

NMR Spectra and HPLC Profiles  
for

**Enantioselective Synthesis of  $\gamma$ -Tetrasubstituted  $\gamma$ -Nitro- $\gamma$ -sulfonyl Carboxylates and Amides *via* L-*tert*-Leucine-Derived-Squaramide Catalyzed Conjugate Addition of  $\alpha$ -Nitrosulfones to Acrylates and Acrylamides**

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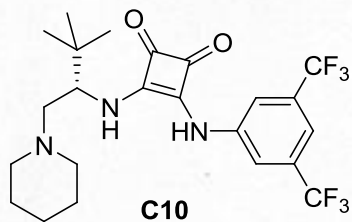
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NAME INN-4-KSB-46B-1H  
EXPNO 10  
PROCNO 1  
Date 20130422  
Time 11.17  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 49340  
SOLVENT DMSO  
NS 13  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.166674 Hz  
AQ 2.9999220 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 294.5 K  
D1 1.00000000 sec  
TDO 1

INN-4-KSB-46B-1H



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PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300364 MHz  
WDW EM  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

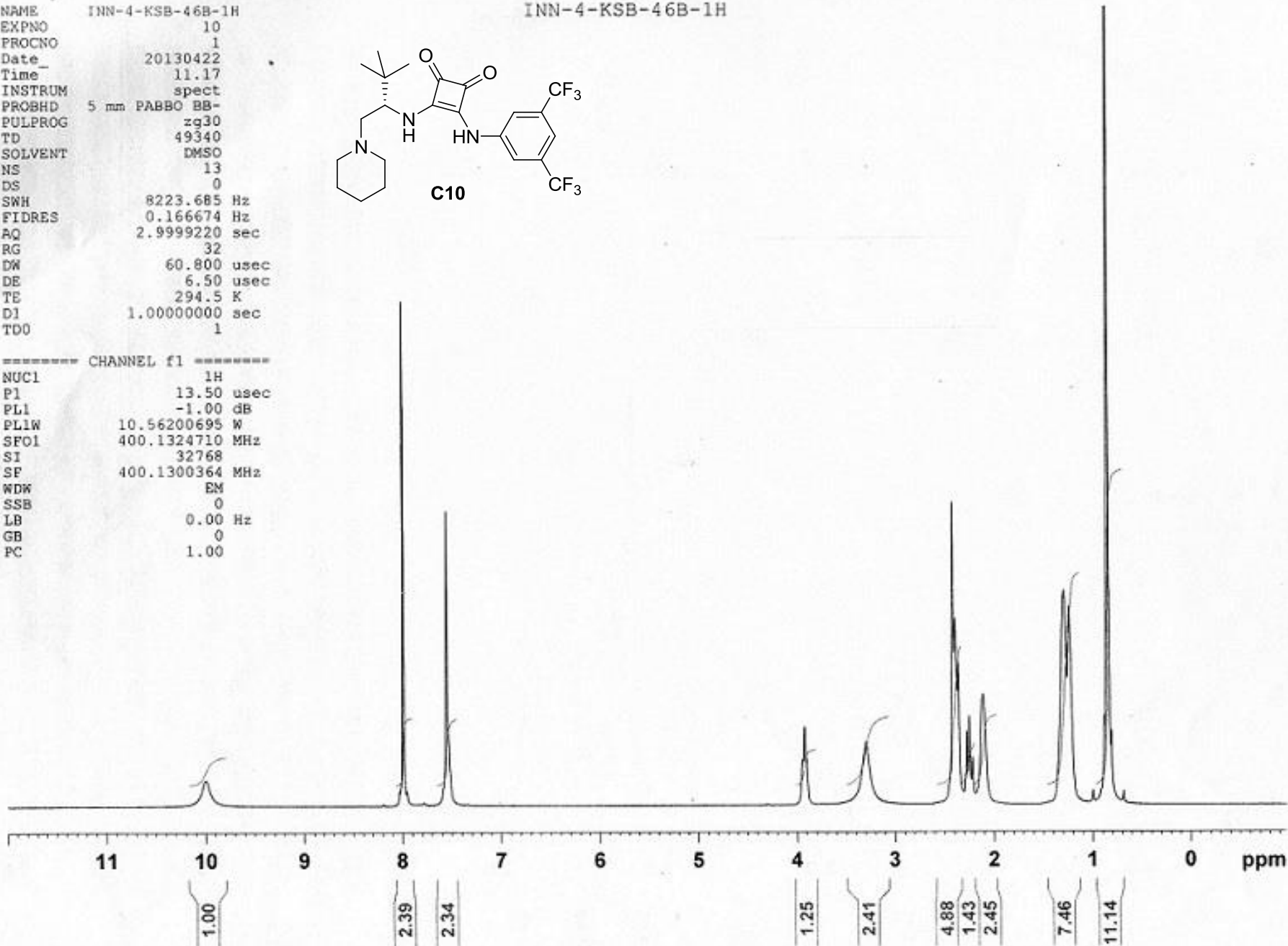


Fig S1. <sup>1</sup>H NMR Spectrum of C10

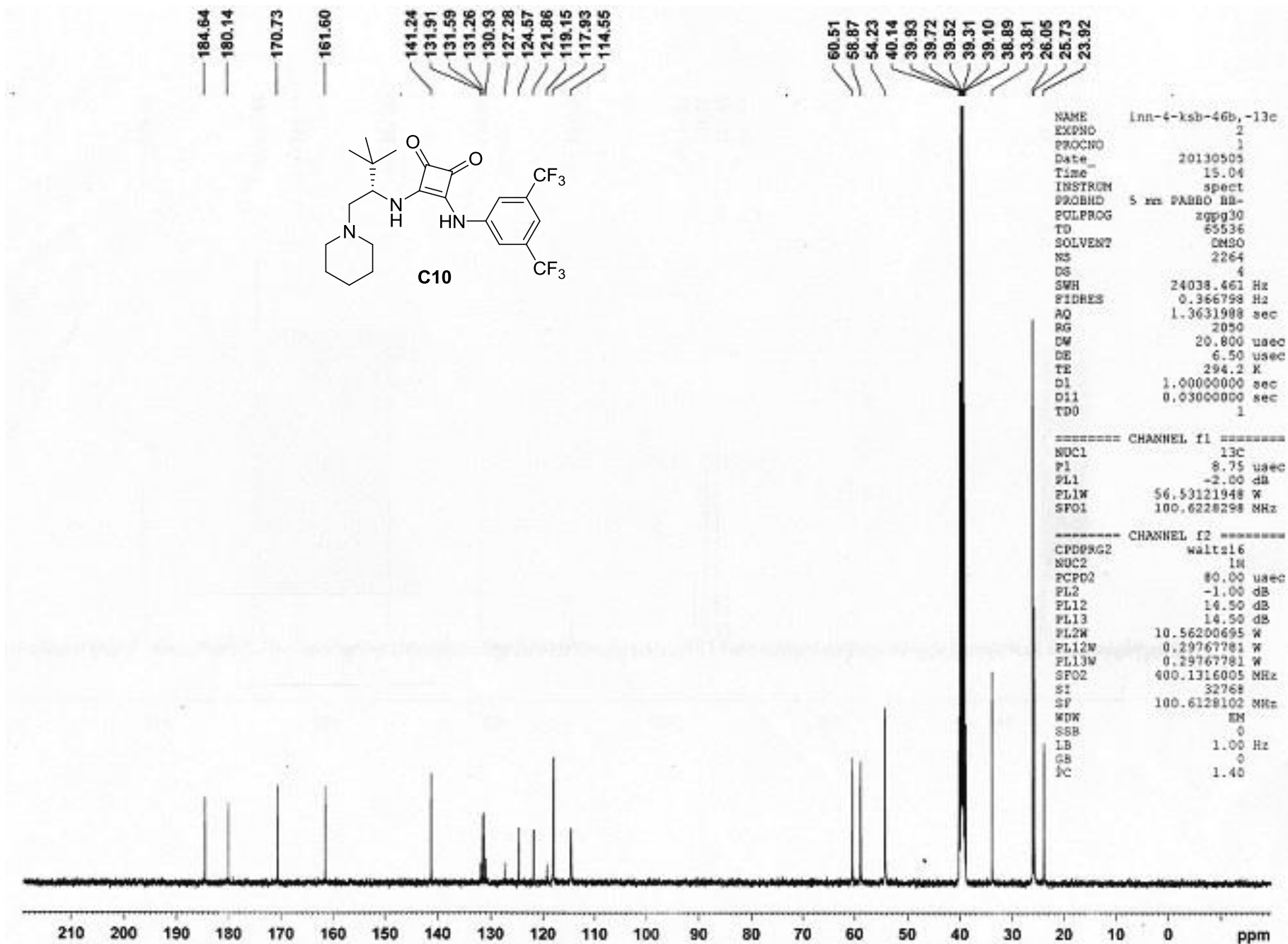
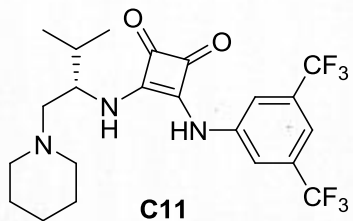


Fig S2. <sup>13</sup>C NMR Spectrum of C10

NAME INN-4-KSB-98C-1H  
 EXPNO 7  
 PROCNO 1  
 Date\_ 20130728  
 Time\_ 19.38  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 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 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  
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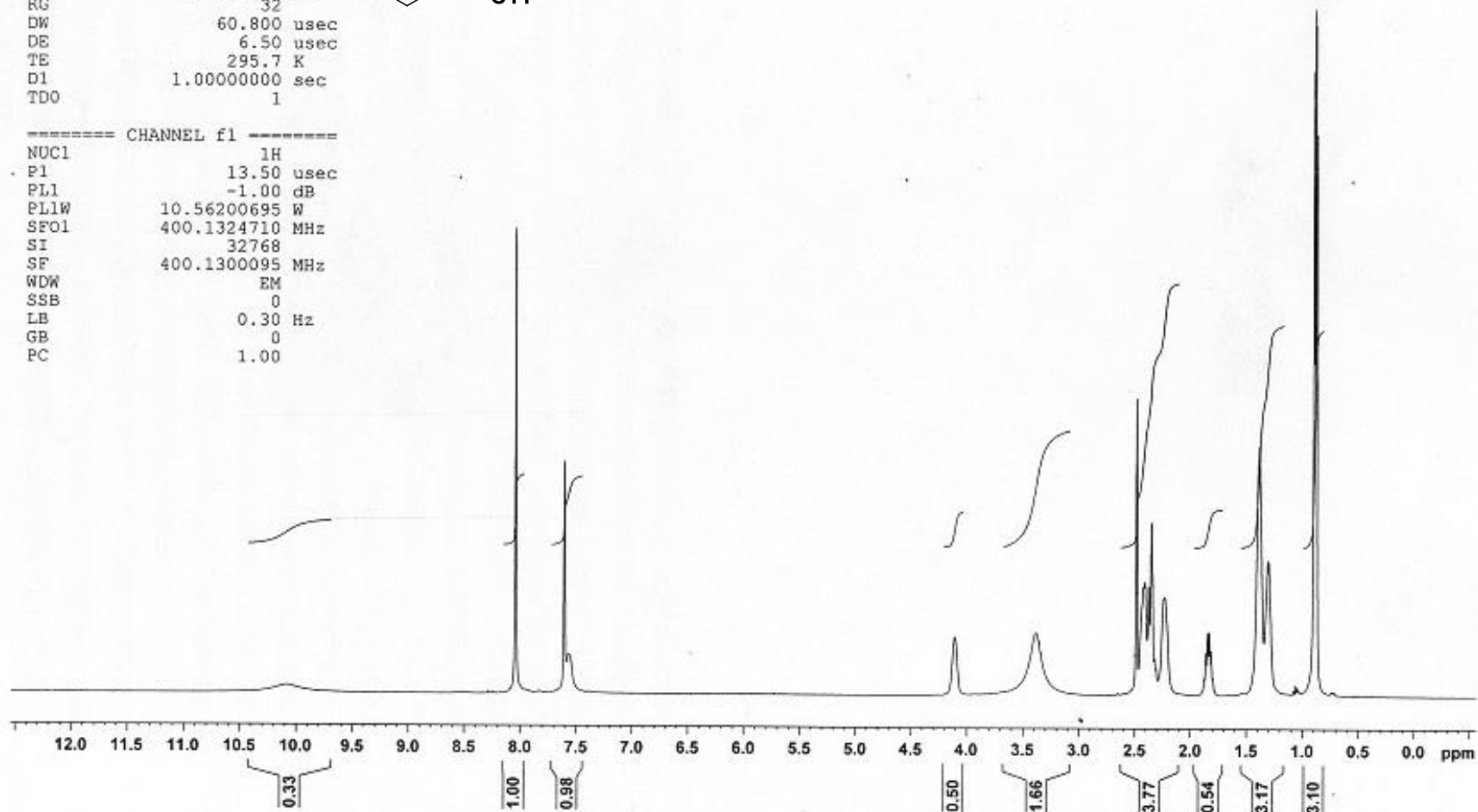


Fig S3. <sup>1</sup>H NMR Spectrum of C11

```

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PROCNO   1
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Time     21.58
INSTRUM  spect
PROBHD   5 mm FAREO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DM80
NS       874
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3031988 sec
RG       2050
CN       20.900 usec
DC       0.50 usec
TE       295.2 K
DL       1.0000000 sec
D11      0.0300000 sec
T00      1

```

```

***** CHANNEL f1 *****
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      56.51121948 W
SFO1      100.6228298 MHz

```

```

***** CHANNEL f2 *****
CPDPRG2  waltz16
NUC2      1H
PCPO2     80.00 usec
PL2       -1.00 dB
PL12      14.50 dB
PL13      14.50 dB
PL2W      10.58200895 W
PL12W     0.29767781 W
PL13W     0.29767781 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127973 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

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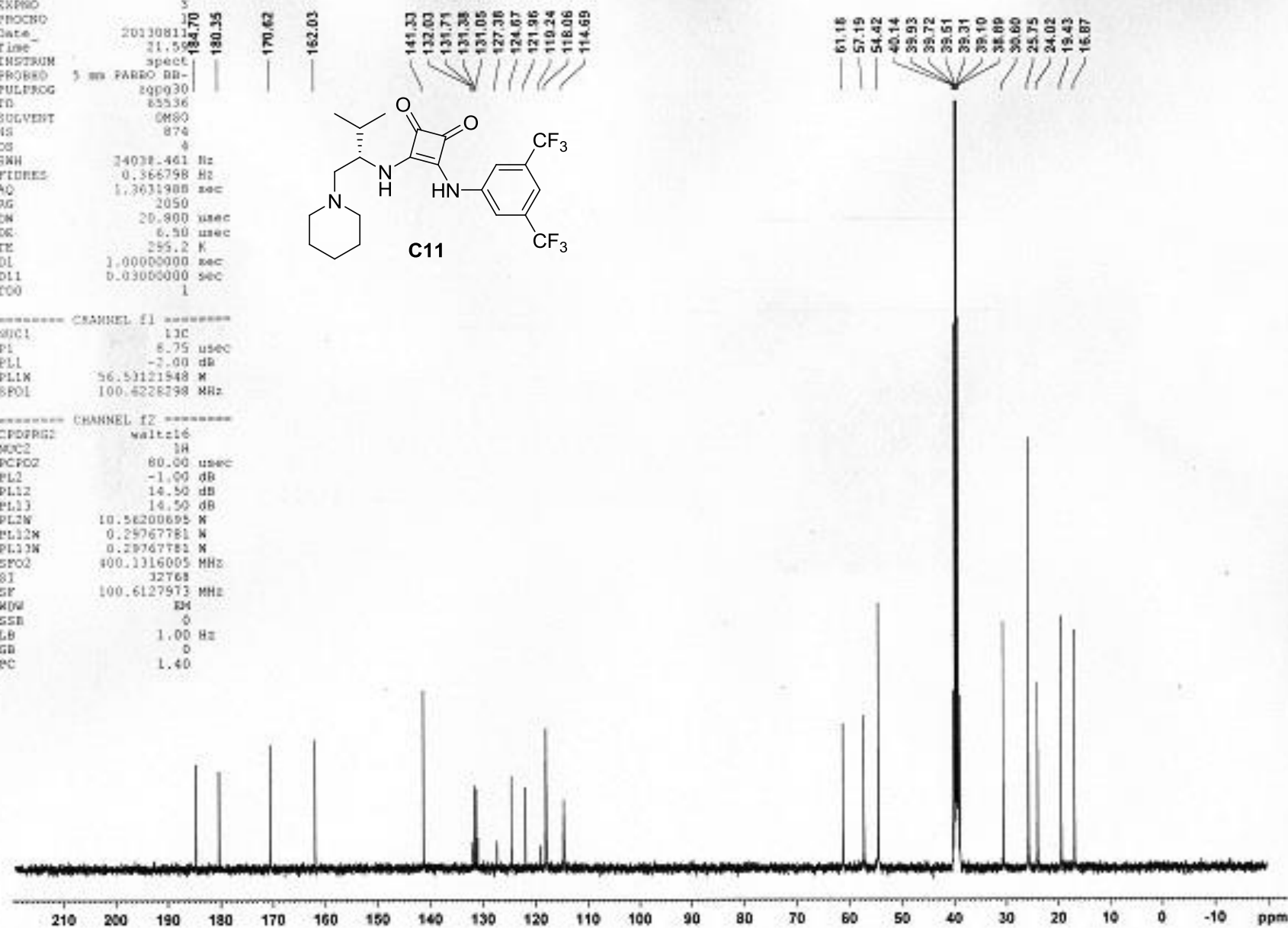
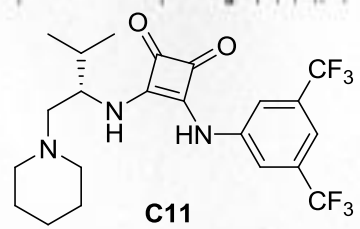


Fig S4. <sup>13</sup>C NMR Spectrum of C11



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 EXPNO 9  
 PROCNO 1  
 Date\_ 20130728  
 Time\_ 19.44  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 7  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.6 K  
 D1 1.00000000 sec  
 TD0 1

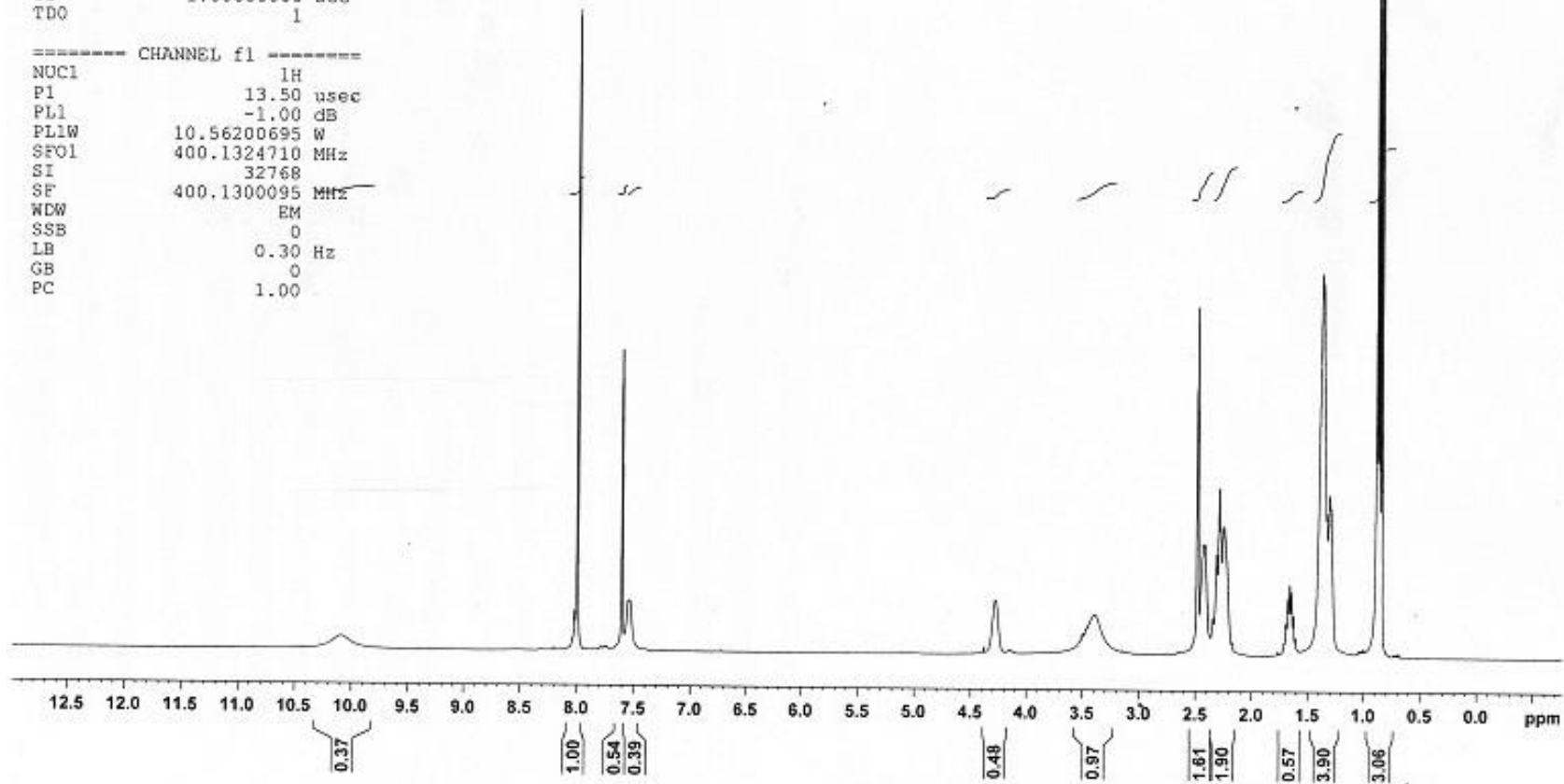
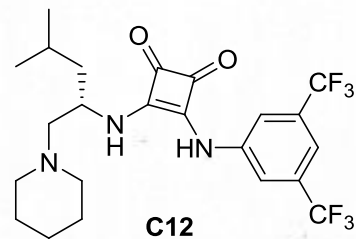


Fig S5. <sup>1</sup>H NMR Spectrum of C12

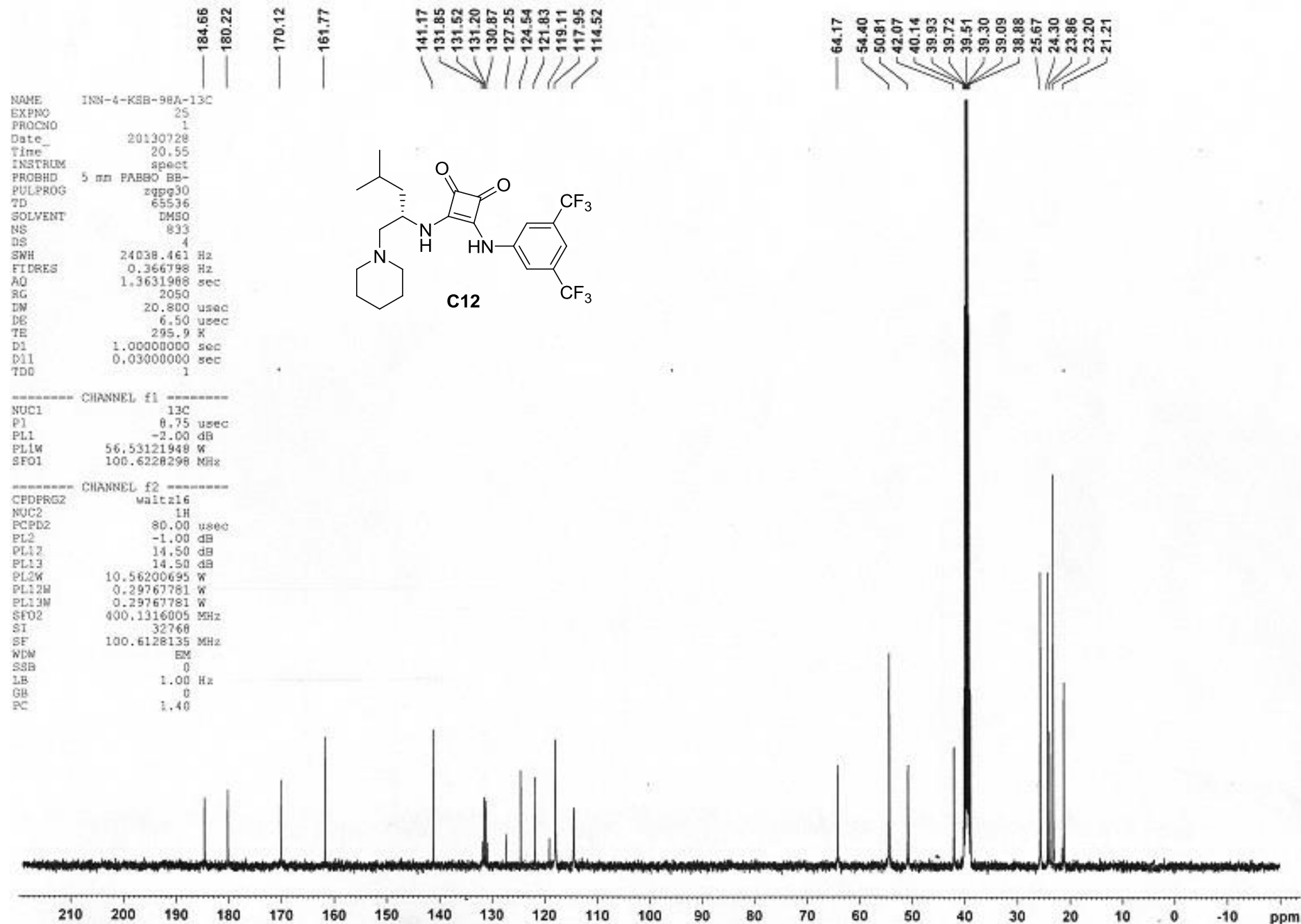
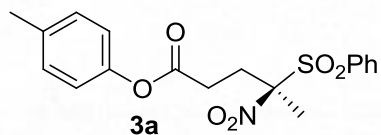


Fig S6. <sup>13</sup>C NMR Spectrum of C12

NAME INN-3-KSB-187A-1H  
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 PROCNO 1  
 Date 20121122  
 Time 20.38  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 16  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 57  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 293.7 K  
 D1 1.00000000 sec  
 TD0 1



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 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

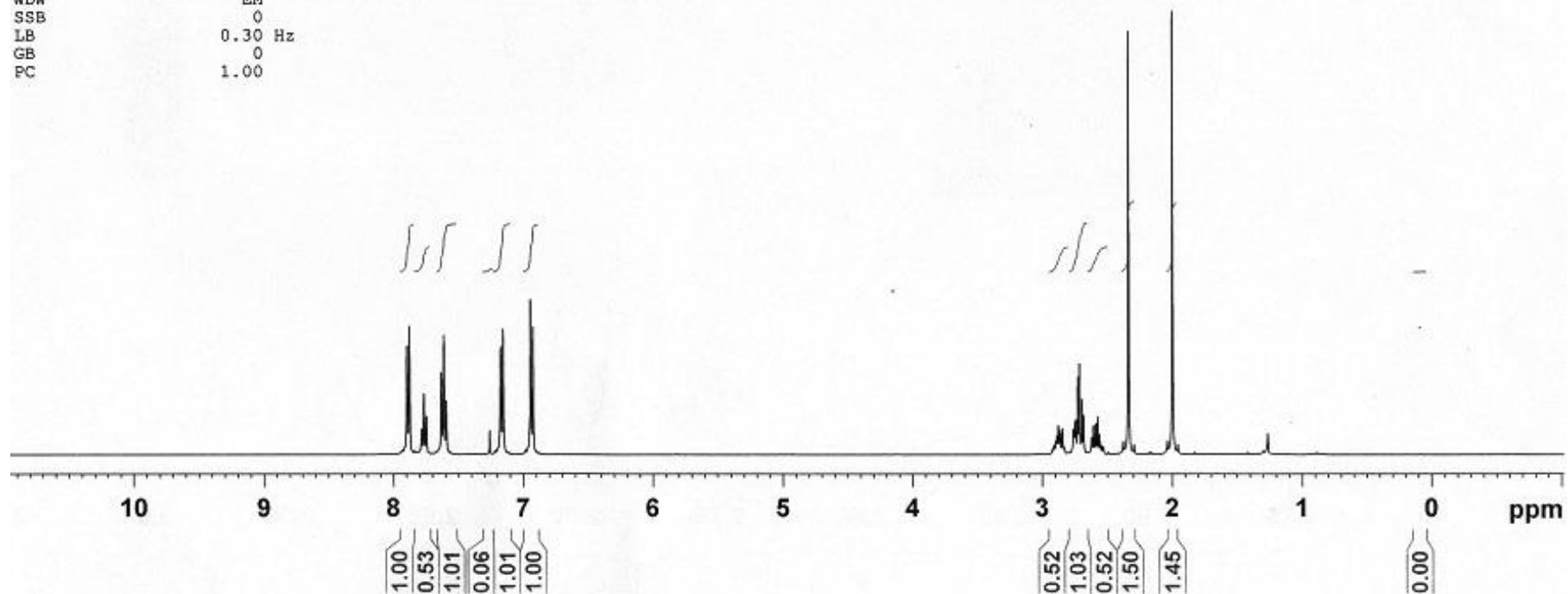


Fig S7. <sup>1</sup>H NMR Spectrum of 3a

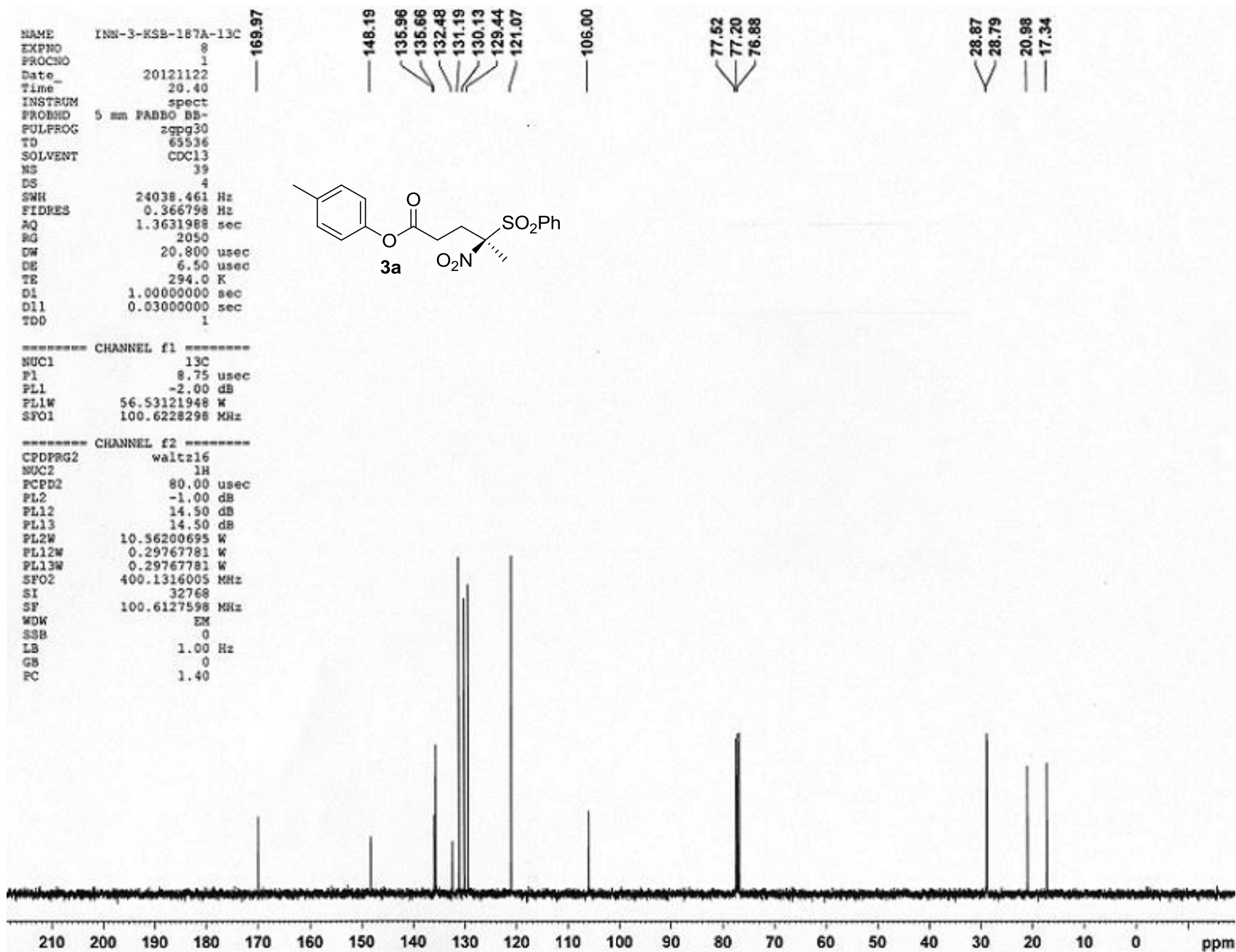
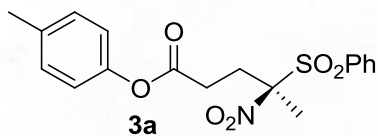


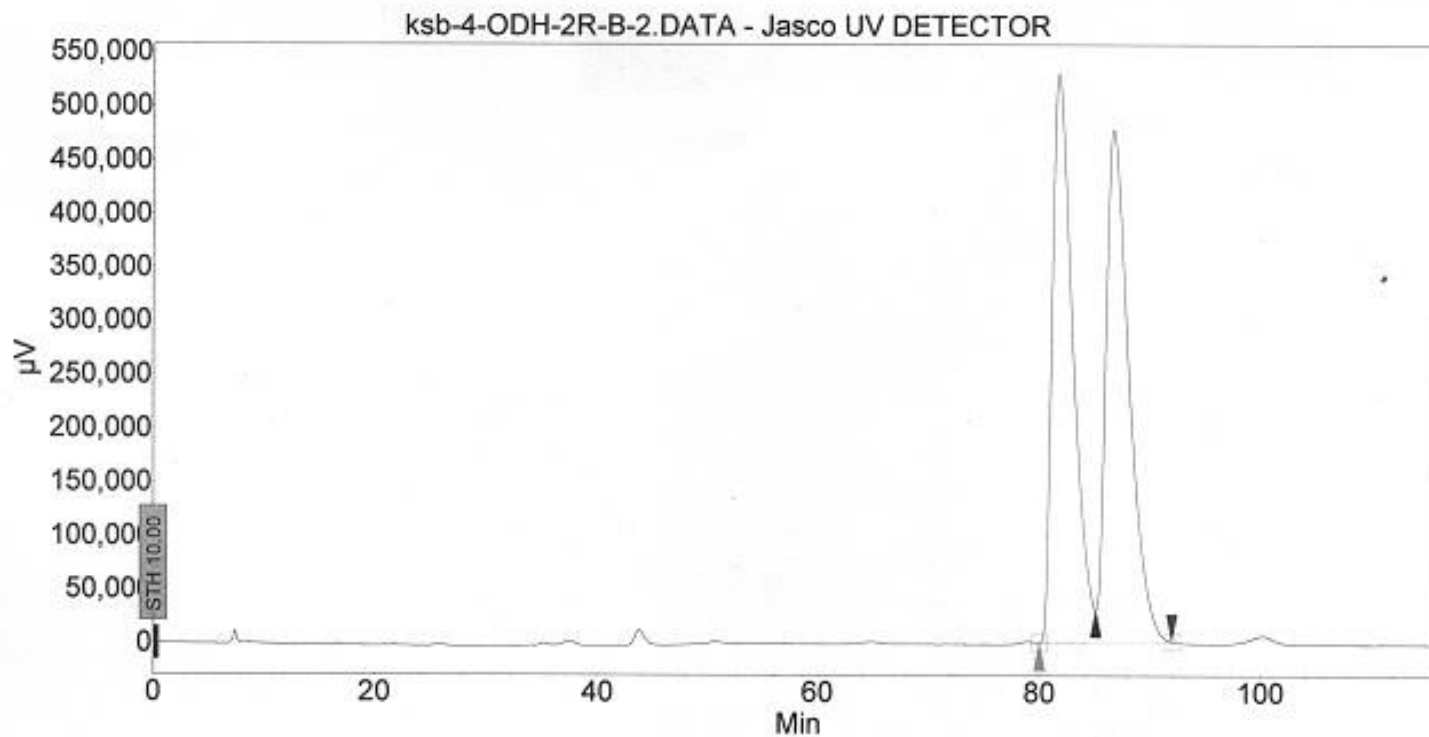
Fig S8. <sup>13</sup>C NMR Spectrum of 3a

# Chromatogram : ksb-4-ODH-2R-B-2\_channel2

System : JASCO HPLC  
Method : ksb-5-05f-216  
User : User1



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Processed : 12/5/2012 9:07:00 PM  
Printed : 5/31/2013 4:31:01 PM



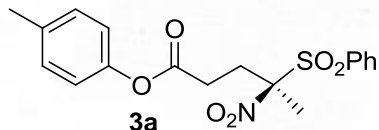
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	81.700	49.70	529339.3	1145375.1	49.703
2	UNKNOWN	86.617	50.30	476969.4	1159066.0	50.297
Total			100.00	1006308.7	2304441.1	100.000

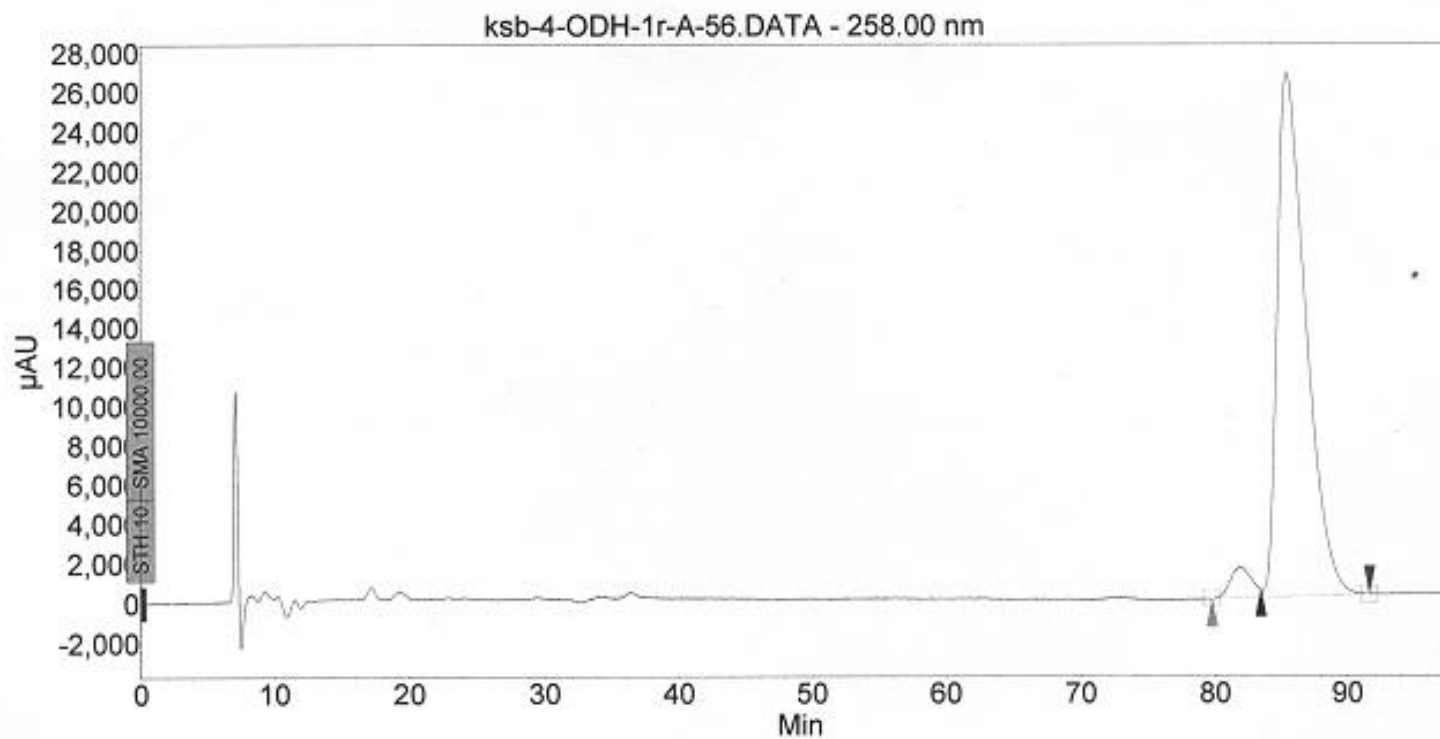
Fig S9. HPLC Profile of Racemic 3a

# Chromatogram : ksb-4-ODH-1r-A-56\_channel5

System : JASCO HPLC  
Method : ksb-5-05f-258  
User : User1



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Printed : 5/31/2013 4:08:37 PM

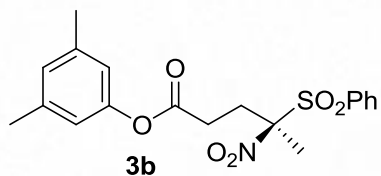


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	81.864	4.18	1539.7	2910.2	4.182
2	UNKNOWN	85.436	95.82	26663.7	66681.6	95.818
Total			100.00	28203.4	69591.7	100.000

Fig S10. HPLC Profile of Enantioenriched 3a

NAME INN-4-KSB-33C-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130223  
 Time 18.54  
 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 293.8 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

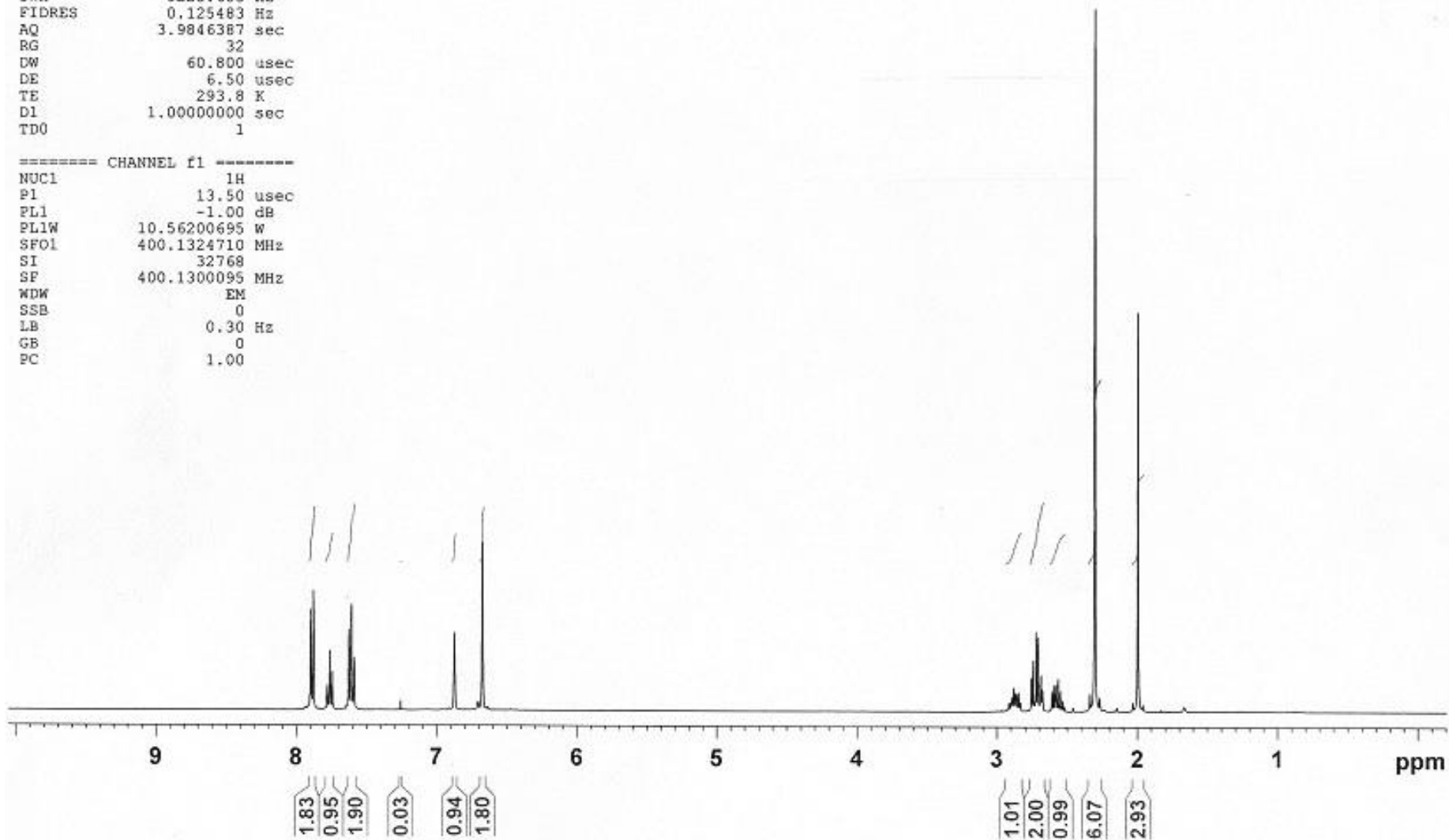
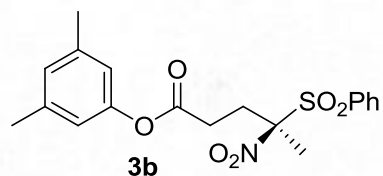


Fig S11. <sup>1</sup>H NMR Spectrum of 3b

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PROCNO   1
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Time     18.54
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       65
DS       4
SMH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       2050
DW       20.800 usec
DE       6.50 usec
TE       293.8 K
D1       1.0000000 sec
D11      0.0300000 sec
TDO      1

```



```

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

```

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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       100.6127657 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       -0
PC       1.40

```

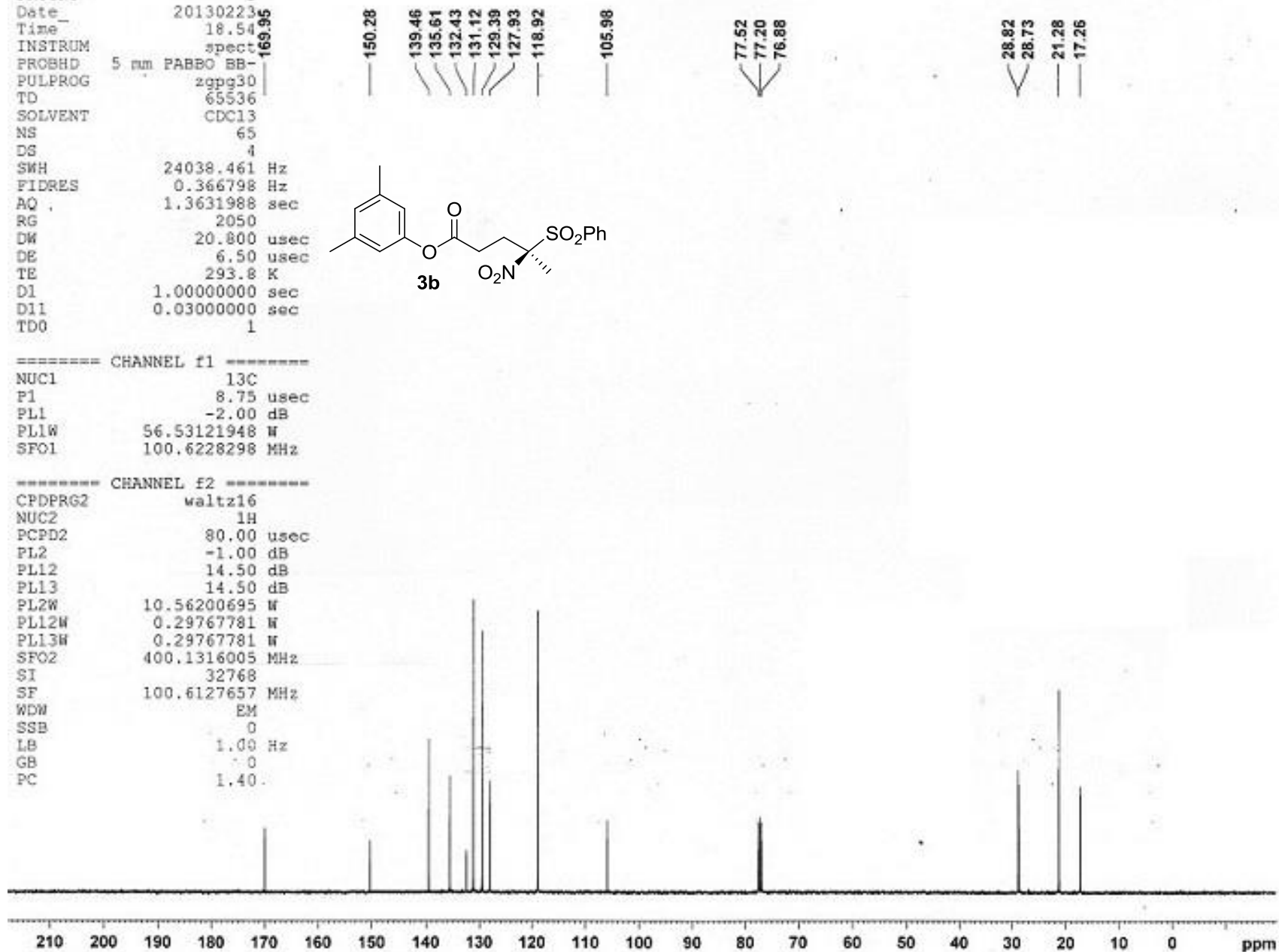
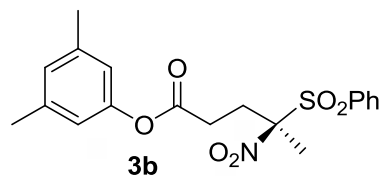


Fig S12. <sup>13</sup>C NMR Spectrum of 3b

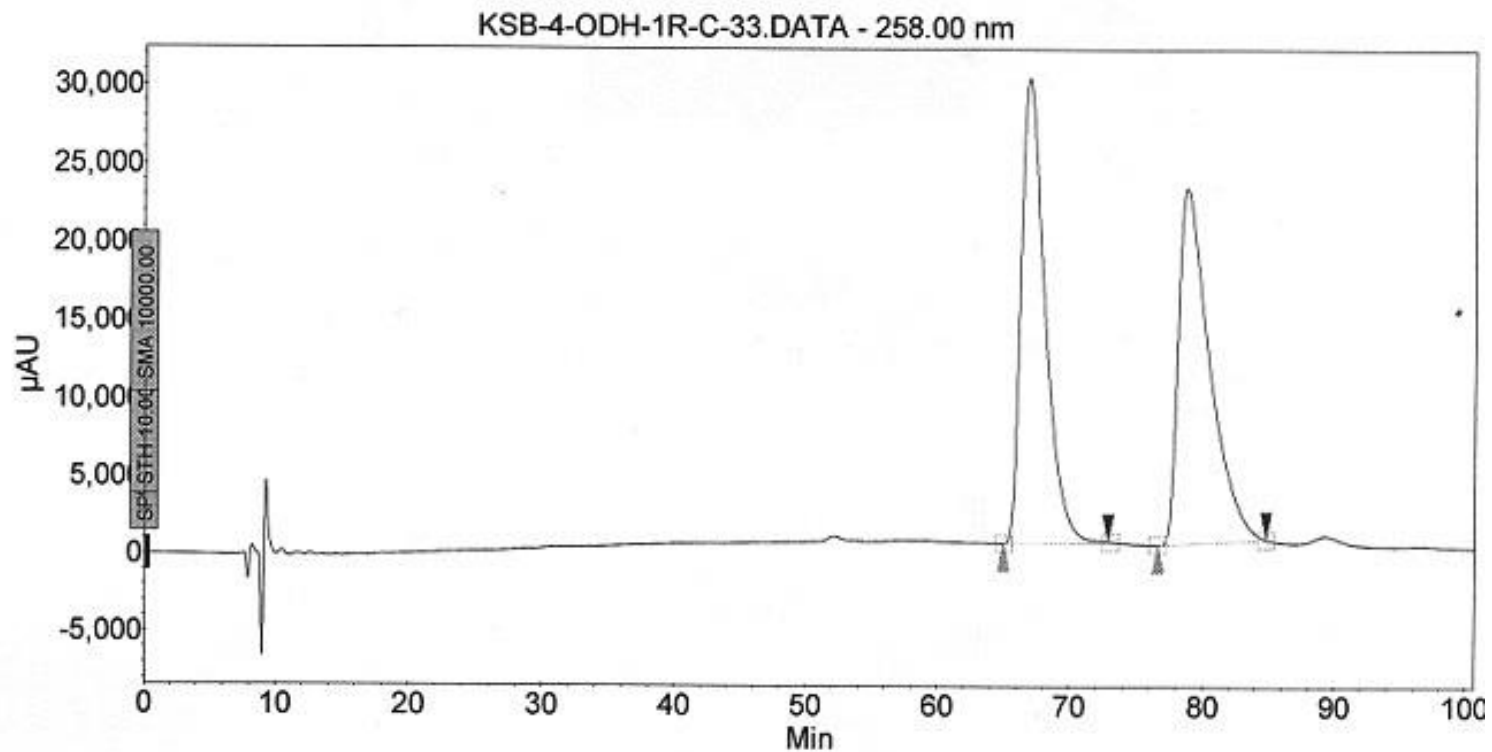


# Chromatogram : KSB-4-ODH-1R-C-33\_channel5

System : JASCO HPLC  
Method : ksb-5-05f-216  
User : User1



Acquired : 2/27/2013 1:46:51 PM  
Processed : 2/27/2013 4:38:00 PM  
Printed : 2/27/2013 8:46:44 PM



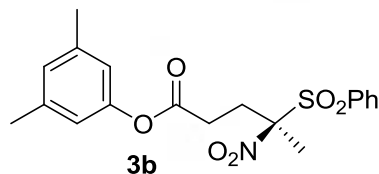
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	66.869	49.90	29726.7	62281.5	49.900
2	UNKNOWN	78.772	50.10	22774.1	62530.7	50.100
Total			100.00	52500.8	124812.2	100.000

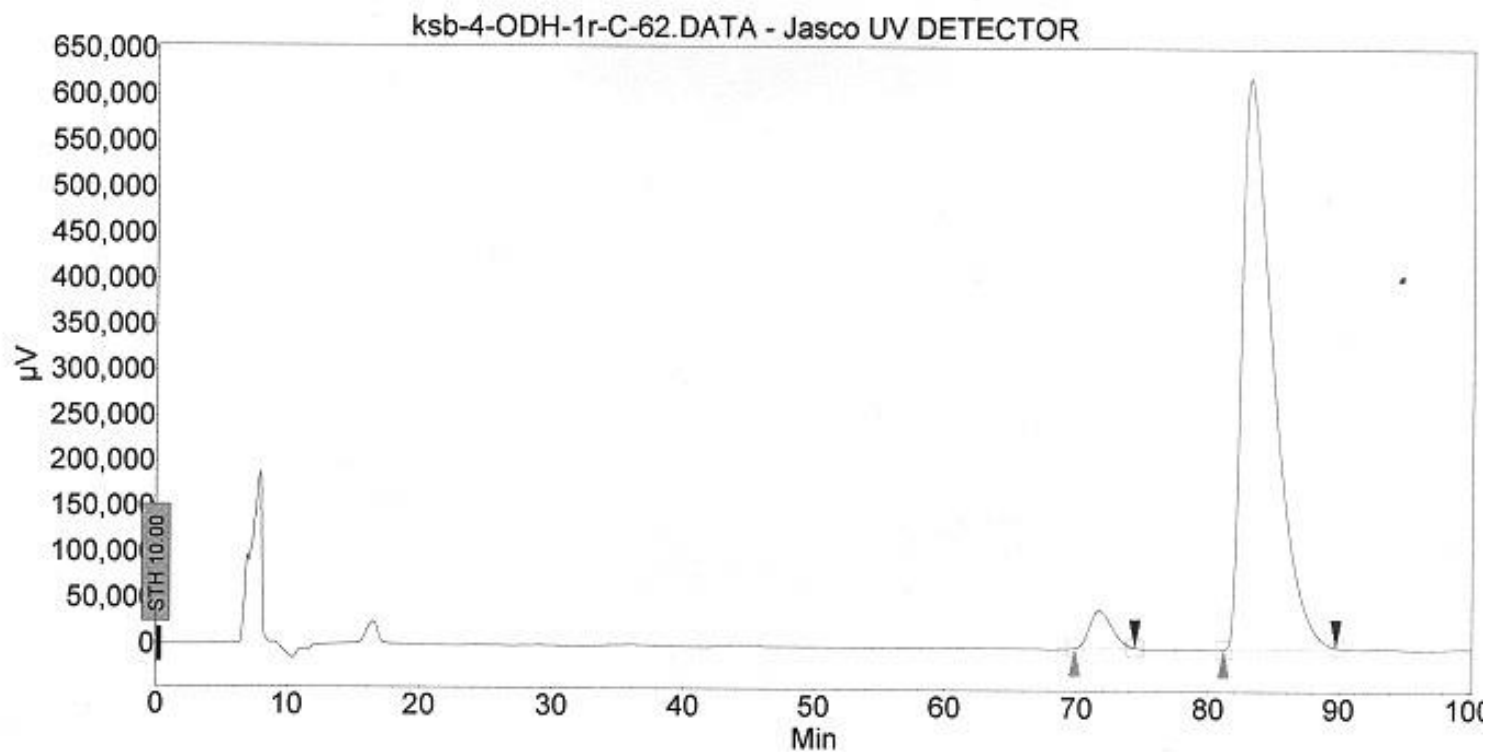
Fig S13. HPLC Profile of Racemic 3b

# Chromatogram : ksb-4-ODH-1r-C-62\_channel2

System : JASCO HPLC  
Method : ksb-5-05f-258  
User : User1



Acquired : 5/6/2013 3:05:46 PM  
Processed : 5/6/2013 4:46:26 PM  
Printed : 5/6/2013 10:07:20 PM

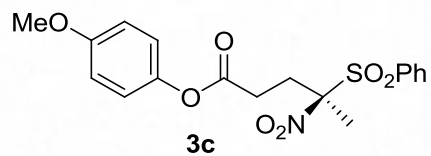


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu$ V]	Area [ $\mu$ V.Min]	Area % [%]
1	UNKNOWN	71.700	4.46	40901.1	80043.6	4.457
2	UNKNOWN	83.075	95.54	622257.7	1715687.9	95.543
Total			100.00	663158.8	1795731.5	100.000

Fig S14. HPLC Profile of Enantioenriched 3b

NAME INN-4-KSB-31B-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20130219  
Time\_ 20.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 14  
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 294.2 K  
D1 1.0000000 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

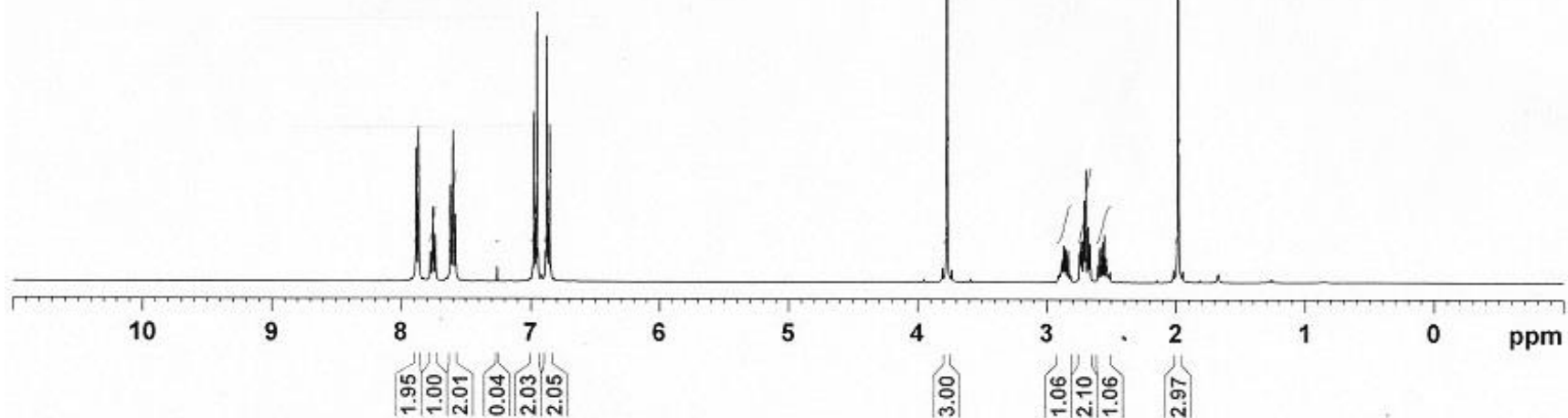


Fig S15. <sup>1</sup>H NMR Spectrum of 3c

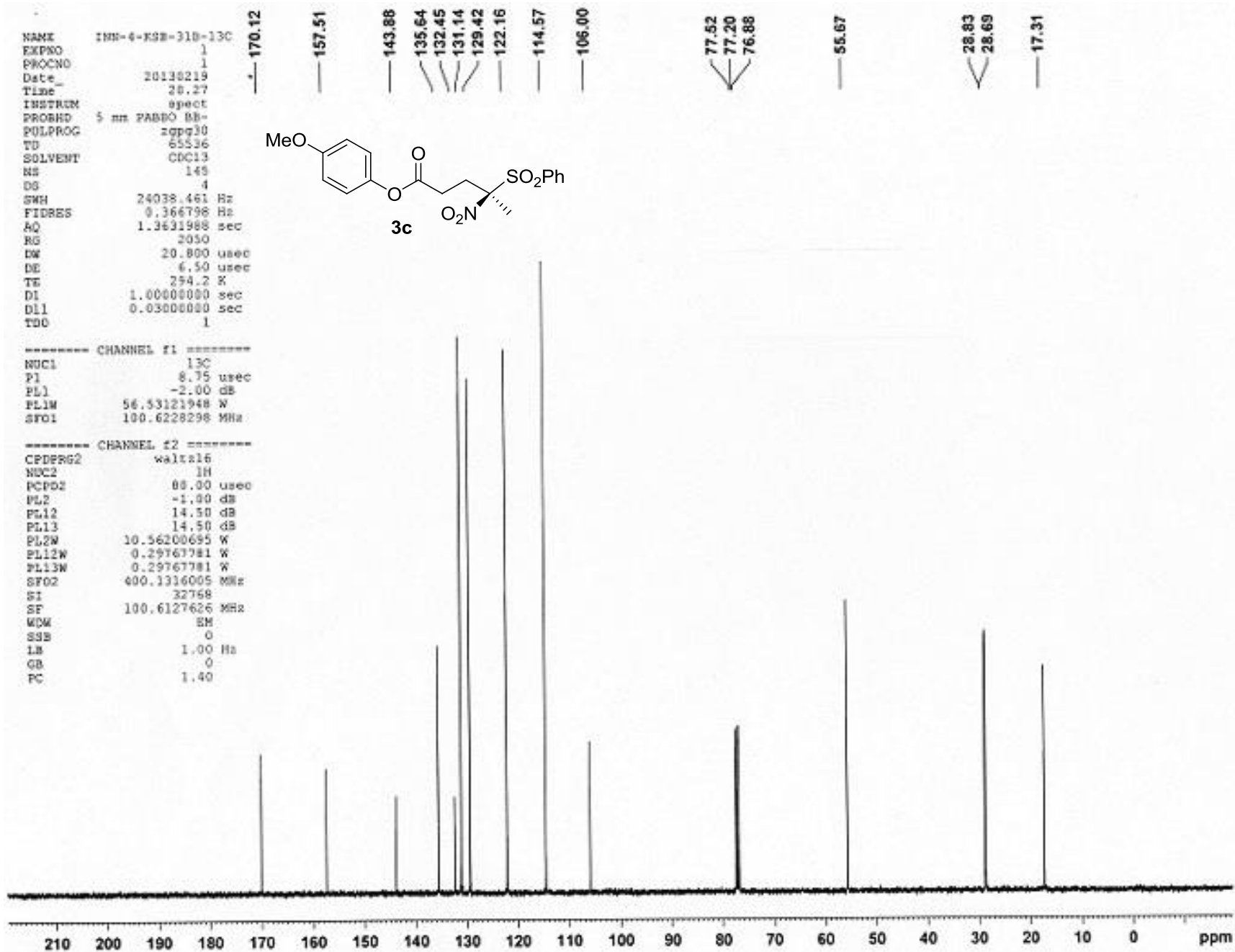
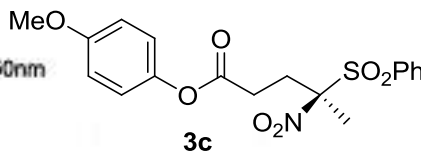


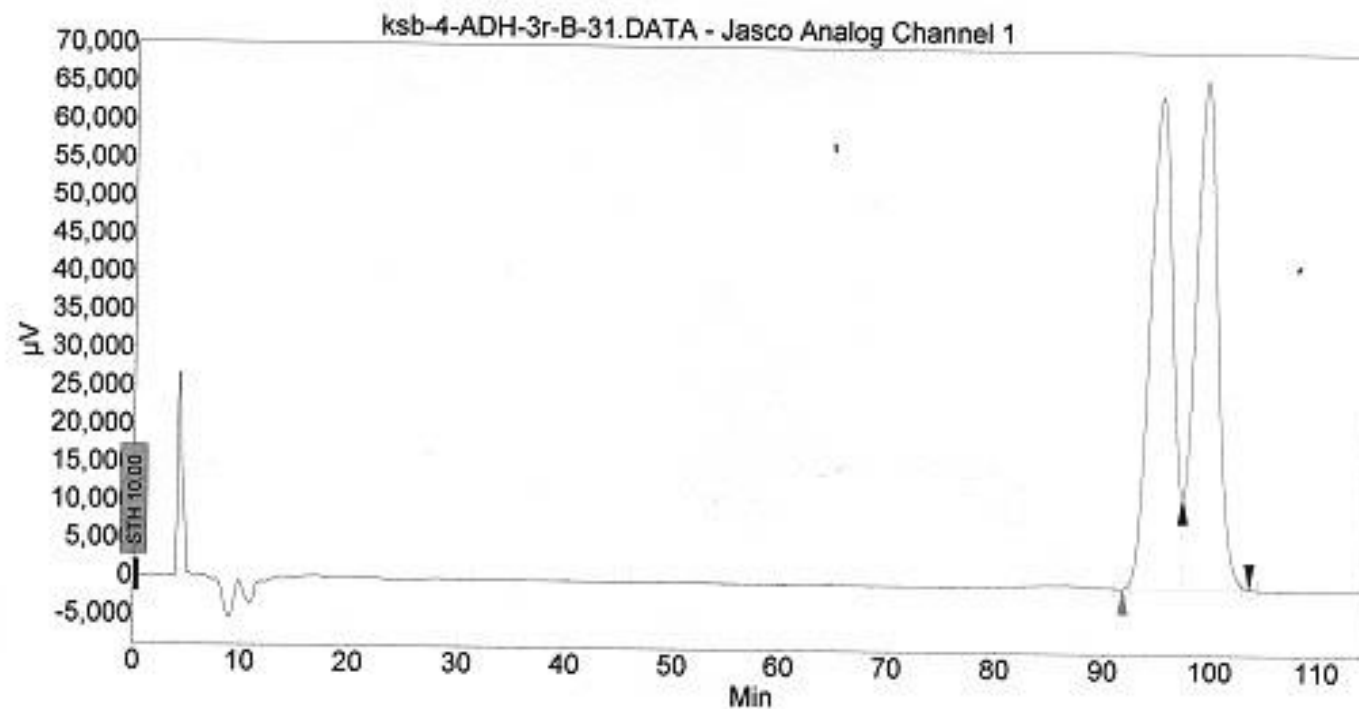
Fig S16. <sup>13</sup>C NMR Spectrum of 3c

## Chromatogram : ksb-4-ADH-3r-B-31\_channel2

System : Jasco HPLC  
 Method : ksb-4%-05-250nm  
 User : User1



Acquired : 5/27/2013 5:33:12 PM  
 Processed : 5/27/2013 7:28:27 PM  
 Printed : 5/27/2013 7:33:39 PM



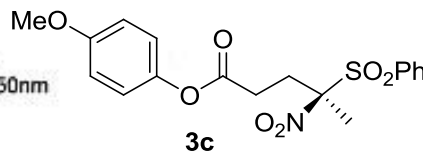
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	95.125	49.52	64755.0	158707.1	49.516
2	UNKNOWN	99.325	50.48	66888.8	161812.4	50.484
Total			100.00	131643.8	320519.5	100.000

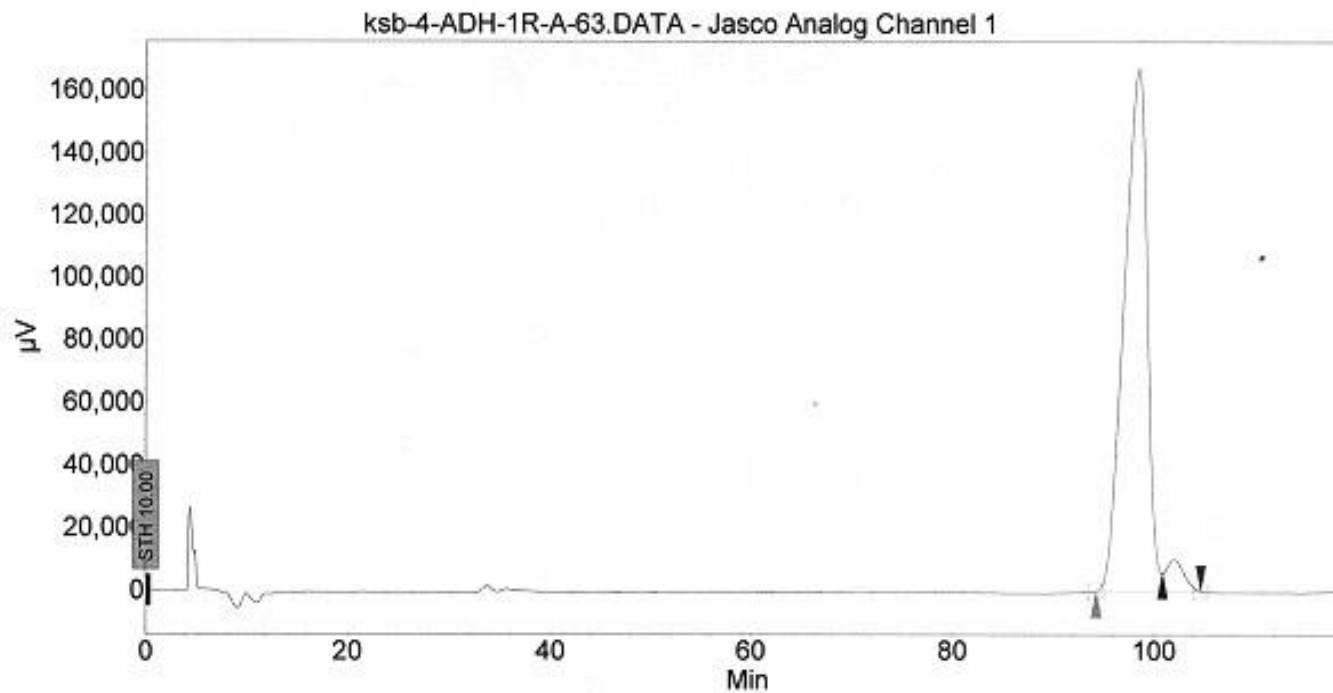
Fig S17. HPLC Profile of Racemic 3c

## Chromatogram : ksb-4-ADH-1R-A-63\_channel2

System : Jasco HPLC  
Method : ksb-4%-05-250nm  
User : User1



Acquired : 5/27/2013 1:41:24 PM  
Processed : 5/27/2013 3:41:06 PM  
Printed : 5/27/2013 3:44:16 PM



### Peak results :

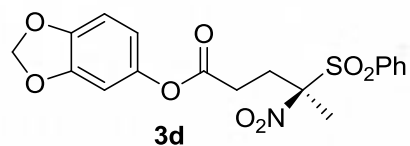
Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	98.483	95.51	167473.7	447971.3	95.511
2	UNKNOWN	102.008	4.49	10125.0	21055.5	4.489
Total			100.00	177598.7	469026.8	100.000

Fig S18. HPLC Profile of Enantioenriched 3c

```

NAME      INN-4-KSB-32C-1H
EXPNO    1
PROCNO   1
Date_    20130219
Time     20.37
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       15
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       294.3 K
D1       1.00000000 sec
TD0      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

```

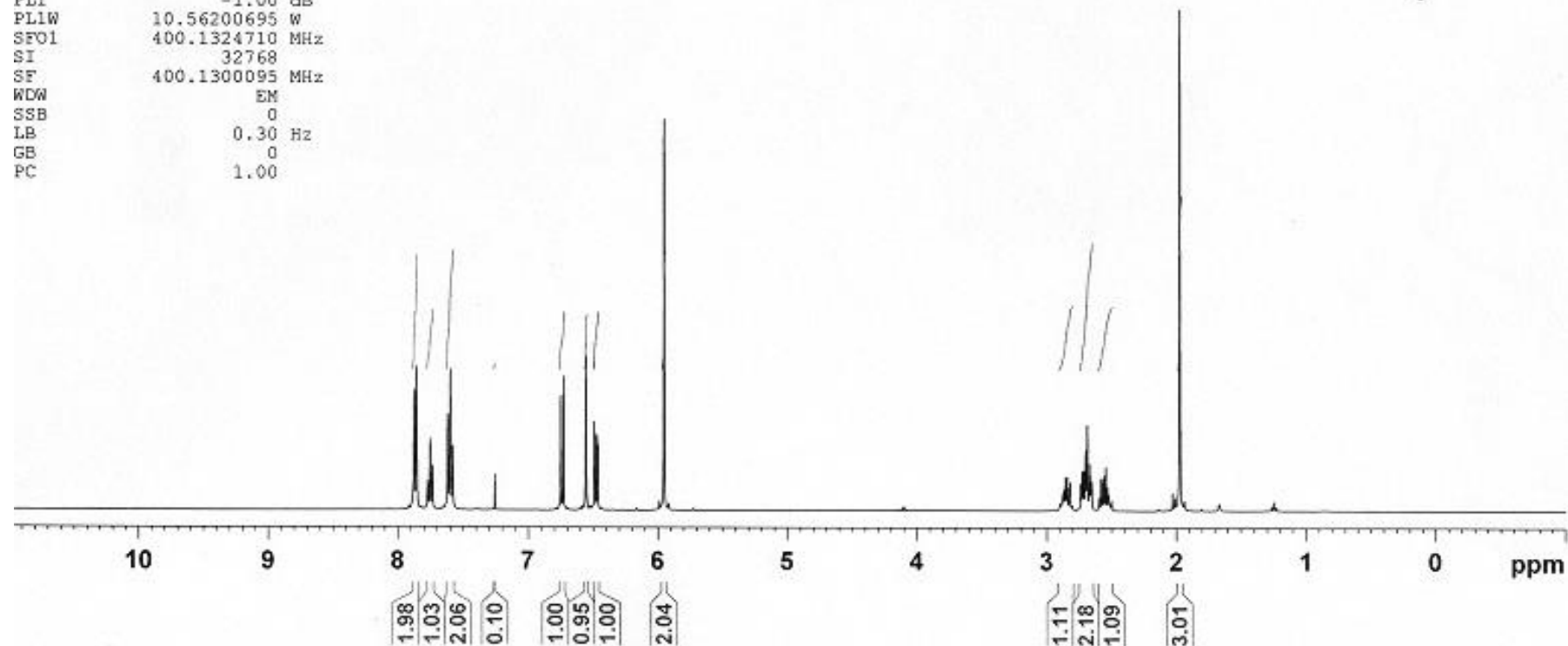


Fig S19. <sup>1</sup>H NMR Spectrum of 3d

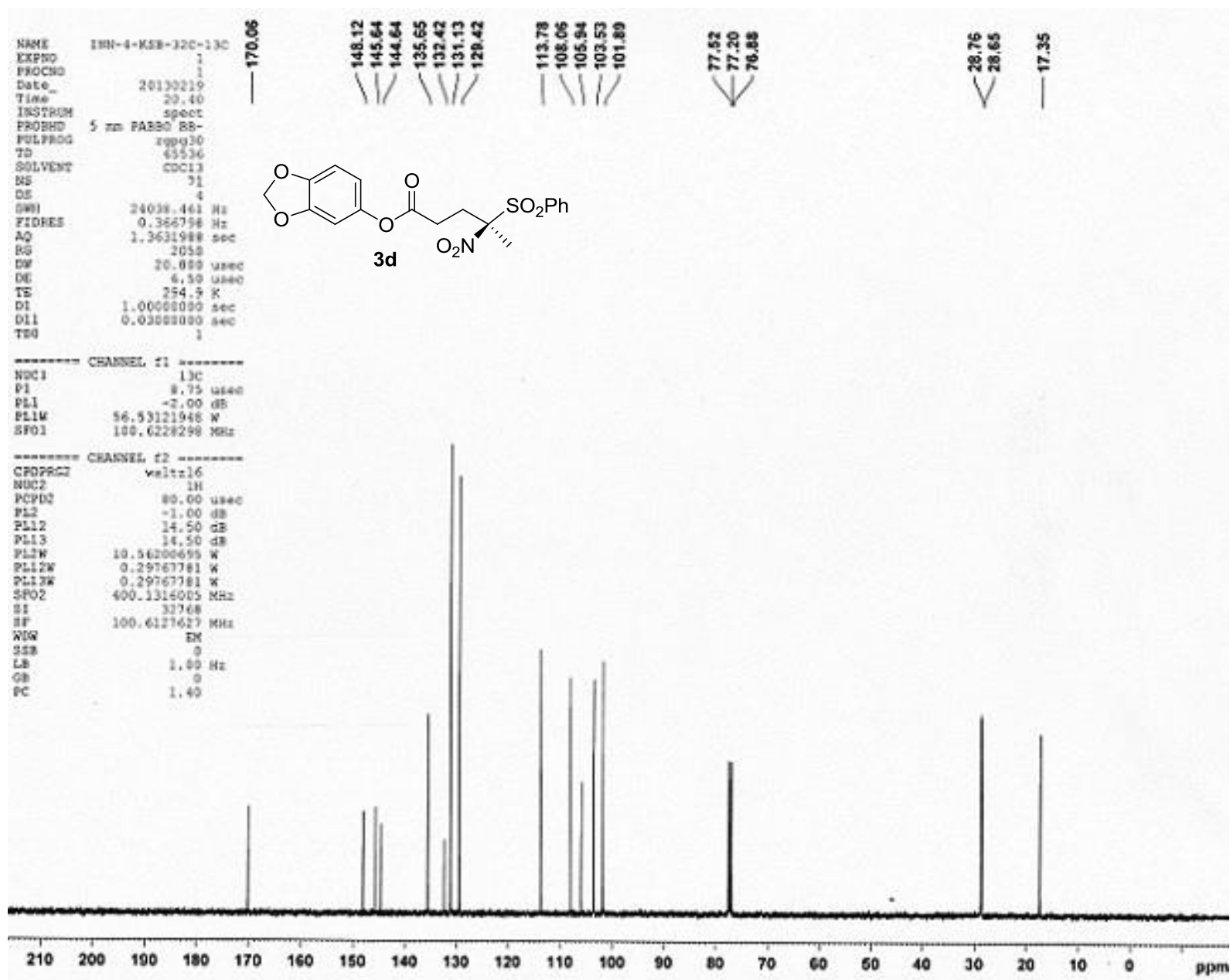
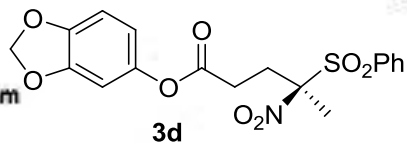


Fig S20. <sup>13</sup>C NMR Spectrum of 3d

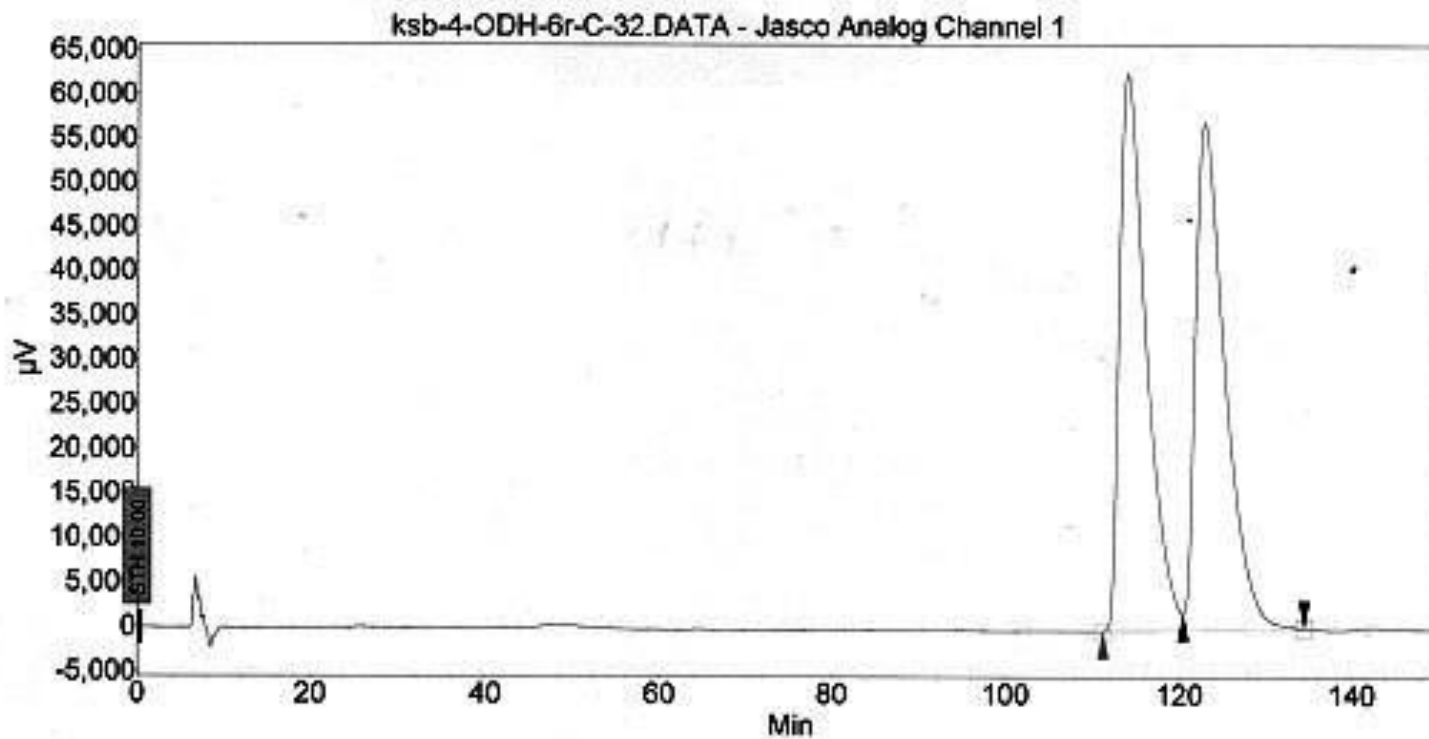


# Chromatogram : ksb-4-ODH-6r-C-32\_channel2

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/9/2013 2:38:15 PM  
Processed : 5/9/2013 8:49:18 PM  
Printed : 5/9/2013 8:50:21 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	113.967	49.89	62674.4	224137.4	49.886
2	UNKNOWN	122.750	50.11	57072.5	225158.7	50.114
Total			100.00	119746.9	449296.1	100.000

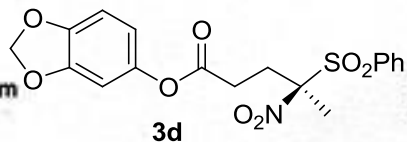
Fig S21. HPLC Profile of Racemic 3d

# Chromatogram : ksb-4-ODH-2r-B-63\_channel4

System : Jasco HPLC

Method : ksb-10%-05f-250nm

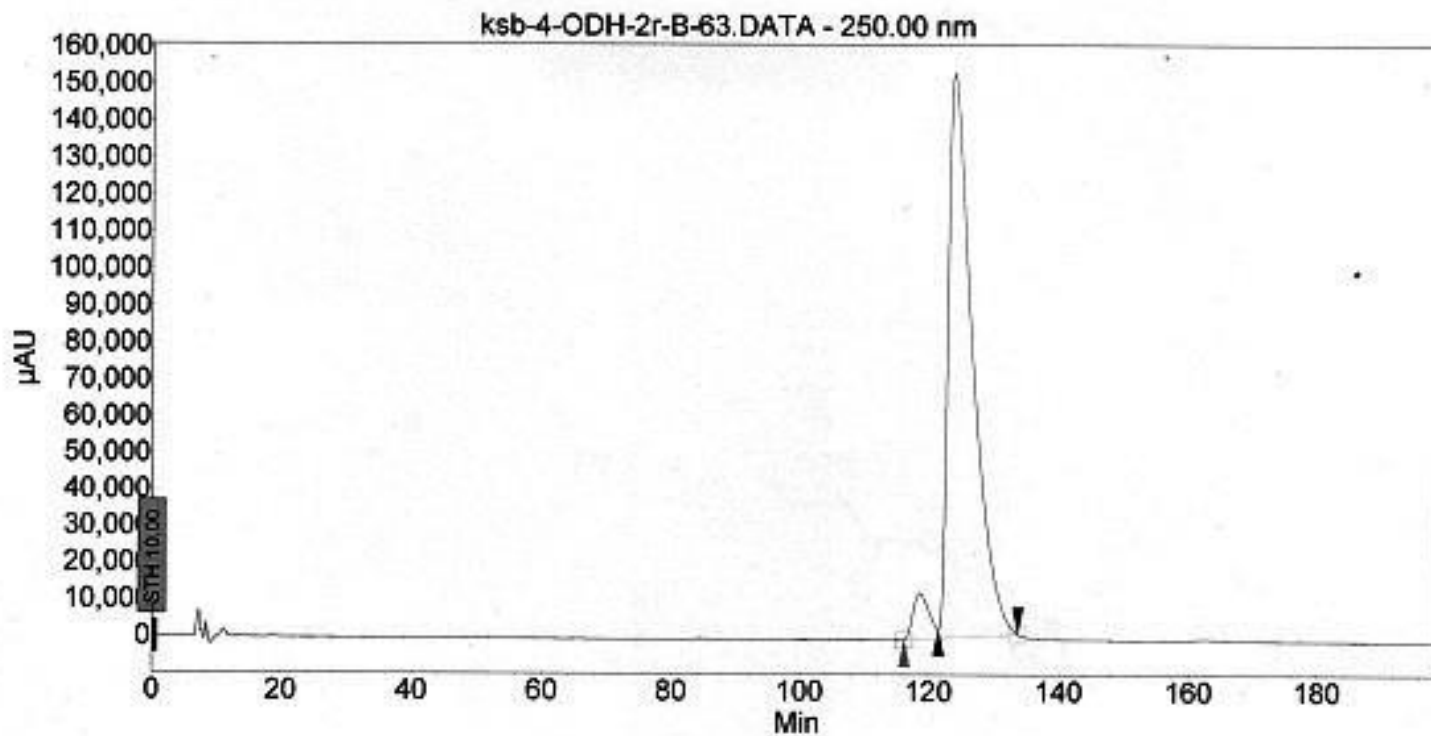
User : User1



Acquired : 5/9/2013 5:22:14 PM

Processed : 5/9/2013 8:42:22 PM

Printed : 5/9/2013 8:50:00 PM

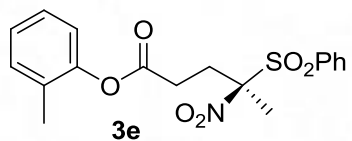


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	118.583	5.07	12373.5	35355.6	5.071
2	UNKNOWN	123.778	94.93	152848.8	661919.6	94.929
Total			100.00	165222.3	697275.3	100.000

Fig S 22. HPLC Profile of Enantioenriched 3d

NAME INN-3-KSB-T0A-1H  
 EXPNO 4  
 PROCNO 1  
 Date\_ 20130518  
 Time 17.34  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 6  
 DS 0  
 SMH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DM 60.800 usec  
 DE 6.50 usec  
 TE 293.5 K  
 D1 1.00000000 sec  
 T00 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

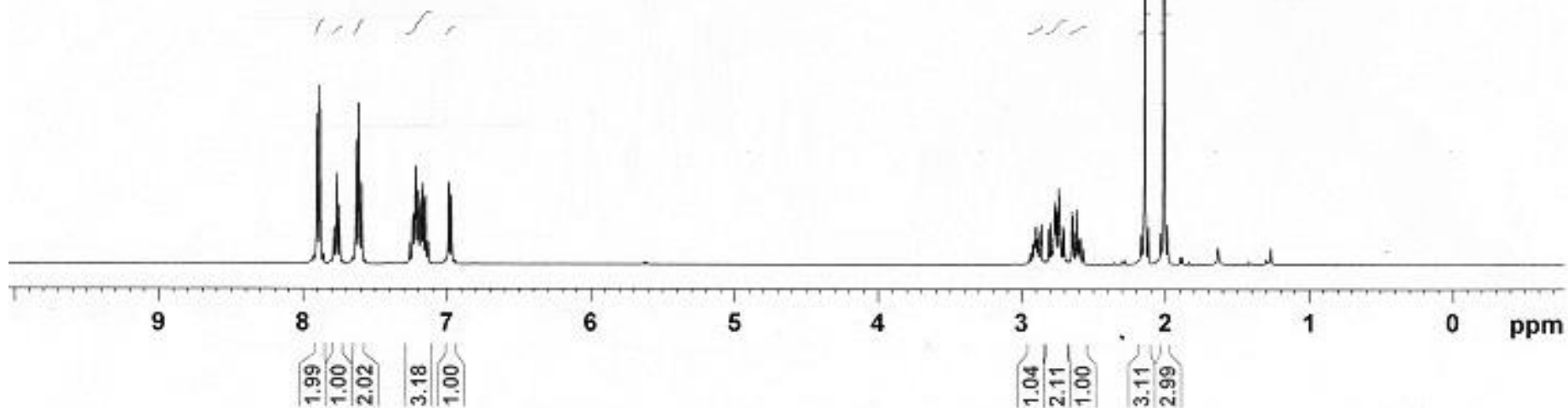


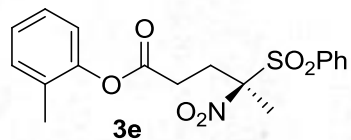
Fig S23. <sup>1</sup>H NMR Spectrum of 3e

```

NAME      INN-3-KSB-70A-13C
EXPNO     4
PROCNO    1
Date_     20130518
Time      17.38
INSTRUM   spect
PROBHD    5 mm PABBO BB-
POLPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        44
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DM        20.800 usec
DE        6.50 usec
TE        293.9 K
D1        1.00000000 sec
D11       0.03000000 sec
TDO       1

```

169.51  
 149.06  
 135.67  
 132.45  
 131.34  
 131.16  
 129.92  
 129.44  
 127.13  
 126.44  
 121.71  
 105.96  
 77.52  
 77.20  
 76.88  
 28.92  
 28.56  
 17.38  
 16.27



```

----- CHANNEL f1 -----
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      56.53121948 W
SFO1      100.6228290 MHz

```

```

===== CHANNEL f2 =====
CFPPRG2   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        100.6127618 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

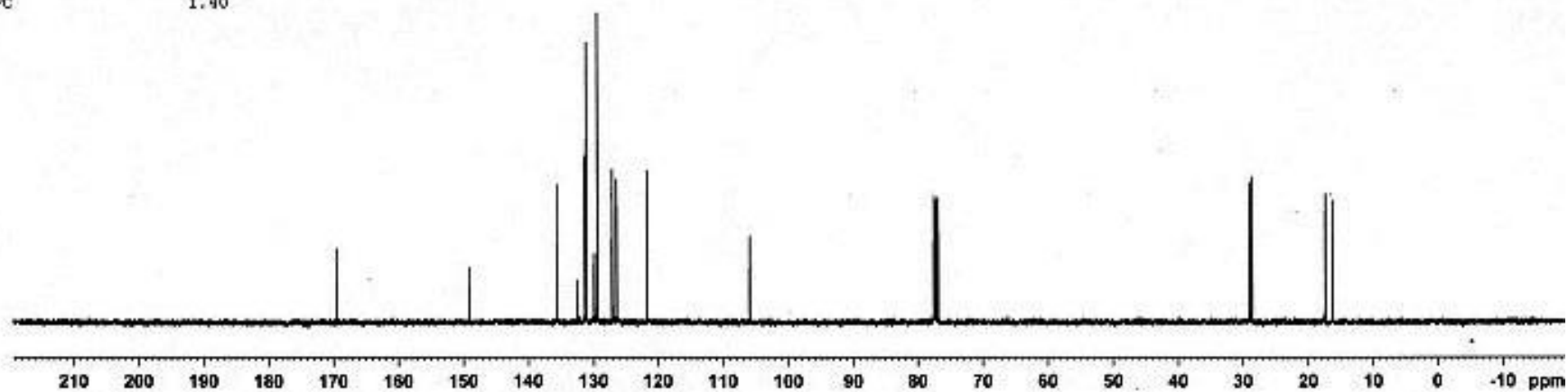
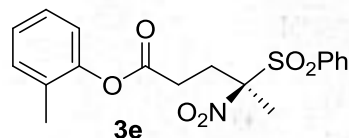


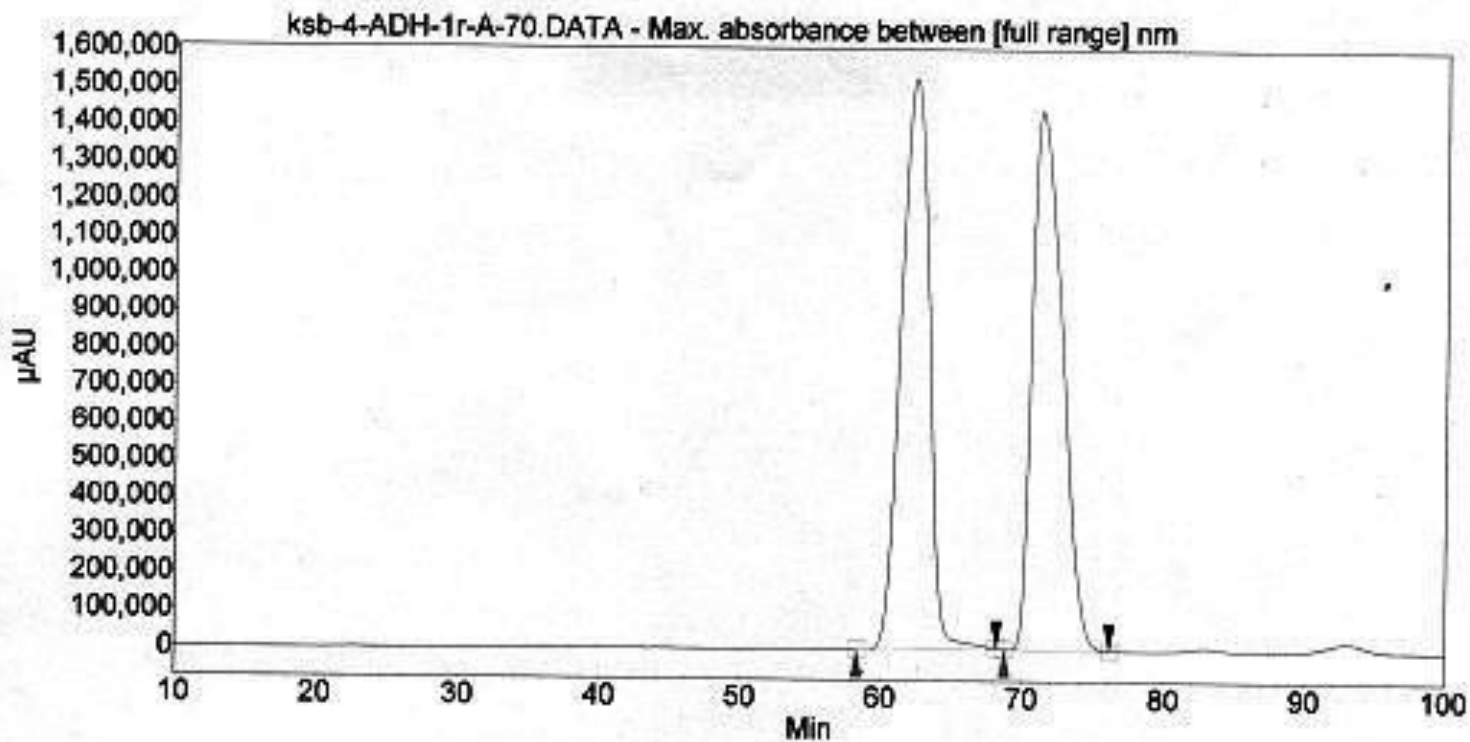
Fig S24. <sup>13</sup>C NMR Spectrum of 3e

# Chromatogram : ksb-4-ADH-1r-A-70\_channel5

System : Jasco HPLC  
 Method : ksb-2%-05f-250nm  
 User : User1



Acquired : 5/25/2013 9:59:25 AM  
 Processed : 5/25/2013 11:54:48 AM  
 Printed : 5/25/2013 11:55:13 AM



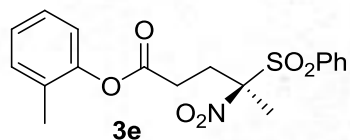
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	62.244	50.08	1522952.0	3534512.9	50.083
1	UNKNOWN	71.188	49.92	1438794.1	3522794.1	49.917
Total			100.00	2961746.1	7057307.0	100.000

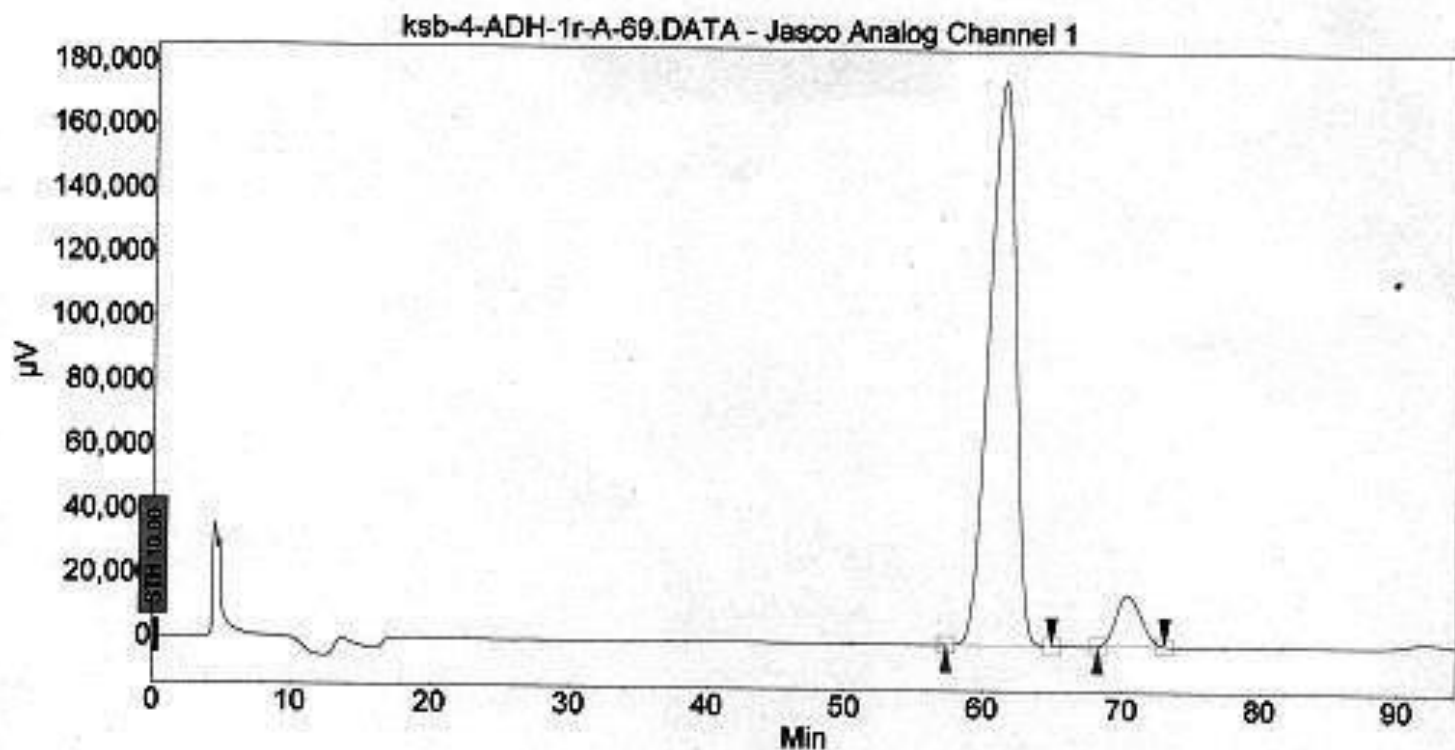
Fig S25. HPLC Profile of Racemic 3e

# Chromatogram : ksb-4-ADH-1r-A-69\_channel2

System : Jasco HPLC  
 Method : ksb-2%-05f-250nm  
 User : User1



Acquired : 5/25/2013 11:41:02 AM  
 Processed : 5/25/2013 1:20:25 PM  
 Printed : 5/25/2013 1:20:37 PM

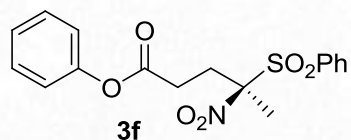


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
2	UNKNOWN	61.242	92.10	176430.2	380455.0	92.102
1	UNKNOWN	70.333	7.90	16185.6	33484.3	7.898
Total			100.00	182615.9	423939.3	100.000

Fig S26. HPLC Profile of Enantioenriched 3e

NAME INN-4-KSB-33A-1H  
EXPNO 2  
PROCNO 1  
Date\_ 20130223  
Time\_ 18.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 293.7 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

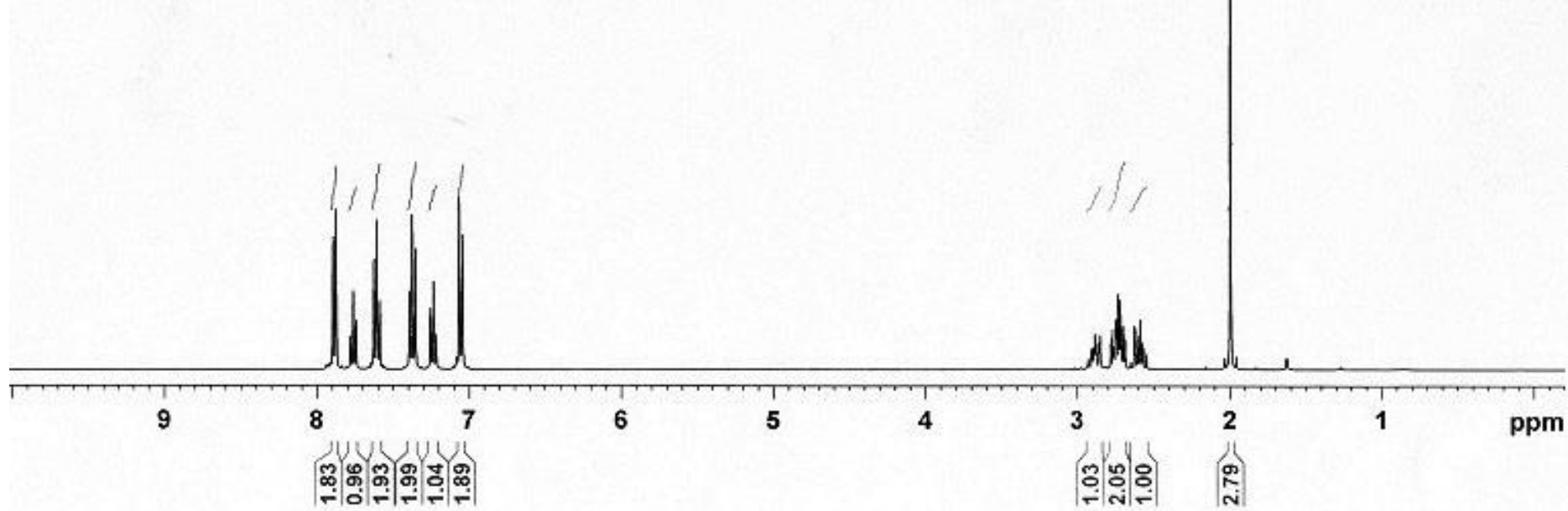


Fig S27. <sup>1</sup>H NMR Spectrum of 3f

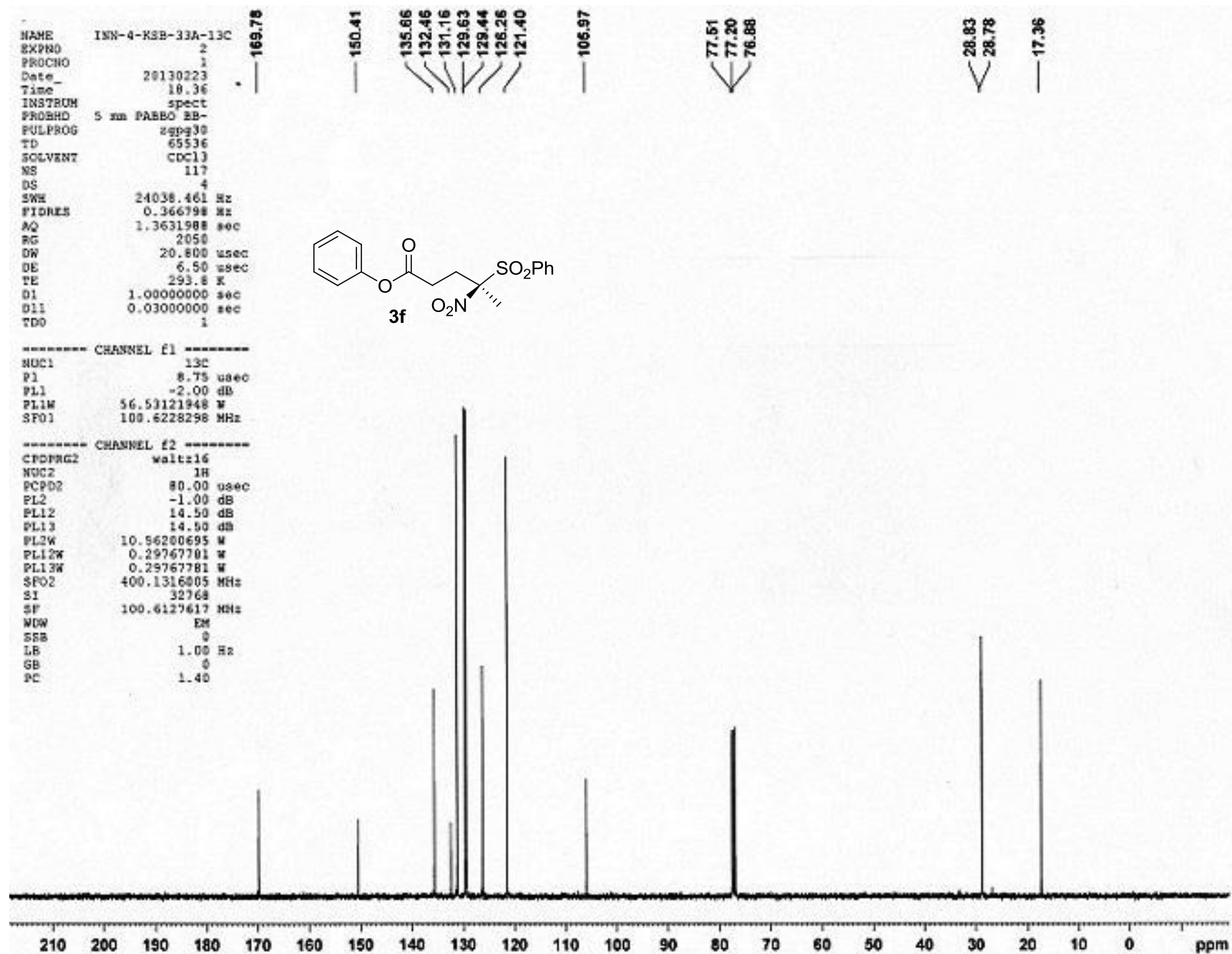
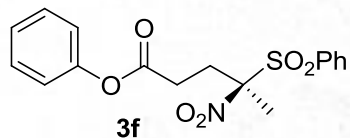


Fig S28. <sup>13</sup>C NMR Spectrum of 3f

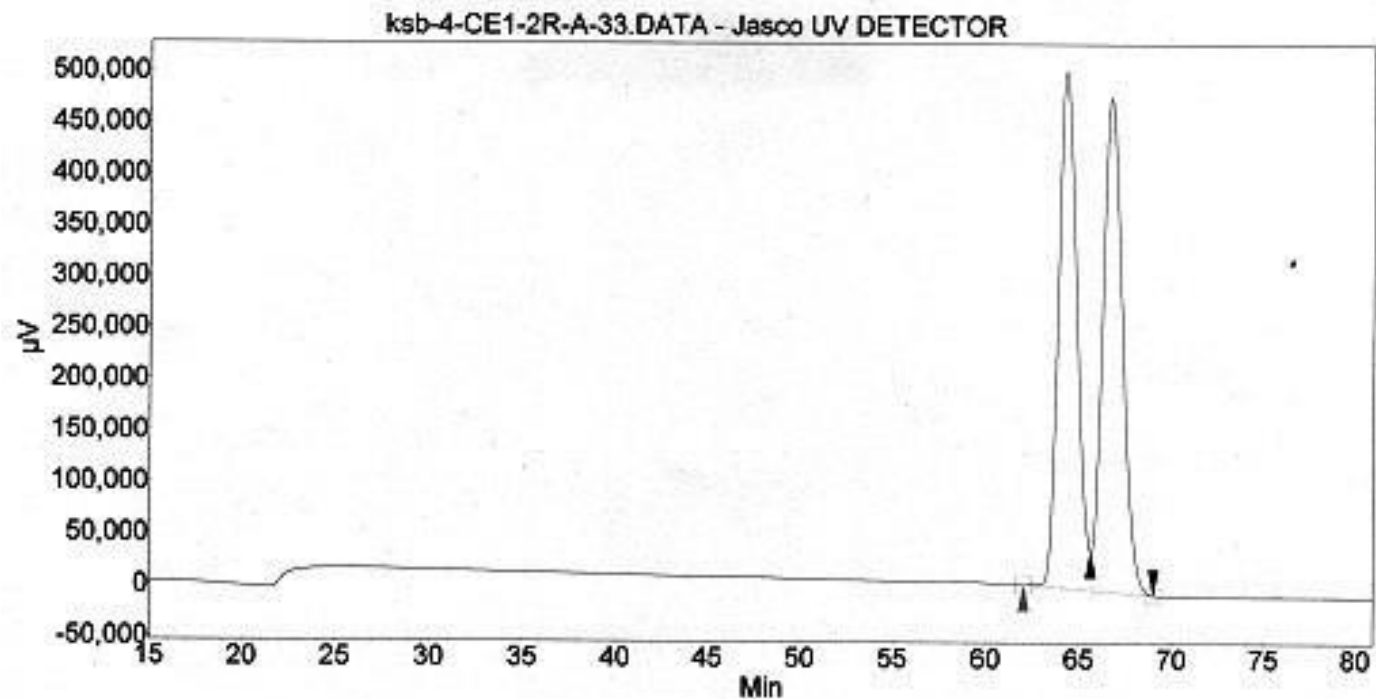


# Chromatogram : ksb-4-CE1-2R-A-33\_channel2

System : JASCO HPLC  
Method : ksb-5-05f-258  
User : User1



Acquired : 5/30/2013 1:52:06 PM  
Processed : 5/30/2013 4:10:58 PM  
Printed : 5/30/2013 4:12:22 PM



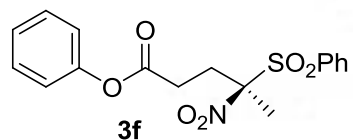
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	64.225	49.31	502032.8	575801.8	49.308
2	UNKNOWN	66.642	50.69	480330.3	591766.9	50.692
Total			100.00	982363.2	1167368.7	100.000

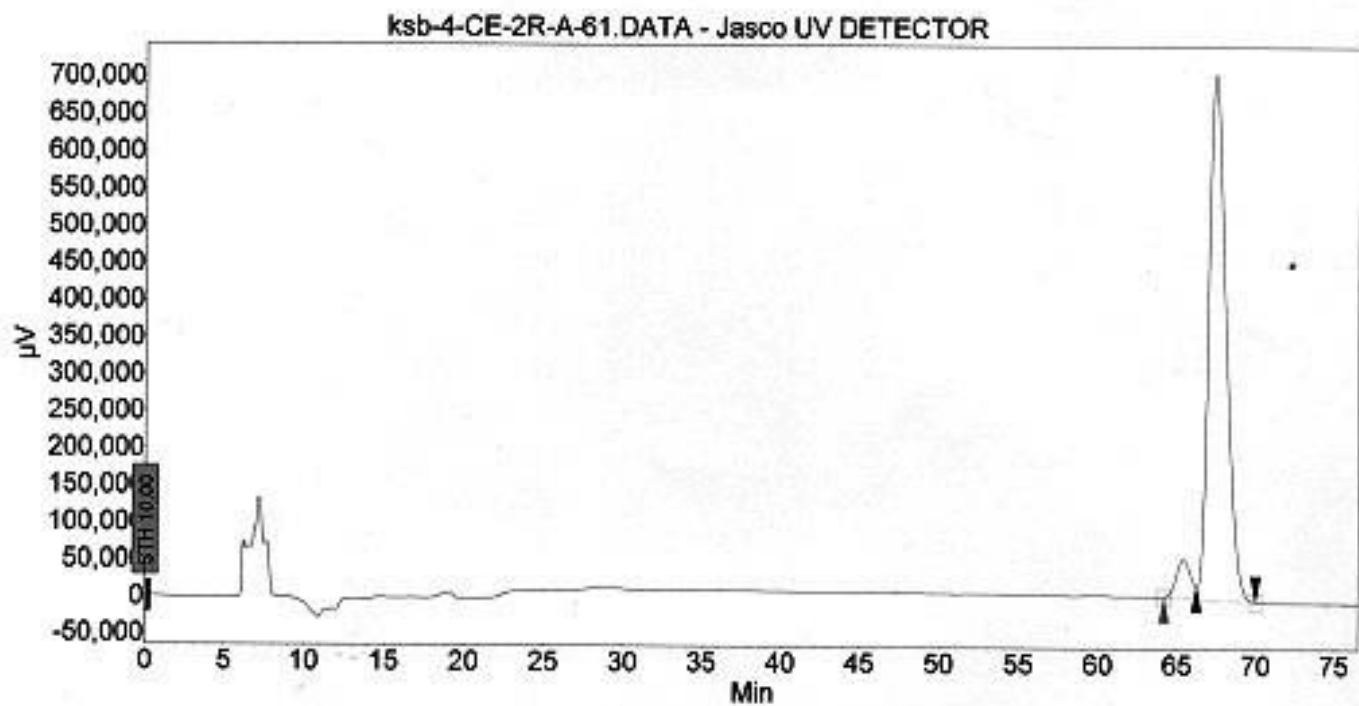
Fig S29. HPLC Profile of Racemic 3f

## Chromatogram : ksb-4-CE-2R-A-61\_channel2

System : JASCO HPLC  
Method : ksb-5-054-258  
User : User1



Acquired : 5/30/2013 4:35:20 PM  
Processed : 5/30/2013 5:52:09 PM  
Printed : 5/30/2013 5:58:20 PM



### Peak results :

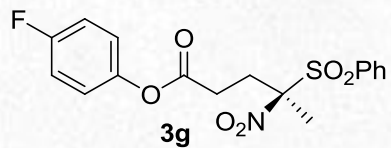
Index	Name	Time (Min)	Quantity (% Area)	Height [µV]	Area [µV.Min]	Area % (%)
1	UNKNOWN	65.358	5.71	51790.2	54436.8	5.710
2	UNKNOWN	67.358	94.29	705410.2	888949.0	94.290
Total			100.00	757200.4	953385.9	100.000

Fig S30. HPLC Profile of Enantioenriched 3f

```

NAME      INN-4-KSB-42B-1H
EXPNO     13
PROCNO    1
Date_     20130314
Time      8.01
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        13
DS        0
SWH       8223.685 Hz
FIDRES    0.125463 Hz
AQ        3.9846367 sec
RG        32
DW        60.800 usec
DE        6.50 usec
TE        292.1 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

```

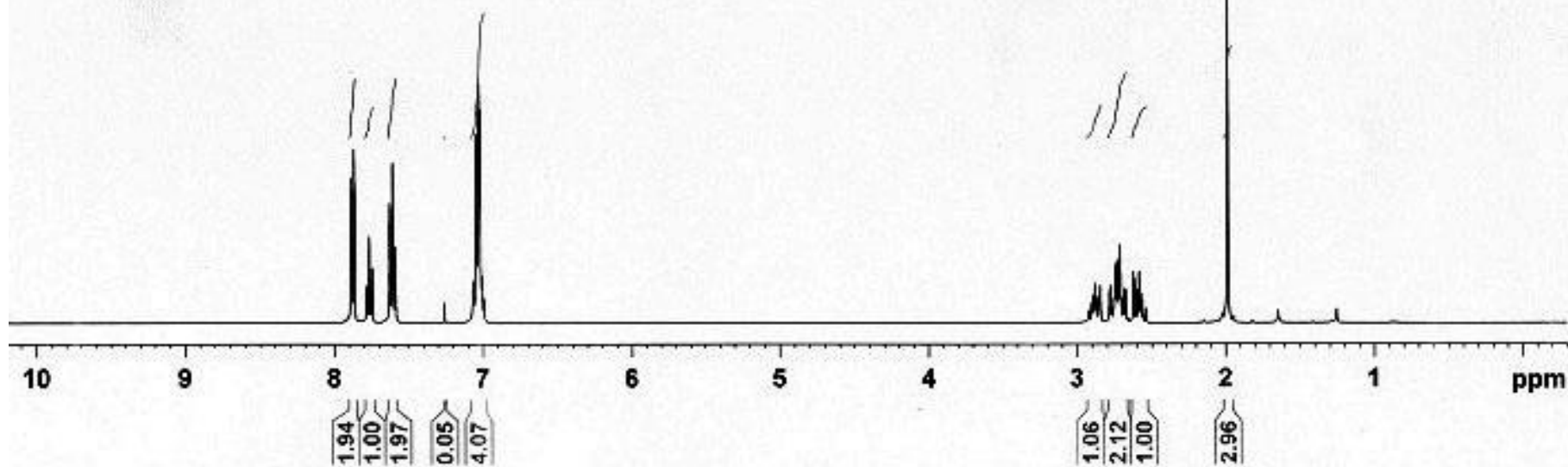


Fig S31. <sup>1</sup>H NMR Spectrum of 3g

```

NAME      INN-4-KSB-42B-13C
EXPNO     14
PROCNO    1
Date_     20130314
Time      8.05
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         100
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         2050
DM         20.800 usec
DE         6.50 usec
TE         292.8 K
D1         1.00000000 sec
D11        0.03000000 sec
TDO        1

```

```

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      56.53121940 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         100.6127605 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
EC         1.40

```

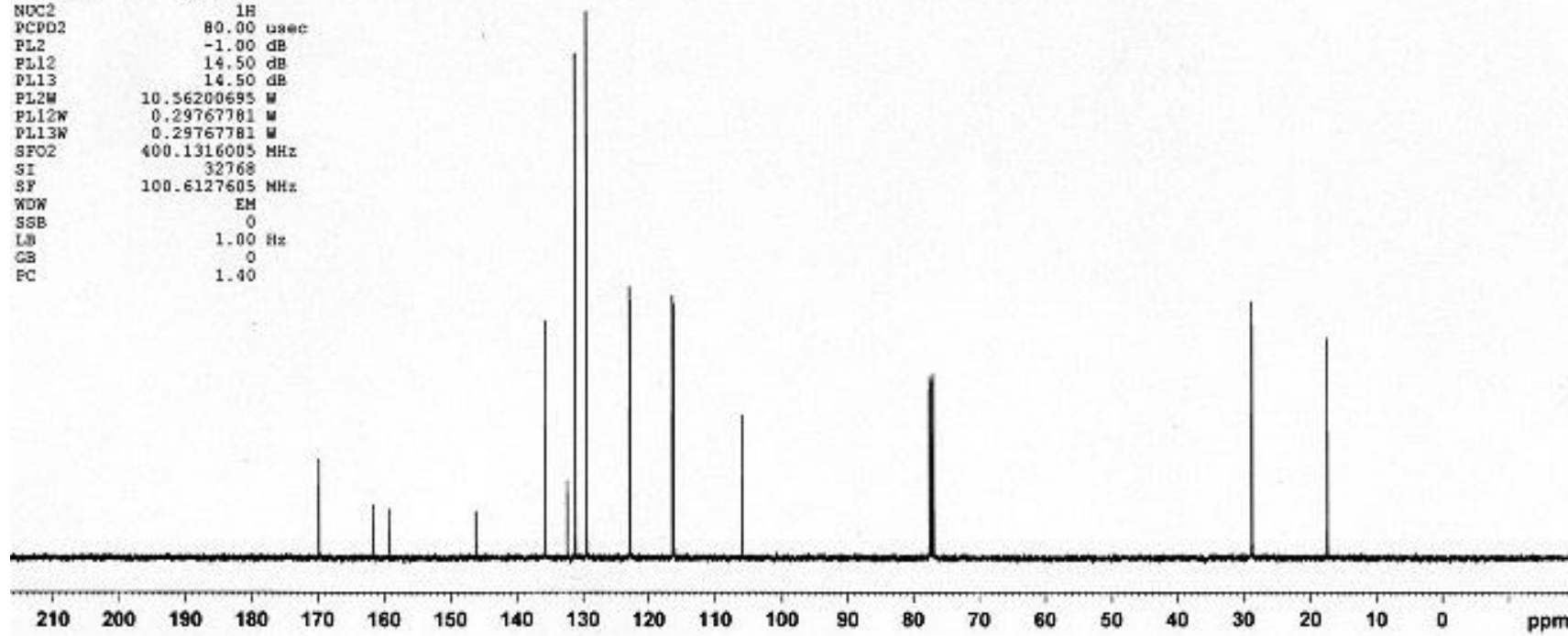
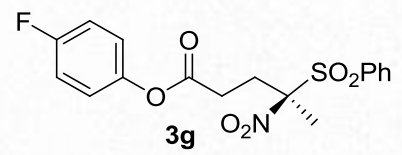


Fig S32. <sup>13</sup>C NMR Spectrum of 3g

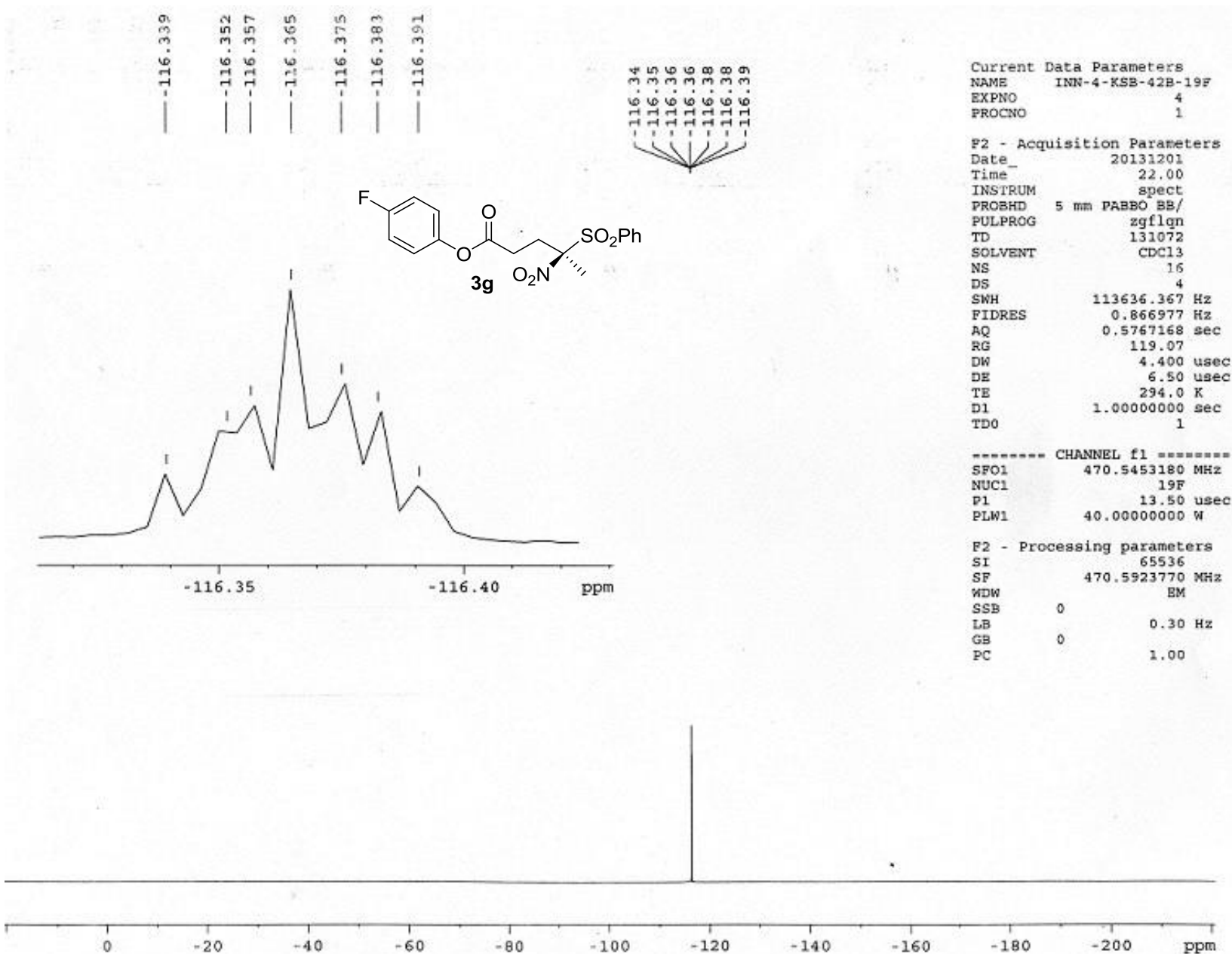
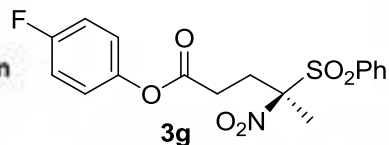


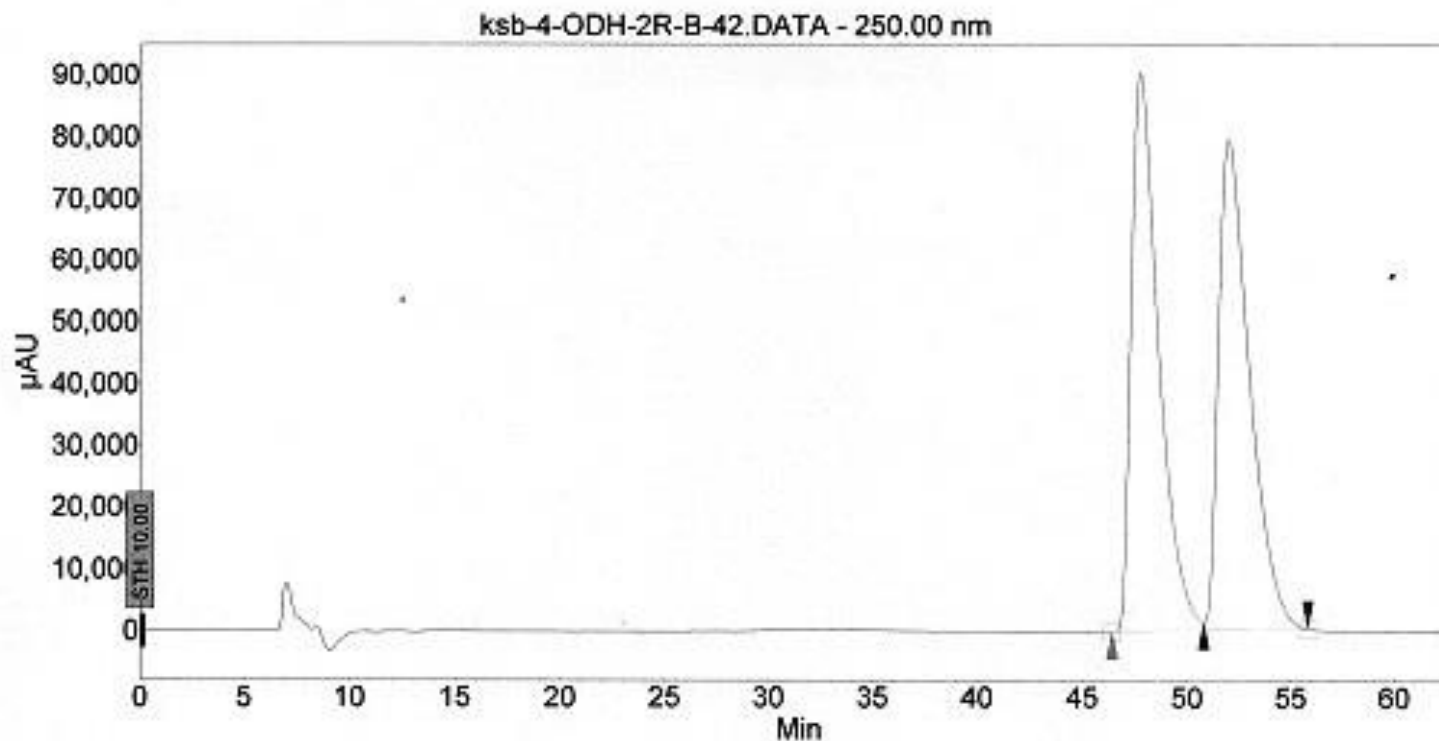
Fig S33. <sup>19</sup>F NMR Spectrum of 3g

# Chromatogram : ksb-4-ODH-2R-B-42\_channel4

System : Jasco HPLC  
 Method : ksb-10%-05f-250nm  
 User : User1



Acquired : 5/16/2013 4:03:38 PM  
 Processed : 5/16/2013 5:08:25 PM  
 Printed : 5/16/2013 5:08:31 PM



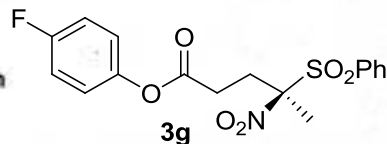
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	47.730	49.91	90311.0	137059.7	49.912
2	UNKNOWN	51.956	50.09	79491.6	137543.5	50.088
Total			100.00	169802.6	274603.2	100.000

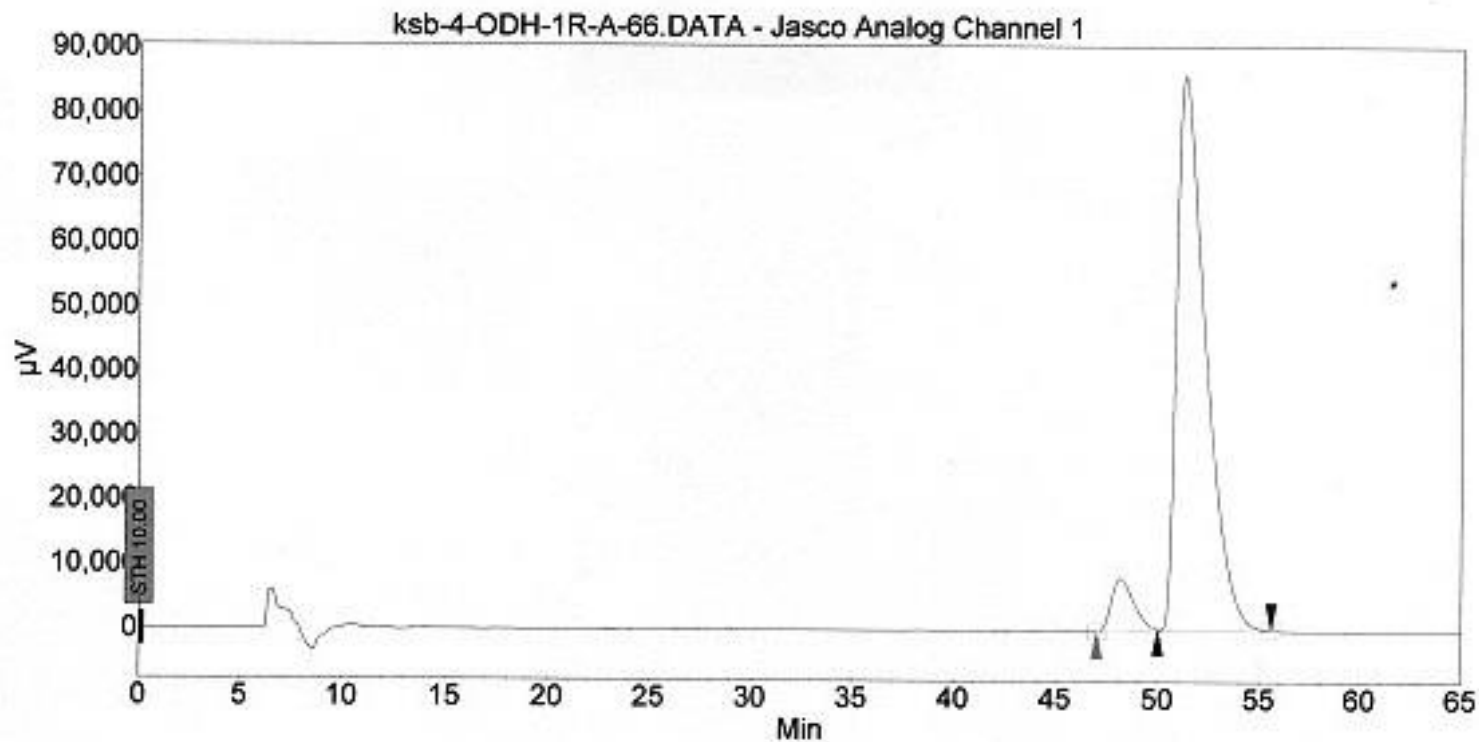
Fig S34. HPLC Profile of Racemic 3g

**Chromatogram : ksb-4-ODH-1R-A-66\_channel2**

System : Jasco HPLC  
 Method : ksb-10%-05f-250nm  
 User : User1



Acquired : 5/16/2013 2:37:35 PM  
 Processed : 5/16/2013 4:05:28 PM  
 Printed : 5/16/2013 4:06:37 PM



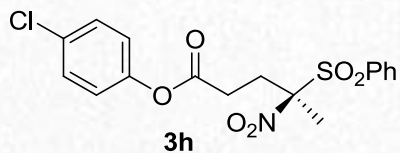
**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	48.158	6.80	8104.1	10416.2	6.804
2	UNKNOWN	51.283	93.20	85829.6	142666.3	93.196
Total			100.00	93933.6	153082.5	100.000

Fig S35. HPLC Profile of Enantioenriched 3g

```

NAME      INN-4-KSB-31A-1H
EXPNO     1
PROCNO    1
Date_     20130219
Time      20.53
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDC13
NS        16
DS        0
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        32
DW        60.800 usec
DE        6.50 usec
TE        294.3 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
    
```

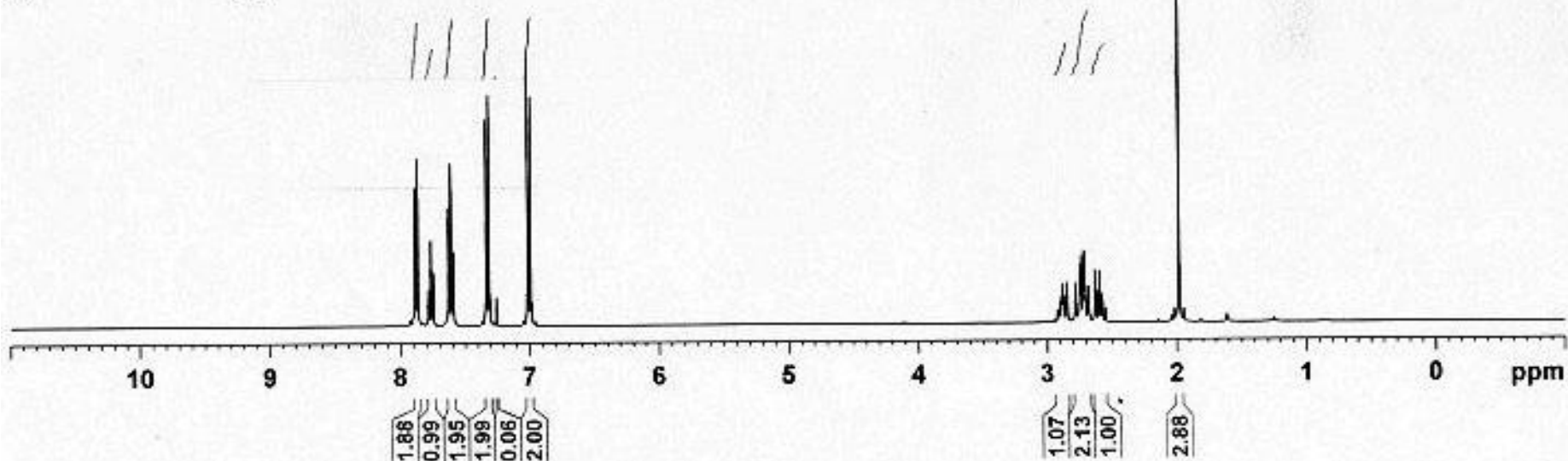




Fig S36. <sup>1</sup>H NMR Spectrum of 3h

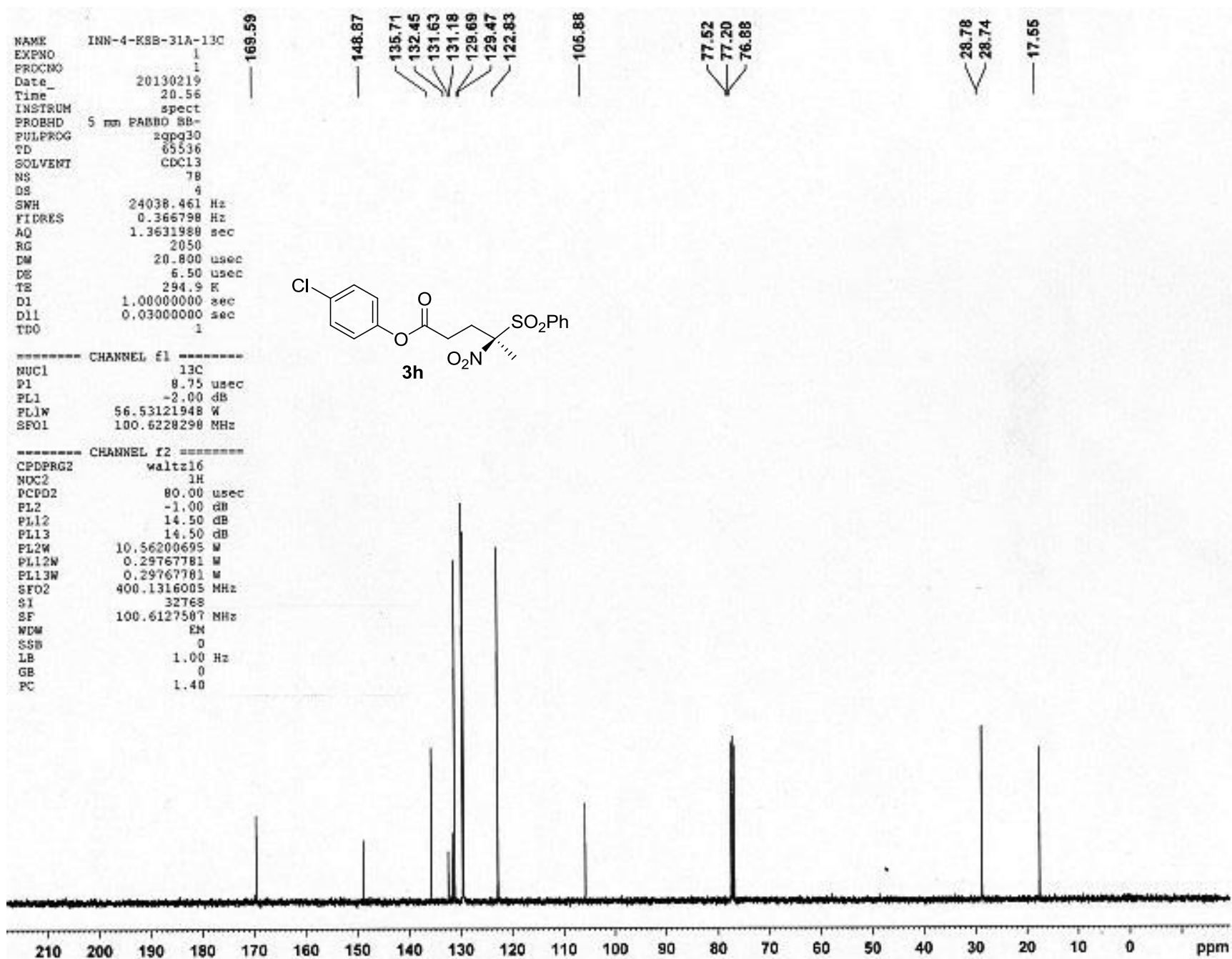
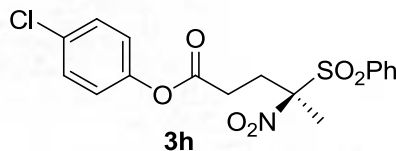


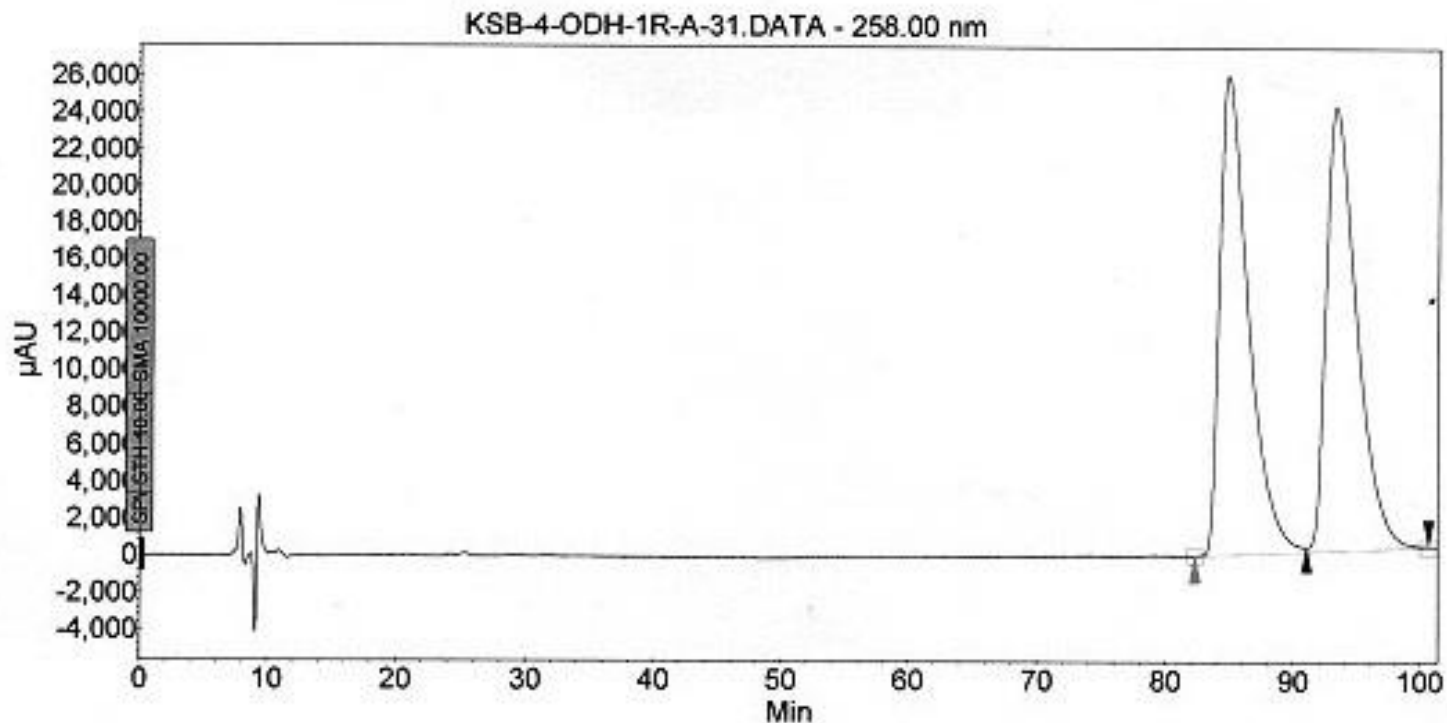
Fig S37. <sup>13</sup>C NMR Spectrum of 3h

Chromatogram : KSB-4-ODH-1R-A-31\_channel5

System : JASCO HPLC  
Method : ksb-5-05f-218  
User : User1



Acquired : 2/27/2013 12:03:55 PM  
Processed : 2/27/2013 1:48:15 PM  
Printed : 2/27/2013 8:47:50 PM



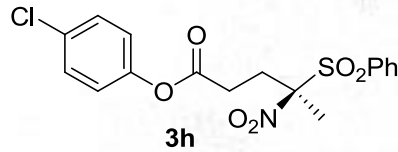
Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	84.836	49.99	25990.2	67368.9	49.989
2	UNKNOWN	93.287	50.01	24081.6	67398.6	50.011
Total			100.00	50071.9	134767.5	100.000

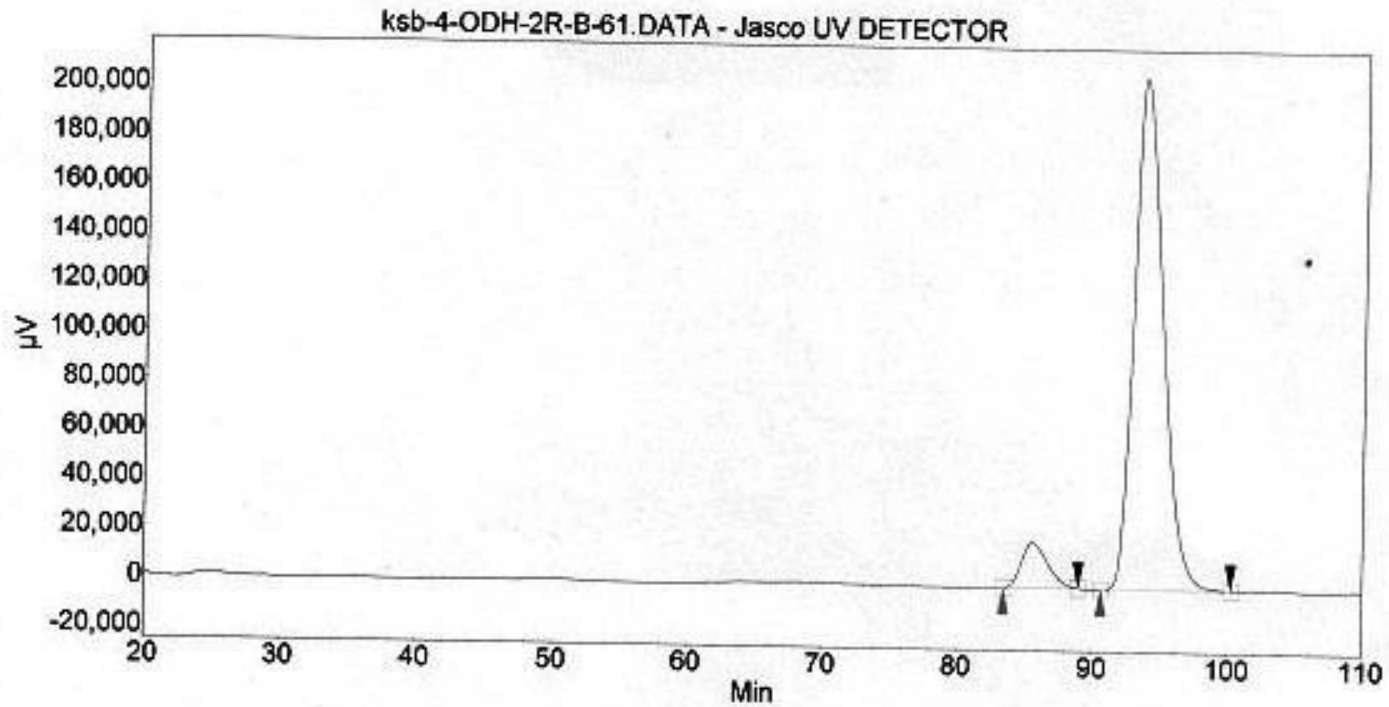
Fig S38. HPLC Profile of Racemic 3h

Chromatogram : ksb-4-ODH-2R-B-61\_channel2

System : JASCO HPLC  
 Method : ksb-5-05f-258  
 User : User1



Acquired : 5/6/2013 1:12:44 PM  
 Processed : 5/6/2013 3:12:49 PM  
 Printed : 5/6/2013 10:12:23 PM



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	85.542	7.34	18254.2	40654.9	7.344
2	UNKNOWN	93.587	92.66	206813.3	515430.1	92.656
Total			100.00	225067.5	556085.1	100.000

Fig S39. HPLC Profile of Enantioenriched 3h

```

NAME      INN-4-XSB-42A-1H
EXPNO     11
PROCNO    1
Date_     20130314
Time      7.52
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        101
DM        60.800 usec
DE        6.50 usec
TE        292.1 K
D1        1.0000000 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
    
```

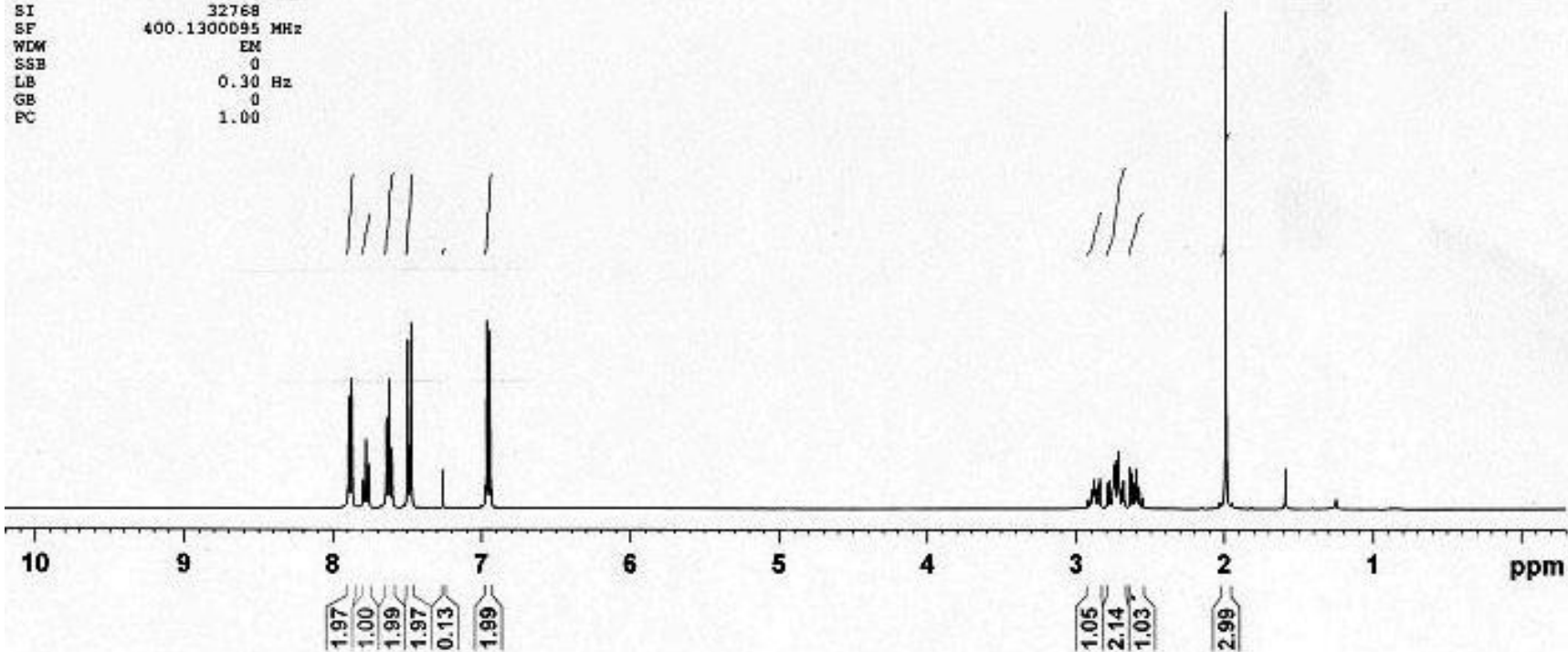


Fig S40. <sup>1</sup>H NMR Spectrum of 3i

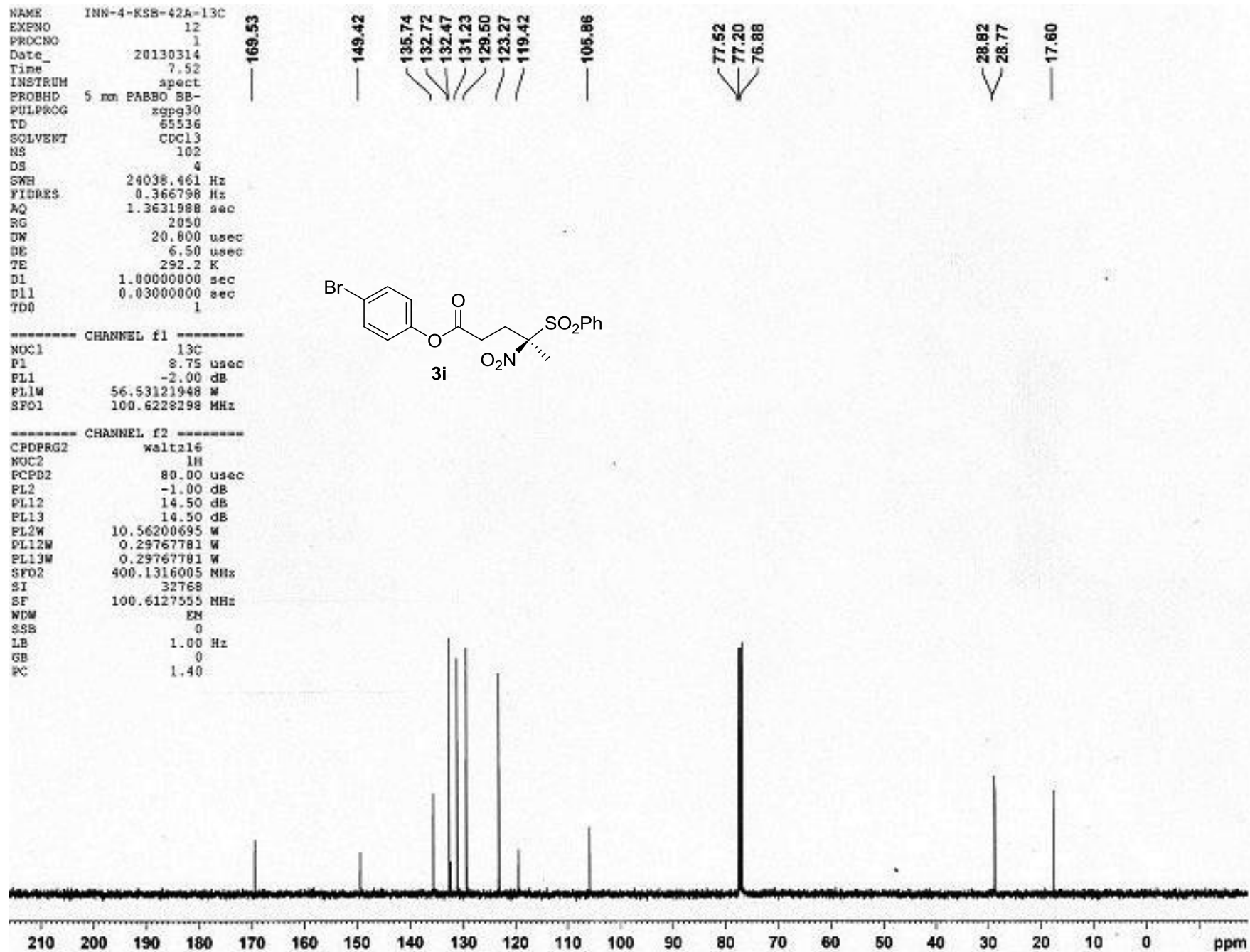


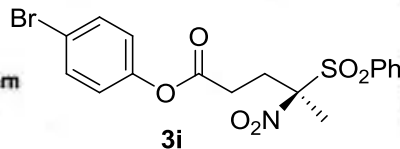
Fig S41. <sup>13</sup>C NMR Spectrum of 3i

Chromatogram : ksb-4-ODH-1R-A-42\_channel4

System : Jasco HPLC

Method : ksb-10%-05f-250nm

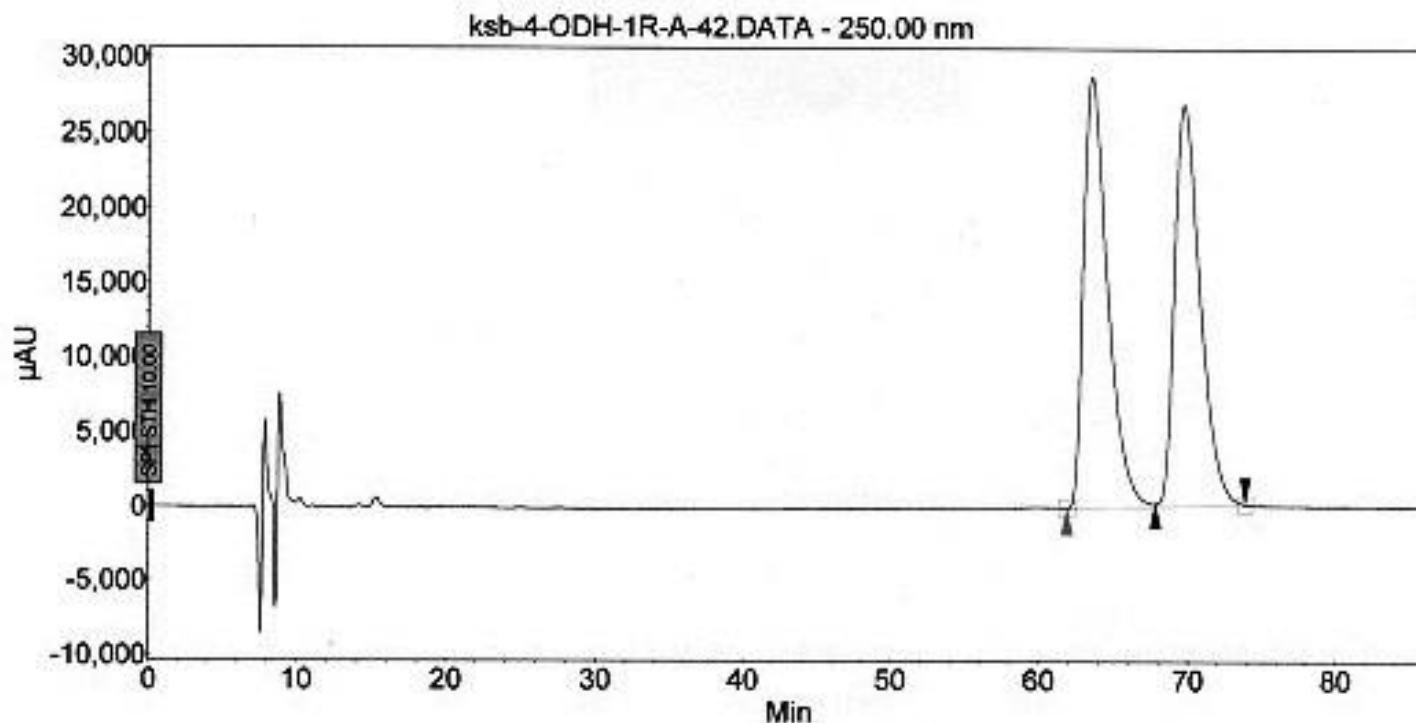
User : User1



Acquired : 3/14/2013 3:01:18 PM

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Printed : 3/14/2013 4:38:14 PM

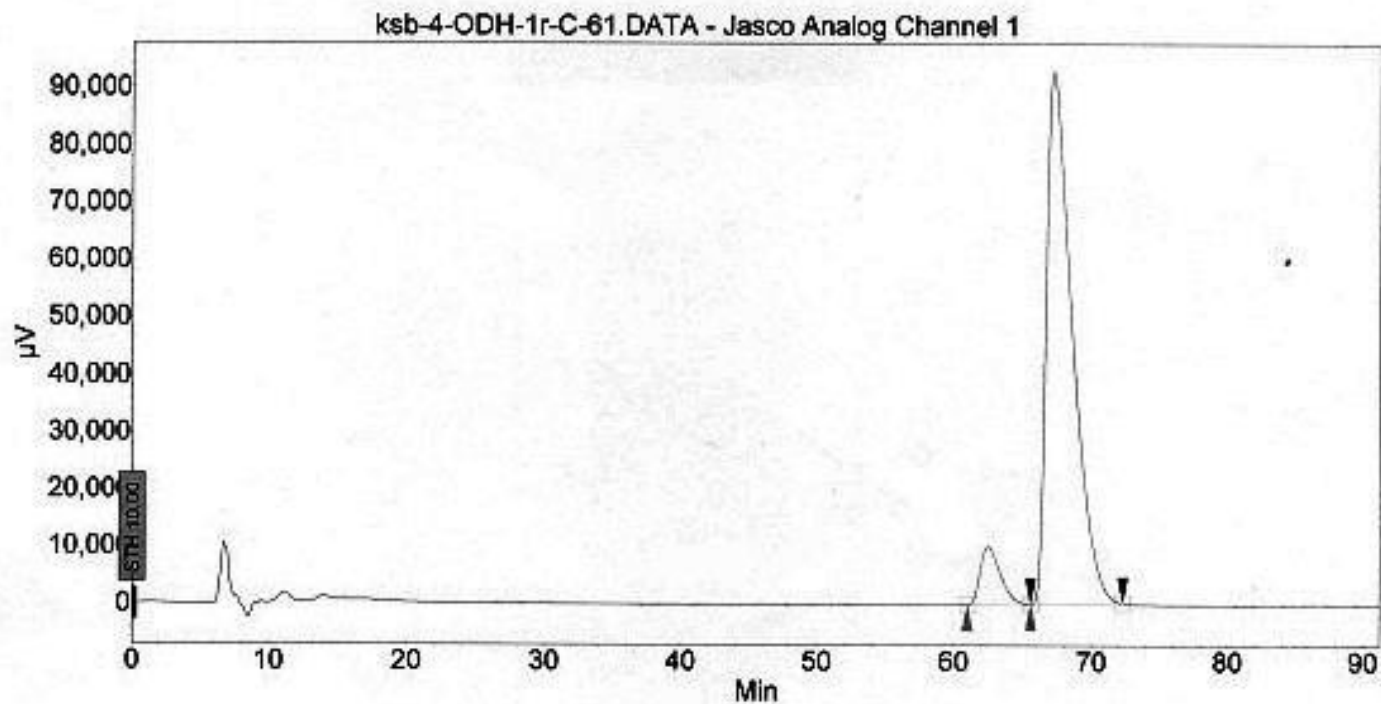
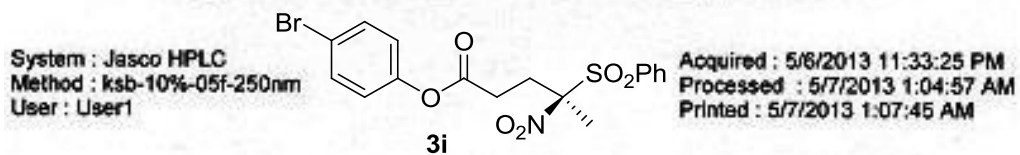


Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [AU]	Area [AU.Min]	Area % [%]
1	UNKNOWN	63.497	50.11	28796.3	53967.4	50.113
2	UNKNOWN	69.695	49.89	26793.6	53723.4	49.887
Total			100.00	55589.9	107690.8	100.000

Fig S42. HPLC Profile of Racemic 3i

Chromatogram : ksb-4-ODH-1r-C-61\_channel2



Peak results :

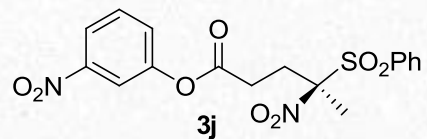
Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV·Min]	Area % [%]
1	UNKNOWN	62.475	7.78	10144.3	17056.2	7.779
2	UNKNOWN	67.167	92.22	92896.8	202203.5	92.221
Total			100.00	103041.1	219259.7	100.000

Fig S43. HPLC Profile of Enantioenriched 3i

```

NAME      INN-4-KSB-31C-1H
EXPNO     1
PROCNO    1
Date_     20130219
Time      21.09
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
ID        65536
SOLVENT   CDCl3
NS        16
DS        0
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        101
DN        60.800 usec
DE        6.50 usec
TE        294.3 K
D1        1.00000000 sec
TD0       1

```



```

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

```

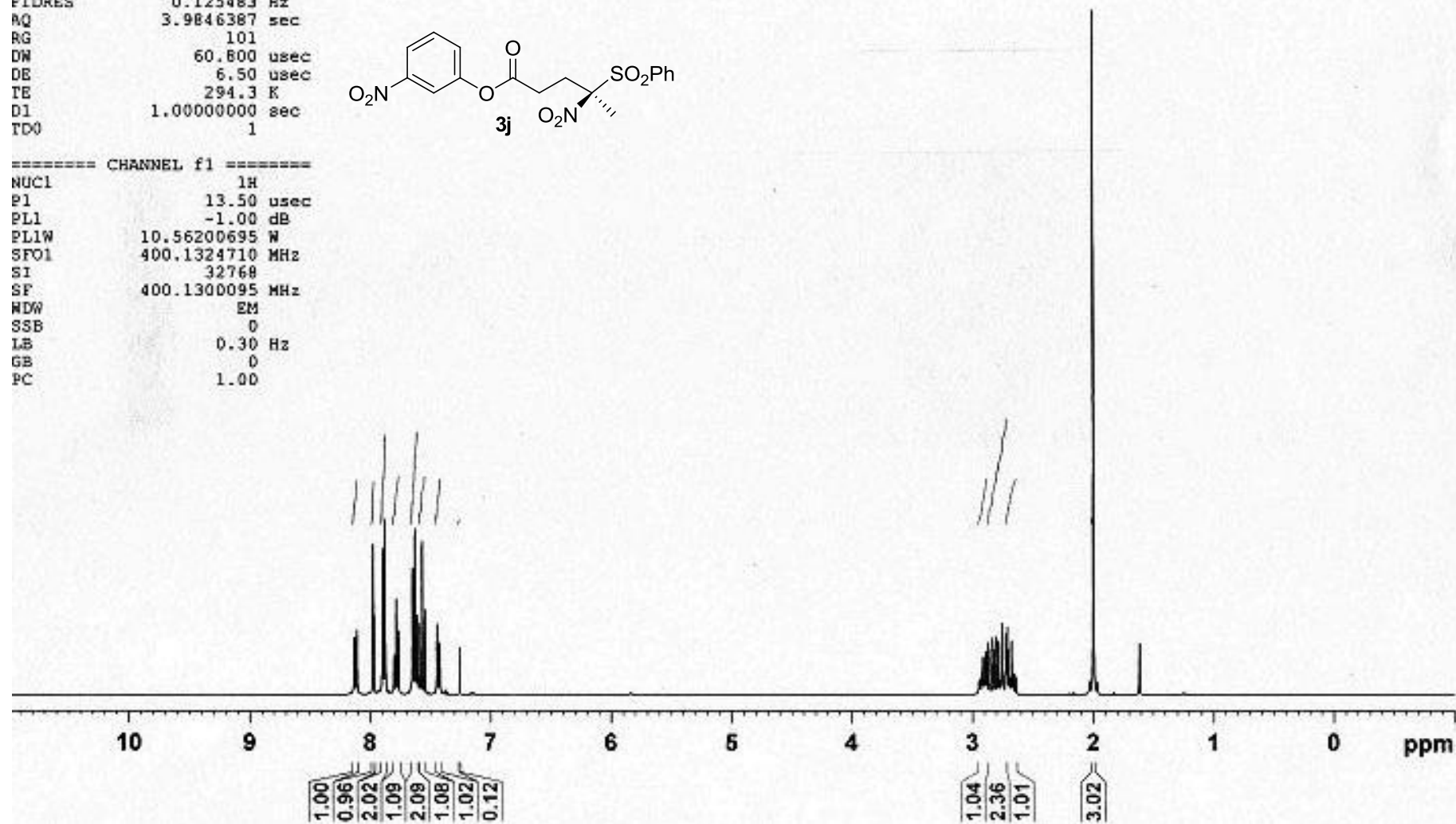


Fig S44. <sup>1</sup>H NMR Spectrum of 3j



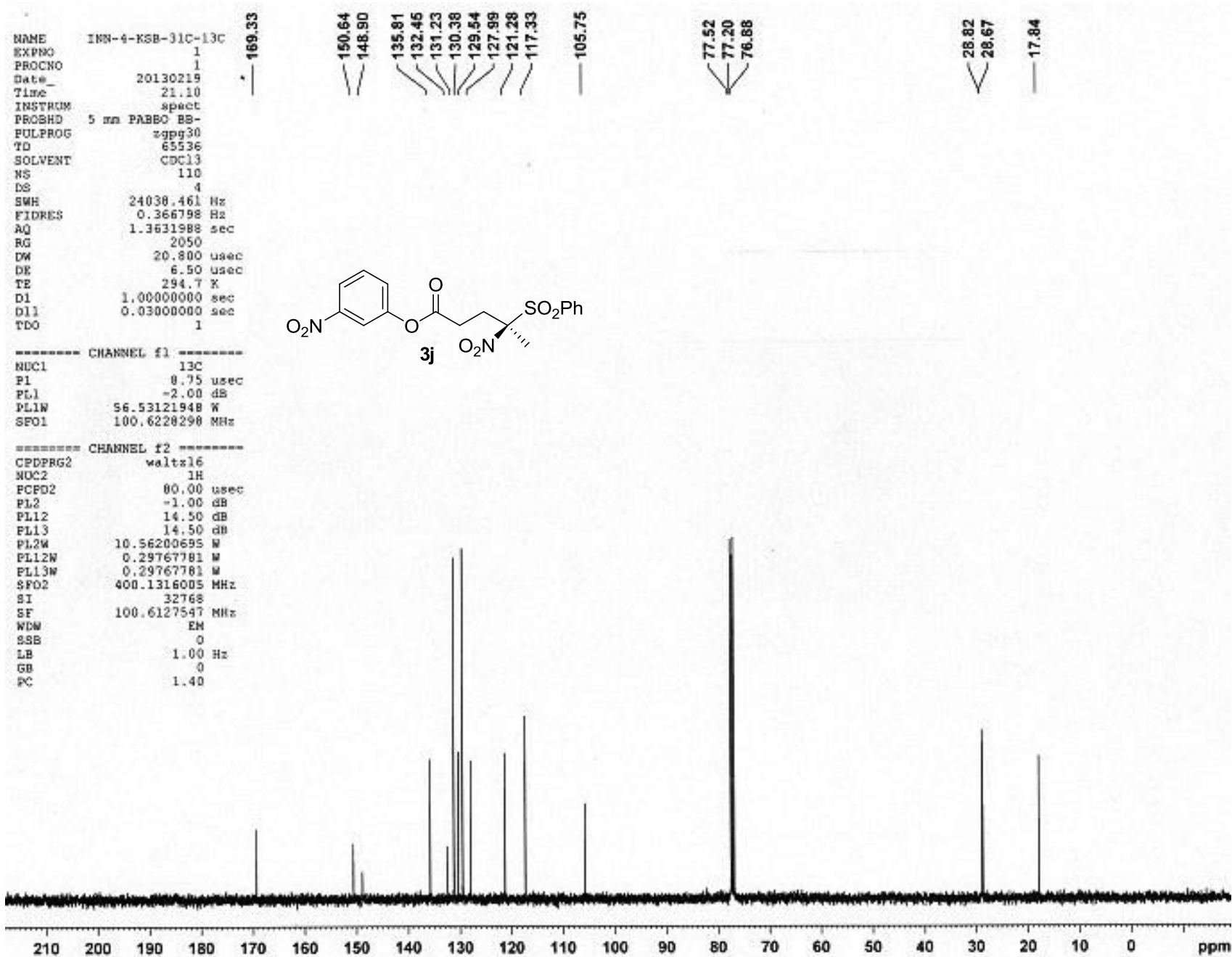
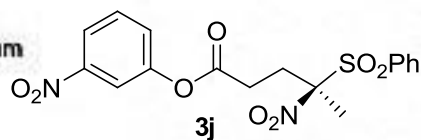


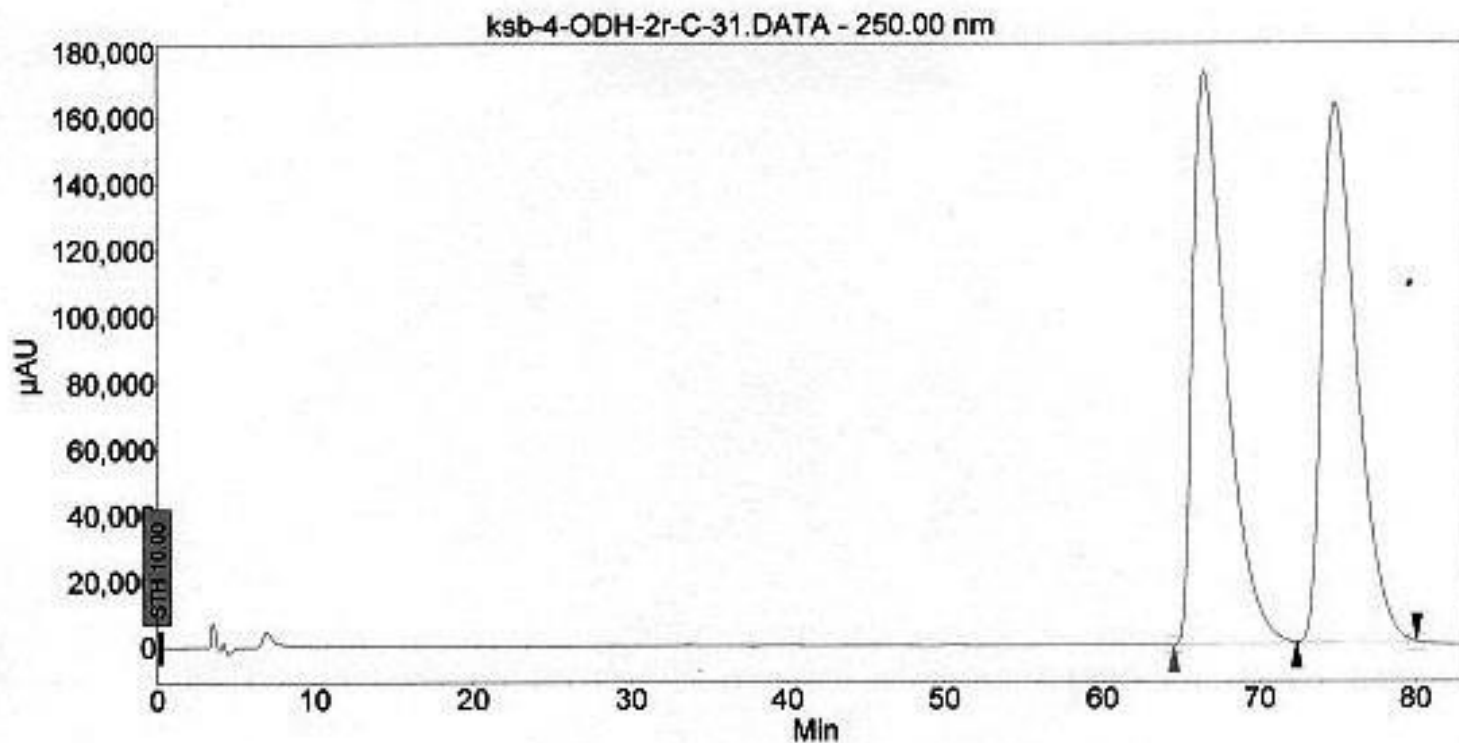
Fig S45. <sup>13</sup>C NMR Spectrum of 3j

# Chromatogram : ksb-4-ODH-2r-C-31\_channel4

System : Jasco HPLC  
 Method : ksb-10%-1f-250nm  
 User : User1



Acquired : 5/11/2013 12:46:37 PM  
 Processed : 5/11/2013 2:10:50 PM  
 Printed : 5/11/2013 4:01:59 PM



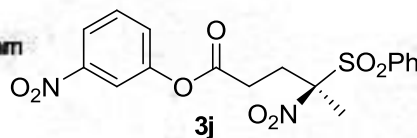
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	66.483	50.00	173161.1	421560.8	50.004
2	UNKNOWN	74.880	50.00	163182.2	421497.9	49.996
Total			100.00	336343.4	843058.6	100.000

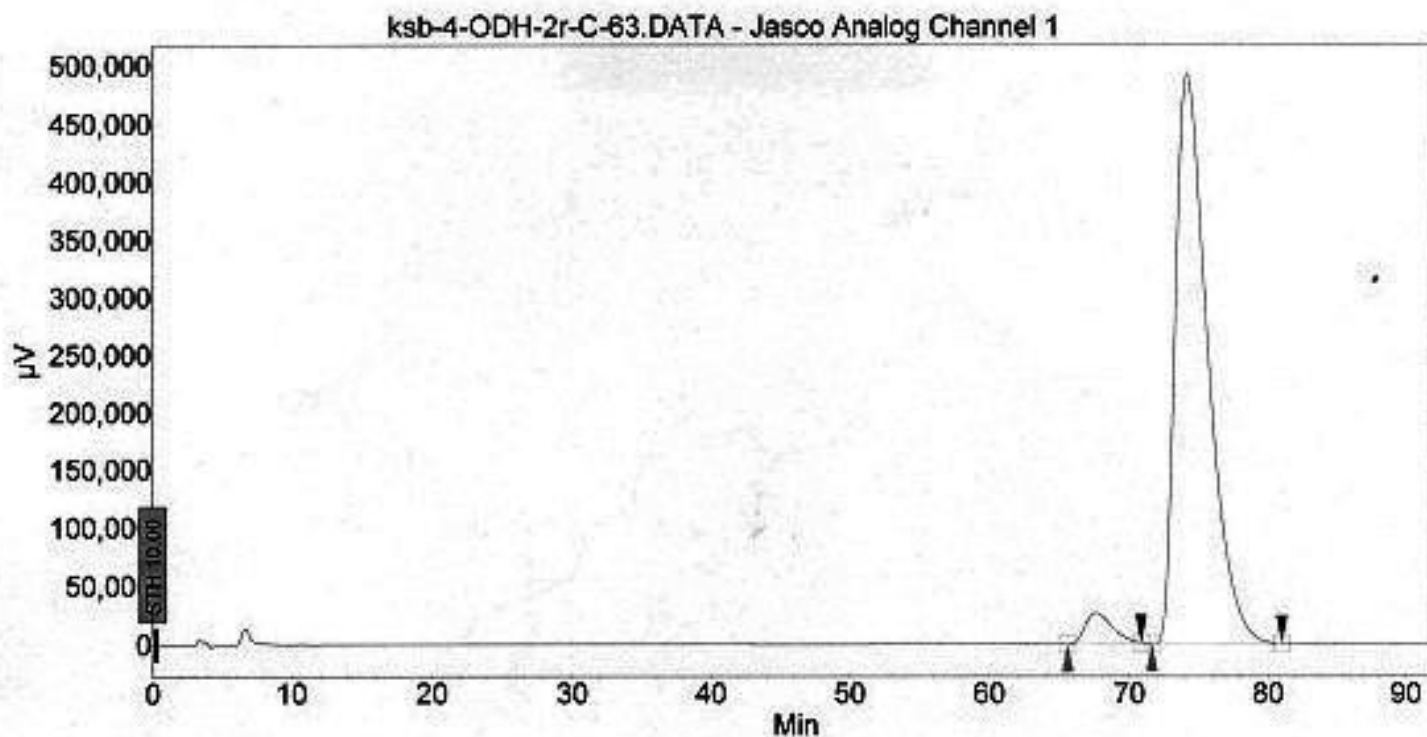
Fig S46. HPLC Profile of Racemic 3j

# Chromatogram : ksb-4-ODH-2r-C-63\_channel2

System : Jasco HPLC  
Method : ksb-10%-1f-250nm  
User : User1



Acquired : 5/11/2013 2:11:41 PM  
Processed : 5/11/2013 4:03:31 PM  
Printed : 5/11/2013 4:04:21 PM

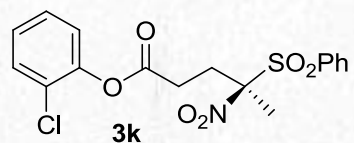


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	67.600	4.18	26166.7	58104.8	4.179
2	UNKNOWN	74.167	95.82	492198.1	1332327.3	95.821
Total			100.00	518364.8	1390432.0	100.000

Fig S47. HPLC Profile of Enantioenriched 3j

NAME INN-4-KSB-33B-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130223  
 Time 18.45  
 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 293.8 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

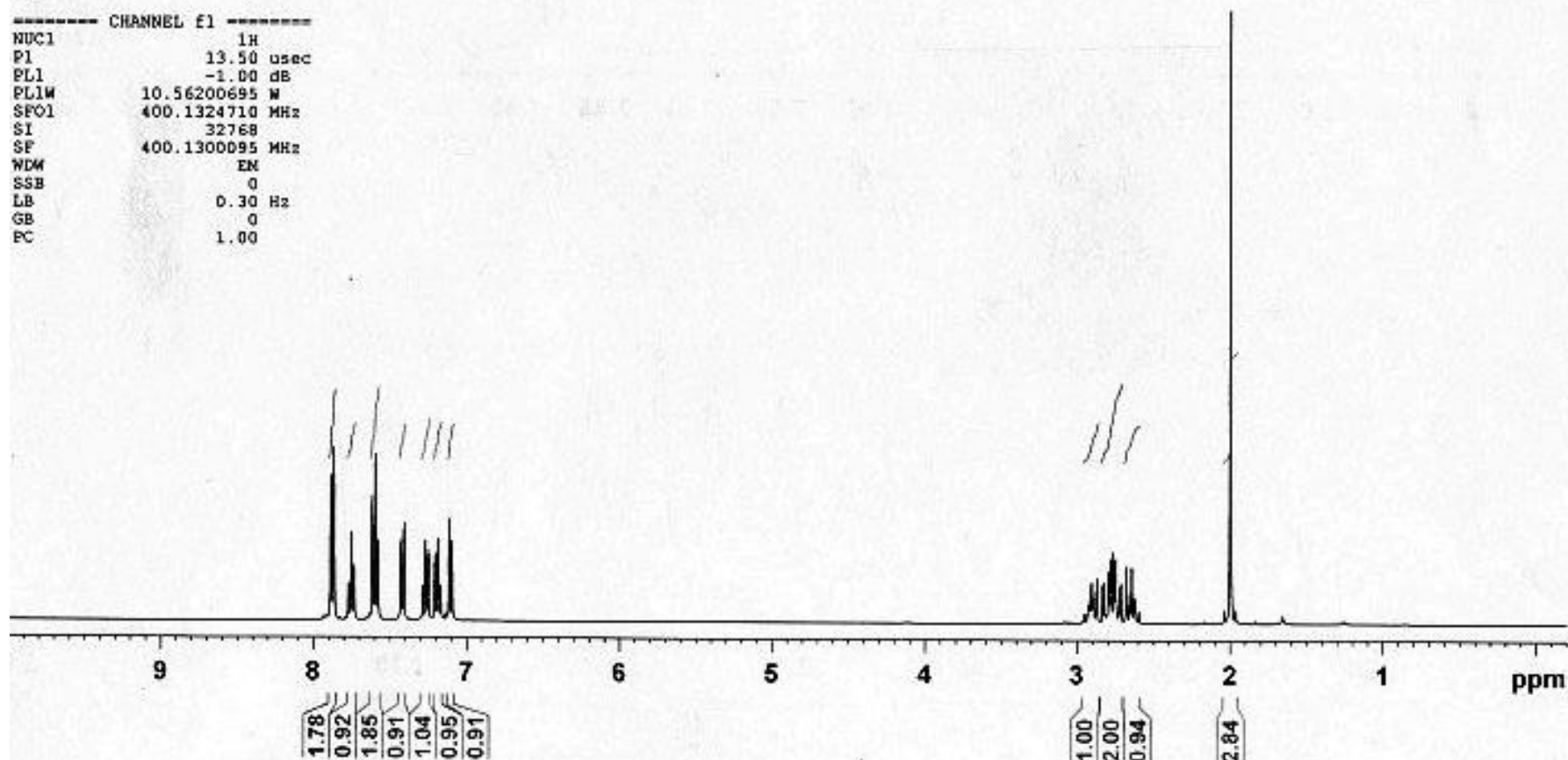


Fig S48. <sup>1</sup>H NMR Spectrum of 3k

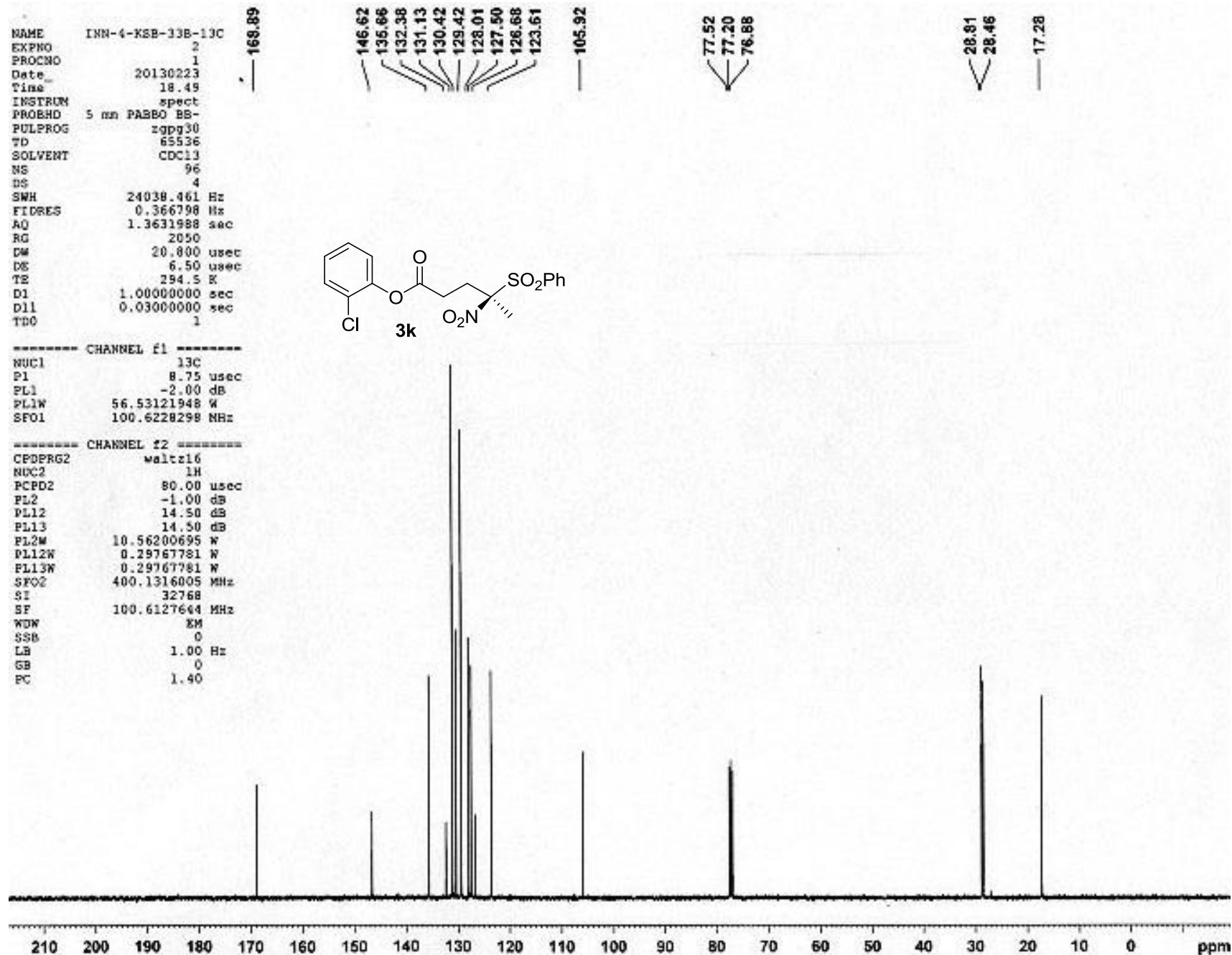
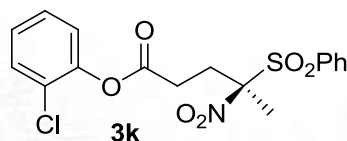


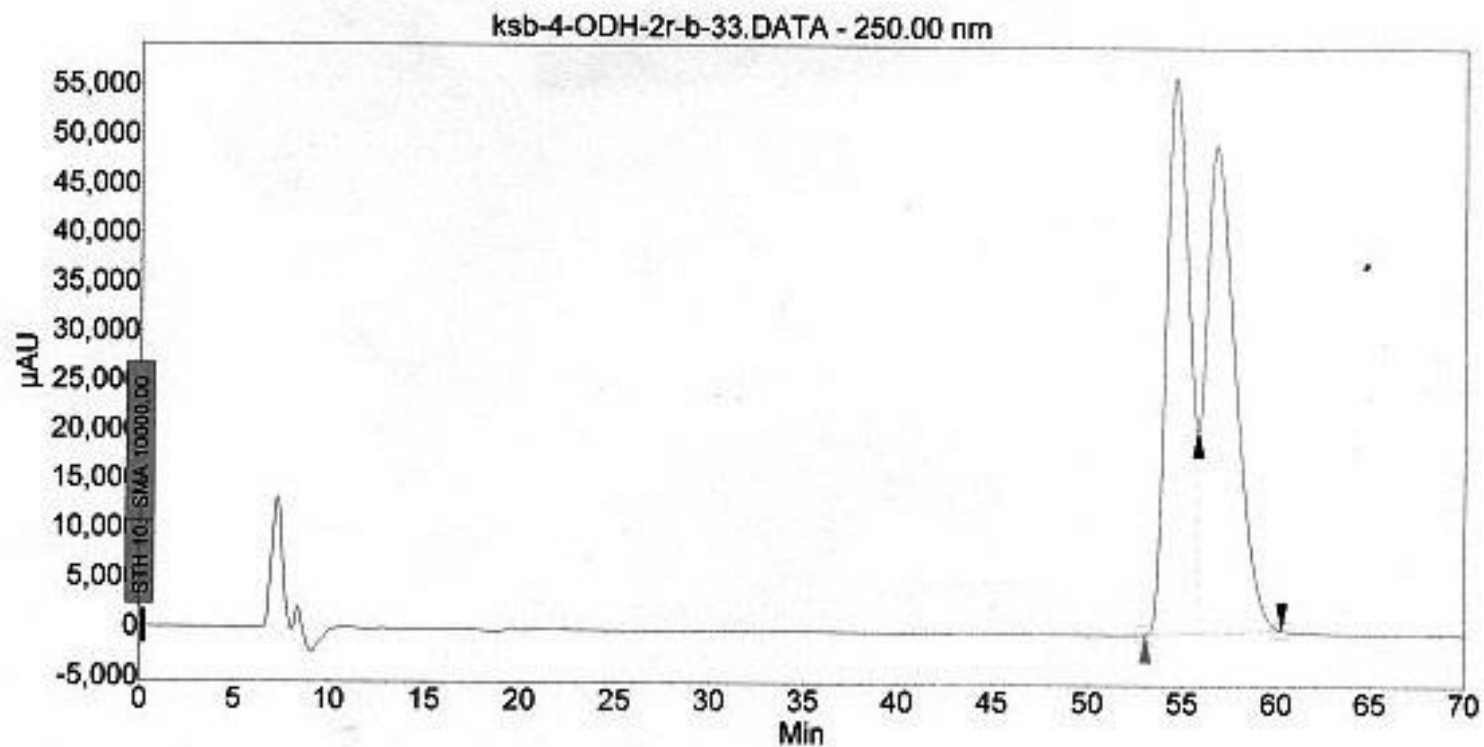
Fig S49. <sup>13</sup>C NMR Spectrum of 3k

# Chromatogram : ksb-4-ODH-2r-b-33\_channel4

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/6/2013 8:13:27 PM  
Processed : 5/6/2013 10:05:41 PM  
Printed : 5/8/2013 10:06:16 PM



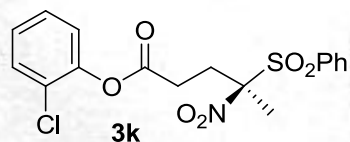
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [AU]	Area [AU.Min]	Area % [%]
1	UNKNOWN	54.540	46.34	56319.2	78928.5	46.342
2	UNKNOWN	56.686	53.66	49482.6	91388.8	53.658
Total			100.00	105801.9	170317.3	100.000

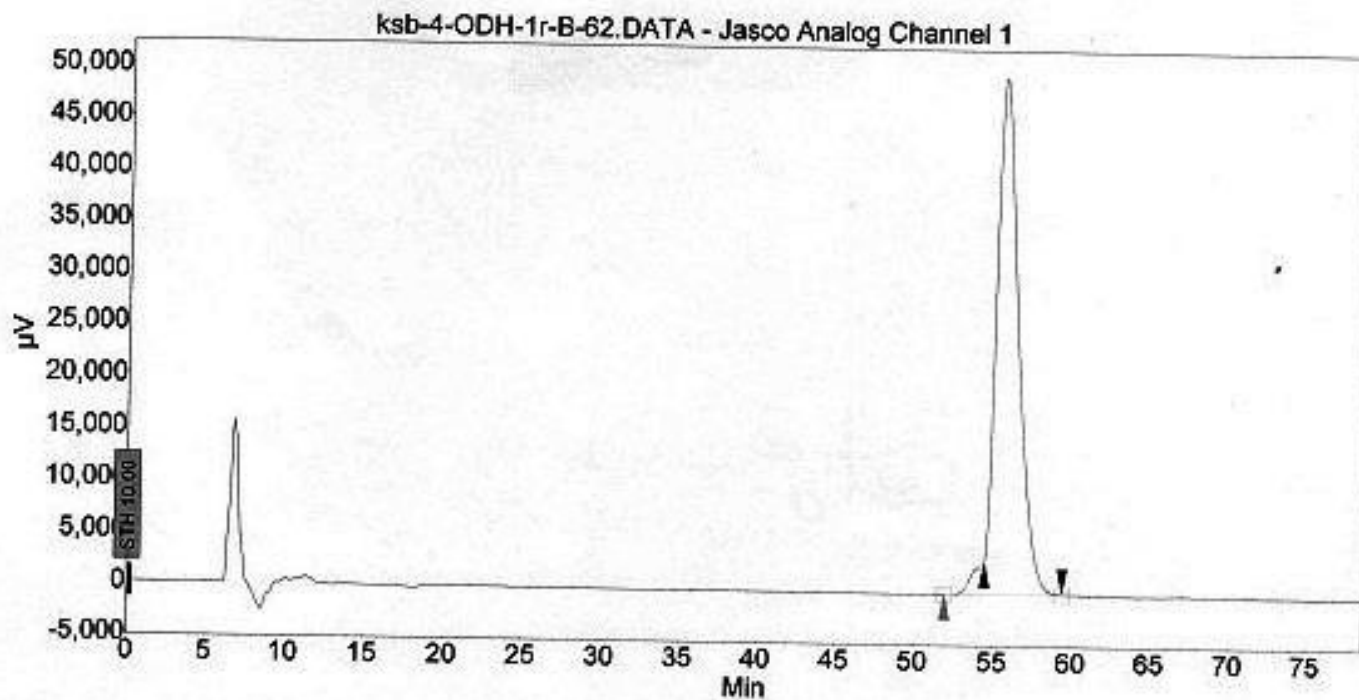
Fig S50. HPLC Profile of Racemic 3k

# Chromatogram : ksb-4-ODH-1r-B-62\_channel2

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/6/2013 5:37:37 PM  
Processed : 5/6/2013 7:28:00 PM  
Printed : 5/6/2013 10:06:42 PM

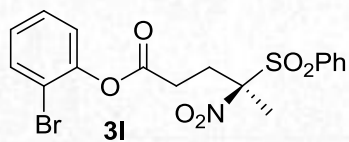


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	54.475	3.20	3106.2	2654.8	3.200
2	UNKNOWN	55.458	96.80	49871.2	80298.6	96.800
Total			100.00	52977.4	82953.4	100.000

Fig S51. HPLC Profile of Enantioenriched 3k

NAME INN-4-KSB-70B-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130523  
 Time\_ 20.25  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 11  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DN 60.800 usec  
 DE 6.50 usec  
 TE 293.9 K  
 D1 1.00000000 sec  
 TD0 1



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

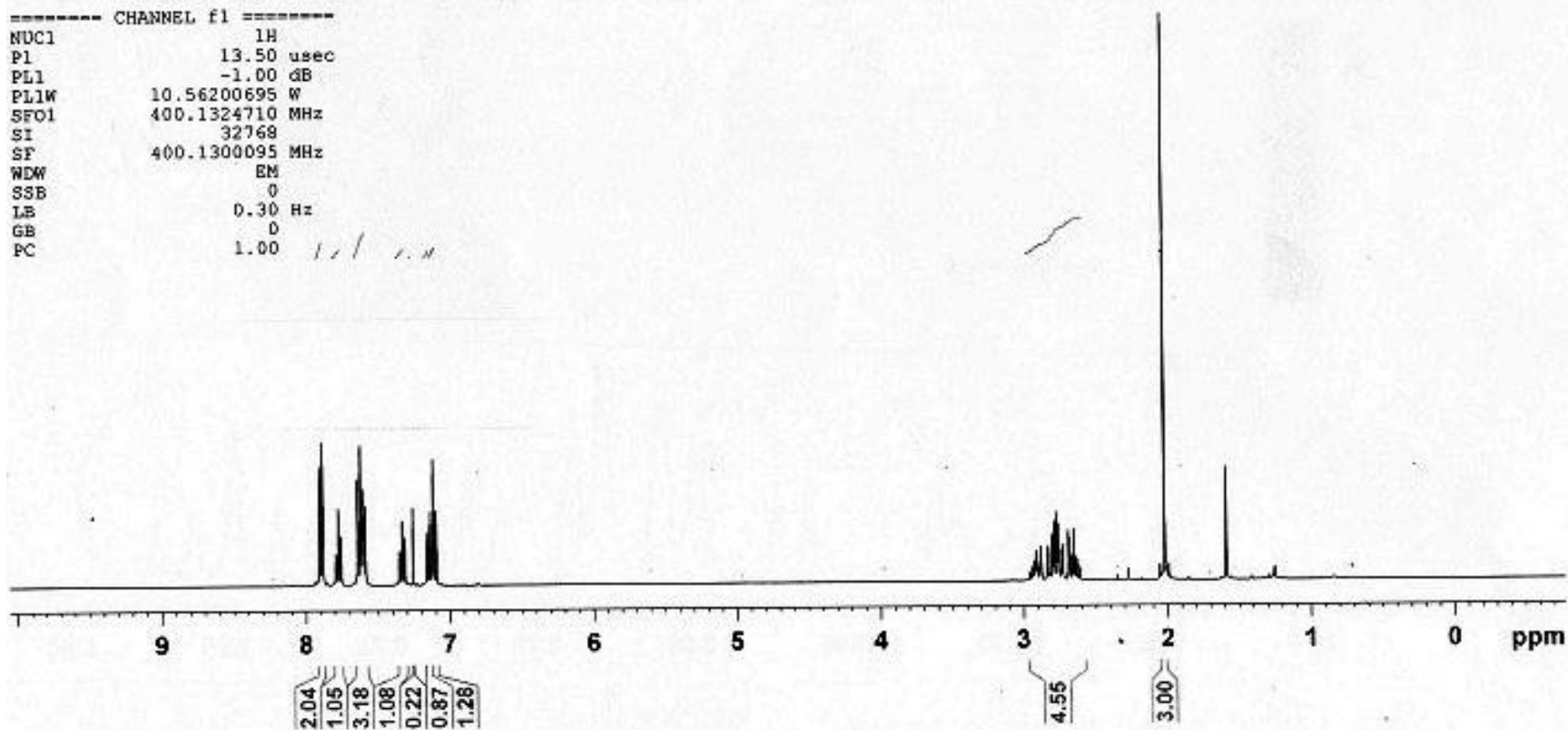


Fig S52. <sup>1</sup>H NMR Spectrum of 3l



```

NAME      INN-3-KSB-70B-13C
EXPNO     1
PROCNO    1
Date_     20130518
Time      16.53
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        84
DS        4
SWH       24030.461 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
TDO       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        100.6127547 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

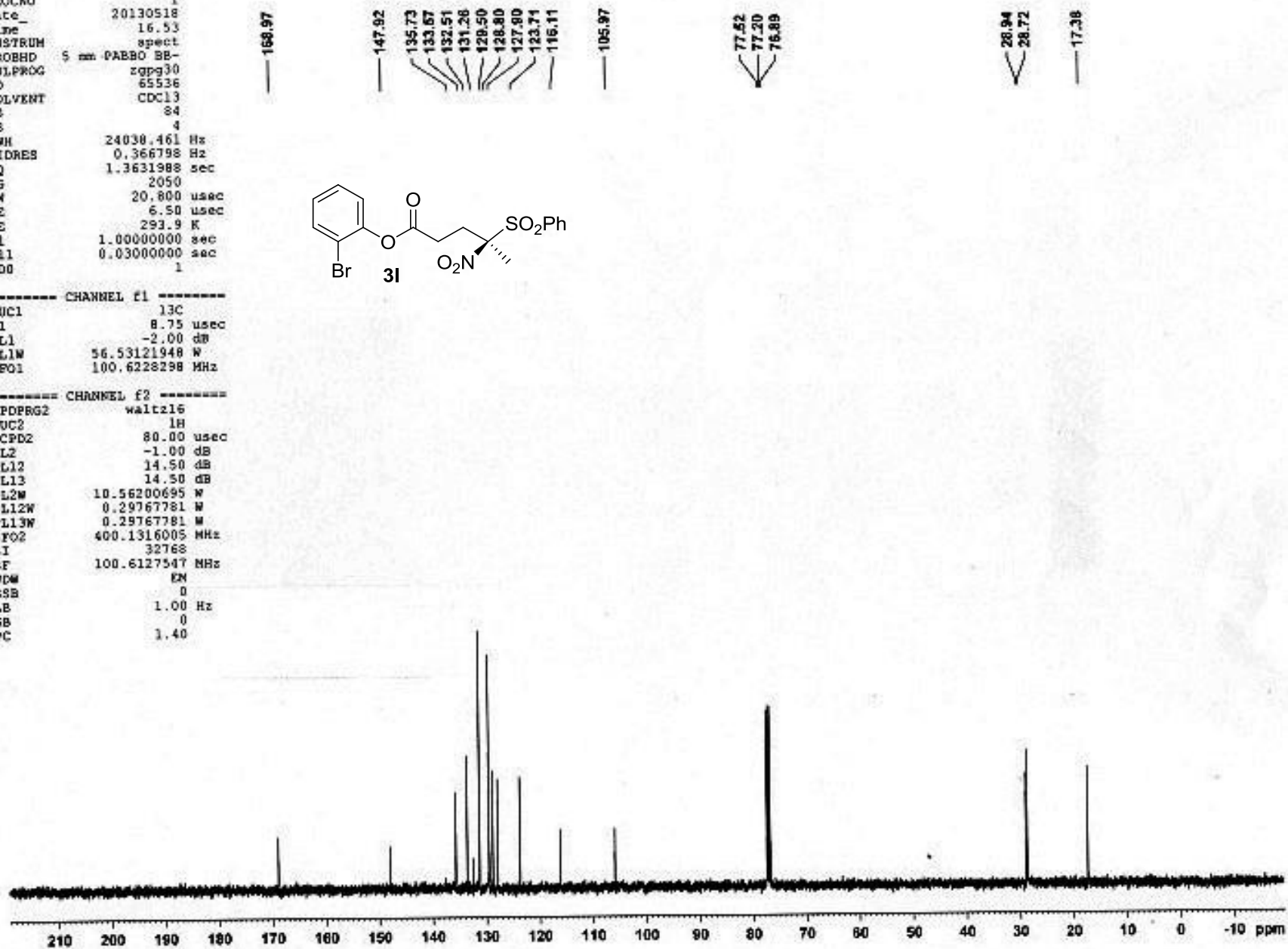
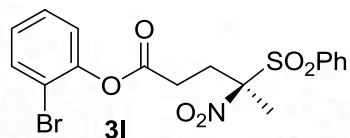
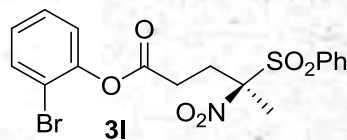


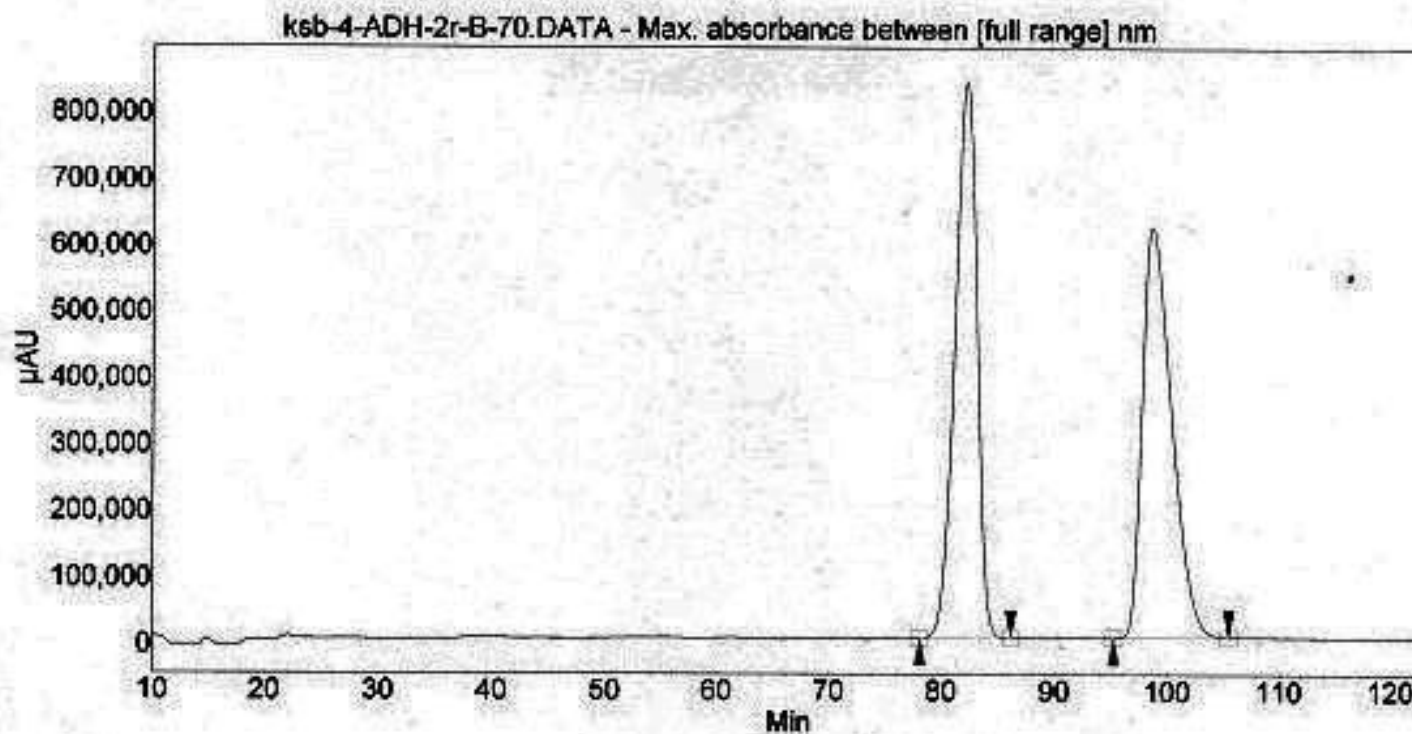
Fig S53. <sup>13</sup>C NMR Spectrum of 31

# Chromatogram : ksb-4-ADH-2r-B-70\_channel5

System : Jasco HPLC  
 Method : ksb-2%-05f-250nm  
 User : User1



Acquired : 5/24/2013 5:46:53 PM  
 Processed : 5/24/2013 7:53:40 PM  
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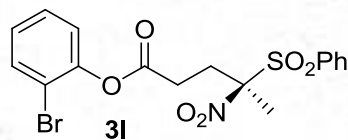
## Peak results :

Index	Name	Time (Min)	Quantity (% Area)	Height (UAU)	Area (UAU·Min)	Area % (%)
1	UNKNOWN	82.171	50.24	836723.9	1688640.7	50.241
2	UNKNOWN	98.618	49.76	618832.7	1070534.9	49.759
Total			100.00	1455556.6	3759175.6	100.000

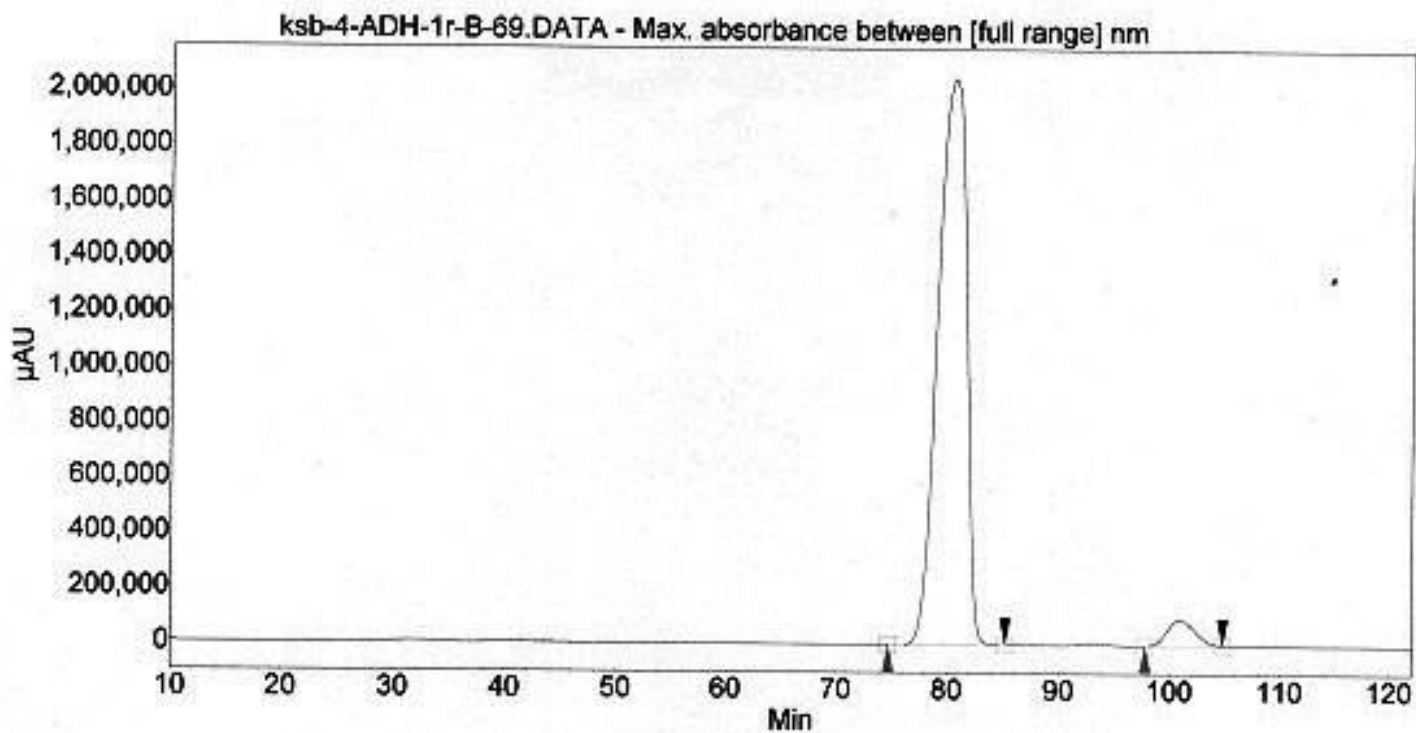
Fig S54. HPLC Profile of Racemic 3l

# Chromatogram : ksb-4-ADH-1r-B-69\_channel5

System : Jasco HPLC  
 Method : ksb-2%-05f-250nm  
 User : User1



Acquired : 5/24/2013 8:12:08 PM  
 Processed : 5/24/2013 11:03:40 PM  
 Printed : 5/24/2013 11:04:36 PM

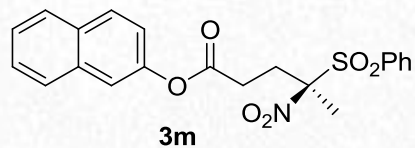


## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	80.512	95.63	2050552.2	6087593.1	95.628
2	UNKNOWN	100.919	4.37	95544.1	278294.0	4.372
Total			100.00	2146096.4	6365887.1	100.000

Fig S55. HPLC Profile of Enantioenriched 3l

NAME INN-4-KSB-32A-1H  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20130219  
 Time\_ 21.00  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 9  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.3 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  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

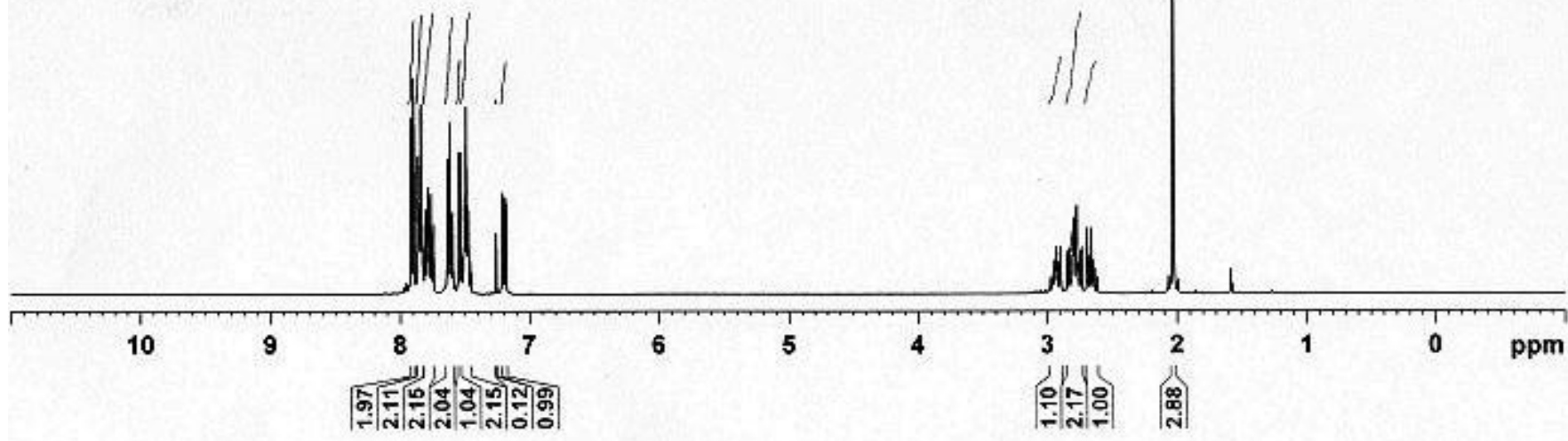


Fig S56. <sup>1</sup>H NMR Spectrum of 3m

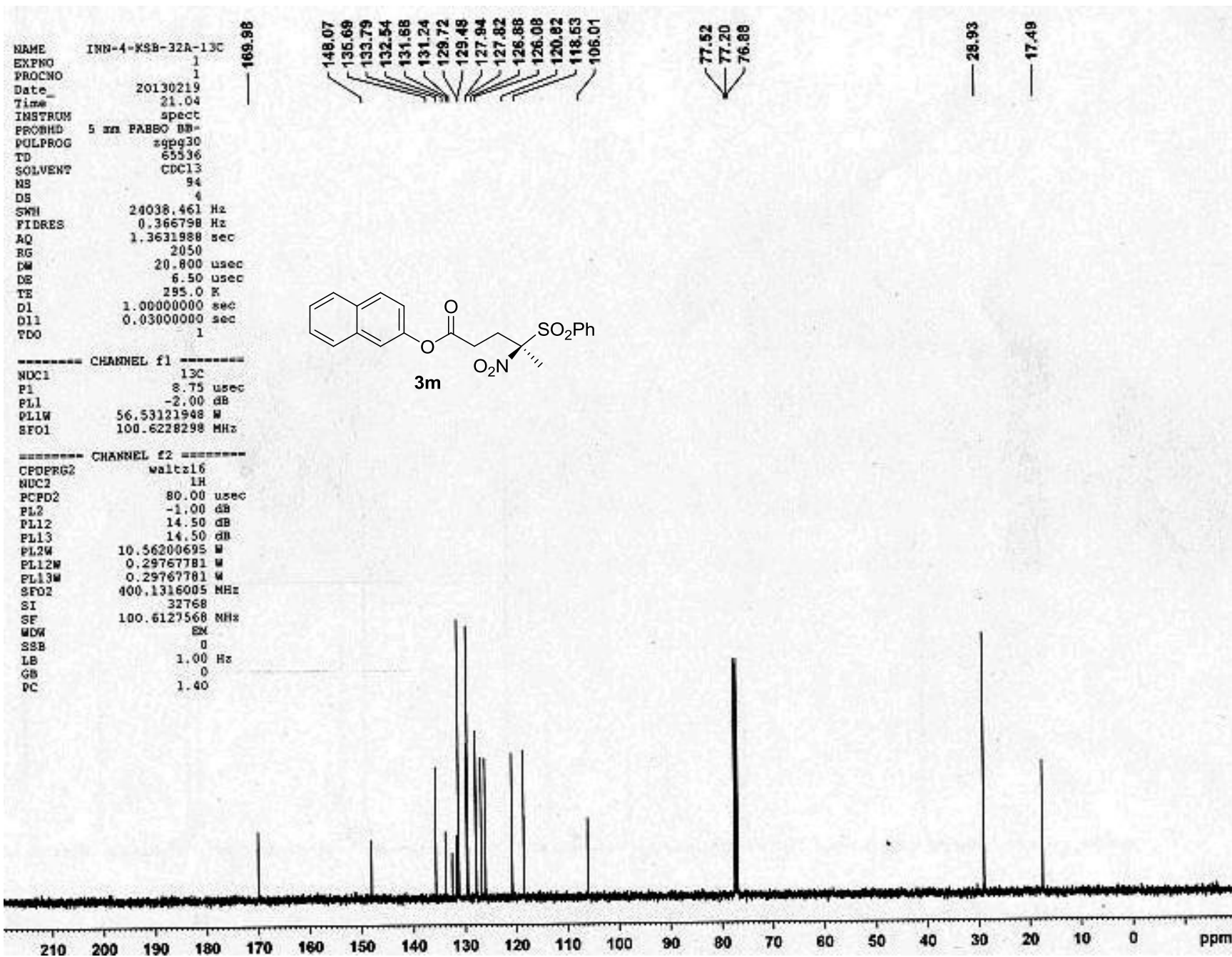


Fig S57. <sup>13</sup>C NMR Spectrum of 3m

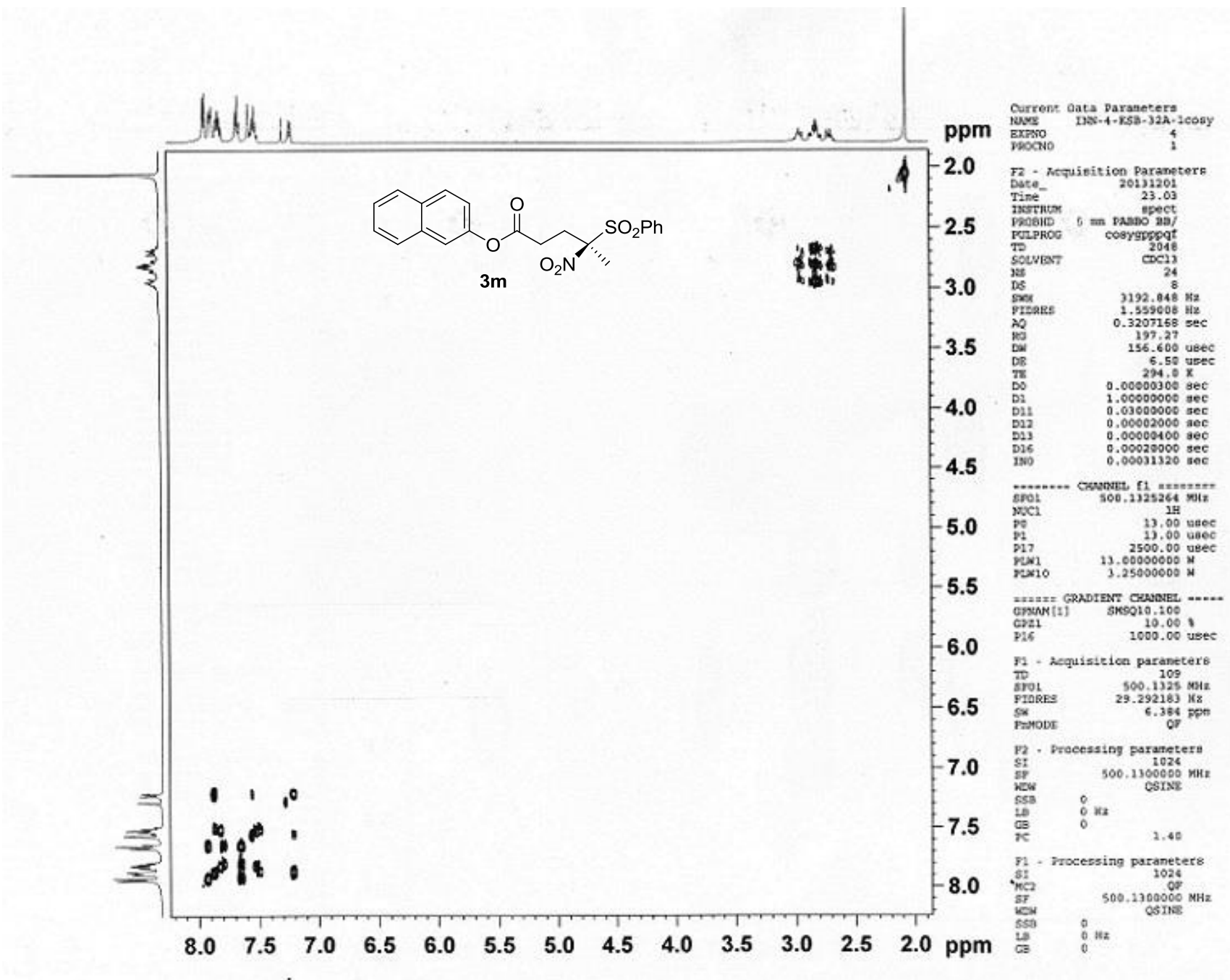


Fig S58.  $^1\text{H}$ - $^1\text{H}$  COSY NMR Spectrum of 3m

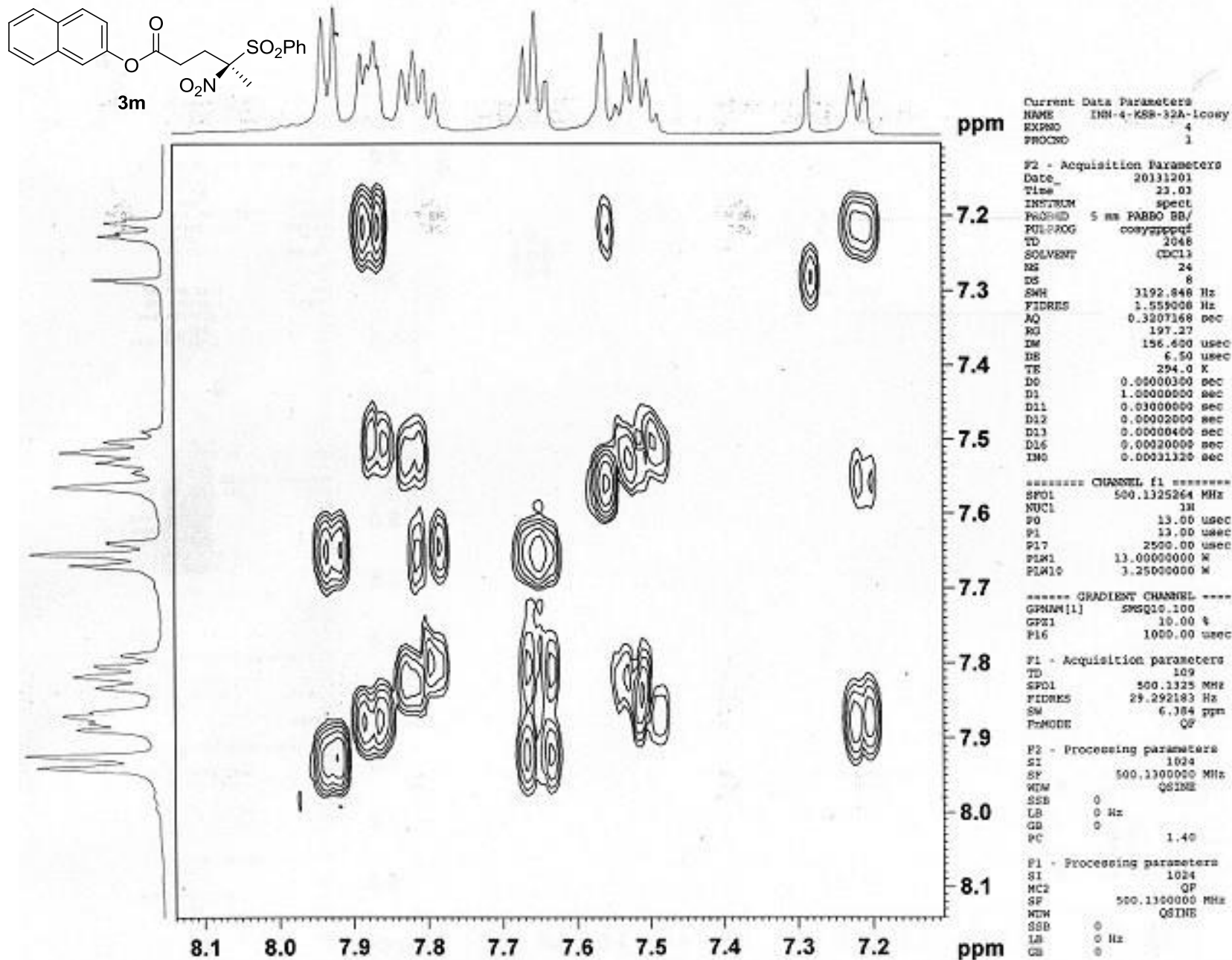
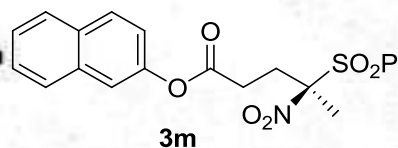


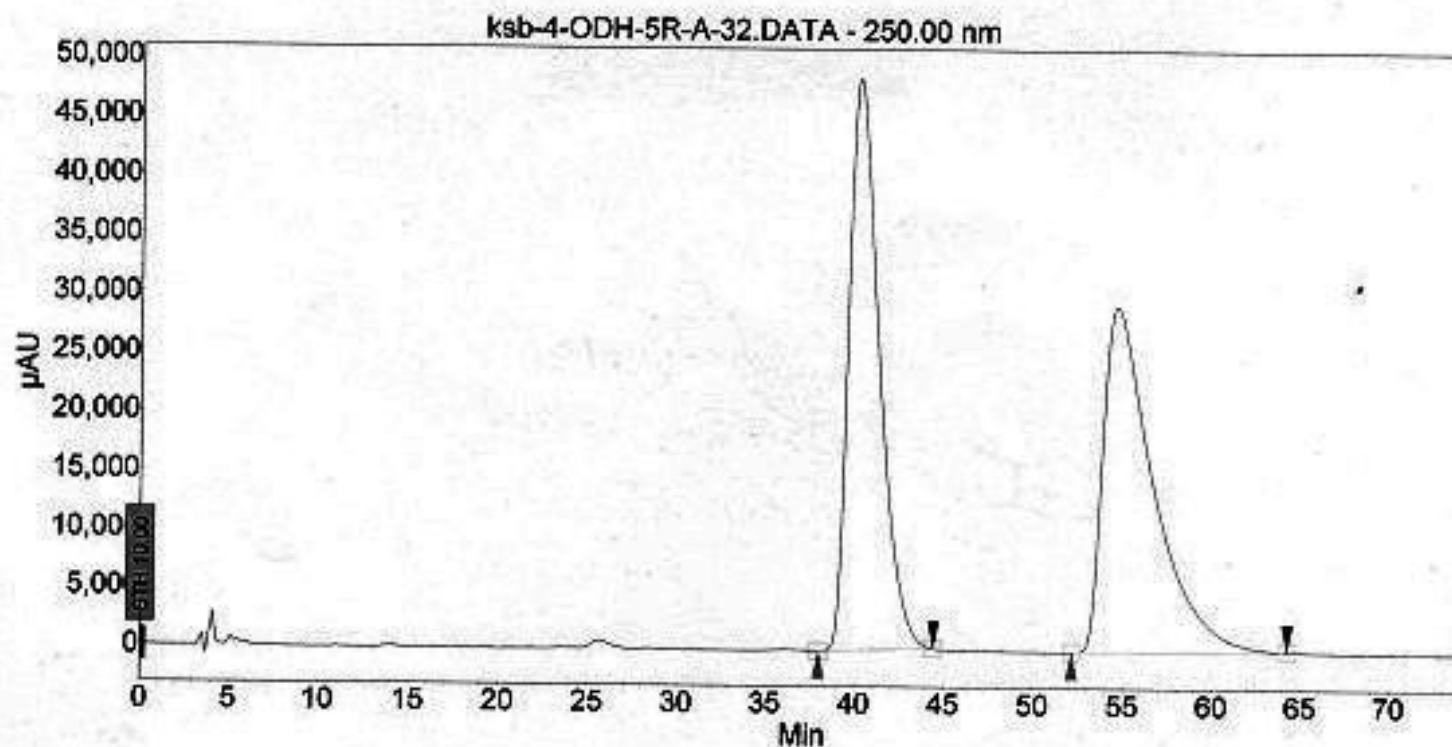
Fig S59.  $^1\text{H}$ - $^1\text{H}$  Expanded COSY NMR Spectrum of 3m

# Chromatogram : ksb-4-ODH-5R-A-32\_channel4

System : Jasco HPLC  
 Method : ksb-30%-1f-250nm  
 User : User1



Acquired : 5/11/2013 7:27:38 PM  
 Processed : 5/11/2013 8:43:04 PM  
 Printed : 5/11/2013 8:43:14 PM



## Peak results :

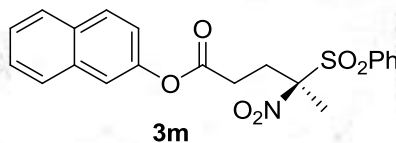
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	40.132	50.15	48430.9	96299.5	50.155
2	UNKNOWN	54.580	49.85	29327.0	95705.7	49.845
Total			100.00	77757.9	192005.2	100.000

Fig S60. HPLC Profile of Racemic 3m

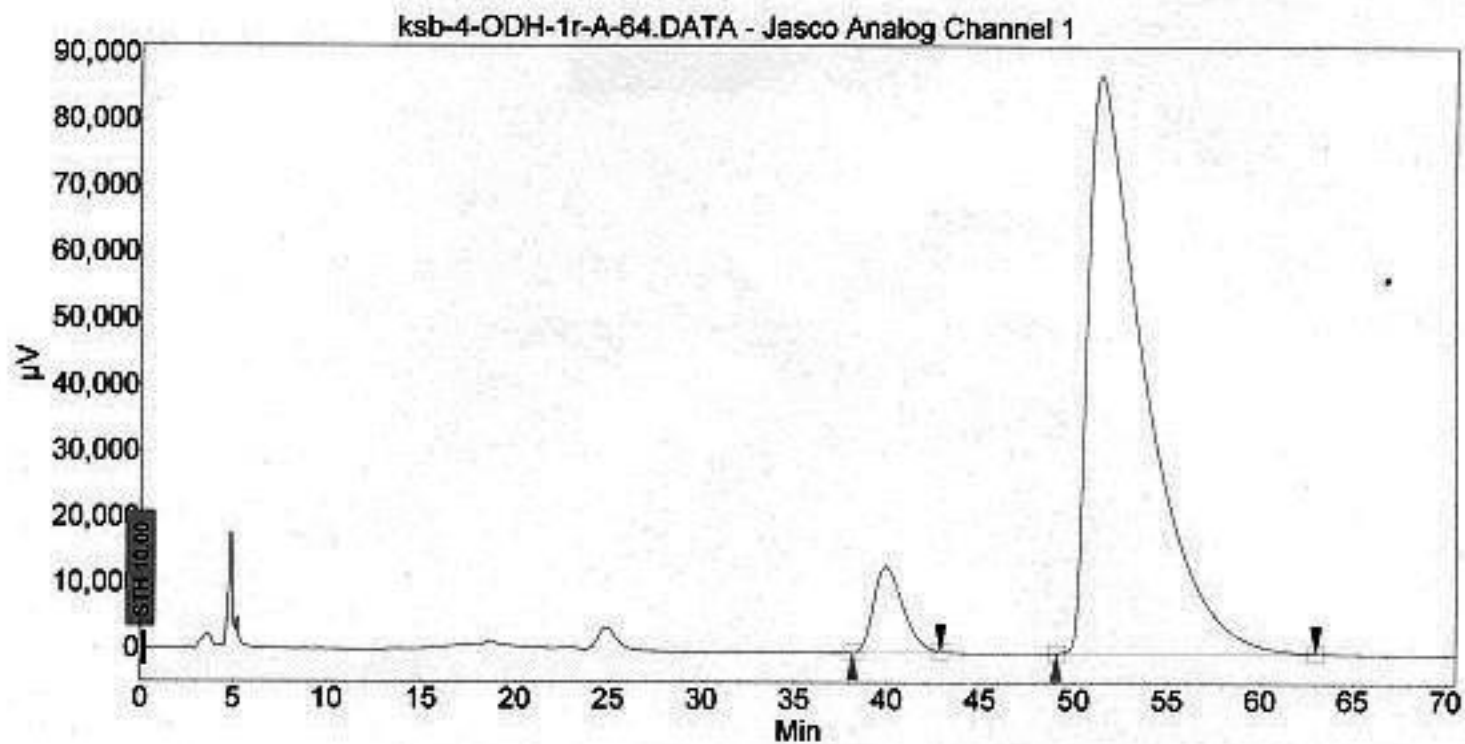


# Chromatogram : ksb-4-ODH-1r-A-64\_channel2

System : Jasco HPLC  
Method : ksb-30%-1f-250nm  
User : User1



Acquired : 5/11/2013 4:52:43 PM  
Processed : 5/11/2013 6:04:25 PM  
Printed : 5/11/2013 6:05:48 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV/Min]	Area % [%]
1	UNKNOWN	39.908	7.22	13095.3	23859.9	7.222
2	UNKNOWN	51.375	92.78	87251.6	308511.0	92.778
Total			100.00	100346.9	330370.9	100.000

Fig S61. HPLC Profile of Enantioenriched 3m

```

NAME      INN-4-KSB-32B-1H
EXPNO     1
PROCNO    1
Date_     20130219
Time      20.45
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         0
SWH        6223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         32
DW         60.800 usec
DE         6.50 usec
TE         294.3 K
D1         1.00000000 sec
TD0        1

```

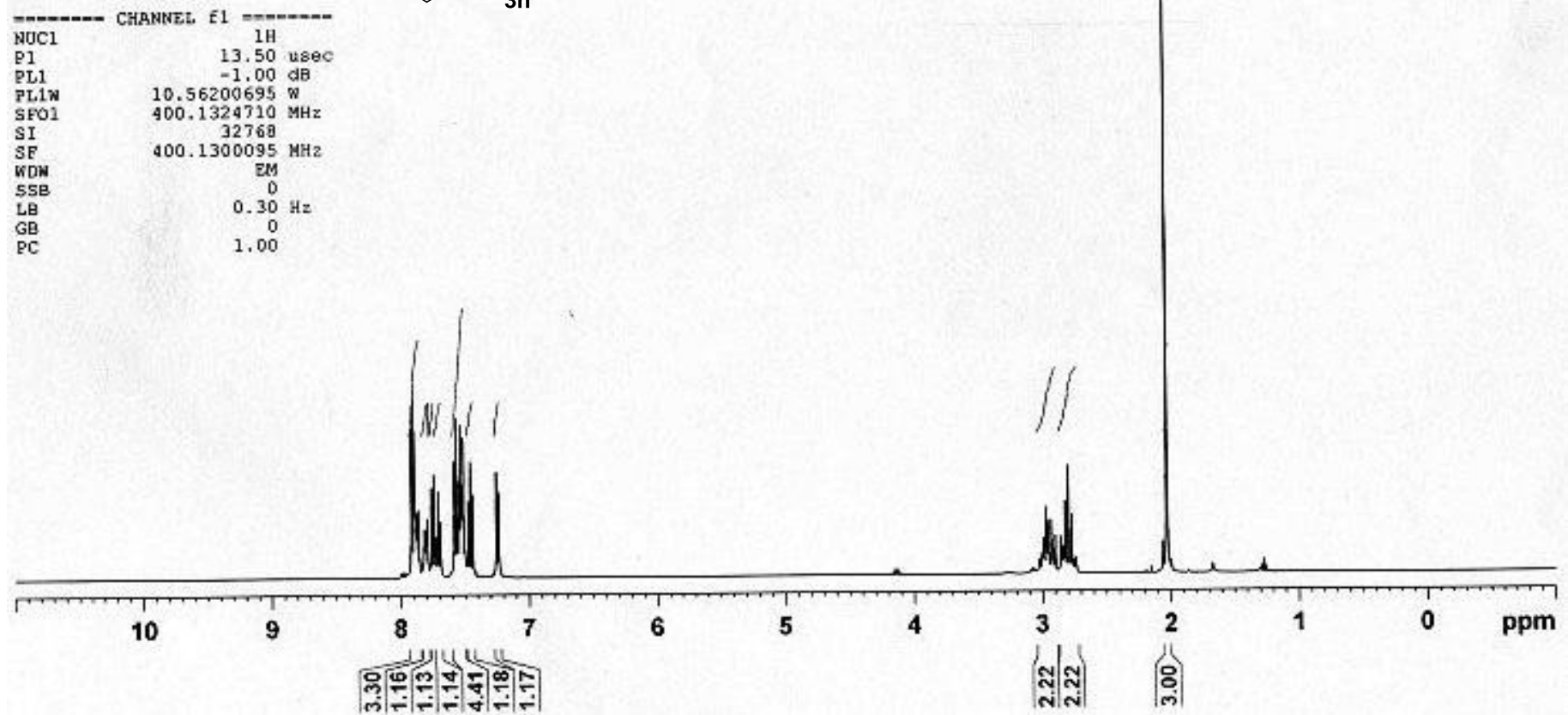
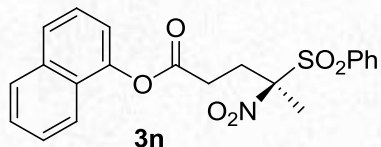


Fig S62. <sup>1</sup>H NMR Spectrum of 3n

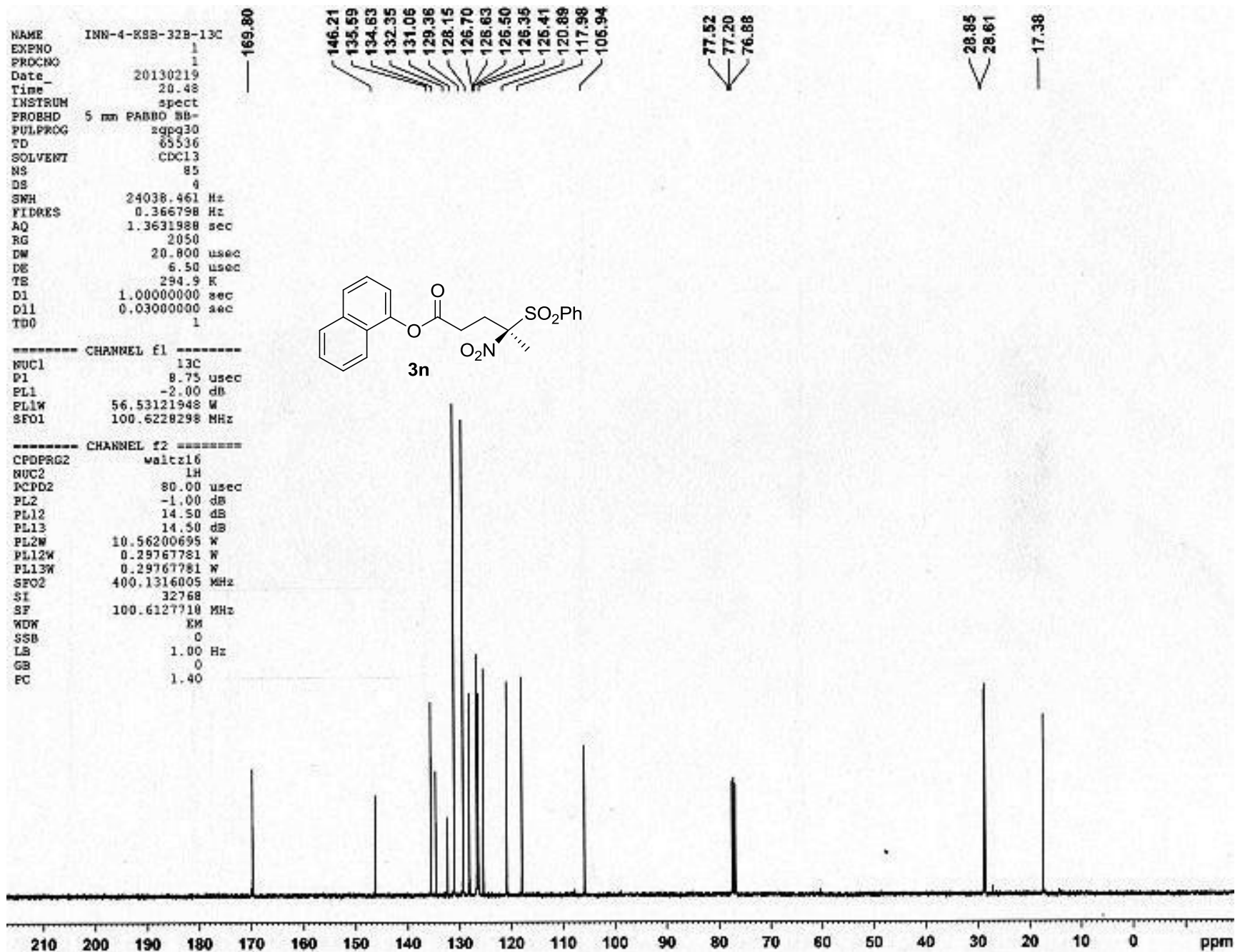
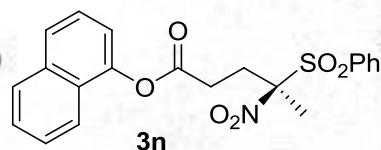


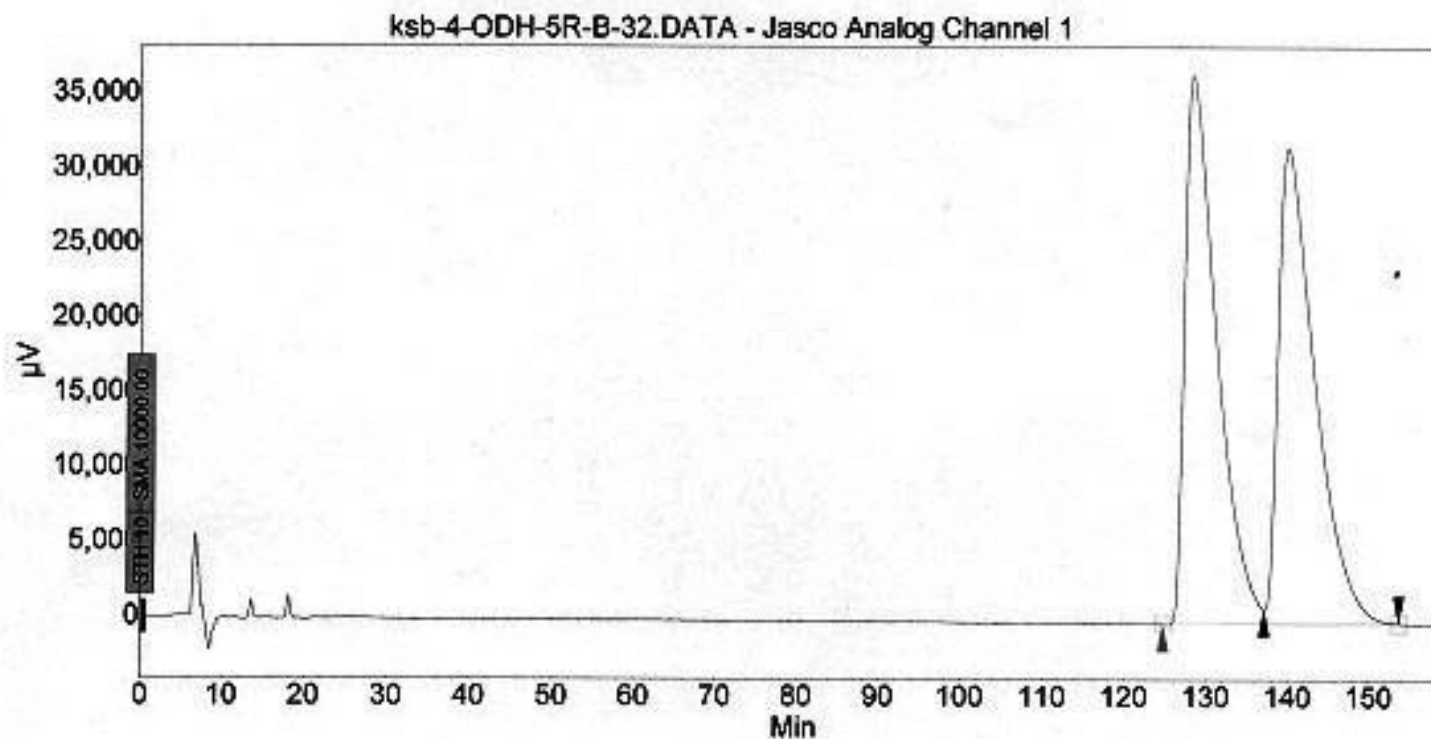
Fig S63. <sup>13</sup>C NMR Spectrum of 3n

# Chromatogram : ksb-4-ODH-5R-B-32\_channel2

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/10/2013 3:19:58 PM  
Processed : 5/10/2013 6:03:45 PM  
Printed : 5/10/2013 6:04:01 PM



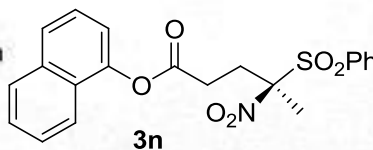
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	128.358	49.92	36620.9	166626.9	49.920
2	UNKNOWN	140.000	50.08	31803.4	167163.4	50.080
Total			100.00	68424.4	333790.3	100.000

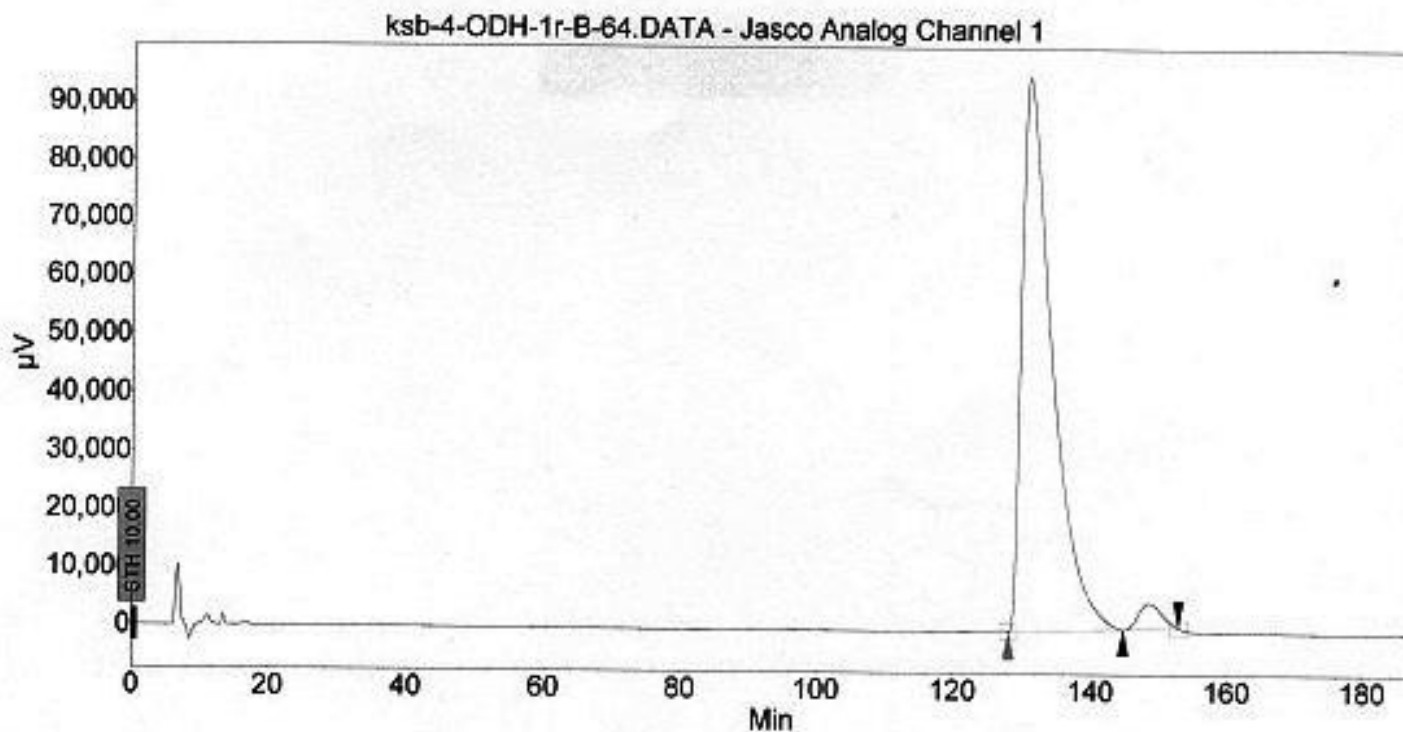
Fig S64. HPLC Profile of Racemic 3n

# Chromatogram : ksb-4-ODH-1r-B-64\_channel2

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/7/2013 1:27:17 PM  
Processed : 5/7/2013 4:34:44 PM  
Printed : 5/7/2013 4:48:00 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	130.692	96.59	95206.1	488193.3	96.590
2	UNKNOWN	148.475	3.41	4417.7	17237.1	3.410
Total			100.00	99623.8	505430.4	100.000

Fig S65. HPLC Profile of Enantioenriched 3n

NAME INN-4-KSB-74-A-1H  
 EXPNO 17  
 PROCNO 1  
 Date 20130524  
 Time 17.09  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 66962  
 SOLVENT CDC13  
 NS 11  
 DS 0  
 SWH 11160.714 Hz  
 FIDRES 0.166672 Hz  
 AQ 2.9999475 sec  
 RG 32  
 DW 44.800 usec  
 DE 6.50 usec  
 TE 294.6 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 13.50 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SFO1 400.1332010 MHz  
 SI 32768  
 SF 400.1300092 MHz  
 WDM EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

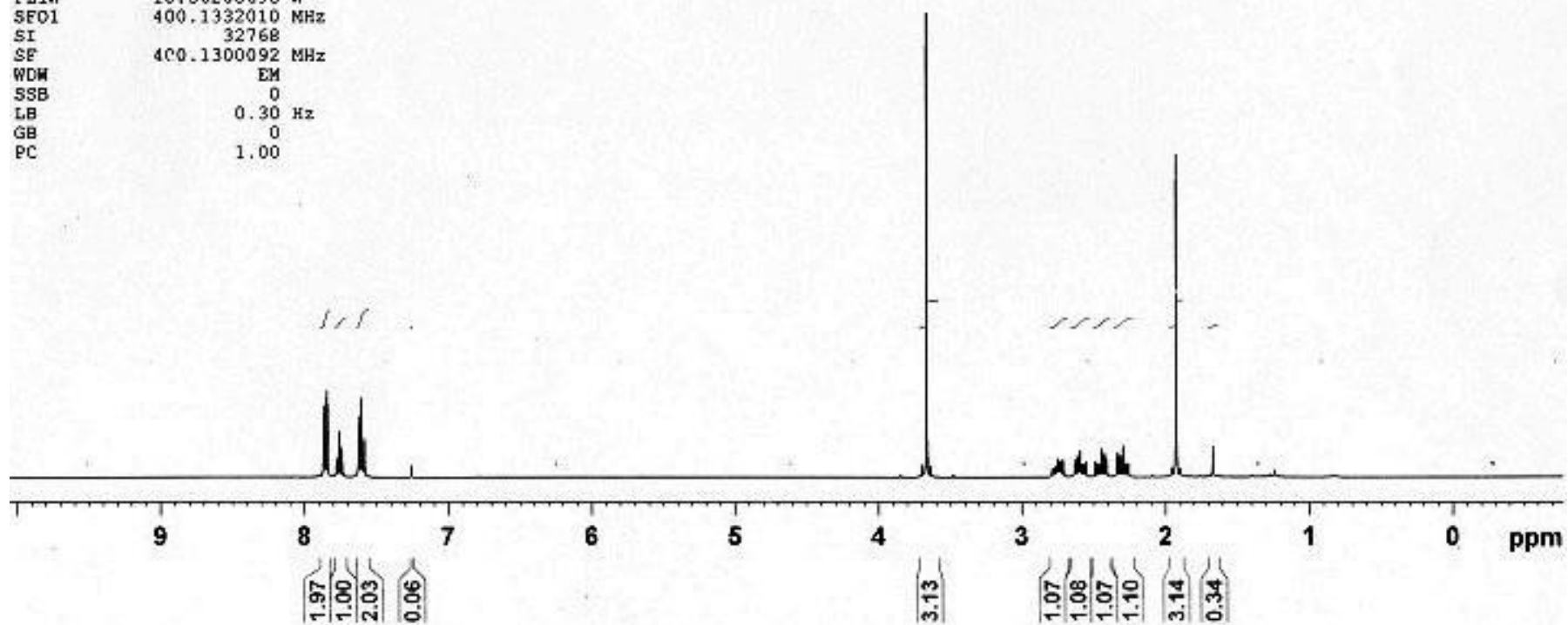
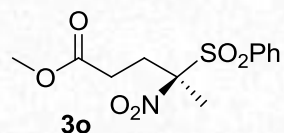


Fig S66. <sup>1</sup>H NMR Spectrum of 3o

NAME \* INN-9-KSB-74-A-13C  
 EXPNO 18  
 PROCNO 1  
 Date 20130524  
 Time 17.11  
 INSTRUM spect  
 PROSHD 5 mm PABBO BR-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 46  
 DS 2  
 SWH 27777.777 Hz  
 FIDRES 0.423855 Hz  
 AQ 1.1796980 sec  
 RG 2050  
 DW 18.000 usec  
 DE 6.50 usec  
 TE 295.1 K  
 D1 1.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 8.75 usec  
 PL1 -2.00 dB  
 PL1W 56.53121948 W  
 SFO1 100.6242389 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 100.6127564 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

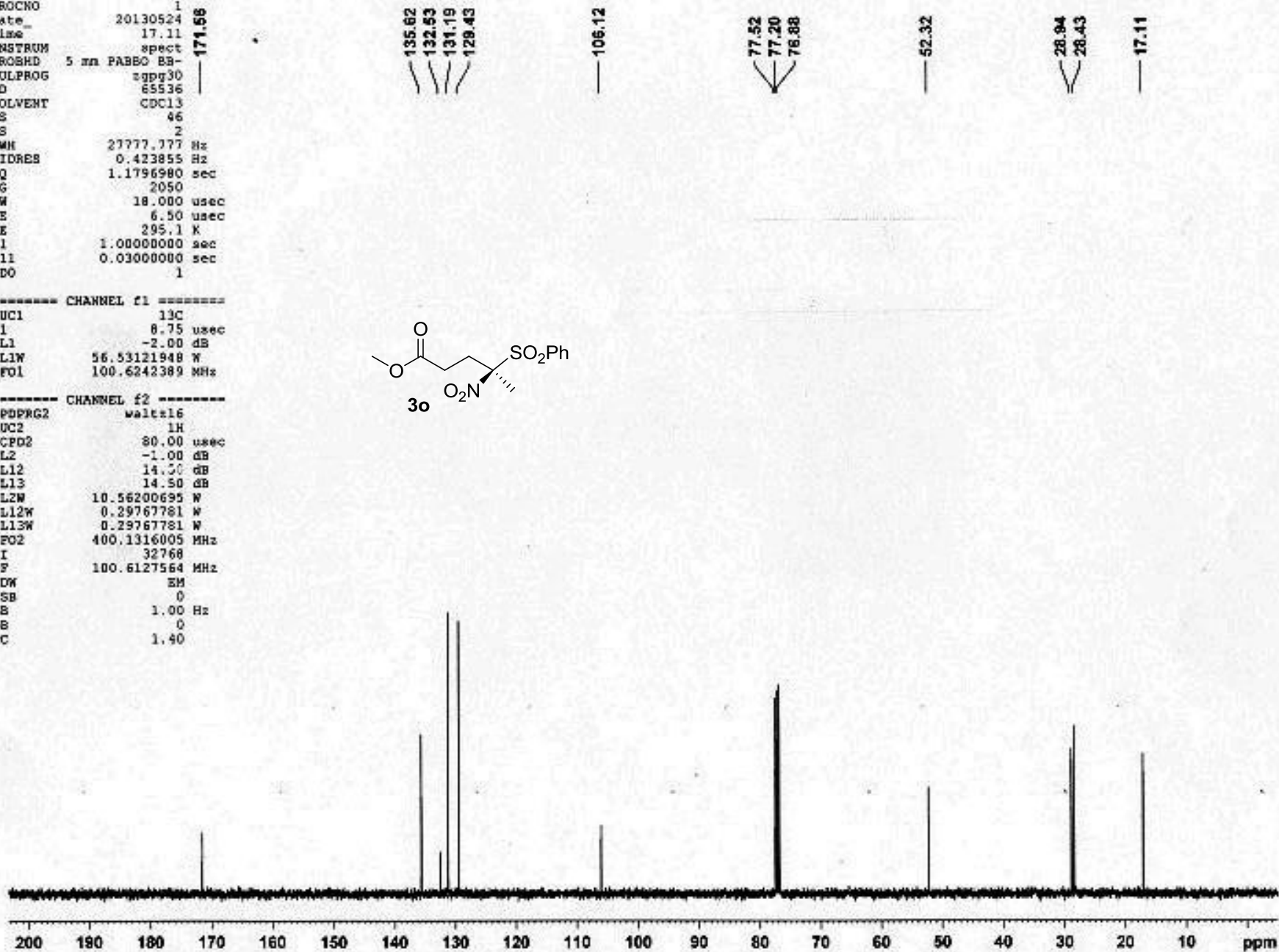
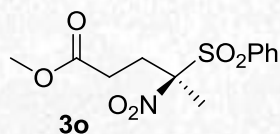
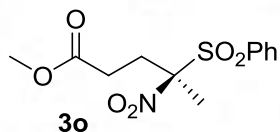


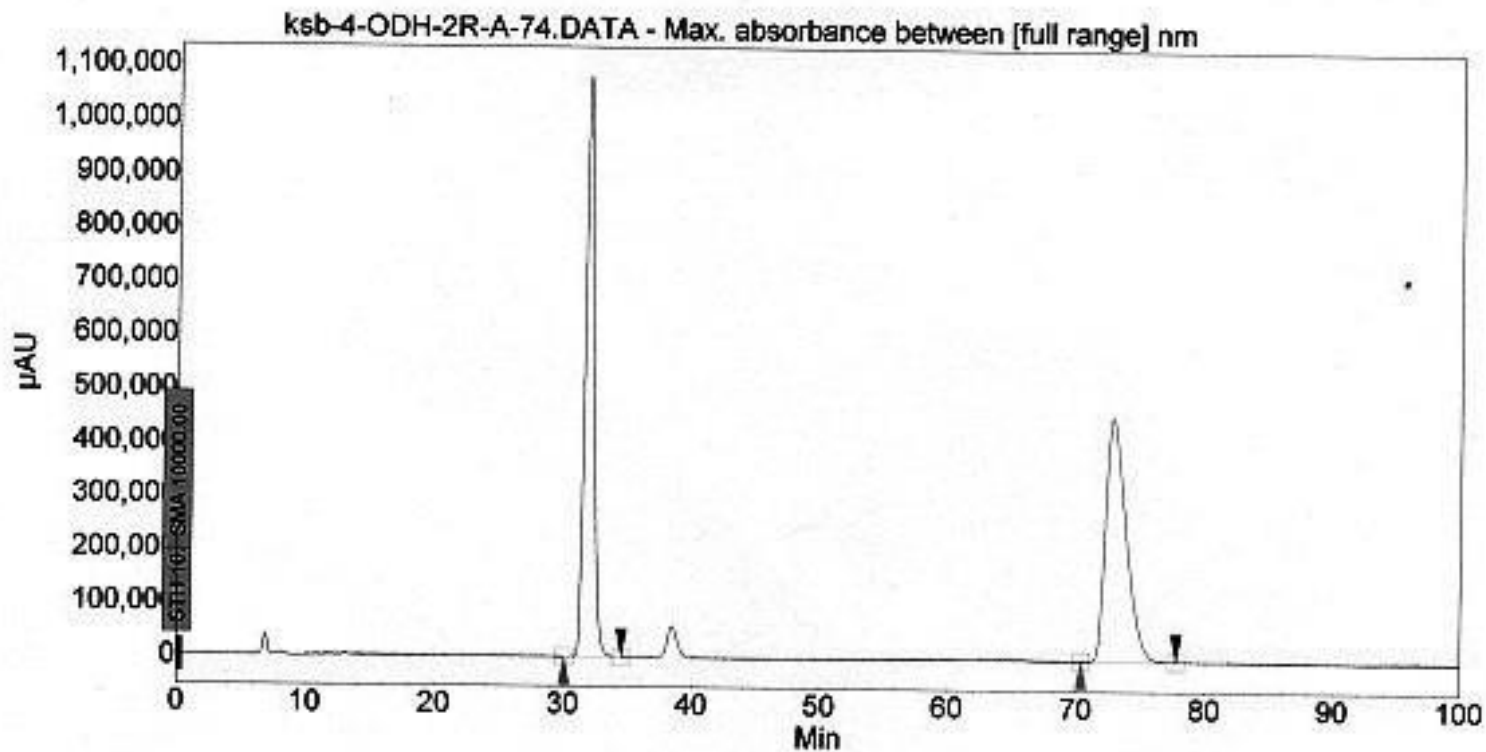
Fig SS67. <sup>13</sup>C NMR Spectrum of 3o

# Chromatogram : ksb-4-ODH-2R-A-74\_channel5

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/24/2013 12:11:57 PM  
Processed : 5/24/2013 2:40:40 PM  
Printed : 5/24/2013 2:40:48 PM



## Peak results :

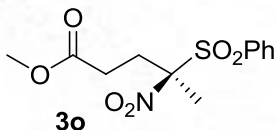
Index	Name	Time [Min]	Quantity [% Area]	Height [AU]	Area [AU.Min]	Area % [%]
2	UNKNOWN	31.757	50.48	1080311.9	855642.3	50.480
1	UNKNOWN	72.732	49.52	454920.9	839369.4	49.520
Total			100.00	1535232.8	1695011.7	100.000

Fig S68. HPLC Profile of Racemic 3o

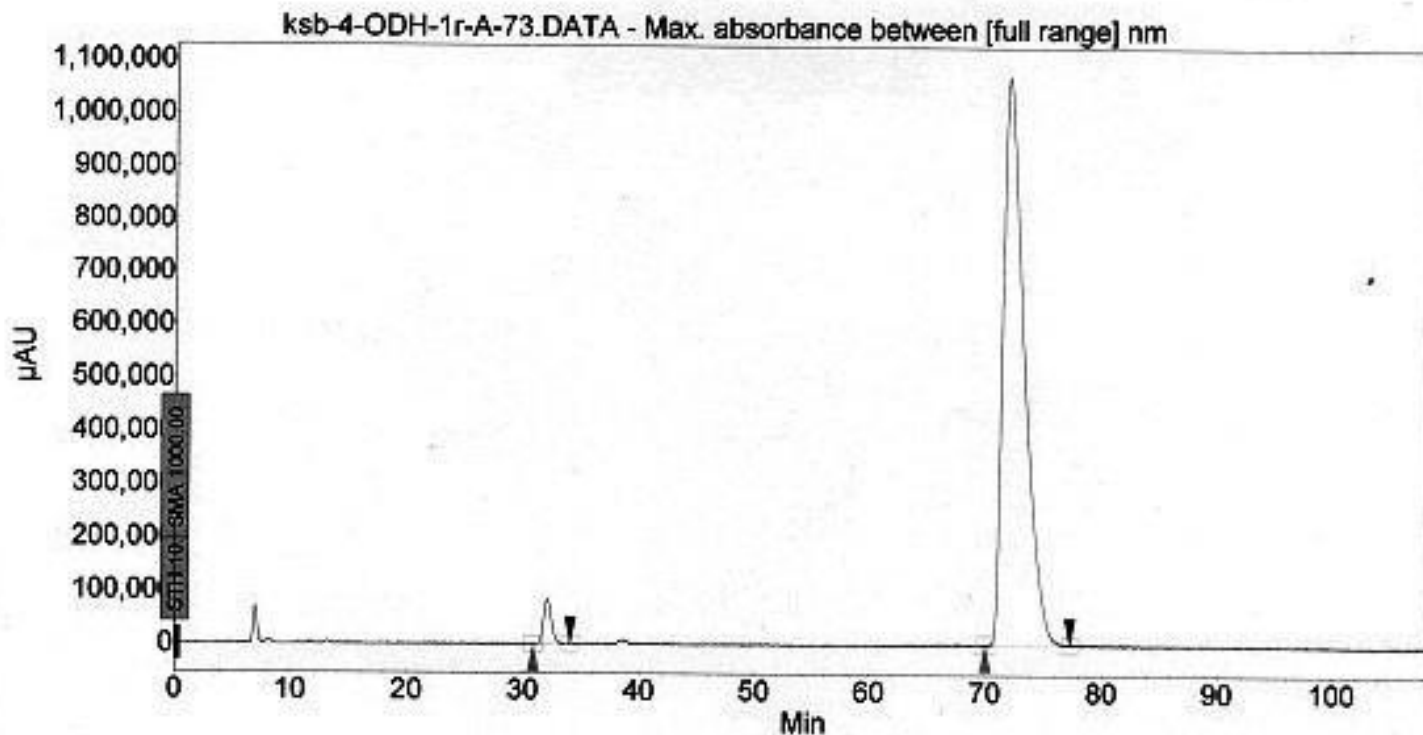


# Chromatogram : ksb-4-ODH-1r-A-73\_channel5

System : Jasco HPLC  
 Method : ksb-10%-05f-250nm  
 User : User1



Acquired : 5/24/2013 10:11:16 AM  
 Processed : 5/24/2013 12:14:01 PM  
 Printed : 5/24/2013 12:14:12 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	32.055	2.67	84682.8	66184.9	2.874
1	UNKNOWN	71.881	97.13	1069963.6	2237037.9	97.126
Total			100.00	1154646.4	2303222.9	100.000

Fig S69. HPLC Profile of Enantioenriched 3o

NAME INN-4-KSB-66C-1H  
 EXPNO 4  
 PROCNO 1  
 Date 20130516  
 Time 18.52  
 INSTRUM spect  
 PROBHD 5 mm F4BBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 9  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.6 K  
 D1 1.00000000 sec  
 TD0 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

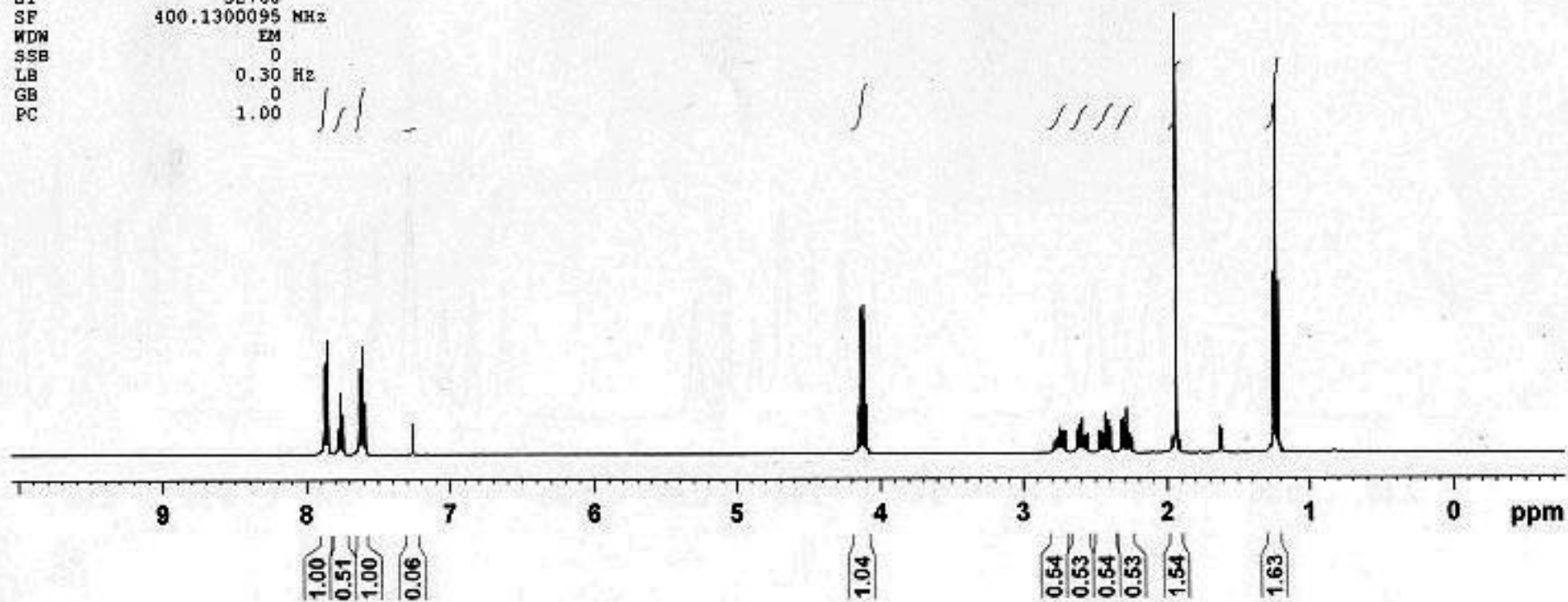
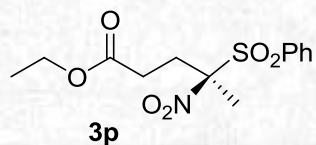


Fig S70. <sup>1</sup>H NMR Spectrum of 3p

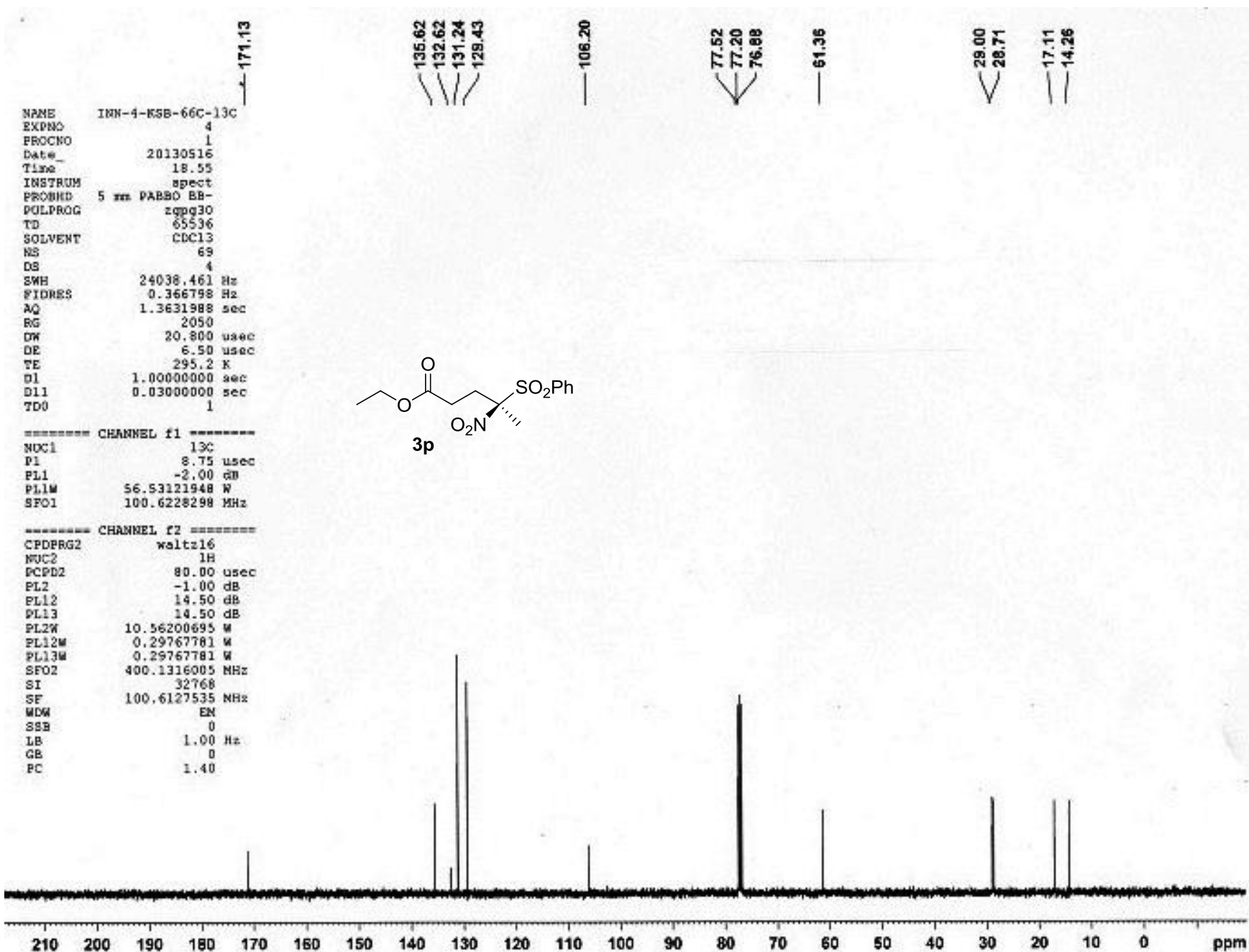
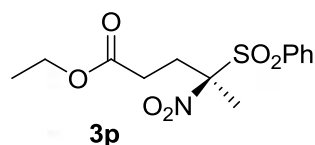


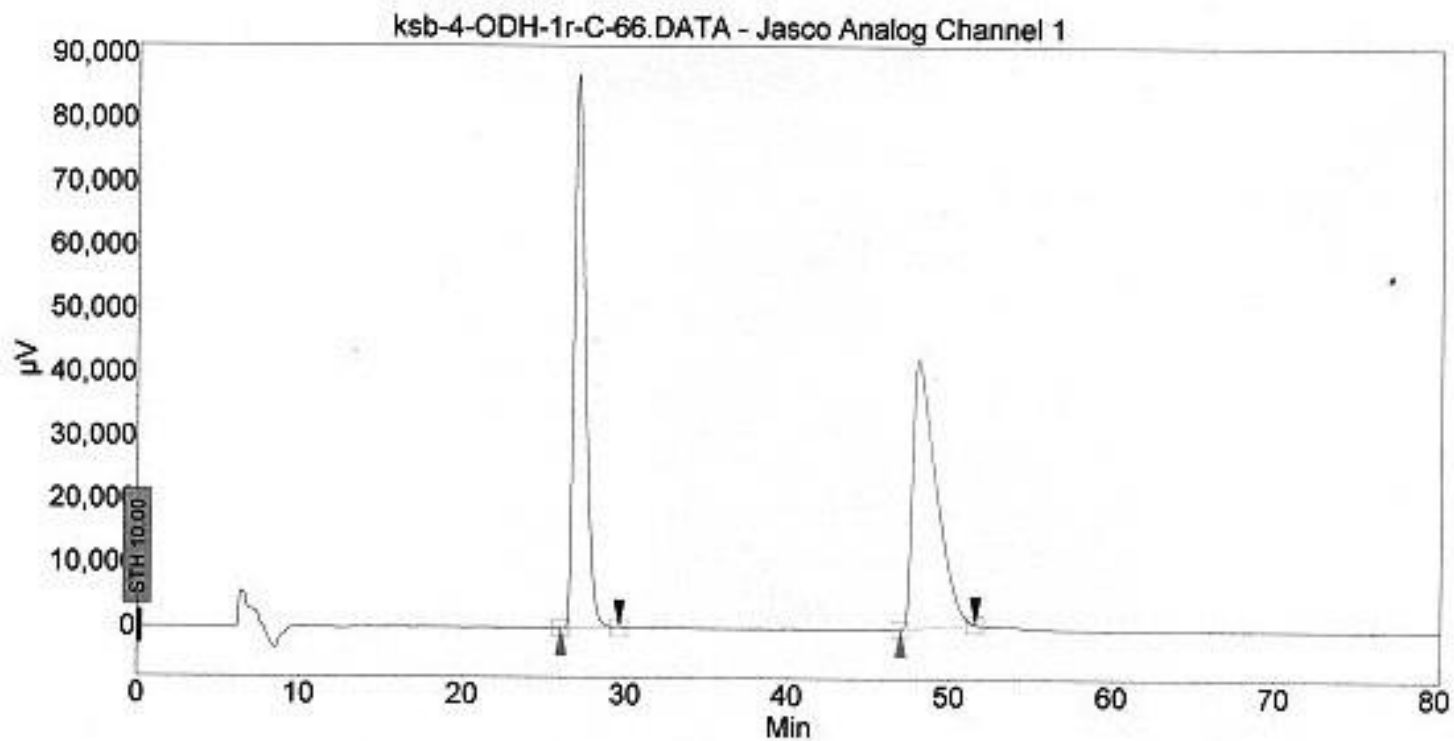
Fig S71. <sup>13</sup>C NMR Spectrum of 3p

# Chromatogram : ksb-4-ODH-1r-C-66\_channel2

System : Jasco HPLC  
 Method : ksb-10%-05f-250nm  
 User : User1



Acquired : 5/16/2013 5:10:34 PM  
 Processed : 5/16/2013 6:37:15 PM  
 Printed : 5/16/2013 6:37:32 PM



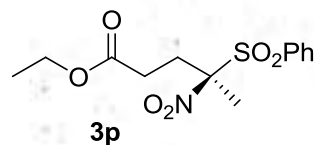
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	26.958	49.80	86860.4	69678.4	49.804
2	UNKNOWN	48.067	50.20	42293.3	70225.9	50.196
Total			100.00	129153.7	139904.3	100.000

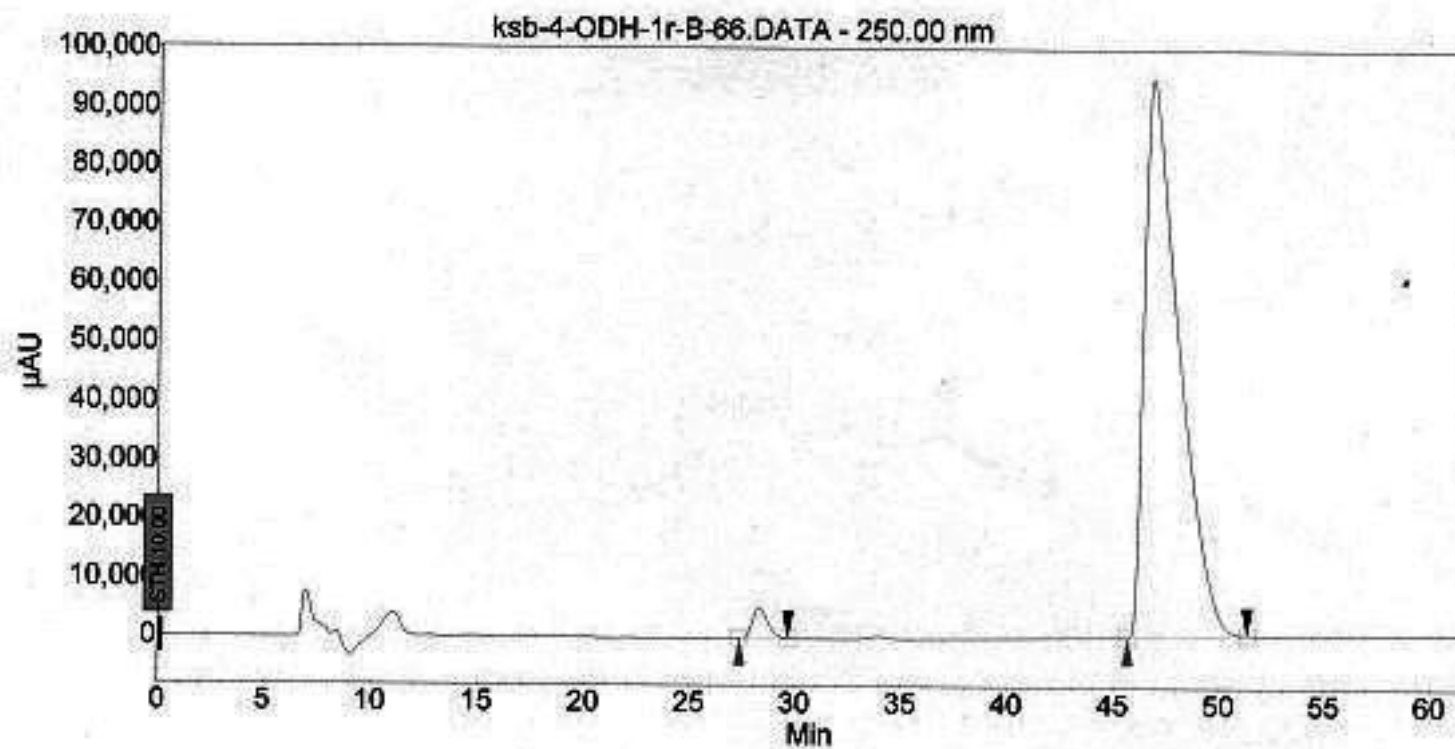
Fig S72. HPLC Profile of Racemic 3p

# Chromatogram : ksb-4-ODH-1r-B-66\_channel4

System : Jasco HPLC  
Method : ksb-10%-05f-250nm  
User : User1



Acquired : 5/16/2013 6:42:35 PM  
Processed : 5/16/2013 7:45:44 PM  
Printed : 5/16/2013 7:47:09 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µAU]	Area [µAU.Min]	Area % [%]
1	UNKNOWN	28.318	2.38	5265.7	4346.8	2.375
2	UNKNOWN	46.730	97.62	94720.4	178668.7	97.625
Total			100.00	99986.1	183015.5	100.000

Fig S73. HPLC Profile of Enantioenriched 3p

NAME INN-4-MSB-B3A-1H  
 EXPNO 5  
 PROCNO 1  
 Date 20130613  
 Time 19.55  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 5  
 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  
 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  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

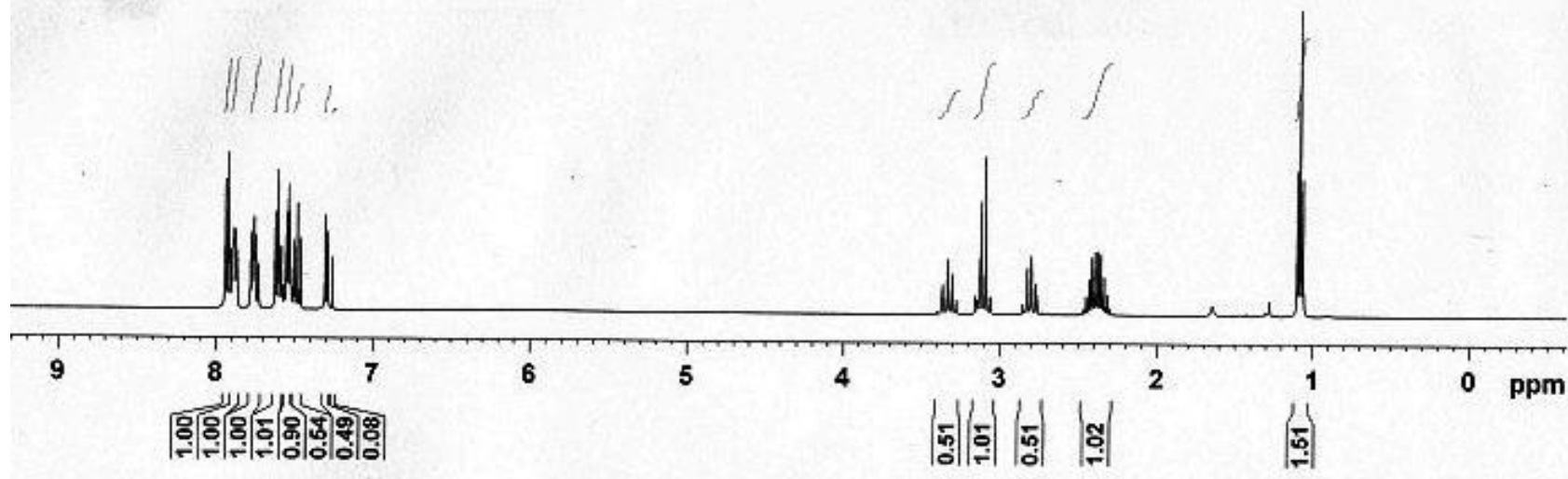
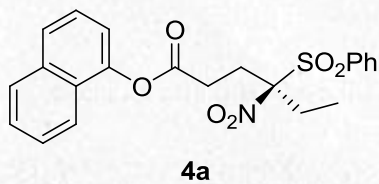


Fig S74. <sup>1</sup>H NMR Spectrum of 4a

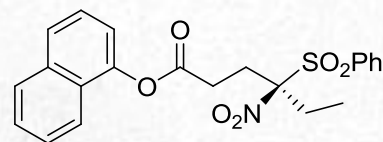
NAME INN-4-KSB-83A-13C  
 EXPNO 5  
 PROCNO 1  
 Date\_ 20130613  
 Time 19.57  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 50  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 294.8 K  
 D1 1.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

146.46  
 135.54  
 134.75  
 133.42  
 131.18  
 129.34  
 128.20  
 126.76  
 126.71  
 126.87  
 126.36  
 125.50  
 121.12  
 118.12  
 109.25

77.52  
 77.20  
 76.88

28.10  
 26.55  
 25.71

8.30



4a

===== 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 100.6127614 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

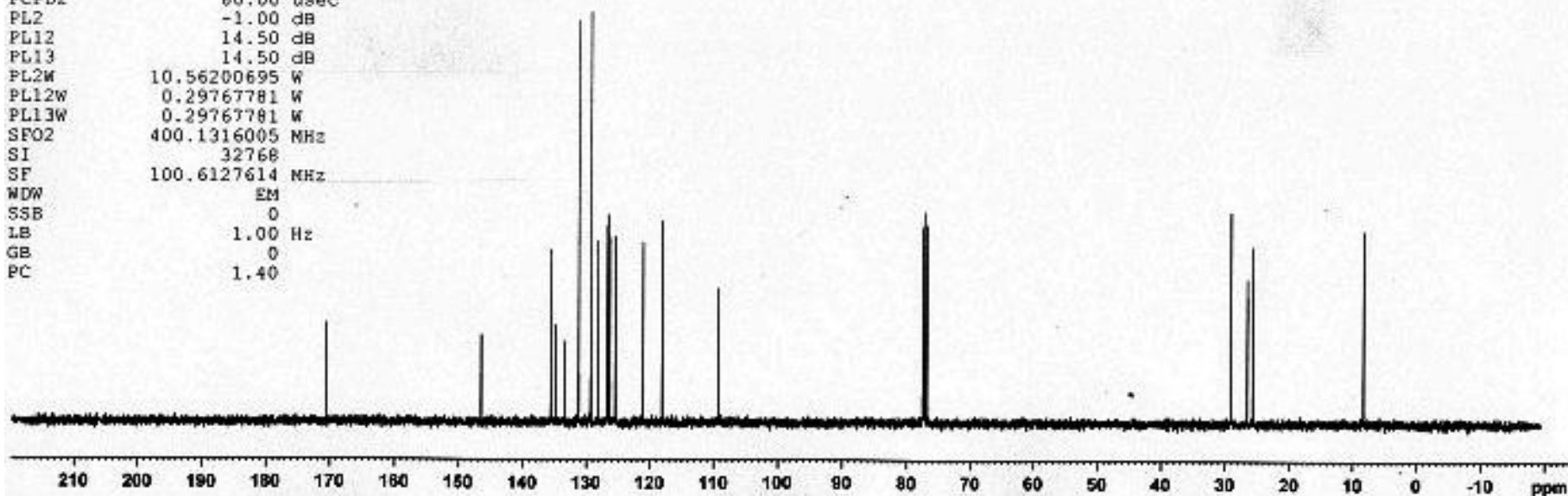
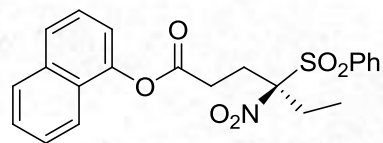


Fig S75. <sup>13</sup>C NMR Spectrum of 4a

# Chromatogram : ksb-4-CE1-2r-A-80\_channel4

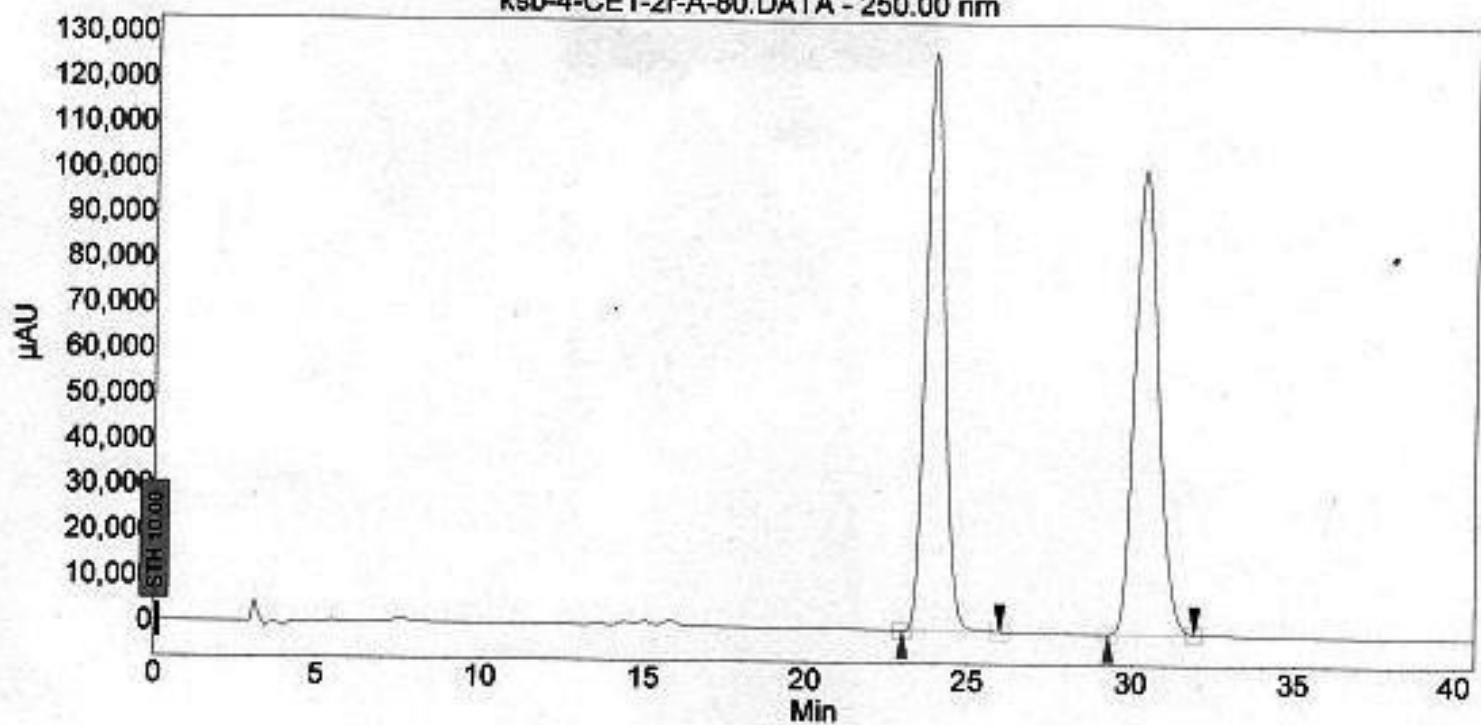
System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



Acquired : 6/12/2013 4:03:10 PM  
Processed : 6/12/2013 5:34:37 PM  
Printed : 6/12/2013 5:34:45 PM

4a

ksb-4-CE1-2r-A-80.DATA - 250.00 nm



## Peak results :

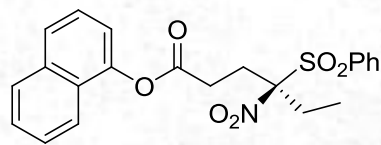
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	23.878	49.53	127526.8	84761.4	49.526
1	UNKNOWN	30.344	50.47	102311.2	86382.5	50.474
Total			100.00	229838.0	171143.8	100.000

Fig S76. HPLC Profile of Racemic 4a



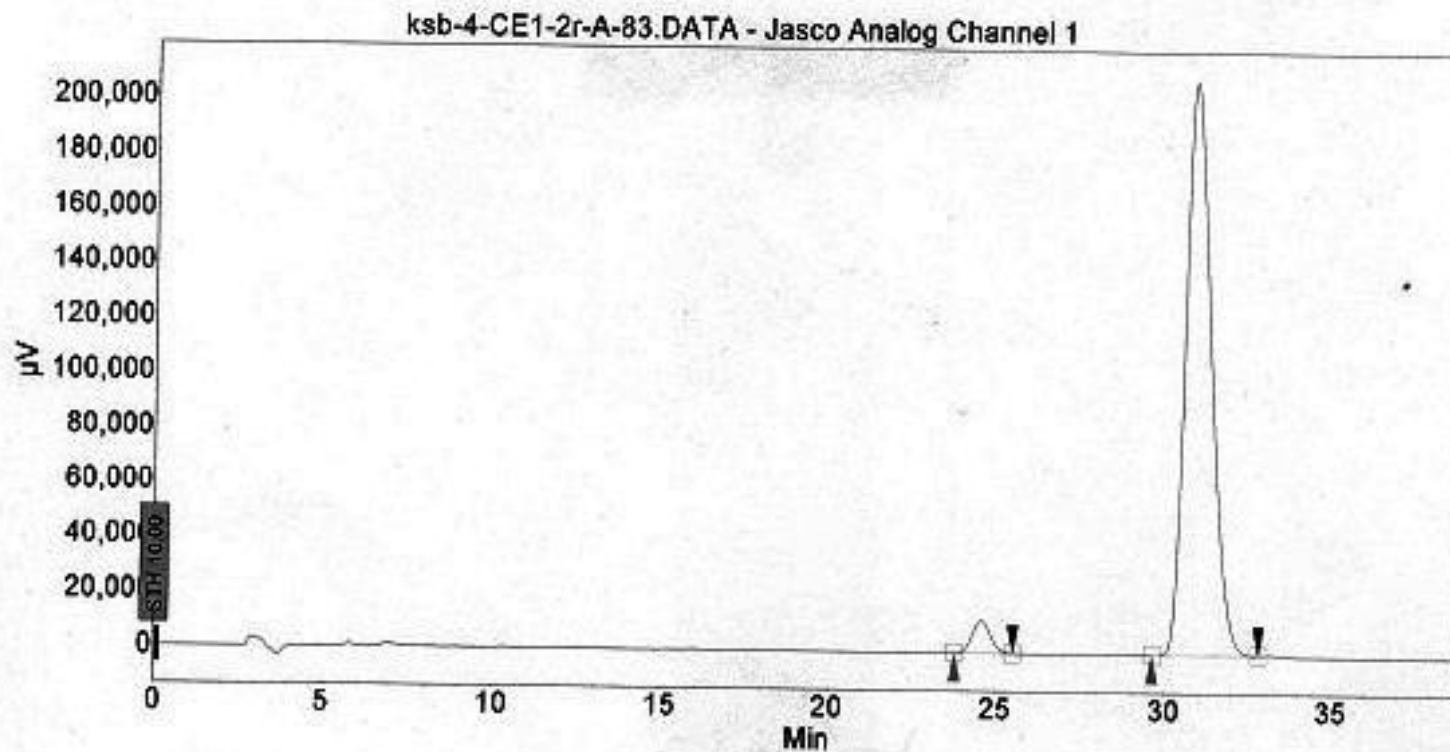
# Chromatogram : ksb-4-CE1-2r-A-83\_channel2

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4a

Acquired : 6/12/2013 6:07:49 PM  
Processed : 6/12/2013 6:51:34 PM  
Printed : 6/12/2013 6:52:10 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	24.567	3.83	11689.9	7317.4	3.833
2	UNKNOWN	30.800	96.17	208287.6	183580.6	96.167
Total			100.00	219977.5	190898.0	100.000

Fig S77. HPLC Profile of Enantioenriched 4a

```

NAME      INN-4-KSB-80B-1H
EXPNO     5
PROCNO    1
Date_     20130613
Time      20.01
INSTRUM   spect
PROBHD    5 mm DABBO 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         294.6 K
D1         1.00000000 sec
TD0        1

```

```

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

```

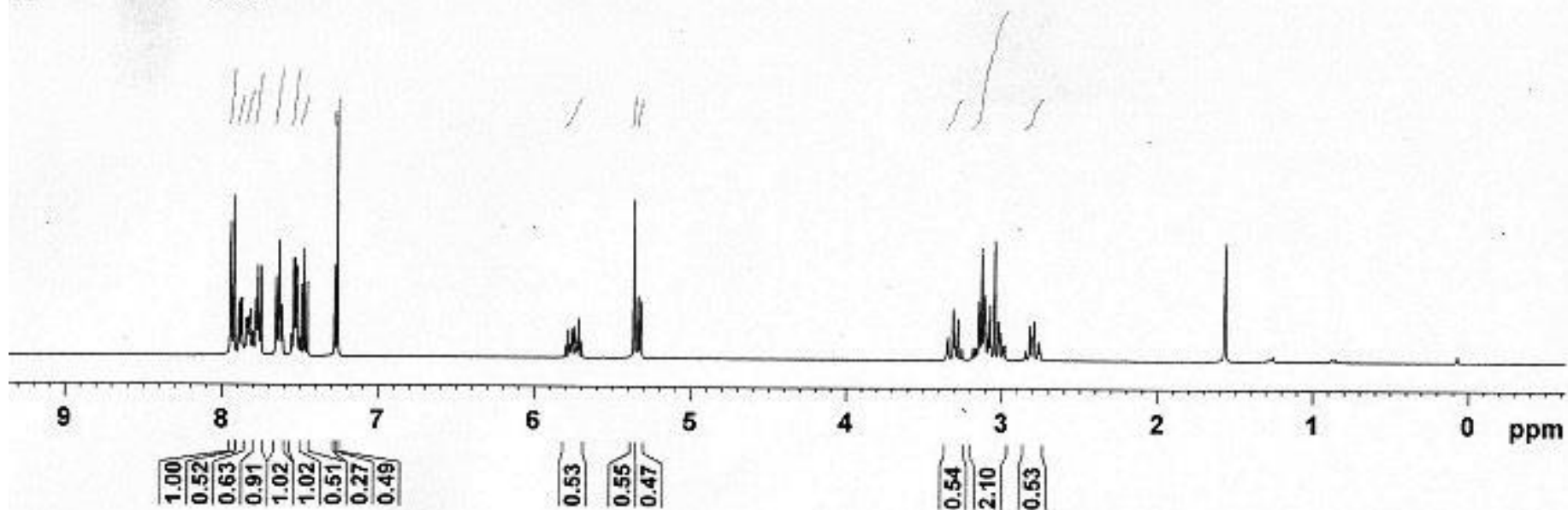
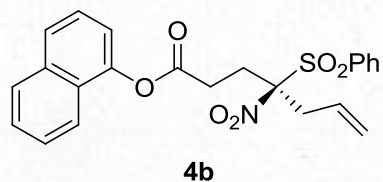


Fig 78. <sup>1</sup>H NMR Spectrum of 4b

```

NAME      inn-4-ksb-80b-13c
EXPNO     1
PROCNO    1
Date_     20130616
Time      18.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         200
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         294.3 K
D1         1.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

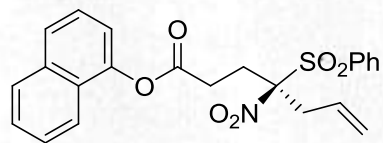
----- CHANNEL f1 -----
NUC1       13C
P1         8.75 usec
PL1        -2.00 dB
PL1W       56.53121948 W
SFO1       100.6226298 MHz

```

```

----- 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          100.6127565 MHz
WDM         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```



4b

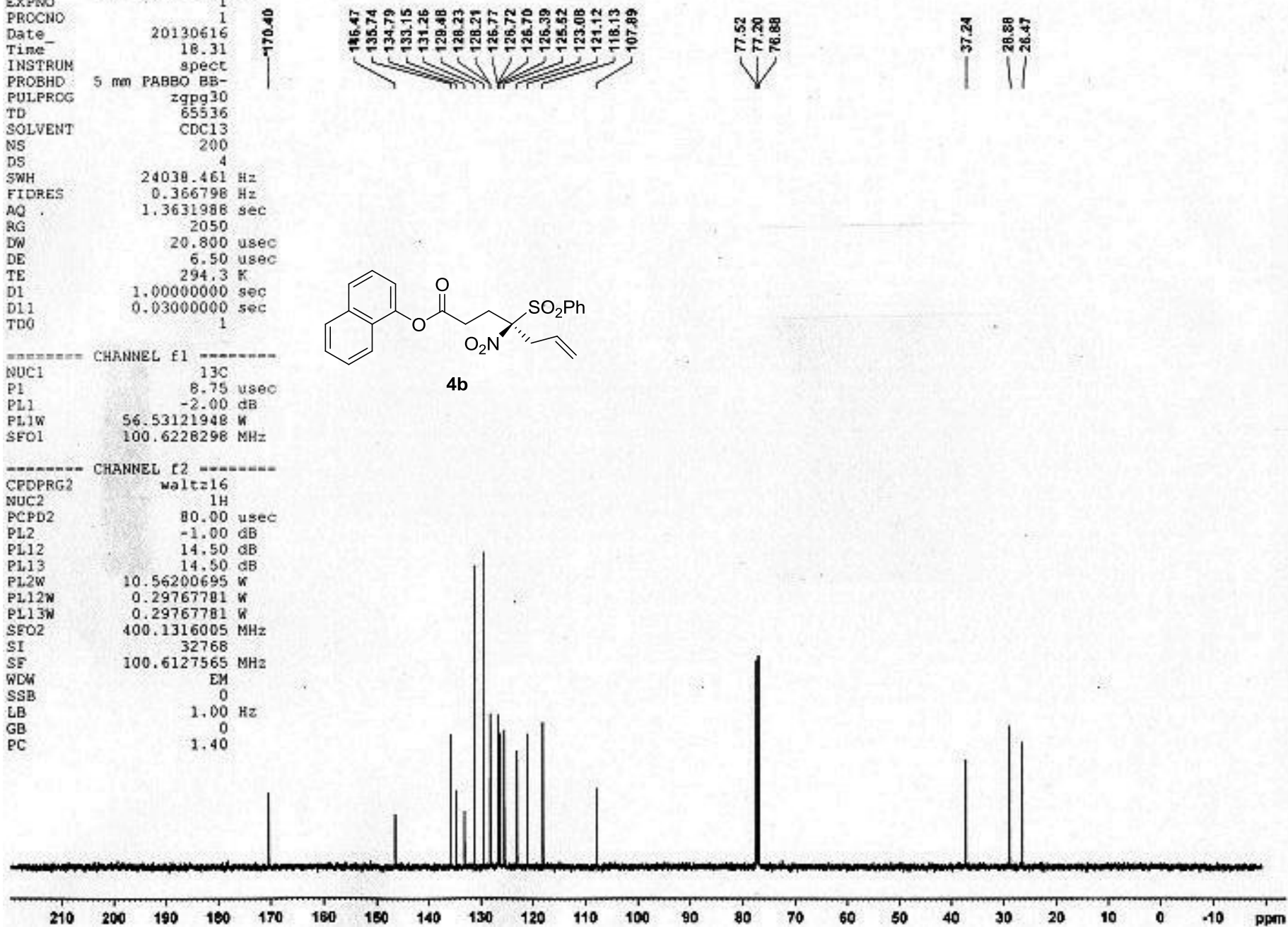
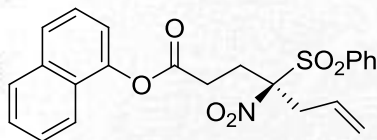


Fig S79. <sup>13</sup>C NMR Spectrum of 4b

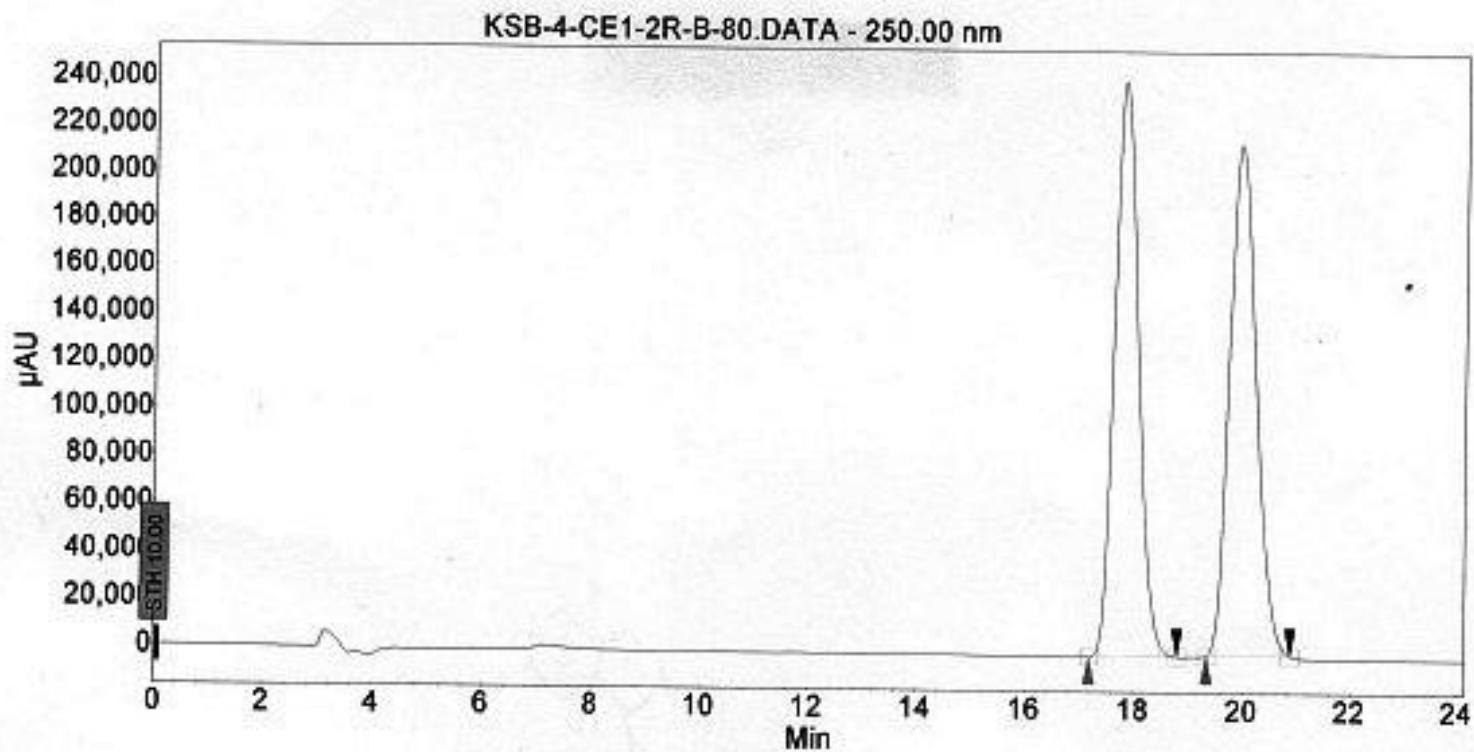
# Chromatogram : KSB-4-CE1-2R-B-80\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4b

Acquired : 6/12/2013 10:52:56 PM  
Processed : 6/12/2013 11:21:53 PM  
Printed : 6/12/2013 11:22:04 PM



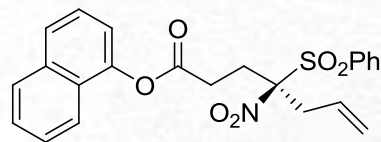
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	17.785	49.97	242702.2	122582.1	49.969
2	UNKNOWN	19.932	50.03	215389.5	122733.6	50.031
Total			100.00	458091.6	245315.7	100.000

Fig S80. HPLC Profile of Racemic 4b

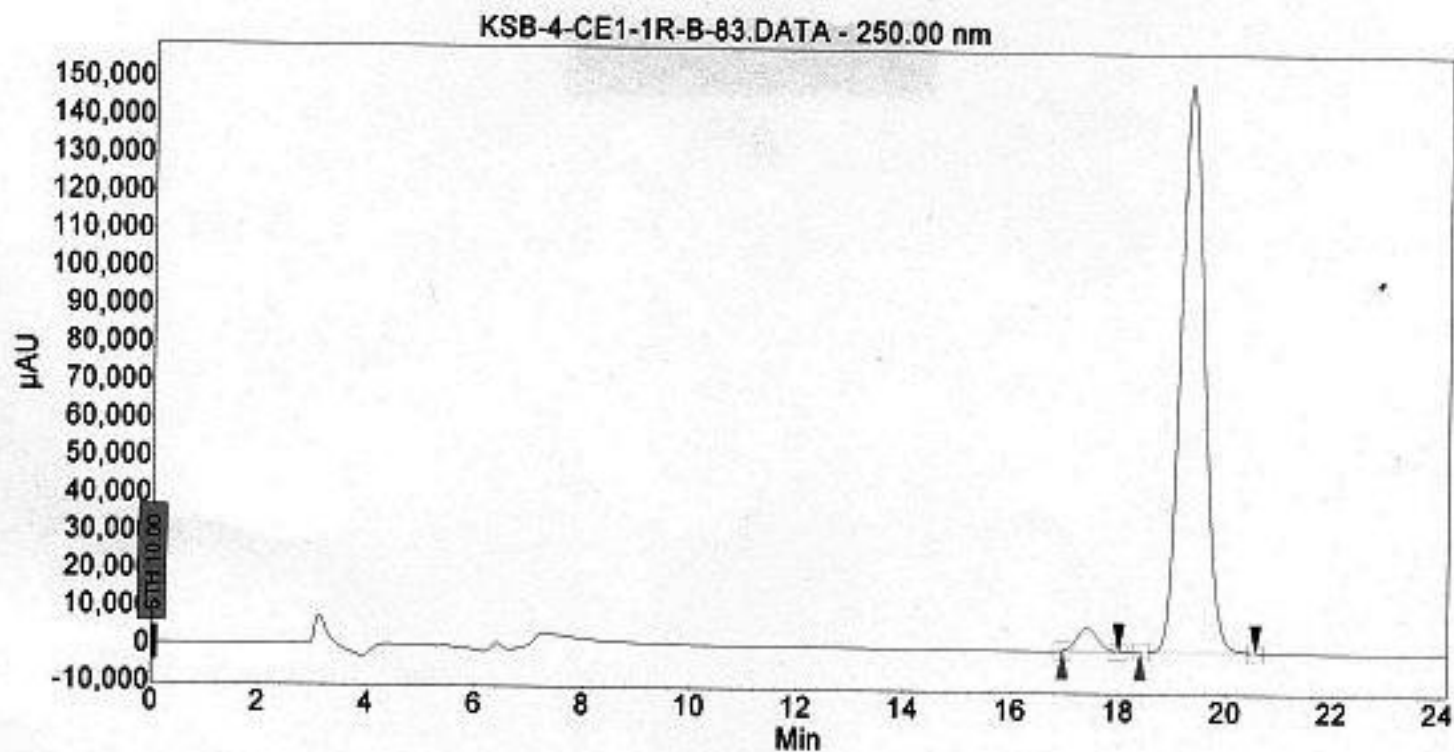
# Chromatogram : KSB-4-CE1-1R-B-83\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4b

Acquired : 6/12/2013 9:50:11 PM  
Processed : 6/12/2013 10:16:53 PM  
Printed : 6/12/2013 11:12:24 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	17.399	3.40	6222.6	2722.8	3.397
1	UNKNOWN	19.279	96.60	150631.7	77435.2	96.603
Total			100.00	156854.2	80158.0	100.000

Fig S81. HPLC Profile of Enantioenriched 4b

```

NAME inn-4-ksb-79b-1h
EXPNO 35
PROCNO 1
Date_ 20130612
Time_ 14.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 57690
SOLVENT CDCl3
NS 12
DS 0
SWH 9615.385 Hz
FIDRES 0.166673 Hz
AQ 2.9999299 sec
RG 128
DW 52.000 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

```

```

===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 10.56200695 W
SFO1 400.1320007 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

```

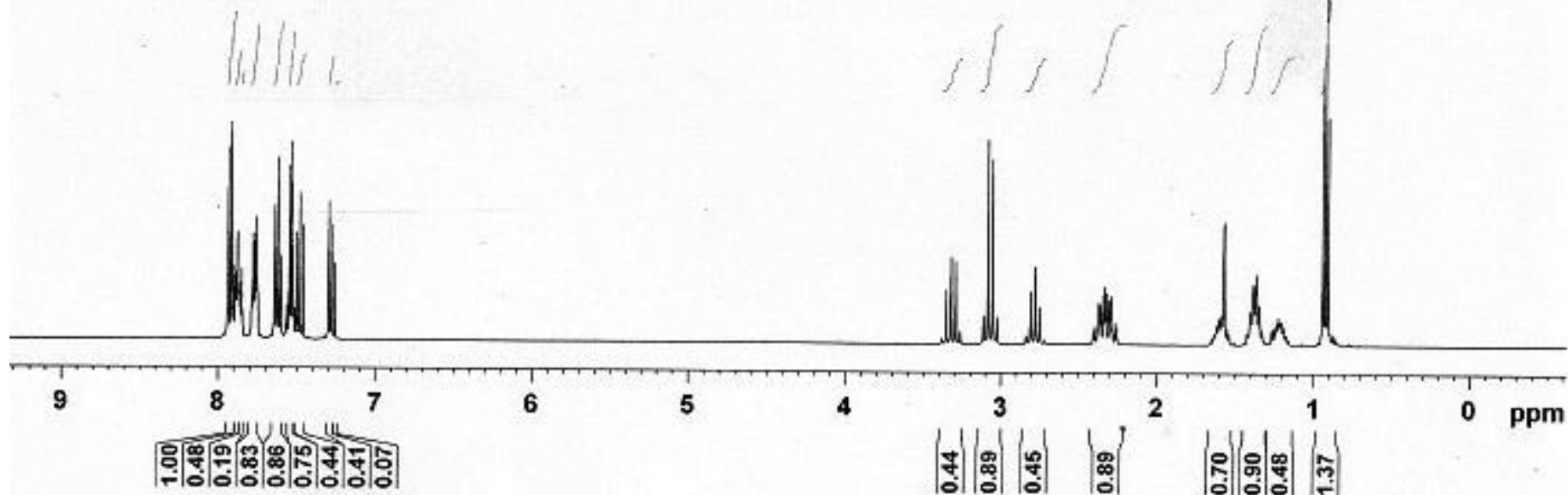
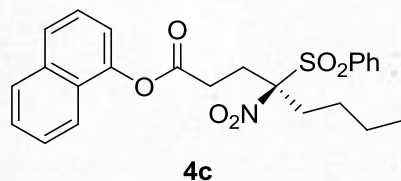


Fig S82. <sup>1</sup>H NMR Spectrum of 4c

```

NAME      inn-4-ksb-79b-13c
EXPNO     36
PROCNO    1
Date_     20130612
Time      15.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        70
DS        2
SWH       27777.777 Hz
FIDRES    0.423855 Hz
AQ        1.1796980 sec
RG        912
DN        18.000 usec
DE        6.50 usec
TE        295.7 K
D1        1.00000000 sec
D11       0.03000000 sec
TDO       1

```

```

===== CHANNEL f1 =====
NUC1      13C
P1        8.75 usec
PL1       -2.00 dB
PL1W      56.53121948 W
SFO1      100.6242389 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        100.6127543 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

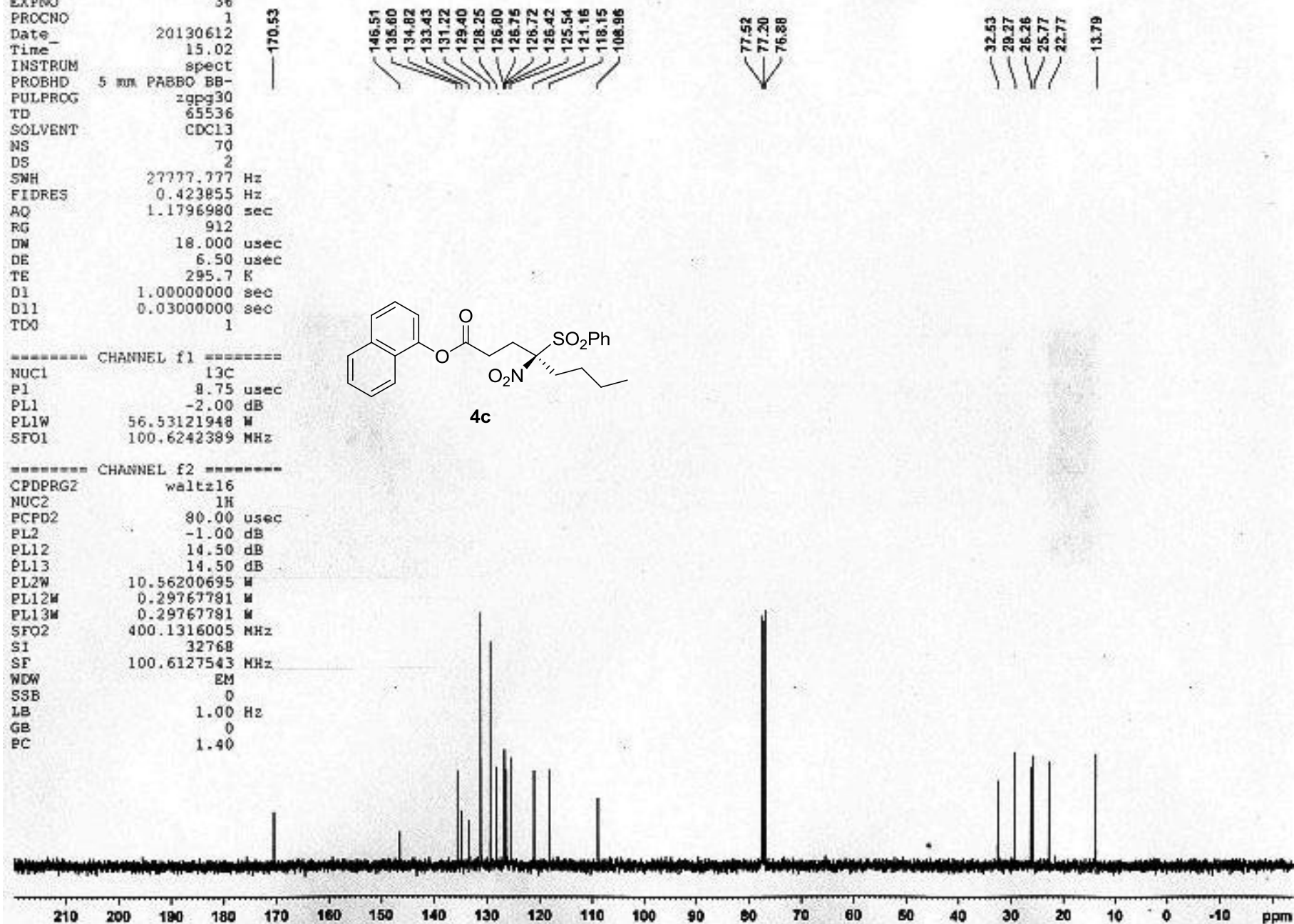
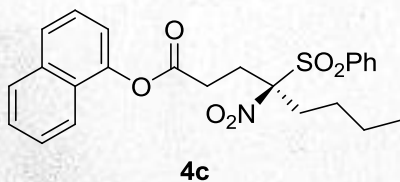


Fig S83. <sup>13</sup>C NMR Spectrum of 4c

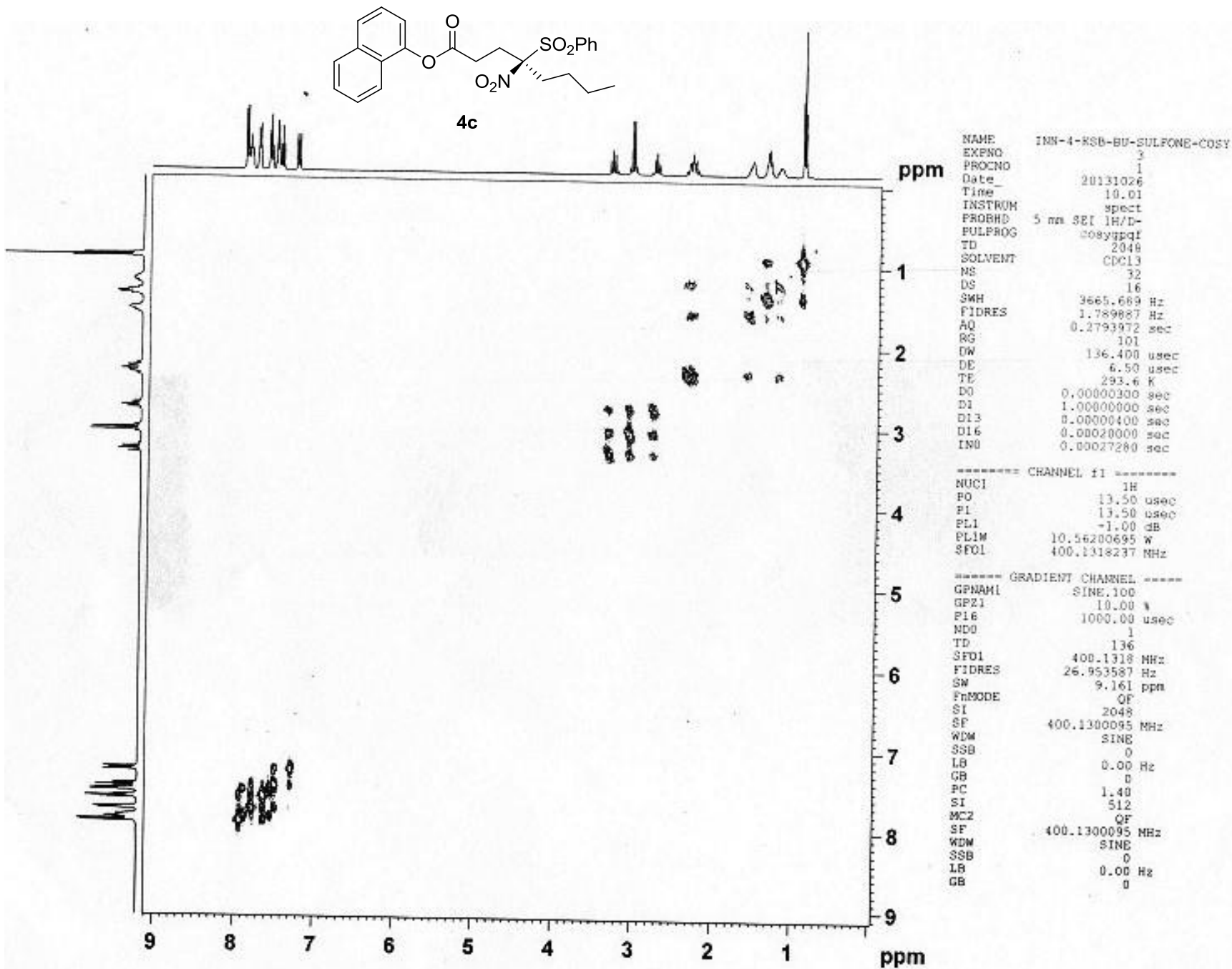


Fig S84.  $^1\text{H}$ - $^1\text{H}$  COSY NMR Spectrum of 4c



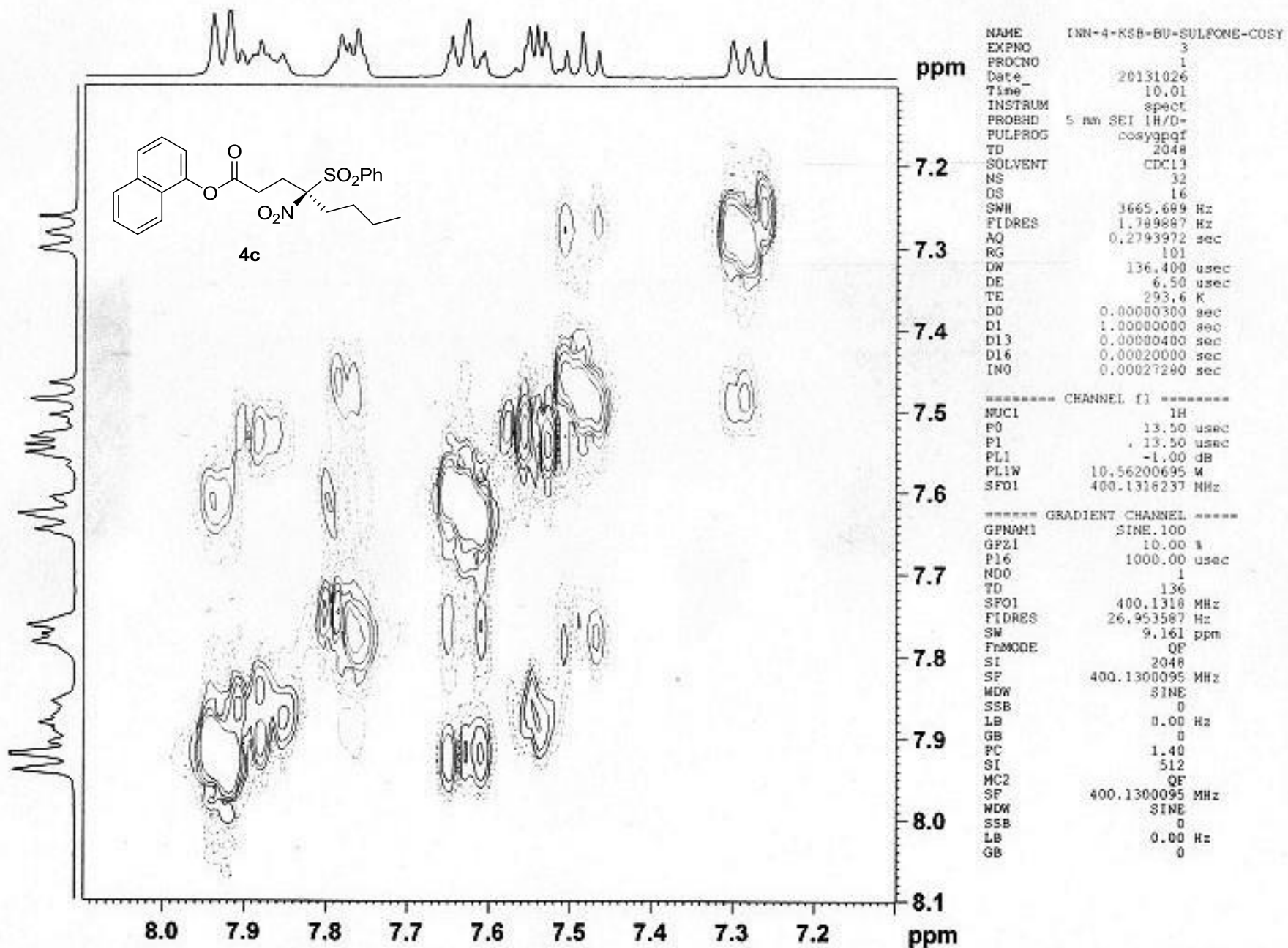
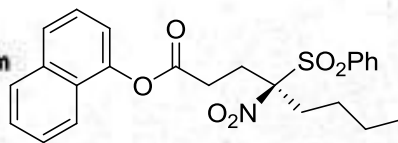


Fig S85. Expanded  $^1\text{H}$ - $^1\text{H}$  COSY NMR Spectrum of 4c

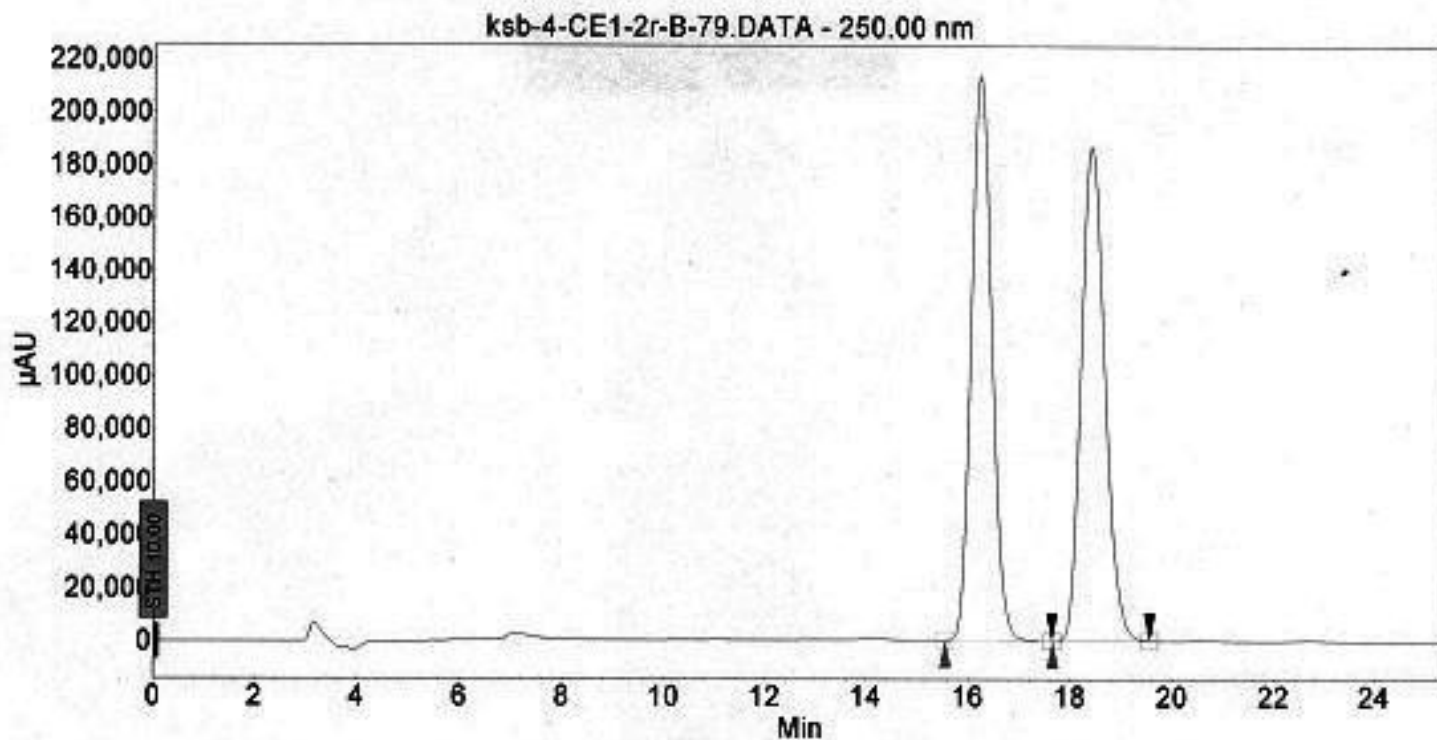
# Chromatogram : ksb-4-CE1-2r-B-79\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4c

Acquired : 6/13/2013 1:31:47 AM  
Processed : 6/13/2013 1:57:40 AM  
Printed : 6/13/2013 1:59:53 AM



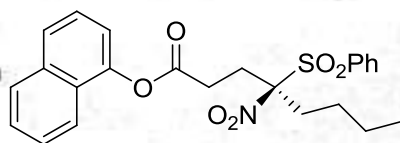
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	16.212	49.72	214001.3	99223.7	49.719
2	UNKNOWN	18.385	50.28	187171.4	100344.7	50.281
Total			100.00	401172.7	199568.4	100.000

Fig S86. HPLC Profile of Racemic 4c

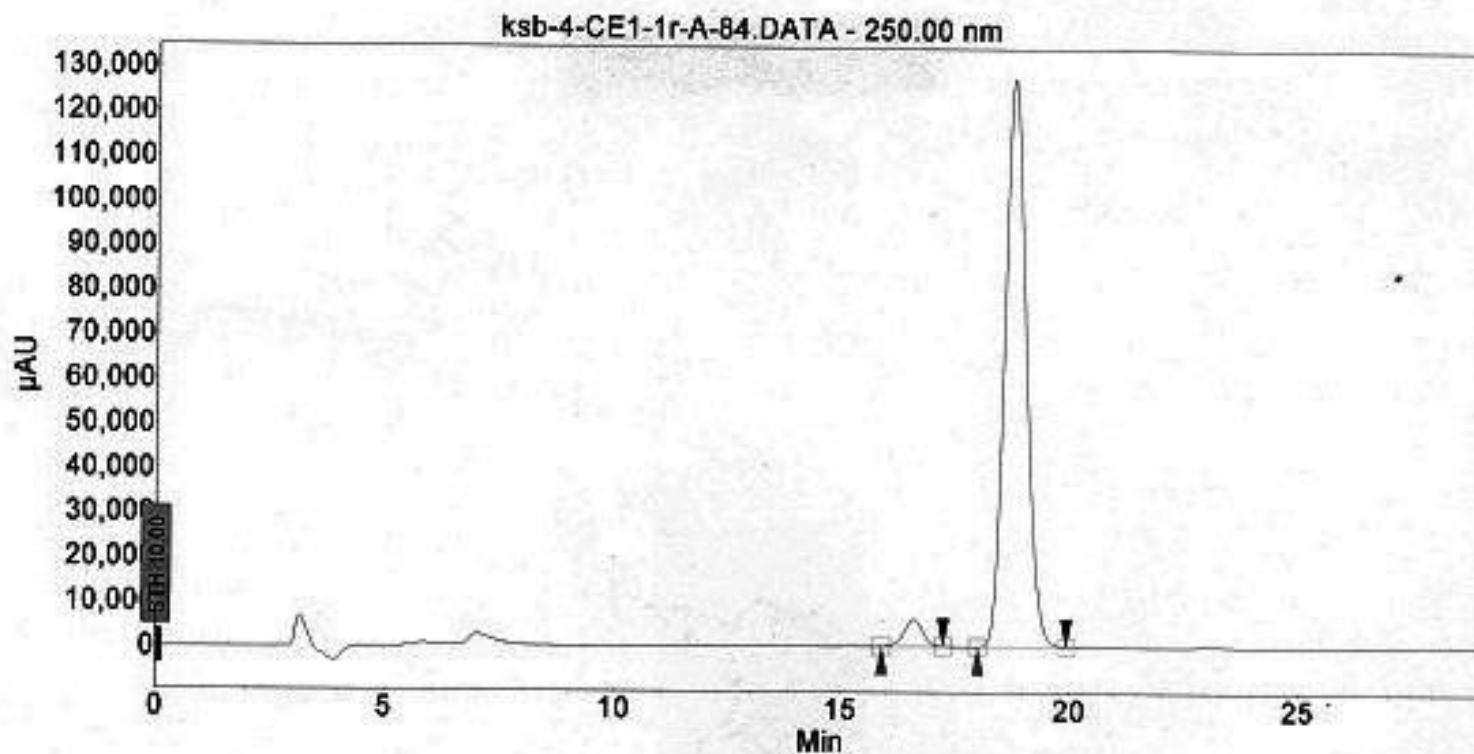
# Chromatogram : ksb-4-CE1-1r-A-84\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4c

Acquired : 6/13/2013 12:57:13 AM  
Processed : 6/13/2013 1:26:41 AM  
Printed : 6/13/2013 1:28:38 AM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU Min]	Area % [%]
1	UNKNOWN	16.585	4.02	6052.8	2856.5	4.016
2	UNKNOWN	18.732	95.98	127532.0	68277.0	95.984
Total			100.00	133584.8	71133.5	100.000

Fig S87. HPLC Profile of Enantioenriched 4c

```

NAME      INN-4-KSB-84B-1H
EXPNO     10
PROCNO    1
Date      20130615
Time      8.42
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        7
DS        0
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        57
DN        60.800 usec
DE        6.50 usec
TE        292.2 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 MH:
SI        32768
SF        400.1300095 MH:
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

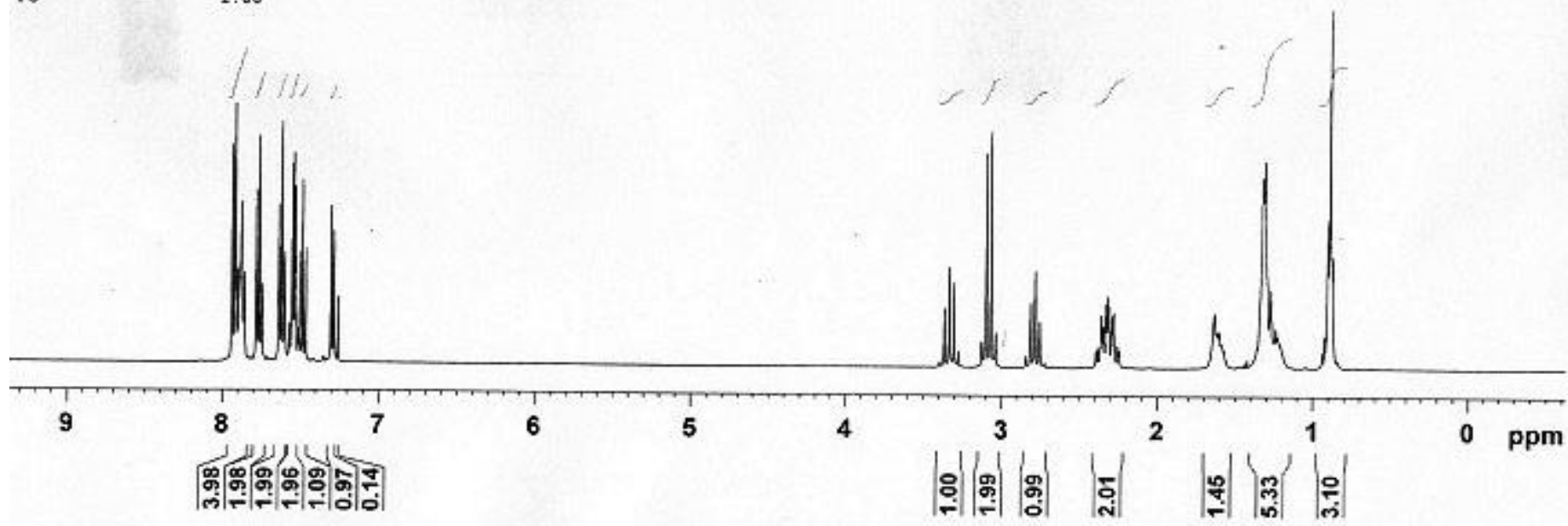
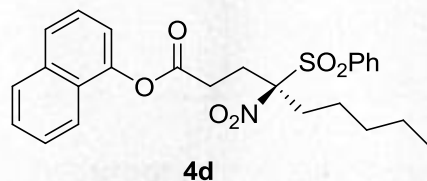


Fig S88. <sup>1</sup>H NMR Spectrum of 4d

```

NAME-      INN-4-KSB-84B-13C
EXPNO      10
PROCNO     1
Date_      20130615
Time       8.44
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         42
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         292.8 K
d1         1.00000000 sec
d11        0.03000000 sec
TDO        1

```

```

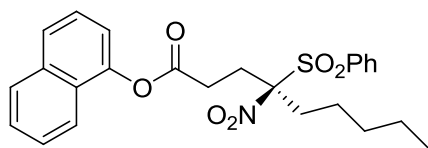
----- CHANNEL f1 -----
NUC1       13C
P1         8.75 usec
PL1        -2.00 dB
PL1W       56.53121948 W
SFO1       100.6228298 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 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         100.6127595 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```



4d

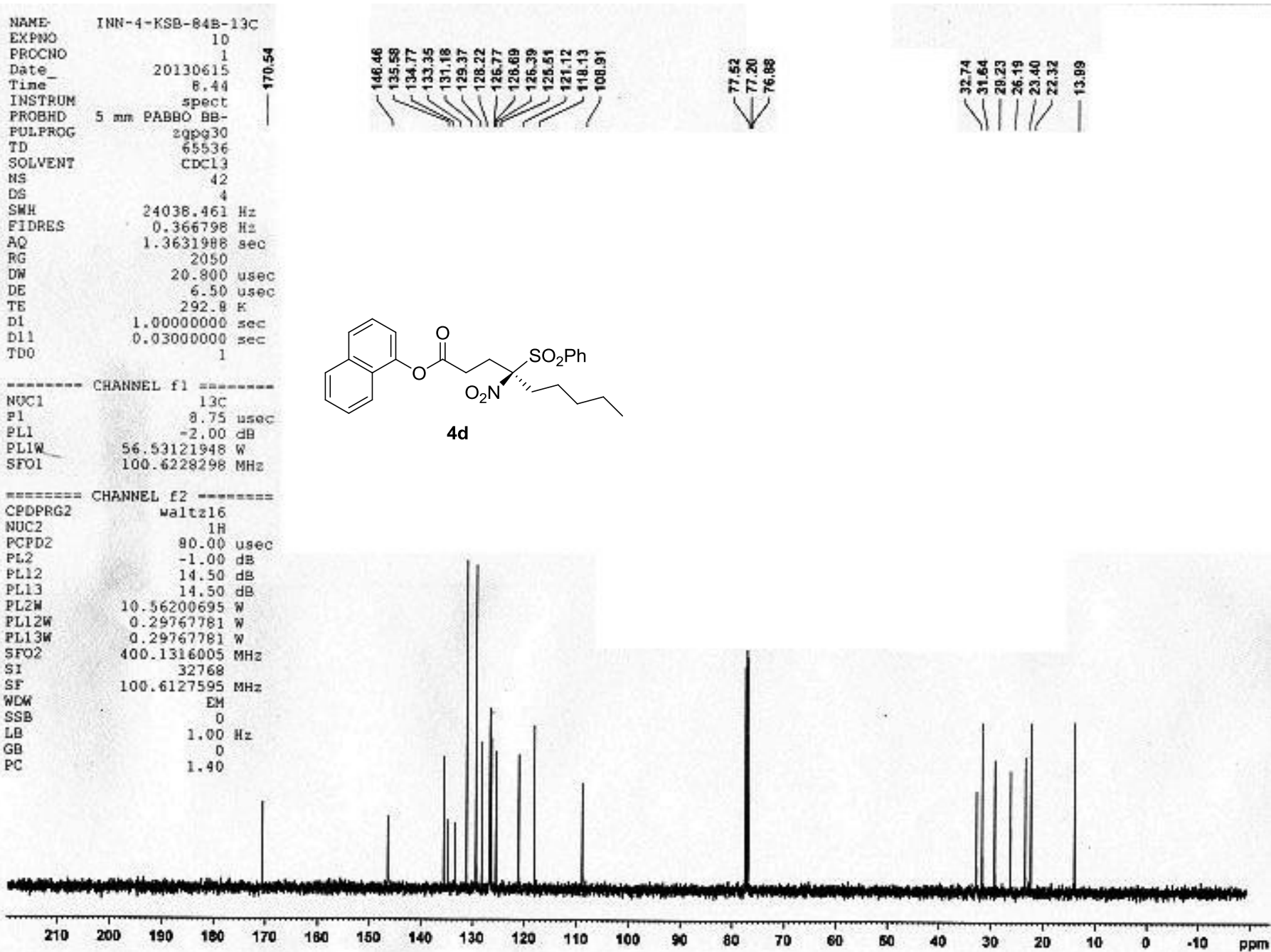
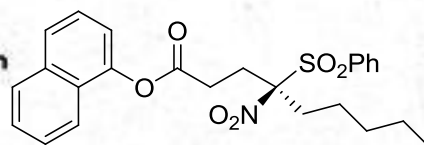


Fig S89. <sup>13</sup>C NMR Spectrum of 4d

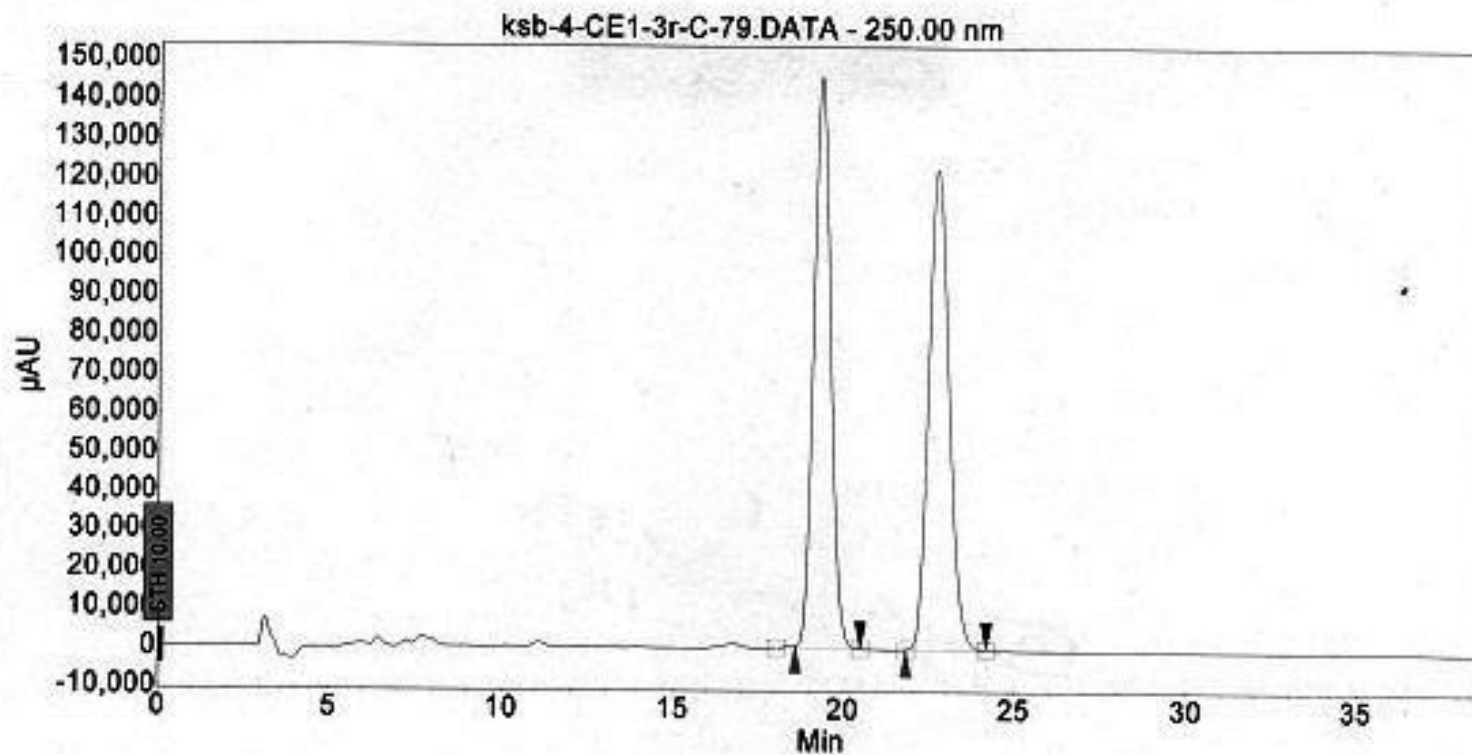
# Chromatogram : ksb-4-CE1-3r-C-79\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4d

Acquired : 6/13/2013 12:08:49 PM  
Processed : 6/13/2013 12:48:00 PM  
Printed : 6/13/2013 12:49:17 PM



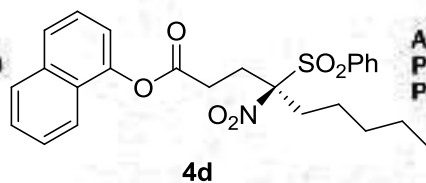
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	19.279	50.00	148037.0	84440.8	50.000
2	UNKNOWN	22.692	50.00	122963.1	84440.3	50.000
Total			100.00	269000.2	168881.1	100.000

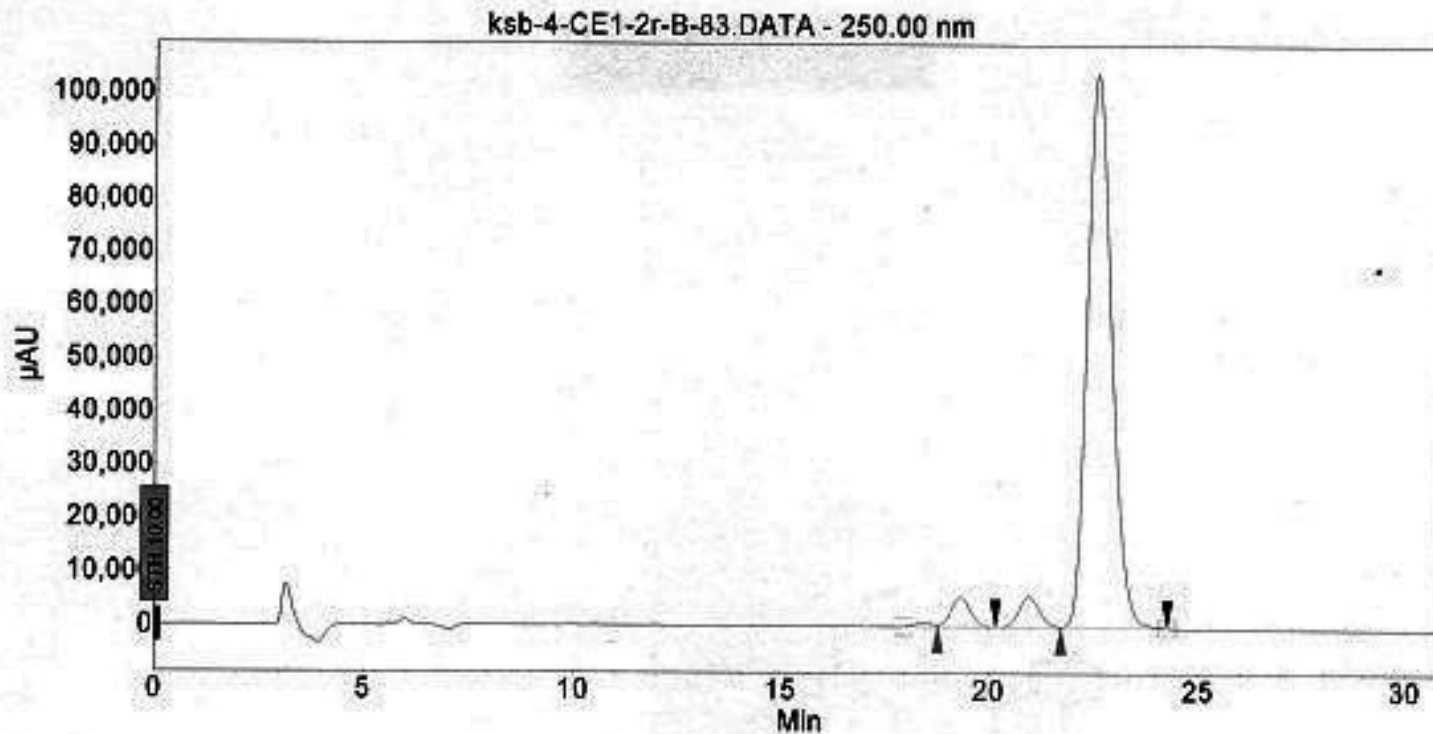
Fig S90. HPLC Profile of Racemic 4d

# Chromatogram : ksb-4-CE1-2r-B-83\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



Acquired : 6/13/2013 11:36:20 AM  
Processed : 6/13/2013 12:08:02 PM  
Printed : 6/13/2013 12:49:27 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	19.332	4.07	5499.6	2971.1	4.069
2	UNKNOWN	22.558	95.93	104090.3	70052.2	95.931
Total			100.00	109590.0	73023.3	100.000

Fig S91. HPLC Profile of Enantioenriched 4d

NAME INN-4-KSB-79A-1h  
 EXPNO 15  
 PROCNO 1  
 Date\_ 20130612  
 Time\_ 11.27  
 INSTRUM spect  
 PROBHD 5 mm PABBO HD-  
 PULPROG zg30  
 TD 49340  
 SOLVENT CDCl3  
 NS 27  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.166674 Hz  
 AQ 2.9999220 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.0 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 400.1300094 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

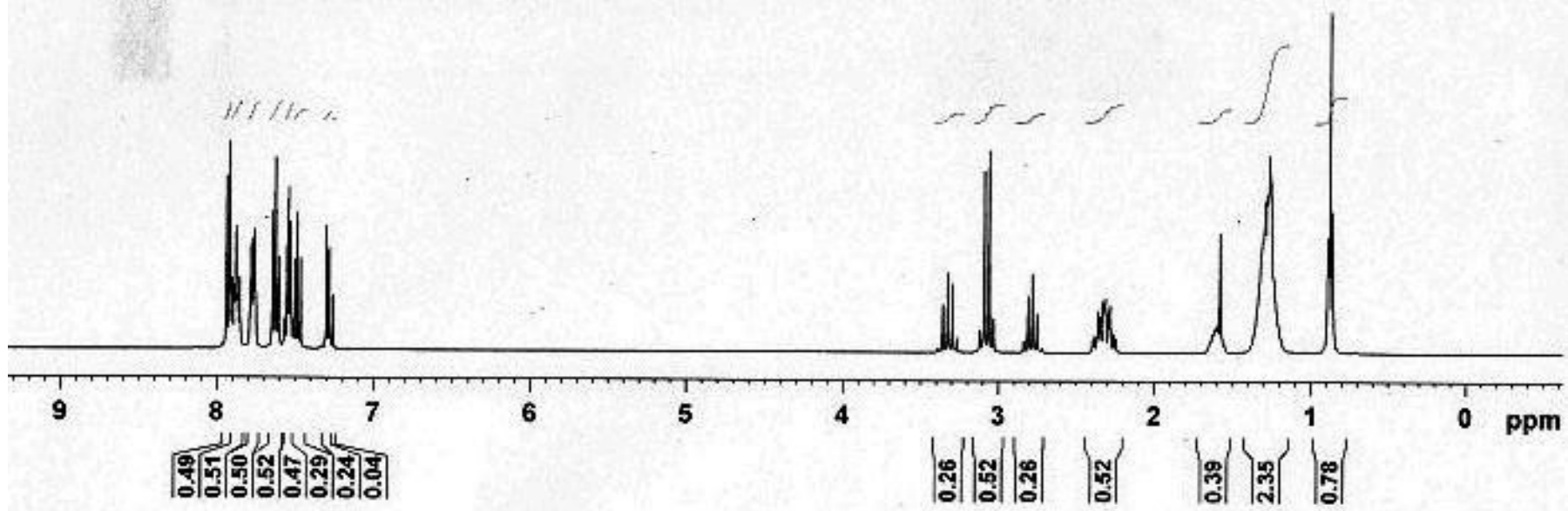
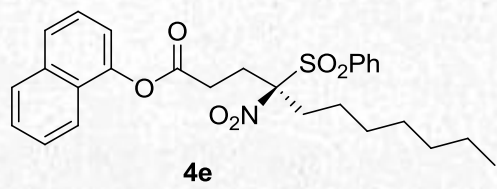


Fig S92. <sup>1</sup>H NMR Spectrum of 4e



```

NAME      INN-4-KSB-79A-13C
EXPNO     4
PROCNO    1
Date_     20130616
Time      17.38
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS        180
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        293.4 K
D1        1.00000000 sec
D11       0.03000000 sec
TDO       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        100.6127567 MHz
WDM       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

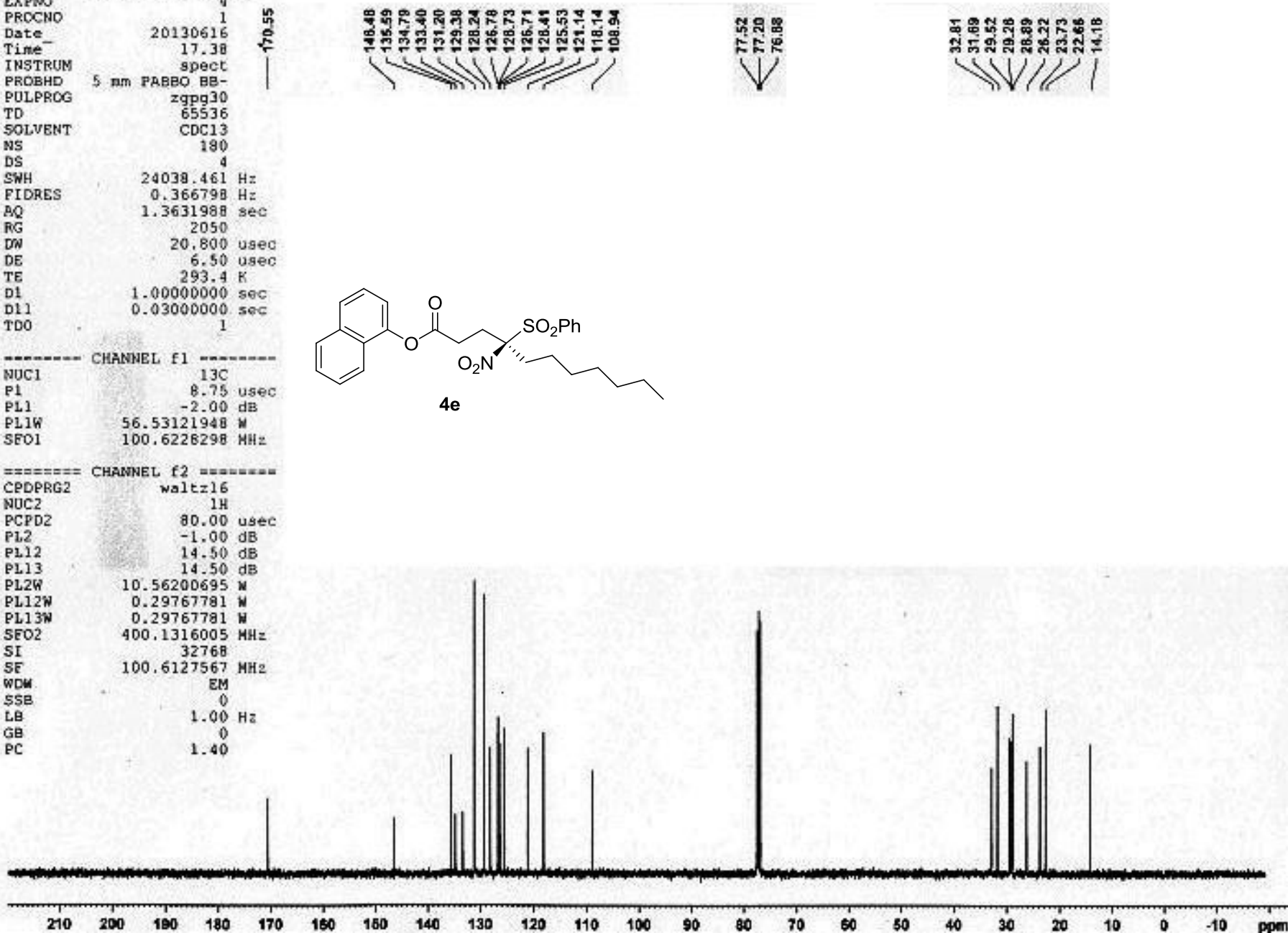
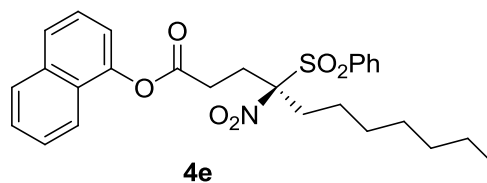
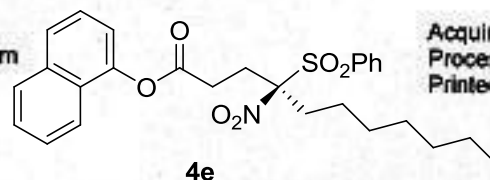


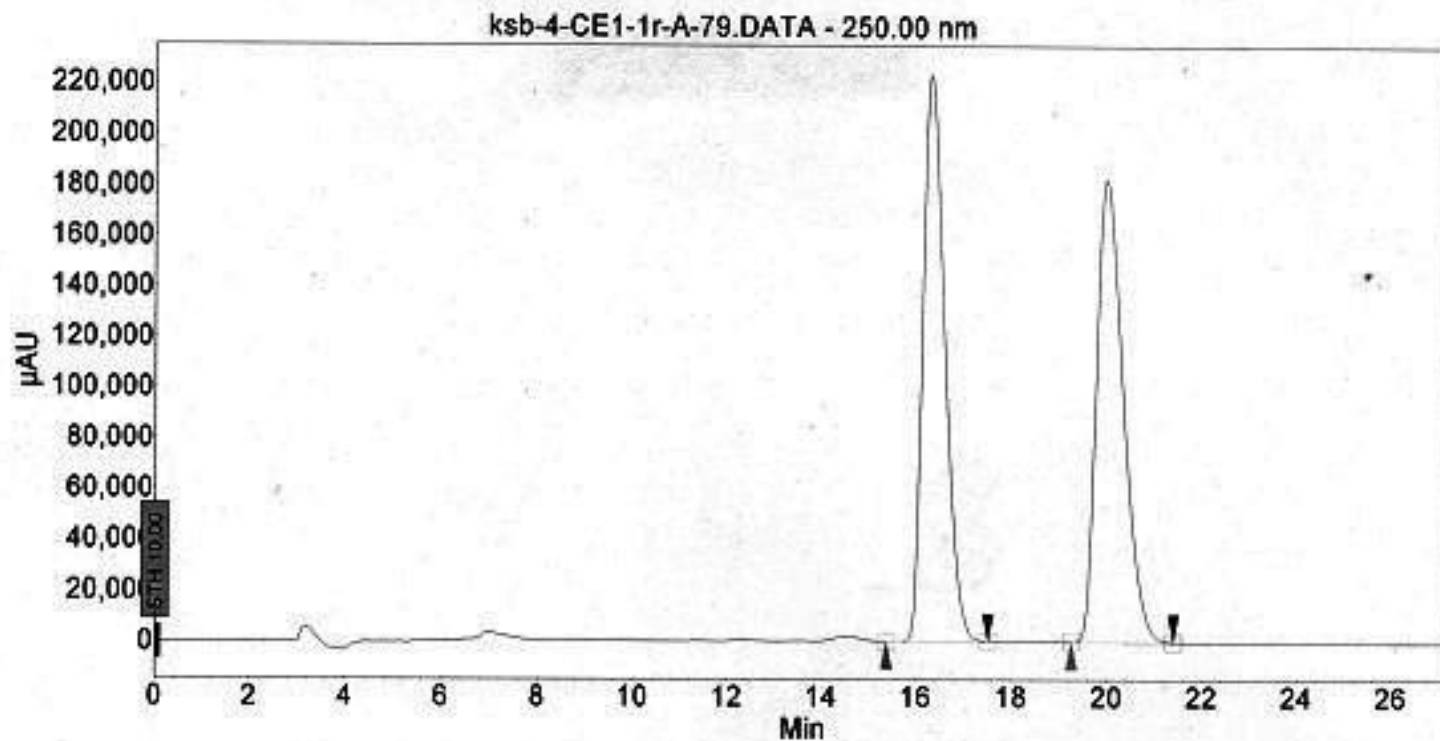
Fig S93. <sup>13</sup>C NMR Spectrum of 4e

# Chromatogram : ksb-4-CE1-1r-A-79\_channel4

System : Jasco HPLC  
 Method : ksb-20%-1f-250nm  
 User : User1



Acquired : 6/13/2013 1:58:46 AM  
 Processed : 6/13/2013 2:26:24 AM  
 Printed : 6/13/2013 2:48:41 AM



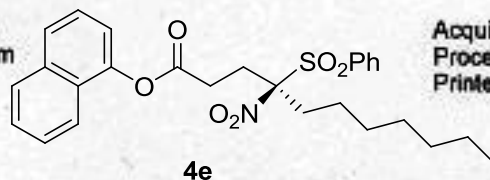
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	16.292	50.04	223315.5	118559.6	50.037
2	UNKNOWN	19.959	49.96	182832.6	118382.7	49.963
Total			100.00	406148.1	236942.3	100.000

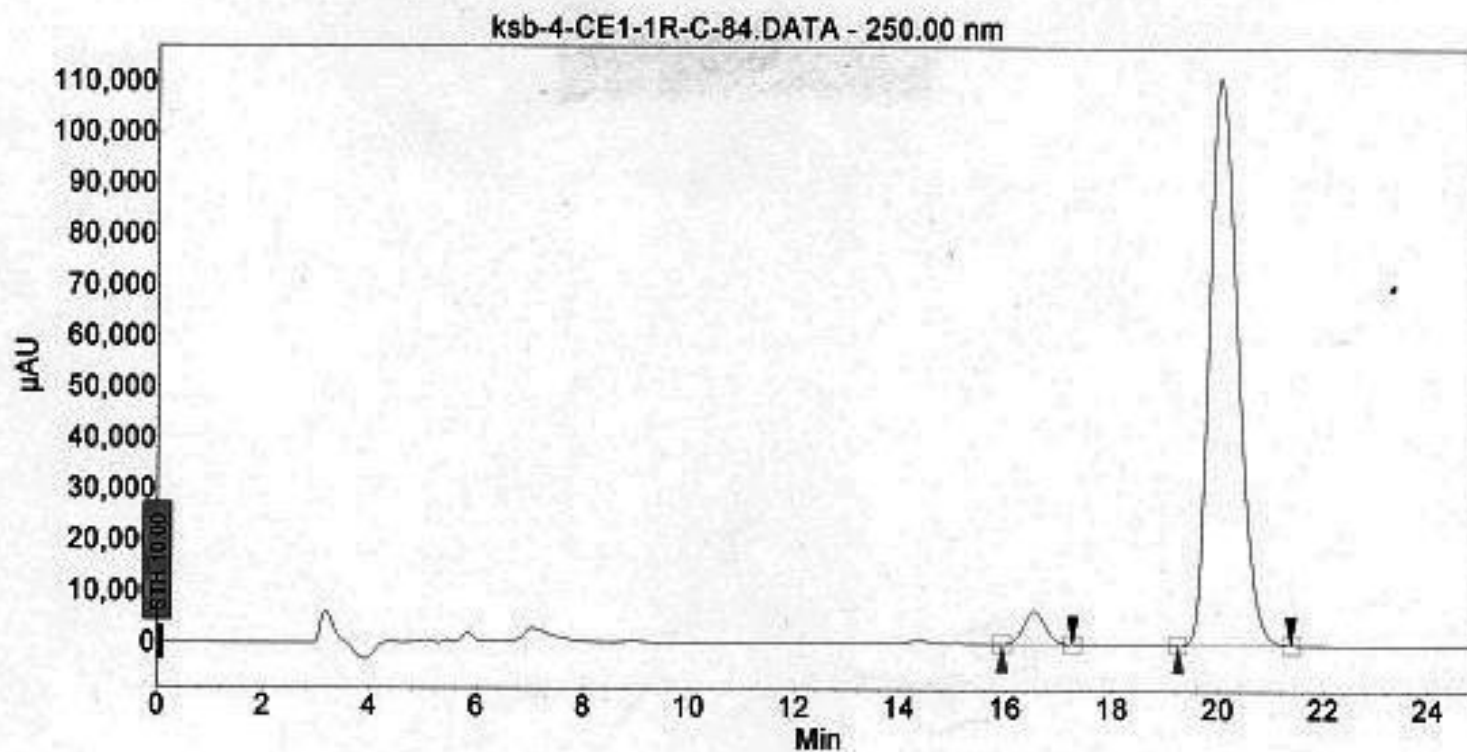
Fig S94. HPLC Profile of Racemic 4e

# Chromatogram : ksb-4-CE1-1R-C-84\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



Acquired : 6/13/2013 2:28:54 AM  
Processed : 6/13/2013 2:54:31 AM  
Printed : 6/13/2013 2:56:34 AM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	16.532	4.20	6403.6	2993.4	4.202
2	UNKNOWN	20.012	95.80	111026.2	68248.4	95.798
Total			100.00	117429.8	71241.8	100.000

Fig S95. HPLC Profile of Enantioenriched 4e

```

NAME      INN-4-KSB-B0C-1H
EXPNO     5
PROCNO    1
Date_     20130613
Time      20.10
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
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         294.5 K
D1         1.00000000 sec
ID0        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

```

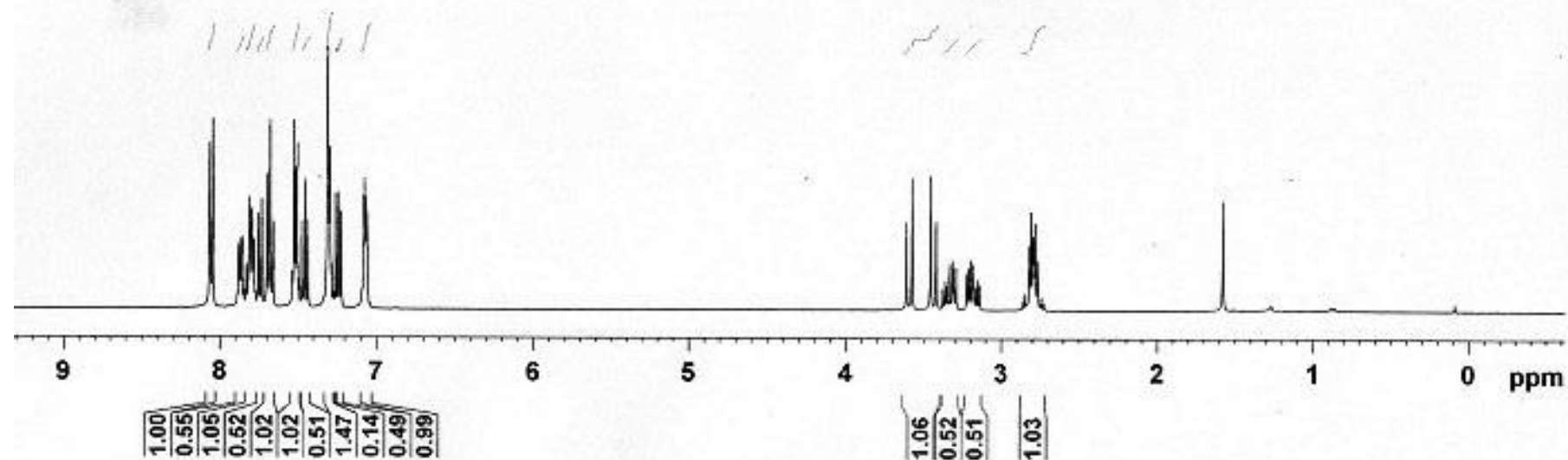
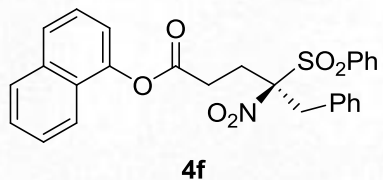


Fig S96. <sup>1</sup>H NMR Spectrum of 4f

```

NAME      INN-4-KSB-80C-13C
EXPNO     5
PROCNO    1
Date_     20130613
Time      20.12
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         62
DS         4
SWH        24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         2050
DM         20.800 usec
DE         6.50 usec
TE         294.9 K
D1         1.00000000 sec
D11        0.03000000 sec
TDO       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         100.6127556 MHz
WDM        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```

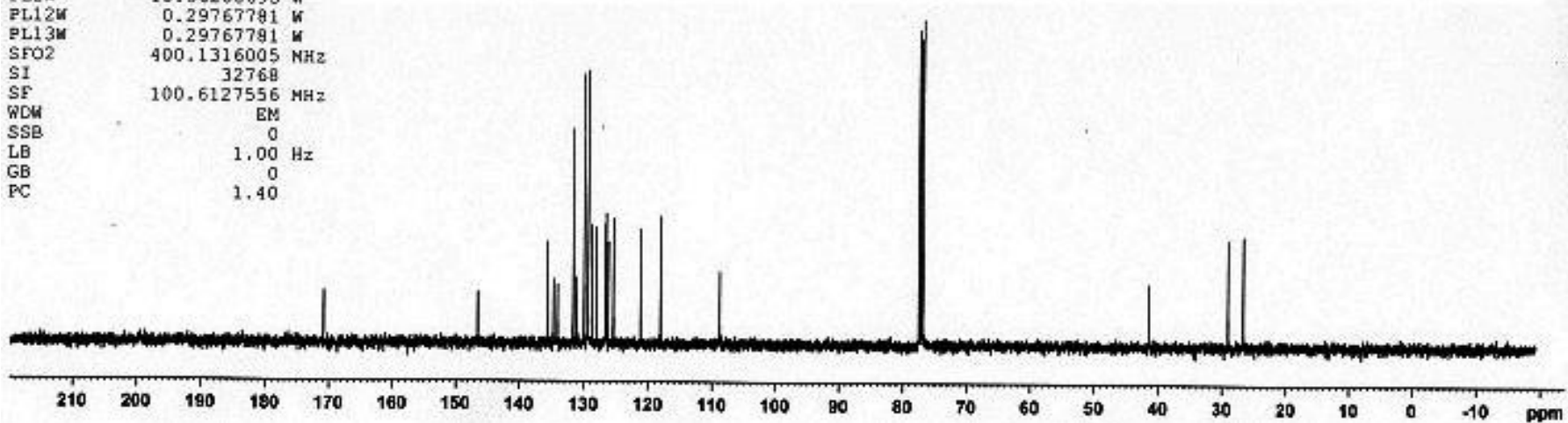
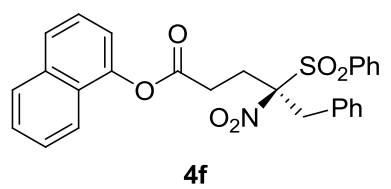
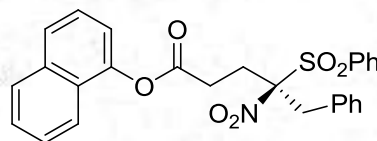


Fig S97. <sup>13</sup>C NMR Spectrum of 4f

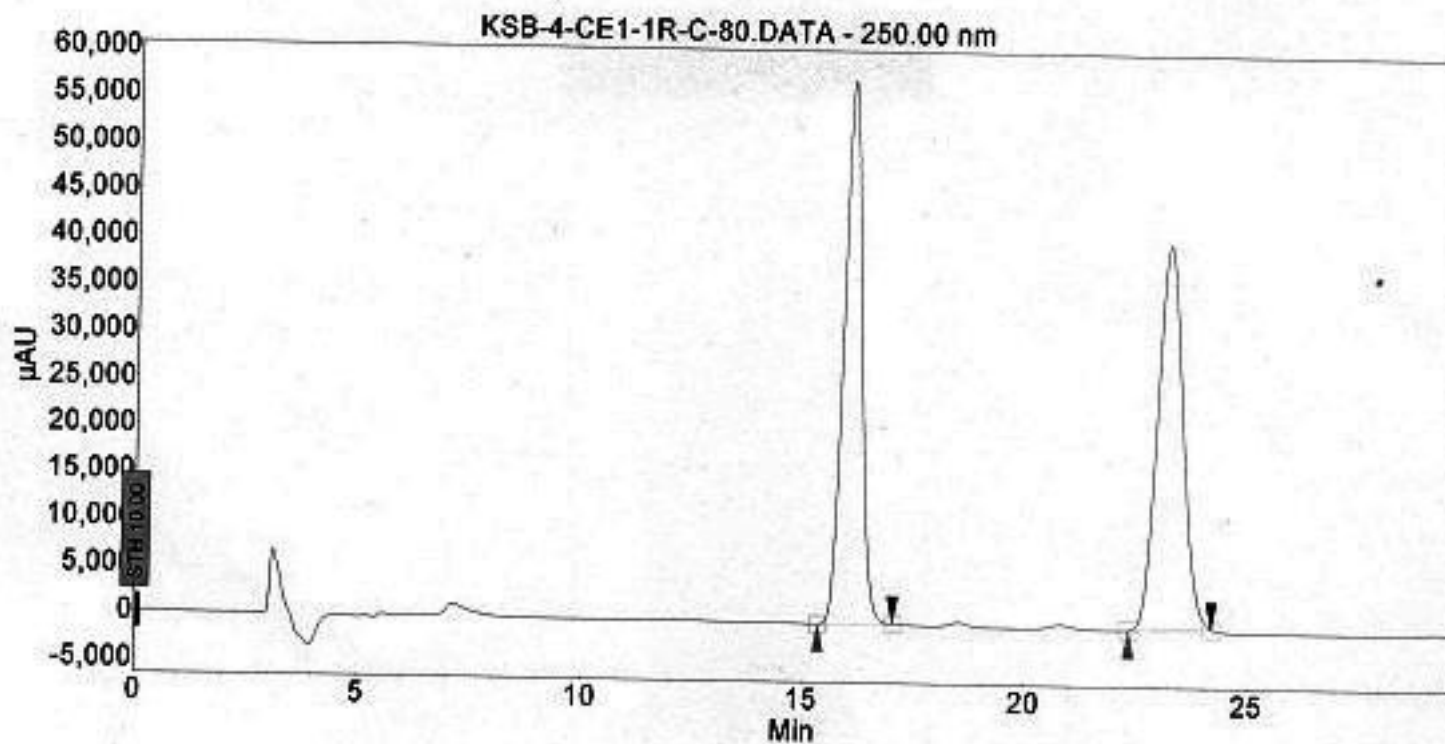
# Chromatogram : KSB-4-CE1-1R-C-80\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4f

Acquired : 6/12/2013 11:20:04 PM  
Processed : 6/12/2013 11:52:23 PM  
Printed : 6/12/2013 11:52:31 PM



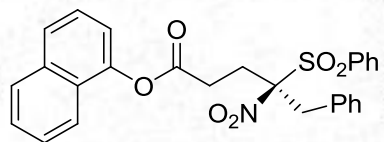
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	16.026	50.00	57740.0	27055.0	49.999
2	UNKNOWN	23.158	50.00	40910.7	27056.0	50.001
Total			100.00	98650.7	54111.0	100.000

Fig S98. HPLC Profile of Racemic 4f

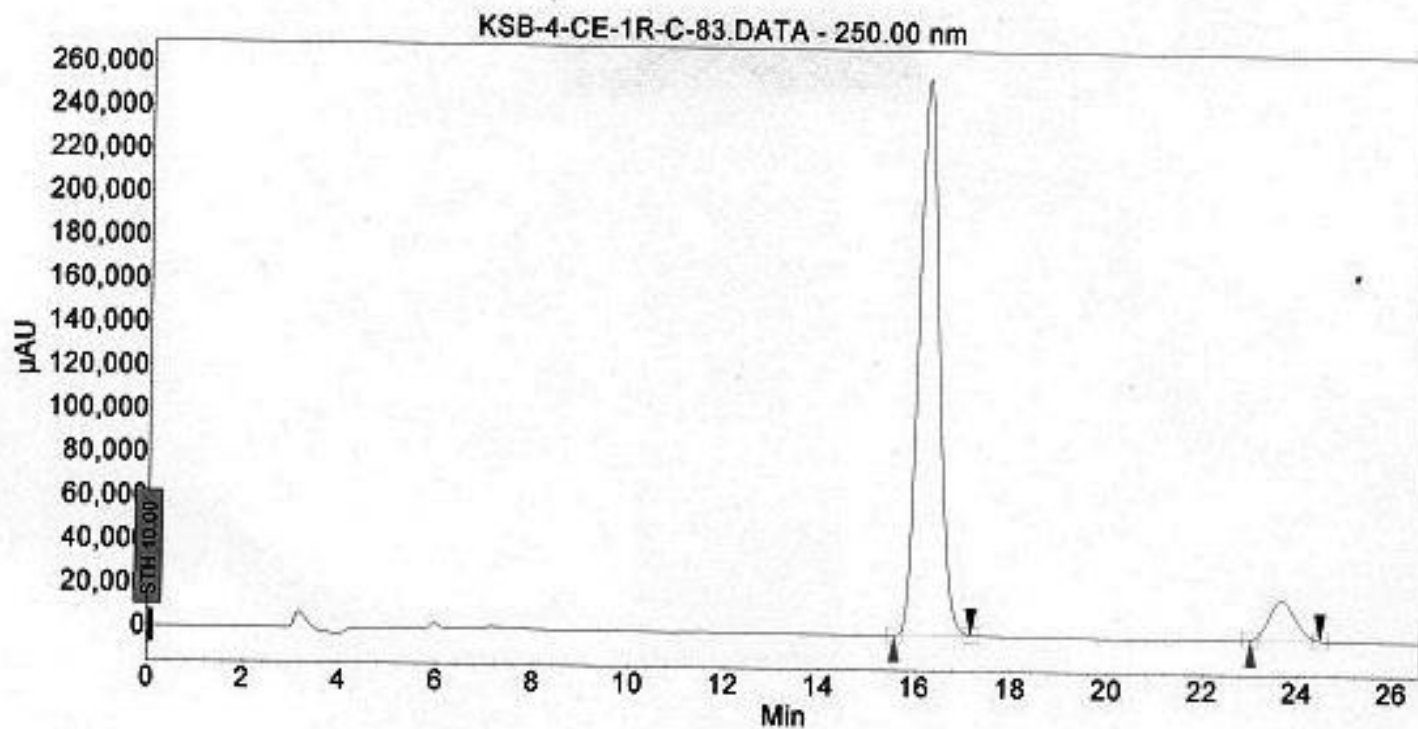
# Chromatogram : KSB-4-CE-1R-C-83\_channel4

System : Jasco HPLC  
Method : ksb-20%-1f-250nm  
User : User1



4f

Acquired : 6/12/2013 11:51:09 PM  
Processed : 6/13/2013 12:20:07 AM  
Printed : 6/13/2013 12:20:16 AM



## Peak results :

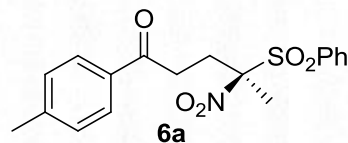
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	16.185	91.36	258465.1	124937.0	91.363
2	UNKNOWN	23.638	8.64	18332.0	11810.2	8.637
Total			100.00	274797.1	136747.2	100.000

Fig S99. HPLC Profile of Enantioenriched 4f

```

NAME      inn-3-ksb-154c-1h
EXPNO     4
PROCNO    1
Date_     20120927
Time      19.12
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         7
DS         0
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         128
DW         60.800 usec
DE         6.50 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

```



```

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

```

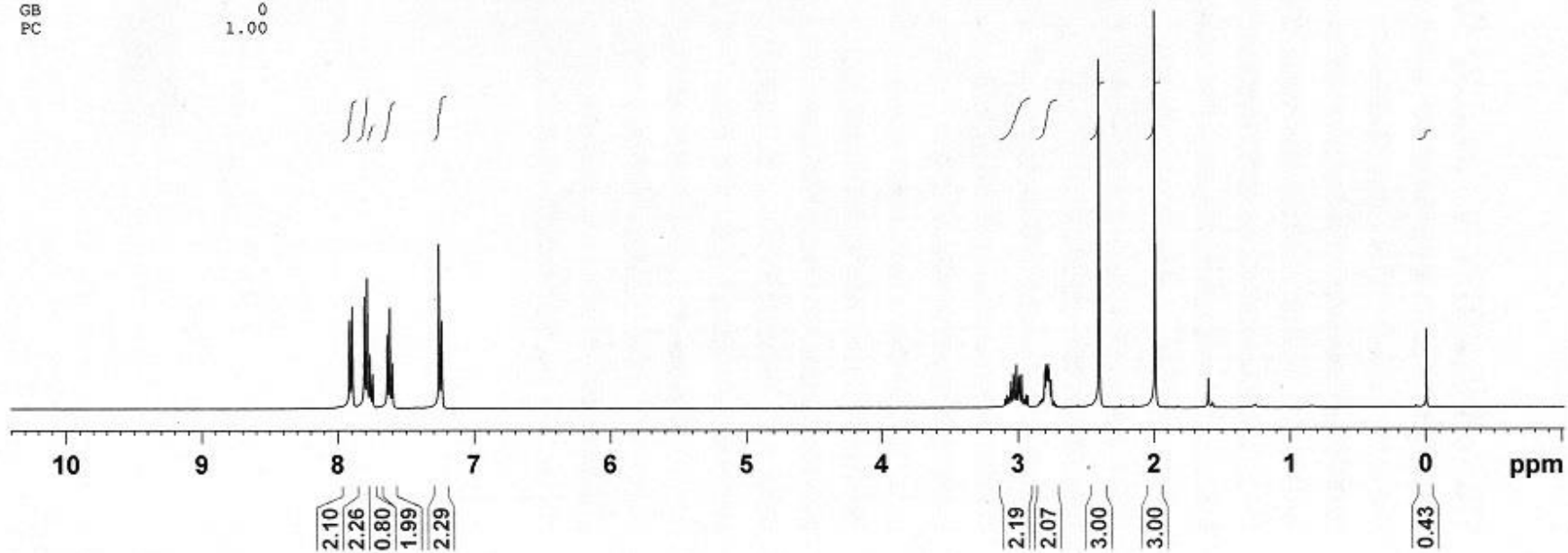


Fig S100. <sup>1</sup>H NMR Spectrum of 6a



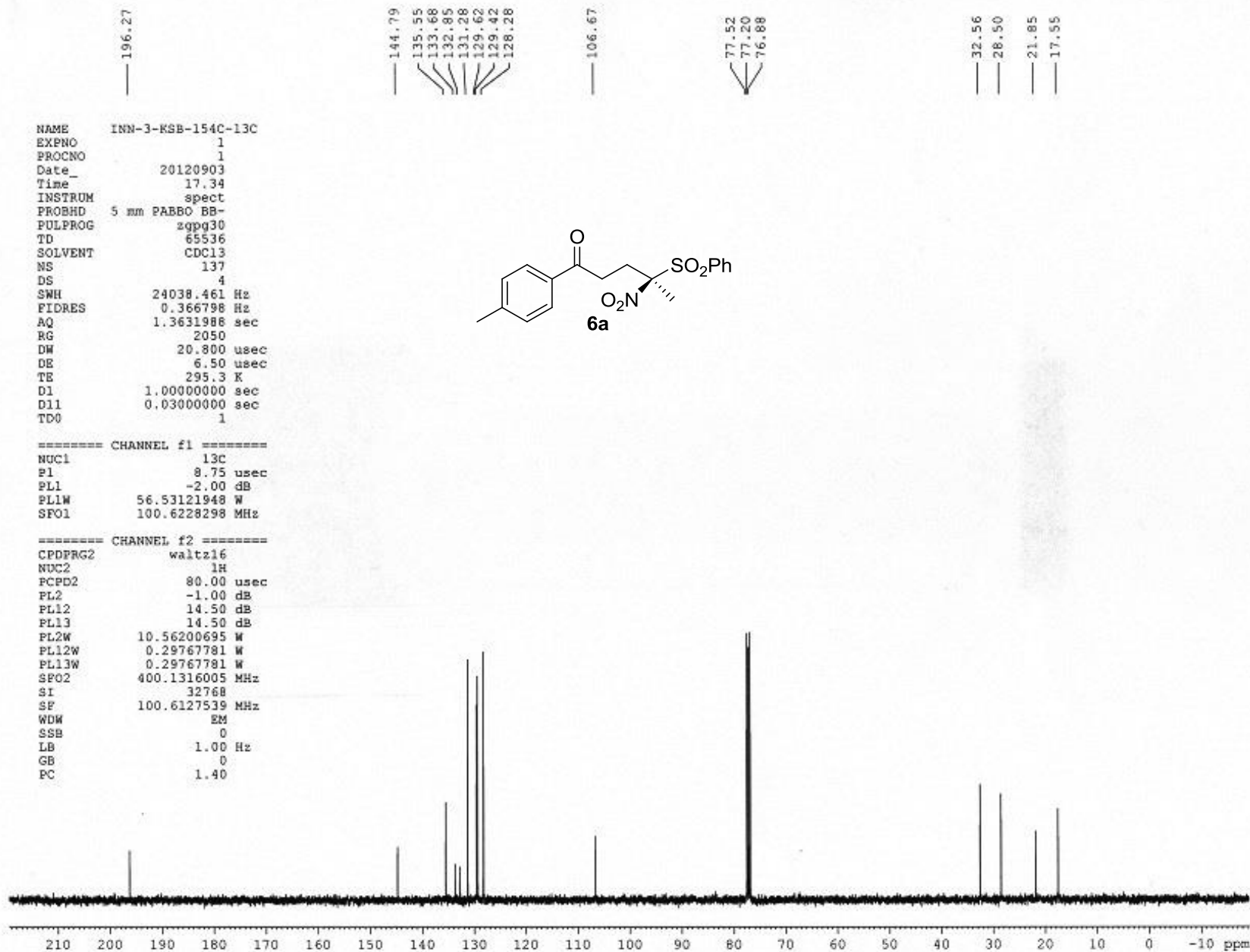
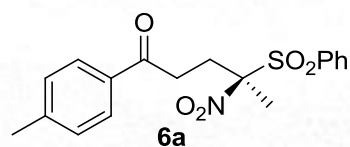


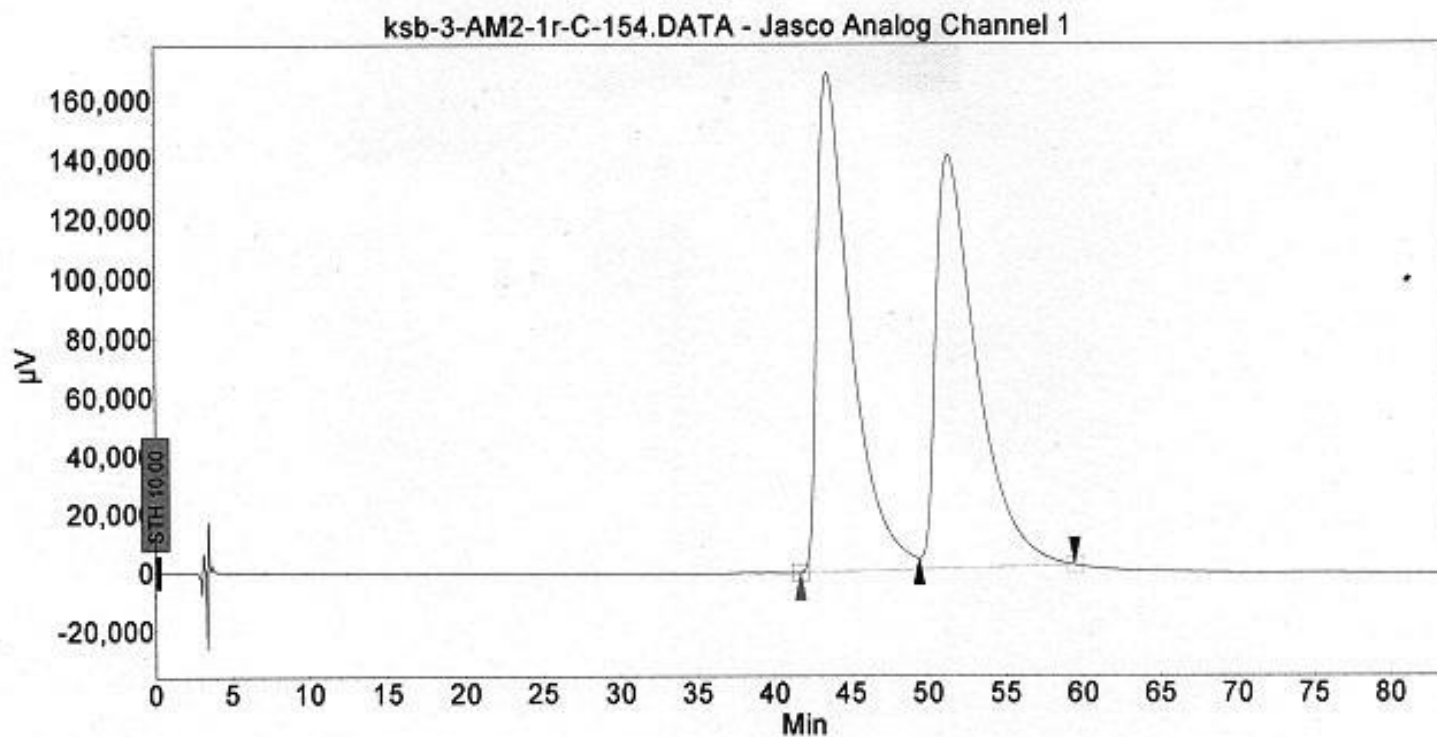
Fig S101. <sup>13</sup>C NMR Spectrum of 6a

# Chromatogram : ksb-3-AM2-1r-C-154\_channel2

System : Jasco HPLC  
 Method : ksb-30%-1f-250nm  
 User : User1



Acquired : 5/7/2014 9:49:36 AM  
 Processed : 5/7/2014 11:13:53 AM  
 Printed : 5/21/2014 1:10:44 PM



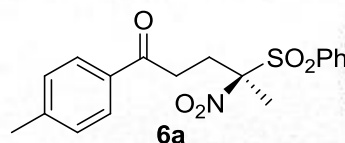
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	43.533	49.97	169000.2	431839.3	49.966
2	UNKNOWN	51.392	50.03	140072.2	432429.8	50.034
Total			100.00	309072.4	864269.1	100.000

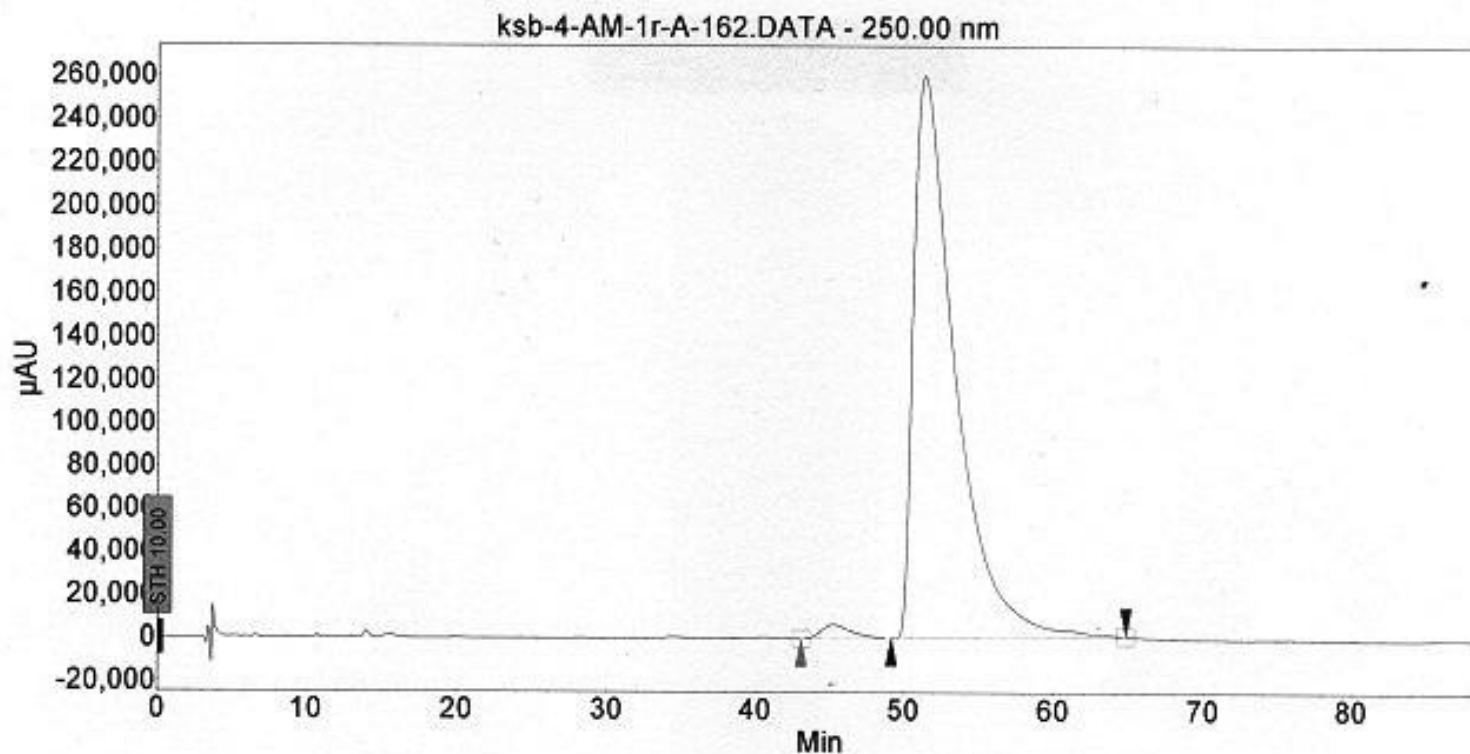
Fig S102. HPLC Profile of Racemic 6a

# Chromatogram : ksb-4-AM-1r-A-162\_channel4

System : Jasco HPLC  
Method : ksb-30%-1f-250nm  
User : User1



Acquired : 5/7/2014 11:15:03 AM  
Processed : 5/7/2014 1:06:21 PM  
Printed : 5/21/2014 1:08:19 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	45.250	1.70	6380.4	14598.2	1.699
2	UNKNOWN	51.328	98.30	259840.5	844374.1	98.301
Total			100.00	266220.9	858972.4	100.000

Fig S103. HPLC Profile of Enantioenriched 6a

Current Data Parameters  
NAME INN-4-KSB-161A-1H  
EXPMO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140507  
Time 15.40  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 59998  
SOLVENT CDCl3  
NS 17  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.166672 Hz  
AQ 2.9999001 sec  
RG 48.36  
DN 50.000 usec  
DE 6.50 usec  
TE 299.3 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 500.1335009 MHz  
NUC1 1H  
P1 11.00 usec  
PLW1 13.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300044 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00

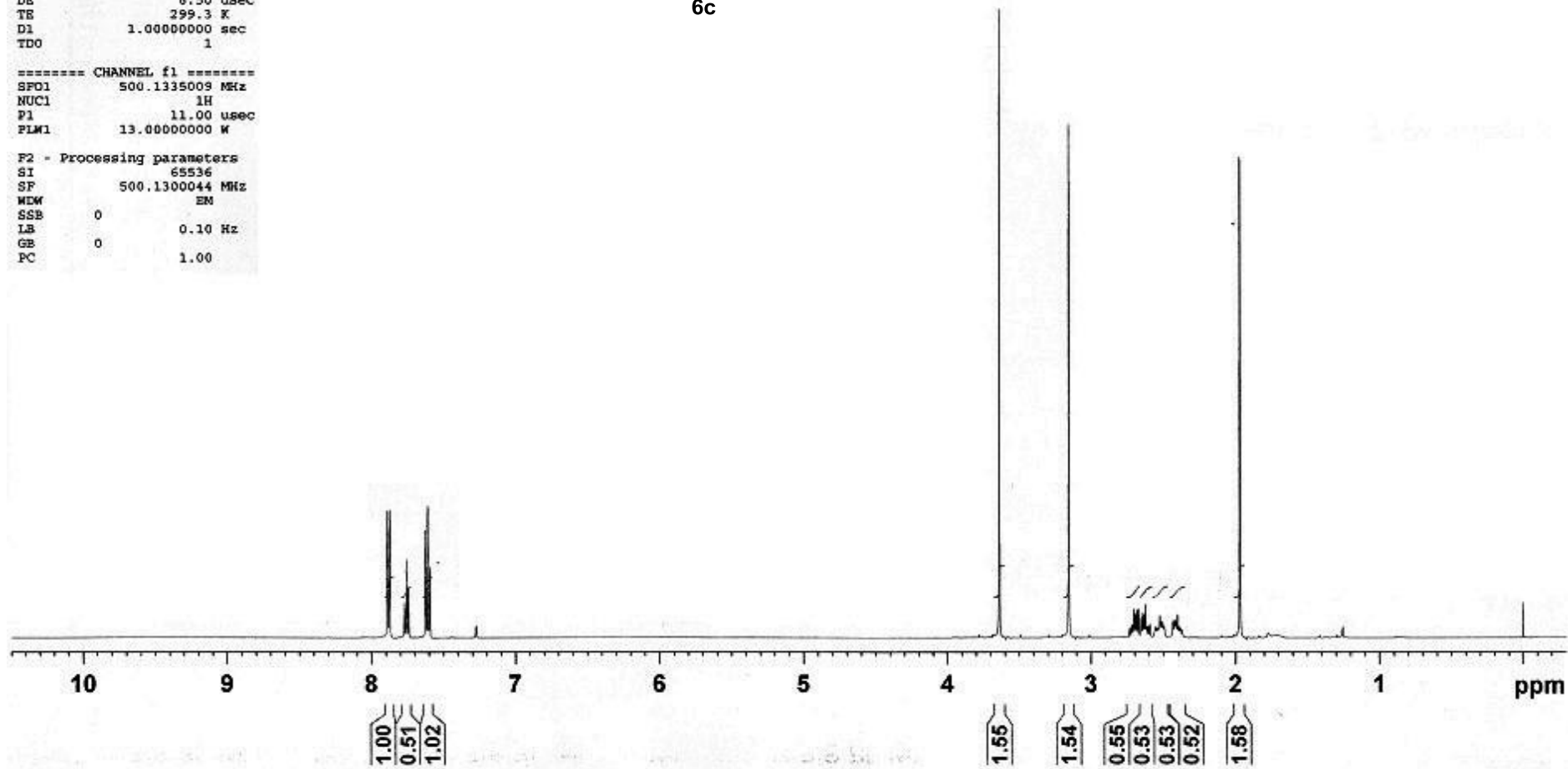
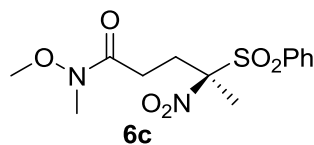


Fig S104. <sup>1</sup>H NMR Spectrum of 6c

```

Current Data Parameters
NAME      INN-4-KSB-161A-1p45
EXPNO    1
PROCNO   1
F2 - Acquisition Parameters
Date_    20140507
Time     16.42
INSTRUM  spect
PROBHD   5 mm BBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       100
DS       4
SNH      29761.904 Hz
FIDRES   0.454131 Hz
AQ       1.1010048 sec
RG       197.27
DW       16.800 usec
DE       6.50 usec
TE       299.9 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
CPDPRG2  waltz16
PCPD2    80.00 usec
PLW2    13.00000000 W
PLW12   0.34327999 W
PLW13   0.21969999 W

```

```

F2 - Processing parameters
SI       32768
SF       125.7577718 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40

```

135.48  
132.83  
131.24  
129.37

106.65

77.45  
77.20  
76.85

61.49

32.43  
28.58  
26.38

17.10

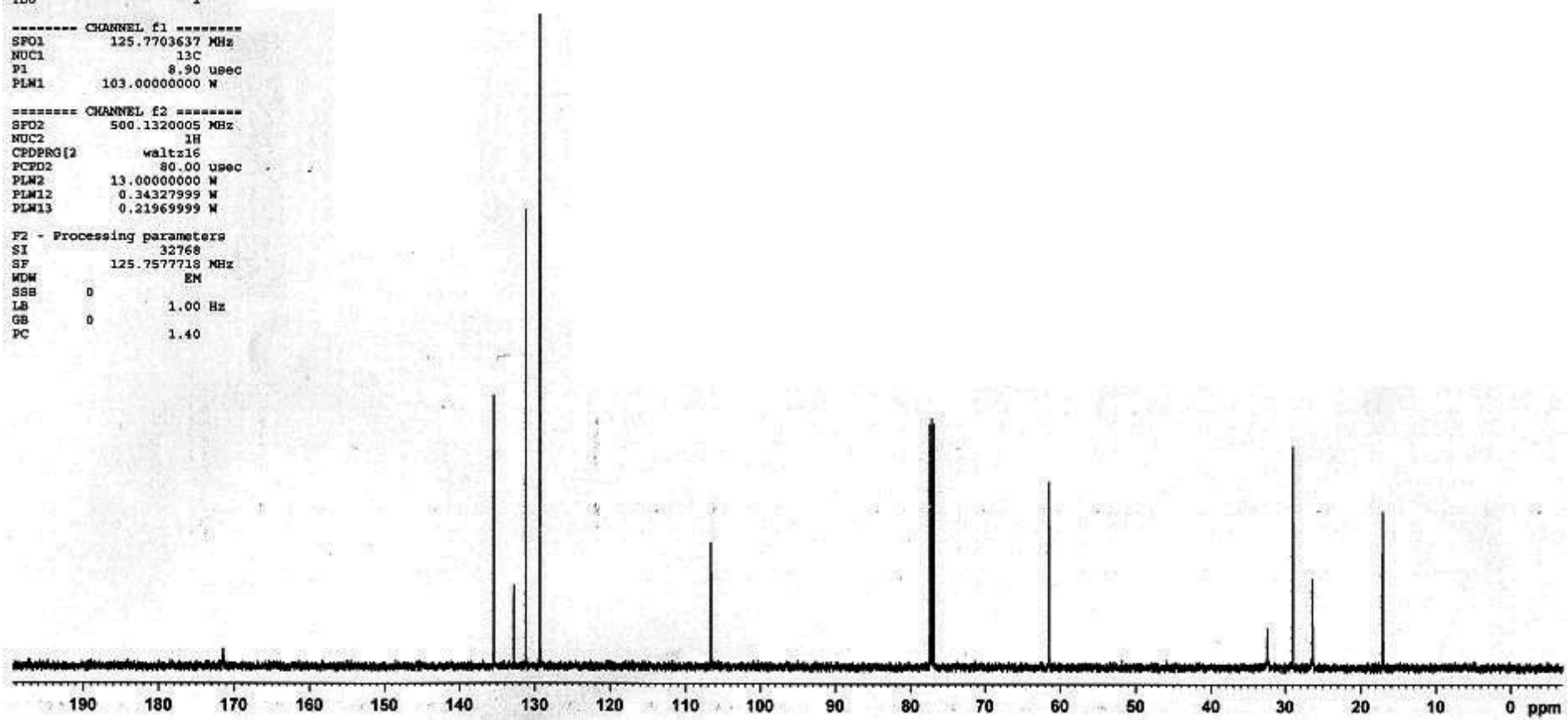
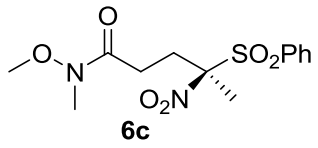
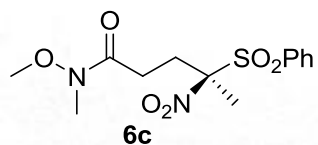


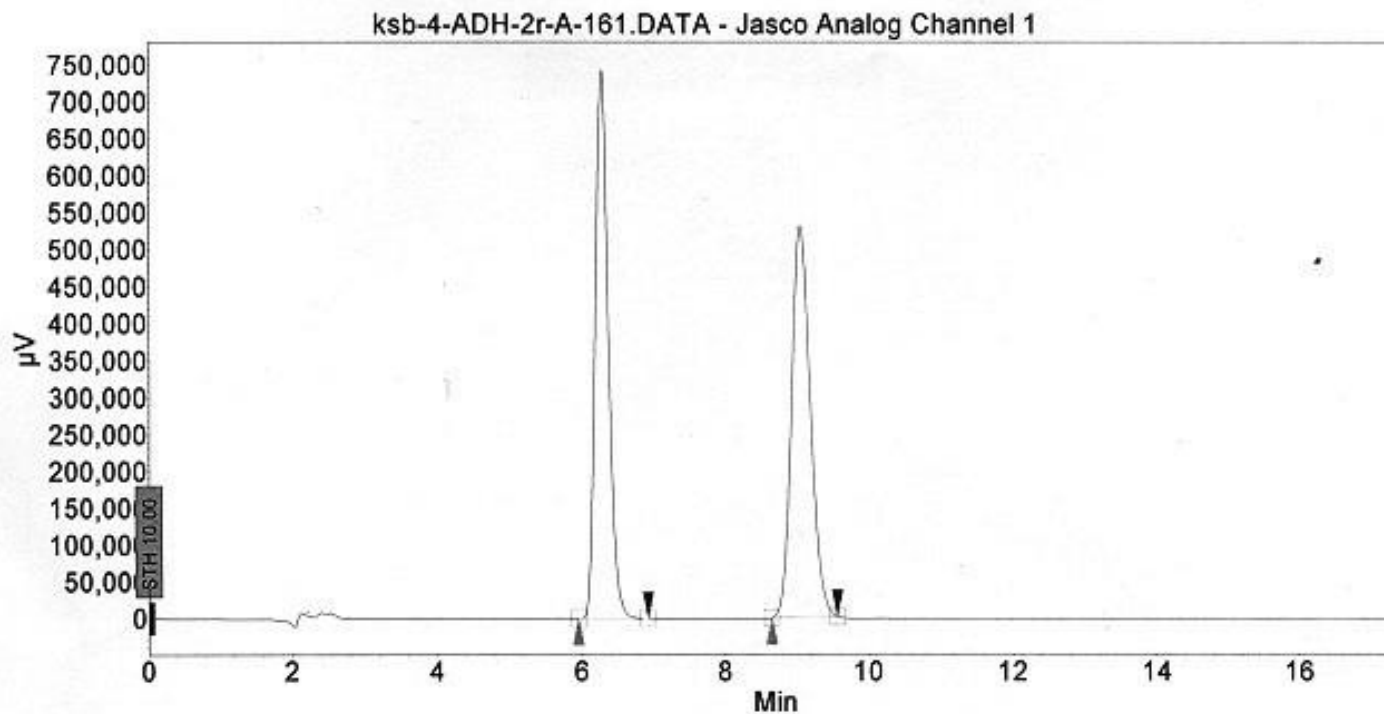
Fig S105. <sup>13</sup>C NMR Spectrum of 6c

# Chromatogram : ksb-4-ADH-2r-A-161\_channel2

System : Jasco HPLC  
Method : 30%-1f-212nm  
User : User1



Acquired : 5/6/2014 10:02:11 PM  
Processed : 5/6/2014 10:23:26 PM  
Printed : 5/6/2014 10:23:35 PM



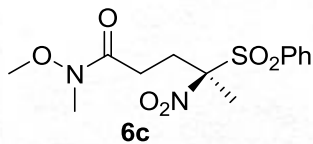
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	6.267	49.16	743491.3	156360.9	49.160
2	UNKNOWN	9.050	50.84	529815.8	161704.6	50.840
Total			100.00	1273307.1	318065.6	100.000

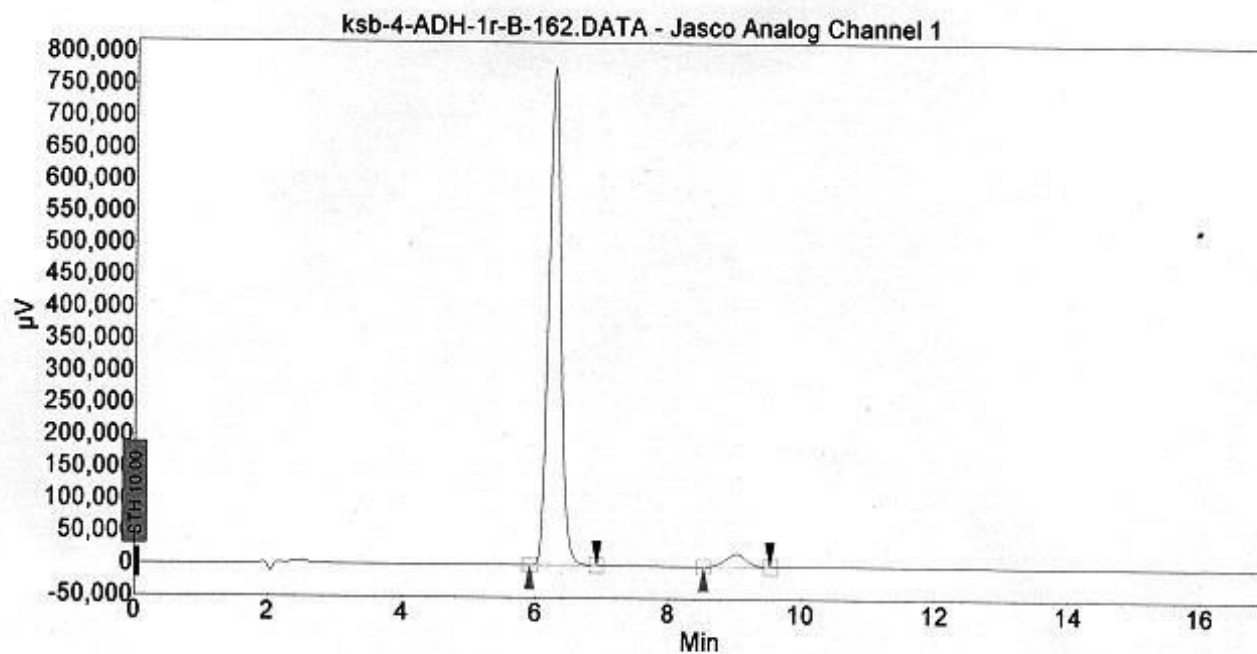
Fig S106. HPLC Profile of Racemic 6c

## Chromatogram : ksb-4-ADH-1r-B-162\_channel2

System : Jasco HPLC  
Method : 30%-1f-212nm  
User : User1



Acquired : 5/6/2014 10:21:59 PM  
Processed : 5/6/2014 10:39:12 PM  
Printed : 5/6/2014 10:42:11 PM



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area % [%]
1	UNKNOWN	6.242	95.86	777788.2	165828.1	95.858
2	UNKNOWN	9.033	4.14	20017.7	7165.0	4.142
Total			100.00	797805.9	172993.1	100.000

Fig S107. HPLC Profile of Enantioenriched 6c

Current Data Parameters  
NAME INN-4-KSB-161b-1H  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140507  
Time 15.47  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 59998  
SOLVENT CDCl3  
NS 15  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.166672 Hz  
AQ 2.9999001 sec  
RG 80.35  
DW 50.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 1.0000000 sec  
TDO 1

----- CHANNEL f1 -----  
SF01 500.1335009 MHz  
NUC1 1H  
P1 11.00 usec  
PLW1 13.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300093 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00

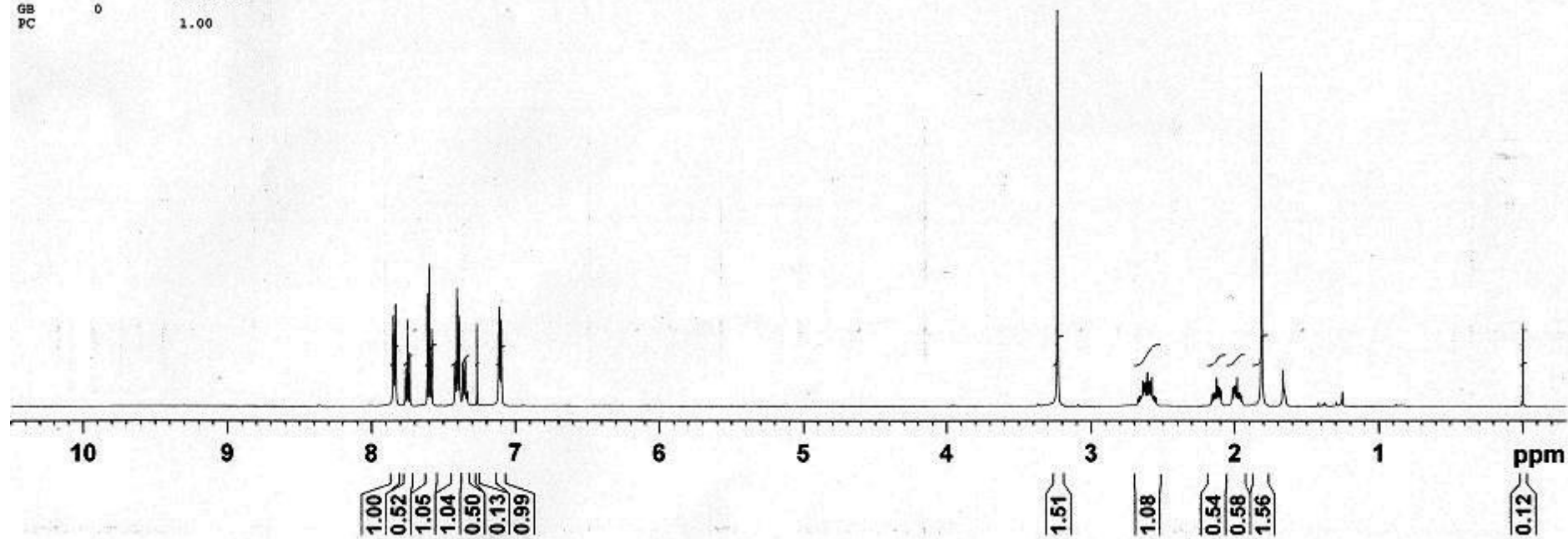
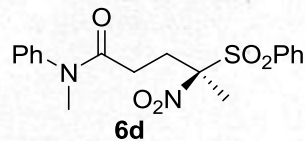


Fig S108. <sup>1</sup>H NMR Spectrum of 6d



Current Data Parameters  
NAME: INM-4-KSB-161B-13C  
EXPMO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date: 20140507  
Time: 16.49  
INSTRUM: spect  
PROBHD: 5 mm PABBO BB/  
PULPROG: zgpg30  
TD: 65536  
SOLVENT: CDCl3  
NS: 64  
DS: 4  
SWH: 29761.904 Hz  
FIDRES: 0.454131 Hz  
AQ: 1.1010048 sec  
RG: 197.27  
SW: 16.800 usec  
DE: 6.50 usec  
TE: 300.4 K  
D1: 1.0000000 sec  
D11: 0.0300000 sec  
TDO: 1

----- CHANNEL f1 -----  
SP01: 125.7703637 MHz  
NUC1: 13C  
P1: 8.90 usec  
PLW1: 103.0000000 W

----- CHANNEL f2 -----  
SP02: 500.1320005 MHz  
NUC2: 1H  
CPDPRG2: waltz16  
PCPD2: 80.00 usec  
PLW2: 13.0000000 W  
PLW12: 0.34327999 W  
PLW13: 0.21969999 W

F2 - Processing parameters  
SI: 32768  
SF: 125.7577682 MHz  
WDW: RM  
SSB: 0  
LB: 1.00 Hz  
GB: 0  
PC: 1.40

169.99

143.31  
135.45  
132.84  
131.27  
130.28  
129.35  
128.52  
127.19

106.57

77.45  
77.20  
76.99

37.69

29.93  
28.43

16.80

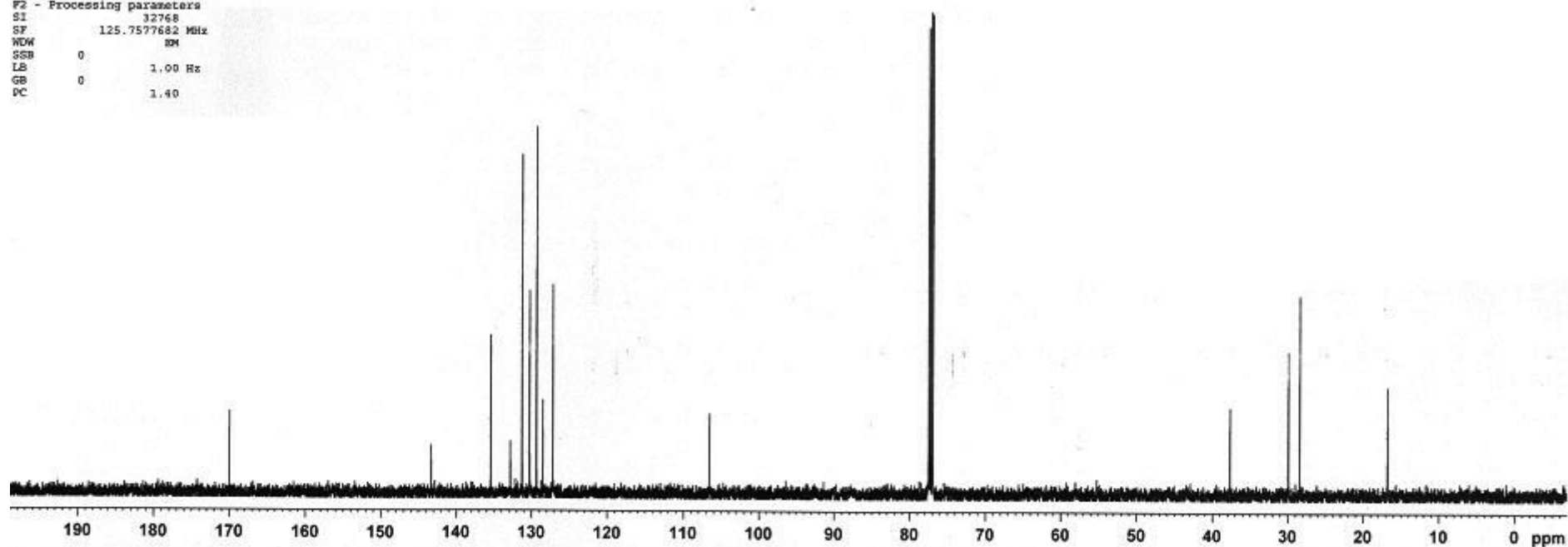
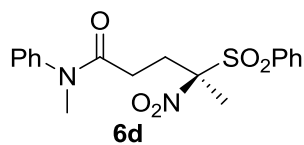
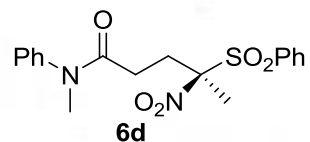


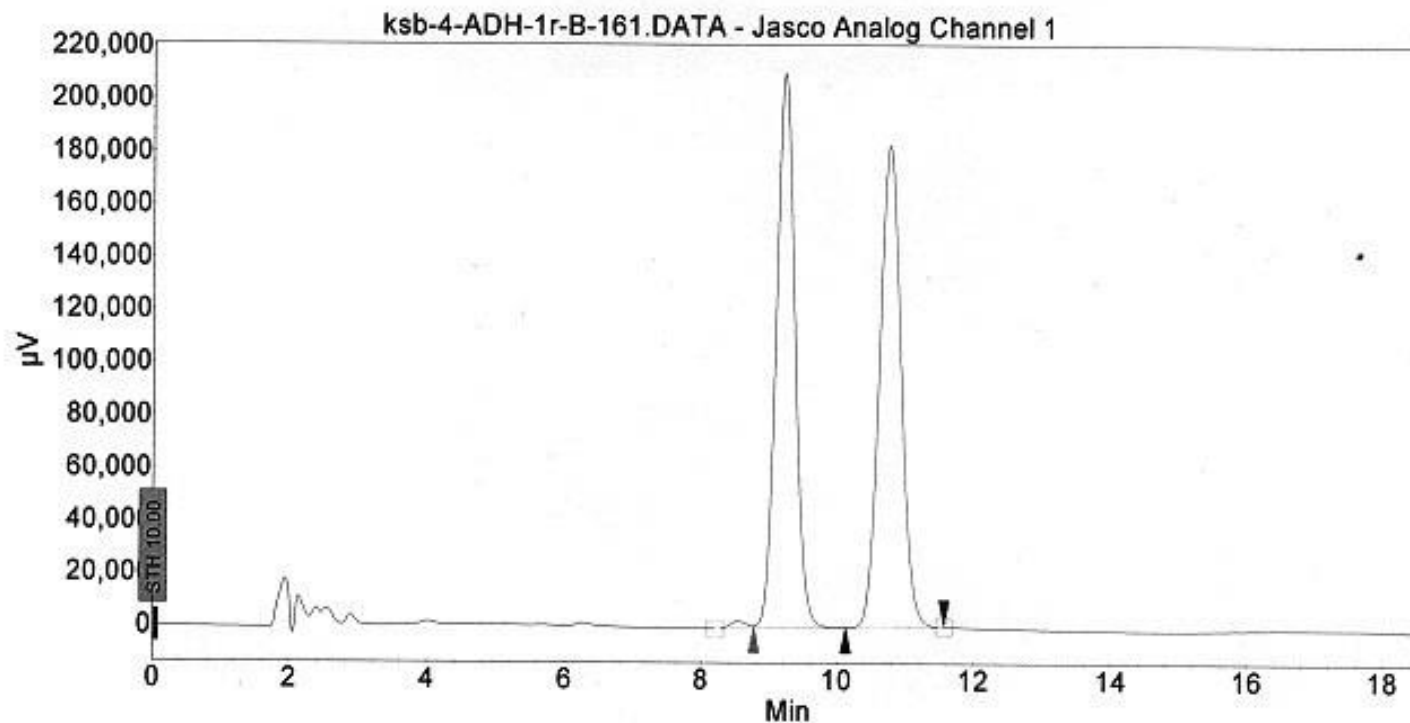
Fig S109. <sup>13</sup>C NMR Spectrum of 6d

# Chromatogram : ksb-4-ADH-1r-B-161\_channel2

System : Jasco HPLC  
Method : 30%-1f-212nm  
User : User1



Acquired : 5/6/2014 10:40:43 PM  
Processed : 5/6/2014 10:59:41 PM  
Printed : 5/6/2014 11:03:15 PM



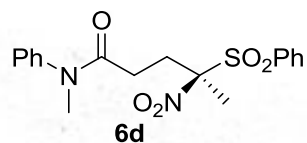
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	9.217	50.09	210822.0	67225.9	50.094
2	UNKNOWN	10.758	49.91	183064.5	66973.6	49.906
Total			100.00	393886.4	134199.5	100.000

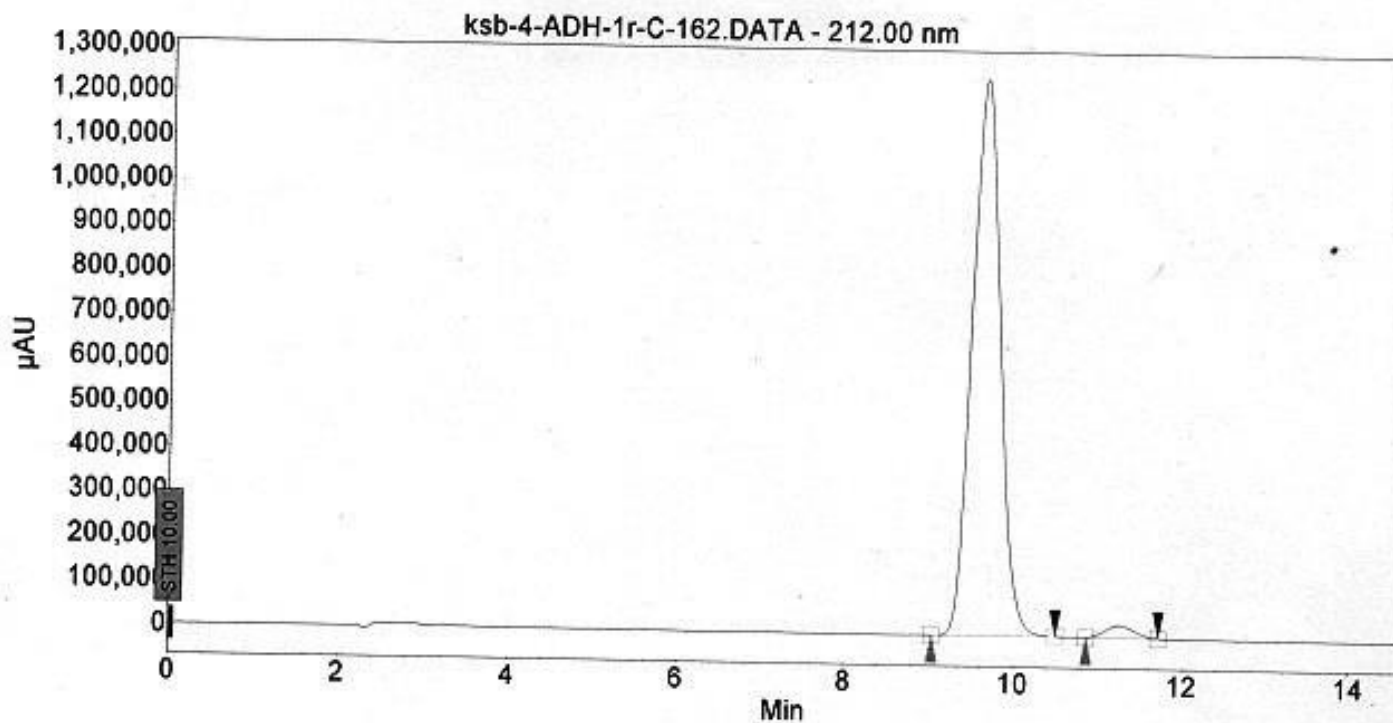
Fig S110. HPLC Profile of Racemic 6d

# Chromatogram : ksb-4-ADH-1r-C-162\_channel4

System : Jasco HPLC  
Method : 30%-1f-212nm  
User : User1



Acquired : 5/6/2014 11:01:22 PM  
Processed : 5/6/2014 11:35:57 PM  
Printed : 5/6/2014 11:36:41 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	9.613	97.76	1248179.3	478862.4	97.755
2	UNKNOWN	11.253	2.24	27847.1	10996.1	2.245
Total			100.00	1276026.4	489858.5	100.000

Fig S111. HPLC Profile of Enantioenriched 6d

NAME INN-3-KSB-14A-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20111112  
 Time\_ 15.18  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT DMSO  
 NS 14  
 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 400.1300026 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

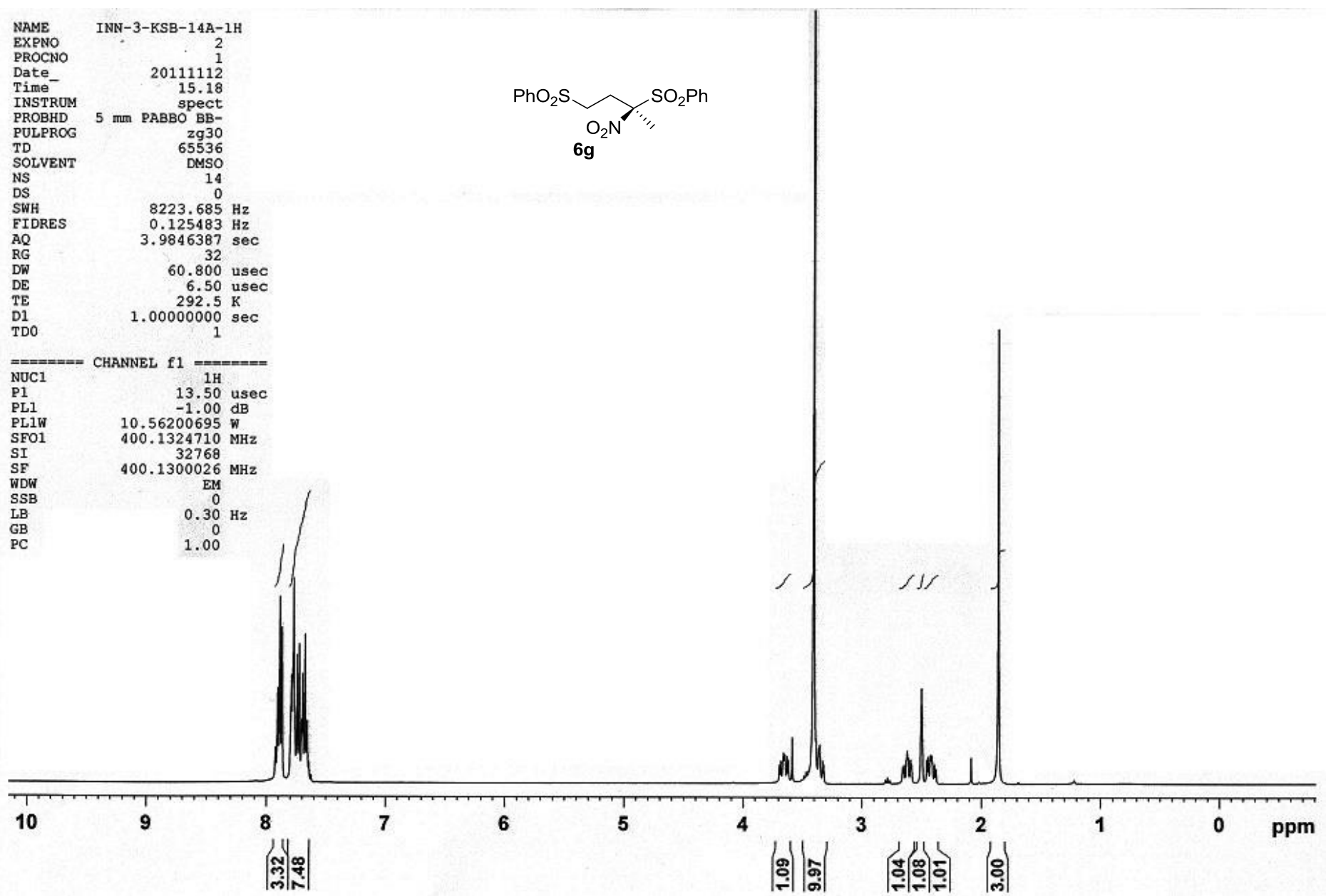
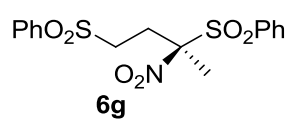


Fig S112. <sup>1</sup>H NMR Spectrum of 6g

NAME INN-3-KSB-14A-13C  
 EXPNO 2  
 PROCNO 1  
 Date 20111112  
 Time 15.24  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT DMSO  
 NS 116  
 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 293.3 K  
 D1 2.00000000 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 100.6128108 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

137.89  
 136.26  
 134.35  
 131.12  
 130.70  
 129.89  
 129.66  
 127.92

105.16

49.07  
 40.13  
 39.92  
 39.71  
 39.50  
 39.29  
 39.08  
 38.87  
 26.37

16.60

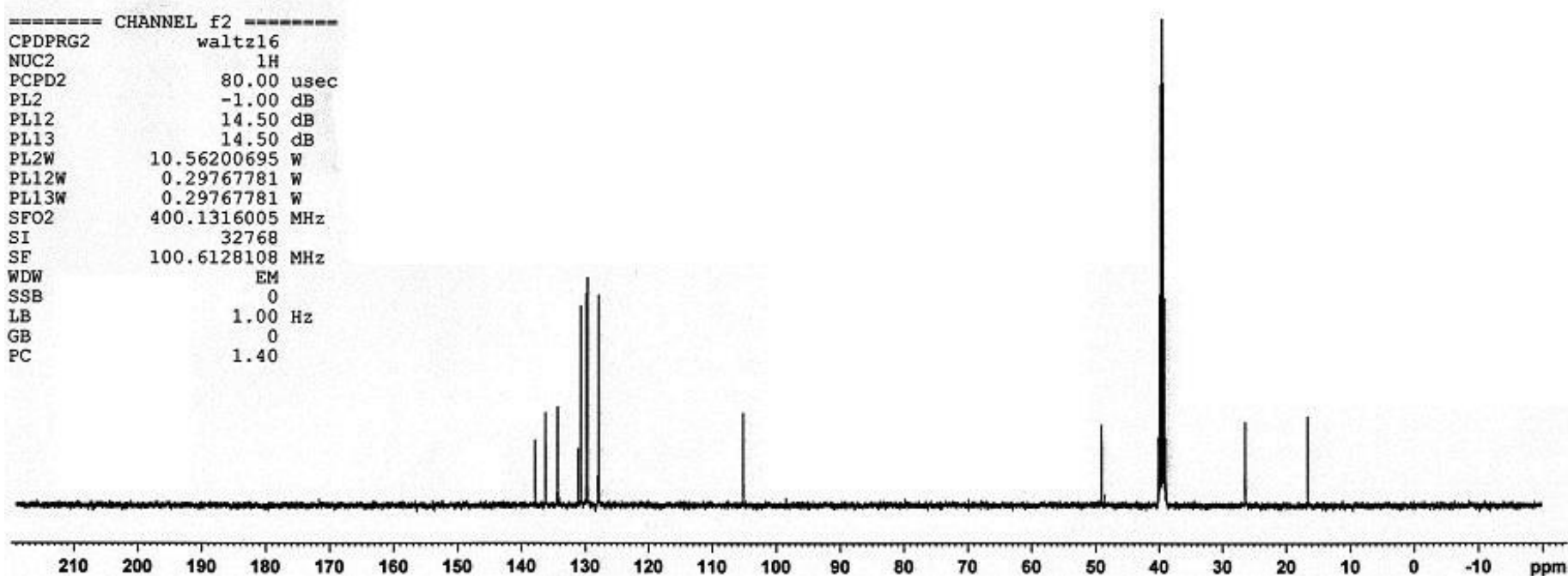
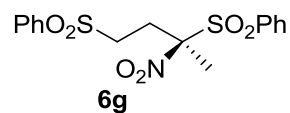
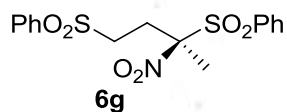


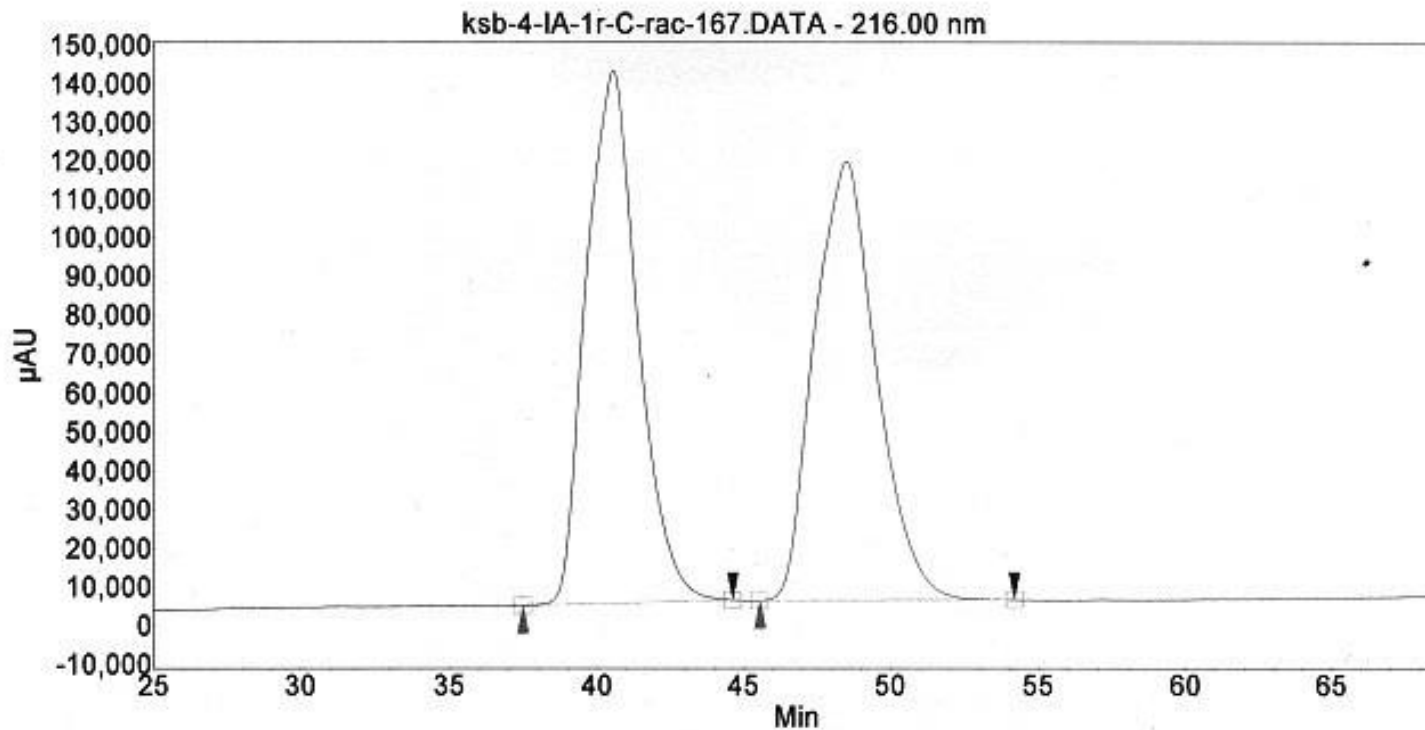
Fig S113. <sup>13</sup>C NMR Spectrum of 6g

# Chromatogram : ksb-4-IA-1r-C-rac-167\_channel4

System : JASCO HPLC  
 Method : ksb-30-1fr-216  
 User : User1



Acquired : 5/20/2014 11:20:45 AM  
 Processed : 5/20/2014 12:33:41 PM  
 Printed : 5/20/2014 1:21:41 PM



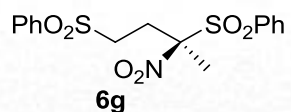
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	40.545	50.18	137678.3	280377.3	50.176
2	UNKNOWN	48.516	49.82	113419.0	278414.2	49.824
Total			100.00	251097.3	558791.5	100.000

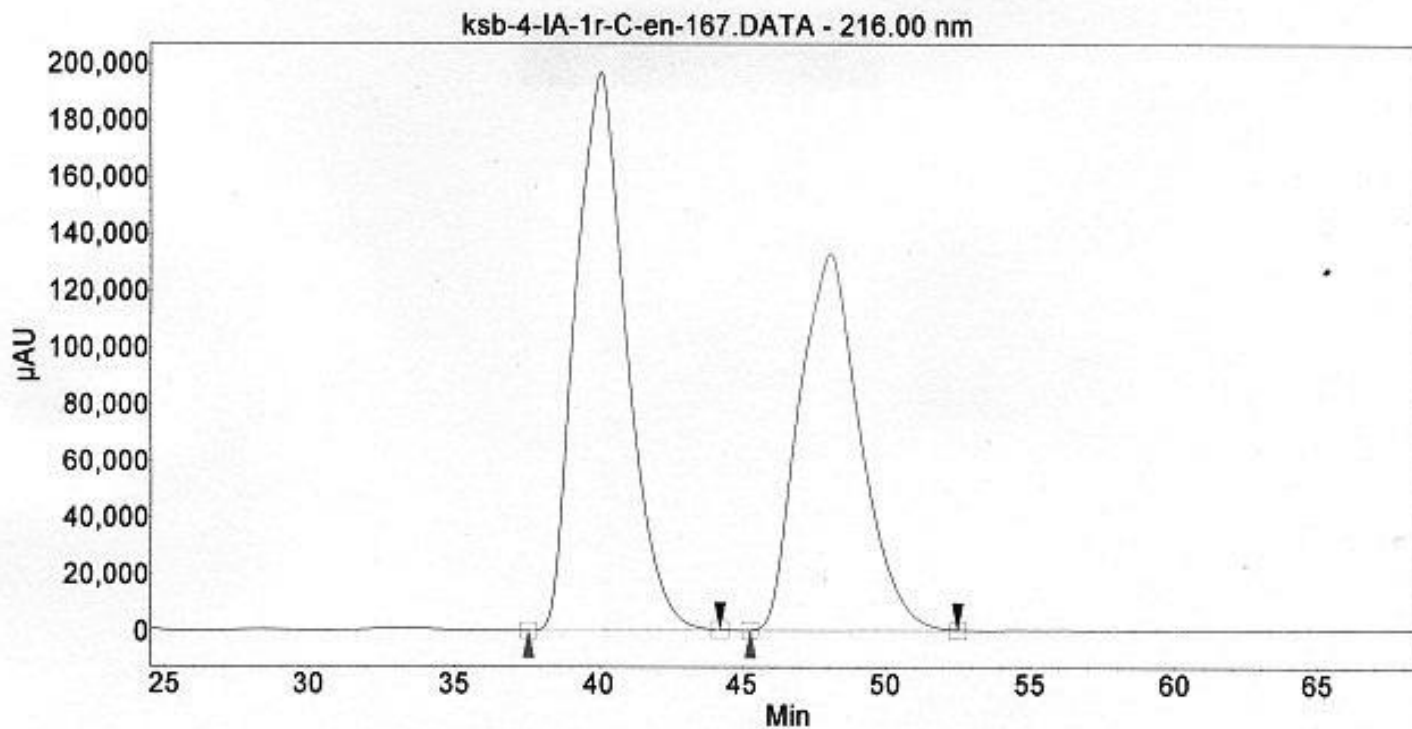
Fig S114. HPLC Profile of Racemic 6g

# Chromatogram : ksb-4-IA-1r-C-en-167\_channel4

System : JASCO HPLC  
Method : ksb-30-1fr-216  
User : User1



Acquired : 5/20/2014 12:32:19 PM  
Processed : 5/20/2014 1:43:36 PM  
Printed : 5/20/2014 1:43:43 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{AU}$ ]	Area [ $\mu\text{AU}\cdot\text{Min}$ ]	Area % [%]
1	UNKNOWN	40.052	55.05	196435.5	401230.7	55.051
2	UNKNOWN	48.063	44.95	132467.6	327606.0	44.949
Total			100.00	328903.1	728836.7	100.000

Fig S115. HPLC Profile of Enantioenriched 6g

Current Data Parameters  
NAME INN-4-KSB-168A-1H  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140518  
Time 12.11  
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 294.9 K  
D1 1.00000000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 11.00 usec  
PLW1 13.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300117 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

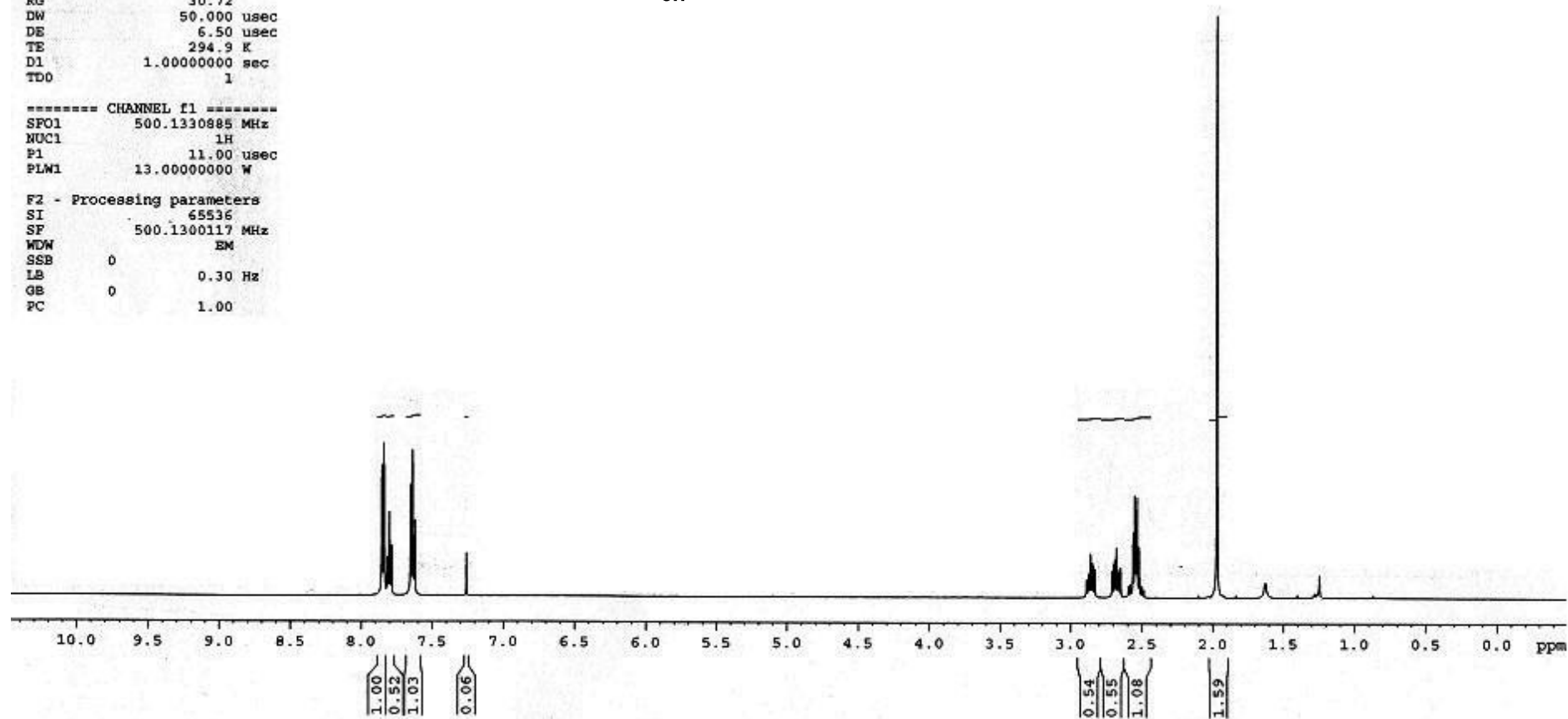
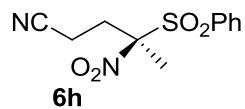


Fig S116. <sup>1</sup>H NMR Spectrum of 6h



Current Data Parameters  
NAME INN-4-KSB-168A-13C  
EXPNO 9  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140518  
Time\_ 12.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 235  
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 295.5 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 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 125.7577728 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

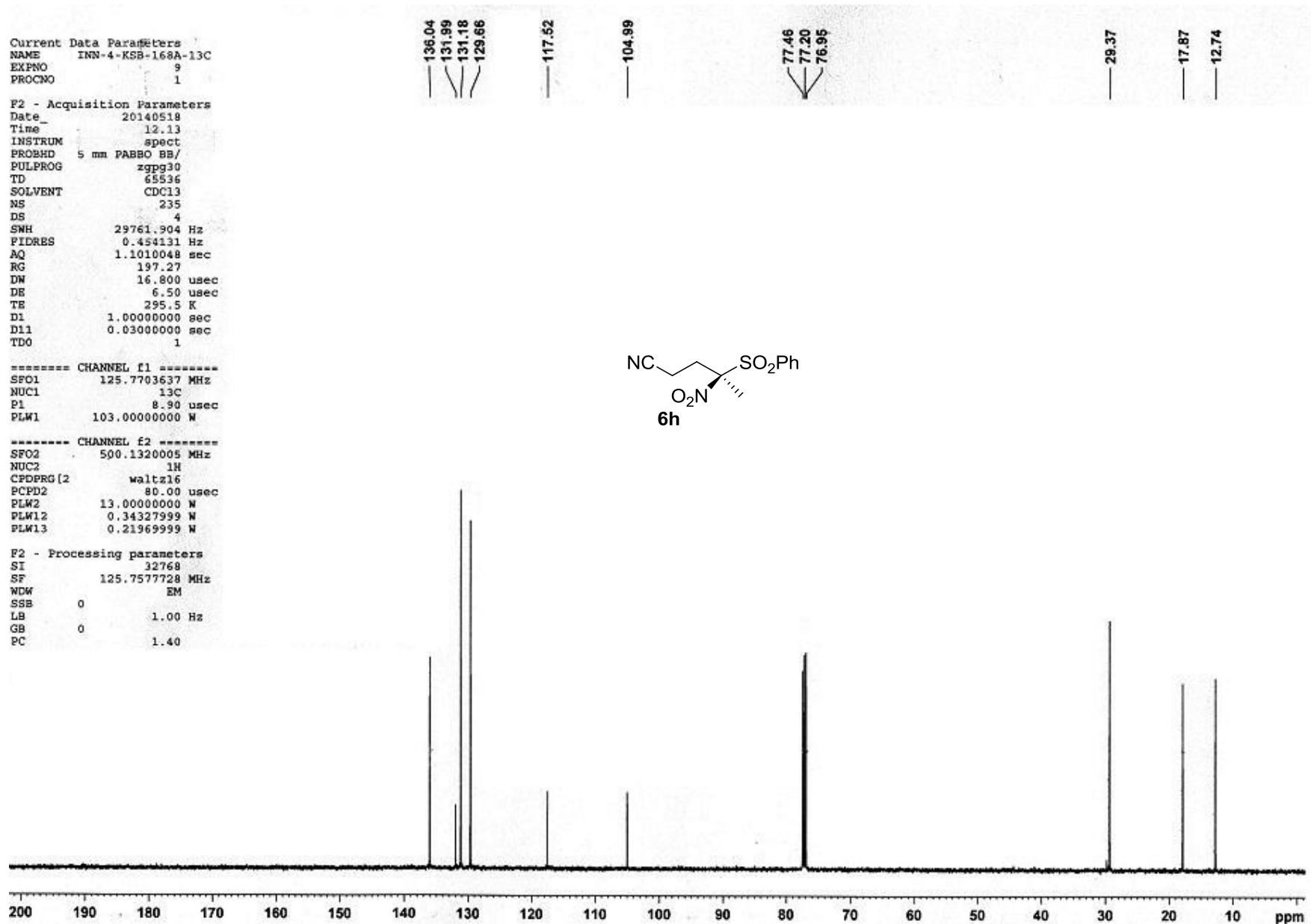
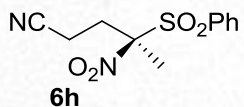


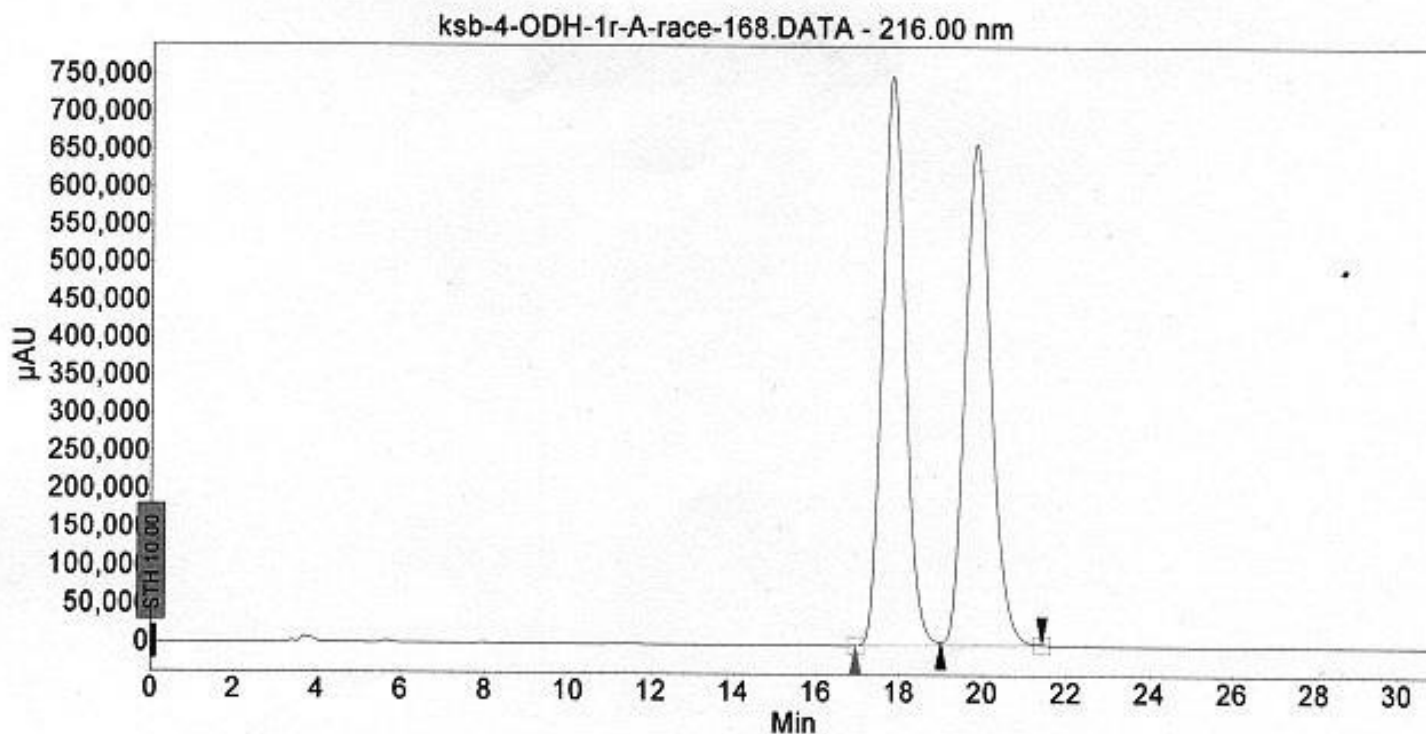
Fig S117. <sup>13</sup>C NMR Spectrum of 6h

# Chromatogram : ksb-4-ODH-1r-A-race-168\_channel4

System : JASCO HPLC  
Method : ksb-30-1r-216  
User : User1



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Printed : 5/19/2014 11:19:46 AM



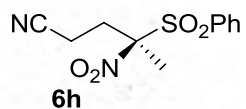
## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	17.785	50.08	752657.8	473959.2	50.077
2	UNKNOWN	19.799	49.92	660839.6	472495.7	49.923
Total			100.00	1413497.5	946454.9	100.000

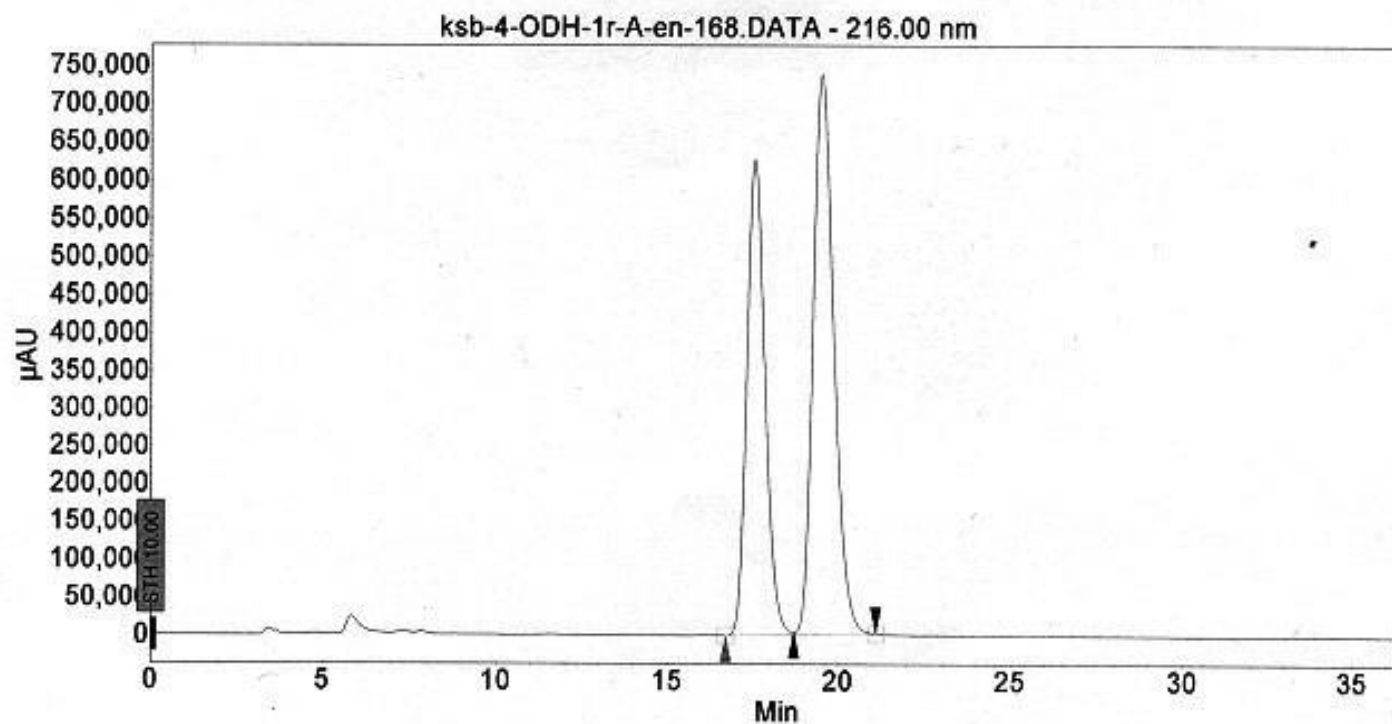
Fig S118. HPLC Profile of Racemic 6h

# Chromatogram : ksb-4-ODH-1r-A-en-168\_channel4

System : JASCO HPLC  
Method : ksb-30-1fr-216  
User : User1



Acquired : 5/19/2014 11:52:03 AM  
Processed : 5/19/2014 12:28:52 PM  
Printed : 5/19/2014 12:29:44 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	17.545	42.86	627529.8	388201.9	42.858
2	UNKNOWN	19.505	57.14	738239.0	517579.7	57.142
Total			100.00	1365768.8	905781.6	100.000

Fig S119. HPLC Profile of Enantioenriched 6h

NAME INN-4-KSB-112A-1H  
 EXPNO 13  
 PROCNO 1  
 Date\_ 20130905  
 Time\_ 23.34  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWH 6223.685 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 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300095 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

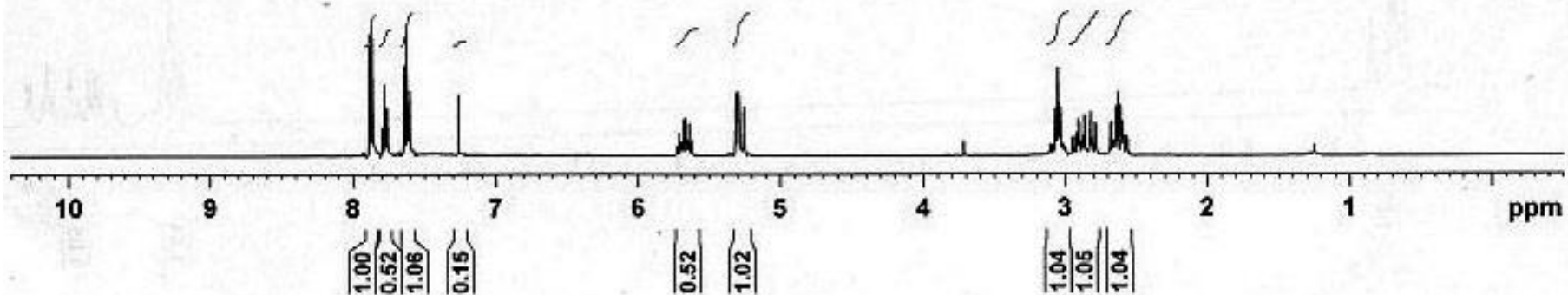
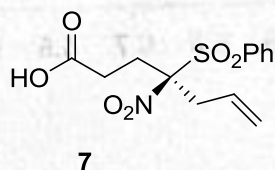


Fig S120. <sup>1</sup>H NMR Spectrum of 7

NAME INN-4-KSB-112A-13C  
 EXPNO 14  
 PROCNO 1  
 Date 20130905  
 Time 23.35  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 178  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631988 sec  
 RG 2050  
 DM 20.800 usec  
 DE 6.50 usec  
 TE 294.7 K  
 D1 1.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

----- CHANNEL f1 -----  
 NUC1 13C  
 P1 13.00 usec  
 PL1 -3.00 dB  
 PL1W 71.16858673 W  
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -3.00 dB  
 PL12 18.48 dB  
 PL13 19.00 dB  
 PL2W 16.73965454 W  
 PL12W 0.11905469 W  
 PL13W 0.10562007 W  
 SFO2 400.1316005 MHz  
 SI 32768  
 SF 100.6127690 MHz  
 MDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

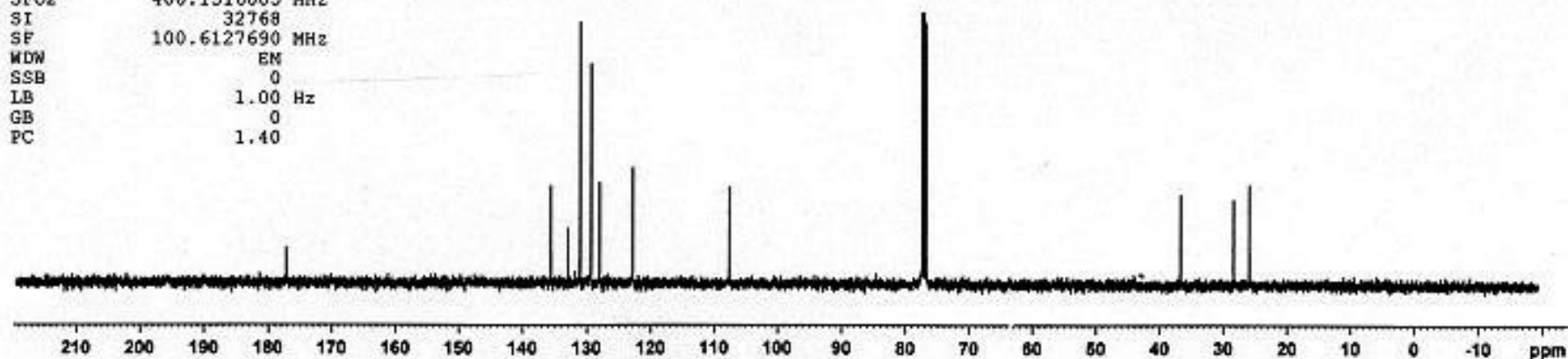
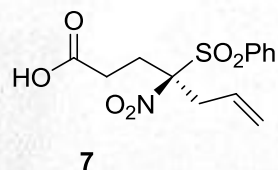


Fig S121. <sup>13</sup>C NMR Spectrum of 7

Current Data Parameters  
NAME inn-4-ksb-125a-1h  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131020  
Time 22.58  
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 294.5 K  
D1 1.0000000 sec  
TD0 1

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

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

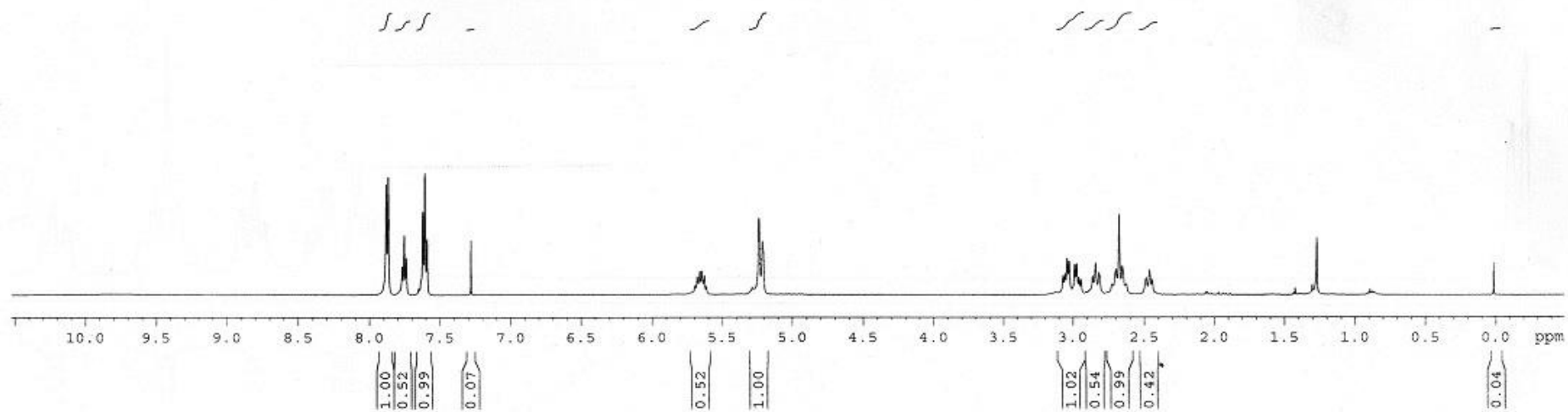
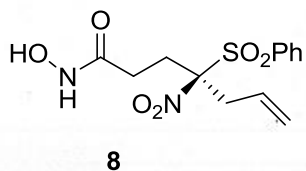


Fig S122. <sup>1</sup>H NMR Spectrum of 8

inn-4-ksb-125a-13c

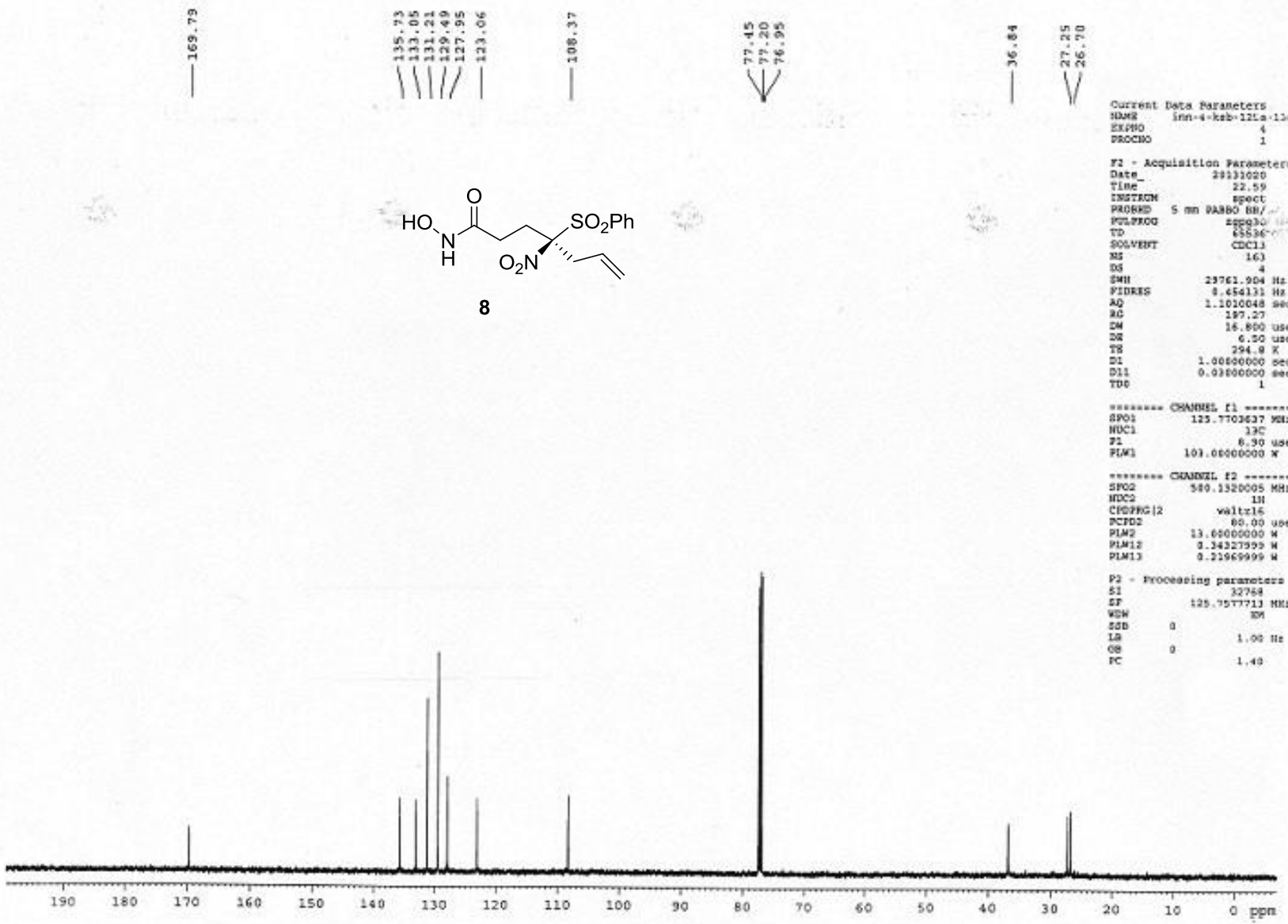
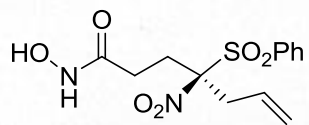


Fig S123. <sup>13</sup>C NMR Spectrum of 8

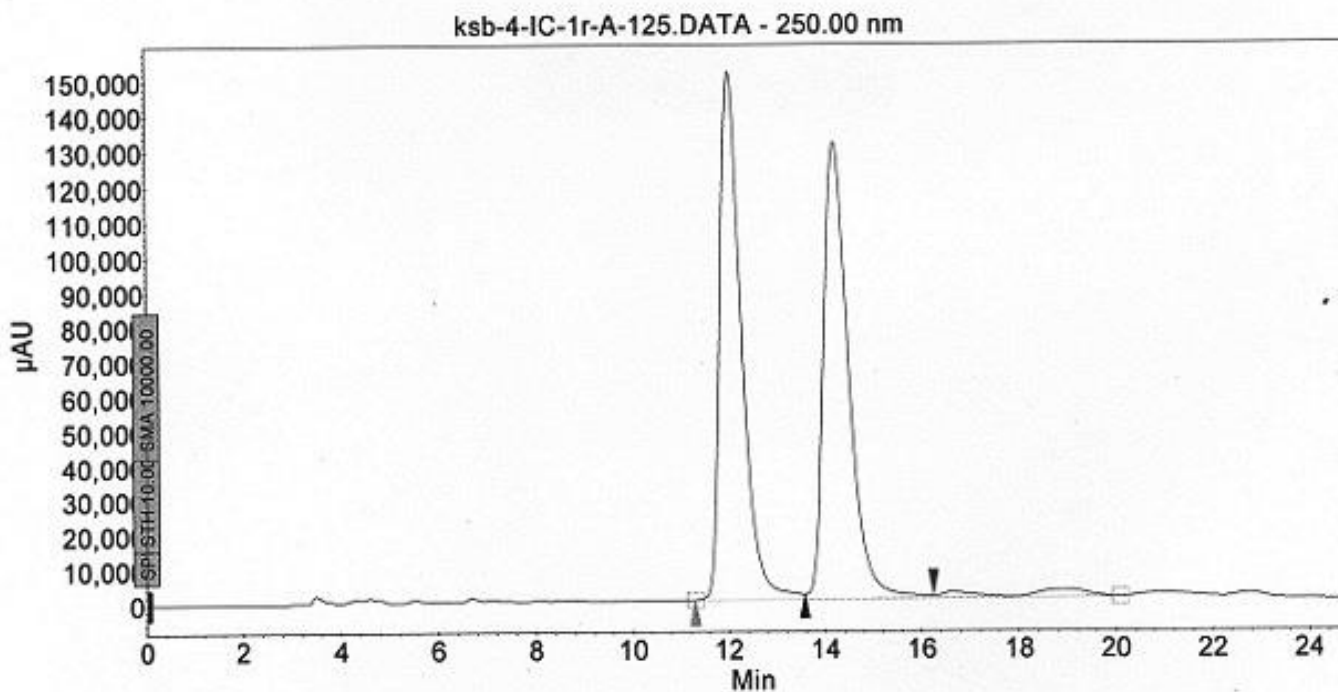
# Chromatogram : ksb-4-IC-1r-A-125\_channel6

System : JASCO HPLC  
 Method : ksb-30-1fr-216  
 User : User1



8

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 Printed : 11/11/2013 8:20:47 PM



## Peak results :

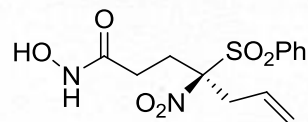
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	12.022	49.86	151181.9	77938.9	49.856
2	UNKNOWN	14.208	50.14	131029.5	78388.6	50.144
Total			100.00	282211.4	156327.5	100.000

Fig S124. HPLC Profile of Racemic 8



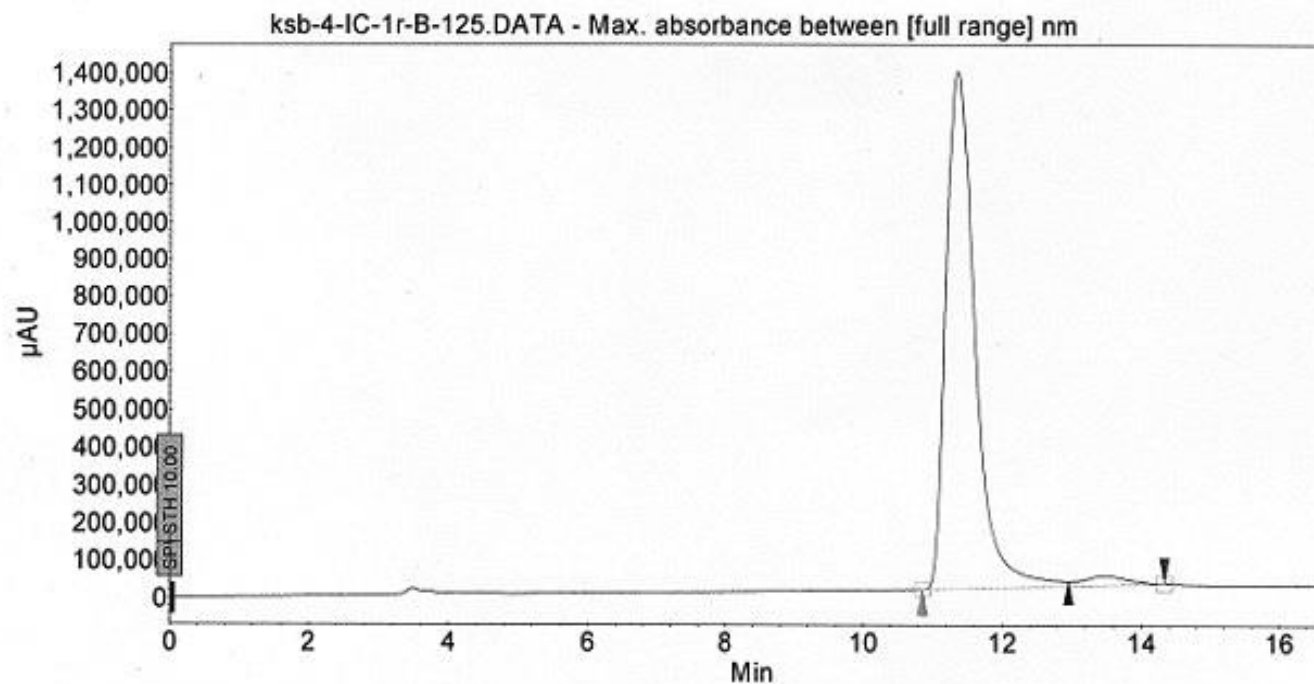
# Chromatogram : ksb-4-IC-1r-B-125\_channel5

System : JASCO HPLC  
 Method : ksb-30-1r-216  
 User : User1



8

Acquired : 11/11/2013 7:13:31 PM  
 Processed : 11/11/2013 7:30:51 PM  
 Printed : 11/11/2013 8:21:27 PM



## Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	11.346	97.21	1380239.6	670184.9	97.211
2	UNKNOWN	13.466	2.79	25816.6	19224.5	2.789
Total			100.00	1406056.3	689409.4	100.000

Fig S125. HPLC Profile of Enantioenriched 8