

Facile Synthesis of Functionalized Spiropyrrolizidine Oxindoles via a Three-Component Tandem Cycloaddition Reaction

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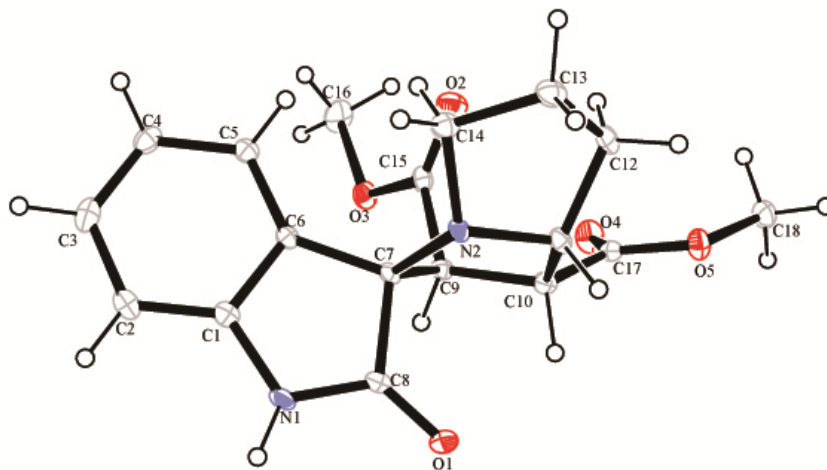
1. Crystallographic data and molecular structure.....	2
2. ¹ H, ¹³ C NMR Spectra.....	3-20

1. Crystallographic data and molecular structure

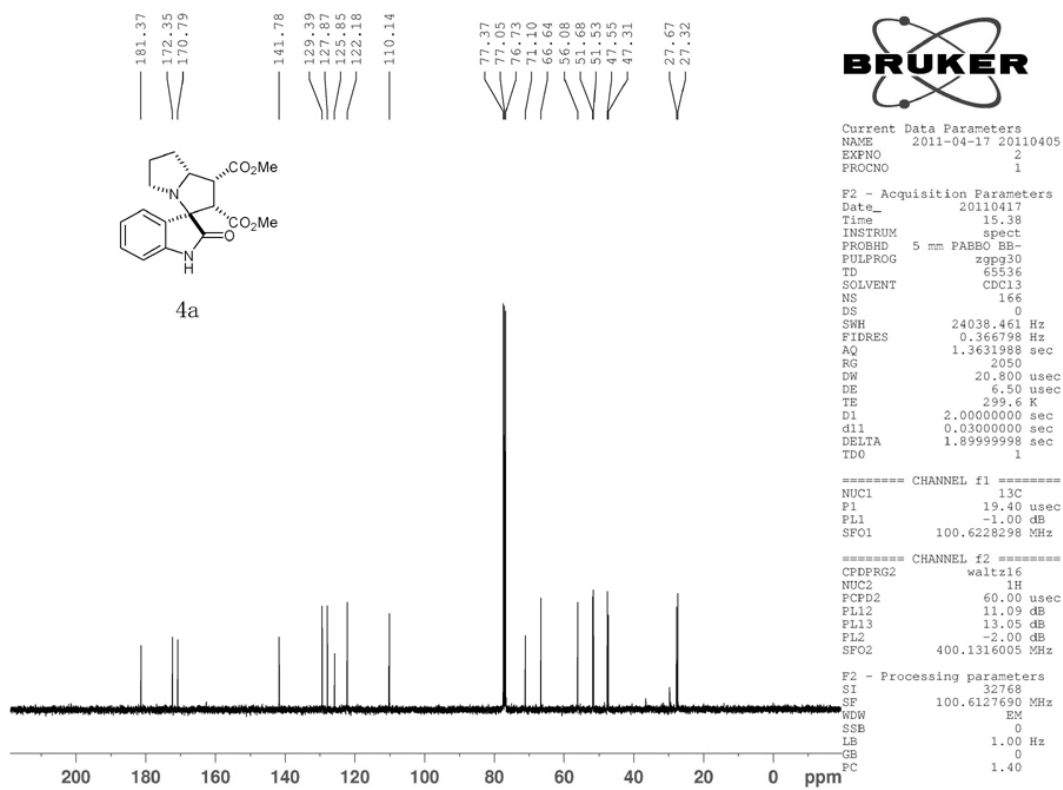
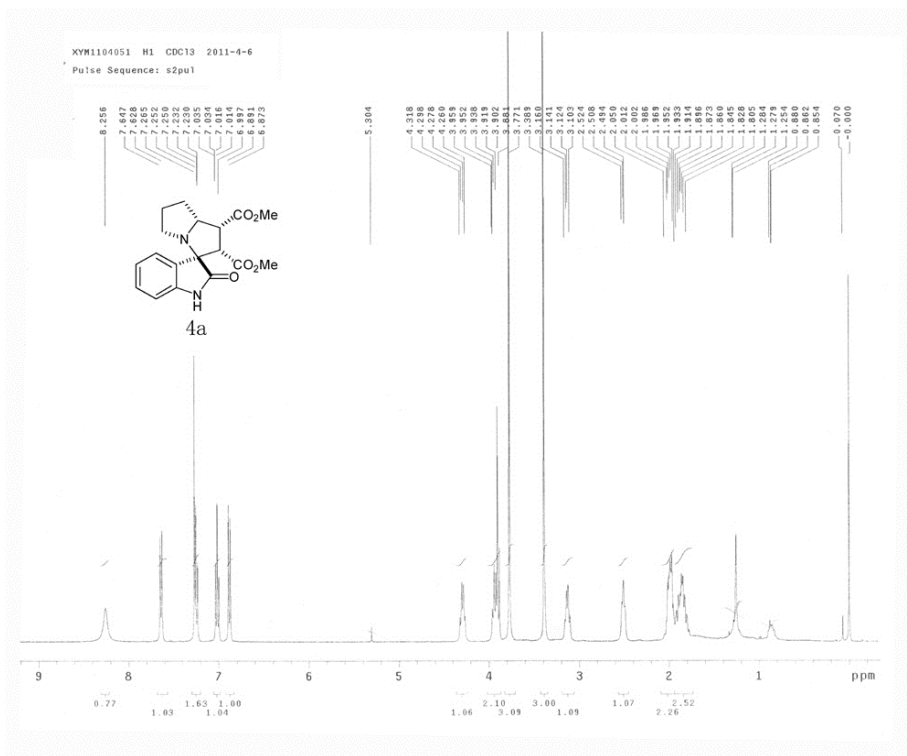
Crystal structure data for compound 4a: CCDC 828257.

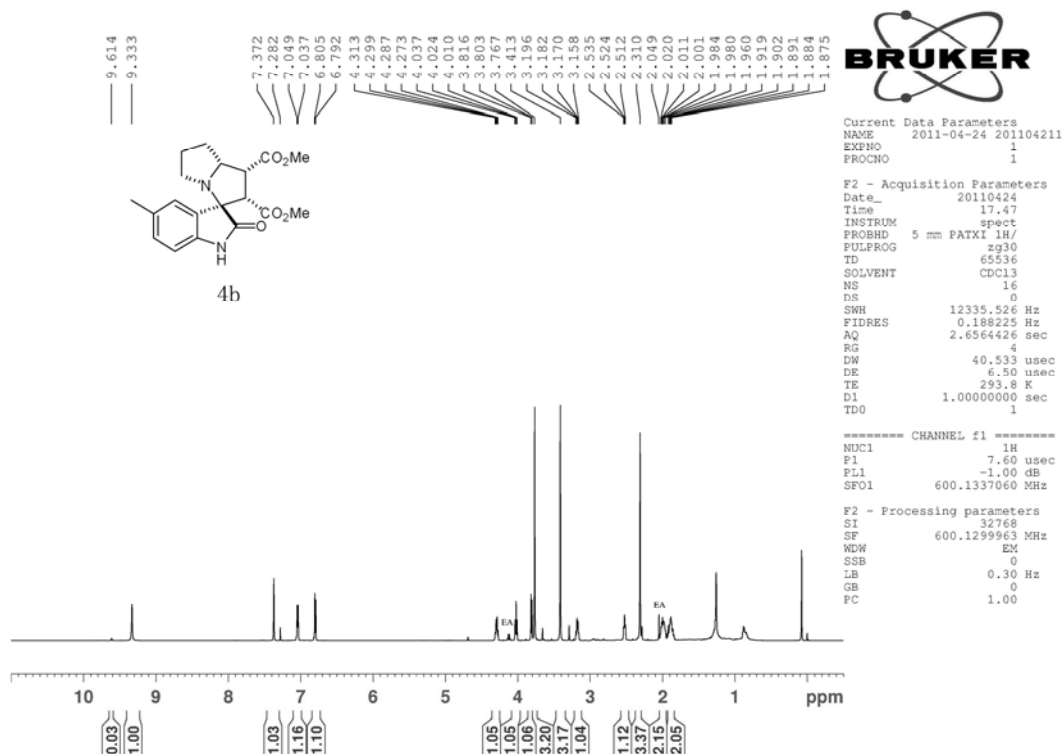
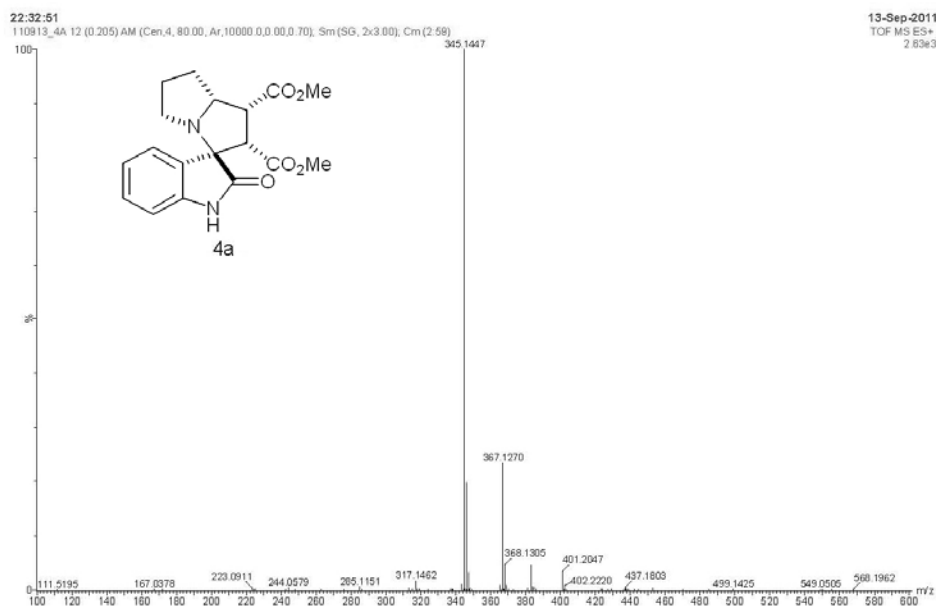
$C_{18}H_{20}N_2O_5$, chemical formula weight: 344.14, monoclinic, space group $P2_1/c$, $a = 7.5912(6)$, $b = 27.380(2)$, $c = 7.8522(6)$ Å; $\alpha = 90.00^\circ$, $\beta = 95.729(8)^\circ$, $\gamma = 90.00^\circ$, $U = 1623.9(2)$ Å³, $T = 145.0$ K, $Z = 4$, $\rho = 1.413$ mg/mm³, $\mu = 0.104$ mm⁻¹, $F(000)732$, crystal size $0.30 \times 0.25 \times 0.20$ mm³, 5840 independent reflections [$R(\text{int}) = 0.0000$], reflections collected 5840, refinement method: full-matrix least-squares on F^2 : goodness-of-fit on F^2 0.721, final R indexes [$I > 2\sigma(I)$], $R_1 = 0.0468$, $wR_2 = 0.0683$, largest diff. peak and hole 0.243 Å⁻³ and -0.246 eÅ⁻³.

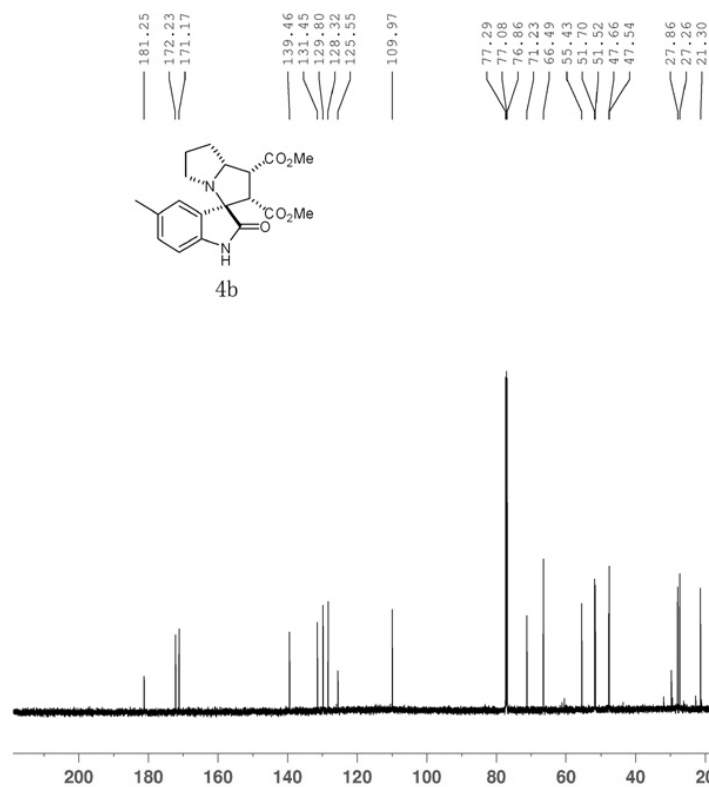
Crystal Structure of 4a:



2. ^1H , ^{13}C NMR Spectra







Current Data Parameters
 NAME 2011-04-24 201104211
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 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110424
 Time 17.54
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 140
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9088159 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 294.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

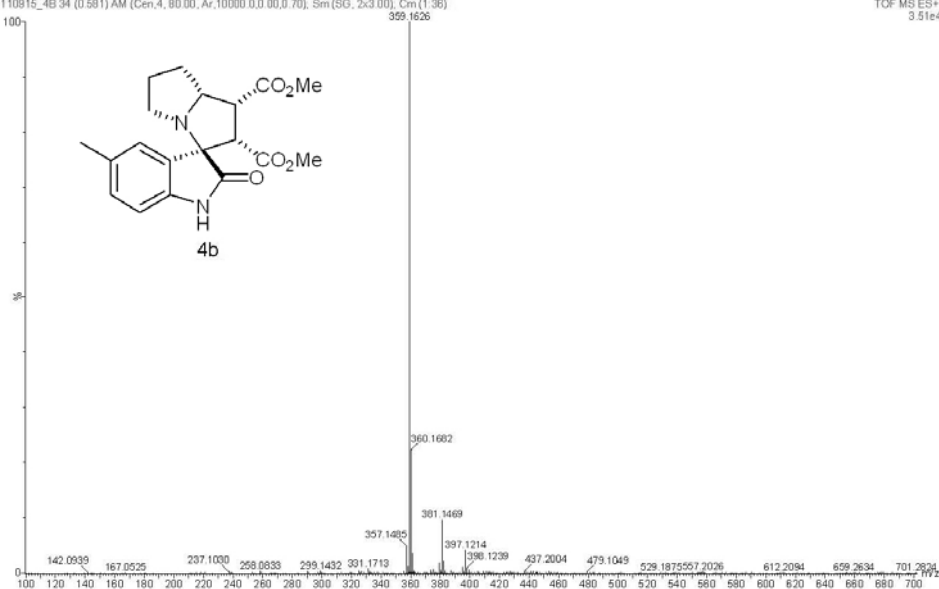
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 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 150.9178988 MHz

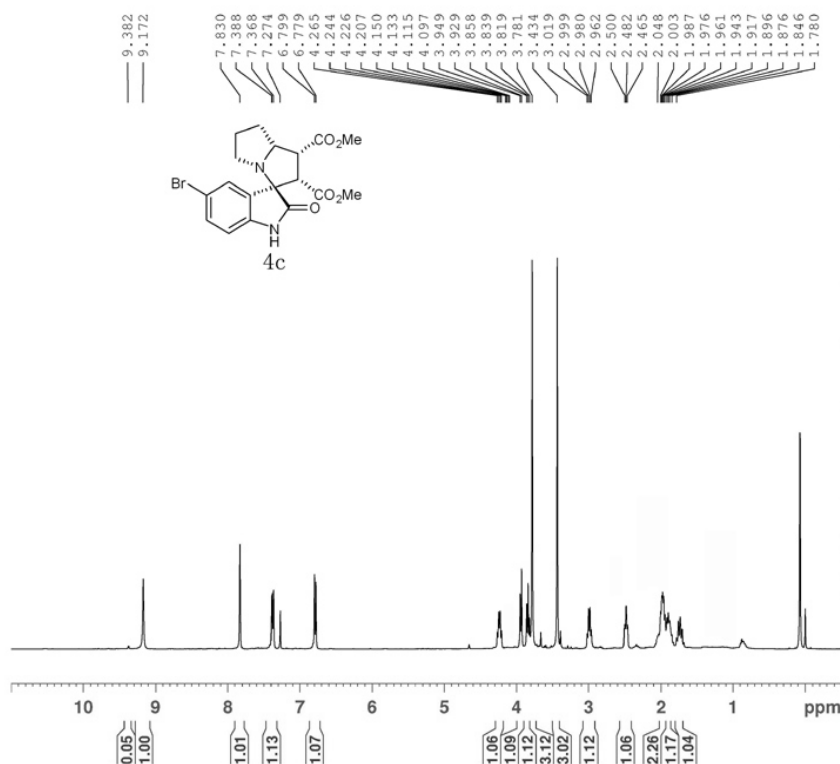
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 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.45 dB
 PL13 19.50 dB
 PL2 -1.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

13:16:01
 110915_4B 34 (0.591) AM (Cen.4, 80.00, Ar.10000 0.0 00.0 70), Sm (SG, 2x3.00), Cm (1.36)

15-Sep-2011
 TOF MS ES+
 3.51e4



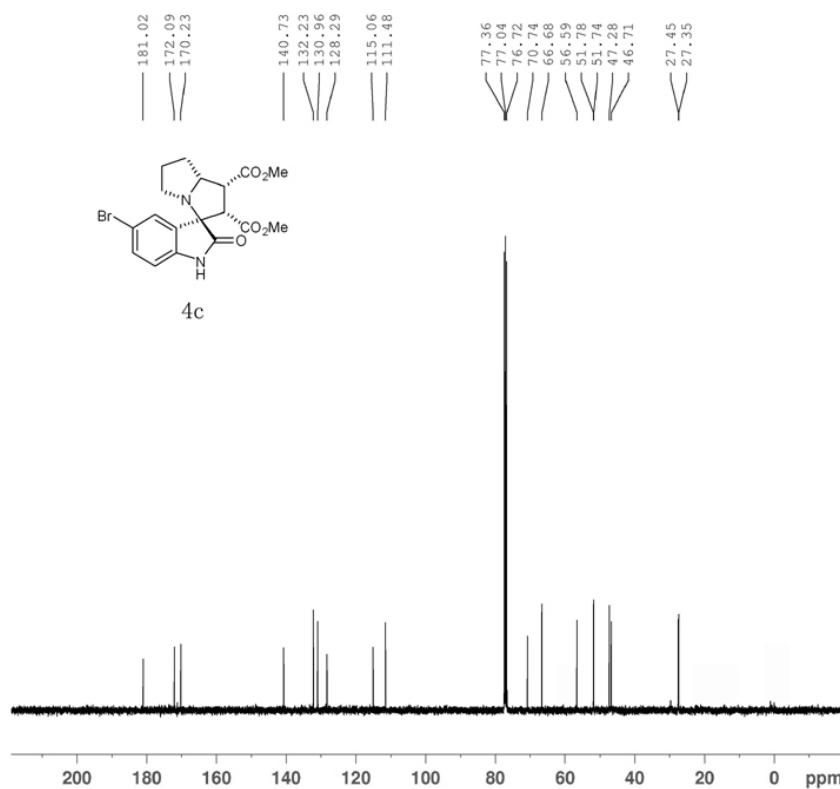


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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 14.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 2011-04-17 201104132
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110417
Time 14.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 239
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 19.40 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

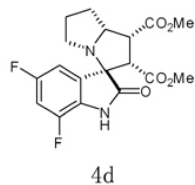
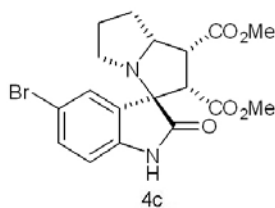
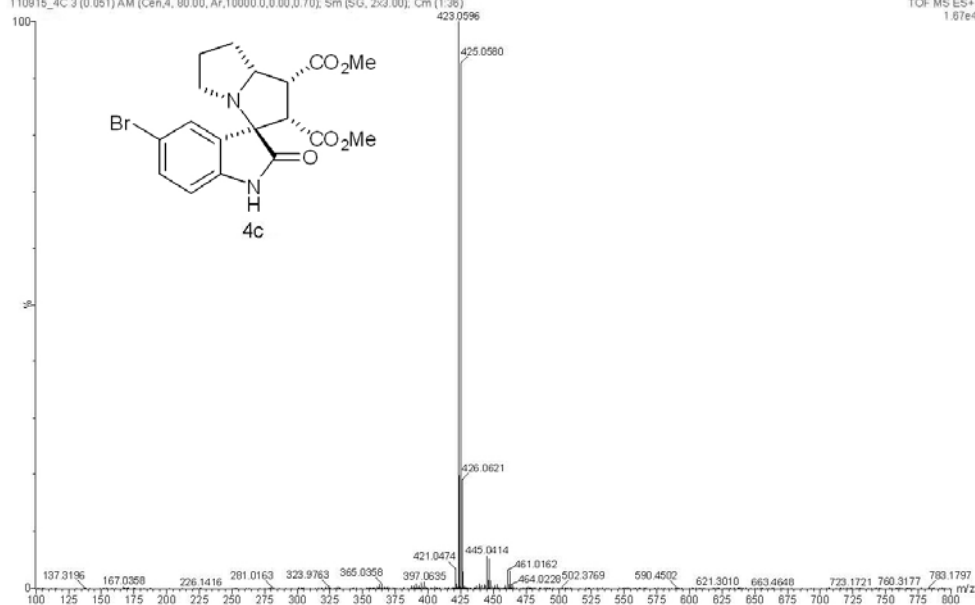
===== CHANNEL f2 =====
CFDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.09 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

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

13:07:31

110915_4C-3 (0.051) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (1.38)

15-Sep-2011

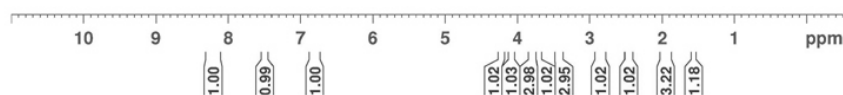
TOF MS ES+
1.87e4

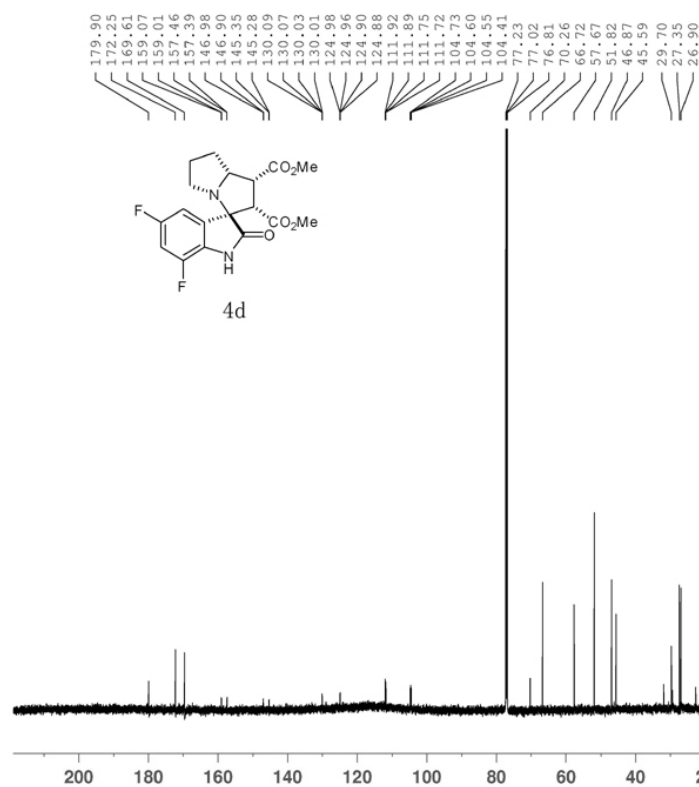
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110424
Time 19.19
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.656426 sec
RG 4
DW 40.533 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.60 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00





Current Data Parameters
 NAME 2011-04-24 201104191
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110424
 Time 19.41
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9088159 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 294.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

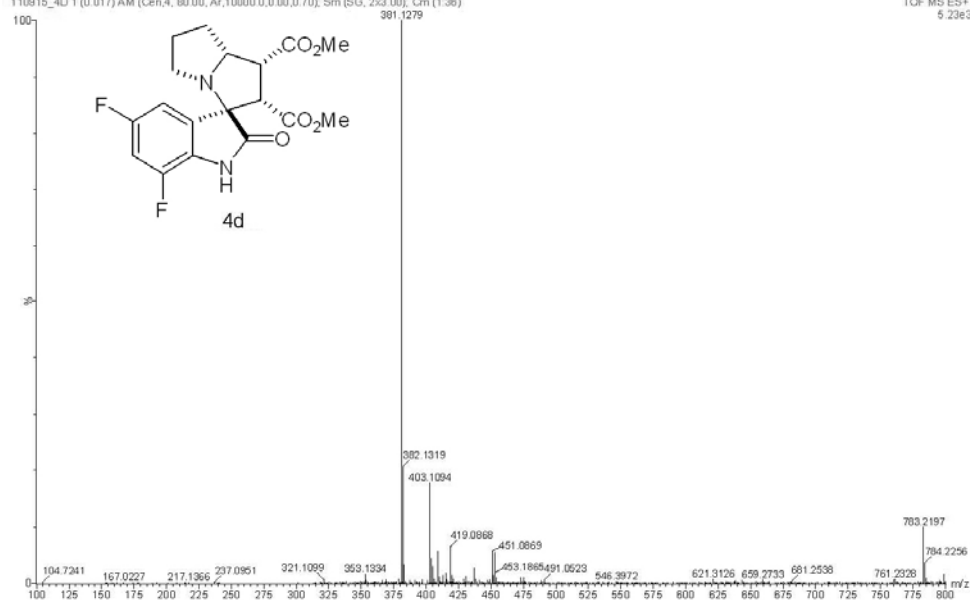
===== CHANNEL f1 =====
 NUC1 13C
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 150.9178988 MHz

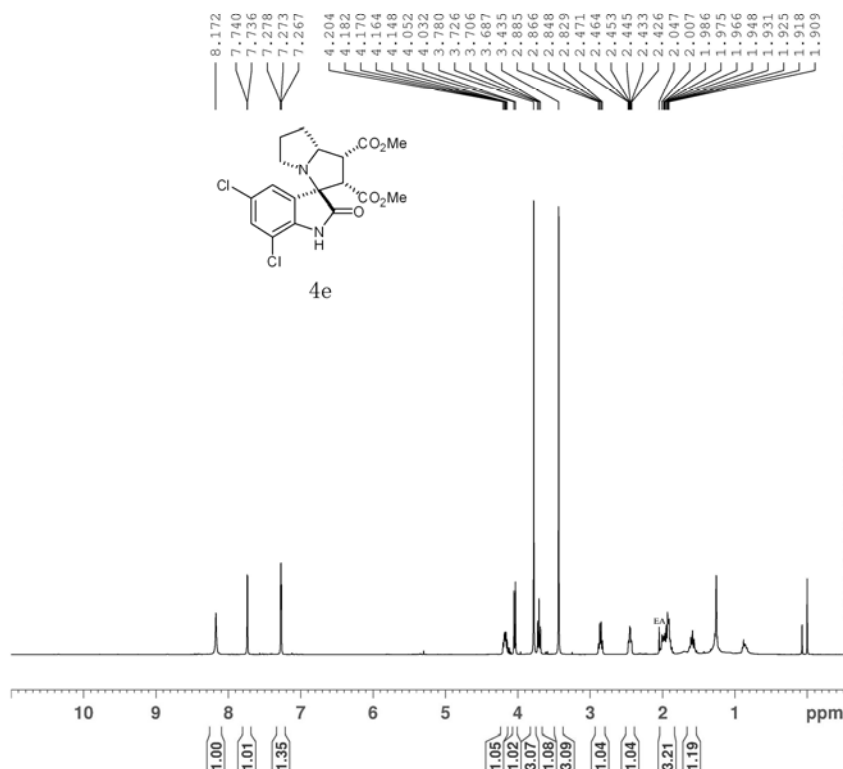
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.45 dB
 PL13 19.50 dB
 PL2 -1.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

12:59:10
 110815_4D 1 (0.017) AM (Cen:4, 60.00, Ar:10000.0, 0.00, 0.70); Sm (SG, 2x3 00); Cm (1.36)

15-Sep-2011
 TOF MS ES+
 5.23e3



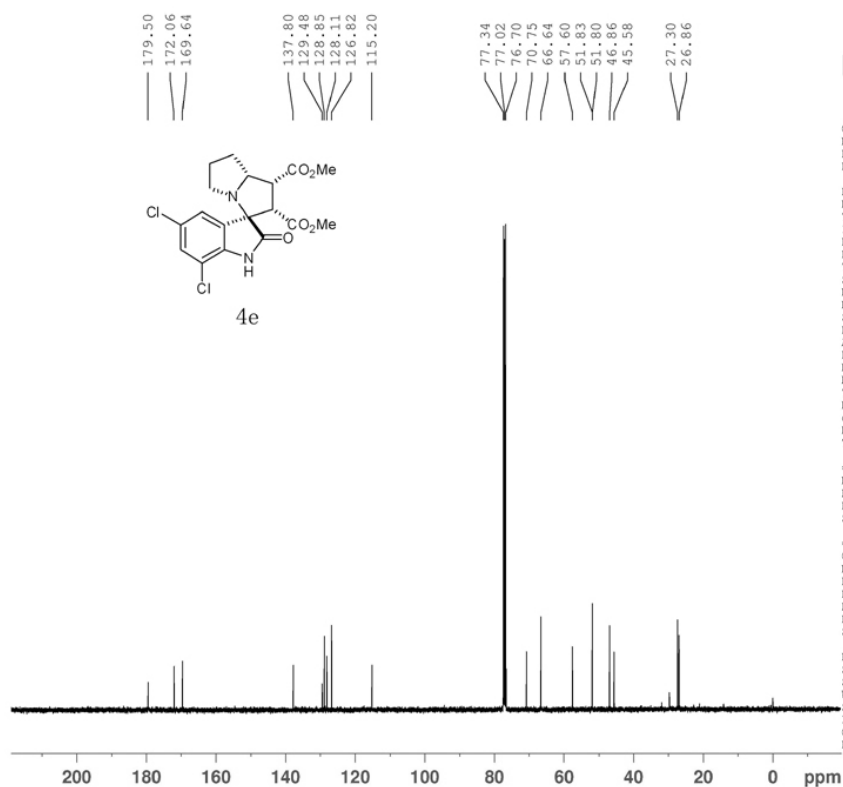


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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110417
Time 15.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300071 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 2011-04-17 201104135
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110417
Time 16.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

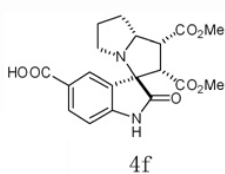
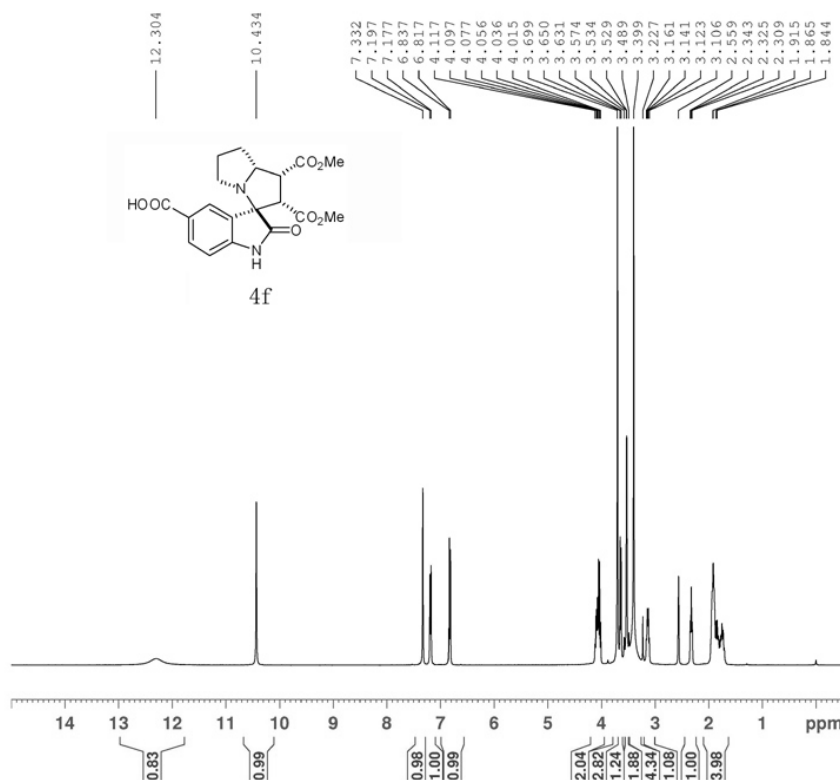
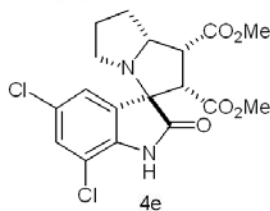
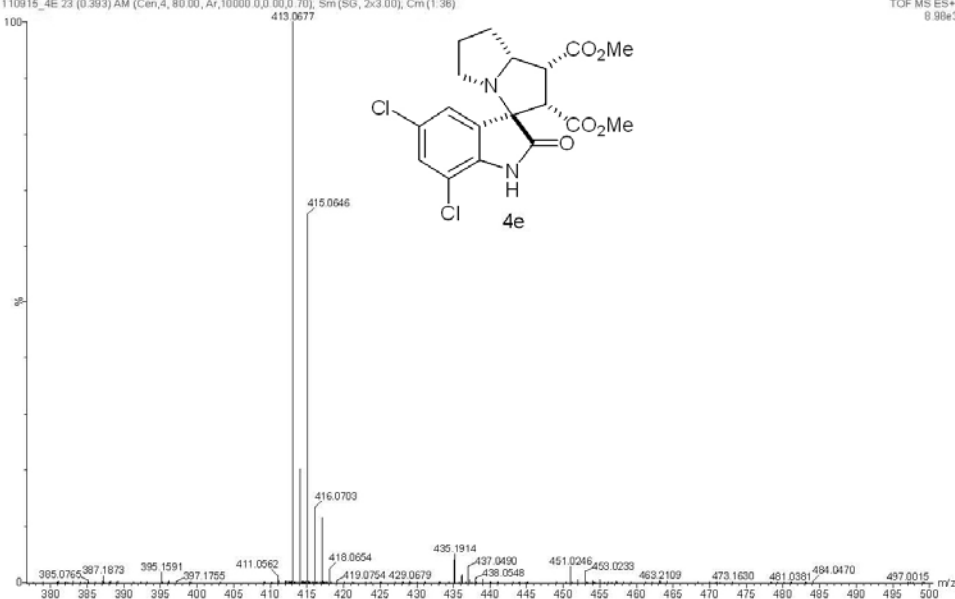
===== CHANNEL f1 =====
NUC1 13C
P1 19.40 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.09 dB
PL13 13.05 dB
PL2 -2.00 dB
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

14:05:32
110915_4E 23 (0.393) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Crn(1:36)

15-Sep-2011
TOF MS ES+
8.98e3

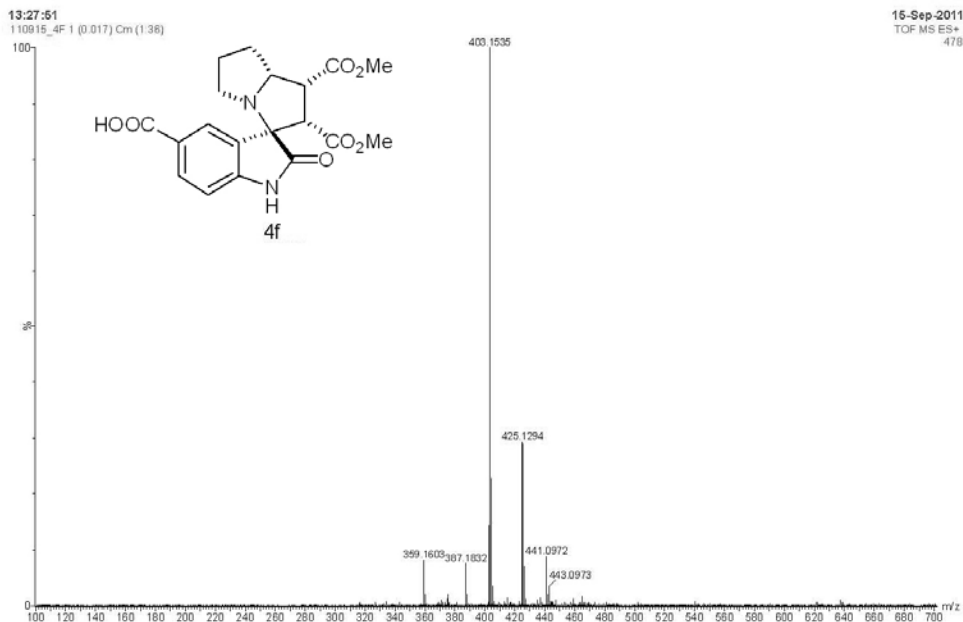
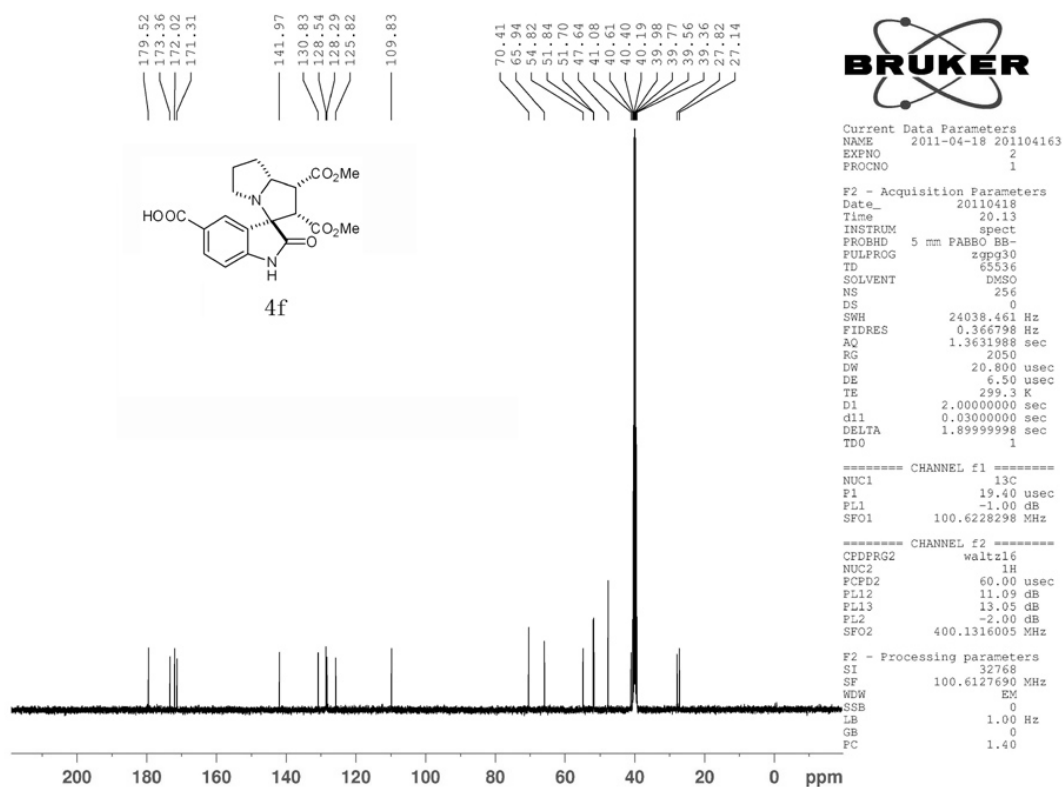


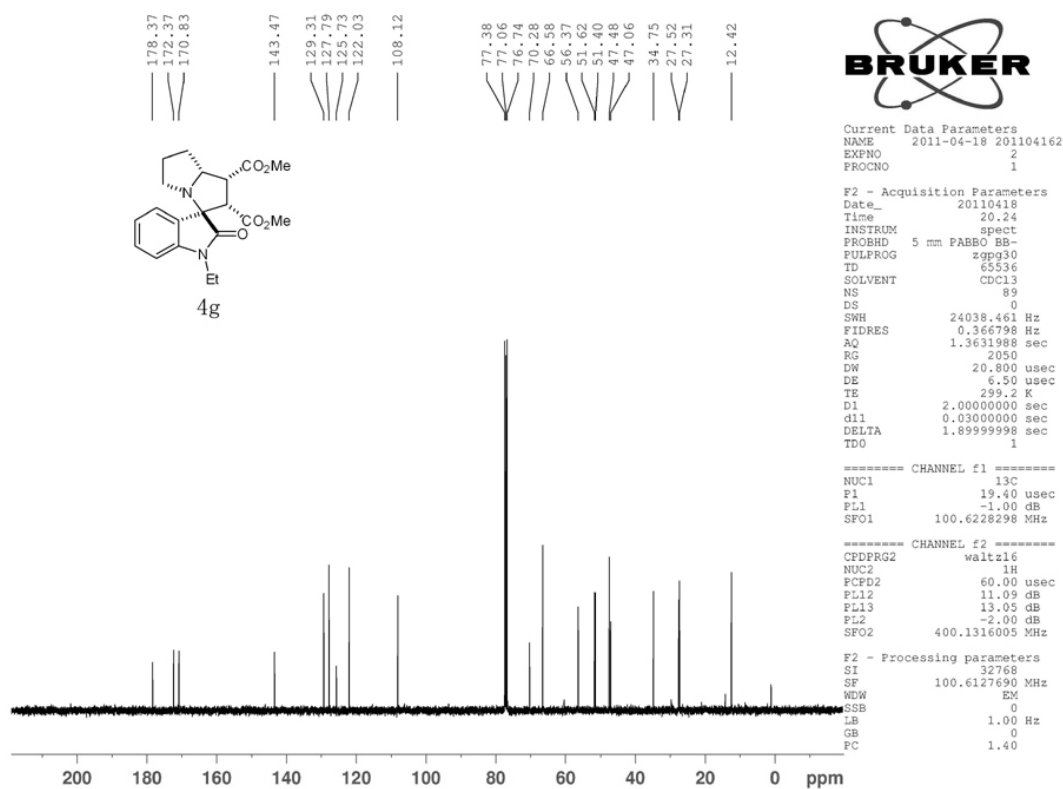
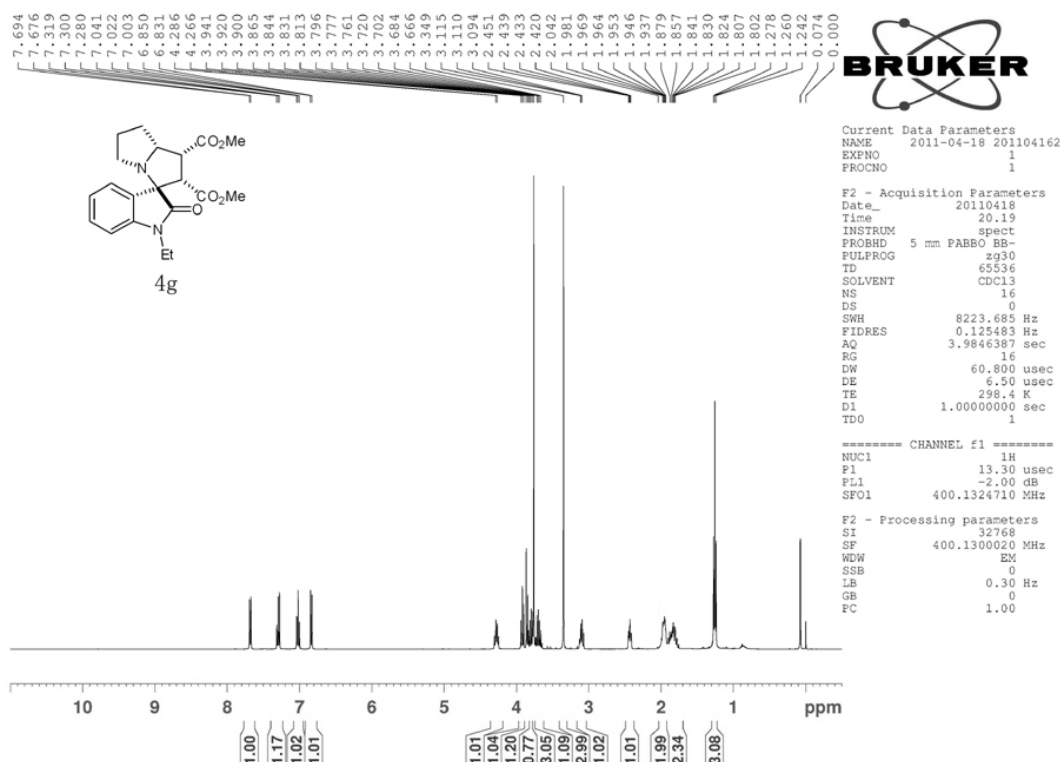
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 19.58
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299803 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00

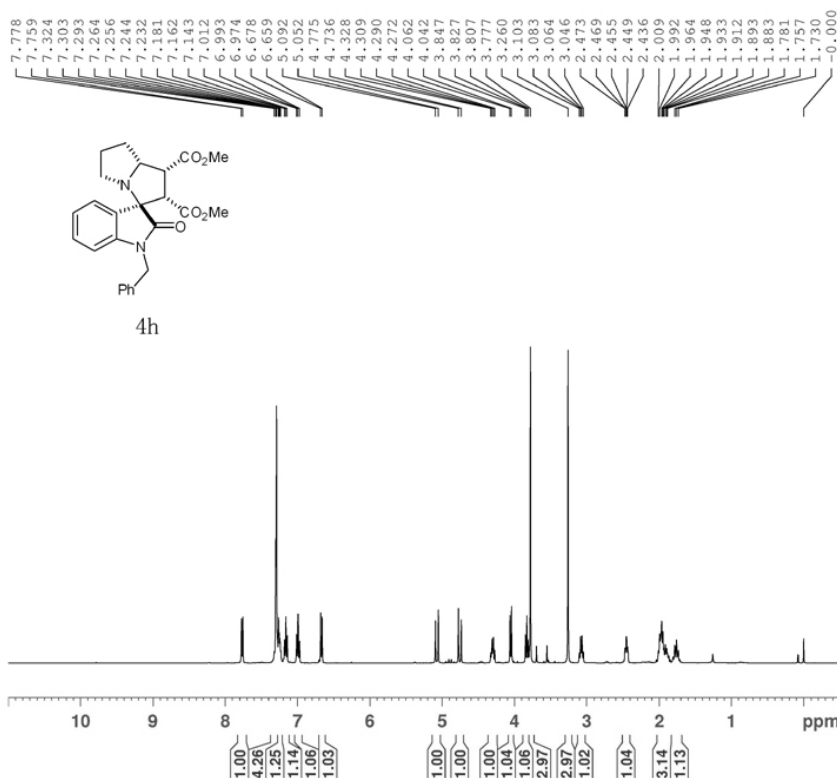
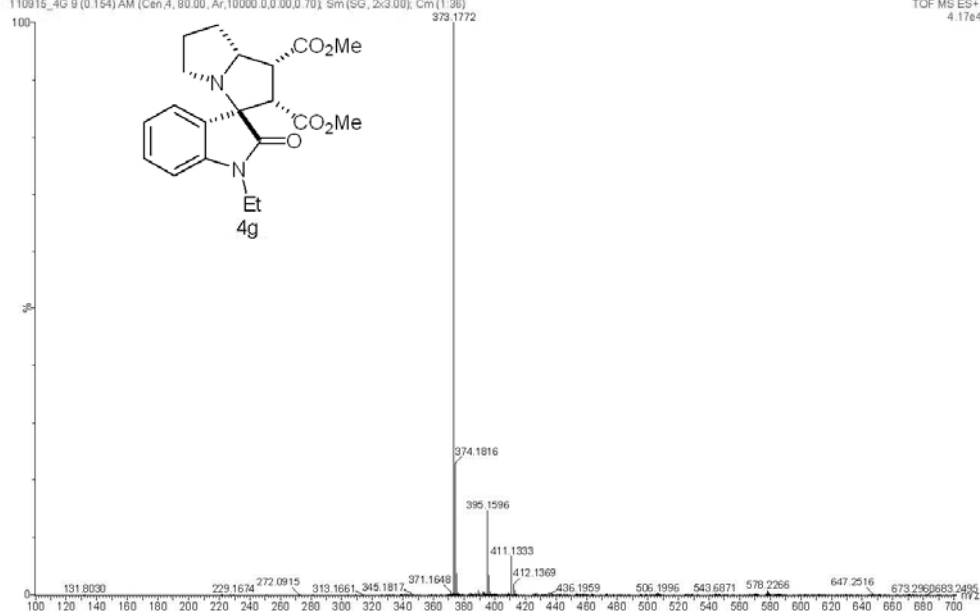




13:59:59

110915_4G 9 (0.154) AM (Cen 4, 80.00, Ar, 10000.0, 0.00, 0.70) Sm (SG, 2x2.00) Cm (1.36)

15-Sep-2011

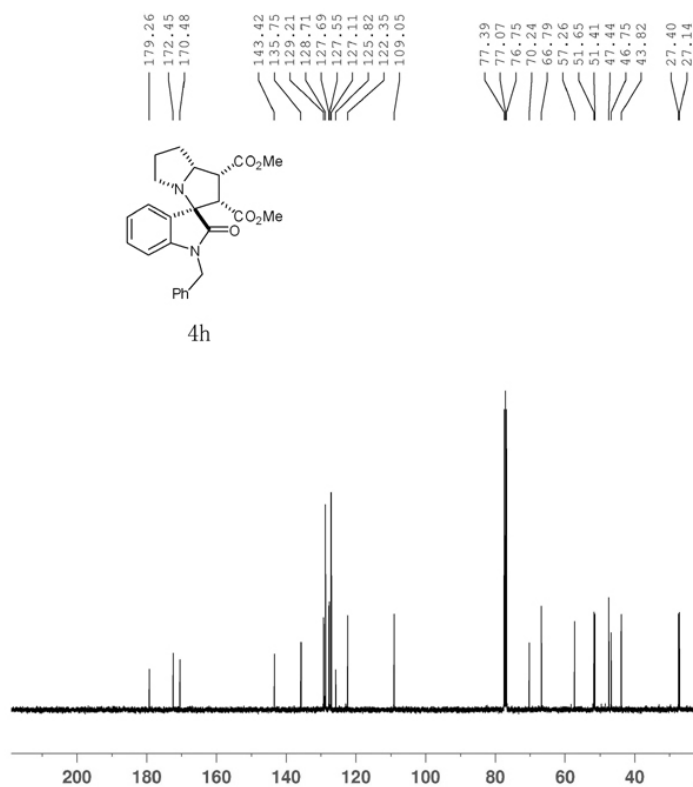
TQF MS ES+
4.17e4

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110417
Time 15.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00



Current Data Parameters
 NAME 2011-04-17 201104133
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110417
 Time 15.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 152
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.50 usec
 TE 299.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

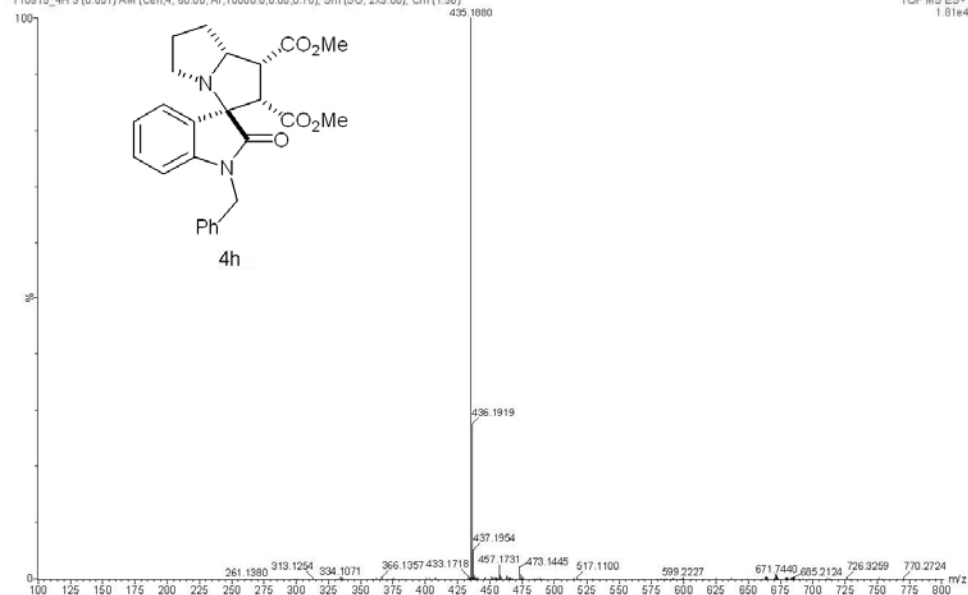
===== CHANNEL f1 =====
 NUC1 13C
 P1 19.40 usec
 PL1 -1.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 60.00 usec
 PL12 11.09 dB
 PL13 13.05 dB
 PL2 -2.00 dB
 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

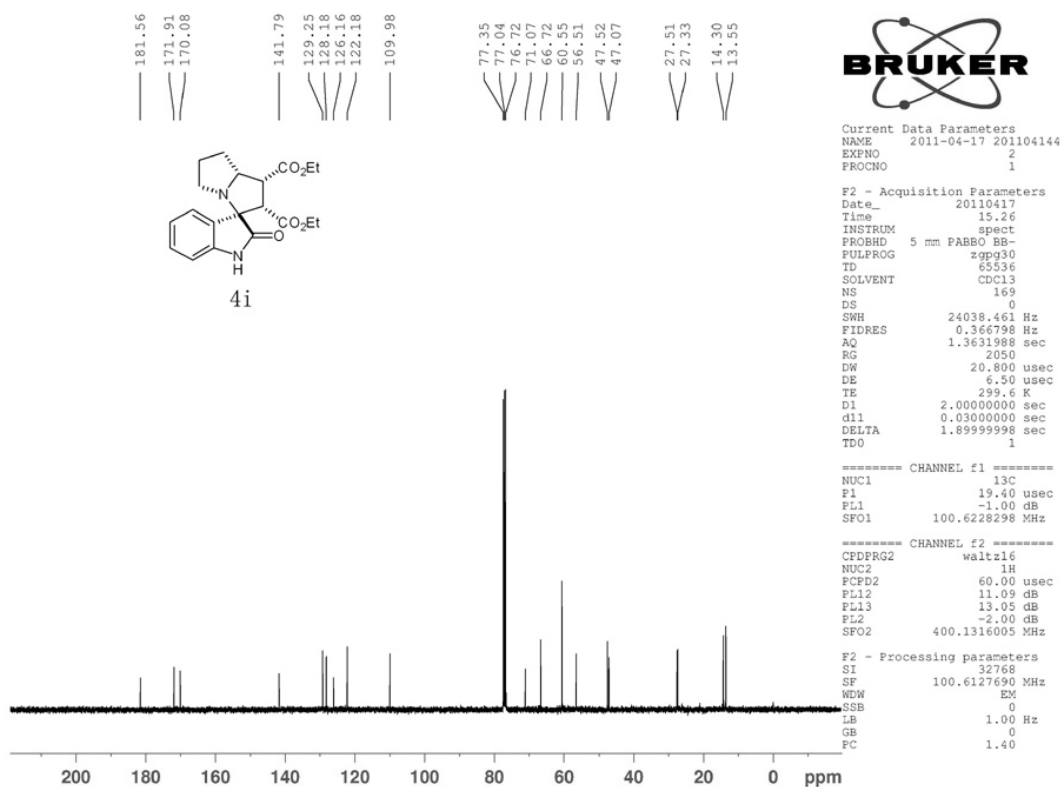
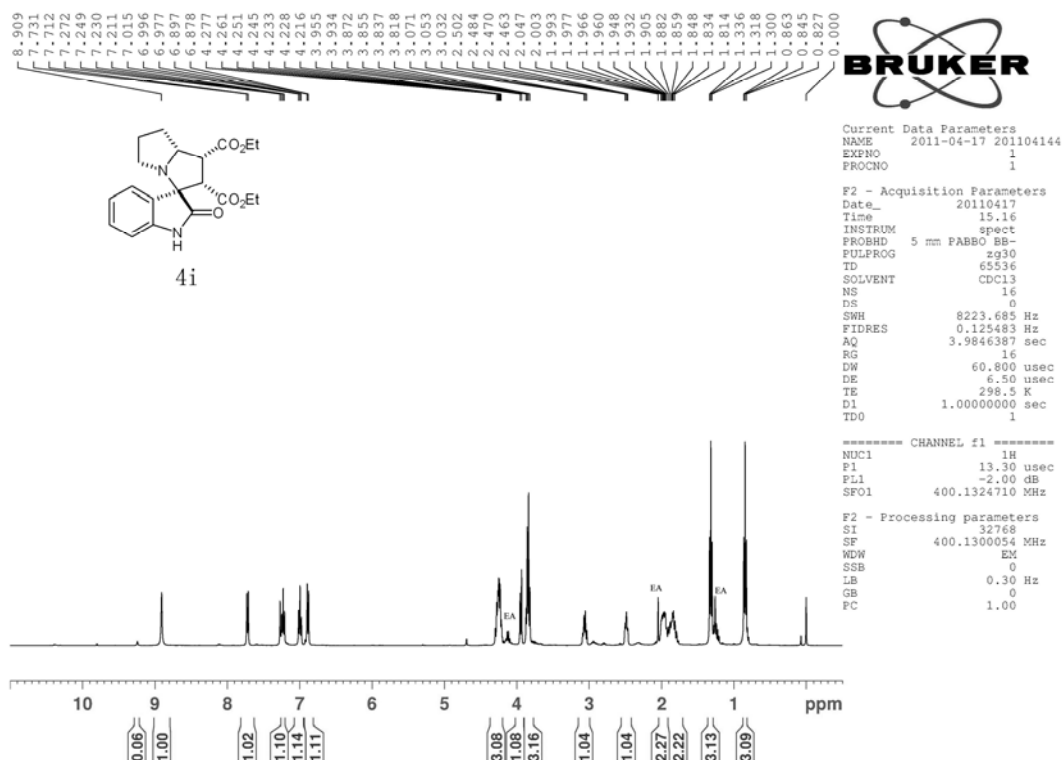
13:44:07

110915_4H 3 (0.051) AM (CenA, 80.00, Ar, 10000 0.000, 0.70), Sm (SG, 2x3.00), Cm (1.38)



15-Sep-2011

TQF MS ES+
 1.81e4

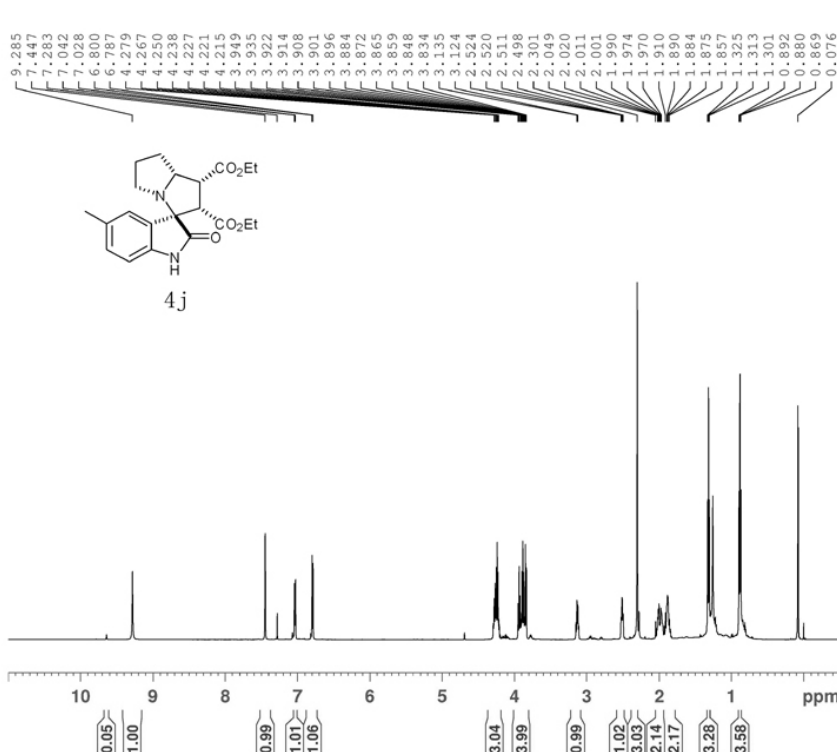
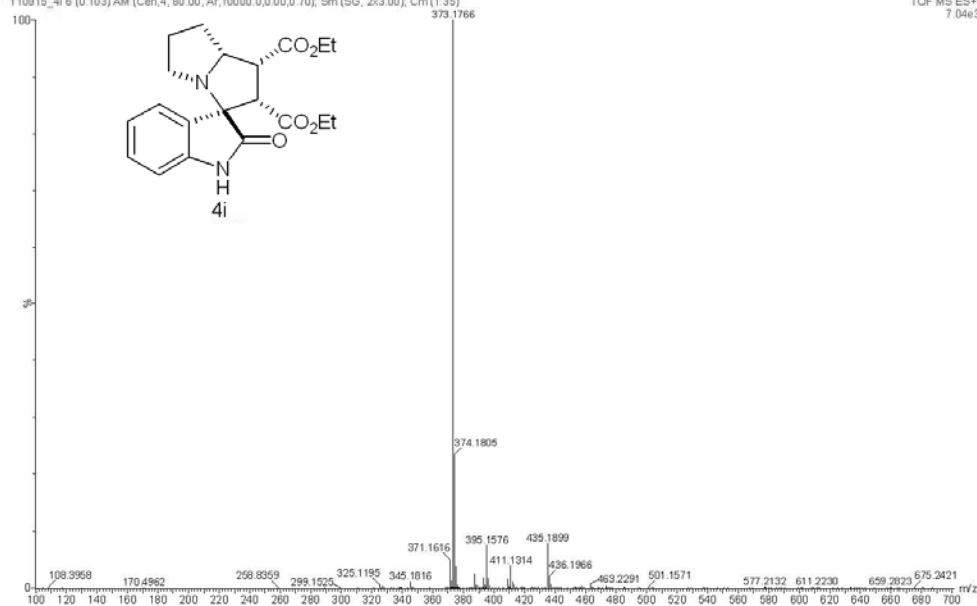


13:55:44

110915_4i 6 (0.103) AM (Cen, 4, 80.00, Ar, 10000.0, 0.00, 0.70); Sm (SG, 2x3.00); Cm (1:35)

15-Sep-2011

TOF MS ES+
7.04e3

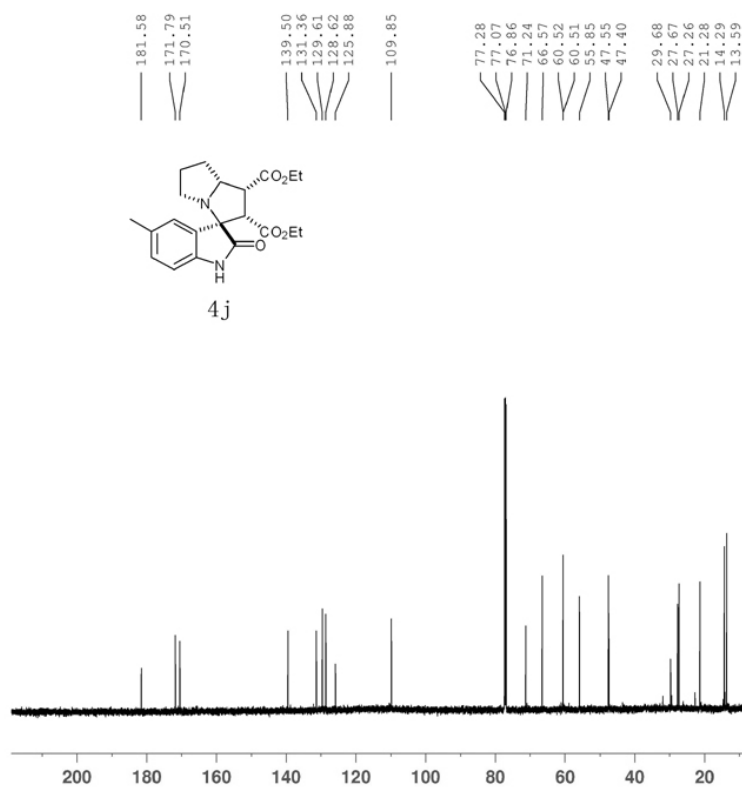


Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110424
Time 17.58
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 4
DW 40.533 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 7.60 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1299958 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00



Current Data Parameters
 NAME 2011-04-24 201104212
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110424
 Time 18.05
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 136
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9088159 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

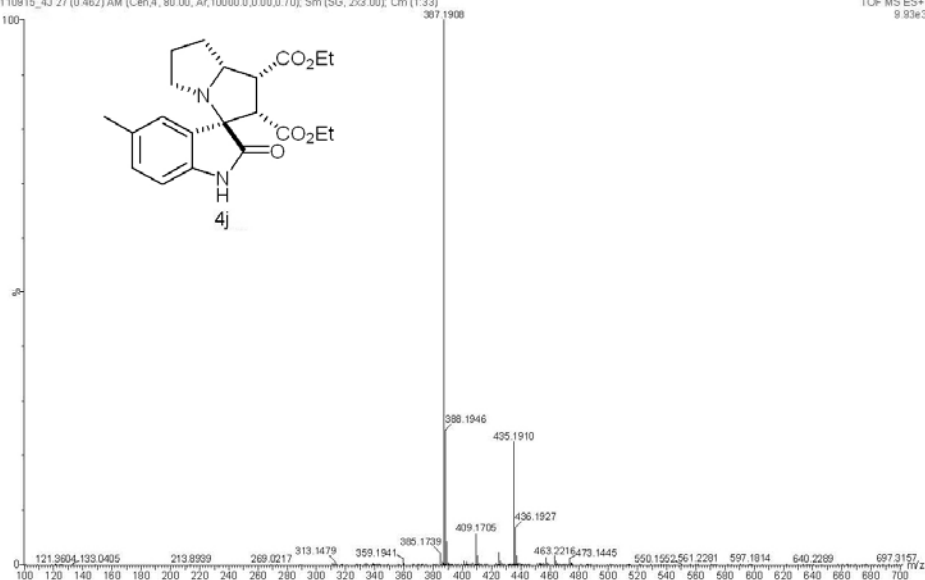
===== CHANNEL f1 =====
 NUC1 13C
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 150.9178988 MHz

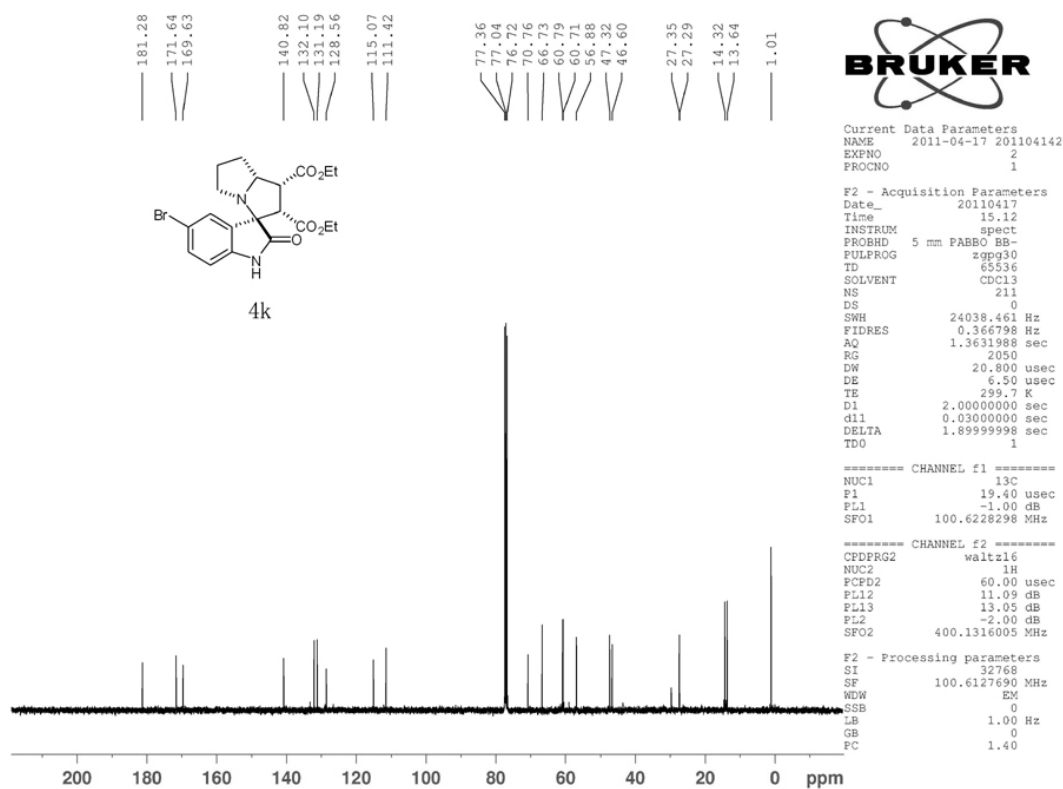
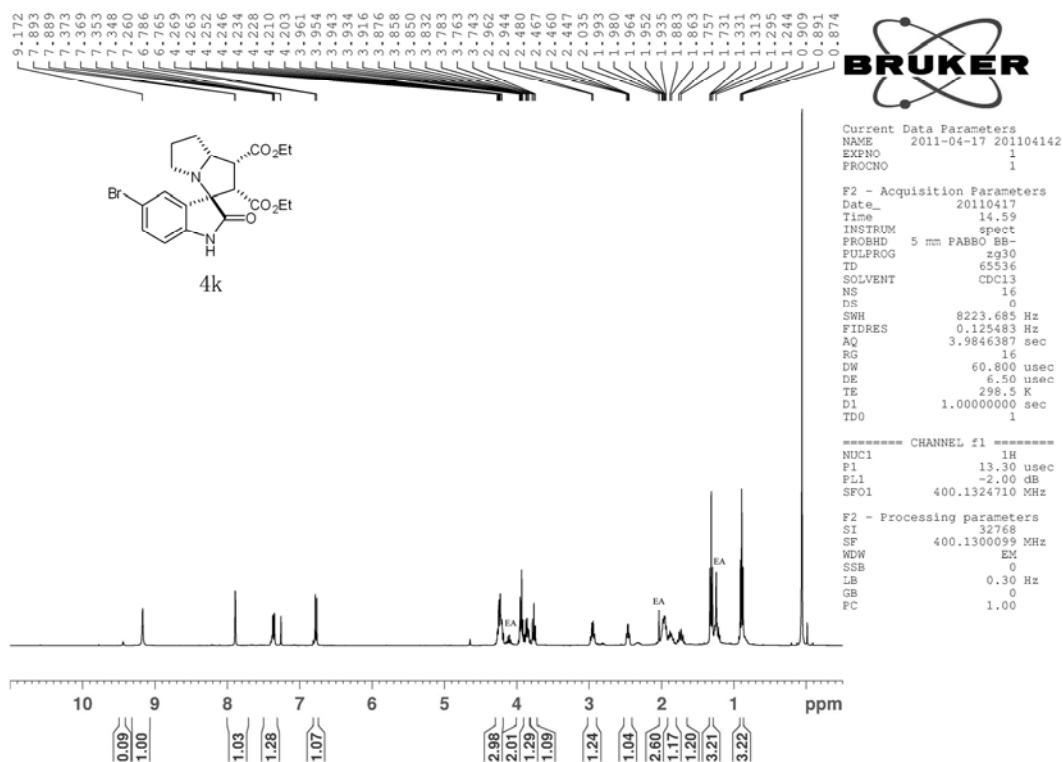
===== CHANNEL f2 =====
 CDPFRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.45 dB
 PL13 19.50 dB
 PL2 -1.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

13:52:48
 110915_4J 27 (0.462) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG; 2x3.00); Cm (1:33)

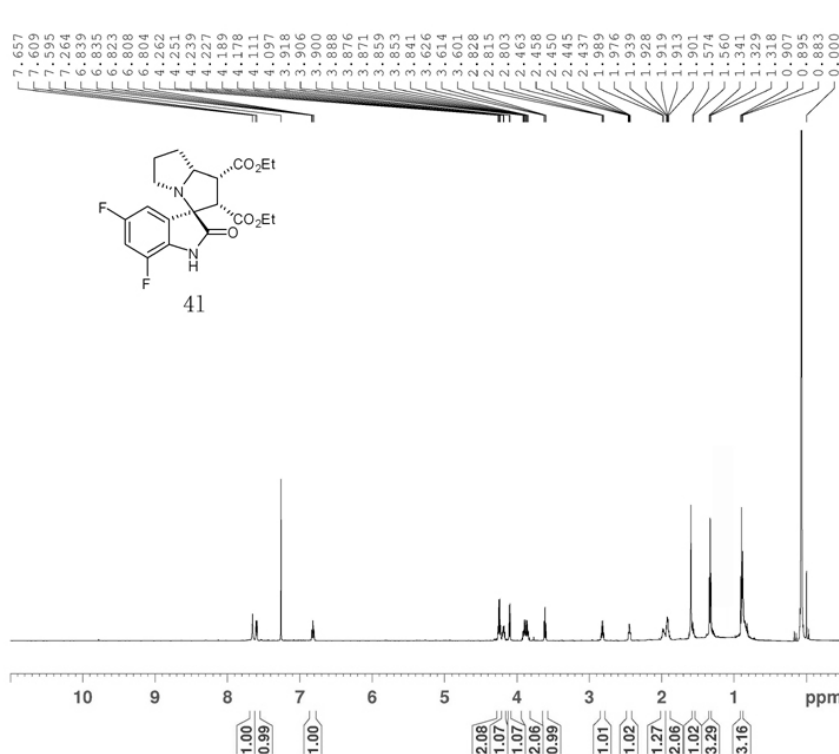
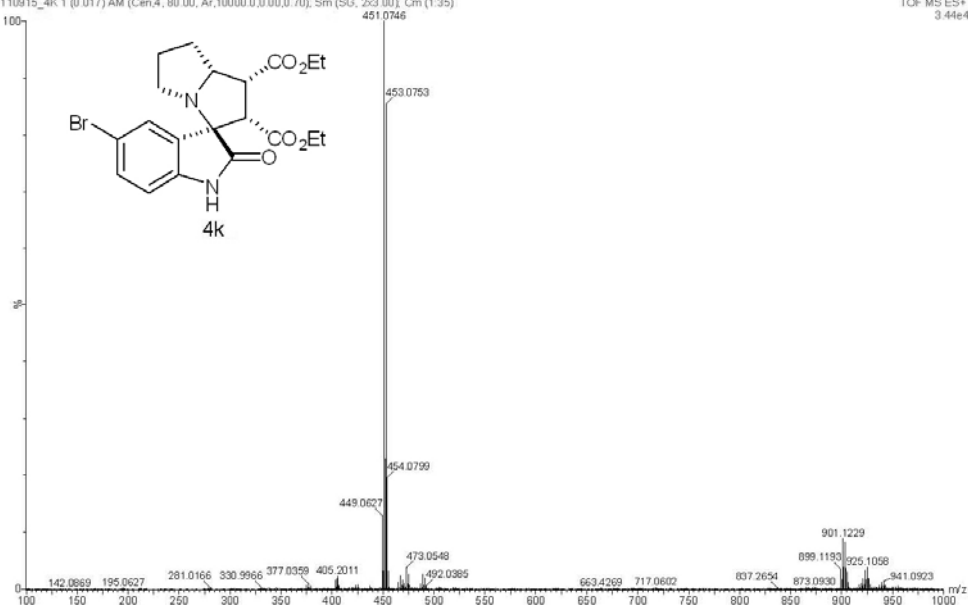
15-Sep-2011
 TOF MS ES+
 8.93e3





12:51:36
110915_4K 1 (0.017) AM (Cen.4, 80.00, Ar, 10000.0, 0.00, 0.70), Sm (50, 2x3.00), Cm (1.35)

15-Sep-2011
TOF MS ES+
3.44e4

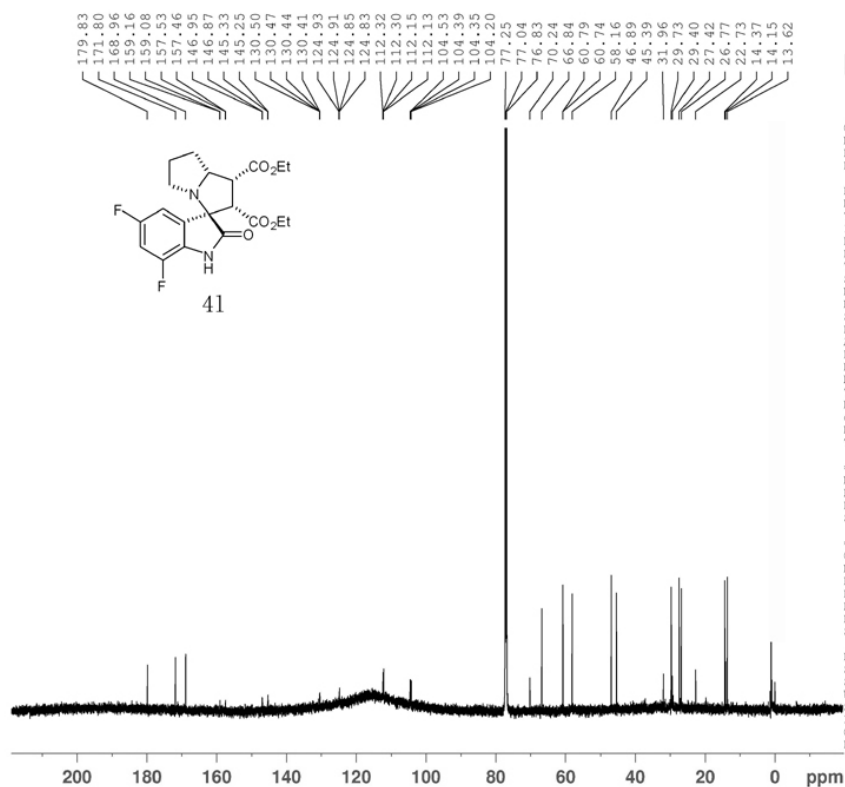


Current Data Parameters
NAME 2011-04-24 201104192
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110424
Time 19.12
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 4
DW 40.533 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.60 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1300076 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00



Current Data Parameters
 NAME 2011-04-24 201104192
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110425
 Time 7.17
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8000
 DS 0
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9088159 sec
 RG 2050
 DW 13.867 usec
 DE 6.50 usec
 TE 295.3 K
 D1 4.00000000 sec
 d11 0.03000000 sec
 DELTA 3.90000010 sec
 TD0 1

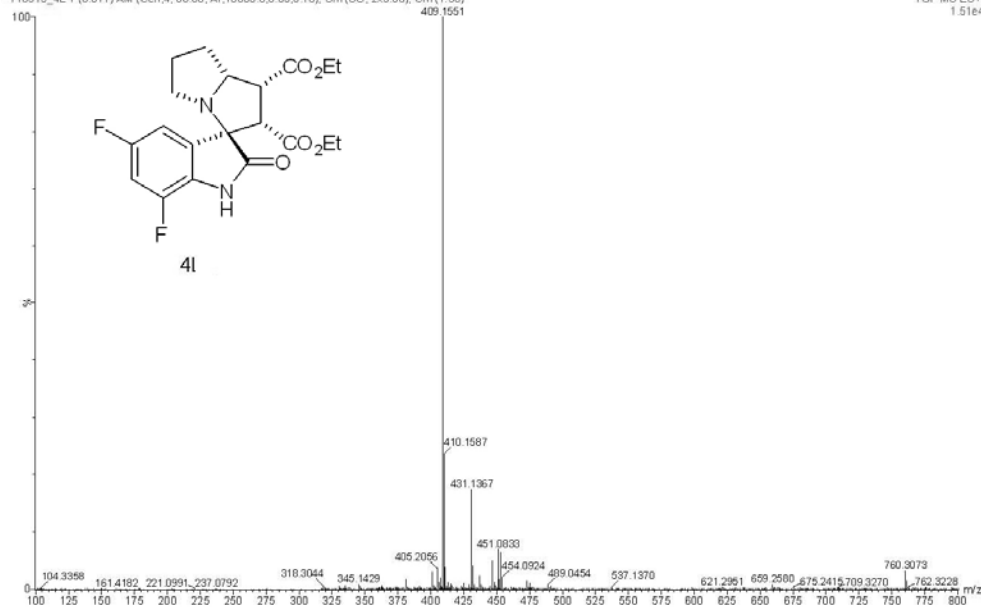
===== CHANNEL f1 =====
 NUC1 13C
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 150.9178988 MHz

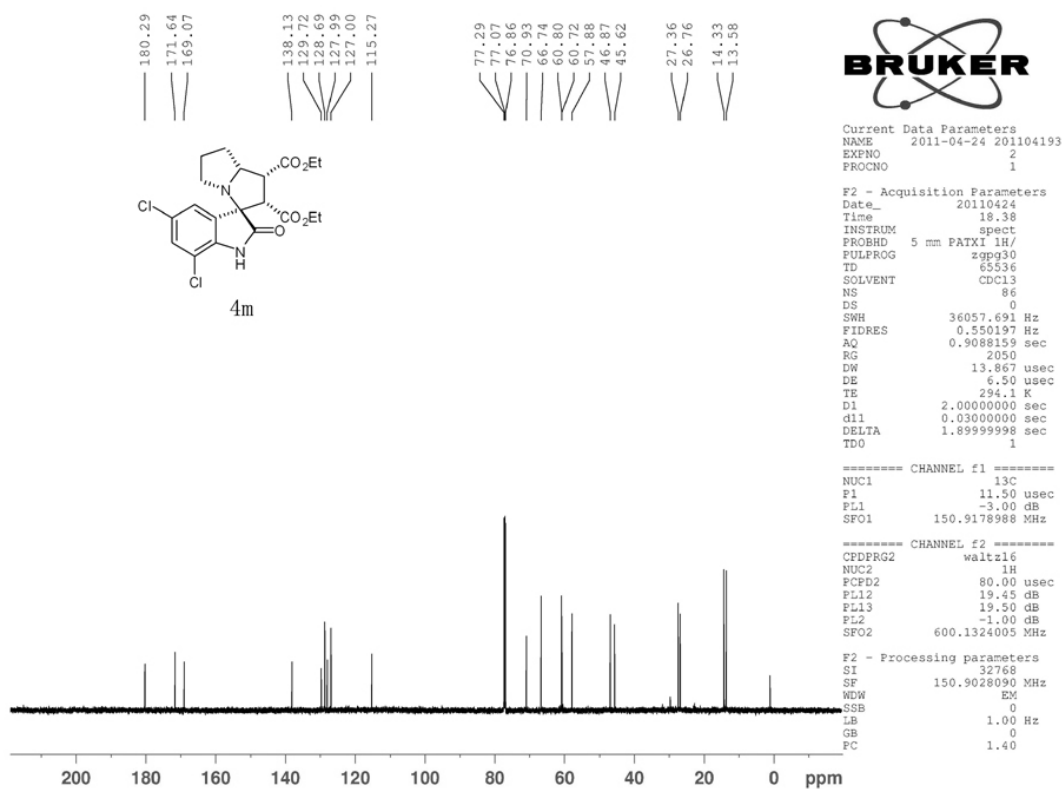
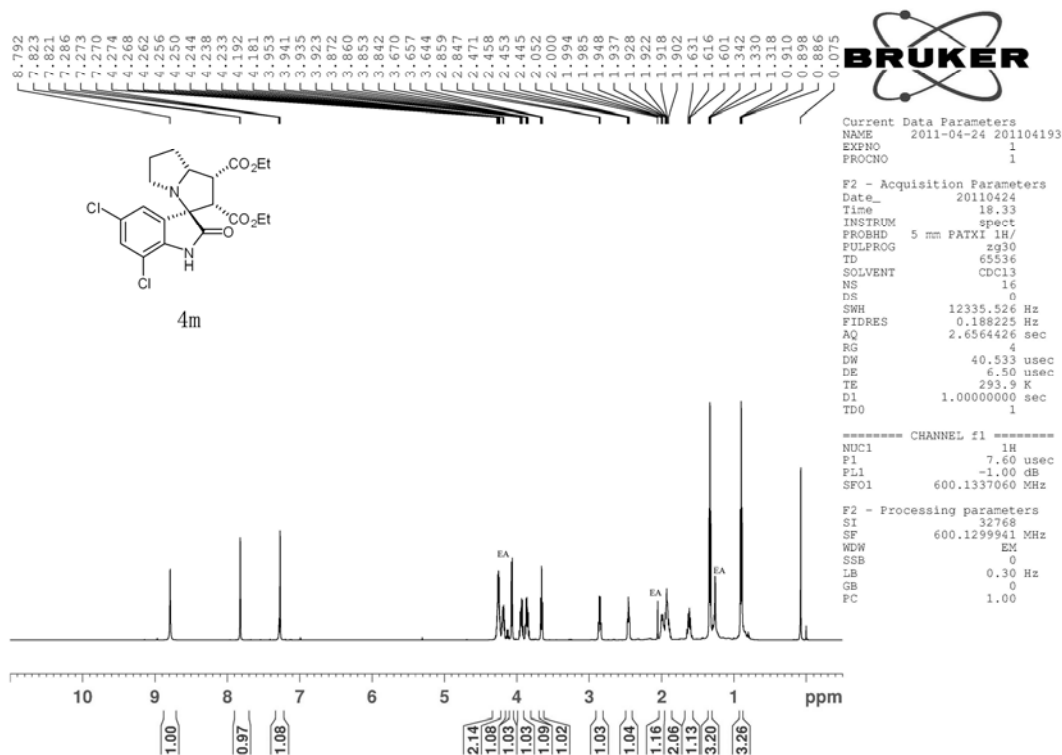
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 19.45 dB
 PL13 19.50 dB
 PL2 -1.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 32768
 SF 150.9028021 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

12:56:02
 110815_4L 1 (0.017) AM (Cen.4, 80.00, Ar, 10000.0, 0.00, 0.70); Sm (SG, 2x3.00); Cm (1:36)

15-Sep-2011
 TOF MS ES+
 1.51e4

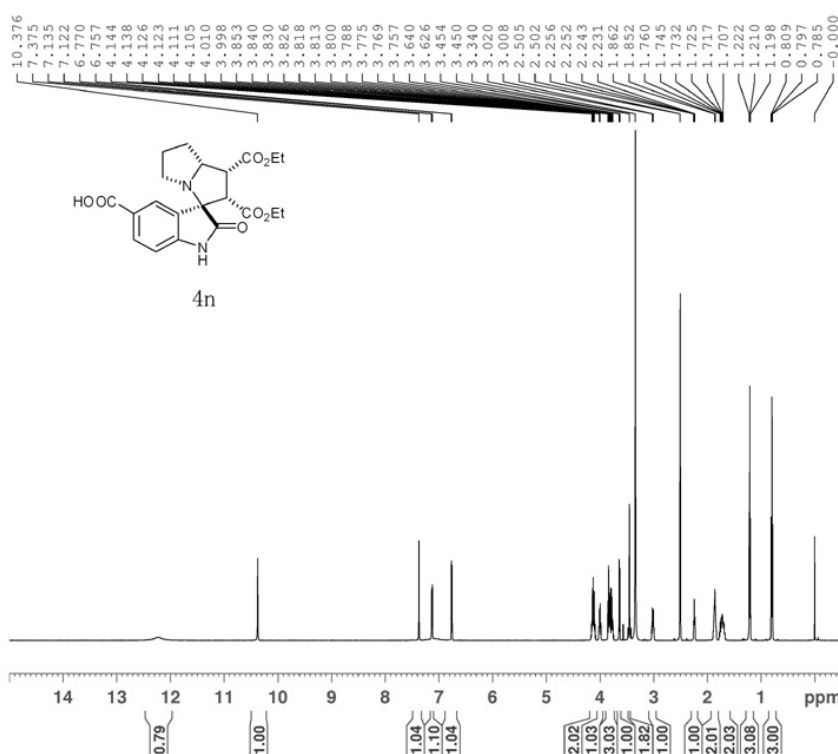
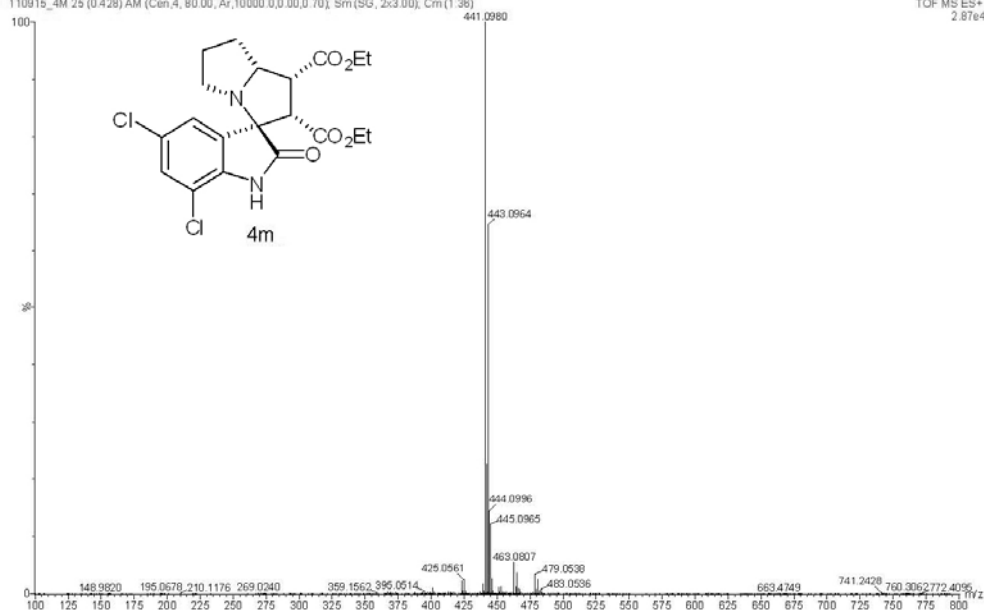




13:11:23

110915_4M 25 (0.428) AM (Cen,4, 80.00, Ar,10000,0.0,0.0,0.70); Sm(SG, 2x3.00); Crn(1:36)

15-Sep-2011

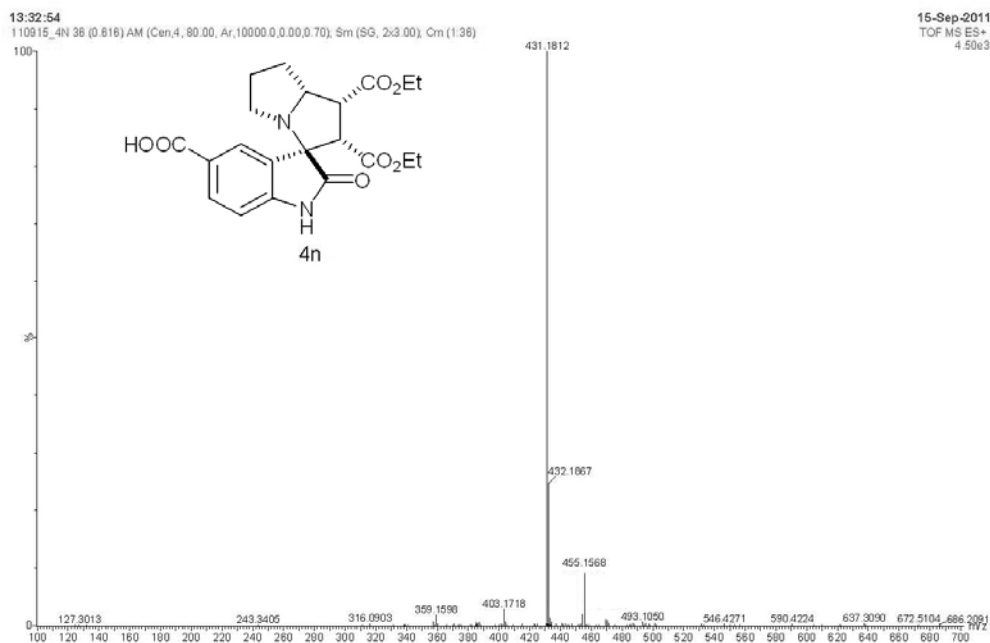
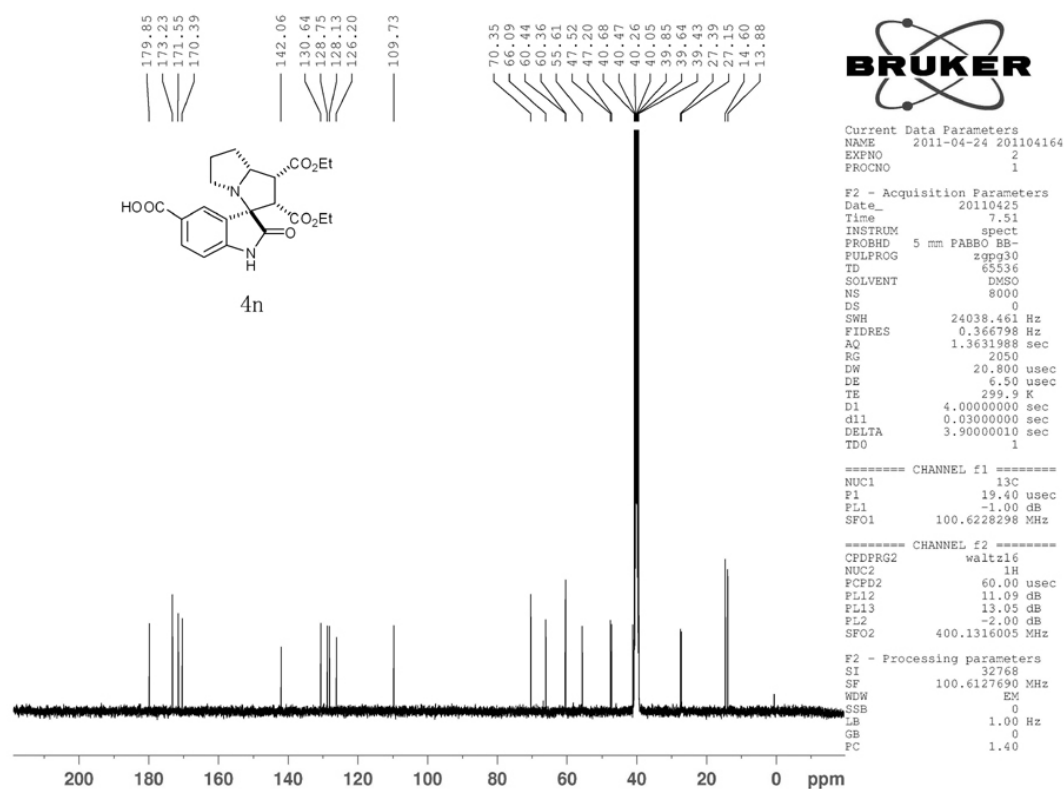
TOF MS ES+
2.87e4

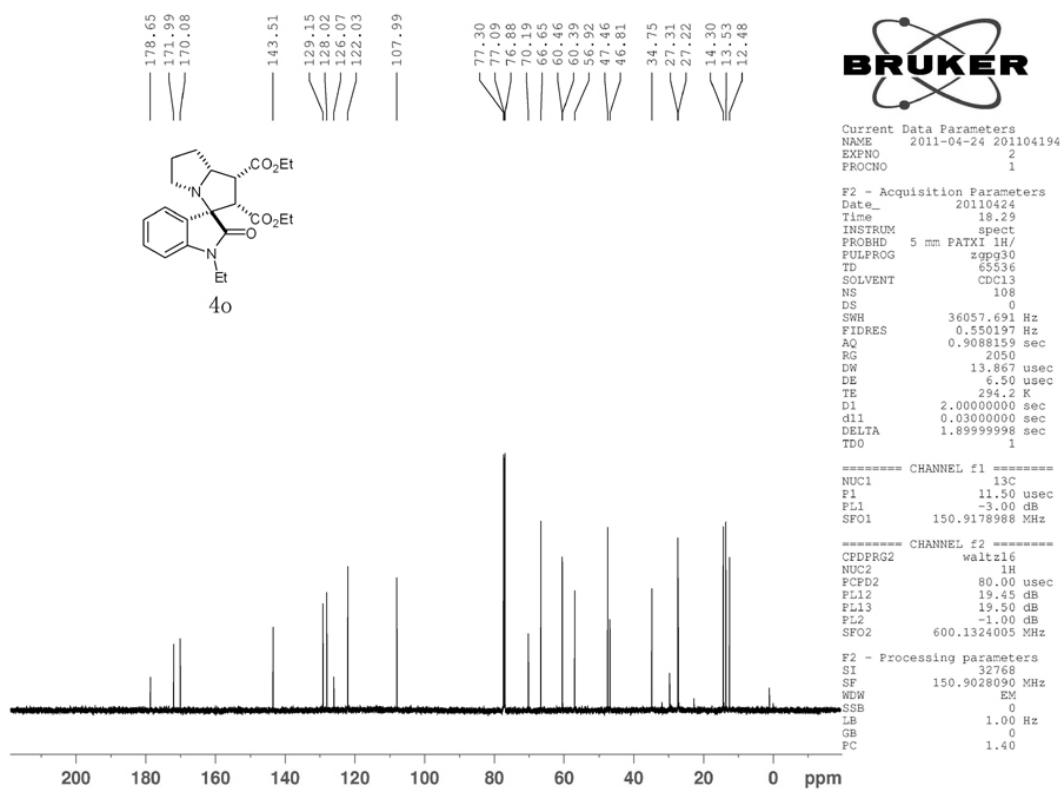
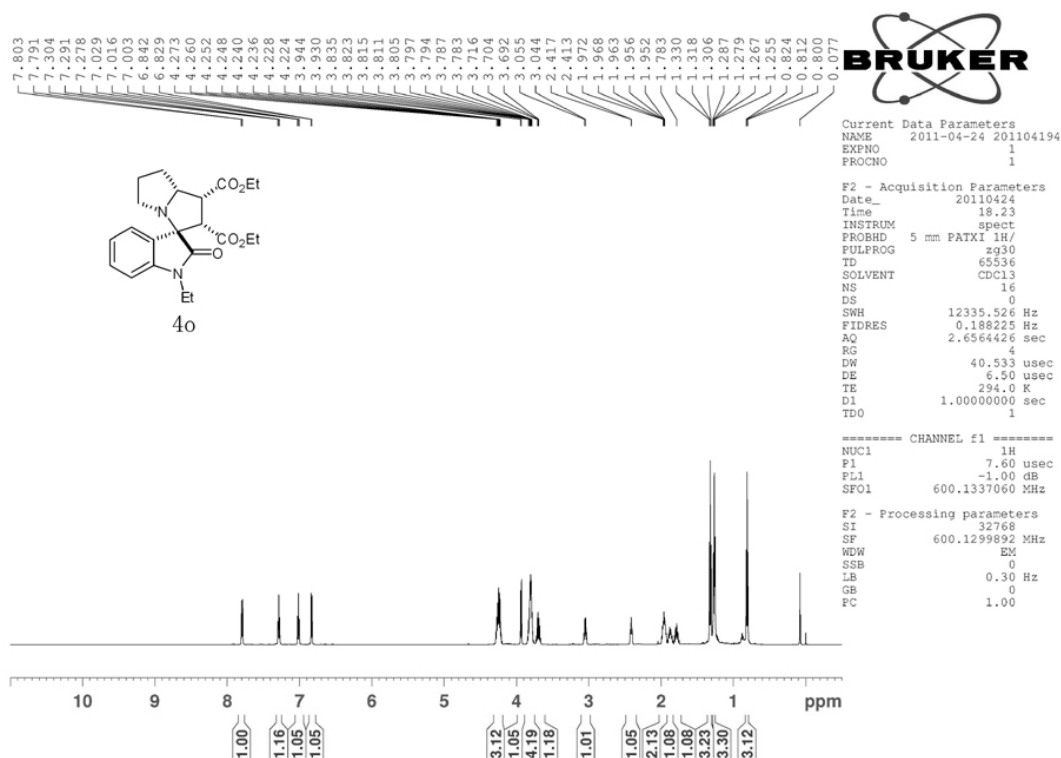
Current Data Parameters
NAME 2011-04-24 201104164
EXPNO 1
PROCNO 1

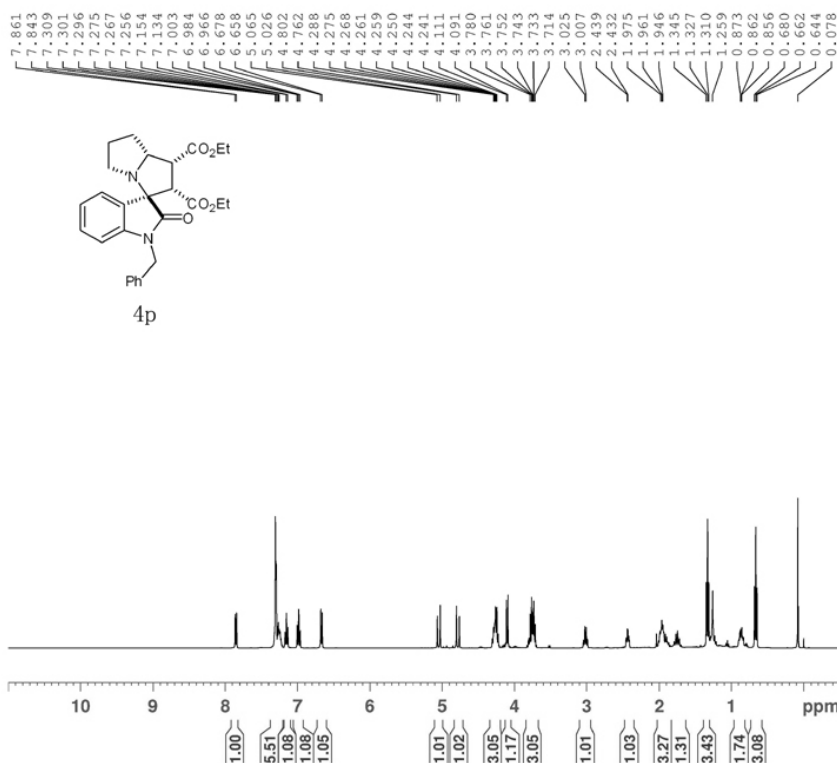
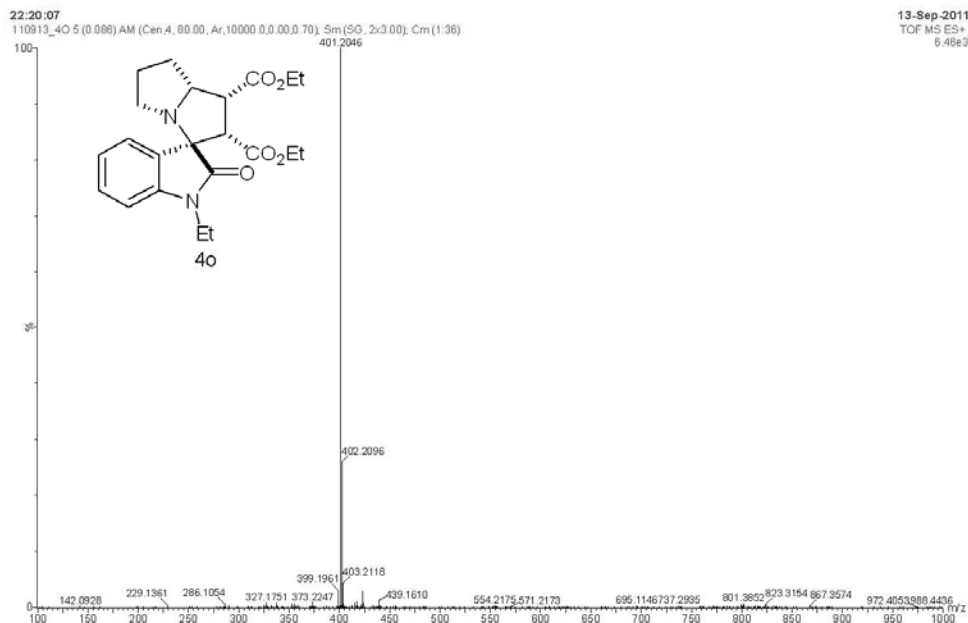
F2 - Acquisition Parameters
Date_ 20110424
Time 18.43
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 4
DW 40.533 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.60 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1299983 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00





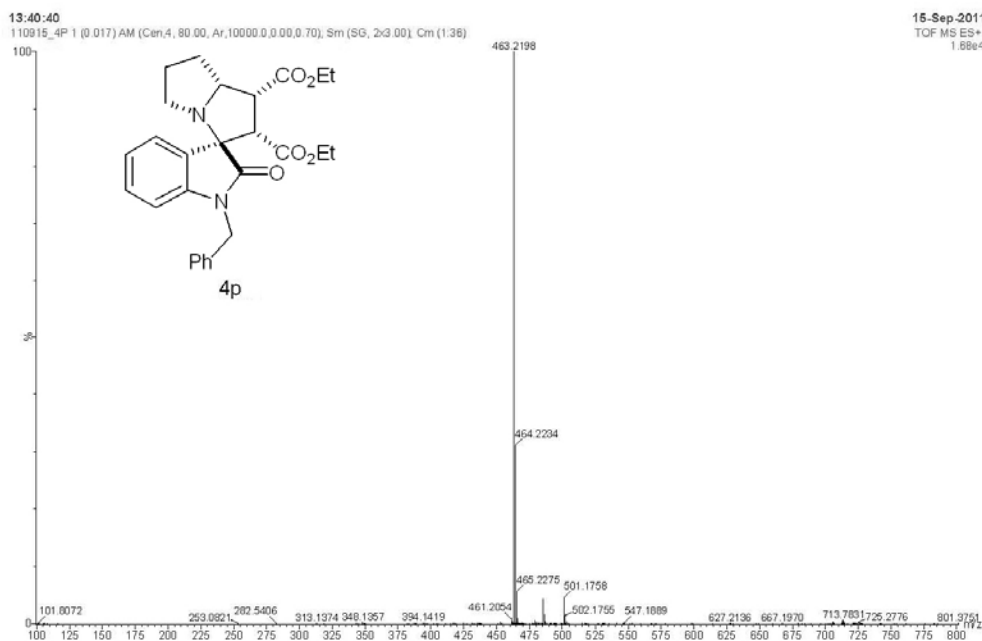
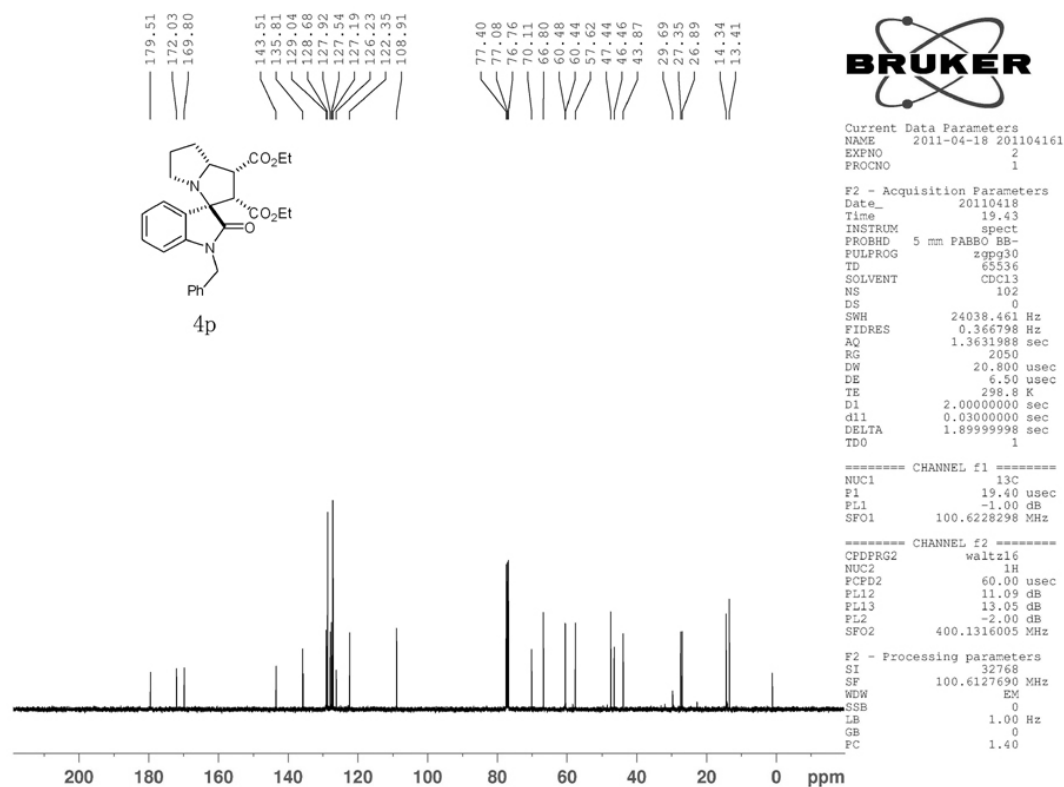


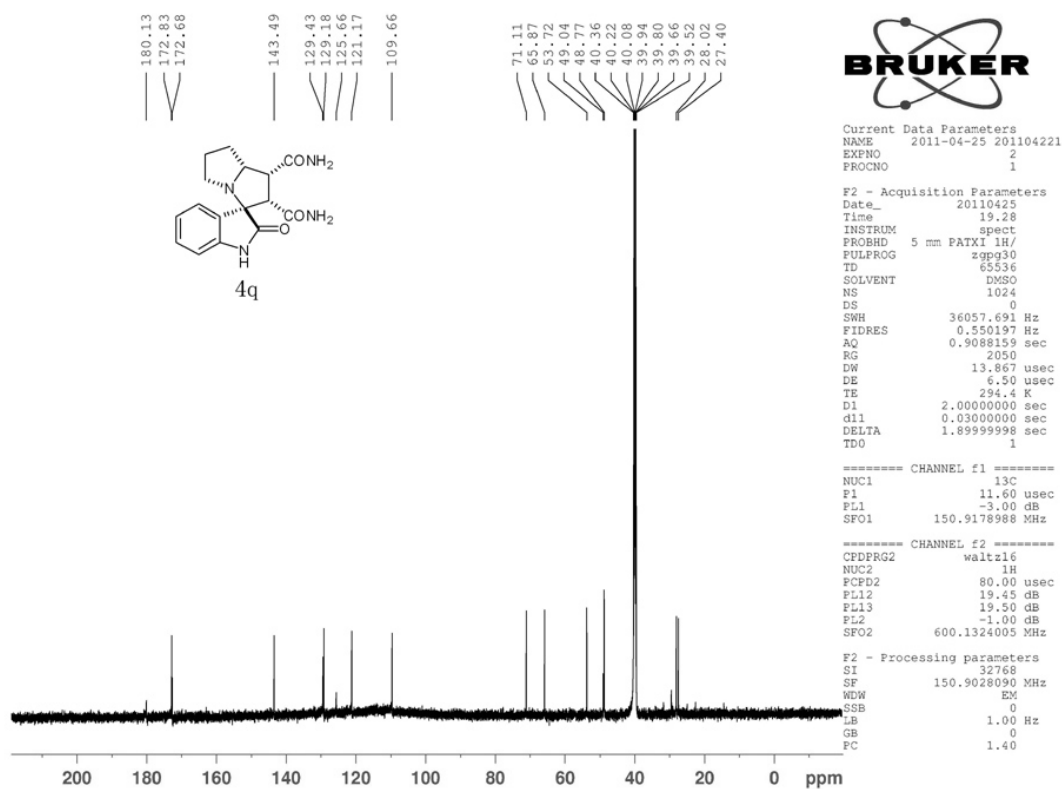
Current Data Parameters
NAME 2011-04-18 201104161
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110418
Time 19.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

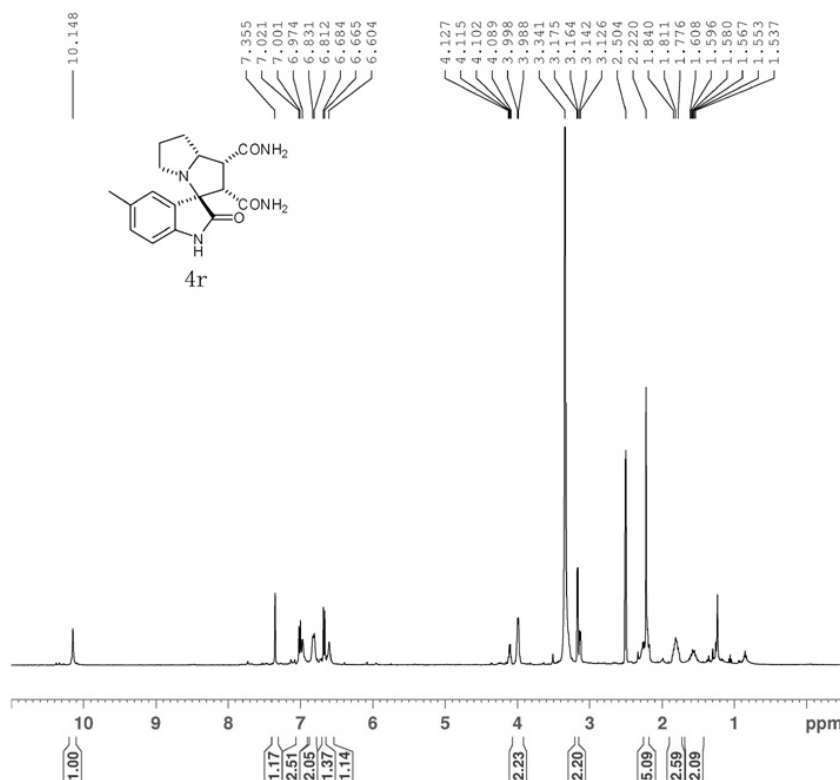
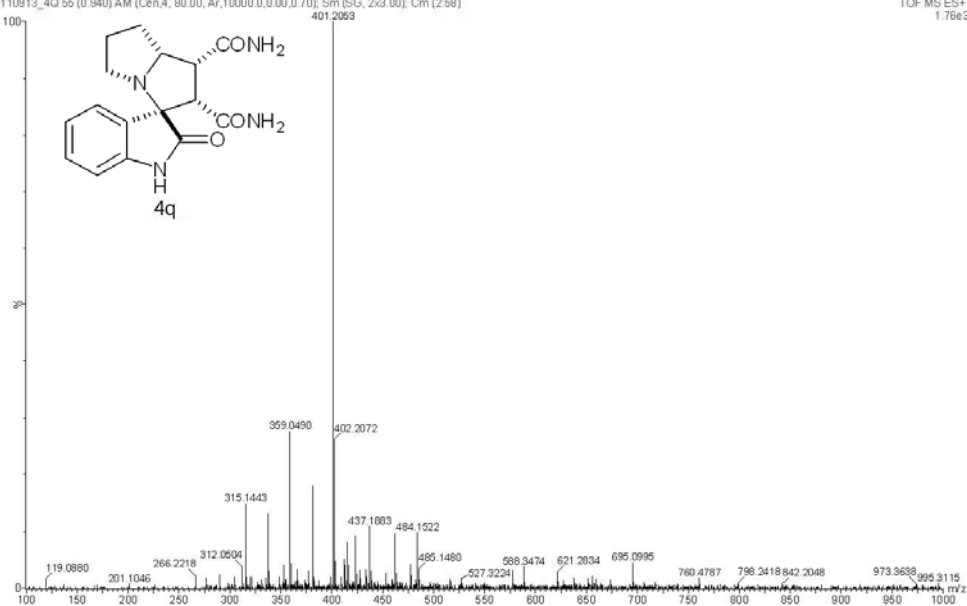
F2 - Processing parameters
SI 32768
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00





22:26:32
110913_4Q 55 (0.940) AM (Cen, 4, 80.00, Ar, 10000.0, 0.00, 0.70); Sm (SG, 2x3.00); Cm (2.58)

13-Sep-2011
TOF MS ES+
1.76e3



Current Data Parameters
NAME 2011-04-29 201104231
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110429
Time 16.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 16
DW 60.800 usec
DE 6.50 usec
TE 300.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300023 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
FC 1.00

