

**NMR Spectra and HPLC Profiles  
for**

**Enantioselective synthesis of quaternary  $\alpha$ -amino acids via L-*tert*-Leucine-derived squaramide catalyzed conjugate addition of  $\alpha$ -nitrocarboxylates to enones**

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Current Data Parameters  
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EXPNO 2  
PROCNO 1

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PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
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DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 297.8 K  
D1 1.0000000 sec  
TDO 1

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NUC1 1H  
P1 13.00 usec  
PLW1 13.00000000 W

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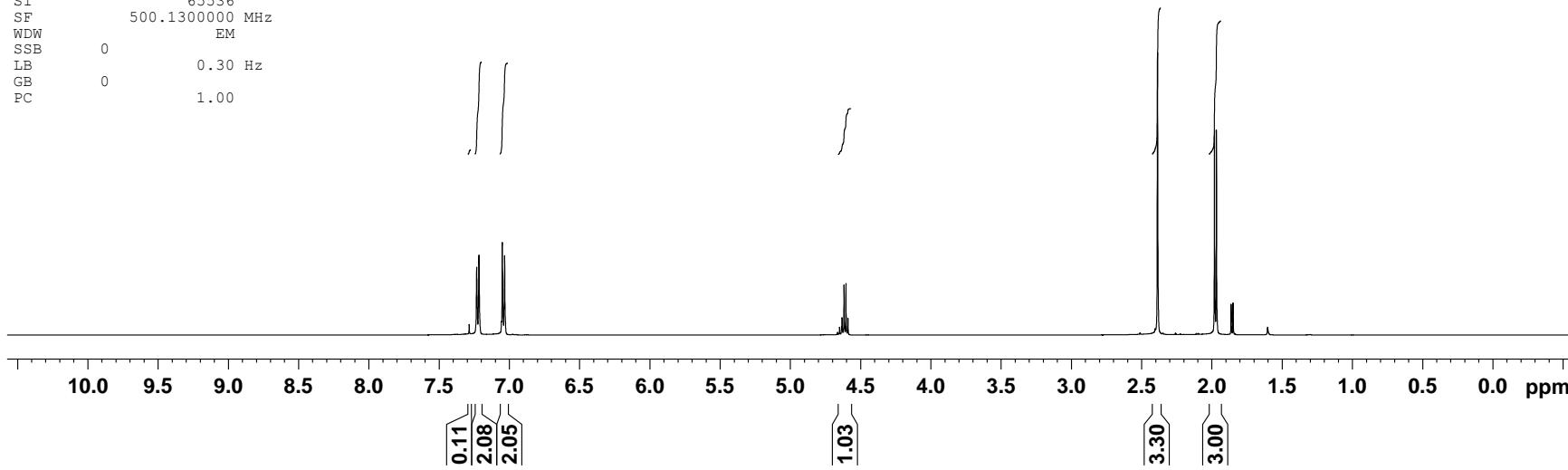
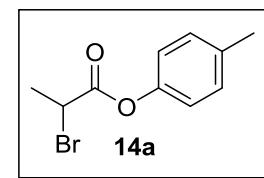


Fig S1. <sup>1</sup>H NMR Spectrum of 14a

Current Data Parameters  
NAME INN-NSS-BE-06-13C  
EXPNO 2  
PROCNO 1

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Time 19.34  
INSTRUM spect  
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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  
DW 16.800 usec  
DE 6.50 usec  
TE 297.9 K  
D1 1.0000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.0000000 W

===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 13.0000000 W  
PLW12 0.34327999 W  
PLW13 0.21969999 W

F2 - Processing parameters  
SI 32768  
SF 125.7577733 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

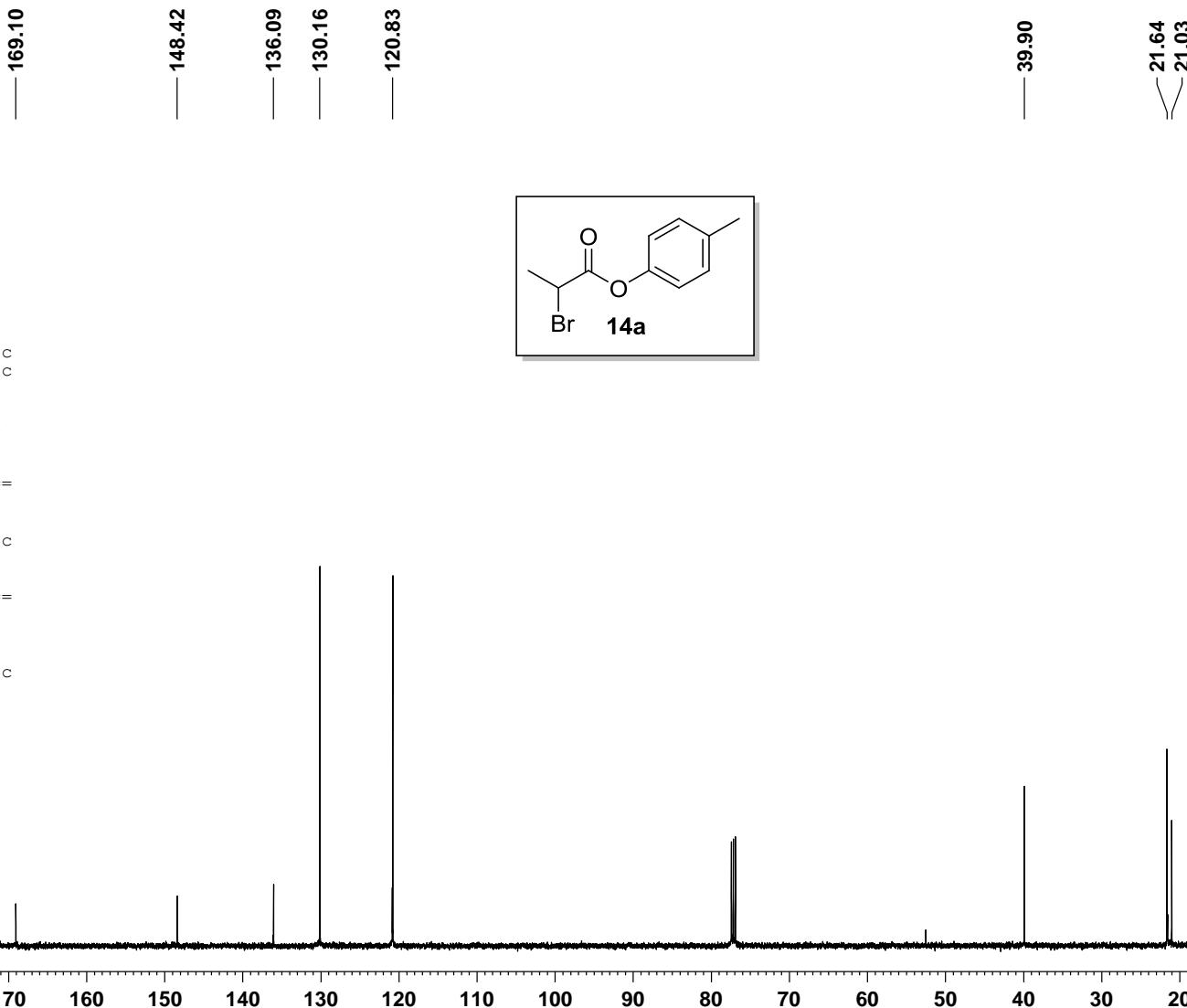


Fig S2. <sup>13</sup>C NMR Spectrum of 14a

Current Data Parameters  
NAME INN-NSS-BE-7-1H  
EXPNO 3  
PROCNO 1

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Time 19.24  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 13  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 90.5  
DW 60.800 usec  
DE 6.50 usec  
TE 294.6 K  
D1 1.0000000 sec  
TD0 1

## ===== CHANNEL f1 =====

NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

## F2 - Processing parameters

SI 32768  
SF 400.1299981 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

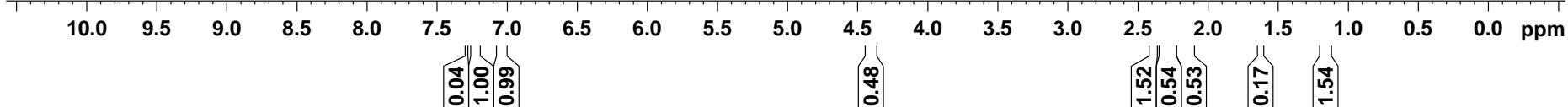
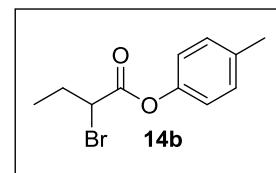


Fig S3. <sup>1</sup>H NMR Spectrum of 14b

Current Data Parameters  
NAME INN-NSS-BE-7-13C  
EXPNO 9  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20130921  
Time 19.28  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 78  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 2050  
DW 20.800 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.0000000 sec  
D11 0.0300000 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 =====

CPDPRG[2 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

## F2 - Processing parameters

SI 32768  
SF 100.6127550 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

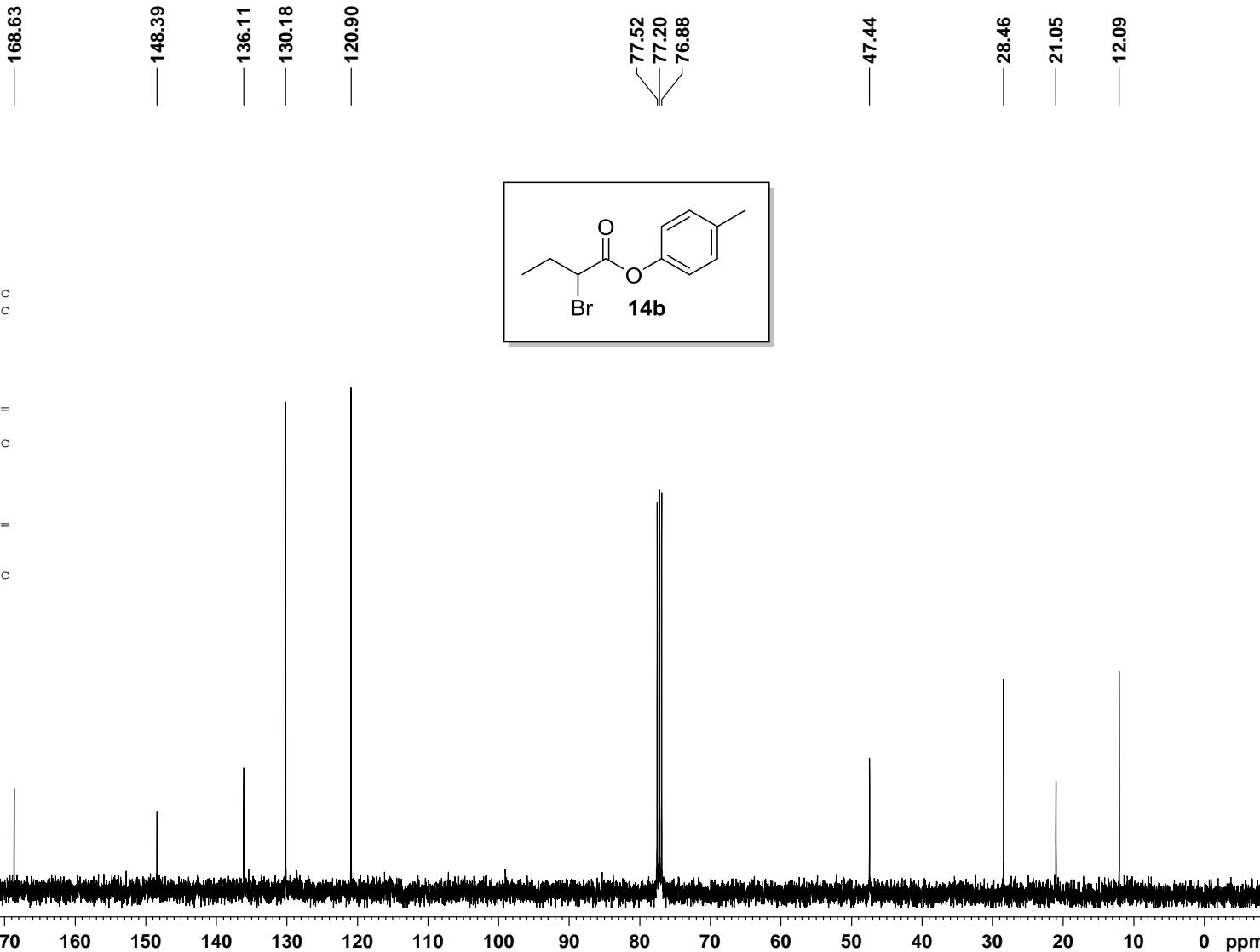


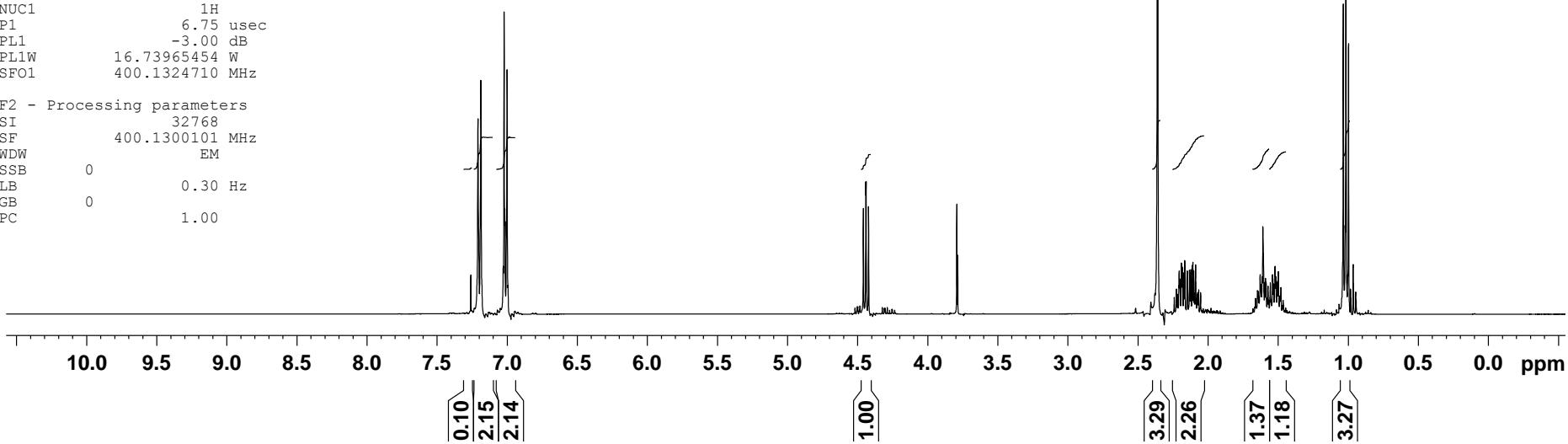
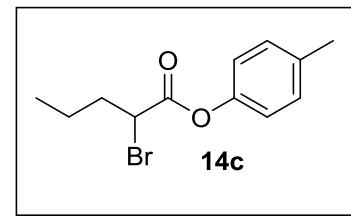
Fig S4. <sup>13</sup>C NMR Spectrum of **14b**

Current Data Parameters  
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 PROCNO 1  
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 Time 17.40  
 INSTRUM spect  
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 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 9  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9845889 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.8 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====

NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S5.** <sup>1</sup>H NMR Spectrum of **14c**

Current Data Parameters  
 NAME INN-NSS-NE-11-13C  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20131011  
 Time 17.41  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 52  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 295.3 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 =====

CPDPRG[2 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

F2 - Processing parameters

SI 32768  
 SF 100.6127579 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

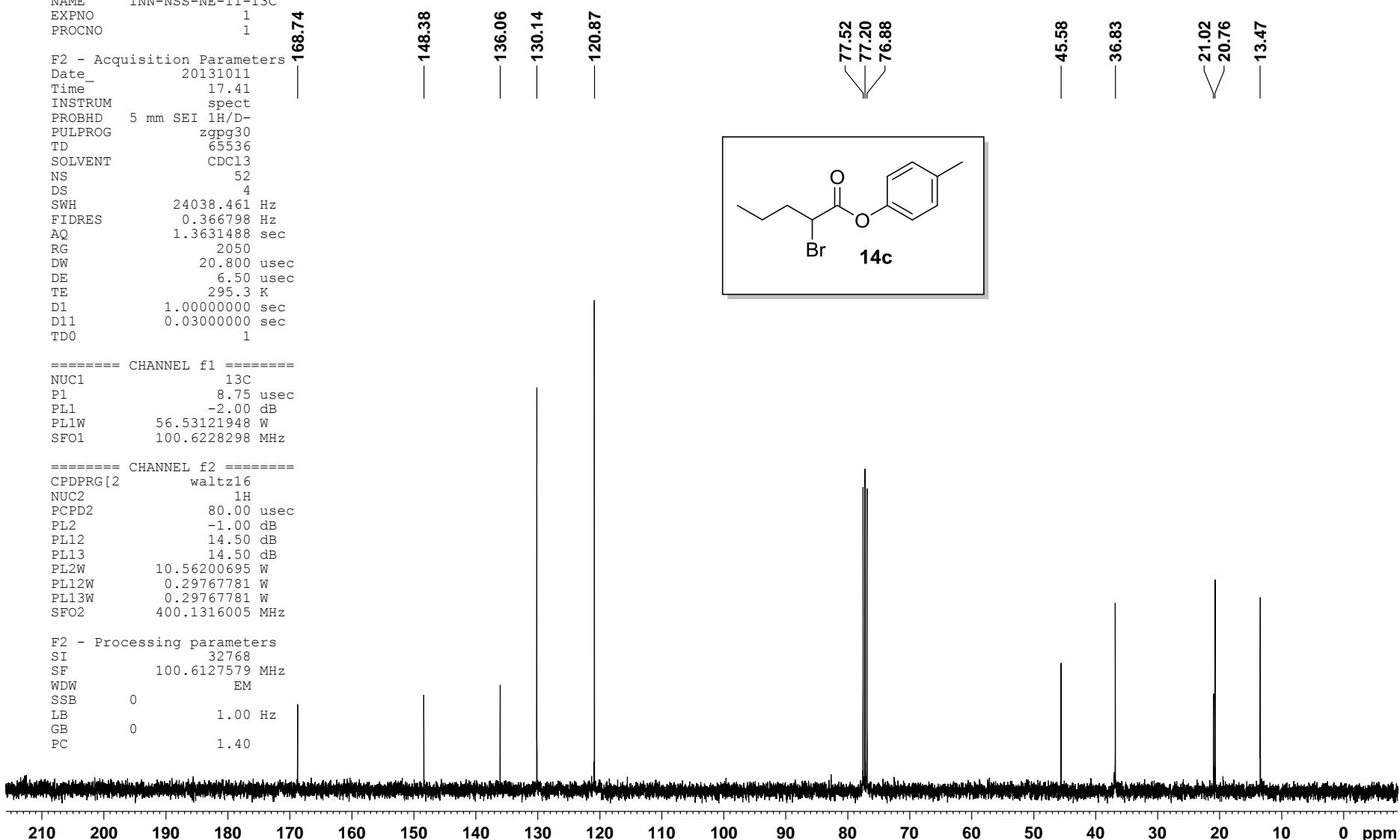


Fig S6. <sup>13</sup>C NMR Spectrum of **14c**

Current Data Parameters  
NAME INN-NSS-BE-10-1H  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131108  
Time 19.25  
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 12.52  
DW 50.000 usec  
DE 6.50 usec  
TE 297.0 K  
D1 1.0000000 sec  
TD0 1

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

F2 - Processing parameters  
SI 65536  
SF 500.1300122 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

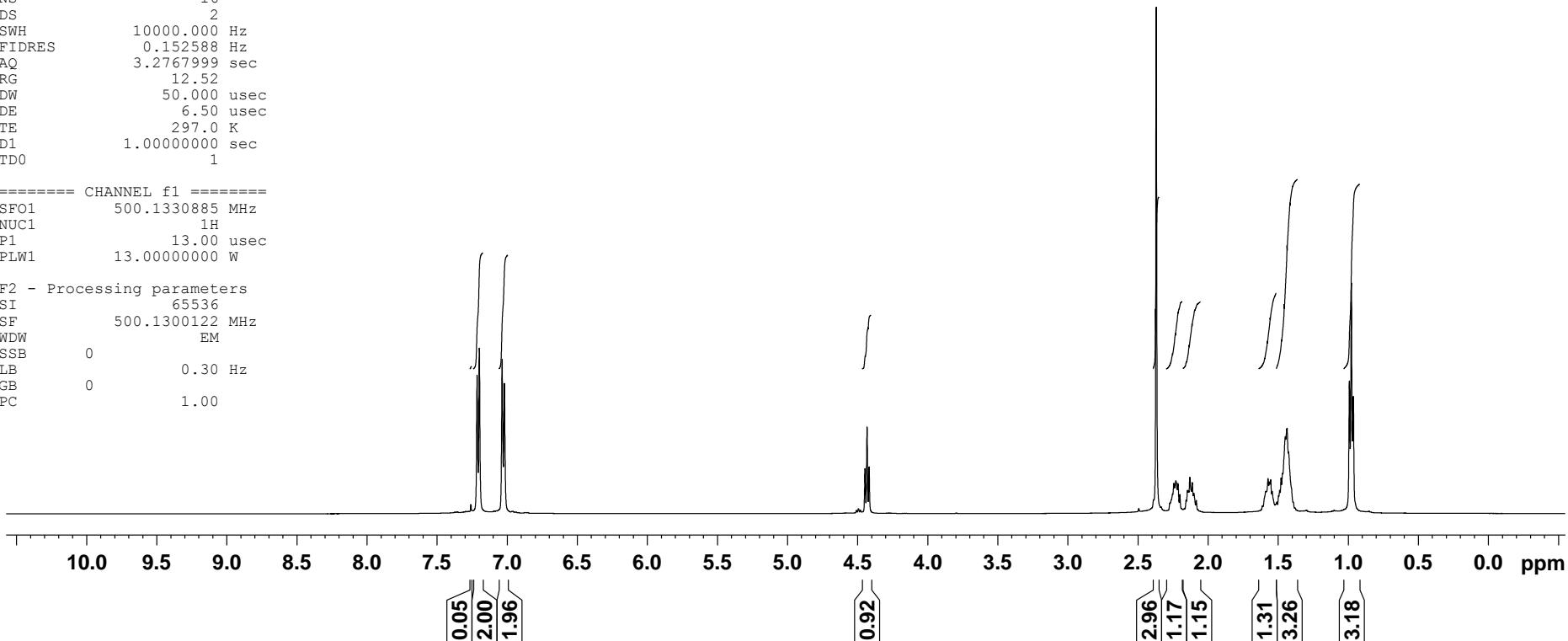
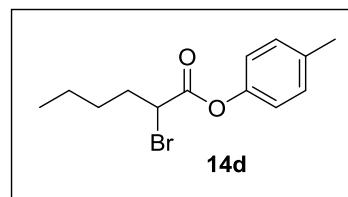


Fig S7. <sup>1</sup>H NMR Spectrum of 14d

Current Data Parameters  
 NAME INN-NSS-NE-10-13C  
 EXPNO 1  
 PROCNO 1

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 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 273  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 296.3 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 ======  
 CPDPRG[2 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

F2 - Processing parameters  
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 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

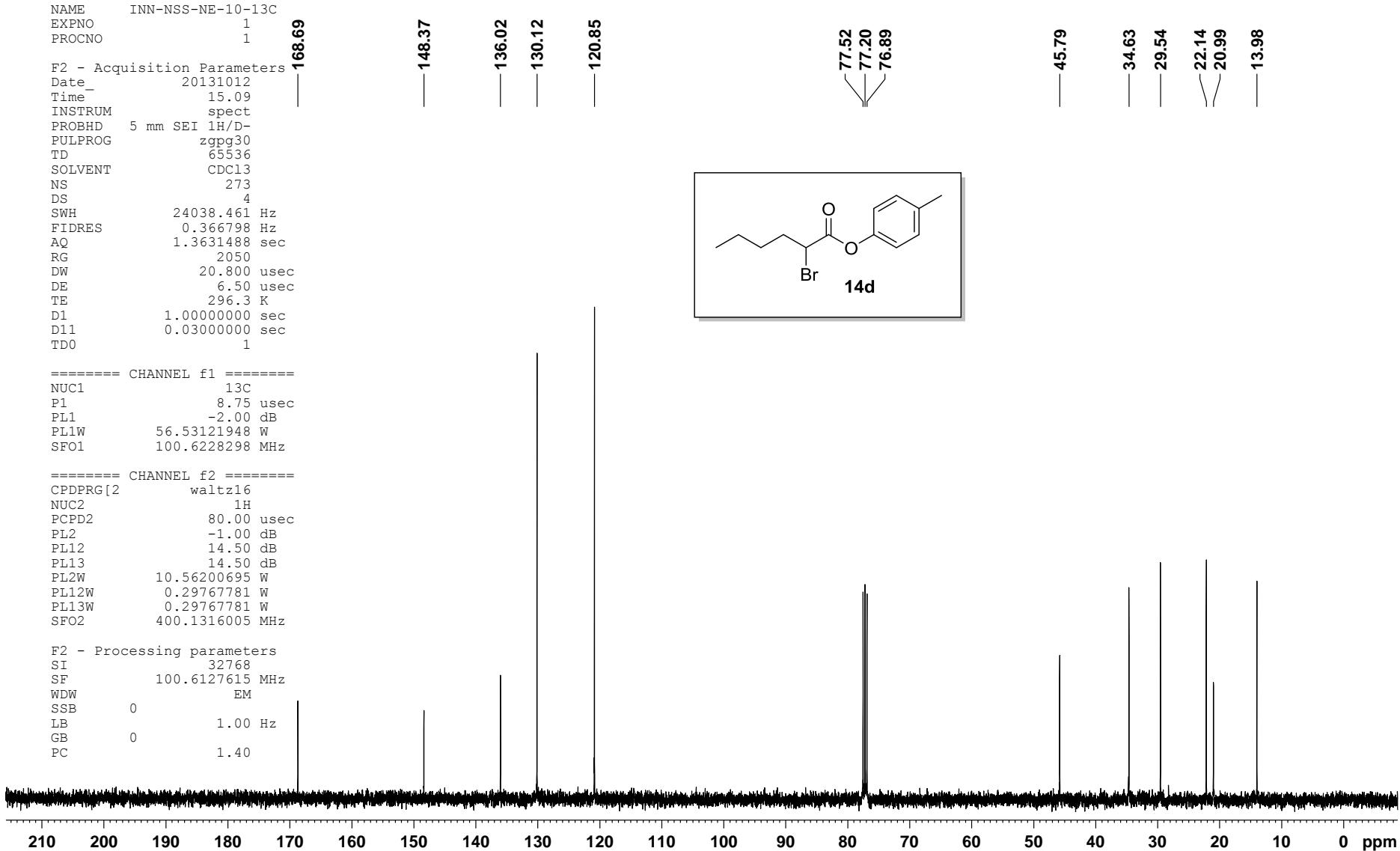


Fig S8.  $^{13}\text{C}$  NMR Spectrum of 14d

Current Data Parameters  
NAME INN-NSS-BE-12-1H  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131201  
Time 22.23  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 11  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 294.0 K  
D1 1.0000000 sec  
TD0 1

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

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

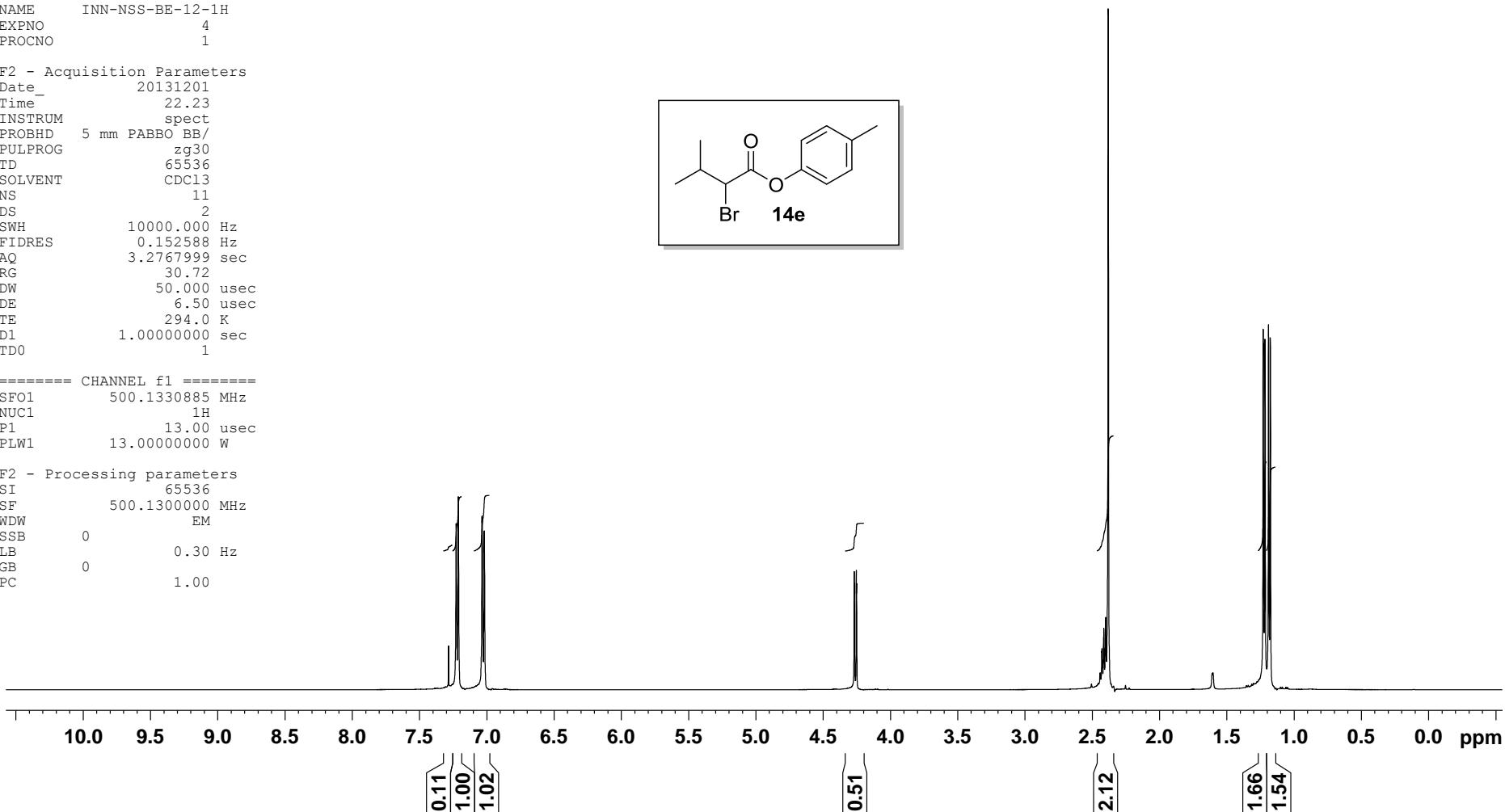
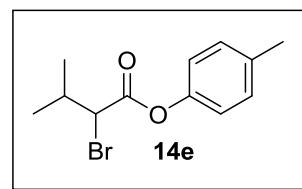


Fig S9. <sup>1</sup>H NMR Spectrum of 14e

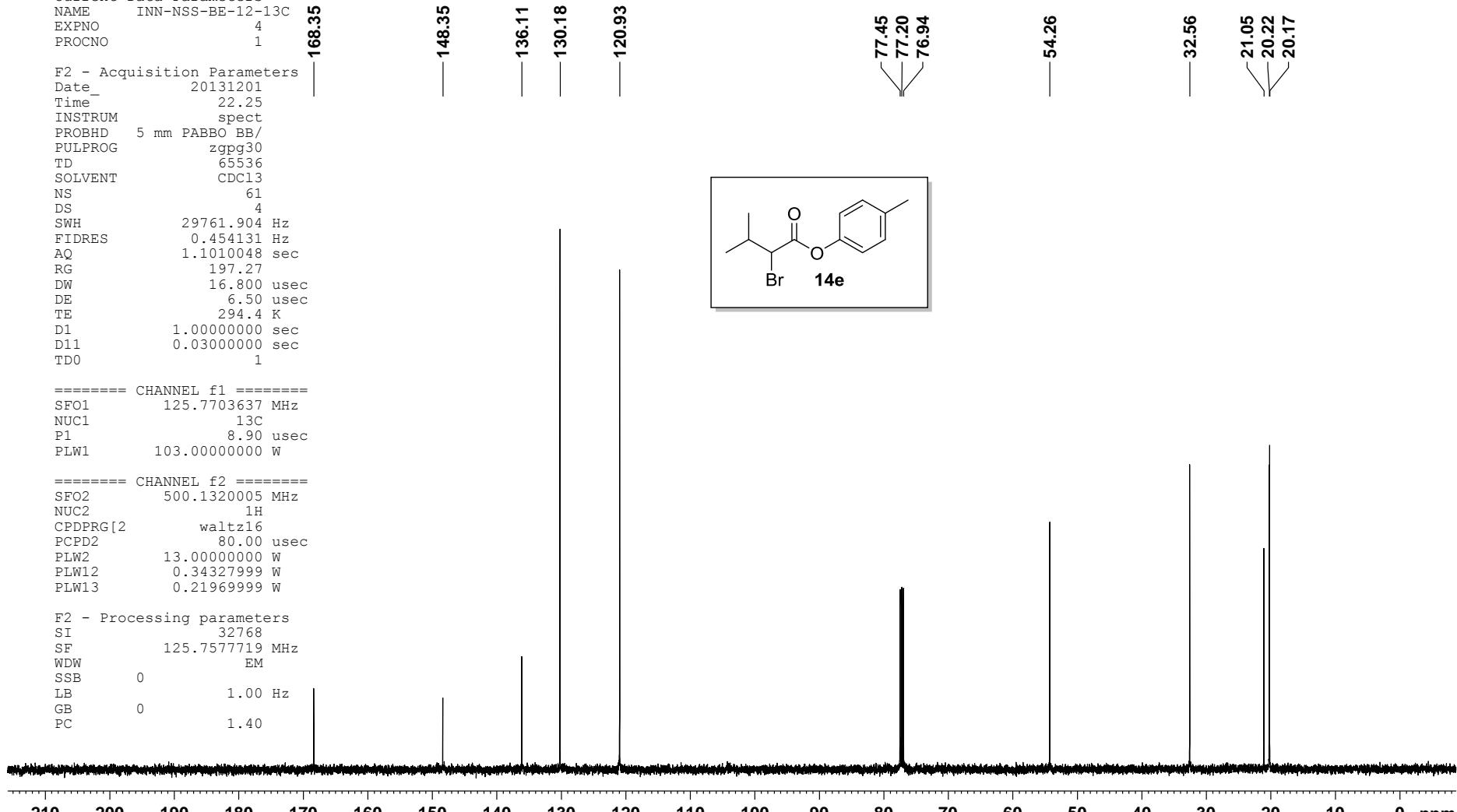
Current Data Parameters  
 NAME INN-NSS-BE-12-13C  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20131201  
 Time 22.25  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 61  
 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 294.4 K  
 D1 1.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

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

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

F2 - Processing parameters  
 SI 32768  
 SF 125.7577719 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0 1.40  
 PC



**Fig S10.**  $^{13}\text{C}$  NMR Spectrum of **14e**

Current Data Parameters  
NAME INN-NSS-BE-06-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date 20130505  
Time 2.33  
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 3.9845889 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 293.9 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

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

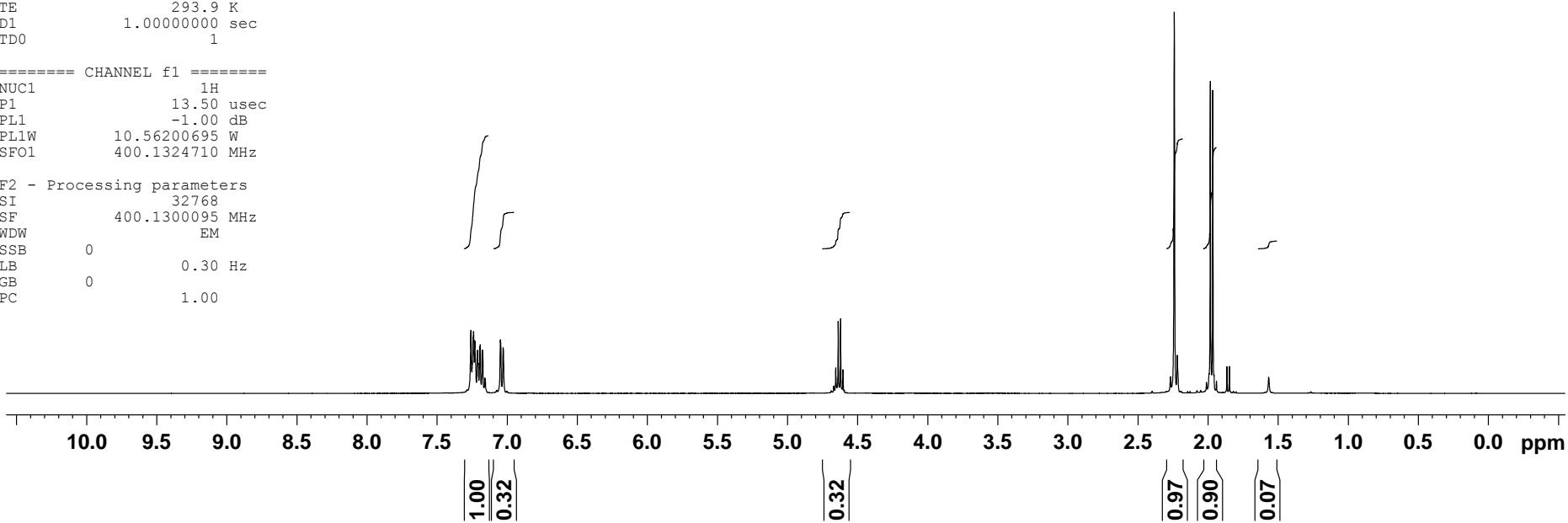
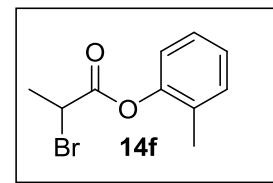


Fig S13. <sup>1</sup>H NMR Spectrum of **14f**

Current Data Parameters  
NAME INN-NSS-BE-06-13C  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130505  
Time 2.34  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 14  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 2050  
DW 20.800 usec  
DE 6.50 usec  
TE 294.0 K  
D1 1.0000000 sec  
D11 0.0300000 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 =====

CPDPRG[2 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

F2 - Processing parameters  
SI 32768  
SF 100.6127551 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

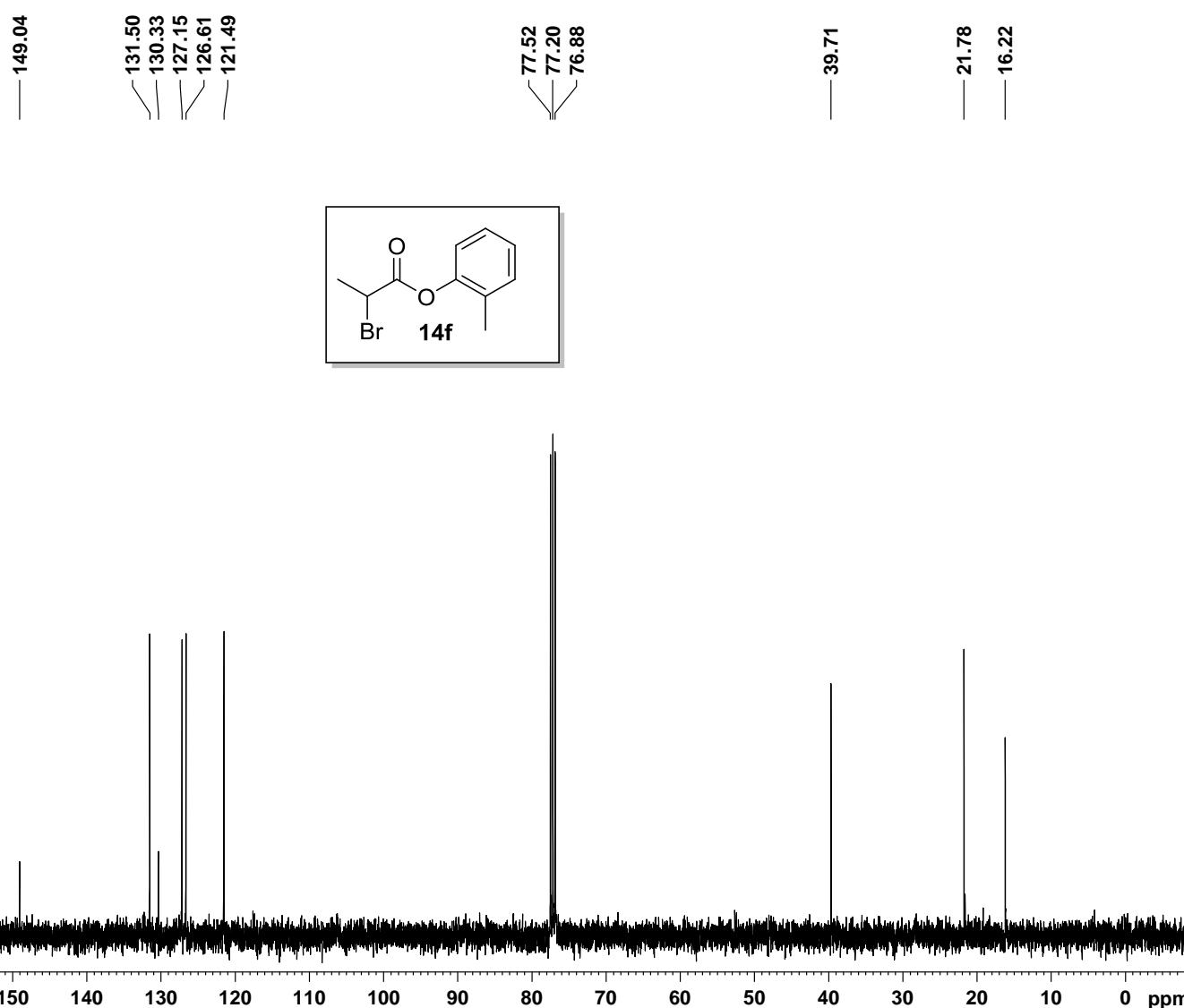


Fig S14.  $^{13}\text{C}$  NMR Spectrum of 14f

Current Data Parameters  
NAME INN-NSS-BE-7-1H  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130507  
Time 22.09  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 12  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 294.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====

NUC1 1H  
P1 13.50 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

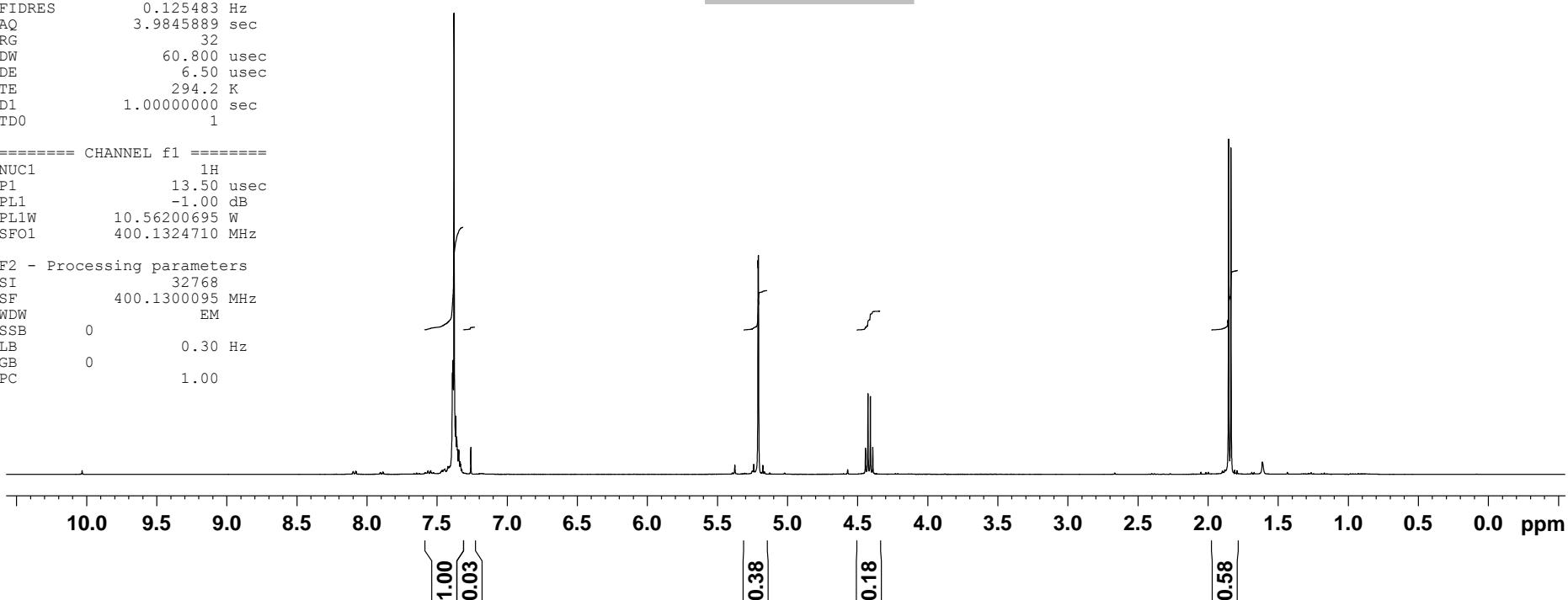
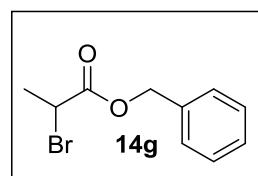


Fig S11. <sup>1</sup>H NMR Spectrum of 14g

Current Data Parameters  
 NAME INN-NSS-BE-7-13C  
 EXPNO 3  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20130507  
 Time 22.12  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 63  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 294.9 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 ======  
 CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127690 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

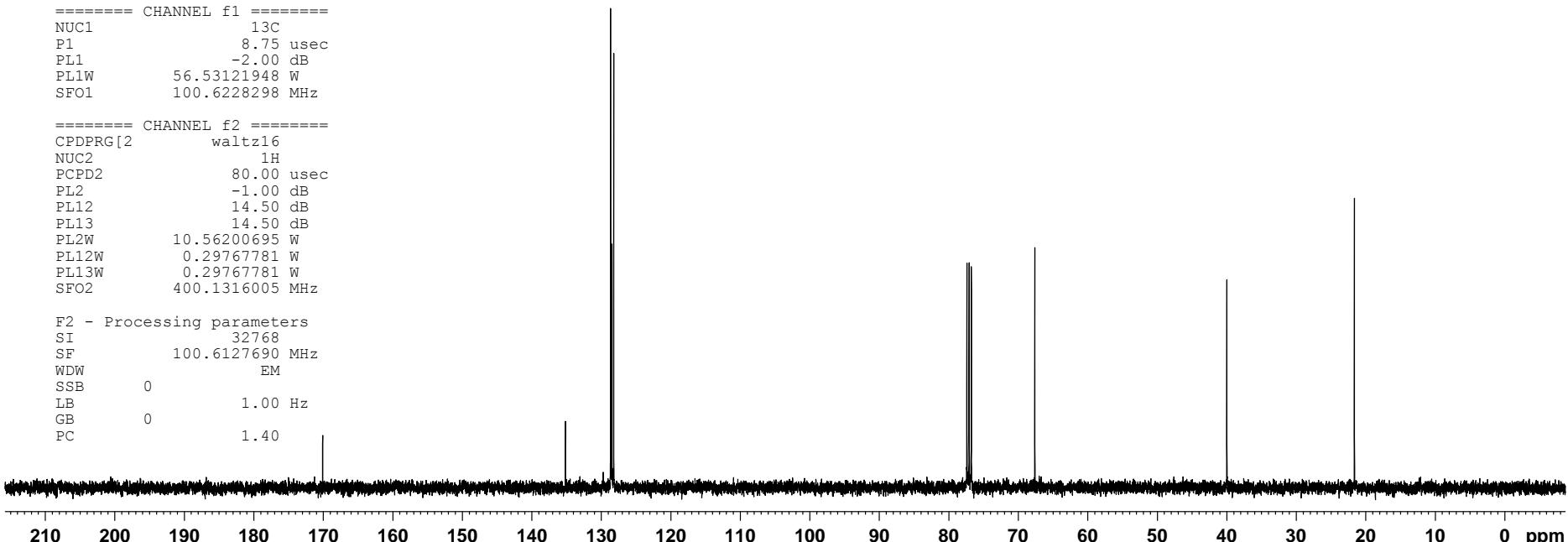
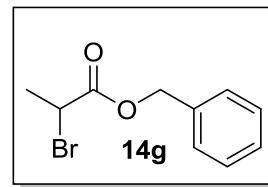
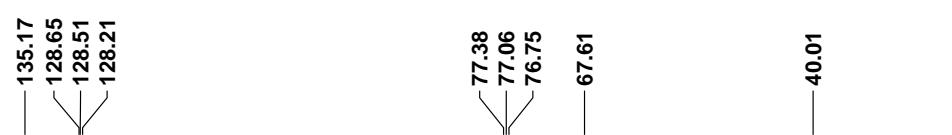


Fig S12.  $^{13}\text{C}$  NMR Spectrum of 14g

Current Data Parameters  
NAME INN-NS-NE-4-1H  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date 20130415  
Time 23.39  
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 3.9845889 sec  
RG 114  
DW 60.800 usec  
DE 6.50 usec  
TE 293.4 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

F2 - Processing parameters  
SI 32768  
SF 400.1300094 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

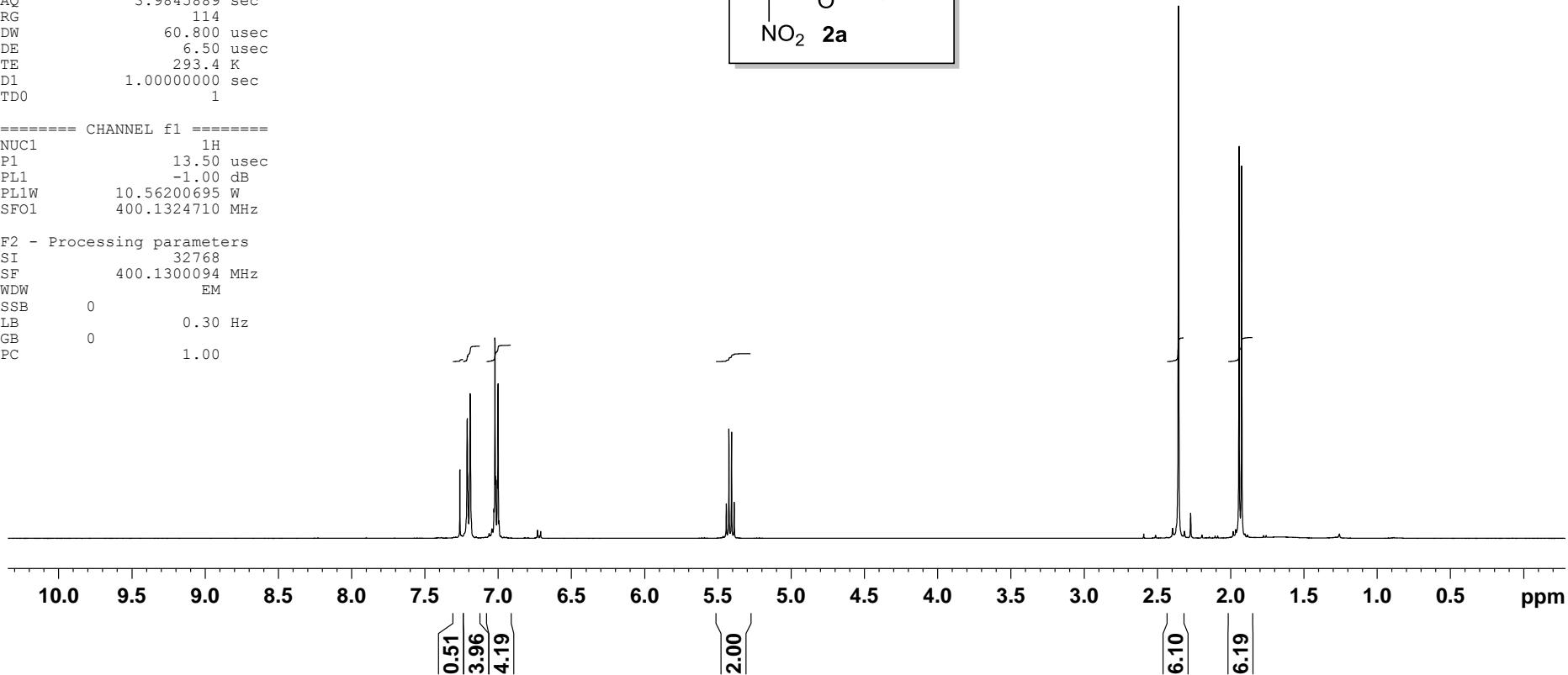
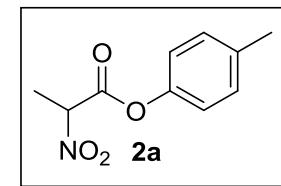


Fig S15. <sup>1</sup>H NMR Spectrum of 2a

Current Data Parameters  
 NAME INN-NS-NE-03-13C  
 EXPNO 14  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20130313  
 Time 19.45  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgppg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 38  
 DS 2  
 SWH 27573.529 Hz  
 FIDRES 0.420739 Hz  
 AQ 1.1883861 sec  
 RG 2050  
 DW 18.133 usec  
 DE 6.50 usec  
 TE 294.8 K  
 D1 1.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

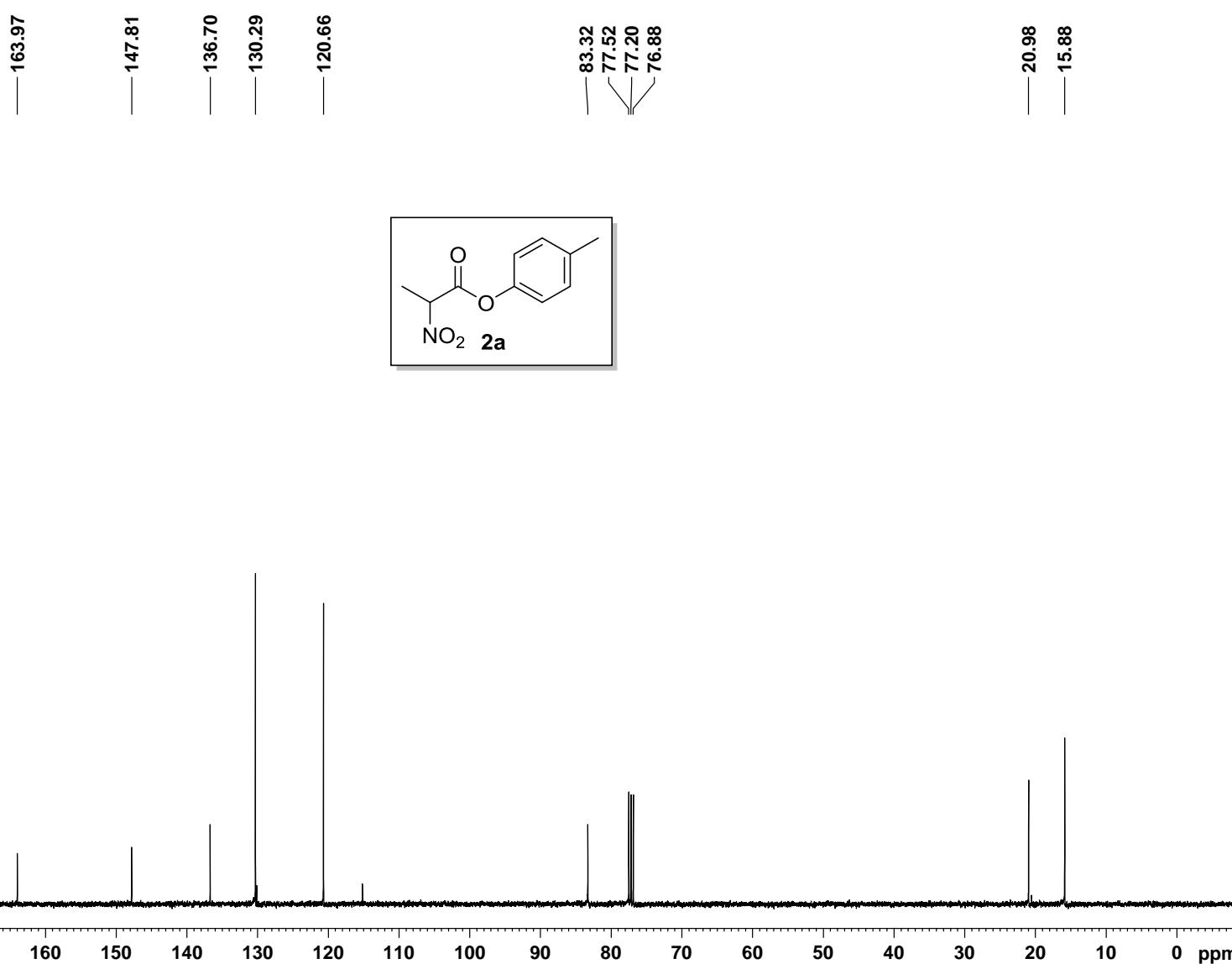
===== CHANNEL f1 =====

NUC1 13C  
 P1 8.75 usec  
 PL1 -2.00 dB  
 PL1W 56.53121948 W  
 SFO1 100.6242389 MHz

===== CHANNEL f2 =====

CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127598 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



**Fig S16.**  $^{13}\text{C}$  NMR Spectrum of **2a**

Current Data Parameters  
NAME INN-NSS-NE-7-1H  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130921  
Time 19.20  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 6  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 90.5  
DW 60.800 usec  
DE 6.50 usec  
TE 295.3 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====

NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1299981 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

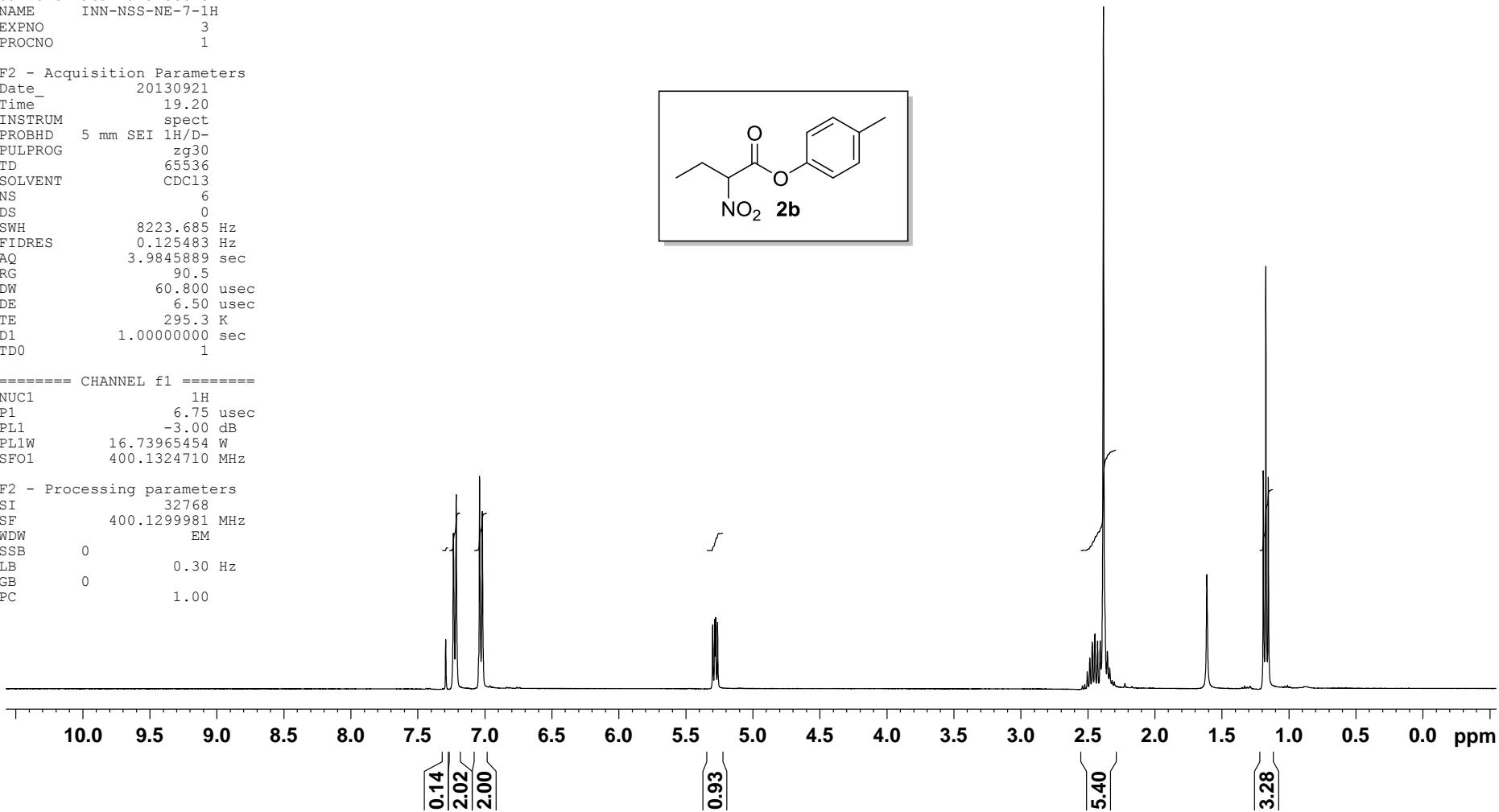
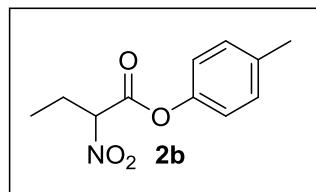


Fig S17. <sup>1</sup>H NMR Spectrum of 2b

Current Data Parameters  
 NAME INN-NSS-NE-7-13C  
 EXPNO 9  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20130921  
 Time 19.13  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 164  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 294.8 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 =====

CPDPRG[2 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

F2 - Processing parameters

SI 32768  
 SF 100.6127530 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

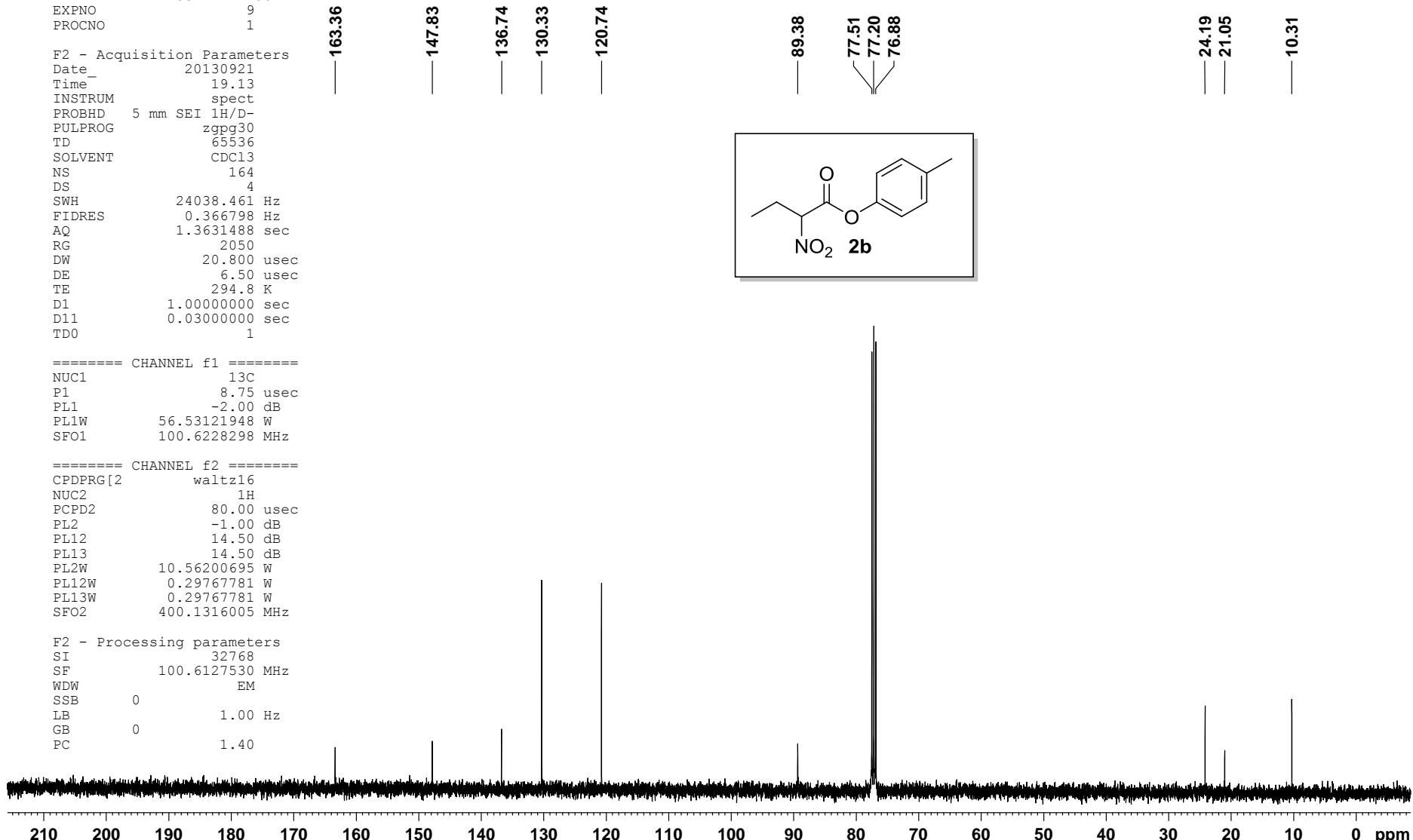


Fig S18. <sup>13</sup>C NMR Spectrum of 2b

Current Data Parameters  
 NAME INN-NSS-NE-11-1H  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20131128  
 Time 22.00  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 15  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9845889 sec  
 RG 114  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 297.2 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====

NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300102 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

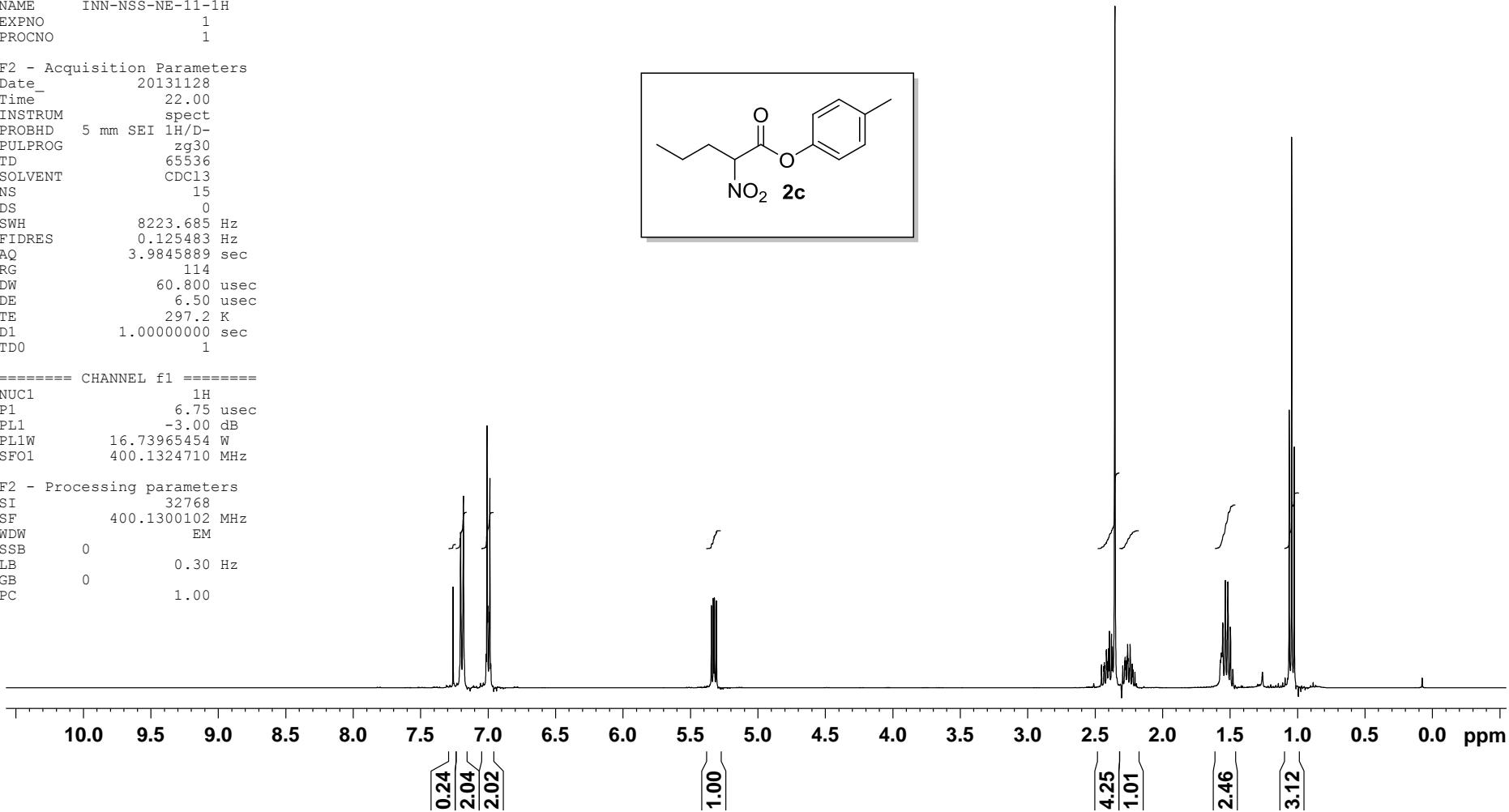
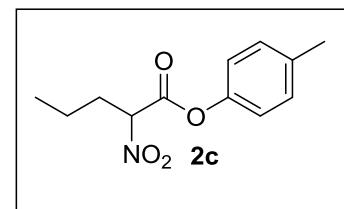
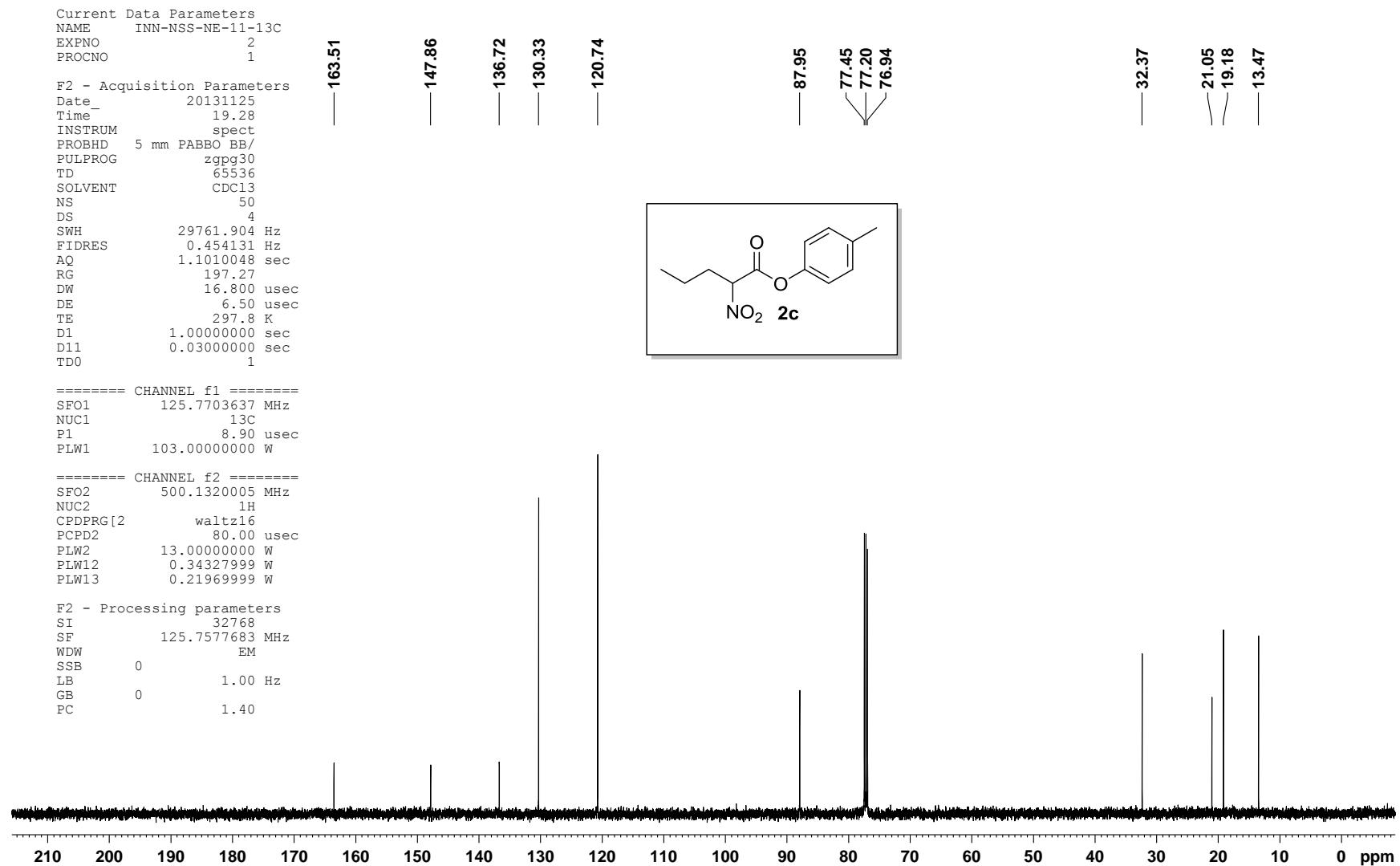


Fig S19.  $^{13}\text{C}$  NMR Spectrum of **2c**



**Fig S20.**  $^{13}\text{C}$  NMR Spectrum of **2c**

Current Data Parameters  
NAME INN-NSS-NE-10-1H  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131201  
Time 22.44  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 5  
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 294.0 K  
D1 1.0000000 sec  
TD0 1

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

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

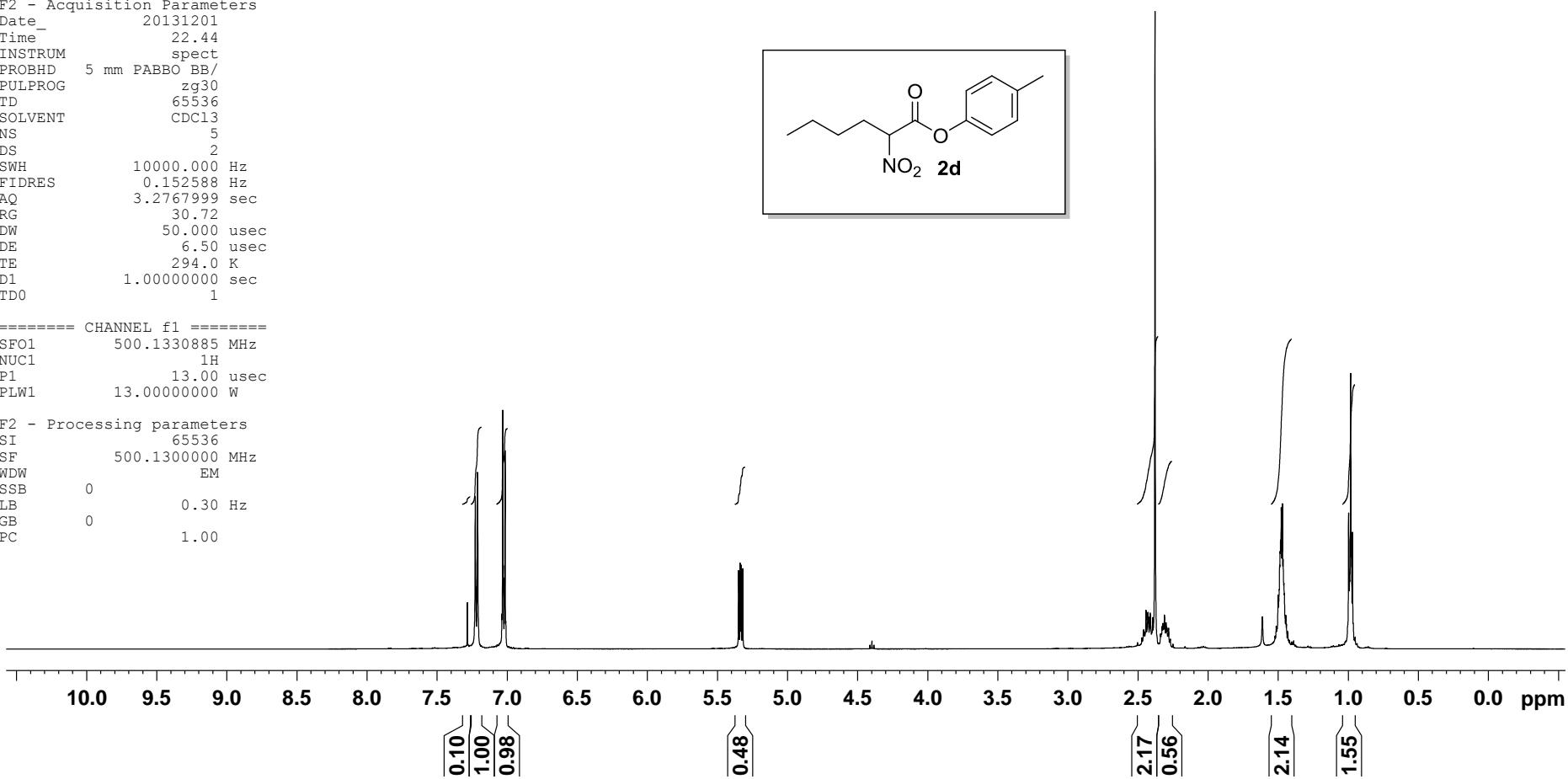
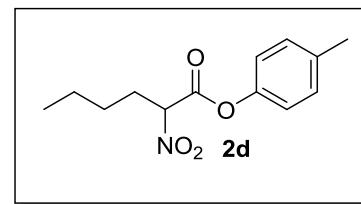


Fig S21. <sup>1</sup>H NMR Spectrum of 2d

Current Data Parameters  
 NAME INN-NSS-NS-10-13c  
 EXPNO 8  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 20131130  
 Time 15.24  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 101  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 296.9 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 =====  
 CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127535 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

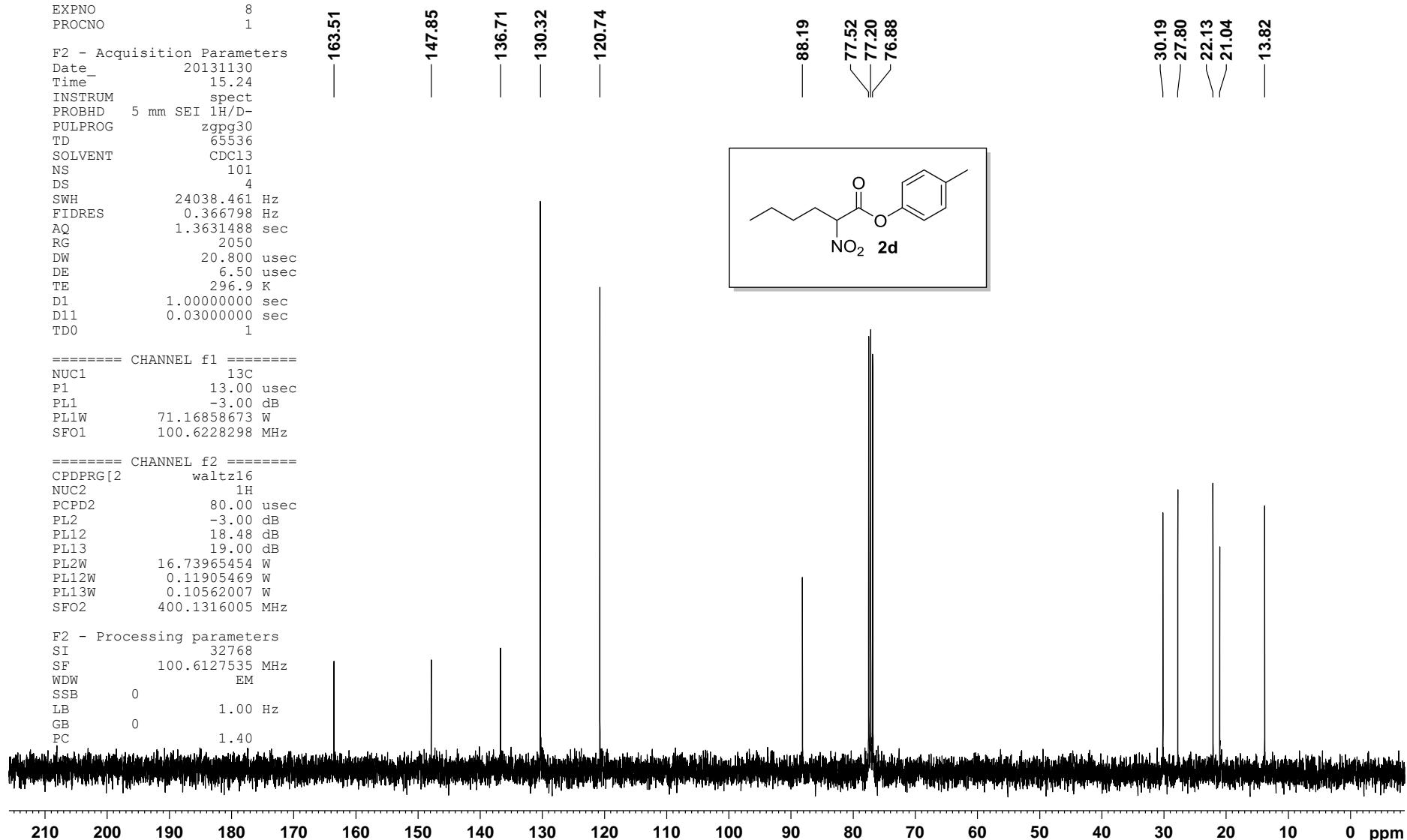


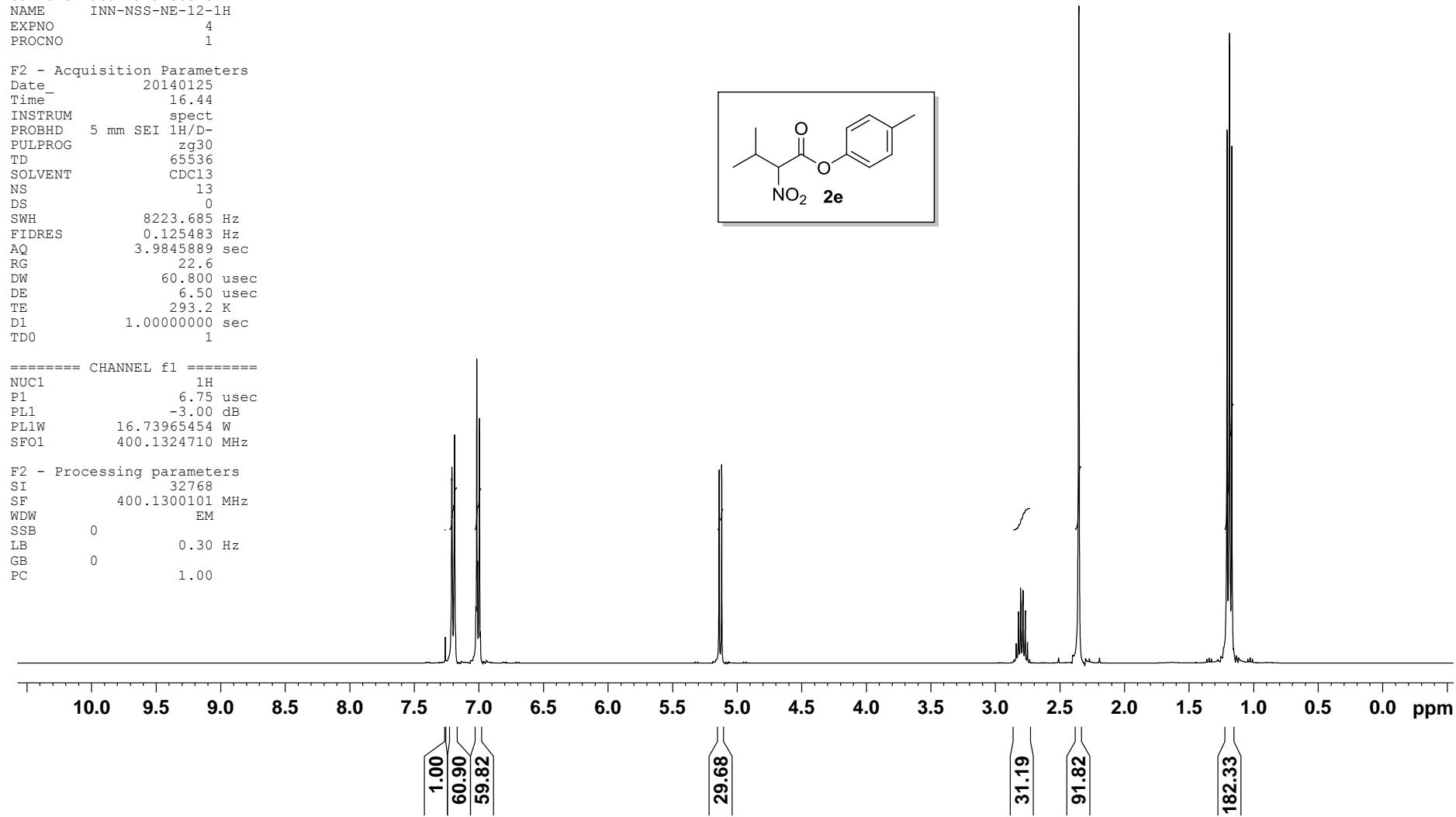
Fig S22. <sup>13</sup>C NMR Spectrum of 2d

Current Data Parameters  
 NAME INN-NSS-NE-12-1H  
 EXPNO 4  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20140125  
 Time 16.44  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 13  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9845889 sec  
 RG 22.6  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 293.2 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====

NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.7396454 W  
 SFO1 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S23.** <sup>1</sup>H NMR Spectrum of **2e**

Current Data Parameters  
 NAME INN-NSS-NE-12-13C  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20140125  
 Time 16.47  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 68  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 293.6 K  
 D1 1.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

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

===== CHANNEL f2 ======  
 CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127614 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

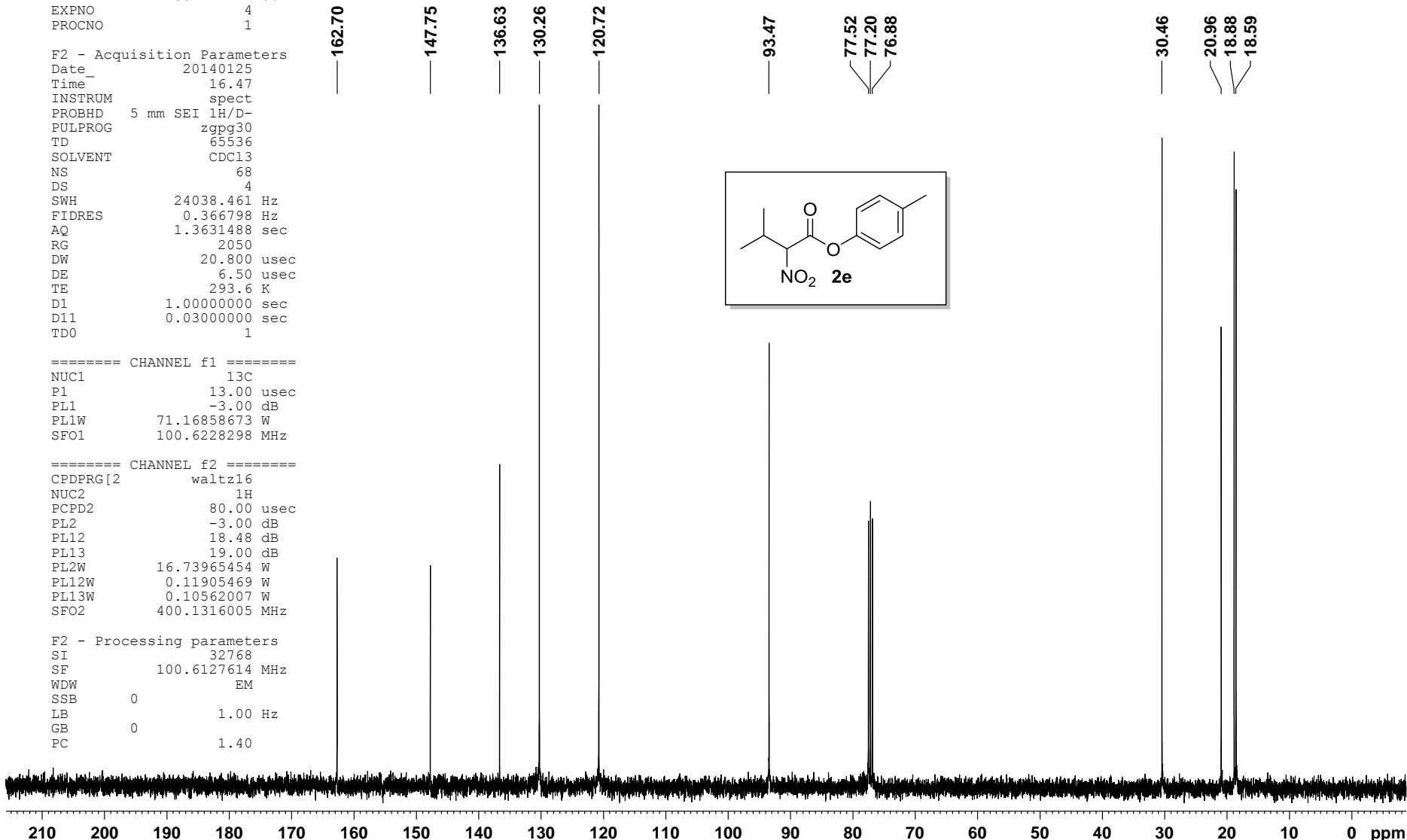


Fig S24.  $^{13}\text{C}$  NMR Spectrum of **2e**

Current Data Parameters  
NAME INN-NSS-NE-6-1H  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date 20130509  
Time 22.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 4  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 295.3 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 13.50 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

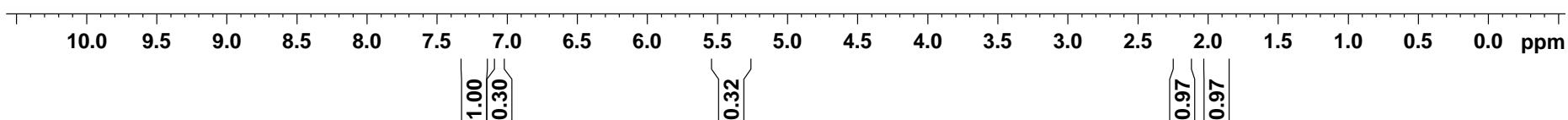
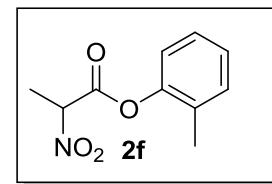


Fig S27. <sup>1</sup>H NMR Spectrum of **2f**

Current Data Parameters  
 NAME INN-NSS-NE-6-<sup>13</sup>C  
 EXPNO 4  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20130509  
 Time 22.28  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl<sub>3</sub>  
 NS 35  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 295.6 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 =====

CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127554 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

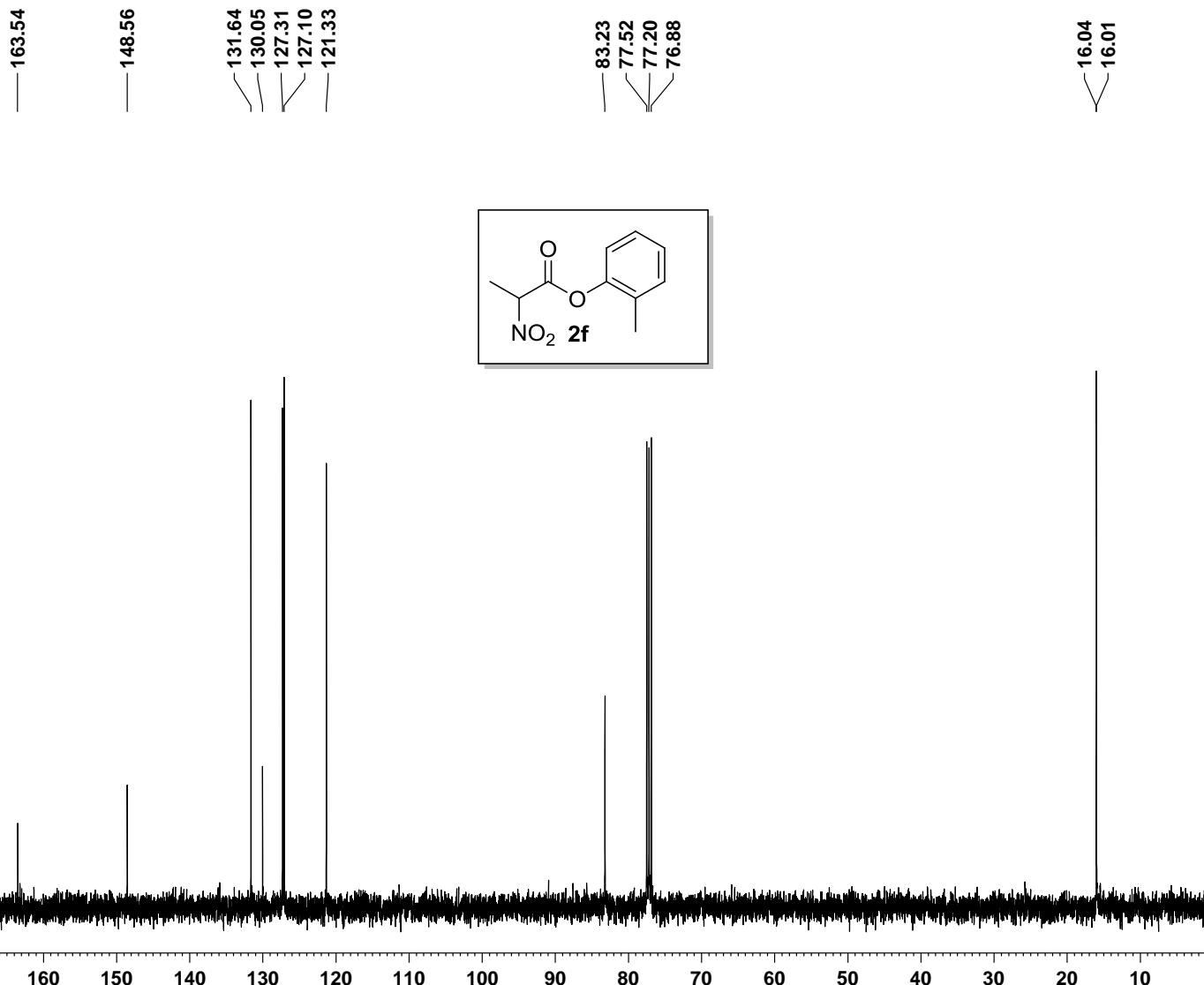


Fig S28. <sup>13</sup>C NMR Spectrum of 2f

Current Data Parameters  
NAME INN-NSS-NE-7-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130511  
Time 21.23  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 90.5  
DW 60.800 usec  
DE 6.50 usec  
TE 294.0 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 13.50 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

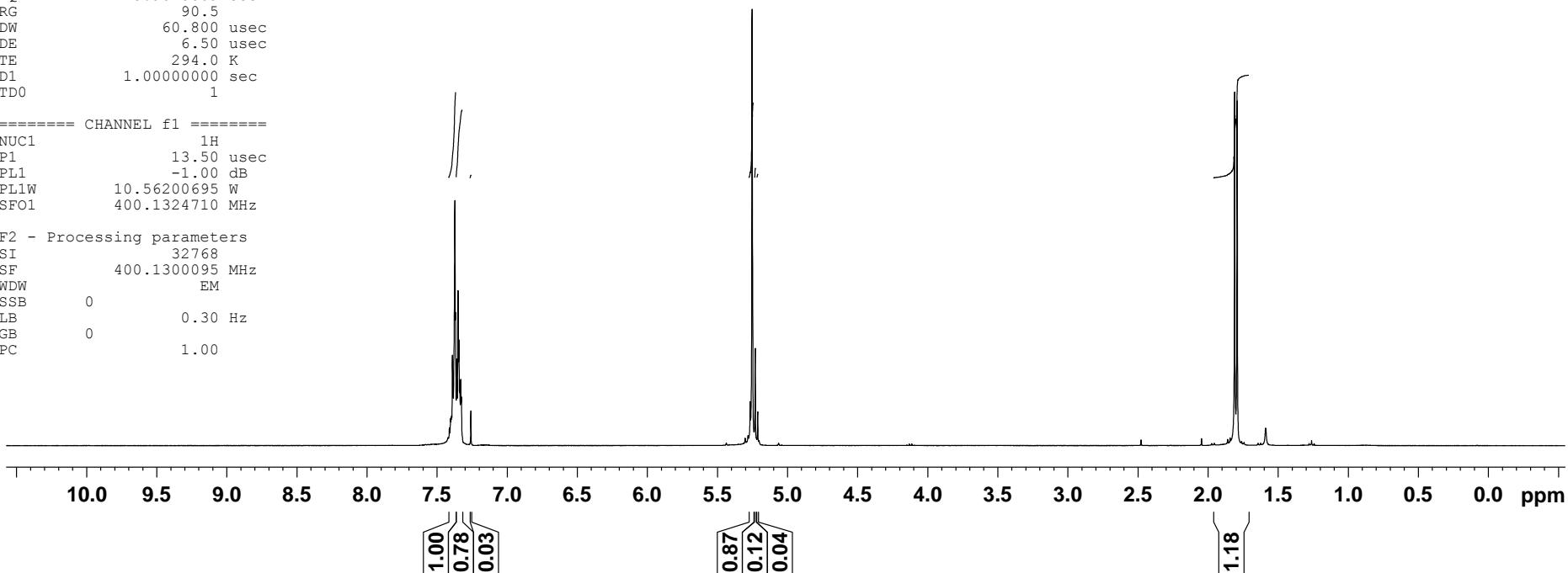
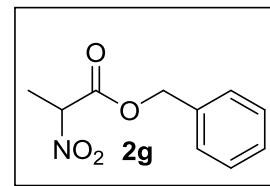


Fig S25.  $^1\text{H}$  NMR Spectrum of 2g

Current Data Parameters  
 NAME INN-NSS-NE-7-13C  
 EXPNO 14  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20130513  
 Time 17.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 903  
 DS 4  
 SWH 27777.777 Hz  
 FIDRES 0.423855 Hz  
 AQ 1.1796480 sec  
 RG 2050  
 DW 18.000 usec  
 DE 6.50 usec  
 TE 296.0 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 ======  
 CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6127552 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

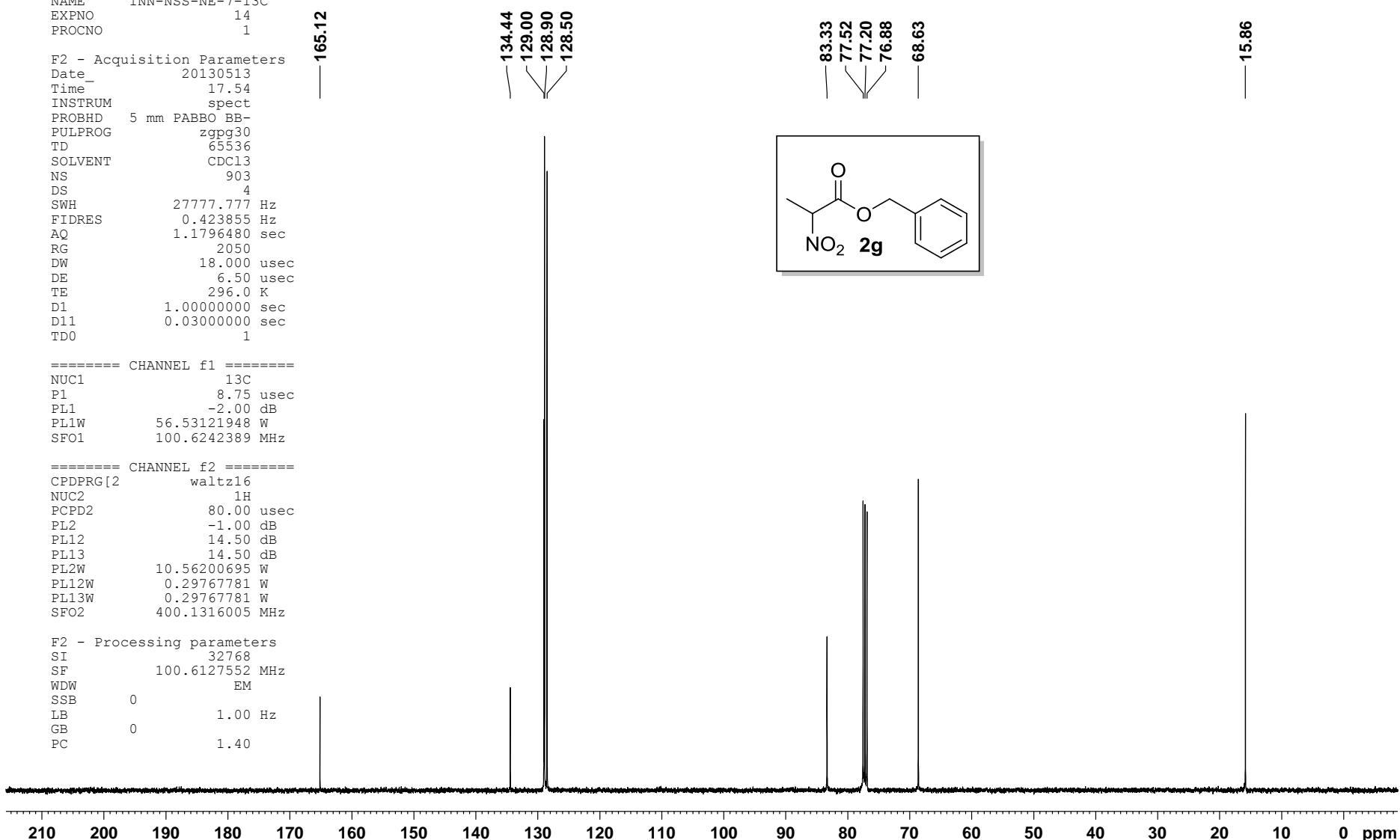
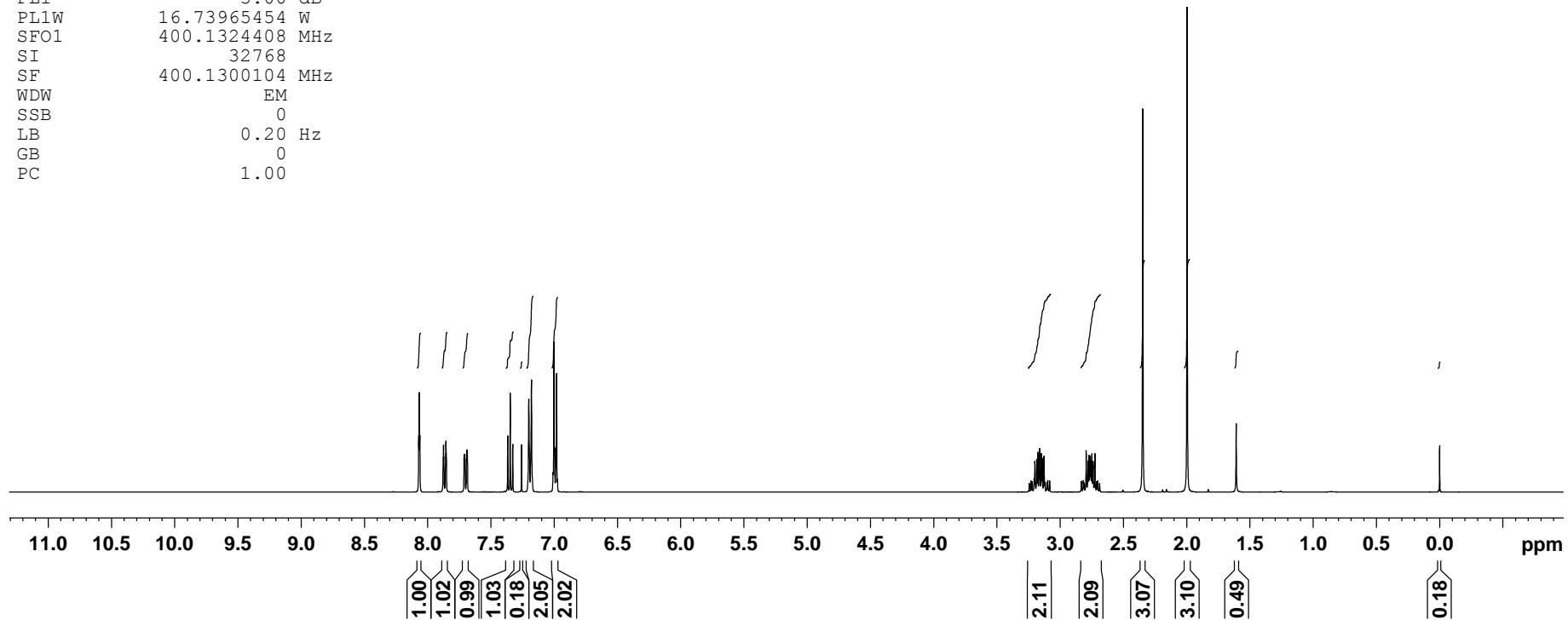
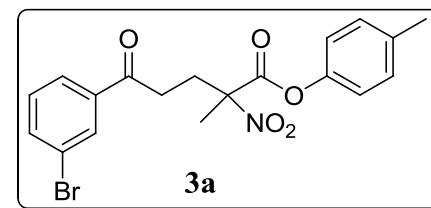


Fig S26.  $^{13}\text{C}$  NMR Spectrum of 2g

NAME INN-4-KSB-100-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130821  
 Time 11.15  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 52816  
 SOLVENT CDCl3  
 NS 14  
 DS 0  
 SWH 8802.817 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.999998 sec  
 RG 45.2  
 DW 56.800 usec  
 DE 6.50 usec  
 TE 293.5 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====

NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324408 MHz  
 SI 32768  
 SF 400.1300104 MHz  
 WDW EM  
 SSB 0  
 LB 0.20 Hz  
 GB 0  
 PC 1.00



**Fig S29.** <sup>1</sup>H NMR Spectrum of 3a

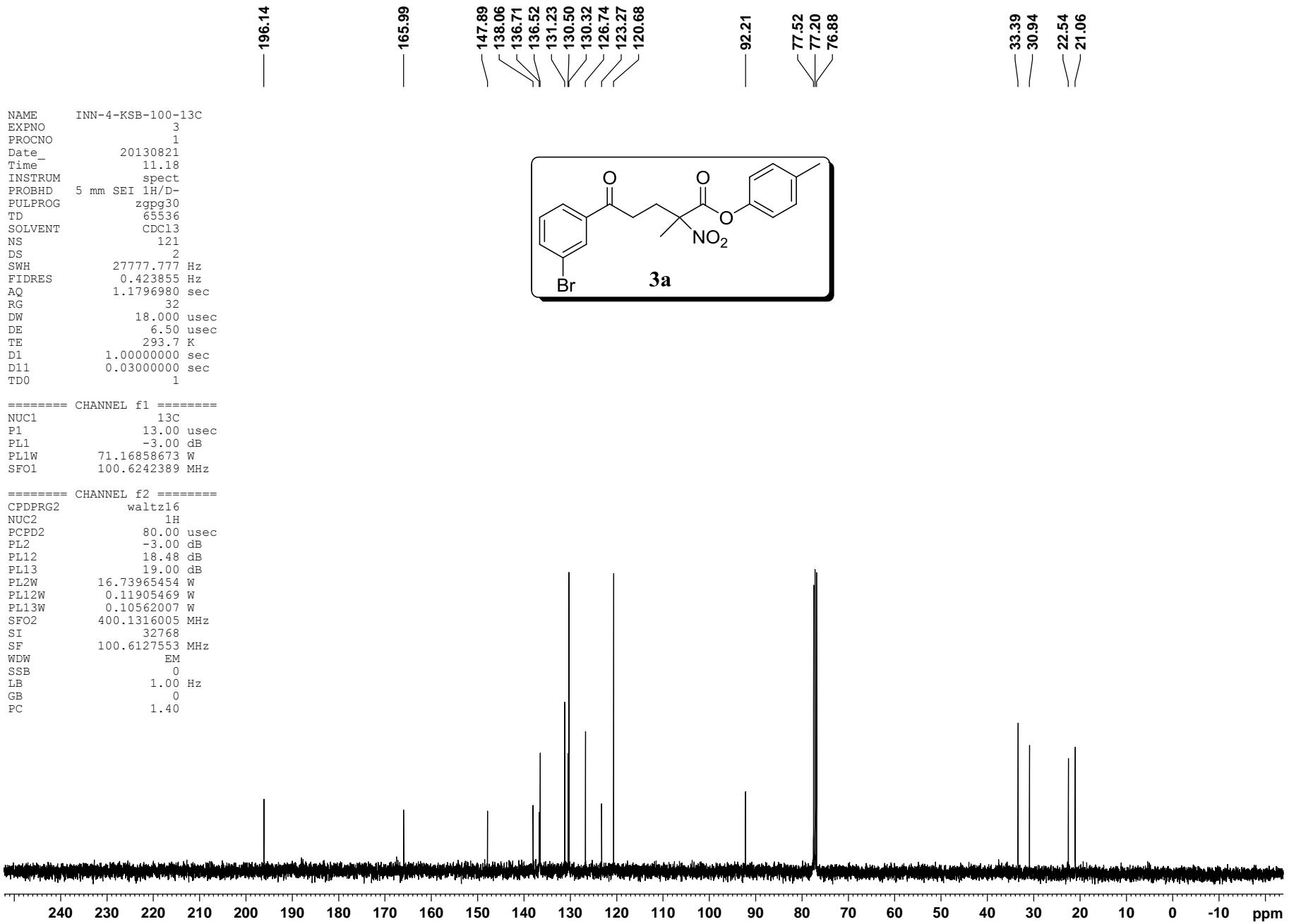
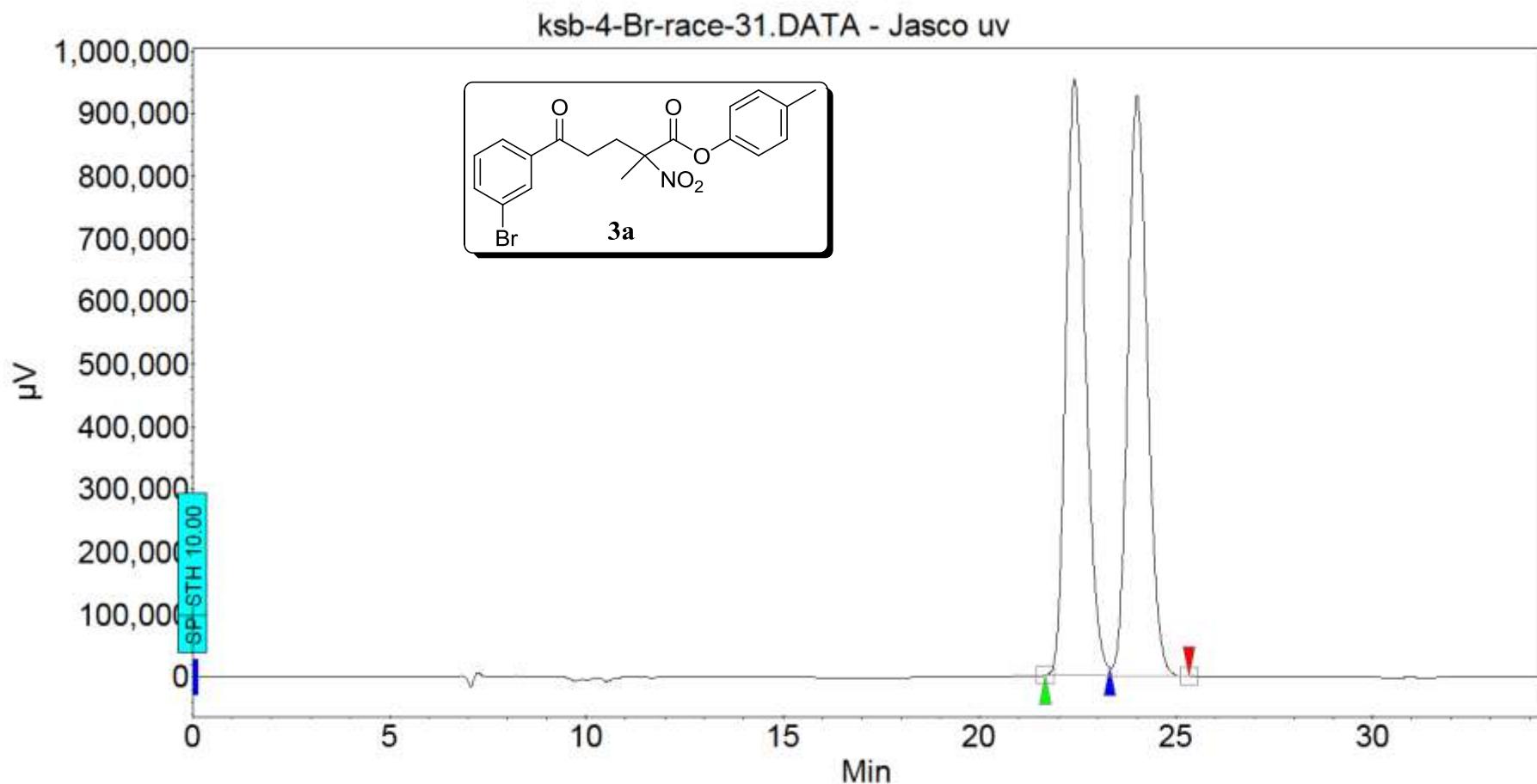


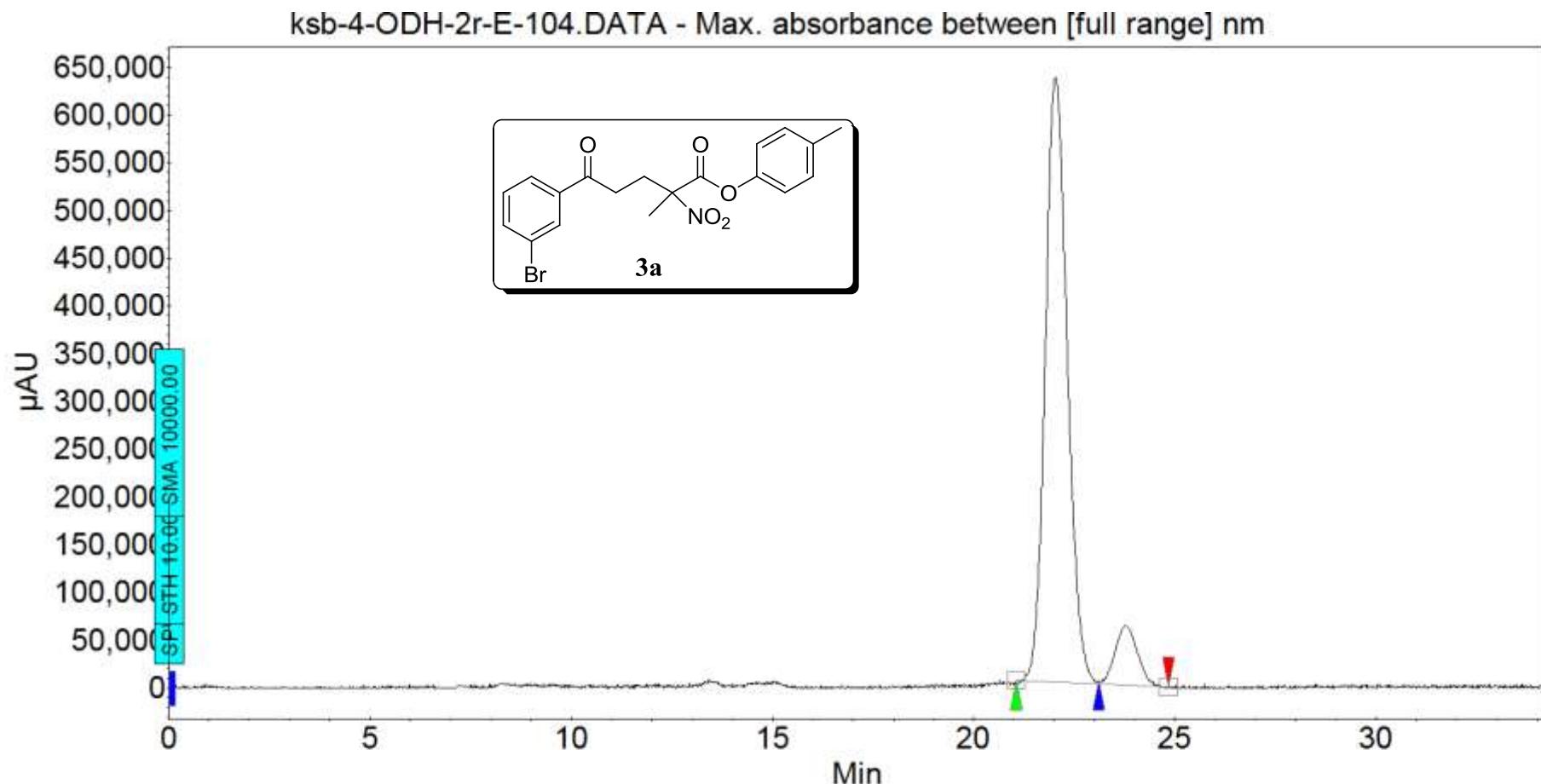
Fig S30.  $^{13}\text{C}$  NMR Spectrum of 3a



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	22.417	50.85	953860.3	550102.0	50.853
2	UNKNOWN	24.000	49.15	928312.7	531655.7	49.147
Total			100.00	1882173.0	1081757.6	100.000

Fig S31. HPLC Profile of Racemic 3a



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	22.025	91.01	632884.3	401347.2	91.013
2	UNKNOWN	23.772	8.99	61597.4	39628.5	8.987
Total			100.00	694481.7	440975.7	100.000

**Fig S32. HPLC Profile of Enantioenriched 3a**

Current Data Parameters  
NAME INN-4-KSB-121A-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date 20131018  
Time 22.22  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

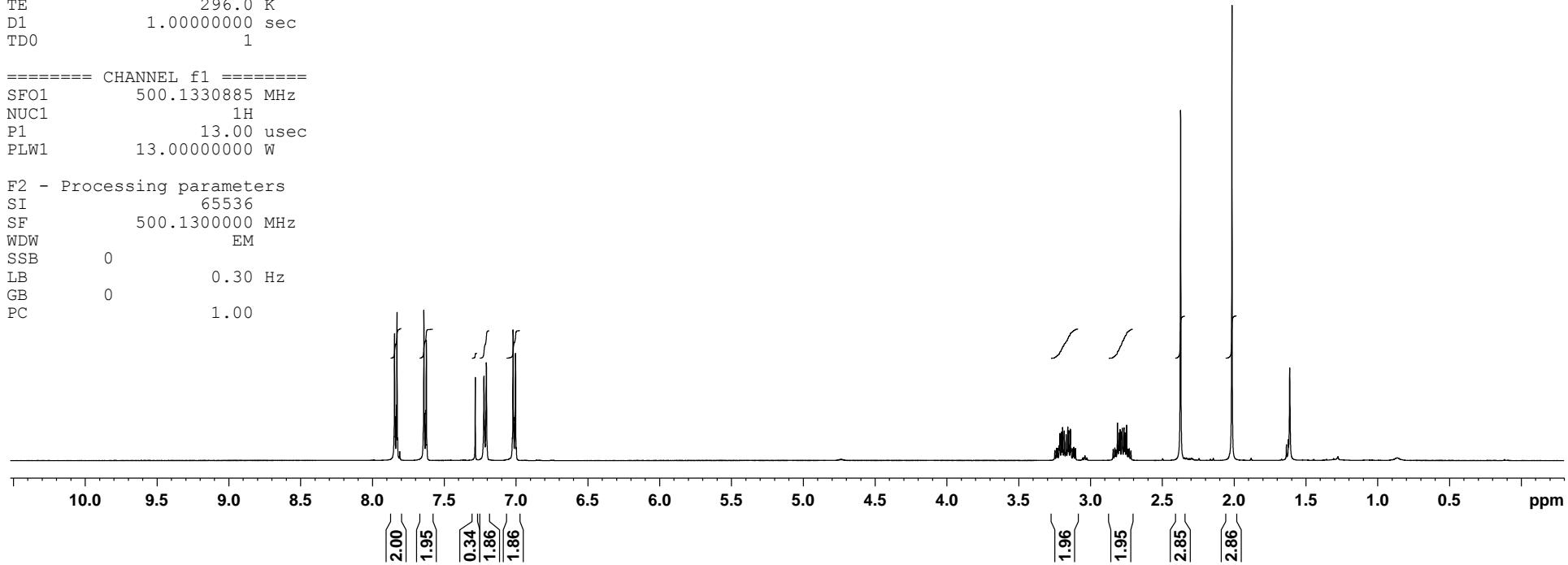
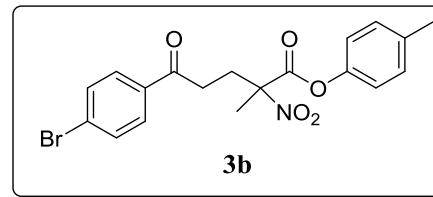


Fig S33. <sup>1</sup>H NMR Spectrum of **3b**

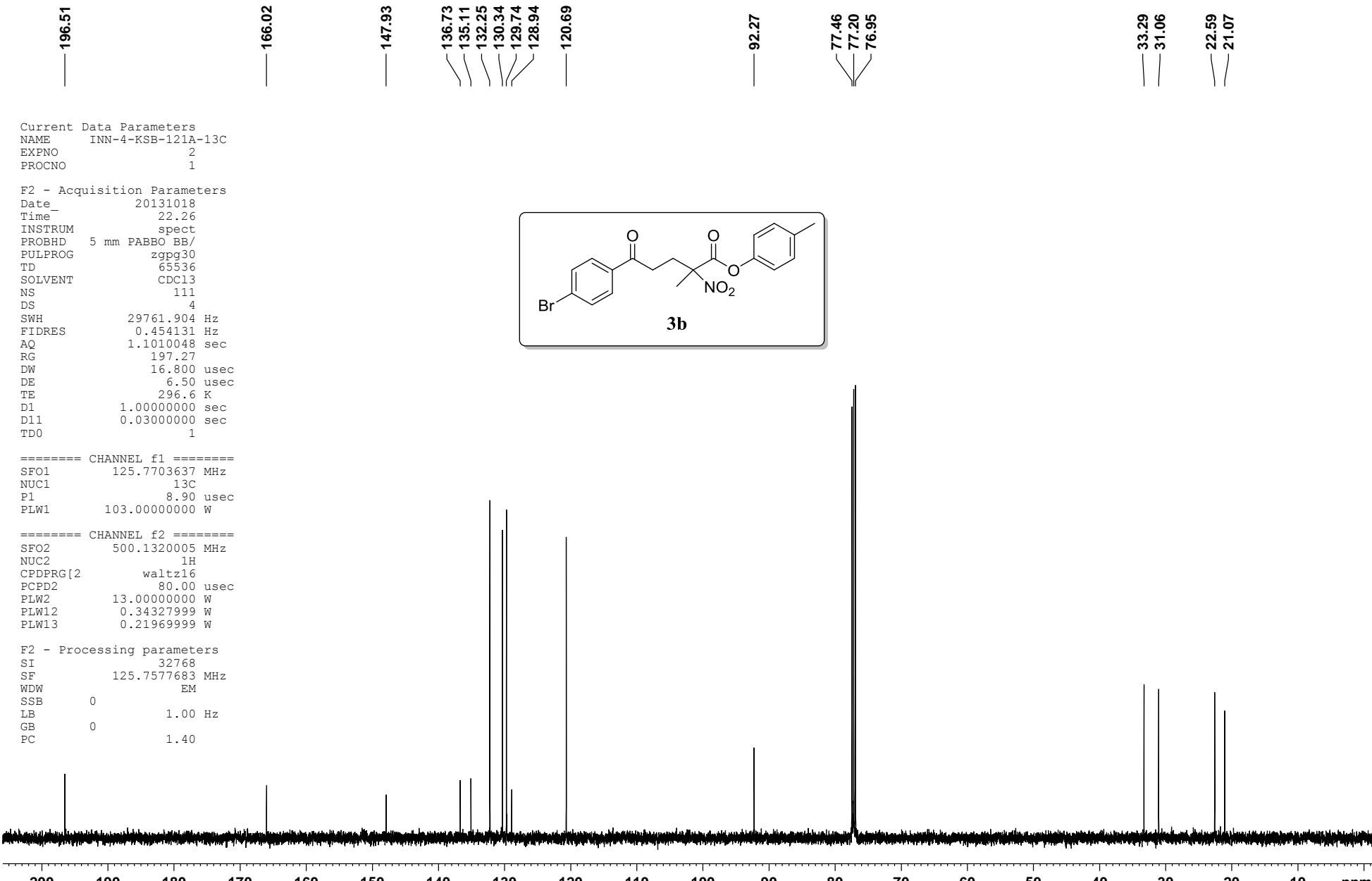
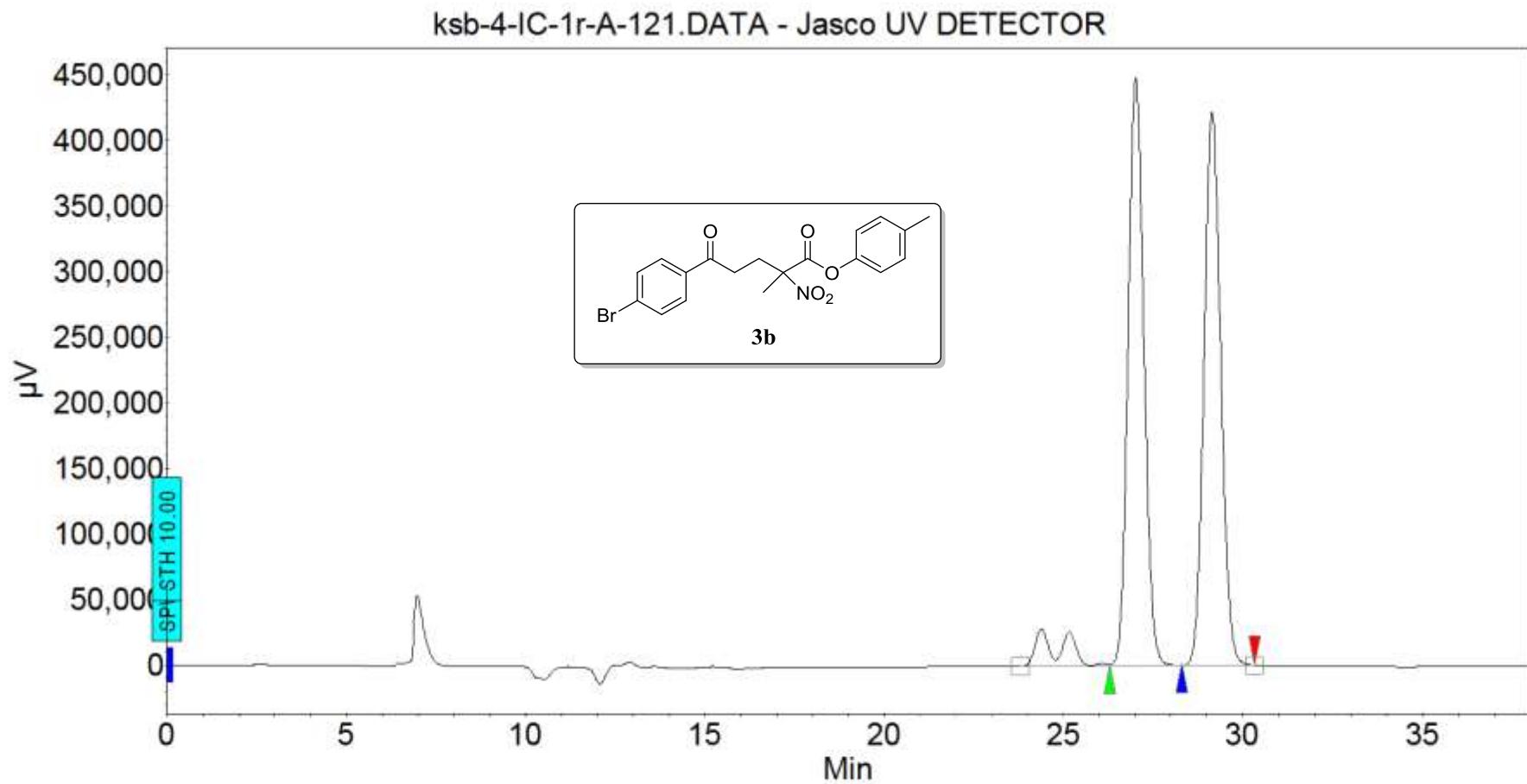


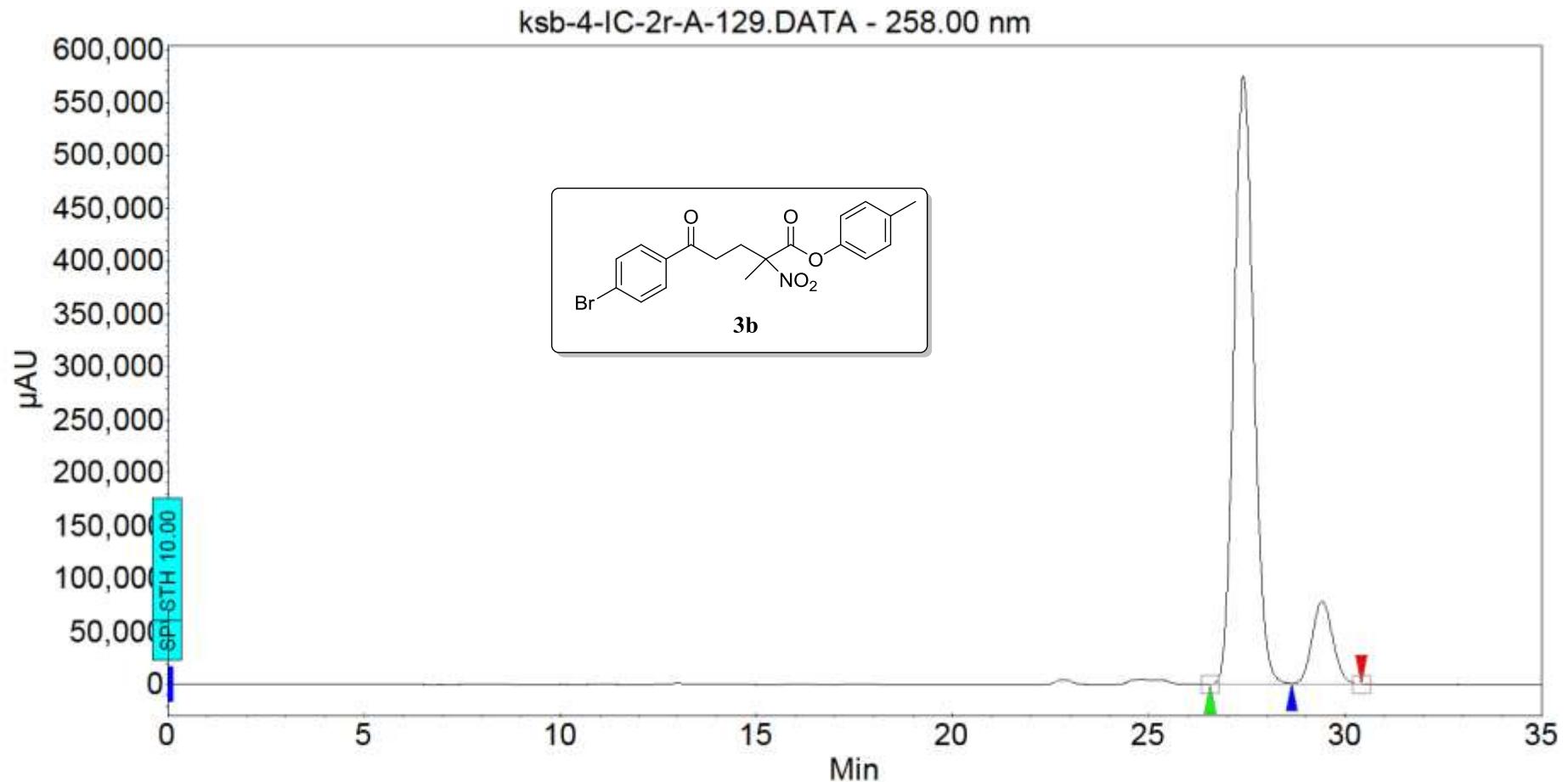
Fig S34.  $^{13}\text{C}$  NMR Spectrum of 3b



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	27.008	50.03	448215.0	229600.4	50.035
2	UNKNOWN	29.133	49.97	421767.7	229282.3	49.965
Total			100.00	869982.7	458882.7	100.000

**Fig S35. HPLC Profile of Racemic 3b**

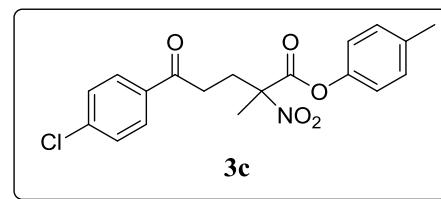


### Peak results :

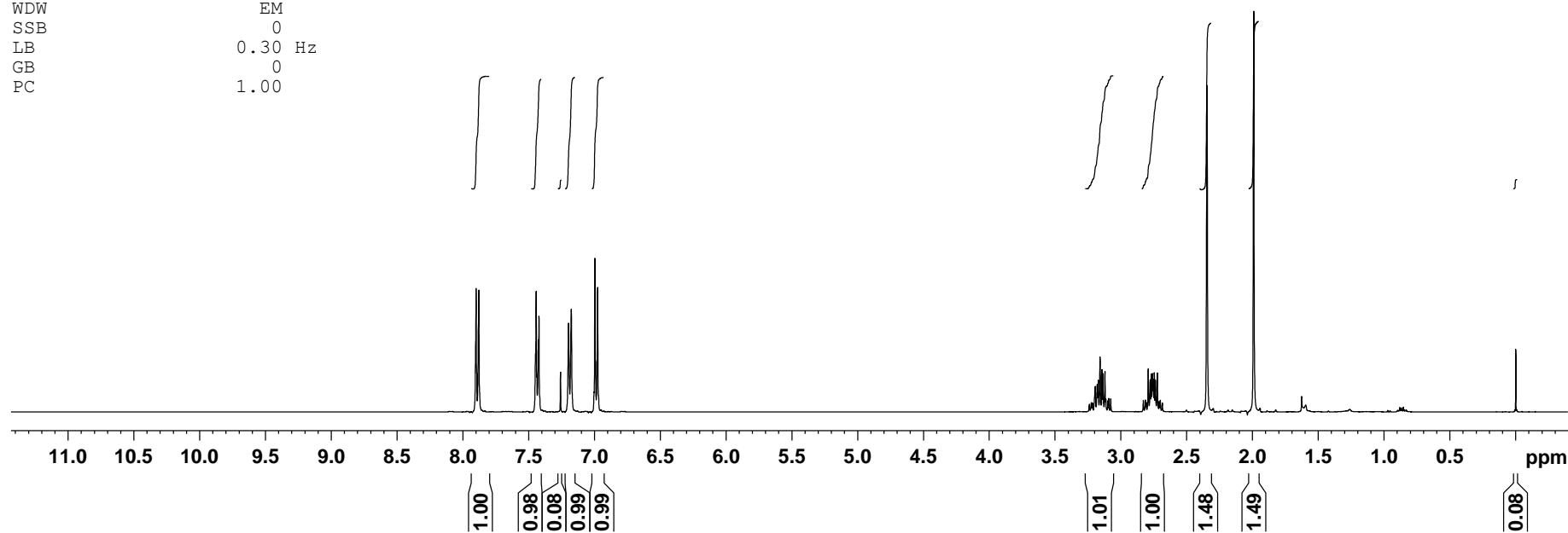
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	27.411	87.40	575054.1	334828.4	87.399
2	UNKNOWN	29.411	12.60	78464.2	48274.4	12.601
Total			100.00	653518.3	383102.8	100.000

**Fig S36. HPLC Profile of Enantioenriched 3b**

NAME INN-4-KSB-105C-1H  
 EXPNO 22  
 PROCNO 1  
 Date 20130820  
 Time 14.45  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 63378  
 SOLVENT CDCl3  
 NS 9  
 DS 0  
 SWH 8802.817 Hz  
 FIDRES 0.138894 Hz  
 AQ 3.5999205 sec  
 RG 57  
 DW 56.800 usec  
 DE 6.50 usec  
 TE 293.6 K  
 D1 1.00000000 sec  
 TDO 1



===== CHANNEL f1 ======  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324008 MHz  
 SI 32768  
 SF 400.1300107 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S37.  $^1\text{H}$  NMR Spectrum of 3c**

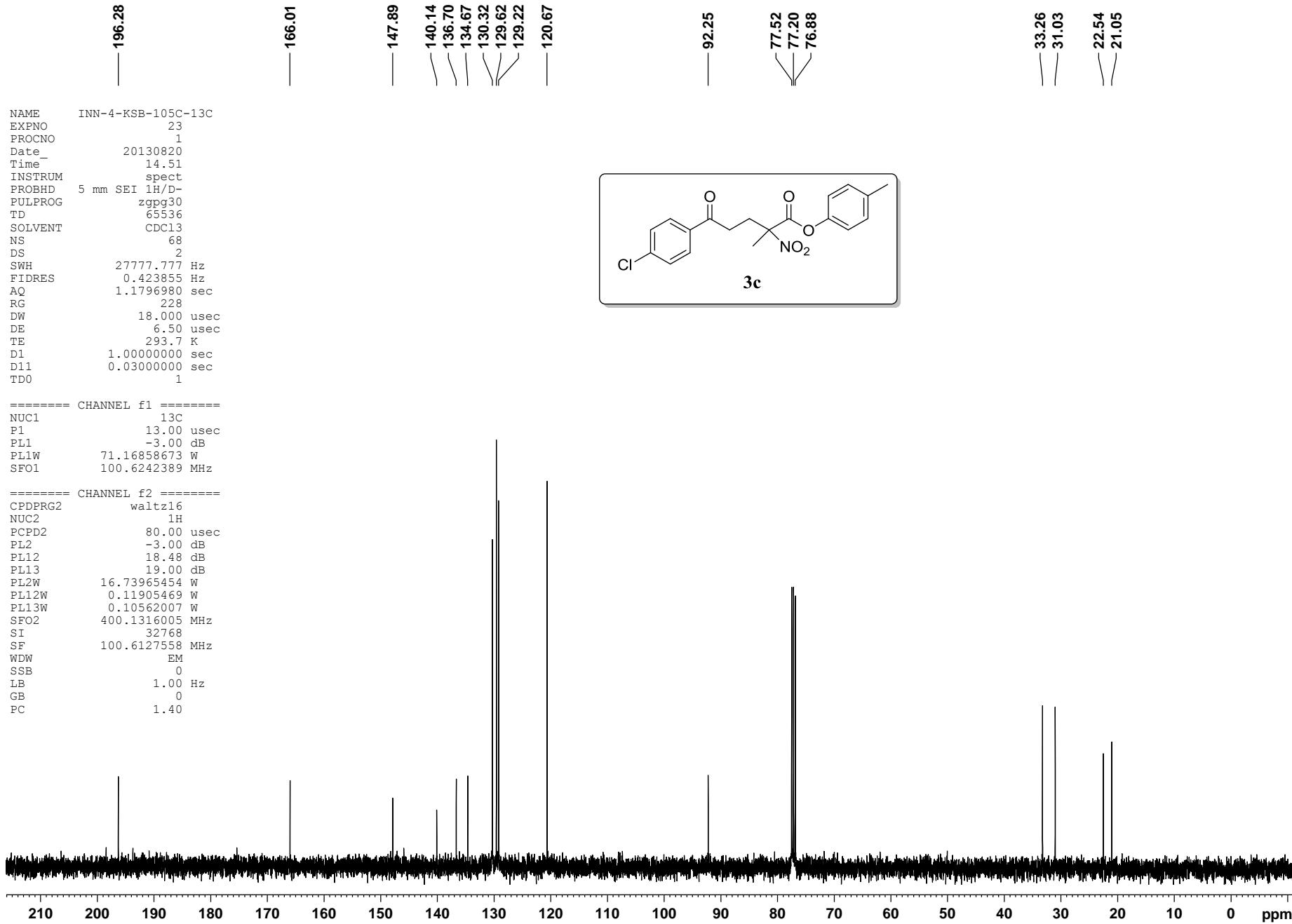
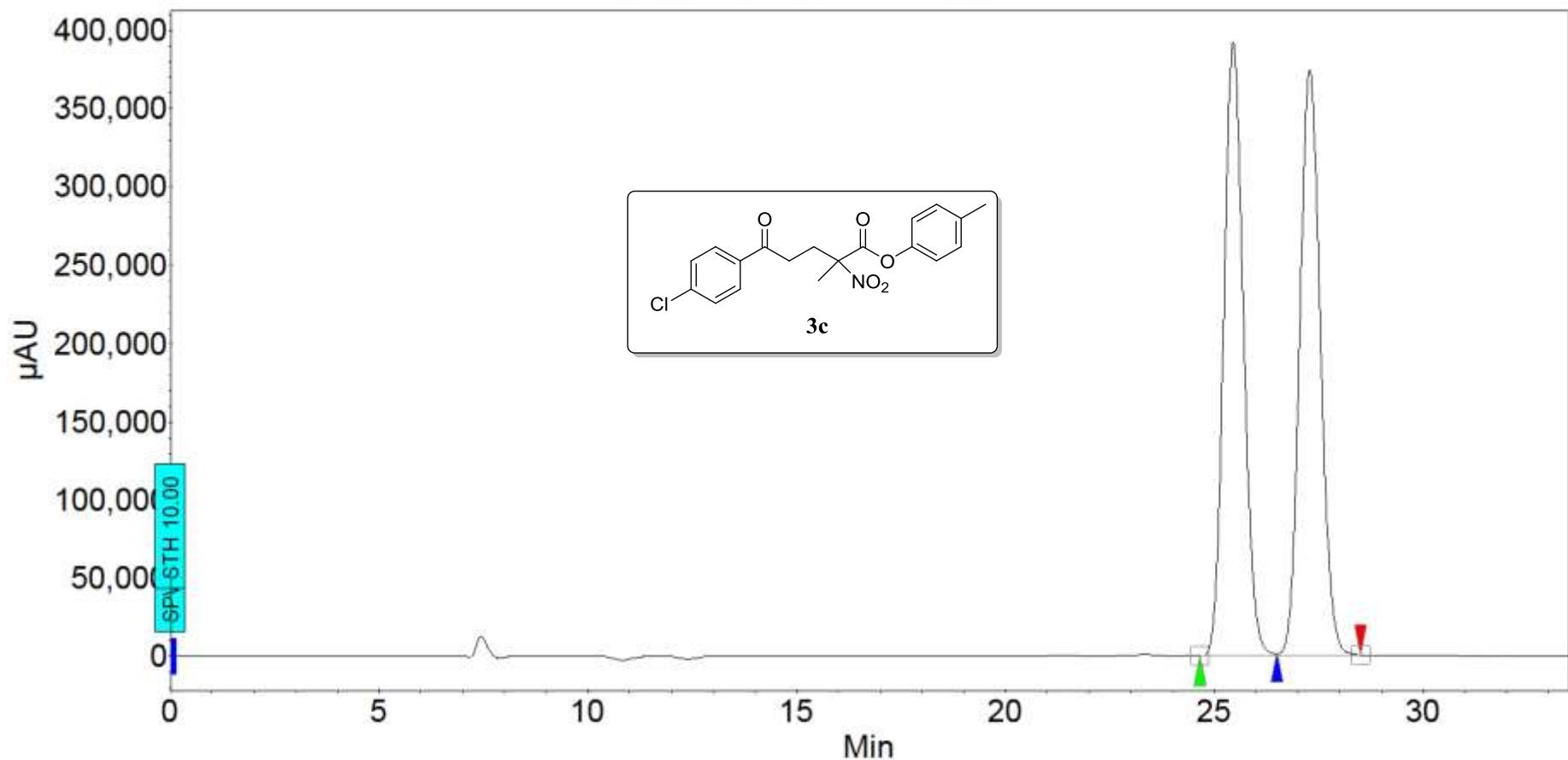


Fig S38.  $^{13}\text{C}$  NMR Spectrum of 3c

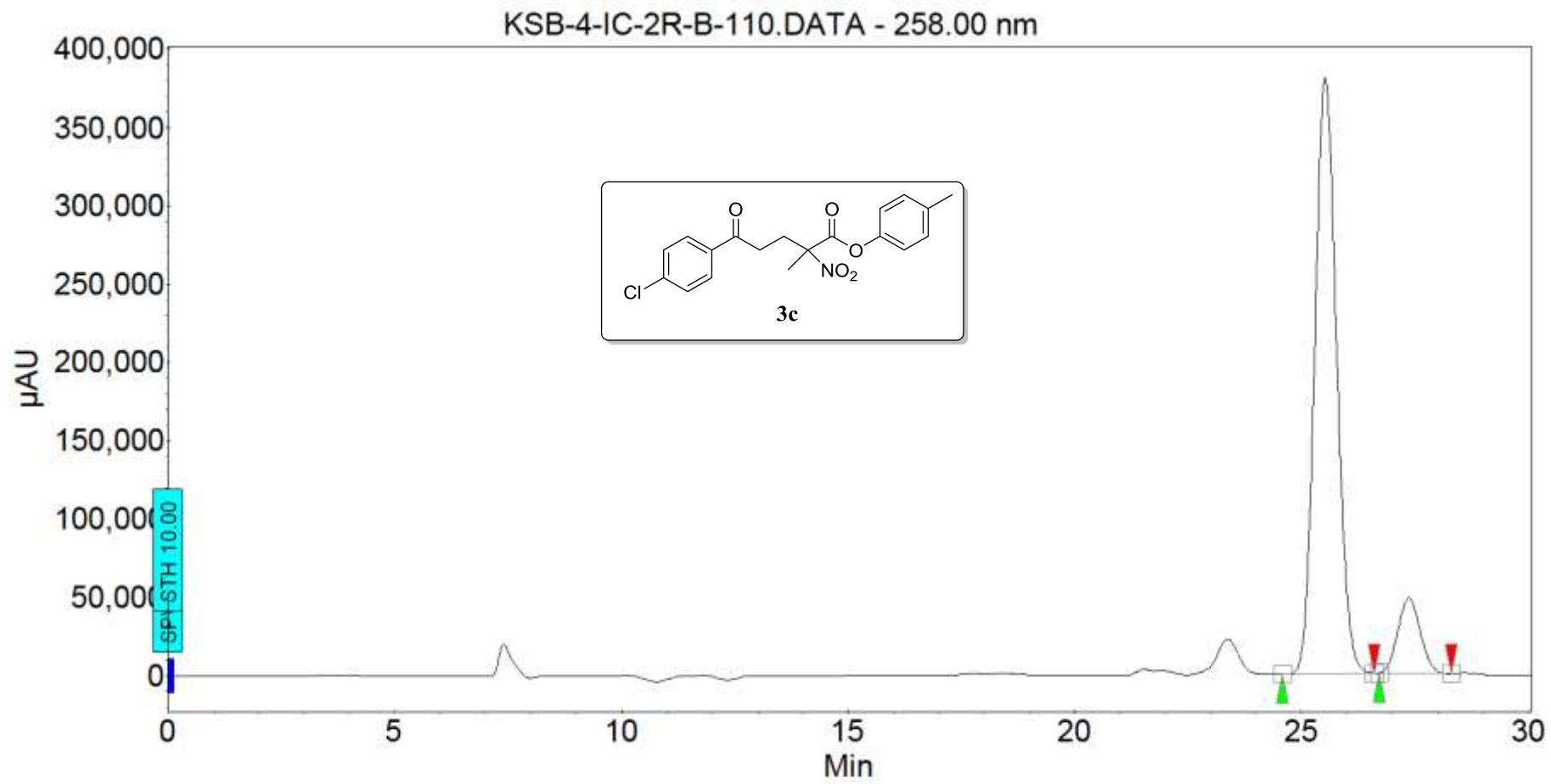
KSB-4-ic-1R-C-105.DATA - 258.00 nm



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	25.451	49.93	391986.7	212932.0	49.926
2	UNKNOWN	27.278	50.07	374331.7	213562.8	50.074
Total			100.00	766318.4	426494.8	100.000

Fig S39. HPLC Profile of Racemic 3c



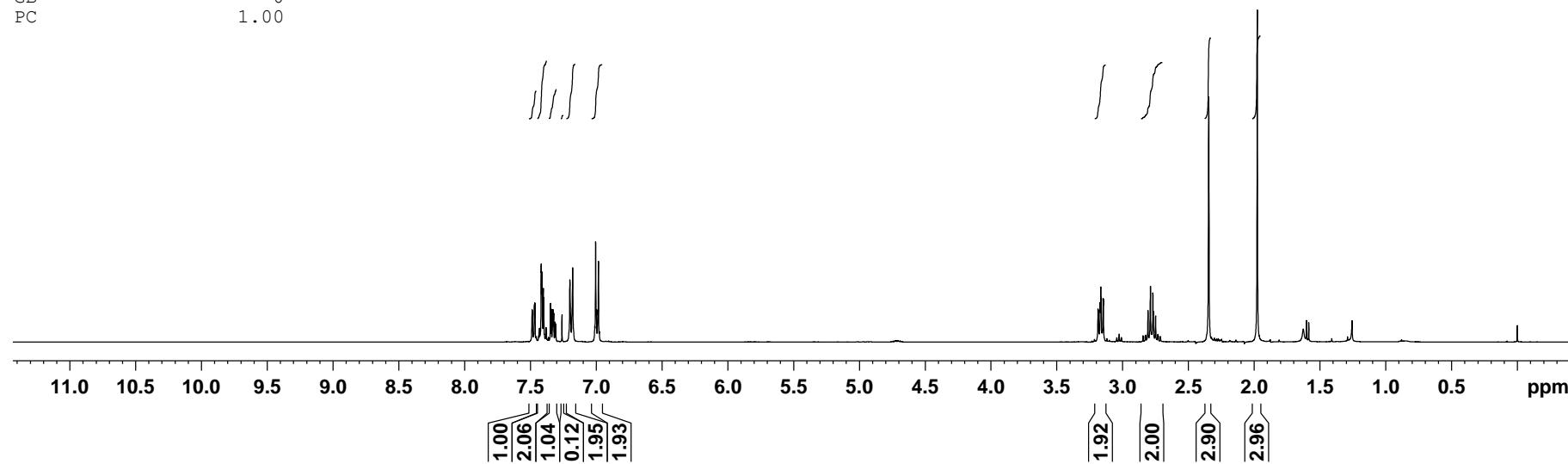
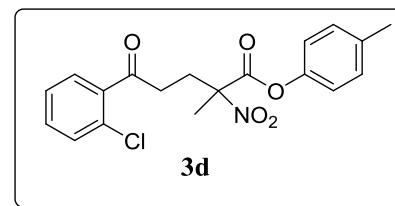
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	25.518	88.54	380971.0	212549.6	88.539
2	UNKNOWN	27.358	11.46	48048.0	27513.4	11.461
Total			100.00	429019.0	240063.1	100.000

Fig S40. HPLC Profile of Enantioenriched 3c

NAME INN-4-KSB-106A-1H  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20130903  
 Time 13.10  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 49340  
 SOLVENT CDC13  
 NS 9  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.166674 Hz  
 AQ 2.9999920 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.3 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 ======  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300103 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S41.**  $^1\text{H}$  NMR Spectrum of 3d

— 200.54

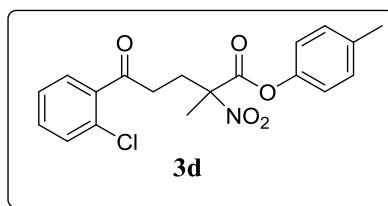
— 165.97

— 147.92  
138.71  
136.66  
132.31  
131.07  
130.82  
130.31  
129.20  
127.26  
120.69

— 92.18

— 77.52  
77.20  
76.89

— 37.56  
— 30.95  
— 22.32  
— 21.04



NAME INN-4-KSB-106A-13C  
EXPNO 2  
PROCNO 1  
Date 20130903  
Time 13.13  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 61  
DS 2  
SWH 27777.777 Hz  
FIDRES 0.423855 Hz  
AQ 1.1796980 sec  
RG 128  
DW 18.000 usec  
DE 6.50 usec  
TE 295.6 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 ======  
NUC1 13C  
P1 13.00 usec  
PL1 -3.00 dB  
PL1W 71.16858673 W  
SFO1 100.6244401 MHz

===== CHANNEL f2 ======  
CPDPKG2 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 100.6127558 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

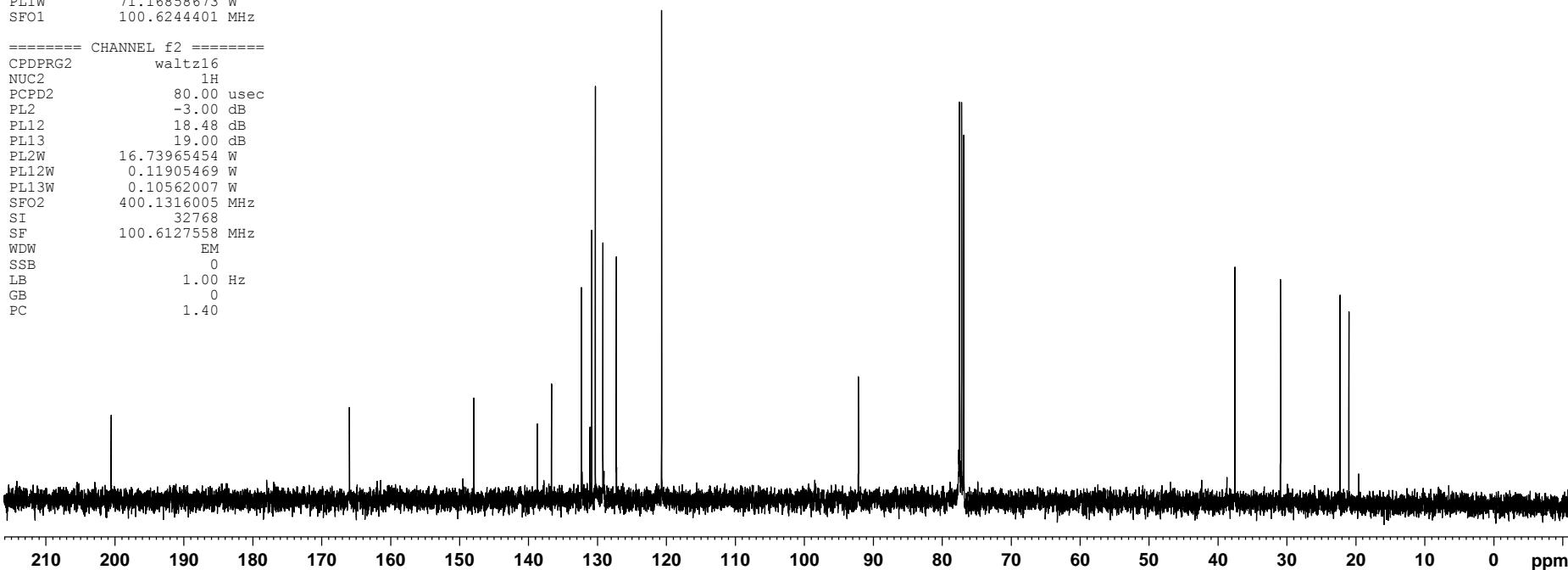
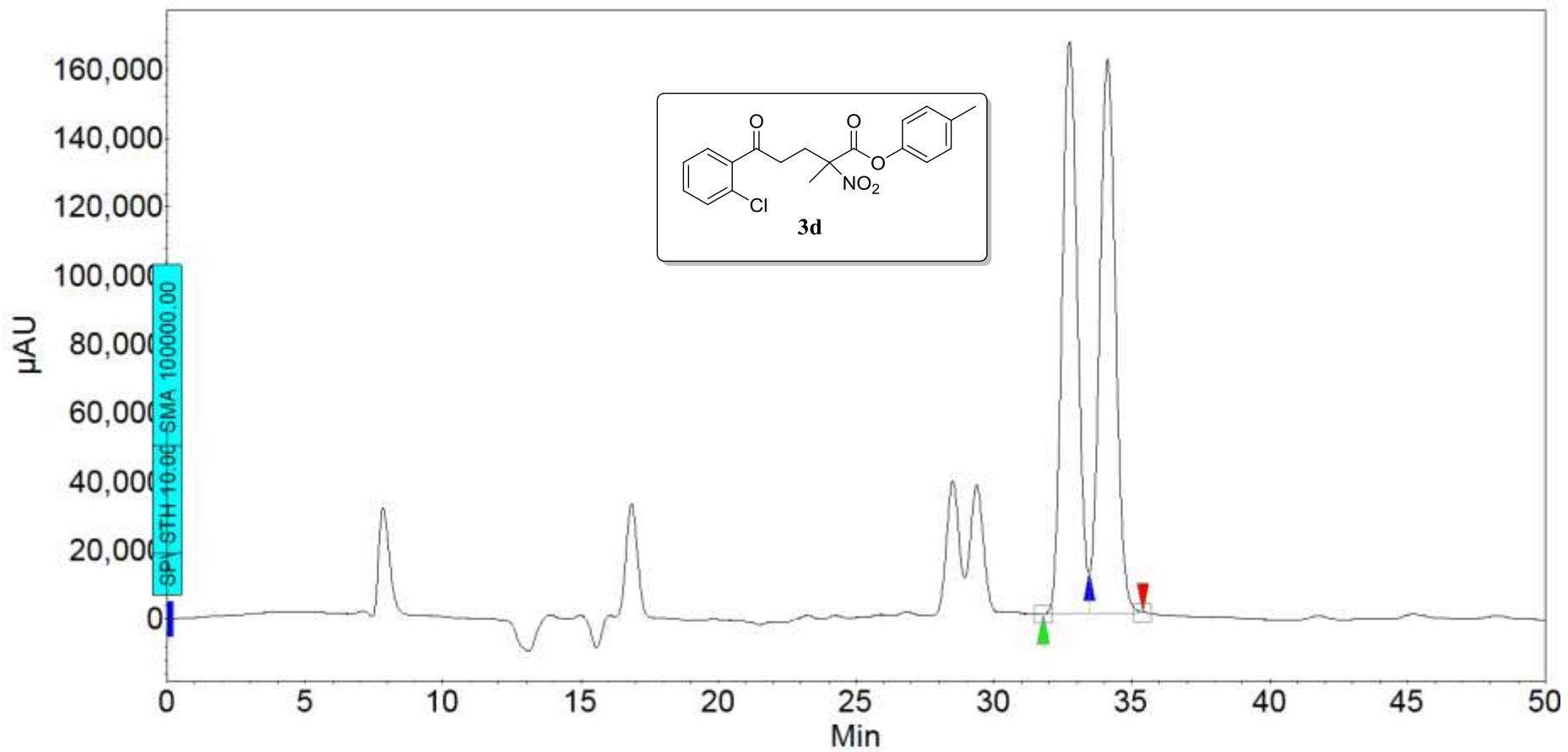


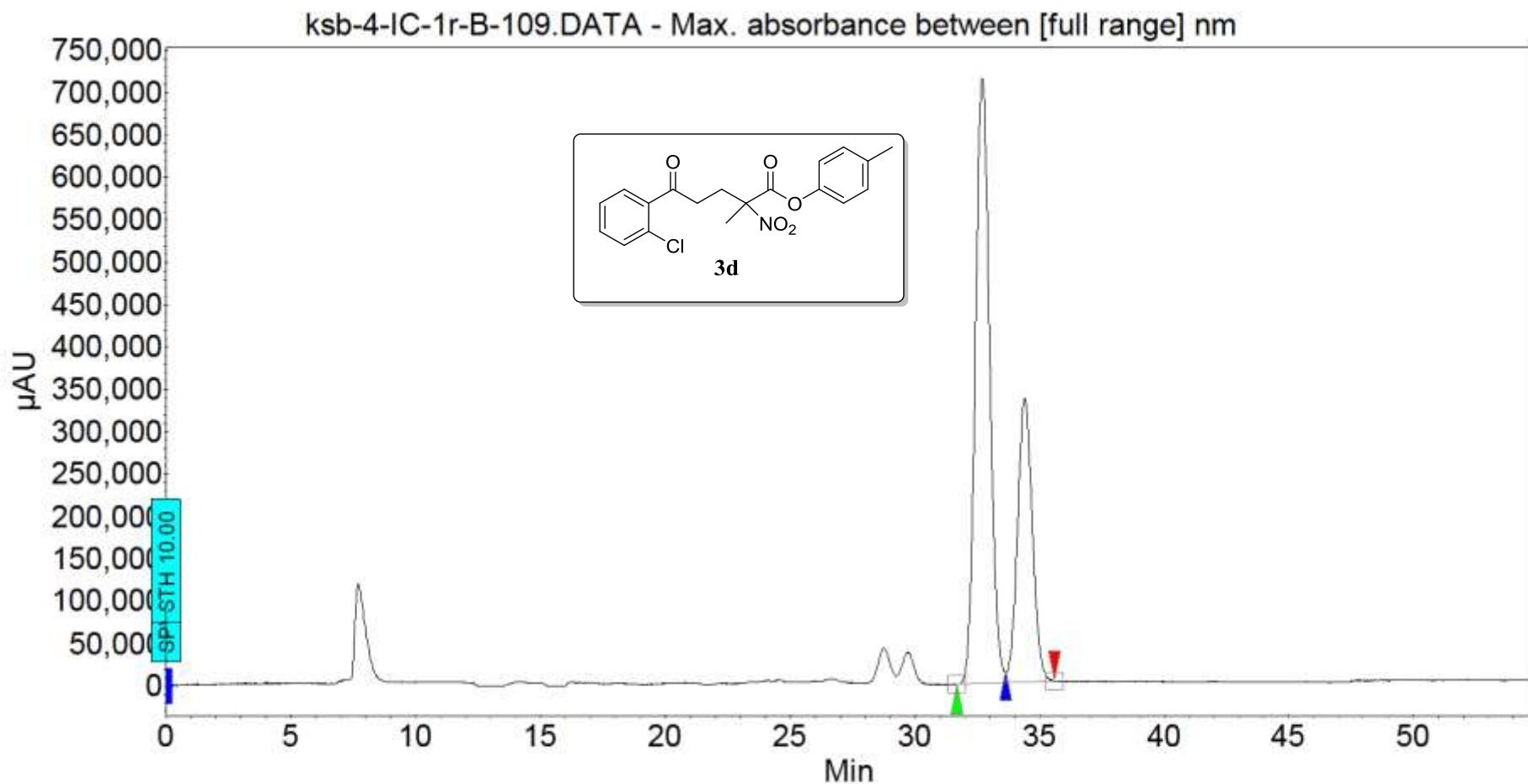
Fig S42. <sup>13</sup>C NMR Spectrum of **3d**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area [%]
1	UNKNOWN	32.730	49.77	166799.5	106717.3	49.770
2	UNKNOWN	34.102	50.23	161440.8	107705.6	50.230
Total			100.00	328240.3	214422.9	100.000

**Fig S43. HPLC Profile of Racemic 3d**

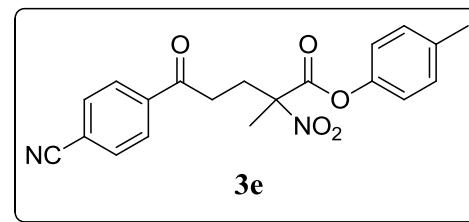


### Peak results :

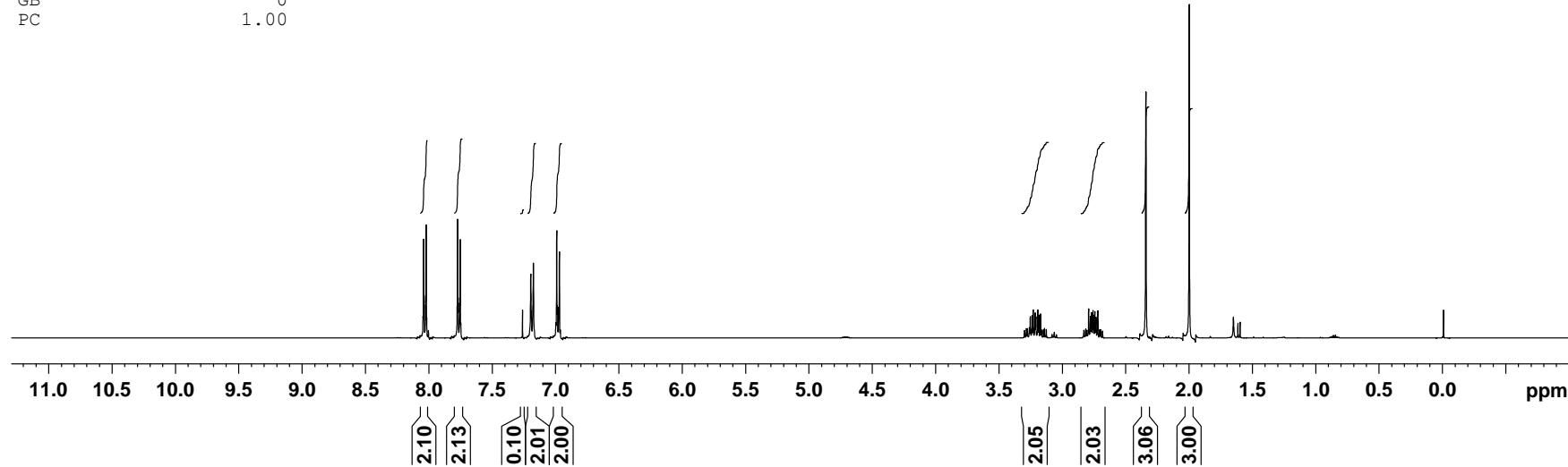
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	32.691	67.34	714485.4	474594.9	67.340
2	UNKNOWN	34.398	32.66	334521.4	230176.3	32.660
Total			100.00	1049006.8	704771.1	100.000

**Fig S44. HPLC Profile of Enantioenriched 3d**

NAME INN-4-KSB-105-B-1H  
 EXPNO 4  
 PROCNO 1  
 Date 20130831  
 Time 13.14  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 11  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 50.8  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.4 K  
 D1 1.0000000 sec  
 TDO 1



===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300104 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S45.**  $^1\text{H}$  NMR Spectrum of **3e**

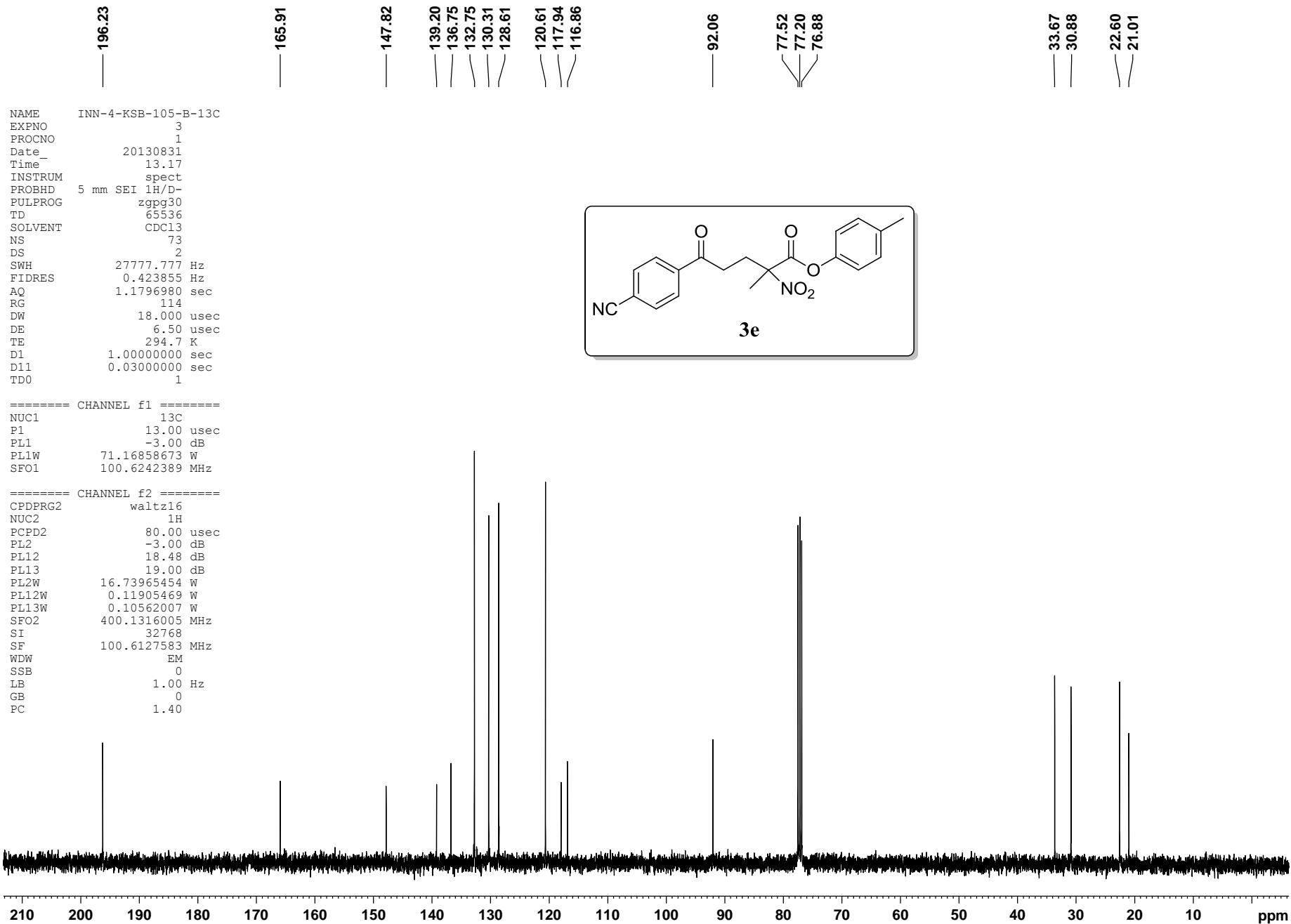
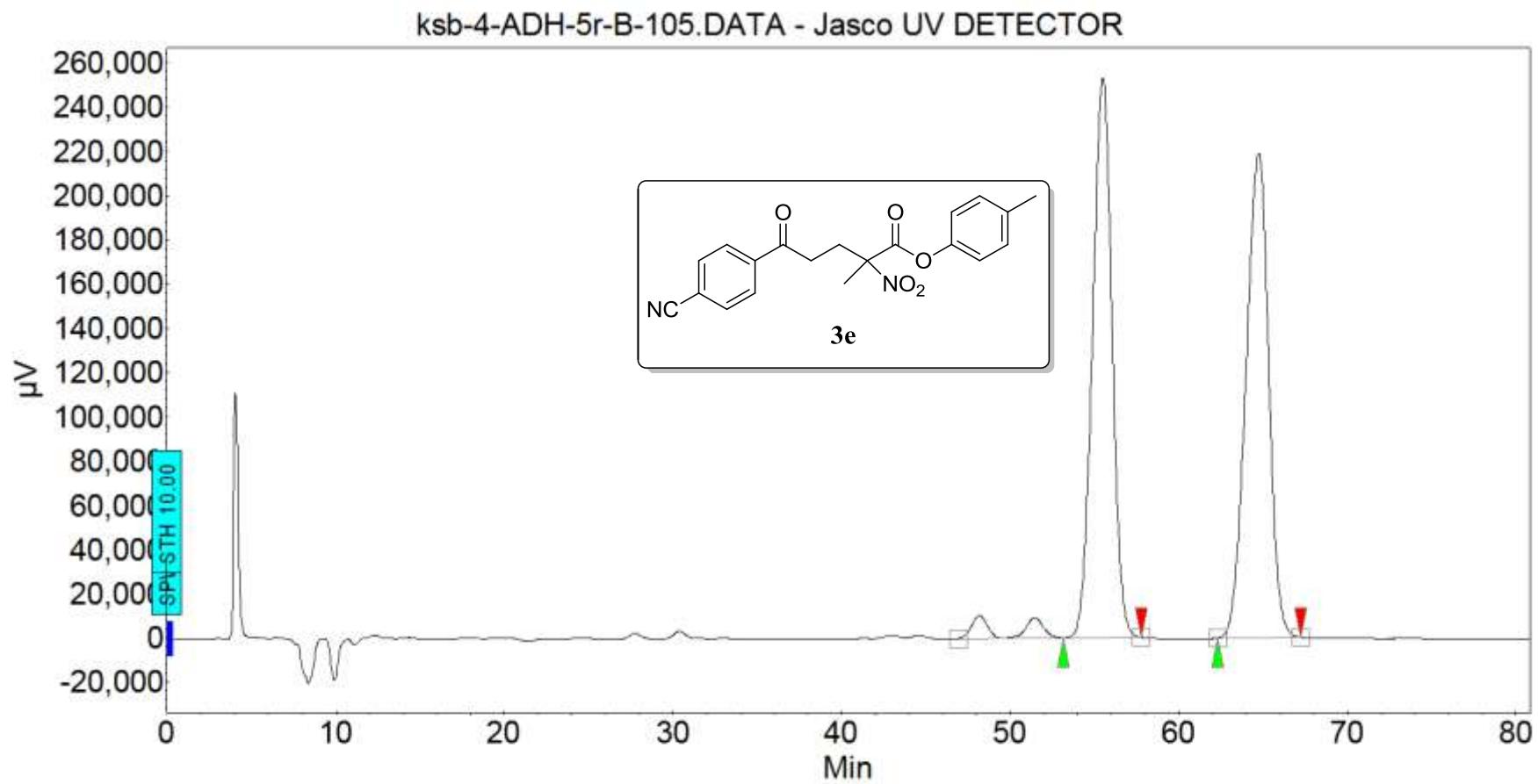


Fig S46.  $^{13}\text{C}$  NMR Spectrum of 3e

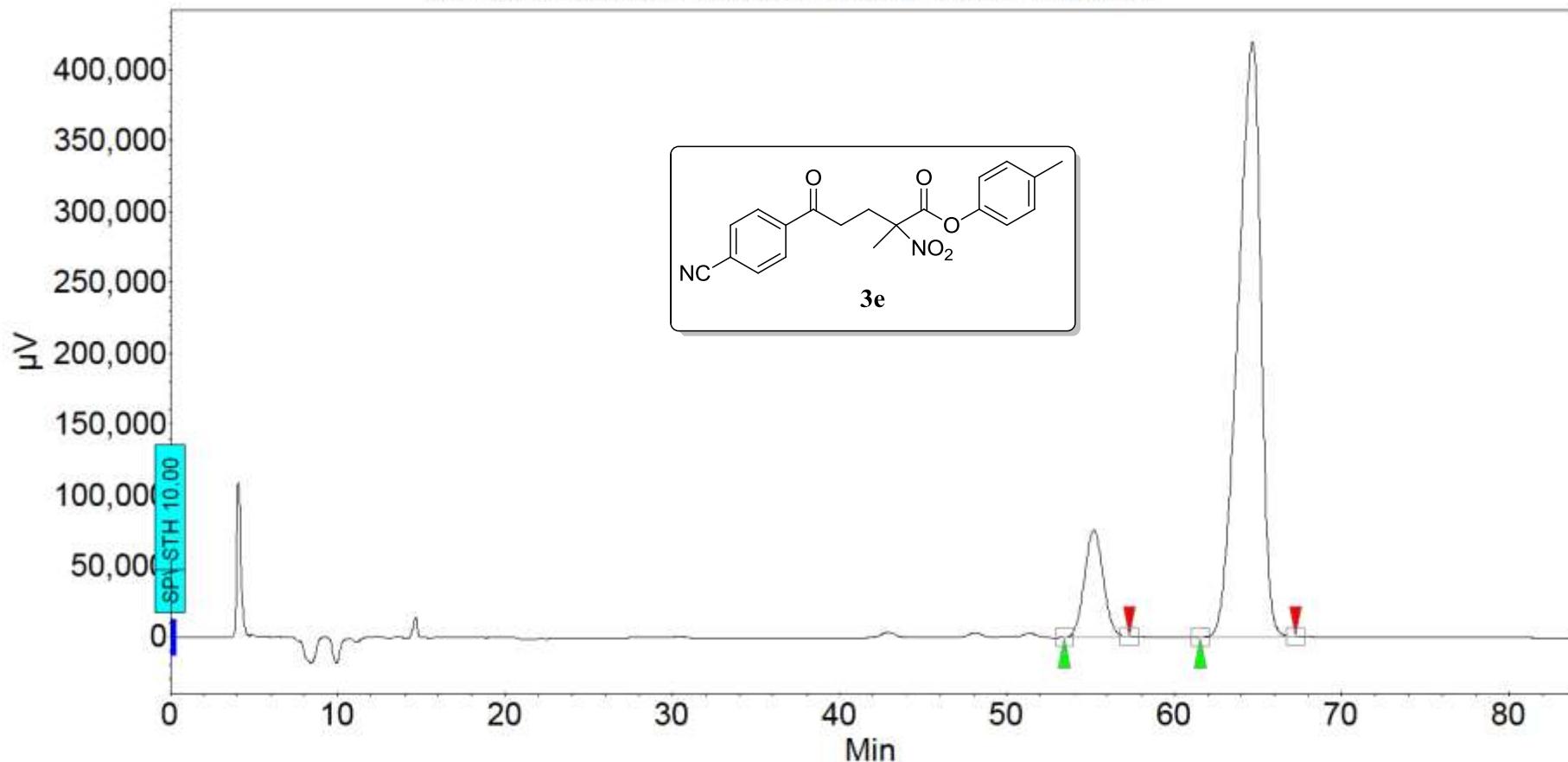


### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{V}$ ]	Area [ $\mu\text{V}.\text{Min}$ ]	Area [%]
1	UNKNOWN	55.492	49.94	252847.1	339144.8	49.941
2	UNKNOWN	64.733	50.06	218833.9	339941.2	50.059
Total			100.00	471681.0	679086.0	100.000

Fig S47. HPLC Profile of Racemic 3e

ksb-4-ADH-2r-B-127.DATA - Jasco UV DETECTOR



**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu$ V]	Area [ $\mu$ V.Min]	Area % [%]
1	UNKNOWN	55.183	12.86	75243.9	98187.1	12.856
2	UNKNOWN	64.667	87.14	419602.2	665561.5	87.144
Total			100.00	494846.1	763748.6	100.000

**Fig S48. HPLC Profile of Enantioenriched **3e****

Current Data Parameters  
NAME INN-4-KSB-105A-1H  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131108  
Time\_ 18.39  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 53.37  
DW 50.000 usec  
DE 6.50 usec  
TE 297.2 K  
D1 1.0000000 sec  
TDO 1

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

F2 - Processing parameters  
SI 65536  
SF 500.1300121 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

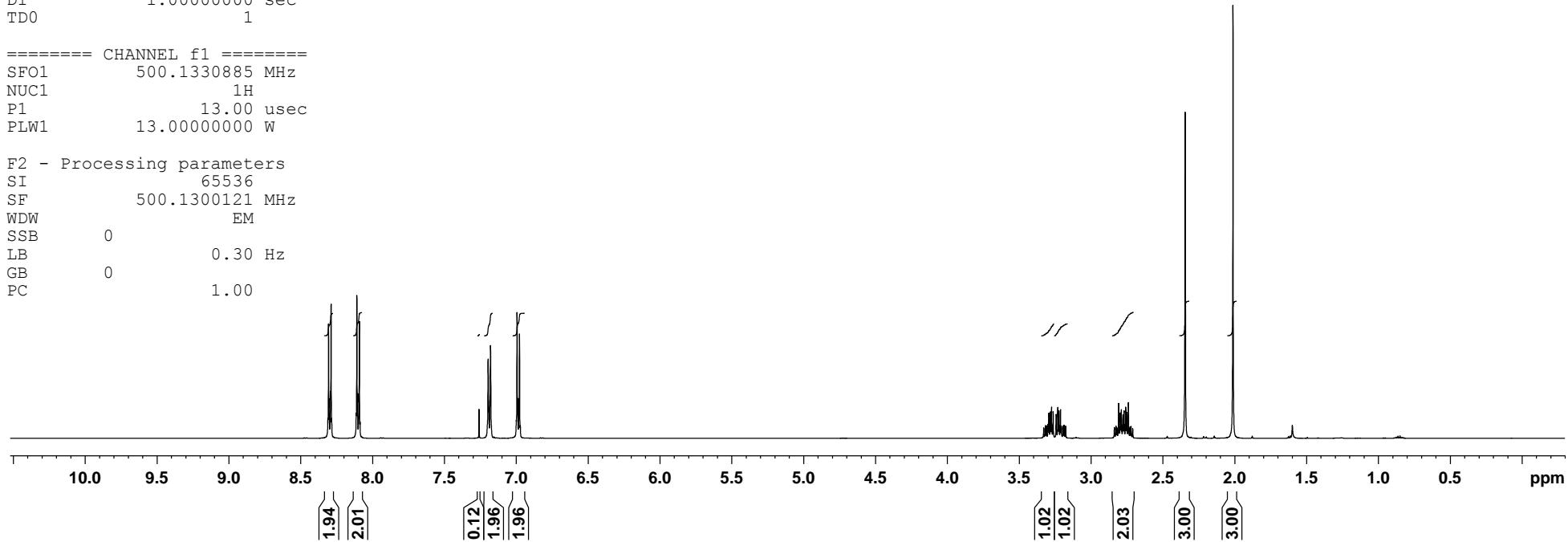
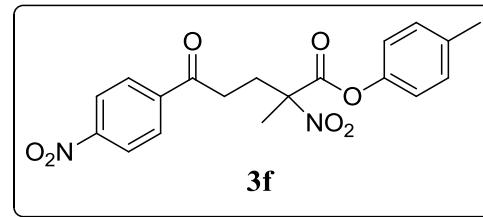


Fig S49.  $^1\text{H}$  NMR Spectrum of **3f**

— 196.05

— 165.92

— 150.70  
— 147.89  
— 140.74  
— 136.79  
— 130.34  
— 129.27  
— 124.12  
— 120.63

— 92.09

— 77.45  
— 77.20  
— 76.94

— 33.96  
— 30.95

— 22.67  
— 21.02

Current Data Parameters  
NAME INN-4-KSB-105A-13C  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131108  
Time\_ 18.42  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zpg30  
TD 65536  
SOLVENT CDCl3  
NS 95  
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 297.6 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 125.7577709 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

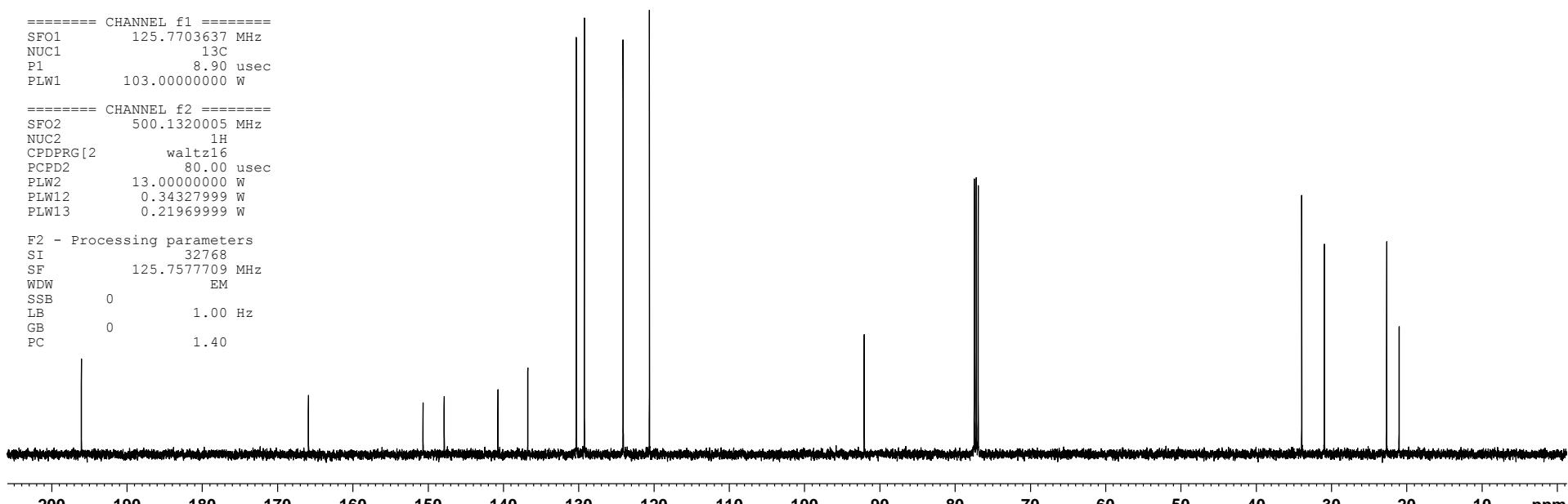
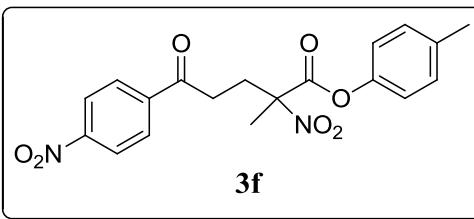
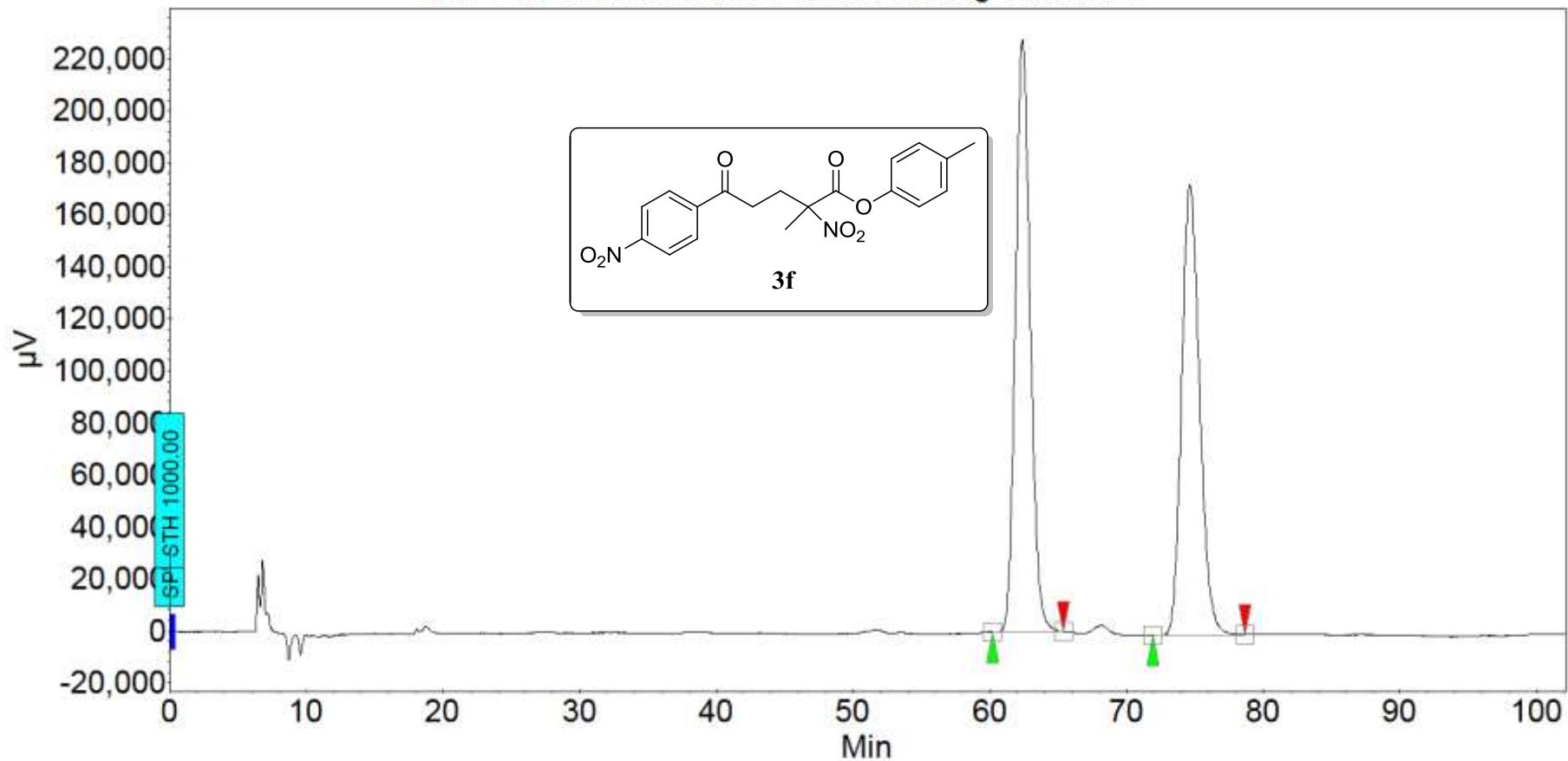


Fig S50. <sup>13</sup>C NMR Spectrum of 3f

ksb-4-IC-3r-A-105.DATA - Jasco Analog Channel 1

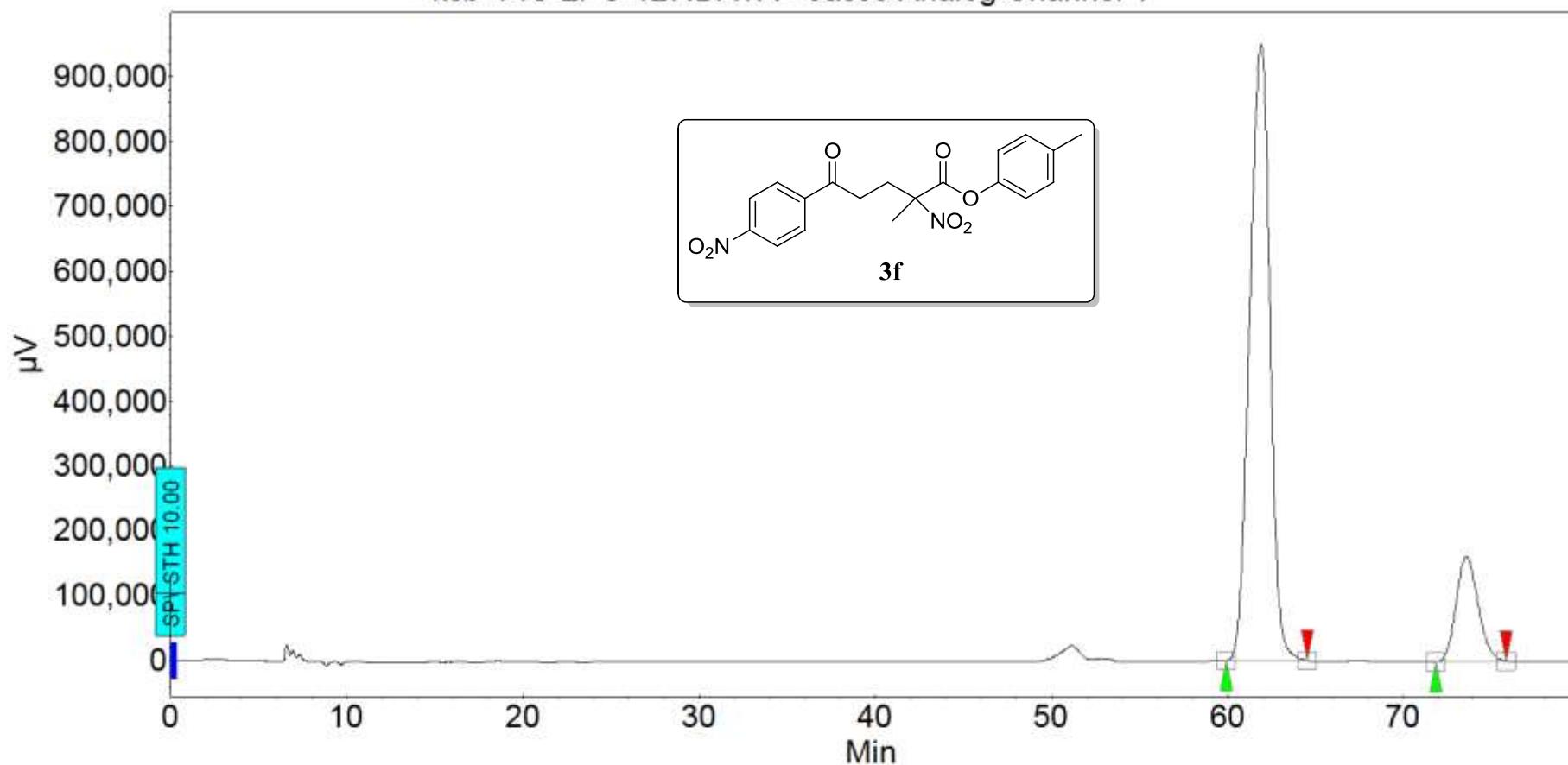


**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{V}$ ]	Area [ $\mu\text{V}.\text{Min}$ ]	Area % [%]
1	UNKNOWN	62.375	52.10	227760.5	286514.3	52.102
2	UNKNOWN	74.625	47.90	173285.7	263398.5	47.898
Total			100.00	401046.2	549912.8	100.000

**Fig S51. HPLC Profile of Racemic 3f**

ksb-4-IC-2r-C-127.DATA - Jasco Analog Channel 1



**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	61.908	84.43	951483.0	1269356.1	84.433
2	UNKNOWN	73.558	15.57	161625.1	234024.8	15.567
Total			100.00	1113108.1	1503380.9	100.000

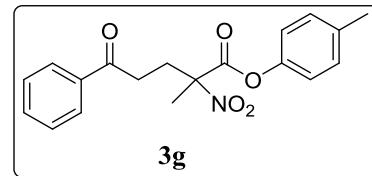
**Fig S52. HPLC Profile of Enantioenriched 3f**

Current Data Parameters  
NAME INN-4-KSB-122A-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131014  
Time 20.05  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TD0 1

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

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



3g

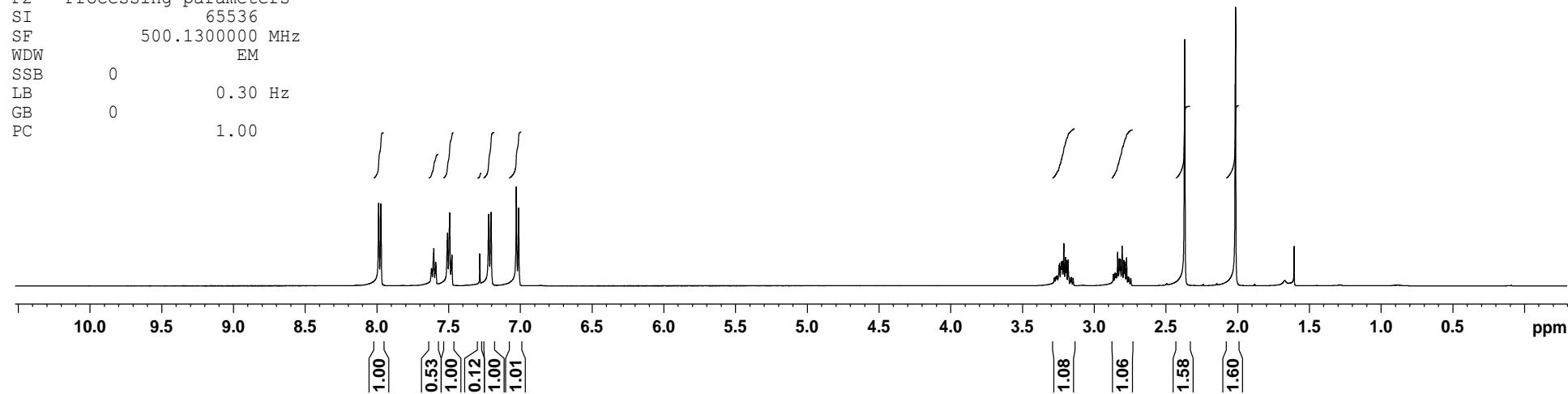


Fig S53. <sup>1</sup>H NMR Spectrum of 3g

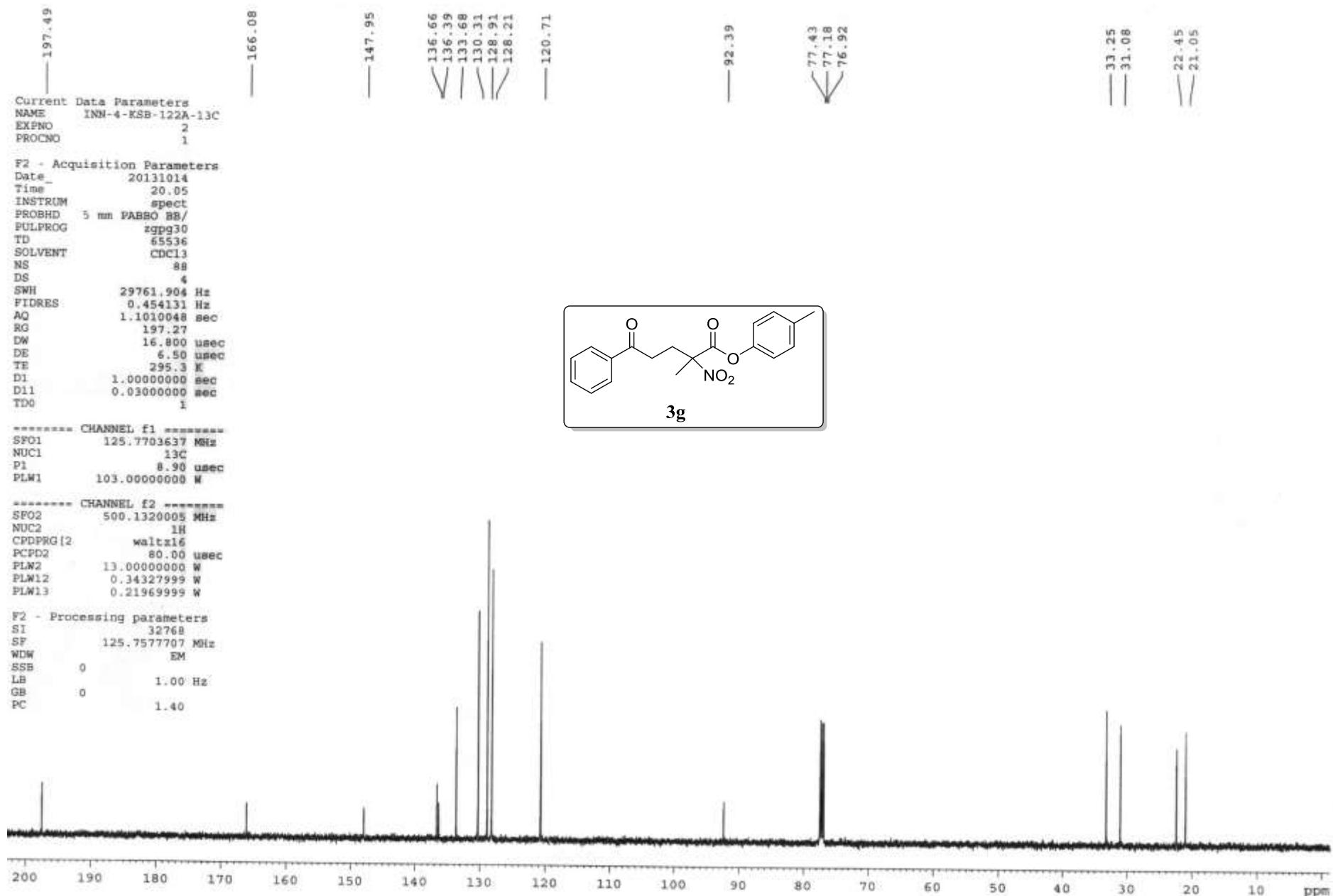
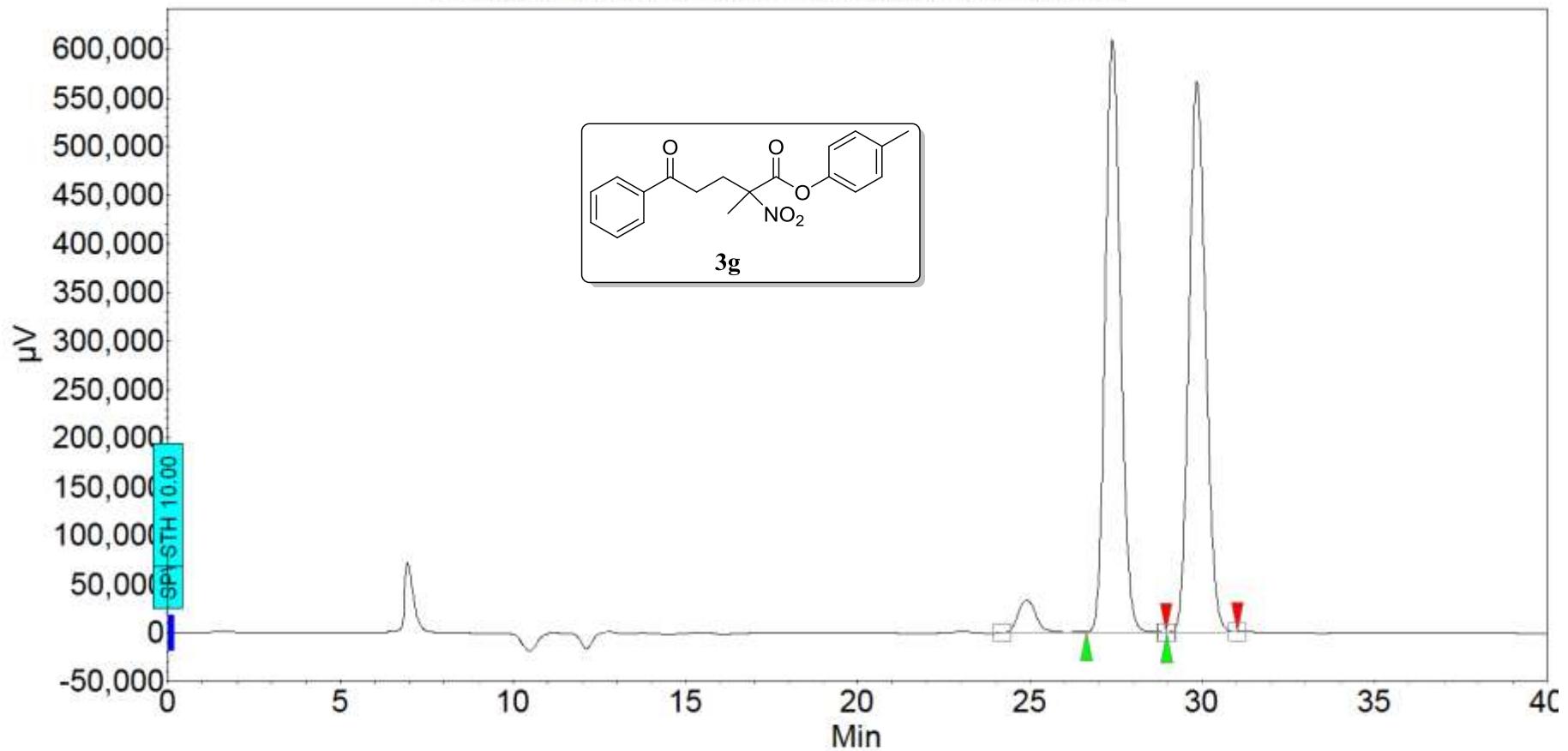


Fig S54.  $^{13}\text{C}$  NMR Spectrum of 3g

ksb-4-IC-1R-A-122.DATA - Jasco UV DETECTOR

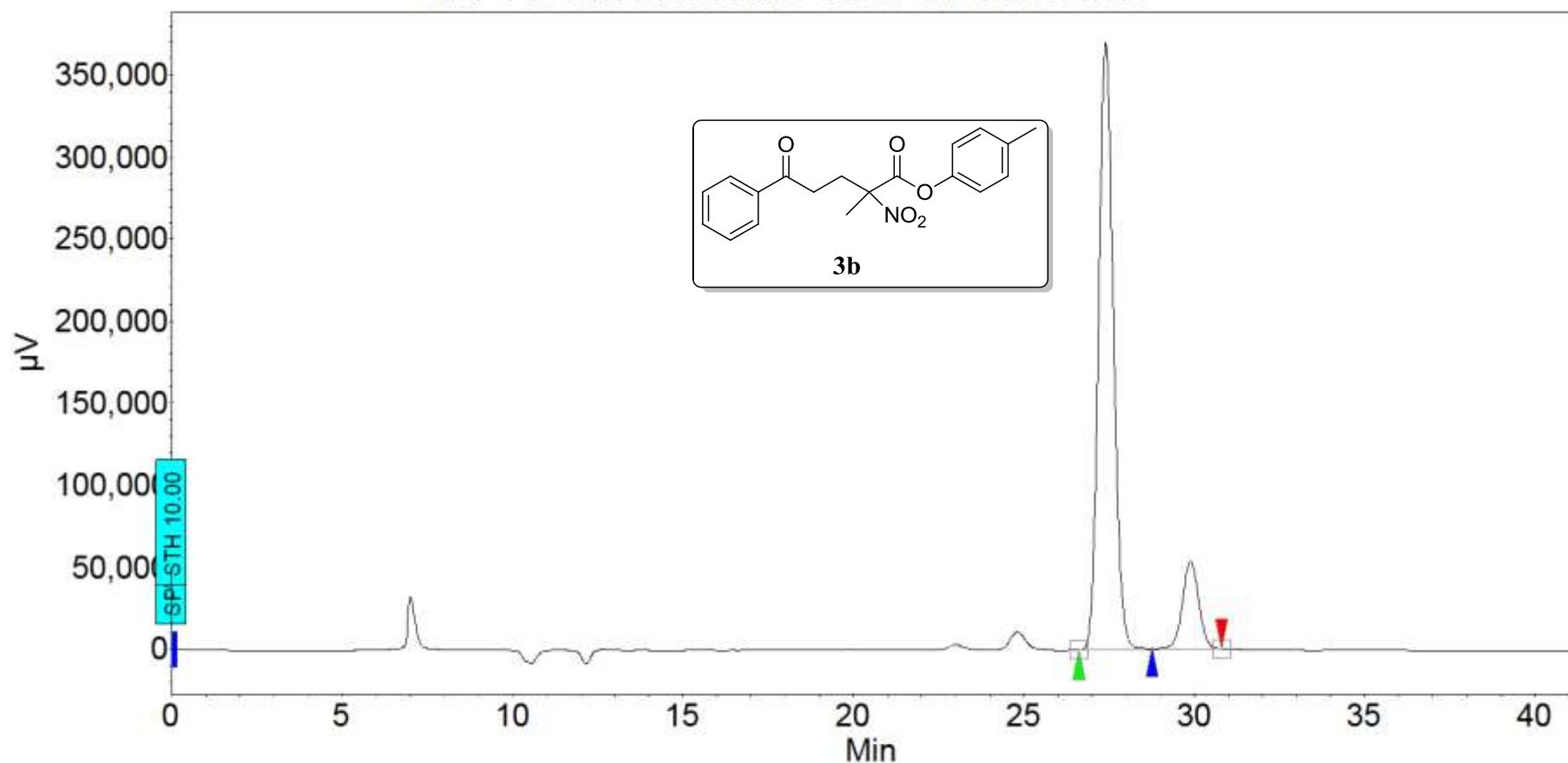


**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	27.383	49.99	609902.8	315794.7	49.986
2	UNKNOWN	29.833	50.01	566037.8	315969.7	50.014
Total			100.00	1175940.6	631764.4	100.000

Fig S55. HPLC Profile of Racemic **3g**

ksb-4-IC-2R-A-126.DATA - Jasco UV DETECTOR



**Peak results :**

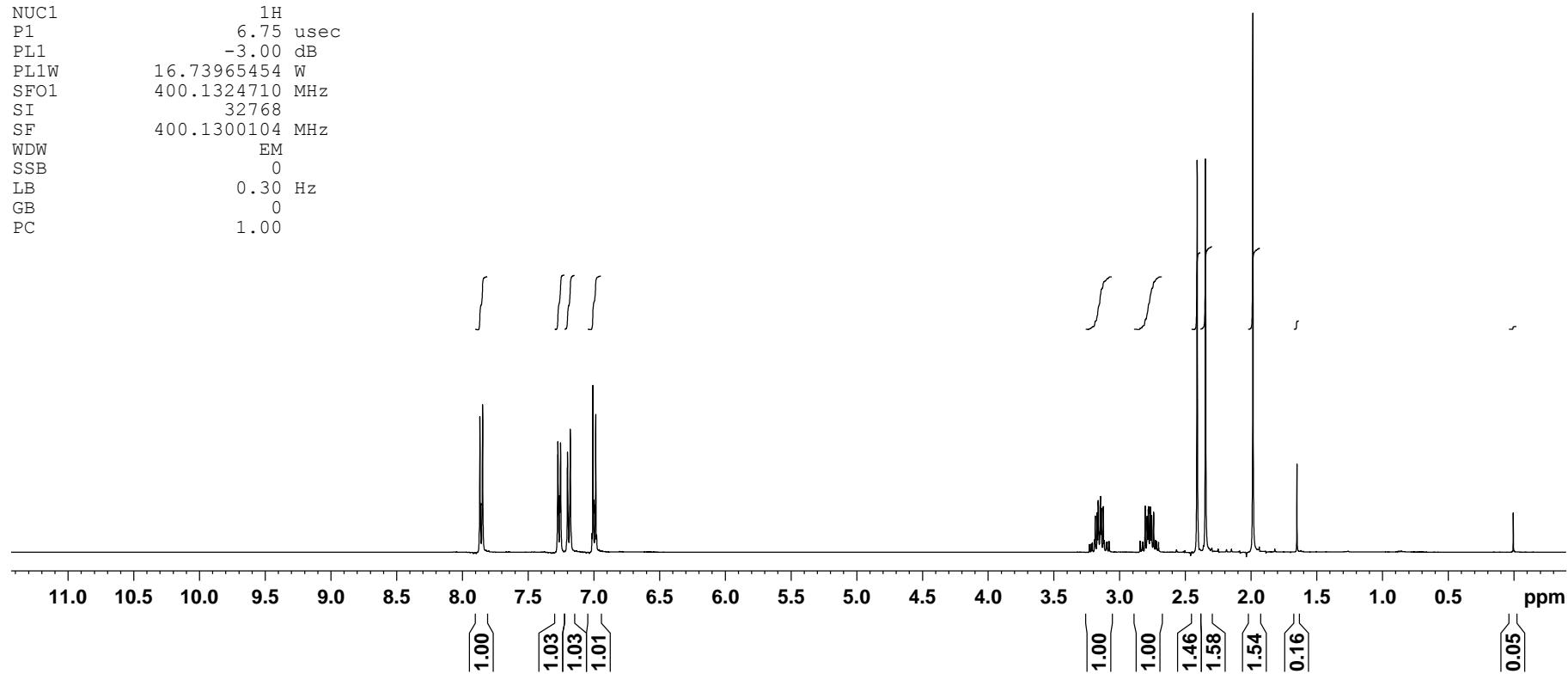
Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV·Min]	Area % [%]
1	UNKNOWN	27.408	86.11	370403.2	187905.3	86.105
2	UNKNOWN	29.892	13.89	53911.4	30322.3	13.895
Total			100.00	424314.7	218227.6	100.000

**Fig S56. HPLC Profile of Enantioenriched 3g**

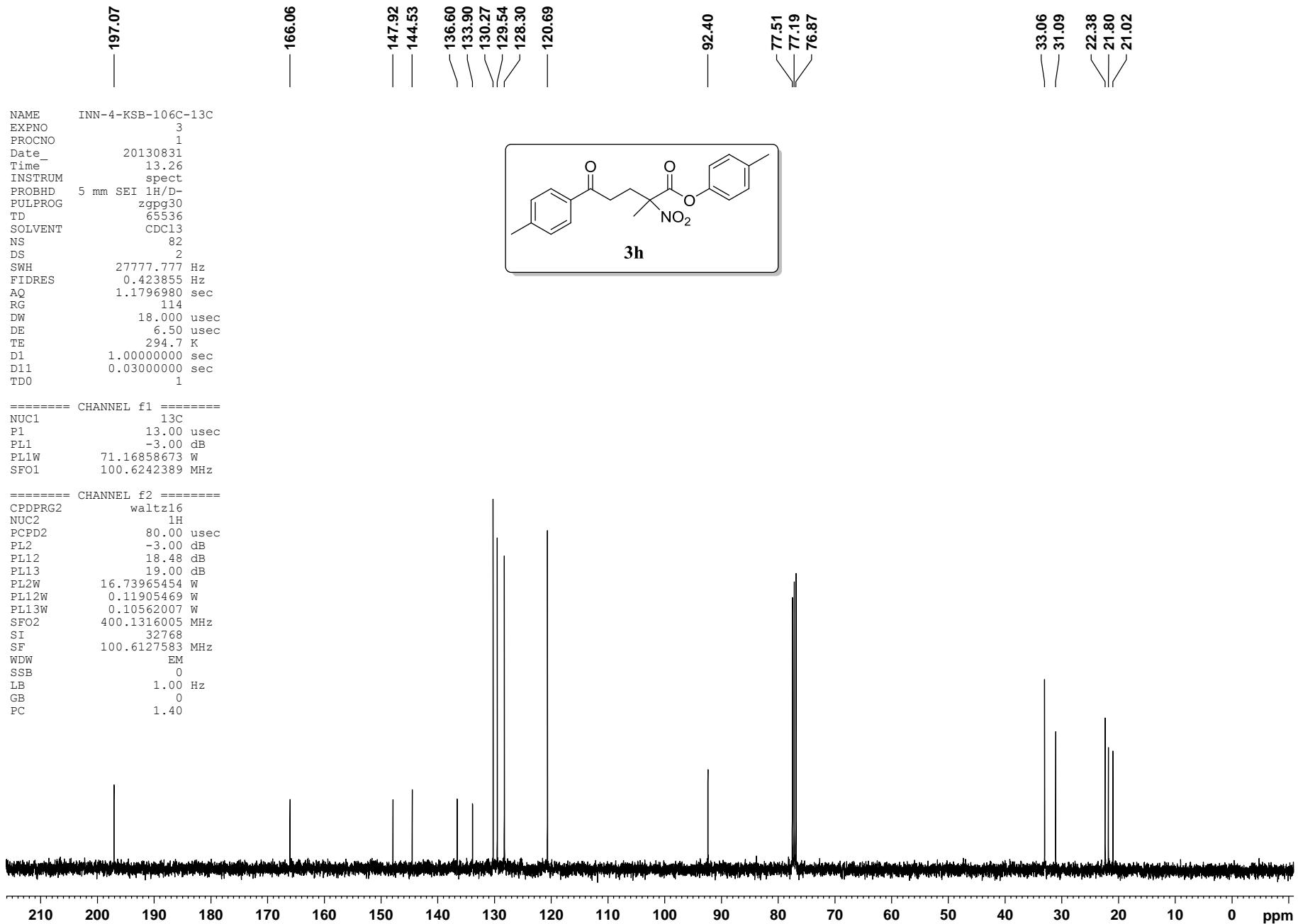
NAME INN-4-KSB-106C-1H  
 EXPNO 4  
 PROCNO 1  
 Date\_ 20130831  
 Time 13.22  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 12  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 50.8  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.4 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====

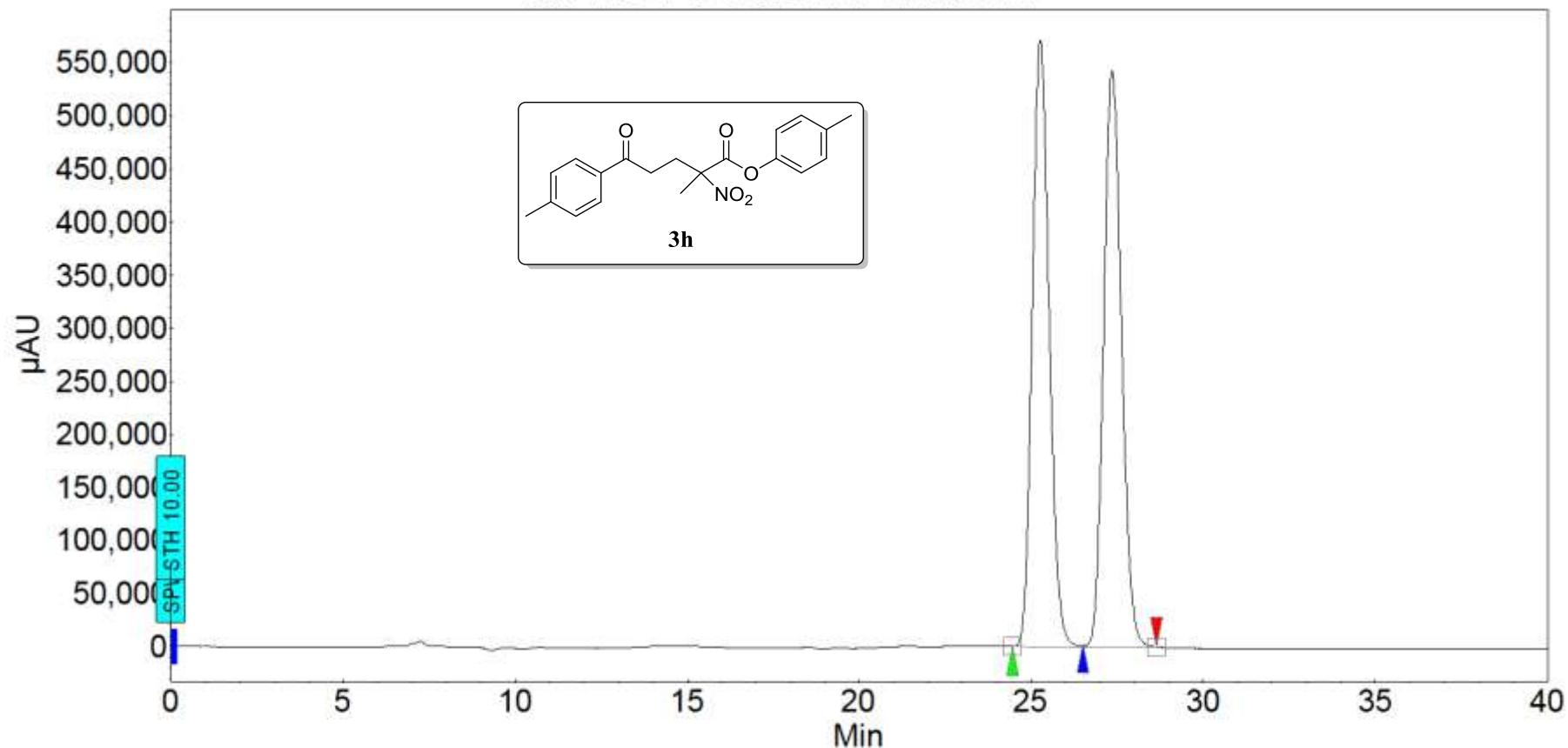
NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300104 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S57. <sup>1</sup>H NMR Spectrum of 3h**



**Fig S58.**  $^{13}\text{C}$  NMR Spectrum of **3h**

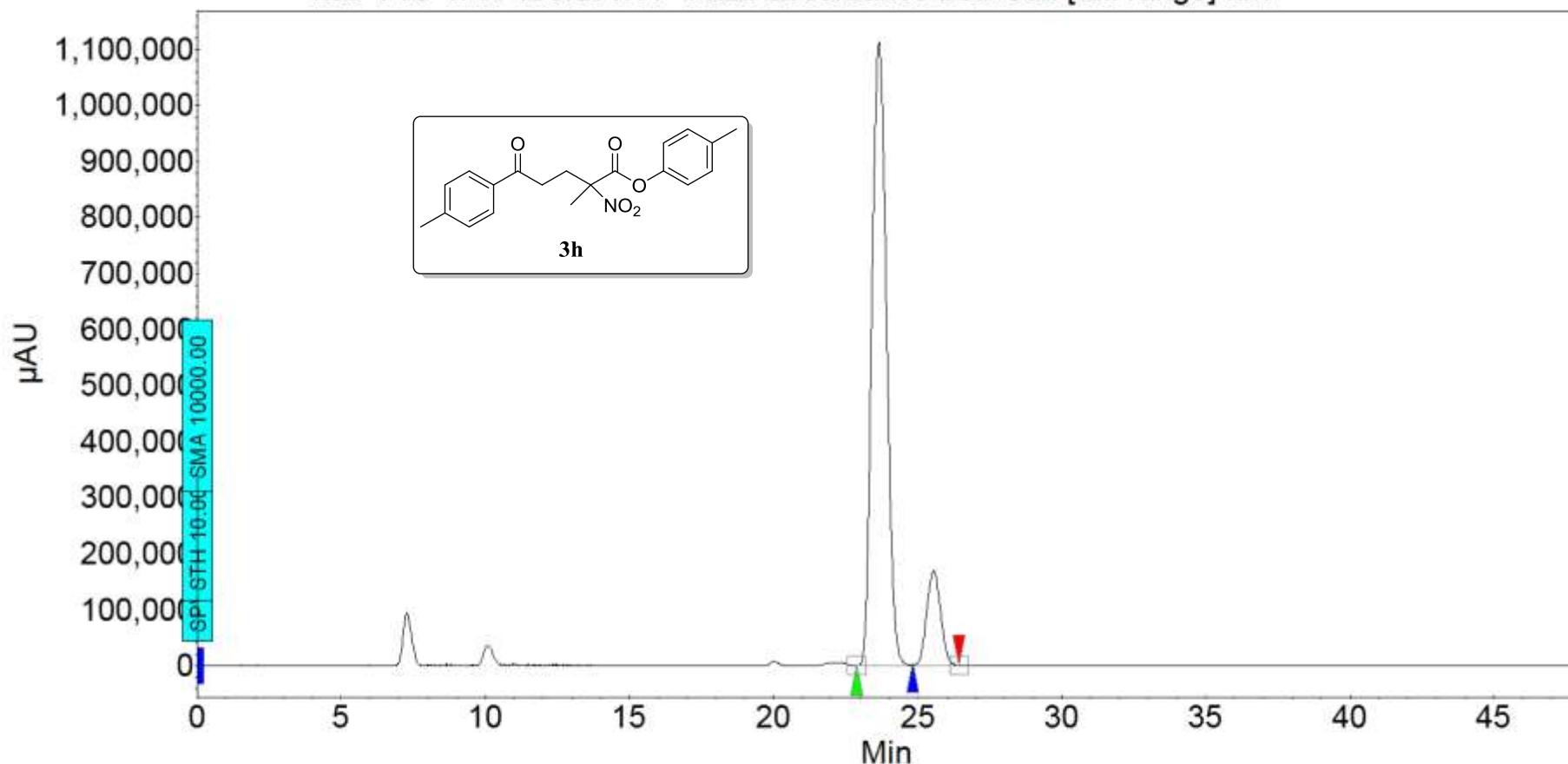


### Peak results :

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

Fig S59. HPLC Profile of Racemic **3h**

ksb-4-IC-1r-A-109.DATA - Max. absorbance between [full range] nm

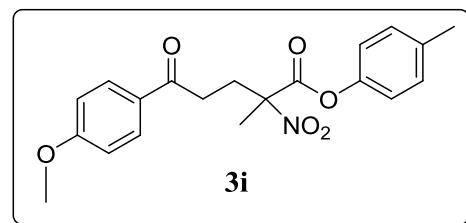


**Peak results :**

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

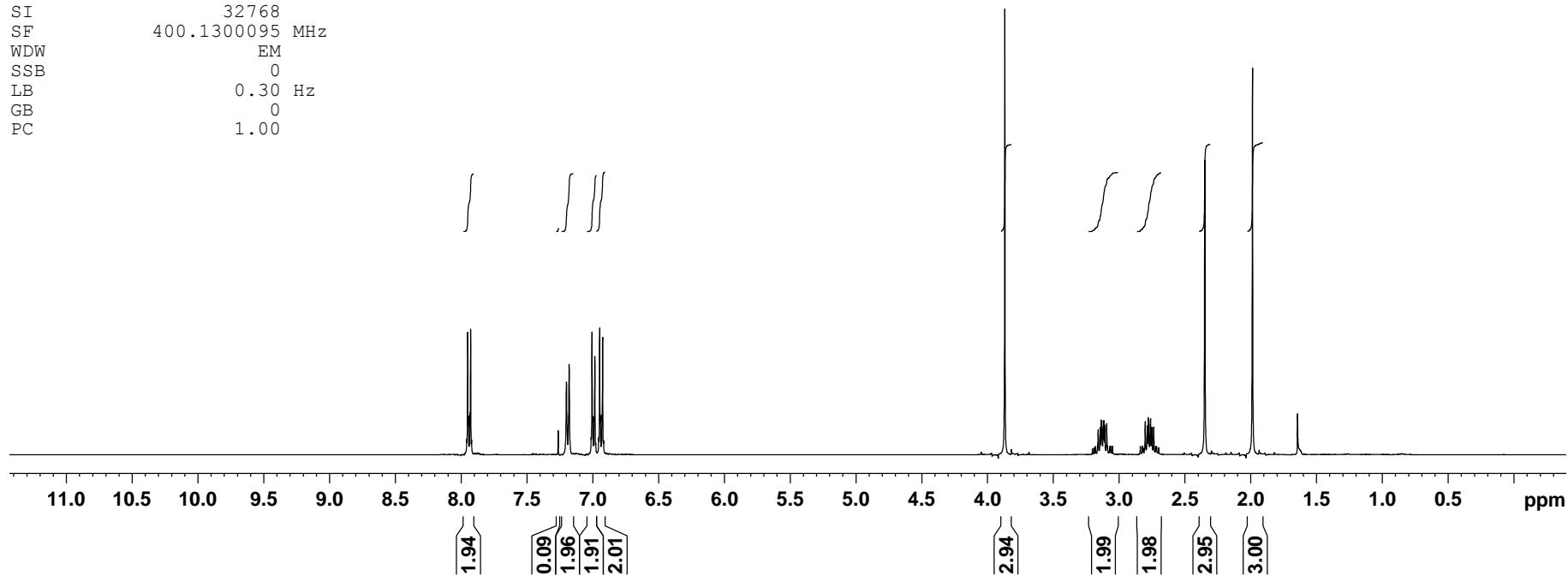
**Fig S60. HPLC Profile of Enantioenriched 3h**

NAME INN-4-KSB-106B-1H  
 EXPNO 3  
 PROCNO 1  
 Date 20130901  
 Time 23.03  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 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 294.7 K  
 D1 1.00000000 sec  
 TDO 1

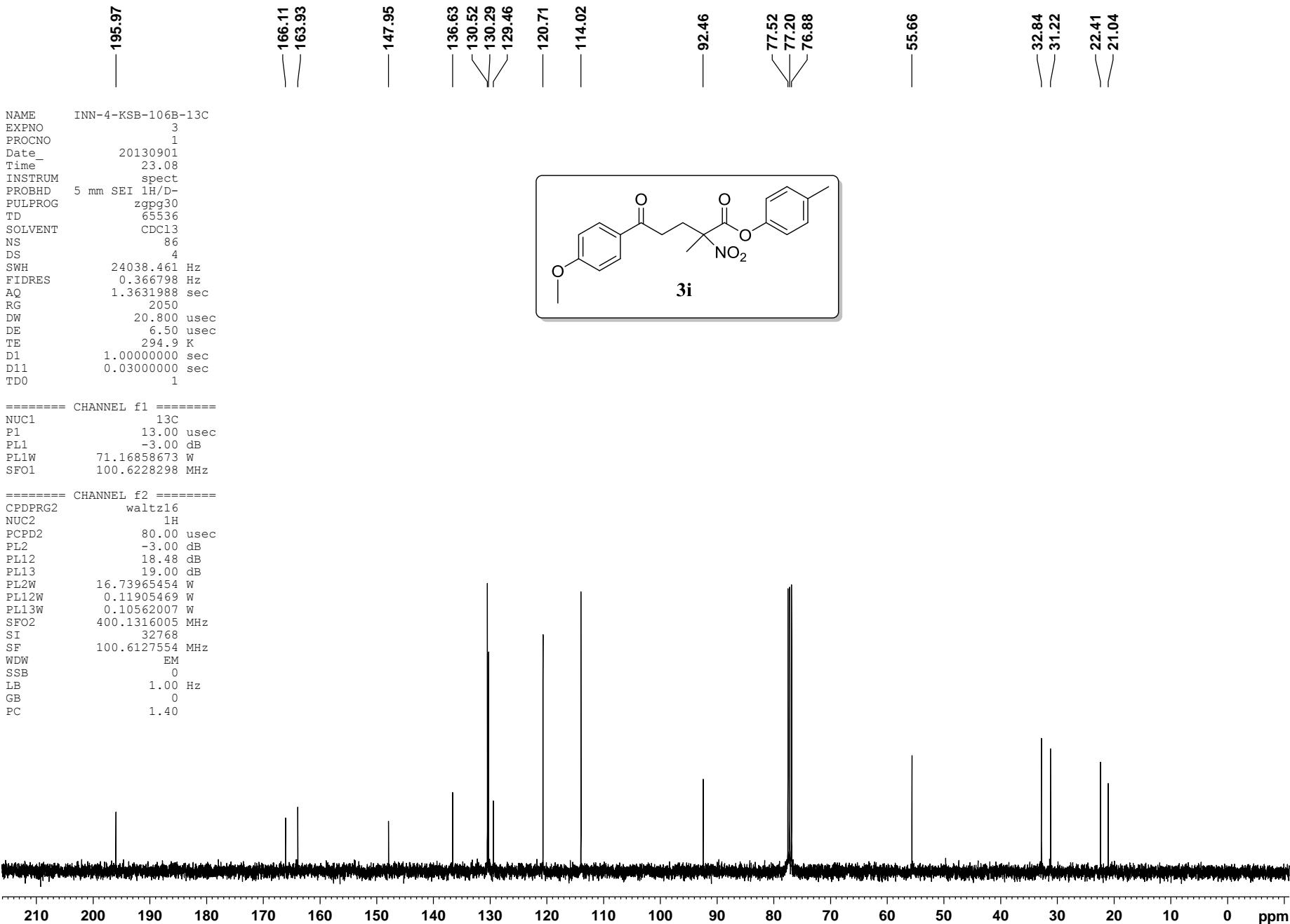


===== CHANNEL f1 =====

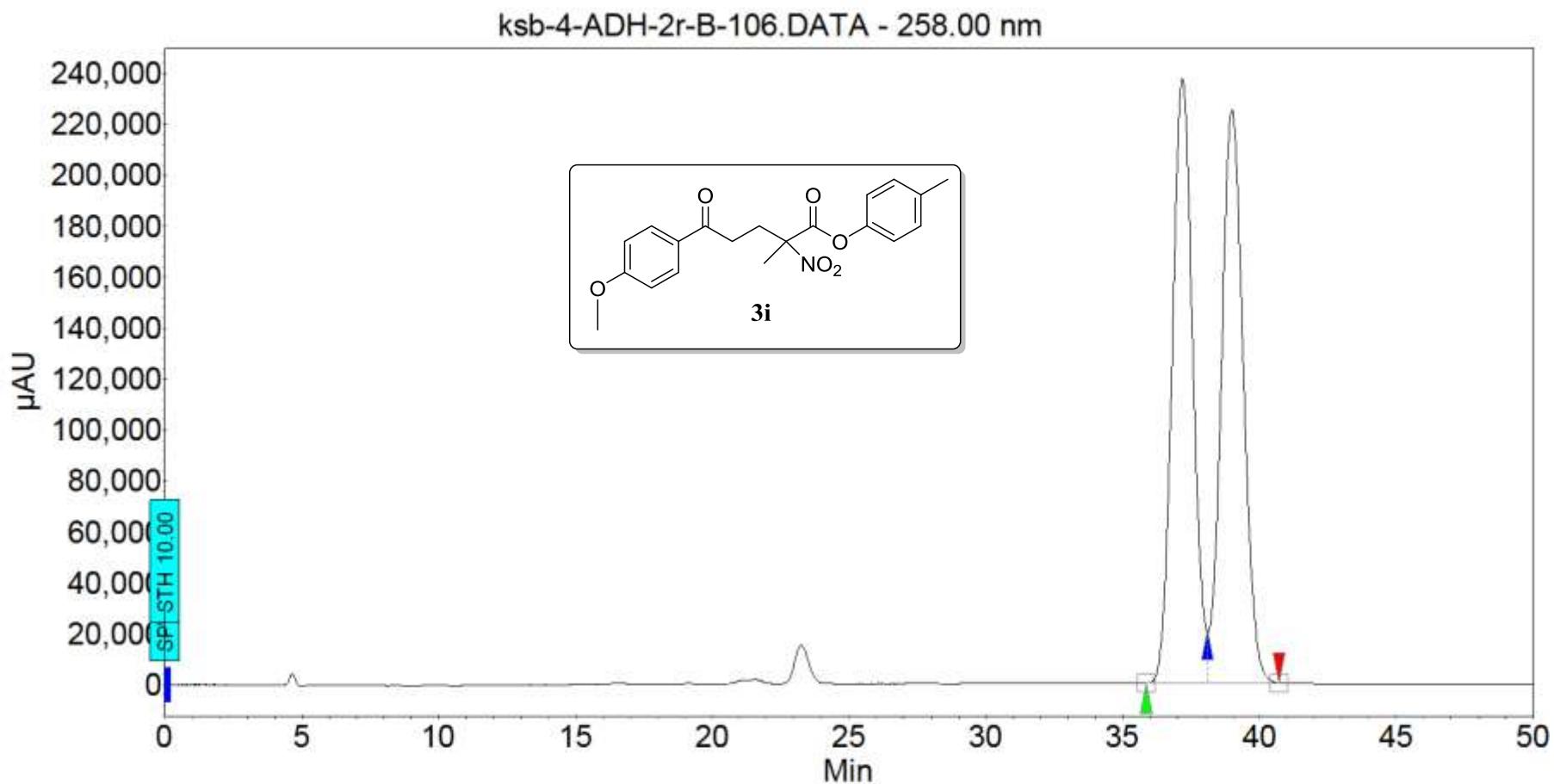
NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300095 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S61.**  $^1\text{H}$  NMR Spectrum of **3i**



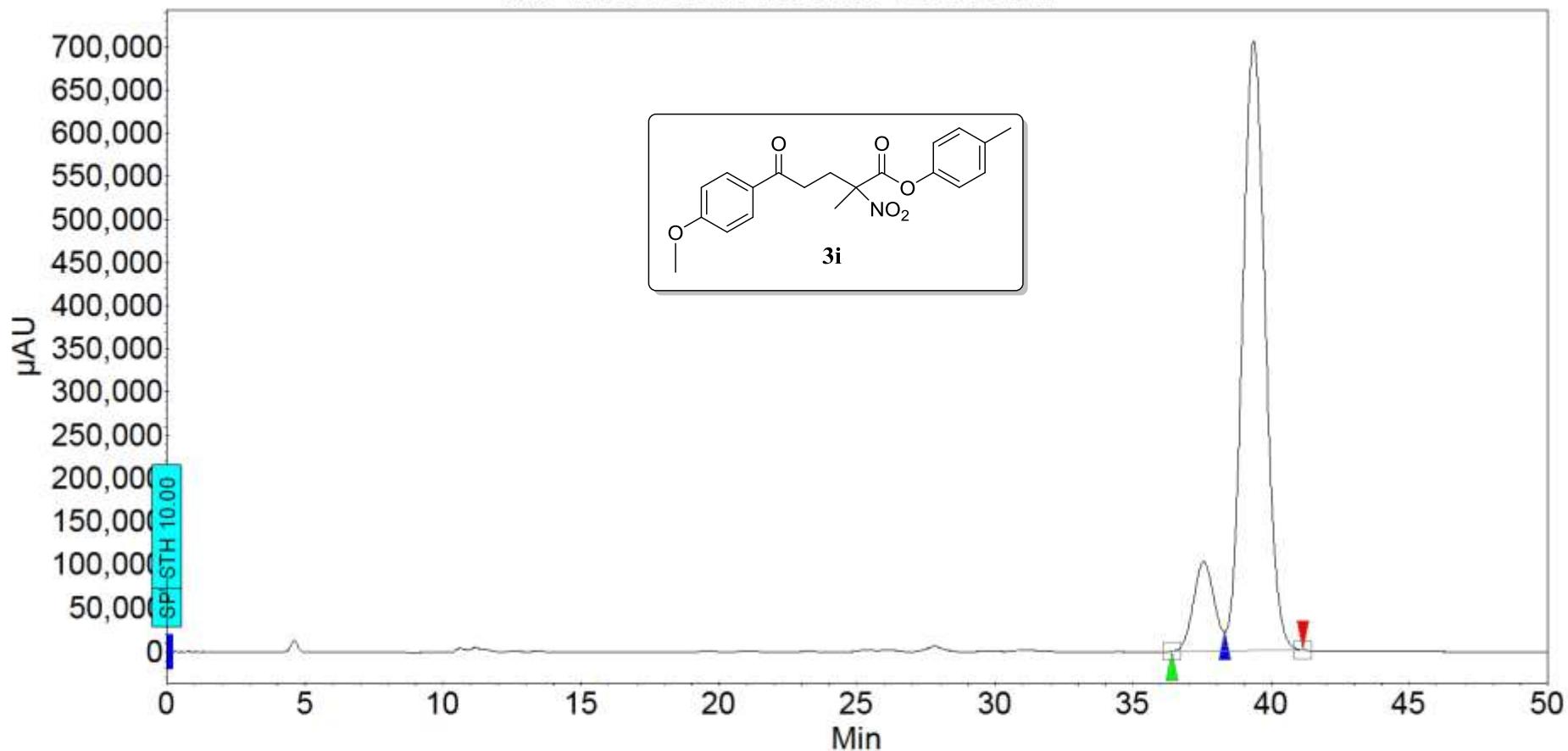
**Fig S62.**  $^{13}\text{C}$  NMR Spectrum of **3i**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	37.160	49.71	237261.5	206486.3	49.711
2	UNKNOWN	38.986	50.29	224863.8	208884.8	50.289
Total			100.00	462125.3	415371.0	100.000

**Fig S63. HPLC Profile of Racemic 3i**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area [%]
1	UNKNOWN	37.533	11.91	103741.5	90813.7	11.909
2	UNKNOWN	39.346	88.09	706068.2	671773.9	88.091
Total			100.00	809809.8	762587.6	100.000

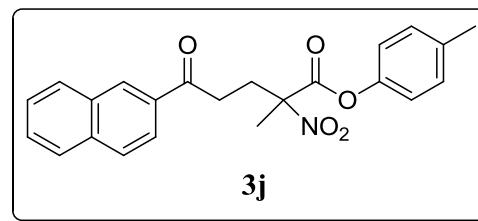
Fig S64. HPLC Profile of Enantioenriched **3i**

Current Data Parameters  
NAME INN-4-KSB-121B-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131207  
Time 9.53  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 52  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 80.35  
DW 50.000 usec  
DE 6.50 usec  
TE 297.0 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300121 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



3j

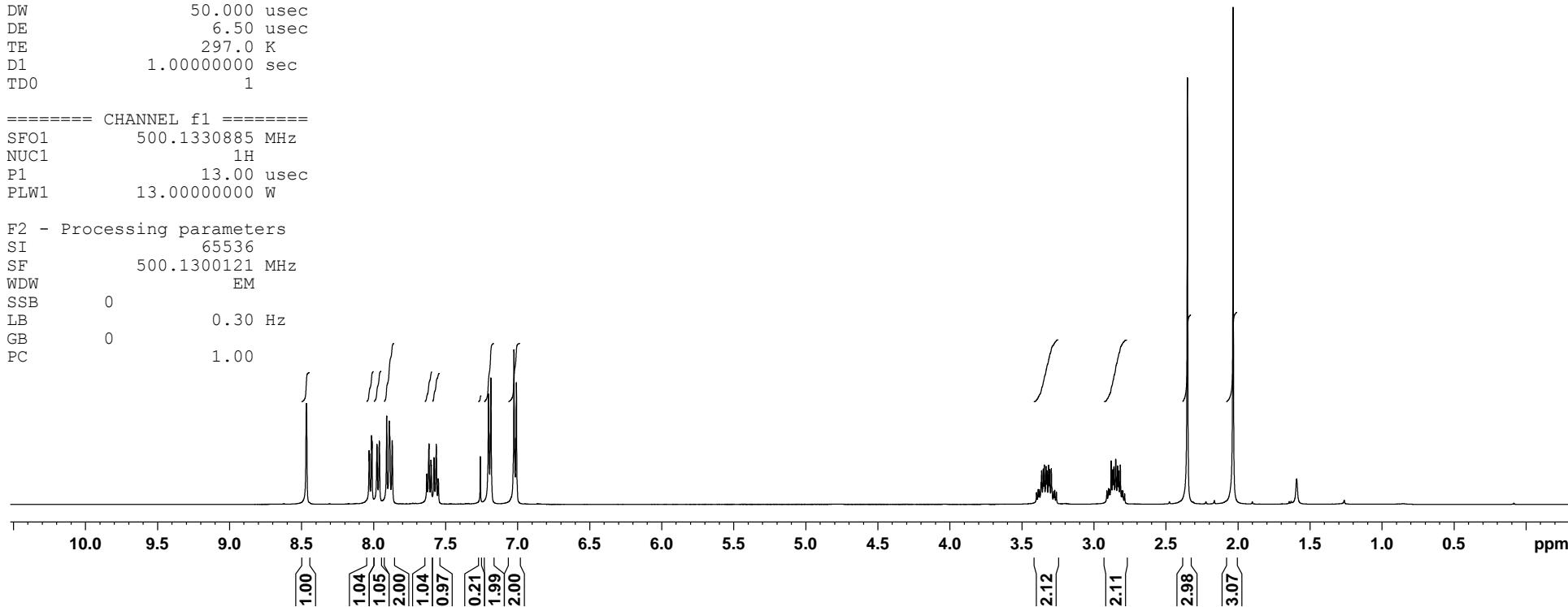


Fig S65. <sup>1</sup>H NMR Spectrum of 3j

— 197.38

— 166.11

— 147.95  
— 136.65  
— 135.88  
— 133.69  
— 132.59  
— 130.30  
— 129.99  
— 129.77  
— 128.85  
— 128.78  
— 127.94  
— 127.08  
— 123.76  
— 120.71

— 92.44

— 77.45  
— 77.20  
— 76.94

— 33.29

— 31.20

— 22.48

— 21.03

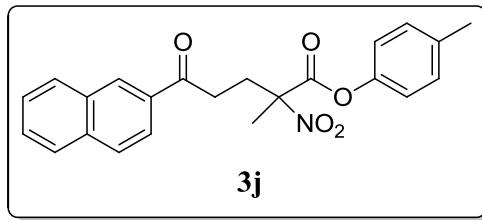
Current Data Parameters  
NAME INN-4-KSB-121B-13C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date 20131014  
Time 20.22  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 157  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AO 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 296.0 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 125.7577737 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



3j

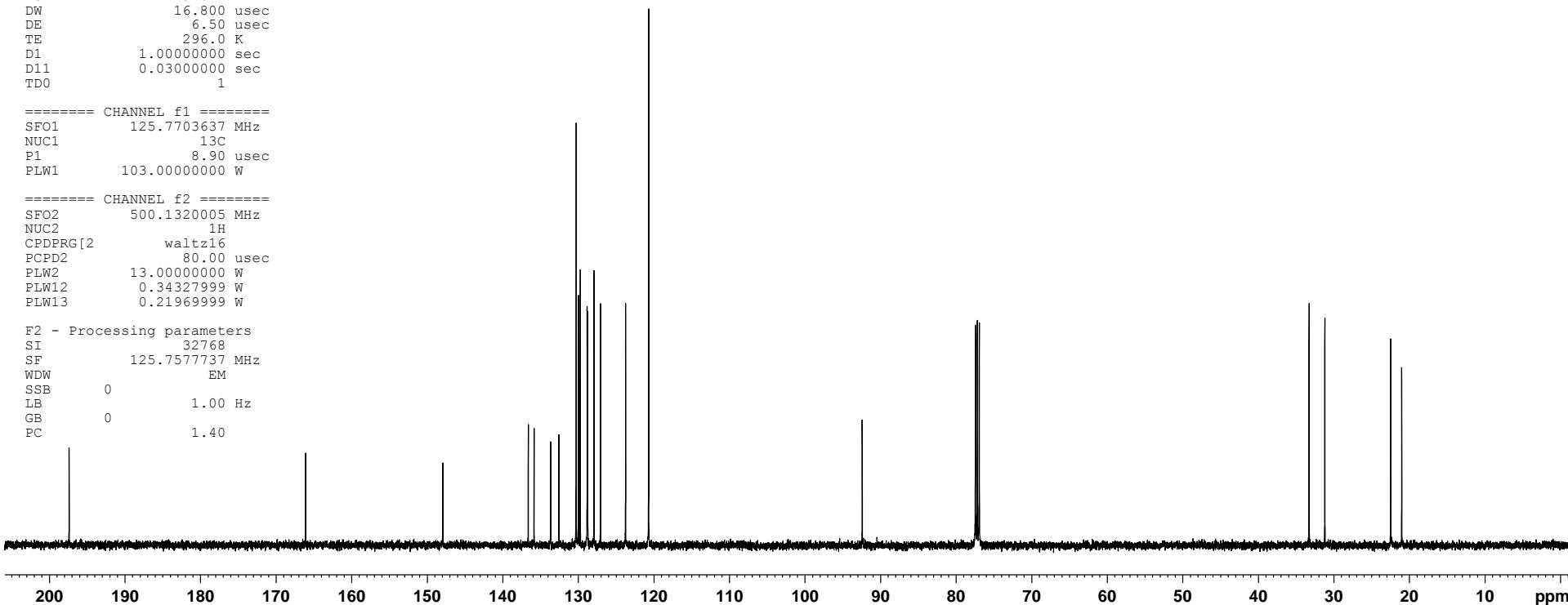
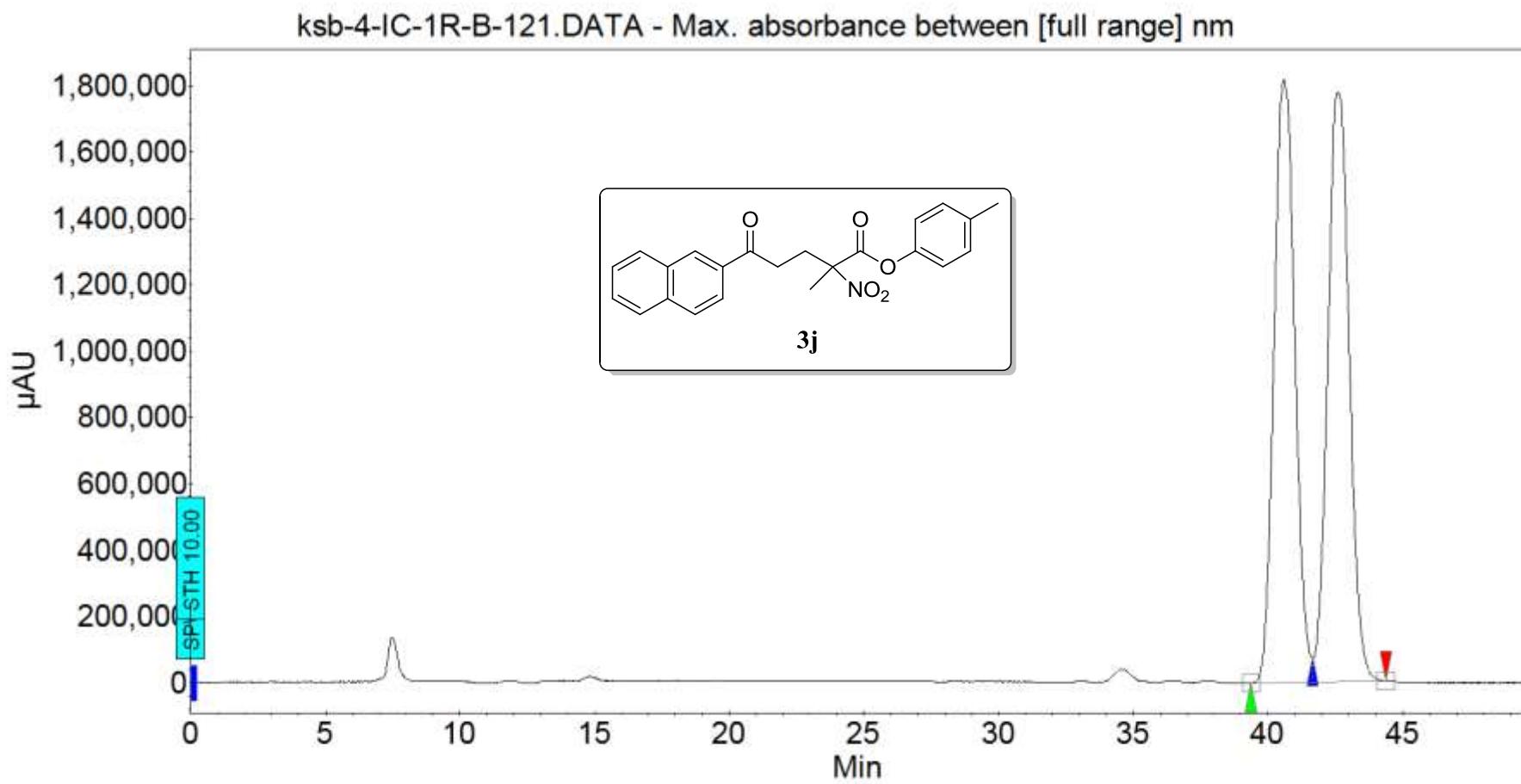


Fig S66. <sup>13</sup>C NMR Spectrum of 3j

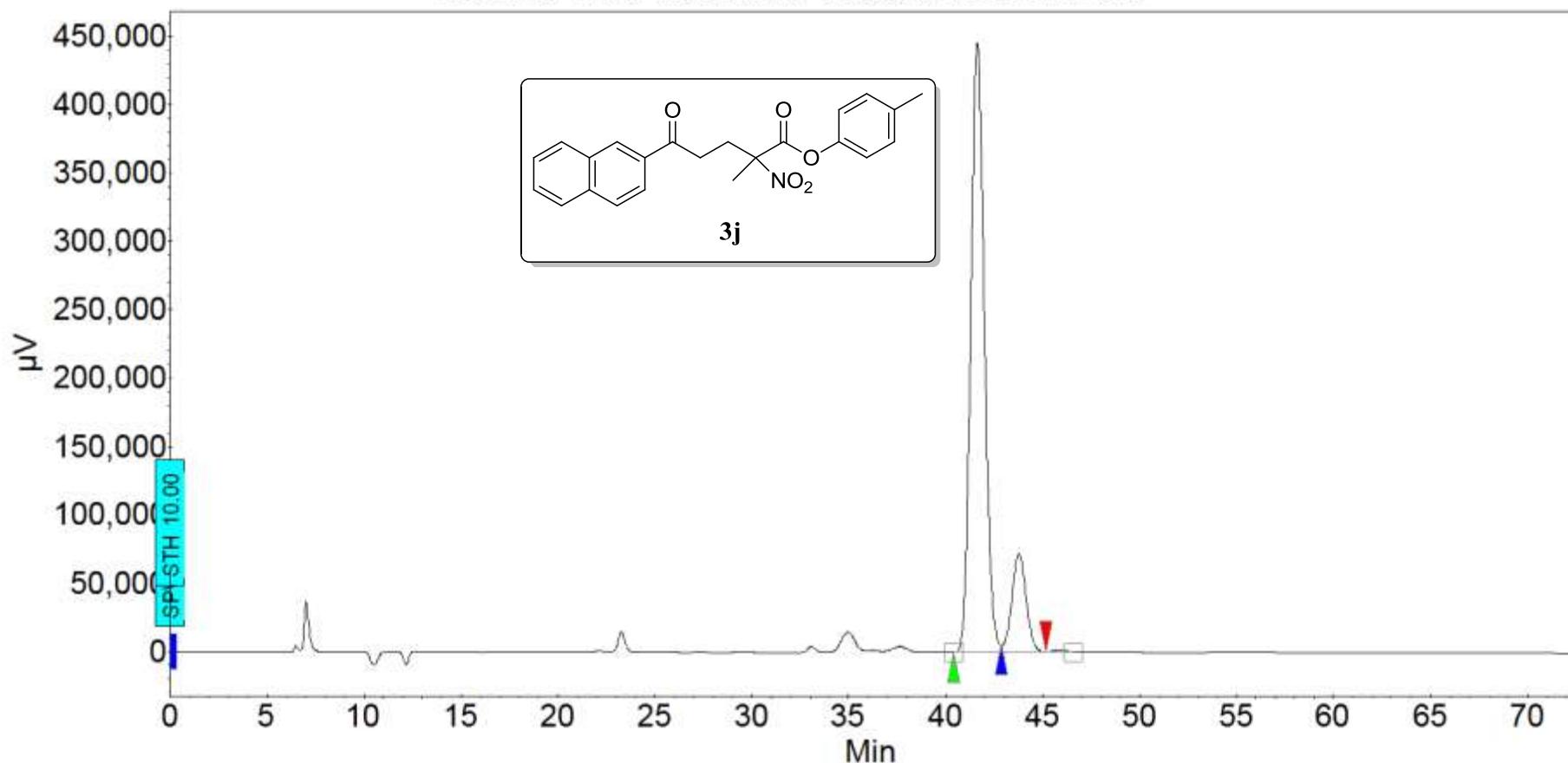


### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	40.597	49.60	1817263.9	1627245.6	49.597
2	UNKNOWN	42.610	50.40	1776214.1	1653720.6	50.403
Total			100.00	3593478.0	3280966.2	100.000

Fig S67. HPLC Profile of Racemic 3j

ksb-4-IC-1R-B-126.DATA - Jasco UV DETECTOR



**Peak results :**

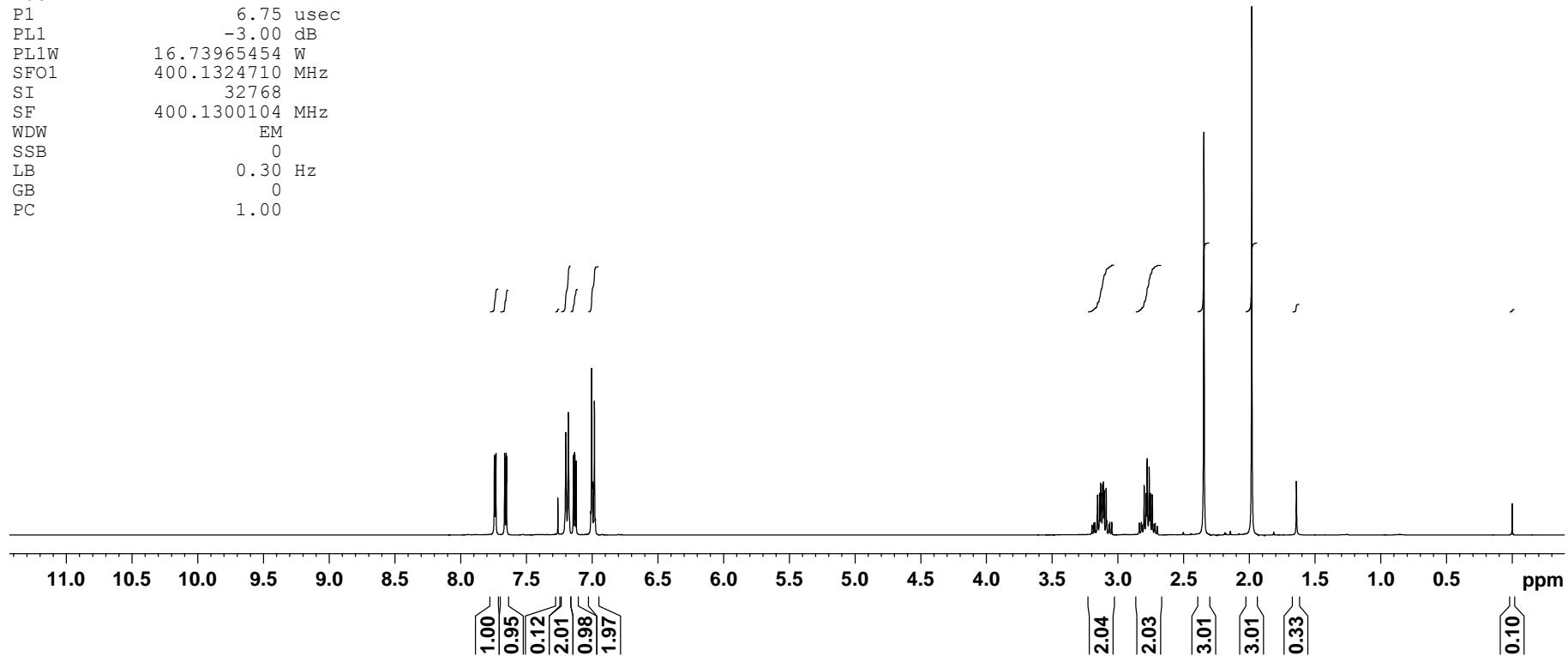
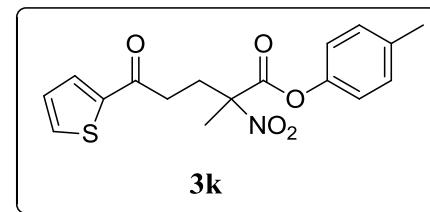
Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu$ V]	Area [ $\mu$ V.Min]	Area % [%]
1	UNKNOWN	41.625	85.50	445824.4	362537.1	85.499
2	UNKNOWN	43.767	14.50	71919.6	61488.2	14.501
Total			100.00	517744.1	424025.4	100.000

Fig S68. HPLC Profile of Enantioenriched 3j

NAME INN-4-KSB-107C-1H  
 EXPNO 4  
 PROCNO 1  
 Date\_ 20130831  
 Time 13.06  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 50.8  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.5 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====

NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300104 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S69.**  $^1\text{H}$  NMR Spectrum of **3k**

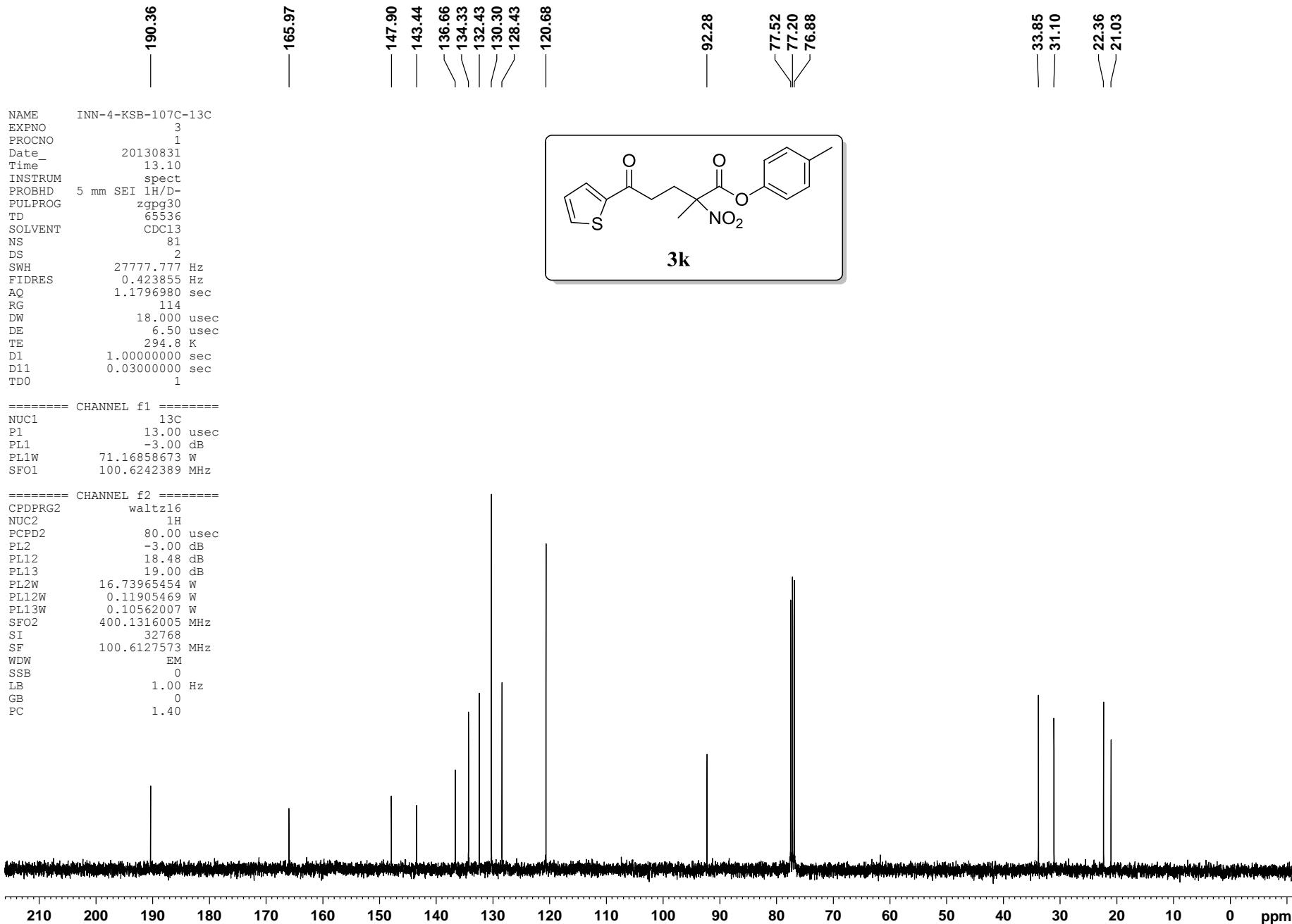
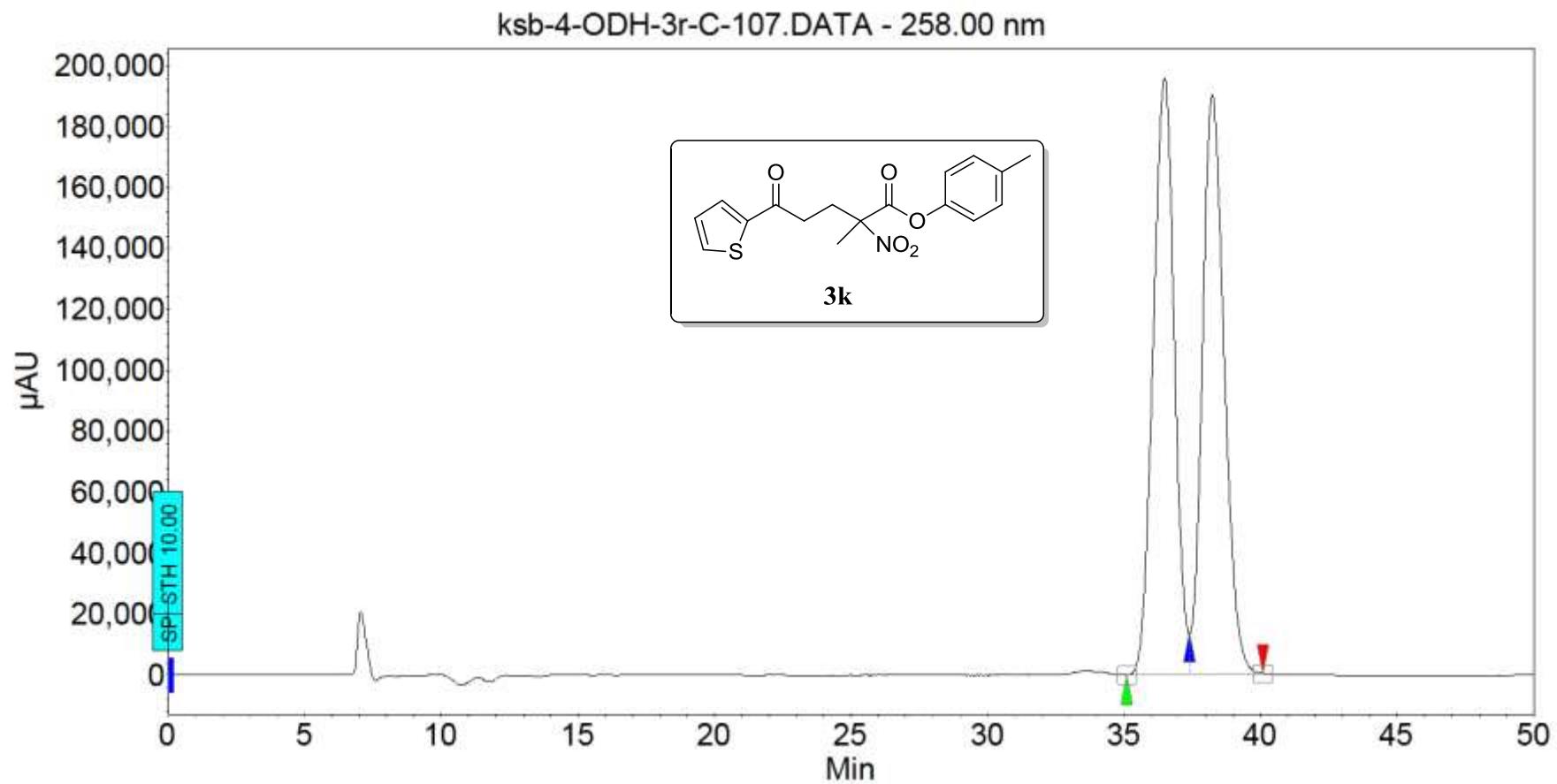


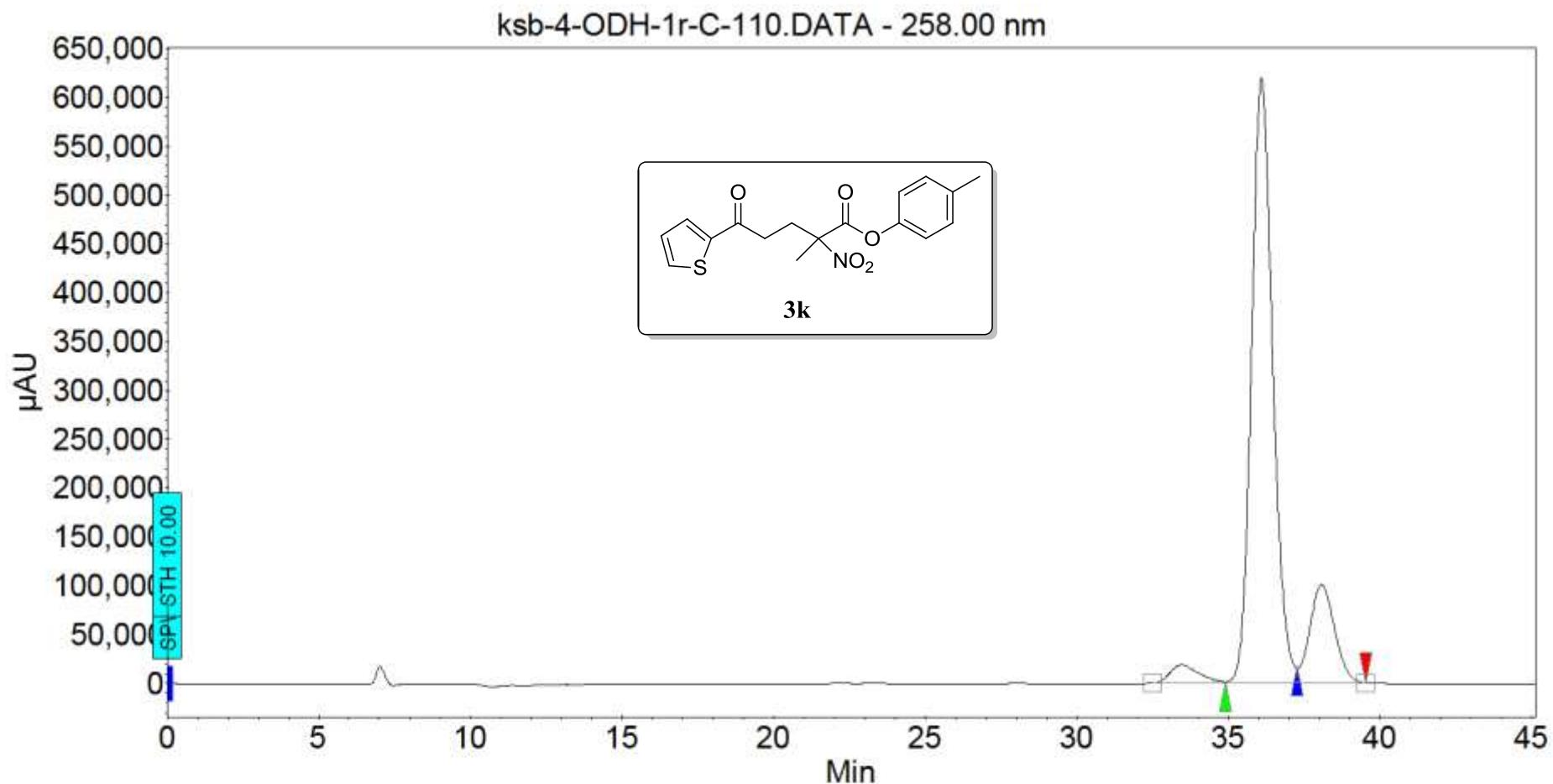
Fig S70.  $^{13}\text{C}$  NMR Spectrum of 3k



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area [%]
1	UNKNOWN	36.473	50.05	195629.9	176421.0	50.052
2	UNKNOWN	38.231	49.95	190077.6	176051.0	49.948
Total			100.00	385707.6	352472.1	100.000

Fig S71. HPLC Profile of Racemic 3k



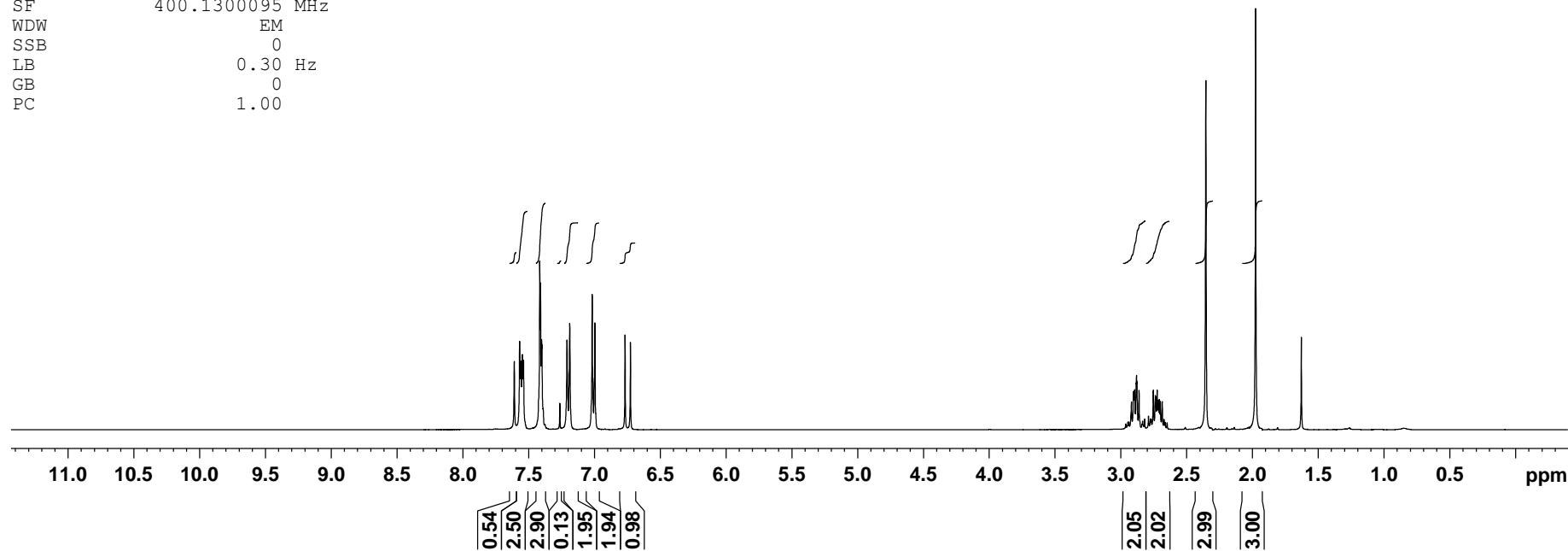
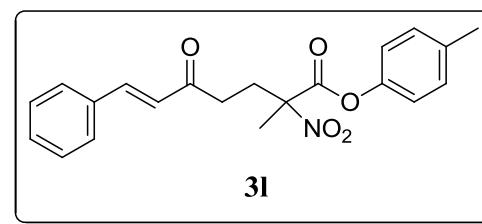
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{AU}$ ]	Area [ $\mu\text{AU}.\text{Min}$ ]	Area % [%]
1	UNKNOWN	36.077	83.98	619142.1	501894.6	83.980
2	UNKNOWN	38.064	16.02	100761.1	95742.2	16.020
Total			100.00	719903.2	597636.8	100.000

Fig S72. HPLC Profile of Enantioenriched 3k

NAME INN-4-KSB-107B-1H  
 EXPNO 3  
 PROCNO 1  
 Date 20130901  
 Time 23.13  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 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 294.7 K  
 D1 1.00000000 sec  
 TDO 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  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S73. <sup>1</sup>H NMR Spectrum of 3l**

— 197.26

— 166.05

— 147.94  
— 143.58  
— 136.65  
— 134.29  
— 130.94  
— 130.31  
— 129.17  
— 128.56  
— 125.59  
— 120.71

— 92.37

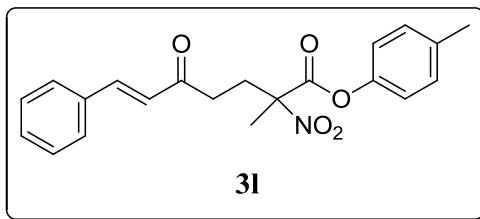
— 77.52  
— 77.20  
— 76.88

— 35.27  
— 30.88  
— 22.36  
— 21.05

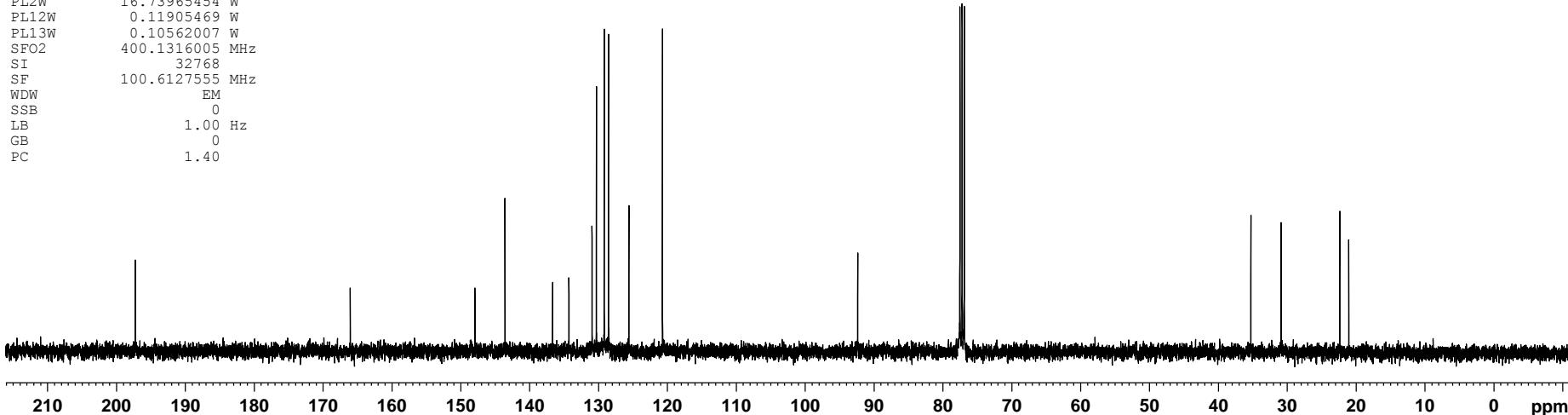
NAME INN-4-KSB-107B-13C  
EXPNO 3  
PROCNO 1  
Date 20130901  
Time 23.16  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 129  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.50 usec  
TE 294.9 K  
D1 1.0000000 sec  
D11 0.03000000 sec  
TDO 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 100.6127555 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

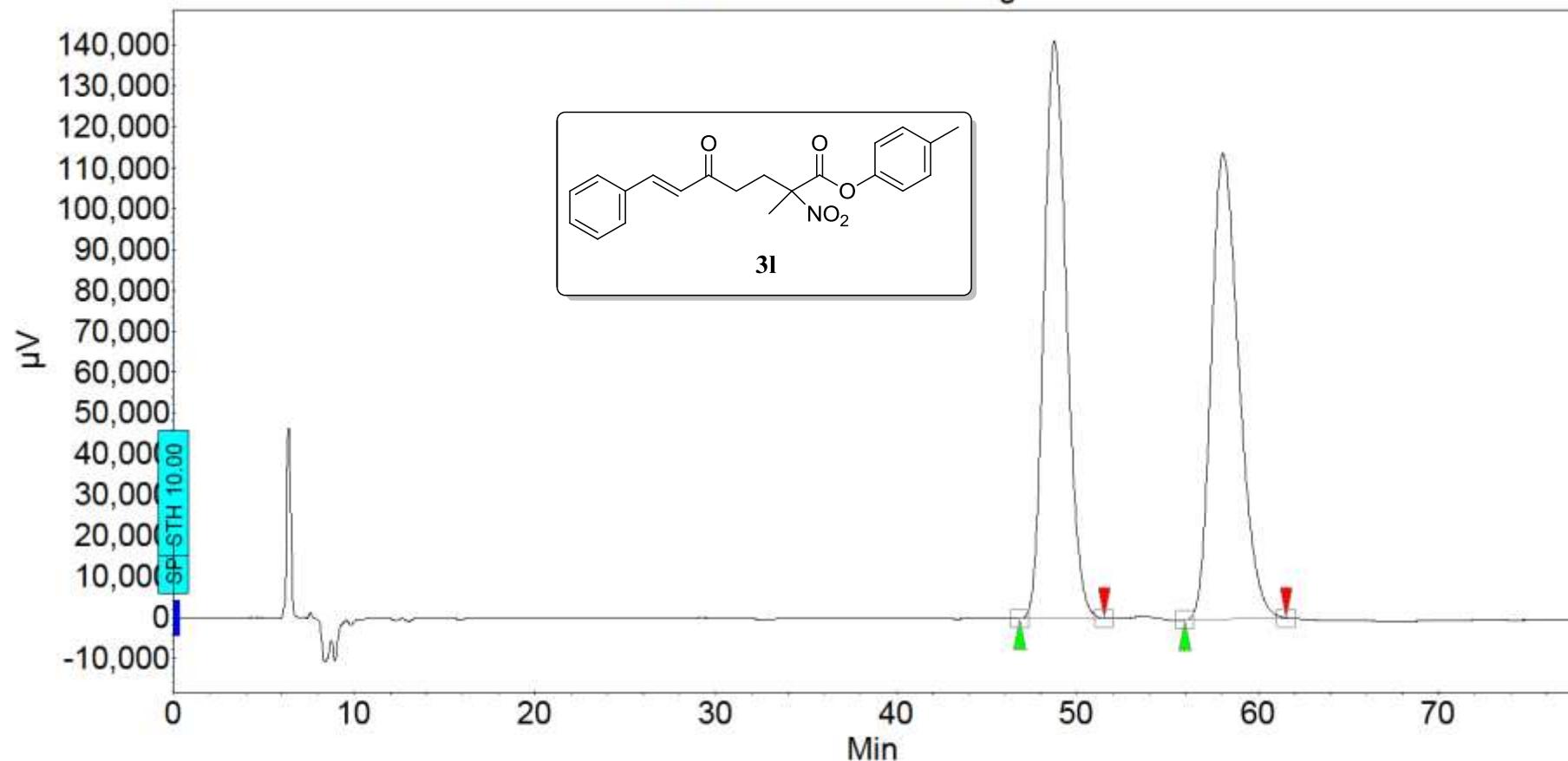


**3l**



**Fig S74. <sup>13</sup>C NMR Spectrum of 3l**

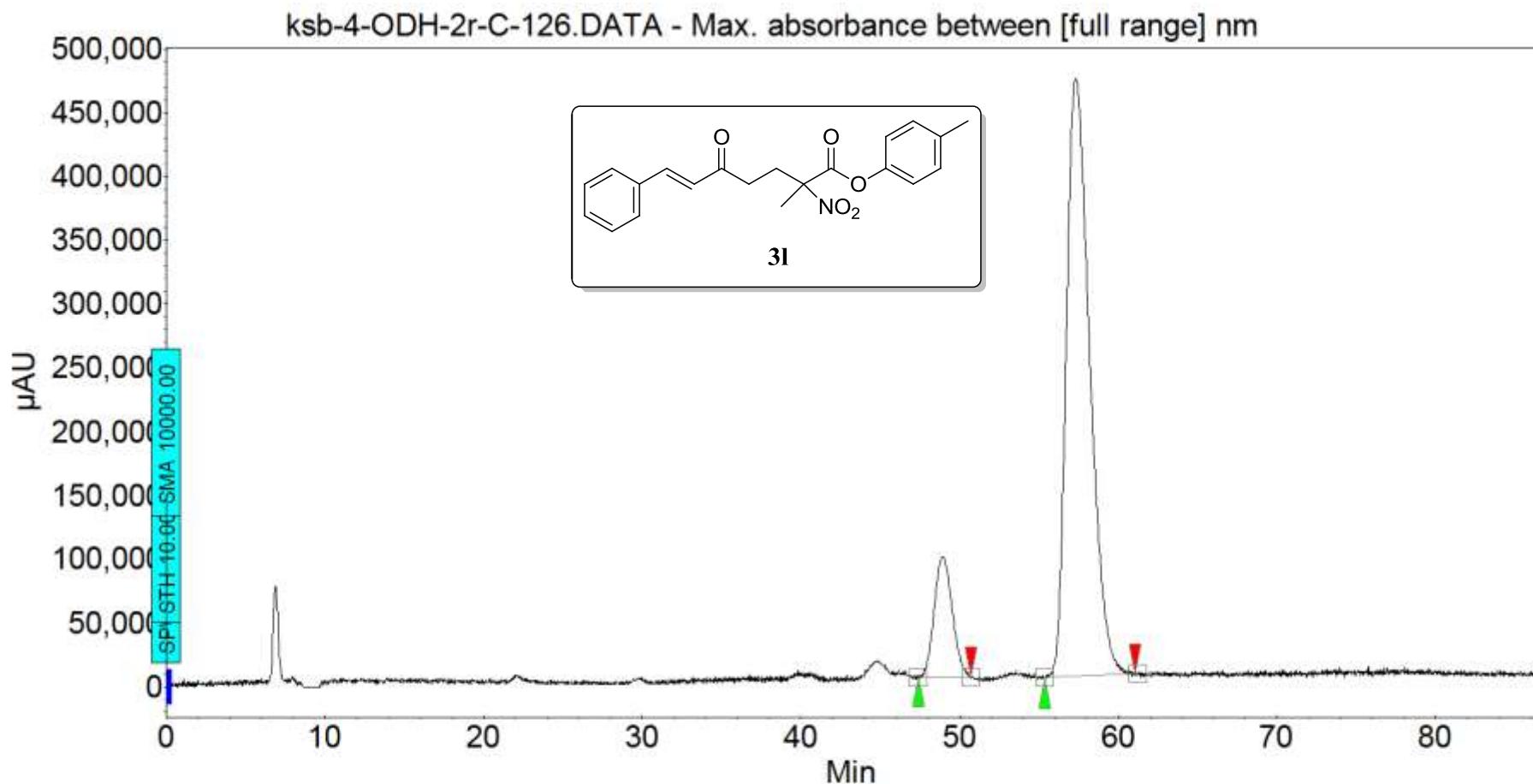
ksb-4-ODH-2r-B-107.DATA - Jasco Analog Channel 1



**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{V}$ ]	Area [ $\mu\text{V}.\text{Min}$ ]	Area % [%]
1	UNKNOWN	48.733	50.01	141506.7	202925.9	50.005
2	UNKNOWN	58.083	49.99	114095.1	202881.4	49.995
Total			100.00	255601.8	405807.3	100.000

**Fig S75. HPLC Profile of Racemic **3l****



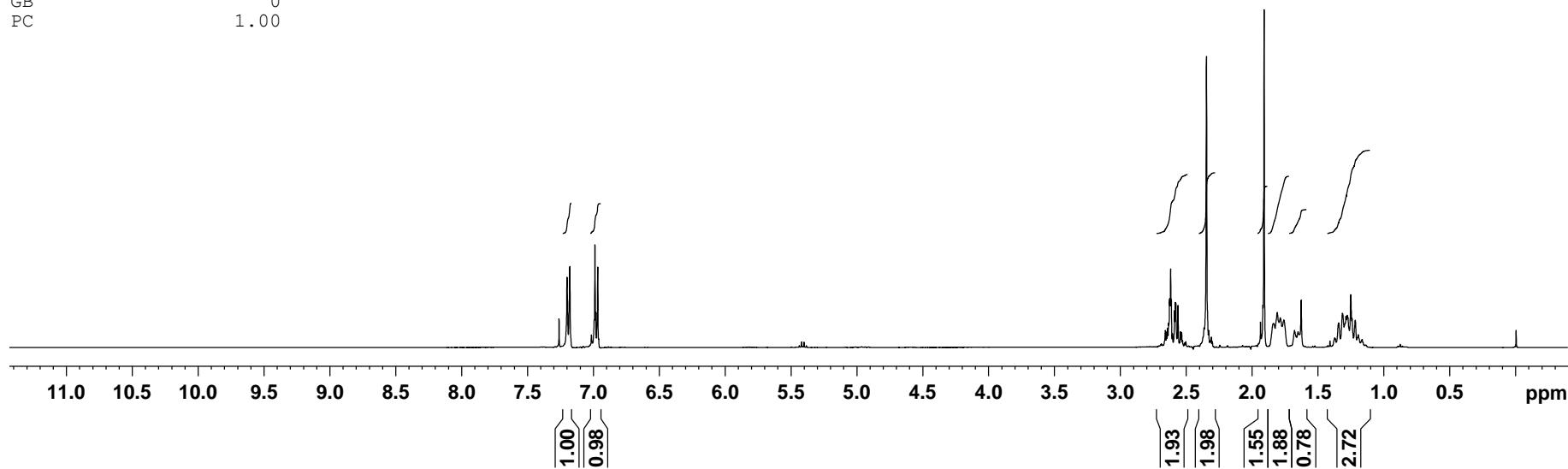
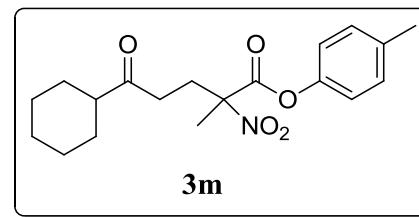
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	48.942	13.54	94497.7	124600.3	13.544
2	UNKNOWN	57.313	86.46	468511.2	795387.4	86.456
Total			100.00	563008.8	919987.7	100.000

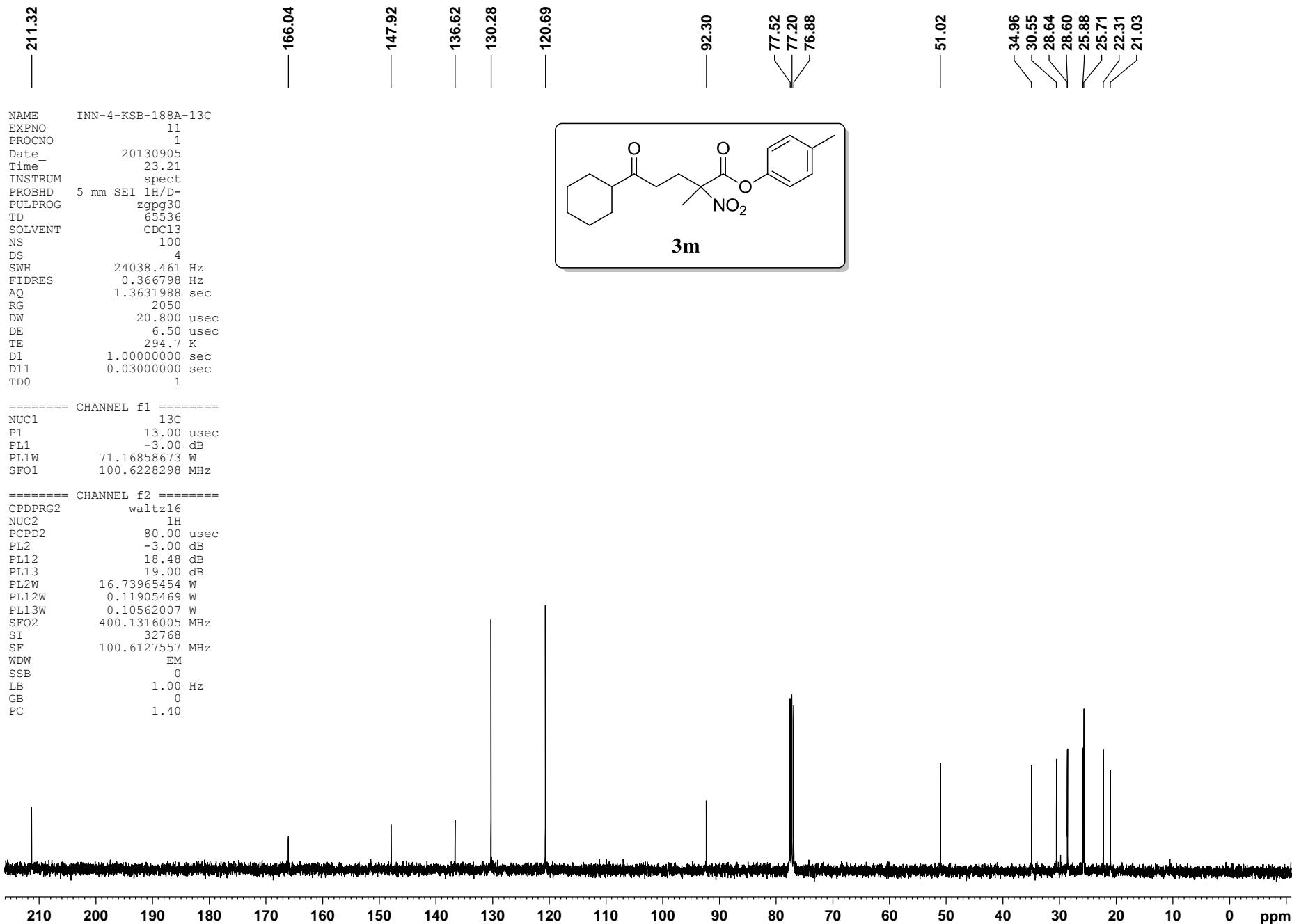
**Fig S76. HPLC Profile of Enantioenriched 3l**

NAME INN-4-KSB-108A-1H  
 EXPNO 1  
 PROCNO 1  
 Date 20130903  
 Time 12.56  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 49340  
 SOLVENT CDCl3  
 NS 12  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.166674 Hz  
 AQ 2.999920 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.4 K  
 D1 1.0000000 sec  
 TD0 1

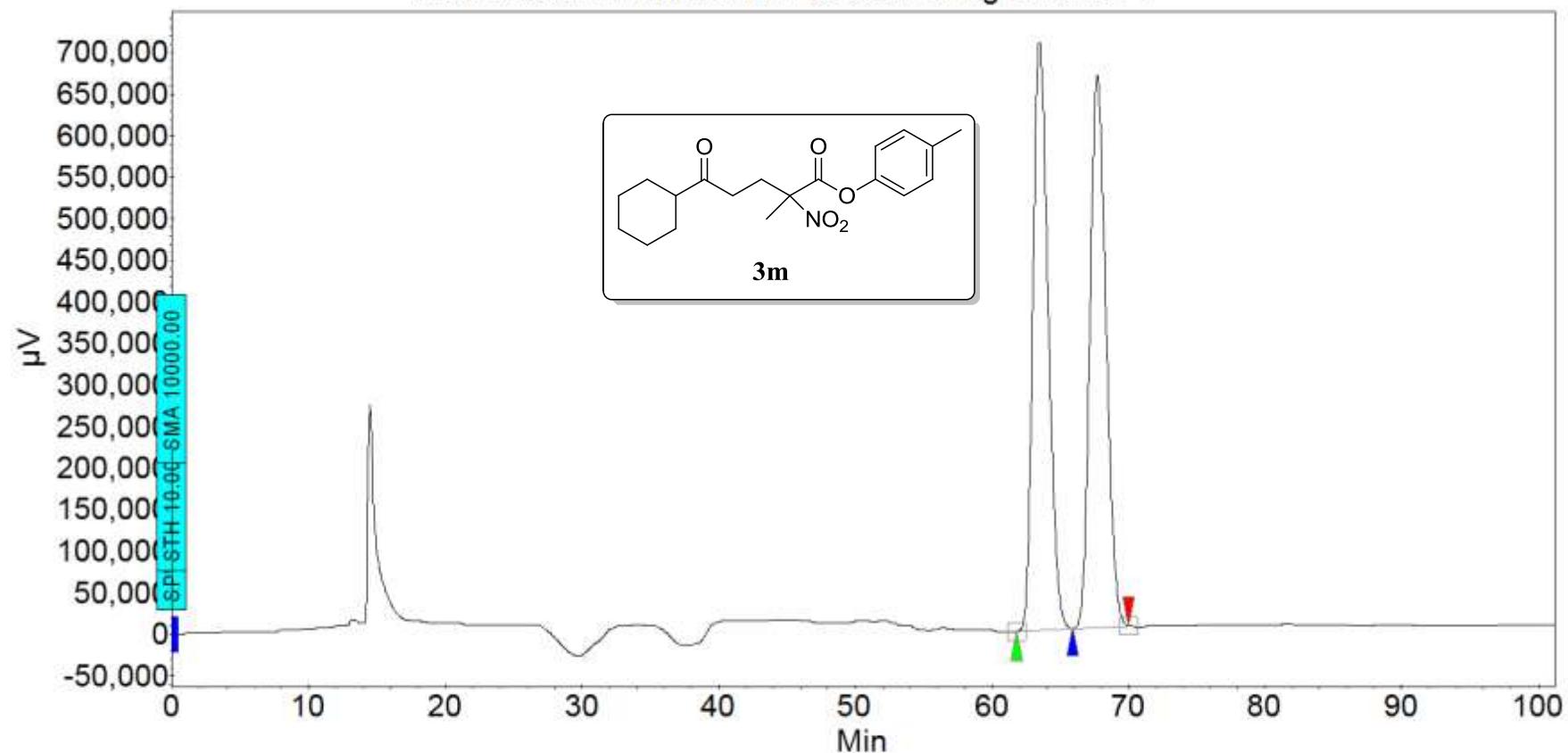
===== CHANNEL f1 ======  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300103 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S77.** <sup>1</sup>H NMR Spectrum of **3m**

Fig S78.  $^{13}\text{C}$  NMR Spectrum of 3m

KSB-4-IC-3R-A-108.DATA - Jasco Analog Channel 1



**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu V$ ]	Area [ $\mu V \cdot Min$ ]	Area % [%]
1	UNKNOWN	63.483	50.11	707520.5	917141.6	50.112
2	UNKNOWN	67.717	49.89	664199.8	913057.7	49.888
Total			100.00	1371720.3	1830199.3	100.000

**Fig S79.** HPLC Profile of Racemic **3m**

KSB-4-IC-1R-A-127.DATA - Max. absorbance between [full range] nm

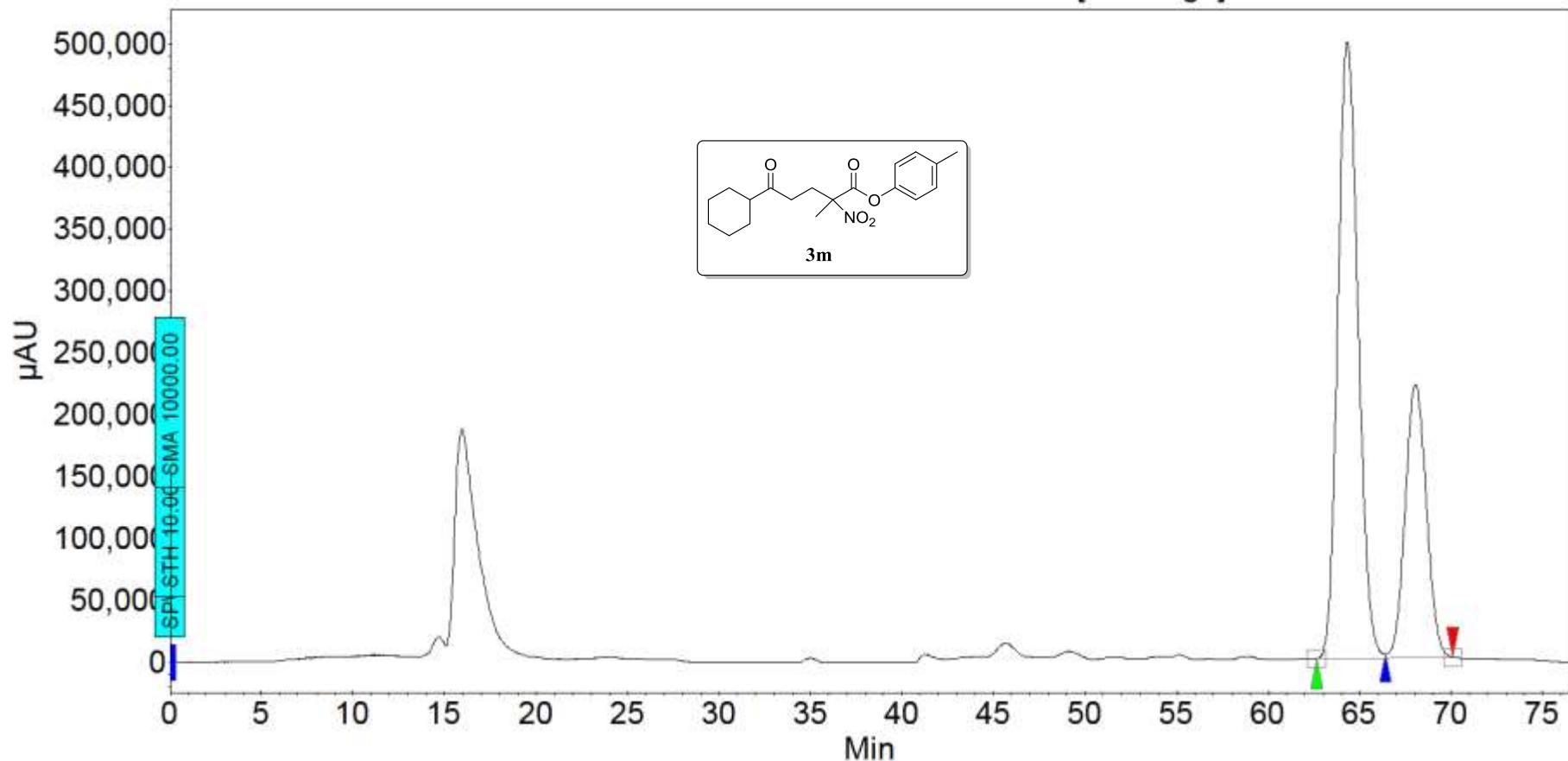


Fig S80. HPLC Profile of Enantioenriched 3m

Current Data Parameters  
NAME INN-2-NSS-129-1H  
EXPNO 9  
PROCNO 1

F2 - Acquisition Parameters  
Date 20160506  
Time 9.35  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 80.6  
DW 60.800 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 ======  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300104 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

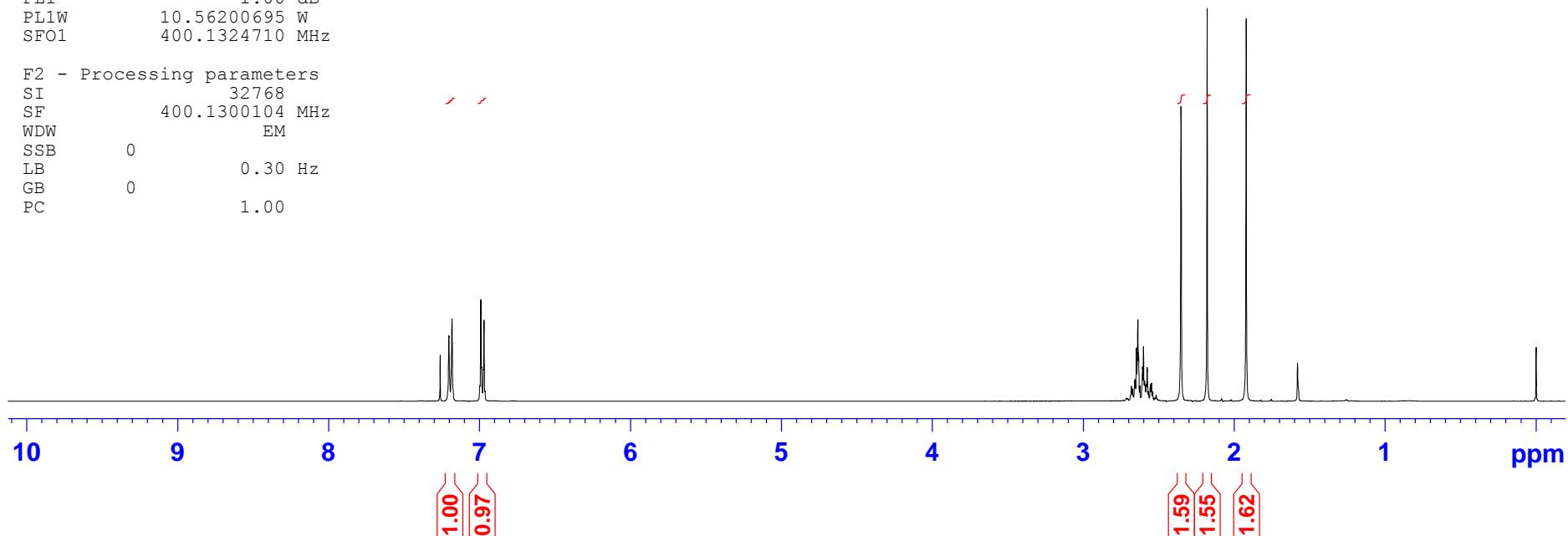
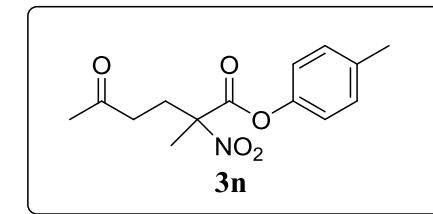
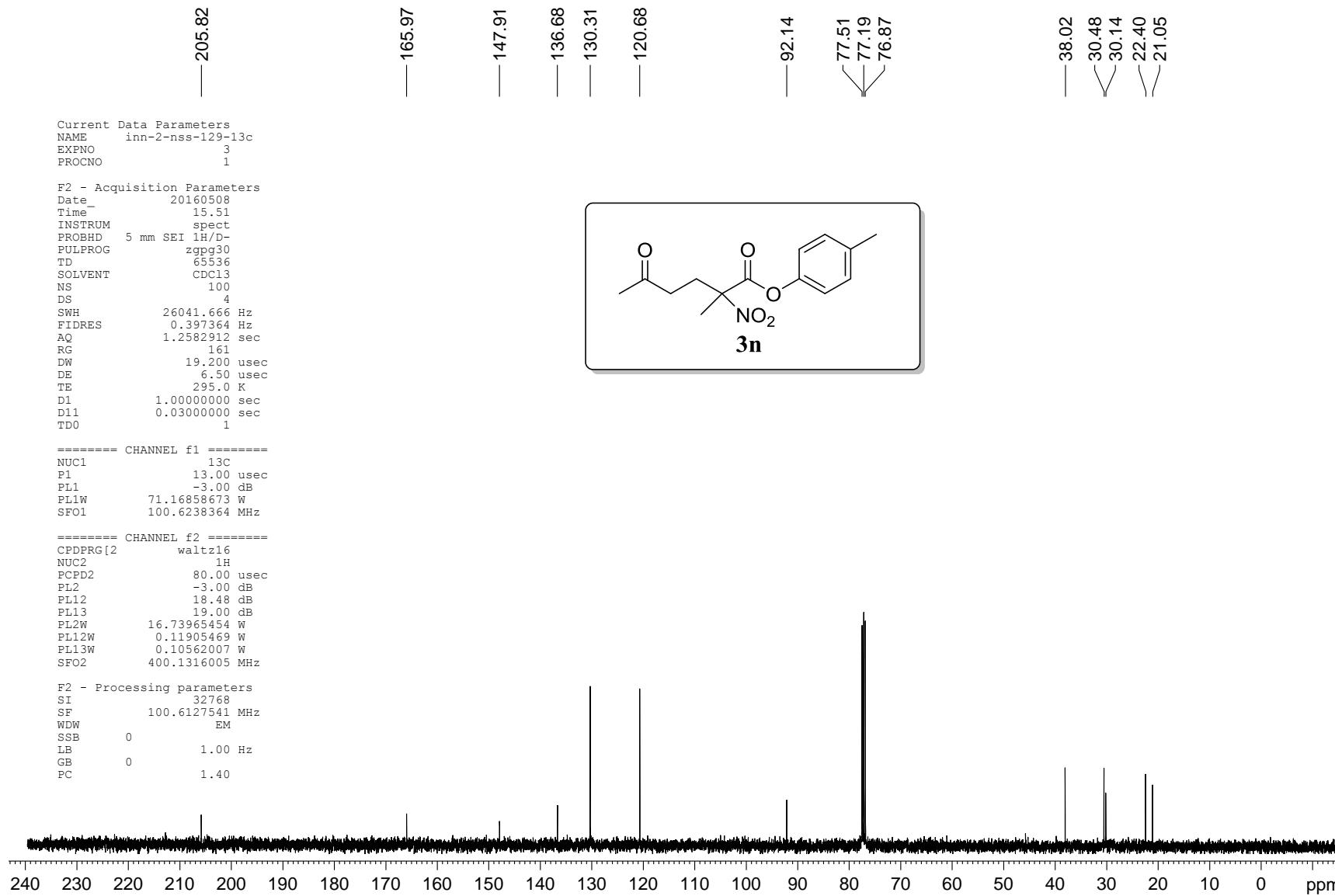
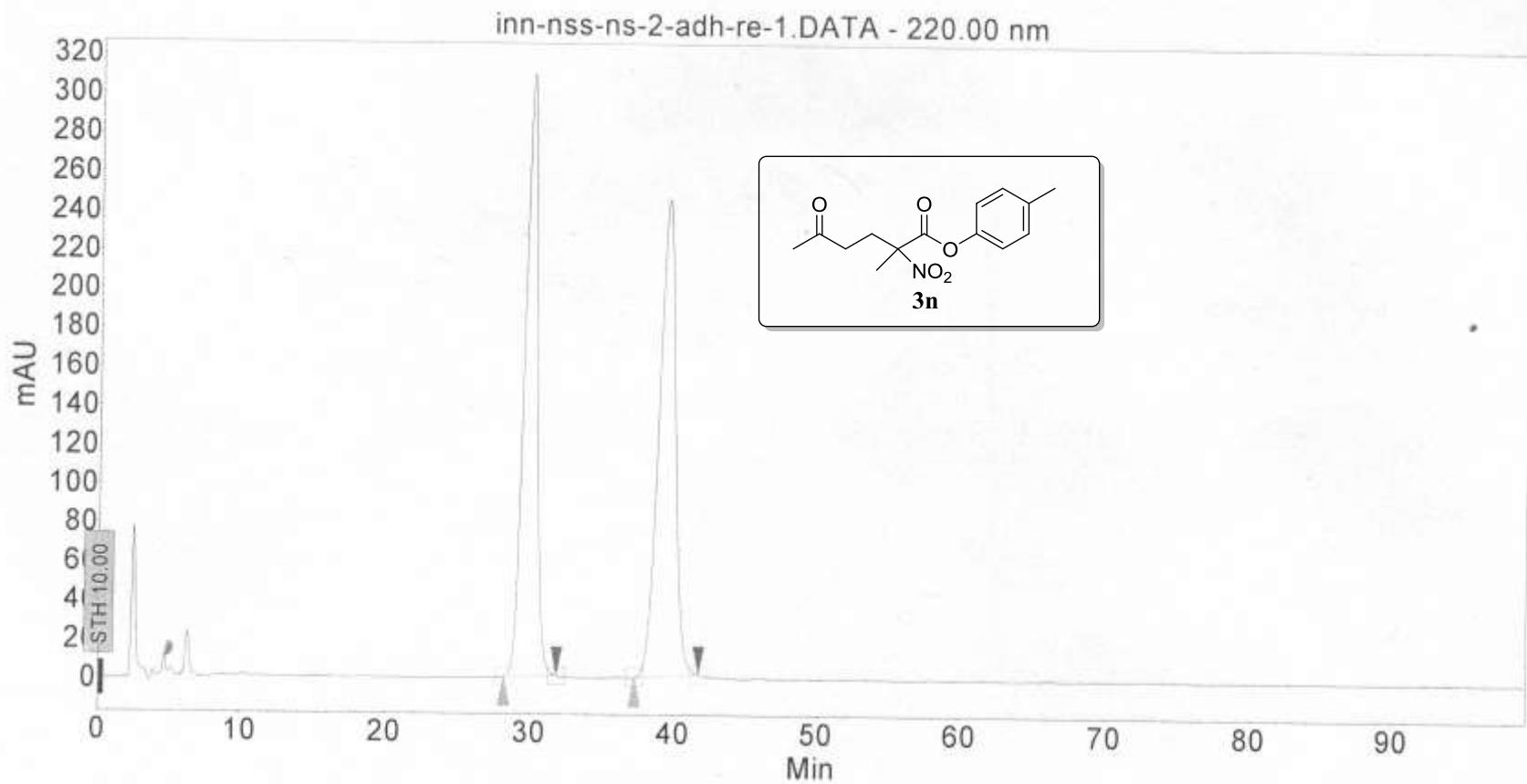


Fig S81.  $^1\text{H}$  NMR Spectrum of **3n**



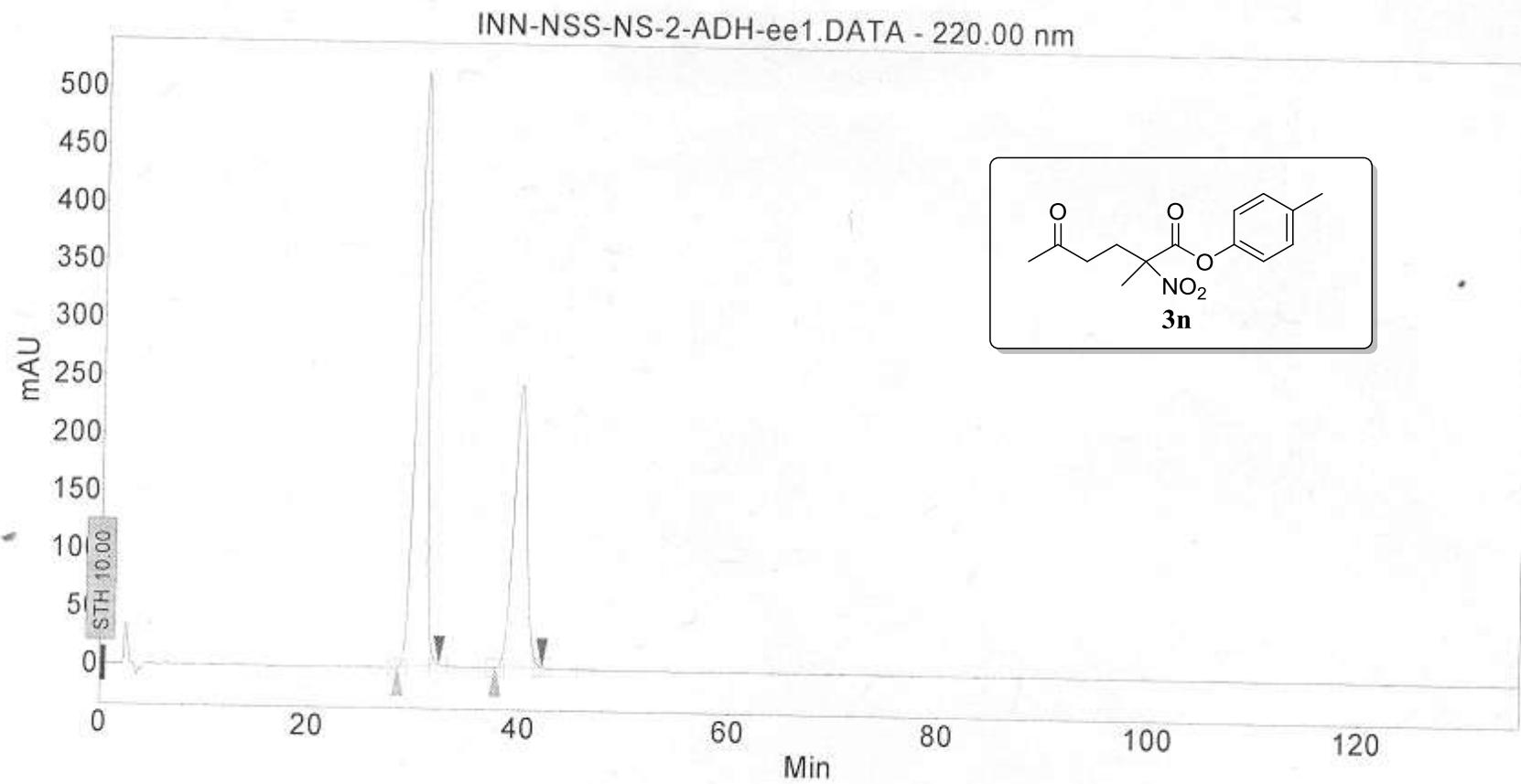
**Fig S82.**  $^{13}\text{C}$  NMR Spectrum of **3n**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area [%] [%]
1	UNKNOWN	29.923	49.13	308.7	317.0	49.127
2	UNKNOWN	39.426	50.87	244.9	328.3	50.873
Total			100.00	553.6	645.3	100.000

**Fig S83. HPLC Profile of Racemic 3n**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	30.505	63.65	512.6	613.1	63.653
2	UNKNOWN	39.896	36.35	244.3	350.1	36.347
Total			100.00	756.9	963.2	100.000

**Fig S84. HPLC Profile of Enantioenriched 3n**

Current Data Parameters  
NAME INN-4-KSB-131A-1H  
EXPNO 22  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131221  
Time\_ 21.27  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 36  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 85.91  
DW 50.000 usec  
DE 6.50 usec  
TE 295.0 K  
D1 1.0000000 sec  
TD0 1

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

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

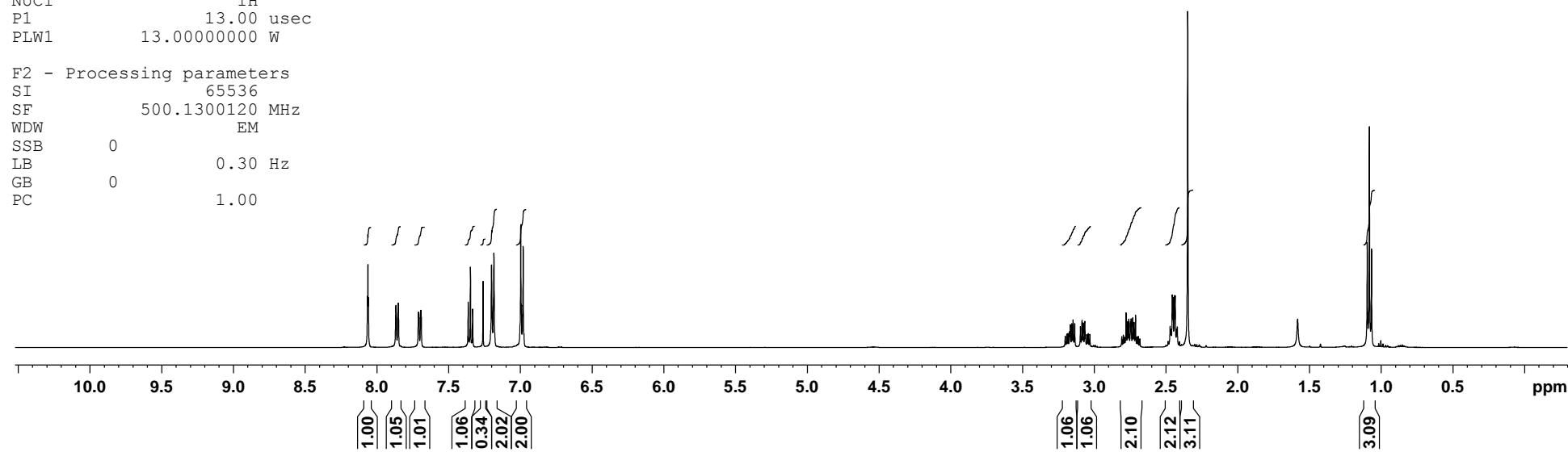


Fig S85. <sup>1</sup>H NMR Spectrum of 4a

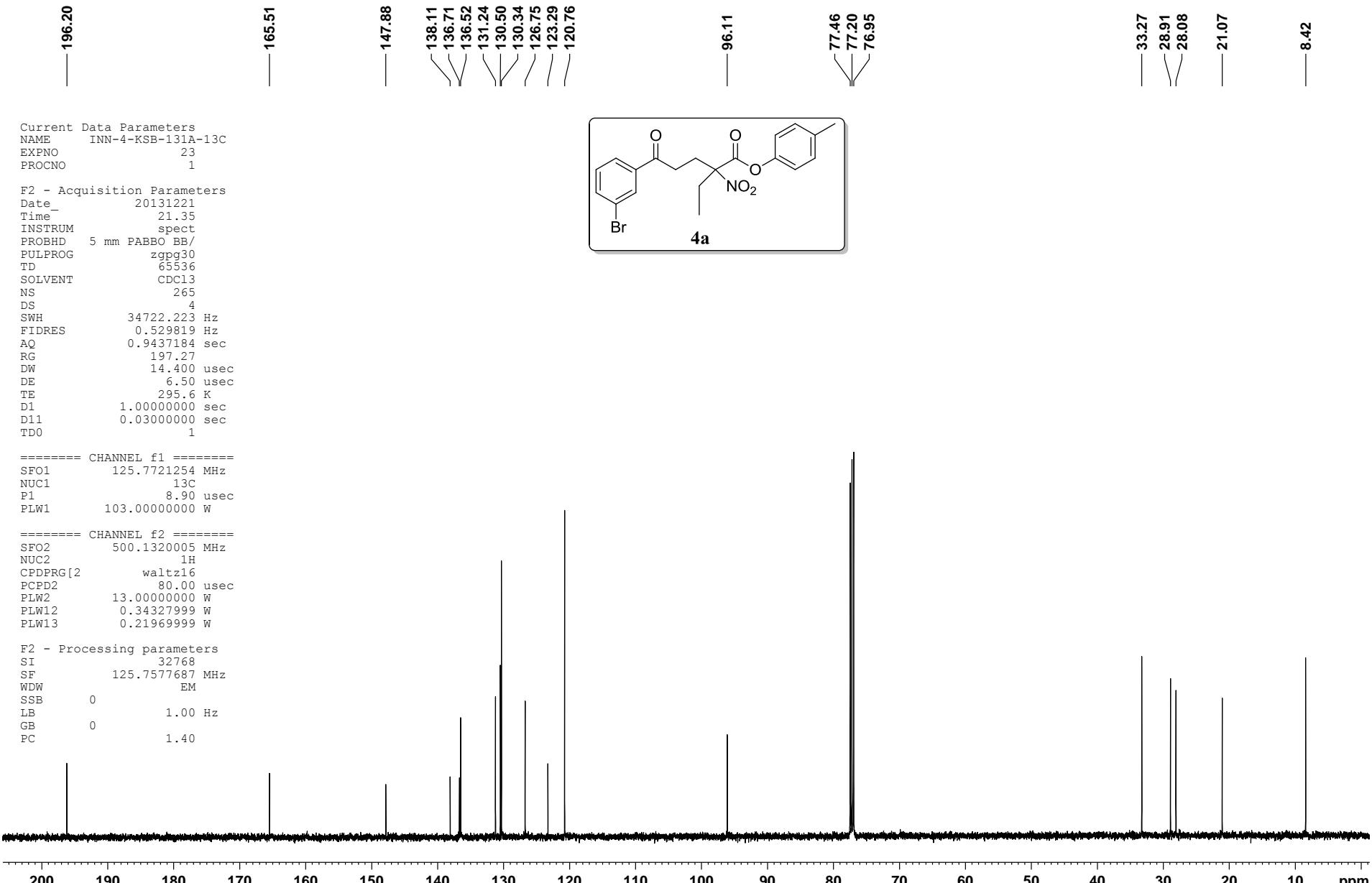
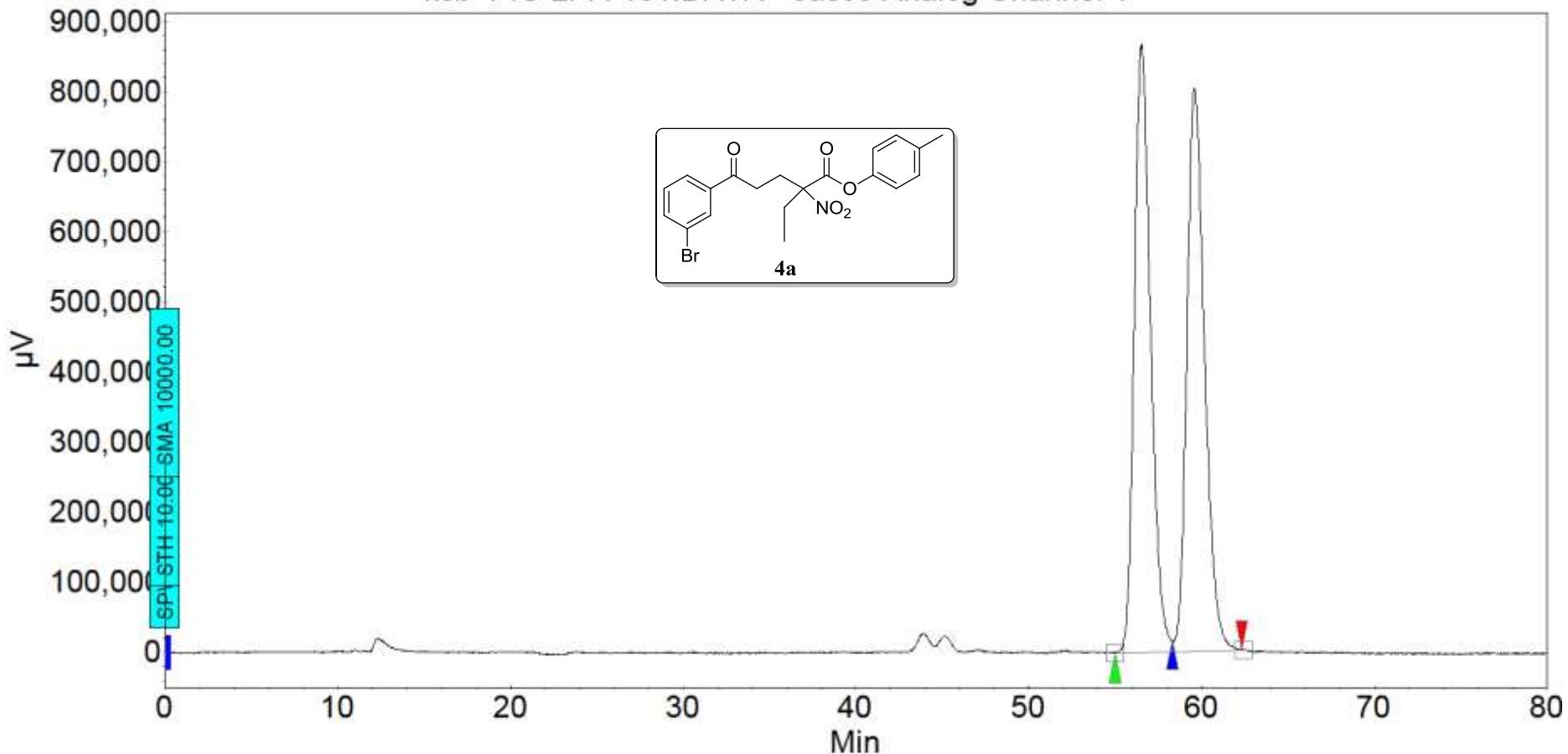


Fig S86.  $^{13}\text{C}$  NMR Spectrum of 4a

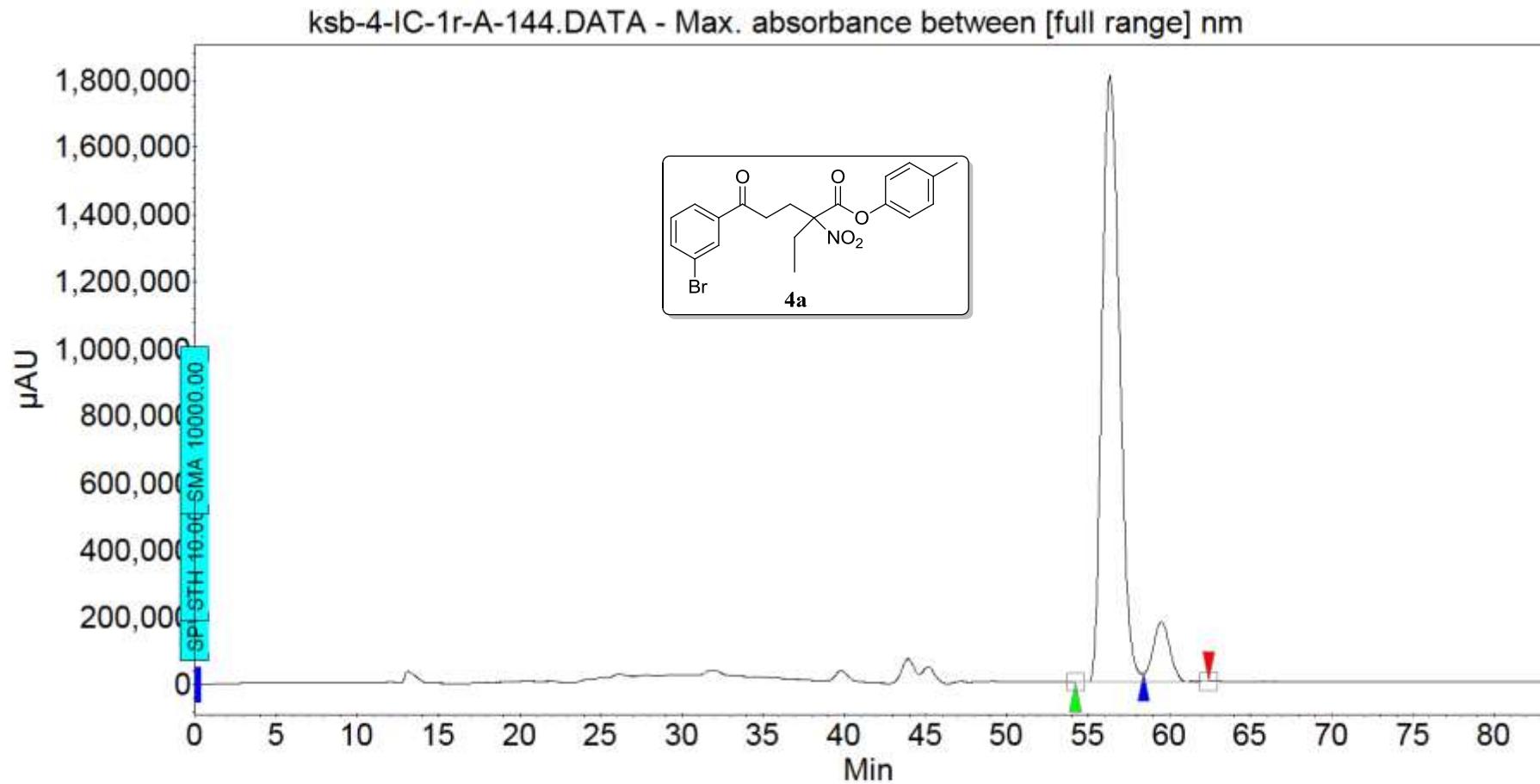
ksb-4-IC-2r-A-131.DATA - Jasco Analog Channel 1



**Peak results :**

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	56.550	49.78	867448.2	947733.9	49.785
2	UNKNOWN	59.575	50.22	802808.8	955933.7	50.215
Total			100.00	1670256.9	1903667.6	100.000

Fig S87. HPLC Profile of Racemic 4a



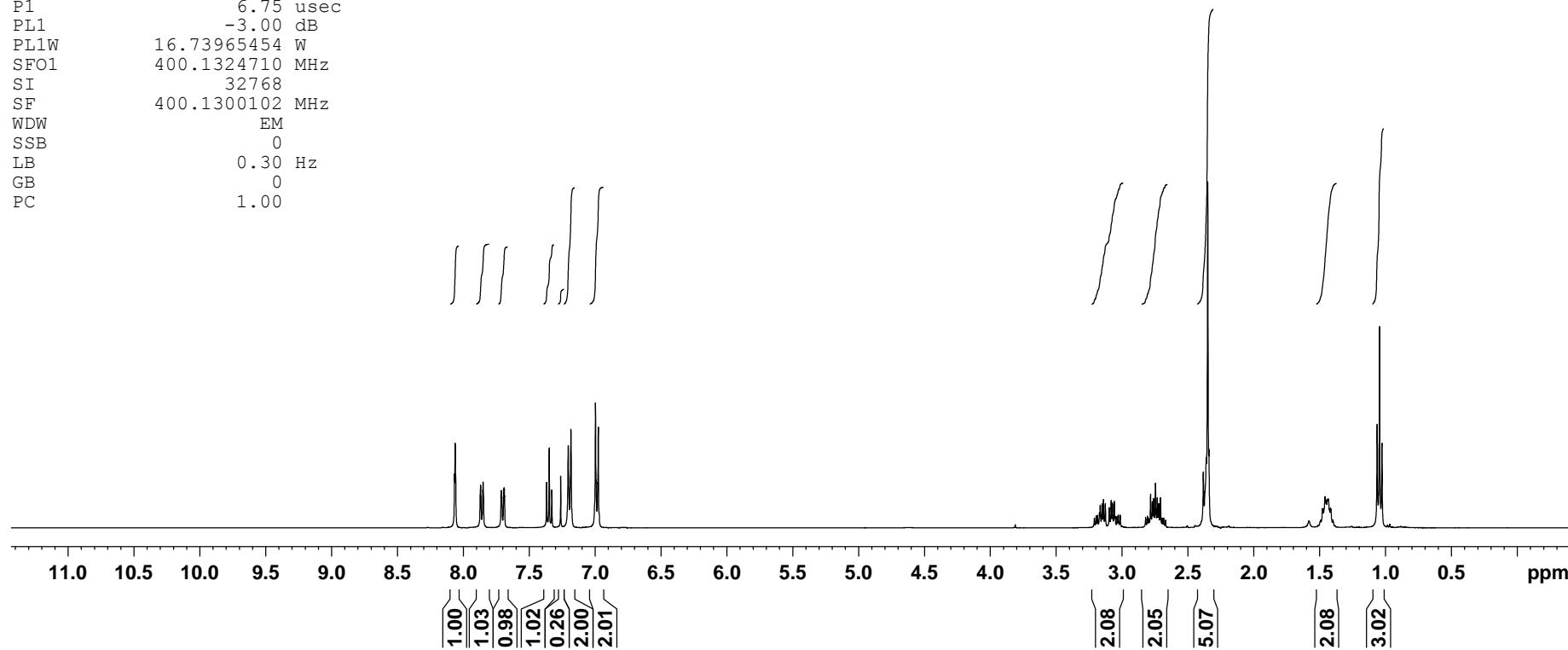
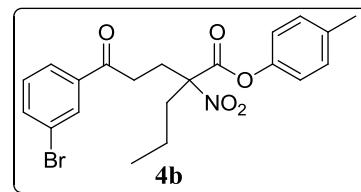
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	56.353	91.38	1804670.9	2247784.3	91.381
2	UNKNOWN	59.512	8.62	178883.2	212007.3	8.619
Total			100.00	1983554.1	2459791.6	100.000

**Fig S88. HPLC Profile of Enantioenriched 4a**

NAME INN-4-KSB-131B-1H  
 EXPNO 4  
 PROCNO 1  
 Date 20131217  
 Time 17.56  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 27  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 71.8  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.8 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 ======  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300102 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S89.** <sup>1</sup>H NMR Spectrum of **4b**

— 196.16

— 165.55

— 147.88

138.12  
136.66  
136.47  
131.20  
130.47  
130.31  
126.73  
123.25  
120.73

— 95.63

77.45  
77.20  
76.94

— 37.51

— 33.31

— 28.61

— 21.03

— 17.41

— 14.18

Current Data Parameters  
NAME INN-4-KSB-131B-13C  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131220  
Time\_ 11.52  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 57  
DS 4  
SWH 34722.223 Hz  
FIDRES 0.529819 Hz  
AQ 0.9437184 sec  
RG 197.27  
DW 14.400 usec  
DE 6.50 usec  
TE 298.3 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 125.7721254 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 125.7577723 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

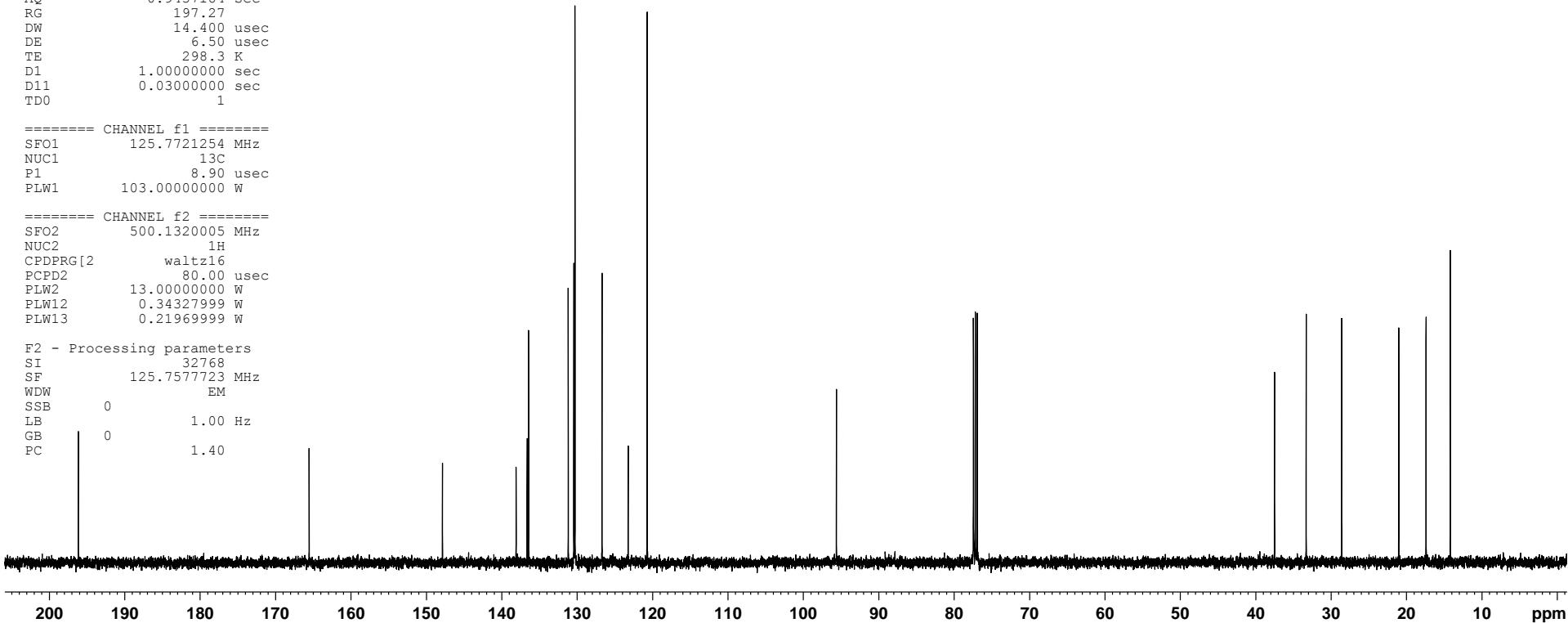
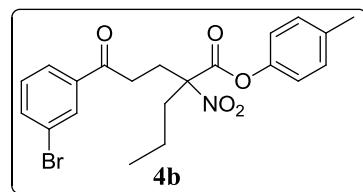
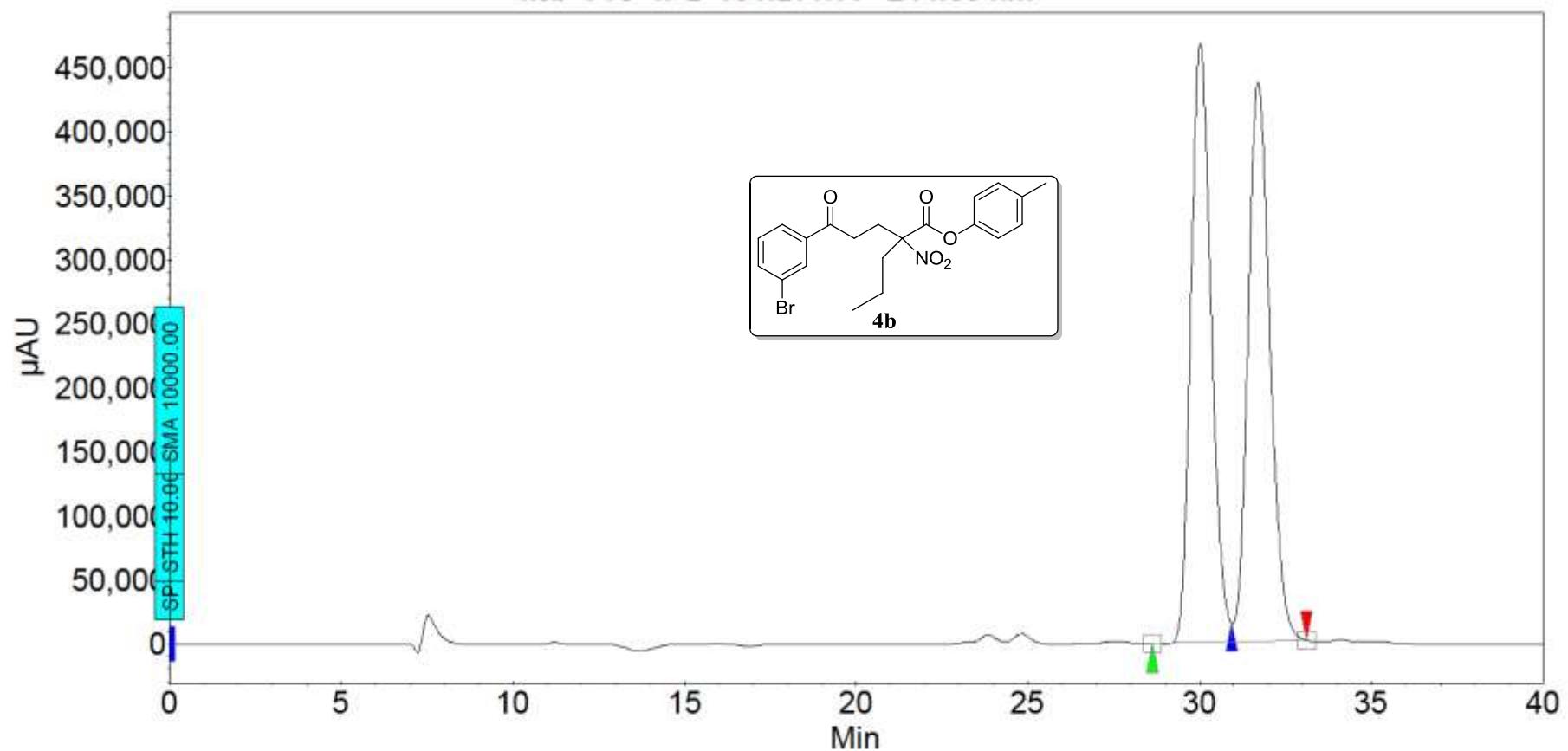


Fig S90. <sup>13</sup>C NMR Spectrum of 4b

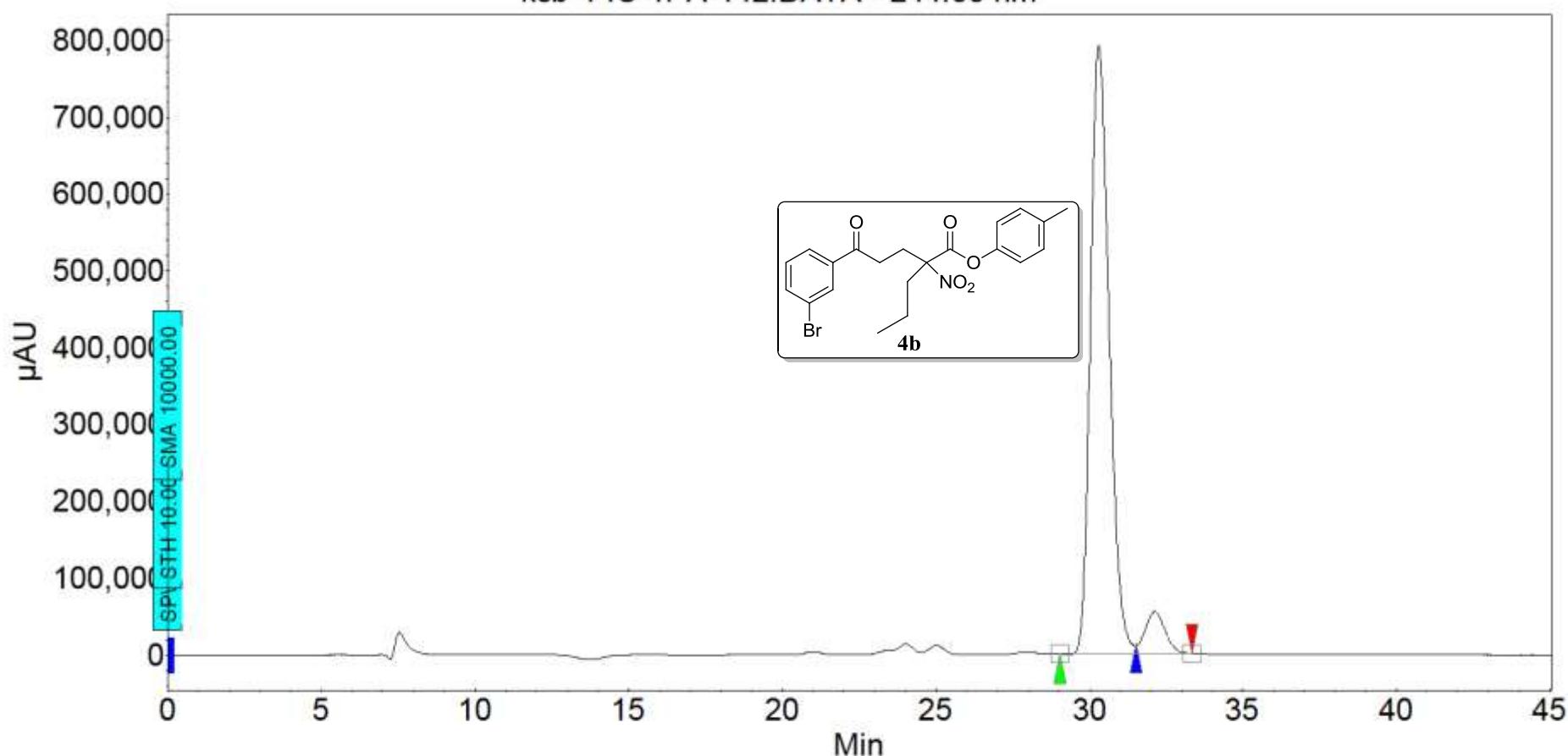


### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	30.011	49.77	467882.9	320224.3	49.770
2	UNKNOWN	31.691	50.23	436893.3	323189.4	50.230
Total			100.00	904776.2	643413.7	100.000

**Fig S91.** HPLC Profile of Racemic 4b

ksb-4-IC-1r-A-142.DATA - 244.00 nm

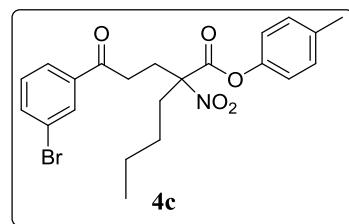


### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	30.304	93.08	792214.3	559612.8	93.079
2	UNKNOWN	32.118	6.92	54715.1	41610.5	6.921
Total			100.00	846929.4	601223.3	100.000

Fig S92. HPLC Profile of Enantioenriched 4b

NAME INN-4-KSB-131C-1H  
 EXPNO 19  
 PROCNO 1  
 Date 20131209  
 Time 21.04  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 10  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 295.5 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 400.1300102 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

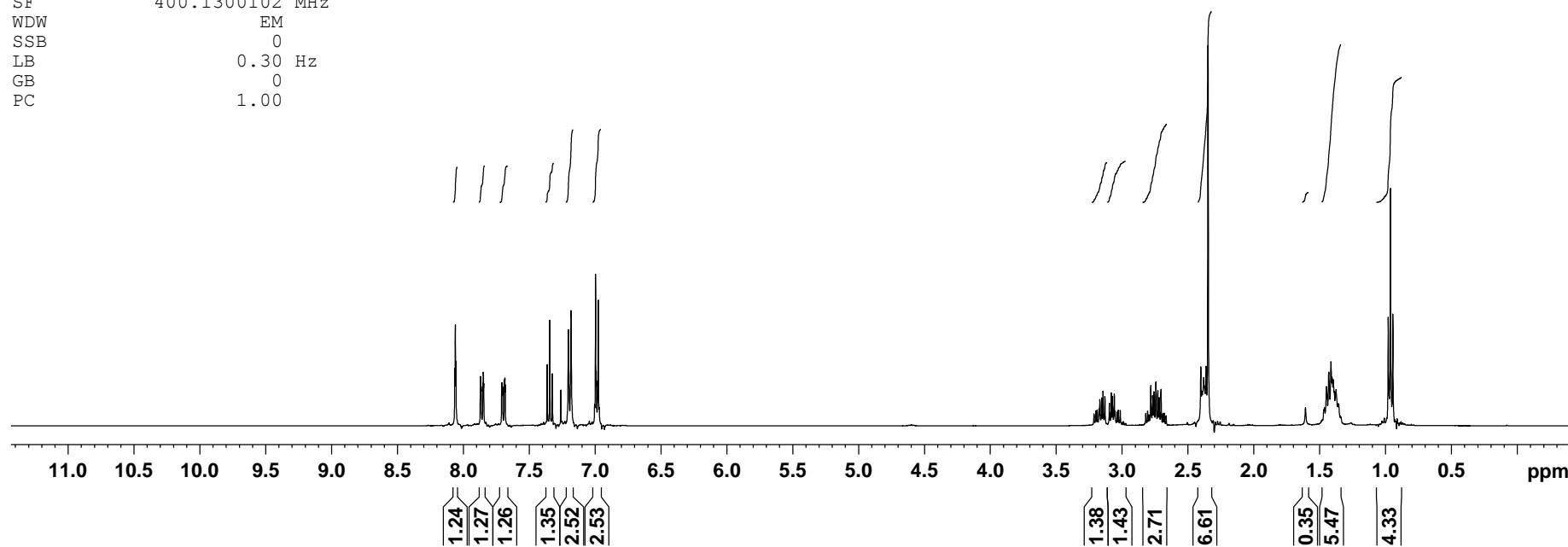


Fig S93. <sup>1</sup>H NMR Spectrum of 4c

— 196.17

— 165.55

— 147.90

138.14  
136.65  
136.46  
131.20  
130.46  
130.31  
126.73  
123.26  
120.73

— 95.70

77.45  
77.20  
76.94

35.22  
33.31  
28.59  
25.93  
22.74  
21.03

— 13.89

Current Data Parameters  
NAME inn-4-ksb-131c-13C  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20131212  
Time 11.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 100  
DS 4  
SWH 34722.223 Hz  
FIDRES 0.529819 Hz  
AQ 0.9437184 sec  
RG 197.27  
DW 14.400 usec  
DE 6.50 usec  
TE 299.4 K  
D1 1.0000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 125.7721254 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 125.7577720 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

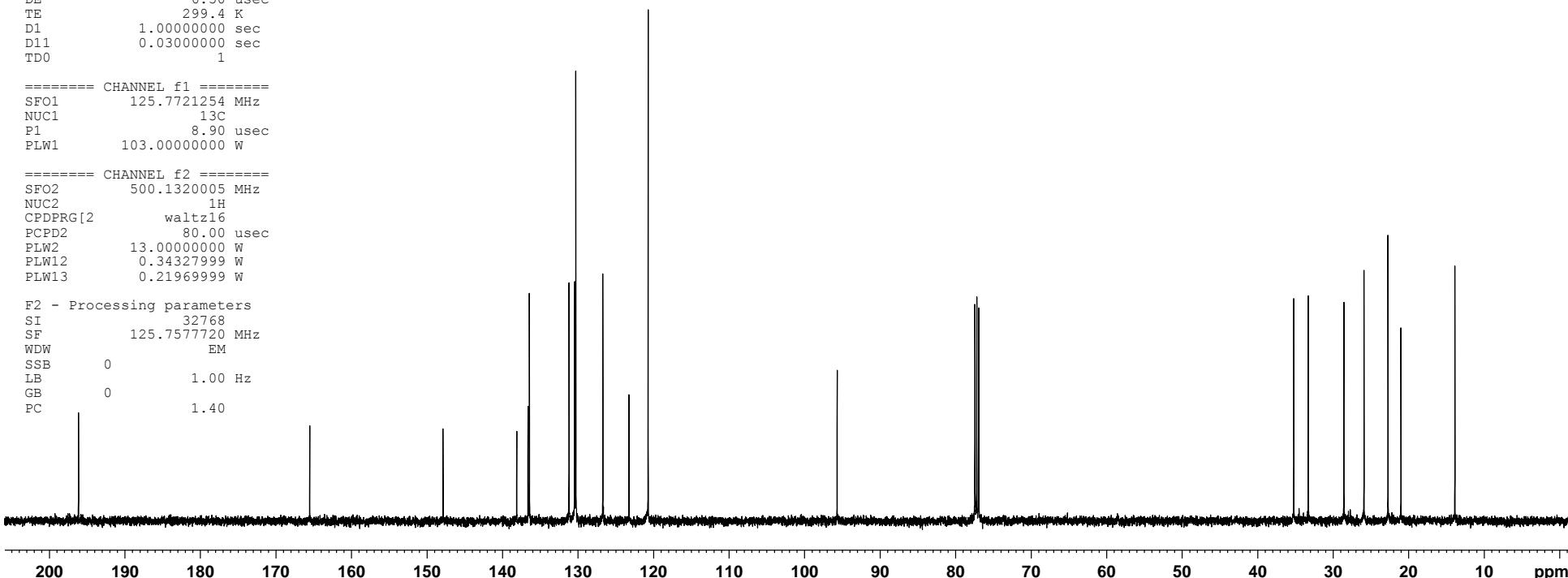
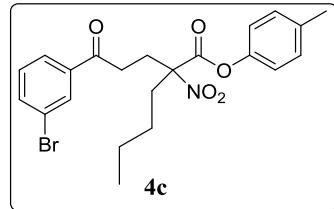


Fig S94. <sup>13</sup>C NMR Spectrum of 4c

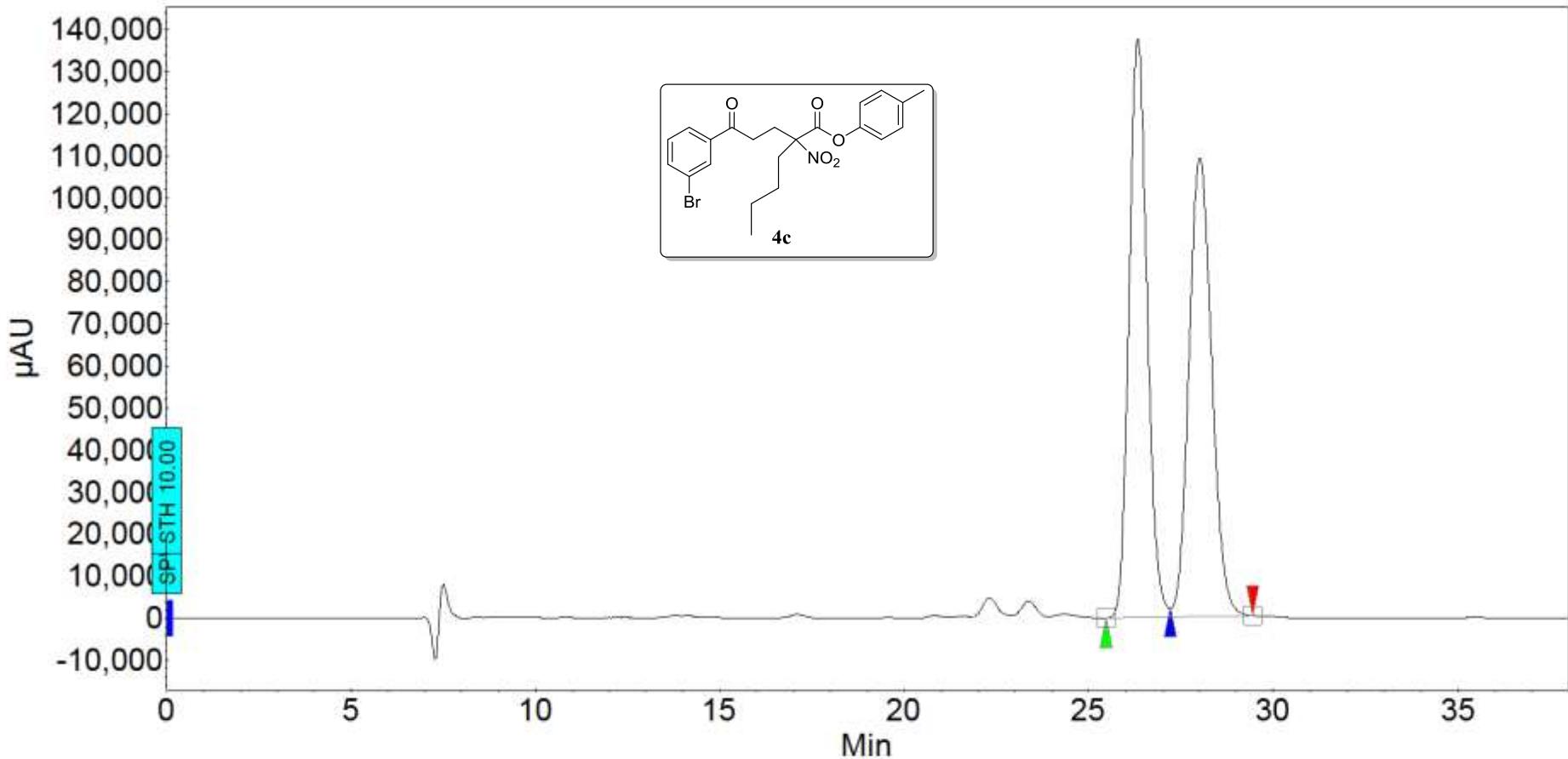
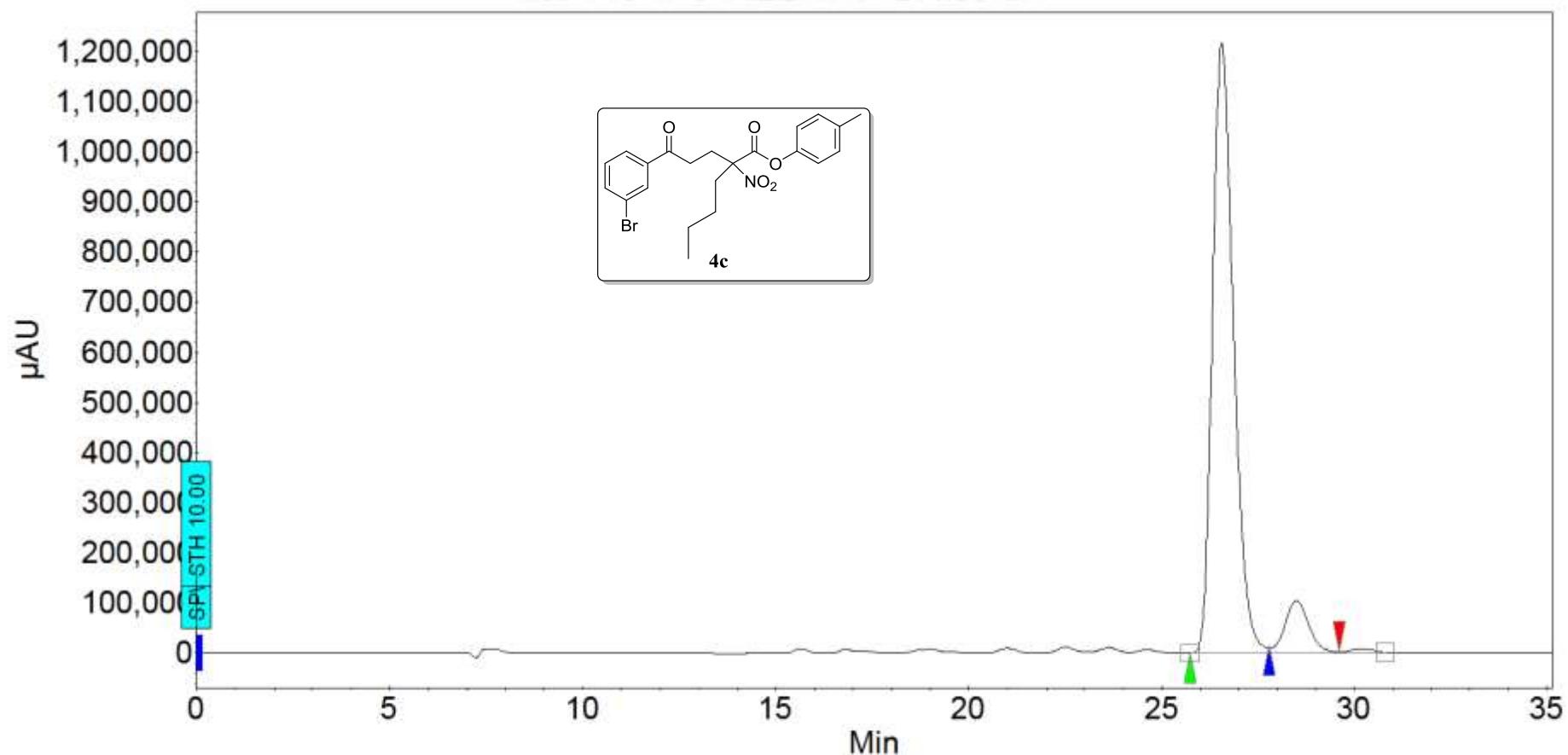


Fig S95. HPLC Profile of Racemic 4c



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area [%]
1	UNKNOWN	26.558	91.29	1216349.6	766813.9	91.285
2	UNKNOWN	28.491	8.71	103577.6	73206.7	8.715
Total			100.00	1319927.2	840020.6	100.000

**Fig S96. HPLC Profile of Enantioenriched 4c**

NAME INN-4-KSB-133b-1H  
 EXPNO 1  
 PROCNO 1  
 Date 20131211  
 Time 22.07  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 25  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 71.8  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 297.6 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 ======  
 NUC1 1H  
 P1 6.75 usec  
 PL1 -3.00 dB  
 PL1W 16.73965454 W  
 SF01 400.1324710 MHz  
 SI 32768  
 SF 400.1300102 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

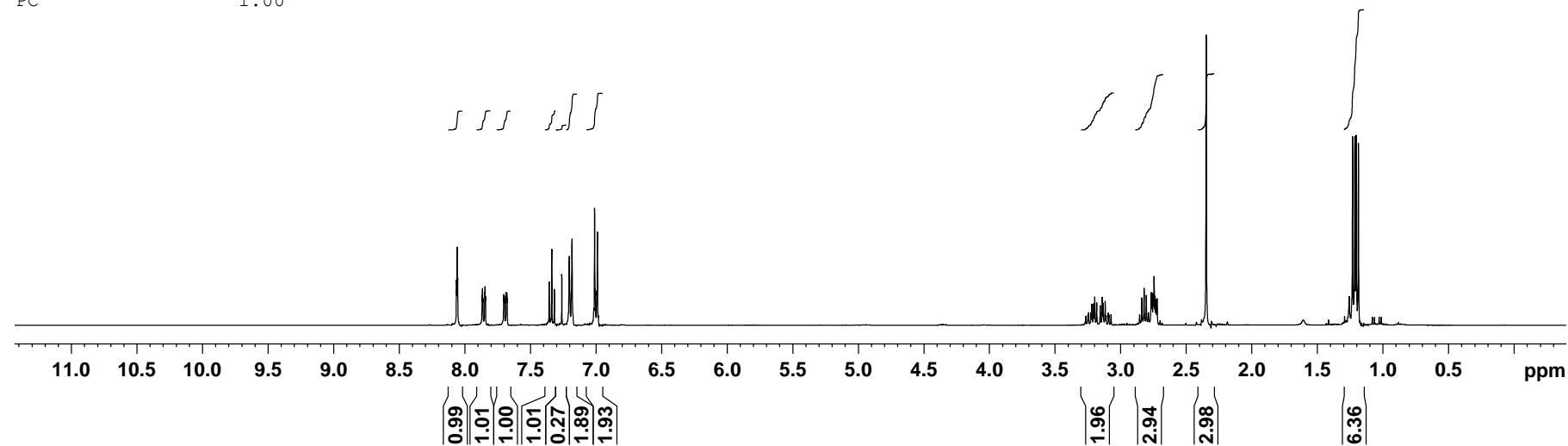
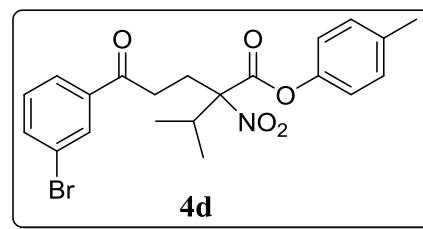


Fig S97.  $^1\text{H}$  NMR Spectrum of 4d

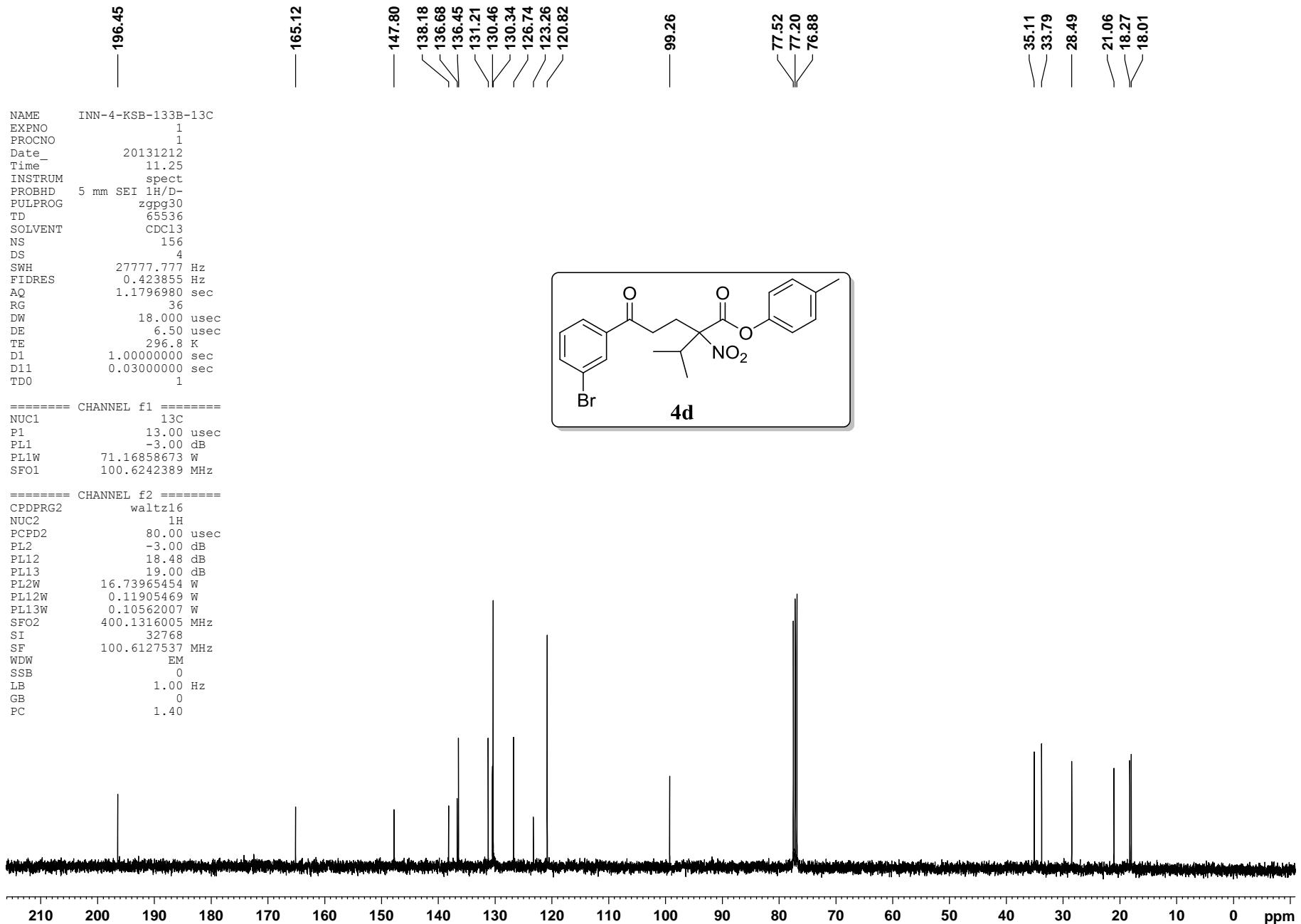
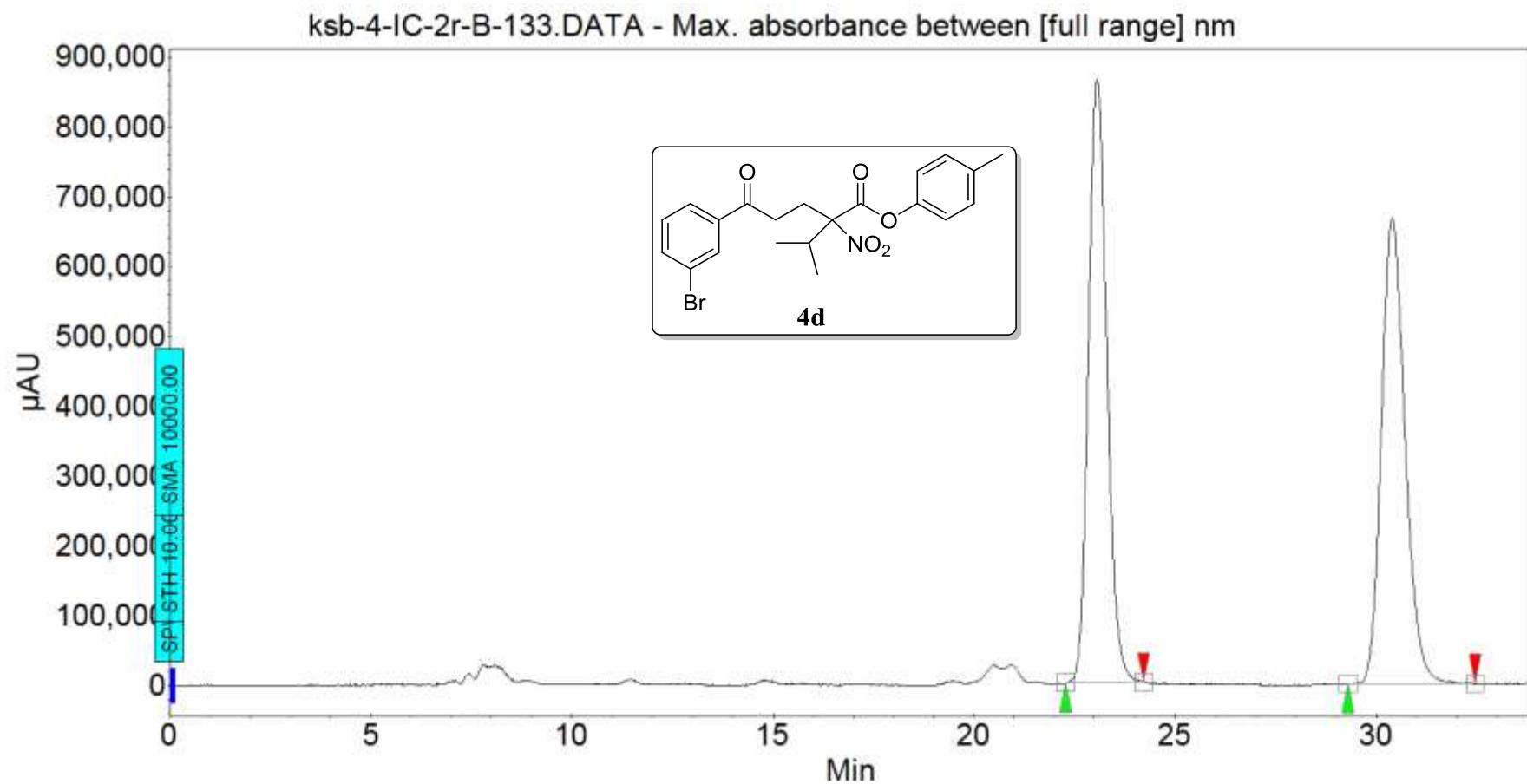


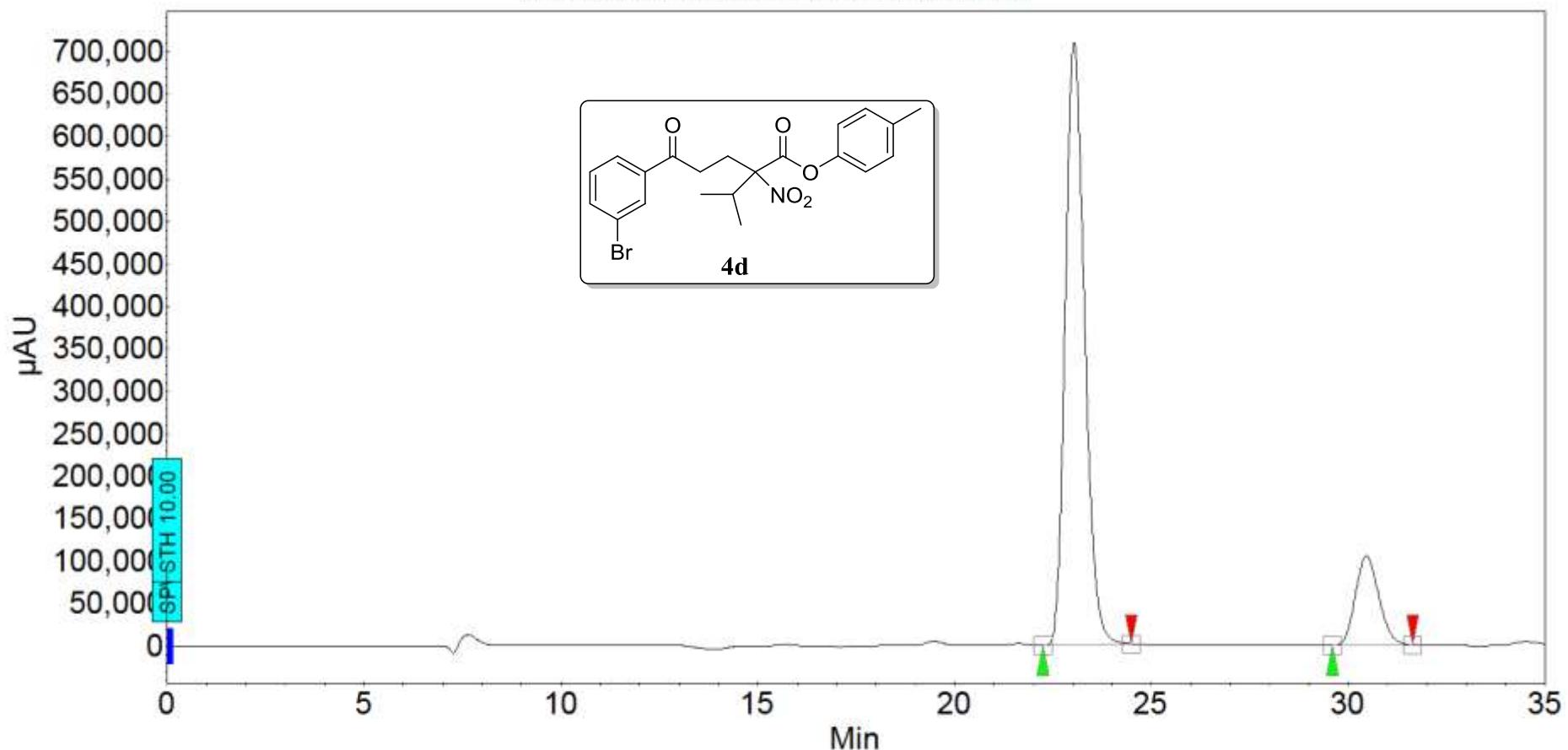
Fig S98.  $^{13}\text{C}$  NMR Spectrum of 4d



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	23.052	50.38	863575.7	461453.8	50.382
1	UNKNOWN	30.398	49.62	666965.3	454454.6	49.618
Total			100.00	1530540.9	915908.4	100.000

**Fig S99. HPLC Profile of Racemic 4d**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
2	UNKNOWN	23.038	84.74	710393.2	405066.3	84.742
1	UNKNOWN	30.464	15.26	105070.5	72932.9	15.258
Total			100.00	815463.7	477999.2	100.000

**Fig S100. HPLC Profile of Enantioenriched 4d**

Current Data Parameters  
NAME INN-2-NSS-125-1H  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20160509  
Time 11.14  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 228  
DW 60.800 usec  
DE 6.50 usec  
TE 294.8 K  
D1 1.0000000 sec  
TD0 1

## ===== CHANNEL f1 =====

NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

## F2 - Processing parameters

SI 32768  
SF 400.1300103 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

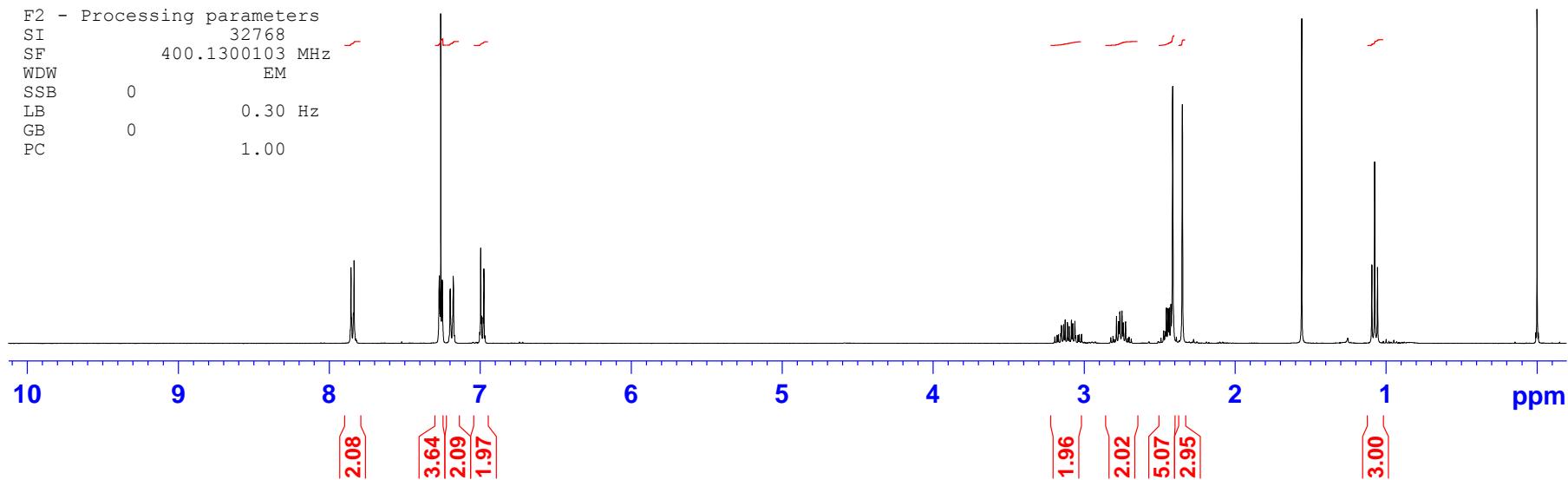
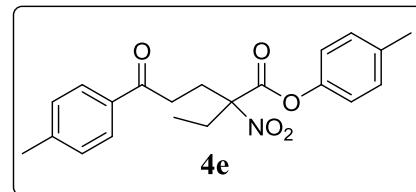
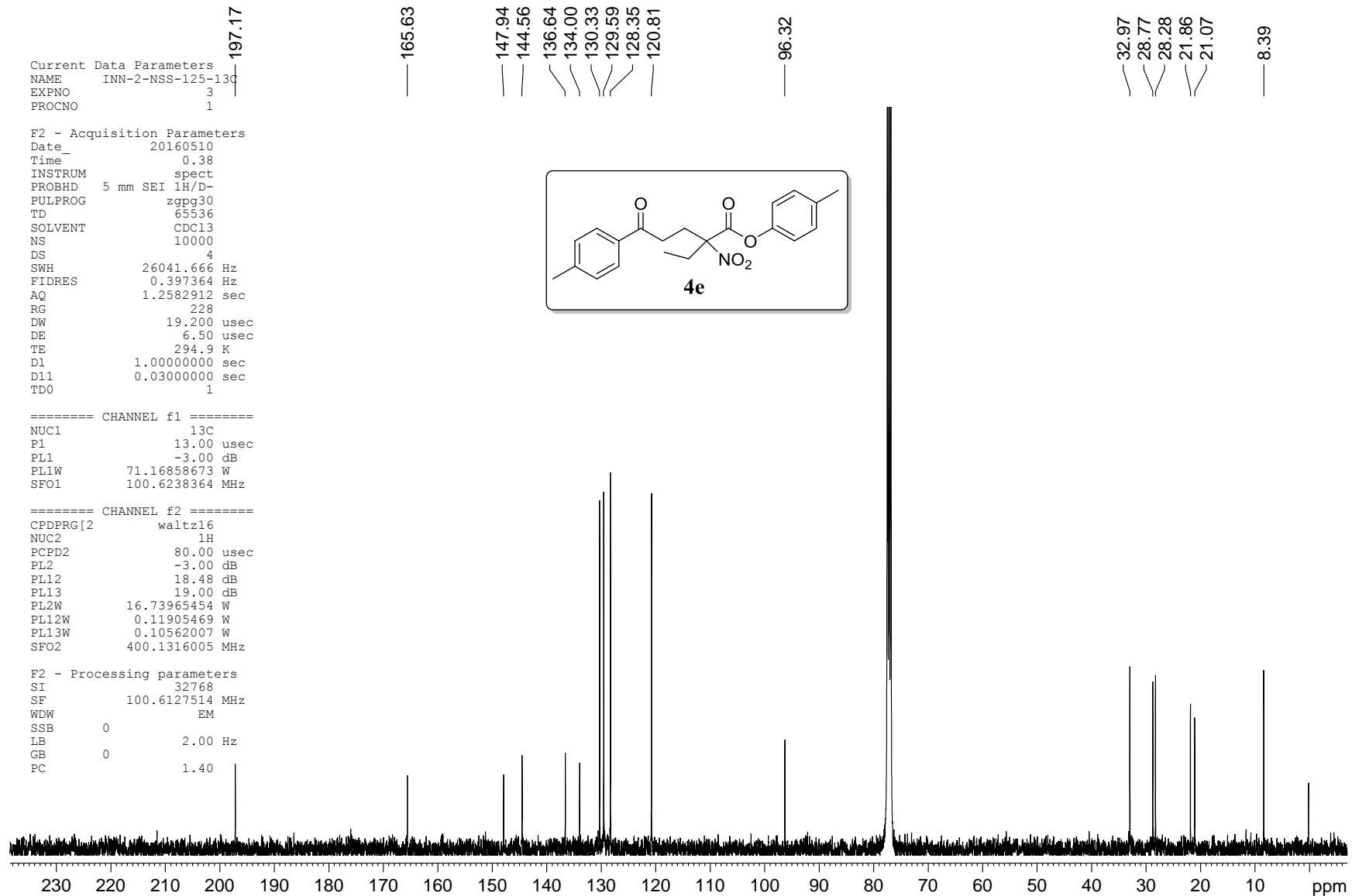
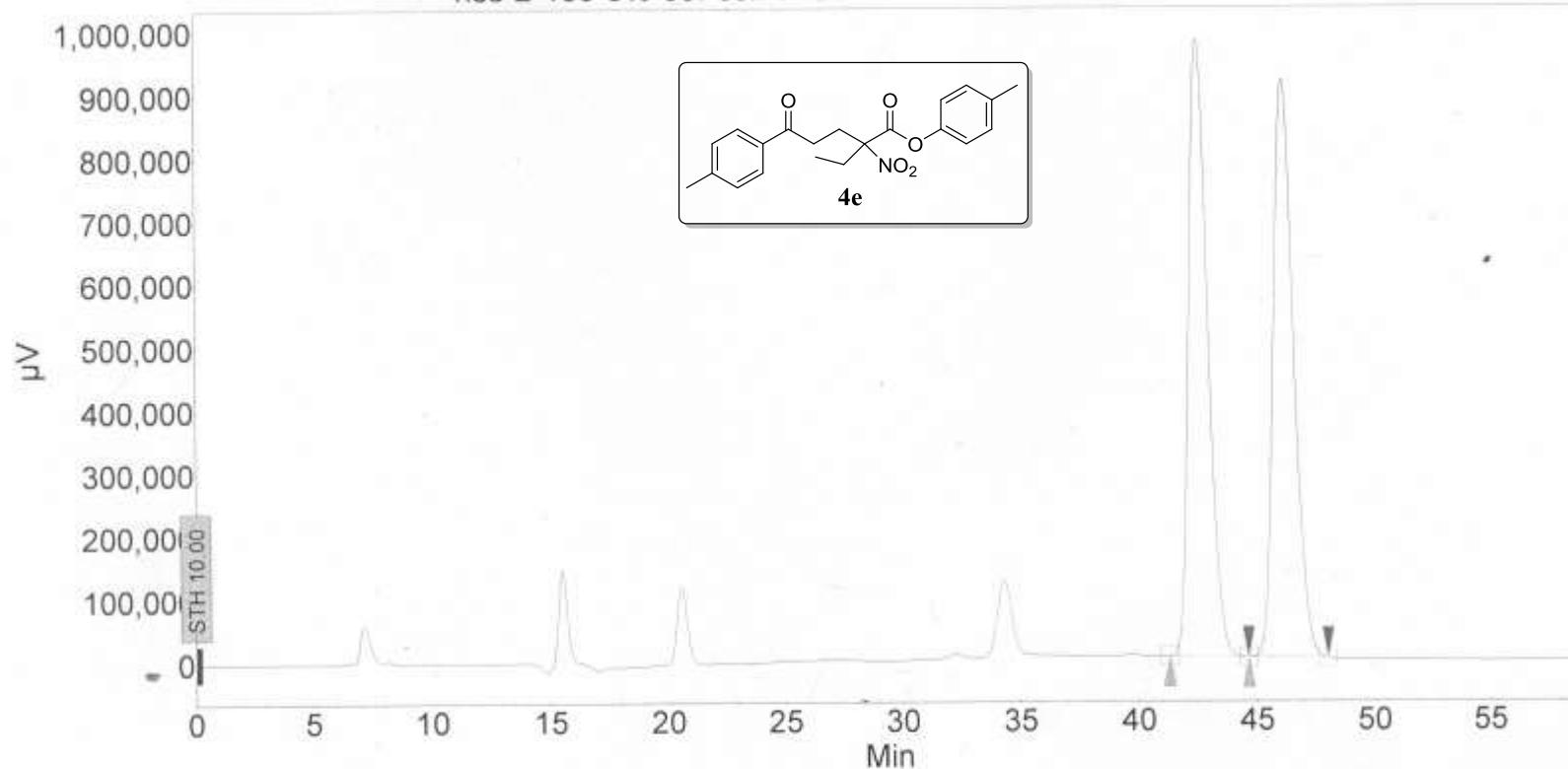


Fig S101. <sup>1</sup>H NMR Spectrum of 4e

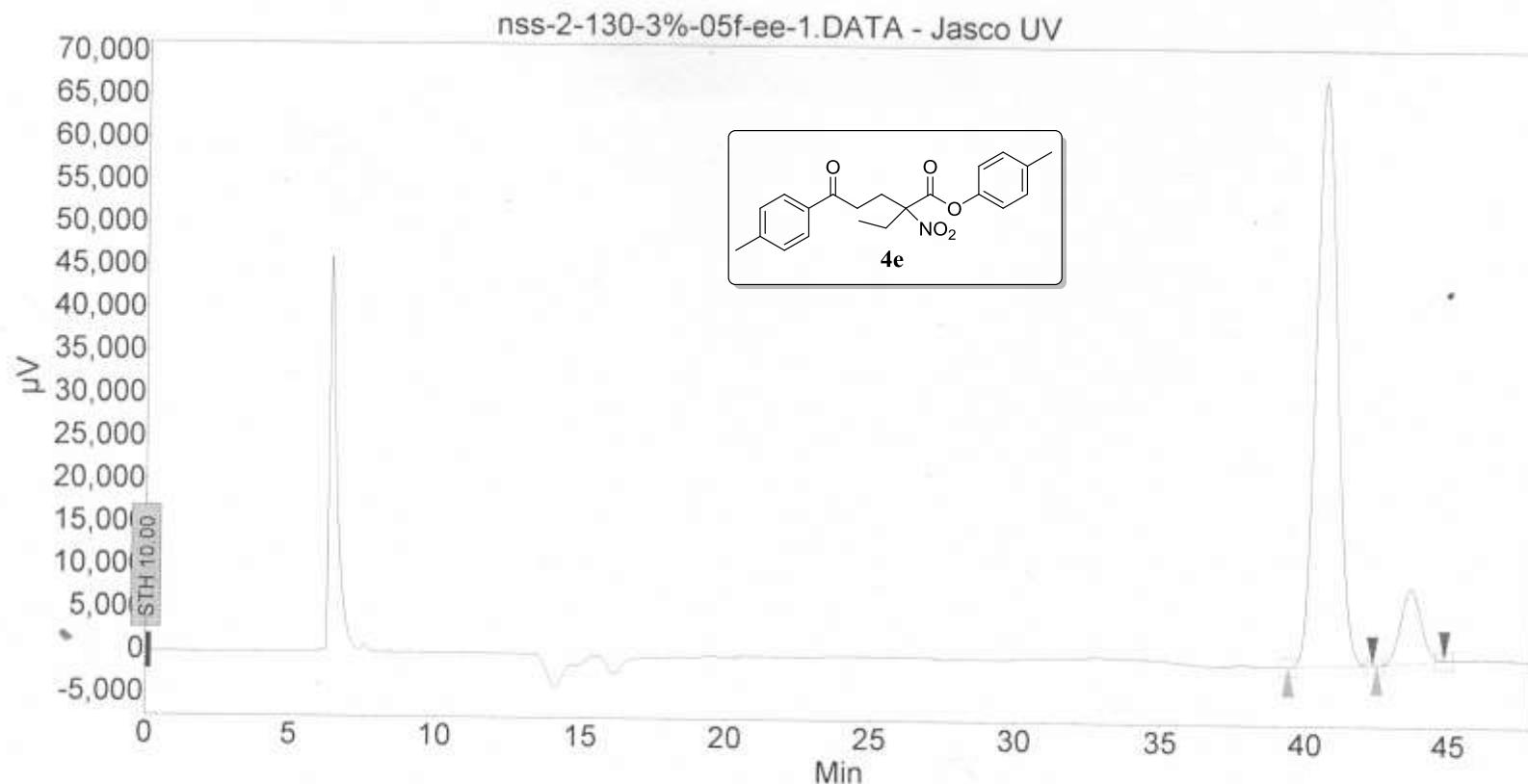




### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	42.477	50.03	977399.7	913277.2	50.025
2	UNKNOWN	46.090	49.97	913869.1	912359.2	49.975
Total			100.00	1891268.8	1825636.4	100.000

**Fig S103. HPLC Profile of Racemic 4e**



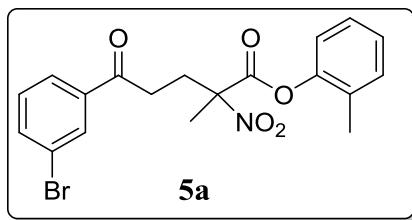
#### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area % [%]
1	UNKNOWN	40.558	88.93	68132.8	57605.1	88.934
2	UNKNOWN	43.625	11.07	8657.9	7168.1	11.066
Total			100.00	76790.7	64773.1	100.000

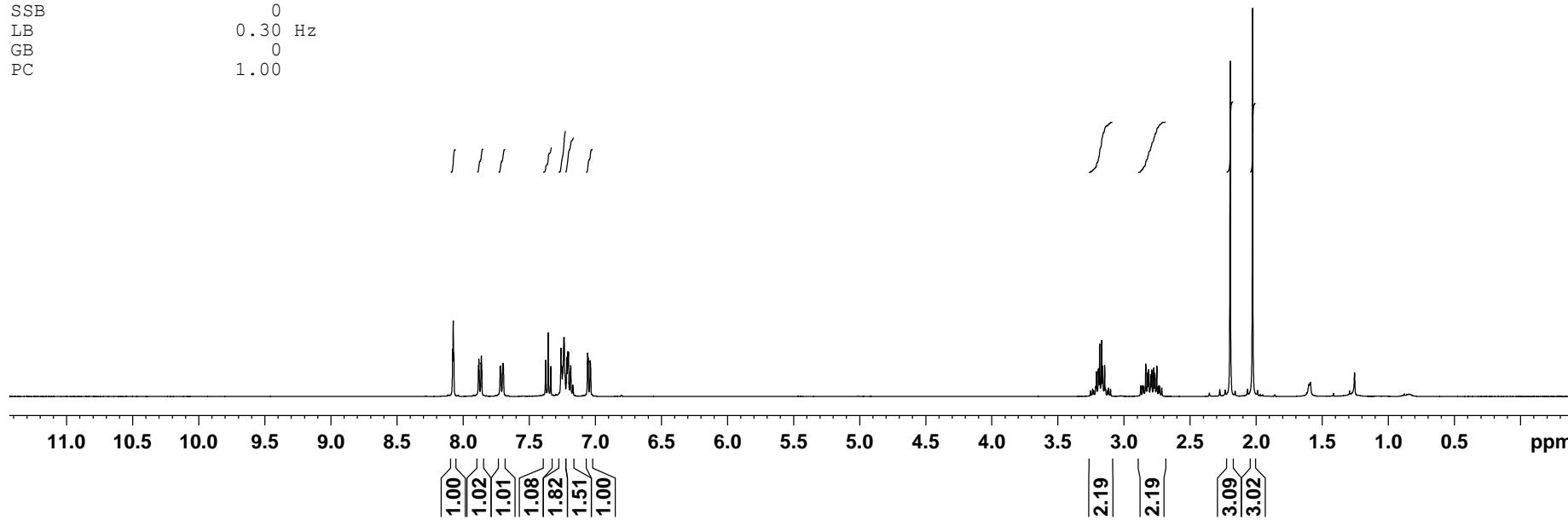
**Fig S104. HPLC Profile of Enantioenriched 4e**

NAME INN-4-KSB-71A-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130523  
 Time 19.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 12  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.1 K  
 D1 1.00000000 sec  
 TDO 1

**5a**



===== 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 105.**  $^1\text{H}$  NMR Spectrum of **5a**

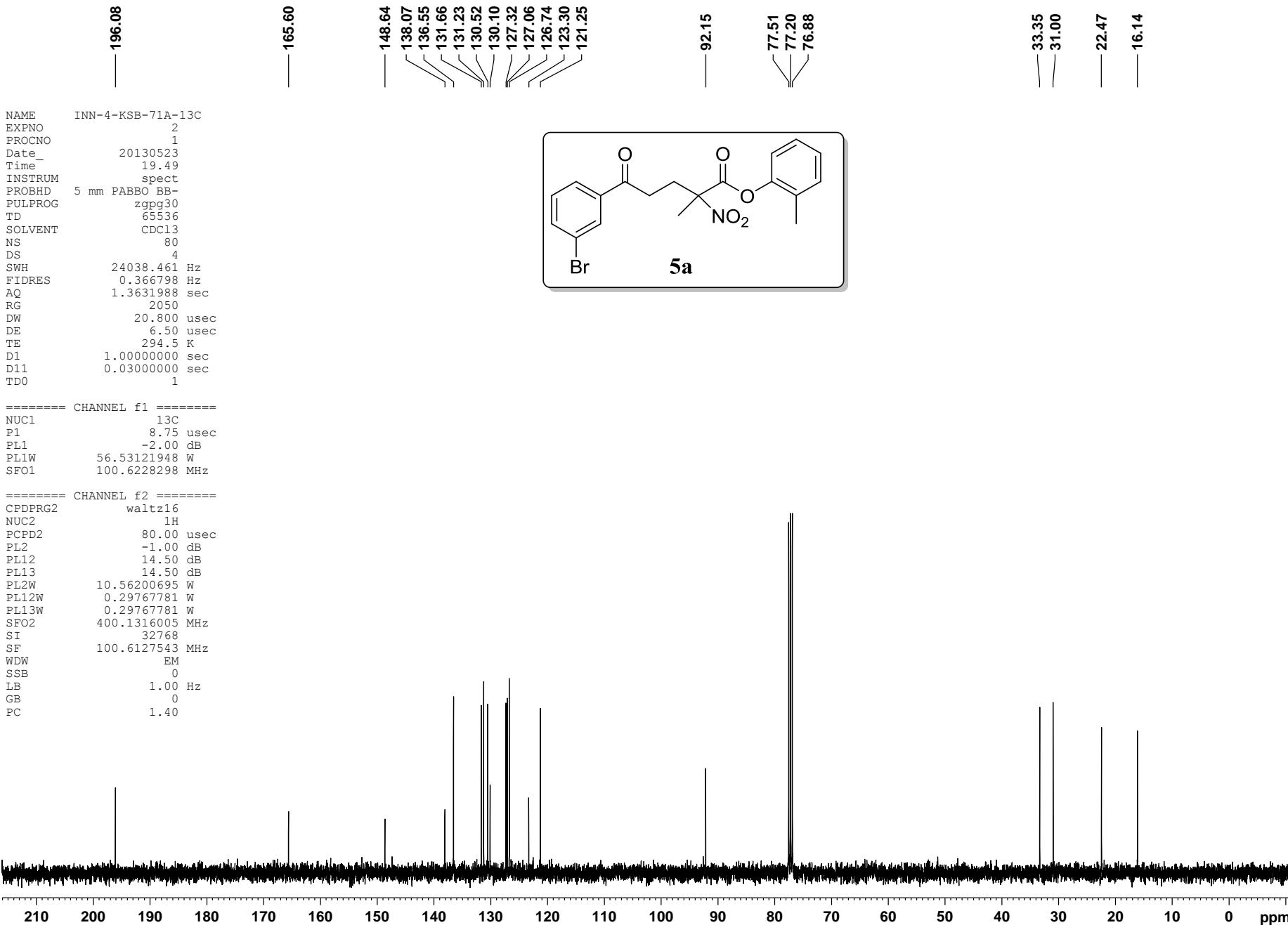
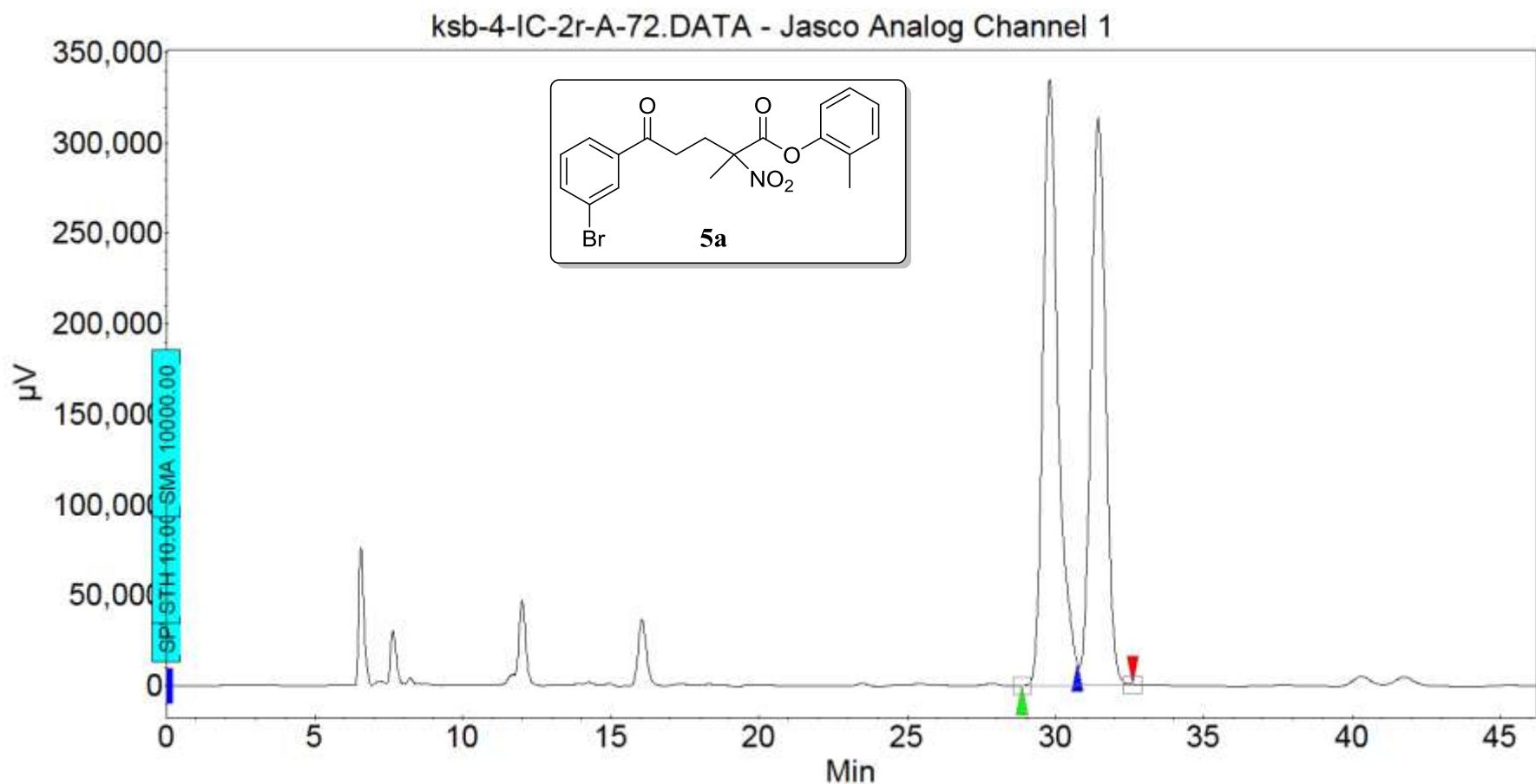


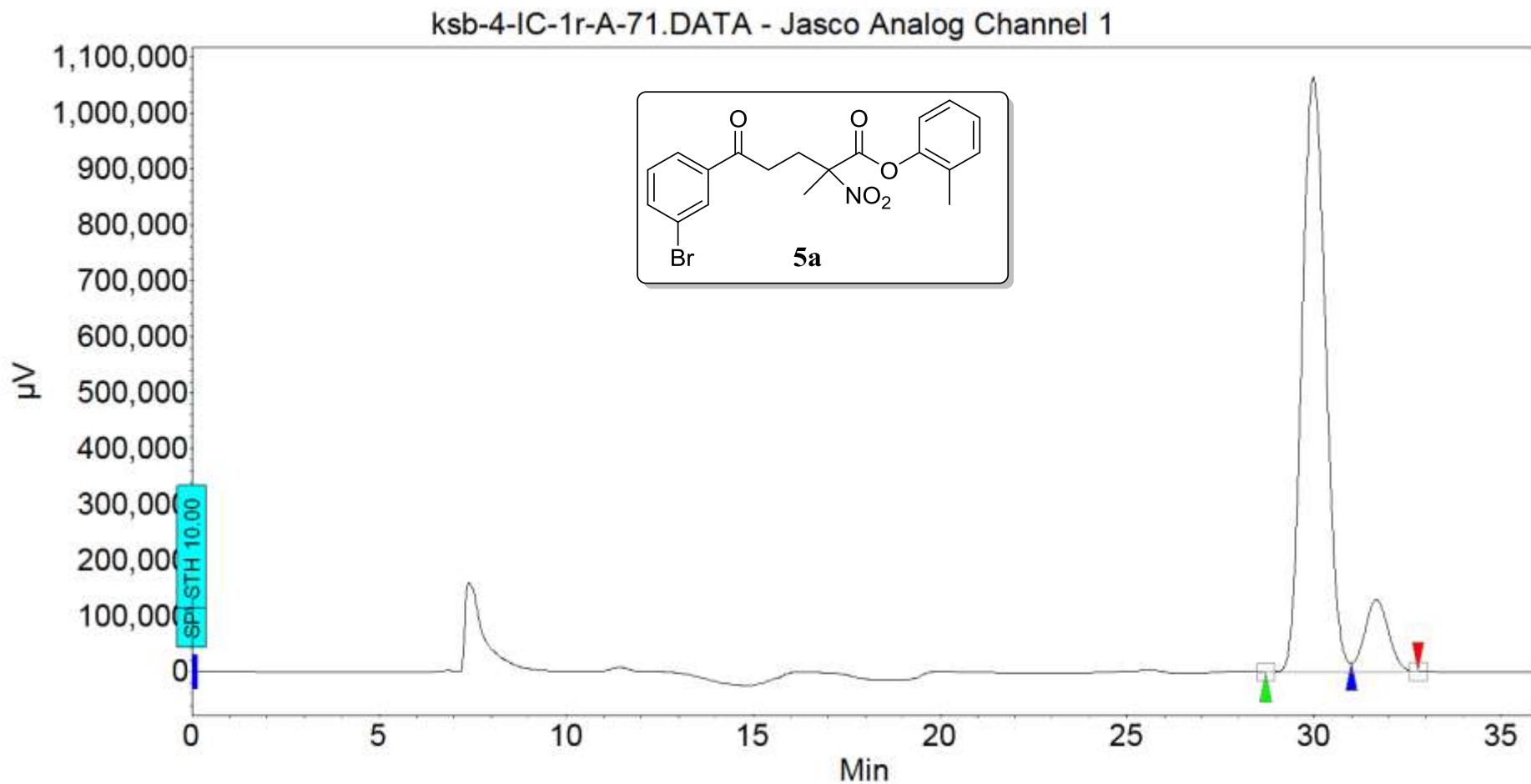
Fig 106.  $^{13}\text{C}$  NMR Spectrum of 5a



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu\text{V}$ ]	Area [ $\mu\text{V}.\text{Min}$ ]	Area % [%]
1	UNKNOWN	29.800	52.99	334759.8	201076.9	52.988
2	UNKNOWN	31.433	47.01	314084.5	178402.4	47.012
Total			100.00	648844.4	379479.3	100.000

**Fig S107. HPLC Profile of Racemic 5a**



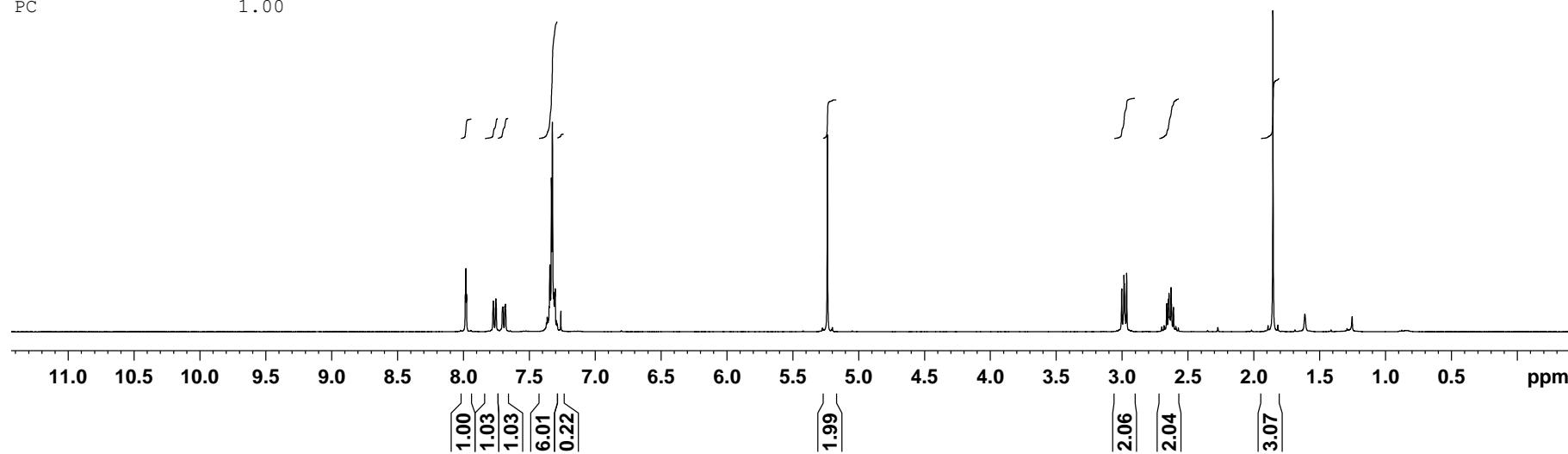
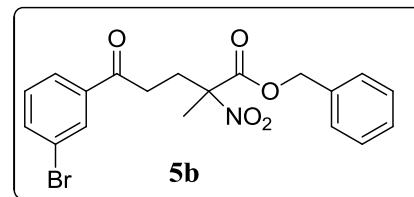
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [ $\mu$ V]	Area [ $\mu$ V.Min]	Area % [%]
1	UNKNOWN	29.983	89.09	1064209.3	777304.7	89.086
2	UNKNOWN	31.667	10.91	128593.8	95229.8	10.914
Total			100.00	1192803.2	872534.5	100.000

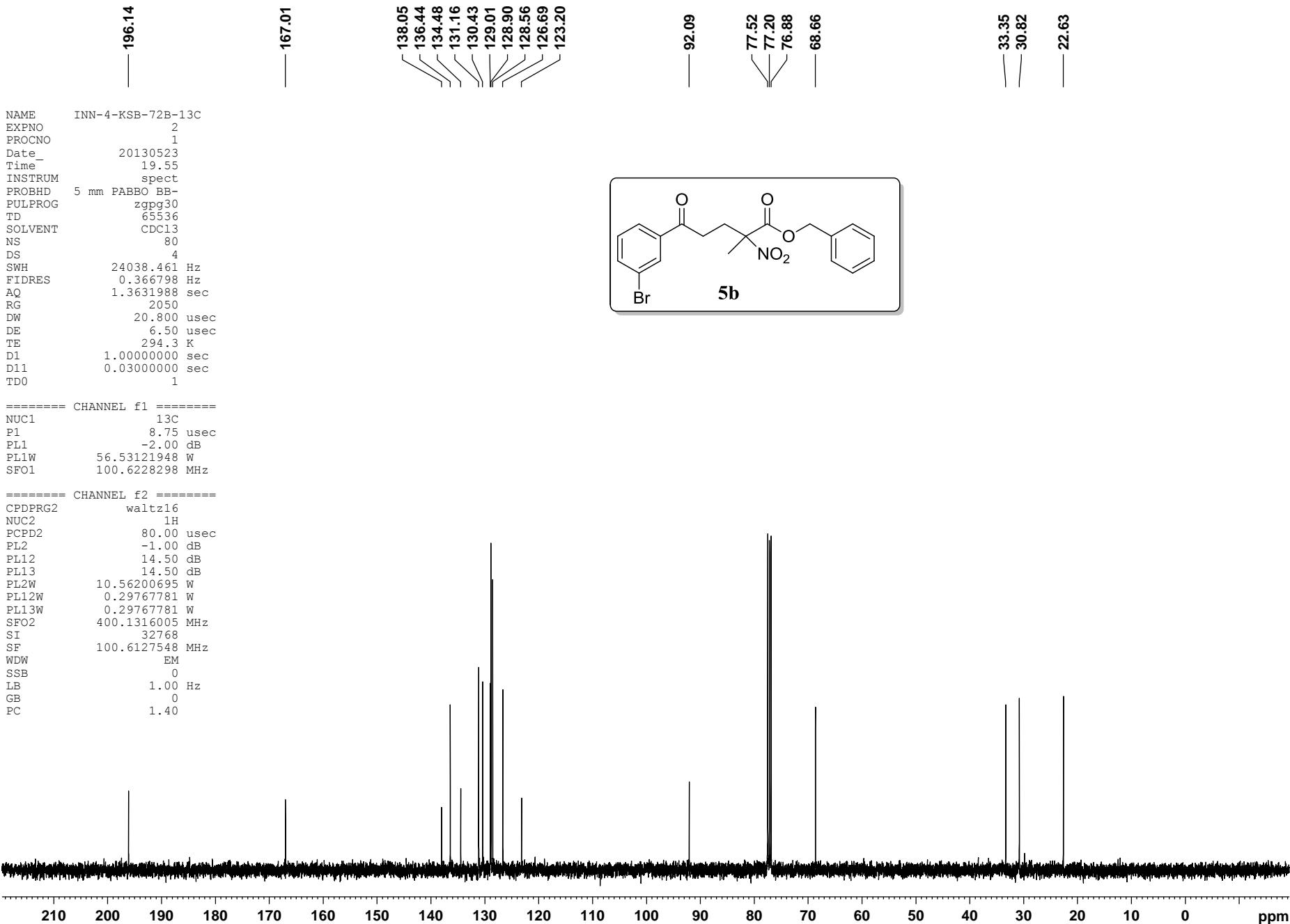
**Fig S108. HPLC Profile of Enantioenriched 5a**

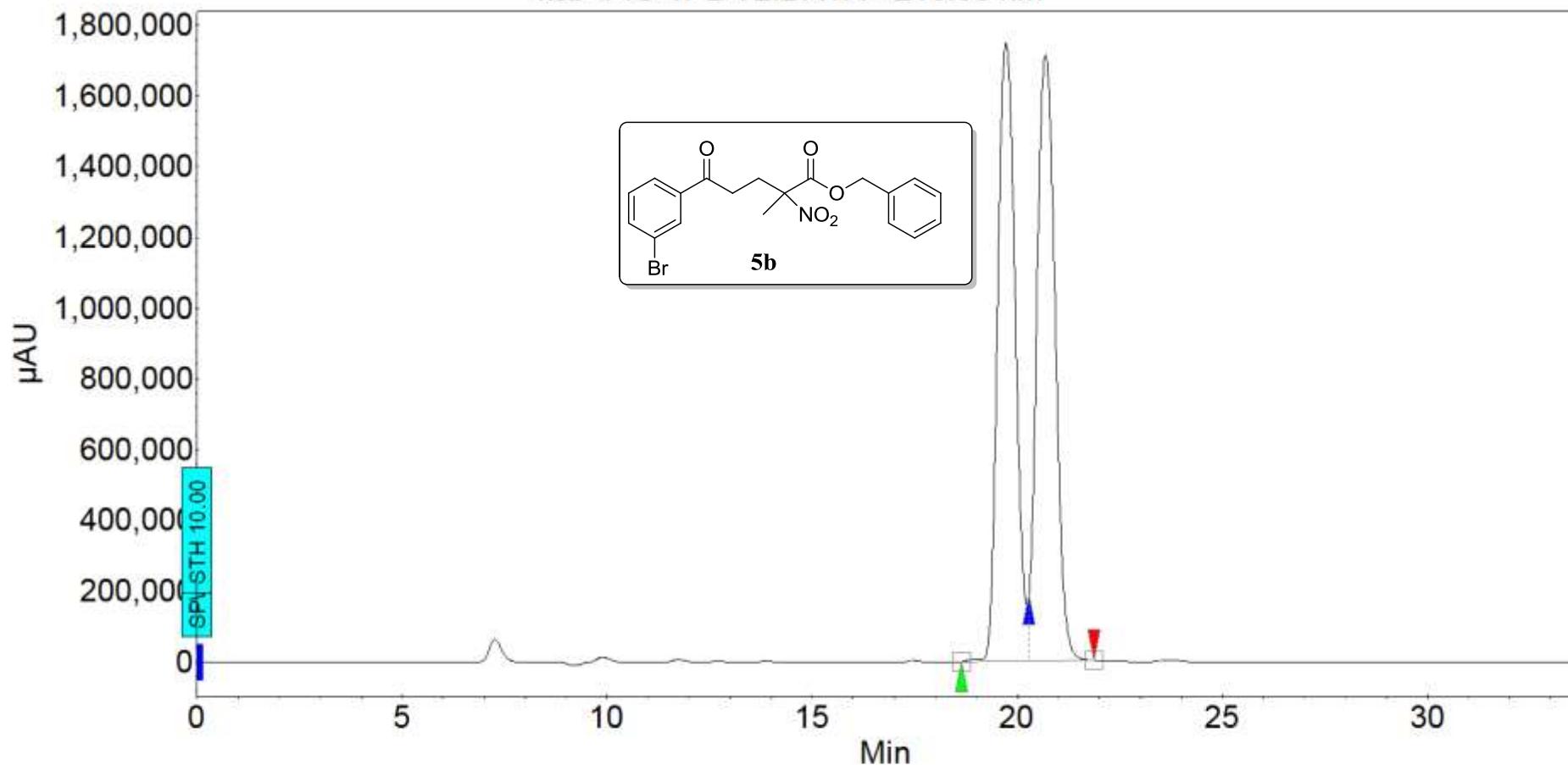
NAME INN-4-KSB-72B-1H  
 EXPNO 2  
 PROCNO 1  
 Date\_ 20130523  
 Time 19.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 4  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 32  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 294.2 K  
 D1 1.00000000 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 S109.**  $^1\text{H}$  NMR Spectrum of **5b**



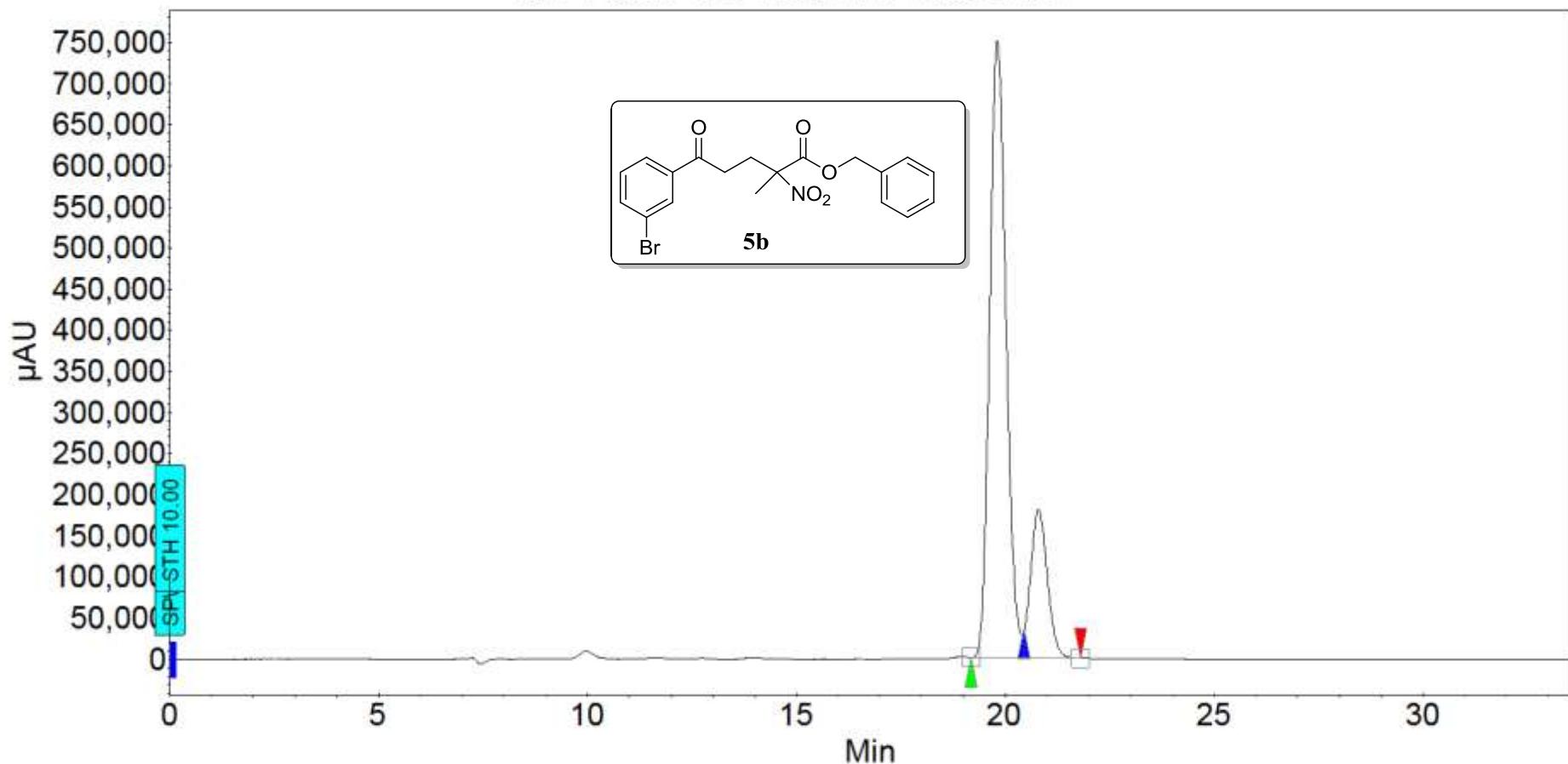


### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU·Min]	Area % [%]
1	UNKNOWN	19.719	49.80	1748879.0	876125.2	49.798
2	UNKNOWN	20.692	50.20	1713630.6	883230.2	50.202
Total			100.00	3462509.5	1759355.5	100.000

Fig S111. HPLC Profile of Racemic 5b

ksb-4-ODH-1r-B-71.DATA - 216.00 nm

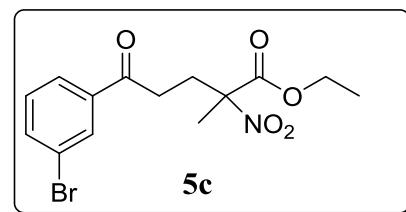


### Peak results :

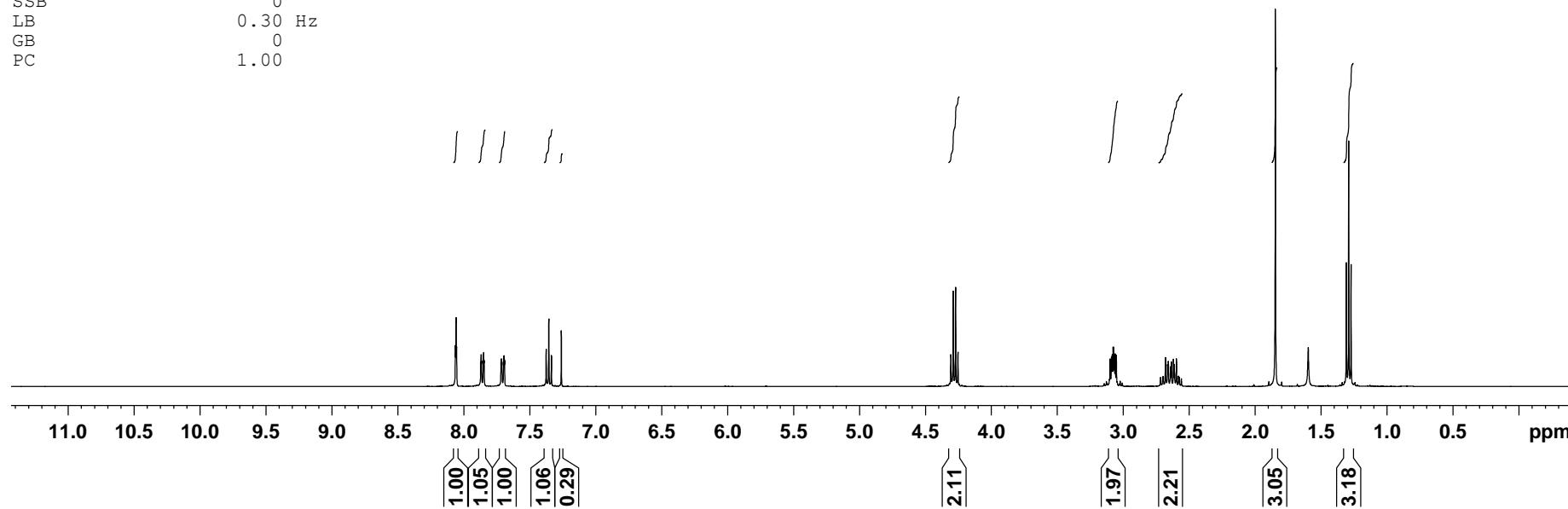
Index	Name	Time [Min]	Quantity [% Area]	Height [μAU]	Area [μAU.Min]	Area % [%]
1	UNKNOWN	19.812	80.37	750105.7	341846.2	80.372
2	UNKNOWN	20.785	19.63	180138.4	83483.9	19.628
Total			100.00	930244.2	425330.1	100.000

Fig S112. HPLC Profile of Enantioenriched **5b**

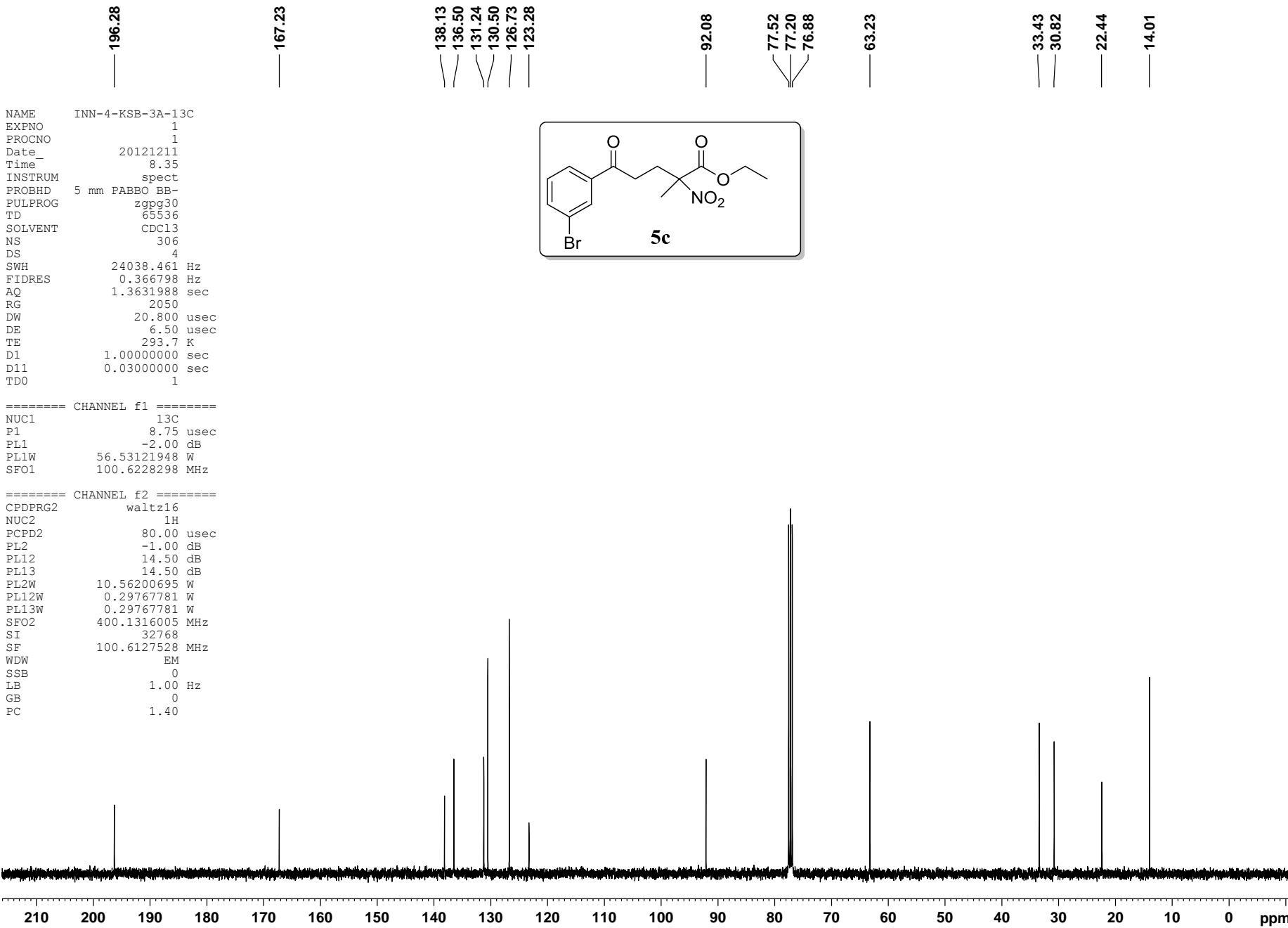
NAME INN-4-KSB-3A-1H  
 EXPNO 1  
 PROCNO 1  
 Date 20121211  
 Time 8.29  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 14  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 sec  
 RG 144  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 293.0 K  
 D1 1.00000000 sec  
 TDO 1



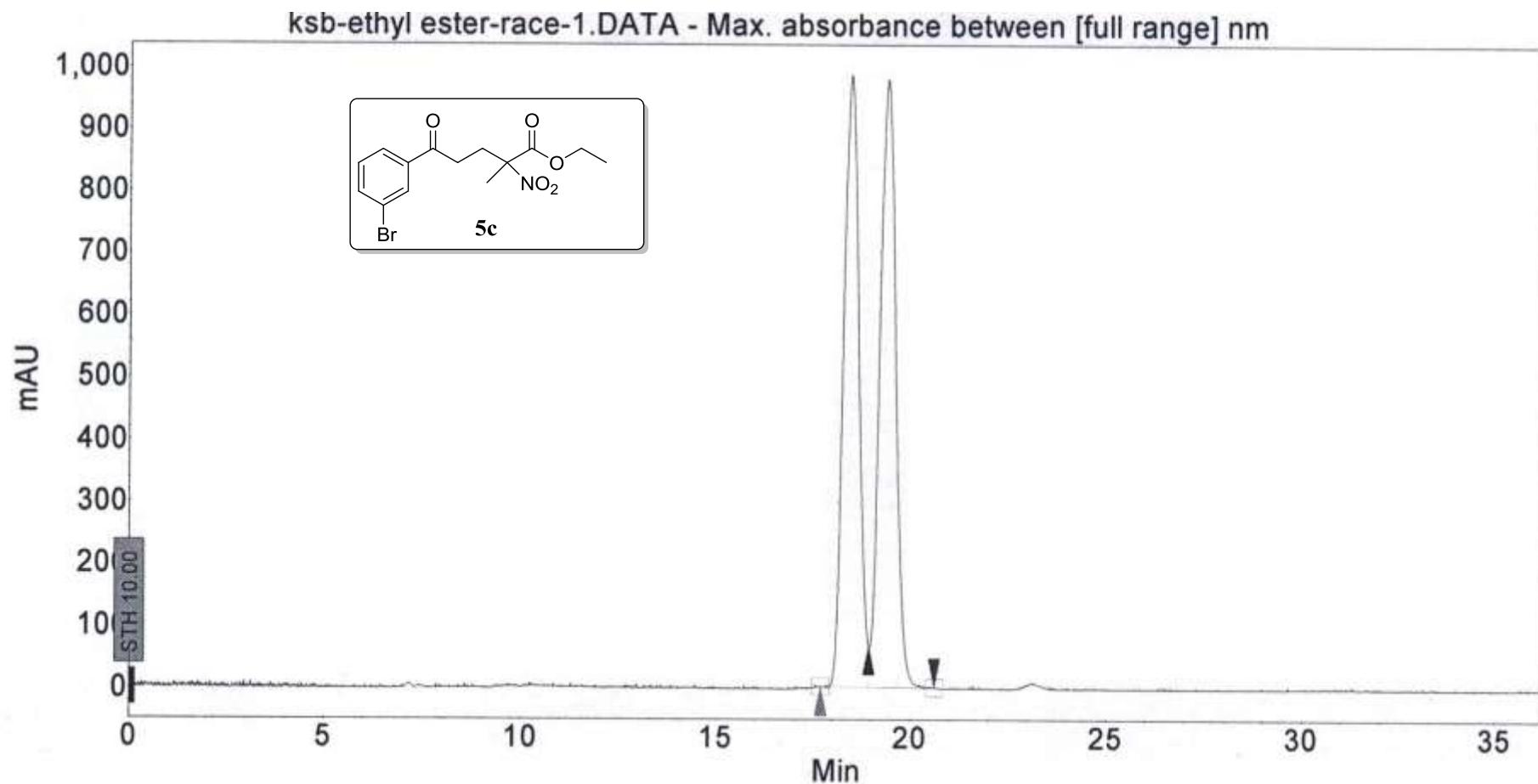
===== CHANNEL f1 ======  
 NUC1 1H  
 P1 13.50 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300095 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S113.**  $^1\text{H}$  NMR Spectrum of **5c**



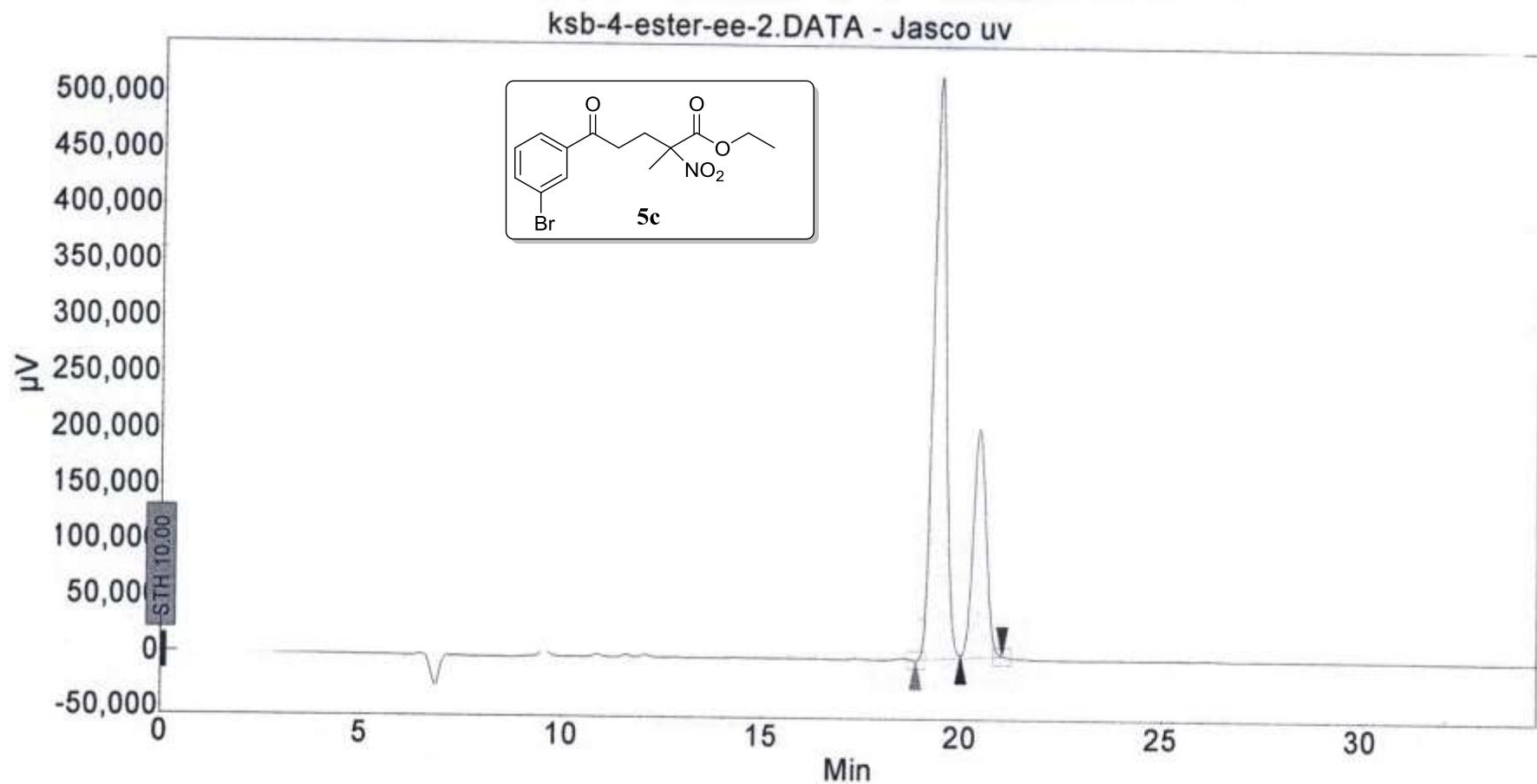
**Fig S114.**  $^{13}\text{C}$  NMR Spectrum of **5c**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18.439	49.12	985.8	447.2	49.122
2	UNKNOWN	19.385	50.88	980.3	463.2	50.878
Total			100.00	1966.1	910.4	100.000

Fig S115. HPLC Profile of Racemic **5c**



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
1	UNKNOWN	19.408	71.28	520587.1	190188.4	71.282
2	UNKNOWN	20.450	28.72	205078.5	76623.1	28.718
Total			100.00	725665.6	266811.5	100.000

**Fig S116. HPLC Profile of Enantioenriched 5c**

Current Data Parameters  
NAME INN-2-NSS-14-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20141118  
Time 16.55  
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 98.91  
DW 50.000 usec  
DE 6.50 usec  
TE 296.7 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300122 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

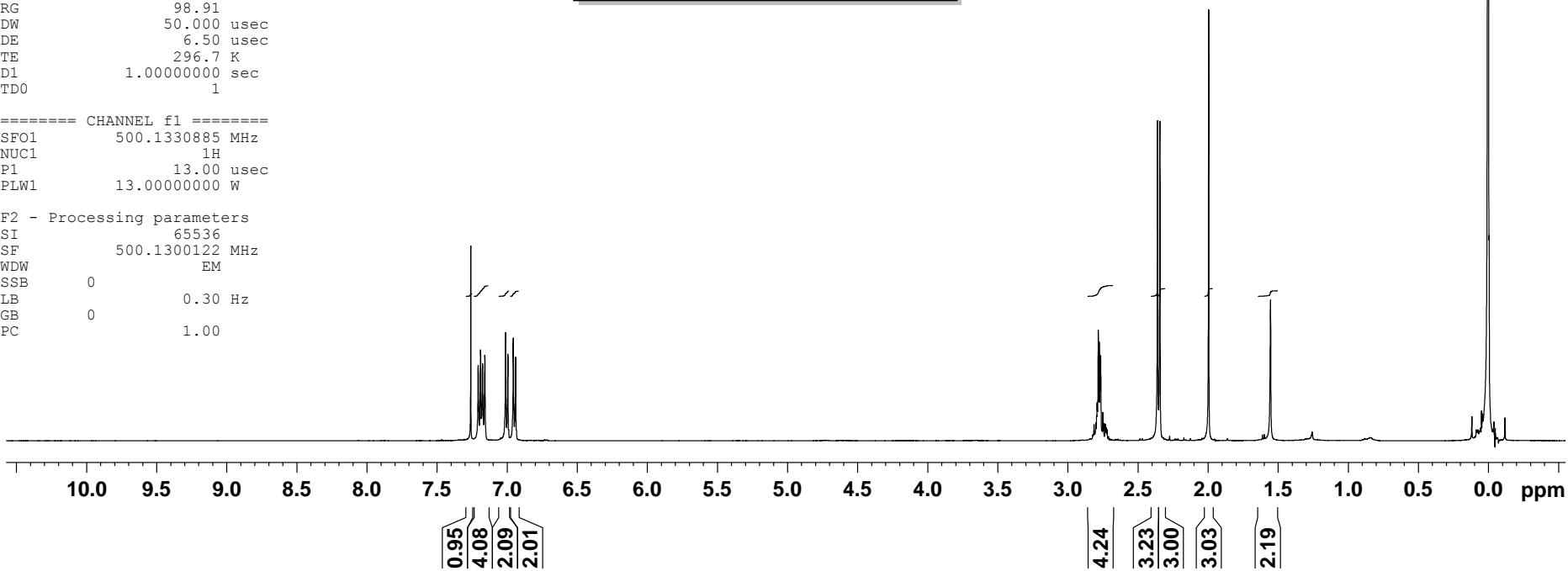
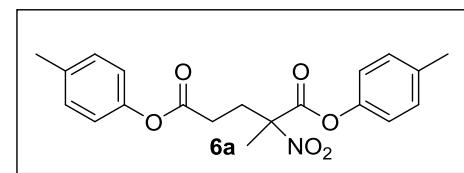


Fig S117.  $^1\text{H}$  NMR Spectrum of 6a

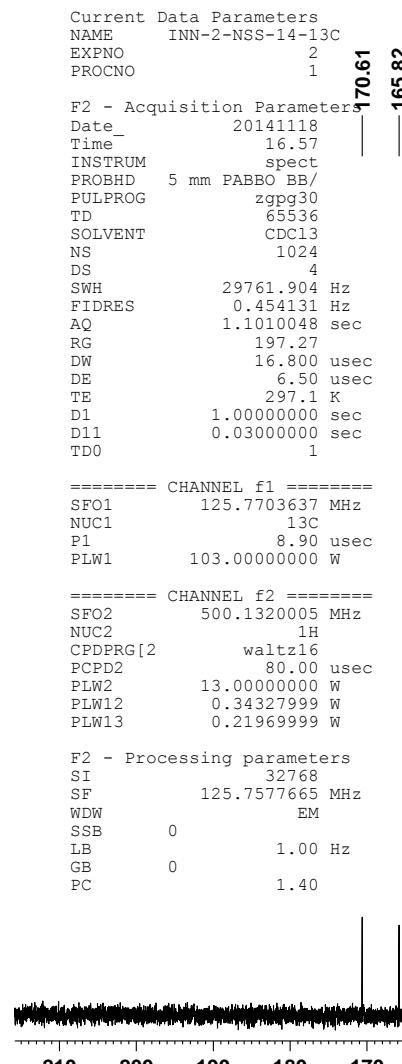
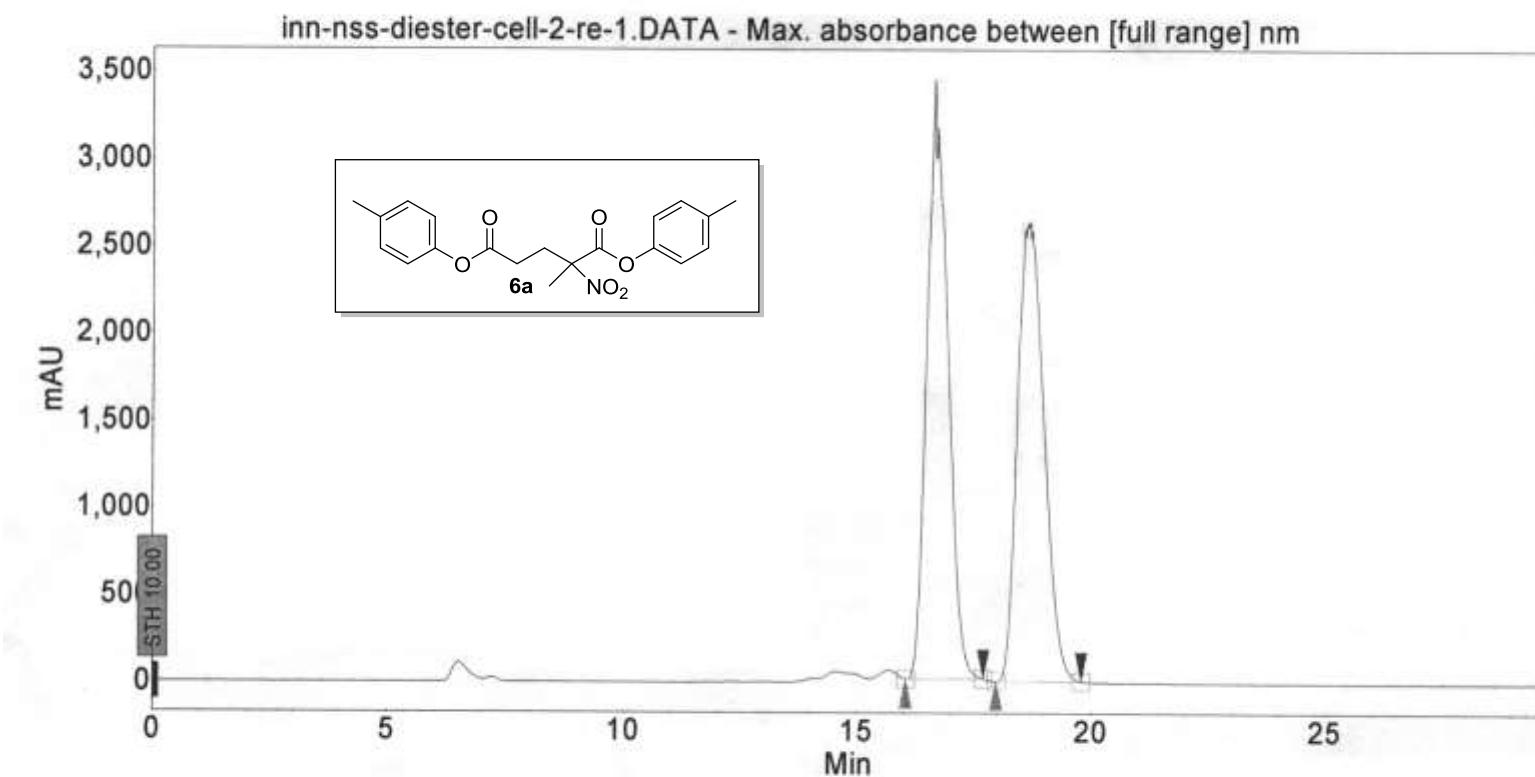


Fig S118.  $^{13}\text{C}$  NMR Spectrum of 6a

System : hplc  
Method : nss-10%05f-216nm  
User : User1

Acquired : 1/23/2005 5:34:15 PM  
Processed : 2/25/2005 6:24:58 PM  
Printed : 2/25/2005 6:26:58 PM



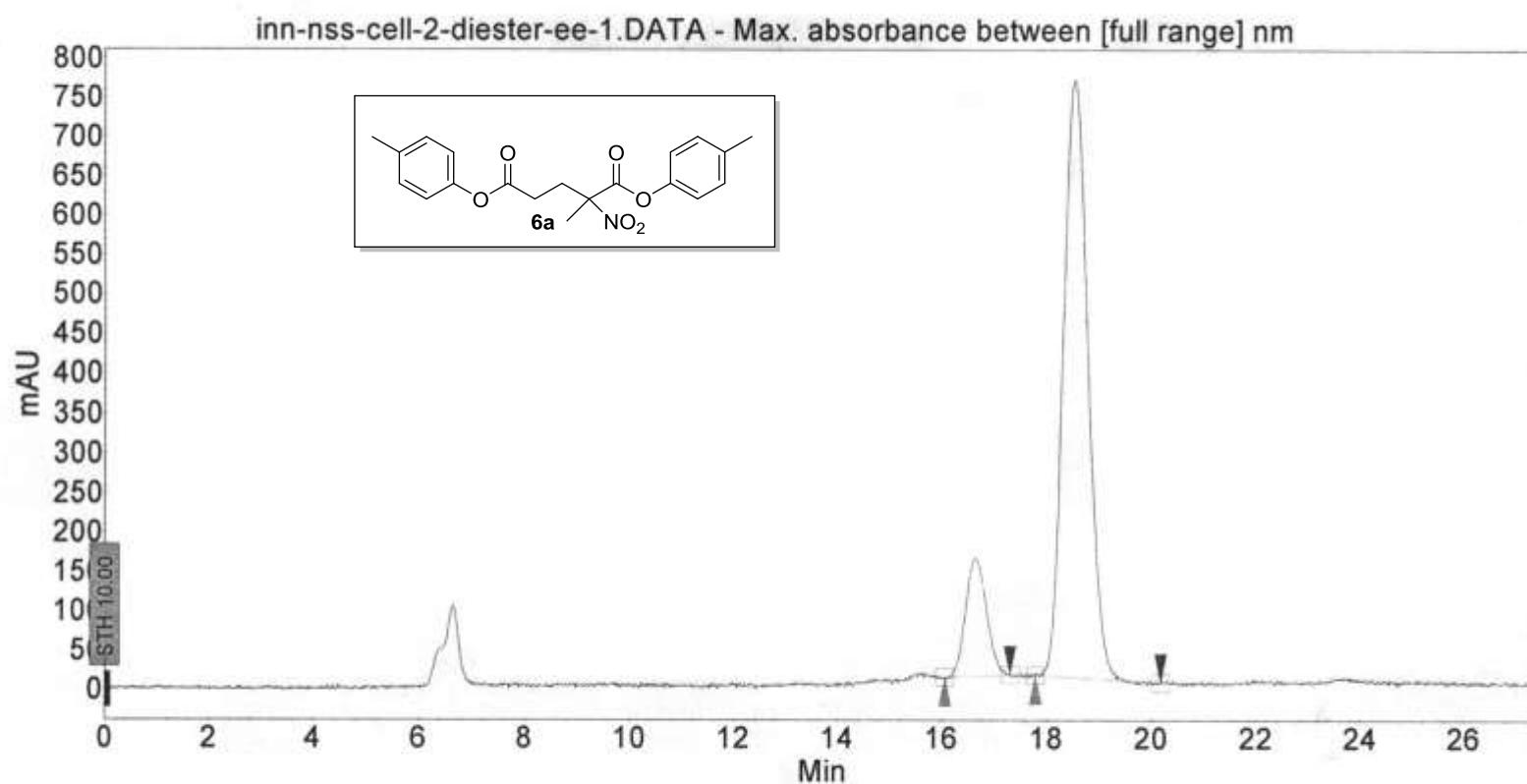
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	16.652	50.53	3433.5	1756.0	50.527
2	UNKNOWN	18.705	49.47	2634.2	1719.4	49.473
Total			100.00	6067.7	3475.5	100.000

Fig S119. HPLC Profile of Racemic 6a

System : hplc  
Method : nss-10%05f-216nm  
User : User1

Acquired : 1/23/2005 7:05:38 PM  
Processed : 1/23/2005 7:35:36 PM  
Printed : 2/25/2005 6:27:57 PM



### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	16.652	14.52	150.1	69.5	14.521
2	UNKNOWN	18.532	85.48	755.2	409.1	85.479
Total			100.00	905.2	478.5	100.000

Fig S120. HPLC Profile of Enantioenriched 6a

Current Data Parameters  
NAME inn-2-nss-131-1h  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date 20160508  
Time 16.29  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 101  
DW 60.800 usec  
DE 6.50 usec  
TE 294.7 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 ======

NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300103 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

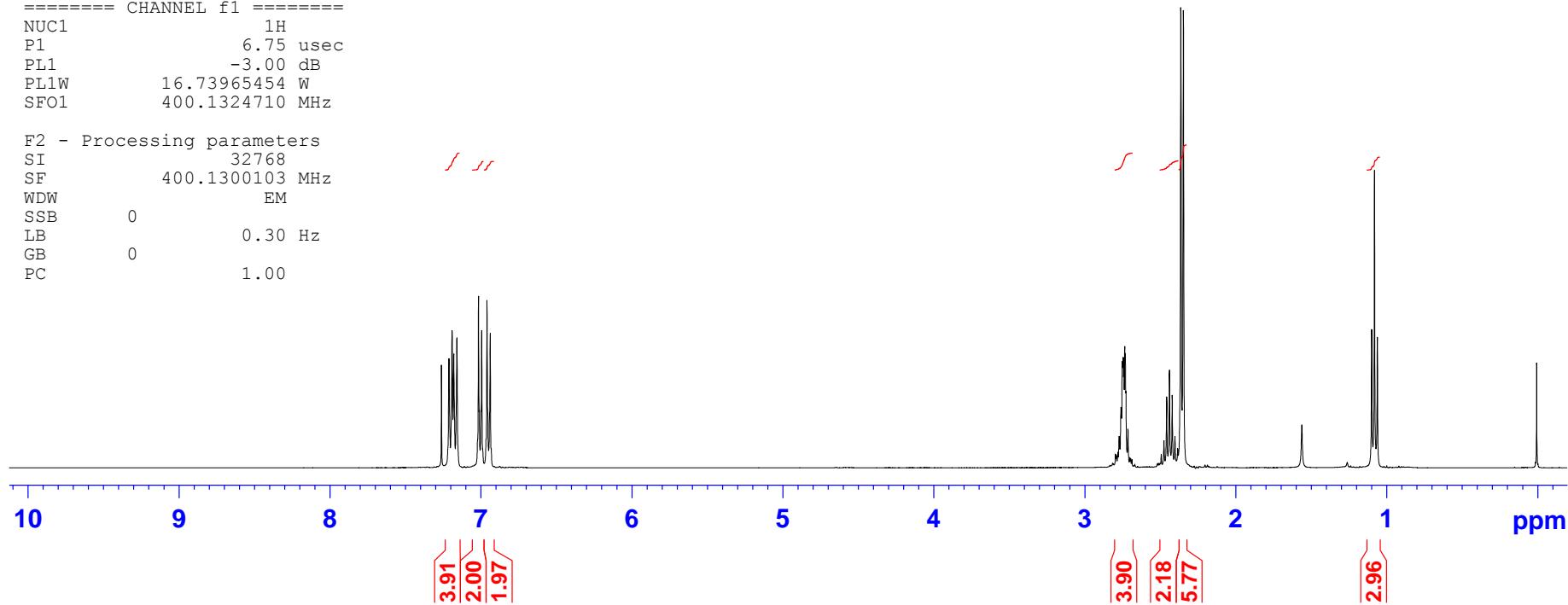
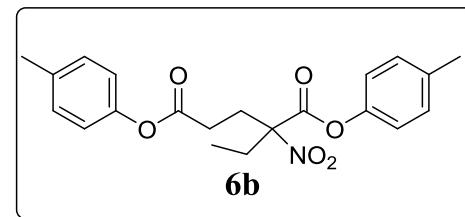


Fig S121.  $^1\text{H}$  NMR Spectrum of **6b**

Current Data Parameters  
 NAME inn-2-nss-131-1<sup>3</sup>C  
 EXPNO 9  
 PROCNO 1 — 170.63 — 165.36

F2 - Acquisition Parameters  
 Date 20160508  
 Time 16.30  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 22  
 DS 4  
 SWH 26041.666 Hz  
 FIDRES 0.397364 Hz  
 AQ 1.2582912 sec  
 RG 256  
 DW 19.200 usec  
 DE 6.50 usec  
 TE 295.1 K  
 D1 1.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

===== CHANNEL f1 =====

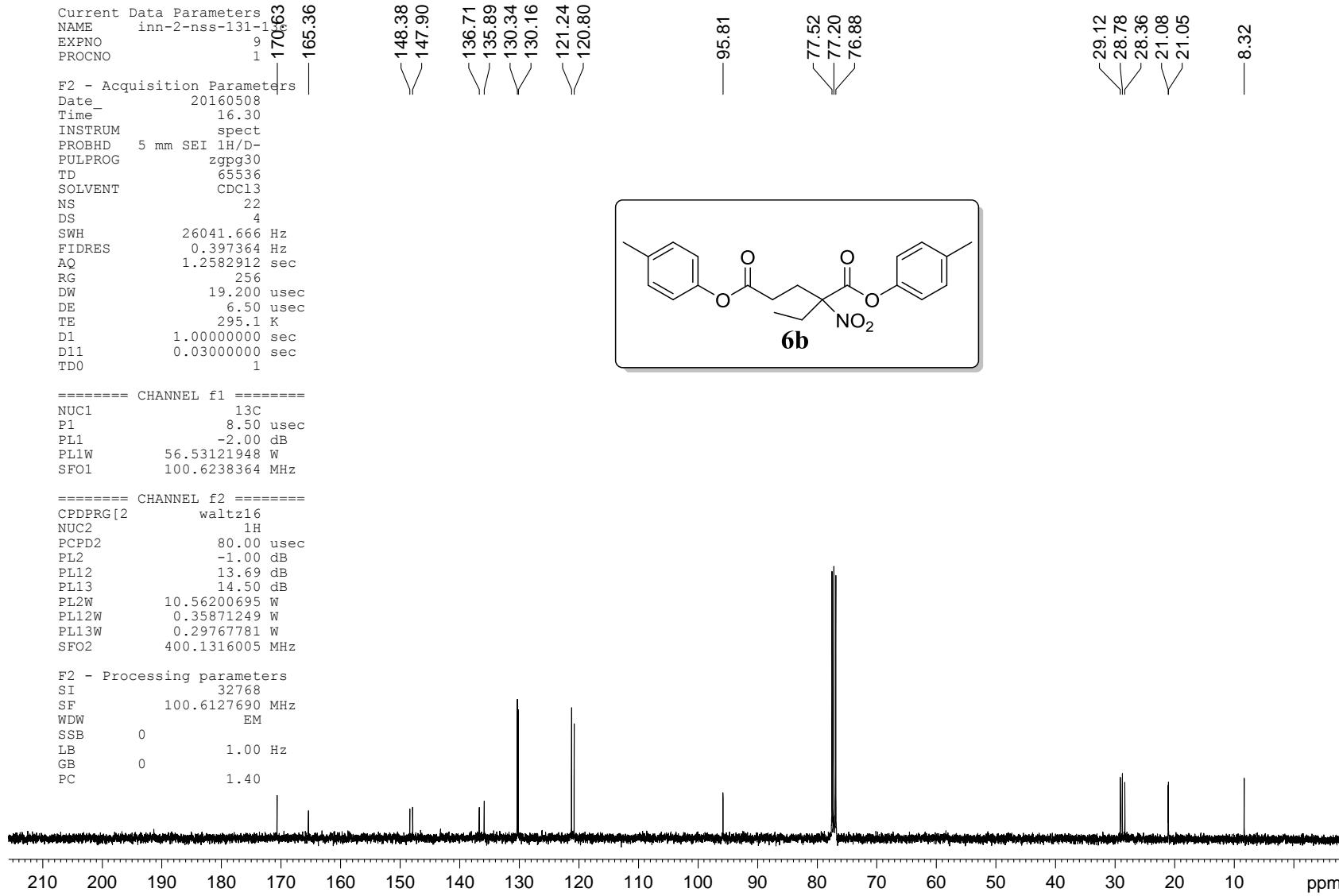
NUC1 13C  
 P1 8.50 usec  
 PL1 -2.00 dB  
 PL1W 56.53121948 W  
 SFO1 100.6238364 MHz

===== CHANNEL f2 =====

CPDPRG[2 waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -1.00 dB  
 PL12 13.69 dB  
 PL13 14.50 dB  
 PL2W 10.56200695 W  
 PL12W 0.35871249 W  
 PL13W 0.29767781 W  
 SFO2 400.1316005 MHz

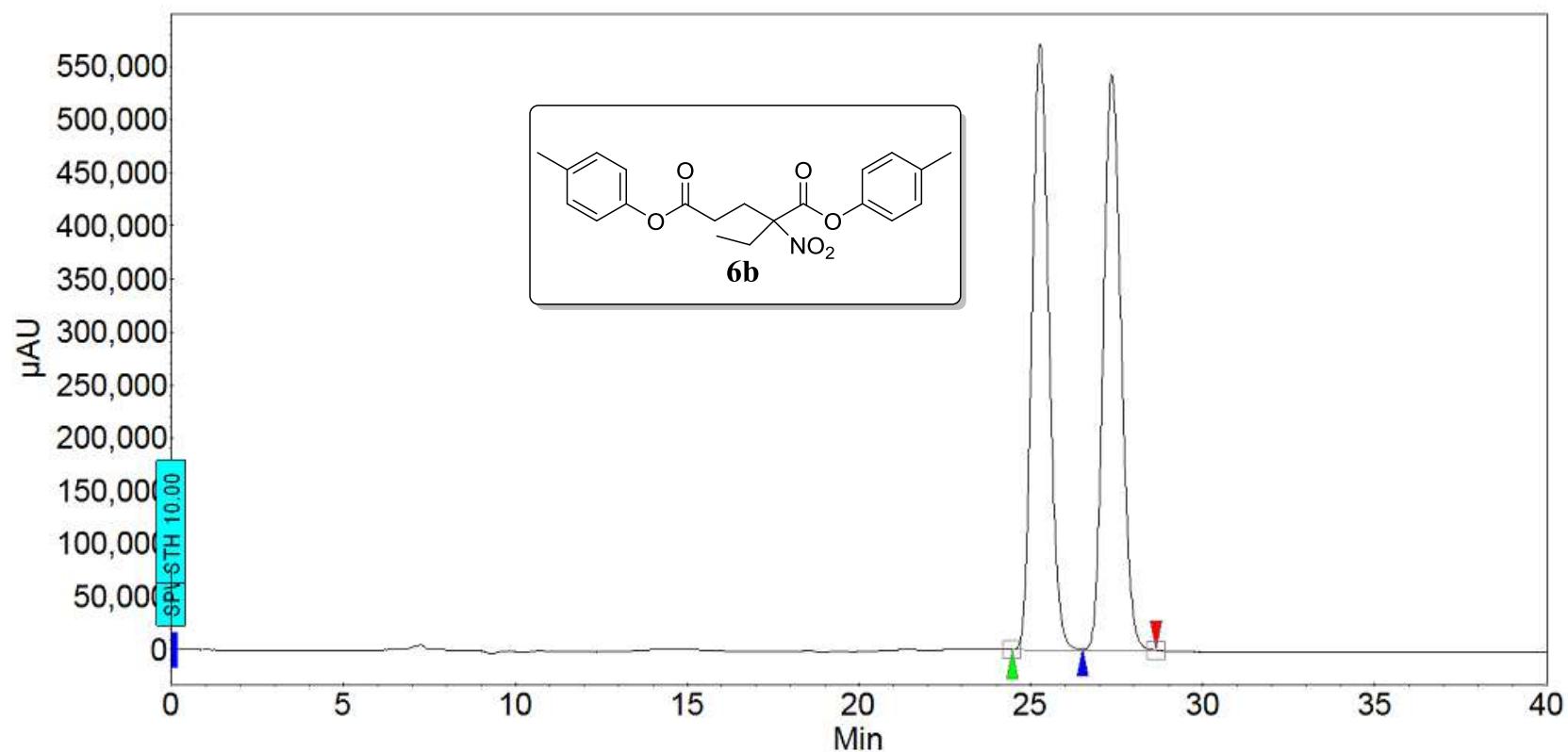
F2 - Processing parameters

SI 32768  
 SF 100.6127690 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



**Fig S122.** <sup>13</sup>C NMR Spectrum of **6b**

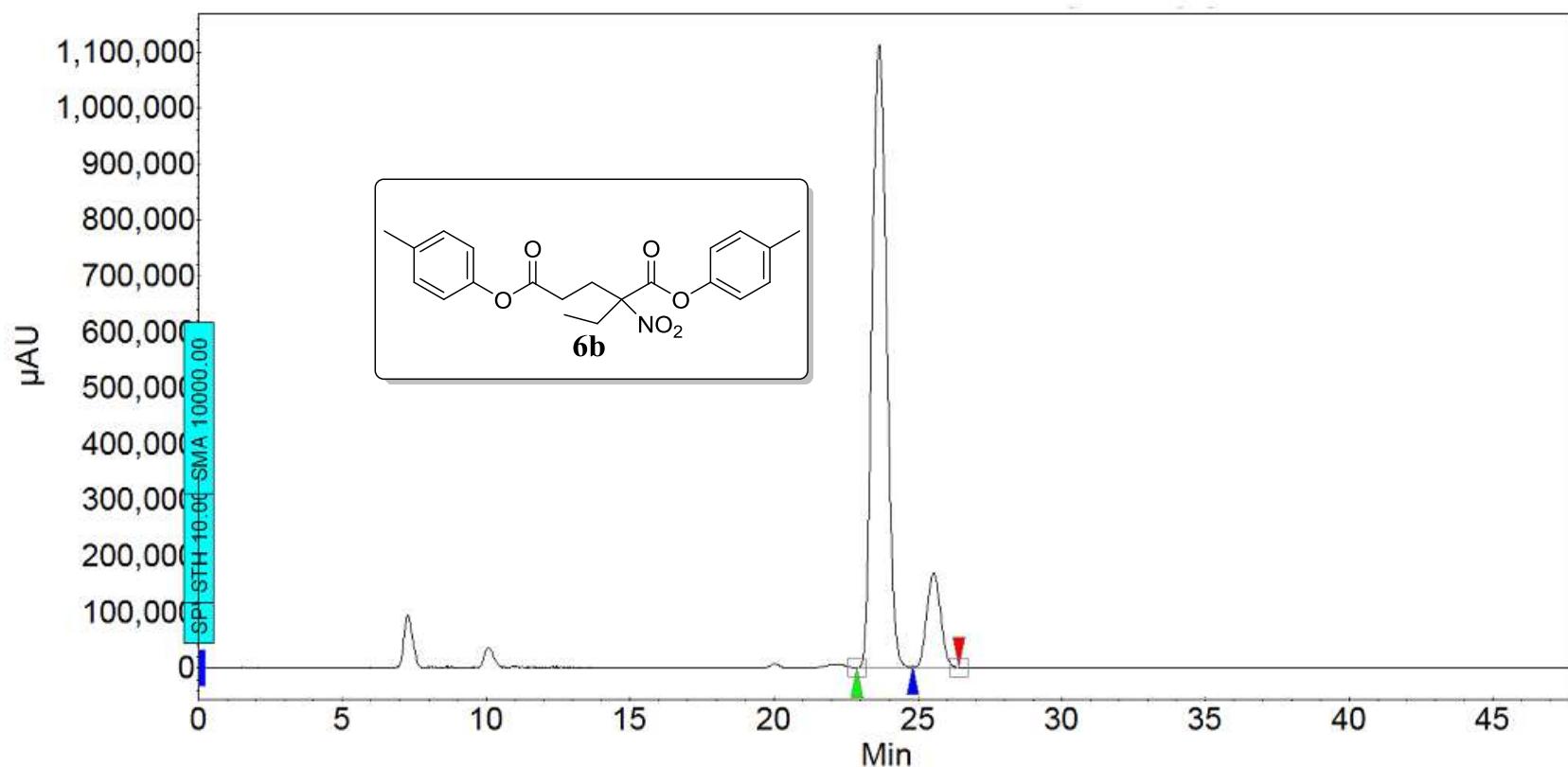
nss-2-131-1%-cell-1-rac-1.DATA-216.00nm



### Peak results :

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

Fig S123. HPLC Profile of Racemic **6b**



### Peak results :

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

Fig S124. HPLC Profile of Enantioenriched **6b**

Current Data Parameters  
NAME INN-1-NSS-185-E-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140908  
Time 22.45  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 12  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 128  
DW 60.800 usec  
DE 6.50 usec  
TE 296.9 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300101 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

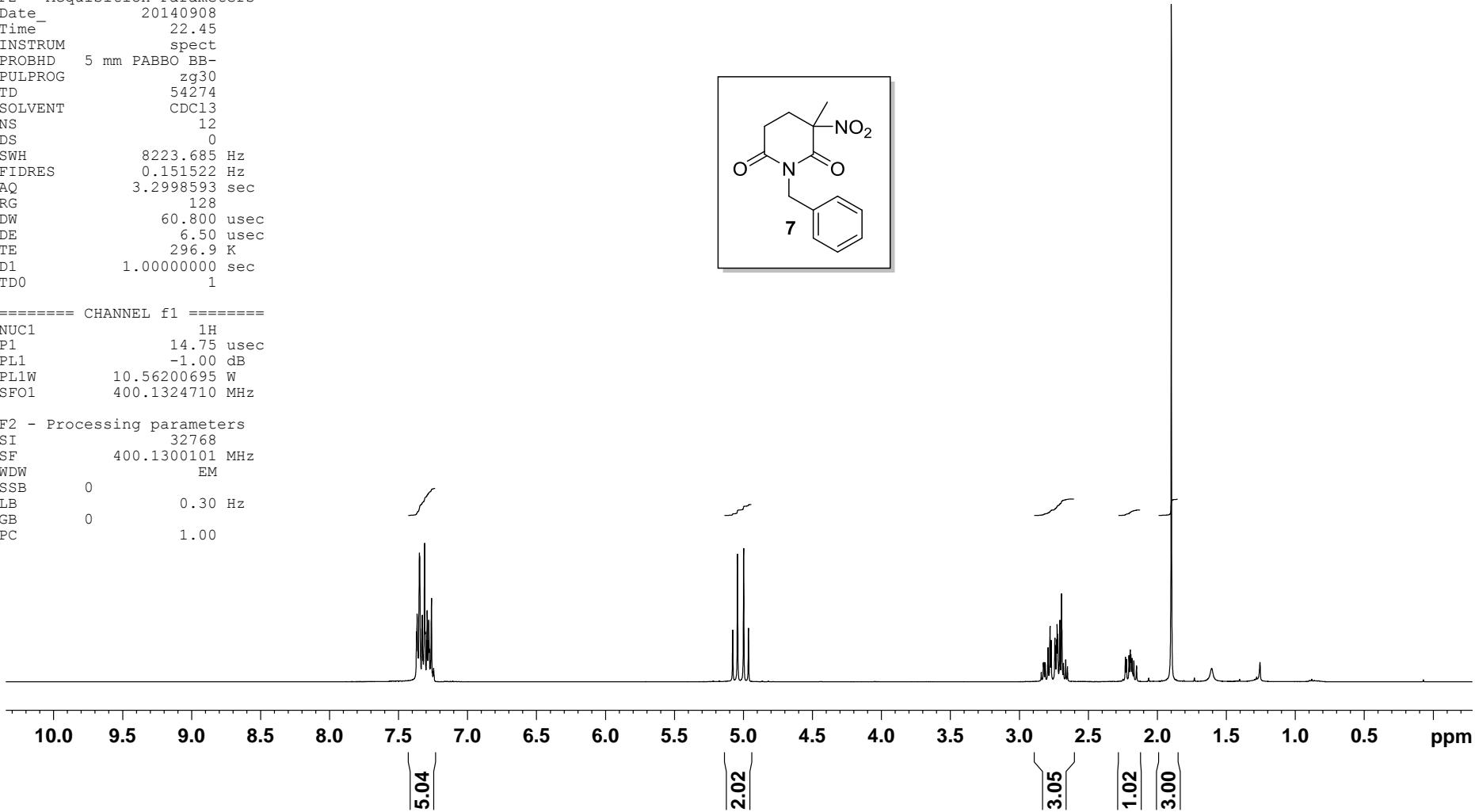
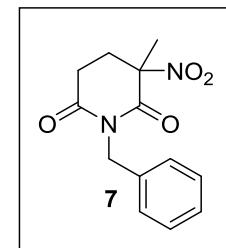


Fig S125. <sup>1</sup>H NMR Spectrum of 7

Current Data Parameters  
NAME INN-1-NSS-185-E-13C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140908  
Time\_ 22.50  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 263  
DS 4  
SWH 27573.529 Hz  
FIDRES 0.420739 Hz  
AQ 1.1883861 sec  
RG 2050  
DW 18.133 usec  
DE 6.50 usec  
TE 297.7 K  
D1 1.0000000 sec  
D11 0.0300000 sec  
TDO 1

===== CHANNEL f1 ======  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6242389 MHz

===== CHANNEL f2 ======  
CPDPGRG[2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127528 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

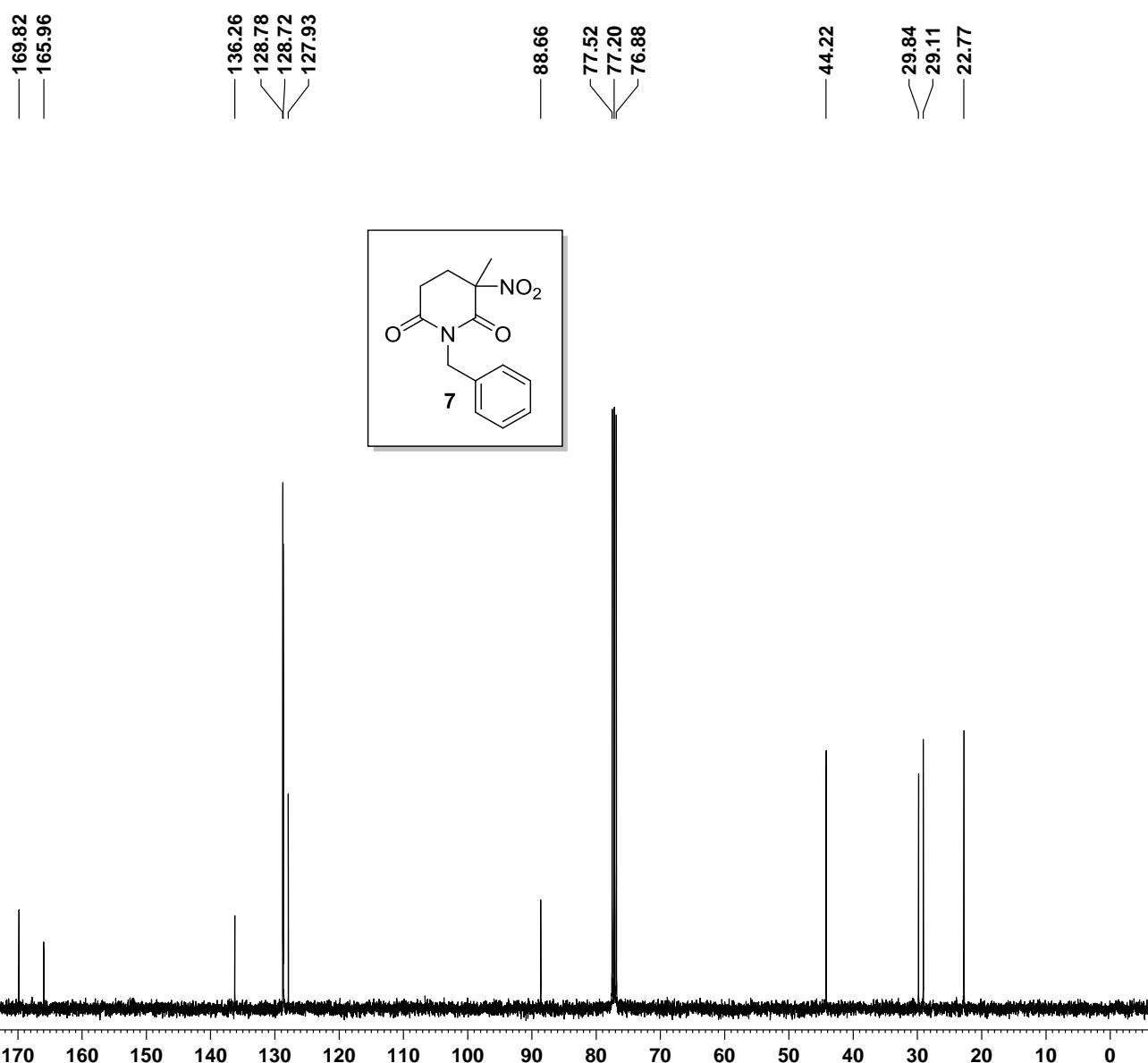
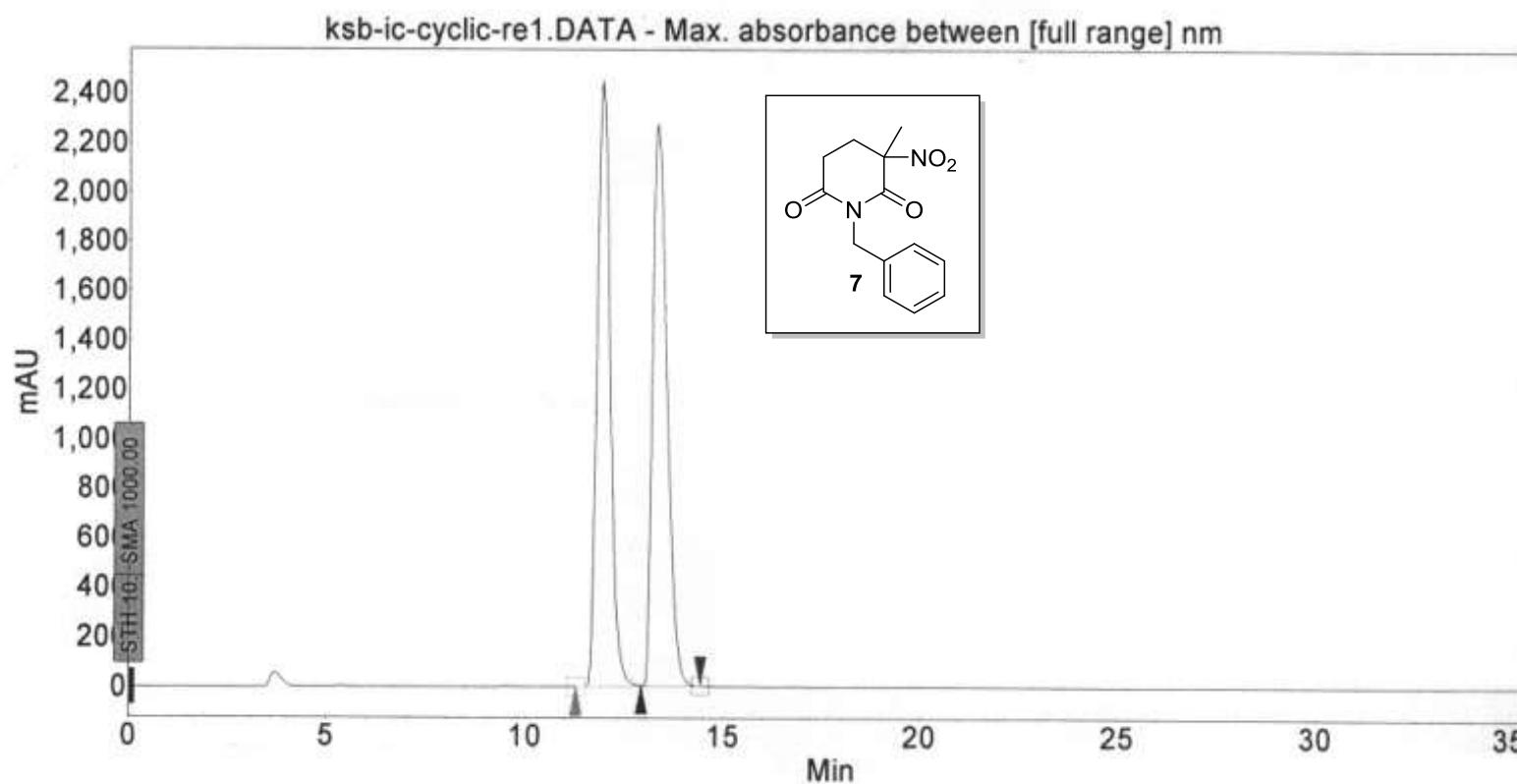


Fig S126. <sup>13</sup>C NMR Spectrum of 7

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

Acquired : 1/29/2005 11:12:40 AM  
Processed : 1/29/2005 12:14:07 PM  
Printed : 1/30/2005 10:51:29 AM



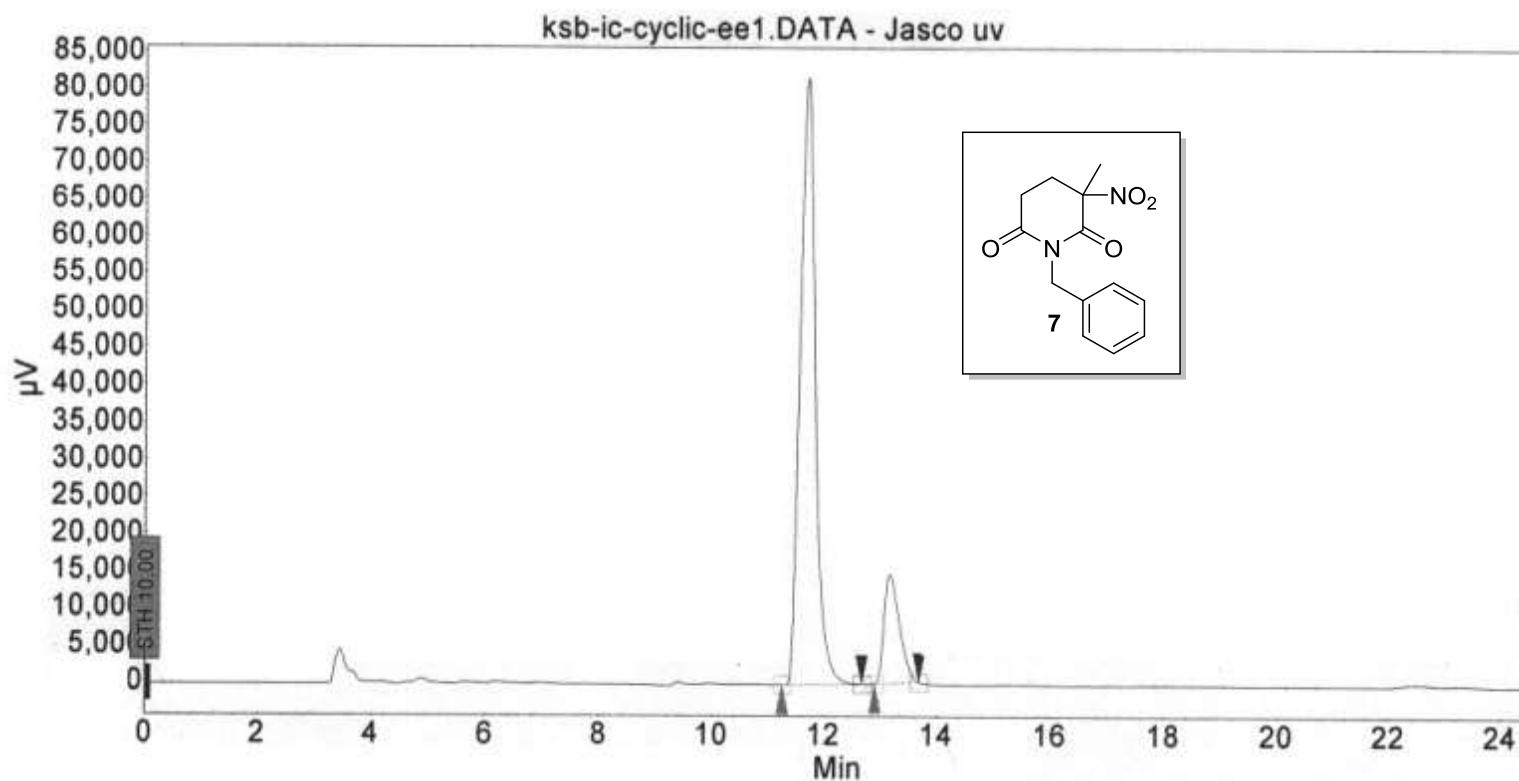
### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	11.919	49.36	2450.8	941.8	49.356
2	UNKNOWN	13.319	50.64	2273.9	966.4	50.644
Total			100.00	4724.8	1908.2	100.000

Fig S127. HPLC Profile of Racemic 7

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

Acquired : 1/29/2005 11:49:55 AM  
Processed : 1/30/2005 11:01:17 AM  
Printed : 1/30/2005 11:01:41 AM



#### Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [µV]	Area [µV.Min]	Area [%]
1	UNKNOWN	11.700	84.62	81761.9	25750.6	84.619
2	UNKNOWN	13.175	15.38	14725.6	4680.5	15.381
Total			100.00	96487.5	30431.1	100.000

Fig S128. HPLC Profile of Enantioenriched 7

Current Data Parameters  
NAME INN-1-NSS-185-RE-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date 2014905  
Time 20.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 14  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 144  
DW 60.800 usec  
DE 6.50 usec  
TE 296.1 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300100 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

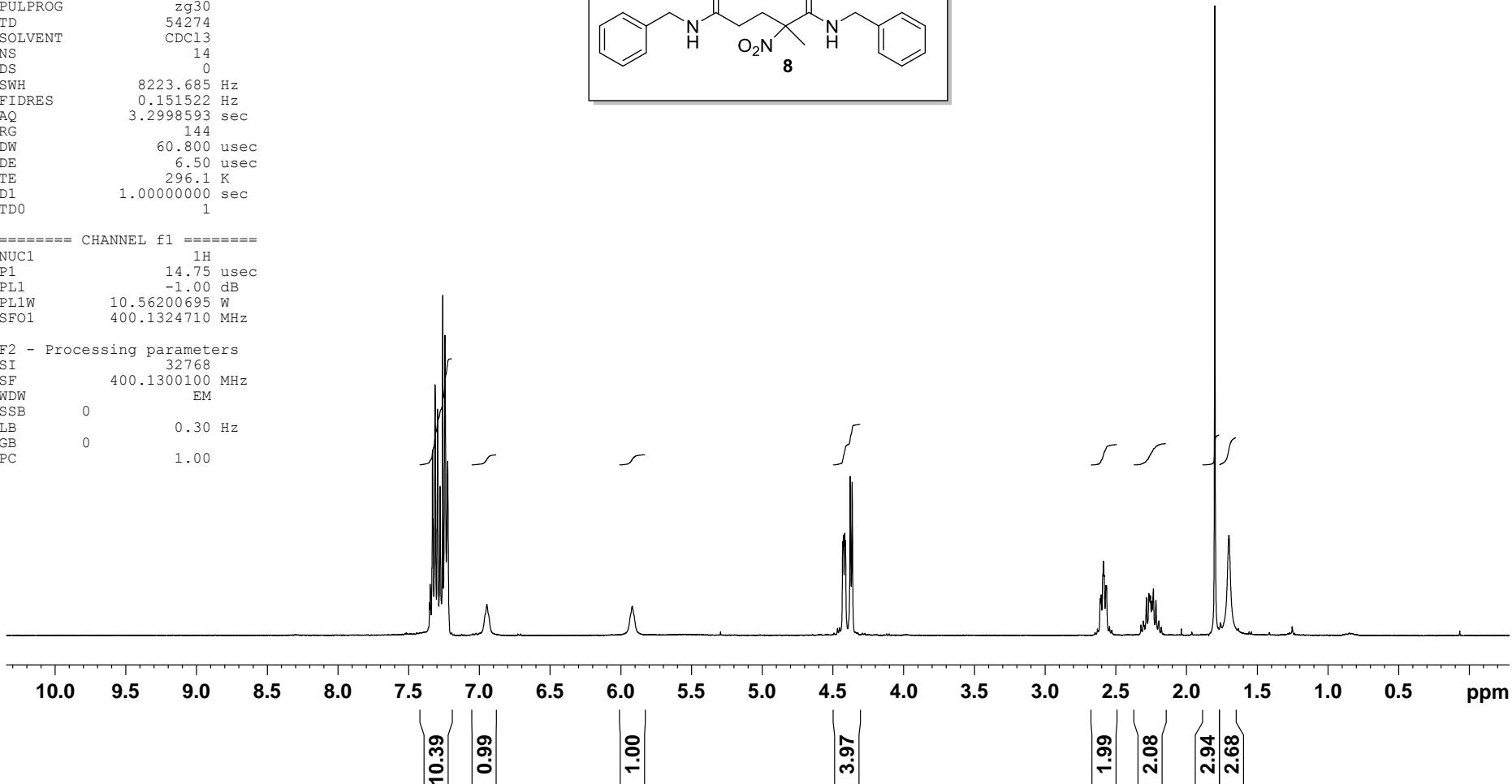
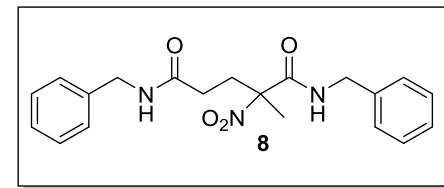


Fig S129.  $^1\text{H}$  NMR Spectrum of 8

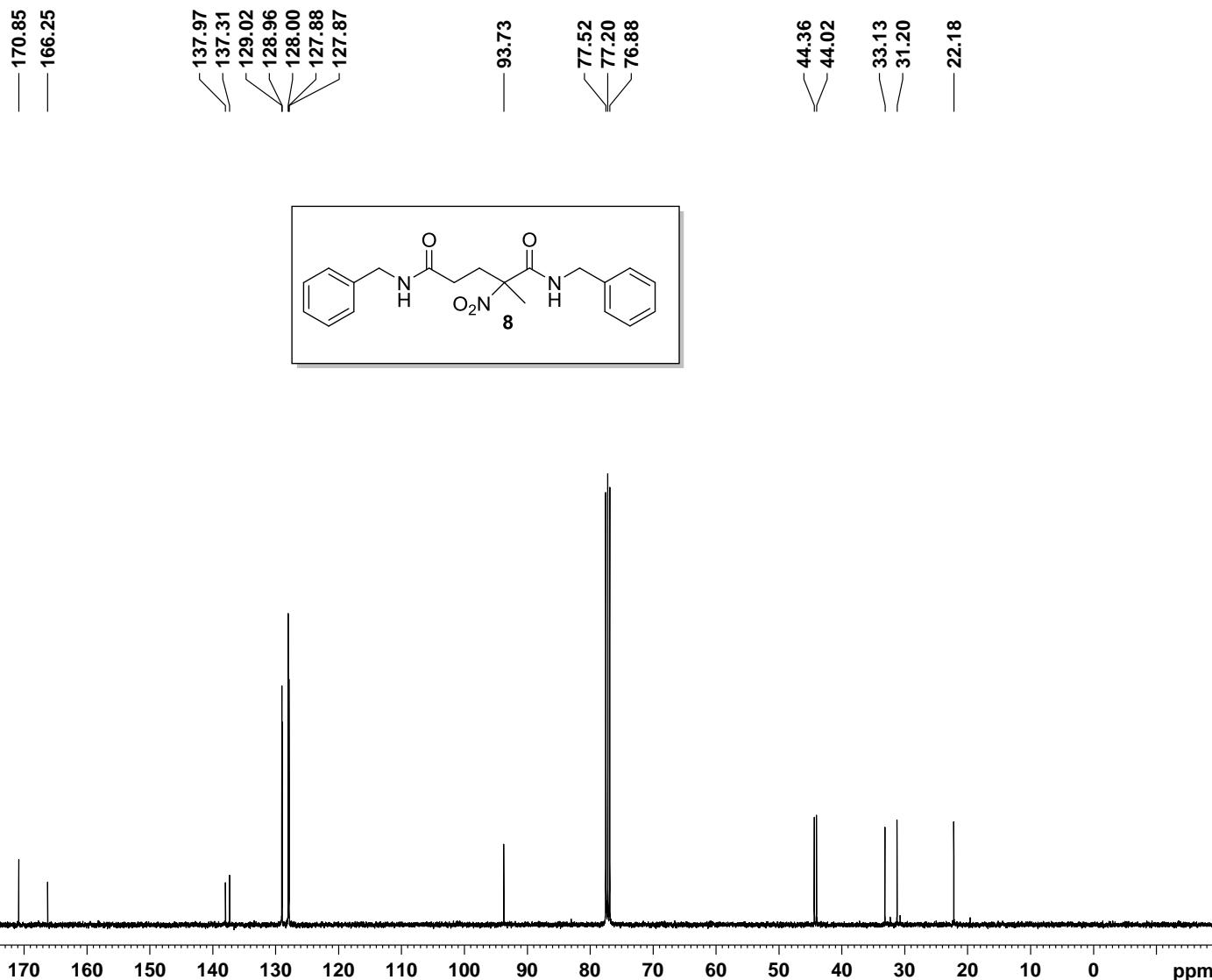
Current Data Parameters  
 NAME INN-NSS-DIAMIDE-13C  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 20141227  
 Time 1.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 1637  
 DS 4  
 SWH 26041.666 Hz  
 FIDRES 0.397364 Hz  
 AQ 1.2582912 sec  
 RG 2050  
 DW 19.200 usec  
 DE 6.50 usec  
 TE 300.2 K  
 D1 1.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

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

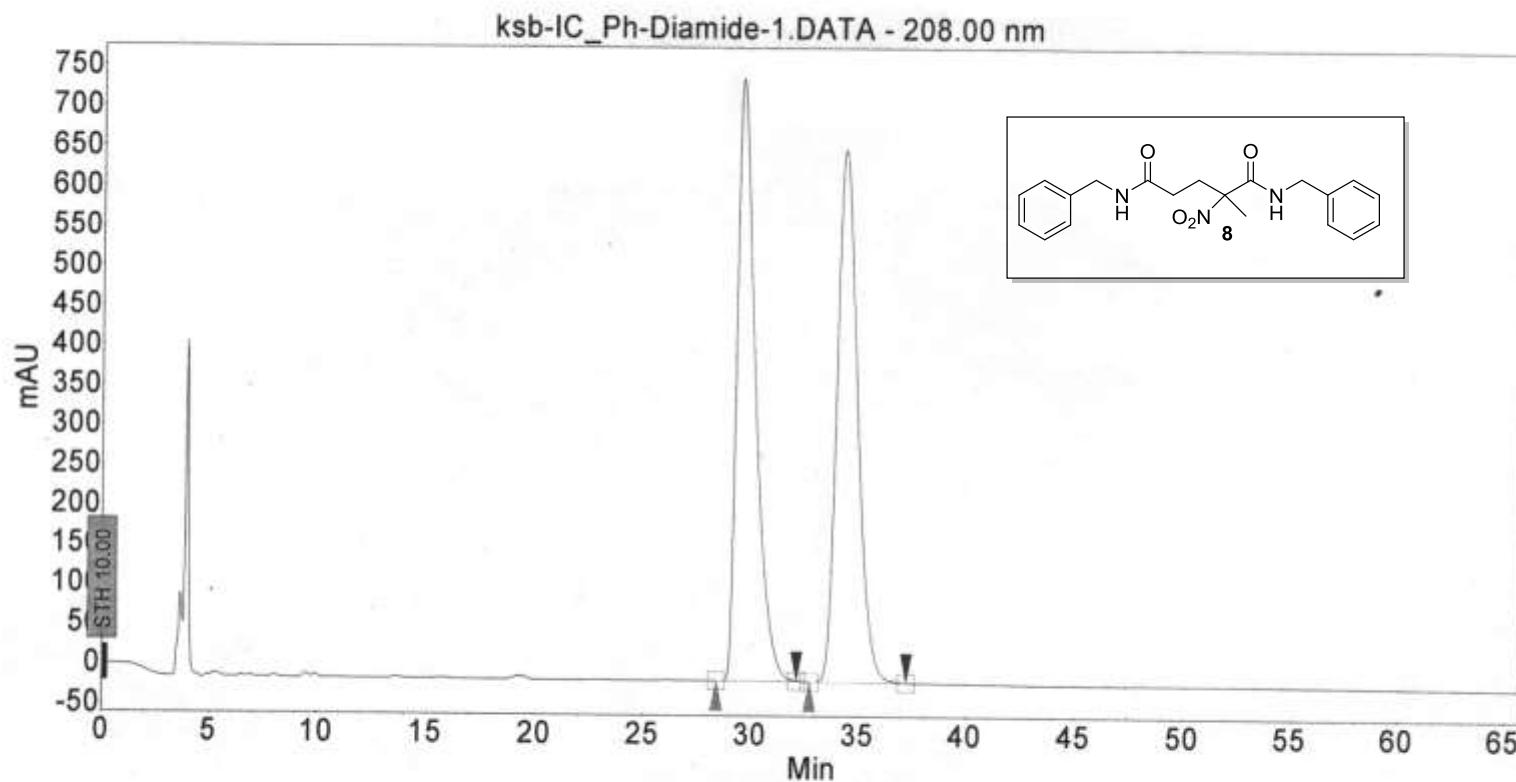
===== CHANNEL f2 ======  
 CPDPRG[2] waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -1.00 dB  
 PL12 13.69 dB  
 PL13 14.50 dB  
 PL2W 10.56200695 W  
 PL12W 0.35871249 W  
 PL13W 0.29767781 W  
 SFO2 400.1316005 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6127507 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



System : hplc  
Method : NOT DEFINED  
User : User1

Acquired : 1/28/2005 6:58:21 PM  
Processed : 1/28/2005 8:08:50 PM  
Printed : 1/30/2005 10:58:03 AM



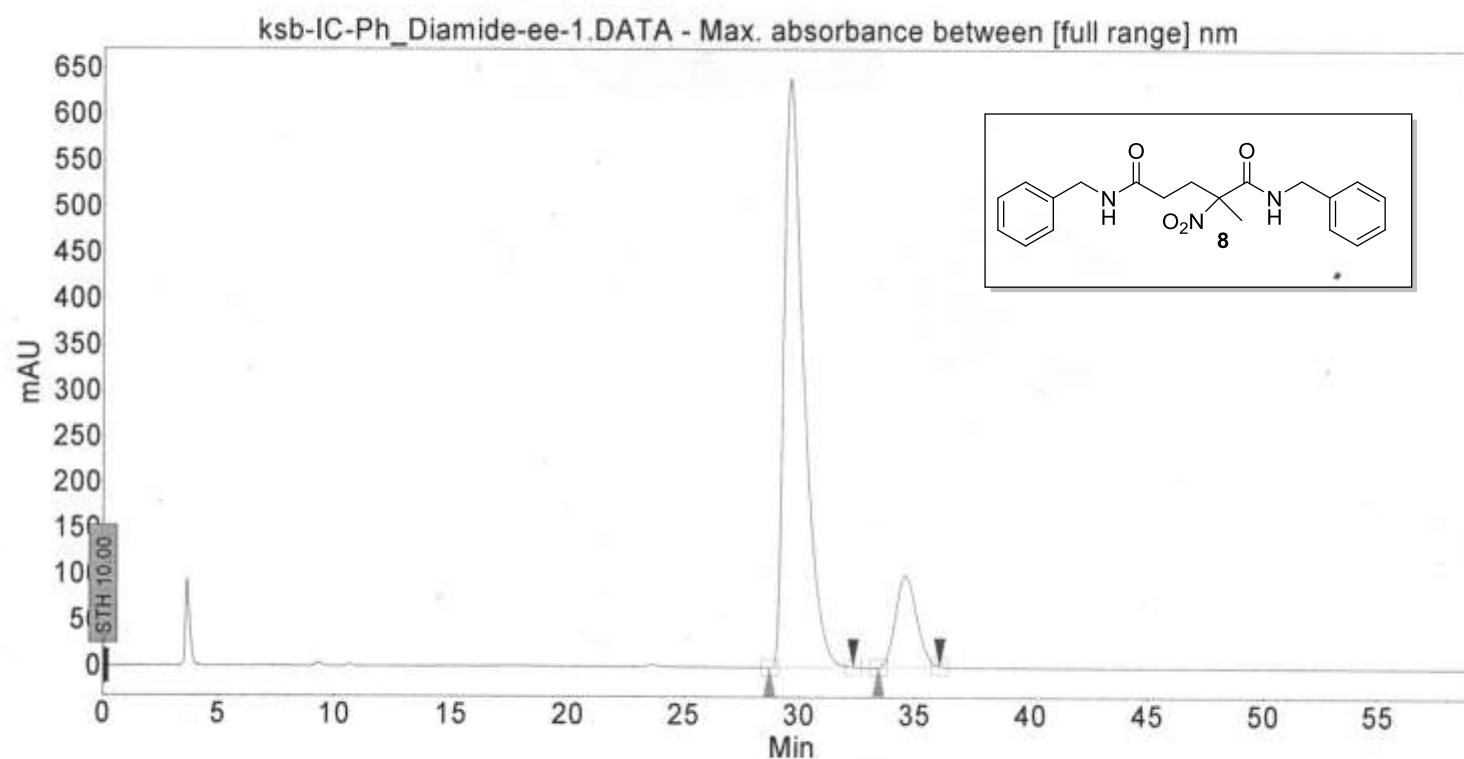
#### Peak results :

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

Fig S131. HPLC Profile of Racemic 8

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

Acquired : 1/28/2005 8:32:06 PM  
Processed : 1/28/2005 9:34:48 PM  
Printed : 1/30/2005 10:55:15 AM



### Peak results :

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

Fig S132. HPLC Profile of Enantioenriched 8

Current Data Parameters  
NAME inn-nss-186-B-1h  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160223  
Time 7.25  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 6  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 114  
DW 60.800 usec  
DE 6.50 usec  
TE 294.6 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 ======  
NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300113 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

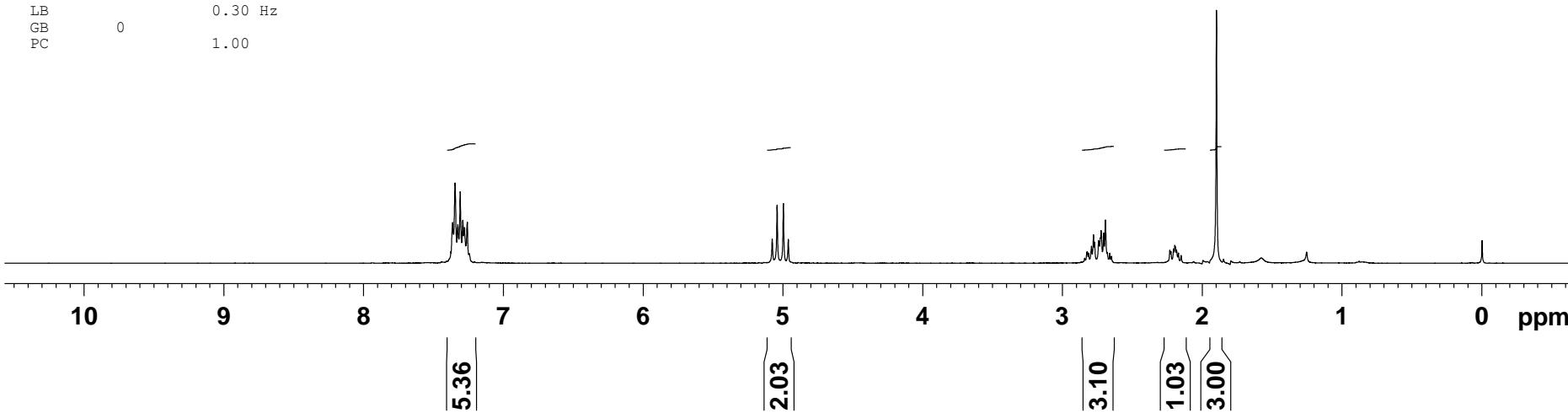
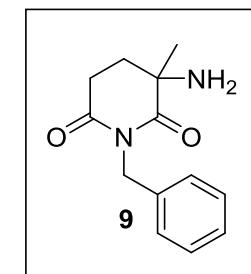


Fig S133. <sup>1</sup>H NMR Spectrum of 9

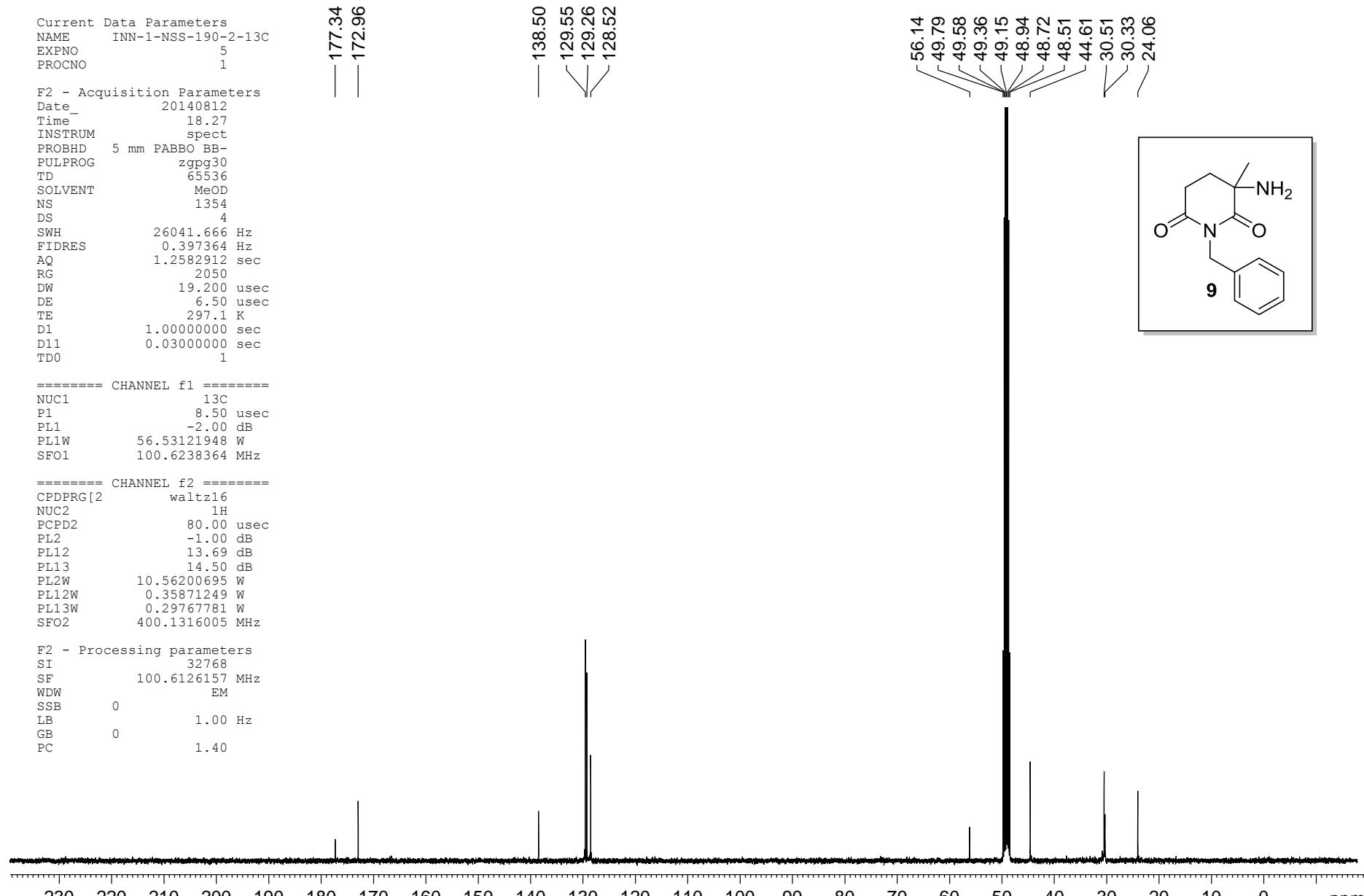
Current Data Parameters  
 NAME INN-1-NSS-190-2-13C  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date 20140812  
 Time 18.27  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT MeOD  
 NS 1354  
 DS 4  
 SWH 26041.666 Hz  
 FIDRES 0.397364 Hz  
 AQ 1.2582912 sec  
 RG 2050  
 DW 19.200 usec  
 DE 6.50 usec  
 TE 297.1 K  
 D1 1.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

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

===== CHANNEL f2 =====  
 CPDPRG[2] waltz16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 -1.00 dB  
 PL12 13.69 dB  
 PL13 14.50 dB  
 PL2W 10.56200695 W  
 PL12W 0.35871249 W  
 PL13W 0.29767781 W  
 SFO2 400.1316005 MHz

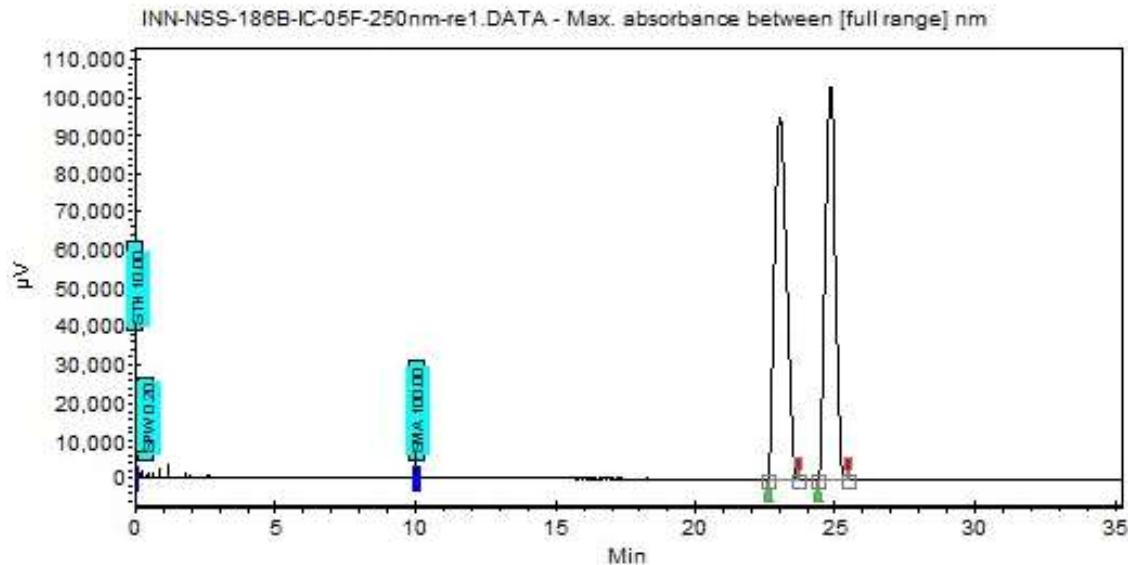
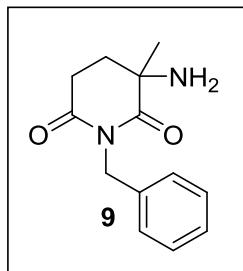
F2 - Processing parameters  
 SI 32768  
 SF 100.6126157 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



### Chromatogram : INN-NSS-186B-IC-05F-250nm-re1\_channel5

System : Jasco HPLC  
Method : nsa-10%-05F-250nm  
User : User1

Acquired : 2/22/2016 3:30:03 PM  
Processed : 2/22/2016 6:08:59 PM  
Printed : 2/22/2016 7:01:36 PM



#### Peak results :

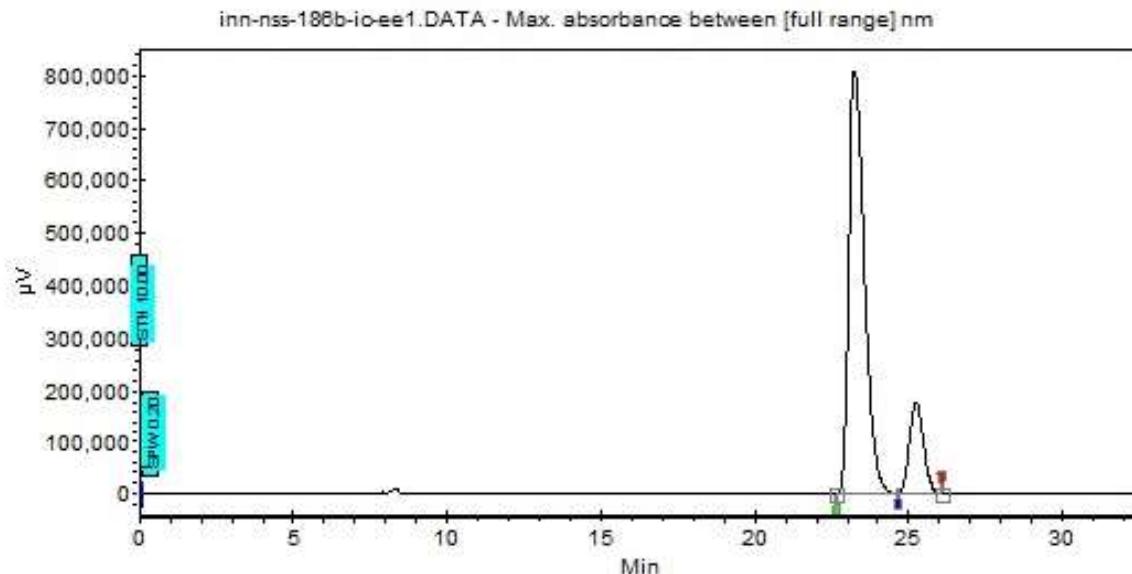
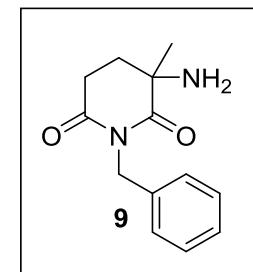
Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV Min]	Area % [%]
1	UNKNOWN	22.992	49.74	95122.8	47108.0	49.737
2	UNKNOWN	24.791	50.26	102944.6	47606.1	50.263
Total			100.00	198067.4	94714.1	100.000

Fig S135. HPLC Profile of Racemic 9

## Chromatogram : inn-nss-186b-ic-ee1\_channel5

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

Acquired : 2/22/2016 5:25:14 PM  
Processed : 2/22/2016 5:58:12 PM  
Printed : 2/22/2016 7:02:51 PM



### Peak results :

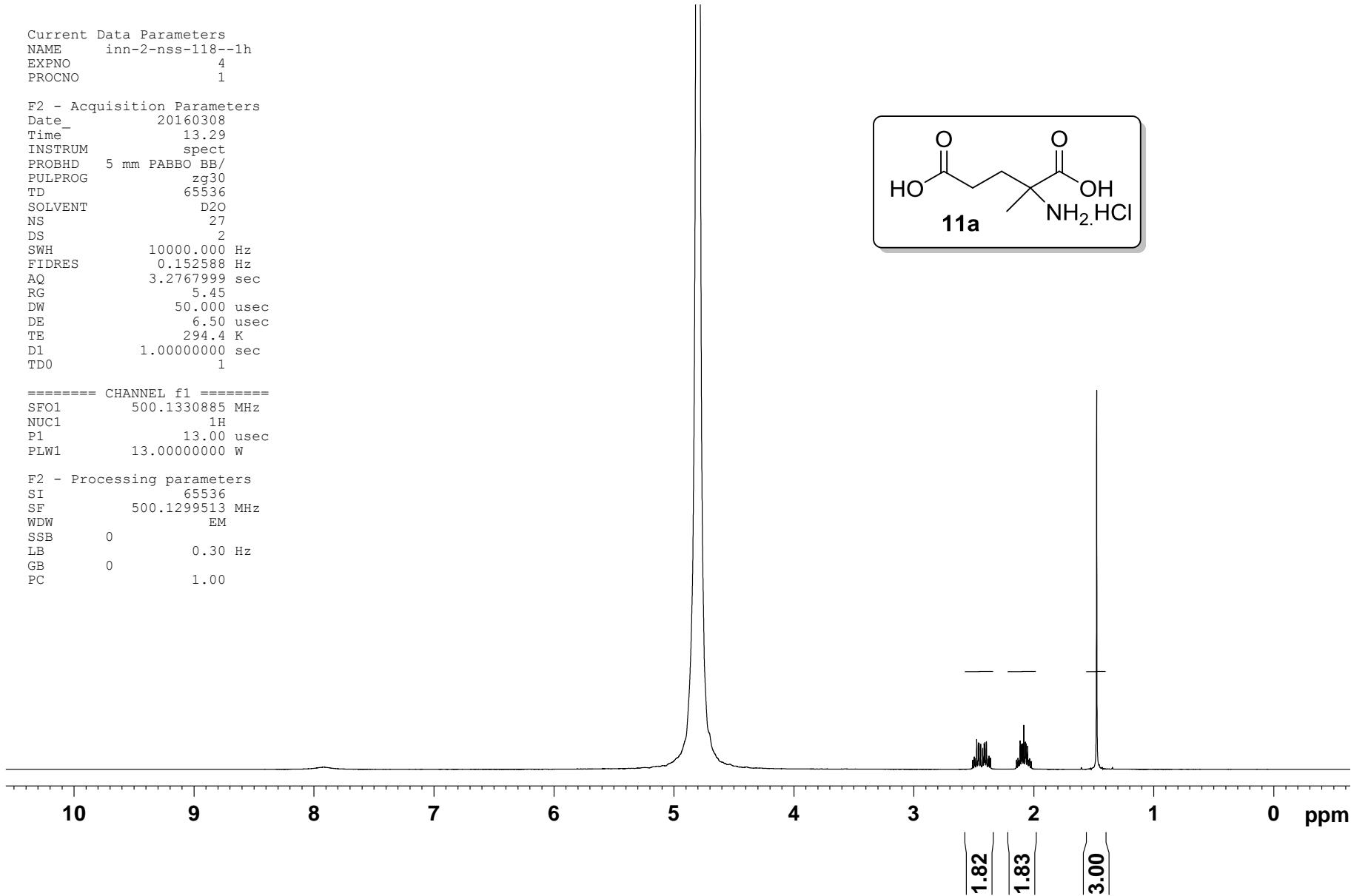
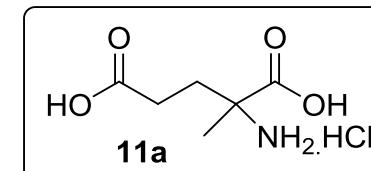
Index	Name	Time [Min]	Quantity [% Area]	Height [μV]	Area [μV.Mln]	Area % [%]
1	UNKNOWN	23.265	84.41	810197.7	514510.2	84.412
2	UNKNOWN	25.265	15.59	177042.6	95015.5	15.588
Total			100.00	987240.2	609525.7	100.000

Fig S136. HPLC Profile of Enantioenriched 9

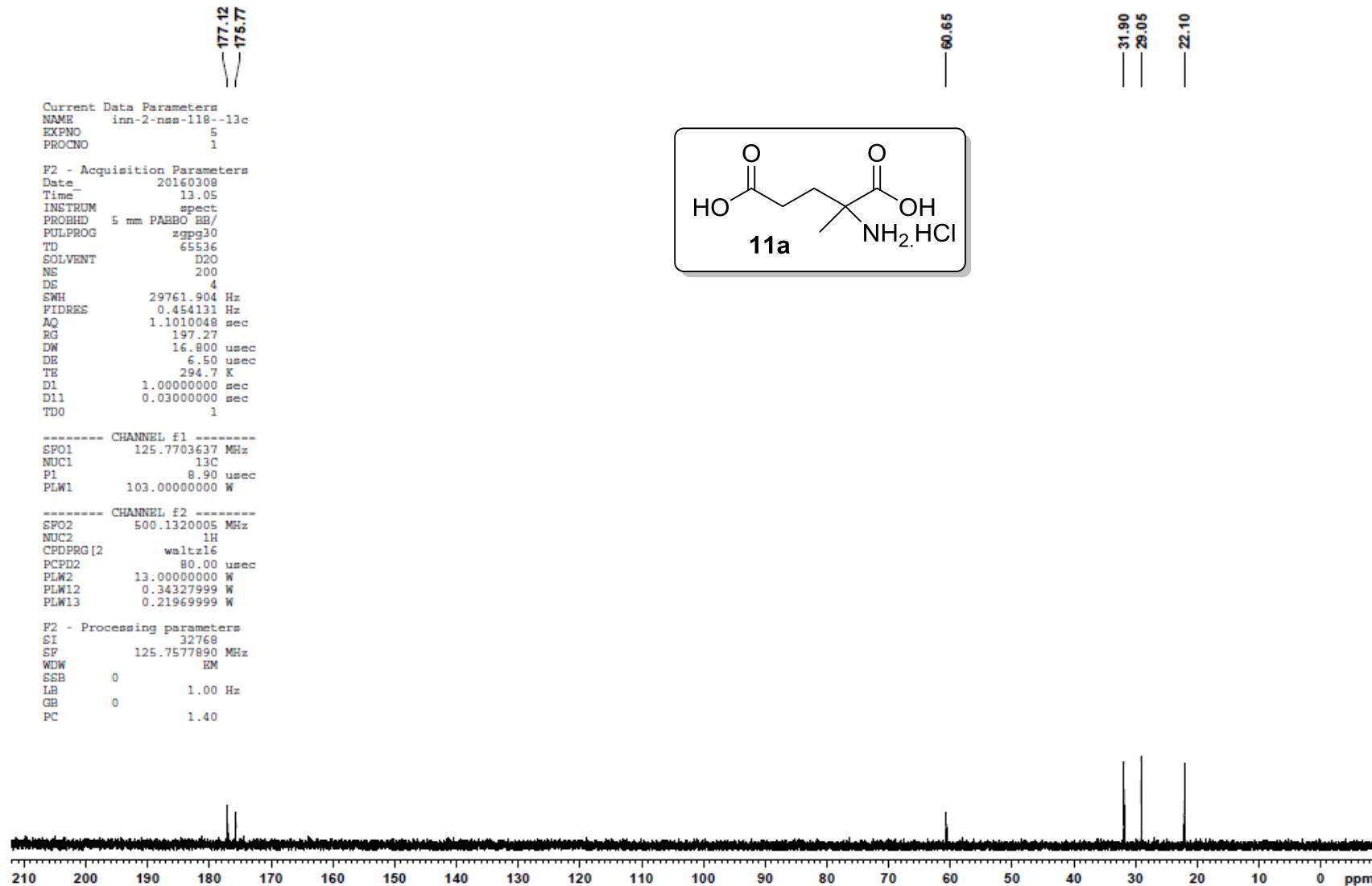
Current Data Parameters  
 NAME inn-2-nss-118--1h  
 EXPNO 4  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date 20160308  
 Time 13.29  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT D2O  
 NS 27  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.152588 Hz  
 AQ 3.2767999 sec  
 RG 5.45  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 294.4 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 SFO1 500.1330885 MHz  
 NUC1 1H  
 P1 13.00 usec  
 PLW1 13.0000000 W

F2 - Processing parameters  
 SI 65536  
 SF 500.1299513 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



**Fig S137.** <sup>1</sup>H NMR Spectrum of 11a



**Fig S138.**  $^{13}\text{C}$  NMR Spectrum of 11a

Current Data Parameters  
NAME inn-2-nss-137-b-1h  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160508  
Time 16.00  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 6  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 294.8 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 6.75 usec  
PL1 -3.00 dB  
PL1W 16.73965454 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

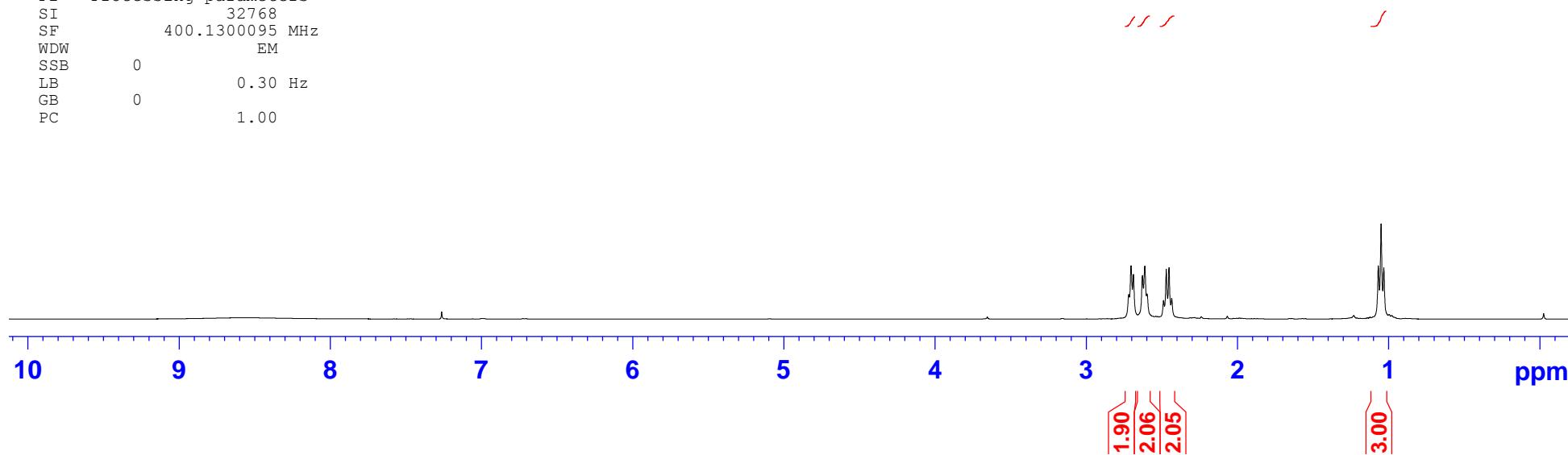
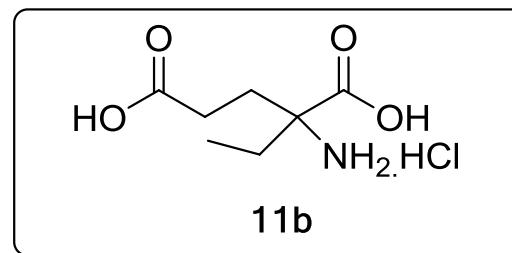


Fig S139. <sup>1</sup>H NMR Spectrum of 11b

Current Data Parameters  
NAME inn-2-nss-137--b-13c  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date 20160508  
Time 16.07  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zgpg30  
TD 65536  
SOLVENT D2O  
NS 217  
DS 4  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2582912 sec  
RG 28.5  
DW 19.200 usec  
DE 6.50 usec  
TE 295.3 K  
D1 1.0000000 sec  
D11 0.0300000 sec  
TD0 1

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

===== CHANNEL f2 =====  
CPDPKG[2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127690 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



Fig S140. <sup>13</sup>C NMR Spectrum of 11b

Current Data Parameters  
NAME INN-4-KSB-98B-1H  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date 20130728  
Time 23.05  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 10  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9845889 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 295.9 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 13.50 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

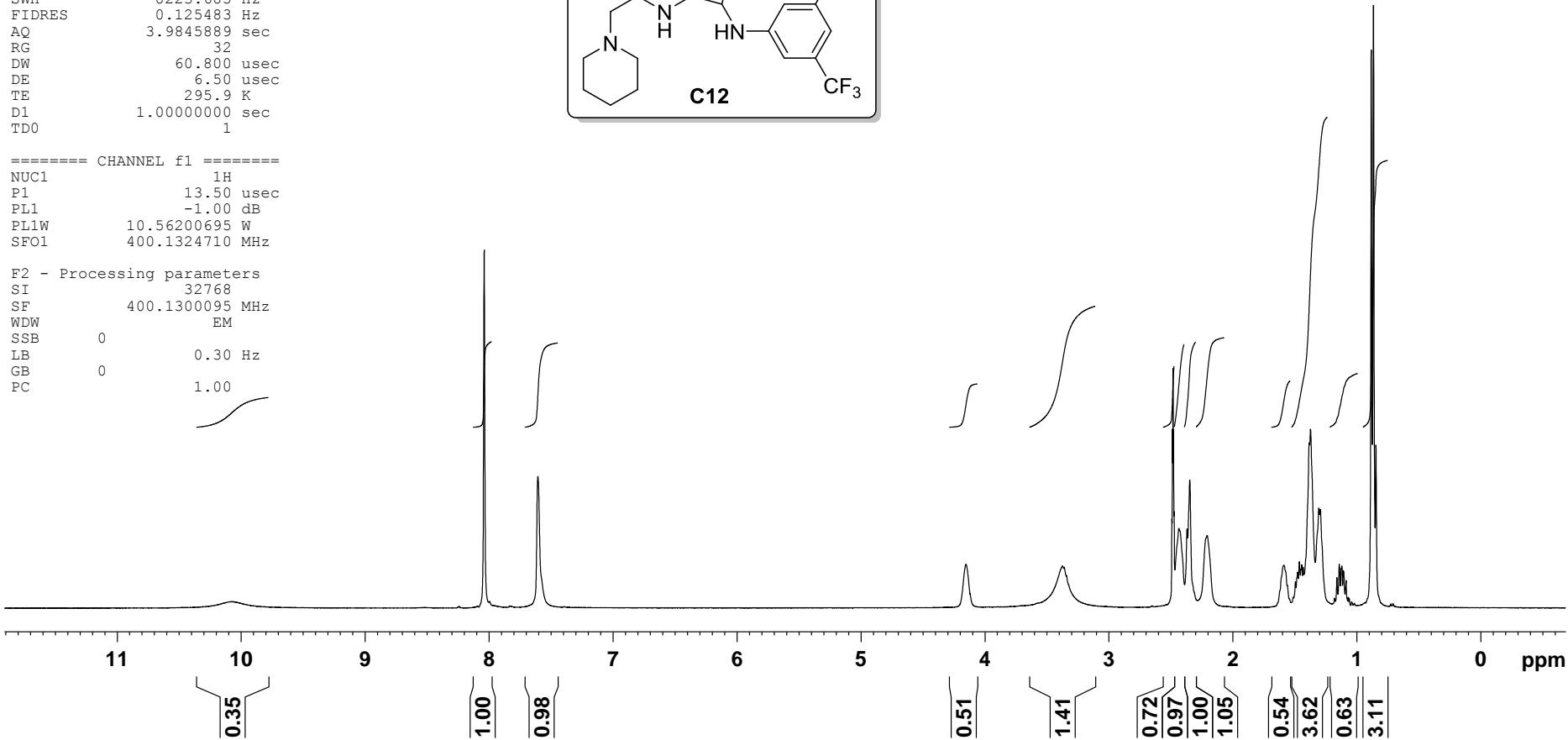
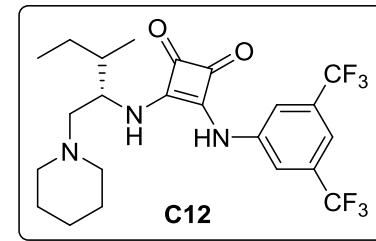


Fig S141. <sup>1</sup>H NMR Spectrum of C12

Current Data Parameters  
 NAME INN-4-KSB-98-B-13C  
 EXPNO 9  
 PROCNO 1  
 F2 - Acquisition parameters  
 Date\_ 20130730  
 Time 12:48  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT DMSO  
 NS 1586  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 2050  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 295.7 K  
 D1 1.0000000 sec  
 D11 0.0300000 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 =====

CPDPRG[2 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

F2 - Processing parameters

SI 32768  
 SF 100.6128116 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

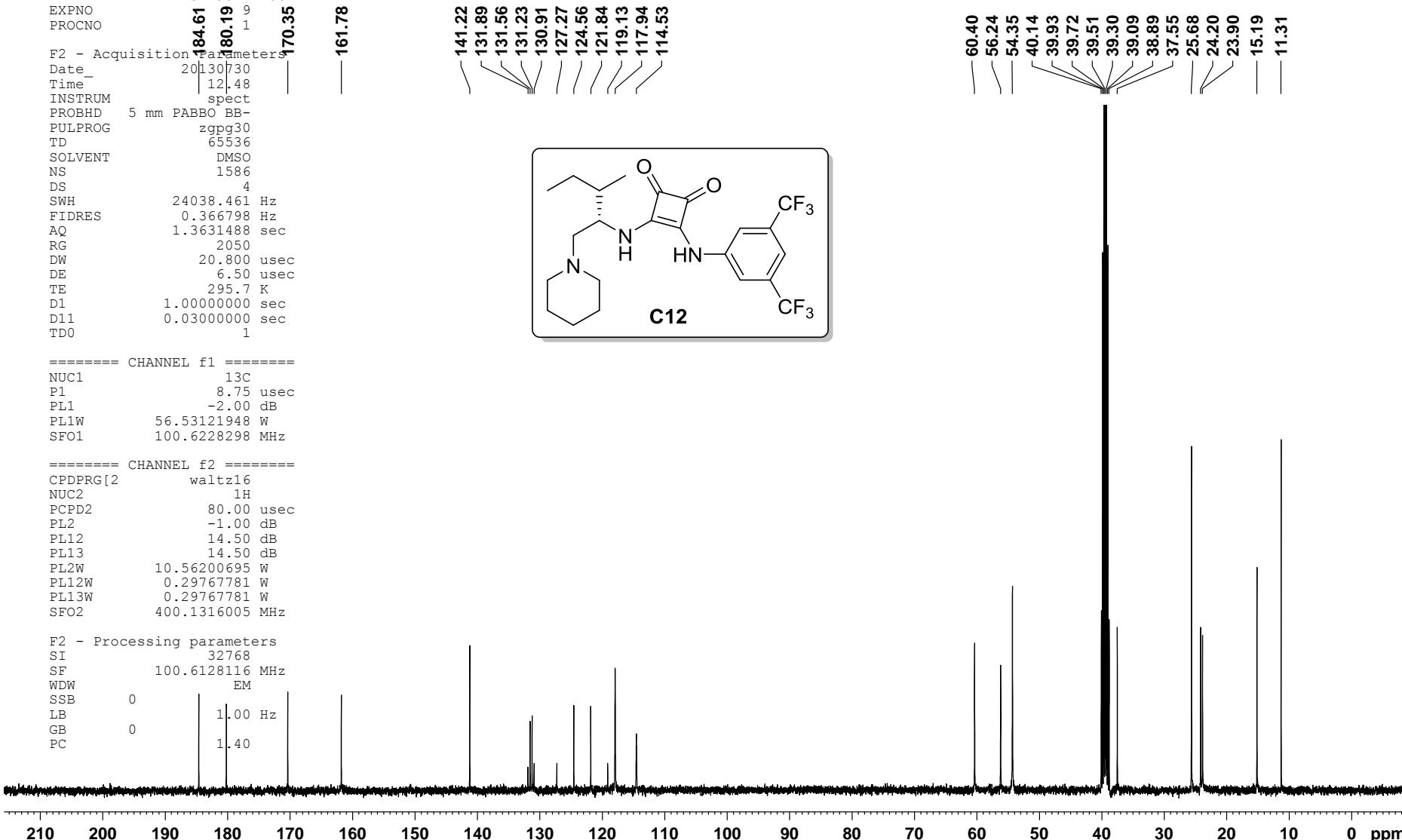


Fig S142. <sup>13</sup>C NMR Spectrum of C12

Current Data Parameters  
NAME INN-4-KSB-99B-1H  
EXPNO 10  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20130728  
Time 23.17  
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 3.9845889 sec  
RG 32  
DW 60.800 usec  
DE 6.50 usec  
TE 295.6 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

## F2 - Processing parameters

SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

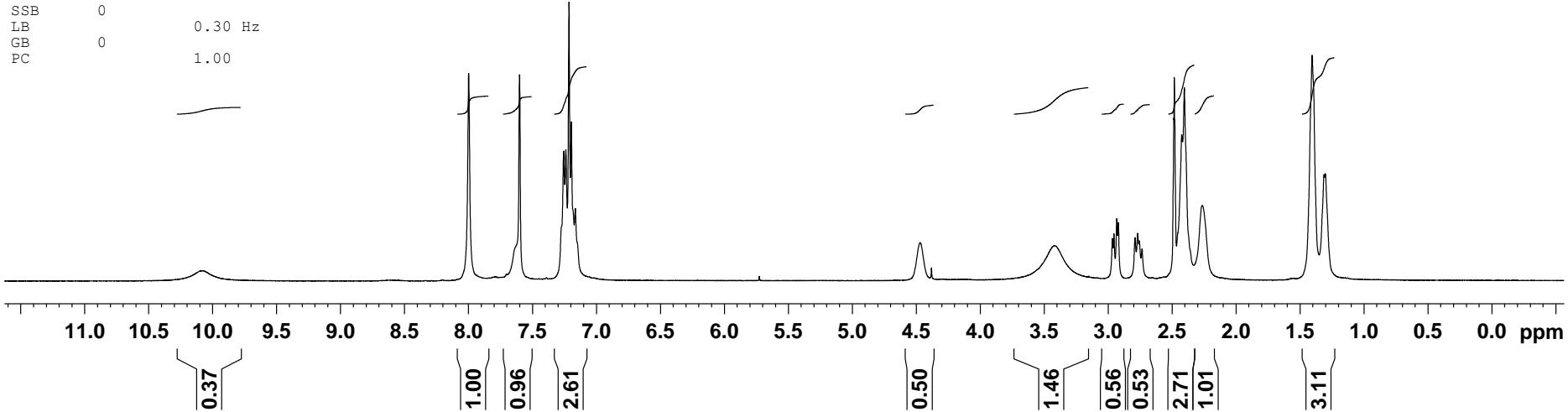
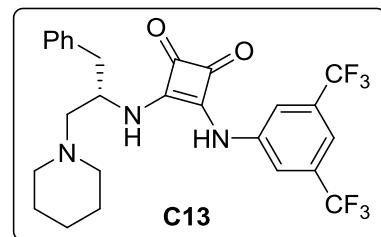


Fig S143. <sup>1</sup>H NMR Spectrum of C13

Current Data Parameters  
NAME inn-4-ksb-d99b-13c  
EXPNO 8  
PROCNO

F2 - Acquisition Parameters  
Date\_ 20130809  
Time 4.43  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT DMSO  
NS 7876  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 2050  
DW 20.800 usec  
DE 6.50 usec  
TE 295.1 K  
D1 1.0000000 sec  
D11 0.0300000 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 ======  
CPDPRG[2 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

F2 - Processing parameters  
SI 32768  
SF 100.6128109 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 0.80



Fig S144.  $^{13}\text{C}$  NMR Spectrum of C13

Current Data Parameters  
NAME INN-4-KSB-99A-1H  
EXPNO 14  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20140106  
Time 23.47  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 69  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 69.35  
DW 50.000 usec  
DE 6.50 usec  
TE 298.1 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.00 usec  
PLW1 13.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300041 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

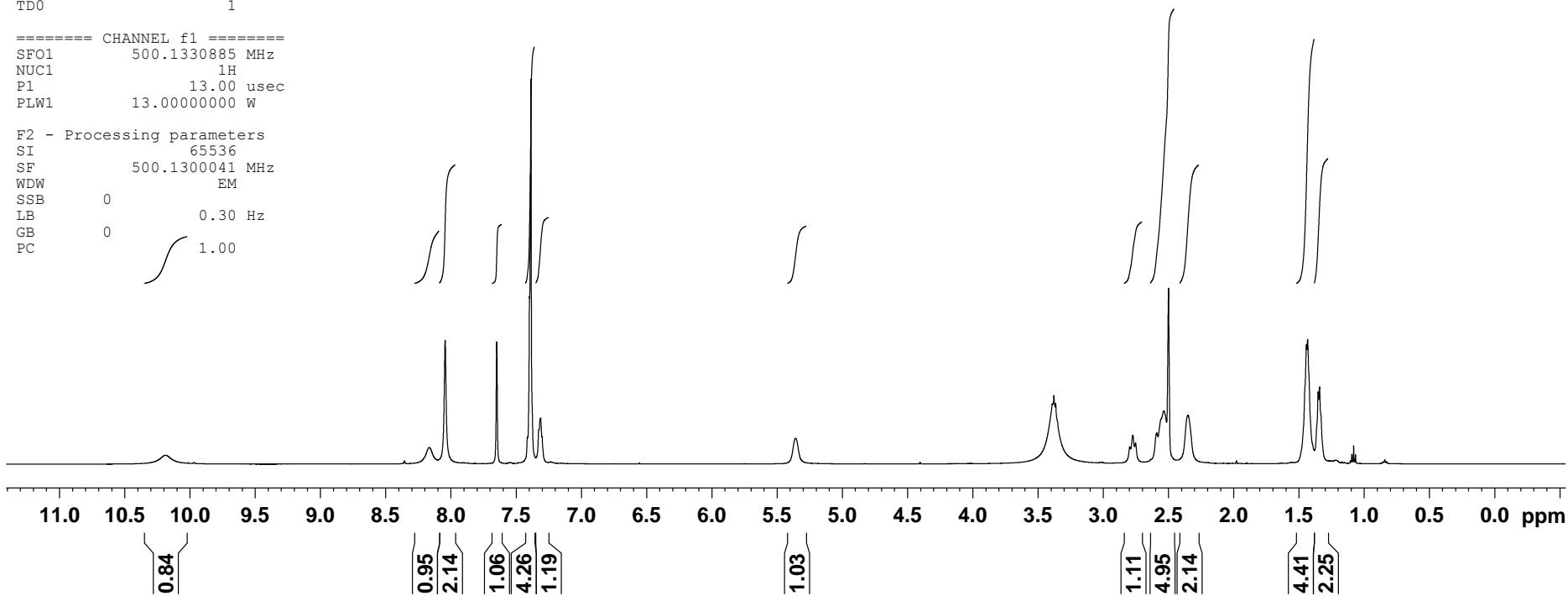
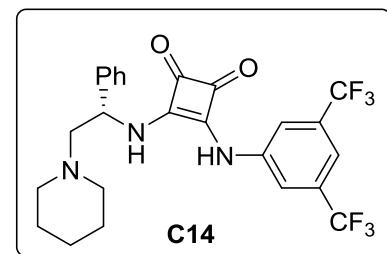


Fig S145. <sup>1</sup>H NMR Spectrum of C14

Current Data Parameters  
 NAME INN-4-KSB-99A-13C  
 EXPNO 11  
 PROCNO 1  
 F2 - Acquisition parameters  
 Date 20140104  
 Time 6.00  
 INSTRUM spect  
 PROBHD 5 mm SEI 1H/D-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT DMSO  
 NS 15336  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 114  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 297.1 K  
 D1 1.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

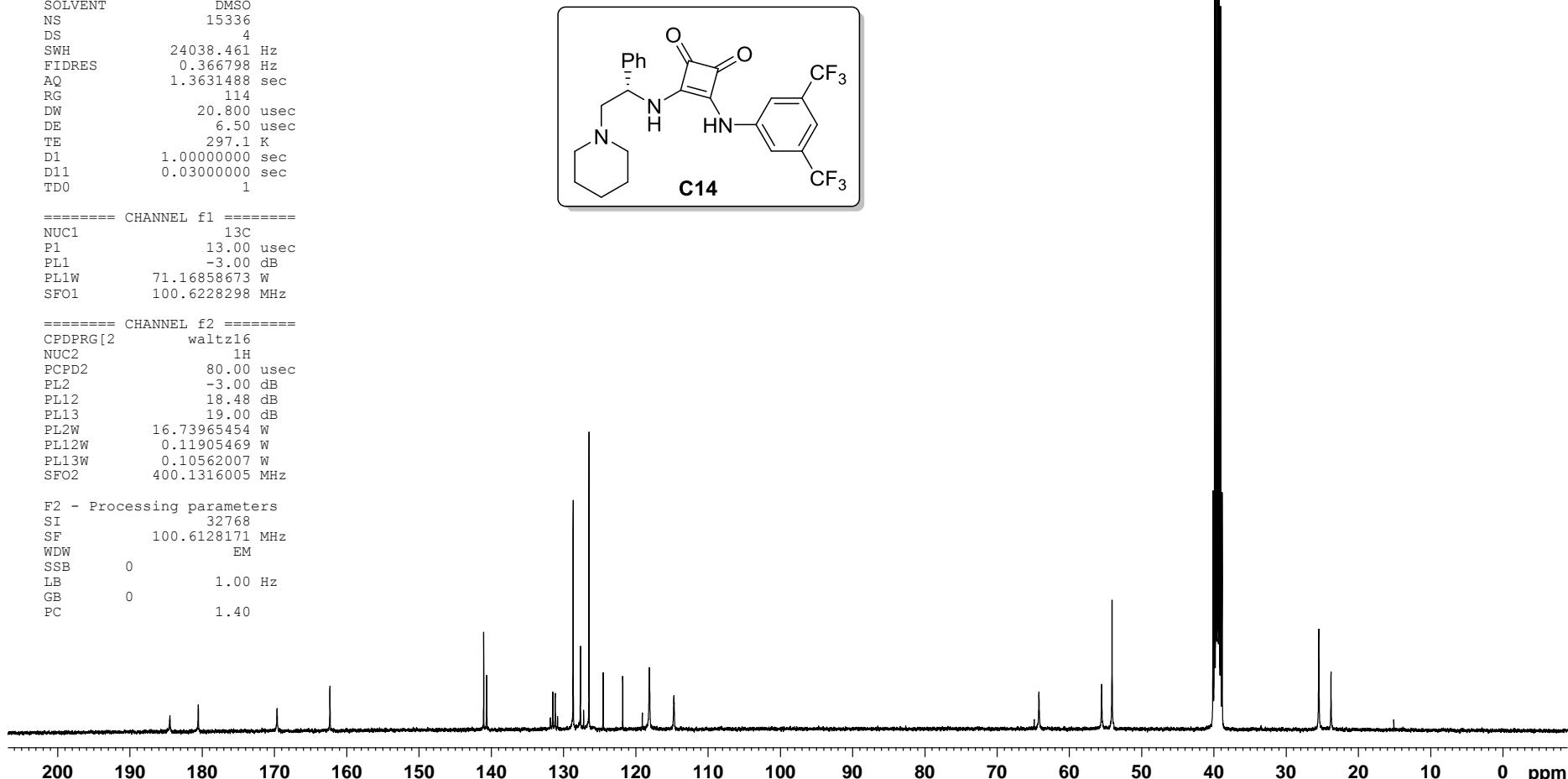
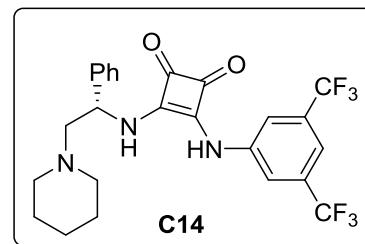
===== CHANNEL f1 =====

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

===== CHANNEL f2 =====

CPDPRG[2 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

F2 - Processing parameters  
 SI 32768  
 SF 100.6128171 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



**Fig S146.**  $^{13}\text{C}$  NMR Spectrum of C14