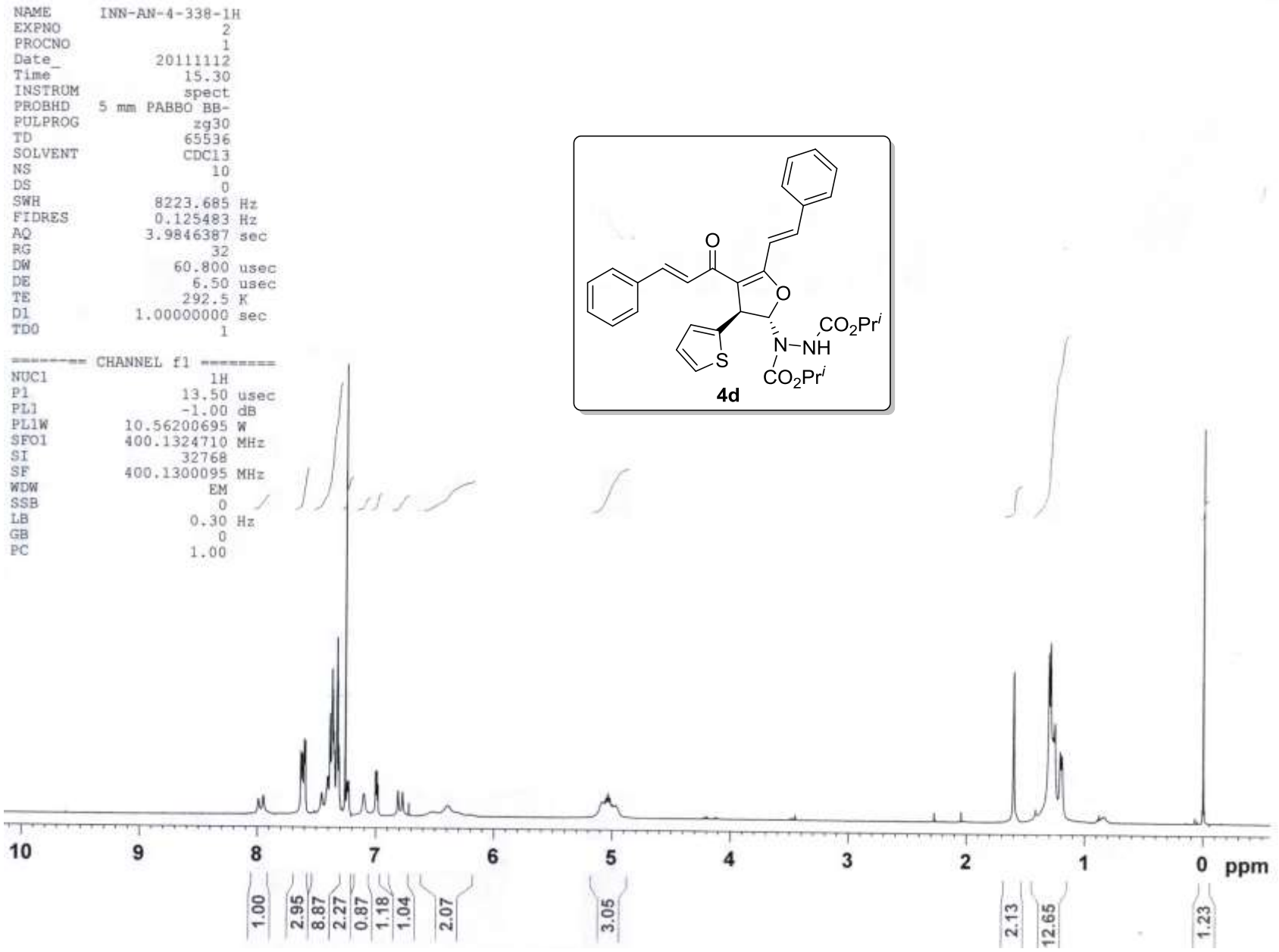

Fig S10. ^{1}H NMR Spectrum of 4d

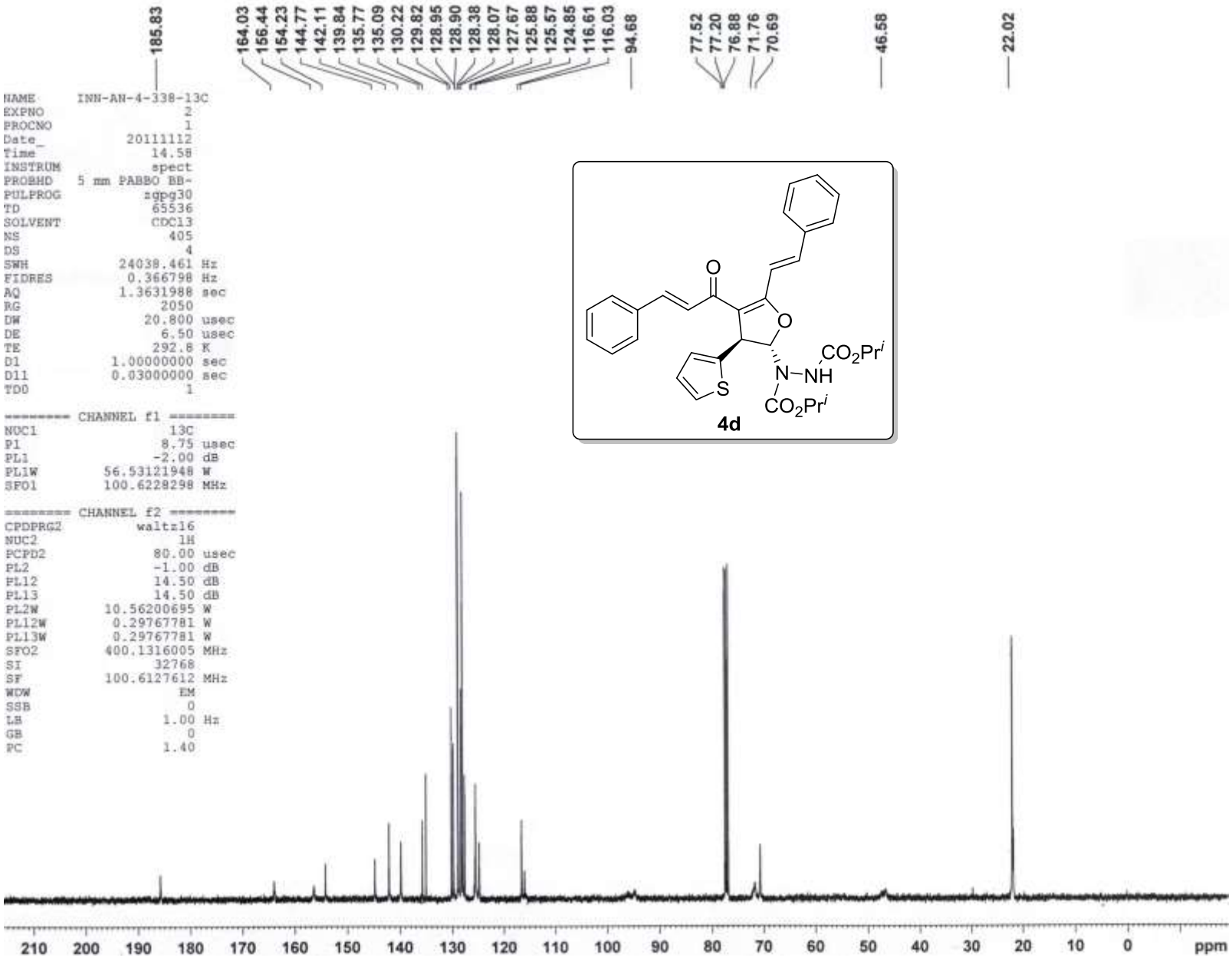

Fig S11. ^{13}C NMR Spectrum of 4d

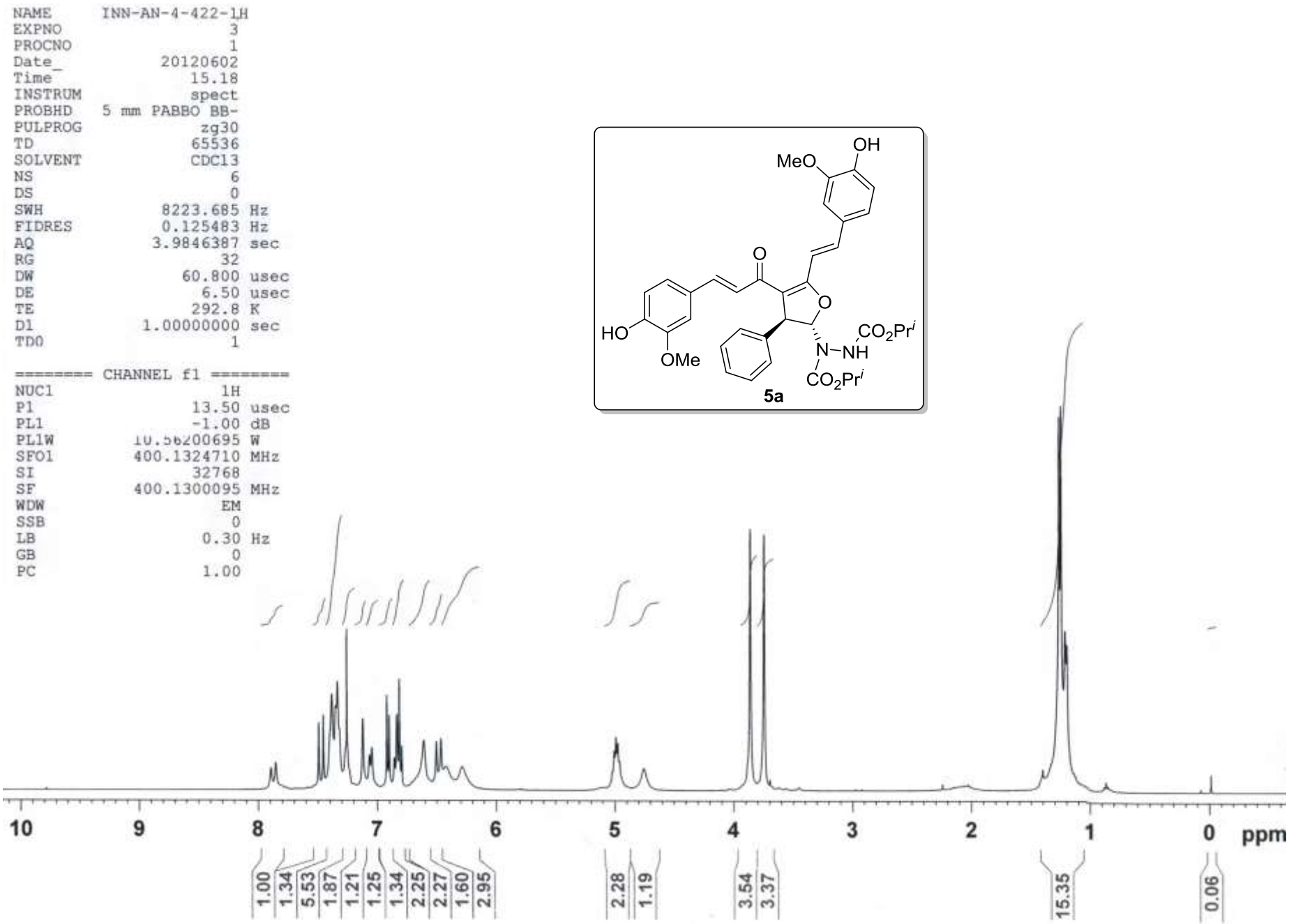

Fig S12. ^{1}H NMR Spectrum of 5a

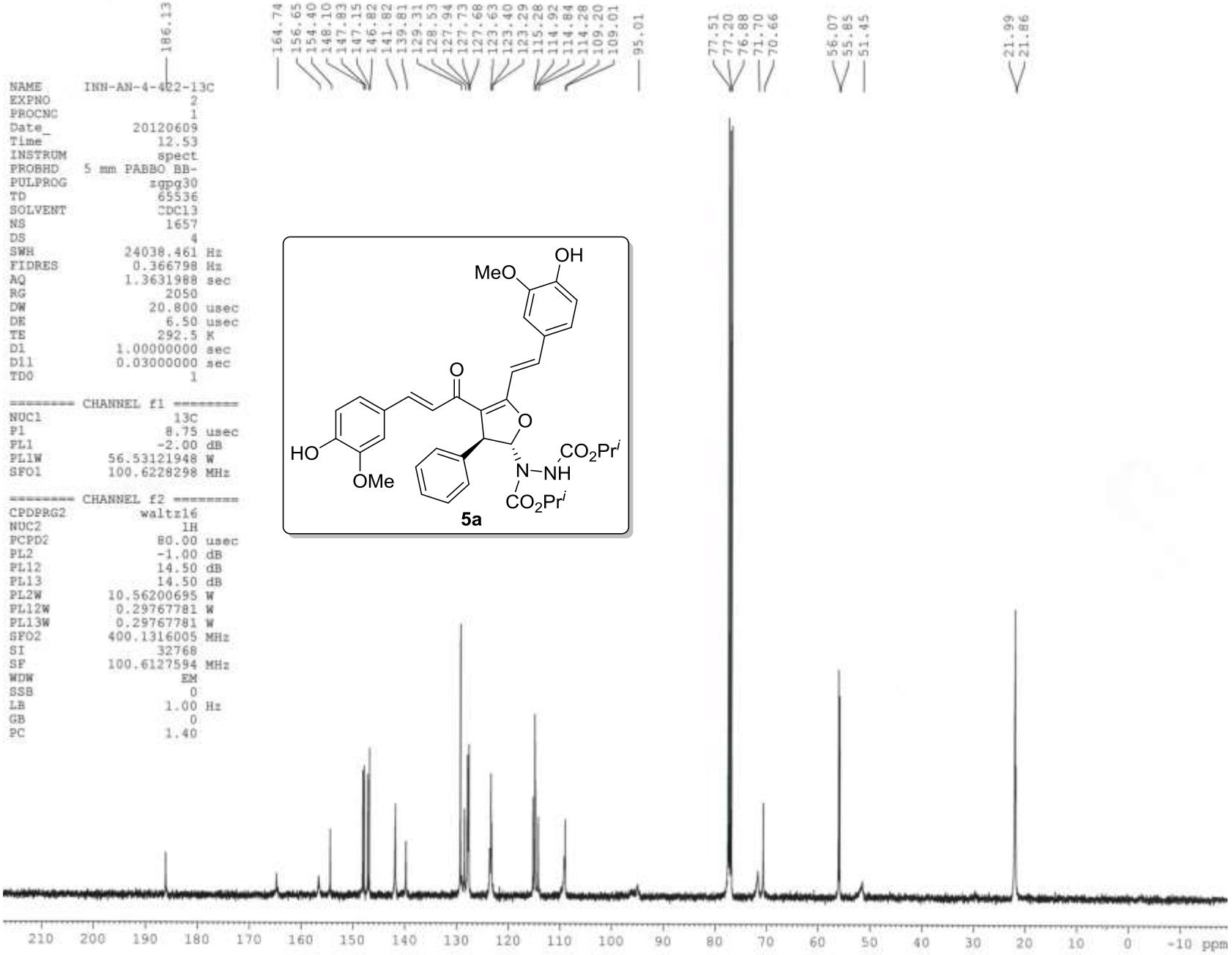

Fig S13. ^{13}C NMR Spectrum of 5a

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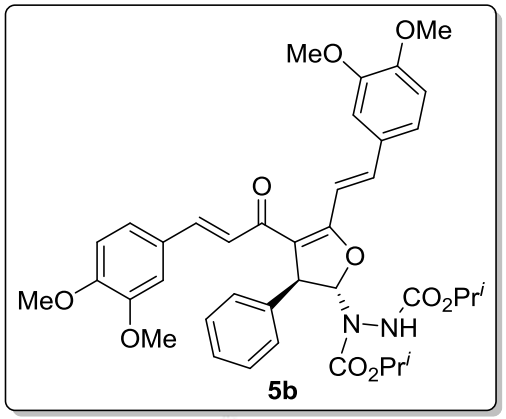
EXPNO                   17
PROCNO                   1
Date_             20121101
Time                 21.09
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH              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
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.50 usec
PL1                 -1.00 dB
PL1W          10.56200695 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300095 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```



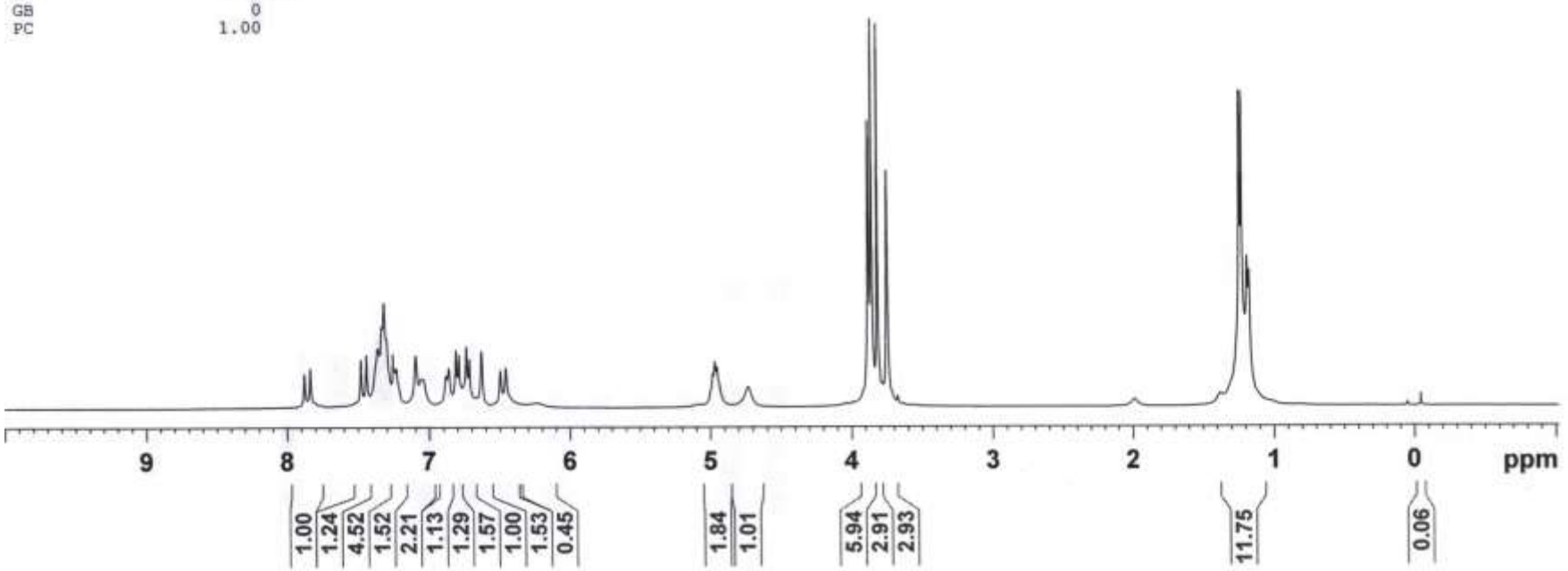


Fig S14. ^{1}H NMR Spectrum of 5b

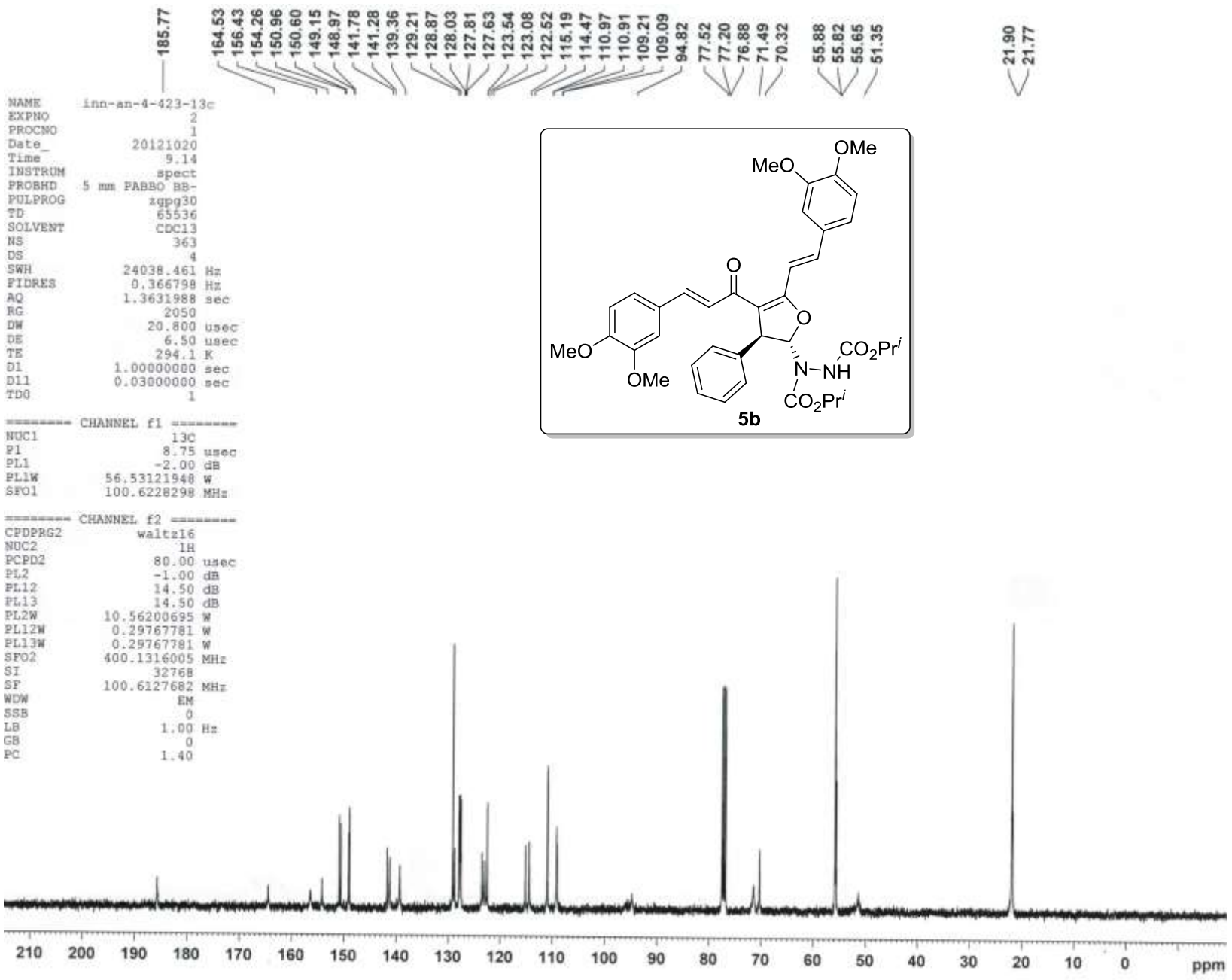


Fig S15. ^{13}C NMR Spectrum of 5b

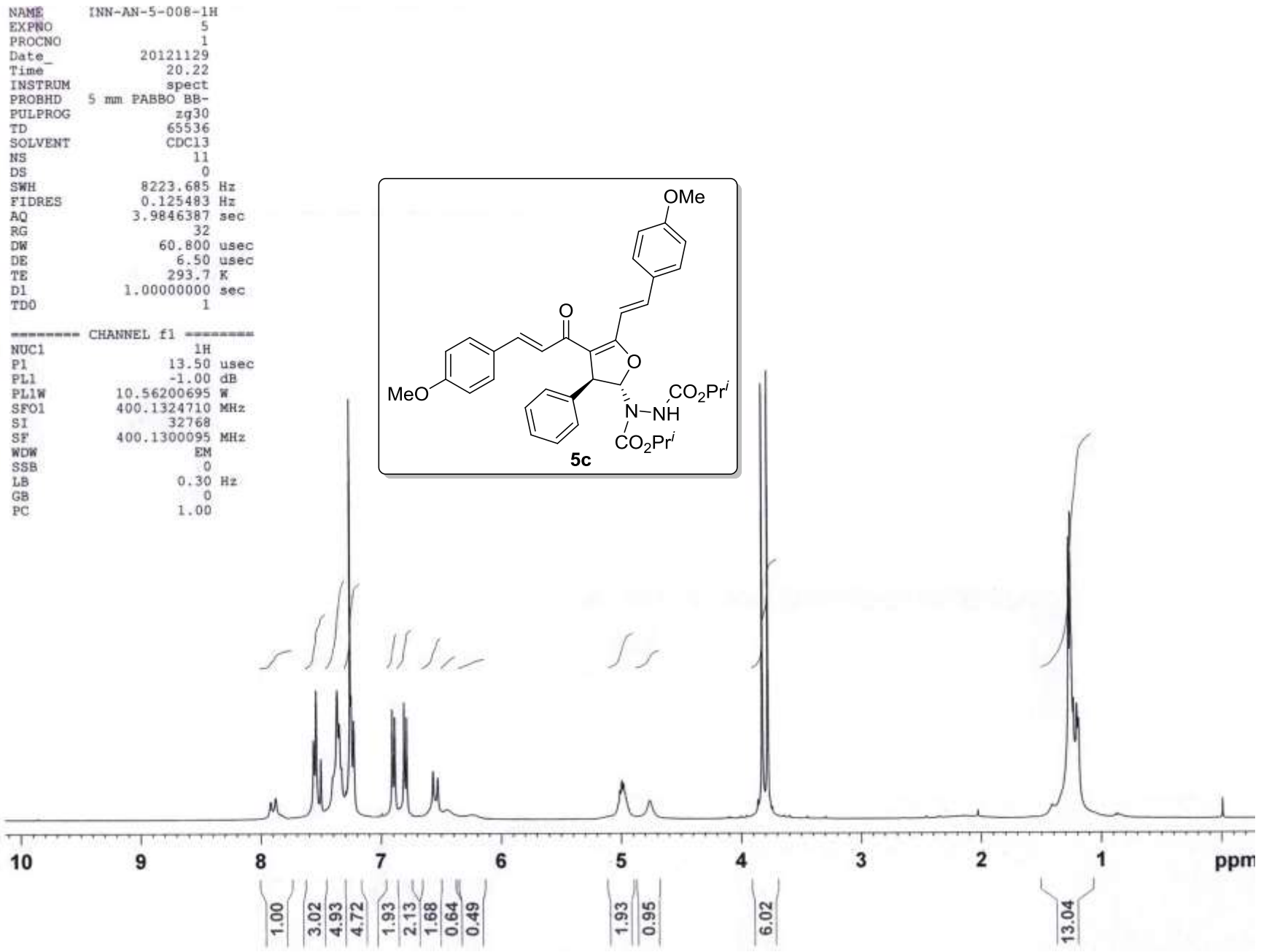


Fig S16. ^{1}H NMR Spectrum of 5c

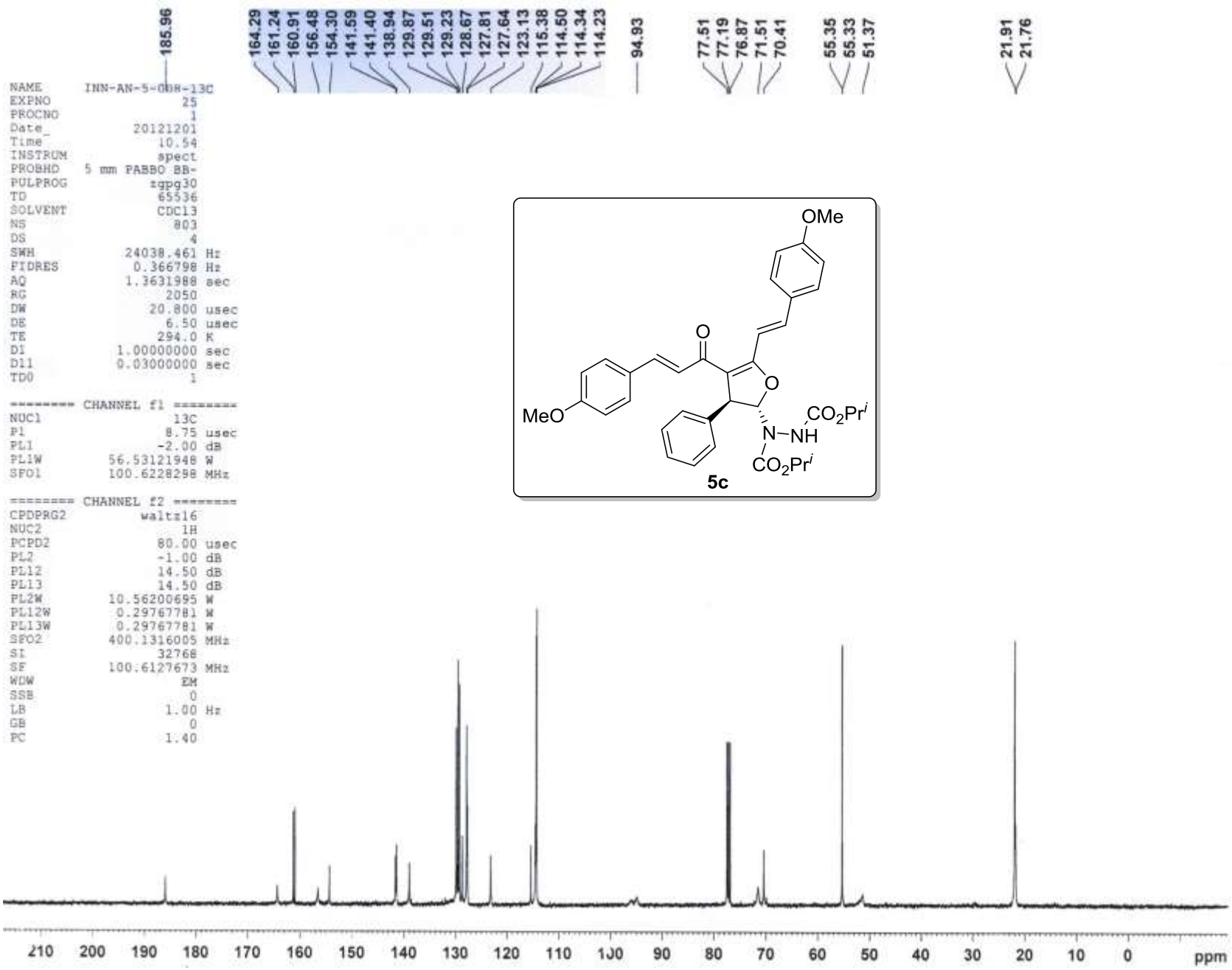


Fig S17. ^{13}C NMR Spectrum of 5c

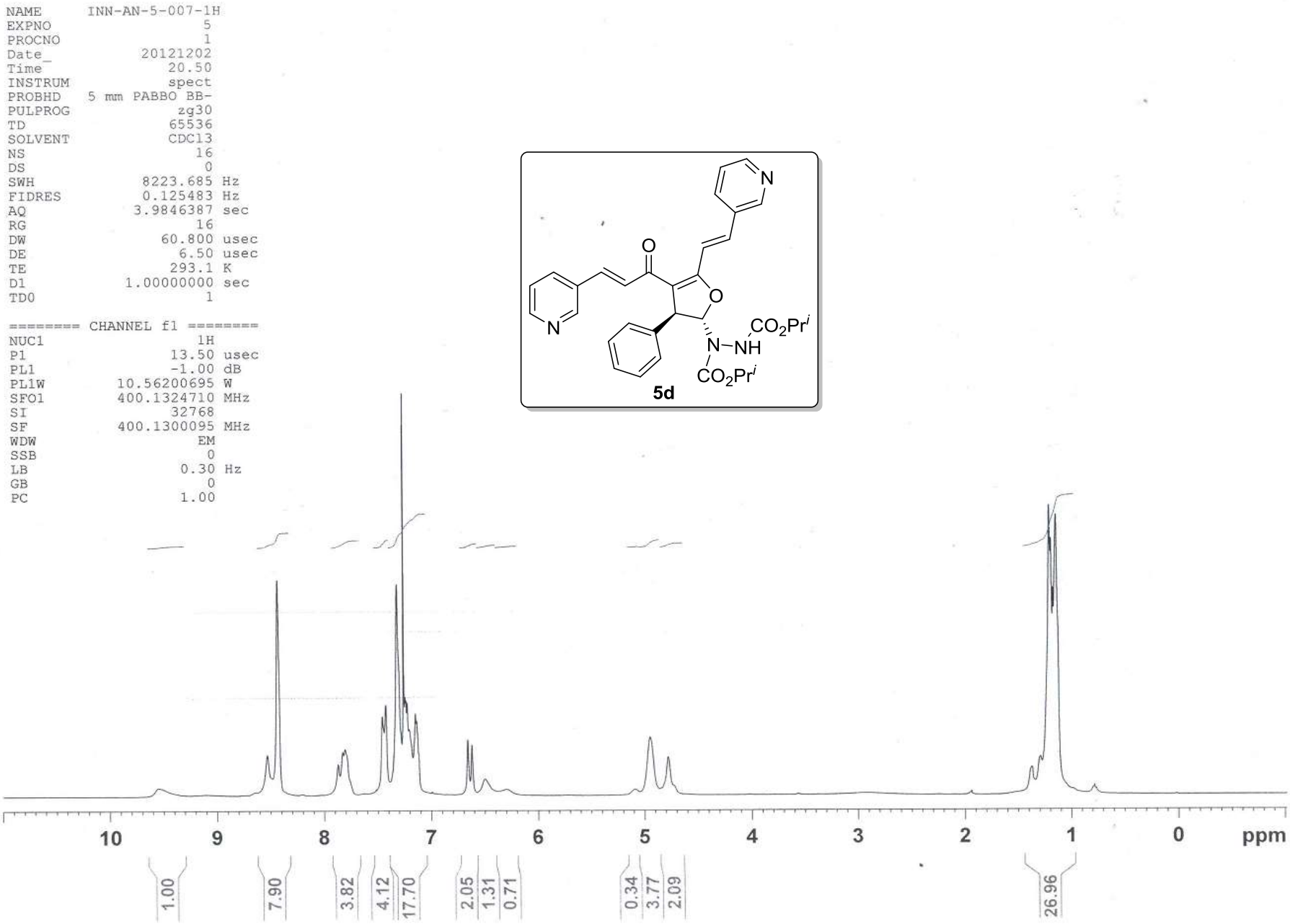


Fig S18. ^{1}H NMR Spectrum of 5d

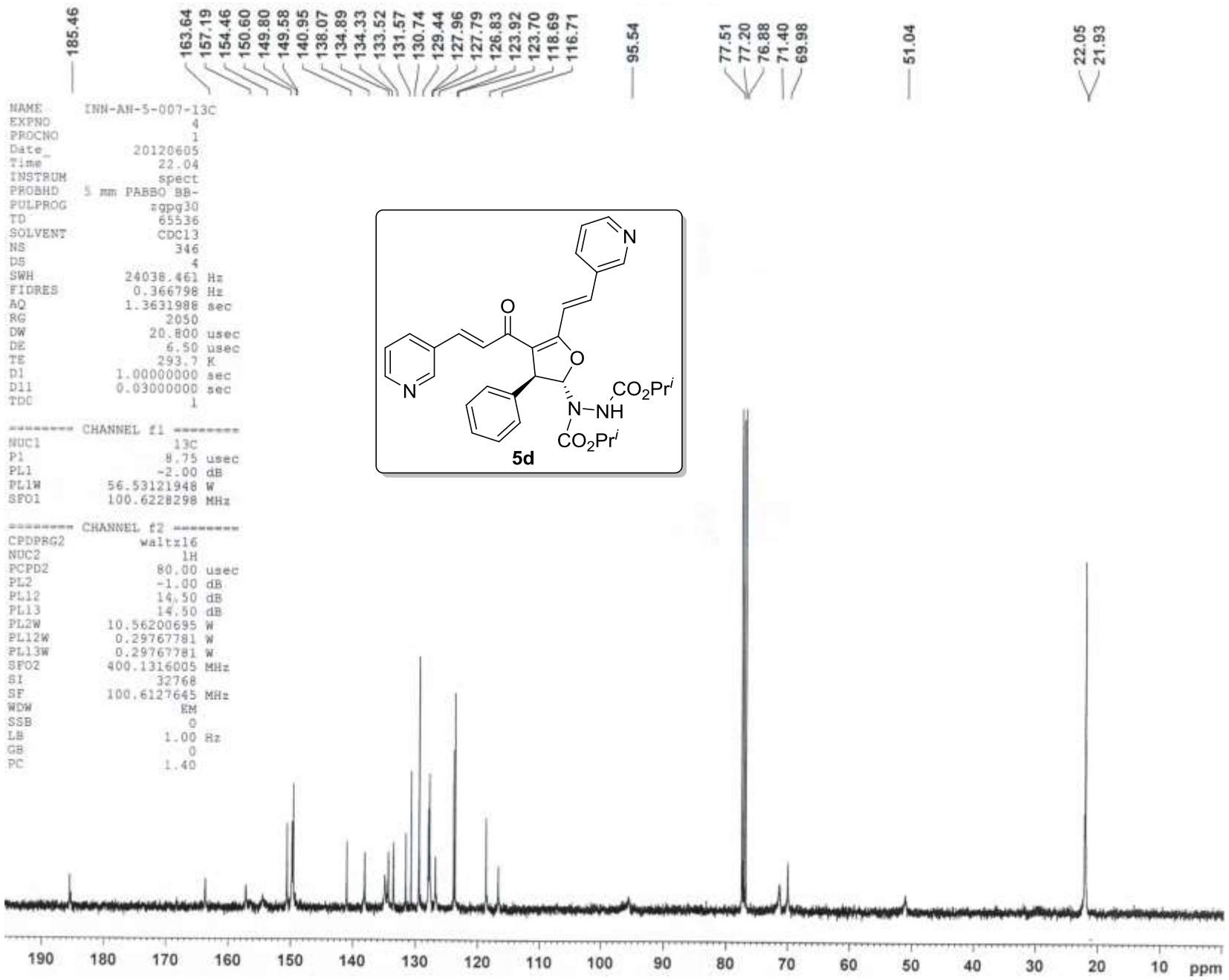


Fig S19. ^{13}C NMR Spectrum of 5d

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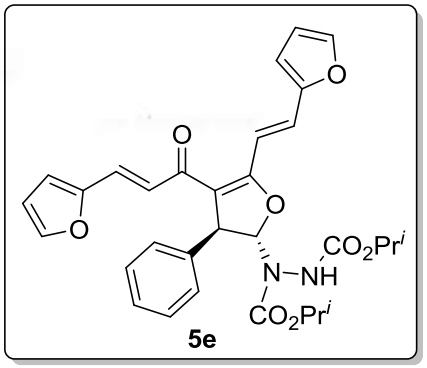
PROCNO	1
Date_	20121206
Time	20.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	32
DW	60.800 usec
DE	6.50 usec
TE	294.5 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	13.50 usec
PL1	-1.00 dB
PL1W	10.56200695 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



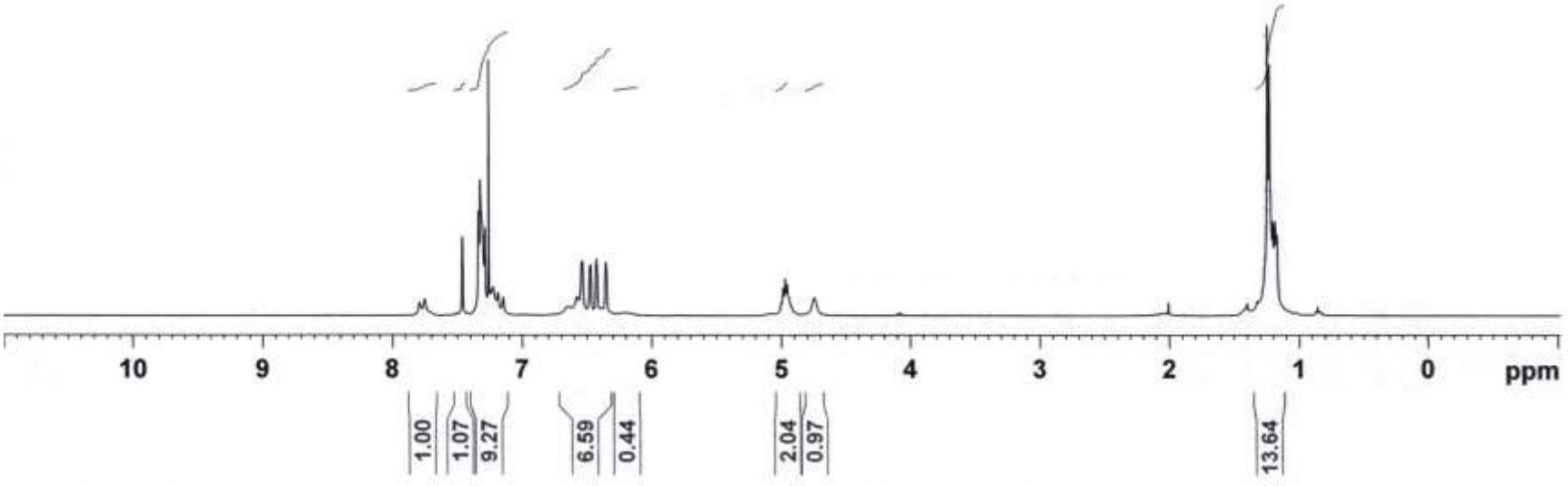


Fig S20. ^{1}H NMR Spectrum of 5e

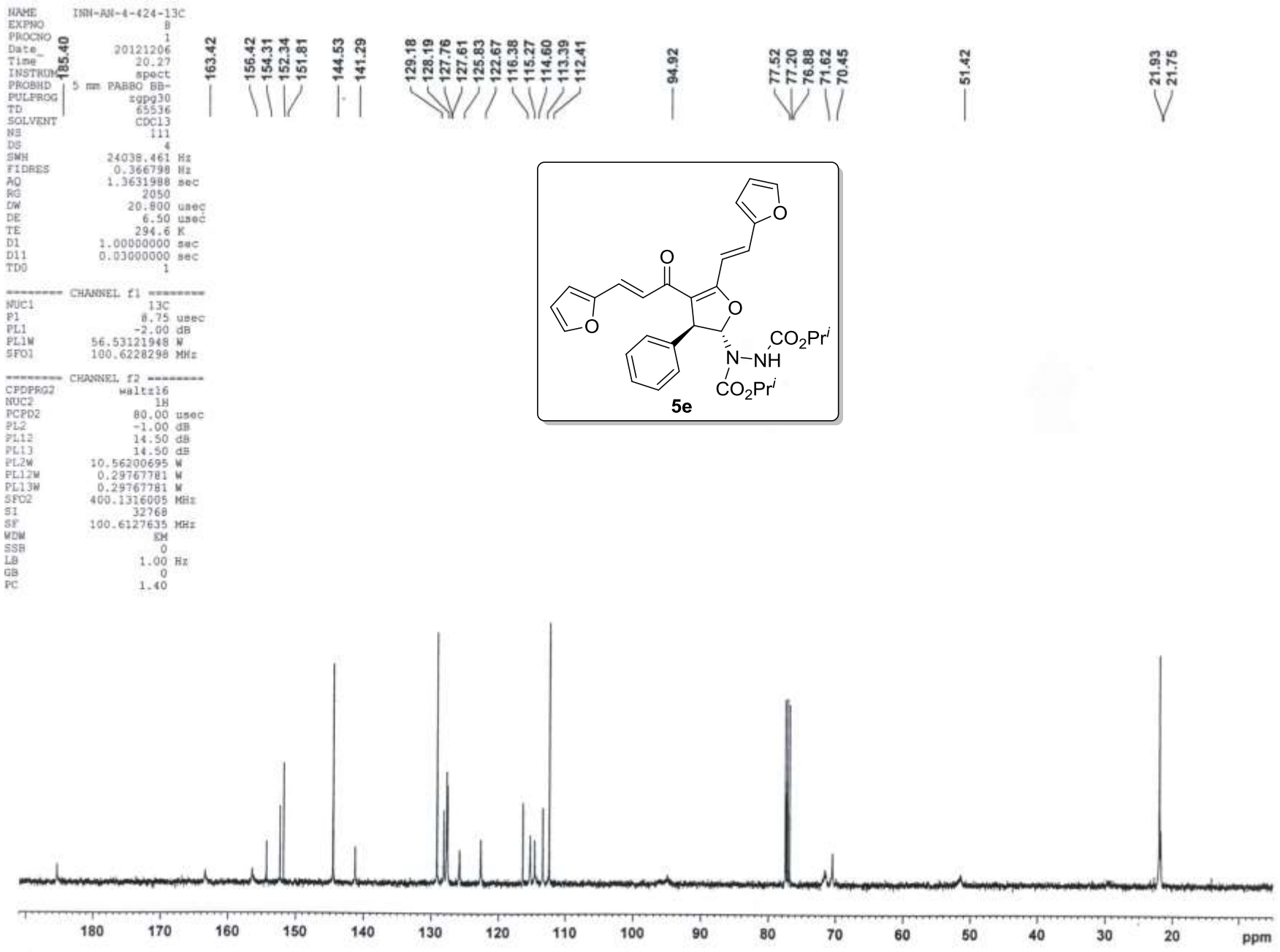


Fig S21. ^{13}C NMR Spectrum of 5e

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PROCNO                 1

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PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
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                295.4 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
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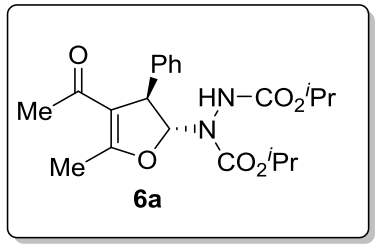

6a

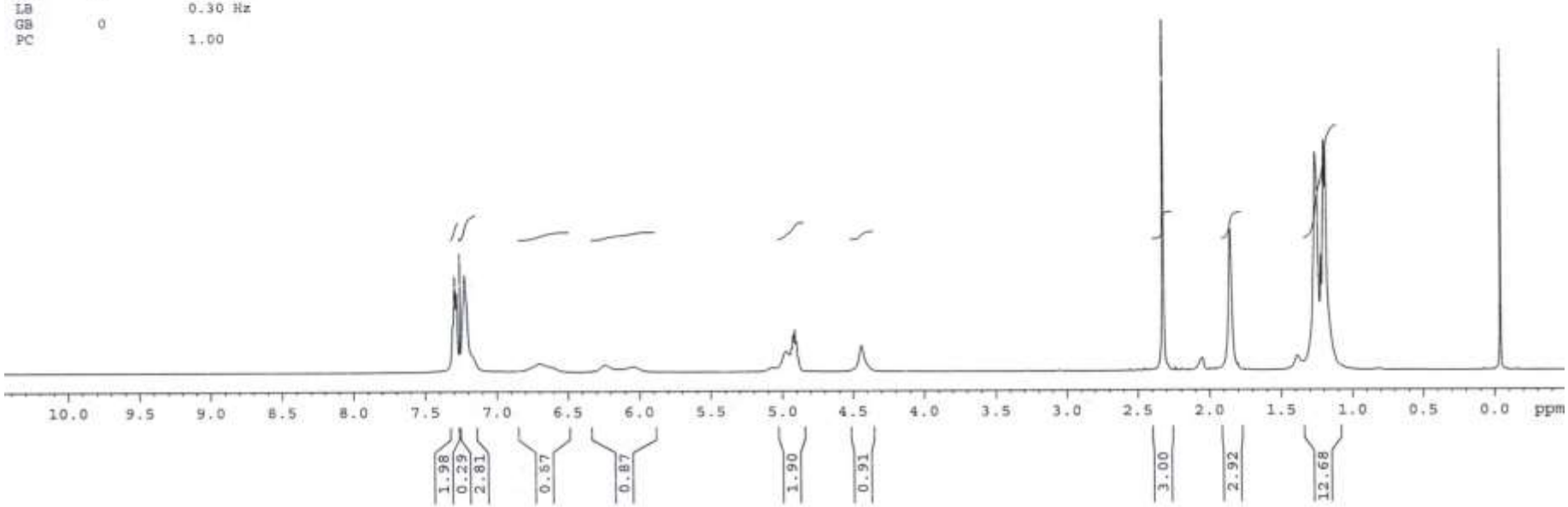

Fig S22. ^{13}H NMR Spectrum of 6a

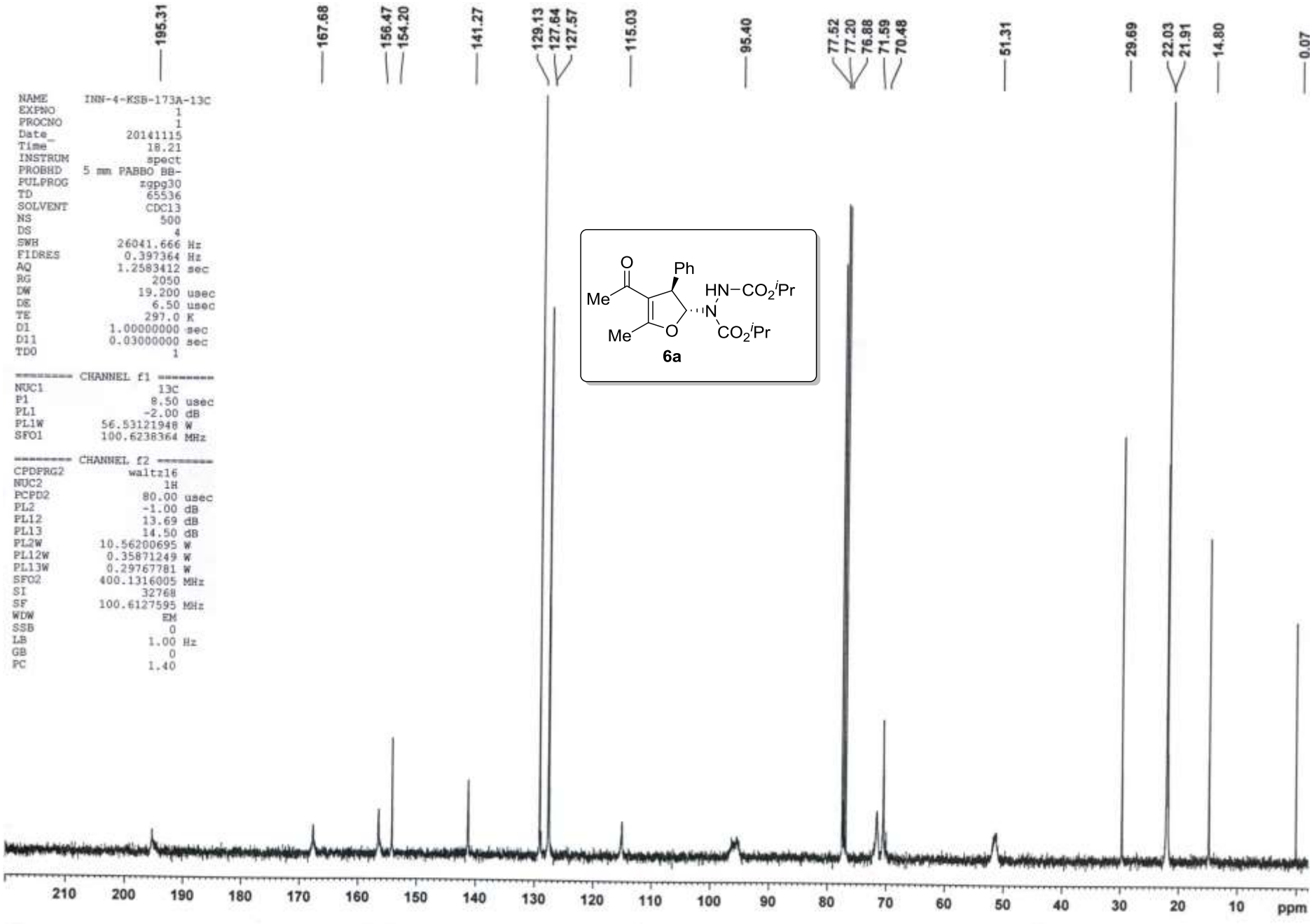

Fig S23. ^{13}C NMR Spectrum of 6a

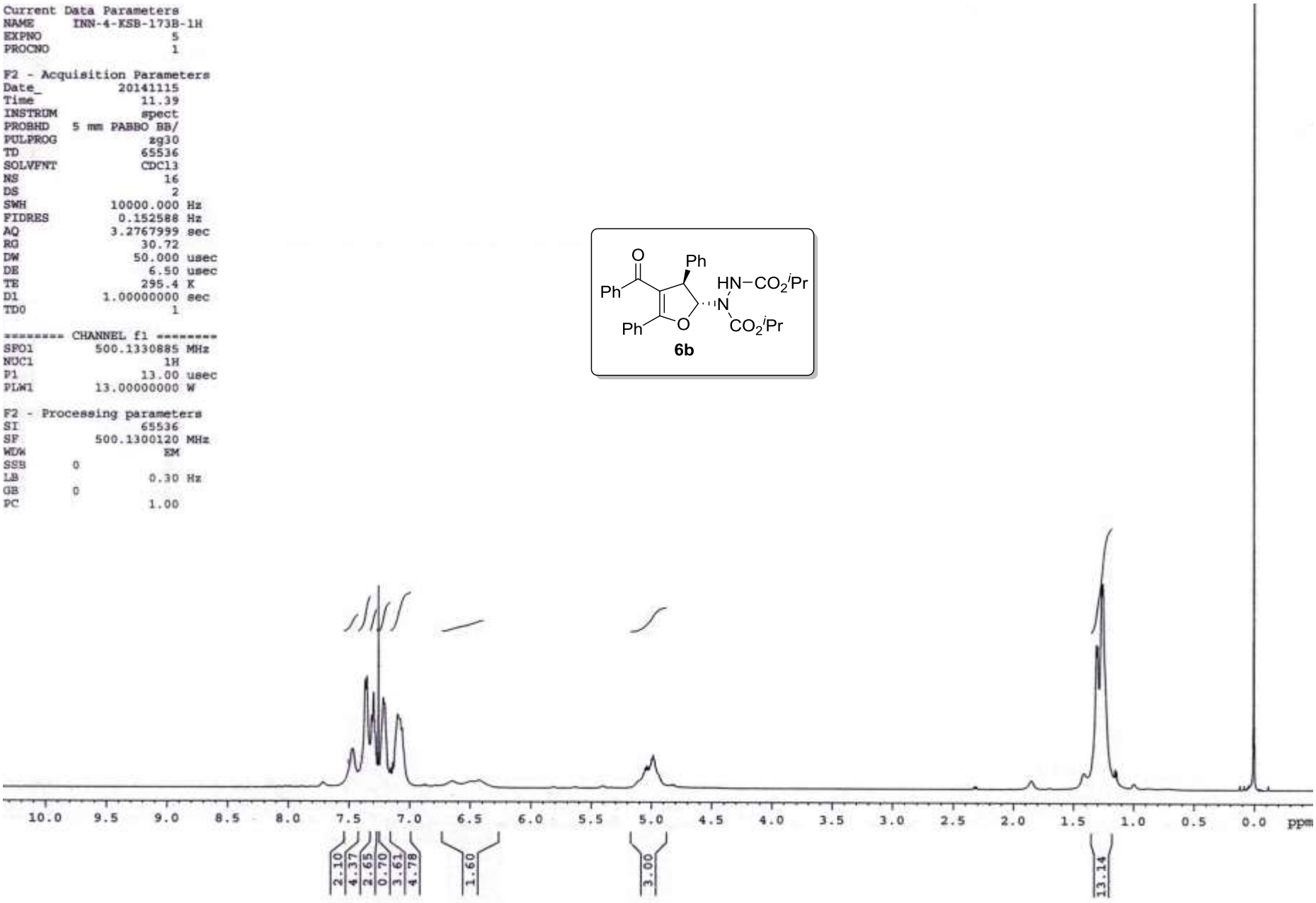

Fig S24. ^{1}H NMR Spectrum of 6b

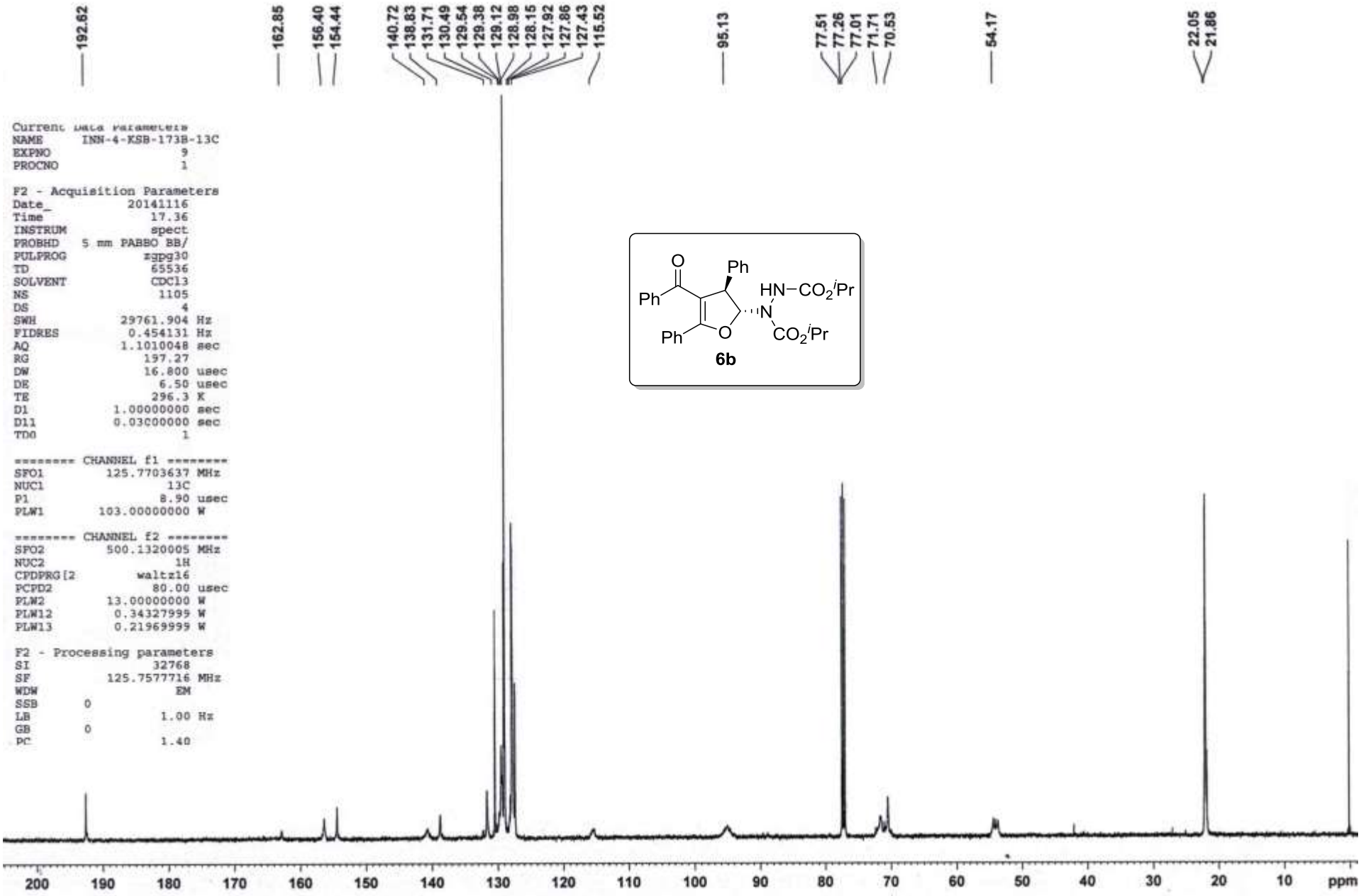

Fig S25. ^{13}C NMR Spectrum of 6b

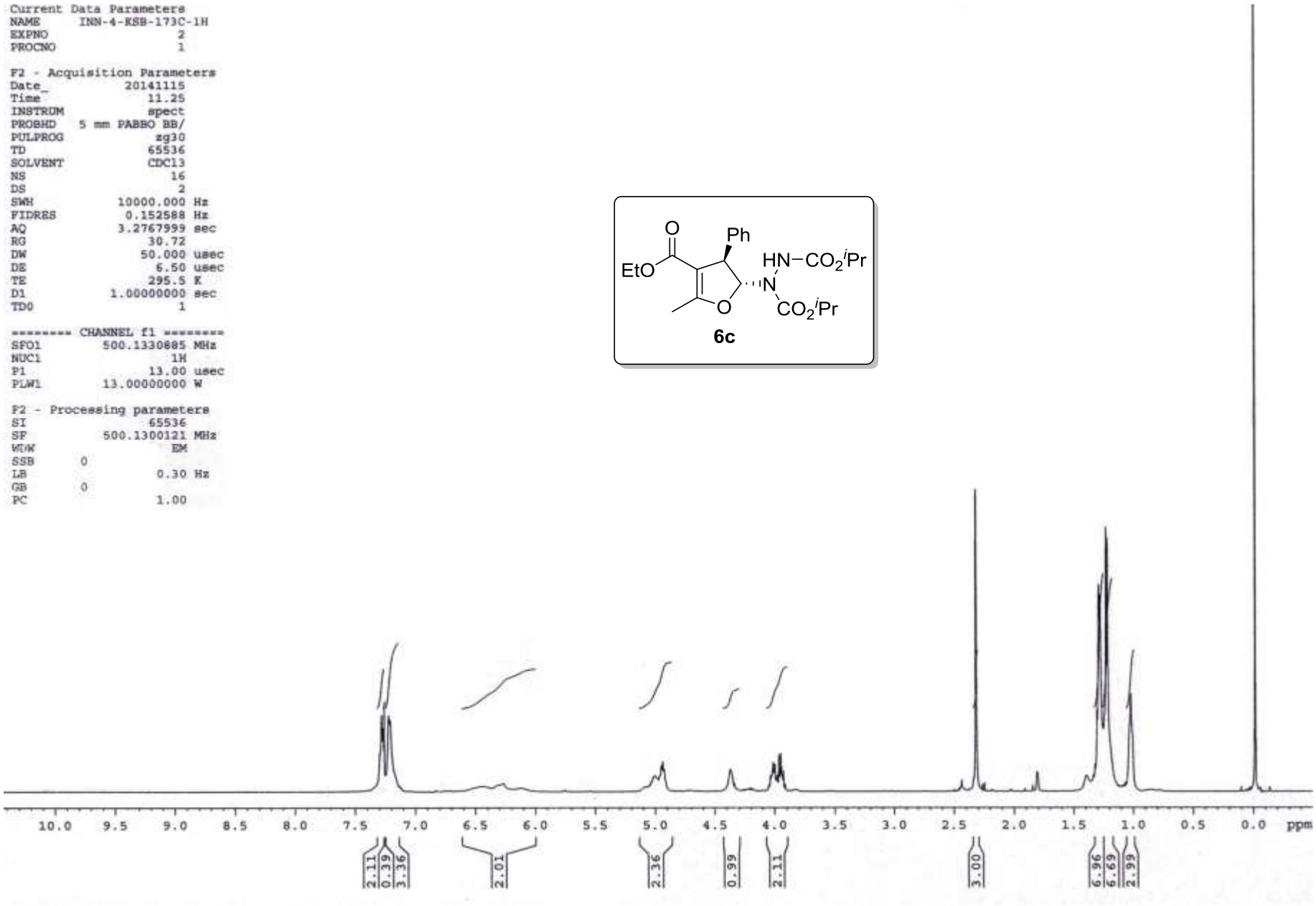

Fig S26. ^{1}H NMR Spectrum of 6c

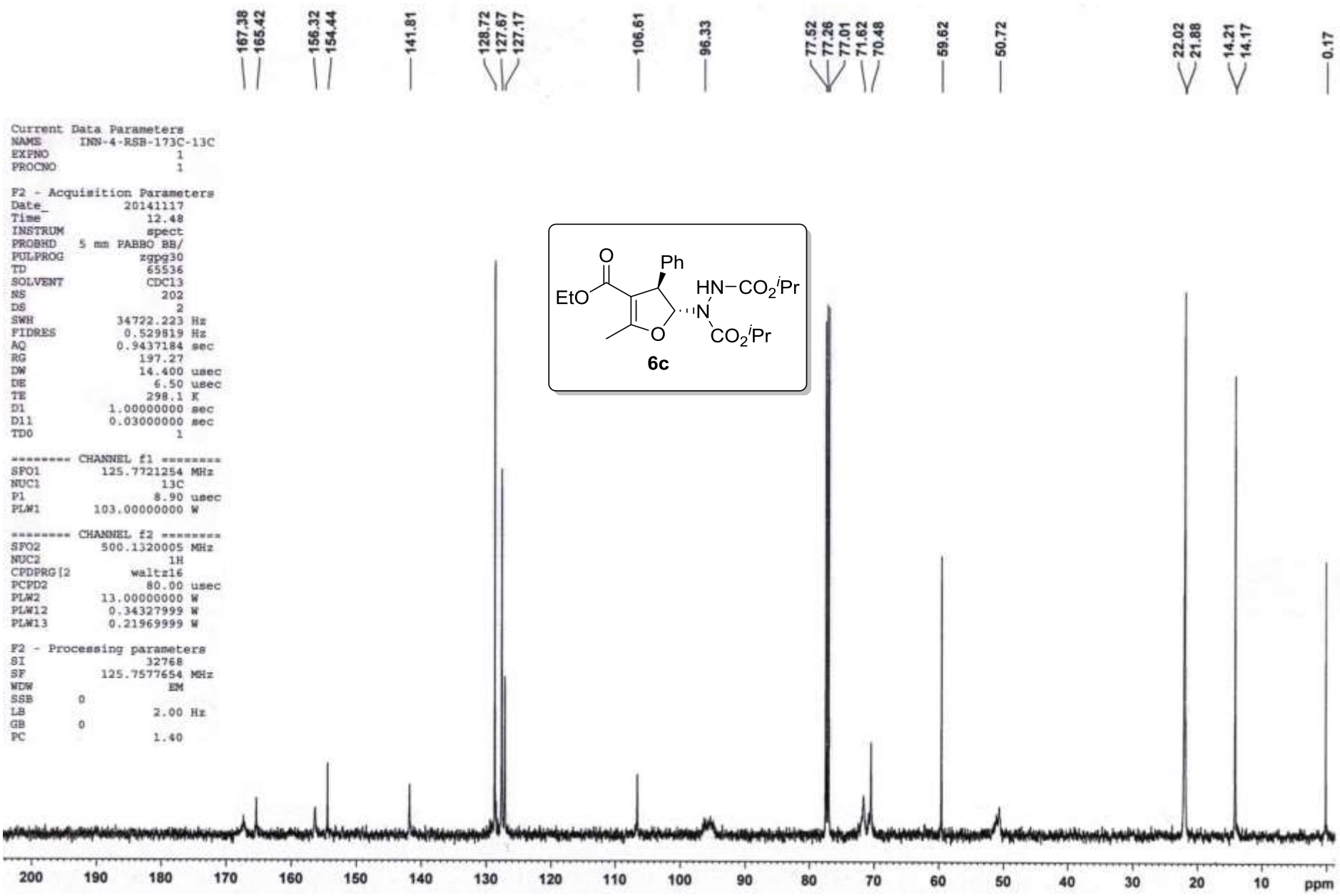

Fig S27. ^{13}C NMR Spectrum of 6c

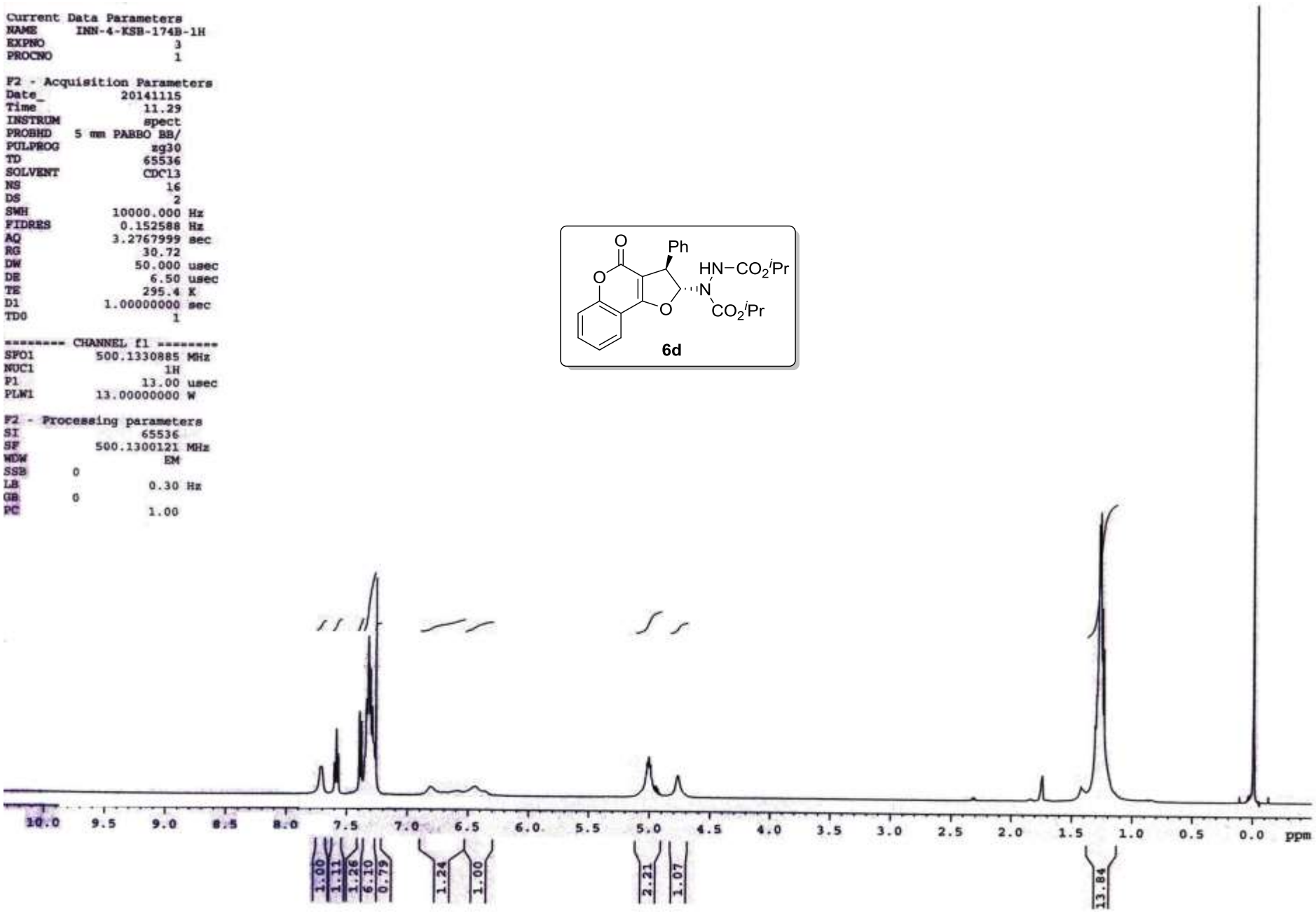

Fig S28. ^{1}H NMR Spectrum of 6d

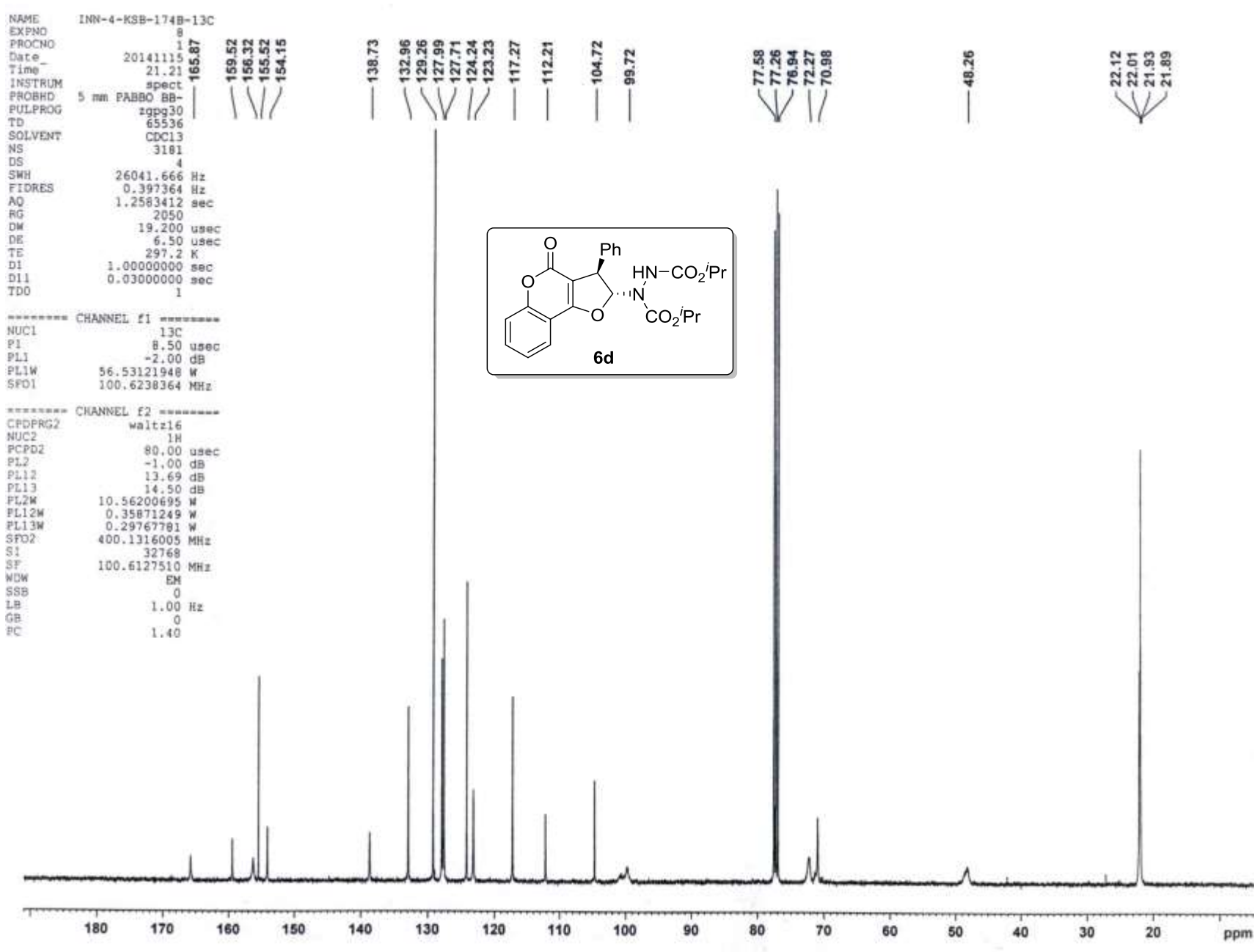

Fig S29. ^{13}C NMR Spectrum of 6d

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PROCNO                    1
Date_              20140724
Time                  18.10
INSTRUM               spect
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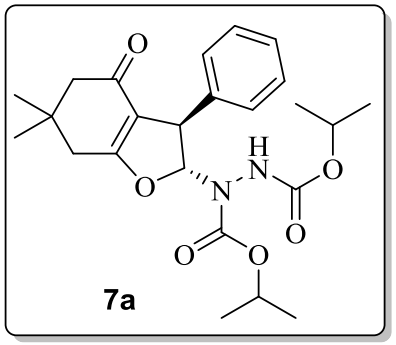
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       13
DS                        0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       32
DW                    60.800 usec
DE                     6.50 usec
TE                    296.4 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
SI                     32768
SF                400.1300103 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



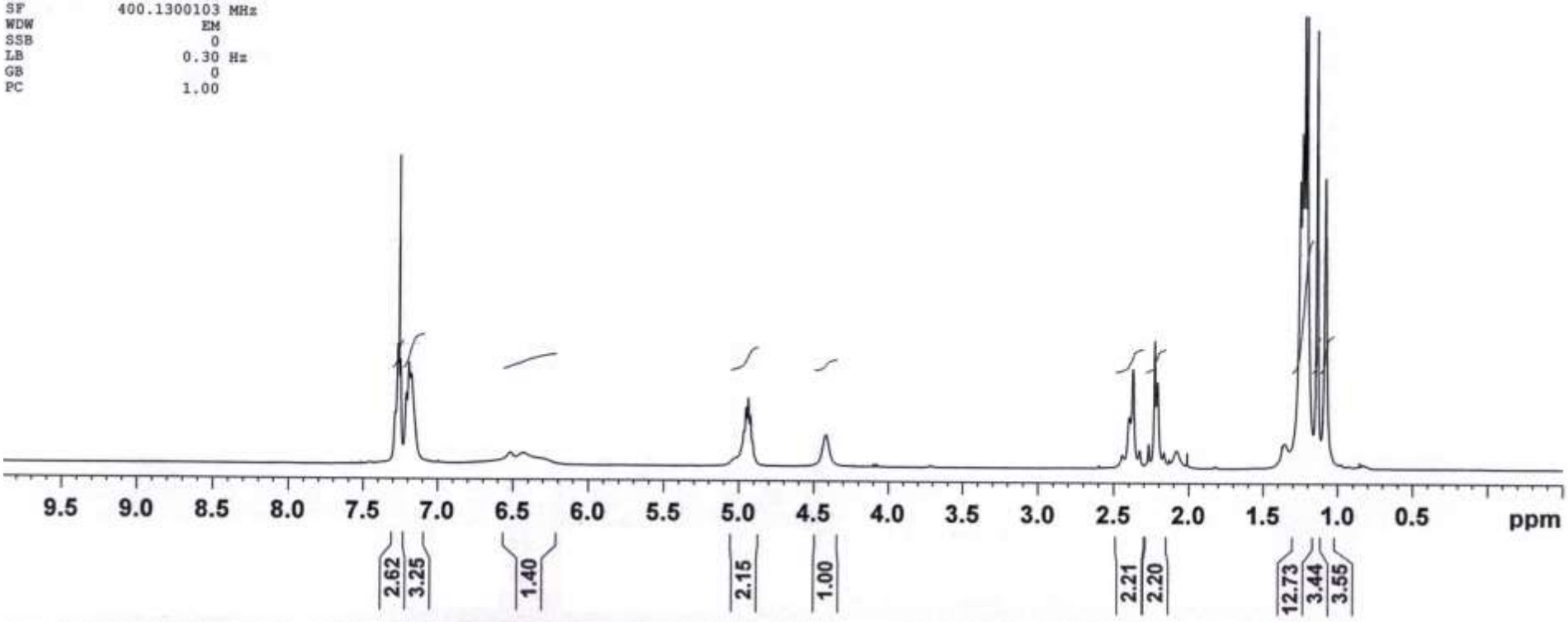


Fig S30. ^{1}H NMR Spectrum of 7a

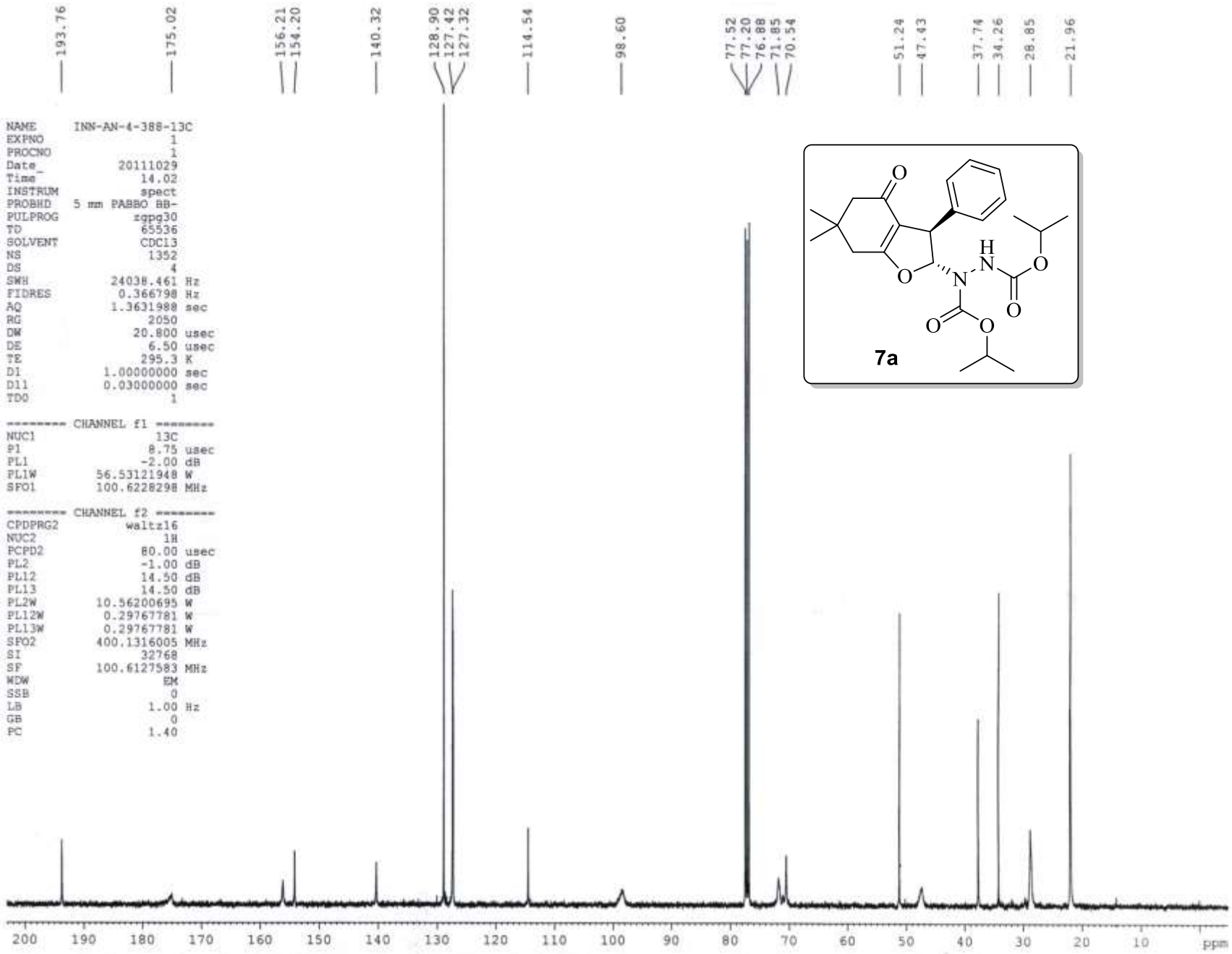
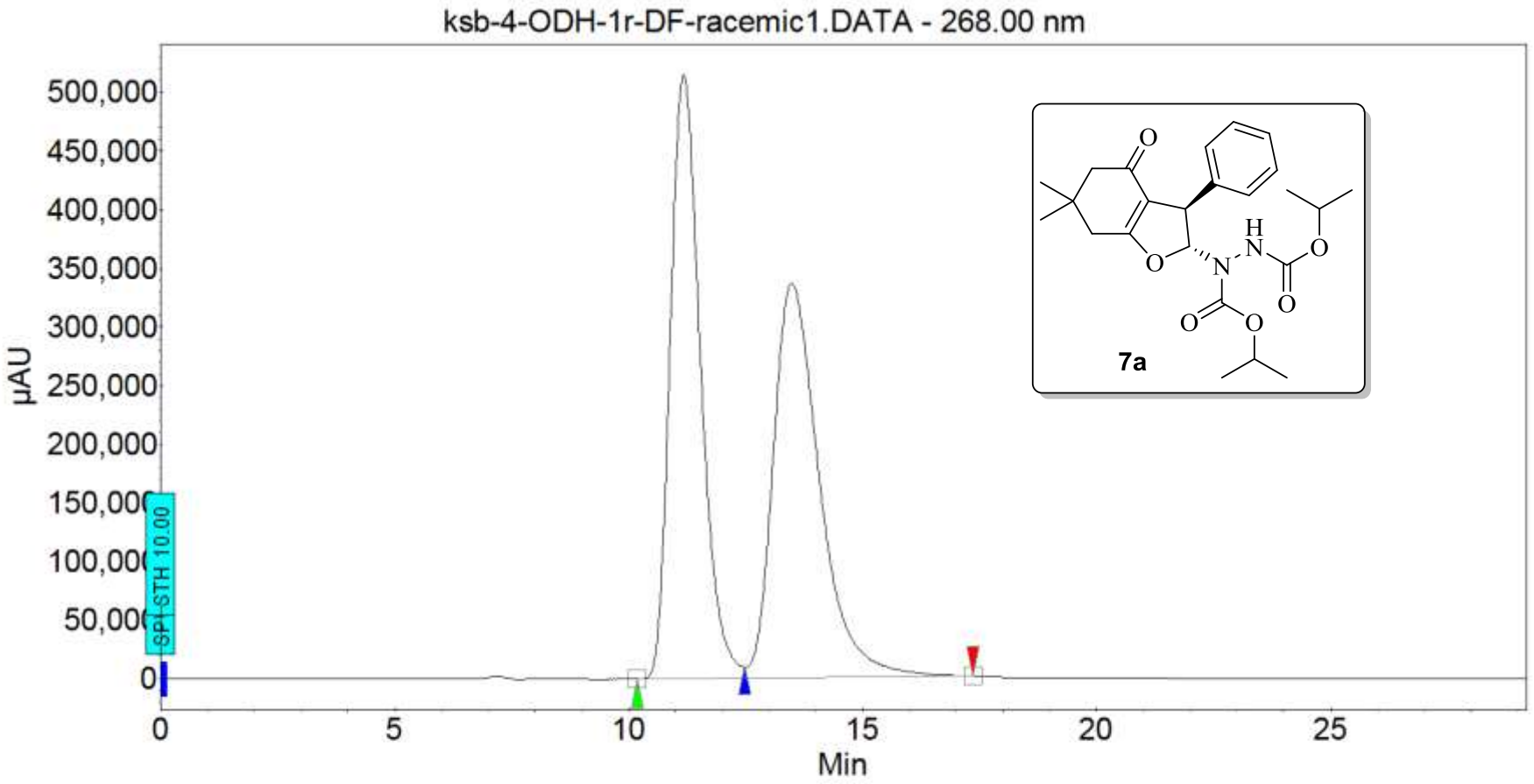


Fig S31. ^{13}C NMR Spectrum of 7a



Peak results :

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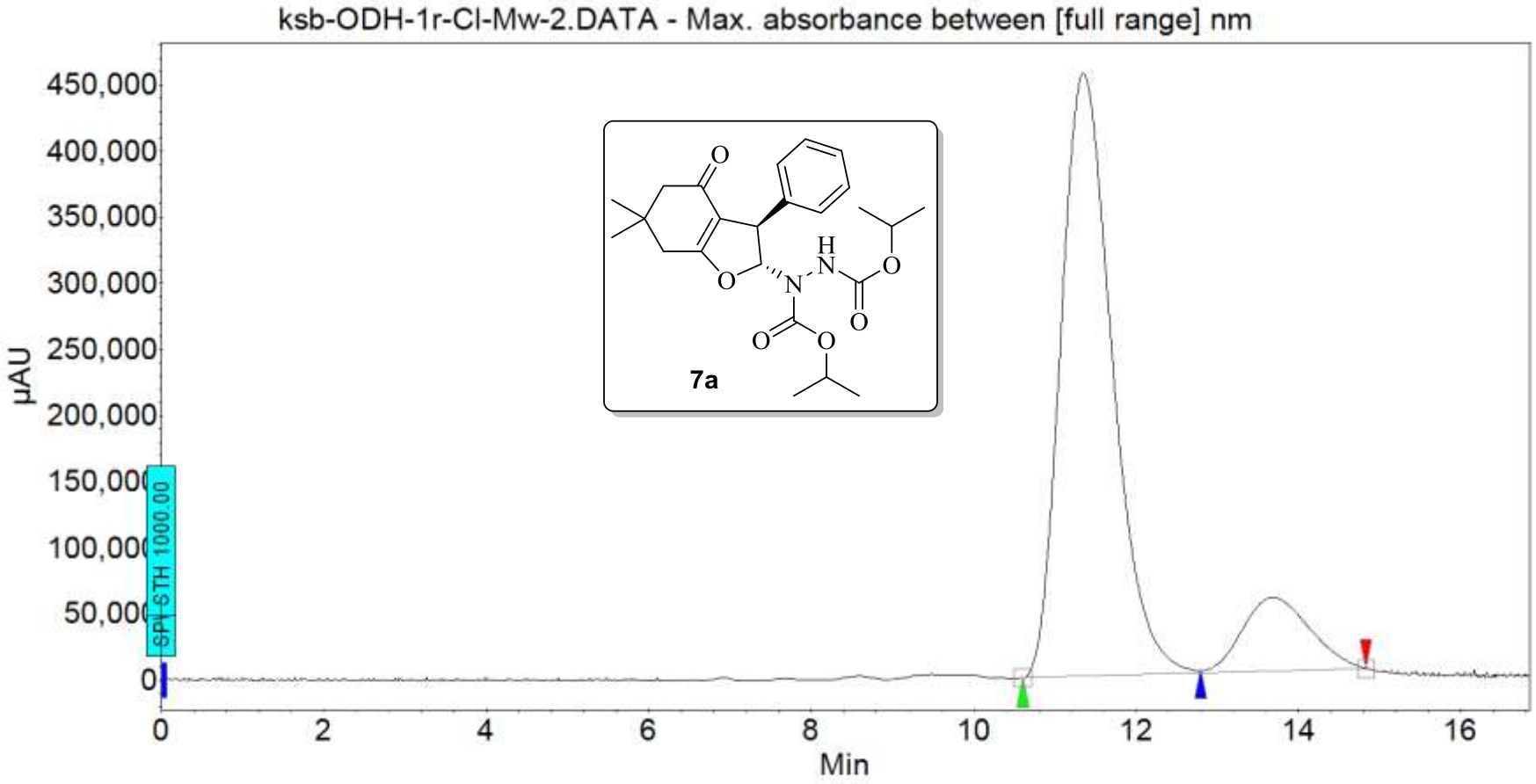
1	UNKNOWN	11.173	50.05	514922.9	386153.6	50.052
2	UNKNOWN	13.479	49.95	336484.9	385350.6	49.948
Total			100.00	851407.9	771504.2	100.000

Fig S32. HPLC Profile of Racemic 7a



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
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2	UNKNOWN	13.679	13.71	55389.6	52939.7	13.710
Total			100.00	511018.8	386136.4	100.000

Fig S33. HPLC Profile of Enantioenriched 7a

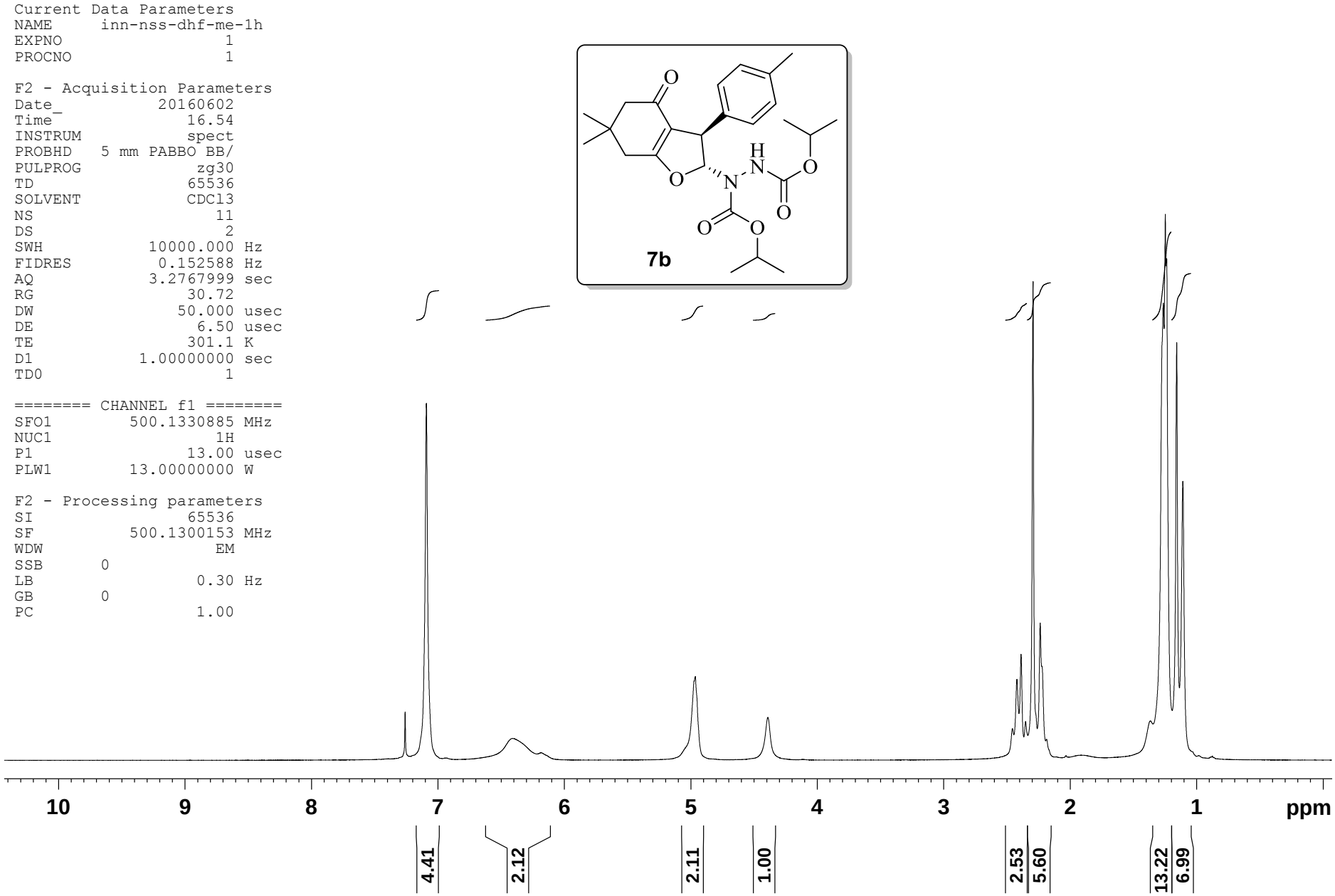


Fig S34. ^{1}H NMR Spectrum of 7b

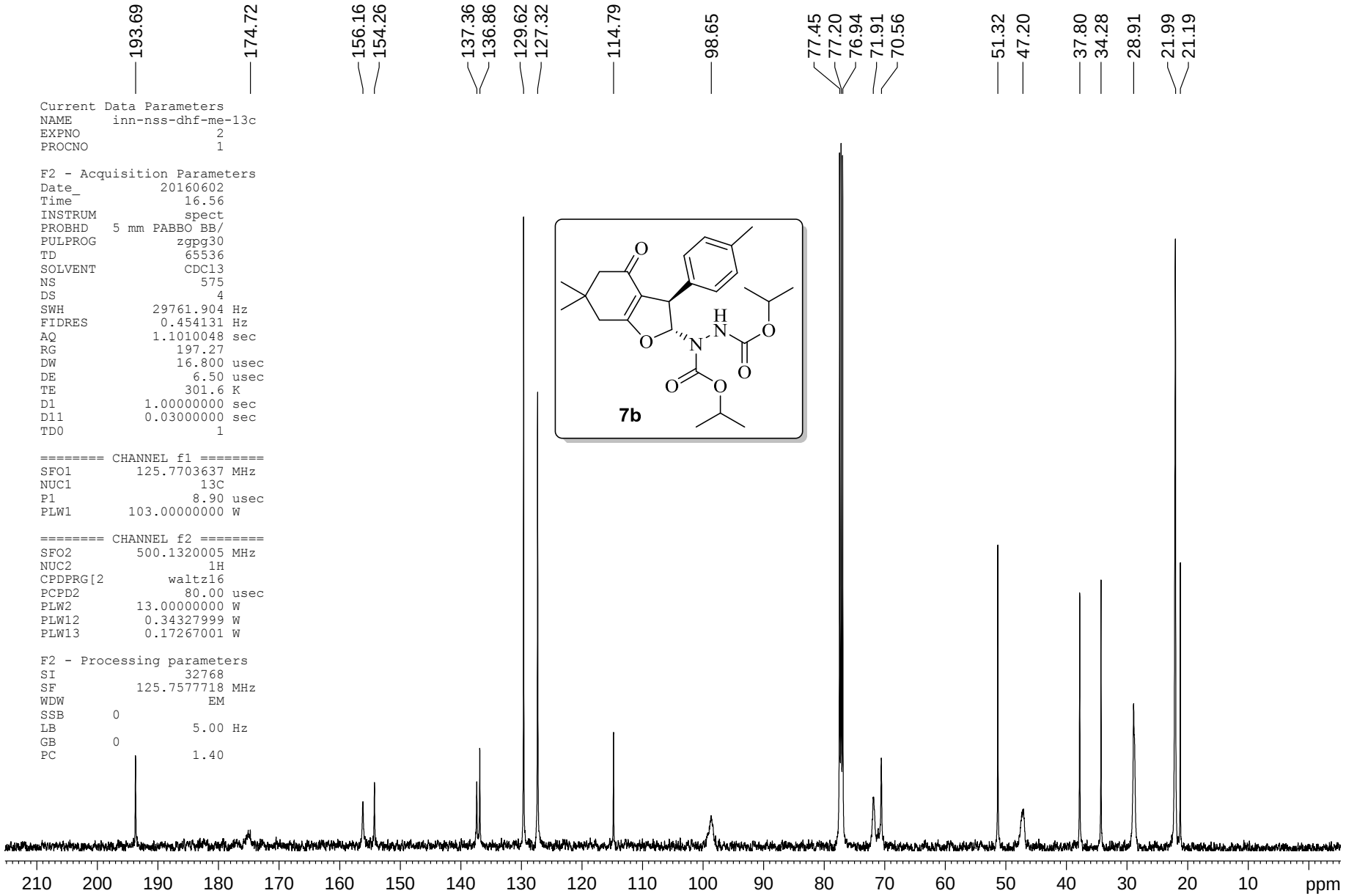
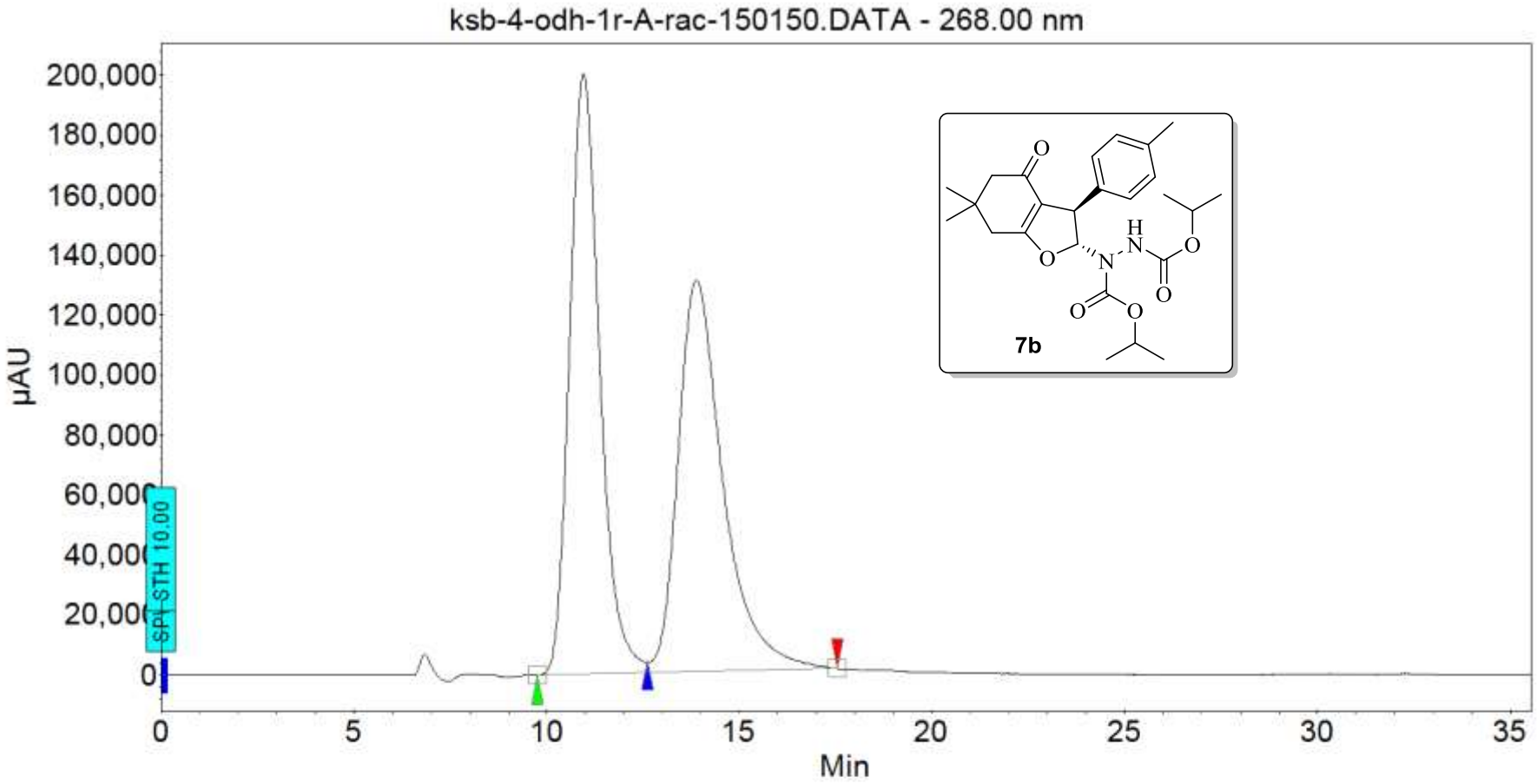


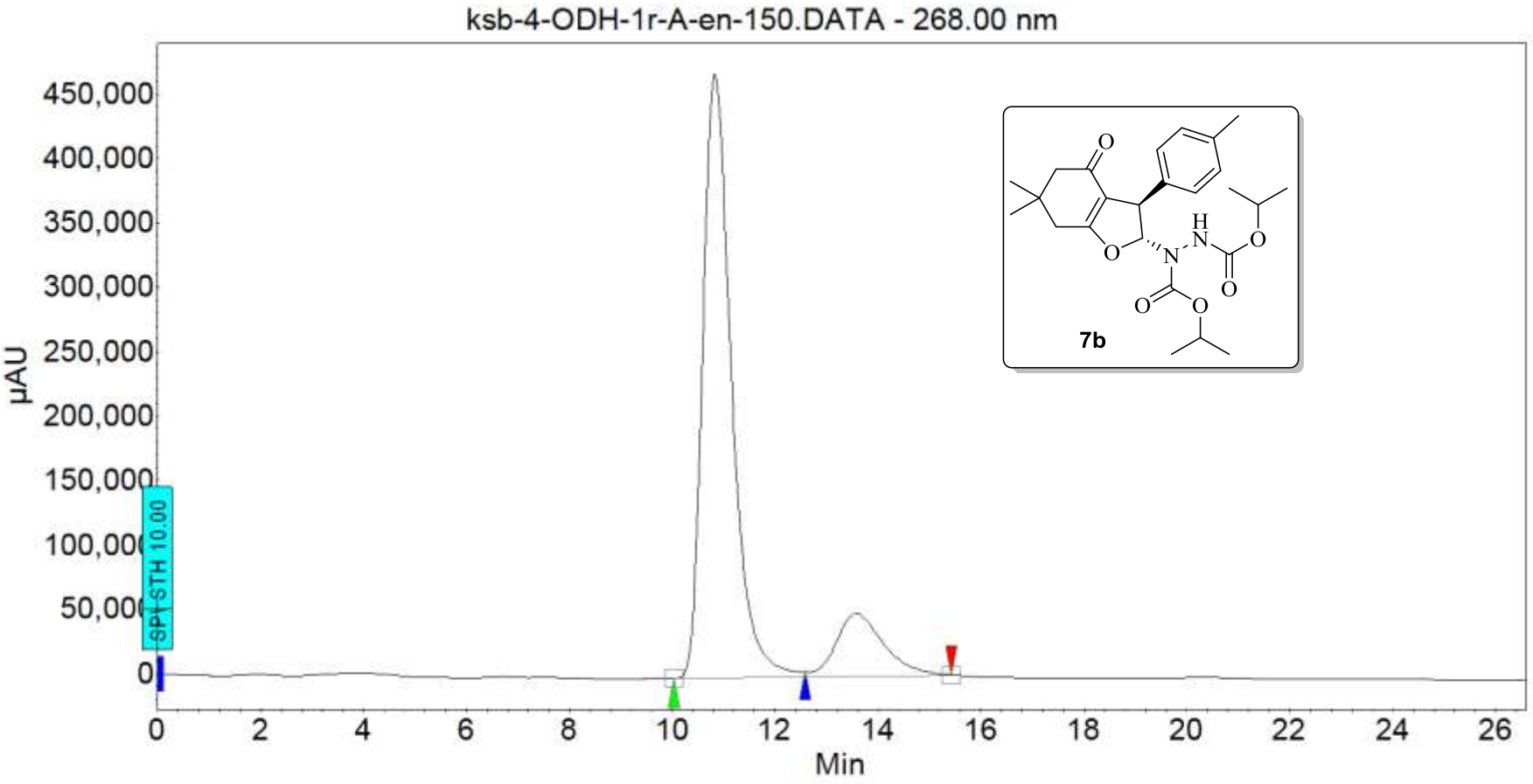
Fig S35. ^{13}C NMR Spectrum of 7b



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	10.946	50.61	199986.5	180830.3	50.607
2	UNKNOWN	13.879	49.39	130355.7	176492.8	49.393
Total			100.00	330342.2	357323.1	100.000

Fig S36. HPLC Profile of Racemic 7b



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	10.826	85.33	468962.5	301858.0	85.327
2	UNKNOWN	13.572	14.67	48579.8	51906.6	14.673
Total			100.00	517542.2	353764.6	100.000

Fig S37. HPLC Profile of Enantioenriched 7b

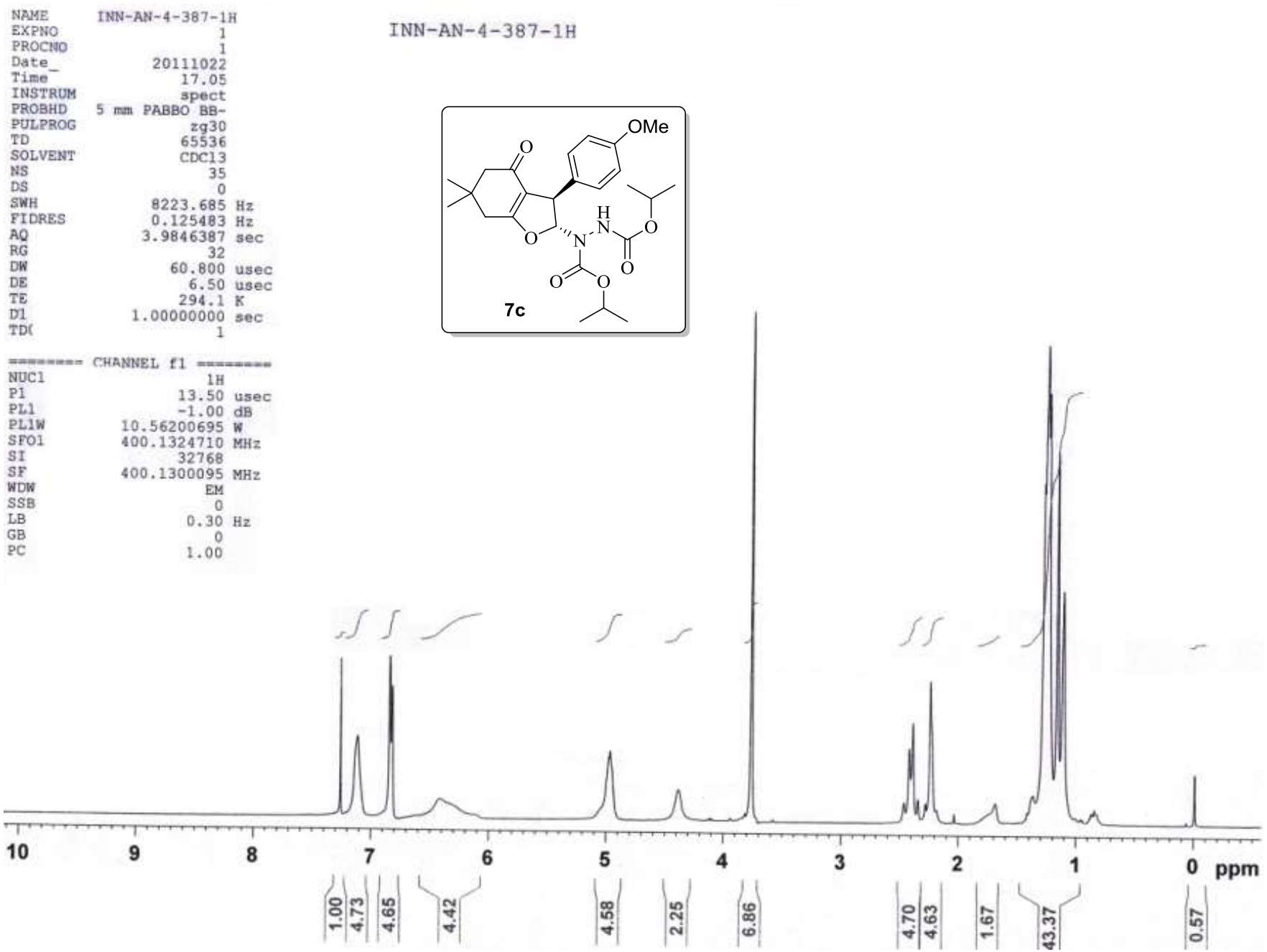


Fig S38. ^{1}H NMR Spectrum of 7c

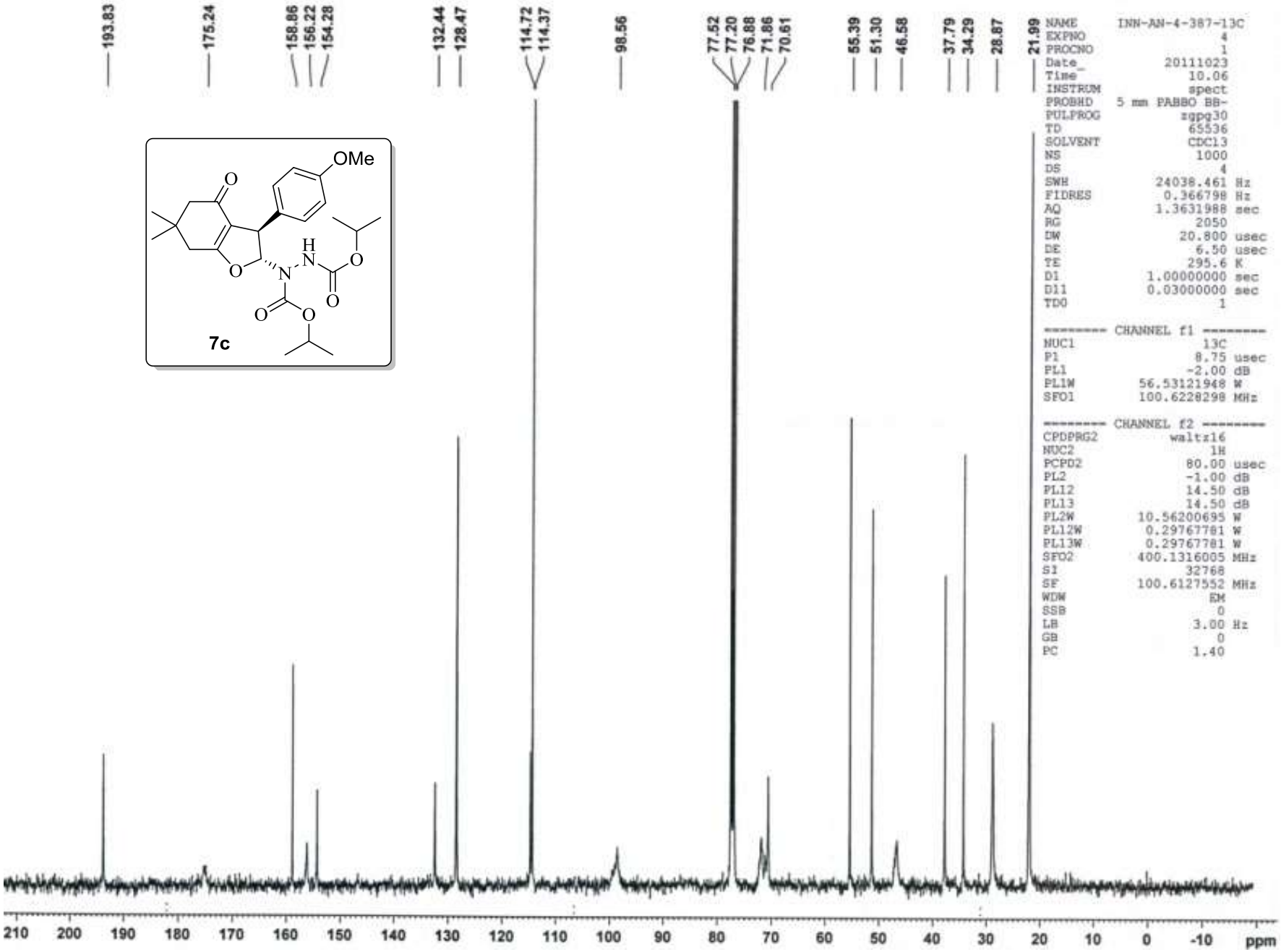
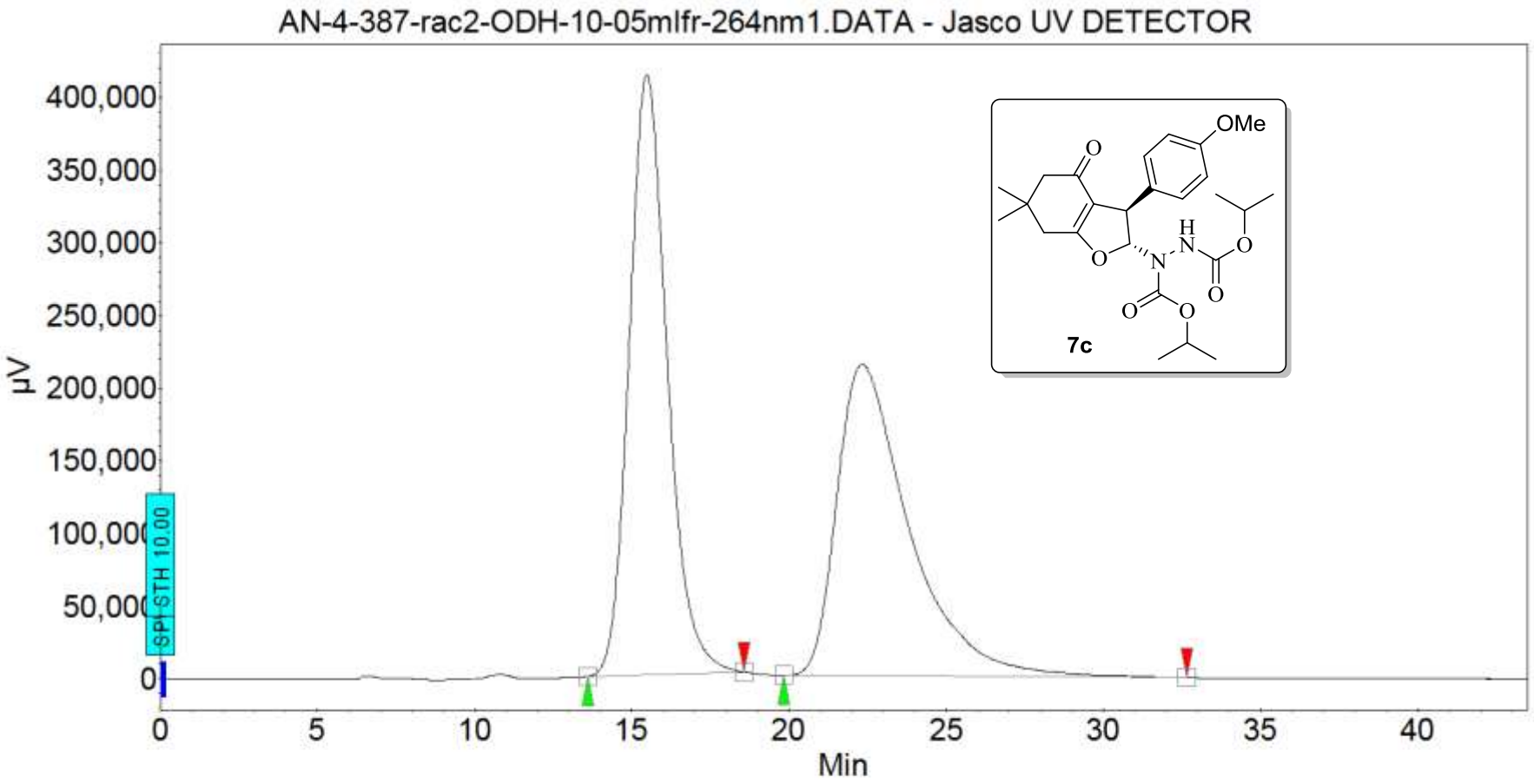


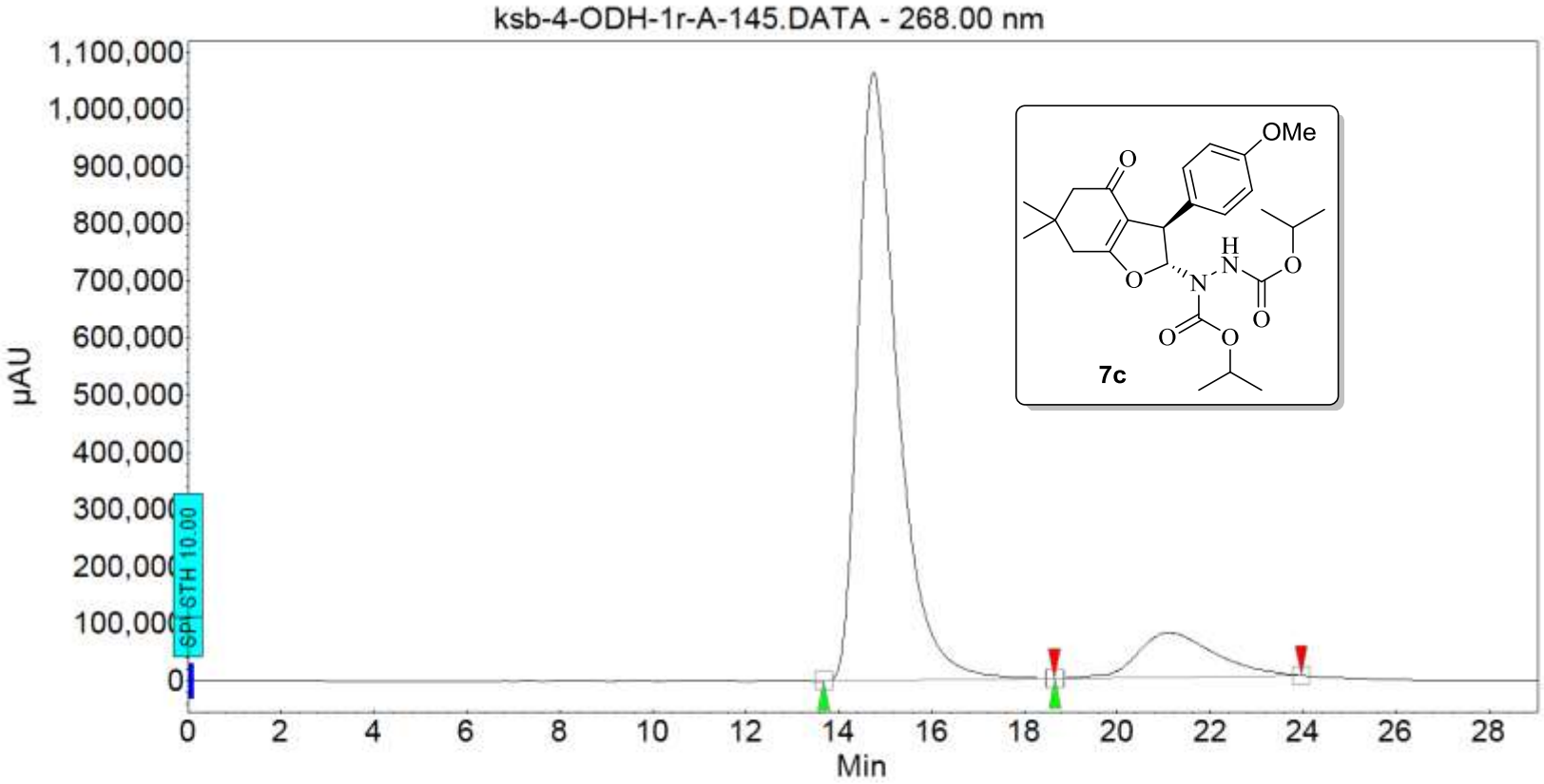
Fig S39. ^{13}C NMR Spectrum of 7c



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	15.467	50.40	412821.1	580592.6	50.398
2	UNKNOWN	22.325	49.60	213996.2	571414.4	49.602
Total			100.00	626817.3	1152007.1	100.000

Fig S40. HPLC Profile of Racemic 7c



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	14.746	87.45	1065035.7	1045245.6	87.451
2	UNKNOWN	21.092	12.55	78096.8	149991.5	12.549
Total			100.00	1143132.5	1195237.1	100.000

Fig S41. HPLC Profile of Enantioenriched 7c

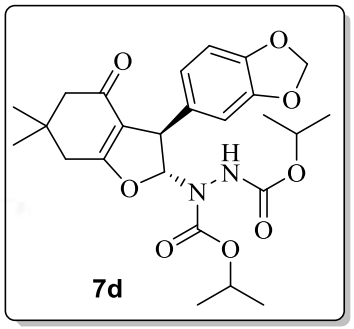
INN-4-KSB-116B-1H

```
Current Data Parameters
NAME      INN-4-KSB-116B-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20131118
Time               19.18
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    11
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ             3.2767999 sec
RG                 30.72
DW                 50.000 usec
DE                  6.50 usec
TE                 295.8 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            500.1300130 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



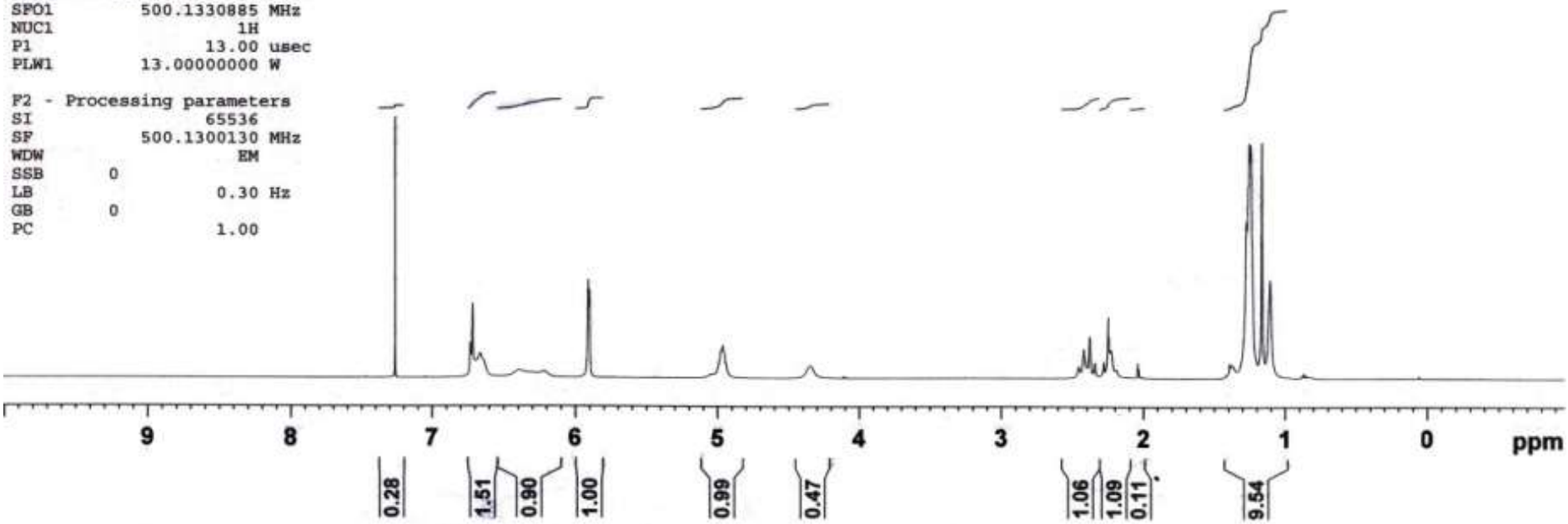


Fig S42. ^{1}H NMR Spectrum of 7d

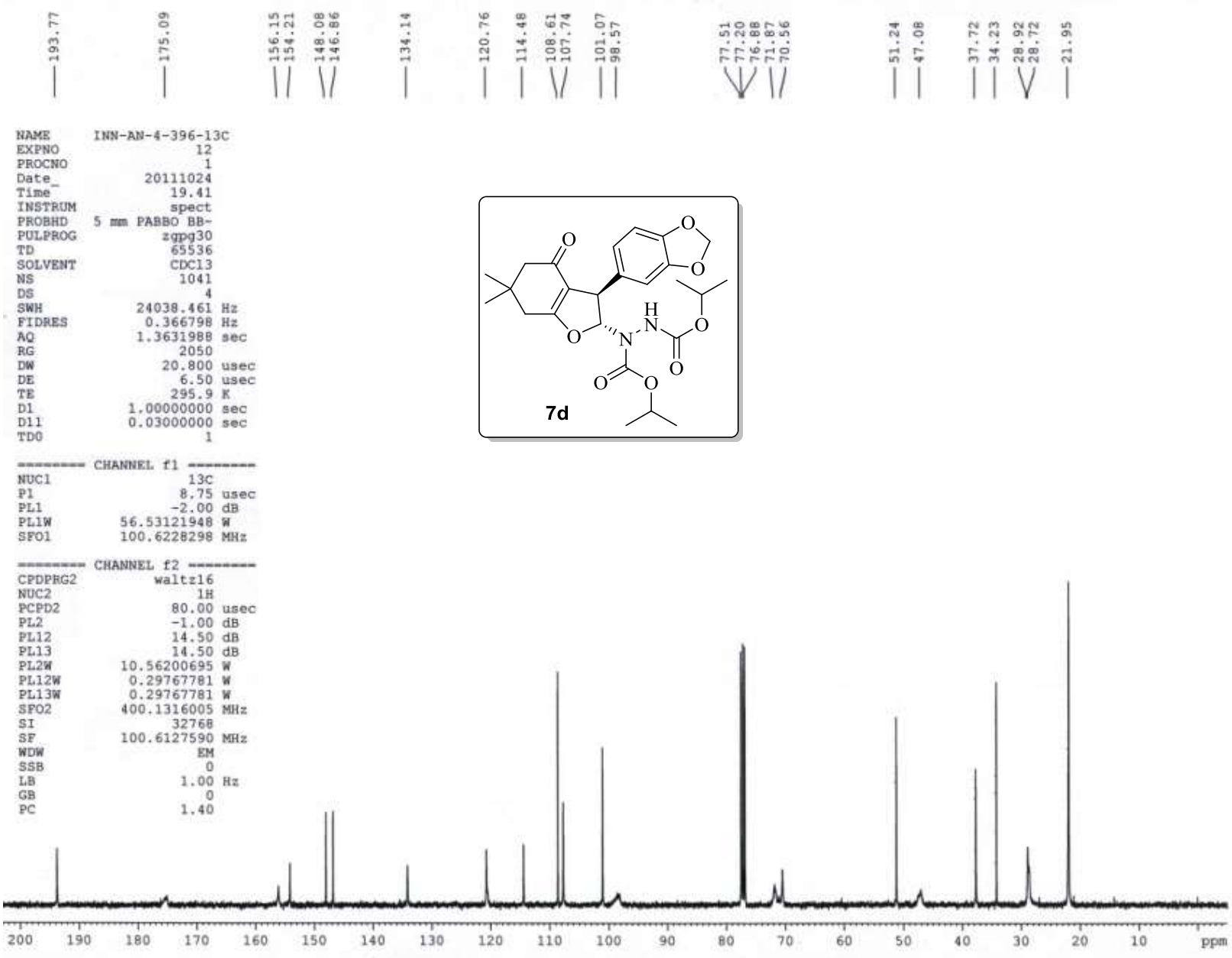
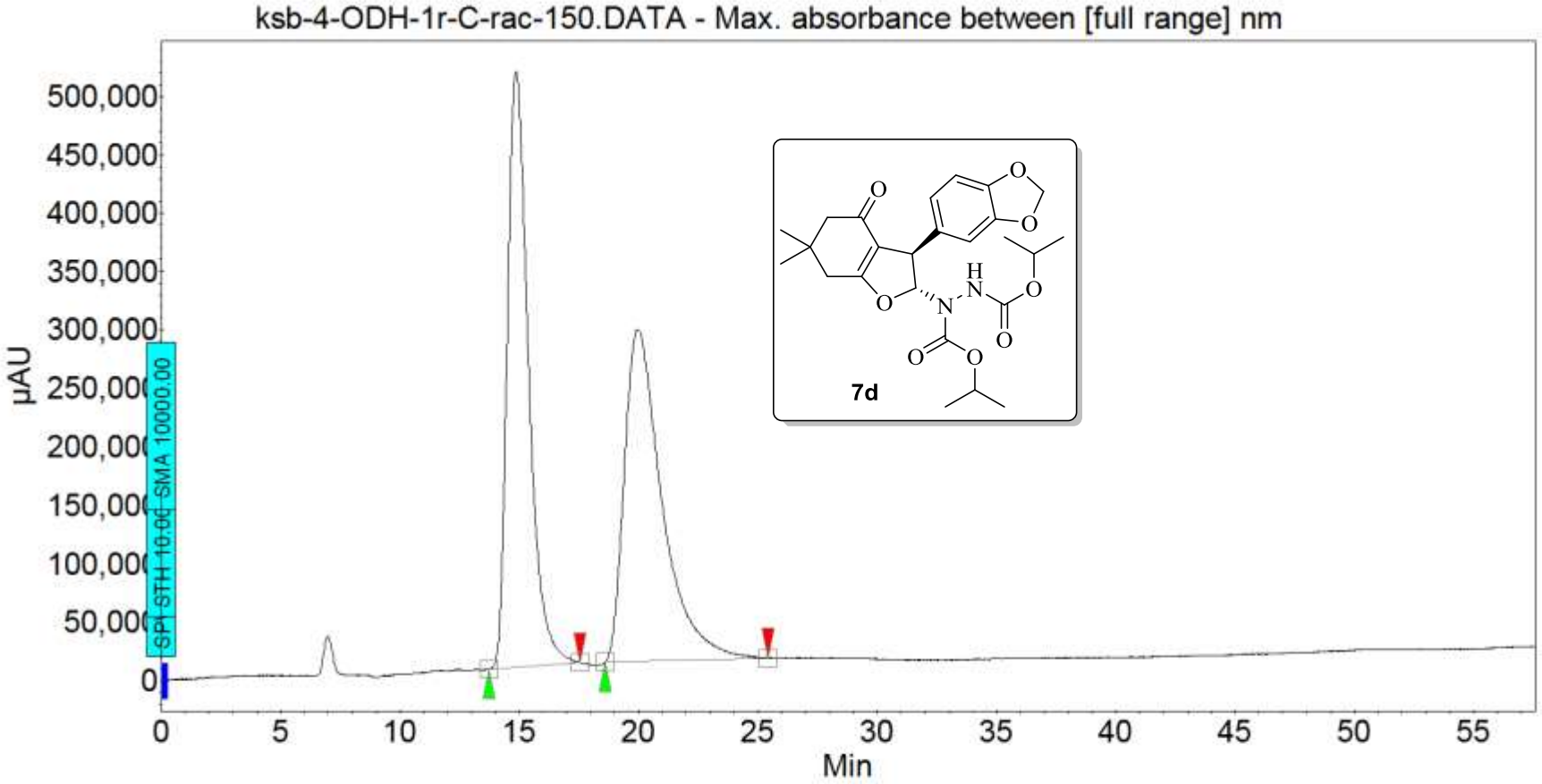


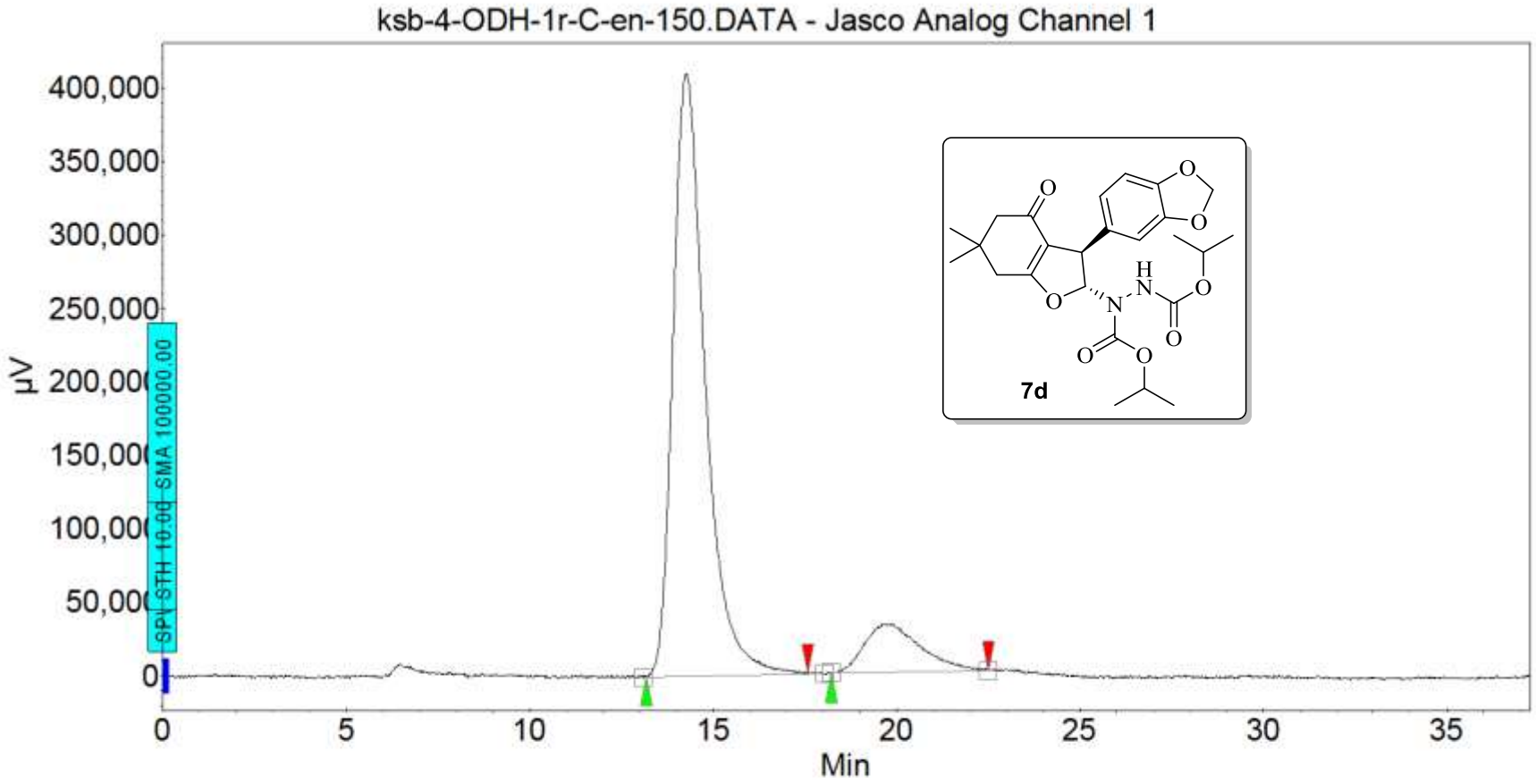
Fig S43. ^{13}C NMR Spectrum of 7d



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
2	UNKNOWN	14.852	50.29	509449.4	524637.8	50.295
1	UNKNOWN	19.959	49.71	283774.9	518485.3	49.705
Total			100.00	793224.3	1043123.1	100.000

Fig S44. HPLC Profile of Racemic 7d



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	14.275	87.97	410281.7	418205.7	87.974
2	UNKNOWN	19.642	12.03	32488.6	57168.7	12.026
Total			100.00	442770.4	475374.4	100.000

Fig S45. HPLC Profile of Enantioenriched 7d

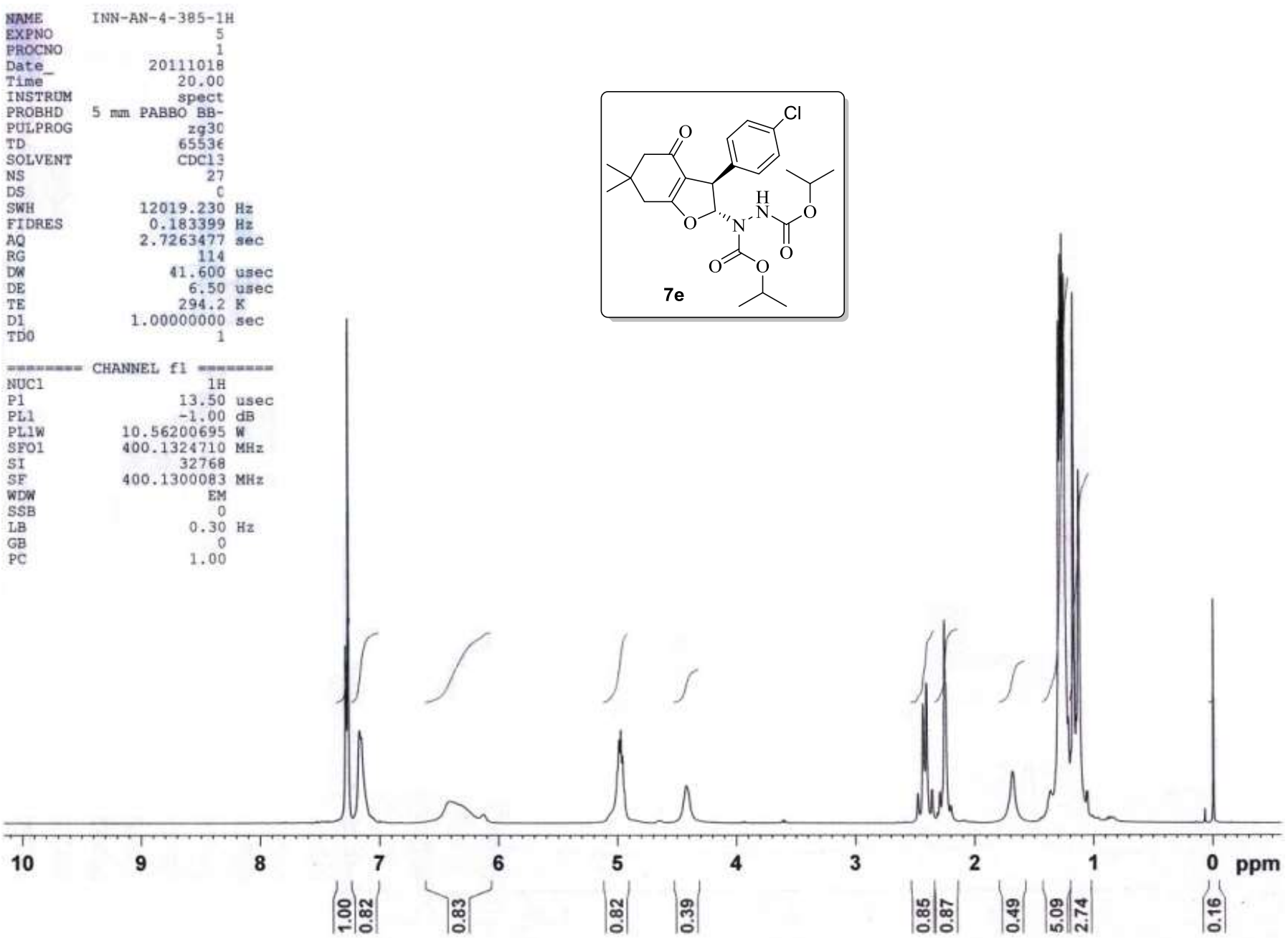


Fig S46. ^{1}H NMR Spectrum of 7e

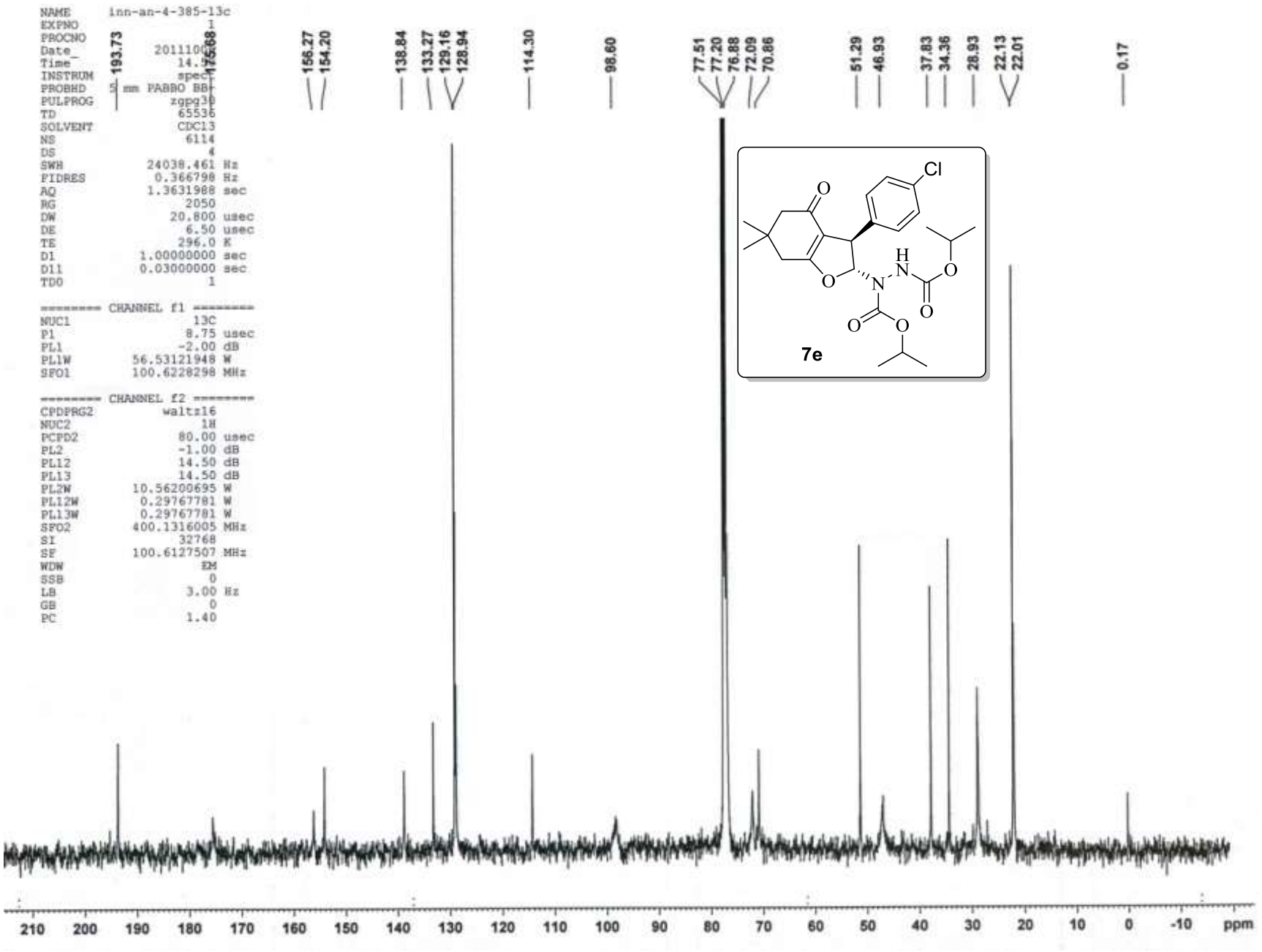
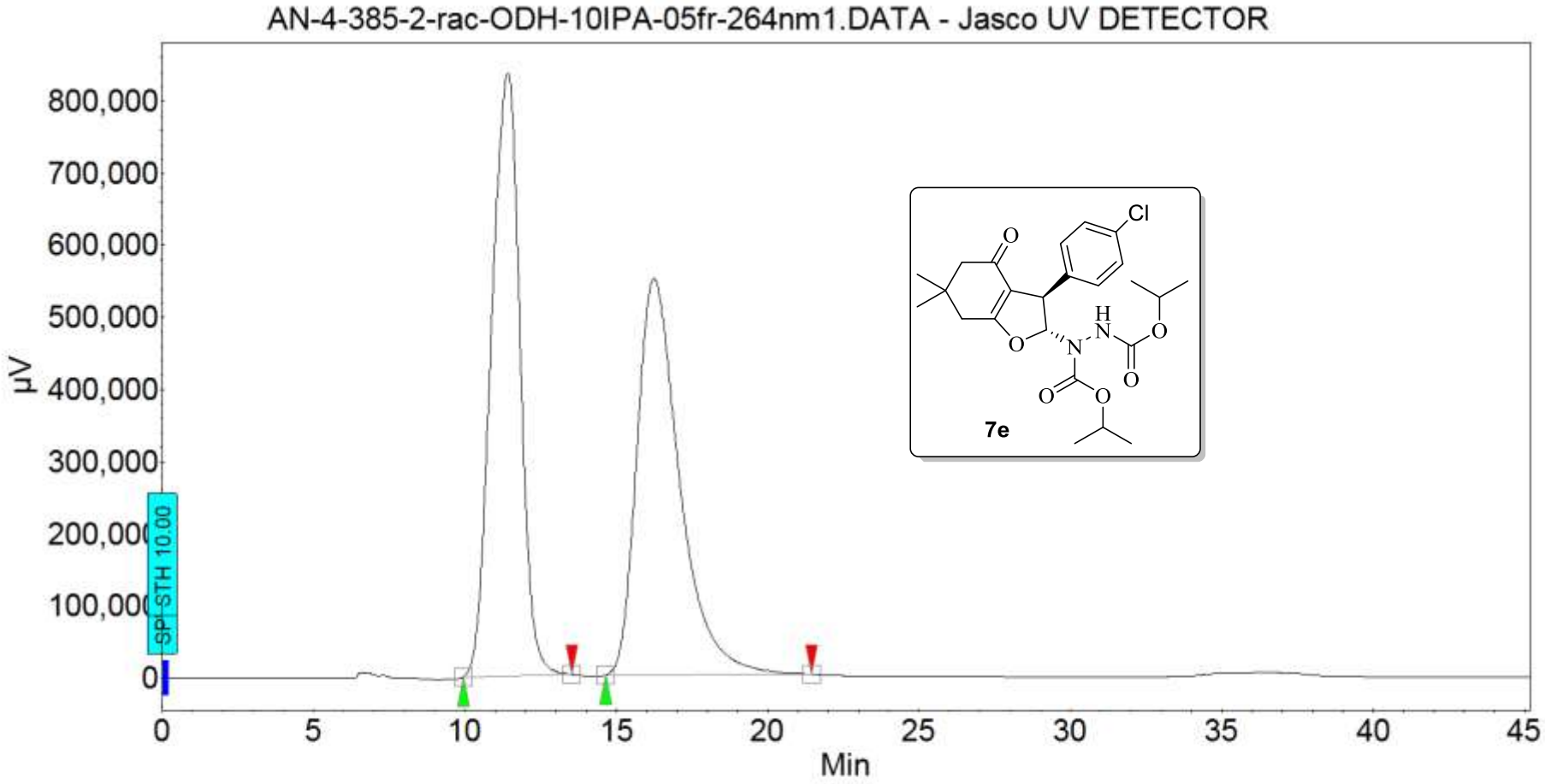


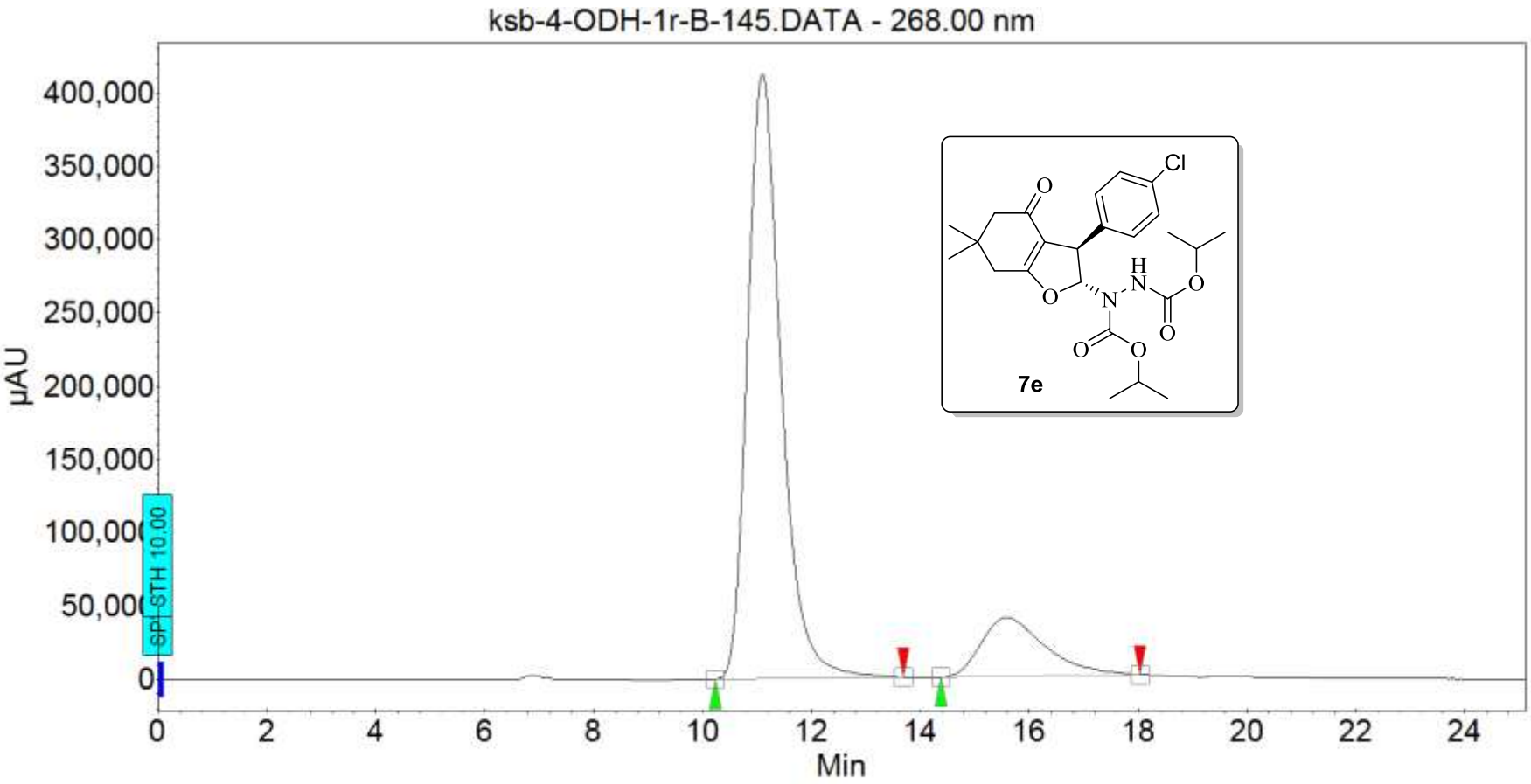
Fig S47. ^{13}C NMR Spectrum of 7e



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	11.417	50.46	837246.2	918050.8	50.463
2	UNKNOWN	16.242	49.54	550096.7	901199.8	49.537
Total			100.00	1387342.9	1819250.6	100.000

Fig S48. HPLC Profile of Racemic 7e



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	11.093	84.47	412679.5	295935.7	84.475
2	UNKNOWN	15.572	15.53	40105.0	54389.6	15.525
Total			100.00	452784.4	350325.3	100.000

Fig S49. HPLC Profile of Enantioenriched 7e

```
Current Data Parameters
NAME      INN-NSS-DHF-2-Br-1H@55°C
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160614
Time               12.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 66174
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            11029.412 Hz
FIDRES          0.166673 Hz
AQ             2.9998879 sec
RG                 33.93
DW                45.333 usec
DE                  6.50 usec
TE                 338.0 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           500.1300102 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

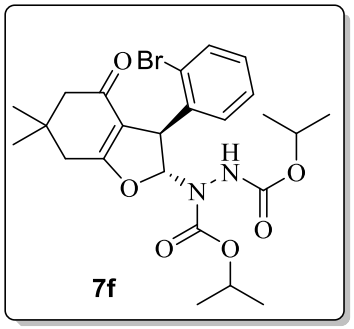


Fig S50. ^{1}H NMR Spectrum of 7f at +55 ^{o}C

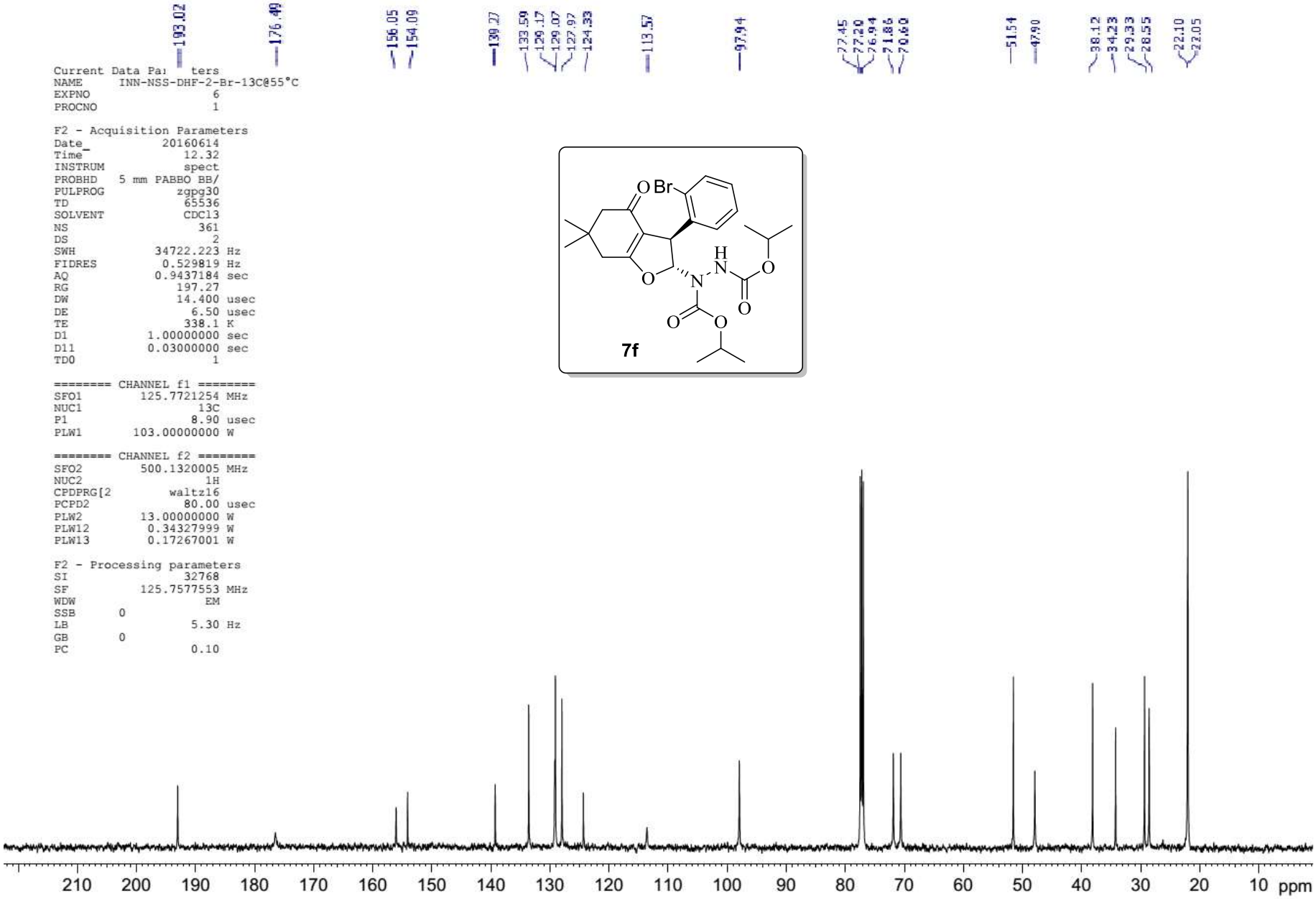


Fig S51. ^{13}C NMR Spectrum of 7f at +55 ^{o}C

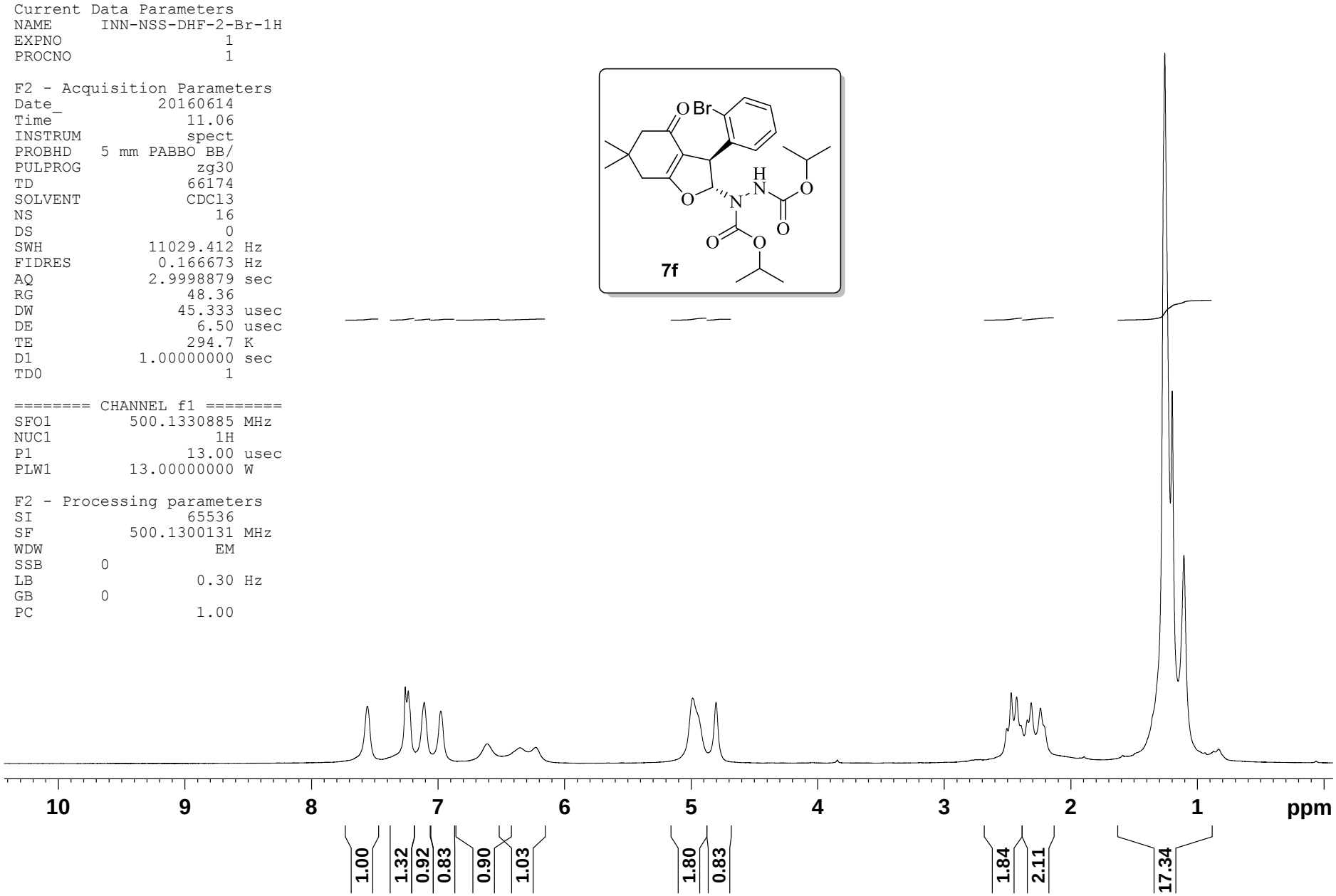


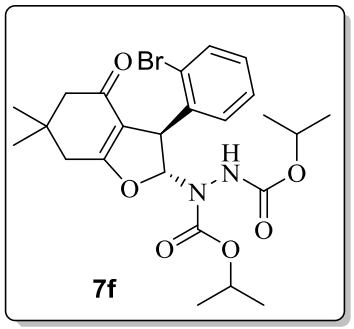
Fig S52. ^{1}H NMR Spectrum of 7f at +25 ^{o}C

```
Current Data Parameters
NAME      INN-NSS-DHF-2-Br-1H@40°C
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160614
Time               11.48
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 66174
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            11029.412 Hz
FIDRES          0.166673 Hz
AQ             2.9998879 sec
RG                 33.93
DW                45.333 usec
DE                 6.50 usec
TE                313.0 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           500.1300112 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



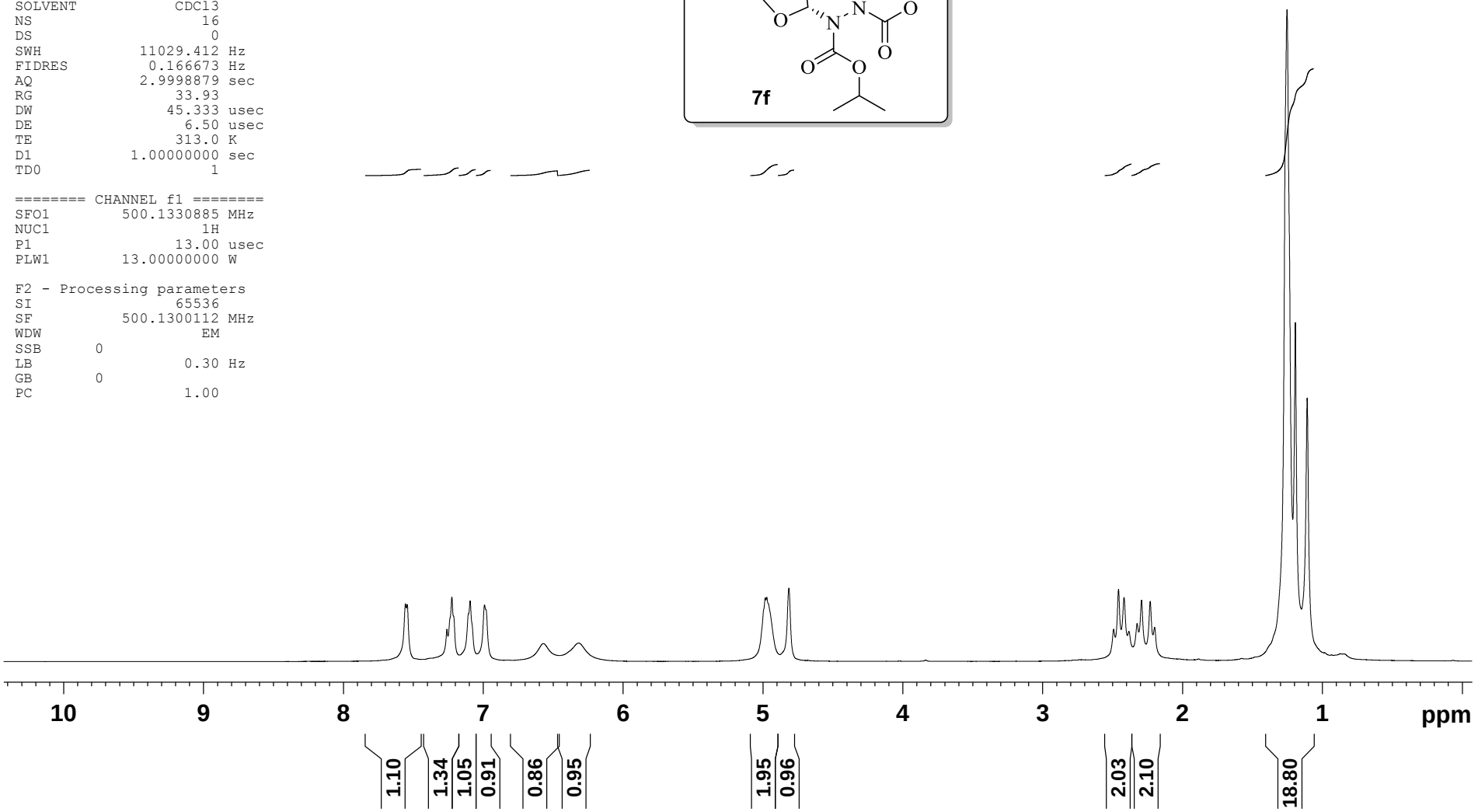


Fig S53. ^{1}H NMR Spectrum of 7f at +40 ^{o}C

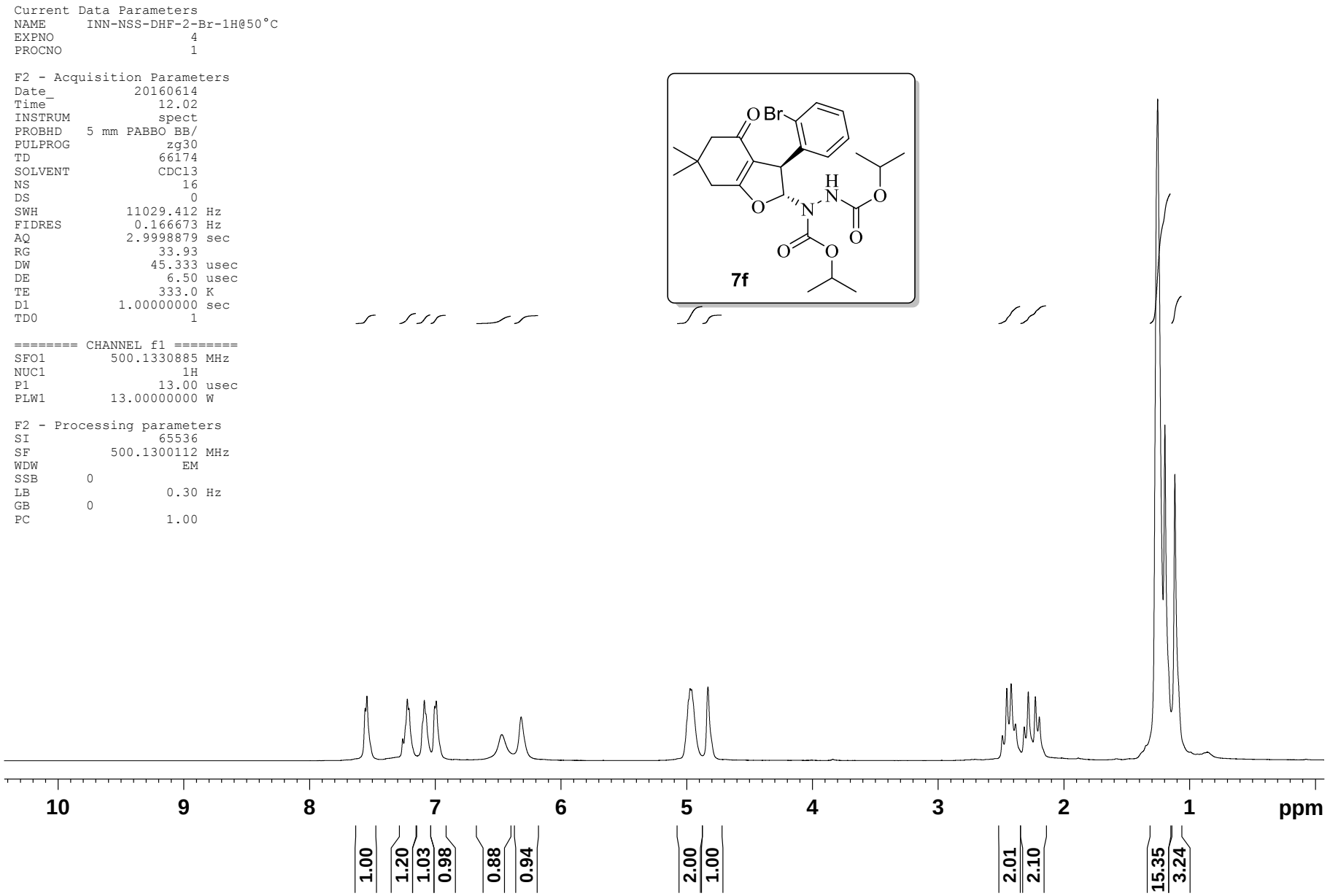
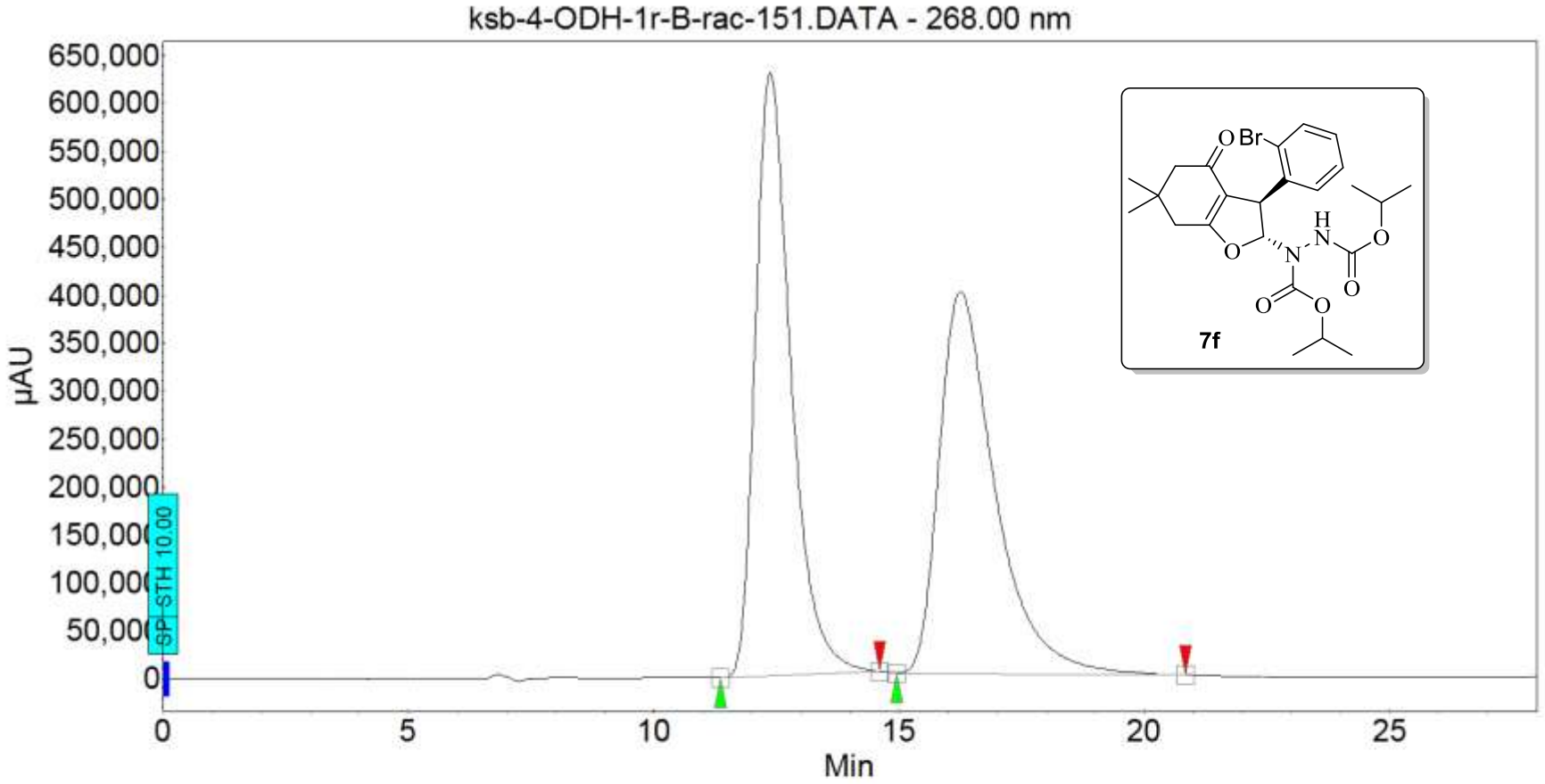


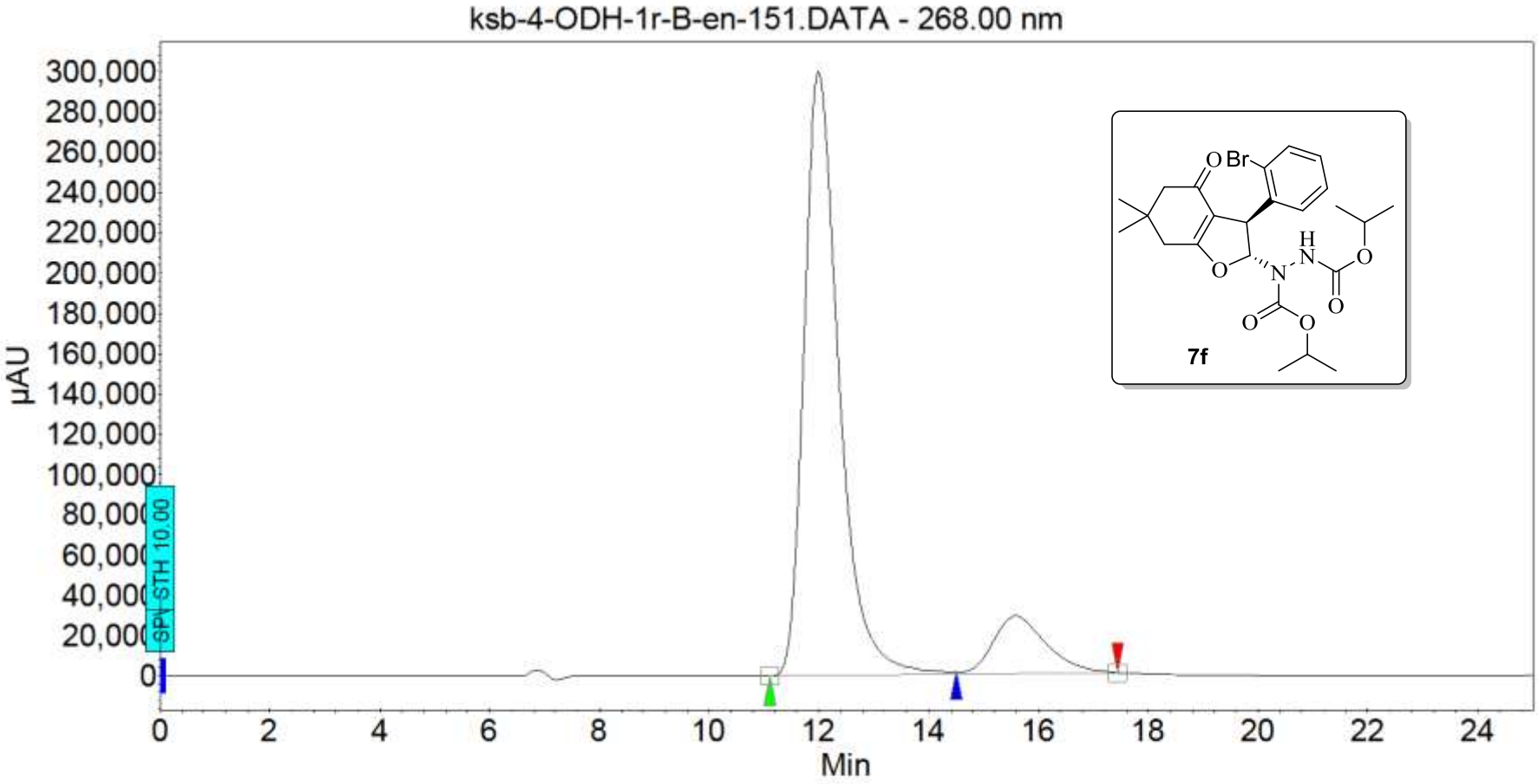
Fig S54. ^{1}H NMR Spectrum of 7f at +50 ^{o}C



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	12.369	50.17	629639.6	531019.4	50.175
2	UNKNOWN	16.248	49.83	398279.4	527324.9	49.825
Total			100.00	1027919.0	1058344.3	100.000

Fig S55. HPLC Profile of Racemic 7f



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	11.986	87.53	299233.0	222404.0	87.528
2	UNKNOWN	15.586	12.47	28498.7	31690.9	12.472
Total			100.00	327731.7	254095.0	100.000

Fig S56. HPLC Profile of Enantioenriched 7f

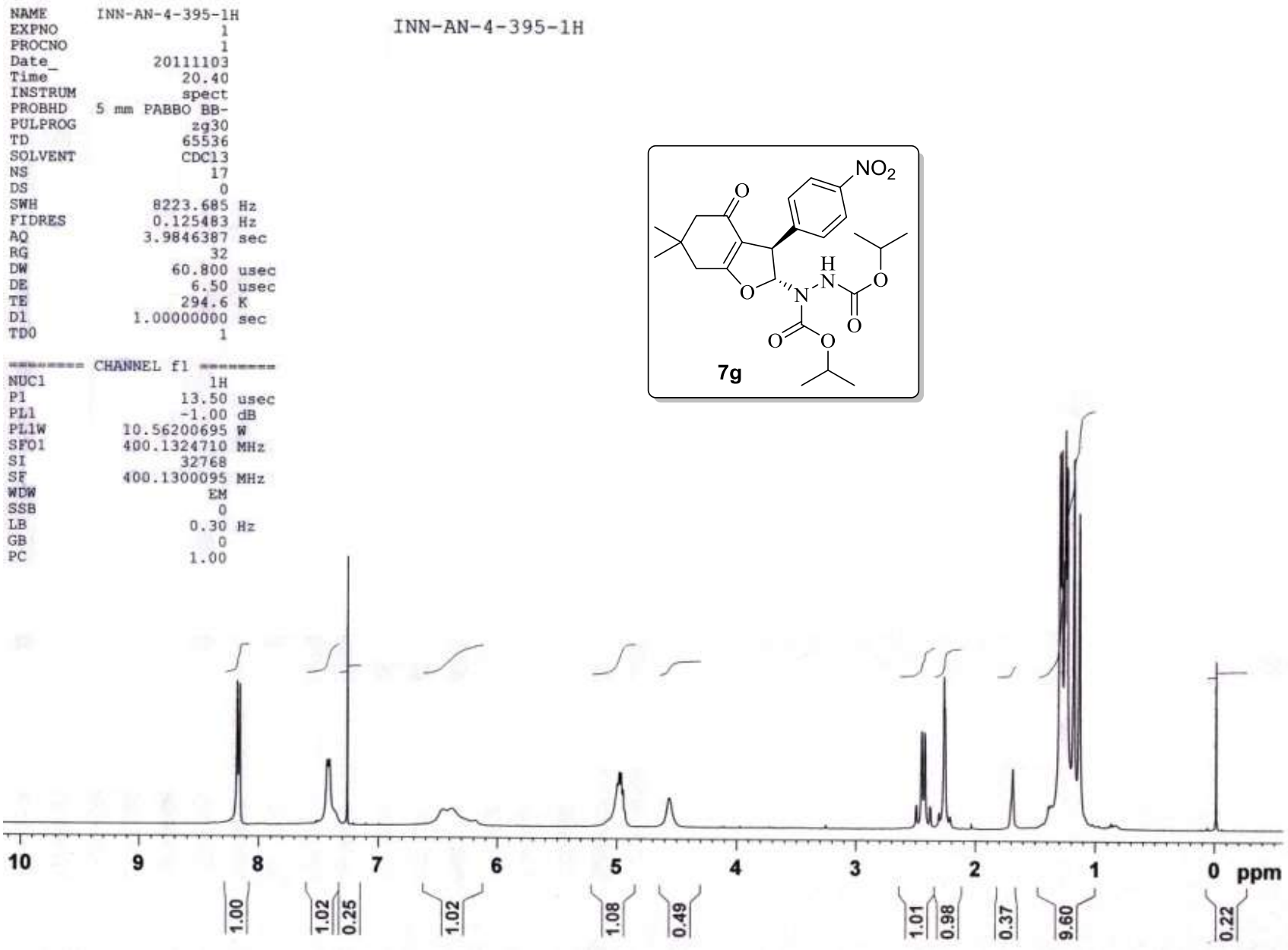


Fig S57. ^{1}H NMR Spectrum of 7g

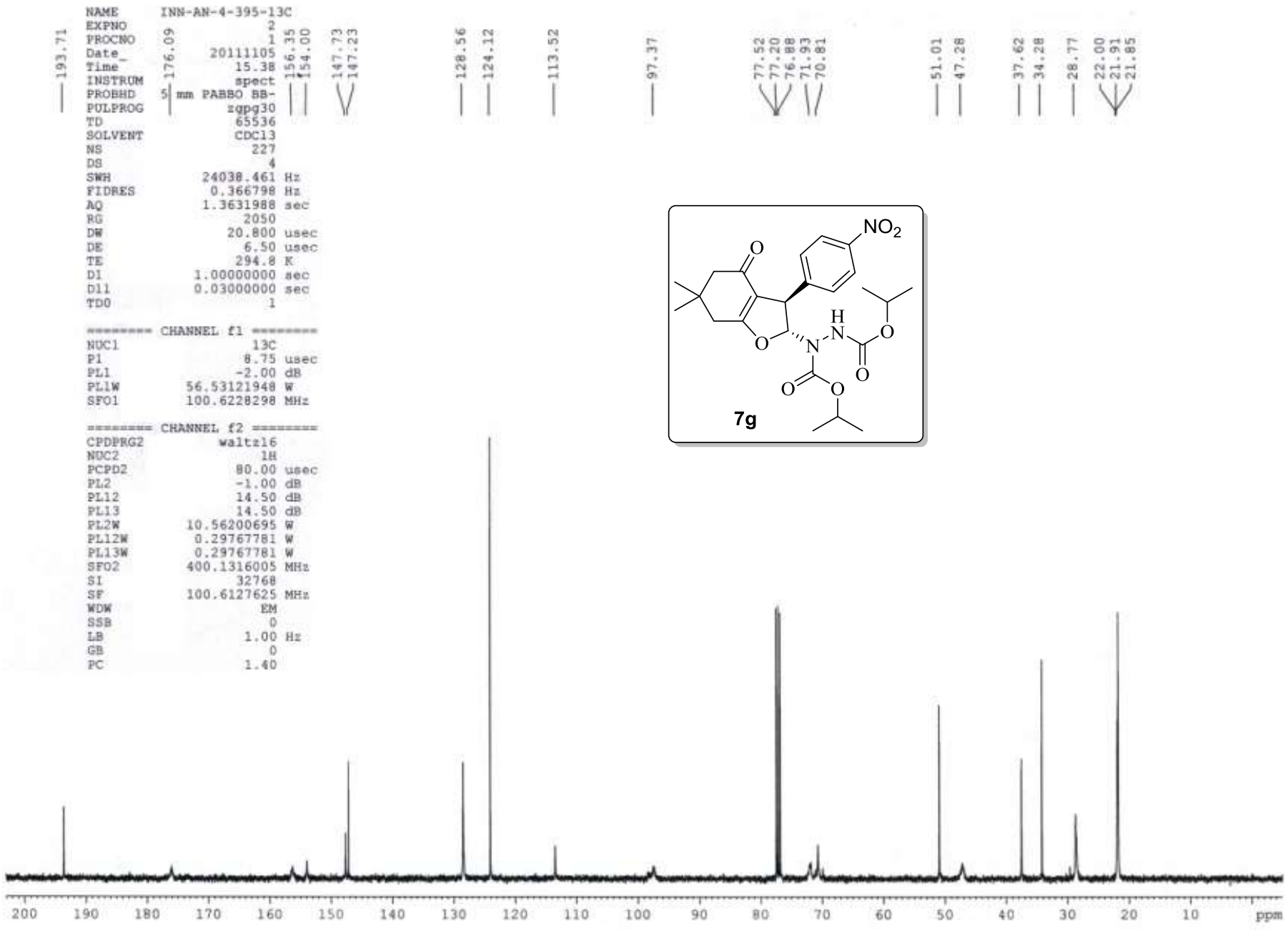
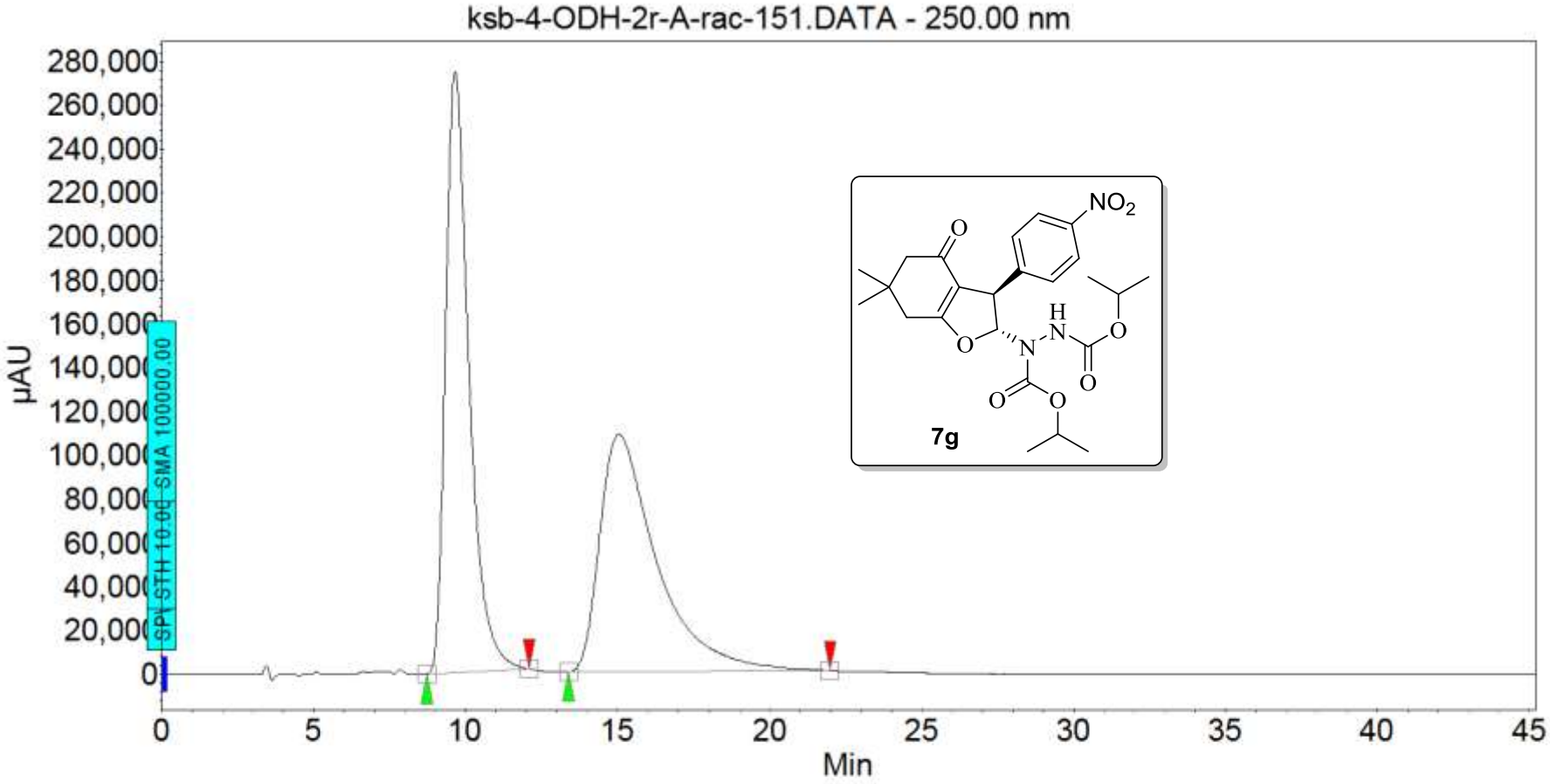


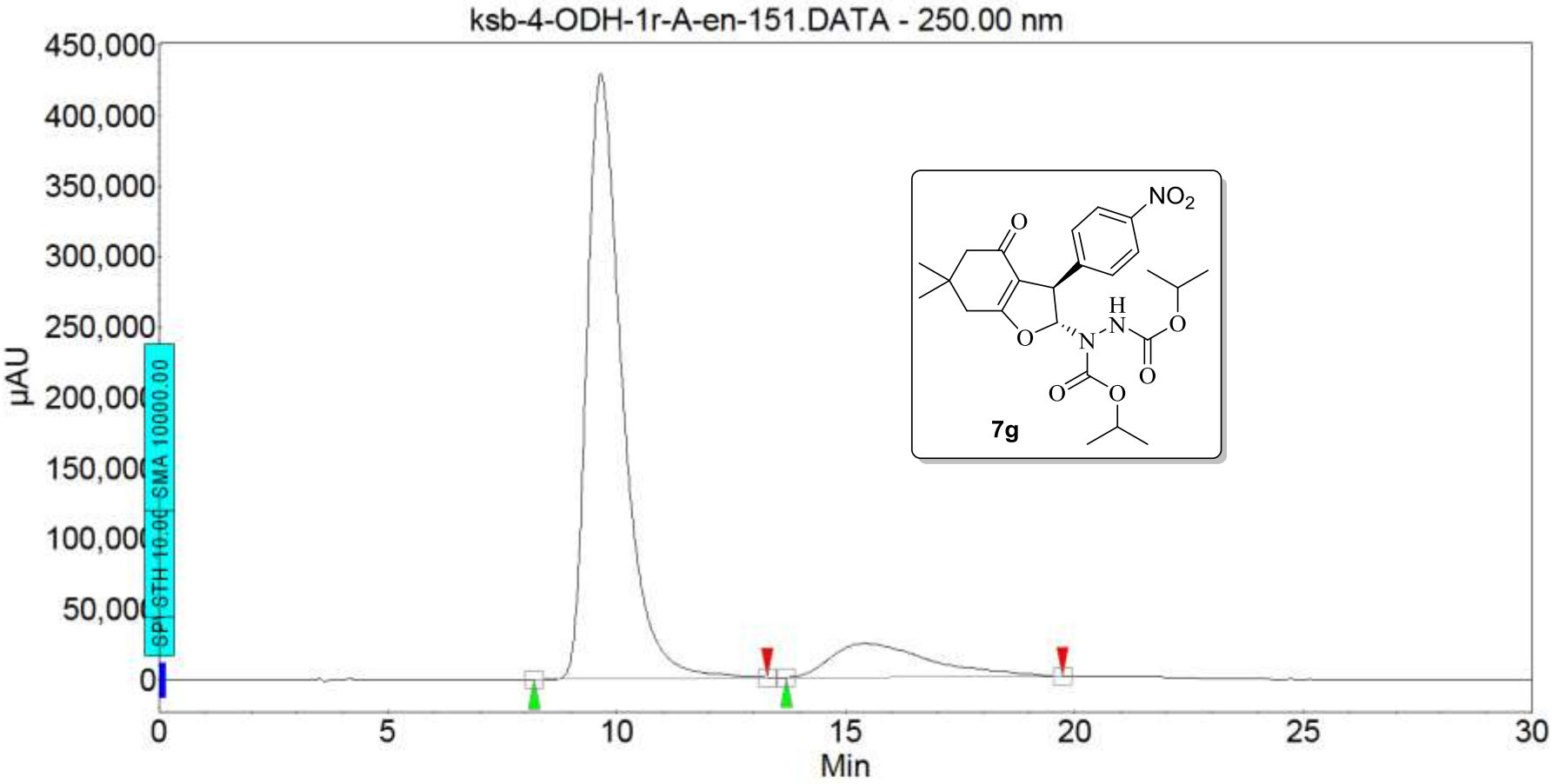
Fig S58. ^{13}C NMR Spectrum of 7g



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	9.653	51.12	274604.6	245530.9	51.125
2	UNKNOWN	15.039	48.88	108344.3	234729.3	48.875
Total			100.00	382949.0	480260.3	100.000

Fig S59. HPLC Profile of Racemic 7g



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	9.637	86.42	429283.0	383923.2	86.418
2	UNKNOWN	15.408	13.58	24071.7	60342.3	13.582
Total			100.00	453354.7	444265.4	100.000

Fig S60. HPLC Profile of Enantioenriched 7g

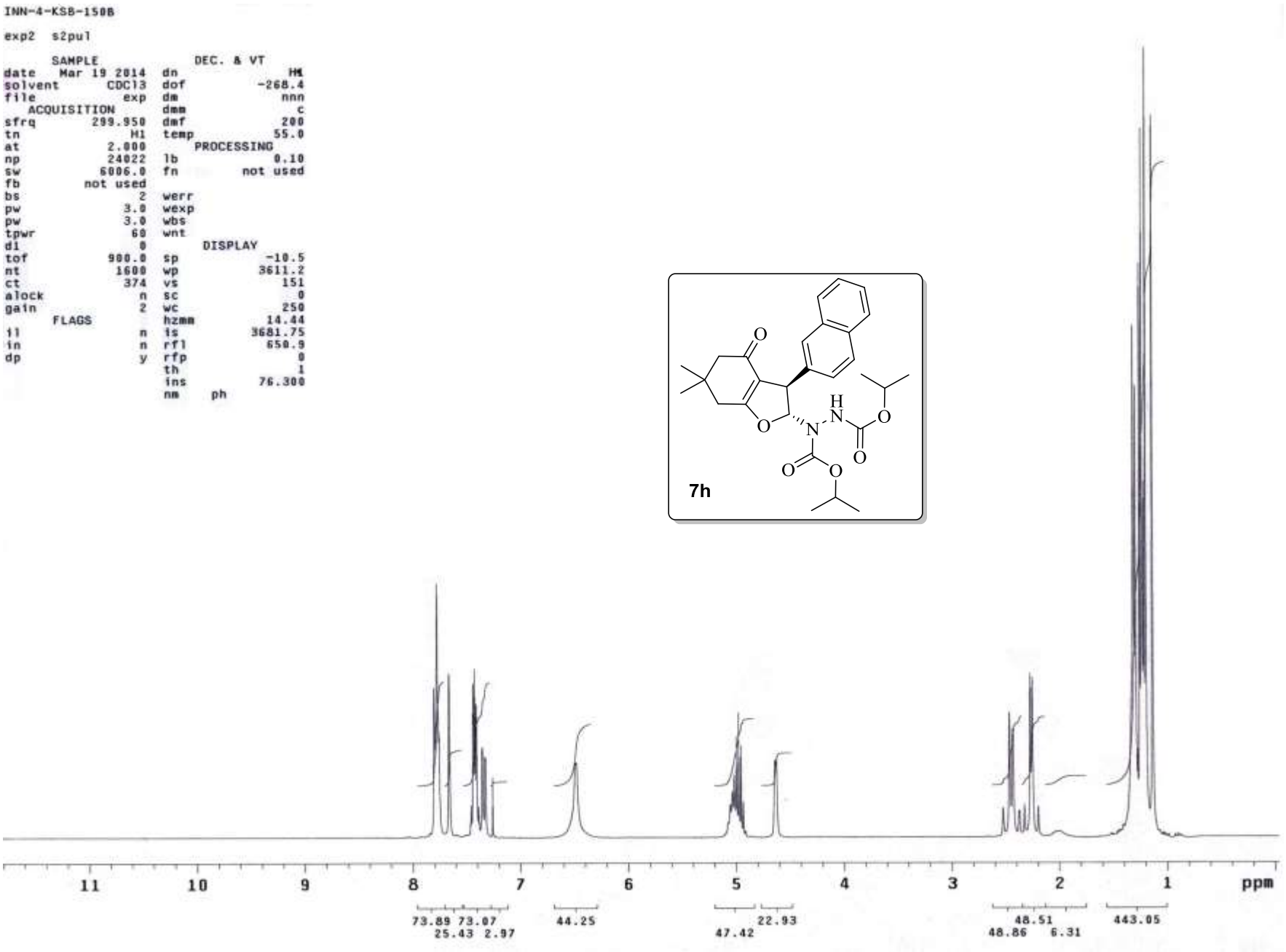


Fig S61. ^{1}H NMR Spectrum of 7h at + 55 ^{o}C

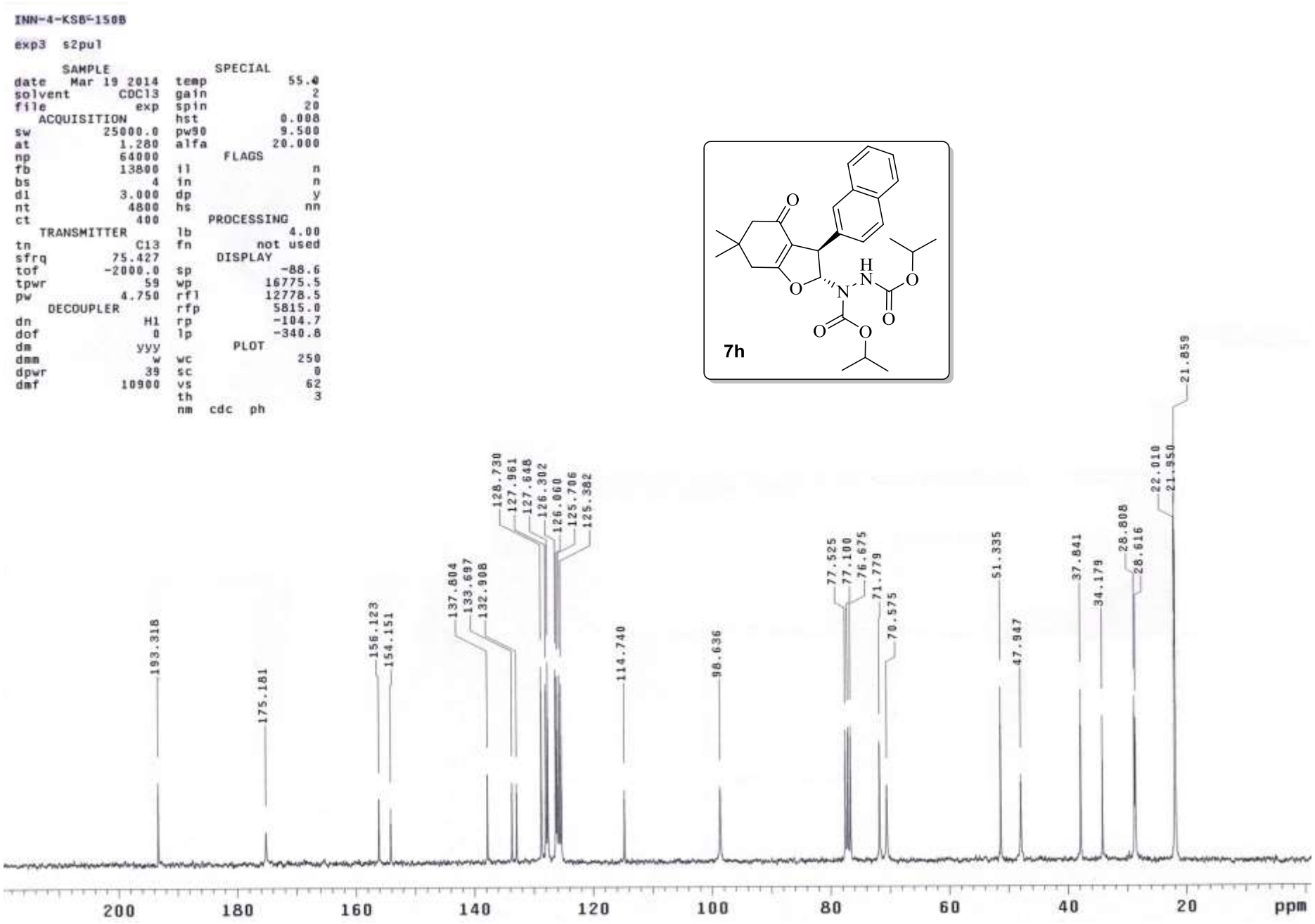


Fig S62. ^{13}C NMR Spectrum of 7h at +55 ^{o}C

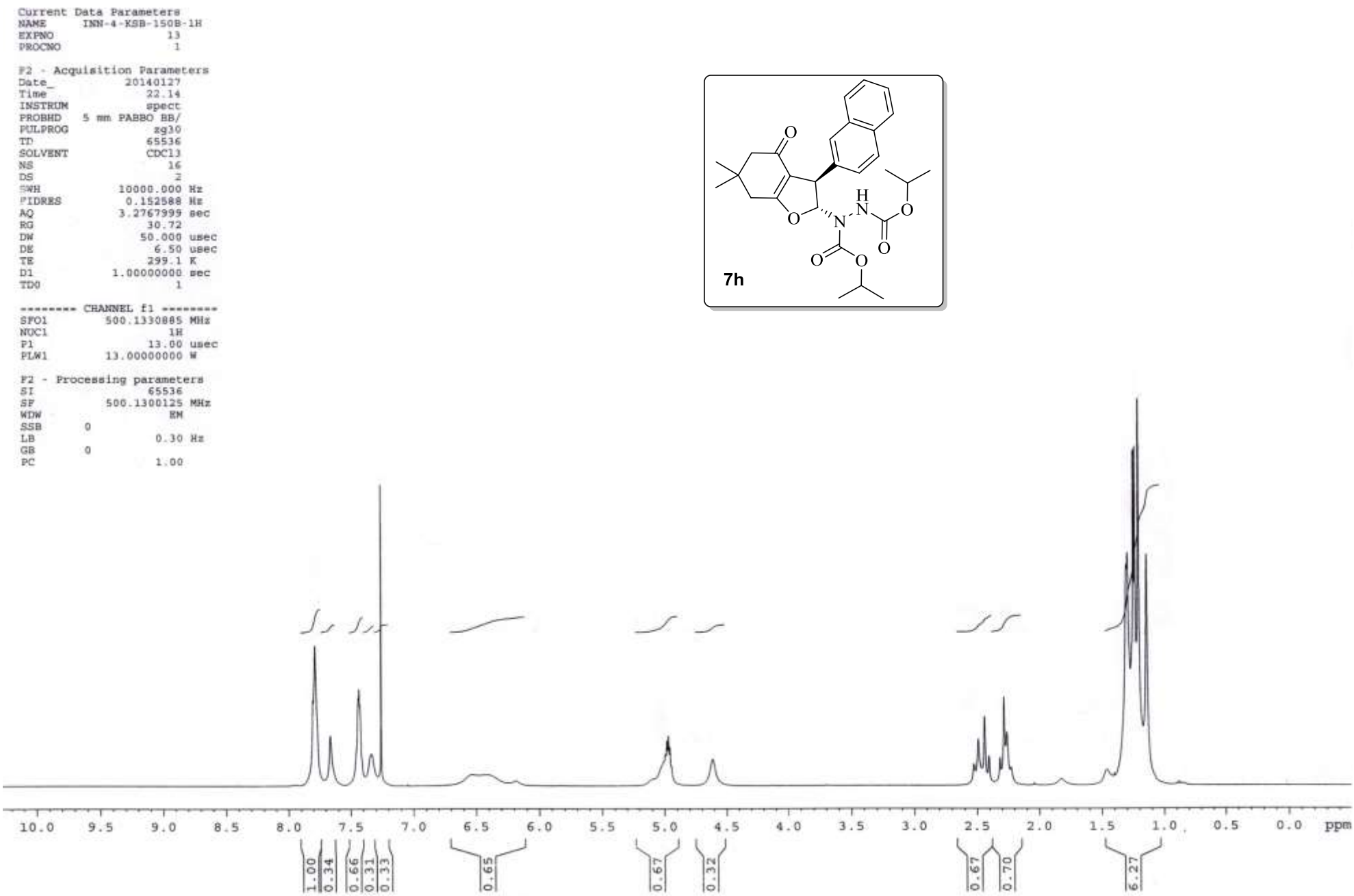


Fig S63 ^{1}H NMR Spectrum of 7h at +25 ^{o}C

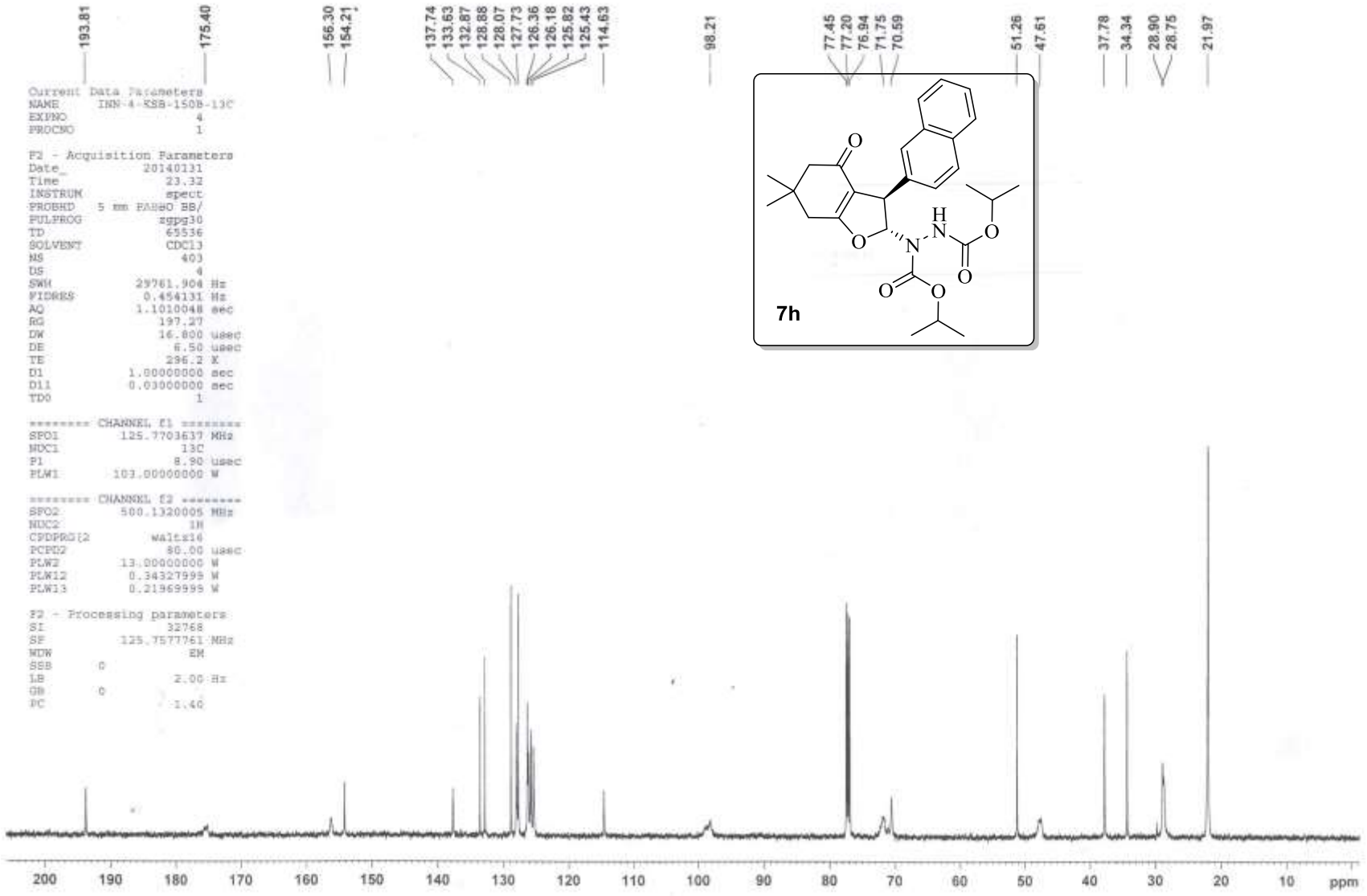
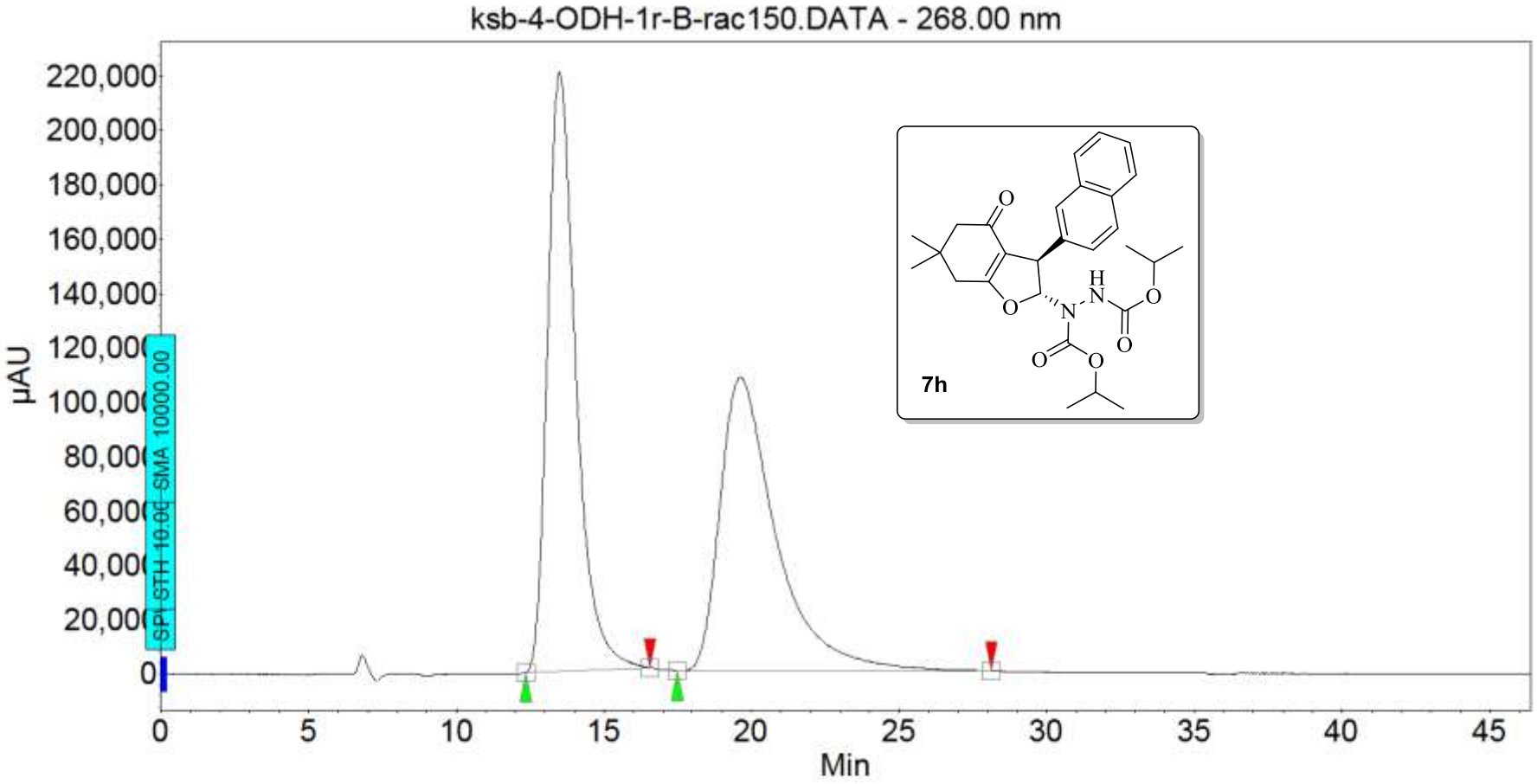


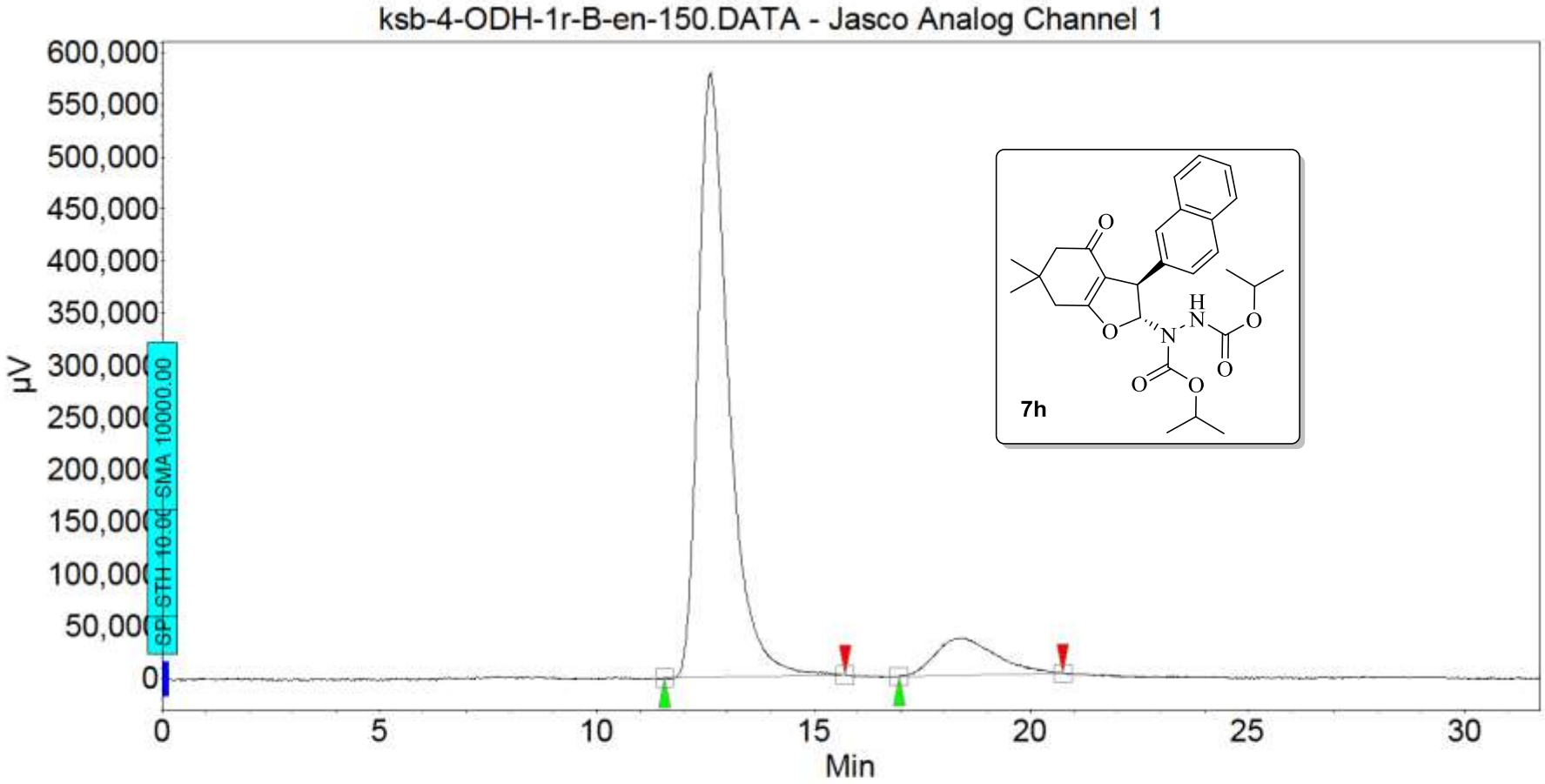
Fig S64. ^{13}C NMR Spectrum of 7h at +25 ^{o}C



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	13.506	50.73	220386.8	244911.2	50.733
2	UNKNOWN	19.612	49.27	108024.3	237834.6	49.267
Total			100.00	328411.1	482745.9	100.000

Fig S65. HPLC Profile of Racemic 7h



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	12.625	89.26	580240.6	469968.2	89.259
2	UNKNOWN	18.408	10.74	35550.8	56555.3	10.741
Total			100.00	615791.4	526523.5	100.000

Fig S66. HPLC Profile of Enantioenriched 7h

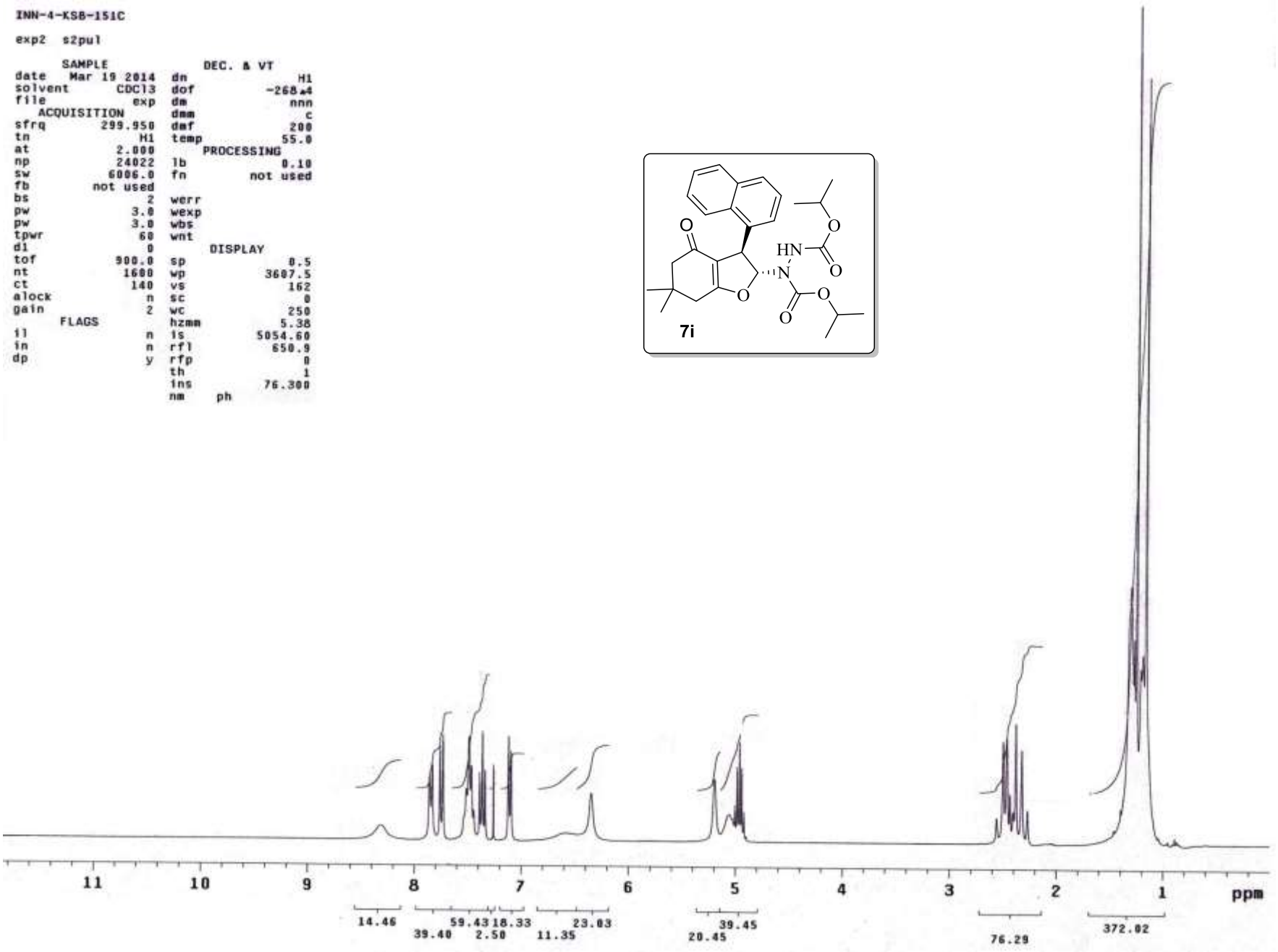


Fig S67. ^{1}H NMR Spectrum of 7i at +55 ^{o}C

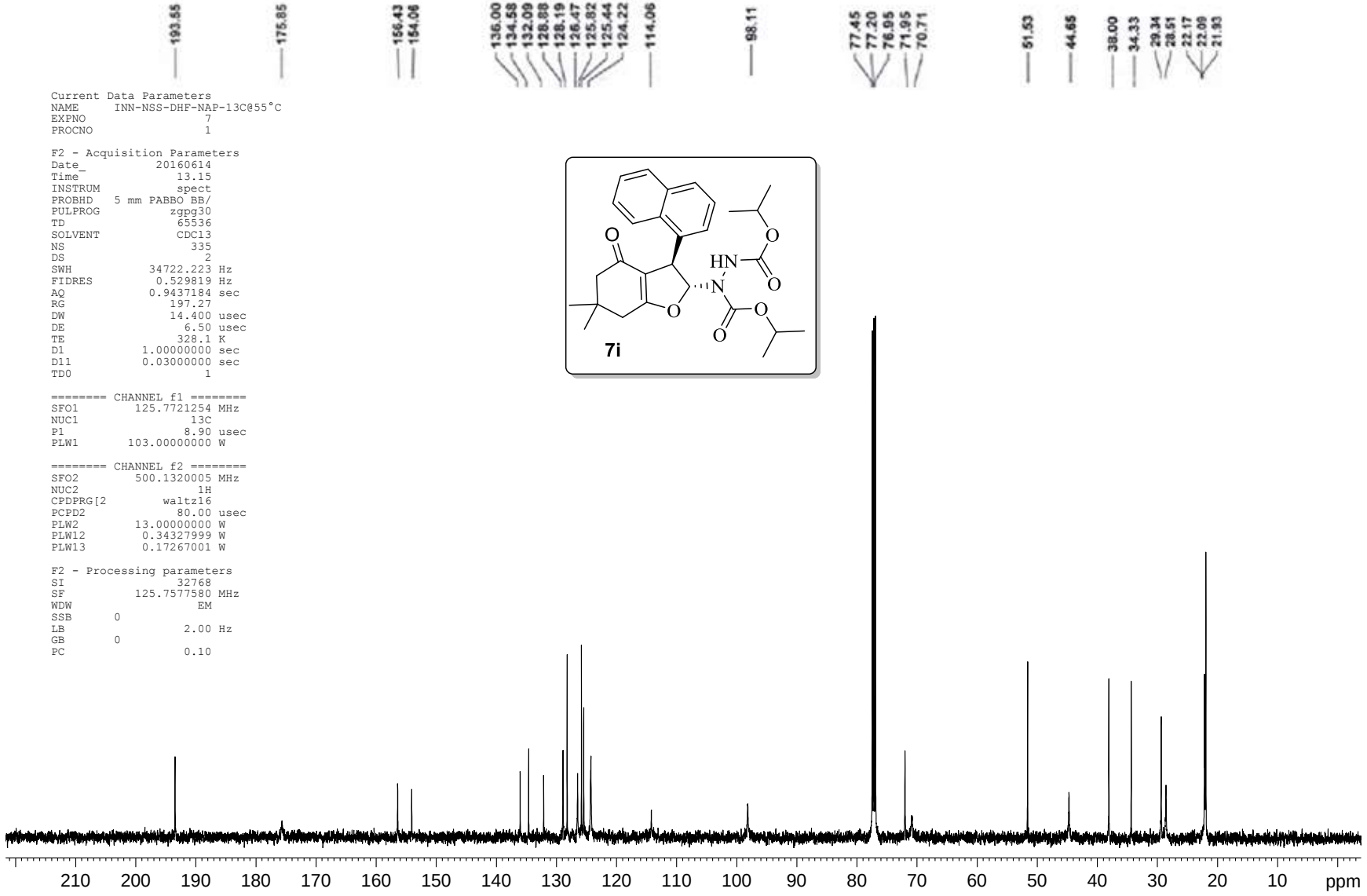
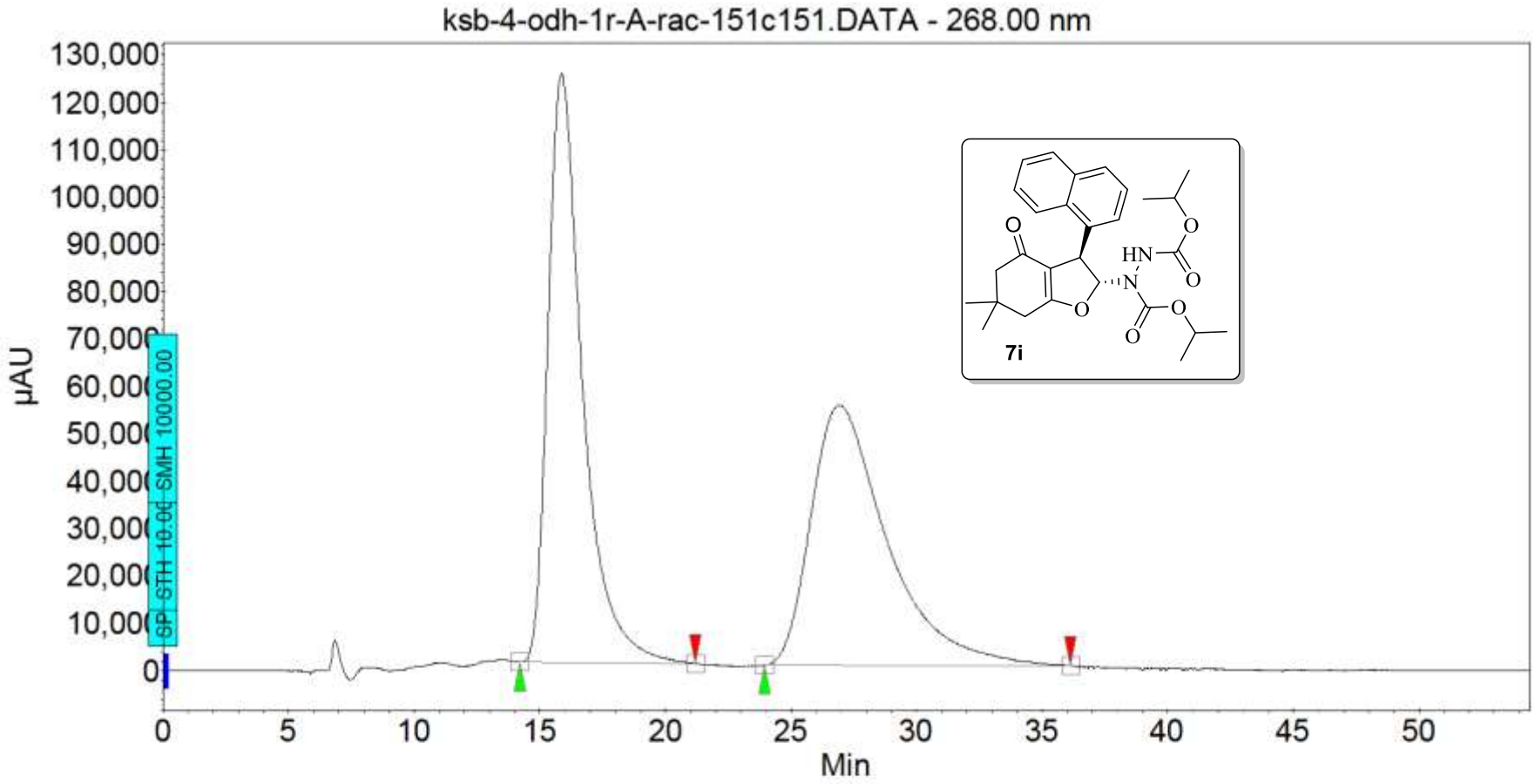


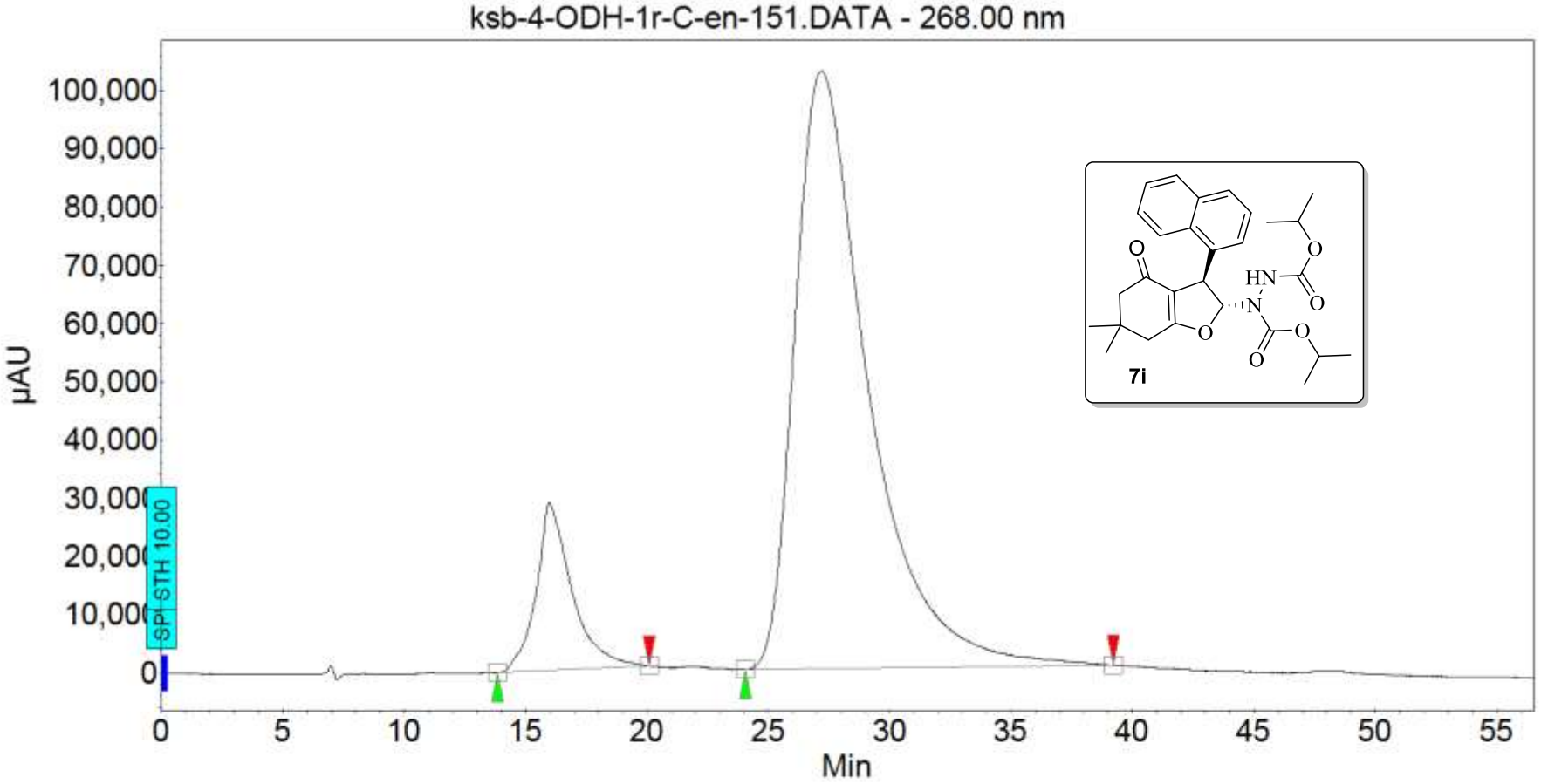
Fig S68. ^{13}C NMR Spectrum of 7i at +55 ^{o}C



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
2	UNKNOWN	15.852	50.72	124324.4	198710.6	50.717
1	UNKNOWN	26.918	49.28	54948.2	193092.0	49.283
Total			100.00	179272.6	391802.6	100.000

Fig S69. HPLC Profile of Racemic 7i



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	15.972	11.95	28729.3	48964.3	11.953
2	UNKNOWN	27.185	88.05	102624.0	360683.0	88.047
Total			100.00	131353.3	409647.3	100.000

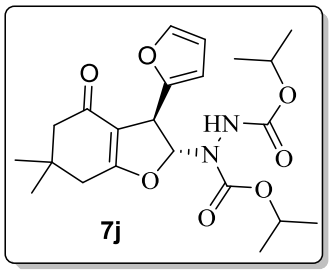
Fig S70. HPLC Profile of Enantioenriched 7i

```
Current Data Parameters
NAME      inn-4-ksb-115c-1h
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20131117
Time                 22.12
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                  294.6 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
```



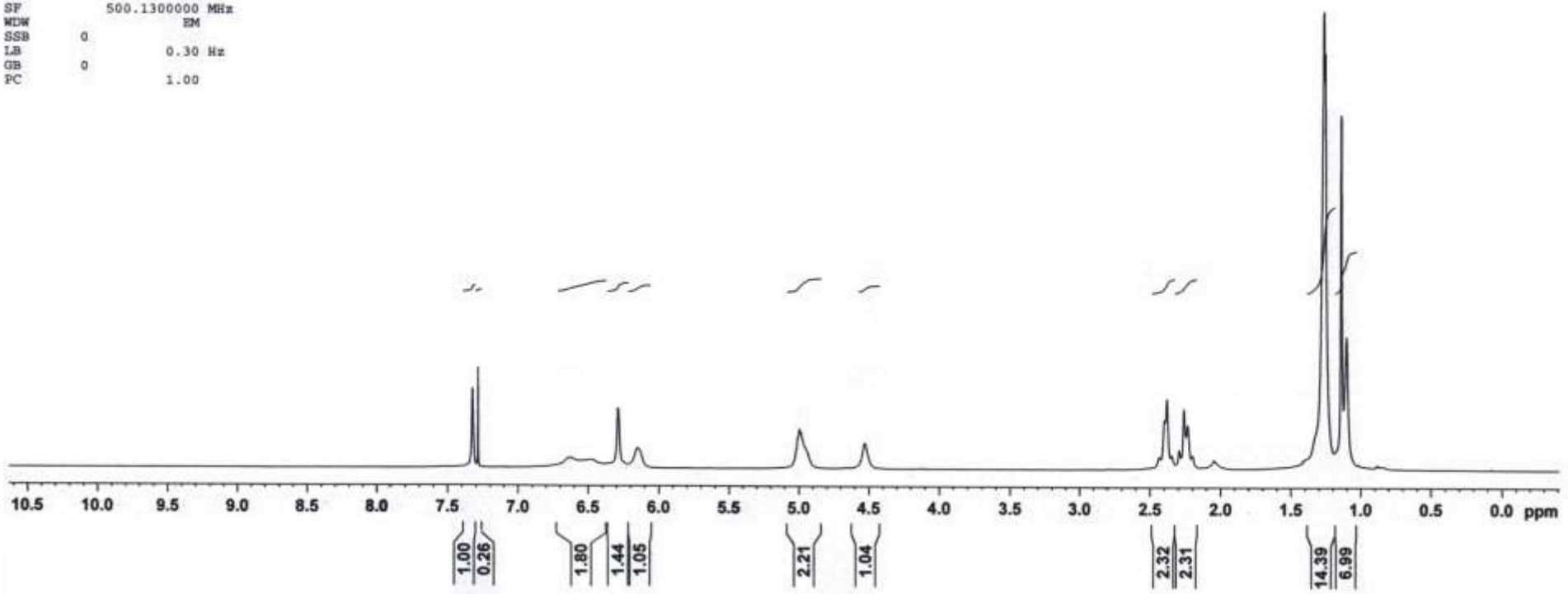


Fig S71. ^{1}H NMR Spectrum of 7j

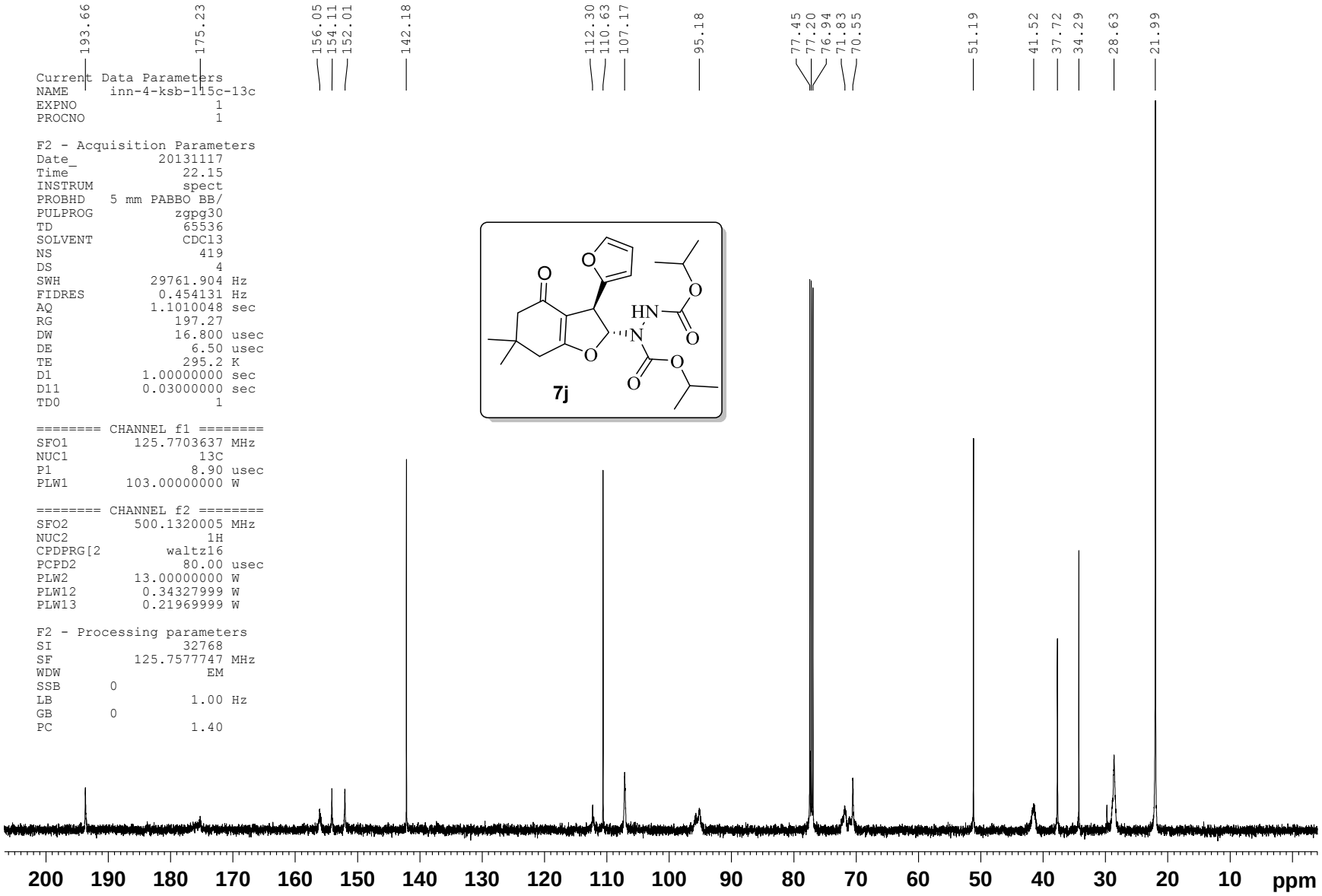
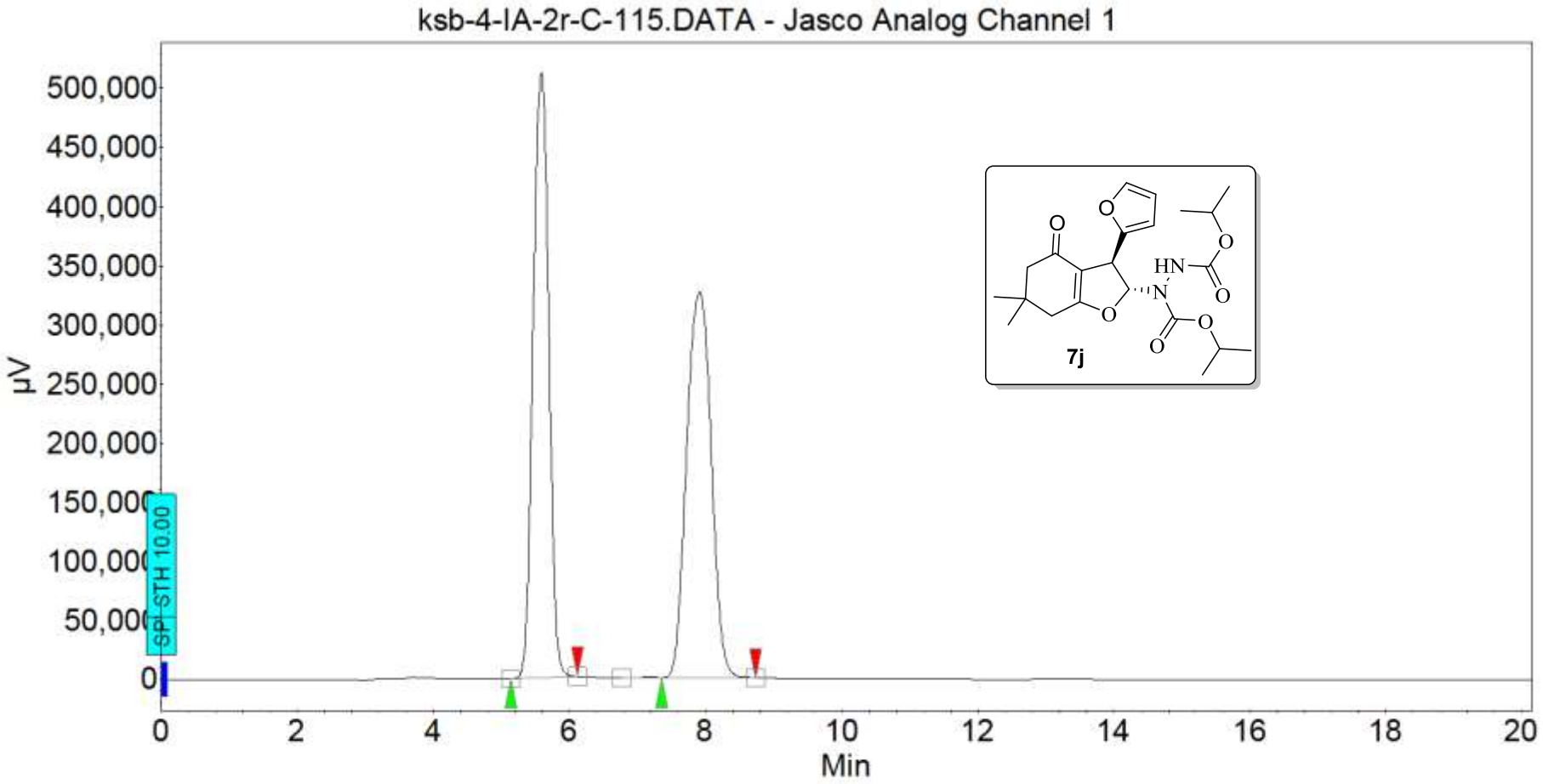


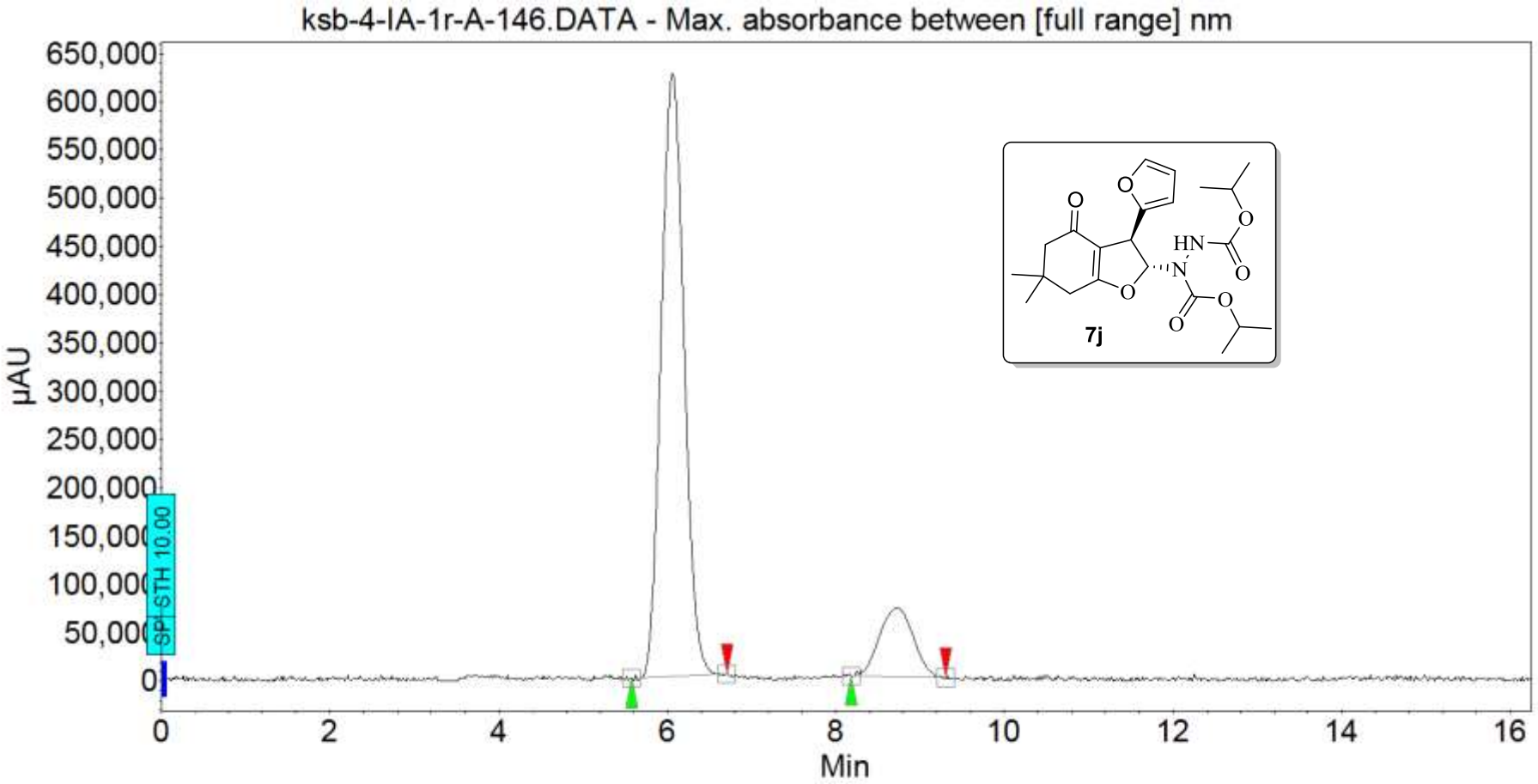
Fig S72. ^{13}C NMR Spectrum of 7j



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
2	UNKNOWN	5.583	50.04	512009.5	137337.9	50.042
1	UNKNOWN	7.908	49.96	327193.9	137107.4	49.958
Total			100.00	839203.4	274445.3	100.000

Fig S73. HPLC Profile of Racemic 7j



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	6.066	85.49	624596.3	195835.8	85.494
2	UNKNOWN	8.733	14.51	71188.5	33226.9	14.506
Total			100.00	695784.8	229062.7	100.000

Fig S74. HPLC Profile of Enantioenriched 7j

```
Current Data Parameters
NAME      INN-4-KSB-115B-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20131117
Time              21.57
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                294.5 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           500.1300000 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
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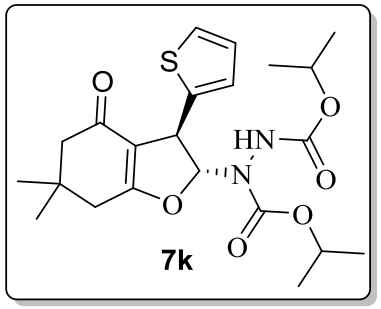


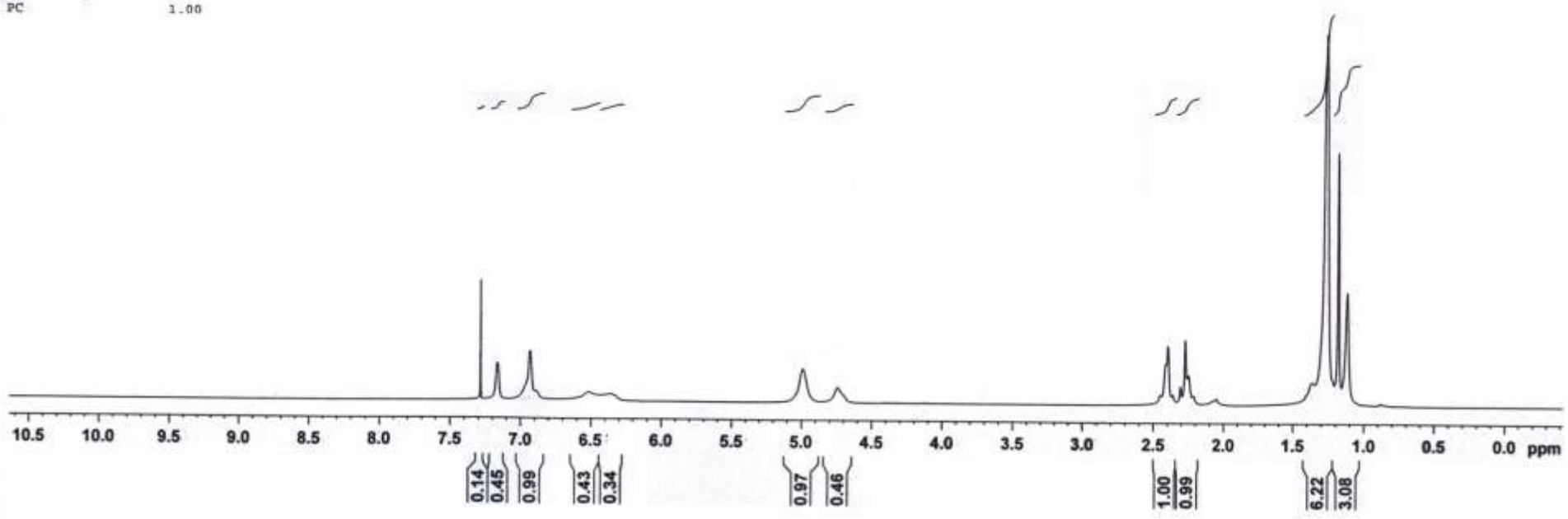


Fig S75. ^{1}H NMR Spectrum of 7k

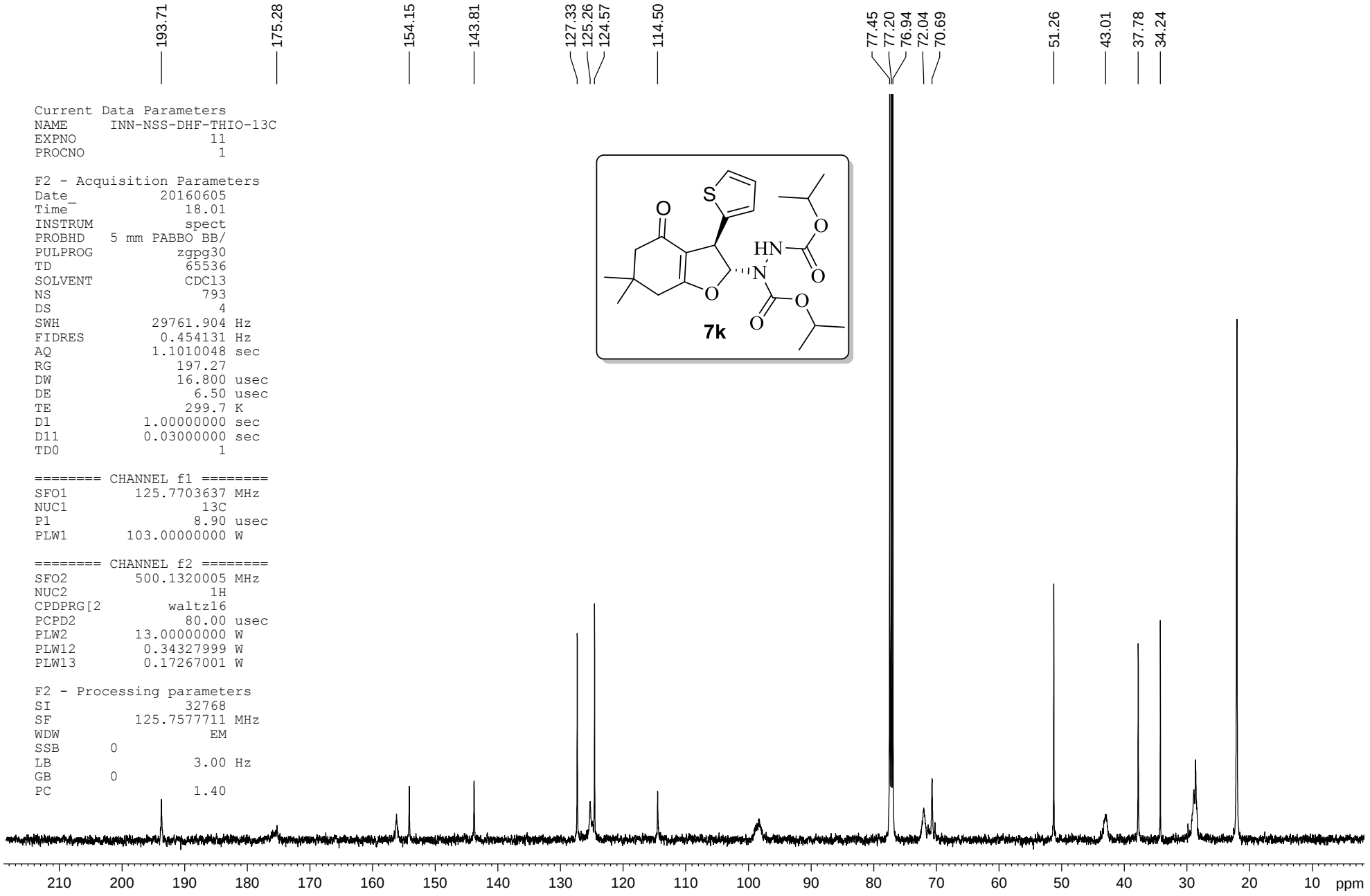
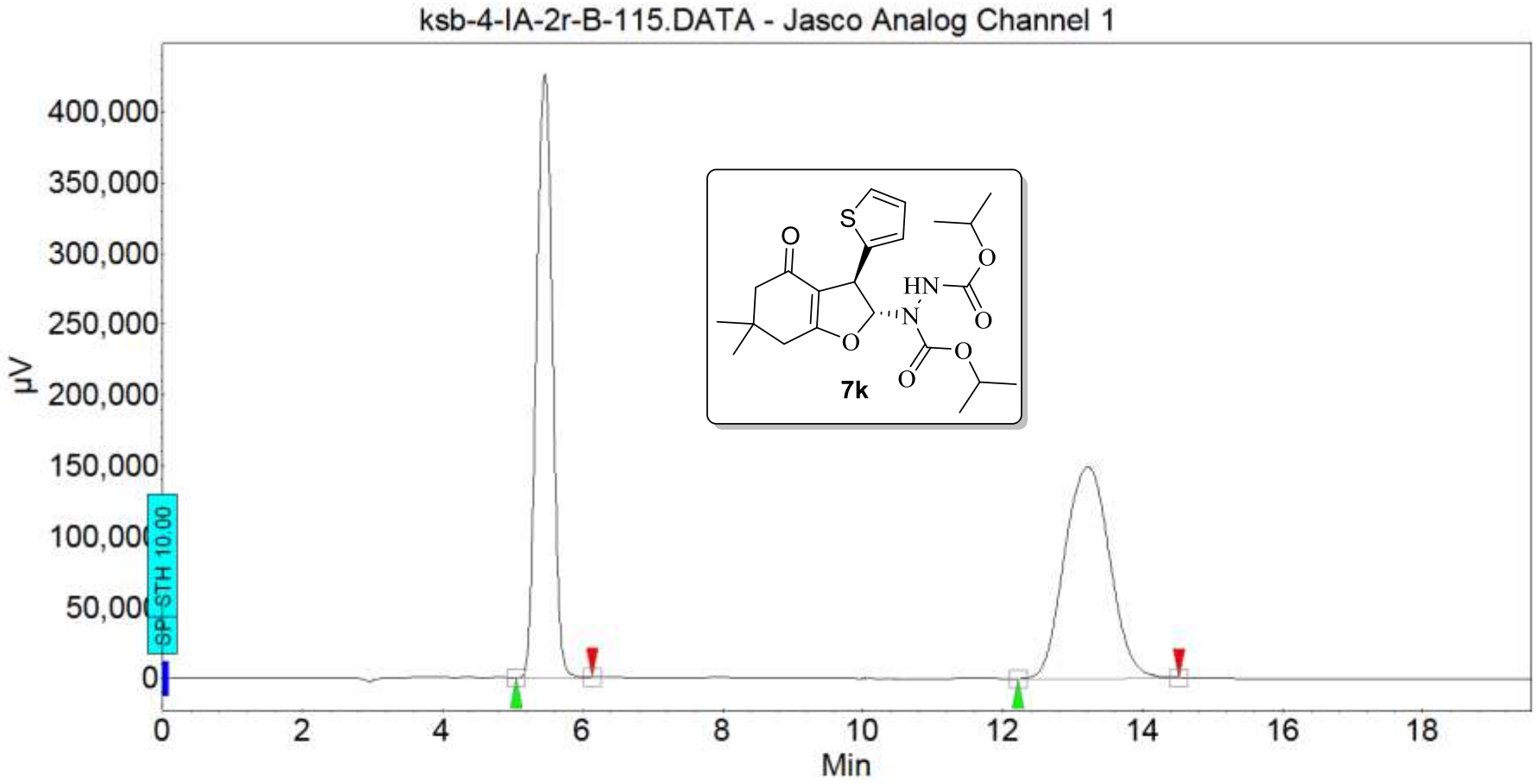


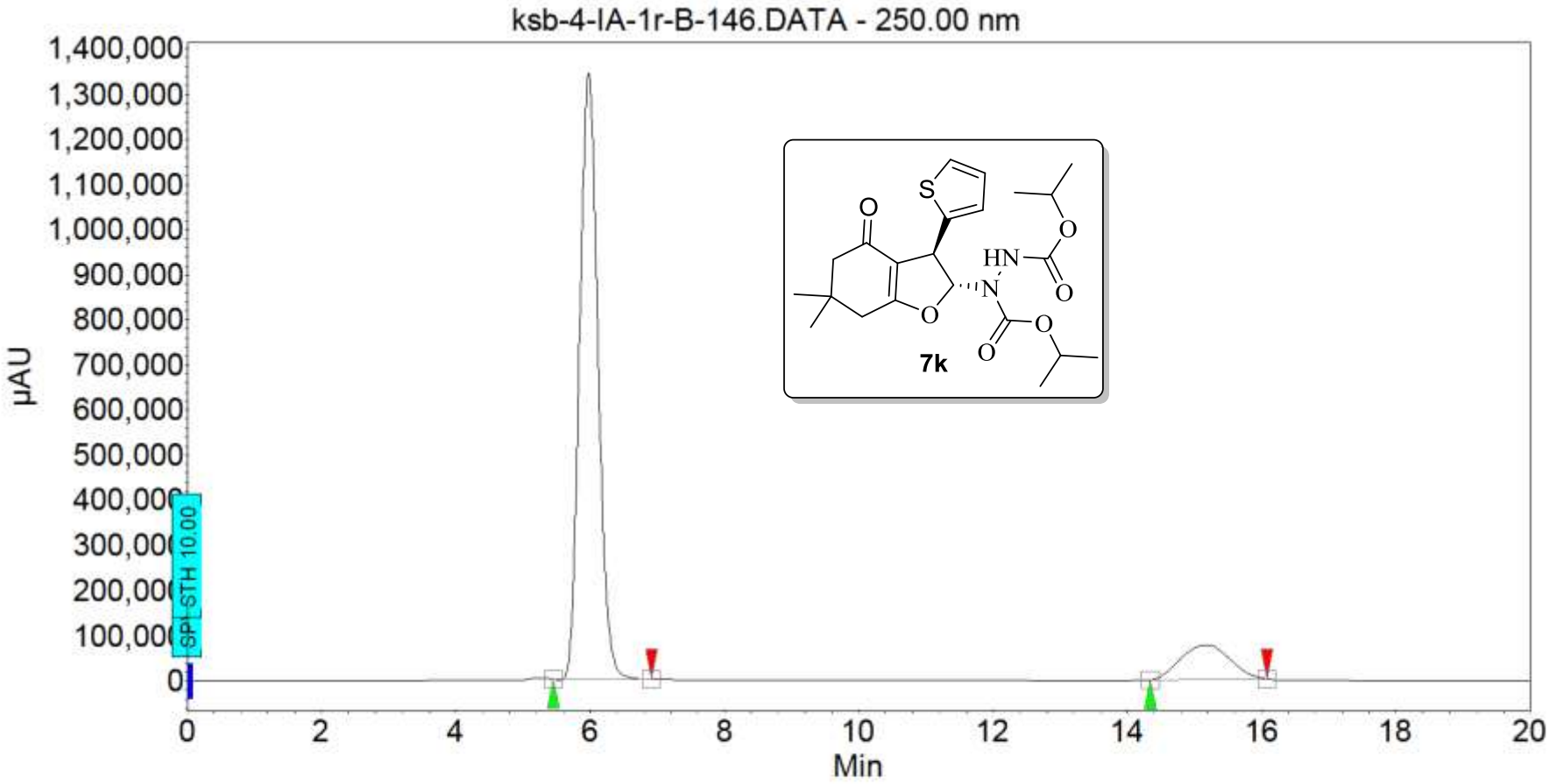
Fig S76. ^{13}C NMR Spectrum of 7k



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	5.458	50.29	426615.4	113047.2	50.292
2	UNKNOWN	13.217	49.71	149274.7	111734.1	49.708
Total			100.00	575890.1	224781.3	100.000

Fig S77. HPLC Profile of Racemic 7k



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	5.973	86.86	1342295.1	425870.4	86.857
2	UNKNOWN	15.172	13.14	76576.9	64444.0	13.143
Total			100.00	1418872.0	490314.3	100.000

Fig S78. HPLC Profile of Enantioenriched 7k

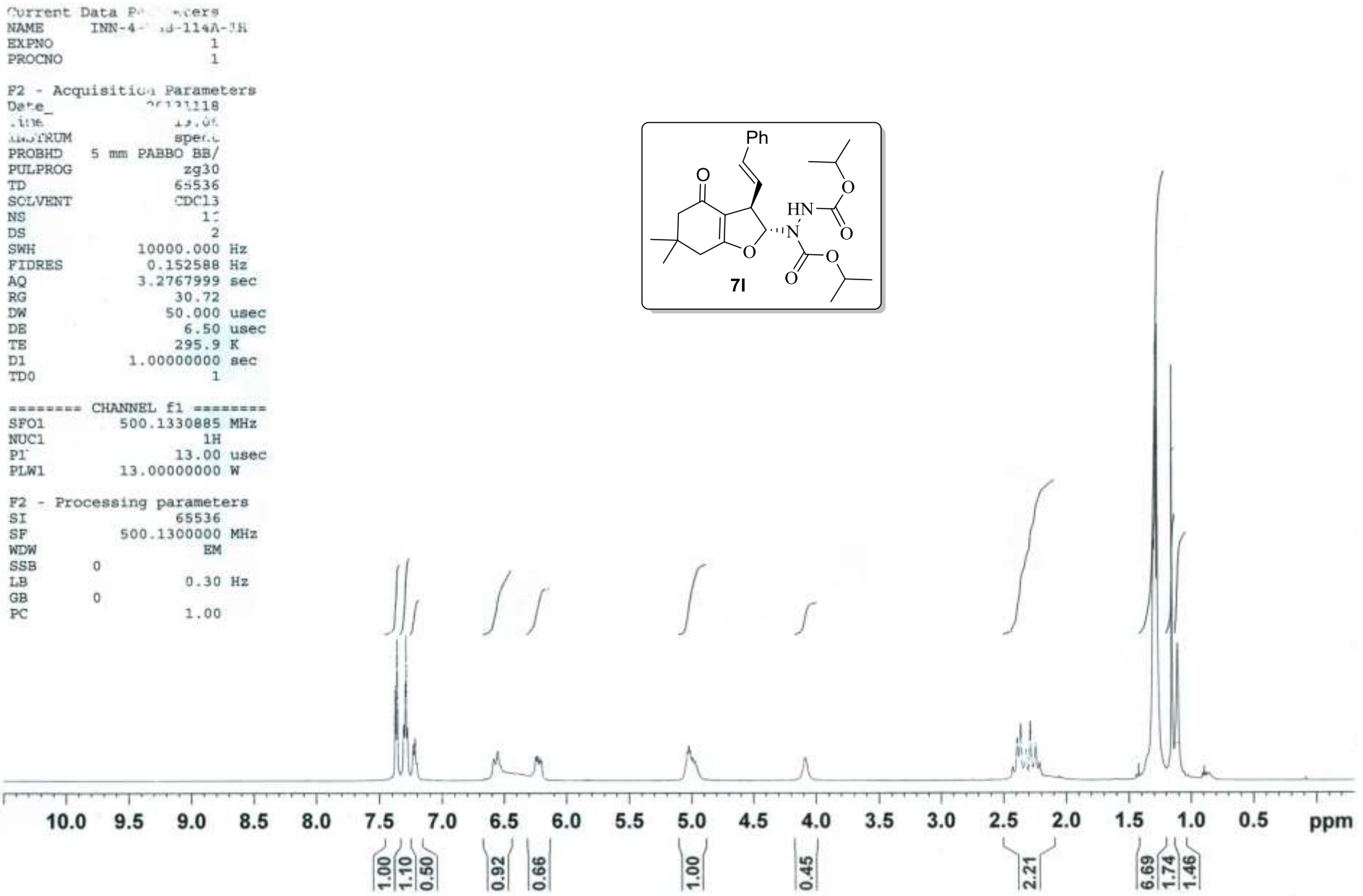


Fig S79. ^{1}H NMR Spectrum of 7l

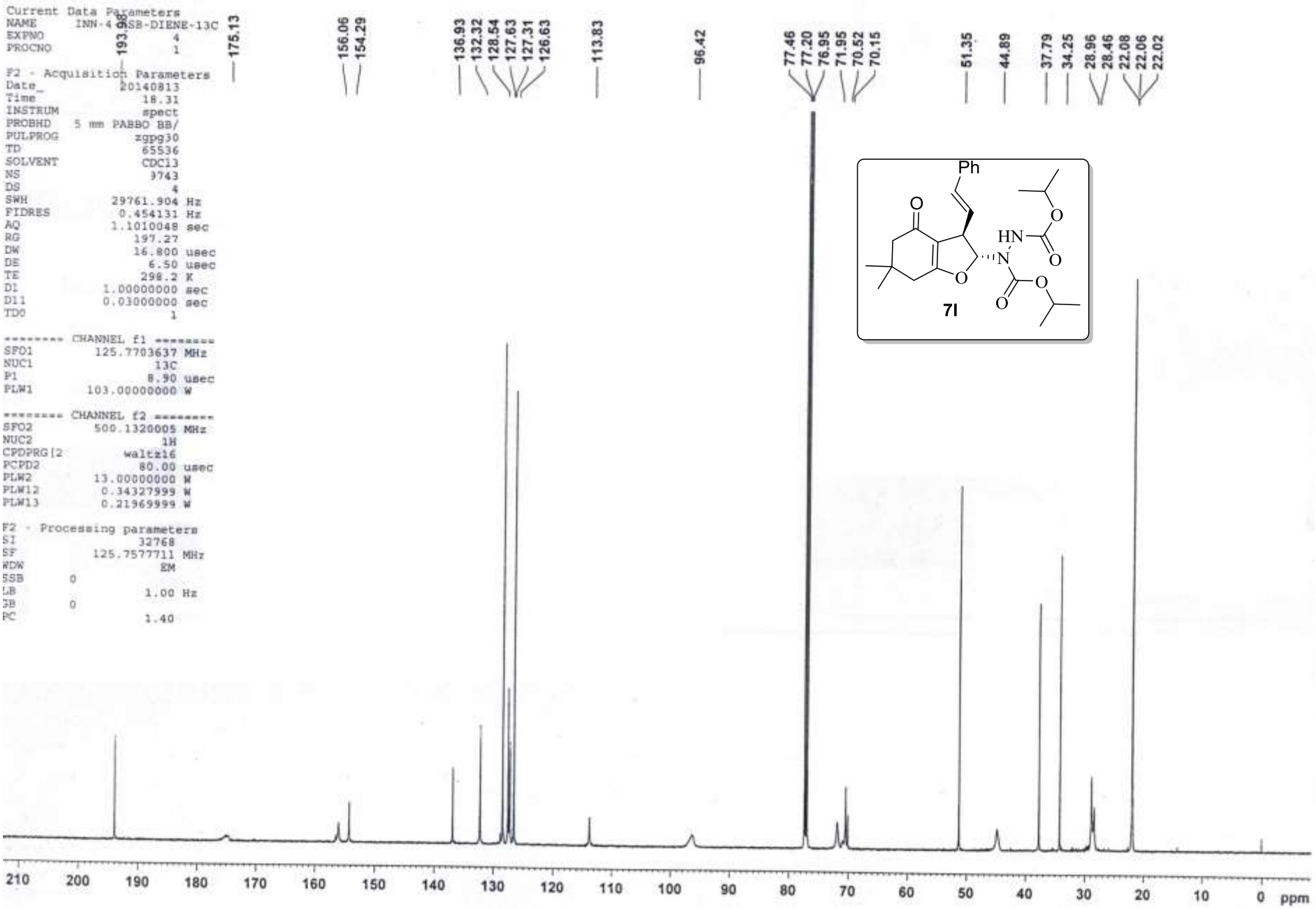
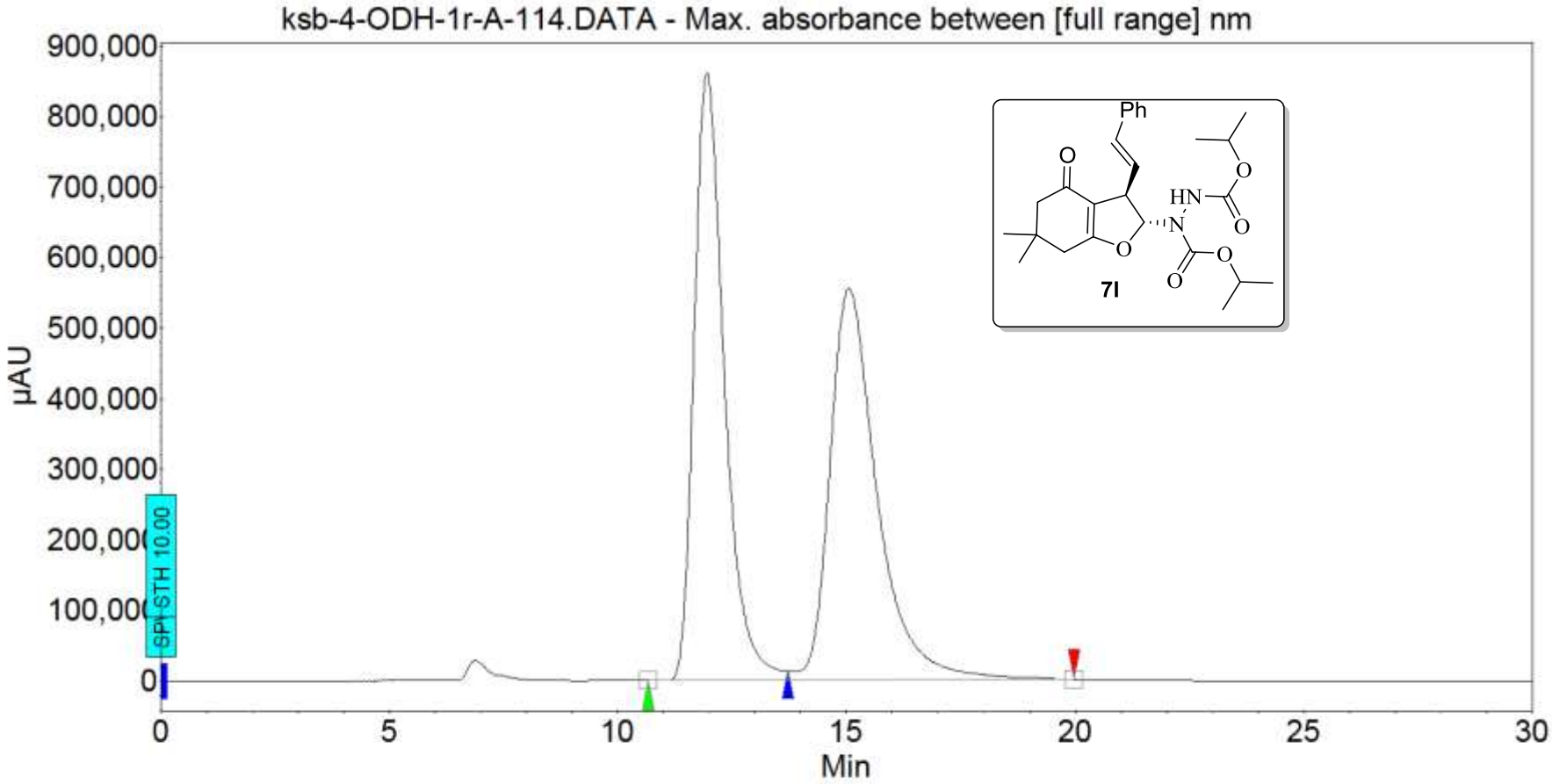


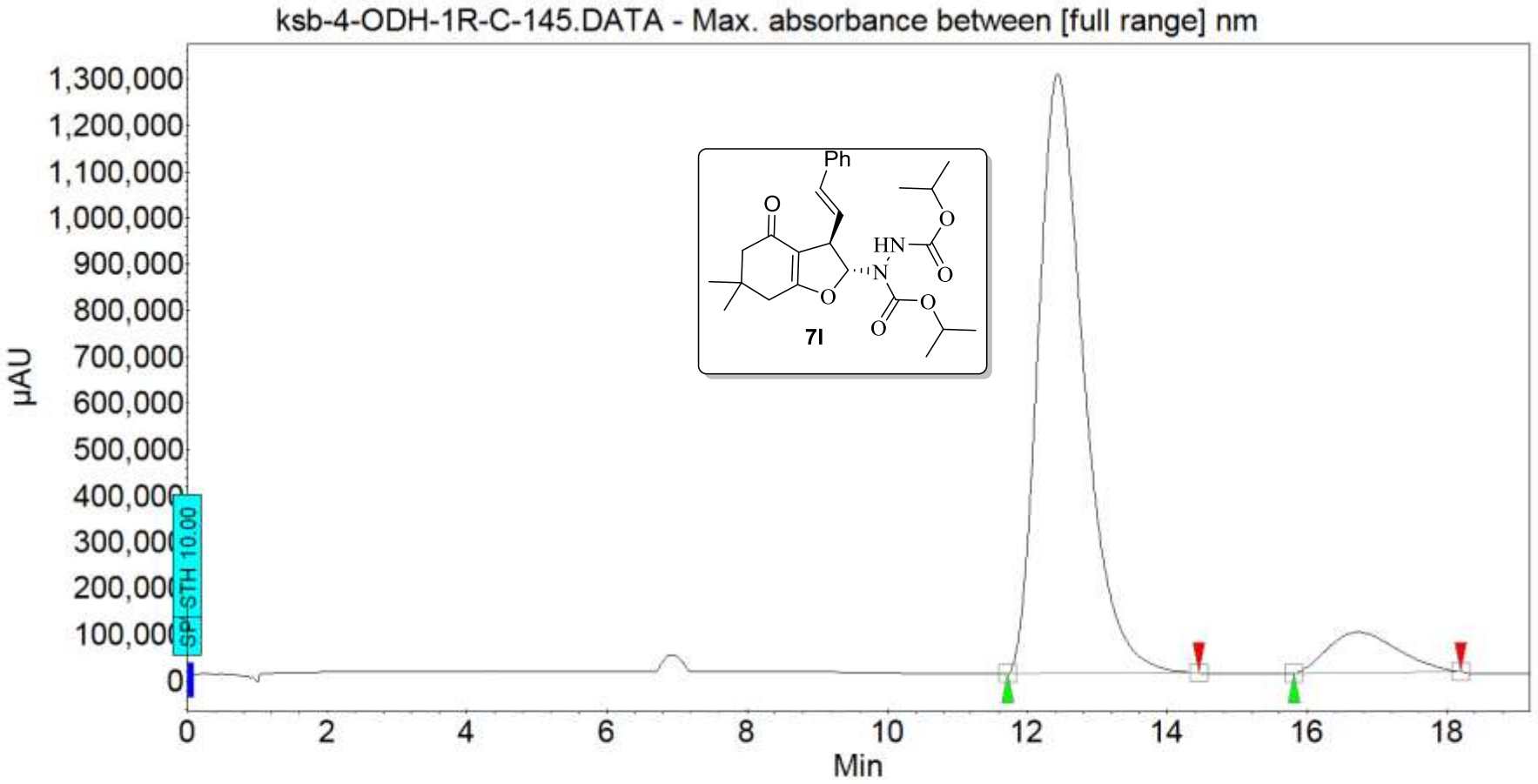
Fig S80. ^{13}C NMR Spectrum of 7l



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	11.932	49.79	860551.1	661218.7	49.795
2	UNKNOWN	15.052	50.21	554531.7	666669.3	50.205
Total			100.00	1415082.8	1327888.0	100.000

Fig S81. HPLC Profile of Racemic 7l



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	12.439	90.44	1294316.1	972003.7	90.443
2	UNKNOWN	16.719	9.56	88126.0	102713.1	9.557
Total			100.00	1382442.1	1074716.8	100.000

Fig S82. HPLC Profile of Enantioenriched 7l

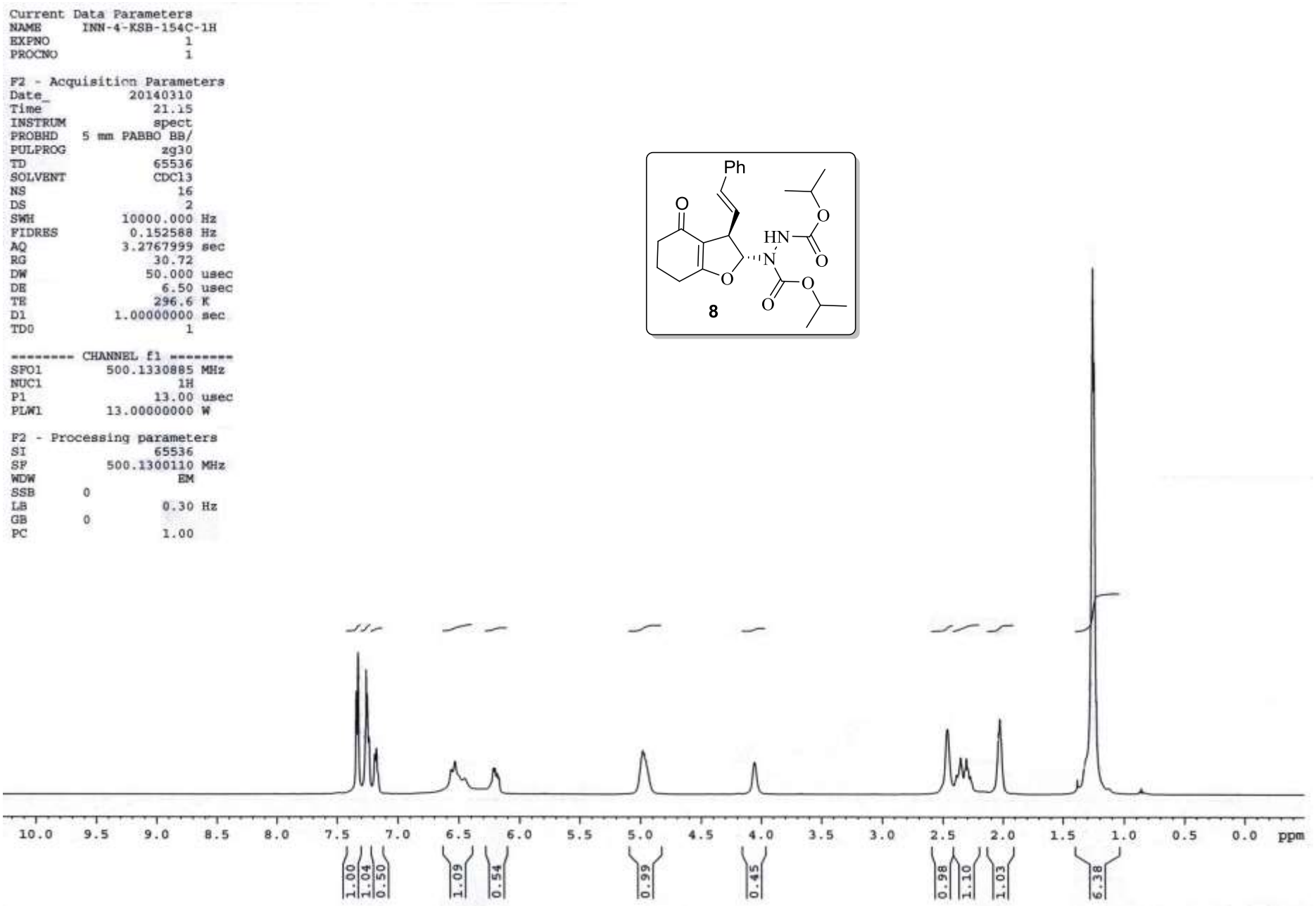


Fig S83. ^{1}H NMR Spectrum of 8

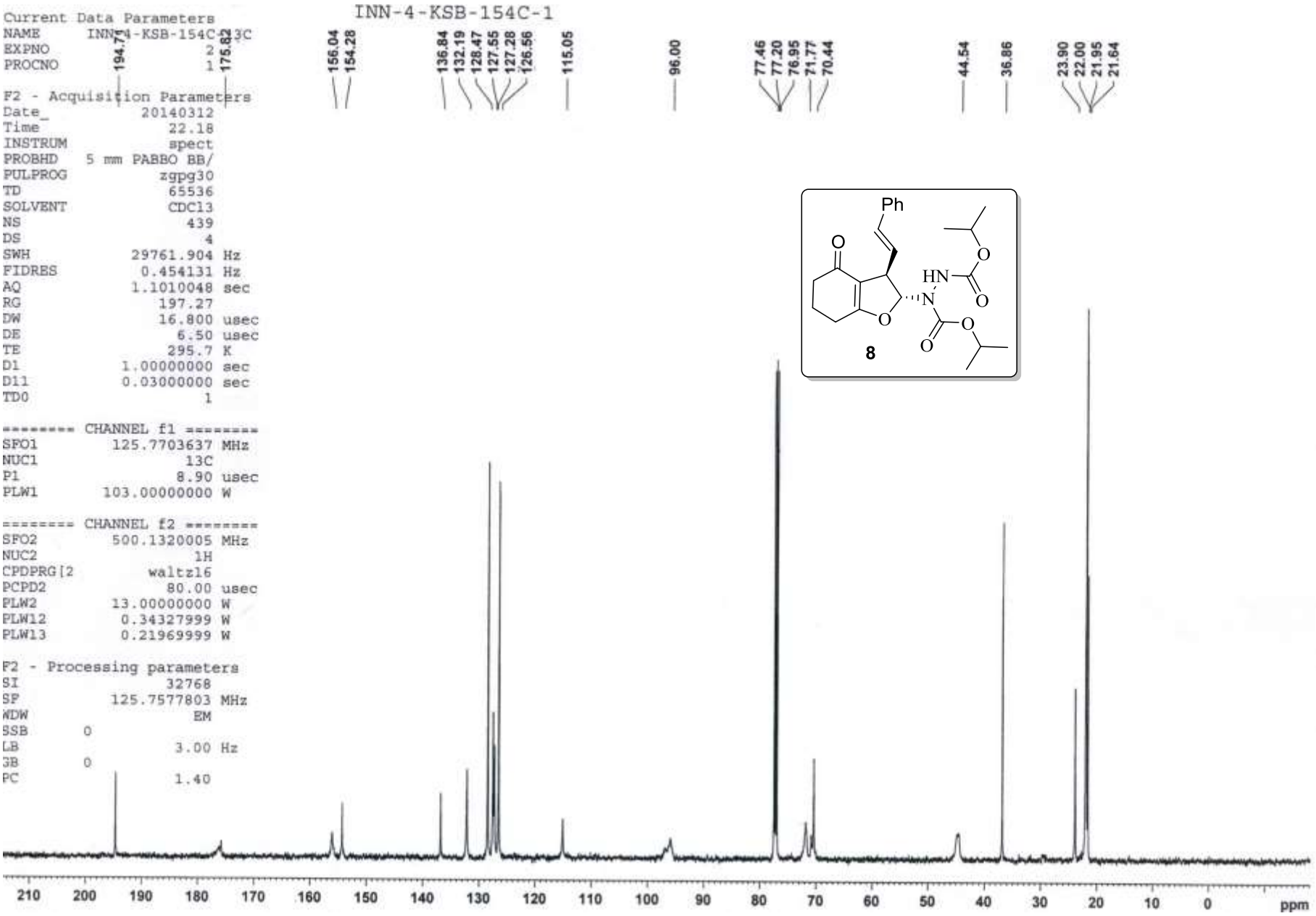
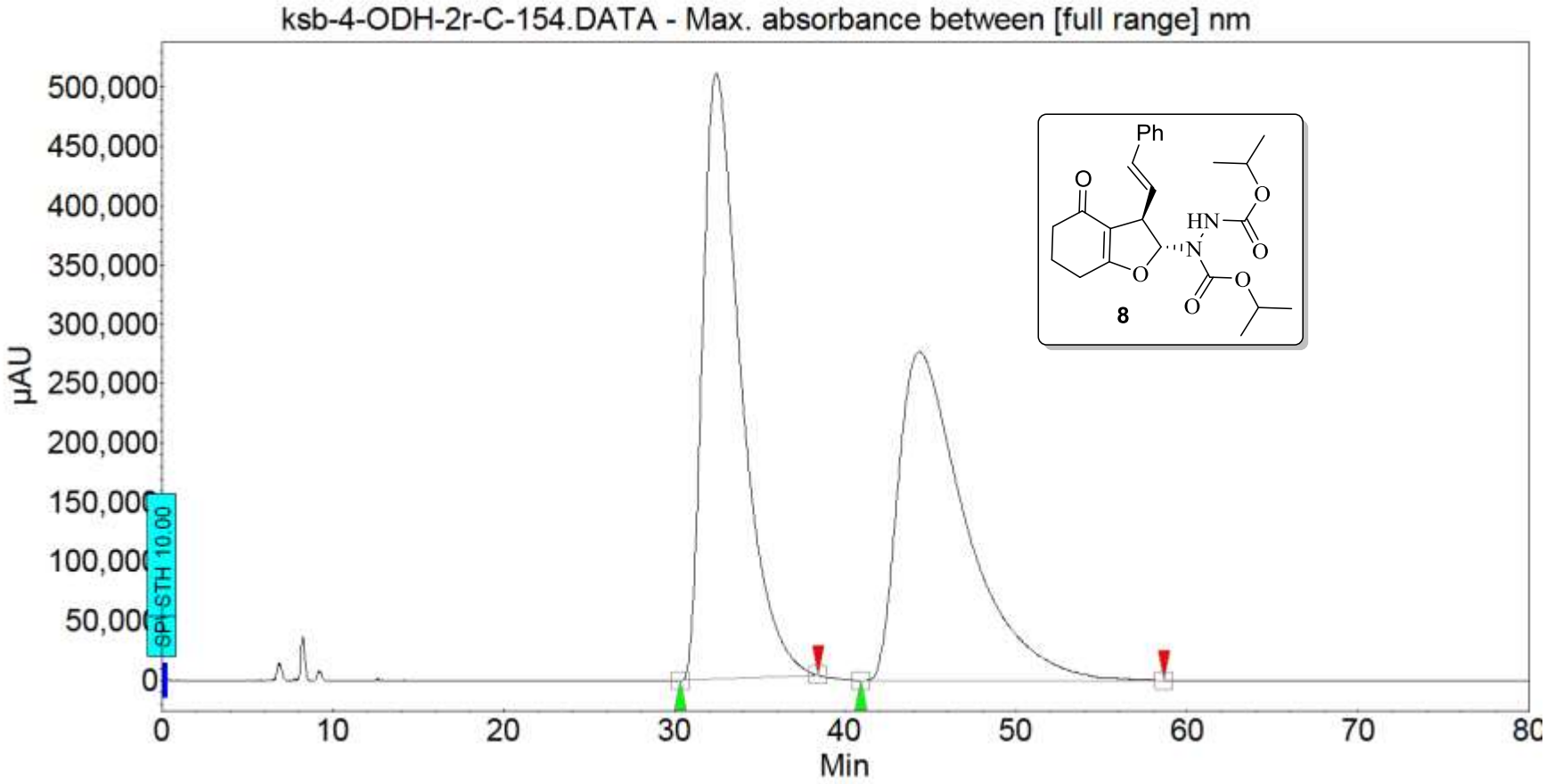


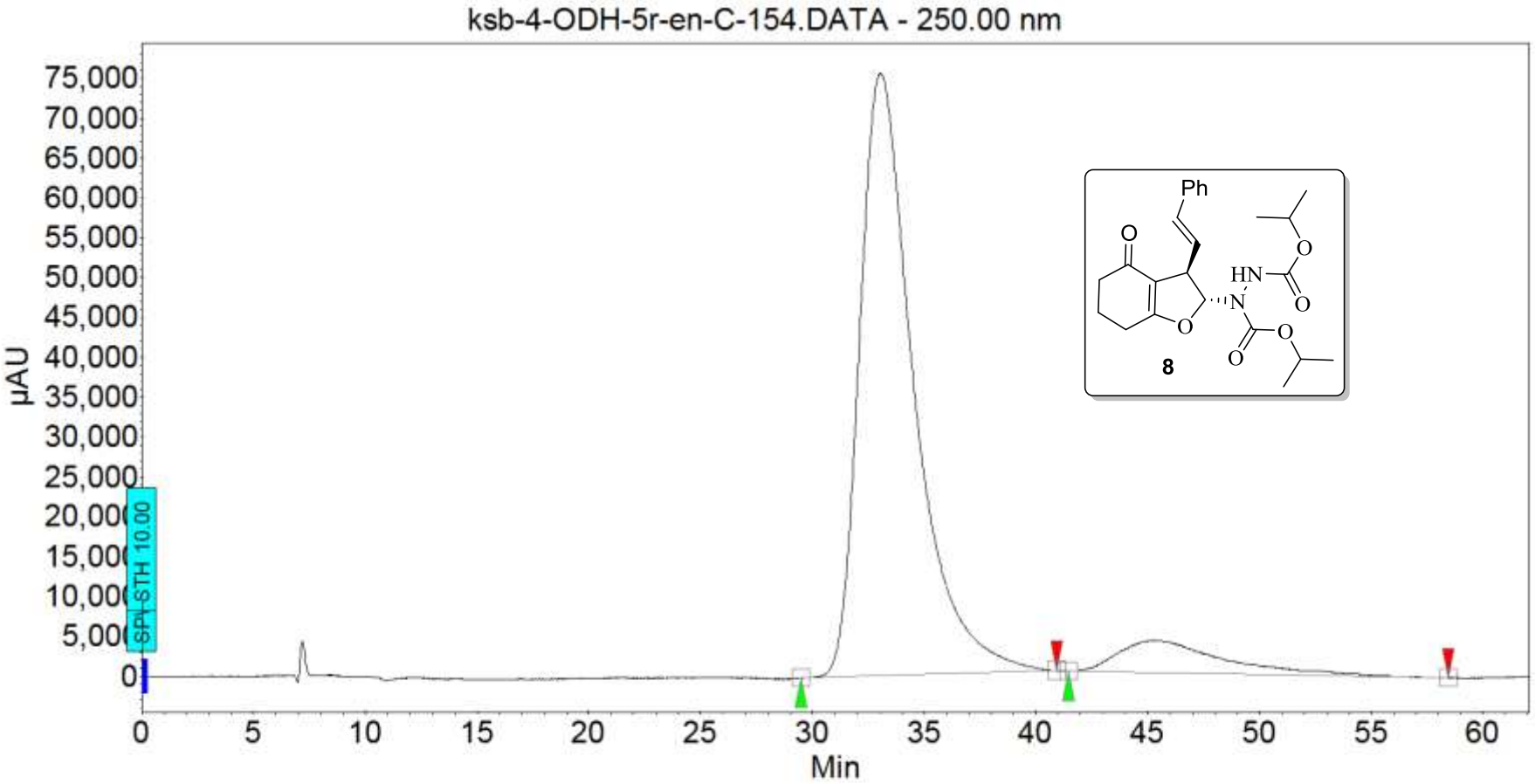
Fig S84. ^{13}C NMR Spectrum of 8



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	32.423	50.99	511411.4	1322305.6	50.992
2	UNKNOWN	44.305	49.01	277021.9	1270849.0	49.008
Total			100.00	788433.2	2593154.6	100.000

Fig S85. HPLC Profile of Racemic 8



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	33.038	90.67	75570.7	212677.2	90.668
2	UNKNOWN	45.317	9.33	3988.9	21888.9	9.332
Total			100.00	79559.6	234566.1	100.000

Fig S86. HPLC Profile of Enantioenriched 8

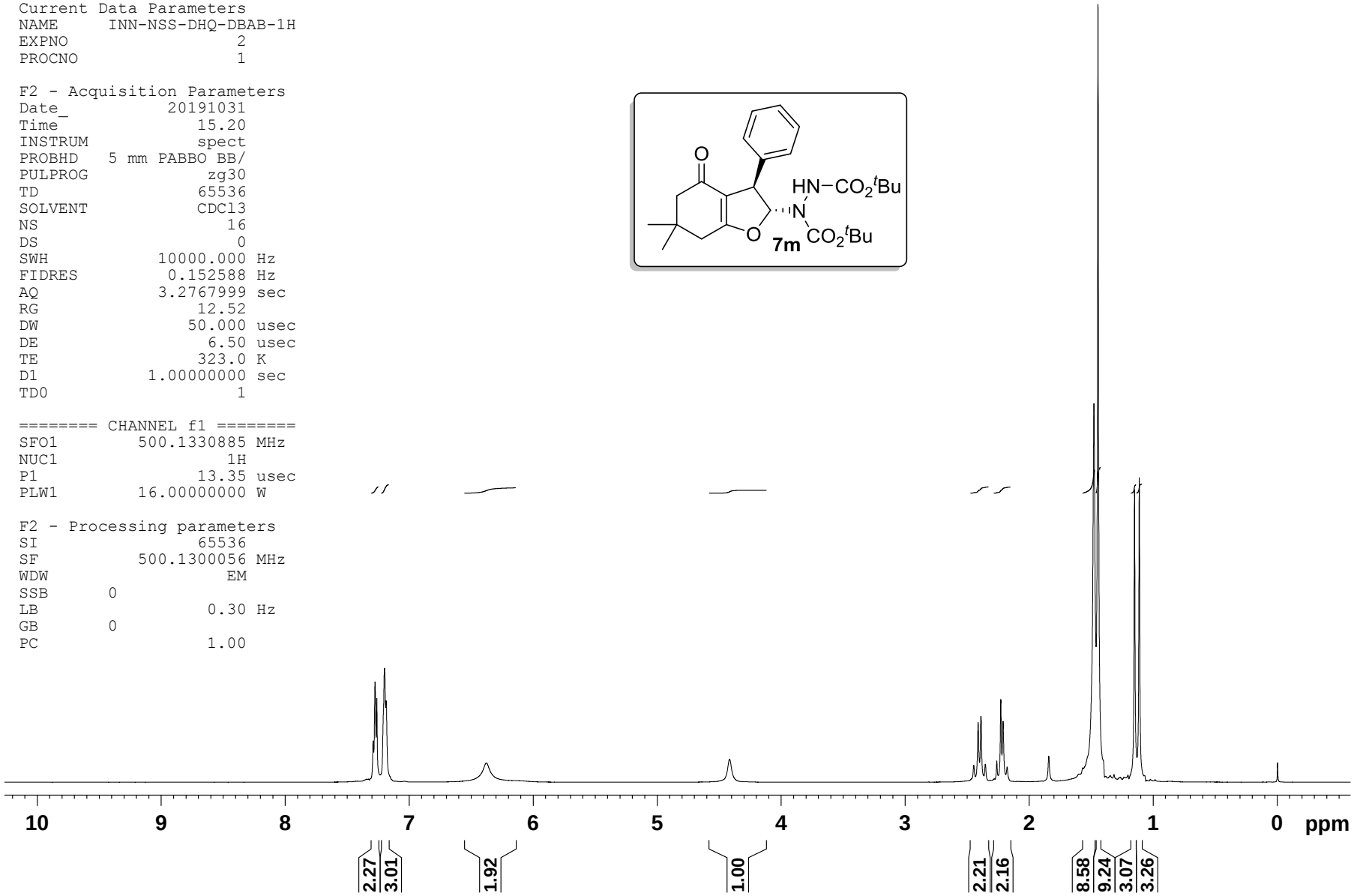


Fig S87. ^{1}H NMR Spectrum of 7m

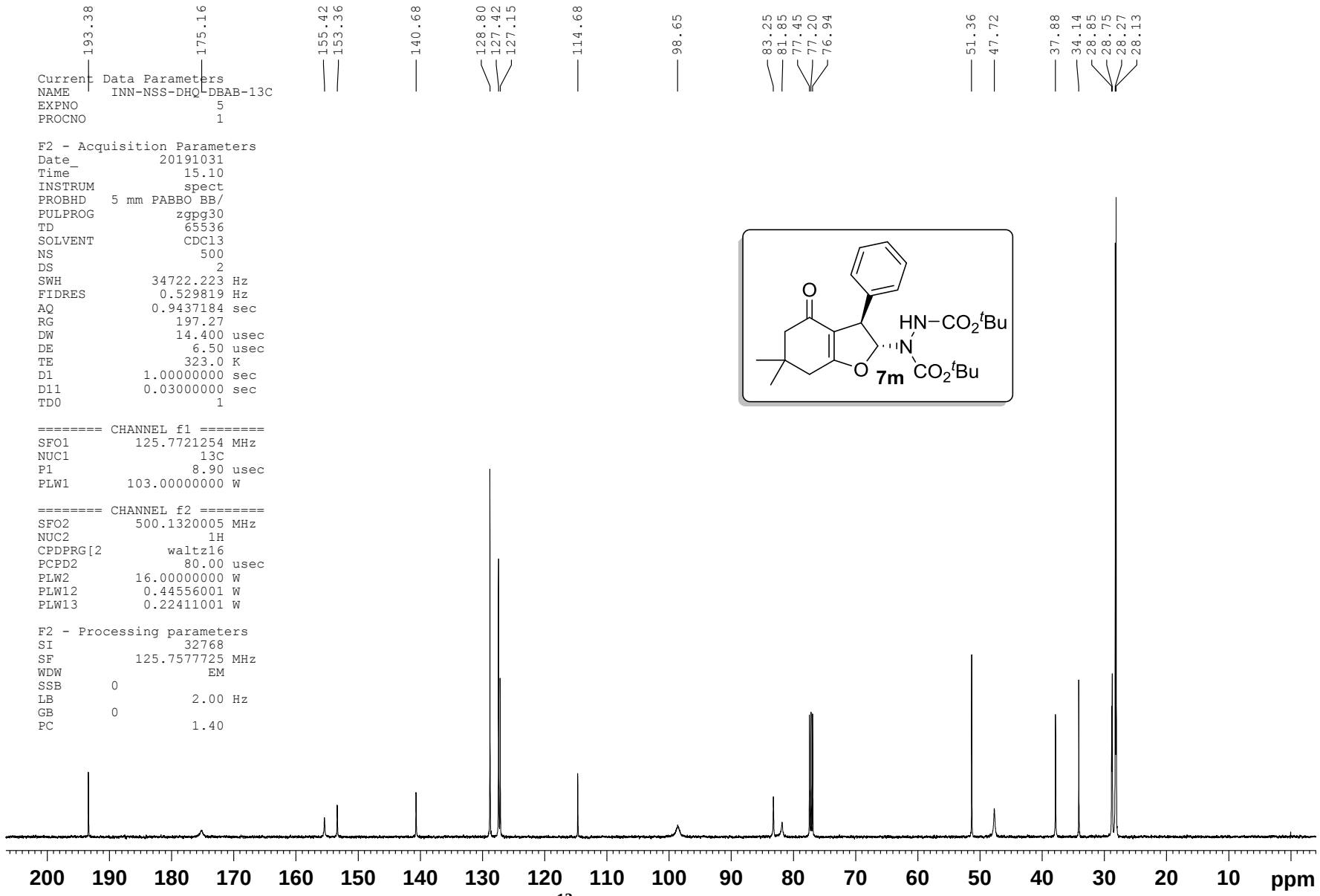
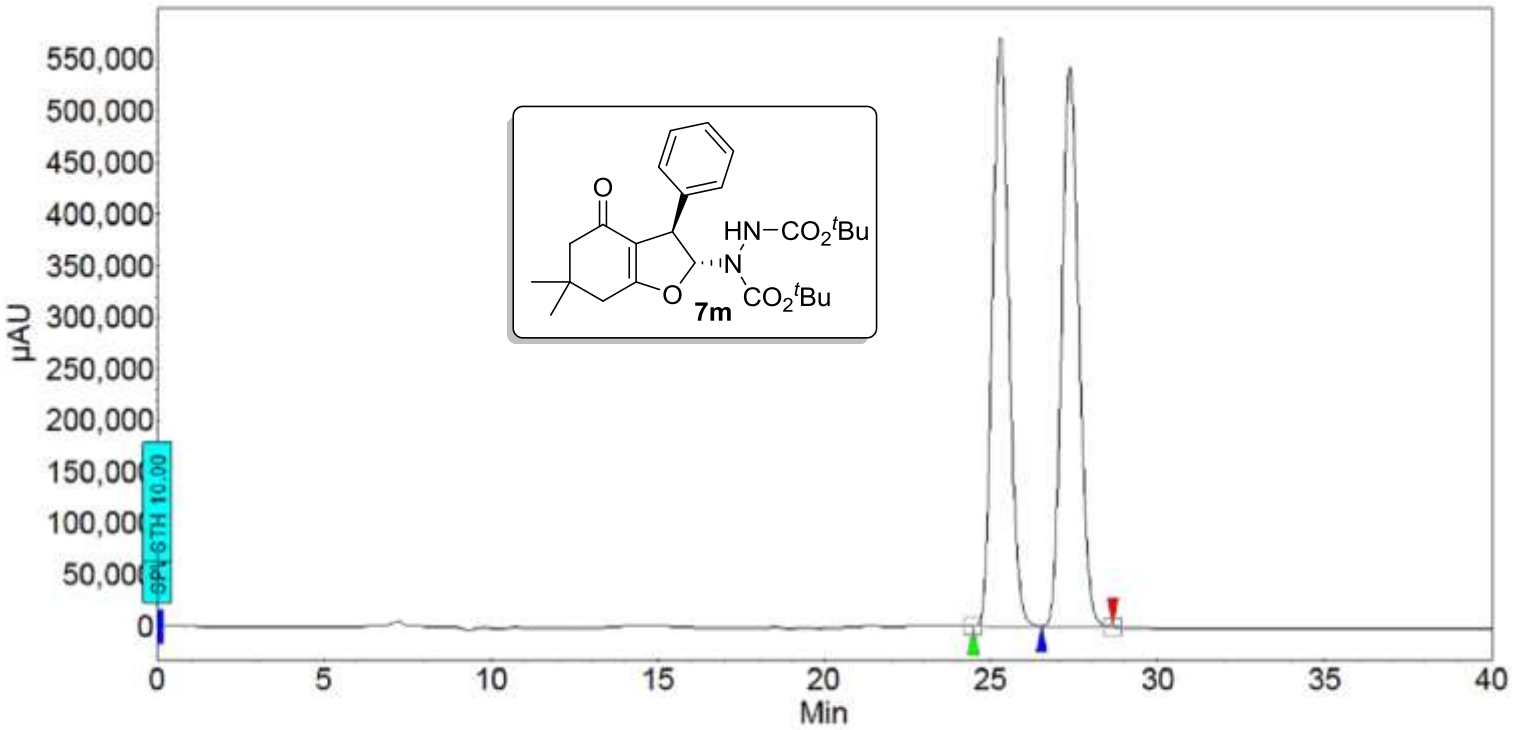


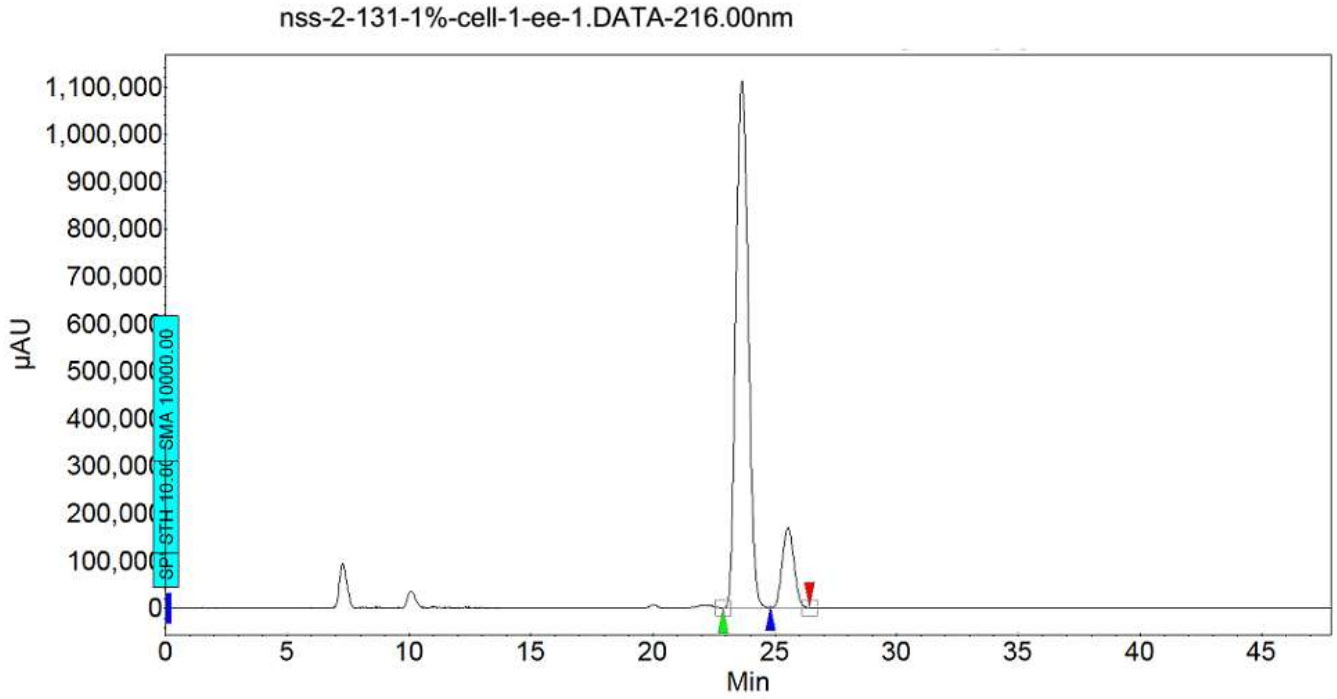
Fig S88. ^{13}C NMR Spectrum of 7m

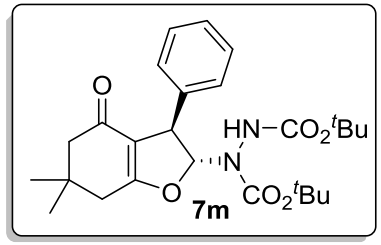


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	25.258	49.97	570998.6	325201.8	49.968
2	UNKNOWN	27.350	50.03	542469.1	325613.6	50.032
Total			100.00	1113467.8	650815.4	100.000

Fig S89. HPLC Profile of Racemic 7m





Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	23.638	87.33	1110902.7	648352.1	87.327
2	UNKNOWN	25.531	12.67	168663.3	94093.7	12.673
Total			100.00	1279566.0	742445.8	100.000

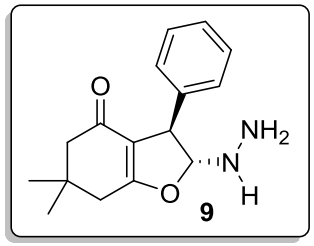
Fig S90. HPLC Profile of Enantioenriched 7m

```
Current Data Parameters
NAME      INN-NSS-DHQ-tBu-Hy-B-1H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191030
Time                2.18
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            10000.000 Hz
FIDRES          0.152588 Hz
AQ             3.2767999 sec
RG                 30.72
DW                50.000 usec
DE                 6.50 usec
TE                 295.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                  1H
P1                 13.35 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300153 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



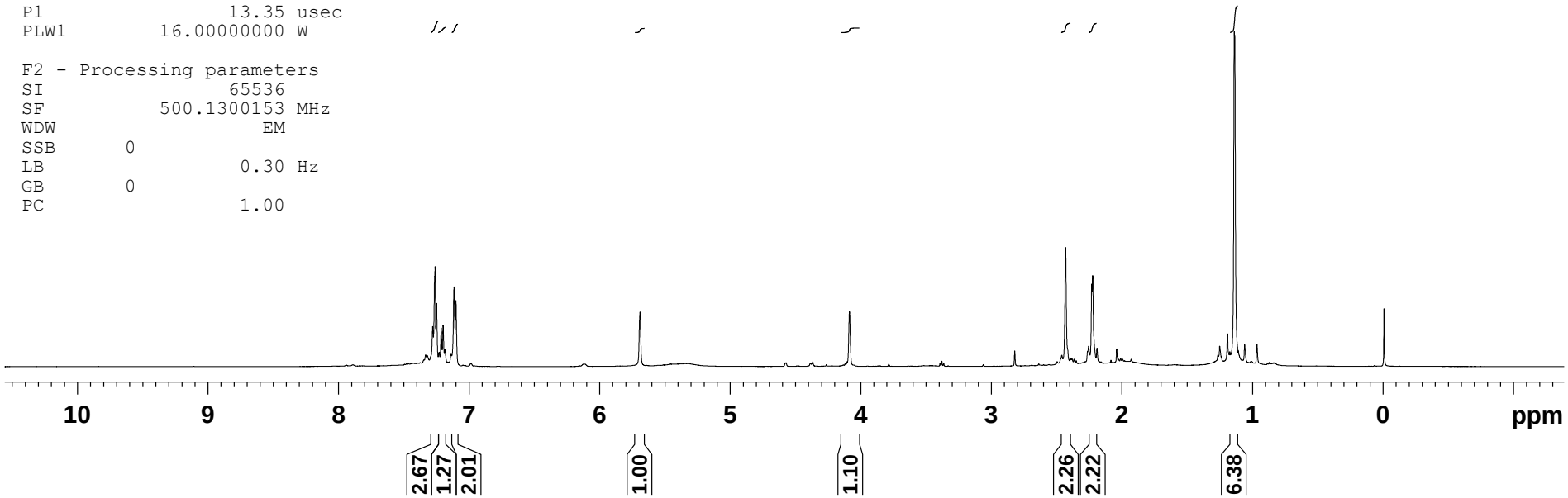


Fig S91. ^{1}H NMR Spectrum of 9

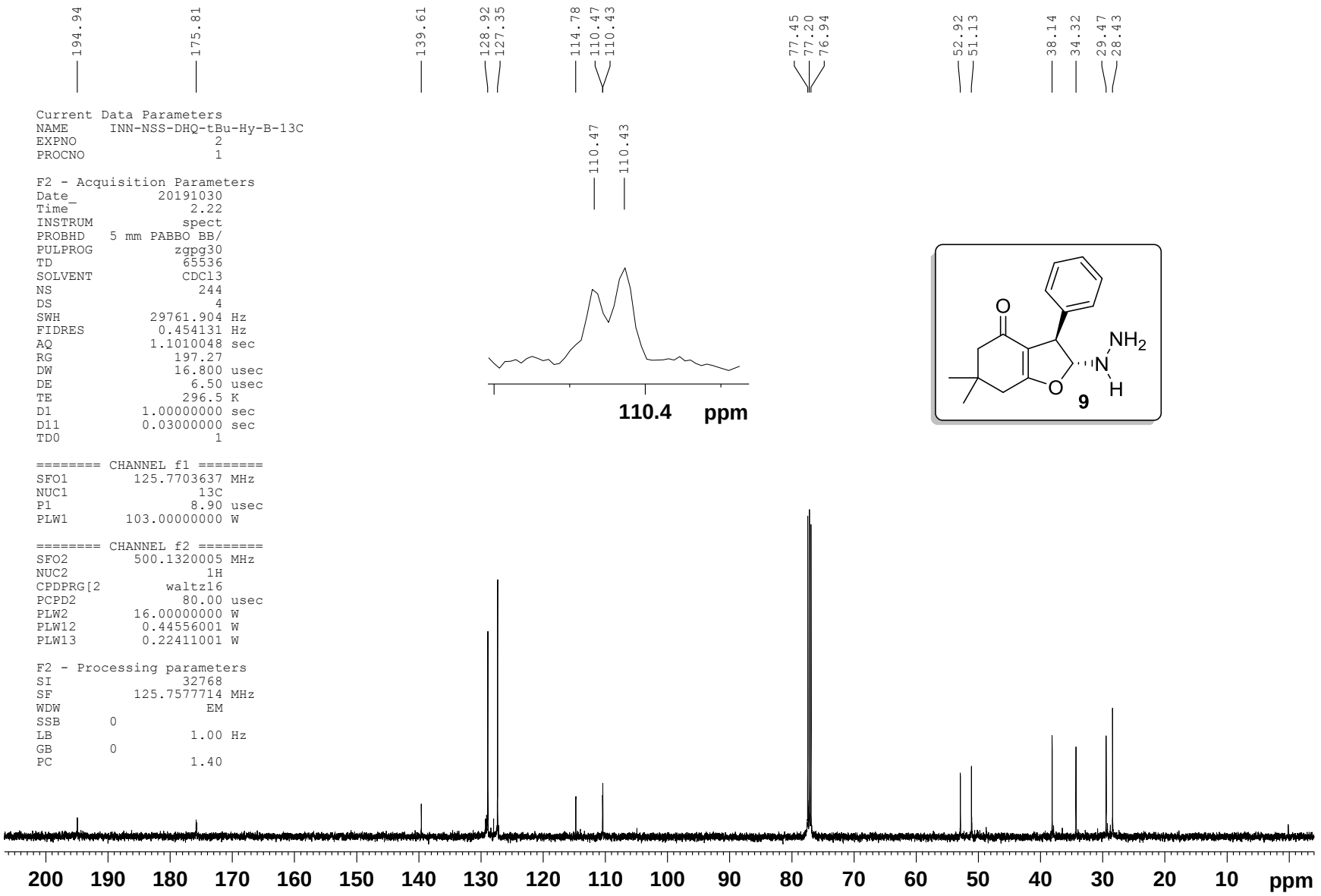


Fig S92. ^{13}C NMR Spectrum of 9

System : hplc
Method : nss-5%-05f-268nm
User : User 1

Accquired : 11/4/2019 6:58:21 PM
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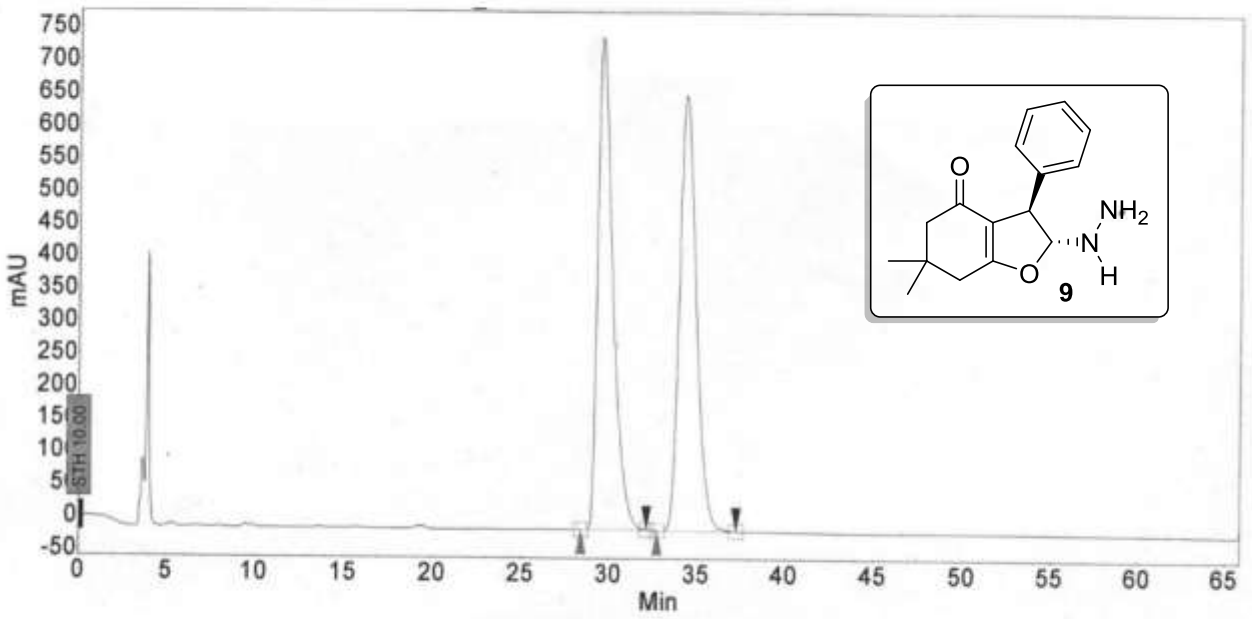
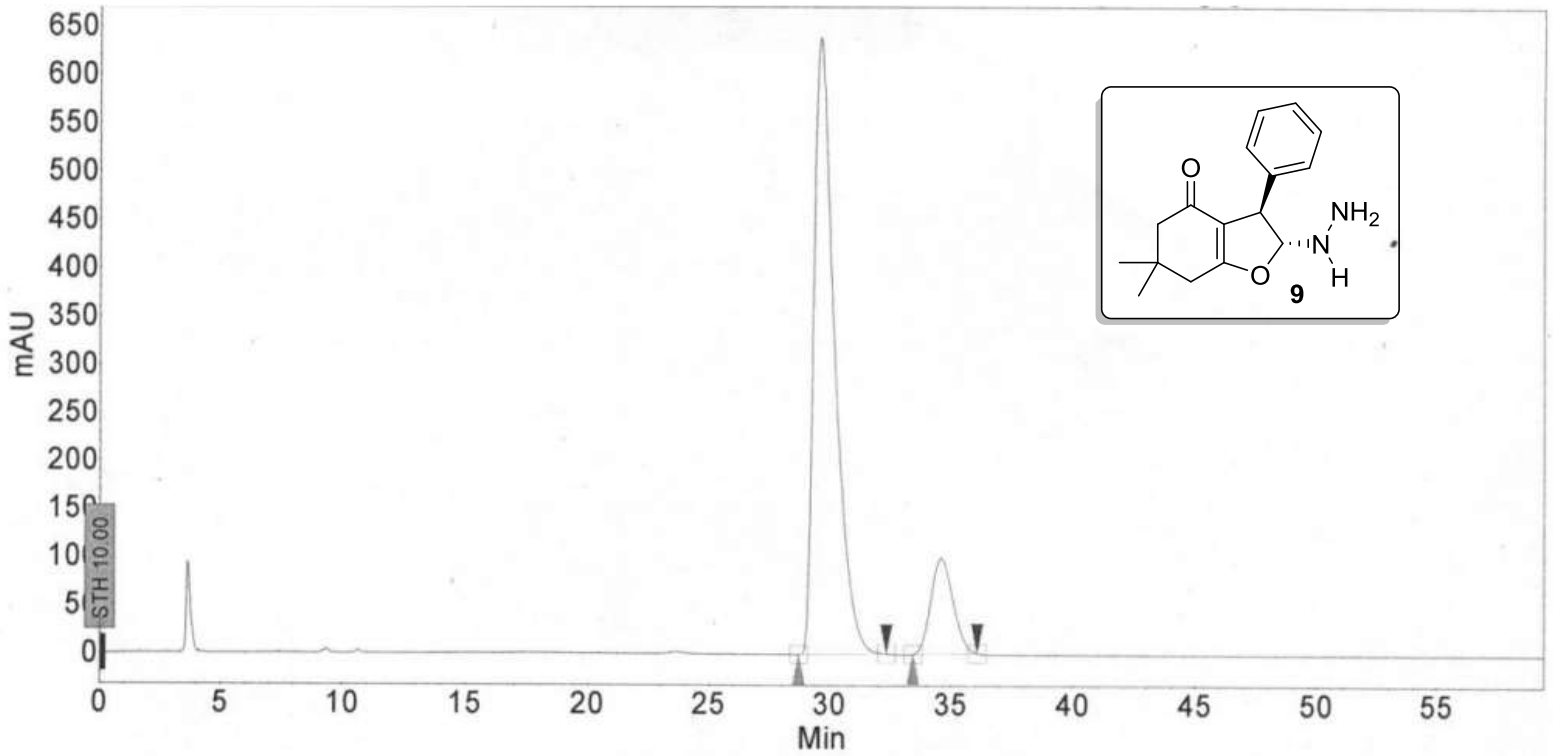


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	29.536	50.32	754.3	749.1	50.323
2	UNKNOWN	34.321	49.68	667.7	739.5	49.677
Total			100.00	1422.0	1488.6	100.000

Fig S93. HPLC Profile of Racemic 9

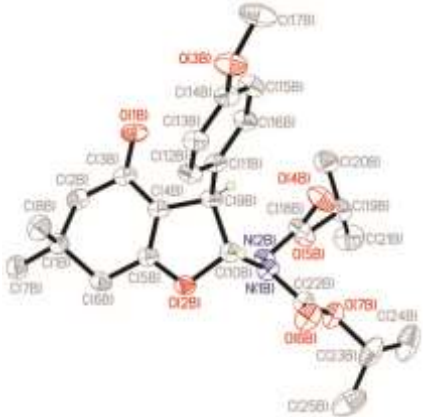


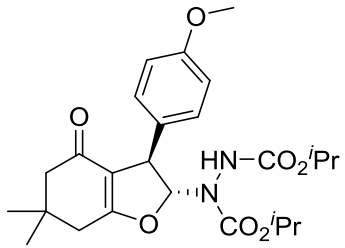
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	29.571	85.80	639.1	634.4	85.803
2	UNKNOWN	34.571	14.20	99.5	105.0	14.197
Total			100.00	738.6	739.3	100.000

Fig S94. HPLC Profile of Enantioenriched 9

Table S1. Crystal data and structure refinement for compound 7c





Crystal data	
Chemical formula	$C_{25}H_{34}N_2O_7 \cdot C_{25}H_{33}NO_8$
M_r	950.06
Crystal system, space group	Triclinic, *P*1
Temperature (K)	293
a, *b*, *c* (Å)	10.4124 (3), 11.3067 (3), 11.3573 (3)
α, β, γ (°)	91.460 (2), 98.011 (2), 104.143 (2)
V (Å^3)	1281.45 (6)
Z	1
Radiation type	Cu *K*α
μ (mm^{-1})	0.75
Crystal size (mm)	0.20 × 0.06 × 0.04
Data collection	
Diffractometer	Dtrek-*CrysAlis PRO*-abstract goniometer imported rigaku-*D*TREK* images diffractometer
Absorption correction	Multi-scan *CrysAlis PRO* 1.171.38.43 (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics,

	implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.663, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	43857, 7928, 7474
R_{int}	0.113
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.588
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.179, 1.08
No. of reflections	7928
No. of parameters	627
No. of restraints	3
H-atom treatment	H-atom parameters constrained
$\Delta\rangle_{max}$, $\Delta\rangle_{min}$ (e Å^{-3})	0.65, -0.47
Absolute structure	Flack x determined using 3030 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	0.0 (2)

$C_{25}H_{34}N_2O_7 \cdot C_{25}H_{33}NO_8$	Z = 1
M_r = 950.06	F(000) = 508
Triclinic, $P\overline{1}$	D_x = 1.231 Mg m^{-3}
a = 10.4124 (3) Å	Cu $K\alpha$ radiation, λ = 1.54184 Å
b = 11.3067 (3) Å	Cell parameters from 27030 reflections
c = 11.3573 (3) Å	θ = 3.9–72.7°
α = 91.460 (2)°	μ = 0.75 mm^{-1}
β = 98.011 (2)°	T = 293 K
γ = 104.143 (2)°	Plate, colourless
V = 1281.45 (6) Å^3	0.20 × 0.06 × 0.04 mm

Data collection

Dtrek-CrysAlisPro-abstract goniometer imported rigaku-d*trek images diffractometer	7928 independent reflections
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source	7474 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.113$
ω scans	$\theta_{max} = 65.0°$, $\theta_{min} = 3.9°$
Absorption correction: multi-scan *CrysAlis PRO* 1.171.38.43 (Rigaku Oxford Diffraction, 2015) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	$h = -12 \rightarrow 12$
$T_{min} = 0.663$, $T_{max} = 1.000$	$k = -13 \rightarrow 13$
43857 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.063$	$w = 1/[\sigma^2(F_o^2) + (0.1282P)^2 + 0.2308P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rangle_{max} = 0.65$ e Å^{-3}
7928 reflections	$\Delta\rangle_{min} = -0.47$ e Å^{-3}
627 parameters	Absolute structure: Flack x determined using 3030 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
3 restraints	Absolute structure parameter: 0.0 (2)