

Supporting Information for

**Asymmetric Construction of Fluorinated Imidazolidines via
Cu(I)-Catalyzed *exo'*-Selective 1,3-Dipolar Cycloaddition of
Azomethine Ylides with Fluorinated Imines**

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I. General Remarks

^1H NMR spectra were recorded on a VARIAN Mercury 300 MHz or Bruker 400 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 100 MHz or 75 MHz spectrometer in CDCl_3 or DMSO-d_6 . 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 ^1H 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, or determined by GC using β -dex 325 column. Chiral ligand (*S,R*_p)-PPFOMe and Fluorinated imines was prepared according to the literature procedure.^{1,2} The racemic adducts were obtained by using AgOAc/ PPh_3 as the catalyst. The absolute configuration of (2*R*,4*R*,5*R*)-**3s** was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.

II. Ligand Screening for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with Fluorinated Imines

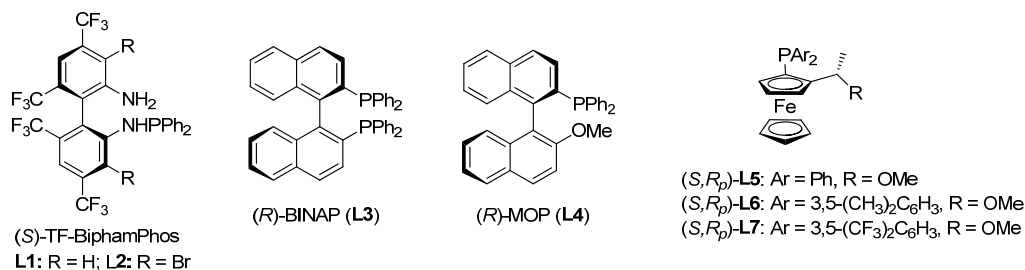
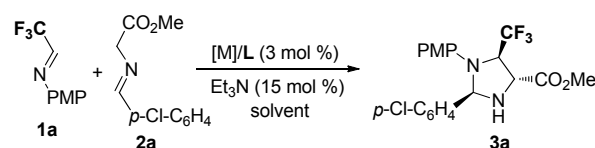


Figure 1. Screened chiral ligands.

Table 1. Optimization for catalytic asymmetric 1,3-dipolar cycloaddition of imino ester **2a** with trifluoromethylated imine **1a**^a



Entry	L	[M] ^b	Solvent	Temp. (°C)	Time (min)	yield ^c (%)	dr ^d	ee ^e (%)
1	L1	AgOAc	DCM	rt	10	85	93:7	23
2	L1	CuBF ₄	DCM	rt	10	88	91:9	35
3	L2	AgOAc	DCM	rt	10	80	83:17	33
4	L2	CuBF ₄	DCM	rt	10	82	62:38	71
5	L3	AgOAc	DCM	rt	60	75	86:14	16
6	L3	CuBF ₄	DCM	rt	60	85	39:61	80
7	L4	AgOAc	DCM	rt	10	86	98:2	55
8	L4	CuBF ₄	DCM	rt	10	88	98:2	55
9	L5	AgOAc	DCM	rt	10	90	98:2	77
10	L5	CuBF ₄	DCM	rt	10	95	85:15	91
11 ^f	L5	CuBF ₄	DCM	rt	10	95	87:13	90
12	L6	CuBF ₄	DCM	rt	10	92	83:17	93
13	L7	CuBF ₄	DCM	rt	10	94	65:32	72
14	L5	CuBF ₄	PhMe	rt	10	90	94:6	93
15	L5	CuBF ₄	THF	rt	10	93	95:5	92
16	L5	CuBF ₄	EtOAc	rt	10	93	95:5	93
17	L5	CuBF ₄	MeOH	rt	10	90	88:12	86
18	L5	CuBF ₄	ether	rt	10	94	96:4	94
19	L5	CuBF ₄	MeOH	-20	60	92	98:2	95
20	L5	CuBF ₄	ether	-20	60	93	98:2	97
21 ^g	L5	CuBF ₄	ether	-20	120	90	98:2	97

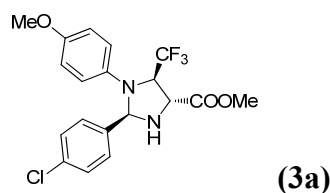
^a All reactions were carried out with 0.35 mmol of **2a** and 0.23 mmol of **1a** in 2 mL of solvent. ^b CuBF₄ = Cu(CH₃CN)₄BF₄. ^c Isolated yield. ^d dr was determined by the crude ¹H NMR and HPLC analysis. ^e ee was determined by chiral HPLC analysis. ^f CuBF₄ is 3 mol % and **L5** is 6.6 mol %. ^g 1 mol % catalyst loading.

III. General Procedure for the Synthesis of Racemic Cycloadducts.

Under argon atmosphere, PPh₃ (6.6 mg, 0.025 mmol) and AgOAc (3.8 mg, 0.023 mmol) were dissolved in 2 mL of DCM, and stirred at room temperature for about 0.5 h. Then, imine substrate (0.35 mmol), Et₃N (0.03 mmol) and fluorinated imines (0.23 mmol) were added sequentially. Once starting material was consumed (monitored by TLC), the organic solvent was removed and the residue was purified by column chromatography to give the cycloaddition product, which was used as the racemic sample for the HPLC analysis.

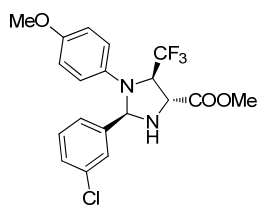
IV. General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides with Fluorinated Imines.

Under argon atmosphere, (*S,R_p*)-PPFOMe (**L5**) (3.3 mg, 0.0077 mmol) and Cu(CH₃CN)₄BF₄ (2.2 mg, 0.007 mmol) were dissolved in 2 mL of ether, and stirred at room temperature for about 0.5 h. After imine substrate (0.35 mmol) was added, the mixture was dropped to -20 °C. Then, fluorinated imines (0.23 mmol) and Et₃N (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.



(2*R*,4*R*,5*R*)-methyl 2-(4-chlorophenyl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

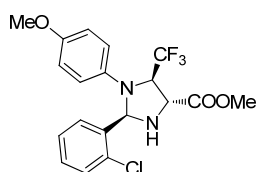
The title compound was prepared according to the general procedure as described above in 95% yield; $[\alpha]_D^{25} = -44.2$ (*c* 0.97, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.49 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 6.76 (d, *J* = 8.4 Hz, 2H), 6.67 (d, *J* = 8.4 Hz, 2H), 5.40 (s, 1H), 4.62 (q, *J* = 6.6 Hz, 1H), 4.32 (s, 1H), 3.79 (s, 3H), 3.72 (s, 3H), 2.66 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 170.4, 154.0, 139.5, 137.5, 134.7, 129.2, 128.1, 125.8 (q, *J* = 280.6 Hz), 117.2, 114.5, 81.2, 65.5 (q, *J* = 30.6 Hz), 60.5, 55.4, 53.0; IR (KBr) ν 3340, 2953, 2845, 1742, 1513, 1450, 1346, 1260, 1175, 1134, 1036, 931, 815, 680, 590 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.49 and 11.47 min.



(3b)

(2R,4R,5R)-methyl 2-(3-chlorophenyl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]_D^{25} = -42.0$ (c 0.29, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.54 (s, 1H), 7.44-7.42 (m, 1H), 7.32-7.31 (m, 2H), 6.77 (d, $J = 9.2$ Hz, 2H), 6.69 (d, $J = 9.2$ Hz, 2H), 5.40 (s, 1H), 4.61 (q, $J = 6.8$ Hz, 1H), 4.31 (s, 1H), 3.79 (s, 3H), 3.72 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.4, 154.1, 141.1, 139.5, 134.8, 130.3, 129.1, 126.9, 125.7 (q, $J = 280.8$ Hz), 124.8, 117.3, 114.6, 81.3, 65.6 (q, $J = 30.6$ Hz), 60.5, 55.4, 53.0; IR (KBr) ν 3318, 2948, 2920, 2815, 1750, 1517, 1395, 1251, 1138, 1036, 950, 759, 590 cm^{-1} . HRMS: calcd. for $\text{C}_{19}\text{H}_{18}\text{ClF}_3\text{N}_2\text{O}_3 + \text{H}^+$: 415.1031, found: 415.1037. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 7.81$ and 11.19 min.

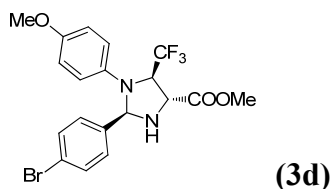


(3c)

(2R,4R,5R)-methyl 2-(2-chlorophenyl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

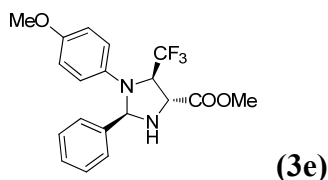
The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]_D^{25} = +15.1$ (c 1.37, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.62-7.60 (m, 1H), 7.45-7.42 (m, 1H), 7.31-7.23 (m, 2H), 6.76 (d, $J = 9.0$ Hz, 2H), 6.63 (d, $J = 9.0$ Hz, 2H), 5.85 (s, 1H), 4.60 (q, $J = 6.9$ Hz, 1H), 4.32 (s, 1H), 3.79 (s, 3H), 3.71 (s, 3H), 2.78 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.6, 153.8, 139.4, 136.3, 133.5, 130.0, 129.9, 127.7, 127.4, 125.7 (q, $J = 280.9$ Hz), 116.6, 114.6, 78.0, 65.4 (q, $J = 30.9$ Hz), 60.4, 55.4, 53.0; IR (KBr) ν 3319, 2910, 2834,

1744, 1510, 1390, 1252, 1157, 1143, 1030, 955, 750, 591 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.58 and 8.47 min.



(2R,4R,5R)-methyl 2-(4-bromophenyl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

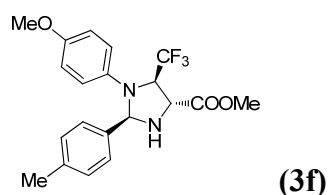
The title compound was prepared according to the general procedure as described above in 94% yield. $[\alpha]_D^{25} = -56.2$ (*c* 0.96, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.51 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.4 Hz, 2H), 6.76 (d, J = 9.2 Hz, 2H), 6.67 (d, J = 9.2 Hz, 2H), 5.39 (s, 1H), 4.61 (q, J = 6.8 Hz, 1H), 4.31 (s, 1H), 3.79 (s, 3H), 3.72 (s, 3H), 2.66 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.4, 154.0, 139.5, 138.0, 132.1, 128.4, 125.7 (q, J = 280.7 Hz), 122.8, 117.2, 114.6, 81.2, 65.6 (q, J = 30.6 Hz), 60.5, 55.4, 53.0; IR (KBr) ν 3340, 2950, 2840, 1714, 1522, 1460, 1251, 1134, 1050, 928, 816, 746, 588 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.01 and 12.77 min.



(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-phenyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

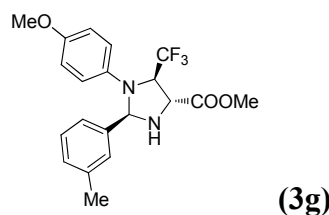
The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]_D^{25} = -35.6$ (*c* 1.35, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.54 (d, J = 7.2 Hz, 2H), 7.40-7.33 (m, 3H), 6.75 (d, J = 9.2 Hz, 2H), 6.69 (d, J =

9.2 Hz, 2H), 5.43 (s, 1H), 4.63 (q, $J = 7.2$ Hz, 1H), 4.32 (s, 1H), 3.78 (s, 3H), 3.71 (s, 3H), 2.69 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 153.7, 139.8, 139.0, 129.0, 128.9, 126.6, 125.9 (q, $J = 280.8$ Hz), 116.9, 114.5, 81.8, 65.5 (q, $J = 30.6$ Hz), 60.6, 55.5, 53.0; IR (KBr) ν 3365, 2950, 2847, 1750, 1514, 1422, 1366, 1240, 1138, 1036, 930, 823, 701, 620 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 7.85$ and 11.61 min.



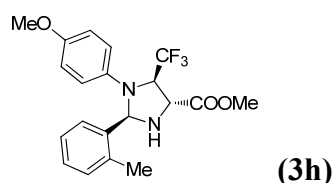
(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-(p-tolyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]_D^{25} = -49.6$ (c 1.37, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.43 (d, $J = 7.6$ Hz, 2H), 7.18 (d, $J = 7.6$ Hz, 2H), 6.75 (d, $J = 9.2$ Hz, 2H), 6.69 (d, $J = 9.2$ Hz, 2H), 5.40 (s, 1H), 4.63 (q, $J = 6.8$ Hz, 1H), 4.31 (s, 1H), 3.78 (s, 3H), 3.71 (s, 3H), 2.69 (brs, 1H), 2.34 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 153.6, 139.9, 138.7, 136.0, 129.6, 126.5, 125.9 (q, $J = 280.9$ Hz), 116.8, 114.5, 81.6, 65.4 (q, $J = 30.5$ Hz), 60.6, 55.4, 52.9, 21.1; IR (KBr) ν 3318, 2930, 2847, 1742, 1513, 1450, 1379, 1138, 1039, 960, 817, 755, 590 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 8.03$ and 9.95 min.



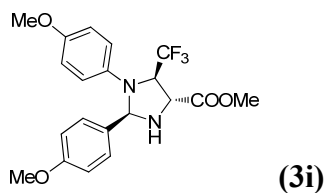
(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-(m-tolyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]_D^{25} = -38.9$ (c 0.85, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.35-7.33 (m, 2H), 7.28-7.24 (m, 1H), 7.16 (d, $J = 7.6$ Hz, 1H), 6.76 (d, $J = 9.2$ Hz, 2H), 6.69 (d, $J = 9.2$ Hz, 2H), 5.38 (s, 1H), 4.64 (q, $J = 6.8$ Hz, 1H), 4.31 (s, 1H), 3.78 (s, 3H), 3.71 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 153.6, 139.9, 138.9, 138.6, 129.6, 128.9, 127.3, 125.9 (q, $J = 283.7$ Hz), 123.5, 116.8, 114.5, 81.8, 65.4 (q, $J = 30.5$ Hz), 60.6, 55.4, 52.9, 21.4; IR (KBr) ν 3345, 2930, 2832, 1747, 1513, 1364, 1217, 1168, 1137, 1040, 961, 812, 756, 588 cm^{-1} . HRMS: calcd. for $\text{C}_{20}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 395.1577, found: 395.1579. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 7.00$ and 10.68 min.



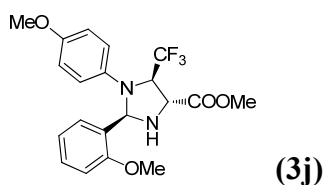
(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-(o-tolyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 82% yield. $[\alpha]_D^{25} = -48.1$ (c 1.35, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.54-7.52 (m, 1H), 7.33-7.23 (m, 2H), 7.18-7.17 (m, 1H), 6.75 (d, $J = 9.2$ Hz, 2H), 6.59 (d, $J = 9.2$ Hz, 2H), 5.59 (s, 1H), 4.59 (q, $J = 6.8$ Hz, 1H), 4.32 (s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 2.57 (s, 3H), 2.53 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 153.7, 139.9, 138.7, 136.0, 129.6, 126.5, 125.9 (q, $J = 280.9$ Hz), 116.8, 114.5, 81.6, 65.4 (q, $J = 30.5$ Hz), 60.6, 55.5, 53.0, 21.2; IR (KBr) ν 3319, 2948, 2926, 1742, 1513, 1449, 1381, 1208, 1165, 1135, 1039, 931, 817, 750, 589 cm^{-1} . HRMS: calcd. for $\text{C}_{20}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 395.1577, found: 395.1575. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 8.03$ and 9.93 min.



(2*R*,4*R*,5*R*)-methyl 1,2-bis(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

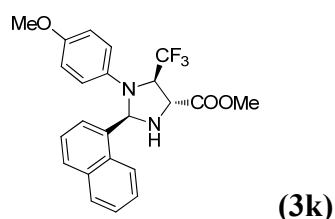
The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]_D^{25} = -50.6$ (c 1.56, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.46 (d, $J = 8.8$ Hz, 2H), 6.90 (d, $J = 8.8$ Hz, 2H), 6.75 (d, $J = 9.2$ Hz, 2H), 6.69 (d, $J = 9.2$ Hz, 2H), 5.37 (s, 1H), 4.62 (q, $J = 7.6$ Hz, 1H), 4.31 (s, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 3.71 (s, 3H), 2.65 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 159.9, 153.7, 139.9, 131.0, 127.9, 125.9 (q, $J = 280.8$ Hz), 117.0, 114.5, 114.2, 81.4, 65.4 (q, $J = 30.5$ Hz), 60.5, 55.4, 55.2, 52.9; IR (KBr) ν 3380, 2960, 2831, 1742, 1513, 1451, 1383, 1249, 1169, 1135, 1036, 931, 840, 690, 593 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 12.73$ and 16.73 min.



(2*R*,4*R*,5*R*)-methyl 2-(2-methoxyphenyl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

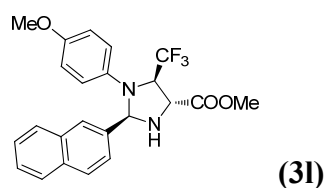
The title compound was prepared according to the general procedure as described above in 76% yield. $[\alpha]_D^{25} = +4.7$ (c 0.58, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.49 (d, $J = 7.6$ Hz, 1H), 7.33-7.31 (m, 1H), 6.95-6.93 (m, 2H), 6.75 (d, $J = 8.8$ Hz, 2H), 6.65 (d, $J = 8.8$ Hz, 2H), 5.84 (s, 1H), 4.59 (q, $J = 6.8$ Hz, 1H), 4.32 (s, 1H), 3.94 (s, 3H), 3.78 (s, 3H), 3.71 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.7, 157.2, 153.5, 139.9, 129.8, 126.8, 126.6, 125.8 (q, $J = 280.9$ Hz), 121.1, 116.5, 114.4, 110.5, 75.6, 65.2 (q, $J = 30.7$ Hz), 60.5, 55.6, 55.4, 52.9; IR (KBr) ν 3340, 2956, 2928, 1741,

1513, 1446, 1388, 1252, 1202, 1176, 1134, 931, 1036, 817, 699, 585 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 10.27 and 15.63 min.



(2*R*,4*R*,5*R*)-methyl 1-(4-methoxyphenyl)-2-(naphthalen-1-yl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

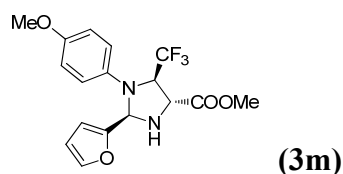
The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]_D^{25} = -58.4$ (*c* 1.42, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.47 (d, J = 8.8 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.85 (d, J = 8.4 Hz, 1H), 7.74 (d, J = 7.6 Hz, 1H), 7.63-7.61 (m, 1H), 7.57-7.55 (m, 1H), 7.44-7.41 (m, 1H), 6.72 (d, J = 9.2 Hz, 2H), 6.62 (d, J = 9.2 Hz, 2H), 6.20 (s, 1H), 4.68 (q, J = 6.8 Hz, 1H), 4.38 (s, 1H), 3.83 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.6, 153.4, 139.8, 133.9, 133.5, 130.9, 129.2, 128.5, 126.6, 125.90, 125.86 (q, J = 281.0 Hz), 125.8, 124.0, 123.3, 116.2, 114.5, 78.8, 65.3 (q, J = 30.7 Hz), 60.5, 55.4, 52.9; IR (KBr) ν 3328, 2956, 1743, 1515, 1425, 1250, 1134, 1037, 929, 817, 763, 669, 588 cm^{-1} . HRMS: calcd. for $\text{C}_{23}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 431.1577, found: 431.1576. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.29 and 8.59 min.



(2*R*,4*R*,5*R*)-methyl 1-(4-methoxyphenyl)-2-(naphthalen-2-yl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

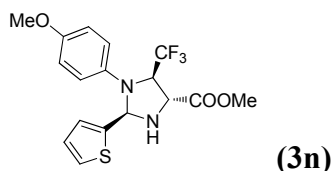
imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 76% yield. $[\alpha]_D^{25} = -68.1$ (c 1.03, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.00 (s, 1H), 7.89-7.83 (m, 3H), 7.68-7.66 (m, 1H), 7.50-7.48 (m, 2H), 6.74 (s, 4H), 5.59 (s, 1H), 4.70 (q, $J = 6.8$ Hz, 1H), 4.36 (s, 1H), 3.81 (s, 3H), 3.69 (s, 3H), 2.78 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 153.8, 139.8, 136.4, 133.6, 133.3, 128.9, 128.2, 127.7, 126.4, 126.3, 126.2, 125.9 (q, $J = 280.9$ Hz), 124.0, 117.1, 114.5, 82.0, 65.5 (q, $J = 30.5$ Hz), 60.6, 55.4, 53.0; IR (KBr) ν 3324, 2956, 1743, 1513, 1425, 1250, 1134, 1037, 953, 815, 765, 677, 588 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 10.63$ and 16.58 min.



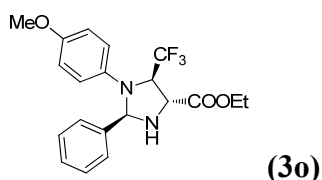
(2R,4R,5R)-methyl 2-(furan-2-yl)-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]_D^{25} = +36.3$ (c 1.29, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.42 (s, 1H), 6.80 (s, 4H), 6.42 (d, $J = 2.7$ Hz, 1H), 6.34 (s, 1H), 5.49 (s, 1H), 4.51 (q, $J = 6.6$ Hz, 1H), 4.33 (s, 1H), 3.78 (s, 3H), 3.74 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.6, 154.5, 152.1, 142.9, 139.6, 125.5 (q, $J = 280.5$ Hz), 118.1, 114.6, 110.6, 108.5, 75.9, 65.1 (q, $J = 30.8$ Hz), 60.5, 55.5, 53.0; IR (KBr) ν 3318, 2956, 2840, 1745, 1513, 1455, 1250, 1134, 1037, 955, 815, 760, 650 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 10.17$ and 21.37 min.



(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-(thiophen-2-yl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

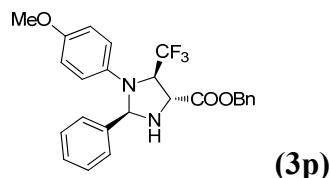
The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]_D^{25} = -52.7$ (*c* 0.87, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.29-7.27 (m, 1H), 7.22-7.21 (m, 1H), 6.99-6.97 (m, 1H), 6.84 (d, $J = 9.2$ Hz, 2H), 6.78 (d, $J = 9.2$ Hz, 2H), 5.69 (s, 1H), 4.52 (q, $J = 6.8$ Hz, 1H), 4.33 (s, 1H), 3.79 (s, 3H), 3.73 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 154.4, 143.5, 139.6, 127.0, 125.94, 125.90, 125.6 (q, $J = 280.5$ Hz), 118.1, 114.5, 77.9, 65.8 (q, $J = 30.7$ Hz), 60.5, 55.4, 52.9; IR (KBr) ν 3318, 2950, 2844, 1743, 1513, 1460, 1250, 1134, 1038, 935, 822, 721, 529 cm^{-1} . The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 9.15$ and 14.01 min.



(2R,4R,5R)-ethyl 1-(4-methoxyphenyl)-2-phenyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

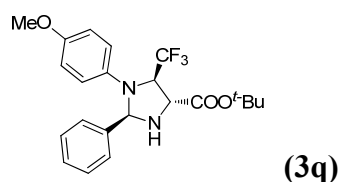
The title compound was prepared according to the general procedure as described above in 80% yield. $[\alpha]_D^{25} = -21.6$ (*c* 1.52, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.55 (d, $J = 6.4$ Hz, 2H), 7.40-7.34 (m, 3H), 6.75 (d, $J = 9.2$ Hz, 2H), 6.69 (d, $J = 9.2$ Hz, 2H), 5.44 (s, 1H), 4.60 (q, $J = 7.2$ Hz, 1H), 4.29 (s, 1H), 4.23 (q, $J = 7.2$ Hz, 2H), 3.71 (s, 3H), 1.25 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.0, 153.8, 140.0, 139.1, 128.94, 128.86, 126.7, 125.9 (q, $J = 280.8$ Hz), 117.0, 114.5, 81.9, 65.6 (q, $J = 30.5$ Hz), 62.0, 60.8, 55.5, 14.0; IR (KBr) ν 3362, 2951, 2855, 1744, 1510, 1436, 1355, 1248, 1135, 1030, 934, 820, 711, 625 cm^{-1} . HRMS: calcd. for $\text{C}_{20}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 395.1577, found: 395.1569. The product was analyzed by HPLC

to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.90 and 10.26 min.



(2R,4R,5R)-benzyl 1-(4-methoxyphenyl)-2-phenyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

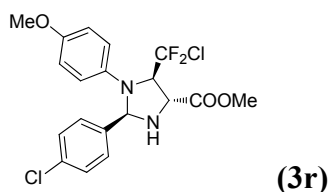
The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]_D^{25} = -12.7$ (*c* 0.93, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.54-7.52 (m, 2H), 7.37-7.22 (m, 8H), 6.73 (d, *J* = 9.0 Hz, 2H), 6.64 (d, *J* = 9.0 Hz, 2H), 5.44 (s, 1H), 5.19 (s, 2H), 4.58 (q, *J* = 6.9 Hz, 1H), 4.34 (s, 1H), 3.72 (s, 3H), 2.68 (brs, 1H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 169.8, 153.8, 139.9, 139.0, 134.9, 128.93, 128.87, 128.6, 128.4, 128.1, 126.7, 125.8 (q, *J* = 281.0 Hz), 117.0, 114.5, 82.0, 67.5, 65.6 (q, *J* = 30.5 Hz), 60.9, 55.5; IR (KBr) ν 3318, 2951, 2844, 1755, 1510, 1442, 1360, 1228, 1123, 1035, 933, 825, 690 cm⁻¹. HRMS: calcd. for C₂₅H₂₃F₃N₂O₃ + H⁺: 457.1734, found: 457.1738. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.33 and 16.08 min.



(2R,4R,5R)-tert-butyl 1-(4-methoxyphenyl)-2-phenyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

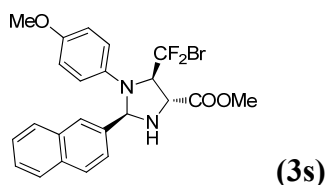
The title compound was prepared according to the general procedure as described above in 84% yield. $[\alpha]_D^{25} = -22.3$ (*c* 0.61, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 7.54 (d, *J* = 6.8 Hz, 2H), 7.39-7.33 (m, 3H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 5.46 (s, 1H), 4.50 (q, *J* = 6.8 Hz, 1H), 4.18 (s, 1H), 3.71 (s, 3H), 2.63

(brs, 3H), 1.40 (s, 9H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 169.2, 153.6, 140.3, 139.3, 128.9, 128.8, 126.7, 125.9 (q, $J = 281.0$ Hz), 116.7, 114.5, 82.7, 82.1, 65.8 (q, $J = 30.4$ Hz), 61.6, 55.5, 27.8; IR (KBr) ν 3320, 2955, 2844, 1755, 1510, 1442, 1360, 1228, 1123, 1035, 933, 825, 690 cm^{-1} . HRMS: calcd. for $\text{C}_{22}\text{H}_{25}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 423.1890, found: 423.1905. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 5.56$ and 6.30 min.



(2R,4R,5R)-methyl 5-(chlorodifluoromethyl)-2-(4-chlorophenyl)-1-(4-methoxyphenyl)imidazolidine-4-carboxylate

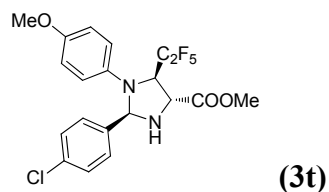
The title compound was prepared according to the general procedure as described above in 76% yield; $[\alpha]_D^{25} = -46.4$ (c 0.80, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.50 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 6.76 (d, $J = 9.2$ Hz, 2H), 6.72 (d, $J = 9.2$ Hz, 2H), 5.40 (s, 1H), 4.72 (dd, $J_1 = 5.2$ Hz, $J_2 = 13.6$ Hz, 1H), 4.37 (s, 1H), 3.80 (s, 3H), 3.72 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.6, 154.4, 139.6, 137.4, 134.6, 129.9 (t, $J = 295.7$ Hz), 129.1, 128.3, 118.3, 114.5, 82.3, 71.2 (t, $J = 24.2$ Hz), 61.6, 55.4, 53.0; IR (KBr) ν 3340, 2951, 2927, 2844, 1744, 1710, 1595, 1513, 1445, 1360, 1160, 1131, 1040, 929, 815, 752, 669 cm^{-1} . HRMS: calcd. for $\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{F}_2\text{N}_2\text{O}_3 + \text{H}^+$: 431.0735, found: 431.0743. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 9.47$ and 12.17 min.



(2R,4R,5R)-methyl 5-(bromodifluoromethyl)-1-(4-methoxyphenyl)-2-(naphtha-

alen-2-yl)imidazolidine-4-carboxylate

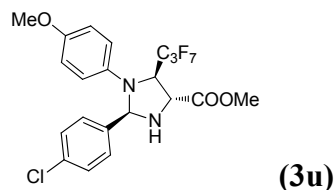
The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]_D^{25} = -31.9$ (c 1.81, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.02 (s, 1H), 7.88-7.81 (m, 3H), 7.71 (dd, $J_1 = 1.2$ Hz, $J_2 = 8.4$ Hz, 1H), 7.51-7.46 (m, 2H), 6.78 (d, $J = 9.2$ Hz, 2H), 6.73 (d, $J = 9.2$ Hz, 2H), 5.58 (s, 1H), 4.84 (dd, $J_1 = 4.4$ Hz, $J_2 = 15.6$ Hz, 1H), 4.44 (s, 1H), 3.83 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.6, 154.2, 139.8, 136.3, 133.6, 133.2, 128.8, 128.2, 127.7, 126.39, 126.36, 126.3, 124.5 (t, $J = 309.8$ Hz), 124.2, 118.2, 114.4, 83.3, 72.7 (t, $J = 21.3$ Hz), 62.0, 55.4, 53.0; IR (KBr) ν 3347, 2957, 2927, 2855, 1742, 1513, 1450, 1220, 1178, 1131, 1038, 929, 756, 669, 590 cm^{-1} . HRMS: calcd. for $\text{C}_{23}\text{H}_{21}\text{BrF}_2\text{N}_2\text{O}_3 + \text{H}^+$: 491.0776, found: 491.0769. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 13.75$ and 17.03 min.



(2R,4R,5R)-methyl 2-(4-chlorophenyl)-1-(4-methoxyphenyl)-5-(perfluoroethyl)-imidazolidine-4-carboxylate

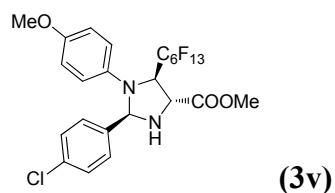
The title compound was prepared according to the general procedure as described above in 80% yield; $[\alpha]_D^{25} = -63.2$ (c 1.10, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.49 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 6.75 (d, $J = 9.2$ Hz, 2H), 6.66 (d, $J = 9.2$ Hz, 2H), 5.32 (s, 1H), 4.76 (dd, $J_1 = 7.6$ Hz, $J_2 = 19.2$ Hz, 1H), 4.41 (s, 1H), 3.80 (s, 3H), 3.71 (s, 3H), 2.65 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 154.4, 139.7, 137.7, 134.7, 129.2, 128.2, 118.4, 114.6, 82.0, 63.9 (m), 60.6, 55.4, 53.1; IR (KBr) ν 3347, 2953, 2940, 1744, 1711, 1595, 1513, 1424, 1361, 1176, 1132, 1040, 929, 815, 752, 669, 626 cm^{-1} . HRMS: calcd. for $\text{C}_{20}\text{H}_{18}\text{ClF}_5\text{N}_2\text{O}_3 + \text{H}^+$: 465.0999, found: 465.1008. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, =

220 nm); $t_r = 6.54$ and 8.30 min.



(2R,4R,5R)-methyl 2-(4-chlorophenyl)-1-(4-methoxyphenyl)-5-(perfluoropropyl)imidazolidine-4-carboxylate

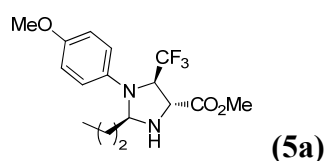
The title compound was prepared according to the general procedure as described above in 80% yield; $[\alpha]_D^{25} = -66.6$ (c 1.30, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.49 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 6.76 (d, $J = 8.8$ Hz, 2H), 6.66 (d, $J = 8.8$ Hz, 2H), 5.34 (s, 1H), 4.86 (dd, $J_1 = 4.4$ Hz, $J_2 = 21.6$ Hz, 1H), 4.41 (s, 1H), 3.79 (s, 3H), 3.71 (s, 3H), 2.65 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 154.4, 139.8, 137.7, 134.7, 129.2, 128.2, 118.3, 117.6 (q, $J = 283.7$ Hz), 114.6, 81.5, 64.1 (m), 60.7, 55.4, 53.1; IR (KBr) ν 3341, 2940, 1740, 1513, 1476, 1425, 1377, 1262, 1175, 1015, 929, 783, 669 cm^{-1} . HRMS: calcd. for $\text{C}_{21}\text{H}_{18}\text{ClF}_7\text{N}_2\text{O}_3 + \text{H}^+$: 515.0967, found: 515.0968. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 5.72$ and 6.75 min.



(2R,4R,5R)-methyl 2-(4-chlorophenyl)-1-(4-methoxyphenyl)-5-(perfluorohexyl)imidazolidine-4-carboxylate

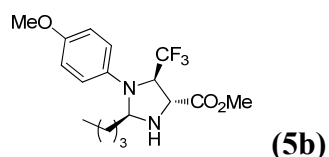
The title compound was prepared according to the general procedure as described above in 88% yield; $[\alpha]_D^{25} = -30.9$ (c 1.02, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.49 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 6.76 (d, $J = 8.8$ Hz, 2H), 6.66 (d, $J = 8.8$ Hz, 2H), 5.35 (s, 1H), 4.88 (dd, $J_1 = 4.0$ Hz, $J_2 = 22.4$ Hz, 1H), 4.42 (s, 1H), 3.79 (s, 3H), 3.70 (s, 3H), 2.65 (brs, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5,

154.3, 139.8, 137.7, 134.7, 129.2, 128.2, 118.2, 114.6, 81.5, 64.3 (m), 60.8, 55.4, 53.0; IR (KBr) ν 3340, 2950, 2834, 1744, 1515, 1446, 1425, 1144, 1016, 929, 771, 669, cm^{-1} . HRMS: calcd. for $\text{C}_{24}\text{H}_{18}\text{ClF}_{13}\text{N}_2\text{O}_3 + \text{H}^+$: 665.0871, found: 665.0861. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralcel OD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 4.21 and 4.77 min.



**(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-propyl-5-(trifluoromethyl)imidazo-
lidine-4-carboxylate**

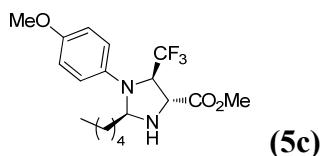
The title compound was prepared according to the general procedure as described above in 80% yield. d.r. = 10:1; $[\alpha]_D^{25} = -26.1$ (c 0.42, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 6.94 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 4.47-4.46 (m, 1H), 4.26 (q, J = 7.2 Hz, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 1.85-1.82 (m, 1H), 1.44-1.40 (m, 3H), 0.94 (t, J = 6.8 Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 171.2, 155.1, 140.5, 125.6 (q, J = 280.0 Hz), 120.9, 114.6, 79.8, 67.2 (q, J = 30.0 Hz), 60.5, 55.5, 52.9, 36.8, 18.1, 14.1; IR (KBr) ν 3336, 2957, 2927, 2855, 1742, 1513, 1450, 1220, 1038, 929, 756, 669 cm^{-1} . HRMS: calcd. for $\text{C}_{16}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 347.1577, found: 347.1584. The product was analyzed by HPLC to determine the enantiomeric excess: 89% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.14 and 6.13 min.



**(2R,4R,5R)-methyl 2-butyl-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazoli-
dine-4-carboxylate**

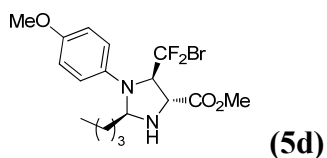
The title compound was prepared according to the general procedure as described

above in 76% yield. d.r. > 20:1; $[\alpha]_D^{25} = -14.3$ (*c* 0.83, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 6.93 (d, *J* = 9.2 Hz, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 4.46-4.45 (m, 1H), 4.26 (q, *J* = 7.2 Hz, 1H), 4.21 (s, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 1.90-1.85 (m, 1H), 1.51-1.29 (m, 5H), 0.88 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.2, 155.1, 140.4, 125.9 (q, *J* = 279.8 Hz), 120.8, 114.7, 80.0, 67.1 (q, *J* = 29.9 Hz), 60.5, 55.5, 52.9, 34.3, 26.9, 22.7, 14.0; IR (KBr) ν 3368, 1736, 1516, 1448, 1425, 1210, 1044, 929, 705, 656 cm⁻¹. HRMS: calcd. for C₁₇H₂₃F₃N₂O₃ + H⁺: 361.1734, found: 361.1742. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.34 and 7.30 min.



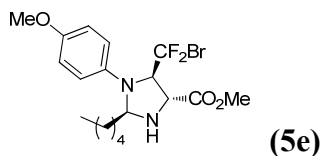
(2*R*,4*R*,5*R*)-methyl 1-(4-methoxyphenyl)-2-pentyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. d.r. = 17:1; $[\alpha]_D^{25} = -19.0$ (*c* 0.55, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 6.93 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 4.46-4.45 (m, 1H), 4.26 (q, *J* = 7.2 Hz, 1H), 4.21 (s, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 1.87-1.85 (m, 1H), 1.51-1.29 (m, 7H), 0.88 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.2, 155.1, 140.4, 125.6 (q, *J* = 281.6 Hz), 120.8, 114.7, 80.0, 67.1 (q, *J* = 30.1 Hz), 60.5, 55.5, 52.9, 34.6, 31.8, 24.5, 22.6, 14.0; IR (KBr) ν 3335, 1756, 1512, 1469, 1255, 1032, 917, 740, 669 cm⁻¹. HRMS: calcd. for C₁₈H₂₅F₃N₂O₃ + H⁺: 375.1890, found: 375.1883. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.34 and 5.88 min.



(2R,4R,5R)-methyl 5-(bromodifluoromethyl)-1-(4-methoxyphenyl)-2-propylimidazolidine-4-carboxylate

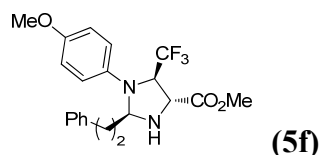
The title compound was prepared according to the general procedure as described above in 80% yield. d.r. = 15:1; $[\alpha]_D^{25} = -12.6$ (*c* 0.27, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 7.03 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 4.38-4.33 (m, 2H), 4.26 (s, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 1.86-1.80 (m, 1H), 1.48-1.45 (m, 2H), 1.32-1.30 (m, 3H), 0.88 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.3, 156.0, 140.3, 125.3 (t, *J* = 309.0 Hz), 123.5, 114.6, 81.8, 74.5 (t, *J* = 20.3 Hz), 62.1, 55.5, 52.9, 34.1, 26.9, 22.8, 14.0; IR (KBr) ν 3317, 2957, 1740, 1515, 1477, 1425, 1250, 1023, 930, 755, 670 cm⁻¹. HRMS: calcd. for C₁₇H₂₃BrF₂N₂O₃ + H⁺: 421.0933, found: 421.0935. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); *t*_r = 6.23 and 6.90 min.



(2R,4R,5R)-methyl 5-(bromodifluoromethyl)-2-ethyl-1-(4-methoxyphenyl)imidazolidine-4-carboxylate

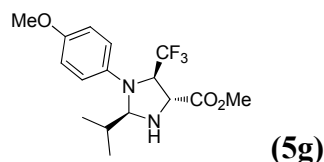
The title compound was prepared according to the general procedure as described above in 76% yield. d.r. = 10:1; $[\alpha]_D^{25} = -20.7$ (*c* 0.15, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 7.03 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 4.37-4.33 (m, 1H), 4.26 (s, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 1.86-1.79 (m, 1H), 1.47-1.26 (m, 7H), 0.87 (t, *J* = 6.8 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.4, 156.0, 140.3, 123.5, 114.6, 81.9, 74.5 (t, *J* = 20.6 Hz), 62.1, 55.5, 52.9, 34.4, 31.9, 24.5, 22.5, 14.0; IR (KBr) ν 2958, 2924, 1743, 1512, 1477, 1435, 1215, 1133, 1021, 935, 783, 670 cm⁻¹. HRMS: calcd. for C₁₈H₂₅BrF₂N₂O₃ + H⁺: 435.1089, found: 435.1076. The

product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 220 nm); t_r = 6.08 and 6.84 min.



(2R,4R,5R)-methyl 1-(4-methoxyphenyl)-2-phenethyl-5-(trifluoromethyl)imidazolidine-4-carboxylate

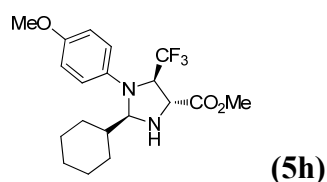
The title compound was prepared according to the general procedure as described above in 80% yield. d.r. > 20:1; $[\alpha]_D^{25}$ = -34.5 (*c* 0.95, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 7.29-7.25 (m, 2H), 7.20-7.17 (m, 3H), 6.91 (d, *J* = 9.2 Hz, 2H), 6.83 (d, *J* = 9.2 Hz, 2H), 4.51-4.49 (m, 1H), 4.26 (q, *J* = 7.2 Hz, 1H), 4.23 (s, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 2.84-2.71 (m, 1H), 2.72-2.69 (m, 1H), 2.18-2.15 (m, 1H), 1.80-1.74 (m, 1H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.2, 155.4, 141.3, 140.3, 128.4, 128.3, 126.0, 125.6 (q, *J* = 279.9 Hz), 121.4, 114.7, 79.6, 67.3 (q, *J* = 30.1 Hz), 60.6, 55.5, 52.9, 36.1, 31.0; IR (KBr) ν 2956, 2840, 1743, 1603, 1512, 1480, 1439, 1214, 1133, 1036, 929, 783, 771, 669 cm⁻¹. HRMS: calcd. for C₂₁H₂₃F₃N₂O₃ + Na⁺: 431.1553, found: 431.1568. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.77 and 8.76 min.



(2R,4R,5R)-methyl 2-isopropyl-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

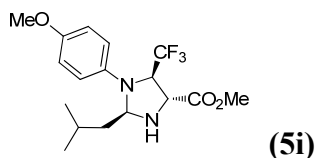
The title compound was prepared according to the general procedure as described above in 76% yield. d.r. > 20:1; $[\alpha]_D^{25}$ = -17.5 (*c* 0.61, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 7.06 (d, *J* = 9.2 Hz, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 4.35 (d, *J* = 3.6

Hz, 1H), 4.18-4.13 (m, 2H), 3.80 (s, 3H), 3.78 (s, 3H), 1.94-1.89 (m, 1H), 0.99 (d, $J = 6.8$ Hz, 3H), 0.94 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 171.6, 156.1, 141.1, 125.7 (q, $J = 280.1$ Hz), 123.9, 114.6, 85.1, 68.6 (q, $J = 29.7$ Hz), 60.5, 55.5, 52.9, 29.7, 19.2, 14.6; IR (KBr) ν 3317, 2957, 2436, 1742, 1603, 1479, 1425, 1214, 1133, 1036, 929, 669 cm^{-1} . HRMS: calcd. for $\text{C}_{16}\text{H}_{21}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 347.1577, found: 347.1585. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 4.79$ and 5.33 min.



(2R,4R,5R)-methyl 2-cyclohexyl-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 76% yield. d.r. > 20:1; $[\alpha]_D^{25} = -32.3$ (c 0.56, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.03 (d, $J = 8.8$ Hz, 2H), 6.85 (d, $J = 8.8$ Hz, 2H), 4.35 (d, $J = 4.0$ Hz, 1H), 4.17-4.13 (m, 2H), 3.79 (s, 3H), 3.78 (s, 3H), 2.60 (brs, 1H), 1.92-1.89 (m, 1H), 1.79-1.65 (m, 5H), 1.19-1.12 (m, 4H), 0.95-0.92 (m, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 171.5, 155.8, 141.3, 125.6 (q, $J = 279.8$ Hz), 123.2, 114.5, 84.5, 68.3 (q, $J = 29.8$ Hz), 60.5, 55.4, 52.8, 40.2, 30.0, 26.6, 26.5, 26.1, 25.7; IR (KBr) ν 3317, 2957, 2434, 1740, 1601, 1469, 1423, 1210, 1131, 1034, 929, 669 cm^{-1} . HRMS: calcd. for $\text{C}_{19}\text{H}_{25}\text{F}_3\text{N}_2\text{O}_3 + \text{H}^+$: 387.1890, found: 387.1894. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 5.43$ and 6.32 min.

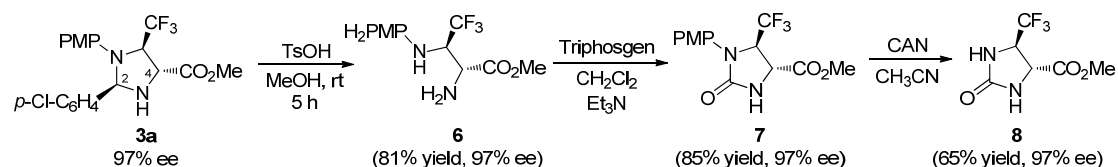


(2R,4R,5R)-methyl 2-isobutyl-1-(4-methoxyphenyl)-5-(trifluoromethyl)imidazolidine-4-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. d.r. = 15:1; $[\alpha]_D^{25} = -37.3$ (*c* 1.12, CHCl₃); ¹H NMR (CDCl₃, TMS, 400 MHz) δ 6.92 (d, *J* = 9.2 Hz, 2H), 6.85 (d, *J* = 9.2 Hz, 2H), 4.56-4.54 (m, 1H), 4.24 (q, *J* = 7.2 Hz, 1H), 4.21 (s, 1H), 3.77 (s, 3H), 3.76 (s, 3H), 1.84-1.71 (m, 2H), 1.35-1.33 (m, 1H), 0.98 (d, *J* = 6.4 Hz, 3H), 0.93 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.1, 154.9, 140.5, 125.6 (q, *J* = 279.9 Hz), 120.4, 114.6, 78.5, 67.0 (q, *J* = 30.0 Hz), 60.6, 55.5, 52.8, 44.3, 25.3, 23.7, 22.0; IR (KBr) ν 2955, 2434, 1741, 1608, 1468, 1421, 1210, 1135, 1037, 929, 668 cm⁻¹. HRMS: calcd. for C₁₇H₂₃F₃N₂O₃ + H⁺: 361.1734, found: 361.1737. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); t_r = 4.81 and 5.16 min.

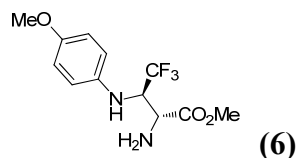
V. Synthetic Transformations.

Due to high volatility of (2R,3R)-2,3-diamino-4,4,4-trifluoro-butanoate, oxidative cleavage of the PMP group could be performed as below: Firstly, diamine **6** was obtained by acidic hydrolysis of the corresponding cycloadduct **3a** in good yield. The diamine **6** was further transformed into cyclic urea **7** with triphosphen, then treatment with Ce(NH₄)₂(NO₃)₆ gave the derived amide **8** without loss of the diastereo-/enantiomeric excess.



3a (207 mg, 0.5 mmol) was dissolved in 3 mL of methanol at room temperature followed by the addition of TsOH·H₂O (380 mg, 2 mmol). The reaction mixture was

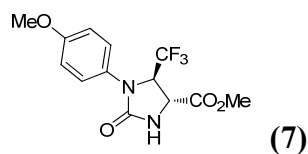
stirred until starting material was consumed (monitored by TLC) and neutralized the mixture by Na₂CO₃. Then the mixture was partitioned between ethyl acetate and water, then the organic layer was washed with brine, dried over MgSO₄, and concentrated in vacuo then the organic solvent was removed and the residue was purified by column chromatography to give compound **6** in 81% yield.



(2R,3R)-methyl 2-amino-4,4,4-trifluoro-3-((4-methoxyphenyl)amino)butanoate

$[\alpha]_D^{25} = -41.2$ (*c* 0.73, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 6.76 (d, *J* = 8.7 Hz, 2H), 6.66 (d, *J* = 8.7 Hz, 2H), 4.54-4.51 (m, 1H), 4.43-4.36 (m, 1H), 4.12 (s, 1H), 3.73 (s, 3H), 3.59 (s, 3H), 1.78 (brs, 2H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 171.7, 153.1, 139.9, 125.6 (q, *J* = 283.1 Hz), 116.0, 114.7, 58.4 (q, *J* = 27.9 Hz), 55.6, 52.7, 52.6; IR (KBr) ν 3368, 2951, 1746, 1516, 1453, 1218, 1035, 929, 669 cm⁻¹. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 220 nm); *t*_r = 11.07 and 20.46 min.

To a solution of **6** (100 mg, 0.34 mmol) and triethylamine (141 μ L, 1.02 mmol) in dry CH₂Cl₂ (15.0 mL) under nitrogen at 0 °C was added a solution of triphosgene (100 mg, 0.34 mmol) in dry CH₂Cl₂ dropwise. The reaction mixture was warmed to room temperature and stirred until the starting material was consumed completely as indicated by TLC. Then the reaction was quenched and purified by column chromatography to give the cyclic urea **7**.

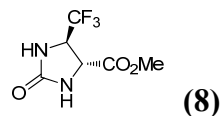


(4R,5R)-methyl 1-(4-methoxyphenyl)-2-oxo-5-(trifluoromethyl)imidazolidine-4-carboxylate

$[\alpha]_D^{25} = +18.8$ (*c* 0.16, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.24 (d, *J* = 9.0 Hz, 2H), 6.92 (d, *J* = 9.0 Hz, 2H), 5.85 (s, 1H), 4.91-4.88 (m, 1H), 4.33 (d, *J* = 2.4 Hz,

1H), 3.89 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 169.7, 158.9, 158.2, 129.3, 126.9, 123.8 (q, $J = 281.5$ Hz), 114.3, 61.1 (q, $J = 32.4$ Hz), 55.3, 53.3, 51.9; IR (KBr) ν 2917, 2846, 2335, 1722, 1515, 1423, 1241, 1166 cm^{-1} . HRMS: calcd. for $\text{C}_{13}\text{H}_{13}\text{F}_3\text{N}_2\text{O}_4 + \text{H}^+$: 319.0897, found: 319.0900. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (Chiralpak AD-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 14.87$ and 16.88 min.

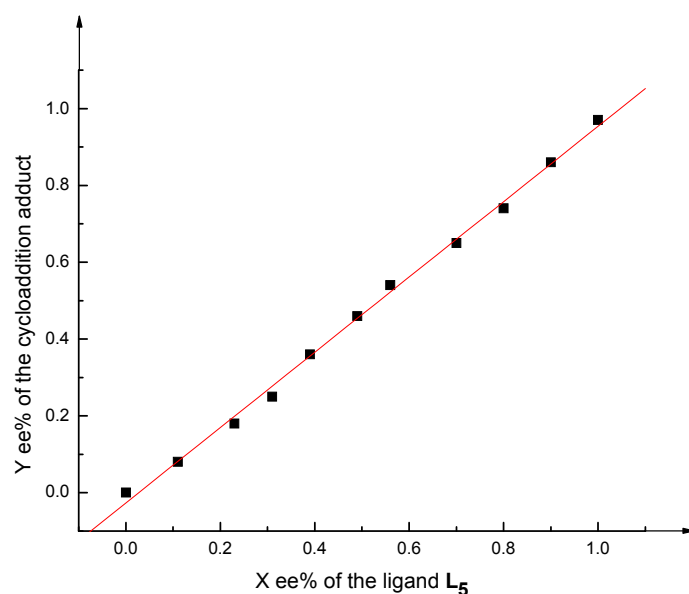
To a solution of **7** (92 mg, 0.29 mmol) in dry acetonitrile (2.0 mL) was added dropwise a solution of CAN (477 mg, 0.87 mmol) in H_2O (1.0 mL) at 0 °C. The reaction was completed immediately and quenched by the addition of saturated NH_4Cl aqueous solution. The phases were separated and the aqueous phase was extracted with ethyl acetate. The combined organic phases were dried over sodium sulfate and concentrated under vacuum. The residue was purified by chromatography to give **8** as a white solid.



(4*R*,5*R*)-methyl 2-oxo-5-(trifluoromethyl)imidazolidine-4-carboxylate

$[\alpha]_D^{25} = -35.0$ (c 0.20, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 6.92 (s, 1H), 6.41 (s, 1H), 4.46 (m, 1H), 4.33 (d, $J = 3.2$ Hz, 1H), 3.85 (s, 3H); ^{13}C NMR ($\text{DMSO}-d_6$, TMS, 100 MHz) δ 170.7, 161.1, 124.7 (q, $J = 279.4$ Hz), 55.2 (q, $J = 32.2$ Hz), 53.4, 52.9; IR (KBr) ν 3243, 2922, 2360, 2341, 1724, 1443, 1240, 1176, 1145 cm^{-1} . HRMS: calcd. for $\text{C}_6\text{H}_7\text{F}_3\text{N}_2\text{O}_3 + \text{Na}^+$: 235.0296, found: 235.0301. The product was analyzed by GC to determine the enantiomeric excess: 97% ee (β -dex 325 column, 30 m x 0.25 mm x 0.25 μm , column temperature: 170 °C, carrier gas: N_2 , 1 mL/min); $t_r = 9.84$ and 14.29 min.

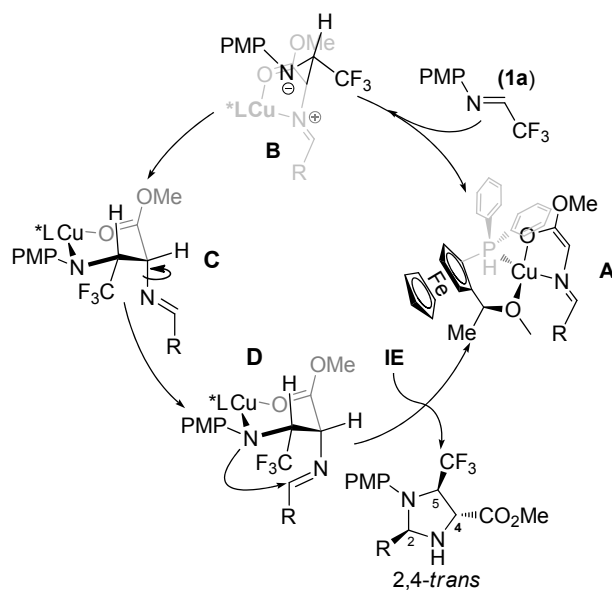
VI. Linear Effect for the 1,3-Dipolar Cycloaddition of Trifluoromethylated Imine **1a with Imino Ester **2a** Catalyzed by $\text{Cu}(\text{CH}_3\text{CN})_4\text{BF}_4/(\text{S},\text{R}_p)\text{-PPFOMe}$ Complex.**



Ee of (*S,R_p*)-**L5** were determined by HPLC: Chiralpak AD-H, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm; t_r = 16.01 and 21.18 min.

L5_{ee} (%)	11	23	31	39	49	56	70	80	90	100
Prod_{ee} (%)	8	18	25	36	46	54	65	74	86	97

VII. Proposed Transition States of the *exo'*-Selectivity for Asymmetric 1,3-Dipolar Cycloaddition of Imino Esters with Fluorinated Imines.



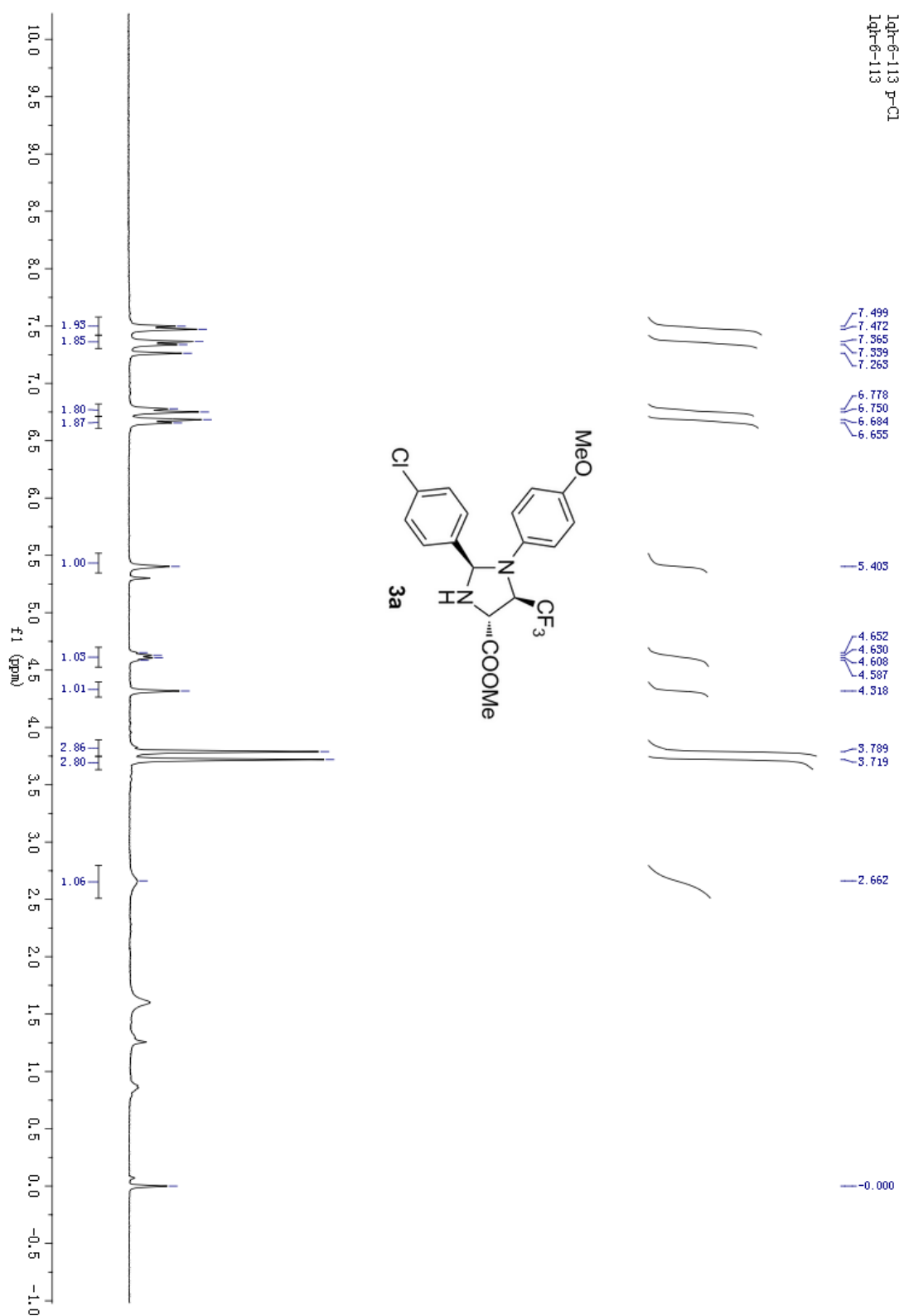
Scheme S1. Postulated catalytic cycle for Cu(I)-catalyzed asymmetric 1,3-DC of azomethine ylide with fluorinated imines.

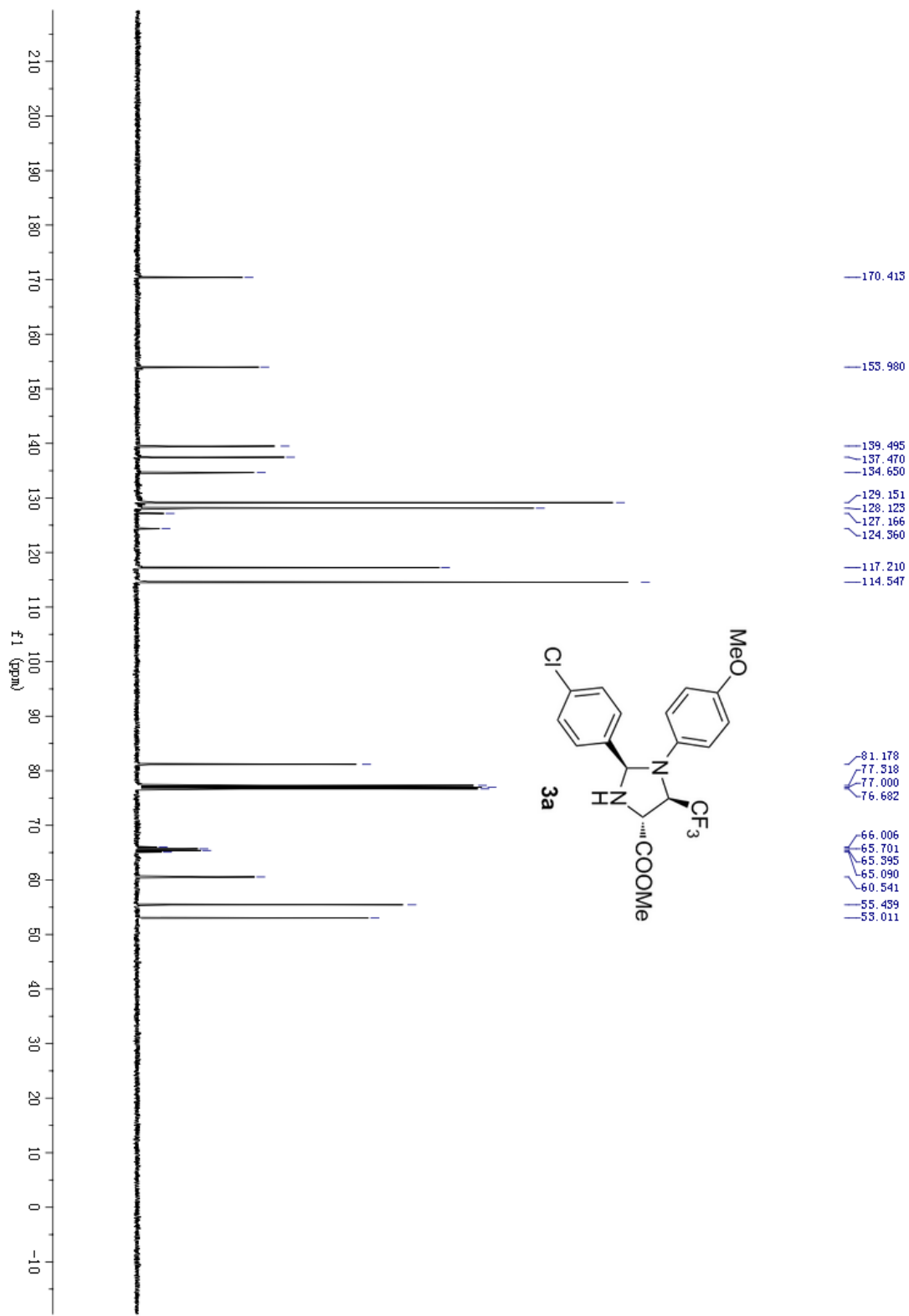
Based on the relative and absolute configuration of (2*R*,4*R*,5*R*)-**3s** and previous studies,³ a plausible stepwise mechanism was proposed to rationalize the observed *exo'*-selectivity for this 1,3-DC. The *in situ*-formed azomethine ylide is coordinated to the Cu complex leading to the catalytically active species (**A**) based on the linear correlation results. Initial Mannich addition of the metalloazomethine ylide (**A**) to the *Re* face (C=N) of the fluorinated imine **1a** through the gauche conformation generates the zwitterionic intermediate (**B**), which could be facilitated by the possible coordination interaction between the imino group of **1a** and the Cu(I) center. After the Mannich reaction, the copper atom spontaneously switches from imino ester to NPMP for forming the six-membered chair-like species (**C**).³ Before the subsequent intramolecular cyclization, the C-N single bond must rotate into the species (**D**) hence the amino unit approaches the *Re* face of the imine moiety to give the *exo'*-diastereomer, in which the substituents at 2 and 4 position of imidazolidine ring are arranged at *trans* configuration. Nevertheless, the real catalytic mechanism still needs further investigation.

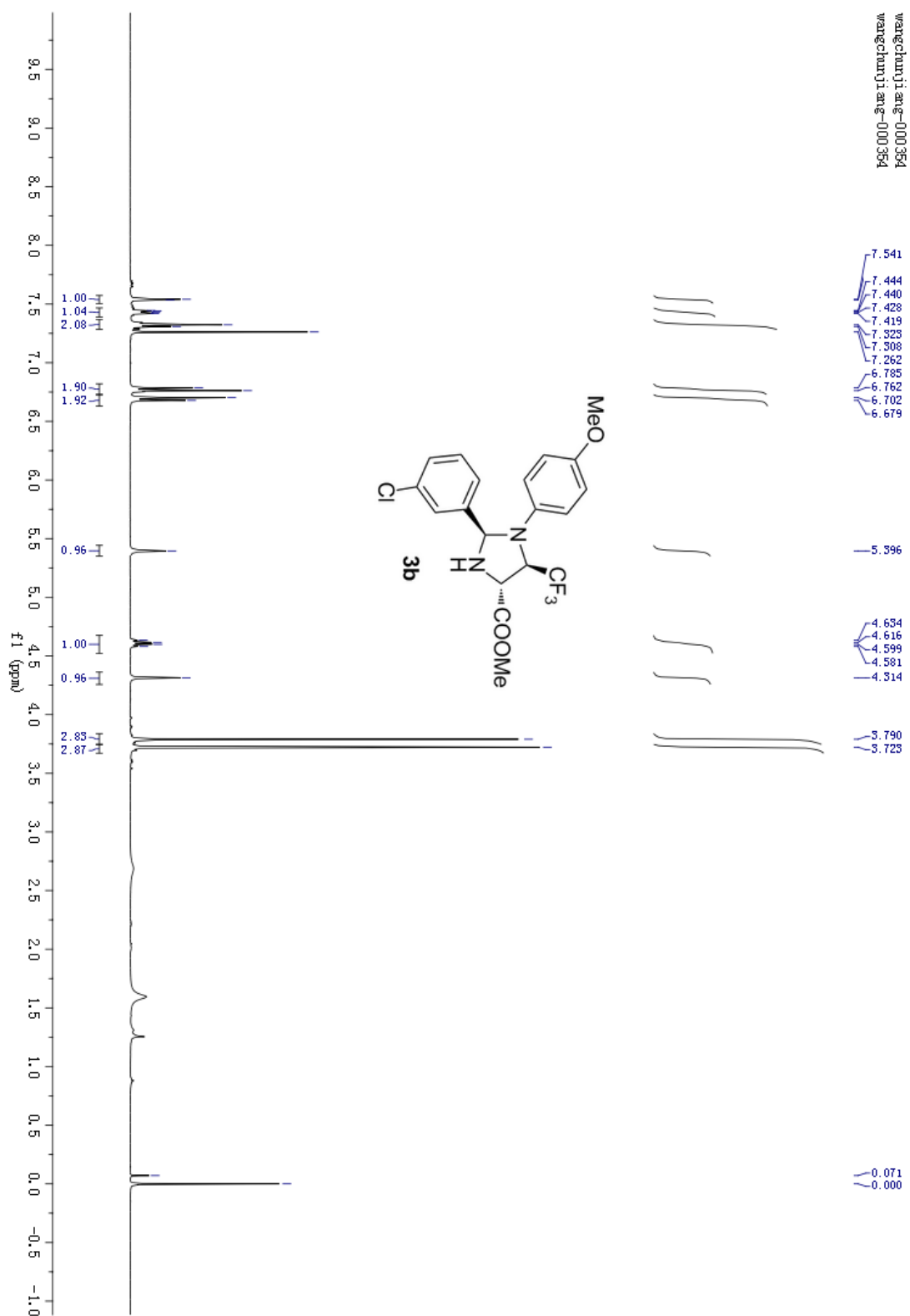
VIII. References.

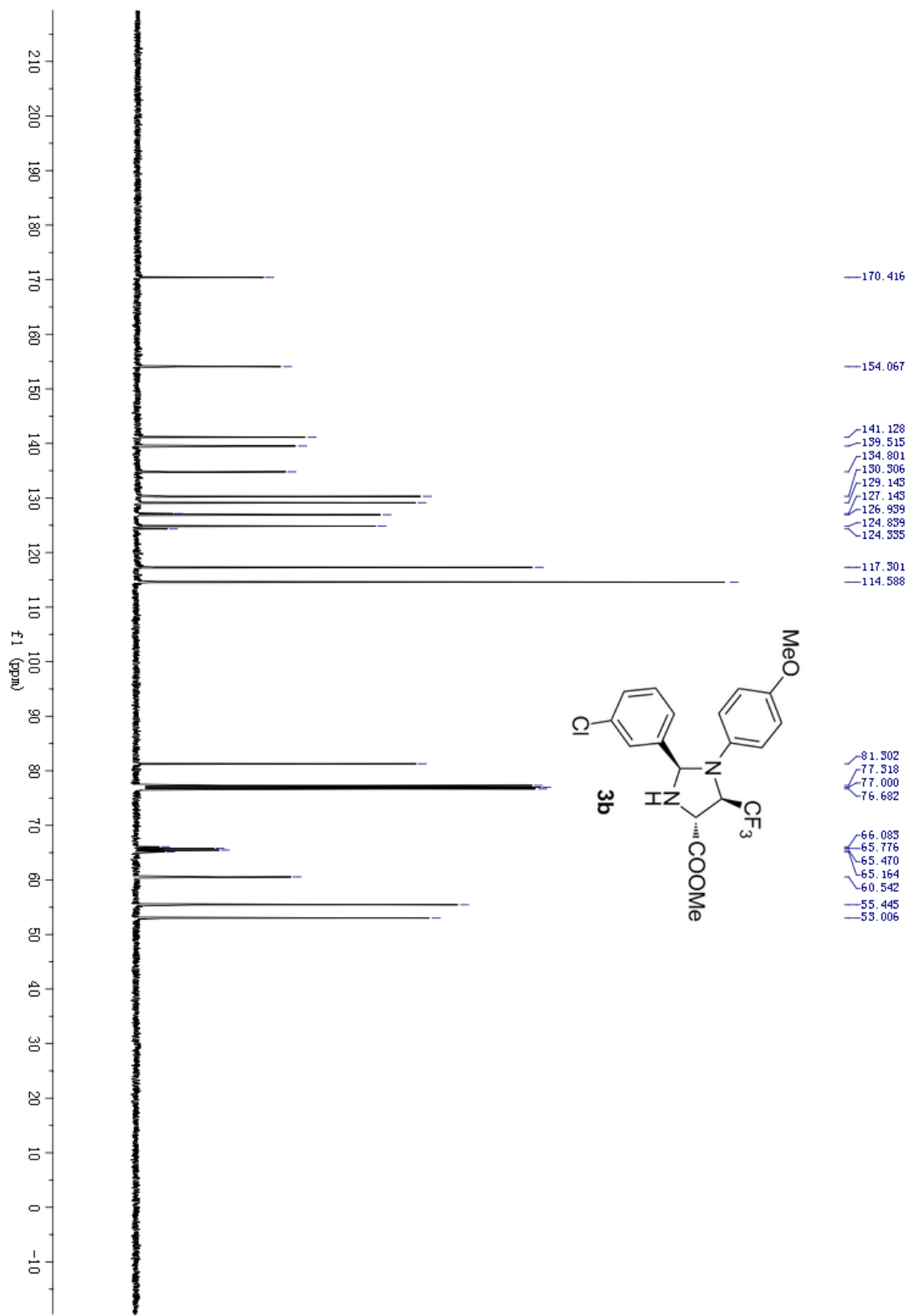
1. T. Hayashi, T. Mise, M. Fukushima, M. Kagotani, N. Nagashima, Y. Hamada, A. Matsumoto, S. Kawakami, M. Konishi, K. Yamamoto, M. Kumada, *Bull. Chem. Soc. Jpn.* **1980**, 53, 1138.
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3. (a) T. Arai, N. Yokoyama, A. Mishiro and H. Sato, *Angew. Chem., Int. Ed.*, **2010**, 49, 7895; (b) A. Awata and T. Arai, *Chem. Eur. J.* **2012**, 18, 8278.

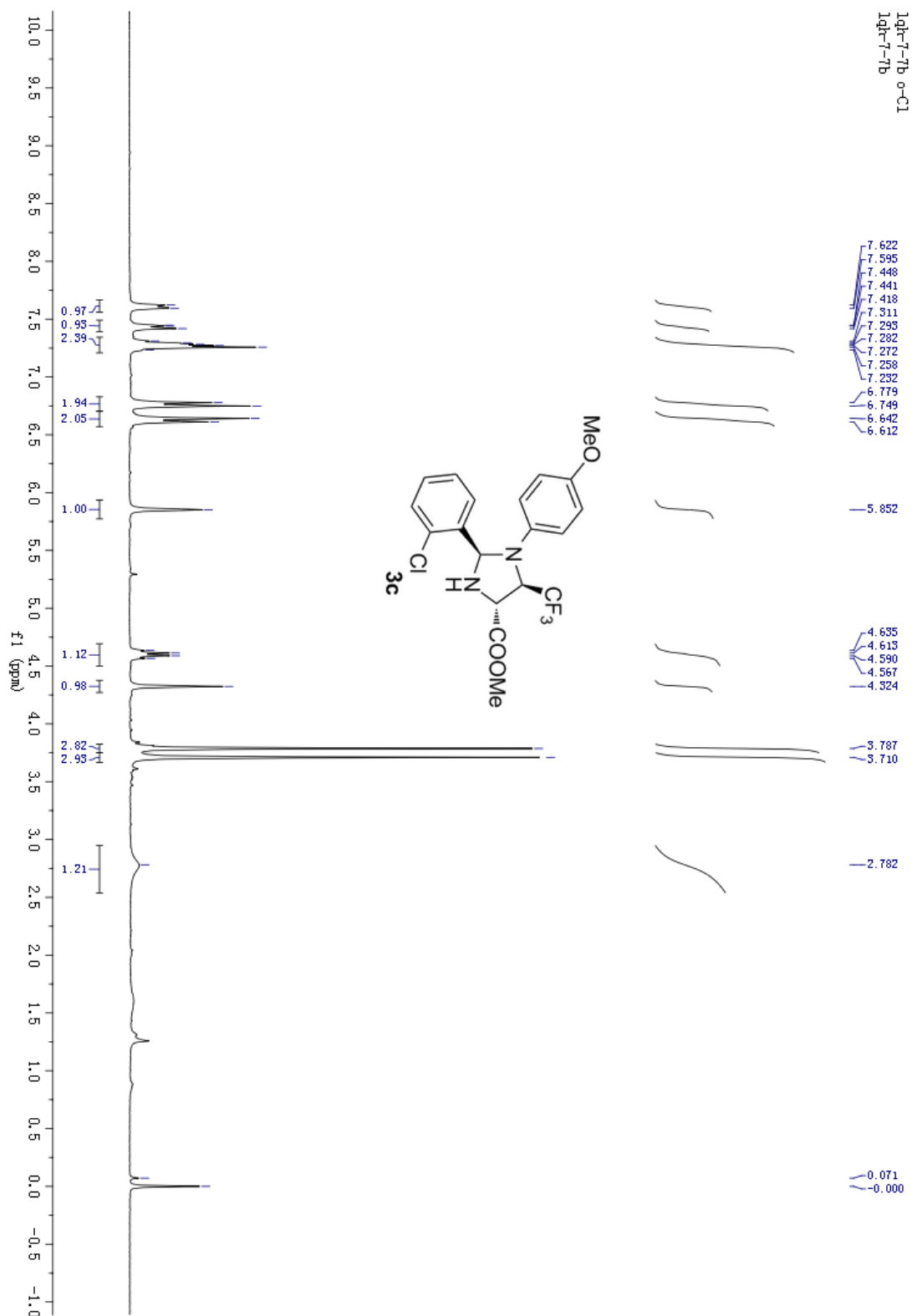
IX. ¹H NMR and ¹³C NMR Spectra.

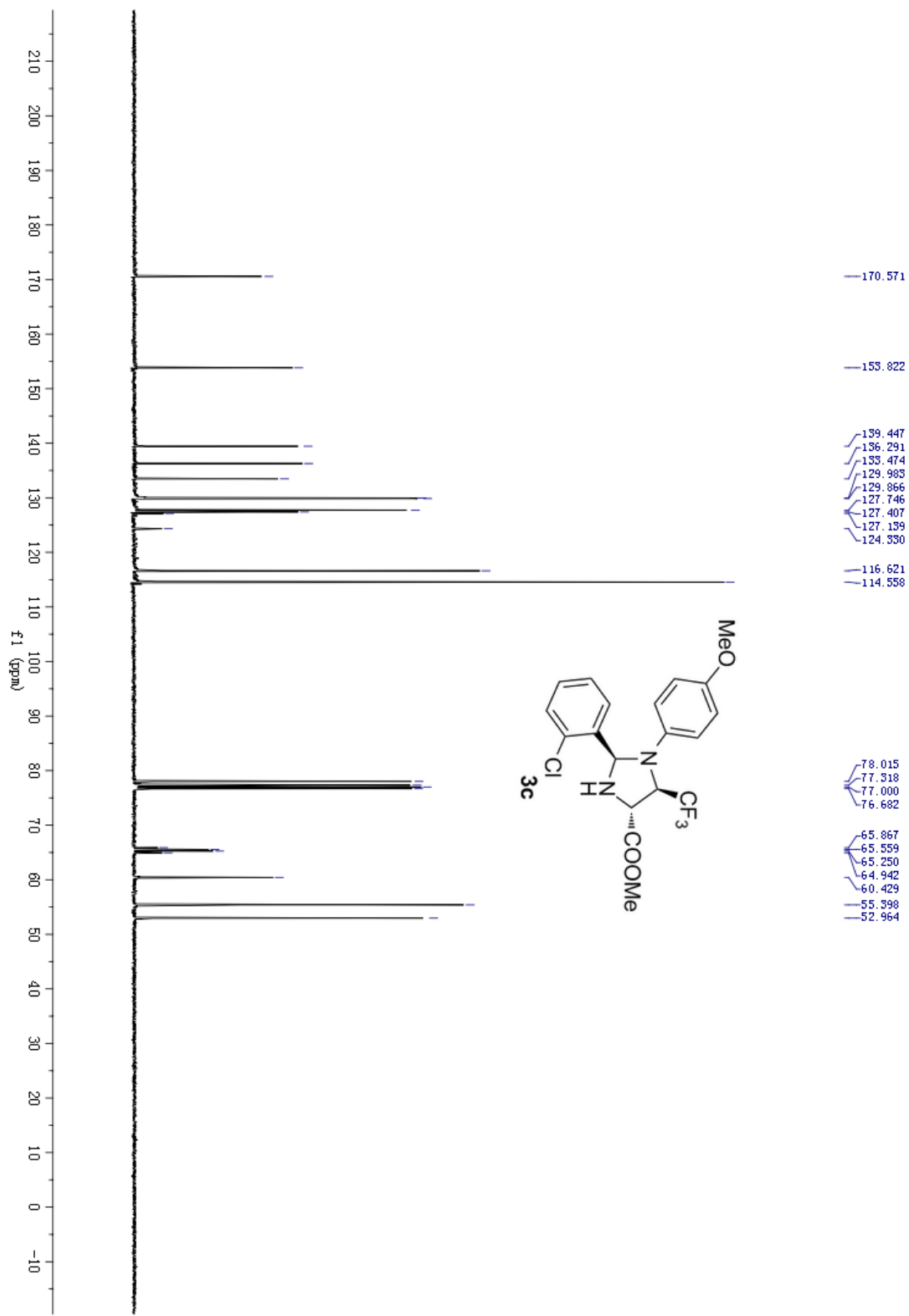


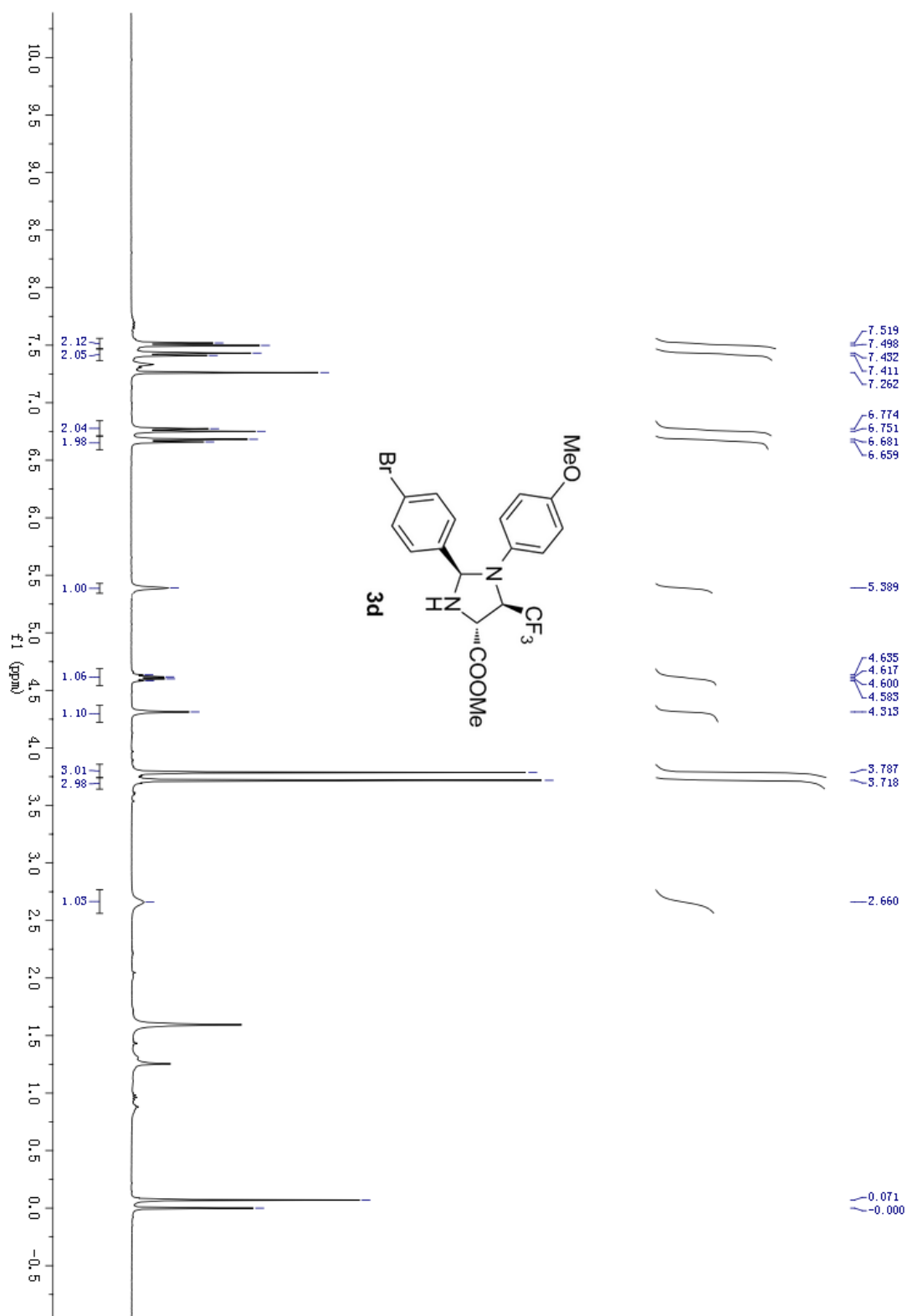


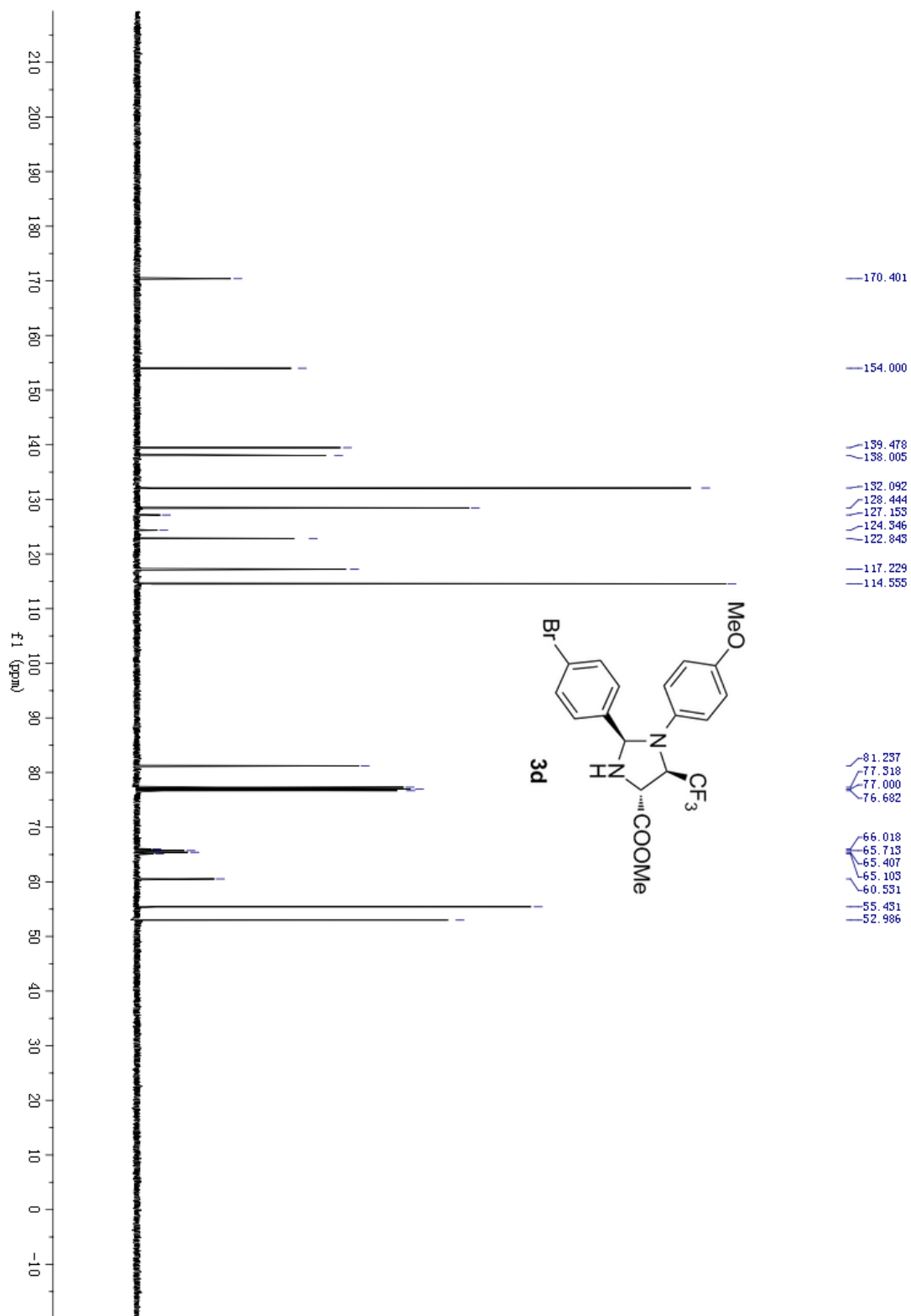


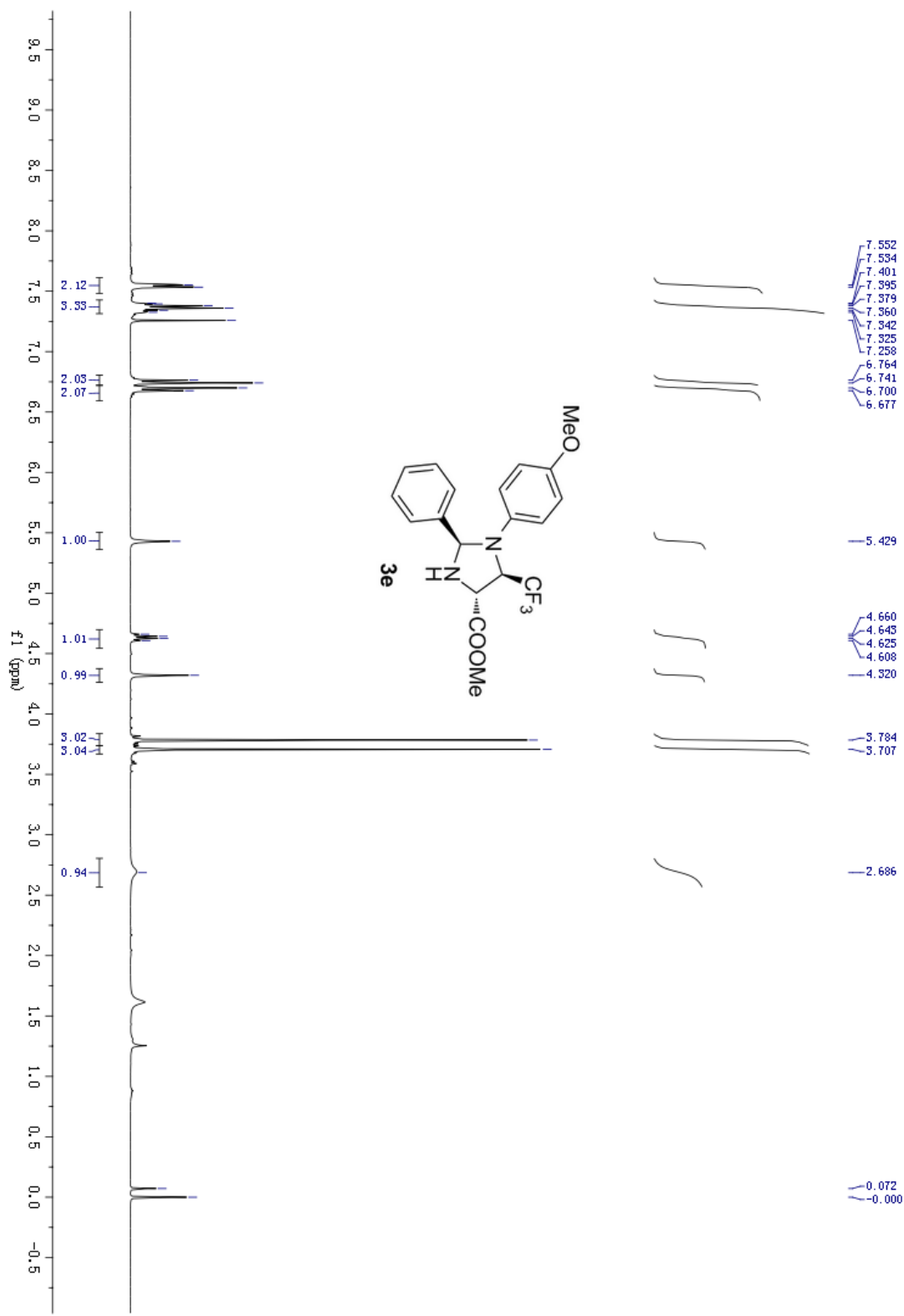


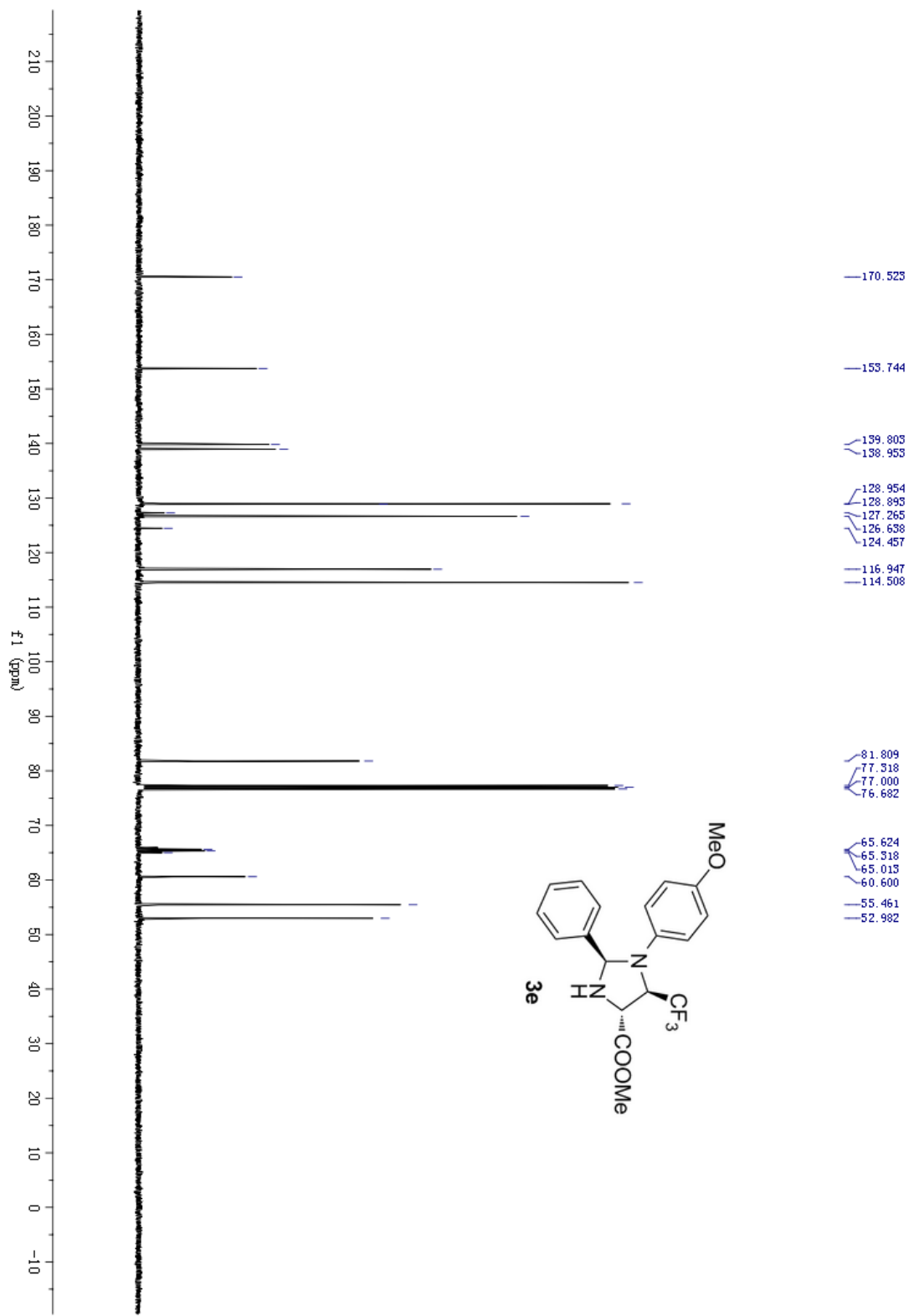


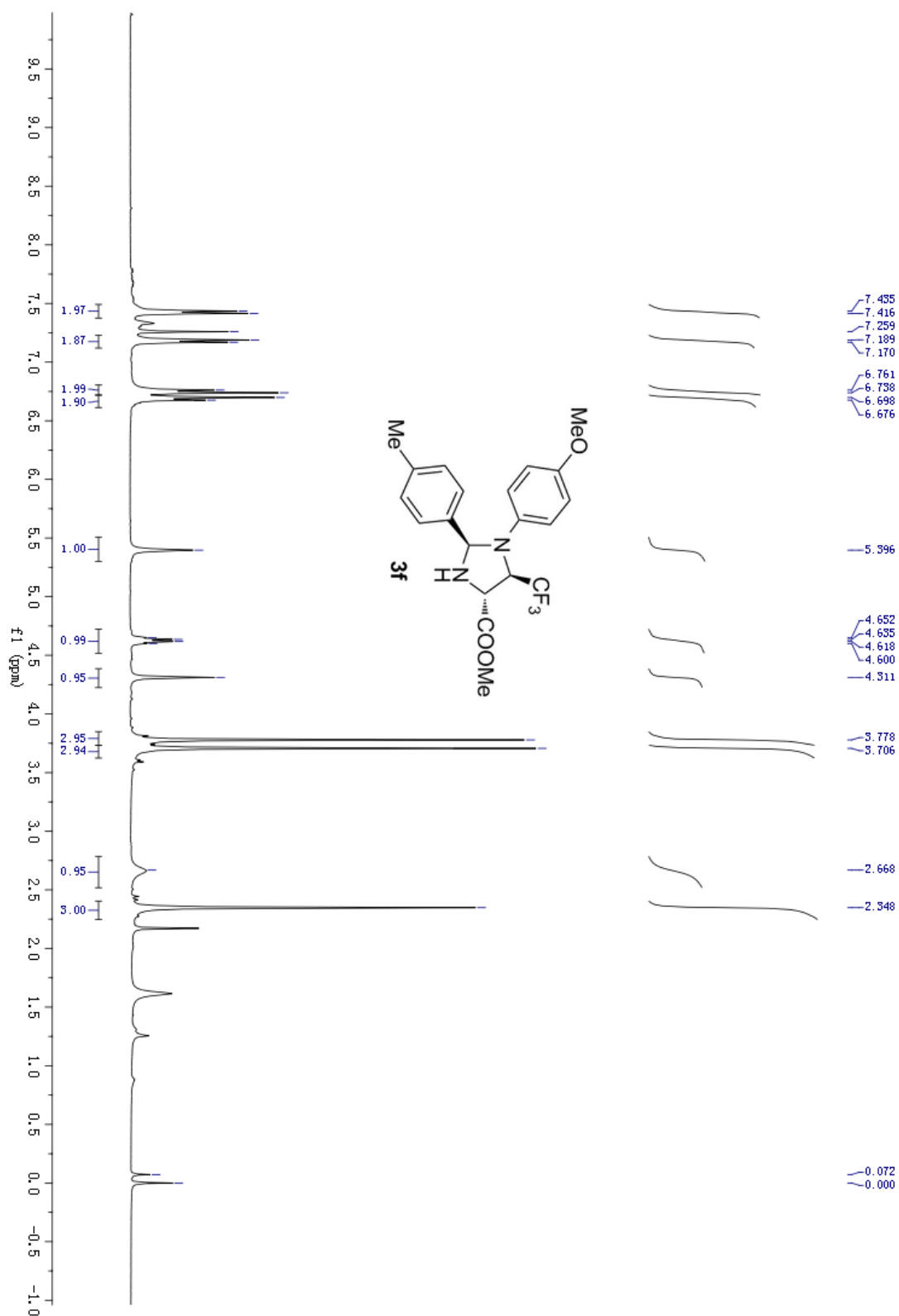


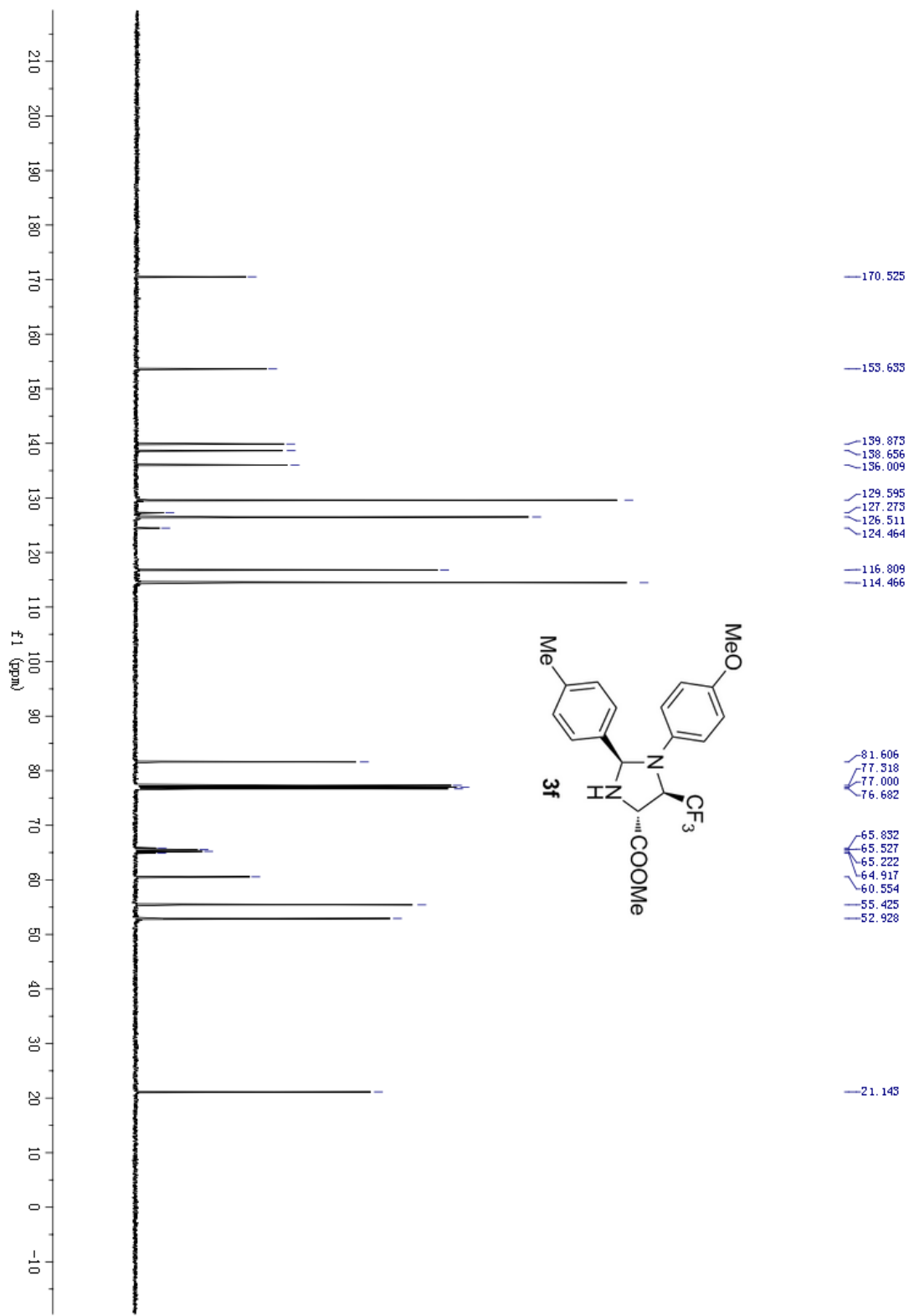


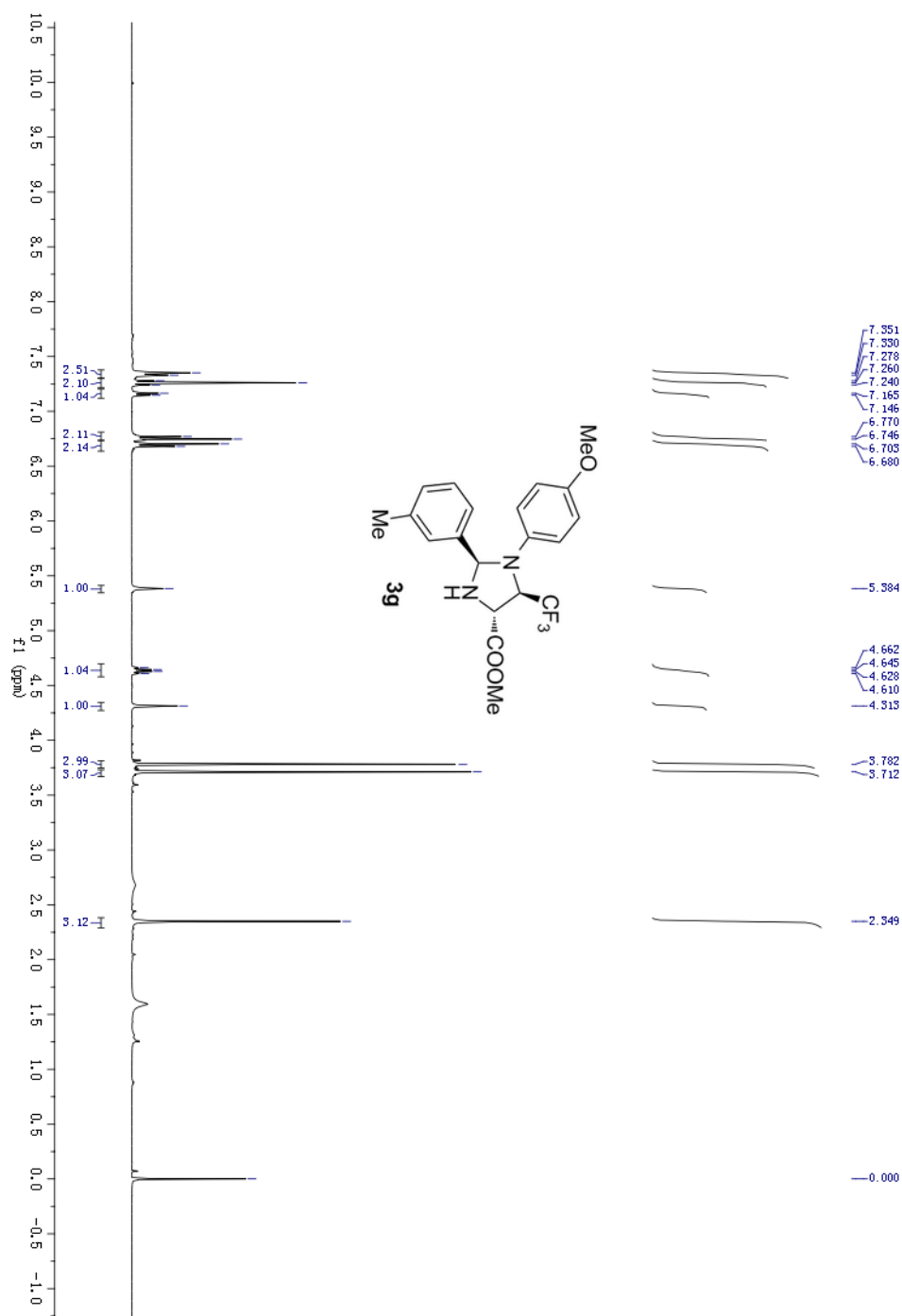


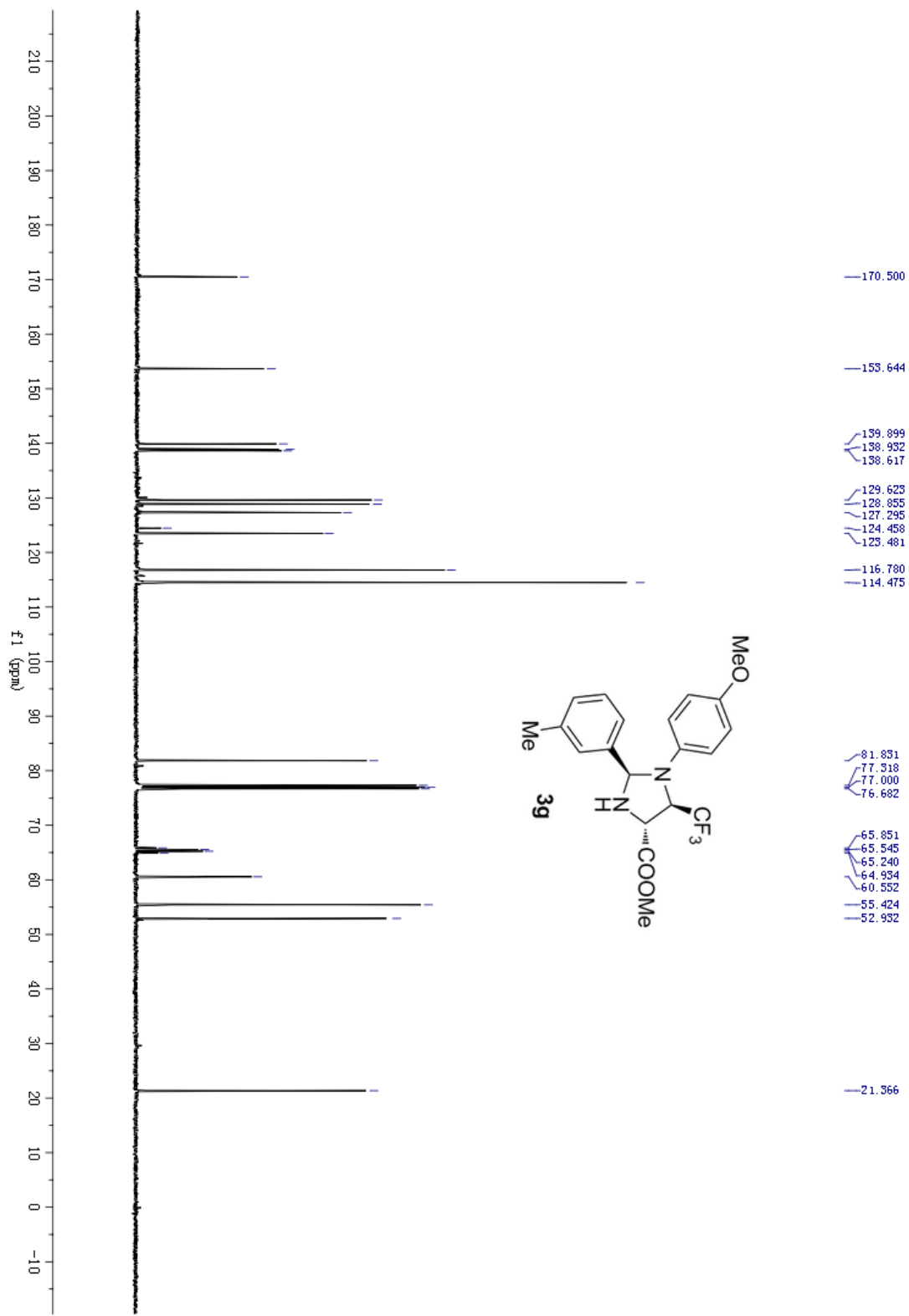


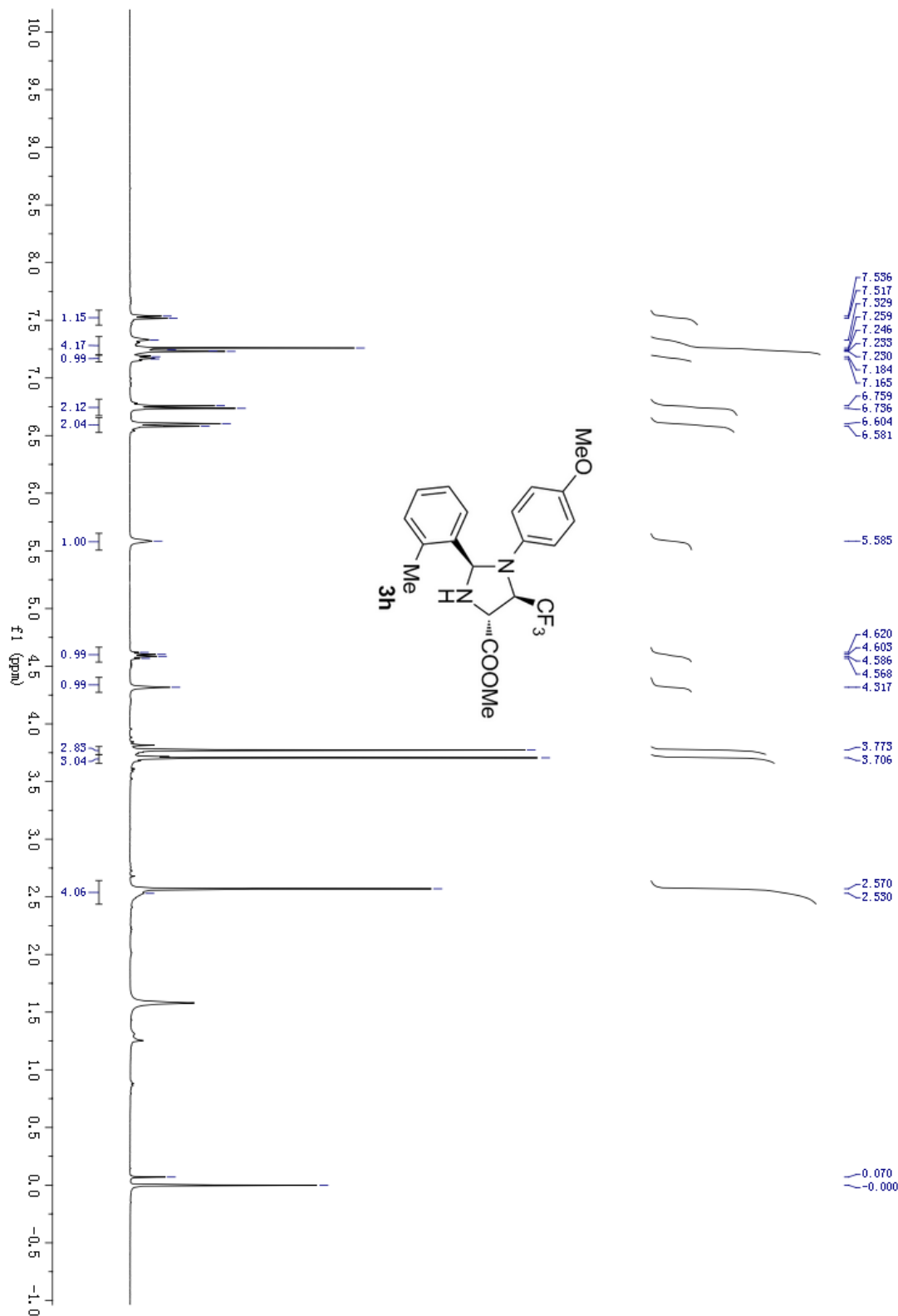


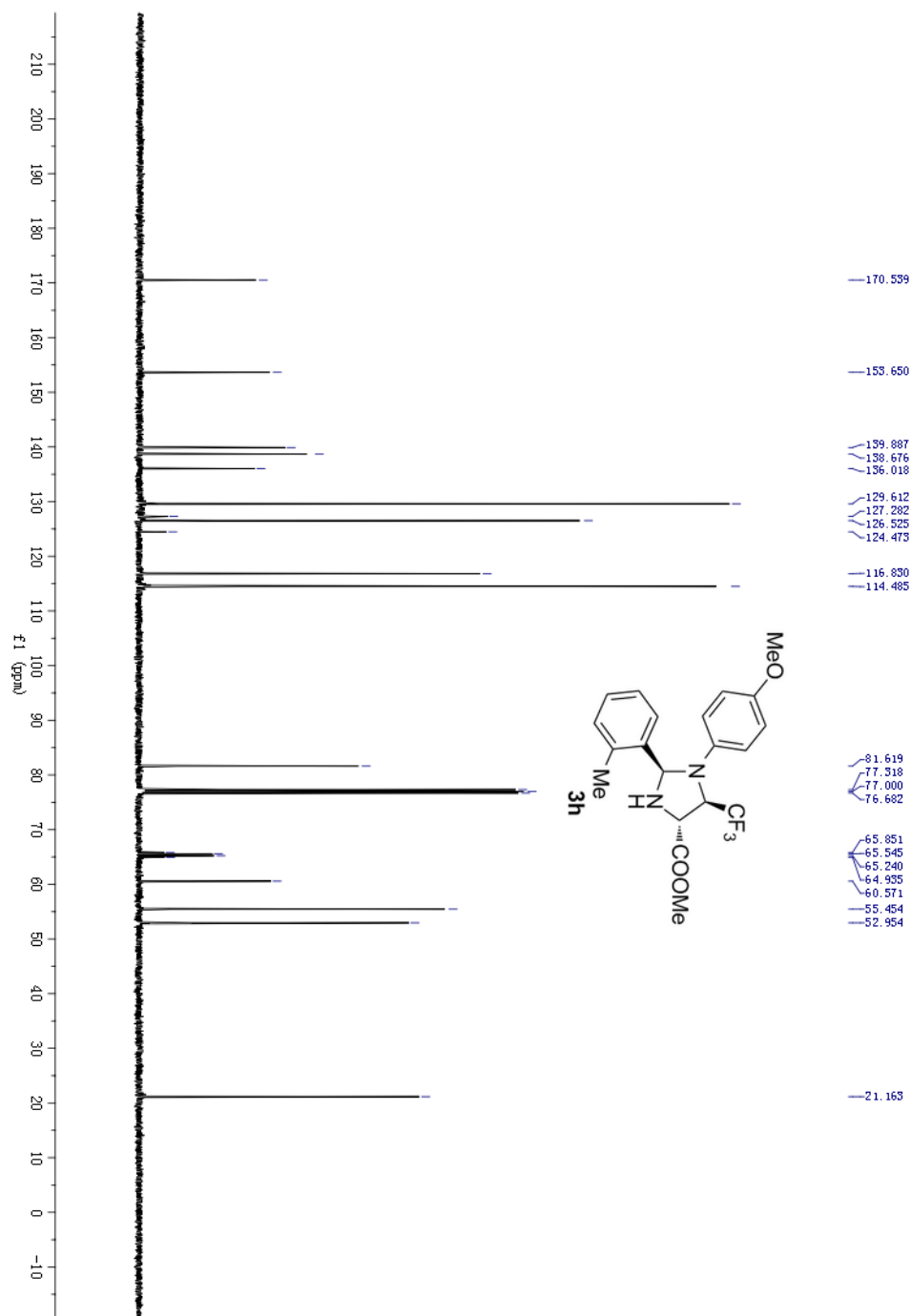


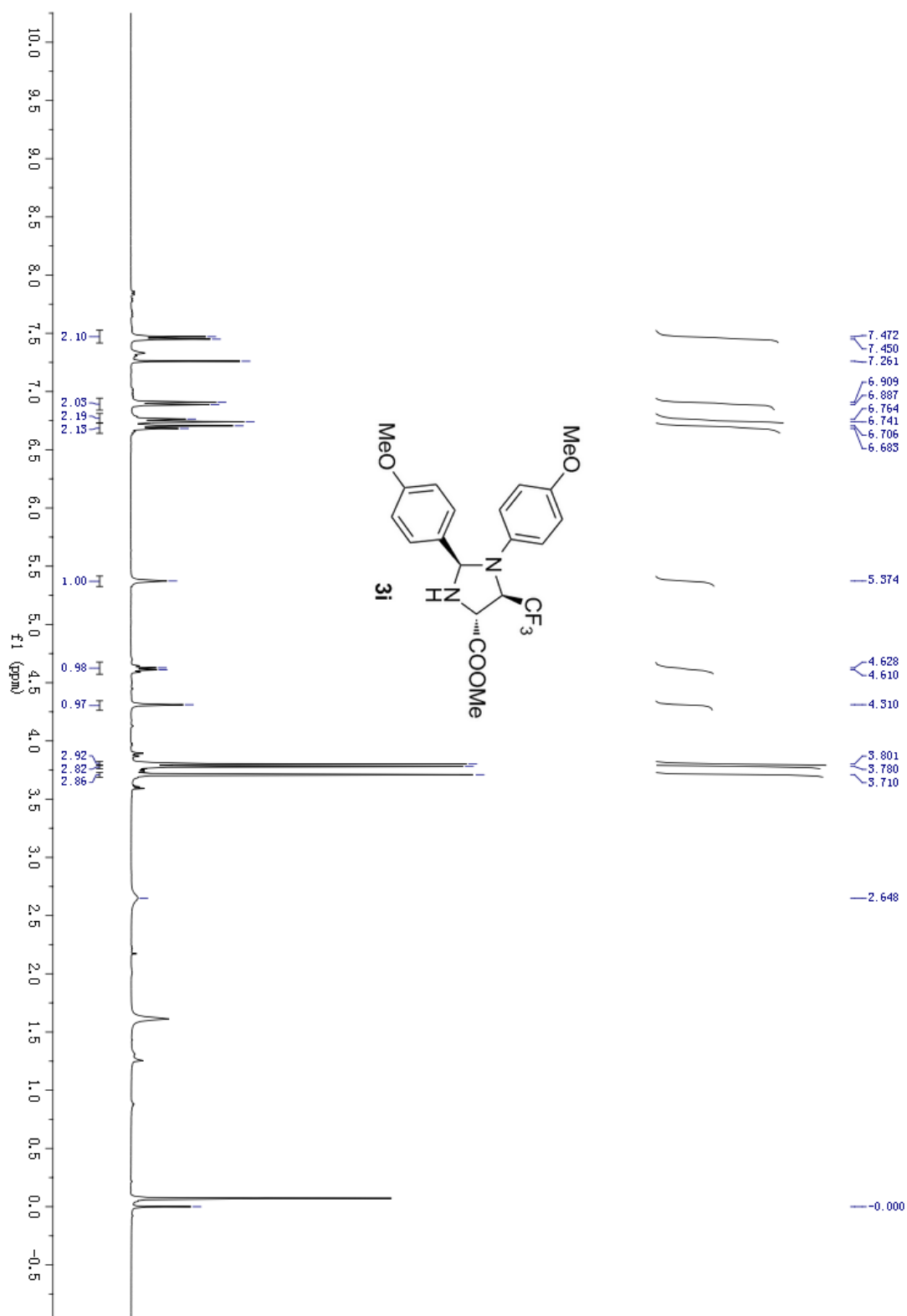


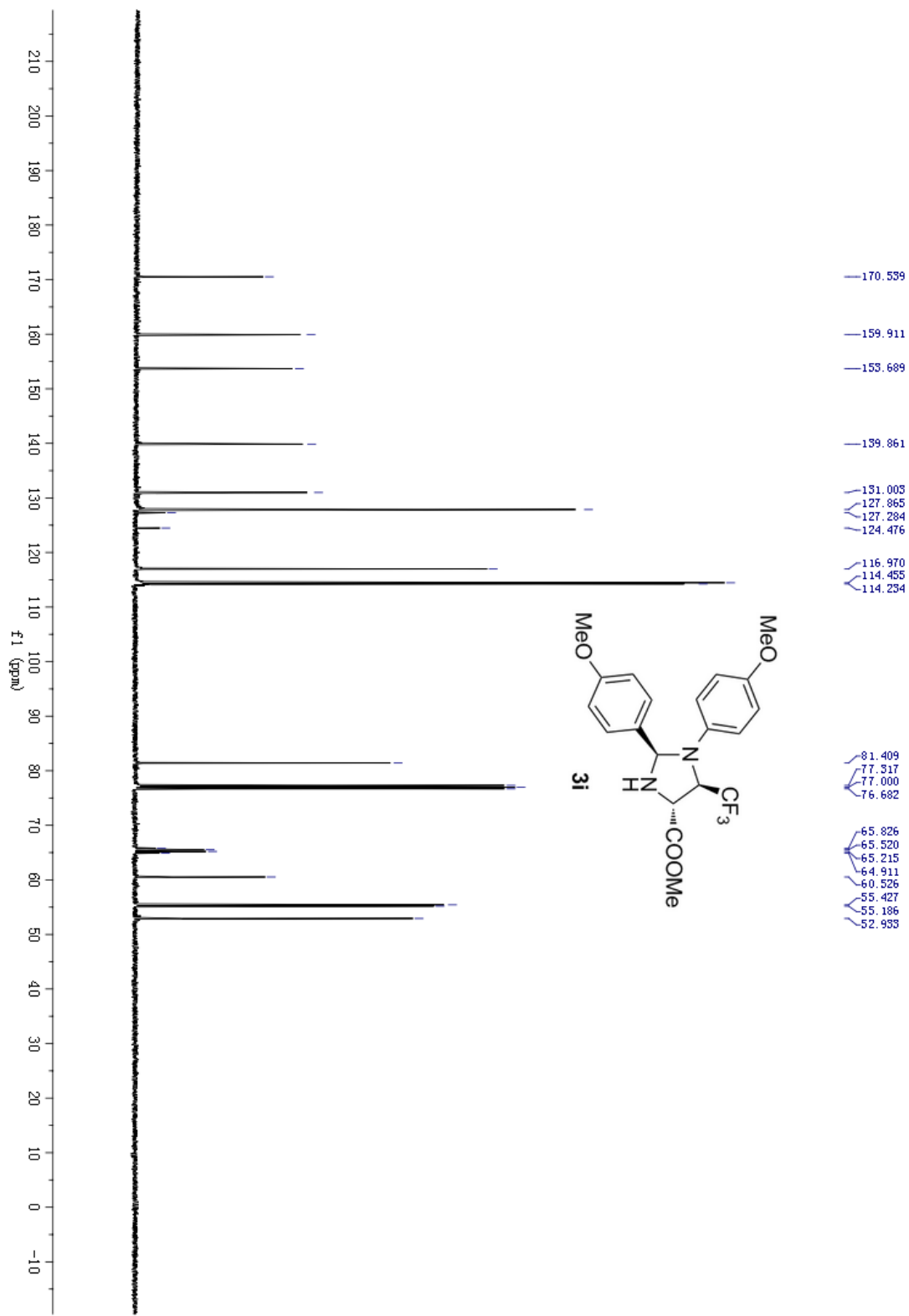


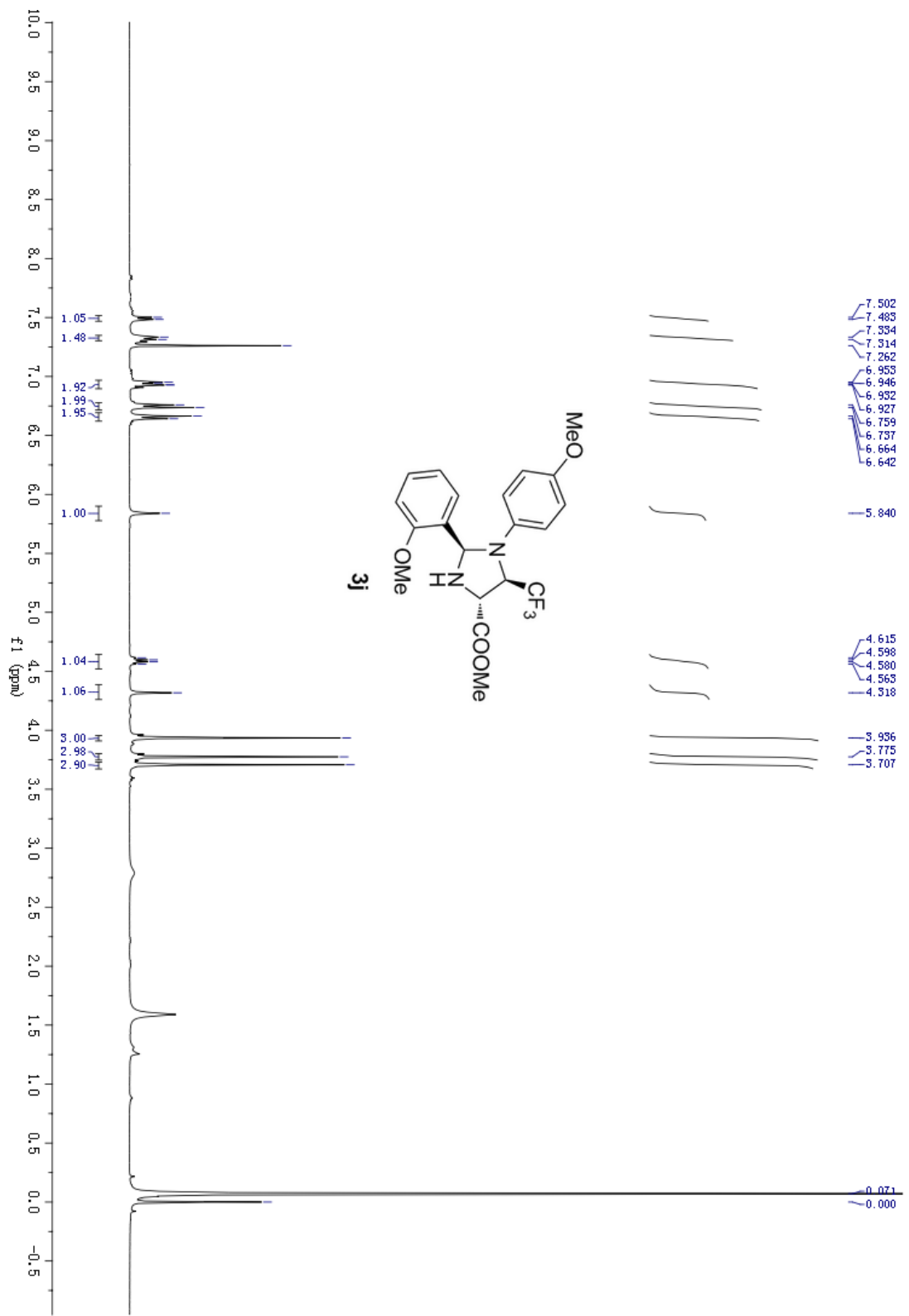


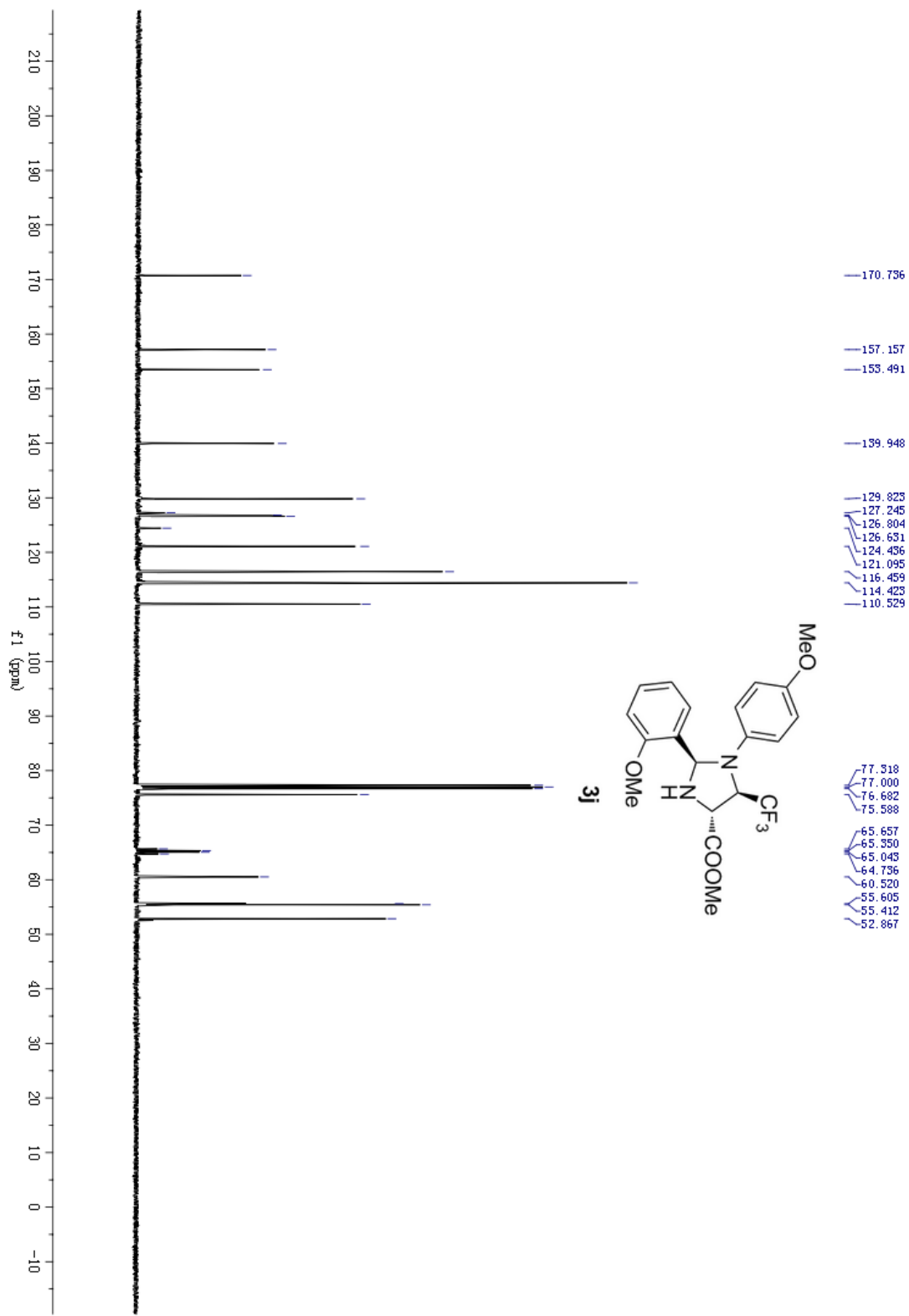


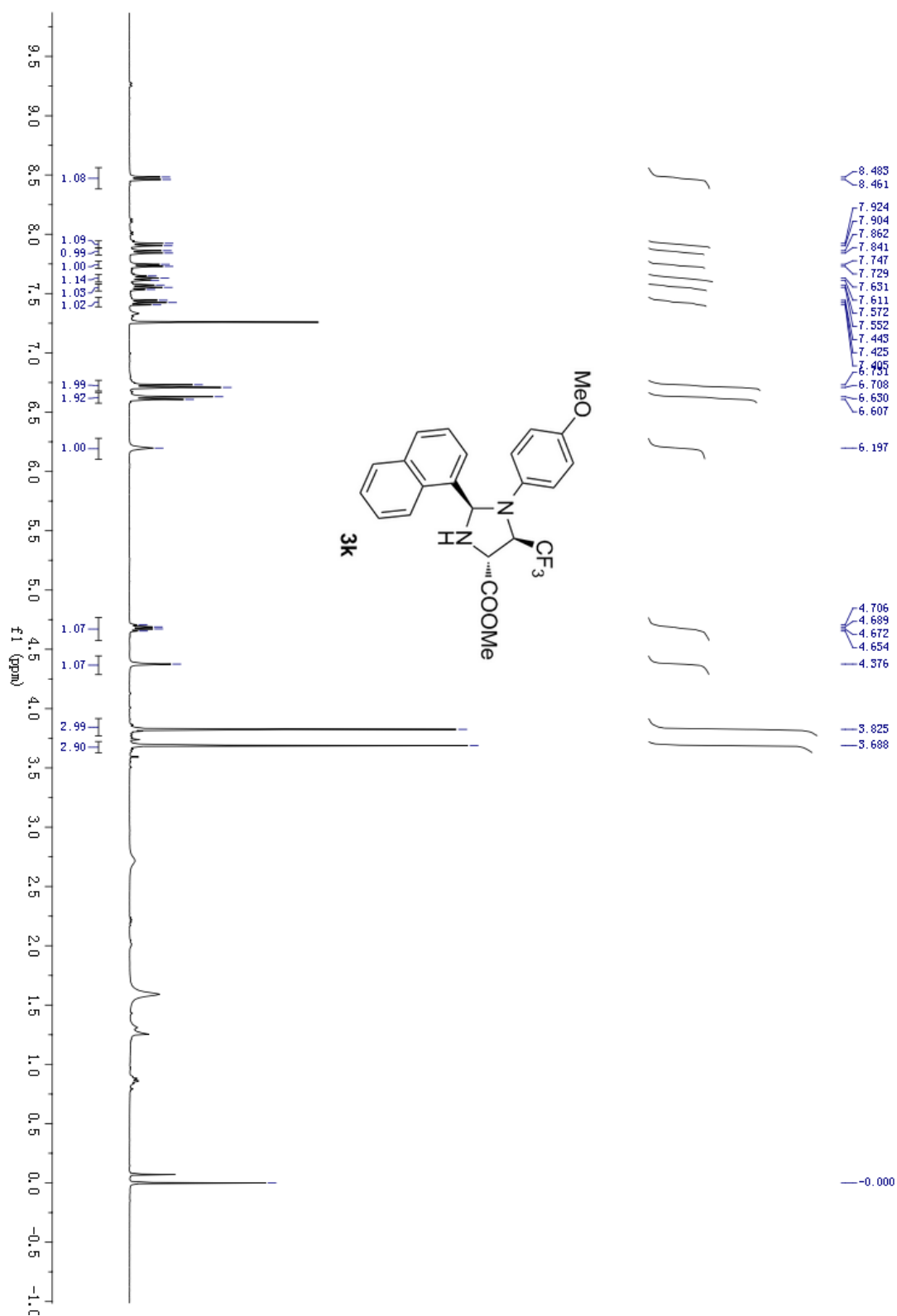


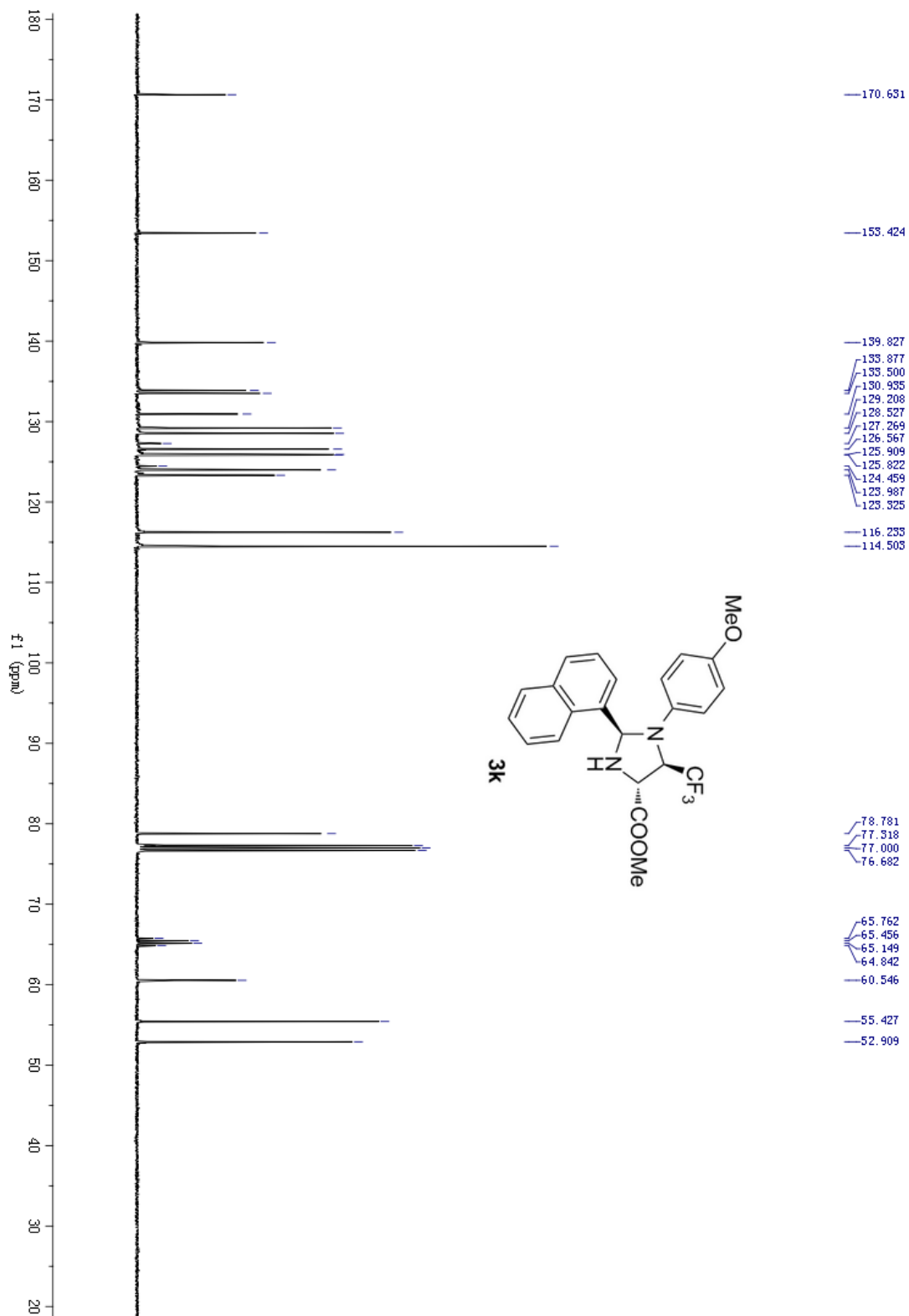


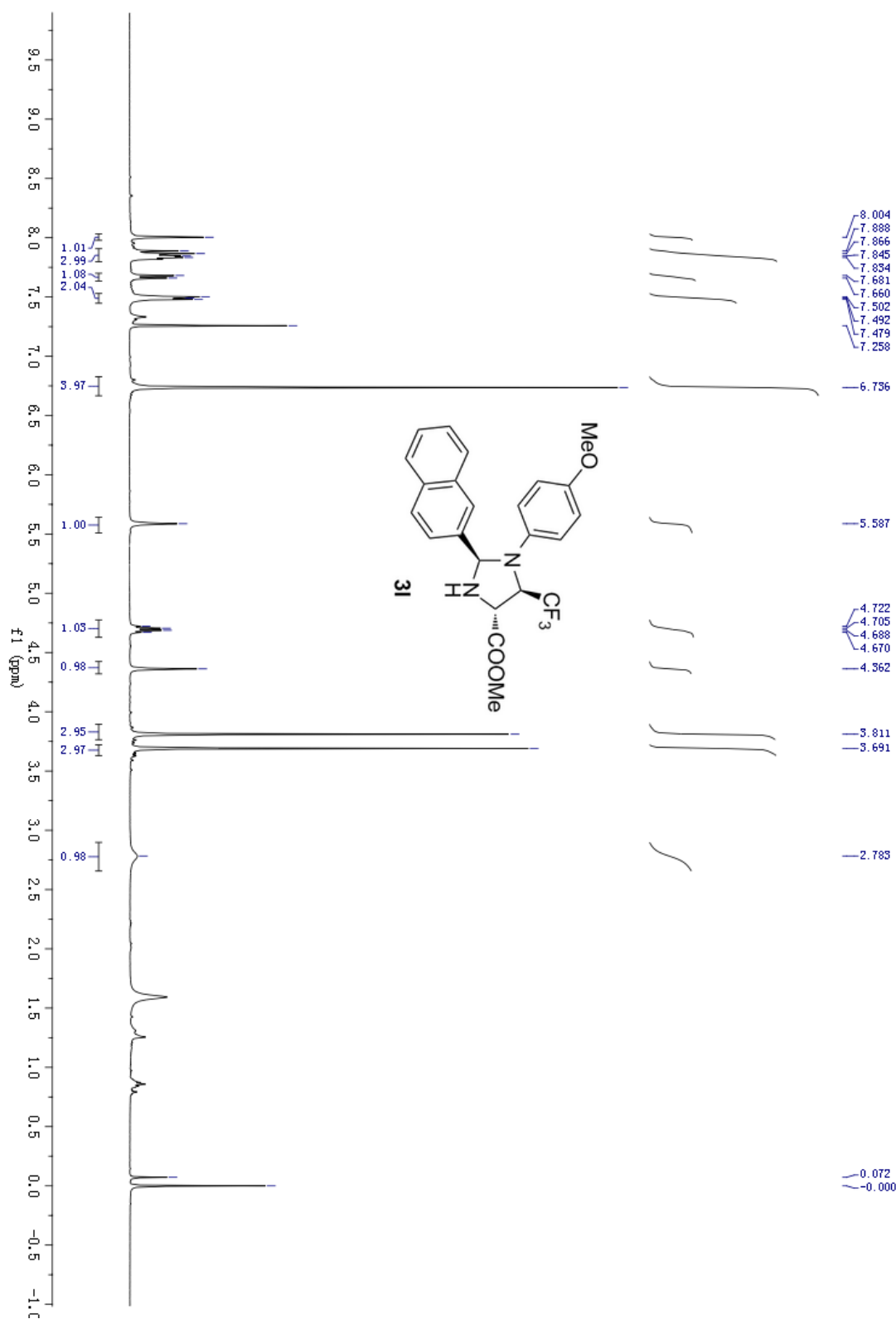


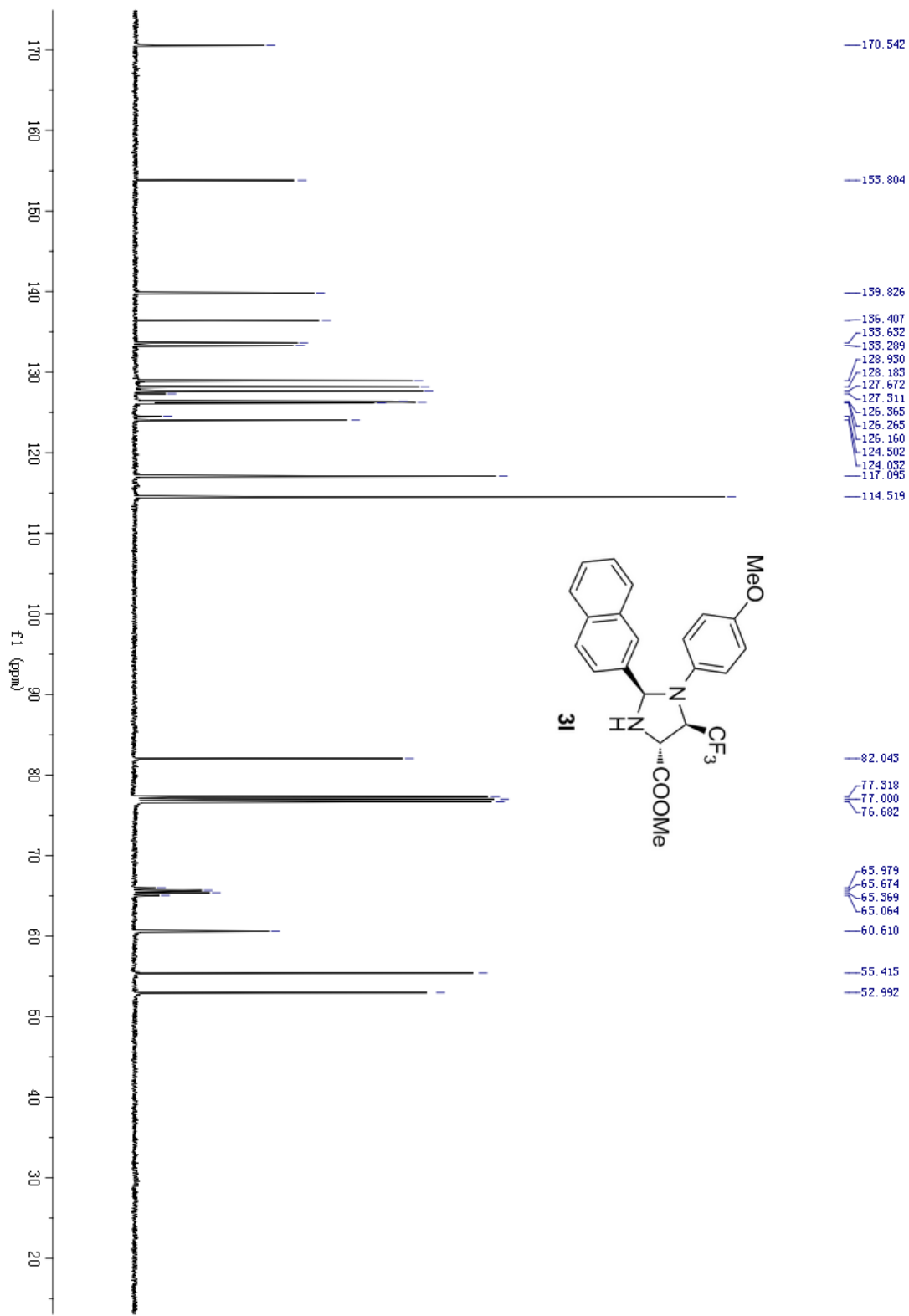


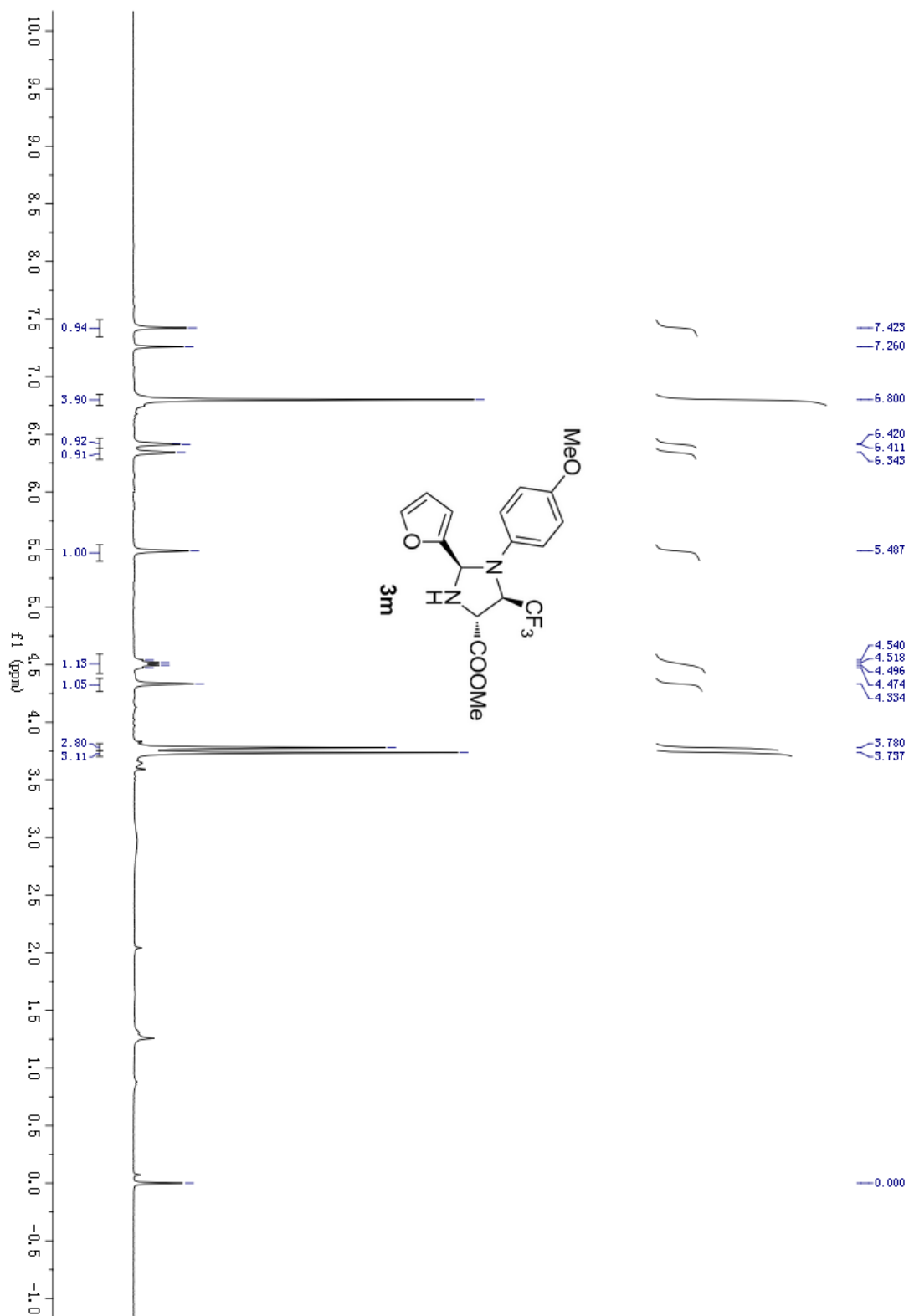


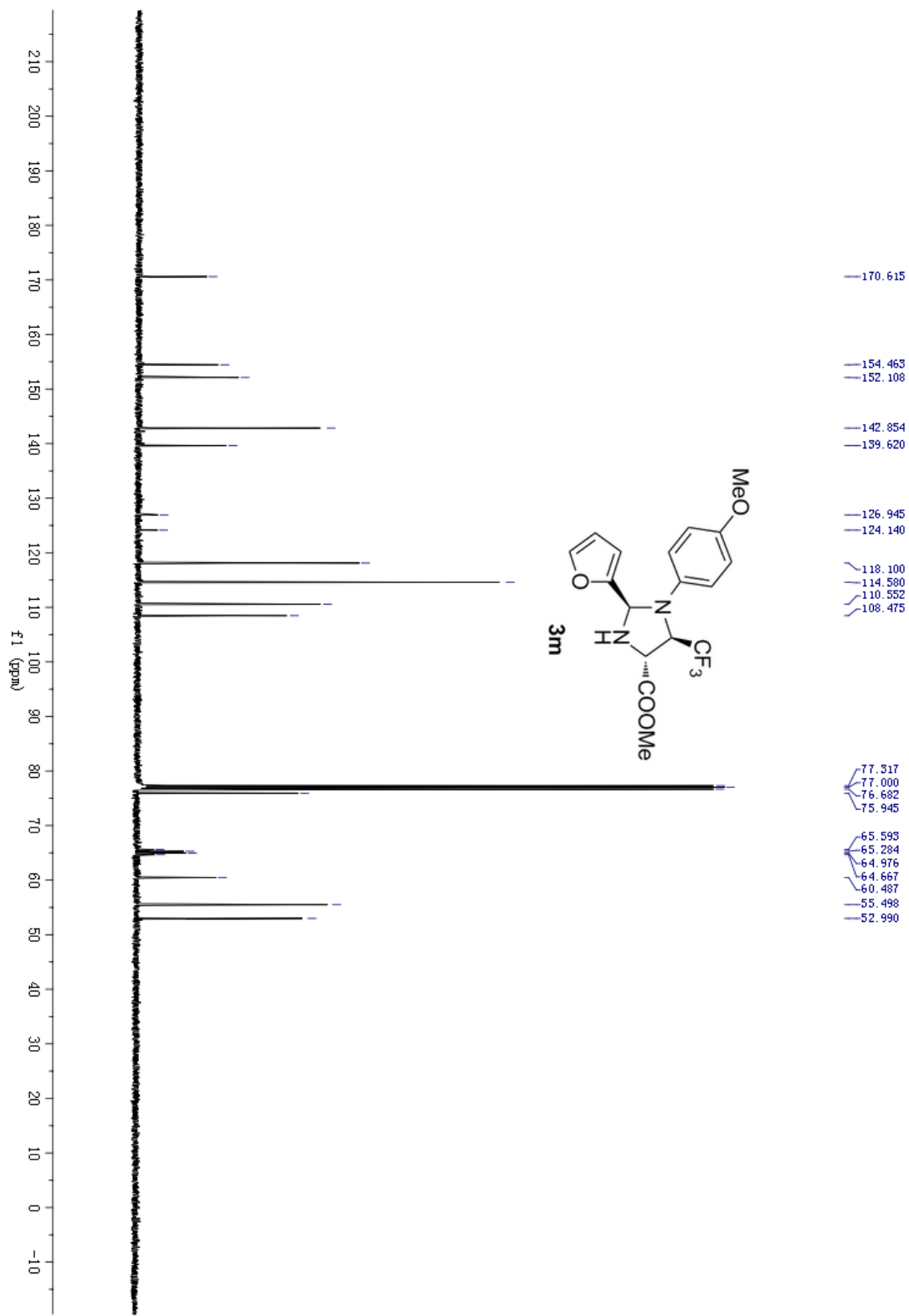


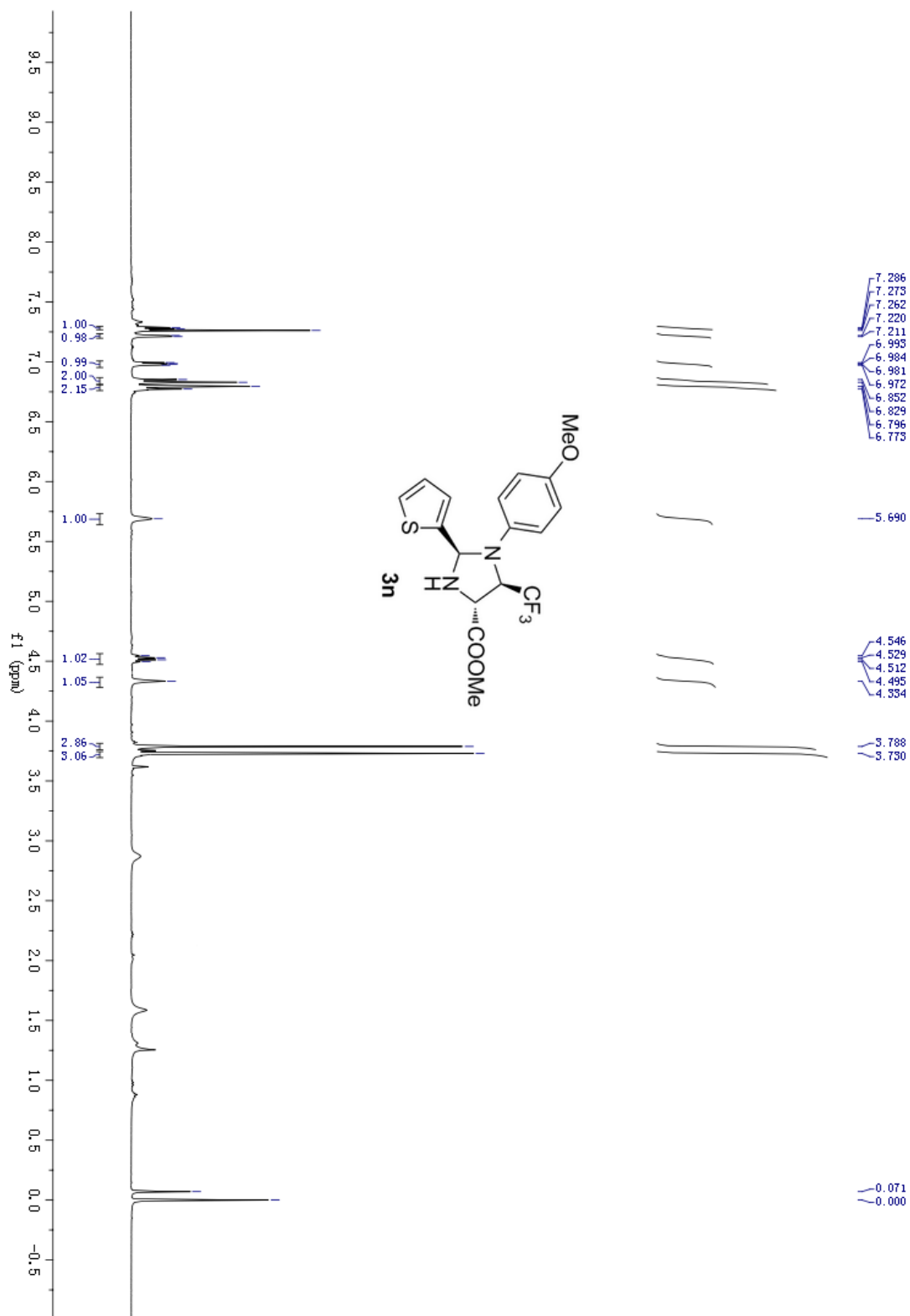


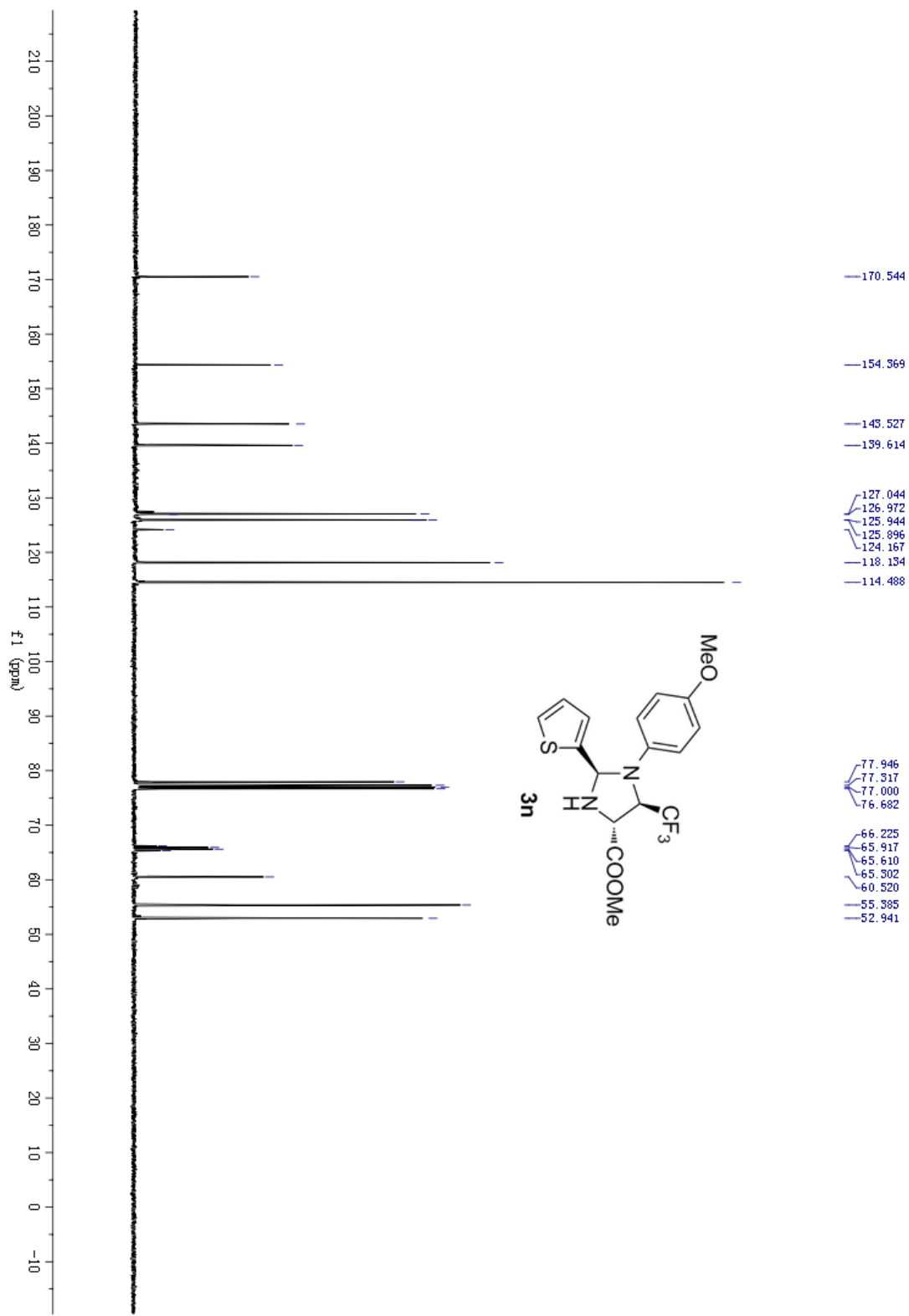


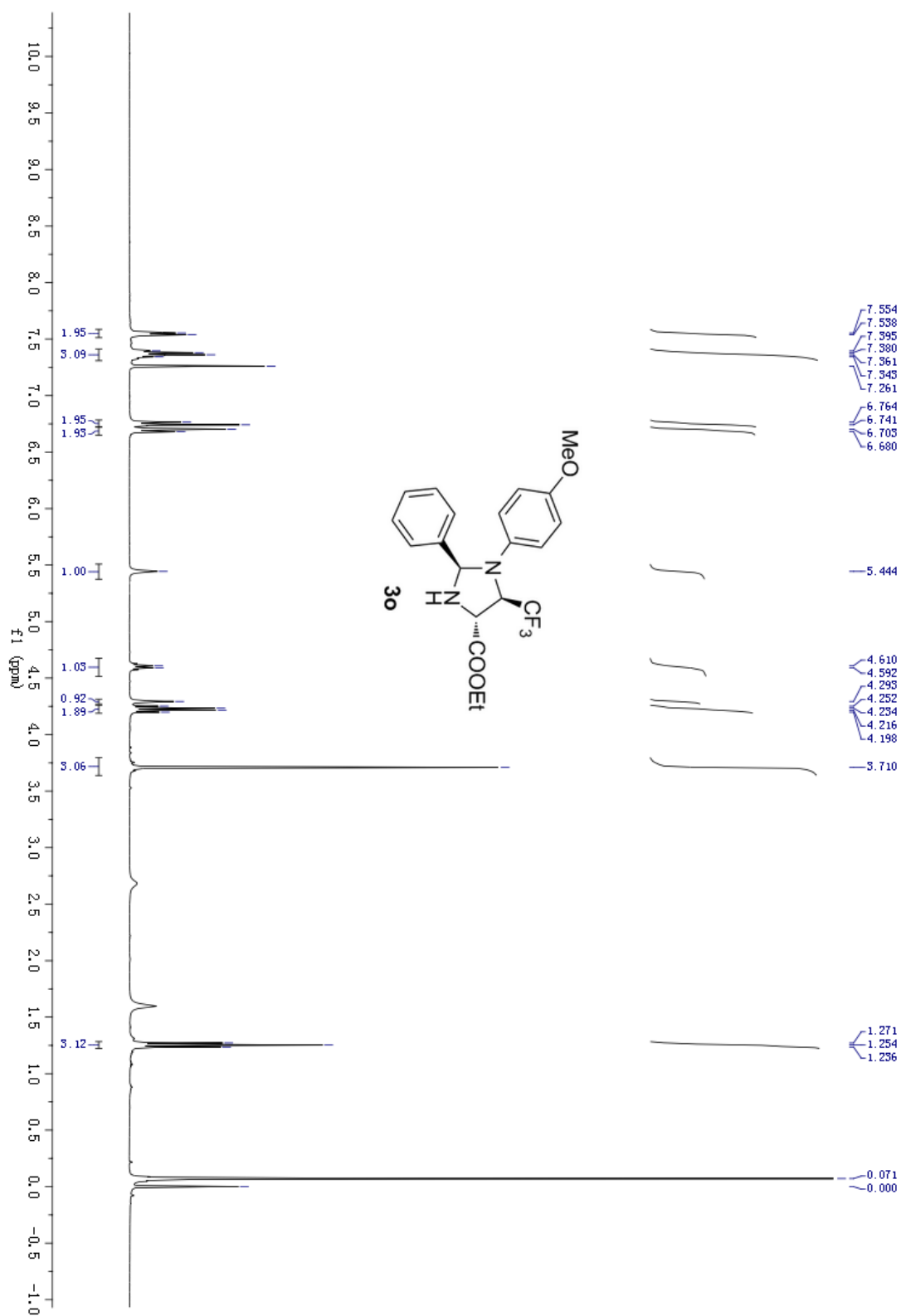


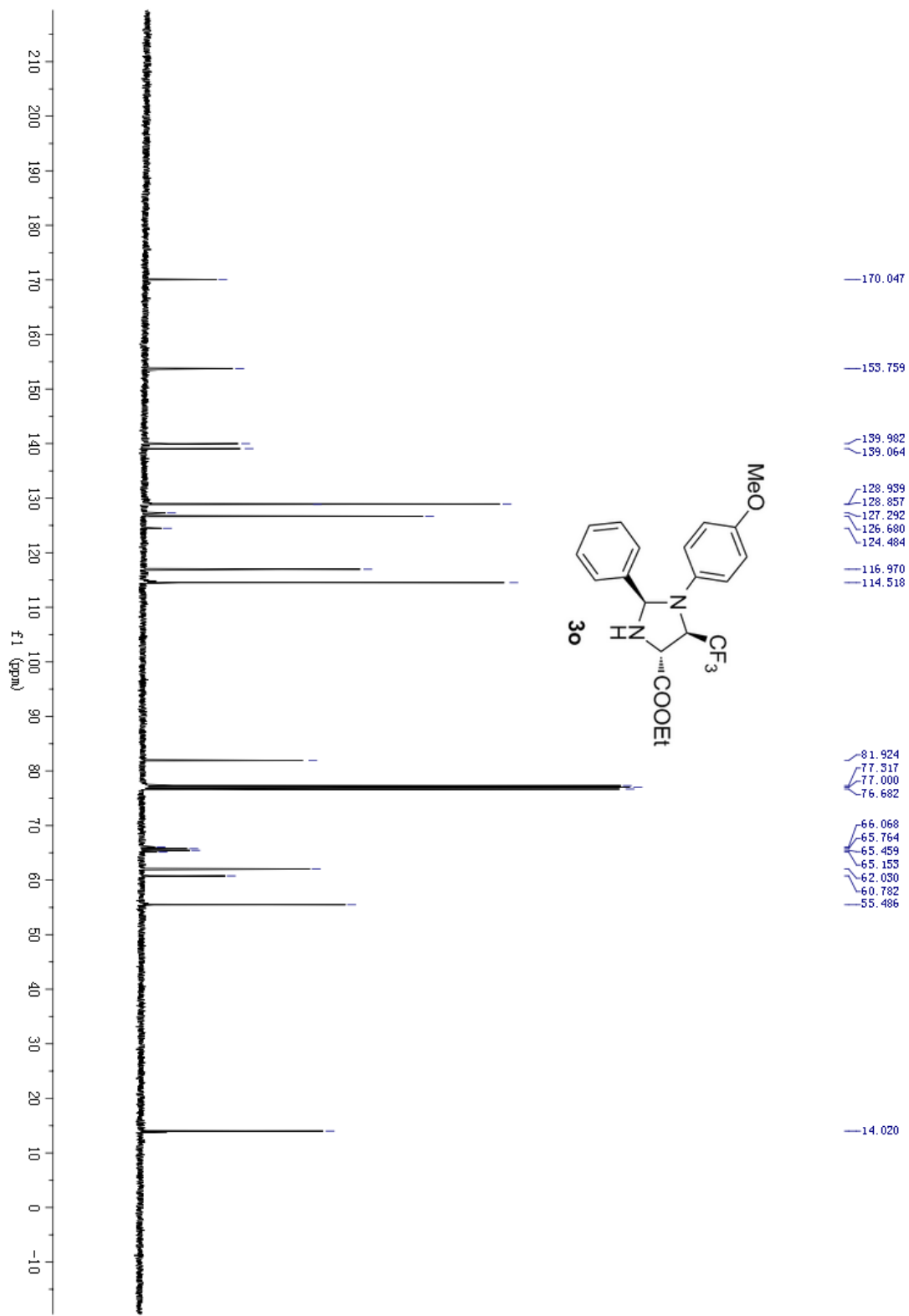


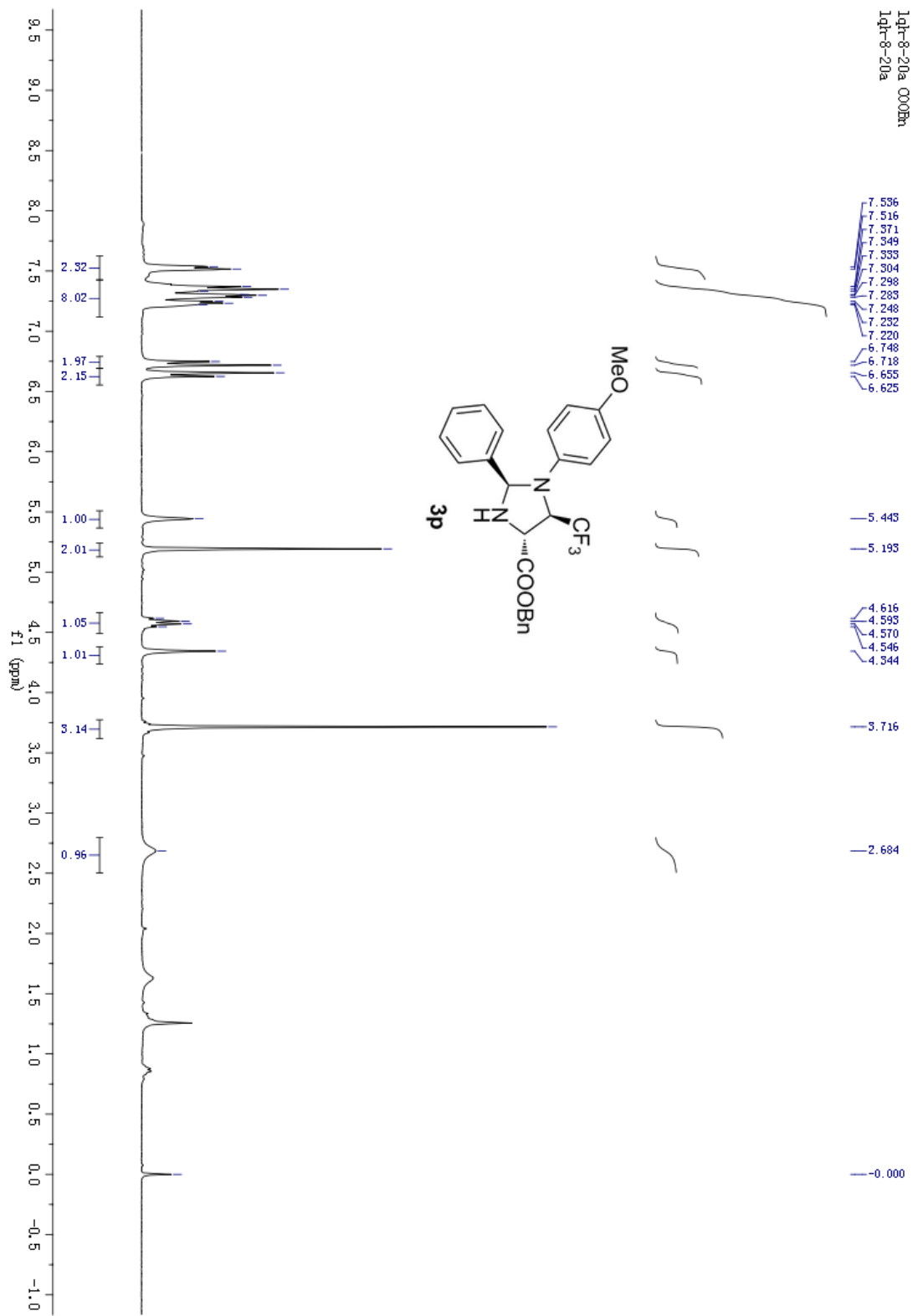


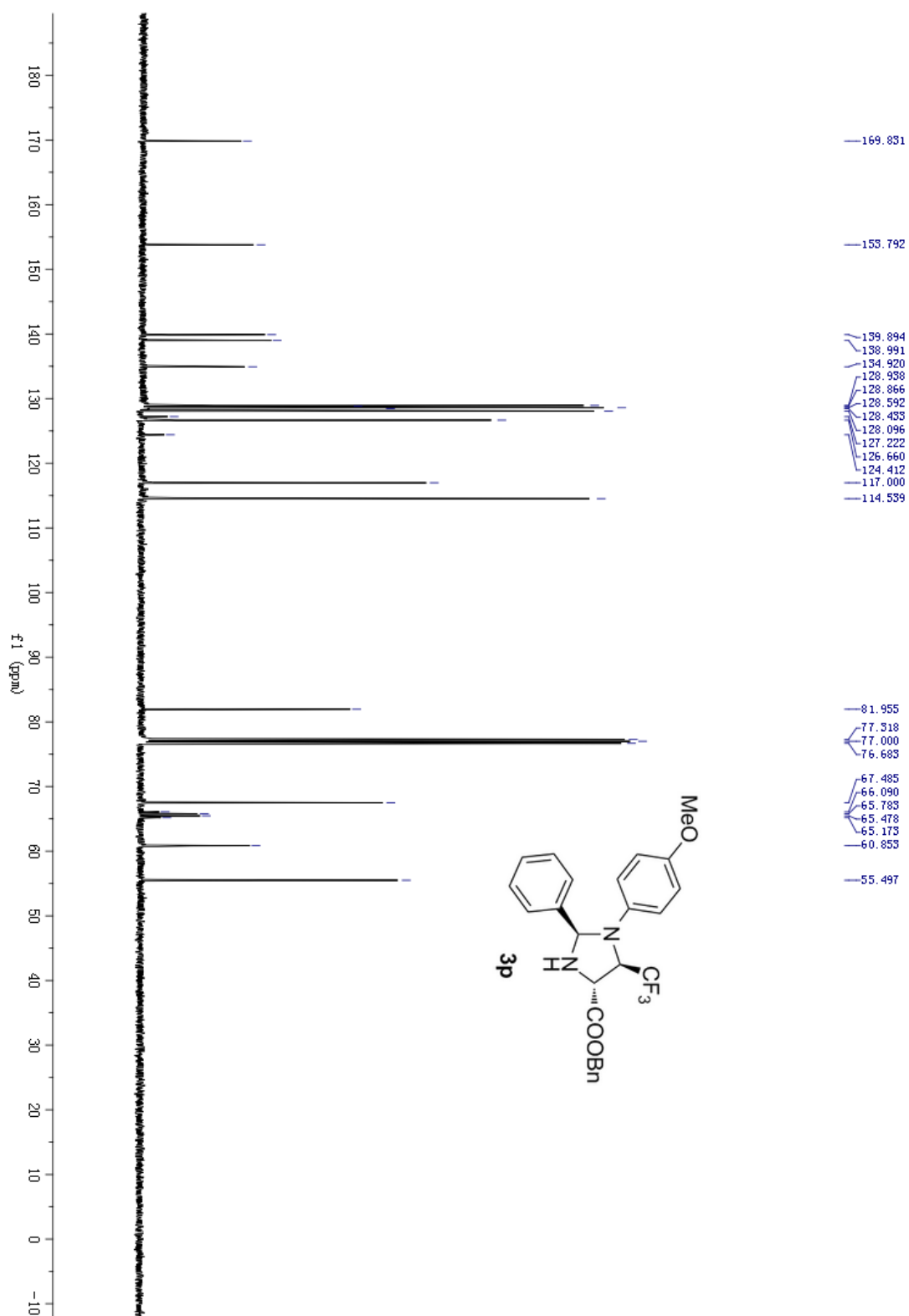


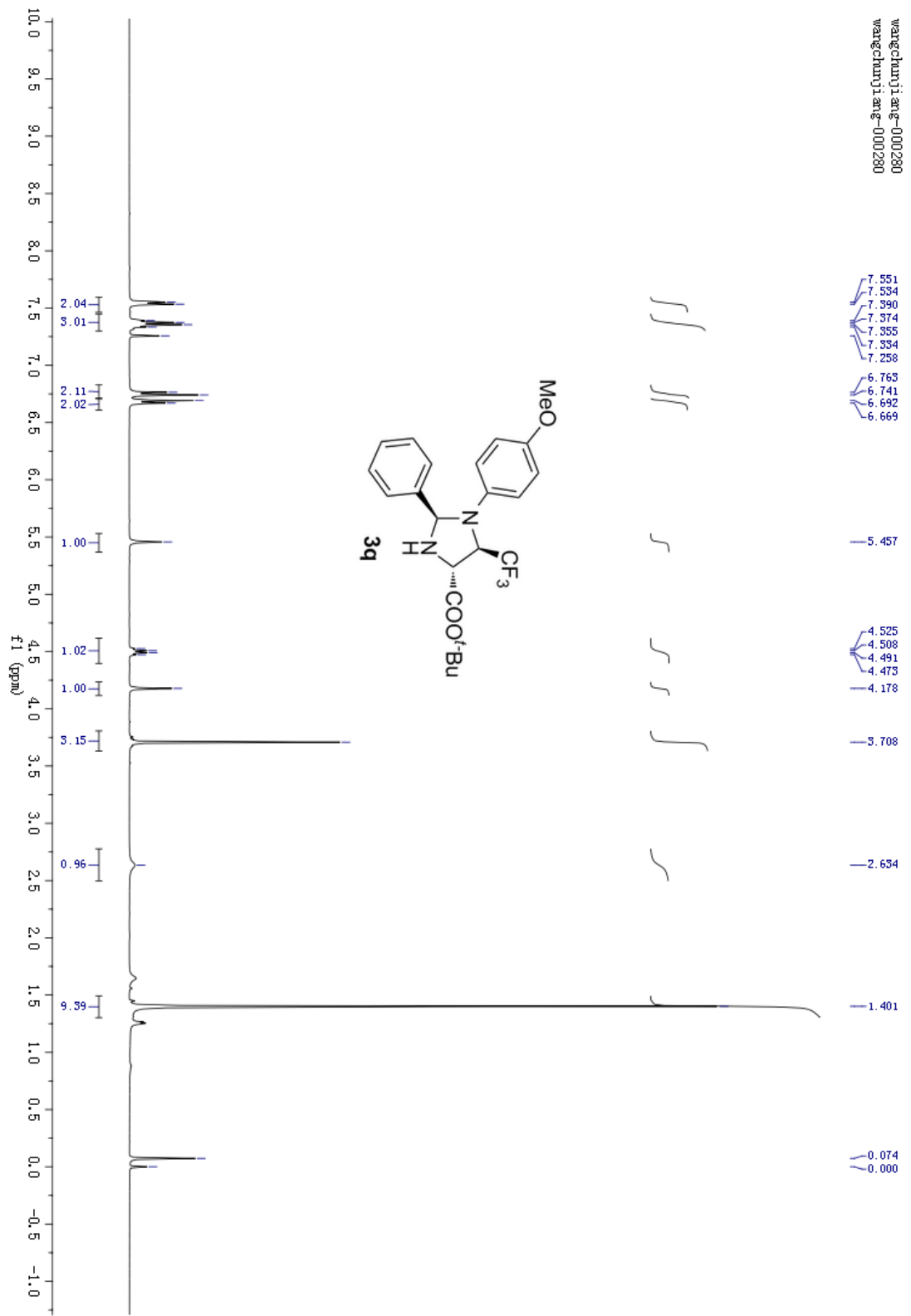


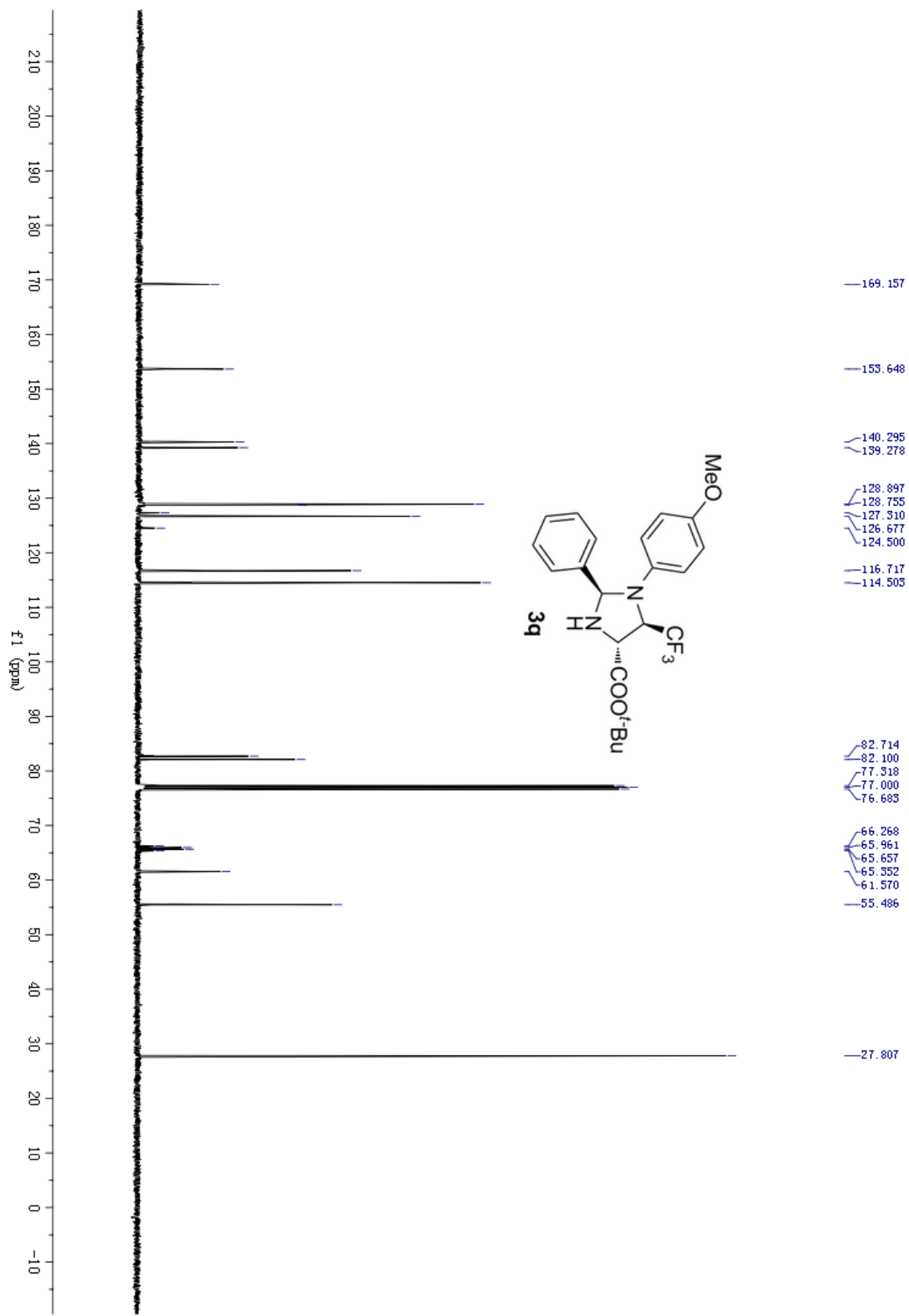


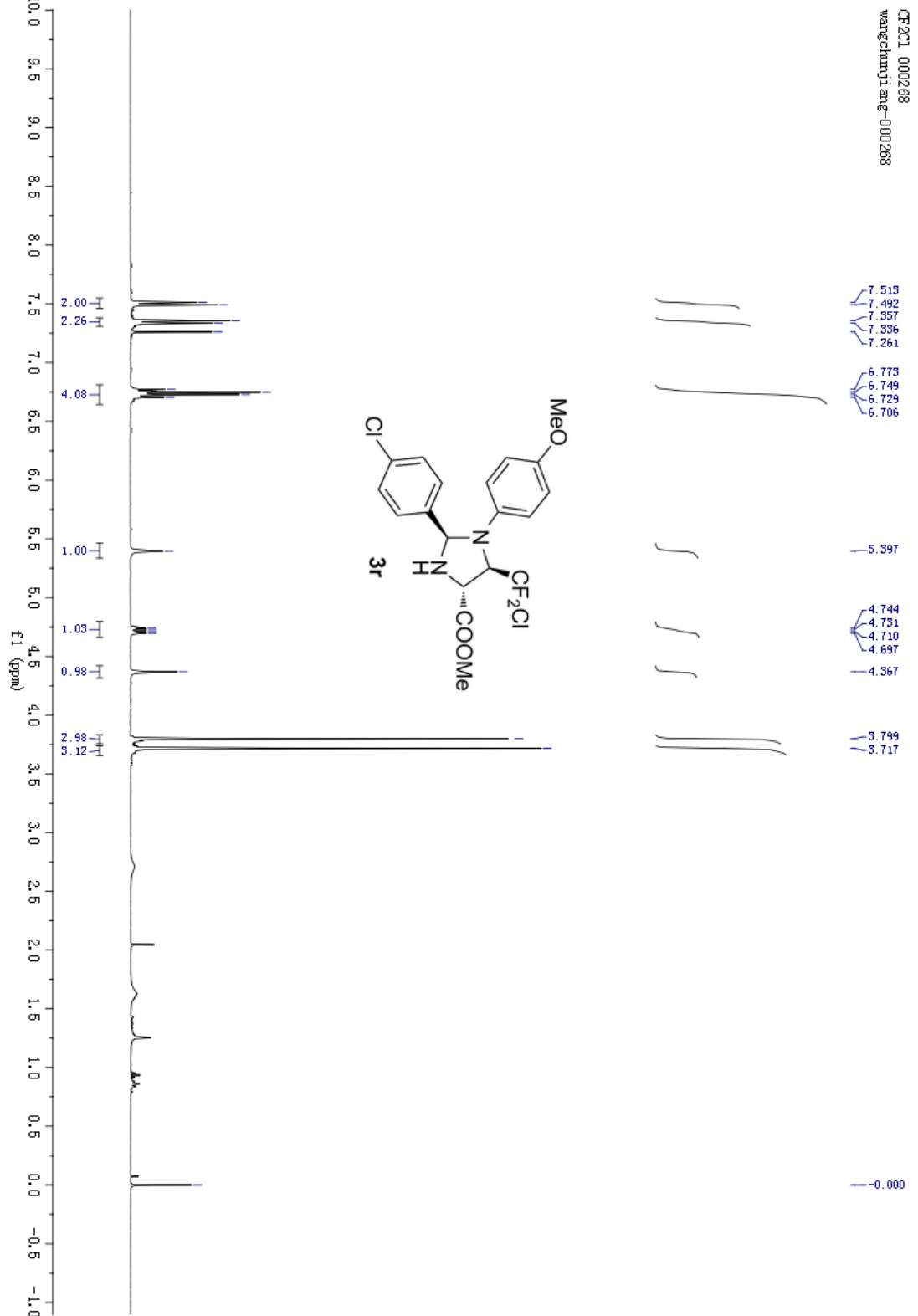


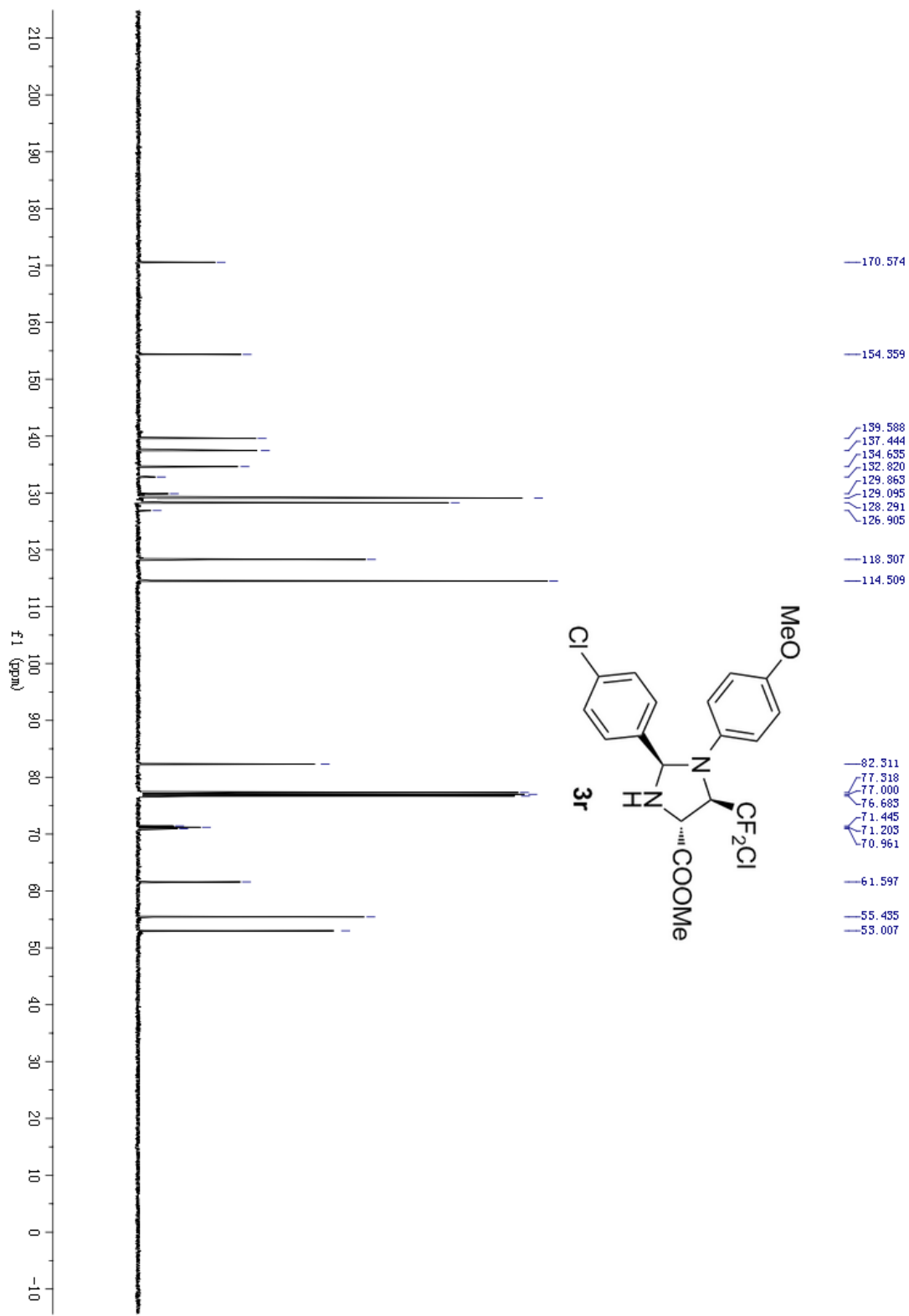


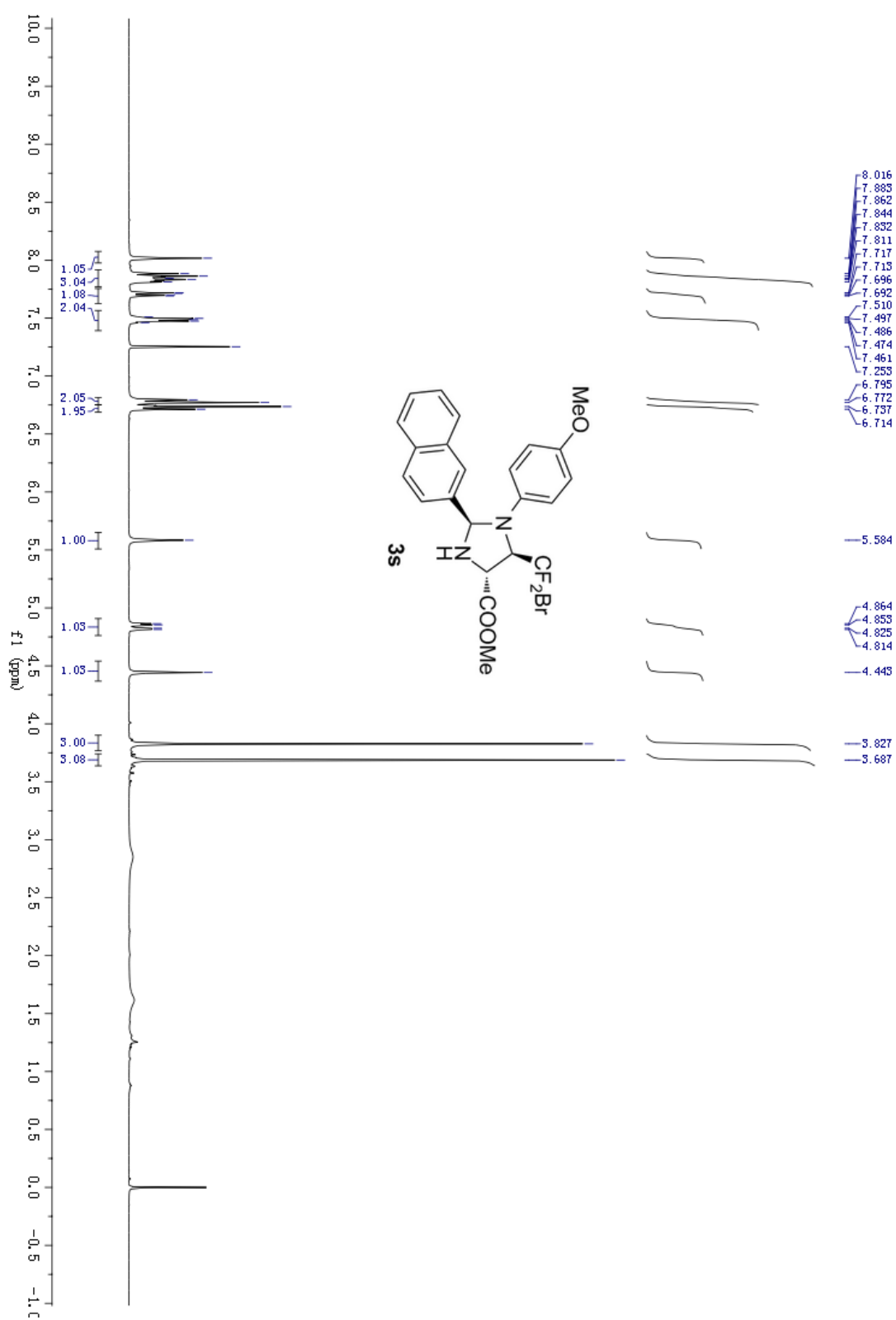


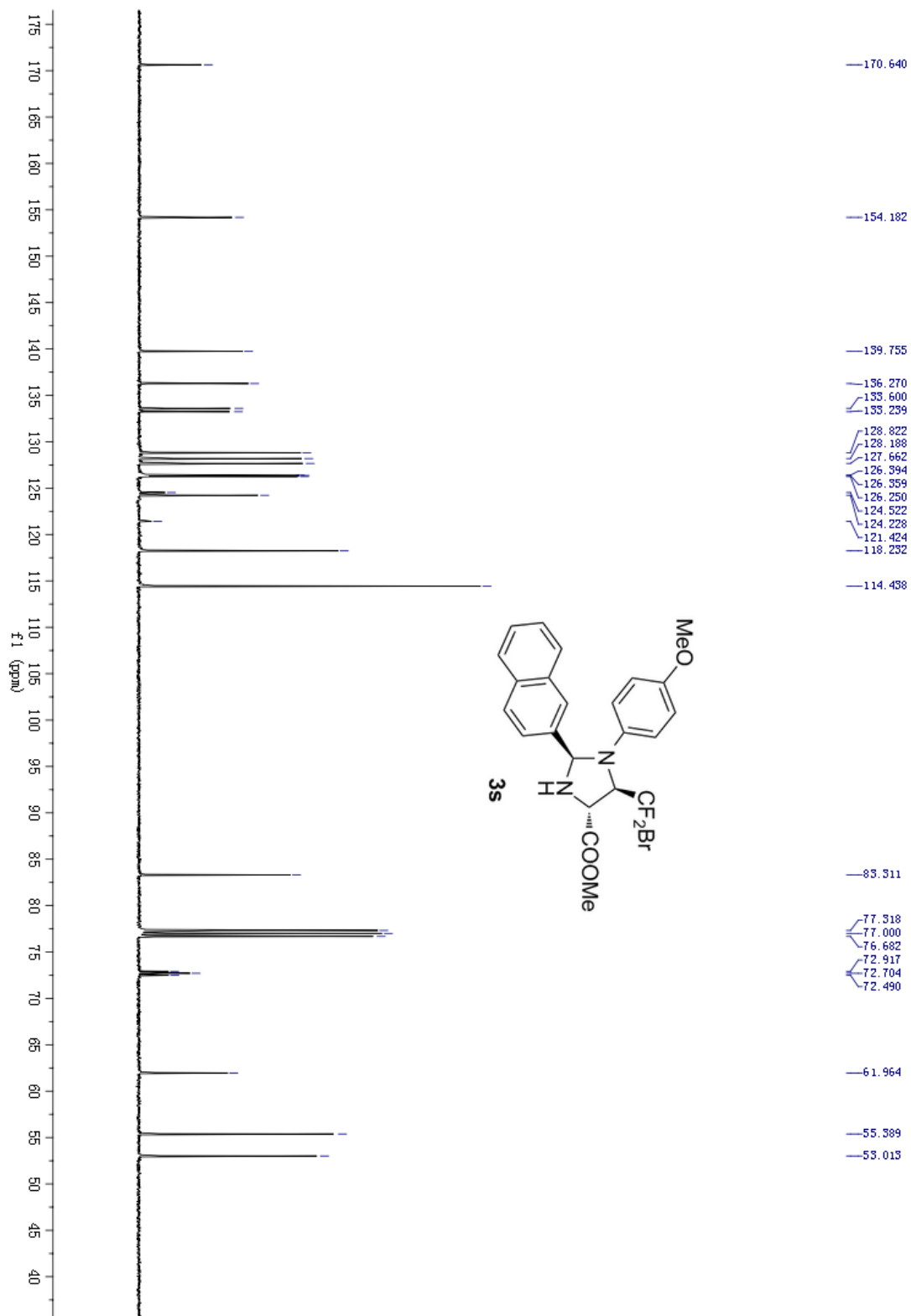


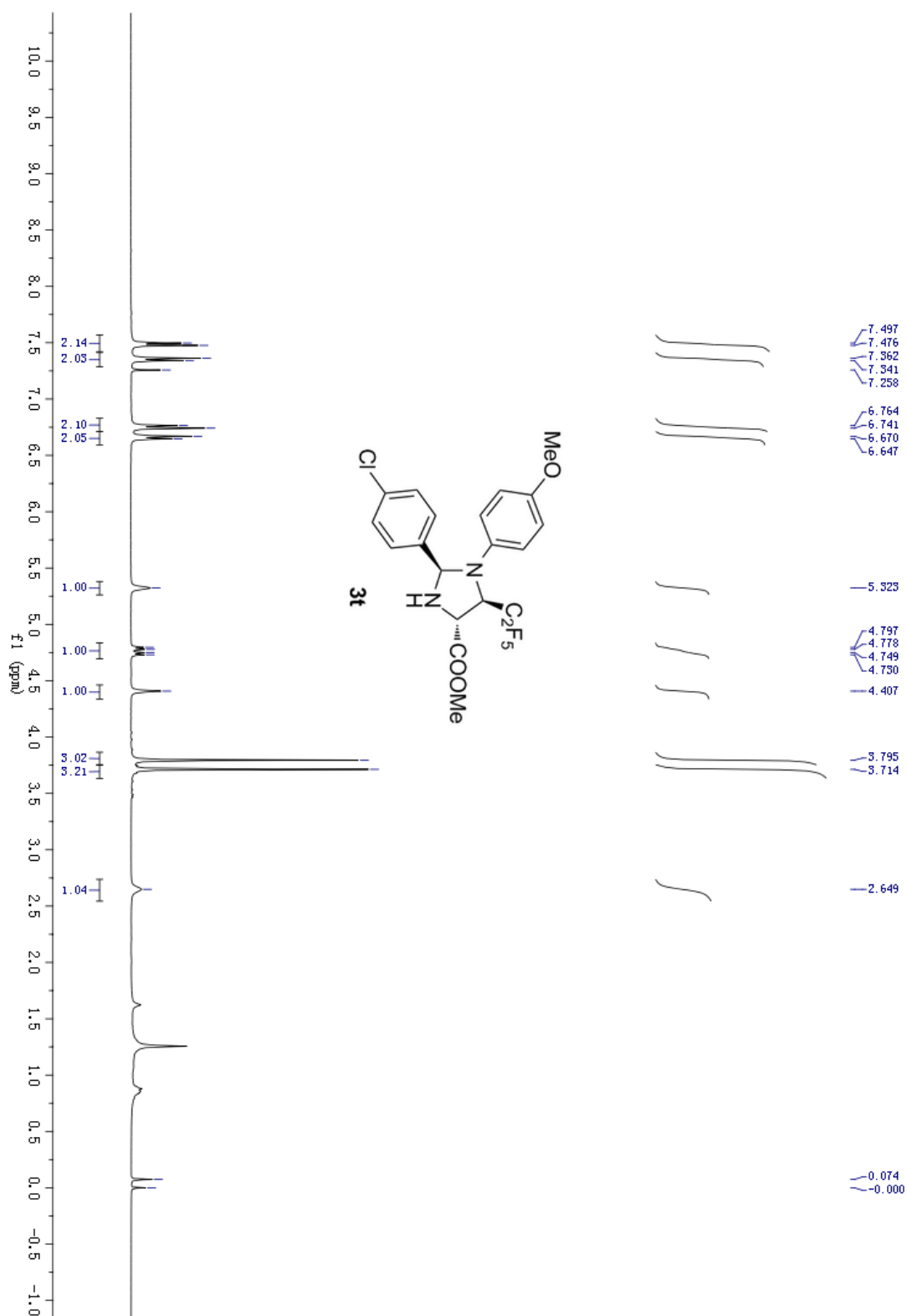


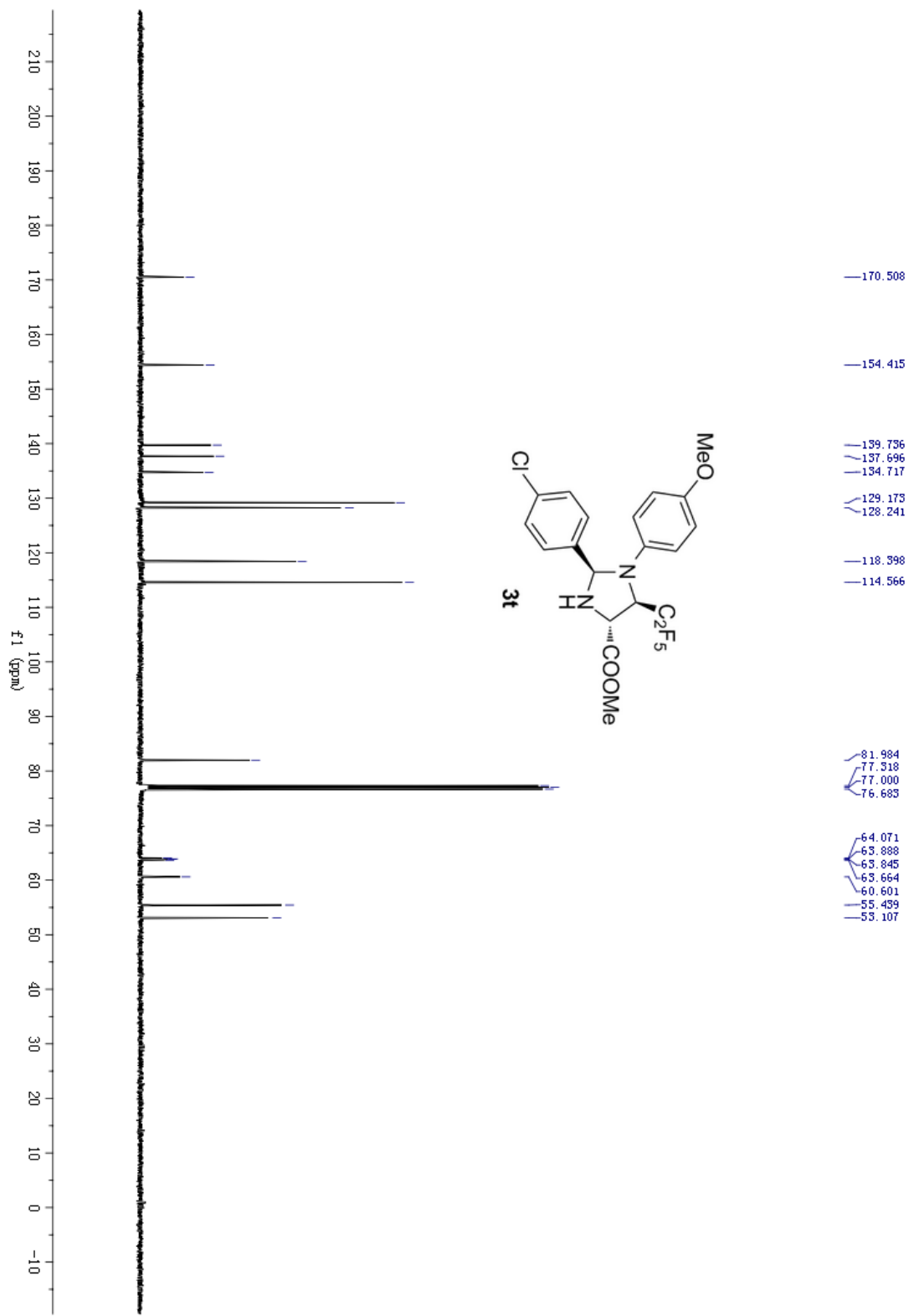


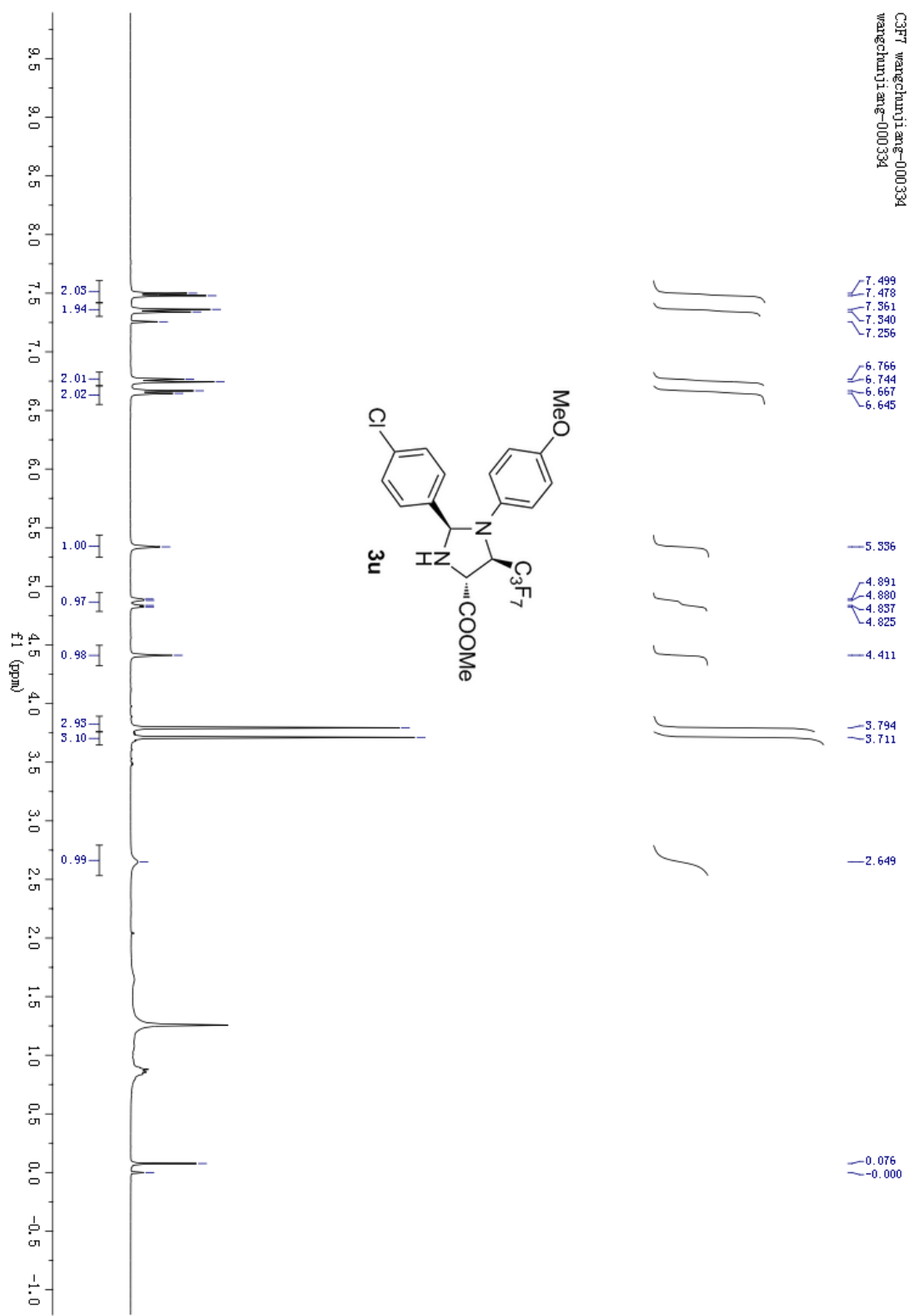


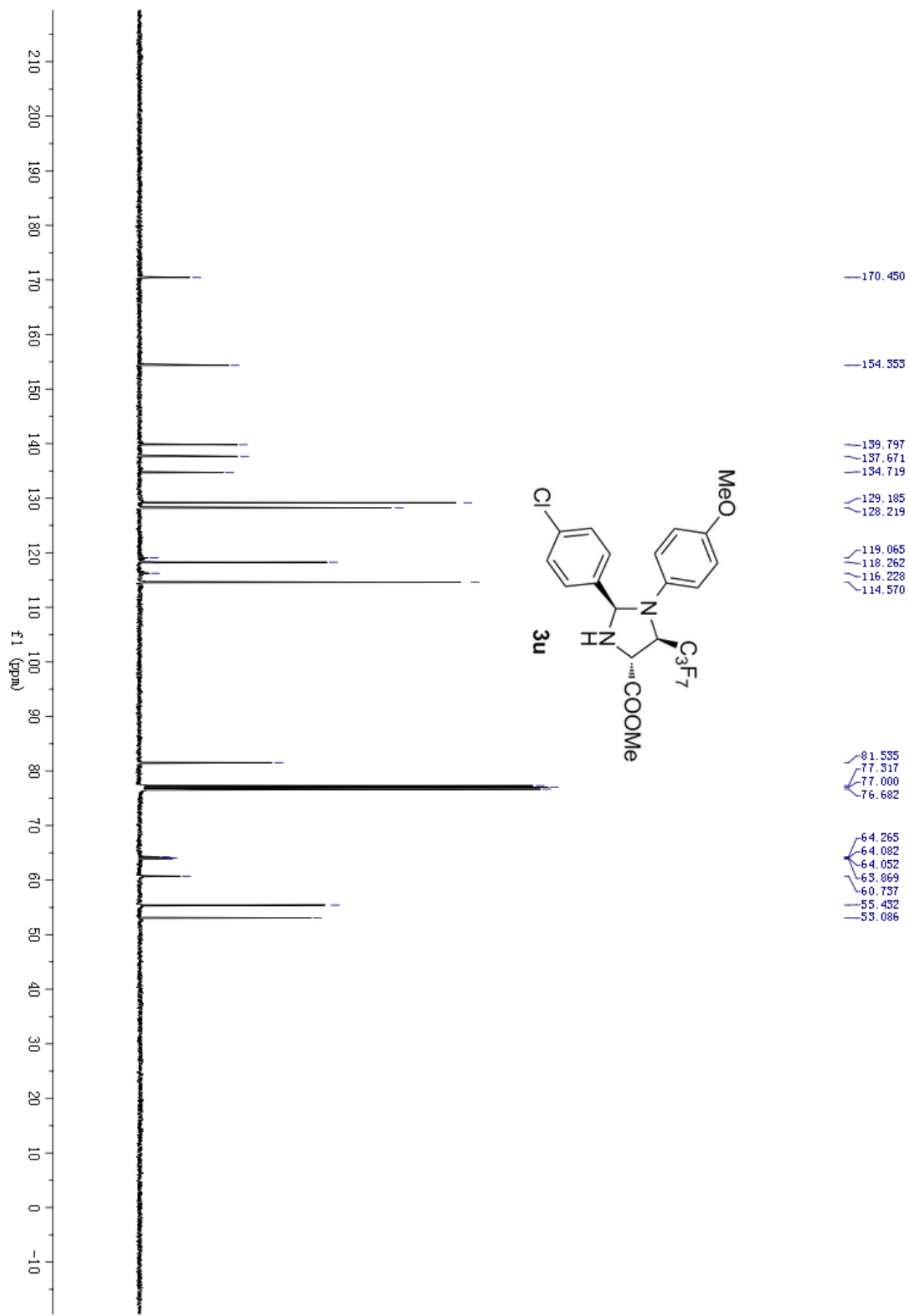


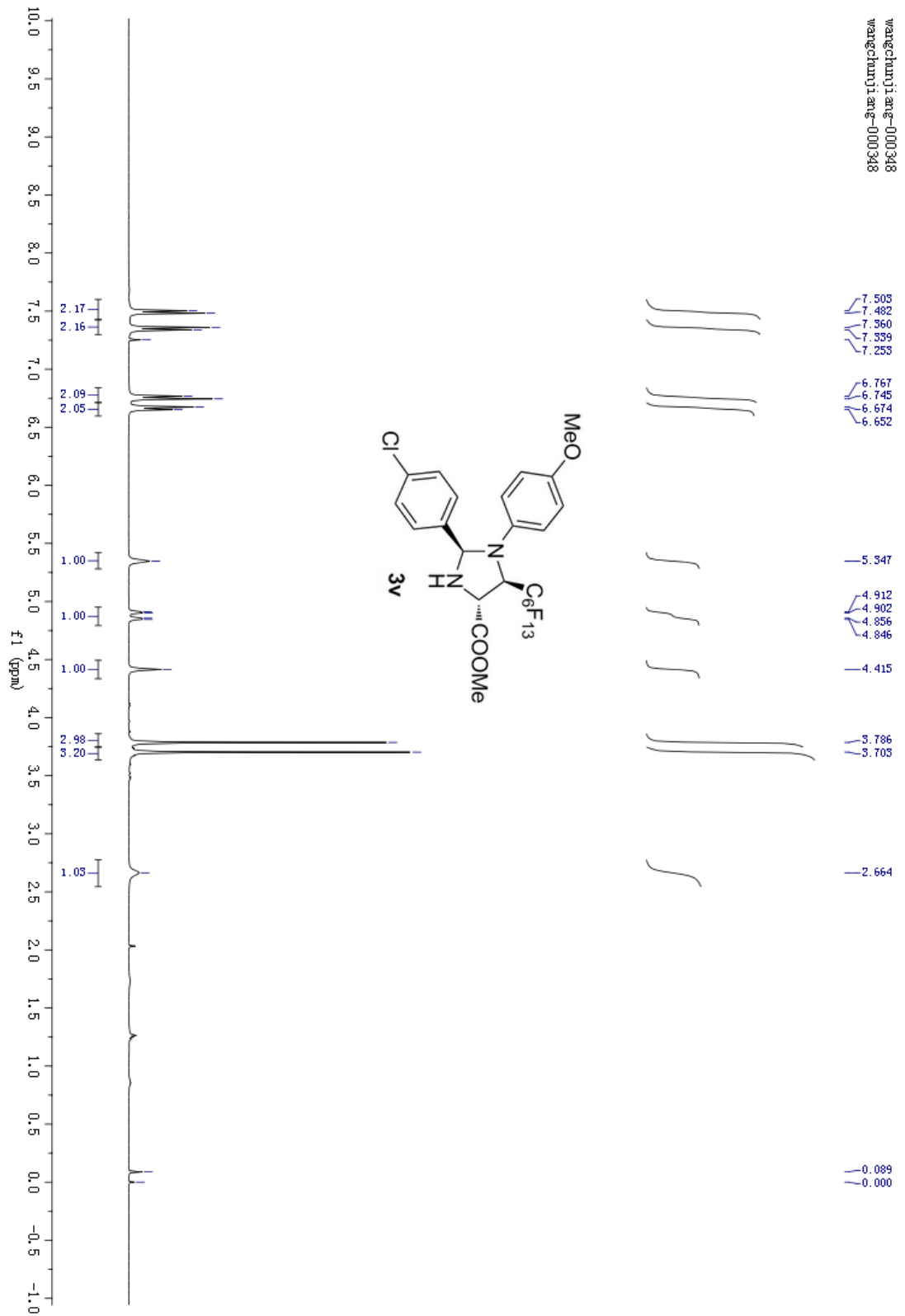


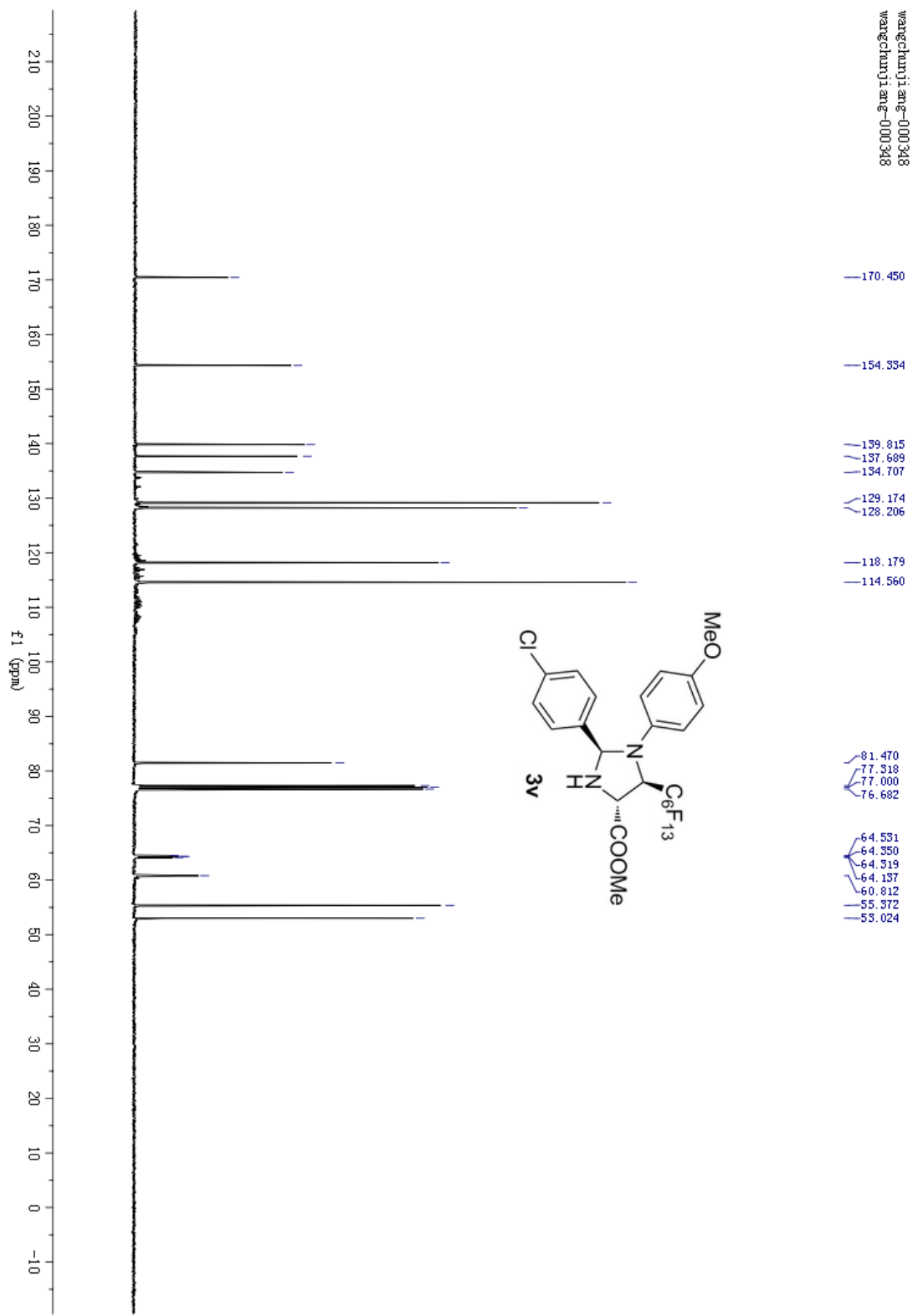


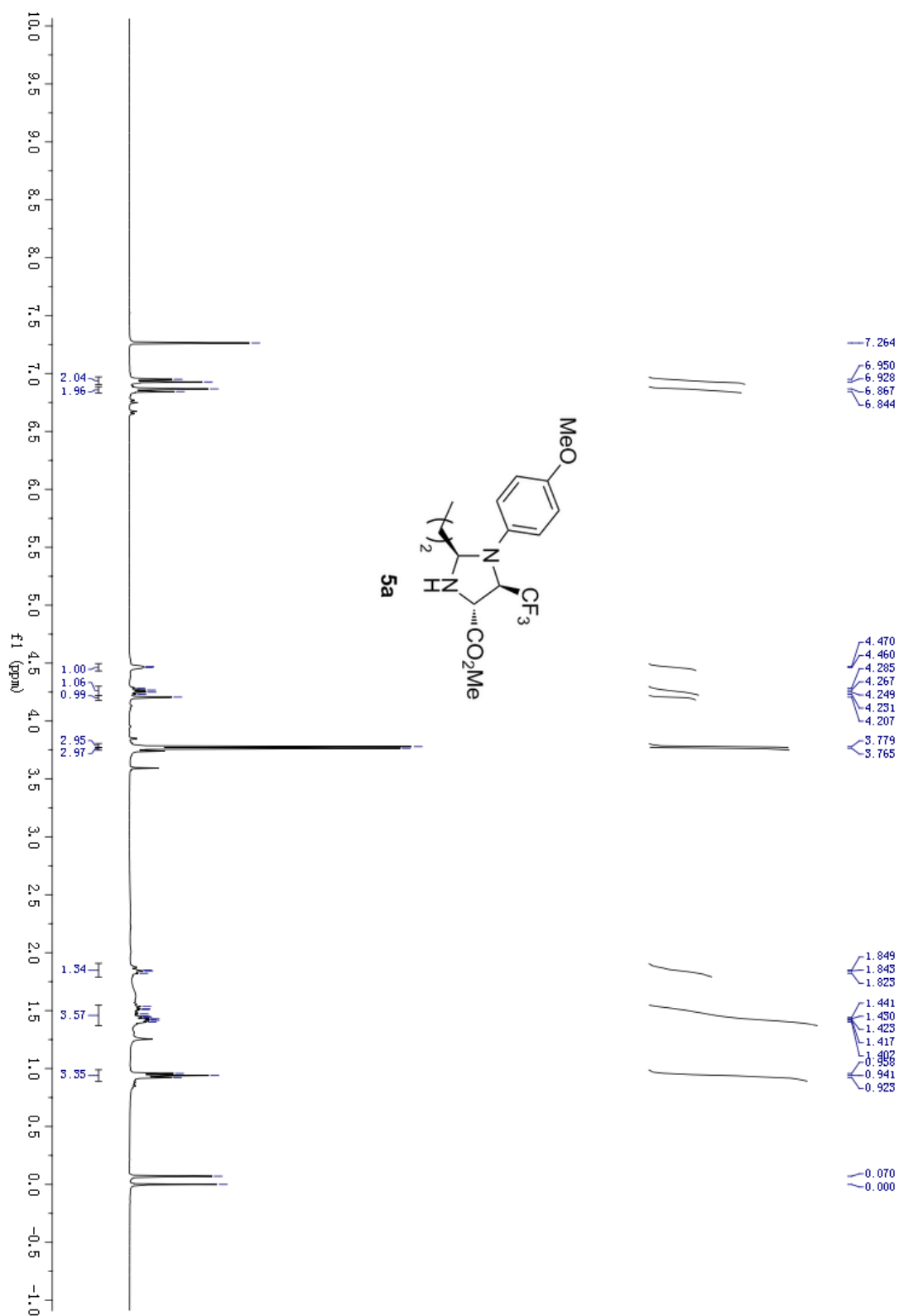


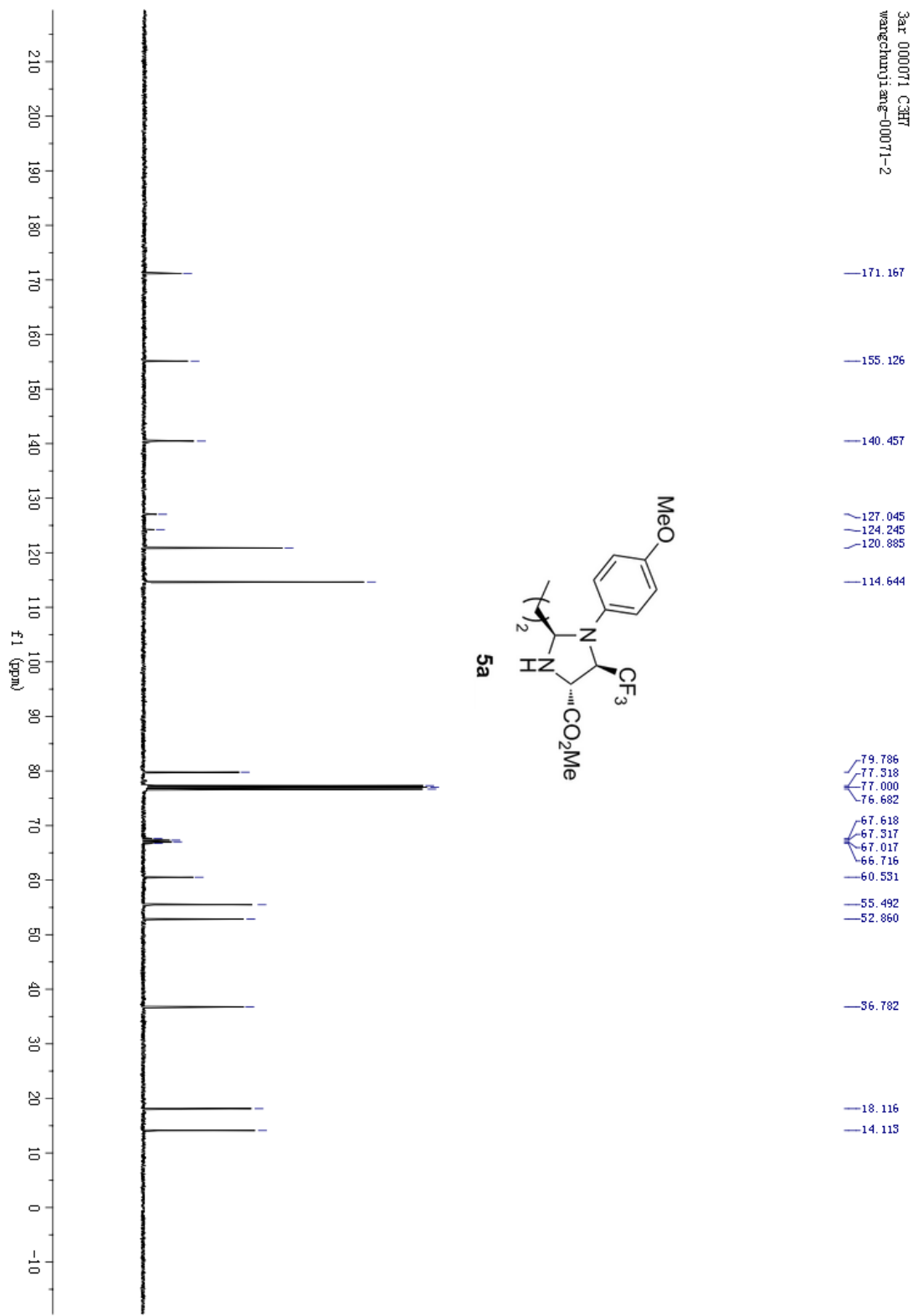


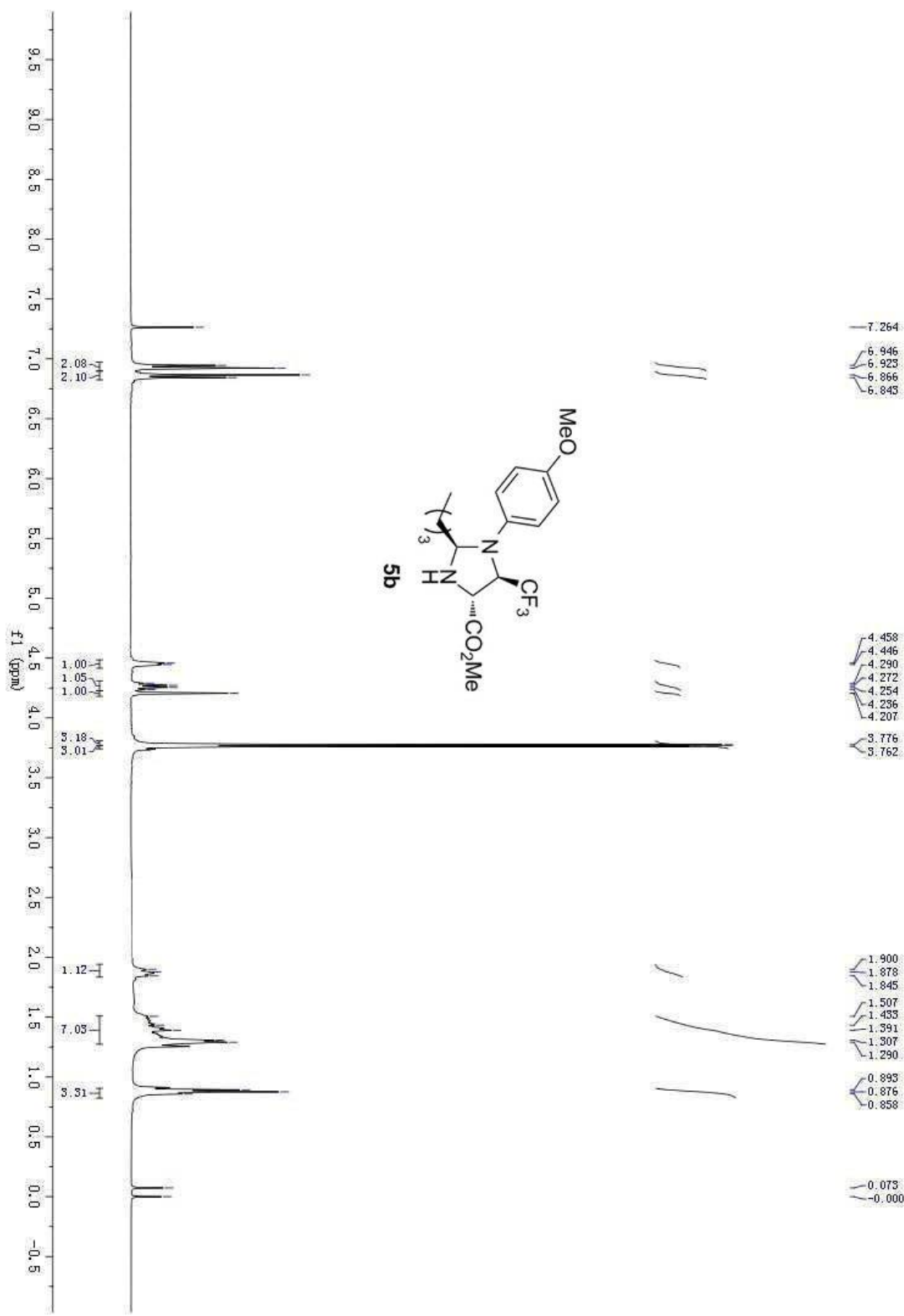


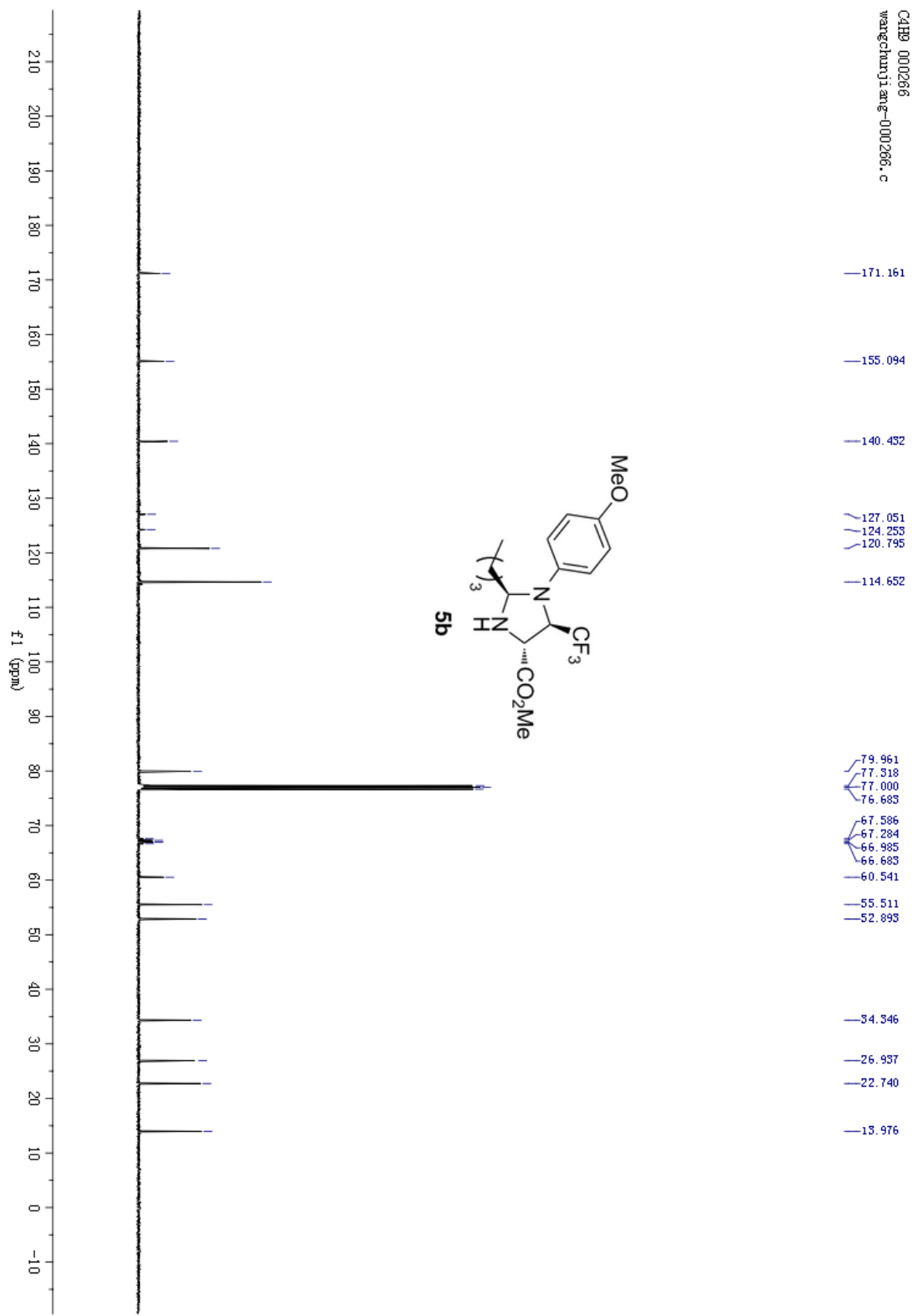


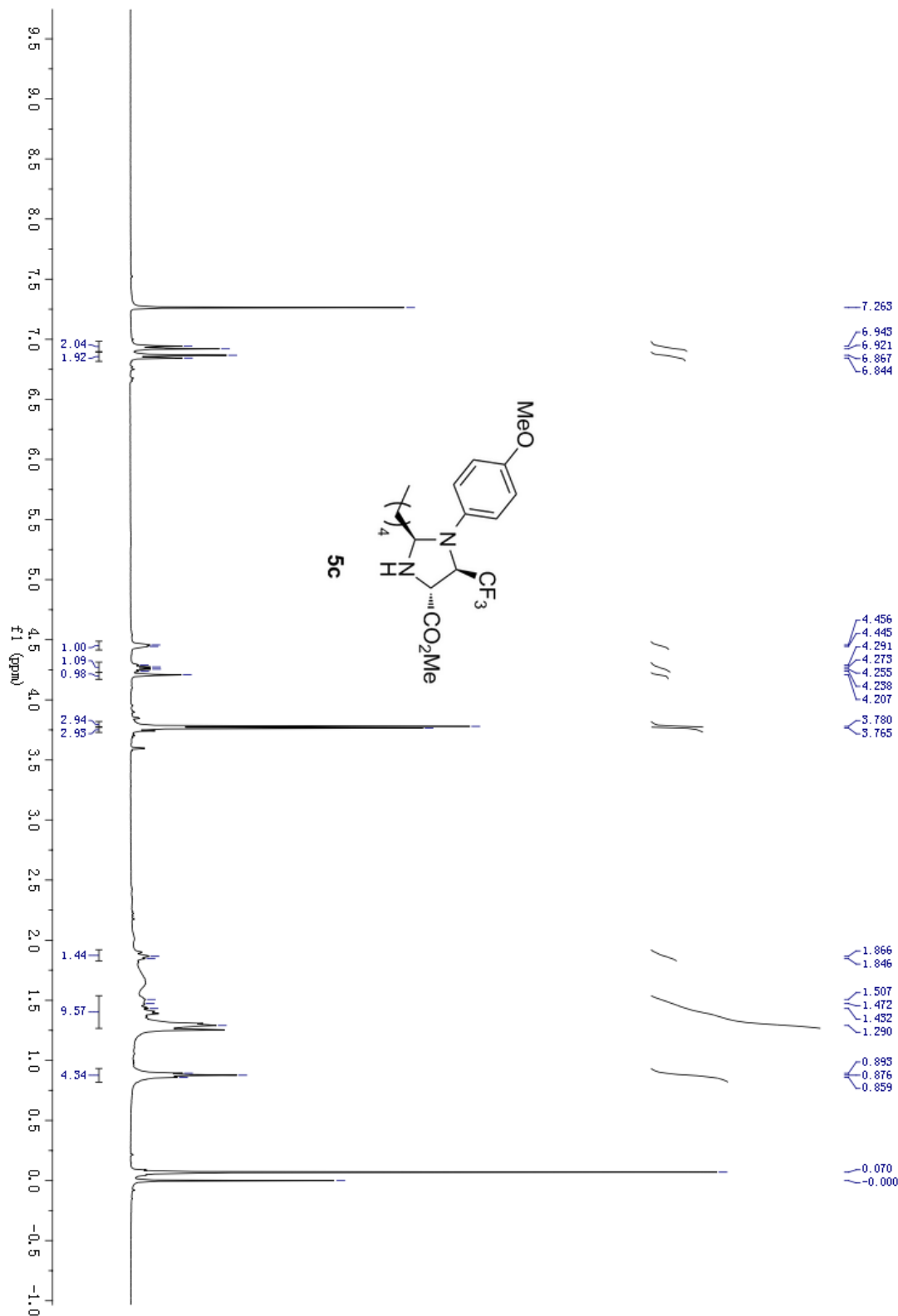


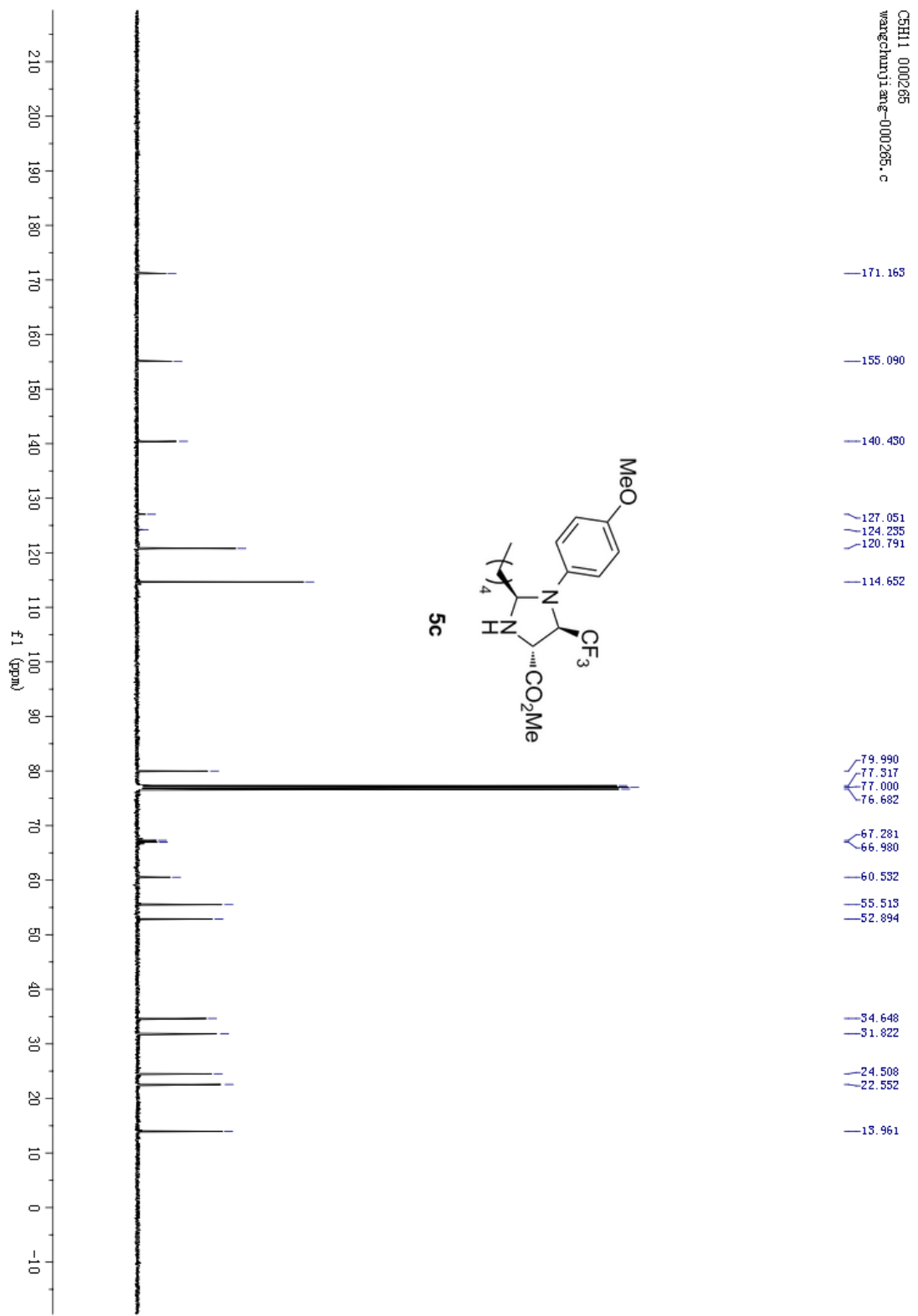


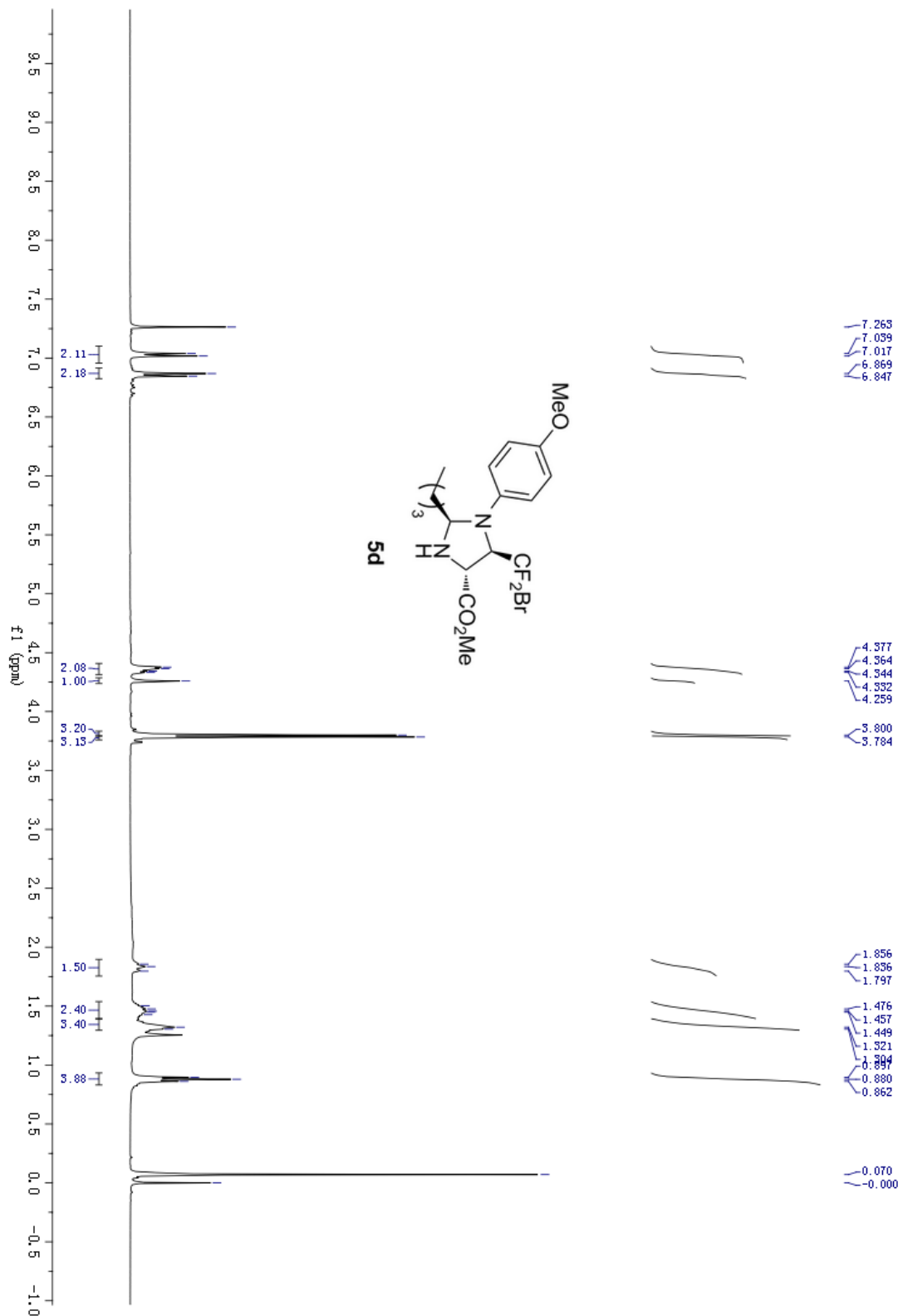


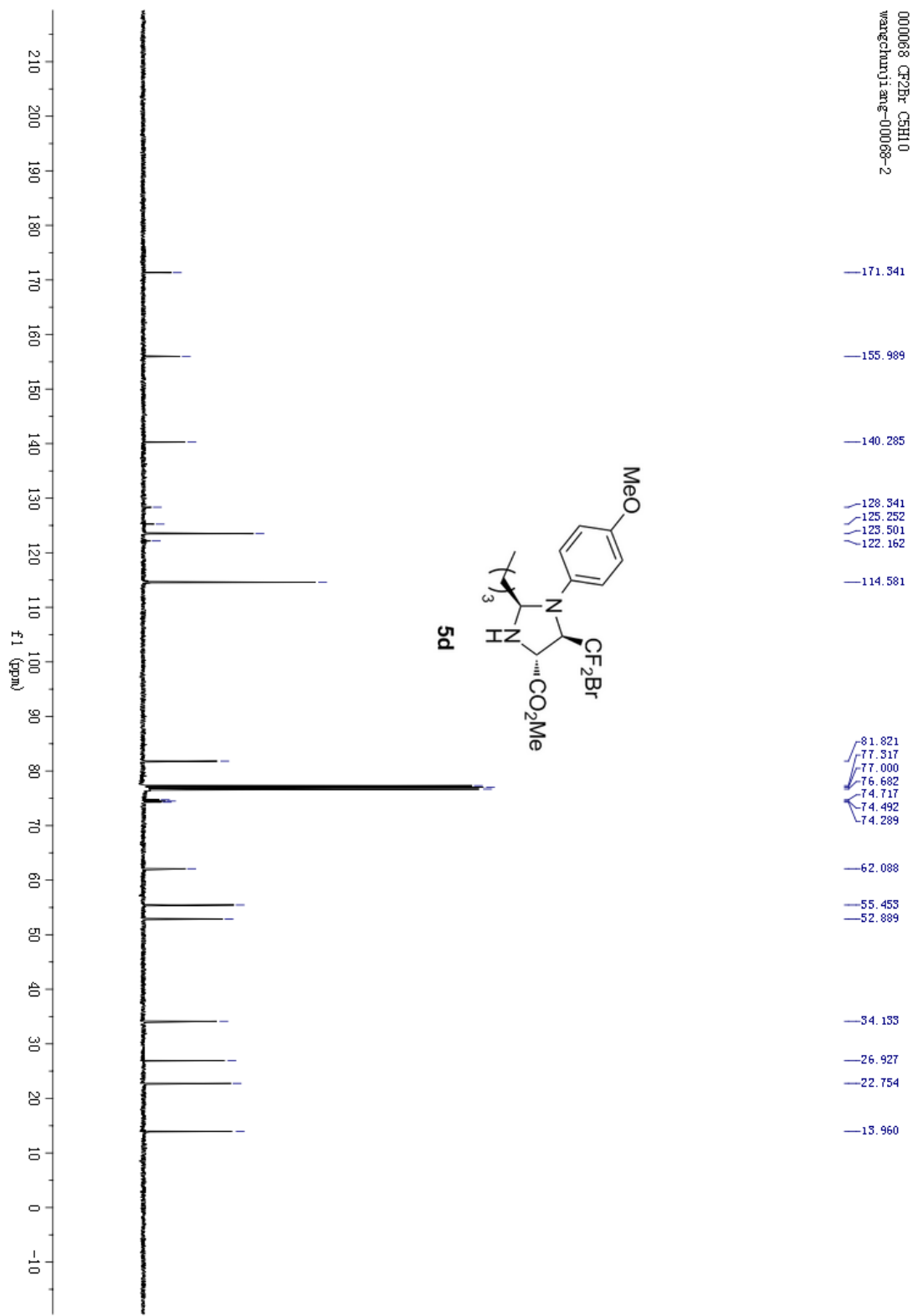


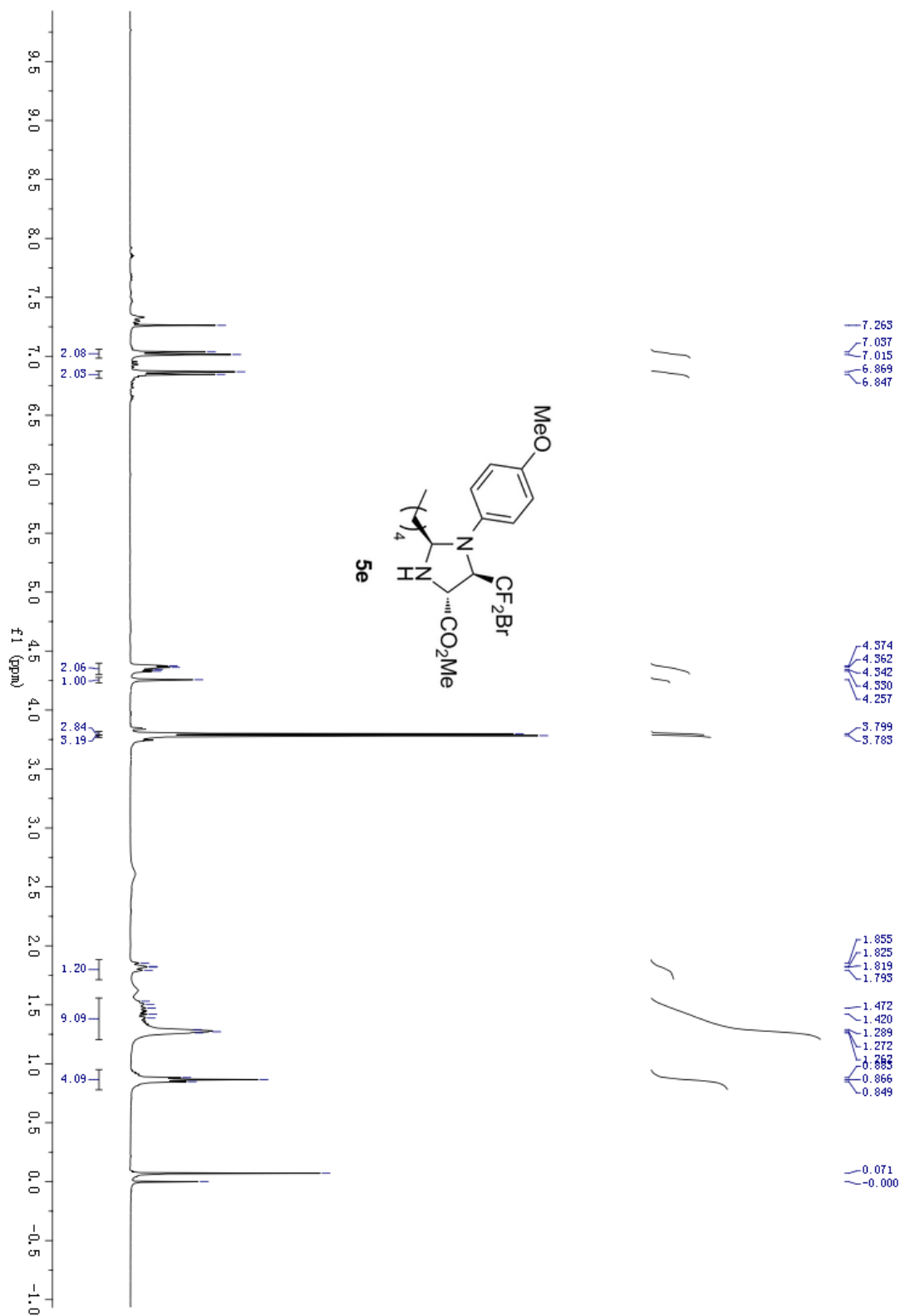


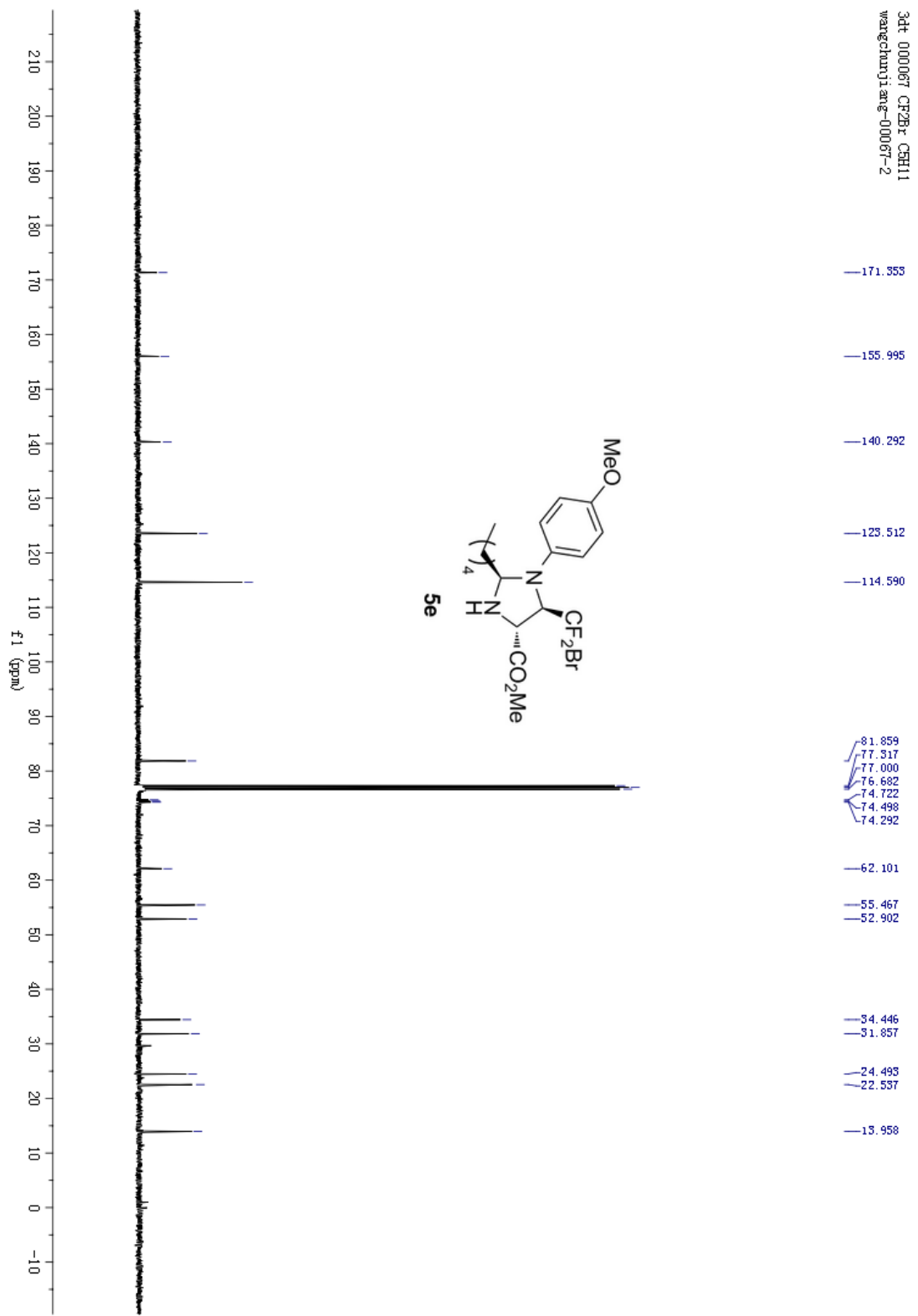


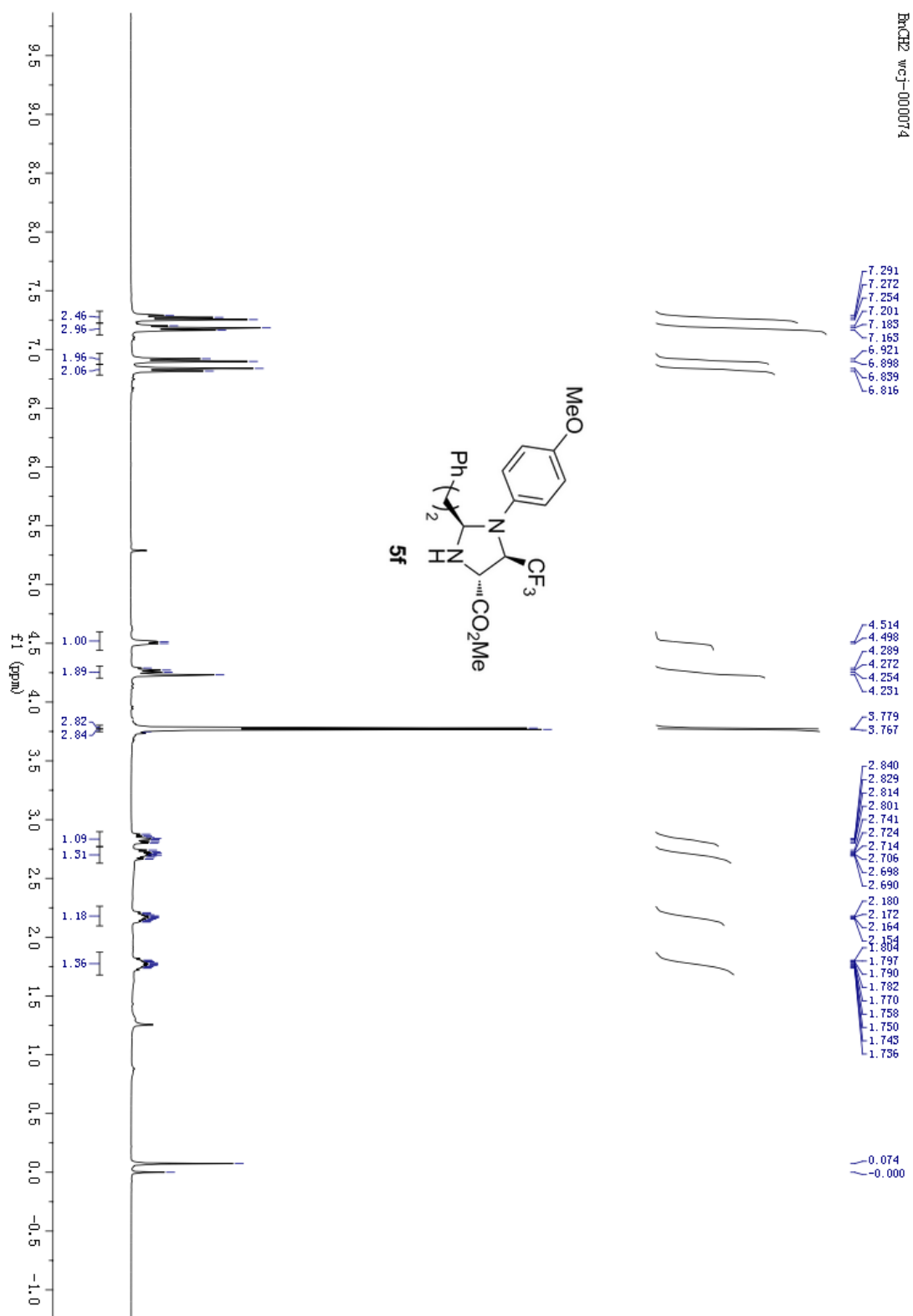


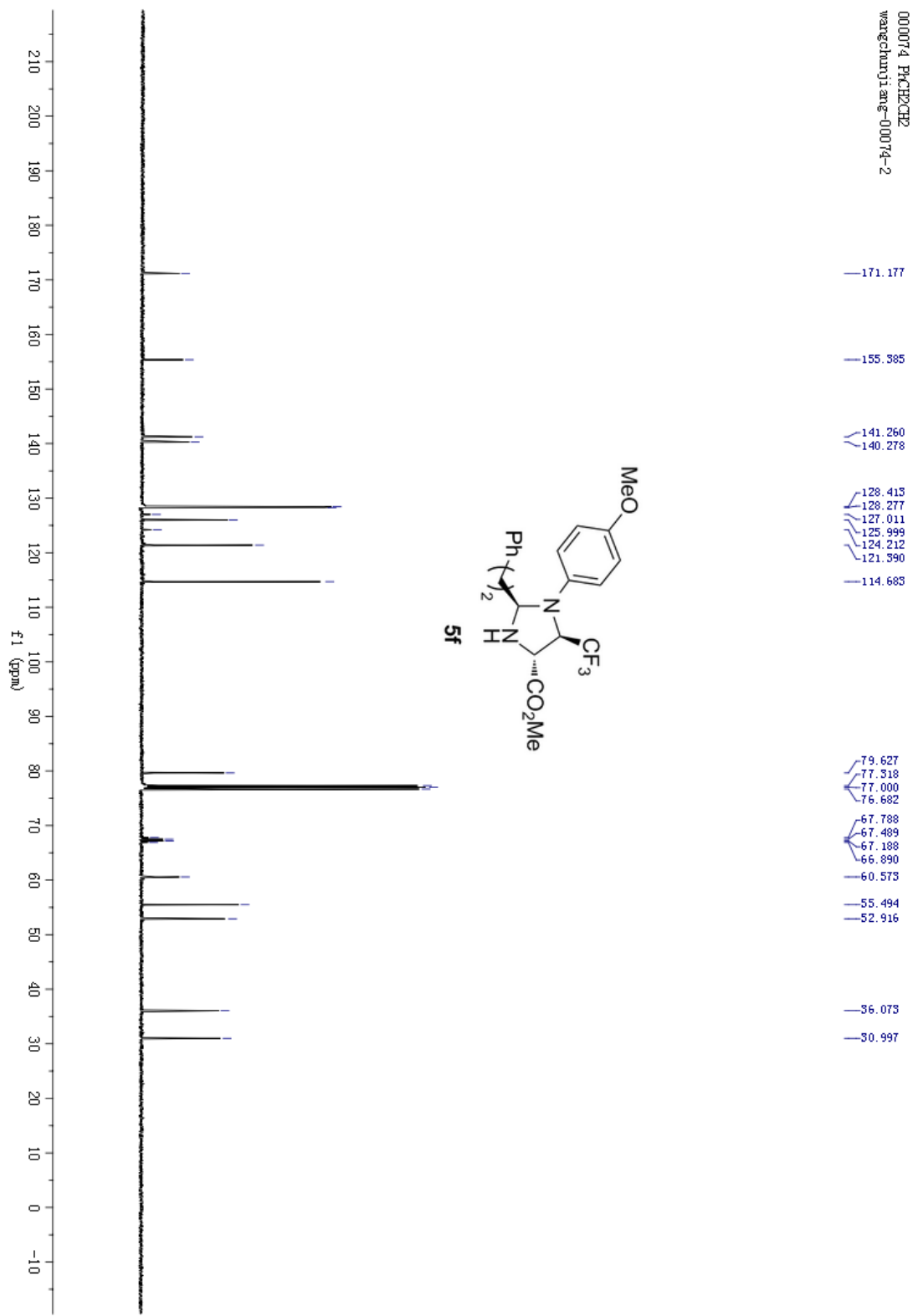


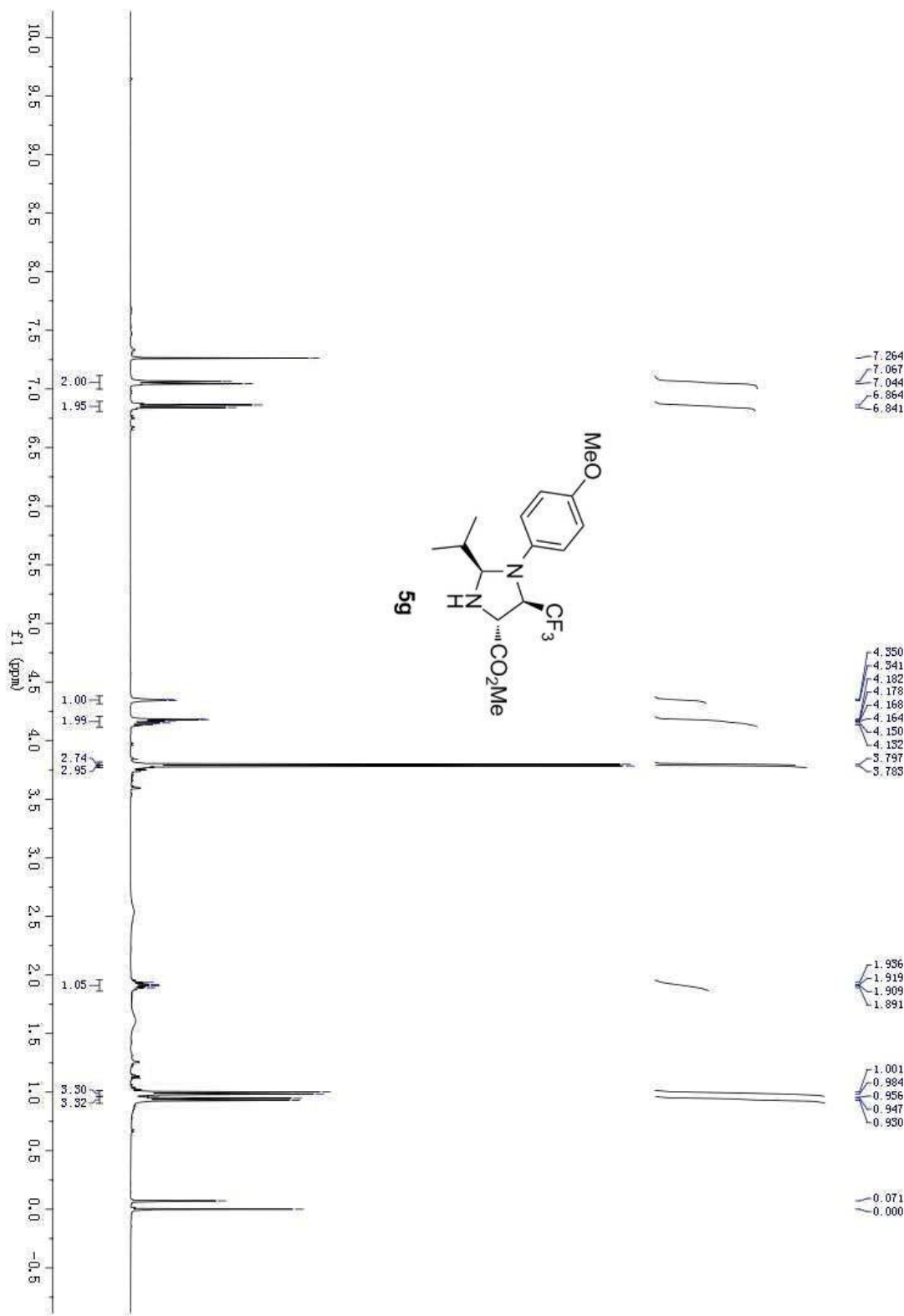


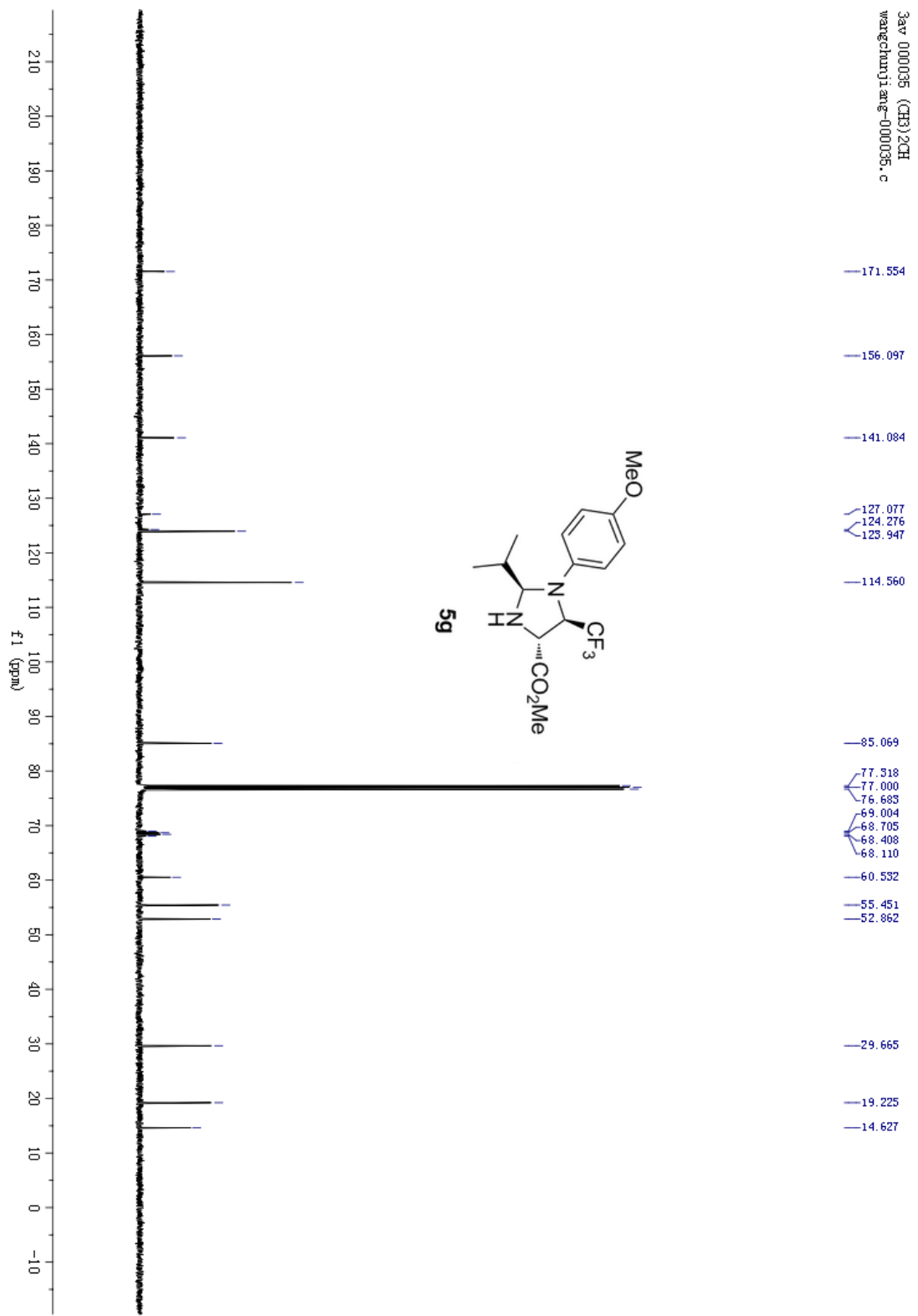


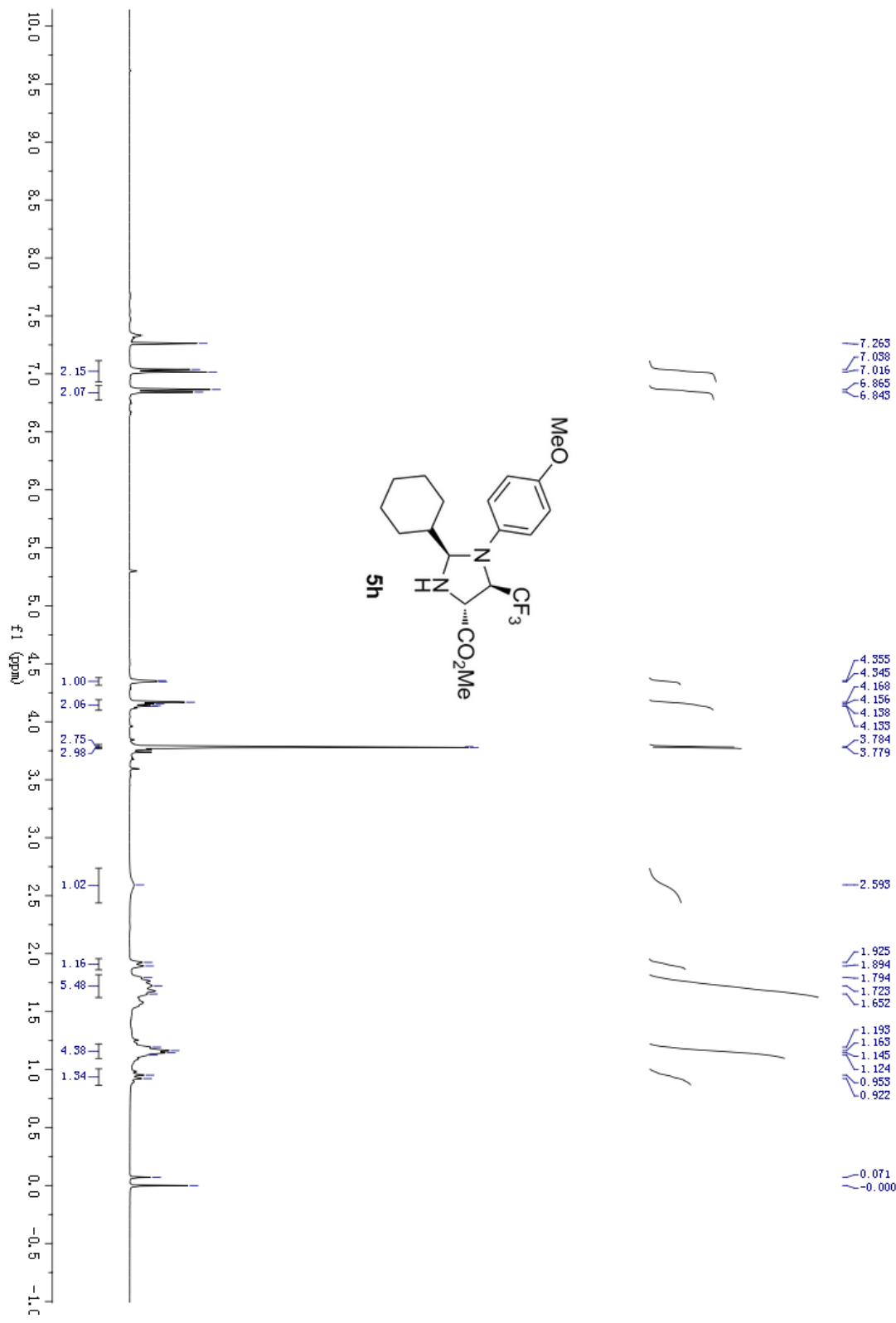


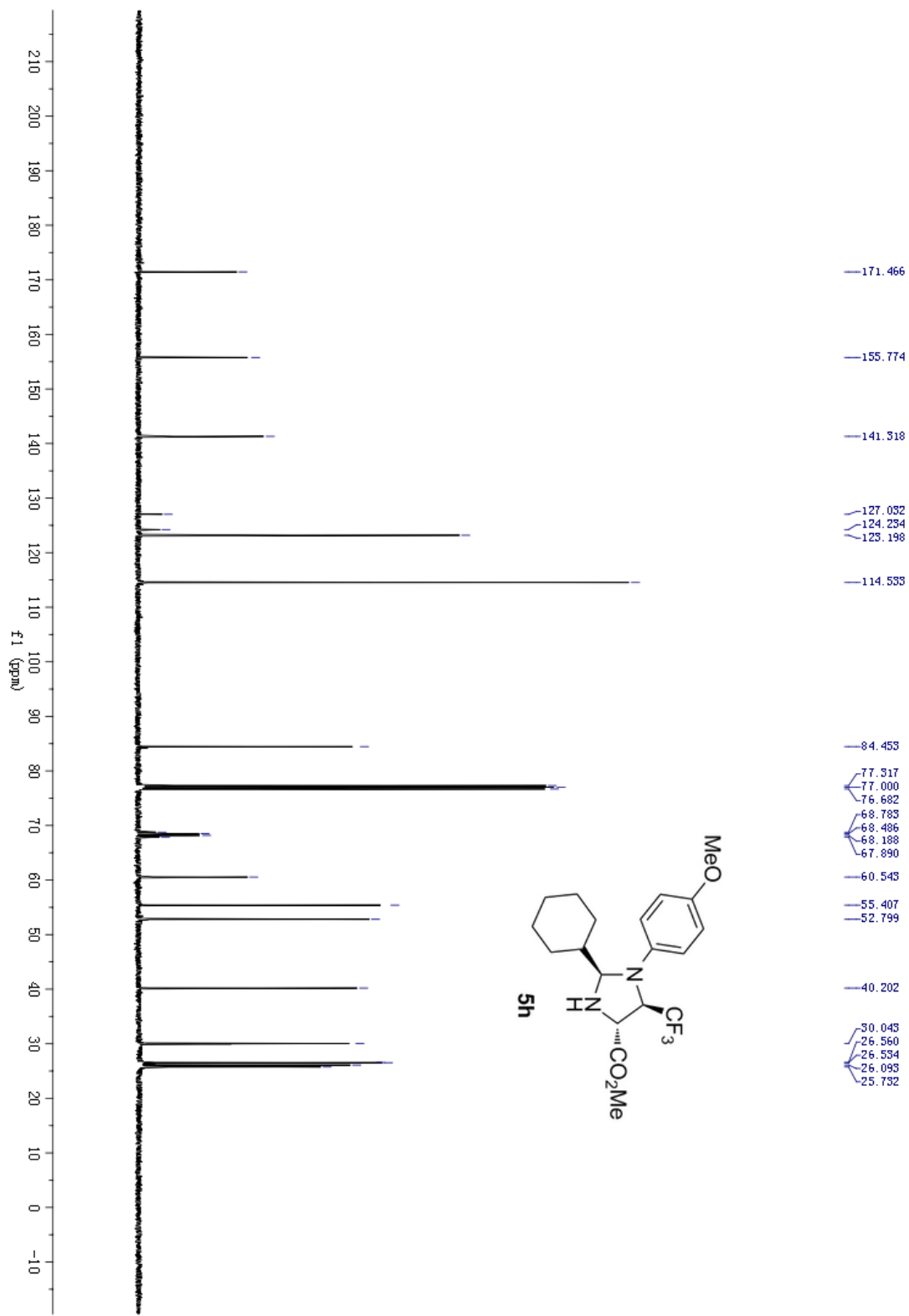


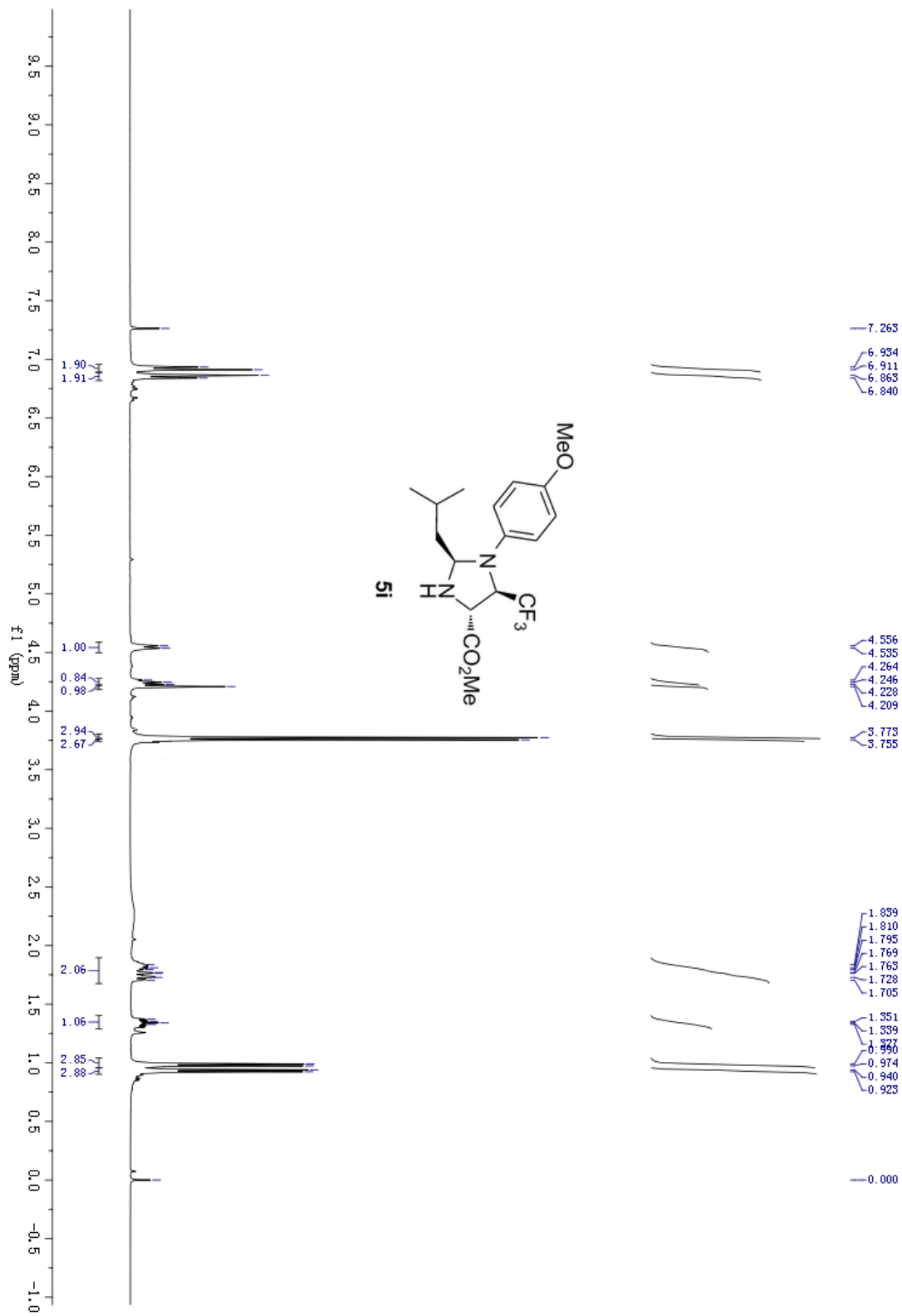


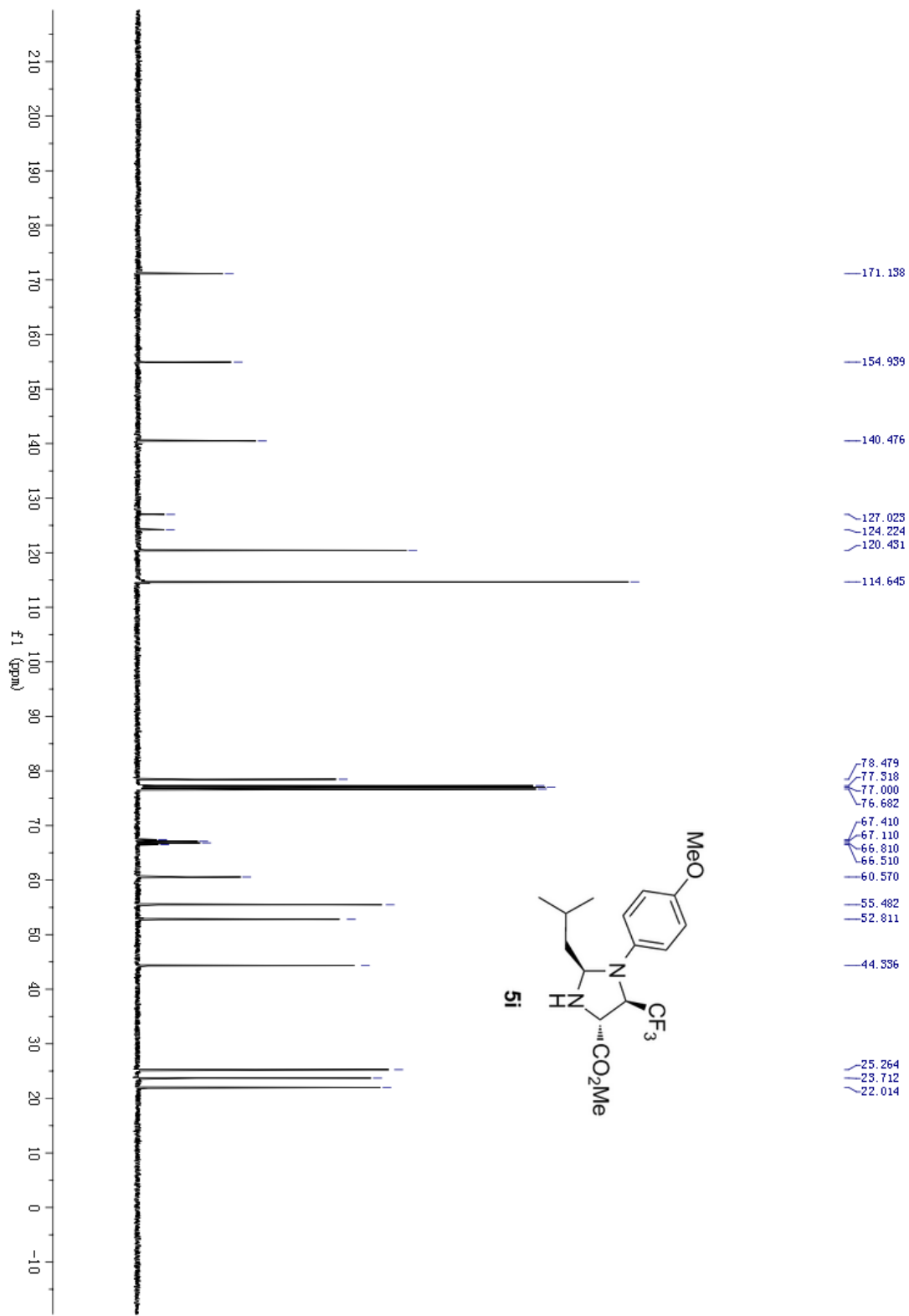


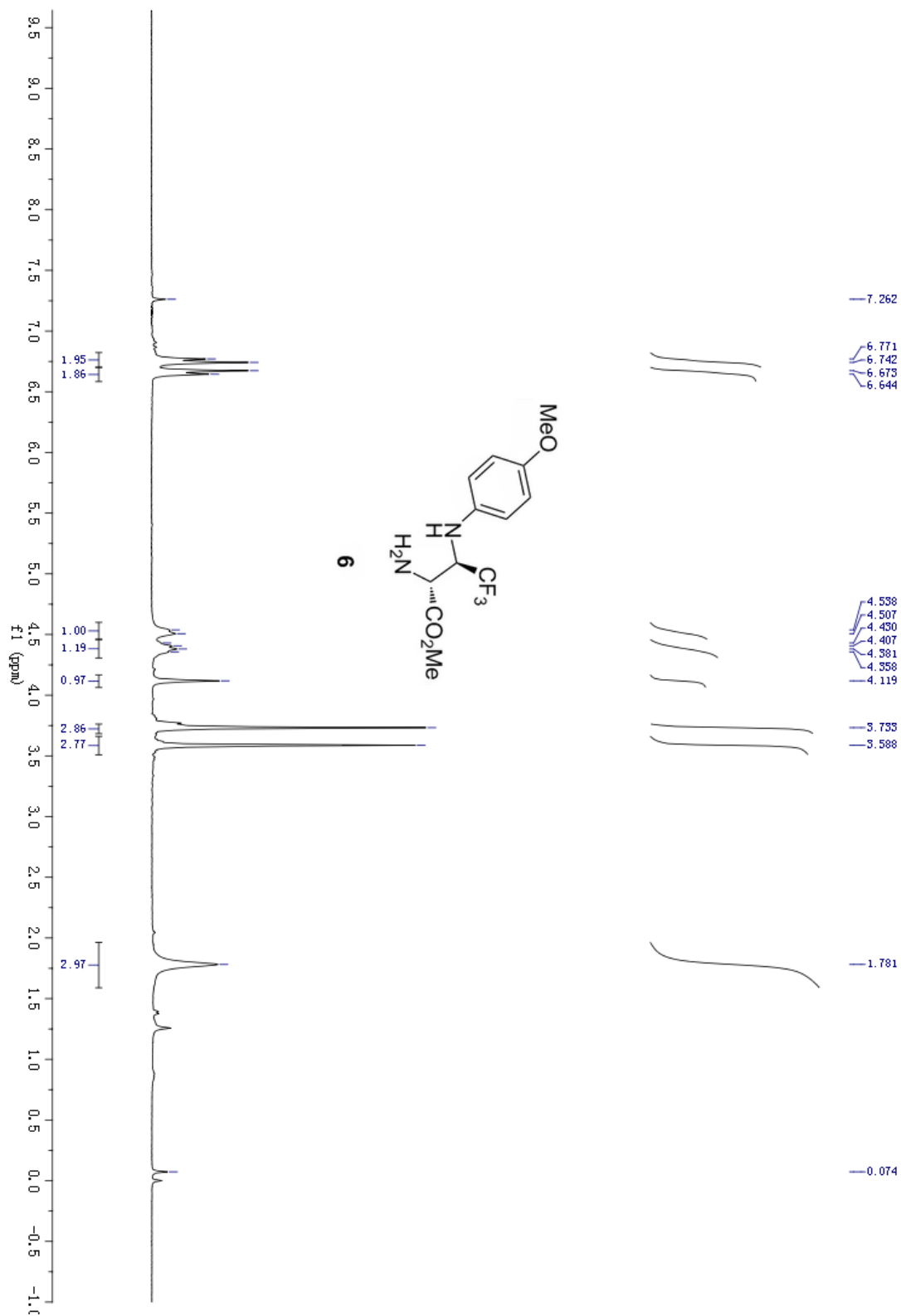


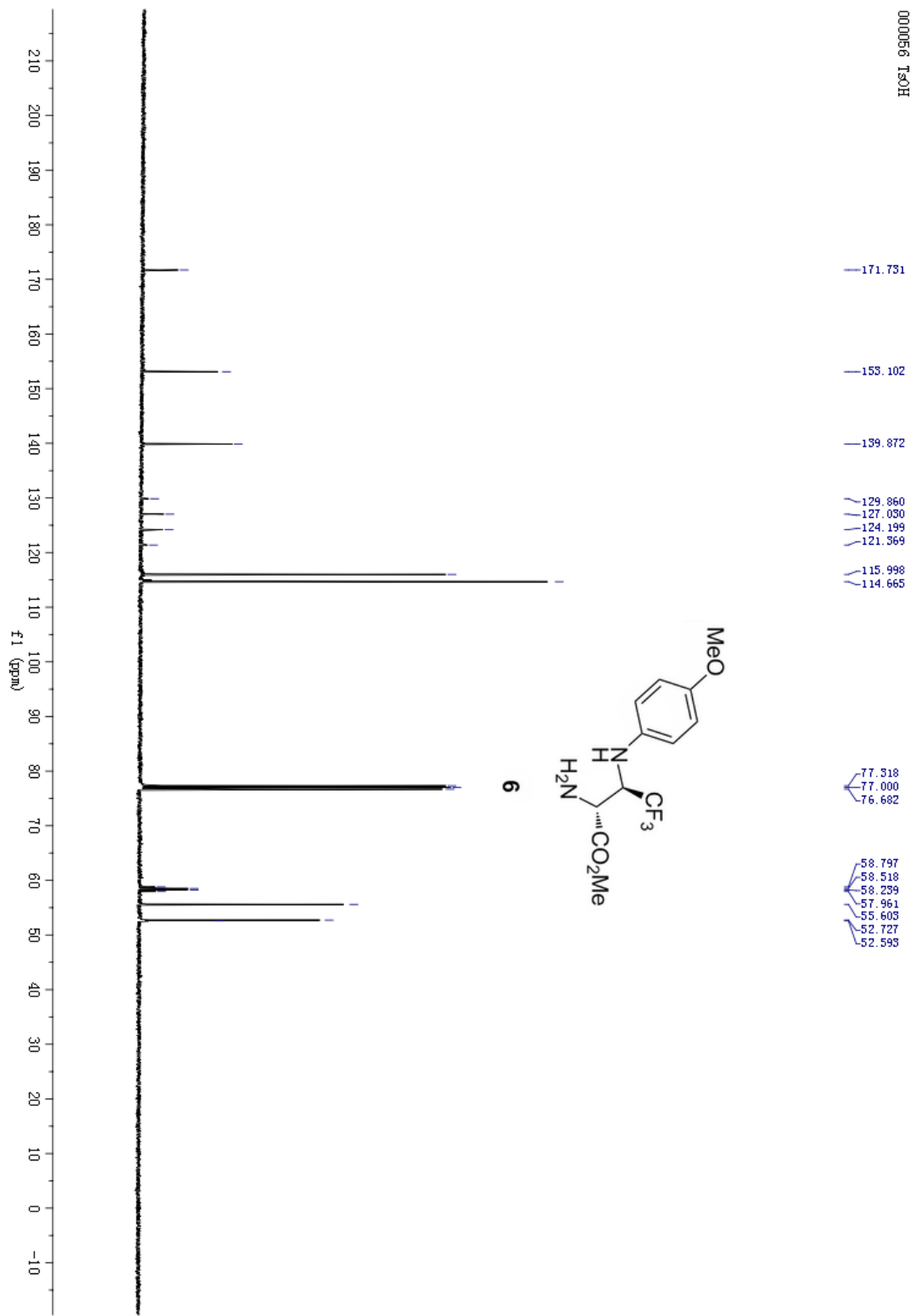


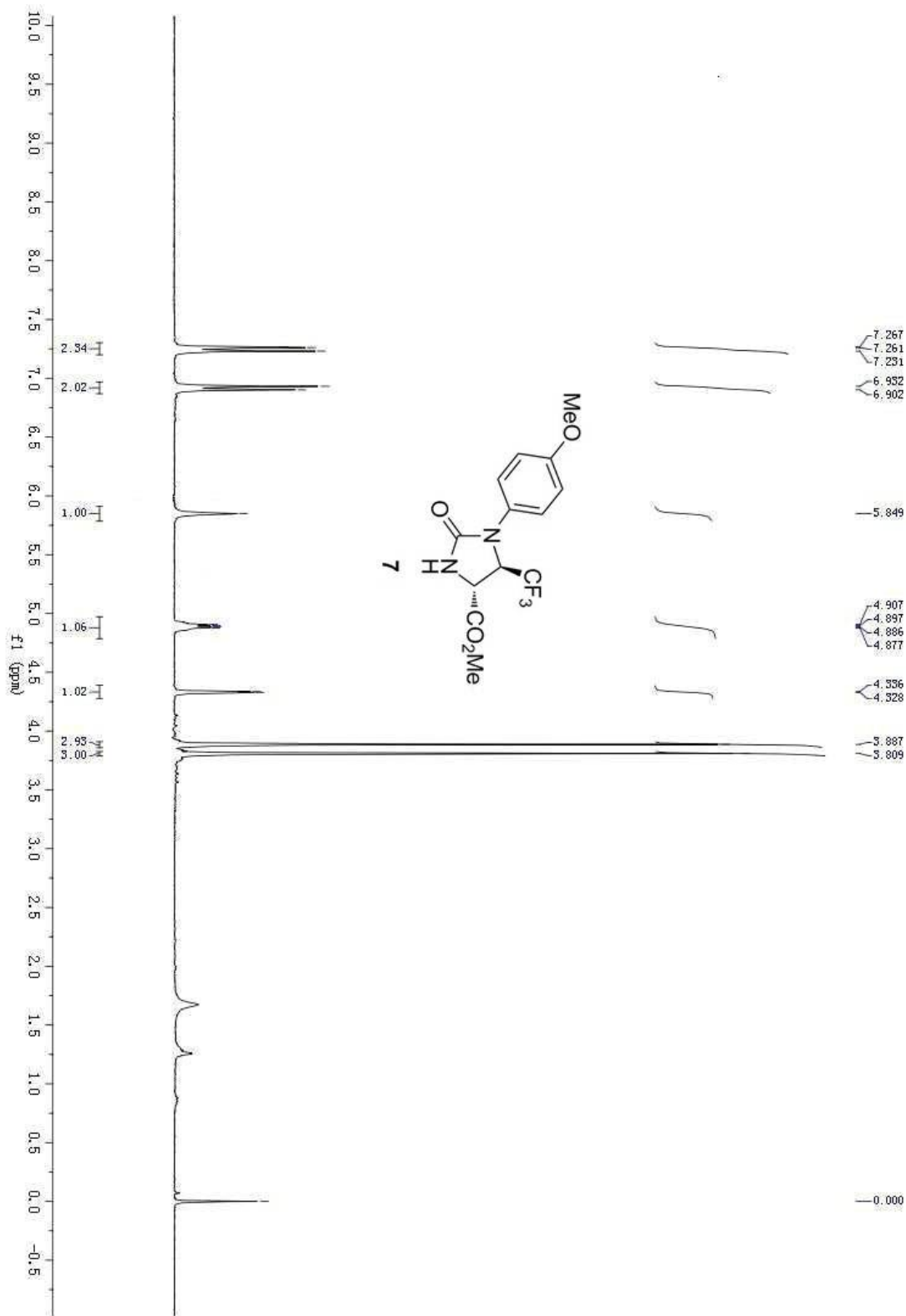


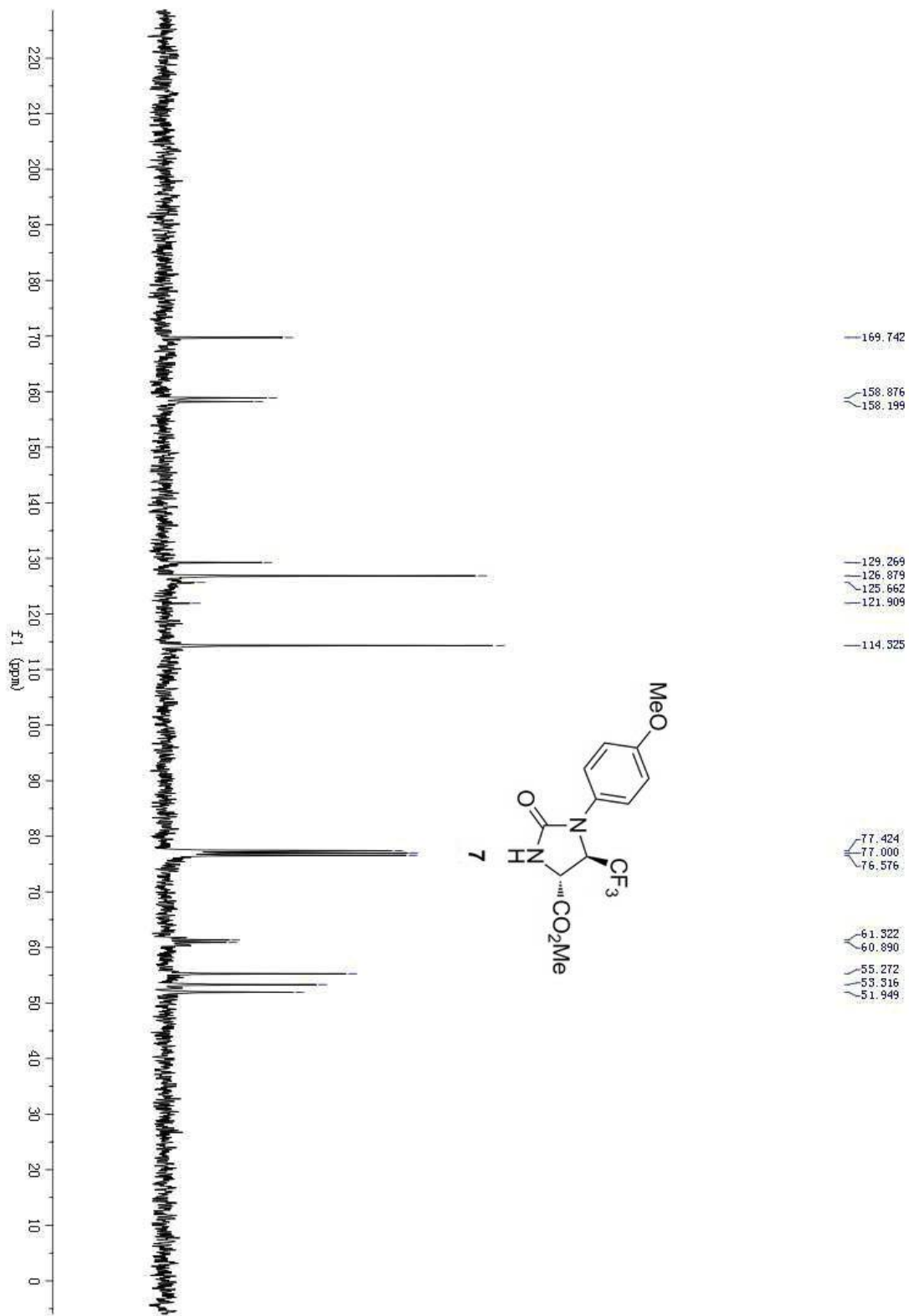


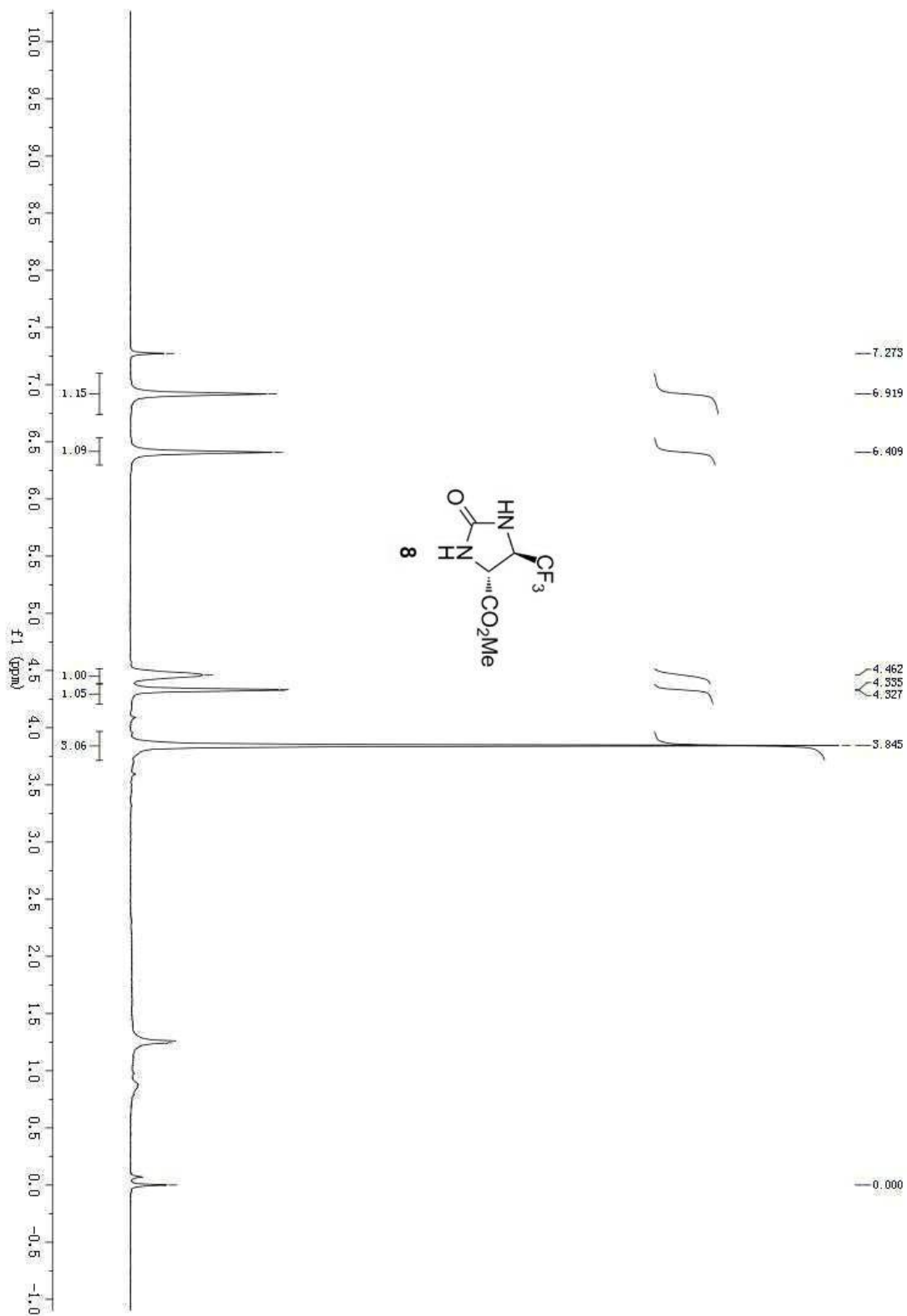


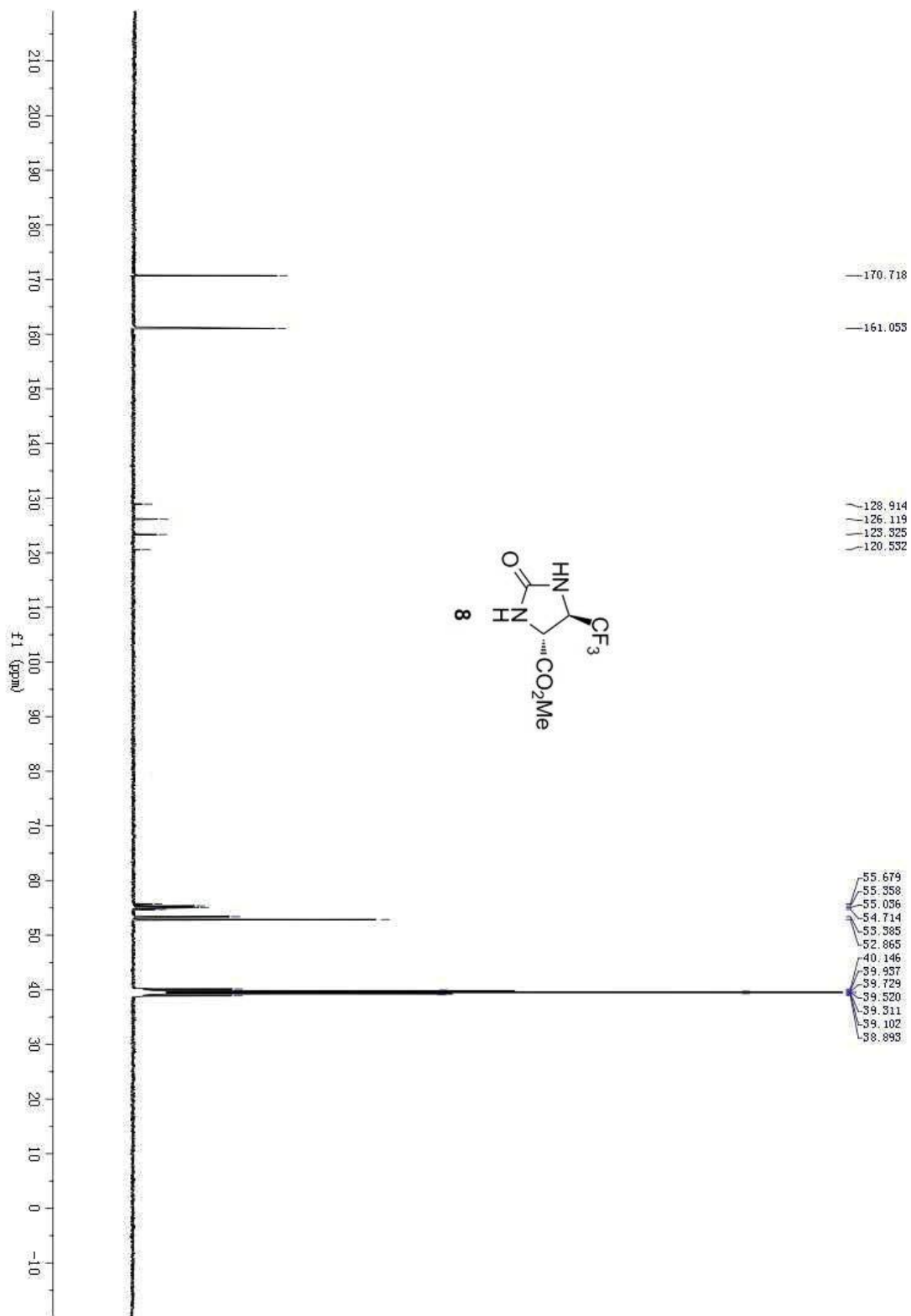








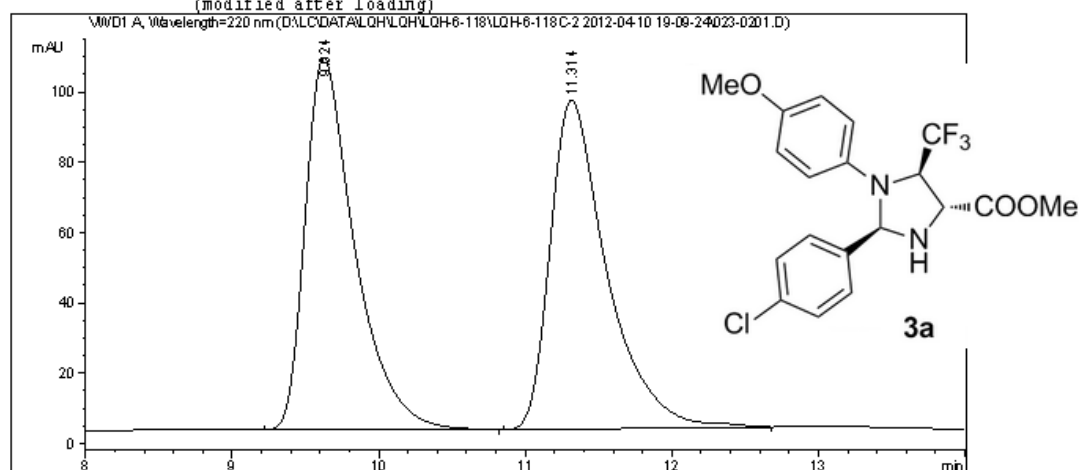




XII. HPLC Chromatograms

Data File D:\LC\DATA\LQH\LQH-6-118\LQH-6-118C-2 2012-04-10 19-09-24\023-0201.D
Sample Name: LQH-6-118C-2

```
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Acq. Operator   : LQH                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 23
Injection Date  : 4/10/2012 7:22:08 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201201\LQH\LQH-6-118\LQH-6-118C-2 2012-04-10 19-09-24\ADH-10-90-
                  10ML-220NM-30MIN.M
Last changed    : 11/9/2011 12:13:31 PM by THL
Analysis Method : D:\LC\DATA\LQH\LQH-6-118\LQH-6-118C-2 2012-04-10 19-09-24\023-0201.D\
                  D.A.M (ADH-10-90-10ML-220NM-30MIN.M)
Last changed    : 7/9/2012 8:40:44 PM by LJ
                  (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	Height [mAU]	Area %
1	9.624	BB	0.3499	2491.14551	105.98347	49.7242
2	11.314	BB	0.3998	2518.77734	93.60684	50.2758

Totals : 5009.92285 199.59032

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

Instrument 1 7/9/2012 8:40:50 PM LJ

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-7-14\LQH-7-14A 2012-03-30 18-49-25\065-0201.D
Sample Name: LQH-7-14A

=====

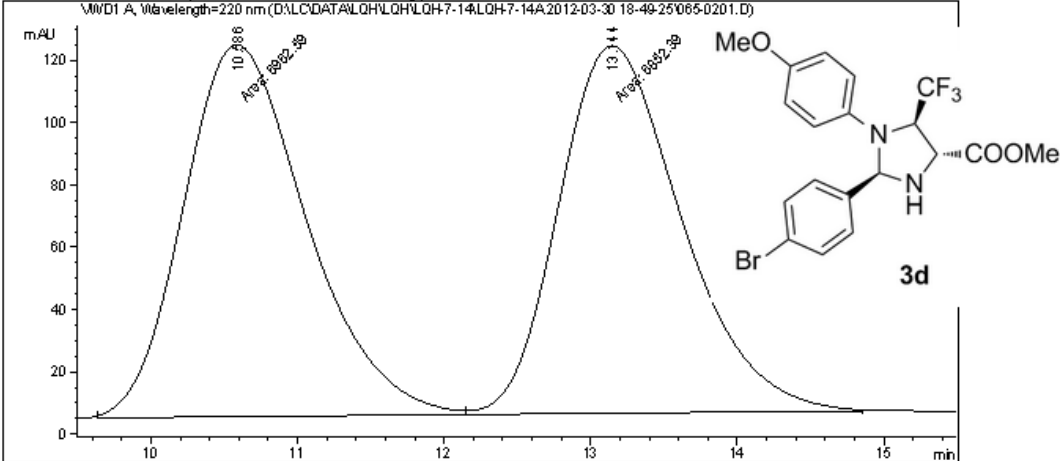
Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 65
Injection Date	: 3/30/2012 7:01:25 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\201201\LQH\LQH-7-14\LQH-7-14A 2012-03-30 18-49-25\ASH-10-90-10ML-220NM.M

Last changed : 3/30/2012 7:23:42 PM by LQH
(modified after loading)

Analysis Method : D:\LC\DATA\LQH\LQH-7-14\LQH-7-14A 2012-03-30 18-49-25\065-0201.D\DA.M
(ASH-10-90-10ML-220NM.M)

Last changed : 9/6/2012 10:53:24 AM by thl
(modified after loading)



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

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

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	10.586	FM	0.9719	6962.59375	119.40109	50.3989
2	13.144	MF	0.9682	6852.38574	117.95641	49.6011

Totals : 1.38150e4 237.35750

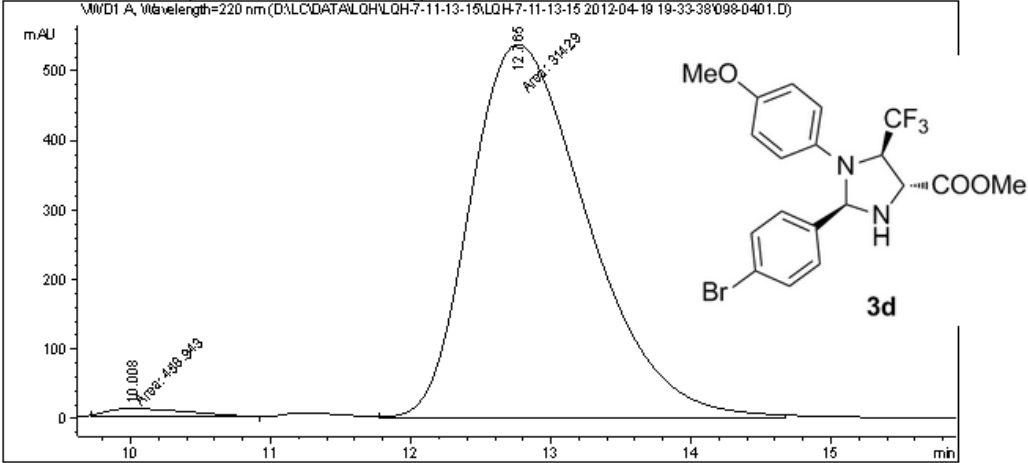
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*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-7-11-13-15\LQH-7-11-13-15 2012-04-19 19-33-38\098-0401.D
Sample Name: LQH-7-15A

=====

Acq. Operator	: LQH	Seq. Line	: 4
Acq. Instrument	: Instrument 1	Location	: Vial 98
Injection Date	: 4/19/2012 8:54:52 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\DATA\LQH\LQH-7-11-13-15\LQH-7-11-13-15 2012-04-19 19-33-38\ASH-10-90-1ML-220NM-25MIN.M
Last changed : 9/10/2011 12:27:52 PM by LTL
Analysis Method : D:\LC\DATA\LQH\LQH-7-11-13-15\LQH-7-11-13-15 2012-04-19 19-33-38\098-0401.D\DA.M (ASH-10-90-1ML-220NM-25MIN.M)
Last changed : 9/6/2012 10:25:07 AM by thl
(modified after loading)



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

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

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	10.008	FM	0.6702	458.94275	11.41303	1.4392
2	12.765	MF	0.9743	3.14290e4	537.61847	98.5608

Totals : 3.18879e4 549.03150

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

Data File D:\LC\DATA\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\063-0301.D
Sample Name: LQH-7-13B

=====

Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 63
Injection Date	: 3/29/2012 10:37:33 PM	Inj	: 1
		Inj Volume	: 5 µl

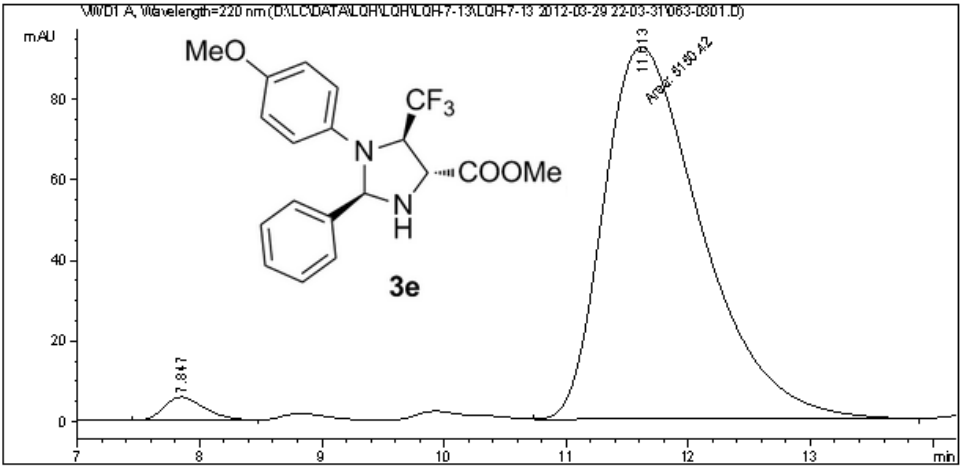
Acq. Method : D:\LC\201201\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\ASH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:17:27 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\063-0301.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)

Last changed : 4/19/2012 2:39:10 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	Height [mAU]	Area %
1	7.847	BV	0.3574	136.32626	5.79601	2.5786
2	11.613	MM	0.9294	5150.42383	92.36421	97.4214

Totals : 5286.75009 98.16022

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

Instrument 1 4/19/2012 2:39:14 PM THL

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\062-0201.D
Sample Name: LQH-7-13A

=====

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 62
Injection Date	: 3/29/2012 10:16:11 PM	Inj	: 1
		Inj Volume	: 5 µl

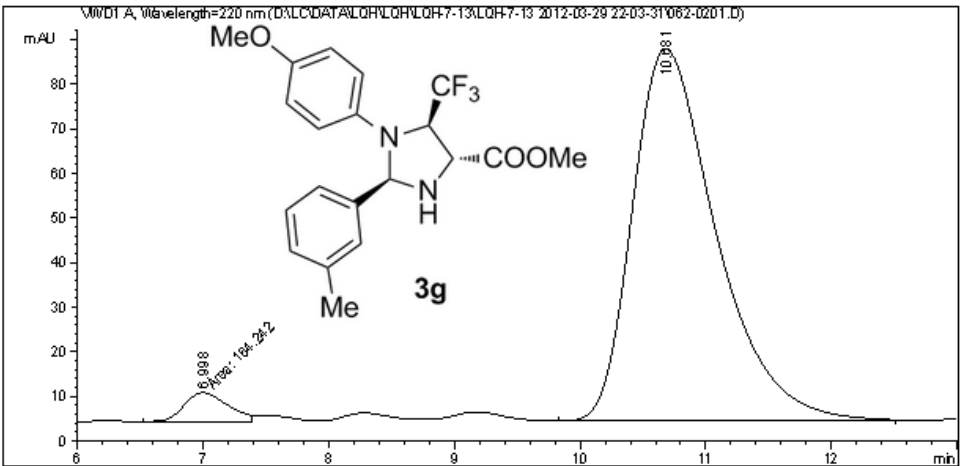
Acq. Method : D:\LC\201201\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\ASH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:17:27 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-7-13\LQH-7-13 2012-03-29 22-03-31\062-0201.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)

Last changed : 5/5/2012 4:19:16 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: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	6.998	MF	0.4128	164.24181	6.63117	4.0823
2	10.681	VB	0.7027	3859.04077	83.42413	95.9177

Totals : 4023.28258 90.05529

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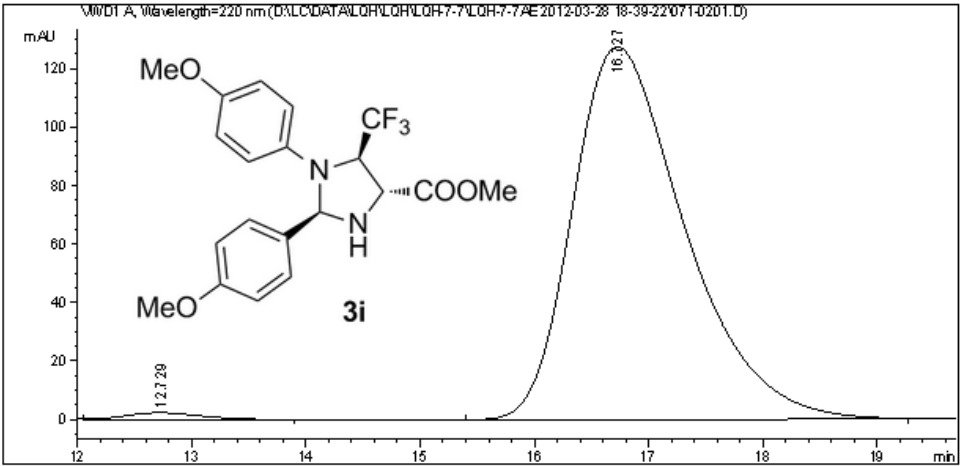
*** End of Report ***

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Data File D:\LC\DATA\LQH\LQH-7-7\LQH-7-7AE 2012-03-28 18-39-22\071-0201.D
Sample Name: LQH-7-7A

=====

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 3/28/2012 6:51:59 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\LQH\LQH-7-7\LQH-7-7AE 2012-03-28 18-39-22\ASH-10-90-10ML-220NM-20MIN.M		
Last changed	: 8/29/2011 8:17:27 PM by LTL		
Analysis Method	: D:\LC\DATA\LQH\LQH-7-7\LQH-7-7AE 2012-03-28 18-39-22\071-0201.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)		
Last changed	: 4/27/2012 11:39:14 AM 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: WWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	12.729	VB	0.6835	118.02040	2.44702	1.3372
2	16.727	BB	1.0437	8708.21582	127.37482	98.6628

Totals : 8826.23622 129.82184

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

Data File D:\LC\DATA\LQH\LQH-8-33\LQH-8-33A 2012-06-21 10-11-38\099-0101.D
Sample Name: LQH-8-33A

=====

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 99
Injection Date	: 6/21/2012 10:13:17 AM	Inj	: 1
		Inj Volume	: 5 µl

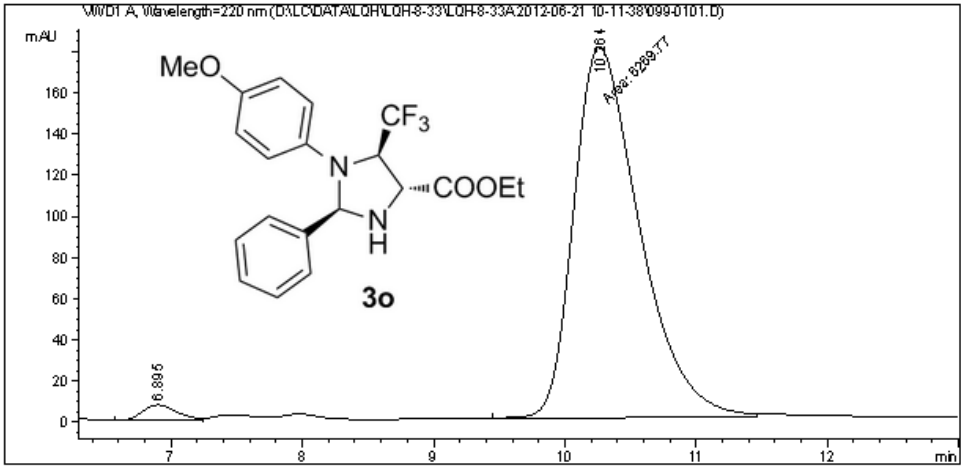
Acq. Method : D:\LC\DATA\LQH\LQH-8-33\LQH-8-33A 2012-06-21 10-11-38\ASH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:17:27 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-8-33\LQH-8-33A 2012-06-21 10-11-38\099-0101.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)

Last changed : 6/26/2012 5:02:45 PM by YDC
(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	Height [mAU]	Area %
1	6.895	W	0.2870	139.97893	7.52152	2.1838
2	10.264	MF	0.5801	6269.77197	180.12724	97.8162

Totals : 6409.75090 187.64876

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

Instrument 1 6/26/2012 5:02:52 PM YDC

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-8-20\LQH-8-20 2012-06-15 16-44-39\081-0201.D
Sample Name: LQH-8-20

=====

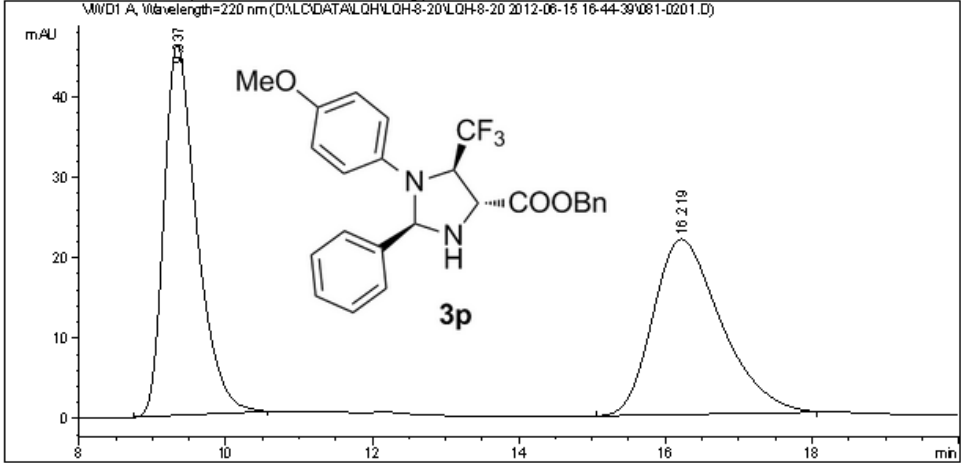
Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 81
Injection Date	: 6/15/2012 4:56:33 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\DATA\LQH\LQH-8-20\LQH-8-20 2012-06-15 16-44-39\ASH-10-90-10ML-220NM.M

Last changed : 8/27/2011 9:19:02 AM by LQH

Analysis Method : D:\LC\DATA\LQH\LQH-8-20\LQH-8-20 2012-06-15 16-44-39\081-0201.D\DA.M (ASH-10-90-10ML-220NM.M)

Last changed : 6/26/2012 4:47:04 PM by YDC
(modified after loading)



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

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

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	9.337	BB	0.4697	1438.75793	46.19259	50.4188
2	16.219	BB	0.9622	1414.85535	21.80758	49.5812

Totals : 2853.61328 68.00017

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

Data File D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\084-0201.D
Sample Name: LQH-8-95

=====

Acq. Operator	: THL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 84
Injection Date	: 8/30/2012 11:07:01 AM	Inj	: 1
		Inj Volume	: 5 µl

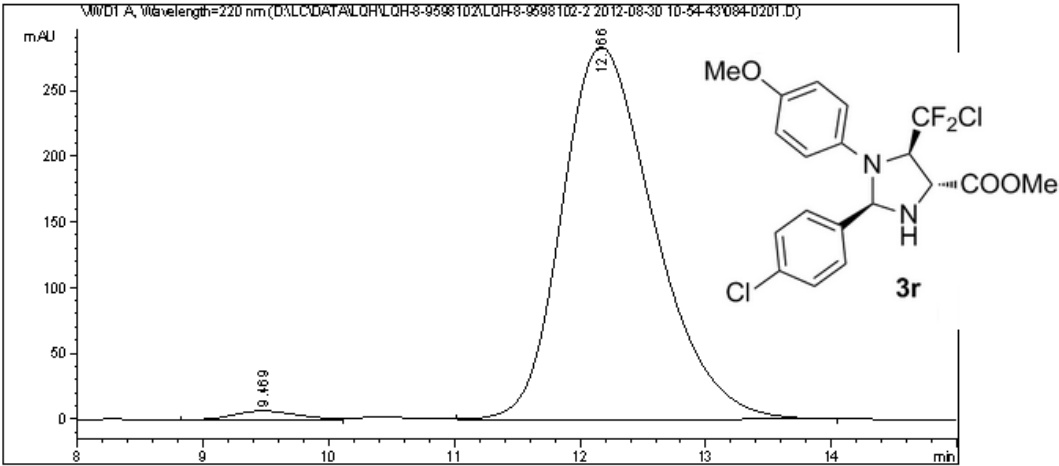
Acq. Method : D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\ASH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:17:27 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\084-0201.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)

Last changed : 8/30/2012 7:27:59 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: WWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	9.469	BV	0.5502	244.45634	6.78117	1.6567
2	12.166	VB	0.7905	1.45110e4	283.63538	98.3433

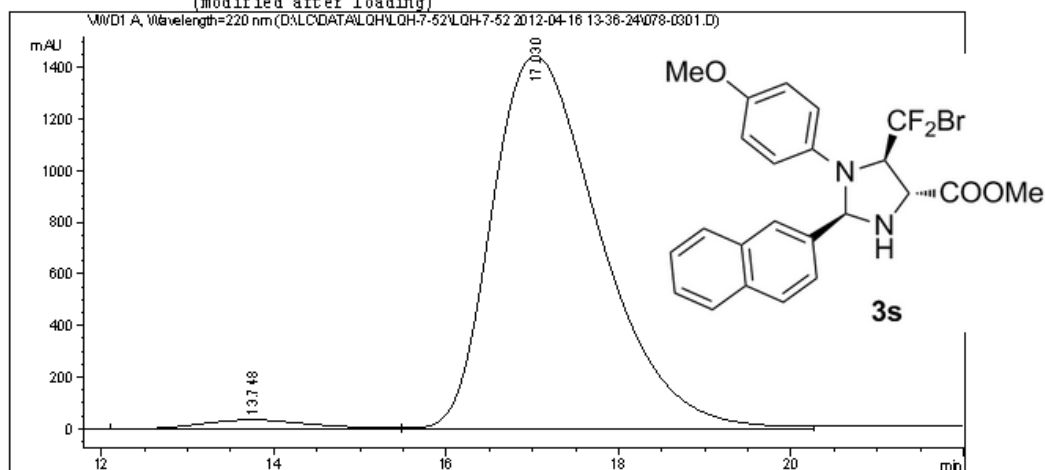
Totals : 1.47555e4 290.41655

=====

*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-7-52\LQH-7-52 2012-04-16 13-36-24\078-0301.D
Sample Name: LQH-7-52B

```
=====
Acq. Operator   : LQH                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : Vial 78
Injection Date  : 4/16/2012 2:15:44 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\DATA\LQH\LQH-7-52\LQH-7-52 2012-04-16 13-36-24\ASH-10-90-10ML-220NM.
                                           M
Last changed    : 4/16/2012 2:14:37 PM by LQH
                  (modified after loading)
Analysis Method : D:\LC\DATA\LQH\LQH-7-52\LQH-7-52 2012-04-16 13-36-24\078-0301.D\DA.M (ASH-
                  10-90-10ML-220NM.M)
Last changed    : 5/5/2012 4:38:02 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	Height [mAU]	Area %
1	13.748	BV	1.3413	3222.45190	35.80244	2.4929
2	17.030	VB	1.3514	1.26045e5	1446.62573	97.5071

Totals : 1.29268e5 1482.42817

*** End of Report ***

Instrument 1 5/5/2012 4:38:08 PM lqh

Page 1 of 1

Data File D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\086-0301.D
Sample Name: LQH-8-102

=====

Acq. Operator	: THL	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 86
Injection Date	: 8/30/2012 11:28:41 AM	Inj	: 1
		Inj Volume	: 5 µl

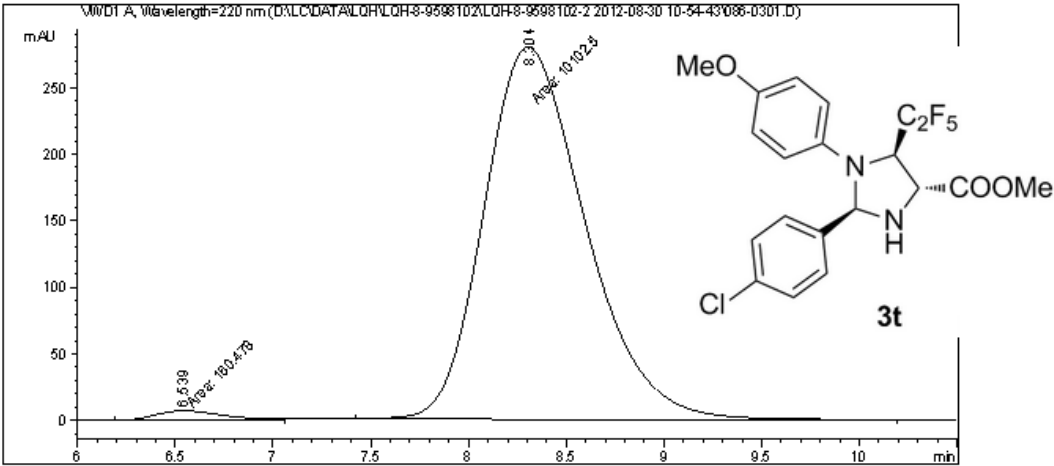
Acq. Method : D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\ASH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:17:27 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-8-9598102\LQH-8-9598102-2 2012-08-30 10-54-43\086-0301.D\DA.M (ASH-10-90-10ML-220NM-20MIN.M)

Last changed : 8/30/2012 7:35:21 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: WWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	6.539	PM	0.3826	160.47826	6.99069	1.5637
2	8.304	PM	0.6017	1.01025e4	279.85178	98.4363

Totals : 1.02630e4 286.84246

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

Data File D:\LC\DATA\LQH\LQH-8-107\LQH-8-107-ODH 2012-09-01 08-58-57\093-0301.D
Sample Name: LQH-8-107

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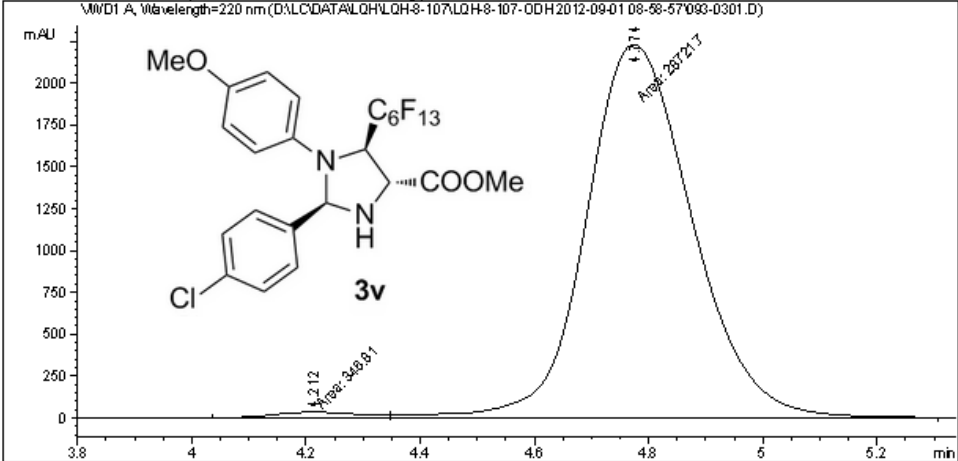
Acq. Operator	: LQH	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 93
Injection Date	: 9/1/2012 9:33:04 AM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\DATA\LQH\LQH-8-107\LQH-8-107-ODH 2012-09-01 08-58-57\ODH-10-90-10ML-220NM-10MIN.M

Last changed : 9/1/2012 8:55:50 AM by LQH

Analysis Method : D:\LC\DATA\LQH\LQH-8-107\LQH-8-107-ODH 2012-09-01 08-58-57\093-0301.D\DA.M (ODH-10-90-10ML-220NM-10MIN.M)

Last changed : 9/4/2012 1:04:11 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	Height [mAU]	Area %
1	4.212	MF	0.1721	346.81021	33.58257	1.1931
2	4.774	FM	0.2146	2.87217e4	2230.45142	98.8069

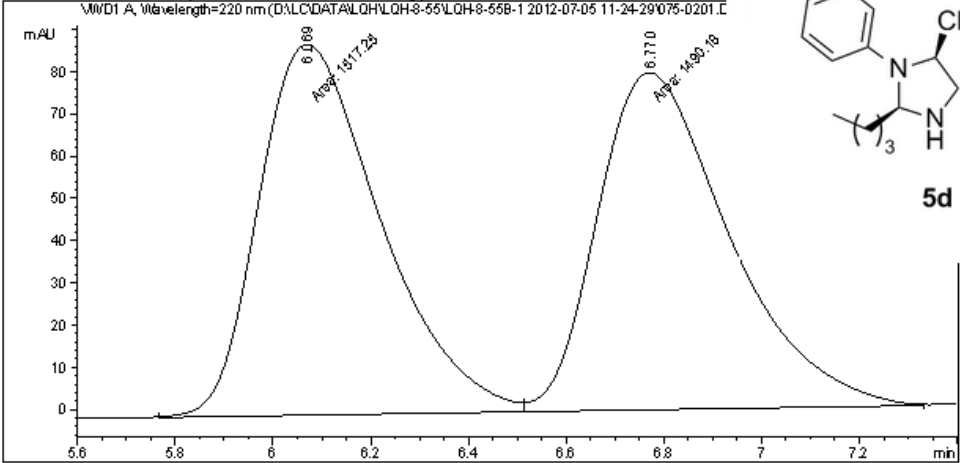
Totals : 2.90686e4 2264.03399

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*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-8-55\LQH-8-55B-1 2012-07-05 11-24-29\075-0201.D
Sample Name: LQH-8-55B

=====

Acq. Operator	: HZL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 75
Injection Date	: 7/5/2012 11:37:11 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LQH\LQH-8-55\LQH-8-55B-1 2012-07-05 11-24-29\ADH-5-95-10ML-220NM.M		
Last changed	: 9/8/2011 7:40:46 PM by LTL		
Analysis Method	: D:\LC\DATA\LQH\LQH-8-55\LQH-8-55B-1 2012-07-05 11-24-29\075-0201.D\A.M (ADH-5-95-10ML-220NM.M)		
Last changed	: 7/24/2012 6:19:18 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	6.069	MF	0.2881	1517.25439	87.78556	50.4501
2	6.770	FM	0.3112	1490.18420	79.80413	49.5499

Totals : 3007.43860 167.58969

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

Data File D:\LC\DATA\LQH\LQH-8-36\LQH-8-36 2012-06-22 08-32-57\087-0201.D
Sample Name: LQH-8-36B

=====

Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 87
Injection Date	: 6/22/2012 8:45:38 AM	Inj	: 1
		Inj Volume	: 5 µl

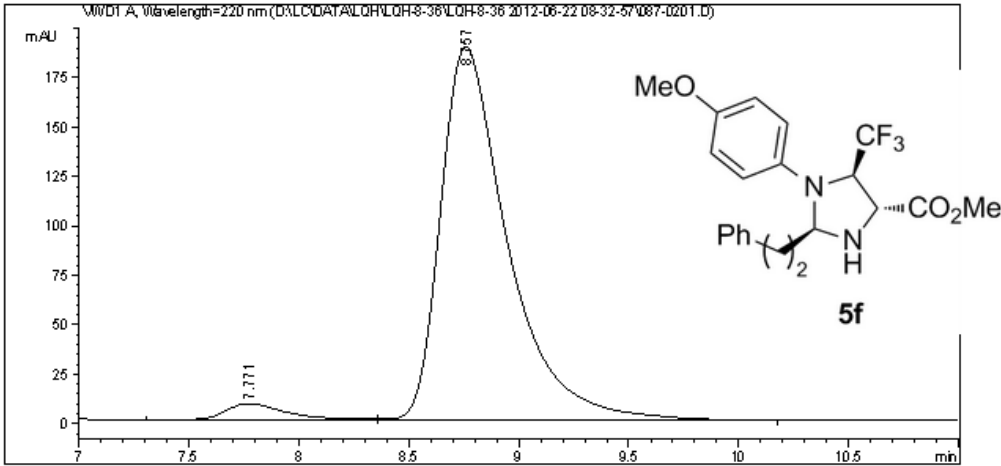
Acq. Method : D:\LC\DATA\LQH\LQH-8-36\LQH-8-36 2012-06-22 08-32-57\ADH-10-90-10ML-220NM-20MIN.M

Last changed : 8/29/2011 8:14:57 PM by LTL

Analysis Method : D:\LC\DATA\LQH\LQH-8-36\LQH-8-36 2012-06-22 08-32-57\087-0201.D\DA.M (ADH-10-90-10ML-220NM-20MIN.M)

Last changed : 6/26/2012 5:16:07 PM by YDC
(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: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.771	BV	0.2959	170.97664	8.44586	4.0805
2	8.757	VB	0.3152	4019.11279	188.87428	95.9195

Totals : 4190.08943 197.32014

=====

*** End of Report ***

Data File D:\LC\DATA\LQH\LQH-7-93\LQH-7-93 2012-05-16 19-01-36\082-0201.D
Sample Name: LQH-7-93

=====

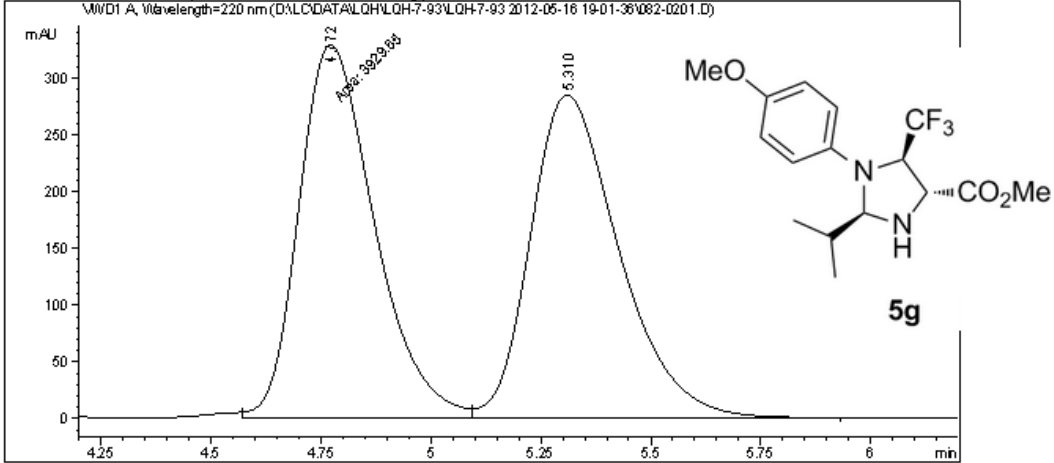
Acq. Operator	: LQH	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 82
Injection Date	: 5/16/2012 7:13:22 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\DATA\LQH\LQH-7-93\LQH-7-93 2012-05-16 19-01-36\ASH-10-90-10ML-220NM.M

Last changed : 8/27/2011 9:19:02 AM by LQH

Analysis Method : D:\LC\DATA\LQH\LQH-7-93\LQH-7-93 2012-05-16 19-01-36\082-0201.D\DA.M (ASH-10-90-10ML-220NM.M)

Last changed : 6/26/2012 6:03:01 PM by YDC
(modified after loading)



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

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

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	4.772	FM	0.1988	3929.65063	329.40140	49.6393
2	5.310	VB	0.2142	3986.76709	284.93958	50.3607

Totals : 7916.41772 614.34097

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

Data File D:\LC\DATA\LQH\LQH-7-73\LQH-7-73 2012-05-05 08-35-24\094-0201.D
Sample Name: LQH-7-73

=====

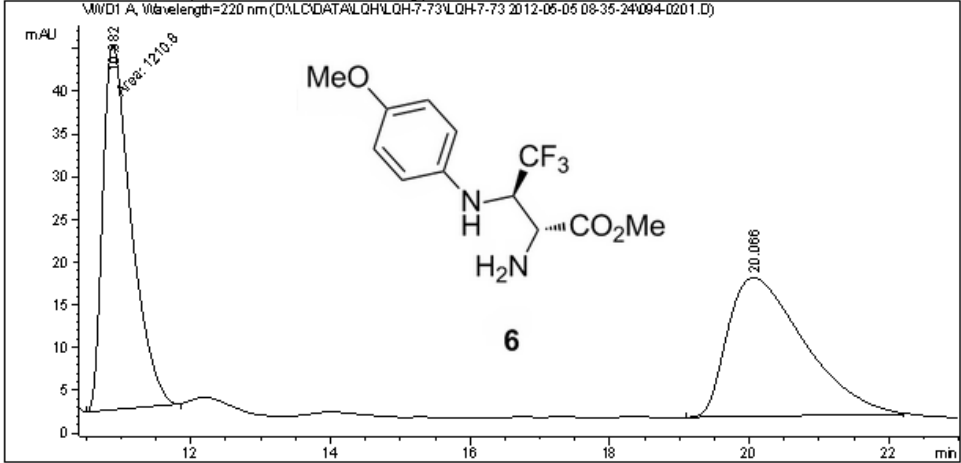
Acq. Operator	: lqh	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 94
Injection Date	: 5/5/2012 8:47:17 AM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : D:\LC\DATA\LQH\LQH-7-73\LQH-7-73 2012-05-05 08-35-24\ASH-10-90-10ML-220NM.M

Last changed : 8/27/2011 9:19:02 AM by LQH

Analysis Method : D:\LC\DATA\LQH\LQH-7-73\LQH-7-73 2012-05-05 08-35-24\094-0201.D\DA.M (ASH-10-90-10ML-220NM.M)

Last changed : 7/9/2012 11:04:22 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: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	10.882	MM	0.4716	1210.79785	42.79110	49.3992
2	20.066	BB	1.0730	1240.24780	16.21302	50.6008

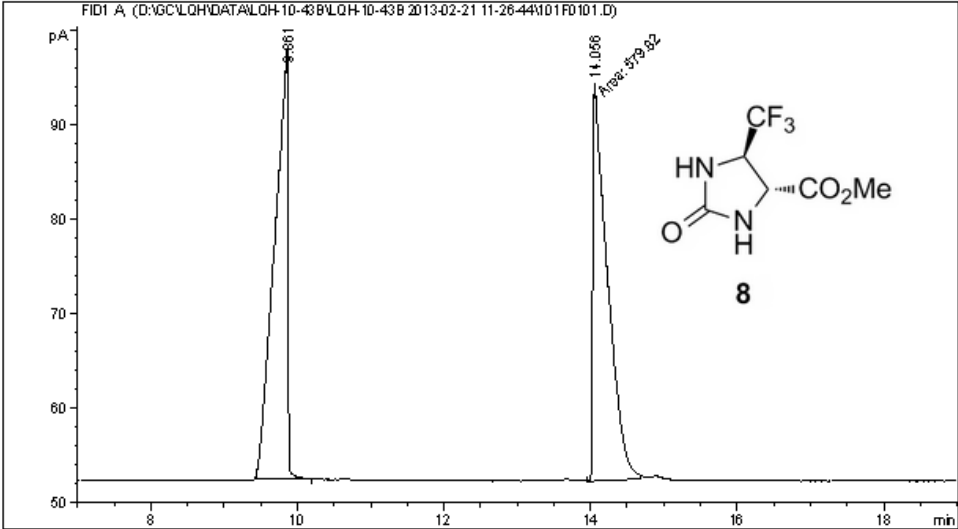
Totals : 2451.04565 59.00412

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

Data File D:\GC\LQH\DATA\LQH-10-43B\LQH-10-43B 2013-02-21 11-26-44\101F0101.D
Sample Name: LQH-10-43B

=====

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 101
Injection Date	: 21-Feb-13, 11:28:27	Inj	: 1
		Inj Volume	: 2 µl
Acq. Method	: D:\GC\LQH\DATA\LQH-10-43B\LQH-10-43B 2013-02-21 11-26-44\BETA-DEX325-1700C.M		
Last changed	: 2/21/2013 11:25:40 AM by LQH		
Analysis Method	: D:\GC\DEF GC OFF.M		
Last changed	: 2/21/2013 12:04:24 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: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	9.861	BB	0.1546	581.82855	45.66846	50.08646
2	14.056	MM	0.2295	579.81989	42.10339	49.91354

Totals : 1161.64844 87.77185

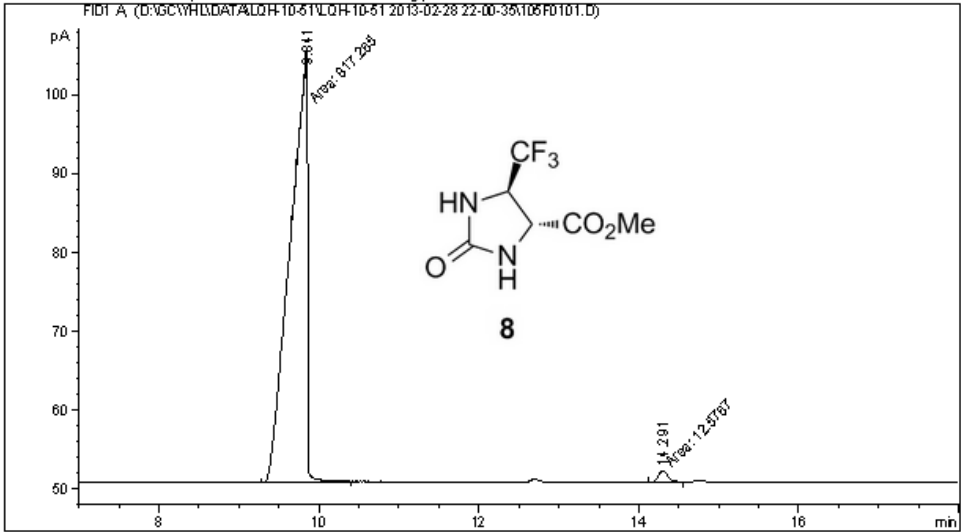
=====
*** End of Report ***

Data File D:\GC\YHL\DATA\LQH-10-51\LQH-10-51 2013-02-28 22-00-35\105F0101.D
Sample Name: LQH-10-51

=====

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 105
Injection Date	: 28-Feb-13, 22:02:18	Inj	: 1
		Inj Volume	: 2 µl

Acq. Method : D:\GC\YHL\DATA\LQH-10-51\LQH-10-51 2013-02-28 22-00-35\BETA-DEX325-1700C.M
Last changed : 2/21/2013 11:25:40 AM by LQH
Analysis Method : D:\GC\DEF GC OFF.M
Last changed : 2/28/2013 10:24:32 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: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	9.841	MM	0.2480	817.26520	54.92947	98.48445
2	14.291	MM	0.1375	12.57668	1.52396	1.51555

Totals : 829.84188 56.45343

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