

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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NAME INN-AN-4-342-1H
 EXPNO 25
 PROCNO 1
 Date_ 20110703
 Time_ 12.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 295.7 K
 D1 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 13.50 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300118 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

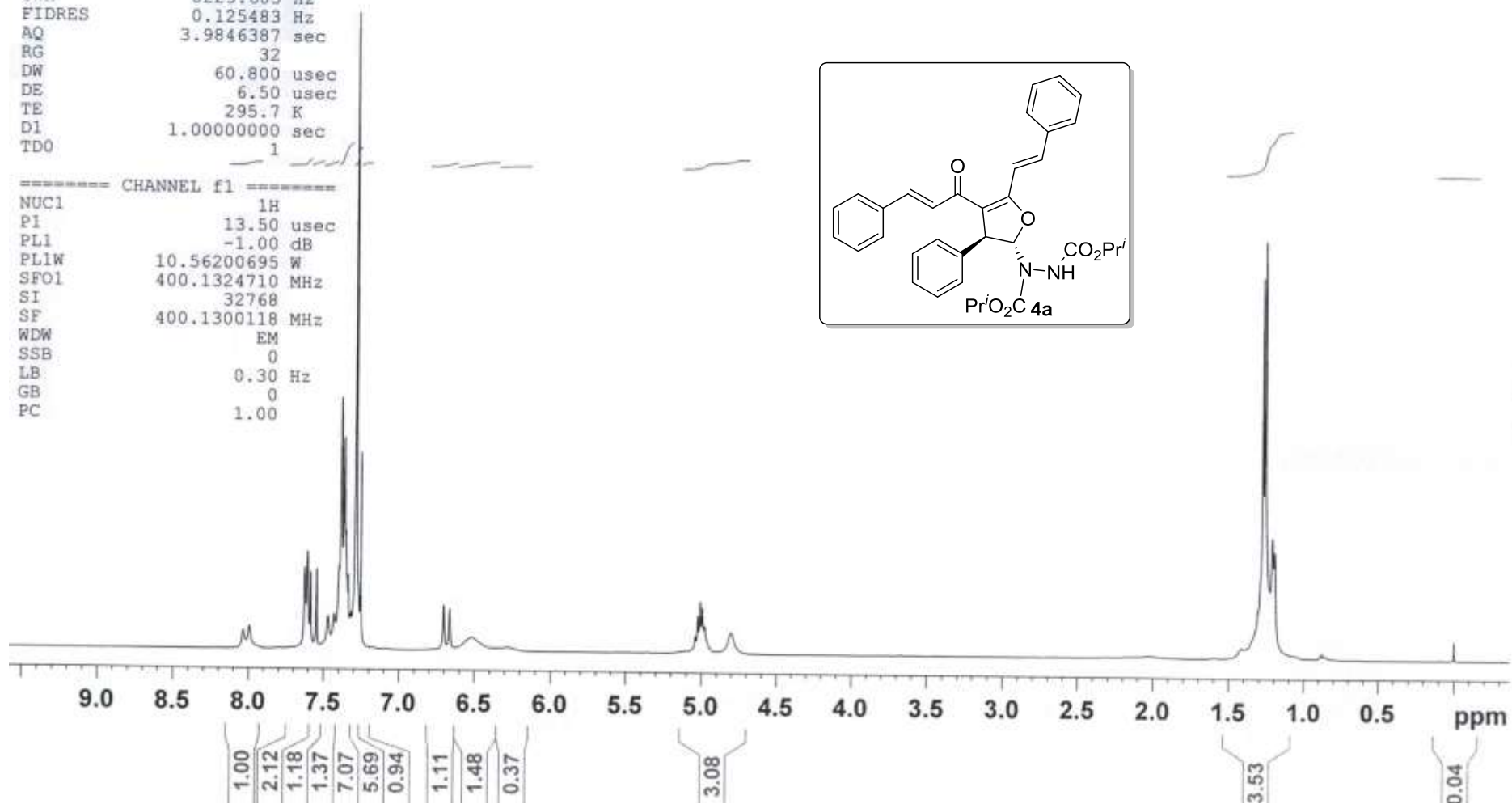
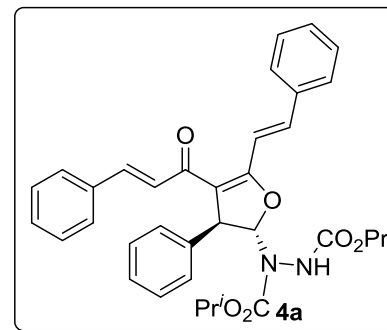
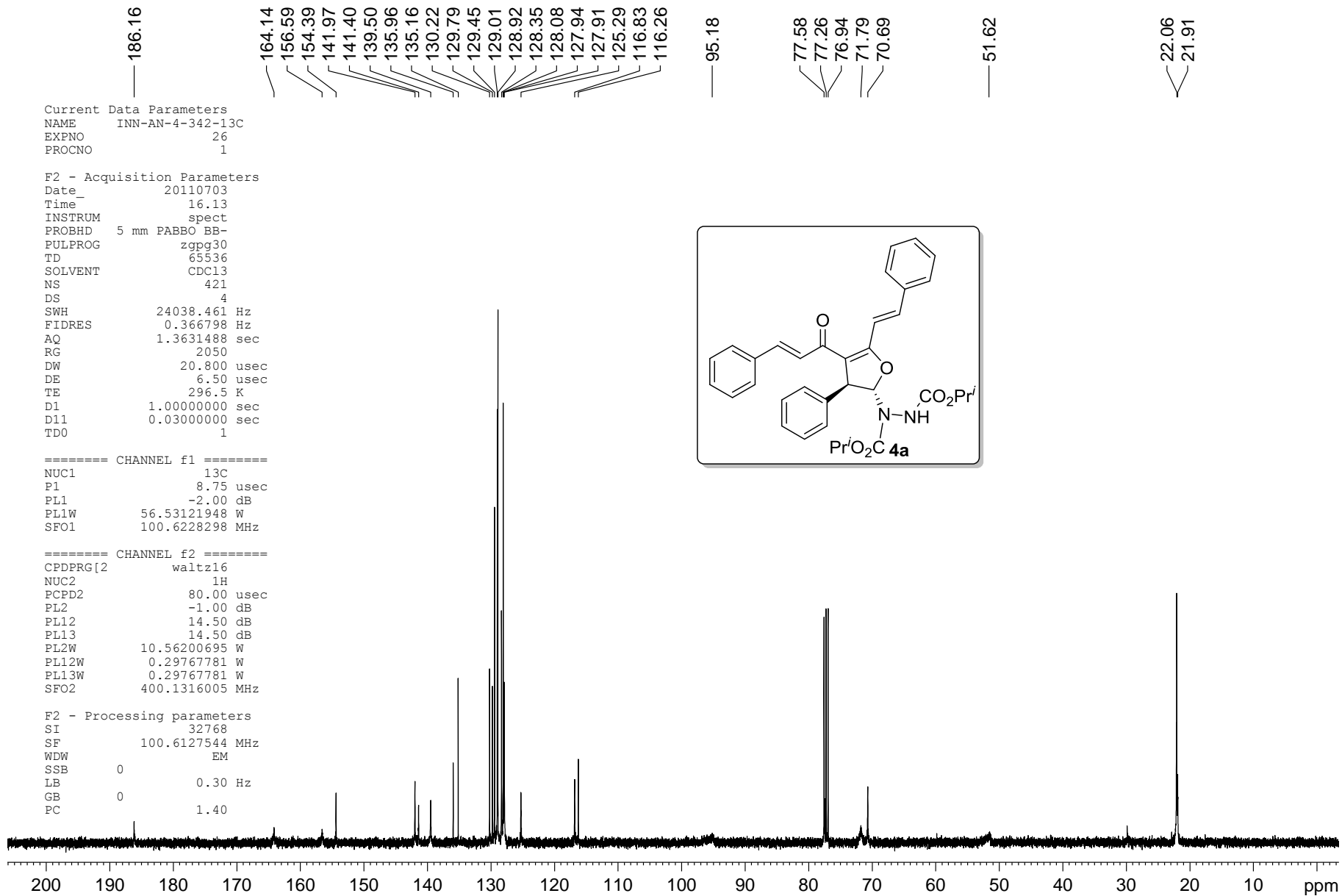


Fig S1. ¹H NMR Spectrum of 4a



```

NAME      INN-AN-4-380-1H
EXPNO     65
PROCNO    1
Date_     20110715
Time      20.34
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        32
DS        0
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        32
DW        60.800 usec
DE        6.50 usec
TE        293.7 K
D1        1.0000000 sec
TD0       1

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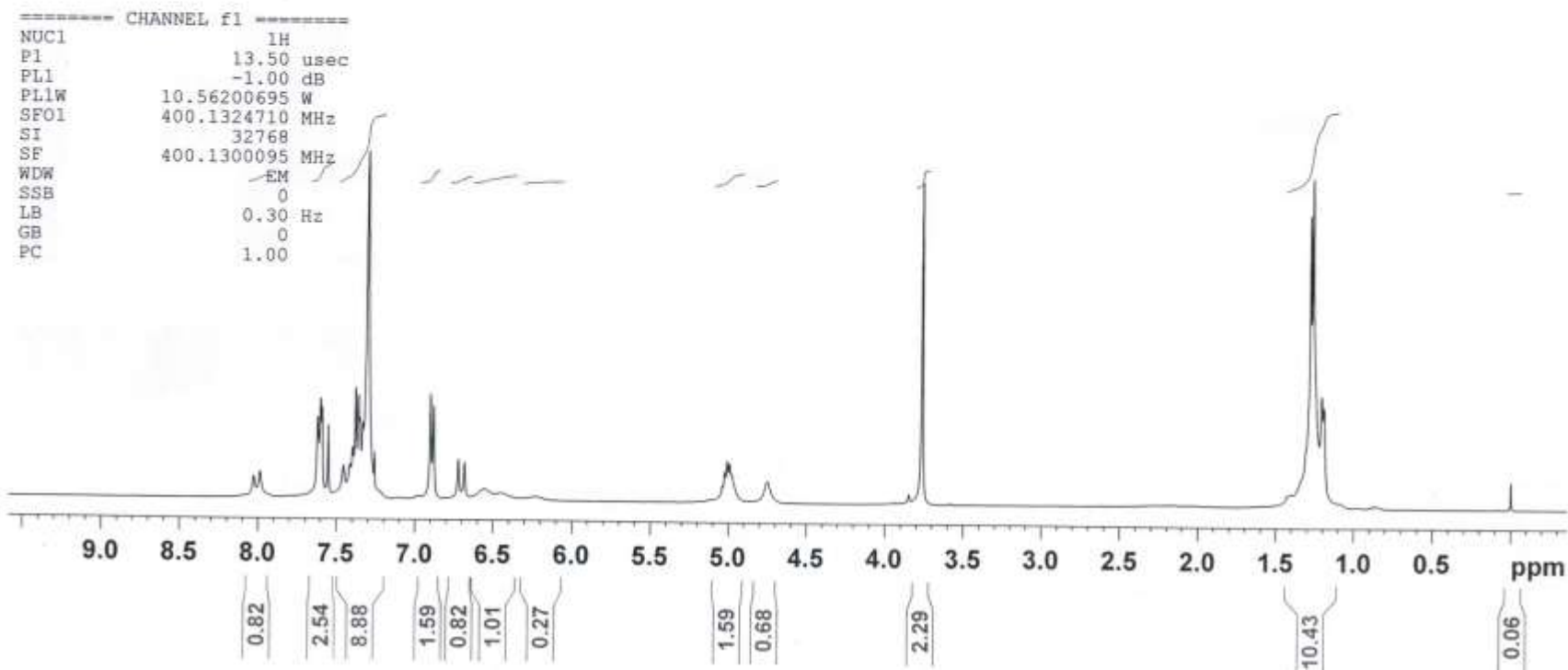
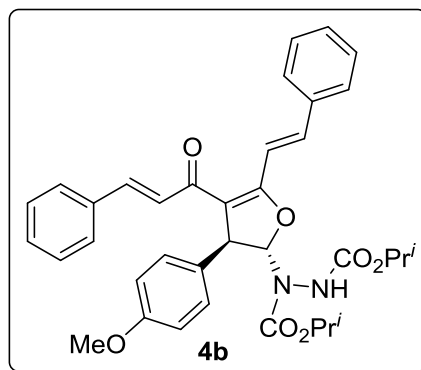


Fig S3. ¹H NMR Spectrum of **4b**

```

NAME      INN-AN-4-380-13C
EXPNO     66
PROCNO    1
Date_     20110715
Time      20.38
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        244
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        293.9 K
D1        1.00000000 sec
D11       0.03000000 sec
TD0       1

----- CHANNEL f1 -----
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      56.53121948 W
SFO1      100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -1.00 dB
PL12      14.50 dB
PL13      14.50 dB
PL2W      10.56200695 W
PL12W     0.29767781 W
PL13W     0.29767781 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127555 MHz
WDW       EM
SSB       0
LB        2.00 Hz
GB        0
PC        1.40

```

186.24
 163.96
 159.22
 156.59
 154.42
 141.90
 139.38
 135.95
 135.16
 133.41
 130.21
 129.75
 128.99
 128.92
 128.36
 128.05
 125.24
 116.86
 116.39
 114.79
 95.29
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 71.67
 70.64
 55.41
 50.65
 22.05
 21.89

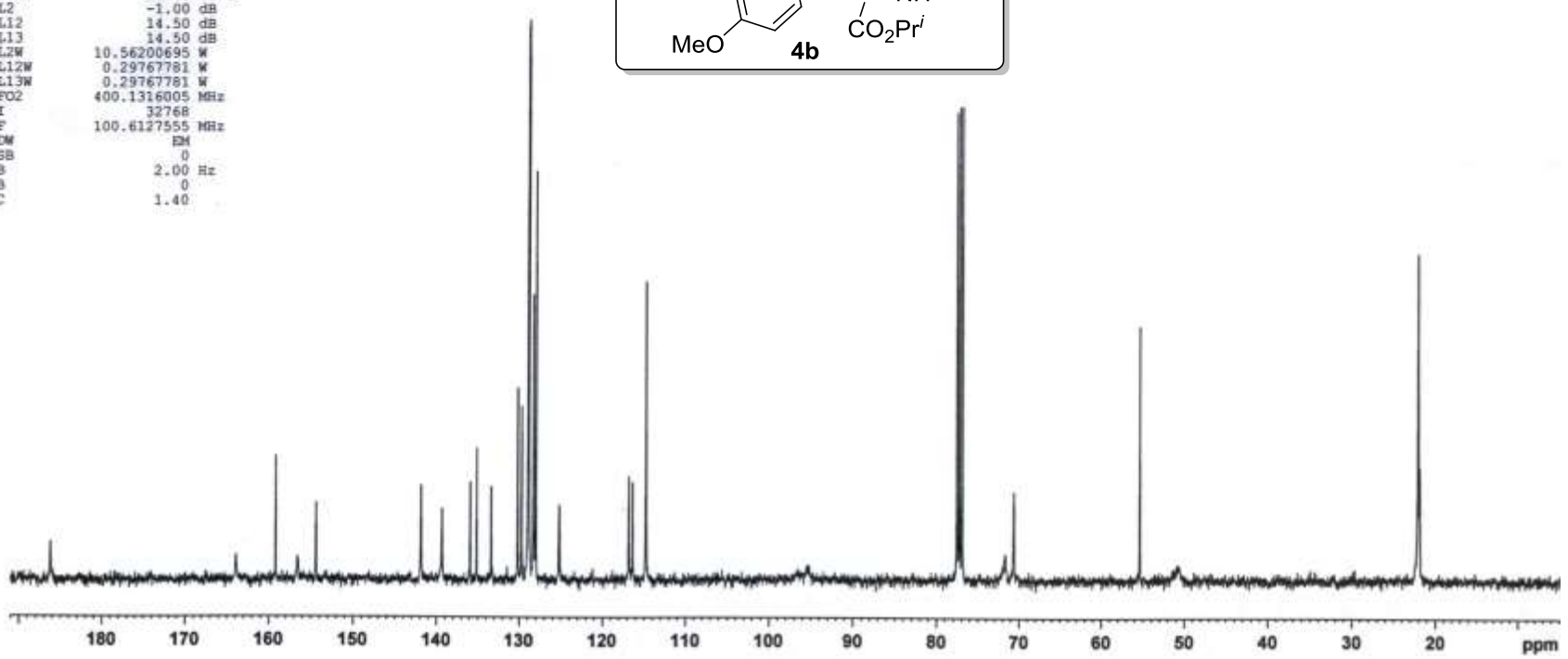
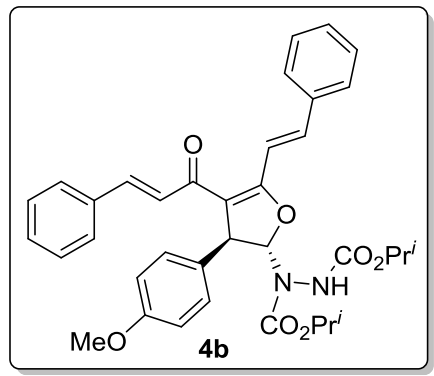


Fig S4. ¹³C NMR Spectrum of 4b

NAME INN-AN-4-341-H1
 EXPNO 20
 PROCNO 1
 Date_ 20110709
 Time 16.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 40
 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 294.0 K
 DI 1.00000000 sec
 TD0 1

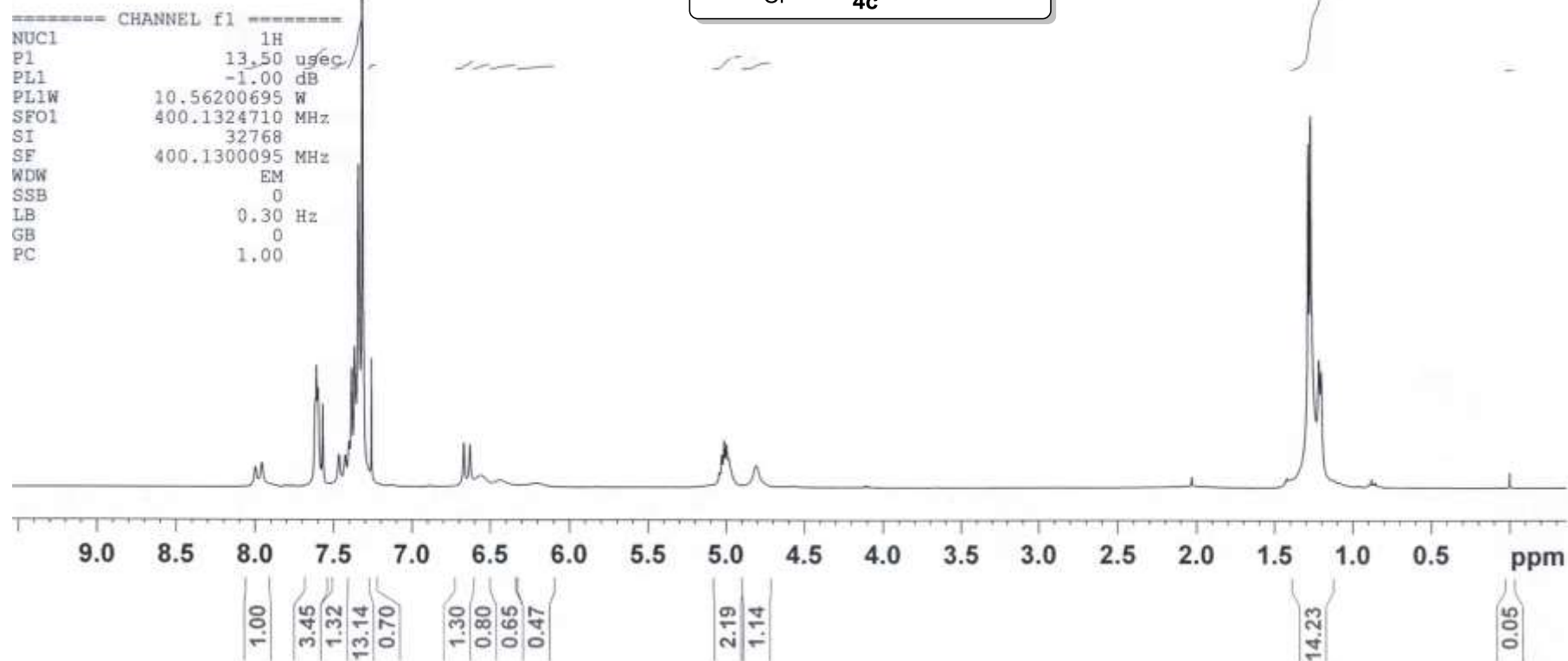
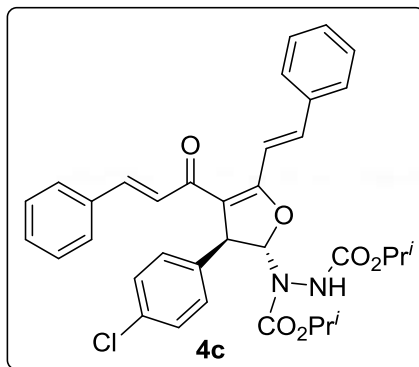


Fig S5. ¹H NMR Spectrum of 4c

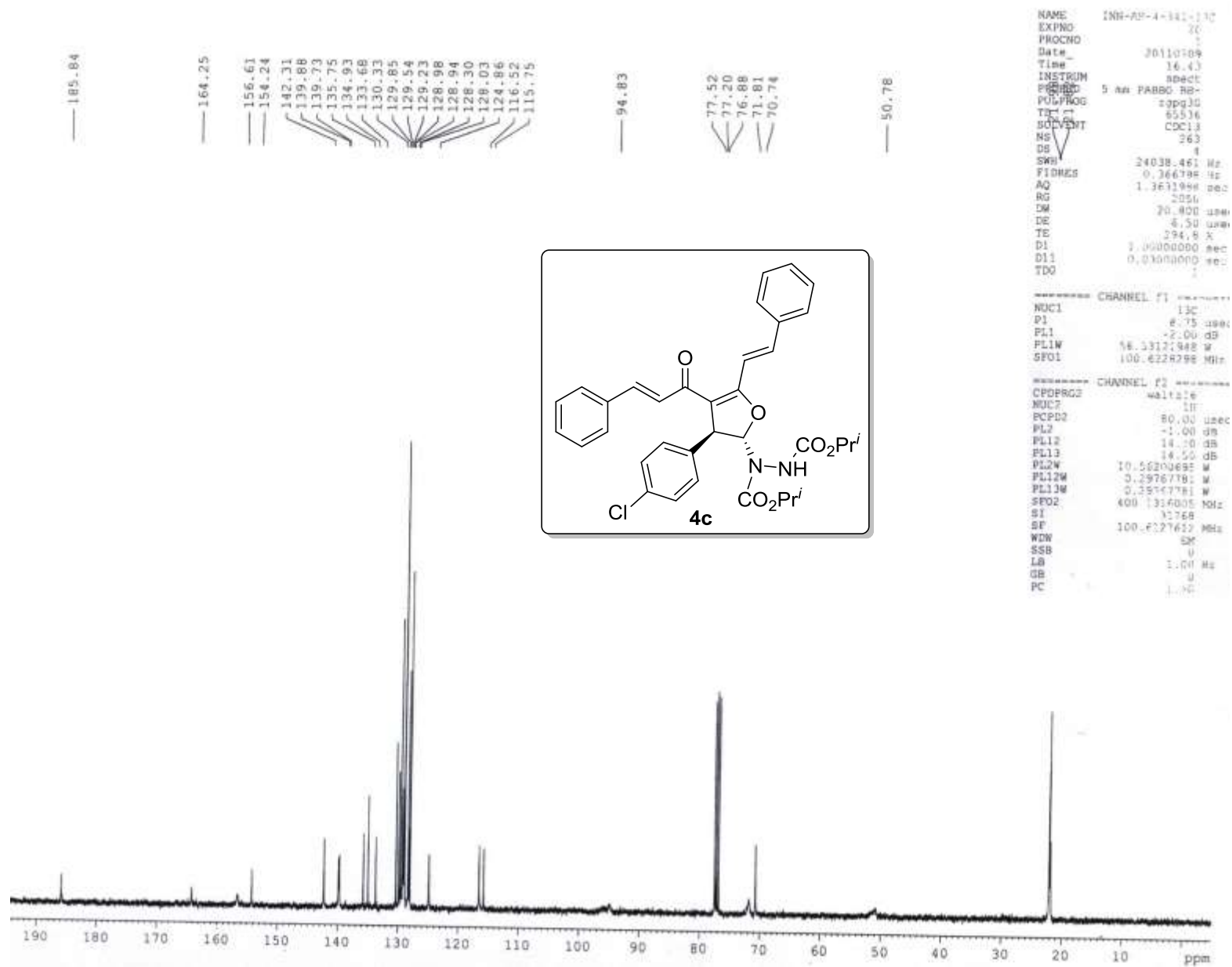


Fig S6. ¹³C NMR Spectrum of 4c

INN-AN-4-341-2-H1-COSEY

```

NAME      INN-AN-4-341-2-H1-COSEY
EXPNO    70
PROCNO   1
Date_    20110709
Time     20.00
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  cosygpgqf
TD       1024
SOLVENT  CDCl3
NS       16
DS       16
SWH      3662.109 Hz
FIDRES   3.576279 Hz
AQ       0.1398601 sec
RG       203
DW       136.533 usec
DE       6.50 usec
TE       294.0 K
DC       0.0000300 sec
D1       1.0000000 sec
D13      0.0000400 sec
D16      0.0002000 sec
IN0      0.00027305 sec
  
```

```

===== CHANNEL f1 =====
NUC1     1H
PQ       13.50 usec
P1       13.50 usec
PL1      -1.00 dB
PL1W     10.56200695 W
SFO1     400.1316171 MHz
  
```

```

===== GRADIENT CHANNEL =====
GPNAM1   SINE.100
GP21     10.00 %
P16      1000.00 usec
ND0      1
TD        256
SFO1     400.1316 MHz
FIDRES   14.305174 Hz
SW        9.152 ppm
InMODE   QF
SI        2048
SF       400.1300095 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.40
ST        512
MC2      QF
SF       400.1300095 MHz
WDW       SINE
SSB       0
LB        0.00 Hz
GB        0
  
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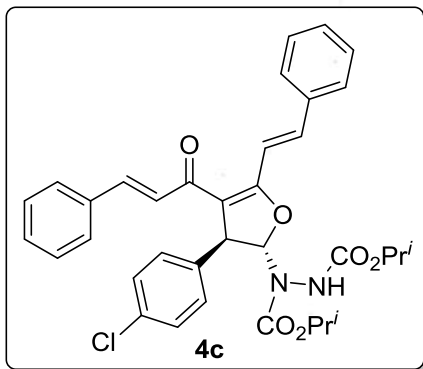
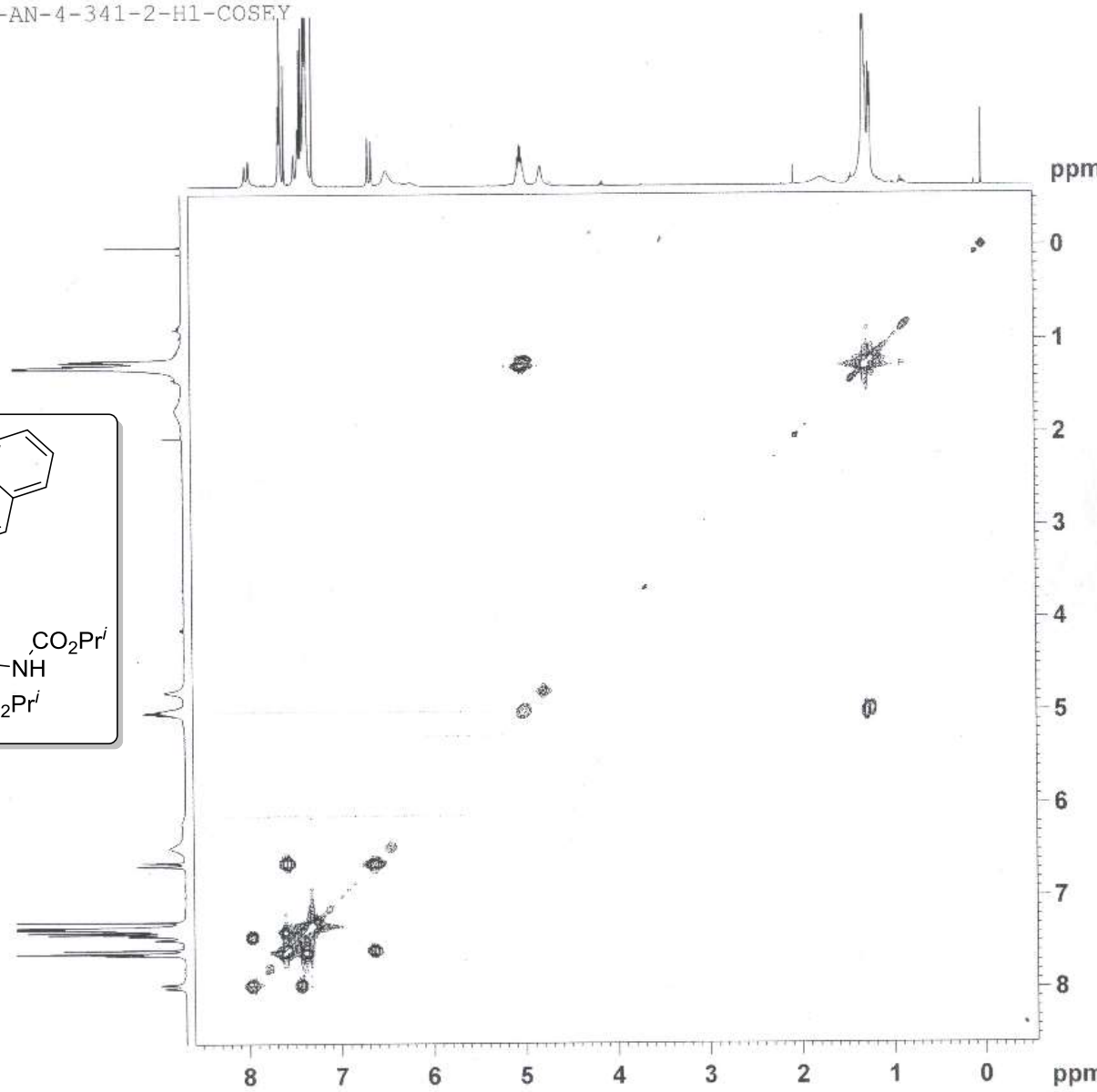
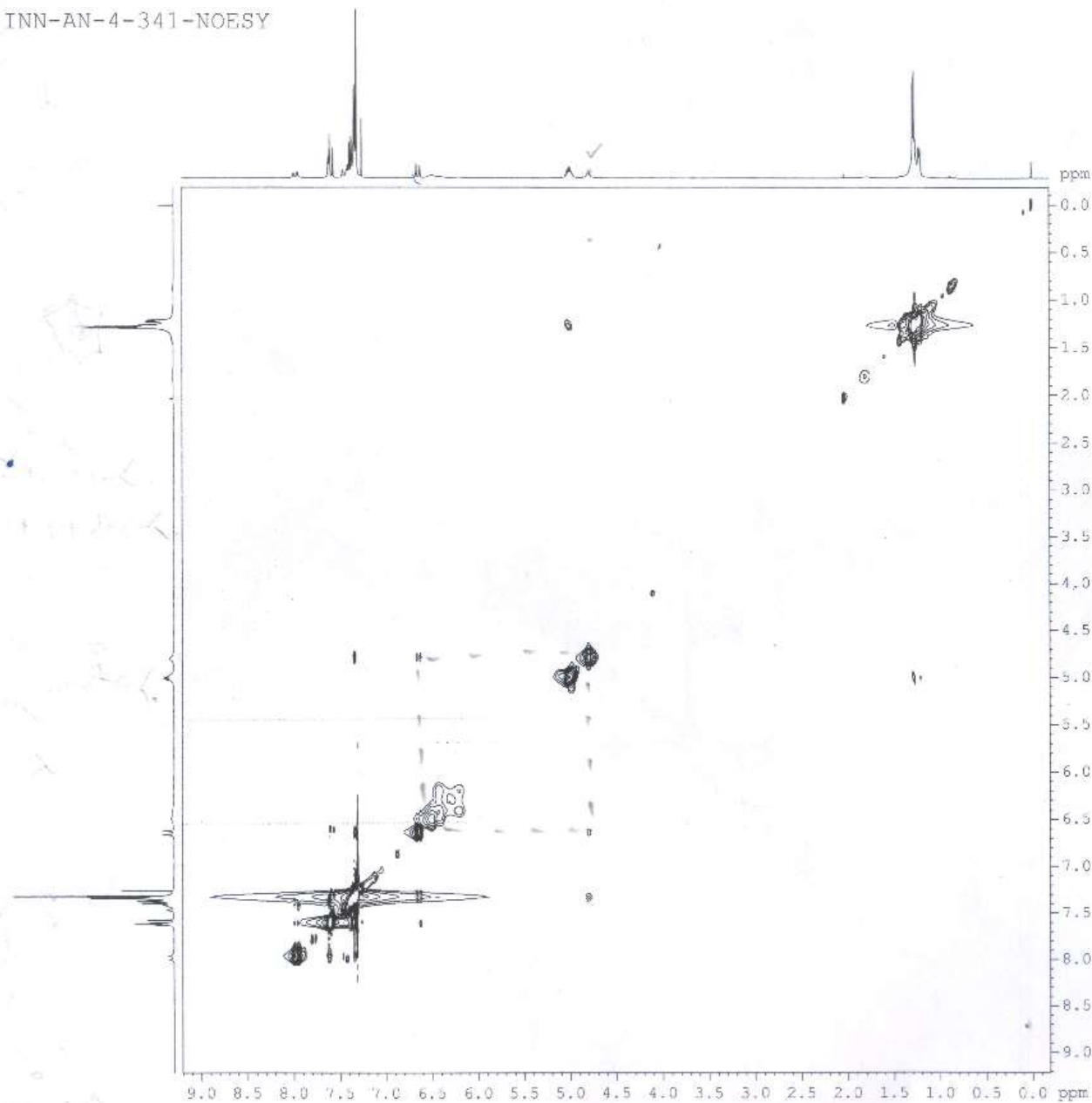


Fig S7. ¹H-¹H COSY NMR Spectrum of 4c

INN-AN-4-341-NOESY



```

NAME      INN-AN-4-341-10
EXPNO    72
PROCNO    1
Date_     20110713
Time      0.35
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   noesyph
TD        3274
SOLVENT   CDCl3
NS        32
DS        16
SWH       3759.399 Hz
FIDRES    1.835644 Hz
AQ        0.2724340 sec
RG        2.56
TM        133.000 usec
DE        6.50 usec
TE        300.0 K
DQ        0.0001075 sec
DI        1.90374398 sec
DB        0.30000001 sec
LNU       0.00026585 sec
    
```

```

----- CHANNEL f1 -----
NUC1      1H
DE        13.50 usec
PULP      -1.00 dB
PL1W      10.56300645 W
SFO1      400.1318128 MHz
NUC2      1
ID        756
SFO2      400.1318 MHz
FIDRES    14.692340 Hz
SW        9.700 ppm
PRMODE    States-T2P1
SI        2048
SF        400.1300095 MHz
WDW       QSHINE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00
ST        512
MC2       States-T2P1
SF        400.1300095 MHz
WDW       QSHINE
SSB       2
LN        0.00 Hz
GB        0
    
```

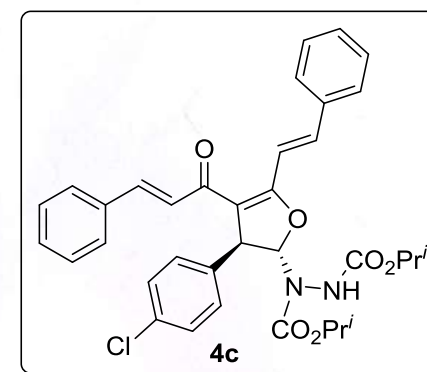


Fig S8. ¹H-¹H NOESY NMR Spectrum of **4c**

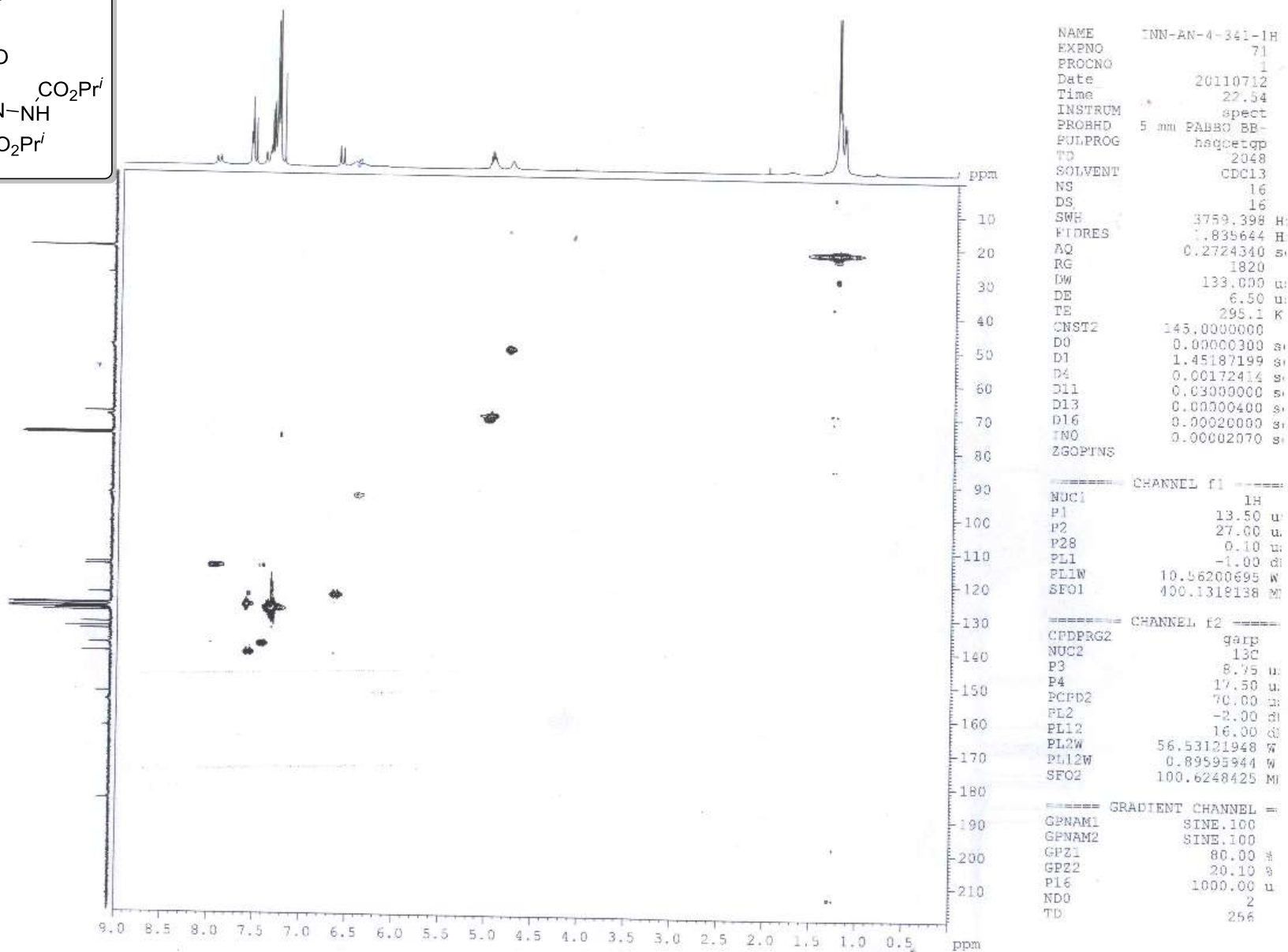
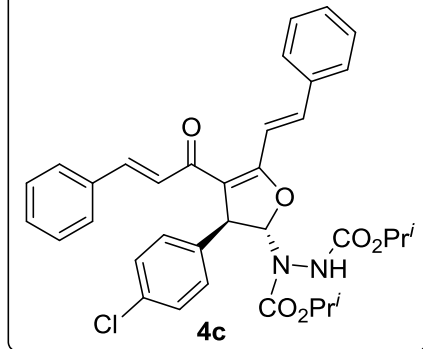


Fig S9. ^1H - ^{13}C HSQC NMR Spectrum of **4c**

NAME INN-AN-4-338-1H
 EXPNO 2
 PROCNO 1
 Date_ 20111112
 Time_ 15.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 10
 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 292.5 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

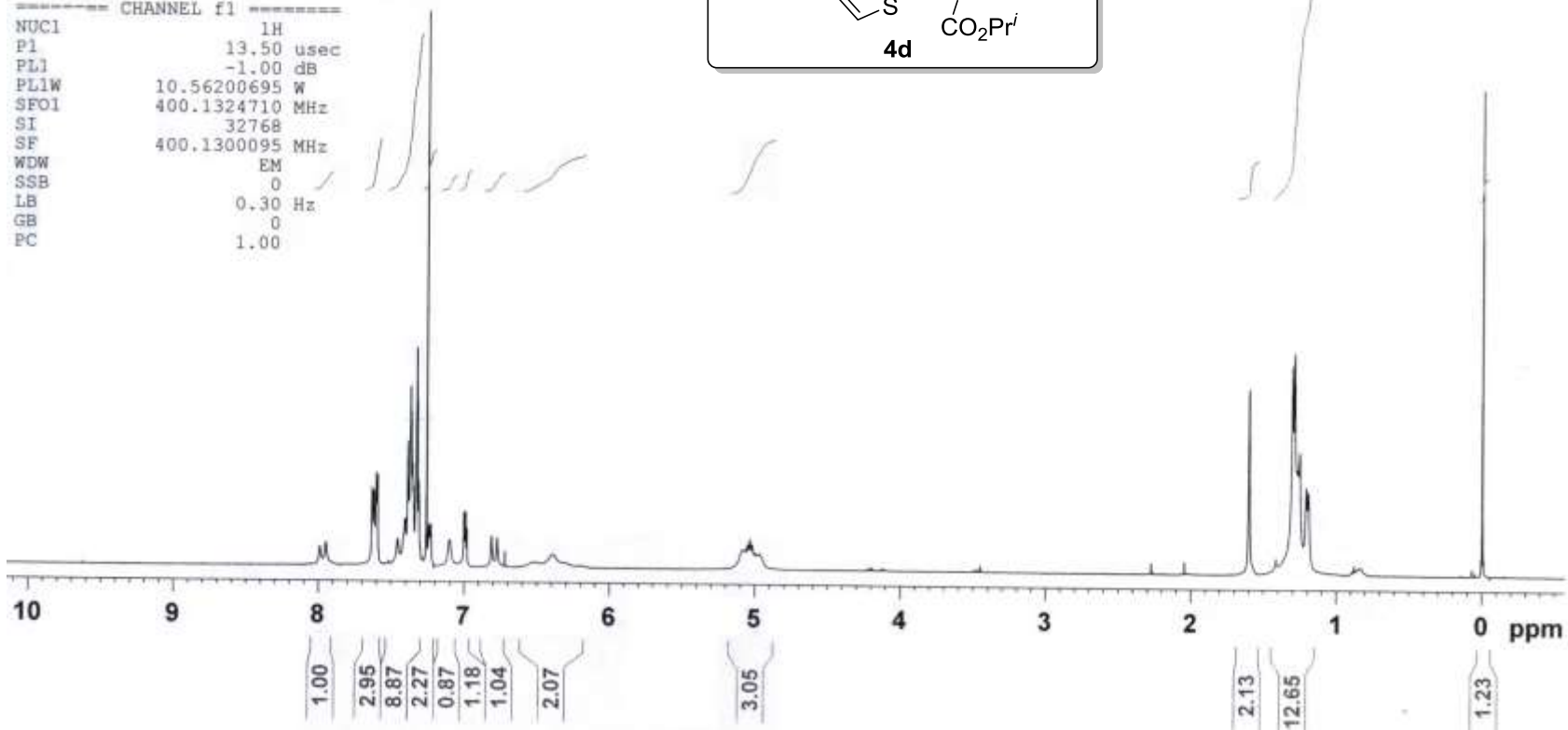
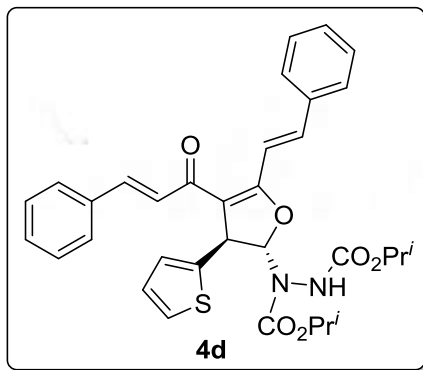


Fig S10. ¹H NMR Spectrum of 4d

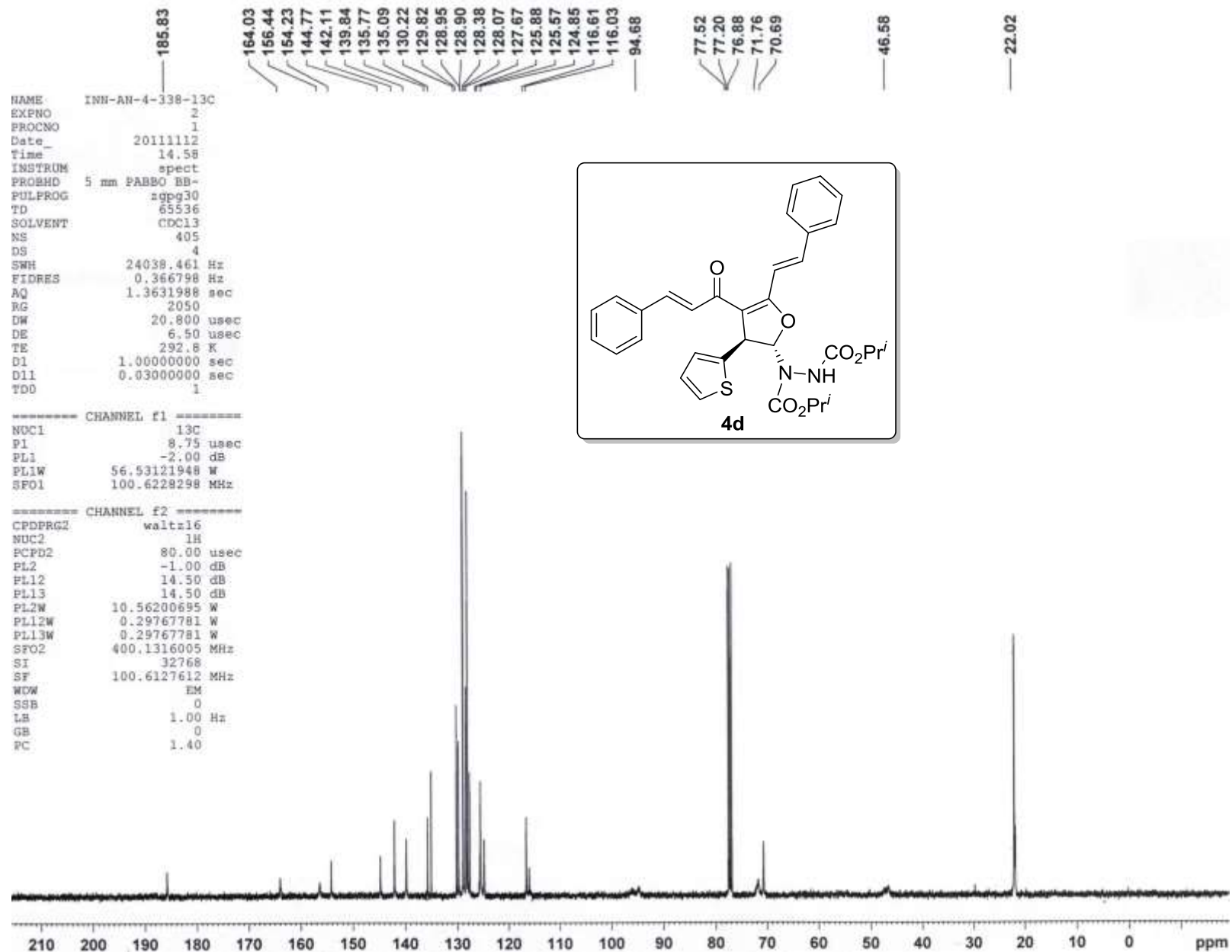


Fig S11. ¹³C NMR Spectrum of 4d

NAME INN-AN-4-422-1H
 EXPNO 3
 PROCNO 1
 Date_ 20120602
 Time_ 15.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 6
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 292.8 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

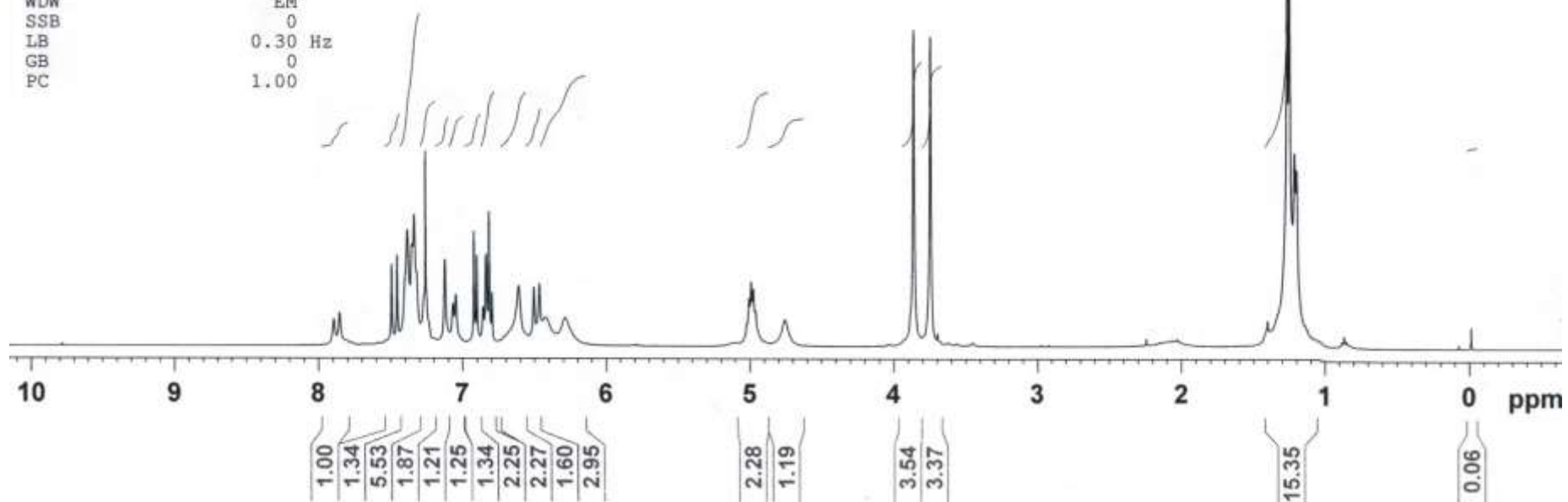
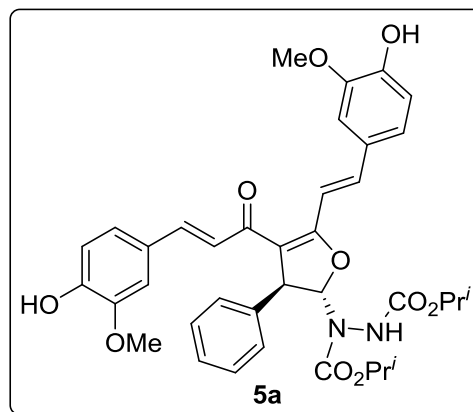


Fig S12. ¹H NMR Spectrum of 5a

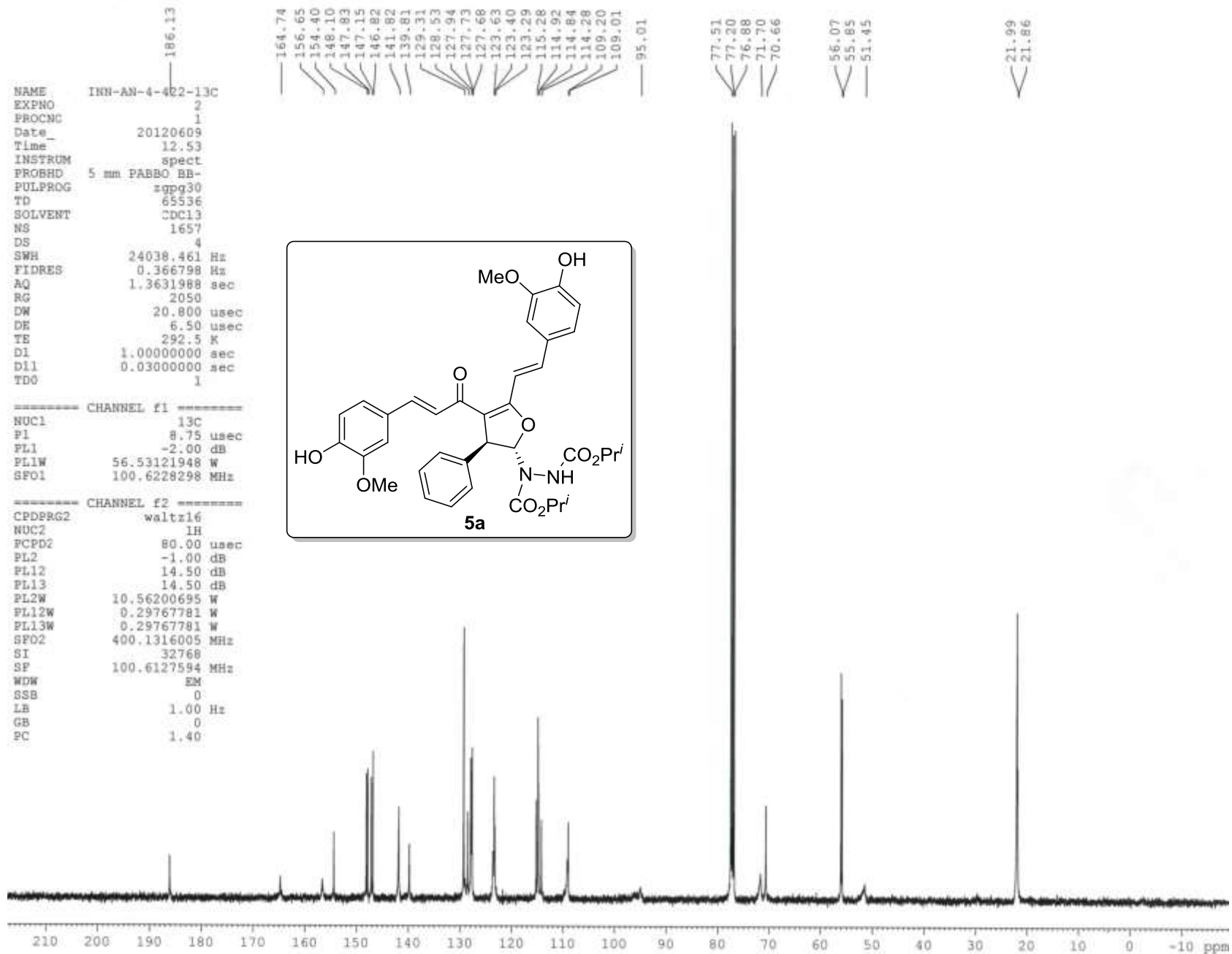


Fig S13. ¹³C NMR Spectrum of 5a

NAME INN-AN-4-423-1H
 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
 TDO 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

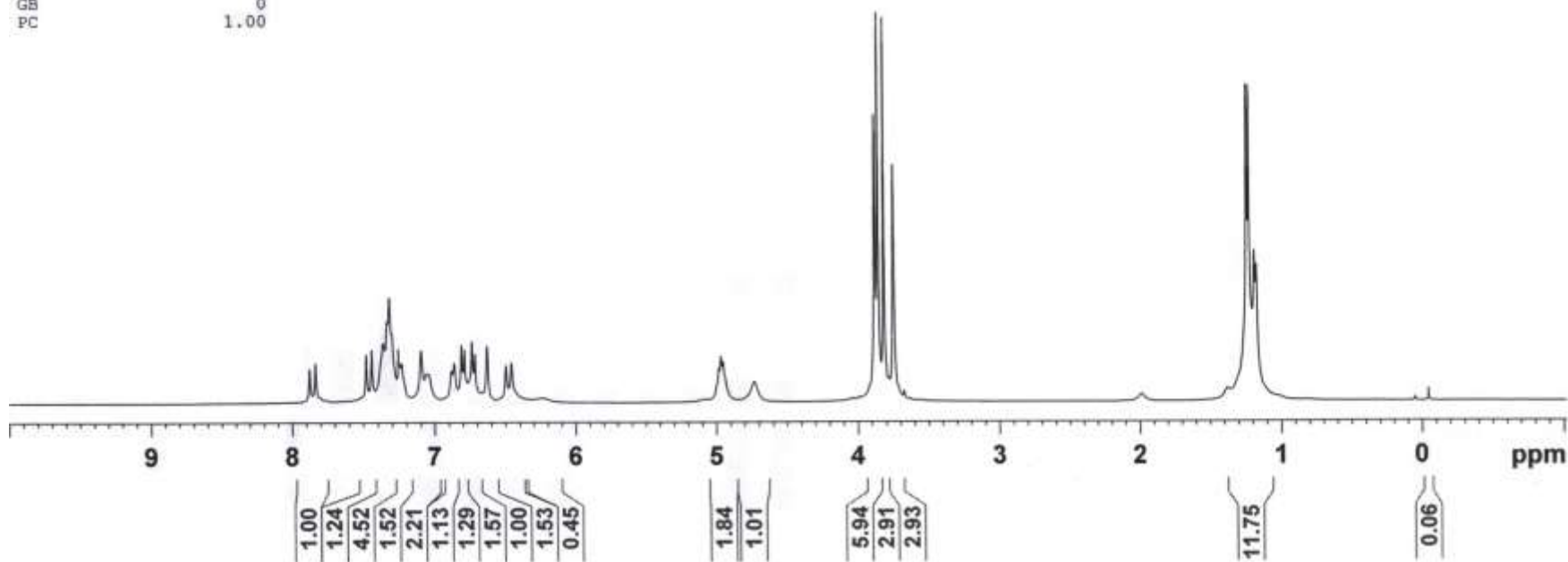
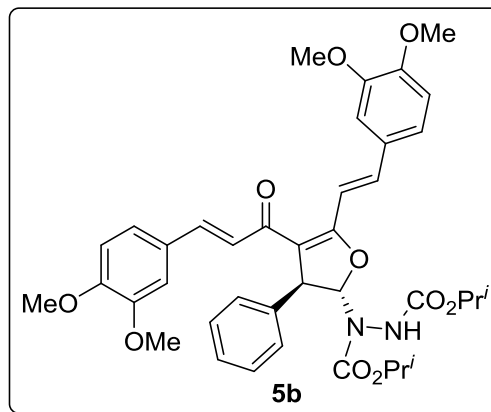


Fig S14. ¹H NMR Spectrum of 5b

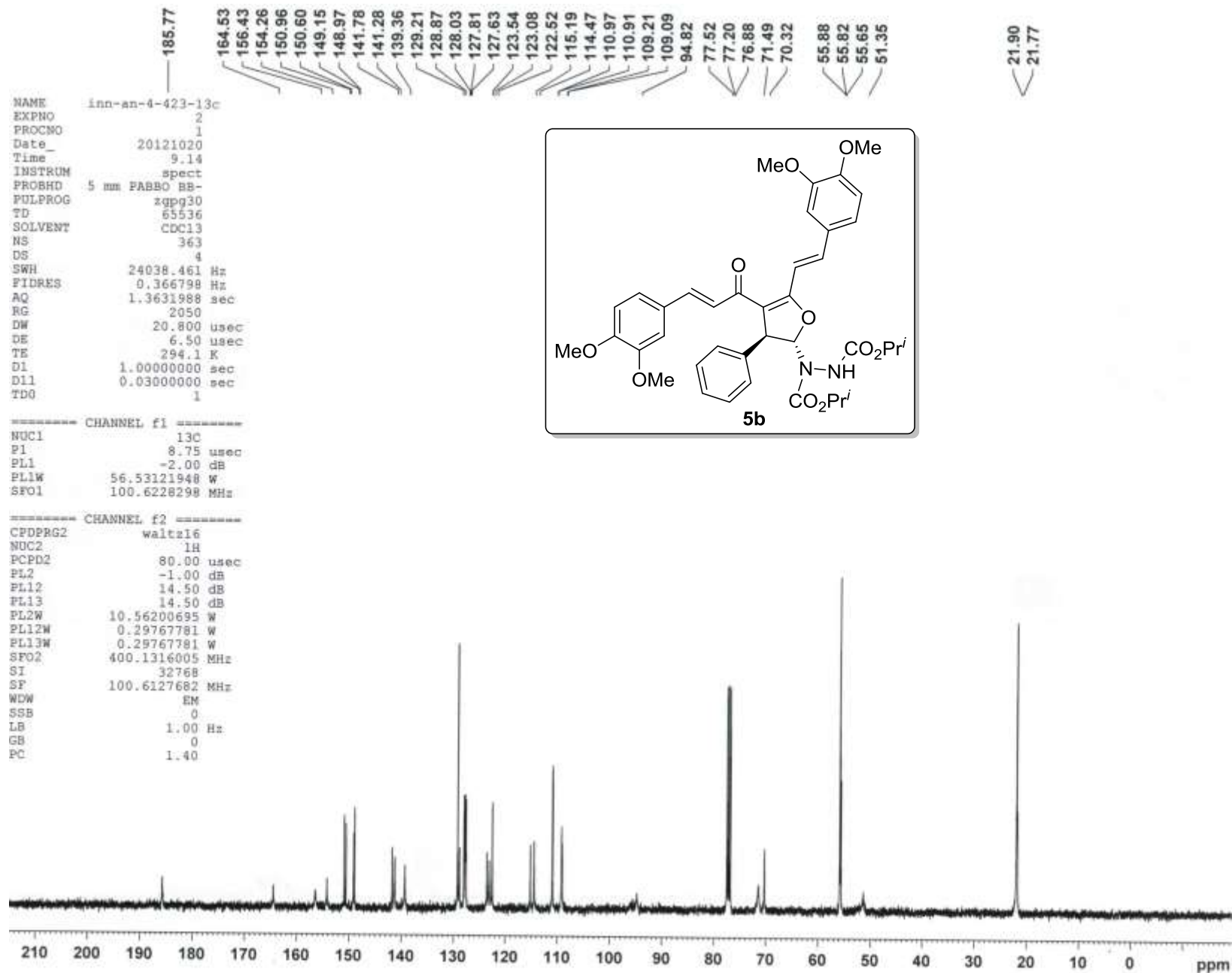


Fig S15. ¹³C NMR Spectrum of 5b

NAME INN-AN-5-008-1H
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 PROCNO 1
 Date_ 20121129
 Time_ 20.22
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 11
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 293.7 K
 DI 1.0000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 13.50 usec
 PL1 -1.00 dB
 PL1W 10.56200695 W
 SF01 400.1324710 MHz
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

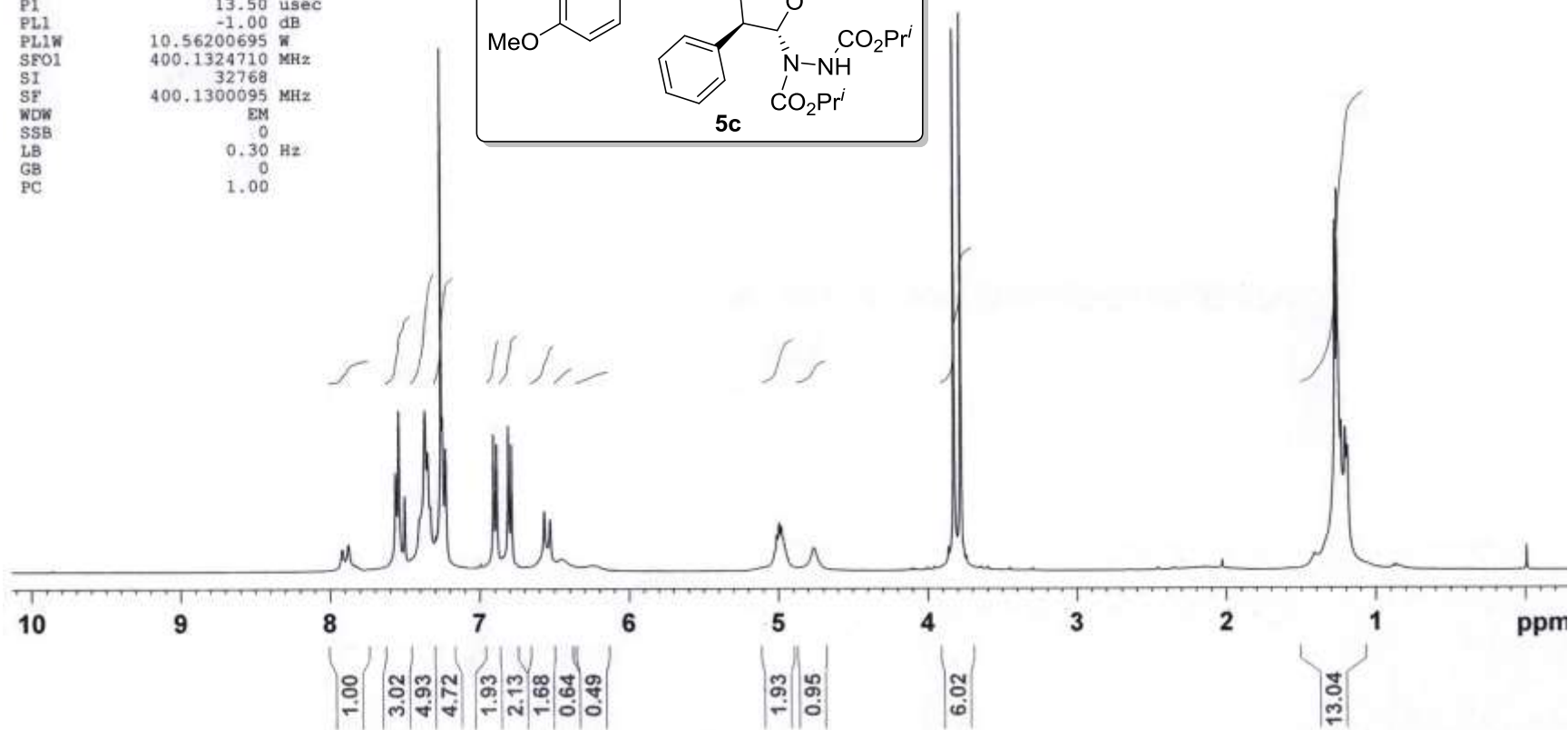
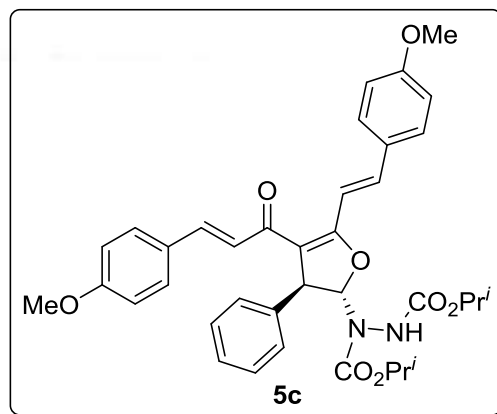


Fig S16. ¹H NMR Spectrum of 5c

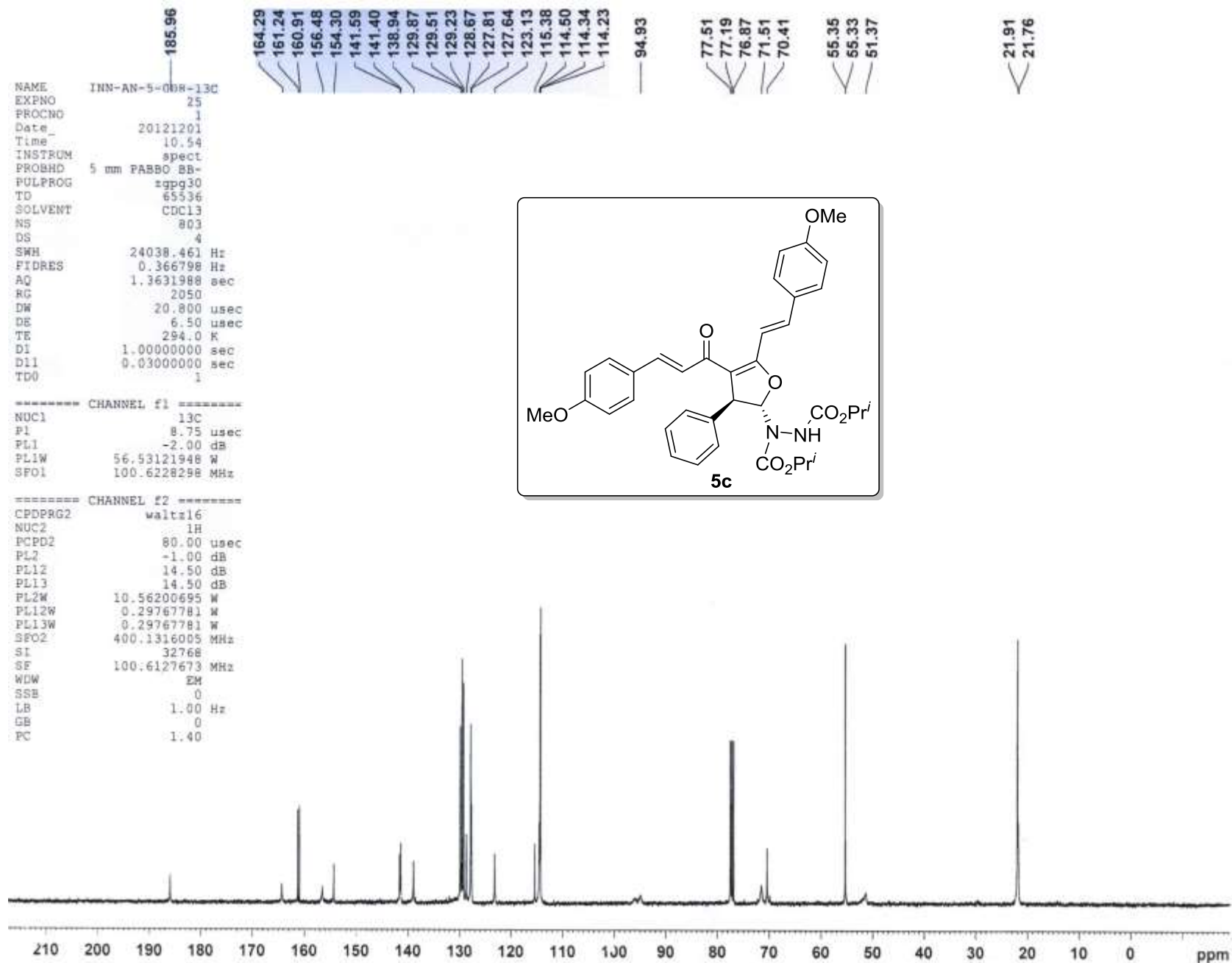
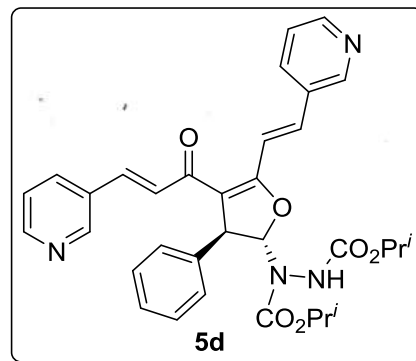


Fig S17. ¹³C NMR Spectrum of 5c

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 EXPNO 5
 PROCNO 1
 Date_ 20121202
 Time_ 20.50
 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 16
 DW 60.800 usec
 DE 6.50 usec
 TE 293.1 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

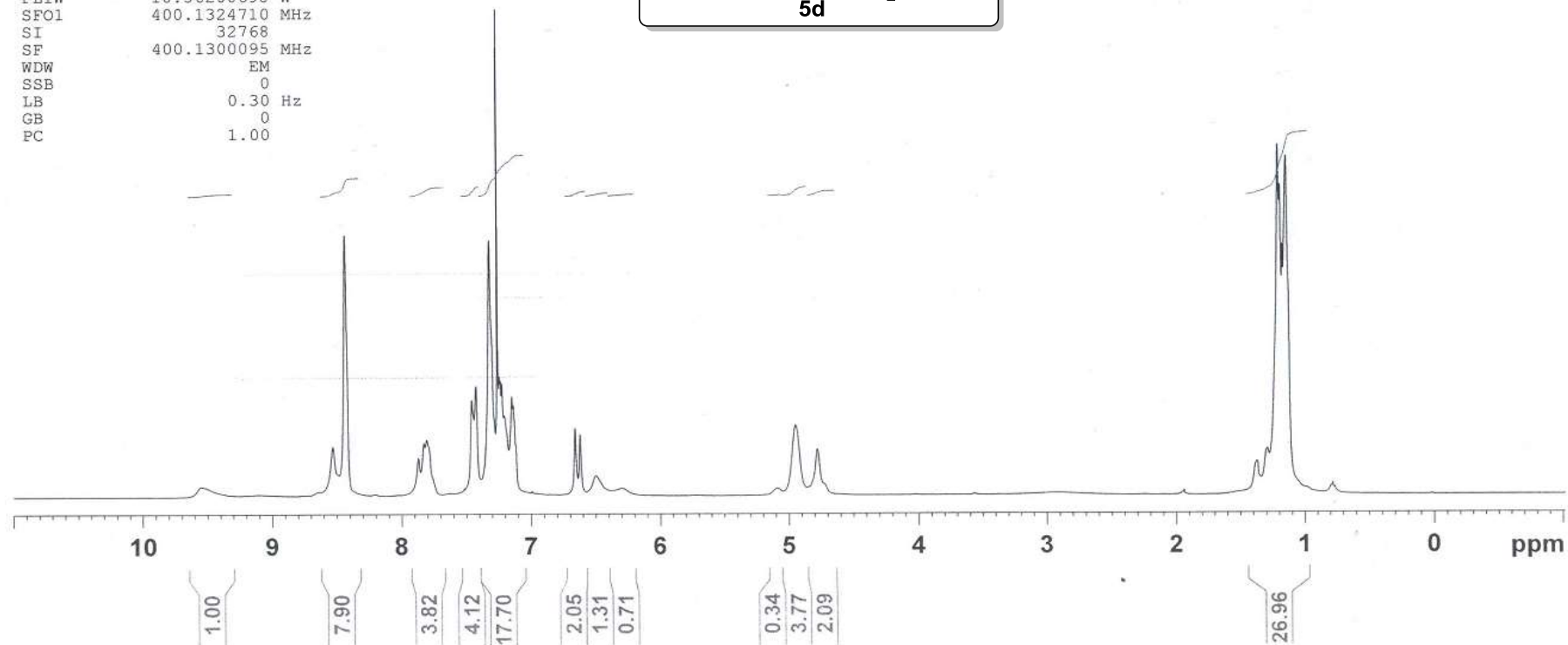


Fig S18. ¹H NMR Spectrum of 5d

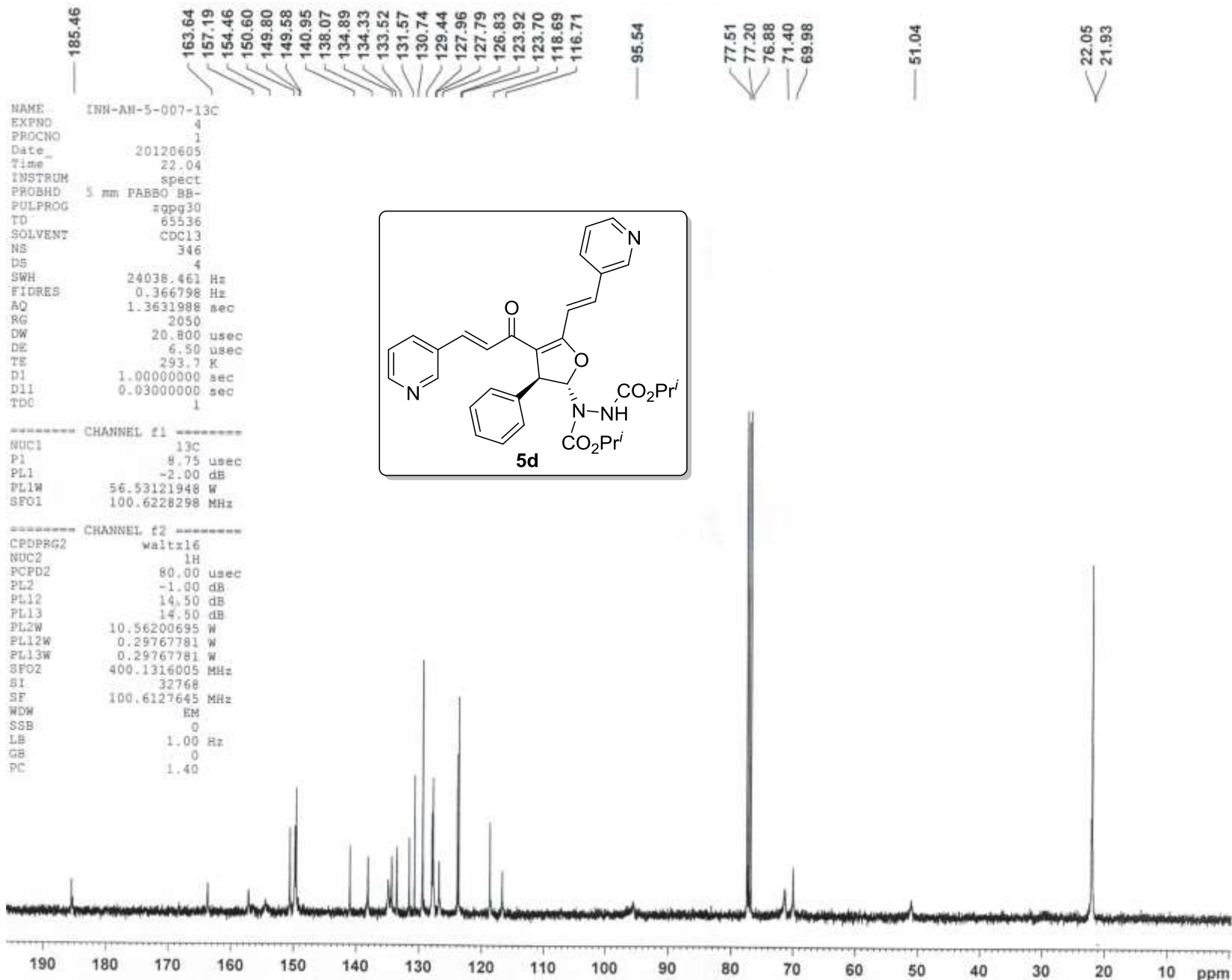


Fig S19. ^{13}C NMR Spectrum of 5d


```

NAME      INN-AN-4-424-13C
EXPNO     8
PROCNO    1
Date_     20121206
Time      20.27
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   COCl3
NS         111
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         294.6 K
D1         1.00000000 sec
D11        0.03000000 sec
TDO        1

----- CHANNEL f1 -----
NUC1      13C
P1         6.75 usec
PL1        -2.00 dB
PL1W       56.53121948 W
SFO1      100.6228298 MHz

----- CHANNEL f2 -----
CFDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       14.50 dB
PL13       14.50 dB
PL2W       10.56200695 W
PL12W      0.29767781 W
PL13W      0.29767781 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127635 MHz
WDW        EM
SSR         0
LB         1.00 Hz
GB         0
PC         1.40

```

185.40
163.42
156.42
154.31
152.34
151.81
144.53
141.29
129.18
128.19
127.76
127.61
125.83
122.67
116.38
115.27
114.60
113.39
112.41

94.92

77.52
77.20
76.88
71.62
70.45

51.42

21.93
21.75

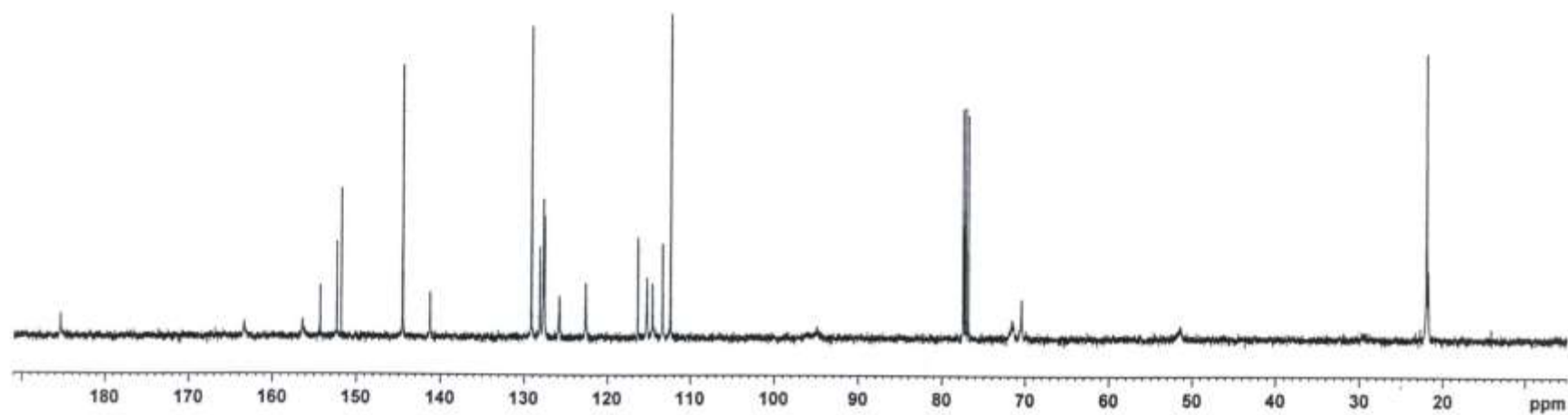
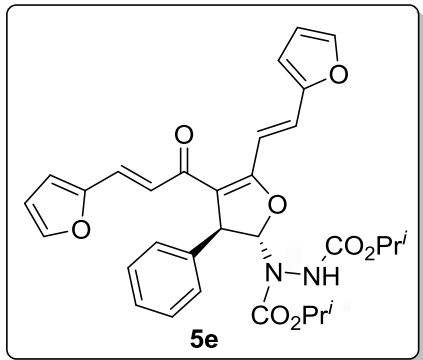


Fig S21. ¹³C NMR Spectrum of 5e

Current Data Parameters
NAME INN-4-KSB-173A
EKPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20141115
Time 11.18
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 13.43
DW 50.000 usec
DE 6.50 usec
TE 295.4 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 500.1300119 MHz
MDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

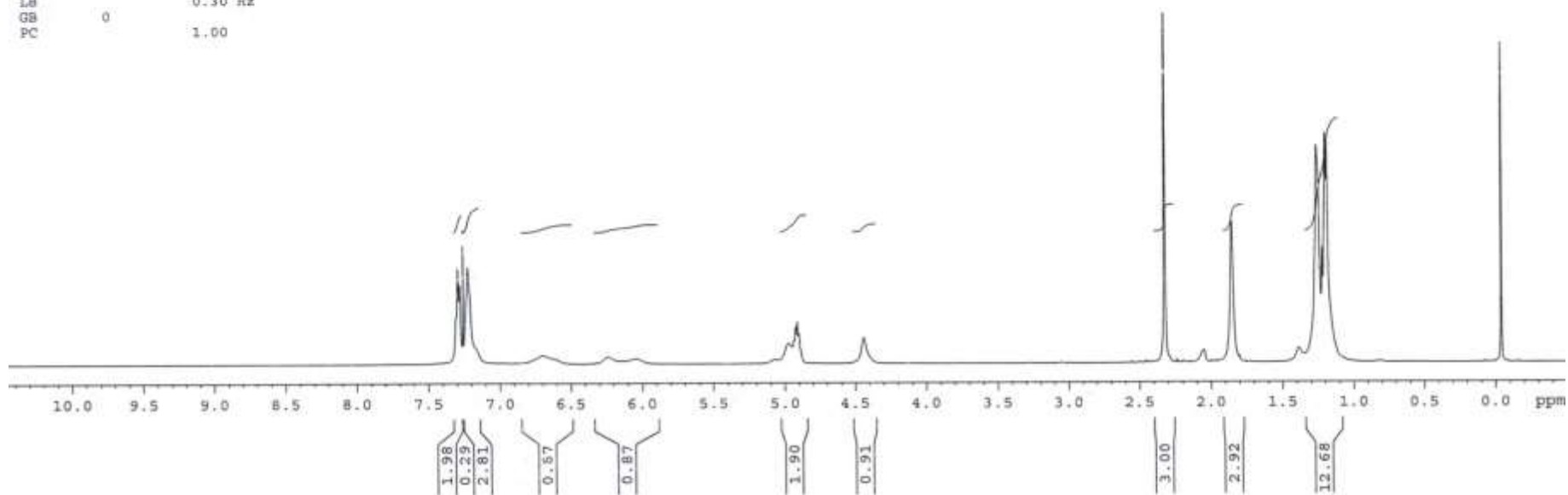
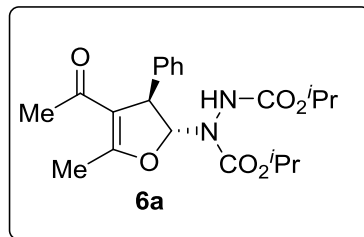


Fig S22. ¹H NMR Spectrum of 6a

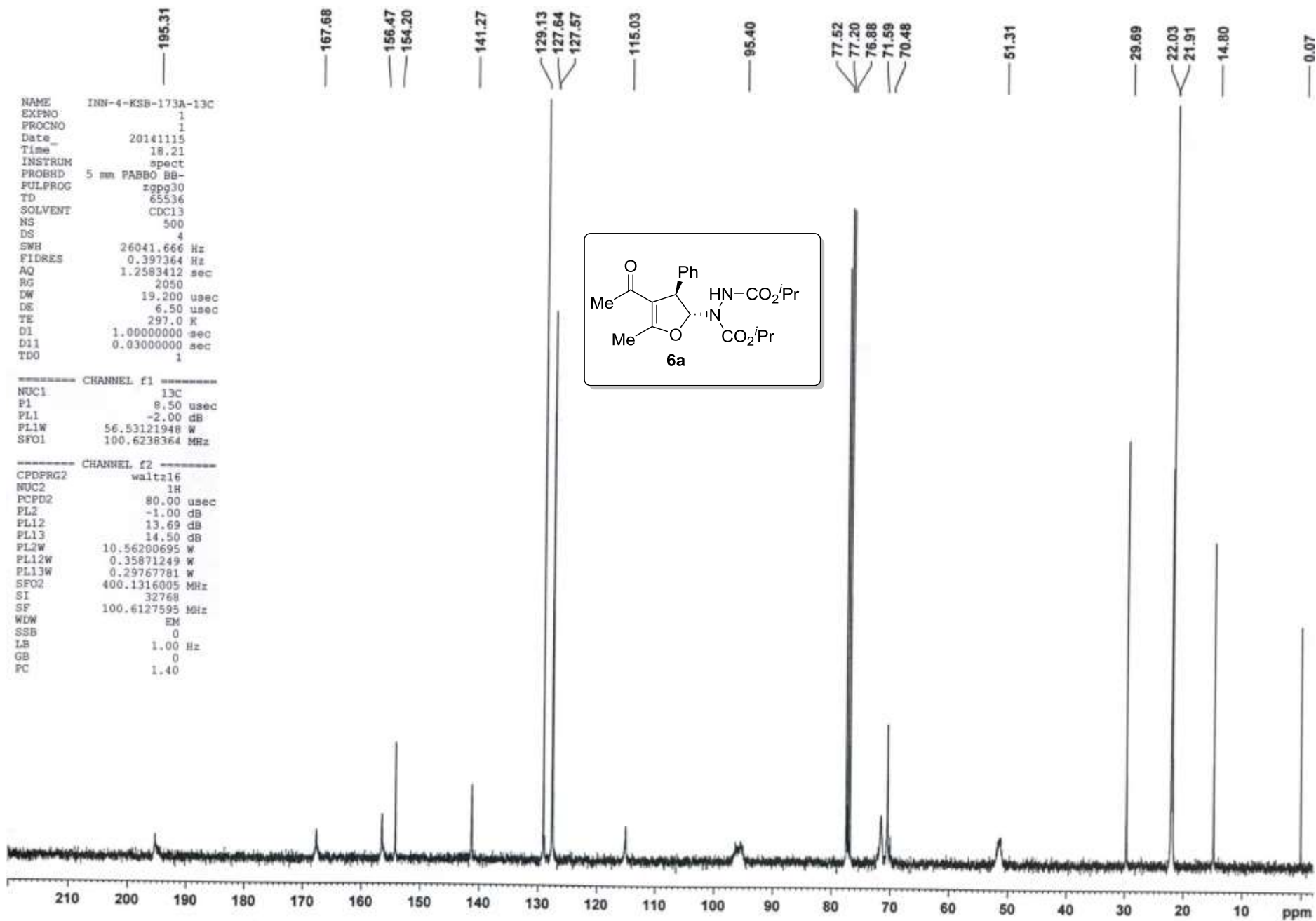


Fig S23. ¹³C NMR Spectrum of 6a

Current Data Parameters
NAME INN-4-KSB-173B-1H
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141115
Time 11.39
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 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.4 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 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

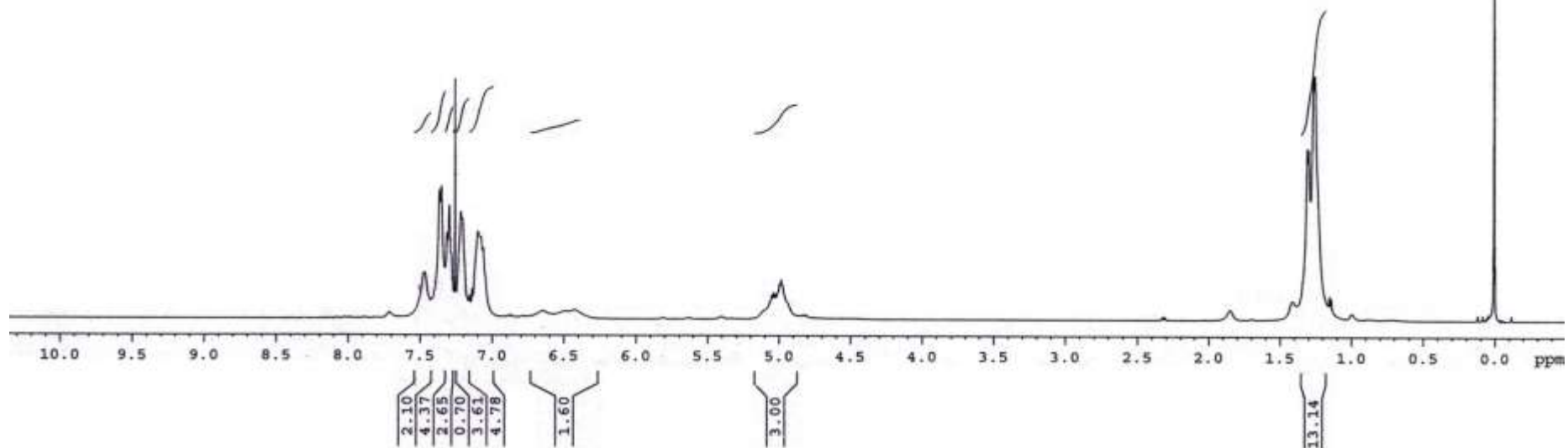
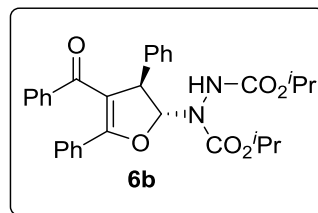


Fig S24. ¹H NMR Spectrum of 6b

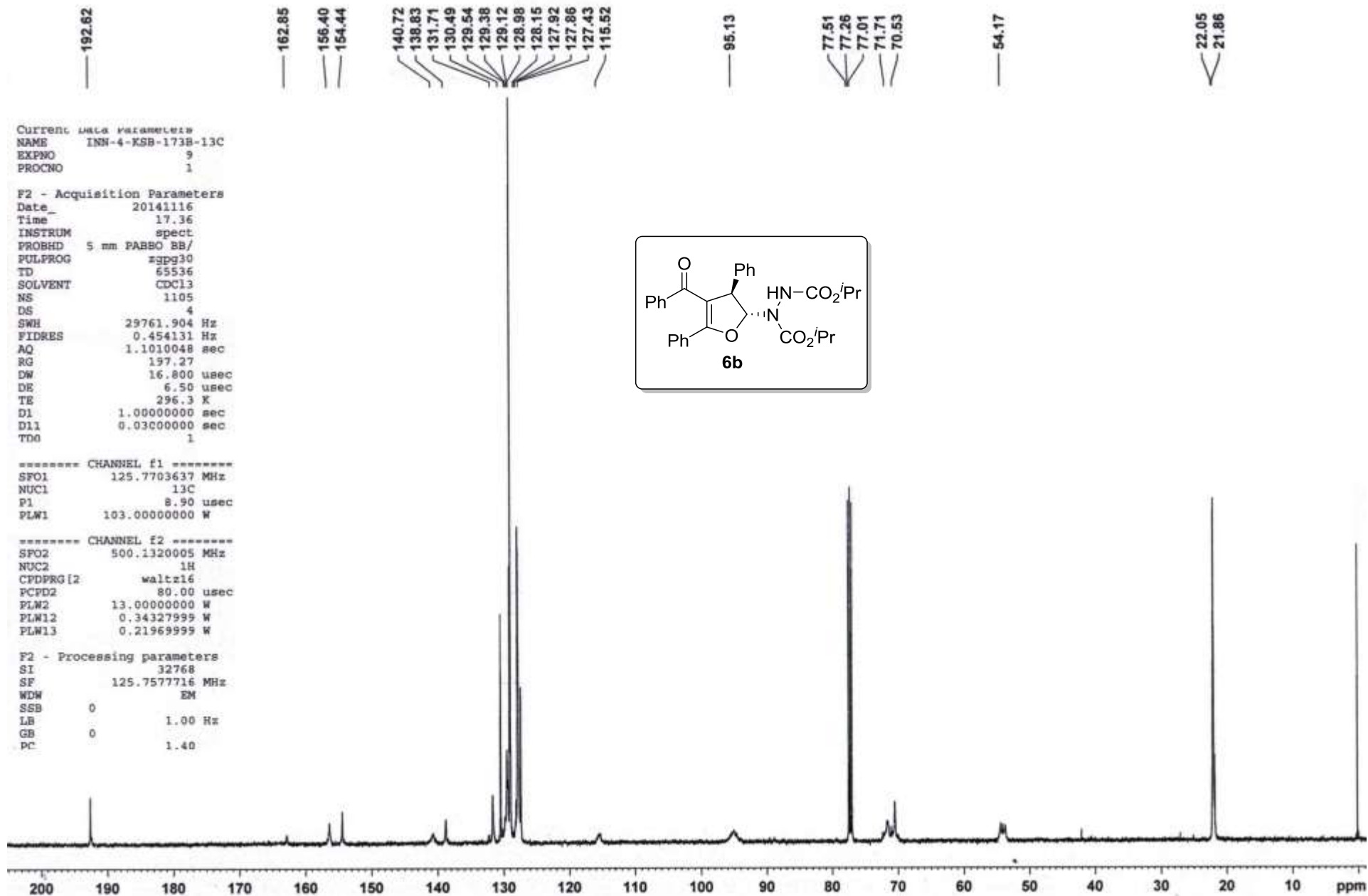


Fig S25. ¹³C NMR Spectrum of 6b

Current Data Parameters
NAME INN-4-RSB-173C-1H
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141115
Time 11.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SMH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.5 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 500.1300121 MHz
W/W EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

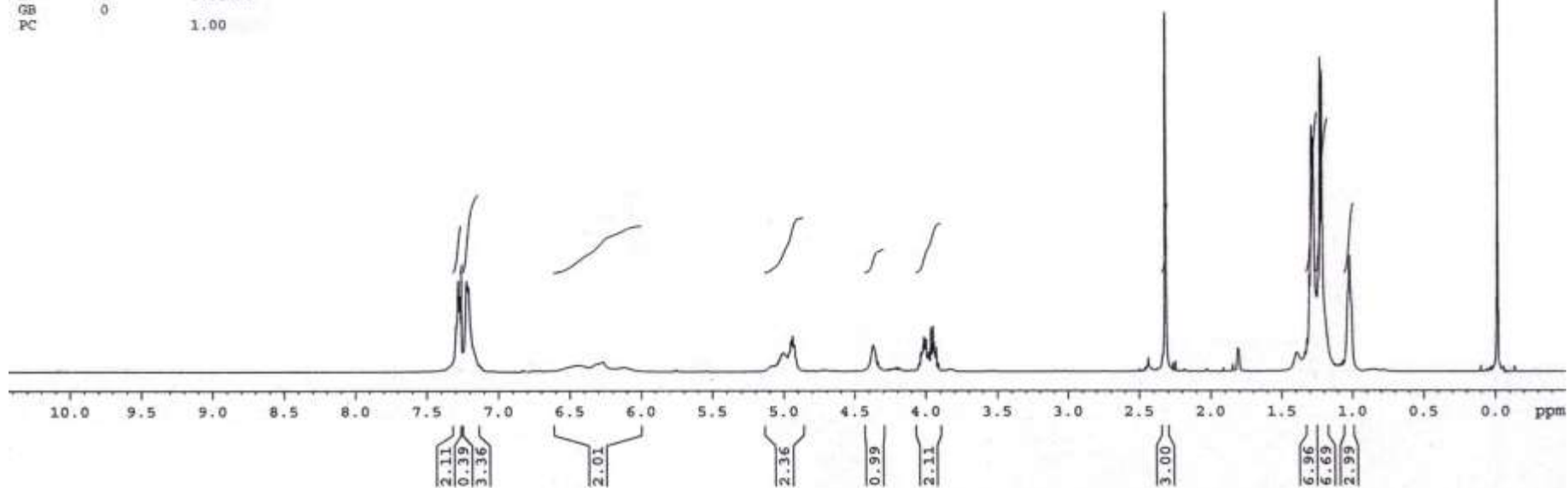
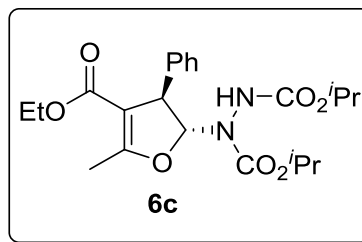


Fig S26. ¹H NMR Spectrum of 6c

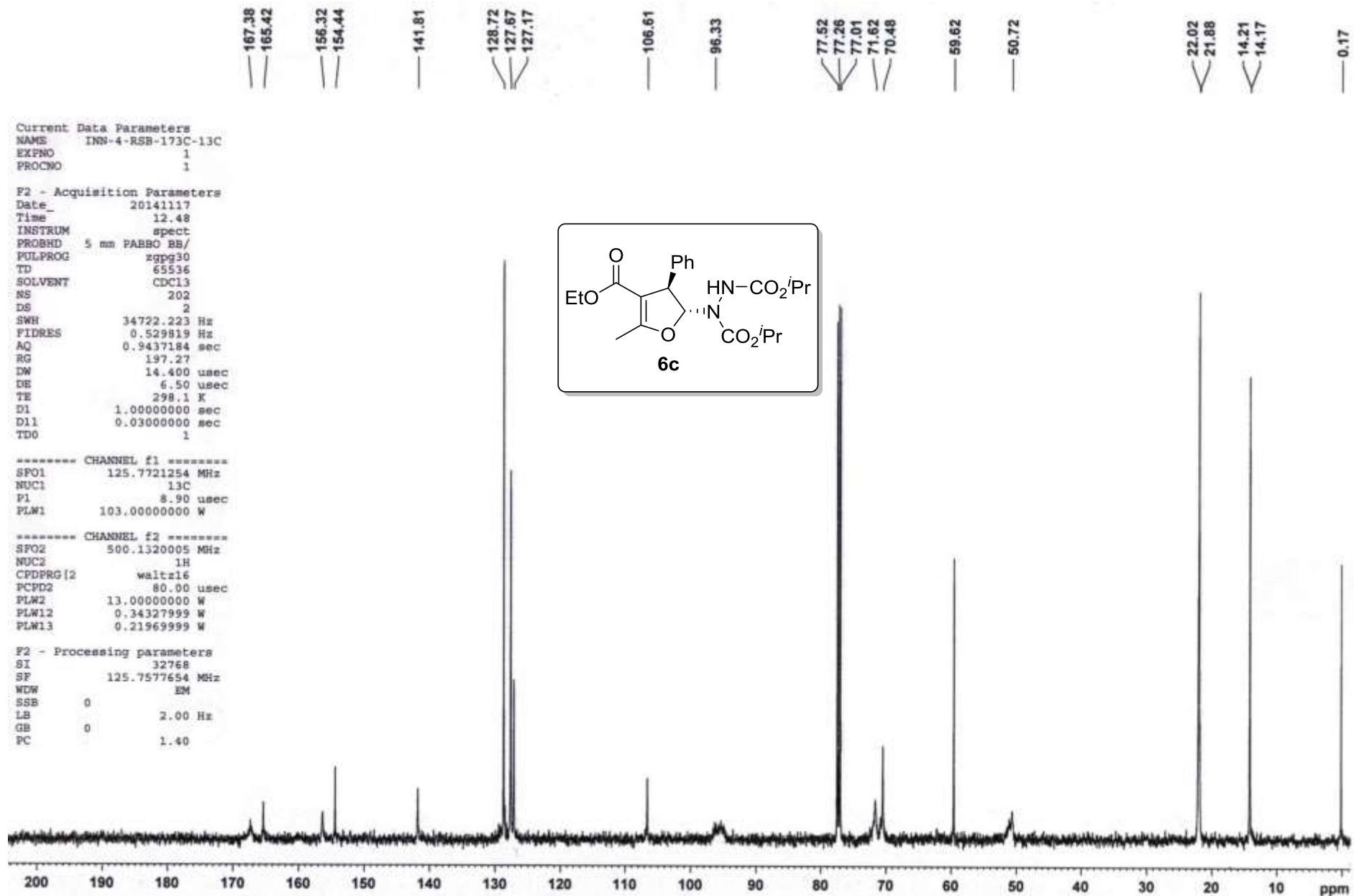


Fig S27. ¹³C NMR Spectrum of 6c

Current Data Parameters
NAME INN-4-KSB-174B-1H
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141115
Time 11.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SMH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.4 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 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

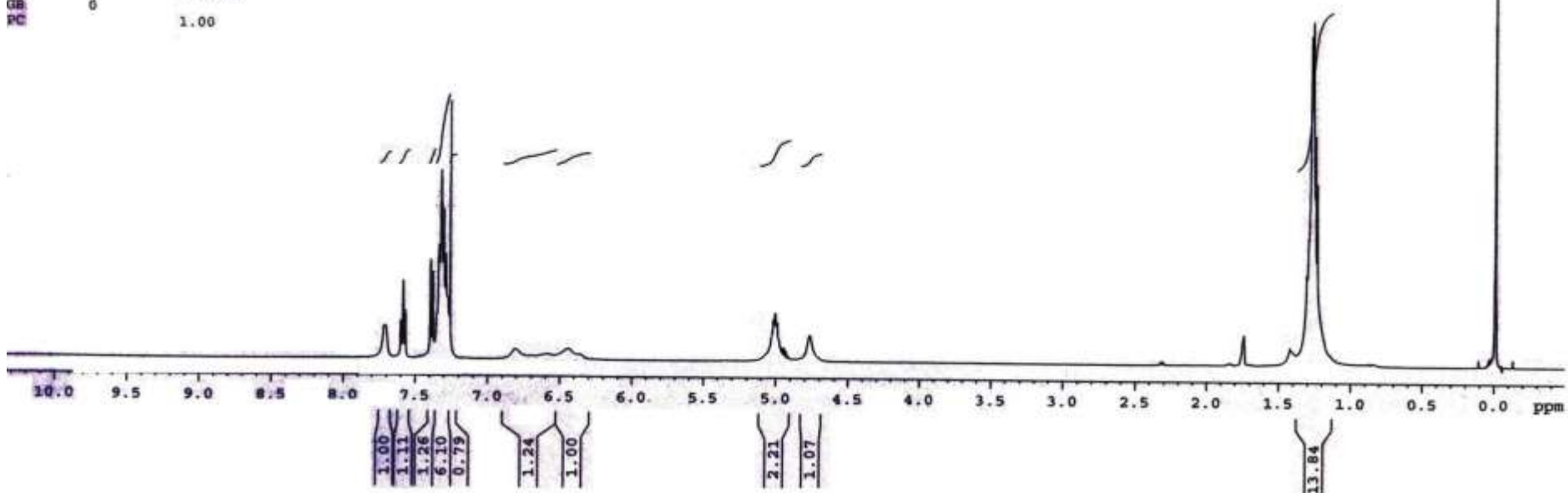
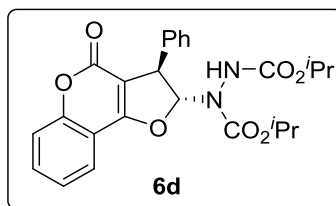


Fig S28. ¹H NMR Spectrum of 6d


```

NAME      INN-4-KSB-174B-13C
EXPNO    8
PROCNO   1
Date_    20141115
Time     21.21
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       3181
DS       4
SWH      26041.666 Hz
FIDRES   0.397364 Hz
AQ       1.2583412 sec
RG       2050
DM       19.200 usec
DE       6.50 usec
TE       297.2 K
D1       1.00000000 sec
D11      0.03000000 sec
TDO      1

```

```

----- CHANNEL f1 -----
NUC1     13C
P1       8.50 usec
PL1      -2.00 dB
PL1W     56.53121948 W
SFO1     100.6238364 MHz

```

```

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      -1.00 dB
PL12     13.69 dB
PL13     14.50 dB
PL2W     10.56200695 W
PL12W    0.35871249 W
PL13W    0.29767781 W
SFO2     400.1316005 MHz
S1       32768
SF       100.6127510 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40

```

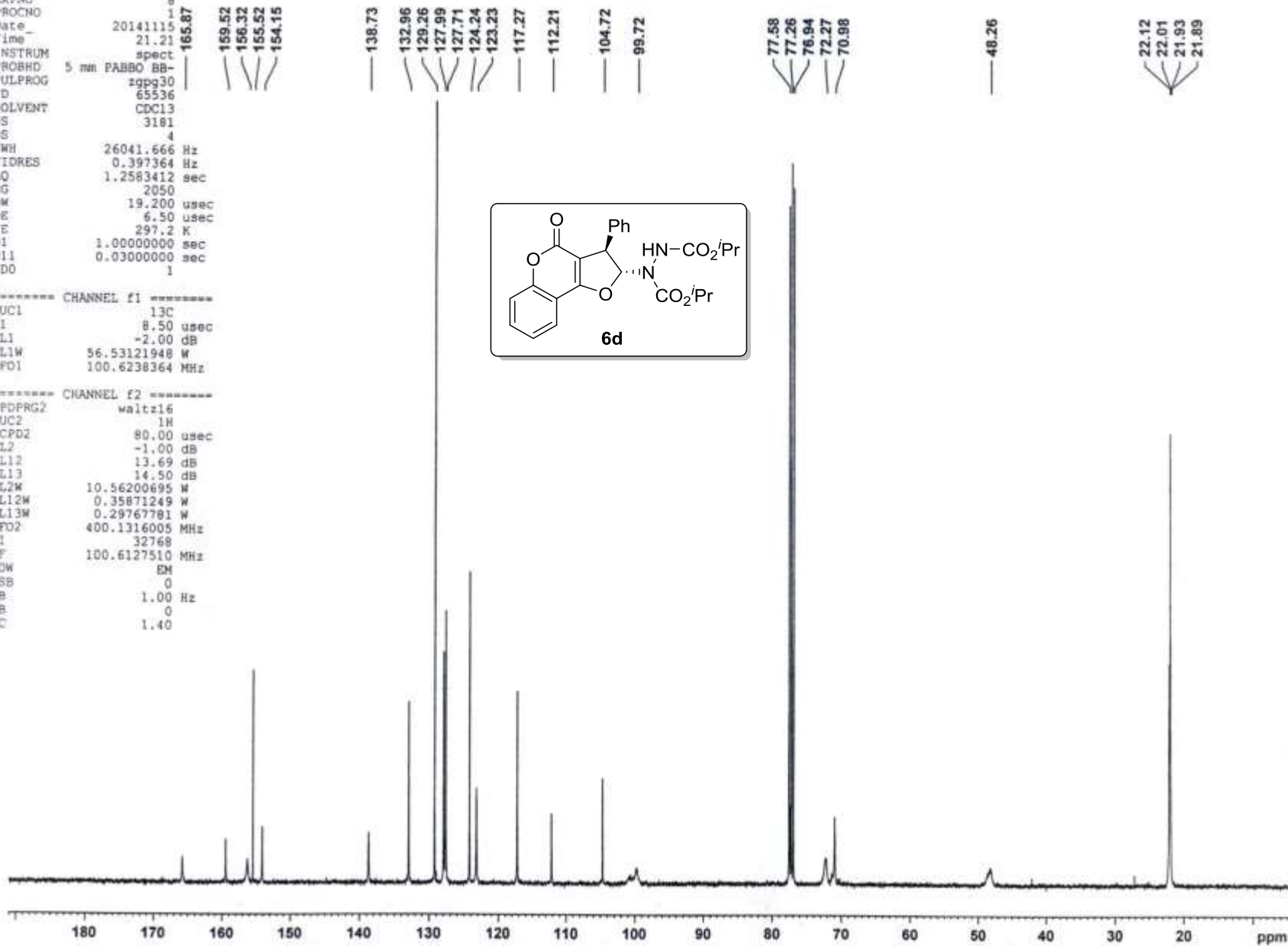


Fig S29. ^{13}C NMR Spectrum of **6d**

NAME INN-4-KSB-Ph-1H
 EXPNO 2
 PROCNO 1
 Date_ 20140724
 Time_ 18.10
 INSTRUM spect
 PROBHD 5 mm SEI 1H/D-
 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
 SF01 400.1324710 MHz
 SI 32768
 SF 400.1300103 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

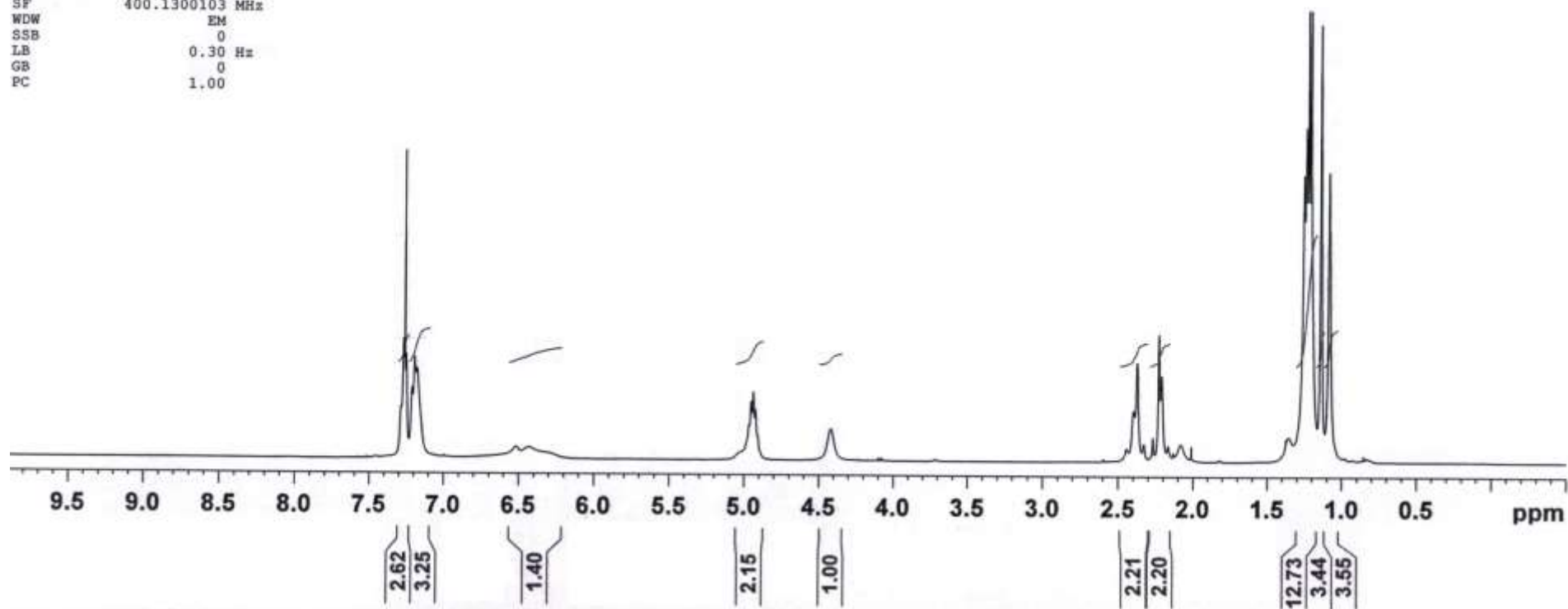
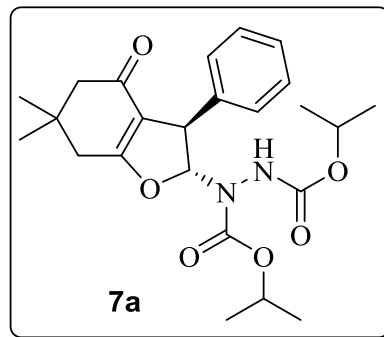


Fig S30. ¹H NMR Spectrum of 7a

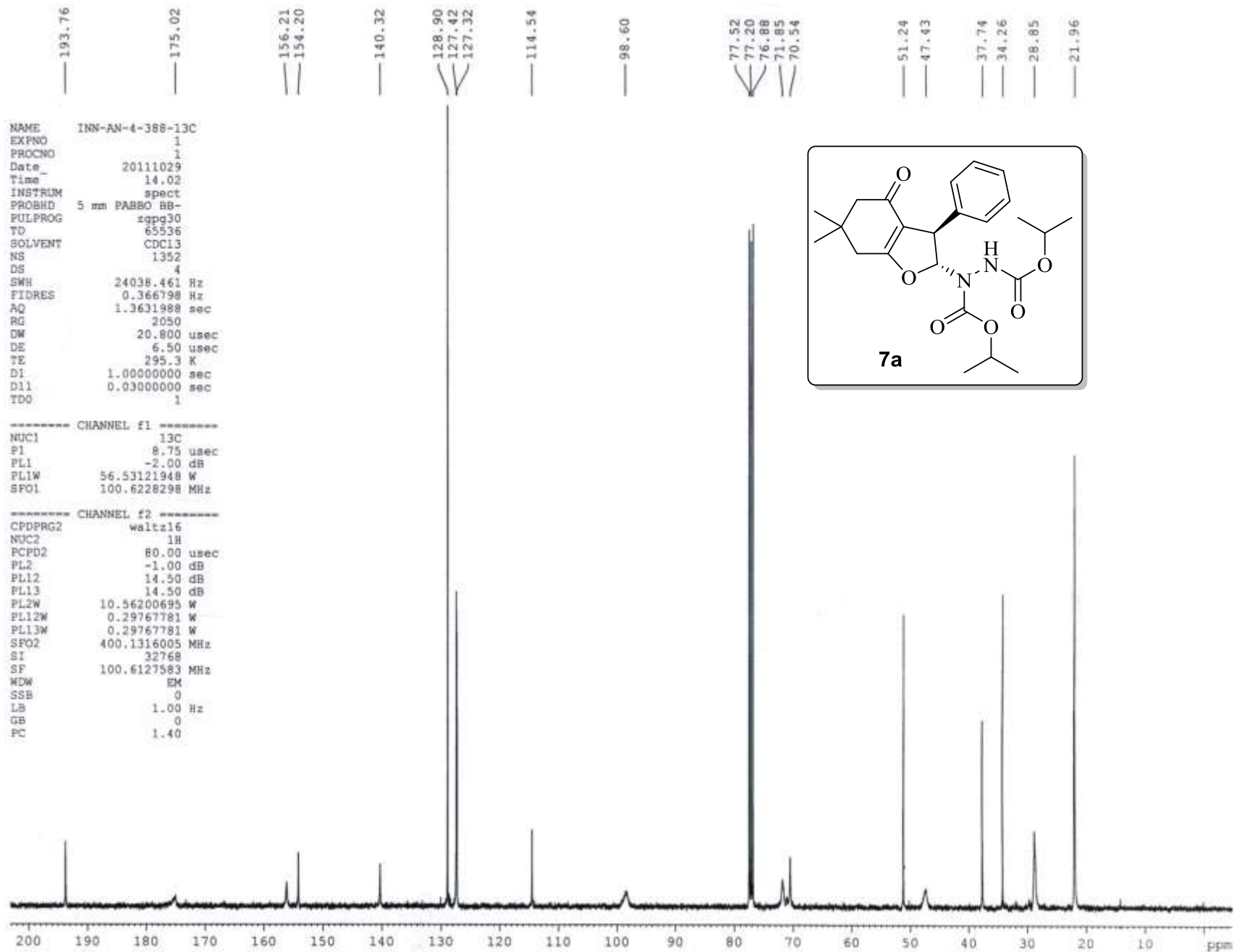
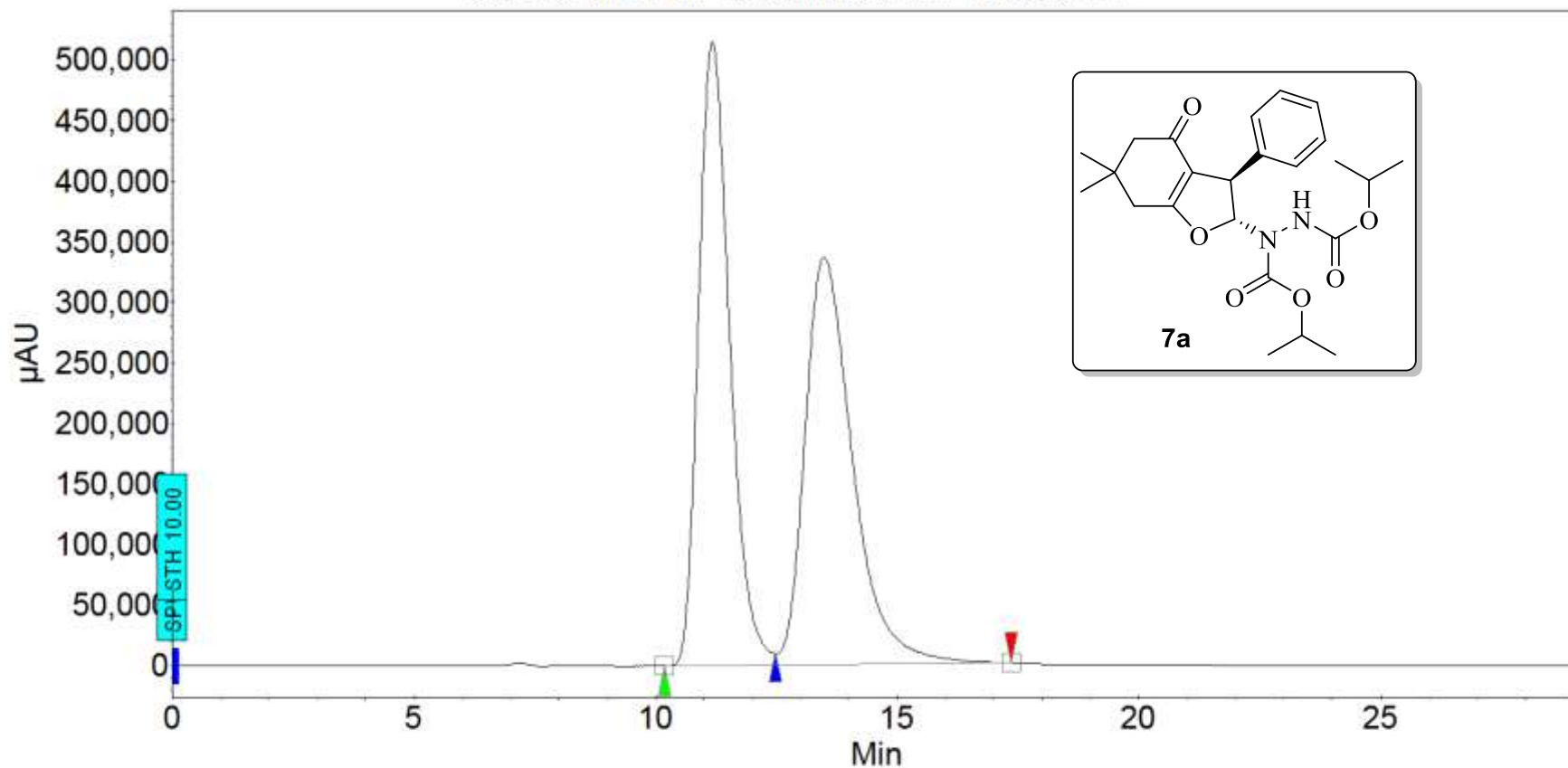


Fig S31. ^{13}C NMR Spectrum of 7a

ksb-4-ODH-1r-DF-racemic1.DATA - 268.00 nm

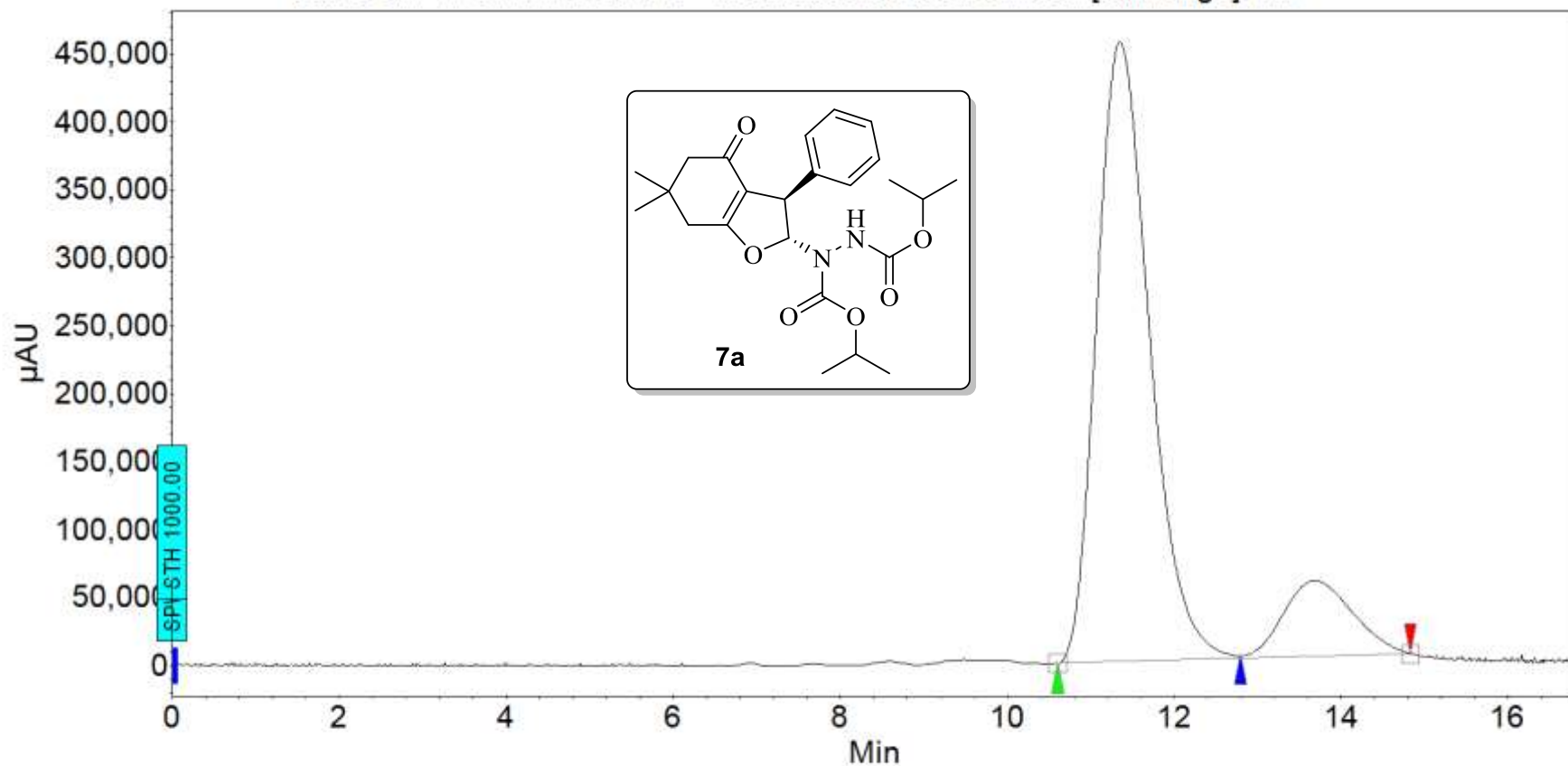


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
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

ksb-ODH-1r-CI-Mw-2.DATA - Max. absorbance between [full range] nm



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	11.346	86.29	455629.2	333196.7	86.290
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

Current Data Parameters
NAME inn-nss-dhf-me-1h
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20160602
Time_ 16.54
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 301.1 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 500.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

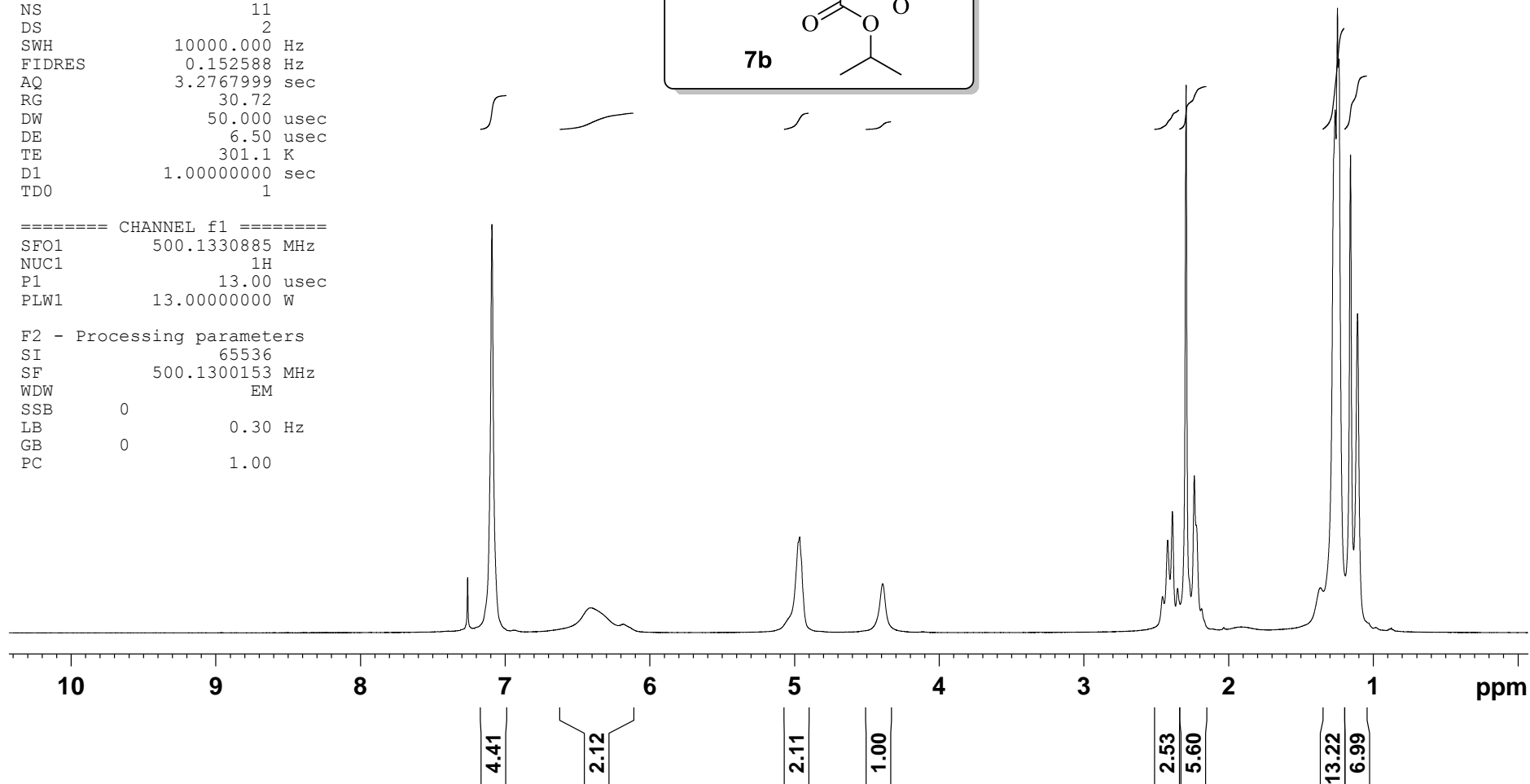
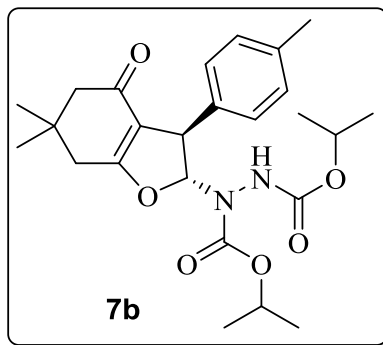


Fig S34. ¹H NMR Spectrum of 7b

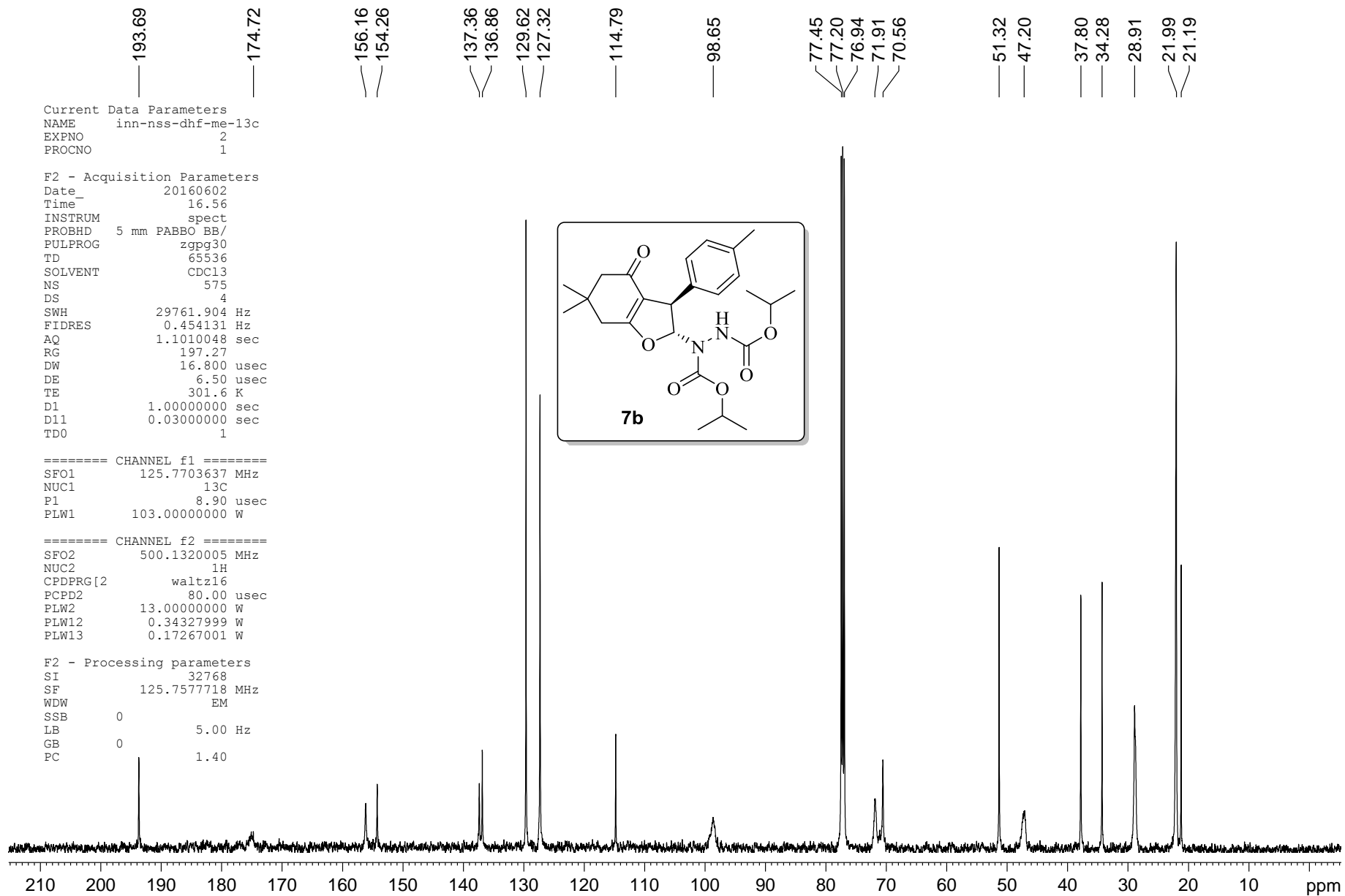
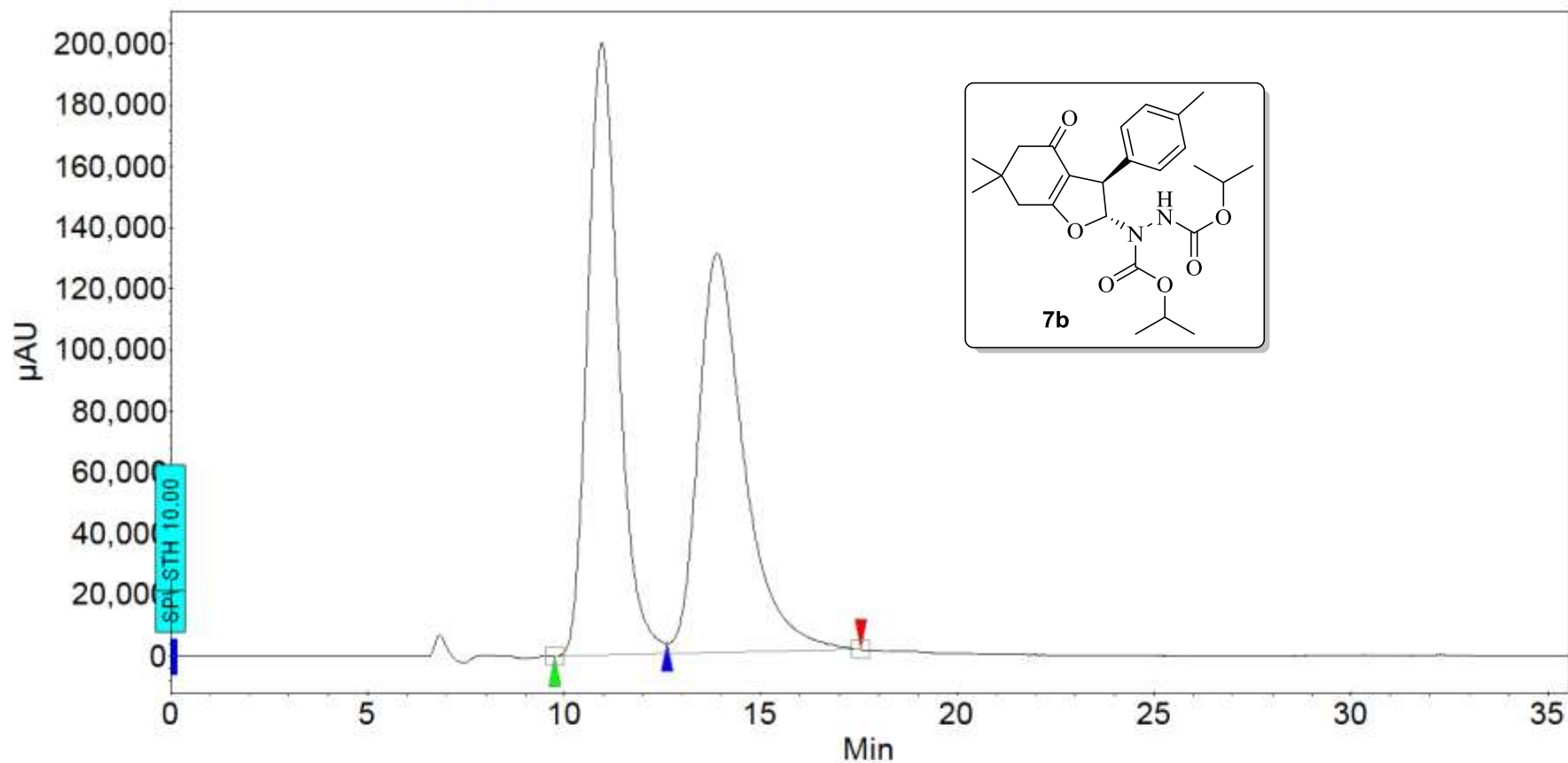


Fig S35. ¹³C NMR Spectrum of 7b

ksb-4-odh-1r-A-rac-150150.DATA - 268.00 nm

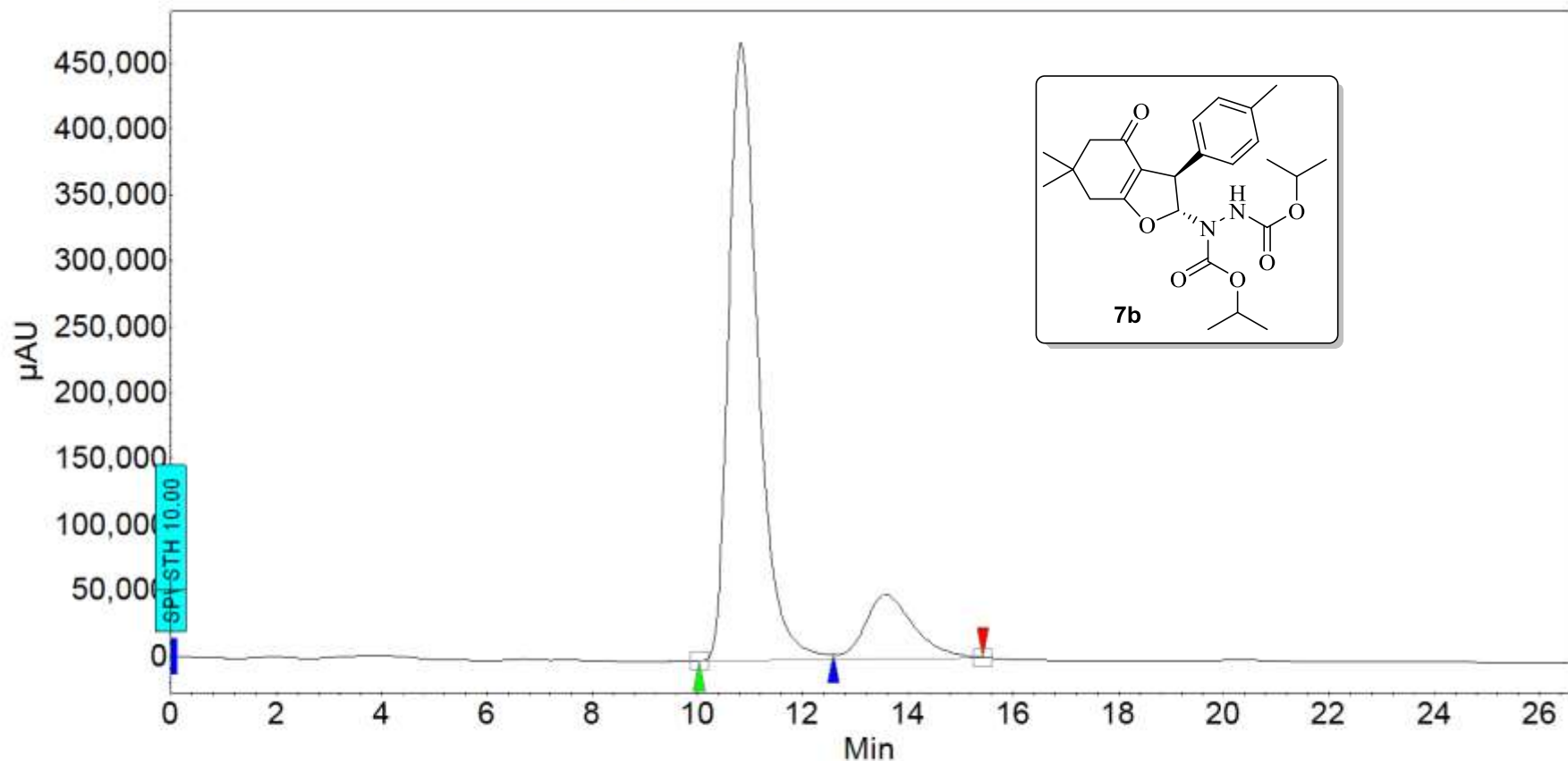


Peak results :

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

ksb-4-ODH-1r-A-en-150.DATA - 268.00 nm



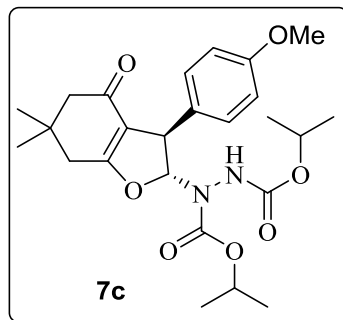
Peak results :

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

NAME INN-AN-4-387-1H
 EXPNO 1
 PROCNO 1
 Date_ 20111022
 Time_ 17.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 35
 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 294.1 K
 D1 1.00000000 sec
 TD(1

INN-AN-4-387-1H



===== 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

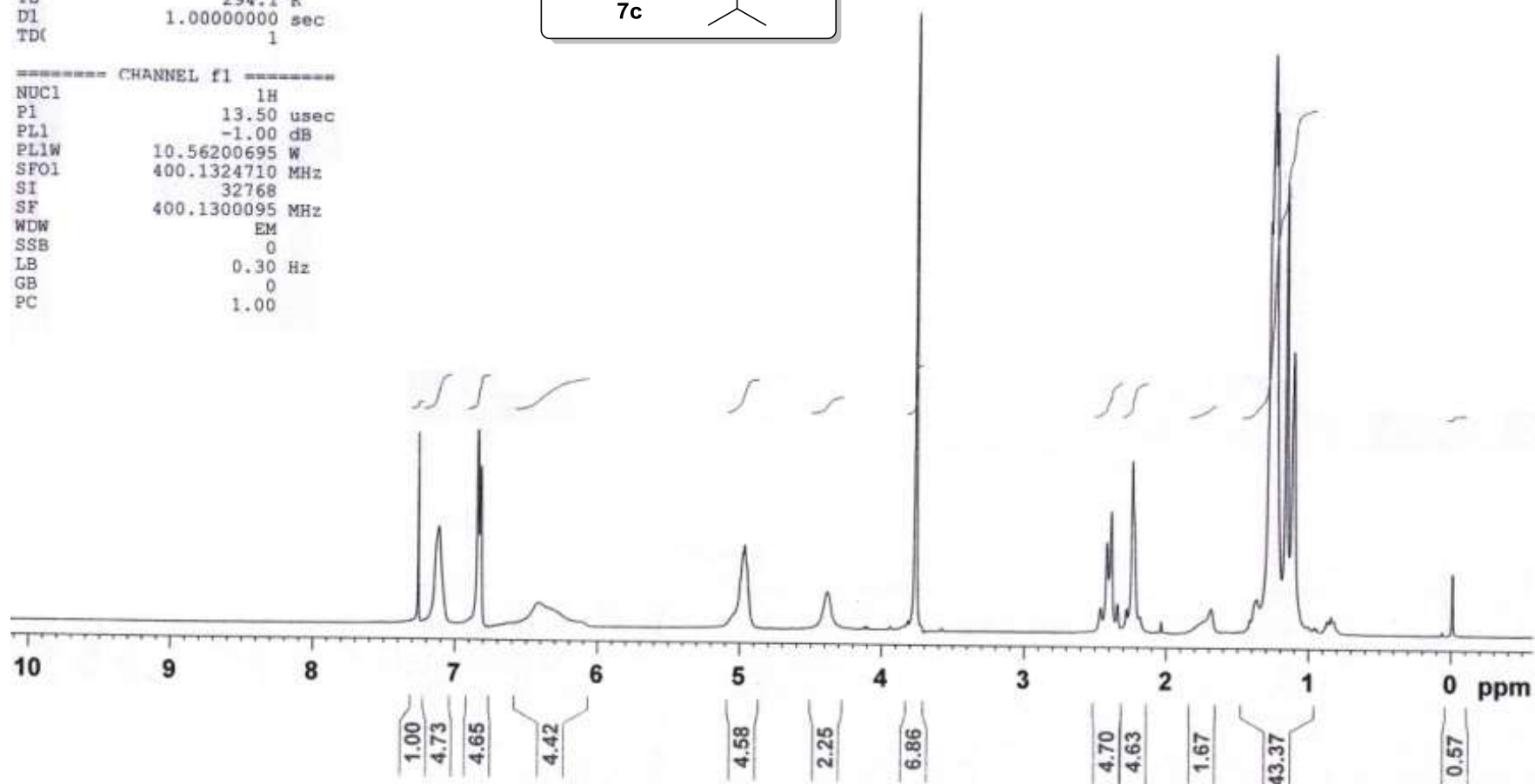


Fig S38. ¹H NMR Spectrum of 7c

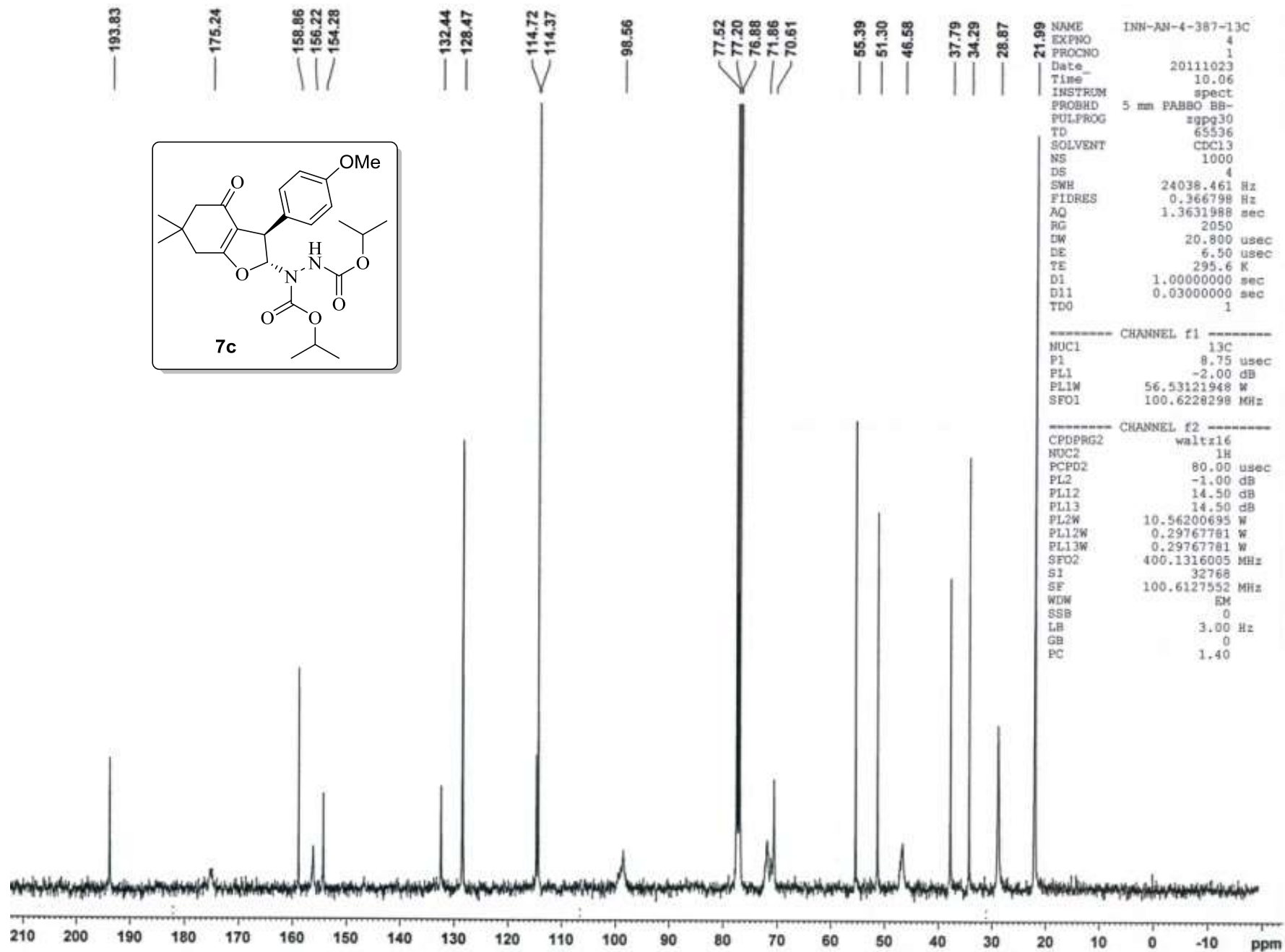
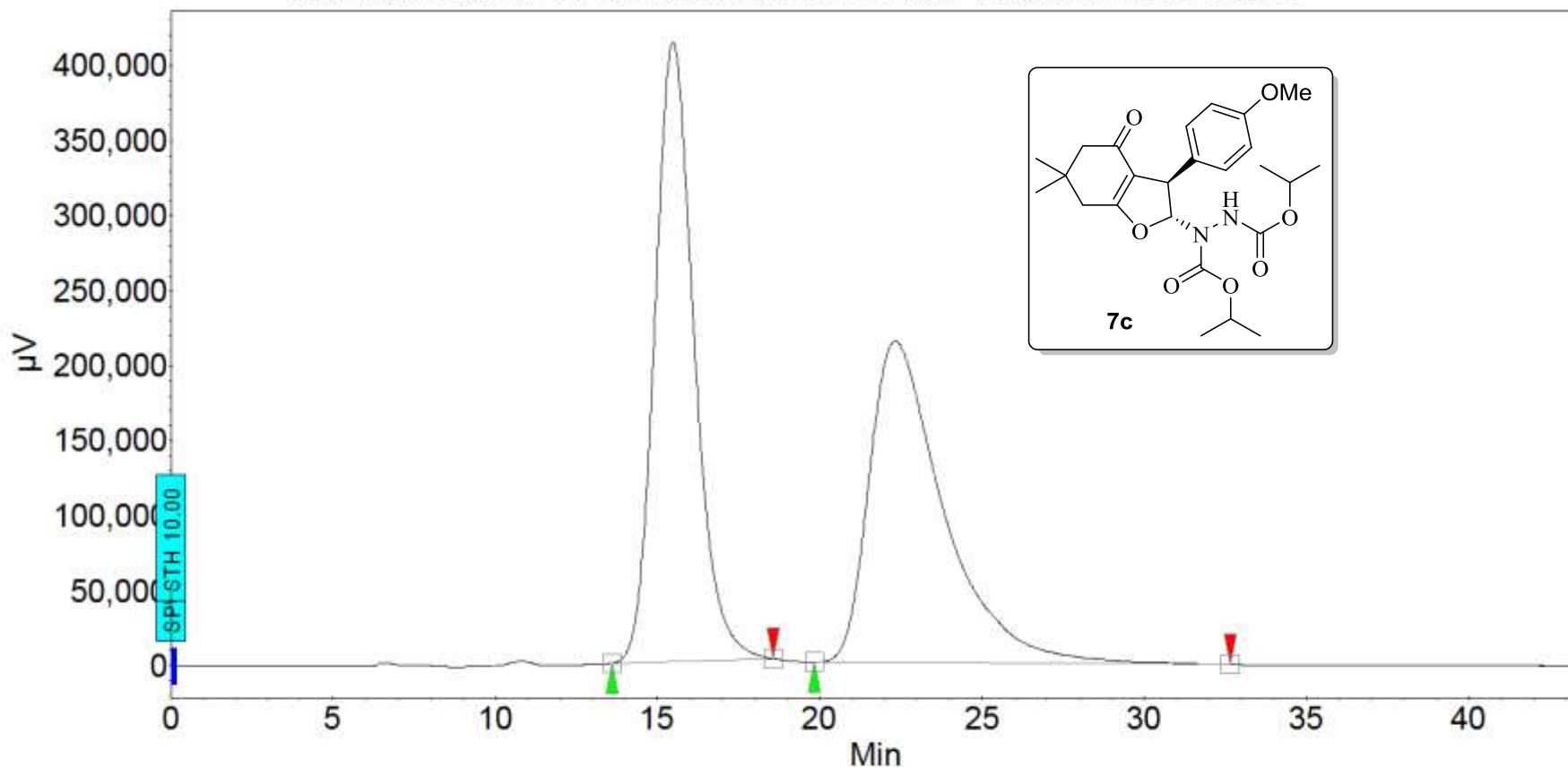


Fig S39. ¹³C NMR Spectrum of 7c



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

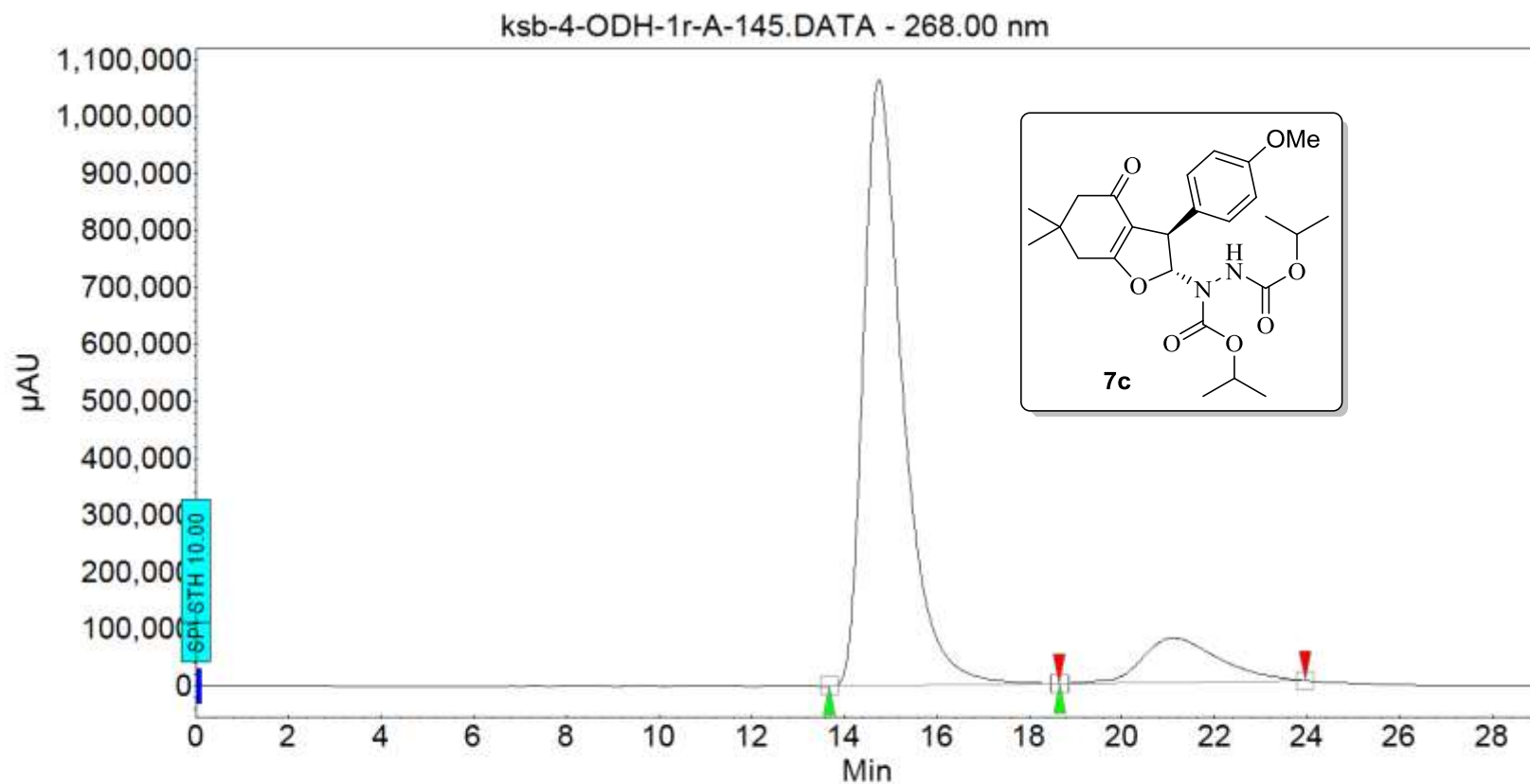
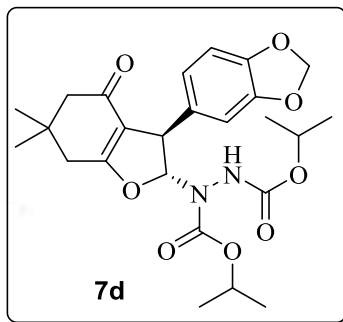


Fig S41. HPLC Profile of Enantioenriched 7c

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

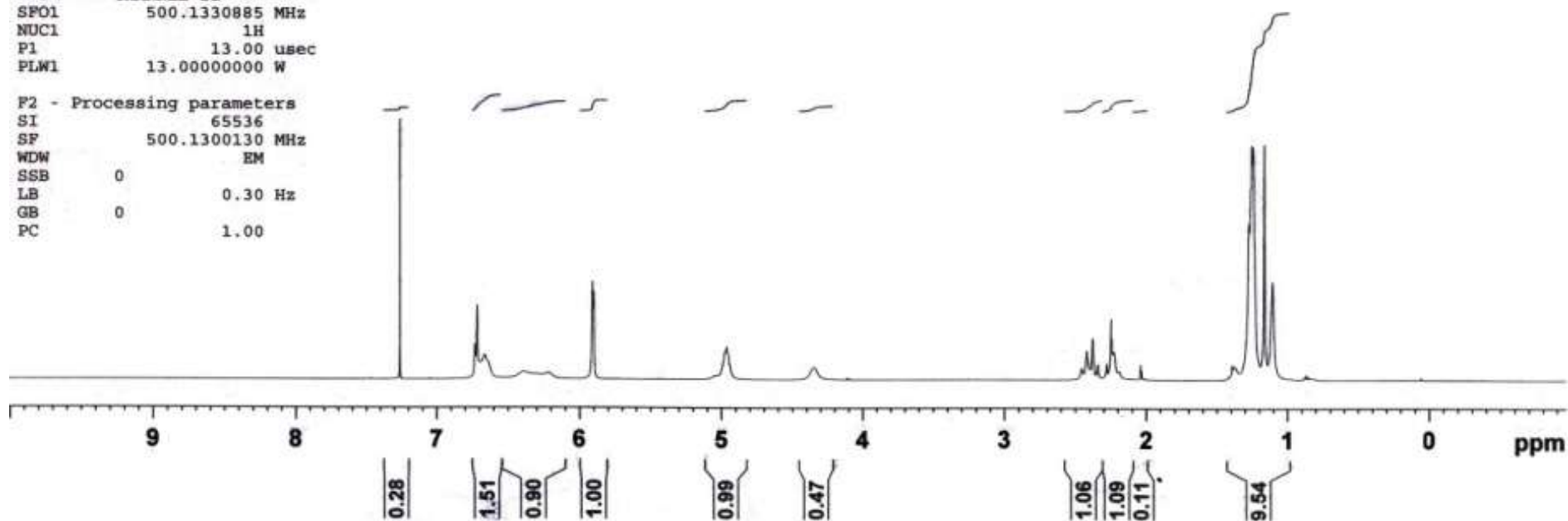


Fig S42. ¹H NMR Spectrum of 7d

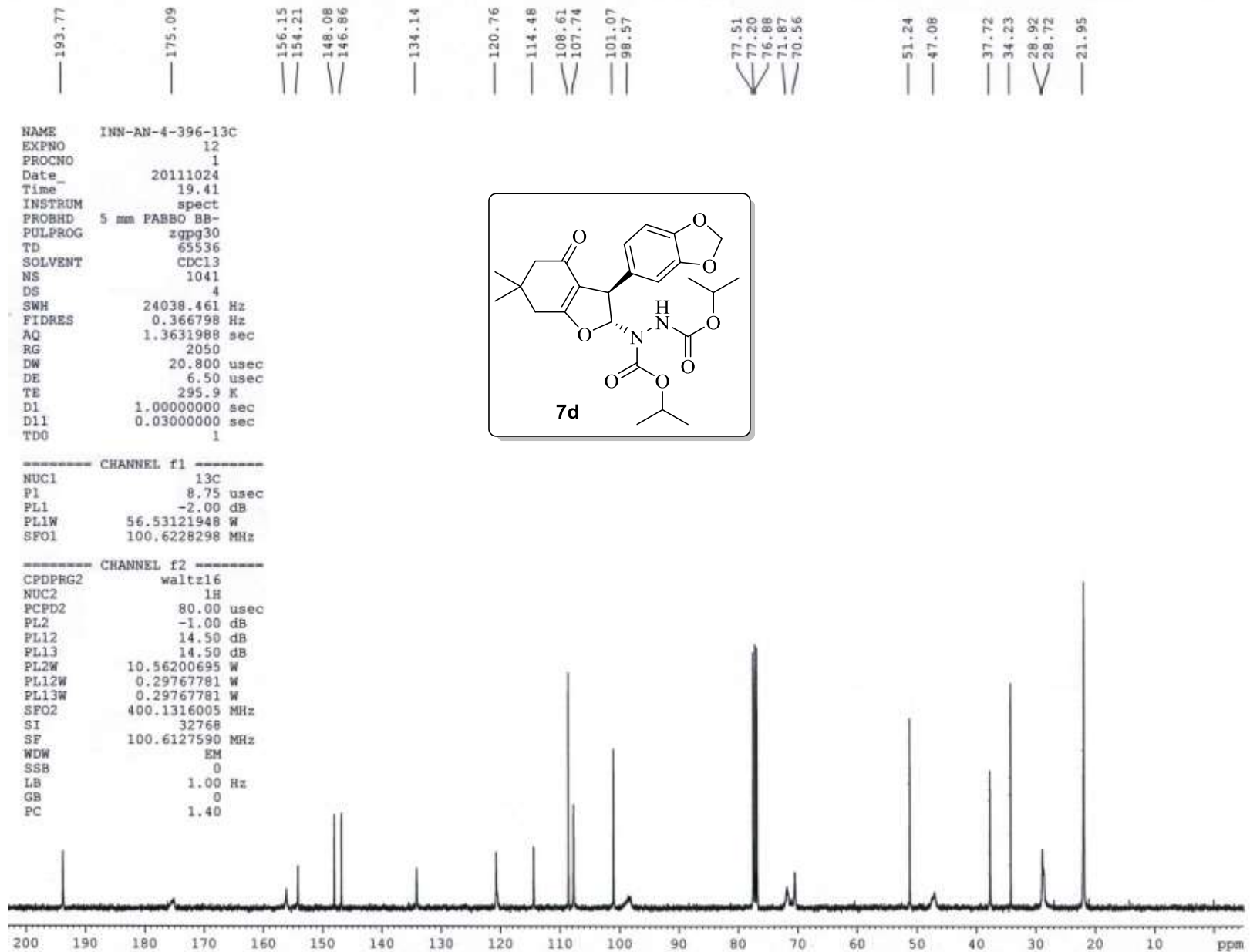
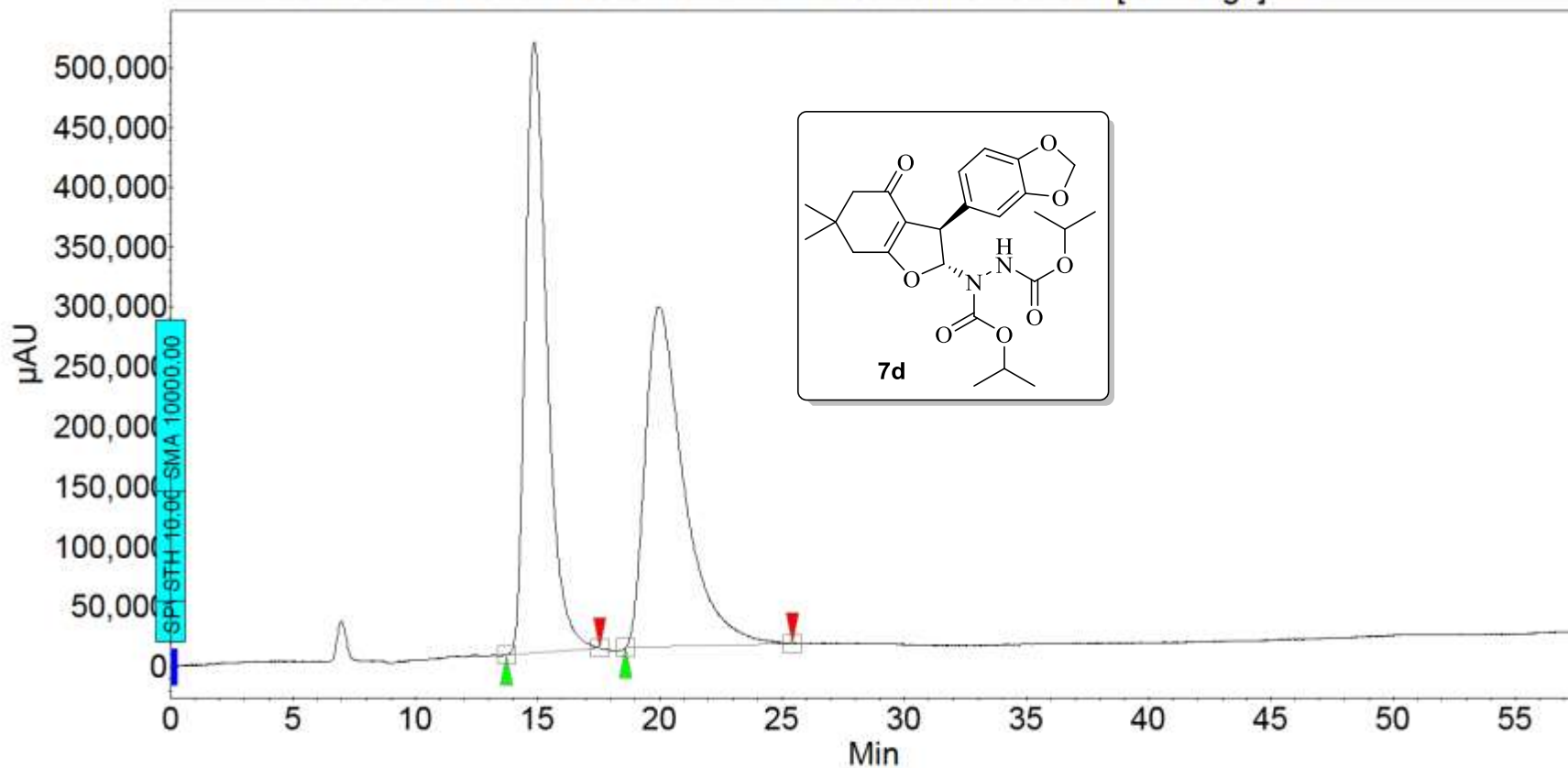


Fig S43. ^{13}C NMR Spectrum of 7d

ksb-4-ODH-1r-C-rac-150.DATA - Max. absorbance between [full range] nm

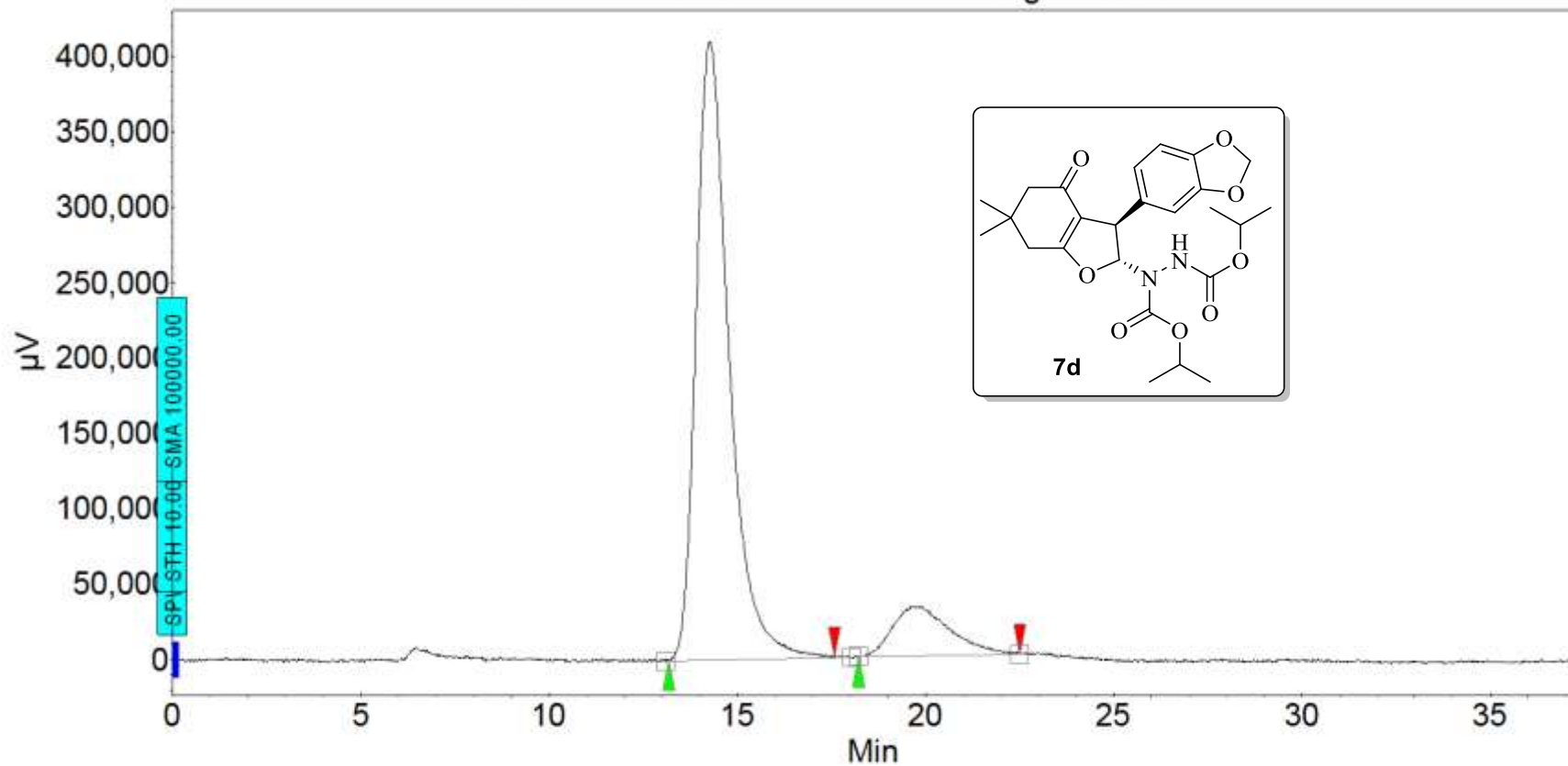


Peak results :

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

ksb-4-ODH-1r-C-en-150.DATA - Jasco Analog Channel 1



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


```

NAME      INN-AN-4-385-1H
EXPNO     5
PROCNO    1
Date_     20111018
Time      20.00
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         27
DS         0
SWH       12019.230 Hz
FIDRES    0.183399 Hz
AQ        2.7263477 sec
RG         114
DW        41.600 usec
DE        6.50 usec
TE        294.2 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        400.1300083 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

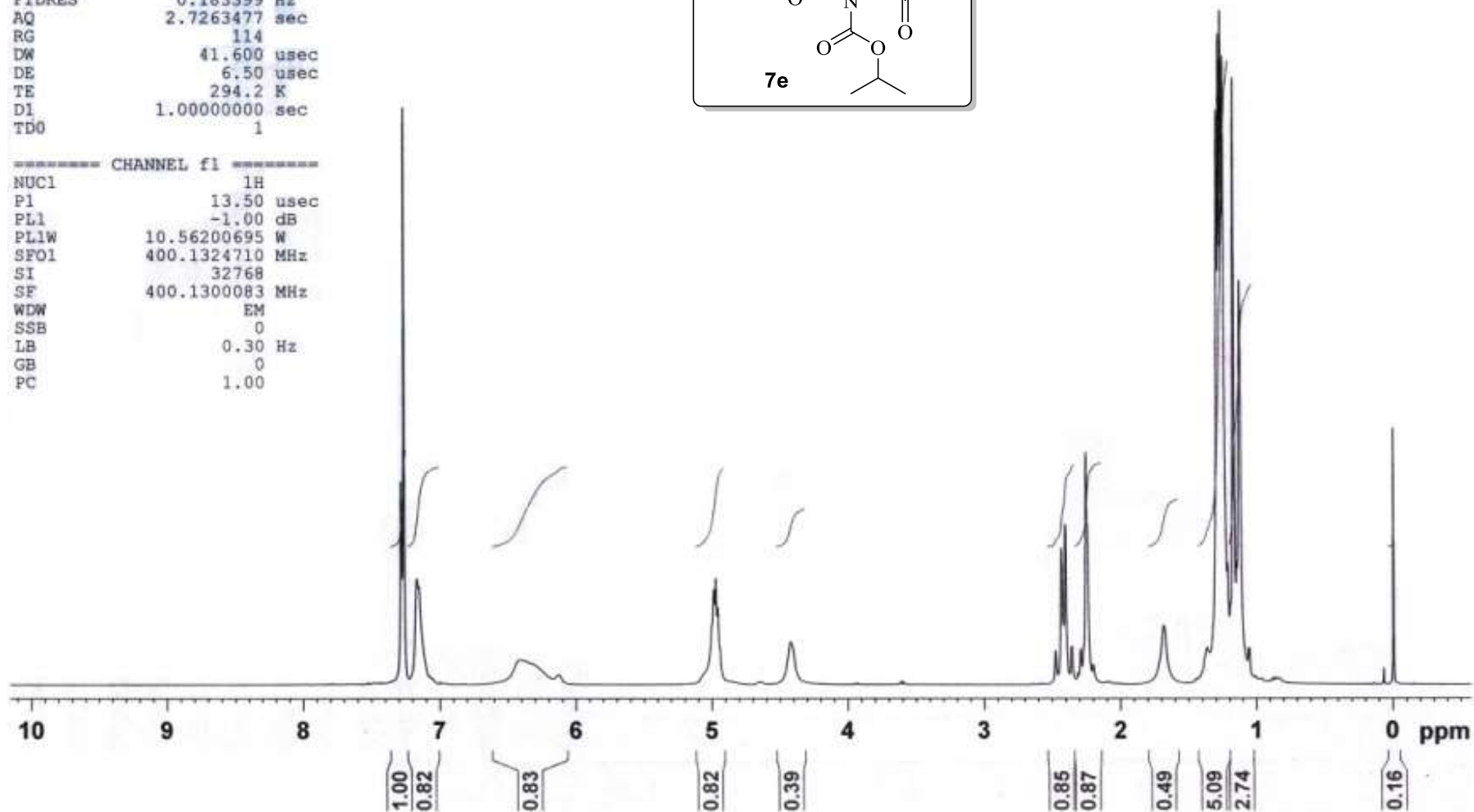
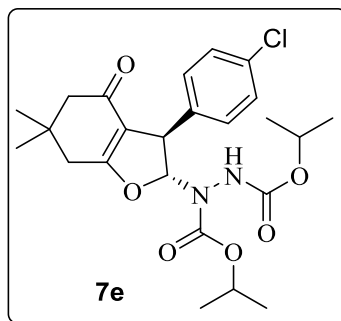


Fig S46. ¹H NMR Spectrum of 7e

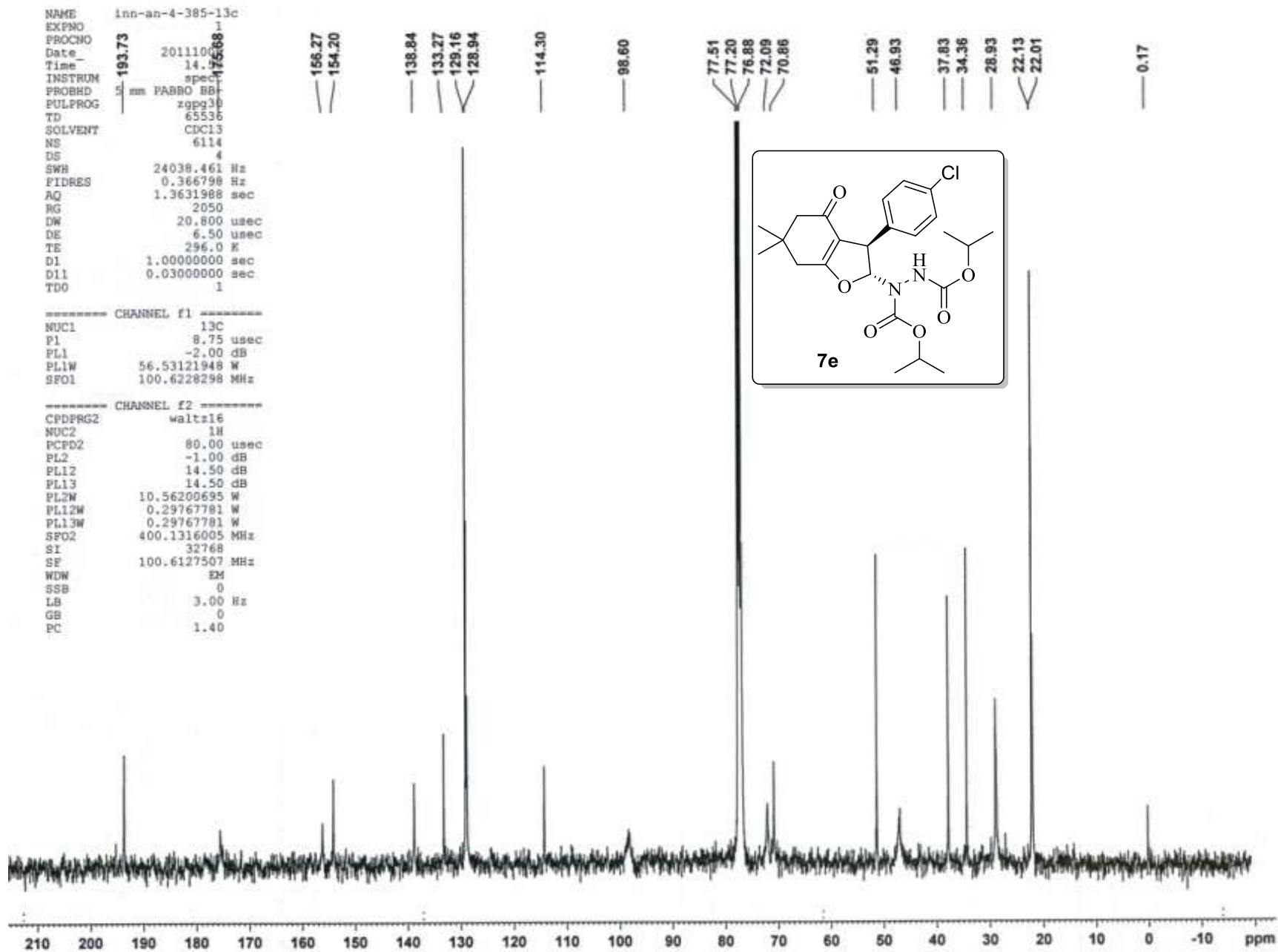
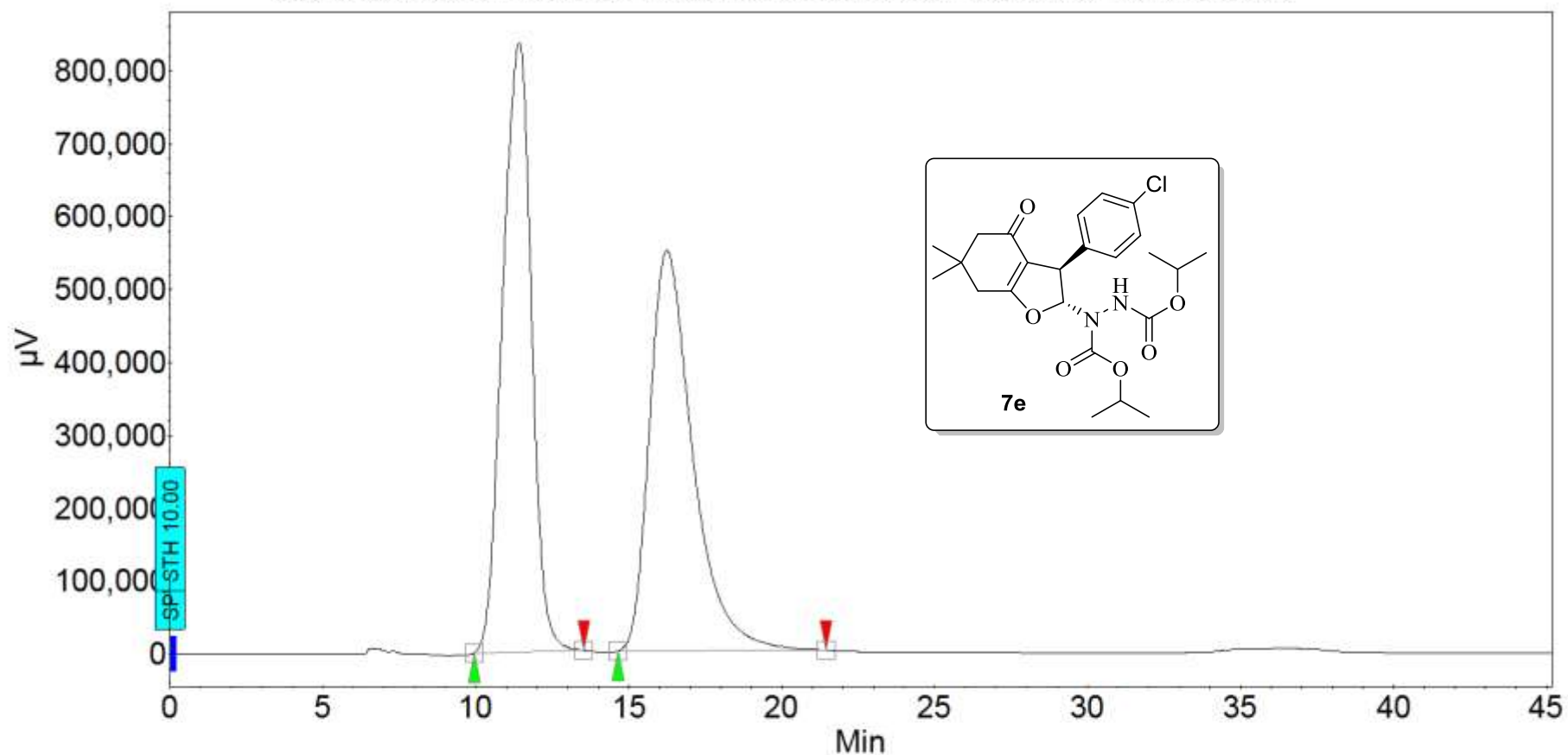


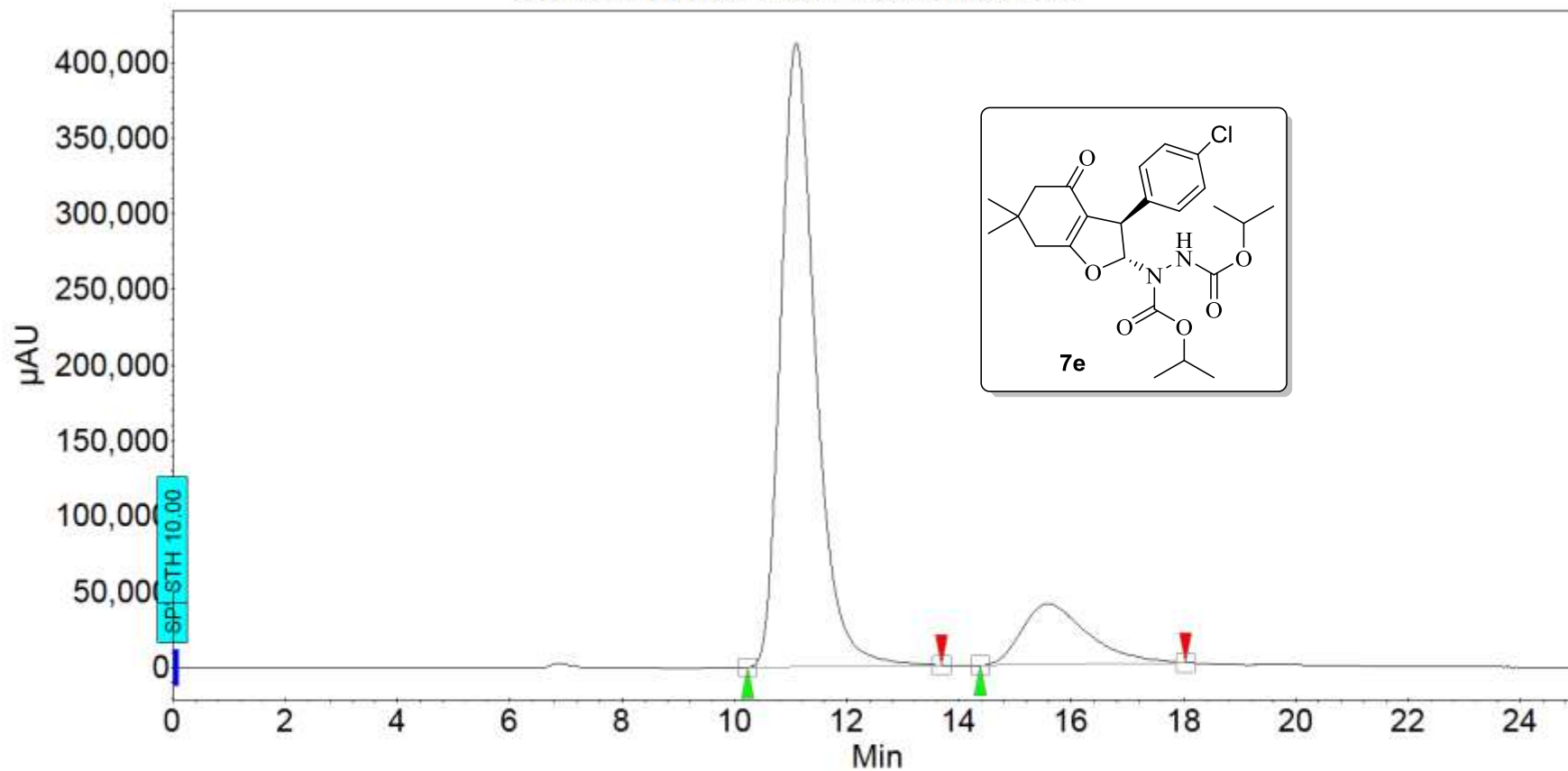
Fig S47. ¹³C NMR Spectrum of 7e

AN-4-385-2-rac-ODH-10IPA-05fr-264nm1.DATA - Jasco UV DETECTOR

**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

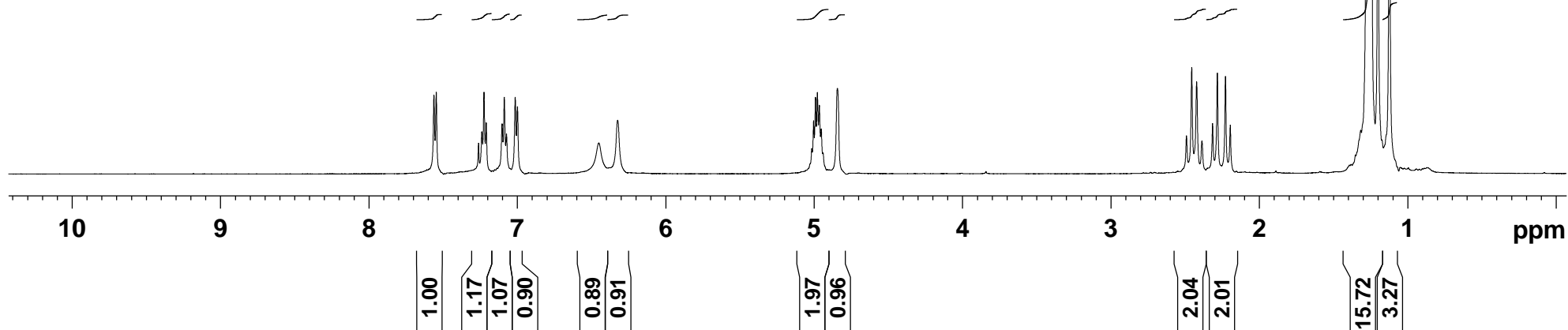
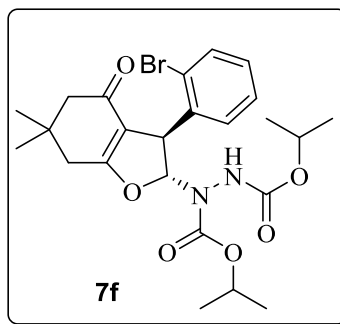


Fig S50. ¹H NMR Spectrum of 7f at +55 °C

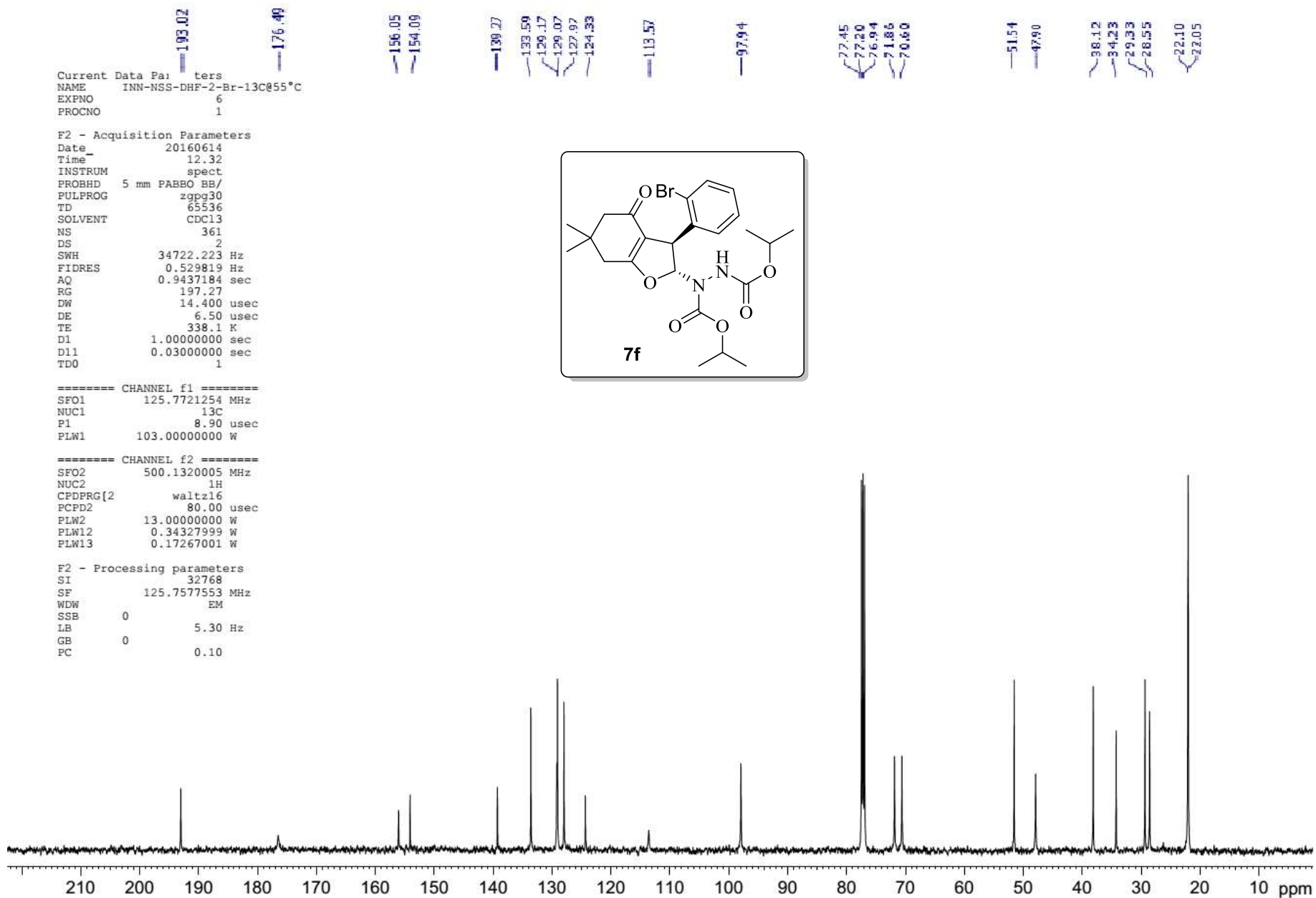


Fig S51. ¹³C NMR Spectrum of 7f at +55 °C

Current Data Parameters
NAME INN-NSS-DHF-2-Br-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160614
Time 11.06
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 48.36
DW 45.333 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 500.1300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

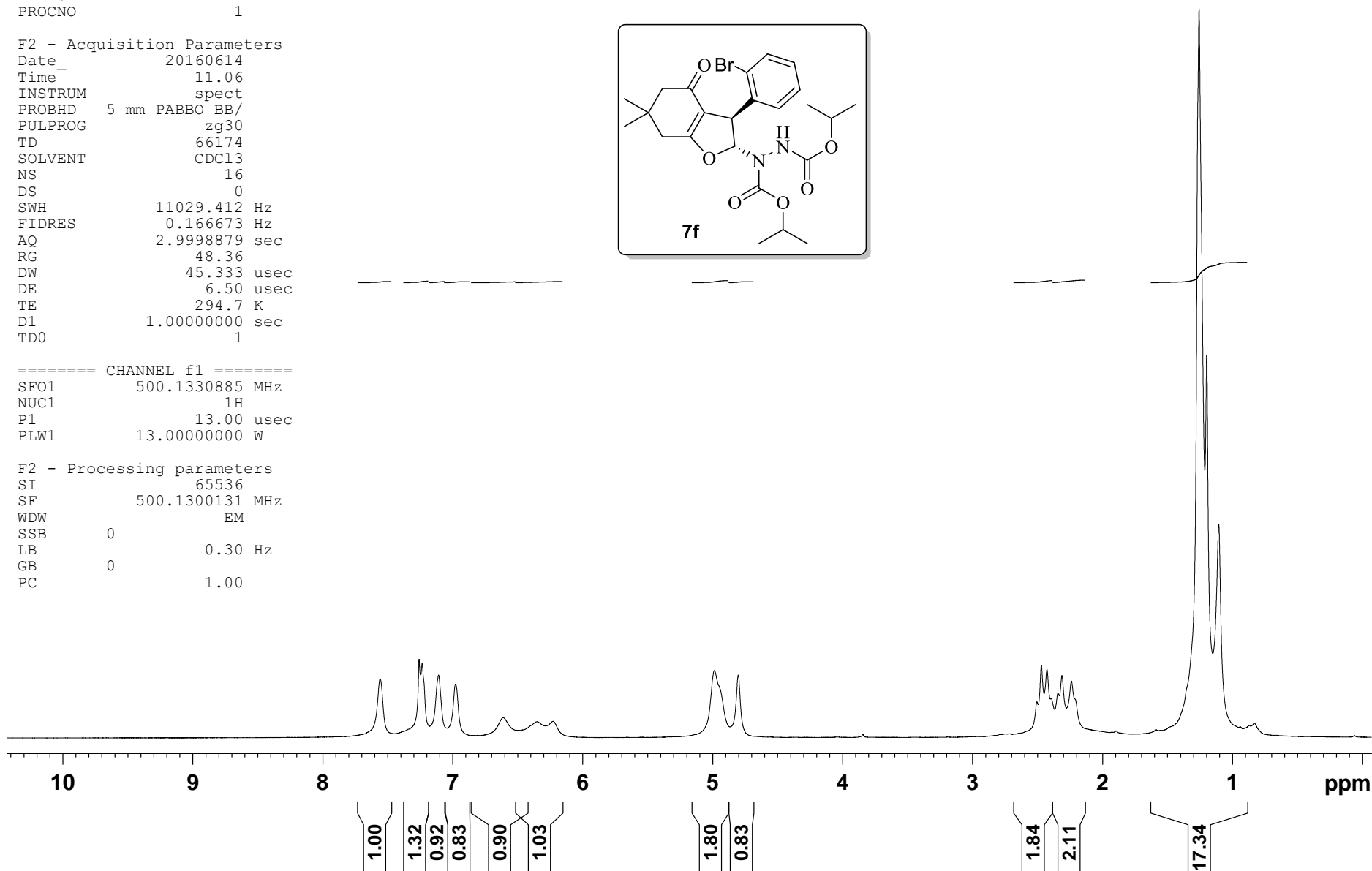
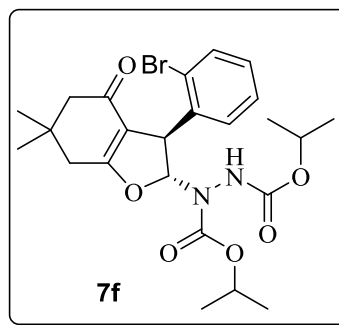


Fig S52. ¹H NMR Spectrum of 7f at +25 °C

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 CDC13
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

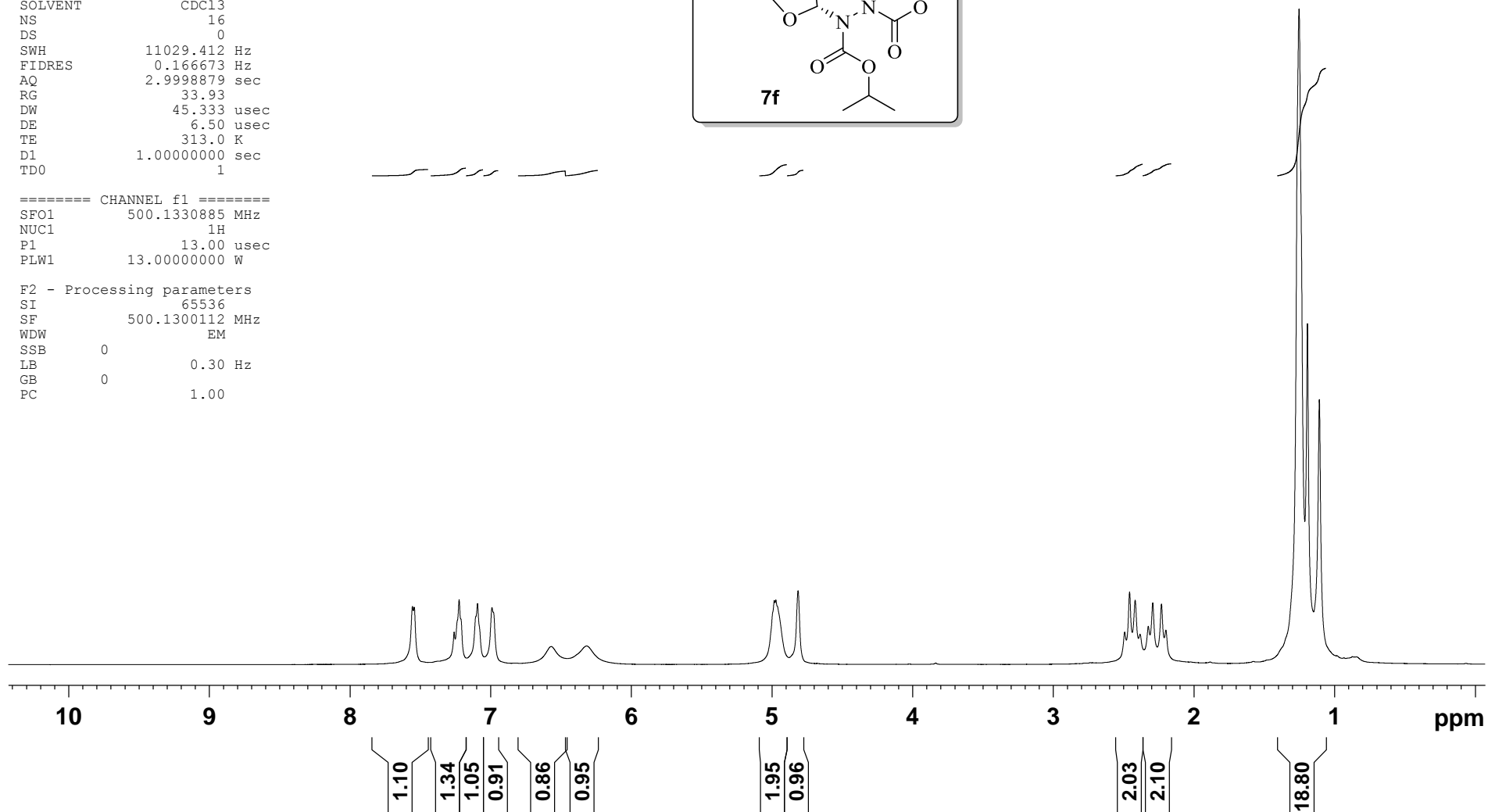
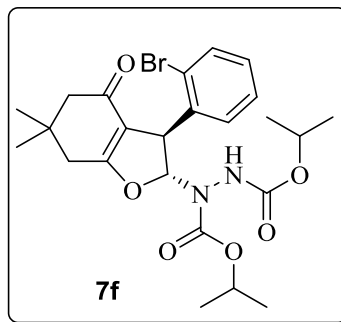


Fig S53. ¹H NMR Spectrum of 7f at +40 °C

Current Data Parameters
NAME INN-NSS-DHF-2-Br-1H@50°C
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20160614
Time_ 12.02
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 333.0 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

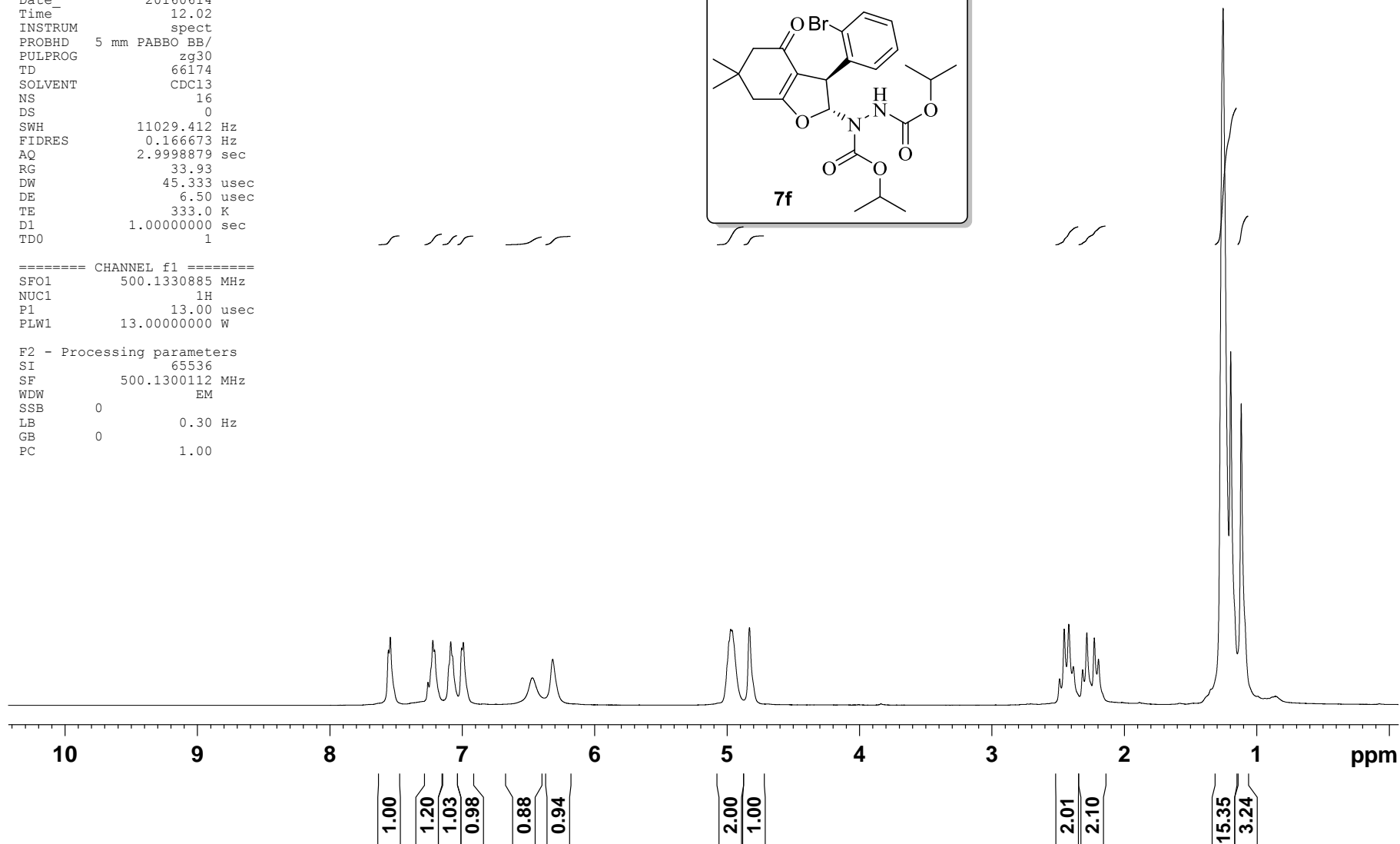
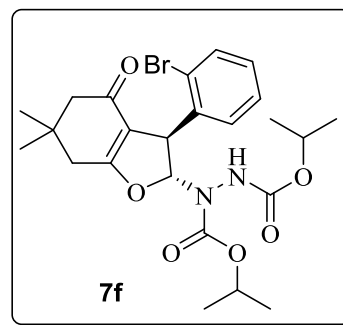
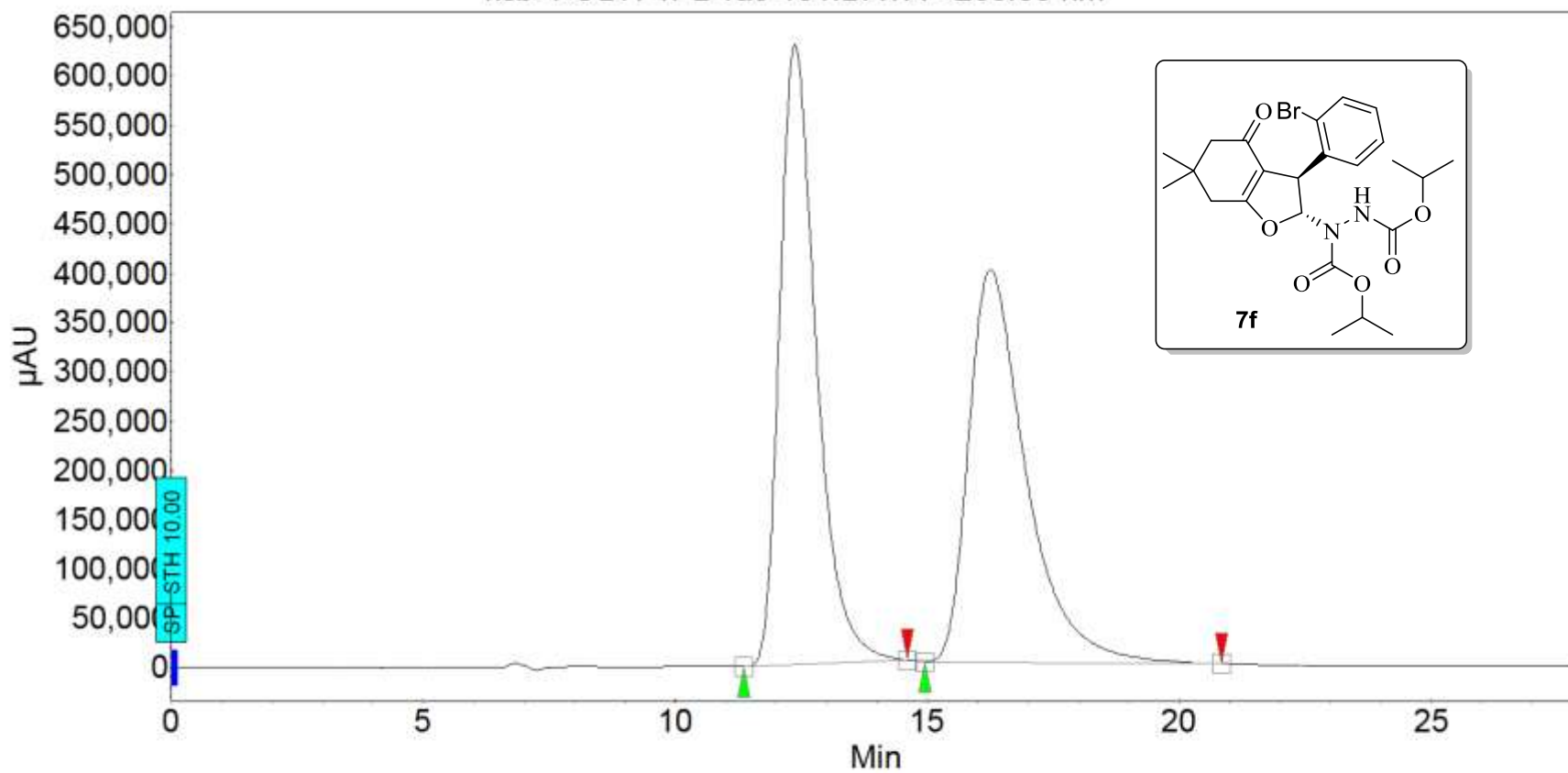


Fig S54. ¹H NMR Spectrum of 7f at +50 °C

ksb-4-ODH-1r-B-rac-151.DATA - 268.00 nm

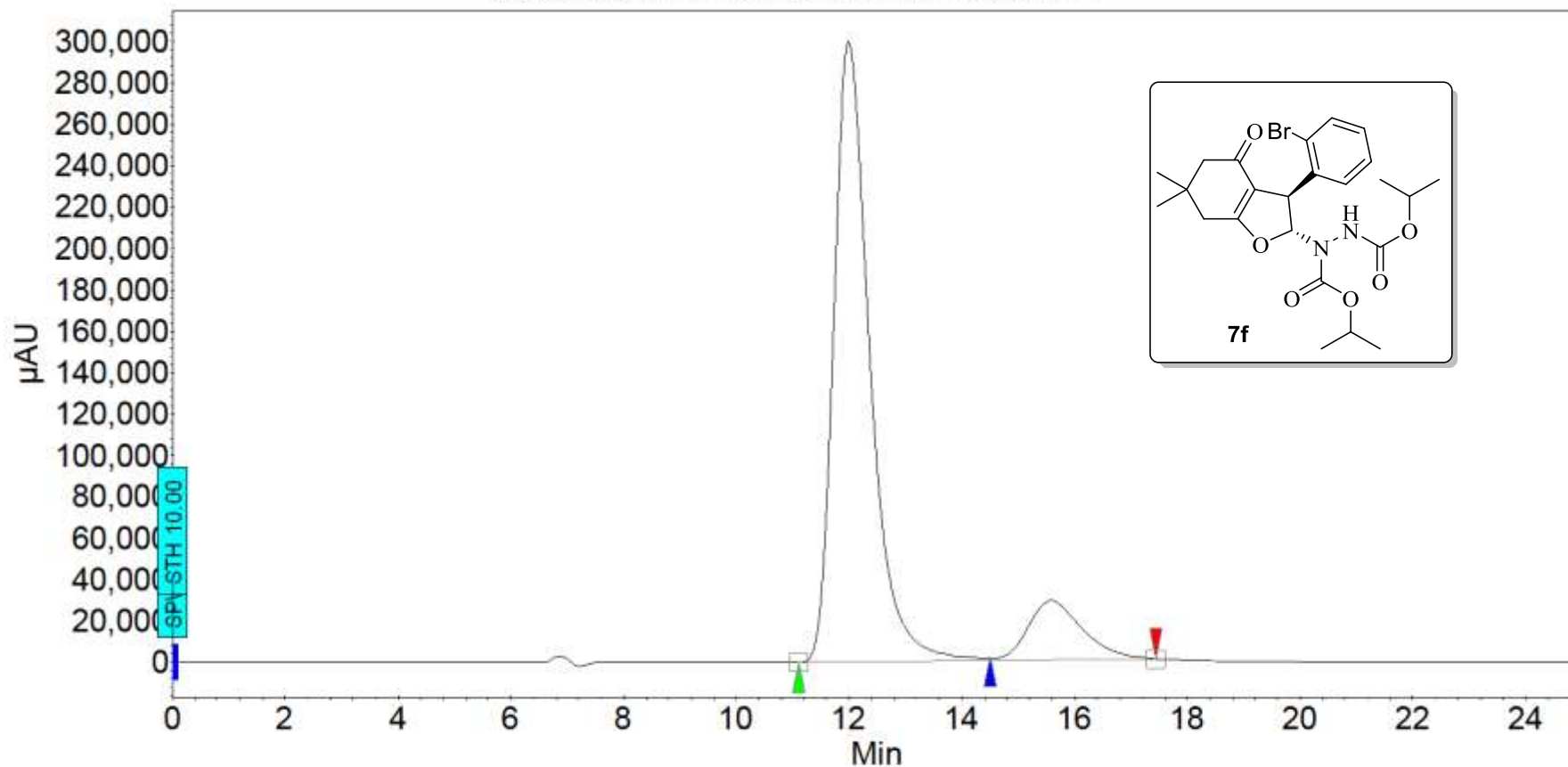


Peak results :

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

ksb-4-ODH-1r-B-en-151.DATA - 268.00 nm



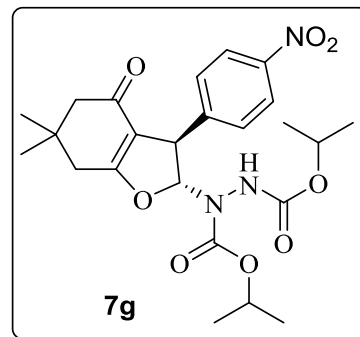
Peak results :

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

NAME INN-AN-4-395-1H
 EXPNO 1
 PROCNO 1
 Date_ 20111103
 Time_ 20.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 17
 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 294.6 K
 DL 1.00000000 sec
 TDO 1

INN-AN-4-395-1H



===== 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

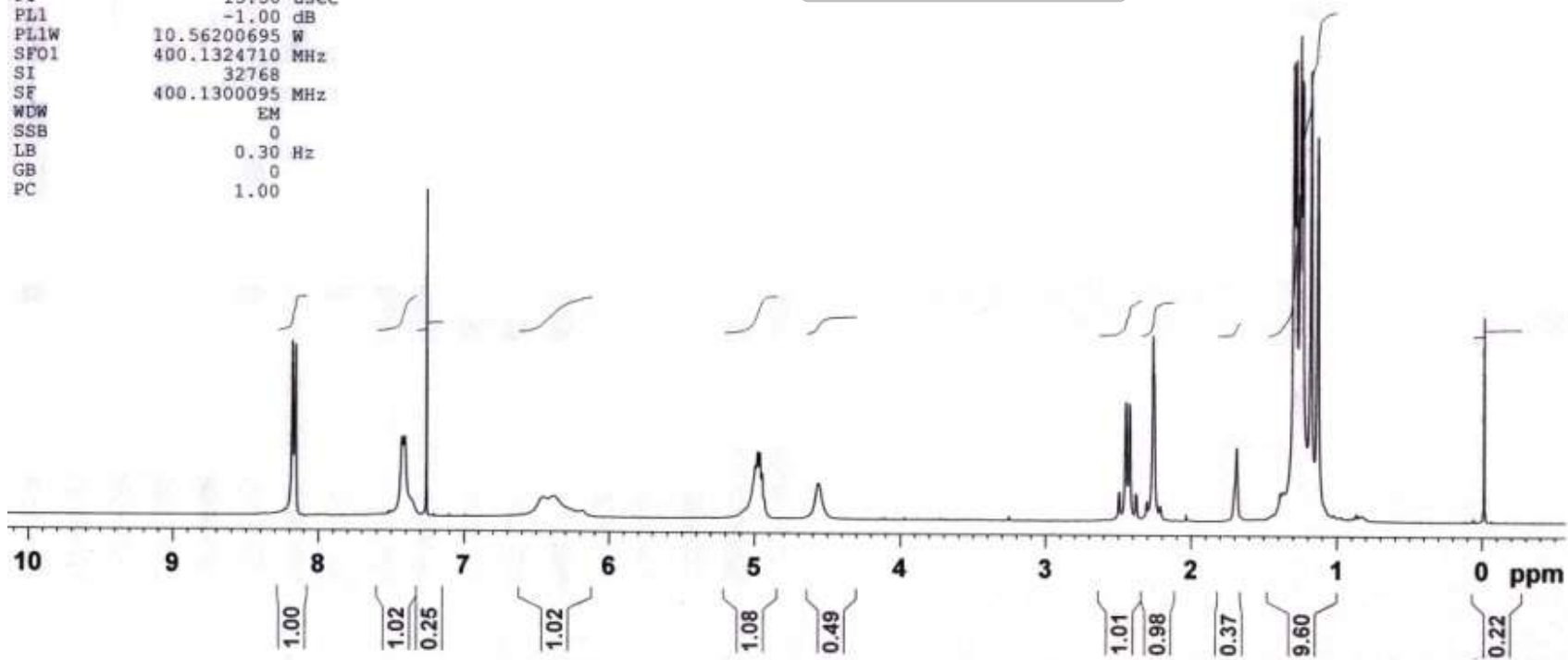


Fig S57. ¹H NMR Spectrum of 7g

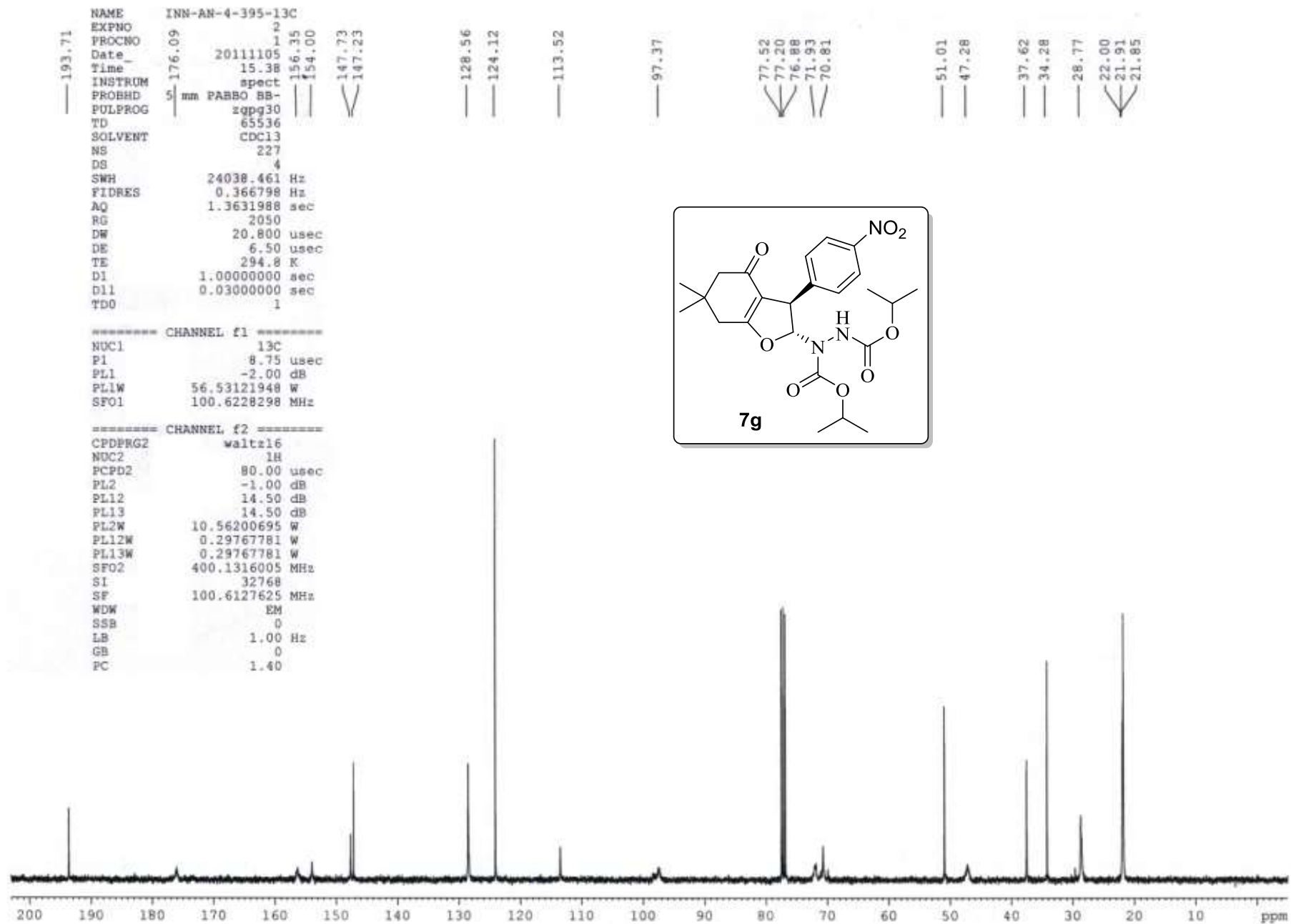
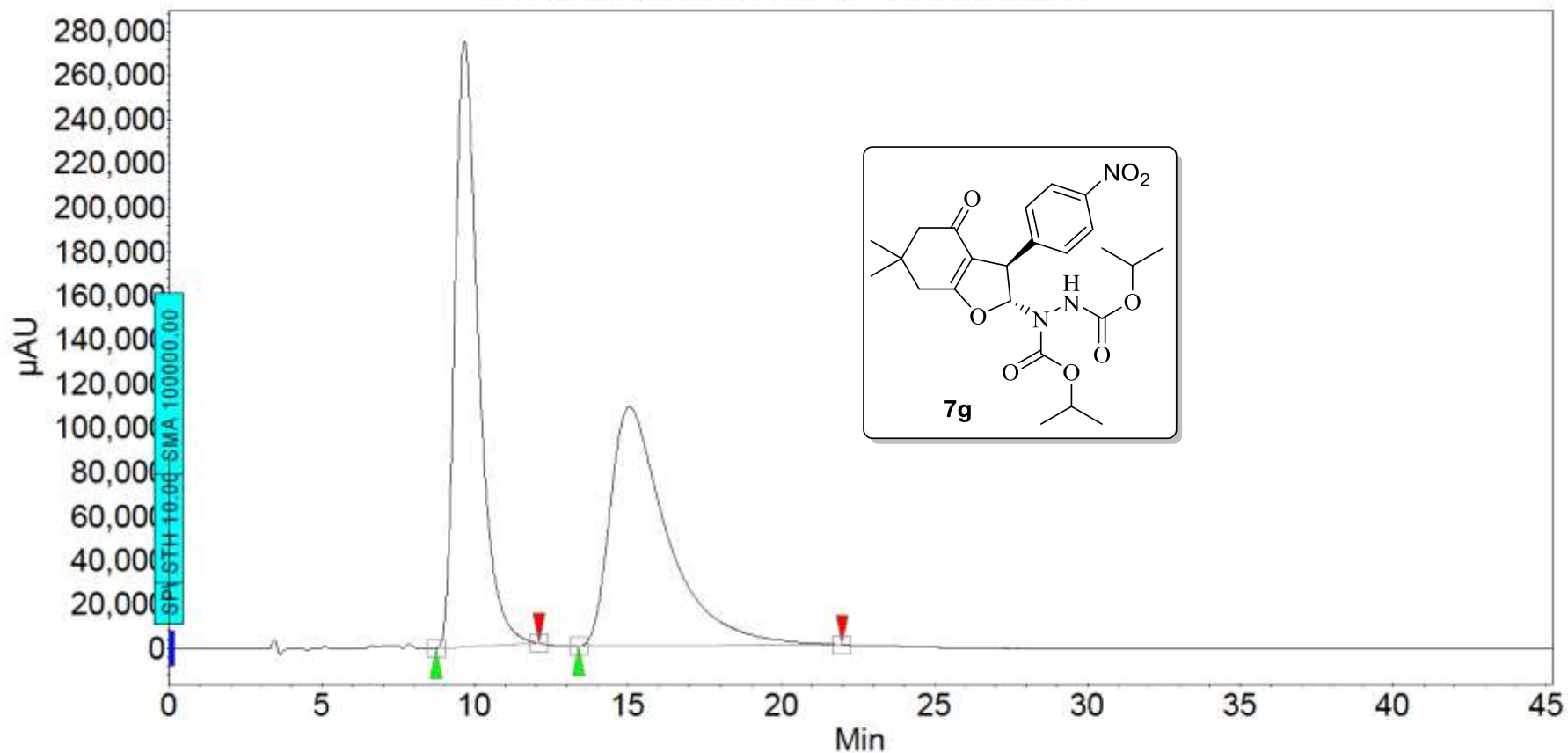


Fig S58. ¹³C NMR Spectrum of 7g

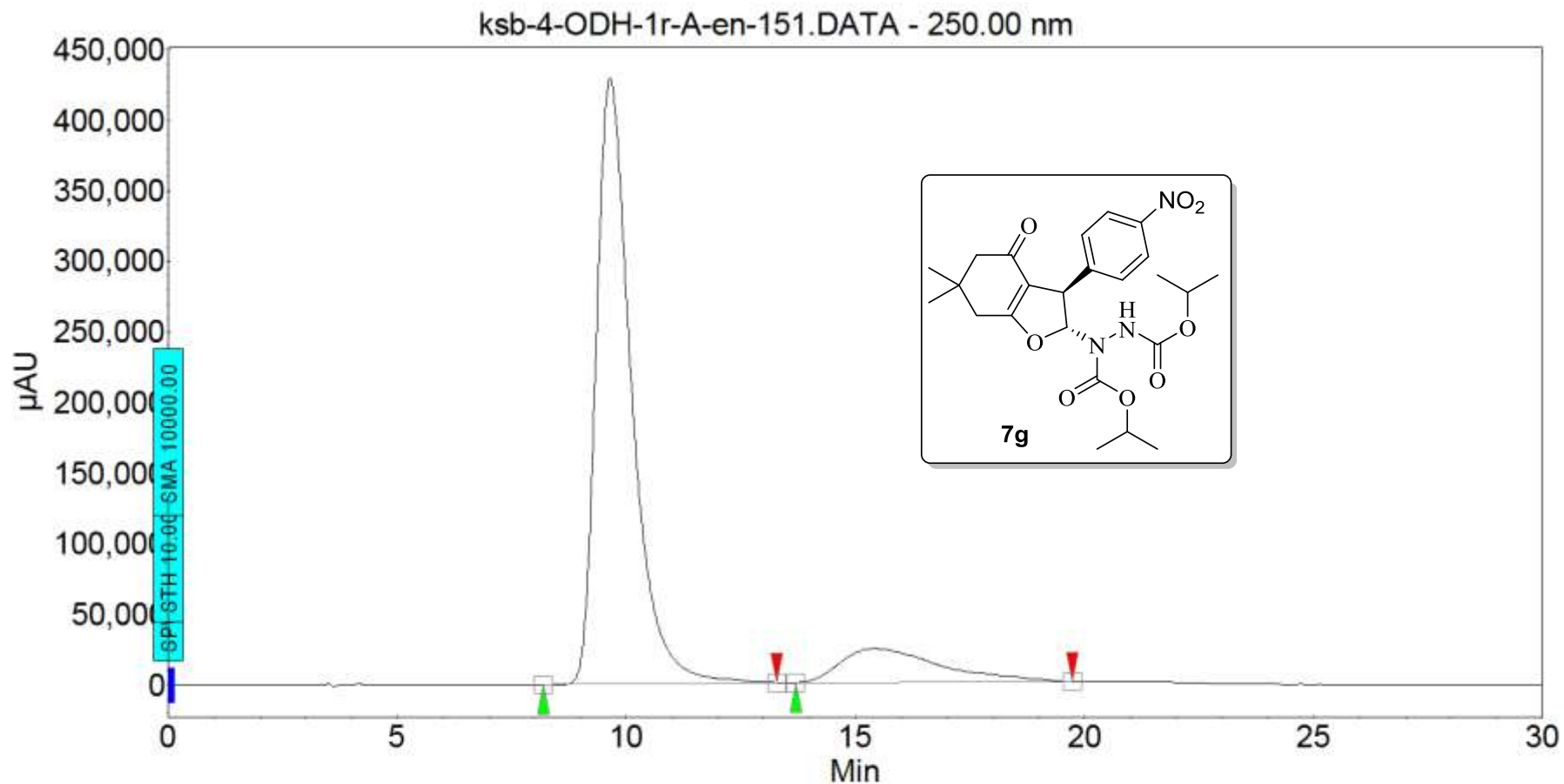
ksb-4-ODH-2r-A-rac-151.DATA - 250.00 nm



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

INN-4-KSB-150B

exp2 s2pu1

SAMPLE		DEC. & VT	
date	Mar 19 2014	dn	HM
solvent	CDCl3	dof	-268.4
file	exp	dm	nnn
ACQUISITION			
sfrq	299.950	dsm	c
tn	H1	daf	200
at	2.000	temp	55.0
PROCESSING			
np	24022	lb	0.10
sw	6006.0	fn	not used
fb	not used		
bs	2	werr	
pw	3.0	wexp	
pw	3.0	wbs	
tpwr	60	wnt	
d1	0		
tof	900.0	DISPLAY	
nt	1600	sp	-10.5
ct	374	wp	3611.2
alock	n	vs	151
gain	2	sc	0
		wc	250
FLAGS			
ll	n	hzmm	14.44
in	n	is	3681.75
dp	y	rfl	650.9
		rfp	0
		th	1
		ins	76.300
		nm	ph

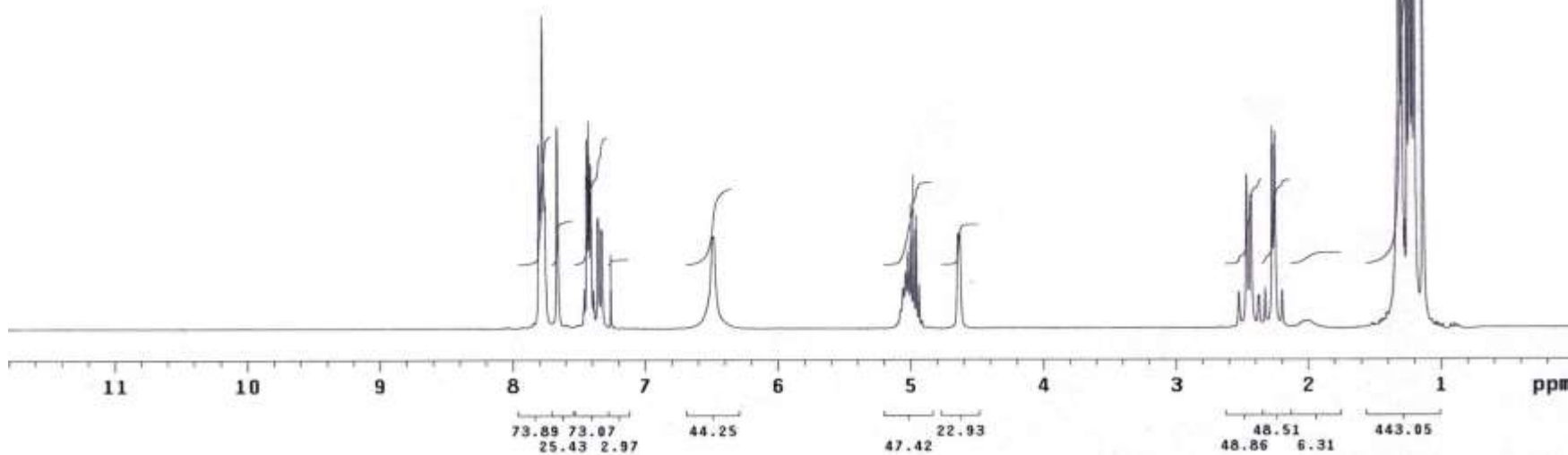
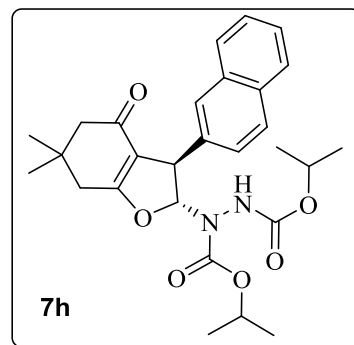


Fig S61. ¹H NMR Spectrum of 7h at + 55 °C

INN-4-KSB-150B

exp3 s2pu1

SAMPLE		SPECIAL	
date	Mar 19 2014	temp	55.0
solvent	CDCl3	gain	2
file	exp	spin	20
ACQUISITION			
sw	25000.0	hst	0.008
at	1.280	pw90	9.500
np	64000	alfa	20.000
fb	13800	FLAGS	
bs	4	il	n
d1	3.000	in	n
nt	4800	dp	y
ct	400	hs	nn
TRANSMITTER		PROCESSING	
tn	C13	lb	4.00
sfrq	75.427	fn	not used
tof	-2000.0	DISPLAY	
tpwr	59	sp	-88.6
pw	4.750	wp	16775.5
DECOUPLER		rfl	12778.5
dn	H1	rff	5815.0
dof	0	rp	-104.7
dm	yyy	lp	-340.8
dmm	w	PLOT	
dpwr	39	wc	250
daf	10900	sc	0
		vs	62
		th	3
		nm	cdc ph

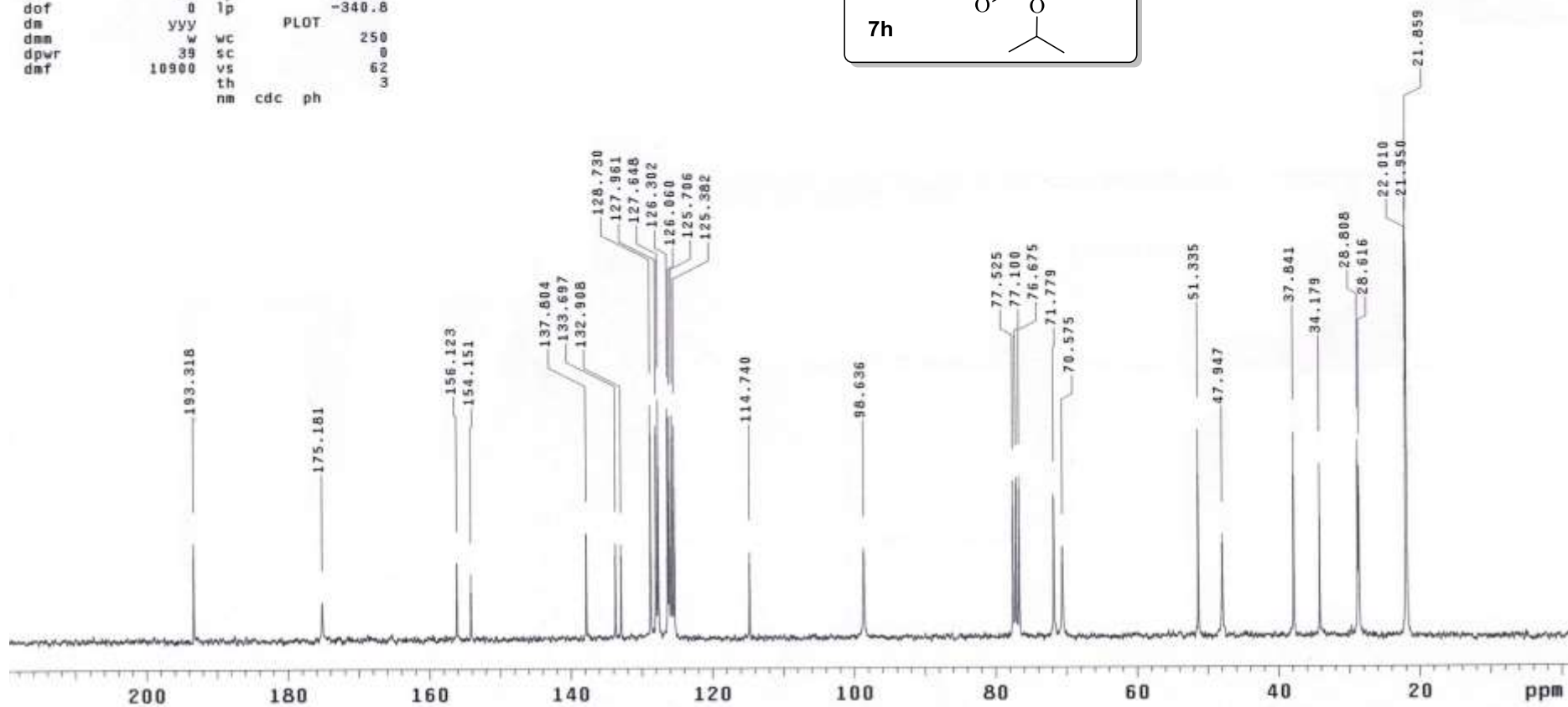
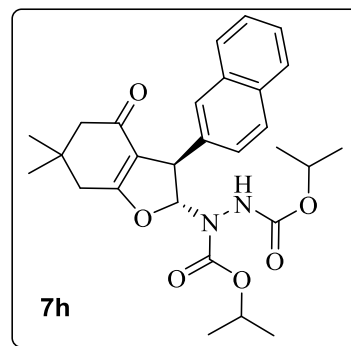


Fig S62. ¹³C NMR Spectrum of 7h at +55 °C

Current Data Parameters
NAME INN-4-KSB-150B-1H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140127
Time 22.14
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 30.72
DW 50.000 usec
DE 6.50 usec
TE 299.1 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

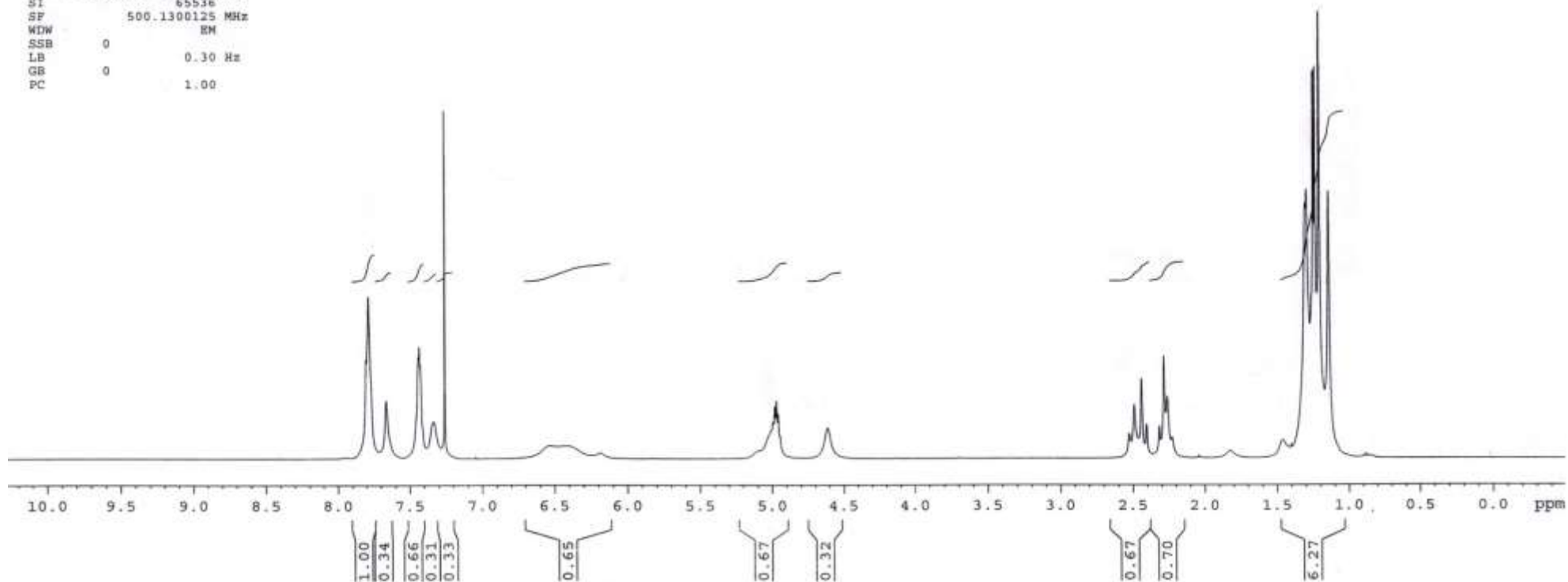
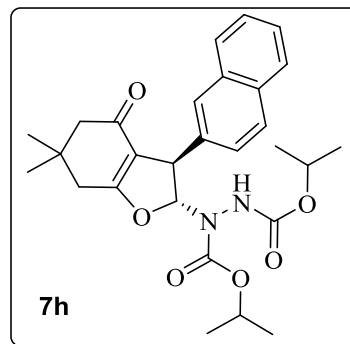


Fig S63 ¹H NMR Spectrum of 7h at +25 °C

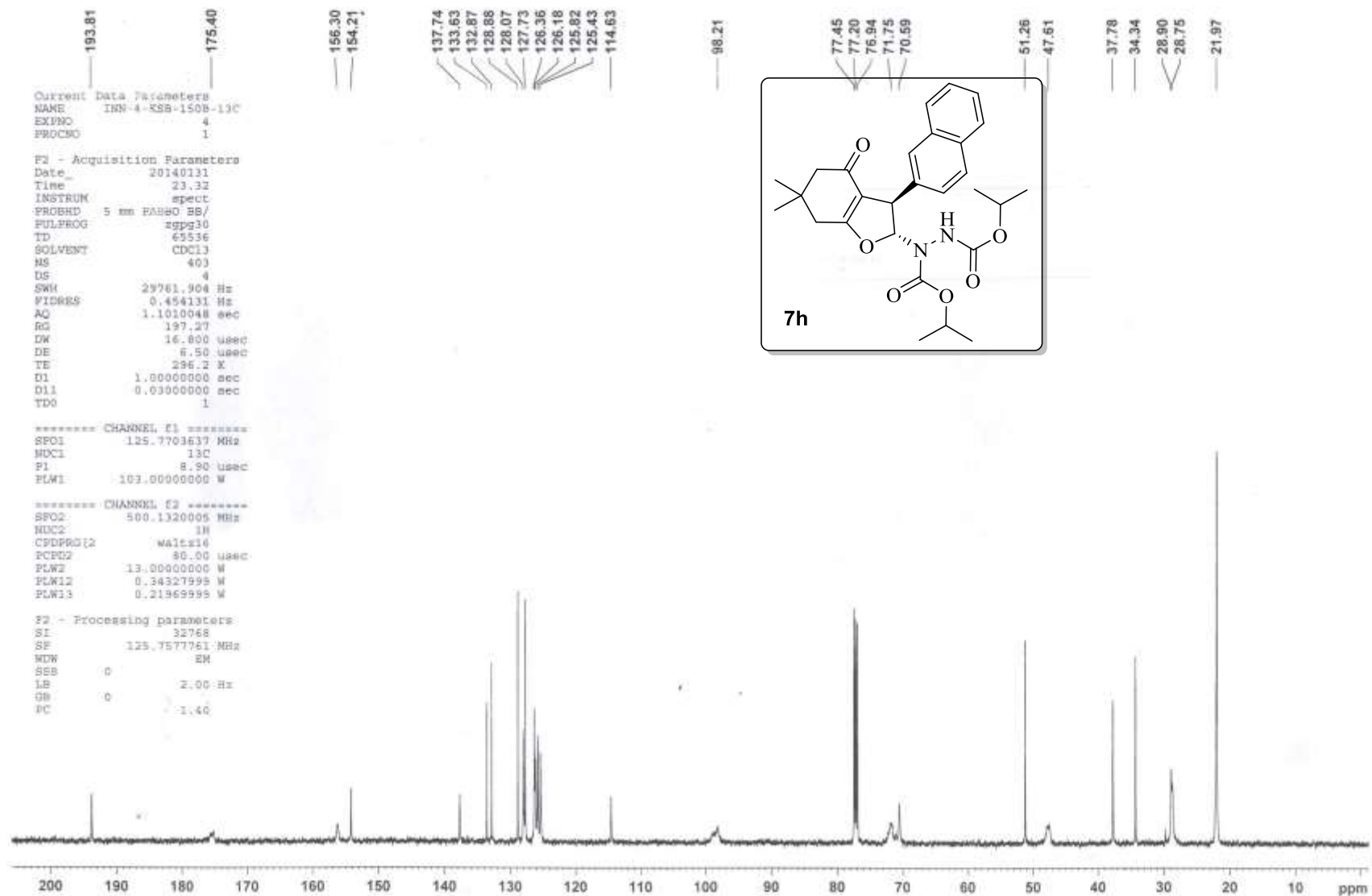
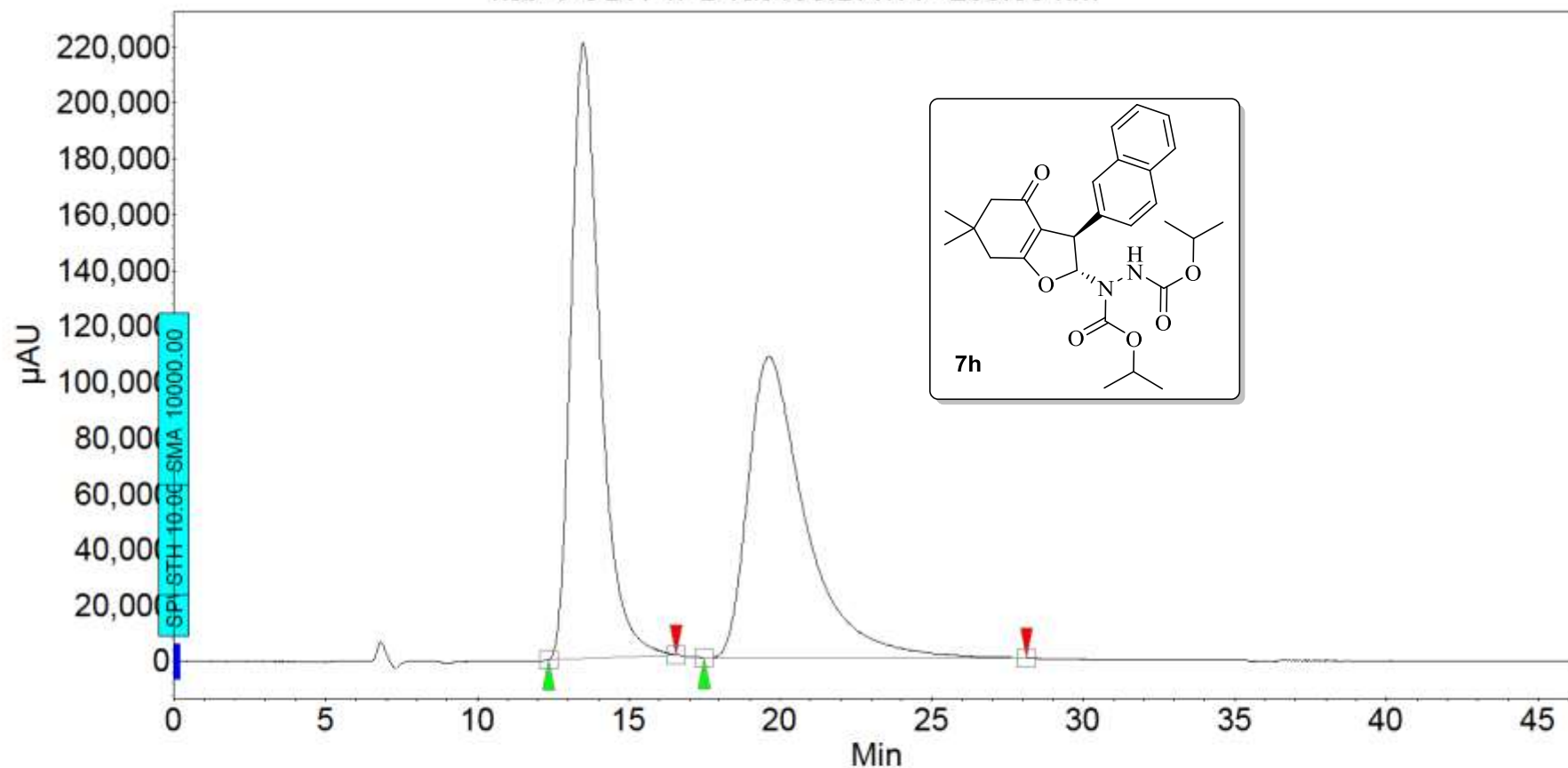


Fig S64. ¹³C NMR Spectrum of 7h at +25 °C

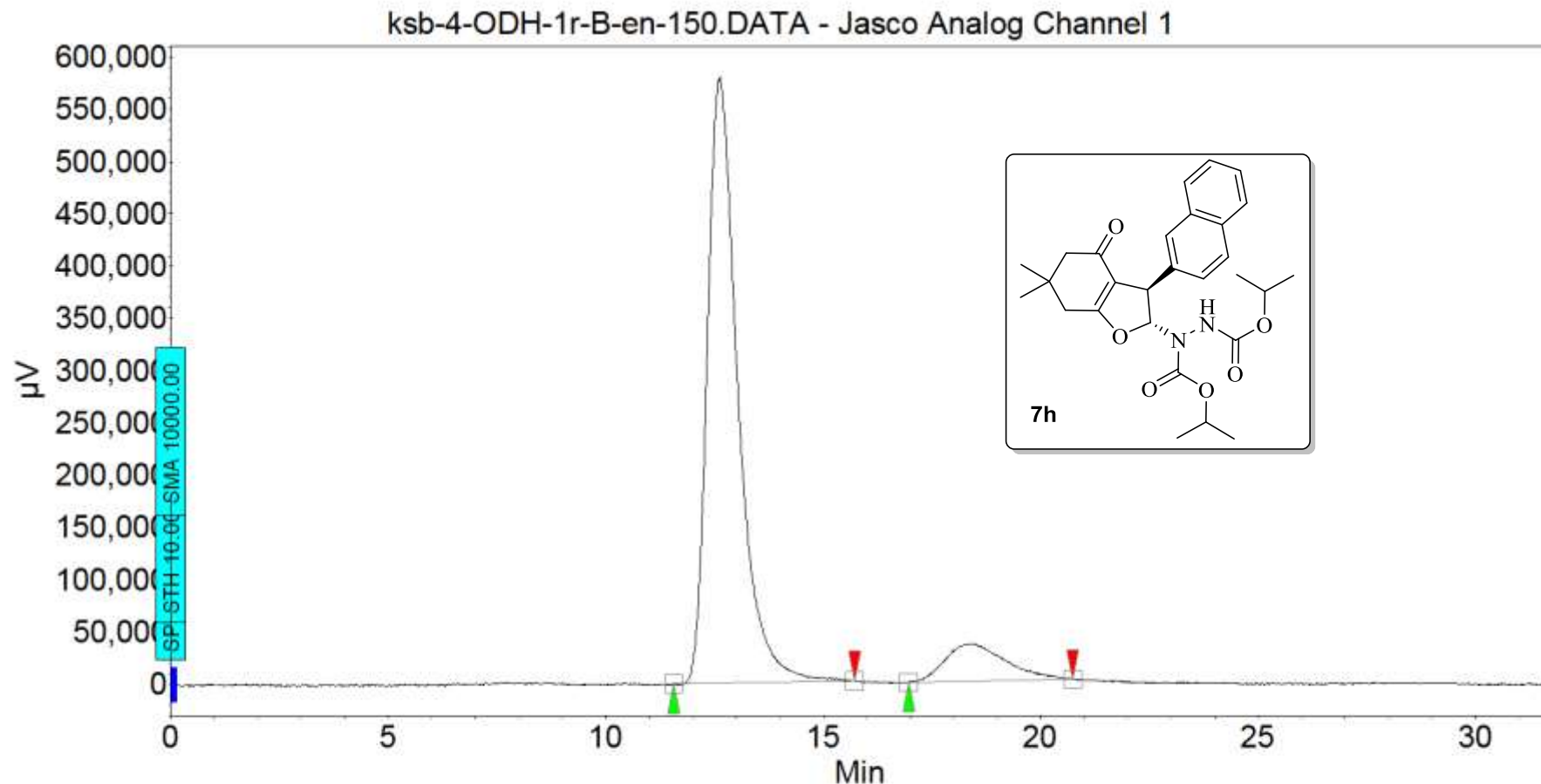
ksb-4-ODH-1r-B-rac150.DATA - 268.00 nm



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

INN-4-KSB-151C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Mar 19 2014	dn	H1
solvent	CDC13	dof	-268.4
file	exp	dm	nnn
ACQUISITION			
sfrq	299.950	dmm	c
tn	H1	dmf	200
at	2.000	temp	55.0
PROCESSING			
np	24022	lb	0.10
sw	6006.0	fn	not used
fb	not used		
bs	2	werr	
pw	3.0	wexp	
pw	3.0	wbs	
tpwr	60	wnt	
d1	0		
DISPLAY			
tof	900.0	sp	0.5
nt	1600	wp	3607.5
ct	140	vs	162
alock	n	sc	0
gain	2	wc	250
FLAGS			
il	n	hzmn	5.38
in	n	is	5054.60
dp	y	rfl	650.9
		rfp	0
		th	1
		ins	76.300
		nm	ph

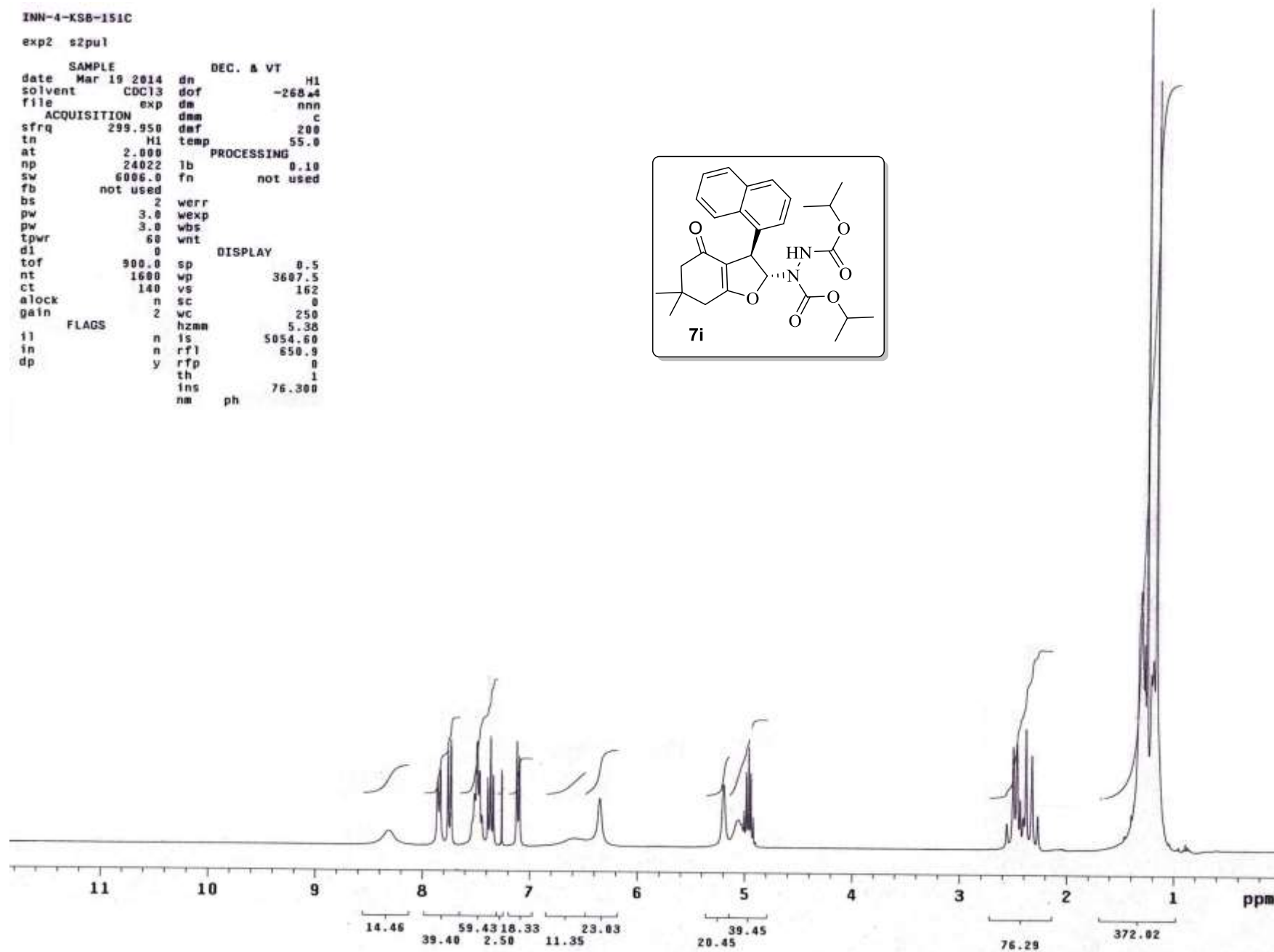
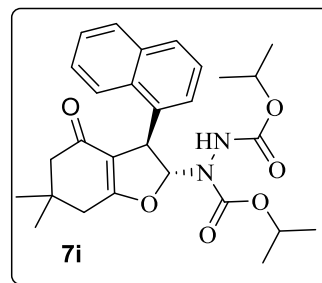


Fig S67. ¹H NMR Spectrum of 7i at +55 °C

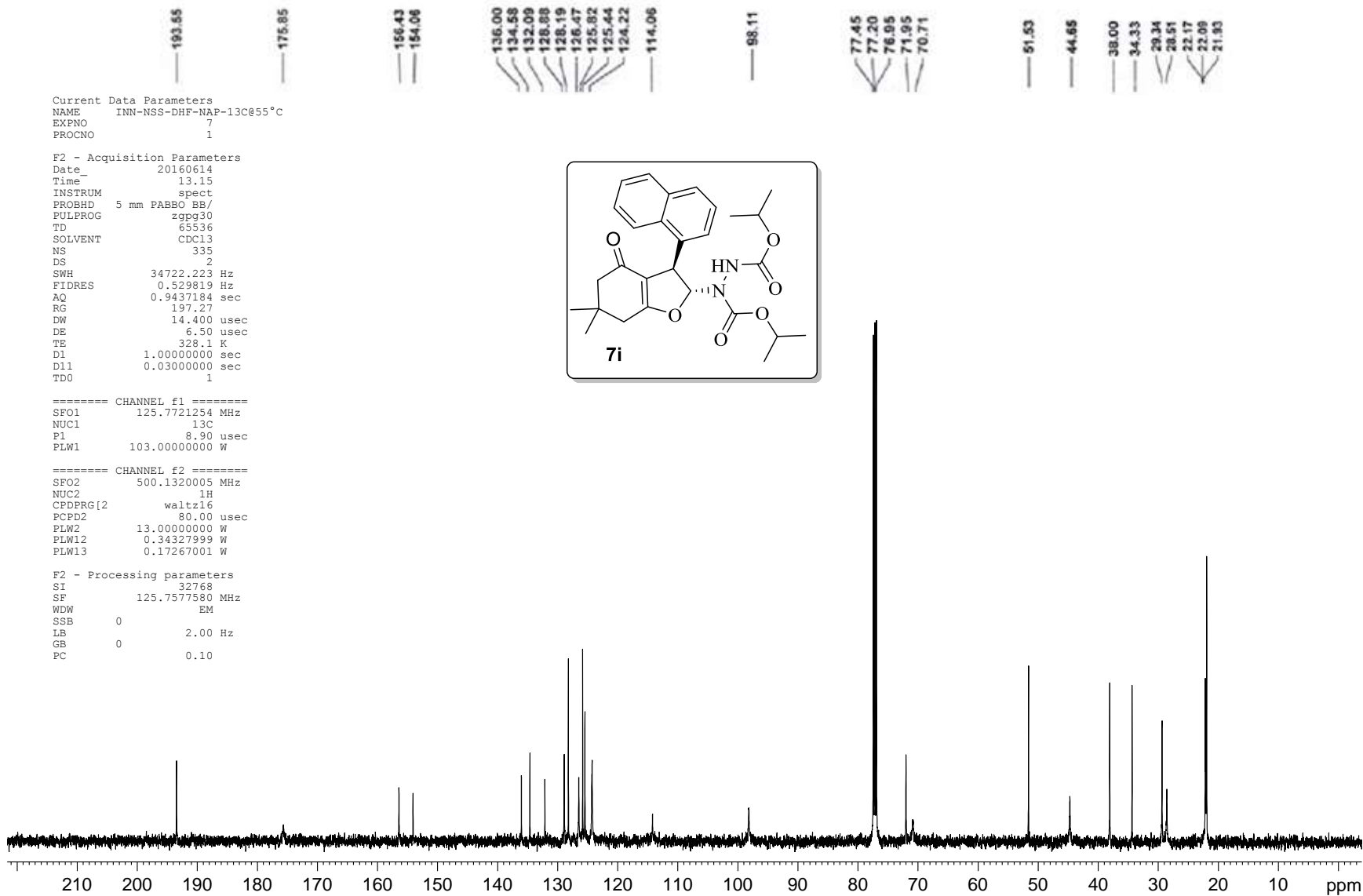
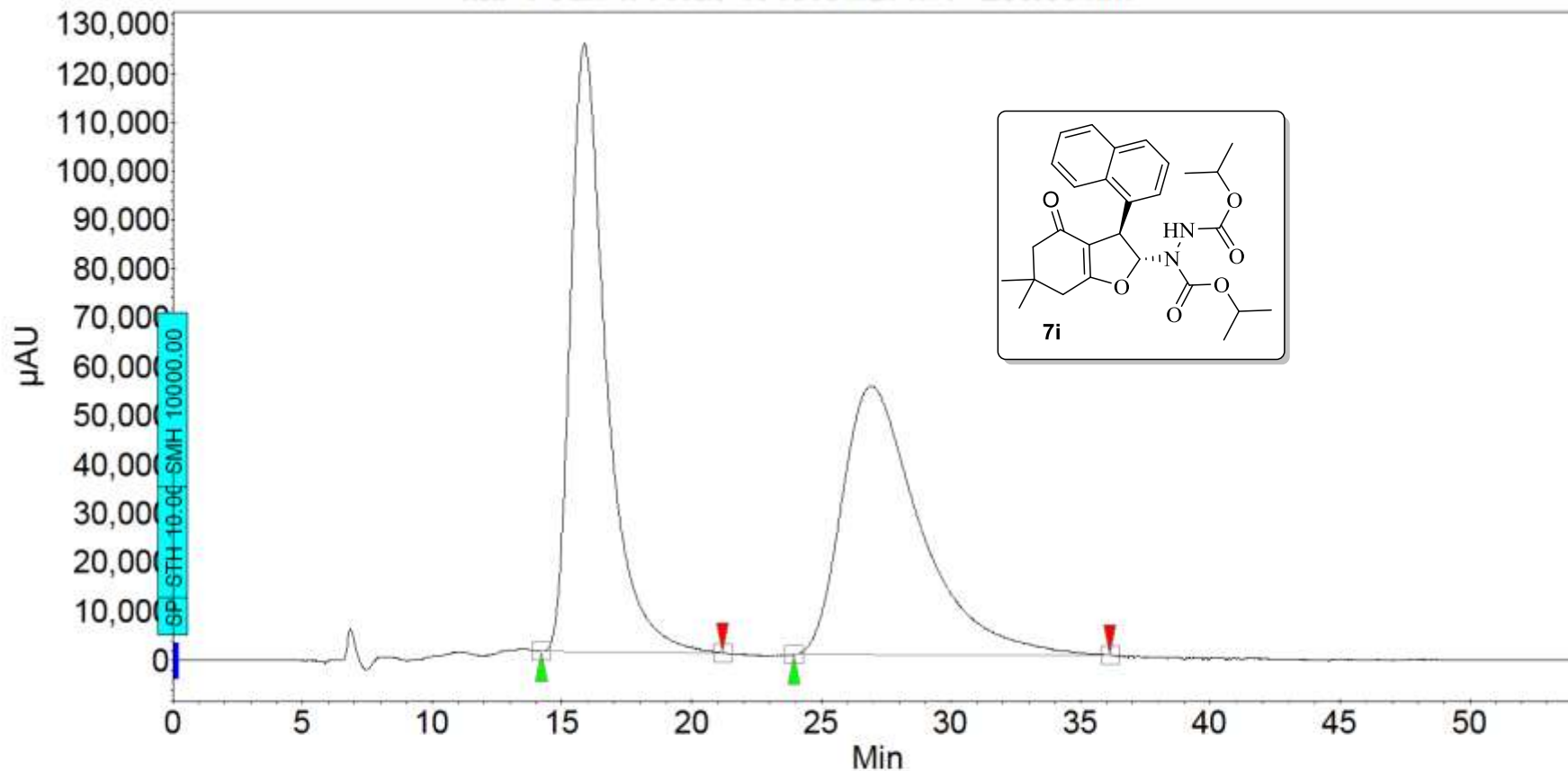


Fig S68. ¹³C NMR Spectrum of 7i at +55 °C

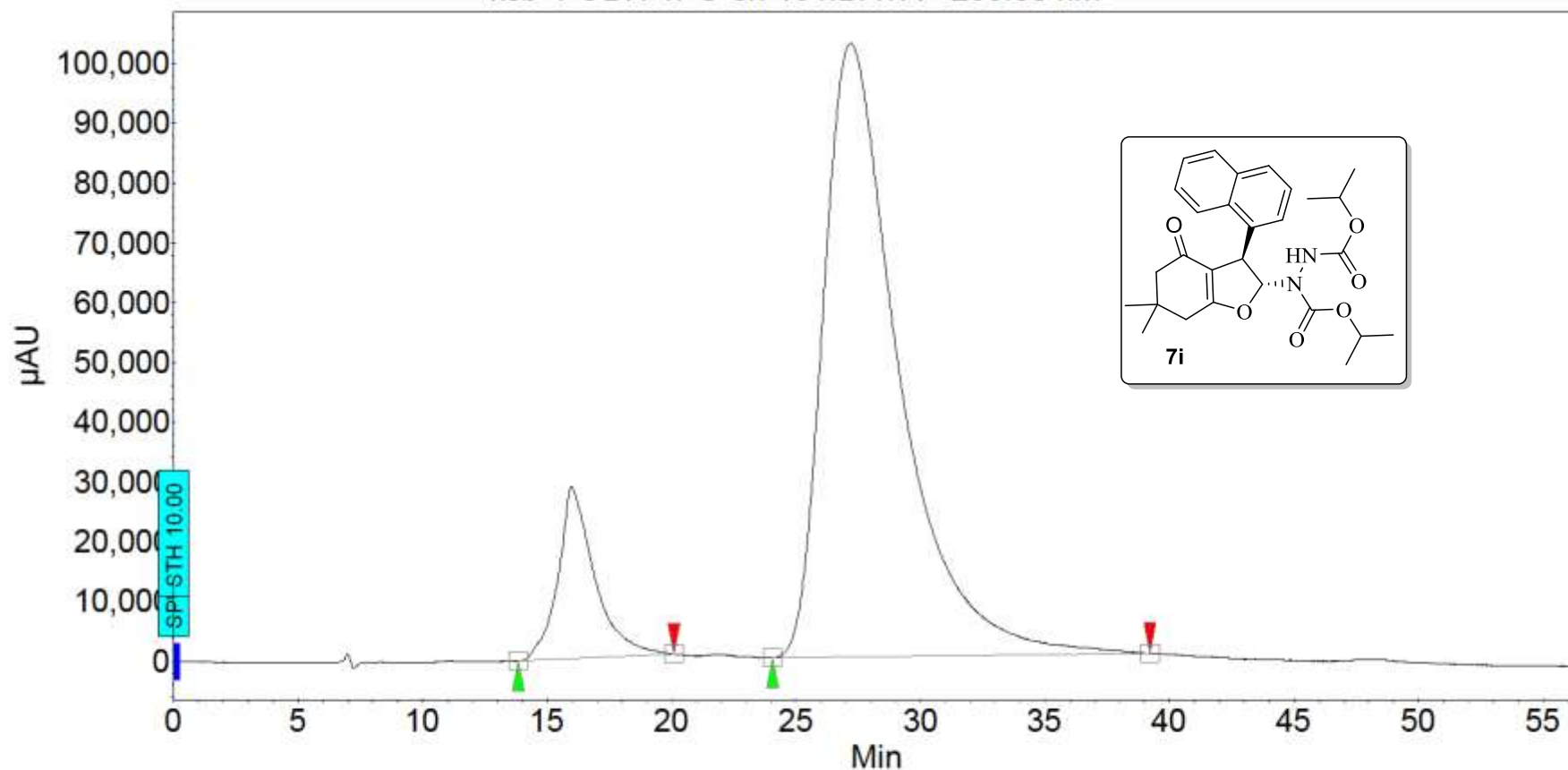
ksb-4-odh-1r-A-rac-151c151.DATA - 268.00 nm



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

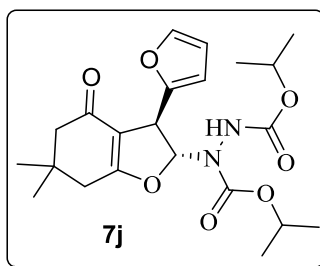


Fig S71. ¹H NMR Spectrum of 7j

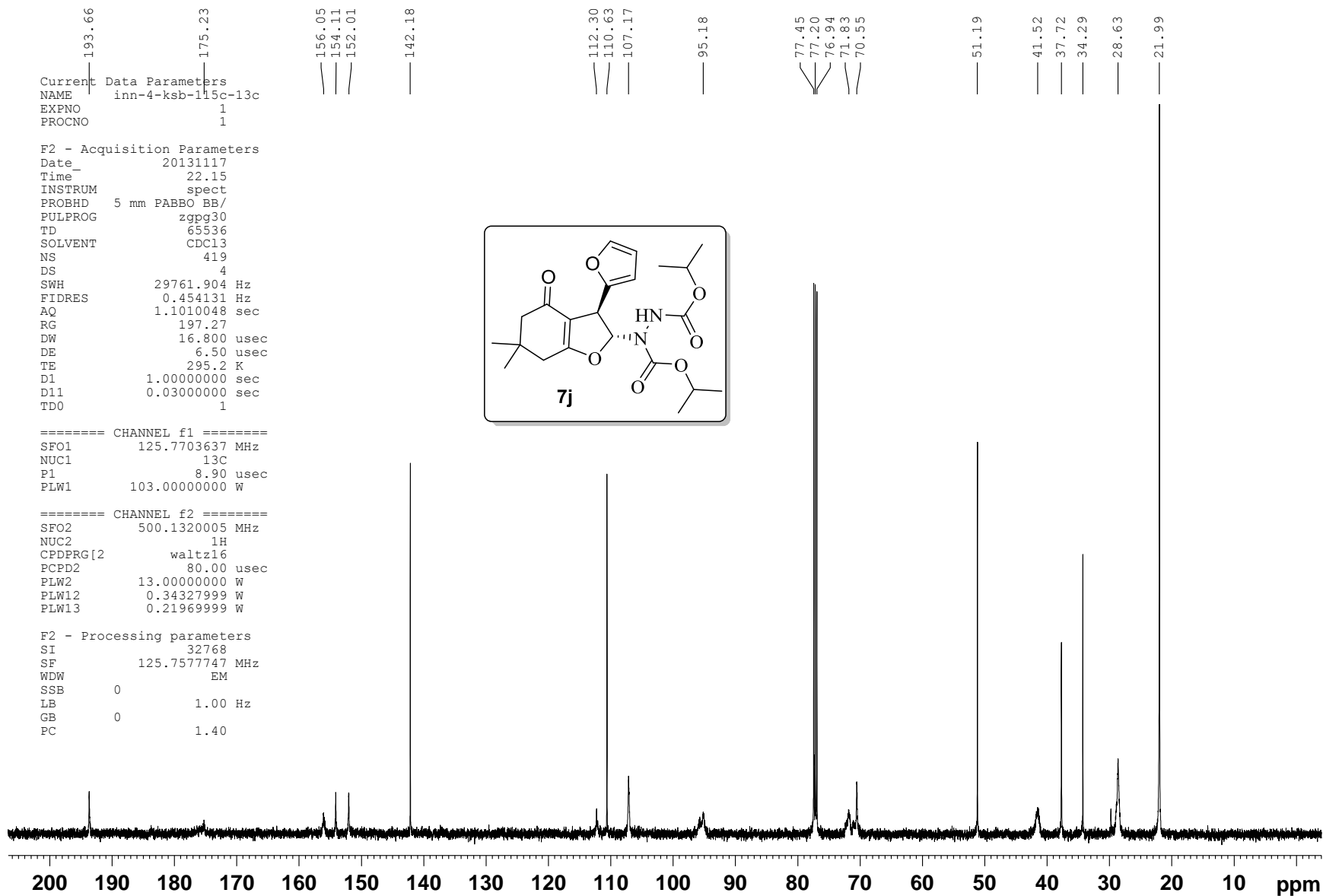
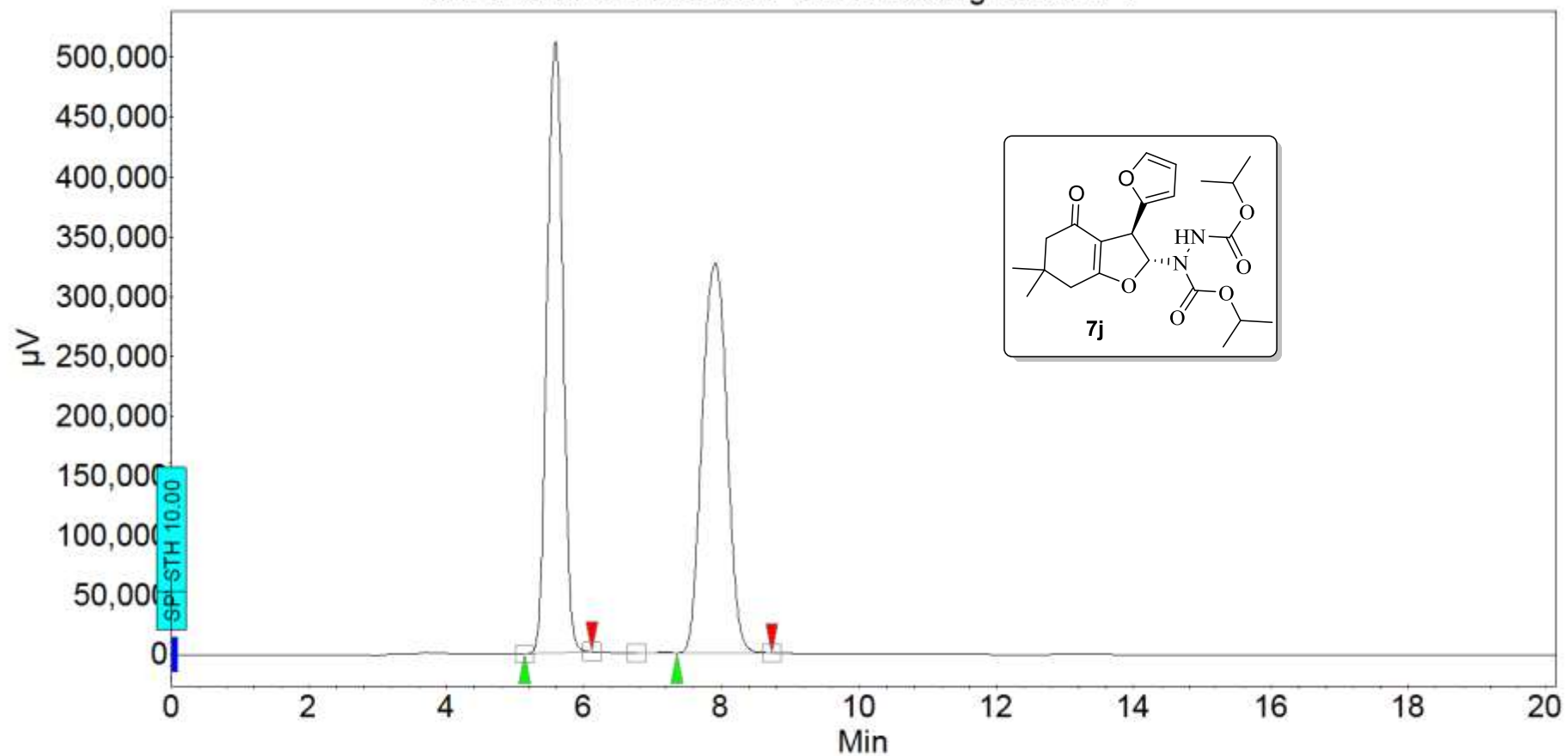


Fig S72. ¹³C NMR Spectrum of 7j

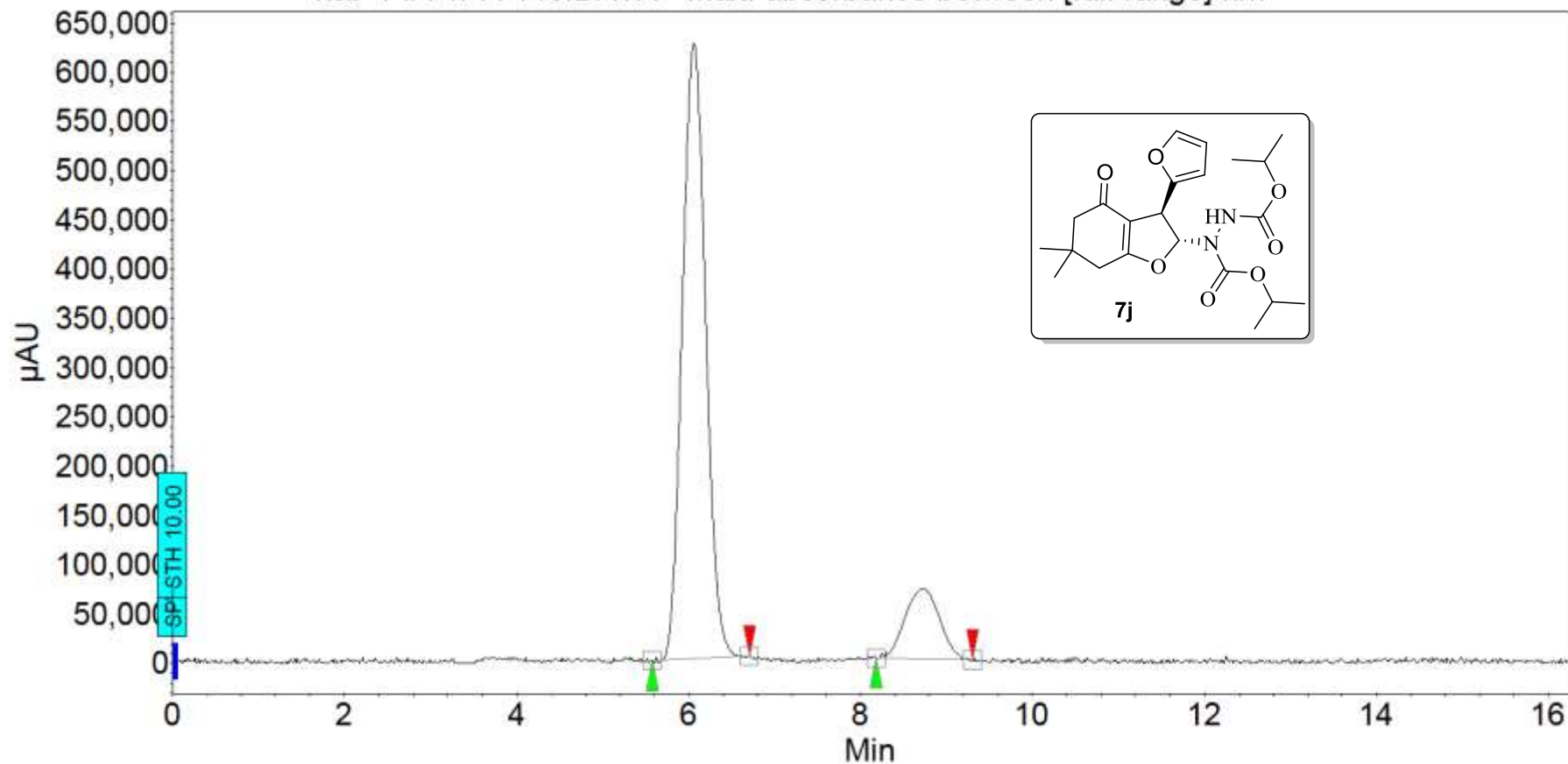


Peak results :

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

ksb-4-IA-1r-A-146.DATA - Max. absorbance between [full range] nm



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
TDO 1

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

F2 - Processing parameters
SI 65536
SF 500.1330000 MHz
WDW RM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

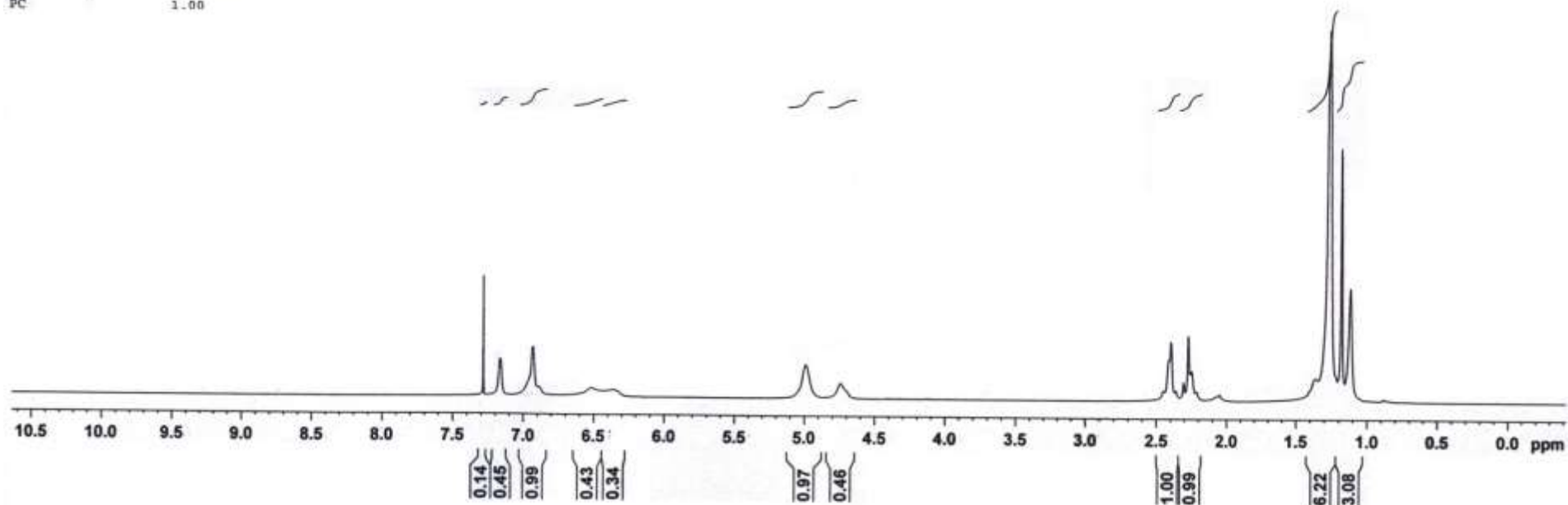
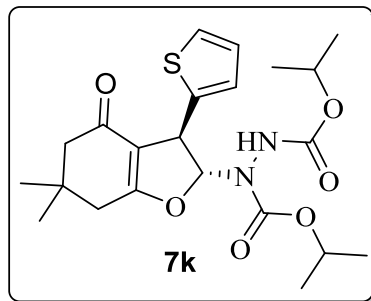


Fig S75. ¹H NMR Spectrum of 7k

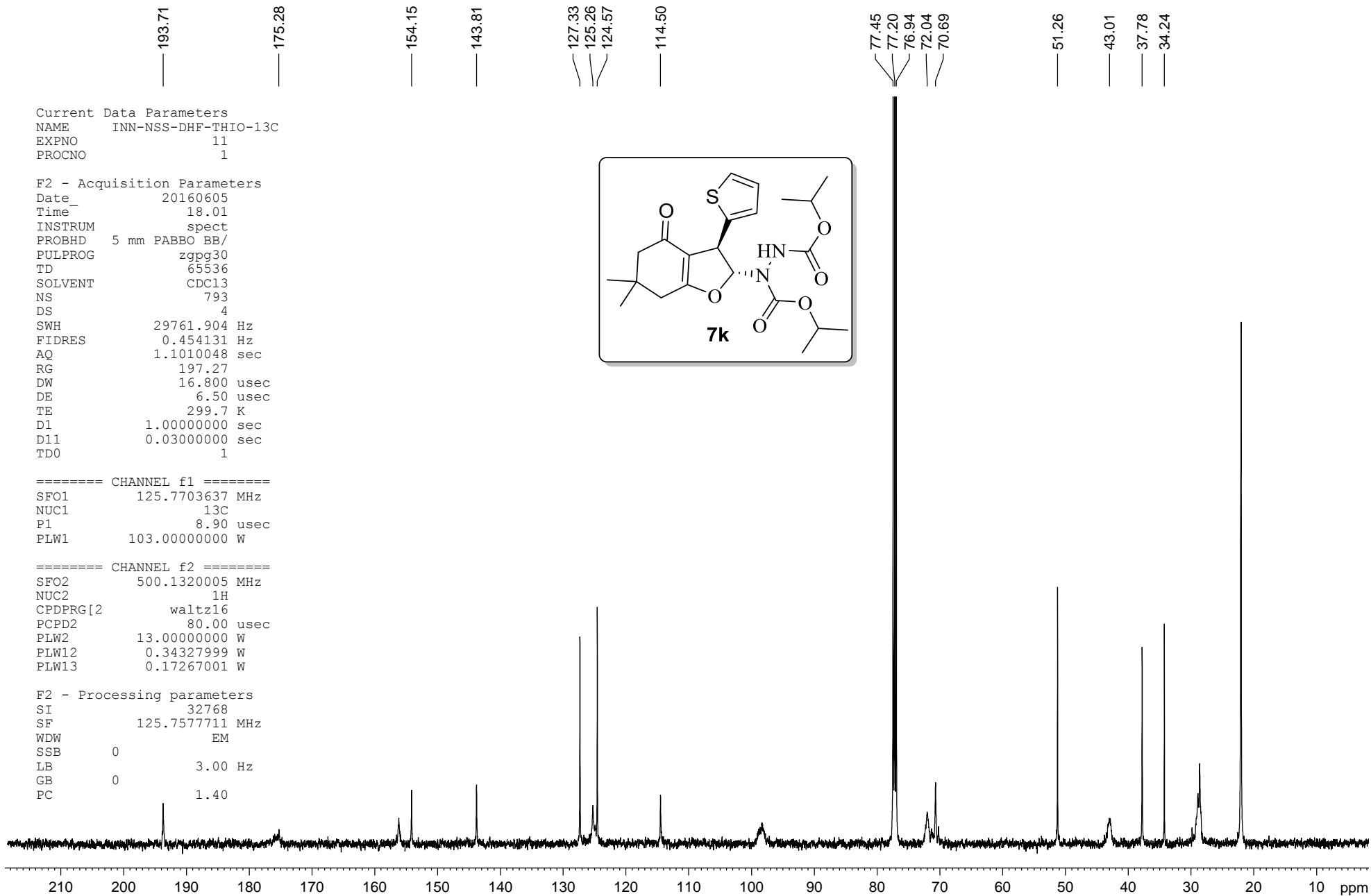
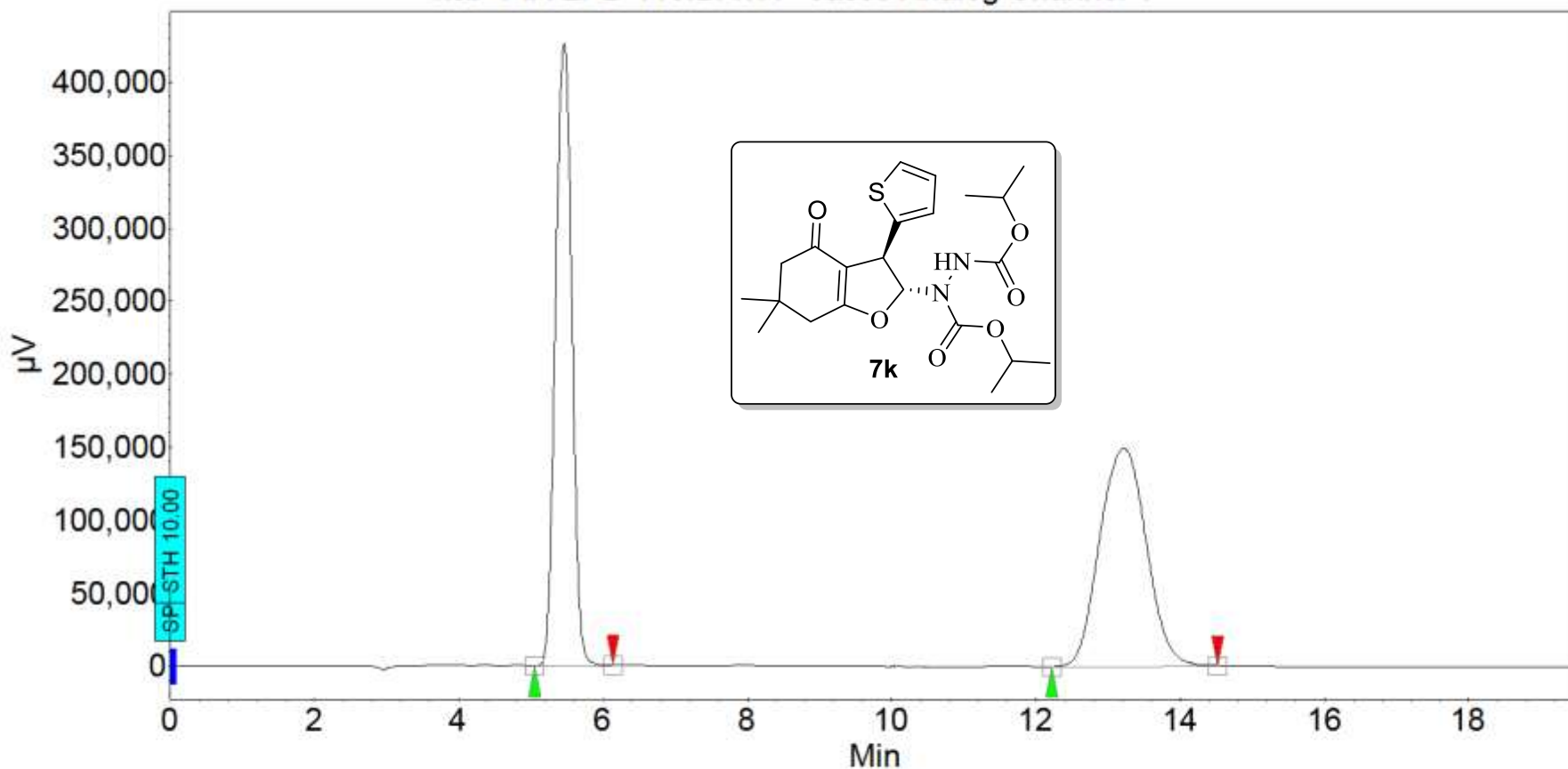


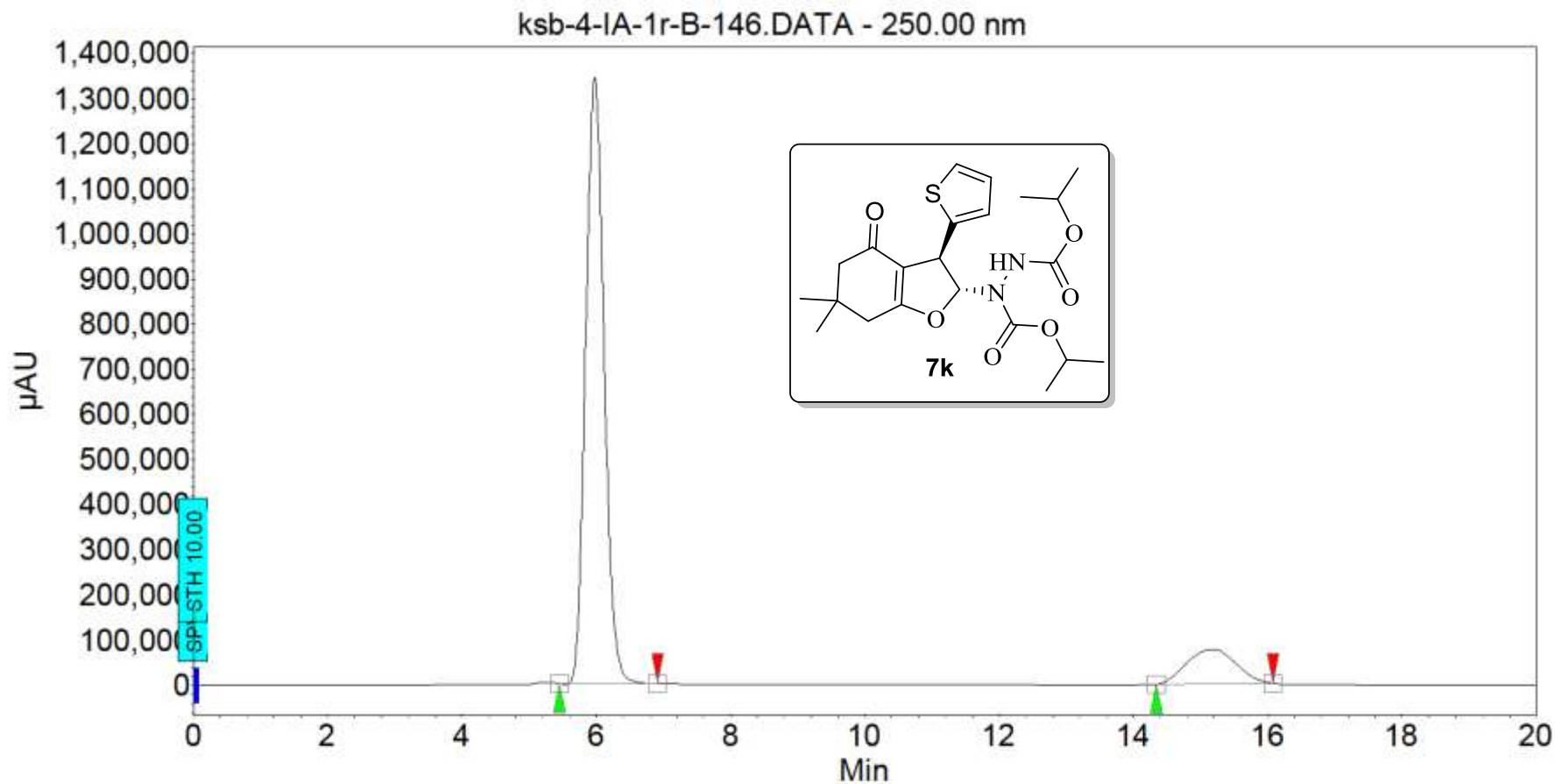
Fig S76. ¹³C NMR Spectrum of 7k



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

Current Data Parameters
NAME INN-4-18-114A-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121118
Time 12.05
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 30.72
DW 50.000 usec
DE 6.50 usec
TE 295.9 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 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

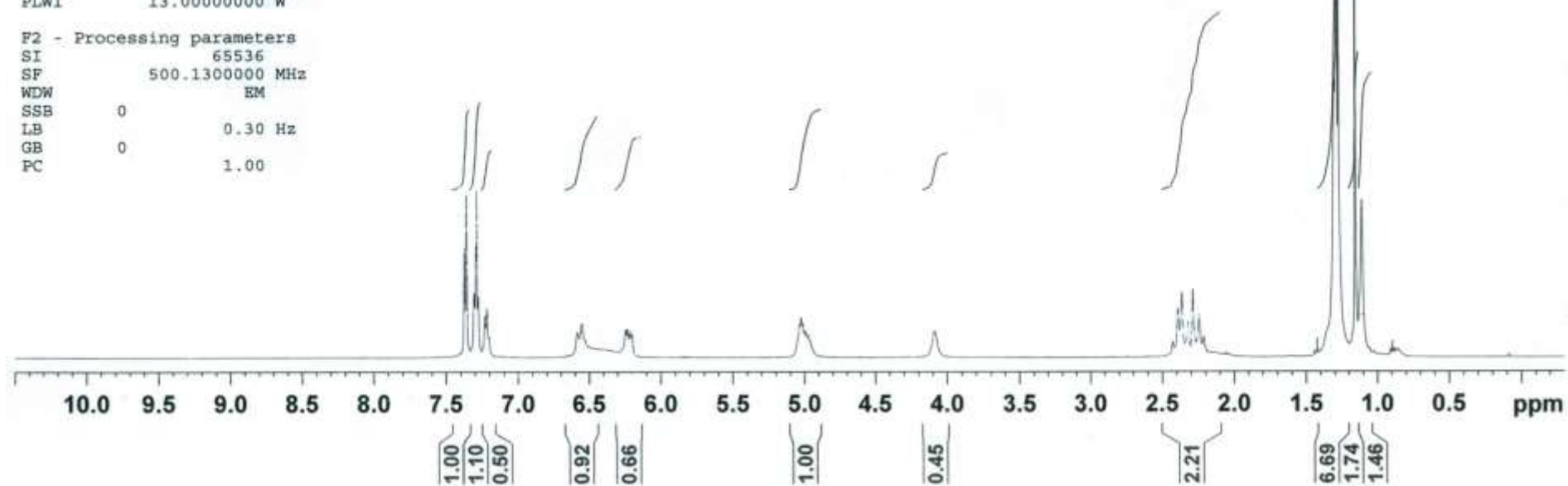
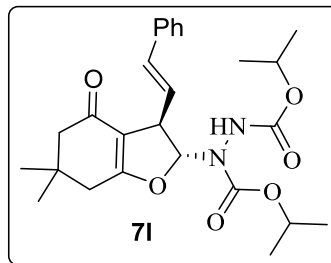


Fig S79. ¹H NMR Spectrum of 71

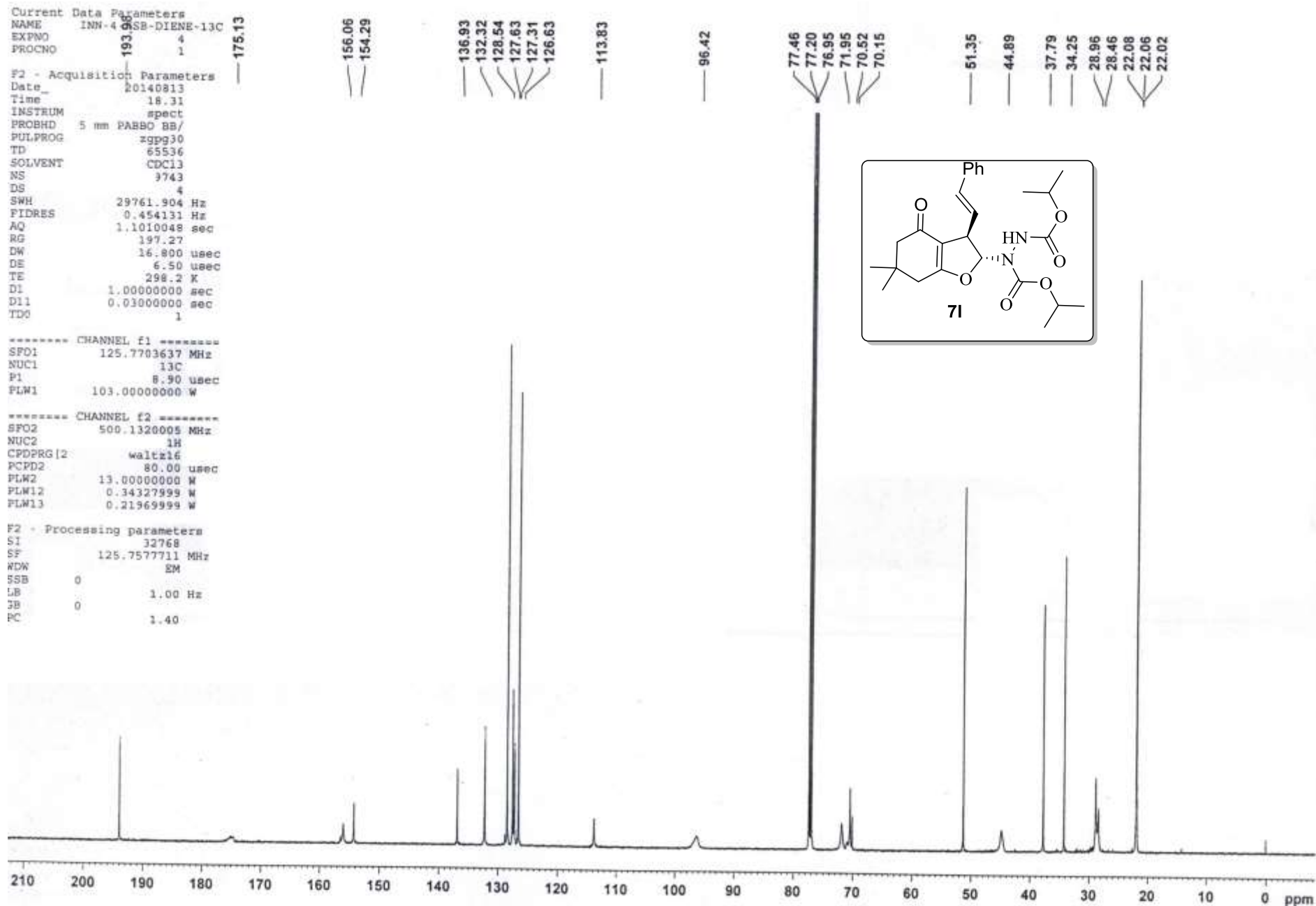


Fig S80. ^{13}C NMR Spectrum of 71

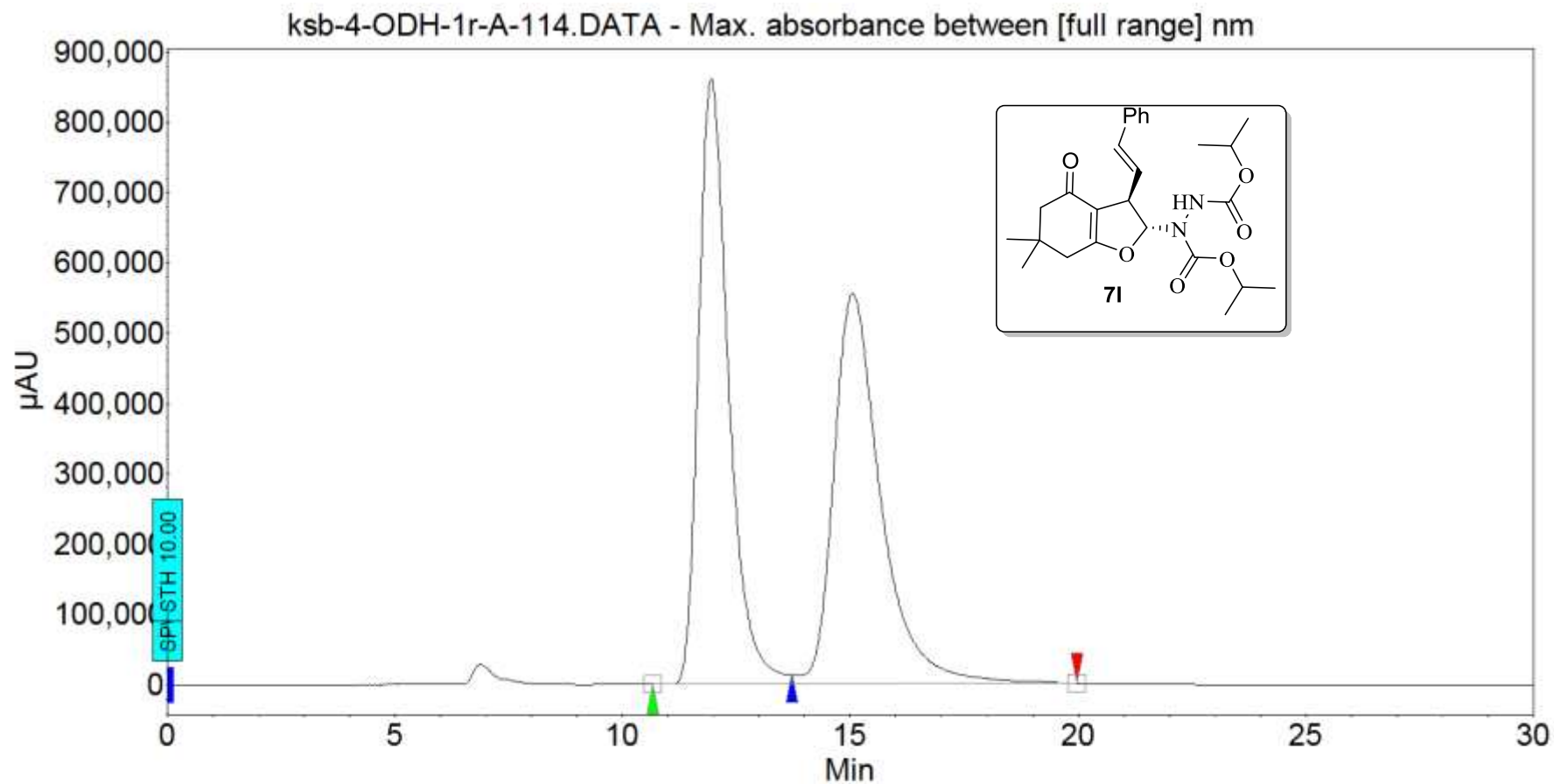
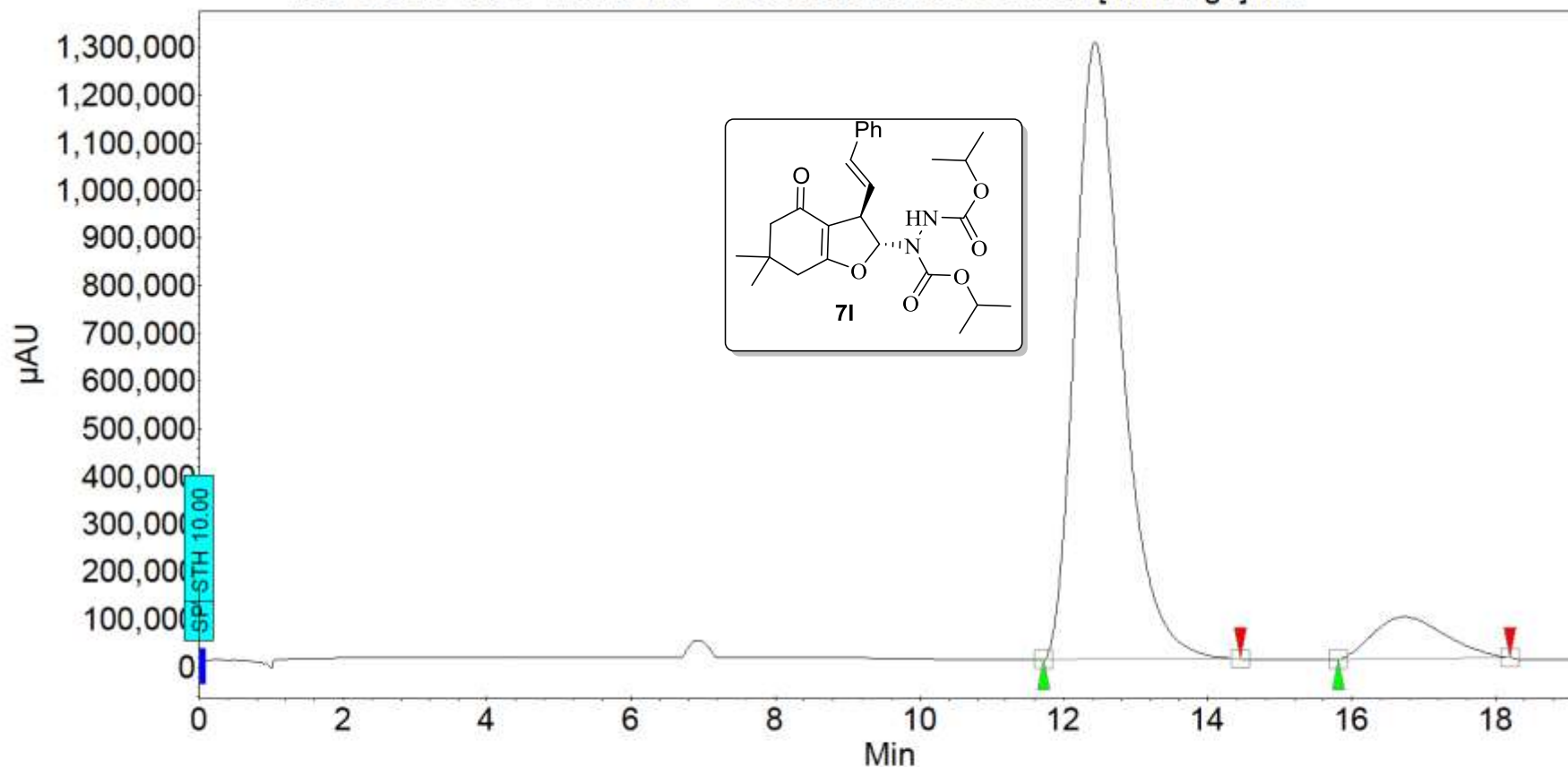


Fig S81. HPLC Profile of Racemic 71

ksb-4-ODH-1R-C-145.DATA - Max. absorbance between [full range] nm



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 71

Current Data Parameters
NAME INN-4-KSB-154C-1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140310
Time 21.15
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 30.72
DW 50.000 usec
DE 6.50 usec
TE 296.6 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 500.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

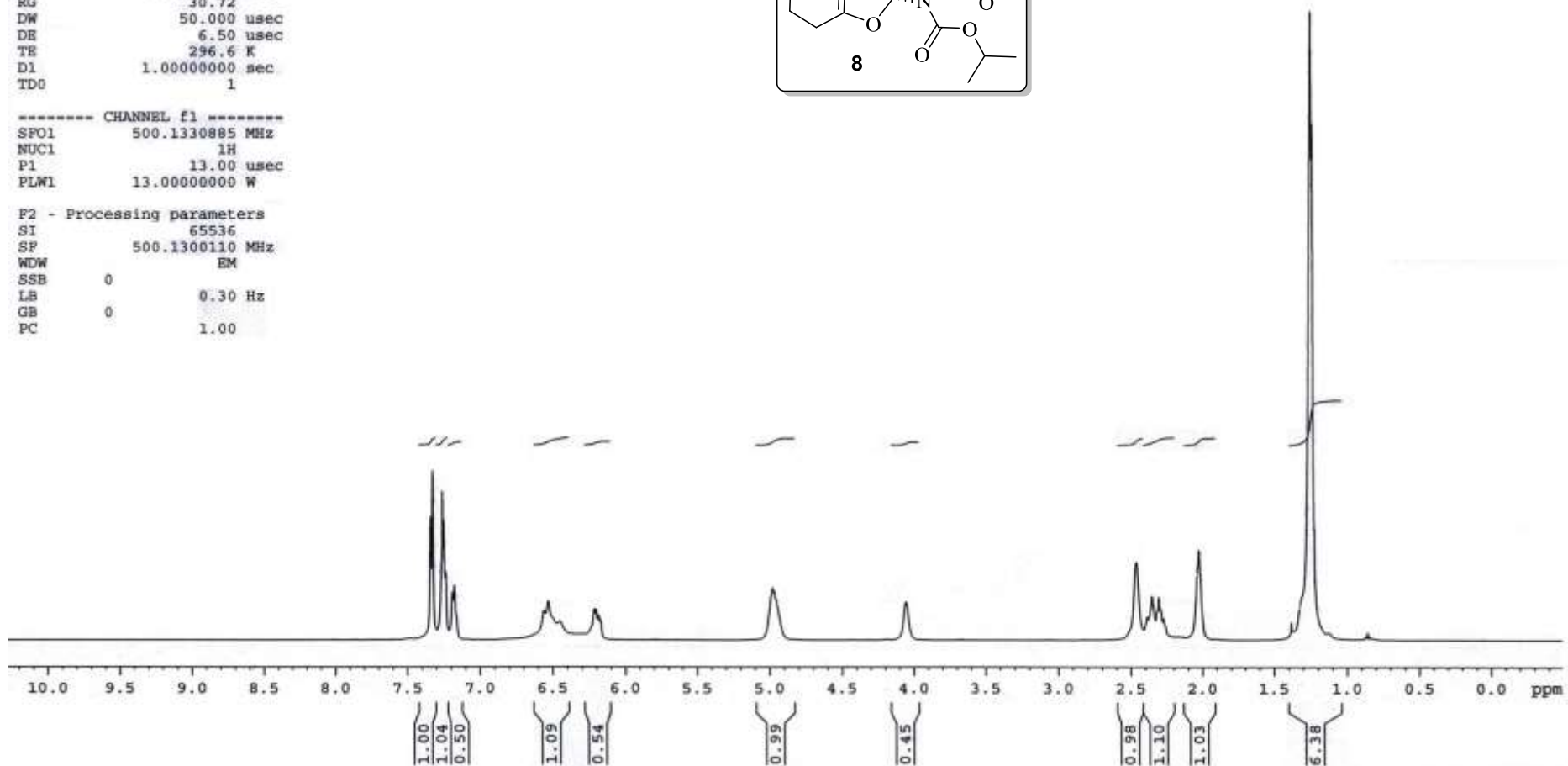
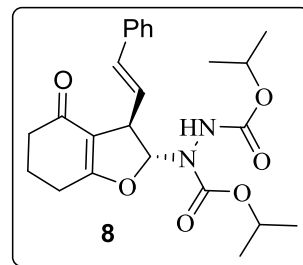


Fig S83. ¹H NMR Spectrum of 8

Current Data Parameters
NAME INN-4-KSB-154C-13C
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140312
Time 22.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 439
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 197.27
DW 16.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
D11 0.03000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 125.7703637 MHz
NUC1 13C
P1 8.90 usec
PLW1 103.00000000 W

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

F2 - Processing parameters
SI 32768
SF 125.7577803 MHz
WDW EM
SSB 0
LB 3.00 Hz
3B 0
PC 1.40

INN-4-KSB-154C-1

156.04 154.28 136.84 132.19 128.47 127.55 127.28 126.56 115.05 96.00 77.46 77.20 76.95 71.77 70.44 44.54 36.86 23.90 22.00 21.95 21.64

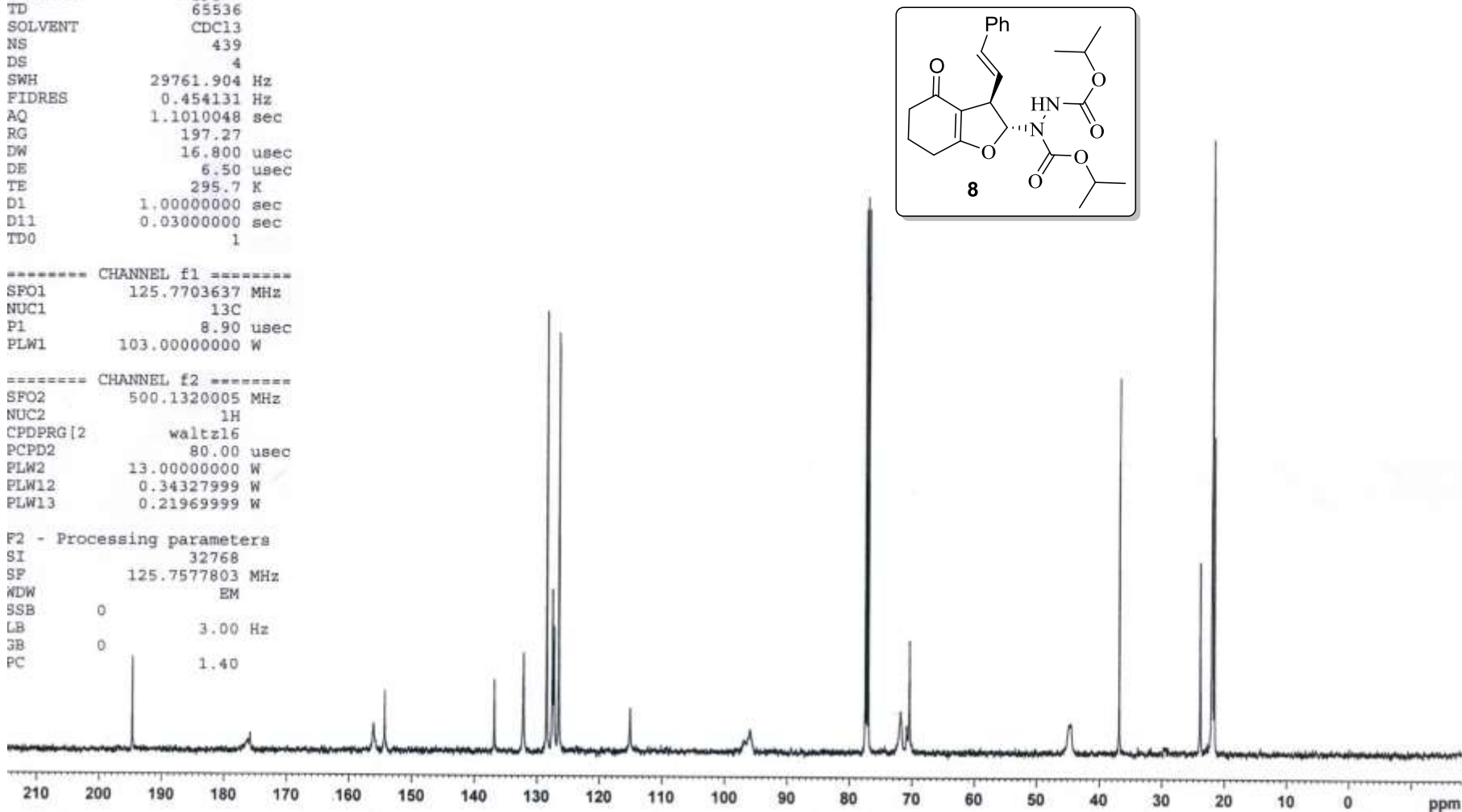
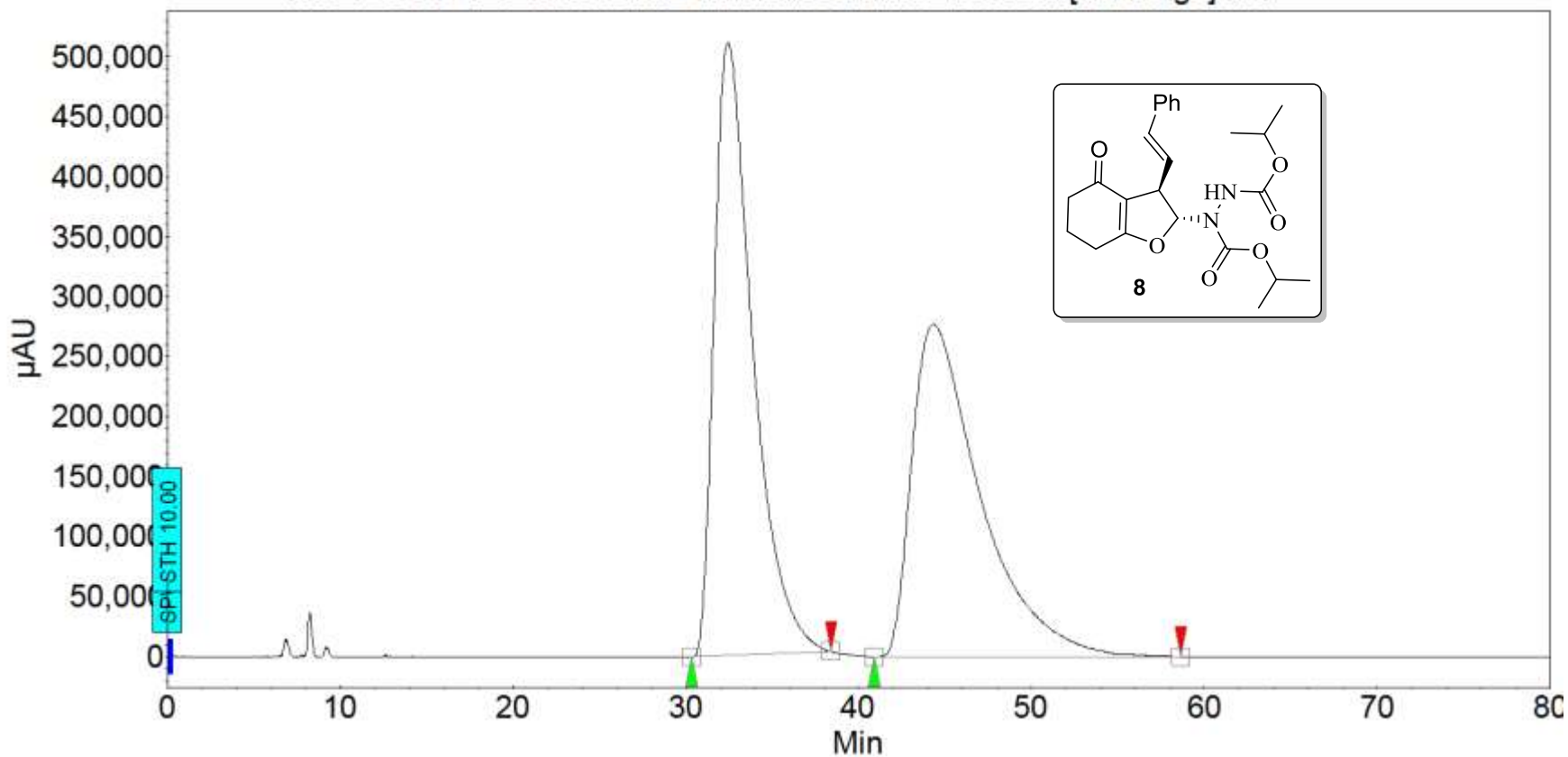


Fig S84. ¹³C NMR Spectrum of 8

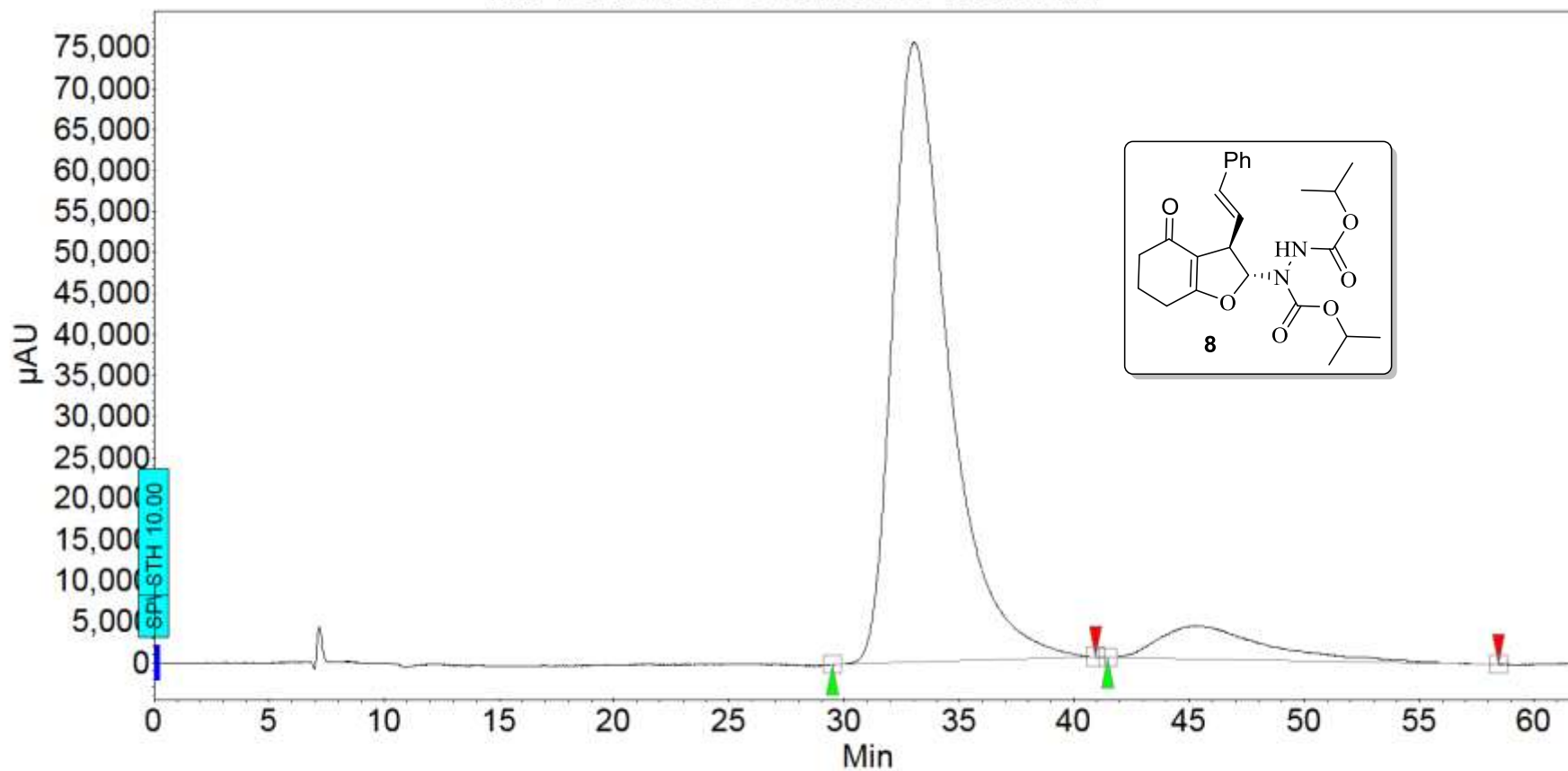
ksb-4-ODH-2r-C-154.DATA - Max. absorbance between [full range] nm



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

Current Data Parameters
NAME INN-NSS-DHQ-DBAB-1H
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191031
Time_ 15.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
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 323.0 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 500.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

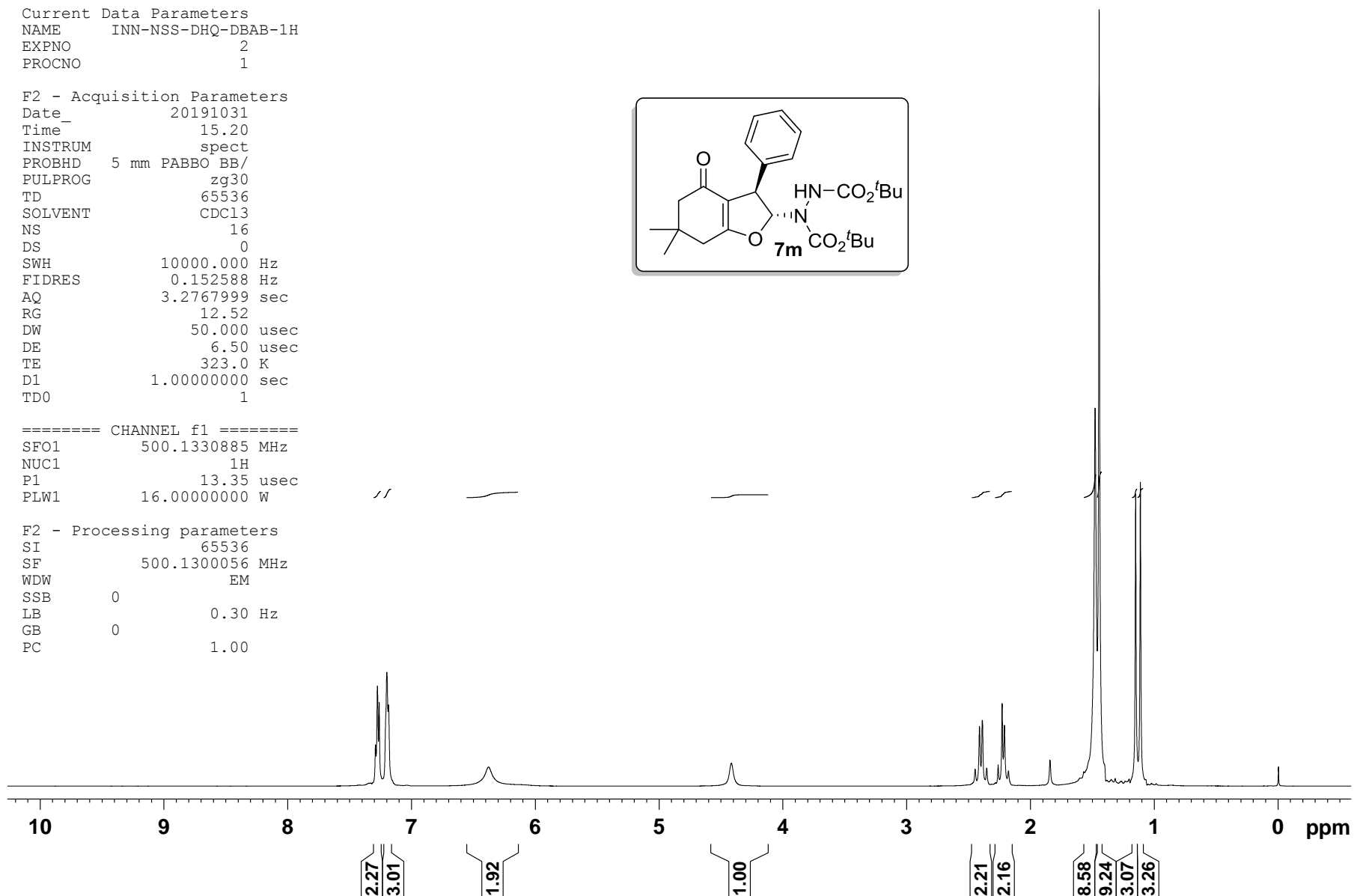
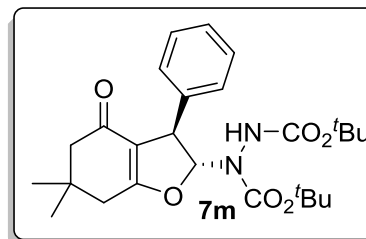


Fig S87. ¹H NMR Spectrum of 7m

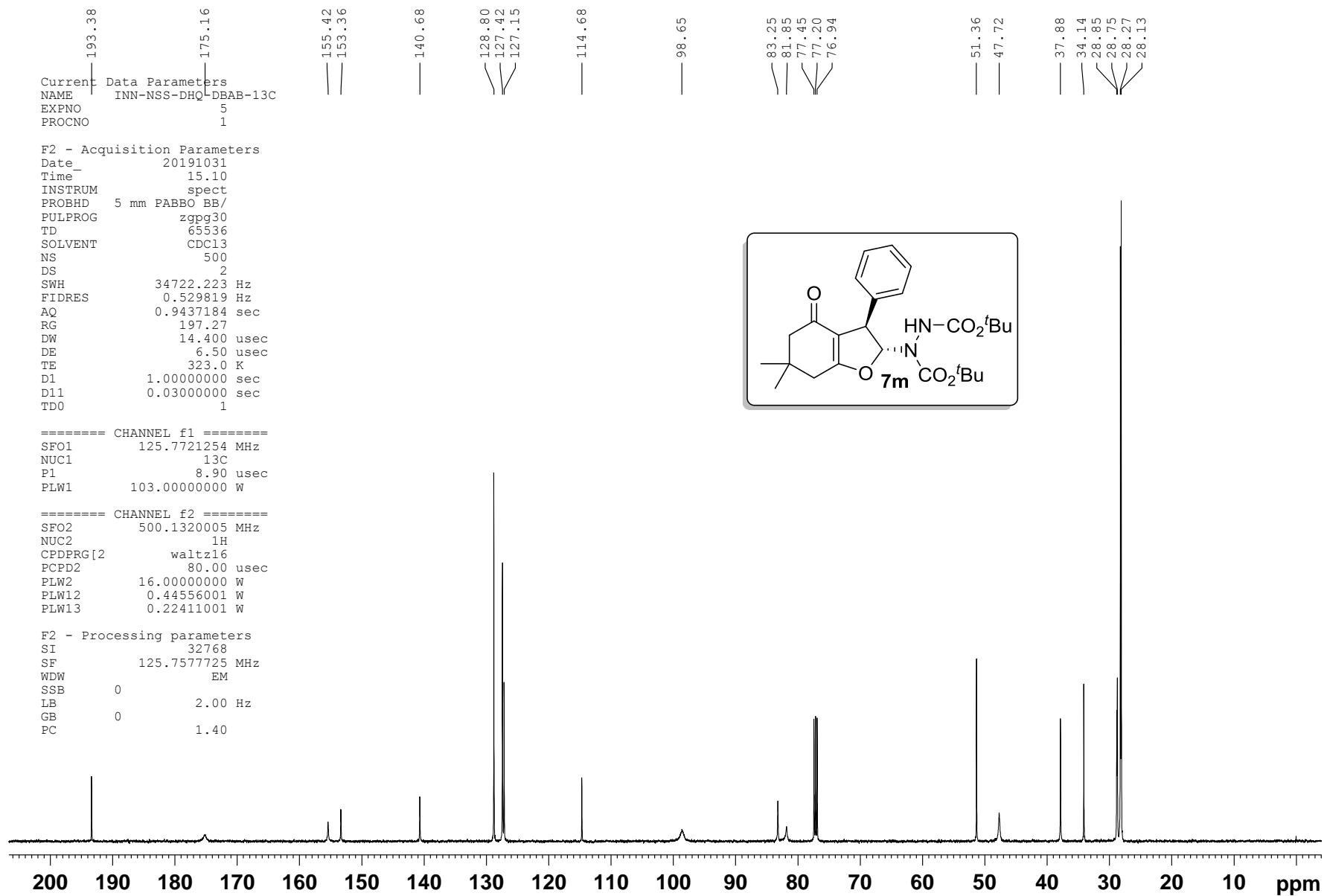
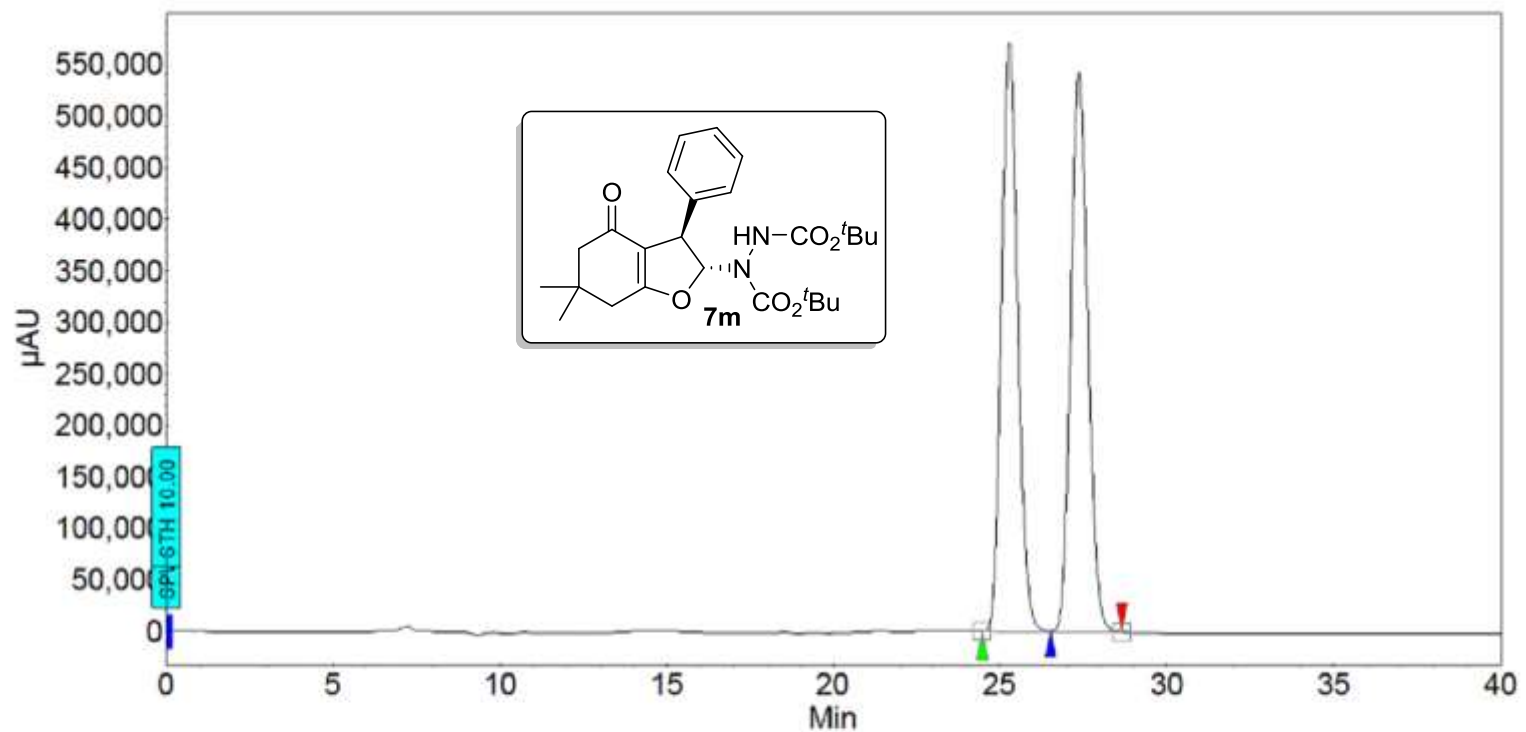


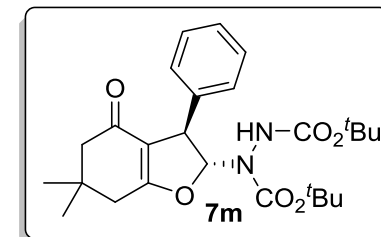
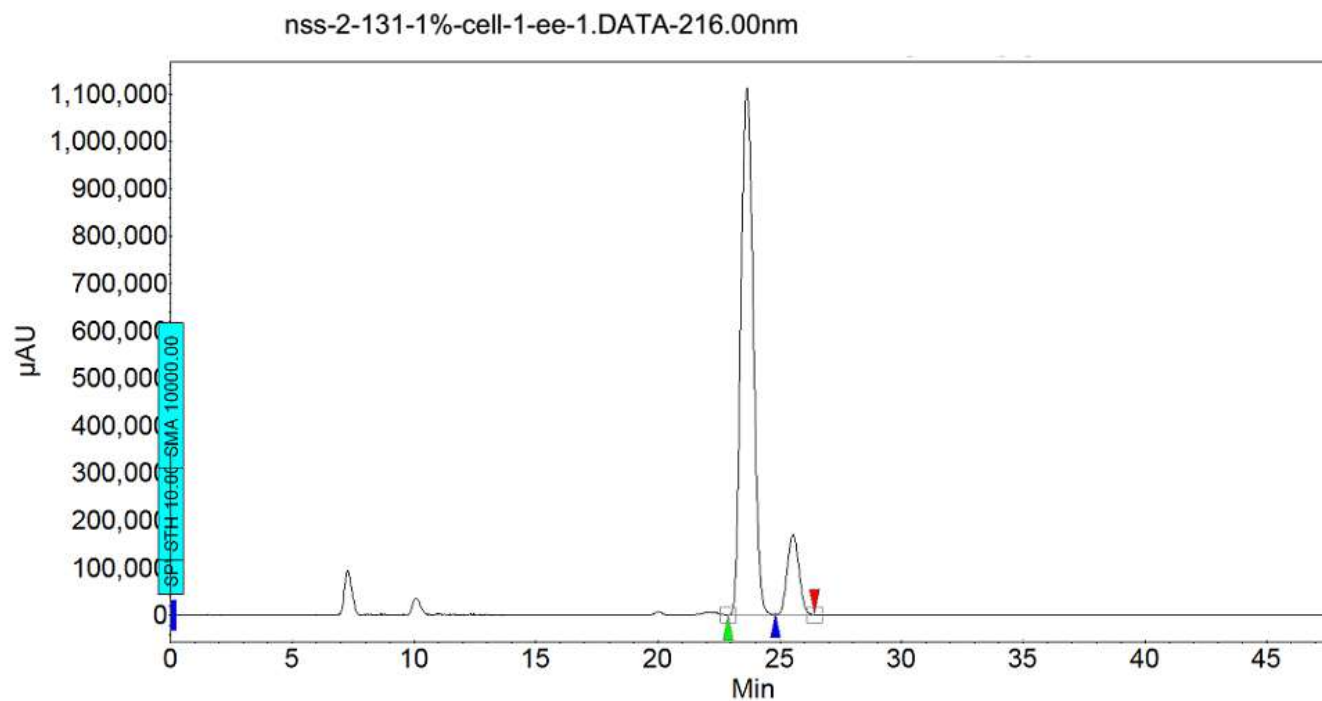
Fig S88. ¹³C NMR Spectrum of 7m



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

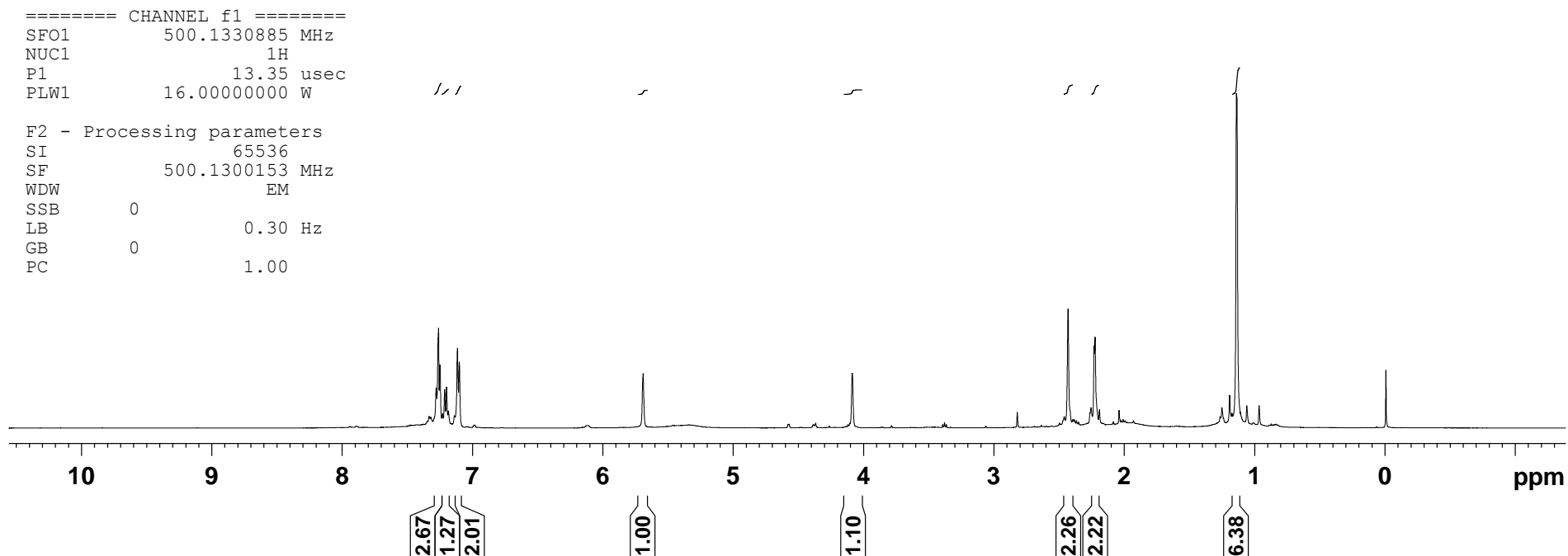
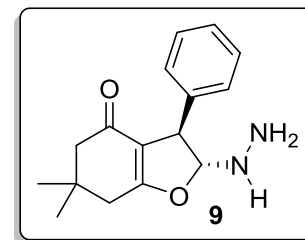


Fig S91. ¹H NMR Spectrum of 9

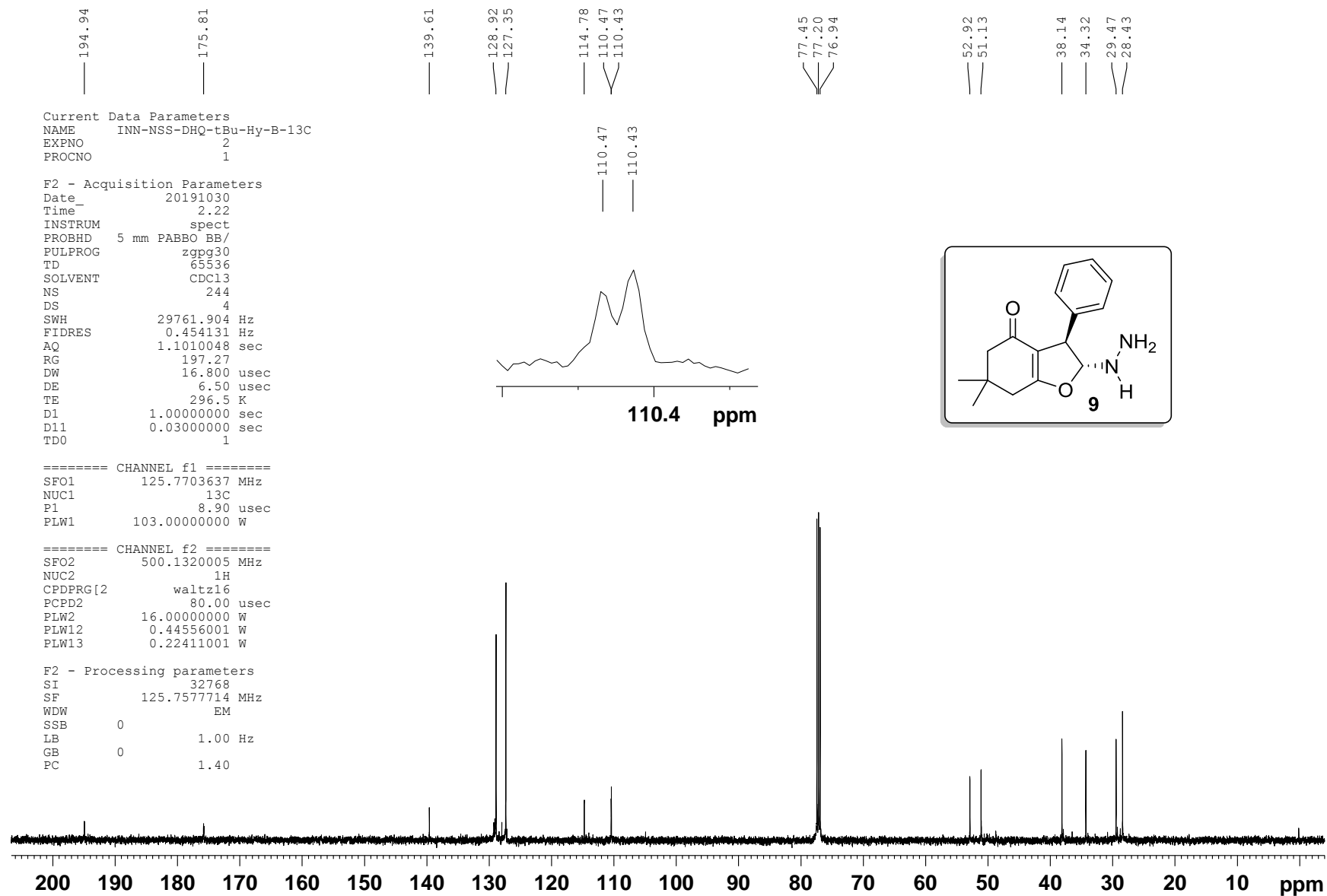
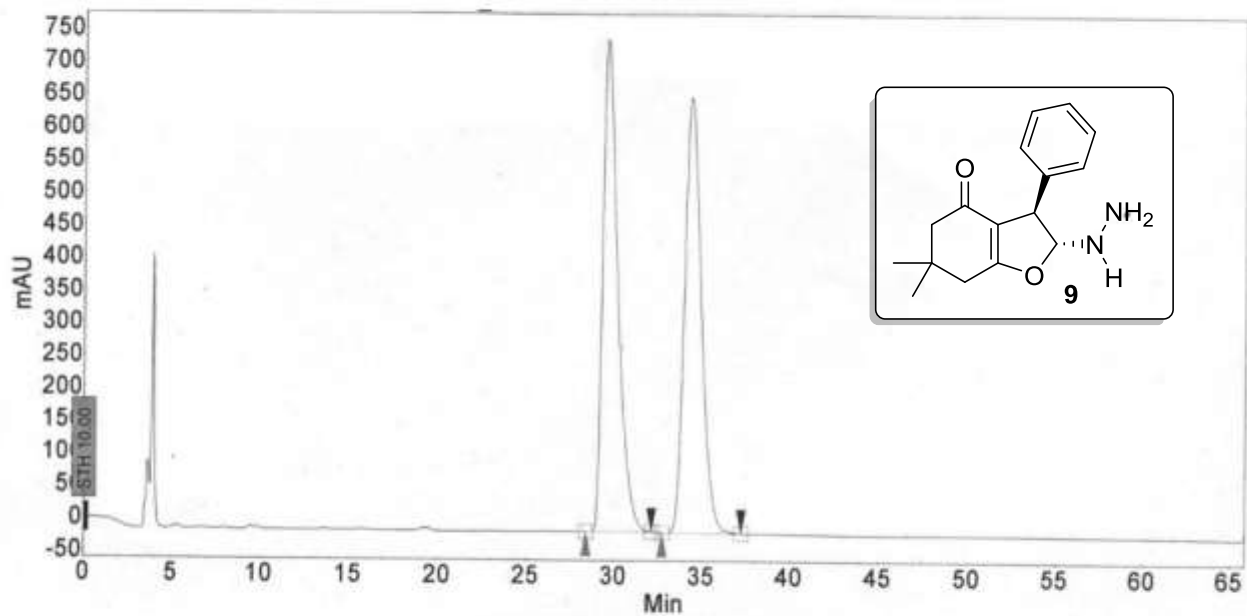


Fig S92. ¹³C NMR Spectrum of 9

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

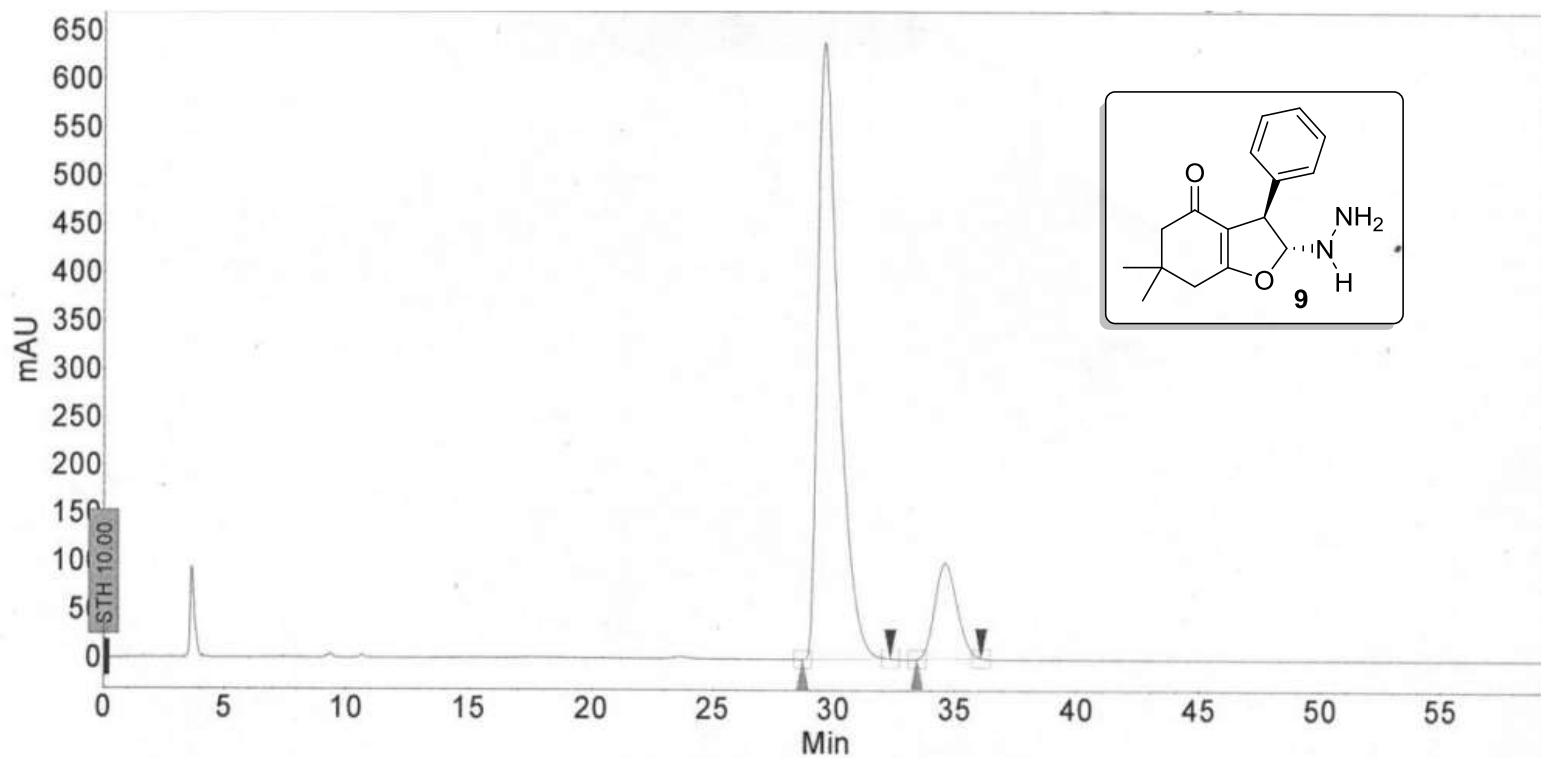
Acquired : 11/4/2019 6:58:21 PM
Processed : 11/4/2019 7:58:21 PM
Printed : 11/8/2019 4:58:21 PM



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

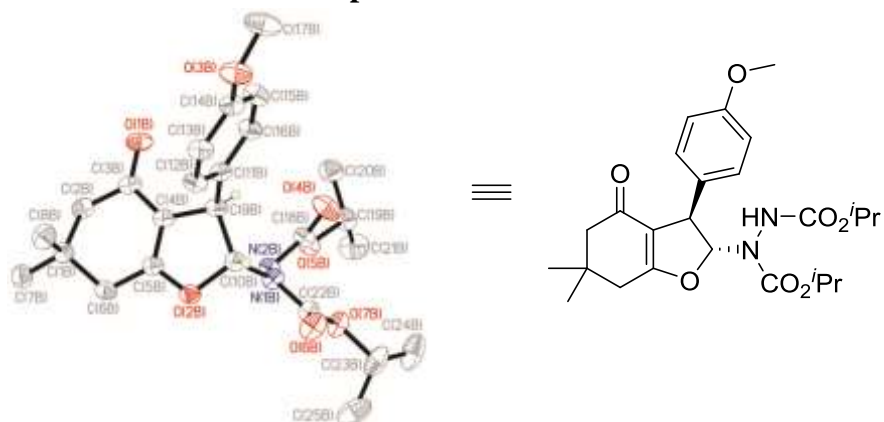


Peak results :

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 ₂ O ₇ ·C ₂₅ H ₃₃ NO ₈
<i>M</i> _r	950.06
Crystal system, space group	Triclinic, <i>P</i> 1
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.4124 (3), 11.3067 (3), 11.3573 (3)
α , β , γ (°)	91.460 (2), 98.011 (2), 104.143 (2)
<i>V</i> (Å ³)	1281.45 (6)
<i>Z</i>	1
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	0.75
Crystal size (mm)	0.20 × 0.06 × 0.04
Data collection	
Diffractometer	Dtrek- <i>CrysAlis PRO</i> -abstract goniometer imported rigaku- <i>D*TREK</i> images diffractometer
Absorption correction	Multi-scan <i>CrysAlis PRO</i> 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
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-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
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-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)

$\text{C}_{25}\text{H}_{34}\text{N}_2\text{O}_7 \cdot \text{C}_{25}\text{H}_{33}\text{NO}_8$	$Z = 1$
$M_r = 950.06$	$F(000) = 508$
Triclinic, $P1$	$D_x = 1.231 \text{ Mg m}^{-3}$
$a = 10.4124 (3) \text{ \AA}$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
$b = 11.3067 (3) \text{ \AA}$	Cell parameters from 27030 reflections
$c = 11.3573 (3) \text{ \AA}$	$\theta = 3.9\text{--}72.7^\circ$
$\alpha = 91.460 (2)^\circ$	$\mu = 0.75 \text{ mm}^{-1}$
$\beta = 98.011 (2)^\circ$	$T = 293 \text{ K}$
$\gamma = 104.143 (2)^\circ$	Plate, colourless
$V = 1281.45 (6) \text{ \AA}^3$	$0.20 \times 0.06 \times 0.04 \text{ mm}$

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	$R_{\text{int}} = 0.113$
ω scans	$\theta_{\text{max}} = 65.0^\circ$, $\theta_{\text{min}} = 3.9^\circ$
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.	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.663$, $T_{\text{max}} = 1.000$	$k = -13 \rightarrow 13$
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 $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta_{\text{max}} = 0.65 \text{ e } \text{\AA}^{-3}$
7928 reflections	$\Delta_{\text{min}} = -0.47 \text{ e } \text{\AA}^{-3}$
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)