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Supporting Information for

exo-Selective Construction of Spiro-[butyrolactone-pyrrolidine] *via* 1,3-Dipolar Cycloaddition of Azomethine Ylides with α-Methylene-γ Butyrolactone Catalyzed by Cu(I)/DTBM-BIPHEP

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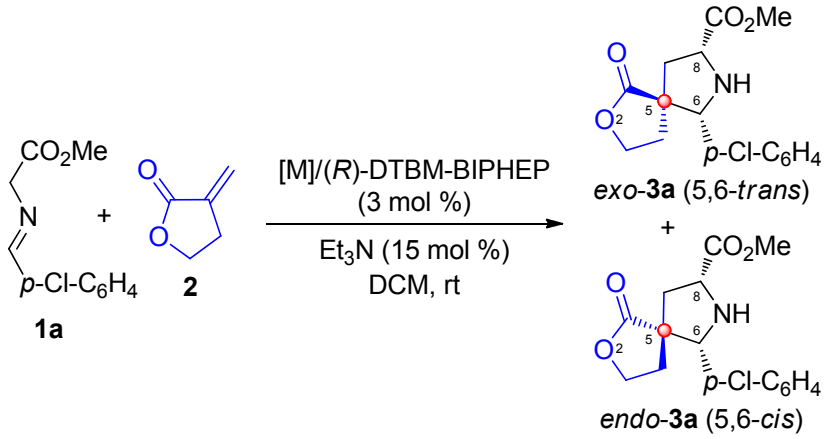
I. General Remarks

^{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, and brs = broad single). ^{13}C NMR spectra were recorded on a Bruker 75 MHz or 100 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially available 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 chiralpak AD-H column, a chiralpak AS-H column or a chiralcel OD-H column with hexane and *i*-PrOH as solvents. α-methylene-γ-butyrolactone was prepared according to the literature procedure.[1] The racemic adducts were obtained by using AgOAc/BINAP as the catalyst. The absolute configuration of **5a** was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

II. Metal salt Screening for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with α-methylene-γ-butyrolactone.

Table S1. Screening studies of the catalytic asymmetric 1,3-DC of imino ester **1a** with α-methylene-γ-butyrolactone **2a**[a]



Entry	[M]	exo/*endo*	yield (%)[b]	ee (%)[c]
1	$AgBF_4$	>98:2	80	98
2	$CuOTf{\cdot}1/2C_6H_6$	>98:2	70	98
3	CuOAc	>98:2	83	97
4	$Cu(OTf)_2$	92:8	68	97

[a] All reactions were carried out with 0.35 mmol of **1a** and 0.23 mmol of **2** in 2 mL solvent for 1-2 h. [b] Isolated yields of *exo*-**3a** and *endo*-**3a**. [c] Ee of *exo*-**3a** was determined by HPLC analysis.

III. General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Imino Esters with α-Methylene-γ-butyrolatone

Under argon atmosphere, *rac*-BINAP (4.7 mg, 0.0076 mmol) and AgOAc (1.1 mg, 0.0069 mmol) were dissolved in 2 mL of DCM, and stirred at room temperature for about 0.5 h. Then, imine substrate (0.35 mmol), Et_3N (0.03 mmol) and α-Methylenebutyrolatone (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.

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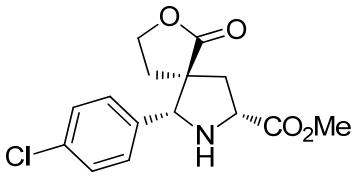
IV. General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with α-Methylenebutyrolatone.

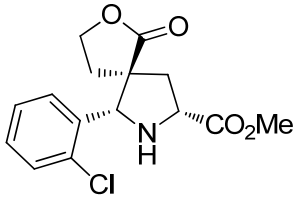
Under argon atmosphere, (*R*)-DTBM-Biphep (**L5**) (8.7 mg, 0.0076 mmol) and $Cu(CH_3CN)_4BF_4$ (2.2 mg, 0.0069 mmol) were dissolved in 2 mL of DCM, and stirred at room temperature for about 0.5 h. After imine substrate (0.35 mmol) was added. Then, α-Methylenebutyrolatone (0.23 mmol) and Et_3N (0.03 mmol) was added sequentially. Once starting material was consumed (monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The residue was purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by HPLC analysis to determine the enantiomeric excess.



(3a)

(5*R*,6*R*,8*R*)-methyl 6-(4-chlorophenyl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

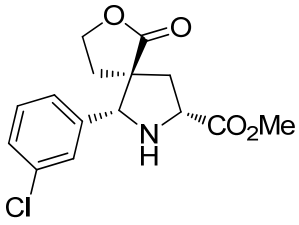
The title compound was prepared according to the general procedure as described above in 87% yield; $[\alpha]^{25}_D$ = +24.0 (*c* 0.75, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.40-7.26 (m, 4H), 4.60 (s, 1H), 4.15-4.09 (m, 1H), 4.06-3.98 (m, 1H), 3.79 (s, 3H), 3.36 (dd, J_1 = 8.1 Hz, J_2 = 15.9 Hz, 1H), 2.80-2.72 (m, 1H), 2.27-2.23 (m, 1H), 2.11-2.02 (m, 1H), 1.95-1.86 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.7, 174.0, 136.2, 134.0, 128.7, 128.2, 67.7, 65.8, 56.9, 52.3, 40.4, 30.7; IR (KBr) ν 3360, 2952, 2820, 2341, 1739, 1513, 1440, 1248, 1216, 1192, 1030, 758 cm^{-1}. HRMS: calcd. for $C_{15}H_{16}ClNO_4 + H^+$: 310.0839, found: 310.0841. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 16.76 and 22.26 min.



(3b)

(5*R*,6*S*,8*R*)-methyl 6-(2-chlorophenyl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]^{25}_{D}$ = -13.1 (*c* 0.76, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.91 (d, *J* = 7.2 Hz, 1H), 7.36-7.21 (m, 3H), 5.12 (s, 1H), 4.22 (t, *J* = 8.1 Hz, 1H), 4.07 (dd, J_1 = 7.8 Hz, J_2 = 15.6 Hz, 1H), 3.79-3.70 (m, 4H), 2.74-2.67 (m, 1H), 2.18-2.04 (m, 2H), 1.82-1.73 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 180.2, 173.5, 137.5, 133.3, 129.4, 129.3, 129.0, 127.0, 66.0, 62.7, 57.5, 52.1, 51.1, 40.4, 32.4; IR (KBr) ν 3350, 2912, 2355, 1730, 1513, 1433, 1240, 1218, 1188, 1028, 759 cm^{-1}. HRMS: calcd. for $C_{15}H_{16}ClNO_4$ + H^+: 310.0841, found: 310.0841. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.78 and 9.78 min.

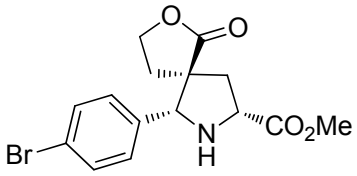


(3c)

(5*R*,6*R*,8*R*)-methyl 6-(3-chlorophenyl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 72% yield. $[\alpha]^{25}_{D}$ = +30.6 (*c* 0.65, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.50 (s, 1H), 7.28 (m, 3H), 4.60 (s, 1H), 4.17-4.13 (m, 1H), 4.05-4.02 (m, 1H), 3.80 (s, 3H), 3.46-3.38 (m, 1H), 2.78-2.71 (m, 1H), 2.27 (dd, J_1 = 4.2 Hz, J_2 = 13.2 Hz, 1H), 2.12-1.97 (m, 1H), 1.92-1.88 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.7, 173.8, 140.0, 134.6, 129.8, 128.4, 126.9, 125.2, 67.7, 65.8, 56.9, 52.4, 52.3, 40.2, 30.8;

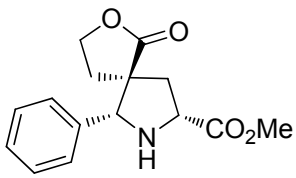
IR (KBr) ν 2945, 2818, 2340, 1720, 1512, 1453, 1240, 1215, 1185, 1023, 757 cm^{-1}. HRMS: calcd. for $C_{15}H_{16}ClNO_4 + H^+$: 310.0828, found: 310.0841. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 17.34 and 21.35 min.



(3d)

(5*R*,6*R*,8*R*)-methyl 6-(4-bromophenyl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_D$ = +27.7 (*c* 0.95, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 4.57 (s, 1H), 4.15-4.12 (m, 1H), 4.02 (dd, J_1 = 8.4 Hz, J_2 = 14.7 Hz, 1H), 3.79 (s, 3H), 3.37 (dd, J_1 = 7.8 Hz, J_2 = 15.0 Hz, 1H), 2.75 (dd, J_1 = 10.2 Hz, J_2 = 13.2 Hz, 1H), 2.25 (dd, J_1 = 4.5 Hz, J_2 = 13.2 Hz, 1H), 2.10-2.01 (m, 1H), 1.95-1.86 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.2, 173.5, 136.3, 131.1, 128.1, 121.6, 67.3, 65.3, 56.4, 51.8, 39.8, 30.2; IR (KBr) ν 3330, 2950, 2810, 2338, 1730, 1508, 1436, 1245, 1208, 1183, 1021, 759 cm^{-1}. HRMS: calcd. for $C_{15}H_{16}BrNO_4 + H^+$: 354.0347, found: 354.0335. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 17.82 and 22.15 min.

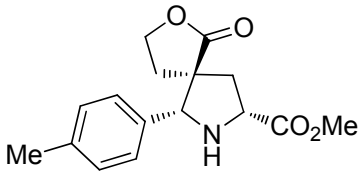


(3e)

(5*R*,6*R*,8*R*)-methyl 1-oxo-6-phenyl-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

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

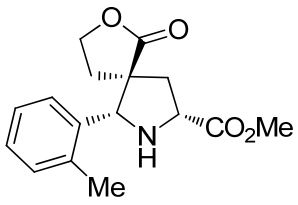
above in 83% yield. $[\alpha]^{25}_{D}$ = +14.3 (*c* 0.40, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.44-7.33 (m, 4H), 4.60 (s, 1H), 4.14 (dd, J_1 = 4.5 Hz, J_2 = 9.9 Hz, 1H), 3.97 (dd, J_1 = 3.3 Hz, J_2 = 8.4 Hz, 1H), 3.80 (s, 3H), 3.19 (dd, J_1 = 7.8 Hz, J_2 = 15.9 Hz, 1H), 2.80 (dd, J_1 = 9.9 Hz, J_2 = 16.2 Hz, 1H), 2.23 (dd, J_1 = 4.5 Hz, J_2 = 10.2 Hz, 1H), 2.16-2.09 (m, 1H), 1.93-1.83 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.0, 174.2, 137.4, 128.5, 128.2, 126.8, 68.6, 65.8, 57.1, 52.6, 52.3, 40.8, 31.0; IR (KBr) ν 3362, 2948, 2807, 2335, 1739, 1513, 1440, 1215, 1180, 1019, 757 cm^{-1}. HRMS: calcd. for $C_{15}H_{17}NO_4 + H^+$: 276.1229, found: 276.1230. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.30 and 21.29 min.



(3f)

(5*R*,6*R*,8*R*)-methyl 1-oxo-6-(p-tolyl)-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

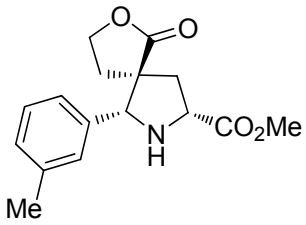
The title compound was prepared according to the general procedure as described above in 78% yield. $[\alpha]^{25}_{D}$ = +9.1 (*c* 1.25, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.30 (d, *J* =7.8 Hz, 2H), 7.14 (d, *J* =7.8 Hz, 2H), 4.55 (s, 1H), 4.12 (m, 1H), 3.96 (dd, J_1 = 8.1 Hz, J_2 = 13.2 Hz, 1H), 3.79 (s, 3H), 3.19 (dd, J_1 = 7.8 Hz, J_2 = 16.8 Hz, 1H), 2.83-2.76 (m, 1H), 2.34 (s, 3H), 2.24-2.10 (m, 2H), 1.91-1.82 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.1, 174.3, 138.0, 134.1, 129.1, 126.6, 68.5, 65.8, 57.0, 52.3, 40.9, 30.9, 21.0; IR (KBr) ν 3350, 2928, 2841, 2341, 1752, 1516, 1440, 1250, 1218, 1175, 1031, 759 cm^{-1}. HRMS: calcd. for $C_{16}H_{19}NO_4 + H^+$: 290.1388, found: 290.1387. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.74 and 21.00 min.



(3g)

(5*R*,6*R*,8*R*)-methyl 1-oxo-6-(o-tolyl)-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 70% yield. $[\alpha]^{25}_{D}$ = +11.5 (*c* 0.78, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.78 (d, J = 6.6 Hz, 1H), 7.27-7.14 (m, 3H), 4.90 (s, 1H), 4.13 (m, 1H), 4.01-3.94 (m, 1H), 3.80 (s, 3H), 3.26 (dd, J_1 = 7.5 Hz, J_2 = 16.2 Hz, 1H), 2.86-2.79 (m, 1H), 2.29 (s, 3H), 2.25-2.14 (m, 2H), 1.92-1.82 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.6, 173.9, 136.3, 136.1, 130.7, 127.8, 127.3, 125.9, 66.1, 63.6, 57.3, 52.2, 41.3, 32.4, 19.4; IR (KBr) ν 3340, 2950, 2825, 2341, 1737, 1512, 1444, 1245, 1210, 1189, 1031, 758 cm^{-1}. HRMS: calcd. for $C_{16}H_{19}NO_4$ + H^+: 290.1389, found: 290.1387. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak OD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.99 and 25.31 min.

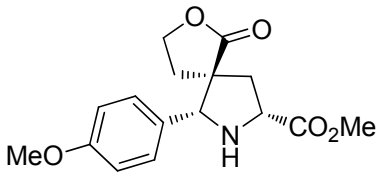


(3h)

(5*R*,6*R*,8*R*)-methyl 1-oxo-6-(m-tolyl)-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 75% yield. $[\alpha]^{25}_{D}$ = +14.9 (*c* 0.62, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.29-7.13 (m, 4H), 4.54 (s, 1H), 4.15-4.12 (m, 1H), 3.97 (dd, J_1 = 8.7 Hz, J_2 = 13.8 Hz, 1H), 3.80(s, 3H), 3.22 (dd, J_1 = 8.1 Hz, J_2 = 16.2 Hz, 1H), 2.83-2.75 (m, 1H), 2.55 (brs, 1H), 2.34 (s, 3H), 2.24-2.10 (m, 2H), 1.92-1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.0, 174.2, 138.2, 137.2, 128.9, 128.3, 127.3, 123.8, 68.6, 65.8, 57.0, 52.5, 52.2, 40.9, 31.0, 21.3; IR (KBr) ν 3351, 2970, 2817, 2340, 1735, 1516, 1236, 1211, 1185, 1025, 759 cm^{-1}. HRMS: calcd. for $C_{16}H_{19}NO_4$ + H^+: 290.1386,

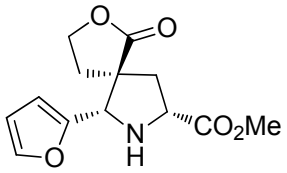
found: 290.1387. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.43 and 16.80 min.



(3i)

(5*R*,6*R*,8*R*)-methyl 6-(4-methoxyphenyl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]^{25}_D$ = -19.0 (*c* 1.09, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.34 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 4.53 (s, 1H), 4.12 (dd, J_1 = 4.5 Hz, J_2 = 10.2 Hz, 1H), 4.01-3.94 (m, 1H), 3.80 (s, 3H), 3.79 (s, 3H), 3.21 (dd, J_1 = 8.1 Hz, J_2 = 16.5 Hz, 1H), 2.78 (dd, J_1 = 10.2 Hz, J_2 = 13.2 Hz, 1H), 2.24-2.11 (m, 2H), 1.92-1.83 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.0, 174.2, 159.4, 129.1, 127.8, 113.8, 68.2, 65.7, 56.9, 55.1, 52.5, 52.2, 40.7, 30.9; IR (KBr) ν 3330, 2935, 2818, 2348, 1725, 1518, 1447, 1236, 1214, 1183, 1017, 759 cm^{-1}. HRMS: calcd. for $C_{16}H_{19}NO_5 + H^+$: 306.1339, found: 306.1336. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 23.50 and 28.21 min.

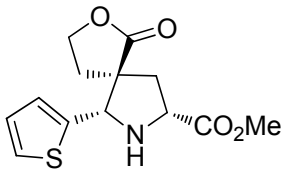


(3j)

(5*R*,6*S*,8*R*)-methyl 6-(furan-2-yl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 74% yield. $[\alpha]^{25}_D$ = +67.3 (*c* 0.42, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.40 (s, 1H), 6.37 (s, 2H), 4.50 (s, 1H), 4.14-4.10 (m, 2H), 3.79 (s, 3H), 3.54

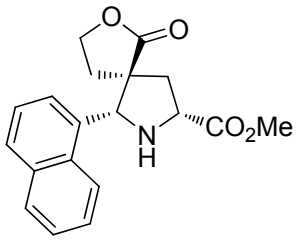
(dd, J_1 = 7.8 Hz, J_2 = 15.9 Hz, 1H), 2.77 (dd, J_1 = 9.6 Hz, J_2 = 13.5 Hz, 1H), 2.29-2.16 (m, 2H), 1.95-1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.2, 174.1, 151.0, 142.4, 110.5, 108.1, 65.6, 63.3, 57.7, 52.7, 52.4, 41.0, 31.2; IR (KBr) ν 3352, 2950, 2816, 2335, 1730, 1514, 1445, 1236, 1215, 1175, 1050, 769 cm^{-1}. HRMS: calcd. for $C_{13}H_{15}NO_5$ + H^+: 266.1023, found: 266.1023. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.39 and 25.99 min.



(3k)

(5*R*,6*S*,8*R*)-methyl 1-oxo-6-(thiophen-2-yl)-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 84% yield. $[\alpha]^{25}_D$ = +43.3 (*c* 0.88, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.29-7.25 (m, 1H), 7.02-6.98 (m, 2H), 4.84 (s, 1H), 4.12-4.06 (m, 2H), 3.79 (s, 3H), 3.53 (dd, J_1 = 7.8 Hz, J_2 = 16.6 Hz, 1H), 2.76-2.69 (m, 2H), 2.30-2.25 (m, 2H), 2.00-1.90 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.4, 173.6, 141.4, 127.0, 124.9, 124.7, 65.9, 64.7, 56.9, 52.8, 52.3, 39.9, 31.0; IR (KBr) ν 2952, 2819, 2345, 1735, 1517, 1449, 1240, 1213, 1185, 1016, 759 cm^{-1}. HRMS: calcd. for $C_{13}H_{15}NO_4S$ + H^+: 282.0792, found: 282.0795. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.43 and 26.17 min.

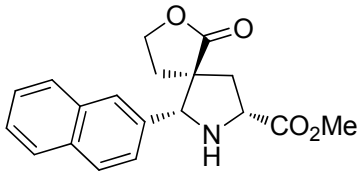


(3l)

(5*R*,6*R*,8*R*)-methyl 6-(naphthalen-1-yl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-car-

boxylate

The title compound was prepared according to the general procedure as described above in 83% yield. $[\alpha]^{25}_{D}$ = -54.3 (*c* 1.04, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.07-8.03 (m, 2H), 7.87-7.80 (m, 2H), 7.52-7.48 (m, 3H), 5.49 (s, 1H), 4.26-4.21 (m, 1H), 3.89-3.82 (m, 4H), 3.03 (dd, J_1 = 7.5 Hz, J_2 = 15.9 Hz, 1H), 2.89 (dd, J_1 = 9.0 Hz, J_2 = 13.2 Hz, 1H), 2.25 (dd, J_1 = 6.6 Hz, J_2 = 12.9 Hz, 1H), 2.16-2.07 (m, 1H), 1.87-1.80 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.8, 173.7, 134.2, 133.6, 131.3, 128.8, 128.6, 126.7, 125.9, 125.3, 125.1, 122.8, 66.0, 62.8, 57.3, 52.3, 51.5, 41.4, 32.6 IR (KBr) ν 3336, 2951, 2815, 2348, 1733, 1526, 1440, 1235, 1215, 1180, 1045, 759 cm^{-1}. HRMS: calcd. for $C_{19}H_{19}NO_4$ + H^+: 326.1387, found: 326.1387. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.41 and 24.37 min.

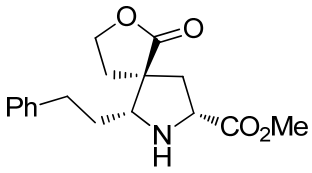


(3m)

(5*R*,6*R*,8*R*)-methyl 6-(naphthalen-2-yl)-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_{D}$ = +44.4 (*c* 0.75, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.93 (s, 1H), 7.82-7.79 (m, 3H), 7.49-7.47 (m, 3H), 4.76 (s, 1H), 4.20-4.16 (m, 1H), 3.93 (dd, J_1 = 8.4 Hz, J_2 = 14.1 Hz, 1H), 3.82 (s, 3H), 3.20 (dd, J_1 = 7.8 Hz, J_2 = 15.6 Hz, 1H), 2.82 (dd, J_1 = 10.8 Hz, J_2 = 12.9 Hz, 1H), 2.27 (dd, J_1 = 4.8 Hz, J_2 = 13.5 Hz, 1H), 2.19-2.10 (m, 1H), 1.94-1.85 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.1, 174.1, 135.0, 133.1, 133.0, 128.2, 128.0, 127.6, 126.3, 126.1, 125.6, 124.8, 68.6, 65.8, 57.1, 52.6, 52.3, 40.8, 31.0; IR (KBr) ν 3356, 2950, 2827, 2365, 1728, 1514, 1245, 1210, 1185, 1031, 756 cm^{-1}. HRMS: calcd. for $C_{19}H_{19}NO_4$ + H^+: 326.1389, found: 326.1387. The product was analyzed by HPLC to determine the

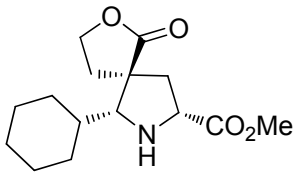
enantiomeric excess: 98% ee (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 21.73 and 31.28 min.



(3n)

(5*R*,6*R*,8*R*)-methyl 1-oxo-6-phenethyl-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

The title compound was prepared according to the general procedure as described above in 62% yield. $[\alpha]^{25}_D$ = +13.5 (*c* 0.24, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.32-7.27 (m, 2H), 7.22-7.18 (m, 3H), 4.25 (t, J = 7.2 Hz, 2H), 4.00 (dd, J_1 = 4.8 Hz, J_2 = 10.2 Hz, 1H), 3.77 (s, 3H), 3.38-3.34 (m, 1H), 2.88-2.81 (m, 1H), 2.69-2.59 (m, 2H), 2.42-2.32 (m, 1H), 2.09 (dd, J_1 = 4.5 Hz, J_2 = 13.5 Hz, 1H), 1.96-1.87 (m, 1H), 1.80-1.73 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.8, 174.4, 141.2, 128.4, 128.1, 126.0, 65.6, 57.8, 52.3, 51.2, 41.6, 33.4, 32.5, 29.7; IR (KBr) ν 3349, 2950, 2817, 2321, 1730, 1517, 1446, 1245, 1210, 1186, 1034, 776 cm^{-1}. HRMS: calcd. for $C_{17}H_{21}NO_4 + H^+$: 304.1541, found: 304.1543. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.68 and 21.92 min.

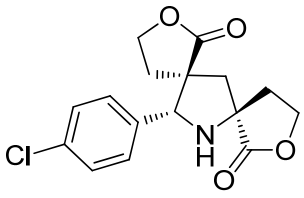


(3o)

(5*R*,6*R*,8*R*)-methyl 6-cyclohexyl-1-oxo-2-oxa-7-azaspiro[4.4]nonane-8-carboxylate

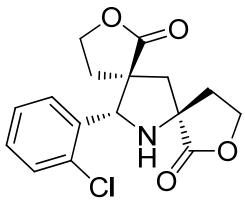
The title compound was prepared according to the general procedure as described above in 65% yield. $[\alpha]^{25}_D$ = +16.1 (*c* 0.74, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 4.45-4.38 (m, 1H), 4.27-4.18 (m, 1H), 3.97-3.95 (m, 1H), 3.76 (s, 3H), 3.14-3.12 (m, 1H), 2.49-2.14 (m, 4H), 1.99-1.87 (m, 2H), 1.77-1.67 (m, 3H), 1.44-1.29 (m, 2H),

1.19-1.00 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.4, 174.4, 70.1, 65.3, 57.2, 52.3, 49.4, 41.9, 39.1, 31.1, 29.6, 28.5, 25.9, 25.6; IR (KBr) ν 2972, 2816, 2337, 1725, 1510, 1436, 1215, 1196, 1035, 759 cm^{-1}. HRMS: calcd. for $C_{15}H_{23}NO_4$ + H^+: 282.1699, found: 282.1700. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); t_r = 15.47 and 17.27 min.



(5a)

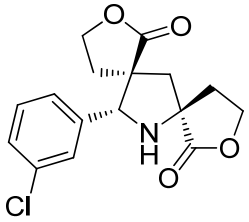
The title compound was prepared according to the general procedure as described above in 82% yield. $[\alpha]^{25}_D$ = +27.7 (*c* 0.48, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.35 (m, 4H), 4.70 (s, 1H), 4.41-4.28 (m, 2H), 4.03 (dd, J_1 = 8.1 Hz, J_2 = 15.0 Hz, 1H), 3.35 (dd, J_1 = 7.8 Hz, J_2 = 16.2 Hz, 1H), 2.60-2.37 (m, 5H), 2.22-2.15 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 179.7, 179.0, 135.3, 134.5, 129.1, 128.3, 68.3, 66.3, 65.5, 63.4, 54.7, 47.2, 38.3, 30.7; IR (KBr) ν 3355, 2951, 2817, 2335, 1737, 1523, 1425, 1240, 1215, 1183, 1021, 776 cm^{-1}. HRMS: calcd. for $C_{16}H_{16}ClNO_4$ + H^+: 322.0834, found: 322.0841. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 20.60 and 24.47 min.



(5b)

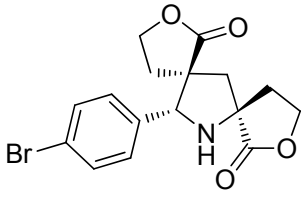
The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]^{25}_D$ = +10.1 (*c* 0.49, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.94 (d, *J* = 7.5 Hz, 1H), 7.39-7.29 (m, 3H), 5.25 (s, 1H), 4.46-4.27 (m, 2H), 4.09-4.02 (m, 1H), 3.53 (dd, J_1 = 7.5 Hz, J_2 = 16.5 Hz, 1H), 2.80-2.64 (m, 2H), 2.48-2.25 (m, 4H); 2.05-1.93 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.1, 178.8,

136.0, 133.4, 129.5, 129.4, 129.3, 127.0, 66.1, 65.2, 63.9, 63.6, 52.2, 46.9, 36.8, 32.9; IR (KBr) ν 3350, 2950, 2815, 2327, 1735, 1510, 1445, 1237, 1210, 1197, 1031, 769 cm^{-1}. HRMS: calcd. for $C_{16}H_{16}ClNO_4 + H^+$: 322.0844, found: 322.0841. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 17.23 and 26.81 min.



(5c)

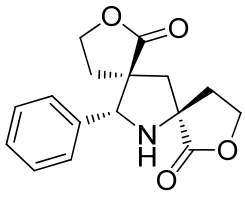
The title compound was prepared according to the general procedure as described above in 81% yield. $[\alpha]^{25}_D$ = +36.4 (*c* 0.57, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.46-7.27 (m, 4H), 4.70 (s, 1H), 4.43-4.30 (m, 2H), 4.05-4.02 (m, 1H), 3.41-3.38 (m, 1H), 2.58-2.37 (m, 5H), 2.22-2.18 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.4, 178.7, 138.9 134.6, 129.9, 128.6, 126.8, 125.1, 67.9, 66.1, 65.3, 63.0, 54.2, 46.7, 37.9, 30.5; IR (KBr) ν 3348, 2951, 2816, 2336, 1730, 1511, 1445, 1242, 1220, 1191, 1033, 769 cm^{-1}. HRMS: calcd. for $C_{16}H_{16}ClNO_4 + H^+$: 322.0825, found: 322.0841. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 17.56 and 24.86 min.



(5d)

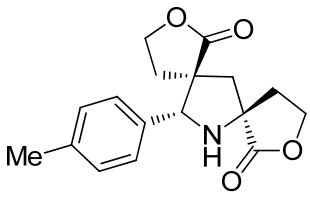
The title compound was prepared according to the general procedure as described above in 84% yield. $[\alpha]^{25}_D$ = +26.0 (*c* 1.16, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.48 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 4.68 (s, 1H), 4.43-4.30 (m, 2H), 4.06-3.98 (m, 1H), 3.35 (dd, J_1 = 7.2 Hz, J_2 = 16.2 Hz, 1H), 2.58-2.36 (m, 5H),

2.20-2.14 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.4, 178.8, 135.7, 131.8, 128.4, 122.3, 68.0, 66.0, 65.3, 63.1, 54.3, 46.9, 38.0, 30.5; IR (KBr) ν 3340, 2951, 2815, 2337, 1730, 1510, 1425, 1240, 1211, 1185, 1017, 758 cm^{-1}. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.63 and 23.89 min.



(5e)

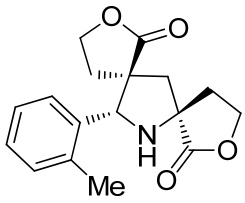
The title compound was prepared according to the general procedure as described above in 83% yield. $[\alpha]^{25}_D$ = +23.1 (*c* 0.86, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.39-7.35 (m, 5H), 4.70 (s, 1H), 4.43-4.31 (m, 2H), 4.05-3.96 (m, 1H), 3.20 (dd, J_1 = 7.8 Hz, J_2 = 16.5 Hz, 1H), 2.65-2.26 (m, 6H), 2.17-2.07 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.7, 179.0, 136.2, 128.5, 128.4, 126.5, 68.6, 65.9, 65.3, 63.1, 54.5, 47.2, 37.9, 30.6; IR (KBr) ν 3365, 2951, 2816, 2340, 1733, 1516, 1425, 1243, 1211, 1176, 1018, 779 cm^{-1}. HRMS: calcd. for $C_{16}H_{17}NO_4$ + H^+: 288.1233, found: 288.1230. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 18.66 and 23.12 min.



(5f)

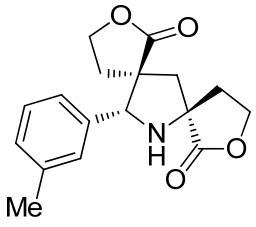
The title compound was prepared according to the general procedure as described above in 75% yield. $[\alpha]^{25}_D$ = +26.0 (*c* 0.9, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.27 (d, *J* = 7.8 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 4.64 (s, 1H), 4.44-4.39 (m, 1H), 4.35-4.27 (m, 1H), 4.01-3.94 (m, 1H), 3.20 (dd, J_1 = 7.8 Hz, J_2 = 16.8 Hz, 1H), 2.62-2.45 (m, 4H), 2.38-2.25 (m, 5H), 2.14-2.04 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75

MHz) 179.7, 179.0, 138.1, 133.0, 129.2, 126.4, 68.6, 66.0, 65.4, 63.1, 54.5, 47.3, 38.0, 30.6, 21.0; IR (KBr) ν 3349, 2943, 2812, 2340, 1726, 1516, 1240, 1211, 1172, 1018, 769 cm^{-1}. HRMS: calcd. for $C_{17}H_{19}NO_4$ + H^+: 302.1392, found: 302.1387. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 16.60 and 24.67 min.



(5g)

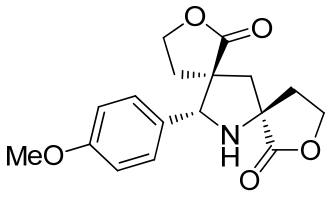
The title compound was prepared according to the general procedure as described above in 68% yield. $[\alpha]^{25}_D$ = +28.8 (*c* 0.87, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.75-7.72 (m, 1H), 7.28-7.17 (m, 3H), 4.99 (s, 1H), 4.42-4.38 (m, 1H), 4.34-4.29 (m, 1H), 4.01-3.94 (m, 1H), 3.24-3.16 (m, 1H), 2.68-2.63 (m, 2H), 2.49-2.30 (m, 7H), 2.10-2.03 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.4, 179.0, 136.3, 134.8, 130.9, 128.0, 126.9, 126.0, 66.3, 65.3, 64.0, 63.3, 52.9, 47.6, 37.4, 32.3, 31.9, 19.4; IR (KBr) ν 3362, 2950, 2825, 2331, 1731, 1517, 1420, 1241, 1210, 1182, 1016, 768 cm^{-1}. HRMS: calcd. for $C_{17}H_{19}NO_4$ + H^+: 302.1382, found: 302.1387. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.73 and 17.72 min.



(5h)

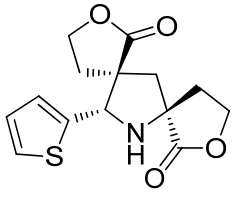
The title compound was prepared according to the general procedure as described above in 75% yield. $[\alpha]^{25}_D$ = +19.0 (*c* 0.81, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.27-7.13 (m, 4H), 4.65 (s, 1H), 4.44-4.30 (m, 2H), 4.01-3.95 (m, 1H), 3.25-3.22 (m, 1H), 2.63-2.45 (m, 4H), 2.39-2.26 (m, 5H), 2.15-2.08 (m, 1H); ^{13}C NMR ($CDCl_3$,

TMS, 75 MHz) 179.8, 179.0, 138.3, 136.0, 129.1, 128.4, 127.1, 123.6, 68.7, 66.0, 65.4, 63.2, 54.5, 47.4, 38.0, 30.7, 21.3; IR (KBr) ν 3355, 2951, 2830, 2335, 1731, 1520, 1438, 1241, 1210, 1196, 1031, 766 cm^{-1}. HRMS: calcd. for $C_{17}H_{19}NO_4 + Na^+$: 324.1203, found: 324.1206. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 13.18 and 18.32 min.



(5i)

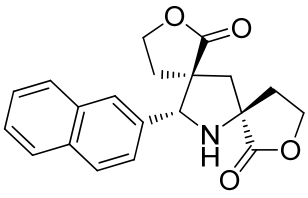
The title compound was prepared according to the general procedure as described above in 80% yield. d $[\alpha]^{25}_D$ = +23.3 (*c* 0.79, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.31 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 7.8 Hz, 2H), 4.63 (s, 1H), 4.44-4.30 (m, 2H), 4.02-3.95 (m, 1H), 3.80 (s, 3H), 3.20 (dd, J_1 = 7.5 Hz, J_2 = 16.5 Hz, 1H), 2.61-2.52 (m, 4H), 2.37-2.26 (m, 2H), 2.15-2.07 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.8, 179.0, 159.5, 128.0, 127.7, 113.9, 68.4, 66.0, 65.4, 63.1, 55.2, 54.5, 47.2, 38.0, 30.6; IR (KBr) ν 2953, 2821, 2351, 1734, 1503, 1429, 1238, 1211, 1190, 1033, 768 cm^{-1}. HRMS: calcd. for $C_{17}H_{19}NO_5 + Na^+$: 340.1163, found: 340.1155. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 34.64 and 61.59 min.



(5j)

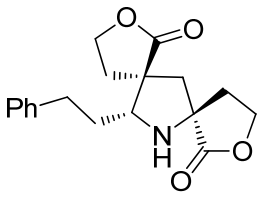
The title compound was prepared according to the general procedure as described above in 84% yield. $[\alpha]^{25}_D$ = +45.5 (*c* 0.80, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.29-7.27 (m, 1H), 7.04-7.02 (m, 2H), 4.93 (s, 1H), 4.43-4.38 (m, 1H), 4.35-4.30 (m, 1H), 4.10-4.05 (m, 1H), 3.52 (dd, J_1 = 7.5 Hz, J_2 = 15.9 Hz, 1H), 2.59-2.39 (m,

6H), 2.25-2.15 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.2, 178.5, 139.5, 127.2, 125.2, 125.1, 66.2, 65.4, 63.3, 55.0, 46.9, 38.1, 30.7; IR (KBr) ν 3340, 2951, 2810, 2340, 1730, 1503, 1426, 1244, 1212, 1186, 1016, 778 cm^{-1}. HRMS: calcd. for $C_{14}H_{15}NO_4S$ + H^+: 294.0803, found: 294.0795. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak OD-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.45 and 17.52 min.



(5k)

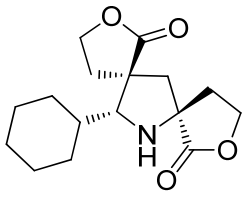
The title compound was prepared according to the general procedure as described above in 82% yield. $[\alpha]^{25}_D$ = +50.4 (*c* 0.36, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.90-7.80 (m, 4H), 7.51-7.43 (m, 3H), 4.87 (s, 1H), 4.46-4.40 (m, 1H), 4.36-4.30 (m, 1H), 3.97-3.90 (m, 1H), 3.19 (dd, J_1 = 8.1 Hz, J_2 = 16.8 Hz, 1H), 2.65-2.44 (m, 4H), 2.43-2.38 (m, 1H), 2.32-2.25 (m, 1H), 2.17-2.10 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.9, 179.0, 138.3, 133.8, 133.0, 128.5, 128.0, 127.6, 126.5, 126.4, 125.5, 124.5, 68.9, 66.1, 65.5, 63.3, 54.6, 47.4, 38.1, 30.7; IR (KBr) ν 3350, 2950, 2819, 2340, 1735, 1506, 1428, 1238, 1205, 1179, 1026, 768 cm^{-1}. HRMS: calcd. for $C_{20}H_{19}NO_4$ + H^+: 338.1393, found: 338.1387. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.56 and 39.97 min.



(5l)

The title compound was prepared according to the general procedure as described above in 60% yield. $[\alpha]^{25}_D$ = +53.0 (*c* 1.15, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.31-7.17 (m, 5H), 4.44-4.37 (m, 1H), 4.29-4.24 (m, 3H), 3.39 (dd, J_1 = 4.5 Hz, J_2 = 9.3 Hz, 1H), 2.85-2.80 (m, 1H), 2.62-2.50 (m, 1H), 2.45-2.34 (m, 5H), 2.20-2.11 (m,

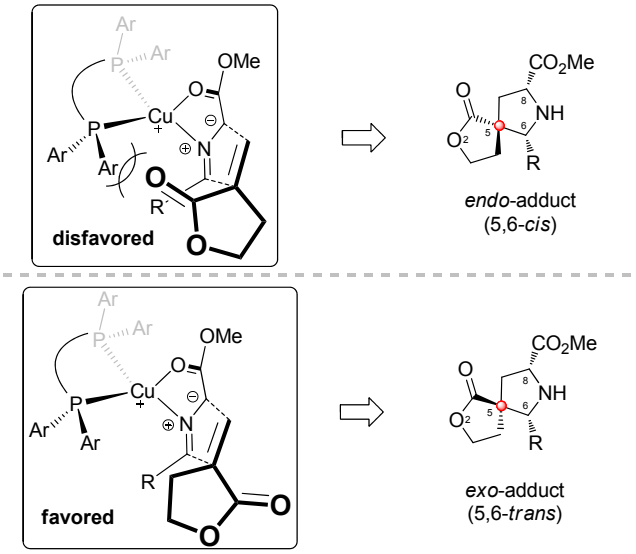
2H), 1.85-1.77 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 179.9, 179.4, 141.2, 128.4, 128.3, 126.1, 74.0, 65.9, 64.5, 53.5, 47.5, 38.5, 33.4, 32.2, 29.6; IR (KBr) ν 3350, 2946, 2826, 2336, 1735, 1510, 1446, 1235, 1215, 1036, 769 cm^{-1}. HRMS: calcd. for $C_{18}H_{21}NO_4$ + H^+: 316.1544, found: 316.1543. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 21.36 and 25.61 min.



(5m)

The title compound was prepared according to the general procedure as described above in 67% yield. $[\alpha]^{25}_D$ = +15.3 (*c* 0.26, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 4.49-4.37 (m, 2H), 4.28-4.18 (m, 2H), 3.21-3.18 (m, 1H), 2.46-2.33 (m, 4H), 2.27-2.16 (m, 2H), 2.00-1.95 (m, 2H), 1.73-1.68 (m, 3H), 1.43-1.40 (m, 2H), 1.26-1.00 (m, 5H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) 180.3, 179.6, 70.6, 65.8, 65.7, 64.0, 52.2, 48.5, 39.0, 35.6, 31.5, 29.8, 28.1, 26.0, 25.7; IR (KBr) ν 2951, 2823, 2335, 1737, 1445, 1228, 1219, 1190, 1035, 759 cm^{-1}. HRMS: calcd. for $C_{16}H_{23}NO_4$ + H^+: 294.1706, found: 294.1700. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 50/50, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.68 and 19.60 min.

V. Proposed Transition States of the *exo*-Selectivity for Asymmetric 1,3-Dipolar Cycloaddition of Imino Esters with α-Methylene-γ-butyrolactone.



Scheme S1. Proposed transition states.

Based on the relative and absolute configuration of cycloadduct (7*R*,9*R*,13*S*)-**5a**, the high *exo*-selectivity observed in the Cu(I)/(*R*)-DTBM-BIPHEP (**L5**) catalyzed asymmetric 1,3-DC reaction of imino ester with α-methylene-γ-butyrolactone can be rationalized by the proposed transition states in Scheme S1. The active species is a copper(I) complex having bulky and electron-donating bisphosphine ligand and an *in situ*-formed azomethine ylide in tetrahedral configuration. [2] An *exo* approach of α-methylene-γ-butyrolactone to the copper(I) complex occurred predominantly because of the disfavored steric repulsion generated in the corresponding *endo* approach between the substituents of α-methylene-γ-butyrolactone and the large bulky aryl group on the phosphorus atom of the chiral ligand.

VI. ^{31}P NMR Spectra of (*R*)-DTBM-BIPHEP Cu(MeCN)$_4$BF$_4$ complex

a) Free ligand (*R*)-DTBM-BIPHEP

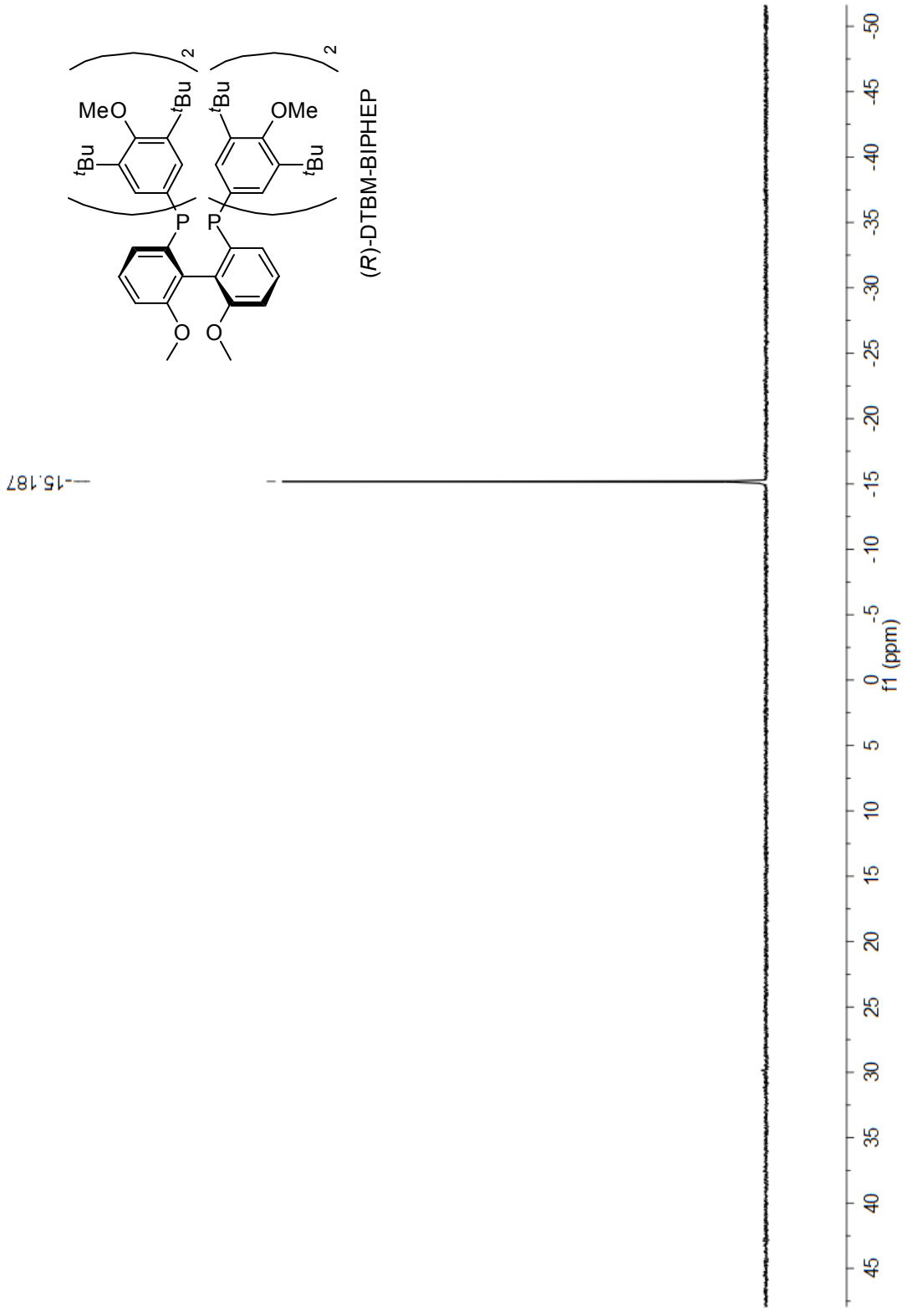


Figure S1. ^{31}P NMR spectrum of free ligand (*R*)-DTBM-BIPHEP in $CDCl_3$.

b) $Cu(MeCN)_4BF_4$:(*R*)-DTBM-BIPHEP = 1:1

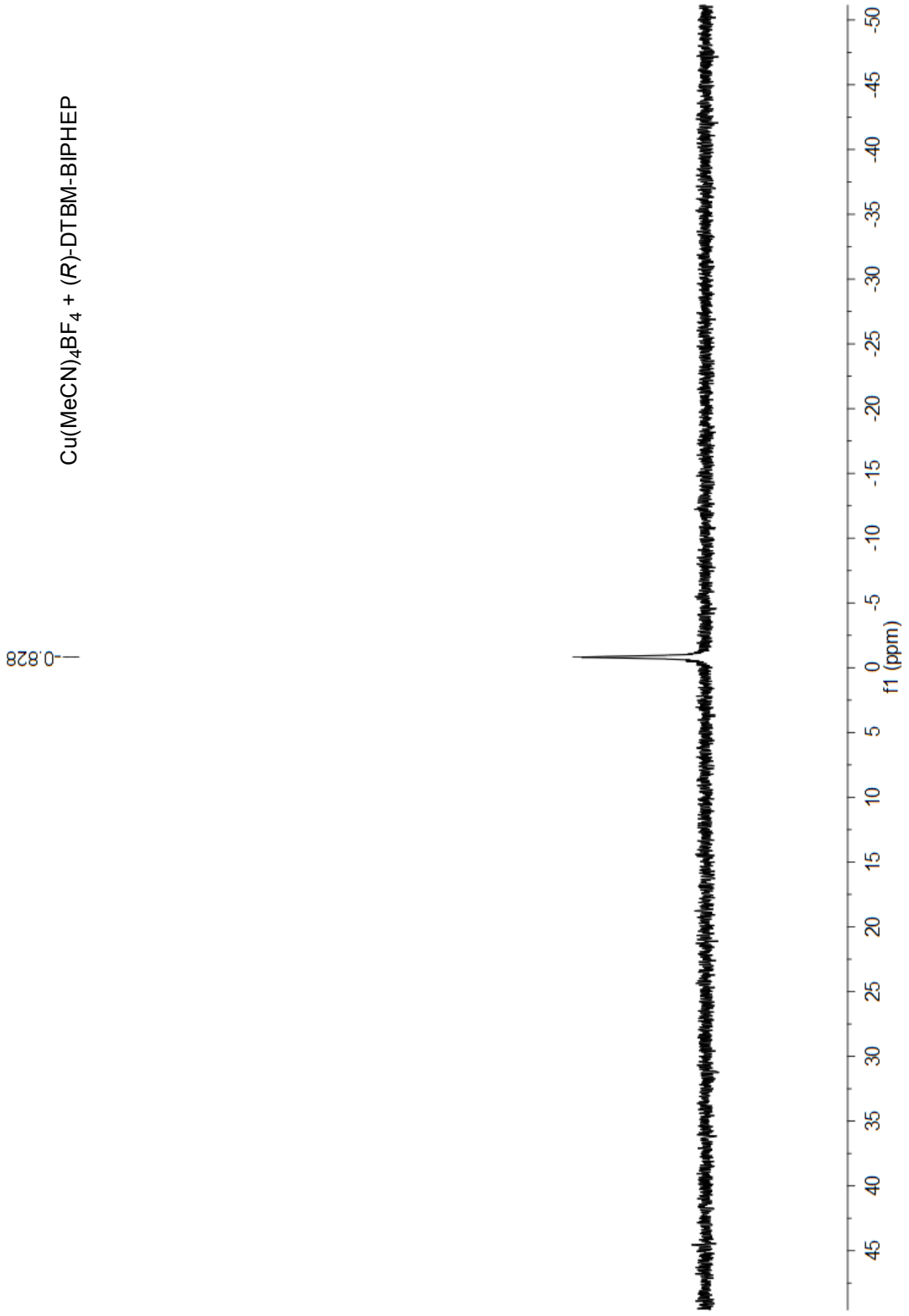


Figure S2. ^{31}P NMR spectrum of (*R*)-DTBM-BIPHEP/$Cu(MeCN)_4BF_4$ (1:1) complex in $CDCl_3$

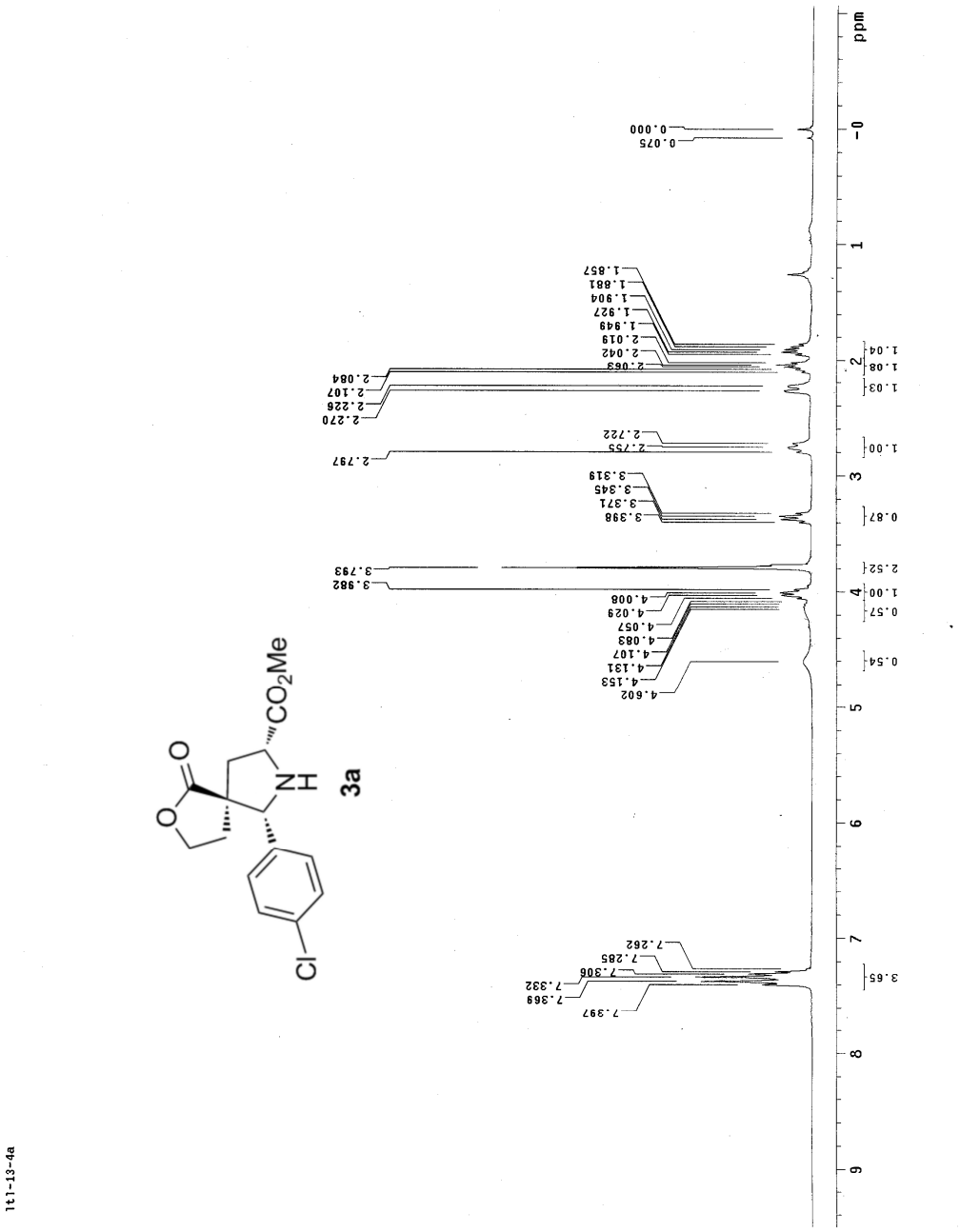
The catalyst structure was examined by ^{31}P NMR study. As shown in Figure 1, ^{31}P NMR spectrum of free ligand (*R*)-DTBM-BIPHEP in $CDCl_3$ showed a singlet peak at -15.19 ppm. After addition of 1 equiv of $Cu(MeCN)_4BF_4$ to the solution, a new single peak at -0.83 ppm was observed with disappearance of the free ligand peak (Figure 2). It showed that (*R*)-DTBM-BIPHEP/$Cu(MeCN)_4BF_4$ complex formed.

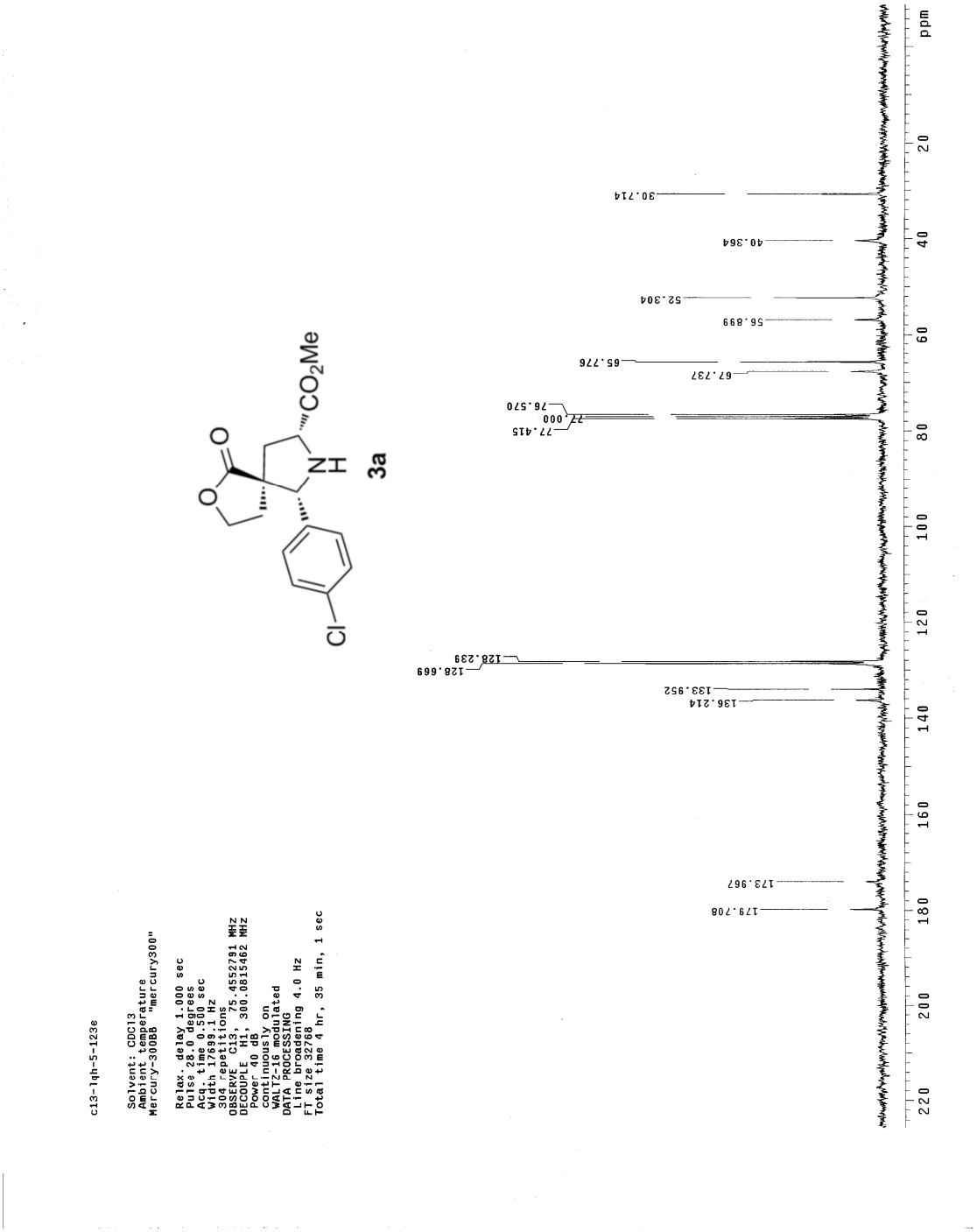
VII. References

1. J. Zhou, A. M. Schmidt, H. Ritter, *Macromolecules* **2010**, *43*, 939-942.

2. Y. Oderaotoshi, W. Cheng, S. Fujitomi, Y. Kasano, S. Minakata and M. Komatsu, *Org. Lett.*, **2003**, *5*, 5043.

VIII. ^{1}H NMR and ^{13}C NMR Spectra.

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Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
3504 repetitions
OBSERVE C13, 75.4552791 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
Cl
3a
30.714
40.364
52.304
56.899
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77.000
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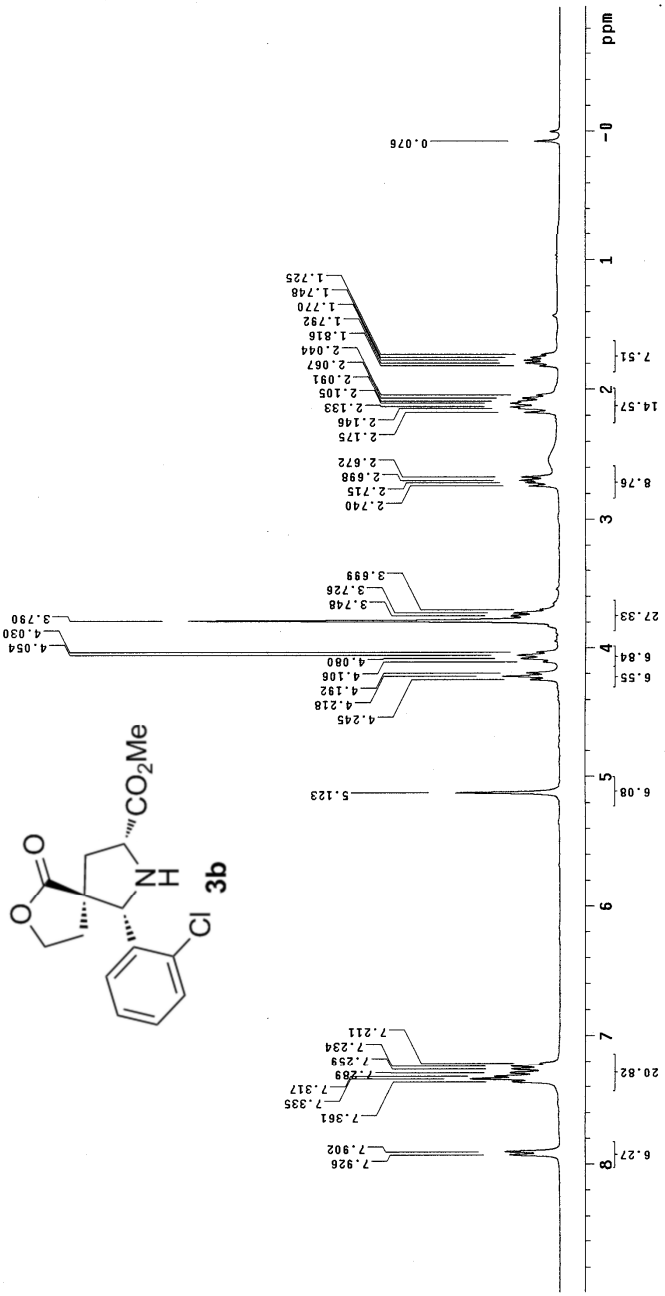
128.669
133.952
136.214
173.967
179.708
ppm


ppm
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1.725
1.748
1.770
1.792
1.816
2.044
2.067
2.091
2.105
2.133
2.146
2.175
2.672
2.698
2.715
2.740
3.699
3.726
3.748
3.790
4.054
4.080
4.106
4.192
4.218
4.245
5.123
7.211
7.234
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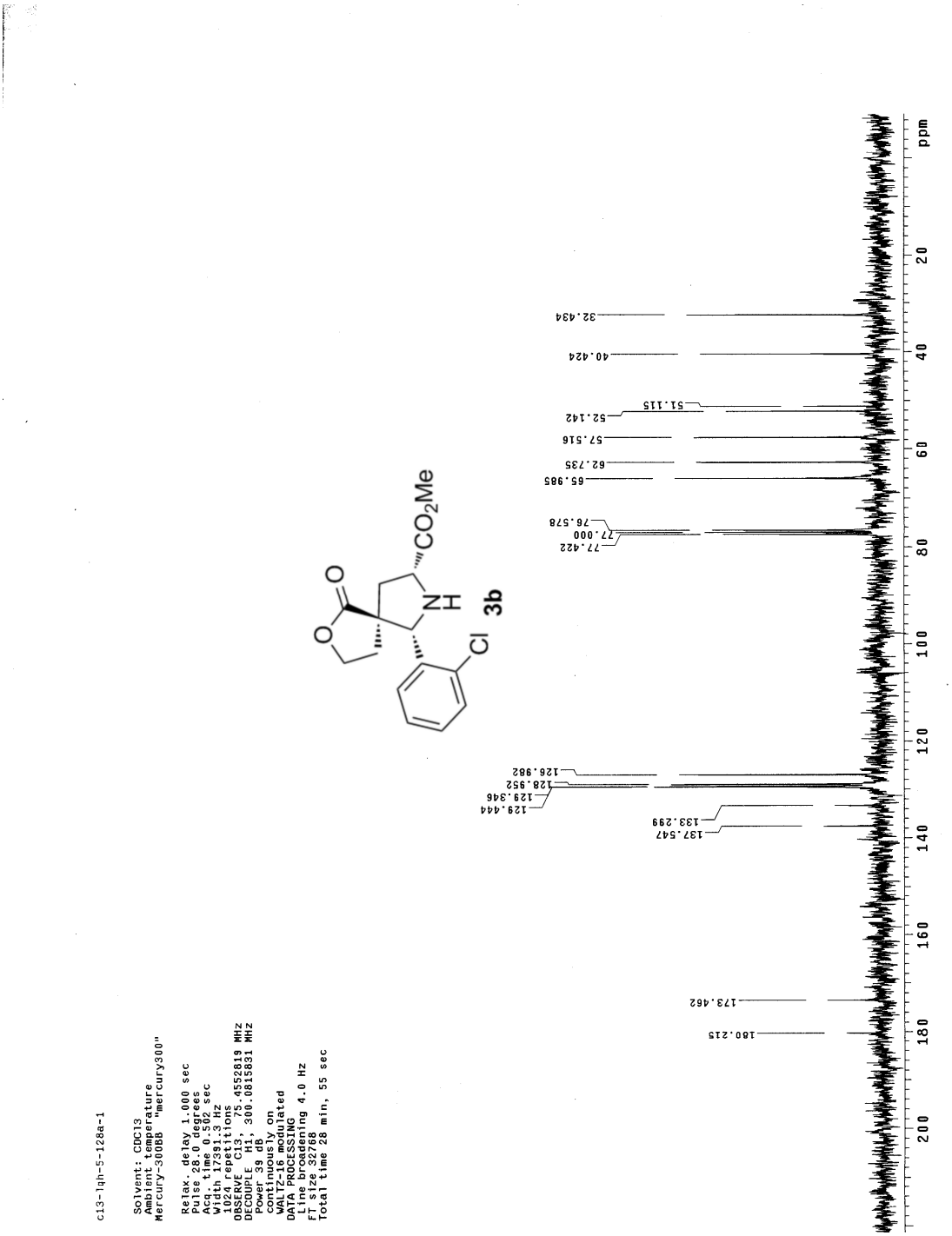
7.926
CO2Me
O
O
N
H
Cl
3b


c13-lqh-5-128a-1
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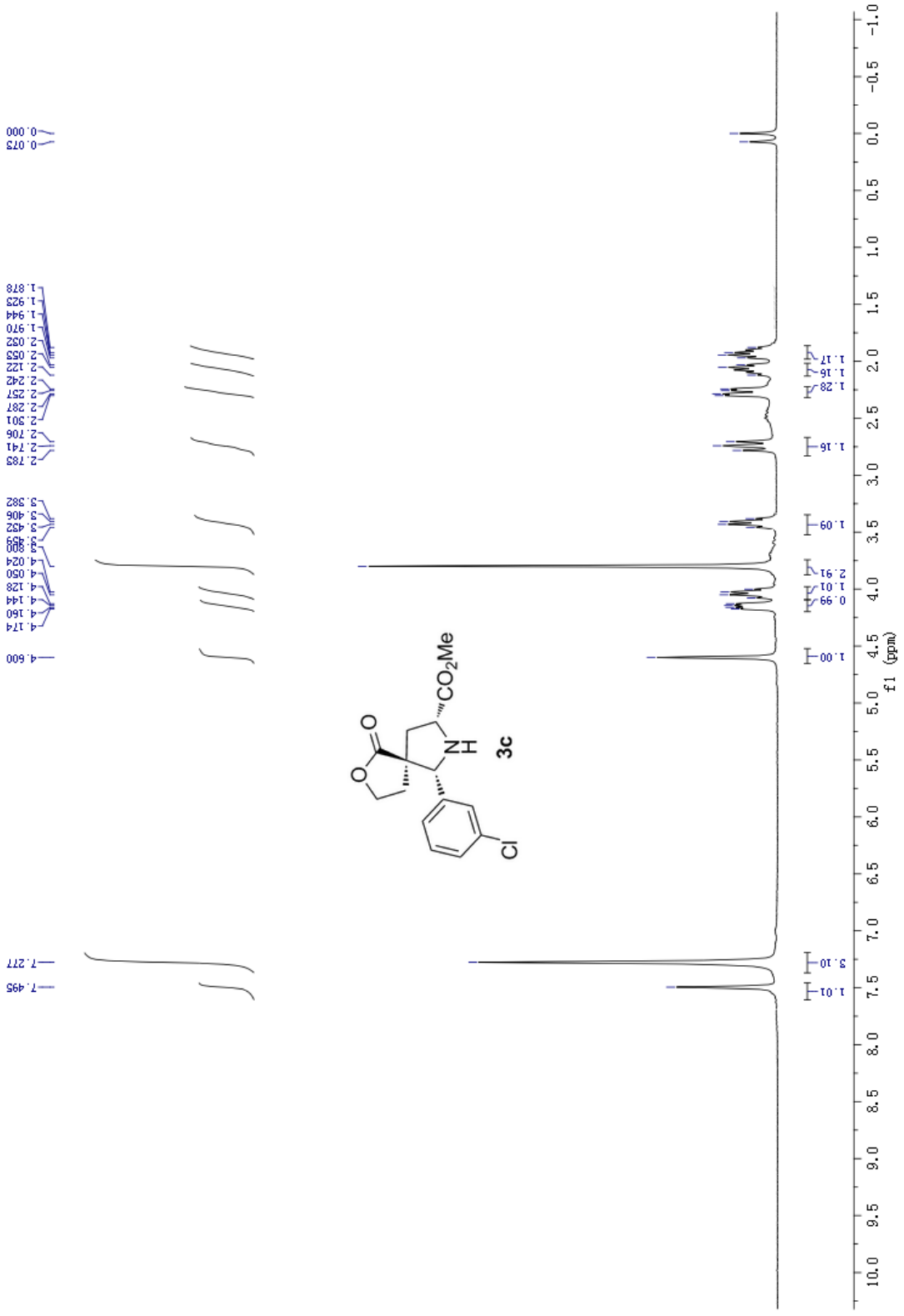
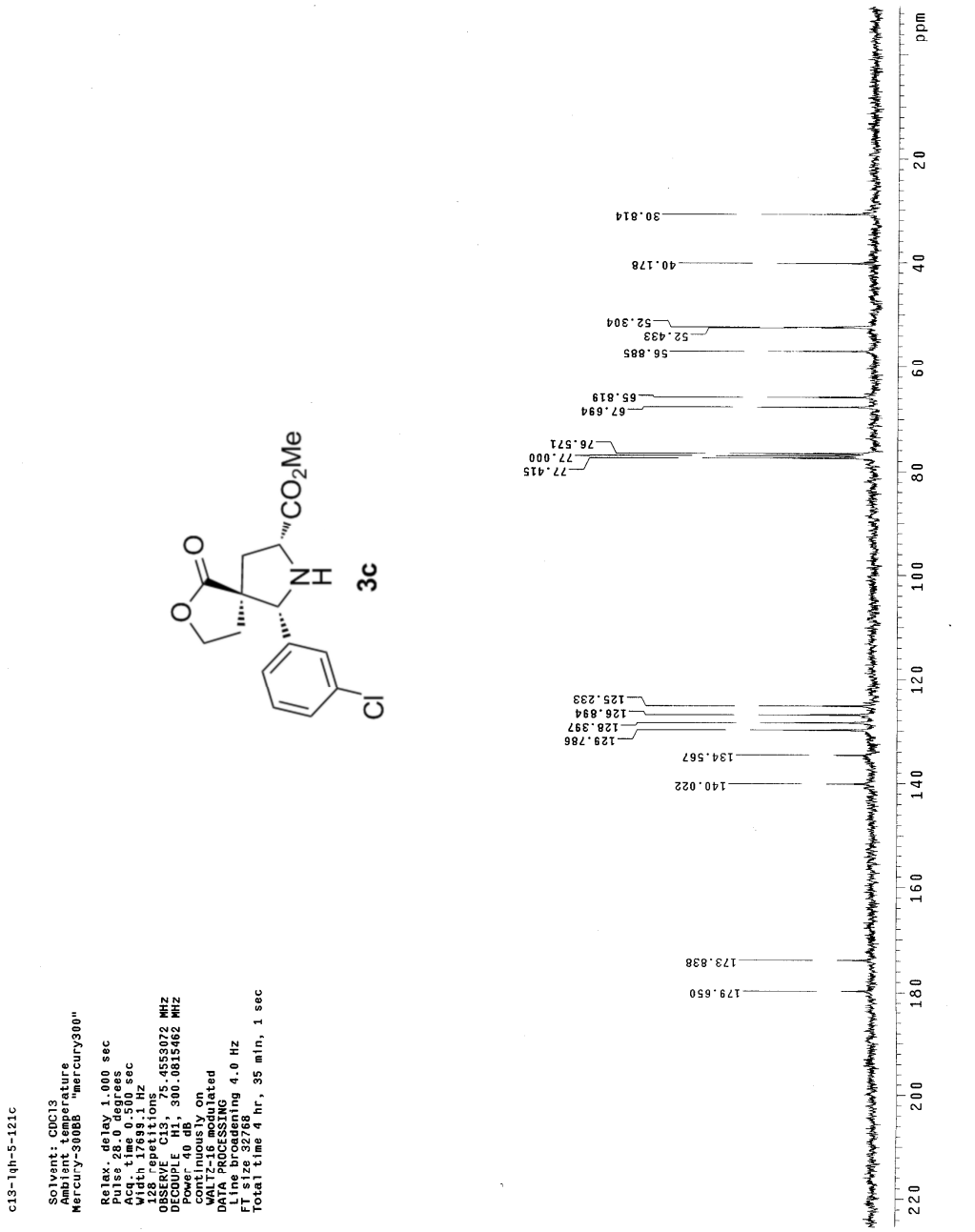
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.582 sec
Width 17391.3 Hz
1024 repetitions
OBSERVE C13, 75.4552819 MHz
DECOUPLE H1, 300.0815831 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 55 sec
CO2Me
N
H
O
O
Cl
3b
32.434
40.424
51.115
52.142
57.516
62.735
65.985
76.578
77.000
77.422
126.982
128.952
129.346
129.444
133.299
137.547
173.462
180.215
ppm


0.000
0.073
1.878
1.923
1.944
1.970
2.032
2.053
2.122
2.242
2.257
2.287
2.301
2.706
2.741
2.783
3.382
3.406
3.432
3.459
3.800
4.024
4.050
4.128
4.144
4.160
4.174
4.600
7.277
7.495
CO2Me
N
H
O
O
Cl
3c
1.17
1.16
1.28
1.16
1.09
2.91
1.01
0.99
1.00
3.10
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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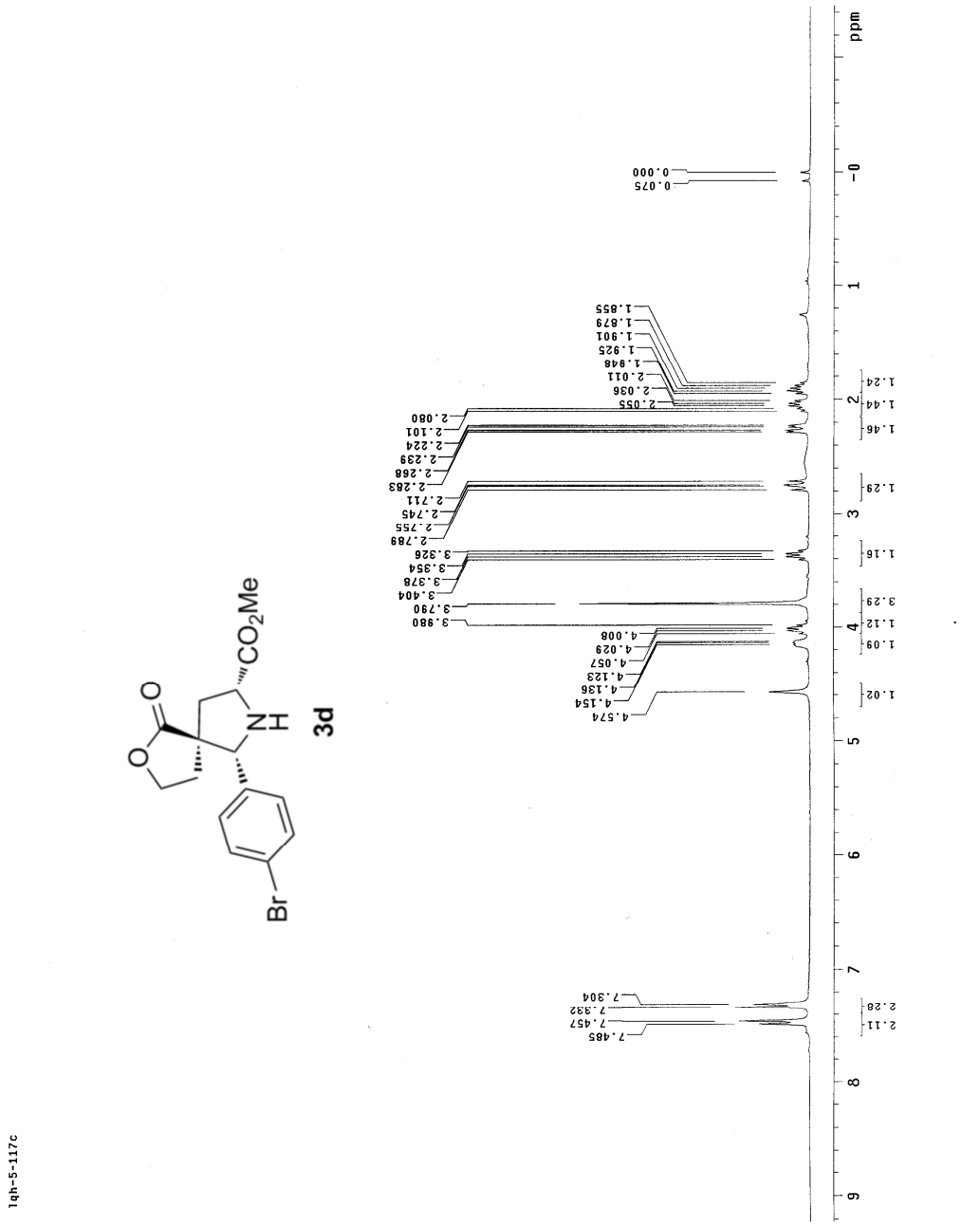
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4553072 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
3c
Cl
179.650
173.838
140.022
134.567
129.786
128.397
126.894
125.233
77.415
77.000
76.571
67.694
65.819
56.885
52.433
52.304
40.178
30.814
ppm


lqh-5-117c
3d
CO2Me
O
O
N
H
Br
ppm
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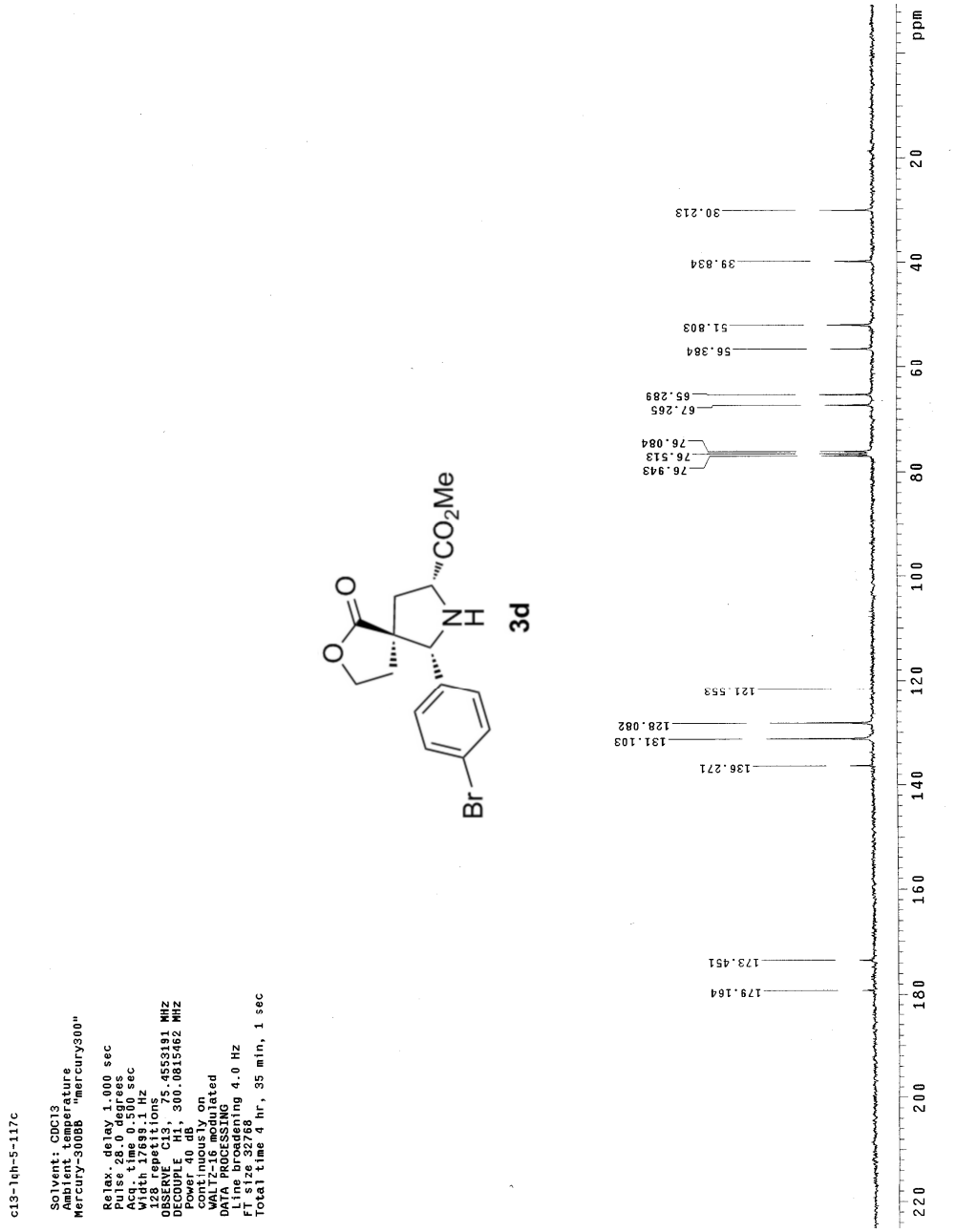
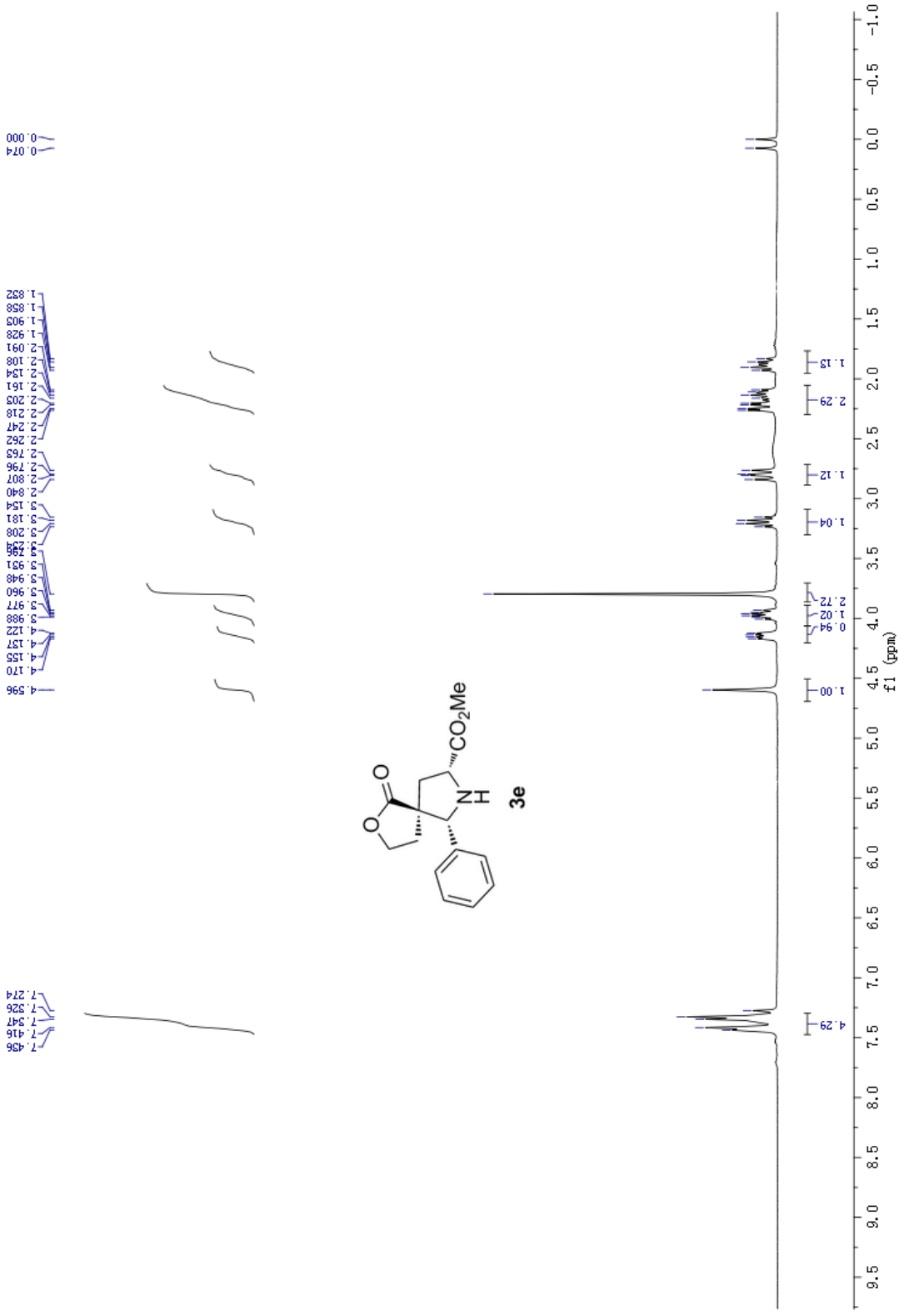
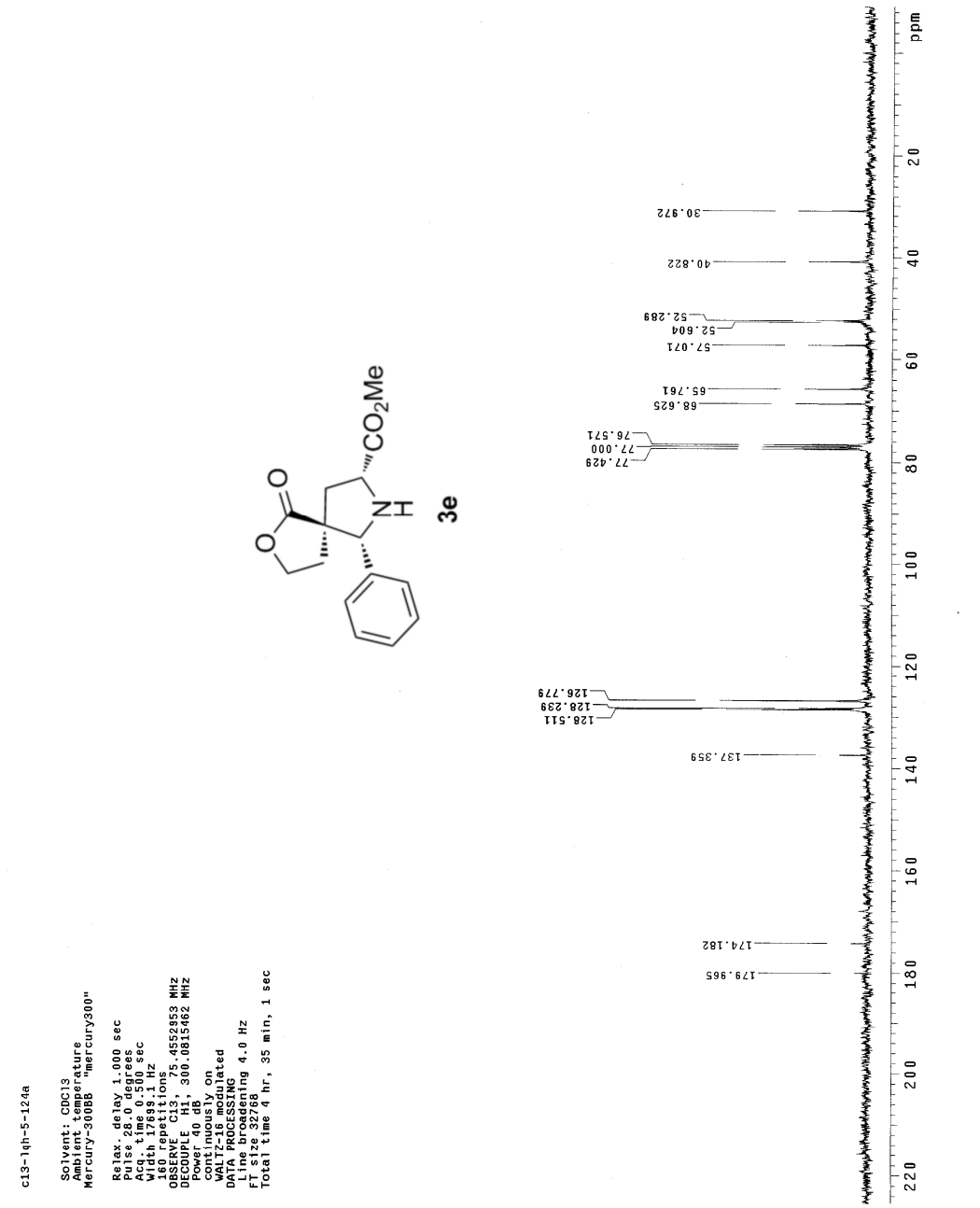
0.000
1.855
1.879
1.901
1.925
1.948
2.011
2.036
2.055
2.080
2.101
2.224
2.239
2.268
2.283
2.711
2.745
2.755
2.789
3.328
3.354
3.378
3.404
3.790
3.980
4.008
4.029
4.057
4.123
4.136
4.154
4.574
7.304
7.332
7.457
7.485
1.24
1.44
1.46
1.29
1.16
3.29
1.12
1.09
1.02
2.28
2.11


c13-lqh-5-117c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4553191 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
N
H
O
O
Br
3d
30.213
39.834
51.803
56.384
65.289
67.265
76.084
76.513
76.943
121.553
128.082
131.103
136.271
173.451
179.164
ppm
220
200
180
160
140
120
100
80
60
40
20


CO2Me
3e
f1 (ppm)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Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17361.1 Hz
160 repetitions
OBSERVE C13, 75.4552853 MHz
DECOUPLE H1, 300.0815402 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
H
O
O
3e
30.972
40.822
52.289
52.604
57.071
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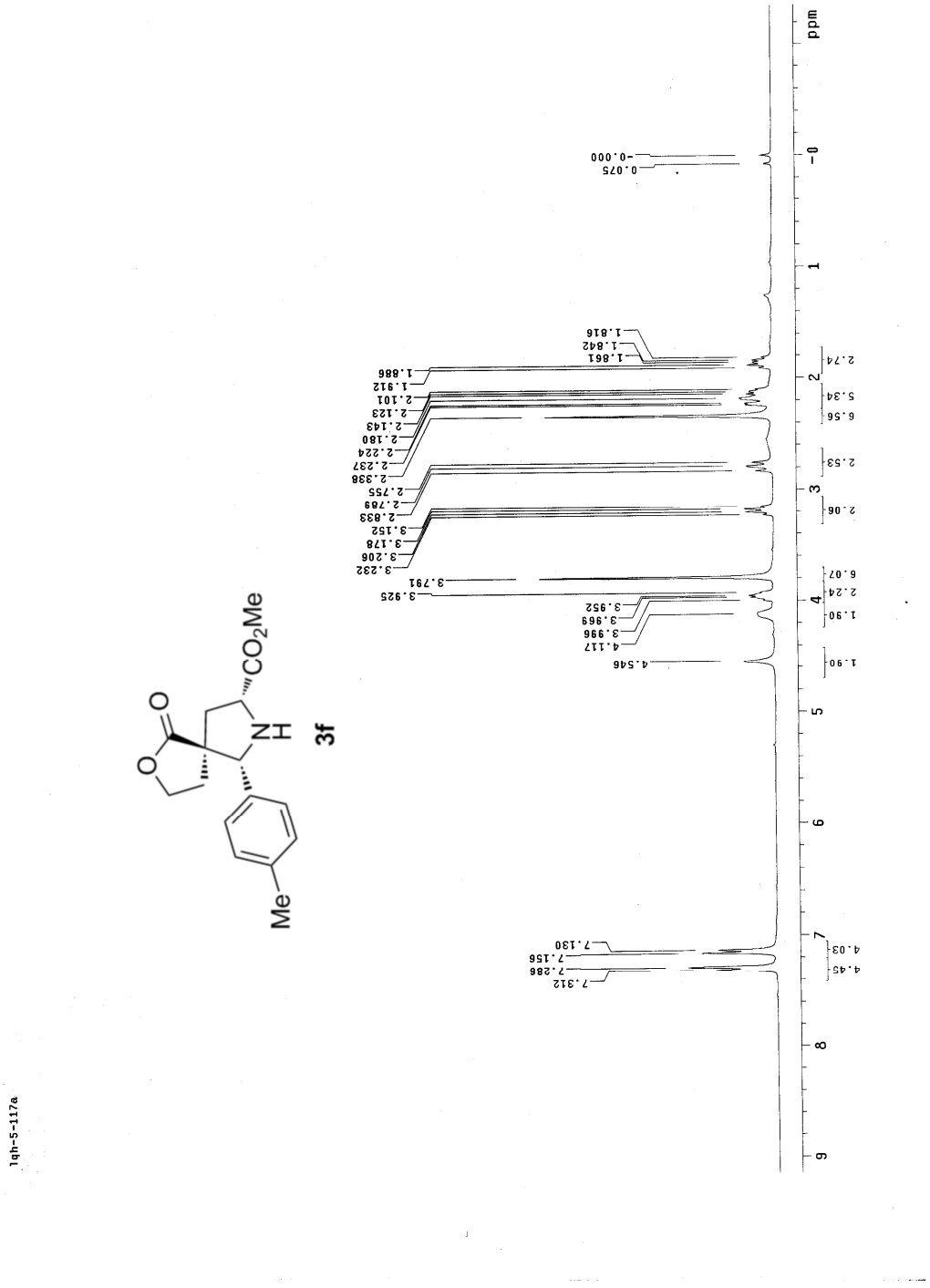
128.239
128.511
137.359
174.182
179.965
ppm


CO2Me
O
O
N
H
Me
3f
lqh-5-117a
ppm
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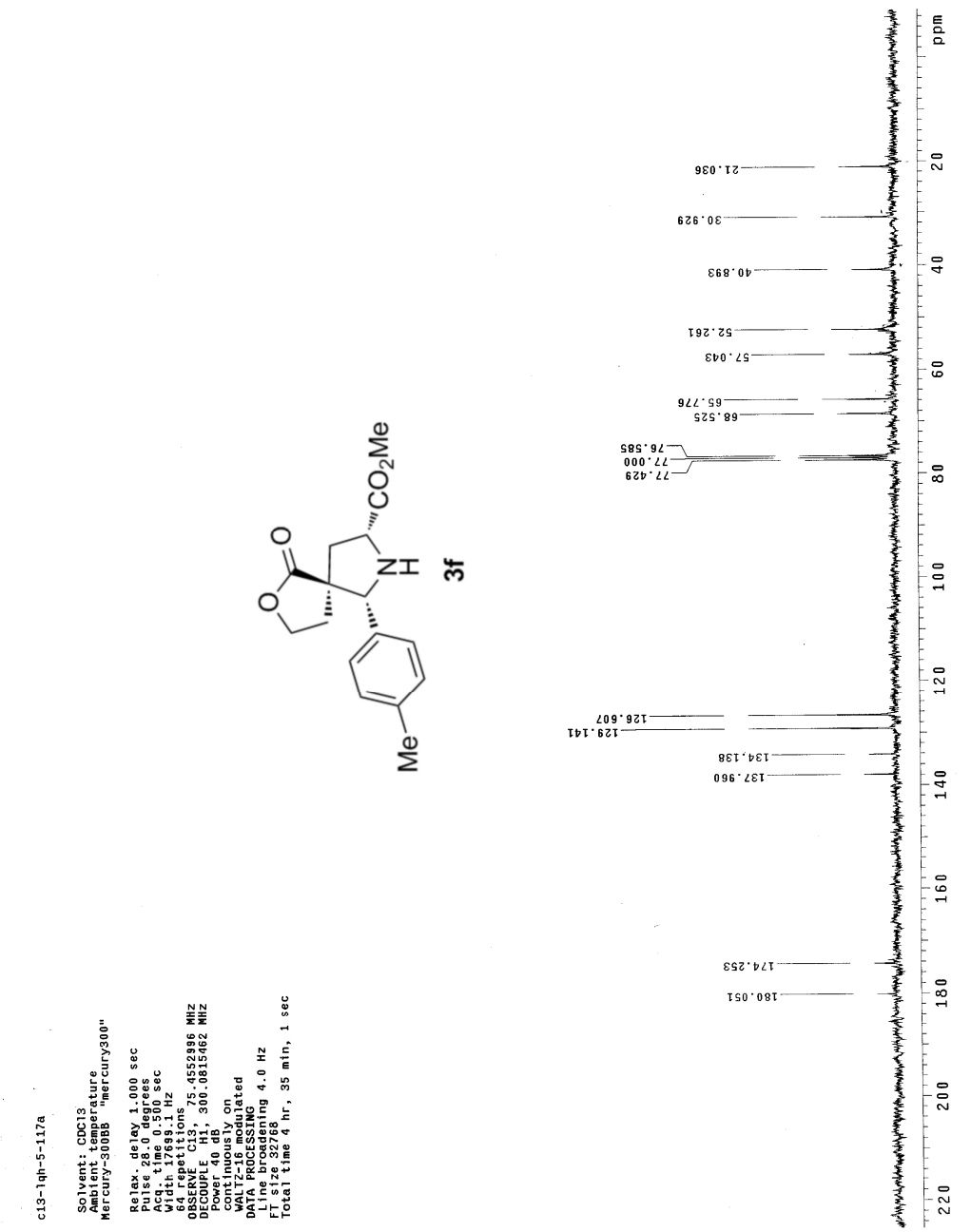
1.861
1.886
1.912
2.101
2.123
2.143
2.180
2.224
2.237
2.338
2.755
2.789
2.833
3.152
3.178
3.206
3.232
3.791
3.925
3.952
3.969
3.996
4.117
4.546
7.130
7.156
7.286
7.312
2.74
5.34
6.56
2.53
2.06
6.07
2.24
1.90
1.90
4.03
4.45


c13-1qh-5-117a
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
64 repetitions
OBSERVE C13, 75.4552986 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
N
H
O
O
Me
3f
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134.138
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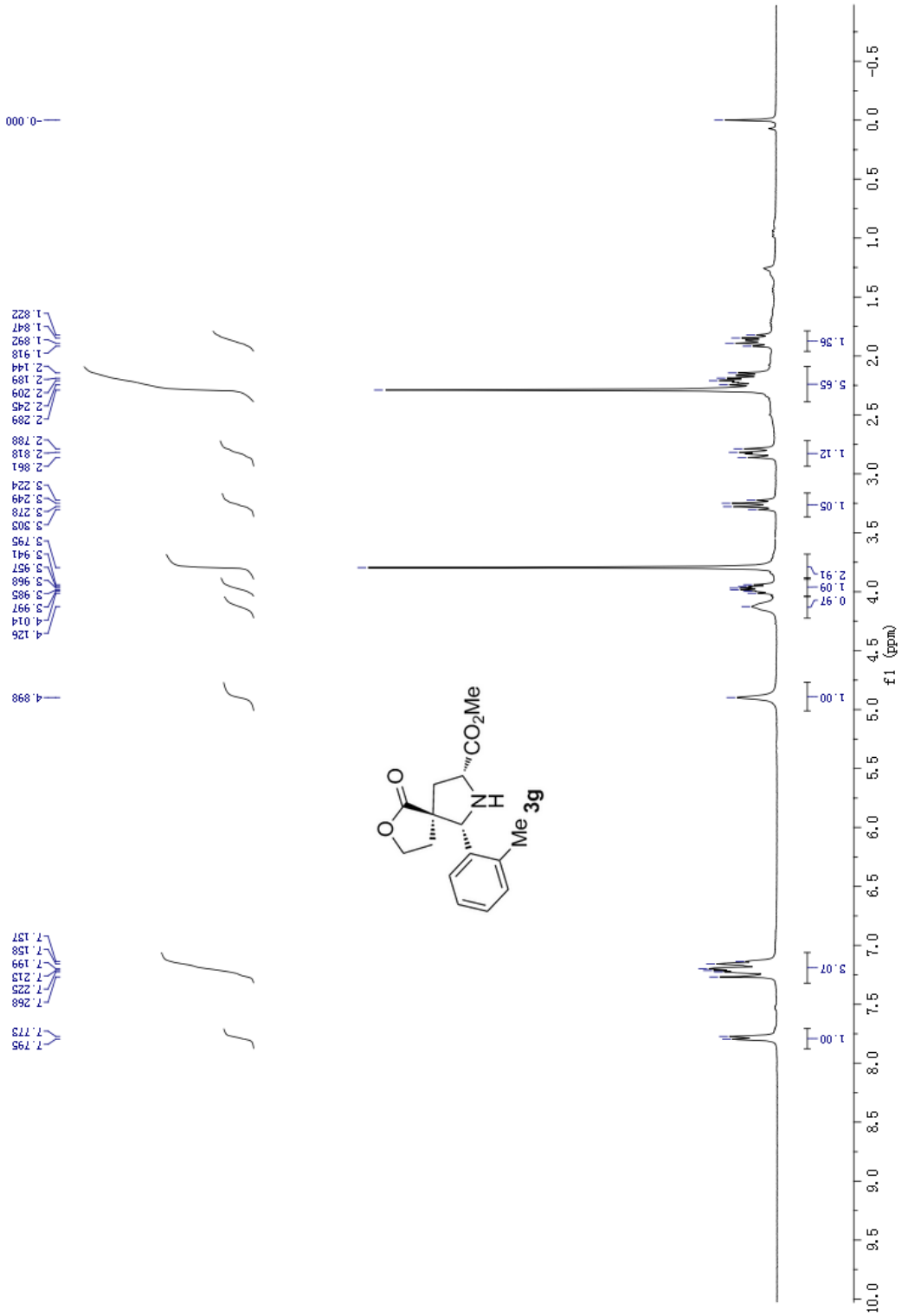
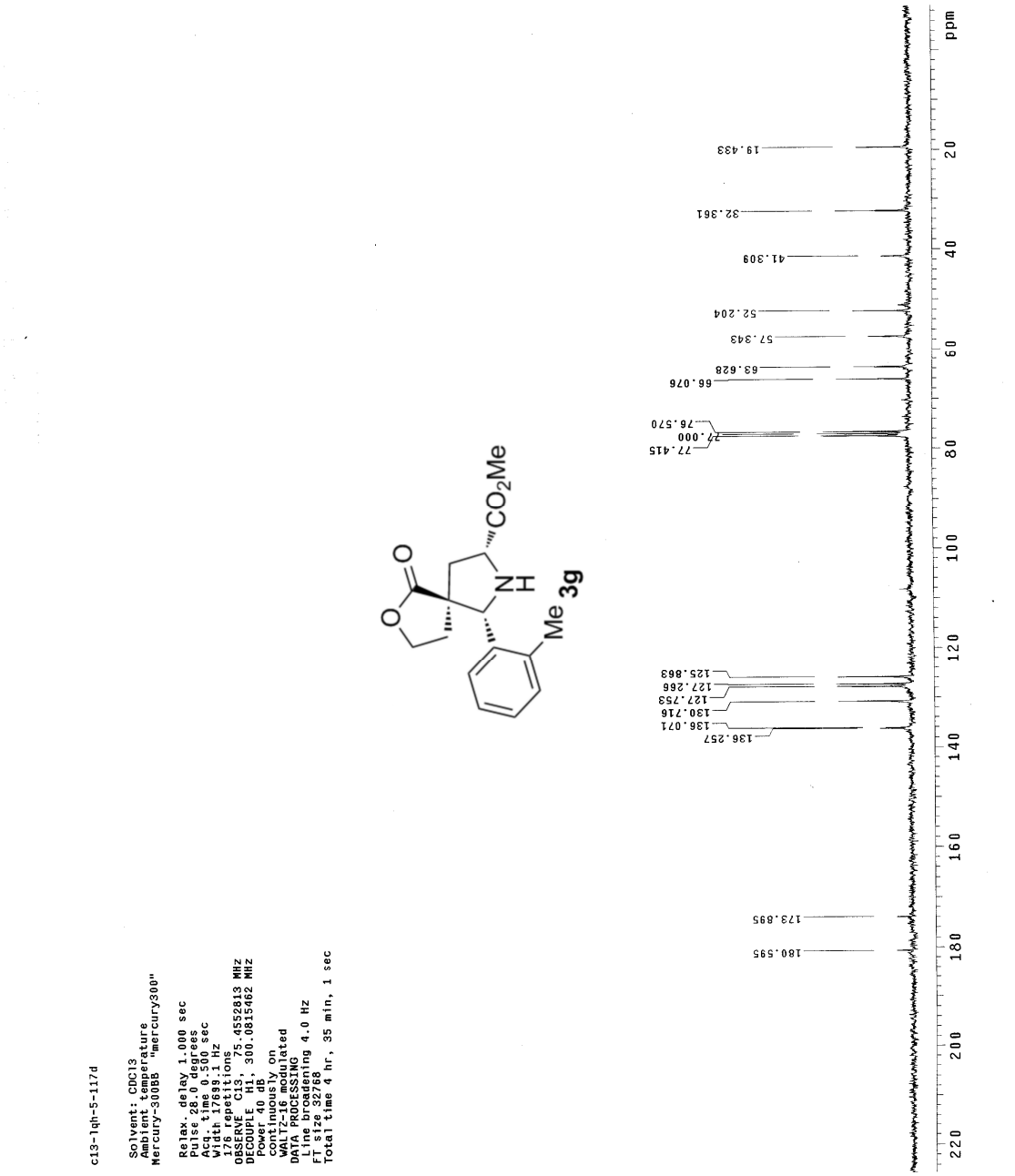
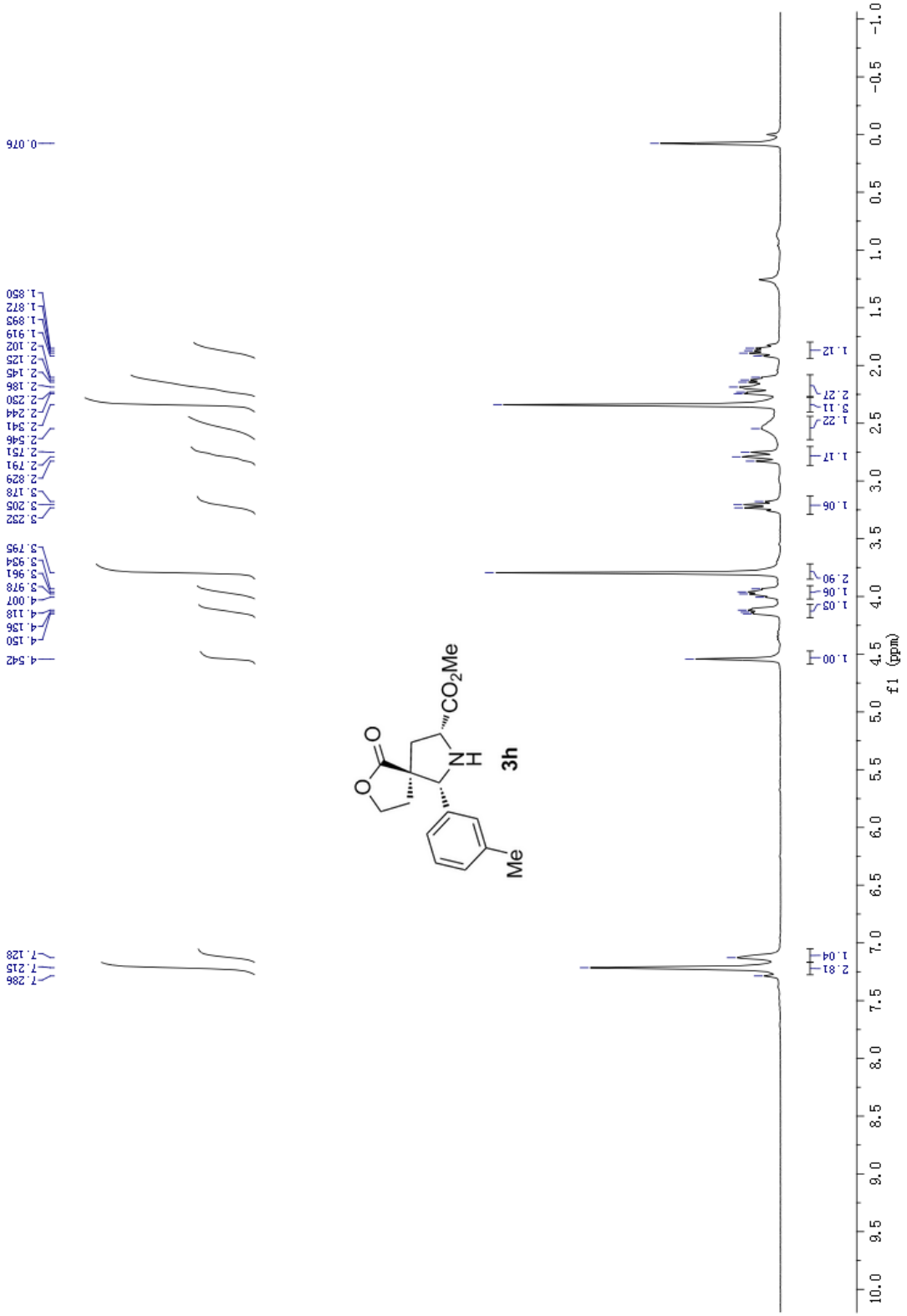
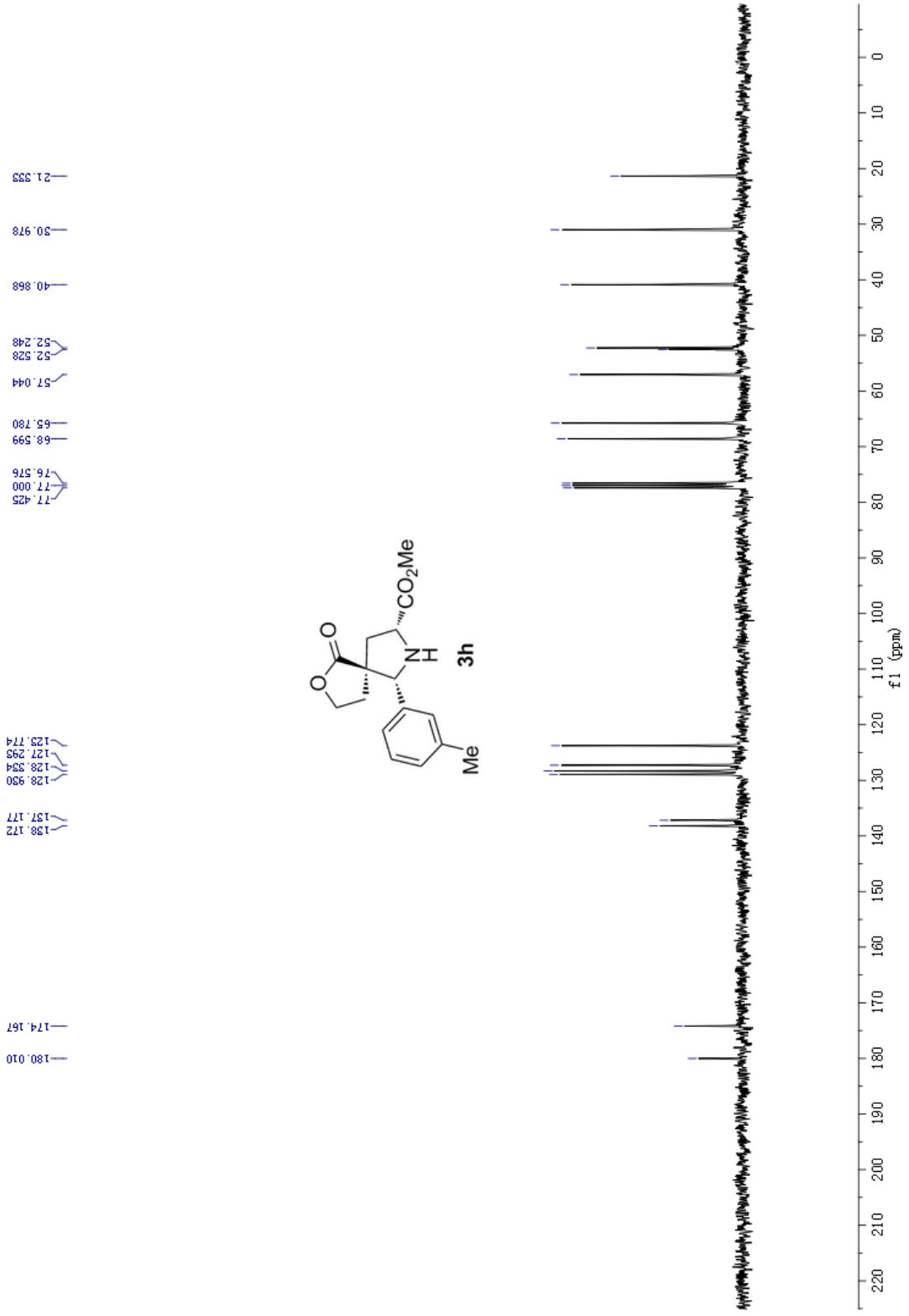
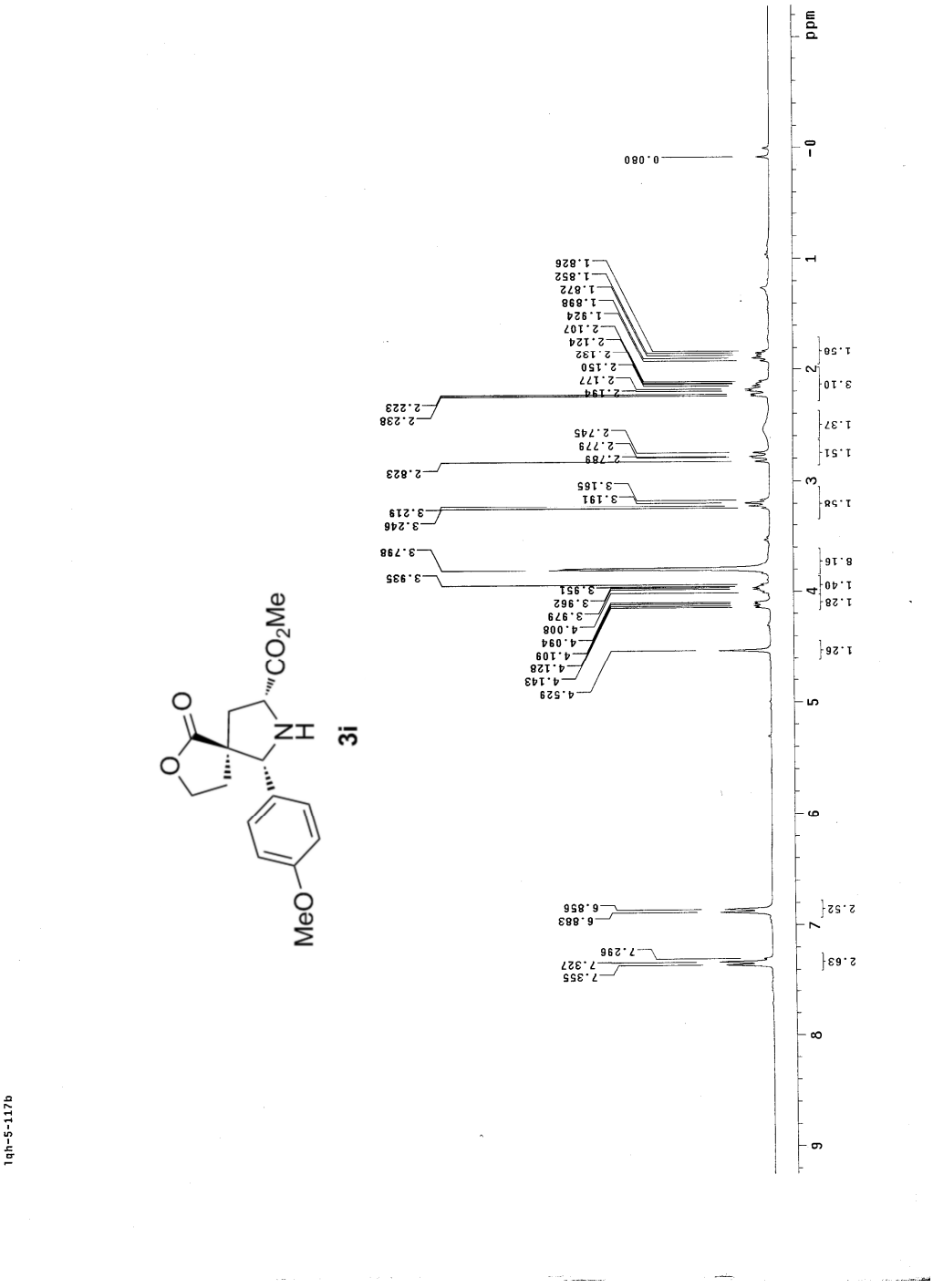
57.043
52.261
40.893
30.929
21.036
ppm

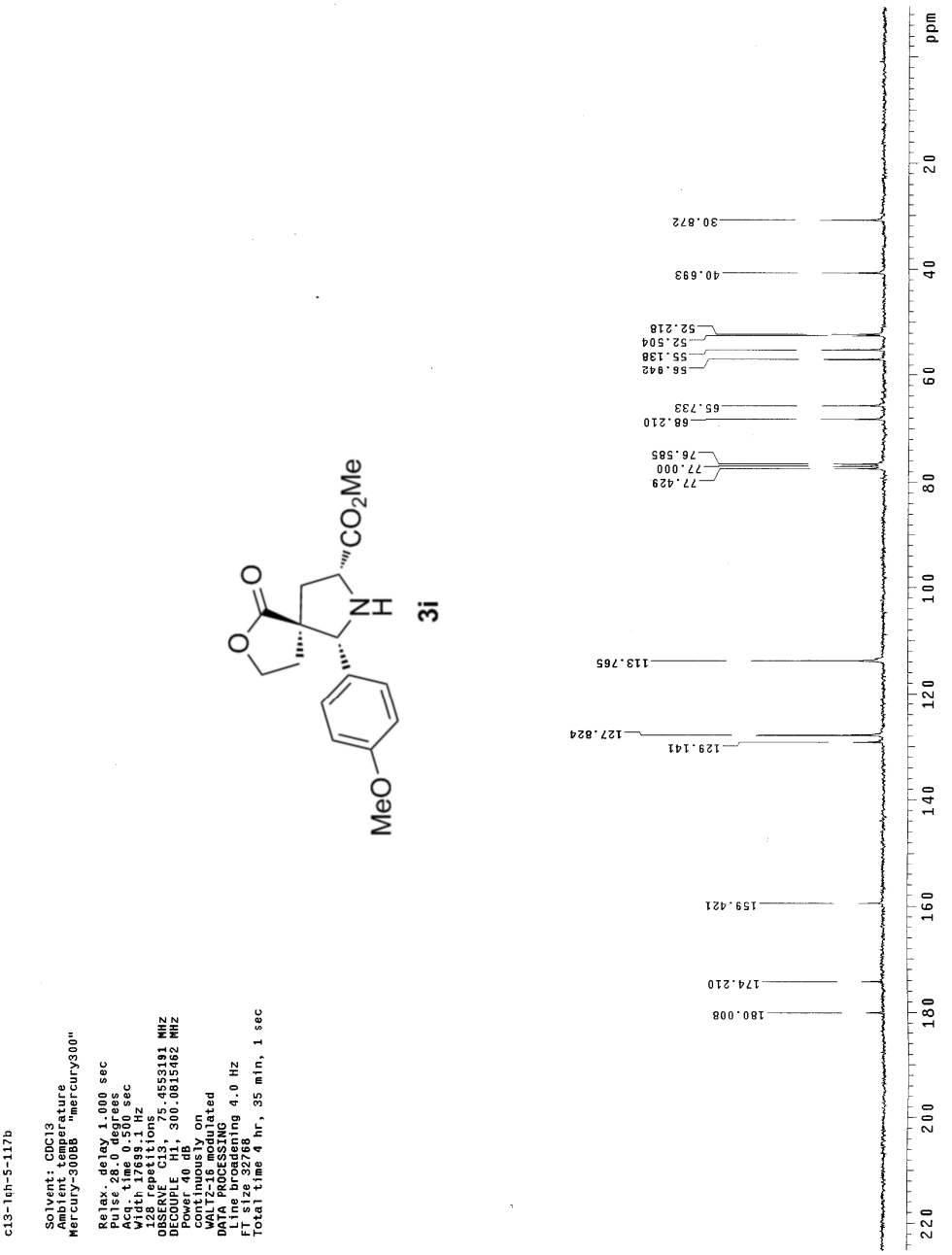

CO2Me
H
N
O
O
Me
3g
f1 (ppm)

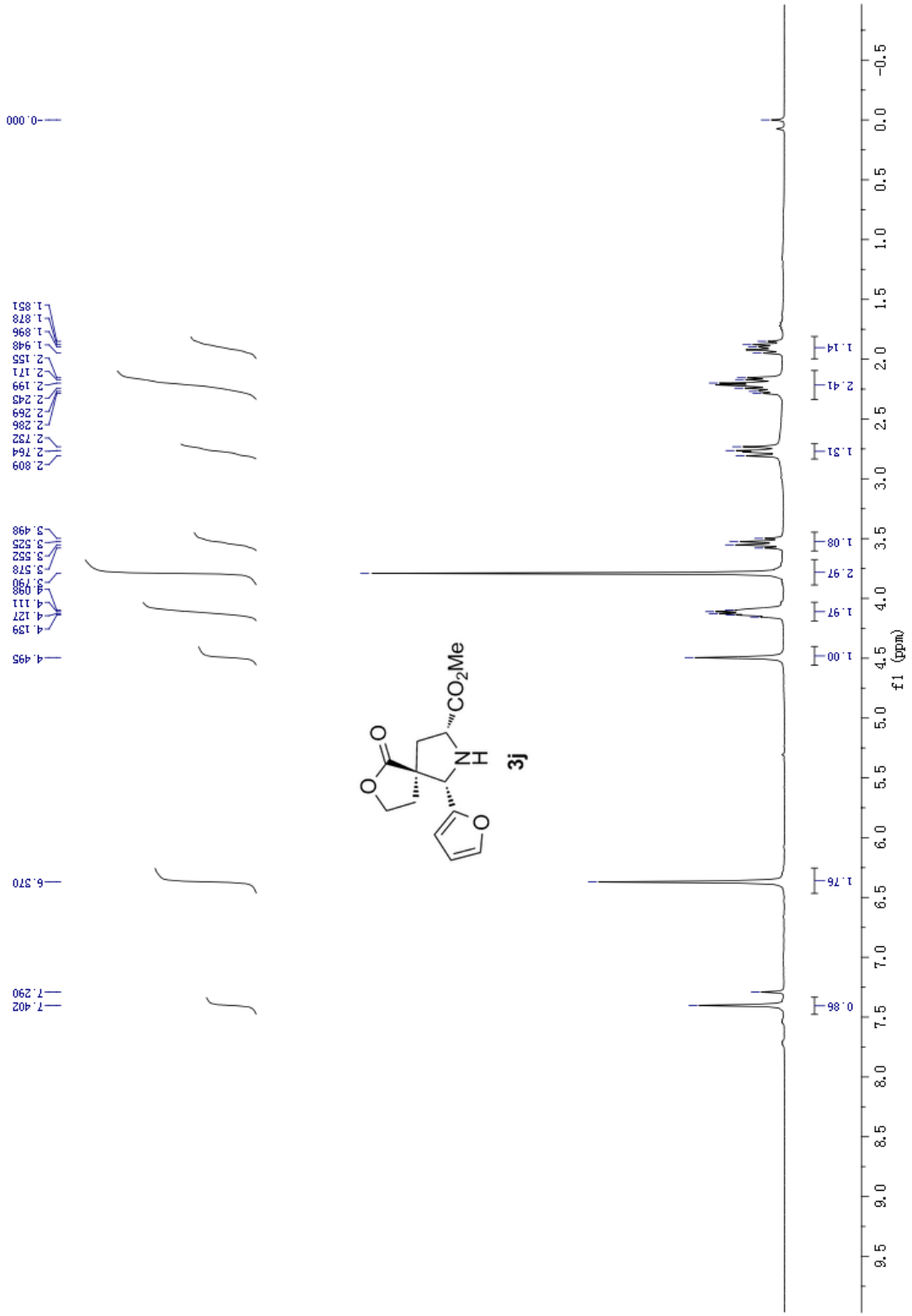

c13-lqh-5-117d
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.000 sec
Width 17689.1 Hz
176 repetitions
OBSERVE C13, 75.4552813 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
Me
3g
19.433
32.361
41.309
52.204
57.343
63.628
66.076
76.570
77.000
77.415
125.863
127.266
127.753
130.716
136.071
136.257
173.895
180.595
ppm

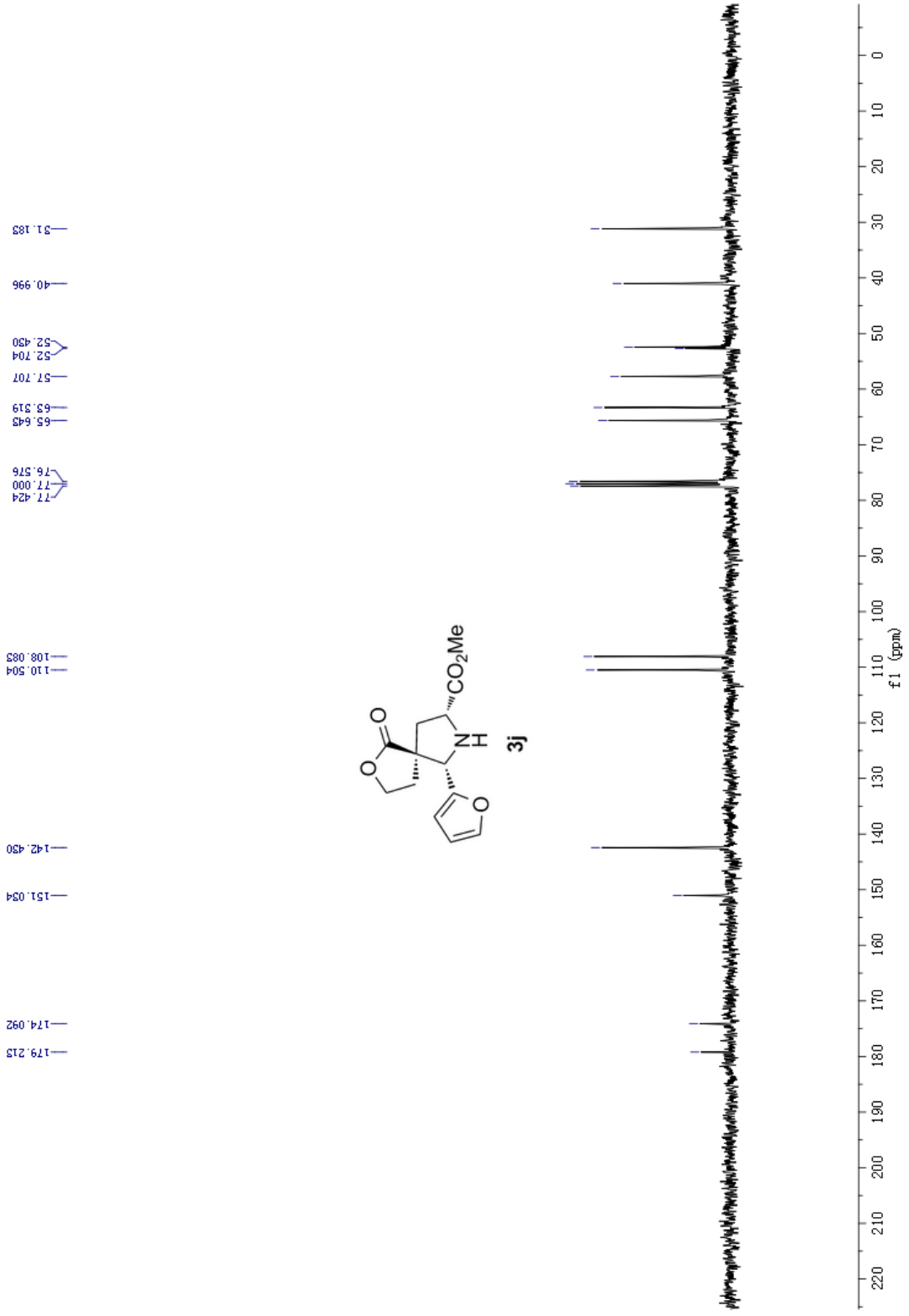

CO2Me
O
O
N
H
Me
3h
f1 (ppm)

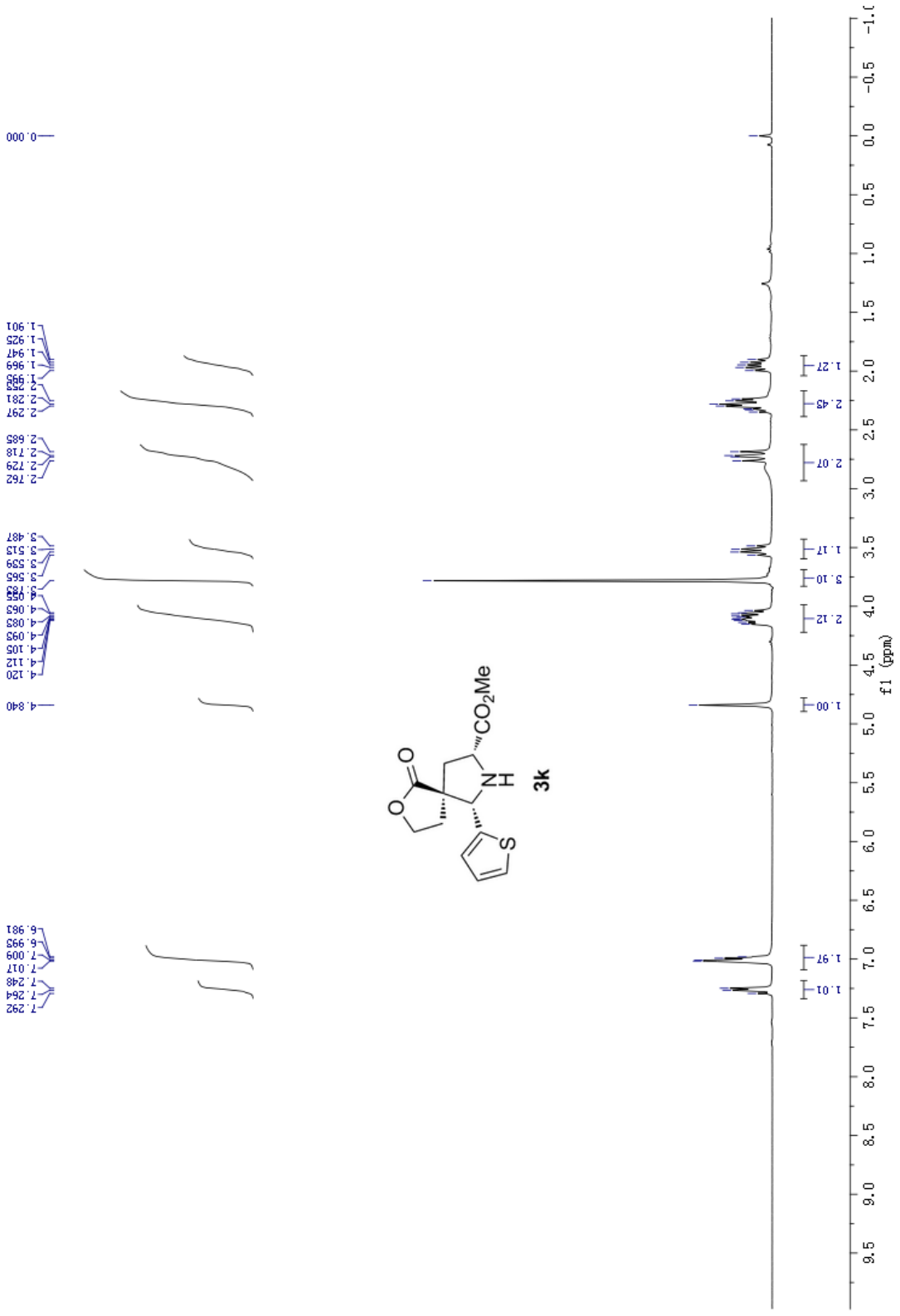

21.333
30.978
40.868
52.248
52.528
57.044
65.780
68.599
76.576
77.000
77.425
123.774
127.293
128.334
128.930
137.177
138.172
174.167
180.010
CO2Me
O
O
N
H
Me
3h
f1 (ppm)

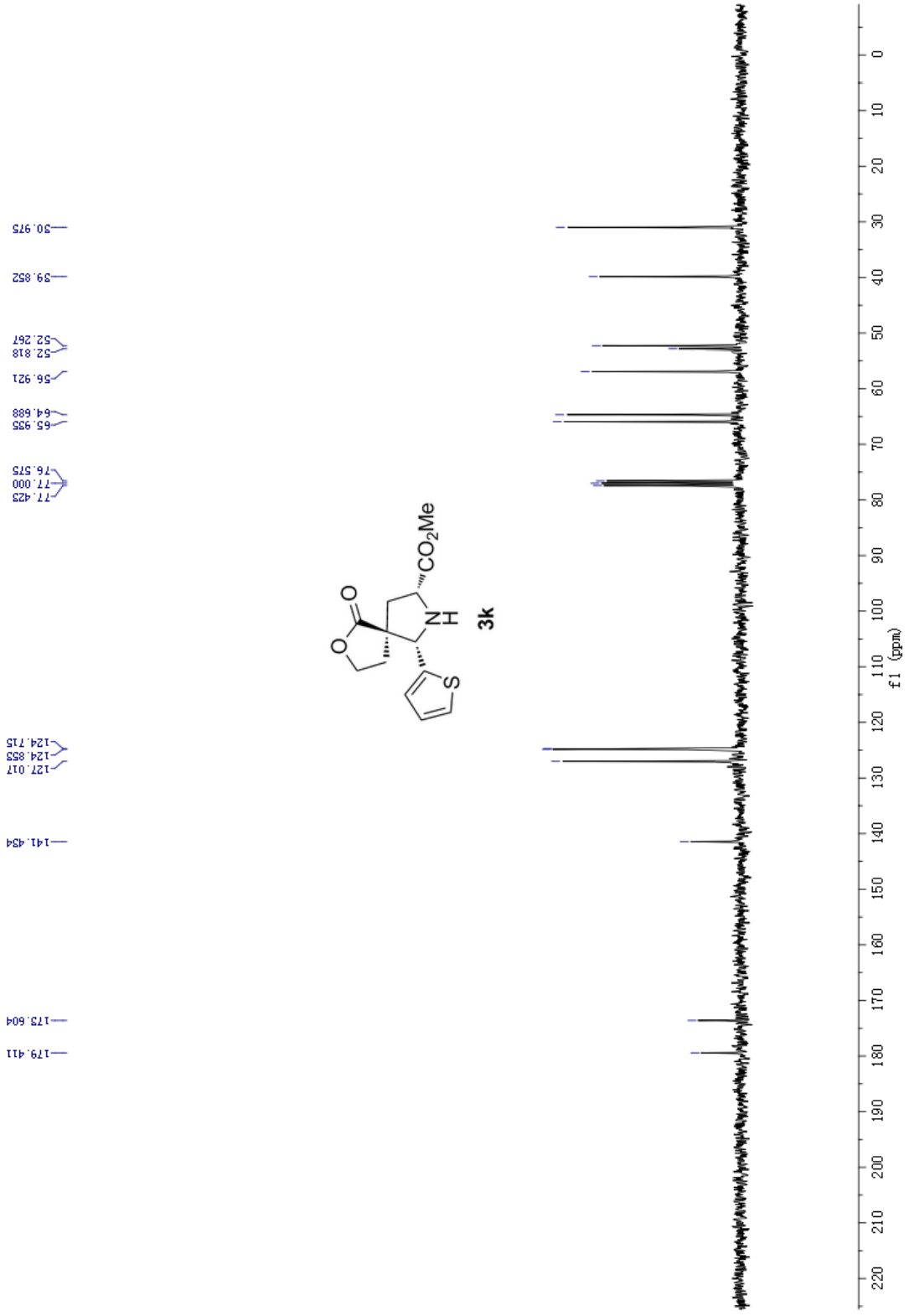

CO2Me
O
O
N
H
MeO
3i
1qh-5-117b
ppm
0.080
1.826
1.852
1.872
1.898
1.924
2.107
2.124
2.132
2.150
2.177
2.194
2.223
2.238
2.745
2.779
2.789
2.823
3.165
3.191
3.219
3.246
3.798
3.935
3.951
3.962
3.979
4.008
4.094
4.109
4.128
4.143
4.529
6.856
6.883
7.296
7.327
7.355
1.56
3.10
1.37
1.51
1.58
8.16
1.40
1.28
1.26
2.52
2.63

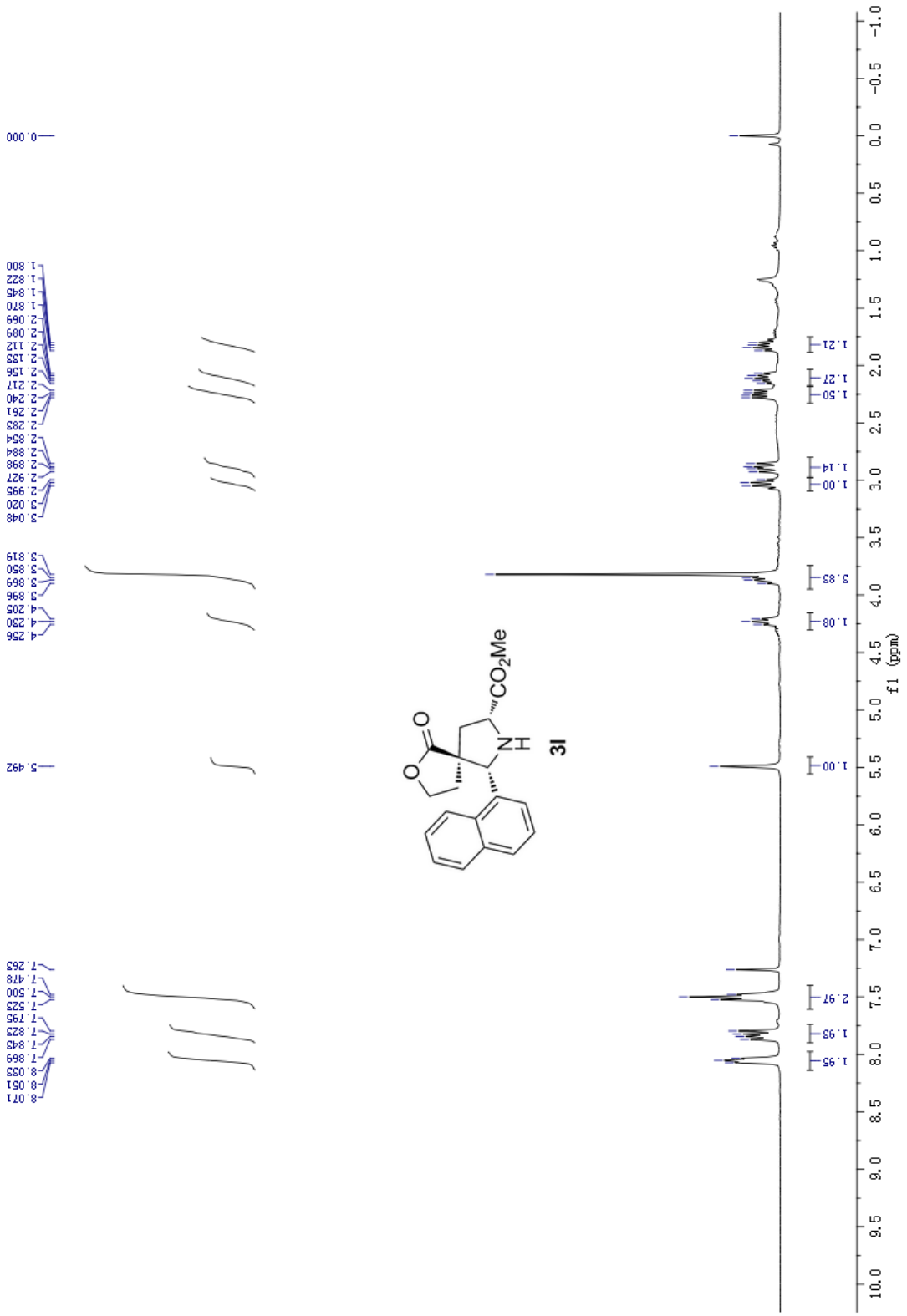

c13-leh-5-117b
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.000 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4553191 MHz
DECOUPLE H1, 300.0815462 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 4 hr, 35 min, 1 sec
CO2Me
N
H
O
O
MeO
3i
180.008
174.210
159.421
129.141
127.824
113.765
77.429
77.000
76.585
68.210
65.733
56.942
55.138
52.504
52.218
40.693
30.872
220
200
180
160
140
120
100
80
60
40
20
ppm

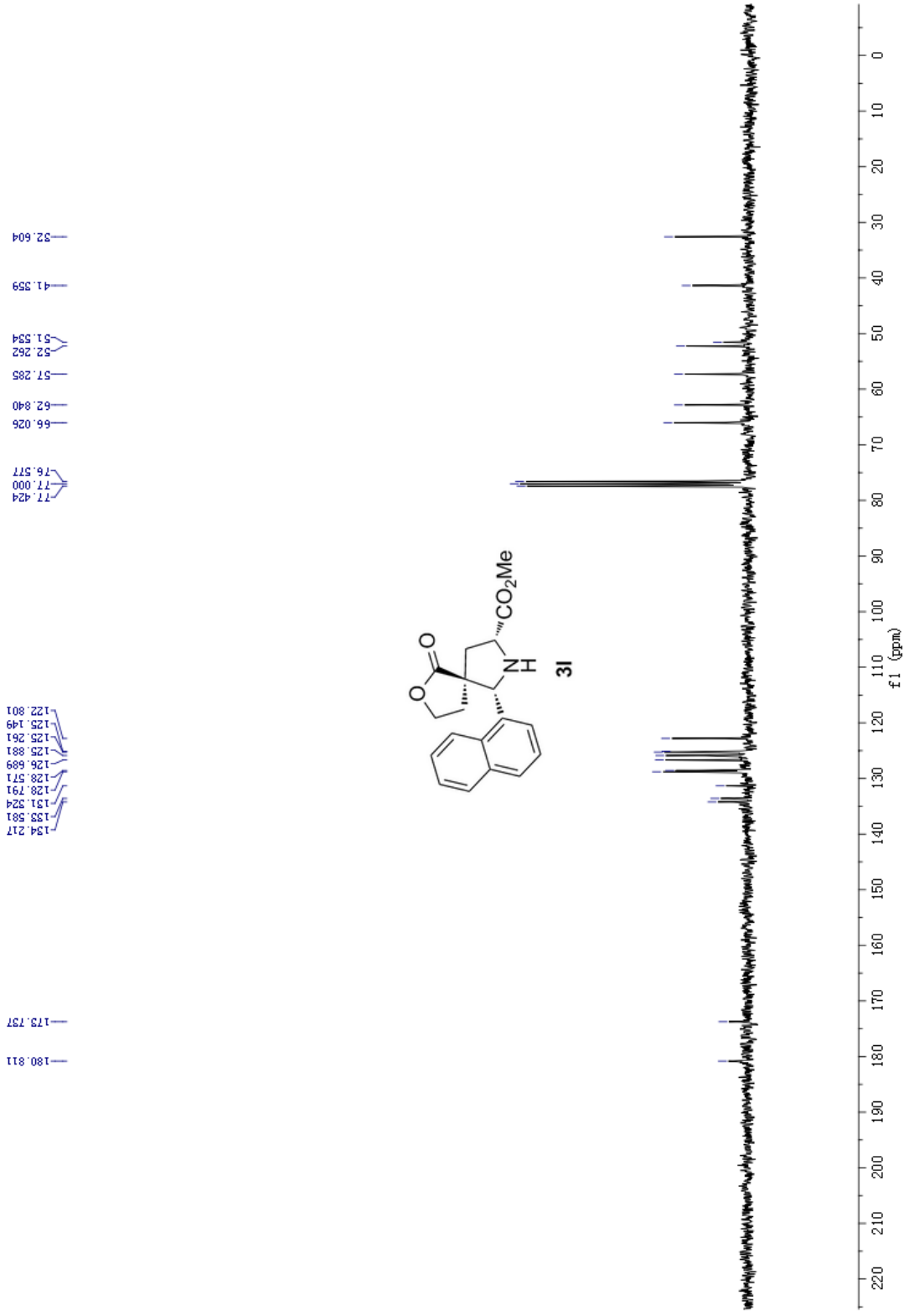

CO2Me
3j
f1 (ppm)

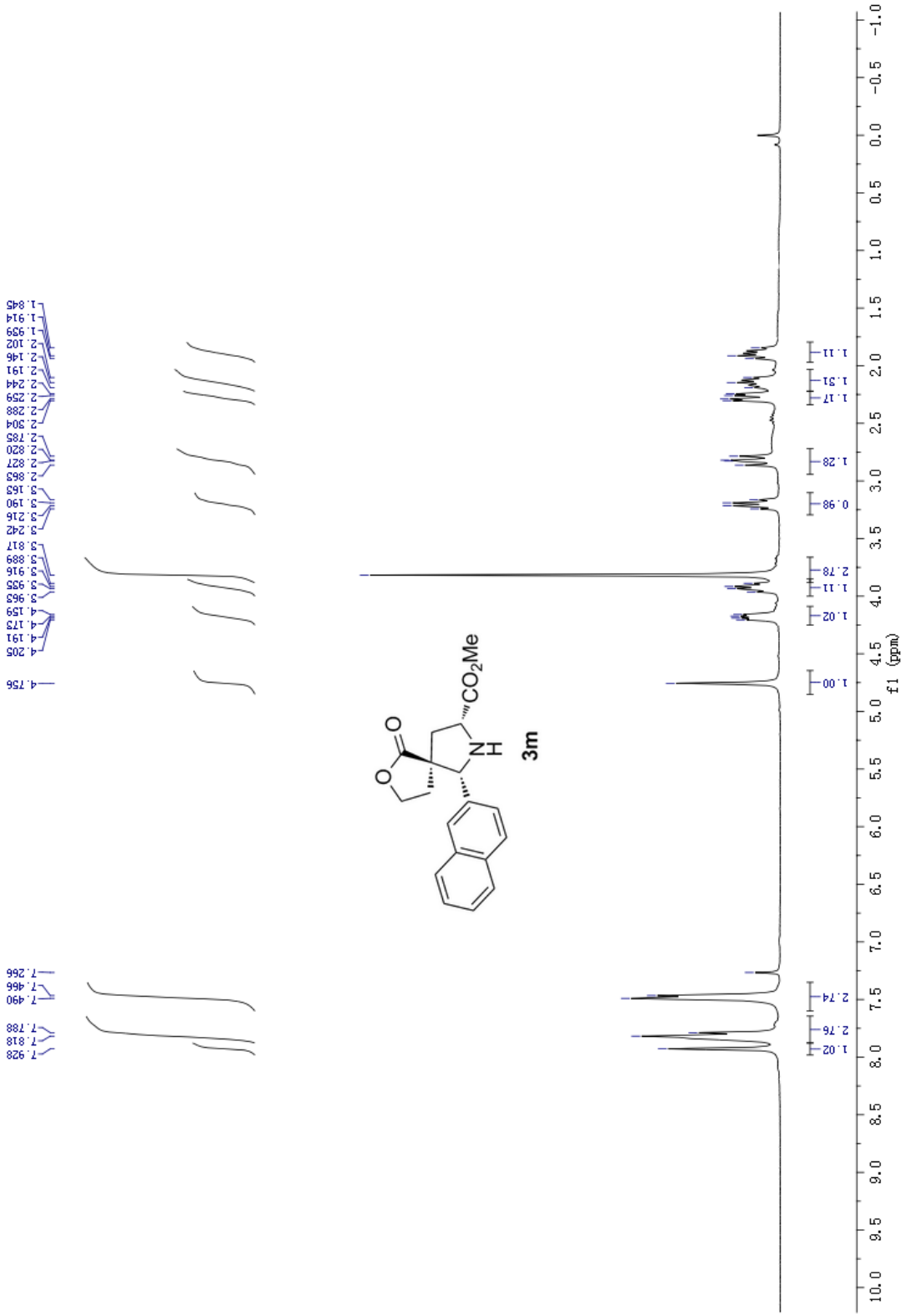

31.183
40.996
52.430
52.704
57.707
63.319
65.643
76.576
77.000
77.424
108.083
110.504
142.430
151.034
174.092
179.213
CO2Me
N
H
O
O
O
3j
f1 (ppm)

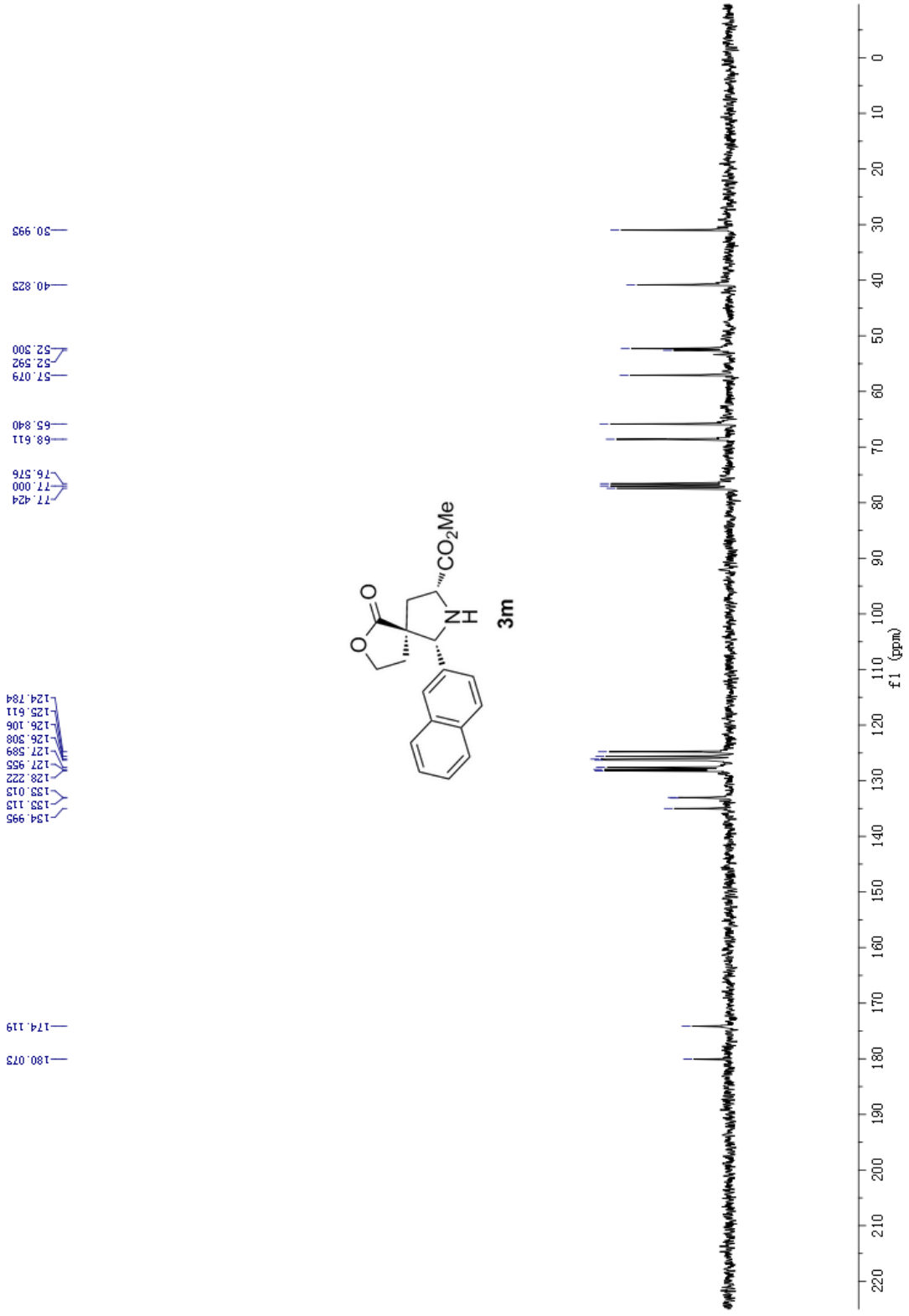

0.000
1.901
1.925
1.947
1.969
1.985
2.255
2.281
2.297
2.685
2.718
2.729
2.762
3.487
3.513
3.539
3.565
3.783
3.955
4.063
4.083
4.093
4.105
4.112
4.120
4.840
6.981
6.993
7.009
7.017
7.248
7.264
7.292
1.27
2.43
2.07
1.17
3.10
2.12
1.00
1.97
1.01
CO2Me
N
H
O
O
S
3k
f1 (ppm)

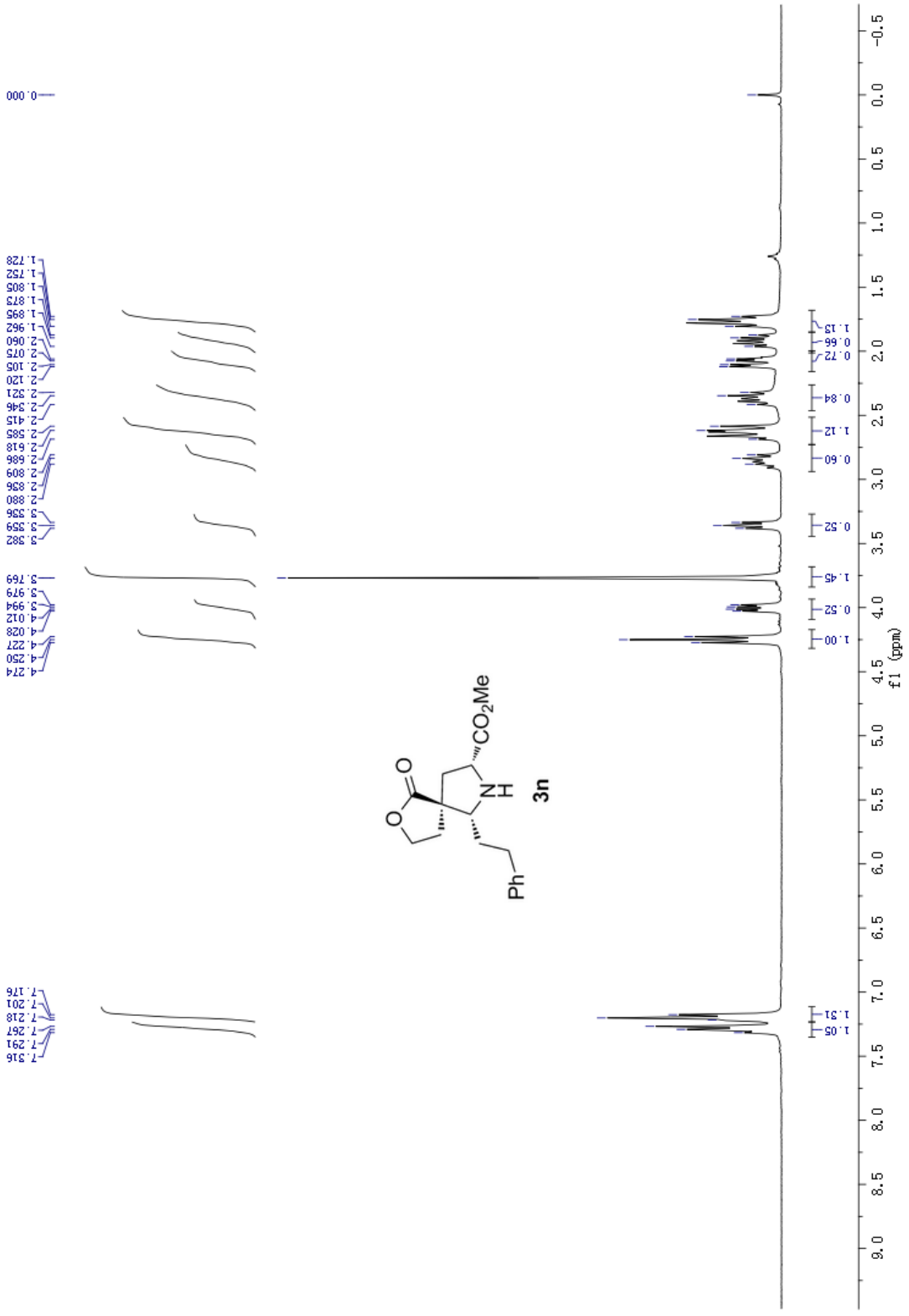

30.975
39.852
52.267
52.818
56.921
64.688
65.935
76.575
77.000
77.423
124.715
124.853
127.017
141.434
173.604
179.411
CO2Me
O
O
N
H
S
3k
f1 (ppm)

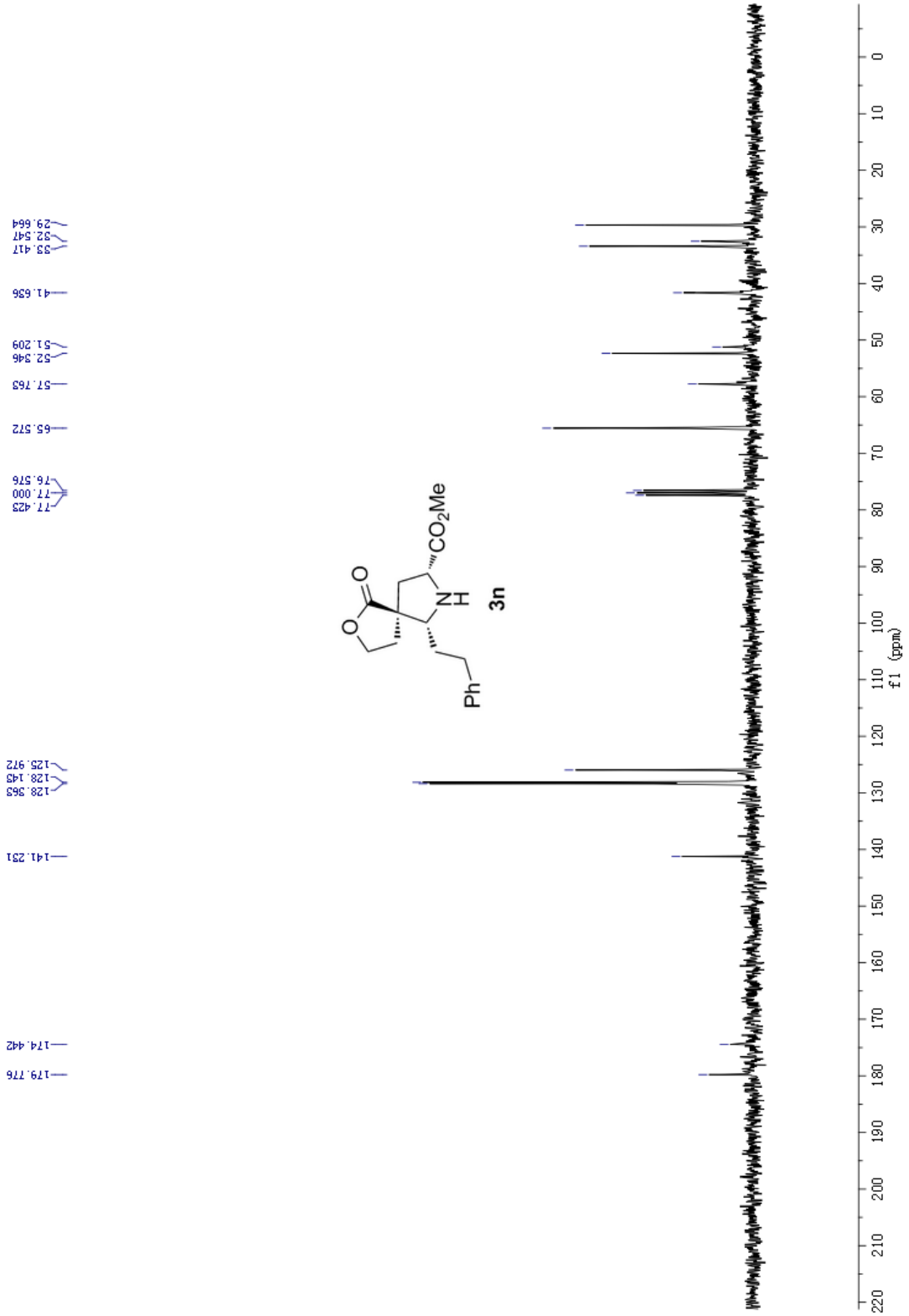

0.000
1.800
1.822
1.845
1.870
2.069
2.089
2.112
2.133
2.156
2.217
2.240
2.261
2.283
2.854
2.884
2.898
2.927
2.995
5.020
5.048
3.819
3.850
3.869
3.896
4.205
4.230
4.256
5.492
7.263
7.478
7.500
7.523
7.795
7.823
7.843
7.869
8.033
8.051
8.071
1.21
1.27
1.50
1.14
1.00
3.83
1.08
1.00
2.97
1.93
1.95
CO2Me
O
O
N
H
3l
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

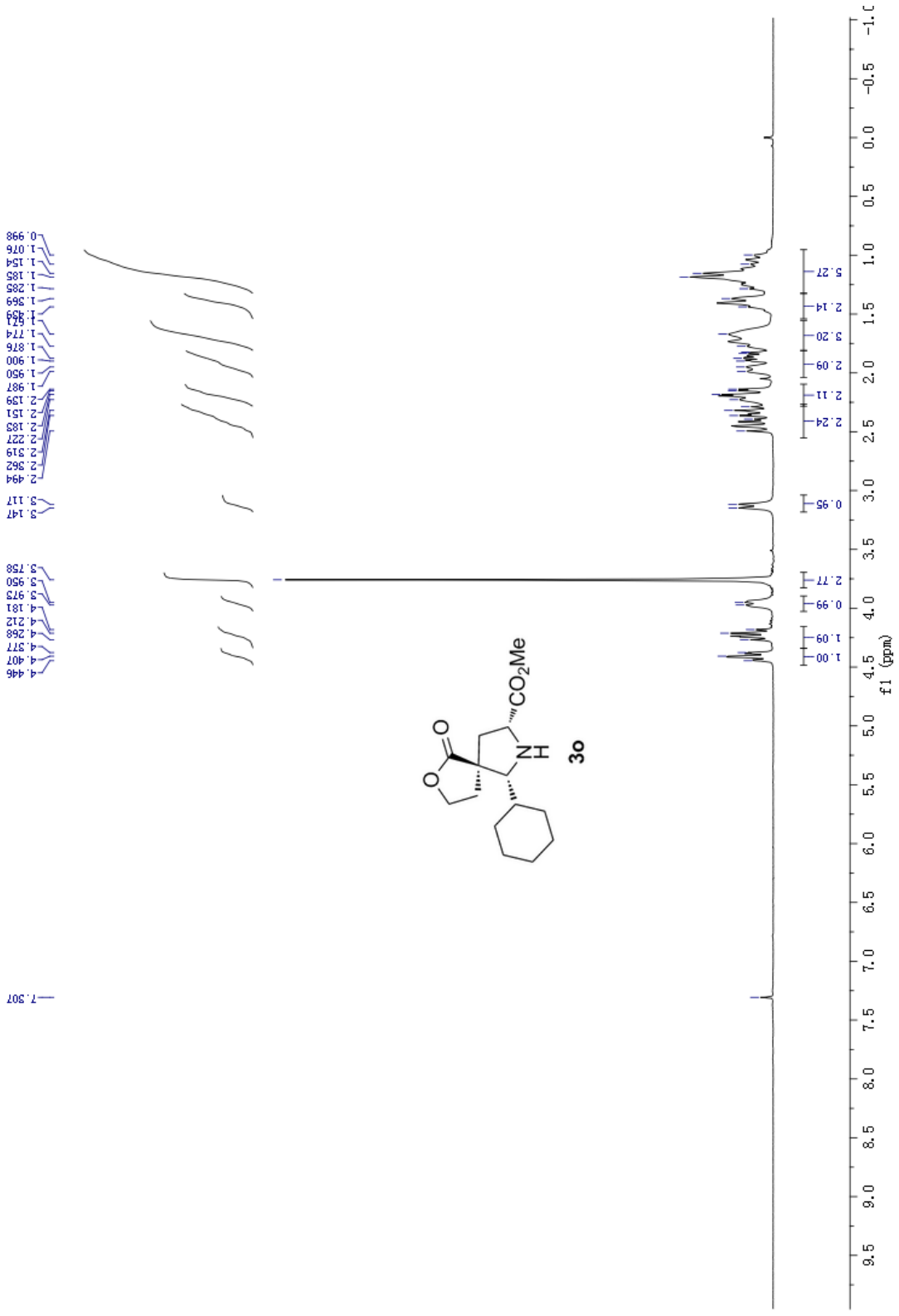

32.604
41.359
51.534
52.262
57.285
62.840
66.026
76.577
77.000
77.424
122.801
125.149
125.261
125.881
126.689
128.571
128.791
131.924
133.581
134.217
173.737
180.811
CO2Me
N
H
O
O
3l
f1 (ppm)

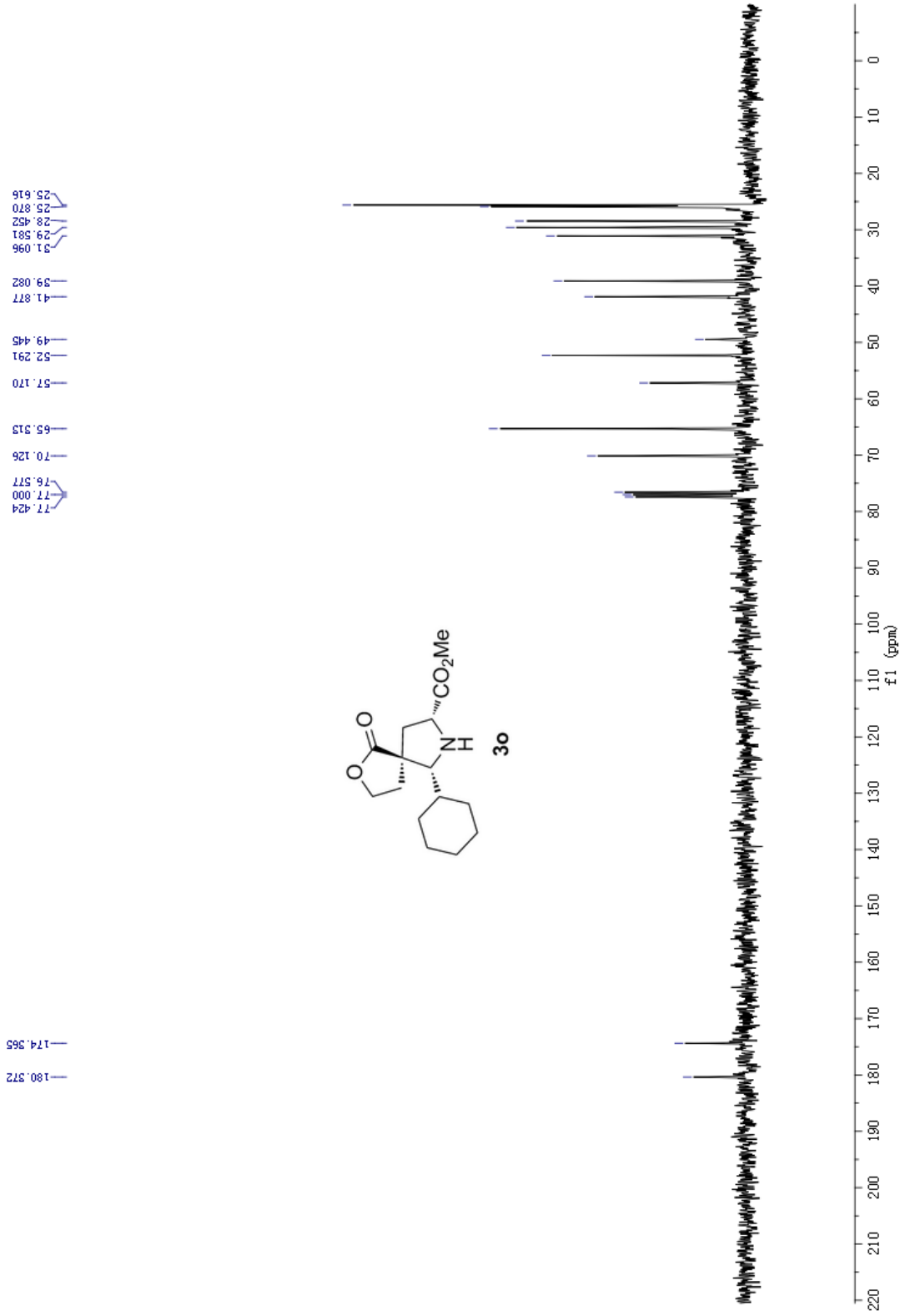

CO2Me
H
N
O
O
3m
f1 (ppm)

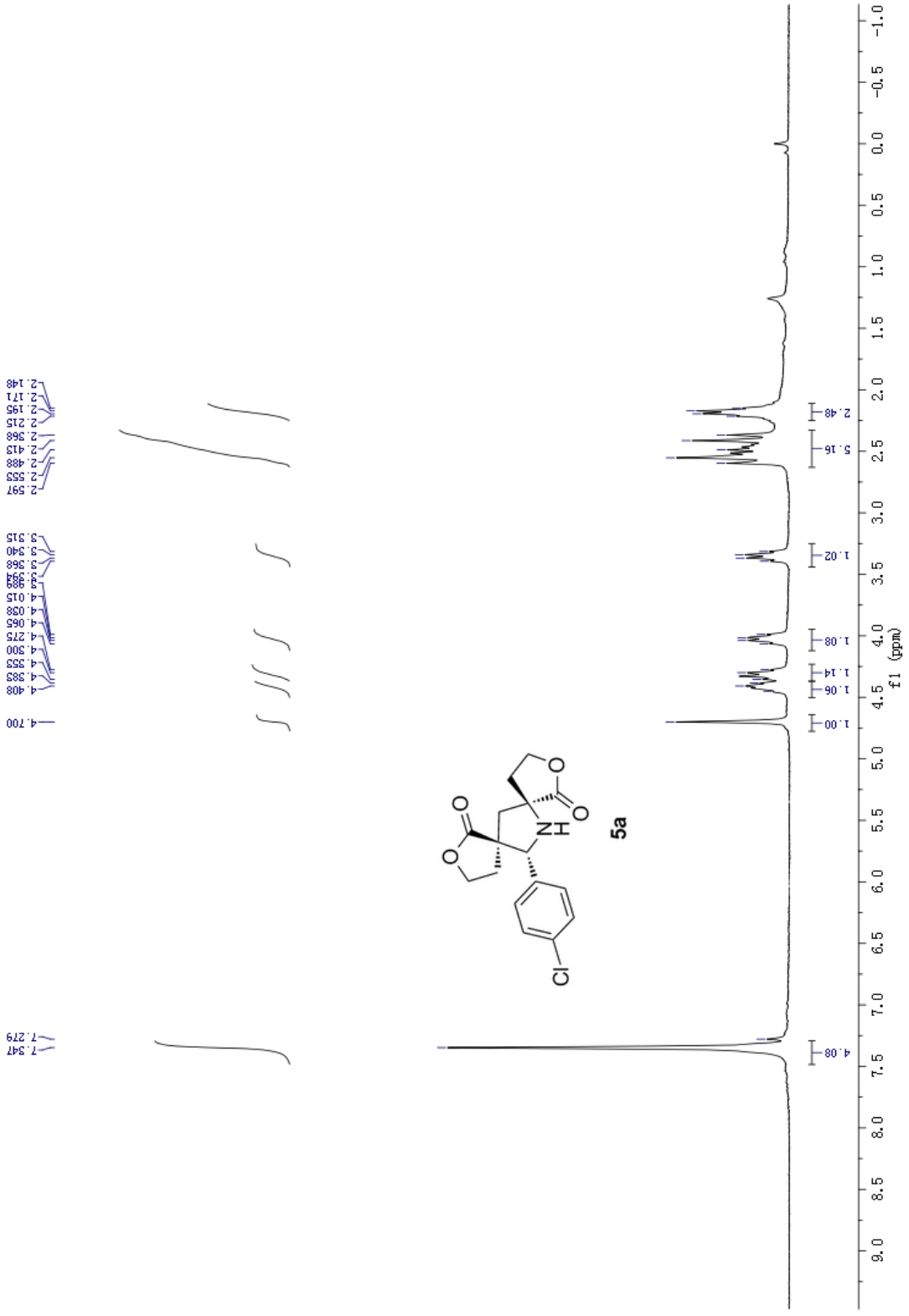

30.993
40.823
52.300
52.592
57.079
65.840
68.611
76.576
77.000
77.424
124.784
125.611
126.106
126.308
127.589
127.955
128.222
133.013
133.113
134.995
174.119
180.073
CO2Me
N
H
O
O
3m
f1 (ppm)

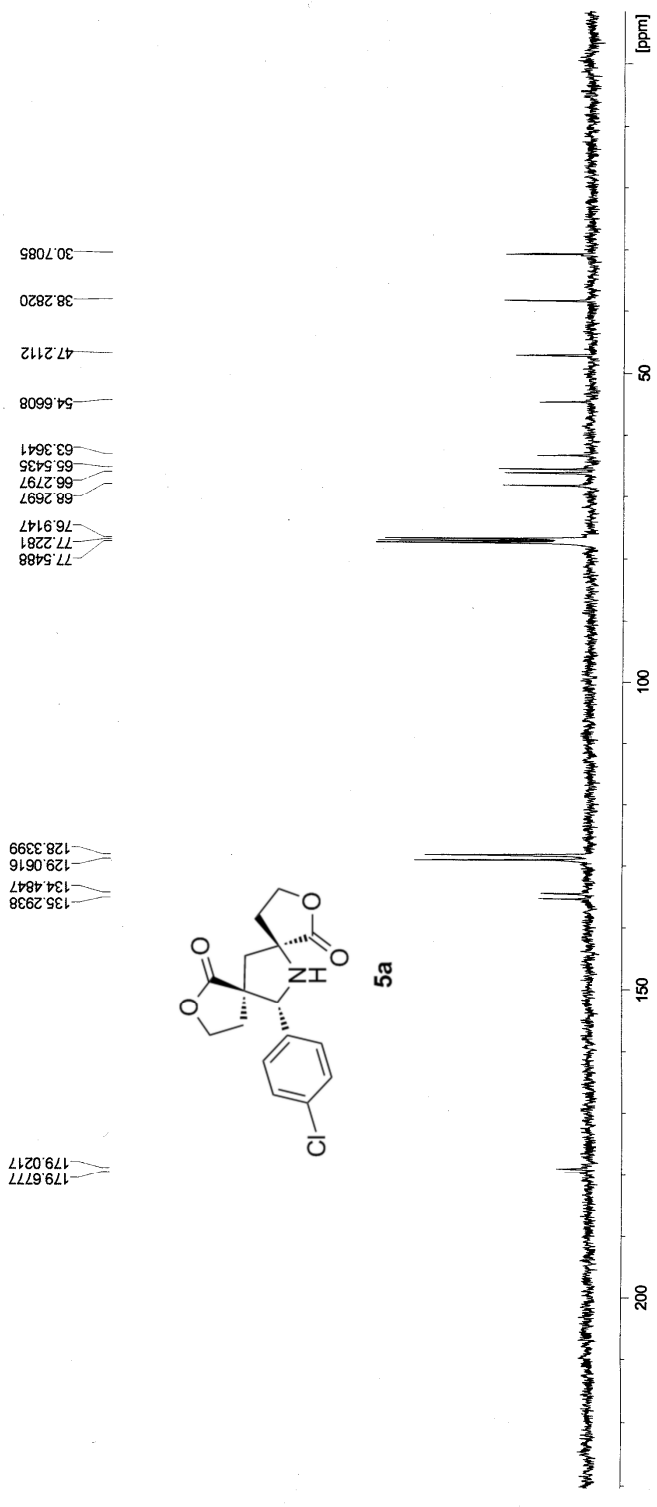

0.000
1.728
1.752
1.805
1.873
1.895
1.962
2.060
2.075
2.105
2.120
2.321
2.346
2.415
2.585
2.618
2.686
2.809
2.836
2.880
3.536
3.359
3.382
5.769
5.979
5.994
4.012
4.028
4.227
4.250
4.274
7.176
7.201
7.218
7.267
7.291
7.316
1.15
0.66
0.72
0.84
1.12
0.60
0.52
1.45
0.52
1.00
1.31
1.05
CO2Me
N
H
O
O
Ph
3n
f1 (ppm)

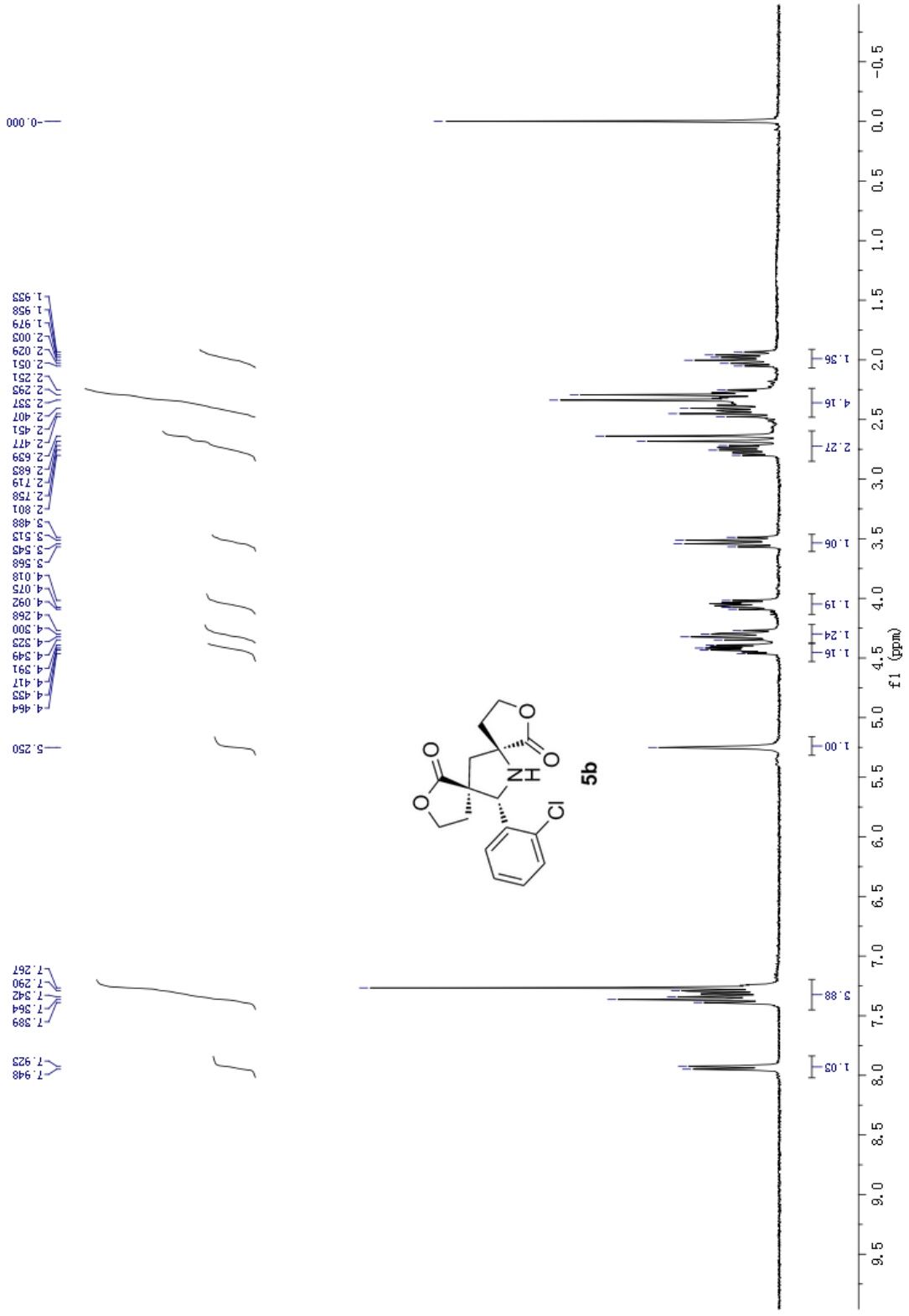

29.664
32.547
33.417
41.636
51.209
52.346
57.763
65.572
76.576
77.000
77.423
125.972
128.143
128.363
141.231
174.442
179.776
CO2Me
O
O
N
H
Ph
3n
f1 (ppm)

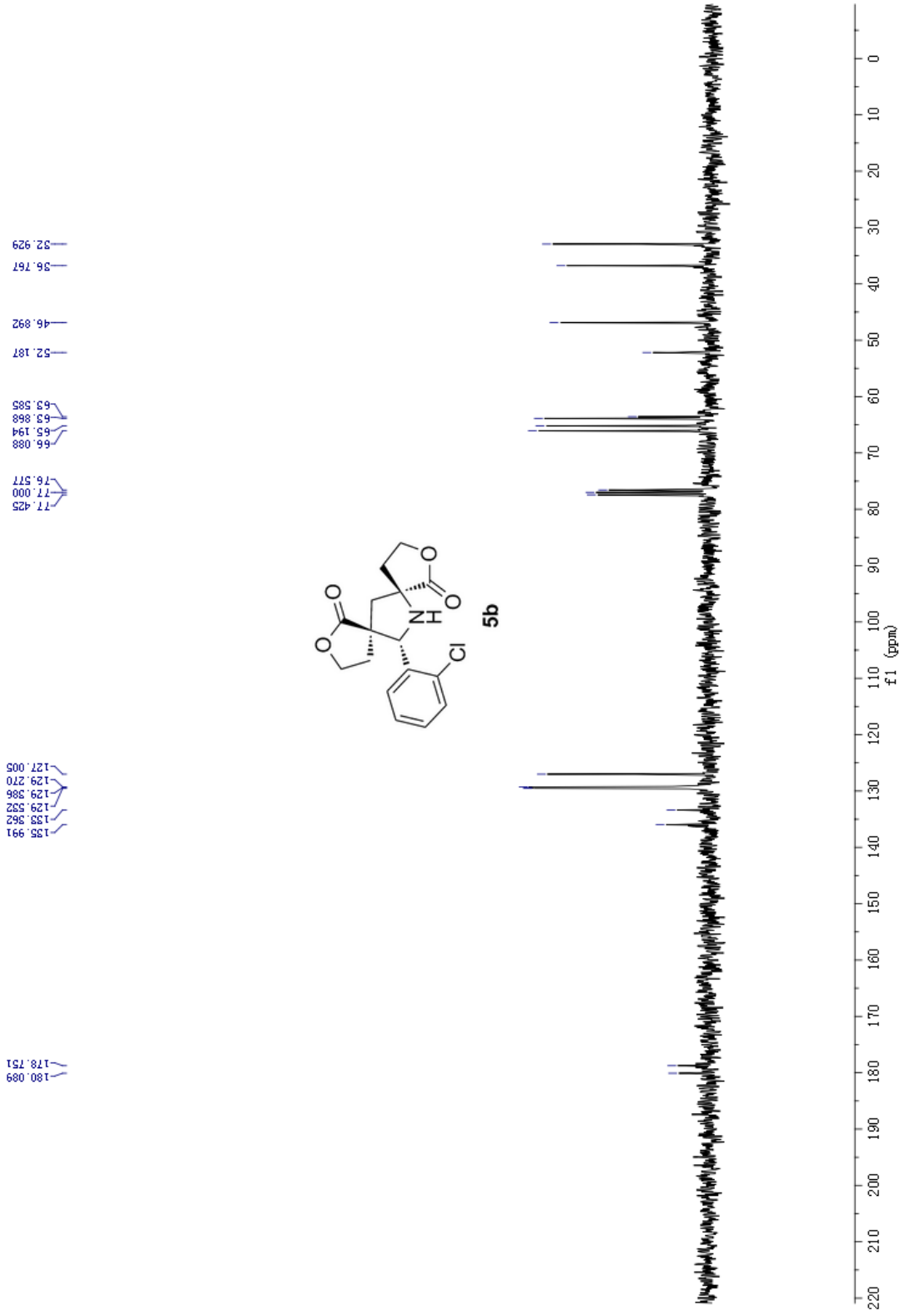

CO2Me
H
N
O
O
3o
f1 (ppm)

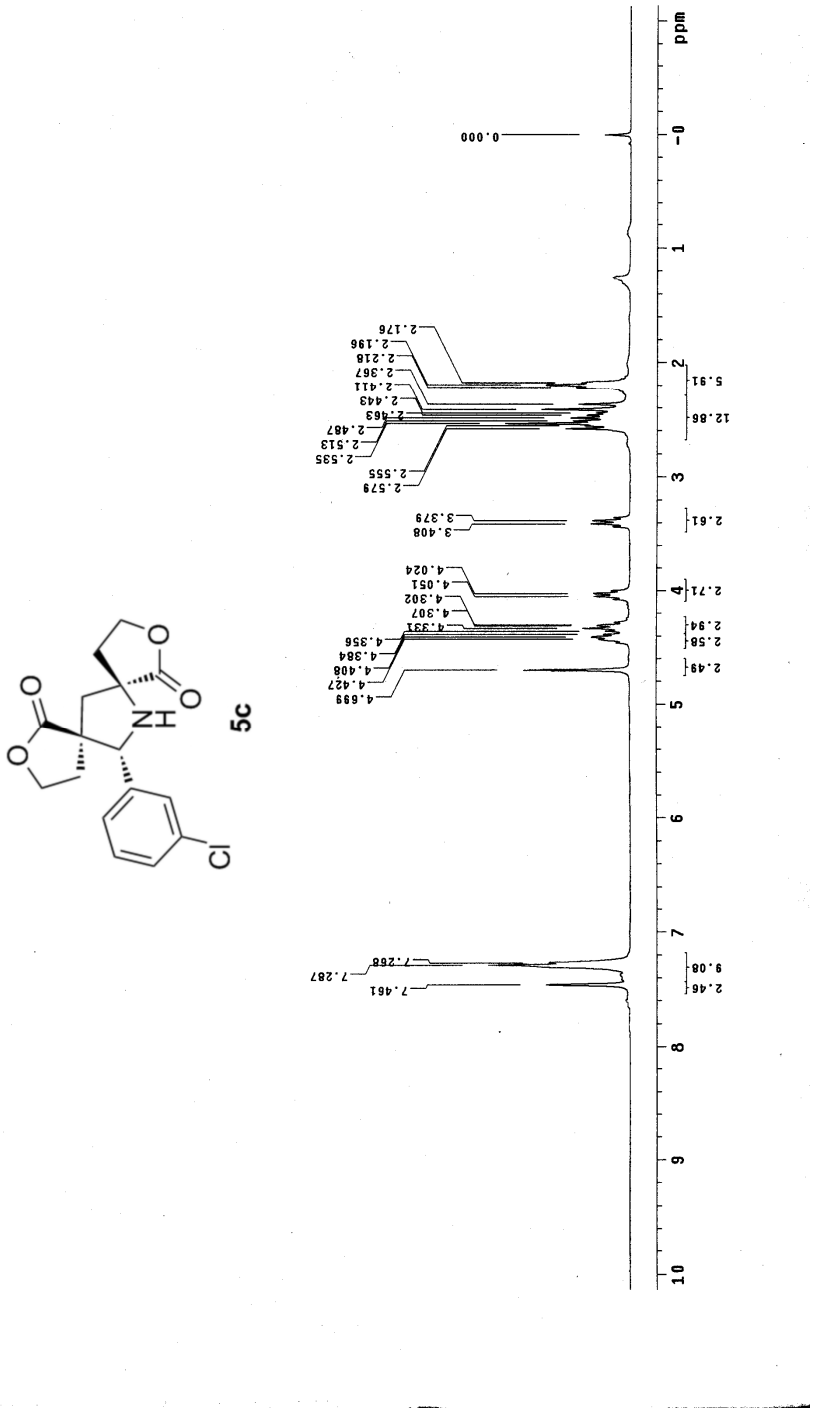

25.616
25.870
28.452
29.581
31.096
39.082
41.877
49.445
52.291
57.170
65.313
70.126
76.577
77.000
77.424
174.365
180.372
CO2Me
O
O
N
H
3o
f1 (ppm)

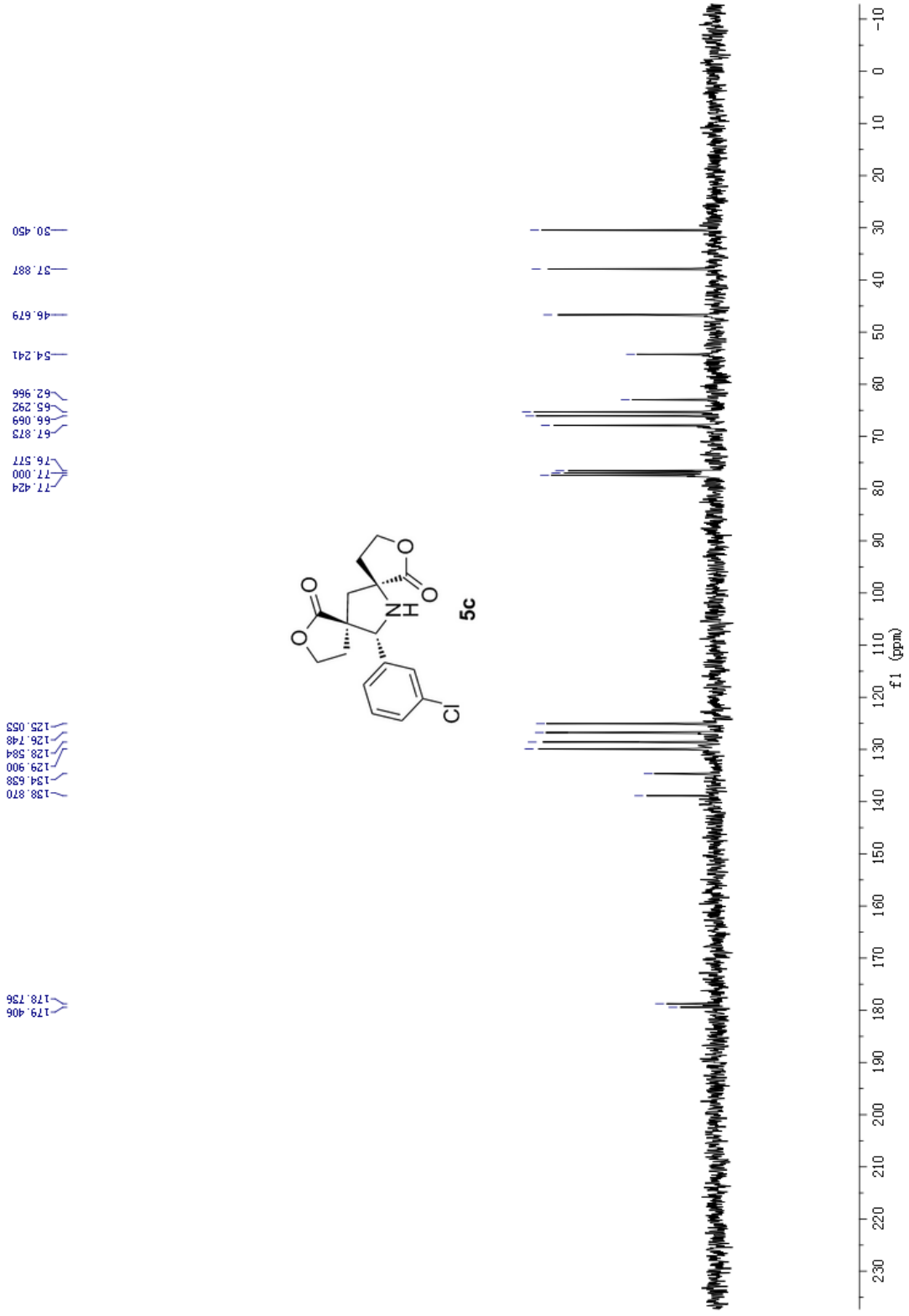

5a
Cl
NH
O
f1 (ppm)

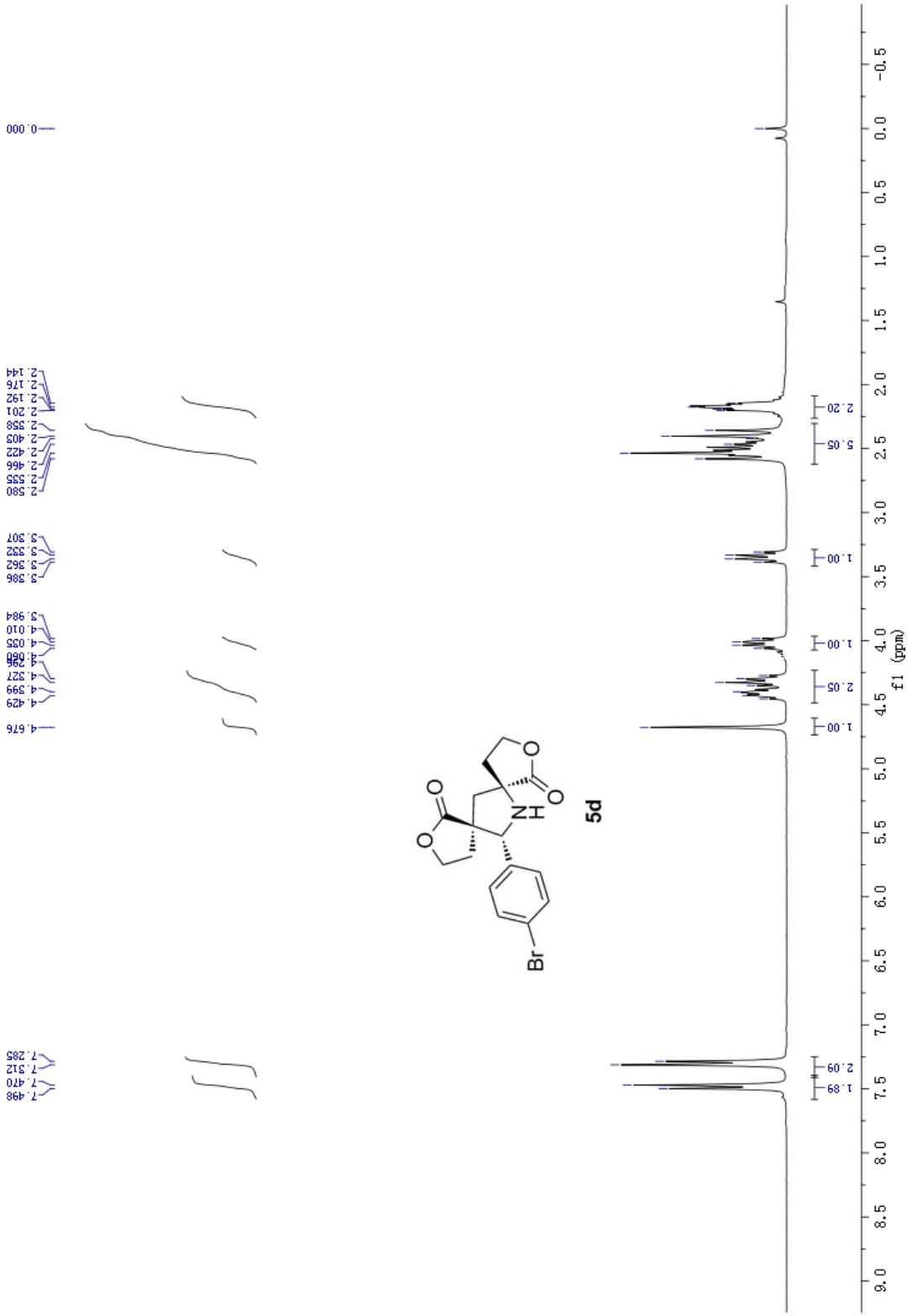

30.7085
38.2820
47.2112
54.6608
63.3641
65.5435
66.2797
68.2697
76.9147
77.2281
77.5488
128.3399
129.0616
134.4847
135.2938
179.6777
179.0217
NH
O
O
O
O
Cl
5a
[ppm]
50
100
150
200

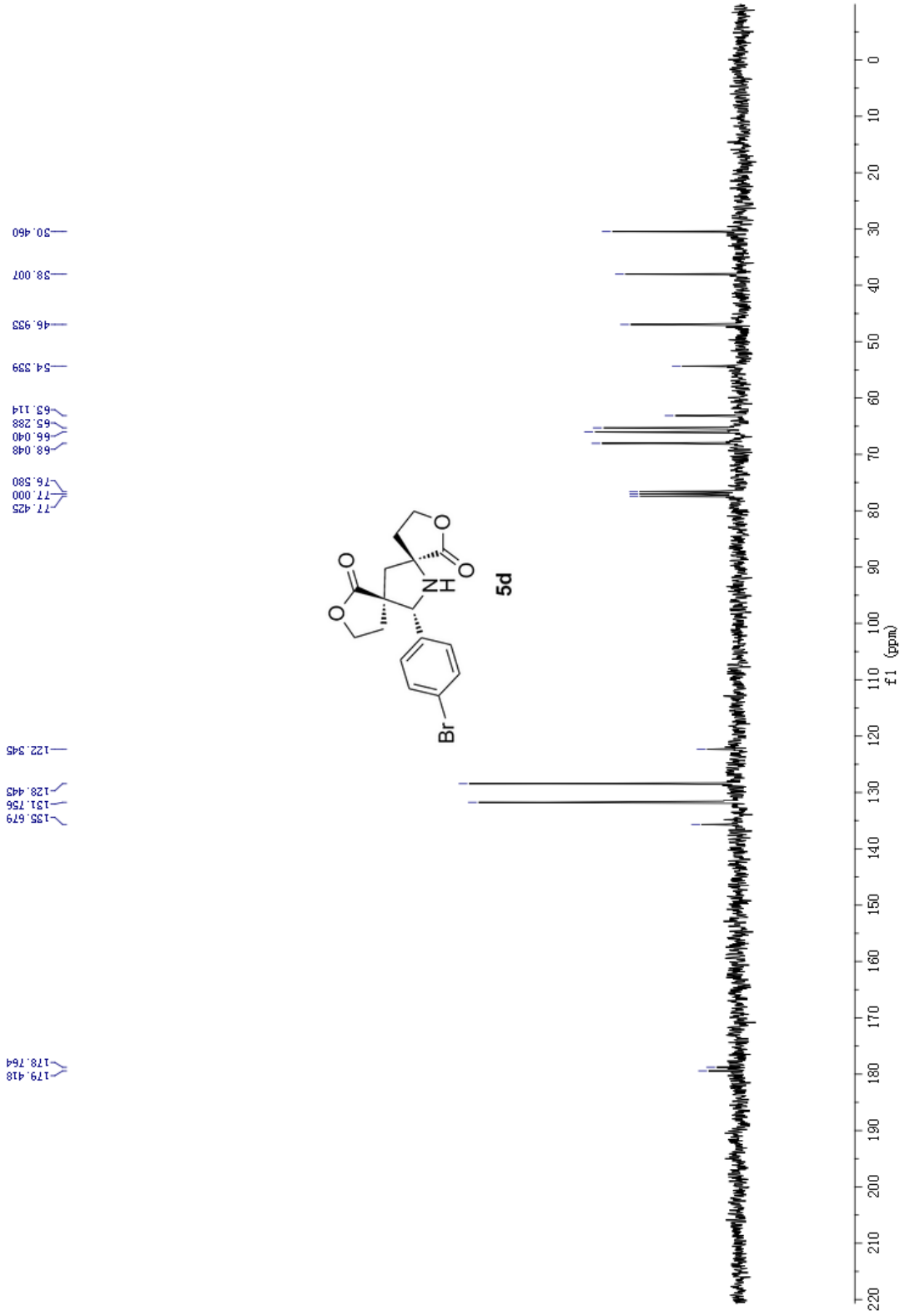

5b
f1 (ppm)

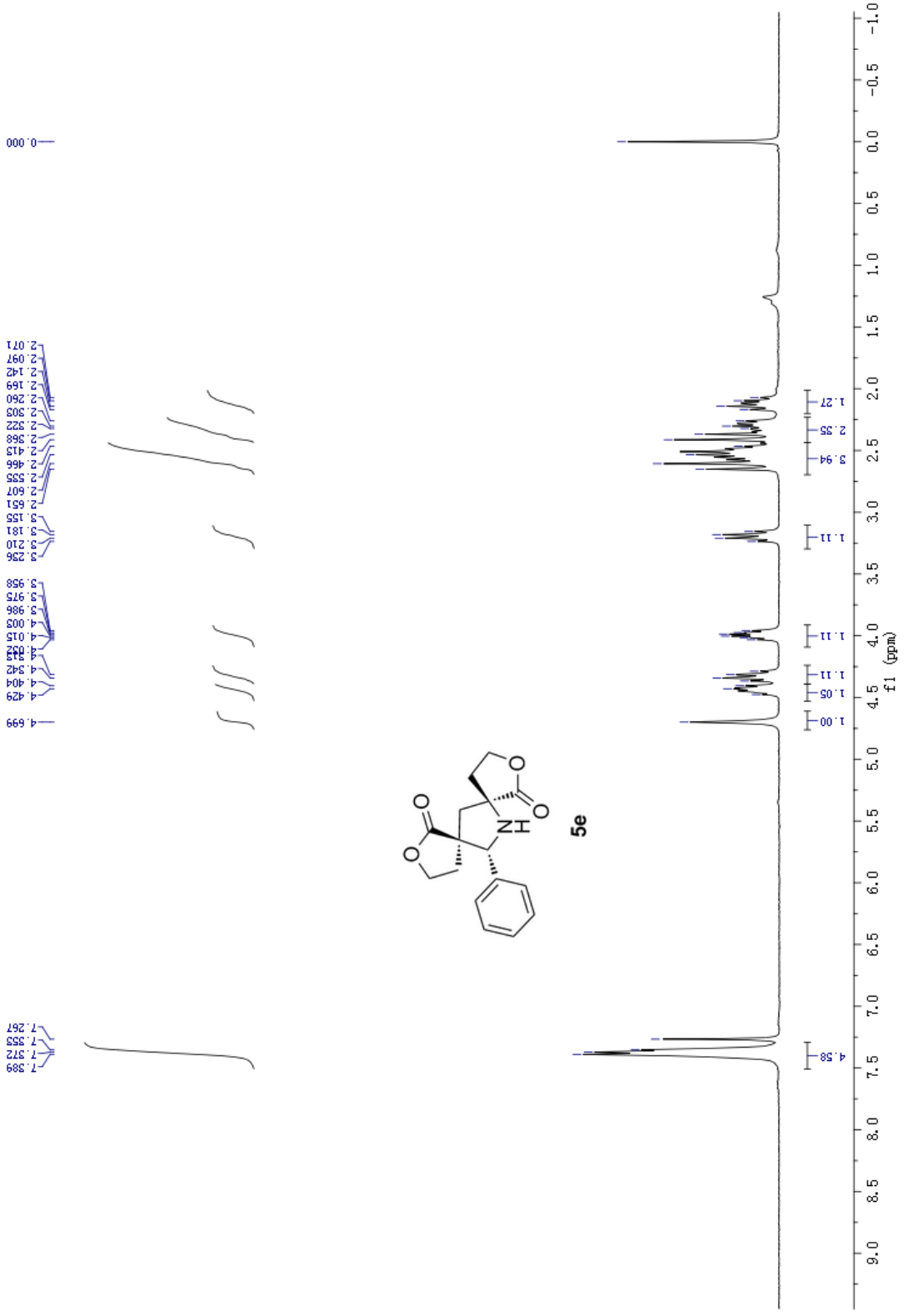

32.929
36.767
46.892
52.187
63.585
63.868
65.194
66.088
76.577
77.000
77.425
127.005
129.270
129.386
129.552
133.362
135.991
178.751
180.089
5b
Cl
O
O
O
O
NH
f1 (ppm)

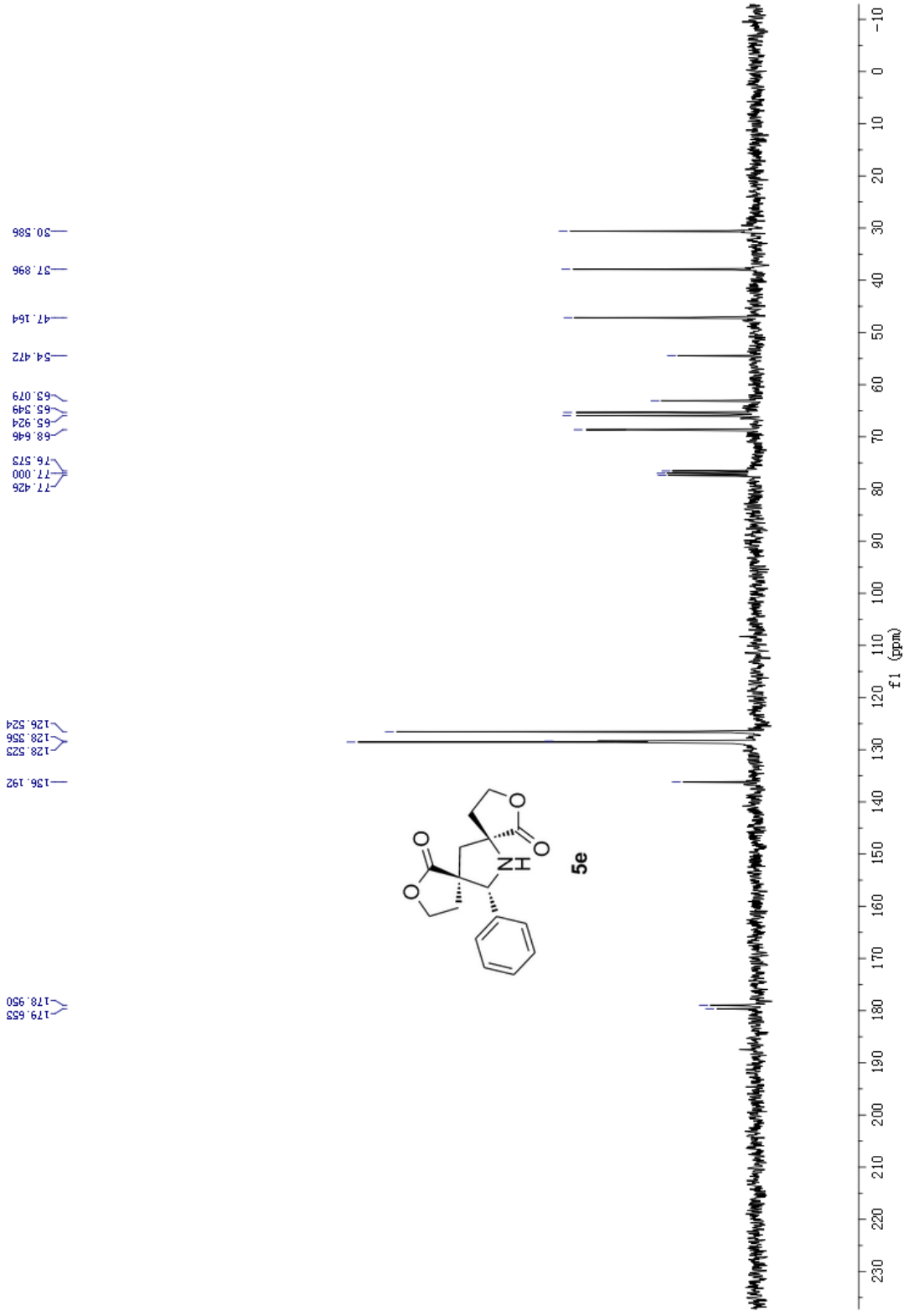

5c
Cl
NH
O
O
O
O
ppm
0.000
2.176
2.196
2.218
2.367
2.411
2.443
2.463
2.487
2.513
2.535
2.555
2.579
3.379
3.408
4.024
4.051
4.302
4.307
4.331
4.356
4.384
4.408
4.427
4.699
7.261
7.268
7.287
7.461
5.91
12.86
2.61
2.71
2.94
2.58
2.49
9.08
2.46

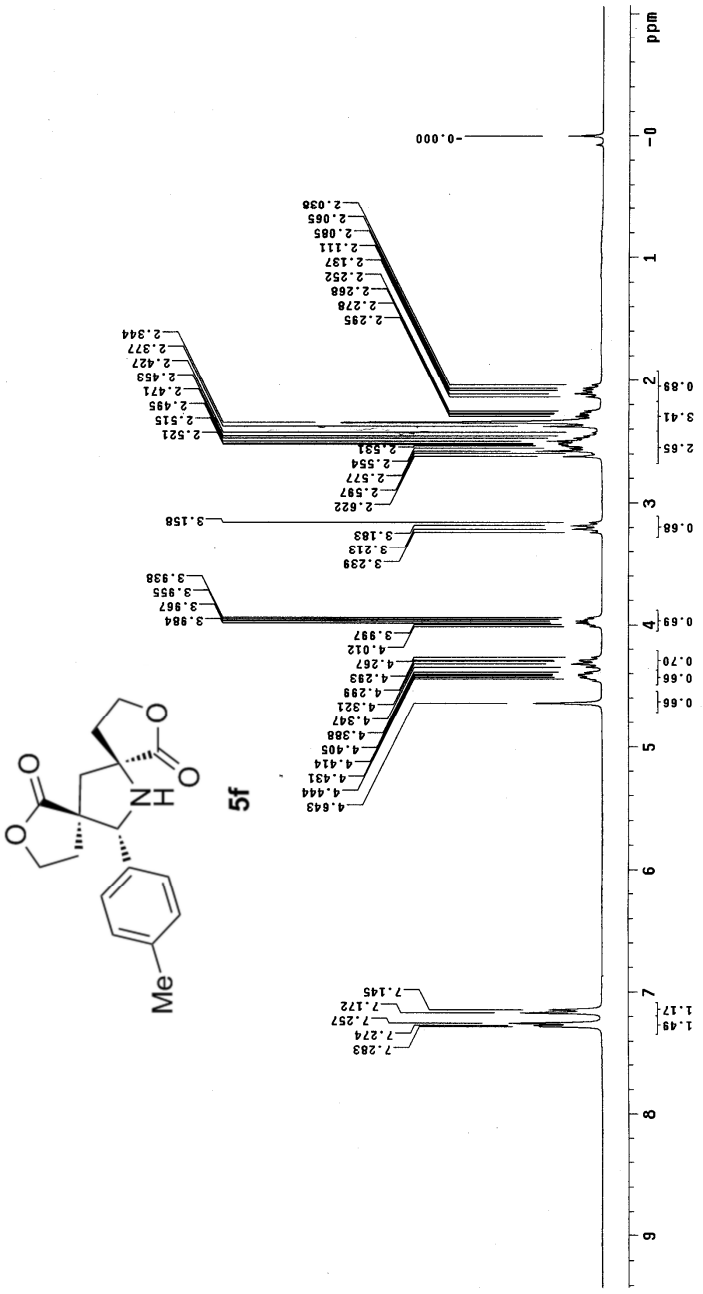

30.450
37.887
46.679
54.241
62.966
65.292
66.069
67.873
76.577
77.000
77.424
125.053
126.748
128.584
129.900
134.638
138.870
178.736
179.406
5c
Cl
N
H
O
f1 (ppm)

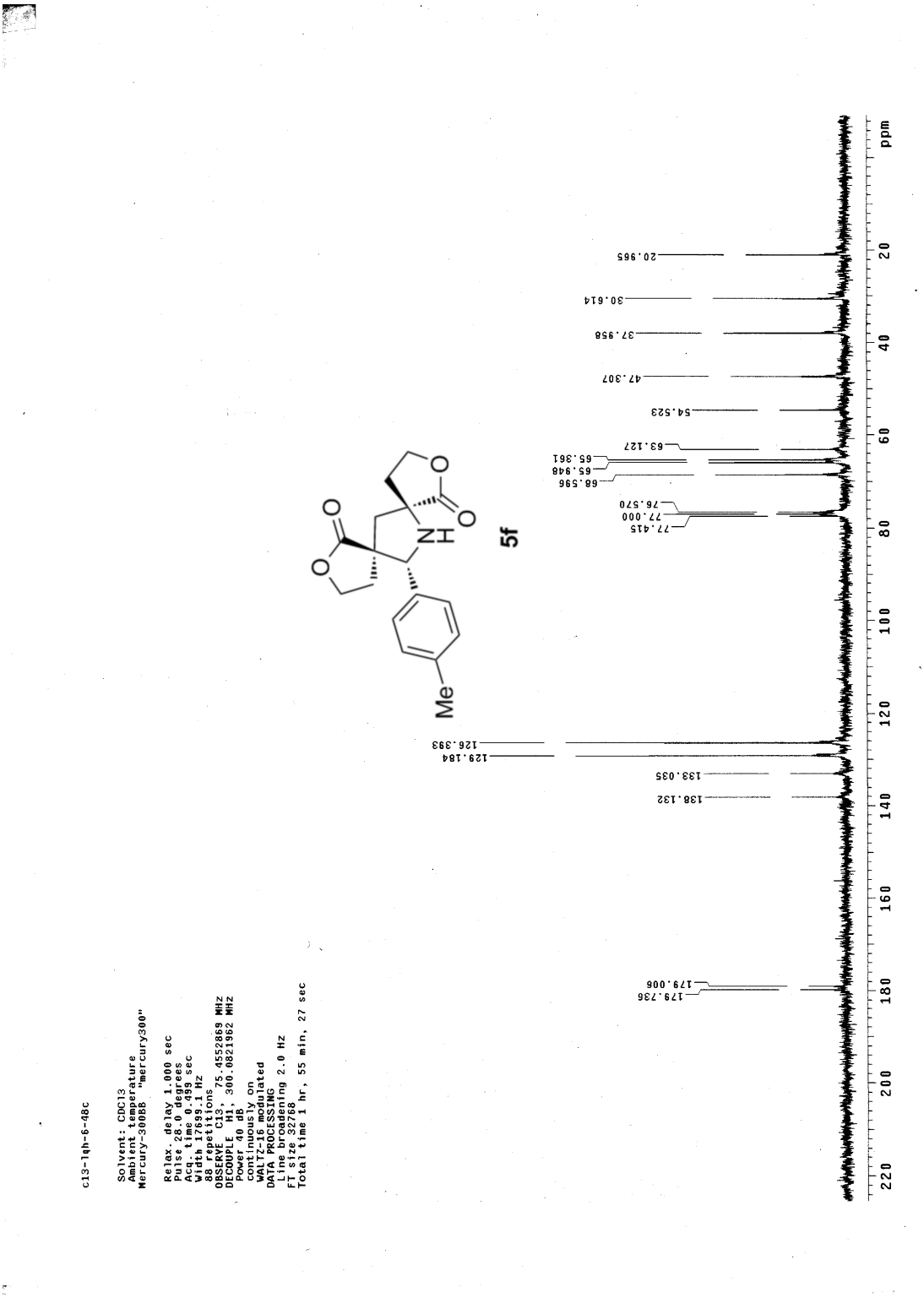

0.000
2.144
2.176
2.192
2.201
2.358
2.403
2.422
2.466
2.535
2.580
3.307
3.332
3.362
3.386
3.984
4.010
4.035
4.060
4.296
4.327
4.399
4.429
4.676
7.285
7.312
7.470
7.498
2.09
1.89
1.00
2.05
1.00
1.00
5.05
2.20
f1 (ppm)
5d
Br
O
O
O
O
N
H

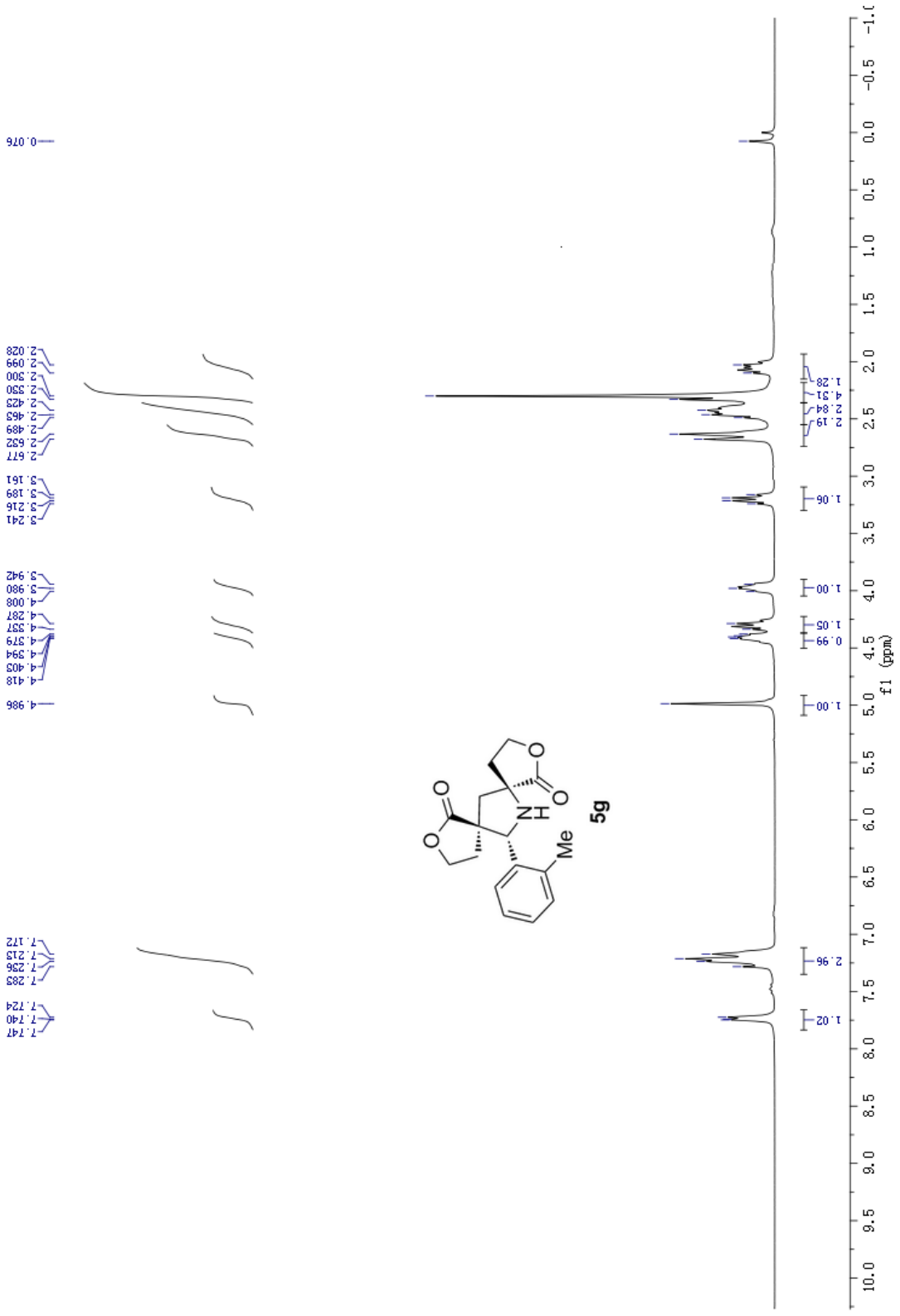

30.460
38.007
46.933
54.339
63.114
65.288
66.040
68.048
76.580
77.000
77.425
122.945
128.443
131.756
135.679
178.764
179.418
5d
Br
f1 (ppm)

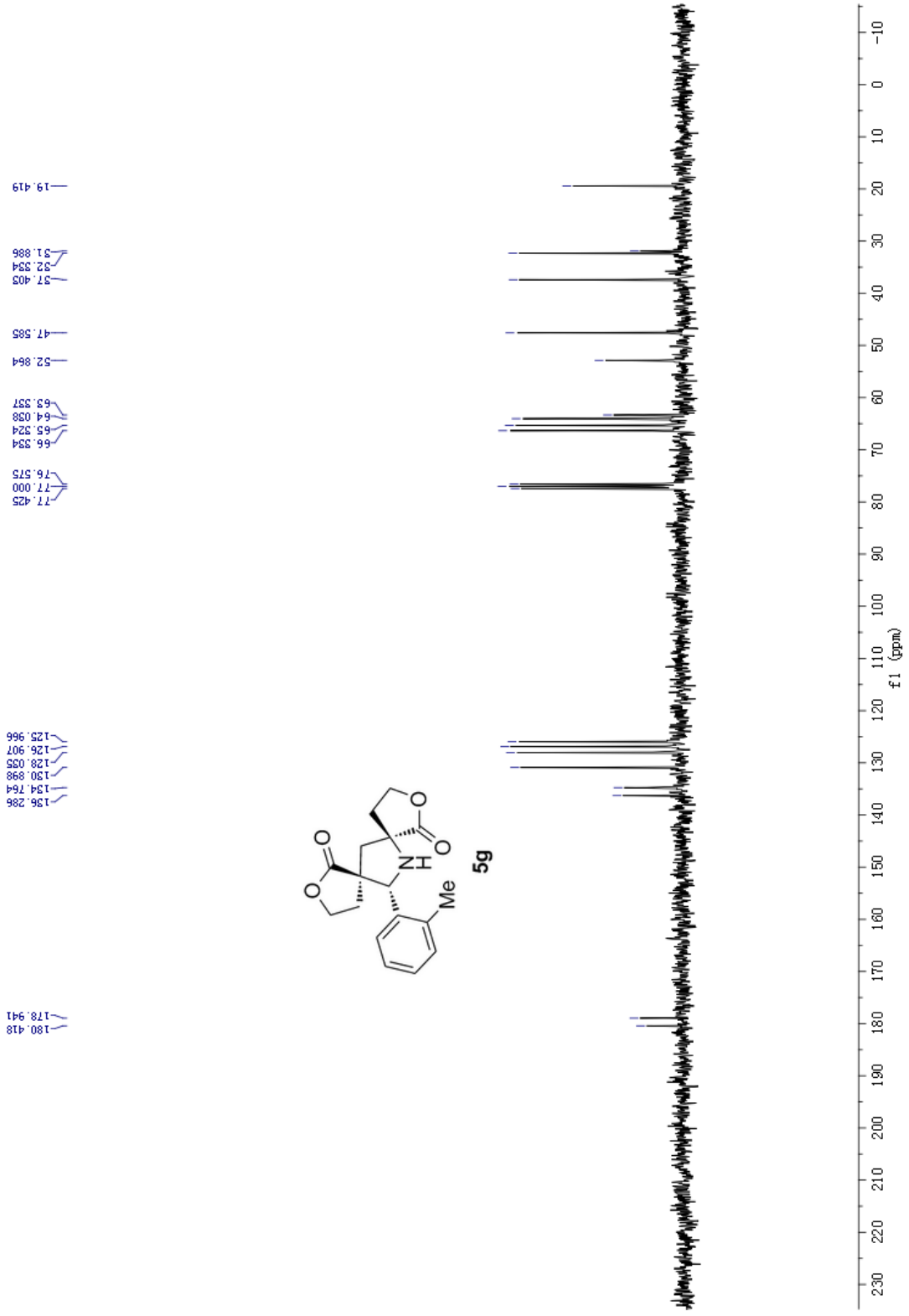

5e
f1 (ppm)

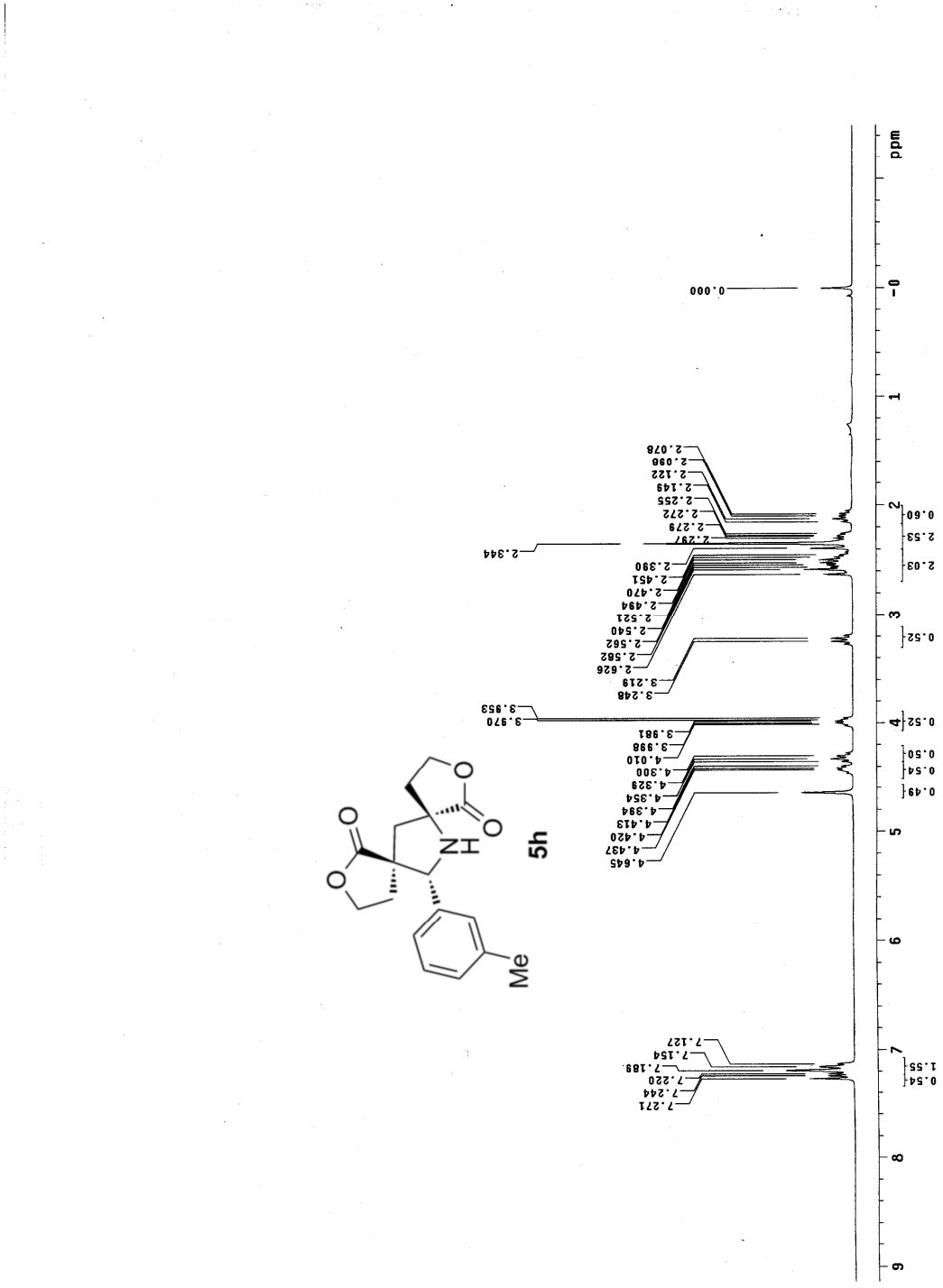

30.586
37.896
47.164
54.472
63.079
65.349
65.924
68.646
76.573
77.000
77.426
128.524
128.356
128.523
136.192
178.950
179.653
5e
f1 (ppm)

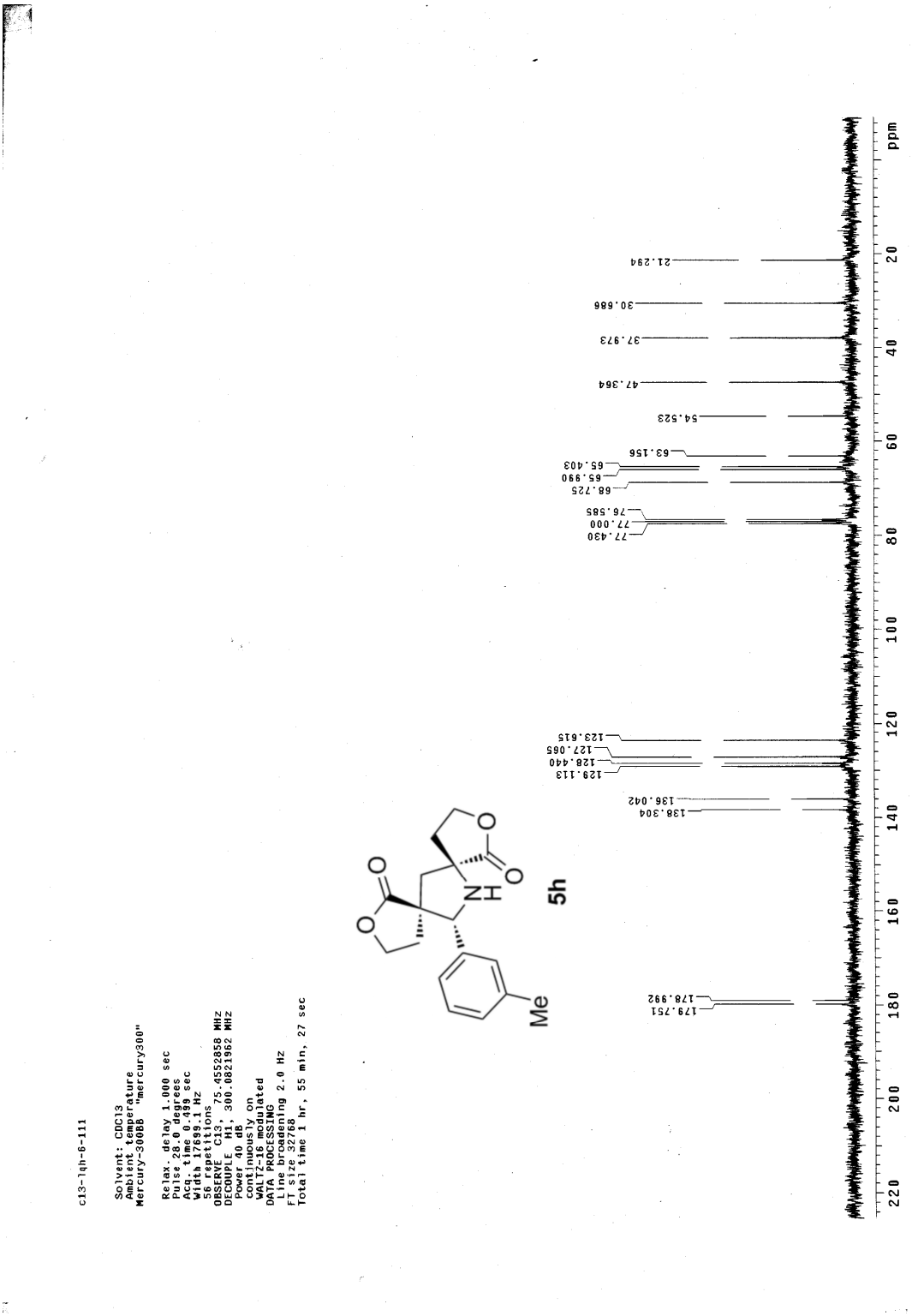

ppm
0.000
2.038
2.065
2.085
2.111
2.137
2.252
2.268
2.278
2.295
2.344
2.377
2.427
2.453
2.471
2.495
2.515
2.521
2.531
2.554
2.577
2.597
2.622
3.158
3.183
3.213
3.239
3.938
3.955
3.967
3.984
3.997
4.012
4.267
4.283
4.299
4.321
4.347
4.388
4.405
4.414
4.431
4.444
4.643
7.145
7.172
7.257
7.274
7.283
0.89
3.41
2.65
0.68
0.69
0.70
0.66
0.66
1.49
1.17
5f
Me

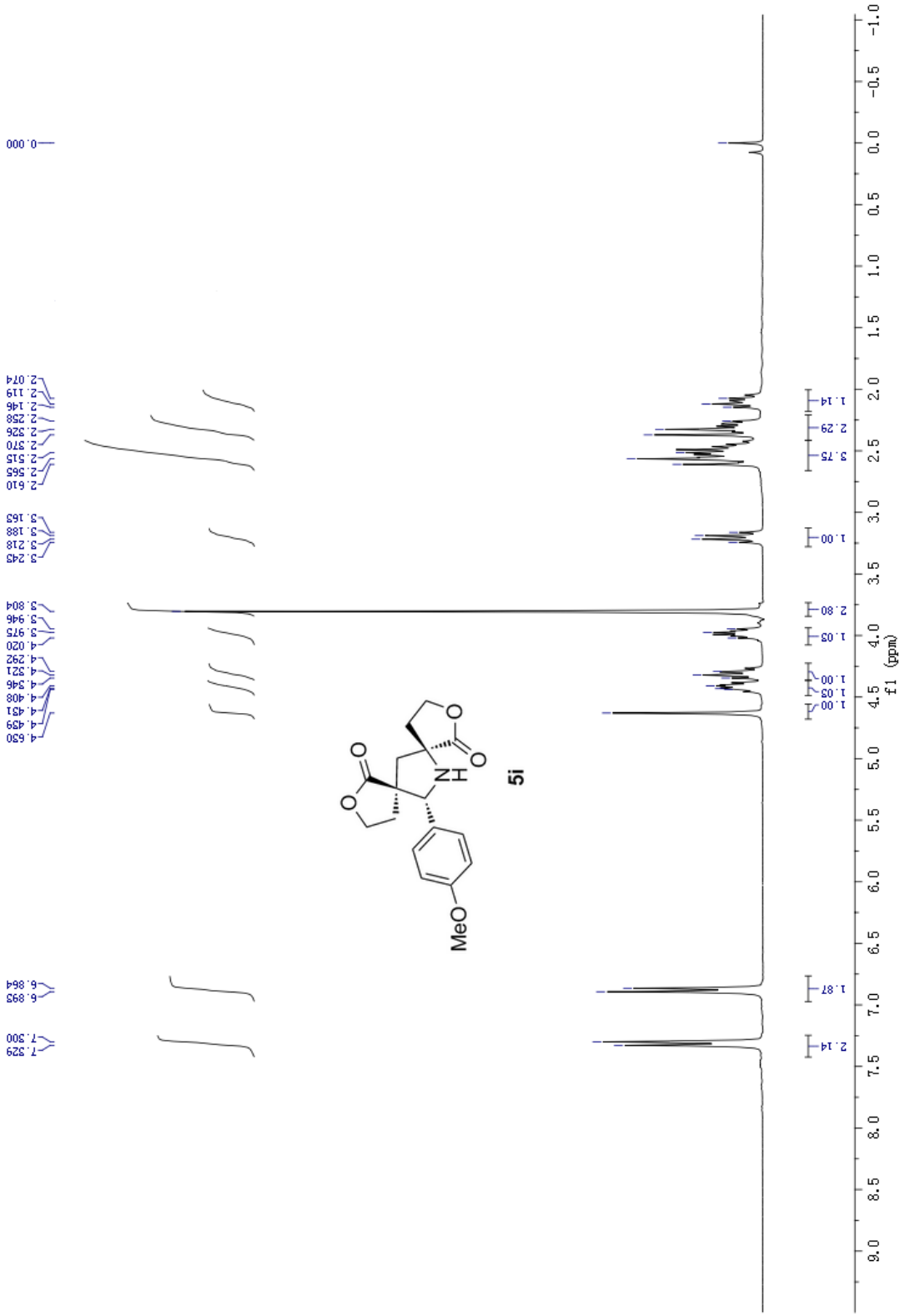

c13-lqh-6-48c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
88 repetitions
OBSERVE C13, 75.4552869 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
5f
Me
N
H
O
O
O
O
179.736
179.006
138.132
133.035
129.184
126.393
77.415
77.000
76.570
68.596
65.948
65.361
63.127
54.523
47.307
37.958
30.614
20.965
ppm

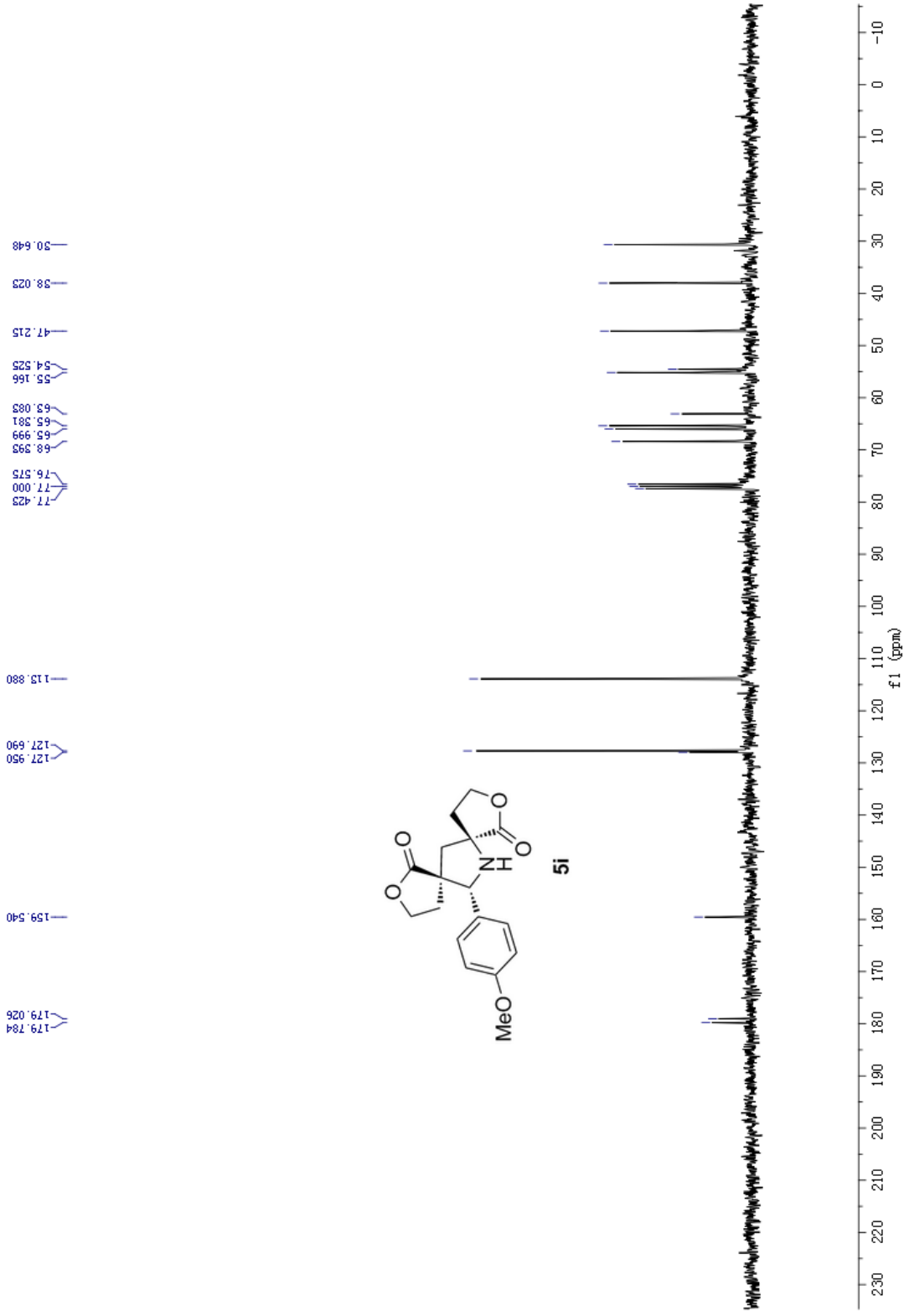

0.076
2.028
2.099
2.300
2.330
2.423
2.463
2.489
2.632
2.677
3.161
3.189
3.216
3.241
3.942
3.980
4.008
4.287
4.337
4.379
4.394
4.403
4.418
4.986
7.172
7.213
7.236
7.283
7.724
7.740
7.747
1.28
4.31
2.84
2.19
1.06
1.00
1.05
0.99
1.00
2.96
1.02
5g
Me
f1 (ppm)


19.419
31.886
32.334
37.403
47.585
52.864
63.337
64.038
65.324
66.334
76.575
77.000
77.425
125.966
126.907
128.035
130.898
134.764
136.286
178.941
180.418
5g
Me
f1 (ppm)


5h
Me
ppm


c13-lqh-6-111
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
56 repetitions
OBSERVE C13, 75.4552858 MHz
DECOUPLE H1, 300.0821562 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
21.294
30.686
37.973
47.364
54.523
63.156
65.403
65.990
68.725
76.585
77.000
77.430
123.615
127.065
128.440
129.113
136.042
138.304
178.751
178.992
ppm
5h
Me


5i
MeO
f1 (ppm)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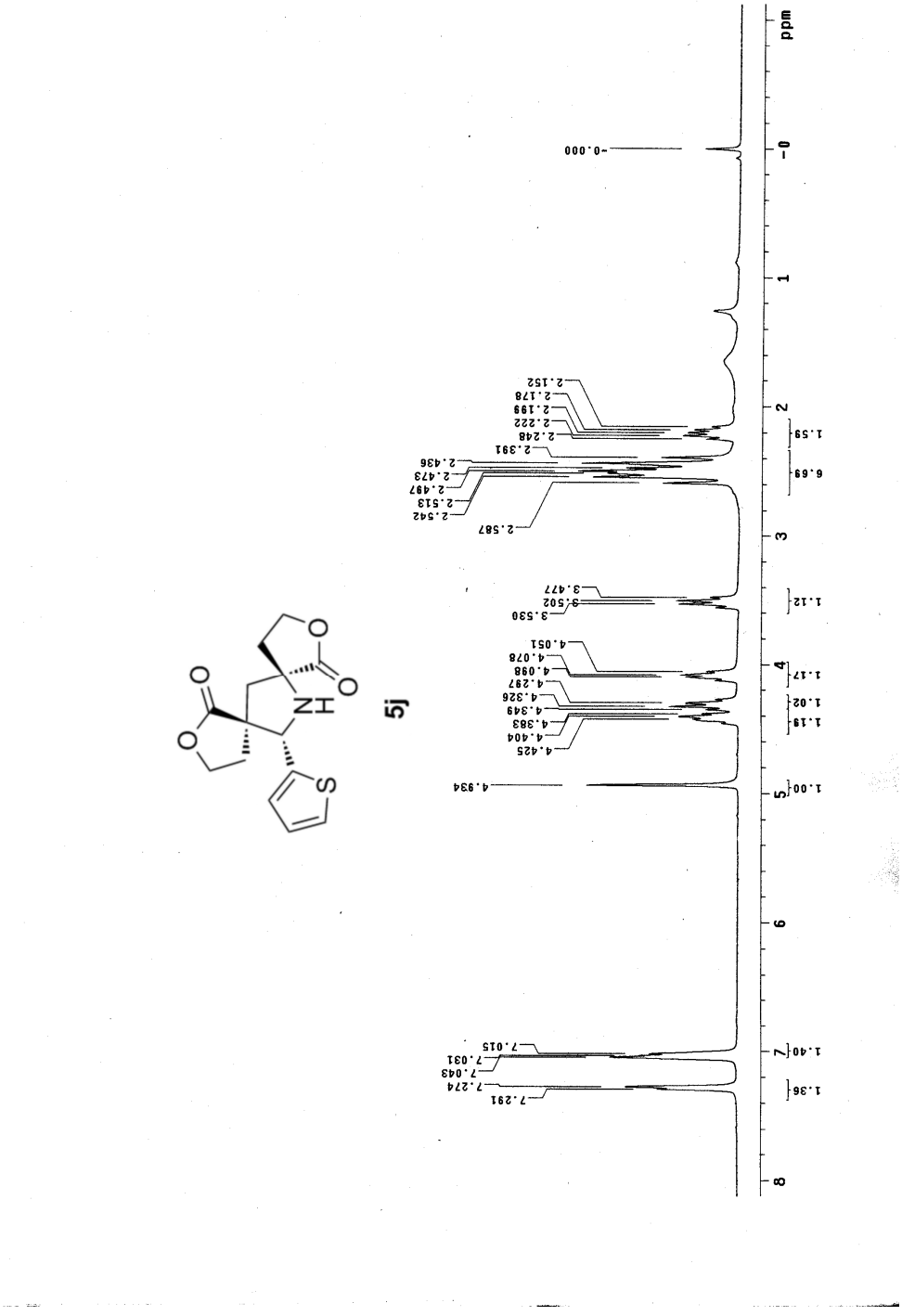
38.023
47.215
54.525
55.166
63.083
65.381
65.999
68.393
76.575
77.000
77.423
113.880
127.690
127.950
159.540
179.026
179.784
f1 (ppm)
MeO
5i


5j
ppm
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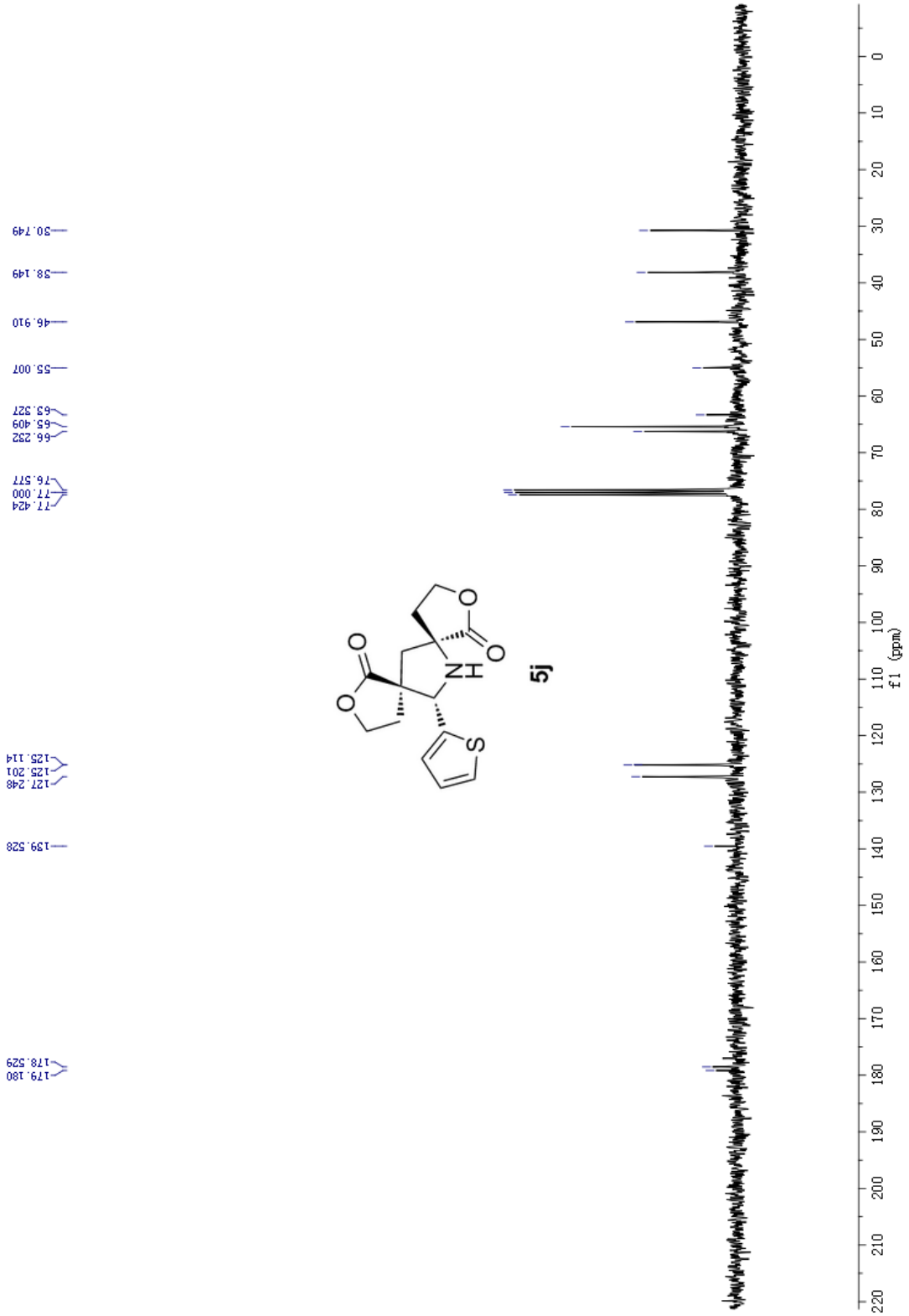
2.178
2.199
2.222
2.248
2.391
2.436
2.473
2.497
2.513
2.542
2.587
3.477
3.502
3.530
4.051
4.078
4.098
4.297
4.326
4.349
4.383
4.404
4.425
4.934
7.015
7.031
7.043
7.274
7.291
1.59
6.69
1.12
1.17
1.02
1.19
1.00
1.40
1.36


30.749
38.149
46.910
55.007
63.327
65.409
66.232
76.577
77.000
77.424
125.114
125.201
127.248
139.528
178.529
179.180
5j
f1 (ppm)

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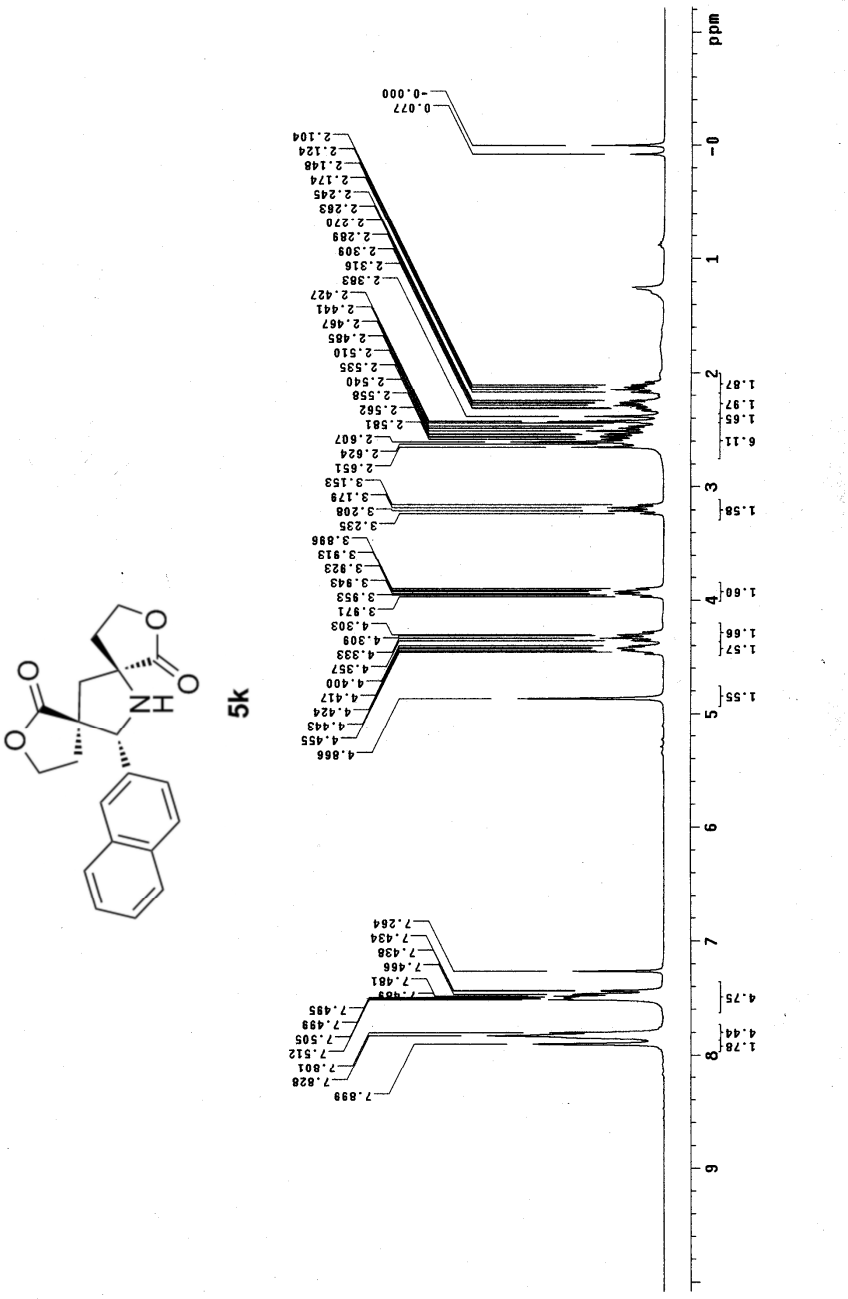
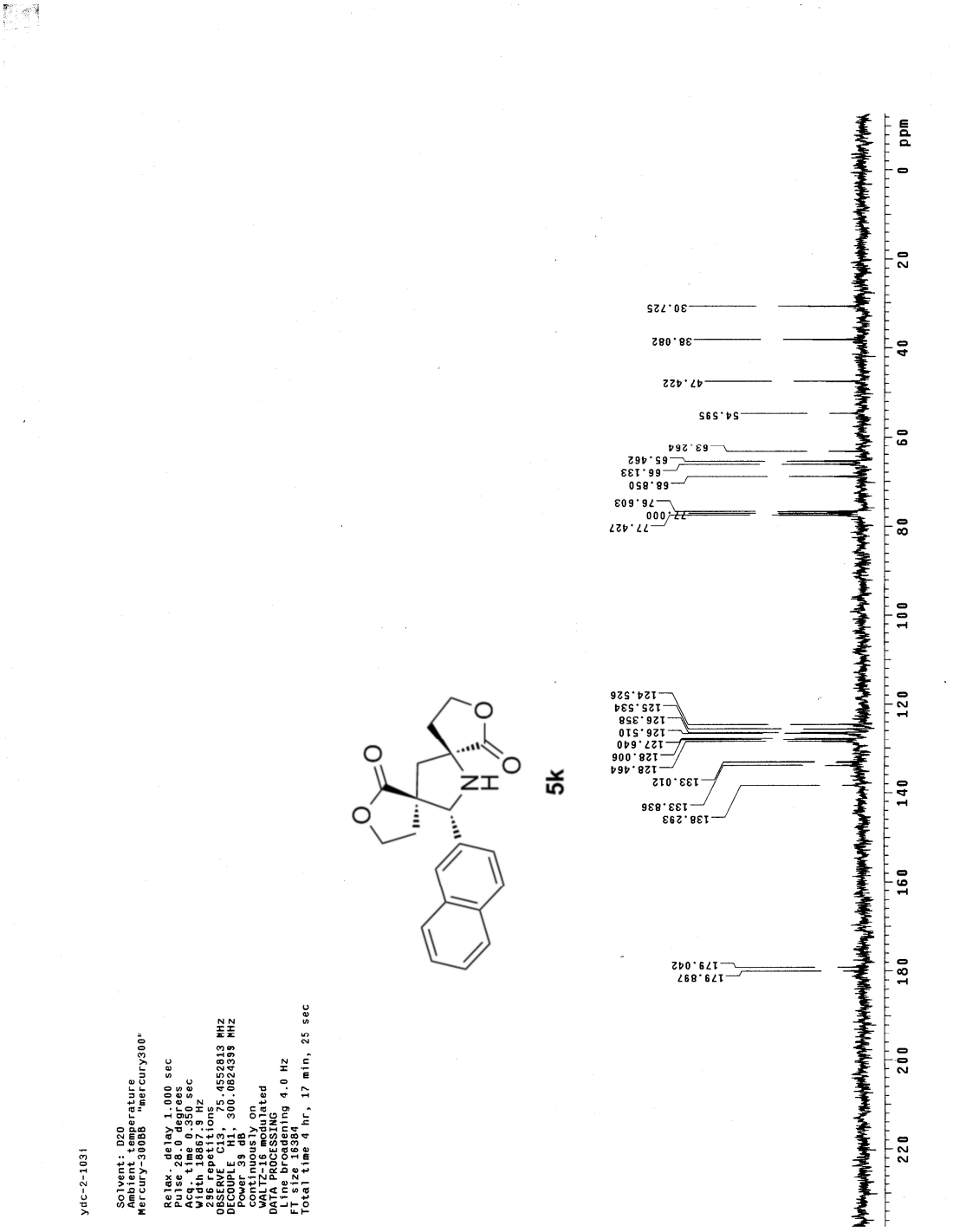
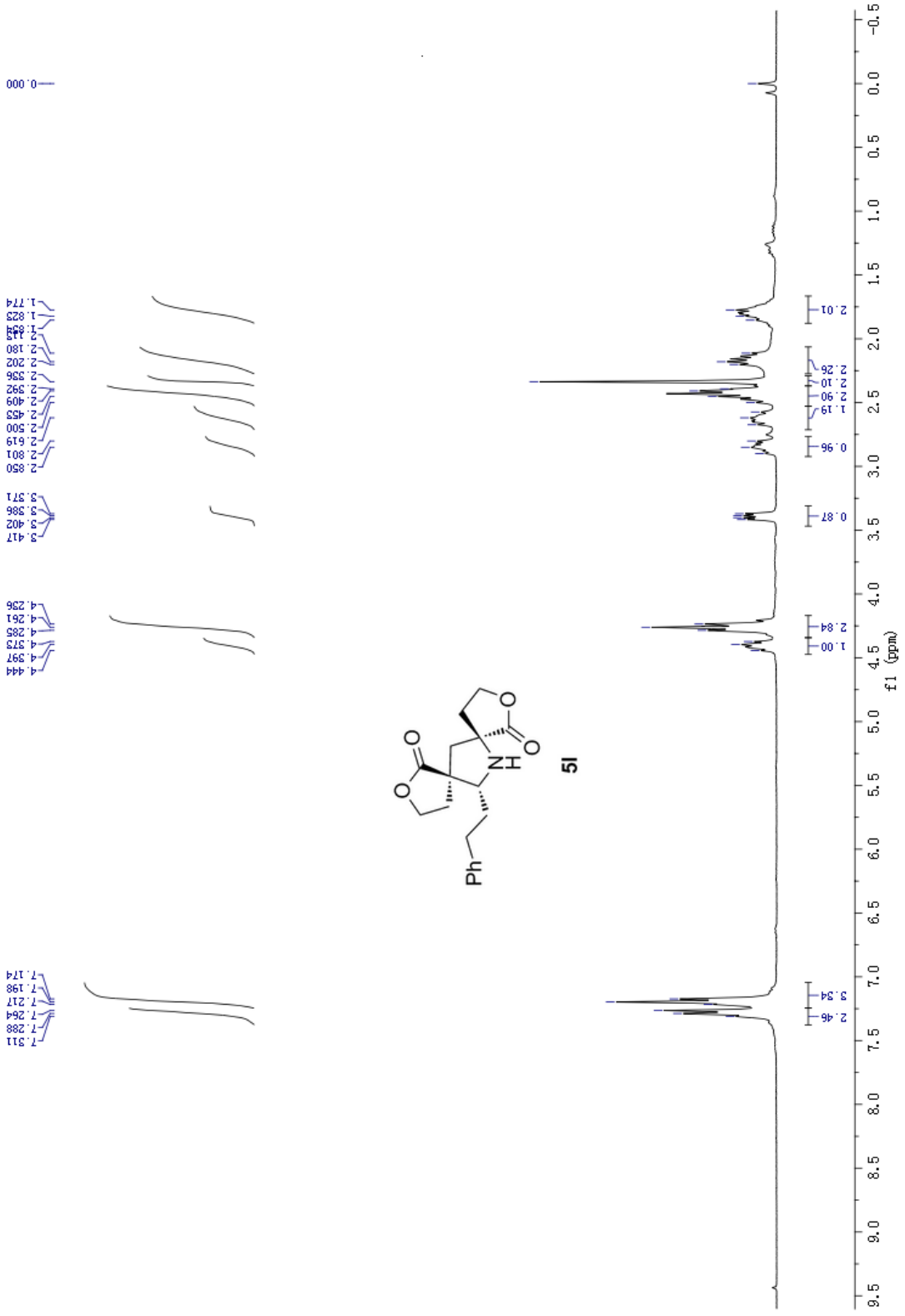
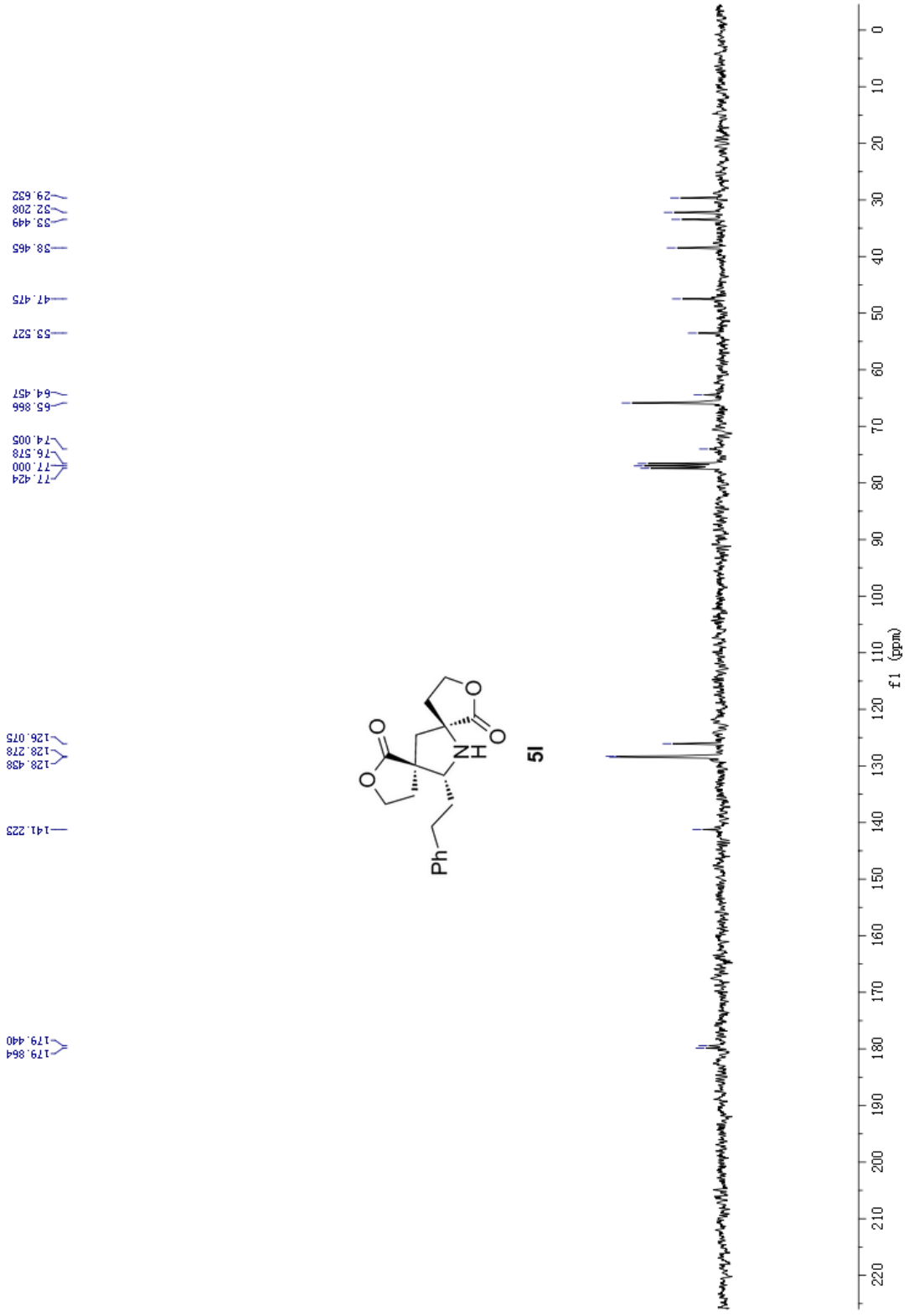
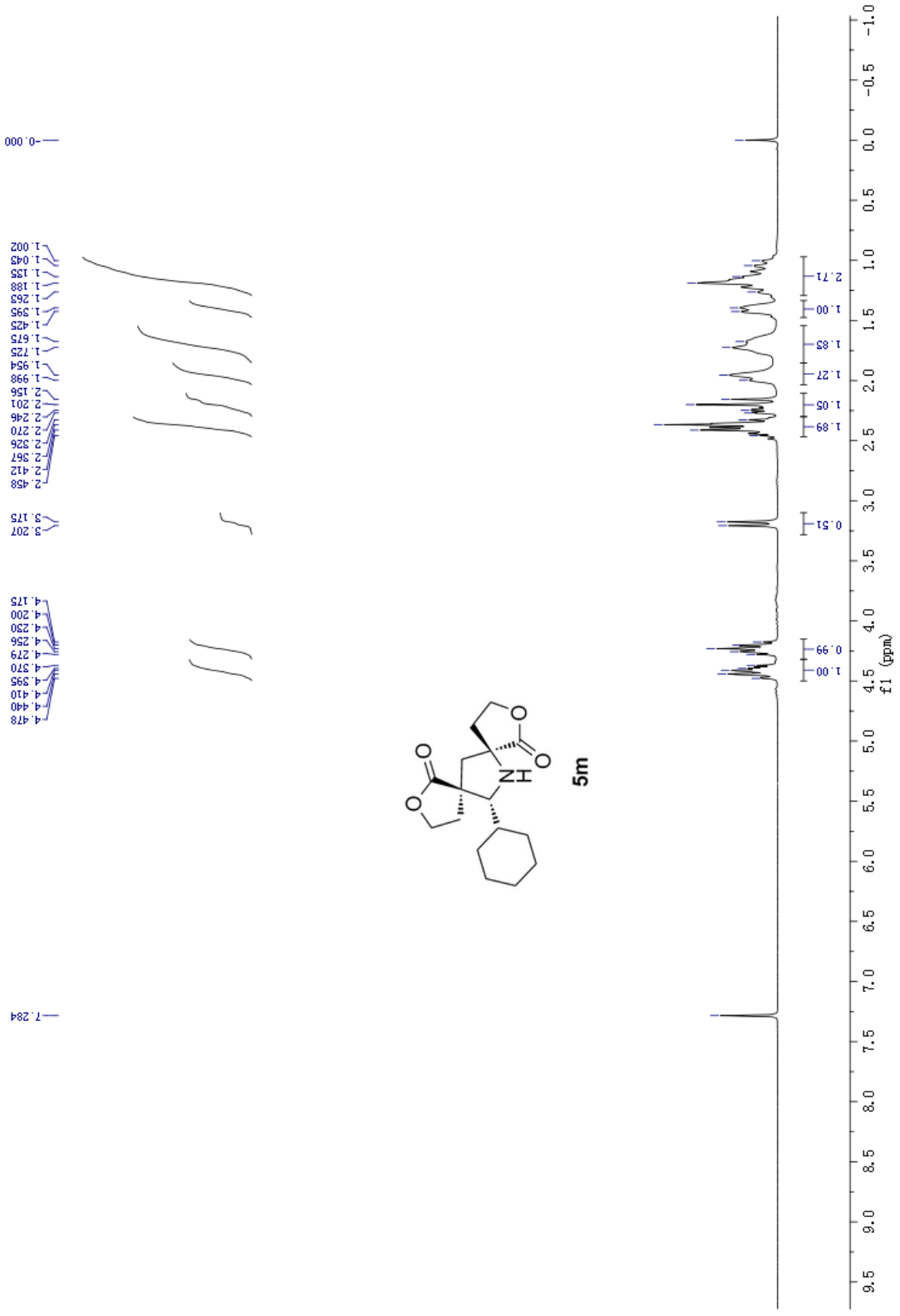
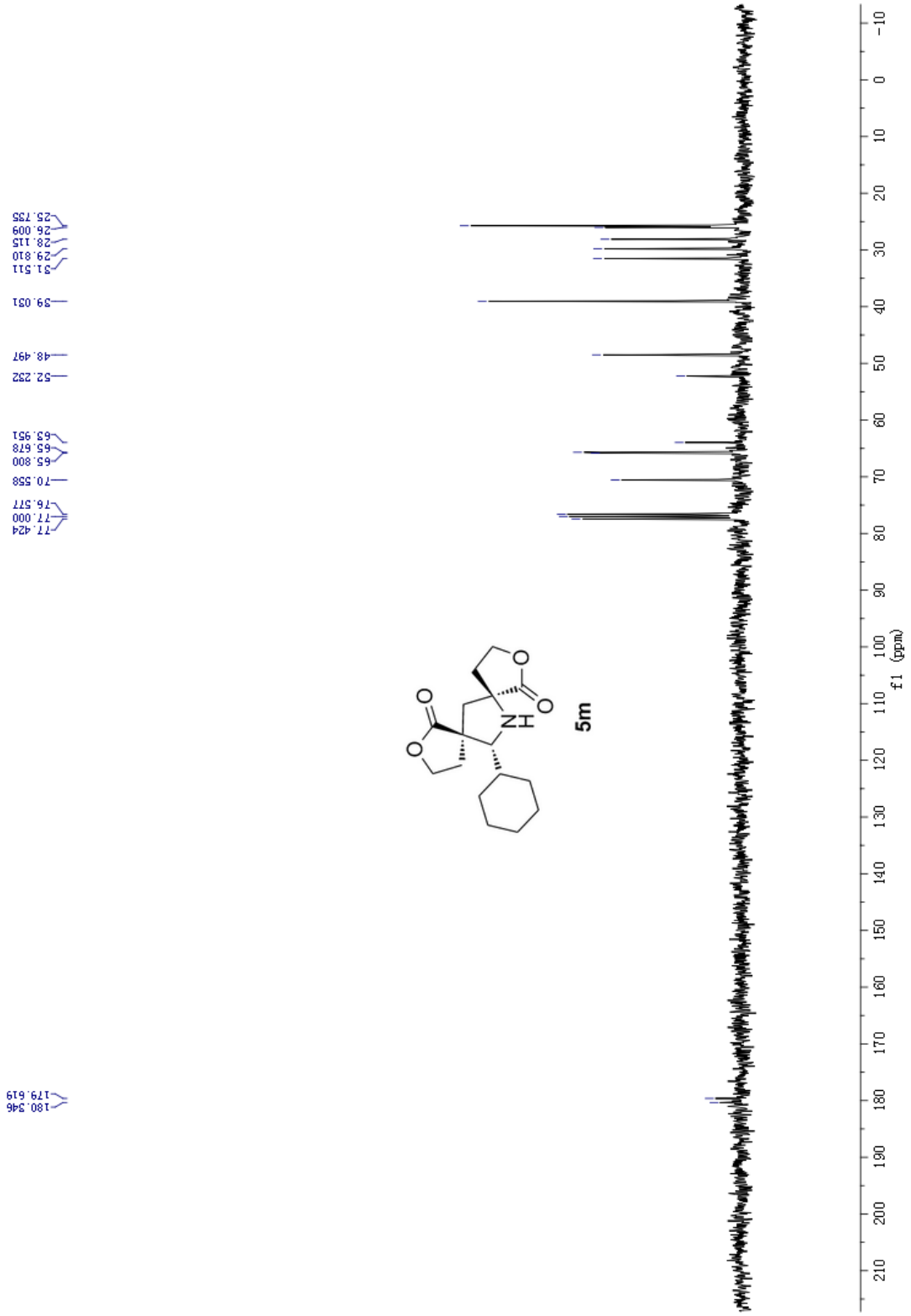



ydc-2-103i
Solvent: D2O
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.350 sec
Width 18867.9 Hz
256 repetitions
OBSERVE C13, 75.4552813 MHz
DECOUPLE H1, 300.0824399 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 16384
Total time 4 hr, 17 min, 25 sec
5k
179.897
179.042
138.293
133.836
133.012
128.464
128.006
127.640
126.510
126.358
125.534
124.526
77.427
77.000
76.603
68.850
66.133
65.462
63.264
54.595
47.422
38.082
30.725
220
200
180
160
140
120
100
80
60
40
20
0 ppm


5l
Ph
O
O
O
O
N
H
f1 (ppm)


29.632
32.208
33.449
38.465
47.475
53.527
64.457
65.866
74.005
76.578
77.000
77.424
126.075
128.278
128.438
141.223
179.864
179.440
Ph
5l
f1 (ppm)


5m
f1 (ppm)


25.735
26.009
28.115
29.810
31.511
39.031
48.497
52.292
63.951
65.678
65.800
70.558
76.577
77.000
77.424
179.619
180.346
5m
f1 (ppm)

IX. HPLC Chromatograms

Data File D:\LC\201111\LTL\LTL-12-79\LTL-12-79 2011-11-15 10-02-07\064-0101.D
Sample Name: LTL-12-79

```
=====================================================================
Acq. Operator   : LTL                              Seq. Line :    1
Acq. Instrument : Instrument 1                      Location : Vial 64
Injection Date  : 11/15/2011 10:03:32 AM                 Inj :    1
                                                  Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\LTL\LTL-10-79\LTL-12-79 2011-11-15 10-02-07\ASH-30-70-1ML-
                  220NM.M
Last changed    : 11/15/2011 10:32:41 AM by LTL
                  (modified after loading)
Analysis Method : D:\LC\201111\LTL\LTL-12-79\LTL-12-79 2011-11-15 10-02-07\064-0101.D\DA.M (
                  ASH-30-70-1ML-220NM.M)
Last changed    : 11/17/2011 5:03:20 PM by hzl
                  (modified after loading)
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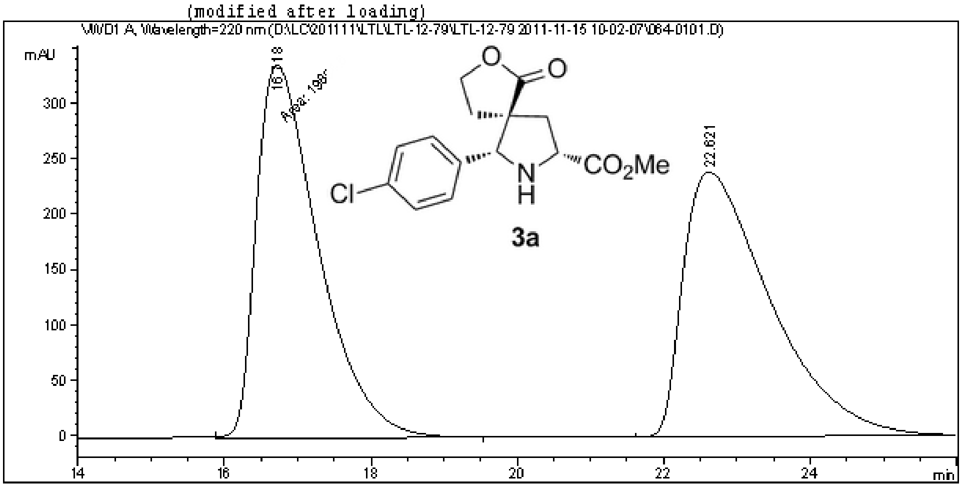



```
=====================================================================
                         Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=220 nm

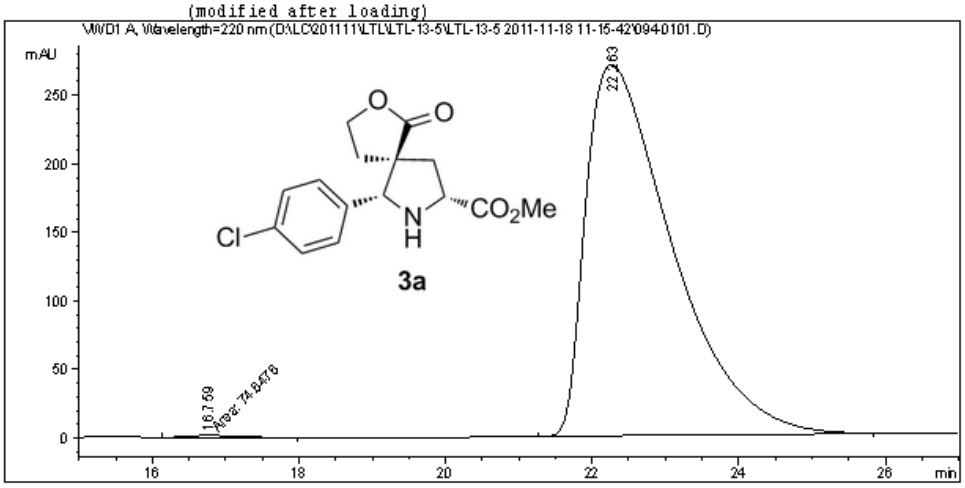
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   mAU   *s   [mAU  ]       %
----|-------|----|-------|----------|----------|--------|
   1  16.718 MM    0.9825 1.98646e4  336.95798  50.0581
   2  22.621 BB    1.2675 1.98185e4  238.95557  49.9419

Totals :                  3.96832e4  575.91354

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

Data File D:\LC\2011ll\LTL\LTL-13-5\LTL-13-5 2011-ll-18 ll-15-42\094-0101.D
Sample Name: LTL-13-5

Acq. Operator	: LTL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 94
Injection Date	: ll/18/2011 ll:17:30 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\2011ll\LTL\LTL-13-5\LTL-13-5 2011-ll-18 ll-15-42\ASH-30-70-1ML-220NM-30MIN.M		
Last changed	: ll/18/2011 ll:14:39 AM by hzl		
Analysis Method	: D:\LC\2011ll\LTL\LTL-13-5\LTL-13-5 2011-ll-18 ll-15-42\094-0101.D\DA.M (ASH-30-70-1ML-220NM-30MIN.M)		
Last changed	: ll/18/2011 12:25:11 PM by hzl (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	16.759	MM	0.7869	74.64757	1.58113	0.3354
2	22.263	BB	1.2419	2.21823e4	270.96863	99.6646
Totals :				2.22569e4	272.54976	

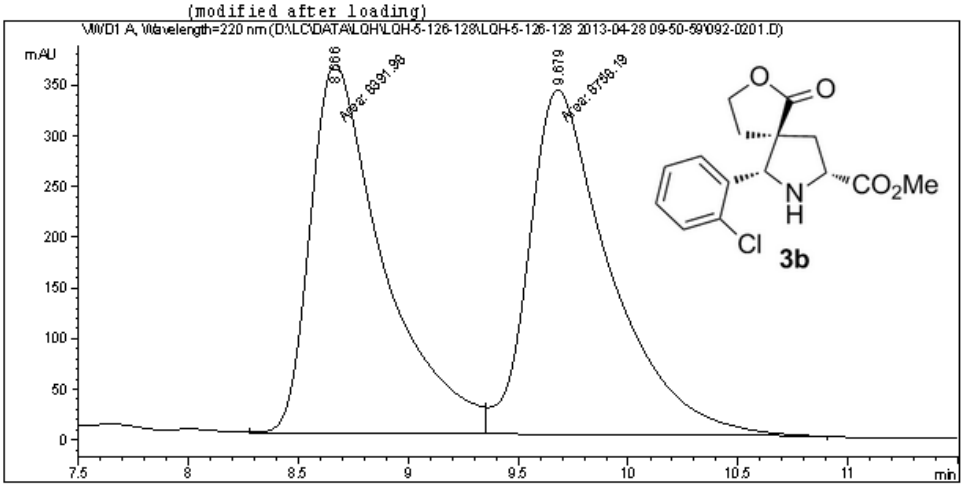
*** End of Report ***

Instrument 1 ll/18/2011 12:25:17 PM hzl

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\092-0201.D
Sample Name: LQH-5-126

```
==================================================================
Acq. Operator   : CX                          Seq. Line :   2
Acq. Instrument : Instrument 1                 Location : Vial 92
Injection Date  : 4/28/2013 10:03:25 AM              Inj :   1
                                             Inj Volume : 5 µl
Acq. Method     : D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\ADH-30-70-
                  10ML-220NM.M
Last changed    : 4/28/2013 10:17:23 AM by CX
                  (modified after loading)
Analysis Method : D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\092-0201.D\
                  DA.M (ADH-30-70-10ML-220NM.M)
Last changed    : 4/28/2013 11:10:53 AM by CX
                  (modified after loading)
```




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

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


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|------|----|-------|----------|----------|-------|
   1   8.666 MF    0.3847 8391.97559  363.57651  48.9323
   2   9.679 FM    0.4296 8758.19141  339.78110  51.0677

Totals :                  1.71502e4   703.35760


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

Instrument 1 4/28/2013 11:11:09 AM CX Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\093-0301.D
Sample Name: LQH-5-128A

```
==================================================================
Acq. Operator   : CX                          Seq. Line :   3
Acq. Instrument : Instrument 1                 Location : Vial 93
Injection Date  : 4/28/2013 10:20:29 AM              Inj :   1
                                             Inj Volume : 5 µl
Acq. Method     : D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\ADH-30-70-
                  10ML-220NM.M
Last changed    : 4/28/2013 10:18:44 AM by CX
                  (modified after loading)
Analysis Method : D:\LC\DATA\LQH\LQH-5-126-128\LQH-5-126-128 2013-04-28 09-50-59\093-0301.D\
                  DA.M (ADH-30-70-10ML-220NM.M)
Last changed    : 4/28/2013 11:12:48 AM by CX
                  (modified after loading)
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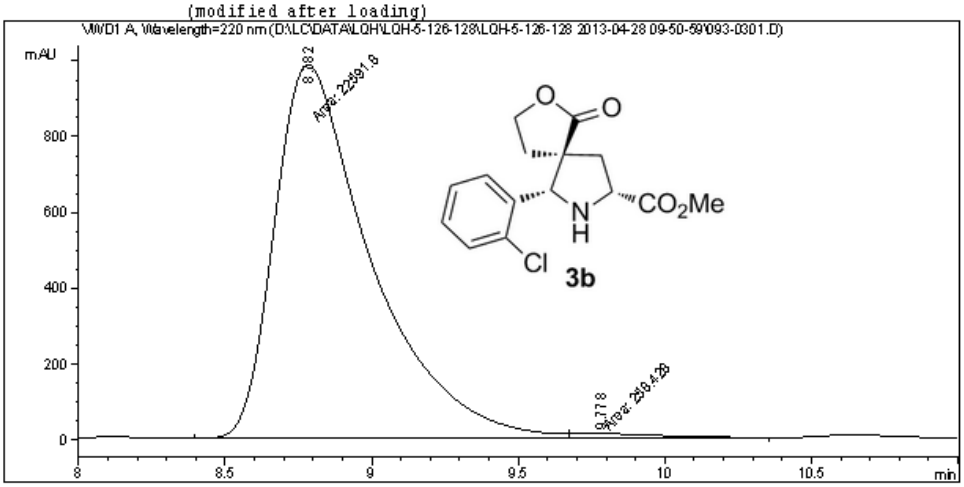
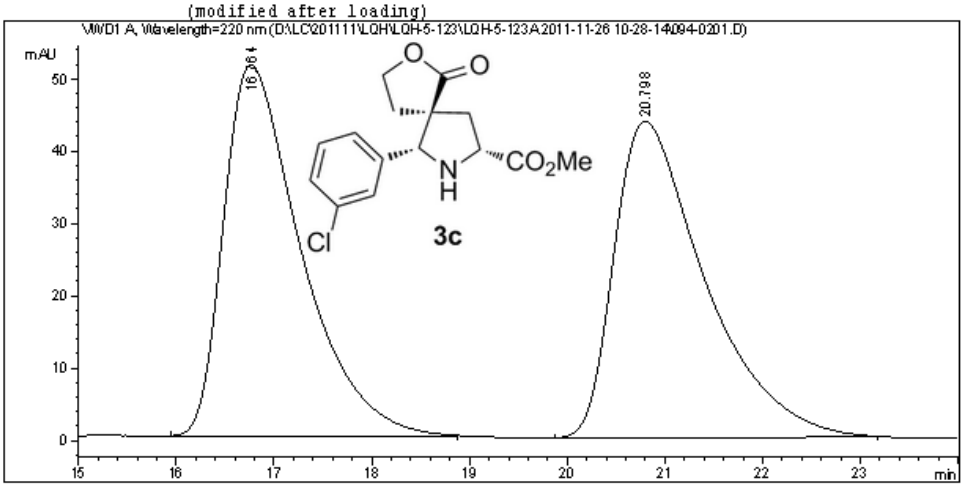



```
==================================================================
                        Area Percent Report
==================================================================

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   8.782 MM    0.3812 2.25918e4   987.80975  98.8690
   2   9.778 MM    0.3674  258.42834   11.72447   1.1310

Totals :                  2.28503e4   999.53422


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

Instrument 1 4/28/2013 11:12:58 AM CX Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\094-0201.D
Sample Name: LQH-5-121C

Acq. Operator	: lqh	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 94
Injection Date	: 11/26/2011 10:52:07 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\ASH-30-70-1ML-220NM.M		
Last changed	: 11/26/2011 11:17:44 AM by lqh (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\094-0201.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/26/2011 11:54:36 AM by HZL (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	16.764	BB	0.8189	2822.21997	51.34835	49.9686
2	20.798	BB	0.9425	2825.76660	43.76609	50.0314
Totals :				5647.98657	95.11444	

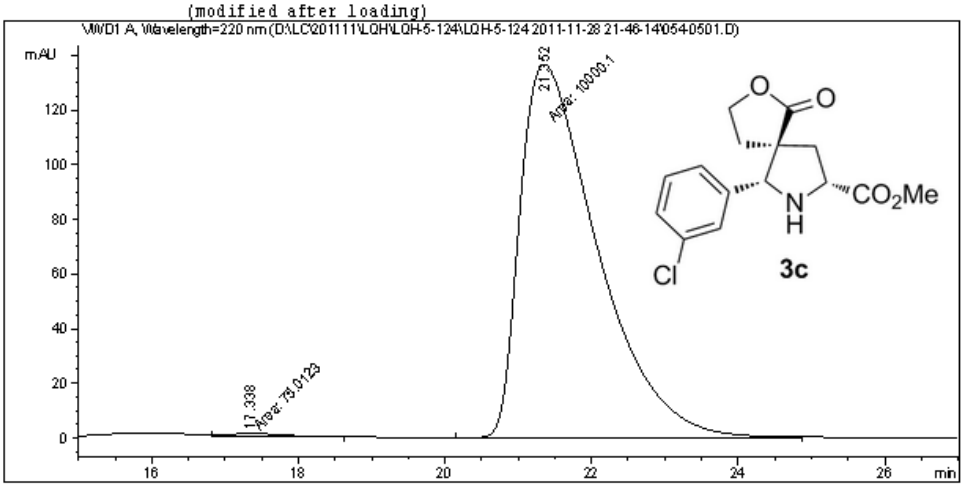
*** End of Report ***

Instrument 1 11/26/2011 11:54:41 AM HZL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\054-0501.D
Sample Name: LQH-5-124C

Acq. Operator	: LQH	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 54
Injection Date	: 11/28/2011 11:48:24 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\ASH-30-70-10ML-220NM-35MIN.M		
Last changed	: 11/28/2011 9:40:40 PM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\054-0501.D\DA.M (ASH-30-70-10ML-220NM-35MIN.M)		
Last changed	: 11/29/2011 12:00:08 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	17.338	MM	0.9675	75.01229	1.29223	0.7445
2	21.352	MM	1.2204	1.00001e4	136.56596	99.2555
Totals :				1.00751e4	137.85819	

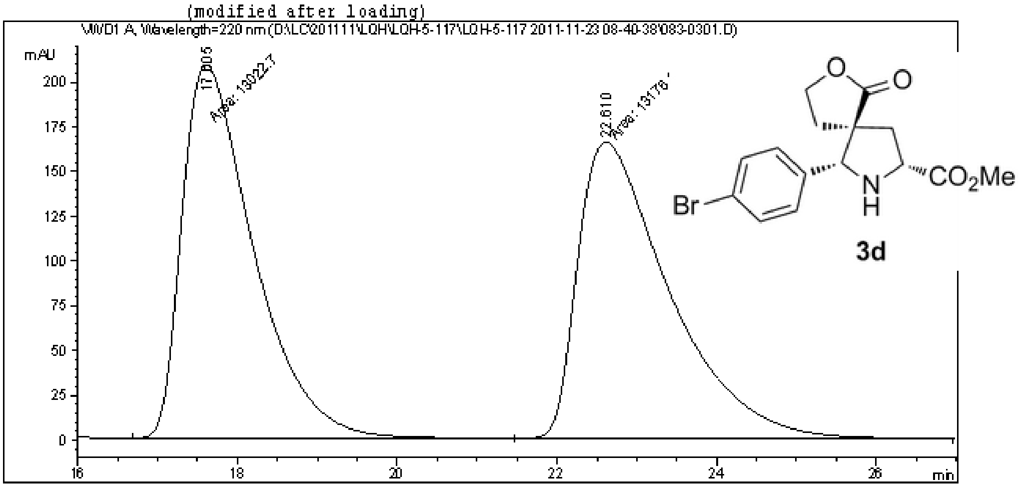
*** End of Report ***

Instrument 1 11/29/2011 12:00:14 PM tmc

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\083-0301.D
Sample Name: LQH-5-117C

===
Acq. Operator : LQH Seq. Line : 3
Acq. Instrument : Instrument 1 Location : Vial 83
Injection Date : 11/23/2011 10:11:32 AM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\ASH-30-70-1ML-220NM.M
Last changed : 11/23/2011 10:40:09 AM by LQH
(modified after loading)
Analysis Method : D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\083-0301.D\DA.M (ASH-30-70-1ML-220NM.M)
Last changed : 11/26/2011 11:40:24 AM by HZL
(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	17.605	MF	1.0402	1.30227e4	208.65845	49.7067
2	22.610	FM	1.3266	1.31764e4	165.53917	50.2933
Totals :				2.61991e4	374.19762	

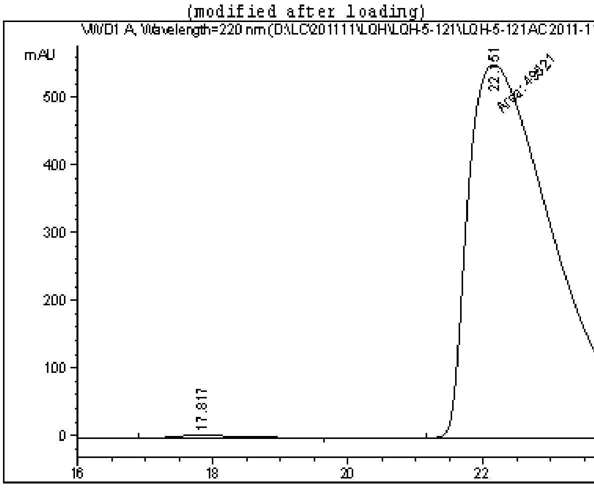
===
*** End of Report ***

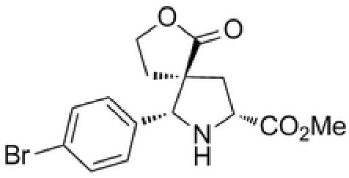
Instrument 1 11/26/2011 11:40:29 AM HZL Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\093-0301.D
Sample Name: LQH-5-121B

===

Acq. Operator : LQH Seq. Line : 3
Acq. Instrument : Instrument 1 Location : Vial 93
Injection Date : 11/24/2011 9:22:45 PM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\ASH-30-70-10ML-220NM-30MIN.M
Last changed : 11/24/2011 8:37:08 PM by HZL
Analysis Method : D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\093-0301.D\DA.M (ASH-30-70-10ML-220NM-30MIN.M)
Last changed : 11/26/2011 12:00:52 PM by HZL
(modified after loading)





3d

===

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	17.817	VB	0.7971	232.72244	3.94681	0.4677
2	22.151	MM	1.4960	4.95210e4	551.71362	99.5323

Totals : 4.97537e4 555.66043

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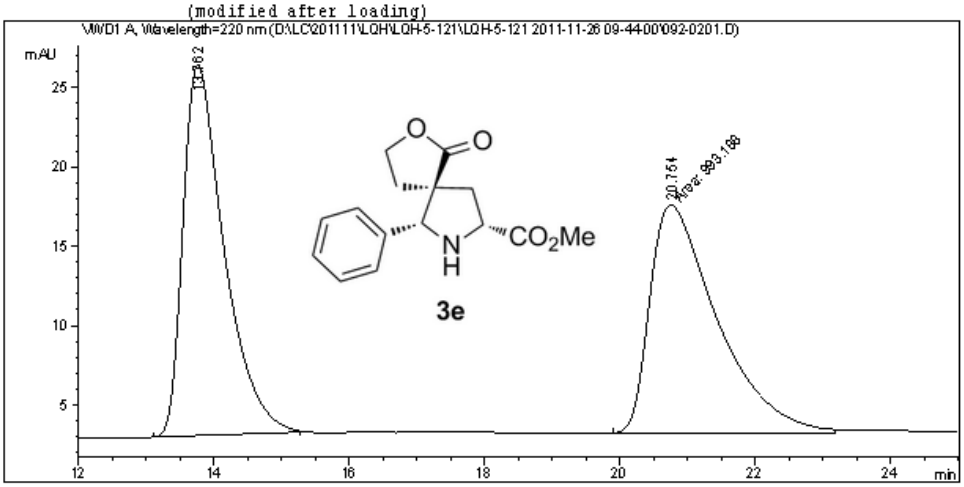
*** End of Report ***

Instrument 1 11/26/2011 12:00:57 PM HZL Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-121\LQH-5-121 2011-11-26 09-44-00\092-0201.D
Sample Name: LQH-5-121A

Acq. Operator	: lqh	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 92
Injection Date	: 11/26/2011 9:56:31 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-121\LQH-5-121 2011-11-26 09-44-00\ASH-30-70-1ML-220NM.M		
Last changed	: 11/26/2011 10:25:37 AM by lqh (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-121\LQH-5-121 2011-11-26 09-44-00\092-0201.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/26/2011 11:44:26 AM by HZL (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	13.762	BB	0.6291	986.78485	23.38632	49.8383
2	20.754	MM	1.1520	993.18842	14.36943	50.1617
Totals :				1979.97327	37.75576	

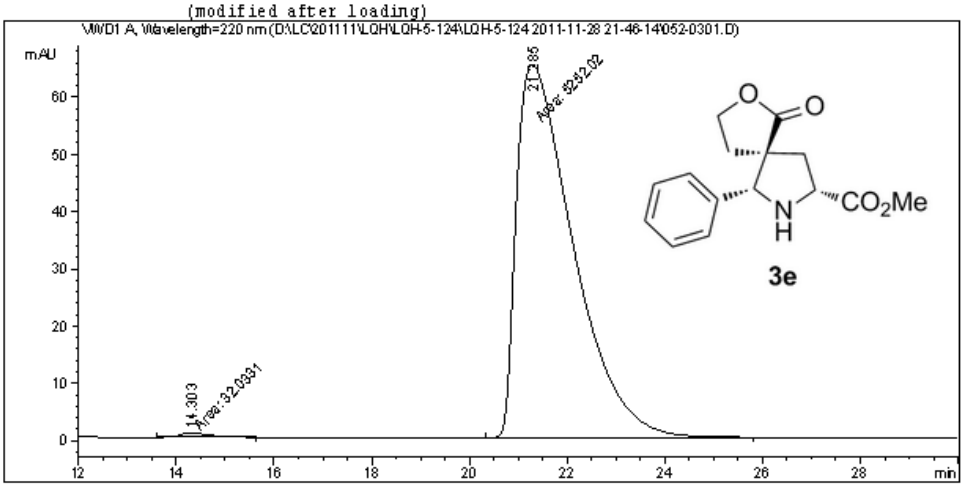
*** End of Report ***

Instrument 1 11/26/2011 11:44:31 AM HZL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\052-0301.D
Sample Name: LQH-5-124A

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 52
Injection Date	: 11/28/2011 10:35:13 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\ASH-30-70-10ML-220NM-35MIN.M		
Last changed	: 11/28/2011 9:40:40 PM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\052-0301.D\DA.M (ASH-30-70-10ML-220NM-35MIN.M)		
Last changed	: 11/29/2011 8:38:54 AM 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	14.303	MM	0.7022	32.03307	7.60255e-1	0.6062
2	21.285	MM	1.3427	5252.01660	65.19064	99.3938
Totals :				5284.04967	65.95090	

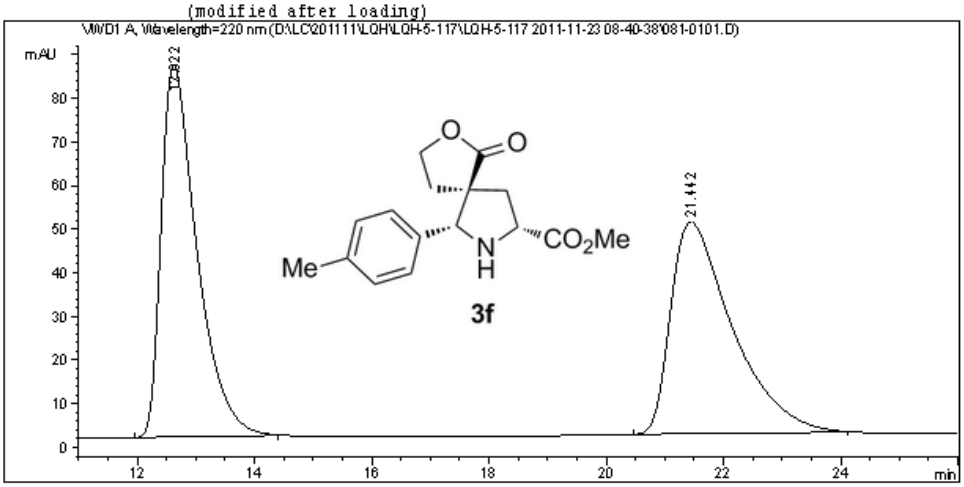
*** End of Report ***

Instrument 1 11/29/2011 8:39:00 AM tmc

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\081-0101.D
Sample Name: LQH-5-117A

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 81
Injection Date	: 11/23/2011 8:41:49 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\ASH-30-70-1ML-220NM.M		
Last changed	: 11/23/2011 9:12:32 AM by LQH (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\081-0101.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/26/2011 11:37:18 AM by HZL (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	12.622	BB	0.6332	3607.51807	85.30511	50.4197
2	21.442	BB	1.0646	3547.45557	48.57446	49.5803
Totals :				7154.97363	133.87958	

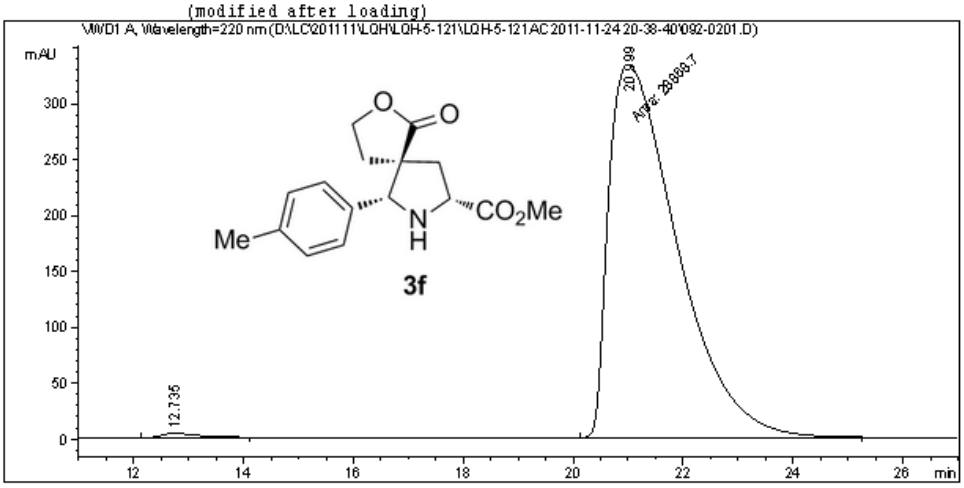
*** End of Report ***

Instrument 1 11/26/2011 11:37:24 AM HZL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\092-0201.D
Sample Name: LQH-5-121A

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 92
Injection Date	: 11/24/2011 8:51:00 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\ASH-30-70-10ML-220NM-30MIN.M		
Last changed	: 11/24/2011 8:37:08 PM by HZL		
Analysis Method	: D:\LC\201111\LQH\LQH-5-121\LQH-5-121AC 2011-11-24 20-38-40\092-0201.D\DA.M (ASH-30-70-10ML-220NM-30MIN.M)		
Last changed	: 11/26/2011 11:57:52 AM by HZL (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	12.735	BB	0.6512	180.80287	3.90208	0.6224
2	20.999	MM	1.4402	2.88687e4	334.07495	99.3776
Totals :				2.90495e4	337.97703	

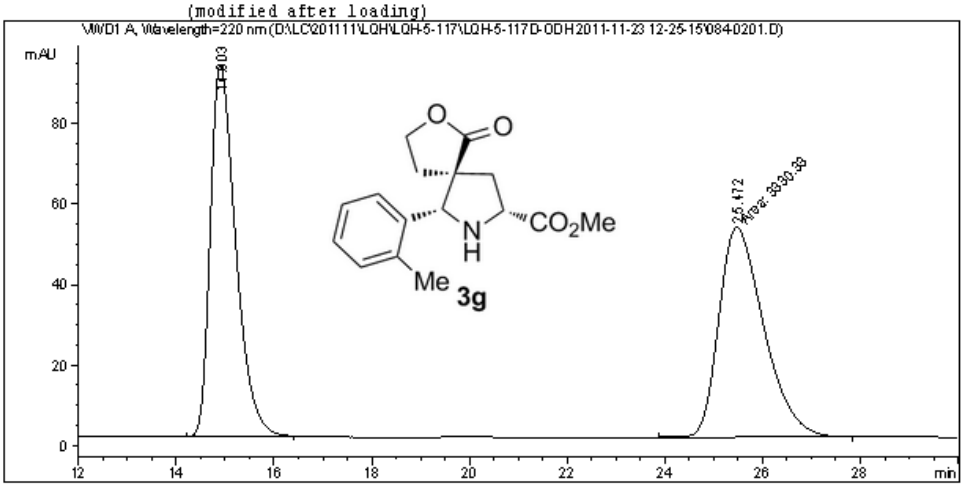
*** End of Report ***

Instrument 1 11/26/2011 11:58:52 AM HZL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-117\LQH-5-117D-ODH 2011-11-23 12-25-15\084-0201.D
Sample Name: LQH-5-117D-ODH

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 84
Injection Date	: 11/23/2011 12:37:38 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-117\LQH-5-117D-ODH 2011-11-23 12-25-15\ODH-20-80-10ML-220NM.M		
Last changed	: 11/23/2011 12:24:25 PM by hzl		
Analysis Method	: D:\LC\201111\LQH\LQH-5-117\LQH-5-117D-ODH 2011-11-23 12-25-15\084-0201.D\DA.M (ODH-20-80-10ML-220NM.M)		
Last changed	: 11/26/2011 11:41:48 AM by HZL (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	14.903	BB	0.5536	3339.64868	92.52085	50.0699
2	25.472	MM	1.0647	3330.32690	52.13207	49.9301
Totals :				6669.97559	144.65292	

*** End of Report ***

Instrument 1 11/26/2011 11:41:53 AM HZL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-119\LQH-5-119D 2011-11-27 18-36-36\081-0201.D
Sample Name: LQH-5-119D

```
=====================================================================
Acq. Operator   : LQH                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 81
Injection Date  : 11/27/2011 6:48:31 PM            Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\LQH\LQH-5-119\LQH-5-119D 2011-11-27 18-36-36\ODH-20-80-10ML-
                  220NM-35MIN.M
Last changed    : 11/27/2011 6:34:35 PM by LTL
Analysis Method : D:\LC\201111\LQH\LQH-5-119\LQH-5-119D 2011-11-27 18-36-36\081-0201.D\DA.M
                  (ODH-20-80-10ML-220NM-35MIN.M)
Last changed    : 12/1/2011 5:08:22 PM by LTL
                  (modified after loading)
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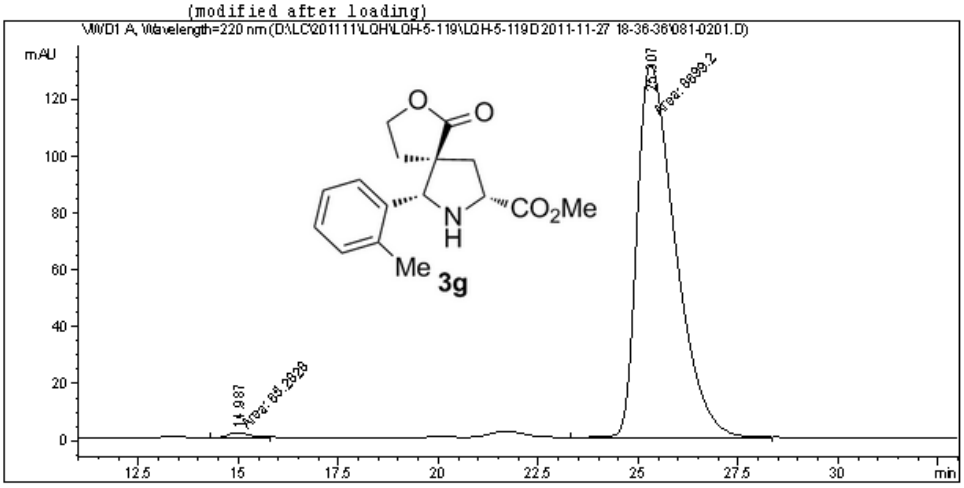


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=====================================================================
                        Area Percent Report
=====================================================================

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  14.987 MF    0.6159   65.28275    1.76669   0.7449
   2  25.307 MF    1.1054 8699.19824  131.15779  99.2551

Totals :                  8764.48100  132.92448


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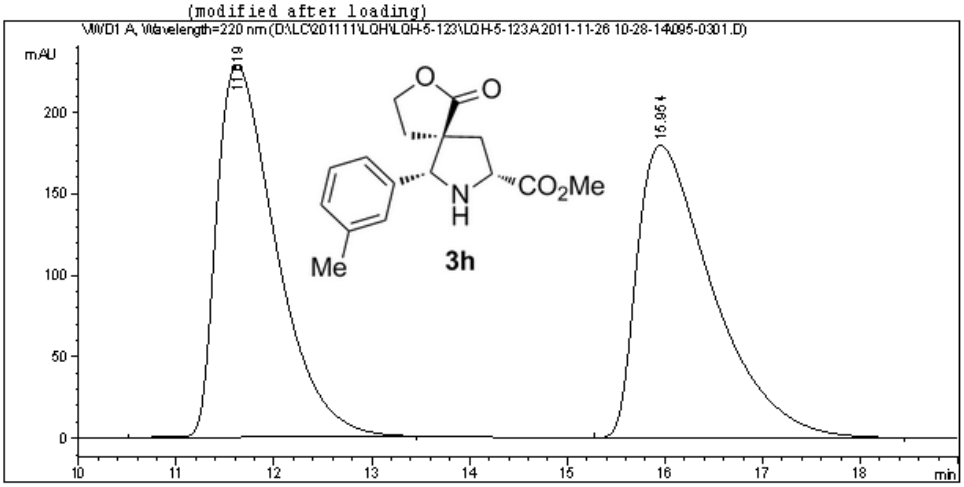

Instrument 1 12/1/2011 5:08:27 PM LTL Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\095-0301.D
Sample Name: LQH-5-121D

Acq. Operator	: lqh	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 95
Injection Date	: 11/26/2011 11:19:34 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\ASH-30-70-1ML-220NM.M		
Last changed	: 11/26/2011 11:18:21 AM by lqh (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A 2011-11-26 10-28-14\095-0301.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/26/2011 11:55:41 AM by HZL (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	11.619	BB	0.6491	9637.21875	228.60901	49.8263
2	15.954	BB	0.8179	9704.40039	179.70007	50.1737
Totals :				1.9341e4	408.30908	

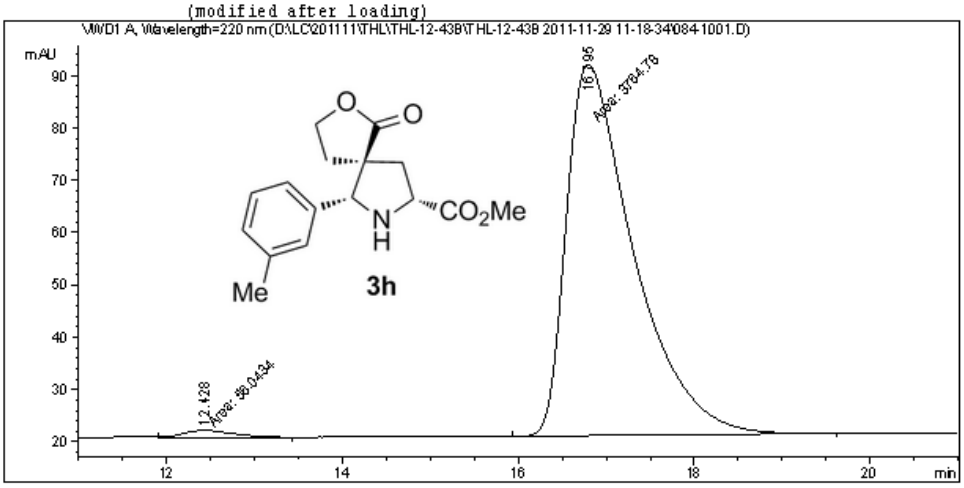
*** End of Report ***

Instrument 1 11/26/2011 11:55:46 AM HZL

Page 1 of 1

Data File D:\LC\201111\THL\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\084-1001.D
Sample Name: LQH-5-124D

Acq. Operator	: THL	Seq. Line	: 10
Acq. Instrument	: Instrument 1	Location	: Vial 84
Injection Date	: 11/29/2011 2:35:16 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\thl\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\ASH-30-70-10ML-220NM-30MIN.M		
Last changed	: 11/24/2011 8:37:08 PM by HZL		
Analysis Method	: D:\LC\201111\THL\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\084-1001.D\DA.M (ASH-30-70-10ML-220NM-30MIN.M)		
Last changed	: 12/1/2011 9:54:26 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	12.428	MM	0.6728	56.04337	1.38823	1.4668
2	16.795	MM	0.8837	3764.78394	71.00076	98.5332
Totals :				3820.82731	72.38900	

*** End of Report ***

Instrument 1 12/1/2011 9:54:32 PM LTL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\082-0201.D
Sample Name: LQH-5-117B

```
==================================================================
Acq. Operator   : LQH                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 82
Injection Date  : 11/23/2011 9:14:33 AM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\ASH-30-70-1ML-
                  220NM.M
Last changed    : 11/23/2011 10:09:43 AM by LQH
                  (modified after loading)
Analysis Method : D:\LC\201111\LQH\LQH-5-117\LQH-5-117 2011-11-23 08-40-38\082-0201.D\DA.M (
                  ASH-30-70-1ML-220NM.M)
Last changed    : 11/26/2011 11:39:06 AM by HZL
                  (modified after loading)
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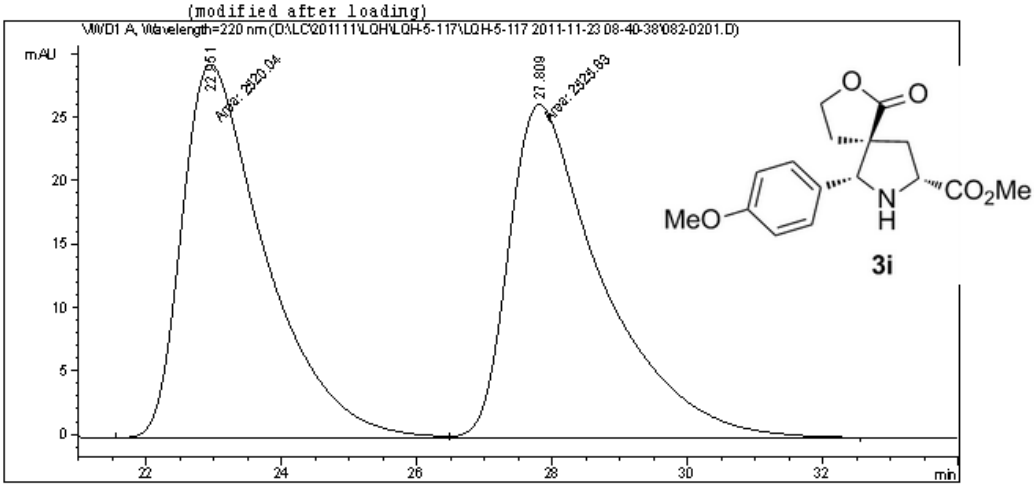


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==================================================================
                        Area Percent Report
==================================================================

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  22.951 MF    1.4289 2520.03613   29.39459  49.9426
   2  27.809 FM    1.6038 2525.83228   26.24825  50.0574

Totals :                  5045.86841   55.64285


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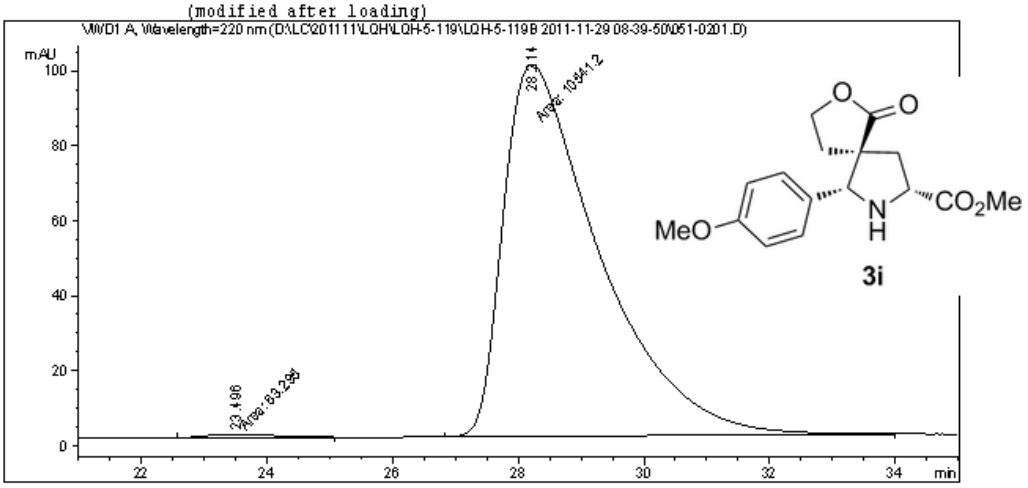

Instrument 1 11/26/2011 11:39:25 AM HZL Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-119\LQH-5-119B 2011-11-29 08-39-50\051-0201.D
Sample Name: LQH-5-119B

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 51
Injection Date	: 11/29/2011 8:52:34 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-119\LQH-5-119B 2011-11-29 08-39-50\ASH-30-70-10ML-220NM-35MIN.M		
Last changed	: 11/28/2011 9:40:40 PM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-119\LQH-5-119B 2011-11-29 08-39-50\051-0201.D\DA.M (ASH-30-70-10ML-220NM-35MIN.M)		
Last changed	: 12/1/2011 5:05:49 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	23.496	MM	1.1791	63.29495	8.94683e-1	0.5969
2	28.214	MM	1.7706	1.05412e4	99.22559	99.4031
Totals :				1.06045e4	100.12028	

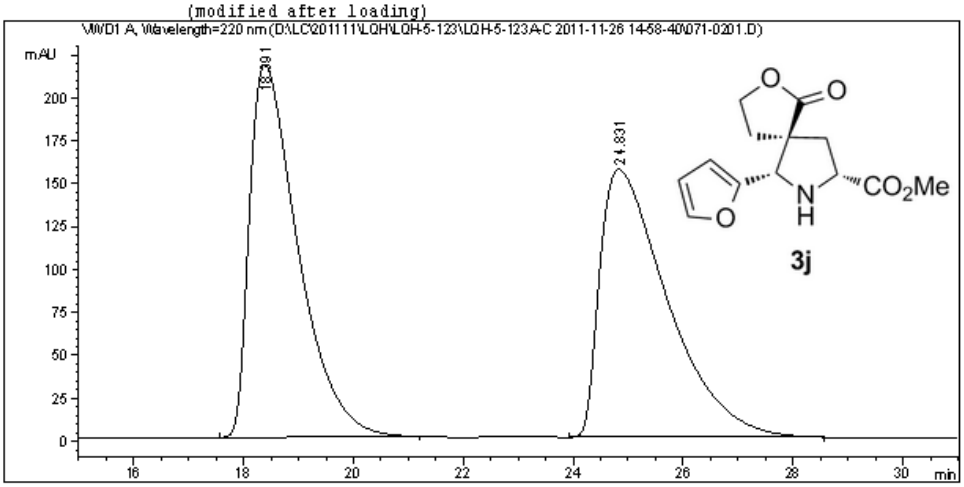
*** End of Report ***

Instrument 1 12/1/2011 5:05:55 PM LTL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\071-0201.D
Sample Name: LQH-5-123A

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/26/2011 3:11:18 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\ASH-30-70-1ML-220NM.M		
Last changed	: 11/26/2011 3:46:33 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\071-0201.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/29/2011 11:45:14 AM 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	18.391	BB	0.9337	1.33091e4	217.06453	49.8675
2	24.831	BB	1.2838	1.33798e4	156.12032	50.1325
Totals :				2.66889e4	373.18484	

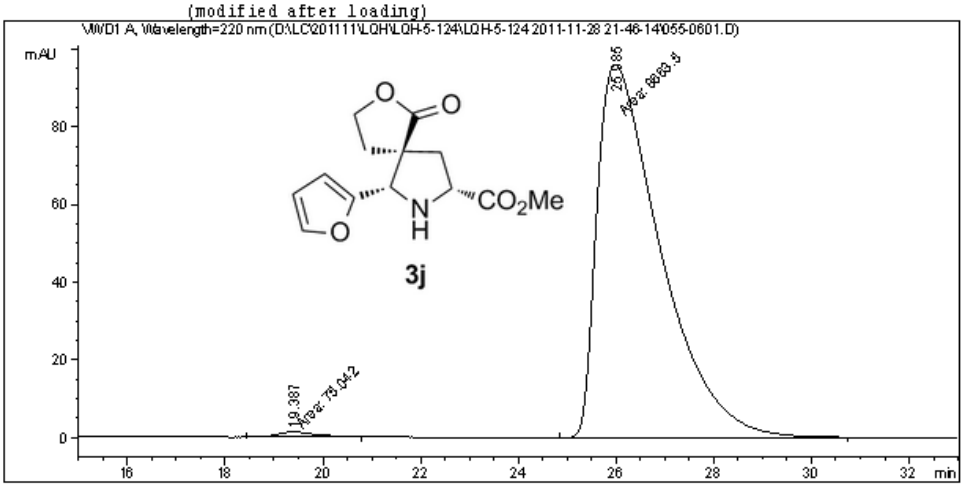
*** End of Report ***

Instrument 1 11/29/2011 11:45:19 AM tmc

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\055-0601.D
Sample Name: LQH-5-124E

Acq. Operator	: LQH	Seq. Line	: 6
Acq. Instrument	: Instrument 1	Location	: Vial 55
Injection Date	: 11/29/2011 12:25:19 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\ASH-30-70-10ML-220NM-35MIN.M		
Last changed	: 11/28/2011 9:40:40 PM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\055-0601.D\DA.M (ASH-30-70-10ML-220NM-35MIN.M)		
Last changed	: 11/29/2011 12:01:44 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	19.387	MM	0.9288	75.04195	1.34653	0.8587
2	25.985	MM	1.5016	8663.50488	96.16138	99.1413
Totals :				8738.54684	97.50791	

*** End of Report ***

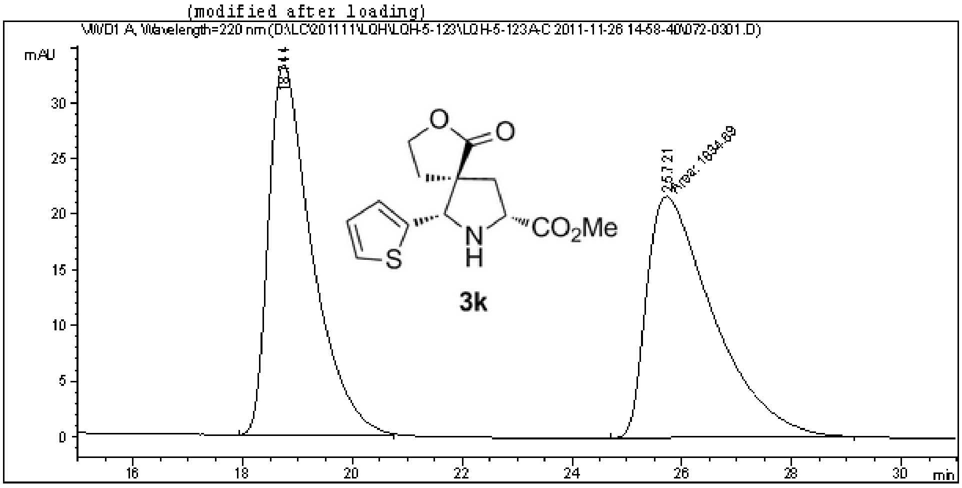
Instrument 1 11/29/2011 12:01:49 PM tmc

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\072-0301.D
Sample Name: LQH-5-123B

===

Acq. Operator : LQH   Seq. Line : 3
Acq. Instrument : Instrument 1   Location : Vial 72
Injection Date : 11/26/2011 3:48:16 PM   Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\ASH-30-70-1ML-220NM.M
Last changed : 11/26/2011 4:19:52 PM by LQH
(modified after loading)
Analysis Method : D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\072-0301.D\DA.M (ASH-30-70-1ML-220NM.M)
Last changed : 11/29/2011 11:46:54 AM 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	18.744	BB	0.8101	1828.05859	33.26278	49.9095
2	25.721	MM	1.4108	1834.68884	21.67430	50.0905

Totals : 3662.74744 54.93708

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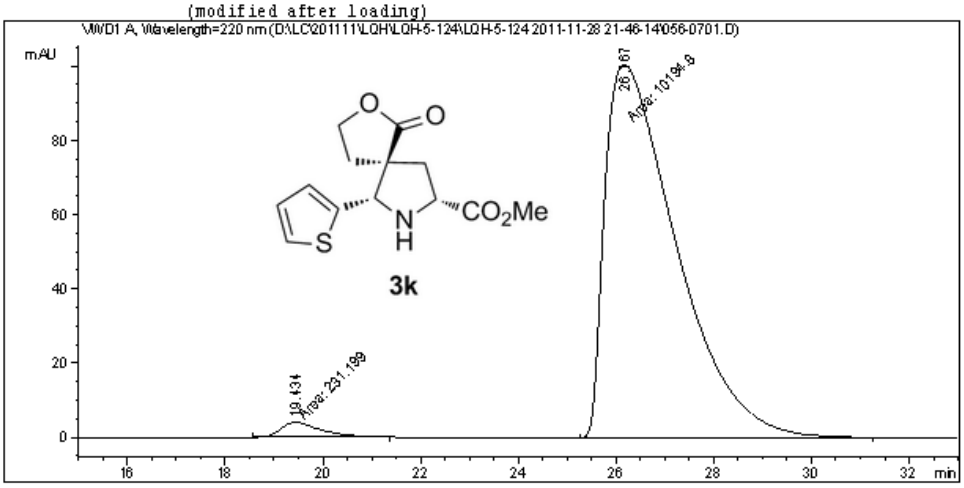
*** End of Report ***

Instrument 1 11/29/2011 11:46:58 AM tmc   Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\056-0701.D
Sample Name: LQH-5-124F

Acq. Operator	: LQH	Seq. Line	: 7
Acq. Instrument	: Instrument 1	Location	: Vial 56
Injection Date	: 11/29/2011 1:01:47 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\ASH-30-70-10ML-220NM-35MIN.M		
Last changed	: 11/28/2011 9:40:40 PM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124 2011-11-28 21-46-14\056-0701.D\DA.M (ASH-30-70-10ML-220NM-35MIN.M)		
Last changed	: 12/1/2011 5:13:39 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	19.434	MM	0.9583	231.19904	4.02083	2.2175
2	26.167	MM	1.6899	1.01948e4	100.54826	97.7825
Totals :				1.04260e4	104.56909	

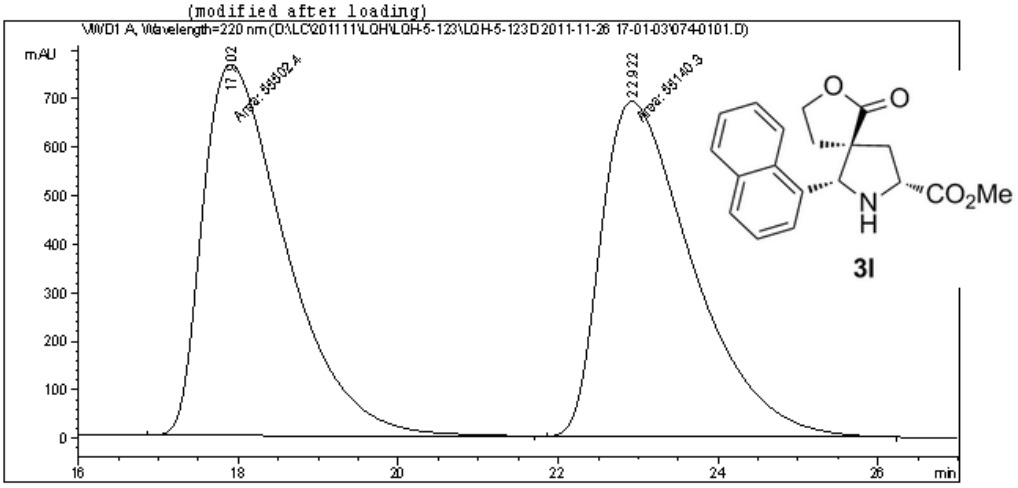
*** End of Report ***

Instrument 1 12/1/2011 5:13:45 PM LTL

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123D 2011-11-26 17-01-03\074-0101.D
Sample Name: LQH-5-123D

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 74
Injection Date	: 11/26/2011 5:02:42 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123D 2011-11-26 17-01-03\ASH-30-70-1ML-220NM.M		
Last changed	: 9/7/2011 3:17:43 PM by thl		
Analysis Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123D 2011-11-26 17-01-03\074-0101.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/29/2011 11:52:00 AM 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	17.902	MM	1.2084	5.55024e4	765.53265	50.1636
2	22.922	MM	1.3314	5.51403e4	690.26672	49.8364
Totals :				1.10643e5	1455.79938	

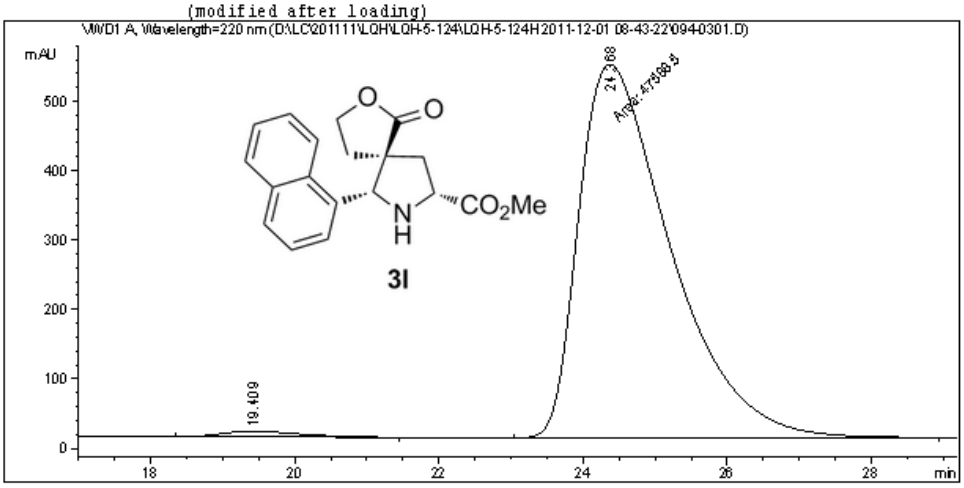
*** End of Report ***

Instrument 1 11/29/2011 11:52:06 AM tmc

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-124\LQH-5-124H 2011-12-01 08-43-22\094-0301.D
Sample Name: LQH-5-124H

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 94
Injection Date	: 12/1/2011 9:32:19 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124H 2011-12-01 08-43-22\ASH-30-70-10ML-220NM-40MIN.M		
Last changed	: 11/29/2011 11:42:14 AM by tmc		
Analysis Method	: D:\LC\201111\LQH\LQH-5-124\LQH-5-124H 2011-12-01 08-43-22\094-0301.D\DA.M (ASH-30-70-10ML-220NM-40MIN.M)		
Last changed	: 12/1/2011 9:52:17 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

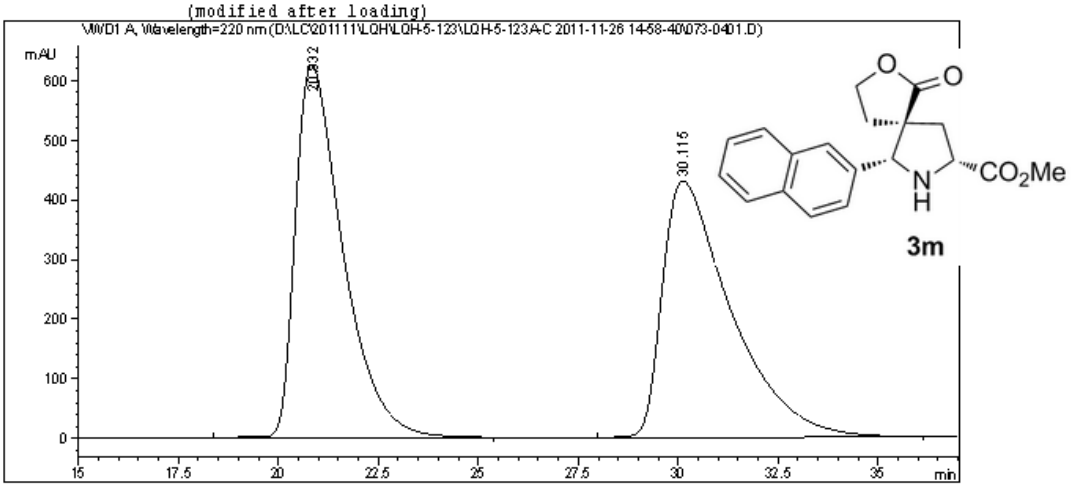
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.409	BB	1.1118	632.18121	8.26942	1.3110
2	24.368	MM	1.4743	4.75885e4	537.98401	98.6890
Totals :				4.82207e4	546.25343	

*** End of Report ***

Instrument 1 12/1/2011 9:52:24 PM LTL Page 1 of 1

Data File D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\073-0401.D
Sample Name: LQH-5-123C

Acq. Operator	: LQH	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 73
Injection Date	: 11/26/2011 4:21:35 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\ASH-30-70-1ML-220NM.M		
Last changed	: 11/26/2011 4:59:25 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-5-123\LQH-5-123A-C 2011-11-26 14-58-40\073-0401.D\DA.M (ASH-30-70-1ML-220NM.M)		
Last changed	: 11/29/2011 11:48:12 AM 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	20.832	BB	1.2513	5.16021e4	625.16650	49.9967
2	30.115	BB	1.7414	5.16089e4	430.29391	50.0033
Totals :				1.03211e5	1055.46042	

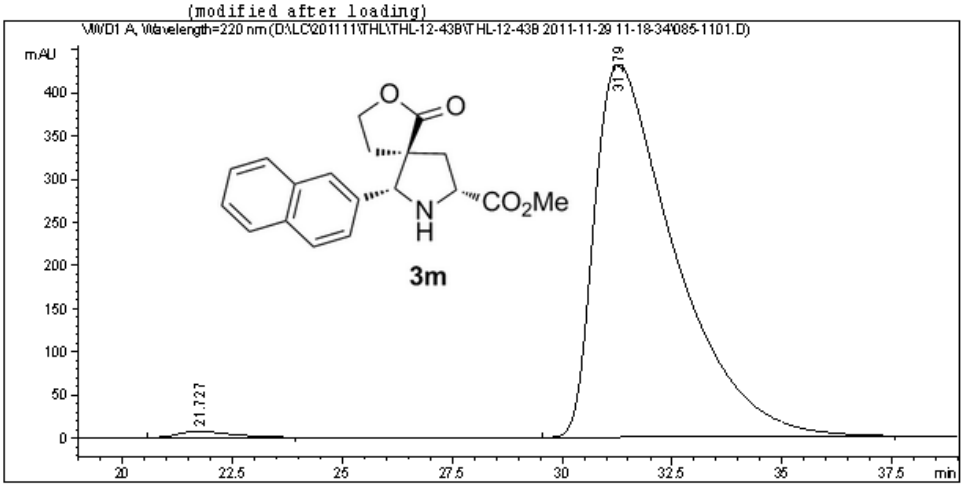
*** End of Report ***

Instrument 1 11/29/2011 11:48:21 AM tmc

Page 1 of 1

Data File D:\LC\201111\THL\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\085-1101.D
Sample Name: LQH-5-124G

Acq. Operator	: THL	Seq. Line	: 11
Acq. Instrument	: Instrument 1	Location	: Vial 85
Injection Date	: 11/29/2011 3:06:32 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\thl\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\ASH-30-70-10ML-220NM-40MIN.M		
Last changed	: 11/29/2011 11:42:14 AM by tmc		
Analysis Method	: D:\LC\201111\THL\THL-12-43B\THL-12-43B 2011-11-29 11-18-34\085-1101.D\DA.M (ASH-30-70-10ML-220NM-40MIN.M)		
Last changed	: 12/1/2011 9:56:27 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

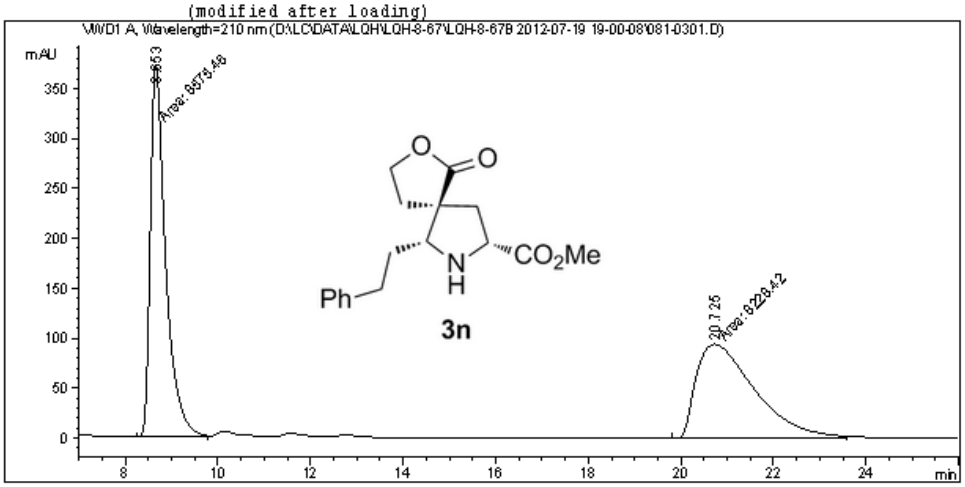
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.727	BB	1.1171	592.79419	7.07624	1.0663
2	31.279	BB	1.8771	5.49990e4	431.95871	98.9337
Totals :				5.55918e4	439.03495	

*** End of Report ***

Instrument 1 12/1/2011 9:56:33 PM LTL

Data File D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\081-0301.D
Sample Name: LQH-8-67B

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 81
Injection Date	: 7/19/2012 8:11:32 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\ADH-30-70-10ML-210NM.M		
Last changed	: 7/19/2012 8:39:58 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\081-0301.D\DA.M (ADH-30-70-10ML-210NM.M)		
Last changed	: 3/16/2013 4:47:33 PM by FX (modified after loading)		



Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

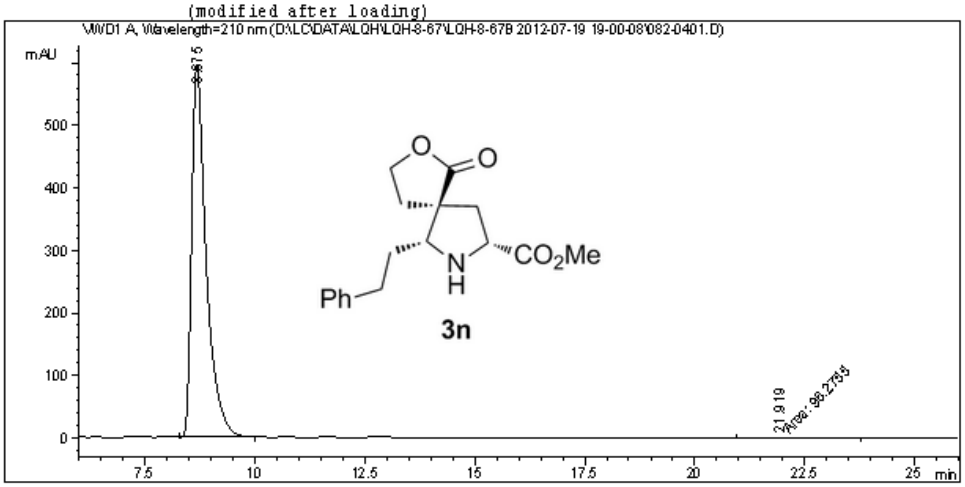
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.653	MM	0.3835	8575.46191	372.67734	51.0326
2	20.725	MF	1.4618	8228.41699	93.81354	48.9674
Totals :				1.68039e4	466.49088	

*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\082-0401.D
Sample Name: LQH-8-68B

Acq. Operator	: LQH	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 82
Injection Date	: 7/19/2012 8:43:27 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\ADH-30-70-10ML-210NM.M		
Last changed	: 7/19/2012 8:41:40 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\DATA\LQH\LQH-8-67\LQH-8-67B 2012-07-19 19-00-08\082-0401.D\DA.M (ADH-30-70-10ML-210NM.M)		
Last changed	: 3/16/2013 4:49:45 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=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.675	VB	0.3392	1.36807e4	595.89819	99.3012
2	21.919	MM	1.3188	96.27553	1.21669	0.6988

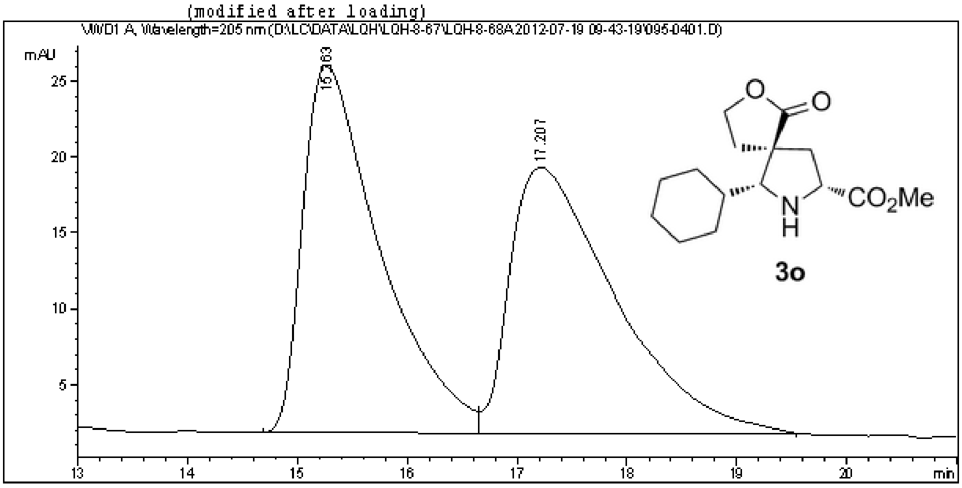
Totals : 1.37770e4 597.11489

*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\095-0401.D
Sample Name: LQH-8-67A

===

Acq. Operator : LQH Seq. Line : 4
Acq. Instrument : Instrument 1 Location : Vial 95
Injection Date : 7/19/2012 11:36:58 AM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\ASH-20-80-10ML-205NM-25MIN.M
Last changed : 7/19/2012 11:28:51 AM by LQH
Analysis Method : D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\095-0401.D\DA.M (ASH-20-80-10ML-205NM-25MIN.M)
Last changed : 3/16/2013 4:36:41 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=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.263	BV	0.7113	1186.18982	24.27481	50.2759
2	17.207	VB	0.9290	1173.17004	17.53744	49.7241
Totals :				2359.35986	41.81225	

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*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\096-0101.D
Sample Name: LQH-8-68A

```
    ====================================================================
    Acq. Operator   : LQH                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 96
    Injection Date  : 7/19/2012 9:44:37 AM               Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\ASH-20-80-10ML-
                      220NM.M
    Last changed    : 7/19/2012 10:09:35 AM by LQH
                      (modified after loading)
    Analysis Method : D:\LC\DATA\LQH\LQH-8-67\LQH-8-68A 2012-07-19 09-43-19\096-0101.D\DA.M (
                      ASH-20-80-10ML-220NM.M)
    Last changed    : 3/16/2013 4:41:58 PM by FX
                      (modified after loading)
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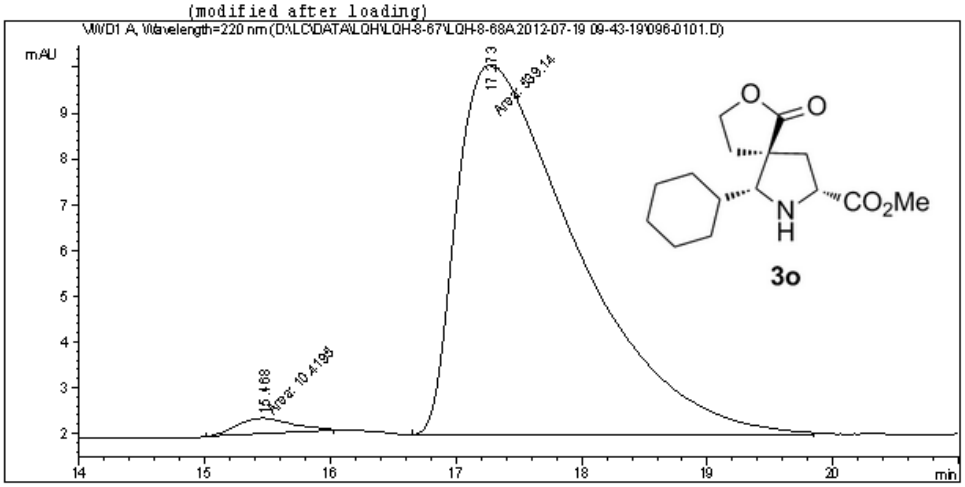



```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

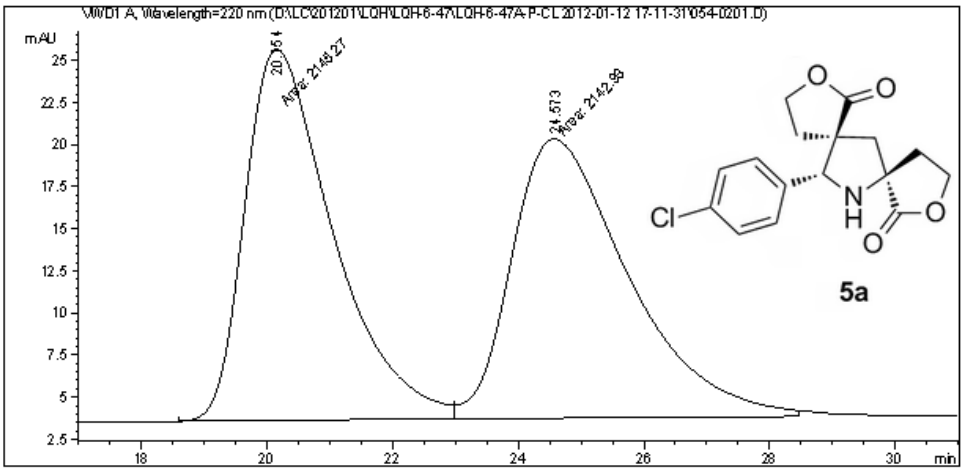
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  15.468 MM    0.3713   10.41946 3.36199e-1   1.8960
       2  17.273 MM    1.1095  539.13971    8.09916  98.1040

    Totals :                   549.55917    8.43536


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

Data File D:\LC\201201\LQH\LQH-6-47\LQH-6-47A-P-CL 2012-01-12 17-11-31\054-0201.D
Sample Name: LQH-6-47A-P-Cl

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 54
Injection Date	: 1/12/2012 5:23:58 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-47A-P-CL 2012-01-12 17-11-31\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-47A-P-CL 2012-01-12 17-11-31\054-0201.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 2/28/2012 1:47:54 PM by LQH (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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

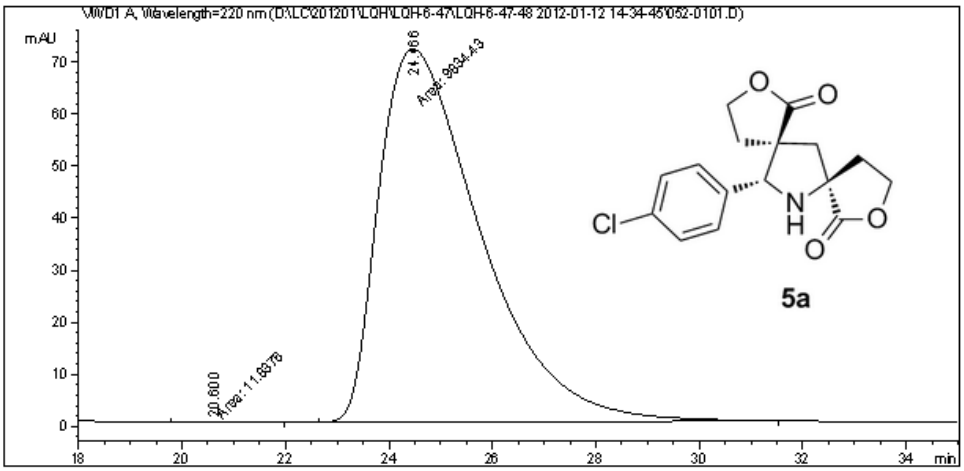
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.154	MF	1.6216	2145.26538	22.04838	50.0272
2	24.573	FM	2.1532	2142.93481	16.58695	49.9728
Totals :				4288.20020	38.63533	

*** End of Report ***

Instrument 1 2/28/2012 1:47:59 PM LQH

Data File D:\LC\201201\LQH\LQH-6-47\LQH-6-47-48 2012-01-12 14-34-45\052-0101.D
Sample Name: LQH-6-48

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 52
Injection Date	: 1/12/2012 2:36:10 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-47-48 2012-01-12 14-34-45\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-47-48 2012-01-12 14-34-45\052-0101.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 2/28/2012 1:50:53 PM by LQH (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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	20.600	MM	1.3168	11.83757	1.49823e-1	0.1202
2	24.466	MM	2.2858	9834.43359	71.70798	99.8798
Totals :				9846.27117	71.85781	

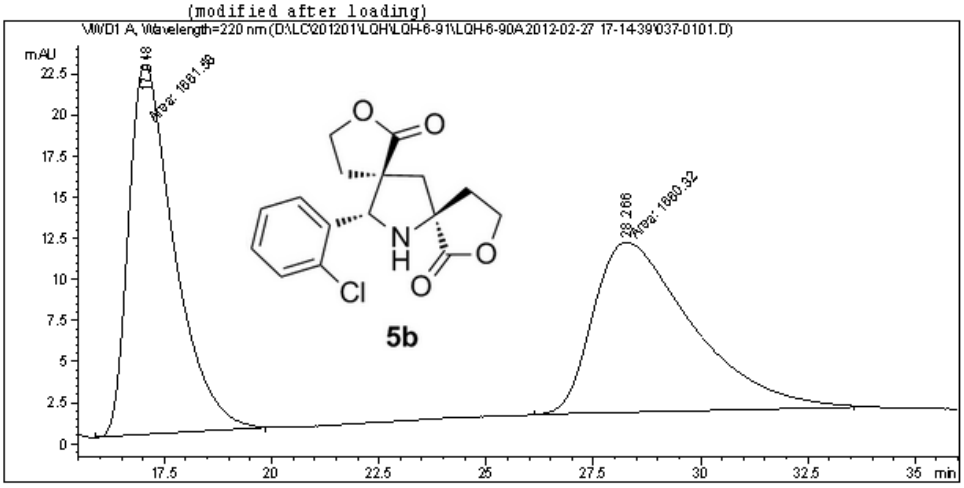
*** End of Report ***

Instrument 1 2/28/2012 1:50:58 PM LQH

Page 1 of 1

Data File D:\LC\201201\LQH\LQH-6-91\LQH-6-90A 2012-02-27 17-14-39\037-0101.D
Sample Name: LQH-6-90A

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 37
Injection Date	: 2/27/2012 5:16:17 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-91\LQH-6-90A 2012-02-27 17-14-39\ASH-50-50-10ML-220NM.M		
Last changed	: 12/6/2011 11:03:10 AM by TMC		
Analysis Method	: D:\LC\201201\LQH\LQH-6-91\LQH-6-90A 2012-02-27 17-14-39\037-0101.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/9/2012 11:17:25 AM 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

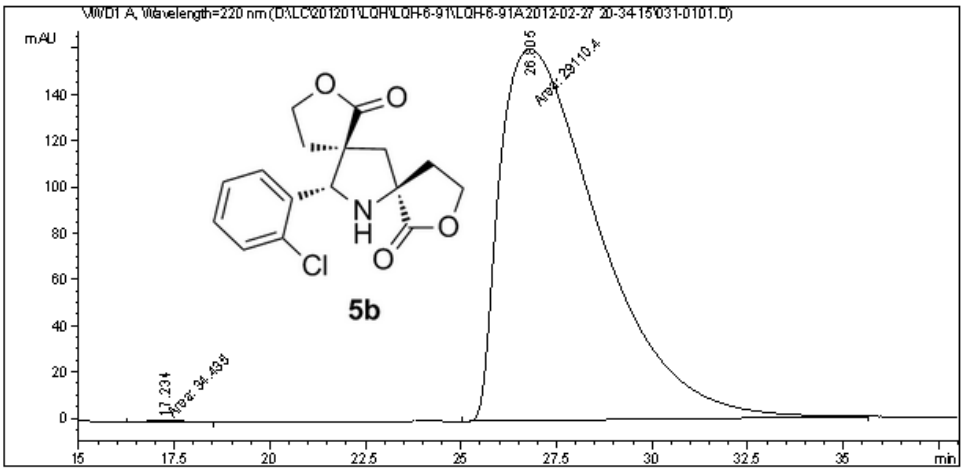
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.048	MM	1.2304	1661.57849	22.50783	50.0190
2	28.266	MM	2.6761	1660.31567	10.34035	49.9810
Totals :				3321.89417	32.84818	

*** End of Report ***

Instrument 1 3/9/2012 11:17:30 AM FX

Data File D:\LC\201201\LQH\LQH-6-91\LQH-6-91A 2012-02-27 20-34-15\031-0101.D
Sample Name: LQH-6-91A

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 2/27/2012 8:35:40 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-91\LQH-6-91A 2012-02-27 20-34-15\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\201201\LQH\LQH-6-91\LQH-6-91A 2012-02-27 20-34-15\031-0101.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 3/9/2012 11:19:39 AM by FX (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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

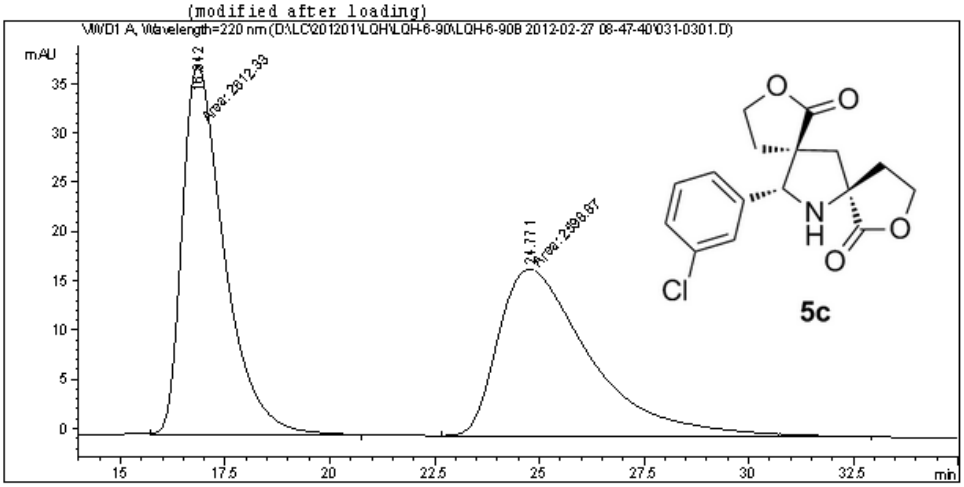
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.234	MM	1.0958	34.43504	5.23737e-1	0.1182
2	26.805	MM	3.0200	2.91104e4	160.65382	99.8818
Totals :				2.91448e4	161.17756	

*** End of Report ***

Instrument 1 3/9/2012 11:19:47 AM FX

Data File D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\031-0301.D
Sample Name: LQH-6-90B

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 2/27/2012 9:37:15 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\ASH-50-50-10ML-220NM.M		
Last changed	: 2/27/2012 10:20:41 AM by LQH (modified after loading)		
Analysis Method	: D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\031-0301.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/9/2012 11:15:17 AM 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	16.842	FM	1.1610	2612.32788	37.50181	50.1291
2	24.771	MM	2.5608	2598.86987	16.91425	49.8709
Totals :				5211.19775	54.41606	

*** End of Report ***

Instrument 1 3/9/2012 11:15:22 AM FX

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-10-73\LQH-10-73 2013-03-19 10-47-46\095-0201.D
Sample Name: LQH-10-73

```
======================================================================
Acq. Operator   : TL                              Seq. Line :   2
Acq. Instrument : Instrument 1                    Location : Vial 95
Injection Date  : 3/19/2013 11:00:30 AM              Inj :   1
                                                  Inj Volume : 5 µl
Acq. Method     : D:\LC\DATA\LQH\LQH-10-73\LQH-10-73 2013-03-19 10-47-46\ASH-50-50-10ML-
                  220NM-60MIN.M
Last changed    : 12/6/2011 9:55:58 PM by TMC
Analysis Method : D:\LC\DATA\LQH\LQH-10-73\LQH-10-73 2013-03-19 10-47-46\095-0201.D\DA.M (
                  ASH-50-50-10ML-220NM-60MIN.M)
Last changed    : 4/27/2013 11:14:30 AM by LFL
                  (modified after loading)
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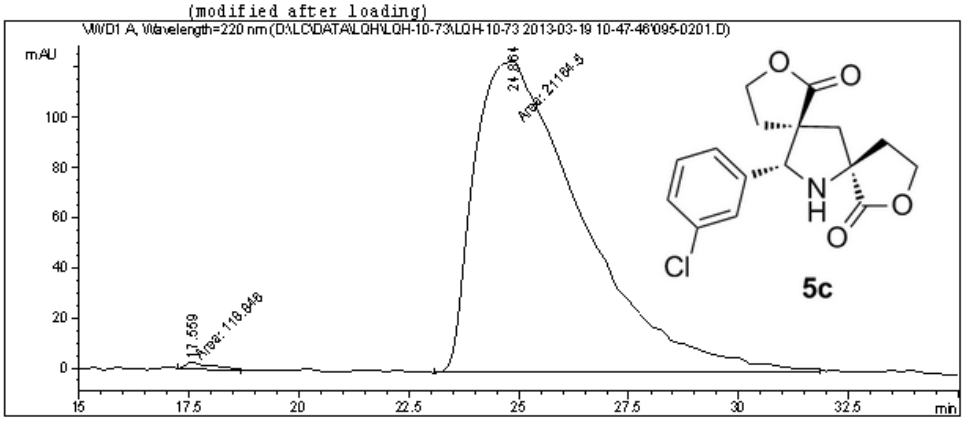
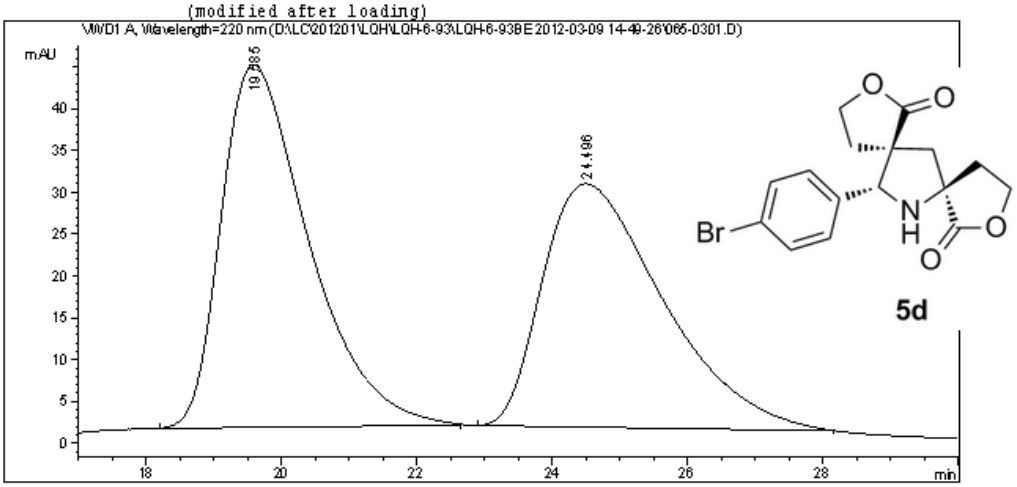



```
======================================================================
                        Area Percent Report
======================================================================

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  17.559 MM    0.7646  118.84649    2.59046   0.5584
   2  24.864 MM    2.8497 2.11645e4   123.78165  99.4416

Totals :                  2.12834e4   126.37211


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

Data File D:\LC\201201\LQH\LQH-6-93\LQH-6-93BE 2012-03-09 14-49-26\065-0301.D
Sample Name: LQH-6-93E

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 65
Injection Date	: 3/9/2012 3:35:13 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-93\LQH-6-93BE 2012-03-09 14-49-26\ASH-50-50-10ML-220NM.M		
Last changed	: 3/9/2012 3:33:52 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201201\LQH\LQH-6-93\LQH-6-93BE 2012-03-09 14-49-26\065-0301.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/12/2012 9:39:45 AM 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

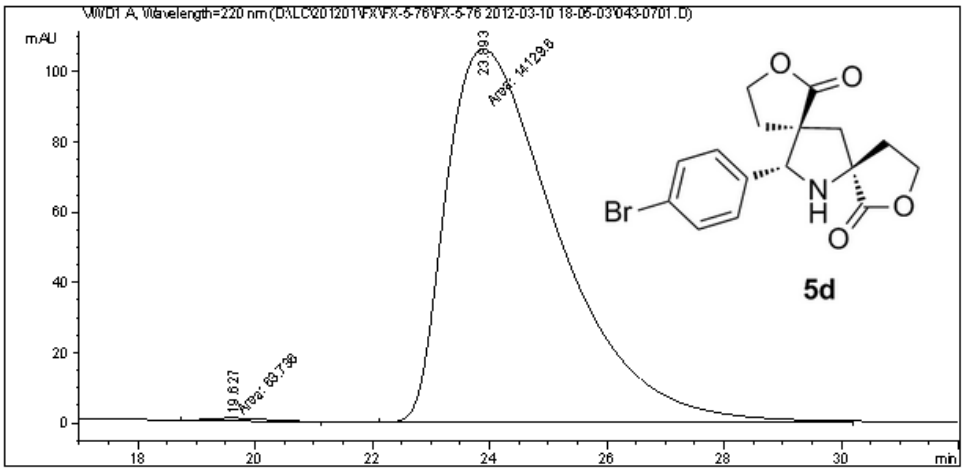
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.585	BB	1.3108	3902.00293	43.34914	51.9619
2	24.496	BB	1.6405	3607.35547	29.08884	48.0381
Totals :				7509.35840	72.43799	

*** End of Report ***

Instrument 1 3/12/2012 9:39:51 AM FX Page 1 of 1

Data File D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\043-0701.D
Sample Name: LQH-6-94E

Acq. Operator	: FX	Seq. Line	: 7
Acq. Instrument	: Instrument 1	Location	: Vial 43
Injection Date	: 3/10/2012 9:35:26 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\043-0701.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 3/12/2012 9:43:14 AM by FX (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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	19.627	MM	1.2066	63.73604	8.80380e-1	0.4491
2	23.893	MM	2.2139	1.41296e4	106.36858	99.5509
Totals :				1.41933e4	107.24896	

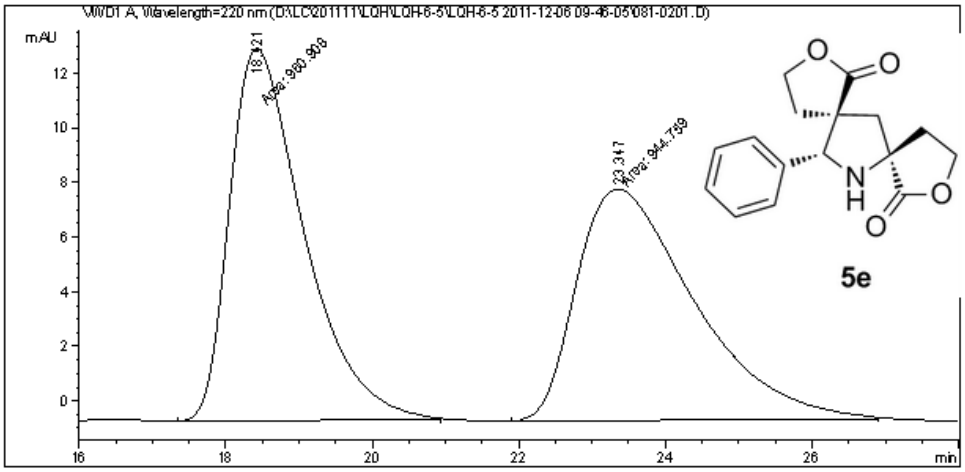
*** End of Report ***

Instrument 1 3/12/2012 9:43:19 AM FX

Page 1 of 1

Data File D:\LC\201111\LQH\LQH-6-5\LQH-6-5 2011-12-06 09-46-05\081-0201.D
Sample Name: LQH-6-5C

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 81
Injection Date	: 12/6/2011 9:58:27 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\LQH\LQH-6-5\LQH-6-5 2011-12-06 09-46-05\ASH-50-50-10ML-220NM.M		
Last changed	: 12/6/2011 10:28:19 AM by LQH (modified after loading)		
Analysis Method	: D:\LC\201111\LQH\LQH-6-5\LQH-6-5 2011-12-06 09-46-05\081-0201.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 1/2/2012 8:26:38 PM by thl (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

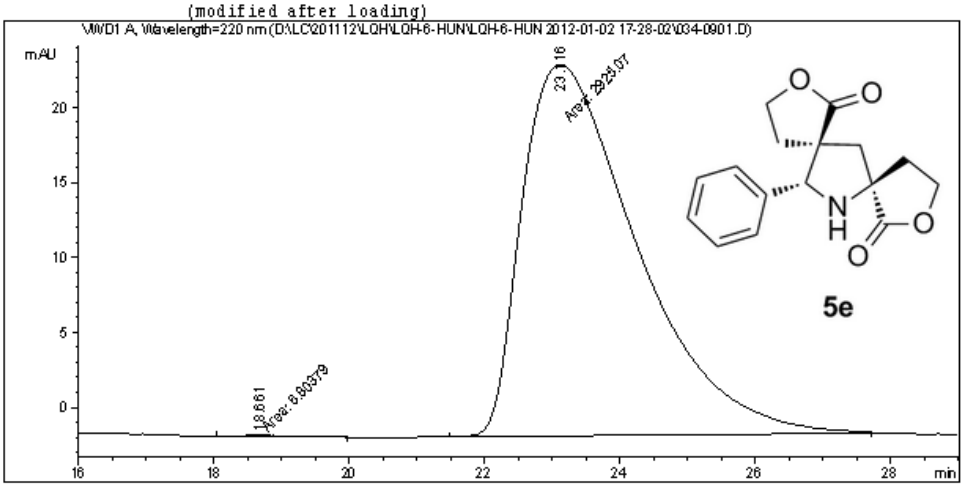
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.421	MM	1.1746	960.90796	13.63483	50.4237
2	23.347	MM	1.8567	944.75897	8.48041	49.5763
Totals :				1905.66693	22.11525	

Data File D:\LC\201112\LQH\LQH-6-HUN\LQH-6-HUN 2012-01-02 17-28-02\034-0901.D
Sample Name: LQH-6-43

Acq. Operator	: LQH	Seq. Line	: 9
Acq. Instrument	: Instrument 1	Location	: Vial 34
Injection Date	: 1/2/2012 7:50:15 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201112\LQH\LQH-6-HUN\LQH-6-HUN 2012-01-02 17-28-02\ASH-50-50-10ML-220NM-30MIN.M		
Last changed	: 1/2/2012 5:39:36 PM by thl		
Analysis Method	: D:\LC\201112\LQH\LQH-6-HUN\LQH-6-HUN 2012-01-02 17-28-02\034-0901.D\DA.M (ASH-50-50-10ML-220NM-30MIN.M)		
Last changed	: 1/2/2012 8:23:40 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	18.661	MM	1.1034	6.80379	1.02772e-1	0.2321
2	23.116	MM	1.9676	2925.06689	24.77680	99.7679
Totals :				2931.87069	24.87957	

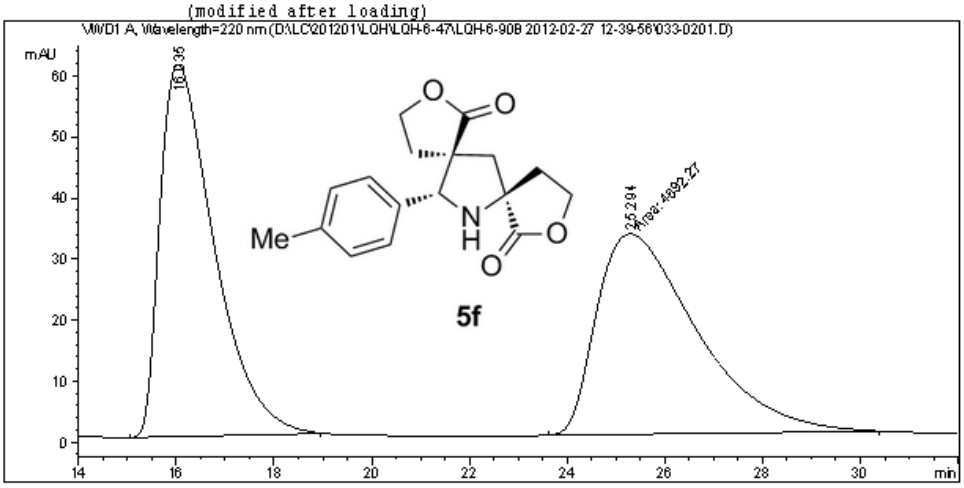
*** End of Report ***

Instrument 1 1/2/2012 8:23:46 PM thl

Page 1 of 1

Data File D:\LC\201201\LQH\LQH-6-47\LQH-6-90B 2012-02-27 12-39-56\033-0201.D
Sample Name: LQH-6-47C

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 33
Injection Date	: 2/27/2012 12:52:36 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-90B 2012-02-27 12-39-56\ASH-50-50-10ML-220NM.M		
Last changed	: 12/6/2011 11:03:10 AM by TMC		
Analysis Method	: D:\LC\201201\LQH\LQH-6-47\LQH-6-90B 2012-02-27 12-39-56\033-0201.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 2/28/2012 1:52:21 PM by LQH (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	16.035	BB	1.1492	4697.03076	60.97140	50.0254
2	25.294	MM	2.3817	4692.26660	32.83550	49.9746
Totals :				9389.29736	93.80690	

*** End of Report ***

Instrument 1 2/28/2012 1:52:26 PM LQH

Page 1 of 1

Data File D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\035-0201.D
Sample Name: LQH-6-48C

```
=====================================================================
Acq. Operator   : LQH                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 35
Injection Date  : 2/27/2012 4:03:25 PM               Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\ASH-50-50-10ML-
                  220NM-40MIN.M
Last changed    : 12/2/2011 2:34:27 PM by HZL
Analysis Method : D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\035-0201.D\DA.M (
                  ASH-50-50-10ML-220NM-40MIN.M)
Last changed    : 3/9/2012 11:34:56 AM by FX
                  (modified after loading)
Method Info     : ASH-50-50-1ML-254NM-50MIN
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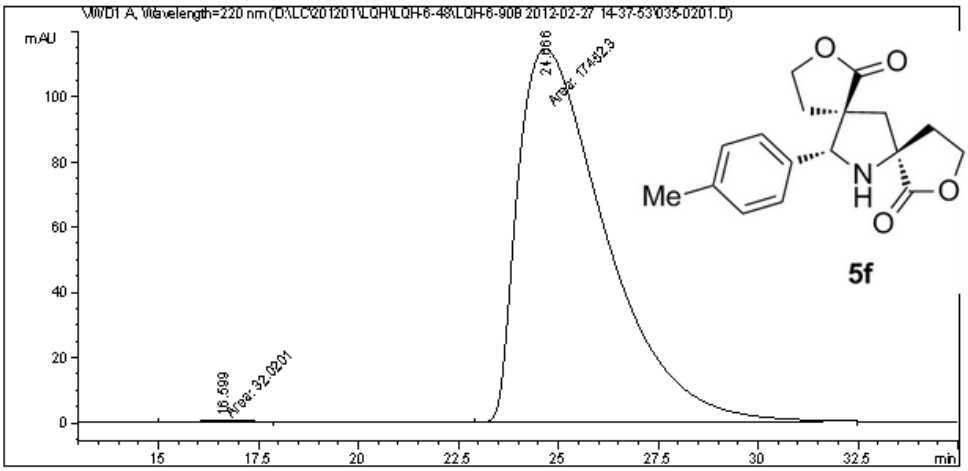


```
=====================================================================
                        Area Percent Report
=====================================================================

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  16.599 MM    1.3422   32.02005 3.97597e-1   0.1831
   2  24.666 MM    2.5451 1.74523e4   114.28714  99.8169

Totals :                  1.74843e4   114.68474


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

Instrument 1 3/9/2012 11:35:02 AM FX                    Page 1 of 1
```

Data File D:\LC\201201\LQH\LQH-6-93\LQH-6-93C 2012-02-28 09-19-17\002-0201.D
Sample Name: LQH-6-93A

```
==================================================================
Acq. Operator   : LQH                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 2
Injection Date  : 2/28/2012 10:00:47 AM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201201\LQH\LQH-6-93\LQH-6-93C 2012-02-28 09-19-17\ASH-50-50-10ML-
                  220NM.M
Last changed    : 2/28/2012 10:27:02 AM by LQH
                  (modified after loading)
Analysis Method : D:\LC\201201\LQH\LQH-6-93\LQH-6-93C 2012-02-28 09-19-17\002-0201.D\DA.M (
                  ASH-50-50-10ML-220NM.M)
Last changed    : 3/9/2012 11:24:19 AM by FX
                  (modified after loading)
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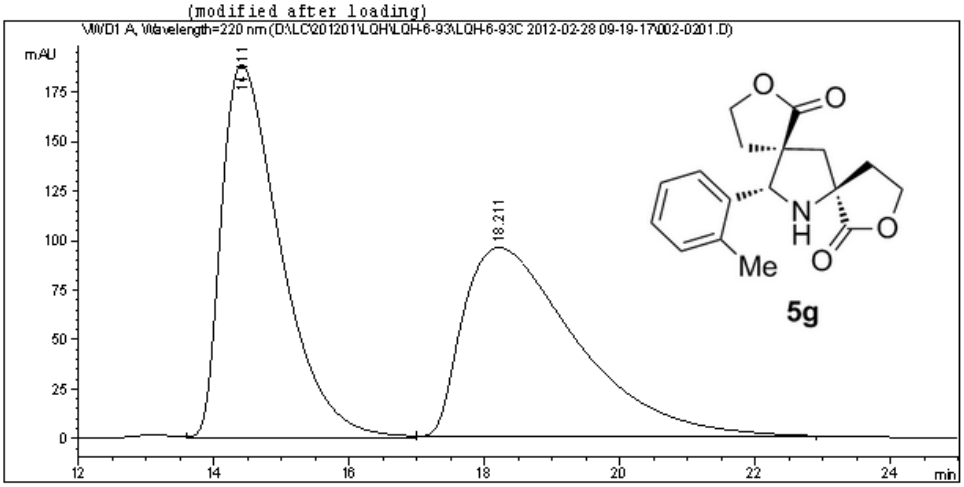
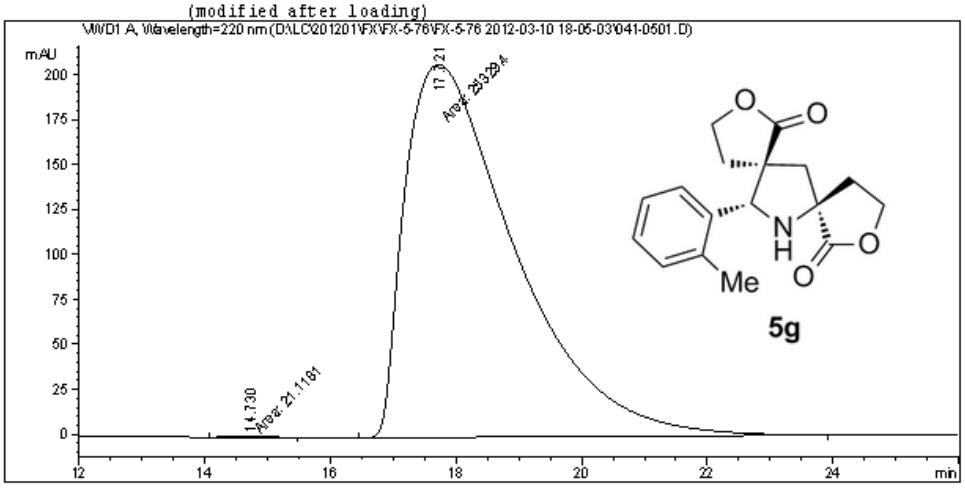



```
==================================================================
                        Area Percent Report
==================================================================

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  14.411 VB    0.9306 1.14309e4   188.41473  50.4420
   2  18.211 BB    1.7569 1.12306e4    95.76410  49.5580

Totals :                  2.26614e4   284.17883


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

Instrument 1 3/9/2012 11:24:25 AM FX Page 1 of 1

Data File D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\041-0501.D
Sample Name: LQH-6-94A

Acq. Operator	: FX	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 41
Injection Date	: 3/10/2012 8:22:21 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\ASH-50-50-10ML-220NM-30MIN.M		
Last changed	: 1/2/2012 5:39:36 PM by thl		
Analysis Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\041-0501.D\DA.M (ASH-50-50-10ML-220NM-30MIN.M)		
Last changed	: 3/12/2012 9:34:50 AM by FX (modified after loading)		



Area Percent Report

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

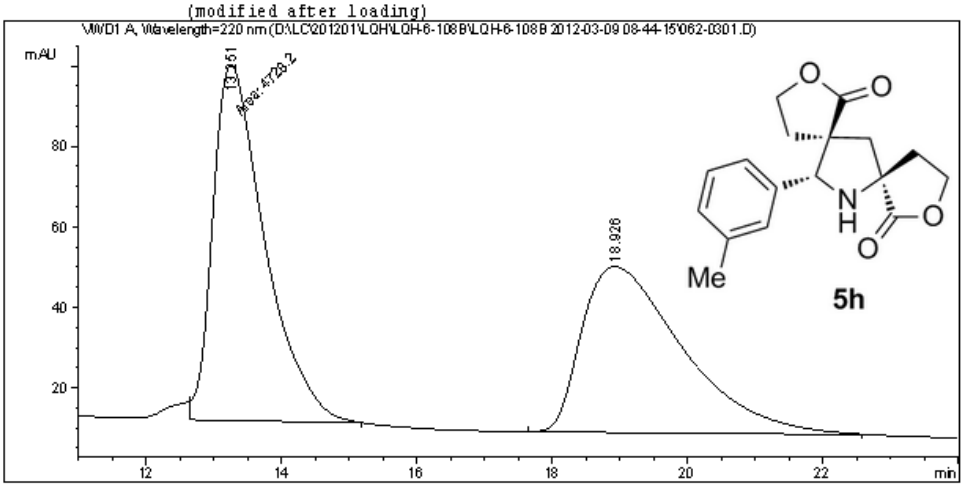
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.730	MM	0.7914	21.11814	4.44722e-1	0.0833
2	17.721	MM	2.0359	2.53294e4	207.35204	99.9167
Totals :				2.53505e4	207.79676	

*** End of Report ***

Data File D:\LC\201201\LQH\LQH-6-108B\LQH-6-108B 2012-03-09 08-44-15\062-0301.D
Sample Name: LQH-6-106A

Acq. Operator	: lqh	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 62
Injection Date	: 3/9/2012 9:51:13 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-108B\LQH-6-108B 2012-03-09 08-44-15\ASH-50-50-10ML-220NM.M		
Last changed	: 12/6/2011 11:03:10 AM by TMC		
Analysis Method	: D:\LC\201201\LQH\LQH-6-108B\LQH-6-108B 2012-03-09 08-44-15\062-0301.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/9/2012 11:38:06 AM by FX (modified after loading)		



Area Percent Report

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

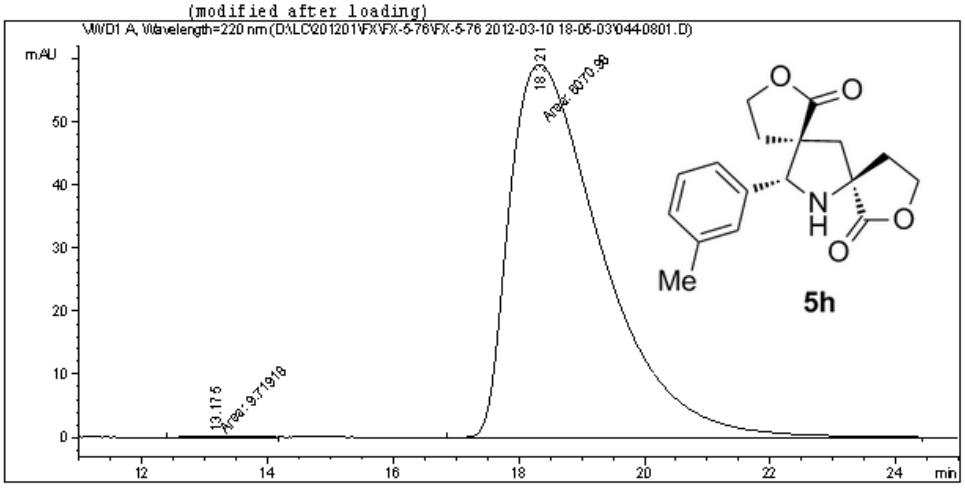
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.251	MM	0.8895	4728.19580	88.59309	52.5801
2	18.926	BB	1.5145	4264.17236	41.20830	47.4199
Totals :				8992.36816	129.80140	

*** End of Report ***

Data File D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\044-0801.D
Sample Name: LQH-6-111

Acq. Operator	: FX	Seq. Line	: 8
Acq. Instrument	: Instrument 1	Location	: Vial 44
Injection Date	: 3/10/2012 10:16:50 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\ASH-50-50-10ML-220NM-30MIN.M		
Last changed	: 1/2/2012 5:39:36 PM by thl		
Analysis Method	: D:\LC\201201\FX\FX-5-76\FX-5-76 2012-03-10 18-05-03\044-0801.D\DA.M (ASH-50-50-10ML-220NM-30MIN.M)		
Last changed	: 3/12/2012 9:44:56 AM 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	13.175	MM	0.8521	9.71918	1.90107e-1	0.1598
2	18.321	MM	1.7092	6070.97803	59.20070	99.8402
Totals :				6080.69720	59.39081	

*** End of Report ***

Instrument 1 3/12/2012 9:45:01 AM FX

Data File D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\032-0401.D
Sample Name: LQH-6-47B

```
=====================================================================
Acq. Operator   : LQH                          Seq. Line :   4
Acq. Instrument : Instrument 1                  Location : Vial 32
Injection Date  : 2/27/2012 10:22:16 AM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\ASH-50-50-10ML-
                  220NM.M
Last changed    : 2/27/2012 10:21:08 AM by LQH
                  (modified after loading)
Analysis Method : D:\LC\201201\LQH\LQH-6-90\LQH-6-90B 2012-02-27 08-47-40\032-0401.D\DA.M (
                  ASH-50-50-10ML-220NM.M)
Last changed    : 3/9/2012 11:08:16 AM by FX
                  (modified after loading)
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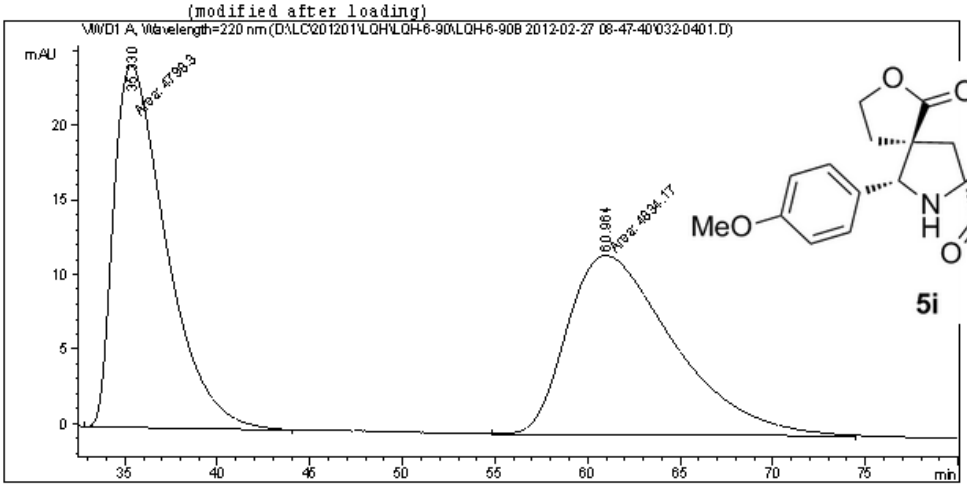



```
=====================================================================
                        Area Percent Report
=====================================================================

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  35.330 MM    3.2880 4798.29834   24.32262  49.8138
   2  60.964 MM    6.7069 4834.17285   12.01297  50.1862

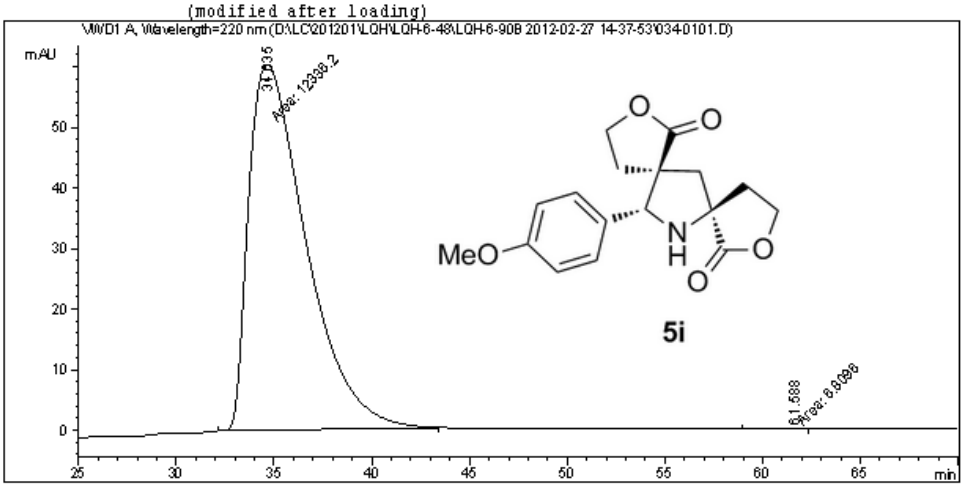
Totals :                  9632.47119   36.33559


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

Instrument 1 3/9/2012 11:08:22 AM FX Page 1 of 1

Data File D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\034-0101.D
Sample Name: LQH-6-48B

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 34
Injection Date	: 2/27/2012 2:39:24 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\ASH-50-50-10ML-220NM.M		
Last changed	: 2/27/2012 4:01:25 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201201\LQH\LQH-6-48\LQH-6-90B 2012-02-27 14-37-53\034-0101.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/9/2012 11:32:06 AM 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

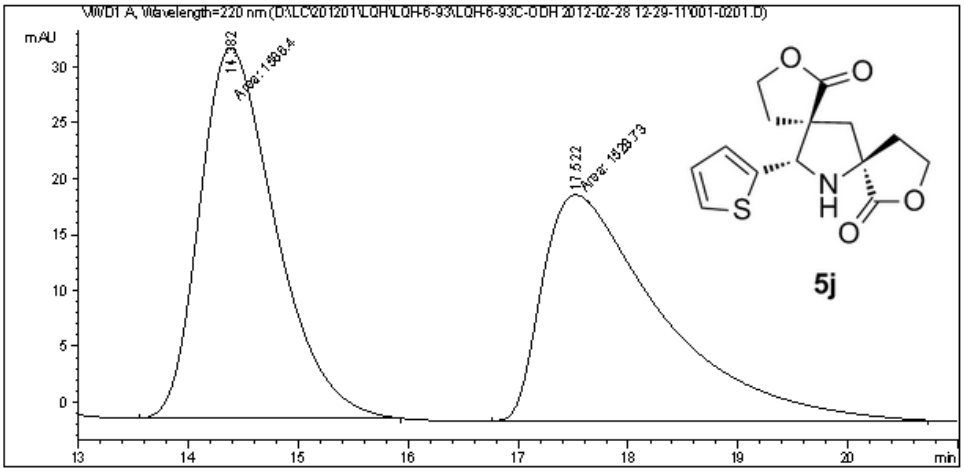
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	34.635	MM	3.4068	1.23362e4	60.35048	99.9448
2	61.588	MM	1.9781	6.80960	5.73759e-2	0.0552
Totals :				1.23430e4	60.40785	

*** End of Report ***

Instrument 1 3/9/2012 11:33:04 AM FX

Data File D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\001-0201.D
Sample Name: LQH-6-93C

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 1
Injection Date	: 2/28/2012 12:41:32 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\ODH-40-60-1ML-220NM.M		
Last changed	: 2/28/2012 1:18:03 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\001-0201.D\DA.M (ODH-40-60-1ML-220NM.M)		
Last changed	: 3/14/2012 9:22:47 PM by FX (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



Area Percent Report

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

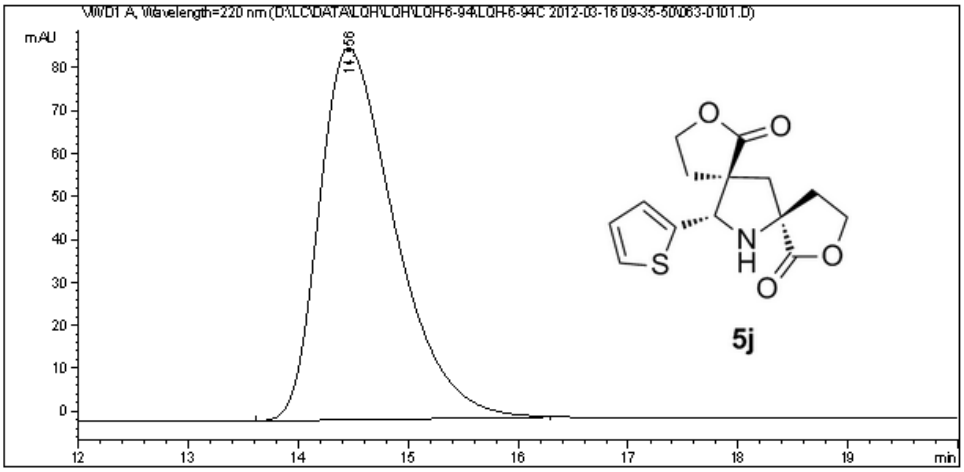
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.382	MM	0.7990	1586.40454	33.09176	50.9256
2	17.522	MM	1.2582	1528.73437	20.25014	49.0744
Totals :				3115.13892	53.34190	

Instrument 1 3/14/2012 9:22:53 PM FX

Data File D:\LC\DATA\LQH\LQH\LQH-6-94\LQH-6-94C 2012-03-16 09-35-50\063-0101.D
Sample Name: LQH-6-94C

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 63
Injection Date	: 3/16/2012 9:37:19 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-94\LQH-6-94C 2012-03-16 09-35-50\ODH-40-60-1ML-220NM.M		
Last changed	: 3/16/2012 9:58:38 AM by LQH (modified after loading)		
Analysis Method	: D:\LC\DATA\LQH\LQH\LQH-6-94\LQH-6-94C 2012-03-16 09-35-50\063-0101.D\DA.M (ODH-40-60-1ML-220NM.M)		
Last changed	: 5/5/2012 7:04:42 PM by lqh (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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	14.456	BB	0.7357	4144.70801	86.42880	100.0000
Totals :				4144.70801	86.42880	

*** End of Report ***

Instrument 1 5/5/2012 7:04:49 PM lqh

Data File D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\003-0301.D
Sample Name: LQH-6-93D

```
==================================================================
Acq. Operator   : LQH                          Seq. Line :   3
Acq. Instrument : Instrument 1                  Location : Vial 3
Injection Date  : 2/28/2012 1:19:47 PM               Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\ASH-50-50-
                  10ML-220NM.M
Last changed    : 2/28/2012 1:53:43 PM by LQH
                  (modified after loading)
Analysis Method : D:\LC\201201\LQH\LQH-6-93\LQH-6-93C-ODH 2012-02-28 12-29-11\003-0301.D\DA.
                  M (ASH-50-50-10ML-220NM.M)
Last changed    : 3/9/2012 11:28:14 AM by FX
                  (modified after loading)
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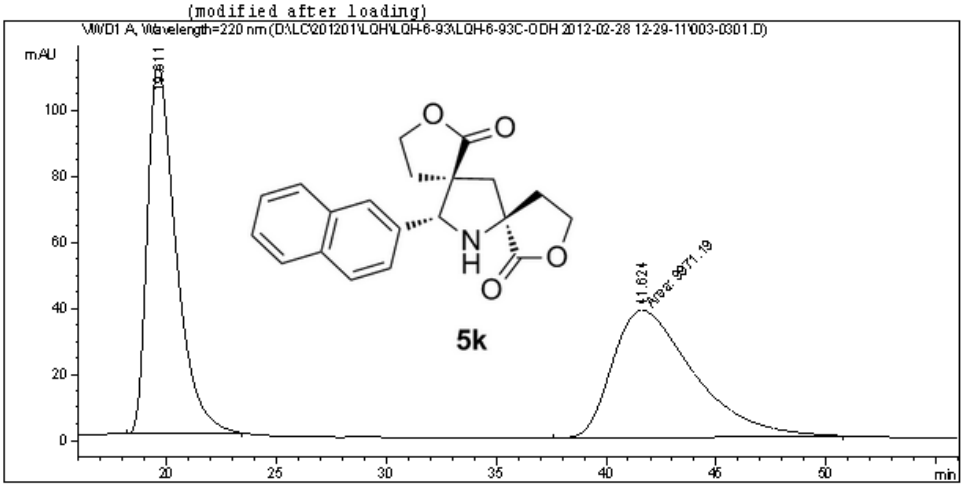
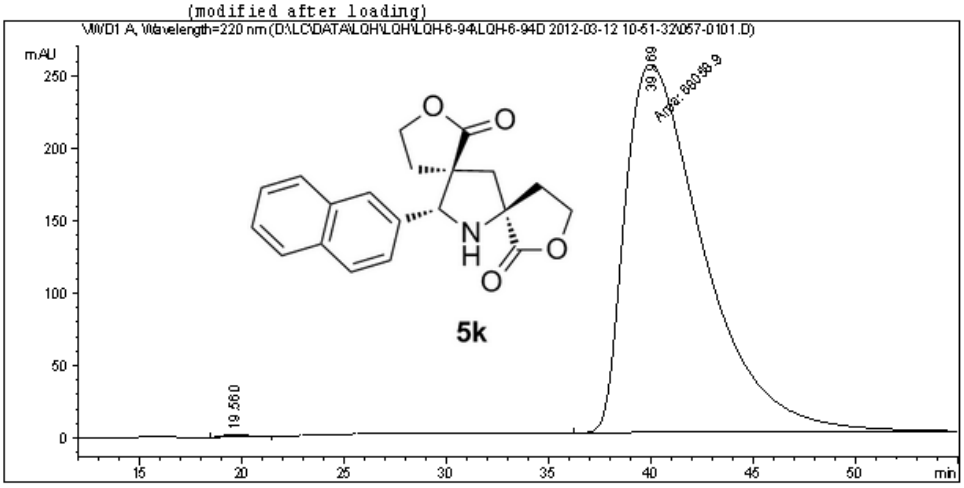



```
==================================================================
                        Area Percent Report
==================================================================

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  19.611 BB    1.3423 1.01271e4   111.79090  50.3879
   2  41.624 MM    4.3220 9971.19336   38.45141  49.6121

Totals :                  2.00983e4   150.24231


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

Data File D:\LC\DATA\LQH\LQH\LQH-6-94\LQH-6-94D 2012-03-12 10-51-32\057-0101.D
Sample Name: LQH-6-94D

Acq. Operator	: lqh	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 57
Injection Date	: 3/12/2012 10:53:09 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-6-94\LQH-6-94D 2012-03-12 10-51-32\ASH-50-50-10ML-220NM-60MIN.M		
Last changed	: 12/6/2011 9:55:58 PM by TMC		
Analysis Method	: D:\LC\DATA\LQH\LQH\LQH-6-94\LQH-6-94D 2012-03-12 10-51-32\057-0101.D\DA.M (ASH-50-50-10ML-220NM-60MIN.M)		
Last changed	: 5/5/2012 7:09:28 PM by lqh (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	19.560	BB	0.8952	142.06021	1.86954	0.2083
2	39.969	MM	4.4642	6.80589e4	254.09404	99.7917
Totals :				6.82010e4	255.96358	

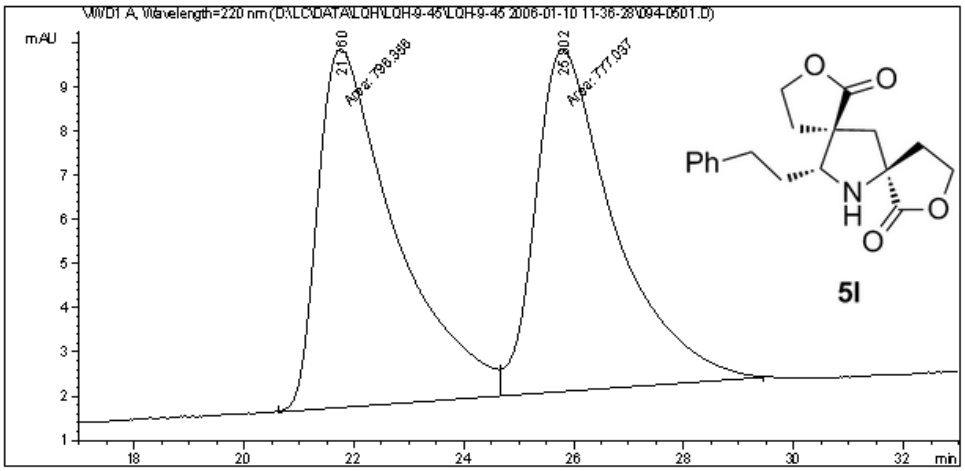
*** End of Report ***

Instrument 1 5/5/2012 7:09:33 PM lqh

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\094-0501.D
Sample Name: LQH-9-45

Acq. Operator	: THL	Seq. Line	: 5
Acq. Instrument	: Instrument 1	Location	: Vial 94
Injection Date	: 1/10/2006 1:20:15 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\094-0501.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 3/16/2013 4:25:49 PM by FX (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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

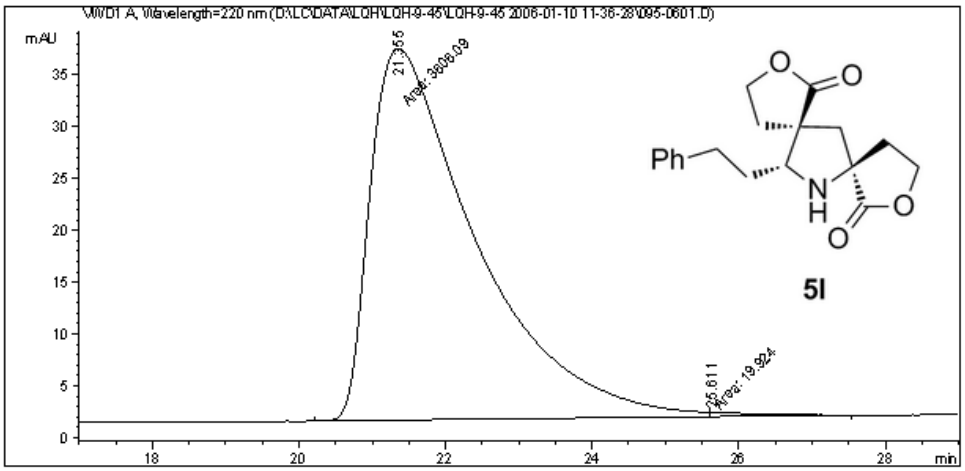
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.760	MF	1.6355	796.35645	8.11544	50.6139
2	25.802	FM	1.6738	777.03687	7.73732	49.3861
Totals :				1573.39331	15.85276	

*** End of Report ***

Instrument 1 3/16/2013 4:26:26 PM FX

Data File D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\095-0601.D
Sample Name: LQH-9-46

Acq. Operator	: THL	Seq. Line	: 6
Acq. Instrument	: Instrument 1	Location	: Vial 95
Injection Date	: 1/10/2006 2:01:31 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\ASH-50-50-10ML-220NM-40MIN.M		
Last changed	: 12/2/2011 2:34:27 PM by HZL		
Analysis Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\095-0601.D\DA.M (ASH-50-50-10ML-220NM-40MIN.M)		
Last changed	: 3/16/2013 4:28:36 PM by FX (modified after loading)		
Method Info	: ASH-50-50-1ML-254NM-50MIN		



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

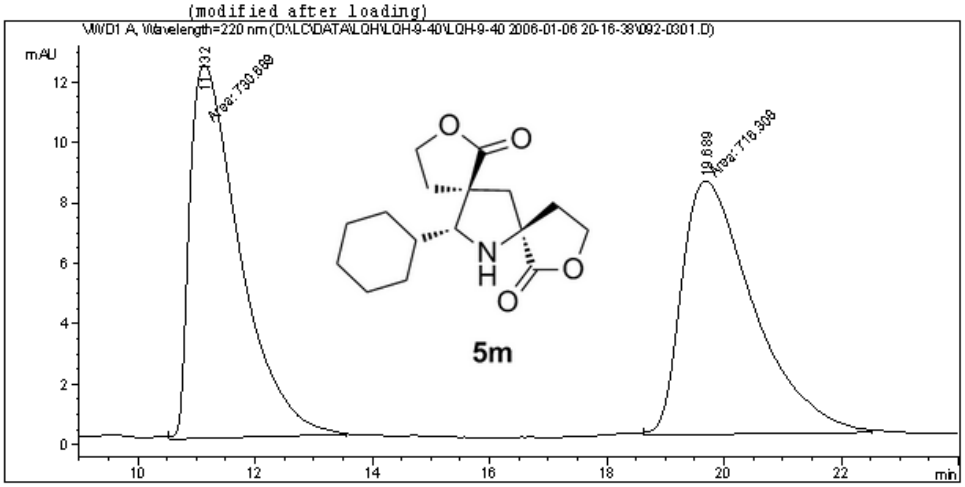
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.355	MF	1.6786	3606.08813	35.80420	99.4505
2	25.611	FM	0.7911	19.92399	4.19765e-1	0.5495
Totals :				3626.01212	36.22396	

*** End of Report ***

Instrument 1 3/16/2013 4:29:06 PM FX

Data File D:\LC\DATA\LQH\LQH-9-40\LQH-9-40 2006-01-06 20-16-38\092-0301.D
Sample Name: LQH-9-40

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 92
Injection Date	: 1/6/2006 8:58:01 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-9-40\LQH-9-40 2006-01-06 20-16-38\ASH-50-50-10ML-220NM.M		
Last changed	: 1/6/2006 8:56:37 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\DATA\LQH\LQH-9-40\LQH-9-40 2006-01-06 20-16-38\092-0301.D\DA.M (ASH-50-50-10ML-220NM.M)		
Last changed	: 3/16/2013 4:19:15 PM by FX (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

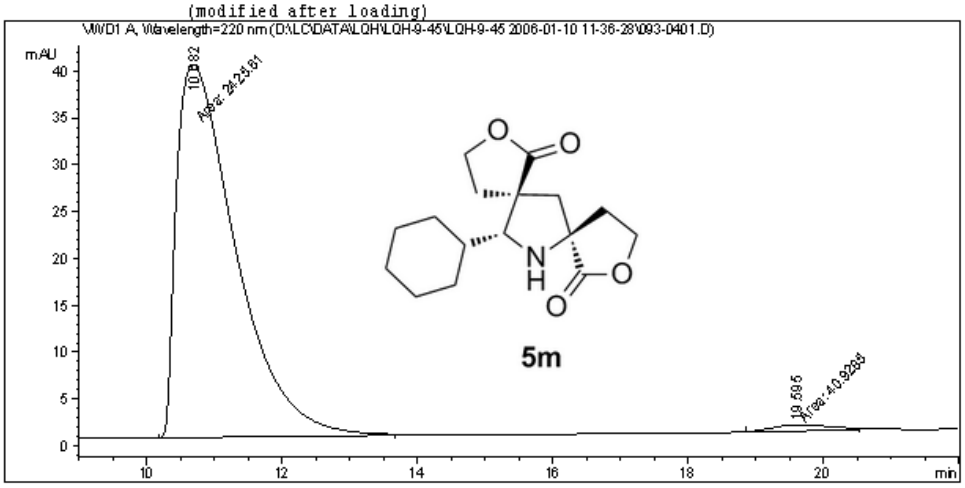
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.132	MM	0.9845	730.68909	12.37048	50.4969
2	19.689	MM	1.4211	716.30835	8.40070	49.5031
Totals :				1446.99744	20.77118	

*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\093-0401.D
Sample Name: LQH-9-40

Acq. Operator	: THL	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 93
Injection Date	: 1/10/2006 12:53:34 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\ASH-50-50-1ML-220NM.M		
Last changed	: 1/10/2006 12:52:17 PM by THL (modified after loading)		
Analysis Method	: D:\LC\DATA\LQH\LQH-9-45\LQH-9-45 2006-01-10 11-36-28\093-0401.D\DA.M (ASH-50-50-1ML-220NM.M)		
Last changed	: 3/16/2013 4:24:15 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	10.682	MF	1.0141	2425.61426	39.86407	98.3407
2	19.595	MM	1.0105	40.92847	6.75068e-1	1.6593
Totals :				2466.54273	40.53914	

*** End of Report ***