Supporting Information

Practical and Stereoselective Synthesis of β-Amino Sulfones from Alkyl Phenyl Sulfones and N-(tert-Butylsulfinyl) Aldimines

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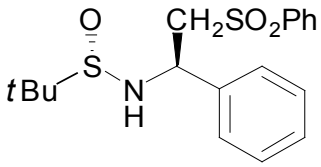
General:

Unless otherwise mentioned, solvents and reagent were purchased from commercial sources and used as received. THF was freshly distilled over sodium. N-(tert-Butanesulfinyl)imines and alkylphenyl sulfones were prepared using known procedures. ^{1}H NMR spectra were recorded on 400 MHz spectrometers with Me_4Si as internal standard. ^{13}C NMR spectra were recorded on 100 MHz spectrometers. Mass spectra were taken on a HP5989A spectrometer. High-resolution mass data were recorded on a high-resolution mass spectrometer in the ESI or MALDI mode.

Preparation and physical data of compounds **3** and **5**

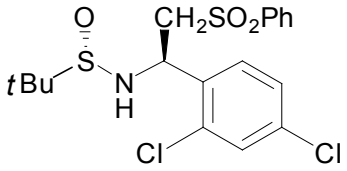
Typical procedure for the synthesis of compound 3a.

LiHMDS (1.3 equiv, 1.3 ML, 1.0 mol/L) was added to a mixture of the imine **2a** (1 mmol) and methylphenyl sulfone **1** (1.3 equiv, 1.3 mmol) in THF (5 mL) at – 70 ^{o}C. Reaction mixtures were stirred over 1 h. Then half-saturated NH_4Cl-H_2O solution (2 mL) was added at lower temperature and the quenched reaction mixture was extracted three times with ethyl acetate. The combined organic layers were dried over anhydrous $MgSO_4$. Evaporation of the solvent afforded the crude product, which was subject to flash chromatography to give the corresponding sulfonamide **3a** (340 mg, 93 %).



3a

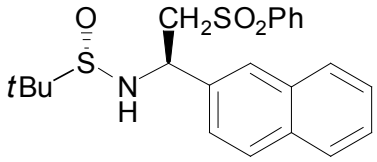
White solid, mp 144.2-145.4 ^{o}C; $[\alpha]_D^{25}$ – 0.16 (c = 0.77, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ/ppm: 7.83-7.94 (m, 2H), 7.61-7.65 (m, 1H), 7.50-7.54（m, 2H), 7.28-7.32（m,5H), 5.01-5.05(m,1H), 4.63(d, J = 3.2 Hz, 1H), 3.93 (d,d J^1 = 14.4 Hz, J^2 = 8.4 Hz,1H), 3.52 (d,d J^1 = 14.4 Hz, J^2 = 4.4 1H),1.24 (s, 9H); ^{13}CNMR (100 MHz, $CDCl_3$) δ/ppm: 139.5, 138.3, 133.8, 129.3, 128.9, 128.6, 127.8, 127.6, 61.7, 56.4, 54.9, 22.4; MALDI calcd. For $C_{18}H_{24}NO_3S_2$ $[M+H]^+$: 366.1192, Found 366.1188



3b

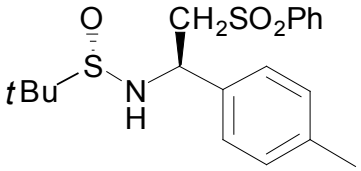
White solid, mp 112.9-115.4^{o}C; $[\alpha]_D^{25}$ 9.29 (c = 0.76, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ/ppm: 7.80-7.90 (m, 2H), 7.66-7.63 (m, 1H), 7.54-7.48 (m,3H), 7.28-7.25 (m,2H), 5.31-5.28 (m,1H), 5.05(d, J = 6 Hz, 1H), 3.91 (dd, J^1 = 14.0 Hz, J^2 = 8.0 Hz, 1H), 3.56 (dd, J^1 = 14.4 Hz, J^2 = 4.0, 1H),1.25 (s, 9H); ^{13}C NMR ($CDCl_3$) δ/ppm: 139.2, 135.1,

134.9, 134.5, 133.9, 133.3, 130.6, 129.7, 129.3, 127.7, 60.0, 56.8, 52.5, 22.4；MALDI calcd.For $C_{18}H_{22}NO_3S_2Cl_2$ $[M + 1]^+$: 434.0413, Found 434.0401



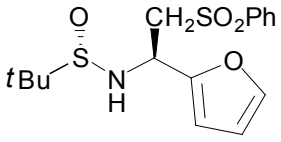
3c

White solid, mp 143.2-144.1°C; $[\alpha]_D^{25}$ 21.59 (c = 0.73, $CHCl_3$);^{1}H NMR ($CDCl_3$) δ/ppm: 7.89-7.42 (m, 12H), 5.88 (d, J = 6.8, 1H), 5.17 (s, 1H), 4.27 (dd, J^1 = 13.6 Hz, J^2 = 9.6Hz, 1H), 3.70 (d , J = 13.6 Hz, 1H),1.19 (s, 9H);^{13}C NMR ($CDCl_3$) δ/ppm:139.3, 133.9, 133.9, 133.0, 129.5, 129.0, 127.8, 126.7, 125.9, 125.6, 122.9, 61.0, 56.0, 50.7, 22.5; MALDI calcd.For $C_{22}H_{26}NO_3S_2$ $[M^+ + H]^+$: 416.1349, Found 416.1340



3d

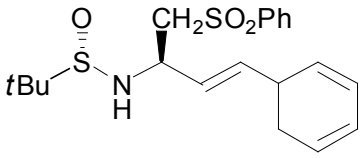
White solid, mp:76.4-78.4°C; $[\alpha]_D^{25}$ 28.89 (c = 0.72, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ/ppm: 7.79 (d, 2H), 7.85-7.82 (m, 2H), 7.63-7.61 (m,1H), 7.54-7.49 (m, 2H), 7.21-7.12 (m, 2H), 7.12-7.10 (m, 2H), 4.99-4.97 (m, 1H), 4.60 (d, J = 3.2, 1H), 3.91 (dd, J^1 = 14.4Hz, J^2 = 8.4Hz, 1H), 3.50 (dd, J^1 = 14.0Hz, J^2 = 9.6 Hz, 1H), 1.23 (s, 9H); ^{13}C NMR ($CDCl_3$) δ: 139.6, 138.5, 135.2, 133.7, 139.5, 129.5, 129.3, 127.8, 127.5, 61.7, 56.3, 54.6, 22.5, 21.0; MALDI calcd.For $C_{19}H_{26}NO_3S_2$ $[M + H]^+$: 380.1349, Found 380.1348



3e

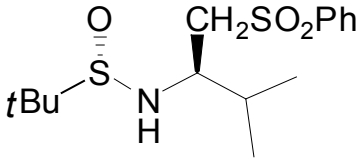
Yellow oil, $[\alpha]_D^{25}$ 37.51 (c = 0.61, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ/ppm: 7.84-7.83 (m, 2H), 7.65-7.61 (m, 1H), 7.55-7.51 (m, 2H), 7.22 (t, 1H), 6.38 (d, J = 3.2 Hz, 1H), 6.27 (m, 1H), 5.06 (m, 1H), 3.91(dd, J^1 = 14.4 Hz, J^2 = 7.6 Hz, 1H), 3.66 (dd, J^1 = 14.4 Hz,

J^2 = 5.2 Hz, 1H); 1.23 (s, 9H); ^{13}C NMR ($CDCl_3$) δ/ppm: 150.7, 142.8, 139.5, 133.7, 127.8, 110.6, 109.2, 59.6, 56.5, 49.6, 22.4; MALDI calcd.For $C_{16}H_{22}NO_4S_2$ $[M + H]^+$: 356.0985, Found 356.0991



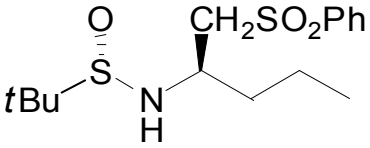
3f

White solid, mp 99.1-103.3°C; $[\alpha]_D^{25}$ 57.82 (c = 0.66, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.93 (m, 2H), 7.67-7.64 (m, 2H), 7.58-7.54 (m, 2H), 7.32-7.31 (m, 5H) ,6.65 (dd, J^1 = 15.6 Hz, J^2 = 0.8Hz, 1H), 6.22 (dd, J^1 = 15.6Hz, J^2 = 7.2Hz, 1H), 3.75 (dd, J^1 = 14.0 Hz, J^2 = 8 Hz,1H), 3.42 (J^1 =14.0 Hz, J^2 = 8 Hz,1H), 1.27(s, 9H); ^{13}C NMR ($CDCl_3$) δ/ppm: 139.7, 135.6, 133.9, 133.9, 129.4, 128.5, 128.3, 127.9, 126.8, 126.5, 61.1, 56.4, 53.6, 22.5; MALDI calcd. For $C_{20}H_{25}NO_3S_2$ $[M + Na]^+$:414.1168, Found 414.1171



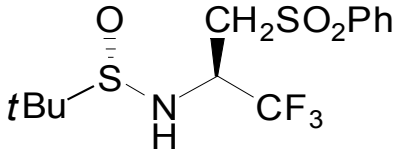
3g

White solid, mp 114.2-115.4°C; $[\alpha]_D^{25}$ 21.65 (c =0.52, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm 7.96-7.94 (m, 2H), 7.69-7.66 (m, 1H), 7.62-7.58 (m, 1H), 4.07 (d, J = 5.6Hz, 1H), 3.71-3.66 (m, 1H), 3.52-3.46 (m, 1H), 3.22 (dd, J^1 = 14.0 Hz, J^2 = 3.2 Hz, 1H), 2.28 (m, 1H), 1.26 (s, 9H), 0.98 (m, 6H); ^{13}C NMR ($CDCl_3$) δ/ppm: 104.2, 75.8, 57.5, 29.1, 22.9, 22.3, 16.9; MALDI calcd.For $C_{15}H_{26}NO_3S_2$ $[M + H]^+$:332.1349, Found 332.1355



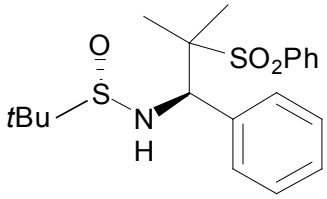
3h

White solid, mp 64.3-67.5°C; $[\alpha]_D^{25}$ 33.27 (c =1.02, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.97 (m, 2H), 7.70-7.62 (m, 1H), 7.60-7.58 (m, 1H), 4.31 (d, J = 4.8 Hz, 1H), 3.92 (m, 1H), 3.72 (dd, J^1 = 14.0 Hz, J^2 = 8.0 Hz, 1H), 3.17 (dd, J^1 = 14.0 Hz, J^2 = 3.2 Hz, 1H), 1.93-1.92 (m, 1H), 1.76-1.72 (m, 1H), 1.45-1.39 (m, 2H), 1.25 (s, 9H), 0.938 (t, J = 7.2Hz, 3H); ^{13}C NMR ($CDCl_3$) δ/ppm: 139.9, 133.8, 129.4, 127.8, 61.4, 56.0, 36.3, 22.5, 19.0, 13.4; MALDI calcd.For $C_{15}H_{26}NO_3S_2$ $[M+H]^+$:332.1349, Found 332.1348



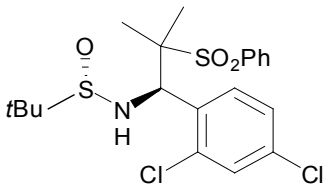
3i

White solid, mp 124.6-125.1°C; $[\alpha]_D^{25}$ 16.32 (c =1.02, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.97 (m, 2H), 7.74-7.71 (m, 1H), 7.65-7.62 (m, 2H), 4.48 (m, 1H), 4.04 (d, J = 9.2 Hz, 1H), 3.61 (dd, J^1 = 14.4 Hz, J^2 = 9.6Hz, 1H), 3.45 (dd, J^1 = 14.4 Hz, J^2 = 2.8 Hz, 1H), 1.30 (s, 9H), 0.938 (t, J = 7.2Hz, 3H); ^{13}C NMR ($CDCl_3$) δ/ppm: 139.3, 134.4, 129.6, 127.9, 57.6, 55.4, 54.2, 53.9, 22.3; ^{19}F NMR (DMSO) δ/ppm: 73.7-73.8 (d, 3F), MALDI calcd.For $C_{13}H_{19}NO_3S_2F_3$ $[M+H]^+$:358.0753, Found 358.0744



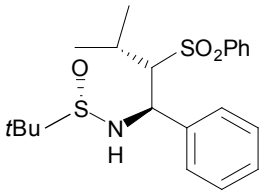
5a

White solid, mp 127.1-129.1 °C; $[\alpha]_D^{25}$ -47.99 (c = 0.70, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.71 (d, 2H), 7.70-7.59 (t, 1H), 7.50-7.48 (t, 2H), 7.42-7.40 (m, 2H), 7.38-7.35 (m, 3H), 5.80 (d, J = 2Hz, 1H), 4.98 (d, J = 2.4Hz, 1H), 1.43 (s, 3H), 1.33 (s,9H), 0.88 (s, 3H); ^{13}C NMR ($CDCl_3$) δ/ppm: 130.5, 129.8, 128.9, 128.4, 127.9, 127.2, 66.1, 63.2, 60.9, 57.4, 55.6, 55.5, 42.3, 22.6, 16.5, 14.0; MALDI calcd.For $C_{20}H_{28}NO_3S_2$ $[M+H]^+$:394.1505, Found 394.1503



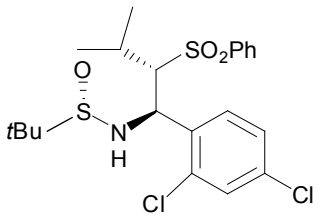
5b

White solid, mp 68.7-70.1°C; $[\alpha]_D^{25}$ -46.49 (c = 0.57,$CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.92 (d, J = 7.6 Hz, 2H), 7.78 (d, J = 8.8 Hz, 1H), 7.72 (t, J = 7.2, 1H), 7.62 (t, J = 8.2Hz, 2H), 7.42-7.36 (m, 2H), 5.80 (d, J = 2.4, 1H), 5.62 (d, J = 2.8, 1H), 1.45 (s, 3H), 1.34 (s, 9H), 0.99 (s, 3H); ^{13}C NMR ($CDCl_3$) δ/ppm: 130.5,129.8,128.9, 128.4, 127.9, 127.2, 66.1, 63.2, 60.9, 57.4, 55.6, 55.5, 42.3, 22.6, 16.5,14.0; ESI-HRMS calcd.For $C_{20}H_{25}Cl_2NO_3S_2$ $[M + Na]^+$:484.0551, Found 484.0554



5c

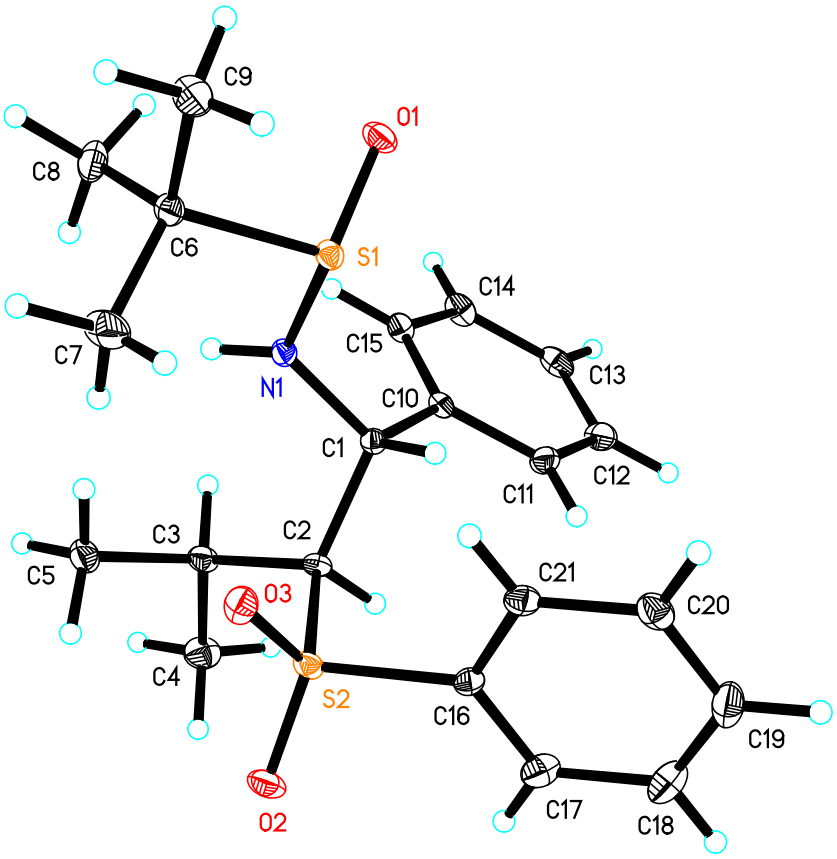
White solid, mp121.0-122.5°C; $[\alpha]_D^{25}$ 3.92 (c = 0.20, $CHCl_3$); ^1H NMR ($CDCl_3$) δ/ppm: 7.73 (d, J = 7.2Hz, 2H), 7.72-7.58 (m, 1H), 7.49 (t, J = 8.0Hz, 2H), 7.36-7.21(m, 5H), 5.11(d, J = 8.0 Hz, 1H), 4.38 (d, J = 8.0 Hz, 1H), 3.49 (dd, J^1 = 4.4 Hz, J^2 = 3.2 Hz, 1H), 2.52 (m, 1H), 1.21(d, J = 2.0 Hz, 3H), 1.19 (d, J = 1.6Hz, 3H); ^{13}C NMR ($CDCl_3$) δ/ppm: 140.7, 139.3, 133.2, 129.1, 128.6, 128.6, 128.0, 127.9, 127.8, 127.4, 127.2, 74.7, 60.9, 59.3, 56.9, 26.9, 22.6, 22.3, 20.2; MALDI calcd.For $C_{21}H_{30}NO_3S_2$ $[M + H]^+$:408.1662, Found 408.1650



5d

White solid, mp 151.3-153.7°C; $[\alpha]_D^{25}$ 5.94 (c = 0.63, $CHCl_3$); ^1H NMR (DMSO) δ/ppm: 7.62-7.61 (m, 1H), 7.58-7.54 (m, 5H), 6.78-6.76 (d, J = 8.4 Hz, 1H), 5.33 (d, J = 11.2 Hz, 1H), 5.11 (m, 1H), 3.97 (d, J = 8.8 Hz,1H); ^{13}C NMR ($CDCl_3$) δ/ppm: 140.3, 134.9, 134.9, 133.1, 132.8, 129.8, 128.9, 127.6, 127.4, 70.0, 57.1, 26.7, 22.5, 21.3, 19.9; ESI-HRMS calcd.For $C_{21}H_{27}NO_3S_2Cl_2$ $[M+Na]^+$: 498.0707, Found 498.0710

Determination of the configuration of **5c** by X-ray analysis

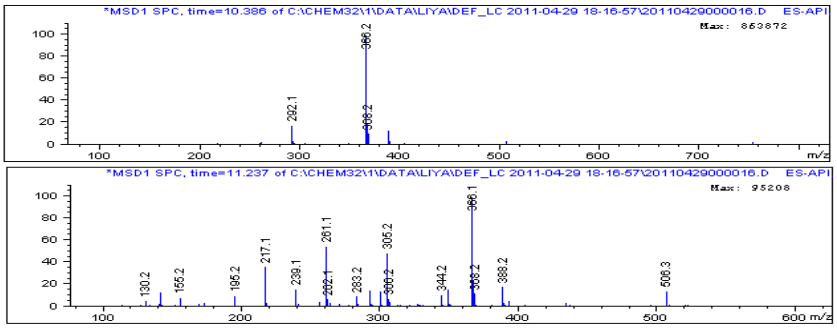


Example of determination of dr ratio for **3a**/**3a'** (entry 1, Table 1) by ^{1}H NMR and HPLC-MS

The dr value has been determined by use of a combination of ^{1}H NMR and HPLC-MS spectra analysis on the crude product. High diastereoselectivity was observed in this reaction, which can be roughly determined based on the ^{1}H NMR on the crude product. The relatively precise dr value was determined by HPLC-MS (Figure 1), and, based on selected ion chromatogram (Figure 1c), the two diasteromers can be found at rt = 10.27 min and 11.00 min respectively.

3a, rt = 10.27 min; **3a'**, rt = 11.10 min

dr = 68.00 : 0.10 = 99 : 1



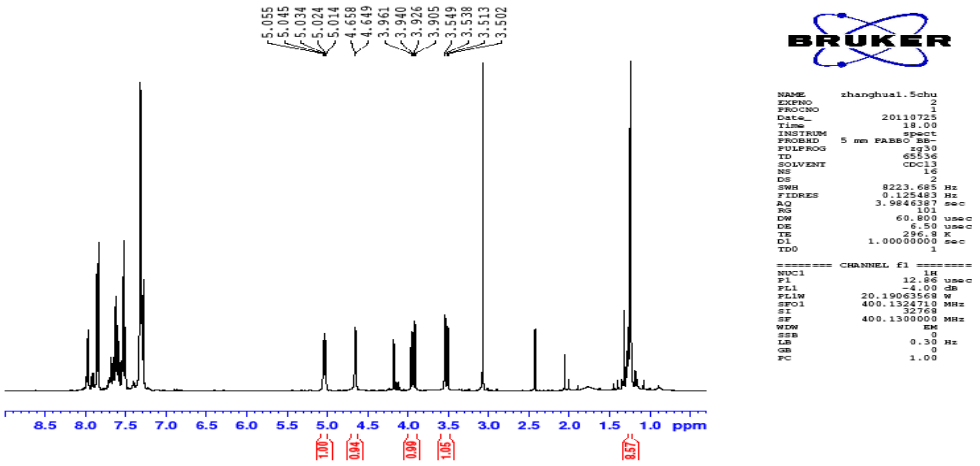
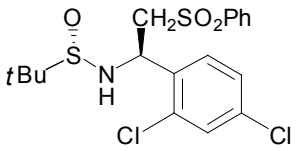


Figure 2. ^{1}H NMR on the crude product **3a**

^{1}H NMR and HPLC-MS spectra on the crude products for determination of dr ratio of **3/3' and 5/5'**



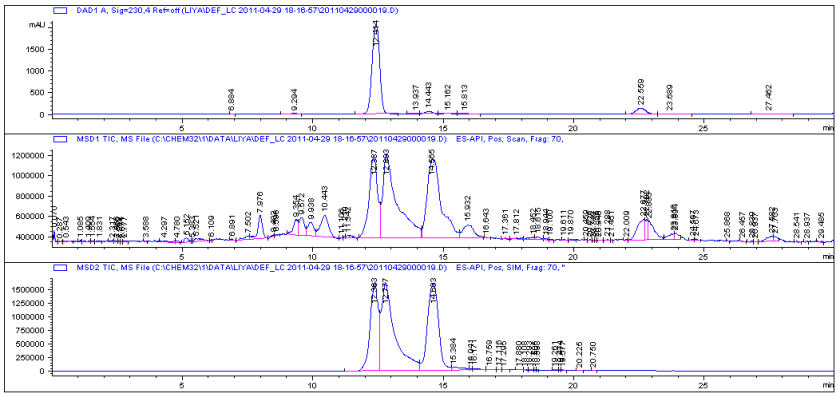
$C_{18}H_{21}Cl_2NO_3S_2$

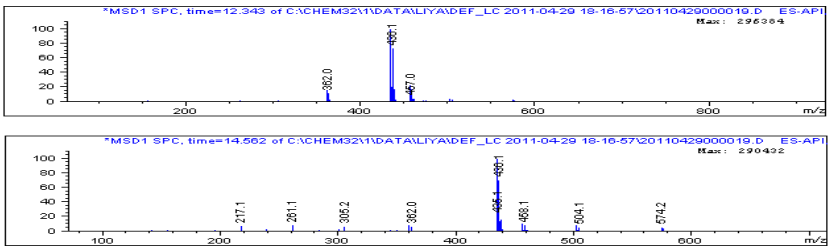
Exact Mass: 433.03

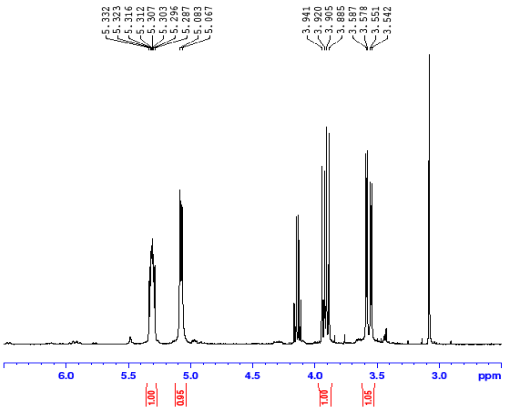
Mol. Wt.: 434.40

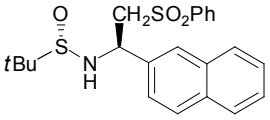
3b, rt = 12.41 min; **3b'**, rt = 14.44 min

dr = 86.49 : 2.38 = 36 : 1









$C_{22}H_{25}NO_3S_2$

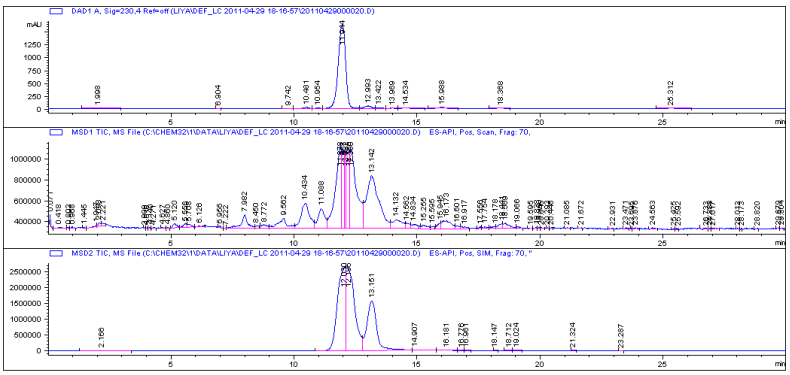
Exact Mass: 415.13

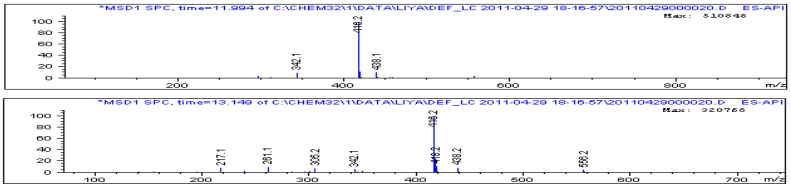
Mol. Wt.: 415.57

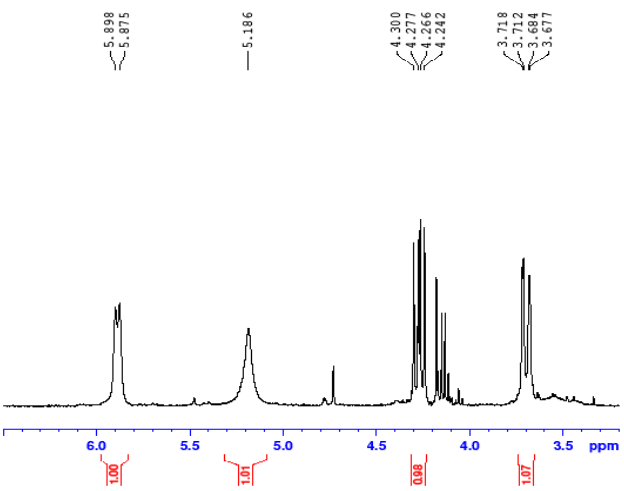
3c

3c, rt = 11.94 min; **3c'**, rt = 12.99 min

dr = 88.49 : 2.95 = 30 : 1





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SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                         144
DW                    60.800 usec
DE                     6.50 usec
TE                     296.8 K
D1                1.00000000 sec
TD0                          1

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NUC1                       1H
P1                    12.86 usec
PL1                   -4.00 dB
PLW1            20.19063568 W
SFO1            400.1324710 MHz
SI                       32768
SF              400.1300000 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00
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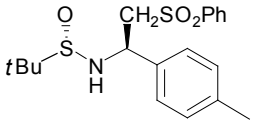


$C_{19}H_{25}NO_3S_2$

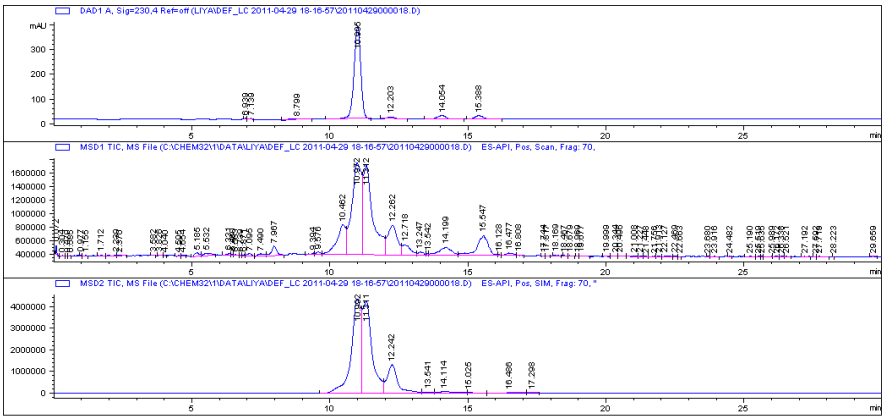
Exact Mass: 379.13

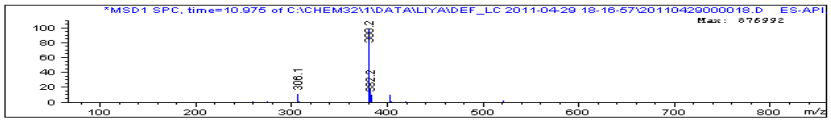
Mol. Wt.: 379.54

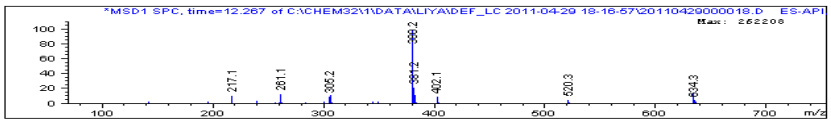
3d

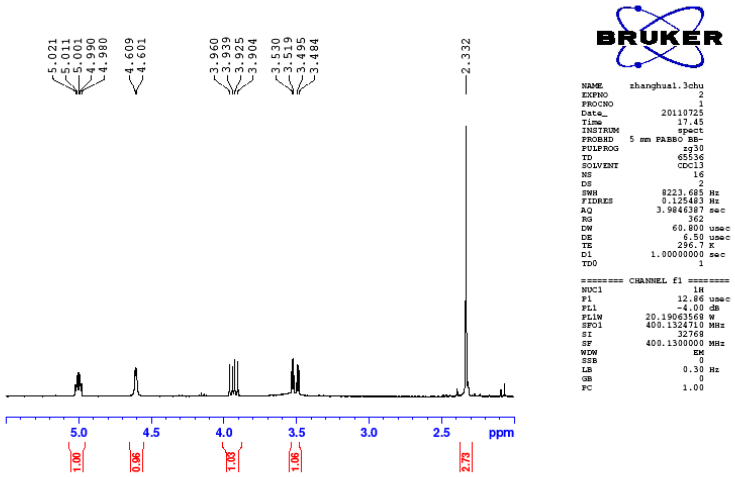
3d, rt = 10.99 min; **3d'**, rt = 12.20 min

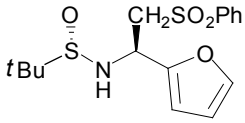
dr = 87.20 : 0.30 = 99 : 1











$C_{16}H_{21}NO_4S_2$

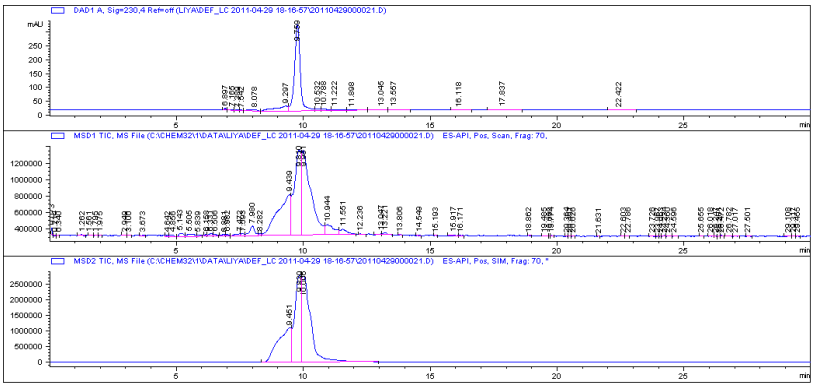
Exact Mass: 355.09

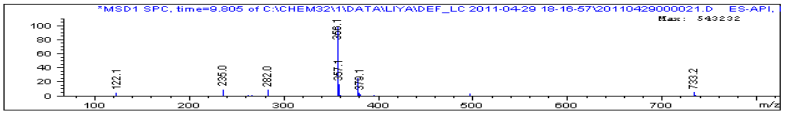
Mol. Wt.: 355.47

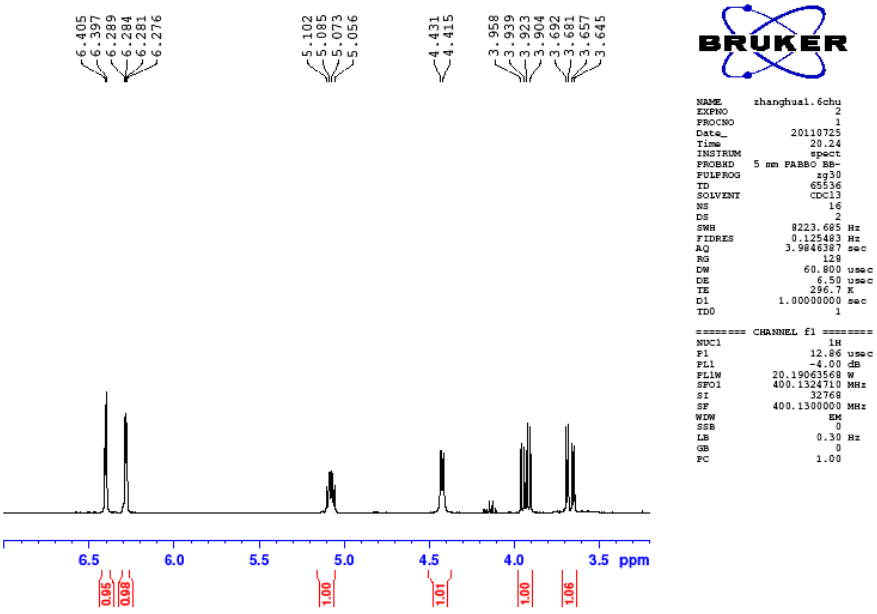
3e

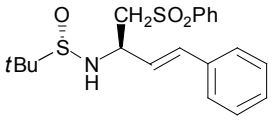
3e, rt = 9.76 min; **3e'**, rt = 9.29 min

dr = 80.20 : 0.80=99 : 1









$C_{20}H_{25}NO_3S_2$

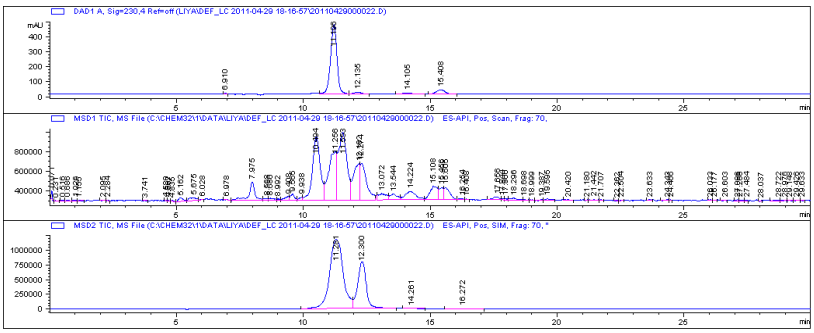
Exact Mass: 391.13

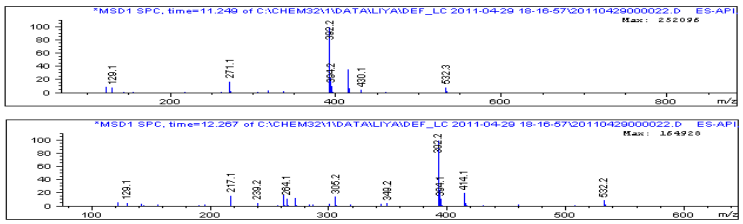
Mol. Wt.: 391.55

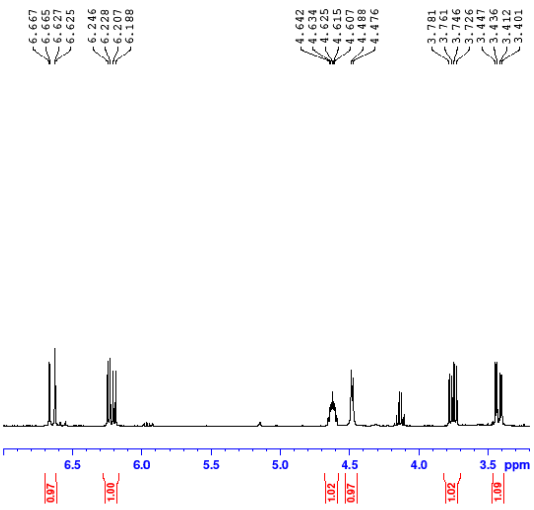
3f

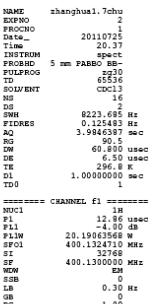
3f, rt = 11.19 min; **3f'**, rt = 12.13 min

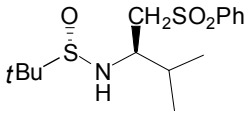
dr = 86.78 : 2.50 = 35 : 1











$C_{15}H_{25}NO_3S_2$

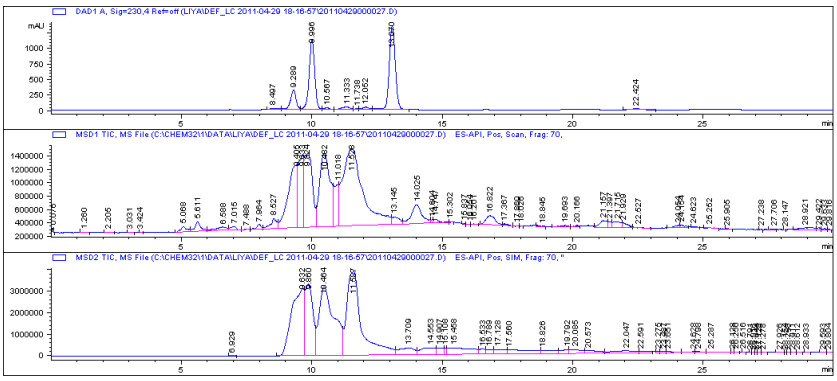
Exact Mass: 331.13

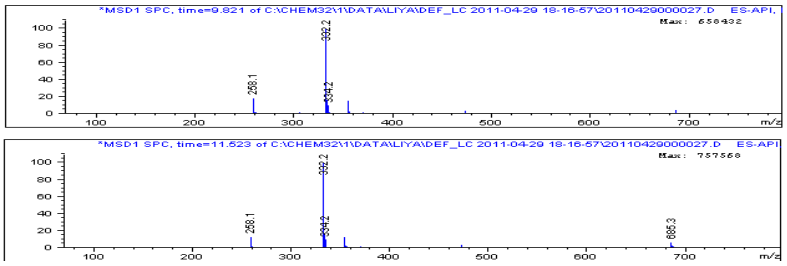
Mol. Wt.: 331.49

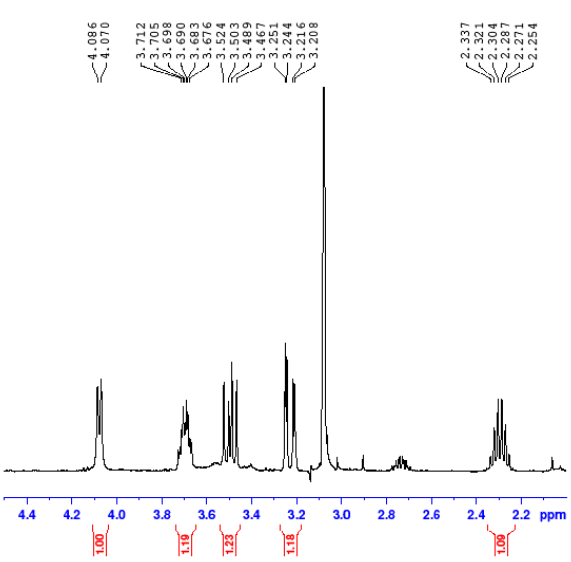
3g

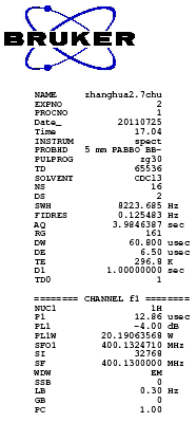
3g, rt = 9.99 min; **3g'**, rt = 11.33 min

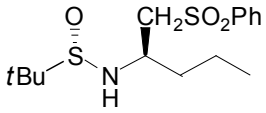
dr = 35.45 : 1.22 = 35 : 1











$C_{15}H_{25}NO_3S_2$

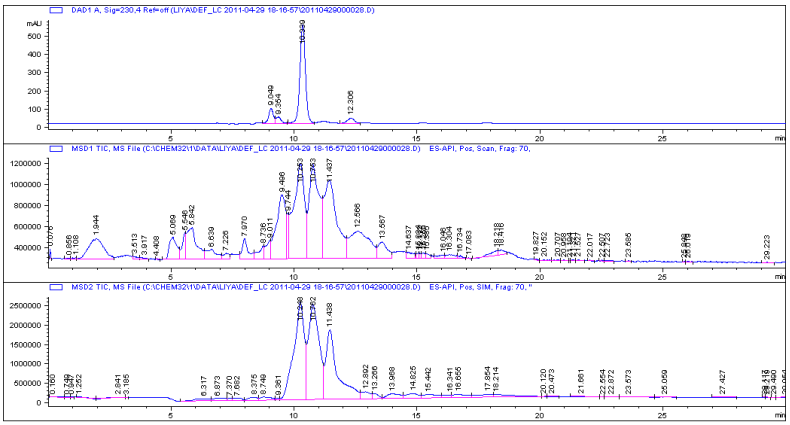
Exact Mass: 331.13

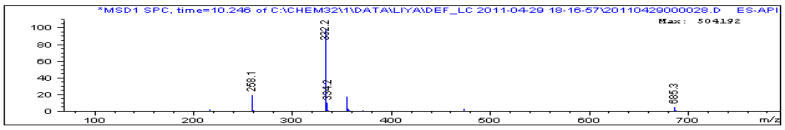
Mol. Wt.: 331.49

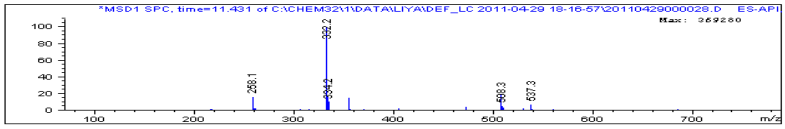
3h

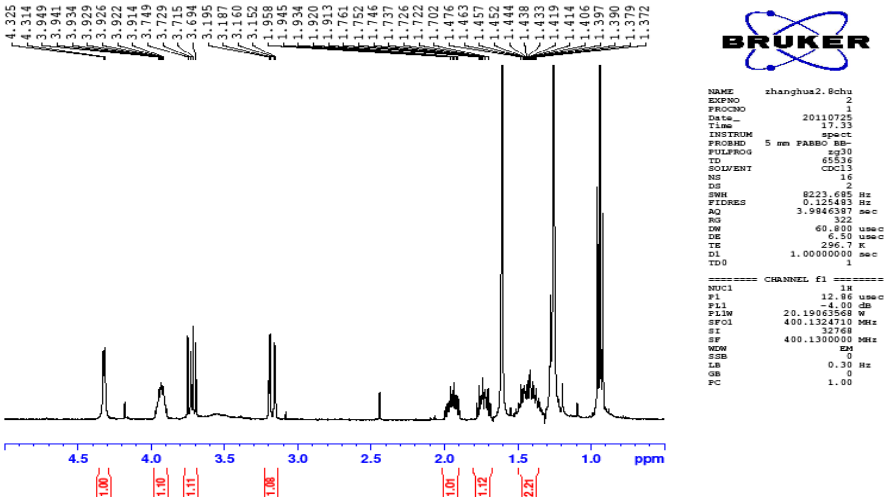
3h, rt = 10.34 min; **3h'**, rt = 11.19 min

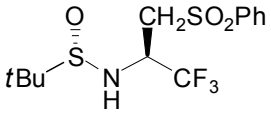
dr = 70.45 : 4.01 = 18 : 1











$C_{13}H_{18}F_3NO_3S_2$

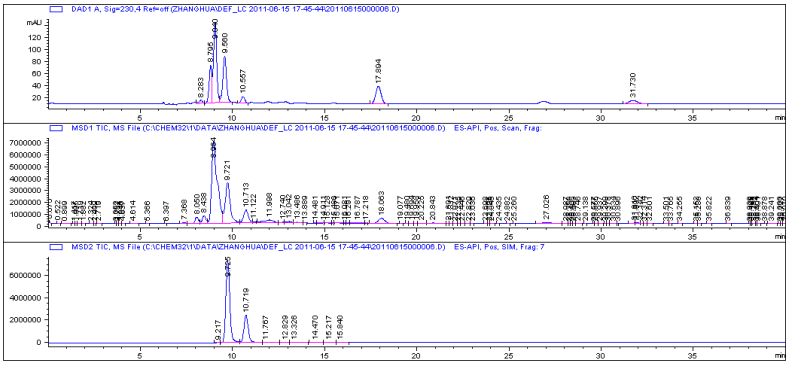
Exact Mass: 357.07

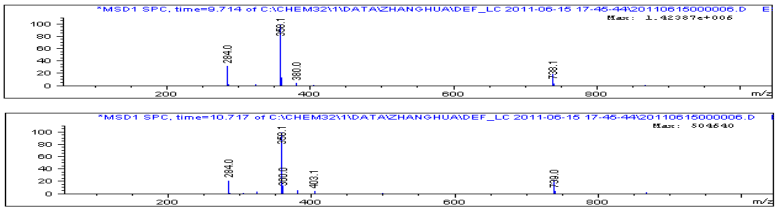
Mol. Wt.: 357.41

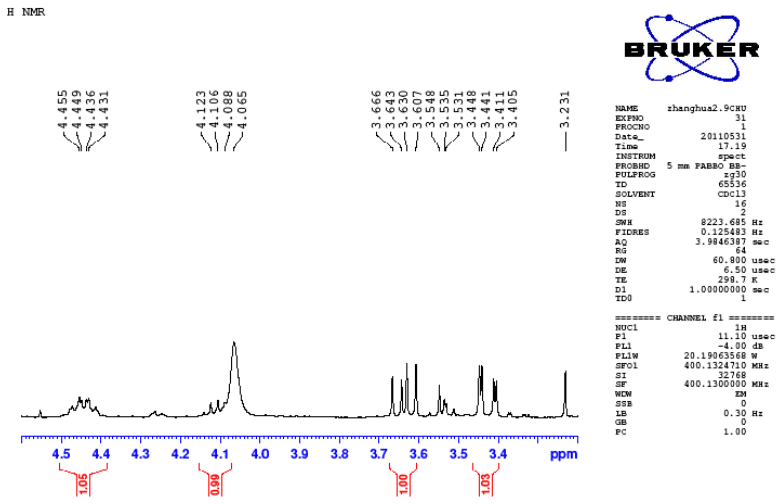
3i

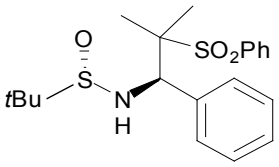
3i, rt = 9.56 min; **3i'**, rt = 10.56 min

dr = 22.18 : 3.12 = 7 : 1









$C_{20}H_{27}NO_3S_2$

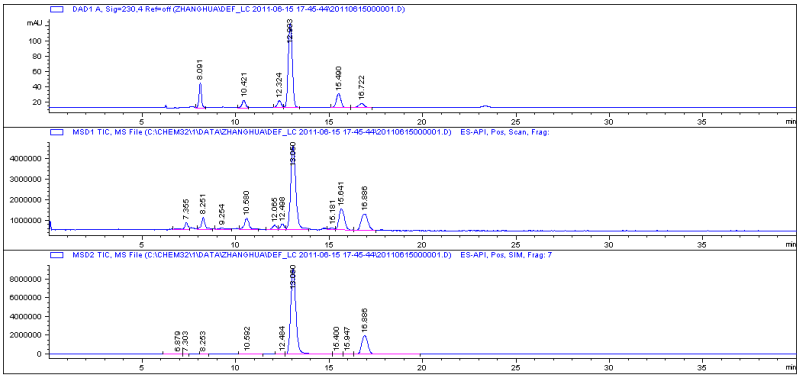
Exact Mass: 393.14

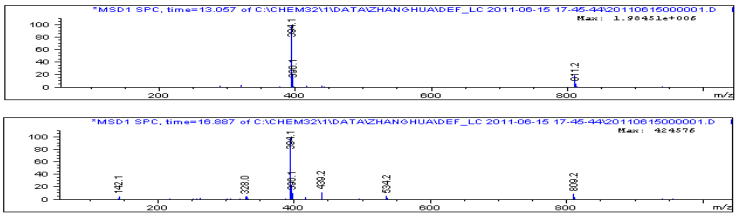
Mol. Wt.: 393.56

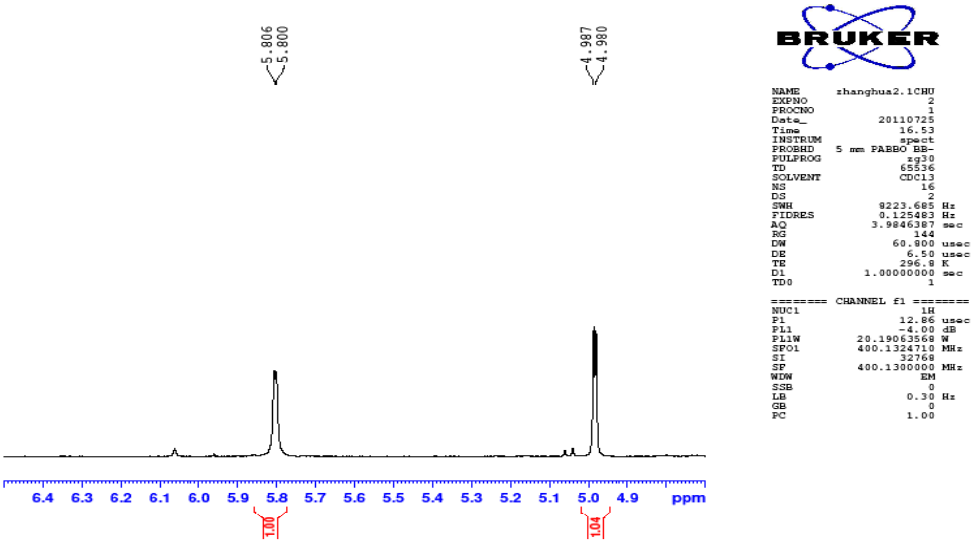
5a

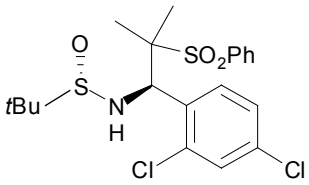
5a rt = 12.90 min; **5a'**, rt = 16.72 min

dr = 62.99 : 4.01 = 16 : 1









$C_{20}H_{25}Cl_2NO_3S_2$

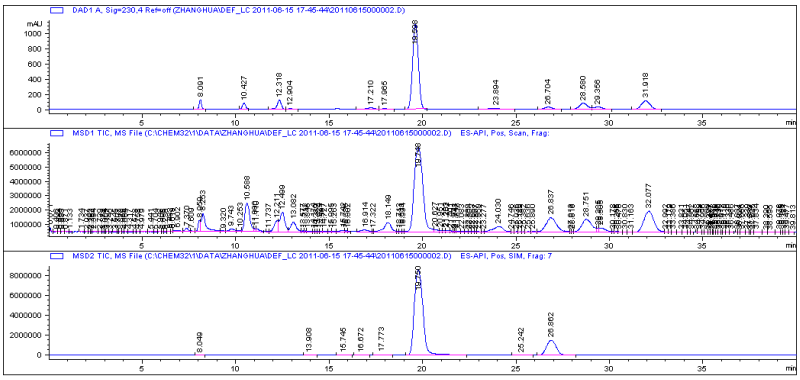
Exact Mass: 461.07

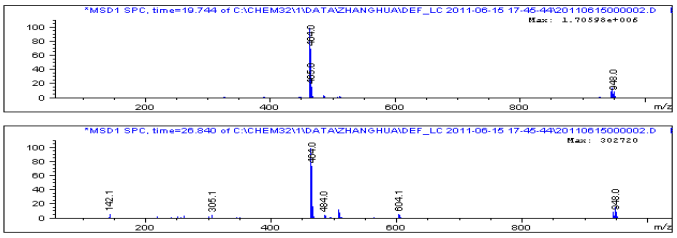
Mol. Wt.: 462.45

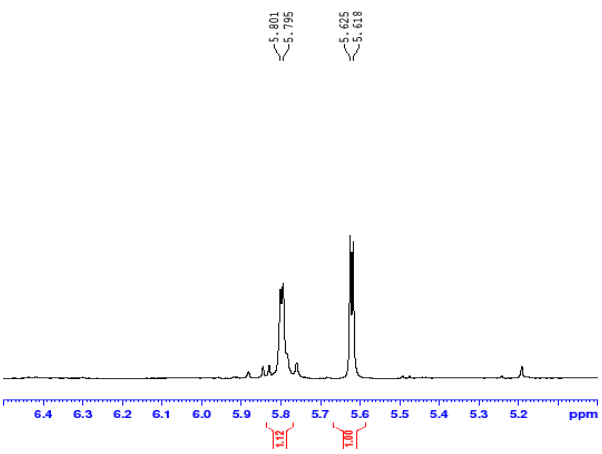
5b

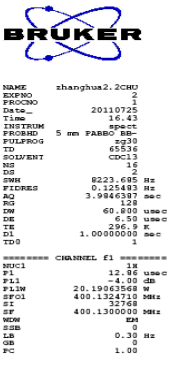
5b, rt = 19.59 min; **5b'**, = 26.70 min

dr = 62.99 : 2.27 = 27 : 1









Determination of the facial selectivity and isomer ratio for entry 3 and entry 4 (Table 2).

A mixture of diasteromers (ratio = 5 : 1 based on ^{1}H NMR), derived from the reaction of **4b** and **2a**, was subject to reductive desulfonylation reaction using Mg/MeOH. Sulfinamide **6** was the single desulfonylated compound that could be detected by ^{1}H NMR on the crude product, thus providing strong evidence that **5c** and **5c'** were produced by attack on the same face of imine **2a**. It should point out that (*E*)-(3-methylbut-1-enyl)benzene **7** was also obtained under the same reaction conditions.

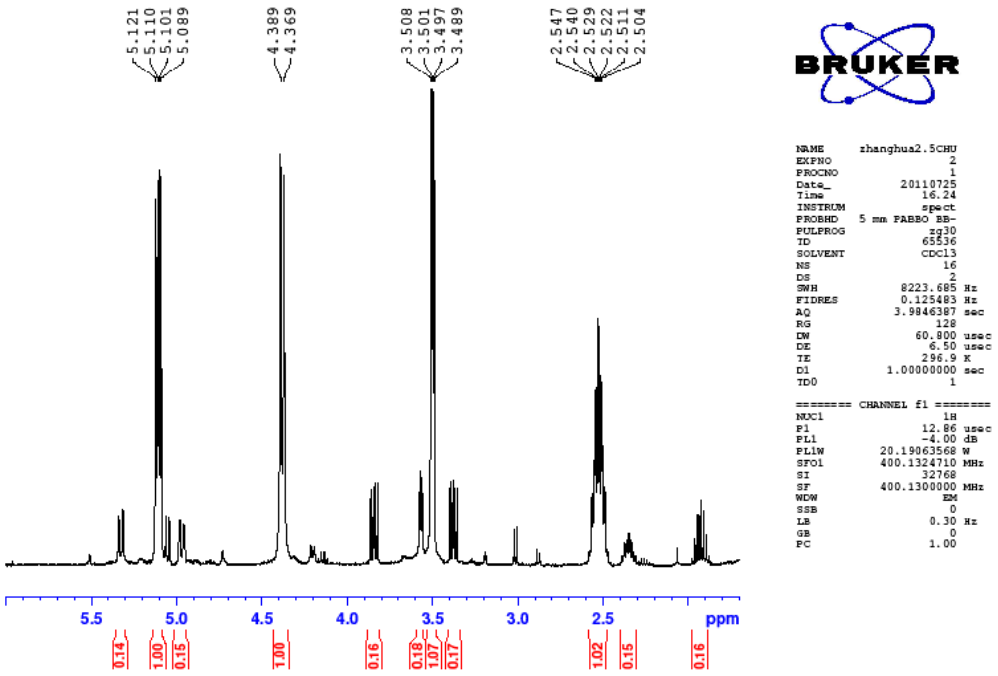


Figure 3. ^{1}H NMR spectroscopy on a mixture of diasteromers (ratio = 5 : 1)

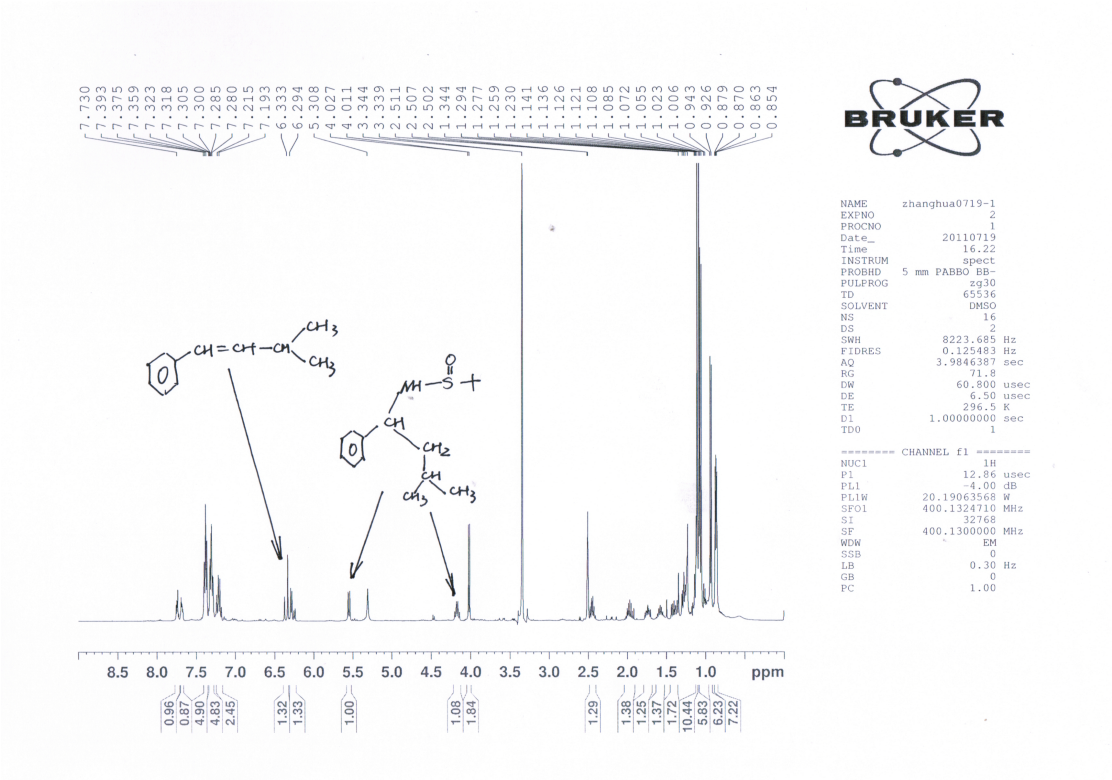
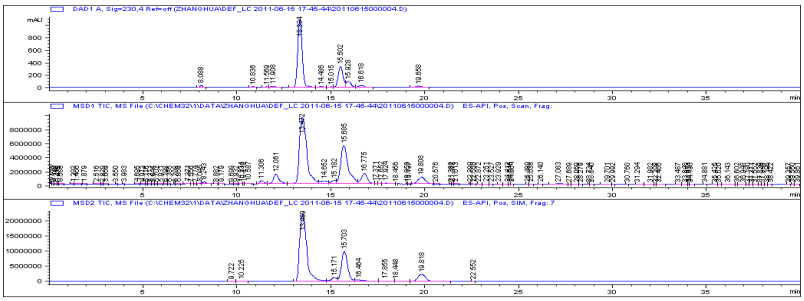


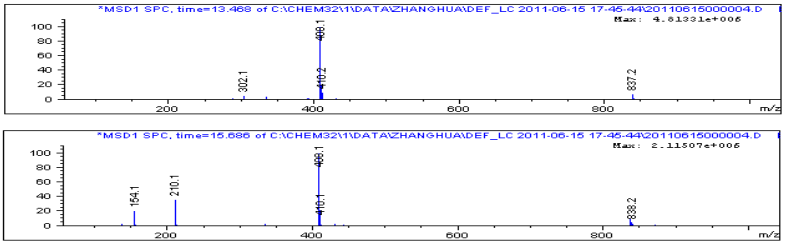
Figure 4. ^{1}H NMR spectroscopy on the crude product **6**

5c, rt = 13.33 min**; 5c' ;** rt = 15.50 min **;5c''** rt=15.01 min **;5c''',**rt=19.65 min

Facial selectivity = (64.24 + 21.72) : (0.04 + 1.43) = 60 : 1

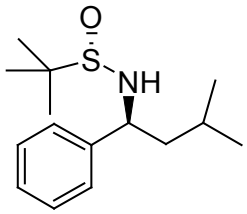
dr = 64.24 : 21.72 = 3 : 1





Experimental details:

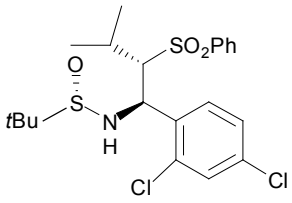
Into a 10-mL flask containing **5c** and **5c'** (183 mg, 0.45 mmol) in 5 mL anhydrous methanol at 0 °C, was added magnesium powder (3.6 mmol). The reaction mixture was stirred 1 h. Then 20 mL brine was added, followed by extracting with EtOAc. The combined organic phase was dried over $MgSO_4$, and the solvent was removed to give product **6** (48 mg, 40 %) and (E)-(3-methylbut-1-enyl)benzene **7** (29 mg, 45 %).



6

White solid, mp 65.3-66.5°C; $[\alpha]_D^{25}$ –41.53(c =0.38, $CHCl_3$); ^{1}H NMR ($CDCl_3$): δ 7.37-7.21 (m, 5H), 5.54(d, J = 8.8 Hz, 1H), 4.17 (m, 1H), 1.72 (m, 1H), 1.57 (m, 1H), 1.40 (m, 1H), 1.10 (s, 9H), 0.85 (m, 6H); ^{13}C NMR ($CDCl_3$): δ 145.2, 128.5, 127.3, 127.1, 58.7, 55.9, 47.7, 24.6, 23.1, 22.2; MALDI calcd. For $C_{15}H_{25}NOSNa$ [M + Na]$^+$: 290.1549, Found 290.1557.

yellow oil; ^{1}H NMR ($CDCl_3$): δ 7.37-7.10 (m, 5H), 6.33 (d, J = 15.9 Hz, 1H), 6.18 (dd,J = 15.9, 6.6 Hz, 1H), 2.54-2.36 (m, 1H), 1.07 (d, J = 6.6 Hz, 6H); ^{13}C NMR ($CDCl_3$): δ 138.2, 128.6, 128.3, 127.0, 126.9, 126.6, 126.1, 31.7, 22.6.



$C_{21}H_{27}Cl_2NO_3S_2$

Exact Mass: 475.08

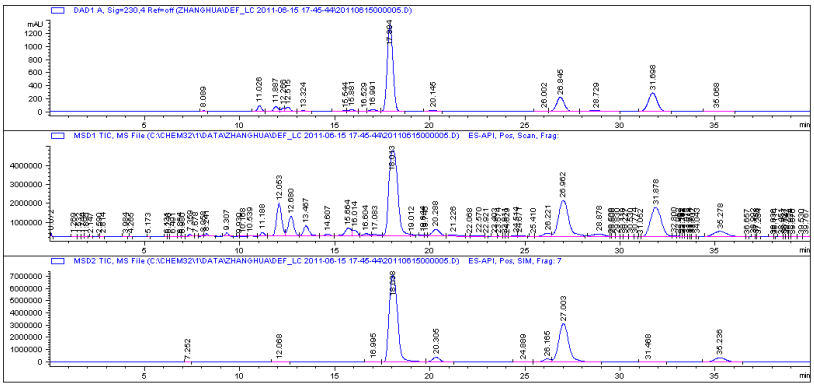
Mol. Wt.: 476.48

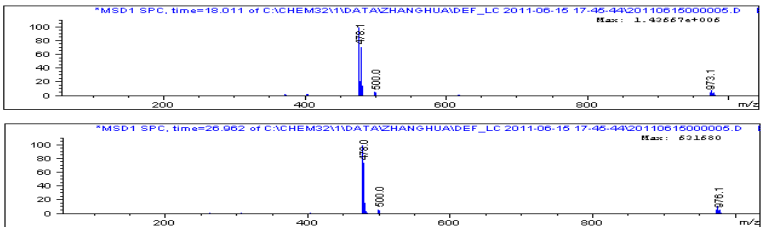
5d

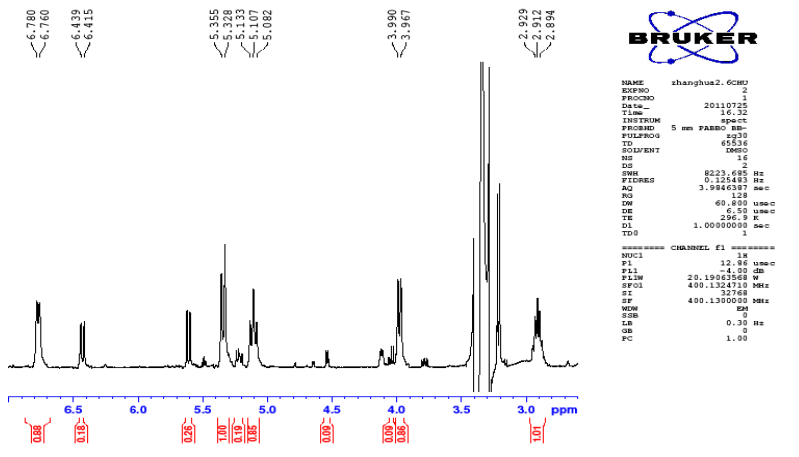
5d, rt = 17.88 min; **5d'**, rt = 26.84 min; **5d''**, rt=20.14 min; **5d'''**, rt=35.06 min

Facial selectivity = (54.16+10.85): (0.04+ 0.86)=75 : 1

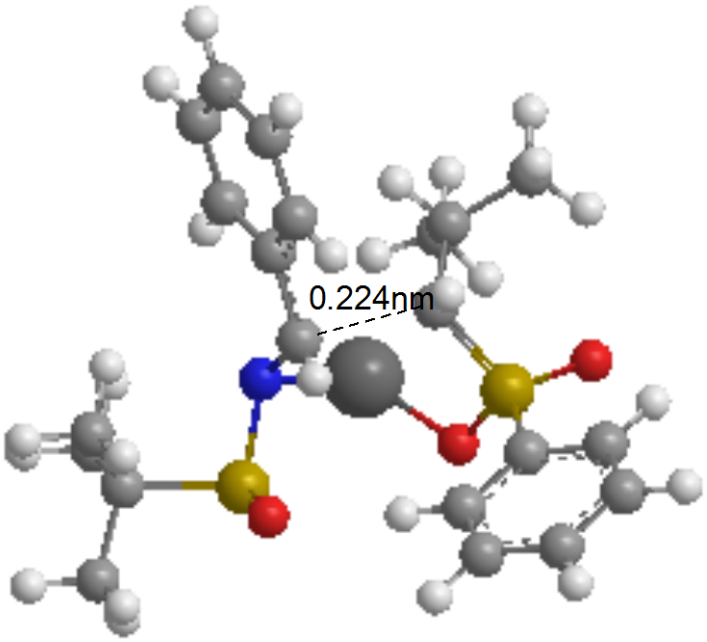
dr =54.16:10.85= 5 : 1







Computational studies for the transition state of the reaction between compound **2a** and **4b**



Geometry optimizations of molecules and transition state were performed at the standard B3LYP/6-31G(d) level. The frequency calculations at the same level provided thermodynamic and zero-point energy corrections at -80℃. Single point energies were then calculated at the B3LYP/6-311++G(2df,2p) level. The calculations were performed with the Gaussian 03 programs.[1]

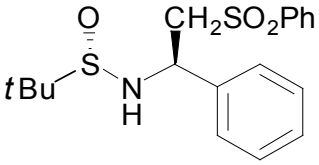
In the transition state(Figure 1c) the forming C-C bond length is 0.224 nm and the free energy of activation for the formation of the transition state ($\Delta G^{\ddagger}$) is 68.5 kJ/mol calculated at -80°C.

reference：

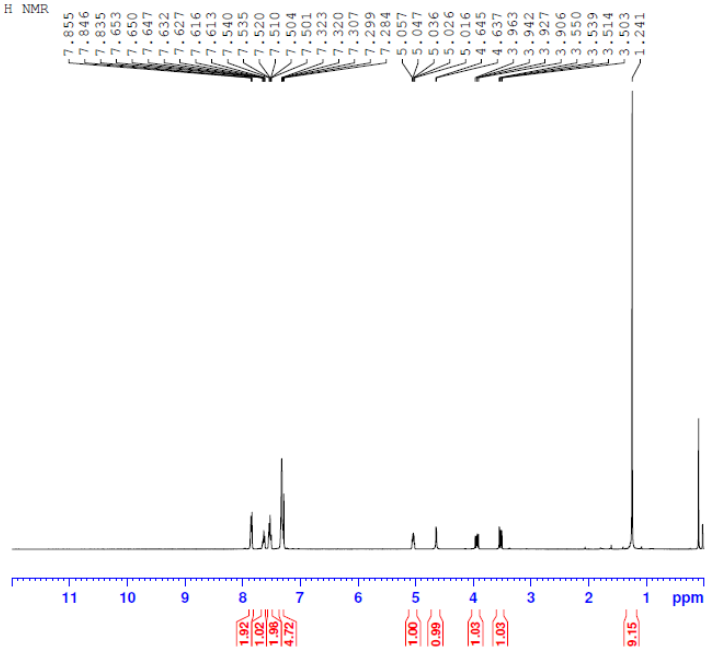
[1] M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, Jr., T. Vreven, K. N. Kudin, J. C. Burant, J. M.

Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakr-zewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y Peng, A. Nanayakkara, M. Challa-combe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C.Gonzalez, J. A. Pople, Gaussian 03, Revision B01, Gaussian, Inc., Pittsburgh, PA, 2003.

NMR spectra for all products



3a (^{1}H NMR and ^{13}C NMR)




```
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EXPNO                    1
PROCNO                   1
Date_             20110411
Time                 12.20
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    144
DW                60.800 usec
DE                 6.50 usec
TE                296.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.10 usec
PL1                -4.00 dB
PL1W         20.19063568 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

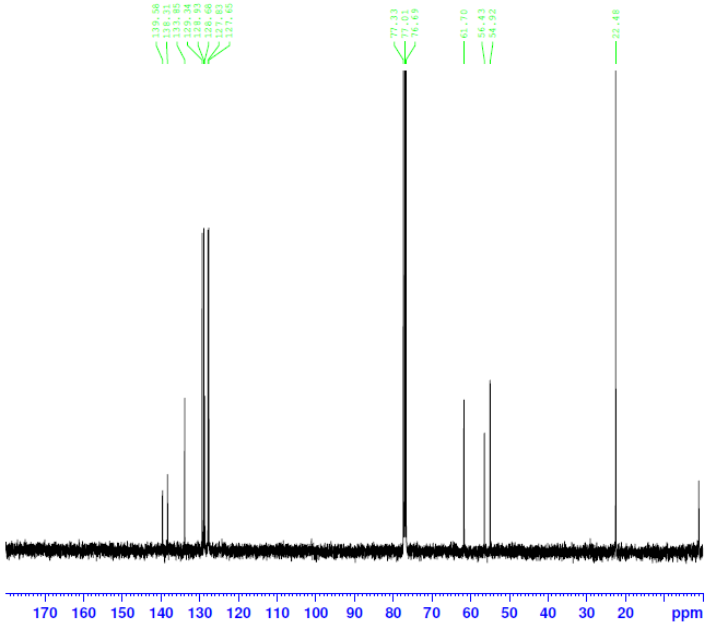


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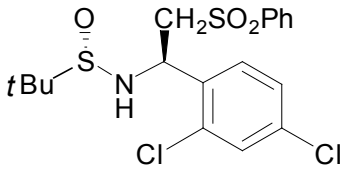
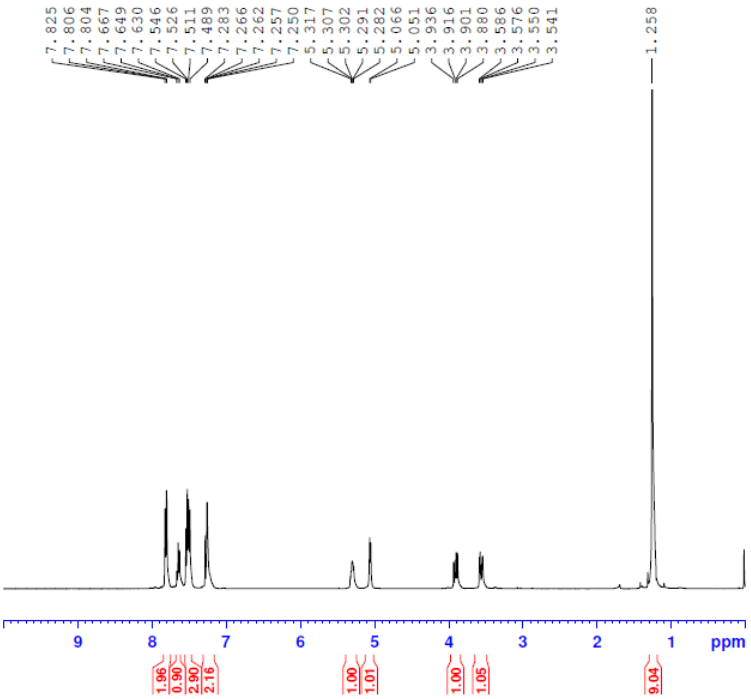
NAME          zhanghua1.1--C
EXPNO                    1
PROCNO                   1
Date_             20110412
Time                 14.44
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    696
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                   1290
DW                20.800 usec
DE                 6.50 usec
TE                300.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

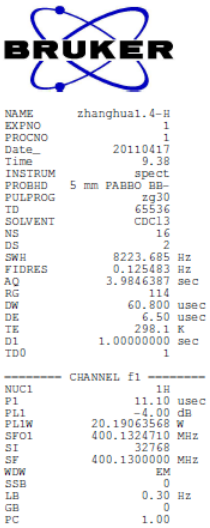
======== CHANNEL f1 ========
NUC1                  13C
P1                 9.30 usec
PL1                -3.00 dB
PL1W         64.15196228 W
SFO1         100.6228298 MHz

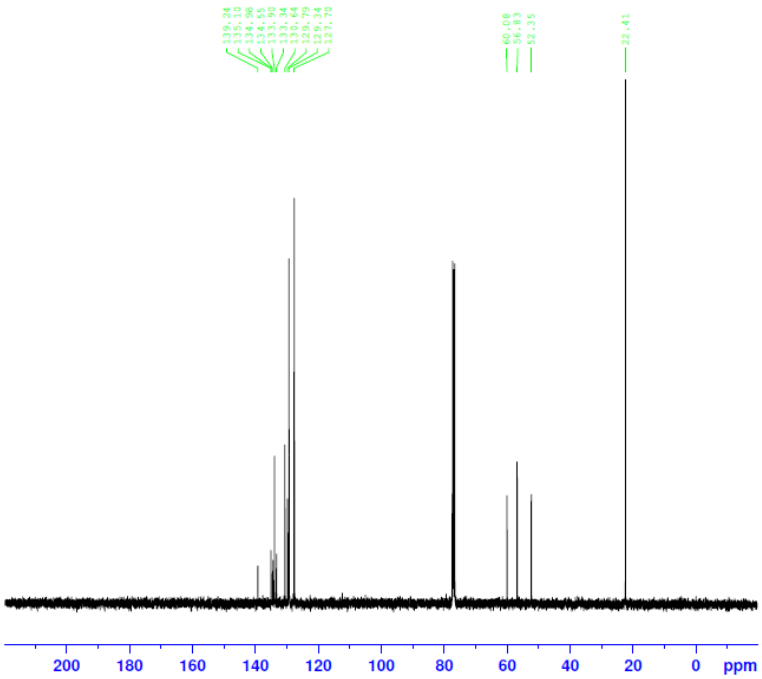
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               13.16 dB
PL13               12.00 dB
PL2W         20.19063568 W
PL12W         0.38828444 W
PL13W         0.50716585 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```

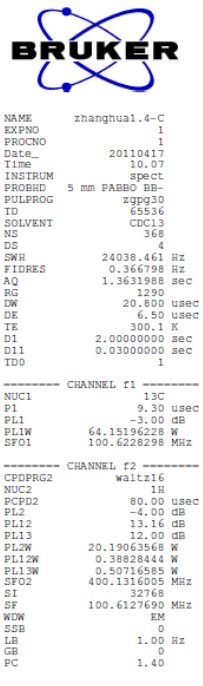


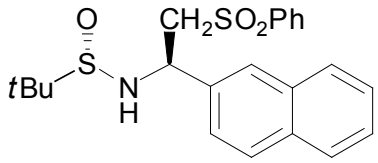
3b (^{1}H NMR and ^{13}C NMR)



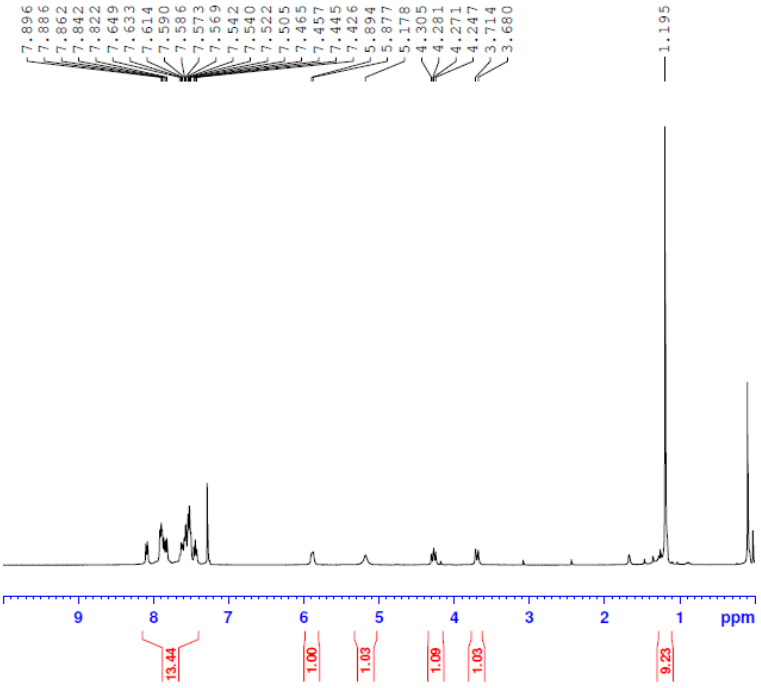








3c (^{1}H NMR and ^{13}C NMR)



BRUKER

```
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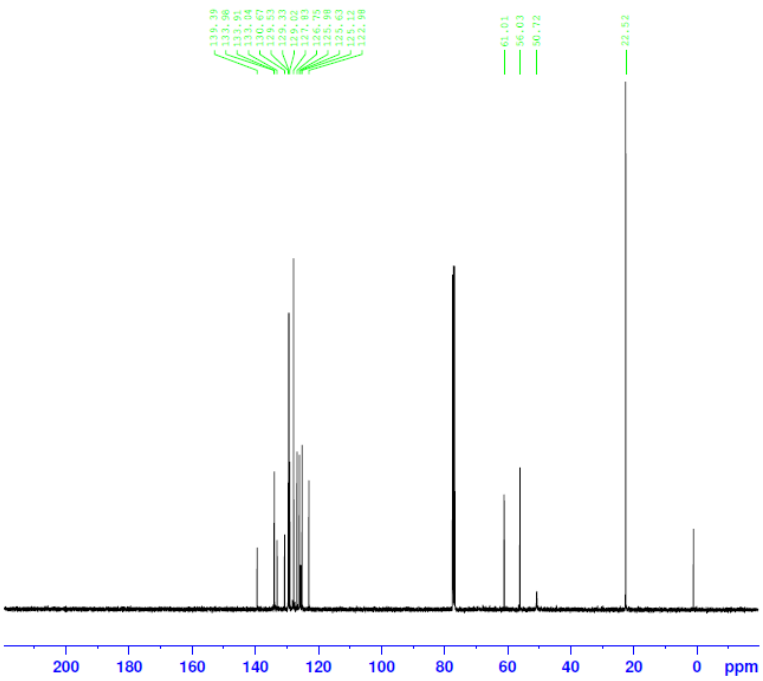
EXPNO                     1
PROCNO                    1
Date_              20110407
Time                 18.44
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      144
DW                 60.800 usec
DE                  6.50 usec
TE                  295.8 K
D1             1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                 11.10 usec
PL1                -4.00 dB
PL1W           20.19063568 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1300000 MHz
WDW                      EM
SSB                       0
LB                 0.30 Hz
GB                        0
PC                 1.00
```

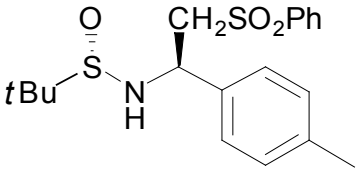


BRUKER

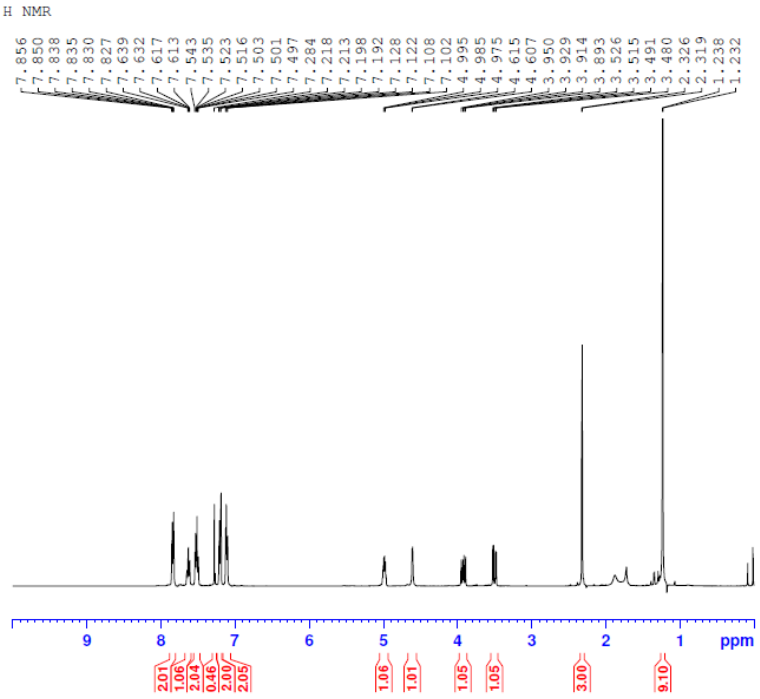
```
NAME          zhanghua1.5r-C
EXPNO                     1
PROCNO                    1
Date_              20110411
Time                 21.16
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      680
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     1030
DW                 20.800 usec
DE                  6.50 usec
TE                  299.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

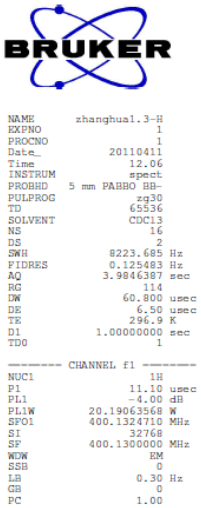
======== CHANNEL f1 ========
NUC1                   13C
P1                  9.30 usec
PL1                -3.00 dB
PL1W           64.15196228 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               13.16 dB
PL13               12.00 dB
PL2W           20.19063568 W
PL12W           0.38828444 W
PL13W           0.50716585 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127690 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                 1.40
```



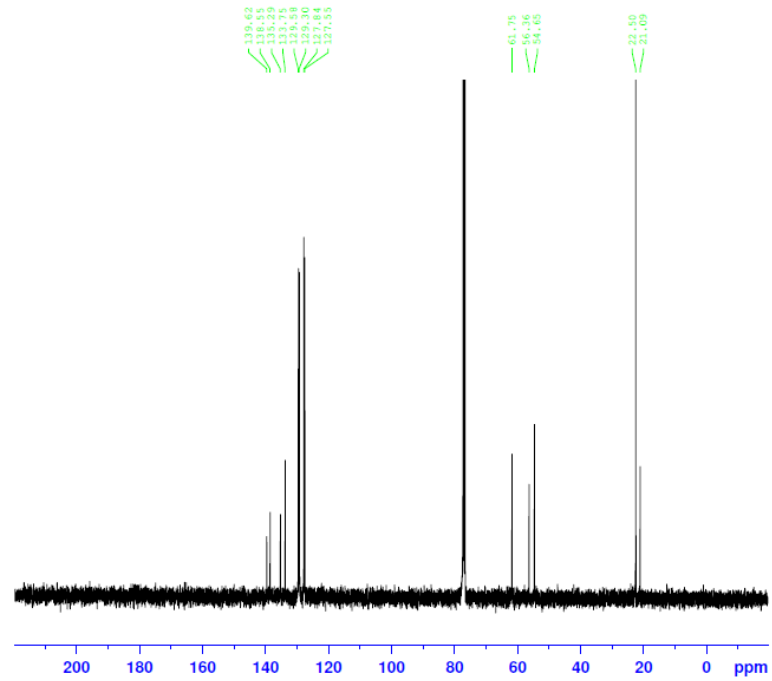
3d (^{1}H NMR and ^{13}C NMR)






```
NAME            zhanghua1.3-H
EXPNO                       1
PROCNO                      1
Date_            20110411
Time                    12.06
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                        114
DW                     60.800 usec
DE                      6.50 usec
TE                     296.9 K
D1                   1.00000000 sec
TD0                         1

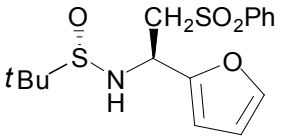
======== CHANNEL f1 ========
NUC1                       1H
P1                      11.10 usec
PL1                     -4.00 dB
PL1W                20.19063568 W
SFO1                400.1324710 MHz
SI                        32768
SF                  400.1300000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



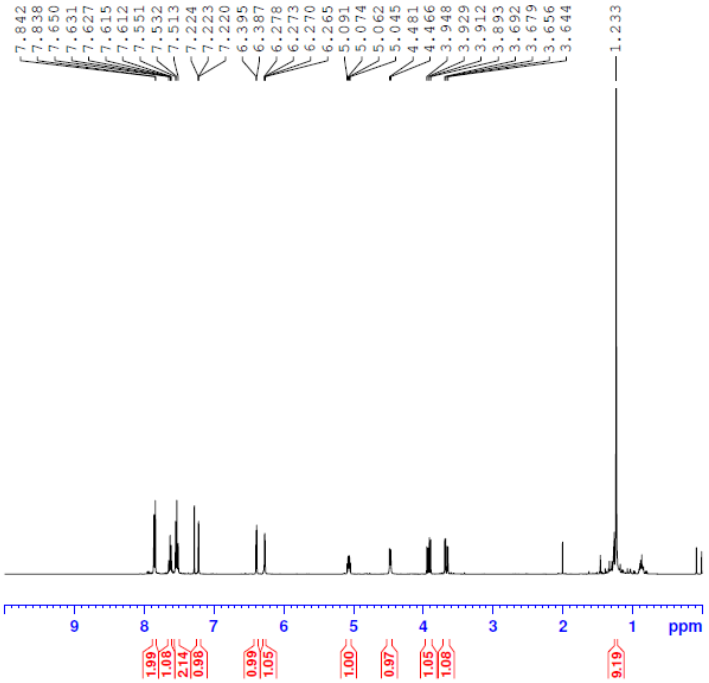

```
NAME            zhanghua1.3--C
EXPNO                       1
PROCNO                      1
Date_            20110412
Time                    15.14
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                     65536
SOLVENT                 CDCl3
NS                        320
DS                          4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631988 sec
RG                        724
DW                     20.800 usec
DE                      6.50 usec
TE                     299.9 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                       9.30 usec
PL1                     -3.00 dB
PL1W                64.15196228 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -4.00 dB
PL12                    13.16 dB
PL13                    12.00 dB
PL2W                20.19063568 W
PL12W                0.38828444 W
PL13W                0.50716585 W
SFO2                400.1316005 MHz
SI                        32768
SF                  100.6127690 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

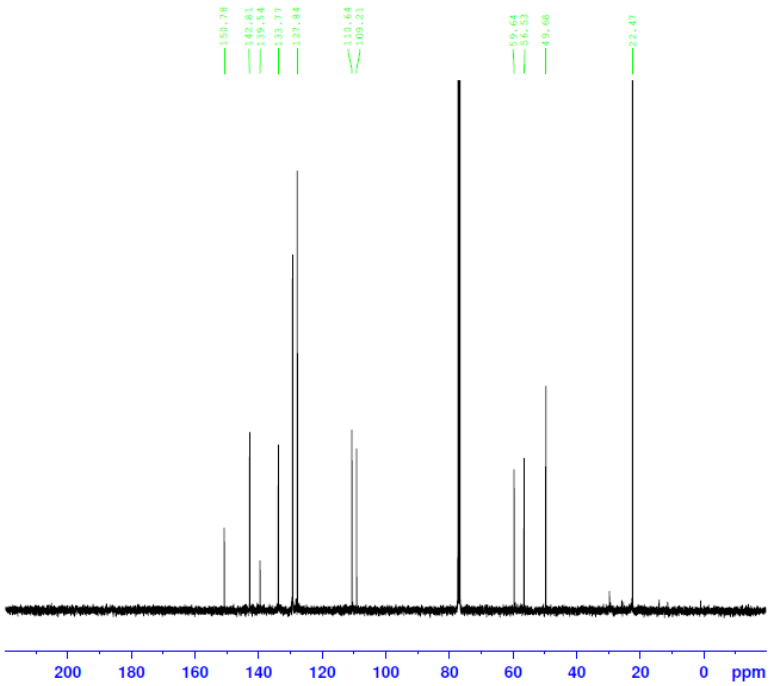


3e (^{1}H NMR and ^{13}C NMR)




```
NAME          zhanghua1.6--H
EXPNO                      1
PROCNO                     1
Date_               20110417
Time                   11.01
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       80.6
DW                    60.800 usec
DE                     6.50 usec
TE                   298.6 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.10 usec
PL1                    -4.00 dB
PL1W            20.19063568 W
SFO1            400.1324710 MHz
SI                      32768
SF              400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




```
NAME          zhanghua1.6-H
EXPNO                      1
PROCNO                     1
Date_               20110417
Time                   10.53
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       296
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                       812
DW                    20.800 usec
DE                     6.50 usec
TE                   300.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.30 usec
PL1                    -3.00 dB
PL1W            64.15196228 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                    -4.00 dB
PL12                   13.16 dB
PL13                   12.00 dB
PL2W            20.19063568 W
PL12W            0.38828444 W
PL13W            0.50716585 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127690 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
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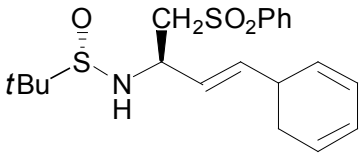
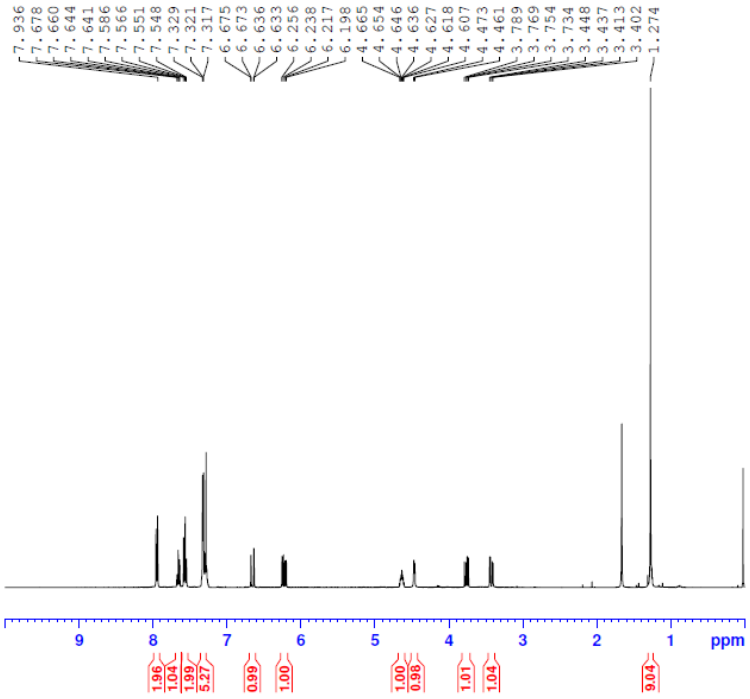
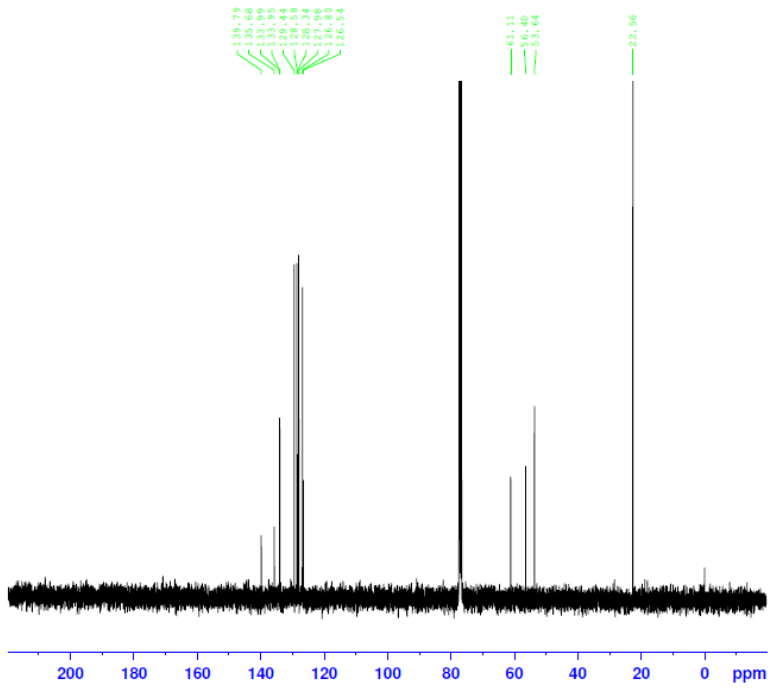


3f (^{1}H NMR and ^{13}C NMR)



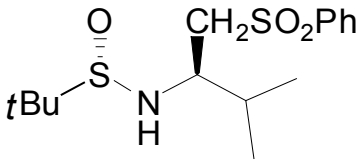
BRUKER

```
NAME          zhanghua1.7-H
EXPNO                     1
PROCNO                    1
Date_              20110407
Time                  19.43
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     181
DW                  60.800 usec
DE                   6.50 usec
TE                  295.8 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.10 usec
PL1                  -4.00 dB
PL1W          20.19063568 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

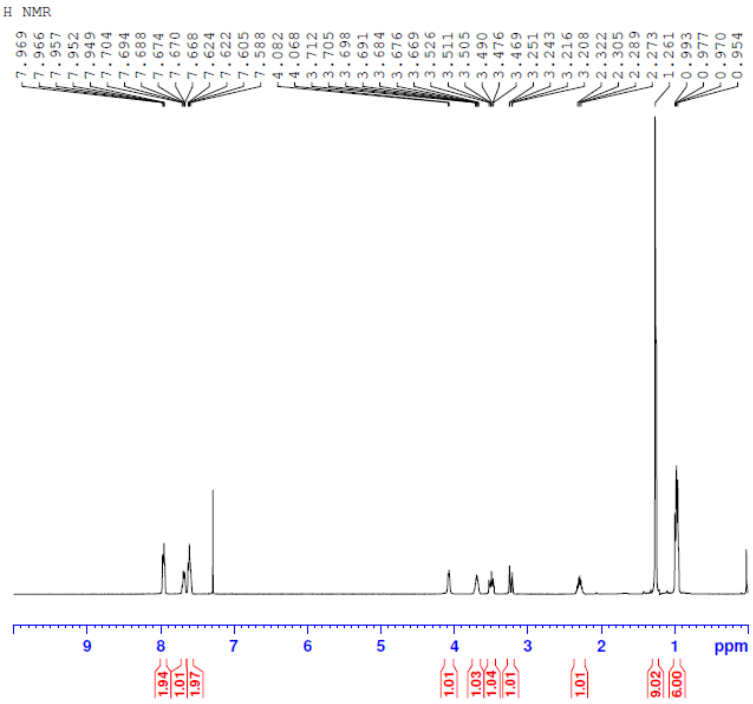


BRUKER

```
NAME          zhanghua1.7-C
EXPNO                     1
PROCNO                    1
Date_              20110411
Time                  19.07
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     472
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                    1290
DW                  20.800 usec
DE                   6.50 usec
TE                  299.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -3.00 dB
PL1W          64.15196228 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 13.16 dB
PL13                 12.00 dB
PL2W          20.19063568 W
PL12W          0.38828444 W
PL13W          0.50716585 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127690 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

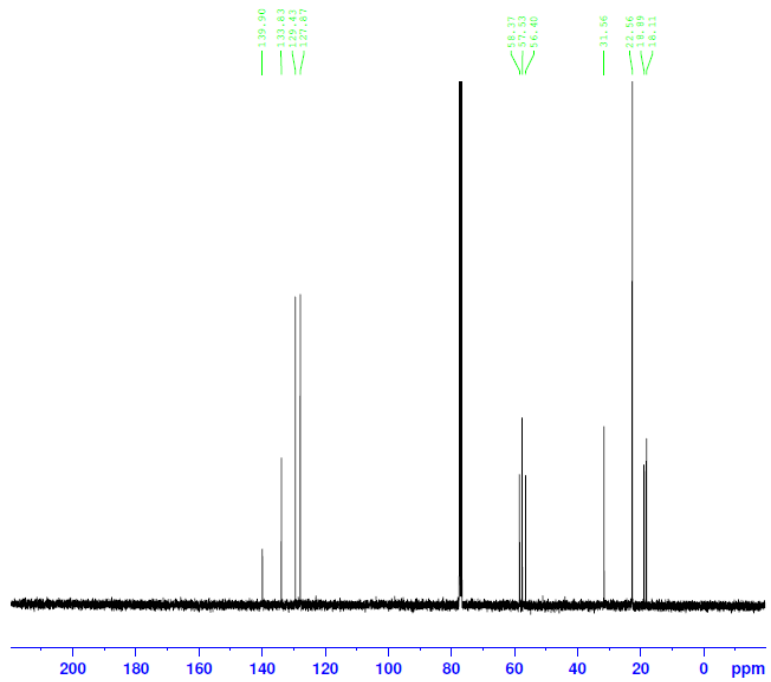


3g (^{1}H NMR and ^{13}C NMR)



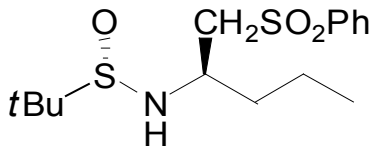
BRUKER

```
NAME          zhanghua2.7-H
EXPNO                     1
PROCNO                    1
Date_              20110411
Time                  13.10
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      144
DW                   60.800 usec
DE                    6.50 usec
TE                   297.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.10 usec
PL1                    -4.00 dB
PL1W            20.19063568 W
SFO1           400.1324710 MHz
SI                     32768
SF             400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

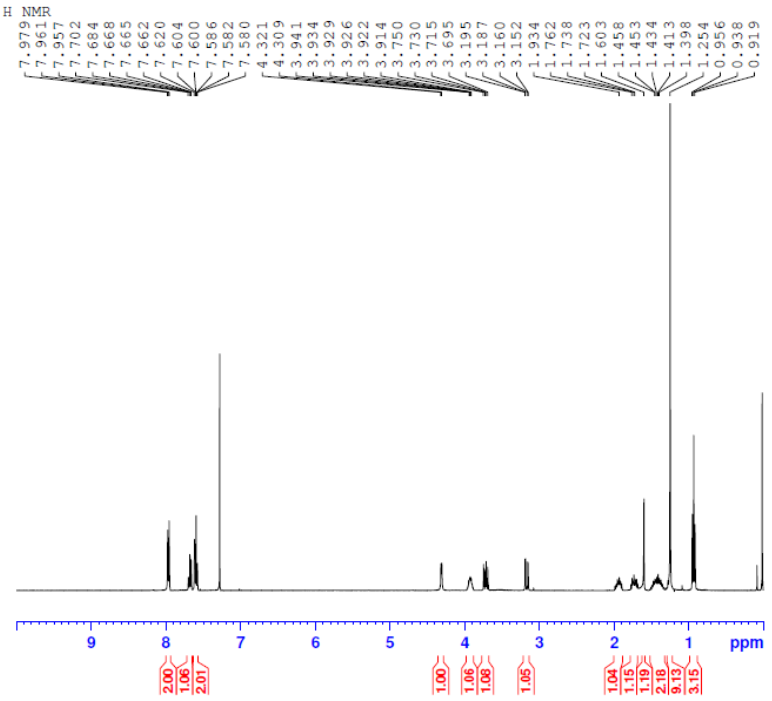


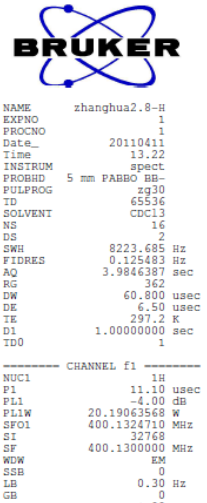
BRUKER

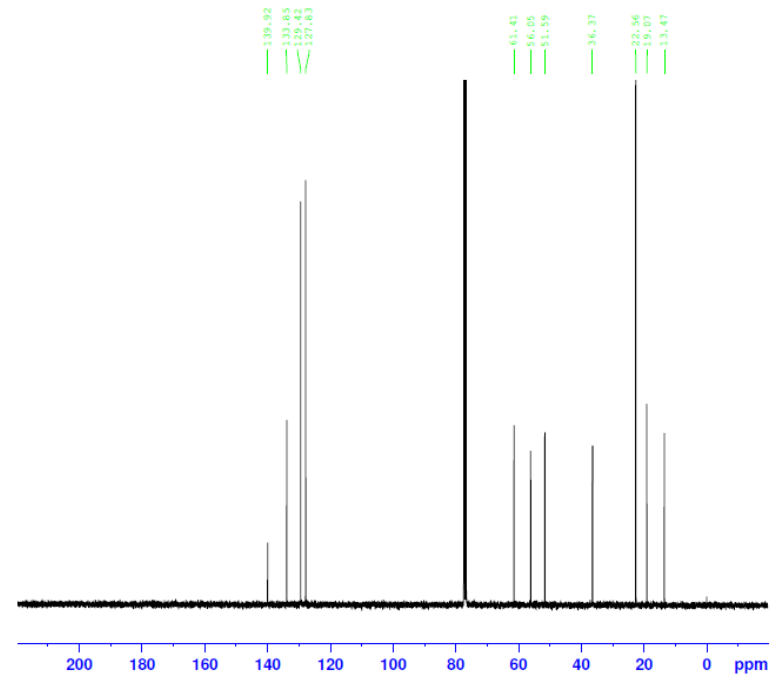
```
NAME          zhanghua2.7-C
EXPNO                     1
PROCNO                    1
Date_              20110411
Time                  20.51
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     1030
DW                   20.800 usec
DE                    6.50 usec
TE                   299.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      13C
P1                      9.30 usec
PL1                    -3.00 dB
PL1W            64.15196228 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                    -4.00 dB
PL12                   13.16 dB
PL13                   12.00 dB
PL2W            20.19063568 W
PL12W            0.38828444 W
PL13W            0.50716585 W
SFO2           400.1316005 MHz
SI                     32768
SF             100.6127690 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

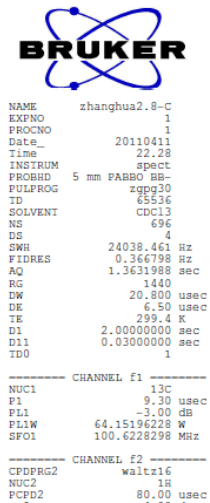


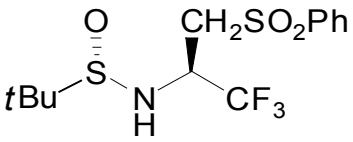
3h (^{1}H NMR and ^{13}C NMR)



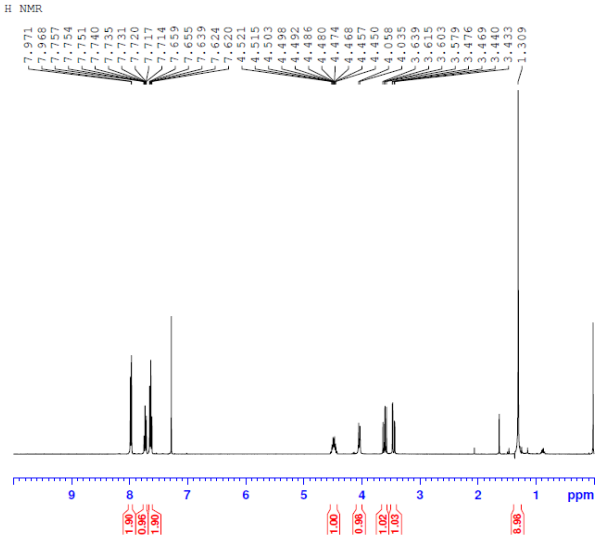


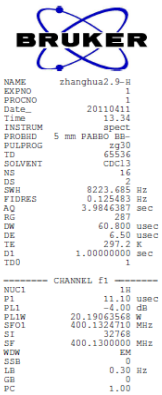


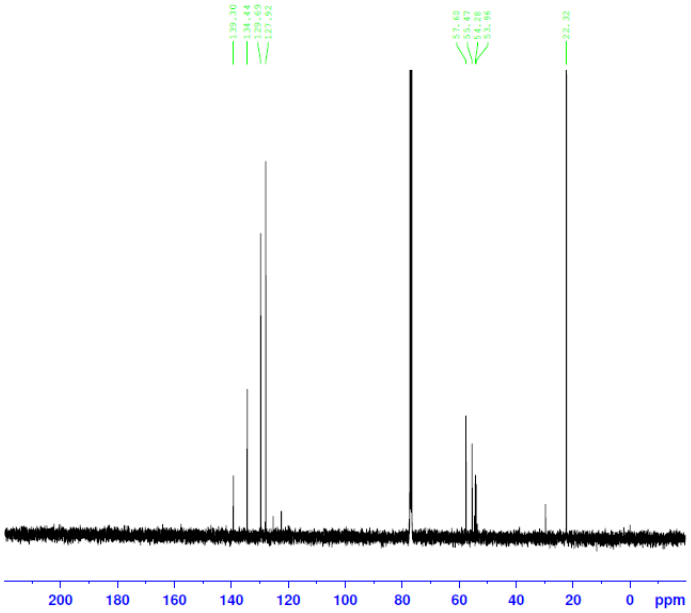




3i (^{1}H NMR ,^{13}C NMR and ^{19}F NMR)

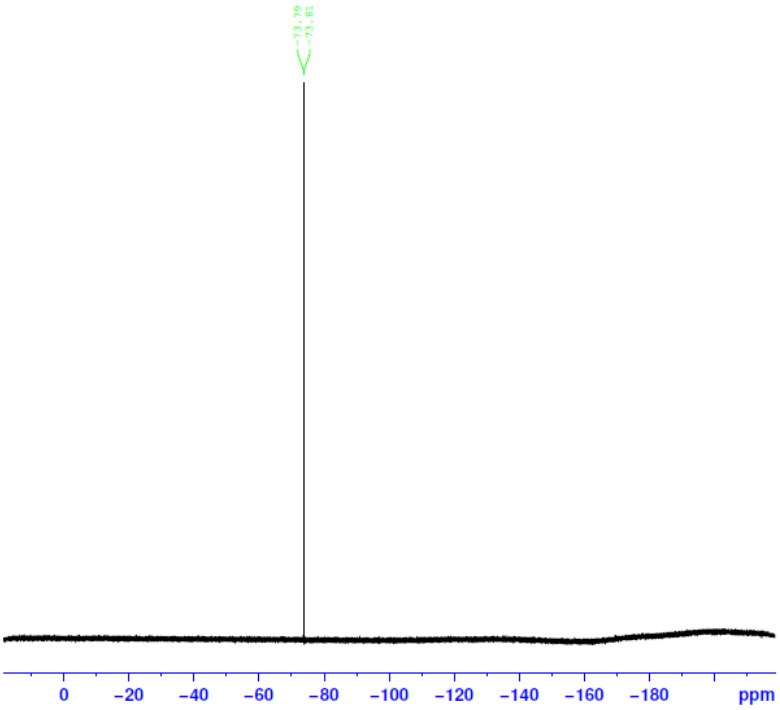






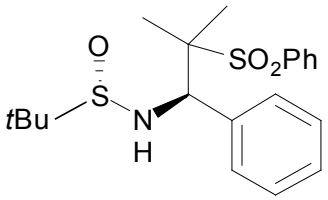
BRUKER

```
NAME            zhanghua2.9--C
EXPNO                        1
PROCNO                       1
Date_             20110412
Time                    16.07
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         824
DS                           4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                         575
DW                   20.800 usec
DE                    6.50 usec
TE                    299.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.30 usec
PL1                      -3.00 dB
PL1W             64.15196228 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                      -4.00 dB
PL12                     13.16 dB
PL13                     12.00 dB
PL2W             20.19063568 W
PL12W             0.38828444 W
PL13W             0.50716585 W
SFO2            400.1316005 MHz
SI                       32768
SF              100.6127690 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

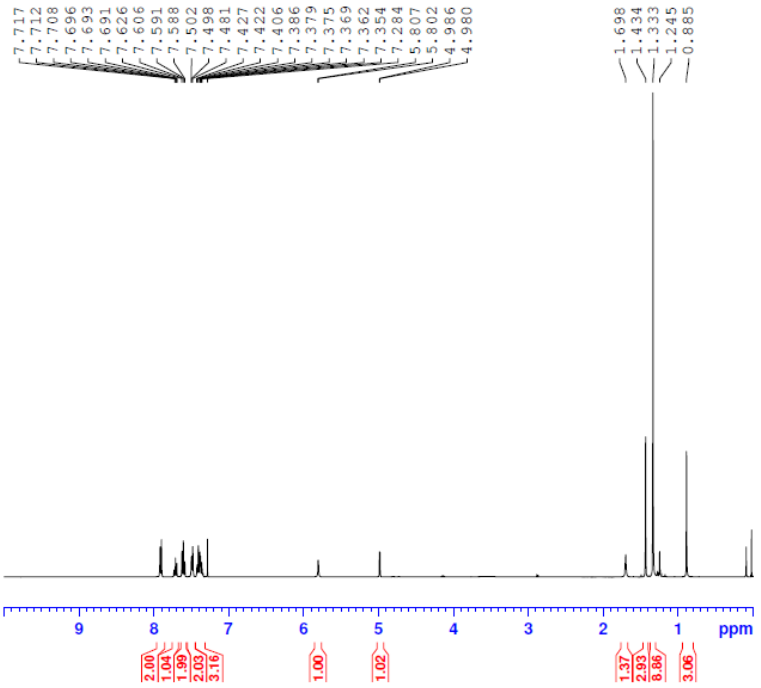


BRUKER

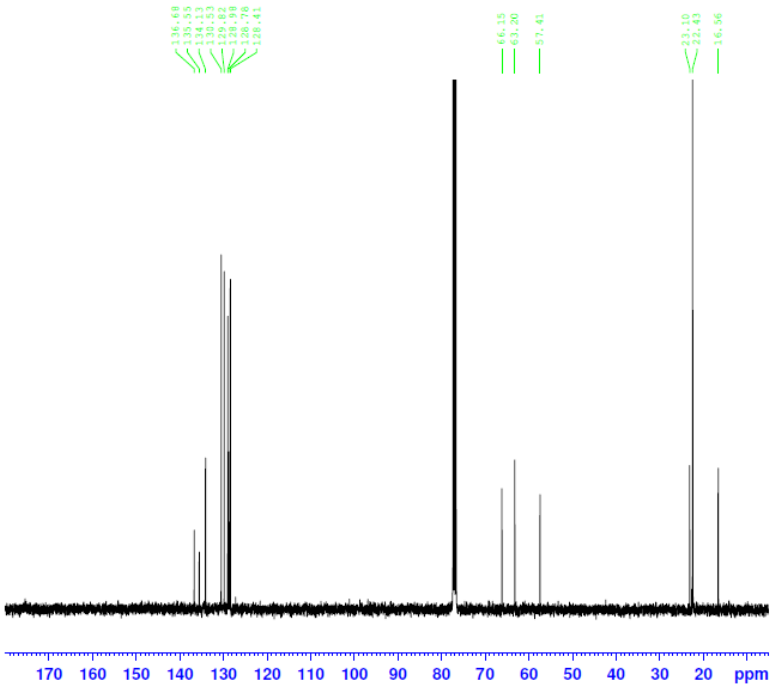
```
NAME          zhanghua0315f
EXPNO                     1
PROCNO                    1
Date_          20110315
Time                 14.17
INSTRUM              spect
PROBHD  5 mm PABBO BB-
PULPROG           zgflqn
TD                131072
SOLVENT              DMSO
NS                     16
DS                      4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ              0.7340532 sec
RG                    912
DW                5.600 usec
DE                6.50 usec
TE                294.8 K
D1             1.00000000 sec
TD0                     1

====== CHANNEL f1 ======
NUC1                   19F
P1                   14.10 usec
PL1                  -3.00 dB
PL1W          14.33395481 W
SFO1         376.4607164 MHz
SI                   65536
SF           376.4983660 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



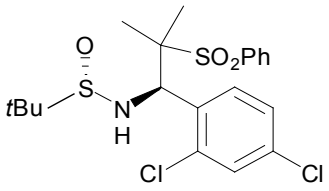
5a (^{1}H NMR and ^{13}C NMR)



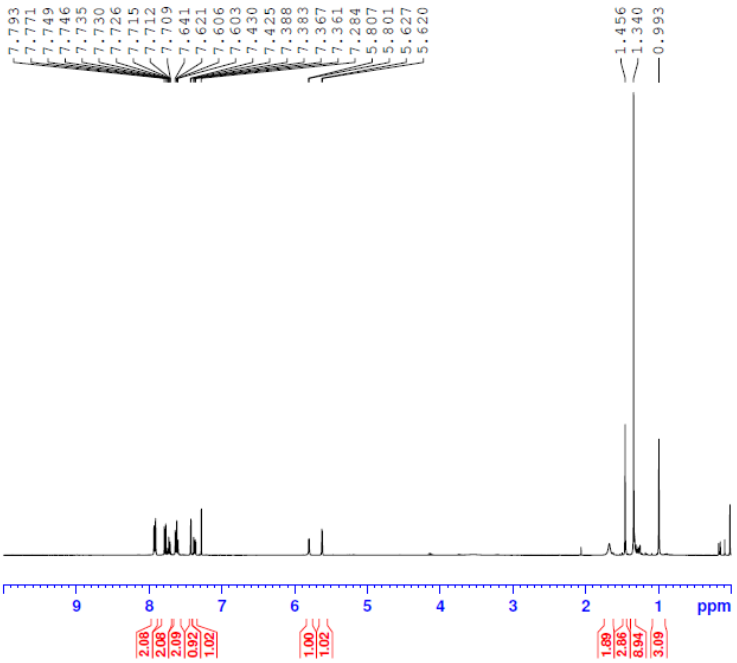

```
NAME            zhanghua110615-2.1
EXPNO                            2
PROCNO                           1
Date_                     20110615
Time                         17.45
INSTRUM                      spect
PROBHD       5 mm PABBO BB-
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                            144
DW                         60.800 usec
DE                          6.50 usec
TE                         296.3 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                         12.86 usec
PL1                        -4.00 dB
PL1W                20.19063568 W
SFO1                400.1324710 MHz
SI                          32768
SF                  400.1300000 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



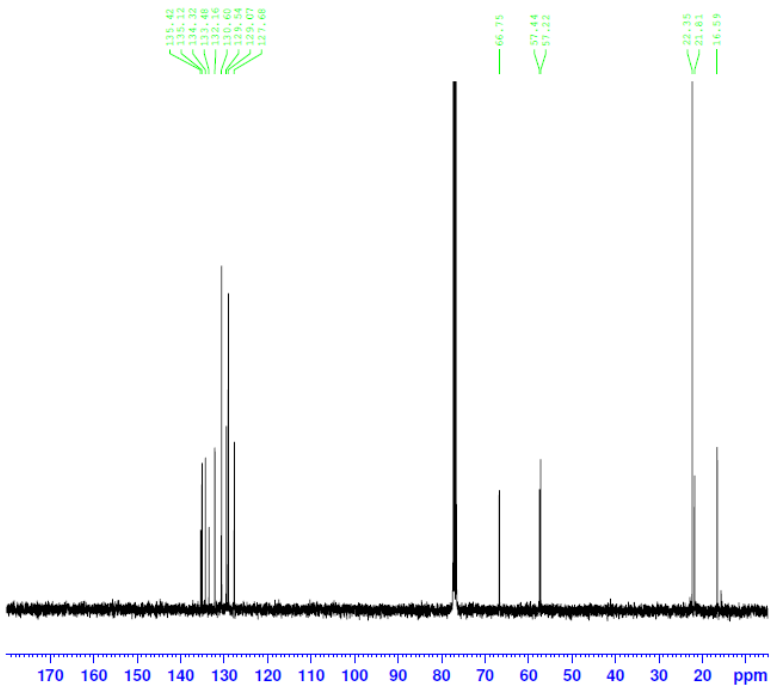

```
NAME            zhanghua110615-2.1-C
EXPNO                            2
PROCNO                           1
Date_                     20110616
Time                         18.32
INSTRUM                      spect
PROBHD       5 mm PABBO BB-
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                           1024
DS                              4
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                           1290
DW                         20.800 usec
DE                          6.50 usec
TE                         299.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                         10.10 usec
PL1                        -3.00 dB
PL1W                64.15196228 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                      80.00 usec
PL2                        -4.00 dB
PL12                       11.88 dB
PL13                       12.00 dB
PL2W                20.19063568 W
PL12W                0.52137470 W
PL13W                0.50716585 W
SFO2                400.1316005 MHz
SI                          32768
SF                  100.6127690 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```



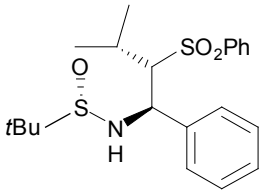
5b (^{1}H NMR and ^{13}C NMR)



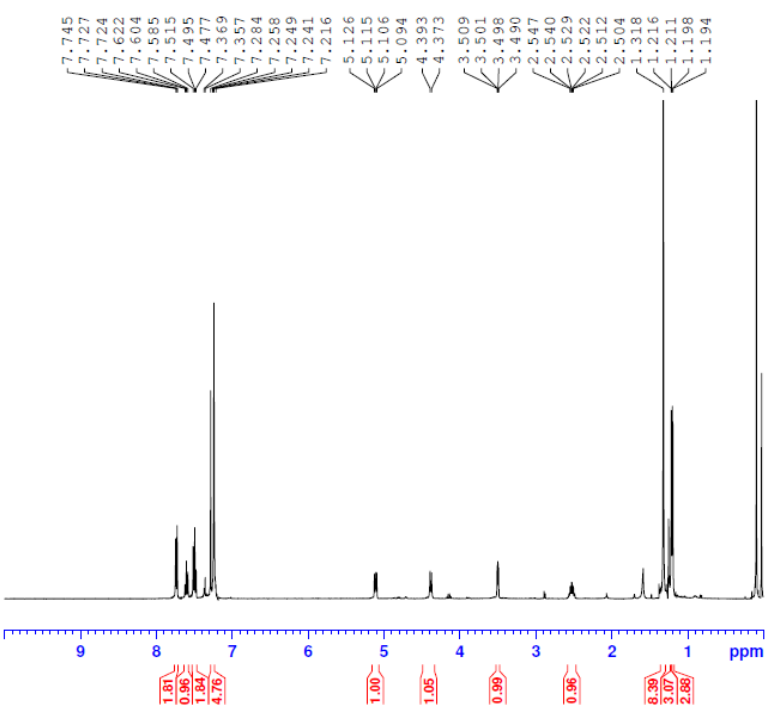

```
NAME          zhanghua110615-2.2
EXPNO                          2
PROCNO                         1
Date_                   20110615
Time                       17.54
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                           161
DW                        60.800 usec
DE                         6.50 usec
TE                        296.2 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                        12.86 usec
PL1                       -4.00 dB
PL1W                20.19063568 W
SFO1                400.1324710 MHz
SI                         32768
SF                  400.1300000 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

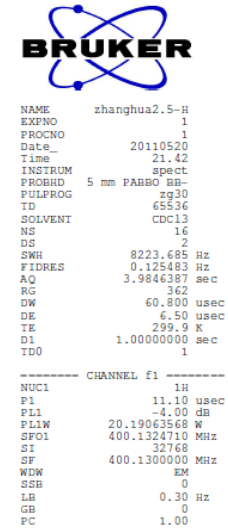



```
NAME          zhanghua110615-2.2-c
EXPNO                          2
PROCNO                         1
Date_                   20110617
Time                       10.01
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                           362
DW                        20.800 usec
DE                         6.50 usec
TE                        299.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                        13C
P1                        10.10 usec
PL1                       -3.00 dB
PL1W                64.15196228 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -4.00 dB
PL12                      11.88 dB
PL13                      12.00 dB
PL2W                20.19063568 W
PL12W               0.52137470 W
PL13W               0.50716585 W
SFO2                400.1316005 MHz
SI                         32768
SF                  100.6127690 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

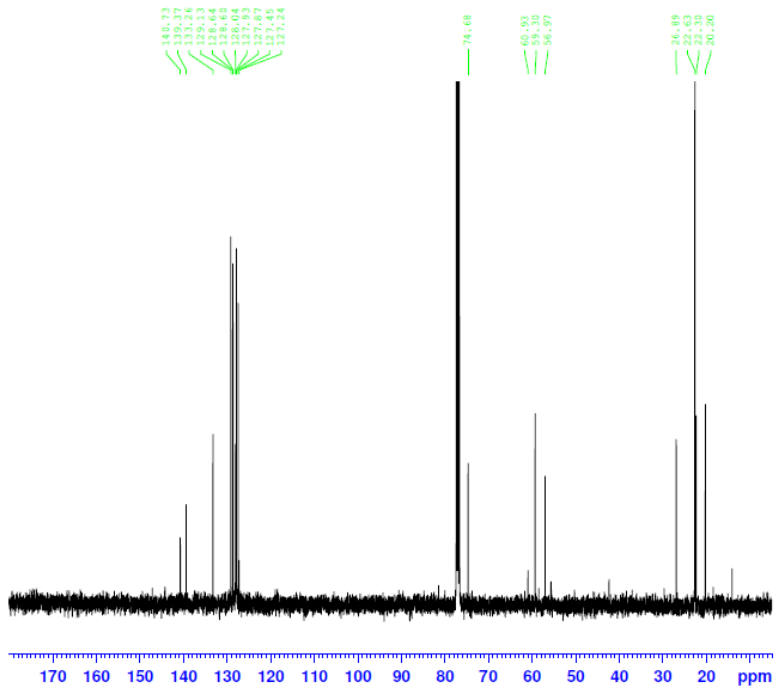


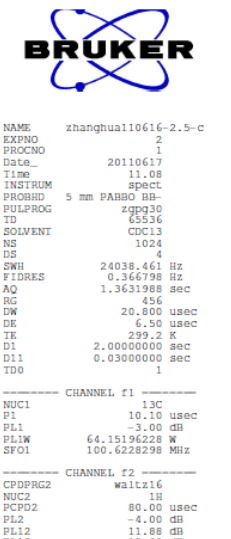
5c (^{1}H NMR and ^{13}C NMR)



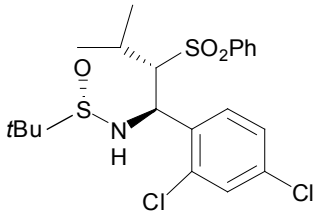


```
NAME          zhanghua2.5-H
EXPNO                     1
PROCNO                    1
Date_              20110520
Time                 21.42
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    362
DW                60.800 usec
DE                 6.50 usec
TE                299.9 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.10 usec
PL1                -4.00 dB
PL1W         20.19063568 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

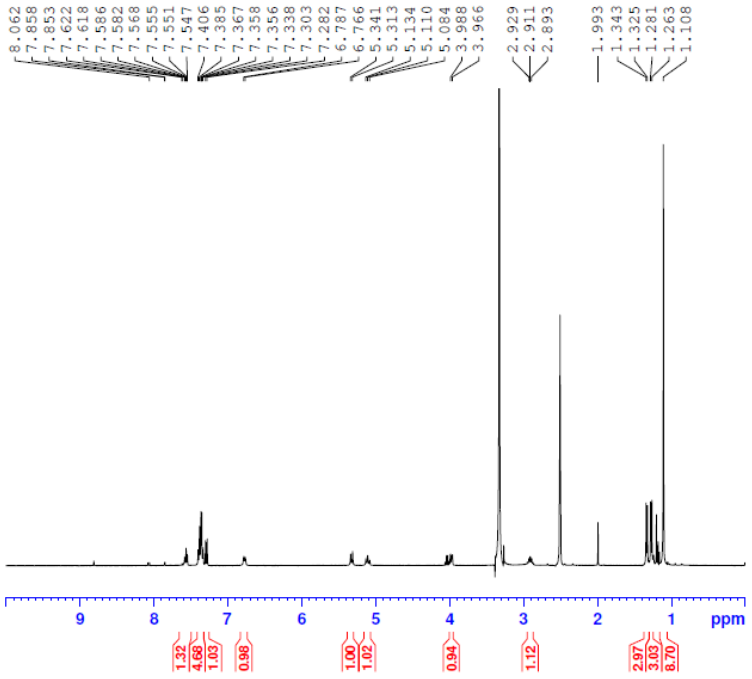




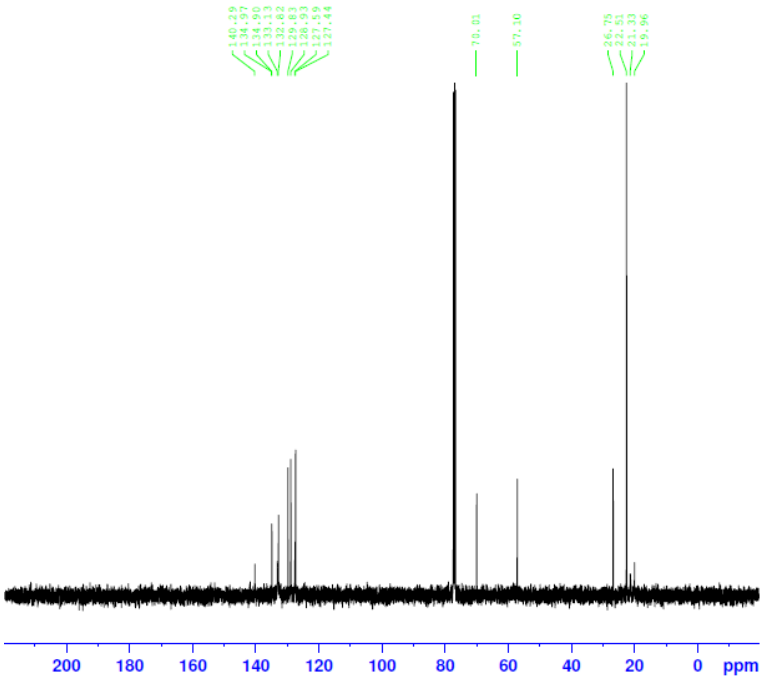
```
NAME      zhanghua110616-2.5-c
EXPNO                       2
PROCNO                      1
Date_              20110617
Time                 11.08
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    456
DW                20.800 usec
DE                 6.50 usec
TE                299.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.10 usec
PL1                -3.00 dB
PL1W         64.15196228 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                -4.00 dB
PL12               11.88 dB
PL13               12.00 dB
PL2W         20.19063568 W
PL12W        0.52137470 W
PL13W        0.50716585 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```



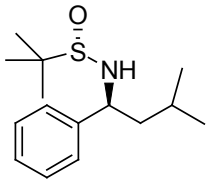
5d (^{1}H NMR and ^{13}C NMR)



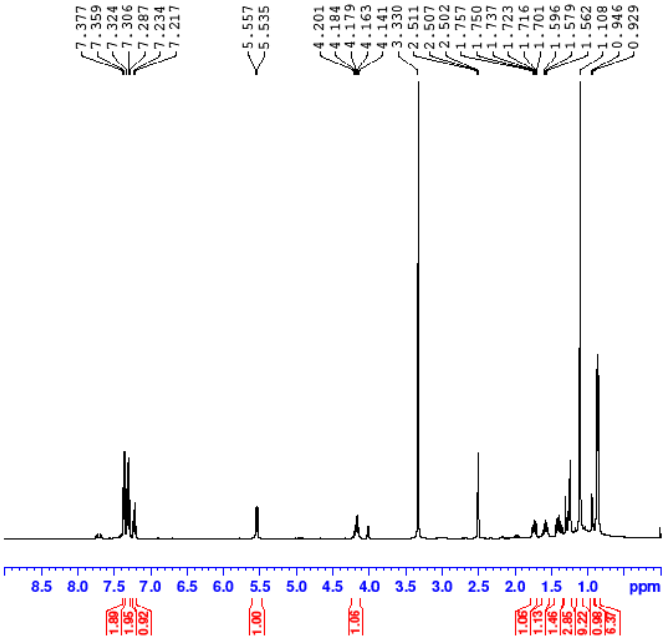

```
NAME          zhanghua2.6-H(DMSO)
EXPNO                          1
PROCNO                         1
Date_                   20110608
Time                       13.41
INSTRUM                    spect
PROBHD   5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                     DMSO
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                           203
DW                        60.800 usec
DE                         6.50 usec
TE                        300.2 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                        11.10 usec
PL1                       -4.00 dB
PL1W                20.19063568 W
SFO1               400.1324710 MHz
SI                         32768
SF                 400.1300000 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```



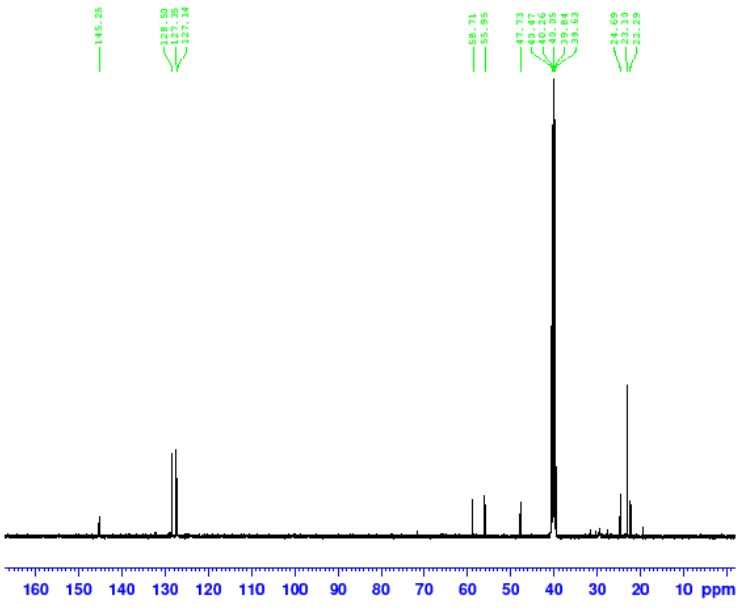

```
NAME                zhanghua2.6-H
EXPNO                           1
PROCNO                          1
Date_                    20110520
Time                        22.50
INSTRUM                     spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                          65536
SOLVENT                  CDCl3
NS                            272
DS                              4
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                            456
DW                         20.800 usec
DE                          6.50 usec
TE                         302.2 K
D1                      2.00000000 sec
D11                     0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                          9.30 usec
PL1                        -3.00 dB
PL1W                 64.15196228 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                        -4.00 dB
PL12                       13.16 dB
PL13                       12.00 dB
PL2W                 20.19063568 W
PL12W                 0.38828444 W
PL13W                 0.50716585 W
SFO2                400.1316005 MHz
SI                         32768
SF                 100.6127690 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



6 (^{1}H NMR and ^{13}C NMR)




```
NAME          zhanghua110718-5
EXPNO                        1
PROCNO                       1
Date_                 20110718
Time                     21.40
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                         16
DS                          2
SWH                 8223.685 Hz
FIDRES              0.125493 Hz
AQ                  3.9846387 sec
RG                         101
DW                    60.800 usec
DE                     6.50 usec
TE                    296.4 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                    12.86 usec
PL1                   -4.00 dB
PL1W          20.19063568 W
SFO1          400.1324710 MHz
SI                       32768
SF            400.1300000 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00
```




```
NAME          zhanghua110718-5c
EXPNO                        1
PROCNO                       1
Date_                 20110718
Time                     22.11
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                  DMSO
NS                         696
DS                           4
SWH                24038.461 Hz
FIDRES              0.366799 Hz
AQ                  1.3631988 sec
RG                         812
DW                    20.800 usec
DE                     6.50 usec
TE                    298.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                   -3.00 dB
PL1W          64.15196228 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  11.98 dB
PL13                  12.00 dB
PL2W          20.19063568 W
PL12W          0.52137470 W
PL13W          0.50716585 W
SFO2          400.1316005 MHz
SI                       32768
SF            100.6127690 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                       1.40
```