Supporting Information

for

Direct asymmetric aldol addition/isomerization of α,β-unsaturated γ-butyrolactam with aryl α-ketoesters: synthesis of MBH-type products

*Jinlong Zhang, Xihong Liu, Xiaojuan Ma and Rui Wang**

Key laboratory of preclinical Study for New Drugs of Gansu Province, School of Basic Medical Sciences, State Key Laboratory of Applied Organic Chemistry, and Institute of Biochemistry and Molecular Biology, Lanzhou University, Lanzhou,730000, P. R. China.

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General information

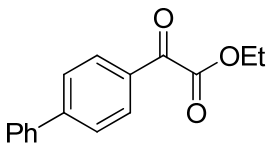
All commercially available reagents were used without further purification unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC), column chromatography purifications were carried out using silica gel. Proton nuclear resonance (^{1}H NMR) spectra were recorded on 300 MHz spectrometer in $CDCl_3$ and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on 75 MHz spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as internal standard. Methanedichloride was freshly distilled from CaH_2 before use; other solvents (THF, Et_2O and toluen) were freshly distilled from sodium. Racemates were obtained by reactions using amounts of thiourea. Catalysts **1a-1c**,[1] **1d**[2] and **1f-1h**[3] were prepared according to the literature, α,β-unsaturated γ-butyrolactam **2a** was synthesized according to the reported procedure.[4]

General procedure for preparation of α-ketoesters [5]

A solution of aryl bromide (24 mmol, 1.2 equiv.) in anhydrous THF was added to Mg turnings (28 mmol, 1.4 equiv.) and external heating under argon atmosphere. The reaction mixture was refluxed gently for 30-40 min. and then it was cooled to room temperature, before conveying it to a dropping funnel. The Grignard reagent was added dropwise to a solution of diethyl oxalate (20 mmol, 1.0 equiv.) in THF at -78°C. The reaction mixture was warmed to 10°C within 1 hour and then was quenched by the addition of 10% HCl solution. The organic layer was separated, the aqueous layer was extracted with diethyl ether and the combined organic layer was washed with brine, dried over $MgSO_4$ and evaporated. The residue was purified by flash column chromatography on silica gel to give pure aryl α-ketoesters in 55-65% yields.

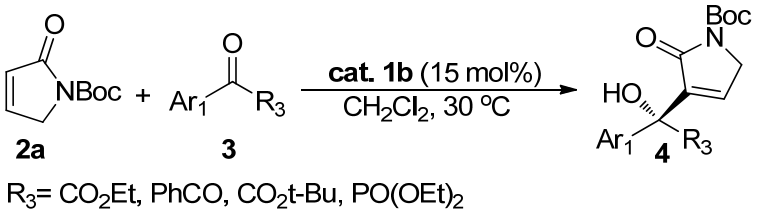
β,γ-Unsaturated a-ketoester **3q** was prepared according to the literature.[6]

Ethyl 4-phenyl-phenylglyoxylate



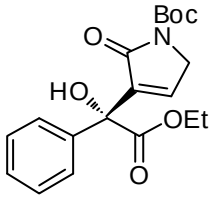
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.5 Hz, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 7.68 – 7.63 (m, 2H), 7.57 – 7.38 (m, 3H), 4.48 (q, *J* = 7.1 Hz, 2H), 1.45 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.88, 163.77, 147.53, 139.37, 131.11, 130.62, 129.00, 128.61, 127.45, 127.30, 62.34, 14.09.

General procedure for the direct asymmetric aldol addition/isomerization of α,β-unsaturated γ-butyrolactam with aryl α-ketoesters



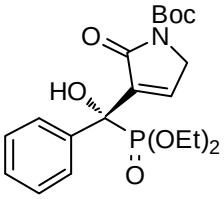
To a solution of aryl a-ketoesters **3** (0.3mmol,1.5 equiv) in CH_2Cl_2 (0.4 mL) was added catalyst **1b** (0.03 mmol, 0.15 equiv) followed by α,β-unsaturated γ-butyrolactam **2a** (0.2 mmol, 1.0 equiv). The reaction mixture was stirred at 30^{o}C until the consumption of **2a**, the progress of which was monitored by TLC analysis (30 h).The solvent was then removed under vacuum. The residue was purified by silica gel chromatography (hexane/ AcOEt= 3/1 to 2/1 as eluent) to afford the desired addition product **4**.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-phenylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4a)



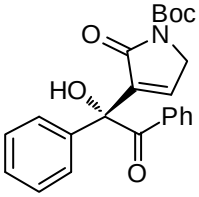
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 11.9 min, t_{minor} = 17.2 min, 99% ee). oily liquid, 78% yield; $[\alpha]_D^{20}$ = -3.39 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 – 7.62 (m, 2H), 7.43 – 7.32 (m, 3H), 6.70 (t, *J* = 2.0 Hz, 1H), 4.60 (s, 1H), 4.41 – 4.15 (m, 4H), 1.55 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.96, 167.59, 149.40, 141.27, 141.10, 137.66, 128.43, 128.30, 126.06, 83.41, 75.36, 62.95, 49.59, 28.04, 13.96 cm^{-1}; HRMS calcd for $C_{19}H_{23}NNaO_6$ $(M+H)^+$ 384.1425, found 384.1418.

tert-Butyl 3-((diethoxyphosphoryl)(hydroxy)(phenyl)methyl) -2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4b)



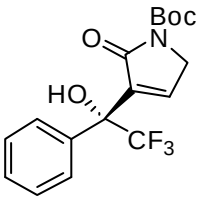
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 19.5 min, t_{minor} = 28.9 min, 0% ee). oily liquid, 45% yield; $[\alpha]_D^{20}$ = 0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69-7.66 (m, 3H), 7.40 – 7.27 (m, 3H), 6.02 (d, *J* = 21.4 Hz, 1H), 4.37 (dd, *J* = 4.0, 2.0 Hz, 2H), 4.32 – 4.13 (m, 2H), 3.94 – 3.80 (m, 1H), 3.73 – 3.54 (m, 1H), 1.53 (s, 9H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.06 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.30, 169.13, 149.02, 141.15, 141.09, 138.21, 135.90, 131.12, 128.85, 128.21, 128.17, 128.08, 128.04, 126.41, 126.35, 83.78, 74.90, 64.42, 64.32, 64.25, 64.14, 49.91, 27.97, 16.52, 16.45, 16.24, 16.17cm^{-1}; ^{31}P NMR (121 MHz, $CDCl_3$) δ 16.93; HRMS calcd for $C_{20}H_{28}NO_7P$ $(M+H)^+$ 426.1684, found 426.1676.

tert-Butyl 3-(1-hydroxy-2-oxo-1,2-diphenylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4c)



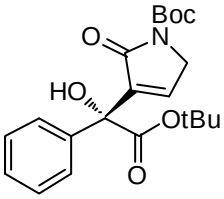
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 15.8 min, t_{minor} = 17.5 min, 0% ee). oily liquid, 53% yield; $[\alpha]_D^{20}$ = 0 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 – 7.89 (m, 2H), 7.60 – 7.52 (m, 2H), 7.47 – 7.24 (m, 4H), 6.47 (t, *J* = 1.9 Hz, 2H), 5.58 (s, 1H), δ 4.38 (dd, *J* = 20.2, 2.0 Hz, 1H), 4.19 (dd, *J* = 20.2, 2.0 Hz, 1H), 1.56 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.38, 169.31, 149.26, 141.82, 140.83, 137.64, 134.07, 132.80, 130.89, 128.83, 128.36, 127.87, 125.44, 83.63, 81.90, 50.00, 28.00cm^{-1}; HRMS calcd for $C_{19}H_{23}NO_6$ $(M+H)^+$ 394.1668, found 394.1649.

(R)-_tert_-butyl 2-oxo-3-(2,2,2-trifluoro-1-hydroxy-1-phenylethyl)-2,5-dihydro-1H-pyrrole-1-carboxylate (4d)



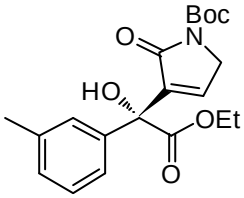
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 90/10, 1.0 ml/min, t_{major} = 7.8 min, t_{minor} = 8.2 min, 0% ee). white solid, 81% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 – 7.52 (m, 2H), 7.43 – 7.34 (m, 3H), 7.31 – 7.17 (m, 1H), 5.99 (s, 1H), 4.41 (d, *J* = 2.0 Hz, 2H), 1.54 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.60, 148.82, 140.85, 135.88, 134.34, 129.15, 128.40, 126.68, 84.13, 49.88, 27.92cm^{-1}; HRMS calcd for $C_{17}H_{18}F_3NNaO_4$ $(M+H)^+$ 380.1088, found 380.1080.

(R)-*tert*-Butyl 3-(2-tert-butoxy-1-hydroxy-2-oxo-1-phenylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4e)



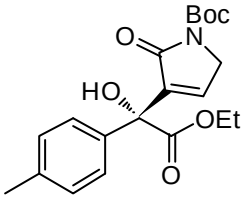
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 100/0.1, 0.5 ml/min, t_{major} = 4.0 min, t_{minor} = 6.1 min, 77% ee). oily liquid, 88% yield; $[\alpha]_D^{20}$ = -0.202 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 – 7.62 (m, 2H), 7.42 – 7.27 (m, 3H), 6.62 (t, *J* = 2.1 Hz, 2H), 4.49 (s, 1H), 4.23 (qd, *J* = 20.1, 2.1 Hz, 2H), 1.55 (s, 9H), 1.48 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.90, 167.21, 149.46, 141.73, 140.84, 138.16, 128.20, 128.14, 126.11, 84.03, 83.12, 75.34, 49.42, 28.03, 27.74cm^{-1}; HRMS calcd for $C_{21}H_{27}NO_6$ $(M+H)^+$ 390.1926, found 390.1911.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-m-tolylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4f)



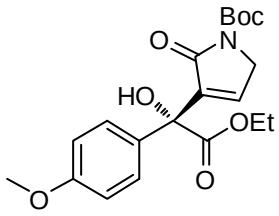
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 11.4 min, t_{minor} = 15.1 min, 99% ee). oily liquid, 79% yield; $[\alpha]_D^{20}$ = -1.87 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (s, 1H), 7.41 – 7.38 (m 1H), 7.30 – 7.25 (m, 1H), 7.72 – 7.71 (m, 1H), 6.71 (t, *J* = 2.0 Hz, 1H), 4.59 (s, 1H), 4.40 – 4.13 (m, 4H), 2.37 (s, 3H), 1.55 (s, 9H), 1.28 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.95, 167.64, 149.35, 141.17, 141.10, 138.02, 137.48, 129.09, 128.08, 126.49, 123.10, 83.32, 75.32, 62.81, 49.55, 27.98, 21.53, 13.91cm^{-1}; HRMS calcd for $C_{20}H_{25}NO_6$ $(M+H)^+$ 376.1758, found 376.1755.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-p-tolylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4g)



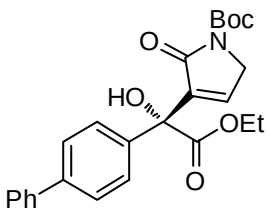
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 9.6 min, t_{minor} = 23.2 min, 99% ee). white solid, 75% yield; $[\alpha]_D^{20}$ = -2.178 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 – 7.50 (m, 2H), 7.21 – 7.18 (m, 2H), 6.71 (t, *J* = 2.0 Hz, 1H), 4.56 (s, 1H), 4.38 – 4.11 (m, 4H), 2.36 (s, 3H), 1.55 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.07, 167.61, 149.41, 141.34, 141.05, 138.19, 134.71, 128.98, 125.94, 83.35, 75.24, 62.85, 49.56, 28.02, 21.05, 13.96cm^{-1}; HRMS calcd for $C_{20}H_{25}NO_6$ $(M+H)^+$ 376.1767 , found 376.1755.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-1(4-methoxyphenyl)-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4h)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 9.3 min, t_{minor} = 10.9 min, 98% ee). oily liquid, 79% yield; $[\alpha]_D^{20}$ = -0.187 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 – 7.53 (m, 2H), 6.93 – 6.90(m, 2H), 6.72 (t, *J* = 2.0 Hz, 1H), 4.56 (s, 1H), 4.40 – 4.14 (m, 4H), 3.82 (s, 3H), 1.55 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.08, 167.57, 159.51, 149.34, 145.12, 141.42, 140.98, 129.61, 127.82, 127.30, 113.54, 83.29, 74.95, 62.79, 55.21, 49.52, 27.97, 13.92cm^{-1}; HRMS calcd for $C_{20}H_{25}NO_7$ $(M+H)^+$ 392.1715, found 392.1704.

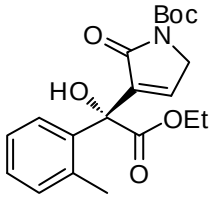
(R)-*tert*-Butyl 3-(1-(biphenyl-4-yl)-2-ethoxy-1-hydroxy-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4i)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{minor} = 10.1 min, t_{major} =

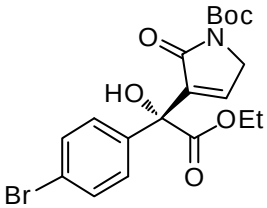
13.9 min, 99% ee). oily liquid, 88% yield; $[\alpha]_D^{20}$ = -2.952 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 – 7.57 (m, 2H), 7.67 – 7.56 (m, 4H), 7.34 – 7.26 (m, 2H), 7.40 – 7.33 (m, 1H), 6.79 (t, *J* = 1.9 Hz, 1H), 4.64 (s, 1H), 4.42 – 4.18 (m, 4H), 1.56 (s, 9H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.91, 167.58, 149.38, 141.24, 141.15, 140.31, 136.65, 128.80, 127.52, 127.05, 126.98, 126.53, 83.40, 75.27, 63.00, 49.61, 28.02, 13.98cm^{-1}; HRMS calcd for $C_{25}H_{27}NO_6$ $(M+H)^+$ 438.1929, found 438.1911.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-o-tolylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4j)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 7.0 min, t_{minor} = 10.4 min, 99% ee). oily liquid, 51% yield; $[\alpha]_D^{20}$ = -1.87 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.14 (m, 4H), 6.67 (t, *J* = 2.0 Hz, 1H), 5.02 (s, 1H), 4.39 – 4.31 (m, 4H), 2.38 (s, 3H), 1.57 (s, 9H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.86, 168.27, 149.30, 141.08, 138.72, 137.08, 136.32, 132.49, 128.54, 126.73, 125.70, 83.60, 78.54, 62.60, 49.62, 28.02, 21.38, 14.03cm^{-1}; HRMS calcd for $C_{20}H_{25}NO_6$ $(M+H)^+$ 376.1760, found 376.1755.

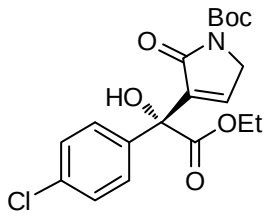
(R)-*tert*-Butyl 3-(1-(4-bromophenyl)-2-ethoxy-1-hydroxy-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4k)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 100/0.5, 1.0 ml/min, t_{major} = 13.5 min, t_{minor} = 17.2 min, 92% ee). oily liquid, 77% yield; $[\alpha]_D^{20}$ = -1.52 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 – 7.35 (m, 4H), 6.71 (t, *J* = 2.0 Hz, 1H), 4.61 (s, 1H), 4.44 – 4.10 (m, 4H), 1.55 (s, 9H), 1.28 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.51, 167.37, 149.24, 141.02, 140.82, 136.76, 131.40, 127.93, 122.71, 83.46, 74.97, 63.13, 49.59, 27.98, 13.91cm^{-1}; HRMS calcd for $C_{19}H_{22}BrNO_6$ $(M+H)^+$462.0545, found 462.0523.

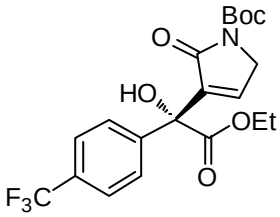
(R)-*tert*-Butyl 3-(1-(4-chlorophenyl)-2-ethoxy-1-hydroxy-2-oxoethyl)-2-oxo-2,5-dihydro-1H-

pyrrole-1-carboxlate (4l)



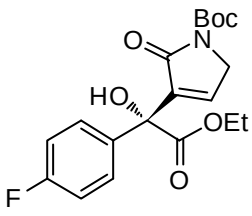
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 7.3 min, t_{minor} = 22.4 min, 99% ee). oily liquid, 75% yield; $[\alpha]_D^{20}$ = -2.581 (*c* = 1.0, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.54 – 7.51 (m, 2H); 7.40 – 7.34 (m, 2H), 6.71 (t, *J* = 2.0 Hz, 1H), 4.63 (s, 1H), 4.39 – 4.15 (m, 4H), 1.55 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.58, 167.38, 149.23, 141.01, 140.88, 136.20, 134.43, 128.42, 127.59, 83.45, 74.91, 63.10, 49.58, 27.96, 13.89cm^{-1}; HRMS calcd for $C_{19}H_{22}ClNO_6$ $(M+H)^+$ 396.1228, found 396.1208.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-(4-(trifluoromethyl)phenyl)ethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4m)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 100/0.1, 0.5 ml/min, t_{major} = 21.6 min, t_{minor} = 23.1 min, 99% ee). oily liquid, 71% yield; $[\alpha]_D^{20}$ = -4.236 (*c* = 1.0, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.82 – 7.80 (m, 2H), 7.68 – 7.66 (m, 2H), 6.71 (t, *J* = 1.9 Hz, 1H), 4.70 (s, 1H), 4.43 – 4.19 (m, 4H), 1.56 (s, 9H), 1.30 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.35, 167.35, 149.25, 141.67, 141.05, 140.72, 126.68, 125.30, 125.25, 83.60, 75.17, 63.33, 49.65, 28.01, 13.93cm^{-1}; HRMS calcd for $C_{20}H_{22}F_3NO_6$ $(M+H)^+$ 430.1491, found 430.1472.

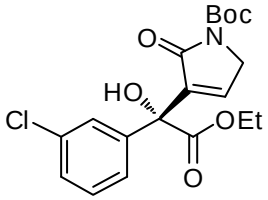
(R)-*tert*-Butyl 3-(2-ethoxy-1-(4-fluorophenyl)-1-hydroxy-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4n)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 8.7 min, t_{minor} = 17.7 min, 96% ee). oily liquid, 67% yield; $[\alpha]_D^{20}$ = -6.96 (*c* = 1.0, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.69 – 7.59 (m, 2H), 7.13 – 7.02 (m, 2H), 6.71 (t, *J* = 2.0 Hz, 1H), 4.63 (s, 1H), 4.42 – 4.16 (m, 4H), 1.55 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.75, 167.43, 161.00, 149.25, 141.10, 140.96, 133.42, 133.38, 128.03, 127.92, 115.27, 114.99, 83.40, 74.90,

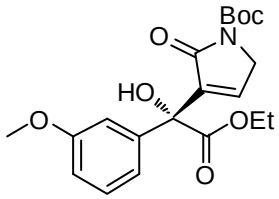
63.00, 49.56, 27.95, 13.88cm^{-1}; HRMS calcd for $C_{19}H_{22}FNO_6$ $(M+H)^+$ 402.1335, found 402.1323.

(R)-*tert*-Butyl 3-(1-(3-chlorophenyl)-2-ethoxy-1-hydroxy-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4o)



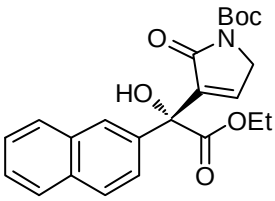
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 9.4 min, t_{minor} = 15.6 min, 99% ee). oily liquid, 74% yield; $[\alpha]_D^{20}$ = -3.061 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 – 7.66 (m, 1H), 7.54 – 7.52 (m, 1H), 7.38 – 7.30 (m, 2H), 6.73 (t, *J* = 2.0 Hz, 1H), 4.64 (s, 1H), 4.39 – 4.17 (m, 4H), 1.56 (s, 9H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.42, 167.35, 149.23, 141.13, 140.70, 139.73, 134.39, 129.52, 128.63, 126.43, 124.33, 83.46, 74.89, 63.17, 49.60, 27.98, 13.90cm^{-1}; HRMS calcd for $C_{19}H_{22}ClNO_6$ $(M+H)^+$396.1214, found 396.1208.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-1(3-methoxyphenyl)-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4p)



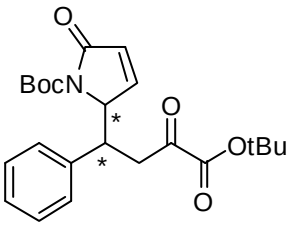
The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 17.2 min, t_{minor} = 19.5 min, 99% ee). oily liquid, 88% yield; $[\alpha]_D^{20}$ = -1.654 (*c* = 1.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.20 (m, 1H), 7.20–7.17 (m, 2H), 6.91– 6.87 (m, 1H), 6.73 (t, *J* = 2.0 Hz, 1H), 4.62 (s, 1H), 4.39 – 4.17 (m, 4H), 3.82 (s, 3H), 1.55 (s, 9H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.79, 167.53, 159.52, 149.30, 141.17, 140.99, 139.20, 129.20, 118.33, 113.91, 111.64, 83.30, 75.20, 62.88, 55.19, 49.54, 27.96, 13.91cm^{-1}; HRMS calcd for $C_{20}H_{25}NO_7$ $(M+H)^+$ 392.1708, found 392.1704.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-1(naphthalene-1-yl)-2-oxoethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4q)



The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 98/2, 1.0 ml/min, t_{major} = 12.2 min, t_{minor} = 41.1 min, 99% ee). oily liquid, 54% yield; $[\alpha]_D^{20}$ = -1.97 (*c* = 1.0, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.89 – 7.84 (m, 3H), 7.70 – 7.67 (m, 1H), 7.53 – 7.50 (m, 2H), 6.71 (t, *J* = 1.9 Hz, 1H), 4.73 (s, 1H), 4.41 – 4.14 (m, 4H), 1.56 (s, 9H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.92, 167.59, 149.37, 141.30, 141.21, 134.96, 133.04, 132.90, 128.45, 127.91, 127.48, 126.58, 126.38, 125.42, 123.81, 83.41, 75.47, 63.03, 49.64, 28.02, 13.98cm^{-1}; HRMS calcd for $C_{23}H_{25}NO_6$ $(M+H)^+$ 412.1775, found 412.1755.

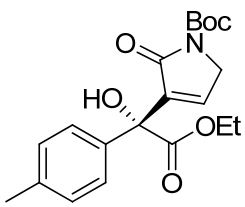
***tert*-Butyl 3-(4-tert-butoxy-3,4-dioxo-1-phenylbutyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (4r)**

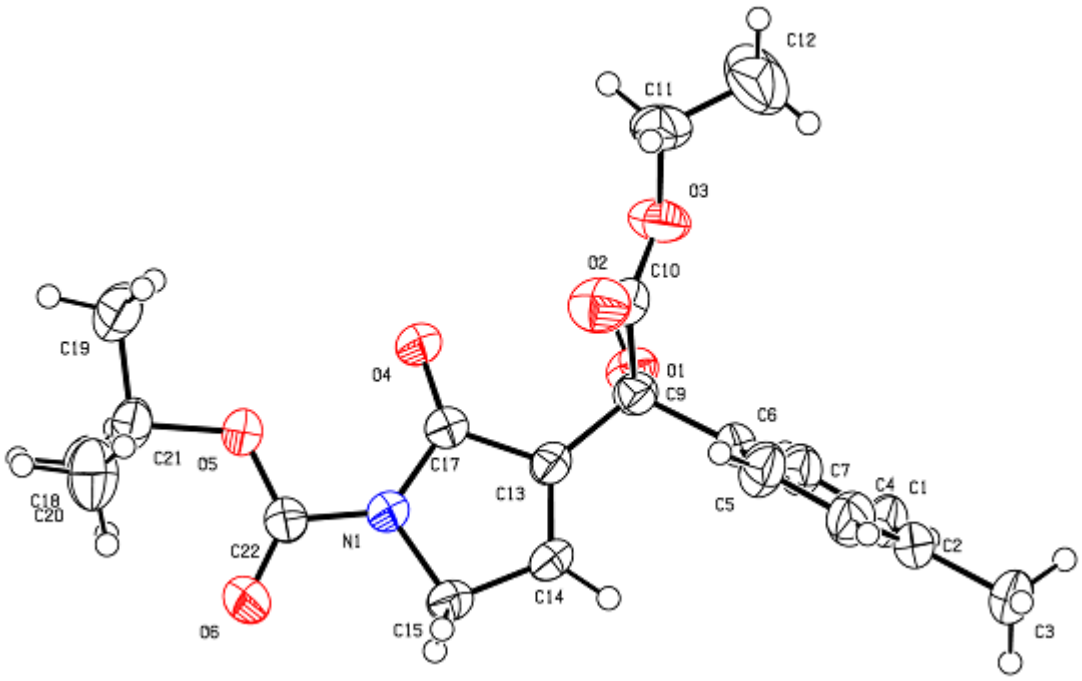


The ee value determination was carried out using chiral high-performance liquid chromatograph (HPLC) with Chiracel IA-H colum (hexane/2-propanol = 80/20, 1.0 ml/min, t_{minor} = 10.2 min, t_{major} = 16.1 min, ee 96%, dr 7:1). oily liquid, 61% yield; $[\alpha]_D^{20}$ = 6.915 (*c* = 1.0, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 3H), 7.21 – 7.17 (m, 1H), 7.17 – 7.16 (m, 1H), 7.00 – 7.97(m, 1H), 4.81– 4.78 (m, 1H), 4.42– 4.36 (m, 1H), 3.16 (dd, *J* = 18.1, 9.7 Hz, 1H), 2.82 (dd, *J* = 18.1, 5.0 Hz, 1H), 1.65 (s, 9H), 1.50 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 192.92, 169.17, 159.94, 149.19, 147.40, 138.37, 129.02, 128.18, 127.88, 127.71, 84.29, 83.79, 66.30, 40.38, 35.58, 28.15, 27.68cm^{-1}; HRMS calcd for $C_{23}H_{29}NO_6$ $(M+H)^+$ 438.1892, found 438.1887.

X-ray structure of 4g.

(R)-*tert*-Butyl 3-(2-ethoxy-1-hydroxy-2-oxo-1-p-tolylethyl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxlate (CCDC: 921456)





Bond precision:	C-C = 0.0032 Å	Wavelength = 0.71073	
Cell:	a = 9.941 (4)	b = 7.947 (3)	c = 12.485 (4)
	Alpha = 90	beta = 94.403 (4)	gamma = 90
Temperature:	296K		

	Calculated	Reported
Volume	983.4 (6)	983.4 (6)
Space group	P 21	P2 (1)
Hall group	P 2yb	
Moiety formula	C20 H25 N O6	
Sum formula	C20 H25 N O6	C20 H25 N O6
Mr	375.41	375.41
Dx, g cm^{-3}	1.268	1.268
Z	2	2
Mu (mm^{-1})	0.094	0.094
F000	400.0	400.0

F000’	400.22	
h, k, lmax	11, 9, 14	11, 9, 14
Nref	1870 [3468]	3376
Tmin, Tmax	0.981, 0.986	0.982, 0.986
Tmin’	0.981	

Correction method = MULTI – SCAN

Date completeness = 1.81/0.97 Theta (max) = 24.990

R (reflections) = 0.0326 (3109) wR2 (reflections) = 0.1070 (3376)

S = 1.031 Npar = 250

Reference

1 T. Okino, Y. Hoashi and Y. Takemoto, *J. Am. Chem. Soc*., 2003, **125**, 12672.

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5 (a) X. Creay, *J. Org. Chem.*, 1987, **52**, 5026–5030; (b) N. J. A. Martin, X. Cheng and B. List, *J. Am. Chem. Soc*., 2008, **130**, 13862–13863.

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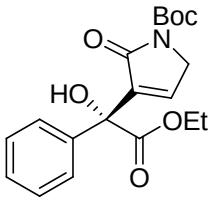
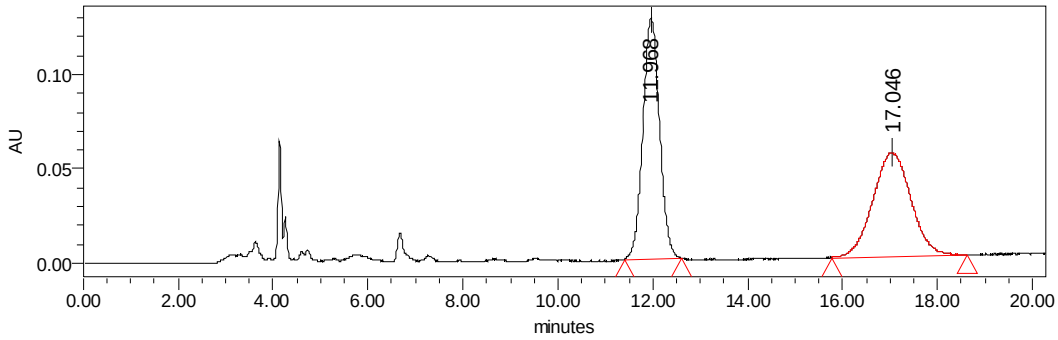
HPLC results

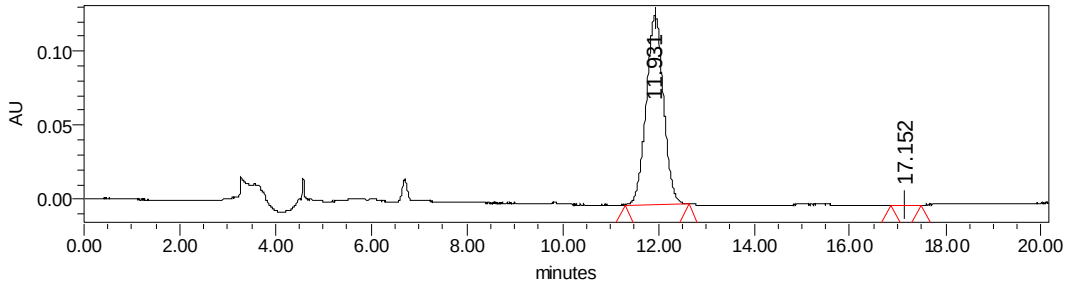


4a

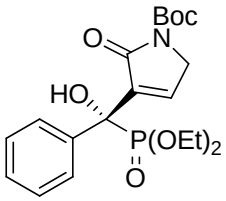
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	11.968	3102131	50.49	127350	bb	Unknow
2	17.046	3041515	49.51	54862	bb	Unknow

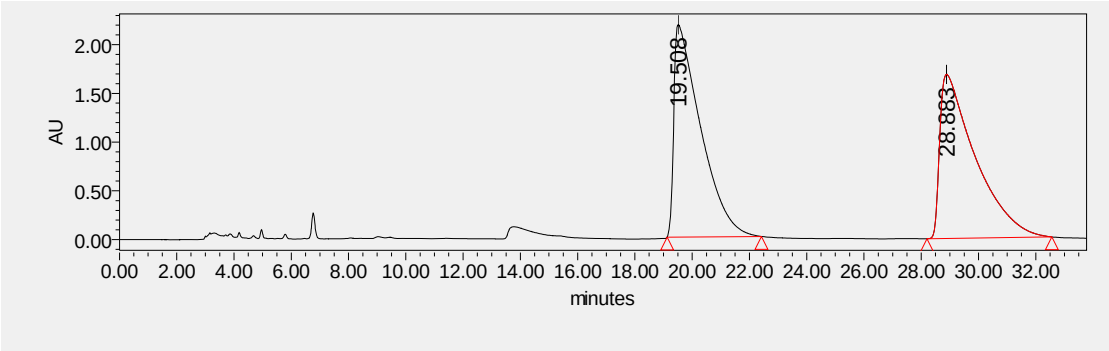


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	11.931	3216219	99.80	127878	bb	Unknow
2	17.152	6481	0.20	339	bb	Unknow



4b

HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	19.508	141235189	49.45	2178662	bb	Unknow
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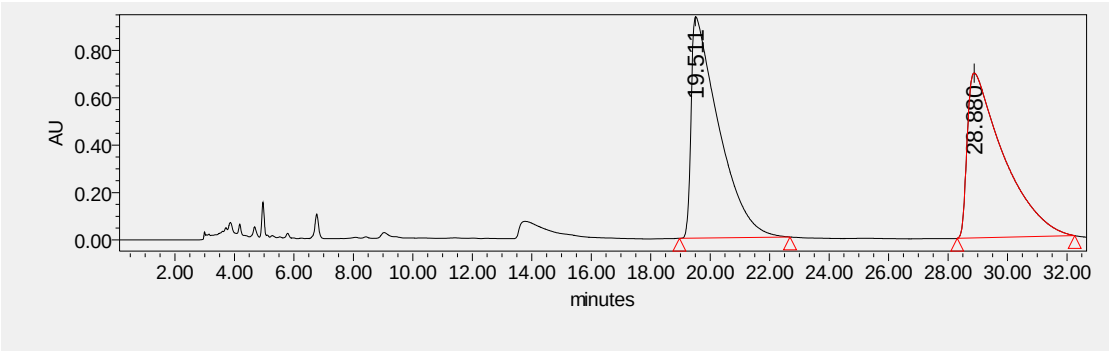
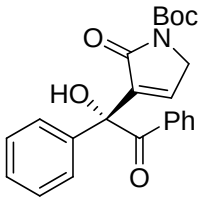
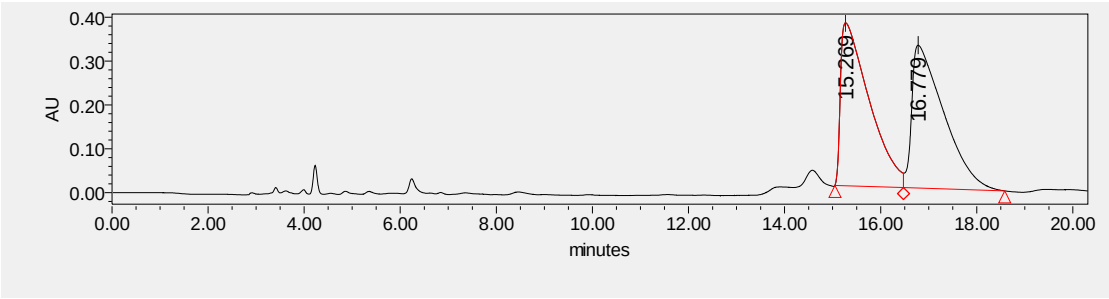


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	19.511	60007529	50.49	934572	bb	Unknow
2	28.880	58851108	49.51	696030	bb	Unknow

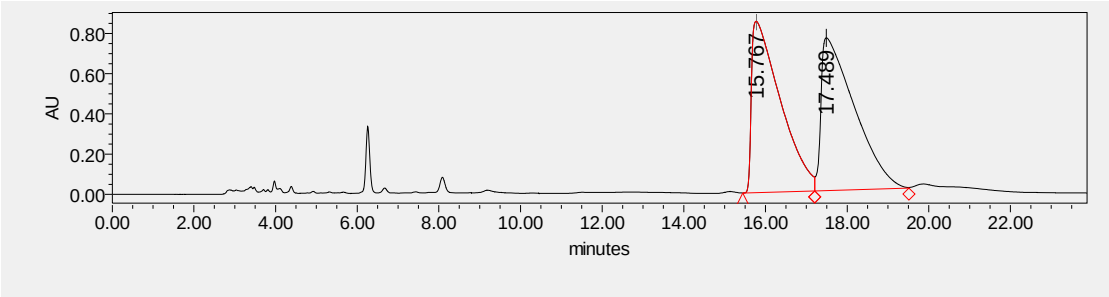


4c

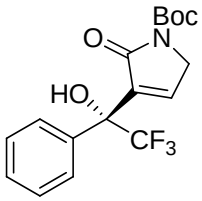
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	15.269	15388825	50.44	371557	bv	Unknow
2	16.779	15123210	49.56	325782	vb	Unknow

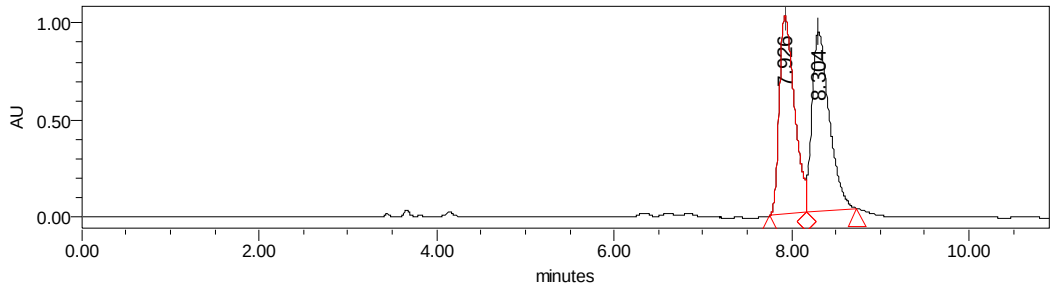


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	15.767	41645092	49.12	851798	bV	Unknow
2	17.489	43137672	50.88	758840	VV	Unknow

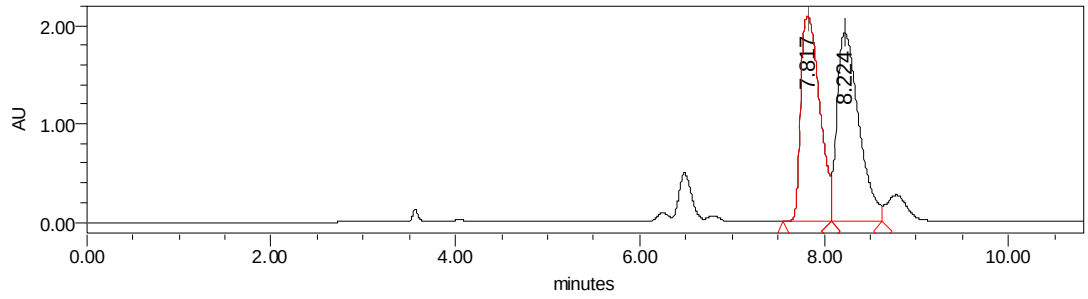


4d

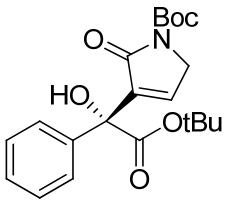
HPLC using an IA-H colum (hexane/2-propanol = 90/10, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.926	12178264	49.18	1020300	bV	Unknow
2	8.304	12586863	50.82	927083	vb	Unknow

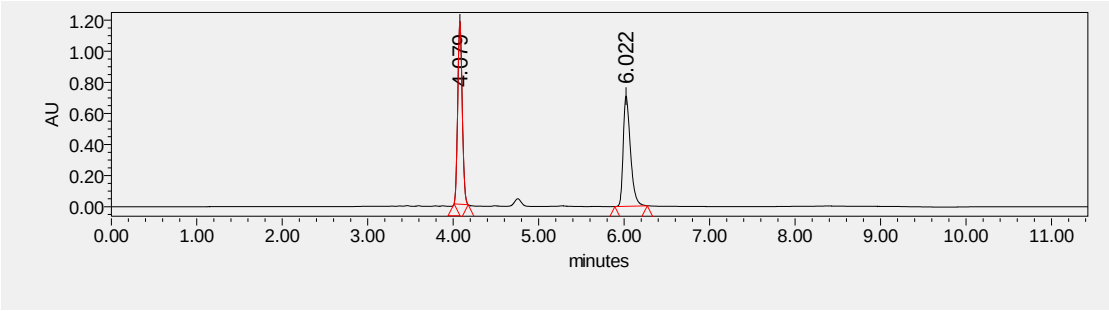


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.817	29811898	48.02	2094248	BV	Unknow
2	8.224	32275676	51.98	1933002	VV	Unknow

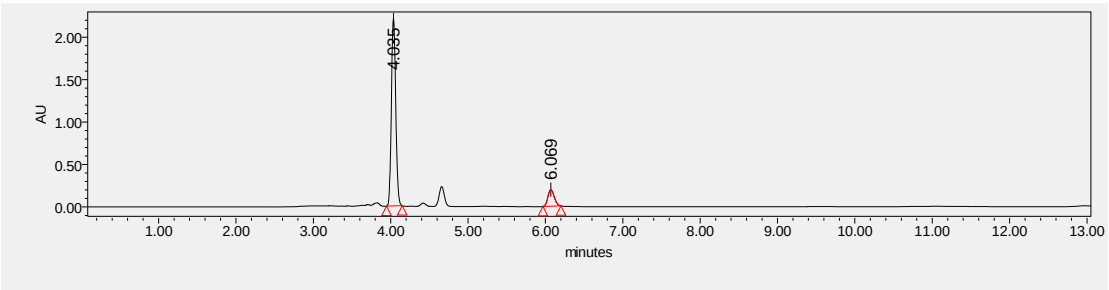


4e

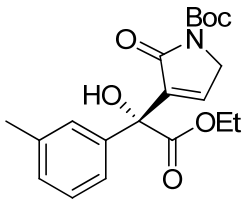
HPLC using an IA-H colum (hexane/2-propanol = 100/0.1, flow rate 0.5 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	4.079	4258411	50.68	1173846	bb	Unknow
2	6.022	4144571	49.32	708904	bb	Unknow

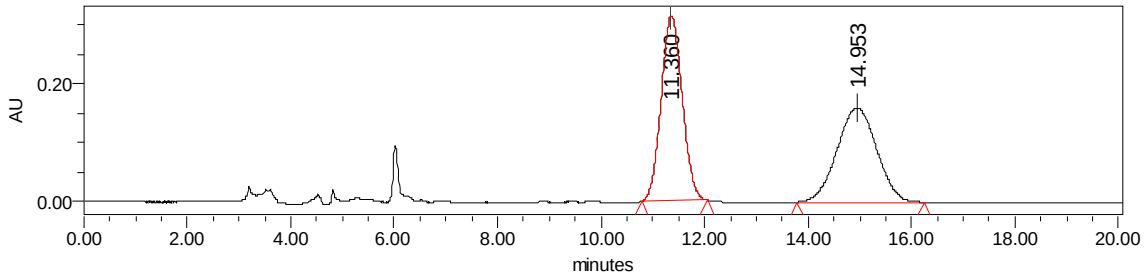


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	4.035	8605742	88.57	2204851	bb	Unknow
2	6.069	1110458	11.43	19638	bb	Unknow



4f

HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	11.360	8863623	50.90	314490	bb	Unknow
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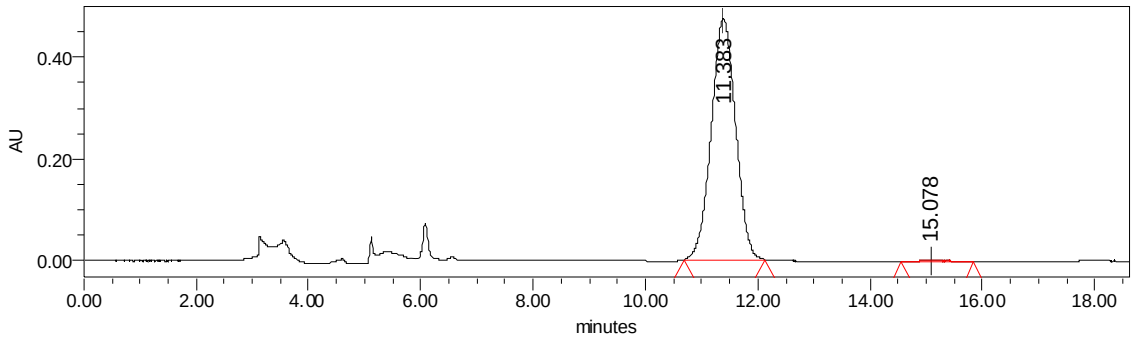
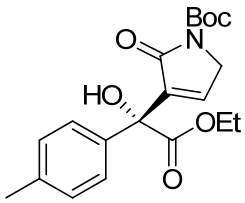
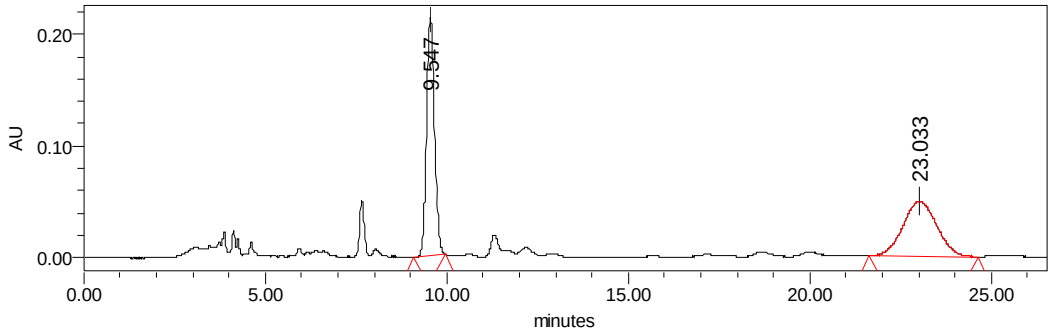


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	11.383	13866305	99.28	462651	bb	Unknow
2	15.078	100158	0.72	2546	bb	Unknow

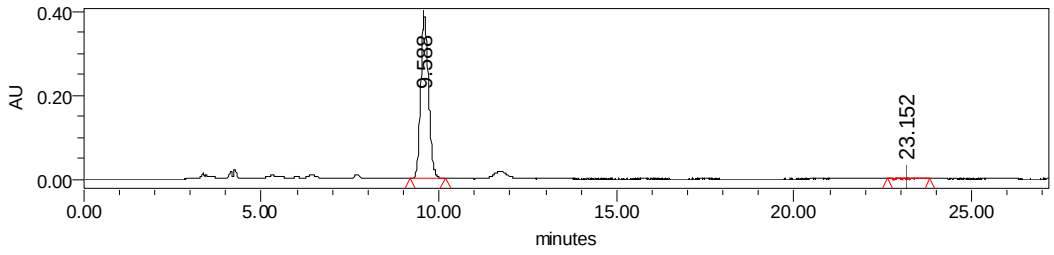


4g

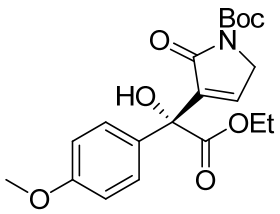
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.547	3229192	50.73	212815	bb	Unknow
2	23.033	3136659	49.27	48658	bb	Unknow

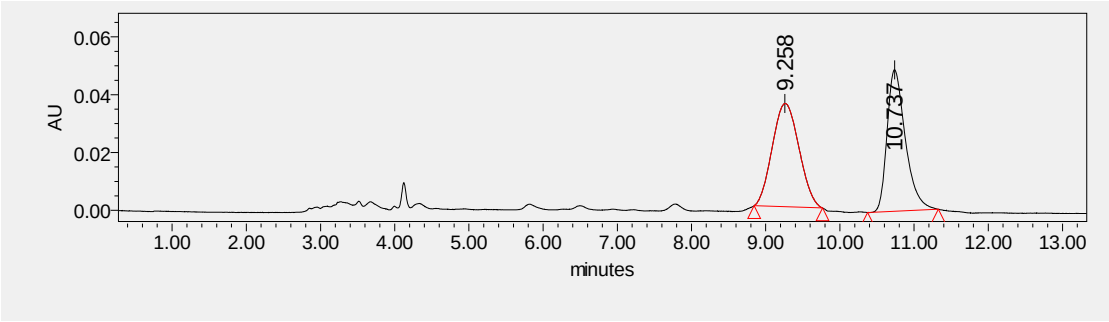


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.588	5960858	99.82	385066	bb	Unknow
2	23.152	10536	0.18	513	bb	Unknow

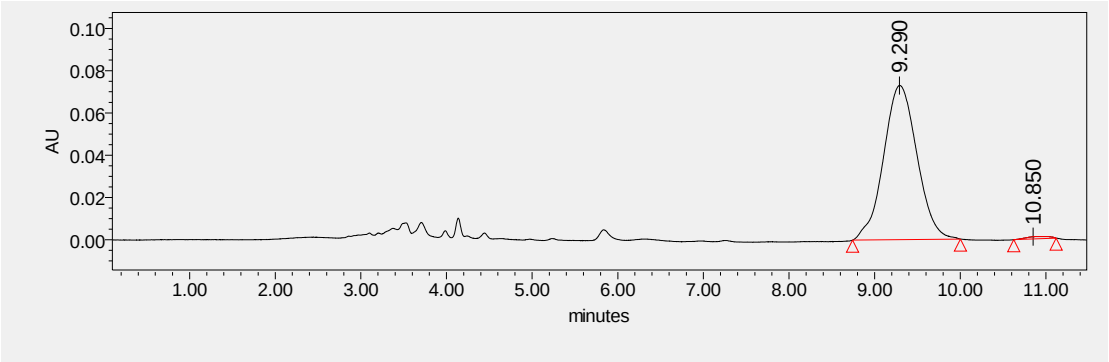


4h

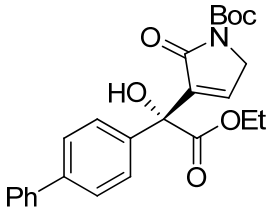
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.258	833791	49.87	34600	bb	Unknow
2	10.737	838290	50.13	48931	bb	Unknow

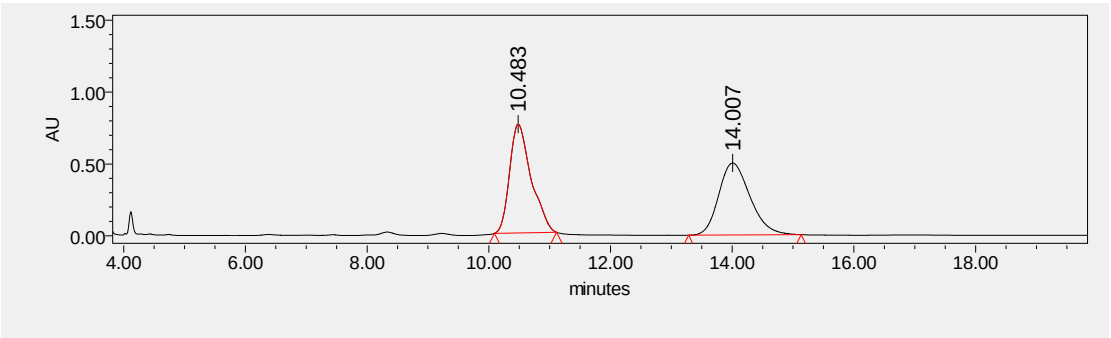


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.290	1975554	99.02	72938	bb	Unknow
2	10.850	19648	0.98	1076	bb	Unknow

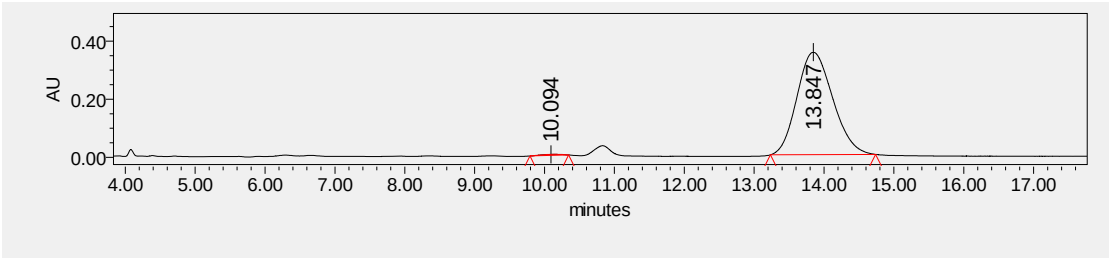


4i

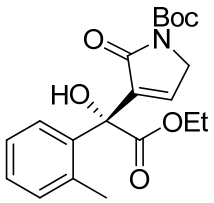
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	10.483	18623349	51.00	757591	bb	Unknow
2	14.007	17891128	49.00	501189	bb	Unknow

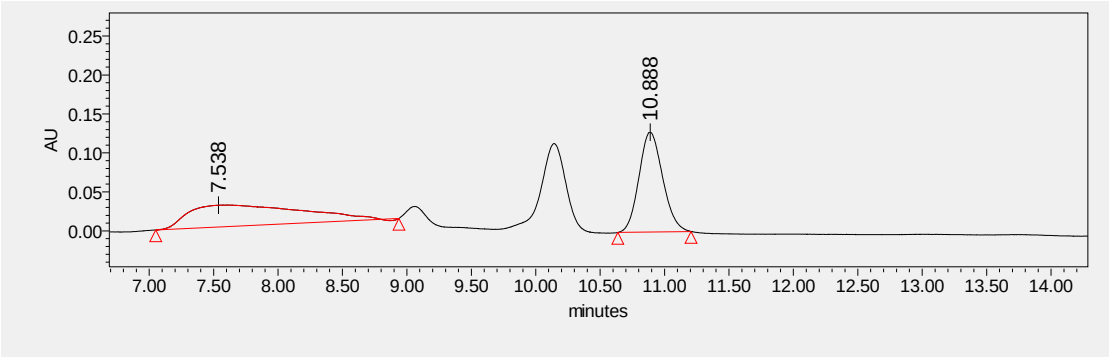


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	10.094	76293	0.61	3769	bb	Unknow
2	13.847	12524924	99.39	352776	bb	Unknow

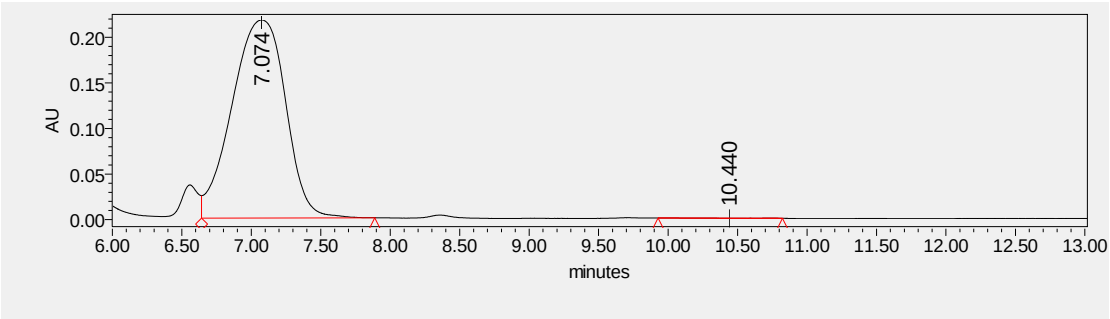


4j

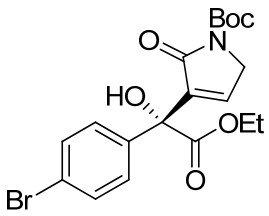
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.538	1686218	50.71	27957	bb	Unknow
2	10.888	1639269	49.29	128164	bb	Unknow

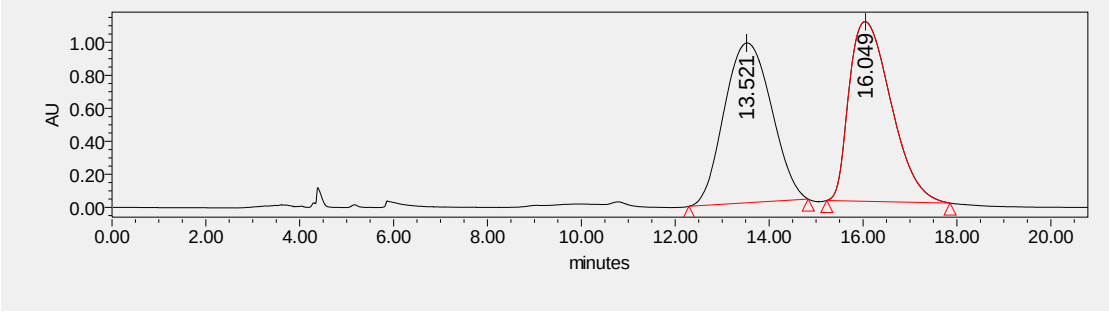


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.074	5832252	99.93	217231	bb	Unknow
2	10.440	3905	0.07	194	bb	Unknow

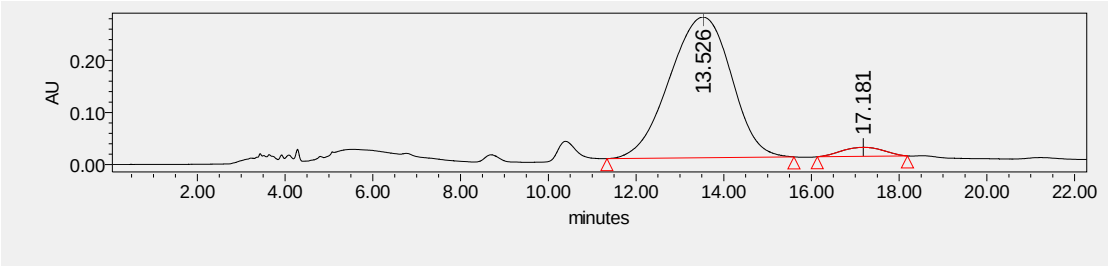


4k

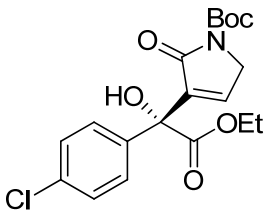
HPLC using an IA-H colum (hexane/2-propanol = 100/0.5, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	13.521	67613342	49.99	968136	bb	Unknow
2	16.049	67653337	50.01	1088316	bb	Unknow

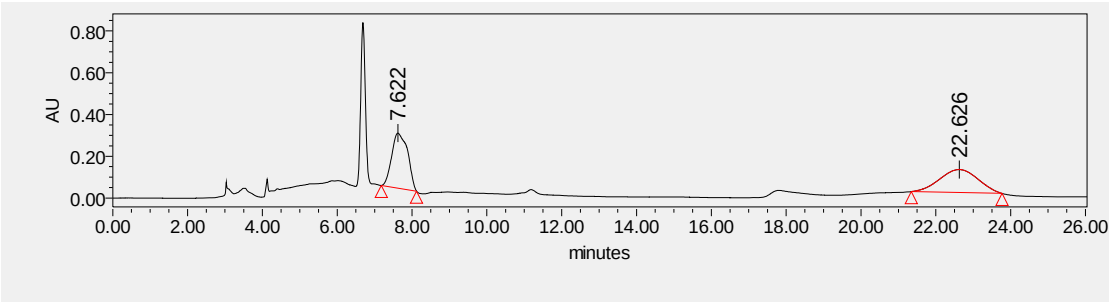


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	13.526	26437259	95.85	269806	bb	Unknow
2	17.181	1145794	4.15	17223	bb	Unknow

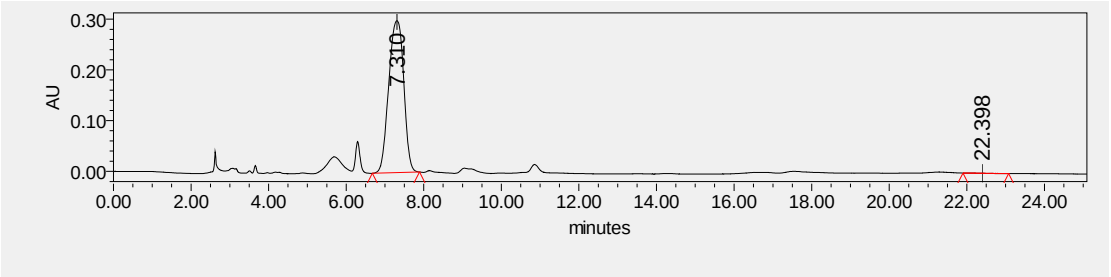


4l

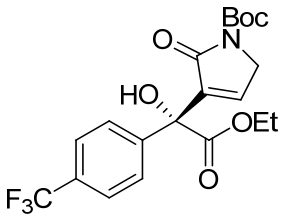
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.622	7757896	50.11	257613	bb	Unknow
2	22.626	7722508	49.89	107293	bb	Unknow

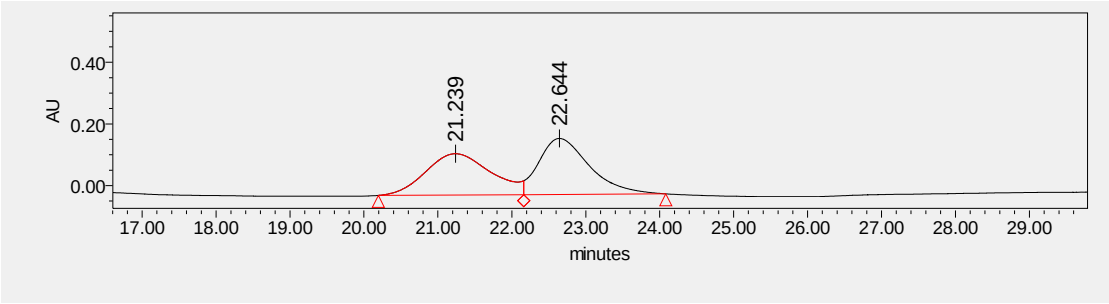


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.310	8032624	99.91	299653	bb	Unknow
2	22.398	6899	0.09	185	bb	Unknow

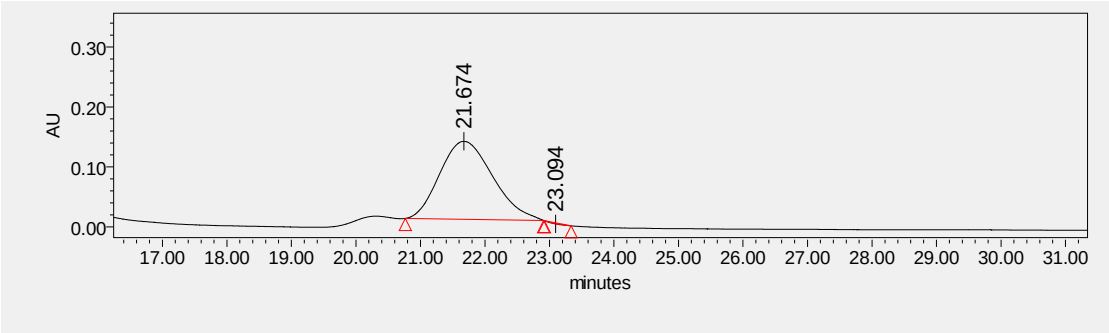


4m

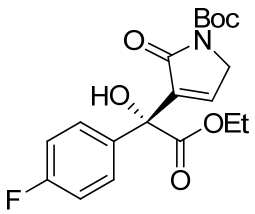
HPLC using an IA-H colum (hexane/2-propanol = 100/0.1, flow rate 0.5 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	21.239	8218503	48.91	133728	bv	Unknow
2	22.644	8584473	51.09	181443	vb	Unknow

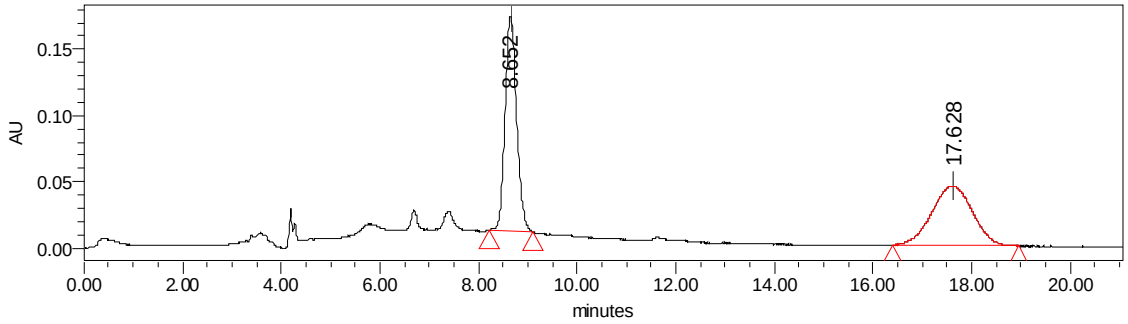


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	21.674	7433210	99.64	130144	bb	Unknow
2	23.094	26825	0.36	1691	bb	Unknow

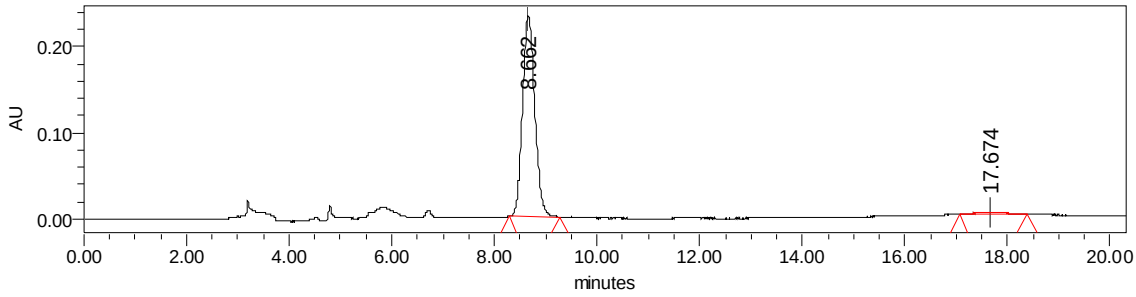


4n

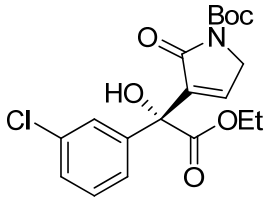
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	8.652	2627773	50.66	161622	bb	Unknow
2	17.628	2559737	49.34	45001	bb	Unknow

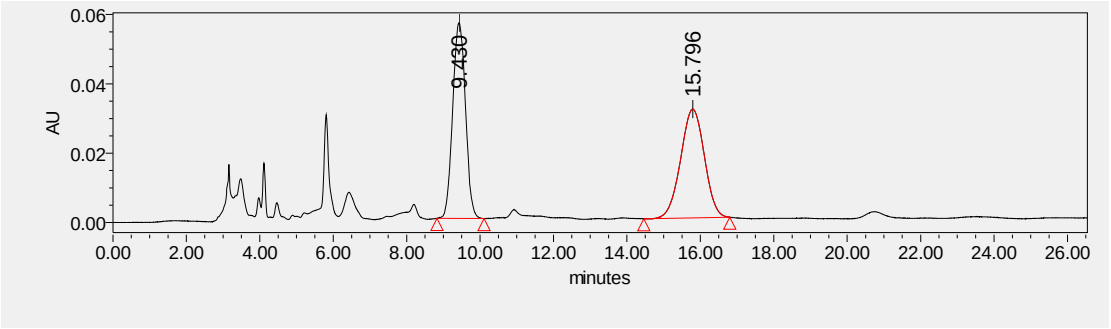


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	8.662	3900009	97.90	233293	bb	Unknow
2	17.674	83735	2.10	2005	bb	Unknow

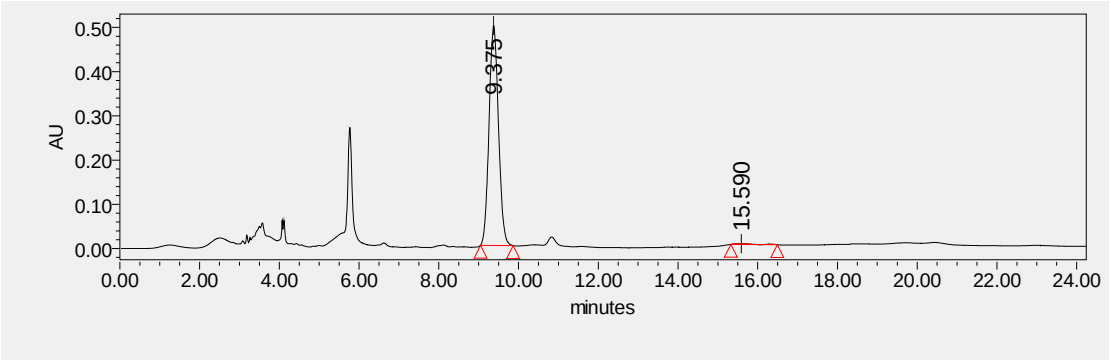


4o

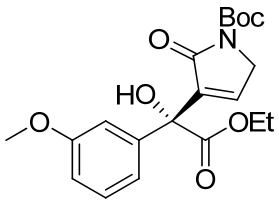
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.430	1364107	49.24	56402	bb	Unknow
2	15.796	1406246	50.76	31349	bb	Unknow

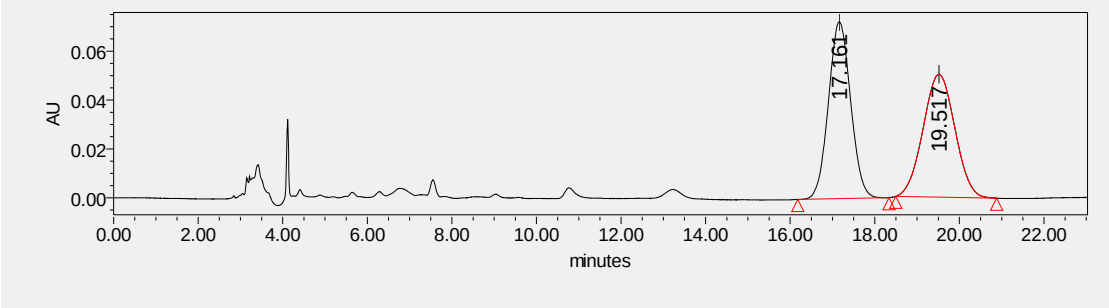


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	9.375	8308825	99.72	500062	bb	Unknow
2	15.590	23220	0.28	1145	bb	Unknow

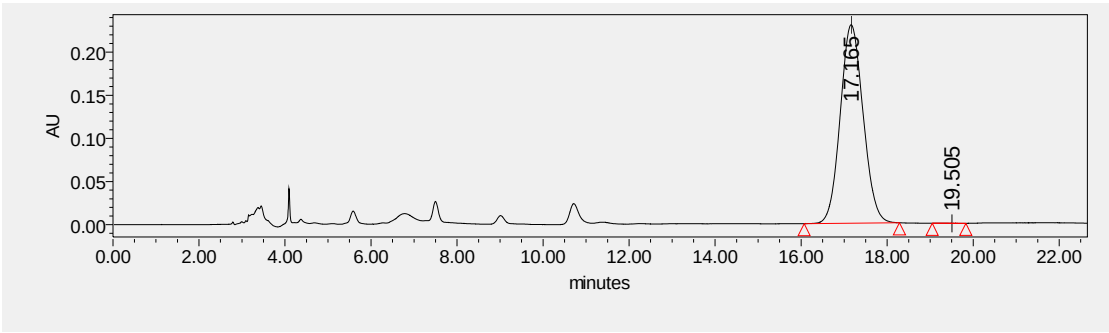


4p

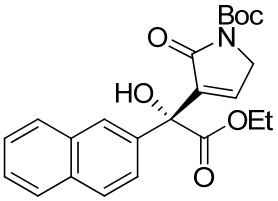
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	17.161	2635303	50.15	72368	bb	Unknow
2	19.517	2619932	49.85	50237	bb	Unknow

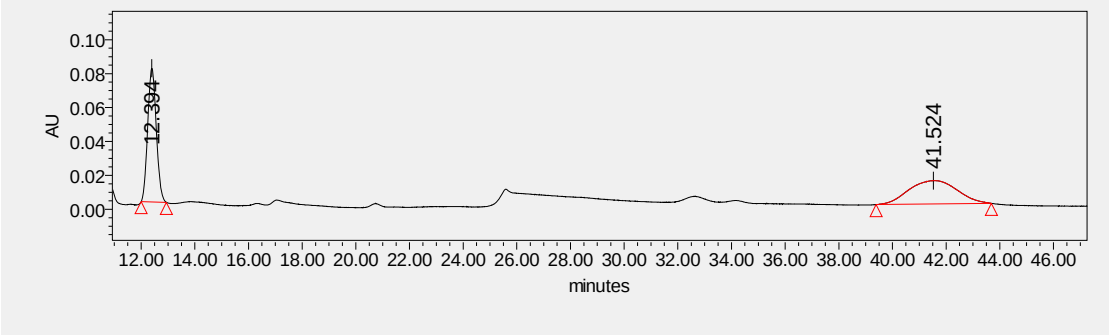


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	17.165	8477278	99.98	72368	bb	Unknow
2	19.505	1747	0.02	86	bb	Unknow

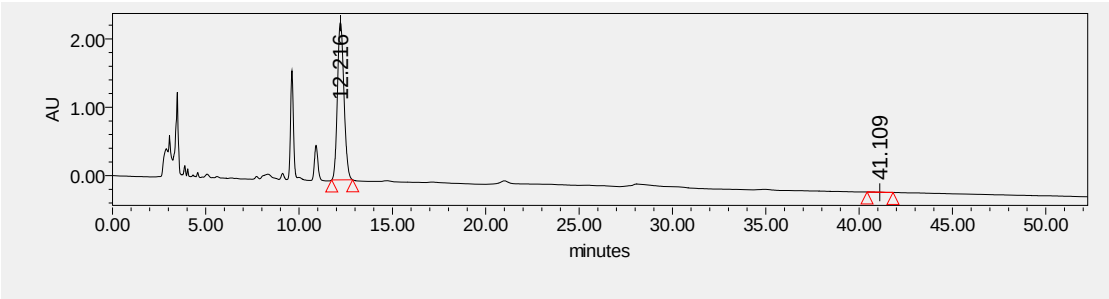


4q

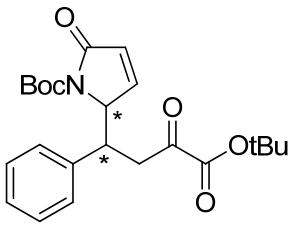
HPLC using an IA-H colum (hexane/2-propanol = 98/2, flow rate 1.0 ml/min)



Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	12.39	1753643	50.37	78901	bb	Unknow
2	41.524	1728081	49.63	13808	bb	Unknow

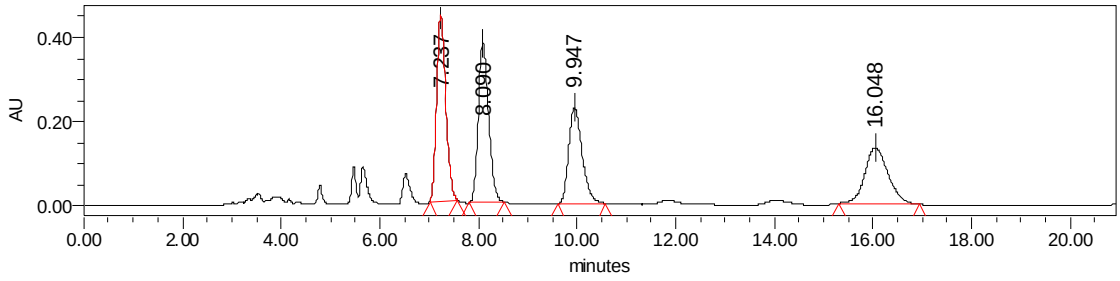


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	12.216	56269146	99.91	2304100	bb	Unknow
2	41.109	5561	0.09	164	bb	Unknow

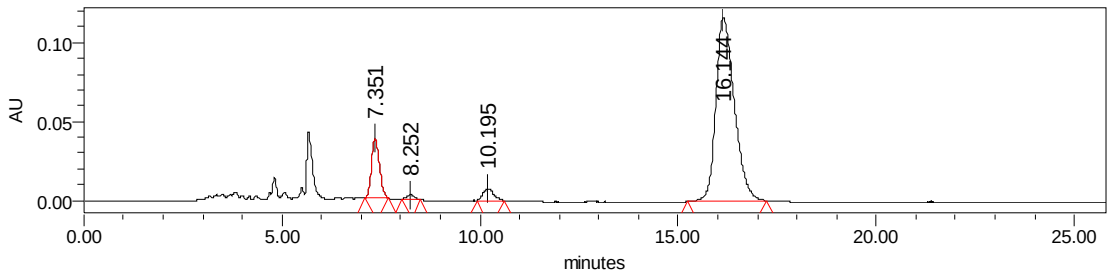


4r

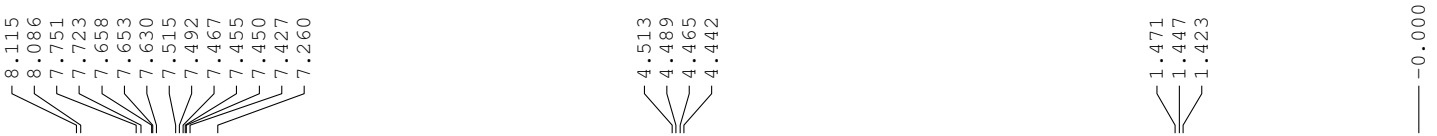
HPLC using an IA-H colum (hexane/2-propanol = 80/20, flow rate 1.0 ml/min)

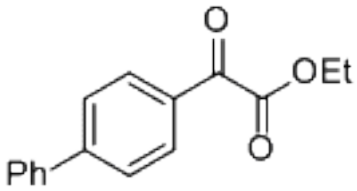


Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.237	5731268	28.59	442328	bb	Unknow
2	8.090	5532718	27.60	379156	bb	Unknow
3	9.947	4389543	21.90	229486	bb	Unknow
4	16.048	4391390	21.91	133351	bb	Unknow

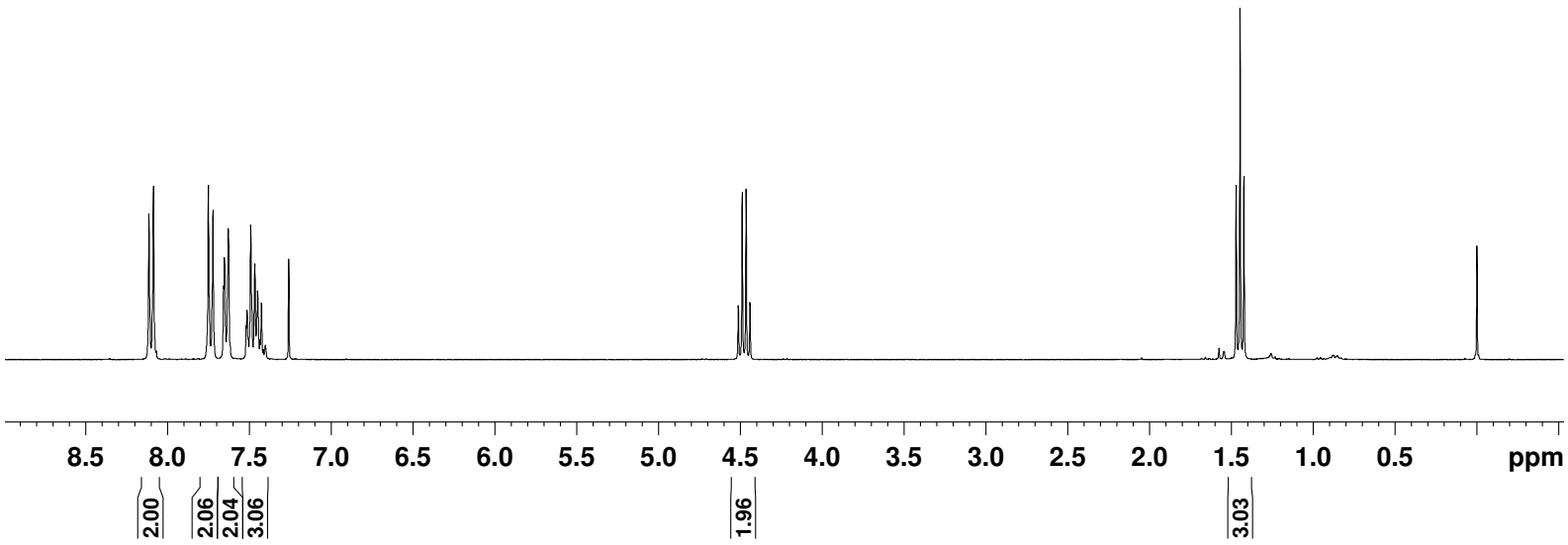


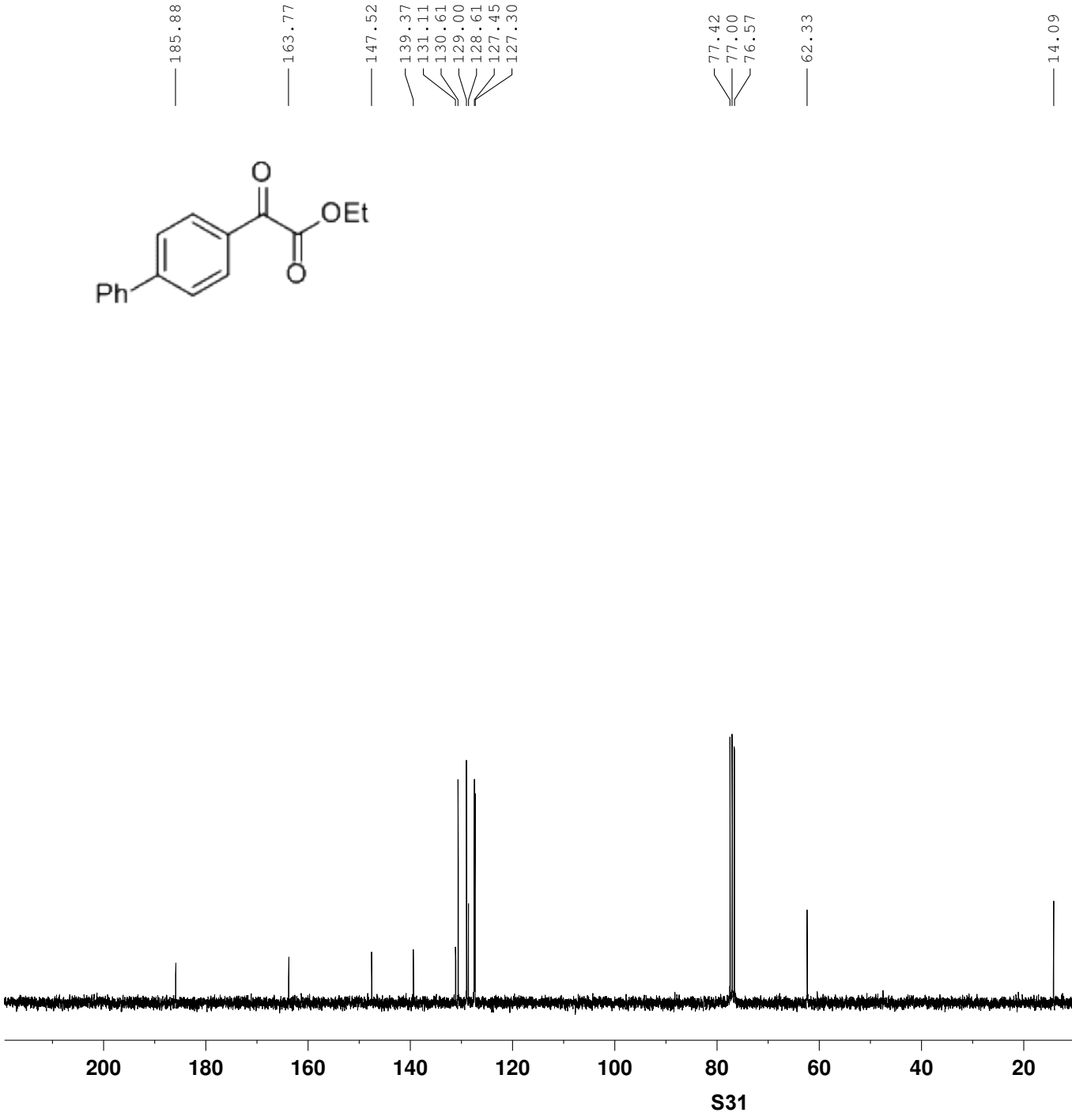
Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	7.351	527315	11.72	37791	bb	Unknow
2	8.252	39744	0.88	2924	bb	Unknow
3	10.195	140679	3.13	7614	bb	Unknow
4	16.144	3792602	84.27	116688	bb	Unknow



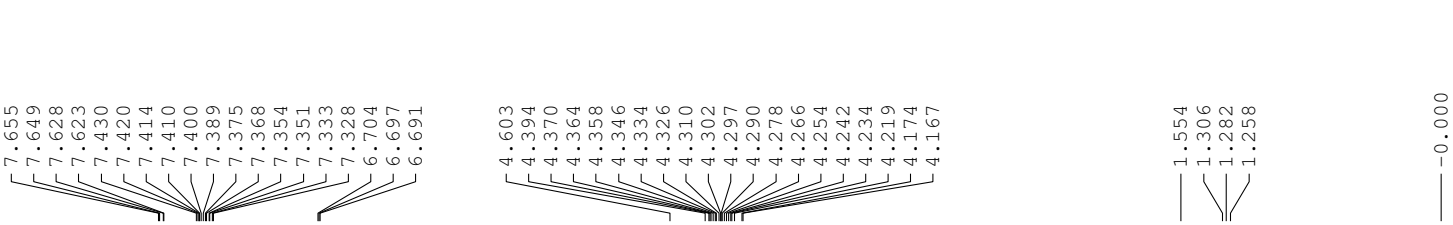


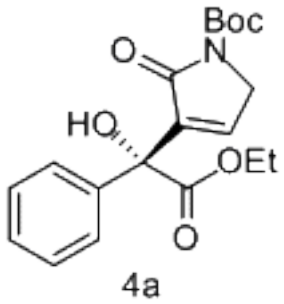

```
NAME            znjl12.11
EXPNO                  22
PROCNO                  1
Date_            20121114
Time                11.32
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                    203
DW                 80.800 usec
DE                  6.50 usec
TE                 288.6 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1300026 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```





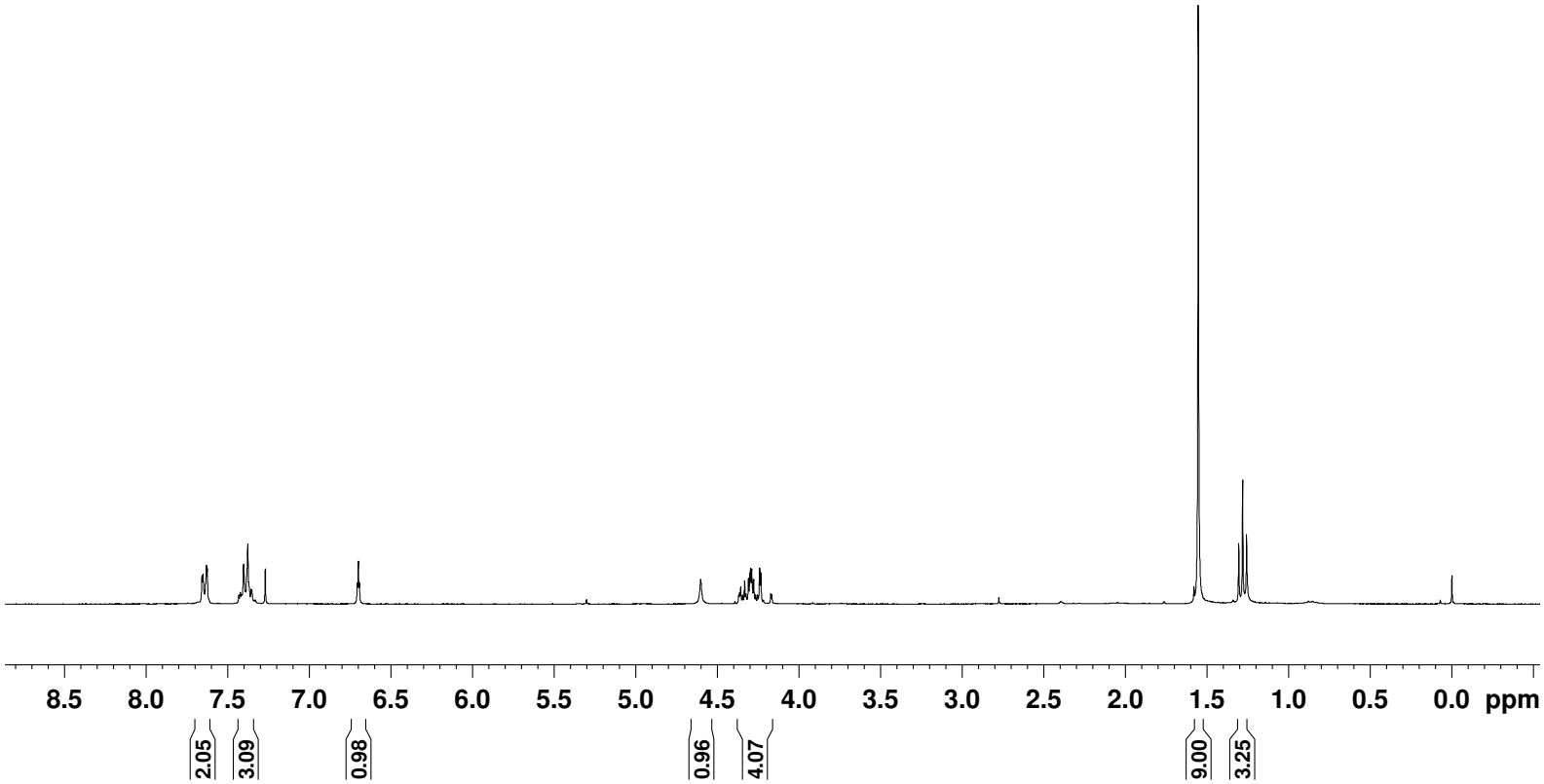

```
NAME           zhjl12.12
EXPNO                 44
PROCNO                 1
Date_           20121212
Time                16.38
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    37
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                288.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677542 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

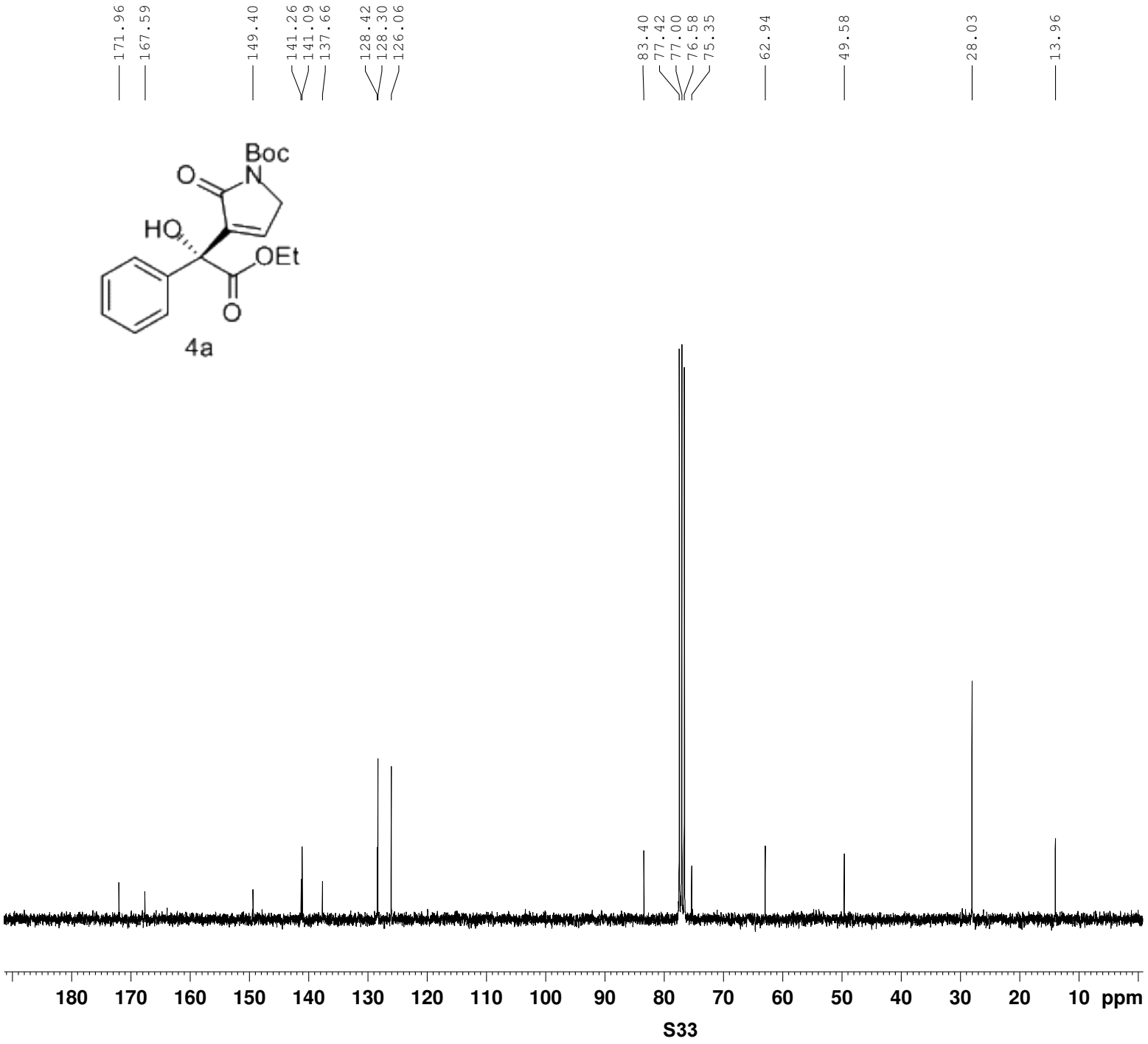




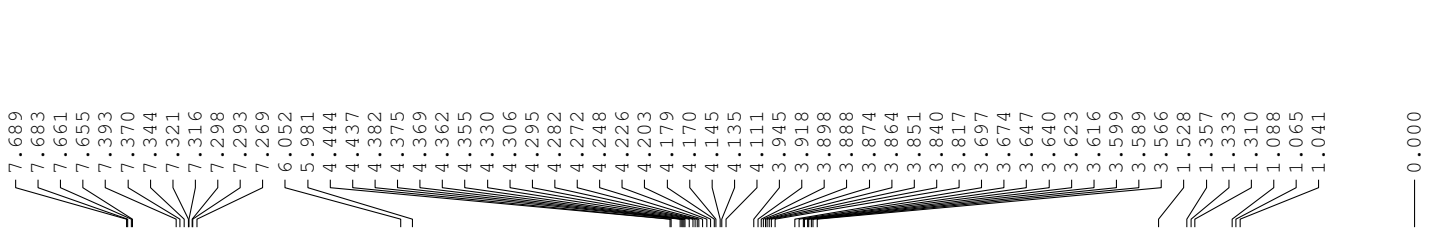
4a

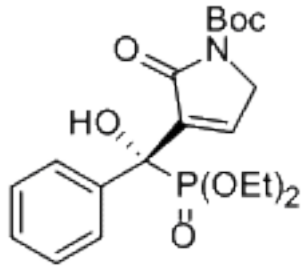
```
NAME            znjl12.10
EXPNO                  57
PROCNO                  1
Date_            20121016
Time                19.56
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                    128
DW                 80.800 usec
DE                  6.50 usec
TE                 288.7 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1300004 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```





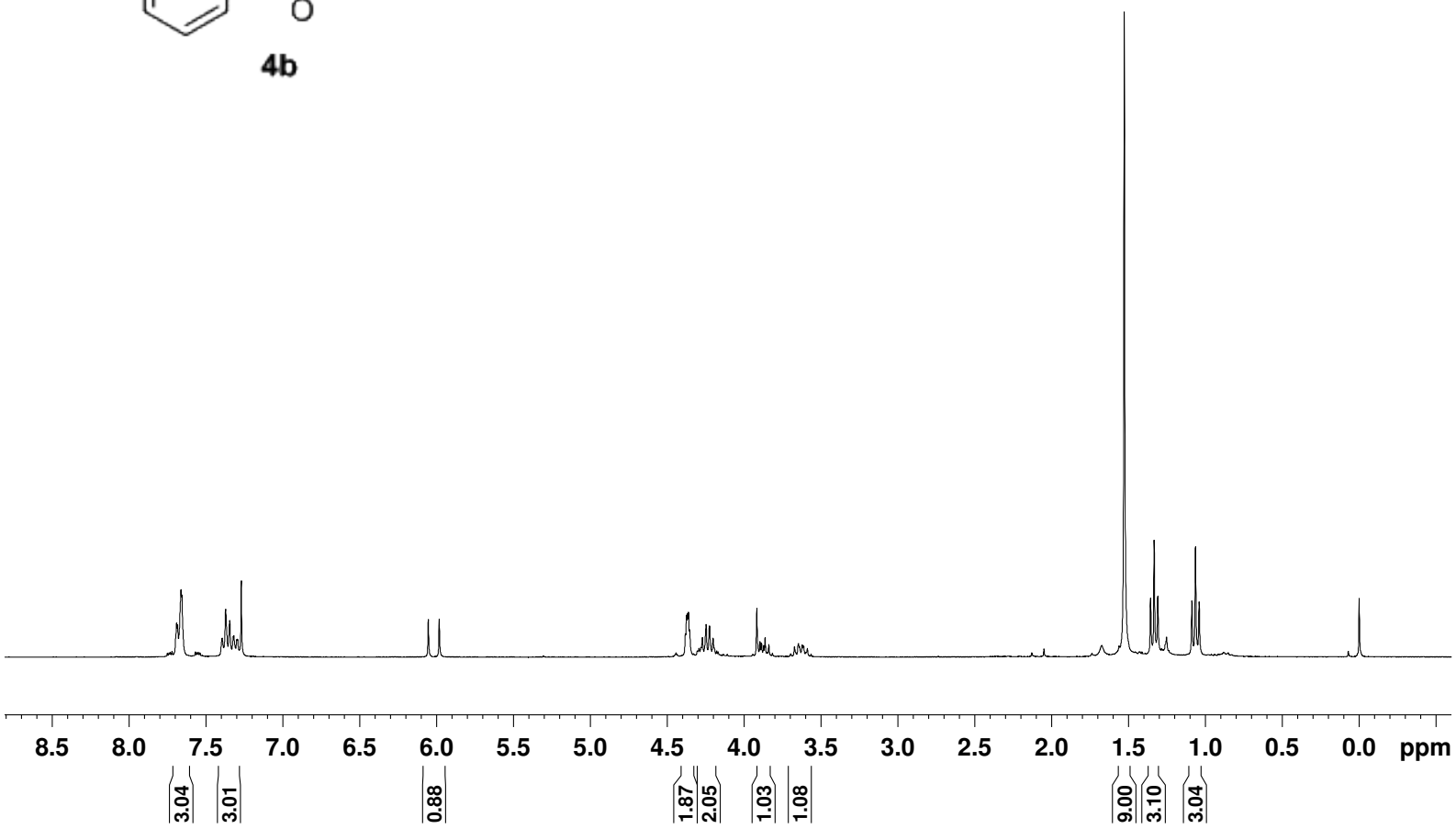

```
NAME           zhjl12.10
EXPNO                 58
PROCNO                 1
Date_           20121016
Time               21.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  363
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW                27.733 usec
DE                 6.50 usec
TE                289.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W        29.38907051 W
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2        300.1312005 MHz
SI                32768
SF           75.4677512 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

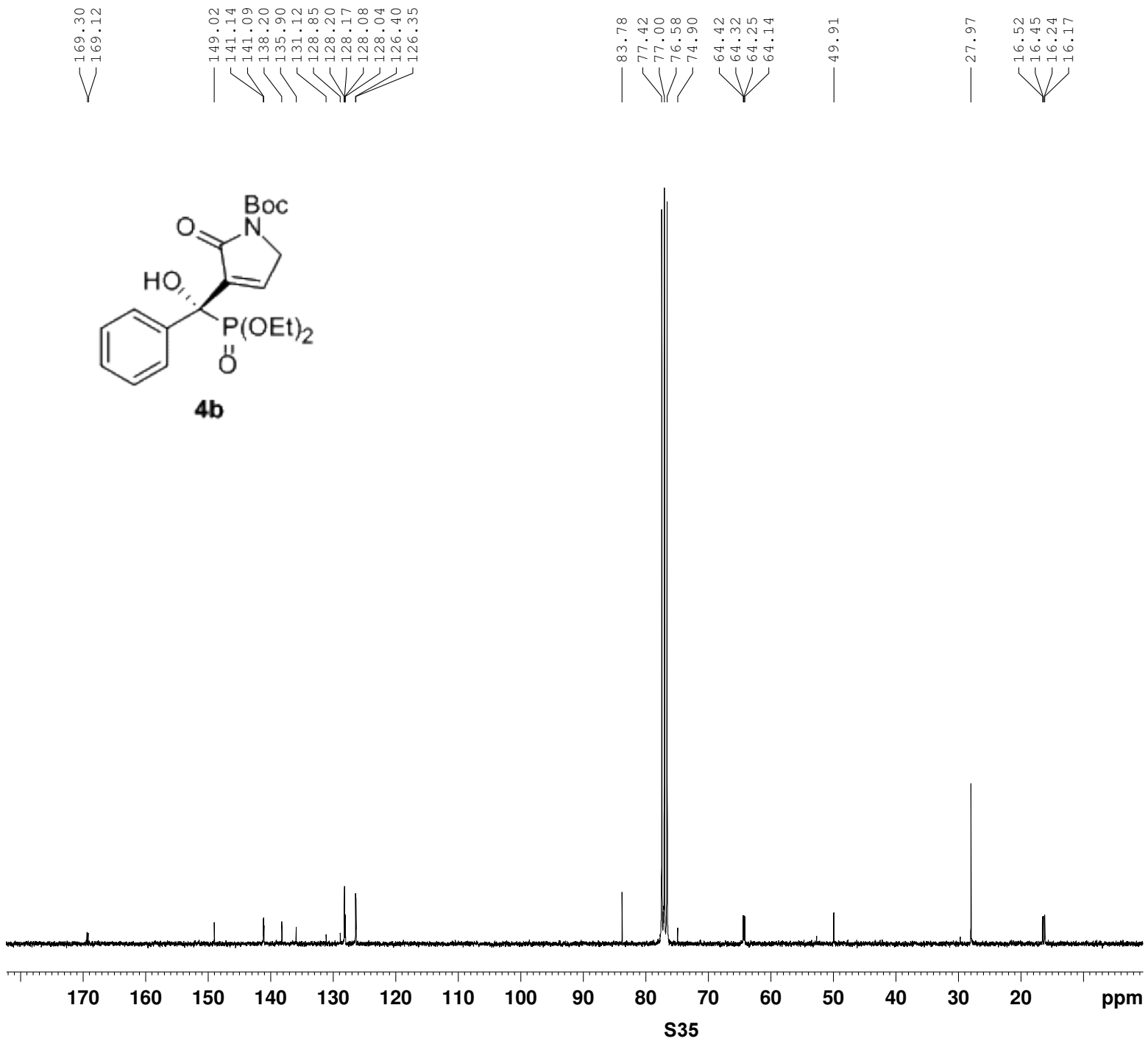




4b

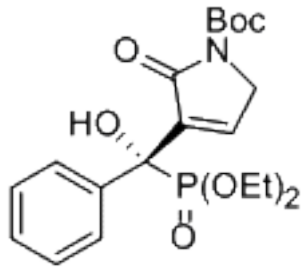
```
NAME            znj112.11
EXPNO                  95
PROCNO                  1
Date_            20121127
Time                10.59
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH           6188.119 Hz
FIDRES        0.094423 Hz
AQ            5.2953587 sec
RG                      4
DW              80.800 usec
DE               6.50 usec
TE               300.0 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W        11.55467796 W
SFO1        300.1318534 MHz
SI                  32768
SF          300.1300000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```





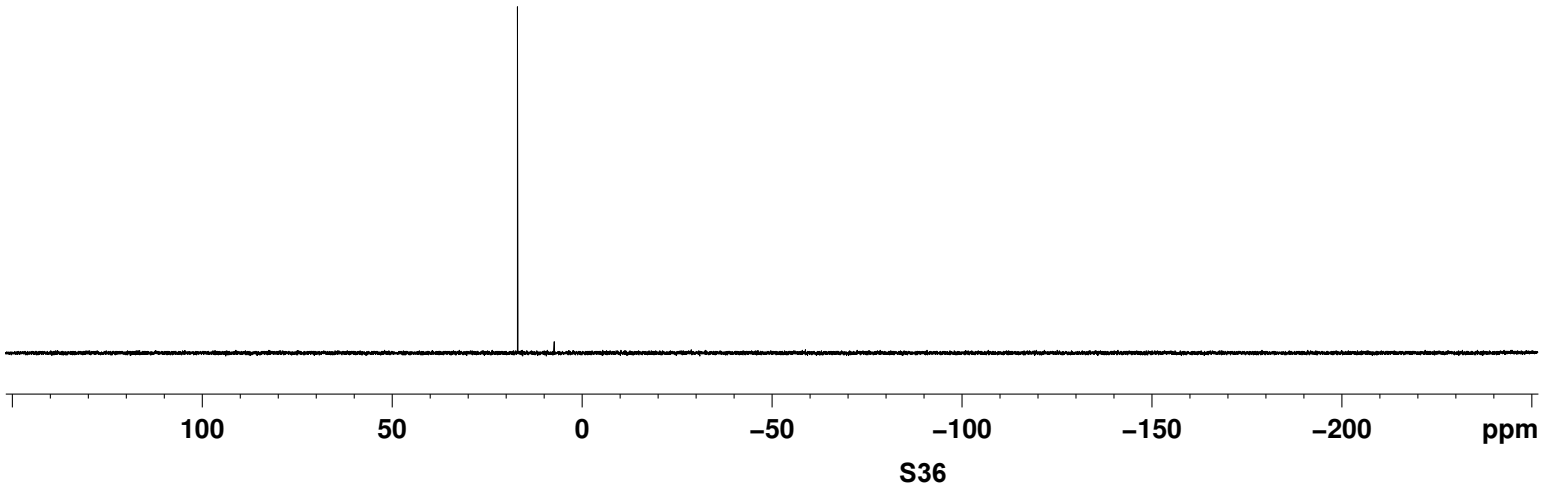

```
NAME            zhjl12.11
EXPNO                  99
PROCNO                  1
Date_            20121127
Time                11.51
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3312
DS                      0
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                  32768
SF            75.4677509 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                  1.40
```

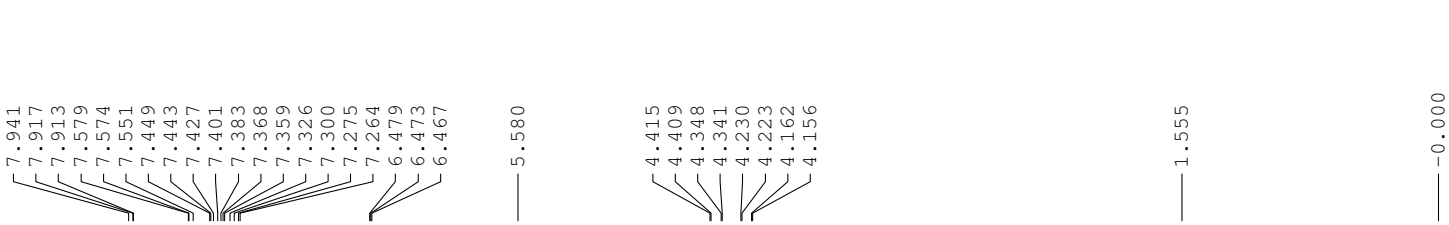
16.93

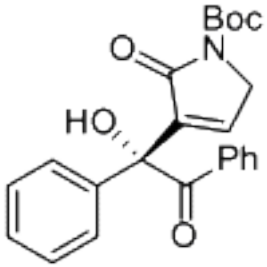


4b

```
NAME           zhjl12.11
EXPNO                 94
PROCNO                 1
Date_           20121127
Time               10.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           49019.609 Hz
FIDRES         0.747980 Hz
AQ             0.6685172 sec
RG                  203
DW                10.200 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                31P
P1                12.90 usec
PL1                3.00 dB
PL1W        18.50620270 W
SFO1        121.4887762 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2        300.1312005 MHz
SI                 32768
SF          121.4948510 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

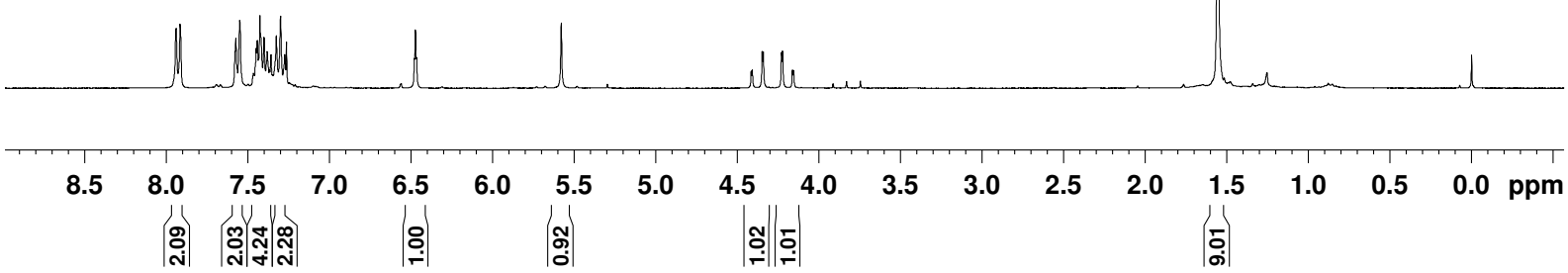




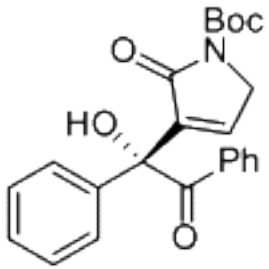


4c

```
NAME            znjl12.11
EXPNO                  68
PROCNO                  1
Date_            20121122
Time                10.37
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             6188.119 Hz
FIDRES          0.094423 Hz
AQ              5.2953587 sec
RG                   128
DW                80.800 usec
DE                 6.50 usec
TE                288.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W        11.55467796 W
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300016 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

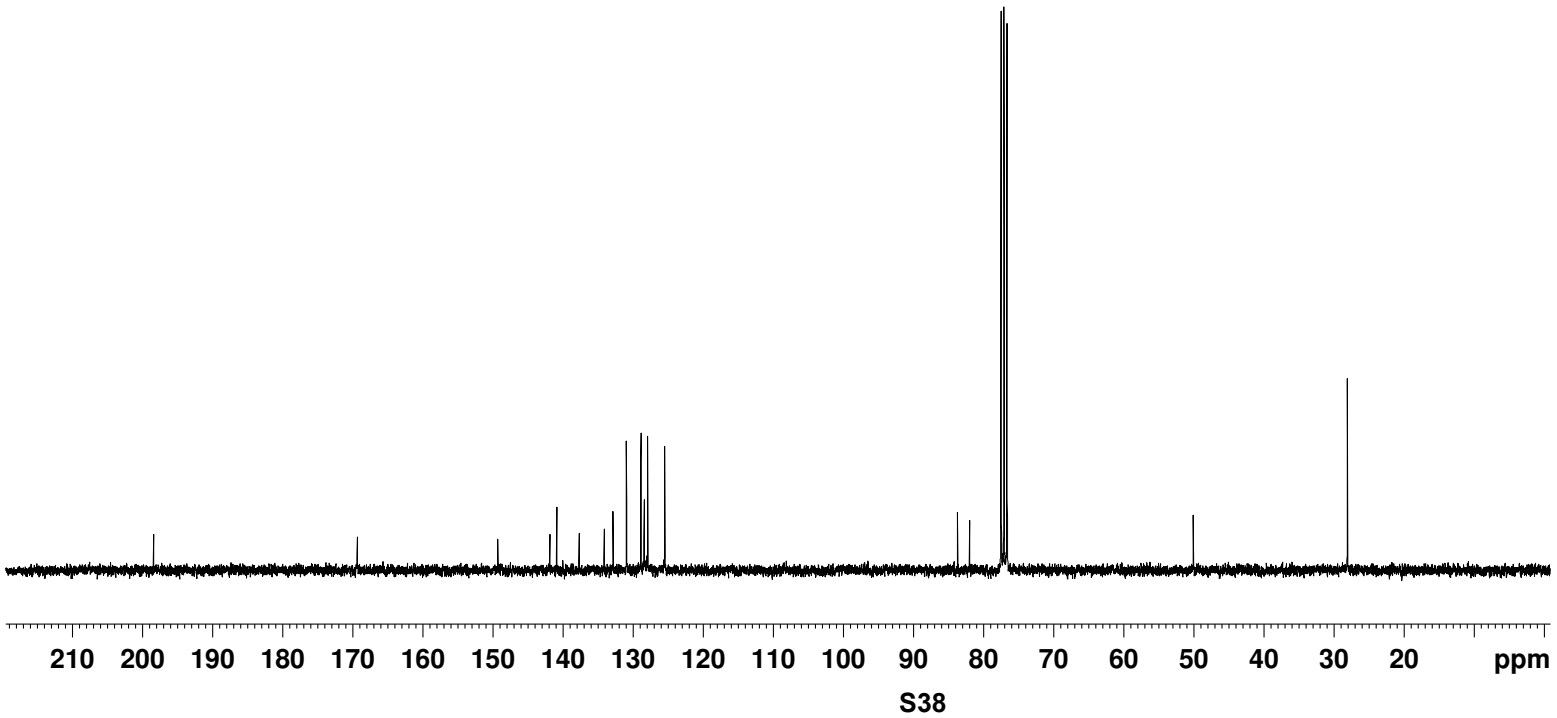


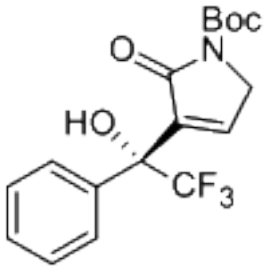
198.38
169.30
149.26
141.82
140.83
137.64
134.06
132.80
130.89
128.82
128.35
127.87
125.43
83.62
81.90
77.42
77.00
76.58
49.99
28.00



4c

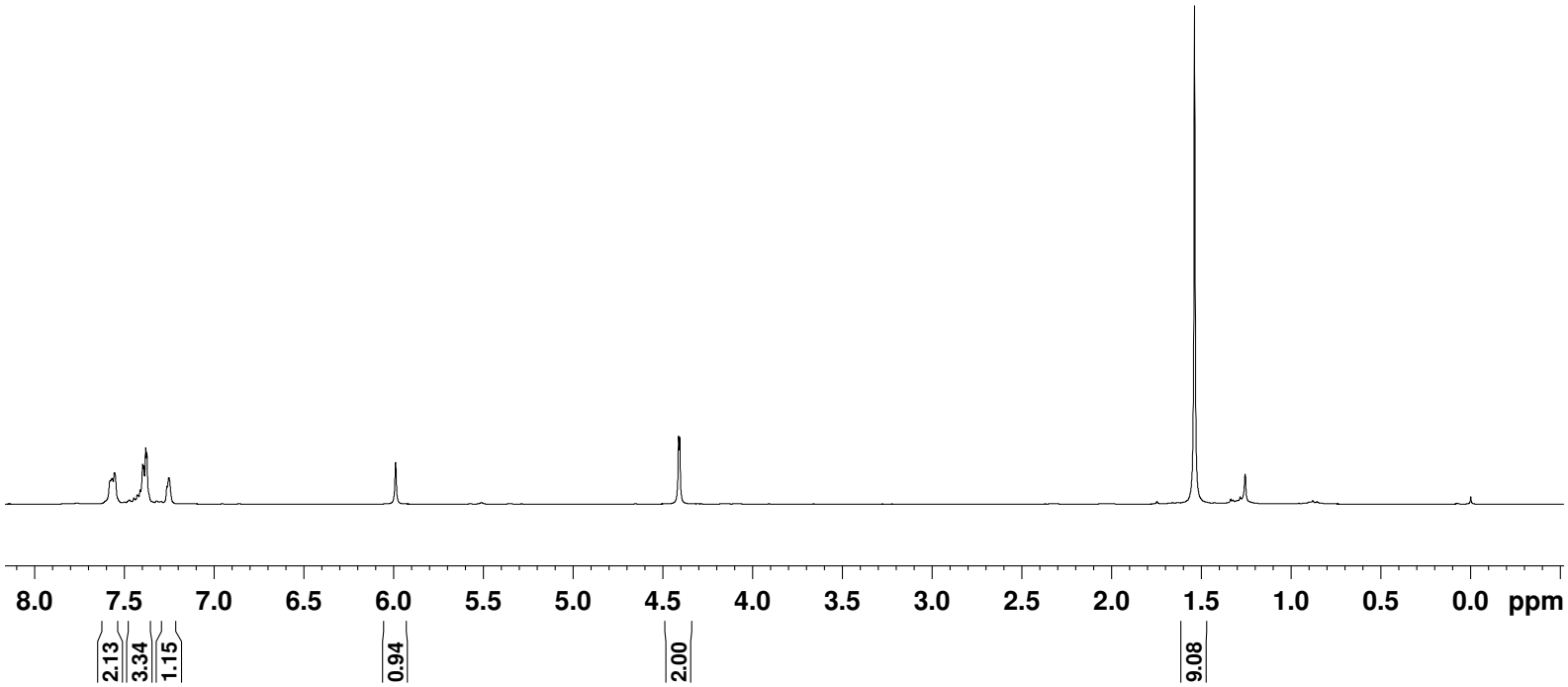
```
NAME           zhjl12.11
EXPNO                 70
PROCNO                 1
Date_           20121122
Time               10.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  202
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW                27.733 usec
DE                 6.50 usec
TE                288.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W        29.38907051 W
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677520 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



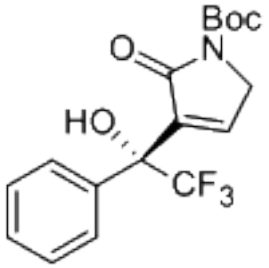


4d

```
NAME                 znj113.03
EXPNO                        8
PROCNO                       1
Date_                 20130302
Time                     16.48
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           2
SWH                 6188.119 Hz
FIDRES              0.094423 Hz
AQ                  5.2953587 sec
RG                          64
DW                     80.800 usec
DE                      6.50 usec
TE                     289.2 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      11.80 usec
PL1                      0.00 dB
PL1W               11.55467796 W
SFO1               300.1318534 MHz
SI                       32768
SF                 300.1300021 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.00
```

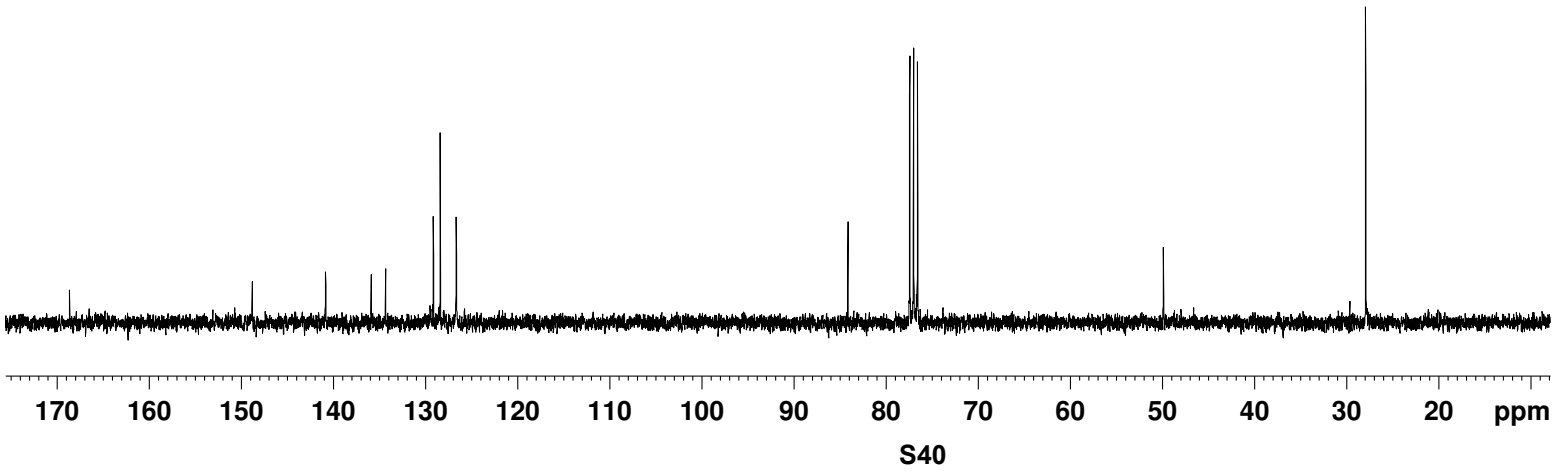


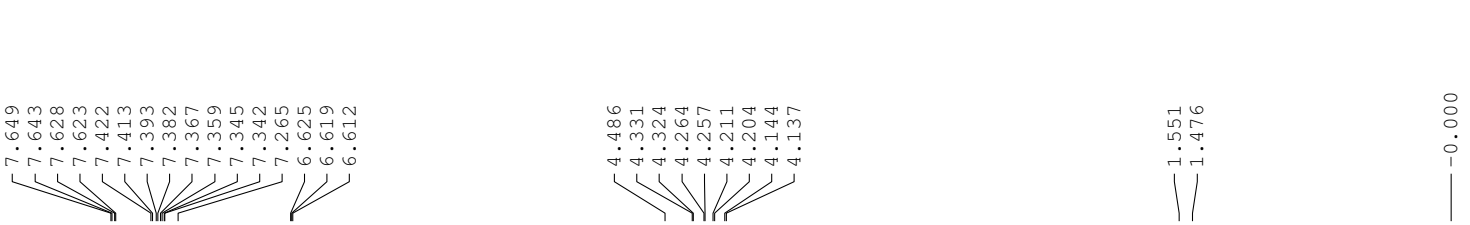
168.60
148.82
140.85
135.88
134.33
129.14
128.40
126.67
84.13
77.42
77.00
76.58
49.88
27.91

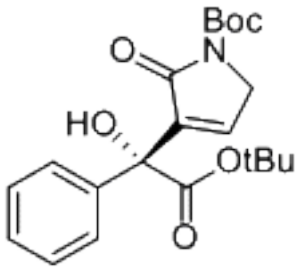


4d

```
NAME           zhjl13.03
EXPNO                  9
PROCNO                 1
Date_           20130302
Time               16.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   35
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW                27.733 usec
DE                 6.50 usec
TE                289.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W        29.38907051 W
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677531 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

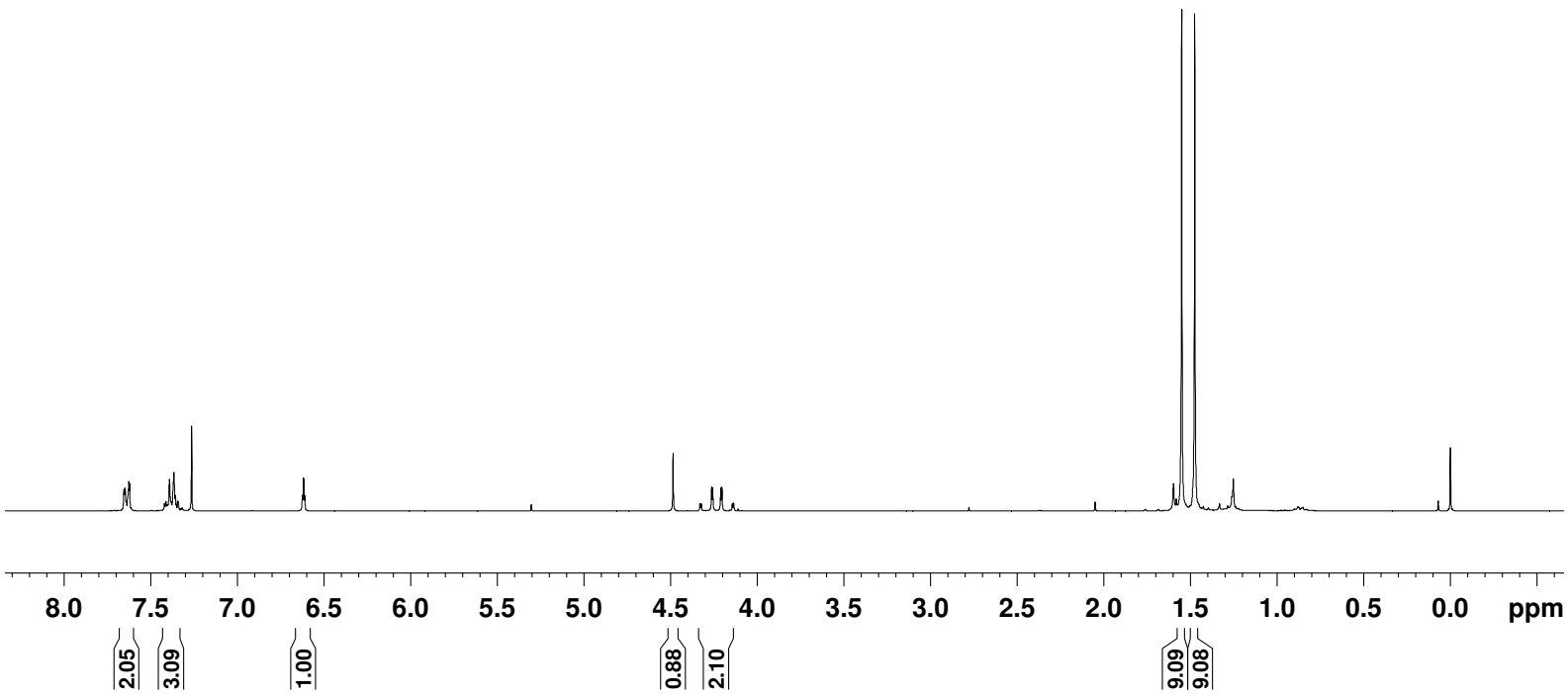


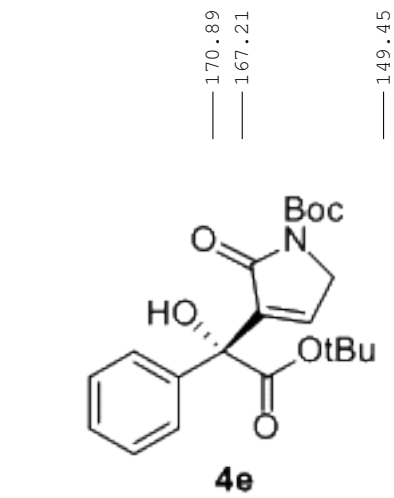




4e

```
NAME                 znj112.12
EXPNO                       33
PROCNO                       1
Date_                 20121211
Time                     16.52
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           2
SWH                 6188.119 Hz
FIDRES              0.094423 Hz
AQ                  5.2953587 sec
RG                         203
DW                    80.800 usec
DE                     6.50 usec
TE                     288.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     11.80 usec
PL1                     0.00 dB
PL1W             11.55467796 W
SFO1             300.1318534 MHz
SI                       32768
SF               300.1300012 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.00
```

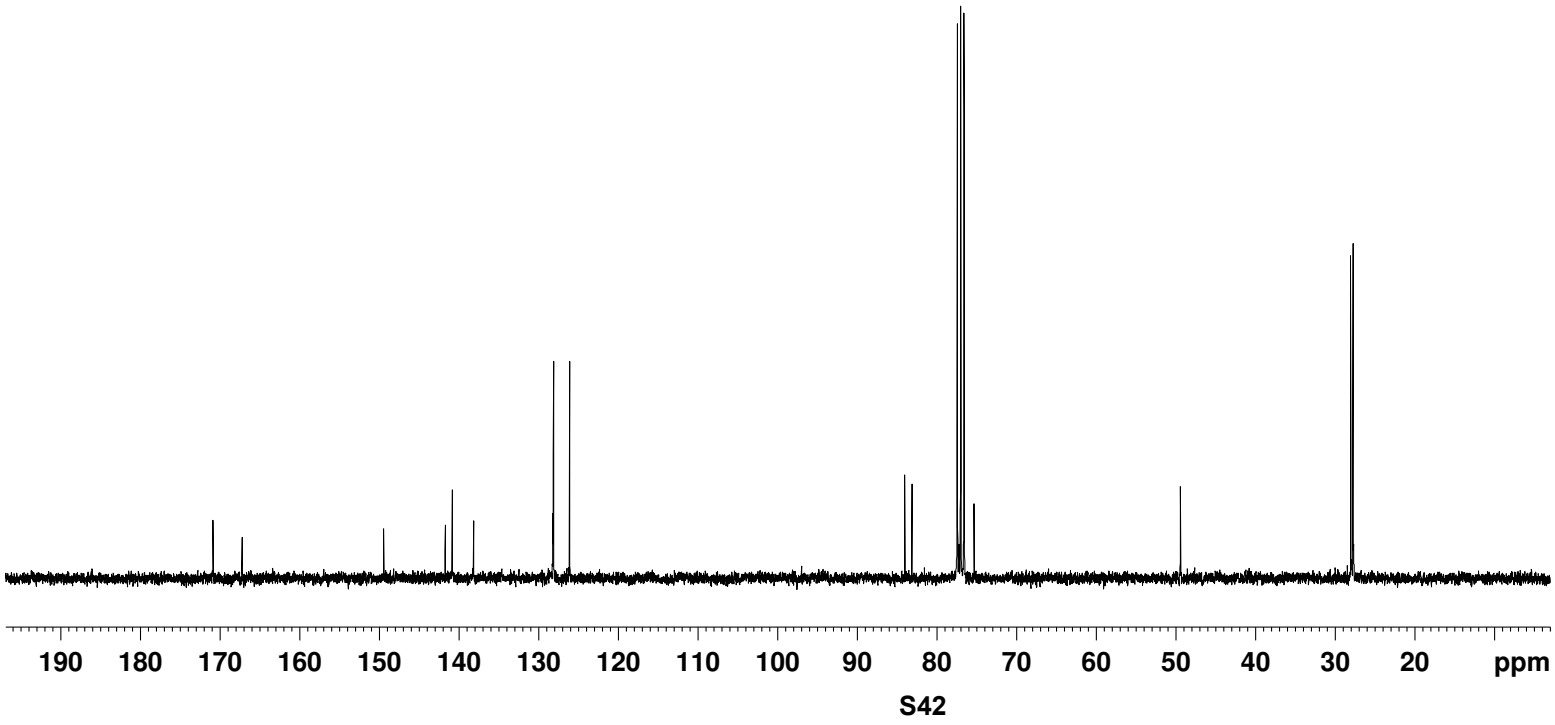


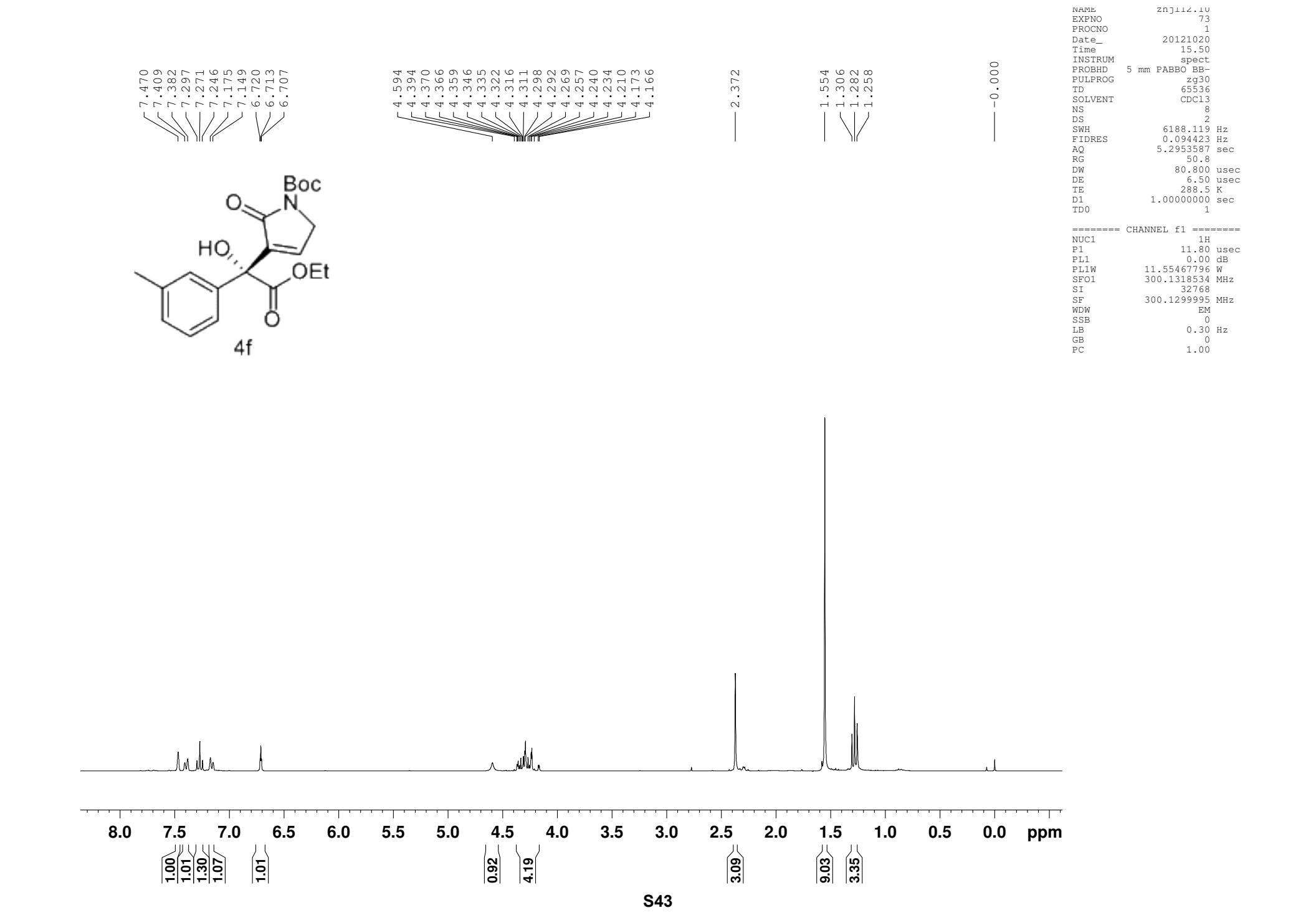


4e

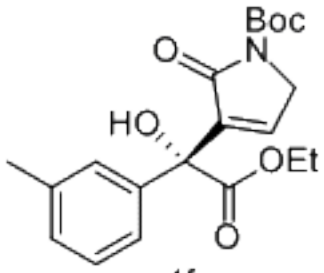
170.89
167.21
149.45
141.73
140.84
138.15
128.20
128.13
126.10
84.03
83.11
77.42
77.00
76.58
75.33
49.42
28.03
27.74

```
NAME           zhjl12.11
EXPNO                 62
PROCNO                 1
Date_           20121121
Time               21.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  179
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175818 sec
RG                  203
DW                27.733 usec
DE                 6.50 usec
TE                289.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W        29.38907051 W
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W         9.17820644 W
PL12W        0.23054613 W
PL13W        0.23054613 W
SFO2        300.1312005 MHz
SI                32768
SF           75.4677525 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



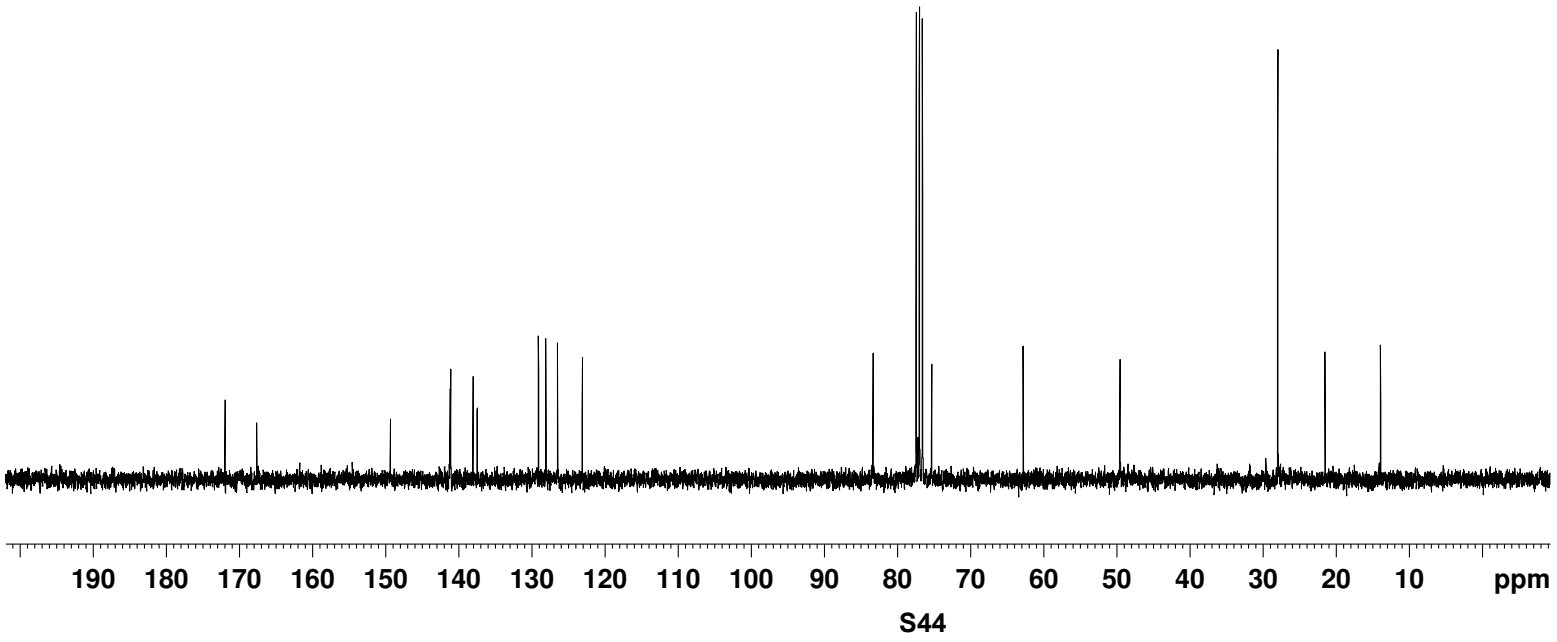

7.470
7.409
7.382
7.297
7.271
7.246
7.175
7.149
6.720
6.713
6.707
4.594
4.394
4.370
4.366
4.359
4.346
4.335
4.322
4.316
4.311
4.298
4.292
4.269
4.257
4.240
4.234
4.210
4.173
4.166
2.372
1.554
1.306
1.282
1.258
0.000
Boc
N
O
HO
OEt
O
4f
NAME zhj112.10
EXPNO 73
PROCNO 1
Date_ 20121020
Time 15.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 50.8
DW 80.800 usec
DE 6.50 usec
TE 288.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299995 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
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
0.5
0.0
ppm
1.00
1.01
1.30
1.07
1.01
0.92
4.19
3.09
9.03
3.35

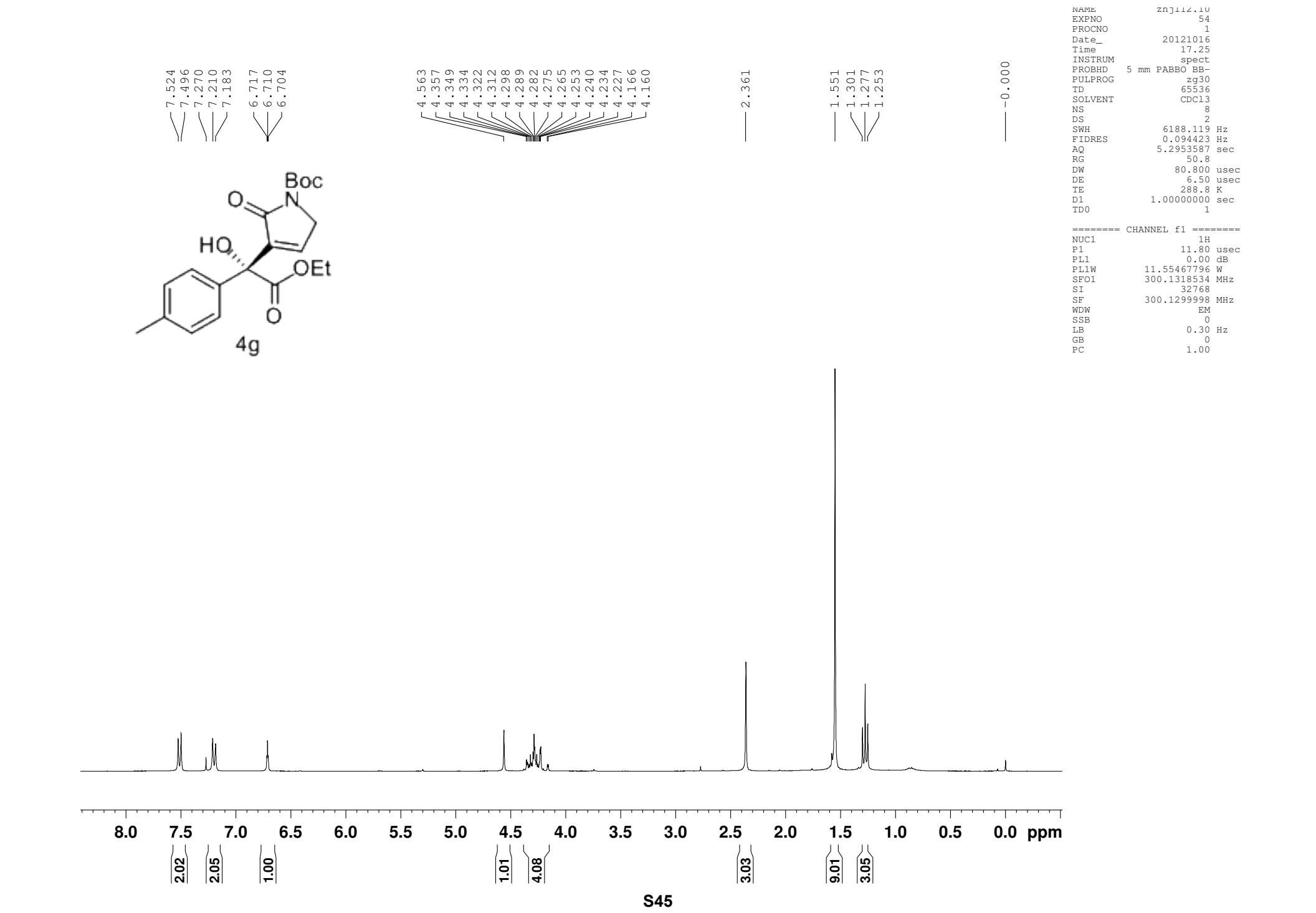
171.95
167.64
149.35
141.17
141.09
138.02
137.48
129.09
128.08
126.49
123.10
83.32
77.42
77.00
76.58
75.32
62.80
49.54
27.97
21.52
13.91

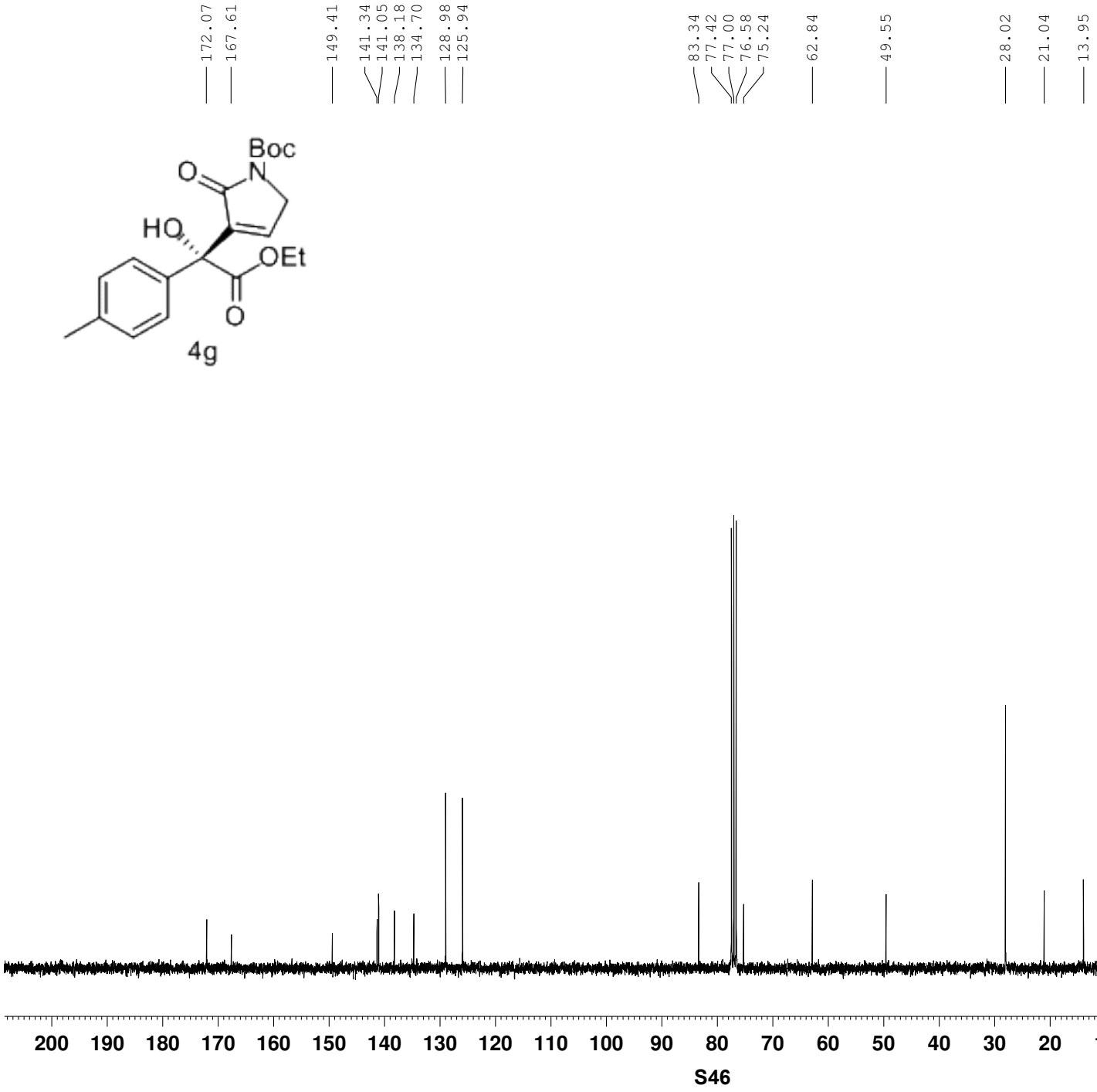


4f

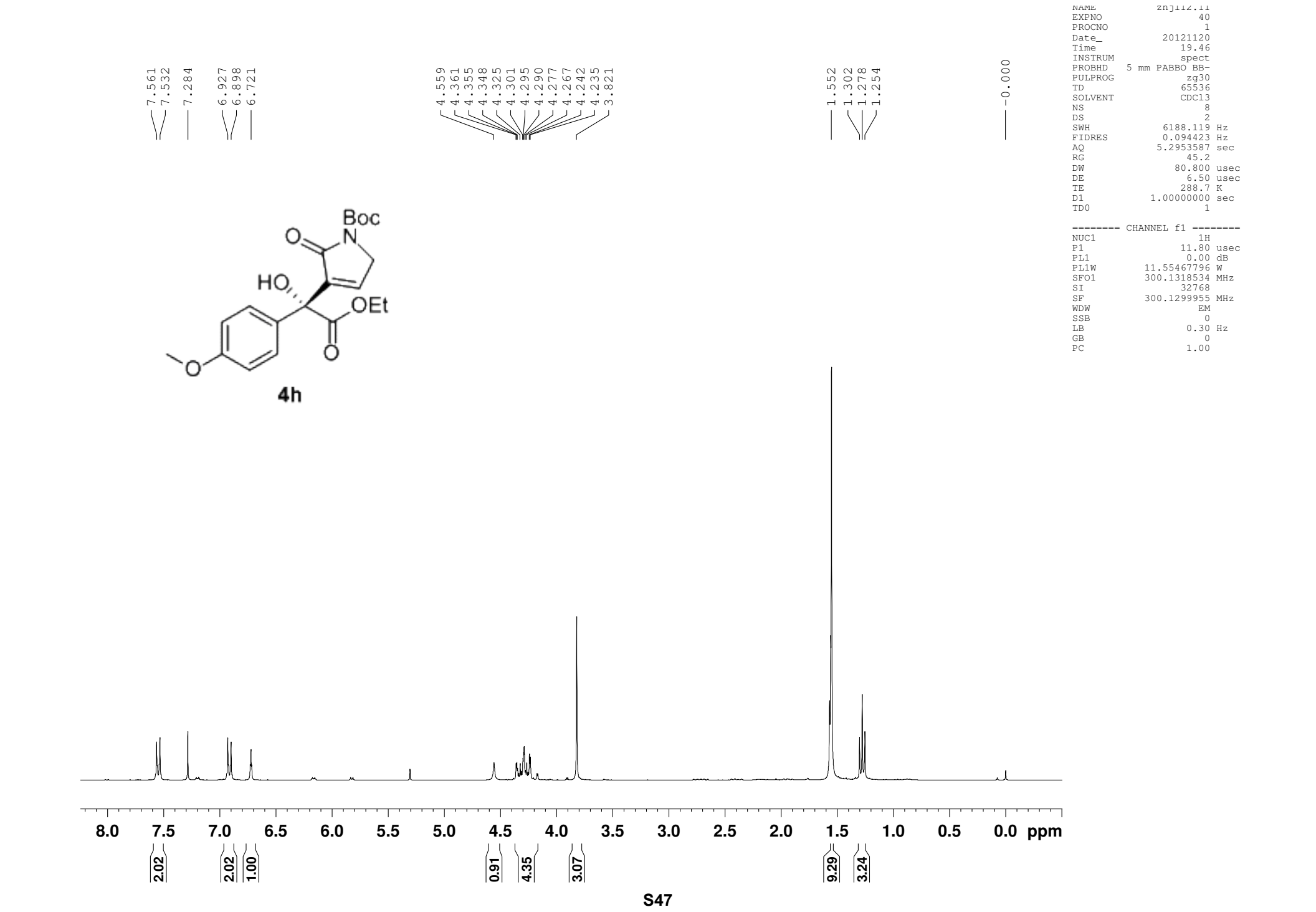
```
NAME            zhjl12.10
EXPNO                  70
PROCNO                  1
Date_            20121020
Time                10.47
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     52
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                288.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677547 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



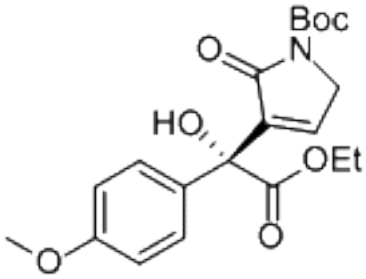




NAME	zhjl12.10
EXPNO	52
PROCNO	1
Date_	20121016
Time	16.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	169
DS	4
SWH	18028.846 Hz
FIDRES	0.275098 Hz
AQ	1.8175818 sec
RG	203
DW	27.733 usec
DE	6.50 usec
TE	289.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	0.00 dB
PL1W	29.38907051 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.00 dB
PL13	17.00 dB
PL2W	9.17820644 W
PL12W	0.23054613 W
PL13W	0.23054613 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677520 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

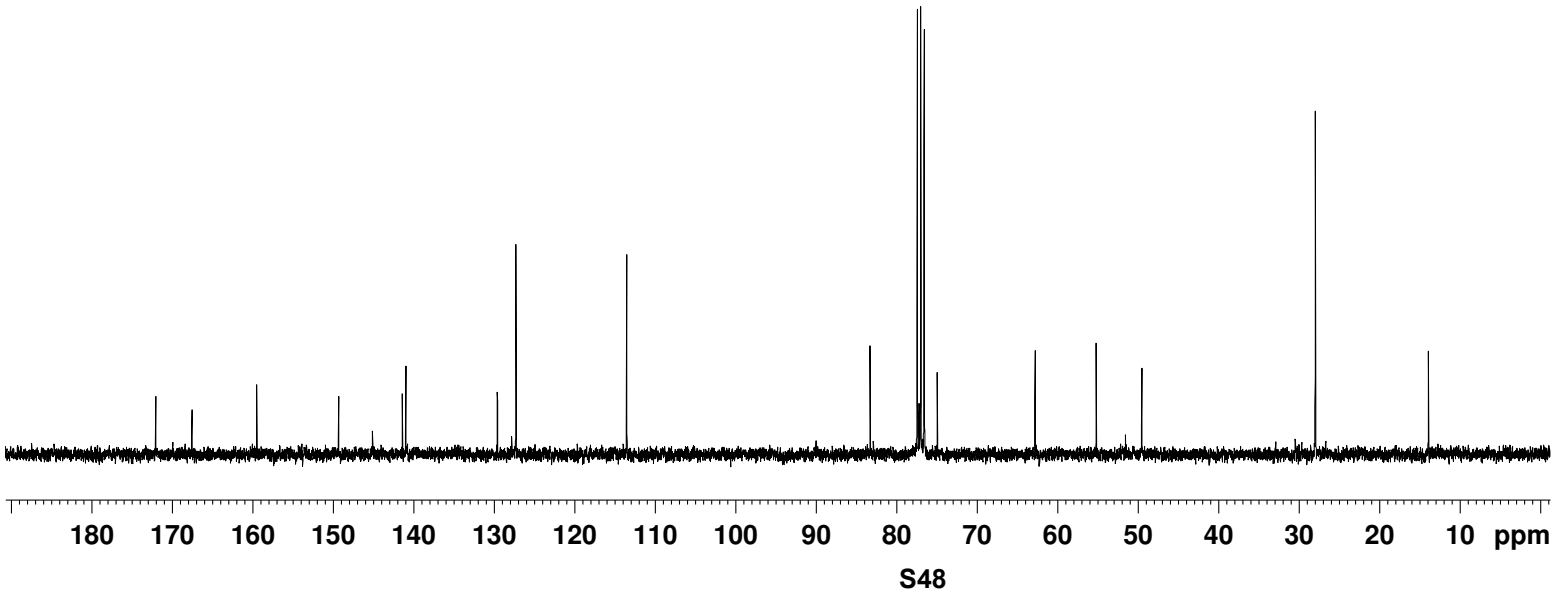


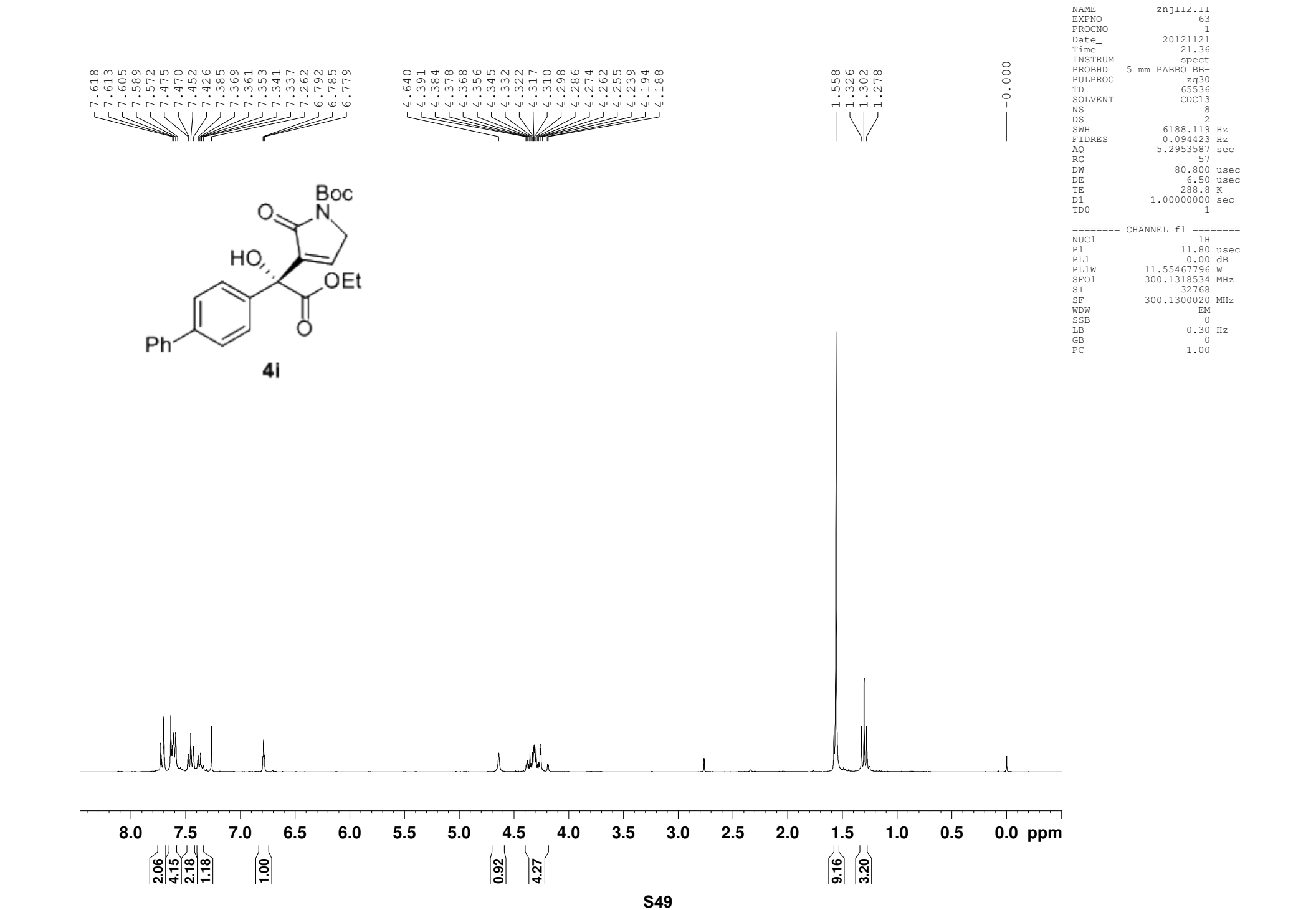
172.07
167.57
159.51
149.34
145.12
141.42
140.98
129.61
127.82
127.30
113.54
83.28
77.42
77.20
77.00
76.57
74.94
62.79
55.21
49.52
27.97
13.92

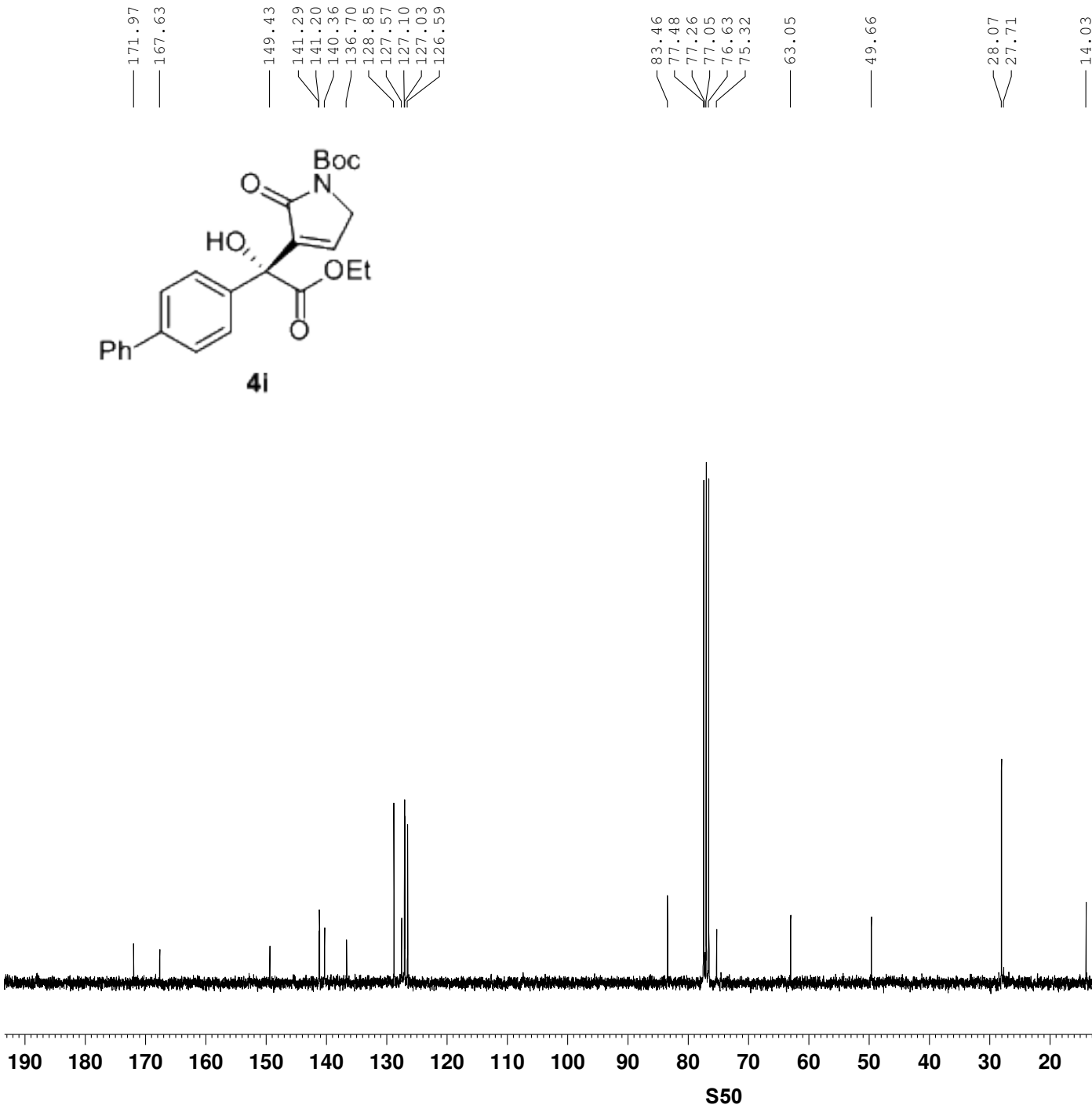


4h

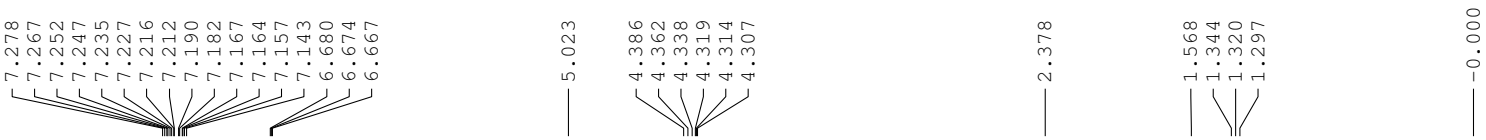
```
NAME            zhjl12.11
EXPNO                  41
PROCNO                  1
Date_            20121120
Time                19.52
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     82
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677547 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

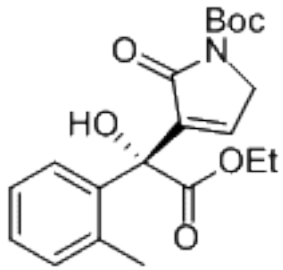



7.618
7.613
7.605
7.589
7.572
7.475
7.470
7.452
7.426
7.385
7.369
7.361
7.353
7.341
7.337
7.262
6.792
6.785
6.779
4.640
4.391
4.384
4.378
4.368
4.356
4.345
4.332
4.322
4.317
4.310
4.298
4.286
4.274
4.262
4.255
4.239
4.194
4.188
1.558
1.326
1.302
1.278
0.000
Boc
N
O
HO
OEt
O
Ph
4l
NAME zhjll2.11
EXPNO 63
PROCNO 1
Date_ 20121121
Time 21.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 57
DW 80.800 usec
DE 6.50 usec
TE 288.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
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 0.5 0.0 ppm
2.06
4.15
2.18
1.18
1.00
0.92
4.27
9.16
3.20



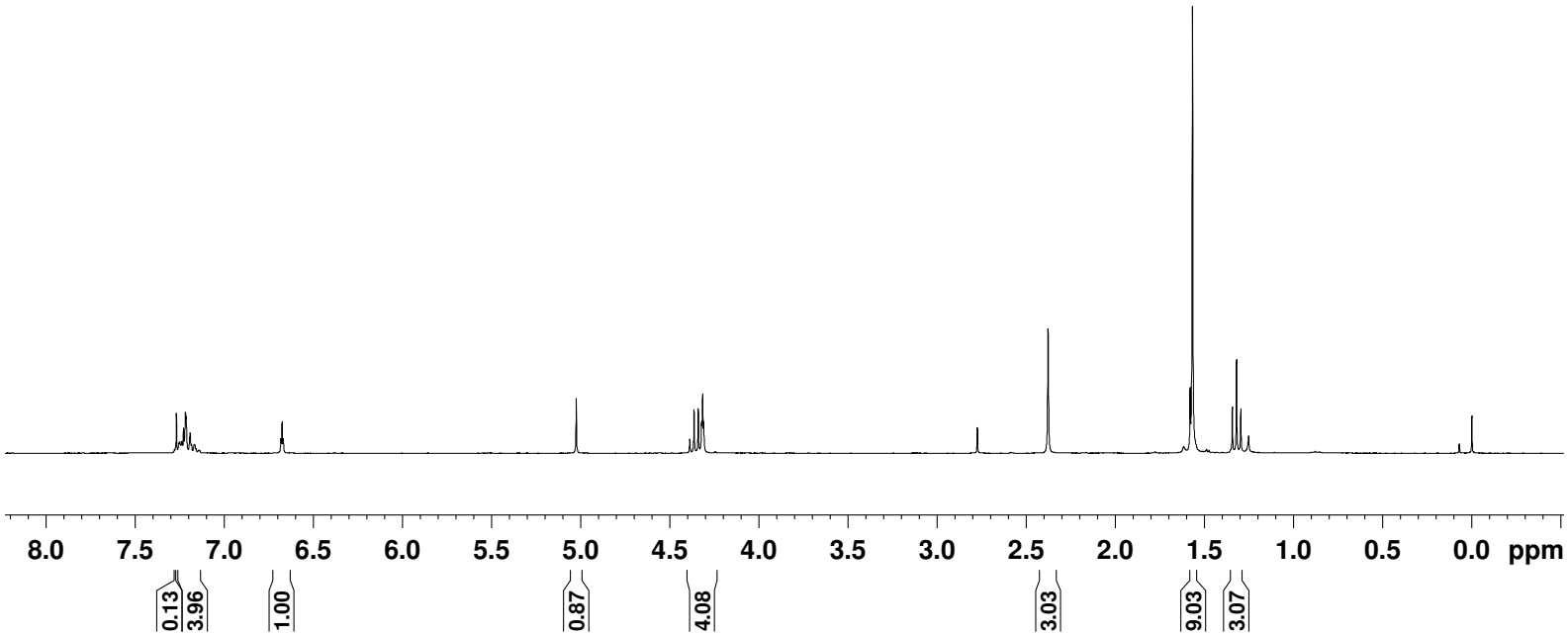

```
NAME            zhjl12.11
EXPNO                  64
PROCNO                  1
Date_            20121121
Time                21.40
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    134
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677531 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

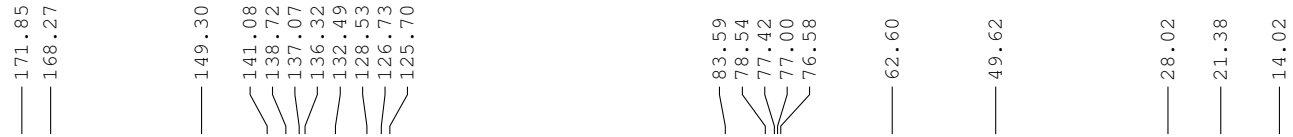


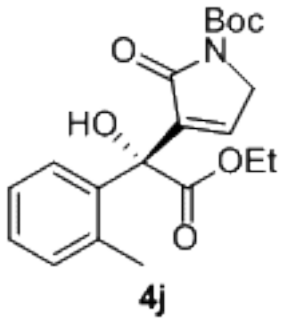


4j

```
NAME                 znjl12.12
EXPNO                       38
PROCNO                       1
Date_                 20121211
Time                     19.27
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          2
SWH                 6188.119 Hz
FIDRES             0.094423 Hz
AQ                 5.2953587 sec
RG                        161
DW                   80.800 usec
DE                    6.50 usec
TE                    288.1 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    11.80 usec
PL1                    0.00 dB
PL1W           11.55467796 W
SFO1           300.1318534 MHz
SI                      32768
SF             300.1300007 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```

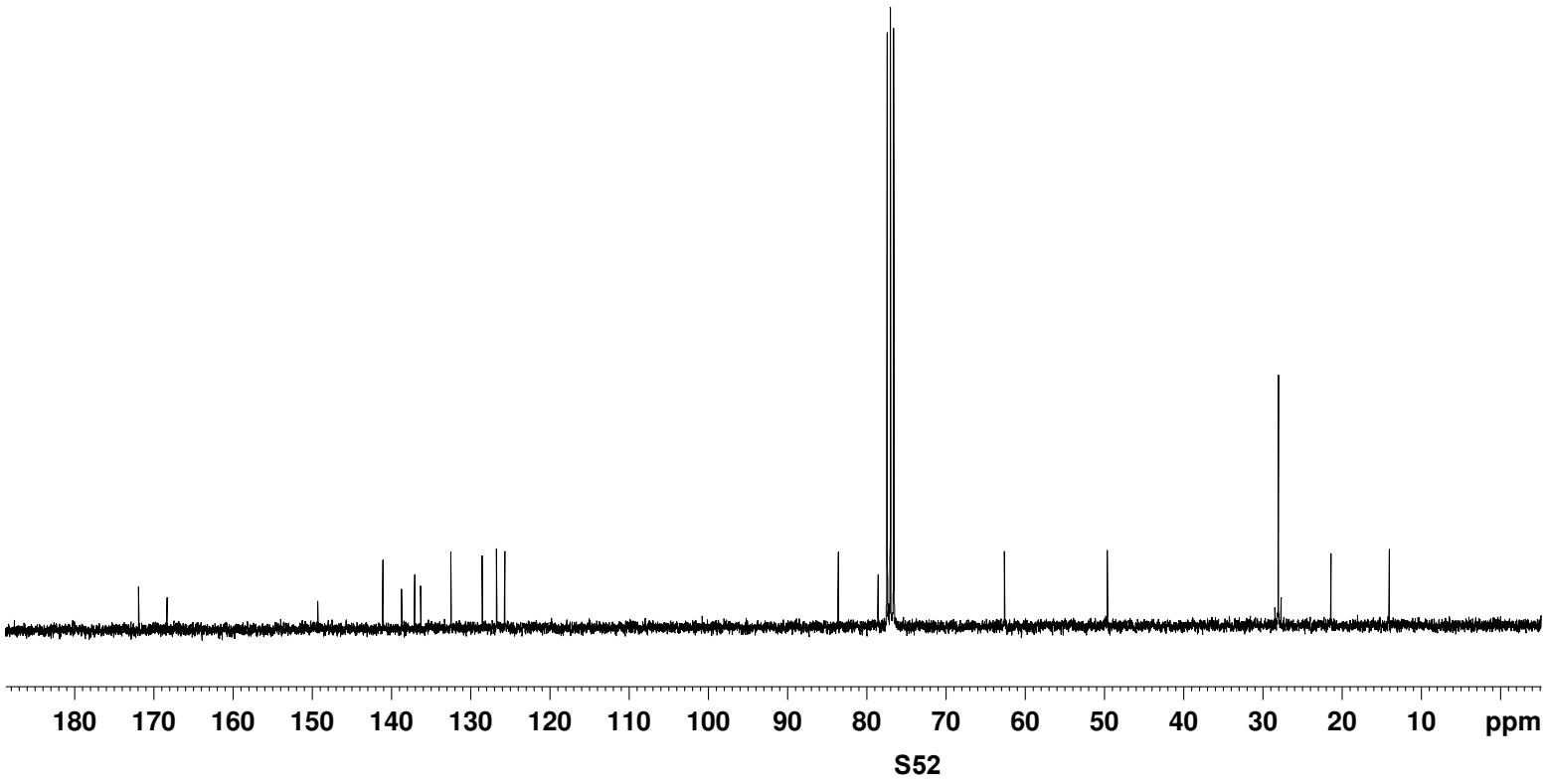


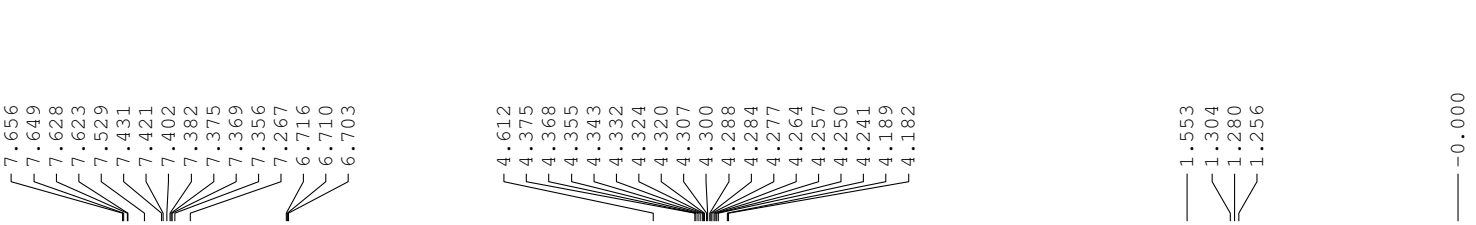


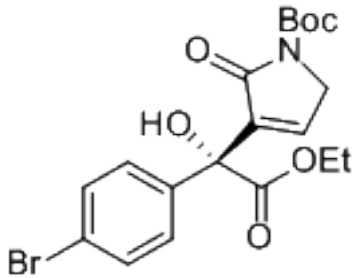


4j

```
NAME            zhjl12.11
EXPNO                  50
PROCNO                  1
Date_            20121120
Time                22.17
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    182
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677520 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

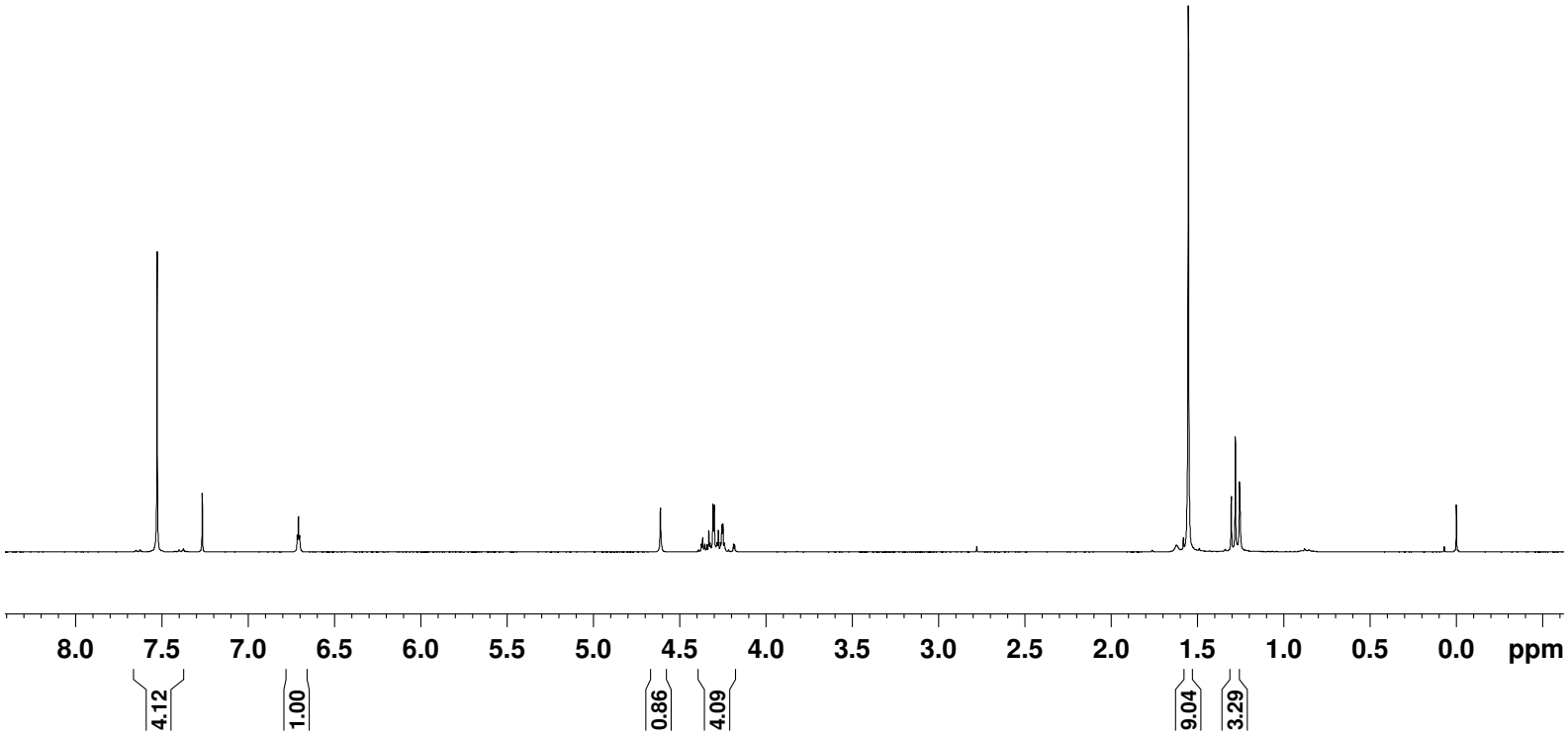


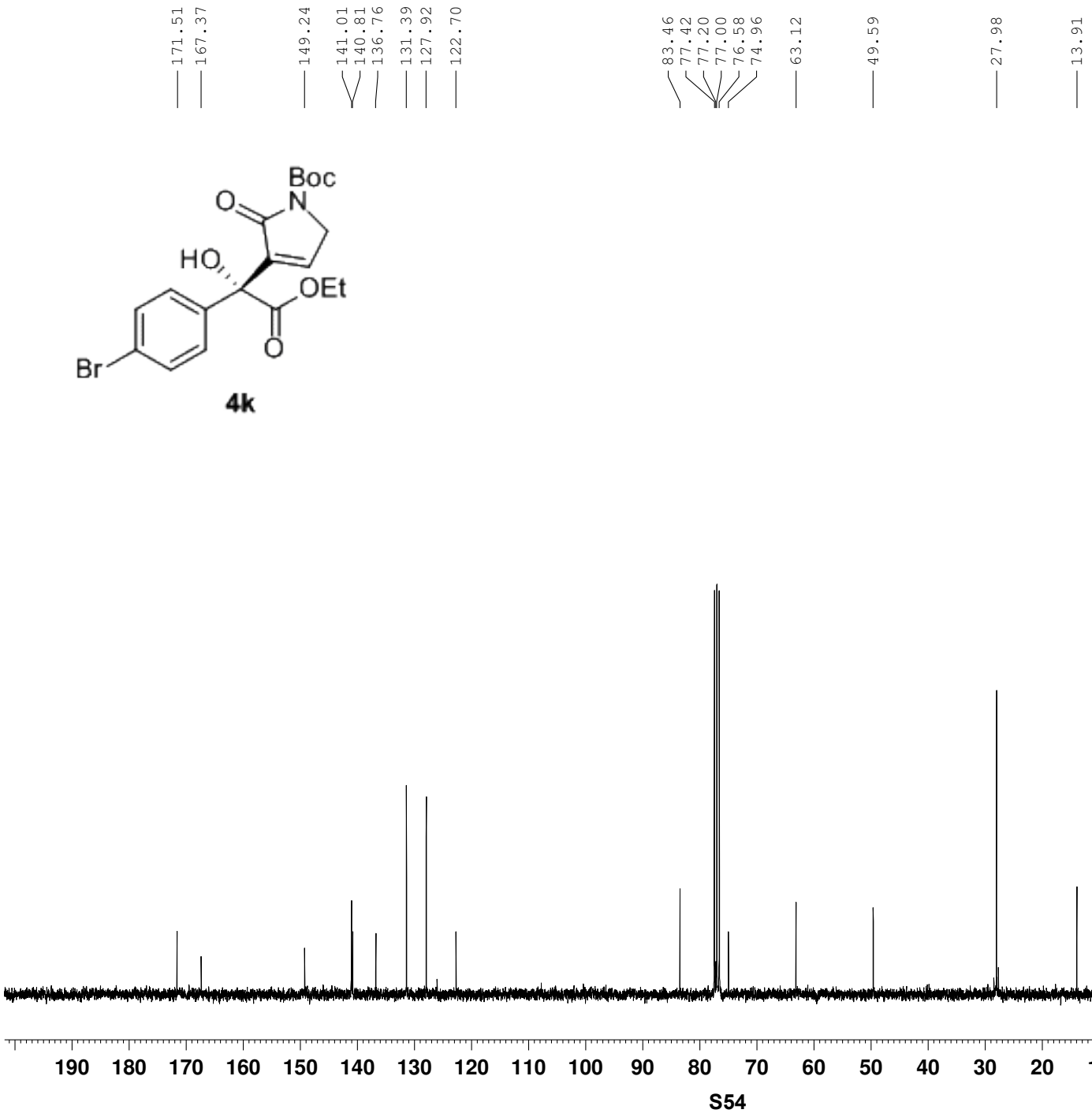




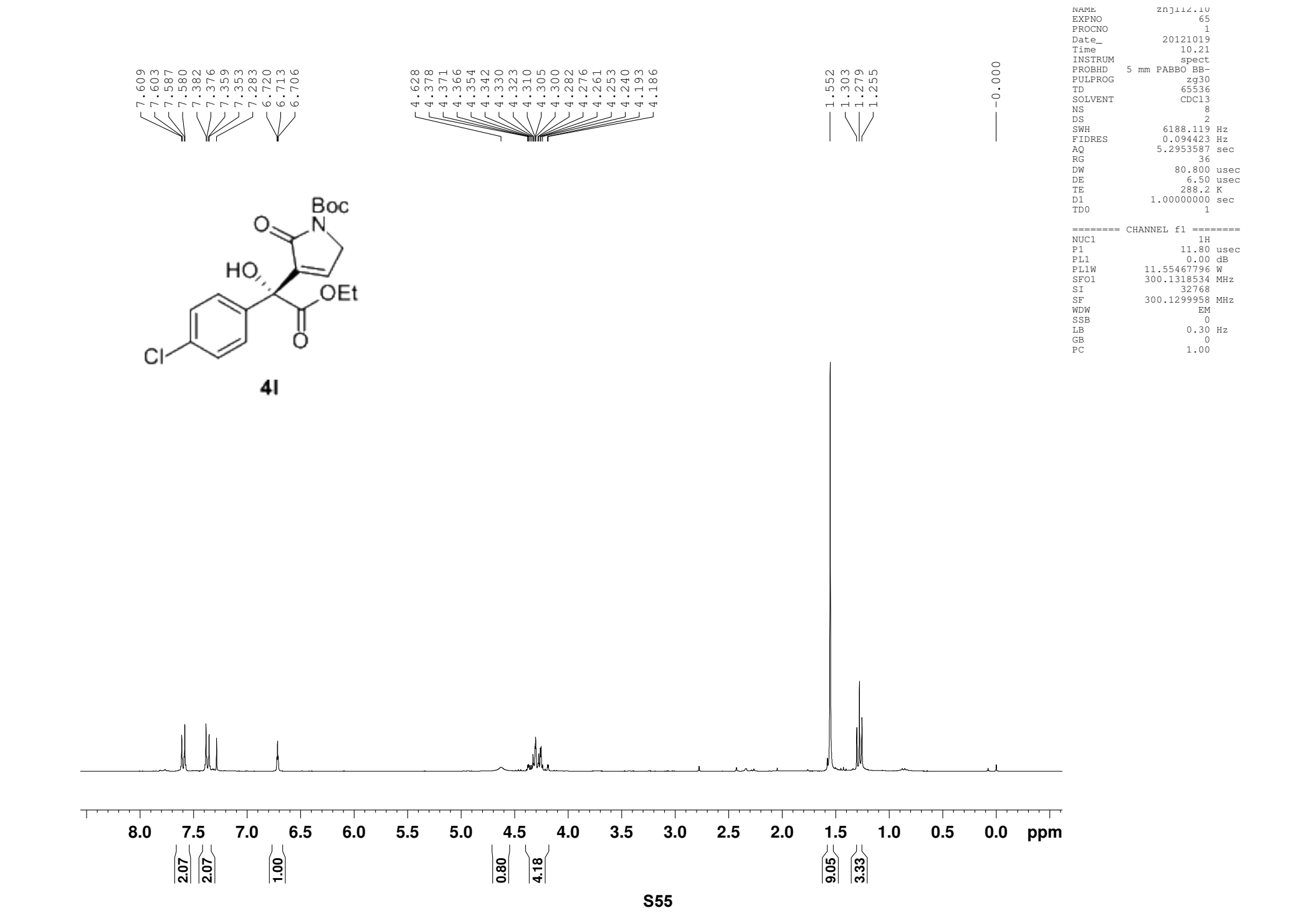
4k

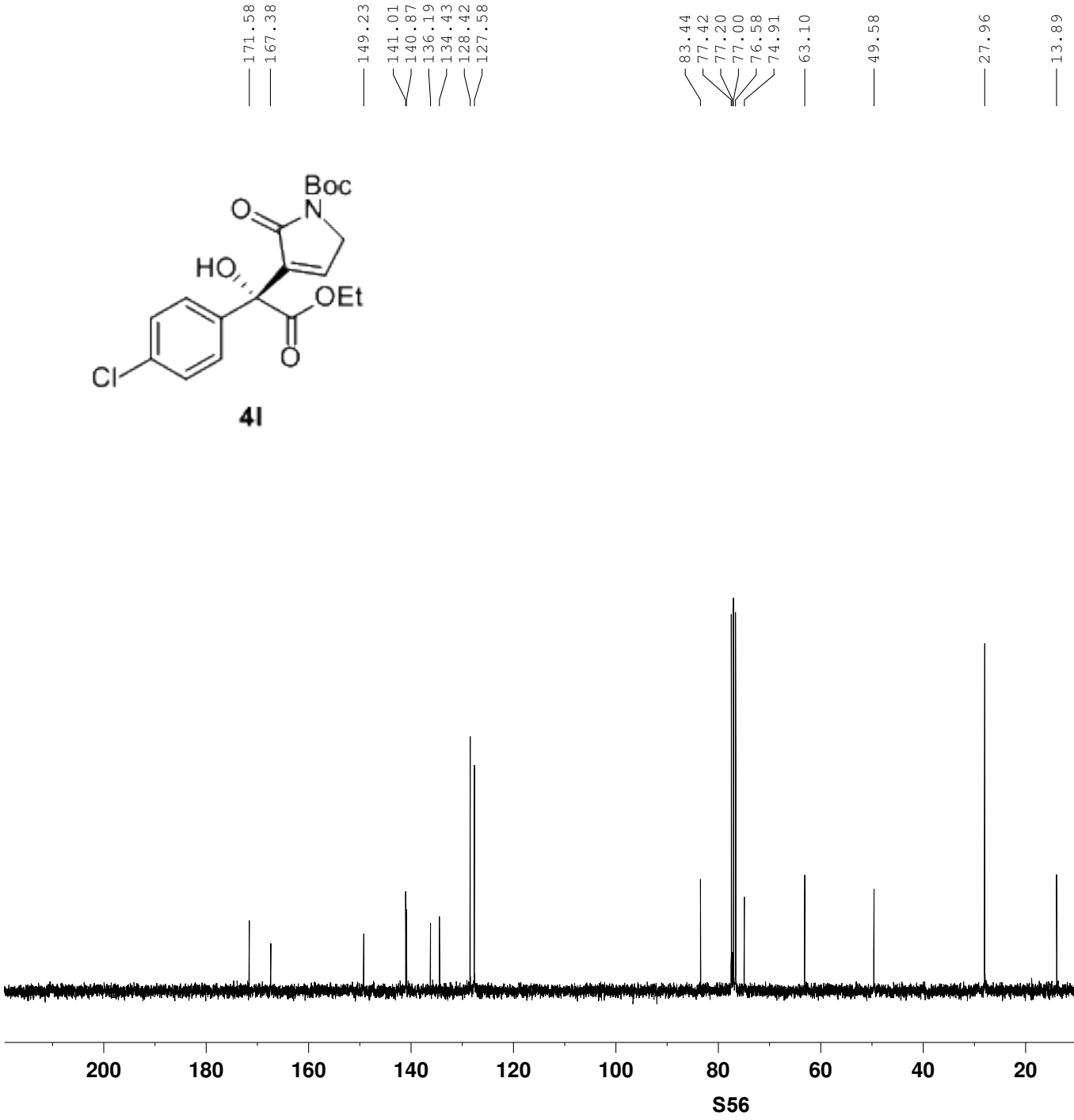
```
NAME            znjl12.12
EXPNO                  31
PROCNO                  1
Date_            20121211
Time                15.32
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                    203
DW                 80.800 usec
DE                  6.50 usec
TE                 288.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1300006 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```



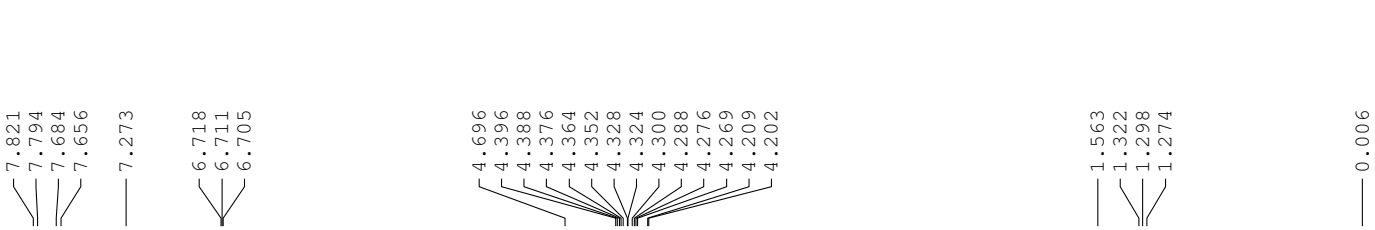


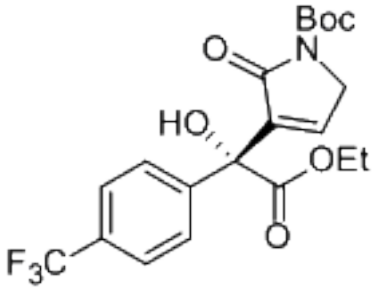

```
NAME            zhjl12.11
EXPNO                  46
PROCNO                  1
Date_            20121120
Time                20.54
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    104
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677539 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```





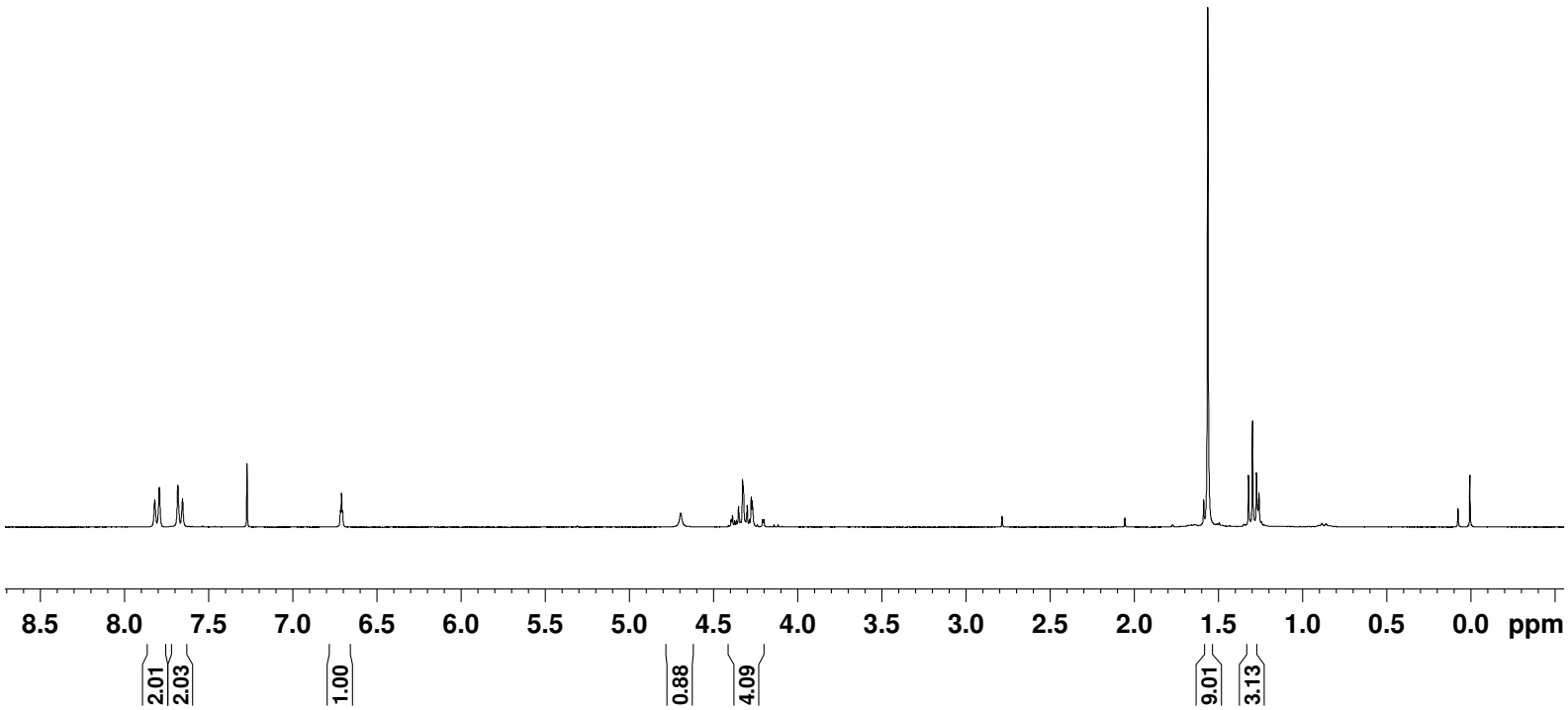

```
NAME            zhjl12.10
EXPNO                  66
PROCNO                  1
Date_            20121019
Time                10.25
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     58
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                 27.733 usec
DE                  6.50 usec
TE                 288.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                  0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                17.00 dB
PL13                17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                  32768
SF            75.4677547 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



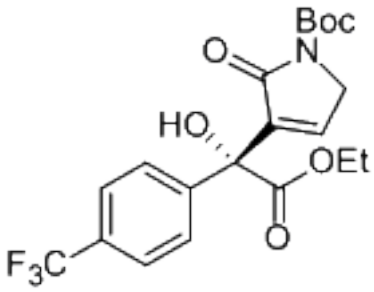


4m

```
NAME            znjl12.12
EXPNO                  36
PROCNO                  1
Date_            20121211
Time                19.15
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH           6188.119 Hz
FIDRES          0.094423 Hz
AQ             5.2953587 sec
RG                    203
DW               80.800 usec
DE                6.50 usec
TE                288.2 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1299987 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

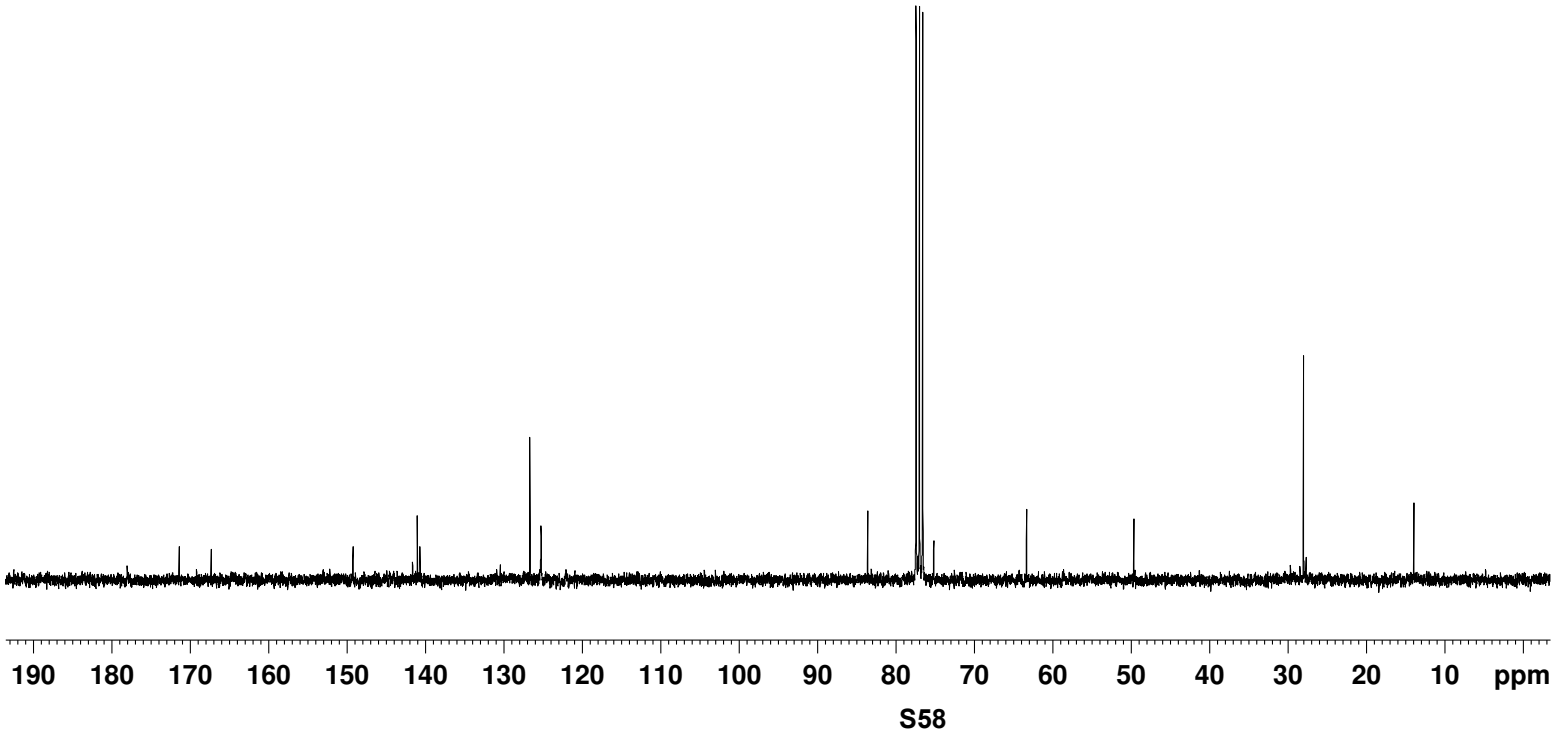


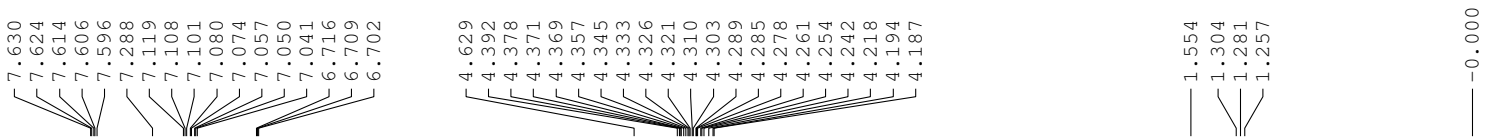
171.35
167.34
149.25
141.67
141.04
140.71
126.68
125.30
125.25
83.60
77.42
77.00
76.57
75.17
63.33
49.65
28.01
27.66
13.93

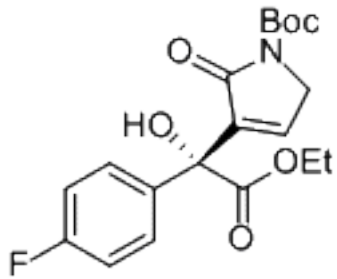


4m

```
NAME            zhjl12.11
EXPNO                  38
PROCNO                  1
Date_            20121120
Time                9.47
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    190
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677514 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

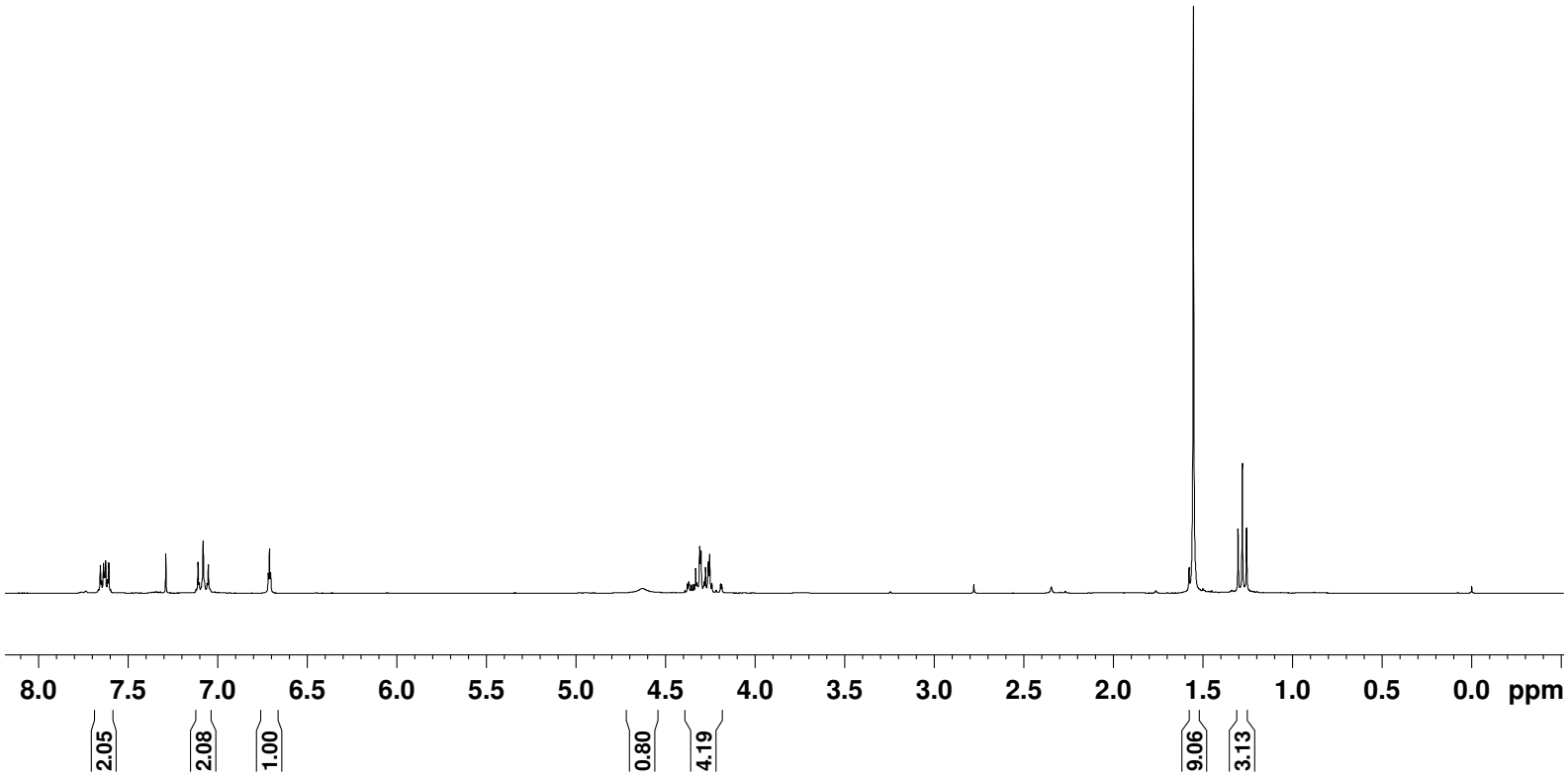


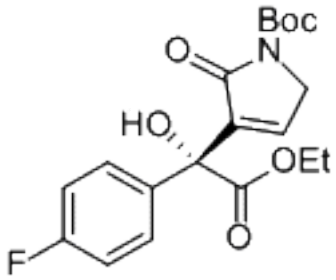




4n

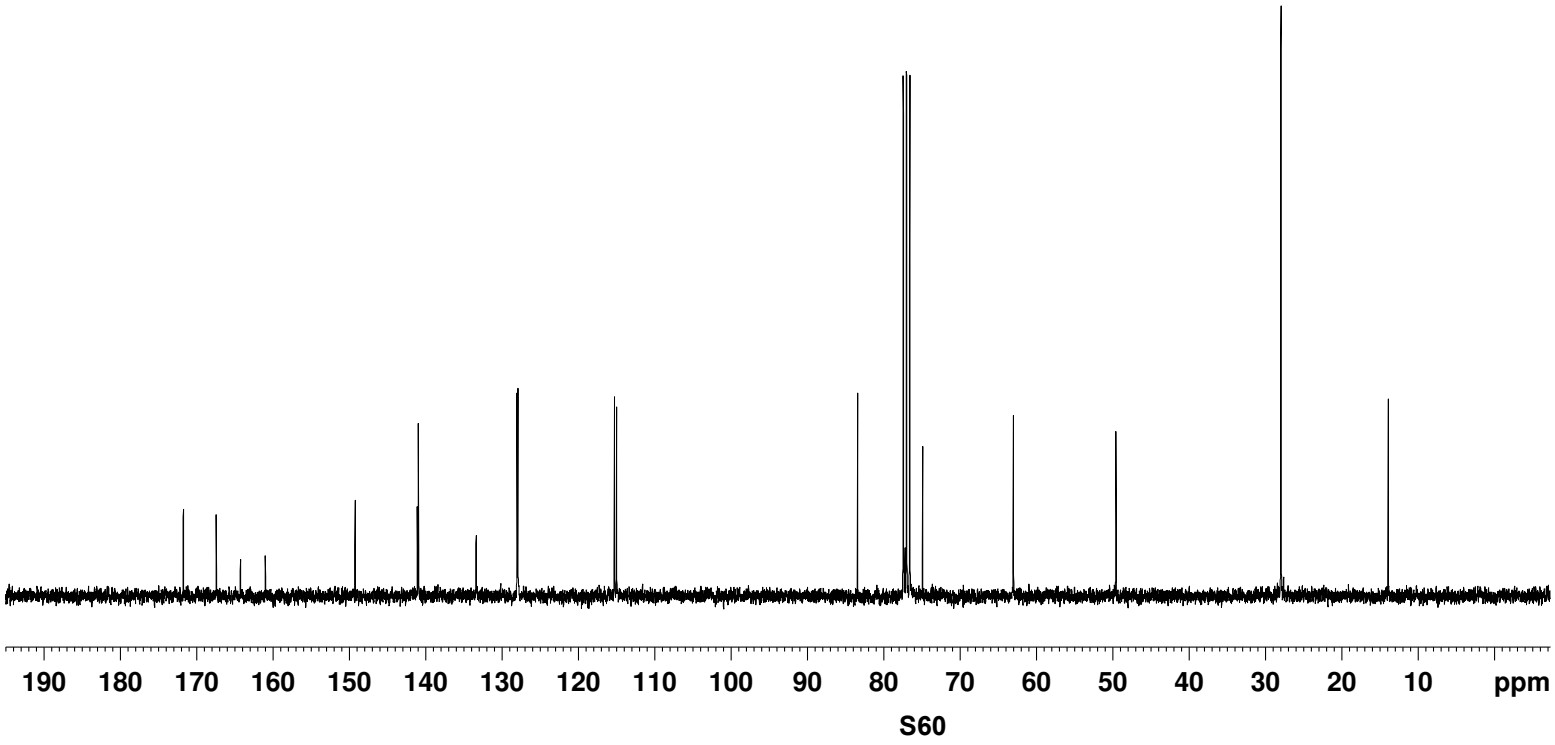
```
NAME            znjl12.10
EXPNO                  60
PROCNO                  1
Date_            20121018
Time                10.16
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                     36
DW                 80.800 usec
DE                  6.50 usec
TE                 288.4 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1299943 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```

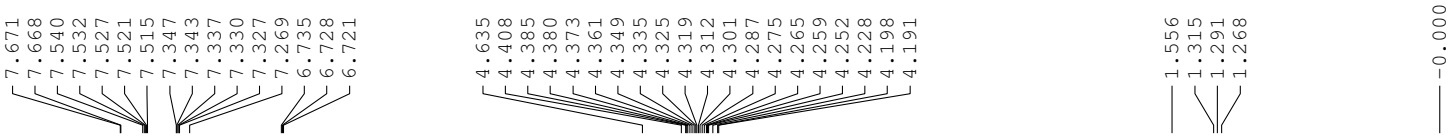


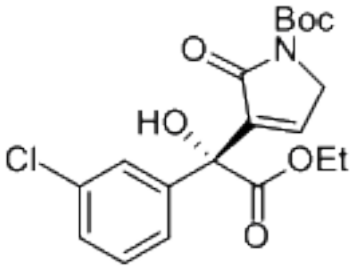


4n

```
NAME            zhjl12.10
EXPNO                  61
PROCNO                  1
Date_            20121018
Time                10.22
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     99
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                288.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677550 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

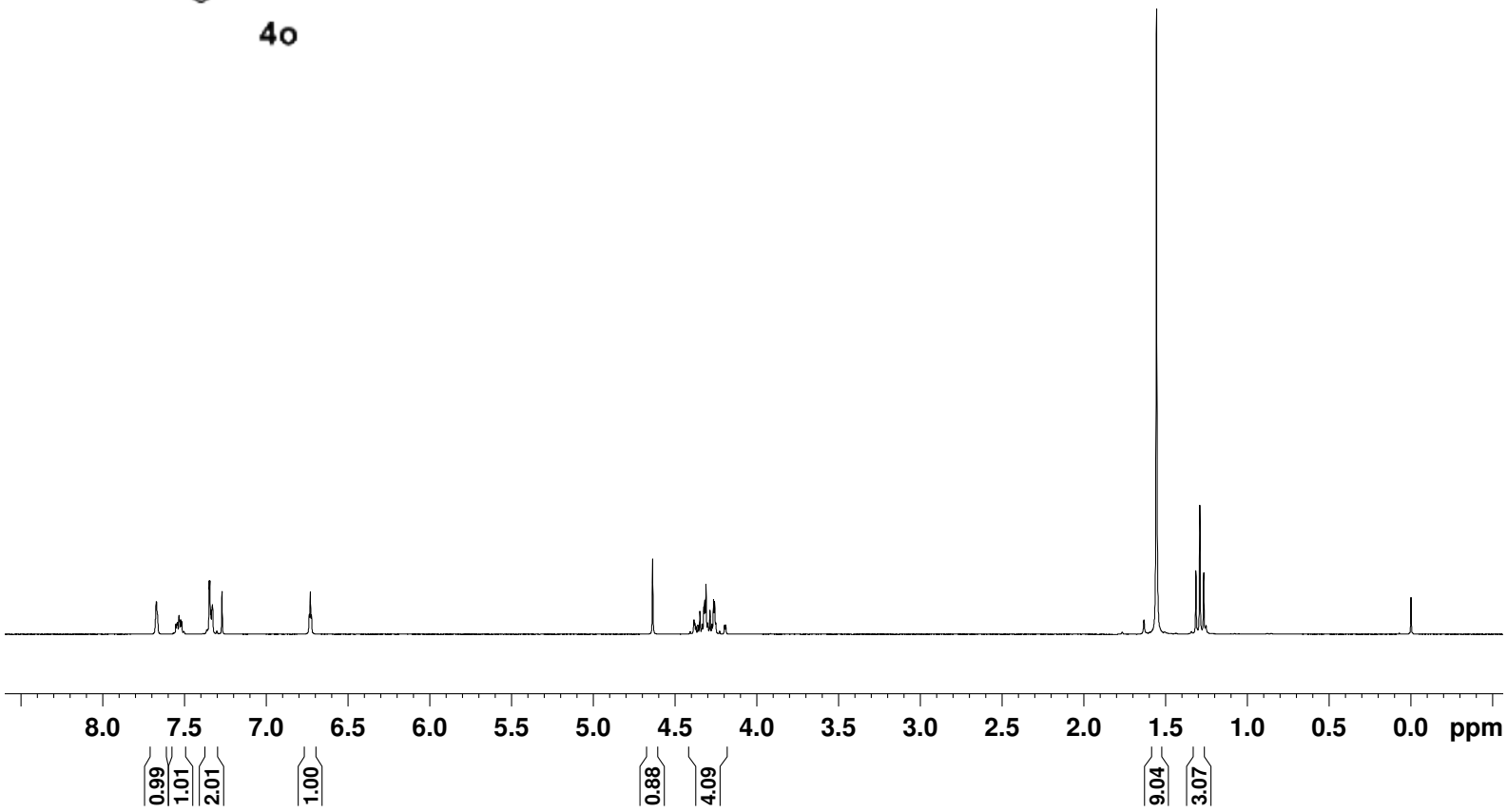




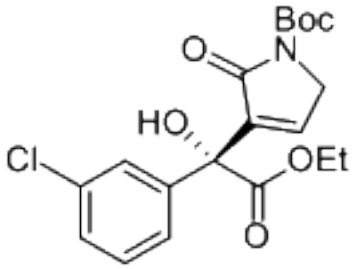


4o

```
NAME            znjl12.12
EXPNO                  43
PROCNO                  1
Date_            20121212
Time                16.20
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                    181
DW                 80.800 usec
DE                  6.50 usec
TE                 288.3 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1300002 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```

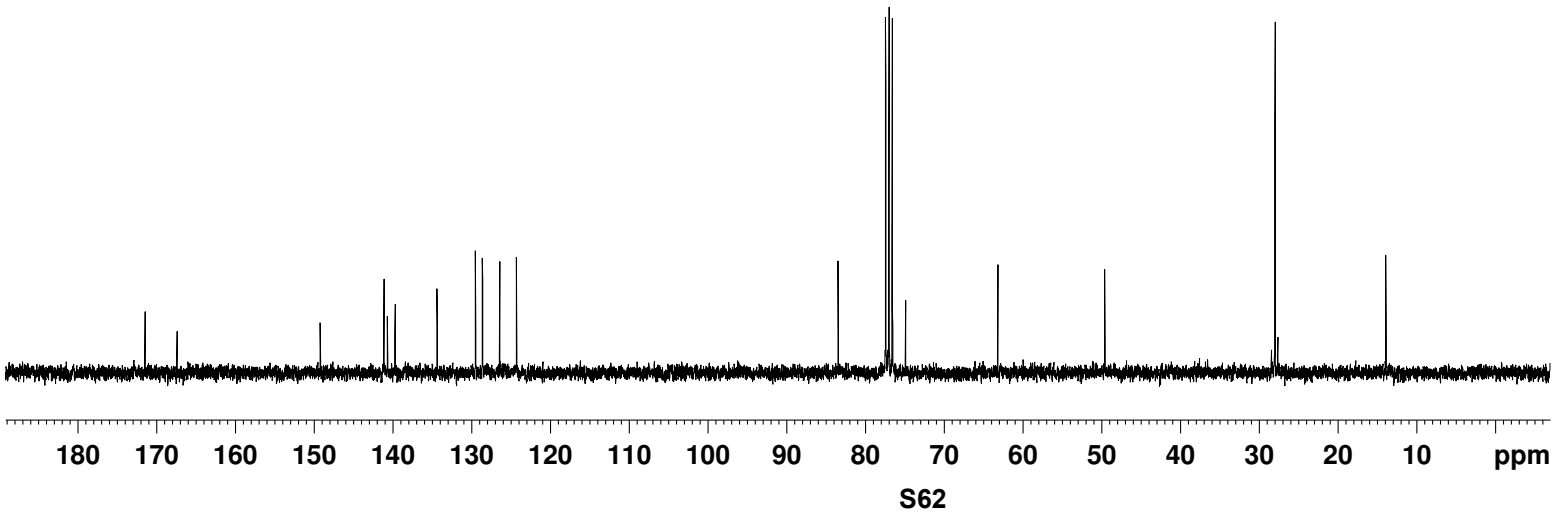


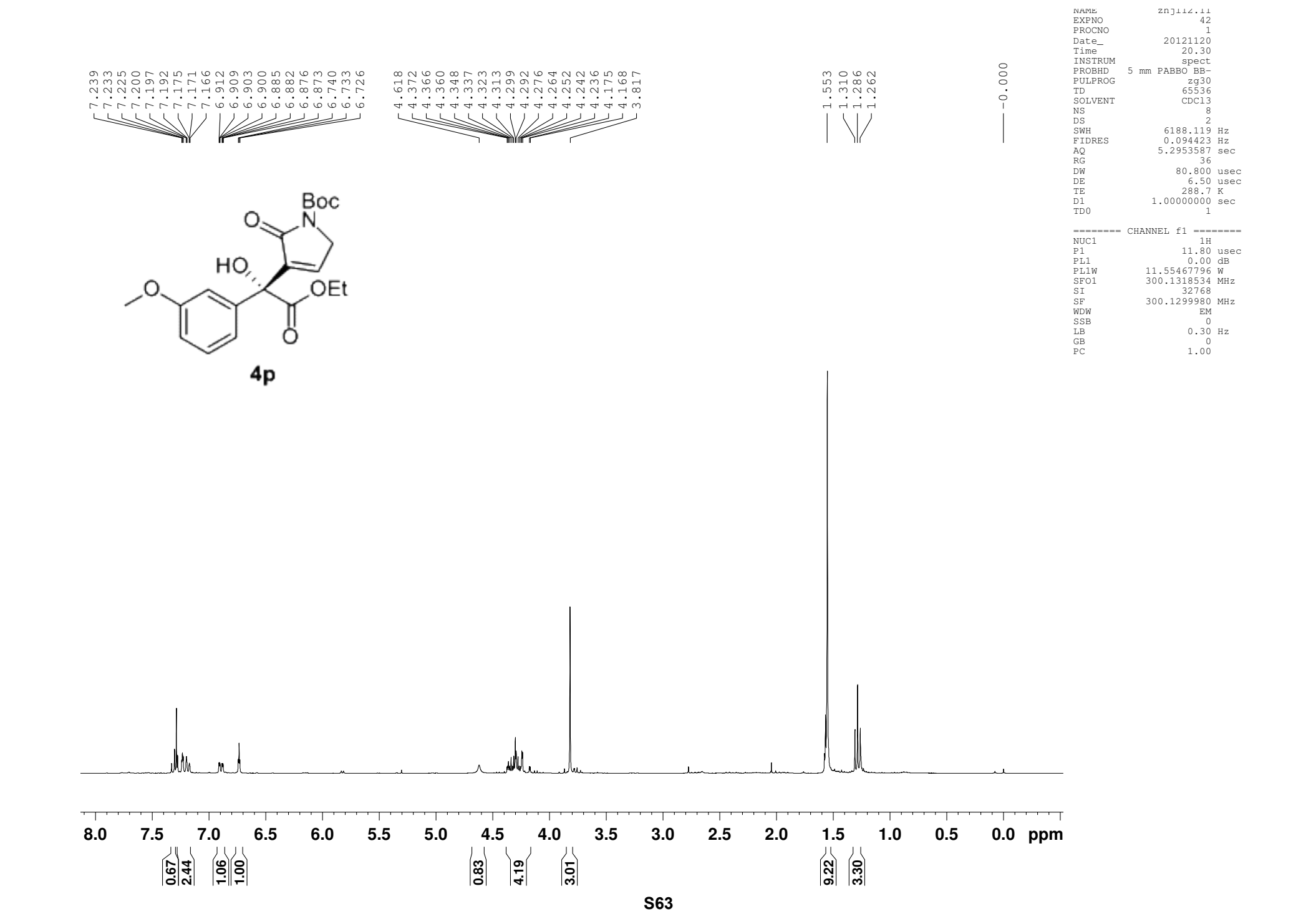
171.41
167.35
149.23
141.13
140.69
139.73
134.38
129.51
128.62
126.42
124.32
83.46
77.42
77.00
76.58
74.88
63.17
49.59
27.97
13.89



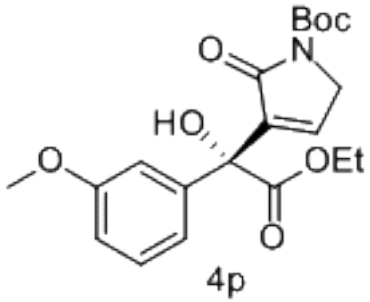
4o

```
NAME            zhjl12.11
EXPNO                  48
PROCNO                  1
Date_            20121120
Time                22.03
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     55
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                  0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                  1.00 dB
PL12                17.00 dB
PL13                17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                  32768
SF            75.4677542 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



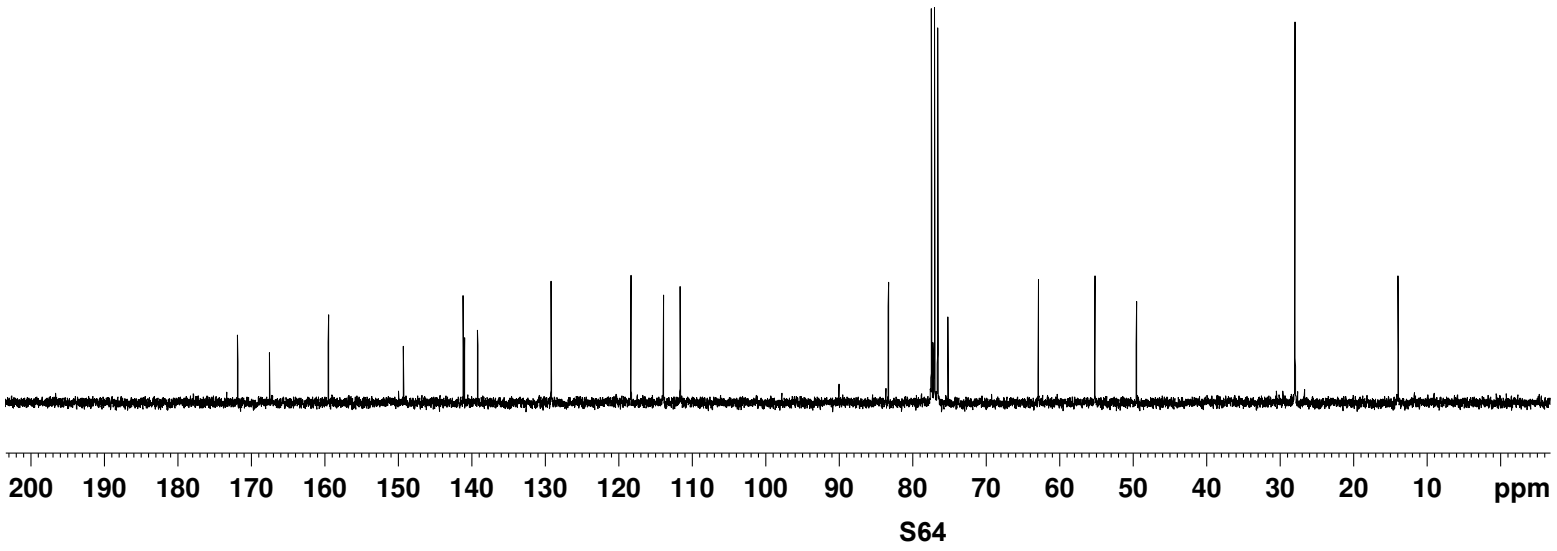

7.239
7.233
7.225
7.200
7.197
7.192
7.175
7.171
7.166
6.912
6.909
6.903
6.900
6.885
6.882
6.876
6.873
6.740
6.733
6.726
4.618
4.372
4.366
4.360
4.348
4.337
4.323
4.313
4.299
4.292
4.276
4.264
4.252
4.242
4.236
4.175
4.168
3.817
1.553
1.310
1.286
1.262
0.000
Boc
N
O
HO
OEt
O
O
4p
NAME zhjl12.11
EXPNO 42
PROCNO 1
Date_ 20121120
Time 20.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 36
DW 80.800 usec
DE 6.50 usec
TE 288.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299980 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
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 0.5 0.0 ppm
0.67
2.44
1.06
1.00
0.83
4.19
3.01
9.22
3.30

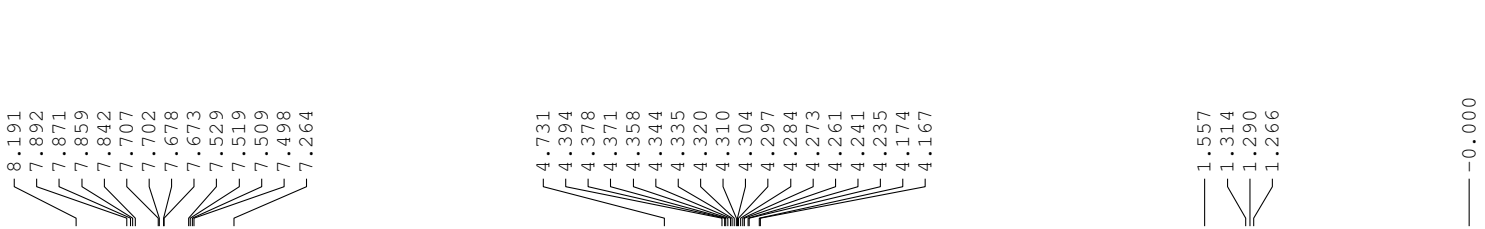
171.79
167.53
159.51
149.30
141.16
140.99
139.19
129.20
118.33
113.91
111.63
83.29
77.42
77.20
77.00
76.57
75.19
62.88
55.18
49.53
27.96
13.91

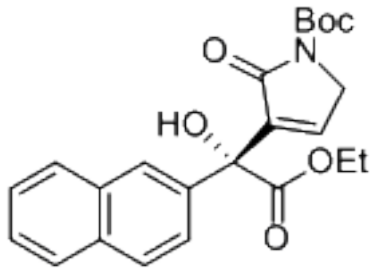


4p

```
NAME            zhjl12.11
EXPNO                  43
PROCNO                  1
Date_            20121120
Time                20.40
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   113
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                289.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677564 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

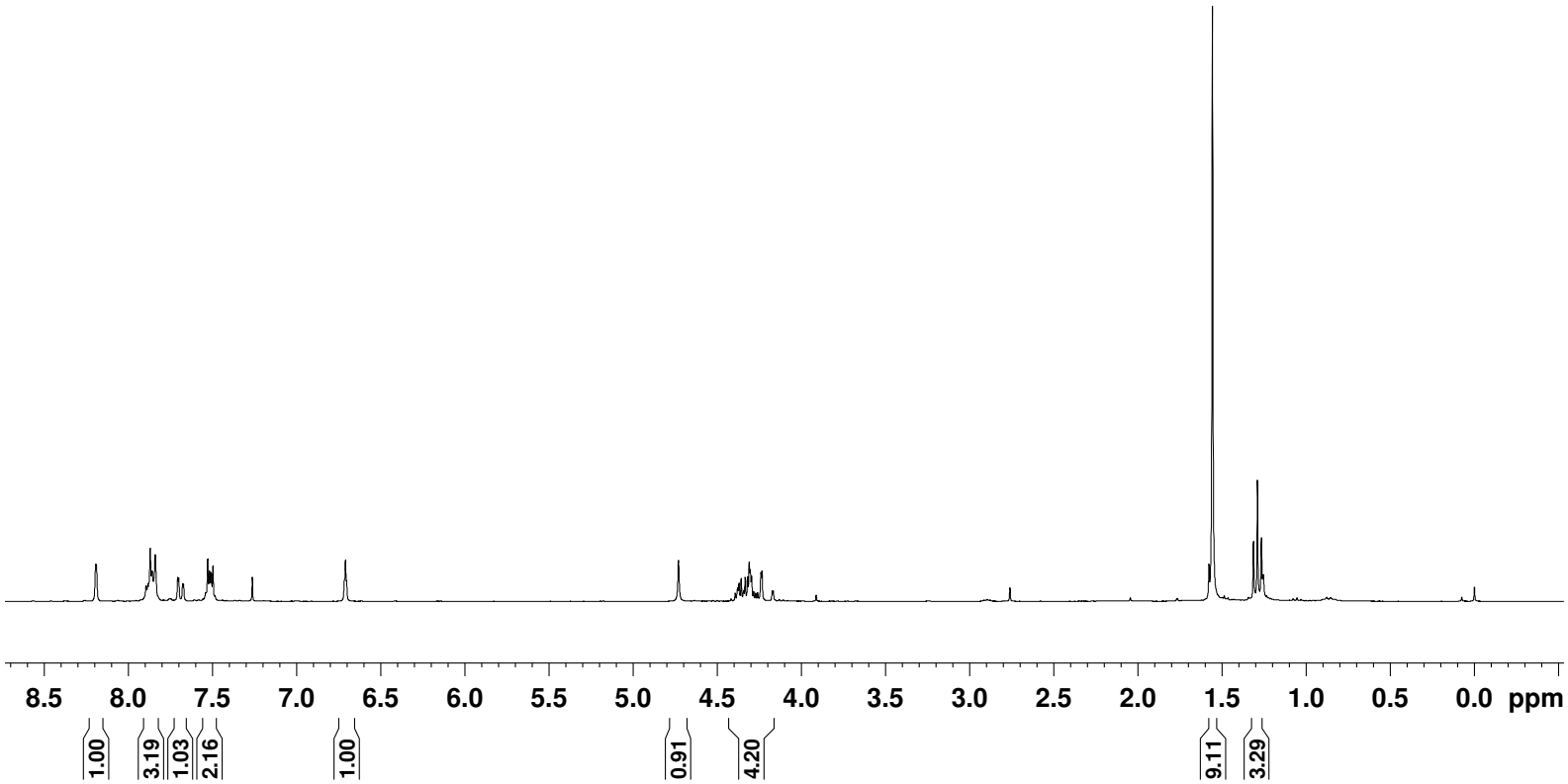


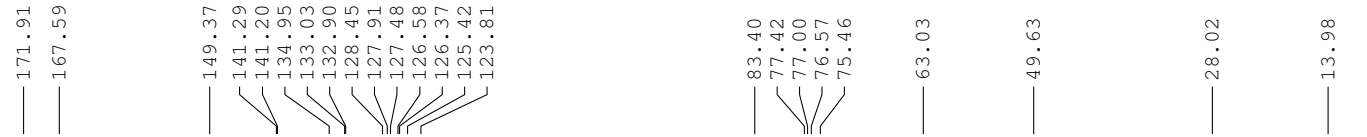


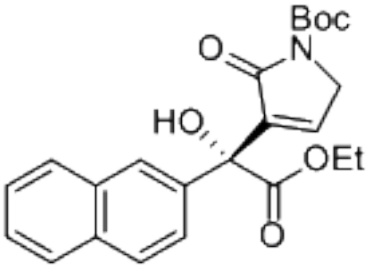


4q

```
NAME            znjl12.11
EXPNO                  32
PROCNO                  1
Date_            20121119
Time                11.55
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6188.119 Hz
FIDRES           0.094423 Hz
AQ               5.2953587 sec
RG                     57
DW                 80.800 usec
DE                  6.50 usec
TE                 288.4 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 11.80 usec
PL1                 0.00 dB
PL1W         11.55467796 W
SFO1         300.1318534 MHz
SI                  32768
SF           300.1300015 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```

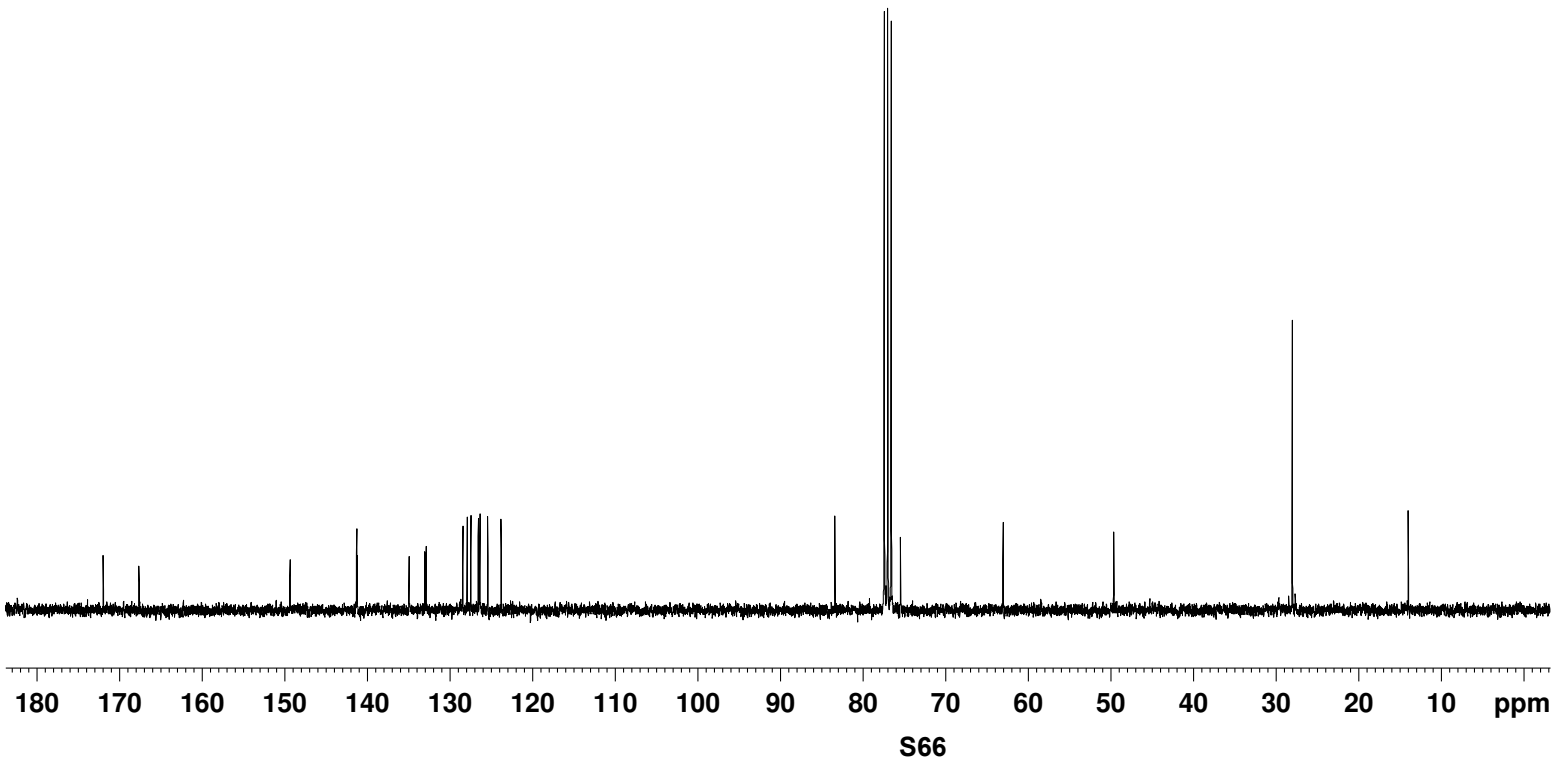


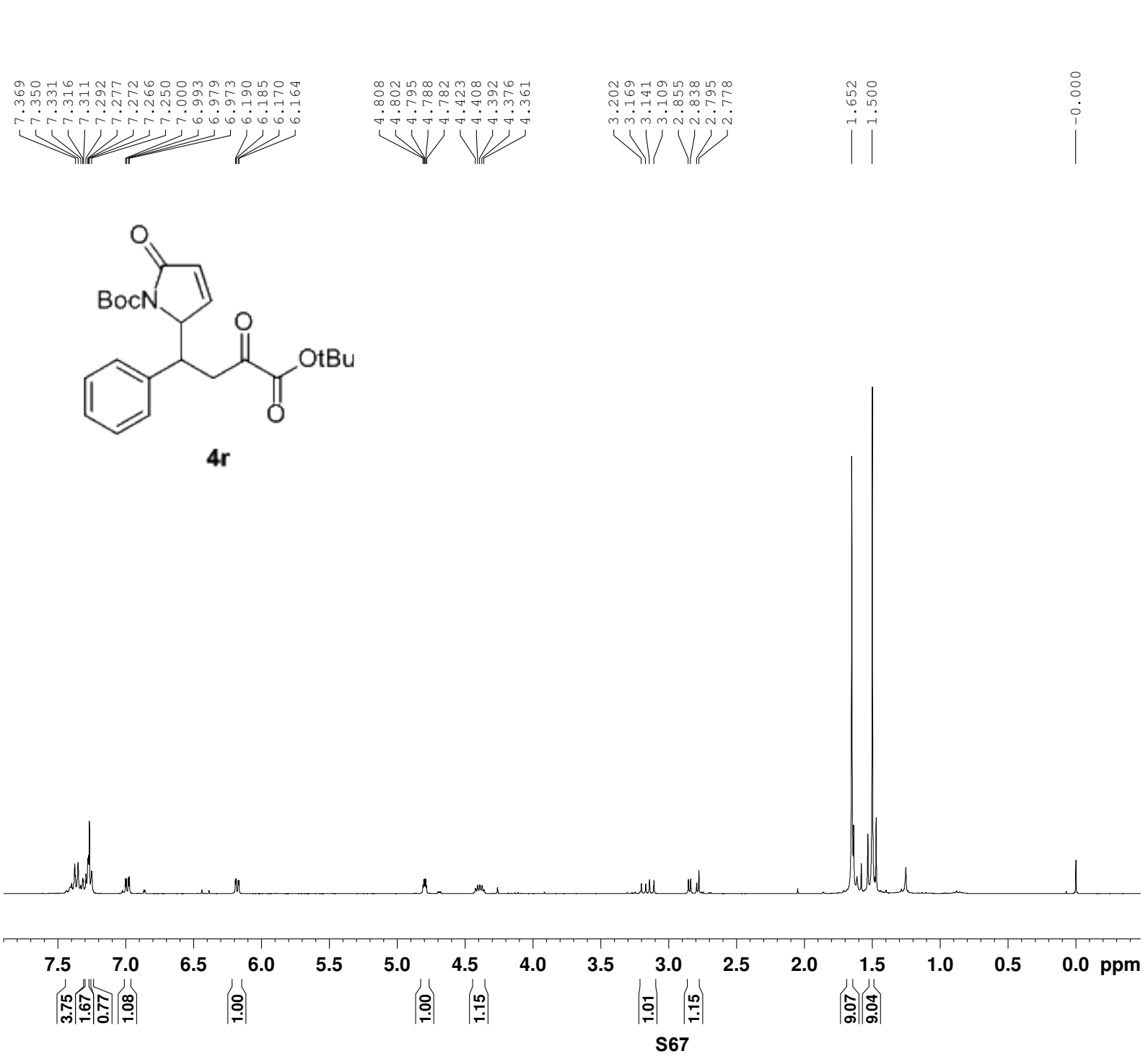




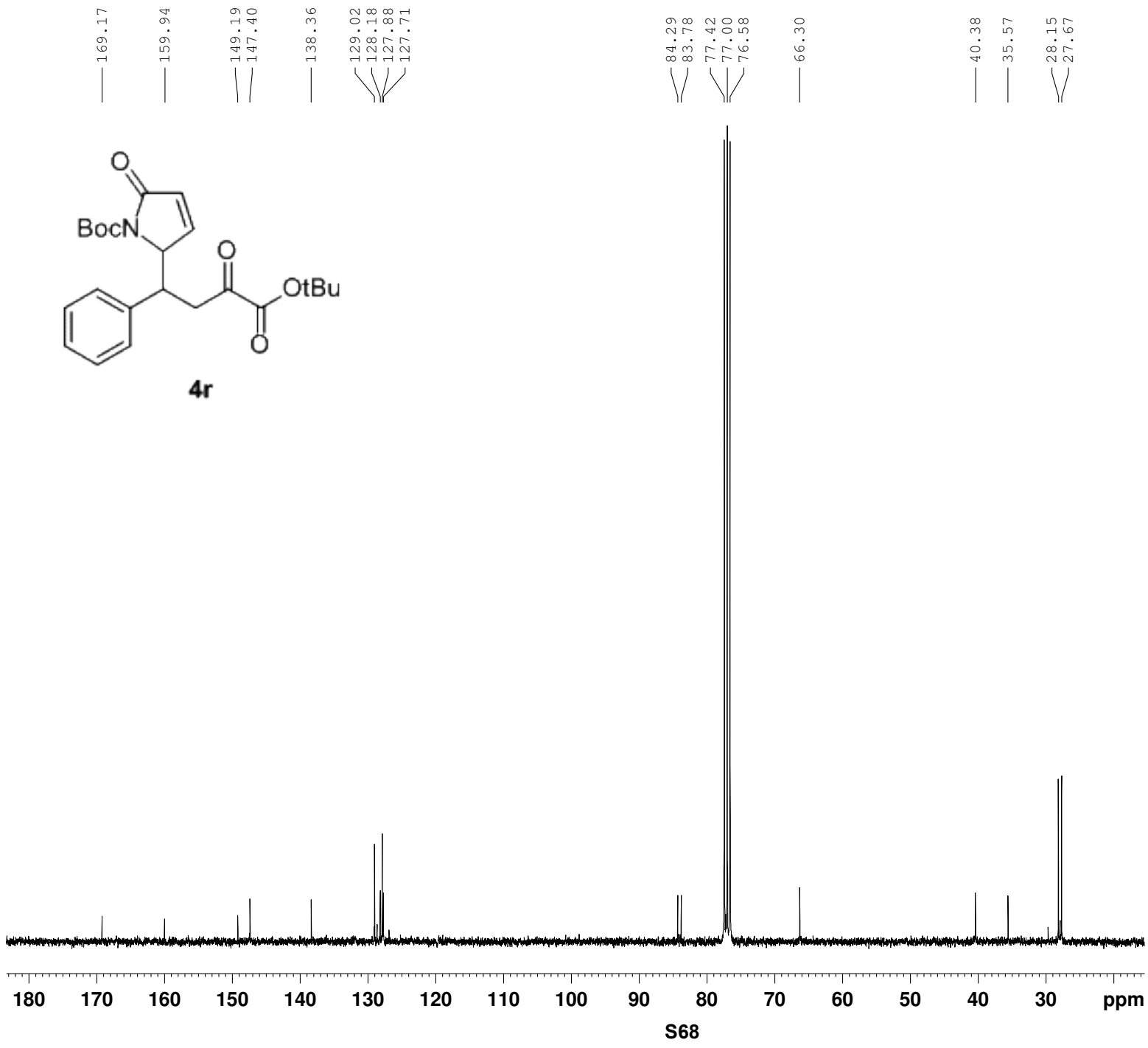
4q

```
NAME            zhjl12.11
EXPNO                  33
PROCNO                  1
Date_            20121119
Time                12.01
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    210
DS                      4
SWH             18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                    203
DW                27.733 usec
DE                 6.50 usec
TE                289.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                 0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677531 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```






```
NAME                 znjl12.11
EXPNO                      100
PROCNO                       1
Date_                 20121127
Time                     15.25
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          2
SWH                  6188.119 Hz
FIDRES               0.094423 Hz
AQ                   5.2953587 sec
RG                        181
DW                     80.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     11.80 usec
PL1                     0.00 dB
PL1W              11.55467796 W
SFO1              300.1318534 MHz
SI                      32768
SF                300.1300008 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```




```
NAME            zhjl12.11
EXPNO                 101
PROCNO                  1
Date_            20121127
Time                15.45
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1054
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                   203
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1                0.00 dB
PL1W         29.38907051 W
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.00 dB
PL13               17.00 dB
PL2W          9.17820644 W
PL12W         0.23054613 W
PL13W         0.23054613 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677509 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```