Highly Efficient Asymmetric Synthesis of 3-Indolyl-(hydroxy)acetates via Friedel-Crafts Alkylation of Indoles

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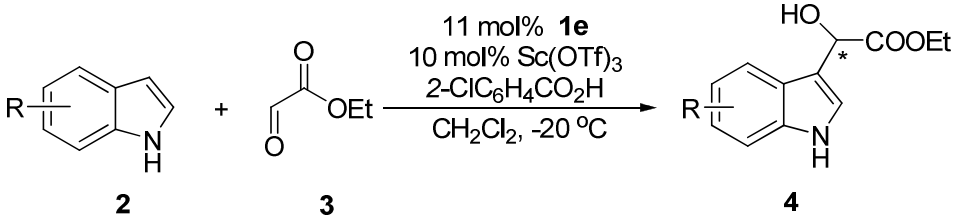
SUPPORTING INFORMATION

1. General

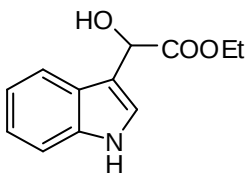
^{1}H NMR spectra were recorded on commerical instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$ and DMSO-d_6). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR spectra were collected on commerical instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$ and DMSO-d_6). The enantiomeric excesses were determined by HPLC analysis on chiral DAICEL CHIRALCEL AS-H and OD-H column. Optical rotations were measured on a commerical polarimeter and reported as follows: $[\alpha]_D^T$ (c = g/100 mL, solvent). Reagents obtained from commercial sources were used without further purification. Anhydrous CH_2Cl_2 were distilled over CaH respectively before use. Chiral *N*,*N'*-dioxide ligands **1a**-**f** were prepared according to the literature procedures.[1]

2. Typical procedure for the enantioselective Friedel-Crafts reaction.

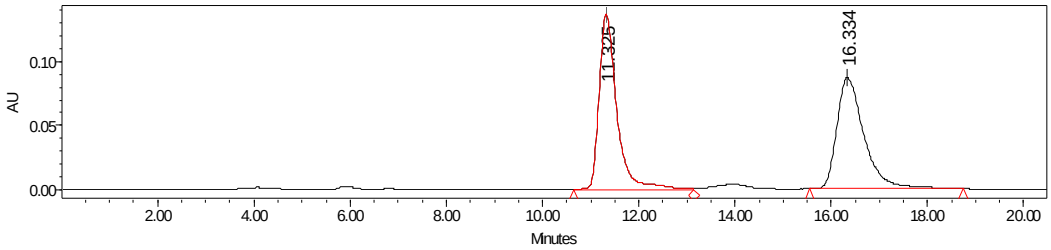


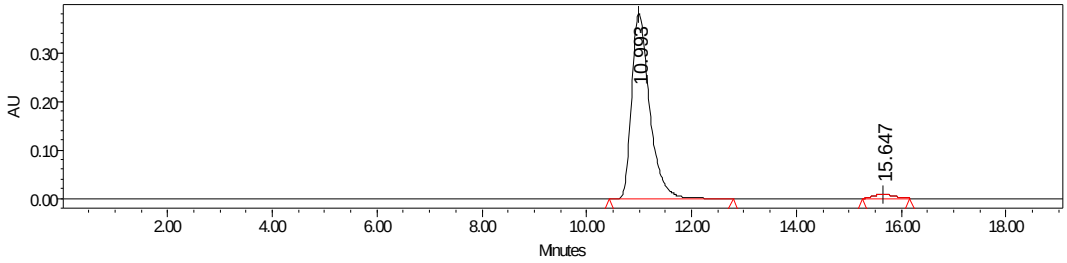
2 **3** **4**

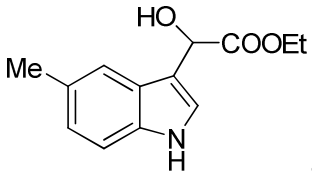
To a test tube, *N*,*N'*-dioxide **1e** (0.011 mmol), $Sc(OTf)_3$ (0.01 mmol), indole **2** (0.1 mmol), *o*-chlorobenzoic acid (0.01 mmol) was added under nitrogen. Then 0.5 mL CH_2Cl_2 was added and stirred at 30°C for 1h. Subsequently, ethyl glyoxylate **3** (0.15 mmol) was added under -20°C, and the reaction was stirred for further 12 - 148 h. The 3- indolyl(hydroxyl)acetates were directly purified by column chromatography on silica gel (ethyl acetate / petroleum ether 1:2 — 1:3)**.**



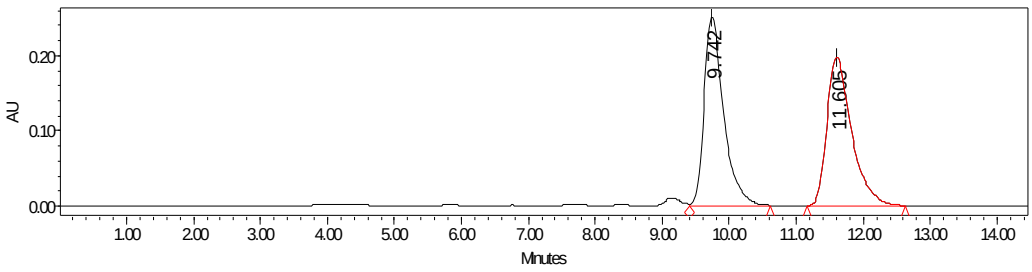
4**a**: colorless oil, The material was determined to be of 95% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min; t_r (major) = 11.33 min, t_r (minor) = 16.33 min]; $[\alpha]_D^{20}$ = +77.5° (c = 3.02 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29 (s, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.33 (t, *J* = 8.0 Hz, 1H), 7.26-7.12 (m, 3H), 5.47 (d, *J* = 6.0 Hz, 1H), 4.33-4.25 (m, 1H), 4.21-4.13 (m, 1H), 3.38 (s, 1H), 1.34-1.20 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 136.5, 123.2, 122.6, 120.2, 119.5, 114.0, 111.3, 67.3, 62.8, 14.1.

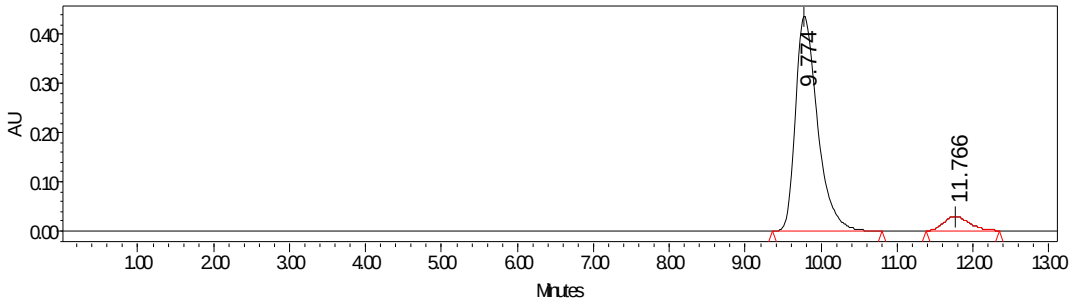


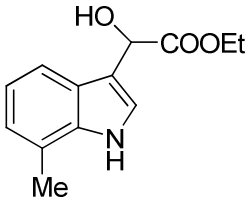




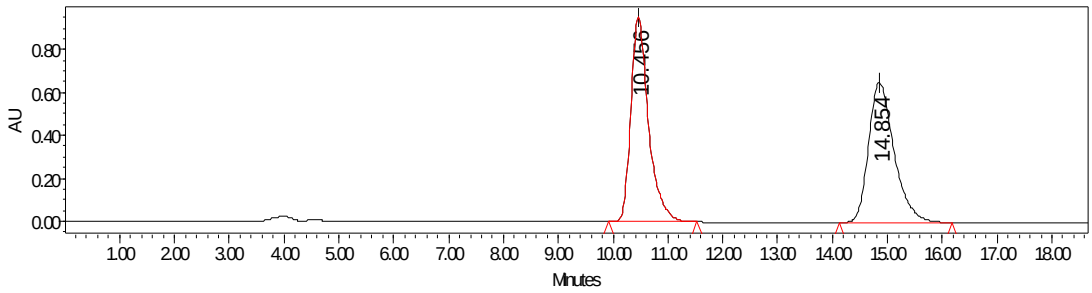
4**b**: colorless oil, The material was determined to be of 86% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min , t_r (major) = 9.74 min, t_r (minor) = 11.80 min]; $[\alpha]_D^{20}$ = +58.2^o (c = 3.68 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.27 (t, *J* = 8.0 Hz, 1H), 7.18(d, 1H), 7.12 (m, 1H), 5.46 (s, 1H), 4.35-4.29 (m, 1H), 4.24-4.18 (m, 1H), 2.45(s, 3H), 1.27-1.24 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 134.8, 129.5, 125.6, 124.2, 123.3, 119.1, 113.4, 111.0, 67.4, 62.0, 21.5, 14.1.

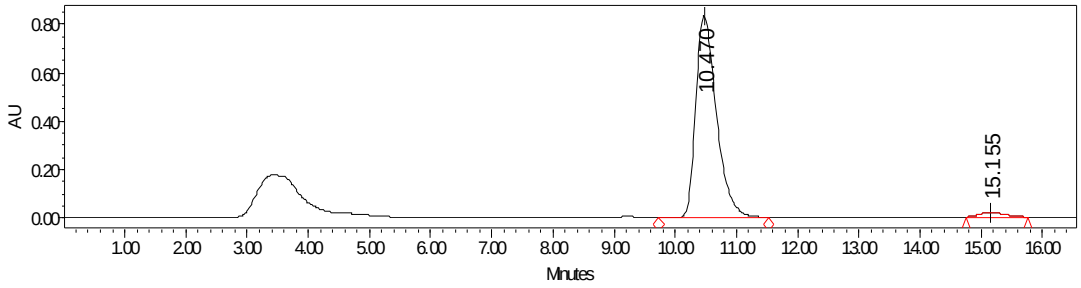


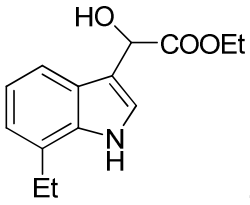




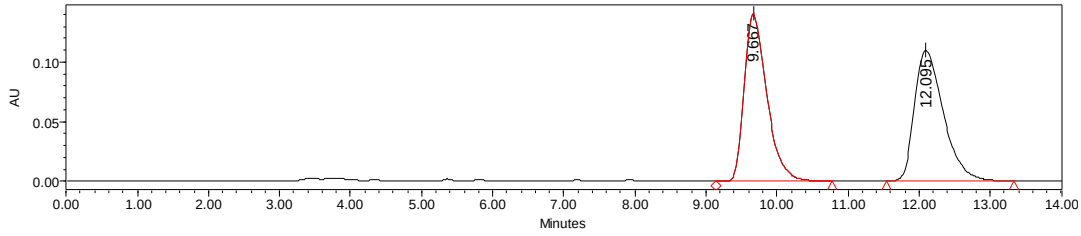
4c: colorless oil, The material was determined to be of 94% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min, t_r (major) = 10.46 min, t_r (minor) = 14.85 min]; $[\alpha]_D^{20}$ = +83.9° (c = 4.10 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.21 (s, 1H), 7.03-6.96 (m, 2H), 5.40 (d, *J* = 6.0 Hz, 1H), 4.27-4.24 (m, 1H), 4.22-4.04 (m, 1H), 3.25 (s, 1H), 2.42(s, 3H), 1.20-1.17 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 136.1, 124.9, 124.6, 123.0, 120.5, 117.9, 117.2, 114.5, 67.4, 62.6, 16.6, 14.1; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 232.0974, Found: 232.1098.

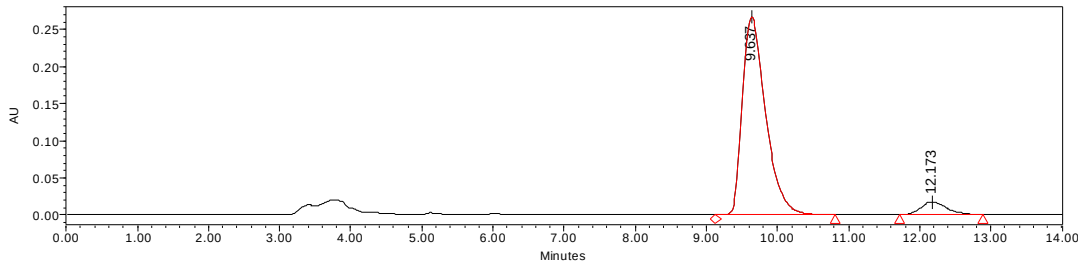


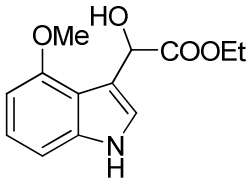




4d. white solid. The material was determined to be of 92% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min, t_r (major) = 9.65 min, t_r (minor) = 12.10 min]; $[\alpha]_D^{20}$ = +71.7° (c = 4.00 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.27 (t, 1H), 7.15-7.07 (m, 2H), 5.48 (d, *J* = 6.0 Hz, 1H), 4.35-4.29 (m, 1H), 4.23-4.16 (m, 1H), 3.30 (s, 1H), 2.90-2.84 (m, 2H), 1.39-1.34 (t, *J* = 4.0 Hz, 3H), 1.28-1.22 (t, *J* = 5.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 135.4, 126.7, 125.1, 122.8, 121.1, 120.5, 117.2, 114.5, 67.4, 62.1, 24.0, 14.1, 13.8; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 246.1130, Found: 246.1260.

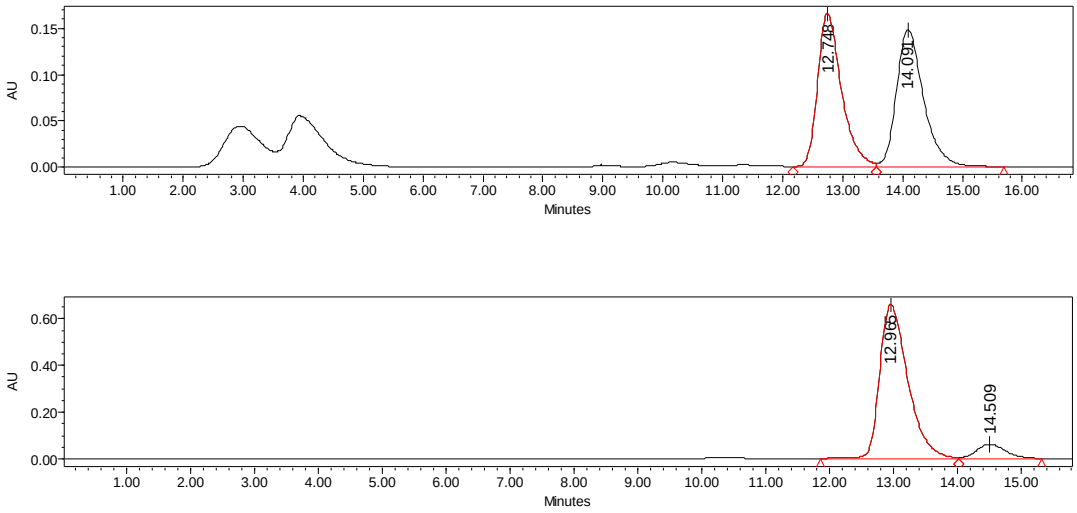


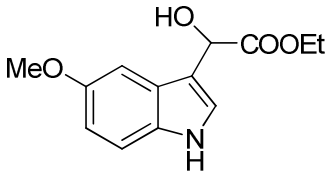




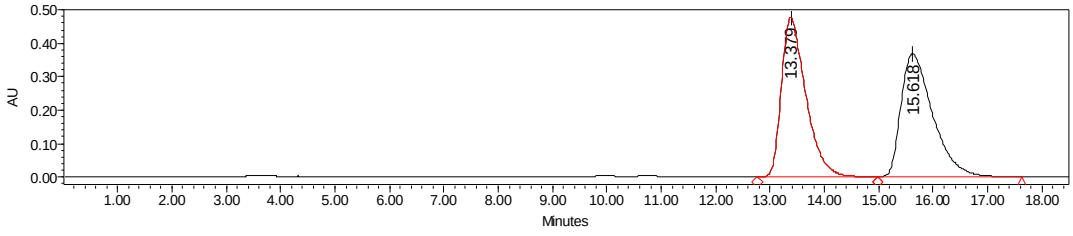
4e: colorless oil. The material was determined to be of 83% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min t_r (major) = 12.75 min, t_r (minor) = 14.10 min]; $[\alpha]_D^{20}$ = +52.7° (c = 3.98 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (s, 1H), 7.28 (s, 1H), 7.16-6.98 (m, 2H), 6.57 (d, *J* = 6.0 Hz,1H), 5.47 (d, *J* = 5.0 Hz, 1H), 4.29-4.23 (m, 1H), 4.21-4.14 (m, 1H), 3.97 (s, 3H), 1.25-1.21 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 152.7, 138.2,

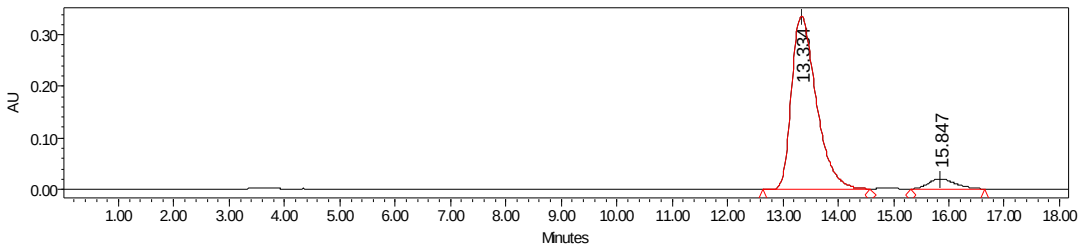
123.4, 122.7, 115.6, 114.1, 105.2, 100.2, 68.0, 61.4, 55.3, 14.2; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 248.0923, Found: 248.1043.

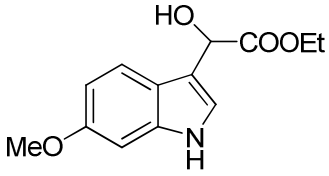




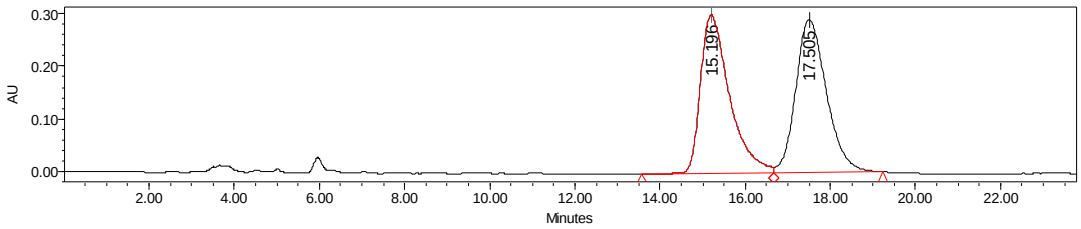
4f: colorless oil**.** The material was determined to be of 88% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min t_r (major) = 13.38 min, t_r (minor) = 15.62 min]; $[\alpha]_D^{20}$ = +57.0° (c = 2.76 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.28-7.17 (m, 2H), 6.91-6.88 (m, 1H), 5.46 (d, *J* = 4.0 Hz, 1H), 4.36-4.28 (m, 1H), 4.25-4.17 (m, 1H), 3.87(s, 3H), 3.29 (d, *J* = 5.0 Hz, 1H), 1.64 (s, 1H), 1.28-1.24(t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 154.4, 131.5, 125.9, 123.8, 113.7, 113.1, 112.1, 101.0, 67.3, 62.1, 55.8, 14.2; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 248.0923, Found: 248.1041.

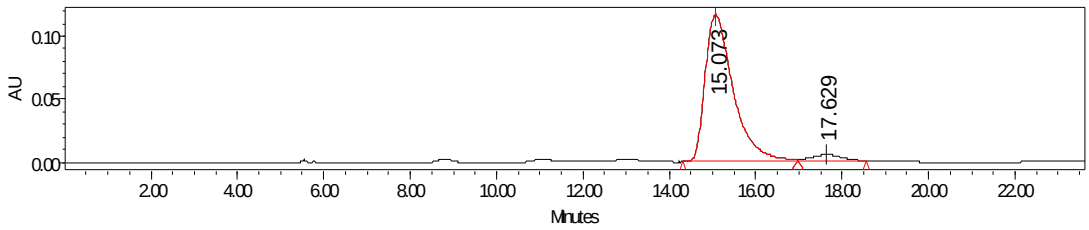


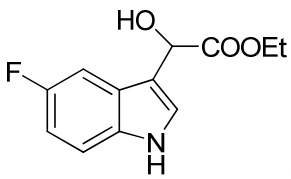




4g: colorless oil. The material was determined to be of 91% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*- hexane /*i*PrOH, 1.0 mL/min, t_r (major) = 15.19 min, t_r (minor) = 17.51 min]; $[\alpha]_D^{20}$ = +43.1^o (c = 2.74 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (s, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.07 (t, 1H), 6.78-6.74 (m, 2H), 5.36 (d, *J* = 6.0 Hz, 1H), 4.26-4.20 (m, 1H), 4.16-4.04 (m, 1H), 3.77(s, 3H), 3.23 (d, *J* = 6.0 Hz, 1H), 1.20-1.17 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 136.5, 123.2, 122.6, 120.2, 119.5, 114.0, 111.3, 67.3, 62.8, 14.1.

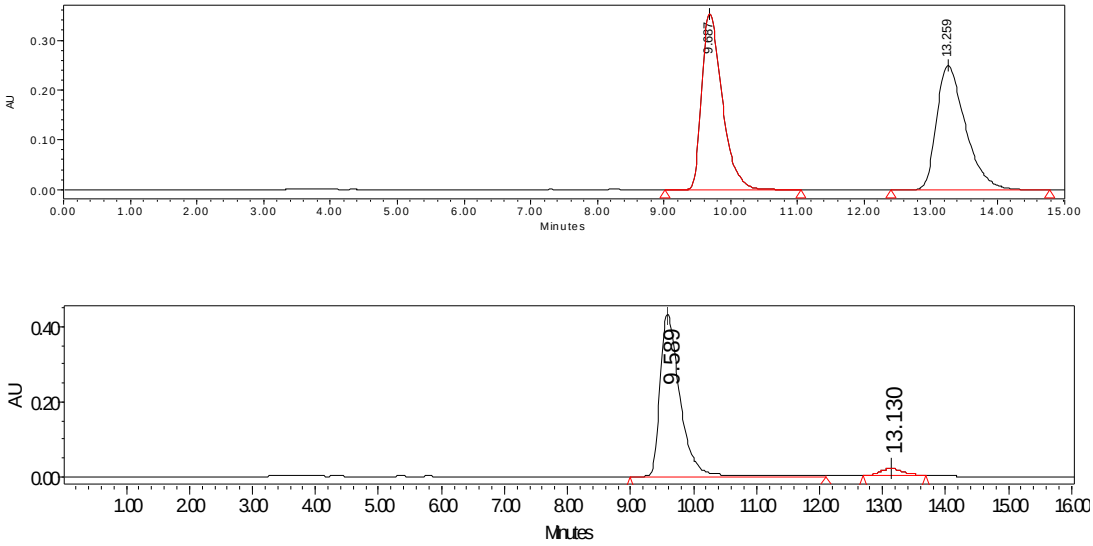


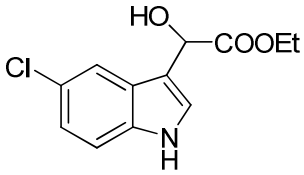




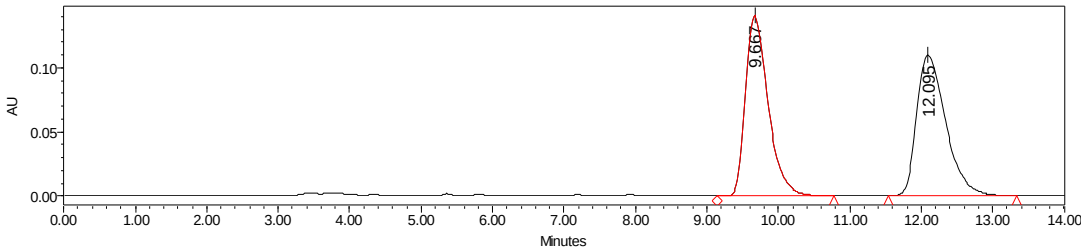
4h: colorless oil. The material was determined to be of 90% ee by chiral HPLC analysis [Chiralpak AS-H,$_{S7}$80:20 *n*-hexane/*i*PrOH, 1.0 mL/min, t_r

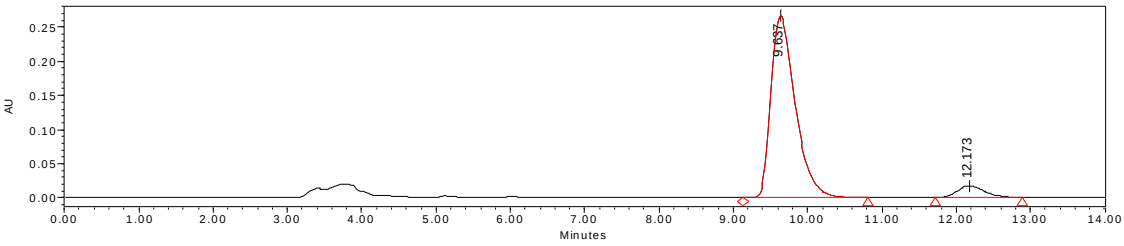
(major) = 9.59 min, t_r (minor) = 13.13 min]; $[\alpha]_D^{20}$ = +57.8^o (c = 3.60 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (s, 1H), 7.40-7.28 (m, 3H), 7.00-6.95 (m, 1H), 5.43 (d, *J* = 5.0 Hz, 1H), 4.34-4.28 (m, 1H), 4.25-4.17 (m, 1H), 3.37 (d, *J* = 6.0 Hz, 1H), 1.27-1.23 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 156.8, 133.0, 125.7, 124.8, 114.1, 112.1, 111.1, 104.5, 67.2, 62.2, 14.1; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 236.0723, Found: 236.0856.

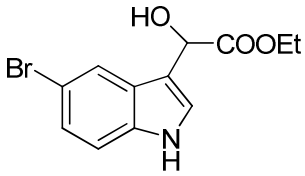




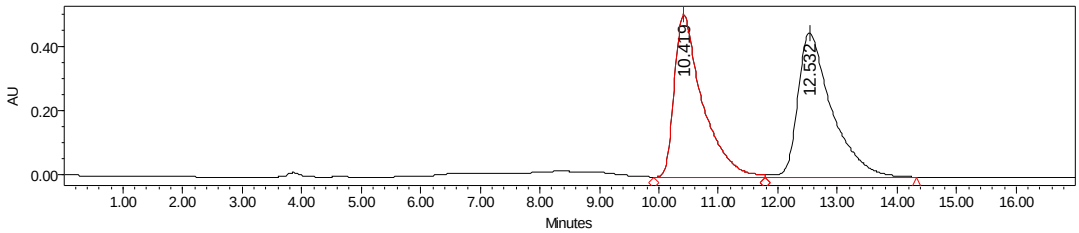
4i: colorless oil**.** The material was determined to be of 87% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min, t_r (major) = 9.61 min, t_r (minor) = 12.10 min]; $[\alpha]_D^{20}$ = +48.3^o (c = 3.80 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31 (s, 1H), 7.74 (s, 1H), 7.30-7.28 (m, 2H), 7.20-7.17 (m, 1H), 5.44 (d, *J* = 5.0 Hz, 1H), 4.36-4.28 (m, 1H), 4.26-4.18 (m, 1H), 3.37 (d, *J* = 6.0 Hz, 1H), 1.28-1.24 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 134.8, 126.4, 125.9, 124.4, 123.0, 119.2, 113.8, 112.4, 67.1, 62.3, 14.1; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 252.0428, Found: 252.0553.

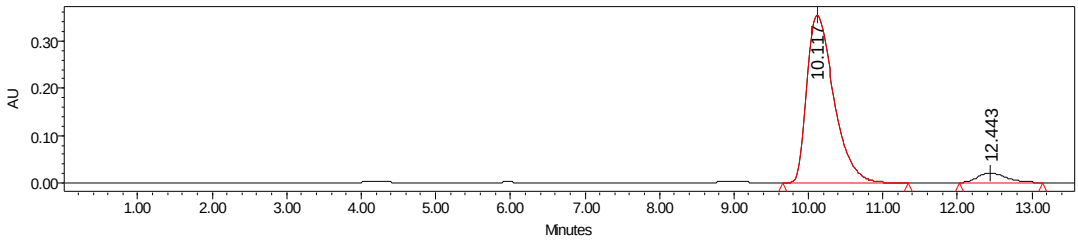


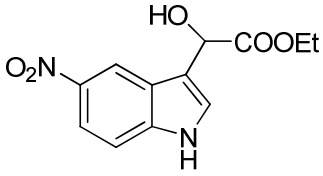




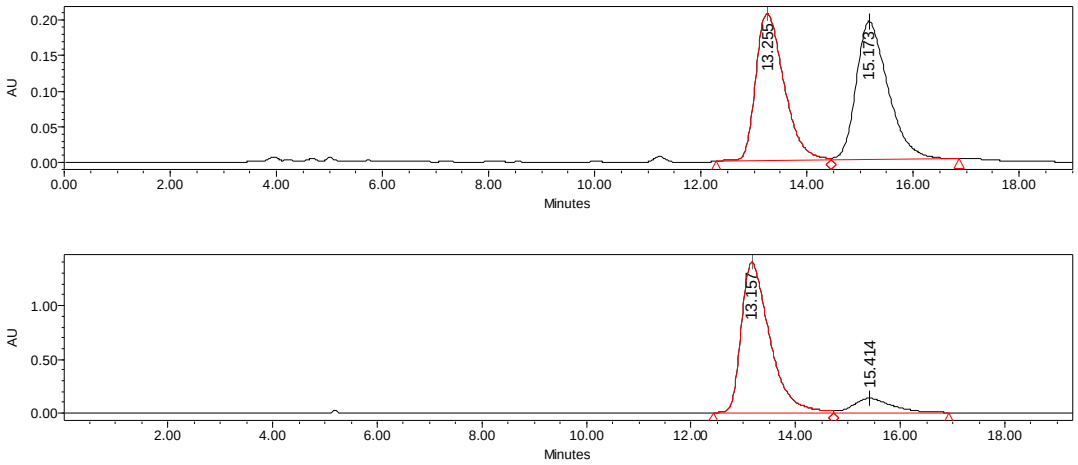
4j: colorless oil**.** The material was determined to be of 88% ee by chiral HPLC analysis [Chiralpak AS-H, 80:20 n-hexane/iPrOH, 1.0 mL/min, t_r (major) = 10.42 min, t_r (minor) = 12.54 min]; $[\alpha]_D^{20}$ = +59.7^o (c = 4.32 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41 (s, 1H), 7.88 (d, J = 2.0 Hz, 1H), 7.32-7.21 (m, 3H), 5.44 (s, 1H), 4.36-4.28 (m, 1H), 4.26-4.18 (m, 1H), 1.28-1.24 (t, J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 135.1, 127.1, 125.5, 124.3, 122.2, 113.5, 113.4, 112.8, 67.2, 62.3, 14.1; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 295.9923, Found: 295.9558.





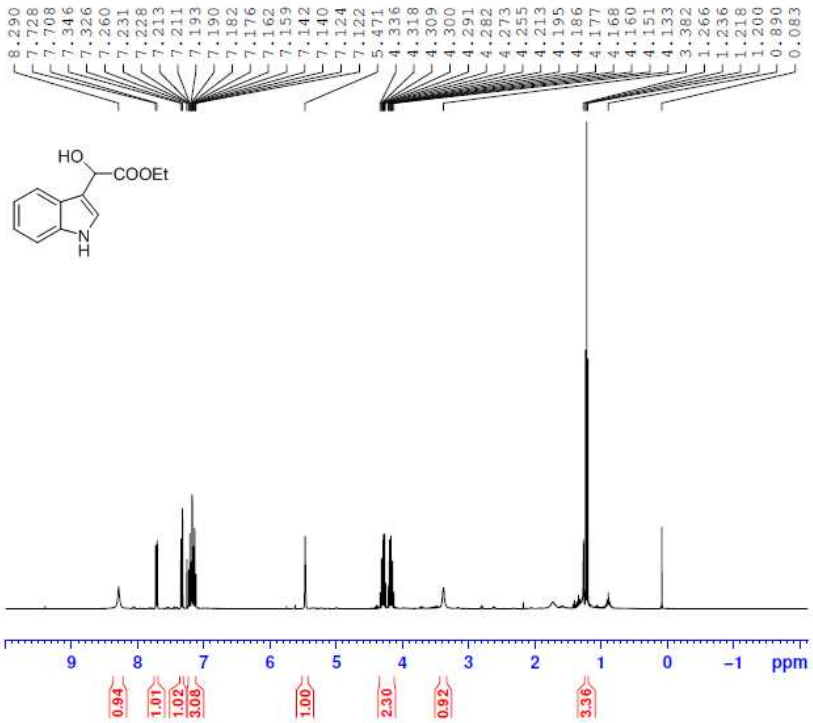


4k: colorless oil. The material was determined to be of 76% ee by chiral HPLC analysis [Chiralpak OD-H, 80:20 *n*-hexane/*i*PrOH, 1.0 mL/min t_r (major) = 13.25 min, t_r (minor) = 15.17 min]; $[\alpha]_D^{20}$ = +90.2° (c = 2.24 in CH_2Cl_2). ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.78 (s, 1H), 8.57 (d, *J* = 2.0Hz, 1H), 8.00-7.97 (m, 1H), 7.57-7.54 (m, 2H), 5.83 (s, 1H), 4.20-4.15 (q, 2H), 1.21-1.18 (t, *J* = 7.0Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.9, 140.9, 140.1, 128.3, 126.0, 117.1, 116.9, 115.1, 112.7, 61.4, 61.1, 14.1; ESI-HRMS Calcd for ($C_{12}H_{13}NO_3$-H^+): 263.0668, Found: 263.0501.



References

1. (a) Wen, Y. H.; Huang, X.; Huang, J. L.; Xiong, Y.; Qin, B.; Feng, X. M. *Synlett* **2005**, 2445. (b) Yu, Z. P.; Liu, X. H.; Dong, Z. H.; Xie, M. S.; Feng, X. M. *Angew. Chem., Int. Ed.* **2008**, *47*, 1308. (c) Zheng, K.; Qin, B.; Liu, X. H.; Feng, X. M. *J. Org. Chem.* **2007**, *72*, 8478. (d) Zhang, X.; Chen, D. H.; Liu, X. H.; Feng, X. M. *J. Org. Chem.* **2007**, *72*, 5227. (e) Zhou, X.; Shang, D. J.; Zhang, Q.; Lin, L. L.; Liu, X. H.; Feng, X. M. *Org. Lett*. **2009**, *11*, 1401.



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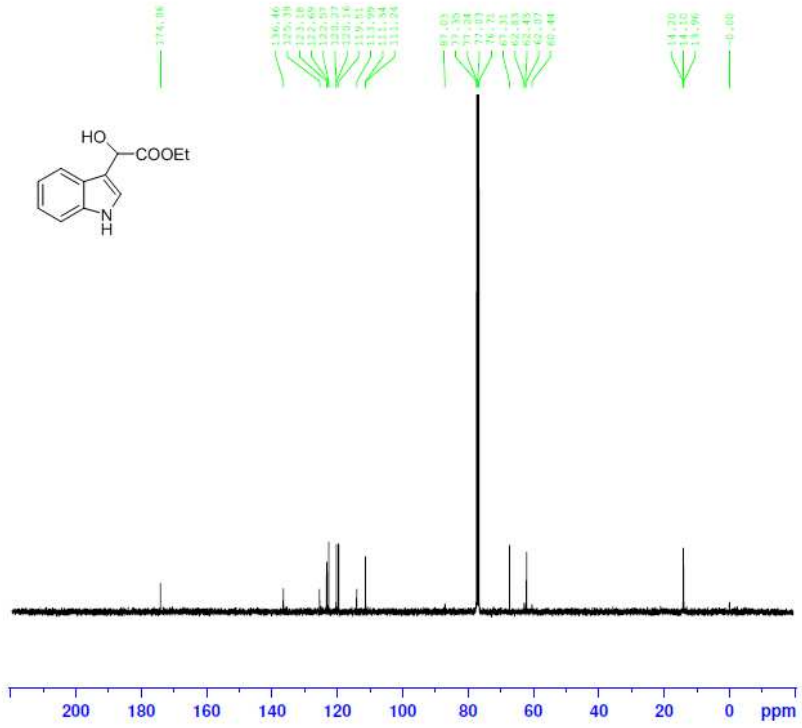
```
Current Data Parameters
NAME      2008-07-10 huiyonghai-A-43
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_              20080710
Time                 19.30
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                     181
DW                  60.800 usec
DE                   6.50 usec
TE                  297.4 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -2.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                   32768
SF            400.1300094 MHz
WDW                     GM
SSB                      0
LB                   -0.20 Hz
GB                     0.1
PC                    1.00
```



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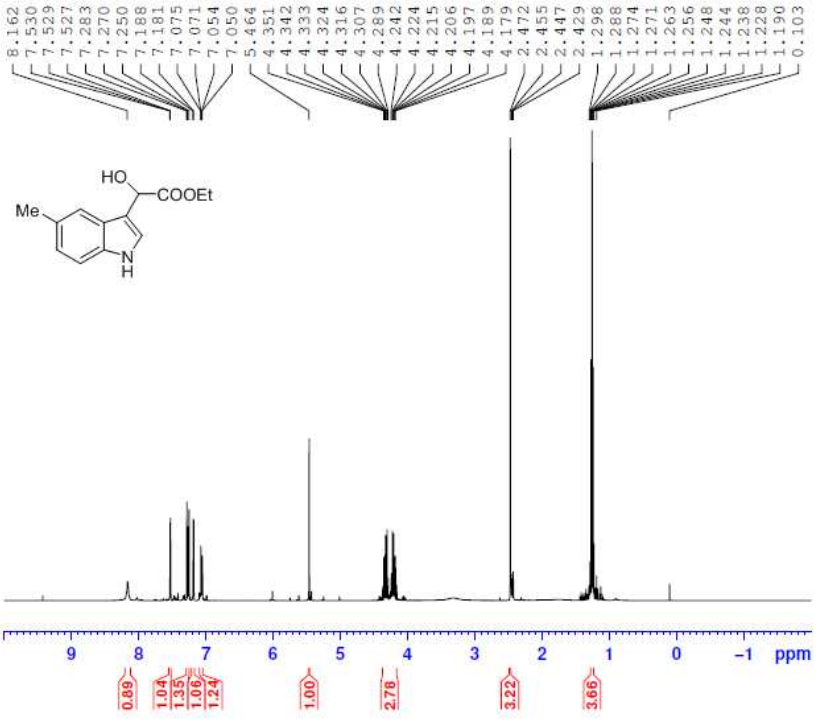
Current Data Parameters
NAME       2008-12-17 huiyonghai DF0
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081217
Time              18.47
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  624
DS                    0
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 1290
DW               20.800 usec
DE                6.50 usec
TE              296.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              11.35 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
```



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Current Data Parameters
NAME       2008-11-21 huiyonghai DF4
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081121
Time              15.14
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                  144
DW               60.800 usec
DE                6.50 usec
TE              295.3 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300000 MHz
WDW                  GM
SSB                   0
LB                -0.20 Hz
GB                   0.1
PC                1.00
```

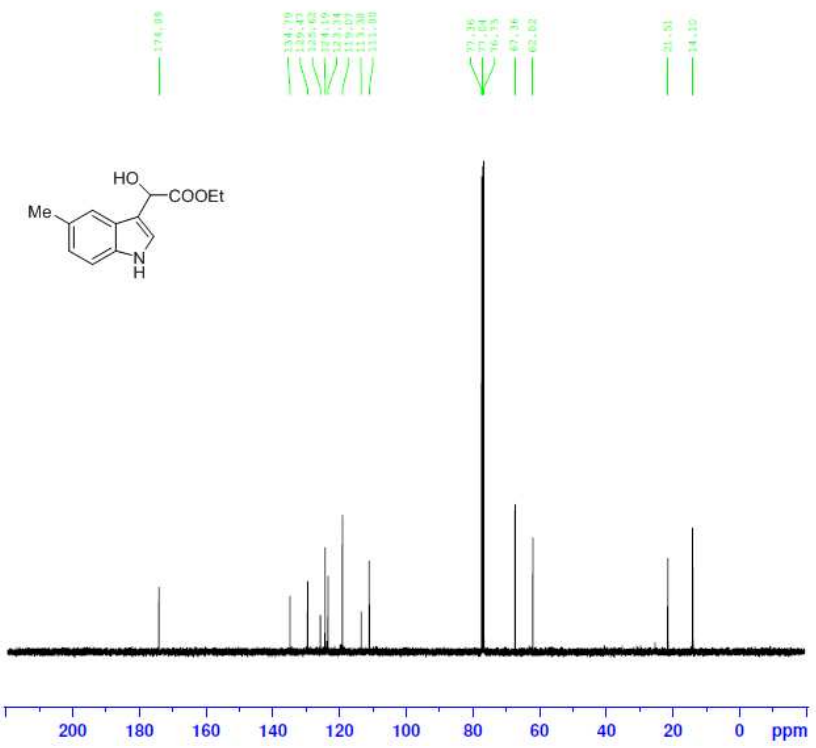


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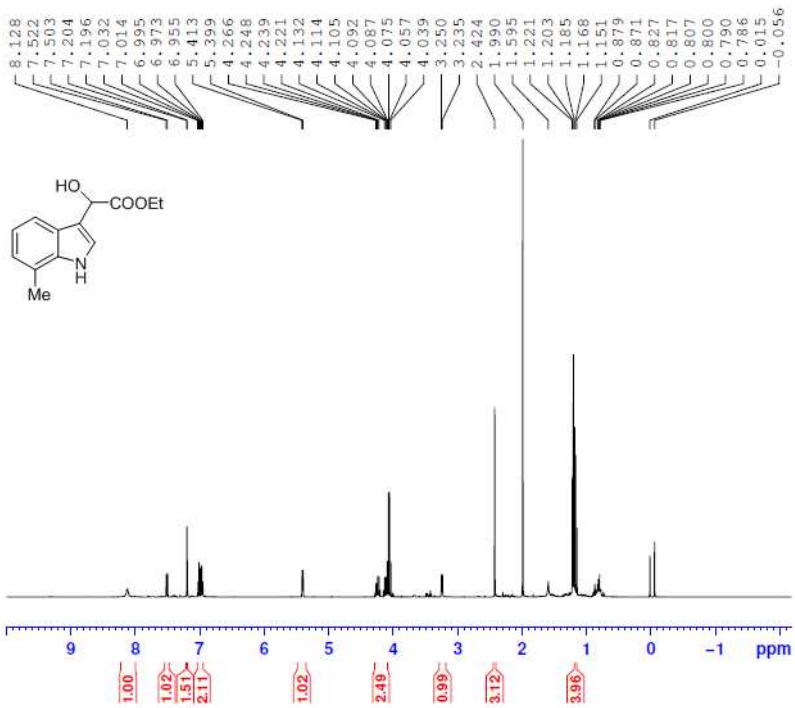
Current Data Parameters
NAME        2008-11-21 huiyonghai DF4
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20081121
Time               15.47
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                 1820
DW               20.800 usec
DE                6.50 usec
TE                297.3 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
PL1               -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             60.00 usec
PL12              11.35 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                 2.00 Hz
GB                    0
PC                 1.40
```

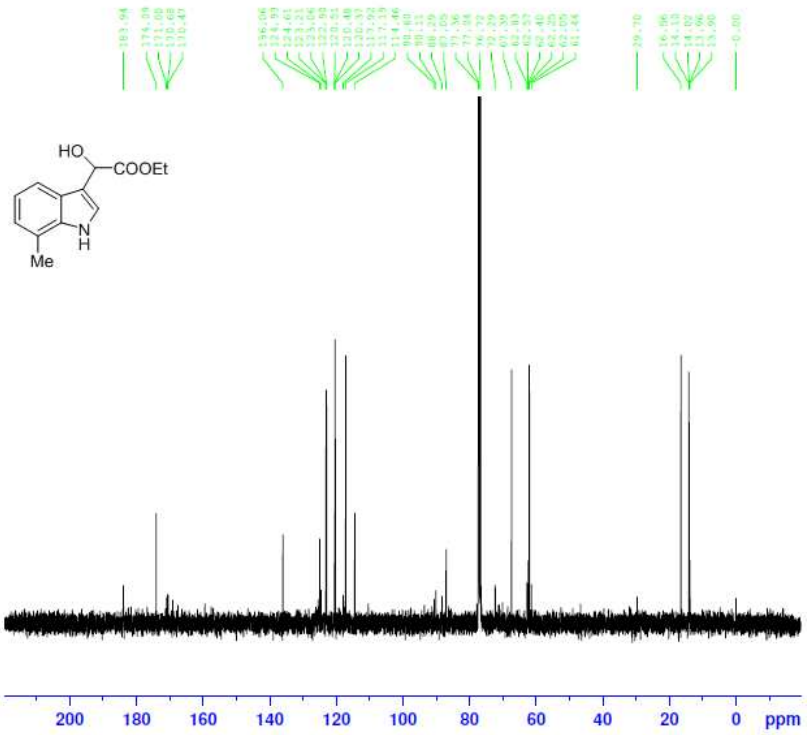



```
Current Data Parameters
NAME        2008-12-16 huiyonghai DF5
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081216
Time               10.51
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                  144
DW               60.800 usec
DE                6.50 usec
TE                295.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300315 MHz
WDW                  GM
SSB                   0
LB                -0.20 Hz
GB                   0.1
PC                 1.00
```

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Current Data Parameters
NAME      2008-12-17 huiyonghai DF5
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081217
Time              20.31
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                 1030
DW               20.800 usec
DE                6.50 usec
TE               297.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
PL1               -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             60.00 usec
PL12              11.35 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
```
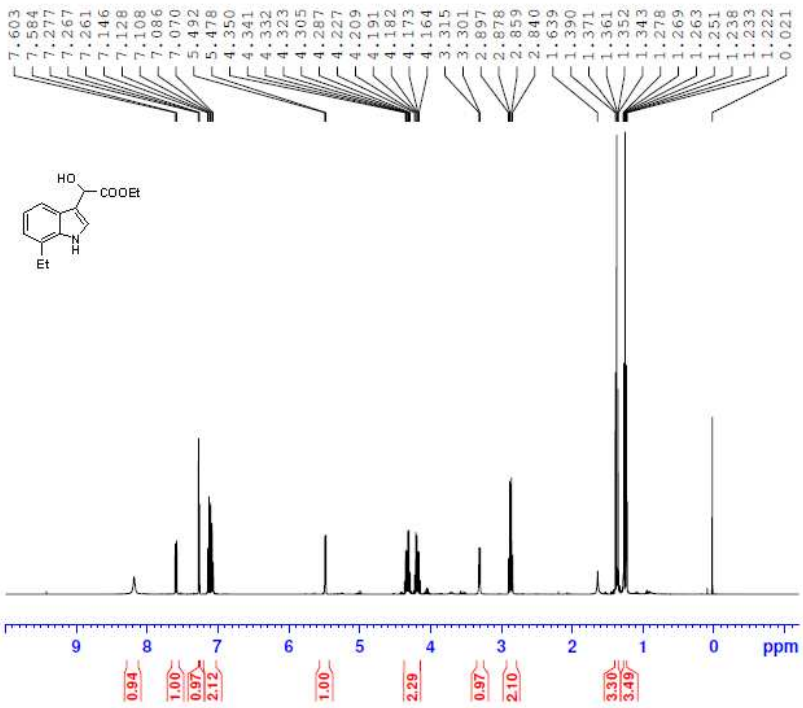
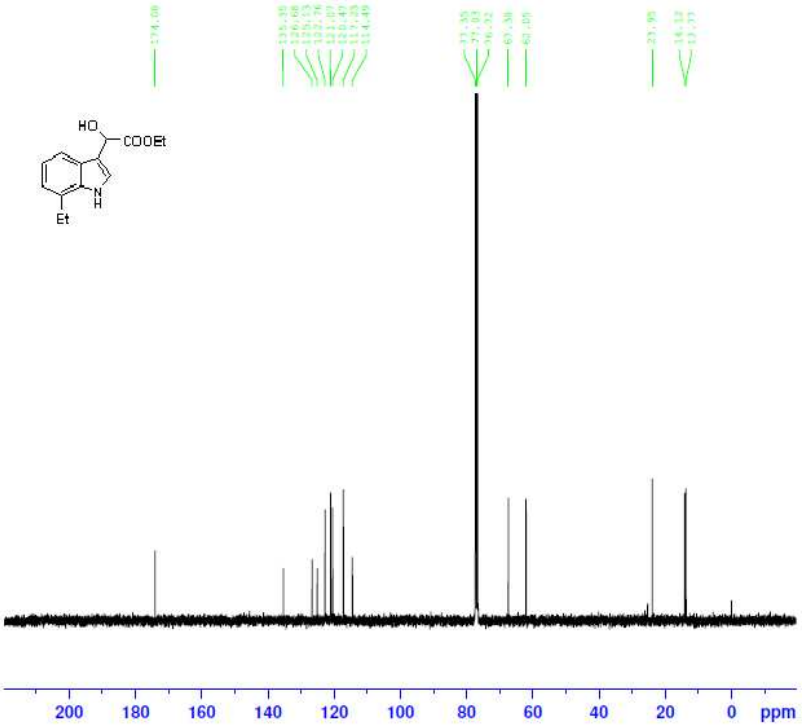




```
Current Data Parameters
NAME      2008-12-23 huiyonghai DF15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081223
Time              11.56
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                  161
DW               60.800 usec
DE                6.50 usec
TE               294.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300025 MHz
WDW                  EM
SSB                   0
LB                -0.20 Hz
GB                   0.1
PC                 1.00
```



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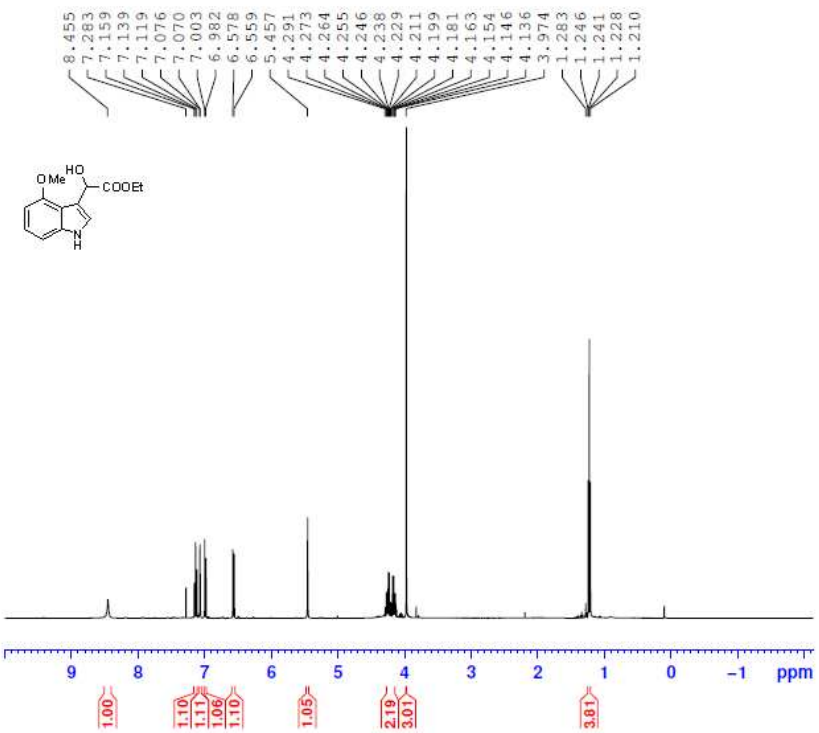
```
Current Data Parameters
NAME       2008-12-24 huiyonghai DF15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081224
Time              21.05
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               296.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
PL1                -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              60.00 usec
PL12               11.35 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
```



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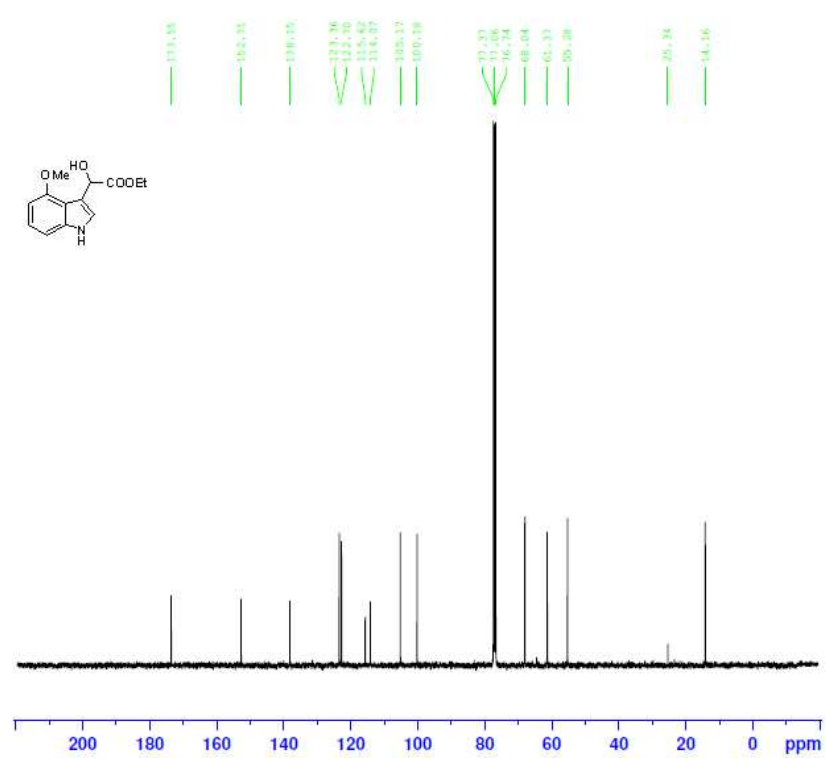
```
Current Data Parameters
NAME      2008-11-21 huiyonghai DF2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20081121
Time              17.33
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            3.9846387 sec
RG                 144
DW              60.800 usec
DE               6.50 usec
TE              295.9 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                -0.20 Hz
GB                  0.1
PC                 1.00
```



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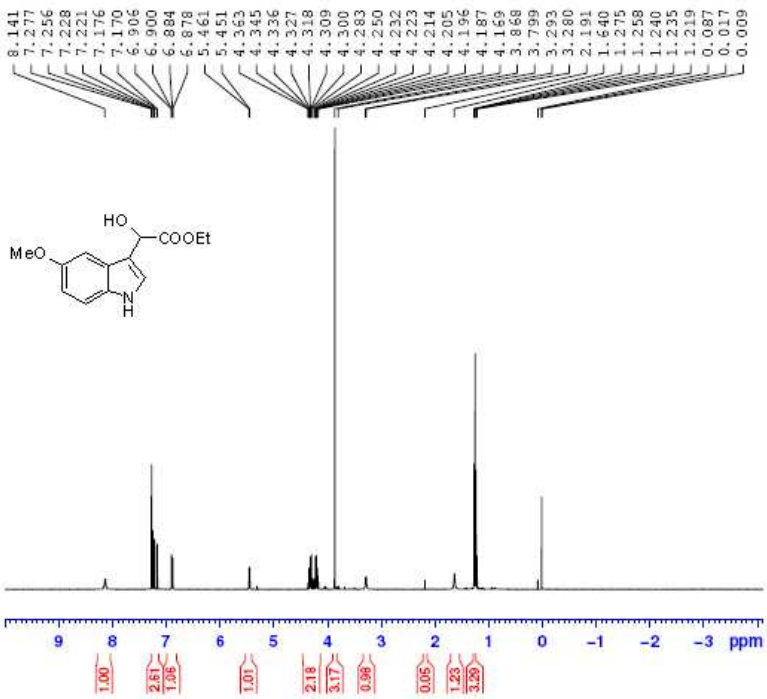
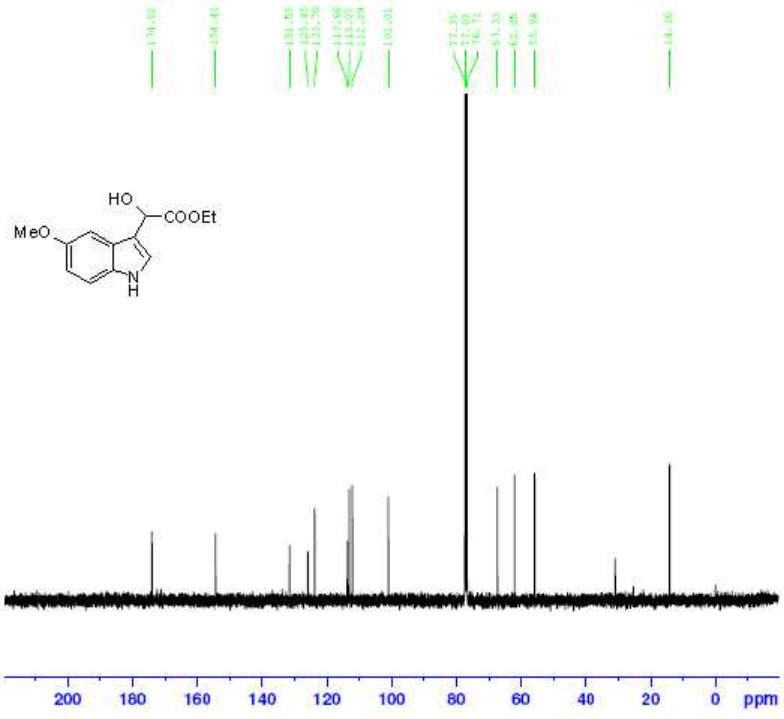
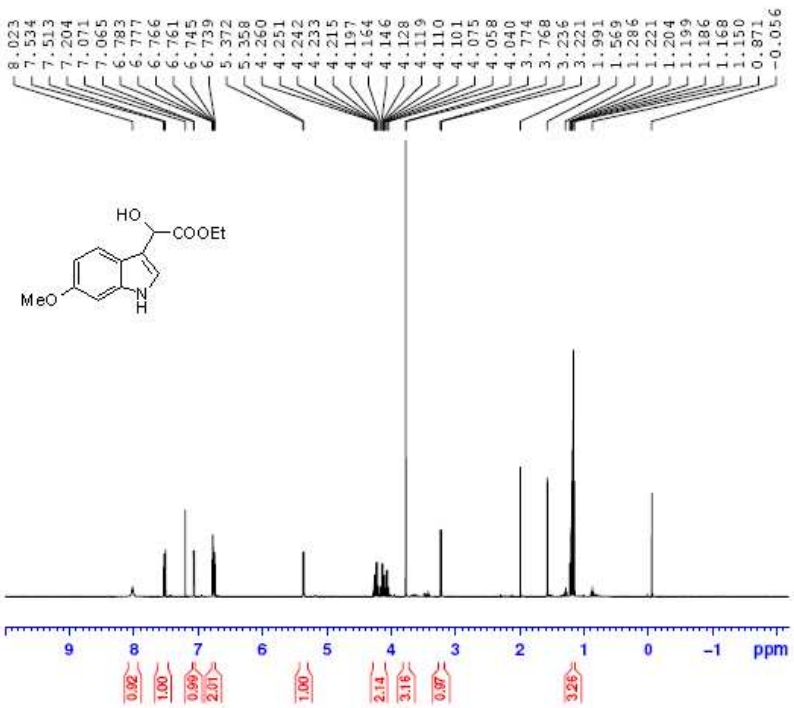
```
Current Data Parameters
NAME      2008-11-21 huiyonghai DF2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081121
Time              18.06
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              297.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              11.35 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                 2.00 Hz
GB                    0
PC                 1.40
```



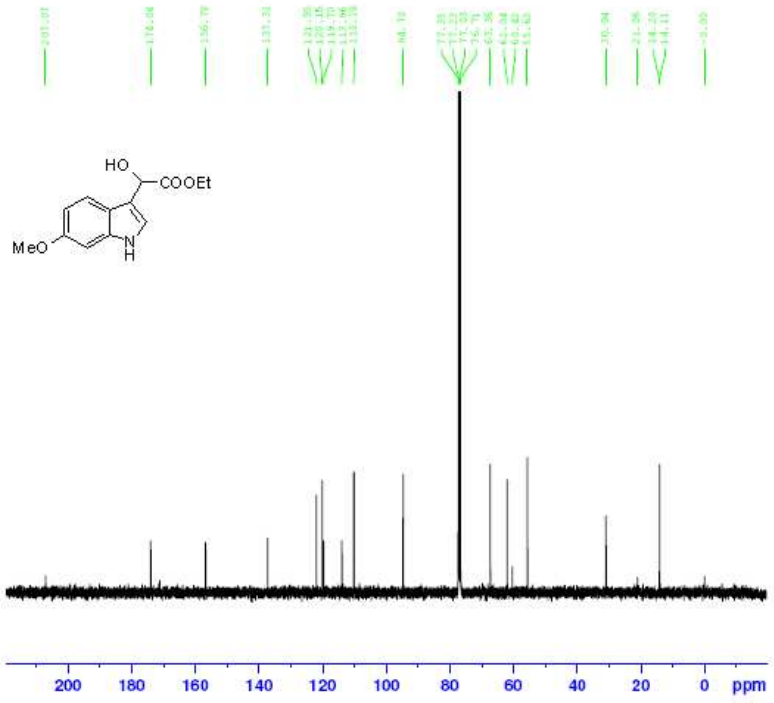

```
Current Data Parameters
NAME      2008-12-23 hulyonghai DP12
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081223
Time              11.34
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           8223.685 Hz
FIDRES        0.125493 Hz
AQ            3.9846387 sec
RG                 161
DW              60.800 usec
DE               6.50 usec
TE              294.3 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300025 MHz
WDW                  EM
SSB                   0
LB               -0.20 Hz
GB                  0.1
PC                 1.00
```



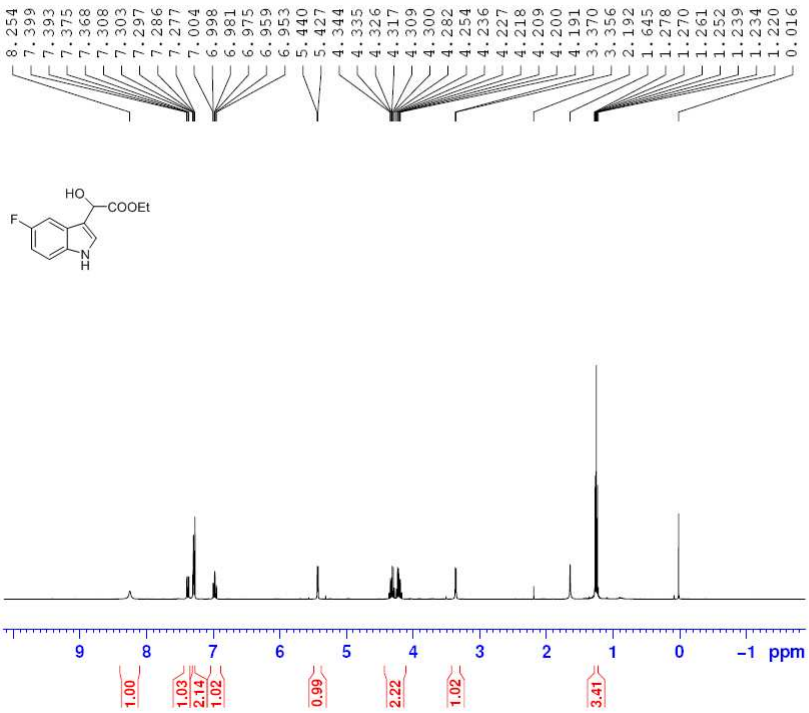

```
Current Data Parameters
NAME      2008-12-24 hulyonghai DP12
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081224
Time              20.33
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              296.6 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.99999999 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1        100.6228299 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              11.35 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
```



```
Current Data Parameters
NAME        2008-12-16 hulyonghai DF3
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20081216
Time              10.43
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                 144
DW               60.800 usec
DE                6.50 usec
TE               295.3 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI               32768
SF          400.1300315 MHz
WDW                 EM
SSB                  0
LB               -0.20 Hz
GB                 0.1
PC                1.00
```



```
Current Data Parameters
NAME        2008-12-17 hulyonghai DF3
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20081217
Time              19.53
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                1030
DW               20.800 usec
DE                6.50 usec
TE               297.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                15.50 usec
PL1               -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              11.35 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
```

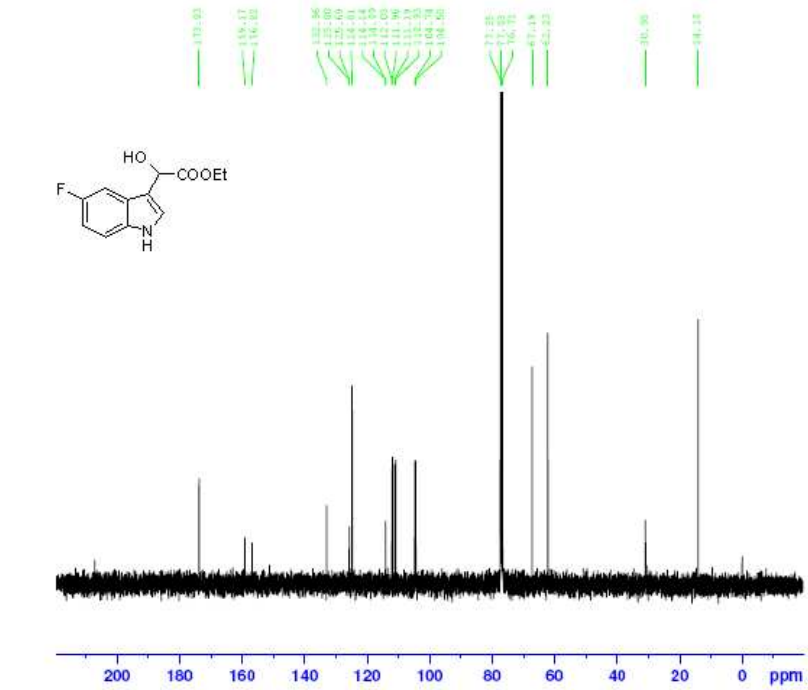


Current Data Parameters
NAME 2008-12-23 huiyonghai DF13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081223
Time 11.40
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 161
DW 60.800 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1

-------- CHANNEL f1 --------
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300025 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00



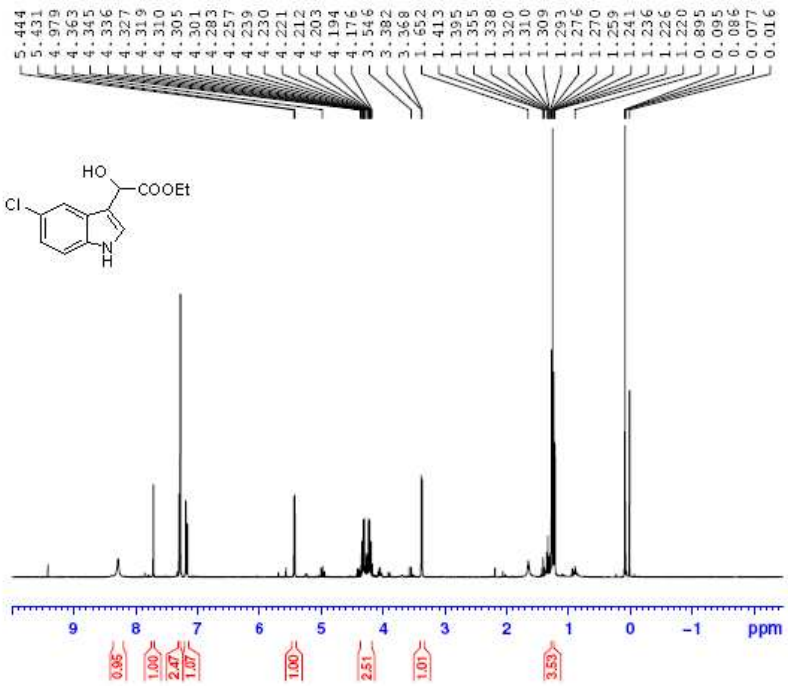
Current Data Parameters
NAME 2008-12-24 huiyonghai DF13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081224
Time 22.15
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 2850
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

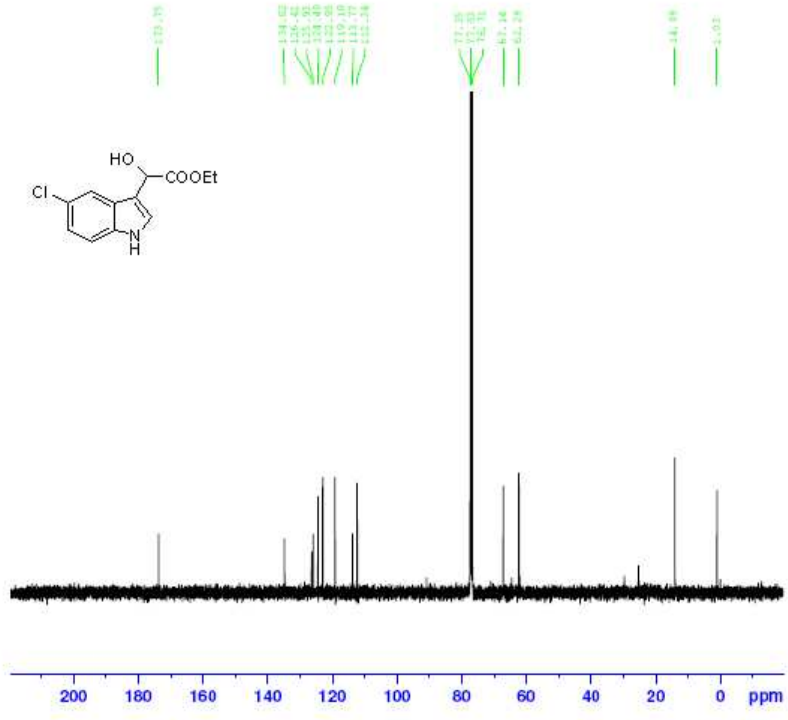
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL12 11.35 dB
PL13 13.05 dB
PL2 -2.00 dB
SFO2 400.1316005 MHz

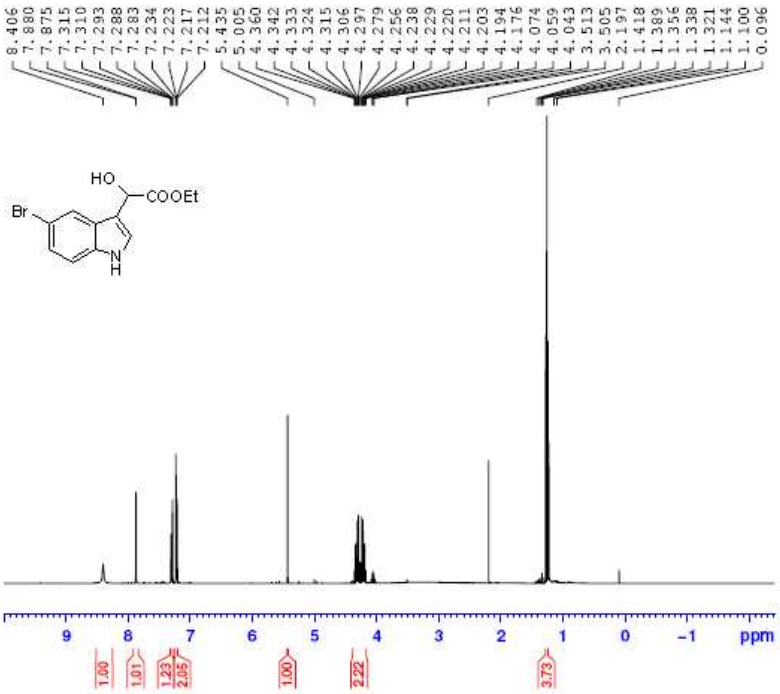
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

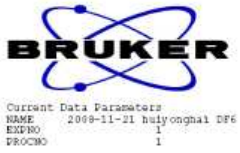


```
Current Data Parameters
NAME        2008-12-23 hulyonghai DF14
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081223
Time              11.48
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            3.9846397 sec
RG                  161
DW             60.800 usec
DE              6.50 usec
TE             300.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300025 MHz
WDW                  EM
SSB                   0
LB               -0.20 Hz
GB                  0.1
PC                 1.00
```

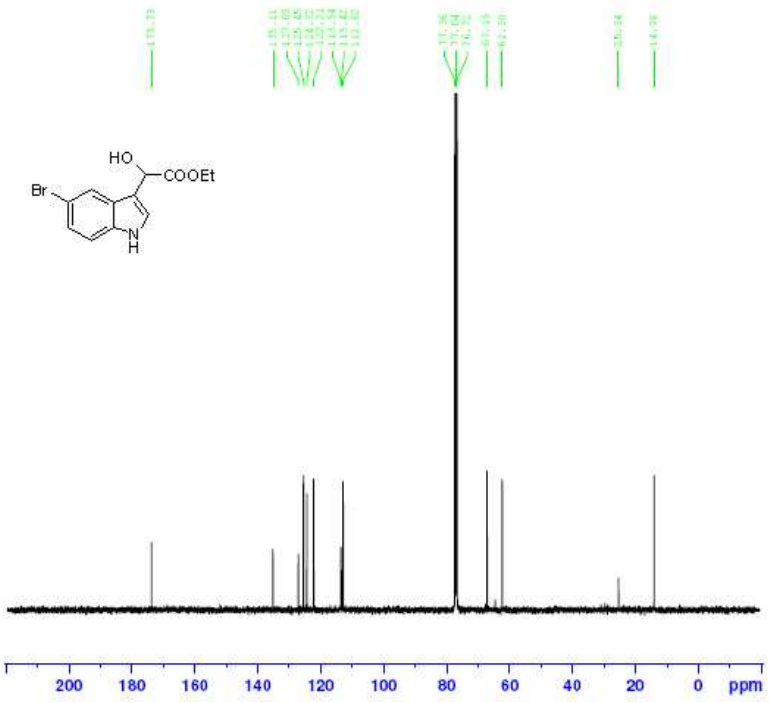


```
Current Data Parameters
NAME        2008-12-24 hulyonghai DF14
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081224
Time              21.43
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
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             295.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
PL1               -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             60.00 usec
PL12              11.35 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
```

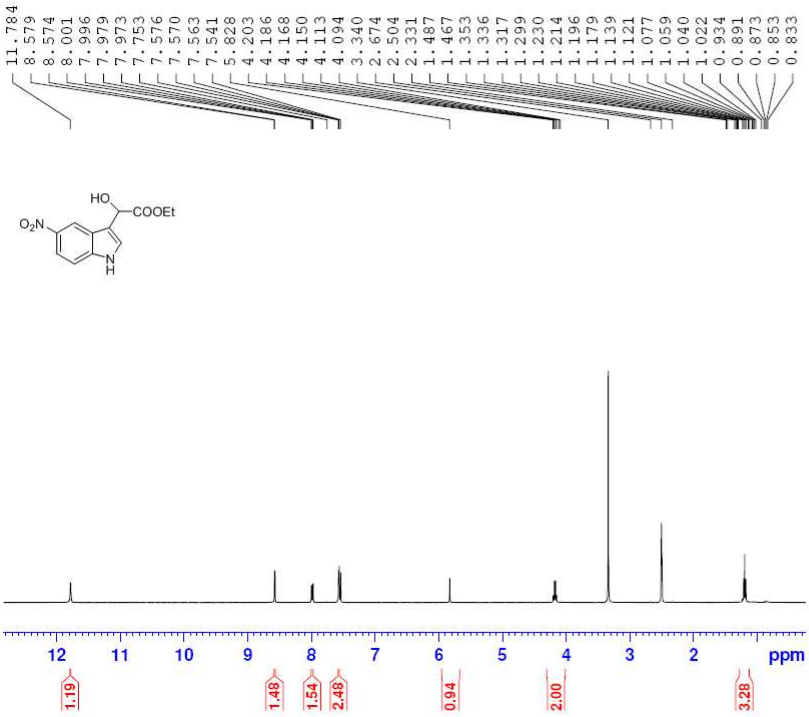




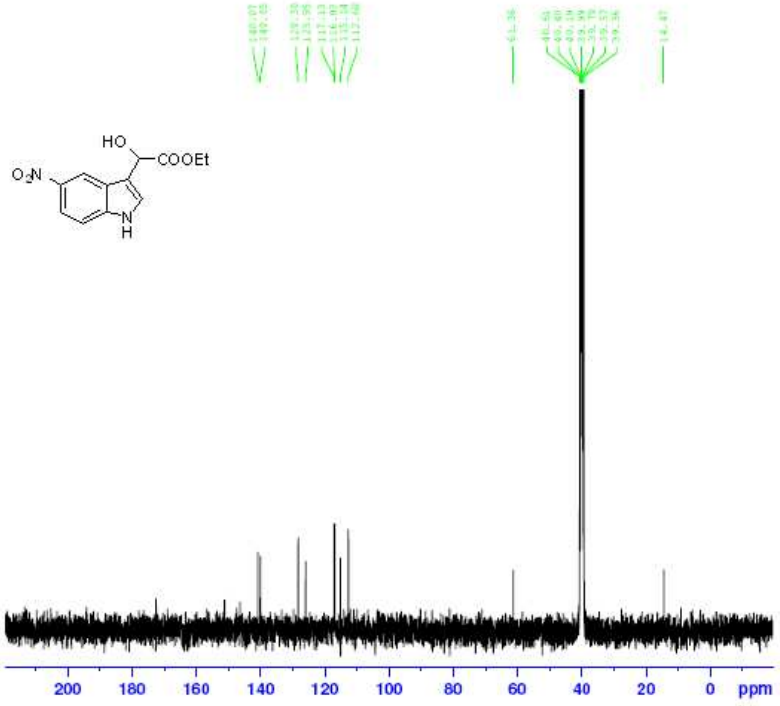
```
Current Data Parameters
NAME      2008-11-21 hulyonghai DF6
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081121
Time              15.52
INSTRUM           spect
PROBHD  5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125493 Hz
AQ            3.9846387 sec
RG                  144
DW             60.800 usec
DE              6.50 usec
TE             300.0 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1             12.00 usec
PL1            -2.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI               32768
SF        400.1300000 MHz
WDW                 EM
SSB                  0
LB             -0.20 Hz
GB                 0.1
PC                1.00
```



```
Current Data Parameters
NAME      2008-11-21 hulyonghai DF6
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20081121
Time              17.26
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             297.6 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1             15.50 usec
PL1            -1.00 dB
SFO1      100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2          60.00 usec
PL12           11.35 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              2.00 Hz
GB                   0
PC                1.40
```



```
Current Data Parameters
NAME      2008-12-25 huiyonghai-DF8
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20081225
Time               17.31
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                   406
DW                60.800 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300007 MHz
WDW                   EM
SSB                    0
LB                  0.60 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME      2008-12-25 huiyonghai-DF8
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20081225
Time               18.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
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                296.9 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
PL1                -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              68.00 usec
PL12               11.35 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                  2.00 Hz
GB                     0
PC                  1.40
```