Supplementary Material (ESI) for Chemical Communications

Supporting information

Highly Enantioselective Synthesis of Tertiary Alcohols: C_2-Symmetric *N*,*N*'-Dioxide-Sc(III) Complex Promoted Direct Aldol Reaction of α-Ketoesters and Diazo Diazoacetate Esters

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1. General

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Data are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR data were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0). The enantiomeric excesses were determined by HPLC analysis on Daicel Chiralcel AD or AD-H column at 254 nm in comparison with the authentic racemates. Optical rotations were measured on a Rudolph Research analytical with a sodium lamp and are reported as follows: $[\alpha]_D^T$ (*c* = g/100 mL, solvent).

Commercial grade reagents were used without further purification. All reactions were carried out under nitrogen atmosphere and monitored by thin-layer chromatography. α-Ketoesters have been prepared in various methods, such as addition of Grignard reagents to oxalates,[1] Friedel-Crafts reaction.[2]

2. General procedure for chiral *N*,*N*′-dioxides preparation

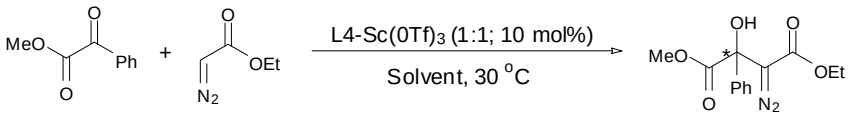
The *N*,*N*′-dioxide ligand **L1-L4, L6-L7** were synthesized by the same procedure in the literature. [3]

3. General procedure for the Aldol Reaction of α-Ketoesters and Diazoacetate Esters

The mixture of ligand **L4** (10.0 mg, 0.015 mmol), $Sc(OTf)_3$ (4.9 mg, 0.010 mmol) in CH_2Cl_2 (0.2 mL) was stirred at 30 ^oC for 30 min under nitrogen atmosphere. Then α-ketoester **1** (15 μ L, 0.1 mmol) and diazoacetate esters **2** (22 μ L, 0.2 mmol) were added sequently under stirring. The reaction mixture was stirred at 30 ^oC for 72 h and directly purified by flash chromatography on silica gel (ethyl acetate : petroleum ether = 1:10) to obtain the corresponding tertiary alcohols.

4. Optimization of the conditions

Table 1. Screening of metals and solvents.[a]

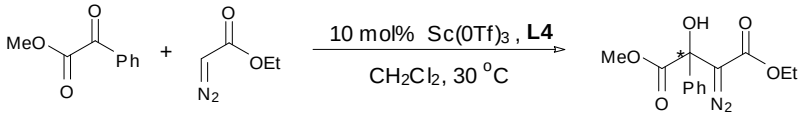


1a **2a** **3a**

entry	metal	solvent	yield (%)[b]	ee (%)[c]
1	$In(OTf)_3$	CH_2Cl_2	trace	-
2	$La(OTf)_3$	CH_2Cl_2	trace	-
3	$Yb(OTf)_3$	CH_2Cl_2	trace	-
4	$Zn(OTf)_3$	CH_2Cl_2	trace	-
5	$Sc(OTf)_3$	CH_2Cl_2	54	92
6	$Sc(OTf)_3$	toluene	25	45
7	$Sc(OTf)_3$	THF	trace	-
8	$Sc(OTf)_3$	MeOH	15	57
9	$Sc(OTf)_3$	$CHCl_3$	46	87
10	$Sc(OTf)_3$	CH_2ClCH_2Cl	50	90

[a] Reactions were carried out with α-ketoesters (0.1 mmol) and ethyl diazoacetate (0.2 mmol) in solvent (1.0 mL) at 30 °C for 48 h. [b] Isolated yield. [c] Determined by chiral HPLC.

Table 2. Optimization of the ratio of ligand to metal and the reaction concentration.[a]



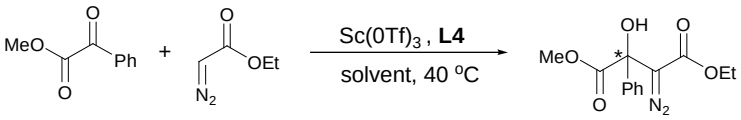
1a **2a** **3a**

entry	Ratio of ligand/metal	Reaction concentration	yield (%)[b]	ee (%)[c]
1	2:1	0.1 M	35	93
2	1.5:1	0.1 M	60	92
3	1:1	0.1 M	54	92
4	1:1.5	0.1 M	40	91

5	1:2	0.1 M	36	90
6	1:1	0.5 M	70	93
7	1:1	0.2 M	56	92
8	1:1	0.5 M	76	93

[a] Reactions were carried out with α-ketoesters (0.1 mmol) and ethyl diazoacetate (0.2 mmol) in CH_2Cl_2 at 30 °C for 48 h. [b] Isolated yield. [c] Determined by chiral HPLC.

Table 3. Optimization of the reaction in DCE and $CHCl_3$ using various catalyst loadings at 40 °C. [a]

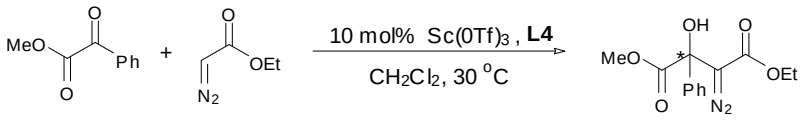


1a **2a** **3a**

entry	Catalyst loading	solvent	yield (%)[b]	ee (%)[c]
1	10	DCE	62	90
2	10	$CHCl_3$	60	90
3	5	DCE	60	89
4	5	$CHCl_3$	56	88

[a] Reactions were carried out with α-ketoesters (0.1 mmol) and ethyl diazoacetate (0.2 mmol) in solvent (0.1 mL) at 40 °C for 72 h. The ratio of ligand/metal was 1.5:1. [b] Isolated yield. [c] Determined by chiral HPLC.

Table 4. Optimization of the ratio between α-ketoester and ethyl diazoacetate. [a]



1a **2a** **3a**

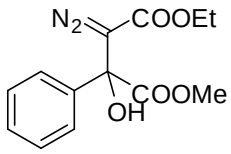
entry	ethyl diazoacetate / α-ketoesters	yield (%)[b]	ee (%)[c]
1	1	70	89
2	1.5	72	90
3	2	76	93

[a] Reactions were carried out with α-ketoesters (0.1 mmol) and various equiv of ethyl

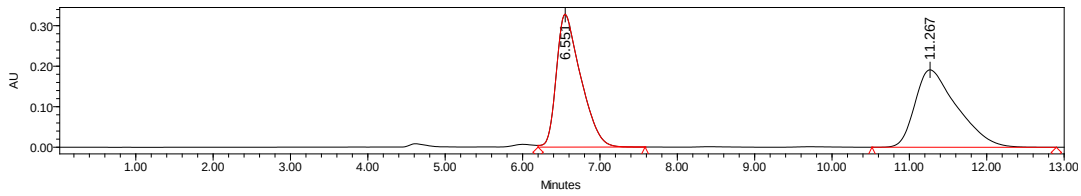
diazoacetate in 0.1 mL CH_2Cl_2 at 30 ^oC for 72 h. The ratio of ligand/metal was 1.5:1.. [b] Isolated yield. [c] Determined by chiral HPLC.

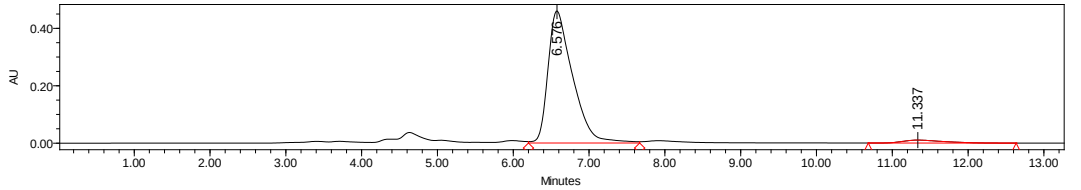
5. The analytical and spectral characterization data of direct Aldol reaction products

4-ethyl 1-methyl 3-diazo-2-hydroxy-2-phenylsuccinate

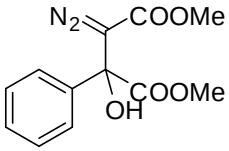


($C_{13}H_{14}N_2O_5$) a yellow viscous liquid; 76% yield, 93% *ee*, $[\alpha]_D^{27}$ = +161.7 (c = 0.24 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.5 min (major) and 11.2 min (minor); ^1H NMR (400 MHz, $CDCl_3$) 1.27 (t, *J* = 7.0 Hz, 3H), 3.79 (s, 3H), 4.17-4.29 (m, 2H), 4.52 (s, 1H), 7.31-7.34 (m, 3H), 7.35-7.36 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.3, 52.5, 60.2, 71.8, 125.3, 127.6, 128.0, 135.2, 164.7, 171.6 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{14}N_2O_5$ ([M+Na^+]) = 301.0800, Found 301.0800.

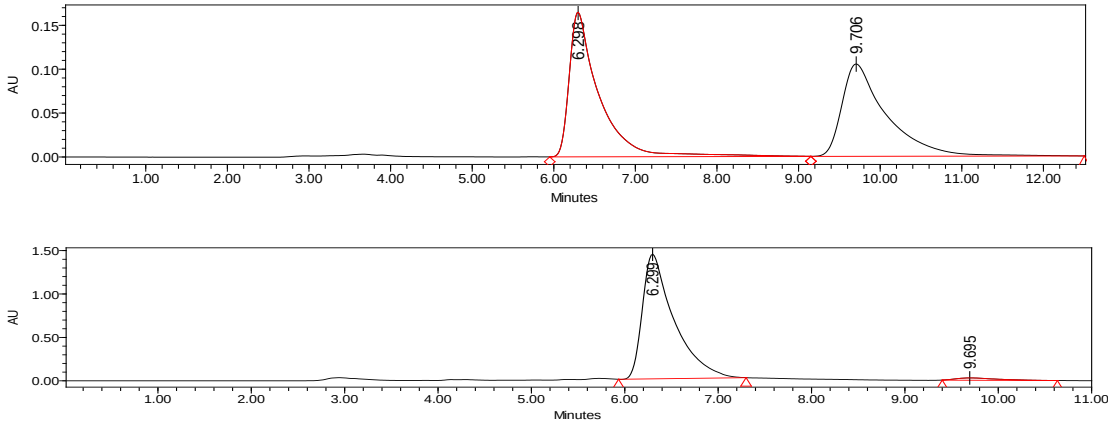




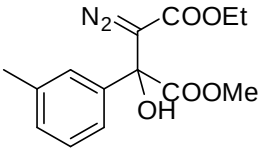
dimethyl 3-diazo-2-hydroxy-2-phenylsuccinate



($C_{12}H_{12}N_2O_5$) a yellow solid; 78% yield, 95% *ee*, $[\alpha]_D^{27}$ = +76.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.2 min (major) and 9.7 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.76-3.83 (m, 6H), 4.52 (s, 1H), 7.36-7.43 (m, 3H), 7.68-7.71 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 51.4, 53.0, 75.6, 125.3, 127.6, 128.1, 135.1, 165.0, 171.5 ppm; HRMS (ESI-TOF) calcd for $C_{12}H_{12}N_2O_5$ ([M+Na$^+$]) = 287.0644, Found 287.0643.

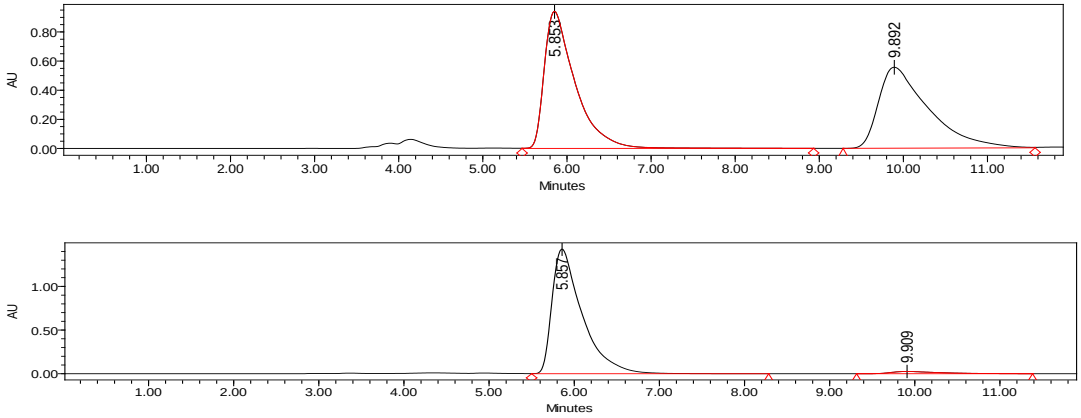


4-ethyl 1-methyl 3-diazo-2-hydroxy-2-*m*-tolylsuccinate

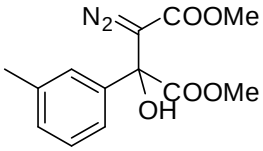


($C_{14}H_{16}N_2O_5$) a yellow viscous liquid; 68% yield, 96% *ee*, $[\alpha]_D^{27}$ = +160.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 5.8 min (major) and 9.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.0 Hz, 3H), 2.17 (s, 3H), 3.82 (s, 3H), 4.40-4.46 (m, 2H), 4.50 (s, 1H), 7.15-7.45 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.3, 20.5, 29.4, 52.9, 60.3, 122.2, 125.6, 127.6, 128.8, 135.1,

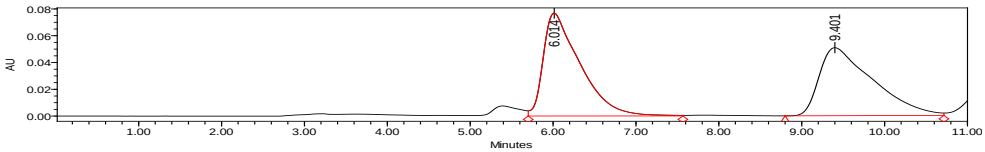
135.5, 164.8, 171.7 ppm; HRMS (ESI-TOF) calcd for $C_{14}H_{16}N_2O_5$ ([M+Na$^+$]) = 315.0957, Found 315.0967.

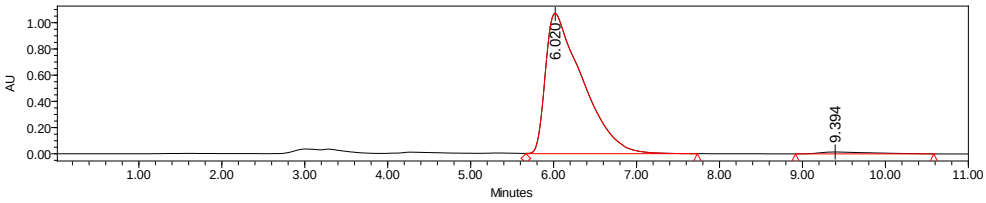


dimethyl 3-diazo-2-hydroxy-2-*m*-tolylsuccinate

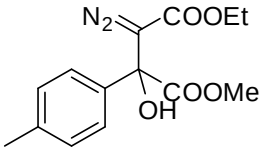


($C_{13}H_{14}N_2O_5$) a yellow solid; 70% yield, 97% *ee*, $[\alpha]_D^{27}$ = +231.5 (c = 0.11 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.0 min (major) and 9.3 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 2.37 (s, 3H), 3.80-3.82 (m, 3H), 4.20-4.30 (m, 3H), 4.50 (s,1H), 7.15-7.45 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.2, 20.4, 52.6, 60.2, 122.3, 125.6, 127.6, 128.8, 135.0, 137.5, 165.1, 171.6 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{14}N_2O_5$ ([M+Na$^+$]) = 301.0800, Found 301.0797.

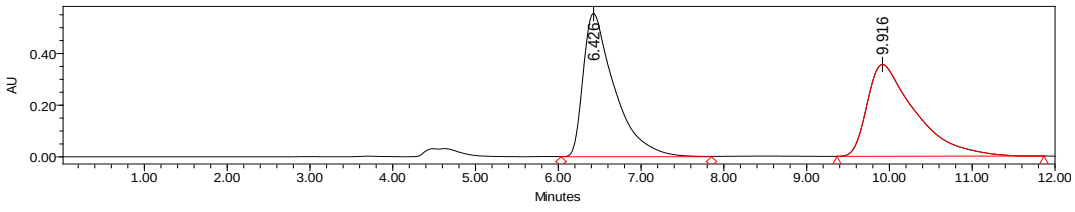


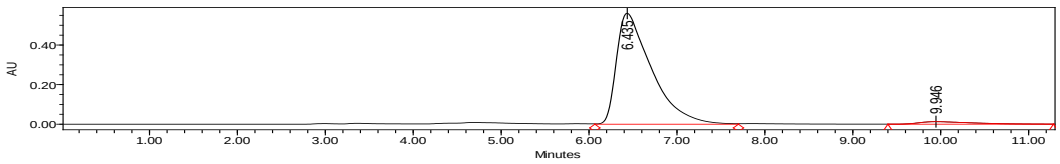


4-ethyl 1-methyl 3-diazo-2-hydroxy-2-*p*-tolylsuccinate

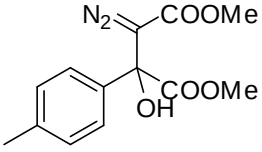


($C_{14}H_{16}N_2O_5$) a yellow viscous liquid; 70% yield, 93% *ee*, $[\alpha]_D^{27}$ = +104.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.4 min (major) and 9.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.2 Hz, 3H), 2.35 (s, 3H), 3.81 (s, 3H), 4.48 (s, 1H), 7.19-7.21 (m, 4H), 7.56-7.58 (m,2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 14.3, 21.0, 30.9, 53.9, 61.3, 77.34, 126.1, 129.4, 139.0, 165.8, 171.2 ppm; HRMS (ESI-TOF) calcd for $C_{14}H_{16}N_2O_5$ ([M+Na^+]) = 315.0957, Found 315.0958.



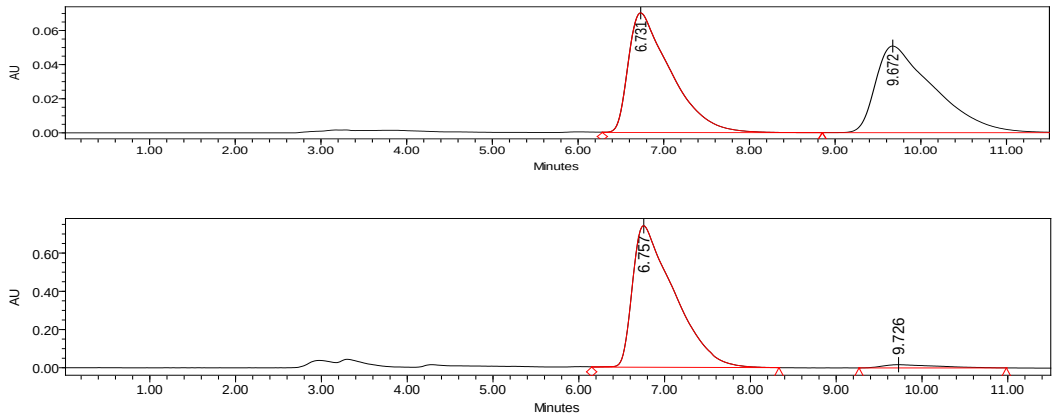


dimethyl 3-diazo-2-hydroxy-2-*p*-tolylsuccinate

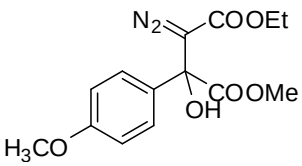


($C_{13}H_{14}N_2O_5$) a yellow solid; 70% yield, 97% *ee*, $[\alpha]_D^{27}$ = +97.5 (c = 0.16 in CH_2Cl_2); HPLC DAICEL

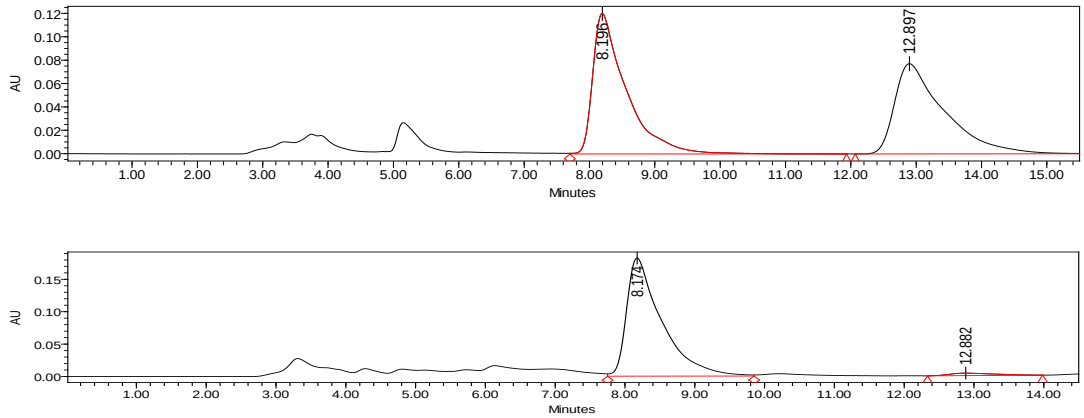
CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.7 min (major) and 9.7 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 2.35 (s, 3H), 3.80 (s, 3H), 3.82 (s, 3H), 4.48 (s,1H), 7.19-7.21 (m, 2H), 7.55-7.57 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 20.0, 51.1, 60.2, 76.2, 125.0, 127.0, 132.2, 138.0, 165.1, 171.7 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{14}N_2O_5$ ([M+Na^+]) = 301.0800, Found 301.0799.



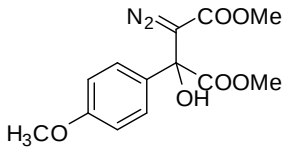
4-ethyl 1-methyl 3-diazo-2-hydroxy-2-(4-methoxyphenyl)succinate



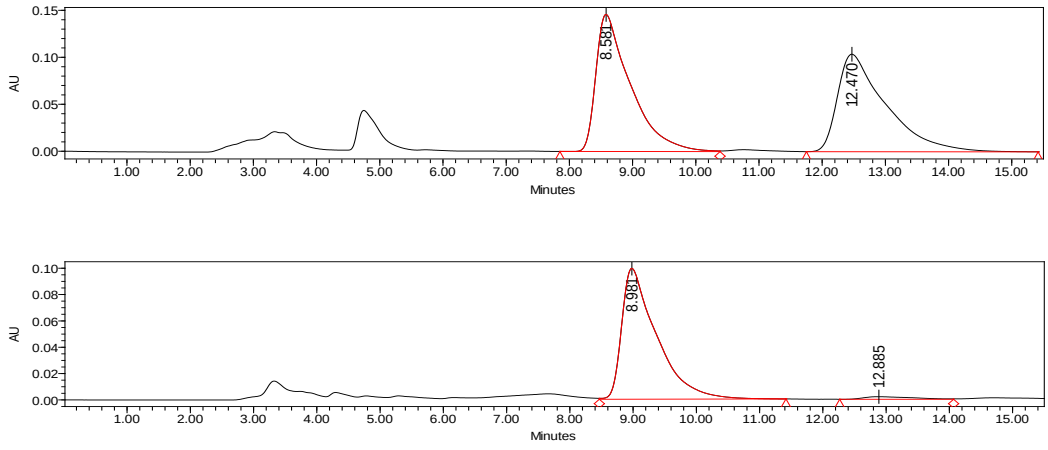
($C_{14}H_{16}N_2O_6$) a yellow liquid; 62% yield, 90% *ee*, $[\alpha]_D^{27}$ = +85.0 (c = 0.11 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 8.7 min (major) and 12.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.26-1.30 (t, *J* = 7.4 Hz, 3H), 3.81 (s, 3H), 3.82 (s, 3H), 4.24-4.48 (s, 2H), 6.90-6.92 (m, 2H), 7.60-7.62 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 14.2, 22.7, 29.5, 31.9, 53.3, 61.3, 114.0, 127.5, 128.1, 160.1, 165.8, 171.9 ppm.



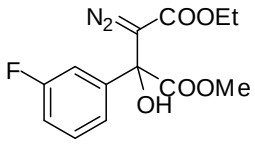
dimethyl 3-diazo-2-hydroxy-2-(4-methoxyphenyl)succinate



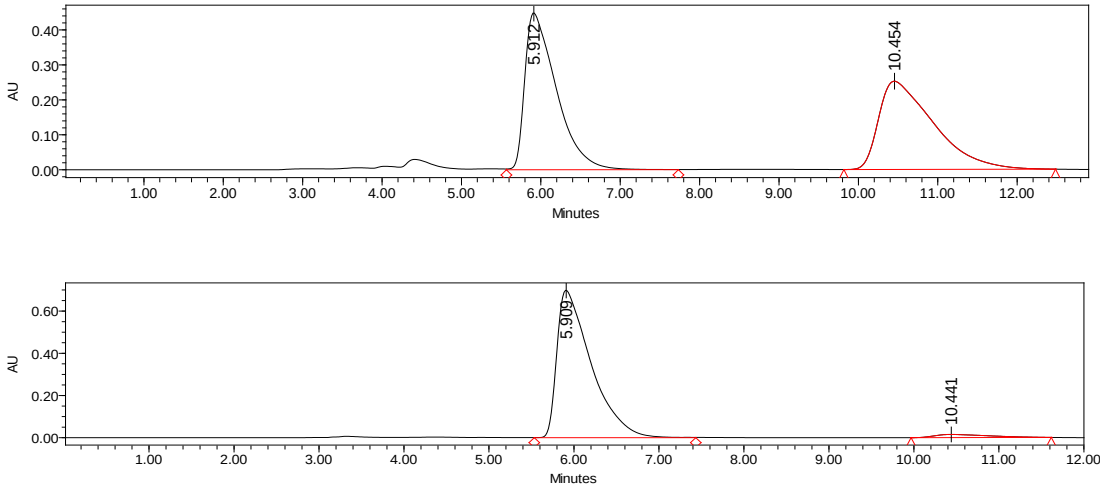
($C_{13}H_{14}N_2O_6$) a yellow solid; 68% yield, 91% *ee*, $[\alpha]_D^{27}$ = +120.0 (c = 0.12 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 8.5 min (major) and 12.4 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.79 (s, 3H), 3.80 (s, 3H), 4.46(s, 1H), 6.89-6.92 (m, 2H), 7.58-7.60 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 22.7, 29.6, 31.9, 54.1, 55.2, 114.1, 127.5, 160.1, 166.1, 172.8 ppm.



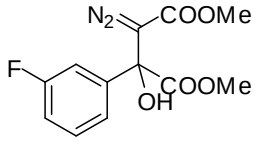
4-ethyl 1-methyl 3-diazo-2-(3-fluorophenyl)-2-hydroxysuccinate



($C_{13}H_{13}FN_2O_5$) a yellow liquid; 62% yield, 94% *ee*, $[\alpha]_D^{27}$ = +450.0 (c = 0.10 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 5.9 min (major) and 10.4 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.0 Hz, 3H), 3.84 (s, 3H), 4.22-4.29 (m, 2H), 4.56 (s, 1H), 7.35-7.39 (m, 2H), 7.40-7.45 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 14.1, 53.2, 60.3, 76.2, 112.6, 115.1, 120.9, 137.8, 160.6, 164.8, 171.0 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{13}FN_2O_5$ ([M+Na^+]) = 319.0706, Found 319.0699.

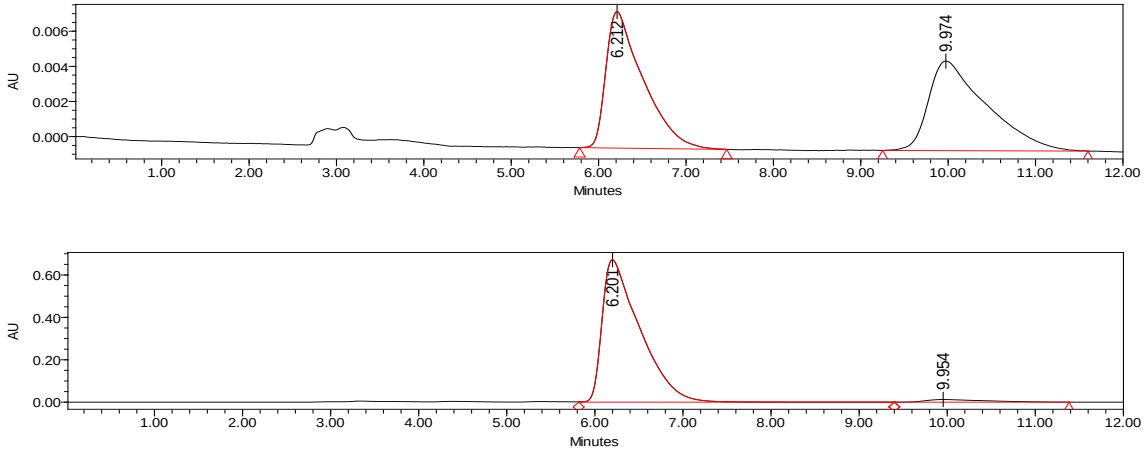


dimethyl 3-diazo-2-(3-fluorophenyl)-2-hydroxysuccinate

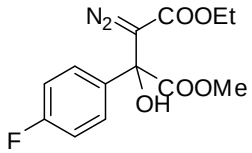


($C_{12}H_{11}FN_2O_5$) a yellow solid; 75% yield, 95% *ee*, $[\alpha]_D^{27}$ = +166.9 (c = 0.32 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.2 min (major) and 9.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.84 (s, 3H), 4.56 (s, 1H),

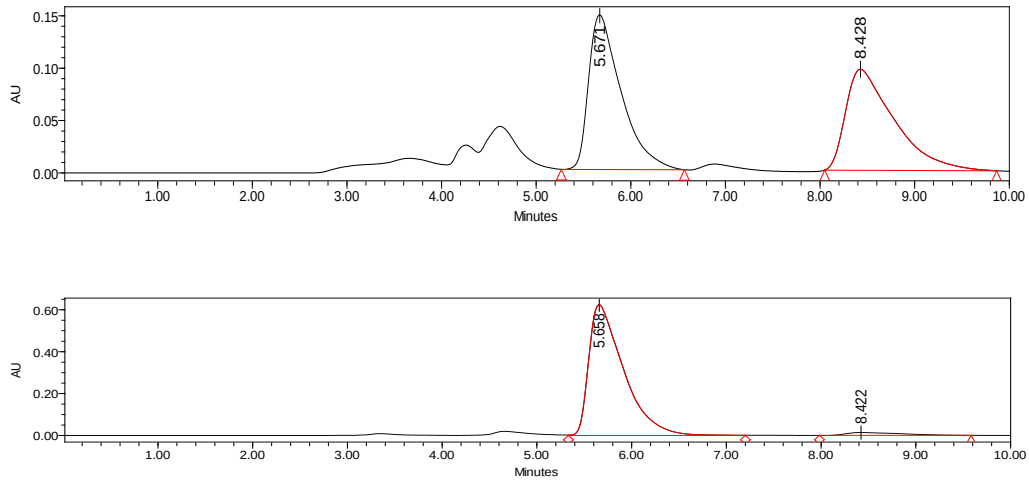
7.35-7.45 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 51.2, 53.1, 76.2, 112.6, 115.1, 120.9, 129.2, 137.8, 160.4, 164.8, 171.0 ppm; HRMS (ESI-TOF) calcd for $C_{12}H_{11}FN_2O_5$ ([M+Na$^+$]) = 315.0550, Found 315.0548.



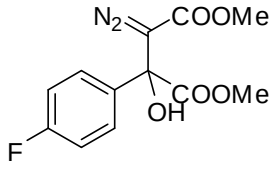
4-ethyl 1-methyl 3-diazo-2-(4-fluorophenyl)-2-hydroxysuccinate



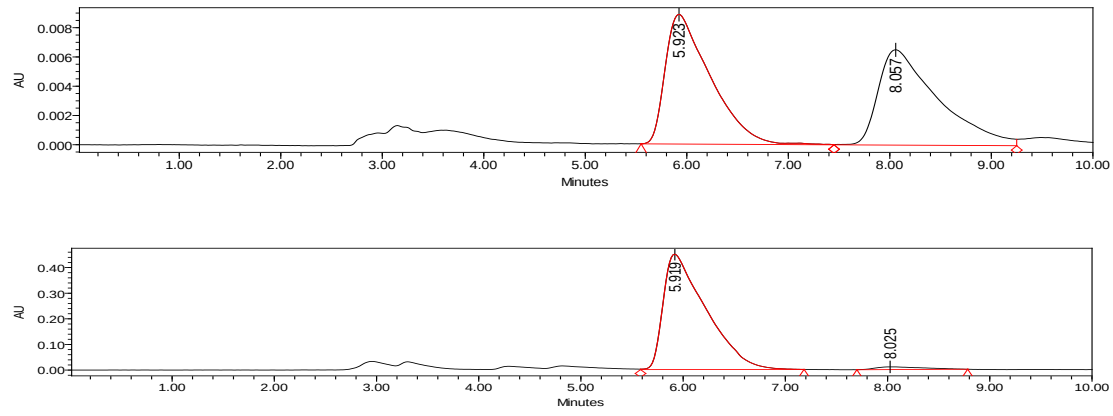
($C_{13}H_{13}FN_2O_5$) a yellow liquid; 66% yield, 93% *ee*, $[\alpha]_D^{27}$ = +125.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 5.6 min (major) and 8.4 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.2 Hz, 3H), 3.83 (s, 3H), 4.24-4.30 (m, 2H), 4.54 (s, 1H), 7.07-7.11 (m, 2H), 7.68-7.69 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.3, 28.6, 53.0, 60.3, 114.6, 127.2, 130.9, 160.6, 164.5, 171.4 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{13}FN_2O_5$ ([M+Na$^+$]) = 319.0706, Found 319.0706.



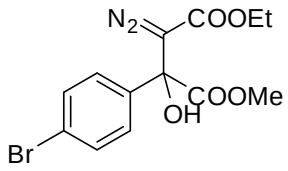
dimethyl 3-diazo-2-(4-fluorophenyl)-2-hydroxysuccinate



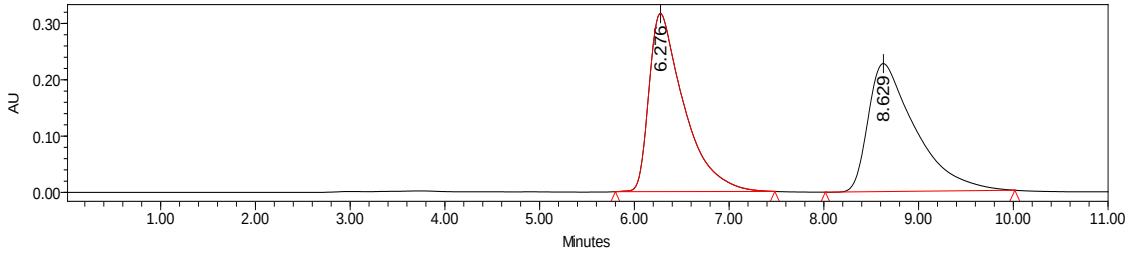
($C_{12}H_{11}FN_2O_5$) a yellow solid; 67% yield, 95% *ee*, $[\alpha]_D^{27}$ = +76.7 (c = 0.24 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 5.9 min (major) and 8.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.83 (s, 3H), 4.54 (s, 1H), 7.07-7.11(m, 2H), 7.67-7.69 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 28.6, 52.1, 114.7, 127.2, 130.9, 160.8, 163.2, 164.9, 171.3 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{14}N_2O_5$ ([M+Na^+]) = 315.0550, Found 315.0545.

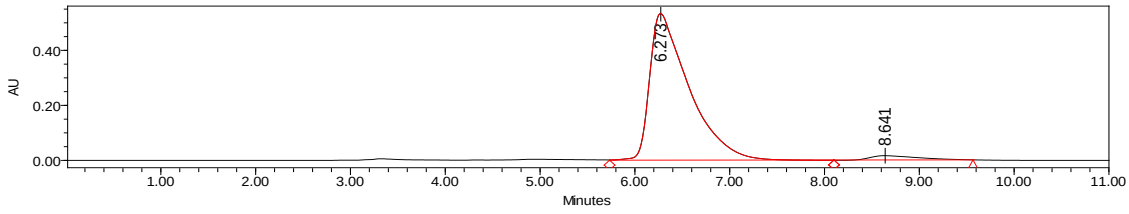


4-ethyl 1-methyl 2-(4-bromophenyl)-3-diazo-2-hydroxysuccinate

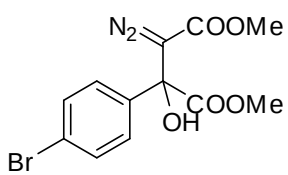


($C_{13}H_{13}BrN_2O_5$) a yellow liquid; 66% yield, 94% *ee*, $[\alpha]_D^{27}$ = +133.9 (c = 0.36 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.2 min (major) and 8.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.2 Hz, 3H), 3.82 (s, 3H), 4.22-4.31 (m, 2H), 4.53 (s, 1H), 7.52-7.54 (m, 2H), 7.58-7.60 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 14.3, 54.2, 61.4, 77.3, 123.6, 128.1, 131.9, 135.4, 165.5, 172.2 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{13}BrN_2O_5$ ([M+Na$^+$]) = 378.9906, Found 378.9902.



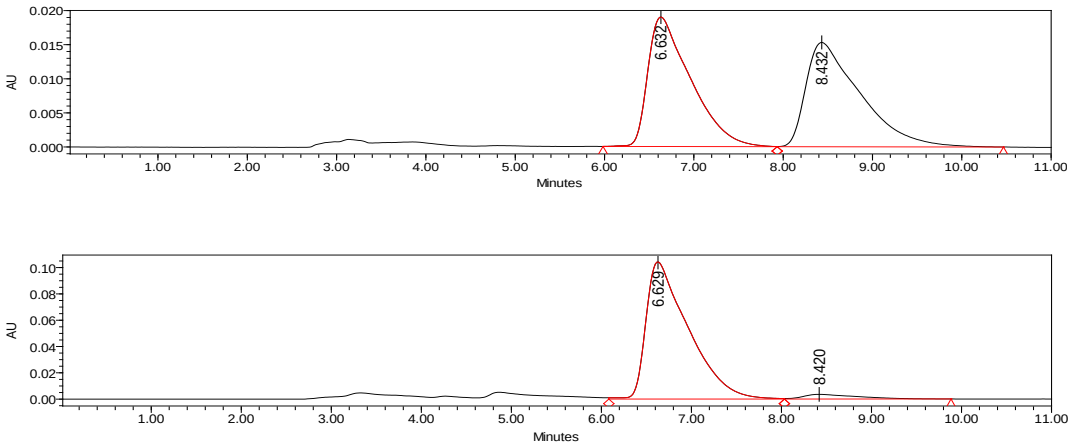


dimethyl 2-(4-bromophenyl)-3-diazo-2-hydroxysuccinate

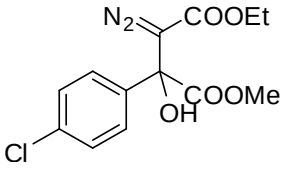


($C_{12}H_{11}BrN_2O_5$) a yellow solid; 85% yield, 94% *ee*, $[\alpha]_D^{27}$ = +120.8 (c = 0.24 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane =

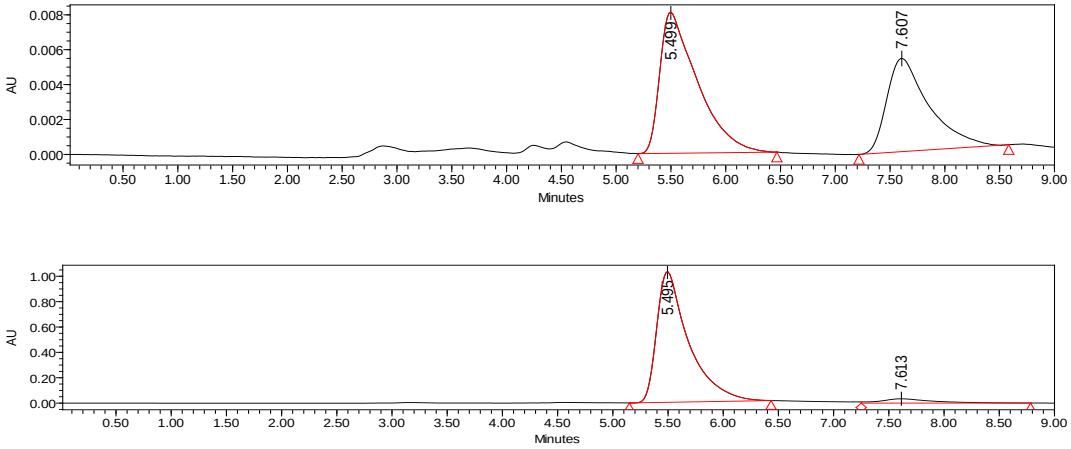
20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.6 min (major) and 8.4 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.76-3.83 (m, 6H), 4.52 (s, 1H), 7.36-7.43 (m, 3H), 7.68-7.71 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 51.1, 53.0, 75.6, 114.7, 125.2, 127.6, 128.1, 165.0, 171.5 ppm; HRMS (ESI-TOF) calcd for $C_{12}H_{11}BrN_2O_5$ ([M+Na$^+$]) = 364.9749, Found 364.9745.



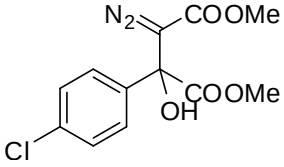
4-ethyl 1-methyl 2-(4-chlorophenyl)-3-diazo-2-hydroxysuccinate



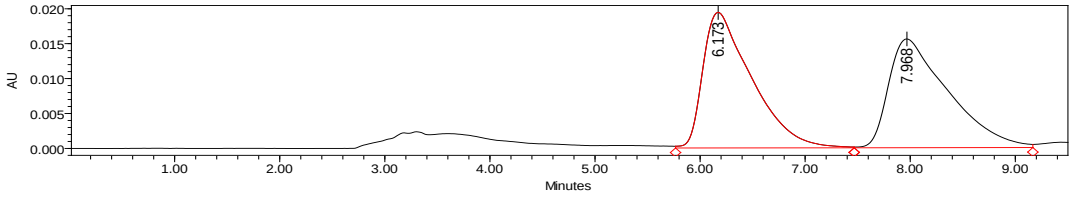
($C_{13}H_{13}ClN_2O_5$) a yellow liquid; 82% yield, 95% *ee*, $[\alpha]_D^{27}$ = +86.0 (c = 0.30 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 5.4 min (major) and 7.6 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.27-1.31 (t, *J* = 7.0 Hz, 3H), 3.82 (s, 3H), 4.21-4.30 (m, 2H), 4.53 (s, 1H), 7.37-7.39 (m, 2H), 7.63-7.64 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.3, 28.6, 53.2, 60.4, 126.8, 127.9, 133.8, 134.3, 164.5, 171.3 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{13}ClN_2O_5$ ([M+Na$^+$]) = 335.0411, Found 335.0409.

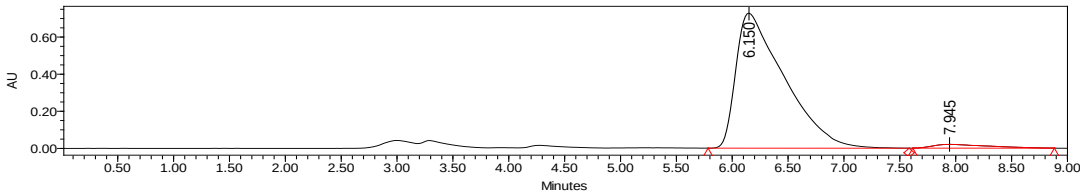


dimethyl 2-(4-chlorophenyl)-3-diazo-2-hydroxysuccinate

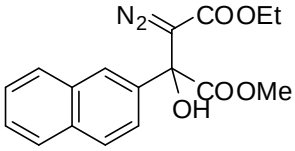


($C_{12}H_{11}ClN_2O_5$) a yellow solid; 84% yield, 94% *ee*, $[\alpha]_D^{27}$ = +125.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 6.1 min (major) and 7.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.83 (s, 3H), 4.53 (s, 1H), 7.37-7.39 (m, 2H), 7.64-7.66 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 29.7, 52.2, 54.2, 127.8, 129.0, 128.1, 134.8, 135.3, 165.9, 172.2 ppm; HRMS (ESI-TOF) calcd for $C_{12}H_{11}ClN_2O_5$ ([M+Na^+]) = 321.0254, Found 321.0247.

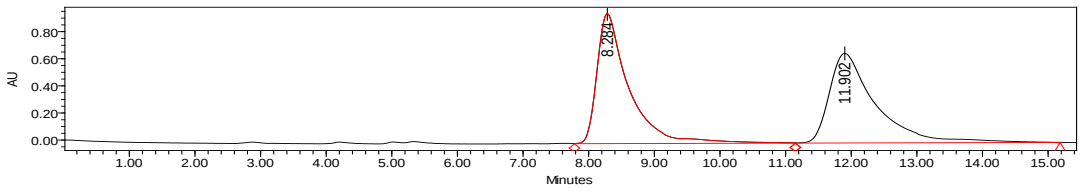


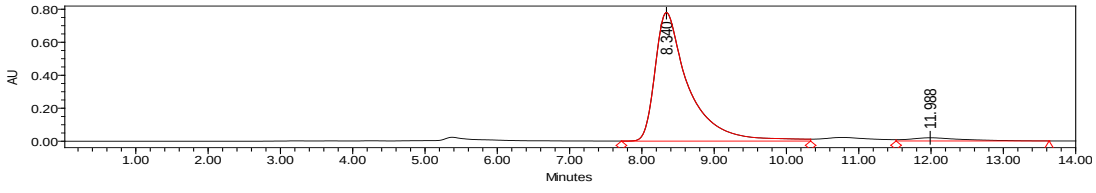


4-ethyl 1-methyl 3-diazo-2-hydroxy-2-(naphthalen-2-yl)succinate

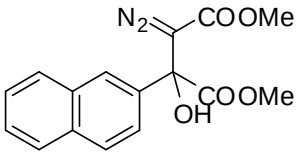


($C_{17}H_{16}N_2O_5$) a yellow solid; 64% yield, 92% *ee*, $[\alpha]_D^{27}$ = +142.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 8.3 min (major) and 11.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.29-1.32 (t, *J* = 7.0 Hz, 3H), 3.82 (s, 3H), 3.82 (s, 3H), 3.83-4.31(m, 2H), 4.34 (s, 1H), 7.51-8.24 (m, 7H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 14.7, 30.0, 54.4, 61.7, 123.8, 126.4, 126.8, 127.3, 127.8, 128.9, 129.0, 129.1, 133.4, 133.6, 165.1, 171.5 ppm.



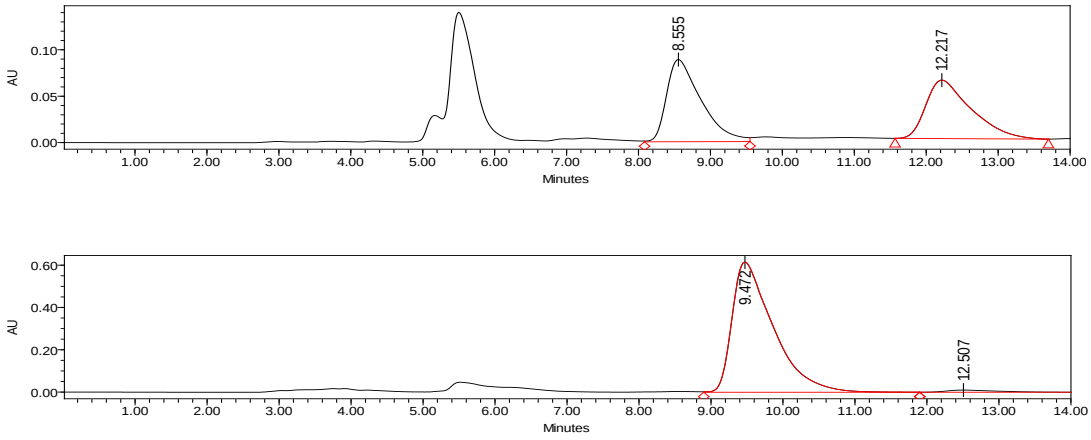


dimethyl 3-diazo-2-hydroxy-2-(naphthalen-2-yl)succinate

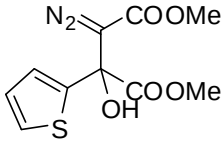


($C_{16}H_{14}N_2O_5$) a yellow solid; 80% yield, 94% *ee*, $[\alpha]_D^{27}$ = +35.0 (c = 0.10 in CH_2Cl_2); HPLC DAICEL

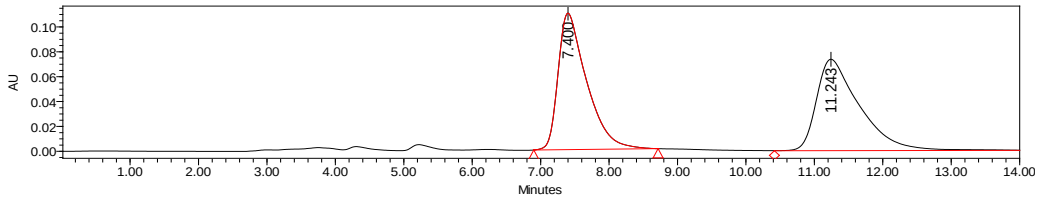
CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 8.5 min (major) and 12.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.82 (s, 3H), 4.63 (s, 1H), 7.49-7.74 (m, 7H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 28.6, 512, 53.1, 122.4, 125.1, 125.5, 125.9, 126.5, 127.6, 127.6, 128.1, 132.0, 132.3, 165.1, 171.5 ppm; HRMS (ESI-TOF) calcd for $C_{16}H_{14}N_2O_5$ ([M+Na^+]) = 321.0254, Found 321.0247.

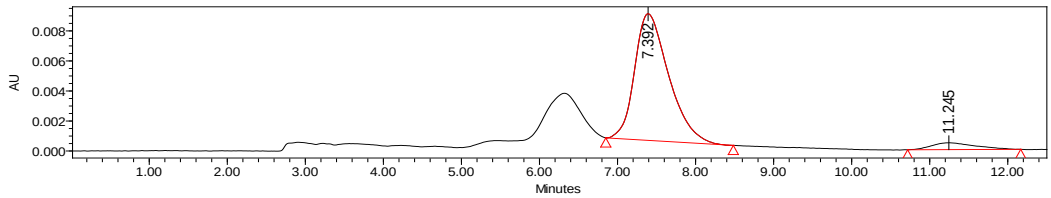


dimethyl 3-diazo-2-hydroxy-2-(thiophen-2-yl)succinate

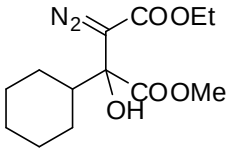


($C_{10}H_{10}N_2O_5S$) a yellow solid; 65% yield, 87% *ee*, $[\alpha]_D^{27}$ = +35.0 (c = 0.10 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 7.4 min (major) and 11.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.82 (s, 3H), 4.63 (s, 1H), 7.49-7.74 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 51.2, 53.1, 65.3, 126.5, 127.6, 127.6, 129.1, 165.1, 171.5 ppm.

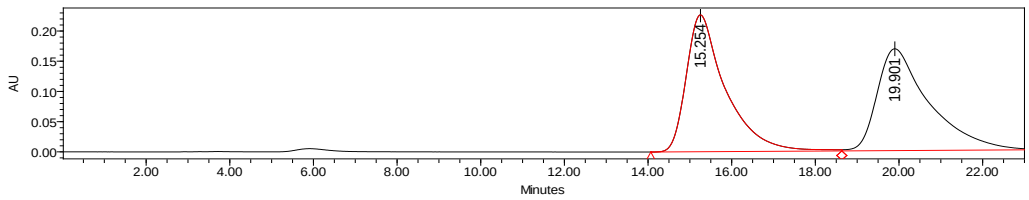


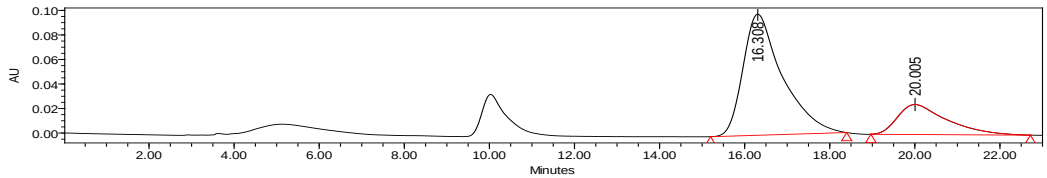


dimethyl 2-cyclohexyl-3-diazo-2-hydroxysuccinate

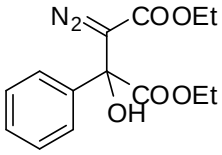


($C_{13}H_{20}N_2O_5$) a yellow solid; 35% yield, 52% *ee*, $[\alpha]_D^{27}$ = +2.6 (c = 0.11 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD-H, 2-propanol/n-hexane = 1/99, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 15.3 min (major) and 20.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 1.25-1.28 (t, *J* = 7.0 Hz, 3H), 1.33-1.36 (m, 4H), 1.61-1.81 (m, 7H), 3.80 (s, 3H), 4.20-4.24 (m, 2H), 4.42 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 13.3, 24.9, 25.1, 25.6, 28.6, 41.7, 52.1, 60.1, 165.4, 173.0 ppm; HRMS (ESI-TOF) calcd for $C_{13}H_{20}N_2O_5$ ([M+Na^+]) = 307.1270, Found 307.1270.

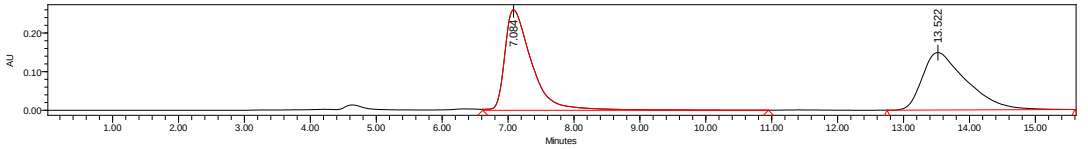


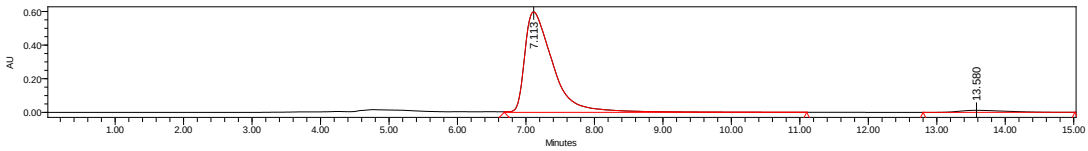


diethyl 3-diazo-2-hydroxy-2-phenylsuccinate

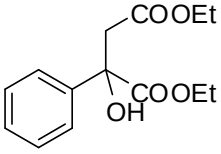


$C_{13}H_{14}N_2O_6$) a yellow solid; 75% yield, 94% *ee*, $[\alpha]_D^{27}$ = +192.0 (c = 0.20 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 7.1 min (major) and 13.5 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) 3.81 (s, 3H), 3.82 (s, 3H), 4.63 (s, 1H), 7.49-7.74 (m, 7H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 28.6, 512, 53.1, 122.4, 125.1, 125.5, 125.9, 126.5, 127.6, 127.6, 128.1, 132.0, 132.3, 165.1, 171.5 ppm.

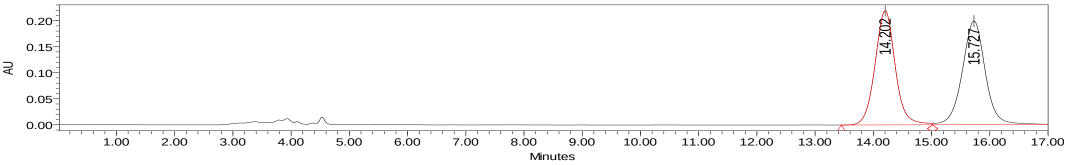




diethyl 2-hydroxy-2-phenylsuccinate



$C_{13}H_{14}N_2O_6$) a yellow liquid; 80% yield, 94% *ee*, $[\alpha]_D^{27}$ = +6.5 (c = 0.10 in CH_2Cl_2); HPLC DAICEL CHIRALCEL AD-H, 2-propanol/n-hexane = 20/80, flow rate = 1.0 mL/min, λ = 254 nm, retention time: 14.2 min (minor) and 15.1 min (major); This compound was identical in all respects (^{1}HNMR, ^{13}CNMR, mass spetra) to that previously reported. [4]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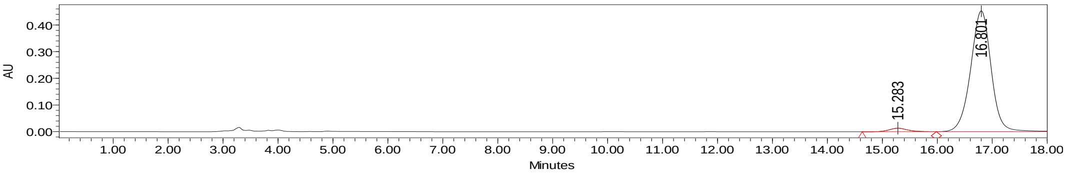
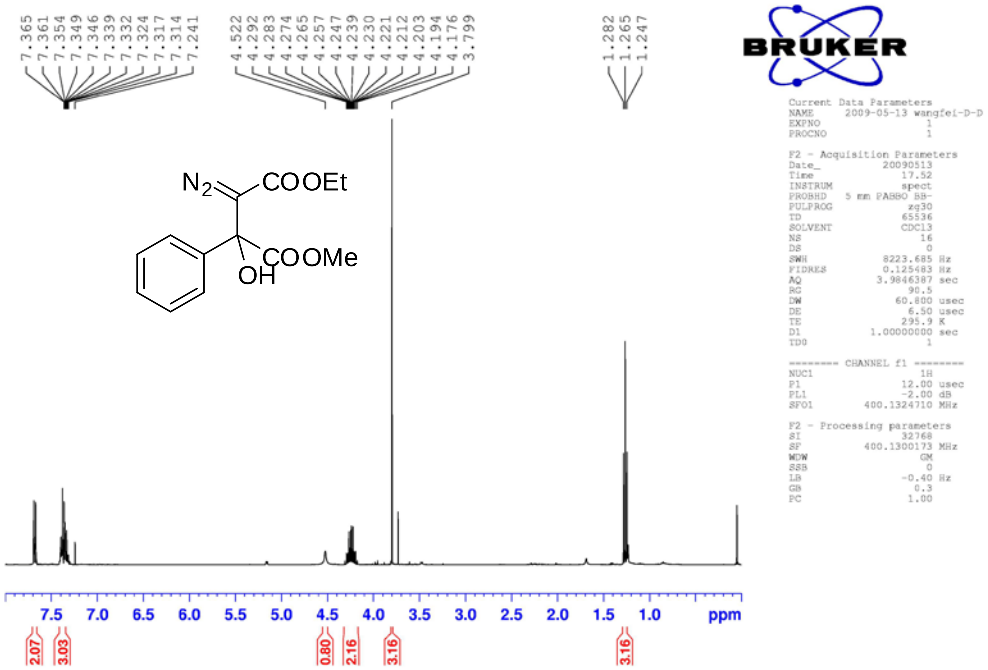
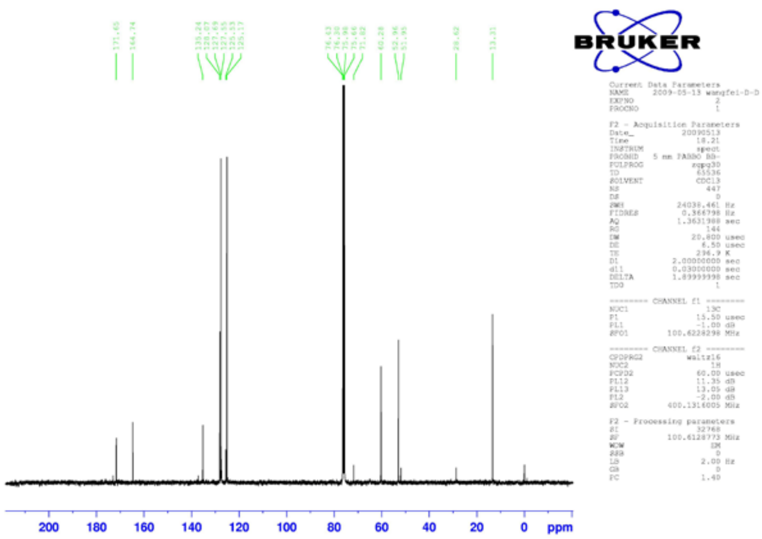
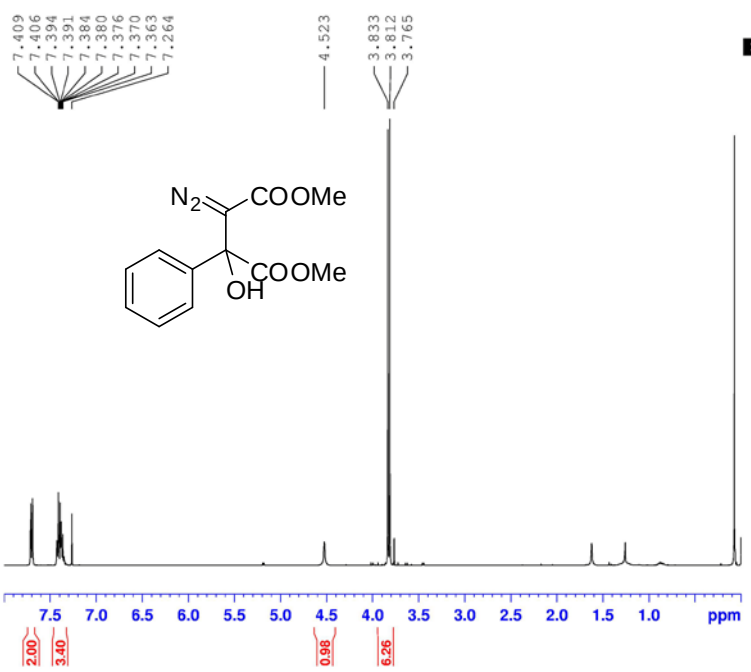
15.727

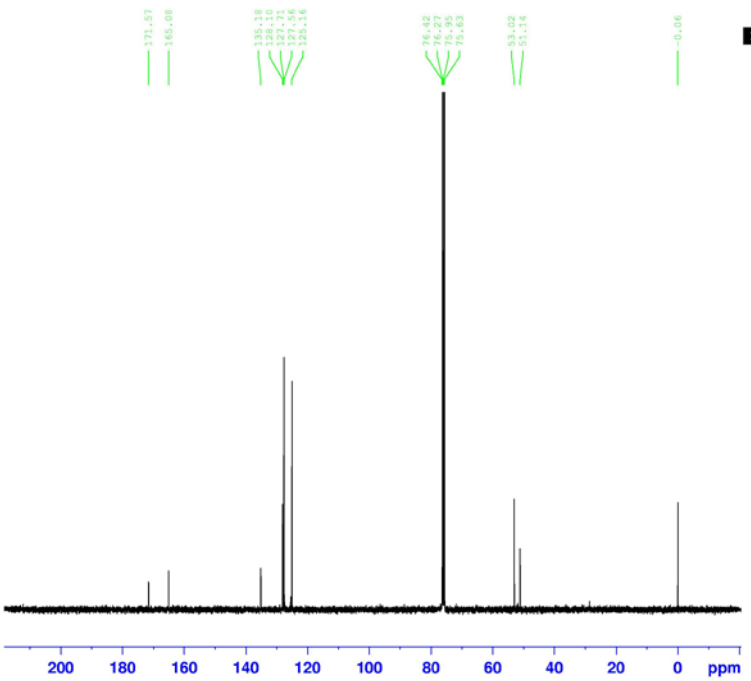

AU
Minutes
15.283
16.801

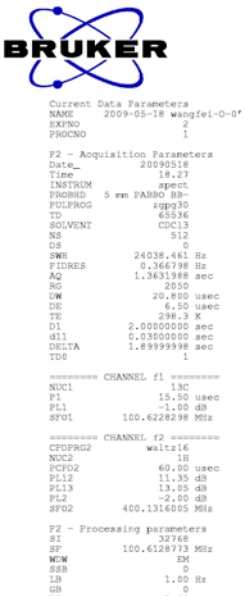

7.365
7.361
7.354
7.349
7.346
7.339
7.332
7.324
7.317
7.314
7.241
4.522
4.292
4.283
4.274
4.265
4.257
4.247
4.239
4.230
4.221
4.212
4.203
4.194
4.176
3.799
1.282
1.265
1.247
BRUKER
N2
COOEt
COOMe
OH
Current Data Parameters
NAME 2009-05-13 wangfei-D-D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 17.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
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 400.1300173 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00
ppm
2.07
3.03
0.80
2.16
3.16
3.16

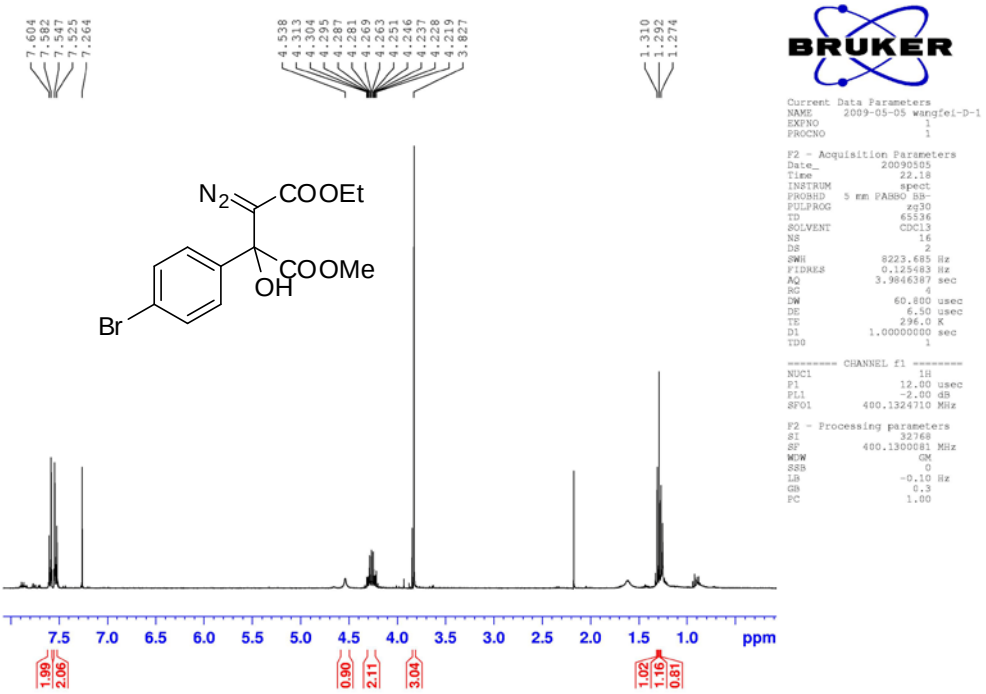

BRUKER
ppm

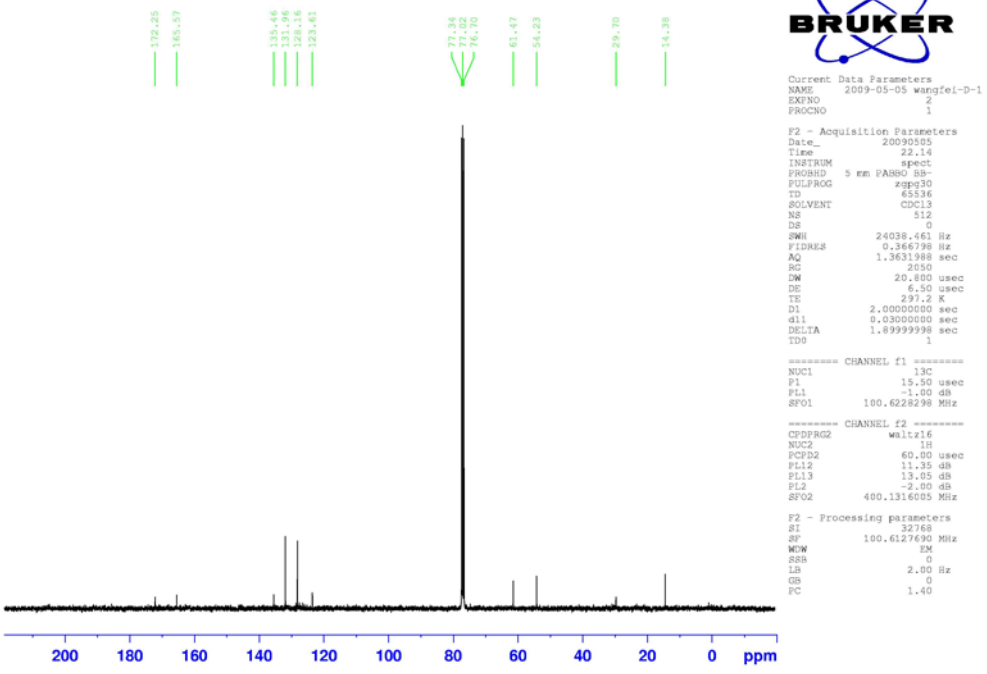

7.409
7.406
7.394
7.391
7.384
7.380
7.376
7.370
7.363
7.264
4.523
3.833
3.812
3.765
N2
COOMe
COOMe
OH
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00
3.40
0.98
6.26

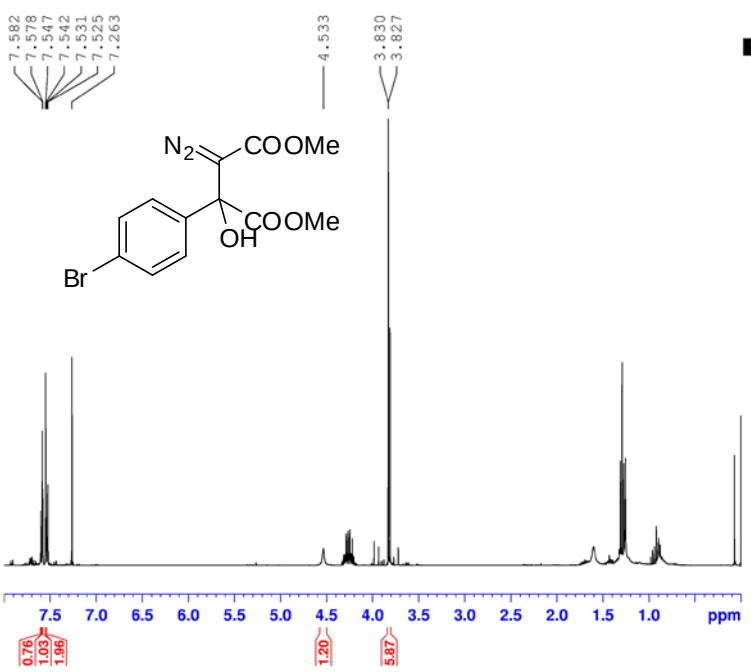

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-0'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 297.0 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 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


171.57
165.08
135.18
132.10
129.71
127.56
126.16
76.42
76.27
75.95
75.63
53.02
51.14
-0.06
200 180 160 140 120 100 80 60 40 20 0 ppm

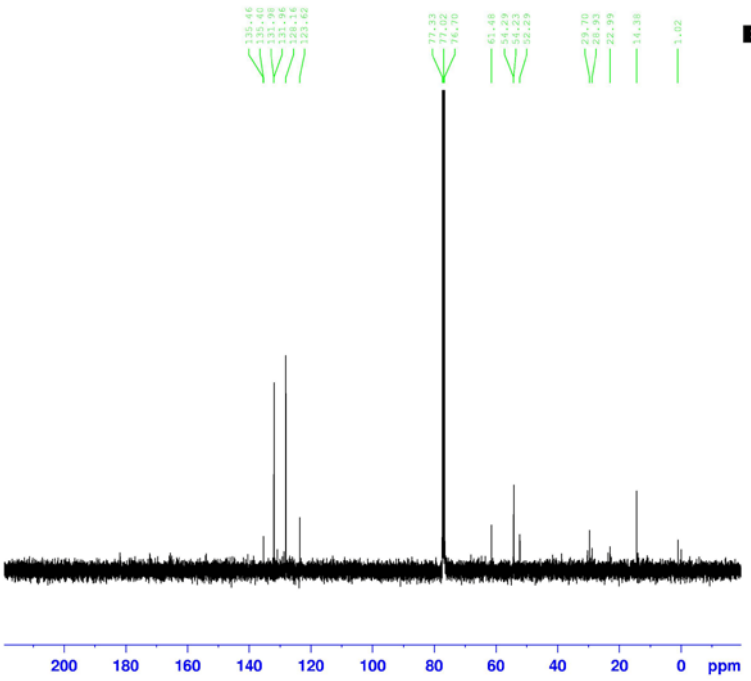

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-0'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 18.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.3 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 100.6128773 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

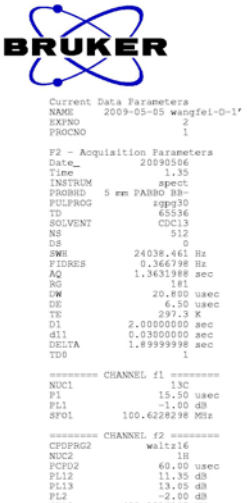

7.604
7.582
7.547
7.525
7.264
4.538
4.313
4.304
4.295
4.287
4.281
4.269
4.263
4.231
4.226
4.217
4.228
4.219
3.827
1.310
1.292
1.274
N2
COOEt
COOMe
OH
Br
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.99
2.06
0.90
2.11
3.04
1.02
1.16
0.81
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090505
Time 22.18
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 4
DW 60.800 usec
DE 6.50 usec
TE 296.0 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 400.1300081 MHz
WDW GM
SSB 0
LB -0.10 Hz
GB 0.3
PC 1.00

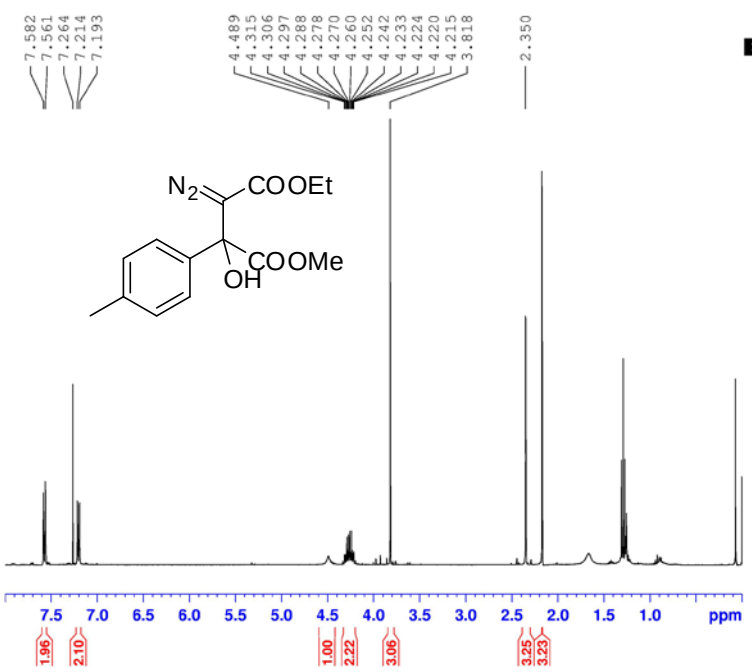

172.25
165.57
135.46
131.96
128.16
123.61
77.34
77.00
76.70
61.47
54.23
29.70
14.38
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090505
Time 22.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
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 297.2 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


7.582
7.578
7.547
7.542
7.531
7.525
7.263
4.533
3.830
3.827
N2
COOMe
COOMe
OH
Br
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.76
1.03
1.96
1.20
5.87

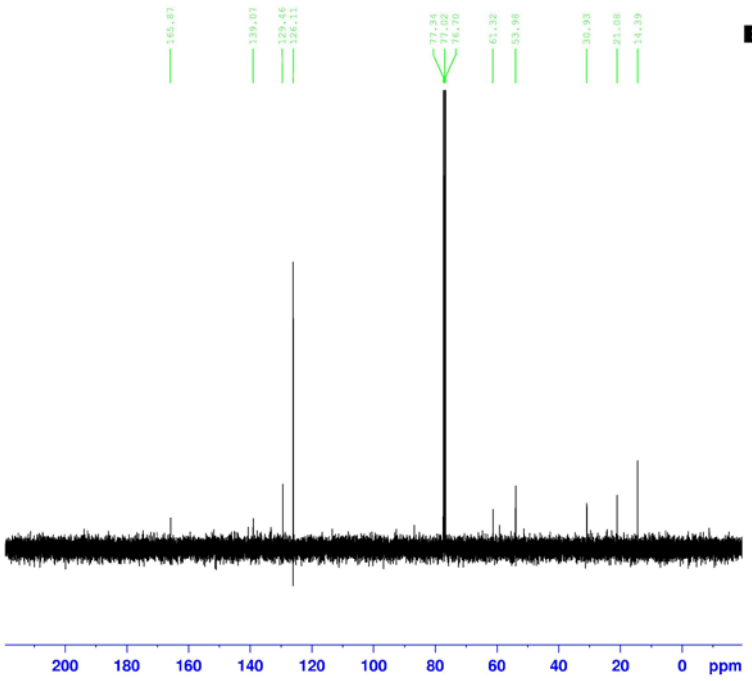

BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-1'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090506
Time 1.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 296.4 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 400.1300081 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.1
PC 1.00

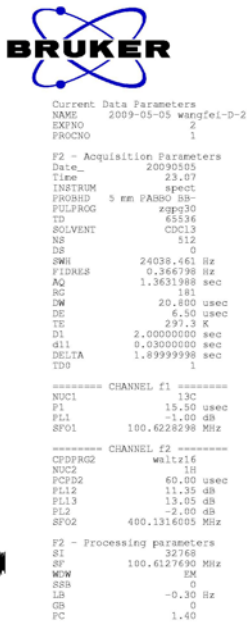

135.46
135.40
131.98
131.96
128.16
123.62
77.33
77.02
76.70
61.46
54.29
54.23
52.29
29.70
28.93
22.99
14.38
1.02
200 180 160 140 120 100 80 60 40 20 0 ppm

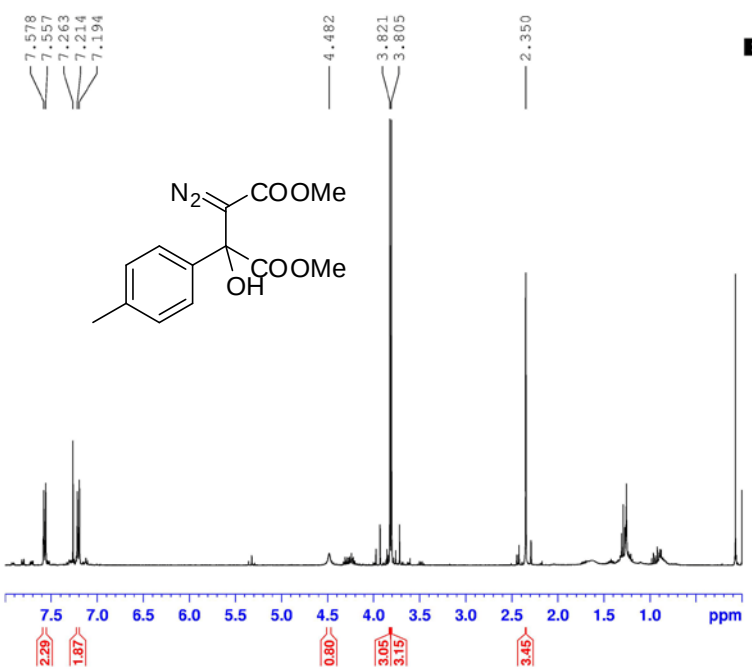

BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-1'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090506
Time 1.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
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 0.50 Hz
GB 0
PC 1.40


7.582
7.561
7.264
7.214
7.193
4.489
4.315
4.306
4.297
4.288
4.278
4.270
4.260
4.252
4.242
4.233
4.224
4.220
4.215
3.818
2.350
N2
COOEt
COOMe
OH
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.96
2.10
1.00
2.22
3.06
3.25
3.23

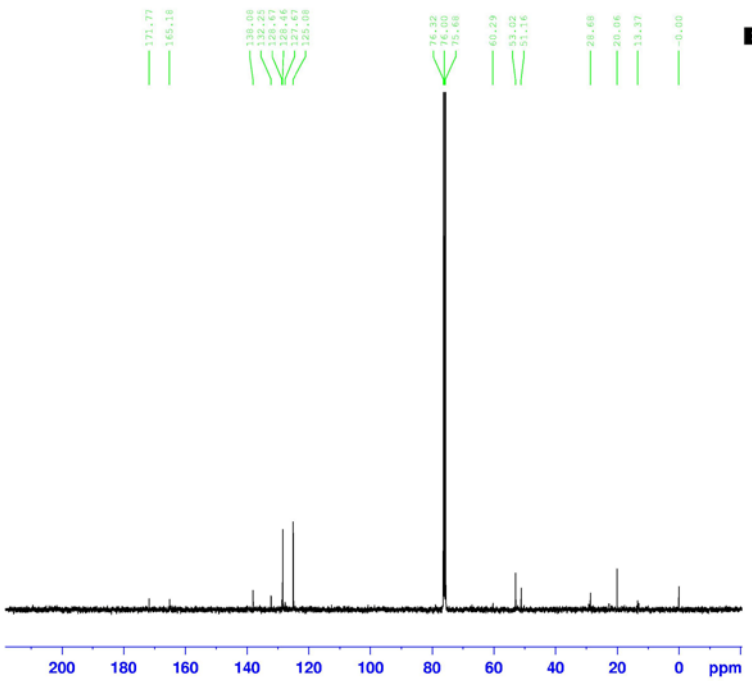

BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090505
Time 22.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 295.7 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 400.1300079 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00

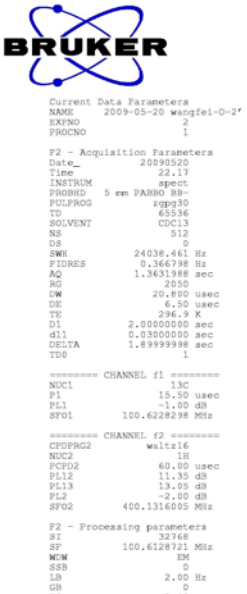

165.87
139.07
129.46
126.11
77.34
77.02
76.70
61.32
53.98
30.93
21.08
14.39
200 180 160 140 120 100 80 60 40 20 0 ppm

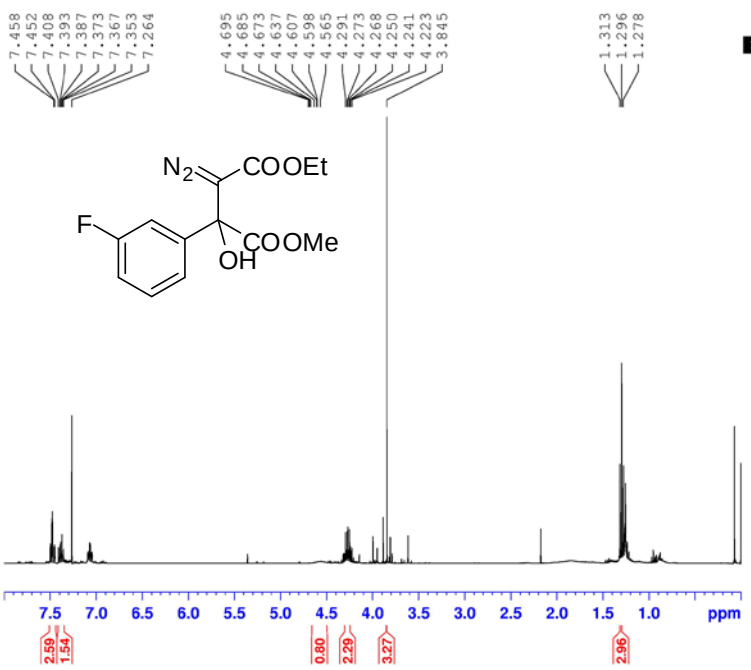

BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090505
Time 23.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
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 -0.30 Hz
GB 0
PC 1.40

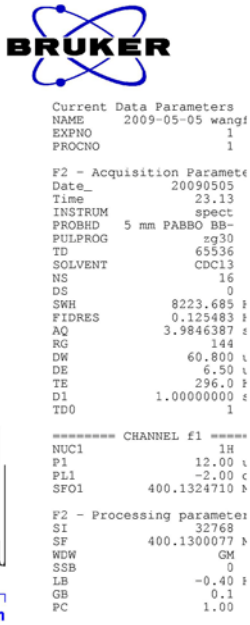

7.578
7.557
7.263
7.214
7.194
4.482
3.821
3.805
2.350
N2
COOMe
COOMe
OH
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.29
1.87
0.80
3.05
3.15
3.45

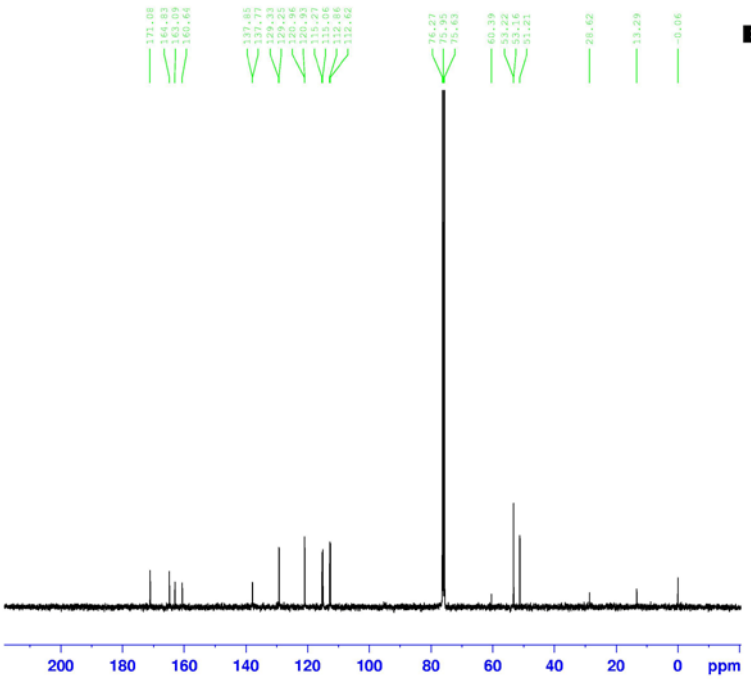

BRUKER
Current Data Parameters
NAME 2009-05-20 wangfei-D-2'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time 21.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.1 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 400.1300082 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00

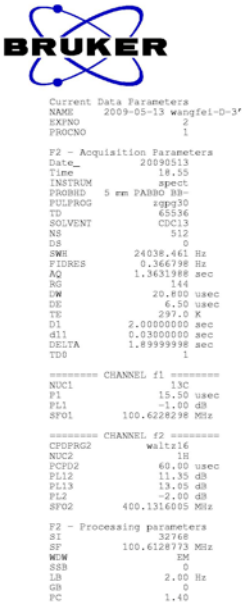

171.77
165.18
138.08
132.25
128.67
128.46
127.67
125.08
76.32
76.00
75.68
60.29
53.02
51.16
28.68
20.06
13.37
-0.00
200 180 160 140 120 100 80 60 40 20 0 ppm

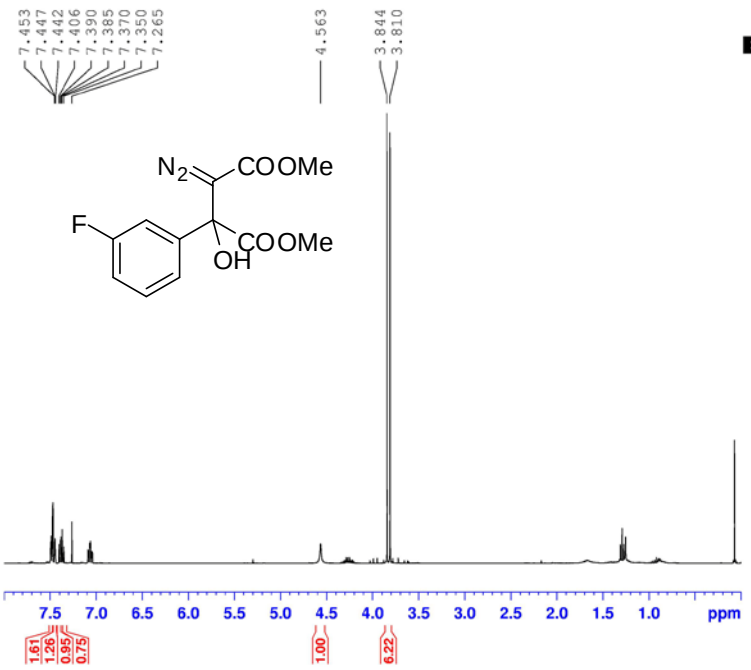

BRUKER
Current Data Parameters
NAME 2009-05-20 wangfei-D-2'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time 22.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.9 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 100.6128721 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


7.458
7.452
7.408
7.393
7.387
7.373
7.367
7.353
7.264
4.695
4.685
4.673
4.637
4.607
4.598
4.565
4.291
4.273
4.268
4.250
4.241
4.223
3.845
1.313
1.296
1.278
N2
COOEt
F
COOMe
OH
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.59
1.54
0.80
2.29
3.27
2.96


BRUKER
Current Data Parameters
NAME 2009-05-05 wangf
EXPNO 1
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090505
Time 23.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685
FIDRES 0.125483
AQ 3.9846387
RG 144
DW 60.800
DE 6.50
TE 296.0
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 12.00
PL1 -2.00
SFO1 400.1324710
F2 - Processing parameter
SI 32768
SF 400.1300077
WDW GM
SSB 0
LB -0.40
GB 0.1
PC 1.00

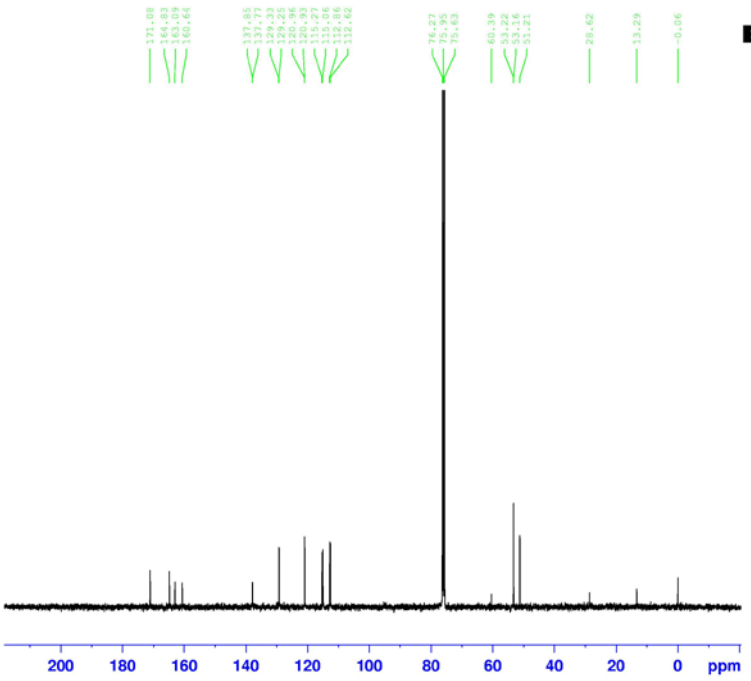

171.08
164.83
163.09
160.64
137.85
137.77
129.23
128.38
120.96
120.93
115.27
115.06
112.96
112.62
76.27
75.95
75.63
60.39
53.22
53.16
51.21
28.62
13.29
-0.06
200 180 160 140 120 100 80 60 40 20 0 ppm


BRUKER
Current Data Parameters
NAME 2009-05-13 wangfei-D-3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 18.55
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 144
DW 20.800 usec
DE 6.50 usec
TE 297.0 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 100.6128773 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40



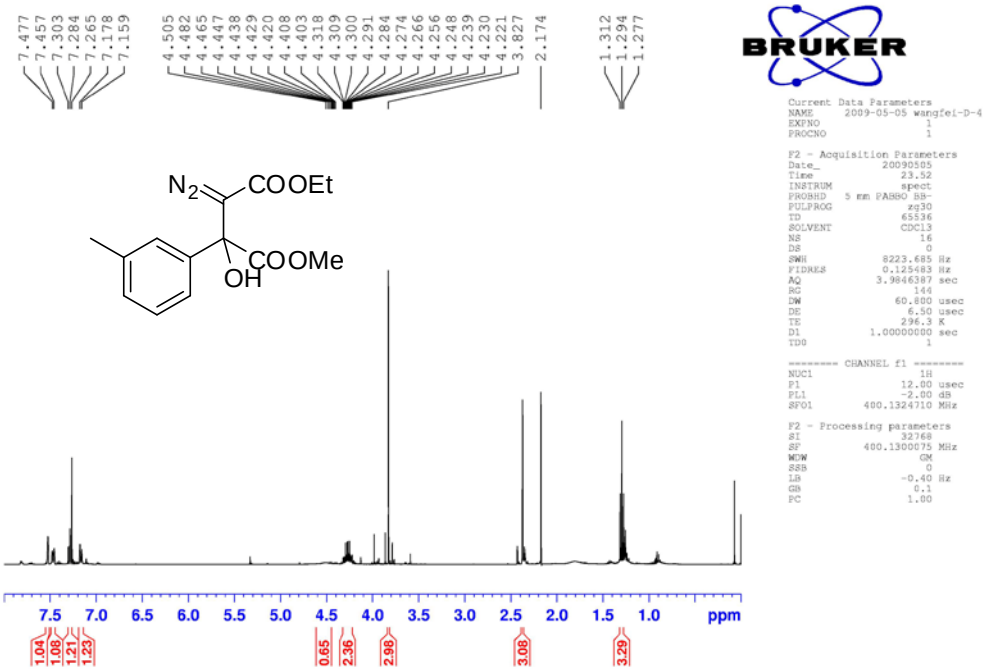
BRUKER

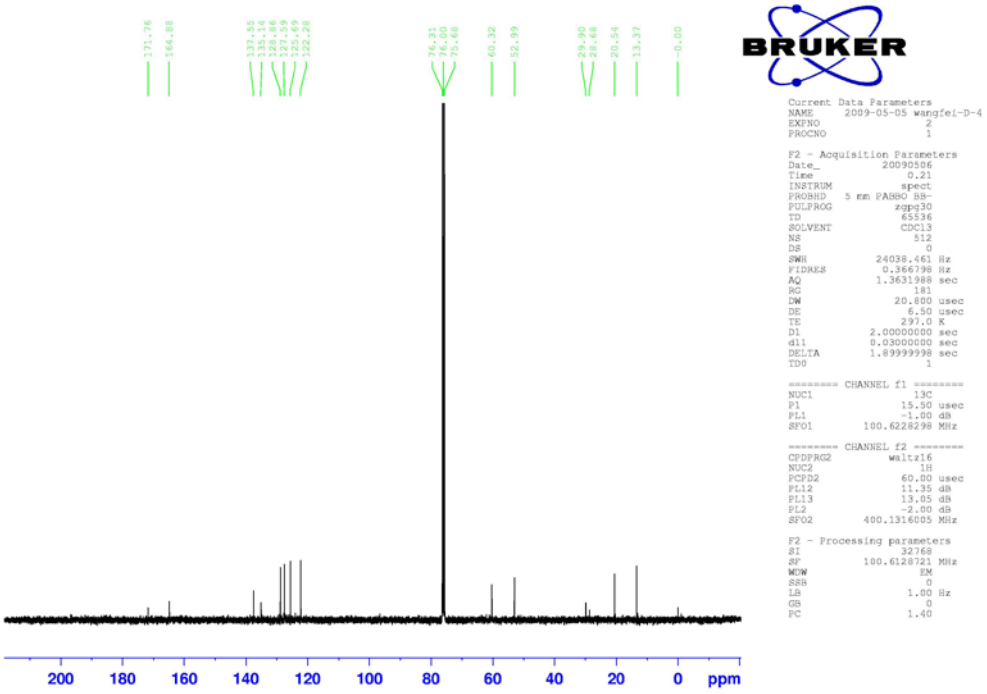
```
Current Data Parameters
NAME      2009-05-13 wangf
EXPNO                   1
PROCNO                  1

F2 - Acquisition Paramete
Date_           20090513
Time               18.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685
FIDRES          0.125483
AQ              3.9846387
RG                  90.5
DW                 60.800
DE                  6.50
TE                296.2 K
D1             1.00000000
TD0                    1

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

F2 - Processing parameter
SI                 32768
SF            400.1300078
WDW                   GM
SSB                    0
LB                 -0.40
GB                   0.3
PC                  1.00
```

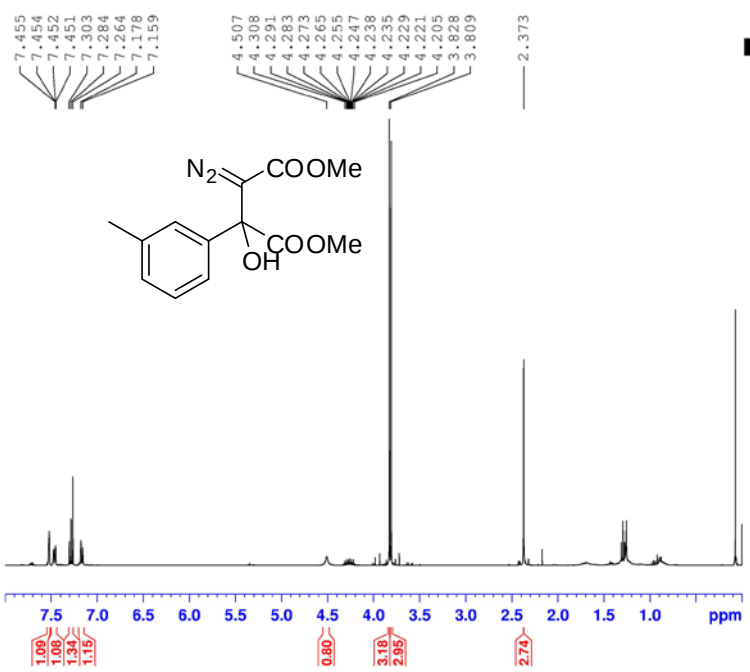


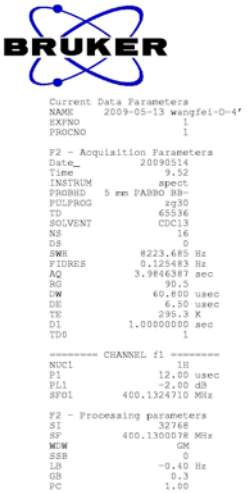
BRUKER

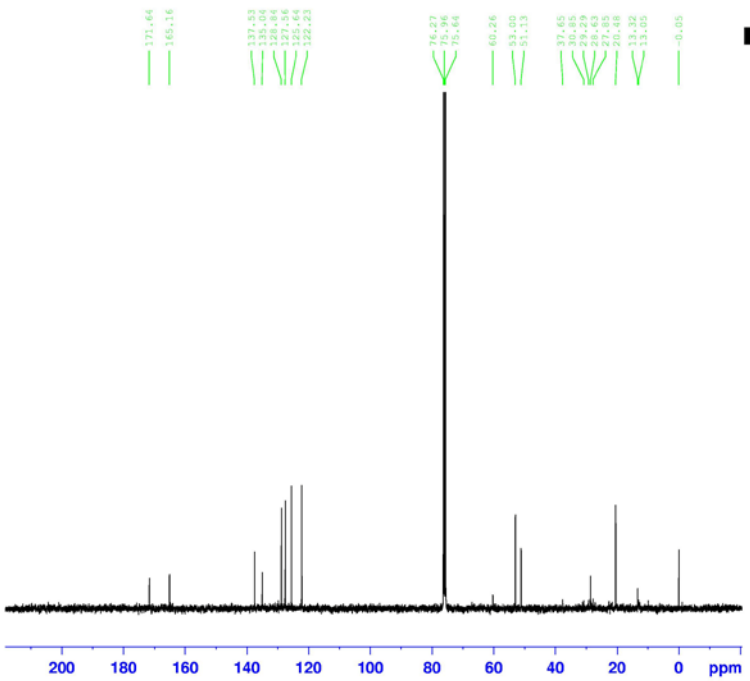
```
Current Data Parameters
NAME      2009-05-13 wangfei-D-3
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20090513
Time               18.55
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                   144
DW               20.800 usec
DE                6.50 usec
TE               297.0 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          100.6128773 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```

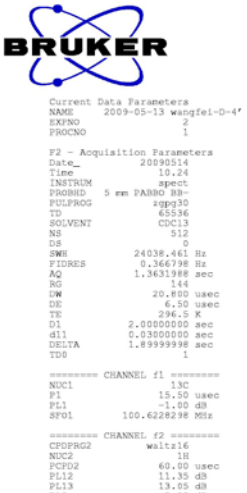

7.477
7.457
7.303
7.284
7.265
7.178
7.159
4.505
4.482
4.465
4.447
4.438
4.429
4.420
4.408
4.403
4.318
4.309
4.300
4.291
4.284
4.274
4.266
4.256
4.248
4.239
4.230
4.221
3.827
2.174
1.312
1.294
1.277
N2
COOEt
COOMe
OH
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090505
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300075 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.1
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.04
1.08
1.21
1.23
0.65
2.36
2.98
3.08
3.29

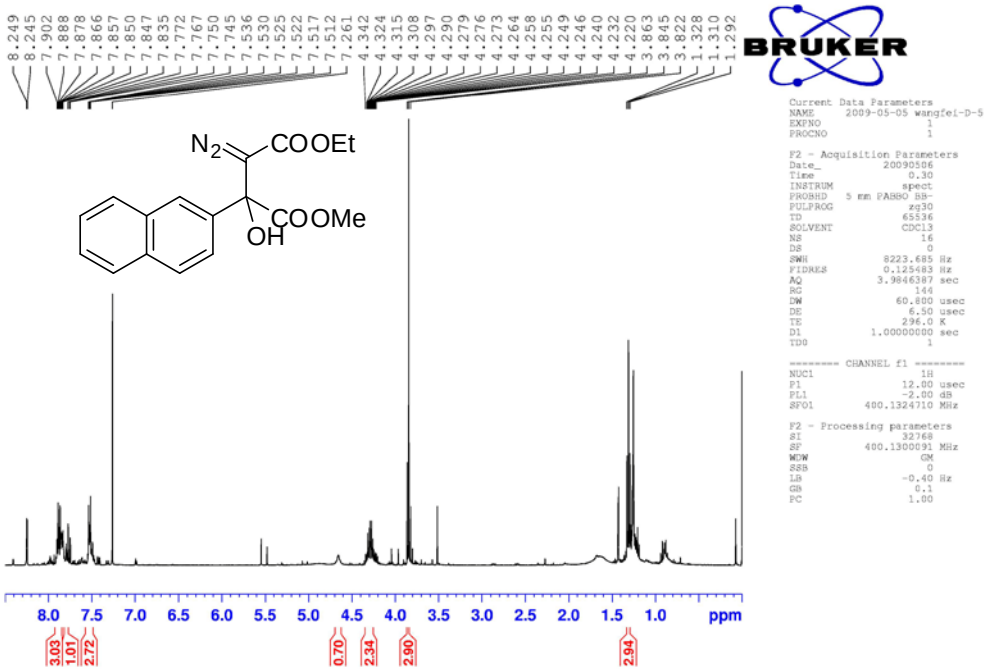

171.76
164.88
137.55
135.14
128.86
127.59
123.69
122.28
76.31
76.00
75.68
60.32
52.99
29.90
28.68
20.54
13.37
-0.00
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-4
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090506
Time 0.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 297.0 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 100.6128721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

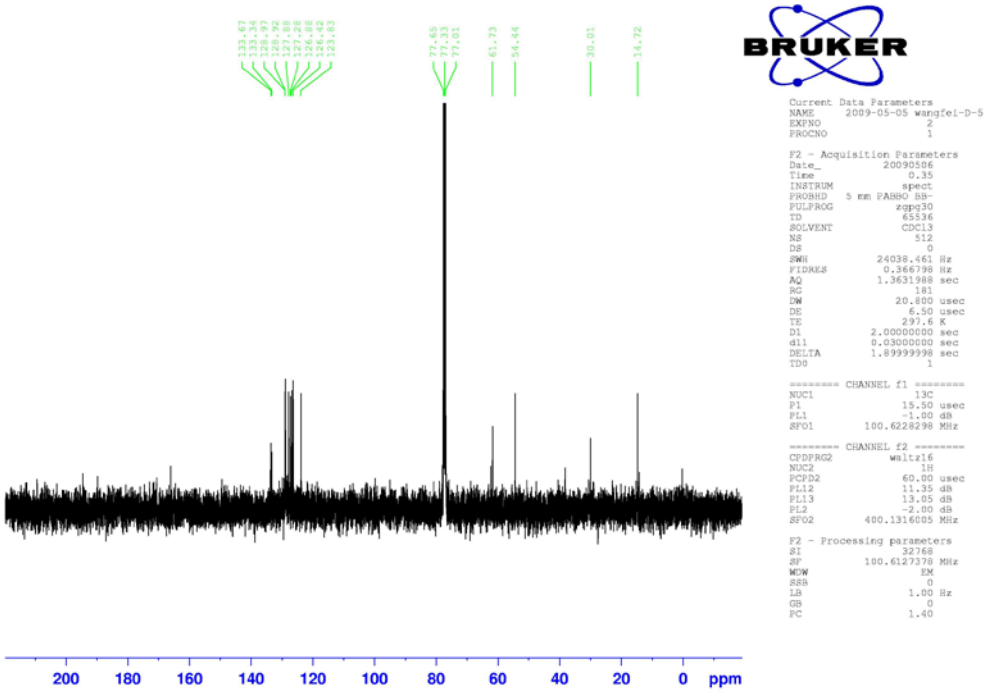

7.455
7.454
7.452
7.451
7.303
7.284
7.264
7.178
7.159
4.507
4.308
4.291
4.283
4.273
4.265
4.255
4.247
4.238
4.235
4.229
4.221
4.205
3.828
3.809
2.373
N2
COOMe
COOMe
OH
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.09
1.08
1.34
1.15
0.80
3.18
2.95
2.74

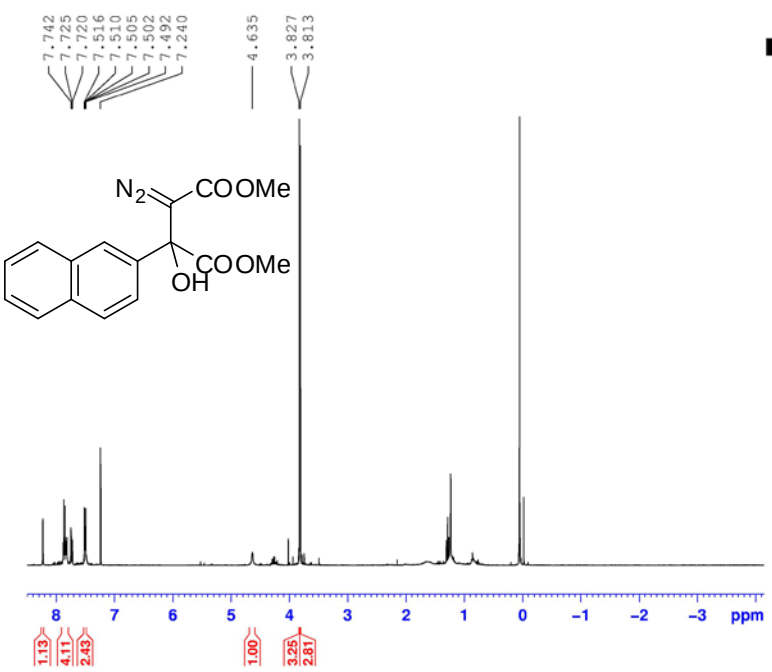

BRUKER
Current Data Parameters
NAME 2009-05-13 wangfei-D-4'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090514
Time 9.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
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 400.1300078 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00


171.64
165.16
137.53
135.04
128.84
127.56
125.64
122.23
76.27
75.96
75.64
60.26
53.00
51.13
37.65
30.85
29.29
28.63
27.85
20.48
13.32
13.05
-0.05
200 180 160 140 120 100 80 60 40 20 0 ppm

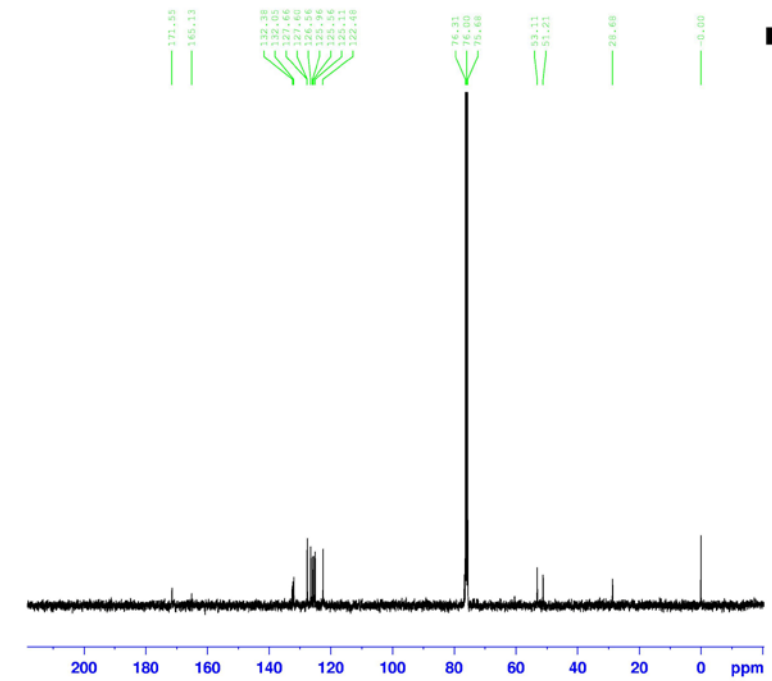

BRUKER
Current Data Parameters
NAME 2009-05-13 wangfei-D-4'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090514
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 144
DW 20.800 usec
DE 6.50 usec
TE 296.5 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 100.6128773 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


8.249
8.245
7.902
7.888
7.878
7.866
7.857
7.850
7.847
7.835
7.772
7.767
7.750
7.745
7.536
7.530
7.525
7.522
7.517
7.512
7.261
4.342
4.324
4.315
4.308
4.297
4.290
4.279
4.276
4.273
4.264
4.258
4.255
4.249
4.246
4.240
4.232
4.220
3.863
3.845
3.822
1.328
1.310
1.292
N2
COOEt
COOMe
OH
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090506
Time 0.30
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 144
DW 60.800 usec
DE 6.50 usec
TE 296.0 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 400.1300091 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.1
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
3.03
1.01
2.72
0.70
2.34
2.90
2.94

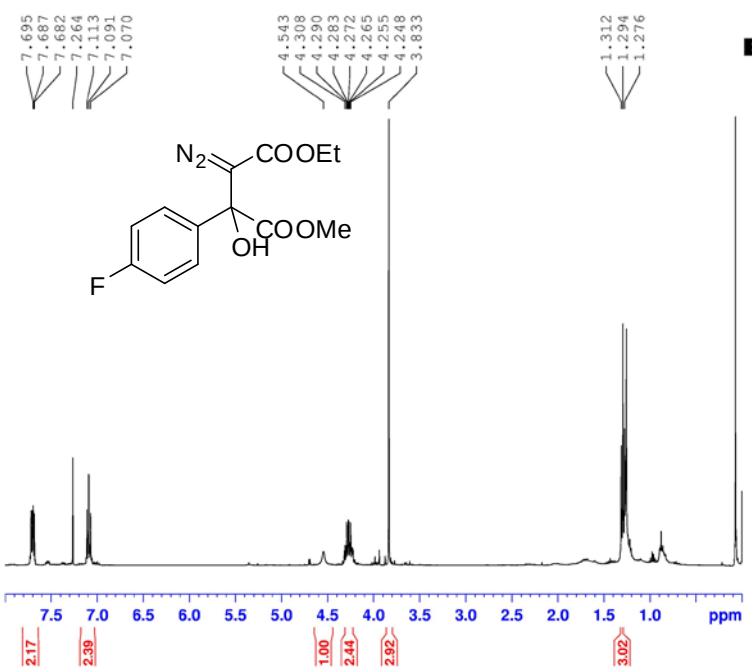

133.67
133.34
128.97
128.92
127.88
127.28
126.88
126.42
123.83
77.65
77.33
77.01
61.73
54.44
30.01
14.72
BRUKER
Current Data Parameters
NAME 2009-05-05 wangfei-D-5
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090506
Time 0.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
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 100.6127370 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


7.742
7.725
7.720
7.516
7.510
7.505
7.502
7.492
7.240
4.635
3.827
3.813
N2
COOMe
COOMe
OH
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
1.13
4.11
2.43
1.00
3.25
2.81

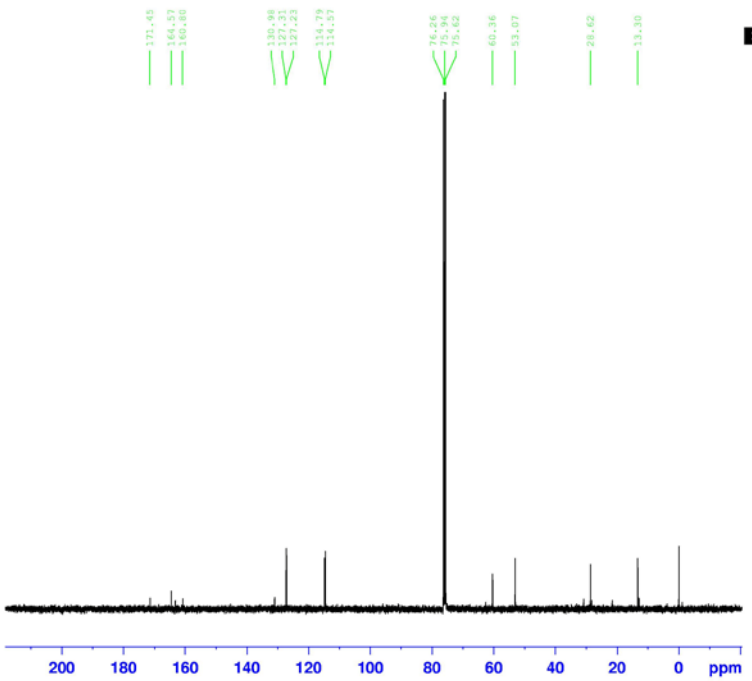

BRUKER
Current Data Parameters
NAME 2009-05-26 wangfei-D-5'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090526
Time 17.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 295.7 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 400.1300176 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00


171.55
165.13
132.38
132.05
127.66
127.60
126.56
125.96
125.56
125.11
122.48
76.31
76.00
75.68
53.11
51.21
28.68
0.00
200
180
160
140
120
100
80
60
40
20
0
ppm

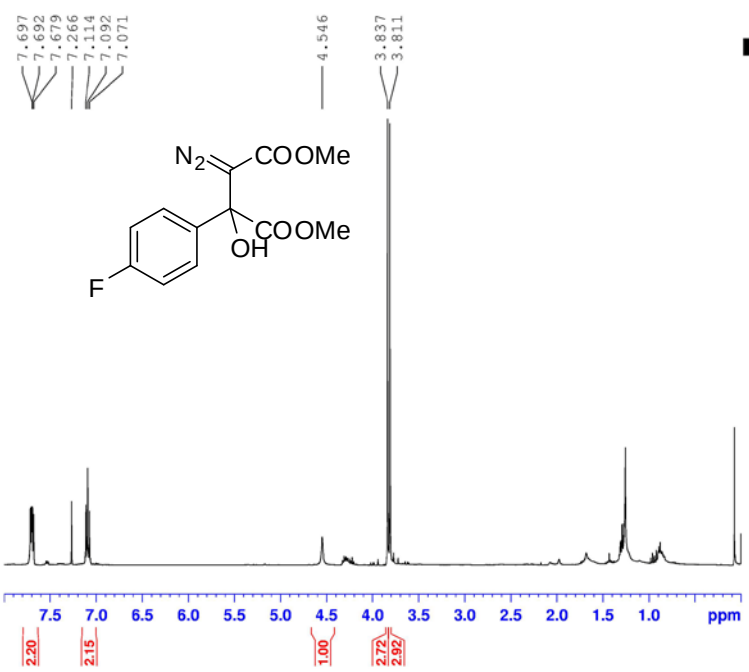

BRUKER
Current Data Parameters
NAME 2009-05-26 wangfei-D-5'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090526
Time 17.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 299.8 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 100.6128696 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


7.695
7.687
7.682
7.264
7.113
7.091
7.070
4.543
4.308
4.290
4.283
4.272
4.265
4.255
4.248
3.833
1.312
1.294
1.276
N2
COOEt
COOMe
OH
F
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.17
2.39
1.00
2.44
2.92
3.02

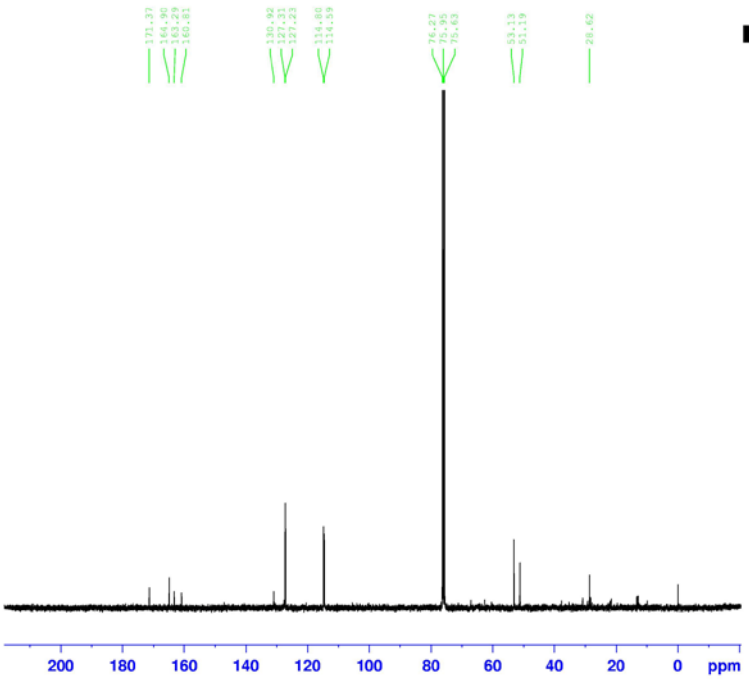

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-6
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 17.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.5 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 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

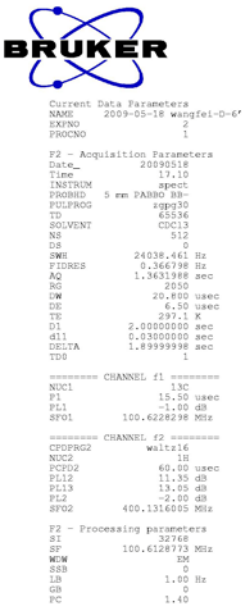

171.45
164.57
160.60
130.98
127.31
121.23
114.79
114.57
76.26
75.94
75.62
60.36
53.07
28.62
13.30
200 180 160 140 120 100 80 60 40 20 0 ppm

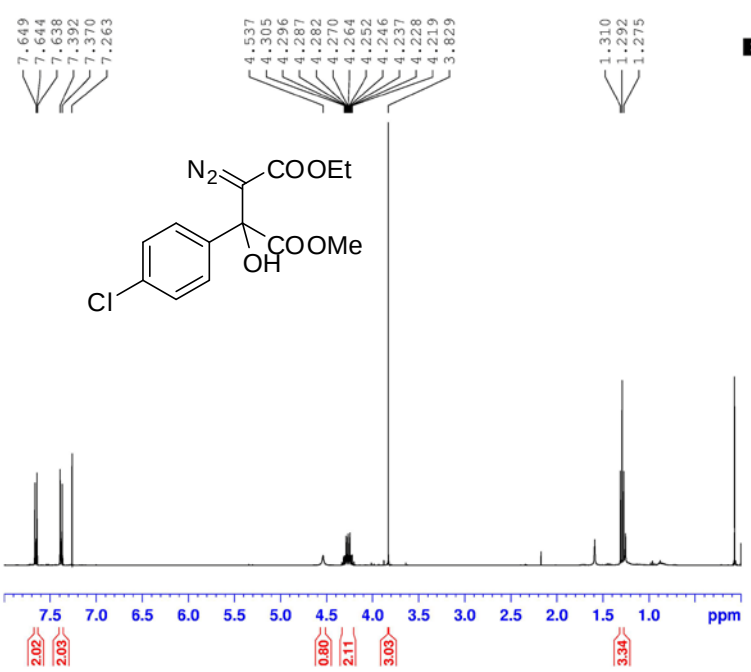

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-6
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.9 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 100.6128773 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

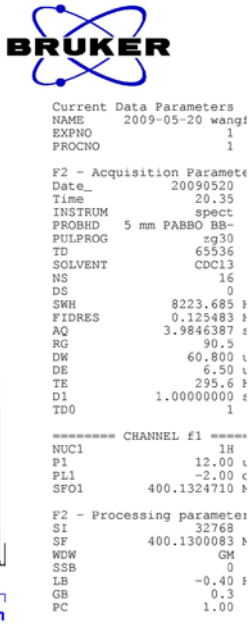

7.697
7.692
7.679
7.266
7.114
7.092
7.071
4.546
3.837
3.811
N2
COOMe
COOMe
OH
F
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.20
2.15
1.00
2.72
2.92

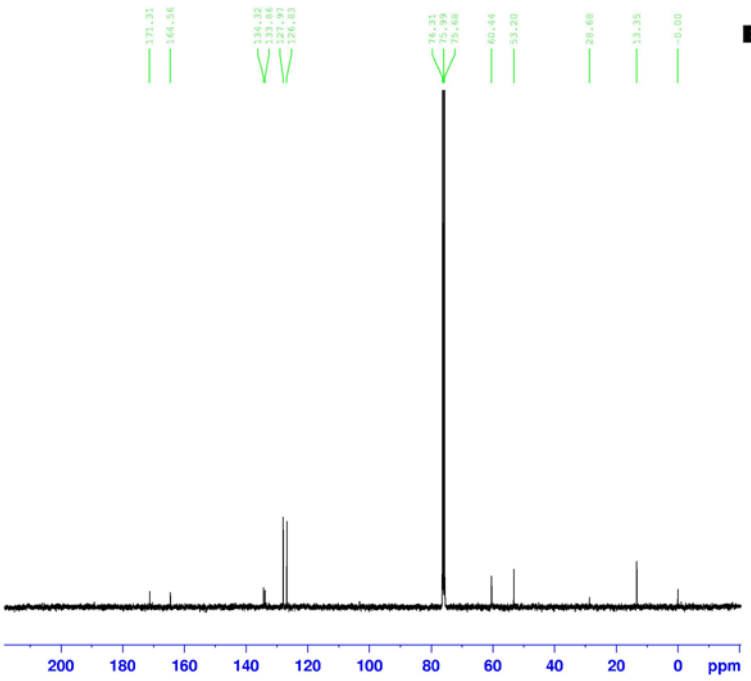

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-6'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 16.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
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 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


171.37
164.90
163.29
160.81
130.92
127.31
121.23
114.80
114.59
76.27
75.95
75.63
53.13
51.19
28.62
200 180 160 140 120 100 80 60 40 20 0 ppm

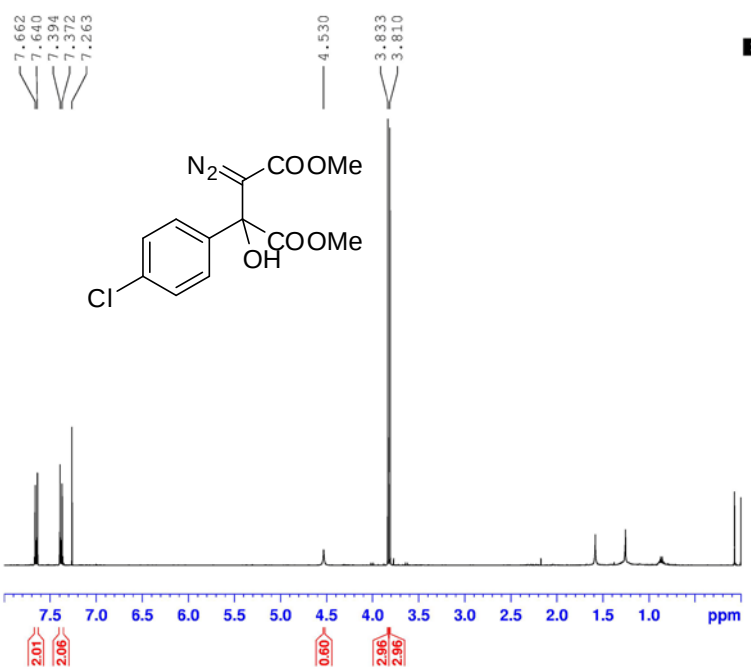

BRUKER
Current Data Parameters
NAME 2009-05-18 wangfei-D-6'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090518
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.1 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 100.6128773 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.649
7.644
7.638
7.392
7.370
7.263
4.537
4.305
4.296
4.287
4.282
4.270
4.264
4.252
4.246
4.237
4.228
4.219
3.829
1.310
1.292
1.275
N2
COOEt
COOMe
OH
Cl
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.02
2.03
0.80
2.11
3.03
3.34

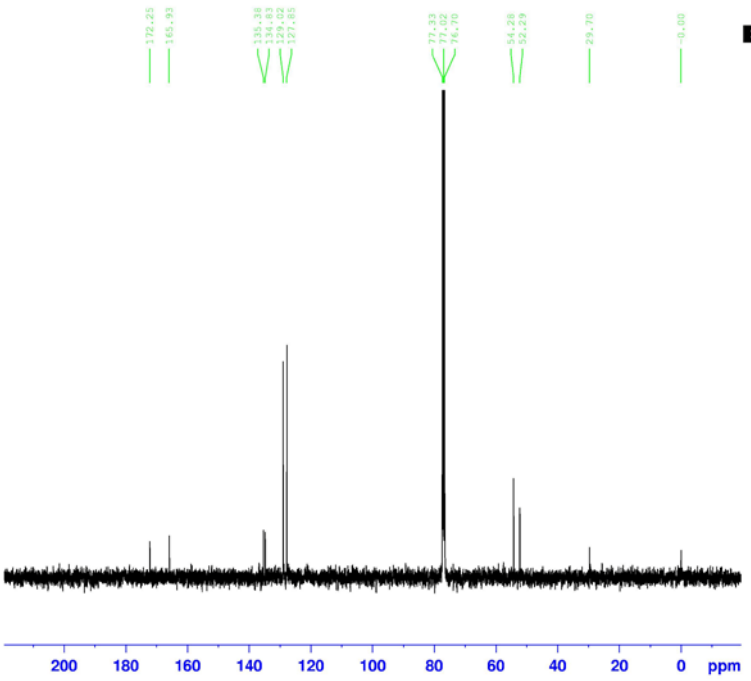

BRUKER
Current Data Parameters
NAME 2009-05-20 wangf
EXPNO 1
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090520
Time 20.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685
FIDRES 0.125483
AQ 3.9846387
RG 90.5
DW 60.800
DE 6.50
TE 295.6
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 12.00
PL1 -2.00
SFO1 400.1324710
F2 - Processing parameter
SI 32768
SF 400.1300083
WDW GM
SSB 0
LB -0.40
GB 0.3
PC 1.00

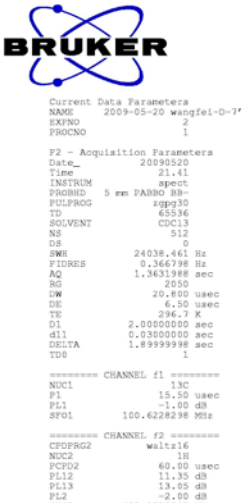

171.31
164.56
134.32
133.46
127.99
126.83
76.31
75.99
75.68
60.44
53.20
28.68
13.35
-0.00
200
180
160
140
120
100
80
60
40
20
0
ppm

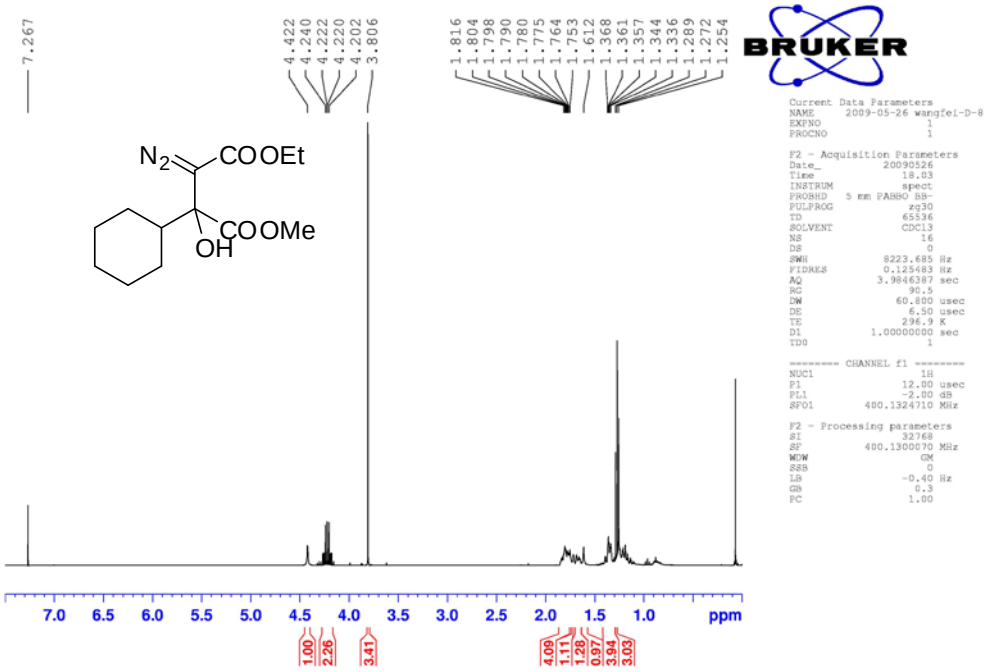

BRUKER
Current Data Parameters
NAME 2009-05-20 wangfei-D-7
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time 21.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.9 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 100.6128720 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

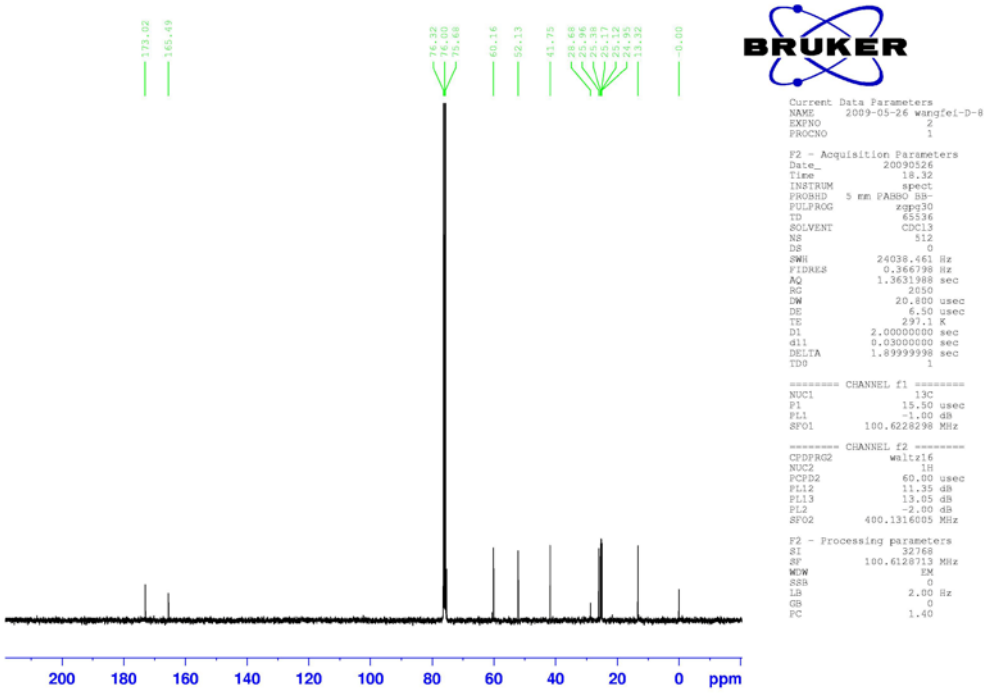

7.662
7.640
7.394
7.372
7.263
4.530
3.833
3.810
N2
COOMe
COOMe
OH
Cl
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.01
2.06
0.60
2.96
2.96

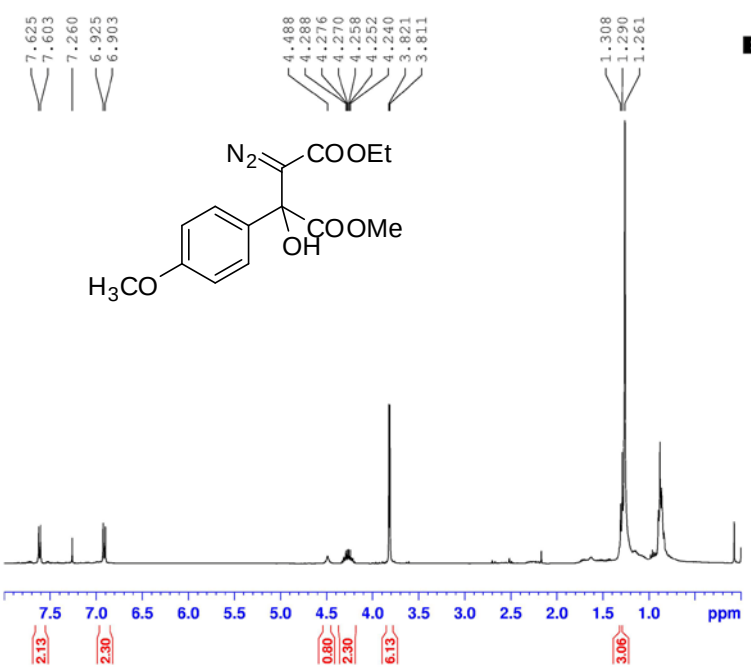

BRUKER
Current Data Parameters
NAME 2009-05-20 wangfei-D-7'
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time 21.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.1 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 400.1300082 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00

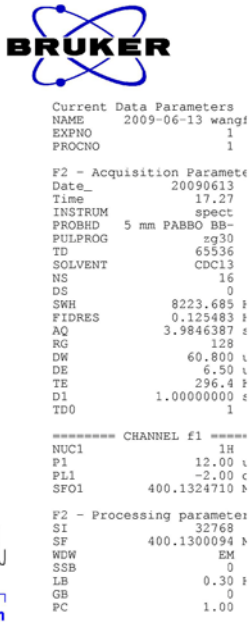

172.25
165.93
135.38
134.83
129.02
127.85
77.33
77.02
76.70
54.28
52.29
29.70
-0.00
200 180 160 140 120 100 80 60 40 20 0 ppm

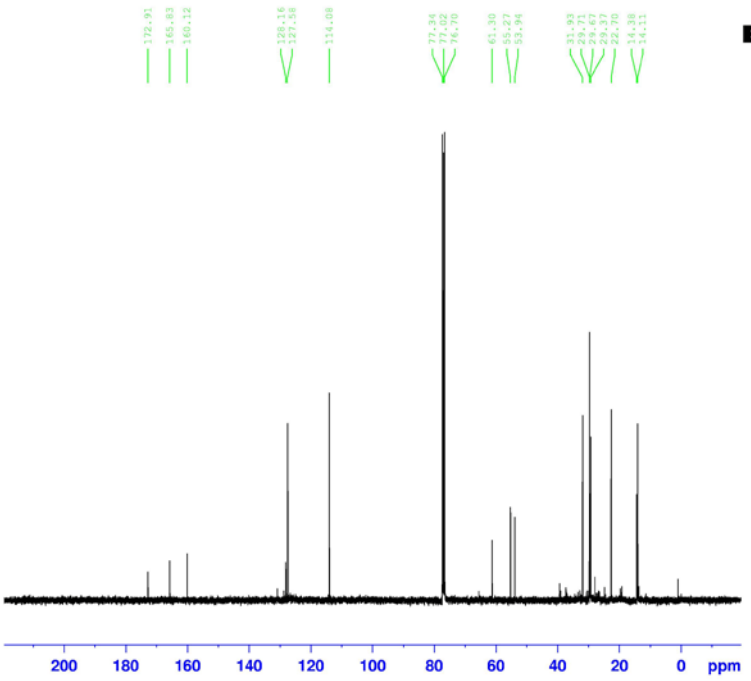

BRUKER
Current Data Parameters
NAME 2009-05-20 wangfei-D-7'
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time 21.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.7 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 100.6127688 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

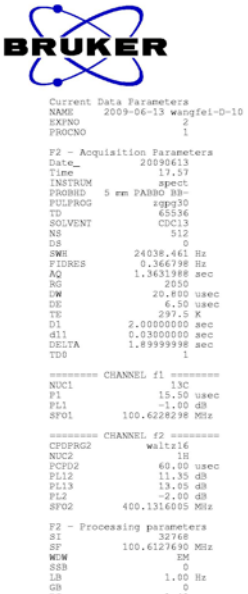

7.267
4.422
4.240
4.222
4.220
4.202
3.806
1.816
1.804
1.798
1.790
1.780
1.775
1.764
1.753
1.612
1.368
1.361
1.357
1.344
1.336
1.289
1.272
1.254
N2
COOEt
COOMe
OH
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
2.26
3.41
4.09
1.11
1.28
0.97
3.94
3.03
BRUKER
Current Data Parameters
NAME 2009-05-26 wangfei-D-8
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090526
Time 18.03
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 90.5
DW 60.800 usec
DE 6.50 usec
TE 296.9 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 400.1300070 MHz
WDW GM
SSB 0
LB -0.40 Hz
GB 0.3
PC 1.00

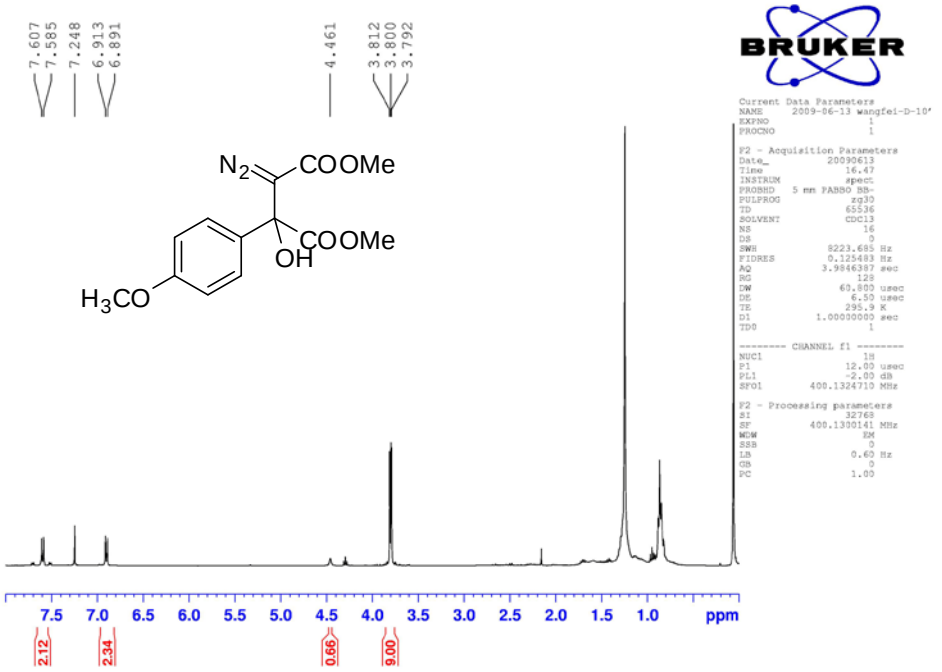

173.02
165.49
76.32
76.00
75.68
60.16
52.13
41.75
28.68
25.96
25.38
25.17
25.12
24.95
13.32
-0.00
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME 2009-05-26 wangfei-D-8
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090526
Time 18.32
INSTRUM spect
PROBHD 5 mm PABBO 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 297.1 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 100.6128713 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

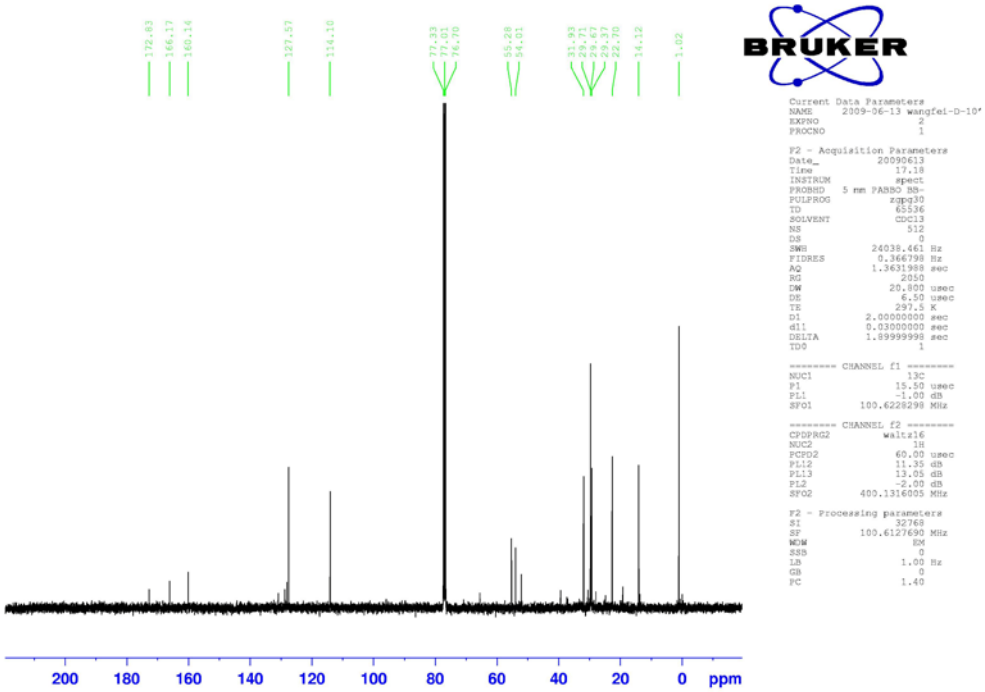

7.625
7.603
7.260
6.925
6.903
4.488
4.288
4.276
4.270
4.258
4.252
4.240
3.821
3.811
1.308
1.290
1.261
N2
COOEt
COOMe
OH
H3CO
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.13
2.30
0.80
2.30
6.13
3.06

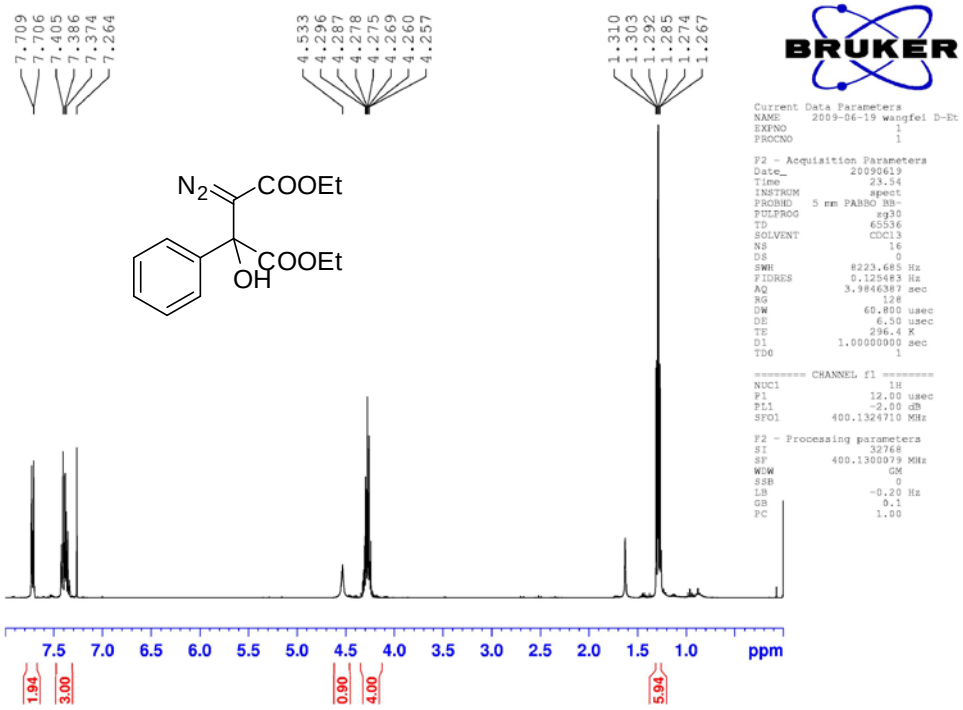

BRUKER
Current Data Parameters
NAME 2009-06-13 wangf
EXPNO 1
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090613
Time 17.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685
FIDRES 0.125483
AQ 3.9846387
RG 128
DW 60.800
DE 6.50
TE 296.4
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 12.00
PL1 -2.00
SFO1 400.1324710
F2 - Processing parameter
SI 32768
SF 400.1300094
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

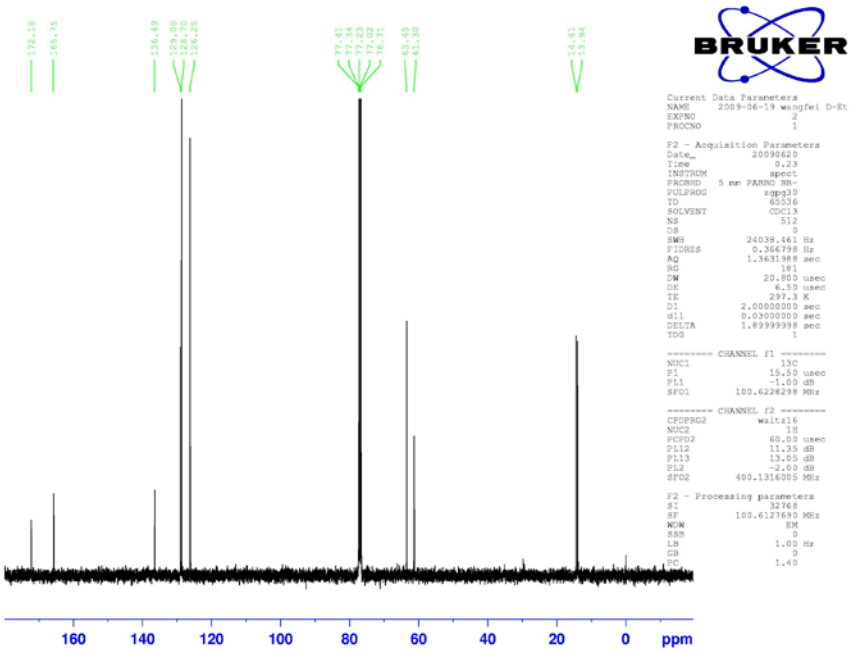

172.91
165.83
160.12
128.16
127.58
114.08
77.34
77.02
76.70
61.30
55.27
53.94
31.93
29.71
29.57
29.33
22.70
14.38
14.11
200 180 160 140 120 100 80 60 40 20 0 ppm


BRUKER
Current Data Parameters
NAME 2009-06-13 wangfei-D-10
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090613
Time 17.57
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 297.5 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


7.607
7.585
7.248
6.913
6.891
4.461
3.812
3.800
3.792
N2
COOMe
COOMe
OH
H3CO
BRUKER
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.12
2.34
0.66
9.00


172.63
166.17
160.14
127.57
114.10
77.33
77.01
76.70
65.28
54.01
31.93
29.71
29.57
29.33
22.70
14.12
1.02
BRUKER
200 180 160 140 120 100 80 60 40 20 0 ppm


7.709
7.706
7.405
7.386
7.374
7.264
4.533
4.296
4.287
4.278
4.275
4.269
4.260
4.257
1.310
1.303
1.292
1.285
1.274
1.267
N2
COOEt
COOEt
OH
BRUKER
Current Data Parameters
NAME 2009-06-19 wangfei D-Et
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090619
Time 23.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 296.4 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 400.1300079 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.1
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.94
3.00
0.90
4.00
5.94


172.18
165.75
136.49
129.88
128.70
126.25
77.41
77.34
77.23
77.02
76.71
63.45
61.30
14.41
13.94
BRUKER
Current Data Parameters
NAME 2009-06-19 wangfei D-Et
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090620
Time 0.23
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 161
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
160 140 120 100 80 60 40 20 0 ppm

6. References

1. M. Rambaud, M. Balasse, G. Duguay and J. Villieras, *Synthesis* **1998**, 564.

2. J. S. Nimitz and H. S. Mosher, *J. Org. Chem.* **1981**, *46*, 211.

3. Z. Yu, X. Liu, Z. Dong, M. Xie and X. Feng, *Angew. Chem. Int. Ed.* **2008**, *47*, 1308.

4. H. Moorlag, R. Kellog, Tetrahedron: Asymmetry, **1991**, 2, 705.