Asymmetric 1,3-Dipolar Cycloaddition of Azomethine ylides with Alkylidene Malonates Catalyzed by AgOAc/TF-BiphamPhos

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Supporting Information

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General Remarks.

^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d3. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quarte, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 100 MHz spectrometer in chloroform-d3. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Diastereomeric ratios were determined from crude ^{1}H NMR or HPLC analysis. Enantiomeric ratios were determined by HPLC, using a chiralcel AD-H column, a chiralpak AS-H column with hexane and *i*-PrOH as solvents. Ligands **1a-e** were prepared according to the literature procedure reported by us.[1] Alkylidene Malonates were prepared according to the literature procedure.[2] The racemic adducts were attained by using AgOAc/PPh_3 as the catalyst. The relative *exo*-configuration of **4dh** and the absolute (2*S*,3*S*,5*S*)-configuration of **4fd** achieved by AgOAc/(*R*)-TF-BiphamPhos was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

General Procedure for racemic 1,3-Dipolar Cycloaddition of Azomethine Ylides with Alkylidene Malonates Catalyzed by AgOAc/PPh_3 Complex

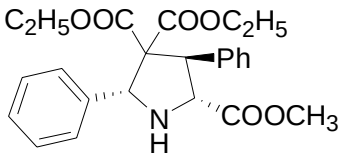
Under argon atmosphere, PPh_3（14.4 mg, 0.055 mmol) and AgOAc (8.6 mg, 0.05 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.5 mmol), Et_3N (0.5 mmol) and alkylidene malonate (0. 3 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the organic solvent was removed and the residue was purified by column chromatography to give the cycloaddition product (65-85% yield), which was used as the racemic sample for the chiral HPLC analysis.

General Procedure (A) for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with Alkylidene Malonates Catalyzed by Ag(I)/(*S*)-TF-BiphamPhos Complex in the Presence of Et_3N as Base

Under argon atmosphere (*S*)-TF-BiphamPhos **1e**（6.0 mg, 0.0075 mmol) and AgOAc (1.2 mg, 0.007 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.45 mmol), Et_3N (0.035 mmol) and alkylidene malonate (0.23 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was analyzed by ^1H NMR to determine the *exo*/*endo* ratio, and then the residue was purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.

General Procedure (B) for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with Alkylidene Malonates Catalyzed by Ag(I)/(*R*)-TF-BiphamPhos Complex in the Presence of K_2CO_3 as Base

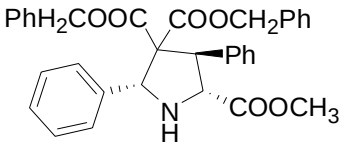
Under argon atmosphere (*R*)-TF-BiphamPhos **1e**（6.0 mg, 0.0075 mmol) and AgOAc (1.2 mg, 0.007 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.45 mmol), K_2CO_3 (62 mg, 0.46 mmol) and alkylidene malonate (0.23 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was analyzed by ^1H NMR to determine the *exo*/*endo* ratio, and then the residue was purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.



(4aa)

(2*R*,3*R*,5*R*)-4,4-diethyl 2-methyl 3,5-diphenylpyrrolidine-2,4,4-tricarboxylate

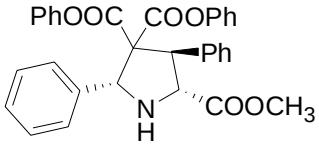
The title compound was prepared according to the general procedure A as described above in 88% yield. It was purified by flash chromatography to afford white solid. m.p. 80-82 ^{o}C; $[\alpha]^{25}_{D}$ = +42.9 (*c* 0.7, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.51-7.48 (m, 2H), 7.33-7.30 (m, 8H), 5.34 (s 1H), 4.42 (d, *J* = 6.6 Hz, 1H), 4.21 (d, *J* = 6.6 Hz, 1H), 3.83-3.78 (m, 5H), 3.47-3.35 (m, 2H), 0.80 (t, *J* = 7.2 Hz, 3H), 0.71 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 12.94, 13.05, 52.12, 55.91, 60.69, 60.98, 66.01, 67.81, 70.64, 128.02, 128.40, 128.84, 138.00, 138.55, 168.45; 169.26, 172.87; IR (KBr) ν 3423, 3055, 2985, 1720, 1265, 739 cm^{-1}. HRMS Calcd. For $C_{24}H_{27}NO_6$: 425.1838, found 425.1835. The product was analyzed by HPLC to determine the enantiomeric excess: 67% ee (Chiralcel AS-H, *i*-propanol/hexane = 30/70, flow rate 1.5 mL/min, λ = 220 nm); t_r = 2.61 and 3.29 min.



(4ba)

(2*R*,3*R*,5*R*)-4,4-dibenzyl 2-methyl 3,5-diphenylpyrrolidine-2,4,4-tricarboxylate

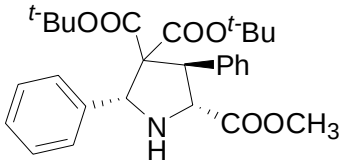
The title compound was prepared according to the general procedure A as described above in 90% yield. It was purified by flash chromatography to afford white solid. m.p. 96-98 ^{o}C; $[\alpha]^{25}_{D}$ = +20.2 (*c* 0.6, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.53-7.51 (m, 2H), 7.33-7.13 (m, 15H), 6.78-6.71 (m, 3H), 5.40 (s 1H), 4.84-4.70 (m, 2H), 4.45 (d, *J* = 6.6 Hz, 1H), 4.25 (d, *J* = 6.6 Hz, 1H), 4.15-4.04 (m, 2H), 3.77 (s, 3H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 50.44, 54.33, 64.31, 65.17, 65.29, 66.32, 69.17, 125.55, 125.67, 125.98, 126.06, 126.14, 126.20, 126.31; 126.44, 126.91, 132.49, 136.09, 136.55, 166.49, 167.32, 171.08; IR (KBr) ν 3425, 1719, 1458, 1261 cm^{-1}. HRMS Calcd. For $C_{34}H_{31}NO_6$: 549.2151, found 549.2157. The product was analyzed by HPLC to determine the enantiomeric excess: 65% ee (Chiralcel AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.62 and 7.38 min.



(4ca)

(2*R*,3*R*,5*R*)-2-methyl 4,4-diphenyl 3,5-diphenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure A as described above in 90% yield. It was purified by flash chromatography to afford white solid. m.p. 146-148 ^{o}C; $[\alpha]^{25}_{D}$ = +97.8 (*c* 0.7, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.66-7.41 (m, 10H), 7.20-7.15 (m, 7H), 6.36-6.26 (m, 3H), 5.58 (s 1H), 4.65 (d, *J* = 6.6 Hz, 1H), 4.33 (d, *J* = 6.0 Hz, 1H), 3.77 (s, 3H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 52.61, 56.99, 66.83, 68.72, 70.97, 120.93, 120.97, 126.11, 128.02, 128.48, 128.73, 128.94, 129.24, 129.28; 129.39, 137.76, 138.86, 149.94, 149.97, 167.49, 168.16, 172.71; IR (KBr) ν 3415, 3054, 1741, 1265, 739 cm^{-1}. HRMS Calcd. For $C_{32}H_{27}NO_6$: 521.1838, found 521.1837. The product was analyzed by HPLC to determine the enantiomeric excess: 78% ee (Chiralcel AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.89 and 10.49 min.



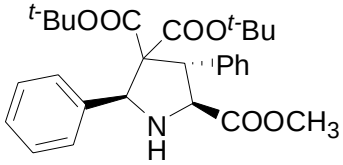
(4da)

(2*R*,3*R*,5*R*)-4,4-di-tert-butyl2-methyl

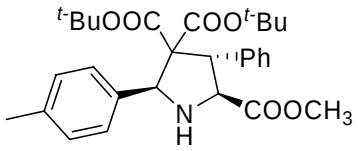
3,5-diphenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 83% yield. It was purified by flash chromatography to afford white solid. m.p. 120-122 ^{o}C; $[\alpha]^{25}_{D}$ = +42.0 (*c* 1.1, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.58-7.51 (m, 2H), 7.36-7.28 (m, 8H), 5.31 (s 1H), 4.36 (d, *J* = 6.6 Hz, 1H), 4.15 (d, *J* = 3.6 Hz, 1H), 3.77 (s, 3H), 1.00 (s, 18H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 27.23, 27.24, 52.36, 56.43, 66.67, 67.81, 71.21, 81.48, 81.83, 127.31, 127.61, 128.03, 128.07, 128.37, 129.50, 138.52, 140.00, 167.93, 168.57, 173.20; IR (KBr) ν 3415,

3054, 2983, 1739， 1714，1265， 739 cm^{-1}. HRMS Calcd. For $C_{28}H_{35}NO_6$: 481.2464, found 481.2462. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.30 and 5.67 min.



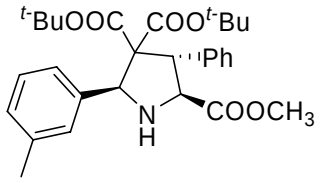
4da: This product was obtained in 83 yield and 81% ee from the reaction catalyzed by AgOAc/(*R*)-**1e** (3 mol %) at 25 °C; [α]^{25}D = -41.0 (*c* 0.7, $CHCl_3$)



(4db)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl2-methyl 3-phenyl-5-p-tolylpyrrolidine-2,4,4-Tricarboxylate

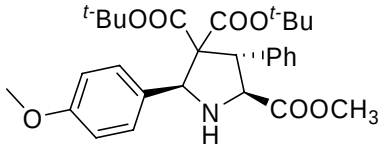
The title compound was prepared according to the general procedure B as described above in 88% yield. It was purified by flash chromatography to afford white solid. m.p. 110-112 °C; [α]$^{25}_D$ = -54.4 (*c* 1.4, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.46-7.44 (m, 2H), 7.35-7.27 (m, 5H), 7.14-7.11 (m, 2H), 5.27 (s 1H), 4.34 (d, *J* = 6.6 Hz, 1H), 4.14 (d, *J* = 6.3 Hz, 1H), 3.75 (s, 3H), 2.33 (s, 3H), 1.00 (s, 9H), 0.99 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 21.06, 27.23, 27.26, 52.35, 56.51, 66.66, 67.68, 71.31, 81.41, 81.78, 127.27, 127.91, 128.35, 128.63, 129.51, 135.38, 137.16, 140.03, 167.99, 168.66, 173.22; IR (KBr) ν 3420, 1719, 1264, 748 cm^{-1}. HRMS Calcd. For $C_{29}H_{37}NO_6$: 495.2621, found 495.2619. The product was analyzed by HPLC to determine the enantiomeric excess: 84% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.12 and 5.24 min.



(4dc)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3-phenyl-5-m-tolylpyrrolidine-2,4,4-Tricarboxylate

The title compound was prepared according to the general procedure B as described above in 81% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D}$ = -53.7 (*c* 1.6, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.38-7.18 (m, 8H), 7.10-7.07 (m, 1H), 5.27 (s 1H), 4.35 (d, *J* = 7.2 Hz, 1H), 4.15 (d, *J* = 6.3 Hz, 1H), 3.75 (s, 3H), 2.34 (s, 3H), 1.00 (s, 18H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 20.45, 26.19, 51.31, 55.46, 65.59, 66.82, 70.24, 80.40, 80.68, 124.33, 126.26, 126.94, 127.28, 127.31, 127.40, 128.50, 136.29, 137.30, 138.92, 166.96, 167.59, 172.11; IR (KBr) ν3424, 1739, 1713, 1265， 739 cm^{-1}. HRMS Calcd. For $C_{29}H_{37}NO_6$: 495.2621, found 495.2619. The product was analyzed by HPLC to determine the enantiomeric excess: 83% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.57 and 6.74 min.

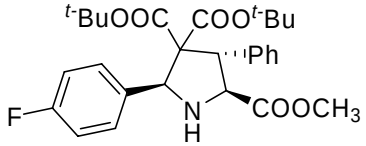


(4dd)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-(4-methoxyphenyl)-3-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 81% yield. It was purified by flash chromatography to afford white solid. m.p. 120-122 ^{o}C; $[\alpha]^{25}_{D}$ = -51.3 (*c* 1.7, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.52-7.49 (m, 2H), 7.38-7.27 (m, 5H), 6.88-6.85 (m, 2H), 5.26 (s 1H), 4.33 (d, *J* = 6.3 Hz, 1H), 4.33 (d, *J* = 6.6 Hz, 1H), 3.81 (s, 3H), 3.75 (s, 3H), 1.04 (s, 9H), 0.99 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 27.22, 27.34, 52.36, 55.34, 56.44, 66.66, 67.39,

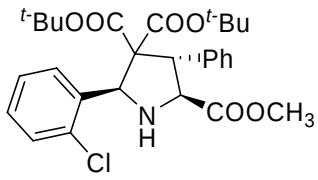
71.24, 81.41, 81.79,113.37, 127.28, 128.36, 129.14, 129.48, 130.62, 140.09, 159.12, 168.02, 168.71, 173.23; IR (KBr) ν 3425, 2979， 1740， 1716, 1250, 738 cm^{-1}. HRMS Calcd. For $C_{29}H_{37}NO_7$: 511.2570, found 511.2568. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm); t_r = 5.14 and 6.84 min.



(4de)

(2S,3S,5S)-4,4-di-tert-butyl 2-methyl 5-(4-fluorophenyl)-3-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 88% yield. It was purified by flash chromatography to afford white solid. m.p. 93-95 °C; $[\alpha]^{25}_D$ = -49.4 (*c* 1.1, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.59 (m, 2H), 7.35-7.32 (m, 5H), 7.05-7.00 (m, 2H), 5.28 (m 1H), 4.35 (d, *J* = 5.4 Hz, 1H), 4.13 (m, 1H), 3.76 (s, 3H), 1.04 (s, 9H), 0.99 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 26.19, 26.31, 51.38, 55.17, 65.64, 66.03, 70.02, 80.60, 81.00, 113.62, 113.83, 126.35, 127.40, 128.43, 128.70, 128.78, 133.37, 133.40, 139.04, 160.12, 162.56, 166.84, 167.46, 172.16; IR (KBr) ν 3423, 1712, 1638, 1265， 738 cm^{-1}. HRMS Calcd. For $C_{28}H_{34}FNO_6$: 499.2370, found 499.2375. The product was analyzed by HPLC to determine the enantiomeric excess: 85% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 5.28 and 7.15 min.

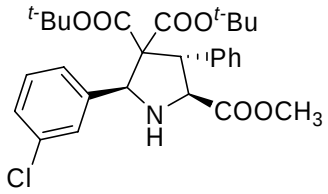


(4df)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-(2-chlorophenyl)-3-phenylpyrrolidine-

2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 69% yield. It was purified by flash chromatography to afford white solid. m.p. 96-98 ^{o}C; $[\alpha]^{25}_{D}$ = -11.6 (*c* 1.3, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.76-7.74 (m, 1H), 7.42-7.20 (m, 8H), 5.82 (s, 1H), 4.55 (d, 1H), 4.46 (d, *J* = 11.7 Hz, 1H), 3.64(s, 3H), 1.07 (s, 9H), 0.99 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 25.84, 26.08, 50.96, 52.78, 60.64, 61.83, 69.12, 80.39, 80.81, 125.75, 125.97, 126.72, 127.51, 127.80, 128.17, 128.88, 133.83, 134.29, 137.97, 166.48, 167.42, 171.25; IR (KBr) ν 3425, 1736, 1712, 1265, 739 cm^{-1}. HRMS Calcd. For $C_{28}H_{34}ClNO_6$: 515.2075, found 515.2079. The product was analyzed by HPLC to determine the enantiomeric excess: 78% ee (Chiralcel AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 11.90 and 15.05 min.

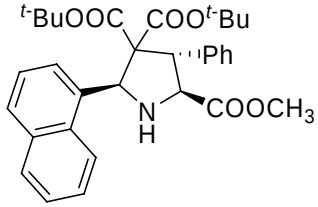


(4dg)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-(3-chlorophenyl)-3-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 98% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D}$ = -49.5 (*c* 1.7, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.60-7.53 (m, 3H), 7.35-7.32 (m, 6H), 5.28 (s, 1H), 4.38 (d, *J* = 6.3 Hz, 1H), 4.14 (d, *J* = 5.7 Hz, 1H), 3.76(s, 3H), 1.05 (s, 9H), 1.00 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 27.20, 27.30, 52.40, 55.94, 66.56, 67.09, 70.99, 81.73, 82.14, 126.42, 127.40, 127.72, 128.23, 128.42, 129.30, 129.48, 133.88, 139.90, 140.87, 167.71, 168.23, 173.00; IR (KBr) ν 3339, 1719, 1597, 1078, 738 cm^{-1}. HRMS Calcd. For $C_{28}H_{34}ClNO_6$: 515.2075, found 515.2064. The product was analyzed by HPLC to determine the enantiomeric excess: 80% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0

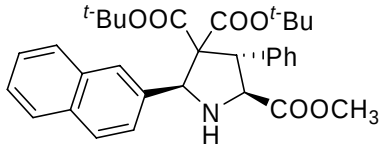
mL/min, λ = 210 nm); t_r = 5.11 and 9.34 min.



(4dh)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-(naphthalen-1-yl)-3-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 80% yield. It was purified by flash chromatography to afford white solid. m.p. 134-136 °C; $[\alpha]^{25}_D$ = +20.0 (*c* 1.2, $CHCl_3$); After simple recrystallization from petroleum ether, the enantionmic excess is improved to 99%, $[\alpha]^{25}_D$ = +26.1 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.23-8.20 (m, 1H), 7.90-7.77 (m, 3H), 7.52-7.41(m, 5H), 7.29-7.26 (m, 3H), 6.23 (s, 1H), 4.66 (d, *J* = 11.1 Hz, 1H), 4.47 (d, *J* = 11.1 Hz, 1H), 3.67 (s, 3H), 1.04 (s, 9H), 0.49 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 26.64, 27.26, 52.25, 55.12, 62.18, 62.41, 70.94, 81.12, 82.02, 124.64, 125.42, 125.49, 125.59, 125.89, 127.21, 128.07, 128.21, 128.41, 129.44, 132.91, 133.51, 136.21, 137.24, 167.98, 169.32, 172.43; IR (KBr) ν 3426, 1707, 1639, 1265, 739 cm^{-1}. HRMS Calcd. For $C_{32}H_{37}NO_6$: 531.2621, found 531.2620. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 5.70 and 9.42 min.

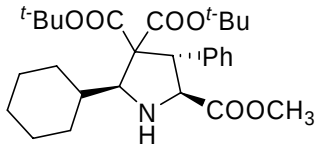


(4di)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-(naphthalen-2-yl)-3-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described

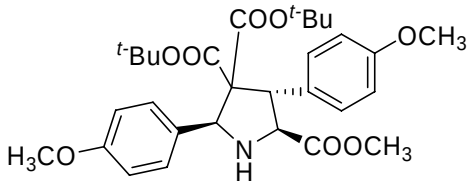
above in 74% yield. It was purified by flash chromatography to afford white solid. m.p. 126-128 ^{o}C; $[\alpha]^{25}_{D}$ = -73.5 (*c* 1.5, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.99 (s, 1H), 7.82-7.78(m, 4H), 7.47-7.29(m, 7H), 5.47(s, 1H), 4.43(d, *J* = 7.5 Hz, 1H), 4.21(d, *J* = 6.6 Hz, 1H), 3.79(s, 3H), 1.00(s, 9H), 0.81(s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 26.07, 26.19, 51.36, 55.37, 65.71, 66.81, 70.35, 80.50, 80.80, 124.77, 124.86, 125.10, 125.83, 126.30, 126.32, 126.37, 127.09, 127.37, 128.53, 131.95, 132.09, 135.06, 139.03, 166.93, 167.53, 172.11; IR (KBr) ν 3417, 1637 cm^{-1}. HRMS Calcd. For $C_{32}H_{37}NO_6$: 531.2621, found 531.2618. The product was analyzed by HPLC to determine the enantiomeric excess: 80% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 5.75 and 10.60 min.



(4dj)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-cyclohexyl-3-phenylpyrrolidine-2,4,4-tricarboxylate

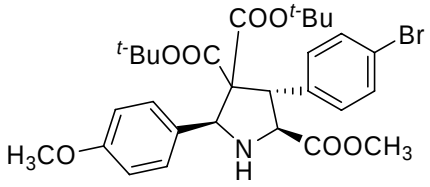
The title compound was prepared according to the general procedure B as described above in 18% yield with 3 mol% catalyst and in 80% yield with 20 mol% catalyst. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_{D}$ = -28.0 (*c* 0.8, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.24 (s, 5H), 4.09 (d, *J* = 5.4 Hz, 1H), 3.94 (d, *J* = 5.1 Hz, 1H), 3.72 (m, 4H), 2.02-1.98(m, 2H), 1.77-1.67(m, 3H), 1.50 (s, 9H), 1.46-1.04(m, 6H), 0.96 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 26.33, 26.48, 26.52, 27.18, 27.96, 31.16, 31.98, 40.19, 52.27, 58.53,68.01, 69.81, 70.73, 80.99, 82.50, 127.24, 128.30, 129.63, 141.06, 167.68, 170.06, 172.91; IR (KBr) ν 3425, 3054, 1740, 1710, 1265 cm^{-1}. HRMS Calcd. For $C_{28}H_{41}NO_6$: 487.2934, found 487.2939. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralcel AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.18 and 4.99 min.



(4ed)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3,5-bis(4-methoxyphenyl)pyrrolidine-2,4,4-Tricarboxylate

The title compound was prepared according to the general procedure B as described above in 94% yield. It was purified by flash chromatography to afford white solid. m.p. 142-144 ^{o}C; $[\alpha]^{25}_{D}$ = -51.3 (*c* 1.3, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.50-7.47 (m, 2H), 7.30-7.27 (m, 3H), 6.87-6.84 (m, 3H), 5.22 (s, 1H), 4.29 (d, *J* = 7.5 Hz , 1H), 4.09 (d, *J* = 6.6 Hz , 1H), 3.80 (s, 6H), 3.75 (s, 3H), 1.04 (s, 18H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 27.33, 52.34, 55.30, 55.32, 55.67, 66.62, 67.25, 71.25, 81.37, 81.69, 113.35, 113.69, 129.12, 130.49, 130.75, 132.02, 158.84, 159.09, 168.15, 168.72, 173.29; IR (KBr) ν 3430, 2979, 1716, 1612, 1035 cm^{-1}. HRMS Calcd. For $C_{30}H_{39}NO_8$: 541.2676, found 541.2673. The product was analyzed by HPLC to determine the enantiomeric excess: 78% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/70, flow rate 1.0 mL/min, λ = 210 nm); t_r = 6.52 and 8.73 min.

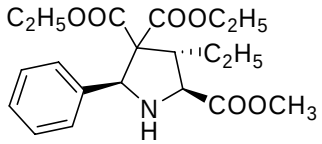


(4fd)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3-(4-bromophenyl)-5-(4-methoxyphenyl) pyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure B as described above in 84% yield. It was purified by flash chromatography to afford white solid. m.p. 116-118 ^{o}C; $[\alpha]^{25}_{D}$ = -32.4 (*c* 1.2, $CHCl_3$); After simple recrystallization from petroleum ether, the enantionmic excess is improved to 99%; $[\alpha]^{25}_{D}$ = -40.0 (*c* 1.3,

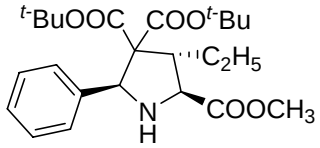
$CHCl_3$); ^1H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.50-7.44 (m, 4H), 7.27-7.24 (m, 2H), 6.88-6.85 (m, 2H), 5.20 (s, 1H), 4.30 (d, *J* = 6.6 Hz , 1H), 4.08 (d, *J* = 7.2 Hz , 1H), 3.80 (s, 3H), 3.74 (s, 3H), 1.04 (s, 9H), 1.03 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 27.29, 27.31, 52.40, 55.33, 55.49, 66.06, 67.33, 71.09, 81.76, 81.95, 113.42, 121.25, 129.17, 130.68, 131.22, 131.36, 138.74, 159.20, 167.97, 168.40, 172.87; IR (KBr) ν 3430, 1711, 1638, 1265, 739 cm^{-1}. HRMS Calcd. For $C_{29}H_{36}BrNO_7$: 589.1675, found 589.1678. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 17.49 and 25.86 min.



(4ga)

(2*S*,3*S*,5*S*)-4,4-diethyl 2-methyl 3-ethyl-5-phenylpyrrolidine-2,4,4-tricarboxylate

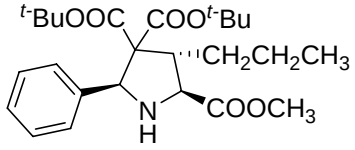
The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford colorless oil. $[\alpha]^{25}_D$ = -41.6 (*c* 0.8, $CHCl_3$); ^1H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.50-7.44 (m, 2H), 7.31-7.24 (m, 3H), 5.01 (s 1H), 4.31-4.14 (m, 2H), 3.84 (s, 3H), 3.81-3.73 (m, 2H), 3.47-3.41 (m, 1H), 3.14-3.09 (m, 1H), 1.62-1.27 (m, 2H), 1.25 (t, *J* = 7.2 Hz , 3H), 1.02 (t, *J* = 7.5 Hz , 3H), 0.82 (t, *J* = 6.0 Hz , 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 12.09, 13.27, 13.97, 23.42, 50.60, 52.34, 60.93, 61.20, 64.01, 67.20, 69.23, 127.73, 127.93, 139.33; 169.18, 169.60, 174.36; IR (KBr) ν 3425, 1719, 1261 cm^{-1}. HRMS Calcd. For $C_{20}H_{27}NO_6$: 377.1838, found 377.1843. The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 3.87 and 5.02 min.



(4ha)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3-ethyl-5-phenylpyrrolidine-2,4,4-Tricarboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. It was purified by flash chromatography to afford white solid. $[\alpha]^{25}_{D}$ = -26.9 (*c* 0.8, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.48-7.46 (m, 2H), 7.31-7.23 (m, 3H), 4.91 (s, 1H), 3.82 (s, 3H), 3.63 (d, *J* = 8.4 Hz, 1H), 3.10-3.09 (m, 1H), 1.73-1.63 (m, 2H), 1.51 (s, 9H), 1.08-1.02 (m , 12H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 12.59, 23.10, 27.15, 27.95, 50.63, 52.23, 64.26, 67.09, 69.83, 81.24, 82.25, 127.50, 127.97, 128.53; 140.88, 167.73, 169.33, 174.54; IR (KBr) ν 3425, 1719, 1261 cm^{-1}; MS (EI) *m*/*e* 434 (M^++1); The product was analyzed by HPLC to determine the enantiomeric excess: 83% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.3 and 5.5 min.

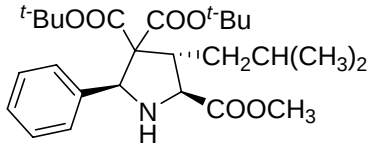


(4ia)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 5-phenyl-3-propylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. It was purified by flash chromatography to afford white solid. $[\alpha]^{25}_{D}$ = -19.5 (*c* 1.2, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.48-7.46 (m, 2H), 7.31-7.23 (m, 3H), 4.91 (s, 1H), 3.80 (s, 3H), 3.63 (d, *J* = 8.4 Hz, 1H), 3.19-3.17 (m, 1H), 1.62 (m, 2H), 1.50 (s, 9H), 1.44-1.40 (m , 2H), 1.01 (s, 9H), 0.92 (t , *J* = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 14.29, 21.02, 27.21, 27.99, 32.63, 48.78, 52.28, 64.57, 67.12, 69.86, 81.25, 82.23, 127.51, 128.00, 128.55, 133.84

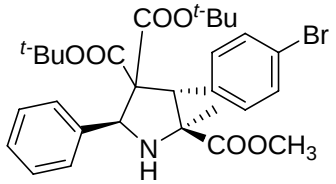
140.87, 167.77, 169.37, 174.51; IR (KBr) ν 3425, 1719, 1261 cm^{-1}; MS (EI) *m*/*e* 448 (M^++1); The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 4.1 and 5.3 min.



(4ja)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3-isobutyl-5-phenylpyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described above in 80% yield. It was purified by flash chromatography to afford white solid. $[\alpha]^{25}_D$ = -34.7 (*c* 1.3, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.52-7.49 (m, 2H), 7.32-7.23 (m, 3H), 4.93 (s, 1H), 3.81 (s, 3H), 3.63 (d, *J* = 6.6 Hz, 1H), 3.27-3.25 (m, 1H), 1.49 (s, 9H), 1.39 (t , *J* = 6.9 Hz, 2H), 1.06-0.88 (m, 16H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 21.41, 23.87, 25.59, 27.19, 27.42, 27.95, 39.88, 46.88, 52.25, 64.66, 66.91, 69.84, 81.21, 82.04, 127.45, 127.72, 127.97, 128.44, 140.55, 167.81, 169.20, 174.63; IR (KBr) ν 3425, 1719, 1261 cm^{-1}; MS (EI) *m*/*e* 462 (M^++1); The product was analyzed by HPLC to determine the enantiomeric excess: 82% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 210 nm); t_r = 3.9 and 5.6 min.



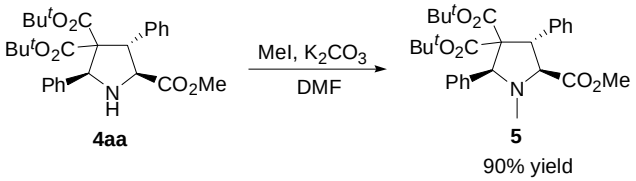
(4fk)

(2*S*,3*S*,5*S*)-4,4-di-tert-butyl 2-methyl 3-(4-bromophenyl)-2-methyl-5-phenyl-pyrrolidine-2,4,4-tricarboxylate

The title compound was prepared according to the general procedure as described

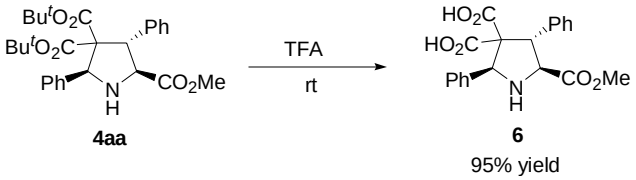
above in 72% yield. It was purified by flash chromatography to afford white solid. $[\alpha]^{25}_{D}$ = -37.8 (*c* 1.1, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.77-7.75 (m, 2H), 7.50-7.47 (m, 2H), 7.35-7.24 (m, 5H), 5.54 (s, 1H), 4.45 (s, 1H), 3.90 (s, 3H), 0.99 (s, 9H), 0.95(s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 26.66, 27.11, 52.89, 55.82, 67.04, 68.87, 71.46, 81.30, 81.88, 121.22, 127.42, 128.13; 128.23, 131.20, 132.26, 137.10, 139.54, 167.34, 168.56, 178.65; IR (KBr) ν 3425, 1719, 1261 cm^{-1}; MS (EI) *m*/*e* 574 (M^++1); The product was analyzed by HPLC to determine the enantiomeric excess: 76% ee (Chiralcel OD-H, *i*-propanol/hexane = 5/90, flow rate 1.0 mL/min, λ = 210 nm); t_r = 3.8 and 4.6 min.

Procedure for *N*-methylation of Cycloadduct 4aa



To a solution of **4aa** (100 mg, 0.21 mmol) and K_2CO_3 (29 mg, 0.21 mmol) in DMF (2 mL) was added MeI (60mg, 0.42 mmol) at room temperature. The mixture was stirred for 4 h and then CH_2Cl_2 (10 mL) was added. The mixture was washed with H_2O and the aqueous phase was extracted with CH_2Cl_2. The combined organic phase was dried over Na_2SO_4. The solvent was removed under reduced pressure, and the residue was purified by flash cromathography to afford **5** as white solid; yield:94 mg (90%).^{1}H NMR ($CDCl_3$, TMS, 300 MHz): δ 7.46 (m, 2H), 7.32-7.15 (m, 8H), 4.58-4.53 (m, 2H), 3.65 (dd, *J* = 9.3 Hz, 1H), 3.59 (s, 3H), 2.56 (s, 3H), 0.92 (s, 9H), 0.90 (s, 9H).^{13}C NMR ($CDCl_3$, TMS, 75 MHz): δ 172.3, 168.4, 167.6, 139.5, 137.8, 129.5, 128.1, 127.9, 127.5, 127.1, 81.6, 81.4, 77.4, 77.0, 69.8, 52.6, 52.0, 39.6, 27.1, 27.0.

Procedure for deprotecting of *tert*-butyl group from 4aa



4aa **6**

95% yield

To **4aa** (400 mg, 0.83 mmol) was added TFA (1ml) at room temperature. The mixture was stirred for 1 h. Then TFA was removed under reduced pressure affording **6** as white solid; yield: 284 mg (95%). ^{1}H NMR (D$_6$-DMSO, TMS, 300 MHz): δ 7.39-7.25 (m, 10H), 5.31 (s, 1H), 5.11 (dd, *J* = 9.9, 1H), 4.34 (dd, *J* = 10.5, 1H), 3.69(s, 3H). ^{13}C NMR (D$_6$-DMSO, TMS, 75 MHz): δ 172.1, 171.91, 168.9, 133.8, 132.1, 129.5, 129.1, 128.8, 128.5, 127.6, 68.9, 65.7, 61.6, 56.1, 54.0.

The relative configuration of the *exo*-adduct 4dh and the absolute configuration of *exo*-(2*S*,3*S*,5*S*)-4fd and *exo*-(2*S*,3*S*,5*S*)-4fk were determined by X-ray diffraction analysis

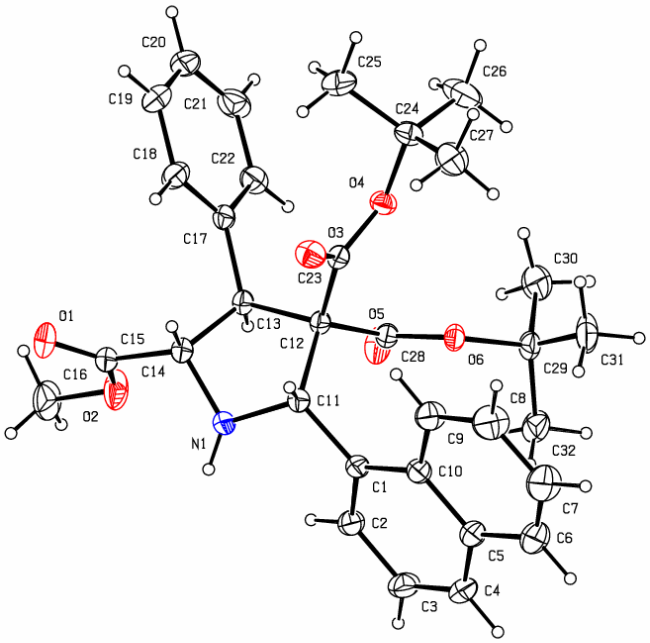


Figure 1. X-ray structure of racemic *exo*-4**dh** (relative configuration)

Crystal data for racemic *exo*-**4dh**: $C_{32}H_{37}NO_6$, M_r = 531.63, T = 293 K, Triclinic, space group P-1, a = 10.0784(12), b = 12.2383(14), c = 12.8767(15) Å, V = 1463.4(3) Å^3, Z = 2, 5649 unique reflections, final R_1 = 0.0462 and wR_2 = 0.1175 for 4300 observed [I>2σ(I)] reflections. CCDC 747638 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

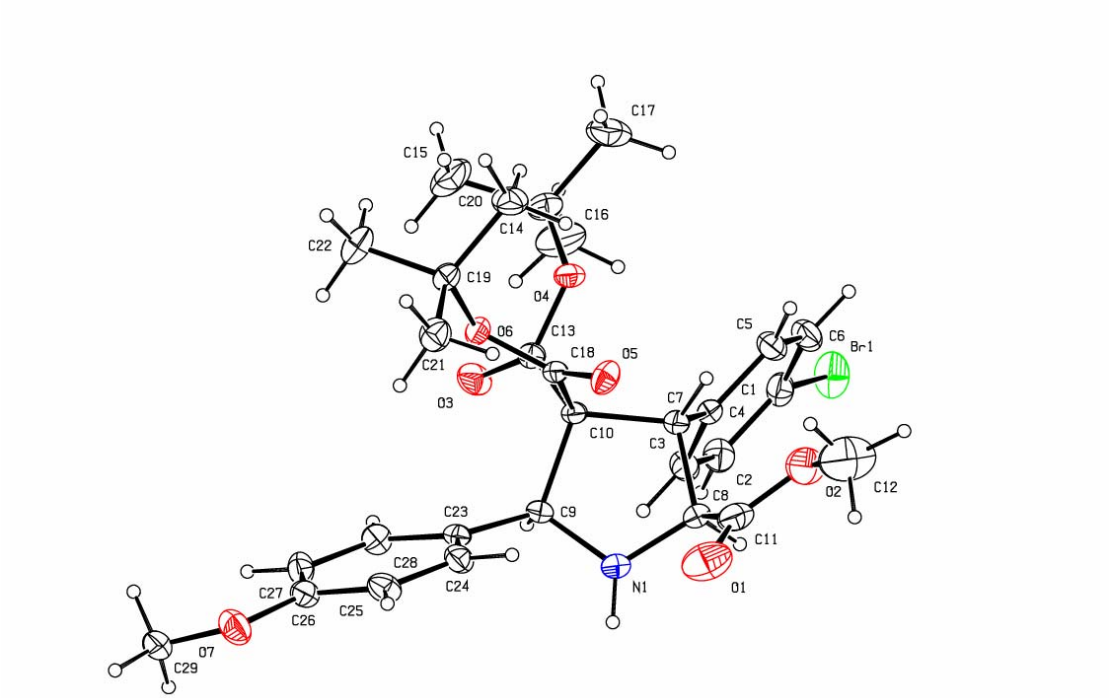


Figure 2. X-ray structure of (2*S*,3*S*,5*S*)-*exo*-4**fd** (absolute configuration)

Crystal data for (2*S*,3*S*,5*S*)-**4fd**: $C_{29}H_{36}BrNO_7$, M_r = 590.50, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 9.7742(8), b = 15.7902(13), c = 18.9829(16) Å, V = 2929.8(4) Å^3, Z = 4, 5717 unique reflections, final R_1 = 0.0437 and wR_2 = 0.1127 for 4283 observed [I>2σ(I)] reflections. CCDC 739450 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge

Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

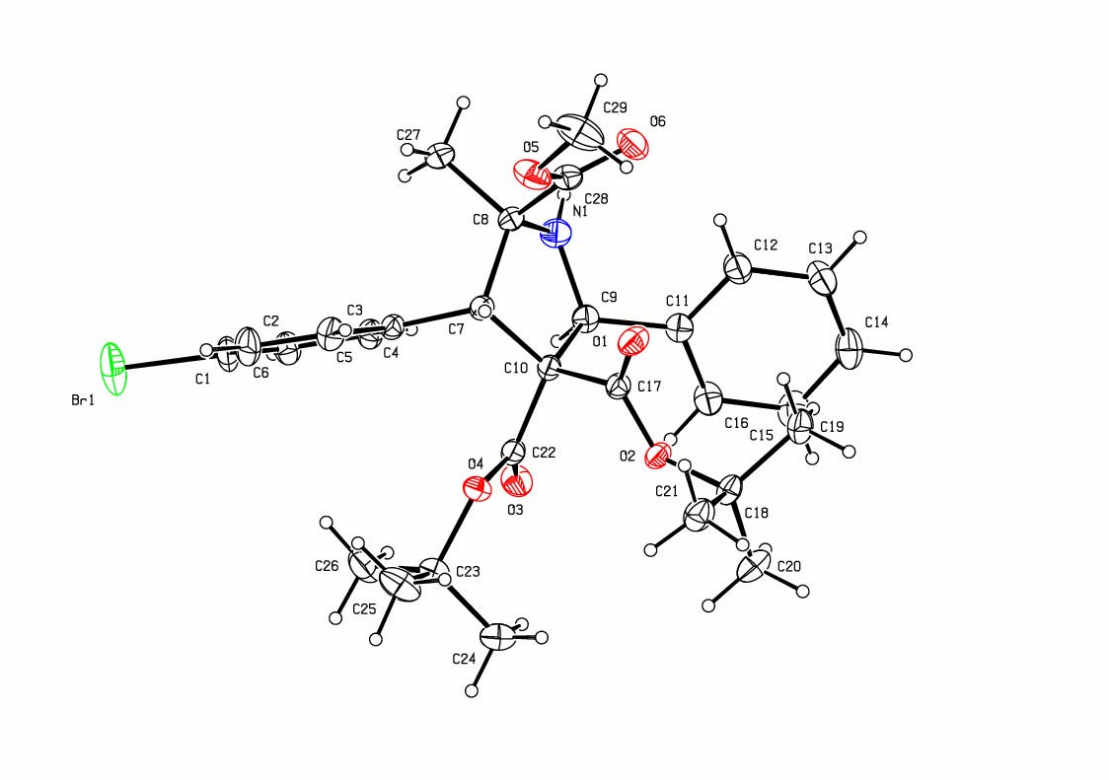


Figure 3. X-ray structure of (2*S*,3*S*,5*S*)-*exo*-4**fk** (absolute configuration)

Crystal data for (2*S*,3*S*,5*S*)-**4fk**: $C_{29}H_{36}BrNO_6$, M_r = 574.50, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 11.202(2), b = 11.863(2), c = 22.167(5) Å, V = 2945.5(10) Å^3, Z = 4, 17768 unique reflections, final R_1 = 0.0523 and wR_2 = 0.1338 for 6077 observed [I>2σ(I)] reflections. CCDC 761891 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk)

Proposed Transition States of the *endo*-selectivity for Dimethyl Maleate and the *exo*-selectivity for Alkylidene malonates

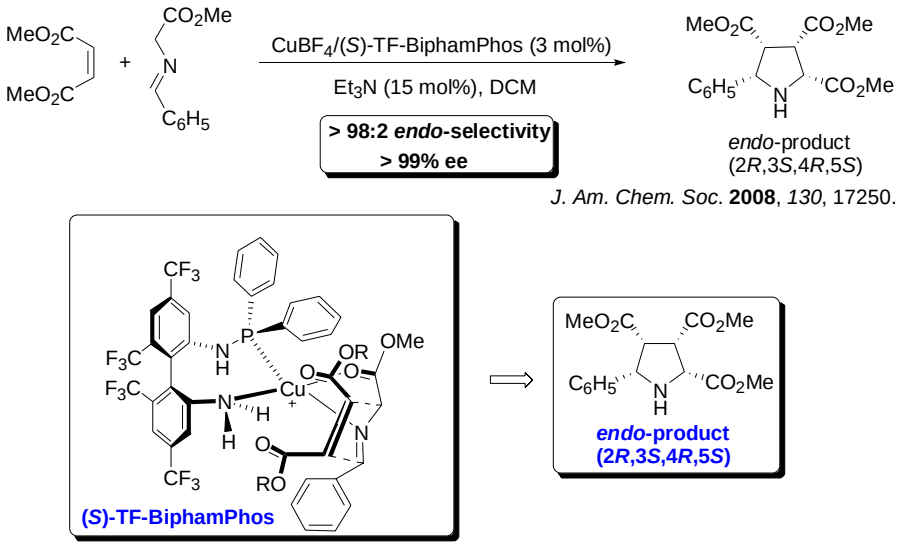


Figure 3. Proposed transition states leading to *endo*-adducts while using dimethyl maleate as the dipolar.

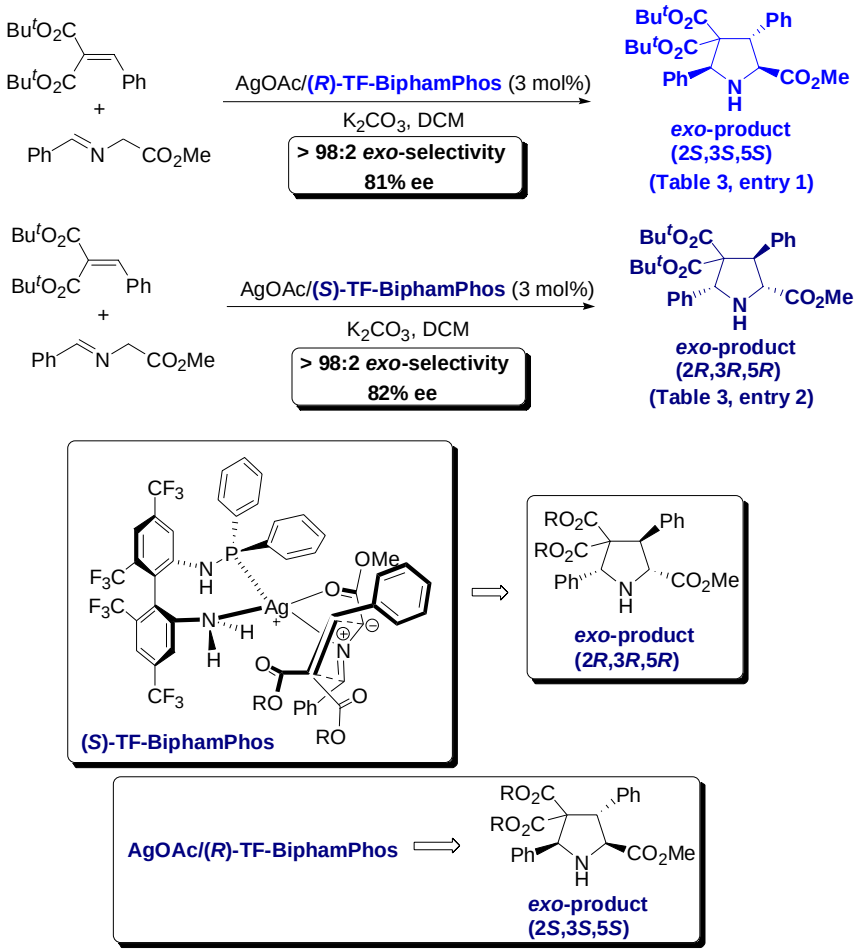


Figure 4. Proposed transition states leading to *exo*-adducts while using alkylidene malonate as the dipolar.

The carbonyl group of the alkylidene malonate or dimethyl maleate could coordinate with the Ag center, which can stabilize the negatively charged oxygen atom in the proposed transition states.[3] It could not rule out the possible hydrogen bond interaction between the carbonyl groups and the NH_2 group of the chiral ligand, which also facilitates stabilizing the proposed transition states.[4,5]

References

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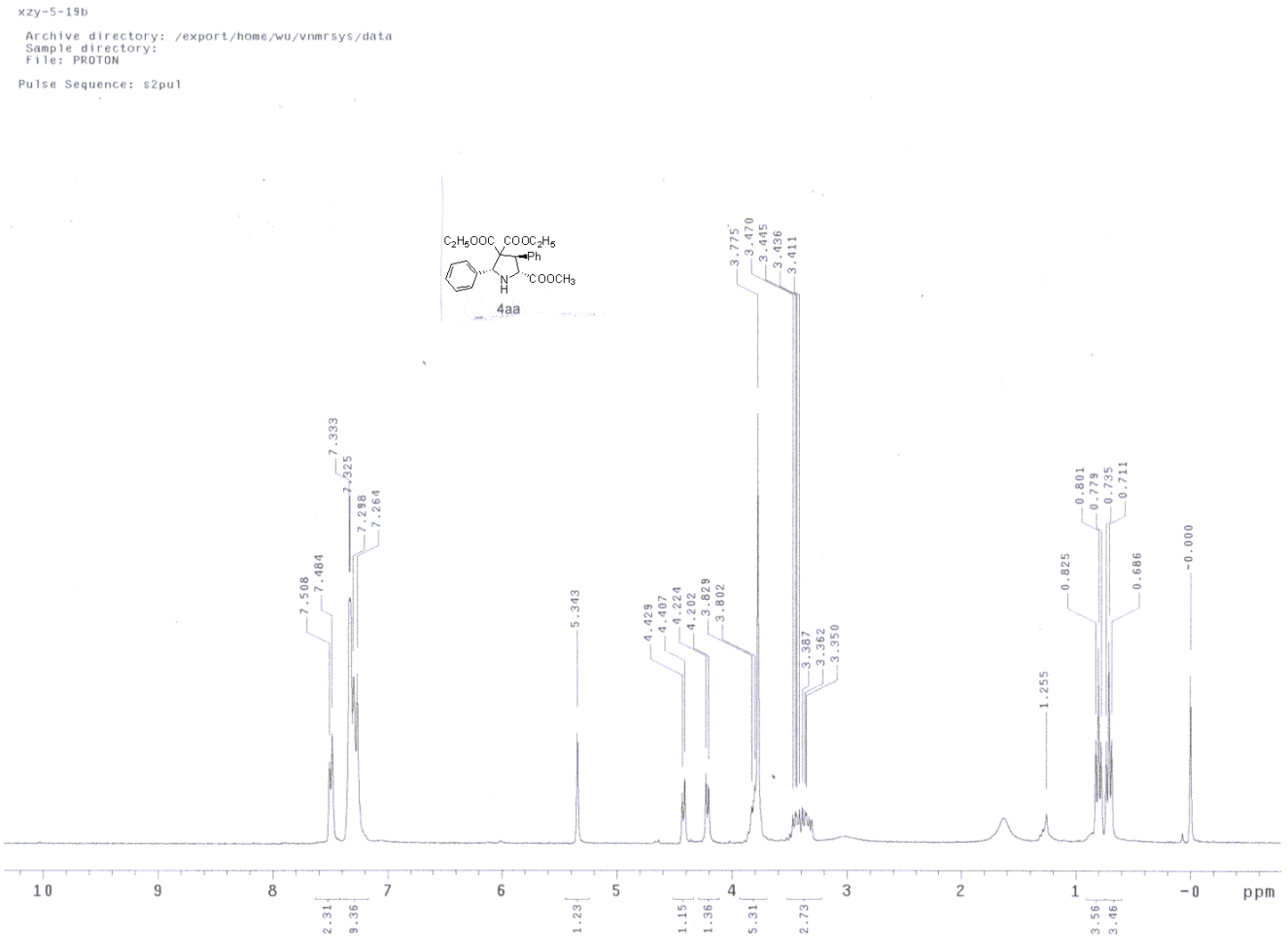

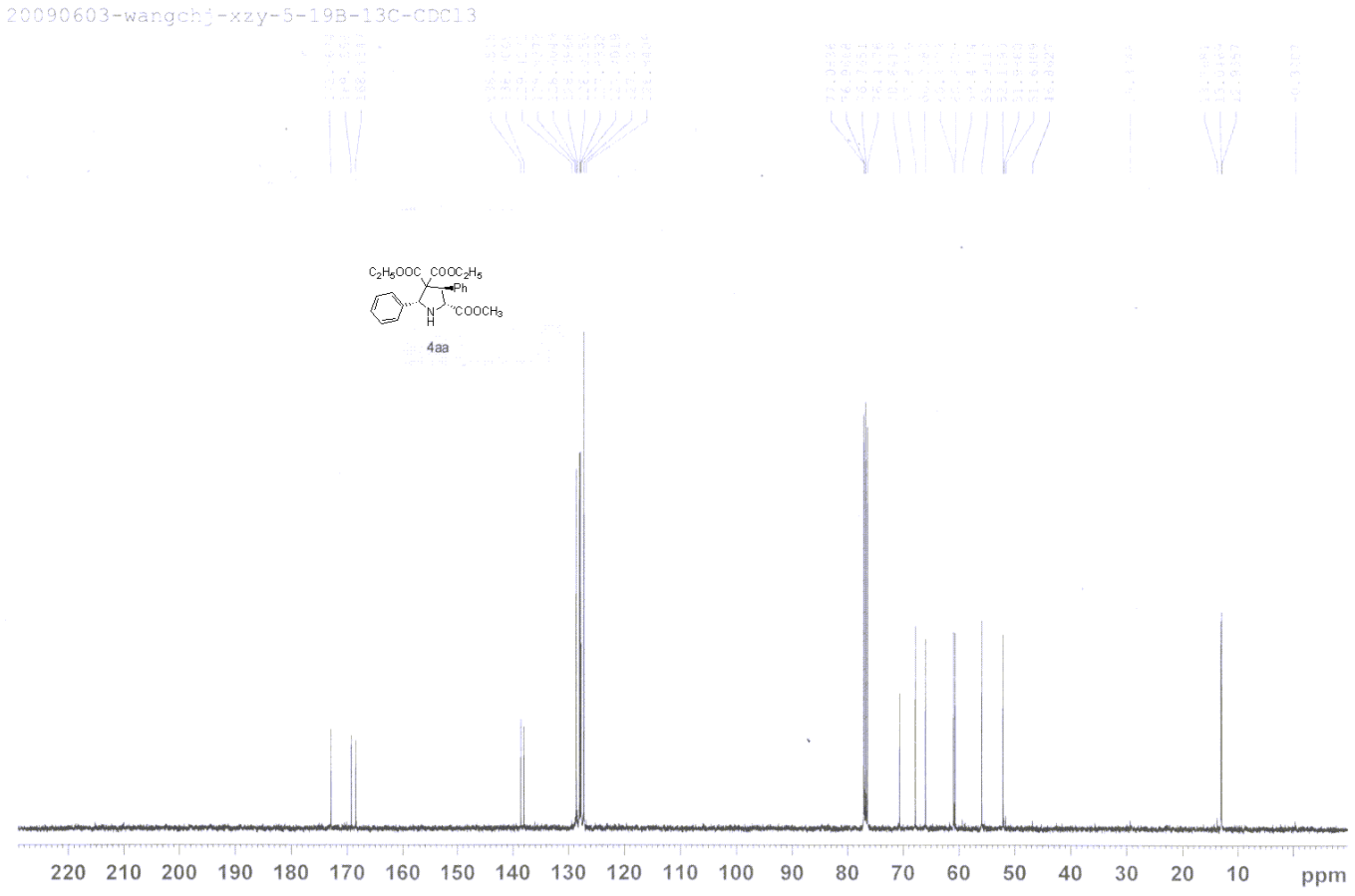

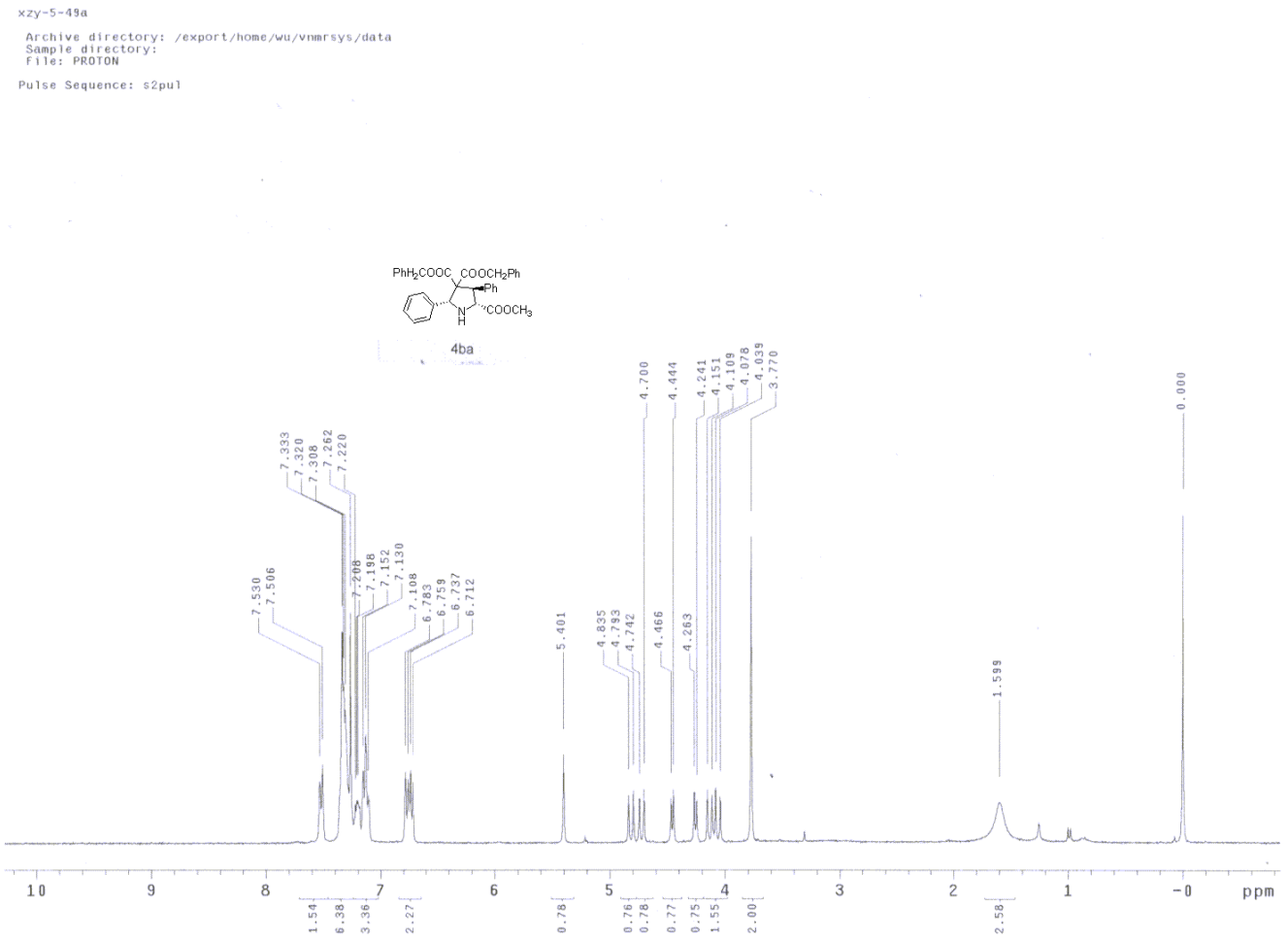
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Ph
COOCH3
N
H
4ba
7.530
7.506
7.333
7.320
7.308
7.262
7.220
7.208
7.198
7.152
7.130
7.108
6.783
6.759
6.737
6.712
5.401
4.835
4.793
4.742
4.700
4.466
4.444
4.263
4.241
4.151
4.109
4.078
4.039
3.770
1.599
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
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3.36
2.27
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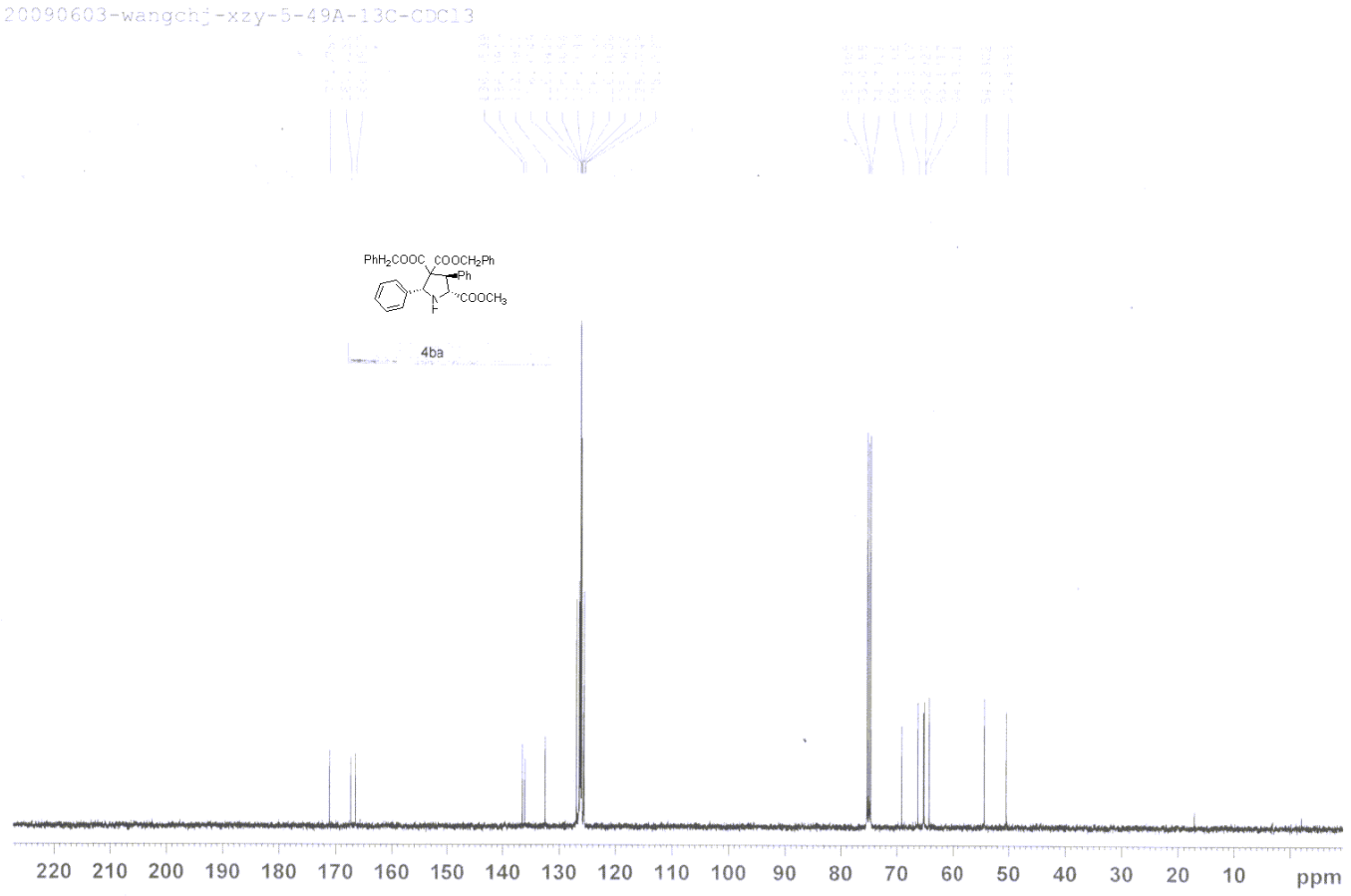
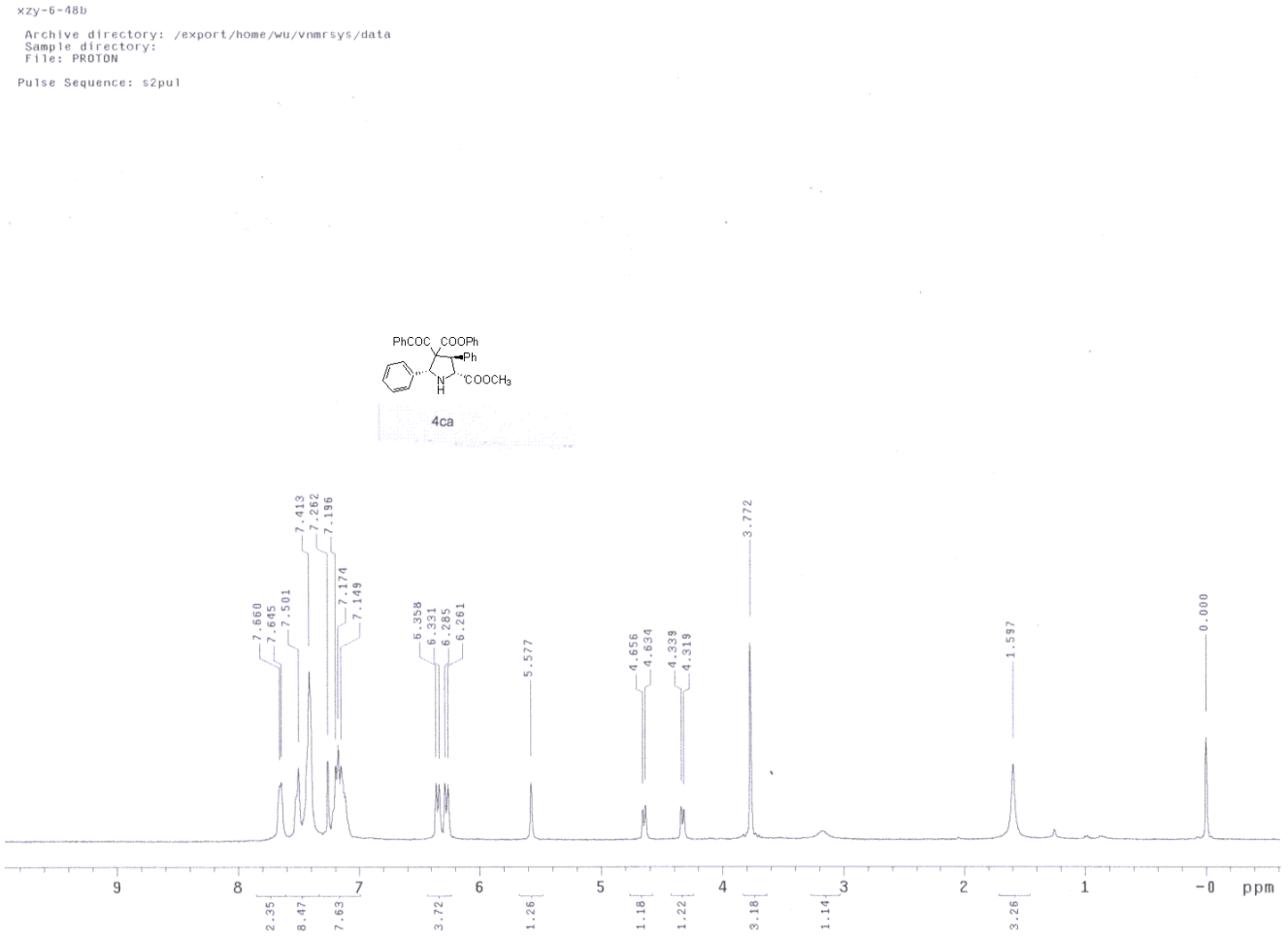
0.76
0.78
0.77
0.75
1.55
2.00
2.58




xzy-6-48b
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COOPh
Ph
COOCH3
N
H
4ca
7.660
7.645
7.581
7.413
7.262
7.196
7.174
7.149
6.358
6.331
6.285
6.261
5.577
4.656
4.634
4.339
4.319
3.772
1.597
0.000
9
8
7
6
5
4
3
2
1
0
ppm
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8.47
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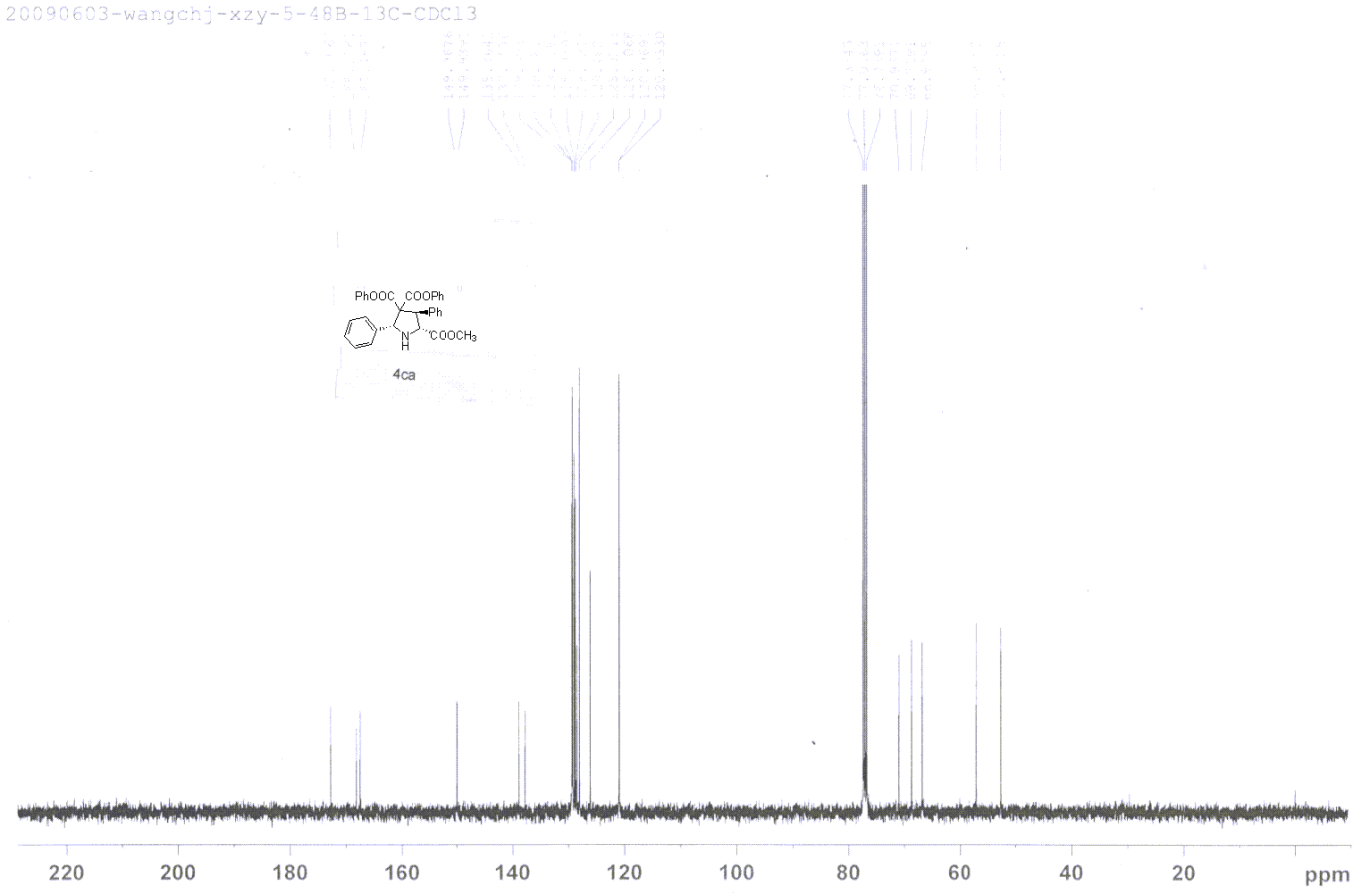
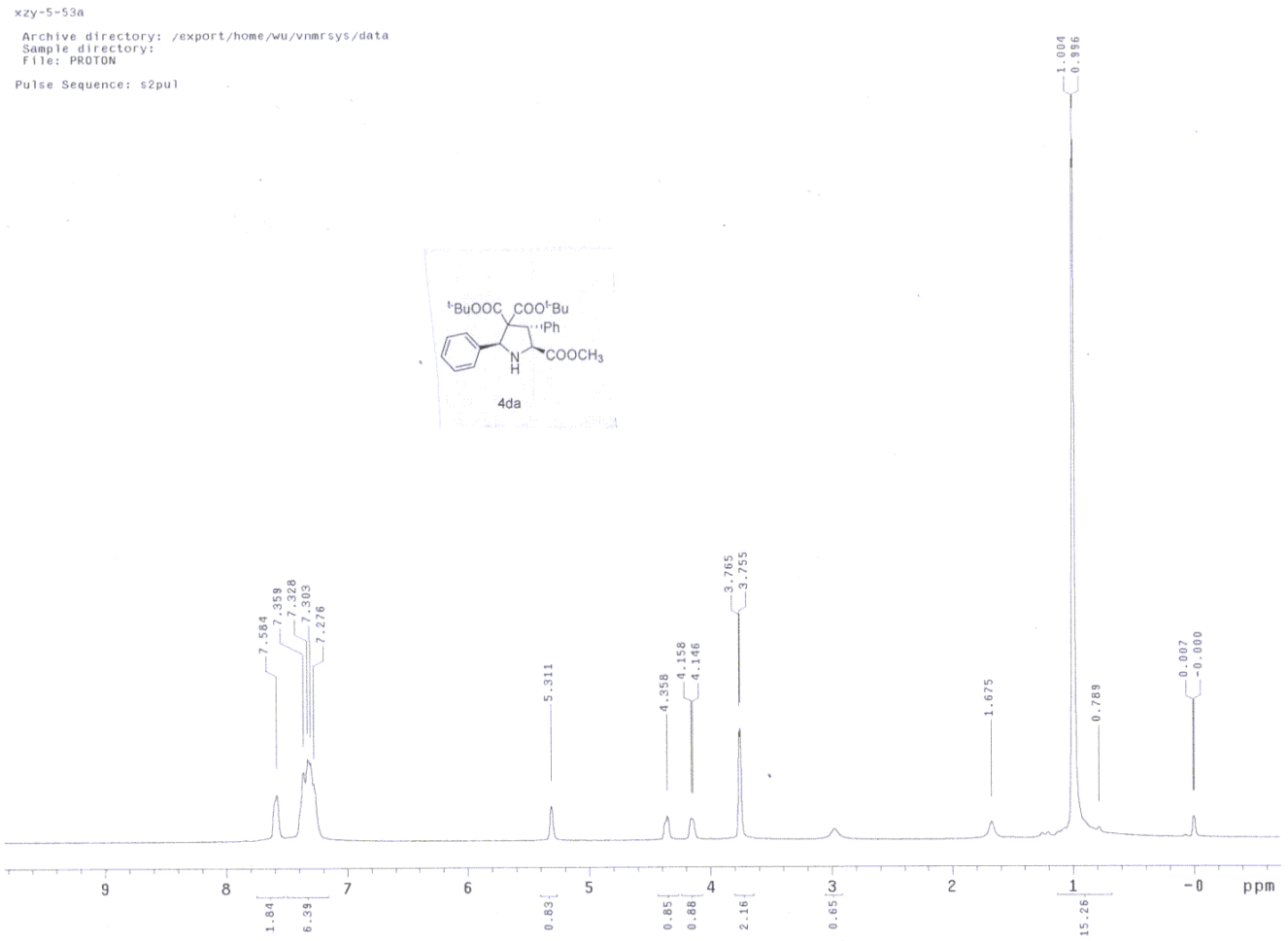
3.72
1.26
1.18
1.22
3.18
1.14
3.26




xzy-5-53a
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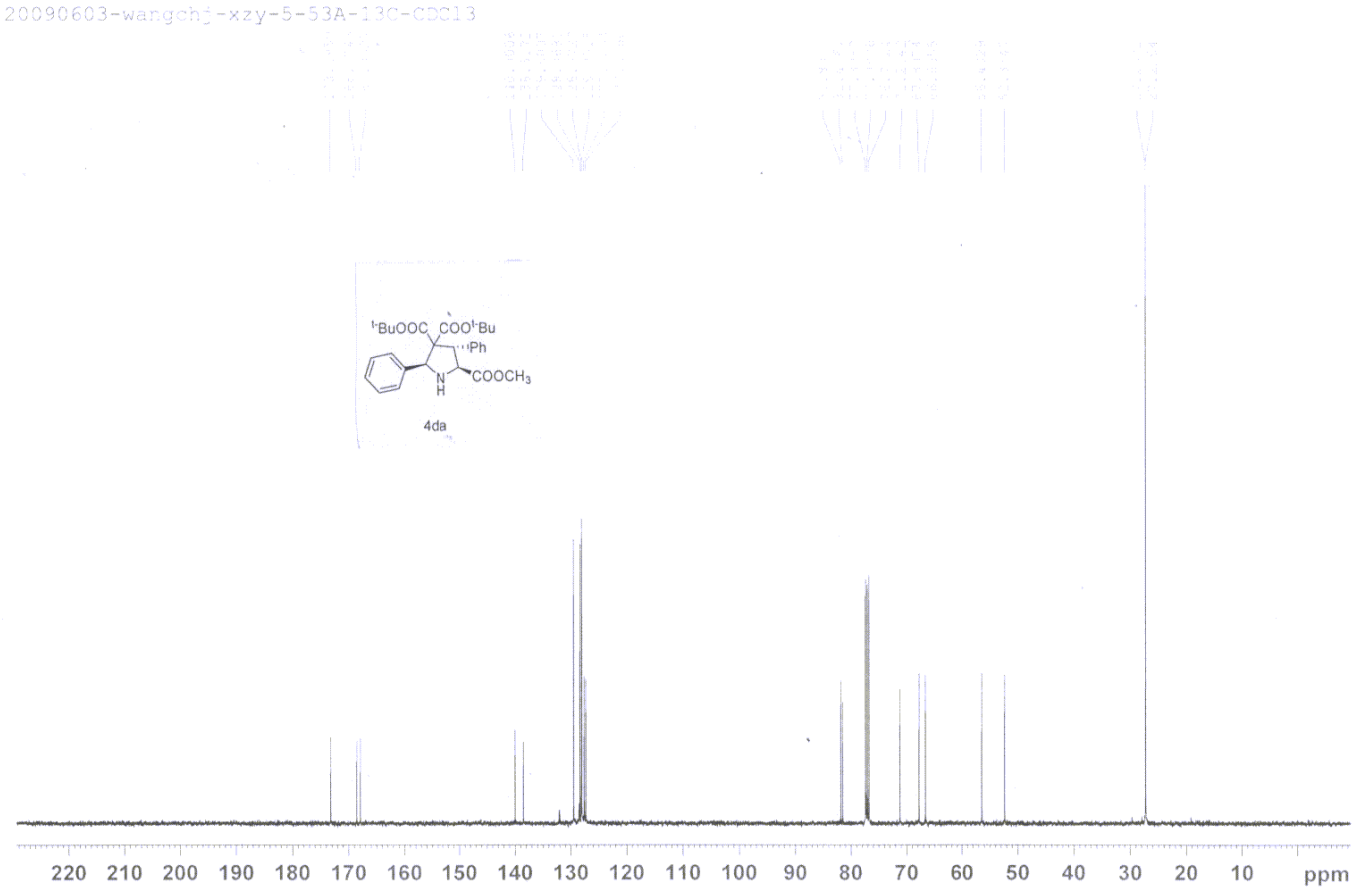
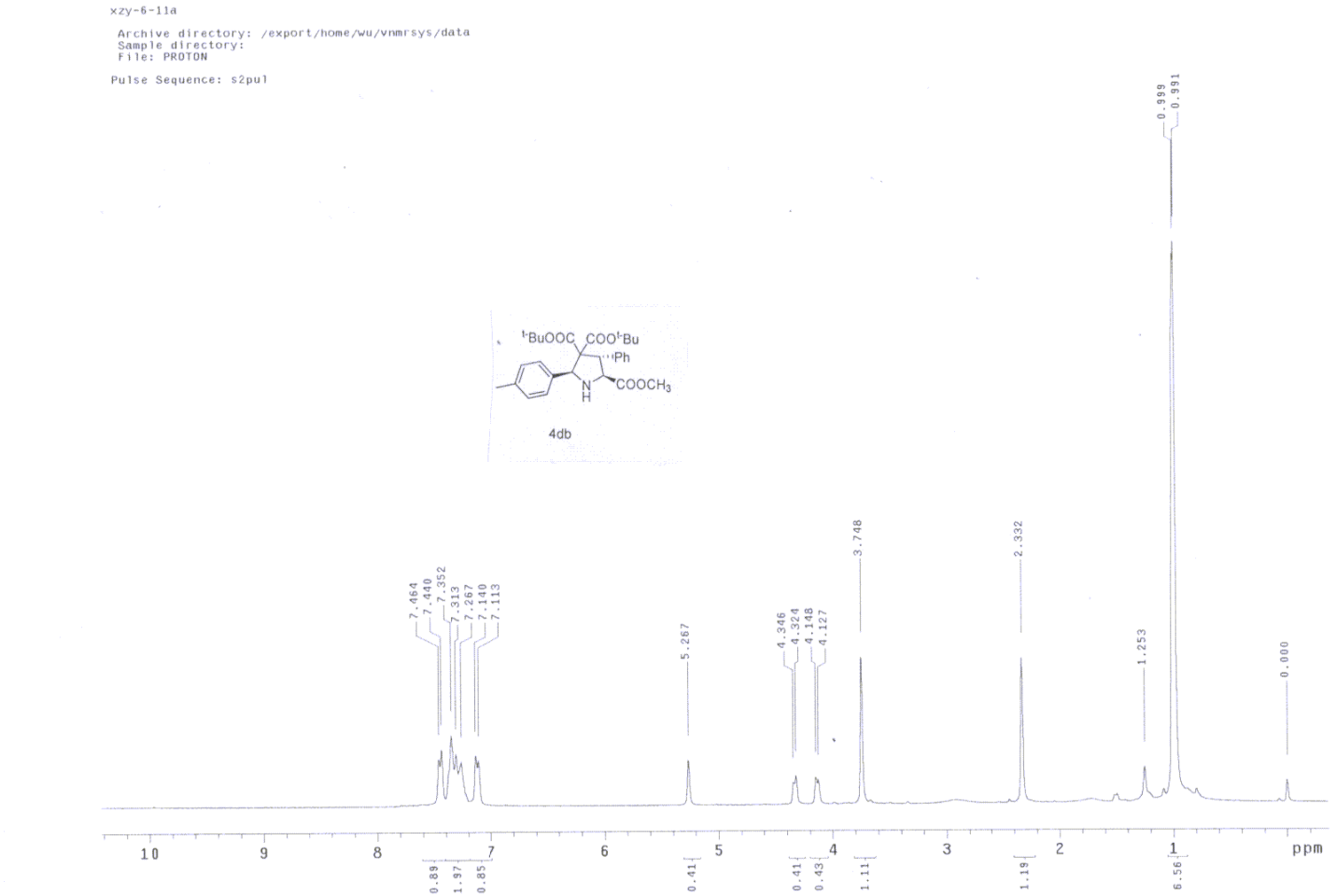
Sample directory:
File: PROTON
Pulse Sequence: s2pul
tBuOOC COOtBu
Ph
COOCH3
H
N
4da
7.584
7.359
7.328
7.303
7.276
5.311
4.358
4.158
4.146
3.765
3.755
1.675
1.004
0.996
0.789
0.007
-0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.84
6.39
0.83
0.85
0.88
2.16
0.65
15.26

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Sample directory:
File: PROTON
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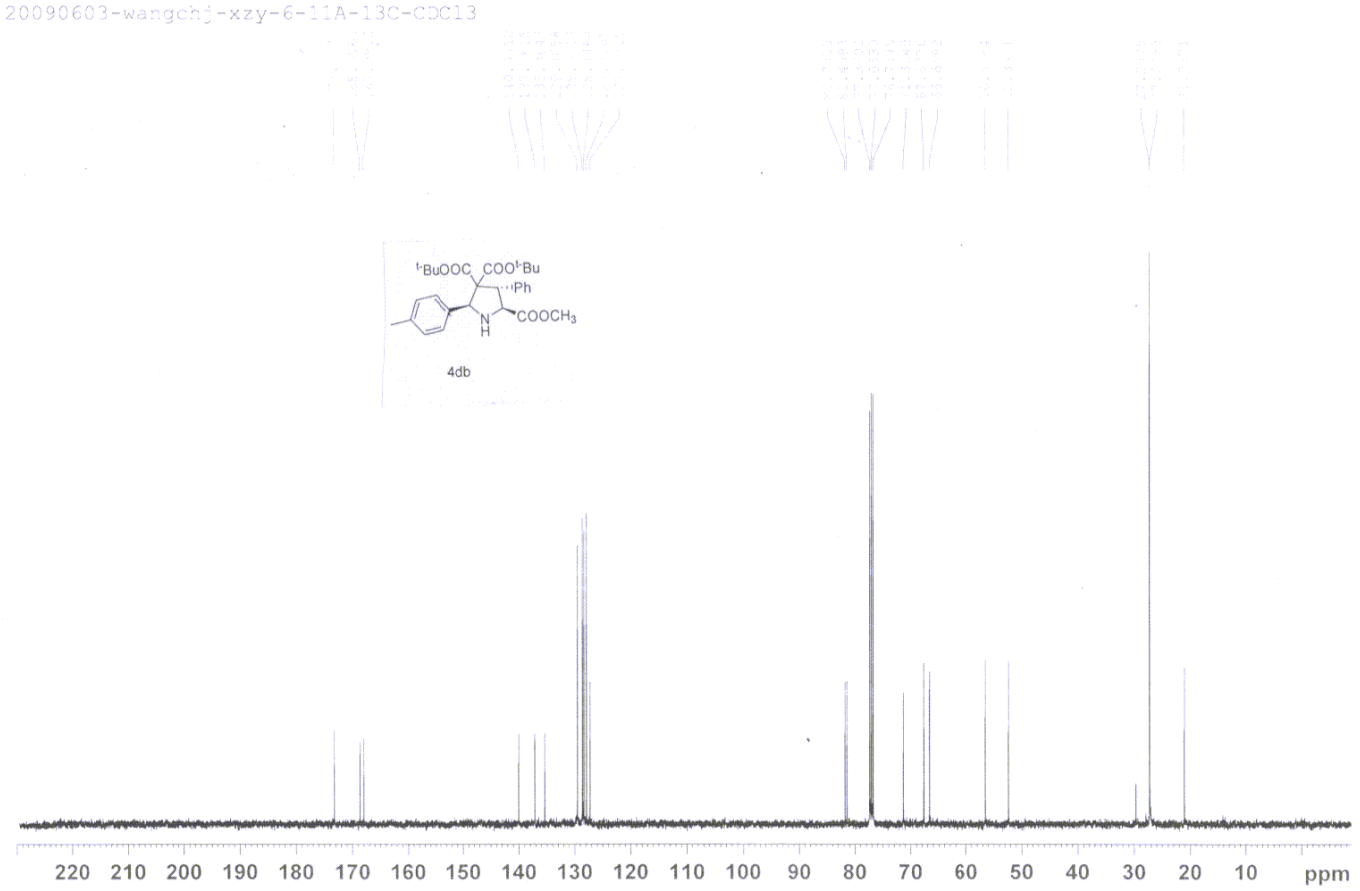
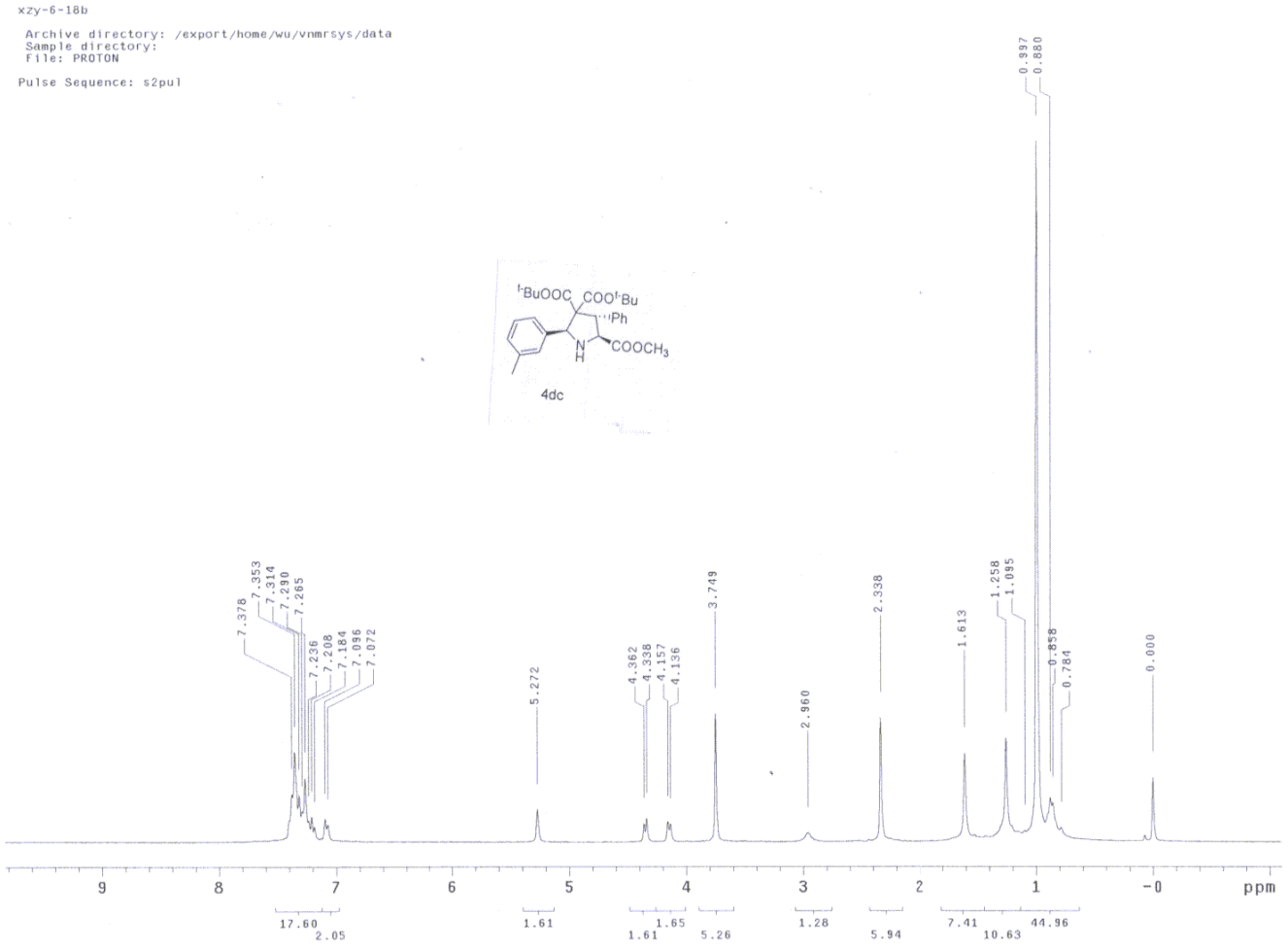
Ph
COOCH3
N
H
4db
0.999
0.991
7.464
7.440
7.352
7.313
7.267
7.140
7.113
5.267
4.346
4.324
4.148
4.127
3.748
2.332
1.253
0.000
10
9
8
7
6
5
4
3
2
1
ppm
0.89
1.97
0.85
0.41
0.41
0.43
1.11
1.19
6.56


20090603-wangchj-xzy-6-11A-13C-CDCl3
tBuOOC
COOtBu
Ph
COOCH3
N
H
4db
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm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Sample directory:
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Pulse Sequence: s2pul
tBuOOC
COOtBu
Ph
N
H
COOCH3
4dc
0.997
0.880
7.378
7.353
7.314
7.290
7.265
7.236
7.208
7.184
7.096
7.072
5.272
4.362
4.338
4.157
4.136
3.749
2.960
2.338
1.613
1.258
1.095
0.858
0.784
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
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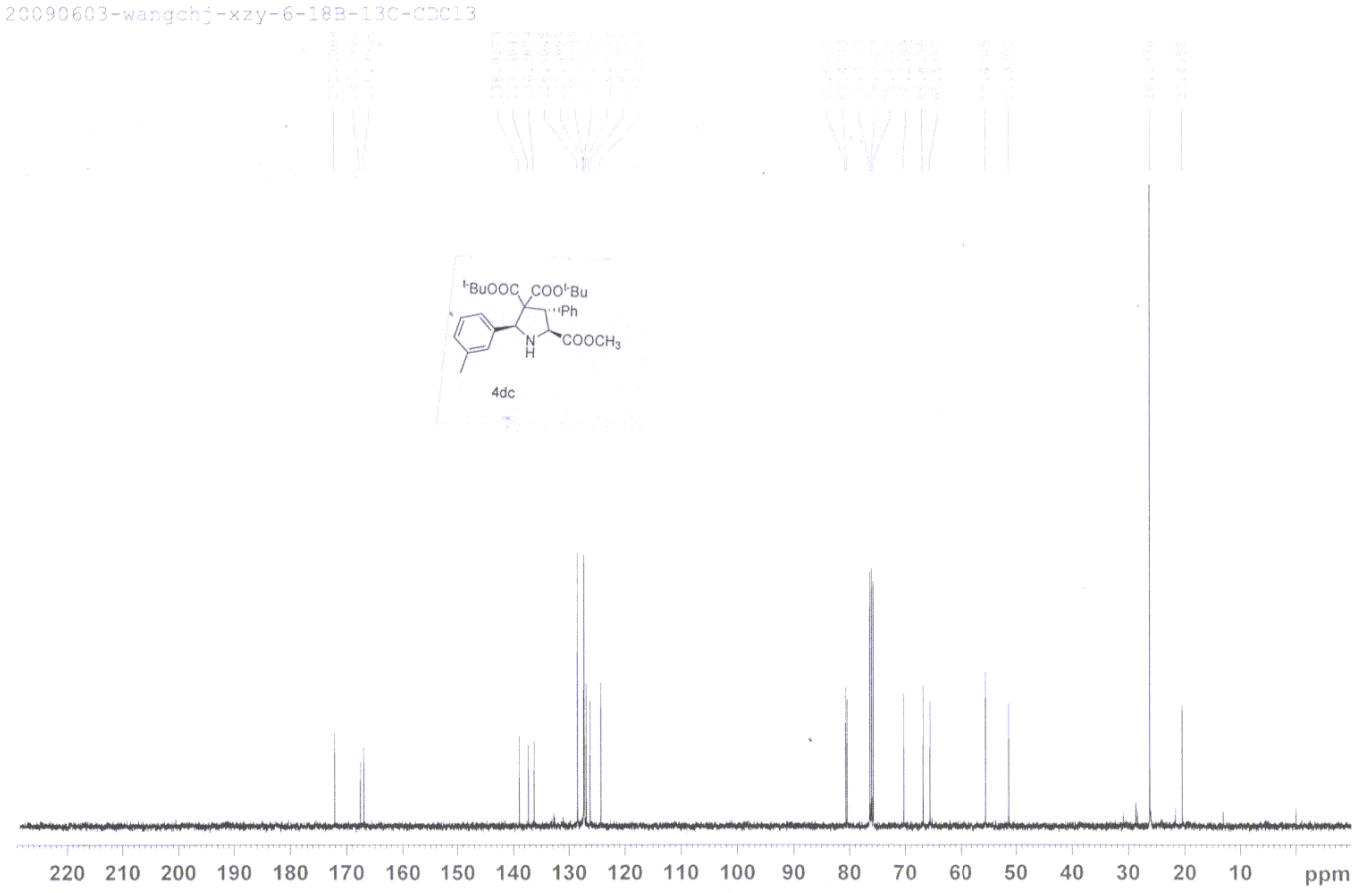
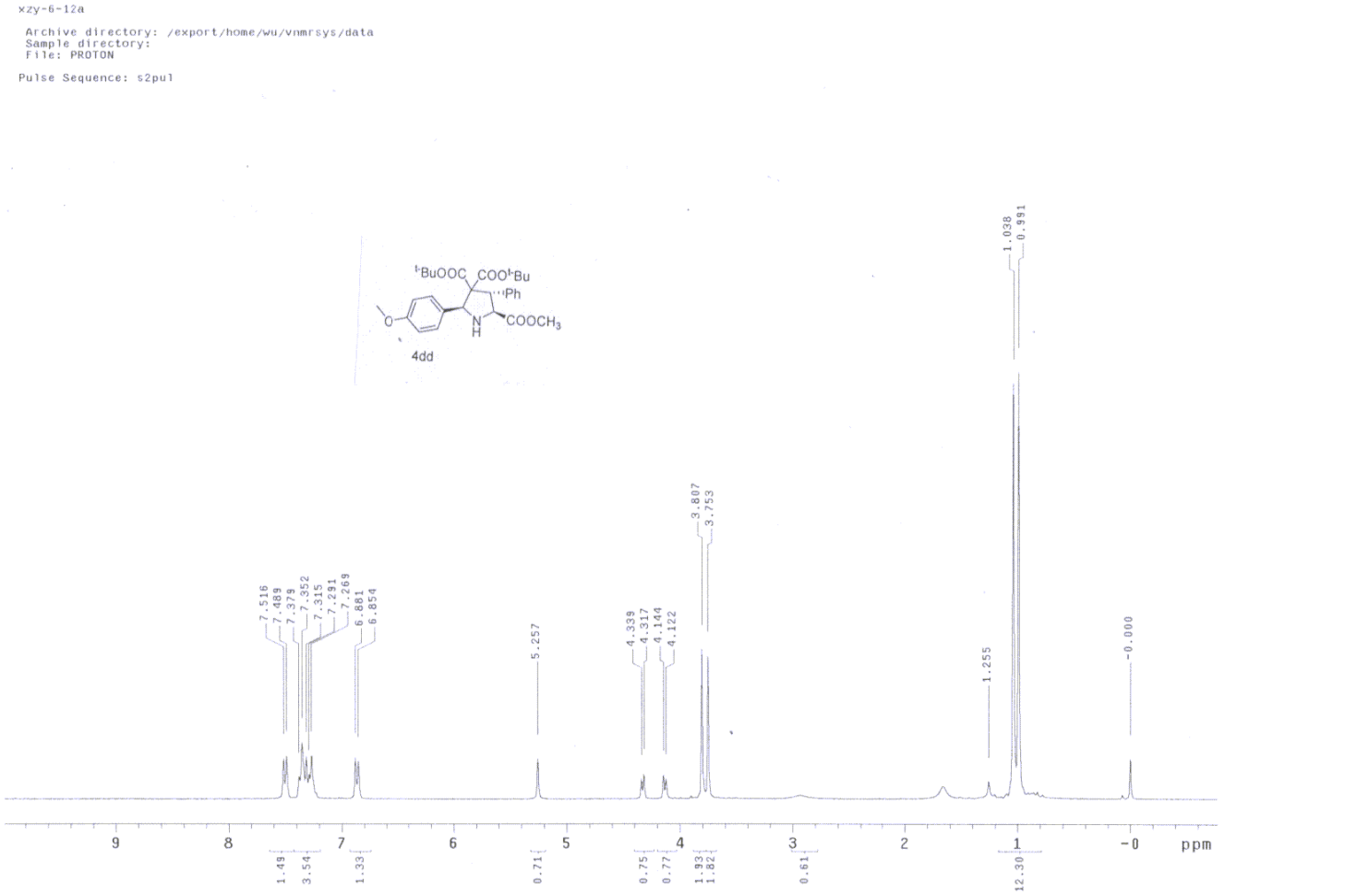
2.05
1.61
1.61
1.65
5.26
1.28
5.94
7.41
10.63
44.96




xzy-6-12a
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tBuOOC
COOtBu
Ph
COOCH3
O
N
H
4dd
7.516
7.489
7.379
7.352
7.315
7.291
7.269
6.881
6.854
5.257
4.339
4.317
4.144
4.122
3.807
3.753
1.255
1.038
0.991
0.000
9
8
7
6
5
4
3
2
1
0
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3.54
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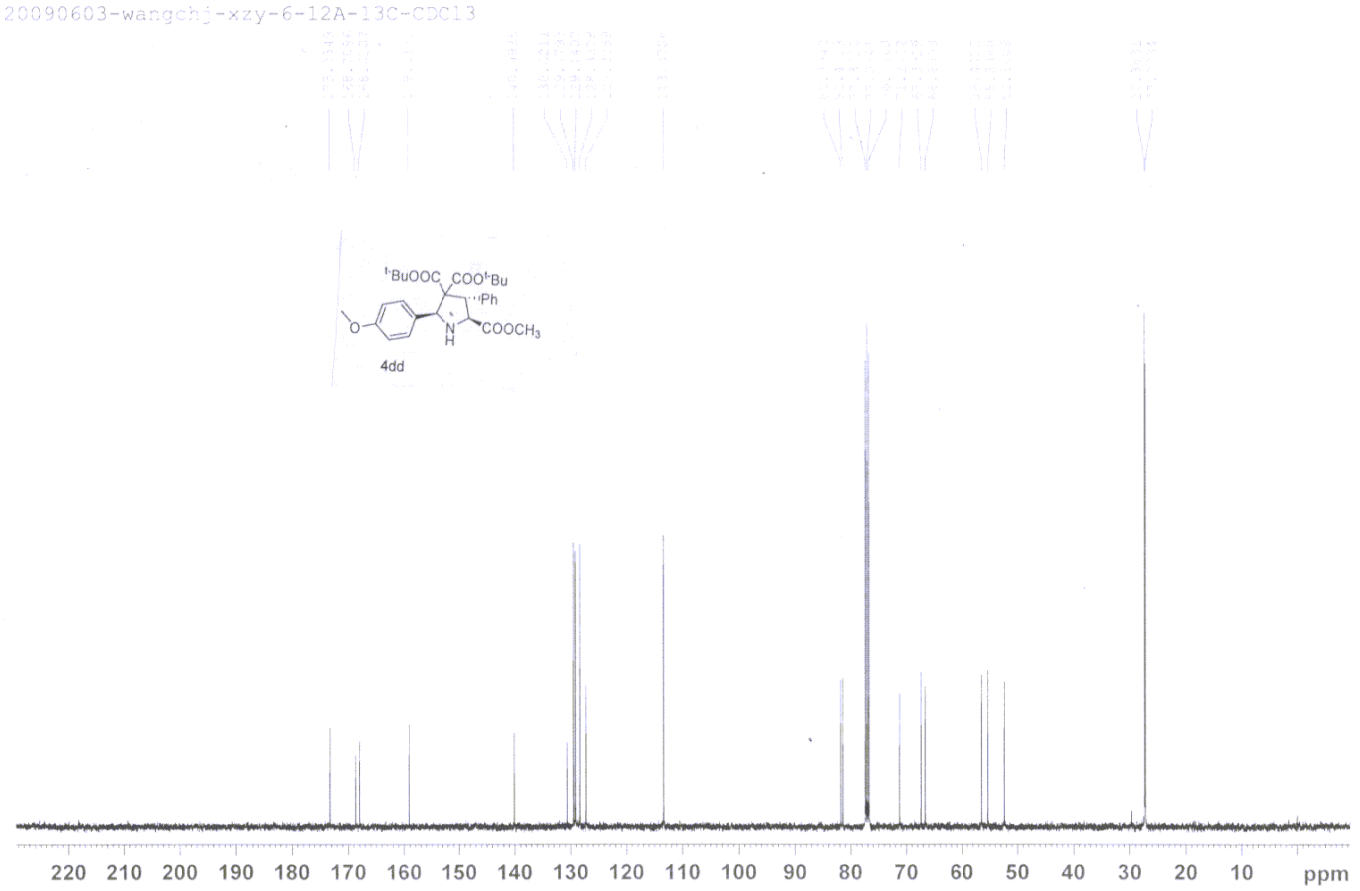
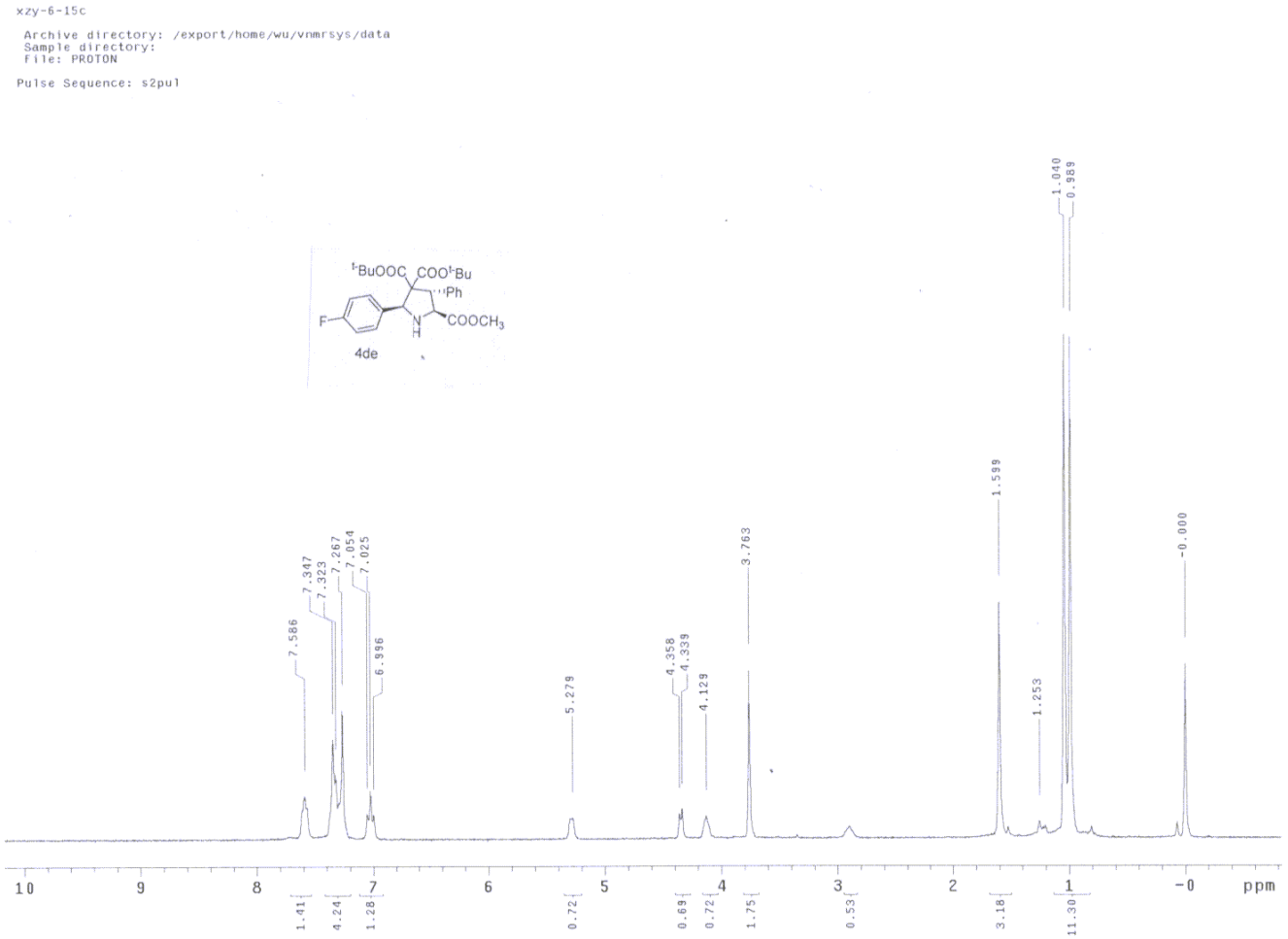
0.71
0.75
0.77
1.93
1.82
0.61
12.30

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Sample directory:
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Pulse Sequence: s2pul
tBuOOC COOtBu
Ph
F
N
H
COOCH3
4de
7.586
7.347
7.323
7.267
7.054
7.025
6.986
5.279
4.358
4.339
4.129
3.763
1.599
1.253
1.040
0.989
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
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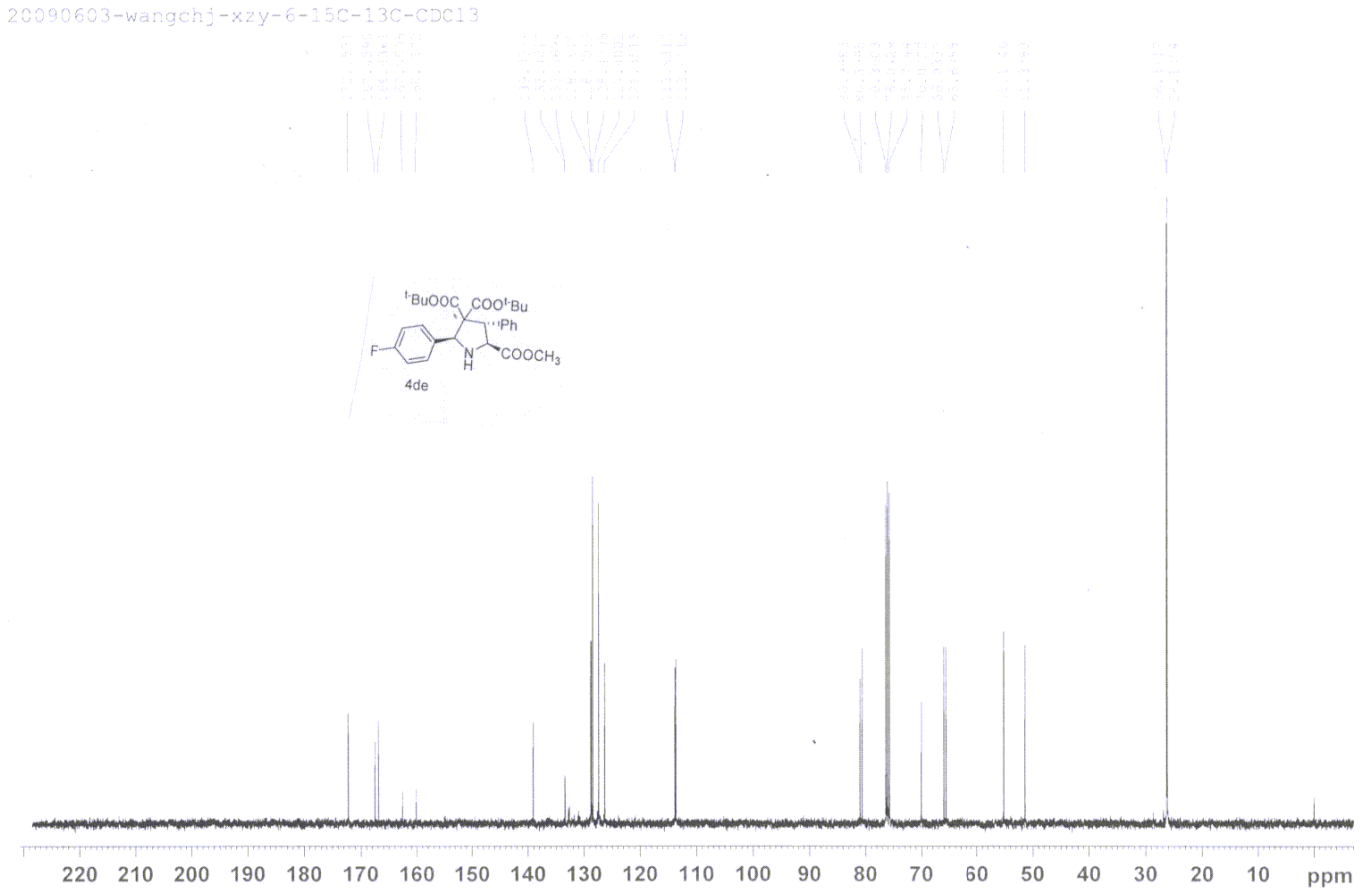
4.24
1.28
0.72
0.69
0.72
1.75
0.53
3.18
11.30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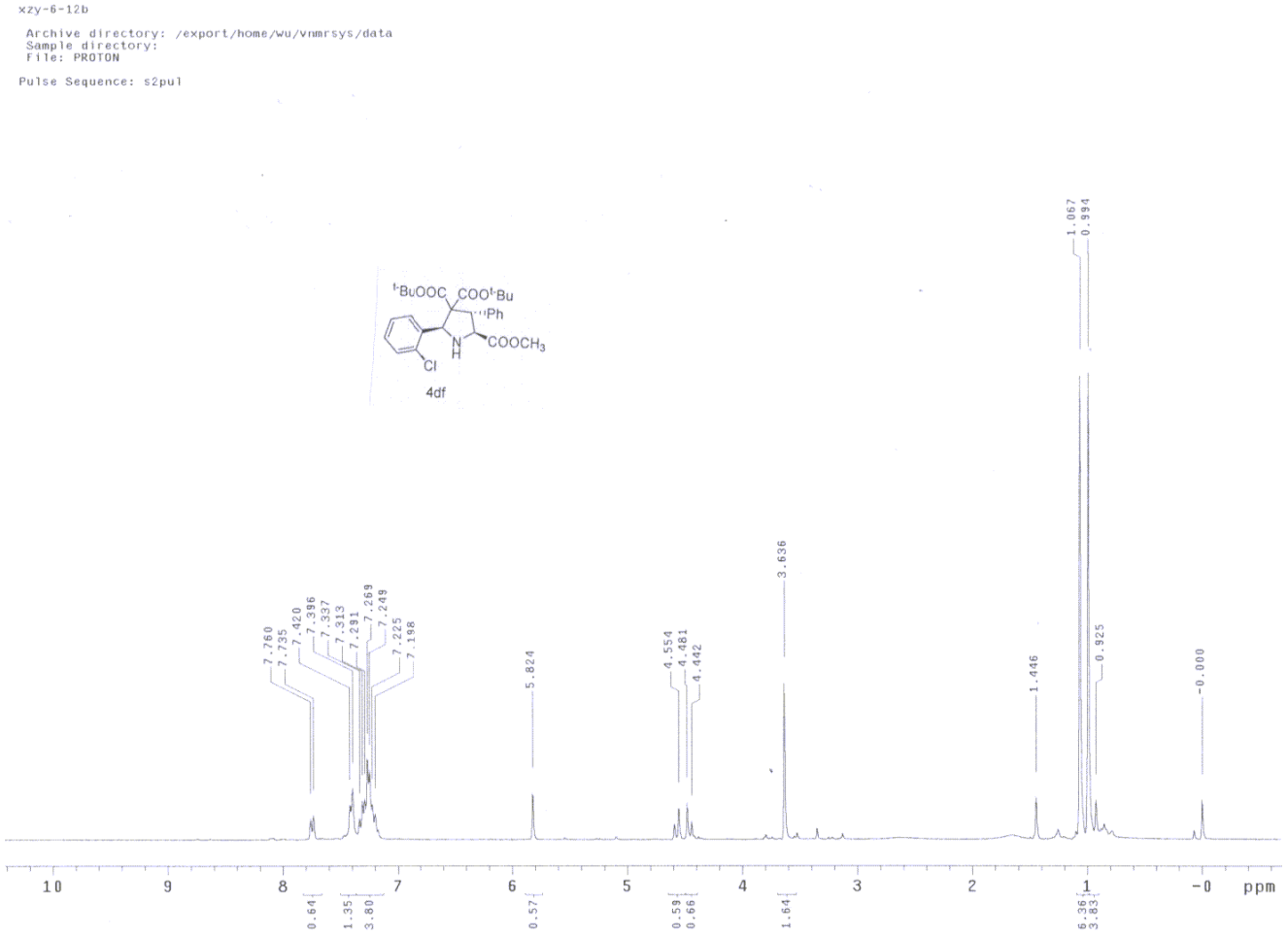
tBuOOC
COOtBu
Ph
F
N
H
COOCH3
4de
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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Sample directory:
File: PROTON
Pulse Sequence: s2pul
tBuOOC
COOtBu
Ph
COOCH3
Cl
N
H
4df
7.760
7.735
7.420
7.396
7.337
7.313
7.291
7.269
7.249
7.225
7.198
5.824
4.554
4.481
4.442
3.636
1.446
1.067
0.994
0.925
-0.000
10
9
8
7
6
5
4
3
2
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ppm
0.64
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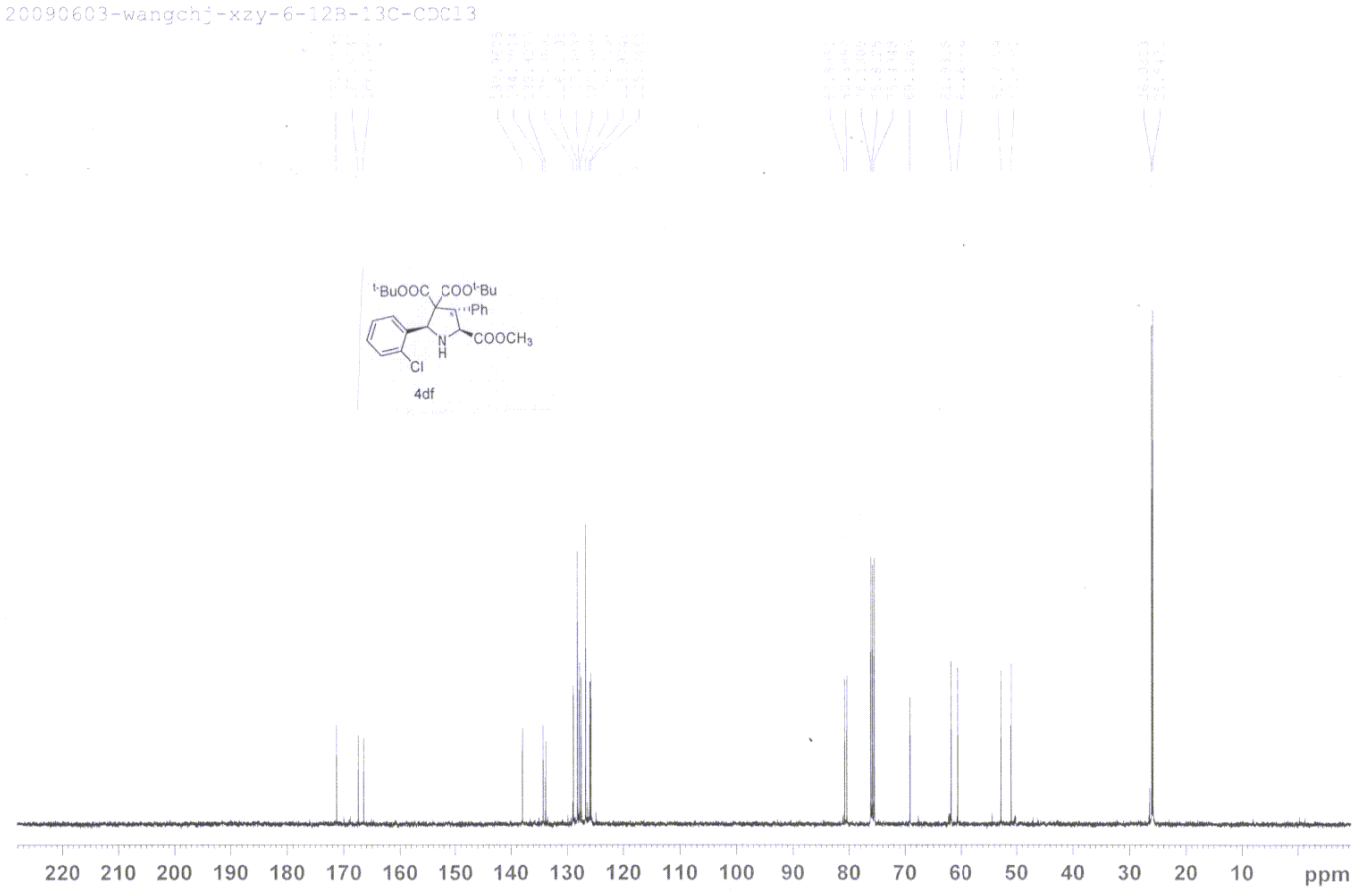
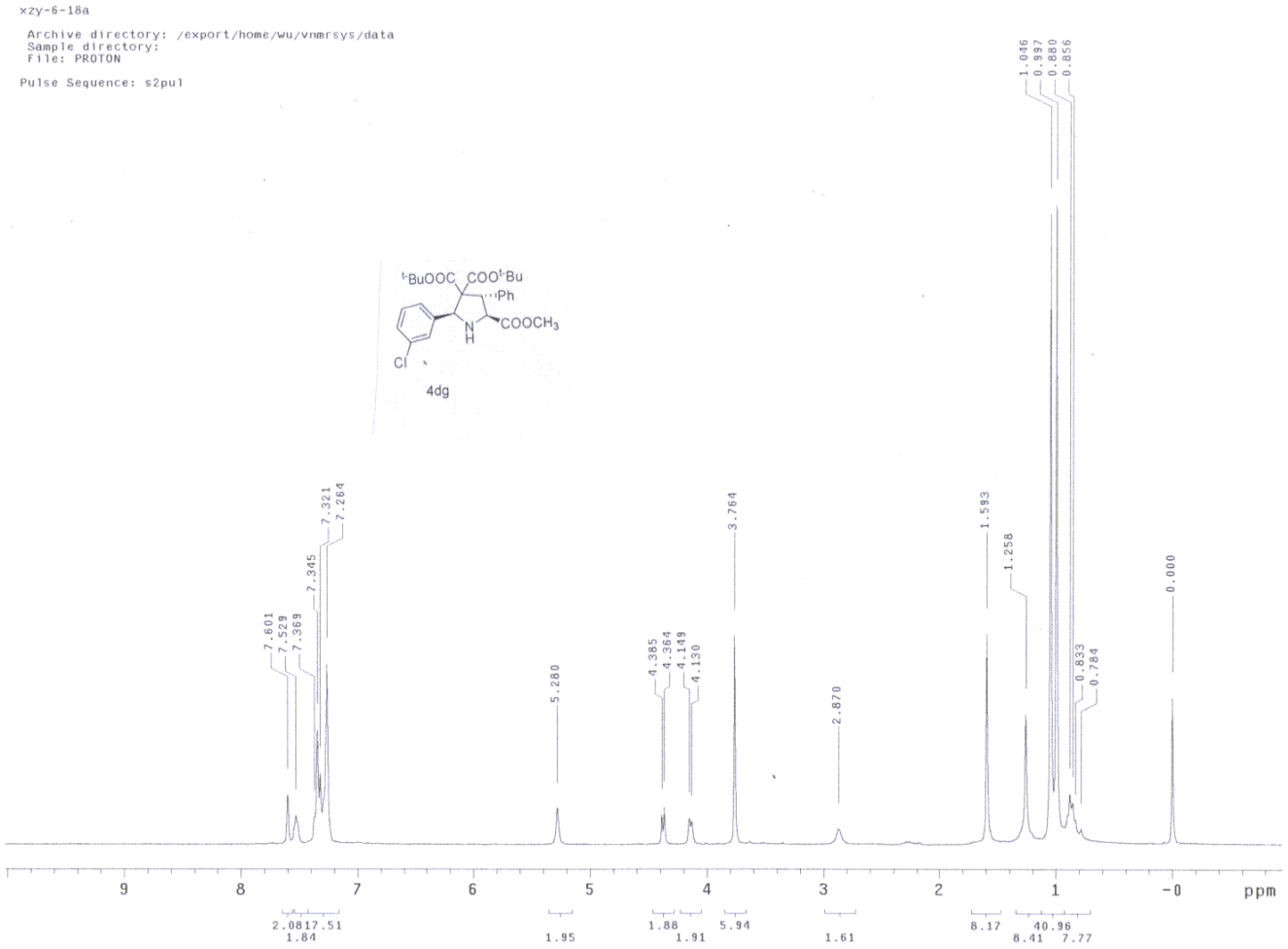
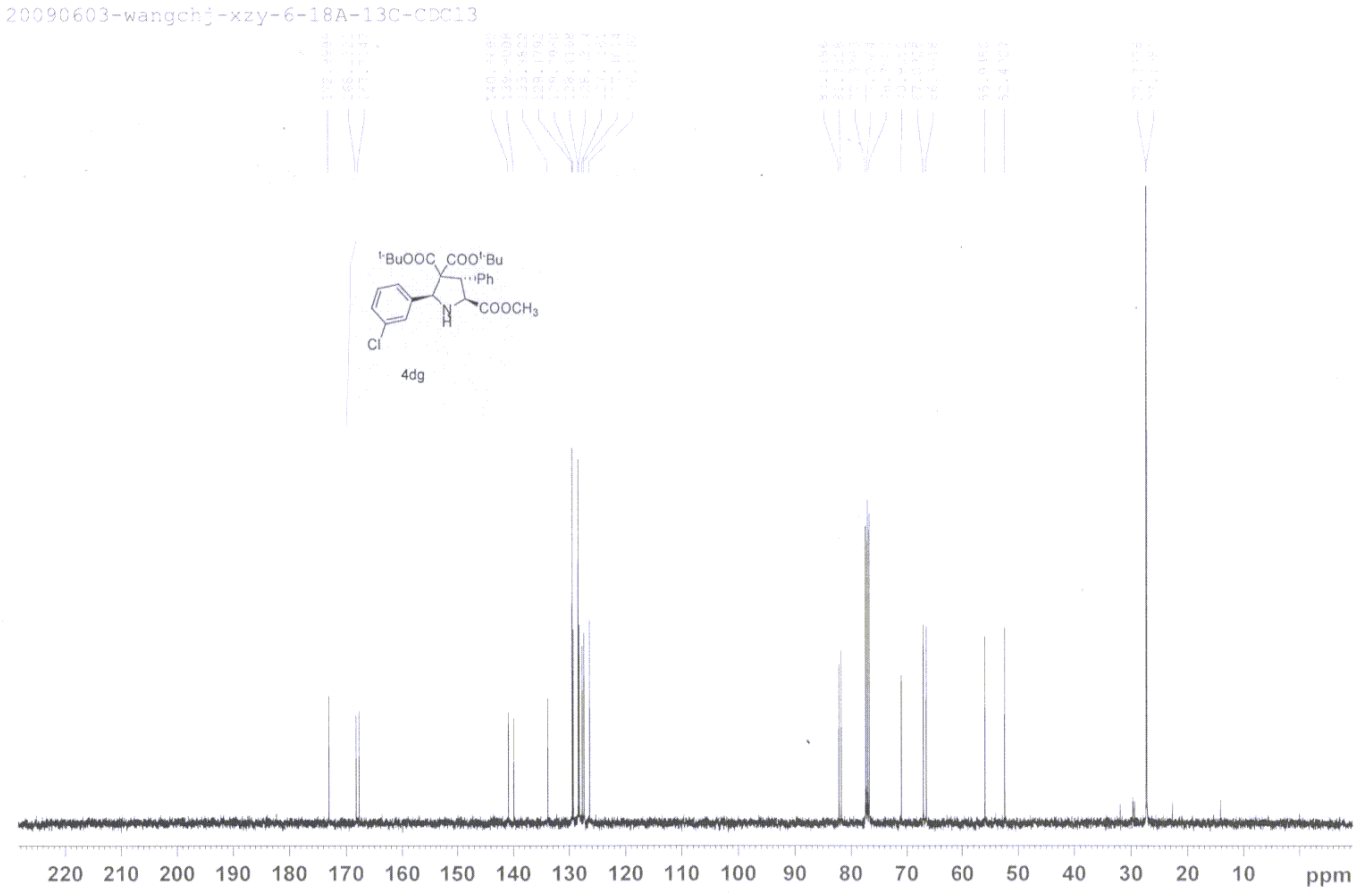
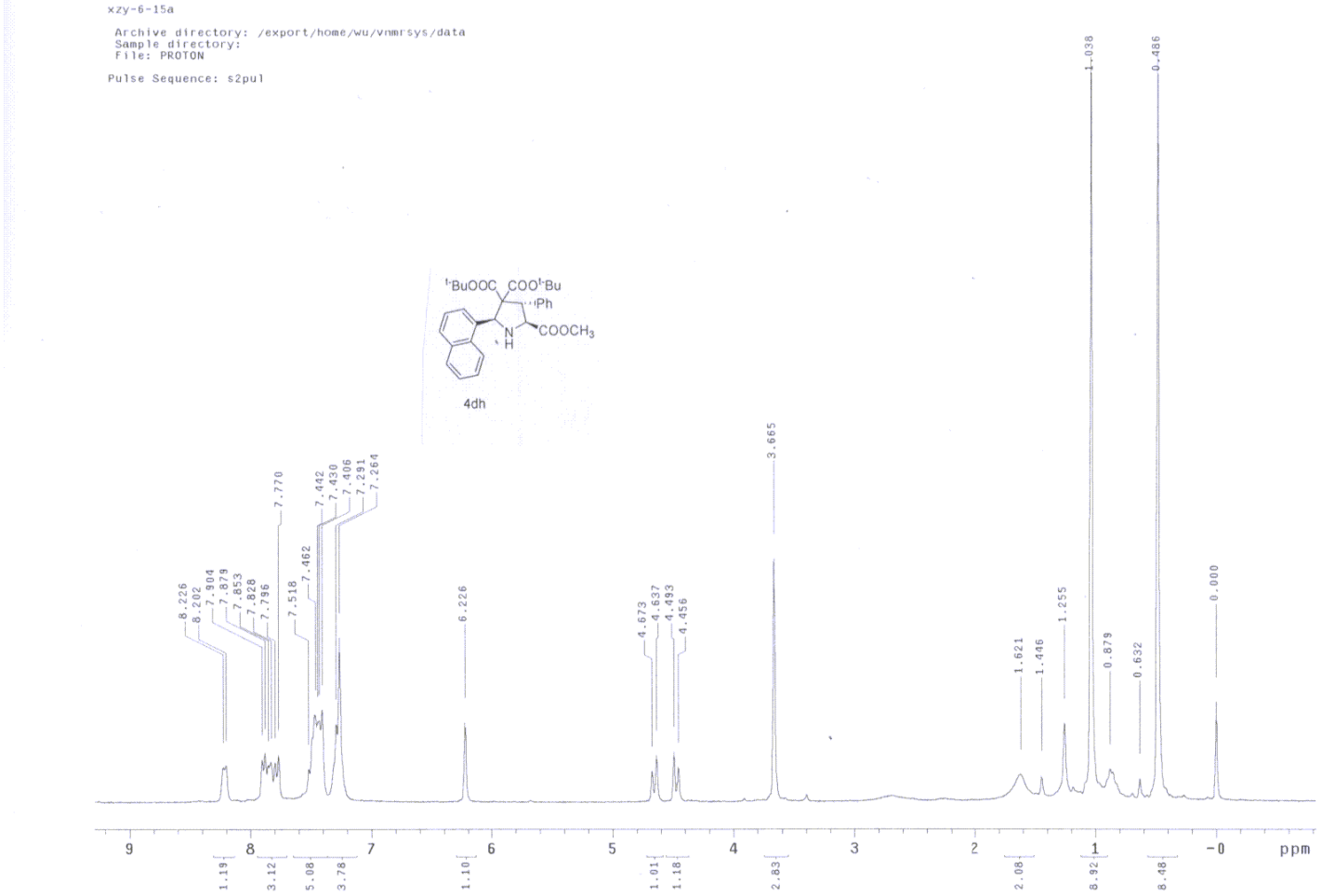
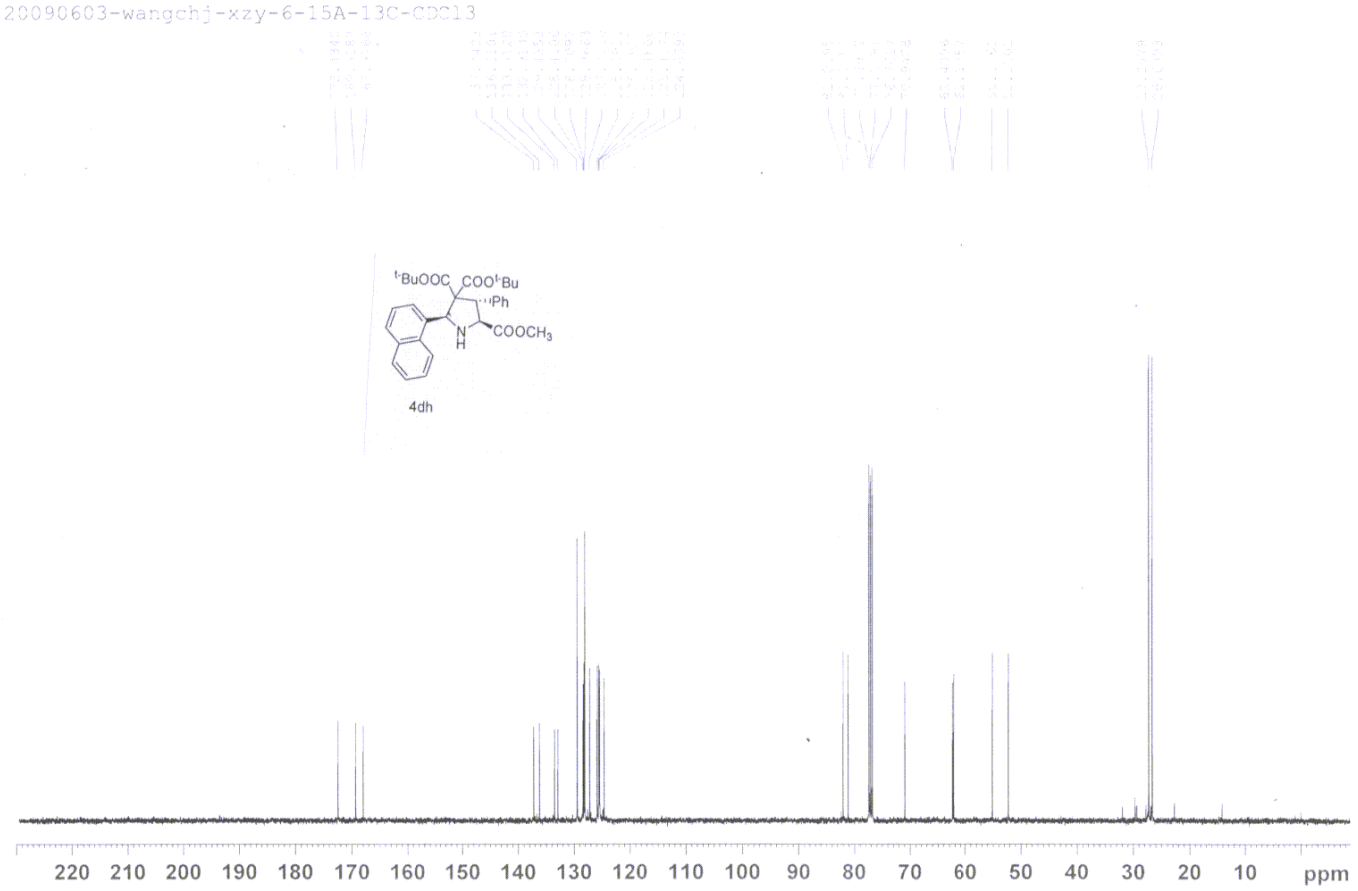
0.59
0.66
1.64
6.36
3.83

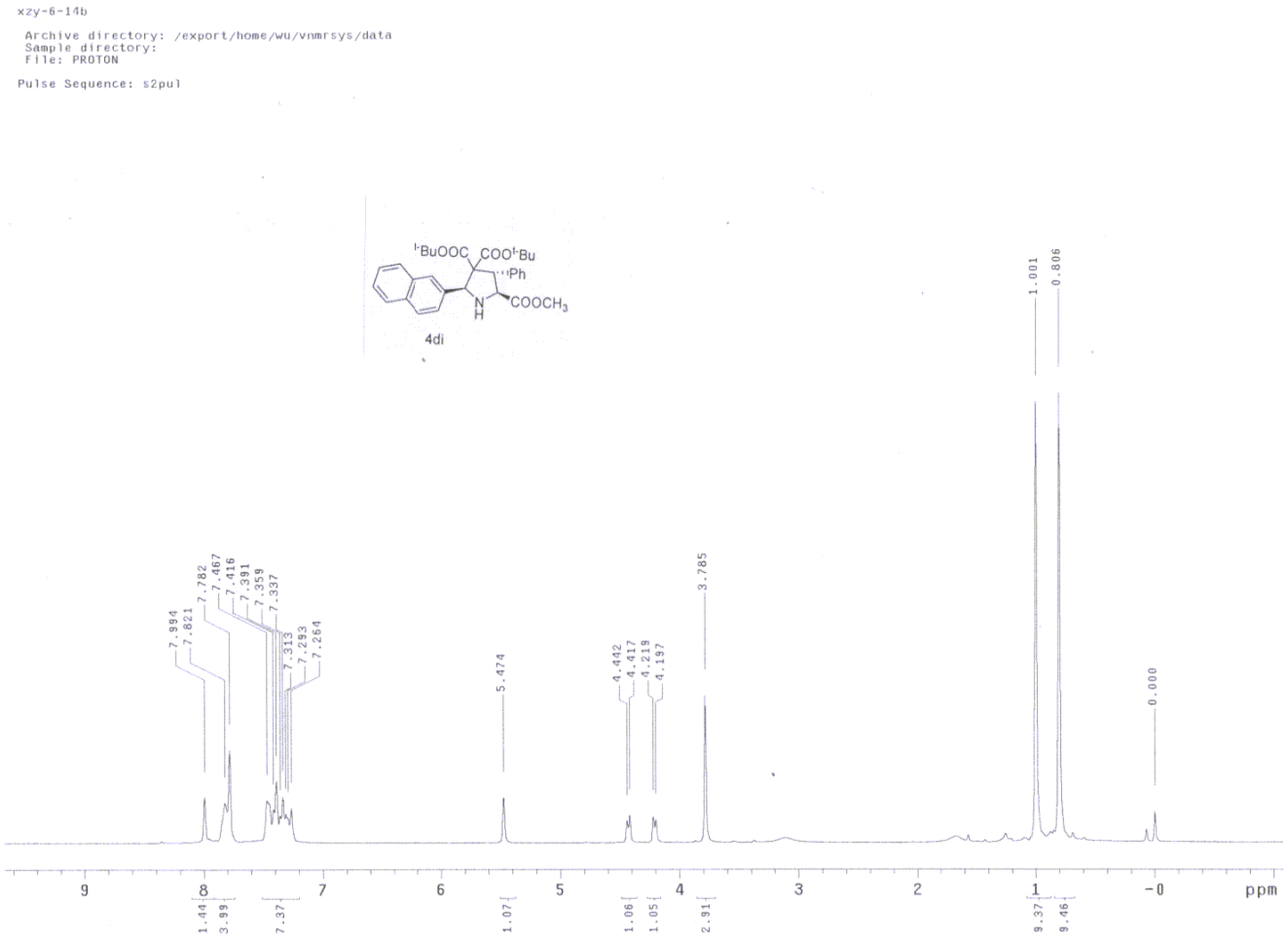





20090603-wangchj-xzy-6-18A-13C-CDCl3
tBuOOC
COOtBu
Ph
COOCH3
Cl
H
N
4dg
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm



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COOtBu
Ph
COOCH3
N
H
4di
7.994
7.821
7.782
7.467
7.416
7.391
7.359
7.337
7.313
7.293
7.264
5.474
4.442
4.417
4.219
4.197
3.785
1.001
0.806
0.000
9
8
7
6
5
4
3
2
1
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ppm
1.44
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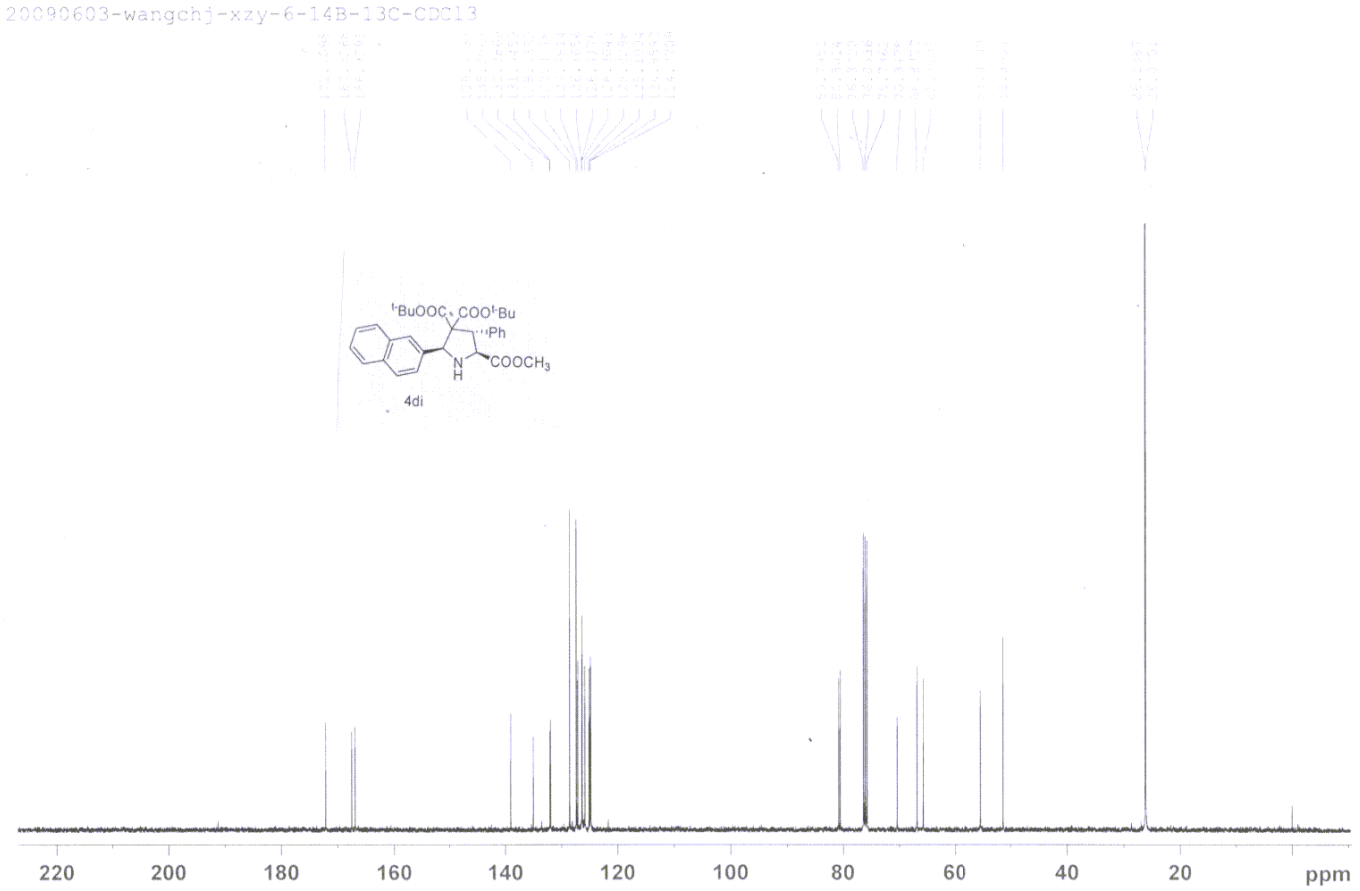
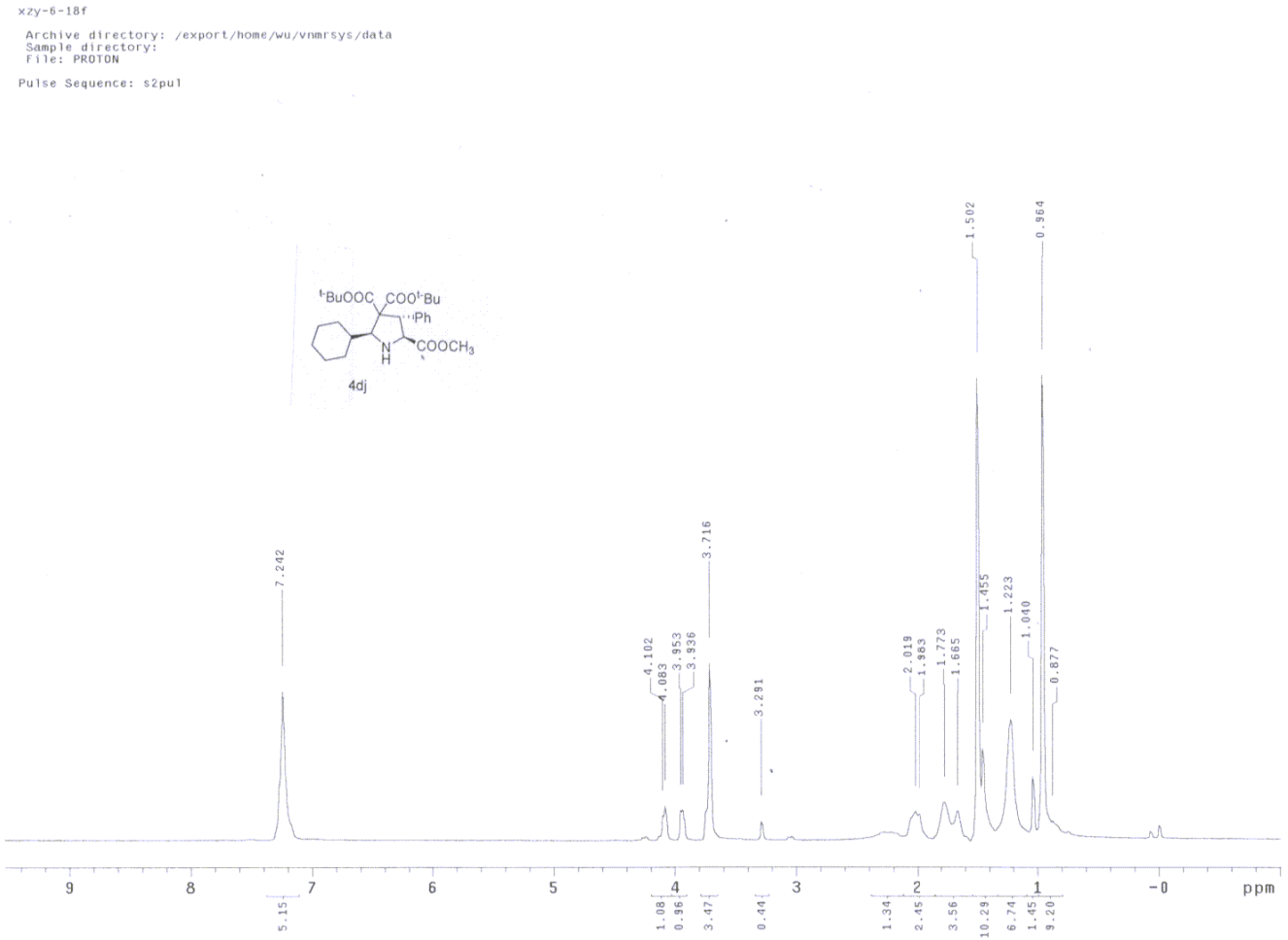
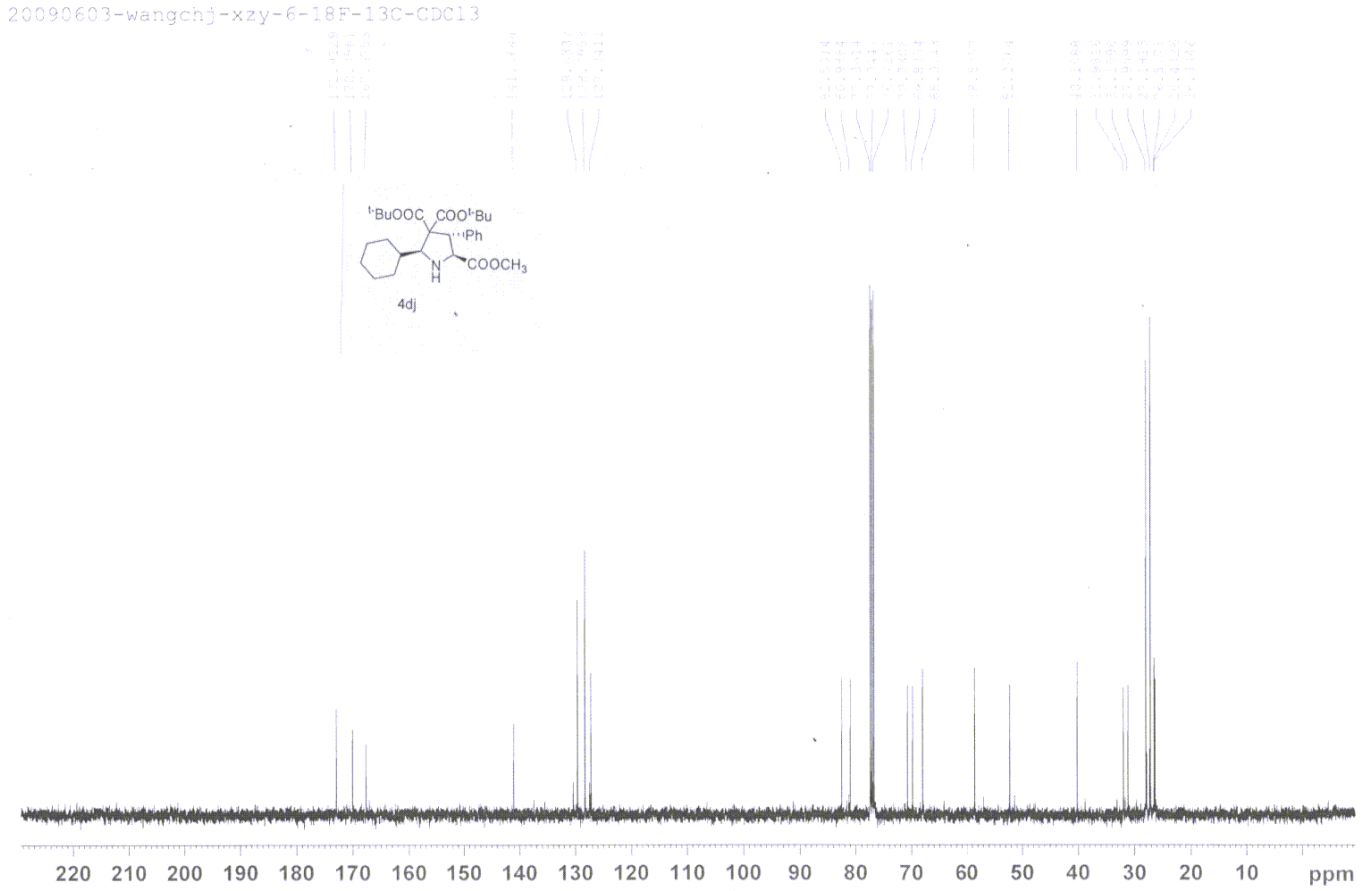
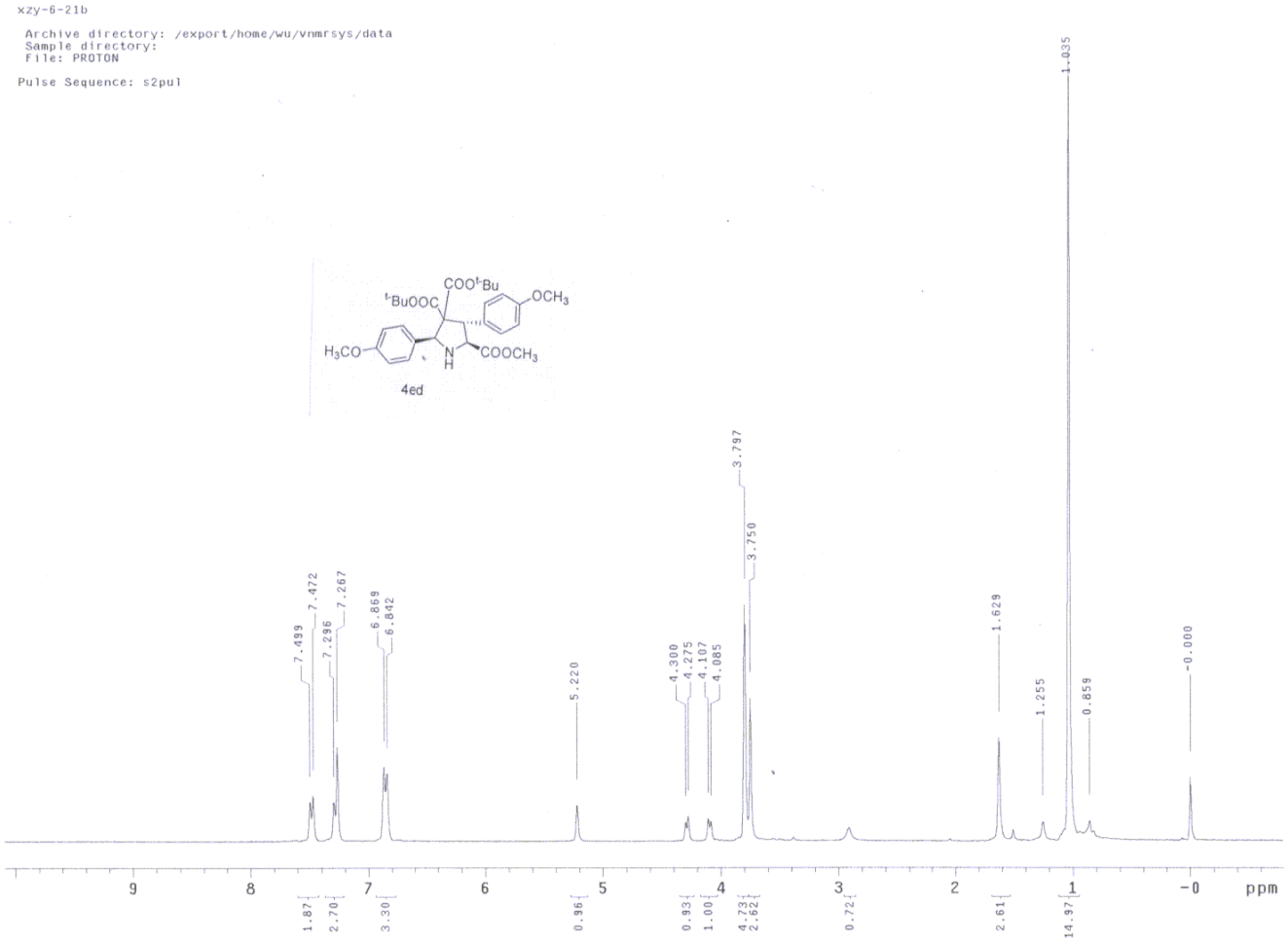
7.37
1.07
1.06
1.05
2.91
9.37
9.46








xzy-6-21b
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Sample directory:
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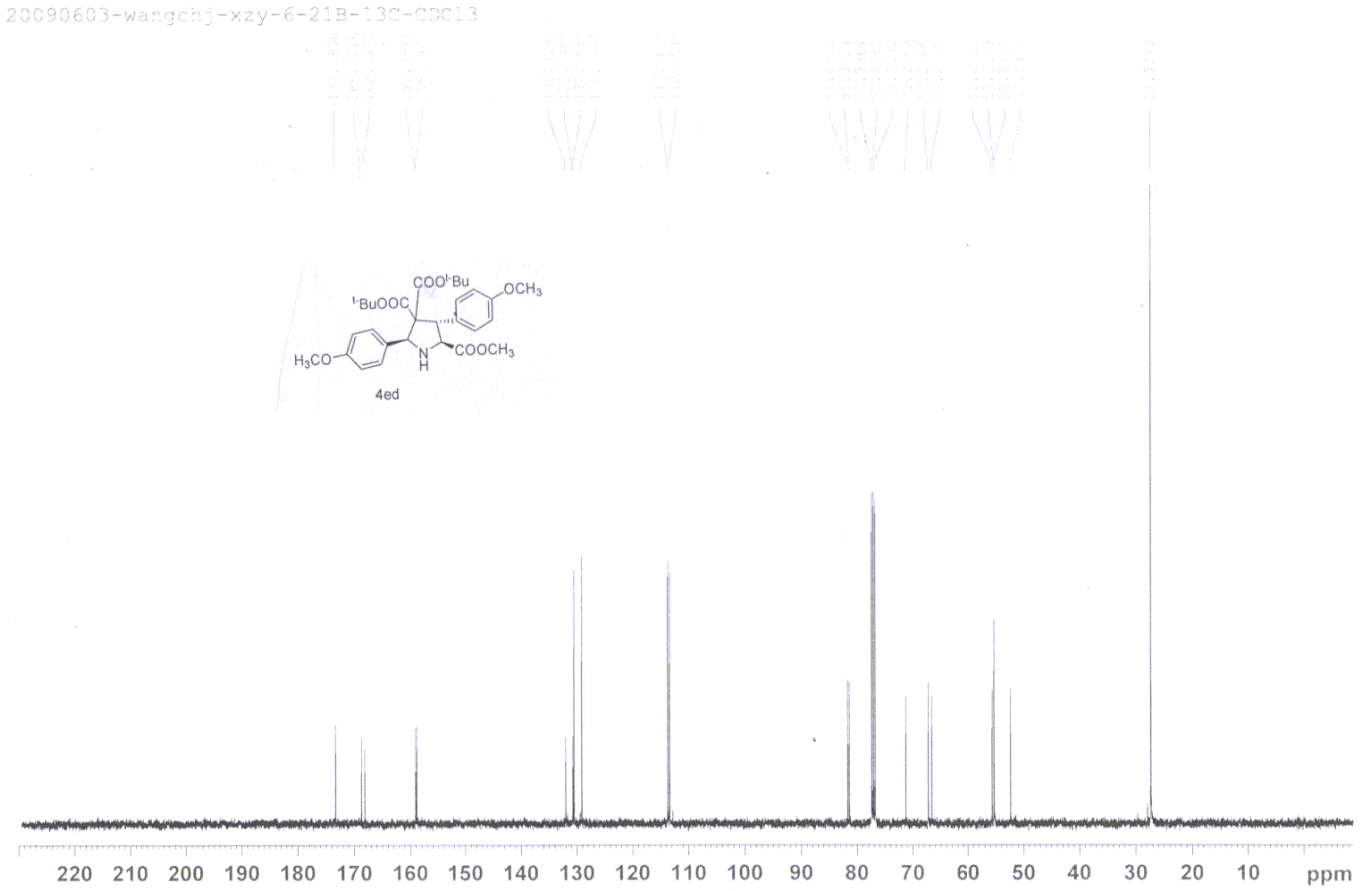
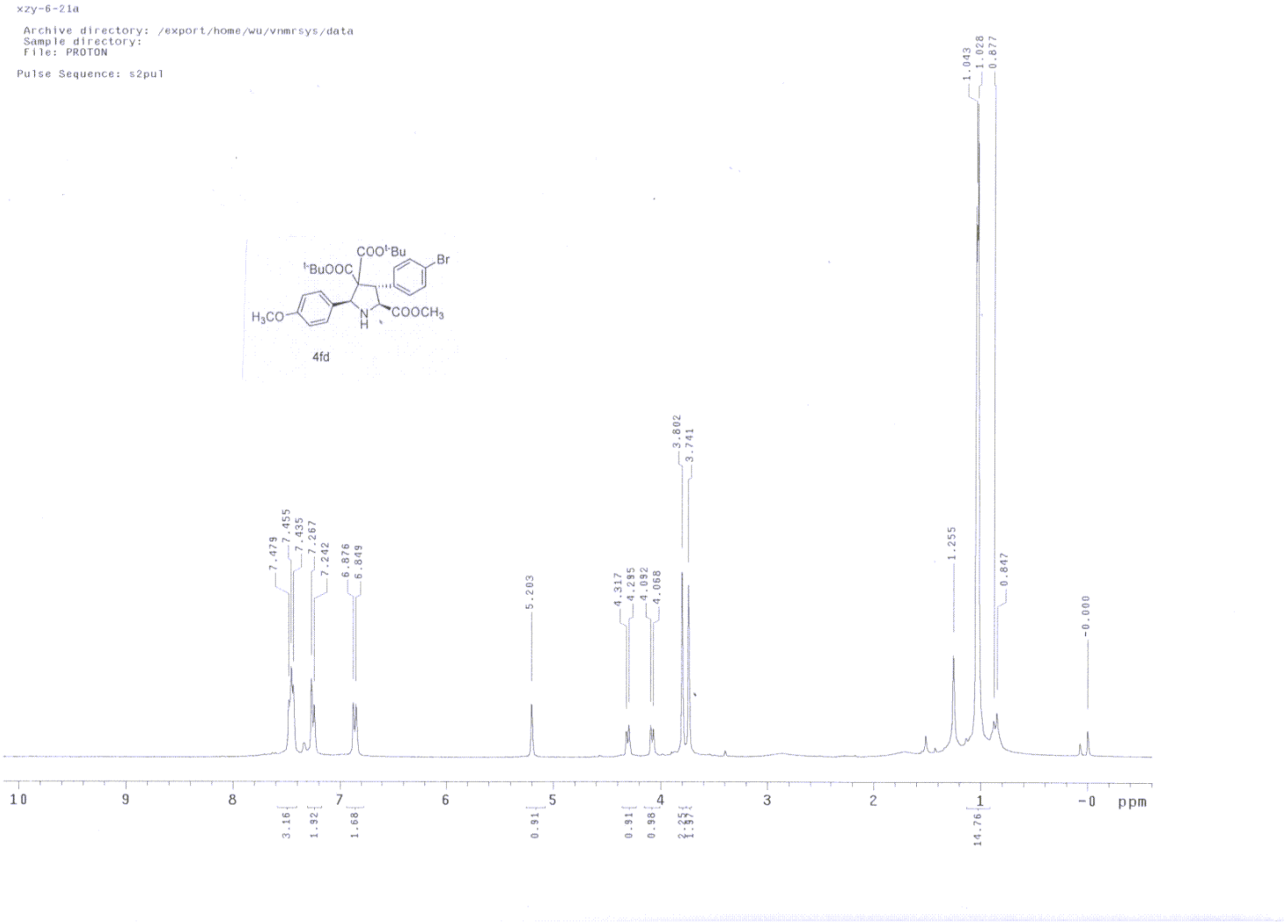
^{t}BuOOC
OCH$_3$
H$_3$CO
COOCH$_3$
N
H
4ed
7.499
7.472
7.296
7.267
6.869
6.842
5.220
4.300
4.275
4.107
4.085
3.797
3.750
1.629
1.255
1.035
0.859
-0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.87
2.70
3.30
0.96
0.93
1.00
4.73
2.62
0.72
2.61
14.97


20090603-wangchj-xzy-6-21B-13C-CDCl3
COO^{t}Bu
^{t}BuOOC
OCH$_3$
H$_3$CO
N
H
COOCH$_3$
4ed
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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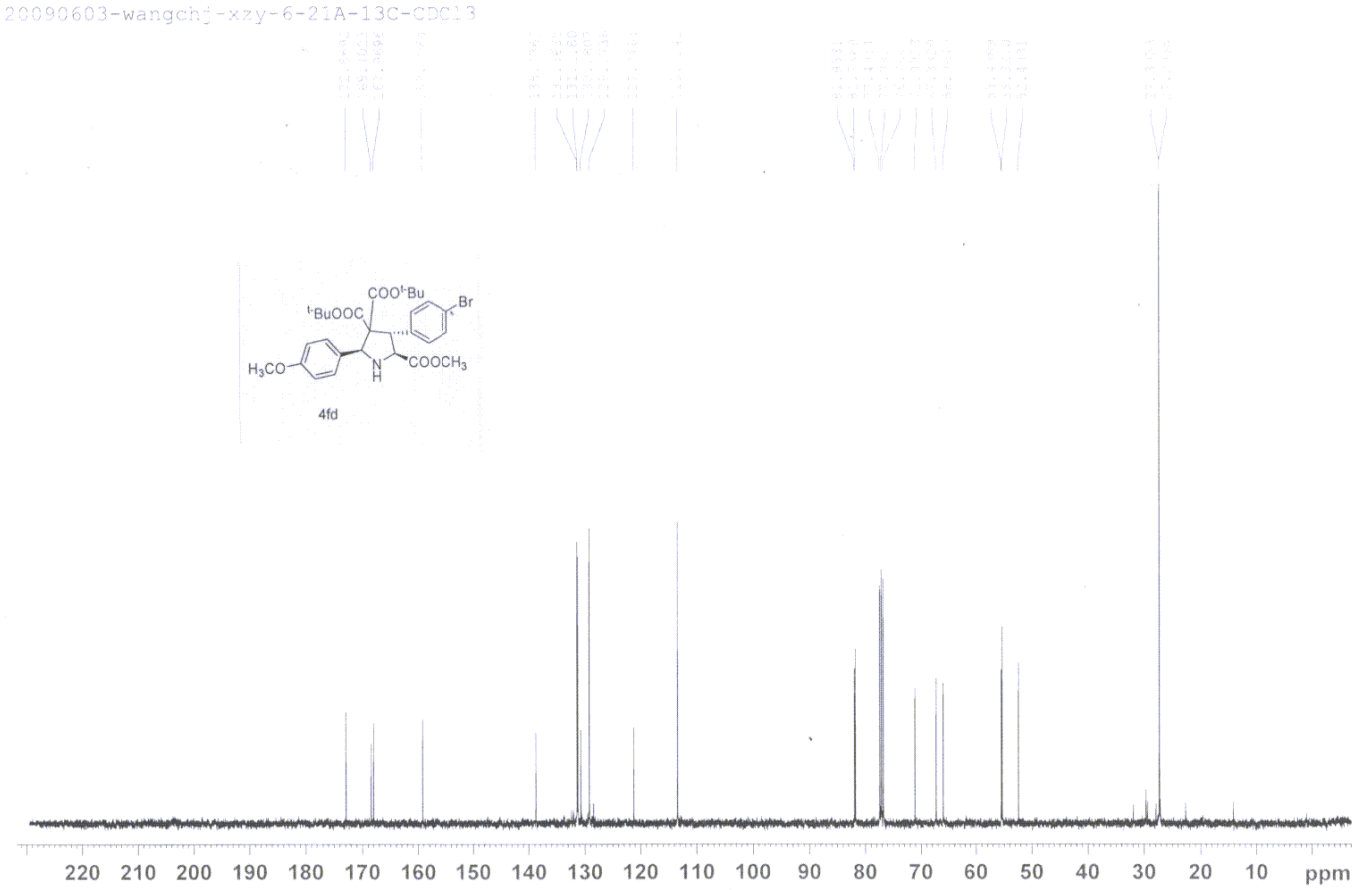
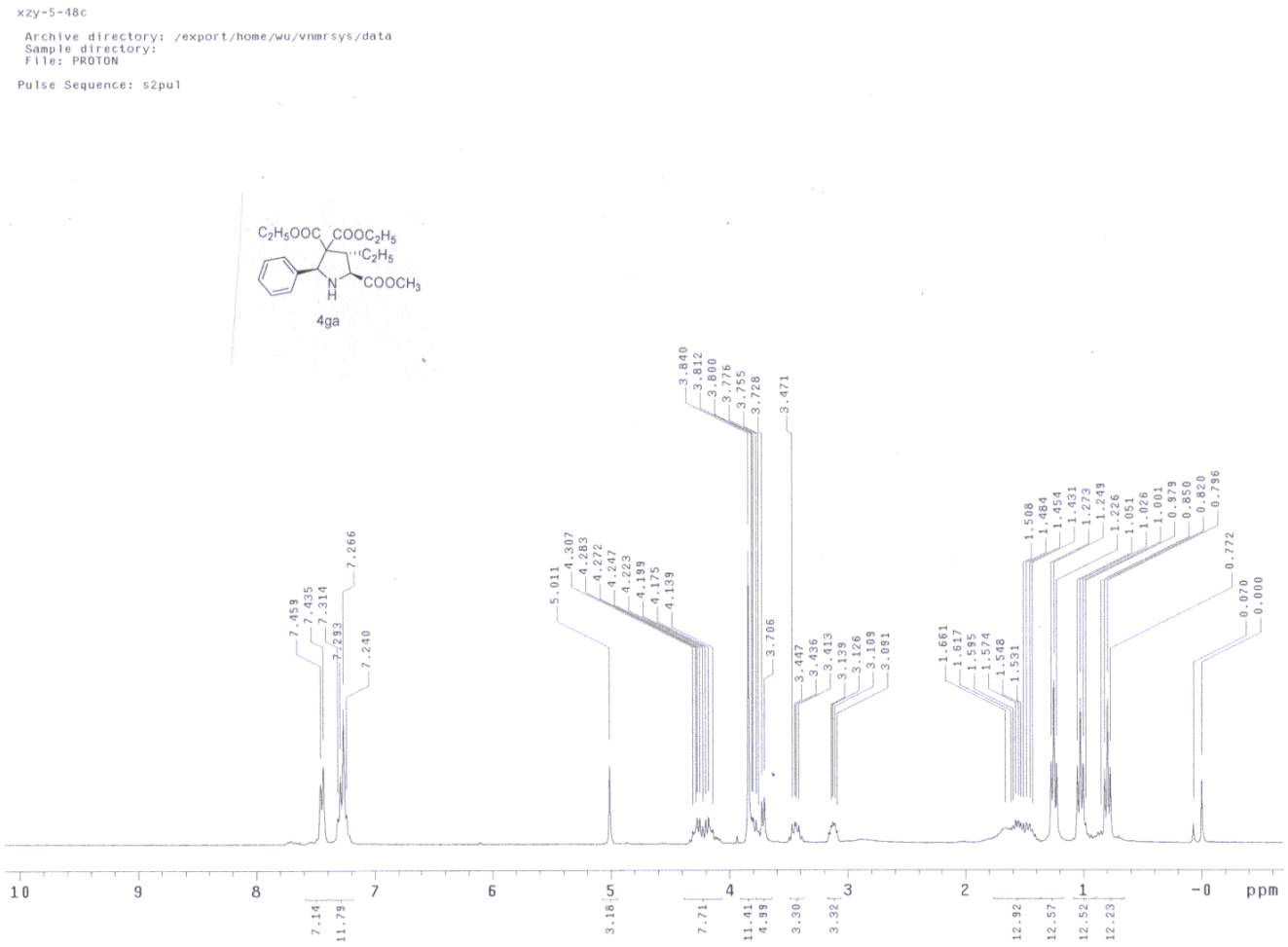
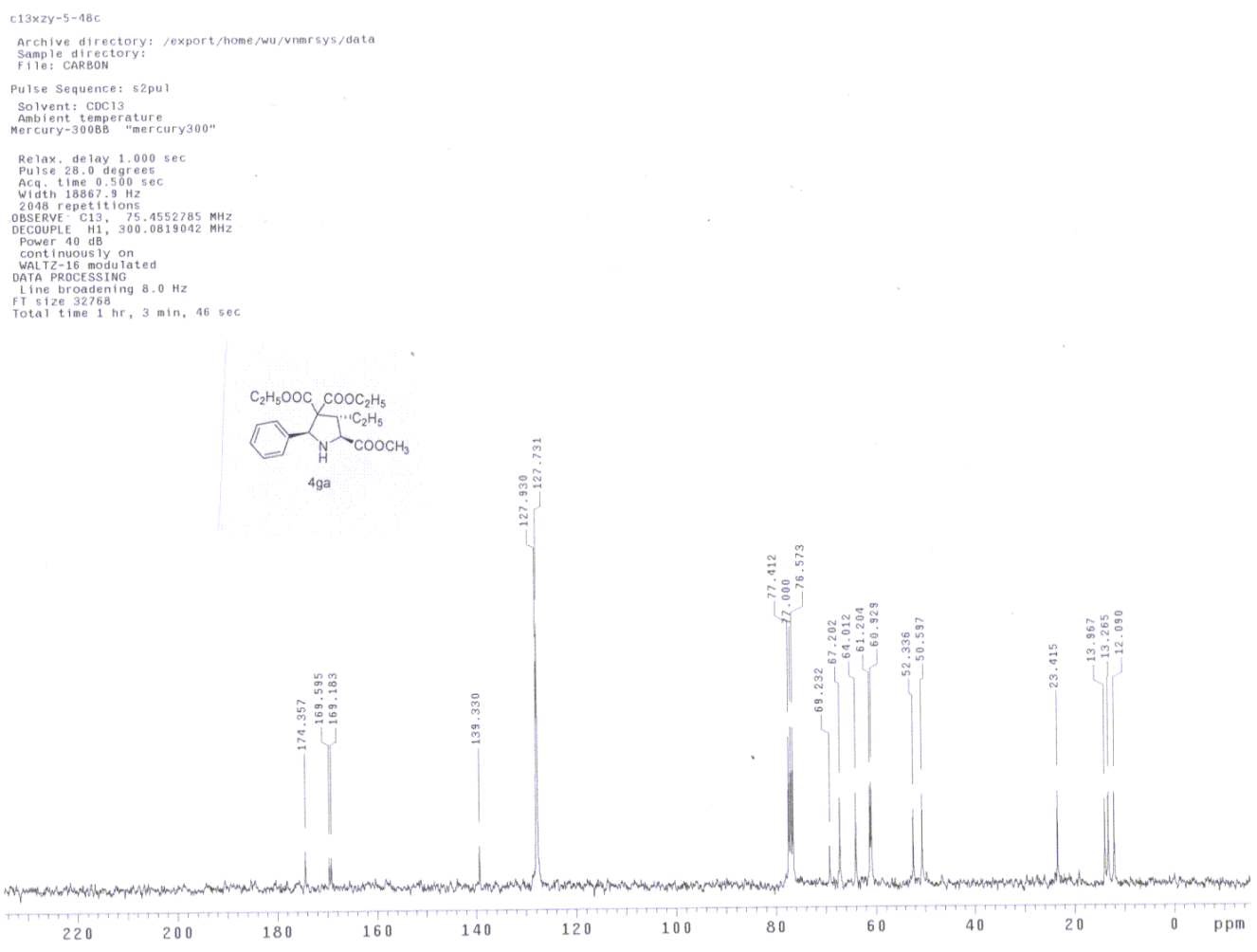
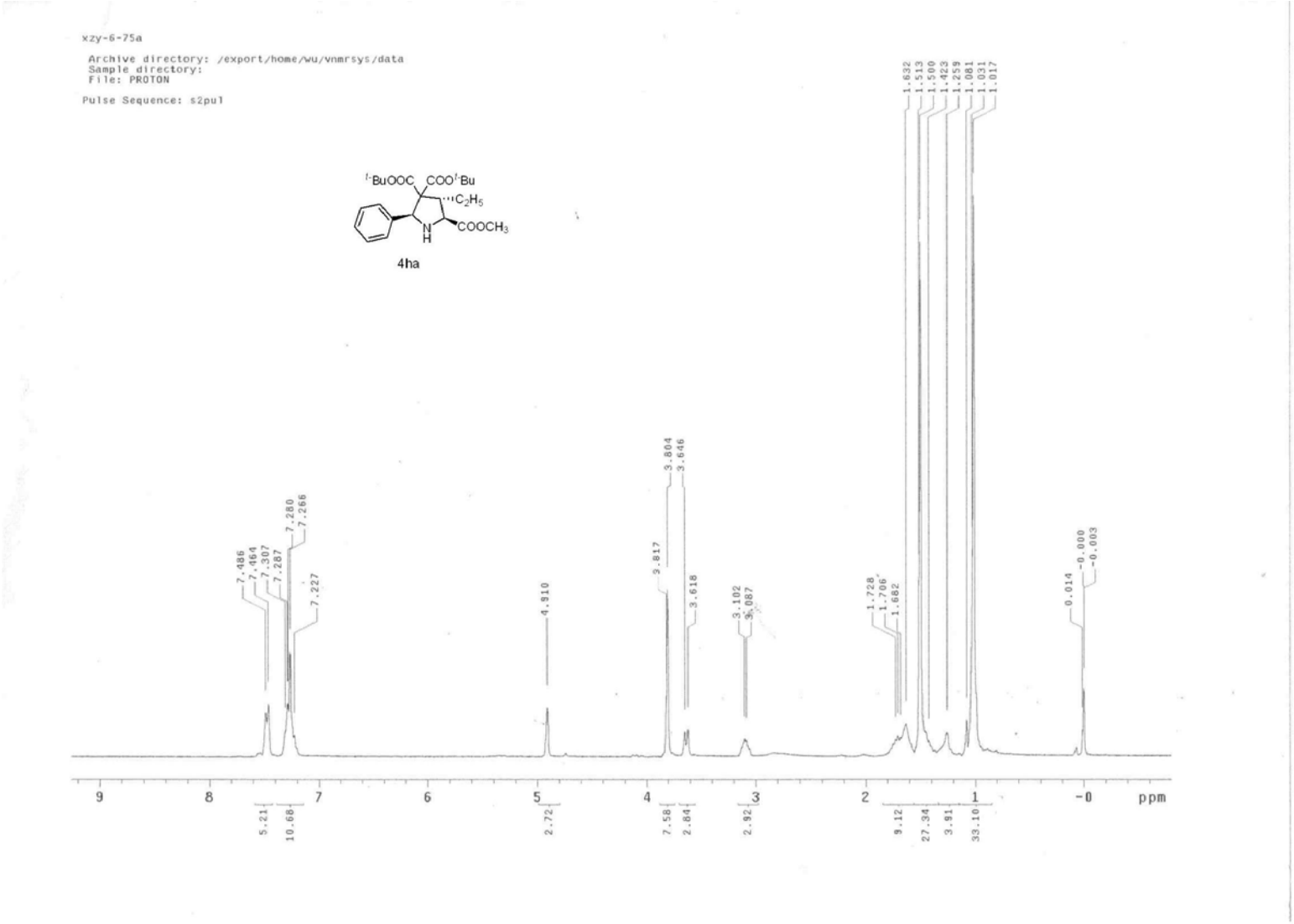
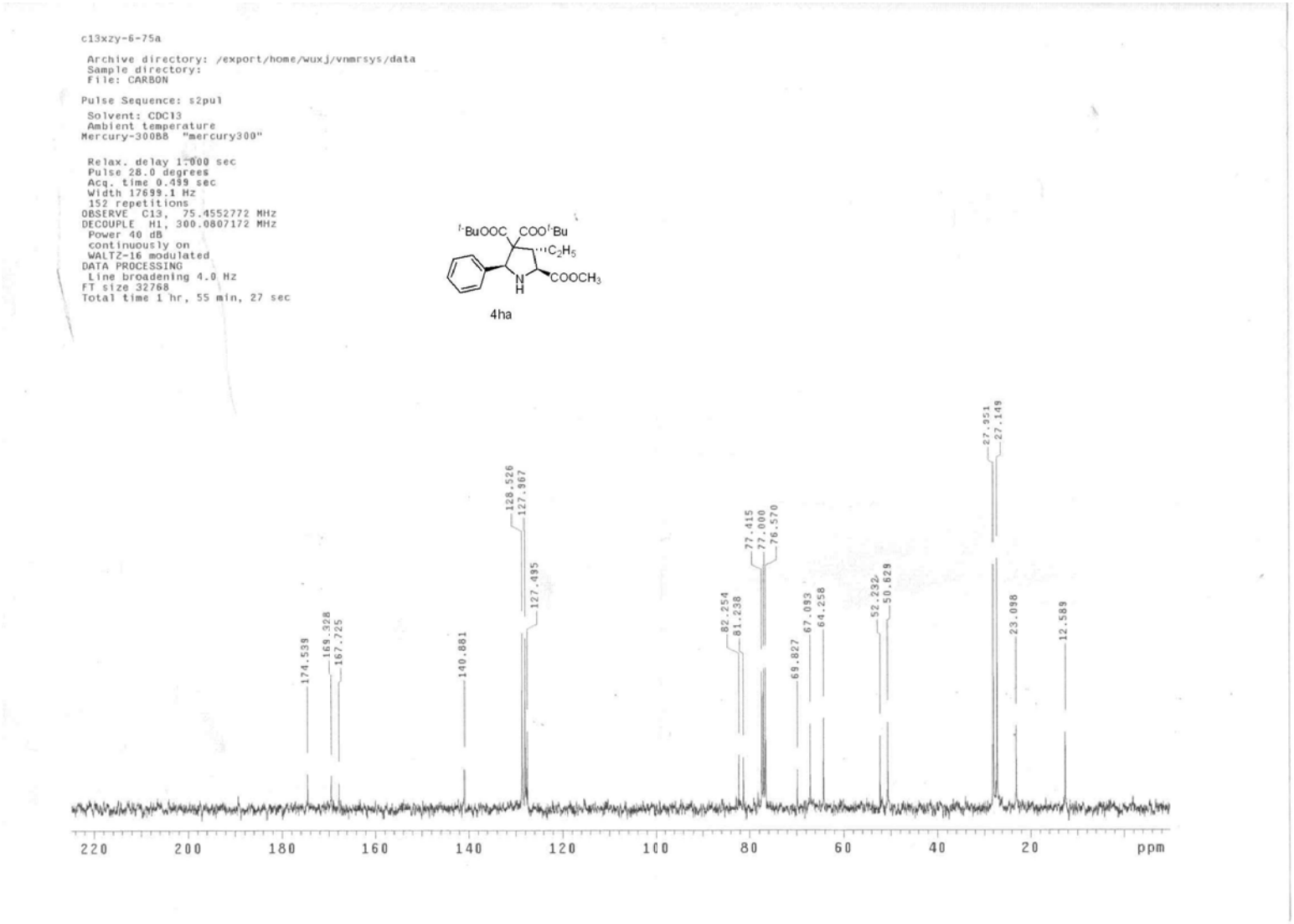
Sample directory:
File: PROTON
Pulse Sequence: s2pul
COO^{t}Bu
^{t}BuOOC
Br
H_3CO
N
H
COOCH$_3$
4fd
7.479
7.455
7.435
7.267
7.242
6.876
6.849
5.203
4.317
4.295
4.092
4.068
3.802
3.741
1.255
1.043
1.028
0.877
0.847
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
3.16
1.92
1.68
0.91
0.91
0.98
2.25
1.97
14.76

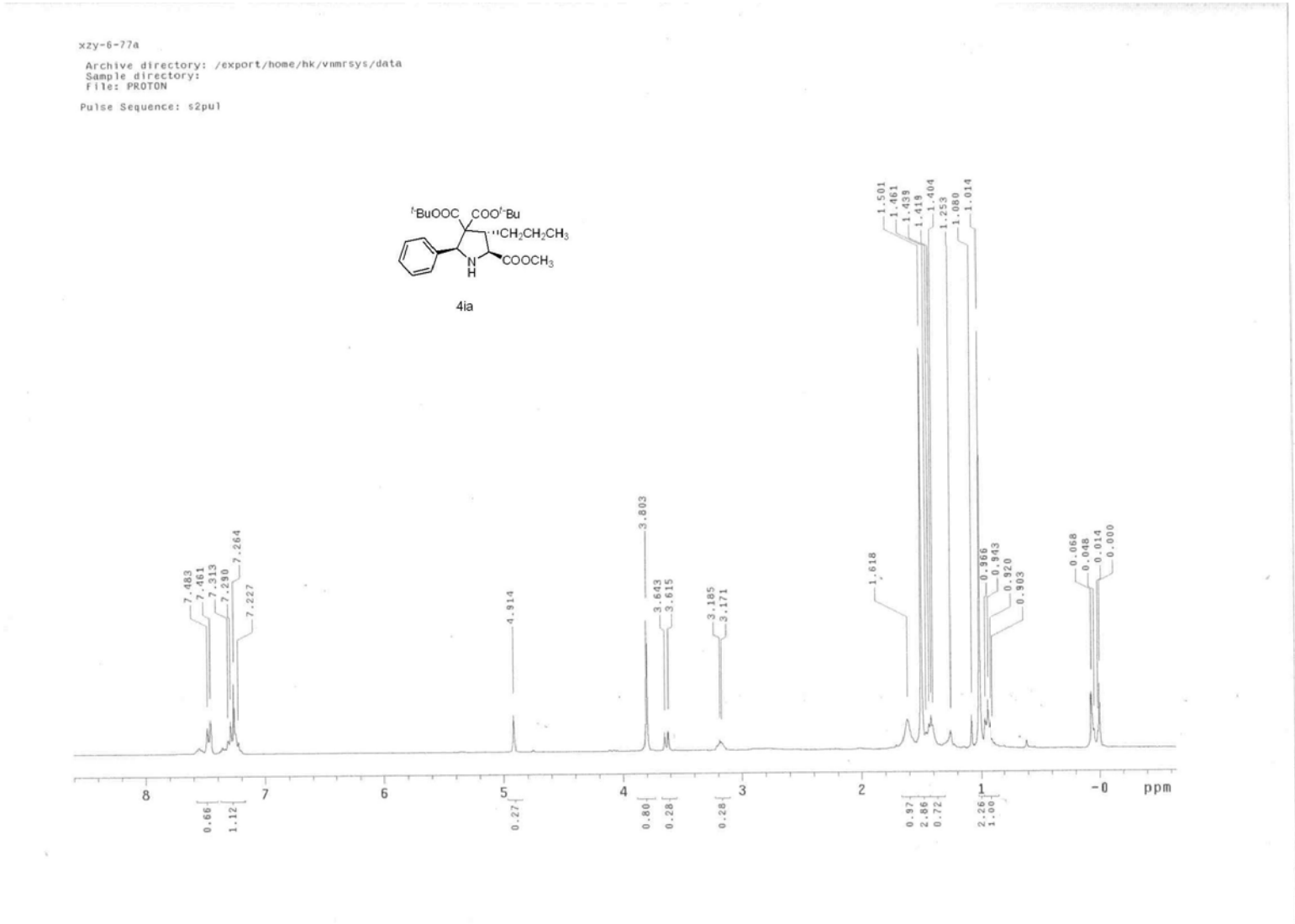

20090603-wangchj-xzy-6-21A-13C-CDCl3
COO^{t}Bu
^{t}BuOOC
Br
H_3CO
COOCH$_3$
N
H
4fd
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

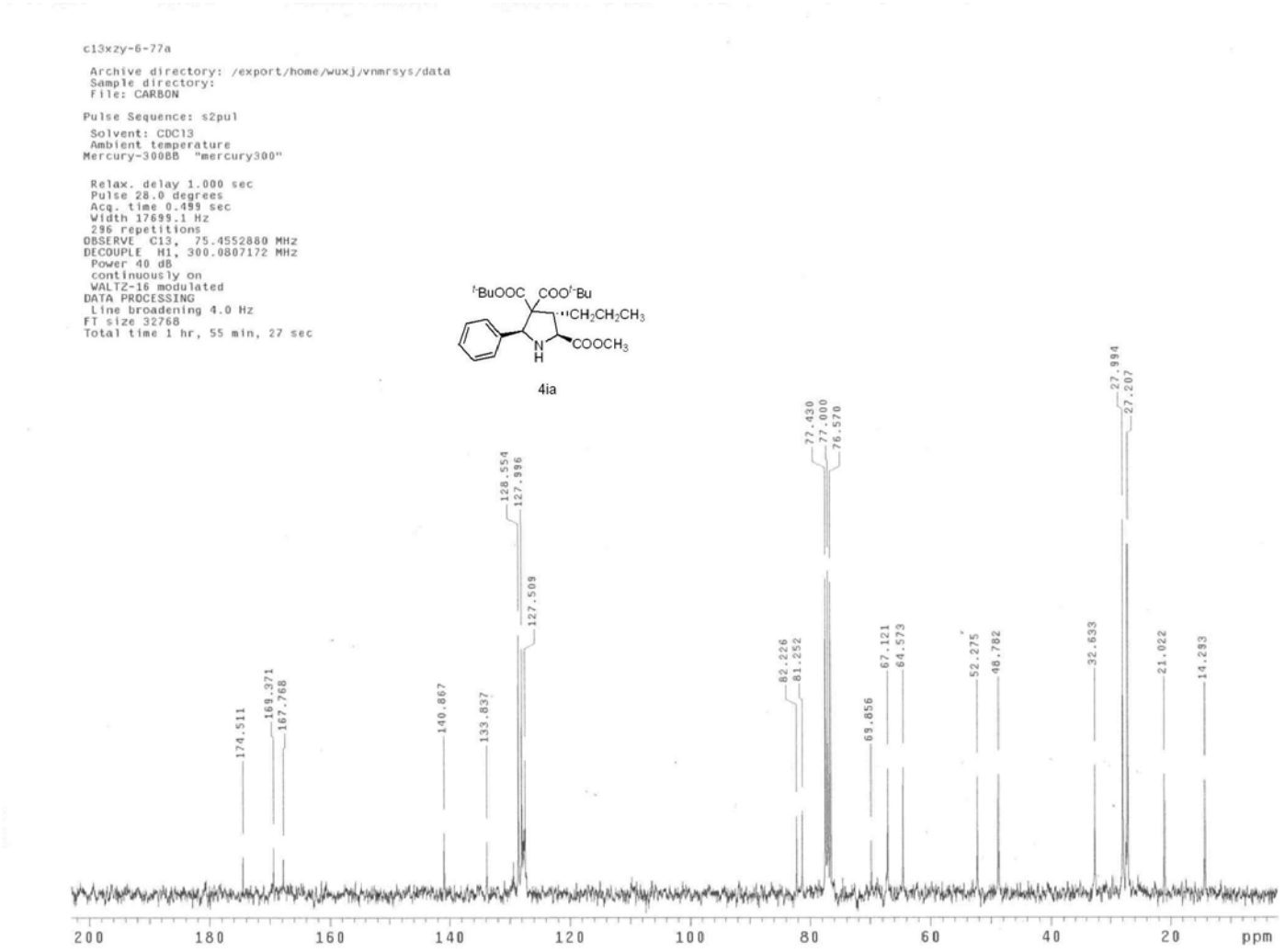


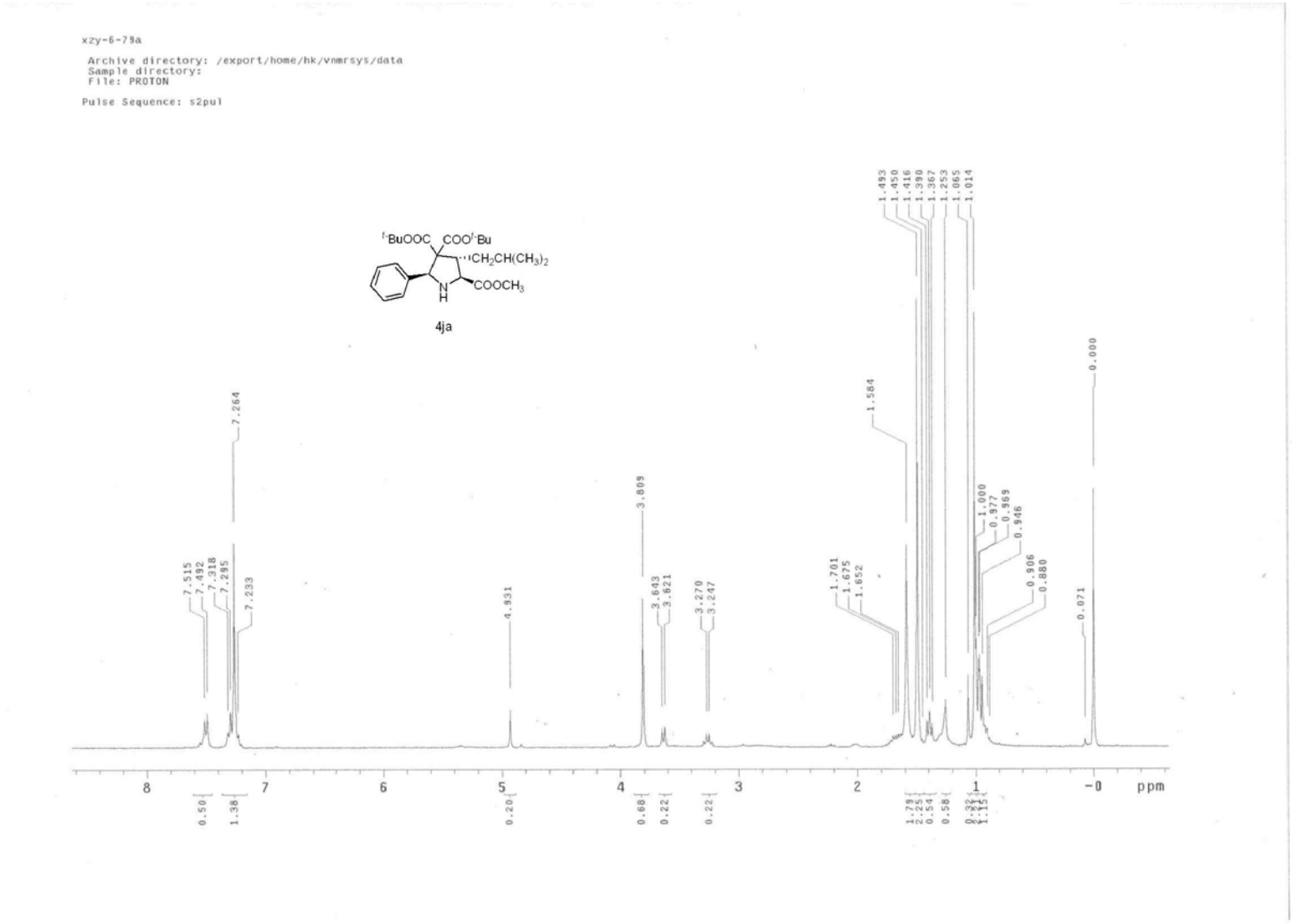

c13xzy-5-48c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 18867.9 Hz
2048 repetitions
OBSERVE C13, 75.4552785 MHz
DECOUPLE H1, 300.0819042 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 8.0 Hz
FT size 32768
Total time 1 hr, 3 min, 46 sec
4ga
174.357
169.595
169.183
139.330
127.930
127.731
77.412
77.000
76.573
69.232
67.202
64.012
61.204
60.929
52.336
50.597
23.415
13.967
13.265
12.090
220
200
180
160
140
120
100
80
60
40
20
0
ppm

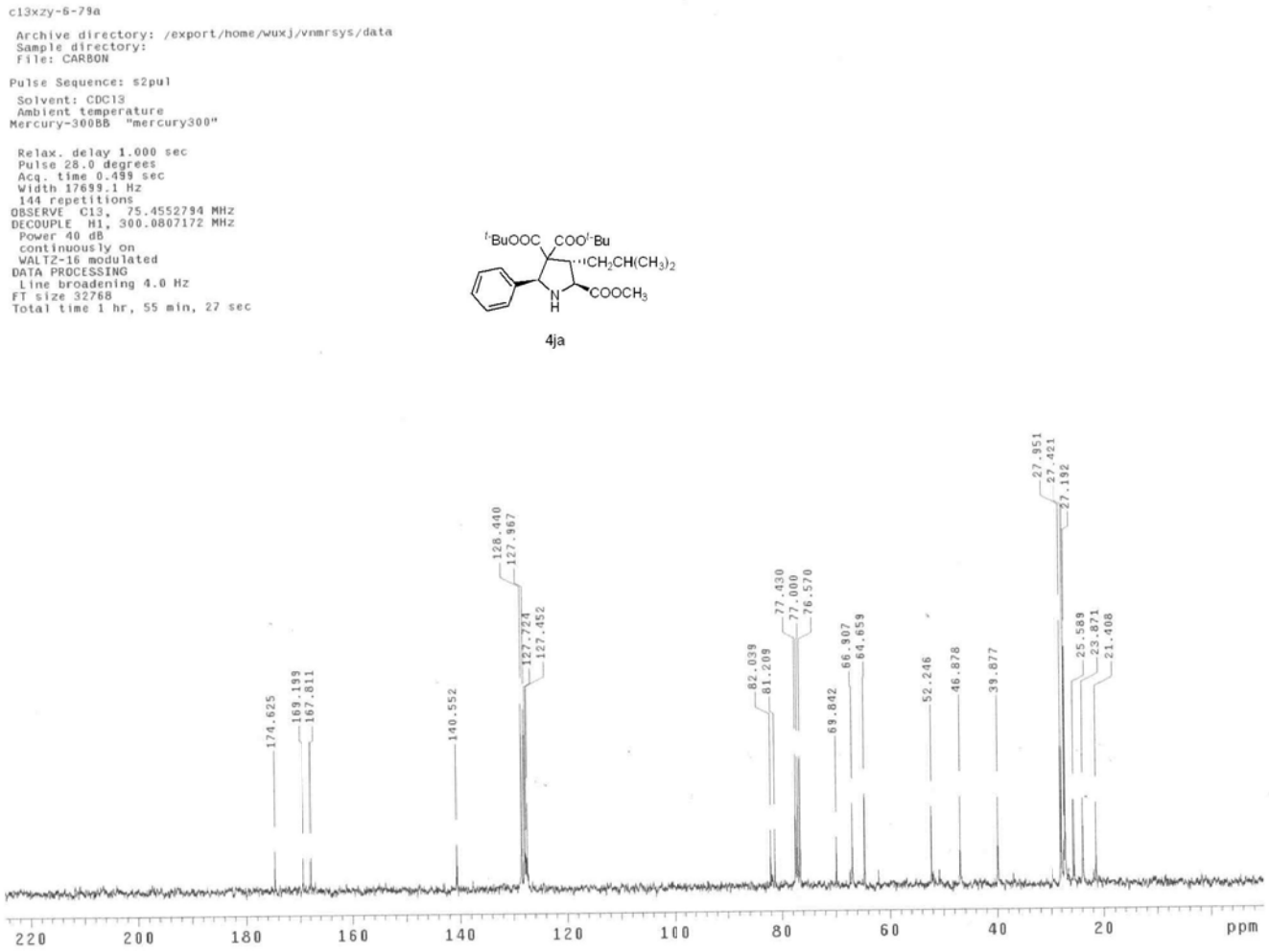

xzy-6-75a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
tBuOOC COOtBu
C2H5
COOCH3
N
H
4ha
1.632
1.513
1.500
1.423
1.259
1.081
1.031
1.017
3.804
3.646
7.486
7.464
7.307
7.287
7.280
7.266
7.227
4.910
3.817
3.618
3.102
3.087
1.728
1.706
1.682
0.014
-0.000
-0.003
9
8
7
6
5
4
3
2
1
-0
ppm
5.21
10.68
2.72
7.58
2.84
2.92
9.12
27.34
3.91
33.10

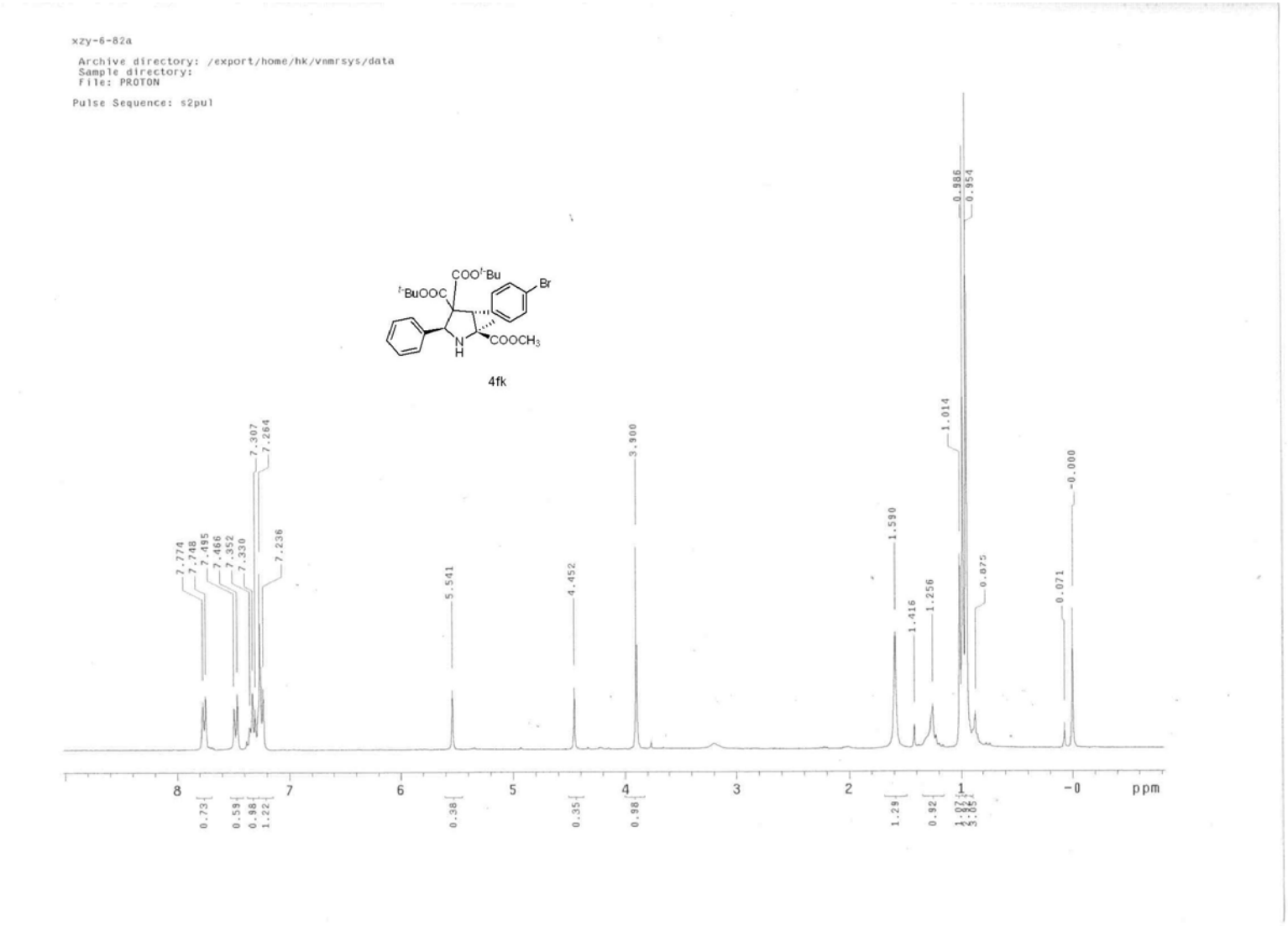

c13xzy-6-75a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
152 repetitions
OBSERVE C13, 75.4552772 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
tBuOOC
COOtBu
C2H5
COOCH3
N
H
4ha
174.539
169.328
167.725
140.881
128.526
127.967
127.495
82.254
81.238
77.415
77.000
76.570
69.827
67.093
64.258
52.232
50.629
27.951
27.149
23.098
12.589
220
200
180
160
140
120
100
80
60
40
20
ppm

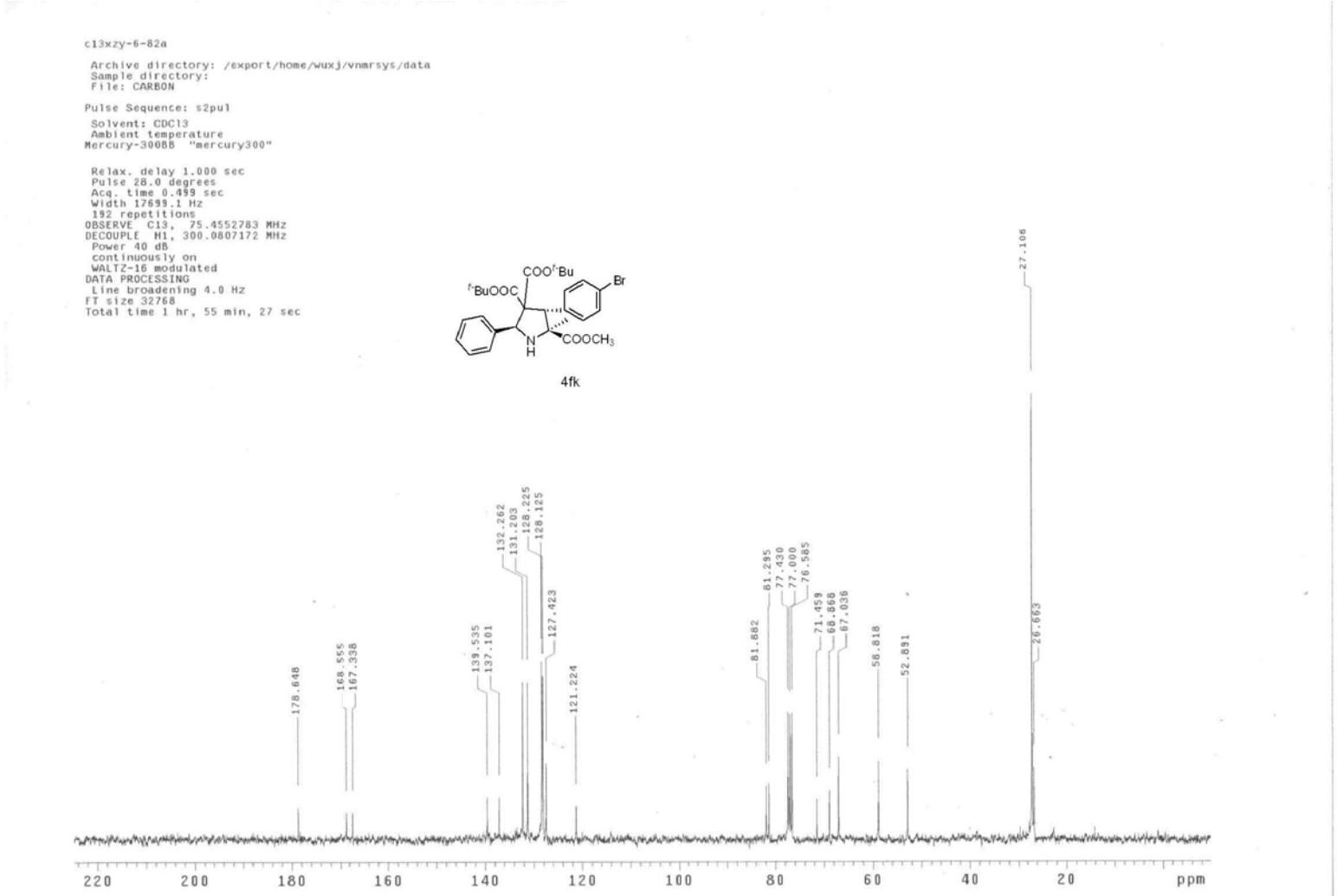

xzy-6-77a
Archive directory: /export/home/hk/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
tBuOOC COOtBu
CH2CH2CH3
COOCH3
N
H
4ia
7.483
7.461
7.313
7.290
7.264
7.227
4.914
3.803
3.643
3.615
3.185
3.171
1.618
1.501
1.461
1.439
1.419
1.404
1.253
1.080
1.014
0.966
0.943
0.920
0.903
0.068
0.048
0.014
0.000
8
7
6
5
4
3
2
1
-0
ppm
0.66
1.12
0.27
0.80
0.28
0.28
0.97
2.86
0.72
2.26
1.00

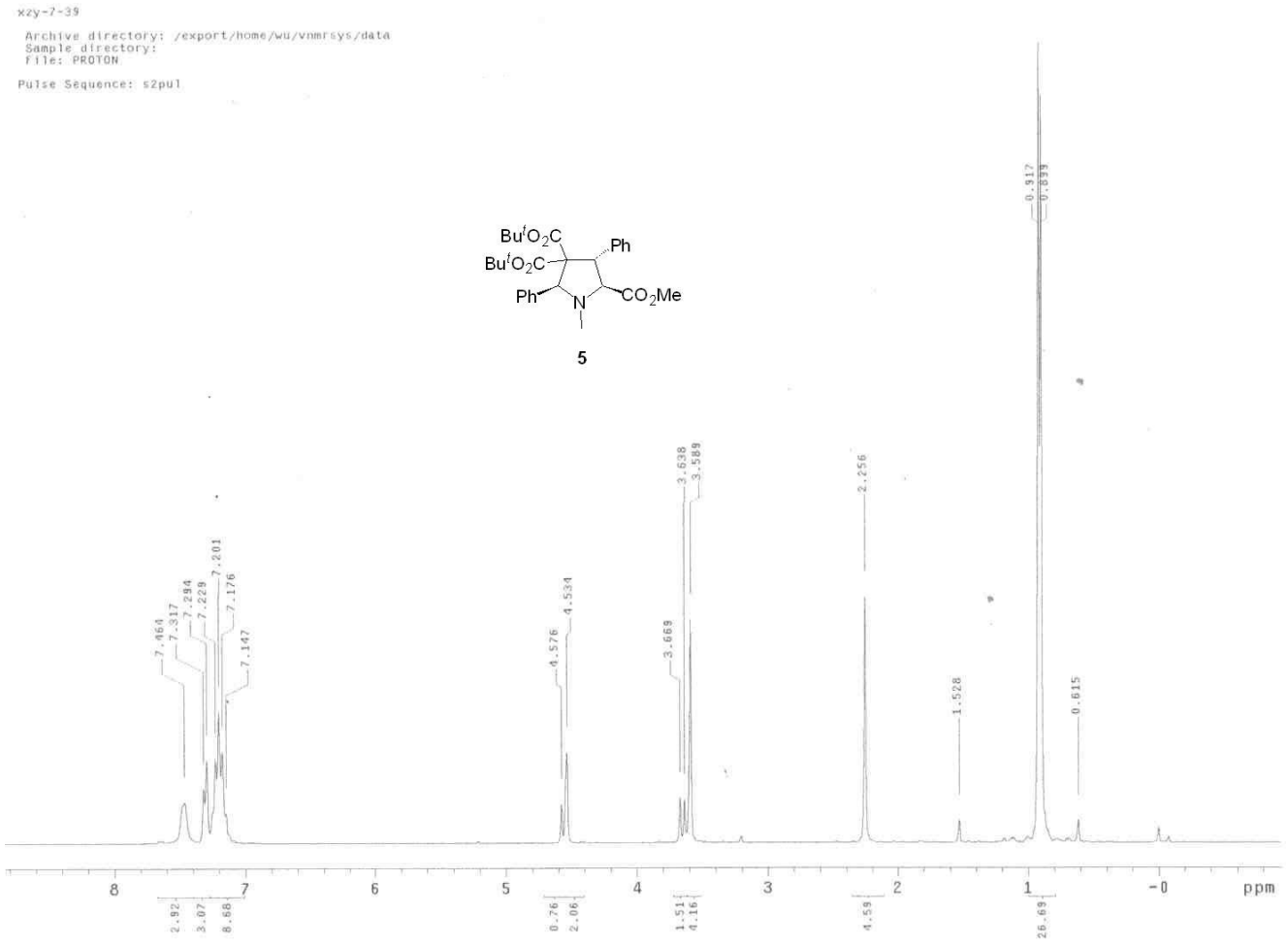

c13xzy-6-77a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
296 repetitions
OBSERVE C13, 75.4552880 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
tBuOOC COOtBu
CH2CH2CH3
COOCH3
N
H
4ia
174.511
169.371
167.768
140.867
133.837
128.554
127.996
127.509
82.226
81.252
77.430
77.000
76.570
69.856
67.121
64.573
52.275
48.782
32.633
27.994
27.207
21.022
14.293
200
180
160
140
120
100
80
60
40
20
ppm

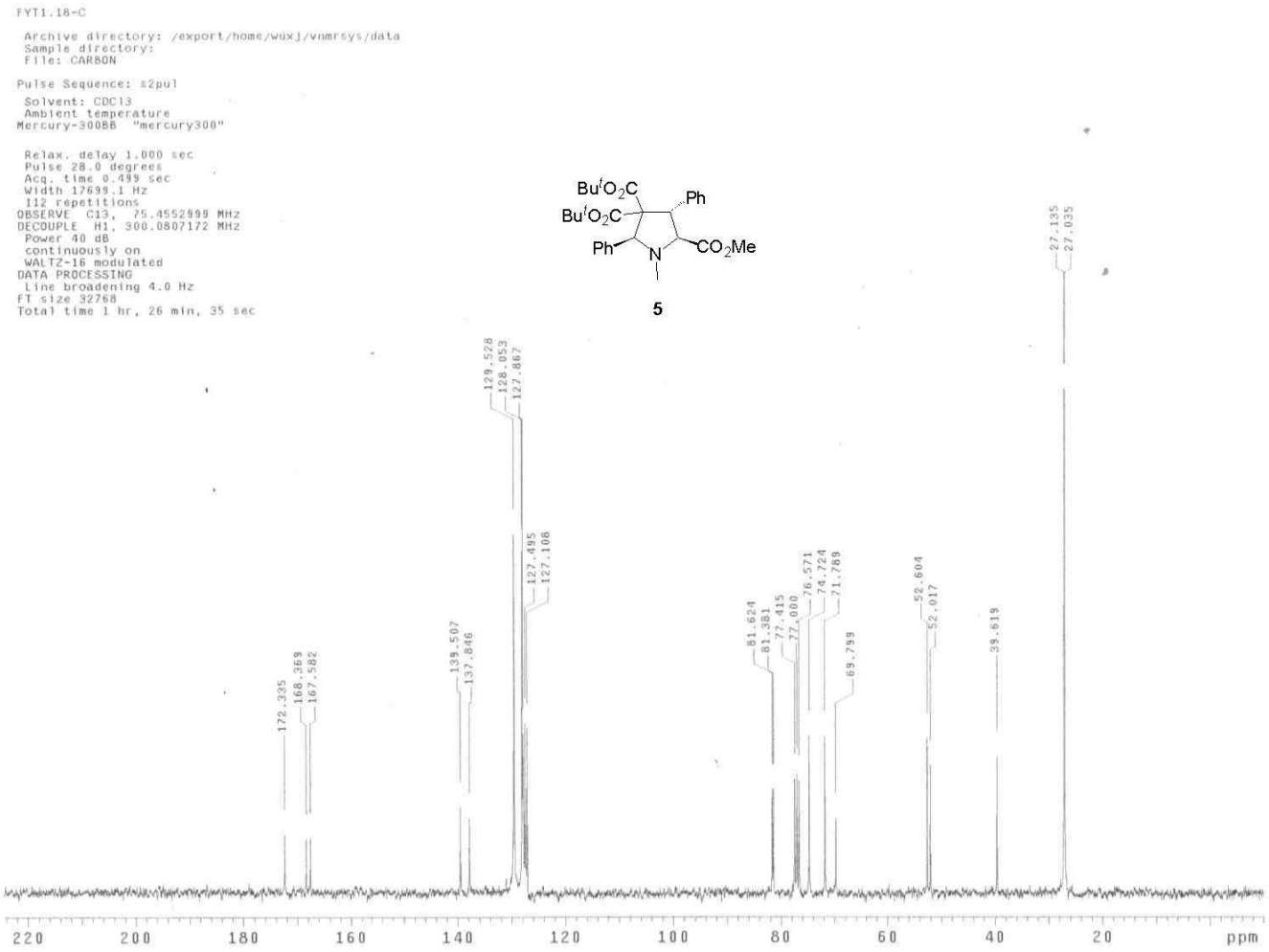

xzy-6-79a
Archive directory: /export/home/hk/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
tBuOOC COOtBu
CH2CH(CH3)2
COOCH3
N
H
4ja
7.515
7.492
7.318
7.295
7.264
7.233
4.931
3.809
3.643
3.621
3.270
3.247
1.701
1.675
1.652
1.584
1.493
1.450
1.416
1.390
1.367
1.253
1.065
1.014
1.000
0.977
0.969
0.946
0.906
0.880
0.071
0.000
8
7
6
5
4
3
2
1
-0
ppm
0.50
1.38
0.20
0.68
0.22
0.22
1.79
2.25
0.54
0.58

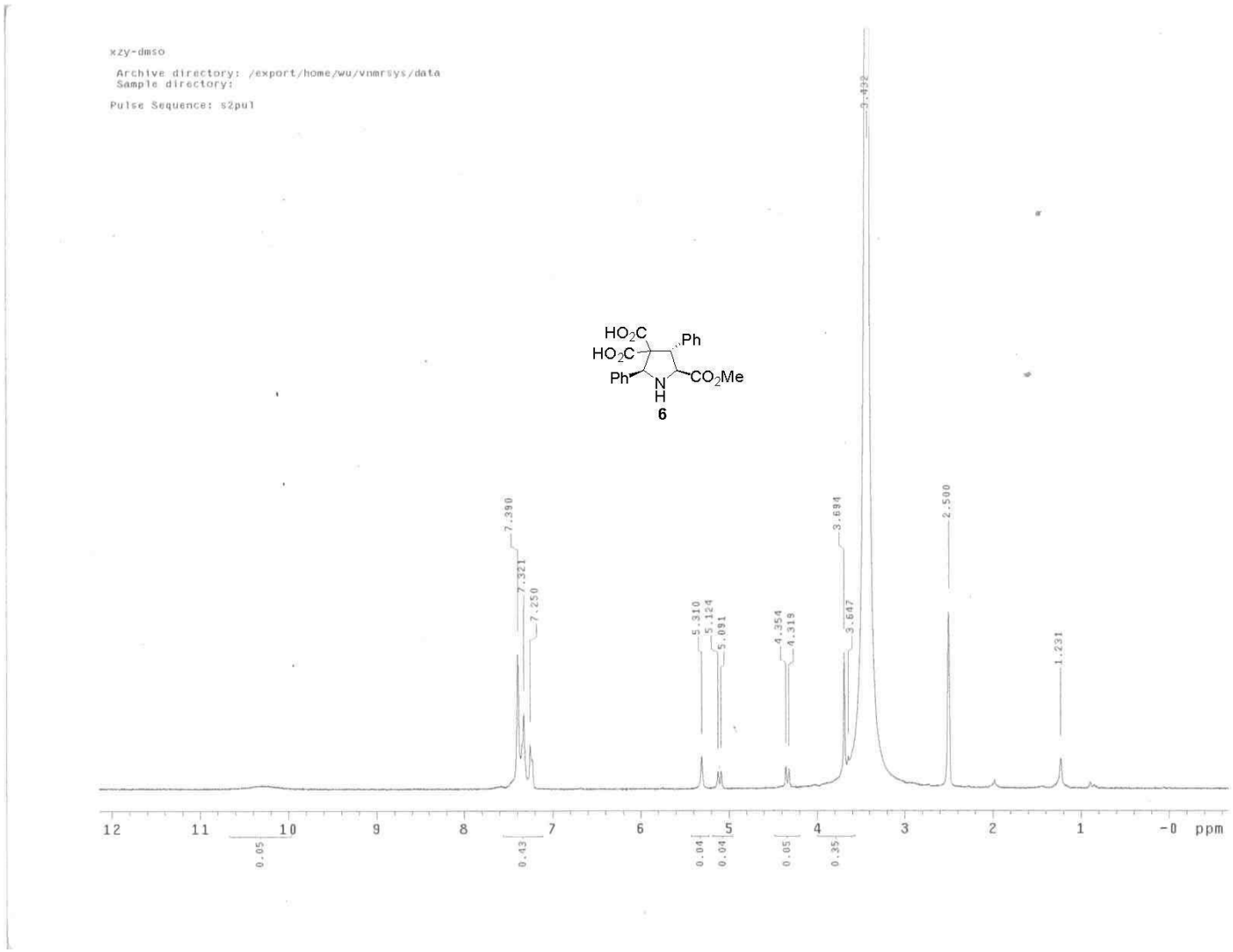

c13xzy-6-79a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
144 repetitions
OBSERVE C13, 75.4552794 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
tBuOOC COOtBu
CH2CH(CH3)2
COOCH3
N
H
4ja
174.625
169.199
167.811
140.552
128.440
127.967
127.724
127.452
82.039
81.209
77.430
77.000
76.570
69.842
66.907
64.659
52.246
46.878
39.877
27.951
27.421
27.192
25.589
23.871
21.408
220
200
180
160
140
120
100
80
60
40
20
ppm

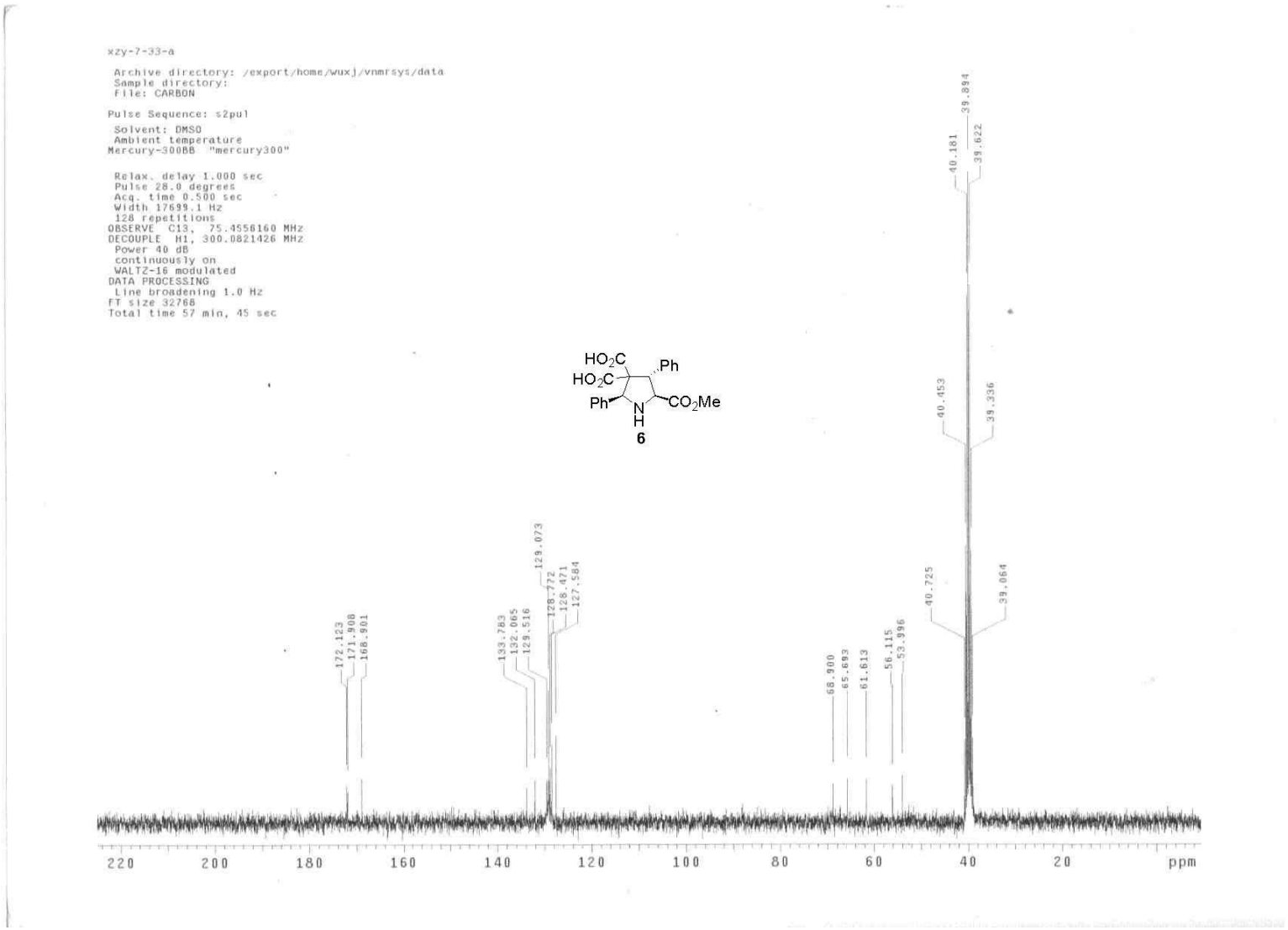

xzy-6-82a
Archive directory: /export/home/hk/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
COO^{t}Bu
^{t}BuOOC
Br
COOCH$_3$
N
H
4fk
7.774
7.748
7.495
7.466
7.352
7.330
7.307
7.264
7.236
5.541
4.452
3.900
1.590
1.416
1.256
1.014
0.886
0.854
0.875
0.071
-0.000
8
7
6
5
4
3
2
1
-0
ppm
0.73
0.59
0.88
1.22
0.38
0.35
0.88
1.29
0.92


c13xzy-6-82a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13, 75.4552783 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
COO^{t}Bu
^{t}BuOOC
Br
COOCH$_3$
N
H
4fk
178.648
168.555
167.338
139.535
137.101
132.262
131.203
128.225
128.125
127.423
121.224
81.882
81.295
77.430
77.000
76.585
71.459
68.868
67.036
58.818
52.891
27.108
26.663
220
200
180
160
140
120
100
80
60
40
20
ppm


xzy-7-39
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Bu^{t}O$_2$C
Bu^{t}O$_2$C
Ph
Ph
N
CO$_2$Me
5
7.464
7.317
7.284
7.229
7.201
7.176
7.147
4.576
4.534
3.669
3.638
3.589
2.256
1.528
0.917
0.899
0.615
8
7
6
5
4
3
2
1
-0
ppm
2.92
3.07
8.68
0.76
2.06
1.51
4.16
4.59
26.69


FYT1.18-C
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
112 repetitions
OBSERVE C13, 75.4552999 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 26 min, 35 sec
Bu^{t}O$_2$C
Bu^{t}O$_2$C
Ph
Ph
N
CO$_2$Me
5
172.335
168.369
167.582
139.507
137.846
129.528
128.053
127.867
127.495
127.108
81.624
81.381
77.415
77.000
76.571
74.724
71.789
69.793
52.604
52.017
39.619
27.135
27.035
220
200
180
160
140
120
100
80
60
40
20
ppm


xzy-dmso
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
HO2C
HO2C
Ph
Ph
N
H
CO2Me
6
7.390
7.321
7.250
5.310
5.124
5.091
4.354
4.319
3.694
3.647
3.432
2.500
1.231
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.05
0.43
0.04
0.04
0.05
0.35


xzy-7-33-a
Archive directory: /export/home/wuxj/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4556160 MHz
DECOUPLE H1, 300.0821426 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 57 min, 45 sec
HO2C
HO2C
Ph
Ph
N
H
CO2Me
6
172.123
171.908
168.901
133.783
132.065
129.516
129.073
128.772
128.471
127.584
68.900
65.693
61.613
56.115
53.996
40.725
40.453
40.181
39.894
39.622
39.336
39.064
220
200
180
160
140
120
100
80
60
40
20
ppm

Data File D:\LC\XZY\DATA\XZY-5-19\XZY-5-19 2009-01-07 10-14-54\SIG1000002.D
Sample Name: xzy-5-19a

```
    ====================================================================
    Acq. Operator   : xzy                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 92
    Injection Date  : 1/7/2009 10:27:11 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-19\XZY-5-19 2009-01-07 10-14-54\ASH-30-70-15ML-220NM.M
    Last changed    : 12/15/2008 10:32:26 AM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-19\XZY-5-19 2009-01-07 10-14-54\SIG1000002.D\DA.M (ASH-
                      30-70-15ML-220NM.M)
    Last changed    : 7/9/2009 3:40:59 PM by LTL
                      (modified after loading)
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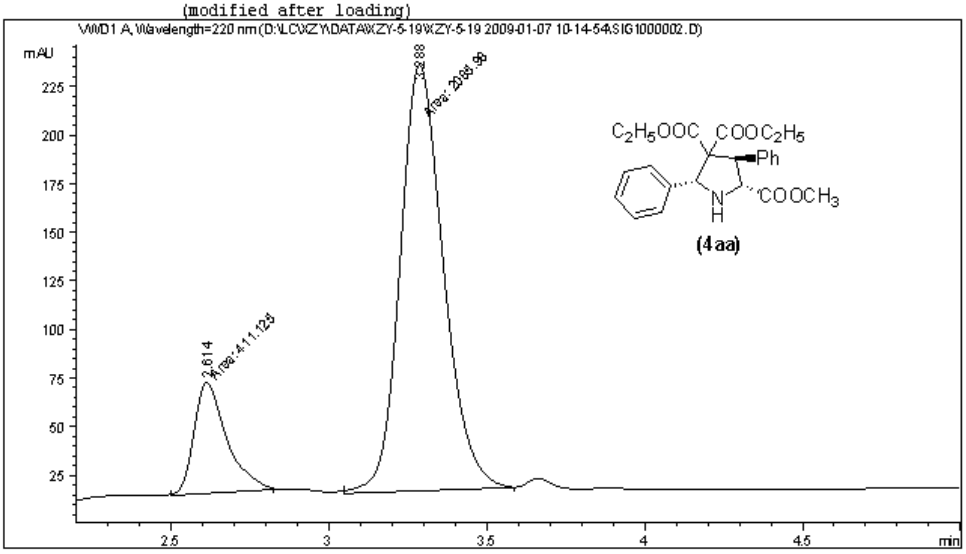



```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

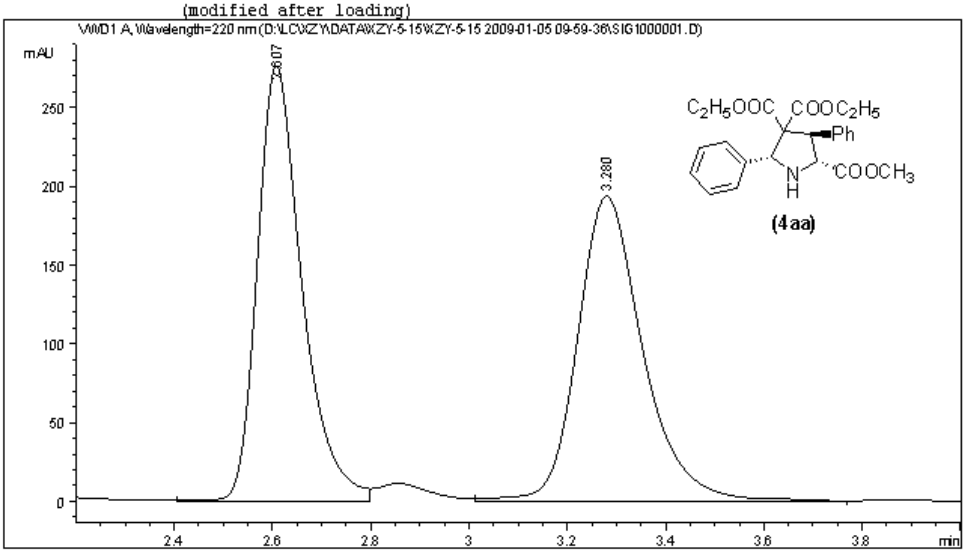
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   2.614 MM    0.1198  411.12451   57.18546  16.4640
       2   3.288 MM    0.1587 2085.98242  219.01387  83.5360

    Totals :                   2497.10693  276.19933


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-5-15\XZY-5-15 2009-01-05 09-59-36\SIG1000001.D
Sample Name: xzy-5-15a

```
    ====================================================================
    Acq. Operator   : xzy                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 86
    Injection Date  : 1/5/2009 10:01:00 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-15\XZY-5-15 2009-01-05 09-59-36\ASH-30-70-15ML-220NM.M
    Last changed    : 12/15/2008 10:32:26 AM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-15\XZY-5-15 2009-01-05 09-59-36\SIG1000001.D\DA.M (ASH-
                      30-70-15ML-220NM.M)
    Last changed    : 7/9/2009 3:35:51 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

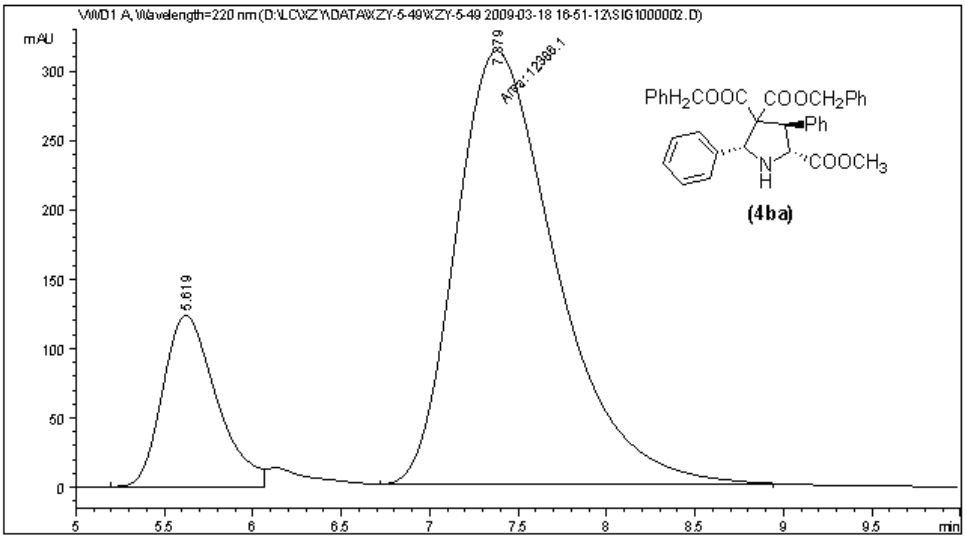
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   2.607 VV    0.0957 1755.90344  277.32416  49.1695
       2   3.280 VV    0.1410 1815.22021  194.27667  50.8305

    Totals :                   3571.12366  471.60083


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-5-49\XZY-5-49 2009-03-18 16-51-12\SIG1000002.D
Sample Name: xzy-5-49a

```
    ====================================================================
    Acq. Operator   : dongxiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                   Location : Vial 61
    Injection Date  : 3/18/2009 5:03:32 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-49\XZY-5-49 2009-03-18 16-51-12\ASH-30-70-10ML-220NM-
                      10MIN.M
    Last changed    : 1/6/2009 9:03:30 AM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-49\XZY-5-49 2009-03-18 16-51-12\SIG1000002.D\DA.M (ASH-
                      30-70-10ML-220NM-10MIN.M)
    Last changed    : 7/9/2009 3:47:00 PM by LTL
                      (modified after loading)
    Method Info     : ASH-30-70-1.0ML-220(20)
```




```
    ====================================================================
                              Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.619 BV    0.3245 2629.22412  124.00349  17.5103
       2   7.379 MM    0.6604 1.23861e4   312.58548  82.4897

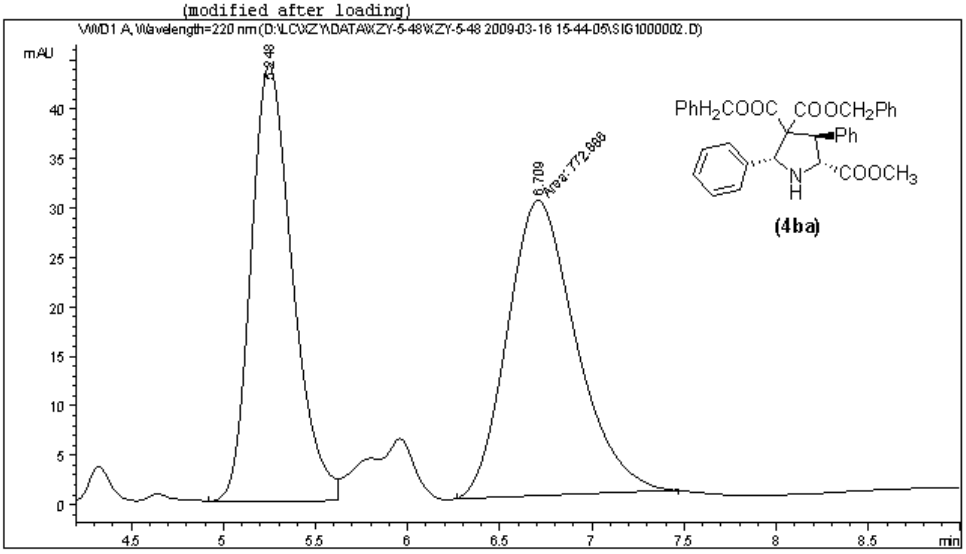
    Totals :                   1.50153e4   436.58897


    ====================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 3:47:03 PM LTL

Data File D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 15-44-05\SIG1000002.D
Sample Name: xzy-5-48a

```
    ==================================================================
    Acq. Operator   : liang qang                    Seq. Line :   2
    Acq. Instrument : Instrument 1                   Location : Vial 21
    Injection Date  : 3/16/2009 3:58:18 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 15-44-05\ASH-30-70-220NM-1ML.M
    Last changed    : 7/1/2008 8:17:03 PM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 15-44-05\SIG1000002.D\DA.M (ASH-
                      30-70-220NM-1ML.M)
    Last changed    : 7/9/2009 3:44:15 PM by LTL
                      (modified after loading)
```




```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

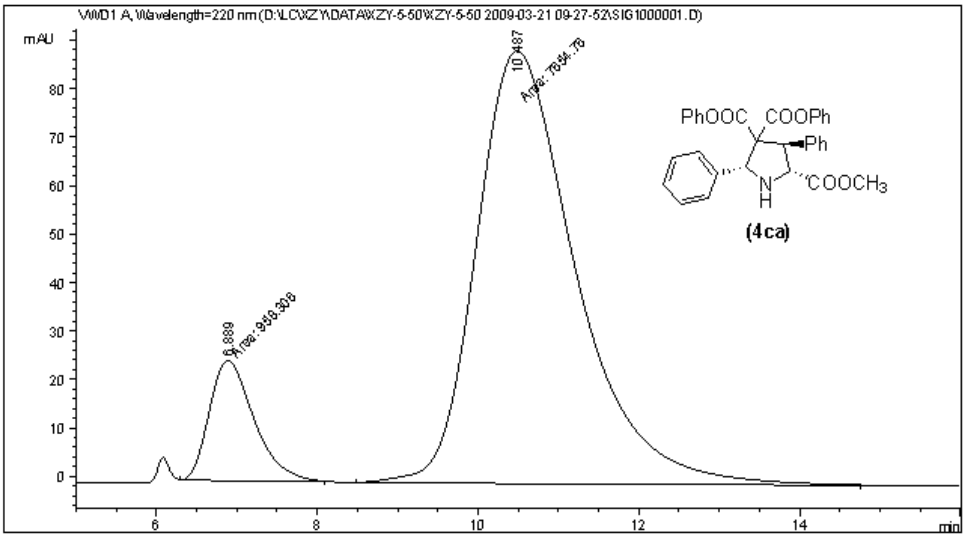
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.248 VV    0.2397  691.40741   44.11064  47.2178
       2   6.709 MM    0.4314  772.88568   29.86179  52.7822

    Totals :                  1464.29309   73.97242


    ==================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-5-50\XZY-5-50 2009-03-21 09-27-52\SIG1000001.D
Sample Name: xzy-5-50a

```
    ==================================================================
    Acq. Operator   : liang gang                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 84
    Injection Date  : 3/21/2009 9:29:04 AM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-50\XZY-5-50 2009-03-21 09-27-52\ASH-30-70-10ML-220NM-
                      20MIN.M
    Last changed    : 3/21/2009 9:28:25 AM by liang gang
                      (modified after loading)
    Analysis Method : D:\LC\XZY\DATA\XZY-5-50\XZY-5-50 2009-03-21 09-27-52\SIG1000001.D\DA.M (ASH-
                      30-70-10ML-220NM-20MIN.M)
    Last changed    : 7/9/2009 3:52:20 PM by LTL
                      (modified after loading)
    Method Info     : ASH-30-70-1.0ML-220(20)
```




```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   6.889 MM    0.6438  958.30609   24.80682  11.1262
       2  10.487 MM    1.4282 7654.76465   89.33189  88.8738

    Totals :                  8613.07074  114.13871


    ==================================================================
```

Instrument 1 7/9/2009 3:52:22 PM LTL

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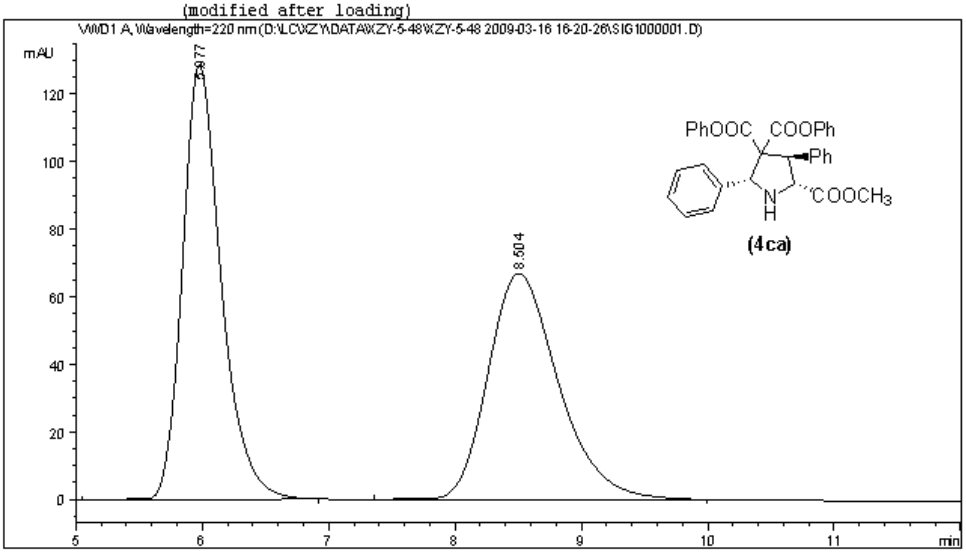
Data File D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-20-26\SIG1000001.D
Sample Name: xzy-5-48b

```
    ====================================================================
    Acq. Operator   : liang qang                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 22
    Injection Date  : 3/16/2009 4:22:24 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-20-26\ASH-30-70-220NM-1ML.M
    Last changed    : 7/1/2008 8:17:03 PM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-20-26\SIG1000001.D\DA.M (ASH-
                      30-70-220NM-1ML.M)
    Last changed    : 7/9/2009 3:49:45 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.977 VB    0.3232 2744.12744  128.60385  51.3001
       2   8.504 BB    0.5960 2605.04102   67.04376  48.6999

    Totals :                   5349.16846  195.64761


    ====================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 3:49:47 PM LTL

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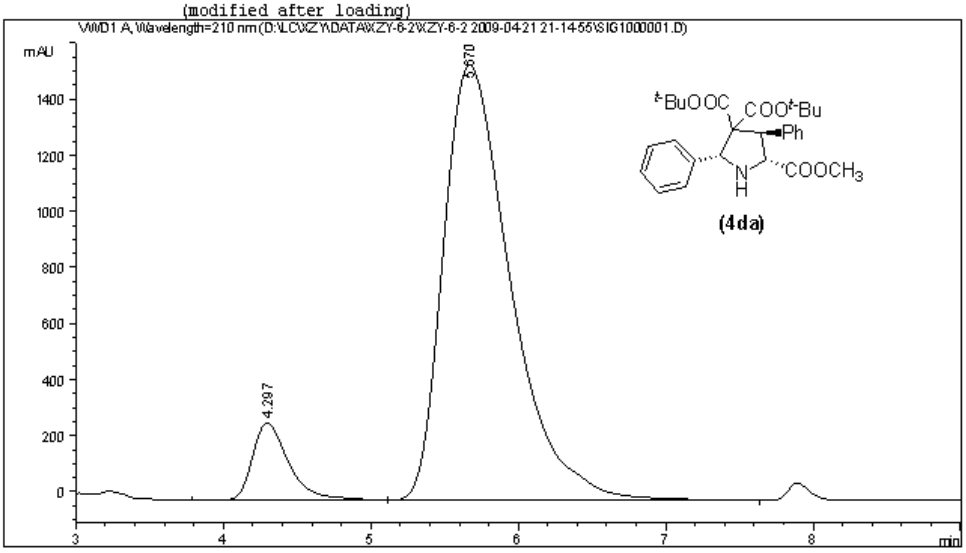
Data File D:\LC\XZY\DATA\XZY-6-2\XZY-6-2 2009-04-21 21-14-55\SIG1000001.D
Sample Name: xzy-6-2e

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 21
    Injection Date  : 4/21/2009 9:16:24 PM                 Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-2\XZY-6-2 2009-04-21 21-14-55\ASH-10-90-10ML-210NM-20MIN.
                      M
    Last changed    : 4/9/2009 5:26:07 PM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-6-2\XZY-6-2 2009-04-21 21-14-55\SIG1000001.D\DA.M (ASH-10-
                      90-10ML-210NM-20MIN.M)
    Last changed    : 7/9/2009 3:56:42 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

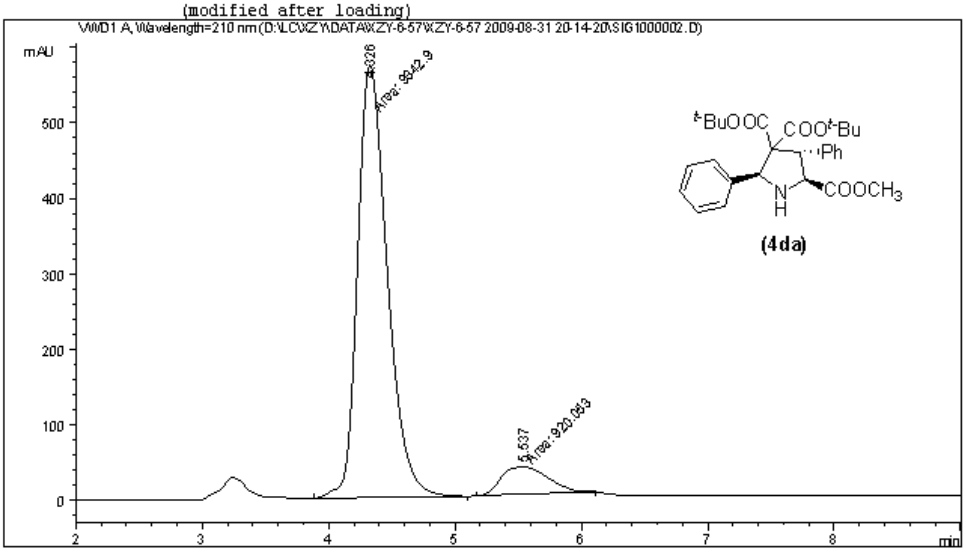
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       1   4.297 VV    0.2646 4878.53174  277.80310   9.0056
       2   5.670 VB    0.4849 4.92939e4  1554.80823  90.9944

    Totals :                   5.41724e4  1832.61133


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-57\XZY-6-57 2009-08-31 20-14-20\SIG1000002.D
Sample Name: xzy-6-57c

```
    ====================================================================
    Acq. Operator   : dxq                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 21
    Injection Date  : 8/31/2009 8:29:37 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-57\XZY-6-57 2009-08-31 20-14-20\ASH-10-90-10ML-210NM-
                      10MIN.M
    Last changed    : 3/4/2009 10:06:55 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-57\XZY-6-57 2009-08-31 20-14-20\SIG1000002.D\DA.M (ASH-
                      10-90-10ML-210NM-10MIN.M)
    Last changed    : 9/14/2009 4:18:24 PM by dxq
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

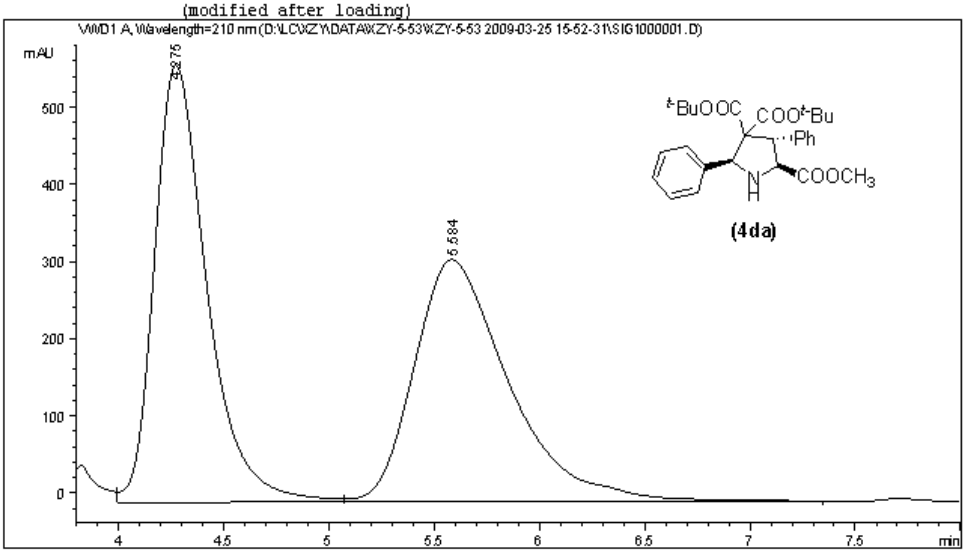
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       1   4.326 MM    0.2716 9342.89648  573.26685  91.0352
       2   5.537 MM    0.4247  920.05310   36.10443   8.9648

    Totals :                  1.02629e4   609.37128


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-5-53\XZY-5-53 2009-03-25 15-52-31\SIG1000001.D
Sample Name: XZY-5-53a

```
    ======================================================================
    Acq. Operator   : zhang zhihai                  Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 75
    Injection Date  : 3/25/2009 3:54:18 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-53\XZY-5-53 2009-03-25 15-52-31\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-5-53\XZY-5-53 2009-03-25 15-52-31\SIG1000001.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 3:54:40 PM by LTL
                      (modified after loading)
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.275 VV    0.2634 9820.85840  566.55017  50.1049
       2   5.584 VB    0.4729 9779.75195  313.73007  49.8951

    Totals :                  1.96006e4   880.28024

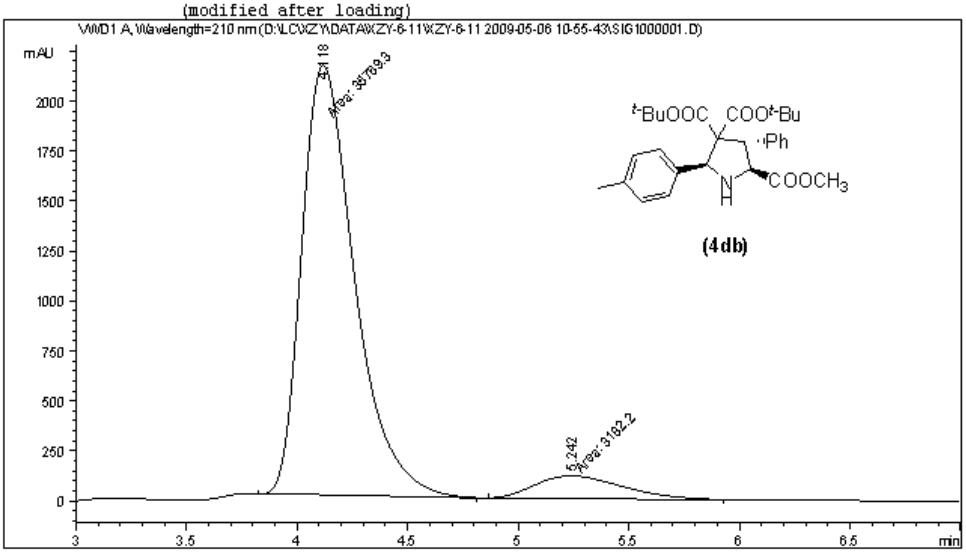

    ======================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 3:54:42 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-55-43\SIG1000001.D
Sample Name: xzy-6-11c

```
    ==================================================================
    Acq. Operator   : xzy                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 62
    Injection Date  : 5/6/2009 10:57:20 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-55-43\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-55-43\SIG1000001.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 4:02:08 PM by LTL
                      (modified after loading)
```



(4db)

```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.118 MM    0.2773 3.57693e4  2149.52466  91.8303
       2   5.242 MM    0.4625 3182.20483  114.66626   8.1697

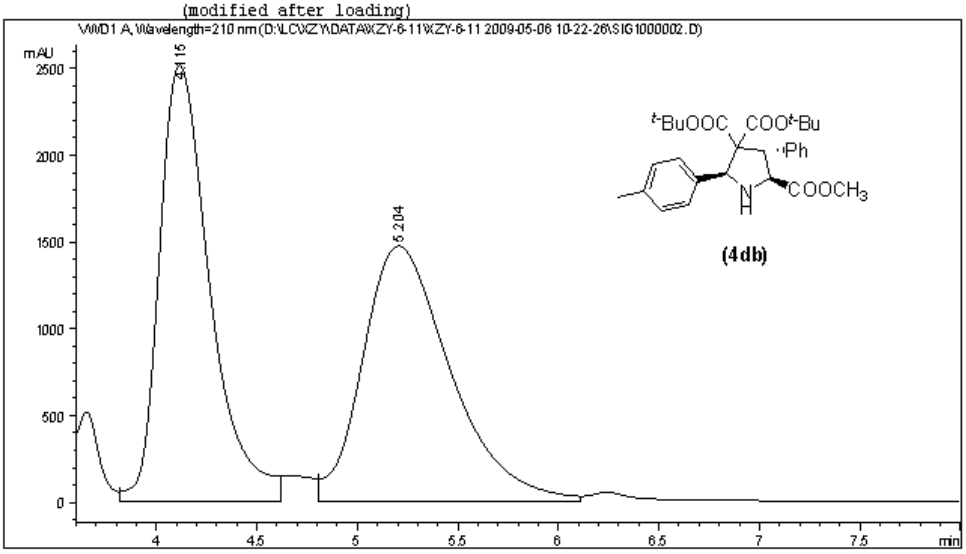
    Totals :                  3.89515e4  2264.19092


    ==================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 4:02:11 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-22-26\SIG1000002.D
Sample Name: xzy-6-11a

```
    ================================================================
    Acq. Operator   : xzy                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 61
    Injection Date  : 5/6/2009 10:34:56 AM               Inj :   1
                                                  Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-22-26\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-11\XZY-6-11 2009-05-06 10-22-26\SIG1000002.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 3:59:04 PM by LTL
                      (modified after loading)
```



(4db)

```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.115 VV    0.2663 4.38980e4  2514.40820  49.6444
       2   5.204 VV    0.4626 4.45269e4  1470.00928  50.3556

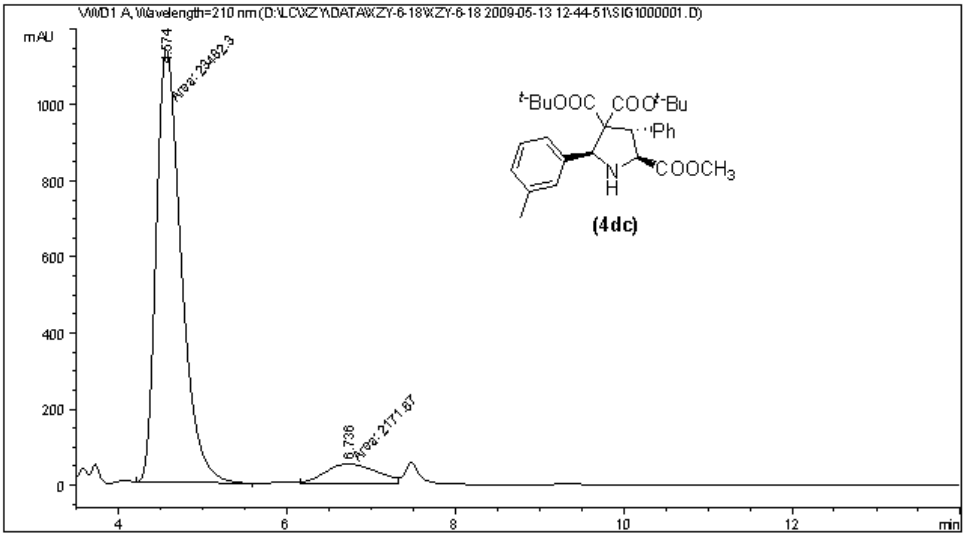
    Totals :                   8.84249e4  3984.41748


    ================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 3:59:09 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-44-51\SIG1000001.D
Sample Name: xzy-6-18e

```
    ======================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 14
    Injection Date  : 5/13/2009 12:46:09 PM              Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-44-51\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-44-51\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:10:44 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.574 MM    0.3437 2.34823e4  1138.55774  91.5348
       2   6.736 MM    0.7093 2171.66675   51.03012   8.4652

    Totals :                  2.56539e4  1189.58786

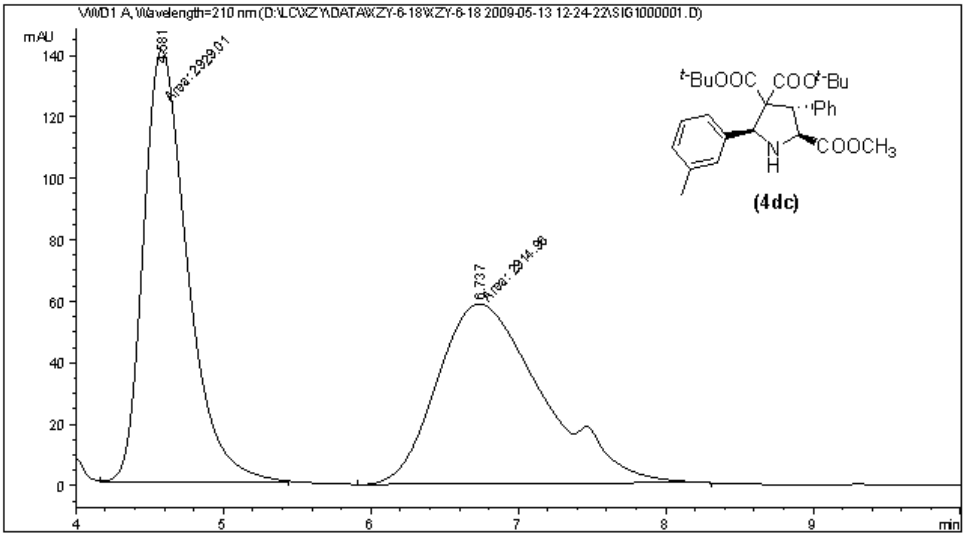
    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:10:46 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-24-22\SIG1000001.D
Sample Name: xzy-6-18b

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 13
    Injection Date  : 5/13/2009 12:25:40 PM              Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-24-22\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-24-22\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:04:17 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.581 MM    0.3466 2929.00806  140.83606  50.1202
       2   6.737 MM    0.8304 2914.96094   58.50456  49.8798

    Totals :                   5843.96899  199.34062

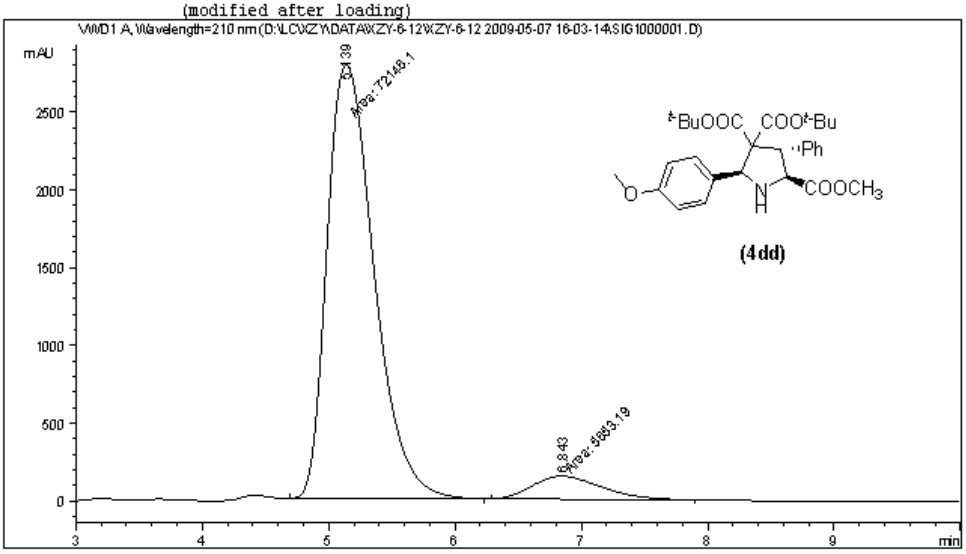
    ====================================================================
                               *** End of Report ***
```

Instrument 1 7/9/2009 4:04:20 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 16-03-14\SIG1000001.D
Sample Name: xzy-6-12b

```
    ====================================================================
    Acq. Operator   : ltl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 32
    Injection Date  : 5/7/2009 4:04:33 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 16-03-14\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 16-03-14\SIG1000001.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 4:16:28 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.139 MM    0.4318 7.21461e4  2784.41455  92.7336
       2   6.843 MM    0.6354 5653.19043  148.28455   7.2664

    Totals :                  7.77993e4  2932.69910

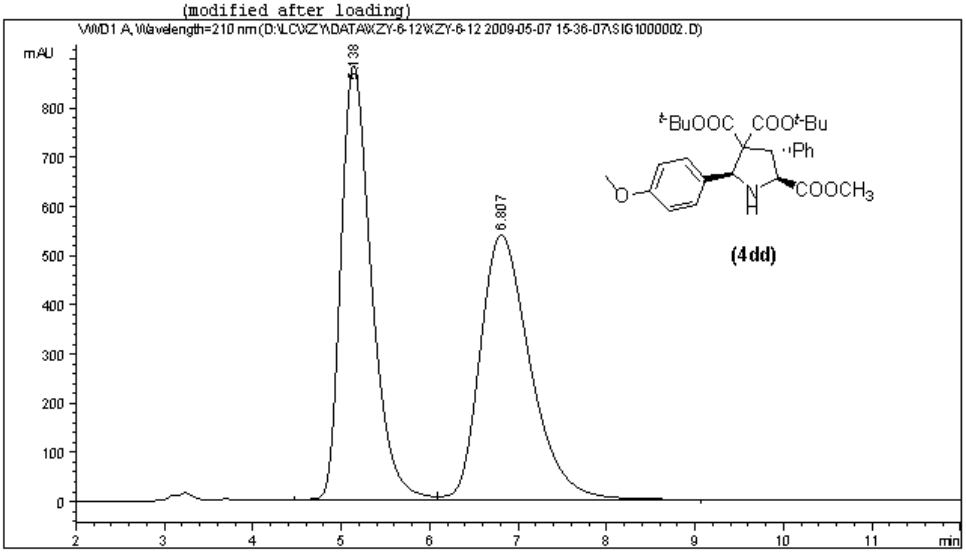

    ====================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 4:16:31 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 15-36-07\SIG1000002.D
Sample Name: xzy-6-12a

```
    ====================================================================
    Acq. Operator   : ltl                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 31
    Injection Date  : 5/7/2009 3:48:29 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 15-36-07\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 15-36-07\SIG1000002.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 4:13:05 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

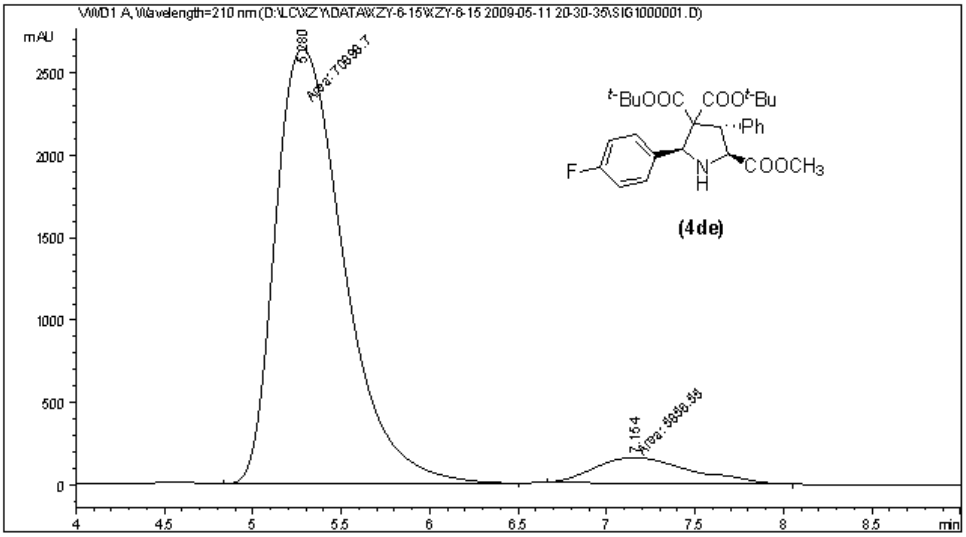
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.138 VV    0.3672 2.12233e4   884.86169  50.0483
       2   6.807 VB    0.6048 2.11823e4   539.87646  49.9517

    Totals :                   4.24057e4  1424.73816


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-30-35\SIG1000001.D
Sample Name: xzy-6-15f

```
    ======================================================================
    Acq. Operator   : dongxiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 86
    Injection Date  : 5/11/2009 8:31:52 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-30-35\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-30-35\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:21:03 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.280 MM    0.4486 7.08987e4  2634.36060  92.3698
       2   7.154 MM    0.6284 5856.55273  155.32179   7.6302

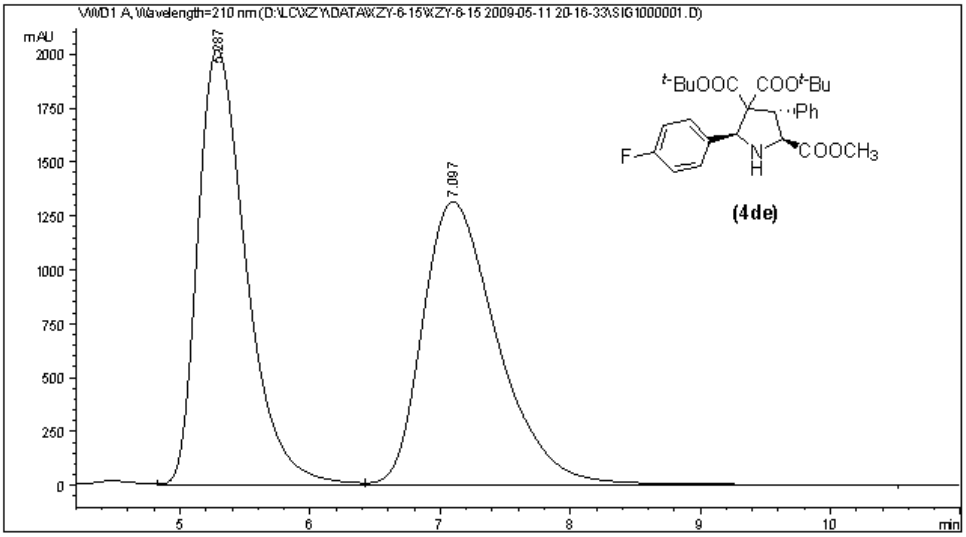
    Totals :                  7.67553e4  2789.68239

    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:21:06 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-16-33\SIG1000001.D
Sample Name: xzy-6-15c

```
    ======================================================================
    Acq. Operator   : dongxiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 85
    Injection Date  : 5/11/2009 8:18:11 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-16-33\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 20-16-33\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:18:15 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.287 VV    0.3981 5.23163e4  2021.11304  49.6239
       2   7.097 VB    0.6214 5.31094e4  1314.42187  50.3761

    Totals :                   1.05426e5  3335.53491

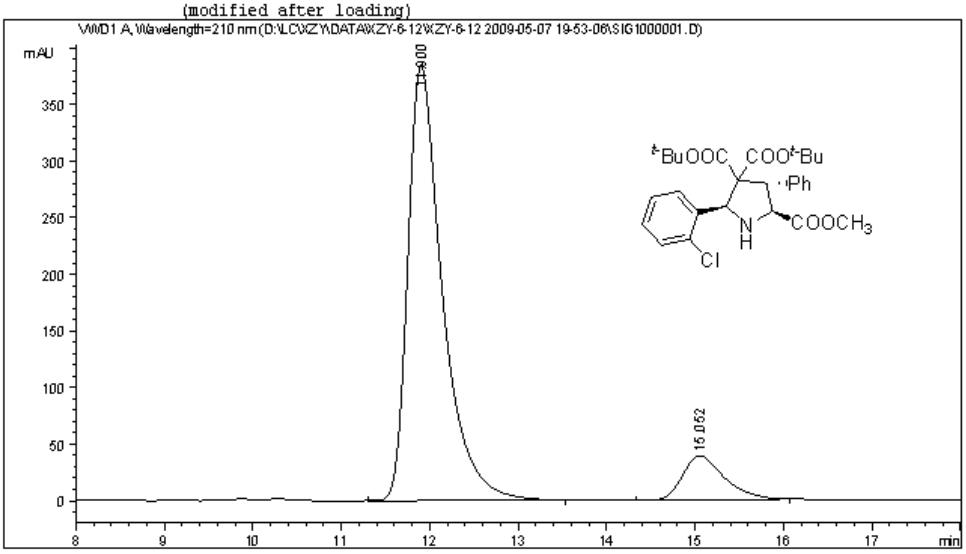
    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:18:17 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-53-06\SIG1000001.D
Sample Name: xzy-6-12d

```
    ================================================================
    Acq. Operator   : ltl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 34
    Injection Date  : 5/7/2009 7:54:31 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-53-06\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-53-06\SIG1000001.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:24:42 PM by LTL
                      (modified after loading)
```




```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

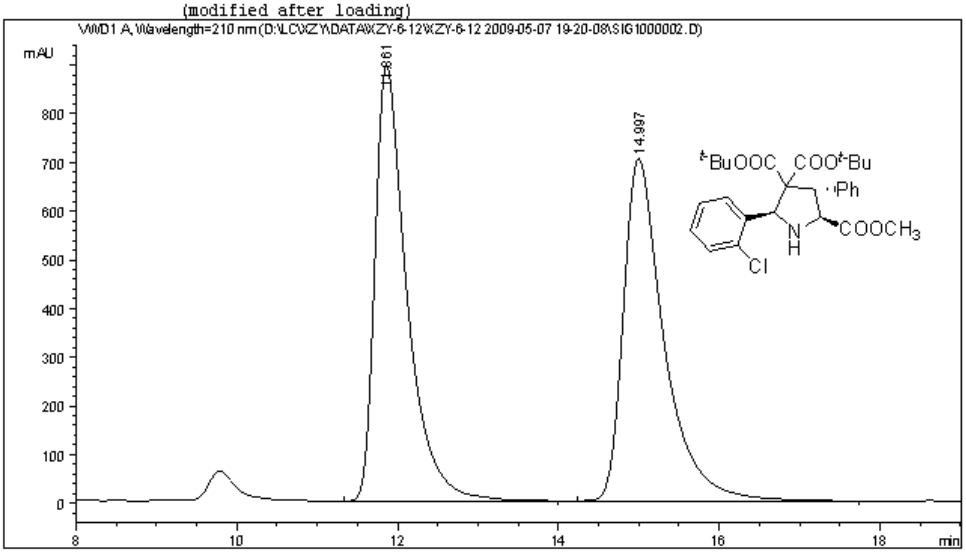
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.900 BB    0.4028 1.03964e4   384.47974  89.0141
       2  15.052 BB    0.4964 1283.09985   38.95367  10.9859

    Totals :                  1.16795e4   423.43341

    ================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-20-08\SIG1000002.D
Sample Name: xzy-6-12b

```
    ================================================================
    Acq. Operator   : ltl                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 33
    Injection Date  : 5/7/2009 7:32:32 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-20-08\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-12\XZY-6-12 2009-05-07 19-20-08\SIG1000002.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:22:41 PM by LTL
                      (modified after loading)
```




```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

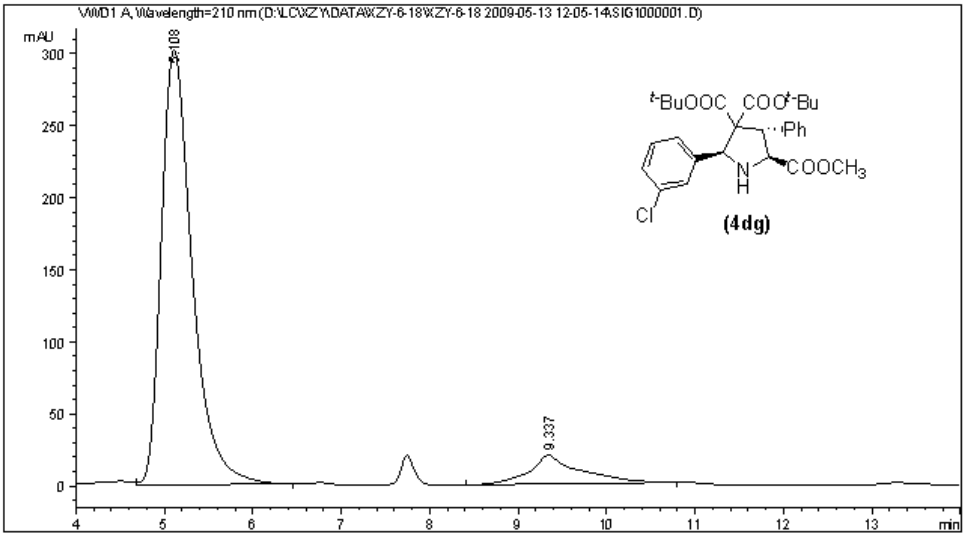
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.861 BB    0.4095 2.47138e4   895.10565  50.0373
       2  14.997 BB    0.5206 2.46769e4   702.24927  49.9627

    Totals :                  4.93907e4  1597.35492


    ================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-05-14\SIG1000001.D
Sample Name: xzy-6-18d

```
    ======================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 12
    Injection Date  : 5/13/2009 12:06:37 PM               Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-05-14\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 12-05-14\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:29:10 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.108 VB    0.3694 7343.61572  302.19672  90.1259
       2   9.337 VB    0.5232  804.55774   20.00068   9.8741

    Totals :                   8148.17346  322.19739

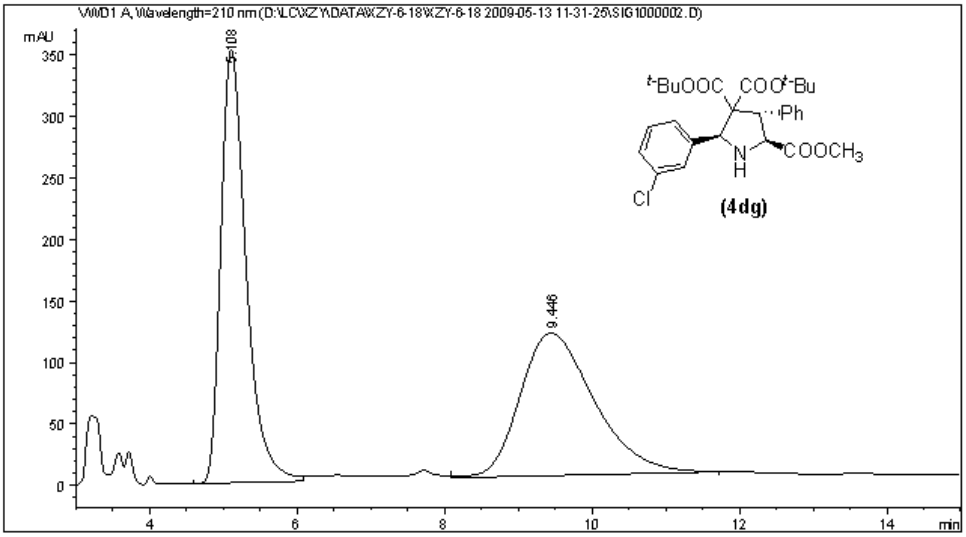
    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:29:12 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 11-31-25\SIG1000002.D
Sample Name: xzy-6-18a

```
    ======================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                    Location : Vial 11
    Injection Date  : 5/13/2009 11:43:53 AM               Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 11-31-25\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 11-31-25\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:27:13 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

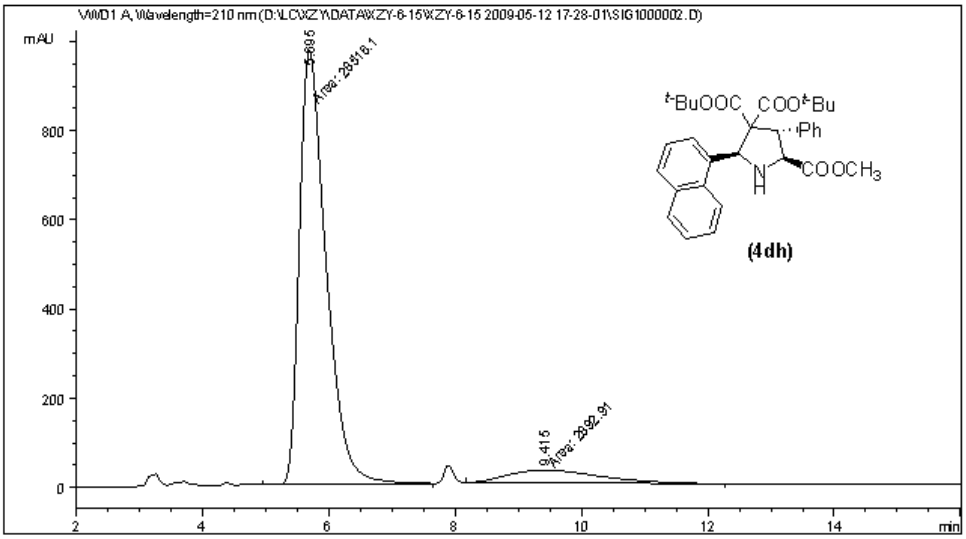
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.108 VB    0.3637 8391.59180  352.37695  50.8417
       2   9.446 VB    1.0755 8113.74609  115.96394  49.1583

    Totals :                  1.65053e4   468.34090

    ======================================================================
                              *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-12 17-28-01\SIG1000002.D
Sample Name: xzy-6-15d

```
    ====================================================================
    Acq. Operator   : ltl                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 74
    Injection Date  : 5/12/2009 5:40:27 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-12 17-28-01\ASH-95-5-10ML-210NM-
                      20MIN.M
    Last changed    : 5/7/2009 5:36:12 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-12 17-28-01\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM-20MIN.M)
    Last changed    : 7/9/2009 4:33:07 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ====================================================================
                              Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.695 MM    0.4893 2.85161e4   971.29913  90.7895
       2   9.415 MM    1.6774 2892.91455   28.74377   9.2105

    Totals :                   3.14090e4  1000.04290

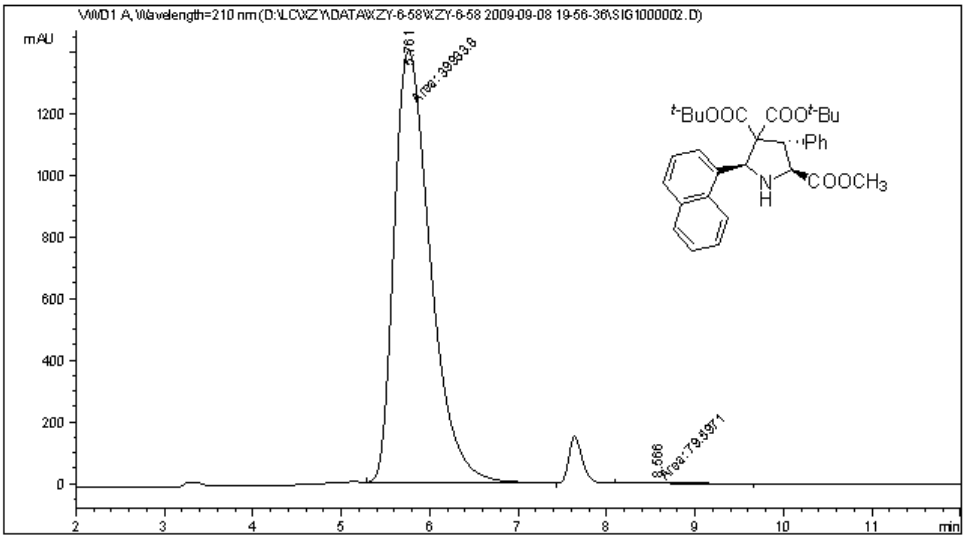

    ====================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:33:08 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-58\XZY-6-58 2009-09-08 19-56-36\SIG1000002.D
Sample Name: xzy-6-58(1)

```
    ================================================================
    Acq. Operator   : zhihai zhang                  Seq. Line :   2
    Acq. Instrument : Instrument 1                   Location : Vial 34
    Injection Date  : 9/8/2009 8:09:04 PM                 Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-58\XZY-6-58 2009-09-08 19-56-36\ASH-95-5-10ML-210NM-
                      20MIN.M
    Last changed    : 5/7/2009 5:36:12 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-58\XZY-6-58 2009-09-08 19-56-36\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM-20MIN.M)
    Last changed    : 9/11/2009 7:06:39 PM by zhihai zhang
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.761 MM    0.4759 3.99338e4  1398.53650  99.8011
       2   8.566 MM    0.6930   79.59708    1.91432   0.1989

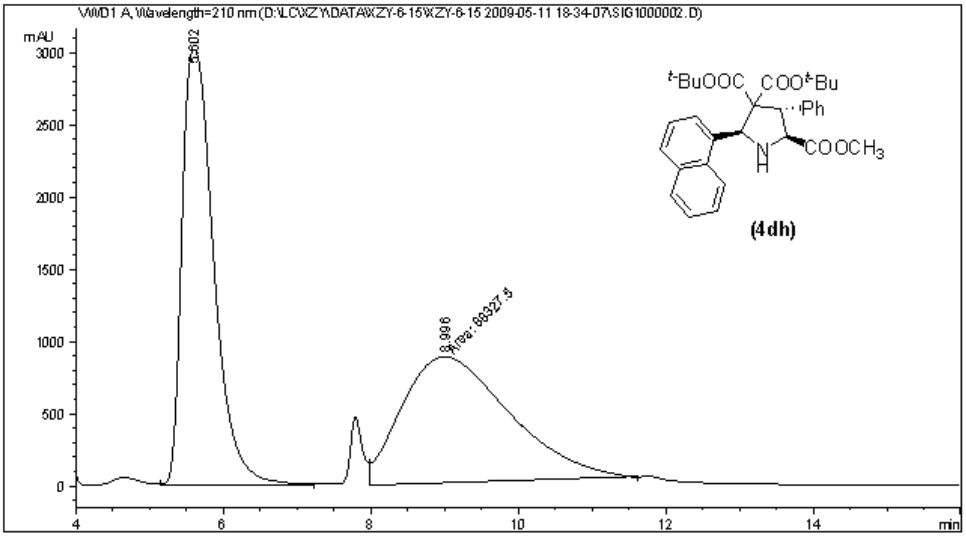
    Totals :                  4.00134e4  1400.45081


    ================================================================
                            *** End of Report ***
```

Instrument 1 9/11/2009 7:06:42 PM zhihai zhang Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 18-34-07\SIG1000002.D
Sample Name: xzy-6-15a

```
    ======================================================================
    Acq. Operator   : dongxiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                   Location : Vial 81
    Injection Date  : 5/11/2009 6:46:22 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 18-34-07\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-15\XZY-6-15 2009-05-11 18-34-07\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:31:13 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.602 VV    0.4659 9.08029e4  3019.55298  50.6910
       2   8.996 MM    1.6865 8.83275e4   872.90399  49.3090

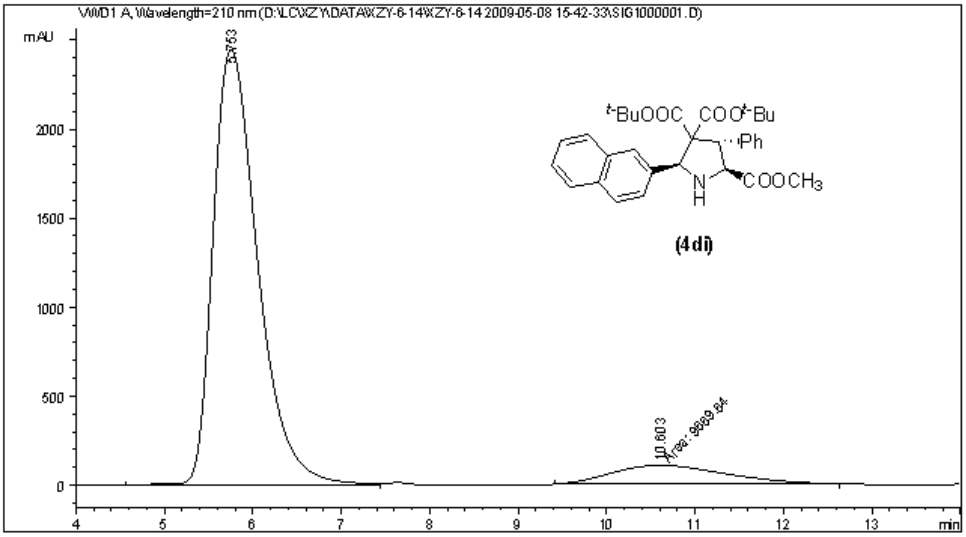
    Totals :                  1.79130e5  3892.45697

    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:31:15 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-42-33\SIG1000001.D
Sample Name: xzy-6-14d

```
    ====================================================================
    Acq. Operator   : ltl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 54
    Injection Date  : 5/8/2009 3:43:53 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-42-33\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-42-33\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:36:32 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```



(4di)

```
    ====================================================================
                              Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.753 VV    0.5424 8.59925e4  2447.60083  89.8919
       2  10.603 MM    1.5208 9669.64355  105.97260  10.1081

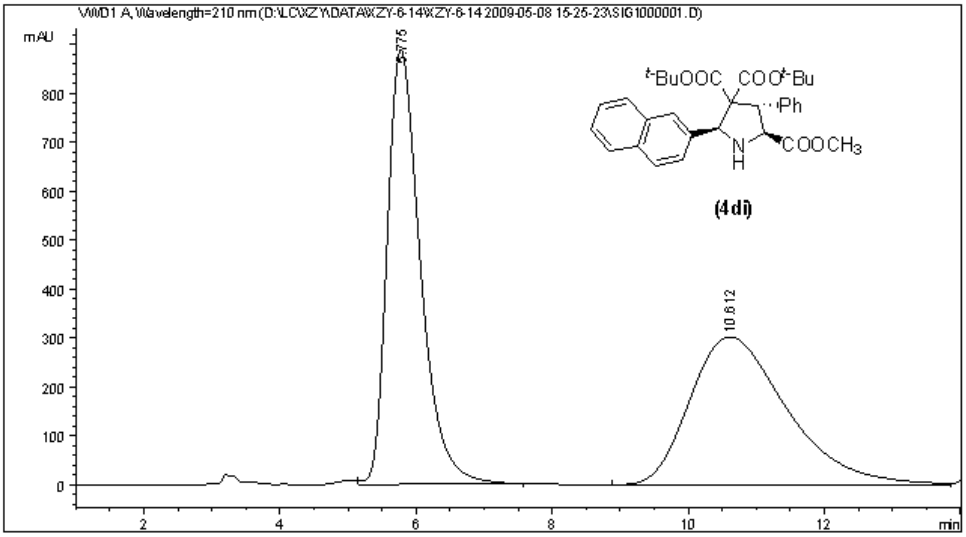
    Totals :                  9.56622e4  2553.57343

    ====================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:36:34 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-25-23\SIG1000001.D
Sample Name: xzy-6-14b

```
    ======================================================================
    Acq. Operator   : ltl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 53
    Injection Date  : 5/8/2009 3:26:47 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-25-23\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-14\XZY-6-14 2009-05-08 15-25-23\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 7/9/2009 4:34:34 PM by LTL
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.775 VB    0.5191 3.01132e4   888.74353  50.1756
       2  10.612 BV    1.5198 2.99024e4   302.22595  49.8244

    Totals :                  6.00156e4  1190.96948

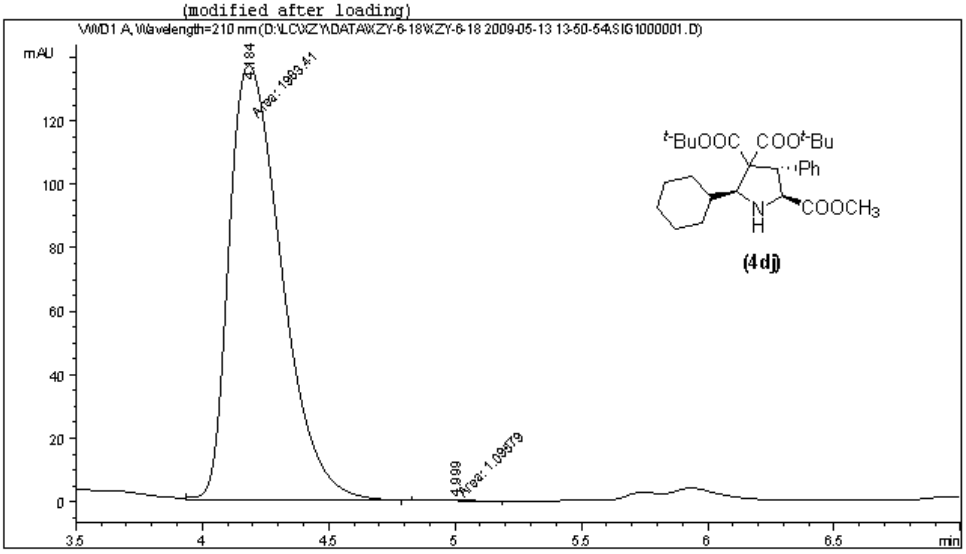
    ======================================================================
                              *** End of Report ***
```

Instrument 1 7/9/2009 4:34:37 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-50-54\SIG1000001.D
Sample Name: xzy-6-18f

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 16
    Injection Date  : 5/13/2009 1:52:14 PM                 Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-50-54\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-50-54\SIG1000001.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:42:18 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                              Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

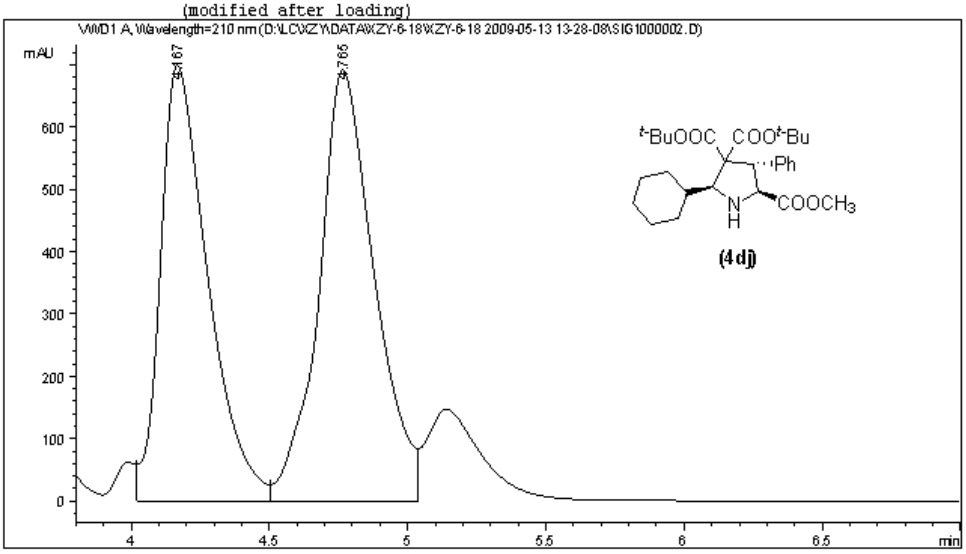
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.184 MM    0.2412 1983.40894  137.03215  99.9448
       2   4.999 MM    0.2061    1.09579 8.85935e-2   0.0552

    Totals :                  1984.50473  137.12074


    ====================================================================
                              *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-28-08\SIG1000002.D
Sample Name: xzy-6-18c

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                    Location : Vial 15
    Injection Date  : 5/13/2009 1:40:31 PM                 Inj :   1
                                                     Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-28-08\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-18\XZY-6-18 2009-05-13 13-28-08\SIG1000002.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:38:32 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

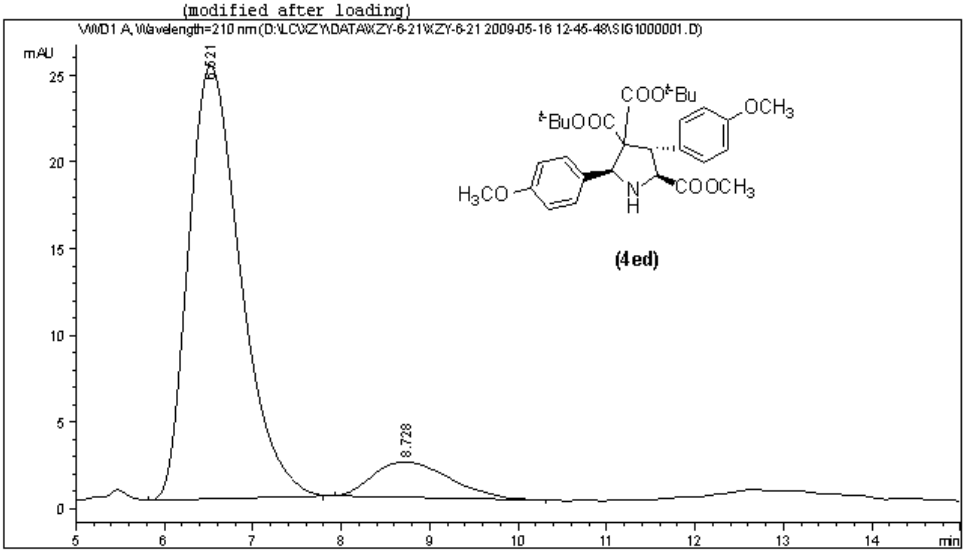
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.167 VV    0.1781 8317.36426  699.23767  46.2907
       2   4.765 VV    0.2050 9650.29883  691.56787  53.7093

    Totals :                  1.79677e4  1390.80554


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-45-48\SIG1000001.D
Sample Name: xzy-6-21d

Acq. Operator	: dong xiuqin	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 6
Injection Date	: 5/16/2009 12:47:31 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-45-48\ASH-10-90-10ML-210NM.M		
Last changed	: 3/4/2009 10:02:27 AM by liang gang		
Analysis Method	: D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-45-48\SIG1000001.D\DA.M (ASH-10-90-10ML-210NM.M)		
Last changed	: 7/9/2009 4:45:54 PM by LTL (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

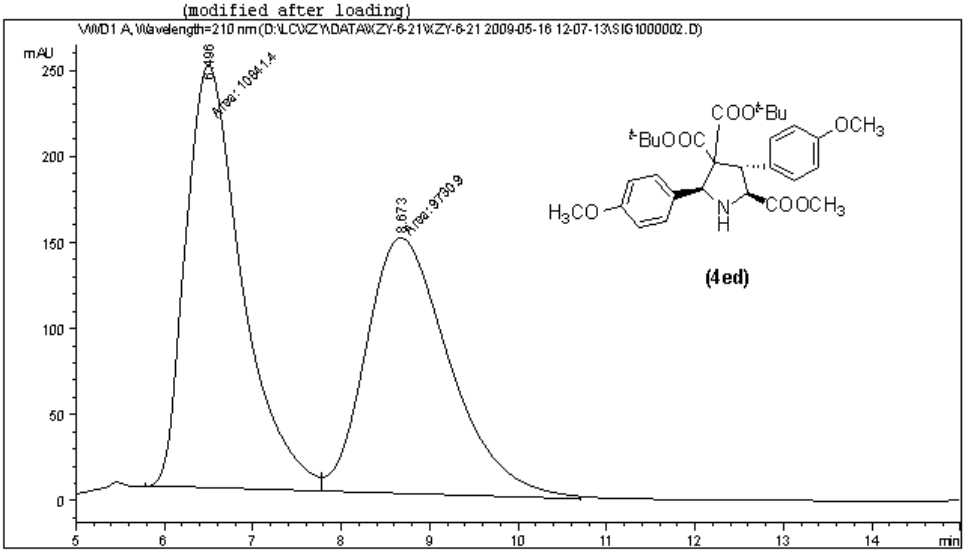
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.521	VB	0.6461	1020.54431	25.02669	89.1785
2	8.728	BB	0.8071	123.84005	2.02379	10.8215
Totals :				1144.38436	27.05048	

*** End of Report ***

Data File D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-07-13\SIG1000002.D
Sample Name: xzy-6-21b

```
    ================================================================
    Acq. Operator   : dong xiuqin                  Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 5
    Injection Date  : 5/16/2009 12:19:36 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-07-13\ASH-10-90-10ML-210NM.M
    Last changed    : 3/4/2009 10:02:27 AM by liang gang
    Analysis Method : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 12-07-13\SIG1000002.D\DA.M (ASH-
                      10-90-10ML-210NM.M)
    Last changed    : 7/9/2009 4:44:04 PM by LTL
                      (modified after loading)
```




```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   6.496 MF    0.7365 1.08414e4   245.34370  52.6991
       2   8.673 FM    1.0921 9730.89844  148.50185  47.3009

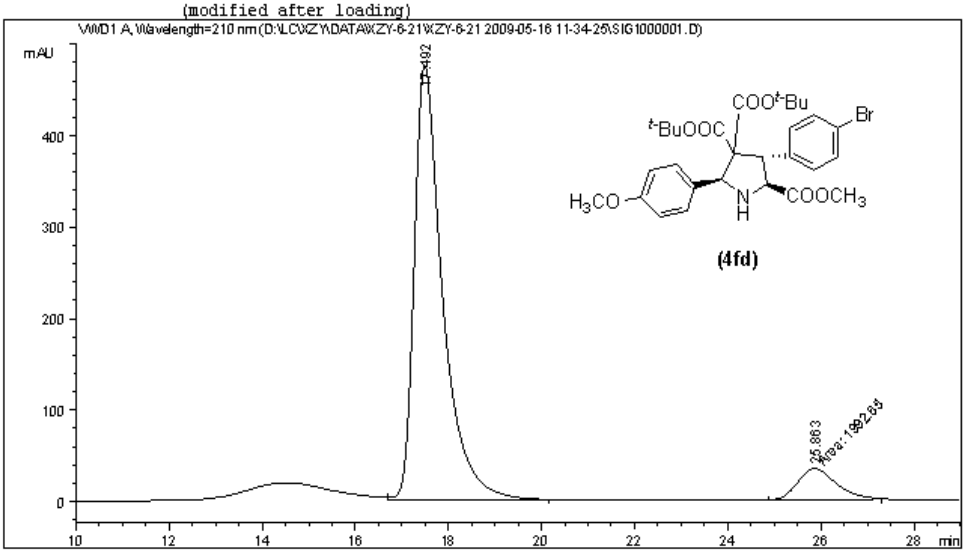
    Totals :                   2.05723e4   393.84555


    ================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 4:44:06 PM LTL Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 11-34-25\SIG1000001.D
Sample Name: xzy-6-21c

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 4
    Injection Date  : 5/16/2009 11:36:16 AM              Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 11-34-25\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 11-34-25\SIG1000001.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:50:22 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  17.492 VB    0.6244 2.01044e4   475.36859  90.9823
       2  25.863 MM    0.9673 1992.65173   34.33250   9.0177

    Totals :                  2.20970e4   509.70110

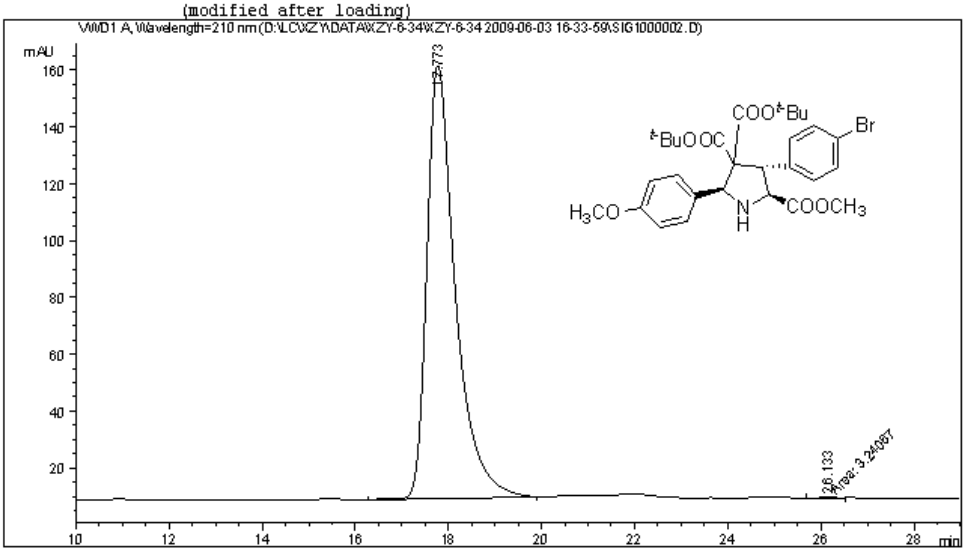

    ====================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 4:50:25 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-34\XZY-6-34 2009-06-03 16-33-59\SIG1000002.D
Sample Name: xzy-6-34

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                    Location : Vial 6
    Injection Date  : 6/3/2009 4:46:20 PM                  Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-34\XZY-6-34 2009-06-03 16-33-59\ADH-5-95-10ML-210NM-
                      30MIN.M
    Last changed    : 5/11/2009 10:01:09 PM by zhang zhihai
    Analysis Method : D:\LC\XZY\DATA\XZY-6-34\XZY-6-34 2009-06-03 16-33-59\SIG1000002.D\DA.M (ADH-5-
                      95-10ML-210NM-30MIN.M)
    Last changed    : 9/11/2009 7:02:43 PM by zhihai zhang
                      (modified after loading)
```




```
    ====================================================================
                              Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       1  17.773 BB    0.6306 6480.78027  152.69122  99.9500
       2  26.133 MM    0.4639    3.24067 1.1644le-1   0.0500

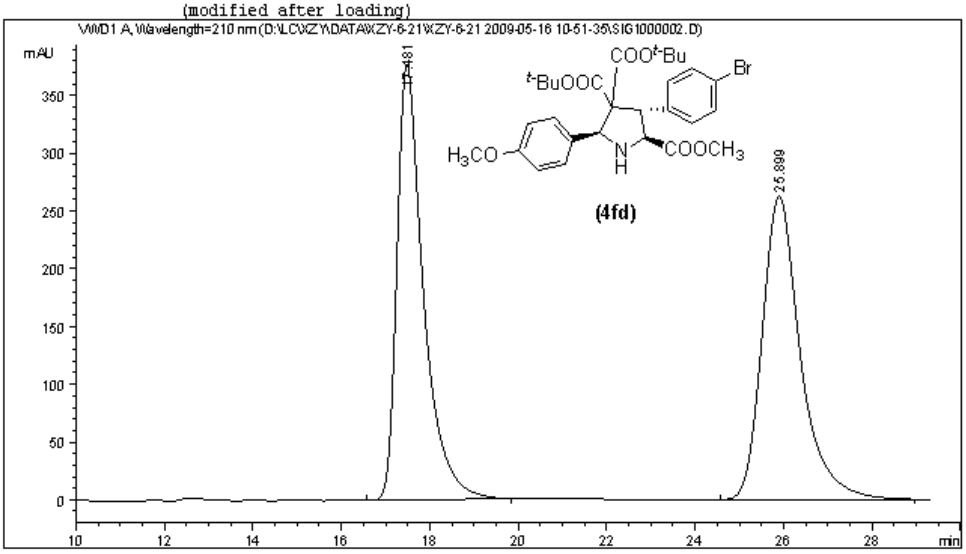
    Totals :                  6484.02095  152.80766


    ====================================================================
                              *** End of Report ***
```

Instrument 1 9/11/2009 7:02:50 PM zhihai zhang Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 10-51-35\SIG1000002.D
Sample Name: xzy-6-21a

```
    ====================================================================
    Acq. Operator   : dong xiuqin                    Seq. Line :   2
    Acq. Instrument : Instrument 1                    Location : Vial 3
    Injection Date  : 5/16/2009 11:03:57 AM               Inj :   1
                                                    Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 10-51-35\ADH-5-95-10ML-210NM.M
    Last changed    : 5/7/2009 7:17:41 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-21\XZY-6-21 2009-05-16 10-51-35\SIG1000002.D\DA.M (ADH-5-
                      95-10ML-210NM.M)
    Last changed    : 7/9/2009 4:47:11 PM by LTL
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

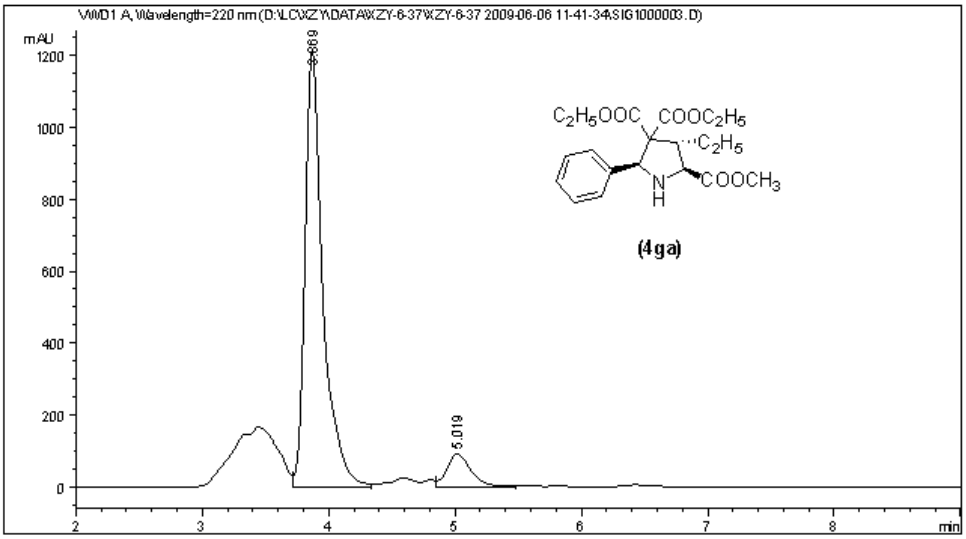
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  17.481 VB    0.6190 1.56936e4   376.33389  49.9464
       2  25.899 BB    0.9013 1.57273e4   262.53937  50.0536

    Totals :                  3.14209e4   638.87326


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-6-37\XZY-6-37 2009-06-06 11-41-34\SIG1000003.D
Sample Name: xzy-6-37b

```
    ==================================================================
    Acq. Operator   : liang gang                    Seq. Line :   3
    Acq. Instrument : Instrument 1                   Location : Vial 42
    Injection Date  : 6/6/2009 12:05:22 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-37\XZY-6-37 2009-06-06 11-41-34\ASH-30-70-10ML-220NM-
                      10MIN.M
    Last changed    : 1/6/2009 9:03:30 AM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-6-37\XZY-6-37 2009-06-06 11-41-34\SIG1000003.D\DA.M (ASH-
                      30-70-10ML-220NM-10MIN.M)
    Last changed    : 7/9/2009 4:55:44 PM by LTL
                      (modified after loading)
    Method Info     : ASH-30-70-1.0ML-220(20)
```



(4ga)

```
    ==================================================================
                              Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.869 VV    0.1450 1.18972e4  1212.60950  90.7729
       2   5.019 VV    0.1999 1209.35132   91.16996   9.2271

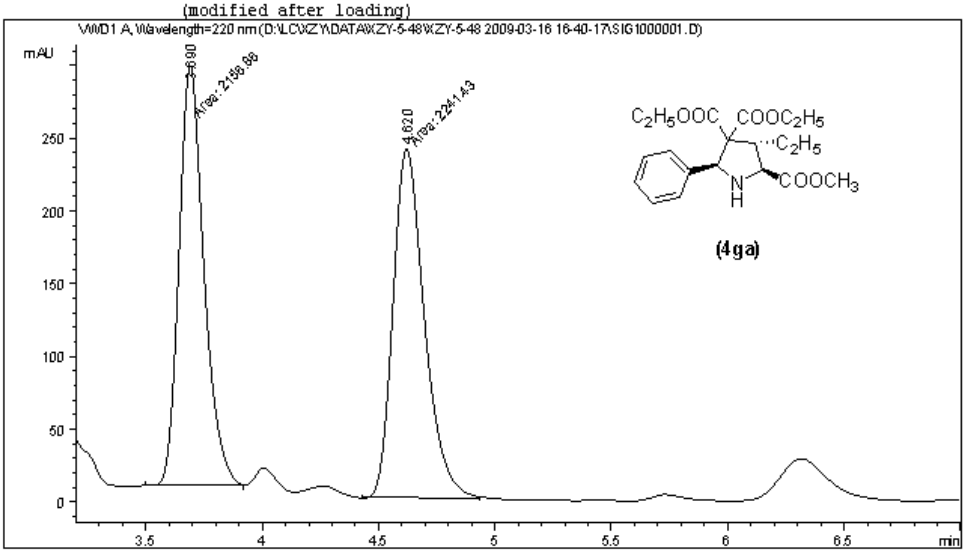
    Totals :                   1.31066e4  1303.77946


    ==================================================================
                              *** End of Report ***

Instrument 1 7/9/2009 4:55:46 PM LTL                          Page 1 of 1
```

Data File D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-40-17\SIG1000001.D
Sample Name: xzy-5-48c

```
    ====================================================================
    Acq. Operator   : liang qang                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 23
    Injection Date  : 3/16/2009 4:41:34 PM                Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-40-17\ASH-30-70-220NM-1ML.M
    Last changed    : 7/1/2008 8:17:03 PM by xzy
    Analysis Method : D:\LC\XZY\DATA\XZY-5-48\XZY-5-48 2009-03-16 16-40-17\SIG1000001.D\DA.M (ASH-
                      30-70-220NM-1ML.M)
    Last changed    : 7/9/2009 4:53:11 PM by LTL
                      (modified after loading)
```



(4ga)

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.690 MM    0.1242 2156.86401  289.54105  49.0387
       2   4.620 MM    0.1558 2241.42749  239.77269  50.9613

    Totals :                   4398.29150  529.31374

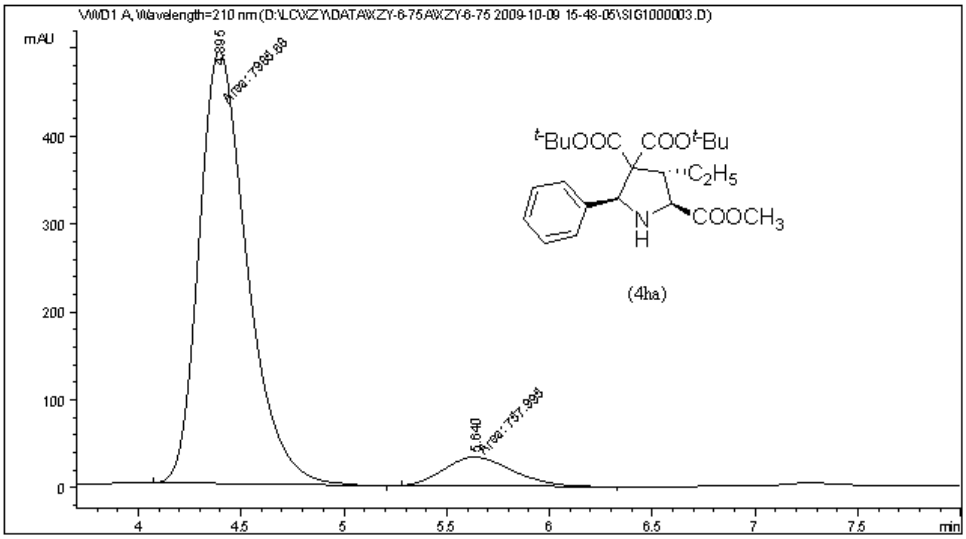

    ====================================================================
                            *** End of Report ***
```

Instrument 1 7/9/2009 4:53:13 PM LTL

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 15-48-05\SIG1000003.D
Sample Name: xzy-6-75b

Acq. Operator	: zzh	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 5
Injection Date	: 10/9/2009 4:22:09 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 15-48-05\ASH-95-5-10ML-210NM-20MIN.M		
Last changed	: 5/7/2009 5:36:12 PM by ltl		
Analysis Method	: D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 15-48-05\SIG1000003.D\DA.M (ASH-95-5-10ML-210NM-20MIN.M)		
Last changed	: 11/16/2009 9:51:49 AM by DXQ (modified after loading)		
Method Info	: ASH-95-5-210NM-1.0ML		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.395	MM	0.2705	7985.67871	492.10446	91.3309
2	5.640	MM	0.3856	757.99506	32.75950	8.6691
Totals :				8743.67377	524.86396	

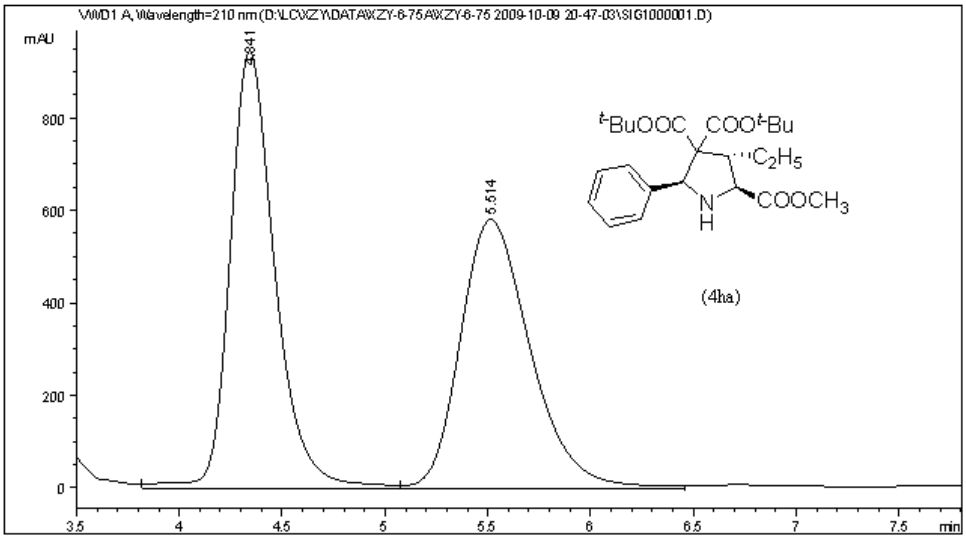
*** End of Report ***

Instrument 1 11/16/2009 9:51:51 AM DXQ

Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 20-47-03\SIG1000001.D
Sample Name: xzy-6-75a-rac

```
    ================================================================
    Acq. Operator   : zzh                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 11
    Injection Date  : 10/9/2009 8:48:24 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 20-47-03\ASH-95-5-10ML-210NM-
                      20MIN.M
    Last changed    : 5/7/2009 5:36:12 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-75A\XZY-6-75 2009-10-09 20-47-03\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM-20MIN.M)
    Last changed    : 11/16/2009 9:45:42 AM by dxq
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.341 VV    0.2307 1.43431e4   946.22614  51.1215
       2   5.514 VV    0.3639 1.37138e4   581.51447  48.8785

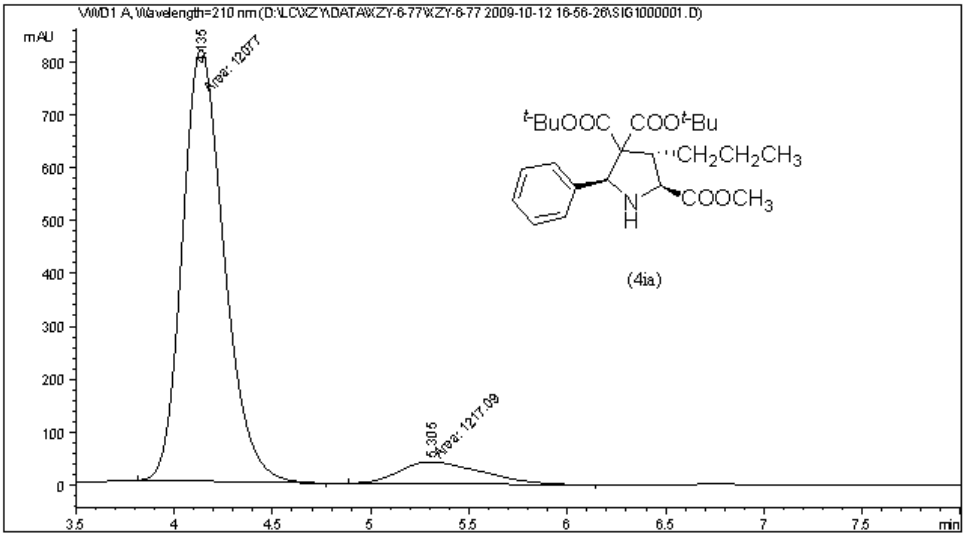
    Totals :                  2.80569e4  1527.74060


    ================================================================
                            *** End of Report ***
```

Instrument 1 11/16/2009 9:45:48 AM dxq Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-56-26\SIG1000001.D
Sample Name: xzy-6-77c

```
    ======================================================================
    Acq. Operator   : liangqang                     Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 42
    Injection Date  : 10/12/2009 4:58:15 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-56-26\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-56-26\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 11/16/2009 9:56:10 AM by DXQ
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.135 MM    0.2466 1.20770e4   816.28619  90.8449
       2   5.305 MM    0.4864 1217.09070   41.70797   9.1551

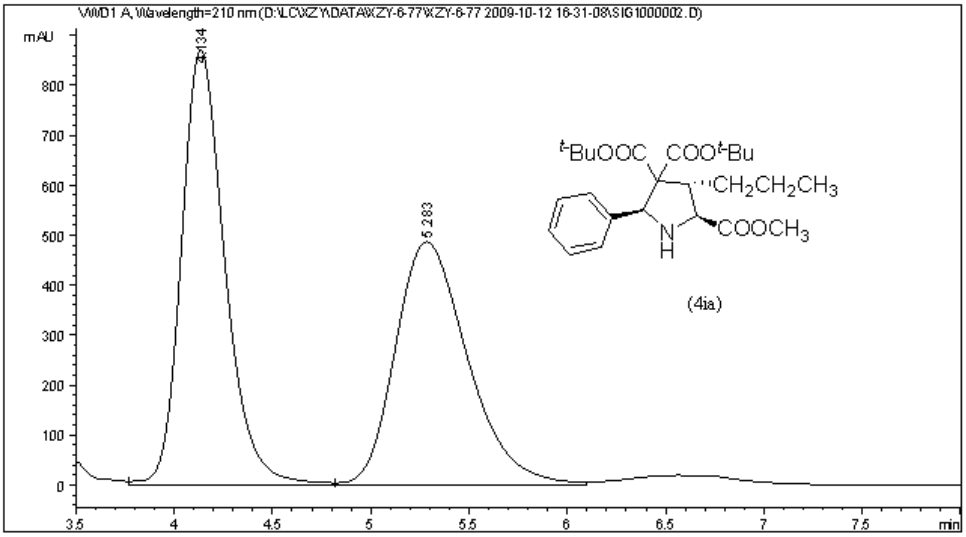
    Totals :                  1.3294le4   857.99416

    ======================================================================
                              *** End of Report ***
```

Instrument 1 11/16/2009 9:56:19 AM DXQ Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-31-08\SIG1000002.D
Sample Name: xzy-6-77a

```
    ======================================================================
    Acq. Operator   : liangqang                    Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 41
    Injection Date  : 10/12/2009 4:43:30 PM            Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-31-08\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-77\XZY-6-77 2009-10-12 16-31-08\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 11/16/2009 9:53:46 AM by DXQ
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   4.134 VV    0.2348 1.3429le4   872.72162  51.8284
       2   5.283 VV    0.3947 1.24816e4   487.61093  48.1716

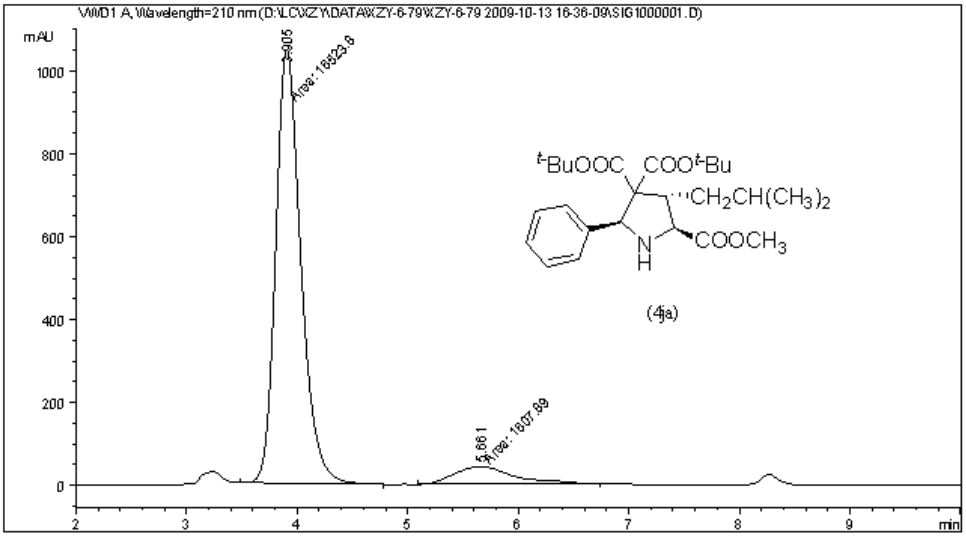
    Totals :                   2.59106e4  1360.33255

    ======================================================================
                               *** End of Report ***
```

Instrument 1 11/16/2009 9:53:49 AM DXQ Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-36-09\SIG1000001.D
Sample Name: xzy-6-79b

```
    ======================================================================
    Acq. Operator   : liang qang                    Seq. Line :   1
    Acq. Instrument : Instrument 1                   Location : Vial 72
    Injection Date  : 10/13/2009 4:37:26 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-36-09\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-36-09\SIG1000001.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 11/16/2009 9:59:38 AM by DXQ
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.905 MM    0.2629 1.65238e4  1047.54309  91.1321
       2   5.661 MM    0.6276 1607.89124   42.69704   8.8679

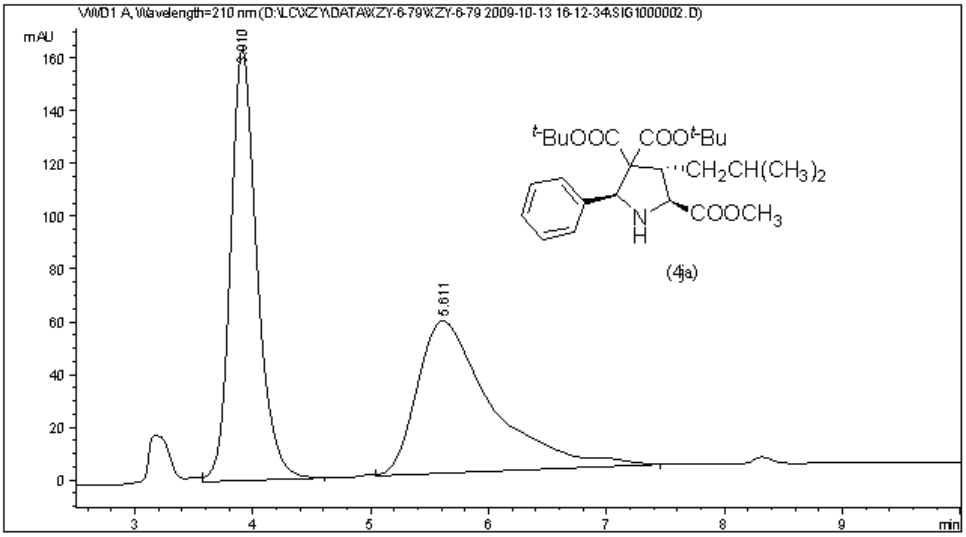
    Totals :                   1.81317e4  1090.24014

    ======================================================================
                              *** End of Report ***
```

Instrument 1 11/16/2009 9:59:40 AM DXQ Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-12-34\SIG1000002.D
Sample Name: xzy-6-79a

```
    ======================================================================
    Acq. Operator   : liang qang                    Seq. Line :   2
    Acq. Instrument : Instrument 1                   Location : Vial 71
    Injection Date  : 10/13/2009 4:24:54 PM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-12-34\ASH-95-5-10ML-210NM.M
    Last changed    : 5/7/2009 4:29:38 PM by ltl
    Analysis Method : D:\LC\XZY\DATA\XZY-6-79\XZY-6-79 2009-10-13 16-12-34\SIG1000002.D\DA.M (ASH-
                      95-5-10ML-210NM.M)
    Last changed    : 11/16/2009 9:57:59 AM by DXQ
                      (modified after loading)
    Method Info     : ASH-95-5-210NM-1.0ML
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.910 VB    0.2373 2549.33057  163.48143  50.1074
       2   5.611 VB    0.6366 2538.39844   57.74630  49.8926

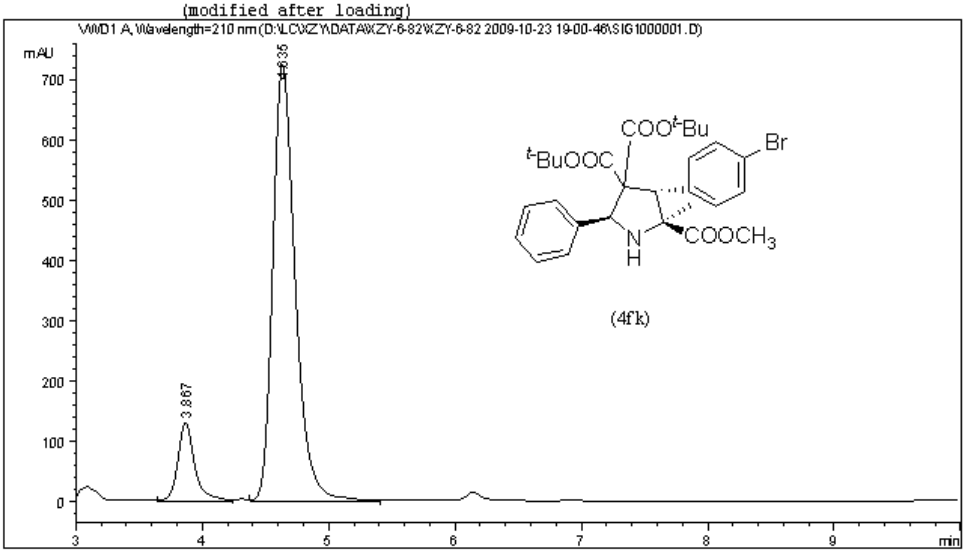
    Totals :                   5087.72900  221.22773

    ======================================================================
                              *** End of Report ***
```

Instrument 1 11/16/2009 9:58:01 AM DXQ Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 19-00-46\SIG1000001.D
Sample Name: XZY-6-82B

```
    ======================================================================
    Acq. Operator   : DXQ                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 2
    Injection Date  : 10/23/2009 7:02:31 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 19-00-46\ODH-5-95-10ML-210NM.M
    Last changed    : 10/23/2009 6:30:15 PM by DXQ
    Analysis Method : D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 19-00-46\SIG1000001.D\DA.M (ODH-5-
                      95-10ML-210NM.M)
    Last changed    : 11/16/2009 10:02:40 AM by DXQ
                      (modified after loading)
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.867 VV    0.1410 1225.56250  131.18359  12.0695
       2   4.635 VV    0.1891 8928.66797  723.46613  87.9305

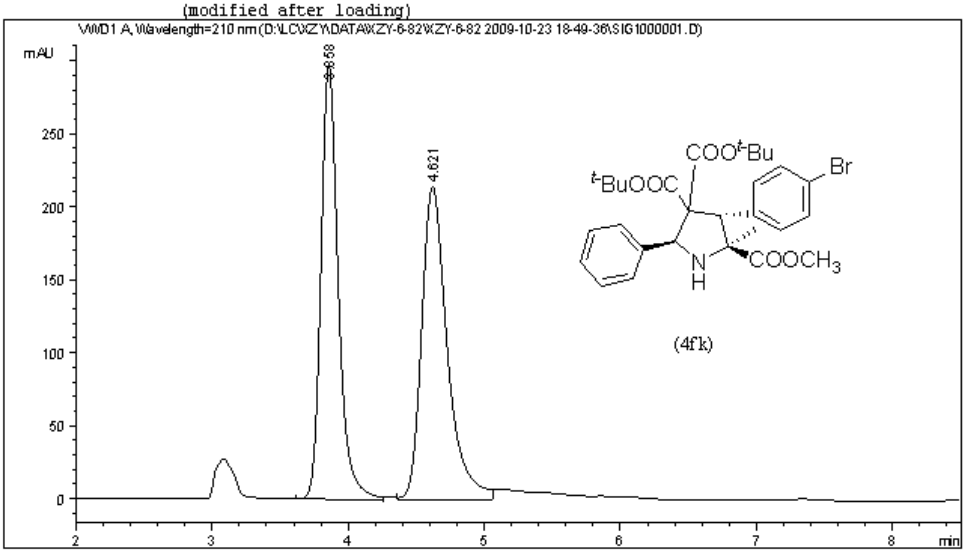
    Totals :                  1.01542e4   854.64972


    ======================================================================
                            *** End of Report ***
```

Instrument 1 11/16/2009 10:02:42 AM DXQ Page 1 of 1

Data File D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 18-49-36\SIG1000001.D
Sample Name: XZY-6-82A

```
    ====================================================================
    Acq. Operator   : DXQ                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 1
    Injection Date  : 10/23/2009 6:51:06 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 18-49-36\ODH-5-95-10ML-210NM.M
    Last changed    : 10/23/2009 6:30:15 PM by DXQ
    Analysis Method : D:\LC\XZY\DATA\XZY-6-82\XZY-6-82 2009-10-23 18-49-36\SIG1000001.D\DA.M (ODH-5-
                      95-10ML-210NM.M)
    Last changed    : 11/16/2009 10:01:15 AM by DXQ
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

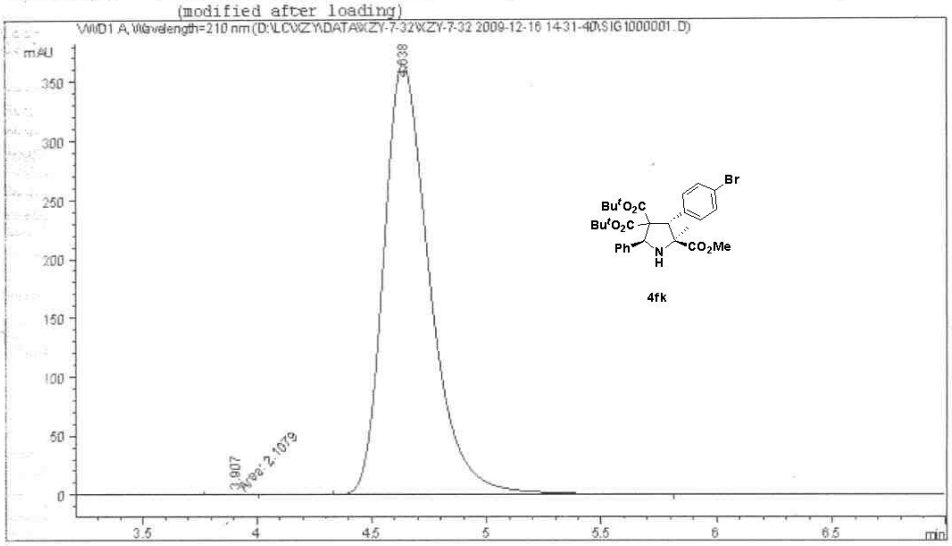
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   3.858 VV    0.1333 2617.43555  297.19235  48.3218
       2   4.621 VV    0.1962 2799.23584  214.09074  51.6782

    Totals :                   5416.67139  511.28310


    ====================================================================
                            *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-7-32\XZY-7-32 2009-12-16 14-31-40\SIG1000001.D
Sample Name: xzy-7-32(crystall-1)-L2

```
==================================================================
Acq. Operator   : ltl                          Seq. Line :   1
Acq. Instrument : Instrument 1                  Location : Vial 1
Injection Date  : 12/16/2009 2:32:55 PM            Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\XZY\DATA\XZY-7-32\XZY-7-32 2009-12-16 14-31-40\ODH-5-95-10ML-210NM-
                  10MIN.M
Last changed    : 12/5/2009 11:27:24 AM by liang gang
Analysis Method : D:\LC\XZY\DATA\XZY-7-32\XZY-7-32 2009-12-16 14-31-40\SIG1000001.D\DA.M (ODH-5-
                  95-10ML-210NM-10MIN.M)
Last changed    : 1/18/2010 8:42:35 PM by THL
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

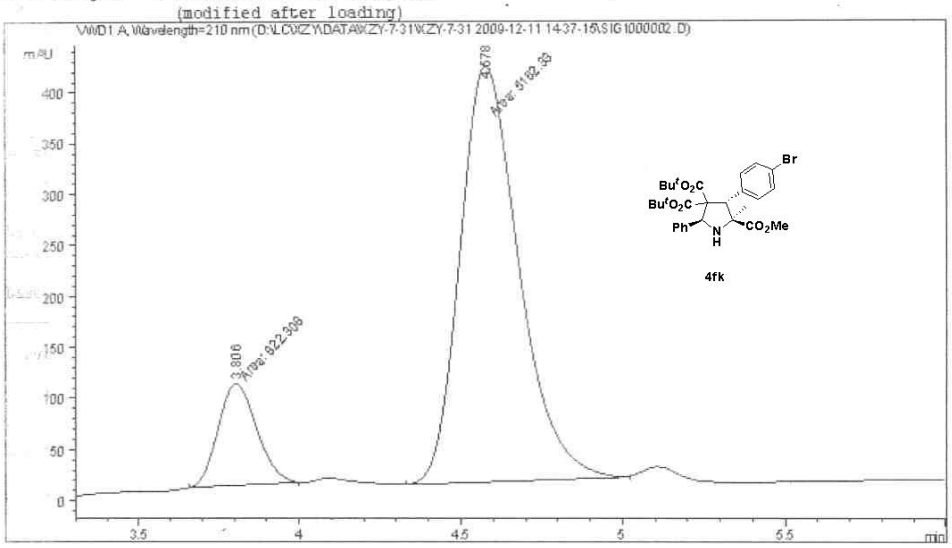
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|------|----------|----------|--------|
   1   3.907 MM    0.1174    2.10790 2.99198e-1   0.0415
   2   4.638 VB    0.2119 5072.39014  364.35315  99.9585

Totals :                   5074.49803  364.65235

==================================================================
                        *** End of Report ***
```

Data File D:\LC\XZY\DATA\XZY-7-31\XZY-7-31 2009-12-11 14-37-15\SIG1000002.D
Sample Name: xzy-7-31A(L-)

```
==================================================================
Acq. Operator   : dxg                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 1
Injection Date  : 12/11/2009 2:49:32 PM            Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\XZY\DATA\XZY-7-31\XZY-7-31 2009-12-11 14-37-15\ODH-5-95-10ML-210NM-
                  10MIN.M
Last changed    : 12/5/2009 11:27:24 AM by liang gang
Analysis Method : D:\LC\XZY\DATA\XZY-7-31\XZY-7-31 2009-12-11 14-37-15\SIG1000002.D\DA.M (ODH-5-
                  95-10ML-210NM-10MIN.M)
Last changed    : 1/18/2010 8:38:58 PM by THL
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
-----|-------|----|-------|----------|----------|--------|
   1   3.806 MM    0.1385  822.30756   98.94784  13.7403
   2   4.578 MM    0.2112 5162.32813  407.37796  86.2597

Totals :                  5984.63568  506.32580

==================================================================
                        *** End of Report ***
```

Instrument 1 1/18/2010 8:39:03 PM THL

Page 1 of 1