Electronic Supplementary Material (ESI) for Chemical Communications

Asymmetric construction of trifluoromethylated pyrrolidines *via* Cu(I)-catalyzed 1,3-dipolar cycloaddition of azomethine ylides with 4,4,4-trifluorocrotonates

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Supporting Information

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General Remarks.

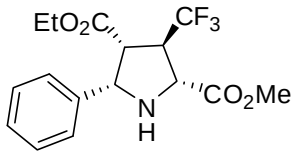
^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quartet, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 or VARIAN Mercury 150 MHz spectrometer in $CDCl_3$. 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 **L3-L7** were prepared according to the literature procedure reported by us.[1] *trans* and *cis*-4,4,4-Trifluorocrotonates were prepared according to the literature procedure.[2] The racemic adducts were obtained by using AgOAc/PPh_3 as the catalyst. The absolute configuration of (2*R*,3*S*,4*R*,5*S*)-**6cg** and (2*R*,3*S*,4*S*,5*S*)-**7cg** achieved by $Cu(CH_3CN)_4BF_4$/(*S*)-TF-BiphamPhos **L7** 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 *trans* or *cis*-4,4,4-Trifluorocrotonates Catalyzed by AgOAc/PPh_3 Complex

Under argon atmosphere, PPh_3 (6.6 mg, 0.0253 mmol) and AgOAc (3.8 mg, 0.023 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.35 mmol), Et_3N (0.03 mmol) and *trans* or *cis*-4,4,4-Trifluorocrotonates (0.23 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, which was used as the racemic sample for the chiral HPLC analysis.

General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with *trans* or *cis*-4,4,4-Trifluorocrotonates Catalyzed by $Cu(CH_3CN)_4BF_4$ /(*S*)-TF-BiphamPhos Complex

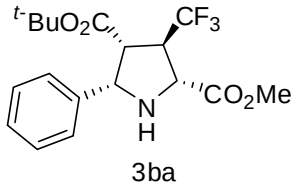
Under argon atmosphere (*S*)-TF-BiphamPhos **L7**（6.1 mg, 0.0076 mmol) and $Cu(CH_3CN)_4BF_4$ (2.1 mg, 0.0069 mmol) were dissolved in 2 mL DCM, and stirred at room temperature for about 1h. Then, imine substrate (0.35 mmol), Et_3N (0.03 mmol) and *trans* or *cis*-4,4,4-Trifluorocrotonates (0.23 mmol) 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 product purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.



(3aa)

(2*R*,3*R*,4*R*,5*S*)-4-ethyl 2-methyl 5-phenyl-3-(trifluoromethyl)pyrrolidine-2,4-di-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 57 ℃; $[\alpha]^{25}_D$ = -30.7 (*c* 0.88, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.24 (m, 5H), 4.55 (d, *J* = 7.8 Hz, 1H), 3.99 (d, *J* = 6.3 Hz, 1H), 3.78 (s, 3H), 3.70-3.60 (m, 1H), 3.56-3.48 (m, 2H); 3.33-3.30 (m, 1H); 2.87 (brs, 1H), 0.68 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 171.17, 170.87, 136.73, 128.49, 128.30, 126.69, 126.32 (q, J_{CF} = 270.4Hz), 65.82, 61.00, 60.40, 52.79, 50.89, 50.11 (q, J_{CF} = 27.2Hz), 13.36; IR (KBr) ν 3683, 3622, 3021, 2975, 2400, 1731, 1516, 1426, 1215, 1045, 929, 756 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 86% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.64 and 6.68 min.

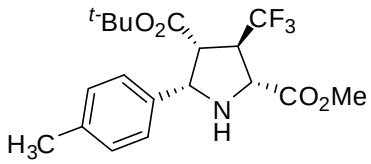


3ba

(3ba)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-phenyl-3-(trifluoromethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 82% yield. m.p. 85 ℃; $[\alpha]^{25}_{D}$ = -29.0 (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.27-7.20 (m, 5H), 4.51 (d, *J* = 7.5 Hz, 1H), 3.95 (d, *J* = 6.6 Hz, 1H), 3.78 (s, 3H), 3.48-3.41 (m, 1H), 3.29-3.26 (m, 1H); 2.77 (brs, 1H), 0.93 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) δ 171.18, 169.91, 136.82, 128.18, 127.70, 126.97, 126.40 (q, J_{CF} = 277.2 Hz), 81.63, 65.51, 60.45, 52.73, 51.31, 50.91 (q, J_{CF} = 28.4 Hz), 27.26; IR (KBr) ν 3684, 3622, 3019, 2977, 2439, 1746, 1715, 1520, 1477, 1370, 1216, 1046, 929, 849, 754, 744, 669 cm^{-1}. HRMS: calcd. for $C_{18}H_{22}F_3NO_4$: 373.1501, found. 373.1502. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.84 and 7.81 min.

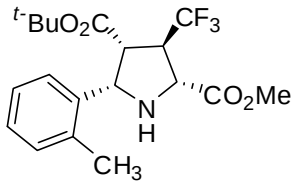


(3bb)

(2*R*,3*R*,4*R*,5S)-4-*tert*-butyl 2-methyl 5-p-tolyl-3-(trifluoromethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 72% yield. m.p. 82 ℃; $[\alpha]^{25}_{D}$ = -52.1 (*c* 0.36, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.24-7.13 (m, 4H), 4.60 (d, *J* = 6.9 Hz, 1H), 4.08 (d, *J* = 6.9 Hz, 1H), 3.87 (s, 3H), 3.52-3.47 (m, 1H), 3.36-3.34 (m, 1H), 2.33 (s, 3H), 1.04 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 171.21, 170.00, 137.44, 133.71, 128.84, 126.87, 126.42 (q, J_{CF} = 277.2 Hz), 81.66, 65.42, 60.54, 52.78, 51.46, 51.03 (q, J_{CF} = 27.2 Hz),

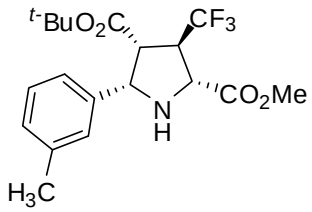
27.35, 20.98; IR (KBr) ν 3684, 3621, 3019, 2898, 2400, 1744, 1521, 1423, 1216, 1045, 929, 776 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_4$: 387.1657, found. 387.1655 The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 4.17 and 4.78 min.



(3bc)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-o-tolyl-3-(trifluoromethyl)pyrrolidine-2,4-dicarboxylate

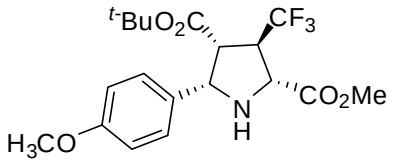
The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 73 ℃; $[\alpha]^{25}_D$ = -68.9 (*c* 0.65, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.40-7.37 (m, 1H), 7.22-7.18 (m, 3H), 4.69 (d, *J* = 7.5 Hz, 1H), 4.06 (d, *J* = 6.9 Hz, 1H), 3.88 (s, 3H), 3.67-3.60 (m, 1H), 3.45-3.41 (m, 1H), 2.38 (s, 3H), 0.96 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 171.24, 170.04, 137.79, 136.66, 128.45, 128.16, 127.58, 126.46 (q, J_{CF} = 276.7 Hz), 124.10, 81.60, 65.63, 60.56, 52.78, 51.41, 51.00 (q, J_{CF} = 27.2 Hz), 27.34, 21.30; IR (KBr) ν 3684, 3620, 3019, 2977, 2400, 1746, 1521, 1423, 1221, 1046, 929, 782, 751, 669 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_4$: 387.1657, found. 387.1659. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.40 and 7.44 min.



(3bd)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-m-tolyl-3-(trifluoromethyl)pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. m.p. 82 ℃; $[\alpha]^{25}_{D}$ = -31.2 (*c* 0.80, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.26-7.08 (m, 4H), 4.59 (d, *J* = 7.2 Hz, 1H), 4.07 (d, *J* = 6.3 Hz, 1H), 3.87 (s, 3H), 3.54-3.47 (m, 1H), 3.37-3.33 (m, 1H), 2.34 (s, 3H), 1.03 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 171.21, 170.02, 137.78, 136.61, 128.45, 128.16, 127.55, 126.37 (q, J_{CF} = 267.8 Hz), 124.07, 81.60, 65.61, 60.52, 52.78, 51.38, 50.97 (q, J_{CF} = 26.1 Hz), 27.32, 21.30; IR (KBr) ν 3685, 3624, 3017, 2977, 2400, 1735, 1519, 1420, 1215, 1046, 929, 753 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_4$: 387.1657, found. 387.1656. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.36 and 6.83 min.

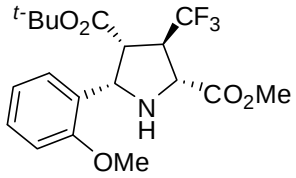


(3be)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-(4-methoxyphenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. m.p. 74 ℃; $[\alpha]^{25}_{D}$ = -32.3 (*c* 0.85, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.29-7.26 (d, *J* = 7.8 Hz, 2H), 6.89-6.86 (d, *J* = 8.1Hz, 2H), 4.57 (d, *J* = 7.2 Hz, 1H), 4.05 (d, *J* = 6.3 Hz, 1H), 3.87 (s, 3H), 3.80 (s, 3H), 3.52 (m, 1H), 3.35-3.32 (m, 1H), 1.06 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 171.21, 170.01, 159.26, 128.91, 128.14, 126.73 (q, J_{CF} = 268.8 Hz), 113.64, 81.67, 65.08, 60.43, 55.30, 52.79, 51.41, 50.91 (q, J_{CF} = 27.2 Hz), 27.41; IR (KBr) ν 3684, 3622, 3019, 2977, 2435, 2400, 1745, 1518, 1424, 1216, 1046, 929, 754, 731, 669 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_5$: 403.1607, found. 403.1610 The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralcel AS-H,

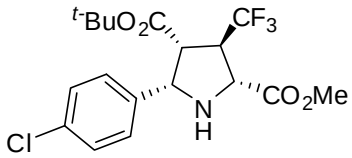
i-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.58 and 6.50 min.



(3bf)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-(2-methoxyphenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 74% yield. m.p. 80 ℃; $[\alpha]^{25}_D$ = -79.0 (*c* 0.88, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.31-7.26 (m, 2H), 6.96-6.86 (m, 2H), 4.67 (d, *J* = 7.2 Hz, 1H), 4.02 (d, *J* = 6.9 Hz, 1H), 3.87 (s, 3H), 3.87 (s, 3H), 3.61-3.58 (m, 1H), 3.50 (m, 1H), 0.97 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 171.31, 170.51, 156.96, 128.77, 126.59 (q, J_{CF} = 277.1 Hz), 126.30, 125.17, 120.32, 109.76, 81.10, 61.44, 60.28, 55.16, 52.72, 51.03 (q, J_{CF} = 27.2 Hz), 49.48, 27.30; IR (KBr) ν 3683, 3621, 3019, 2977, 2400, 1731, 1521, 1423, 1216, 1046, 929, 770, 629 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_5$: 403.1607, found. 403.1609 The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.27 and 8.60 min.

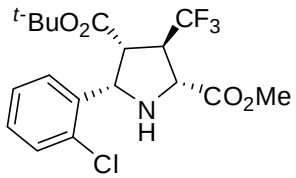


(3bg)

(2*R*,3*R*,4*R*,5S)-4-*tert*-butyl 2-methyl 5-(4-chlorophenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 76% yield. m.p. 71 ℃; $[\alpha]^{25}_D$ = -32.9 (*c* 0.76, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.32 (m, 4H), 4.60 (d, *J* = 6.0 Hz, 1H,), 4.08 (d, *J* = 6.0 Hz, 1H), 3.87 (s, 3H), 3.54 (m, 1H), 3.34 (m, 1H); 1.06 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150

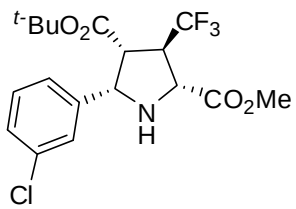
MHz) δ 171.06, 169.64, 135.59, 133.59, 128.47, 128.31, 126.33 (q, J_{CF} = 277.2 Hz), 81.95, 64.78, 60.30, 52.81, 51.10, 50.46 (q, J_{CF} = 26.9 Hz), 27.37; IR (KBr) ν 3683, 3583, 3020, 2400, 2361, 1744, 1522, 1421, 1216, 1016, 929, 770, 669 cm^{-1}. HRMS: calcd. for $C_{18}H_{21}ClF_3NO_4$: 407.1111, found. 407.1108 The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 4.91 and 5.72 min.



(3bh)

(2*R*,3*R*,4*R*,5S)-4-*tert*-butyl 2-methyl 5-(2-chlorophenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 104 ℃; $[\alpha]^{25}_D$ = -74.7 (*c* 0.61, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.51-7.24 (m, 4H), 4.79 (d, *J* = 7.2 Hz, 1H), 4.00 (d, *J* = 6.3 Hz, 1H), 3.85 (s, 3H), 3.66-3.64 (m, 2H), 0.98 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) δ 171.03, 169.72, 134.94, 133.90, 129.21, 128.97, 127.62, 126.79, 126.47 (q, J_{CF} = 277.1 Hz), 81.58, 62.53, 59.62, 52.82, 49.89 (q, J_{CF} = 25.8 Hz), 48.37, 27.28; IR (KBr) ν 3684, 3622, 3019, 2977, 2400, 1747, 1520, 1423, 1216, 1046, 929, 757, 669 cm^{-1}. HRMS: calcd. for $C_{18}H_{21}ClF_3NO_4$: 407.1111, found. 407.1110 The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.13 and 9.23 min.

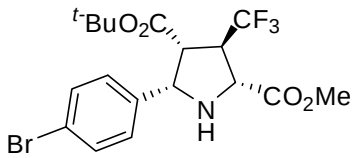


(3bi)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 5-(3-chlorophenyl)-3-(trifluoromethyl)

pyrrolidine-2,4-dicarboxylate

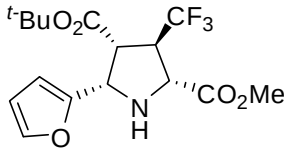
The title compound was prepared according to the general procedure as described above in 80% yield. m.p. 115 ℃; $[\alpha]^{25}_{D}$ = -68.0 (*c* 0.25, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.37-7.26 (m, 4H), 4.73 (d, *J* = 6.0 Hz, 1H), 4.25 (d, *J* = 6.3 Hz, 1H), 3.91 (s, 3H), 3.55-3.43 (m, 2H), 1.11 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 170.98, 169.58, 139.15, 134.26, 129.58, 127.97, 127.40, 125.26, 124.53, 82.05, 64.88, 60.31, 52.87, 51.05, 27.39; IR (KBr) ν 3615, 3584, 3019, 2965, 2400, 1747, 1520, 1422, 1216, 1046, 929, 772, 757, 669 cm^{-1}. HRMS: calcd. for $C_{18}H_{21}ClF_3NO_4$: 407.1111, found. 407.1114 The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.88 and 9.57min.



(3bj)

(2*R*,3*R*,4*R*,5S)-4-*tert*-butyl 2-methyl 5-(4-bromophenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

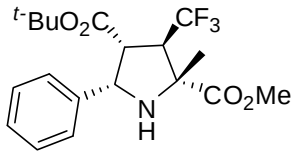
The title compound was prepared according to the general procedure as described above in 83% yield. m.p. 108 ℃; $[\alpha]^{25}_{D}$ = -32.9 (*c* 0.52, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.50-7.47 (m, 2H), 7.28-7.25 (m, 2H), 4.57 (d, *J* = 7.5 Hz, 1H), 4.06 (d, *J* = 5.7 Hz, 1H), 3.87 (s, 3H), 3.54 (m, 1H), 3.36-3.34 (m, 1H); 1.07 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) δ 171.01, 169.59, 136.05, 131.27, 128.82, 126.30 (q, J_{CF} = 276.0 Hz), 121.65, 81.99, 64.79, 60.27, 52.85, 51.04, 50.60 (q, J_{CF} = 27.0 Hz), 27.37; IR (KBr) ν 36845, 3622, 3019, 2977, 2400, 1745, 1521, 1423, 1216, 1046, 929, 773, 669 cm^{-1}. HRMS: calcd. for $C_{18}H_{21}BrF_3NO_4$: 451.0606, found. 451.0611 The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.07 and 6.02 min.



(3bk)

((2*R*,3*R*,4*R*,5*S*))-4-*tert*-butyl 2-methyl 5-(furan-2-yl)-3-(trifluoromethyl) pyrrolidine-2,4-di-carboxylate

The title compound was prepared according to the general procedure as described above in 90% yield. m.p. 76 ℃; $[\alpha]^{25}_{D}$ = +7.4 (*c* 0.61, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.33 (m, 1H), 6.32-6.26 (m, 2H), 4.64 (d, *J* = 7.2 Hz, 1H), 4.02 (d, *J* = 5.7 Hz, 1H), 3.80-3.75 (m, 4H), 3.41-3.37 (m, 1H), 1.24 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 171.73, 168.84, 151.51, 141.96, 126.51 (q, J_{CF} = 277.2 Hz), 110.35, 107.54, 81.88, 60.19, 59.26, 52.84, 50.34, 49.14 (q, J_{CF} = 26.9 Hz), 27.53; IR (KBr) ν 3684, 3621, 3019, 2977, 2400, 1742, 1522, 1423, 1217, 1046, 929, 770, 669 cm^{-1}. HRMS: calcd. for $C_{16}H_{20}F_3NO_5$: 363.1294, found. 363.1292. The product was analyzed by HPLC to determine the enantiomeric excess: 88% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.41 and 10.39 min.

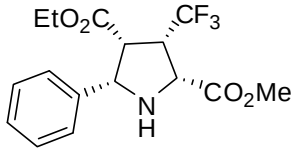


(3bl)

(2*R*,3*R*,4*R*,5*S*)-4-*tert*-butyl 2-methyl 2-methyl-5-phenyl-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 65% yield. m.p. 88 ℃; $[\alpha]^{25}_{D}$ = -22.2 (*c* 0.35, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.35-7.30 (m, 5H), 4.82 (d, *J* = 9.0 Hz, 1H), 3.92-3.86 (m, 4H), 3.51-3.46 (m, 1H), 1.60 (s, 3H), 0.99 (s, 9H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 173.57, 169.64, 137.90, 128.17, 127.93, 127.75, 126.08 (q, J_{CF} = 277.1 Hz), 81.40, 65.91, 62.66, 52.98, 52.73 (q, J_{CF} = 28.1 Hz), 51.08, 27.25, 19.79; IR (KBr) ν 3684,

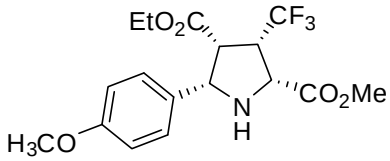
3621, 3019, 2977, 2400, 1731, 1522, 1423, 1216, 1046, 929, 770, 669 cm^{-1}. HRMS: calcd. for $C_{19}H_{24}F_3NO_4$: 387.1657, found. 387.1654 The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.66 and 7.00 min.



(4ca)

(2*R*,3*S*,4*R*,5*S*)- 4-ethyl 2-methyl 5-phenyl-3-(trifluoromethyl)pyrrolidine -2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 118 ℃; $[\alpha]^{25}_D$ = -44.3 (*c* 1.05, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.36-7.29 (m, 5H), 4.49 (d, *J* = 3.9 Hz, 1H), 4.32 (d, *J* = 9.6 Hz, 1H), 3.84- 3.80 (m, 5H), 3.74-3.69 (m, 1H), 3.56-3.52 (m, 1H), 3.25 (brs, 1H), 0.91 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 169.97, 168.82, 136.11, 128.36, 127.64, 126.18, 125.04 (q, J_{CF} = 277.2 Hz), 64.70, 60.68, 58.56, 52.55, 50.20, 13.51; IR (KBr) ν 3684, 3622, 3019, 2977, 2400, 1750, 1522, 1437, 1216, 1046, 929, 770, 669 cm^{-1}. HRMS: calcd. for $C_{16}H_{18}F_3NO_4$: 345.1188, found. 345.1192. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee 96:4 dr (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); t_r = 11.02 and 35.62 min.

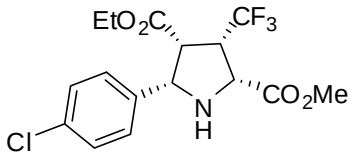


(4ce)

(2*R*,3*S*,4*R*,5*S*)-4-ethyl 2-methyl 5-(4-methoxyphenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

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

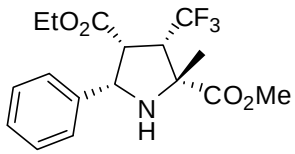
above in 82% yield. m.p. 120 ℃; $[\alpha]^{25}_{D}$ = -34.7 (*c* 0.56, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.29-7.26 (m, 2H), 6.89-6.86 (m, 2H), 4.42 (d, *J* = 4.2 Hz, 1H), 4.30 (d, *J* = 10.2 Hz, 1H), 3.88- 3.80 (m, 8H), 3.70-3.61 (m, 1H), 3.50-3.48 (m, 1H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 169.97, 168.89, 158.87, 128.11, 127.25, 125.03 (q, J_{CF} = 277.1 Hz) 113.59, 64.17, 60.50, 58.46, 55.04, 52.52 (q, J_{CF} = 28.1 Hz), 52.35, 50.18, 13.48; IR (KBr) ν 3684, 3621, 3019, 2976, 2400, 1744, 1518, 1424, 1216, 1045, 929, 758, 669 cm^{-1}. HRMS: calcd. for $C_{17}H_{20}F_3NO_5$: 375.1294, found. 375.1296. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee >98:2 dr (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.54 and 26.19 min.



(4cg)

(2*R*,3*S*,4*R*,5*S*)-4-ethyl 2-methyl 5-(4-chlorophenyl)-3-(trifluoromethyl)pyrrolidine -2,4-dicarboxylate

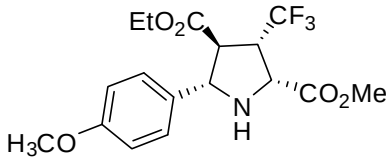
The title compound was prepared according to the general procedure as described above in 78% yield. m.p. 118 ℃; $[\alpha]^{25}_{D}$ = -36.0 (*c* 0.22, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.30-7.18 (m, 4H), 4.33 (d, *J* = 4.2 Hz, 1H), 4.19 (d, *J* = 9.9 Hz, 1H), 3.86-3.71 (m, 5H), 3.63-3.51(m, 1H), 3.47-3.41 (m, 1H), 2.96 (brs, 1H), 0.88 (d, *J* = 7.2Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 169.84, 168.71, 134.78, 133.57, 128.59, 127.69, 124.95 (q, J_{CF} = 277.1 Hz), 64.17, 60.93, 58.56, 52.72 (q, J_{CF} = 27.0 Hz), 49.95, 13.63; IR (KBr) ν 3681, 3583, 3019, 2400, 1710, 1523, 1421, 1216, 1018, 929, 773, 669 cm^{-1}. HRMS: calcd. for $C_{16}H_{17}ClF_3NO_4$: 379.0798, found. 379.0794. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee 96:4 dr (Chiralcel AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.30 and 17.09 min.



(4cl)

(2*R*,3*S*,4*R*,5*S*)-4-ethyl 2-methyl 2-methyl-5-phenyl-3-(trifluoromethyl)pyrrolidine -2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 83% yield. $[\alpha]^{25}_{D}$ = -59.0 (*c* 0.10, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.35-7.27 (m, 5H), 4.55 (d, *J* = 4.8 Hz, 1H), 3.84-3.78 (m, 5H), 3.54-3.50 (m, 1H), 3.23-3.17 (m, 1H), 1.69 (s, 3H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 172.46, 168.80, 136.27, 128.31, 127.58, 126.25, 125.18 (q, J_{CF} = 278.3 Hz), 65.37, 62.85, 60.69 (q, J_{CF} = 26.2 Hz), 52.75, 52.23, 29.61, 29.33, 13.52; IR (KBr) ν 3684, 3622, 3019, 2977, 2400, 1746, 1522, 1424, 1216, 1046, 929, 773, 669 cm^{-1}. HRMS: calcd. for $C_{17}H_{20}F_3NO_4$: 359.1344, found. 359.1342. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee >98:2 dr (Chiralcel AS-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.68 and 9.19 min.

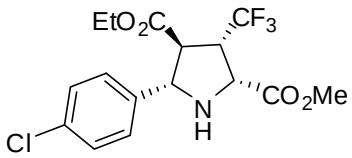


(5ce)

(2*R*,3*S*,4*S*,5*S*)-4-ethyl 2-methyl 5-(4-methoxyphenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 75% yield. $[\alpha]^{25}_{D}$ = +18.0 (*c* 0.14, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.37 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.4 Hz, 2H), 4.25-4.23 (m, 1H), 4.17-4.10 (m, 3H), 3.81(s, 3H), 3.80 (s, 3H), 3.69-3.60 (m, 1H), 3.16-3.10 (m, 1H), 2.50 (brs, 1H), 1.17 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 171.46, 170.48, 159.59, 130.74, 128.09, 125.88 (q, J_{CF} = 278.3 Hz), 114.11, 66.40, 61.38,

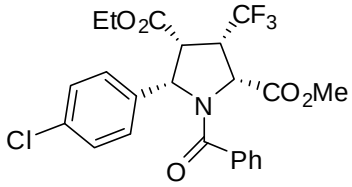
60.13, 55.16, 52.44, 51.02, 13.93; IR (KBr) ν 3682, 3583, 3020, 2400, 1735, 1518, 1476, 1425, 1216, 929, 758, 669 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee >98:2 dr (Chiralcel AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); t_r = 23.96 and 33.23 min.



(5cg)

(2*R*,3*S*,4*S*,5*S*)-4-ethyl 2-methyl 5-(4-chlorophenyl)-3-(trifluoromethyl)pyrrolidine -2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_D$ = +17.3 (*c* 1.00, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.42-7.33 (m, 4H), 4.29 (d, *J* = 9.0 Hz, 1H), 4.18-4.12 (m, 3H), 3.81(s, 3H), 3.69-3.60 (m, 1H), 3.14-3.09 (m, 1H), 2.49 (brs, 1H), 1.19 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 171.18, 170.41, 137.60, 134.15, 128.92, 128.37, 125.74 (q, J_{CF} = 278.3 Hz), 65.93, 61.61, 60.12, 52.66, 52.55, 50.95 (q, J_{CF} = 26.9 Hz), 13.99; IR (KBr) ν 3681, 3583, 3020, 2400, 2256, 1735, 1523, 1216, 1089, 909, 765, 669 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralcel AS-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); t_r = 13.84 and 18.97 min.

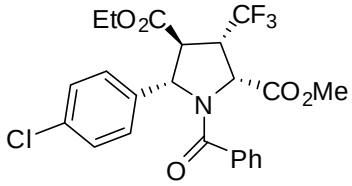


(6cg)

(2*R*,3*S*,4*R*,5*S*)-4-ethyl 2-methyl 1-benzoyl-5-(4-chlorophenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

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

above in 87% yield. m.p. 208 ℃; $[\alpha]^{25}_{D}$ = -32.5 (*c* 1.16, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.48-7.24 (m, 9H), 5.61 (m, 1H), 4.82 (m, 1H), 3.87 (s, 3H), 3.75-3.54 (m, 3H), 3.40 (m, 1H), 0.83 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 171.31, 168.39, 167.55, 135.51, 135.21, 133.43, 130.26, 128.66, 128.34, 128.07, 126.47, 123.45 (q, J_{CF} = 277.1 Hz) , 64.24, 61.05, 60.29, 52.43, 48.33, 31.16, 13.35; IR (KBr) ν 3683, 3583, 3020, 2400, 1751, 1655, 1523, 1216, 1017, 929, 747, 669 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralcel AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 22.67 and 69.76 min.



(7cg)

(2*R*,3*S*,4*S*,5*S*)-4-ethyl 2-methyl 1-benzoyl-5-(4-chlorophenyl)-3-(trifluoromethyl) pyrrolidine-2,4-dicarboxylate

The title compound was prepared according to the general procedure as described above in 88% yield. m.p. 160 ℃; $[\alpha]^{25}_{D}$ = -15.0 (*c* 0.20, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.14 (m, 9H), 5.08 (m, 2H), 4.14-4.10 (m, 2H), 3.87 (s, 3H), 3.64-3.49 (m, 2H), 1.13 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 150 MHz) 170.06, 169.41, 136.92, 134.97, 133.68, 129.92, 128.51, 127.98, 126.45, 123.87(q, J_{CF} = 277.2 Hz), 67.24, 61.91, 59.35, 52.88, 50.17, 47.15, 13.77; IR (KBr) ν 3684, 3586, 3019, 2400, 1724, 1650, 1525, 1216, 1019, 929, 747, 669 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); t_r = 16.50 and 26.73 min.

X-ray crystal structures of (2*R*,3*S*,4*R*,5*S*)-6cg and (2*R*,3*S*,4*S*,5*S*)-7cg

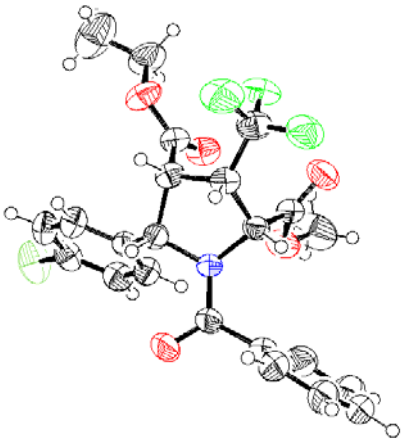


Figure 1. X-ray crystal of (2*R*,3*S*,4*R*,5*S*)-**6cg**.

For (2*R*,3*S*,4*R*,5*S*)-**6cg**: CCDC 827404, $C_{23}H_{21}ClF_3NO_5$, M_r = 483.86, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 8.6745(12), b = 13.3919(18), c = 19.262(3) Å, V = 2237.6(5) Å^3, Z = 4,, 12954 reflections measured, 3670 unique (R_{int} = 0.0353) which were used in all caclculations. The final wR_2 = 0.0845(all data), Flack χ = 0.08(7).

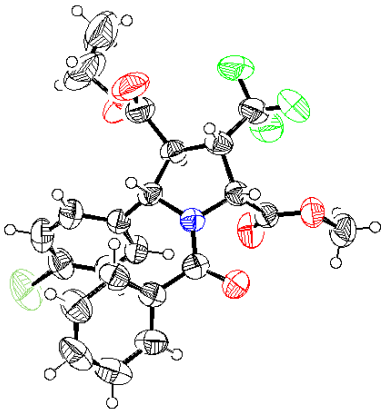


Figure 2. X-ray crystal of (2*R*,3*S*,4*R*,5*S*)-**7cg**.

For (2*R*,3*S*,4*S*,5*S*)-**7cg**: CCDC 827405, $C_{23}H_{21}ClF_3NO_5$, M_r = 483.86, T = 293 K, Orthorhombic, space group $P2_12_12_1$, a = 14.7950(17), b = 15.6582(17), c = 20.390(2) Å, V = 4723.6(9) Å^3, Z = 8, 26602 reflections measured, 6467 unique (R_{int} = 0.0422) which were used in all caclculations. The final wR_2 = 0.0945 (all data), Flack χ = -0.02(6).

Proposed transition states of the *endo*-selectivity for asymmetric 1,3-dipolar cycloaddition of azomethine ylides with *trans* or *cis*-4,4,4-Trifluorocrotonate

Based on the relative and absolute configuration of the cycloadducts, the high *endo*-selectivity observed in the $Cu(CH_3CN)_4BF_4$/TF-BiphamPhos catalyzed asymmetric 1,3-dipolar cycloaddition reaction of azomethine ylide with *trans*- or *cis*-4,4,4-trifluorocrotonate can be rationalized by the proposed tetracoordinated complex[3] shown in Figure 3. The *in situ*-formed azomethine ylide is coordinated to the metallic center and oriented in such transition state because of the steric repulsion between the phenyl group in the ylide and the phenyl ring on the phosphorus atom of the chiral ligand. The highly steric congestion imposed by the latter effectively blocks the dipolarophiles (*trans*- or *cis*-4,4,4-trifluorocrotonate) approach from the *Re* (C=N) face of the azomethine ylide and forms the corresponding *endo*-(2*R*,3*R*,4*R*,5*S*) or *endo*-(2*R*,3*S*,4*R*,5*S*) adduct through *Si* face attack. The carbonyl group of the *trans*- or *cis*-4,4,4-trifluorocrotonate could coordinate with the Cu(I) 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 group of dipolarophile **1a** and the NH_2 group of the chiral (*S*)-TF-BiphamPhos ligand (**L1**), which also facilitates stabilizing the proposed transition states.[3b,3c]

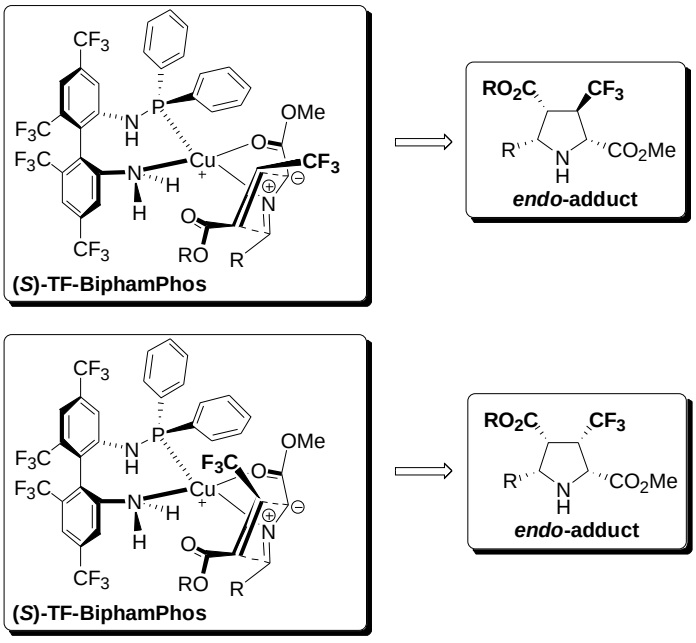


Figure 3. Proposed transition states leading to *endo*-adducts.

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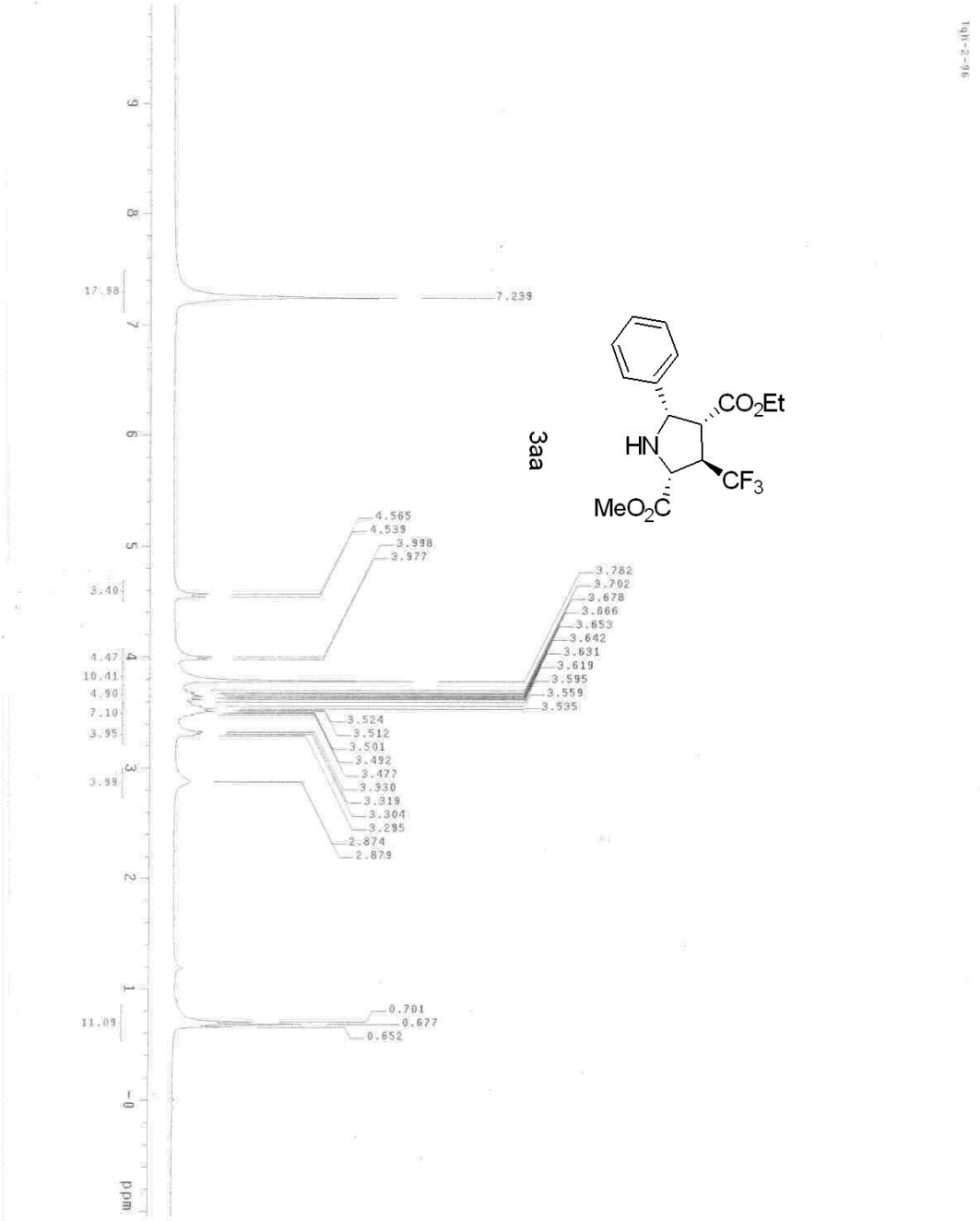
2. a) B.-L. Wang, F. Yu, X.-L. Qiu, Z. Jiang, F.-L. Qing, *J. Fluorine Chem*. **2006**, *127*, 580; b) E. T. Mcbee, M. J. Keogh, R. P. Levek, E. P. Wesseler, *J. Org. Chem.*, **1973**, *38*, 632; c) S. K. Guha, A. Shibayama, D. Abe, M. Sakaguchi, Y. Ukaji, K. Inomata, *Bull. Chem. Soc. Jpn.*, **2004**, *77*, 2147; d) P. F. Bevilacqua, D. D. Keith, J. L. Roberts, *J. Org. Chem.*, **1984**, *49*, 1430; e) B. C. Hamper, *Org. Synth.*, **1998**, *49*, 1430; f) J. Leroy, N. Fischer, C. Wakselman, *J. Chem. Soc. Perkin Trans. 1* **1990**, 1281.

3. a) S. Cabrera, R. G. Arrayás, B. Martín-Matute, F. P. Cossío, J. C. Carretero, *Tetrahedron* **2007**, *63*, 6587; b) C. Nájera, M. D. G. Retamosa, J. Sansano, *Angew. Chem., Int. Ed.* **2008**, *47*, 6055; c) X.-X. Yan, Q. Peng, Y. Zhang, K. Zhang, W. Hong, X.-L. Hou, Y.-D. Wu, *Angew. Chem., Int. Ed.* **2006**, *45*, 1979.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3.319
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0.652
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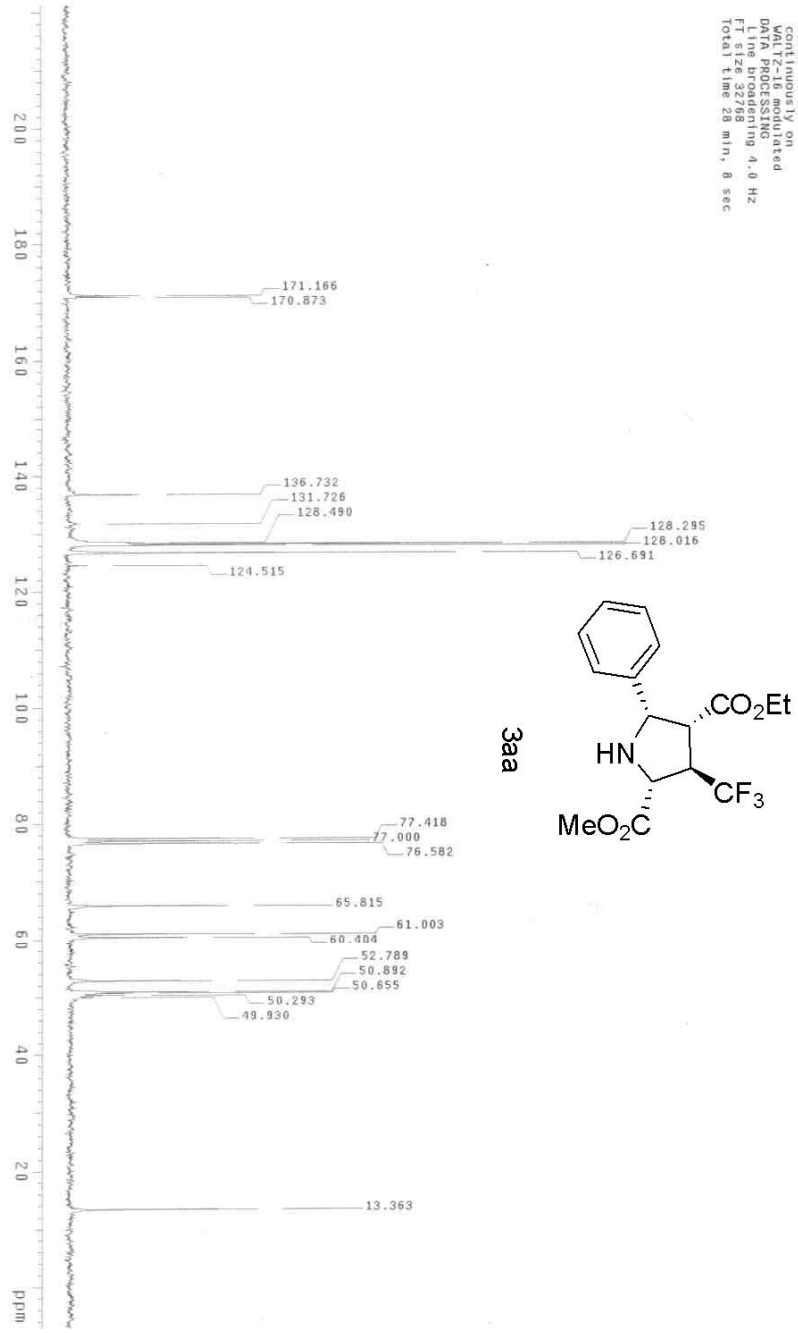
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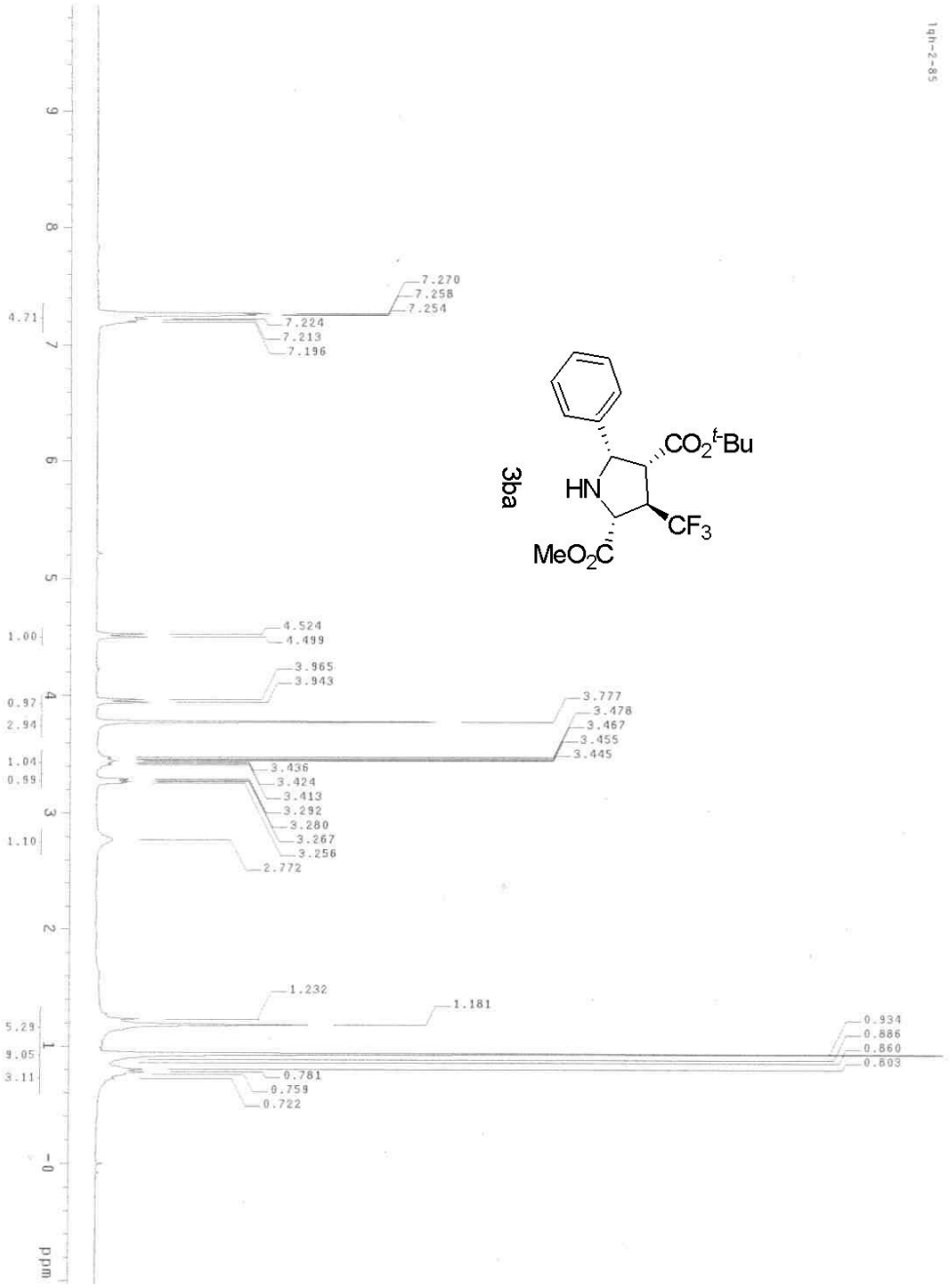
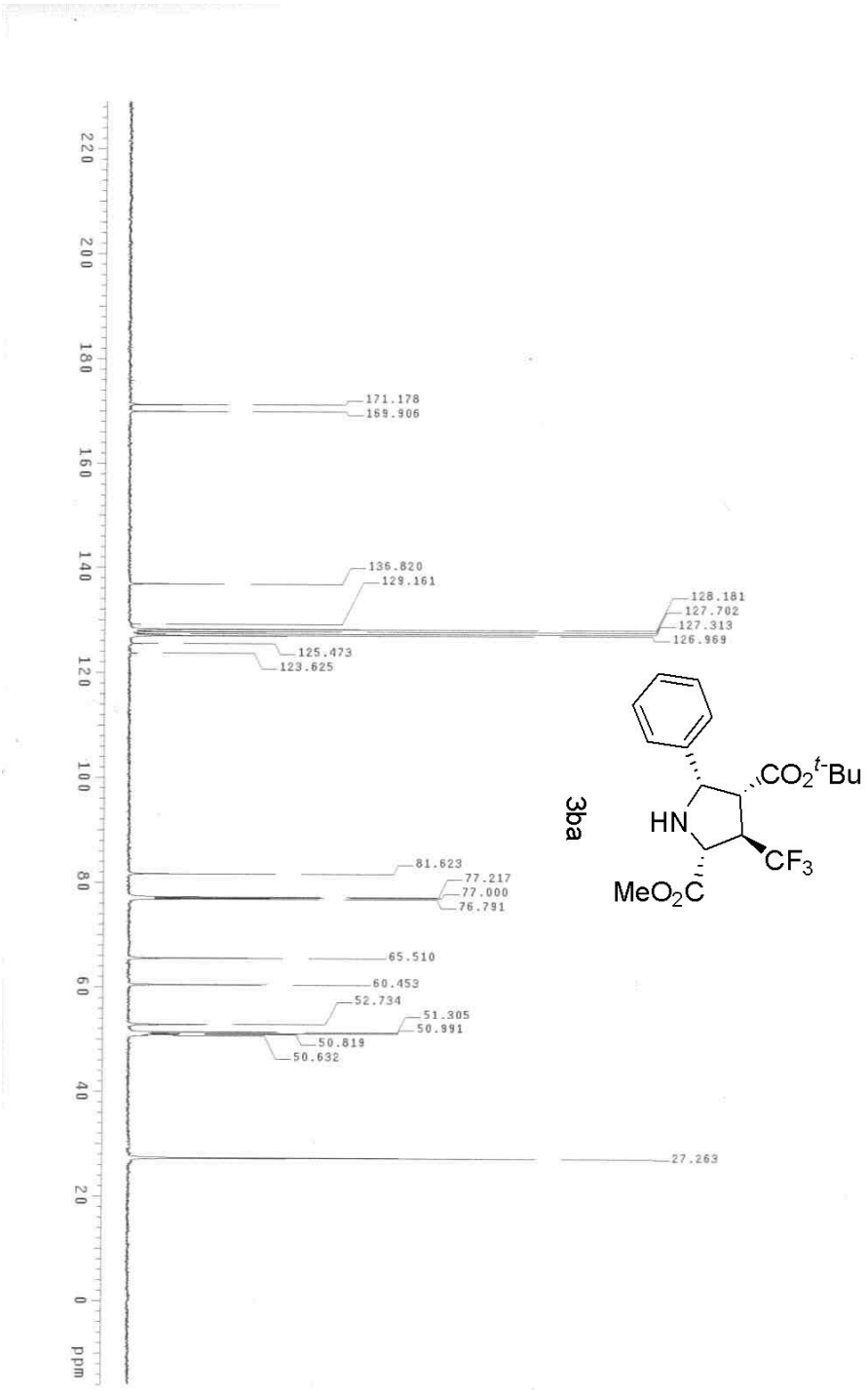
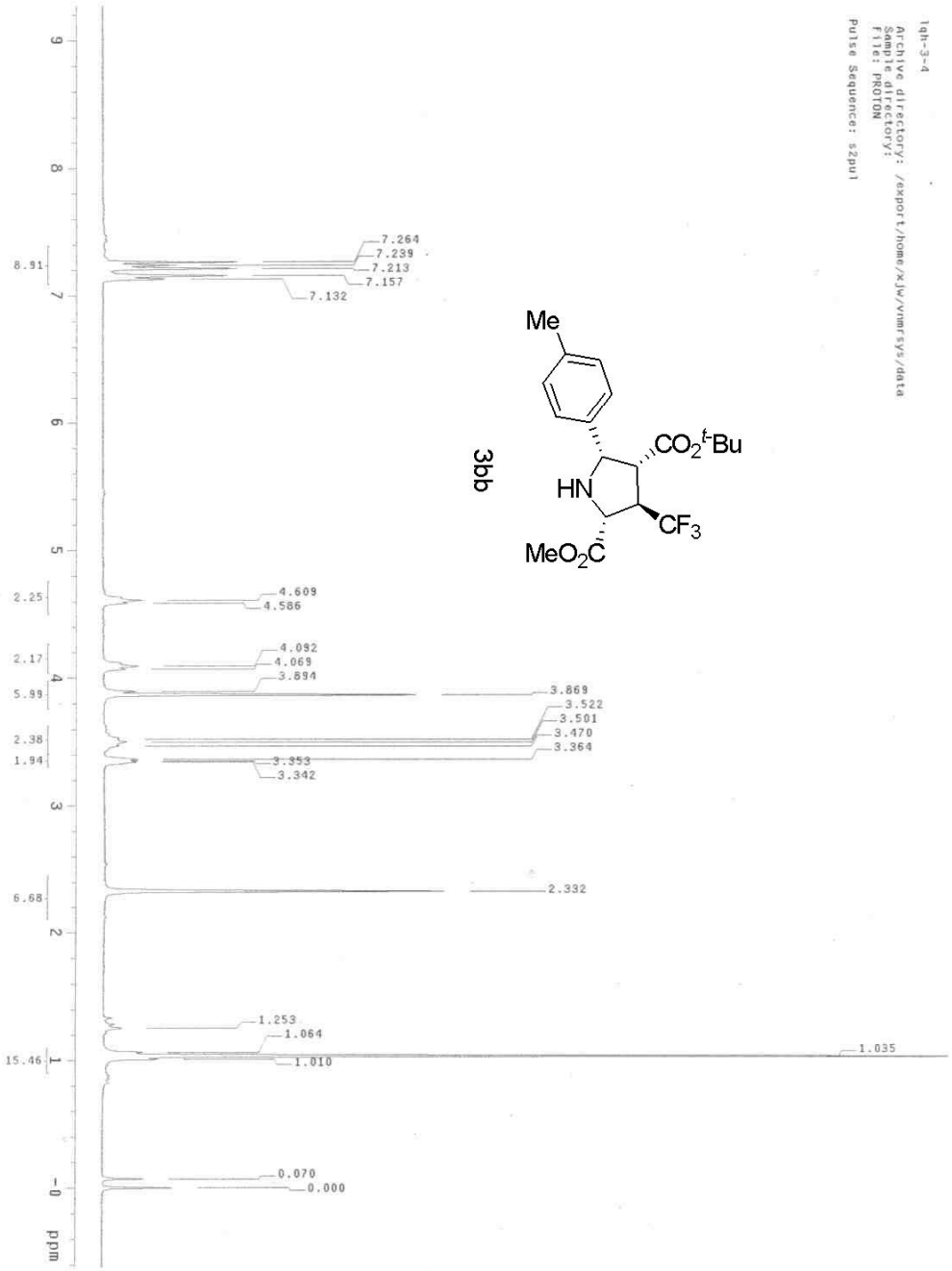
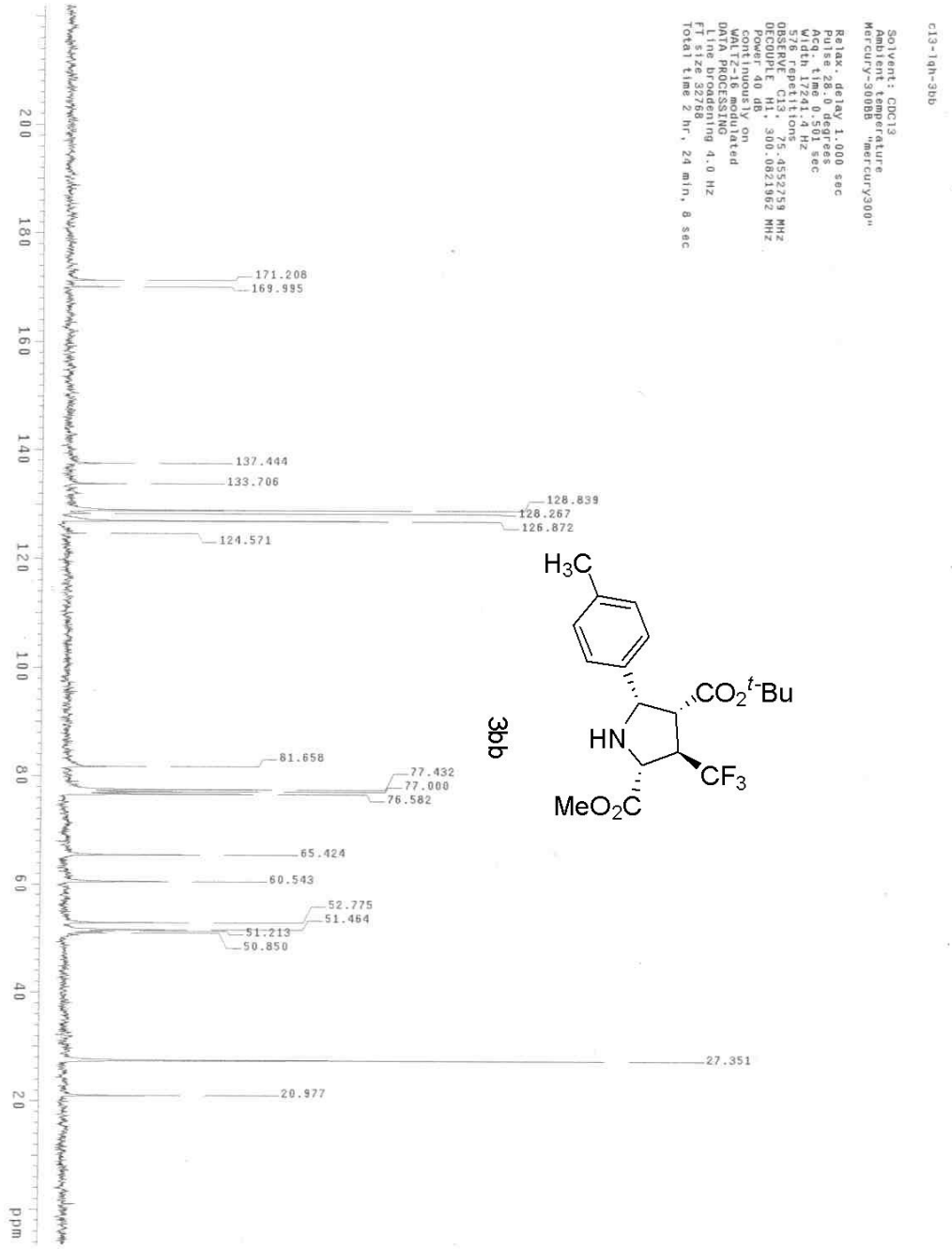
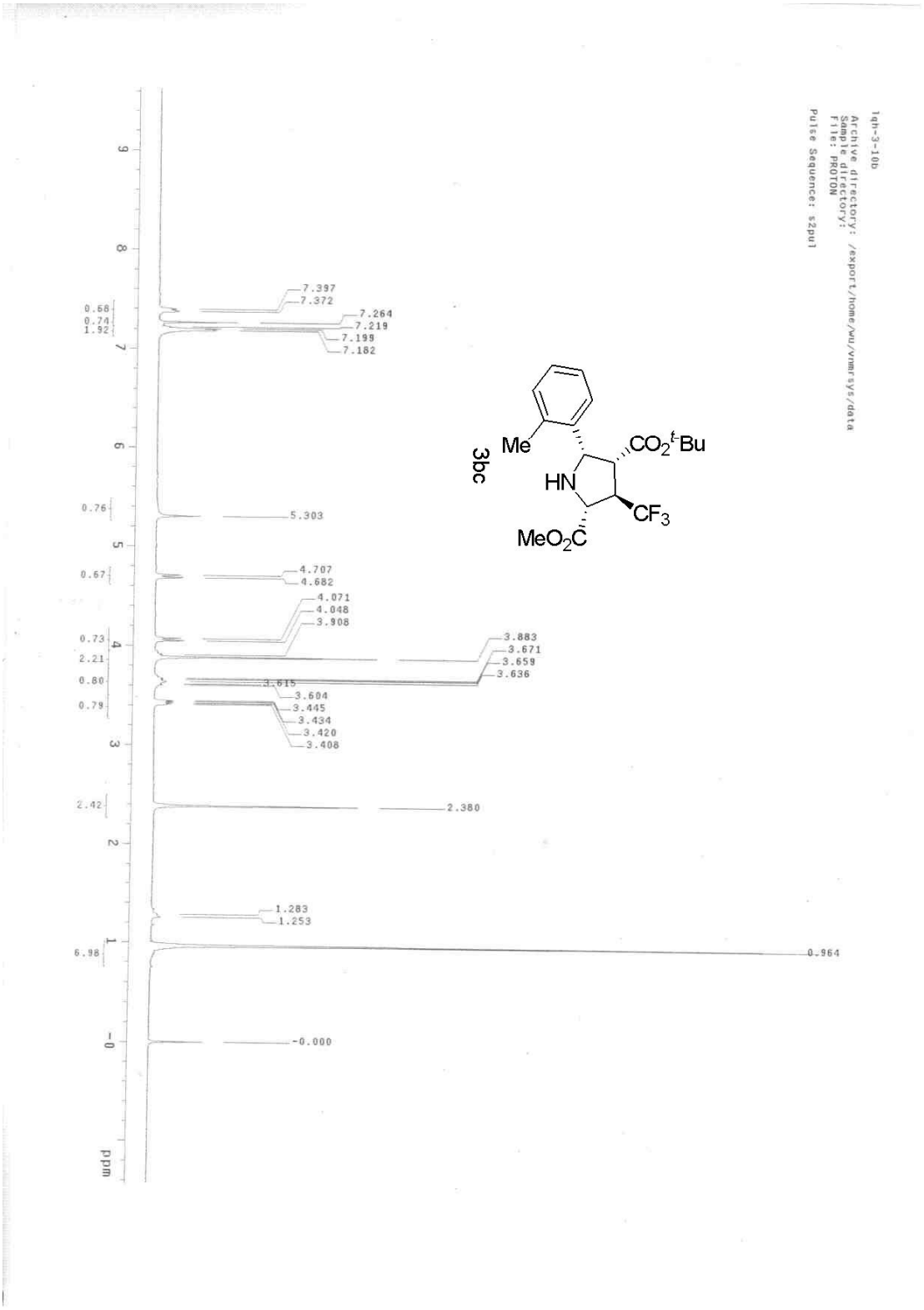
Acq. time 0.500 sec
Width 17241.4 Hz
1000 repetitions
OBSERVE C13, 75.453222 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 8 sec
171.166
170.873
136.732
131.726
128.490
128.295
128.016
126.691
124.515
77.418
77.000
76.582
65.815
61.003
60.404
52.789
50.892
50.655
50.293
49.930
13.363
200
180
160
140
120
100
80
60
40
20
ppm
3aa
CO2Et
HN
CF3
MeO2C

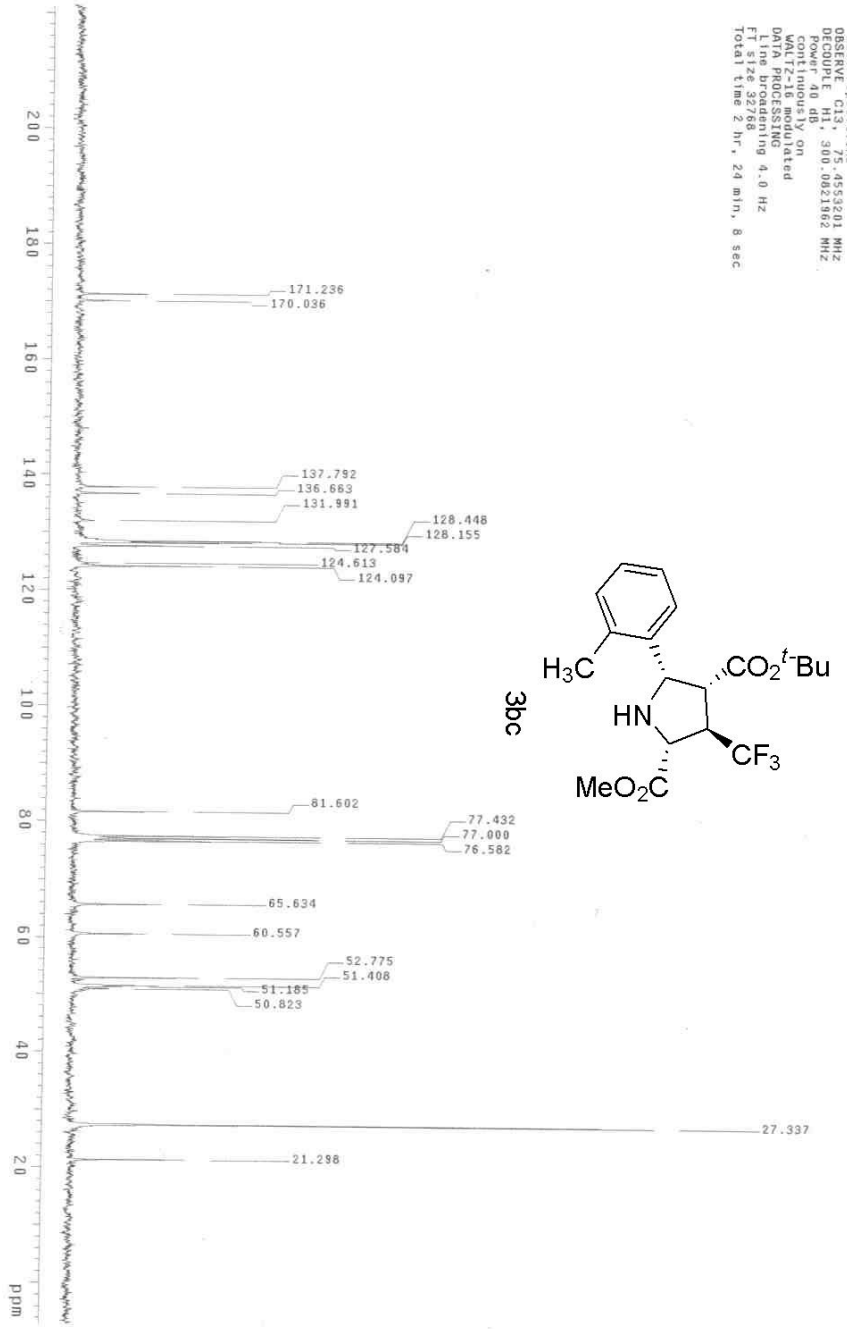

7.270
7.258
7.254
7.224
7.213
7.196
4.524
4.499
3.965
3.943
3.777
3.478
3.467
3.455
3.445
3.436
3.424
3.413
3.292
3.280
3.267
3.256
2.772
1.232
1.181
0.934
0.886
0.860
0.803
0.781
0.759
0.722
4.71
1.00
0.92
2.94
1.04
0.99
1.10
5.29
9.05
3.11
ppm
3ba
HN
CO2tBu
CF3
MeO2C

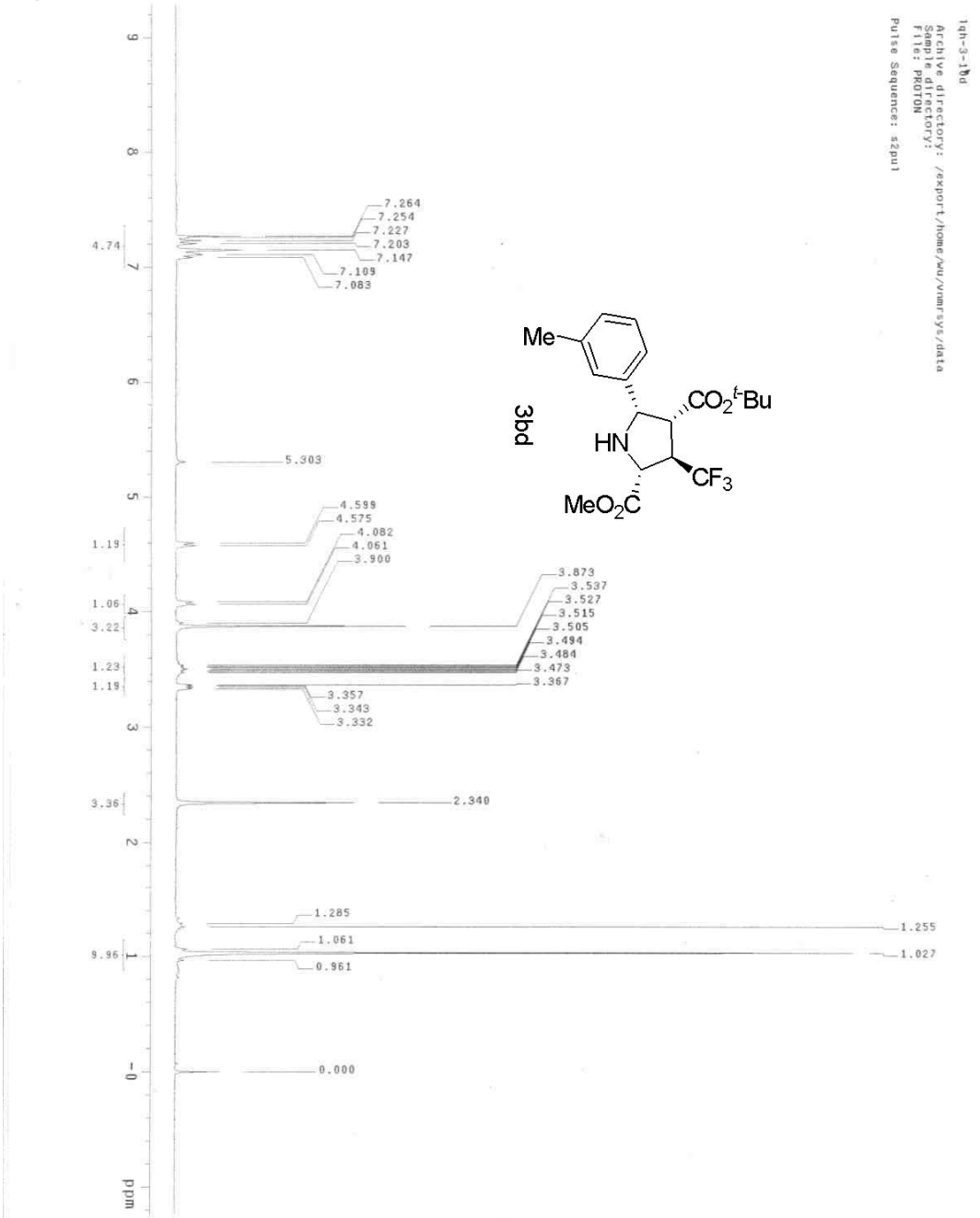

c13-1qh-3ba
171.178
169.906
136.820
129.161
128.181
127.702
127.313
126.969
125.473
123.625
81.623
77.217
77.000
76.791
65.510
60.453
52.734
51.305
50.991
50.819
50.632
27.263
220
200
180
160
140
120
100
80
60
40
20
0
ppm
3ba
$CO_2{}^tBu$
HN
CF_3
MeO_2C

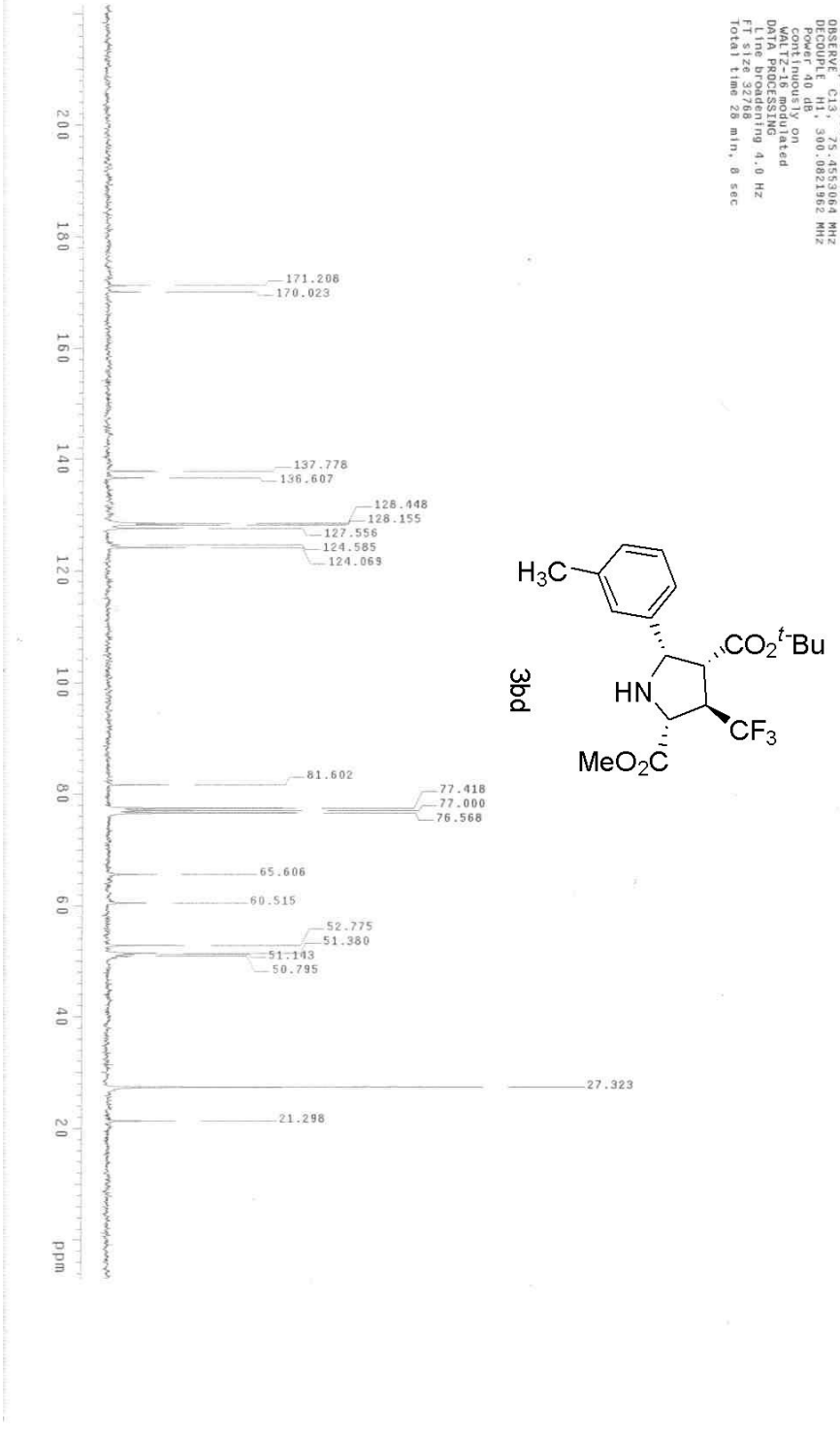

lqh-3-4
Archive directory: /export/home/xjw/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.264
7.239
7.213
7.157
7.132
8.91
4.609
4.586
2.25
4.092
4.069
3.894
2.17
3.869
3.522
3.501
3.470
3.364
5.99
2.38
1.94
3.353
3.342
2.332
6.68
1.253
1.064
1.035
1.010
15.46
0.070
0.000
ppm
Me
CO2tBu
HN
CF3
MeO2C
3bb

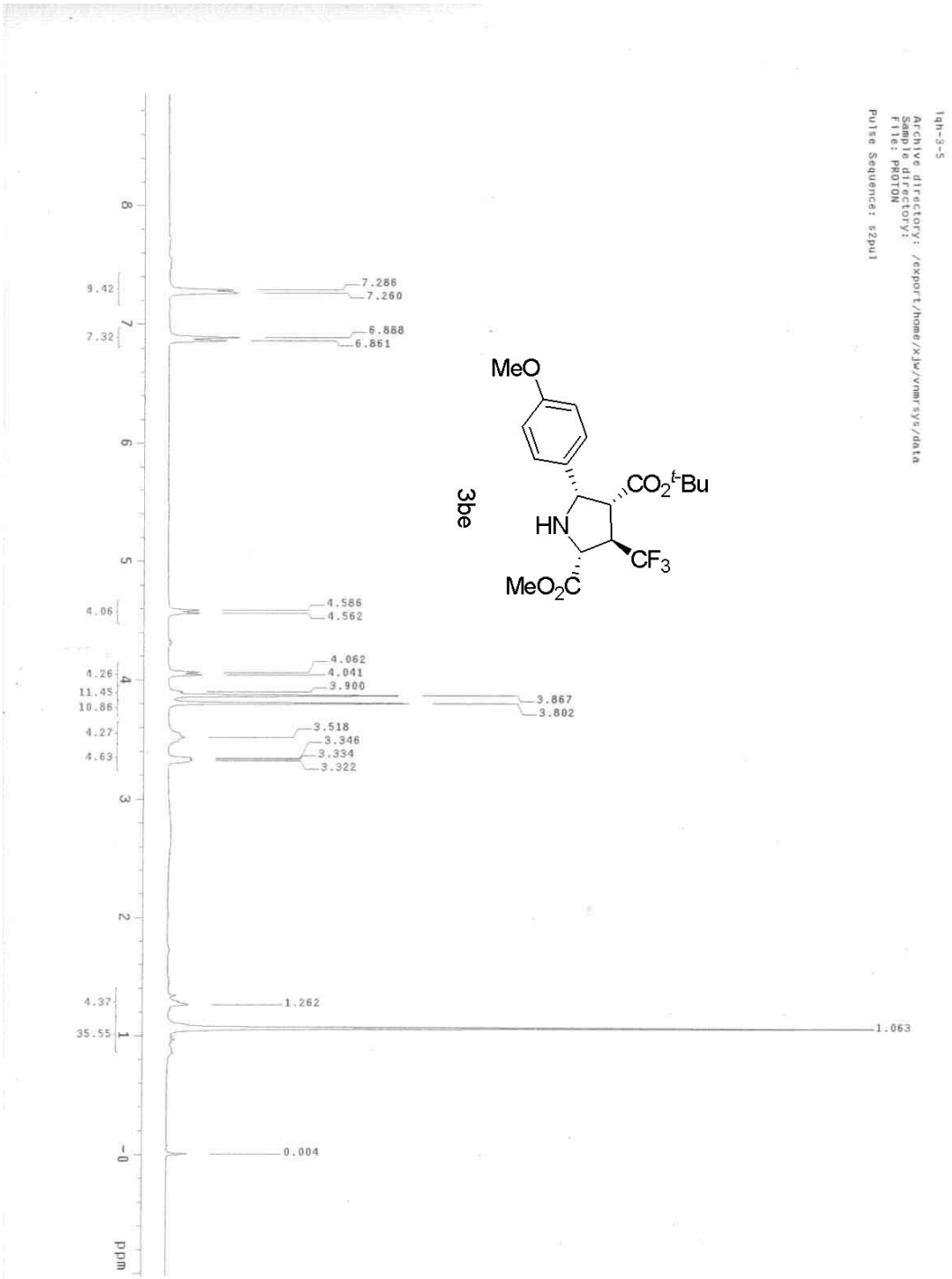

c13-lqh-3bb
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.501 sec
Width 17241.4 Hz
576 repetitions
OBSERVE C13, 75.4552259 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 8 sec
171.208
169.995
137.444
133.706
128.839
128.267
126.872
124.571
81.658
77.432
77.000
76.582
65.424
60.543
52.775
51.464
51.213
50.850
27.351
20.977
ppm
3bb
H3C
CO2t-Bu
HN
CF3
MeO2C

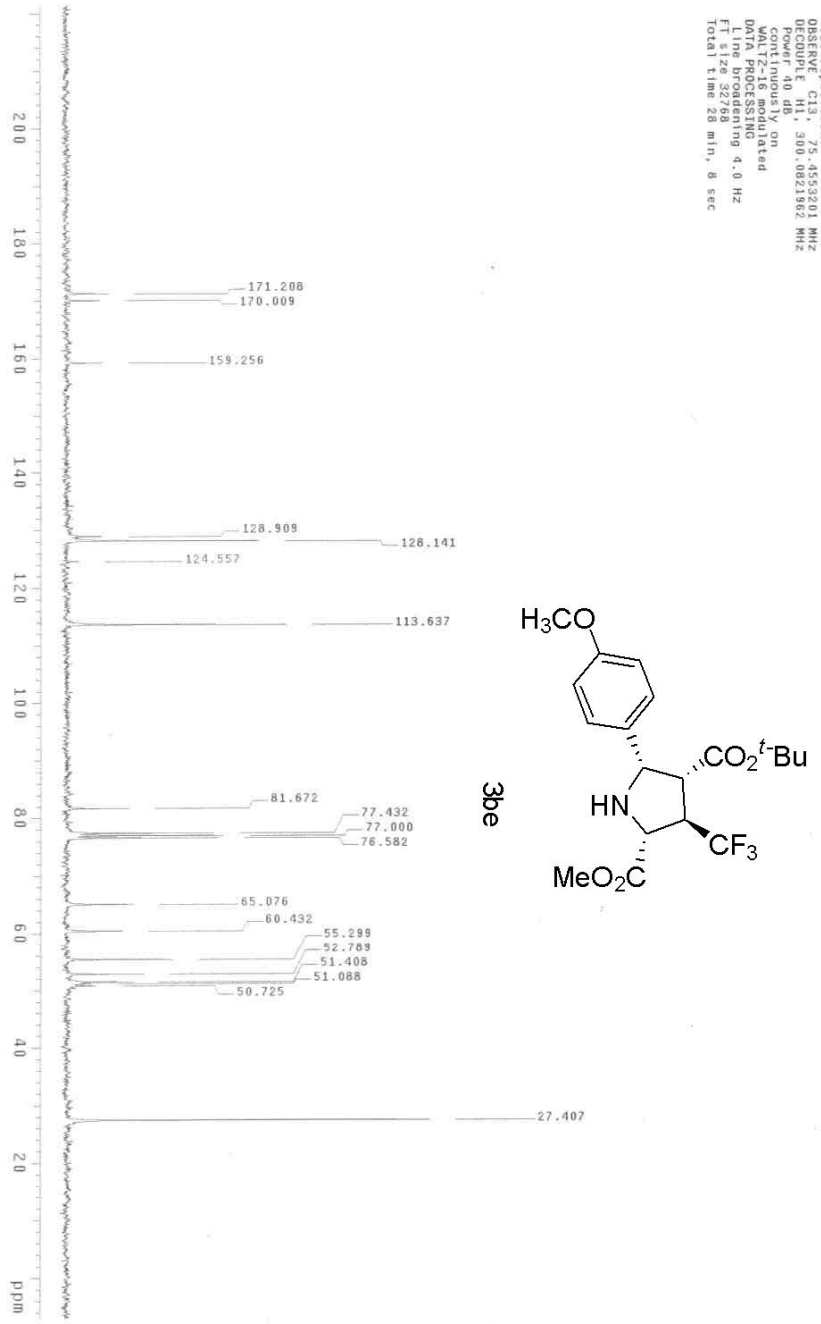

lqh-3-10b
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.397
7.372
7.264
7.219
7.199
7.182
5.303
4.707
4.682
4.071
4.048
3.908
3.883
3.671
3.659
3.636
3.615
3.604
3.445
3.434
3.420
3.408
2.380
1.283
1.253
0.964
-0.000
0.68
0.74
1.92
0.76
0.67
0.73
2.21
0.80
0.79
2.42
6.98
ppm
Me
CO2tBu
HN
CF3
MeO2C
3bc

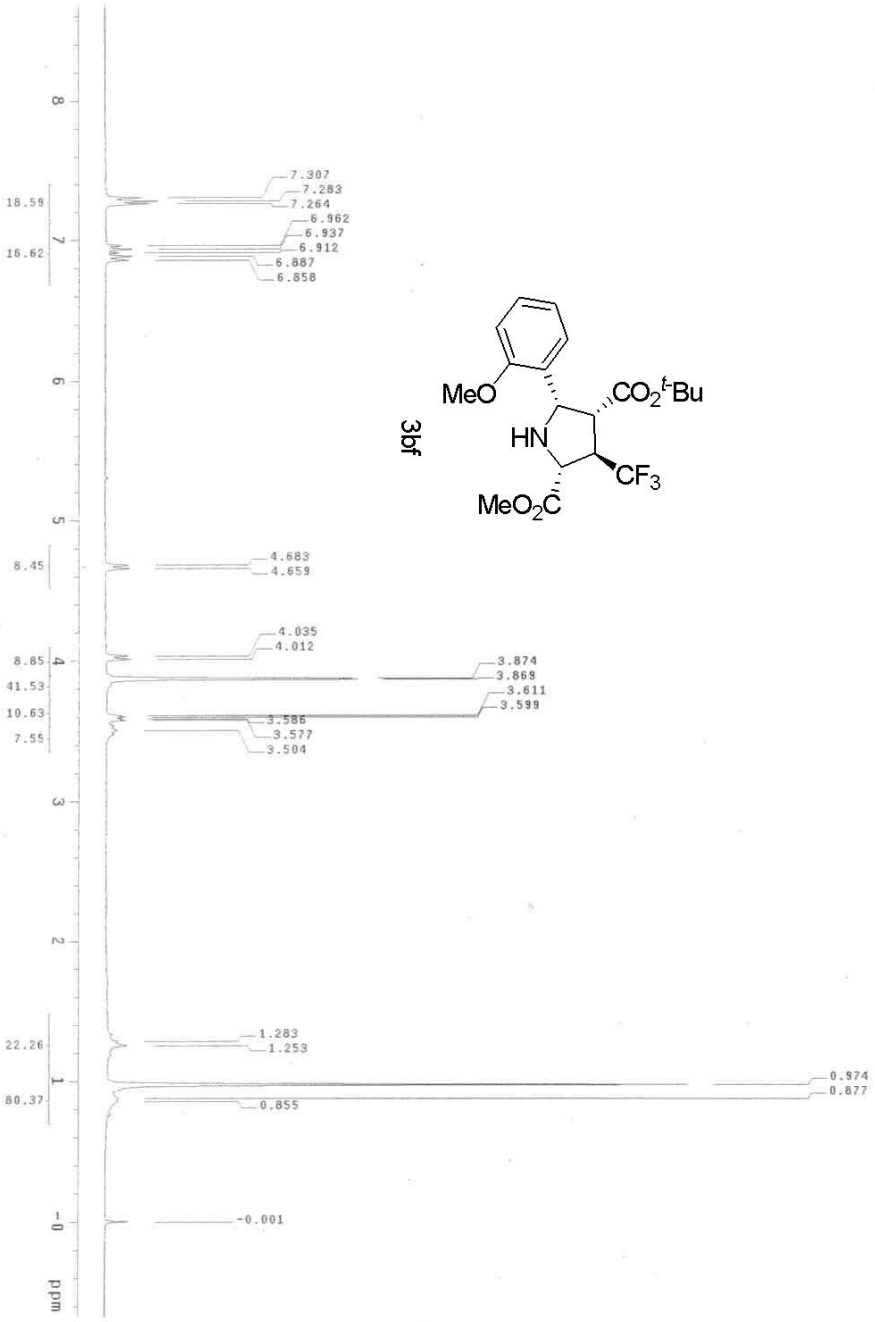

c13-1qh-3bc
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.501 sec
Width 17241.4 Hz
1152 repetitions
OBSERVE C13, 75.4553201 MHz
DECOUPLE H1, 300.0021962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 8 sec
171.236
170.036
137.792
136.663
131.991
128.448
128.155
127.584
124.613
124.097
81.602
77.432
77.000
76.582
65.634
60.557
52.775
51.408
51.185
50.823
27.337
21.298
200
180
160
140
120
100
80
60
40
20
ppm
3bc
H3C
CO2t-Bu
HN
CF3
MeO2C

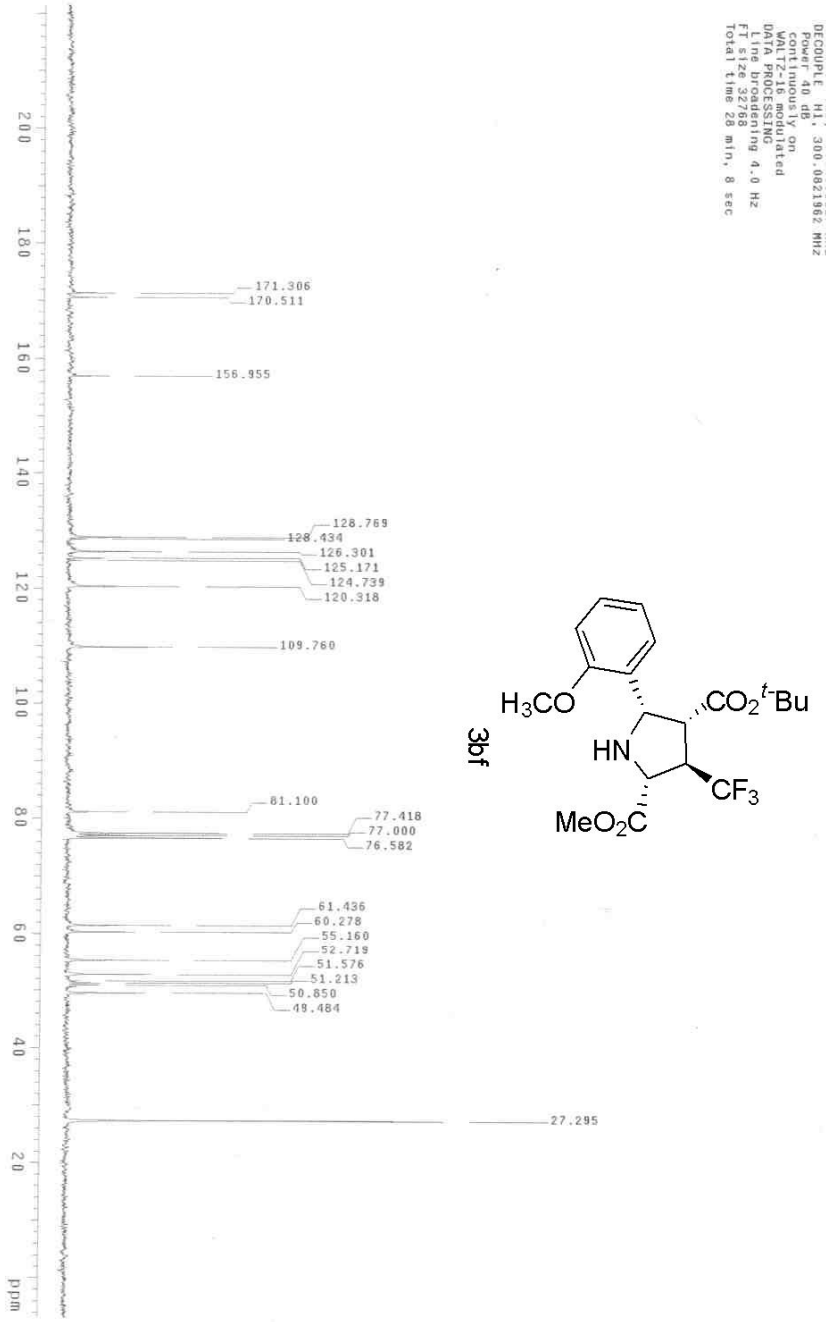

1qh-3-1bd
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
3bd
Me
CO2tBu
HN
CF3
MeO2C
7.264
7.254
7.227
7.203
7.147
7.109
7.083
5.303
4.599
4.575
4.082
4.061
3.900
3.873
3.537
3.527
3.515
3.505
3.494
3.484
3.473
3.367
3.357
3.343
3.332
2.340
1.285
1.255
1.061
1.027
0.961
0.000
4.74
1.19
1.06
3.22
1.23
1.19
3.36
9.96
ppm

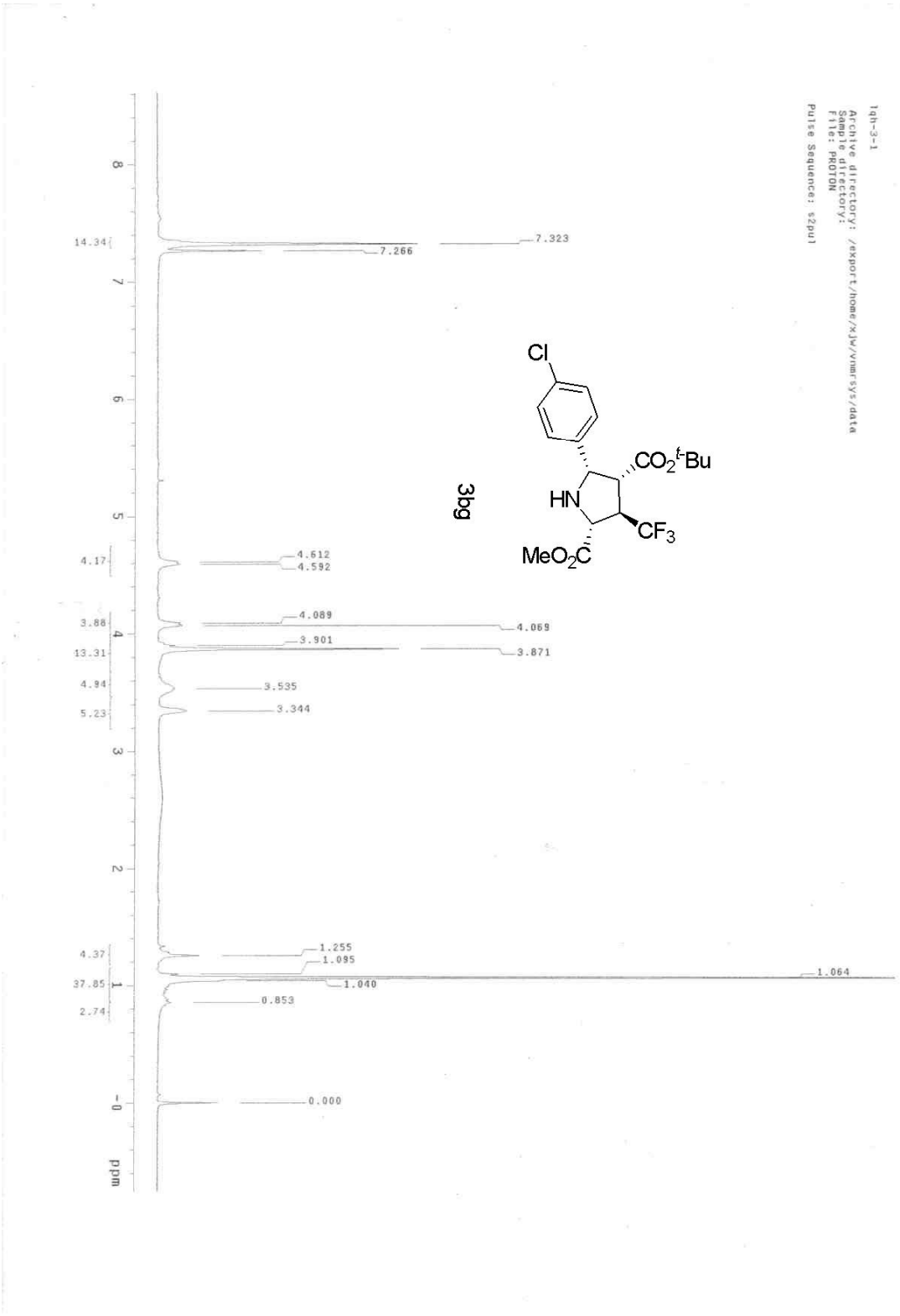

1qh-3bd
Solvent: CDCl3
Ambient temperature
File: 1qh-3bd
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 12019.2 Hz
688 repetitions
OBSERVE C13, 75.453064 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 20 min, 8 sec
171.208
170.023
137.778
136.607
128.448
128.155
127.556
124.585
124.069
81.602
77.418
77.000
76.568
65.606
60.515
52.775
51.380
51.143
50.795
27.323
21.298
200
180
160
140
120
100
80
60
40
20
ppm
3bd
H3C
CO2t-Bu
HN
CF3
MeO2C

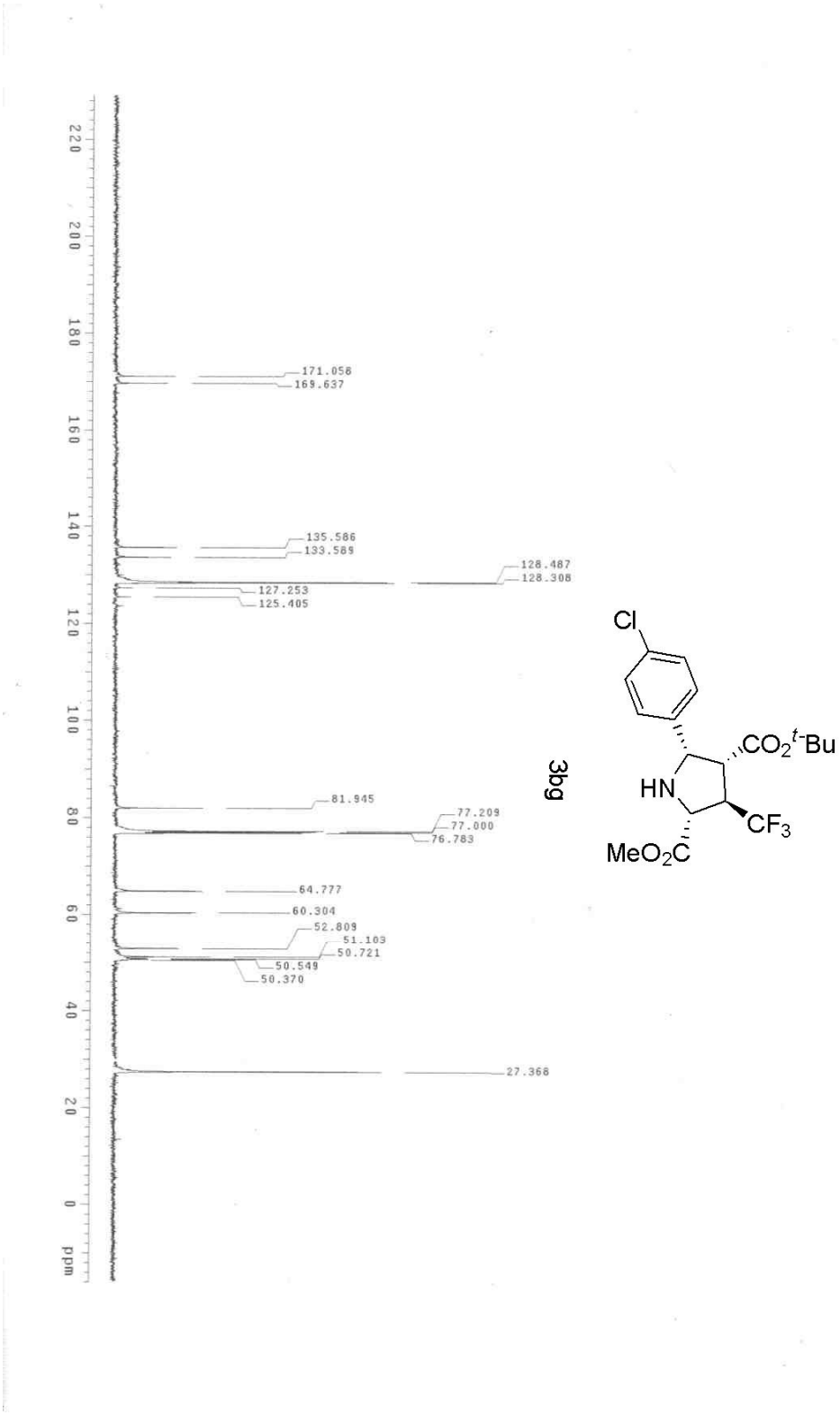

lqh-3-5
Archive directory: /export/home/xjw/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
MeO
$CO_2{}^tBu$
HN
CF_3
MeO_2C
3be
9.42
7.32
4.06
4.26
11.45
10.86
4.27
4.63
4.37
35.55
7.286
7.260
6.888
6.861
4.586
4.562
4.062
4.041
3.900
3.867
3.802
3.518
3.346
3.334
3.322
1.262
1.063
0.004
ppm

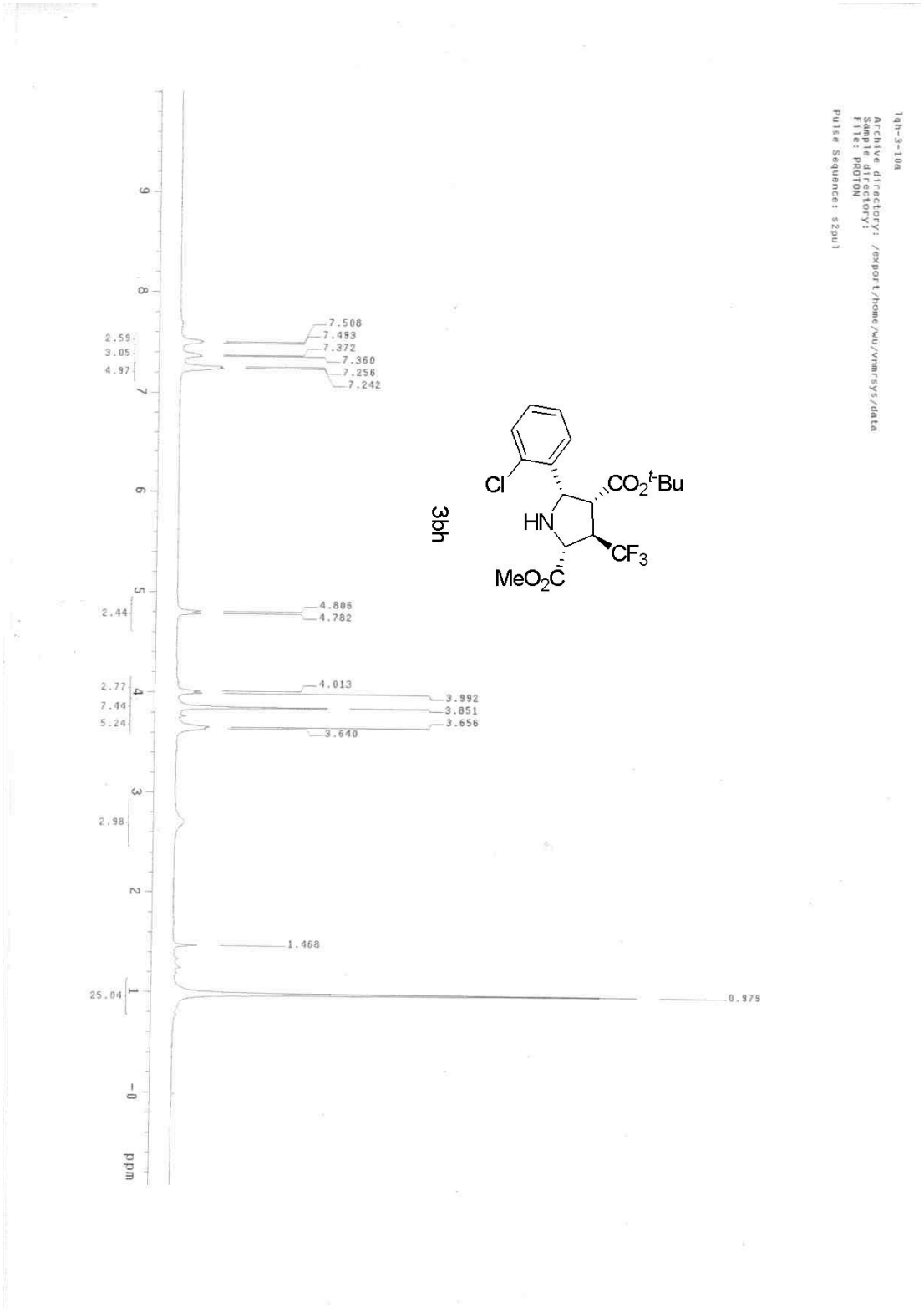

lqh-3be
Solvent: CDCl3
Ambient temperature
File: lqh-3be
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17241.4 Hz
520 repetitions
OBSERVE C13, 75.455201 MHz
DECOUPLE H1, 300.082962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 8 sec
171.208
170.009
159.256
128.909
128.141
124.557
113.637
81.672
77.432
77.000
76.582
65.076
60.432
55.299
52.789
51.408
51.088
50.725
27.407
200
180
160
140
120
100
80
60
40
20
ppm
3be
H3CO
CO2t-Bu
HN
CF3
MeO2C

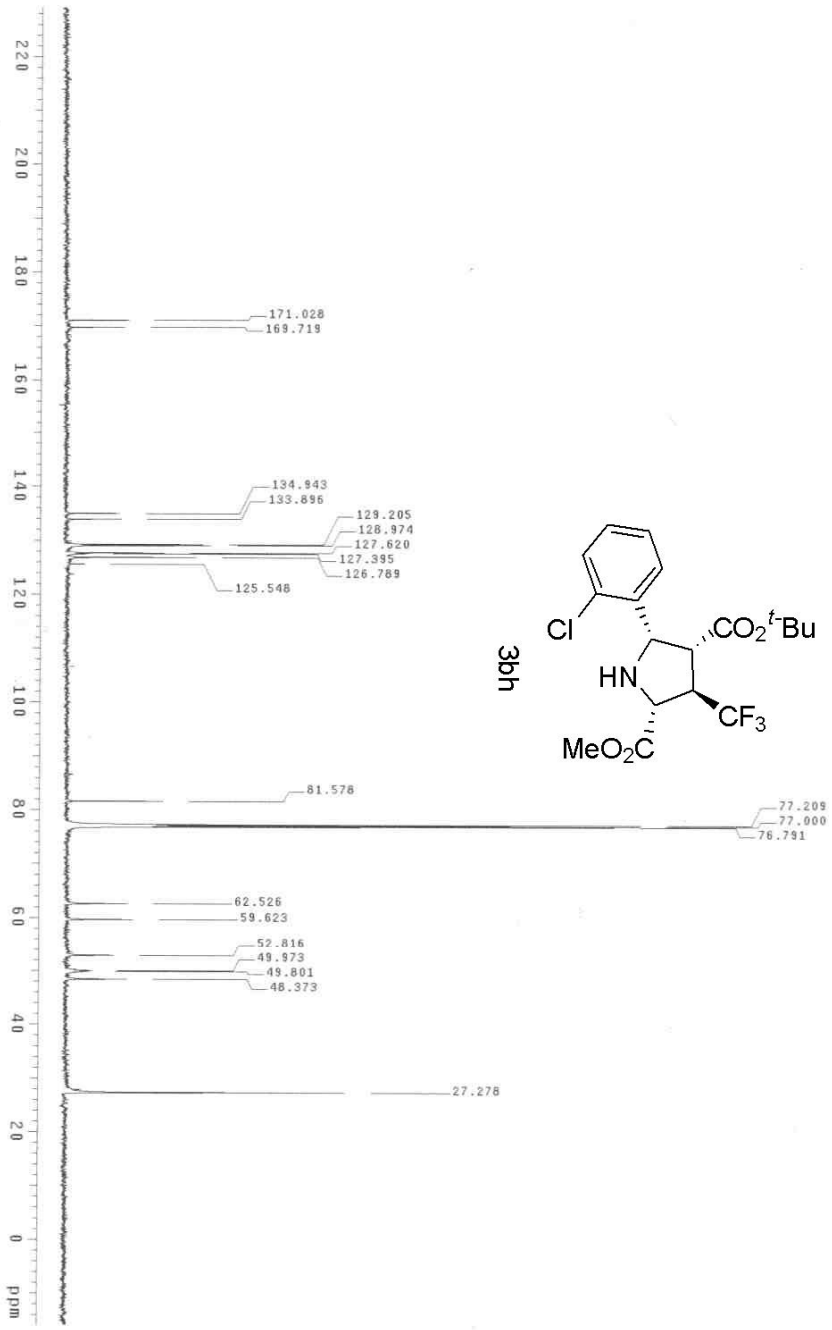

lqh-3-12a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.307
7.283
7.264
6.962
6.937
6.912
6.887
6.858
4.683
4.659
4.035
4.012
3.874
3.869
3.611
3.599
3.586
3.577
3.504
1.283
1.253
0.974
0.877
0.855
-0.001
18.59
16.62
8.45
8.85
41.53
10.63
7.55
22.26
80.37
MeO
$CO_2{}^tBu$
HN
CF_3
MeO_2C
3bf
ppm

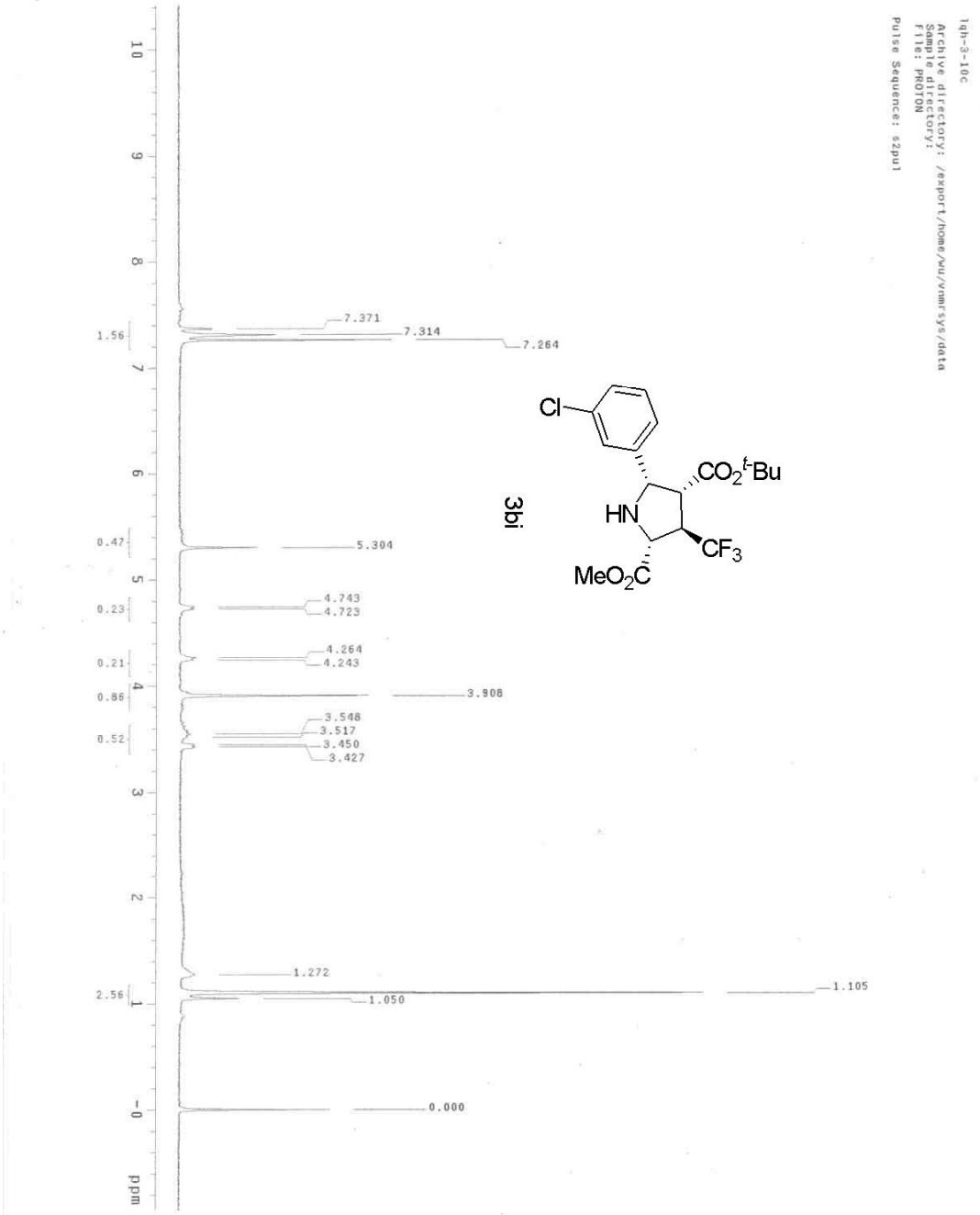

lqh-3bf
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17241.4 Hz
616 repetitions
OBSERVE C13, 75.4553117 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 8 sec
171.306
170.511
156.955
128.769
128.434
126.301
125.171
124.739
120.318
109.760
81.100
77.418
77.000
76.582
61.436
60.278
55.160
52.719
51.576
51.213
50.850
49.484
27.295
200
180
160
140
120
100
80
60
40
20
ppm
3bf
H3CO
CO2tBu
HN
CF3
MeO2C

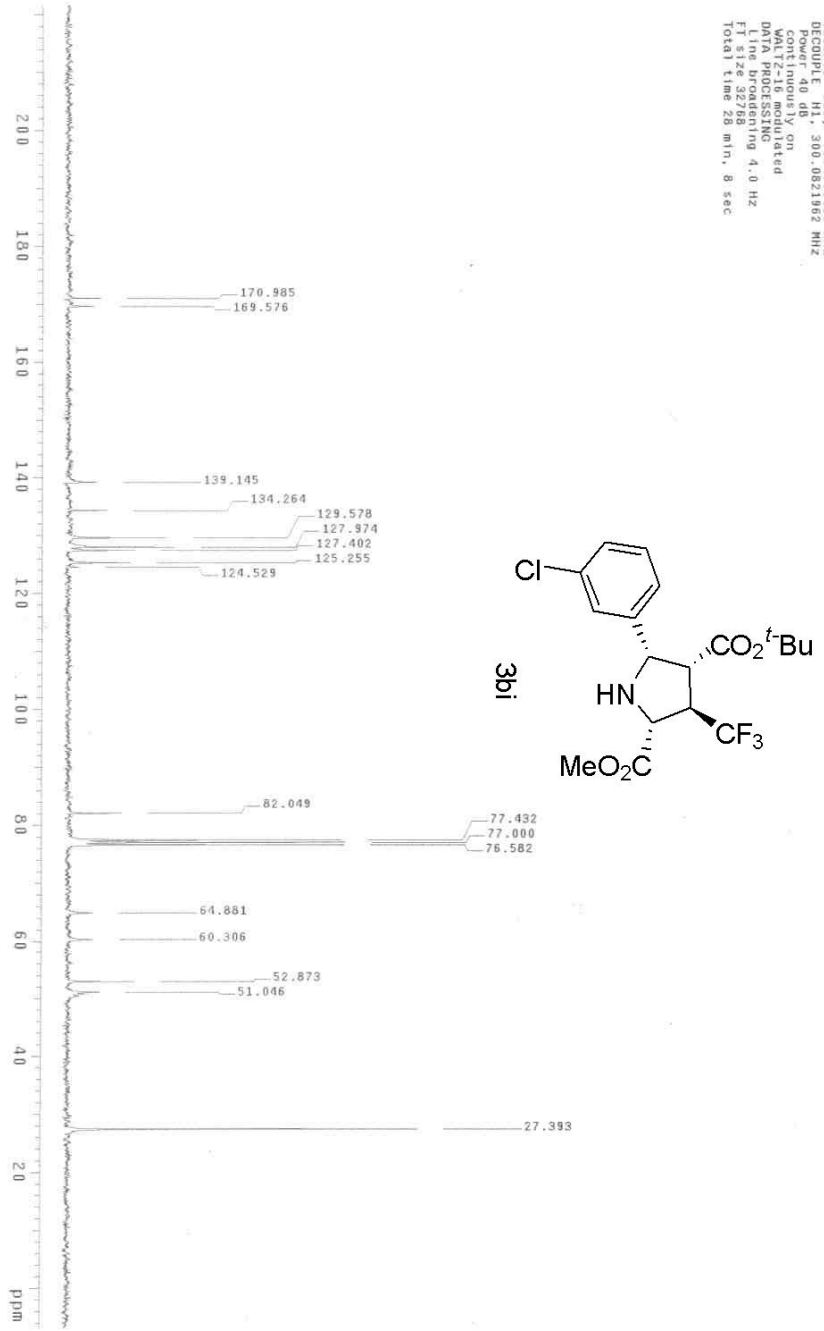

lqh-3-1
Archive directory: /export/home/xjw/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
14.34
4.17
3.88
13.31
4.94
5.23
4.37
37.85
2.74
7.323
7.266
4.612
4.592
4.089
4.069
3.901
3.871
3.535
3.344
1.255
1.095
1.064
1.040
0.853
0.000
ppm
3bg
Cl
CO2tBu
HN
CF3
MeO2C

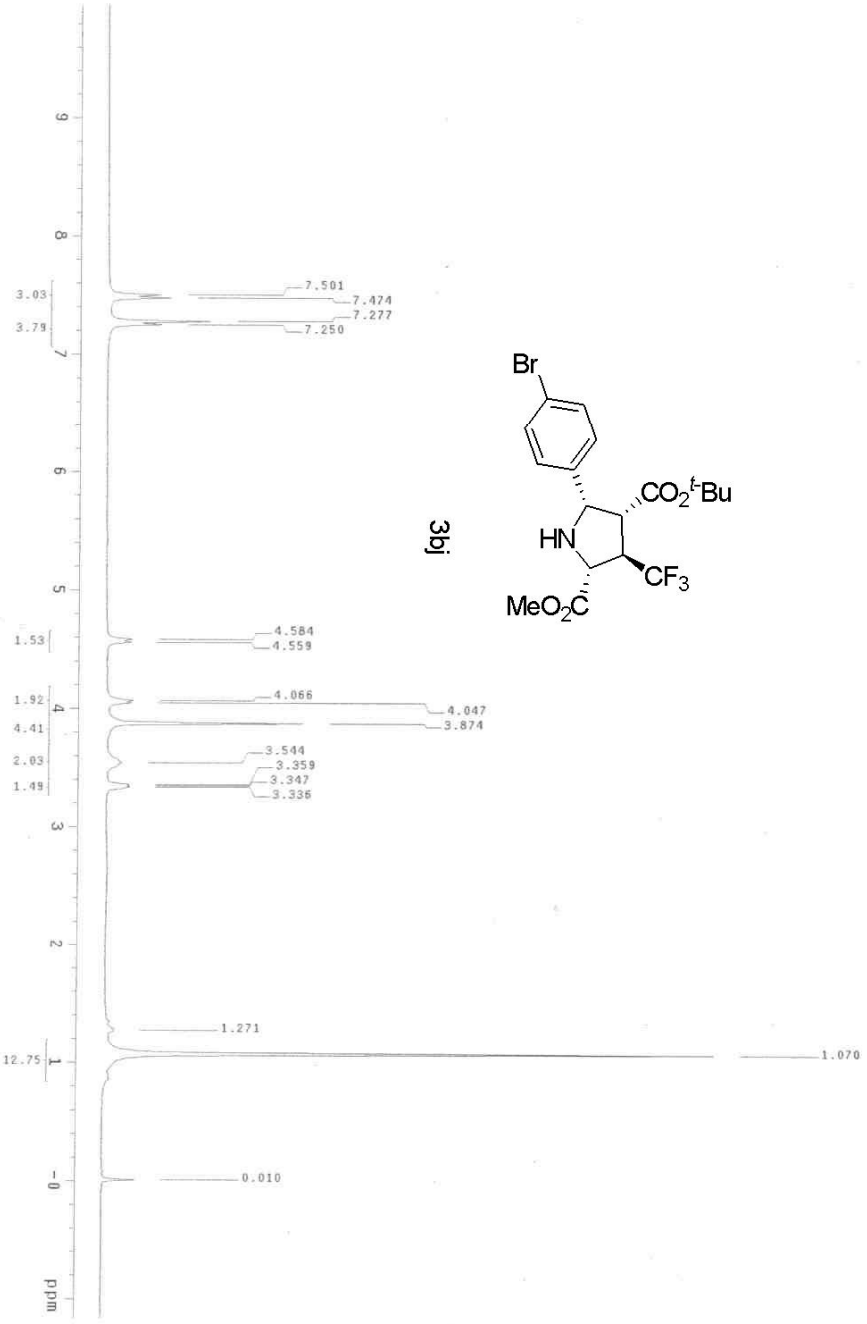

c13-1qh-3bg
171.058
169.637
135.586
133.589
128.487
128.308
127.253
125.405
81.945
77.209
77.000
76.783
64.777
60.304
52.809
51.103
50.721
50.549
50.370
27.368
ppm
3bg
Cl
CO2t-Bu
HN
CF3
MeO2C

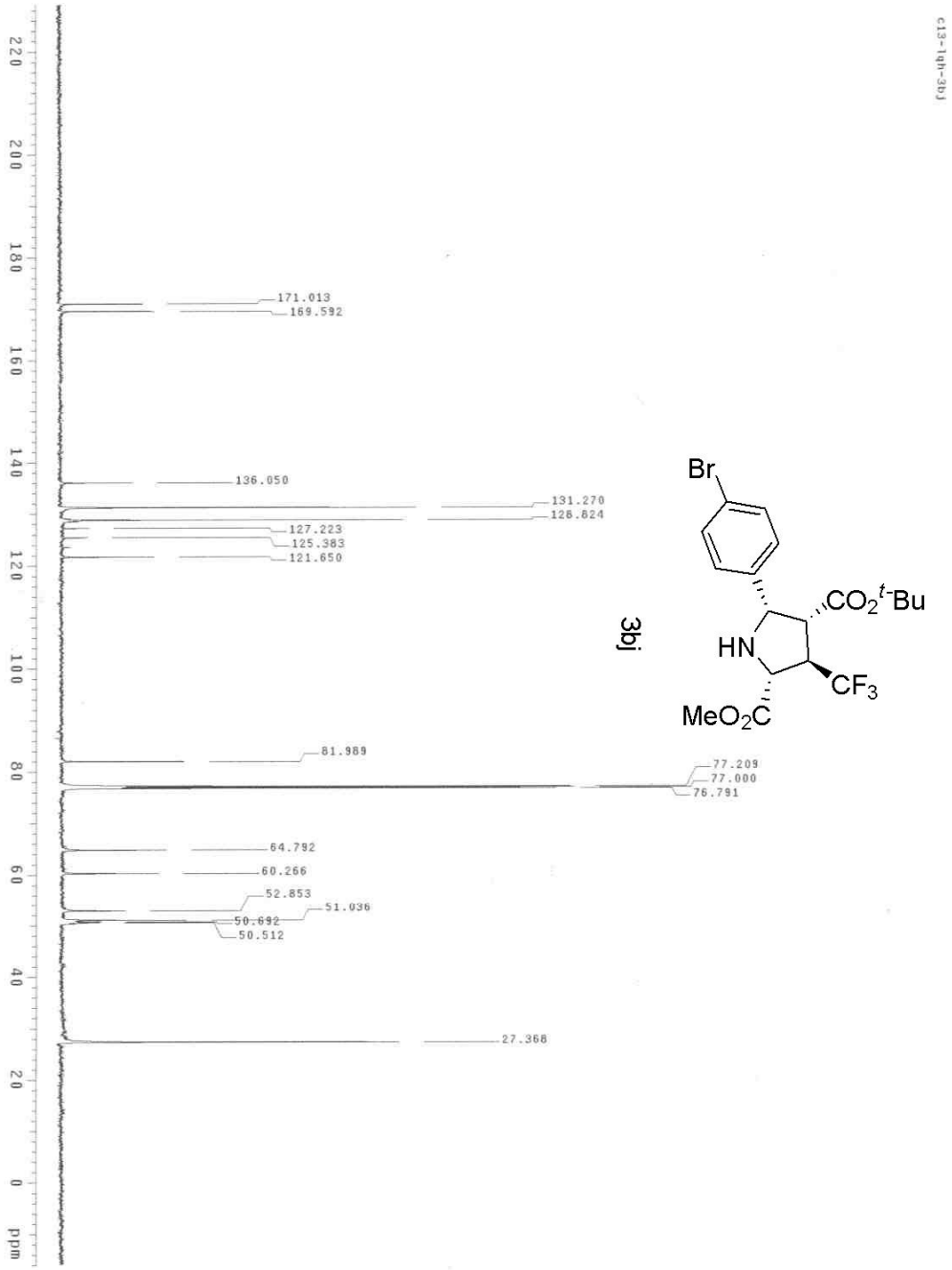

1qh-3-10a
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.508
7.493
7.372
7.360
7.256
7.242
4.806
4.782
4.013
3.992
3.851
3.656
3.640
1.468
0.979
2.59
3.05
4.97
2.44
2.77
7.44
5.24
2.98
25.04
ppm
3bh
Cl
CO2tBu
HN
CF3
MeO2C

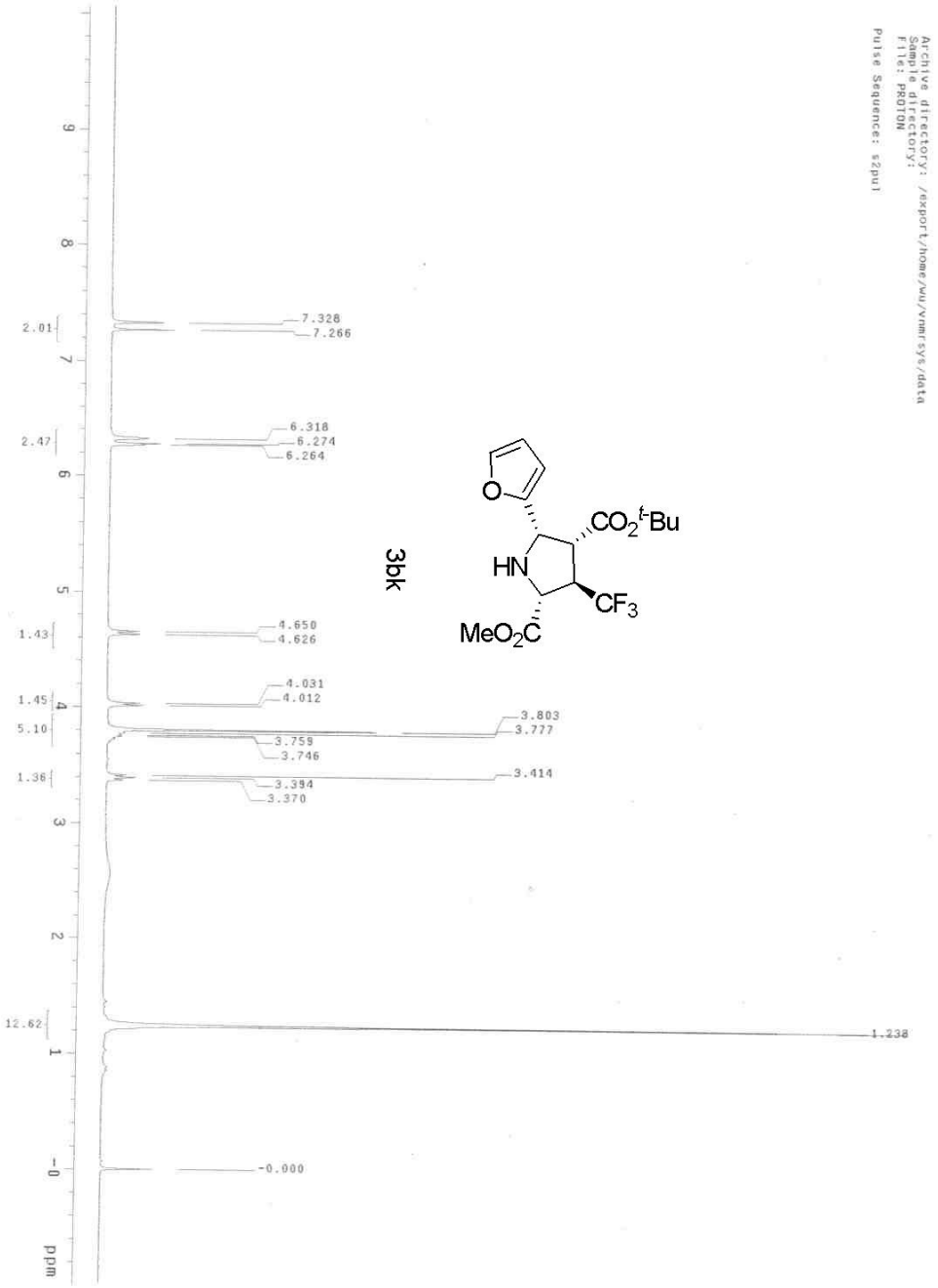

c13-1qh-3bh
171.028
169.719
134.943
133.896
129.205
128.974
127.620
127.395
126.789
125.548
81.578
77.209
77.000
76.791
62.526
59.623
52.816
49.973
49.801
48.373
27.278
220
200
180
160
140
120
100
80
60
40
20
0
ppm
3bh
Cl
CO2tBu
HN
CF3
MeO2C

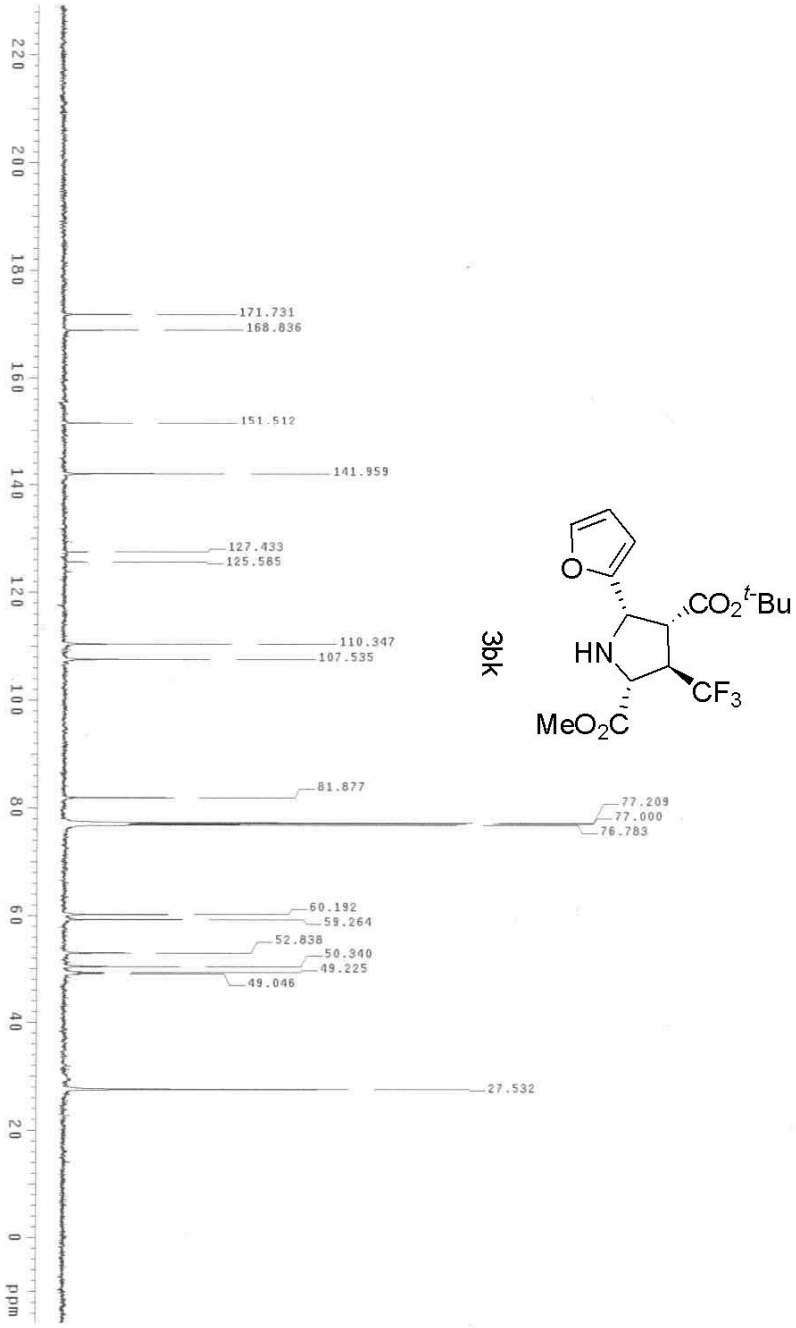

lqh-3-10c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.371
7.314
7.264
5.304
4.743
4.723
4.264
4.243
3.908
3.548
3.517
3.450
3.427
1.272
1.105
1.050
0.000
1.56
0.47
0.23
0.21
0.86
0.52
2.56
ppm
Cl
$CO_2{}^tBu$
HN
CF_3
MeO_2C
3bi

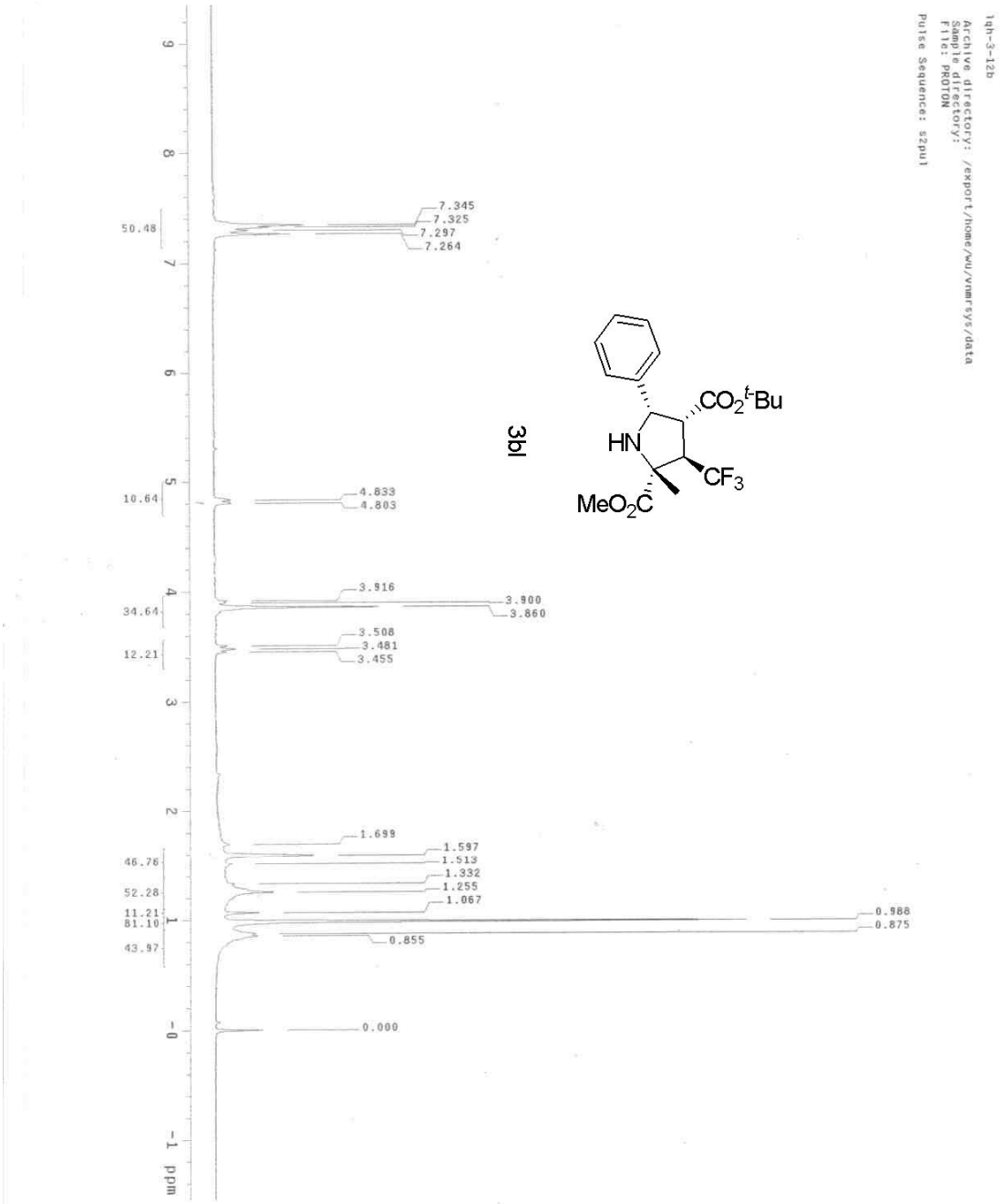

c13-lqh-3bi
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degree
Acq. time 1.500 sec
Width 12041.4 Hz
928 repetitions
OBSERVE C13, 75.4553033 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 8 sec
170.985
169.576
139.145
134.264
129.578
127.974
127.402
125.255
124.529
82.049
77.432
77.000
76.582
64.881
60.306
52.873
51.046
27.393
ppm
Cl
CO2tBu
HN
CF3
MeO2C
3bi

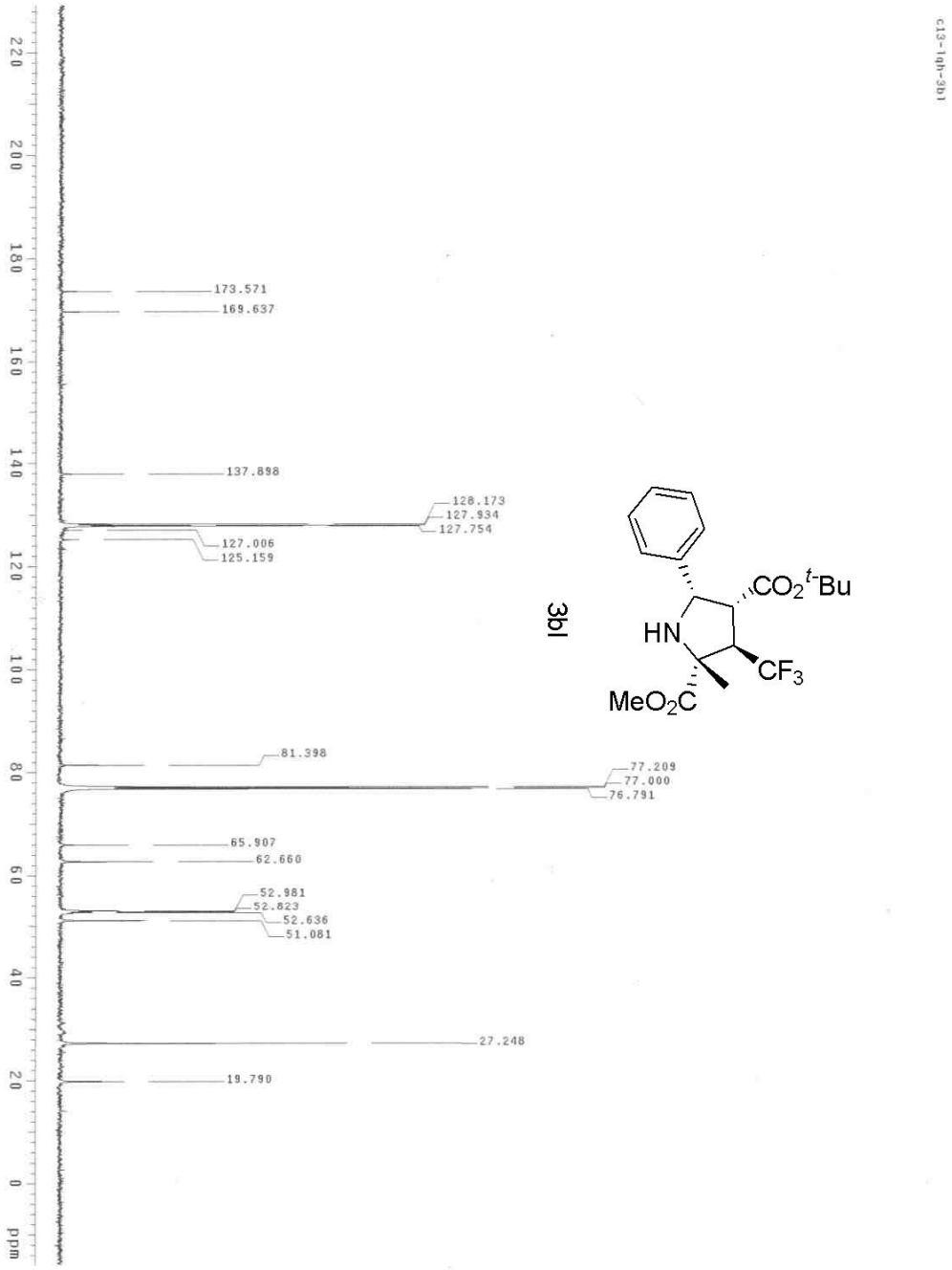

lqh-3-7-1
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.501
7.474
7.277
7.250
4.584
4.559
4.066
4.047
3.874
3.544
3.359
3.347
3.336
1.271
1.070
0.010
3.03
3.79
1.53
1.92
4.41
2.03
1.49
12.75
ppm
Br
CO2tBu
HN
CF3
MeO2C
3bj

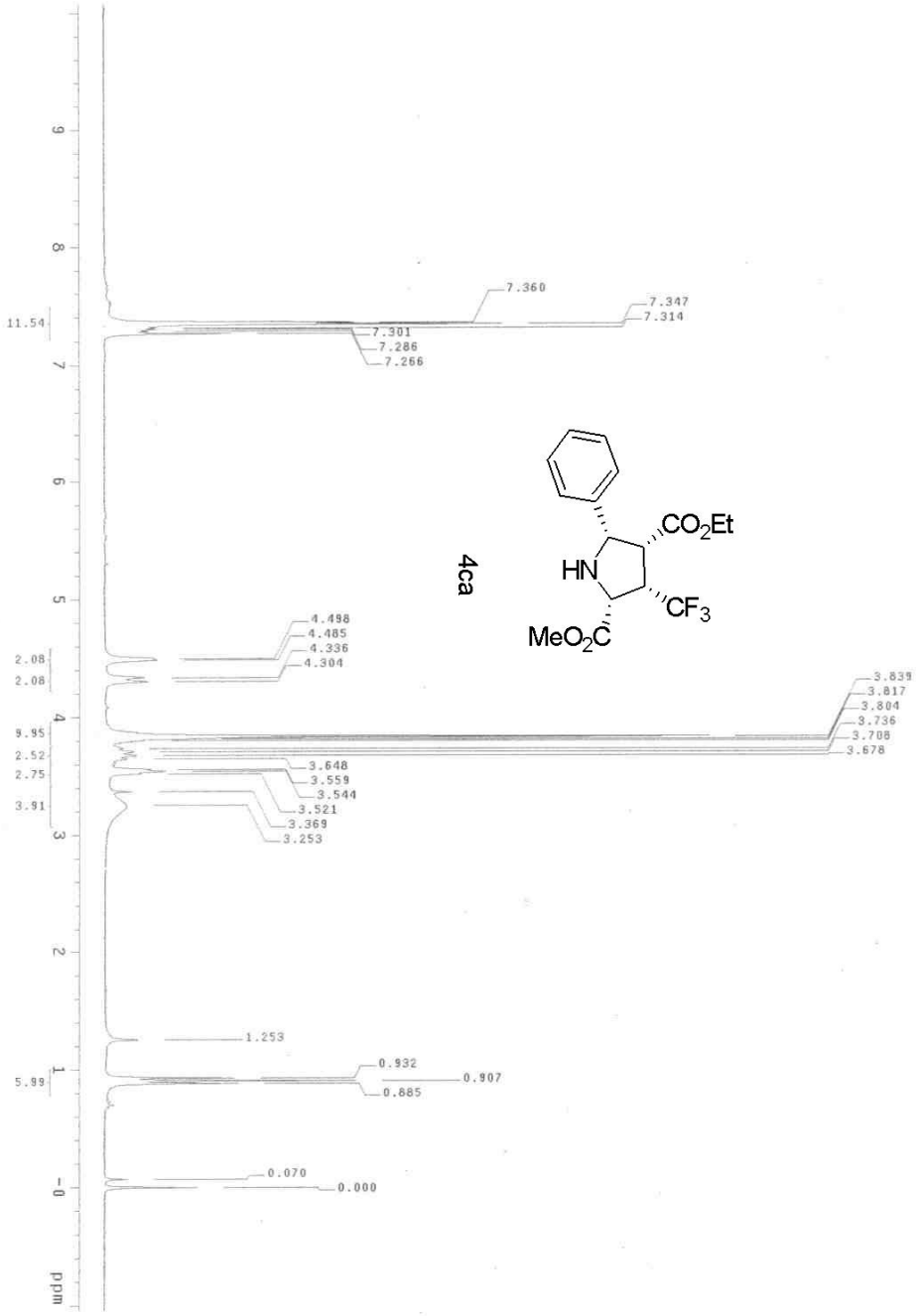

c13-1qh-3bj
171.013
169.592
136.050
131.270
128.824
127.223
125.383
121.650
81.989
77.209
77.000
76.791
64.792
60.266
52.853
51.036
50.692
50.512
27.368
ppm
3bj
Br
CO2t-Bu
HN
CF3
MeO2C

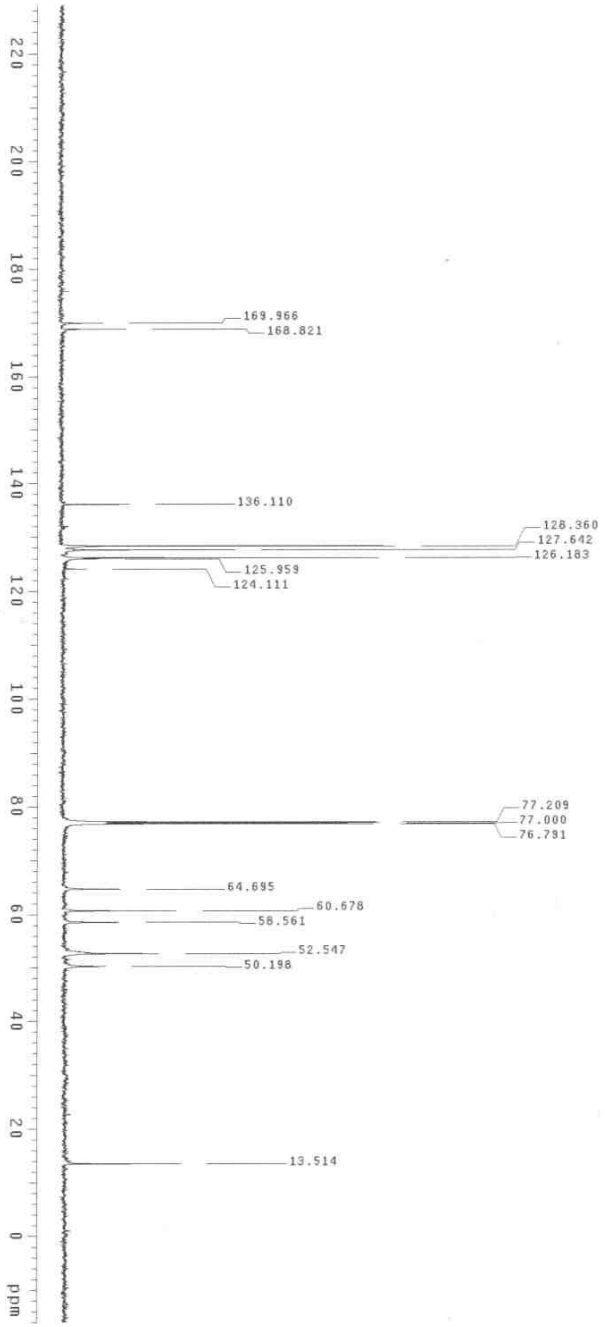

lqh-3-17d
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
7.328
7.266
6.318
6.274
6.264
4.650
4.626
4.031
4.012
3.803
3.777
3.759
3.746
3.414
3.394
3.370
1.238
-0.000
2.01
2.47
1.43
1.45
5.10
1.36
12.62
ppm
3bk
O
HN
CO2tBu
CF3
MeO2C

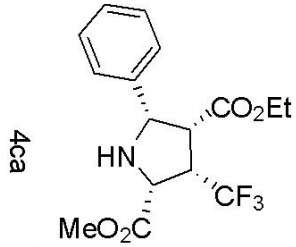

c13-1gh-3bk
171.731
168.836
151.512
141.959
127.433
125.585
110.347
107.535
81.877
77.209
77.000
76.783
60.192
59.264
52.838
50.340
49.225
49.046
27.532
ppm
3bk
O
HN
CO2tBu
CF3
MeO2C

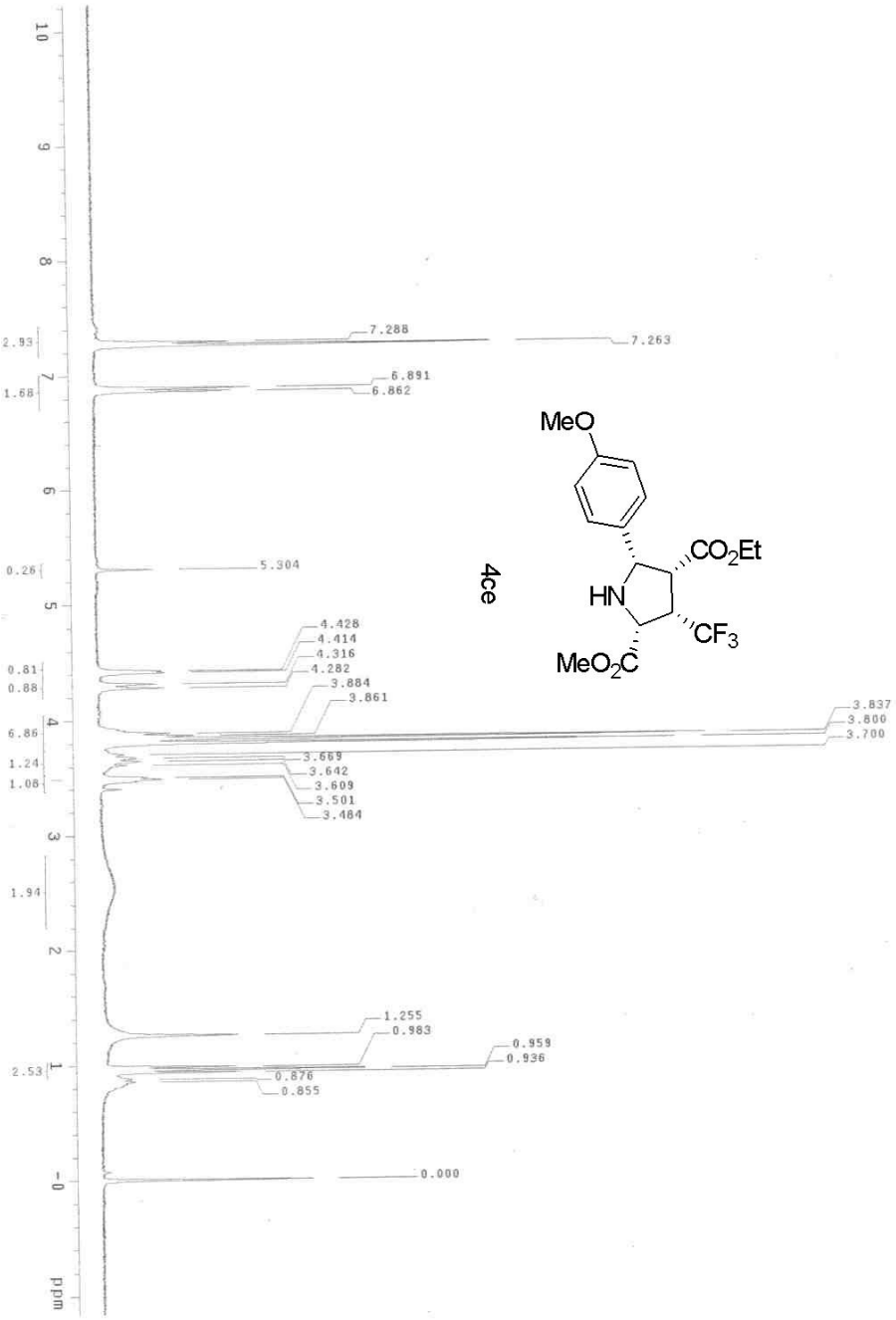

lqh-3-12b
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse sequence: s2pul
3bl
CO2tBu
HN
CF3
MeO2C
7.345
7.325
7.297
7.264
4.833
4.803
3.916
3.900
3.860
3.508
3.481
3.455
1.699
1.597
1.513
1.332
1.255
1.067
0.988
0.875
0.855
0.000
50.48
10.64
34.64
12.21
46.76
52.28
11.21
81.10
43.97
ppm

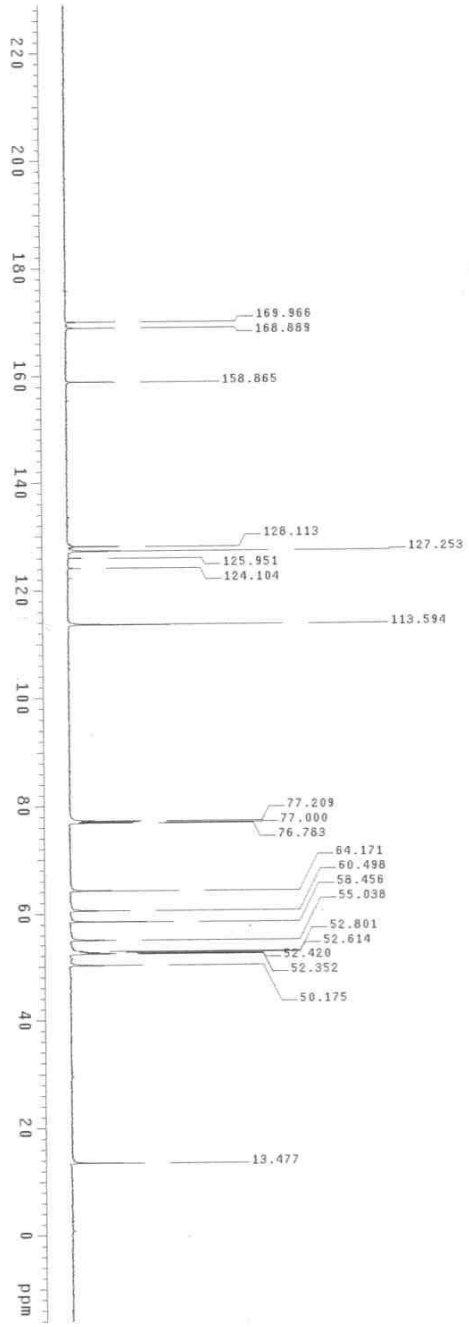

c13-1qh-3bl
220
200
180
160
140
120
100
80
60
40
20
0
ppm
173.571
169.637
137.898
128.173
127.934
127.754
127.006
125.159
81.398
77.209
77.000
76.791
65.907
62.660
52.981
52.823
52.636
51.081
27.248
19.790
3bl
$CO_2{}^tBu$
HN
CF_3
MeO_2C

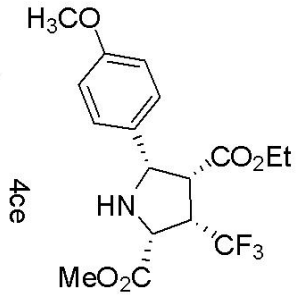

1qb-3-41a
7.360
7.347
7.314
7.301
7.286
7.266
11.54
4.488
4.485
4.336
4.304
2.08
2.08
3.839
3.817
3.804
3.736
3.708
3.678
9.95
2.52
2.75
3.91
3.648
3.559
3.544
3.521
3.369
3.253
1.253
5.99
0.932
0.907
0.885
0.070
0.000
ppm
4ca
CO2Et
HN
CF3
MeO2C


c13-1qh-4ca
220
200
180
160
140
120
100
80
60
40
20
0
ppm
169.966
168.821
136.110
128.360
127.642
126.183
125.959
124.111
77.209
77.000
76.791
64.695
60.678
58.561
52.547
50.198
13.514

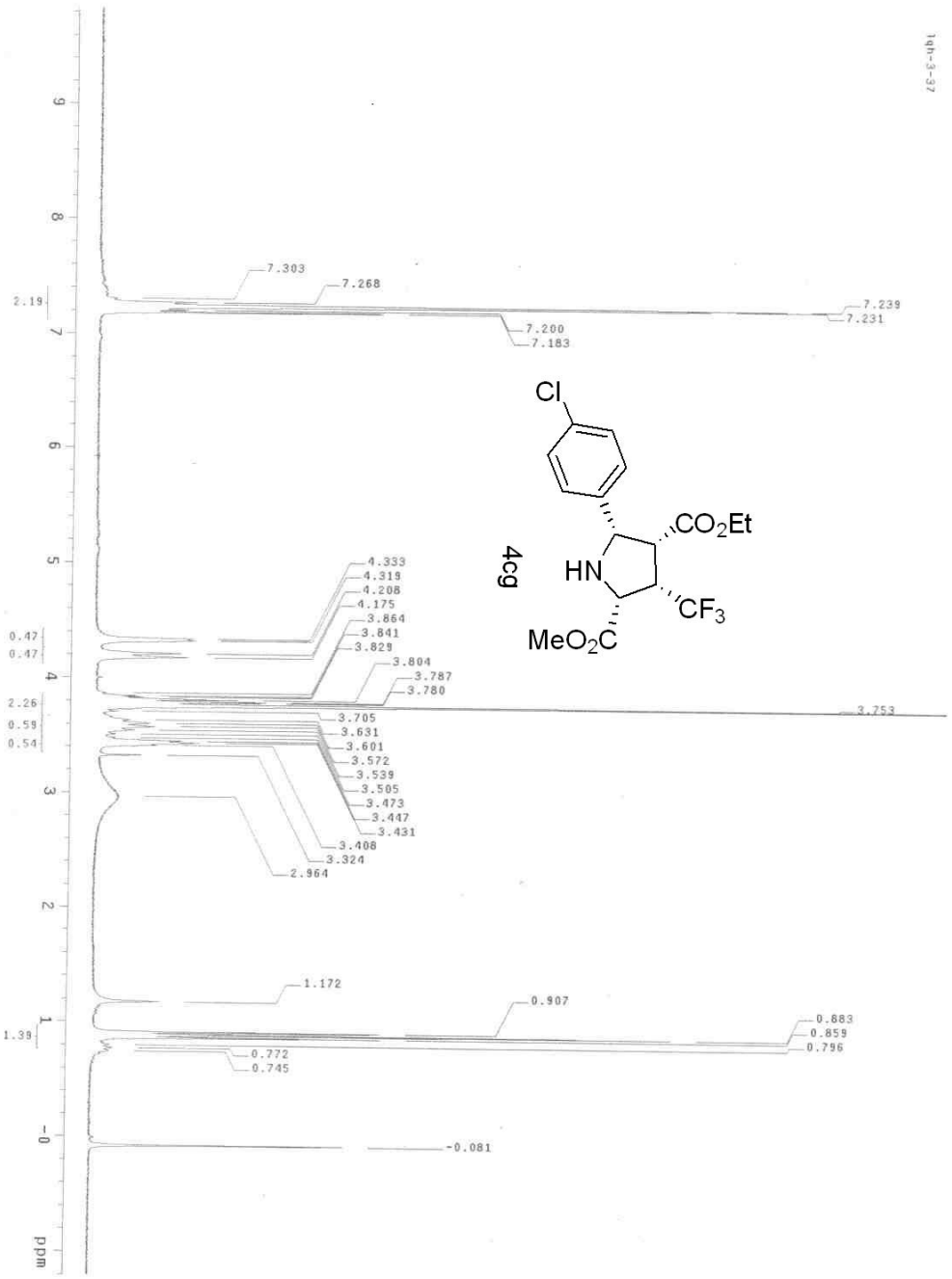

4ca
CO2Et
HN
CF3
MeO2C

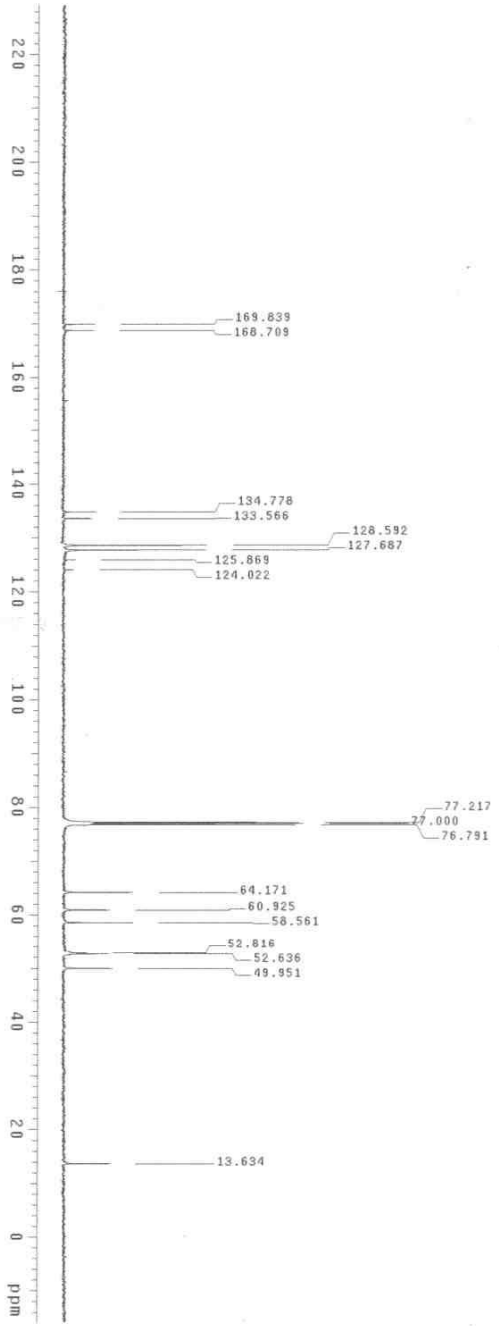

lqh-3-41b
7.288
7.263
6.891
6.862
5.304
4.428
4.414
4.316
4.282
3.884
3.861
3.837
3.800
3.700
3.669
3.642
3.609
3.501
3.484
1.255
0.983
0.959
0.936
0.876
0.855
0.000
2.93
1.68
0.26
0.81
0.88
6.86
1.24
1.08
1.94
2.53
ppm
MeO
CO2Et
HN
CF3
MeO2C
4ce

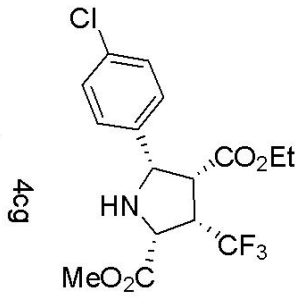

c13-1qh-4ce
220
200
180
160
140
120
100
80
60
40
20
0
ppm
169.966
168.889
158.865
128.113
127.253
125.951
124.104
113.594
77.209
77.000
76.783
64.171
60.498
58.456
55.038
52.801
52.614
52.420
52.352
50.175
13.477


H3CO
CO2Et
HN
CF3
MeO2C

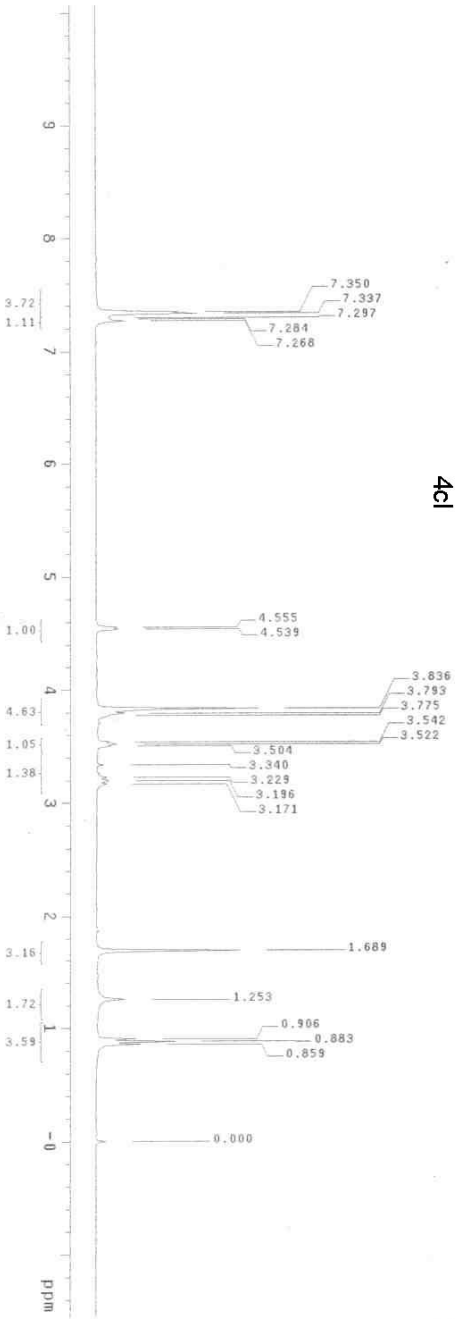
4ce


7.303
7.268
7.239
7.231
7.200
7.183
4.333
4.319
4.208
4.175
3.864
3.841
3.829
3.804
3.787
3.780
3.753
3.705
3.631
3.601
3.572
3.539
3.505
3.473
3.447
3.431
3.408
3.324
2.964
1.172
0.907
0.883
0.859
0.796
0.772
0.745
-0.081
2.19
0.47
0.47
2.26
0.59
0.54
1.39
ppm
4cg
Cl
CO2Et
HN
CF3
MeO2C

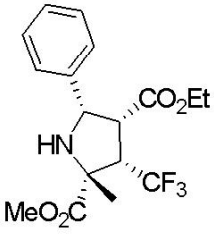

c13-1qh-4cg
220
200
180
160
140
120
100
80
60
40
20
0
ppm
169.839
168.709
134.778
133.566
128.592
127.687
125.869
124.022
77.217
77.000
76.791
64.171
60.925
58.561
52.816
52.636
49.951
13.634

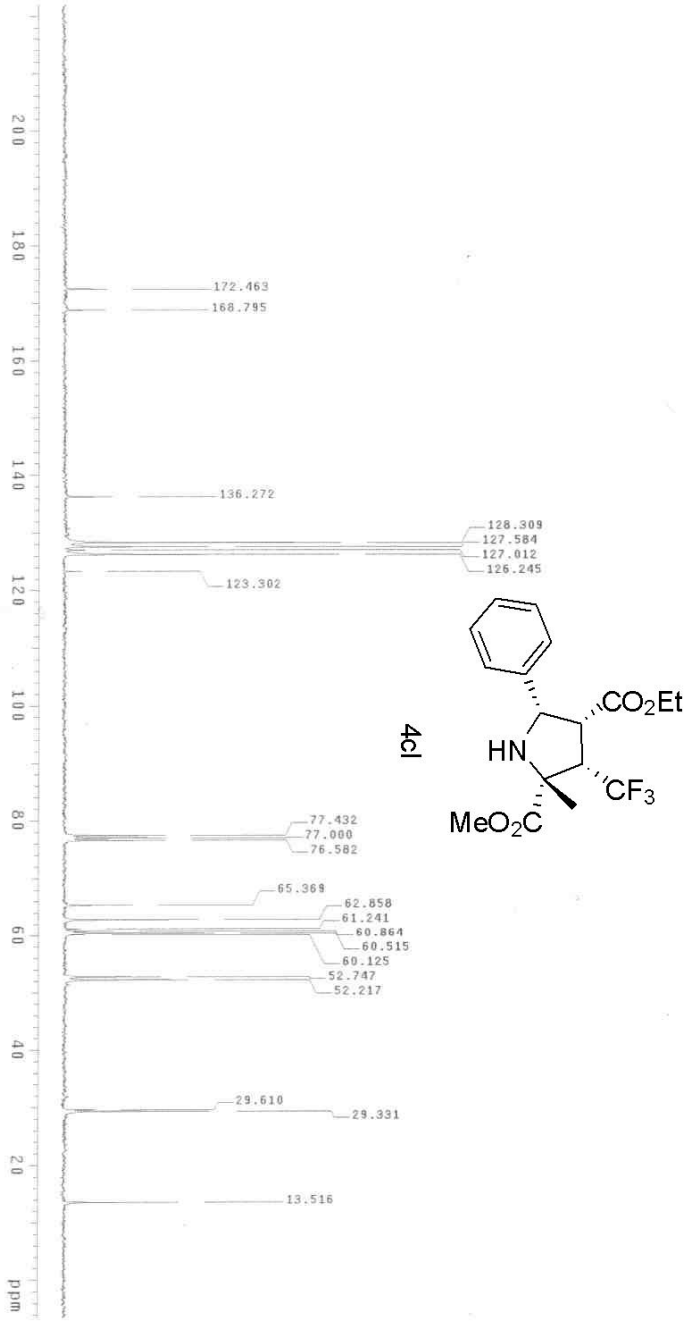

Cl
CO2Et
HN
CF3
MeO2C

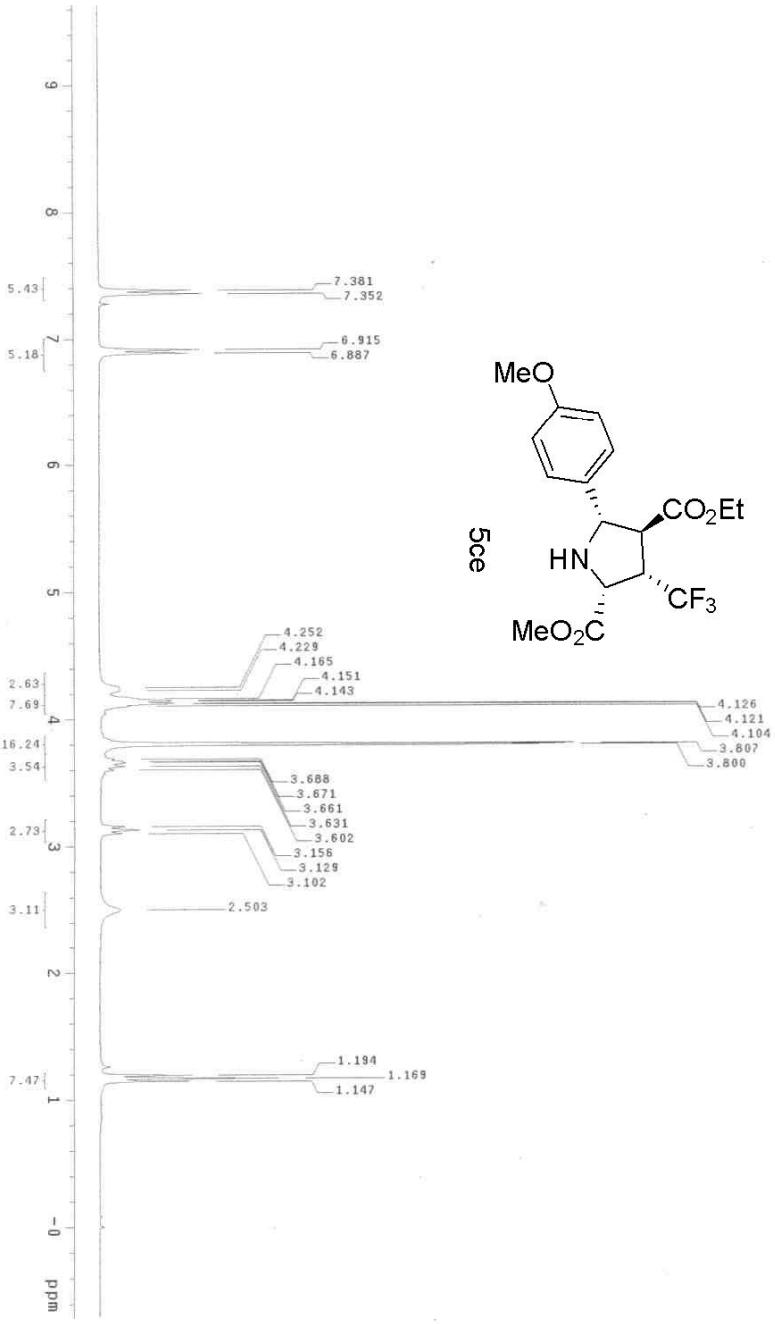
4cg

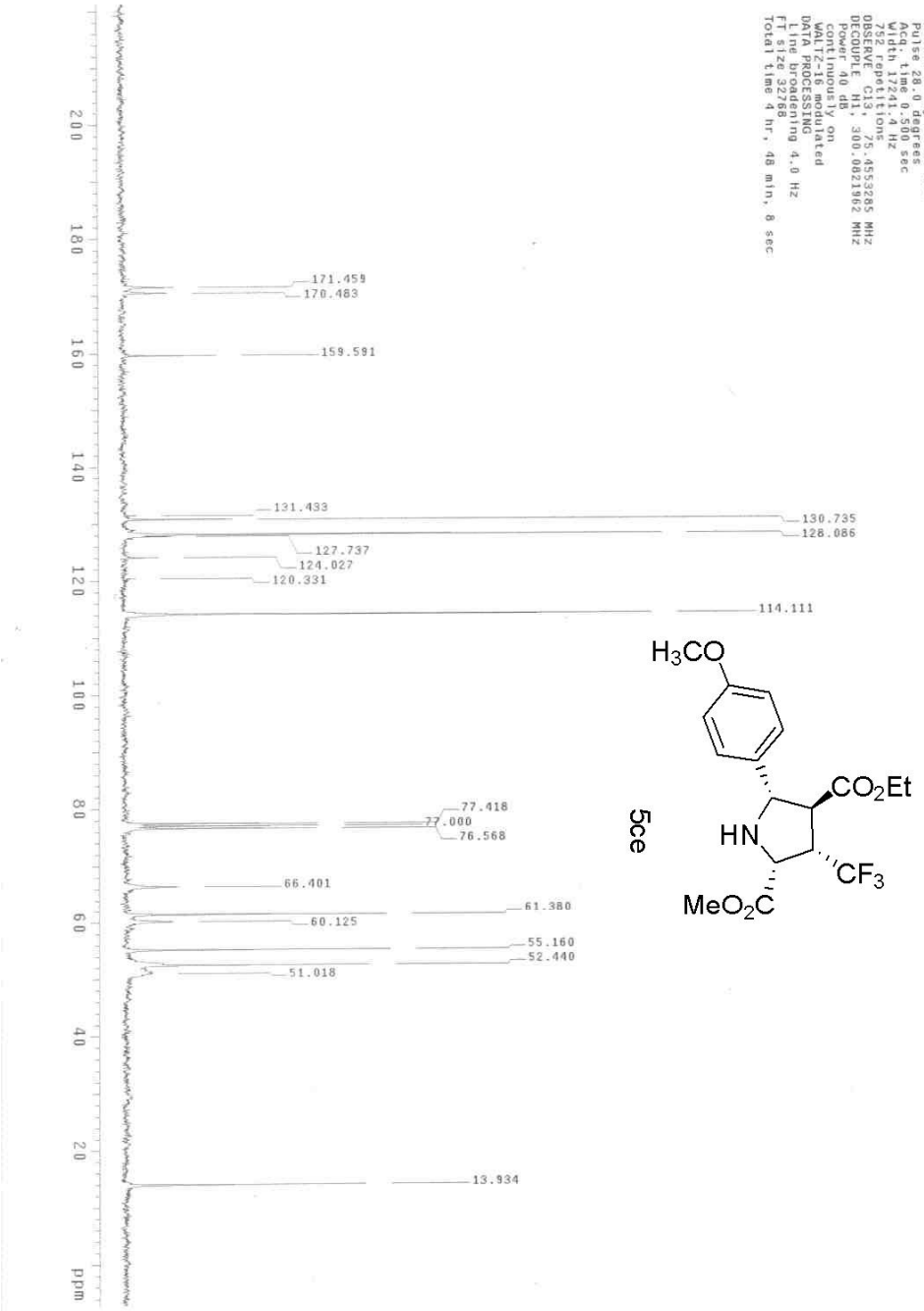

7.350
7.337
7.297
7.284
7.268
3.72
1.11
4.555
4.539
1.00
3.836
3.793
3.775
3.542
3.522
4.63
1.05
3.504
3.340
3.229
3.196
3.171
1.38
1.689
3.16
1.253
1.72
0.906
0.883
0.859
3.59
0.000
ppm

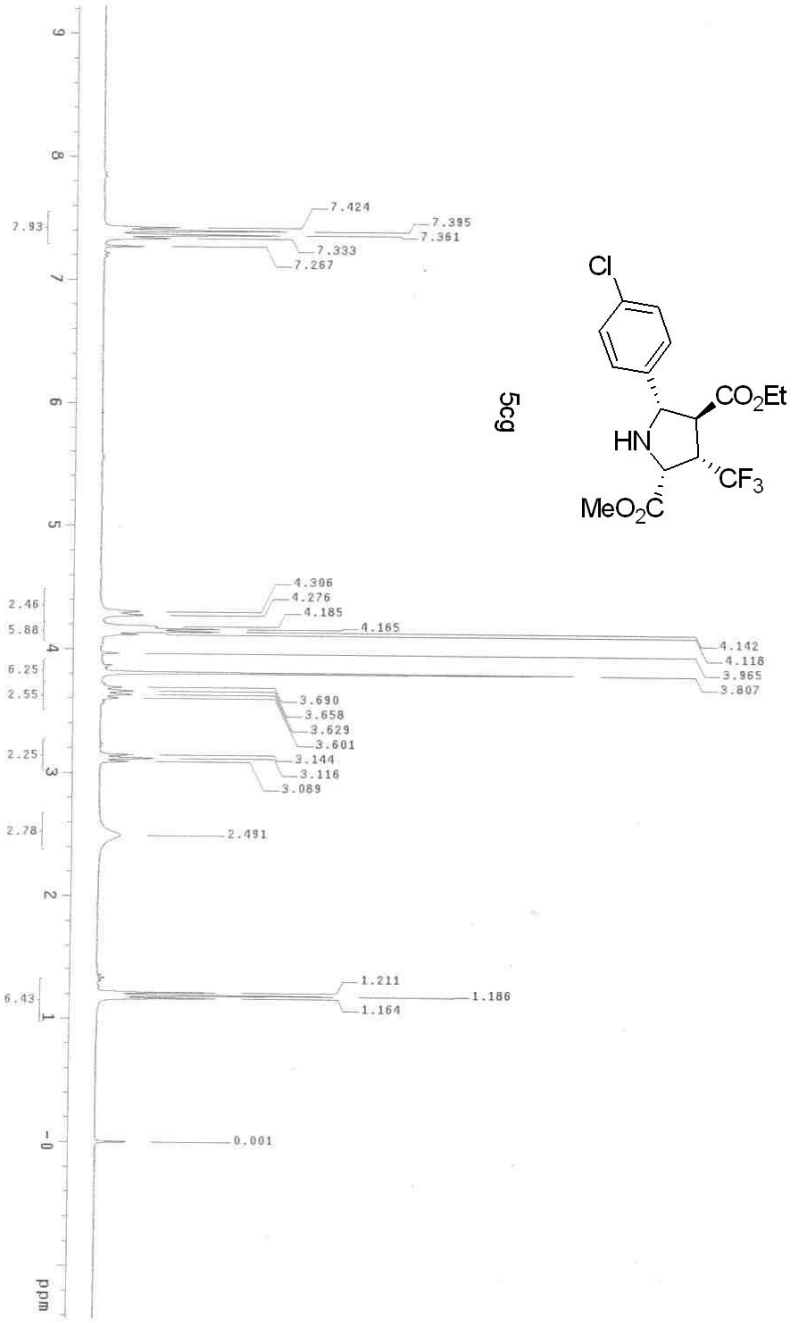
4cl

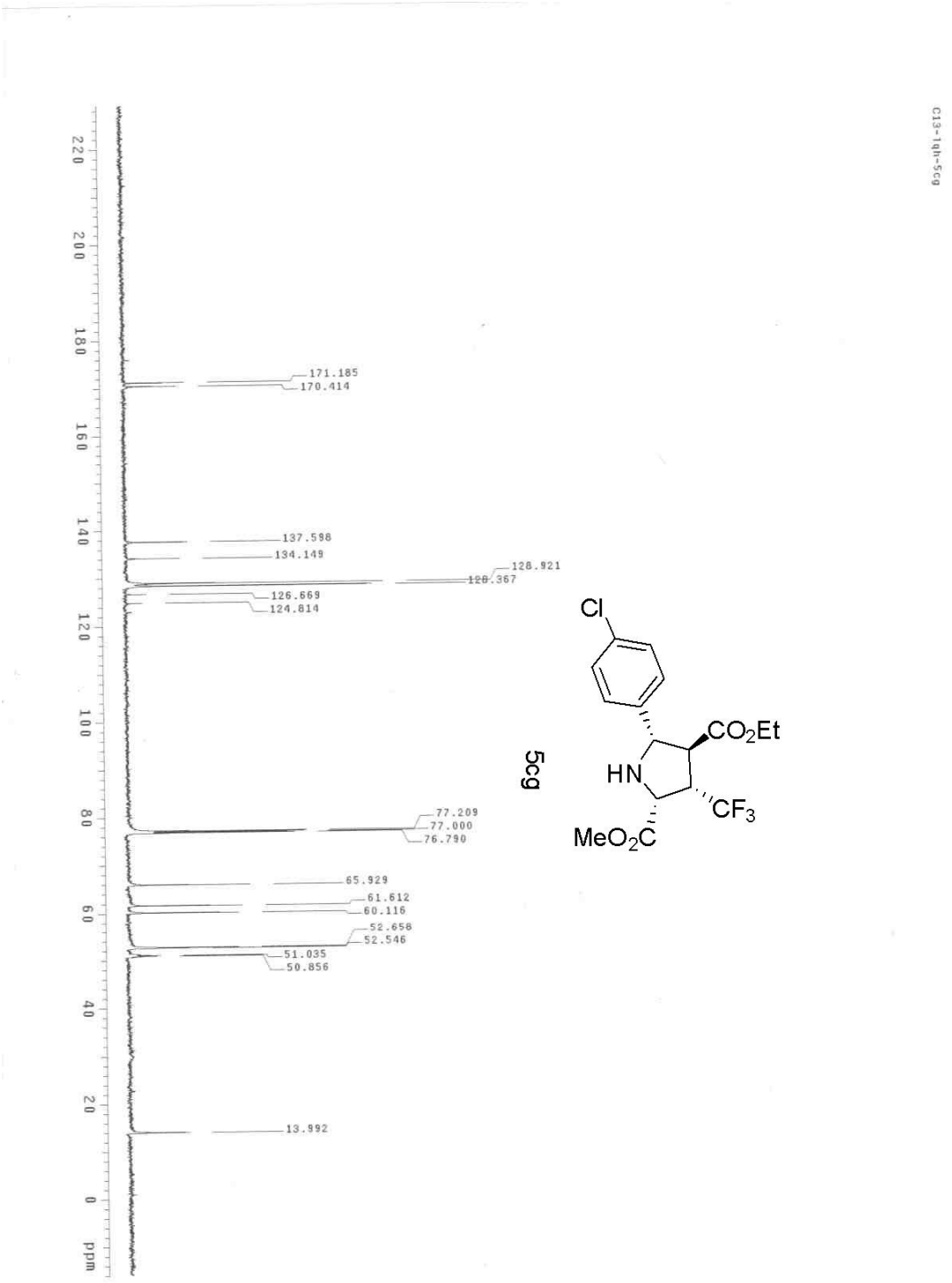

CO2Et
HN
CF3
MeO2C

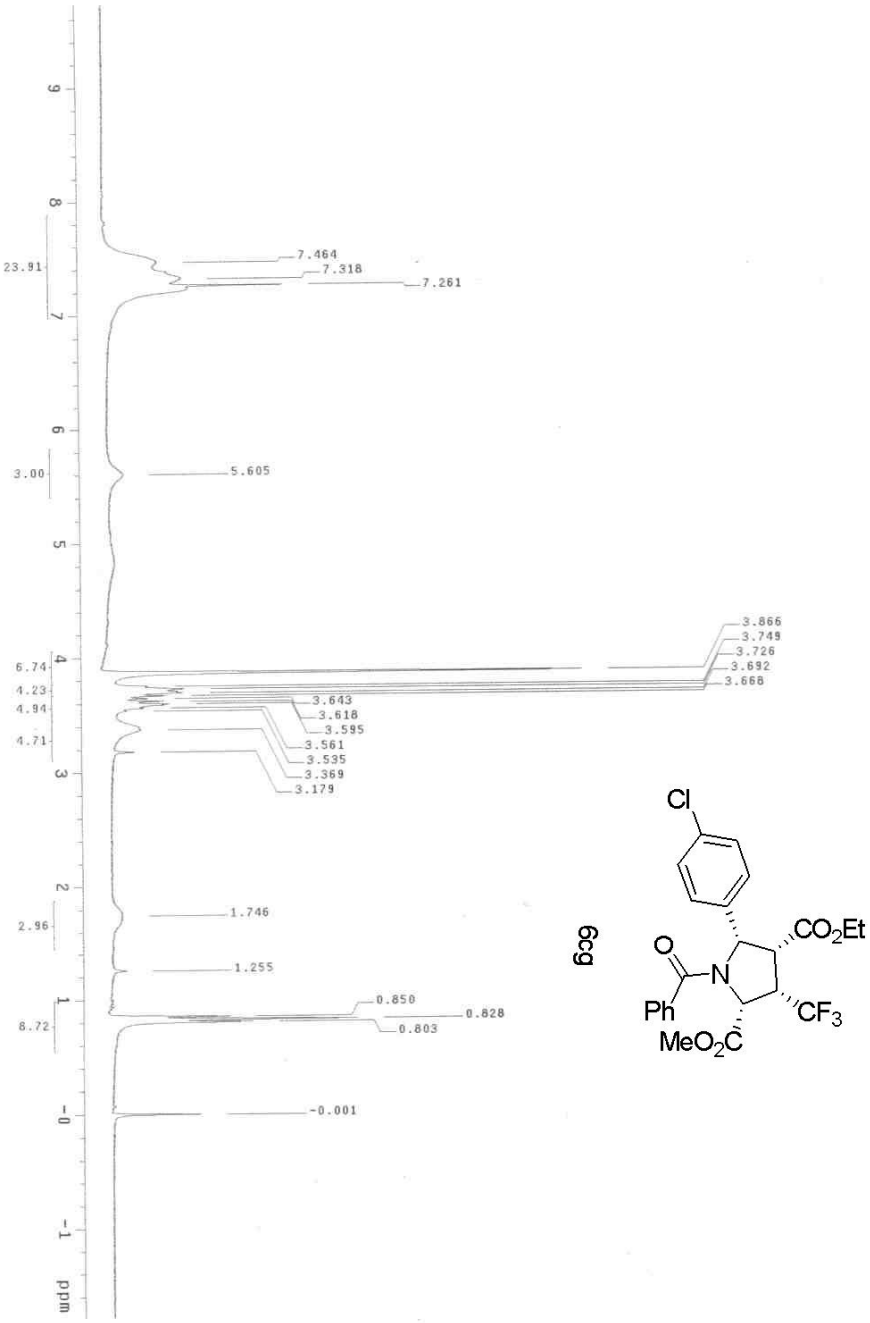

1qh-4cl
Solvent: CDCl3
Ambient temperature
File: 1qh-4cl
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.000 sec
Width 12041.4 Hz
808 repetitions
OBSERVE C13, 75.4552780 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 8 sec
172.463
168.795
136.272
128.309
127.584
127.012
126.245
123.302
77.432
77.000
76.582
65.369
62.858
61.241
60.864
60.515
60.125
52.747
52.217
29.610
29.331
13.516
ppm
4cl
HN
CO2Et
CF3
MeO2C

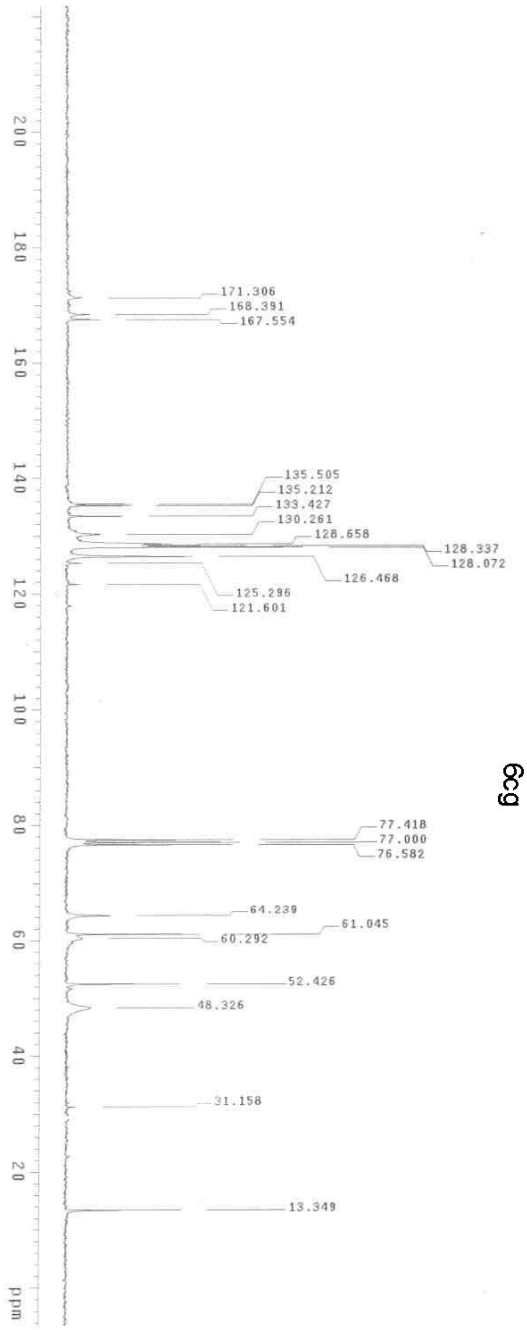

lqh-0530
5ce
MeO
CO2Et
HN
CF3
MeO2C
7.381
7.352
6.915
6.887
4.252
4.229
4.165
4.151
4.143
4.126
4.121
4.104
3.807
3.800
3.688
3.671
3.661
3.631
3.602
3.156
3.129
3.102
2.503
1.194
1.169
1.147
5.43
5.18
2.63
7.69
16.24
3.54
2.73
3.11
7.47
ppm

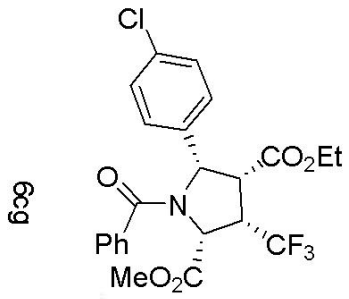

1qh-5ce
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17241.4 Hz
1 repetition
OBSERVE C13, 75.4553285 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 48 min, 8 sec
171.459
170.483
159.591
131.433
130.735
128.086
127.737
124.027
120.331
114.111
77.418
77.000
76.568
66.401
61.380
60.125
55.160
52.440
51.018
13.934
ppm
5ce
H3CO
CO2Et
HN
CF3
MeO2C


lqh-110527-2
7.424
7.395
7.361
7.333
7.267
7.93
4.306
4.276
4.185
4.165
4.142
4.118
3.965
3.807
2.46
5.88
6.25
2.55
3.690
3.658
3.629
3.601
3.144
3.116
3.089
2.25
2.78
2.491
1.211
1.186
1.164
6.43
0.001
ppm
5cg
Cl
CO2Et
HN
CF3
MeO2C

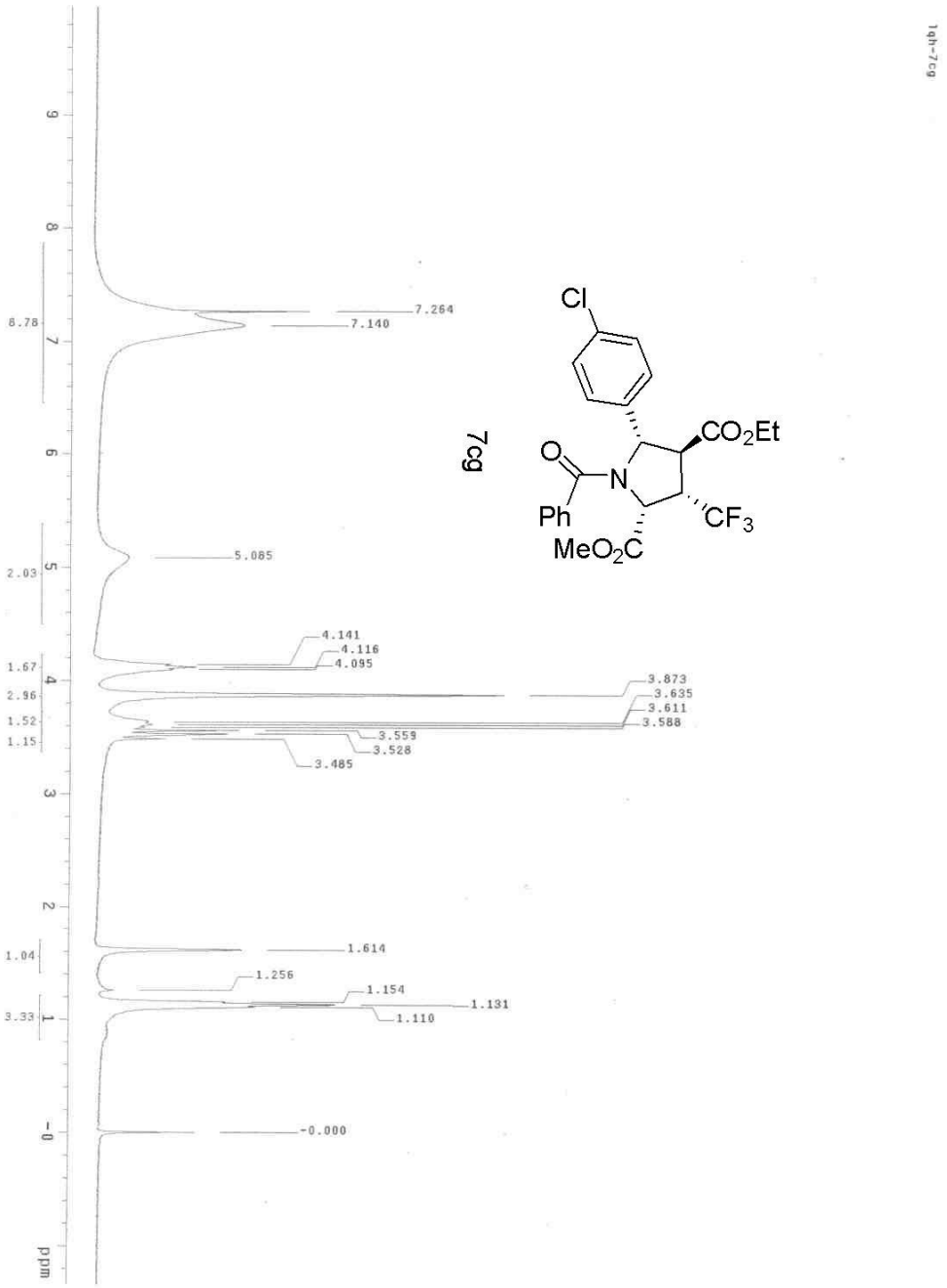

C13-1qh-5cg
171.185
170.414
137.598
134.149
128.921
128.367
126.669
124.814
77.209
77.000
76.790
65.929
61.612
60.116
52.658
52.546
51.035
50.856
13.992
ppm
5cg
Cl
CO2Et
HN
CF3
MeO2C

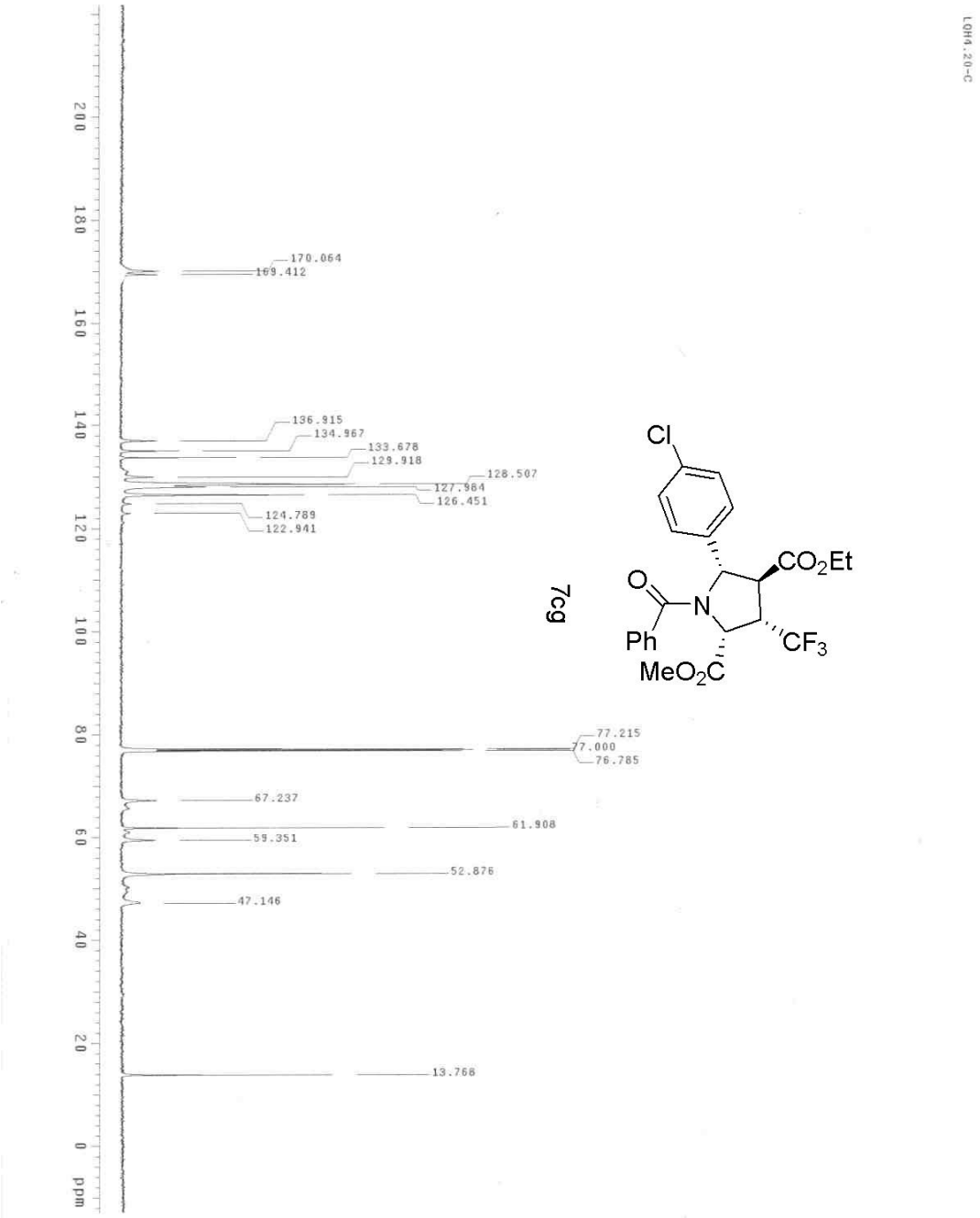

lqh-11027-1
7.464
7.318
7.261
5.605
3.866
3.749
3.726
3.692
3.668
3.643
3.618
3.595
3.561
3.535
3.369
3.179
1.746
1.255
0.850
0.828
0.803
-0.001
23.91
3.00
6.74
4.23
4.94
4.71
2.96
6.72
ppm
6gg
Cl
CO2Et
O
N
Ph
CF3
MeO2C


c13-1qh-6cg
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.091 sec
Width 12741.3 Hz
2784 repetitions
OBSERVE C13, 75.4552791 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 8 sec
171.306
168.391
167.554
135.505
135.212
133.427
130.261
128.658
128.337
128.072
126.468
125.296
121.601
77.418
77.000
76.582
64.239
61.045
60.292
52.426
48.326
31.158
13.349
ppm


Cl
CO2Et
O
N
Ph
CF3
MeO2C

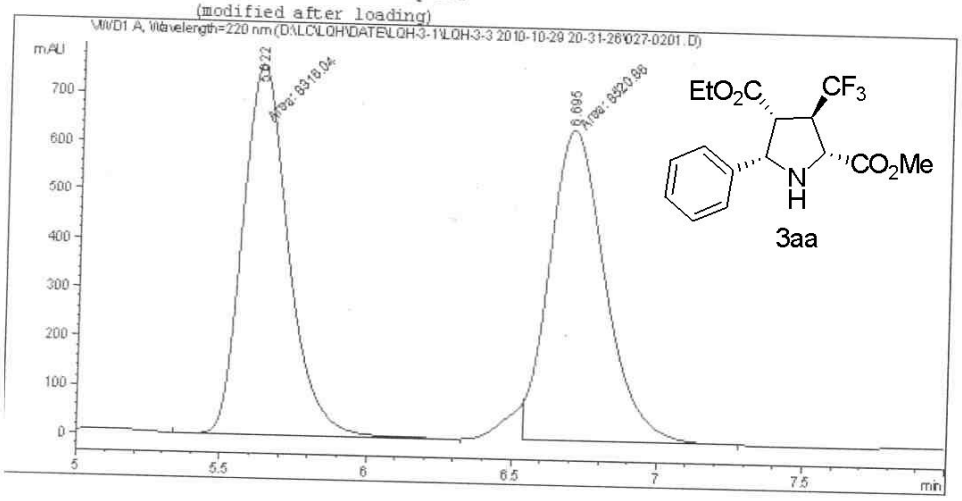
6cg


1qh-7cg
7cg
Cl
CO2Et
O
N
Ph
CF3
MeO2C
7.264
7.140
5.085
4.141
4.116
4.095
3.873
3.635
3.611
3.588
3.559
3.528
3.485
1.614
1.256
1.154
1.131
1.110
-0.000
8.78
2.03
1.67
2.96
1.52
1.15
1.04
3.33
ppm


LQH4.20-C
170.064
169.412
136.915
134.967
133.678
129.918
128.507
127.984
126.451
124.789
122.941
77.215
77.000
76.785
67.237
61.908
59.351
52.876
47.146
13.768
200
180
160
140
120
100
80
60
40
20
0
ppm
7cg
Cl
CO2Et
O
N
Ph
CF3
MeO2C

ta File D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\027-0201.D
mple Name: LQH-3-3

Acq. Operator : DXQ
Acq. Instrument : Instrument 1
Injection Date : 10/29/2010 8:43:51 PM
Seq. Line : 2
Location : Vial 27
Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\ASH-2-98-10-220NM-20MIN.M
Last changed : 10/26/2010 8:40:56 AM by TMC
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\027-0201.D\DA.M (ASH-2-98-10-220NM-20MIN.M)
Last changed : 5/29/2011 7:05:04 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=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.622	MM	0.1826	8316.03711	759.20740	49.3918
2	6.695	FM	0.2228	8520.85645	637.48792	50.6082
Totals :				1.68369e4	1396.69531	

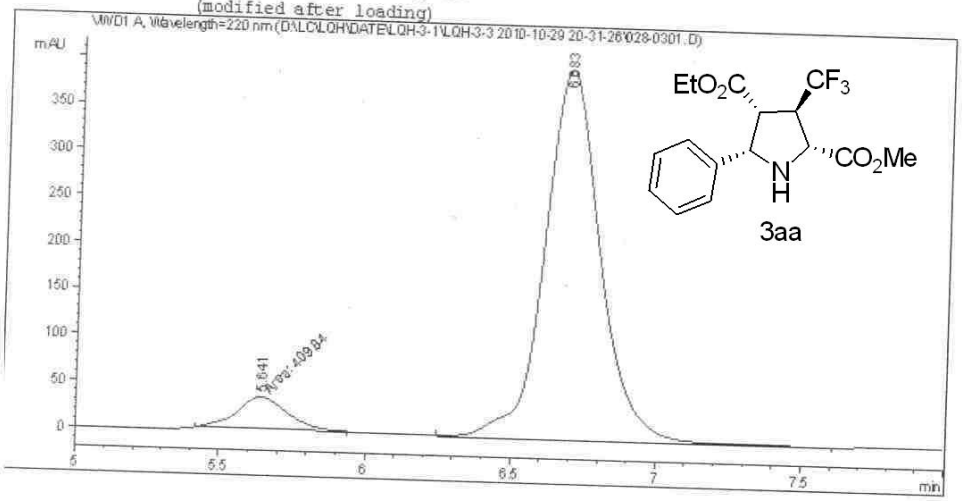
*** End of Report ***

ment 1 5/29/2011 7:05:30 PM LTL

Page 1 of 1

ta File D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\028-0301.D
mple Name: LQH-2-96B

Acq. Operator : DXQ
Acq. Instrument : Instrument 1
Injection Date : 10/29/2010 9:05:24 PM
Seq. Line : 3
Location : Vial 28
Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\ASH-2-98-10-220NM-20MIN.M
Last changed : 10/26/2010 8:40:56 AM by TMC
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-3 2010-10-29 20-31-26\028-0301.D\DA.M (ASH-2-98-10-220NM-20MIN.M)
Last changed : 5/29/2011 7:07:12 PM by LTL
(modified after loading)



Area Percent Report

orted By : Signal
ultiplier : 1.0000
ilution : 1.0000
se Multiplier & Dilution Factor with ISTDs

ignal 1: VWD1 A, Wavelength=220 nm

eak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.641	MM	0.1983	409.84015	34.45402	7.1710
2	6.683	BB	0.2019	5305.40674	398.54132	92.8290
tals :				5715.24689	432.99534	

*** End of Report ***

ment 1 5/29/2011 7:07:18 PM LTL

a File D:\LC\LQH\DATE\LQH-2-81\LQH-2-81 2010-10-12 18-58-21\092-0201.D
ple Name: LQH-2-81

```
=====================================================================
Acq. Operator   : DXQ                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 92
Injection Date  : 10/12/2010 7:10:42 PM            Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-2-81\LQH-2-81 2010-10-12 18-58-21\ASH-2-98-1ML-220NM.M
Last changed    : 10/12/2010 6:57:54 PM by DXQ
Analysis Method : D:\LC\LQH\DATE\LQH-2-81\LQH-2-81 2010-10-12 18-58-21\092-0201.D\DA.M (ASH-
                  2-98-1ML-220NM.M)
Last changed    : 10/12/2010 7:21:20 PM by DXQ
                  (modified after loading)
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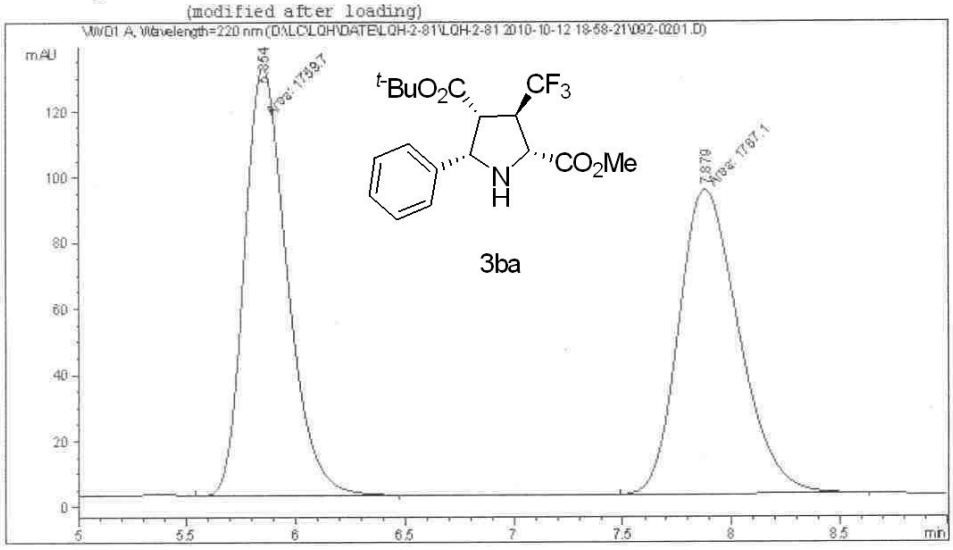



```
=====================================================================
                        Area Percent Report
=====================================================================

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   5.854 MM    0.2261 1759.69812  129.72574  49.8950
   2   7.879 MM    0.3191 1767.10266   92.28297  50.1050

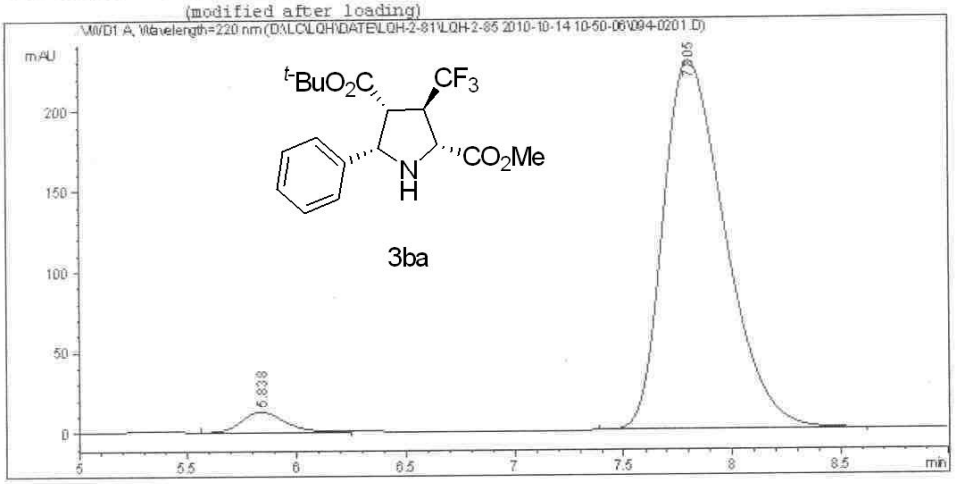
Totals :                  3526.80078  222.00871

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

strument 1 10/12/2010 7:21:24 PM DXQ Page 1 of 1

a File D:\LC\LQH\DATE\LQH-2-81\LQH-2-85 2010-10-14 10-50-06\094-0201.D
ple Name: lqh-2-85

Acq. Operator : tmc
Acq. Instrument : Instrument 1
Injection Date : 10/14/2010 11:02:25 AM
Seq. Line : 2
Location : Vial 94
Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\LQH\DATE\LQH-2-81\LQH-2-85 2010-10-14 10-50-06\ASH-2-98-1ML-220NM-10MIN.M
Last changed : 9/29/2010 2:44:36 PM by DXQ
Analysis Method : D:\LC\LQH\DATE\LQH-2-81\LQH-2-85 2010-10-14 10-50-06\094-0201.D\DA.M (ASH-2-98-1ML-220NM-10MIN.M)
Last changed : 1/20/2011 8:14:41 PM by THL-7-95-97
(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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.838	VB	0.2094	174.49124	12.72870	3.7309
2	7.805	BB	0.3067	4502.38525	228.79823	96.2691
Totals :				4676.87650	241.52693	

*** End of Report ***

ta File D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\025-0401.D
mple Name: LQH-3-4

```
==========================================================================
Acq. Operator   : DXQ                          Seq. Line :   4
Acq. Instrument : Instrument 1                  Location : Vial 25
Injection Date  : 10/29/2010 7:29:45 PM            Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\ASH-2-98-1ML-220NM.M
Last changed    : 10/29/2010 7:39:19 PM by DXQ
                  (modified after loading)
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\025-0401.D\DA.M (ASH-
                  2-98-1ML-220NM.M)
Last changed    : 11/2/2010 6:39:25 PM by dxq
                  (modified after loading)
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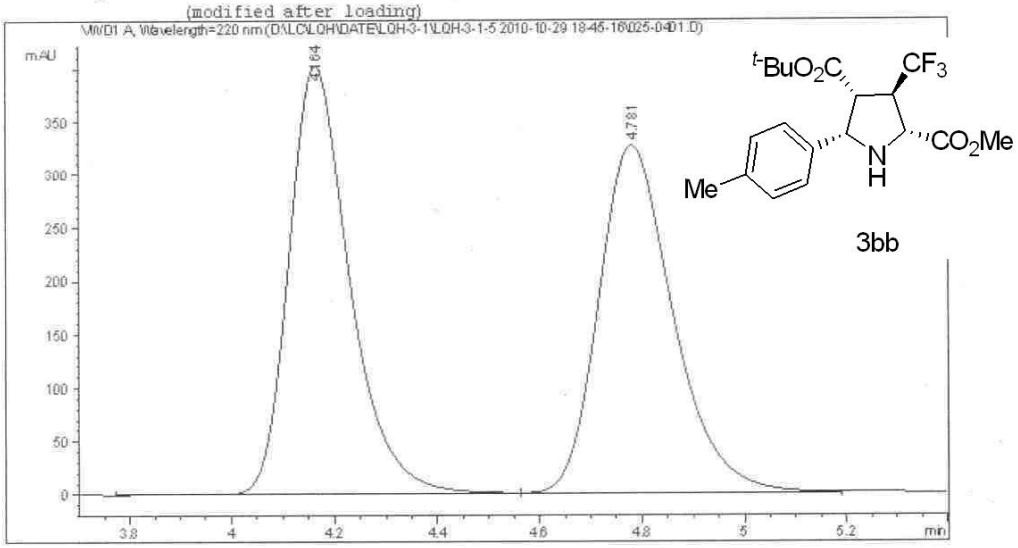


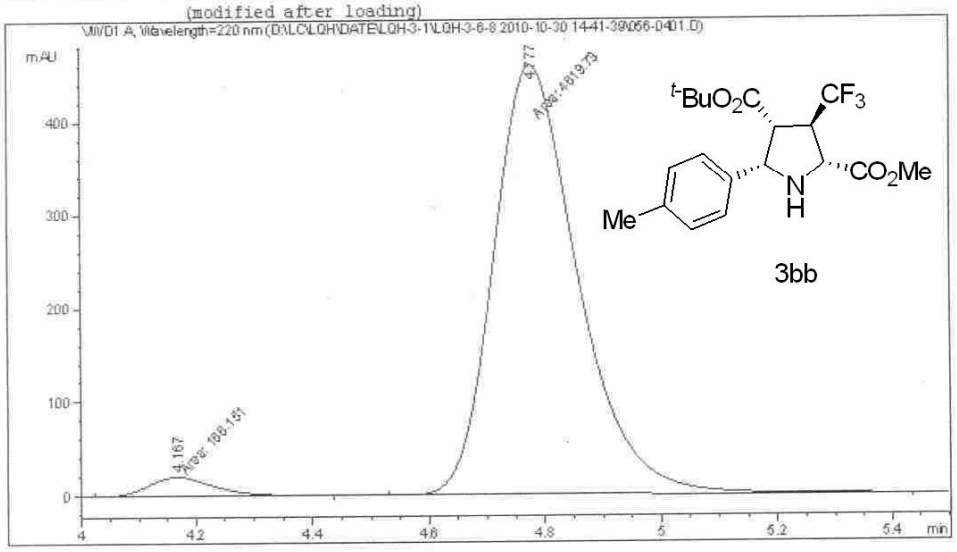
3bb

```
==========================================================================
                        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   4.164 VV    0.1249 3304.16870  402.28793  49.9972
   2   4.781 VV    0.1544 3304.54370  326.61957  50.0028

Totals :                  6608.71240  728.90750

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

ta File D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\056-0401.D
mple Name: LQH-3-8

Acq. Operator : DXQ
Acq. Instrument : Instrument 1
Injection Date : 10/30/2010 3:17:15 PM
Seq. Line : 4
Location : Vial 56
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\ASH-2-98-1ML-220NM-10MIN.M
Last changed : 9/29/2010 2:44:36 PM by DXQ
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\056-0401.D\DA.M (ASH-2-98-1ML-220NM-10MIN.M)
Last changed : 11/8/2010 8:54:01 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=220 nm

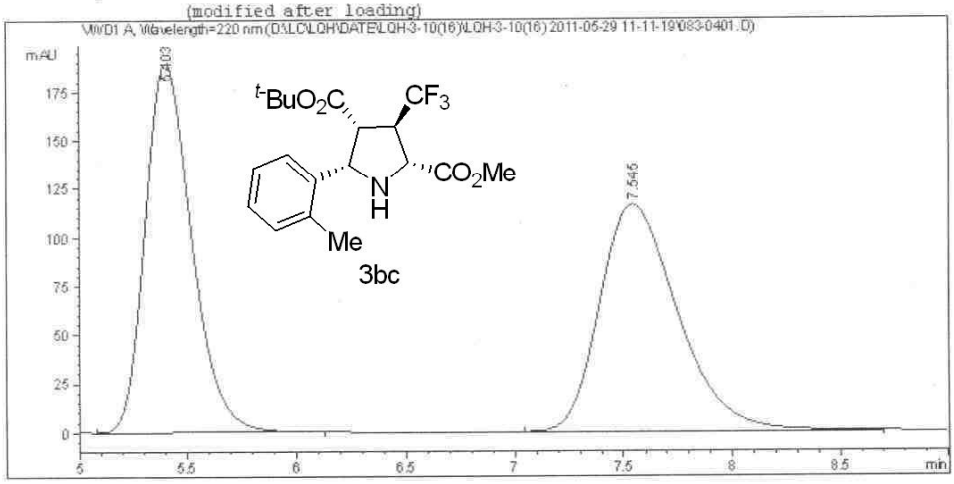
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.167	MM	0.1336	166.15074	20.73029	3.4717
2	4.777	MM	0.1676	4619.73438	459.49329	96.5283
Totals :				4785.88512	480.22358	

nstrument 1 11/8/2010 8:54:07 PM THL

Page 1 of 1

a File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\083-0401.D
ple Name: LQH-3-10B

Acq. Operator	: LTL	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 83
Injection Date	: 5/29/2011 11:56:32 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-220NM-15MIN.M		
Last changed	: 5/27/2011 12:01:57 PM by THL		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\083-0401.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)		
Last changed	: 5/29/2011 3:17:30 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

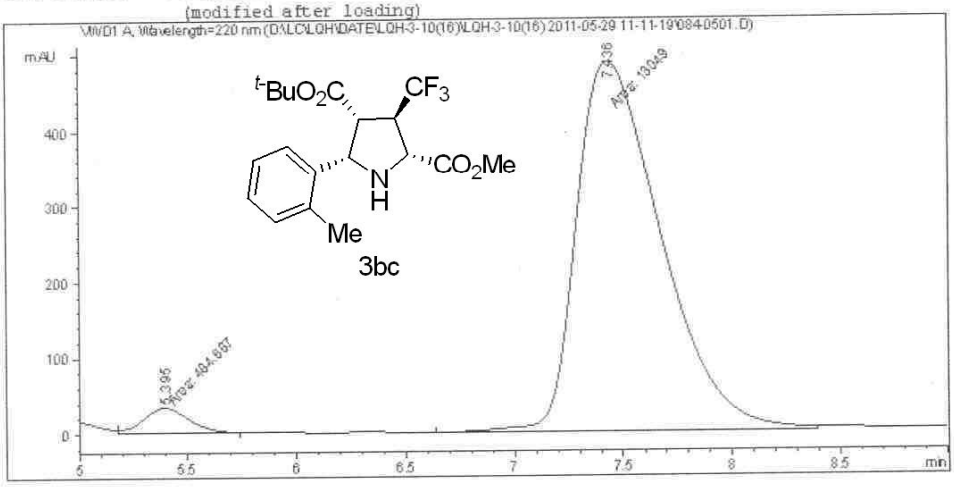
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	5.403	BB	0.2324	2852.84888	189.49812	49.8339
2	7.545	BB	0.3820	2871.87134	115.99537	50.1661
Totals :				5724.72021	305.49349	

*** End of Report ***

strument 1 5/29/2011 3:17:35 PM LTL Page 1 of 1

a File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\084-0501.D
ple Name: LQH-3-16B

Acq. Operator	: LTL	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 84
Injection Date	: 5/29/2011 12:12:59 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-220NM-15MIN.M		
Last changed	: 5/27/2011 12:01:57 PM by THL		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\084-0501.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)		
Last changed	: 5/29/2011 3:21:07 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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.395	MM	0.2463	484.68695	32.79442	3.5813
2	7.436	MM	0.4449	1.30490e4	488.84824	96.4187
Totals :				1.35337e4	521.64265	

*** End of Report ***

strument 1 5/29/2011 3:21:12 PM LTL Page 1 of 1

a File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\087-0801.D
ple Name: LQH-3-10D

```
Acq. Operator   : LTL                              Seq. Line :   8
Acq. Instrument : Instrument 1                     Location : Vial 87
Injection Date  : 5/29/2011 1:02:41 PM                 Inj :   1
                                                   Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-
                  220NM-15MIN.M
Last changed    : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\087-0801.D\
                  DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed    : 5/29/2011 3:25:47 PM by LTL
                  (modified after loading)
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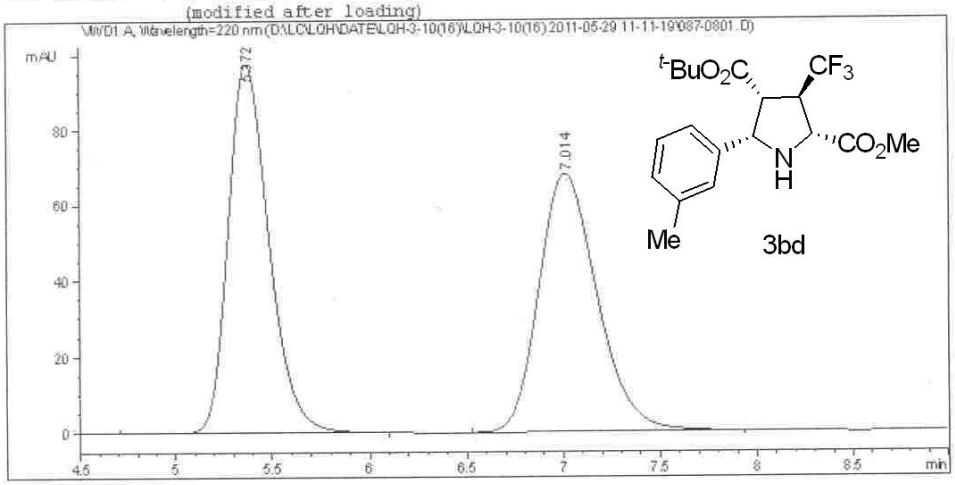


```
=====================================================================
                      Area Percent Report
=====================================================================

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   5.372 BB    0.2252 1420.12427   97.52248  49.9521
   2   7.014 BB    0.3243 1422.84985   67.96291  50.0479

Totals :                  2842.97412  165.48539

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

ata File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\088-0901.D
ample Name: LQH-3-16D

```
==========================================================================
Acq. Operator   : LTL                                 Seq. Line :   9
Acq. Instrument : Instrument 1                        Location : Vial 88
Injection Date  : 5/29/2011 1:19:07 PM                    Inj :   1
                                                    Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-
                  220NM-15MIN.M
Last changed    : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\088-0901.D\
                  DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed    : 5/29/2011 3:27:28 PM by LTL
                  (modified after loading)
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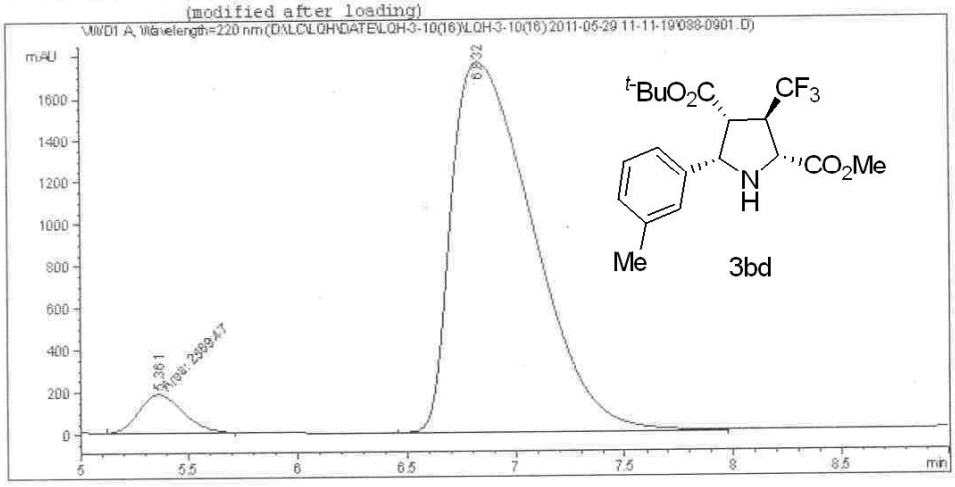


```
==========================================================================
                        Area Percent Report
==========================================================================

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   5.361 MM    0.2363 2569.47046  181.25473   5.2680
   2   6.832 VV    0.4193 4.62060e4  1763.39197  94.7320

Totals :                  4.87755e4  1944.64670

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

ata File D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\026-0501.D
ample Name: LQH-3-5

```
Acq. Operator   : DXQ                                Seq. Line :   5
Acq. Instrument : Instrument 1                       Location : Vial 26
Injection Date  : 10/29/2010 7:40:57 PM                 Inj :   1
                                                    Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\ASH-2-98-1ML-220NM.M
Last changed    : 10/29/2010 7:53:32 PM by DXQ
                  (modified after loading)
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\026-0501.D\DA.M (ASH-
                  2-98-1ML-220NM.M)
Last changed    : 11/2/2010 6:40:52 PM by dxq
                  (modified after loading)
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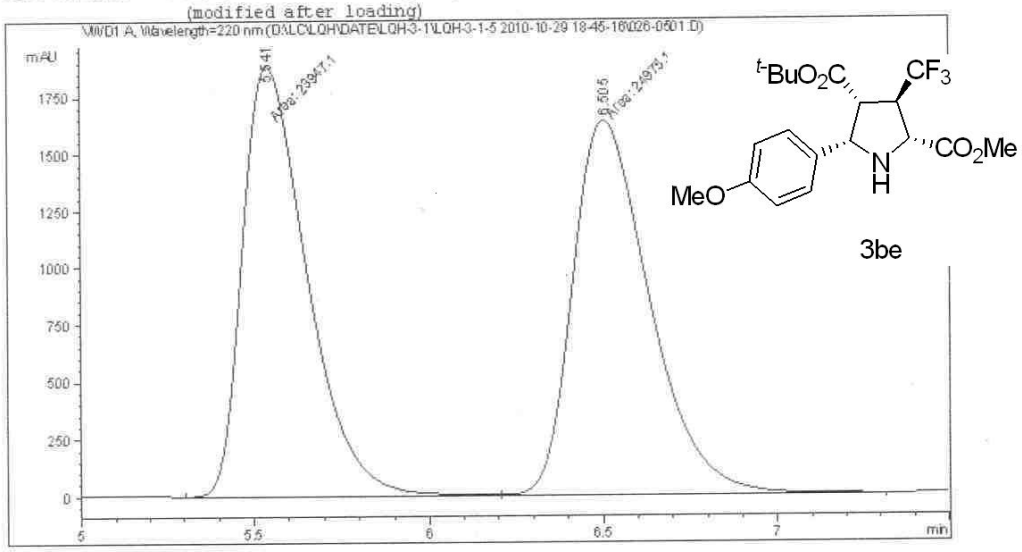


3be

```
                          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

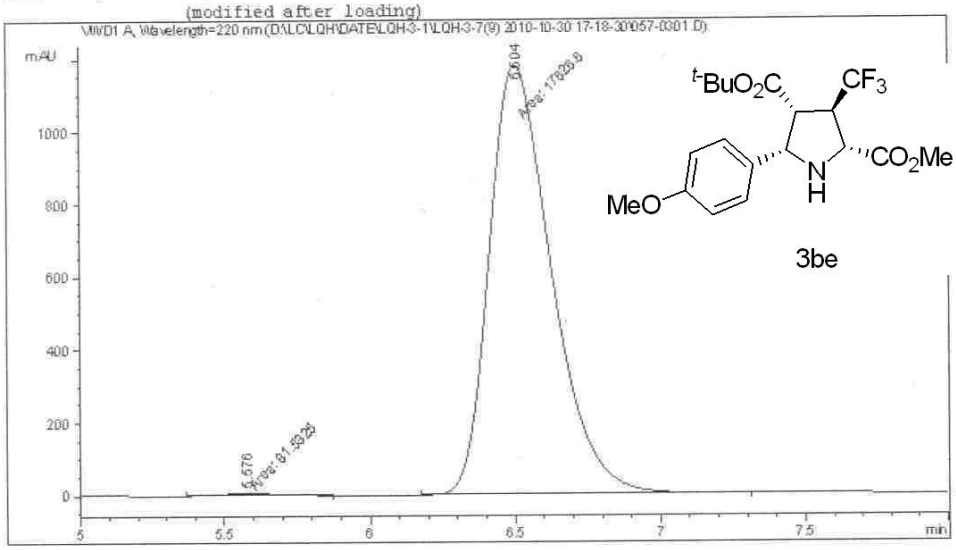
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]     %
----|-------|----|-------|----------|----------|--------|
   1   5.541 MF    0.2122 2.39471e4  1881.01147  48.9494
   2   6.505 FM    0.2538 2.49751e4  1640.17615  51.0506

Totals :                  4.89223e4  3521.18762
```

Instrument 1 11/2/2010 6:40:56 PM dxq Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\057-0301.D
ample Name: LQH-3-9

```
Acq. Operator   : DXQ                          Seq. Line :   3
Acq. Instrument : Instrument 1                  Location : Vial 57
Injection Date  : 10/30/2010 5:42:29 PM            Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\ASH-2-98-1ML-220NM-
                  10MIN.M
Last changed    : 9/29/2010 2:44:36 PM by DXQ
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\057-0301.D\DA.M (
                  ASH-2-98-1ML-220NM-10MIN.M)
Last changed    : 10/30/2010 6:03:54 PM by thl
                  (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   5.576 MM    0.1902   81.53249    7.14318   0.4604
   2   6.504 MM    0.2489 1.76266e4  1180.07593  99.5396

Totals :                  1.77081e4  1187.21911

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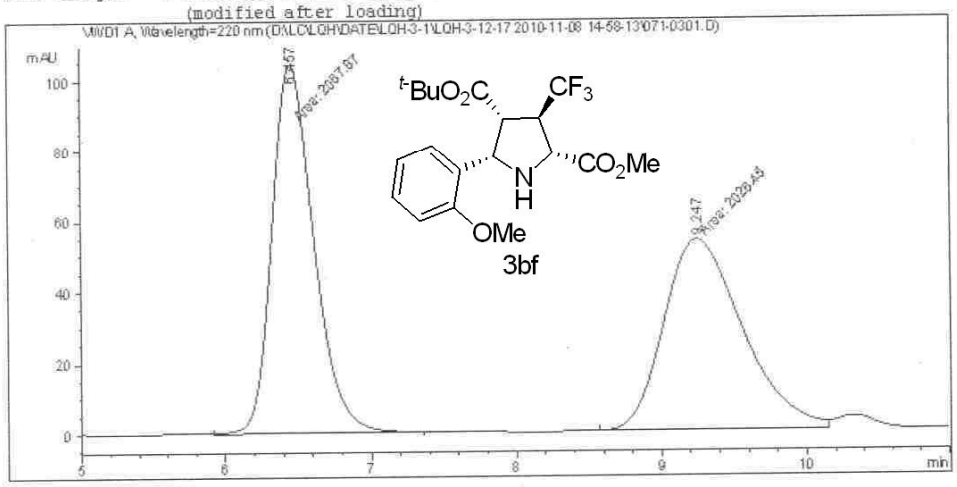

Instrument 1 10/30/2010 6:03:59 PM thl Page 1 of 1

ta File D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\071-0301.D
mple Name: LQH-3-12A

Acq. Operator	: DXQ	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/8/2010 3:27:14 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\ASH-2-98-1ML-220NM.M		
Last changed	: 11/8/2010 3:45:08 PM by DXQ (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\071-0301.D\DA.M (ASH-2-98-1ML-220NM.M)		
Last changed	: 11/8/2010 5:31:38 PM by TMC (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

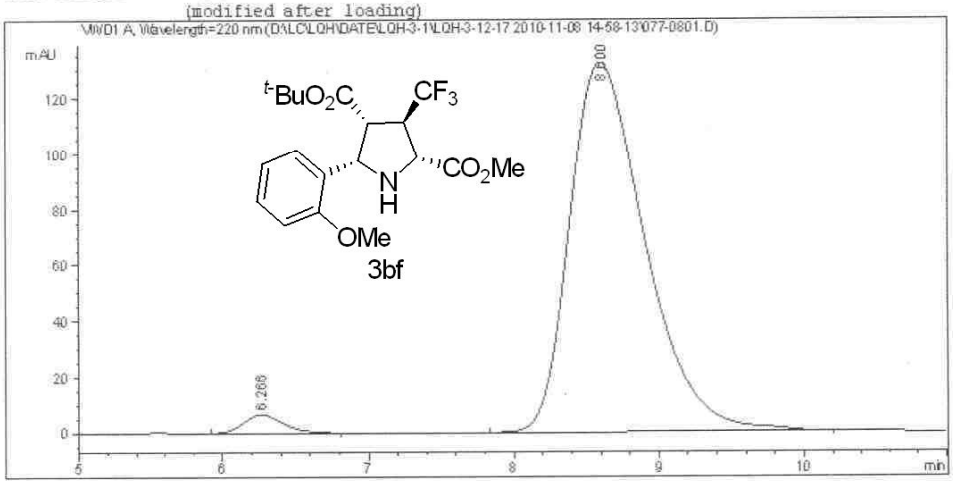
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.457	FM	0.3305	2067.86523	104.27755	50.5057
2	9.247	MF	0.6288	2026.45288	53.71293	49.4943
Totals :				4094.31812	157.99048	

*** End of Report ***

Data File D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\077-0801.D
Sample Name: LQH-3-17B

Acq. Operator	: DXQ	Seq. Line	: 8
Acq. Instrument	: Instrument 1	Location	: Vial 77
Injection Date	: 11/8/2010 4:46:08 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\ASH-2-98-1ML-220NM-15MIN.M
Last changed : 11/8/2010 4:59:47 PM by DXQ
(modified after loading)
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\077-0801.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed : 11/8/2010 5:35:28 PM by TMC
(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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.266	BB	0.2898	125.38615	6.65074	2.5636
2	8.600	BB	0.5548	4765.54150	132.57668	97.4364
Totals :				4890.92765	139.22741	

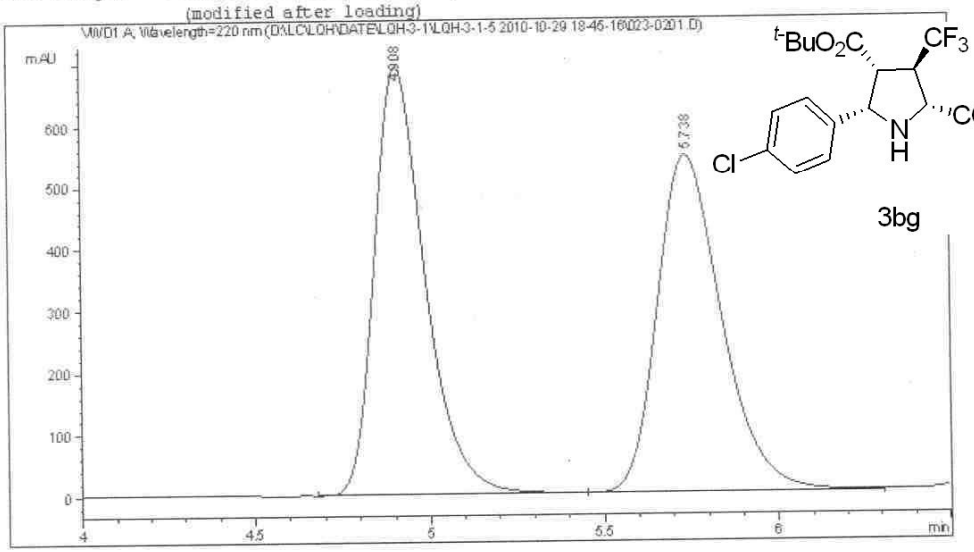
*** End of Report ***

Instrument 1 11/8/2010 5:35:32 PM TMC Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\023-0201.D
ample Name: LQH-3-1

Acq. Operator : DXQ
Acq. Instrument : Instrument 1
Injection Date : 10/29/2010 6:58:28 PM
Seq. Line : 2
Location : Vial 23
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\ASH-2-98-1ML-220NM.M
Last changed : 10/29/2010 7:13:41 PM by DXQ
(modified after loading)
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\023-0201.D\DA.M (ASH-2-98-1ML-220NM.M)
Last changed : 11/2/2010 6:35:19 PM by dxq
(modified after loading)



3bg

Area Percent Report

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

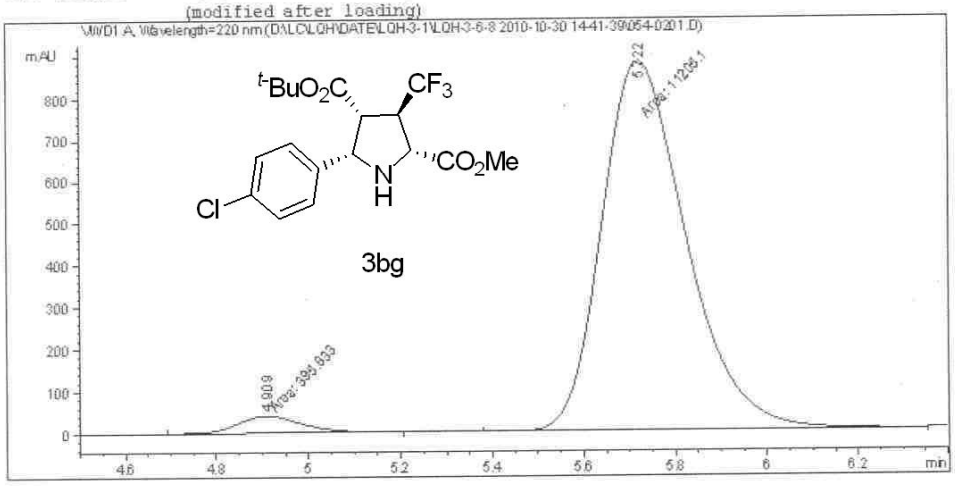
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.908	VV	0.1532	6944.77637	693.40997	49.9862
2	5.738	VV	0.1955	6948.62305	544.48187	50.0138
Totals :				1.38934e4	1237.89185	

Instrument 1 11/2/2010 6:35:29 PM dxq

Data File D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\054-0201.D
Sample Name: LQH-3-6

Acq. Operator : DXQ
Seq. Line : 2
Acq. Instrument : Instrument 1
Location : Vial 54
Injection Date : 10/30/2010 2:54:21 PM
Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\ASH-2-98-1ML-220NM-10MIN.M
Last changed : 9/29/2010 2:44:36 PM by DXQ
Analysis Method : D:\LC\LQH\DATE\LQH-3-1\LQH-3-6-8 2010-10-30 14-41-39\054-0201.D\DA.M (ASH-2-98-1ML-220NM-10MIN.M)
Last changed : 11/8/2010 8:53:03 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=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.909	MM	0.1628	395.83264	40.51372	3.4121
2	5.722	MM	0.2119	1.12051e4	881.12811	96.5879
Totals :				1.16010e4	921.64183	

*** End of Report ***

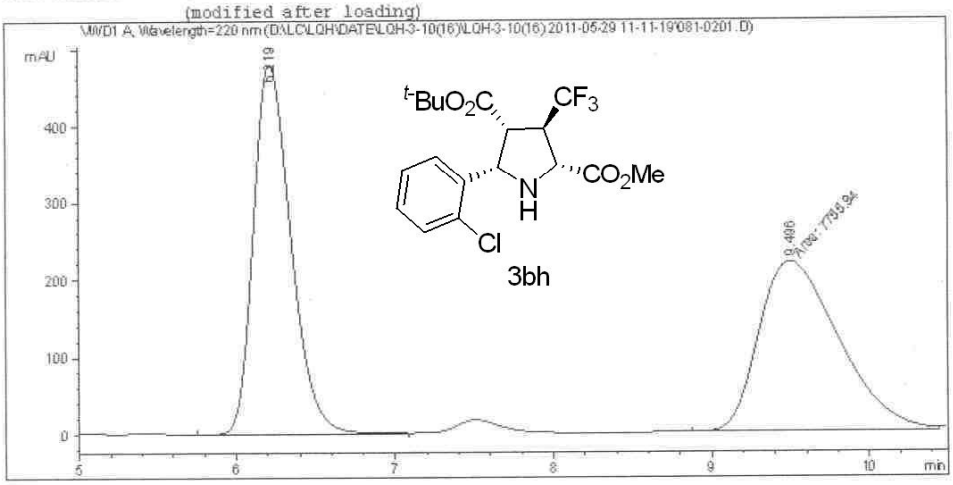
Instrument 1 11/8/2010 8:53:10 PM THL

Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\081-0201.D
Sample Name: LQH-3-10A

Acq. Operator	: LTL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 81
Injection Date	: 5/29/2011 11:23:38 AM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-220NM-15MIN.M
Last changed : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\081-0201.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed : 5/29/2011 3:13:44 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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.219	VB	0.2524	7812.69580	480.23087	50.1823
2	9.496	MM	0.5888	7755.93555	219.52803	49.8177
Totals :				1.55686e4	699.75890	

*** End of Report ***

ata File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\082-0301.D
ample Name: LQH-3-16A

```
==========================================================================
Acq. Operator   : LTL                              Seq. Line :   3
Acq. Instrument : Instrument 1                     Location : Vial 82
Injection Date  : 5/29/2011 11:40:06 AM               Inj :   1
                                                   Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-
                  220NM-15MIN.M
Last changed    : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\082-0301.D\
                  DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed    : 5/29/2011 3:16:31 PM by LTL
                  (modified after loading)
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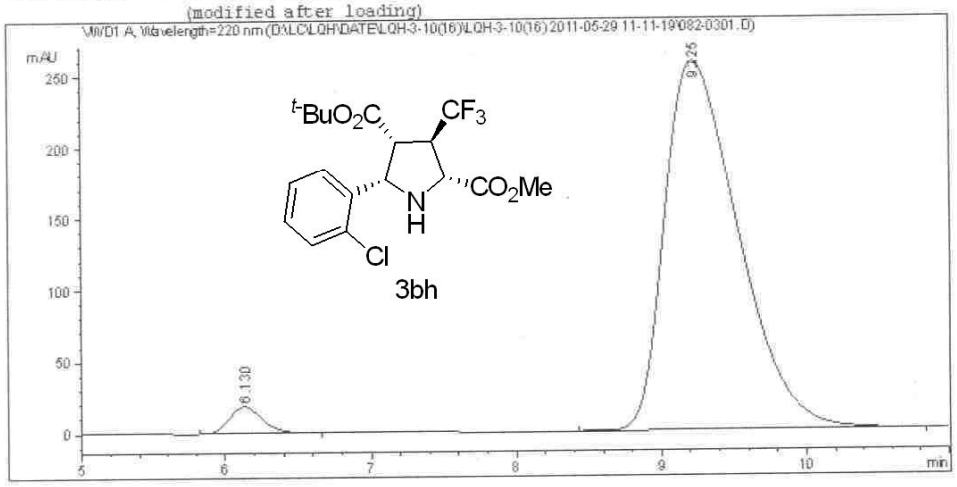


```
==========================================================================
                        Area Percent Report
==========================================================================

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   6.130 BB    0.2438  295.05249   18.69994   3.1433
   2   9.225 BB    0.5489 9091.71289  258.31909  96.8567

Totals :                  9386.76538  277.01903

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

Instrument 1 5/29/2011 3:16:36 PM LTL Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\085-0601.D
ample Name: LQH-3-10C

```
==========================================================================
Acq. Operator   : LTL                                 Seq. Line :   6
Acq. Instrument : Instrument 1                        Location : Vial 85
Injection Date  : 5/29/2011 12:29:31 PM                    Inj :   1
                                                     Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-
                  220NM-15MIN.M
Last changed    : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\085-0601.D\
                  DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed    : 5/29/2011 3:22:20 PM by LTL
                  (modified after loading)
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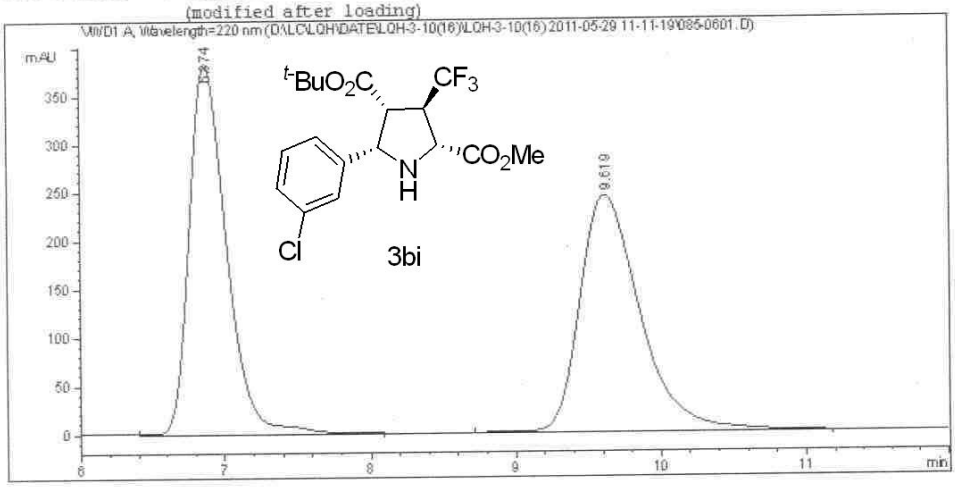


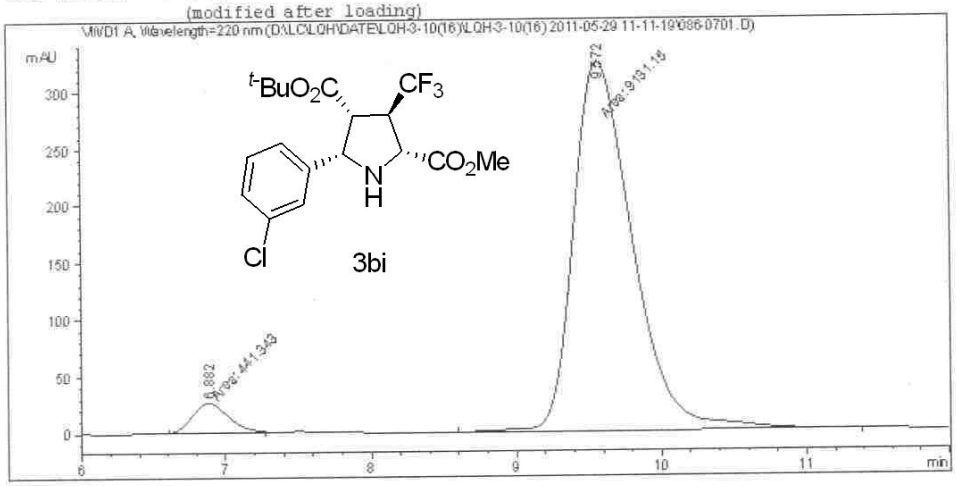

```
==========================================================================
                        Area Percent Report
==========================================================================

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   6.874 VB    0.2750 6820.43066  382.43506  49.4482
   2   9.619 BB    0.4417 6972.64600  243.60837  50.5518

Totals :                  1.3793le4   626.04343

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

Instrument 1 5/29/2011 3:22:24 PM LTL Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\086-0701.D
Sample Name: LQH-3-16C

Acq. Operator : LTL
Acq. Instrument : Instrument 1
Injection Date : 5/29/2011 12:46:02 PM
Seq. Line : 7
Location : Vial 86
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\ASH-2-98-1ML-220NM-15MIN.M
Last changed : 5/27/2011 12:01:57 PM by THL
Analysis Method : D:\LC\LQH\DATE\LQH-3-10(16)\LQH-3-10(16) 2011-05-29 11-11-19\086-0701.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed : 5/29/2011 3:24:53 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

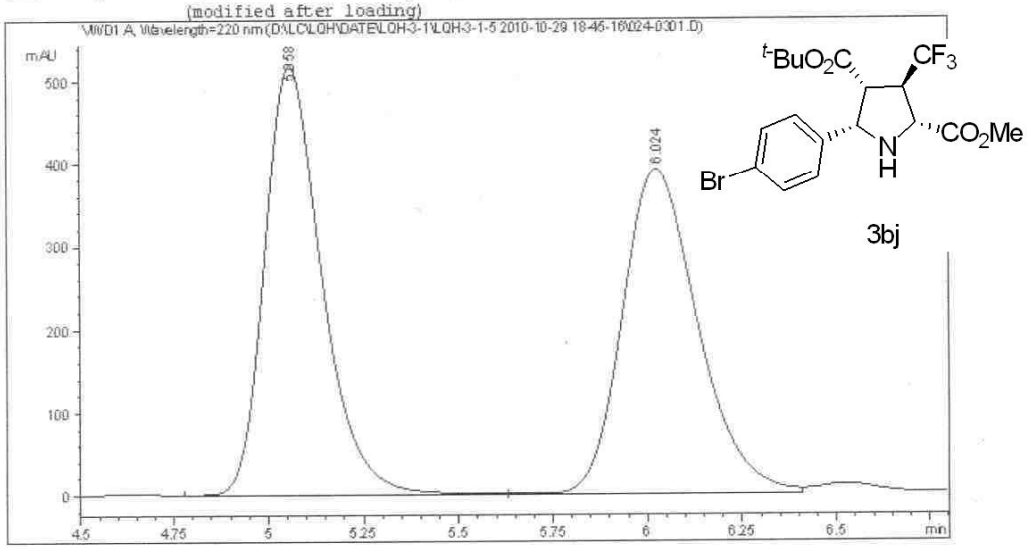
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.882	MM	0.2847	441.34271	25.83364	4.6105
2	9.572	MM	0.4690	9131.14941	324.51608	95.3895
Totals :				9572.49213	350.34972	

*** End of Report ***

Instrument 1 5/29/2011 3:24:57 PM LTL Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\024-0301.D
Sample Name: LQH-3-2

Acq. Operator	: DXQ	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 24
Injection Date	: 10/29/2010 7:15:17 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\ASH-2-98-1ML-220NM.M		
Last changed	: 10/29/2010 7:28:10 PM by DXQ (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-1-5 2010-10-29 18-45-16\024-0301.D\DA.M (ASH-2-98-1ML-220NM.M)		
Last changed	: 11/2/2010 6:38:05 PM by dxq (modified after loading)		



3bj

Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

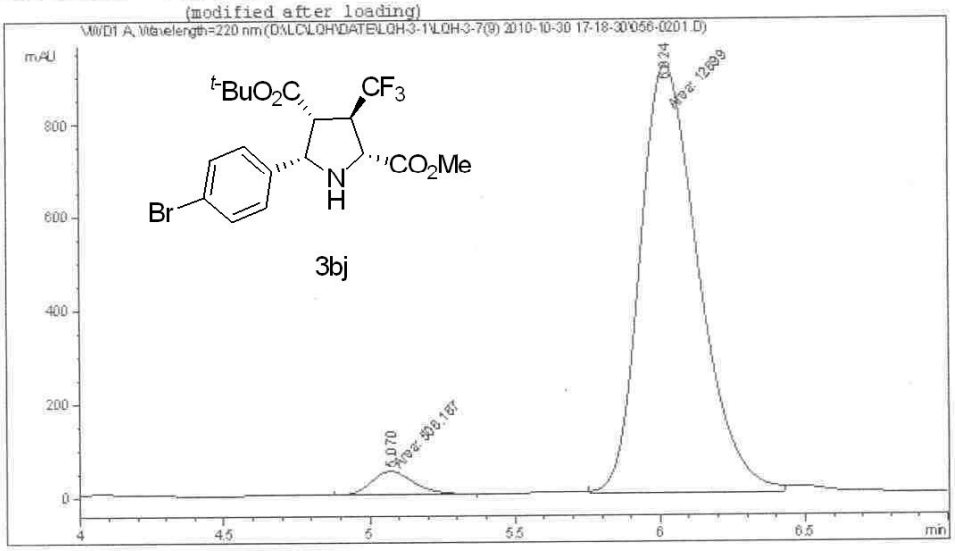
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.058	VV	0.1584	5423.21045	518.48712	50.0595
2	6.024	VV	0.2123	5410.32178	391.31061	49.9405
Totals :				1.08335e4	909.79773	

ata File D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\056-0201.D
ample Name: LQH-3-7

Acq. Operator	: DXQ	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 56
Injection Date	: 10/30/2010 5:31:13 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\ASH-2-98-1ML-220NM-10MIN.M		
Last changed	: 9/29/2010 2:44:36 PM by DXQ		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-7(9) 2010-10-30 17-18-30\056-0201.D\DA.M (ASH-2-98-1ML-220NM-10MIN.M)		
Last changed	: 10/30/2010 6:06:05 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=220 nm

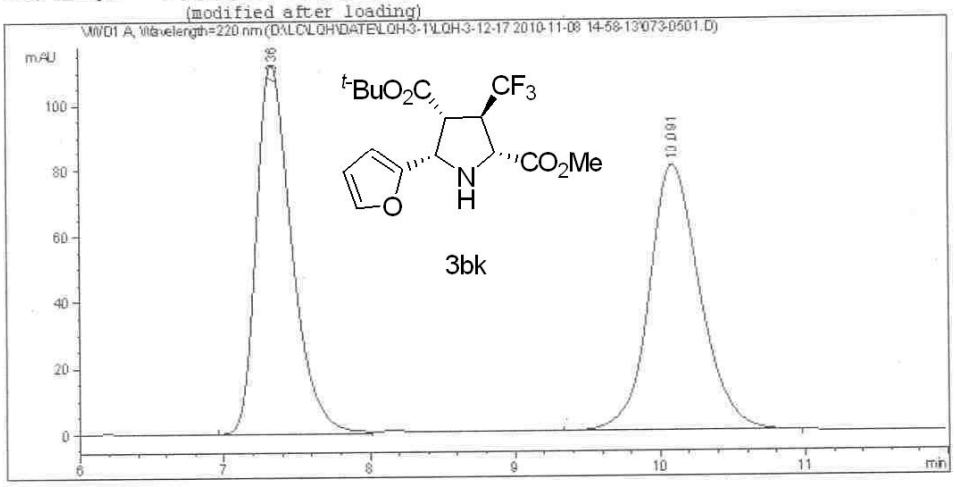
Peak #	RetTime [min]	Type	Width [min]	Area mAU * s	Height [mAU]	Area %
1	5.070	MM	0.1683	506.16711	50.11565	3.8331
2	6.024	MF	0.2312	1.26990e4	915.46576	96.1669
Totals :				1.32052e4	965.58141	

Instrument 1 10/30/2010 6:06:09 PM thl

Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\073-0501.D
Sample Name: LQH-3-13A

Acq. Operator	: DXQ	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 73
Injection Date	: 11/8/2010 4:01:37 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\ASH-2-98-1ML-220NM.M		
Last changed	: 11/8/2010 4:13:06 PM by DXQ (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\073-0501.D\DA.M (ASH-2-98-1ML-220NM.M)		
Last changed	: 11/8/2010 5:33:08 PM by TMC (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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.336	BB	0.2566	1881.96692	112.34785	49.0302
2	10.091	BB	0.3680	1956.41455	80.50997	50.9698
Totals :				3838.38147	192.85782	

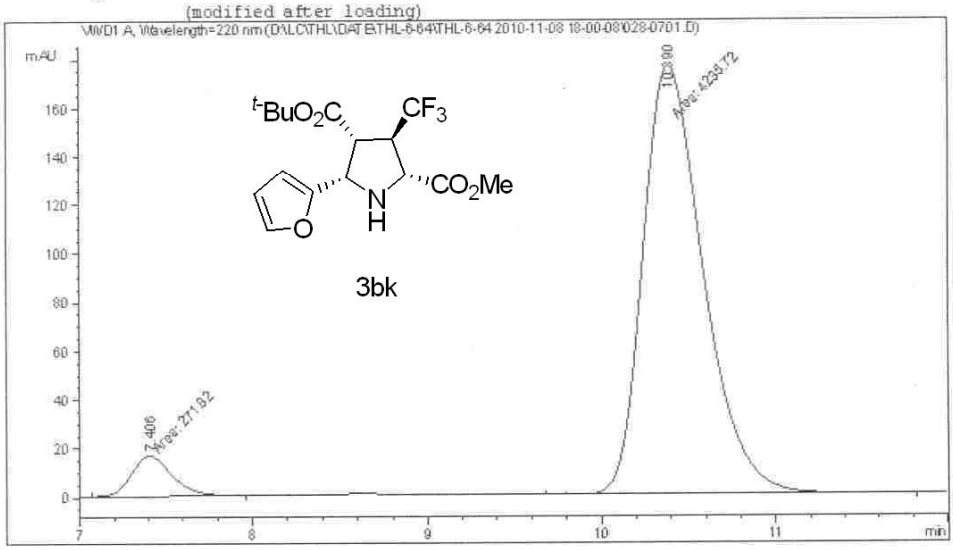
*** End of Report ***

Instrument 1 11/8/2010 5:33:12 PM TMC Page 1 of 1

ata File D:\LC\THL\DATE\THL-6-64\THL-6-64 2010-11-08 18-00-08\028-0701.D
ample Name: LQH-3-17D

Acq. Operator	: DXQ	Seq. Line	: 7
Acq. Instrument	: Instrument 1	Location	: Vial 28
Injection Date	: 11/8/2010 10:18:05 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\THL\DATE\THL-6-64\THL-6-64 2010-11-08 18-00-08\ASH-2-98-1ML-220NM-15MIN.M
Last changed : 9/29/2010 2:43:57 PM by DXQ
Analysis Method : D:\LC\THL\DATE\THL-6-64\THL-6-64 2010-11-08 18-00-08\028-0701.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)
Last changed : 11/9/2010 8:14:55 AM by THL
(modified after loading)



Area Percent Report

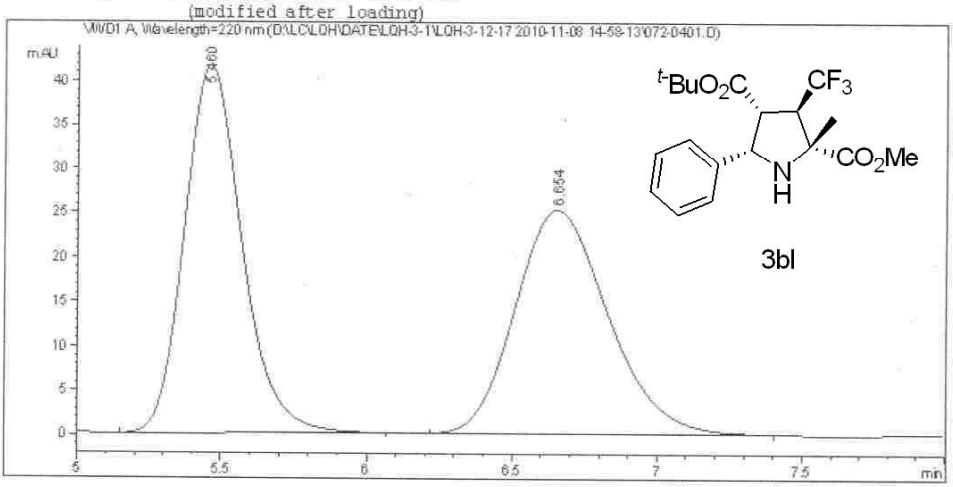
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.406	MM	0.2756	271.82016	16.43653	6.0303
2	10.390	MM	0.4003	4235.71973	176.34241	93.9697
Totals :				4507.53989	192.77894	

Data File D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\072-0401.D
Sample Name: LQH-3-12B

Acq. Operator	: DXQ	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 72
Injection Date	: 11/8/2010 3:46:36 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\ASH-2-98-1ML-220NM.M		
Last changed	: 11/8/2010 4:00:06 PM by DXQ (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-12-17 2010-11-08 14-58-13\072-0401.D\DA.M (ASH-2-98-1ML-220NM.M)		
Last changed	: 11/8/2010 5:32:23 PM by TMC (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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.460	BB	0.2116	573.44189	41.63863	50.4344
2	6.654	BB	0.3454	563.56415	25.19586	49.5656
Totals :				1137.00604	66.83449	

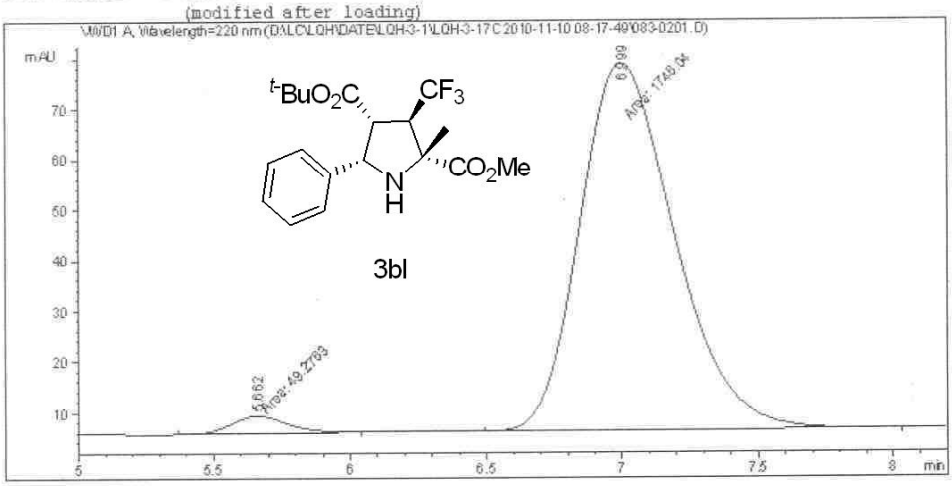
*** End of Report ***

Instrument 1 11/8/2010 5:32:27 PM TMC

Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-1\LQH-3-17C 2010-11-10 08-17-49\083-0201.D
ample Name: LQH-3-17C

Acq. Operator	: TMC	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 83
Injection Date	: 11/10/2010 8:30:11 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-17C 2010-11-10 08-17-49\ASH-2-98-1ML-220NM-15MIN.M		
Last changed	: 9/29/2010 2:43:57 PM by DXQ		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-1\LQH-3-17C 2010-11-10 08-17-49\083-0201.D\DA.M (ASH-2-98-1ML-220NM-15MIN.M)		
Last changed	: 11/10/2010 6:22:09 PM by THL (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

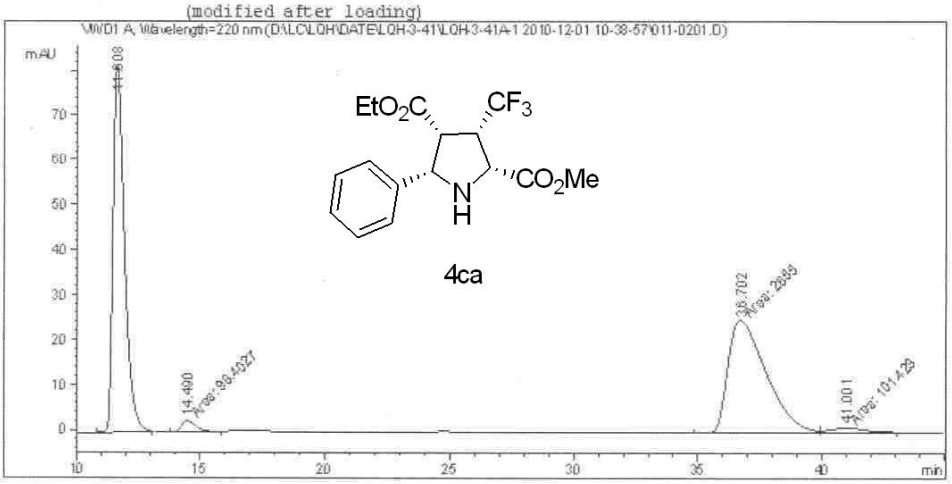
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.662	MM	0.2396	49.27632	3.42749	2.7447
2	6.999	MM	0.4006	1746.03809	72.64720	97.2553
Totals :				1795.31441	76.07469	

*** End of Report ***

ata File D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\011-0201.D
ample Name: LQH-3-41A-1

Acq. Operator	: dxq	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 11
Injection Date	: 12/1/2010 10:51:38 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\ASH-5-95-1ML-220NM.M		
Last changed	: 12/1/2010 11:38:10 AM by dxq (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\011-0201.D\DA.M (ASH-5-95-1ML-220NM.M)		
Last changed	: 1/20/2011 5:32:33 PM by THL-7-95-97 (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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.608	BB	0.4921	2650.05811	81.98250	48.1402
2	14.490	MM	0.6489	98.40275	2.52749	1.7876
3	36.702	MF	1.7722	2654.99585	24.96951	48.2299
4	41.001	FM	1.6090	101.42349	1.05059	1.8424
Totals :				5504.88020	110.53008	

ata File D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\014-0801.D
ample Name: LQH-3-42A

```
=====================================================================
Acq. Operator   : dxq                          Seq. Line :   8
Acq. Instrument : Instrument 1                  Location : Vial 14
Injection Date  : 12/1/2010 1:09:15 PM             Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\ASH-5-95-1ML-
                  220NM-50MIN.M
Last changed    : 12/1/2010 11:45:27 AM by dxq
Analysis Method : D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\014-0801.D\DA.M (
                  ASH-5-95-1ML-220NM-50MIN.M)
Last changed    : 1/20/2011 7:07:55 PM by THL-7-95-97
                  (modified after loading)
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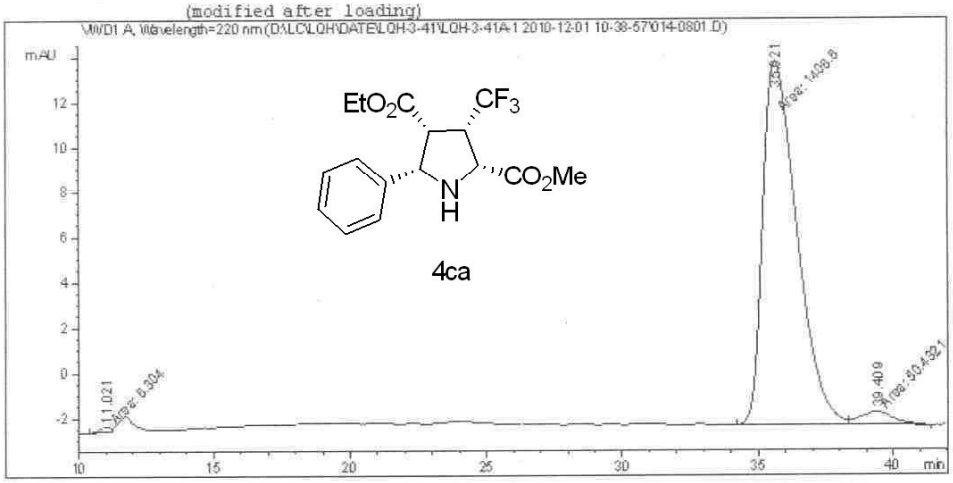



```
=====================================================================
                        Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=220 nm

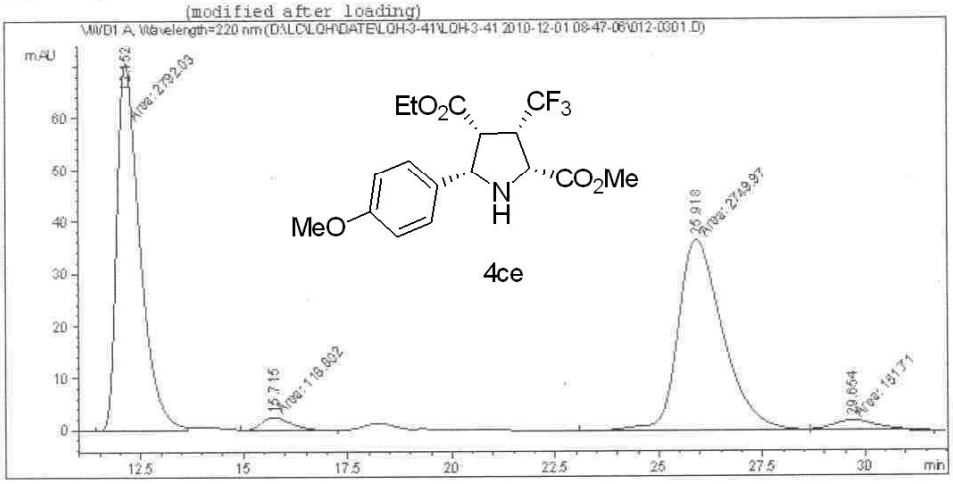
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.021 MF    0.4790    6.30400 2.19324e-1   0.4308
   2  35.621 MF    1.4566 1406.60132   16.09430  96.1228
   3  39.409 FM    1.4088   50.43205 5.96647e-1   3.4464

Totals :                  1463.33737   16.91027

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

Data File D:\LC\LQH\DATE\LQH-3-41\LQH-3-41 2010-12-01 08-47-06\012-0301.D
Sample Name: LQH-3-41B

Acq. Operator	: dxq	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 12
Injection Date	: 12/1/2010 9:32:10 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-41\LQH-3-41 2010-12-01 08-47-06\ASH-10-90-1ML-220NM.M		
Last changed	: 12/1/2010 10:03:10 AM by dxq (modified after loading)		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-41\LQH-3-41 2010-12-01 08-47-06\012-0301.D\DA.M (ASH-10-90-1ML-220NM.M)		
Last changed	: 1/20/2011 5:36:40 PM by THL-7-95-97 (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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.152	MF	0.6584	2792.02637	70.67298	48.0348
2	15.715	MM	0.8053	118.80157	2.45881	2.0439
3	25.918	MF	1.2493	2749.96582	36.68673	47.3112
4	29.654	FM	1.3164	151.70990	1.92081	2.6101
Totals :				5812.50365	111.73933	

*** End of Report ***

Instrument 1 1/20/2011 5:36:44 PM THL-7-95-97 Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\013-0701.D
ample Name: LQH-3-42B

```
==========================================================================
Acq. Operator   : dxq                          Seq. Line :   7
Acq. Instrument : Instrument 1                  Location : Vial 13
Injection Date  : 12/1/2010 12:28:11 PM            Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\ASH-10-90-220NM-
                  1ML-40MIN.M
Last changed    : 8/31/2010 11:21:45 AM by LTL
Analysis Method : D:\LC\LQH\DATE\LQH-3-41\LQH-3-41A-1 2010-12-01 10-38-57\013-0701.D\DA.M (
                  ASH-10-90-220NM-1ML-40MIN.M)
Last changed    : 1/20/2011 7:13:58 PM by THL-7-95-97
                  (modified after loading)
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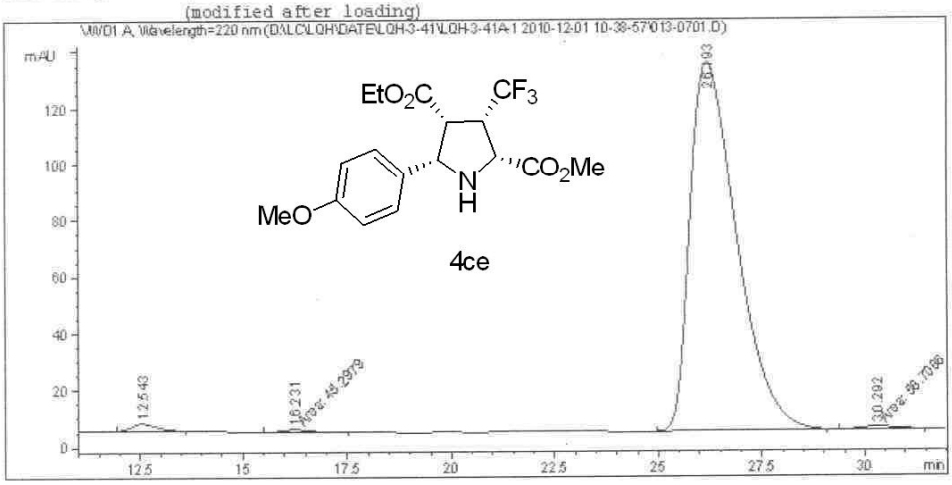
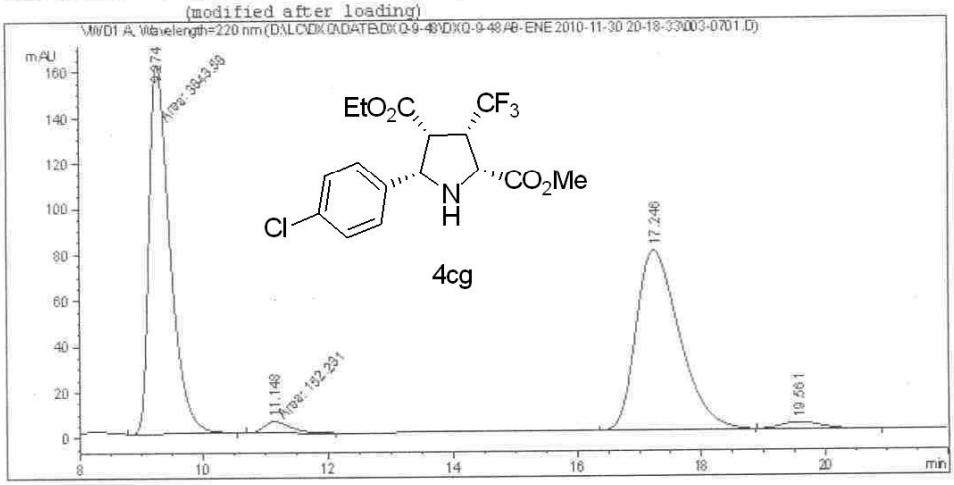



```
==========================================================================
                        Area Percent Report
==========================================================================

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  12.543 BB    0.5997  102.32714    2.44828   0.9882
   2  16.231 MM    0.8015   45.29786 9.41941e-1   0.4375
   3  26.193 BB    1.1999 1.01486e4   130.51140  98.0074
   4  30.292 MM    1.0206   58.70856 9.58704e-1   0.5670

Totals :                  1.03549e4   134.86033

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

Instrument 1 1/20/2011 7:14:04 PM THL-7-95-97 Page 1 of 1

Data File D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\003-0701.D
Sample Name: LQH-3-37

Acq. Operator : dxq
Acq. Instrument : Instrument 1
Injection Date : 11/30/2010 11:08:20 PM
Seq. Line : 7
Location : Vial 3
Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\ASH-10-90-220NM-1ML-40MIN.M
Last changed : 8/31/2010 11:21:45 AM by LTL
Analysis Method : D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\003-0701.D\DA.M (ASH-10-90-220NM-1ML-40MIN.M)
Last changed : 12/1/2010 11:53:38 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=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.274	MM	0.3967	3843.58301	161.48431	48.1590
2	11.148	MM	0.5021	152.23082	5.05339	1.9074
3	17.246	BB	0.7537	3827.99316	78.51288	47.9636
4	19.561	BB	0.7067	157.22655	3.25235	1.9700
Totals :				7981.03354	248.30293	

*** End of Report ***

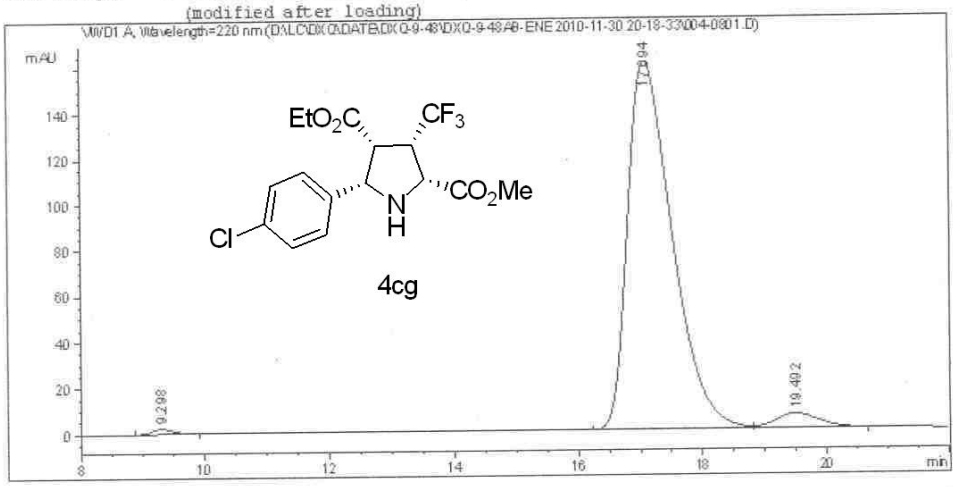
Instrument 1 12/1/2010 11:53:43 AM dxq

Page 1 of 1

Data File D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\004-0801.D
Sample Name: LQH-3-39

Acq. Operator : dxq
Acq. Instrument : Instrument 1
Injection Date : 11/30/2010 11:49:52 PM
Seq. Line : 8
Location : Vial 4
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\ASH-10-90-220NM-1ML-40MIN.M
Last changed : 8/31/2010 11:21:45 AM by LTL
Analysis Method : D:\LC\DXQ\DATE\DXQ-9-48\DXQ-9-48AB-ENE 2010-11-30 20-18-33\004-0801.D\DA.M (ASH-10-90-220NM-1ML-40MIN.M)
Last changed : 12/1/2010 11:57:02 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=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.298	BB	0.3579	56.63172	2.36617	0.6772
2	17.094	BB	0.7624	7990.20801	161.40883	95.5413
3	19.492	BB	0.7361	316.25586	6.45516	3.7816
Totals :				8363.09559	170.23015	

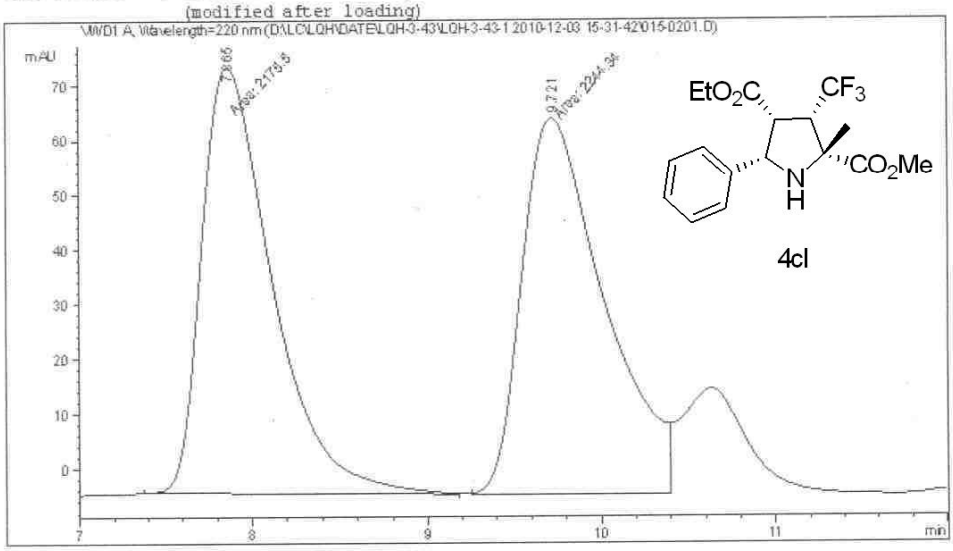
*** End of Report ***

Instrument 1 12/1/2010 11:57:07 AM dxq

Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-43\LQH-3-43-1 2010-12-03 15-31-42\015-0201.D
Sample Name: LQH-3-43-1

Acq. Operator	: dxq	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 15
Injection Date	: 12/3/2010 3:44:08 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-43\LQH-3-43-1 2010-12-03 15-31-42\ASH-2-98-1ML-220NM.M		
Last changed	: 11/1/2010 2:36:30 PM by DXQ		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-43\LQH-3-43-1 2010-12-03 15-31-42\015-0201.D\DA.M (ASH-2-98-1ML-220NM.M)		
Last changed	: 12/4/2010 8:57:22 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=220 nm

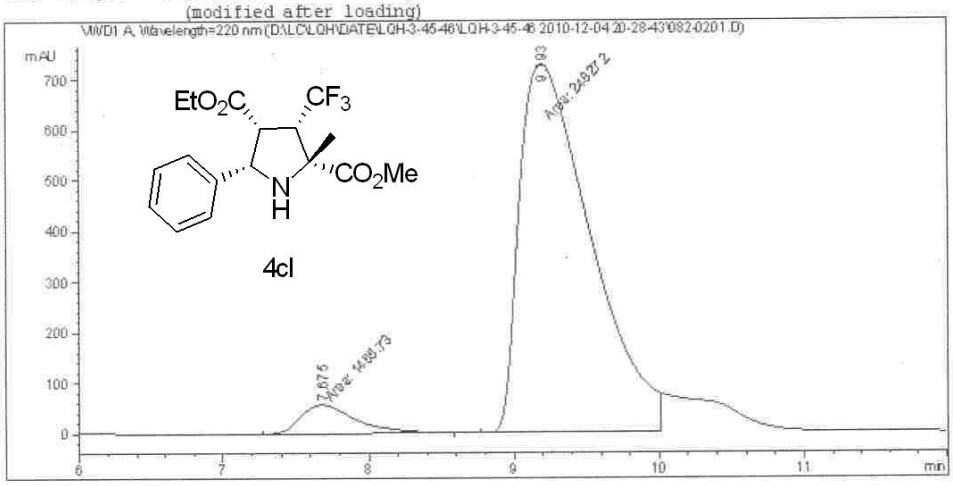
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.865	MM	0.4659	2175.50000	77.82289	49.2212
2	9.721	MF	0.5453	2244.34399	68.59515	50.7788
Totals :				4419.84399	146.41805	

Instrument 1 12/4/2010 8:57:50 PM THL

Data File D:\LC\LQH\DATE\LQH-3-45-46\LQH-3-45-46 2010-12-04 20-28-43\082-0201.D
Sample Name: lqh-3-46

Acq. Operator	: THL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 82
Injection Date	: 12/4/2010 8:41:06 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-45-46\LQH-3-45-46 2010-12-04 20-28-43\ASH-2-98-10-220NM-20MIN.M
Last changed : 12/4/2010 8:55:36 PM by THL
(modified after loading)
Analysis Method : D:\LC\LQH\DATE\LQH-3-45-46\LQH-3-45-46 2010-12-04 20-28-43\082-0201.D\DA.M
(ASH-2-98-10-220NM-20MIN.M)
Last changed : 12/4/2010 9:00:09 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=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.675	MM	0.4328	1465.73108	56.44477	5.5746
2	9.193	MF	0.5679	2.48272e4	728.68579	94.4254
Totals :				2.62929e4	785.13057	

*** End of Report ***

Instrument 1 12/4/2010 9:00:14 PM THL Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-4-41\LQH-4-41C2-AD 2011-05-06 19-25-49\012-0101.D
Sample Name: LQH-4-41C2-AD

```
==================================================================
Acq. Operator   : TMC                          Seq. Line :   1
Acq. Instrument : Instrument 1                  Location : Vial 12
Injection Date  : 5/6/2011 7:27:36 PM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-4-41\LQH-4-41C2-AD 2011-05-06 19-25-49\ADH-5-95-220NM-
                  1M.M
Last changed    : 1/18/2011 8:38:44 PM by LYY
Analysis Method : D:\LC\LQH\DATE\LQH-4-41\LQH-4-41C2-AD 2011-05-06 19-25-49\012-0101.D\DA.M
                  (ADH-5-95-220NM-1M.M)
Last changed    : 5/25/2011 6:11:28 PM by FX
                  (modified after loading)
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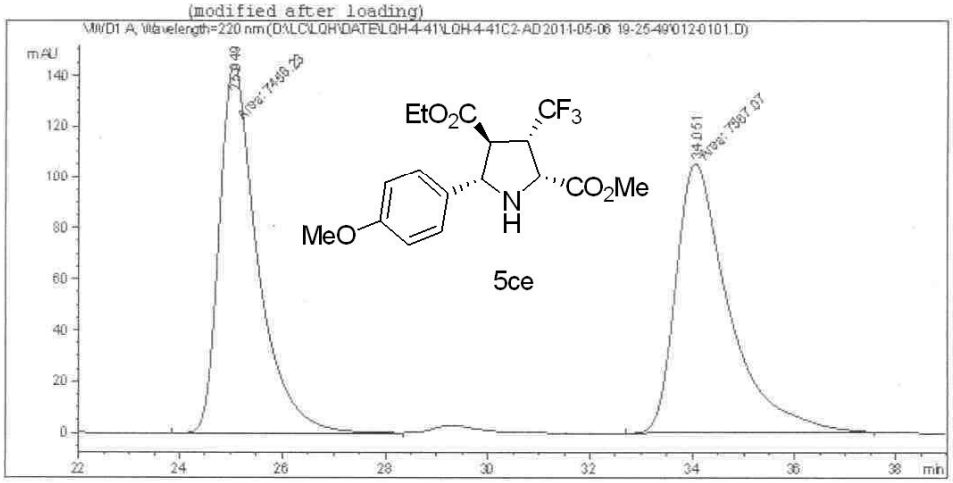


```
==================================================================
                        Area Percent Report
==================================================================

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  25.049 MM    0.8601 7458.23291  144.52231  49.6378
   2  34.051 MM    1.1973 7567.07422  105.33887  50.3622

Totals :                  1.50253e4   249.86118

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

ta File D:\LC\THL\DATE\YDC-1-13\YDC-1-13 2011-05-06 21-46-15\005-0701.D
mple Name: LQH-4-45-1-AD

```
=====================================================================
Acq. Operator   : THL                          Seq. Line :   7
Acq. Instrument : Instrument 1                  Location : Vial 5
Injection Date  : 5/6/2011 11:47:02 PM             Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\THL\DATE\YDC-1-13\YDC-1-13 2011-05-06 21-46-15\ADH-5-95-1ML-220NM-
                  40MIN.M
Last changed    : 3/29/2011 12:09:30 PM by HZL
Analysis Method : D:\LC\THL\DATE\YDC-1-13\YDC-1-13 2011-05-06 21-46-15\005-0701.D\DA.M (ADH-
                  5-95-1ML-220NM-40MIN.M)
Last changed    : 5/25/2011 6:20:48 PM by FX
                  (modified after loading)
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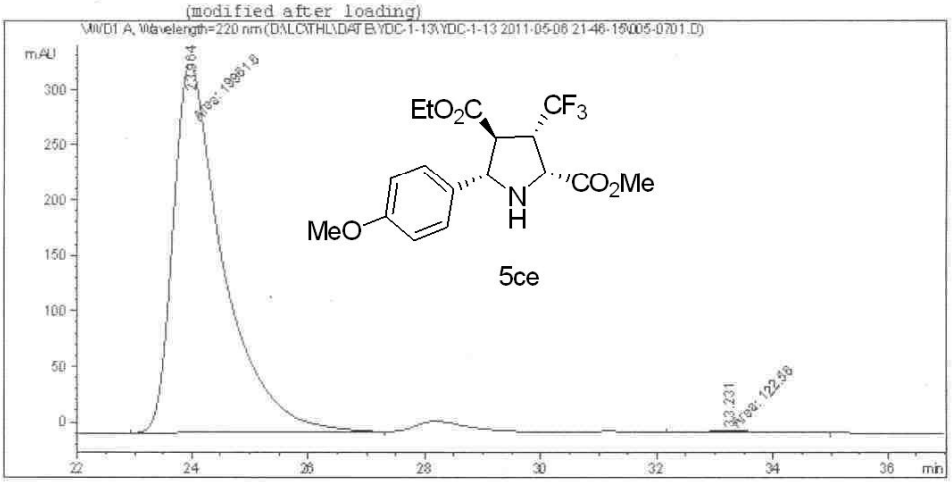
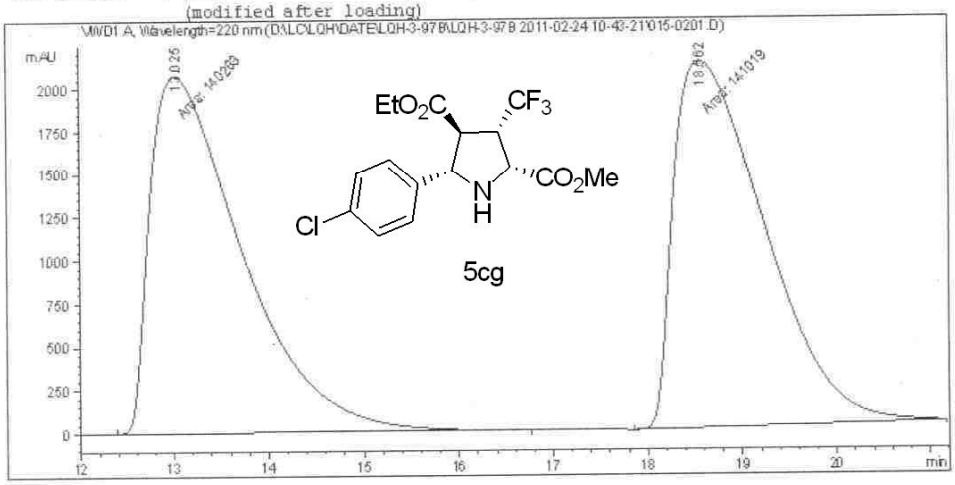



```
=====================================================================
                        Area Percent Report
=====================================================================

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  23.964 MM    1.0000 1.99616e4   332.67972  99.3898
   2  33.231 MM    1.1930  122.56023    1.71216   0.6102

Totals :                  2.00842e4   334.39188

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

Data File D:\LC\LQH\DATE\LQH-3-97B\LQH-3-97B 2011-02-24 10-43-21\015-0201.D
Sample Name: LQH-3-97B

Acq. Operator	: LTL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 15
Injection Date	: 2/24/2011 10:55:48 AM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-97B\LQH-3-97B 2011-02-24 10-43-21\ASH-5-95-1ML-220NM.M
Last changed : 8/30/2010 3:52:51 PM by tmc
Analysis Method : D:\LC\LQH\DATE\LQH-3-97B\LQH-3-97B 2011-02-24 10-43-21\015-0201.D\DA.M (ASH-5-95-1ML-220NM.M)
Last changed : 5/25/2011 5:00:10 PM by FX
(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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.025	MM	1.1316	1.40283e5	2066.08105	49.8692
2	18.562	MM	1.1050	1.41019e5	2127.02661	50.1308

Totals : 2.81302e5 4193.10767

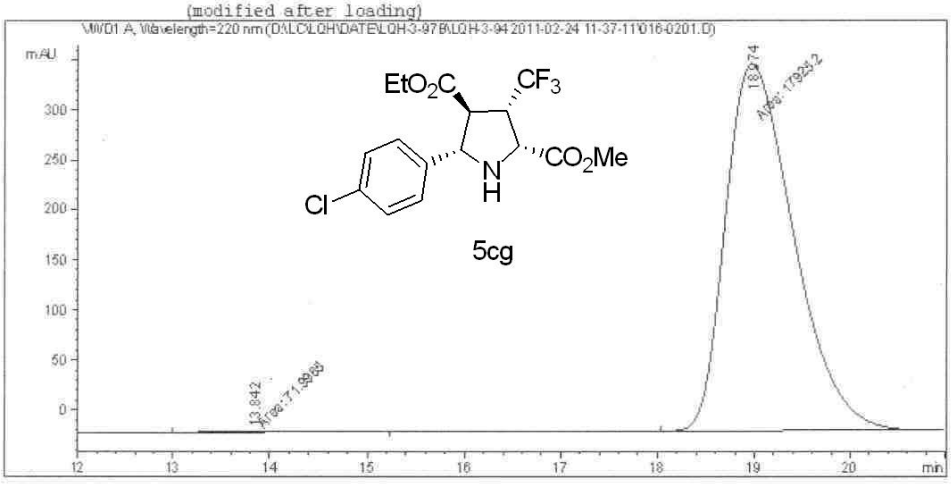
*** End of Report ***

Instrument 1 5/25/2011 5:00:16 PM FX

Page 1 of 1

Data File D:\LC\LQH\DATE\LQH-3-97B\LQH-3-94 2011-02-24 11-37-11\016-0201.D
Sample Name: LQH-3-94

Acq. Operator	: LTL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 16
Injection Date	: 2/24/2011 11:49:34 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\LQH\DATE\LQH-3-97B\LQH-3-94 2011-02-24 11-37-11\ASH-5-95-1ML-220NM-30MIN.M		
Last changed	: 2/24/2011 11:36:08 AM by LTL		
Analysis Method	: D:\LC\LQH\DATE\LQH-3-97B\LQH-3-94 2011-02-24 11-37-11\016-0201.D\DA.M (ASH-5-95-1ML-220NM-30MIN.M)		
Last changed	: 5/25/2011 5:02:33 PM by FX (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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.842	MM	1.0234	71.99645	1.17246	0.4000
2	18.974	MM	0.8132	1.79252e4	367.36295	99.6000
Totals :				1.79972e4	368.53541	

*** End of Report ***

Instrument 1 5/25/2011 5:02:41 PM FX

Page 1 of 1

ata File D:\LC\LQH\DATE\LQH-3-83\LQH-3-83-AD-3 2011-01-21 19-20-52\081-0101.D
ample Name: LQH-3-83-AD

```
==========================================================================
Acq. Operator   : THL-7-95-97                    Seq. Line :   1
Acq. Instrument : Instrument 1                   Location : Vial 81
Injection Date  : 1/21/2011 7:22:06 PM              Inj :   1
                                                 Inj Volume : 5 µl
Acq. Method     : D:\LC\LQH\DATE\LQH-3-83\LQH-3-83-AD-3 2011-01-21 19-20-52\ADH-10-90-220NM-
                  1M.M
Last changed    : 7/28/2010 2:40:06 PM by LTL
Analysis Method : D:\LC\LQH\DATE\LQH-3-83\LQH-3-83-AD-3 2011-01-21 19-20-52\081-0101.D\DA.M
                  (ADH-10-90-220NM-1M.M)
Last changed    : 5/29/2011 3:52:05 PM by LTL
                  (modified after loading)
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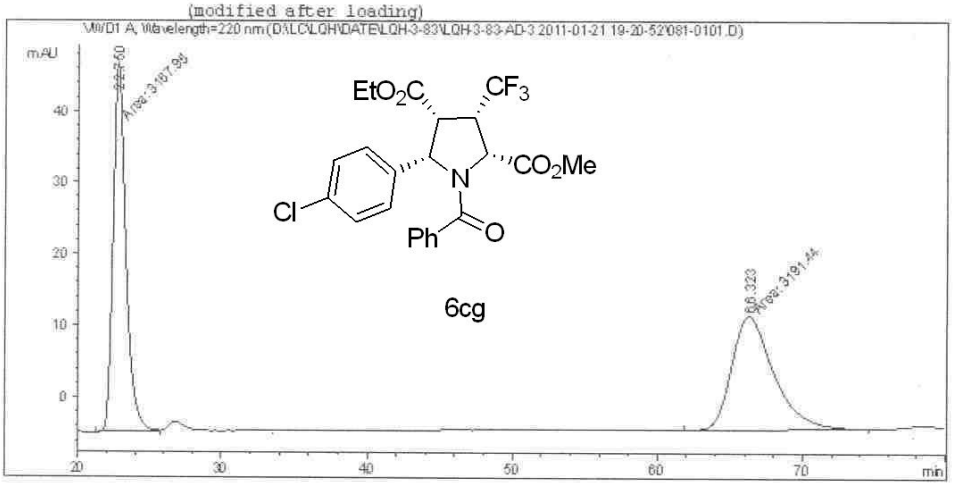


```
==========================================================================
                          Area Percent Report
==========================================================================

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  22.750 MM    1.0256 3167.94897   51.48317  49.8153
   2  66.323 MM    3.3791 3191.44409   15.74130  50.1847

Totals :                 6359.39307   67.22447


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

:a File D:\LC\LQH\DATE\LQH-3-84-2\LQH-3-84-2C 2011-03-01 10-39-18\057-0201.D
mple Name: LQH-3-84-2C

Acq. Operator : THL
Acq. Instrument : Instrument 1
Injection Date : 3/1/2011 10:51:44 AM
Seq. Line : 2
Location : Vial 57
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-84-2\LQH-3-84-2C 2011-03-01 10-39-18\ADH-10-90-220NM-1M-80MIN.M
Last changed : 1/21/2011 9:18:46 PM by THL-7-95-97
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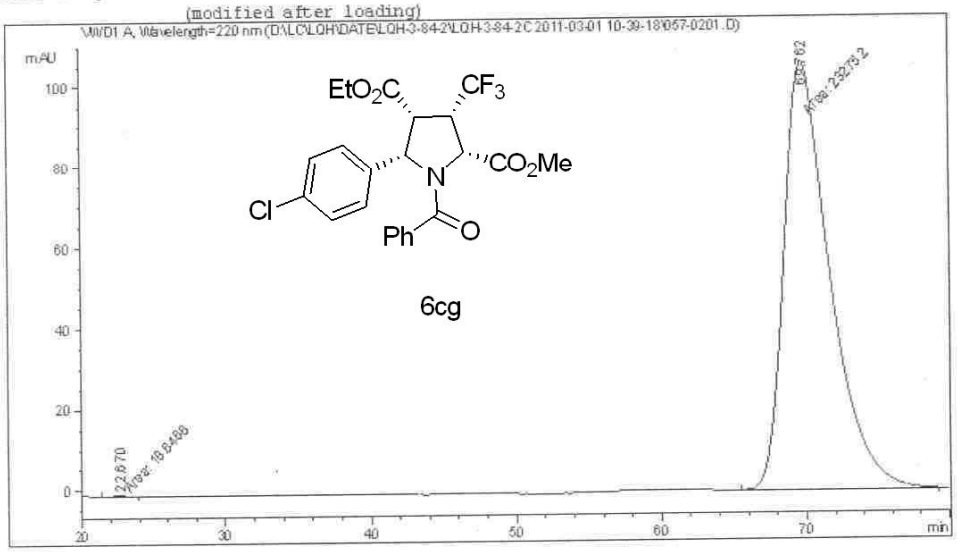
Last changed : 5/29/2011 4:03:57 PM by LTL
(modified after loading)



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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.670	MM	1.1020	18.64658	2.82010e-1	0.0800
2	69.762	MM	3.6592	2.32752e4	106.01153	99.9200
Totals :				2.32939e4	106.29354	

nstrument 1 5/29/2011 4:04:03 PM LTL

ta File D:\LC\LQH\DATE\LQH-3-106\LQH-3-106-RAC-3-20-80 2011-03-17 18-51-35\025-0201.D
mple Name: LQH-3-106-RAC-3

Acq. Operator : THL
Acq. Instrument : Instrument 1
Injection Date : 3/17/2011 7:04:08 PM
Seq. Line : 2
Location : Vial 25
Inj : 1
Inj Volume : 5 µl

Acq. Method : D:\LC\LQH\DATE\LQH-3-106\LQH-3-106-RAC-3-20-80 2011-03-17 18-51-35\ASH-80-20-1ML-220NM.M
Last changed : 3/17/2011 7:42:06 PM by THL
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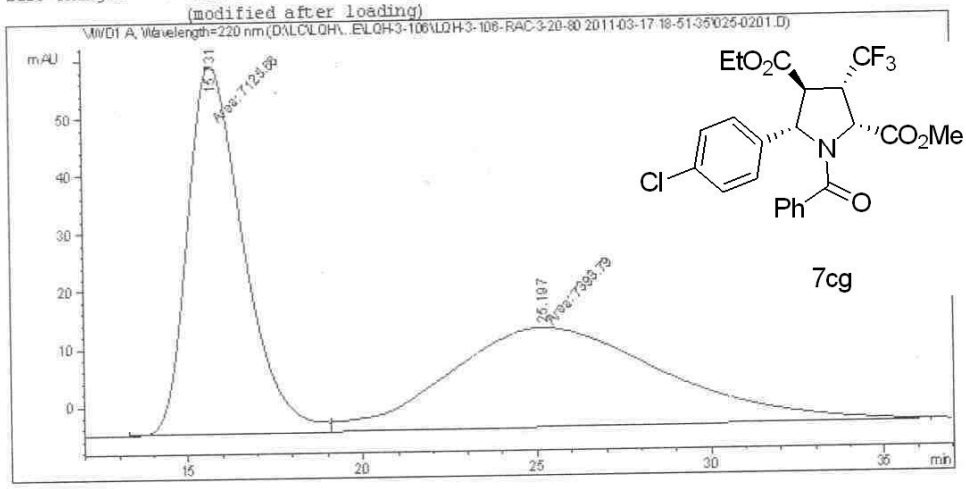
Analysis Method : D:\LC\LQH\DATE\LQH-3-106\LQH-3-106-RAC-3-20-80 2011-03-17 18-51-35\025-0201.D\DA.M (ASH-80-20-1ML-220NM.M)
Last changed : 5/29/2011 4:18:06 PM by LTL
(modified after loading)



7cg

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 [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.731	MF	1.8589	7125.66357	63.68663	49.0767
2	25.197	FM	7.1756	7393.79297	17.17337	50.9233
Totals :				1.45195e4	81.06001	

*** End of Report ***

Instrument 1 5/29/2011 4:18:12 PM LTL

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Data File D:\LC\DXQ\DATE\FX-2-7675-LQH\FX-2-7675 2011-03-17 22-09-18\008-0901.D
Sample Name: LQH-3-106-3

```
==================================================================
Acq. Operator   : THL                          Seq. Line :   9
Acq. Instrument : Instrument 1                  Location : Vial 8
Injection Date  : 3/18/2011 1:30:33 AM             Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\LC\DXQ\DATE\FX-2-7675-LQH\FX-2-7675 2011-03-17 22-09-18\ASH-20-80-1ML-
                  220NM-40MIN.M
Last changed    : 3/17/2011 9:40:45 PM by LYY
Analysis Method : D:\LC\DXQ\DATE\FX-2-7675-LQH\FX-2-7675 2011-03-17 22-09-18\008-0901.D\DA.M
                  (ASH-20-80-1ML-220NM-40MIN.M)
Last changed    : 5/29/2011 4:47:07 PM by LTL
                  (modified after loading)
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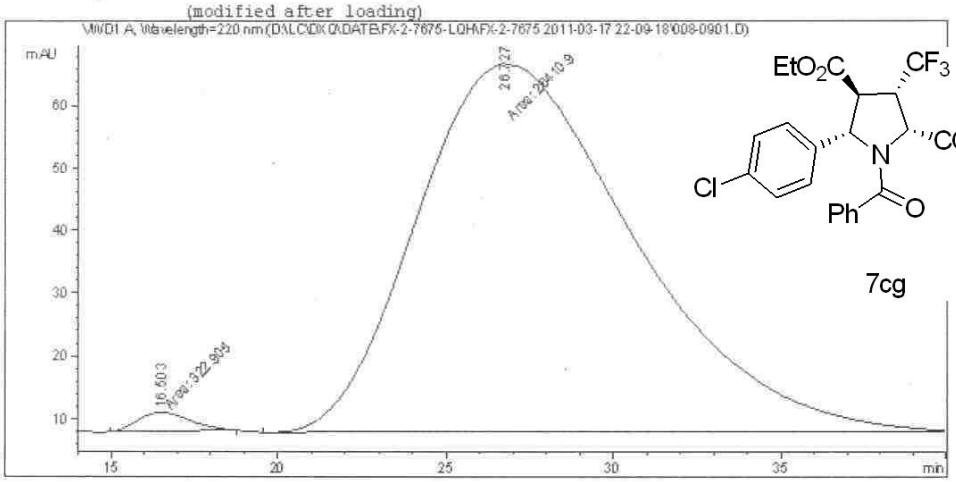


```
==================================================================
                      Area Percent Report
==================================================================

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  16.503 MM    1.8070  322.90475    2.97835   1.2079
   2  26.727 MM    7.4774 2.64109e4    58.86829  98.7921

Totals :                  2.67338e4    61.84664

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

Instrument 1 5/29/2011 4:47:57 PM LTL

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