

Supporting Information For

An Efficient Coupling of *N*-Tosylhydrazones with 2-Halopyridines: Synthesis of 2- α -Styrylpyridines Endowed with Antitumor Activity

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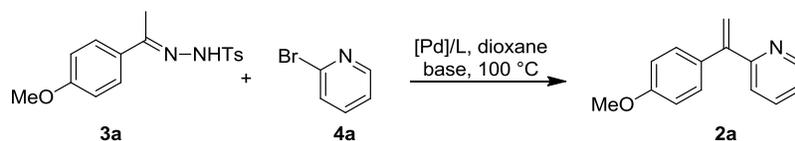
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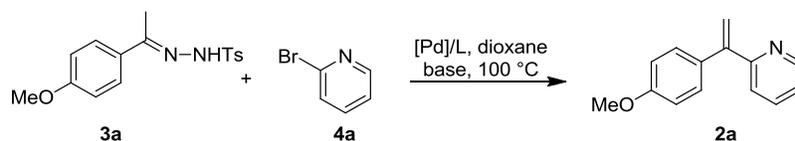
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Table 1. Optimization Coupling Reaction of *N*-Tosylhydrazones 3a with 2-Bromopyridine 4a under Various Conditions.^a



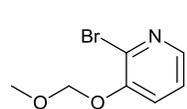
Essais	[Pd]	Ligand	Solvent	Base	Yield of 2a ^b	
1	Pd₂dba₃	Xphos	dioxane	LiO ^t Bu	15	Effect of base source
2	PdCl₂(MeCN)₂	DPPP	dioxane	Cs ₂ CO ₃	22	
3	PdCl ₂ (MeCN) ₂	DPPP	dioxane	LiO ^t Bu	42	
4	PdCl ₂ (MeCN) ₂	DPPP	dioxane	NaO ^t Bu	32	
5	PdCl ₂ (MeCN) ₂	DPPP	dioxane	KO ^t Bu	27	
6	PdCl ₂ (MeCN) ₂	DPPP	dioxane	NaOMe	5	
7	PdCl ₂ (MeCN) ₂	DPPB	dioxane	LiO ^t Bu	30	Effect of ligand source
8	PdCl ₂ (MeCN) ₂	DPPE	dioxane	LiO ^t Bu	28	
9	PdCl ₂ (MeCN) ₂	DPPM	dioxane	LiO ^t Bu	27	
10	PdCl₂(MeCN)₂	DPPF	dioxane	LiO^tBu	84^c	
11	PdCl ₂ (MeCN) ₂	D ^t PrPF	dioxane	LiO ^t Bu	25	
12	PdCl ₂ (MeCN) ₂	D ^t BPF	dioxane	LiO ^t Bu	28	
13	PdCl ₂ (MeCN) ₂	PPh ₃	dioxane	LiO ^t Bu	33	
14	PdCl ₂ (MeCN) ₂	DPEPhos	dioxane	LiO ^t Bu	32	
15	PdCl ₂ (MeCN) ₂	JohnPhos	dioxane	LiO ^t Bu	6	
16	PdCl ₂ (MeCN) ₂	DavePhos	dioxane	LiO ^t Bu	44	
17	PdCl ₂ (MeCN) ₂	PCy ₃	dioxane	LiO ^t Bu	31	
18	PdCl ₂ (MeCN) ₂	^t Bu ₃ P-HBF ₄	dioxane	LiO ^t Bu	8	
19	PdCl₂(MeCN)₂	^tBu₂MeP-HBF₄	dioxane	LiO^tBu	82	



Essais	[Pd]	Ligand	Solvent	Base	Yield of 2a ^b	
20	PdCl ₂ (MeCN) ₂	DPPF	dioxane	LiO ^t Bu	84	Effect of solvent
21	PdCl ₂ (MeCN) ₂	DPPF	CPME ^d	LiO ^t Bu	46	
22	PdCl ₂ (MeCN) ₂	DPPF	Toluene	LiO ^t Bu	51	
23	PdCl ₂ (MeCN) ₂	DPPF	THF	LiO ^t Bu	62	
24	PdCl ₂ (MeCN) ₂	DPPF	CH ₃ CN	LiO ^t Bu	60	
25	PdCl ₂ (MeCN) ₂	DPPF	DMF	LiO ^t Bu	46	
26	PdCl ₂ (MeCN) ₂	DPPF	PhF	LiO ^t Bu	40	
27	PdCl ₂ (MeCN) ₂	DPPF	DME	LiO ^t Bu	53	
28	Pd ₂ dba ₃	DPPF	dioxane	LiO ^t Bu	40	Effect of Pd source
29	PdCl ₂ (PhCN) ₂	DPPF	dioxane	LiO ^t Bu	45	
30	Pd(OAc) ₂	DPPF	dioxane	LiO ^t Bu	35	
31	PdCl ₂ (dppf)	-	dioxane	LiO ^t Bu	60	
32	PdCl ₂ (dppf)	^t Bu ₂ MeP-HBF ₄	dioxane	LiO ^t Bu	58	
33	PdCl ₂ (dppf)	DavePhos	dioxane	LiO ^t Bu	51	
34	PdCl ₂ (PPh ₃) ₂	-	dioxane	LiO ^t Bu	30	
35	-	DPPF	dioxane	LiO ^t Bu	0	
36	PdCl ₂ (MeCN) ₂	-	dioxane	LiO ^t Bu	15	

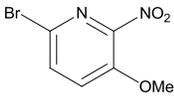
^a The reactions were carried out in a sealed tube with **3a** (1.5 mmol), **4a** (1 mmol), [Pd] (5 mol %), Ligand (10 mol %), base (2.2 equiv) at 100 °C in 3.0 mL of solvent. ^b Isolated yield of **2a**. ^c Performing the coupling with a ratio of Pd/ligand (1:1) give the desired product **2a** in 60% isolated yield. ^d Cyclopentyl methyl ether (CPME).

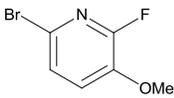
Experimental procedures for the synthesis of starting materials 4d, 4i, 4j.



2-Bromo-3-(methoxymethoxy)pyridine 4d.¹ To a solution of 2-bromopyridin-3-ol (1.0 g, 5.75 mmol, 1.0 equiv) in dry THF (20 mL) in a dry flask under an argon atmosphere was added anhydrous DIPEA (1.5 mL, 8.6 mmol, 1.5 equiv). The suspension was stirred for 15 min then cooled to 5 °C and a solution of technical grade chloromethyl methyl ether (0.8 mL, 8.6 mmol, 1.5 equiv) in dry THF (5 mL) was added in one portion. The suspension was stirred at 5-10 °C for 2 h, then warmed to room temperature and stirred for 15 h at reflux. The reaction was poured into water (60 mL), extracted with diethyl ether (4 x 50 mL), then the combined organic phases were washed with water (1 x 50 mL) and brine (1 x 50 mL) before being dried over MgSO₄. Removal of solvents *in vacuo* yielded a clear oil which was purified by flash chromatography on silica gel (4 x 15 cm) eluting with 1:4 ethyl acetate/hexanes to afford **4d** as colorless crystals, yield 0.68 g, 55%; mp 44-46 °C; TLC: R_f = 0.8 (EtOAc/Cyclohexane, 5/5, SiO₂); IR (neat) 1566, 1453, 1418, 1401, 1308, 1271, 1206, 1155, 1134, 1095, 1069 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ (ppm): 8.00 (dd, *J* = 4.7, 1.5 Hz, 1H), 7.44 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.16 – 7.10 (m, 1H), 5.23 (s, 2H), 3.47 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 149.6 (C), 141.9 (CH), 141.5 (C), 123.6 (CH), 123.2 (CH), 95.1 (OCH₂O), 56.4 (OCH₃); HRMS (ESI): for C₇H₉BrNO₂ (M + H)⁺: *m/z* calcd 217.9817, found 217.9825.

(1) Robert, N.; Hoarau, C.; Célanire, S.; Ribéreau, P.; Godard, A.; Quéguiner, G.; Marsais, F., *Tetrahedron* **2005**, *61*, 4569.

 **6-Bromo-3-methoxy-2-nitropyridine 4i.**² A mixture of 6-bromo-3-hydroxy-2-nitropyridine (2.87 g, 13.71 mmol), and potassium carbonate (3.78 g, 27.4 mol) in acetone (30 mL) was treated with iodomethane (1.8 ml, 28 mmol) and stirred at 50°C. Two further additions of 1.2 equivalents of iodomethane were made over a period of approximately four hours. The solvent was filtered, evaporated, and the residue dissolved in ethyl acetate (50 mL) and washed with water (30 mL), saturated sodium hydrogen carbonate solution (30 mL), water (30 mL) and brine (30 mL). The organic layer was dried (MgSO₄) and evaporated. Purification by column chromatography eluting with 0- 100% ethyl acetate in hexane gave the title compound as a cream yellow solid (3.1 g, 98% yield); mp 98-100 °C; ¹H NMR (300 MHz, CDCl₃) δ (ppm): ¹H NMR (300 MHz,) δ 7.71 (d, *J* = 8.7 Hz, 1H), 7.44 (d, *J* = 8.7 Hz, 1H), 4.00 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 147.5 (C), 133.3 (CH), 128.5 (2C), 125.6 (CH), 57.2 (OCH₃). HRMS (ESI): for C₆H₆BrN₂O₃ (M + H)⁺ calcd 232.9556, found 232.9572.

 **6-Bromo-2-fluoro-3-methoxypyridine 4j.**³ To a stirred solution of 6-bromo-2-fluoropyridin-3-ol (1.55 g, 8.06 mmol) and sodium methoxide (0.46 mg, 8.45 mmol) in DMF (17 mL) was added iodomethane (0.53 mL, 8.45 mmol) at 0 °C, and the mixture was stirred at room temperature for 12 hours. The mixture was treated with H₂O and extracted with ethyl acetate. The combined organic layer was dried and evaporated. The residue was purified by chromatography on silica gel, eluting with ethyl acetate/cyclohexane (5/5) afford compound **4j** as a yellow solid (0.57 g, 44%); mp 58-60 °C. ¹H NMR (300 MHz, CDCl₃) δ (ppm): 7.30 (d, *J* = 8.2 Hz, 1H), 7.18 (dd, *J* = 9.6, 8.2 Hz, 1H), 3.90 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm): 151.9

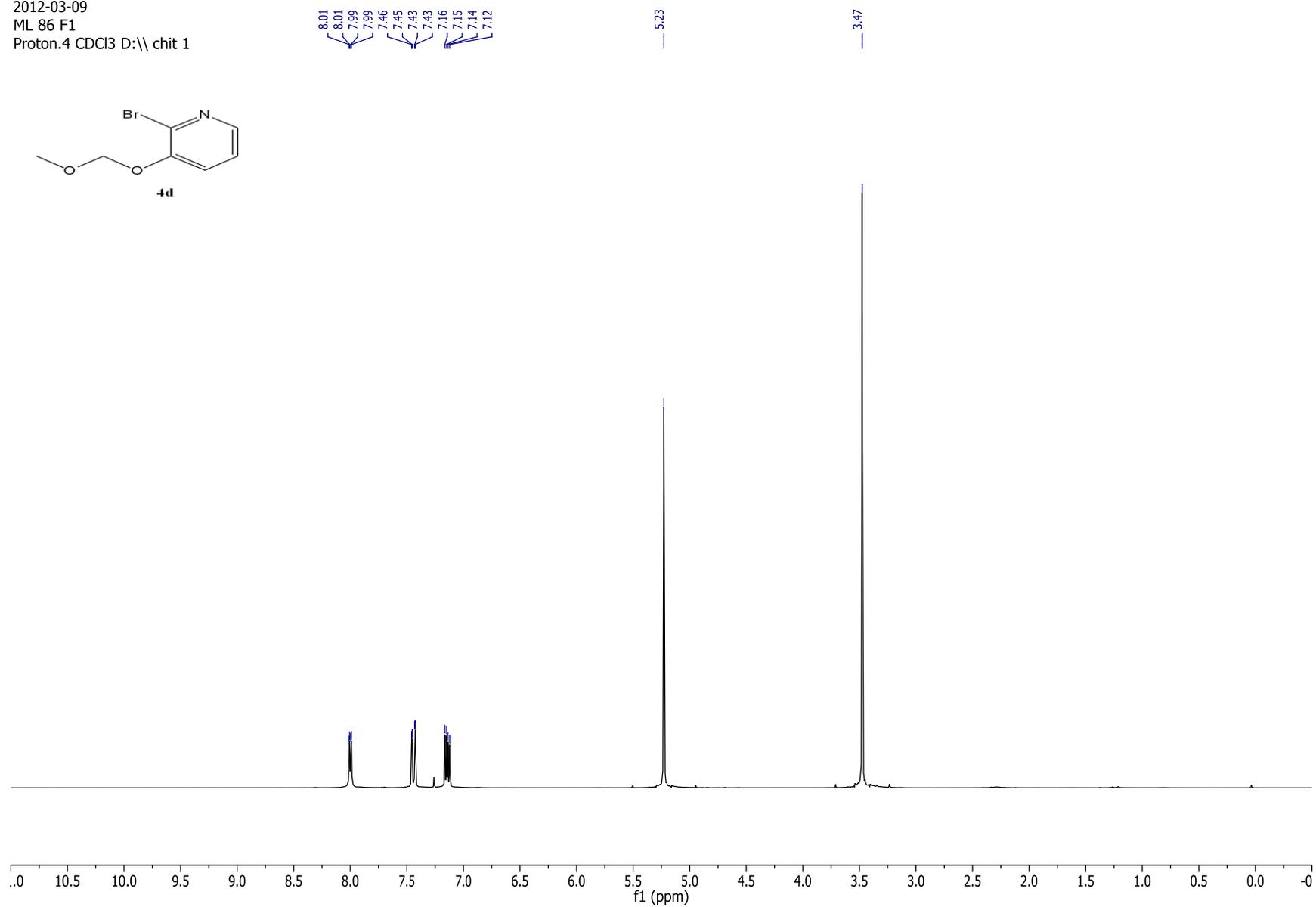
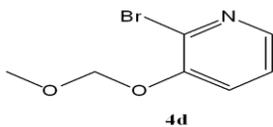
(2) Blaney, E. L.; King, N. P.; Witherington, J. Piperazine derivatives as growth hormone secretagogue (GHS) receptor agonists. WO/2007/113202A1, 2007.

(3) Kazuo, A.; HIROTA, M. 1-2-(4-Hydroxyphenyl)-2-hydroxyethyl-piperidin-4-ol compounds as NMDA receptor antagonists. WO2005035522A1, 2005.

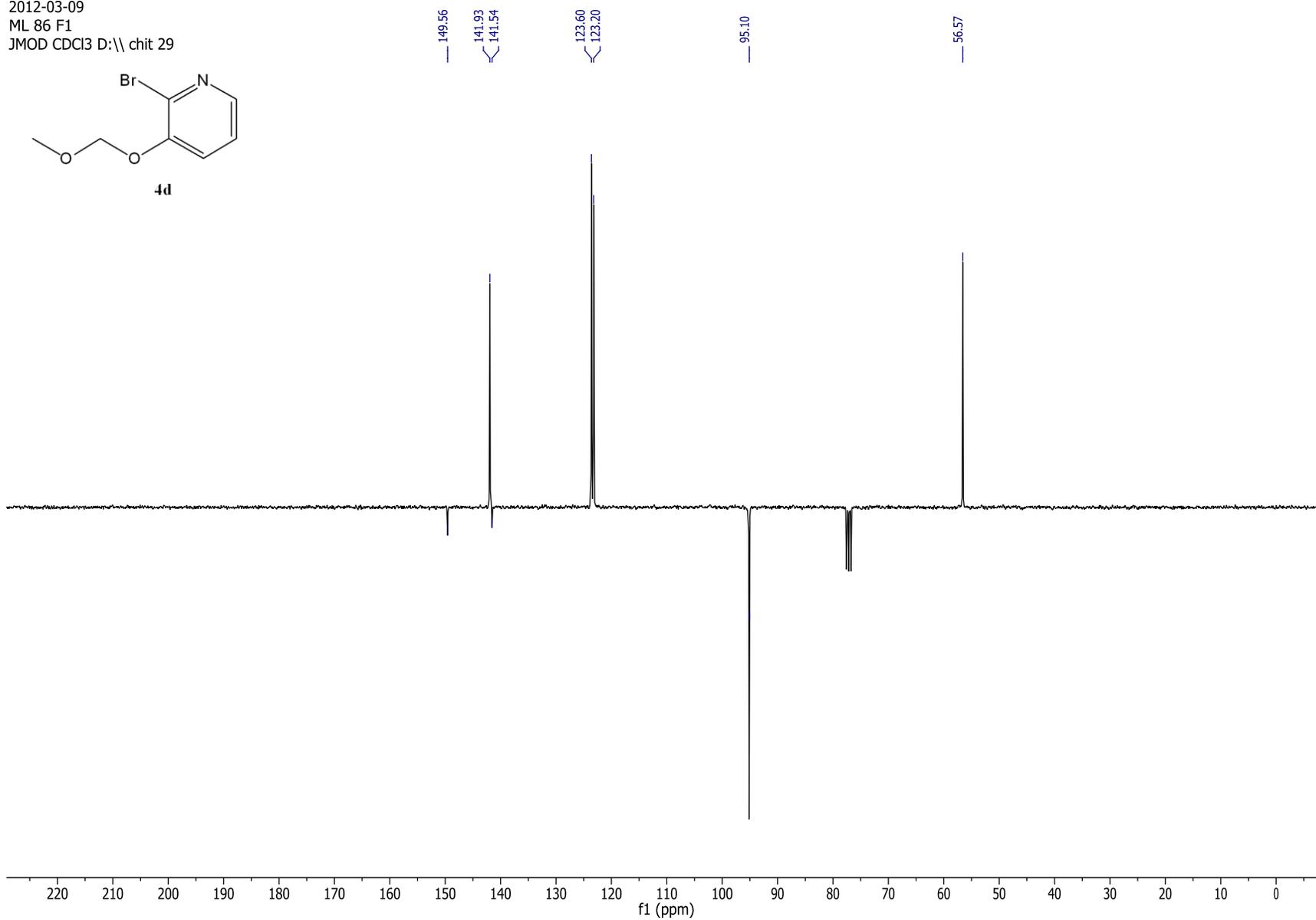
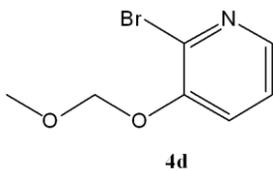
(d, $J = 240$ Hz) (C), 142.45 (d, $J = 23.3$ Hz) (C), 125.6 (d, $J = 2.25$ Hz) (CH), 125.5 (C), 124.2 (d, $J = 4.5$ Hz) (CH), 56.60 (OCH₃); ¹⁹F NMR (188 MHz, CDCl₃) δ -80.03.

^1H NMR and ^{13}C Spectra

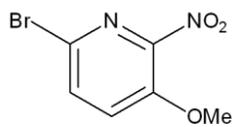
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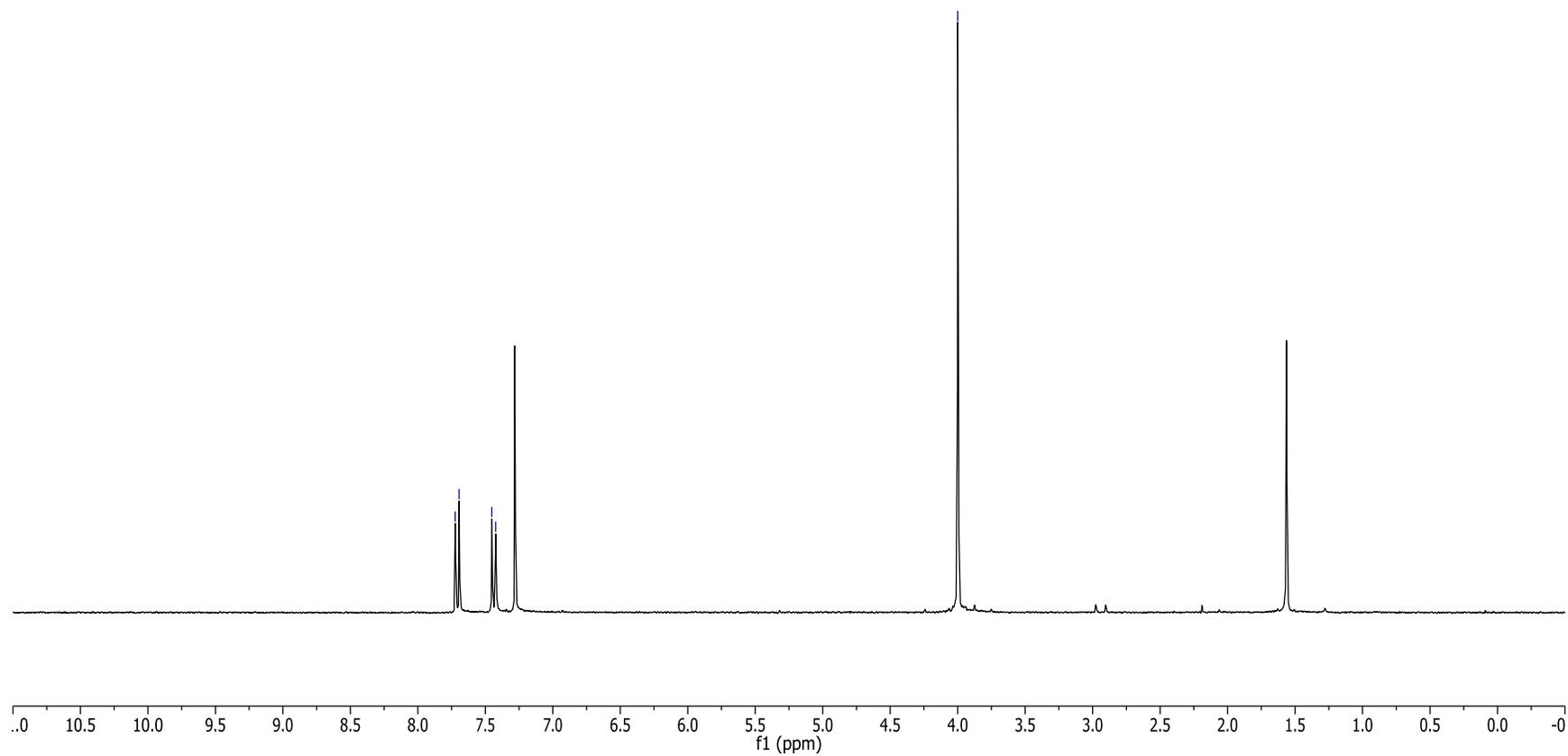
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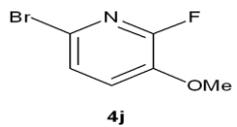
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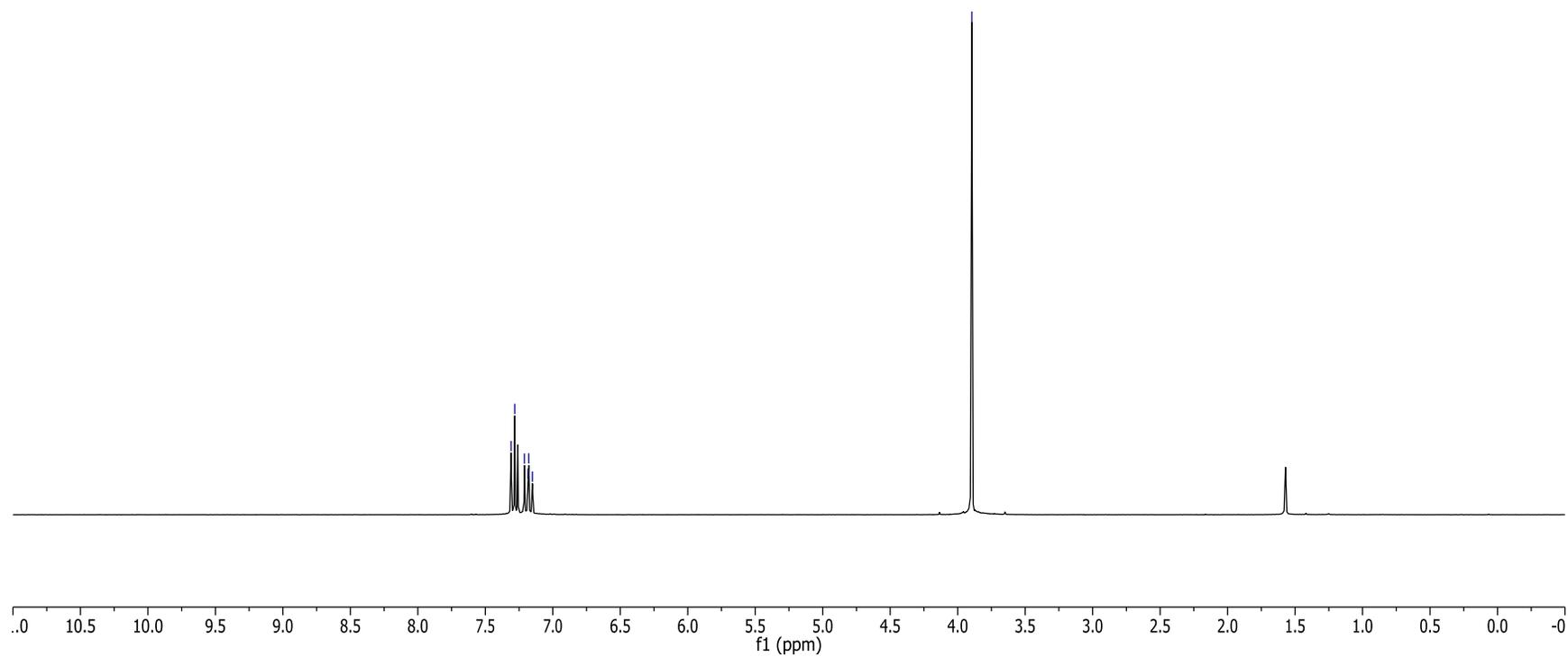


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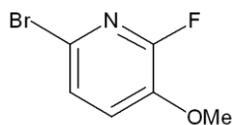


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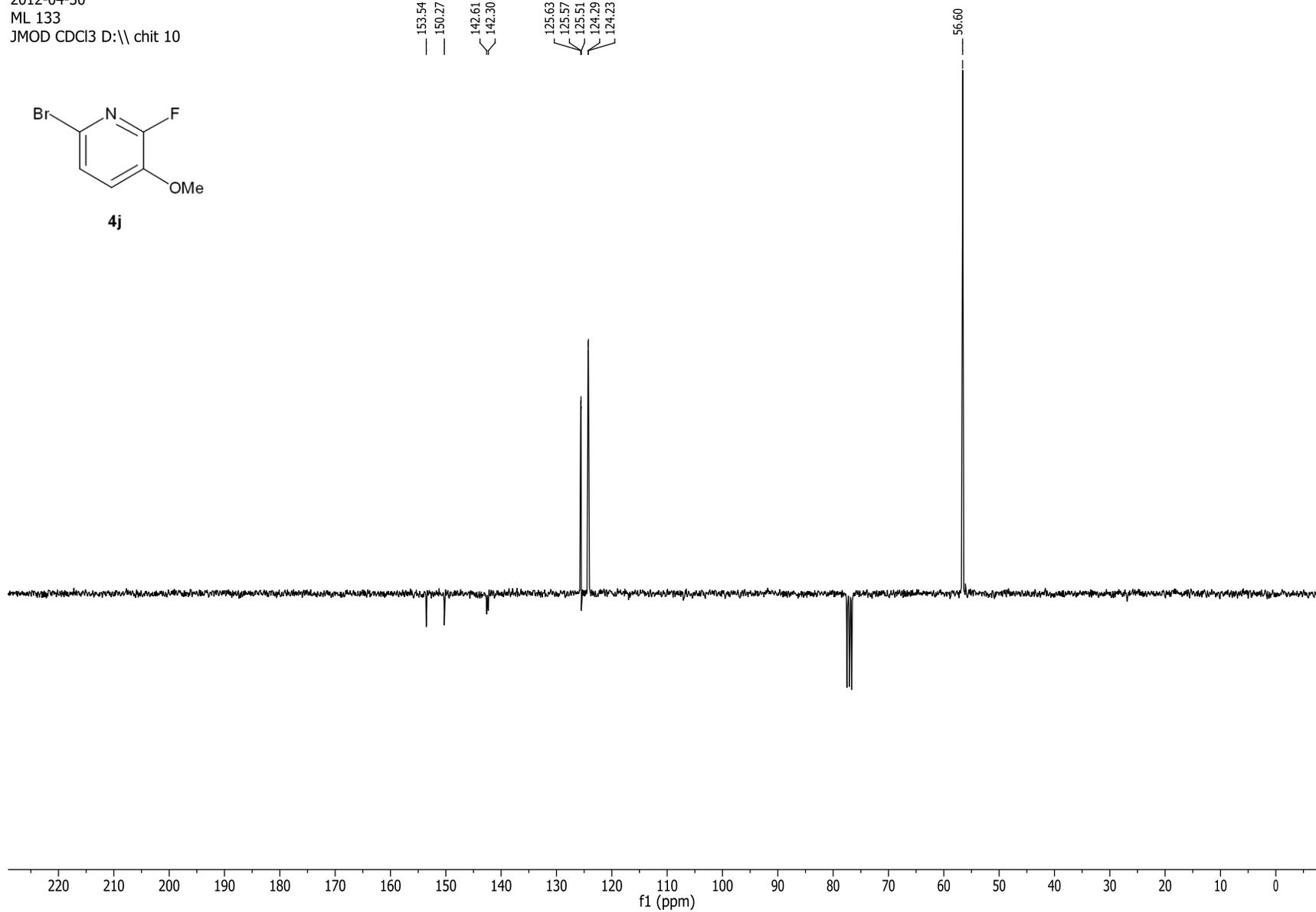


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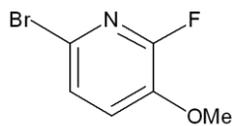


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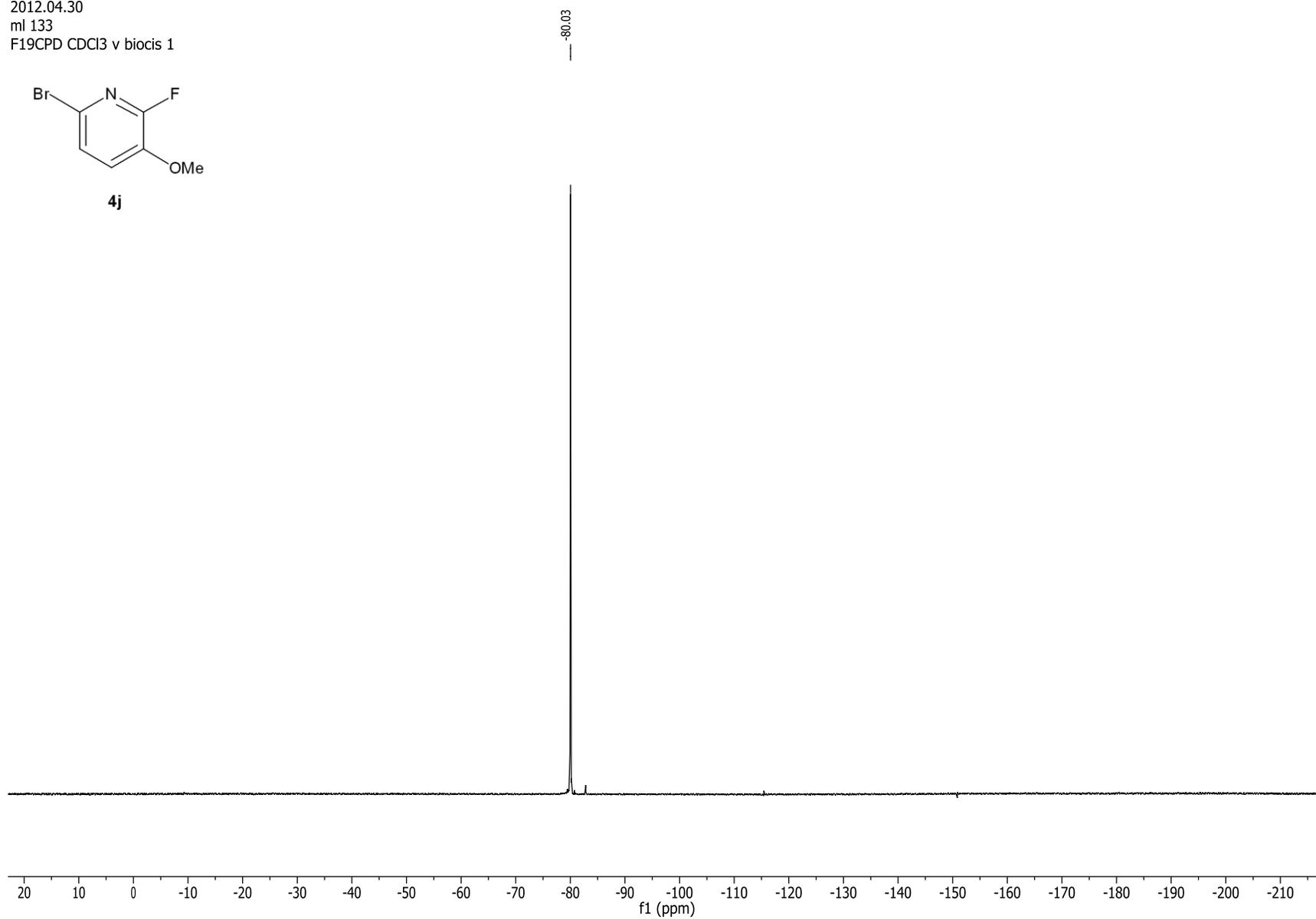
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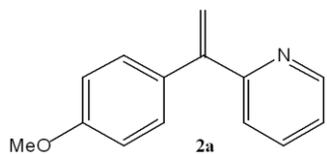
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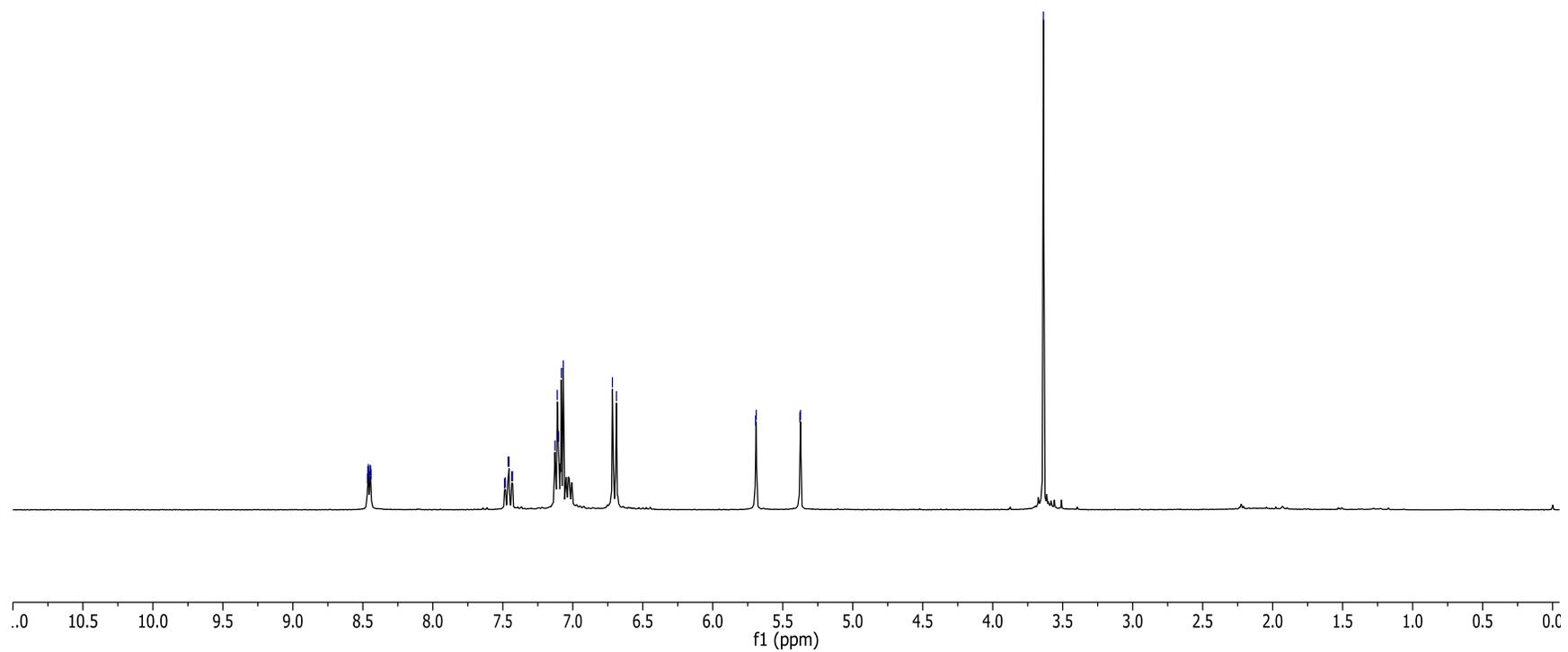
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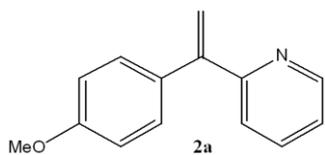
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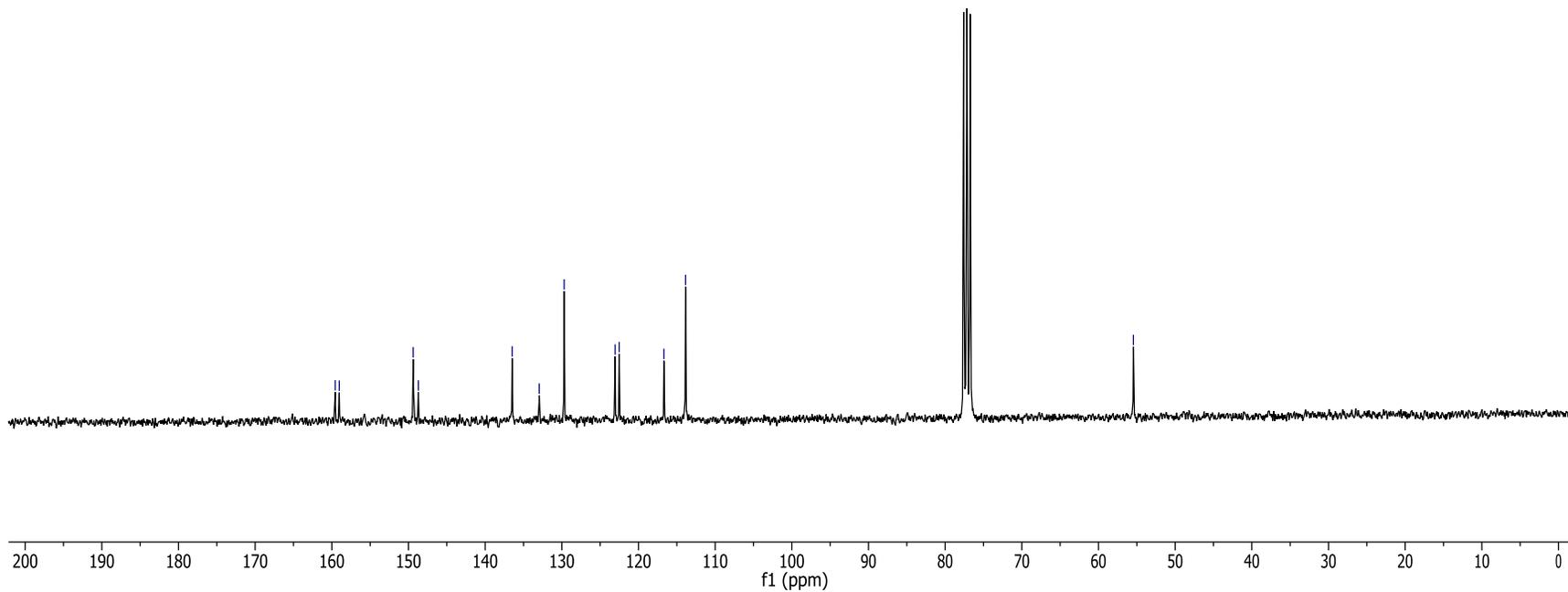
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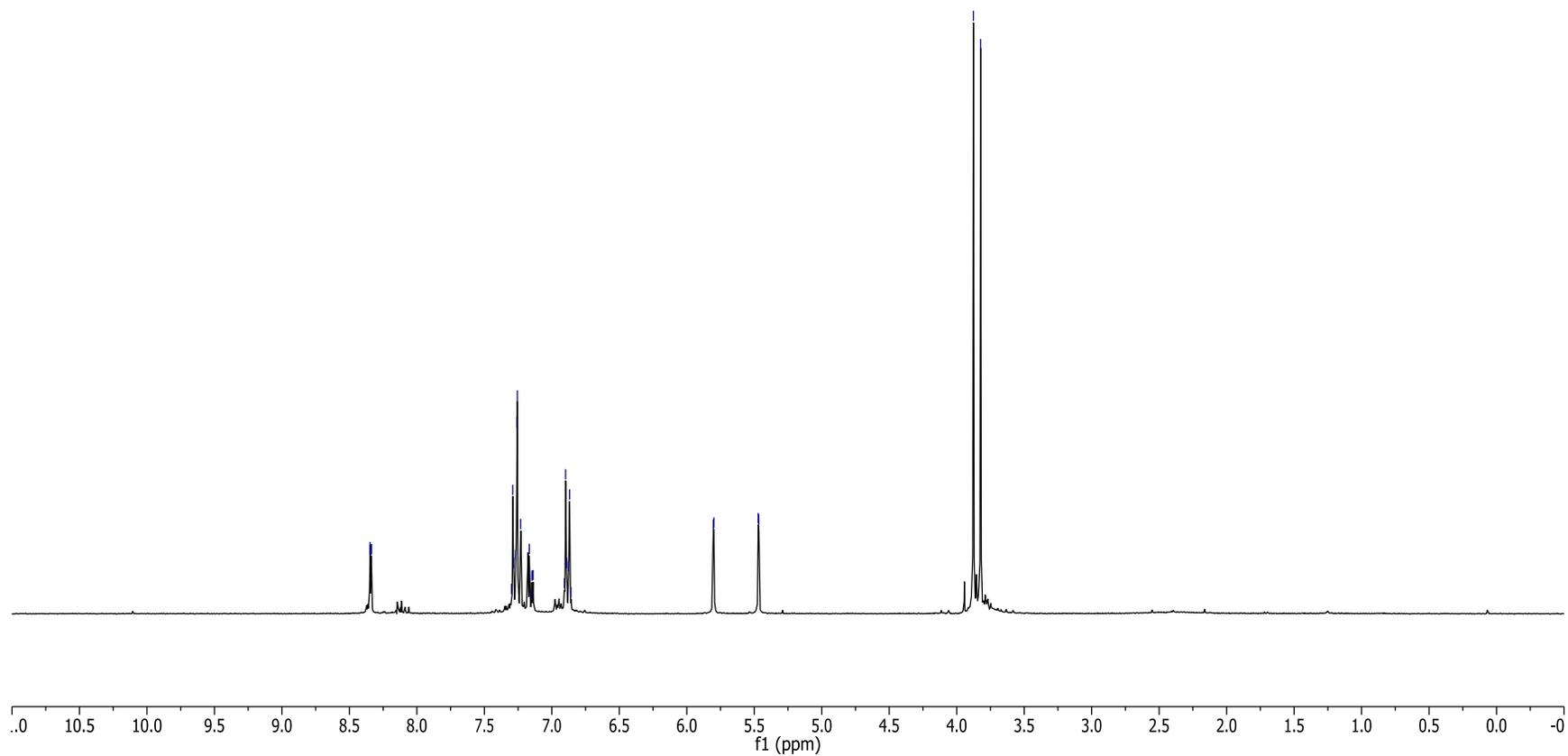
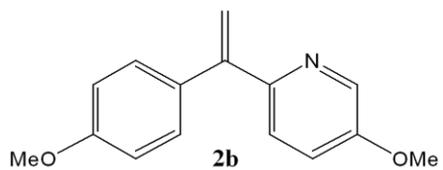
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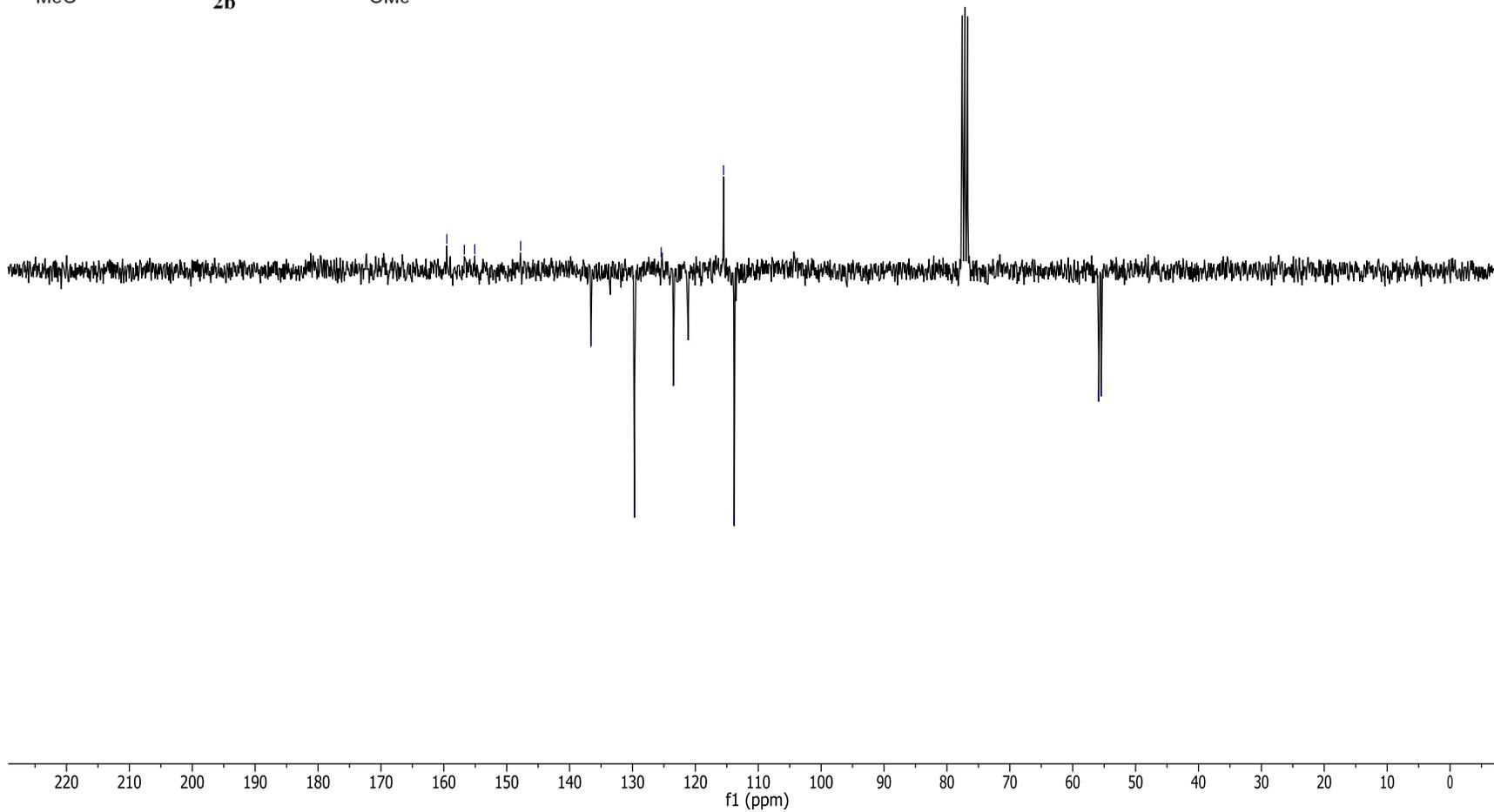
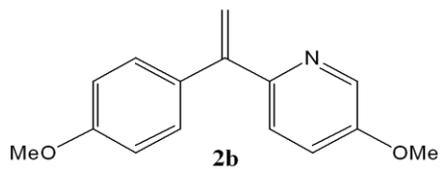
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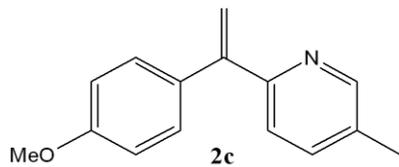
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2012-03-20
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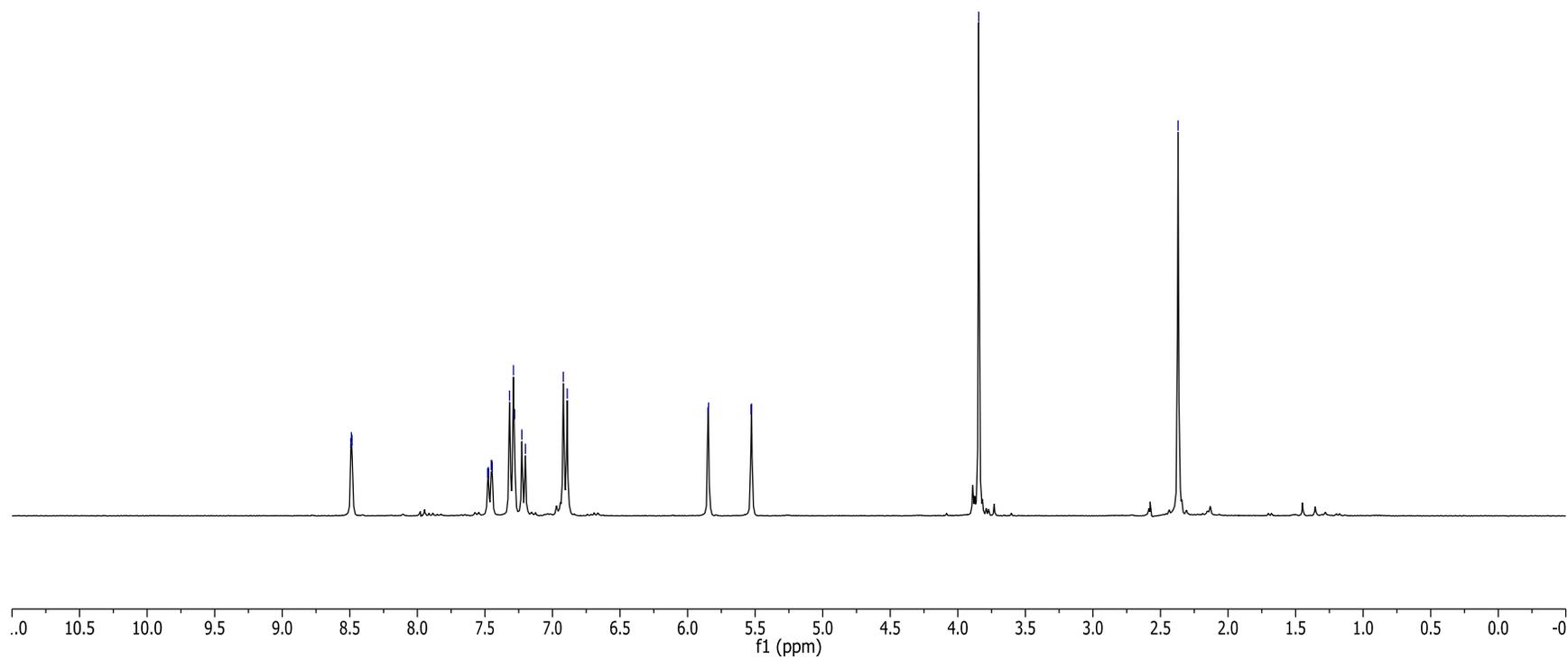


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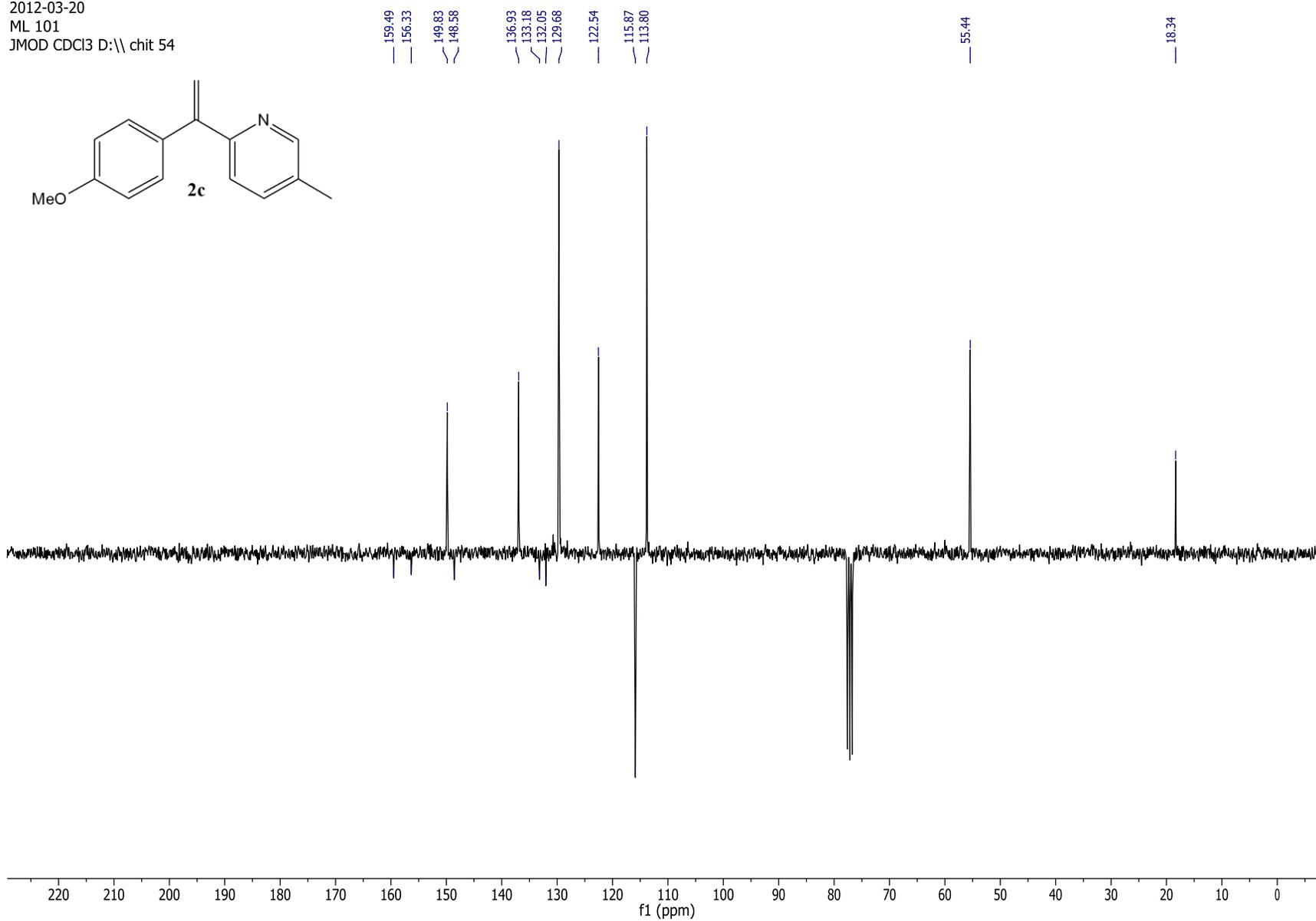
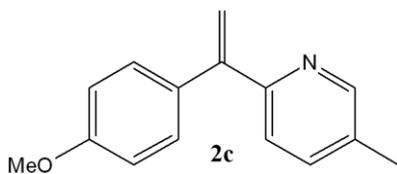
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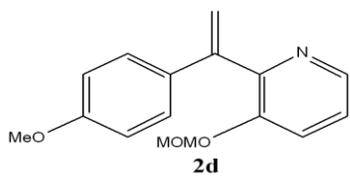
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2012-03-26
ML 107
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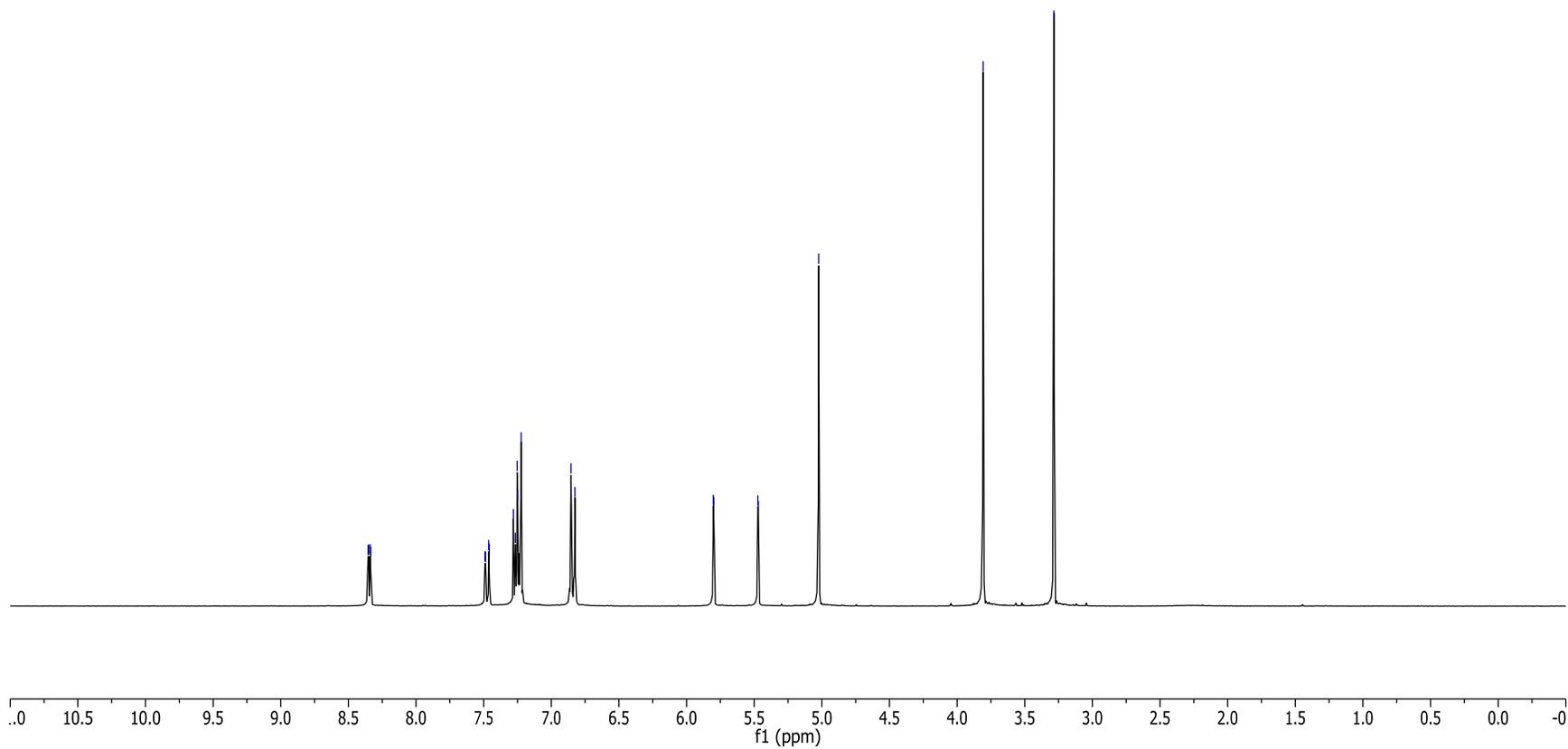
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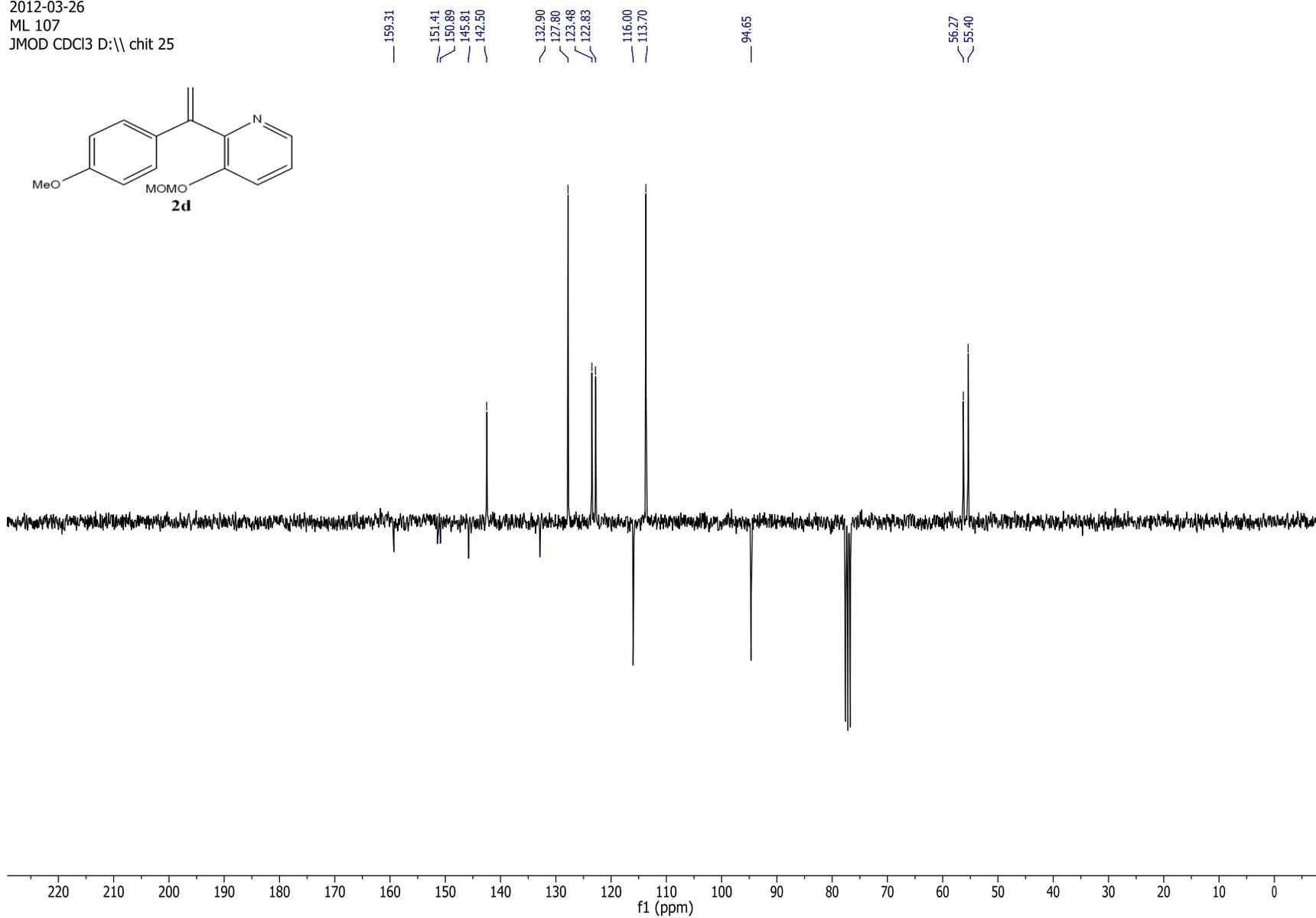
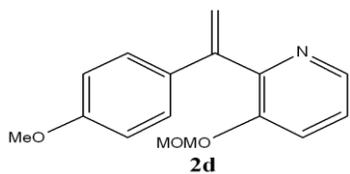
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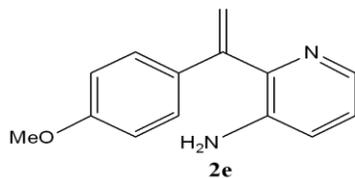
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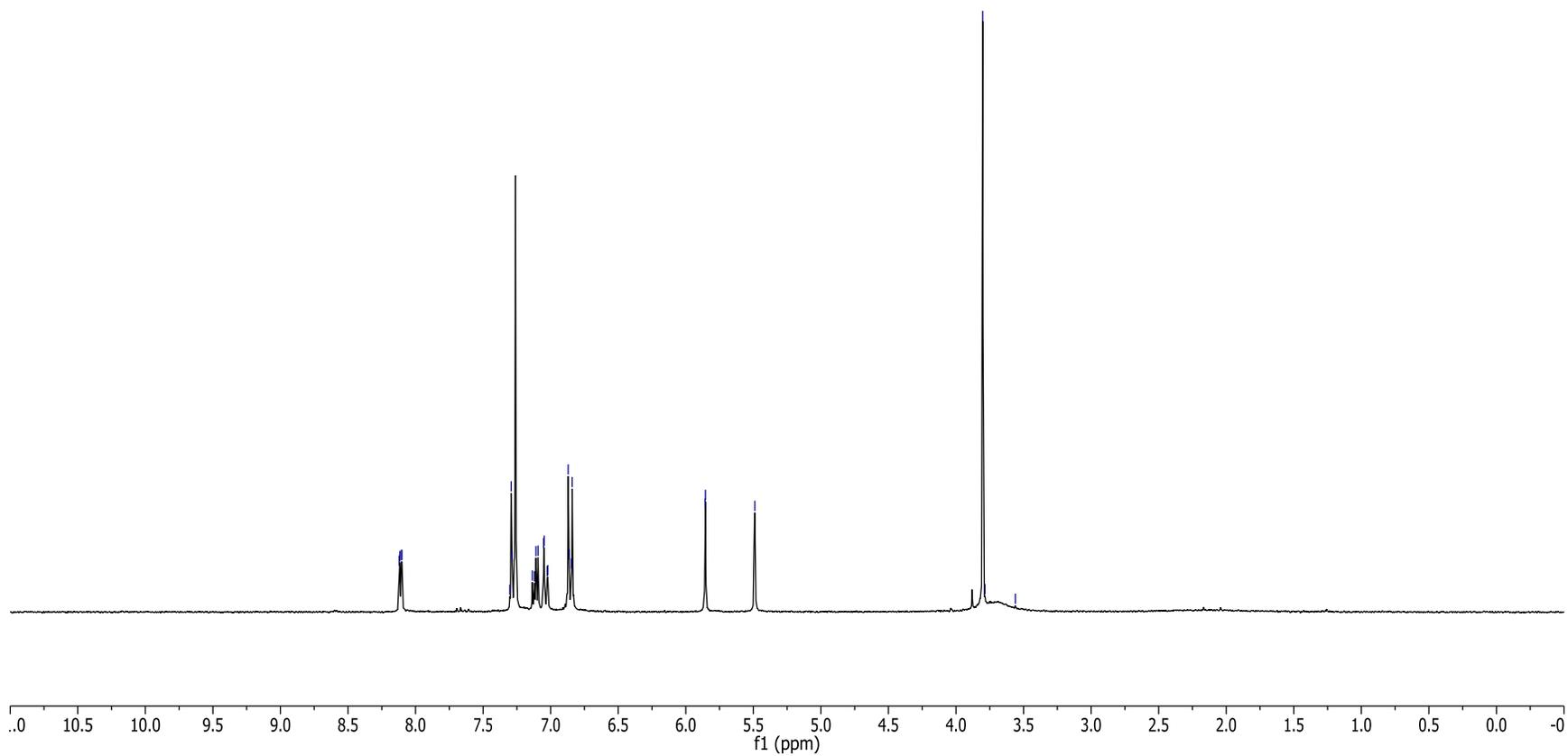


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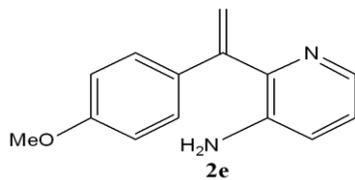


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2012-05-25
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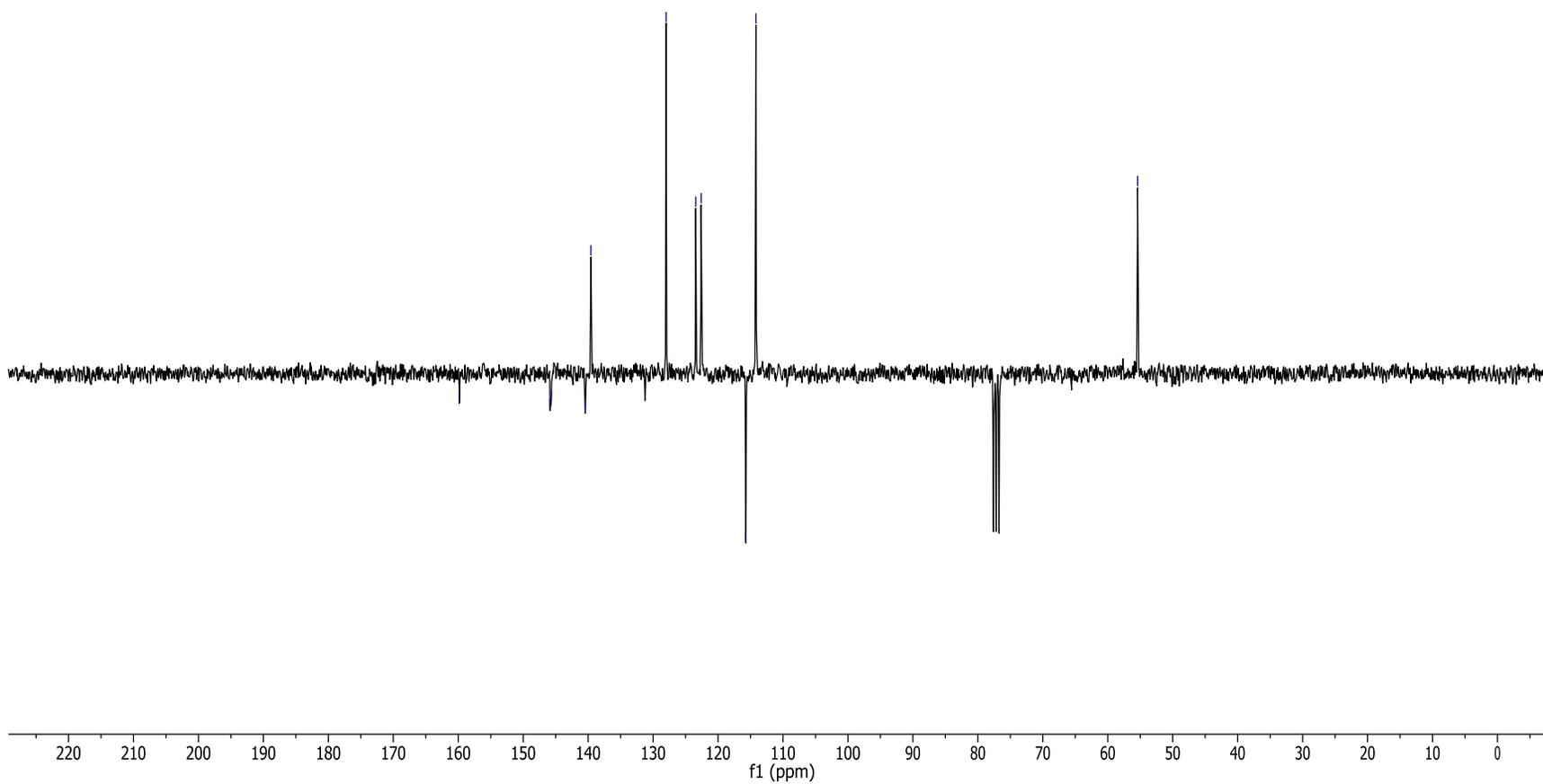
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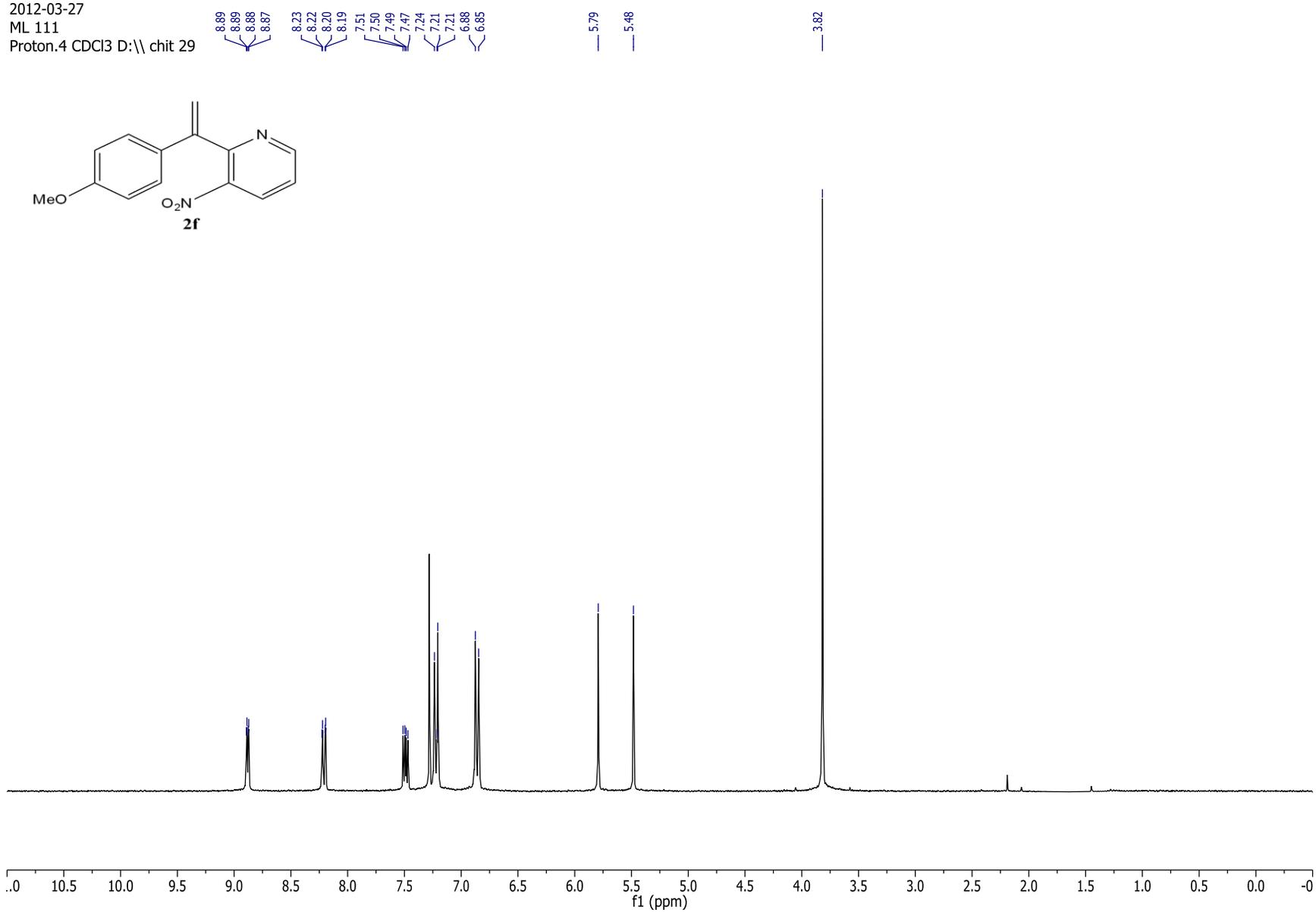
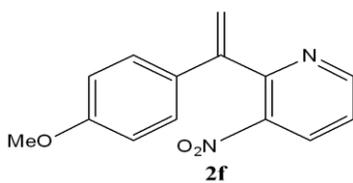
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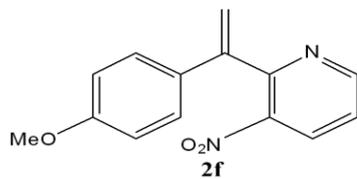
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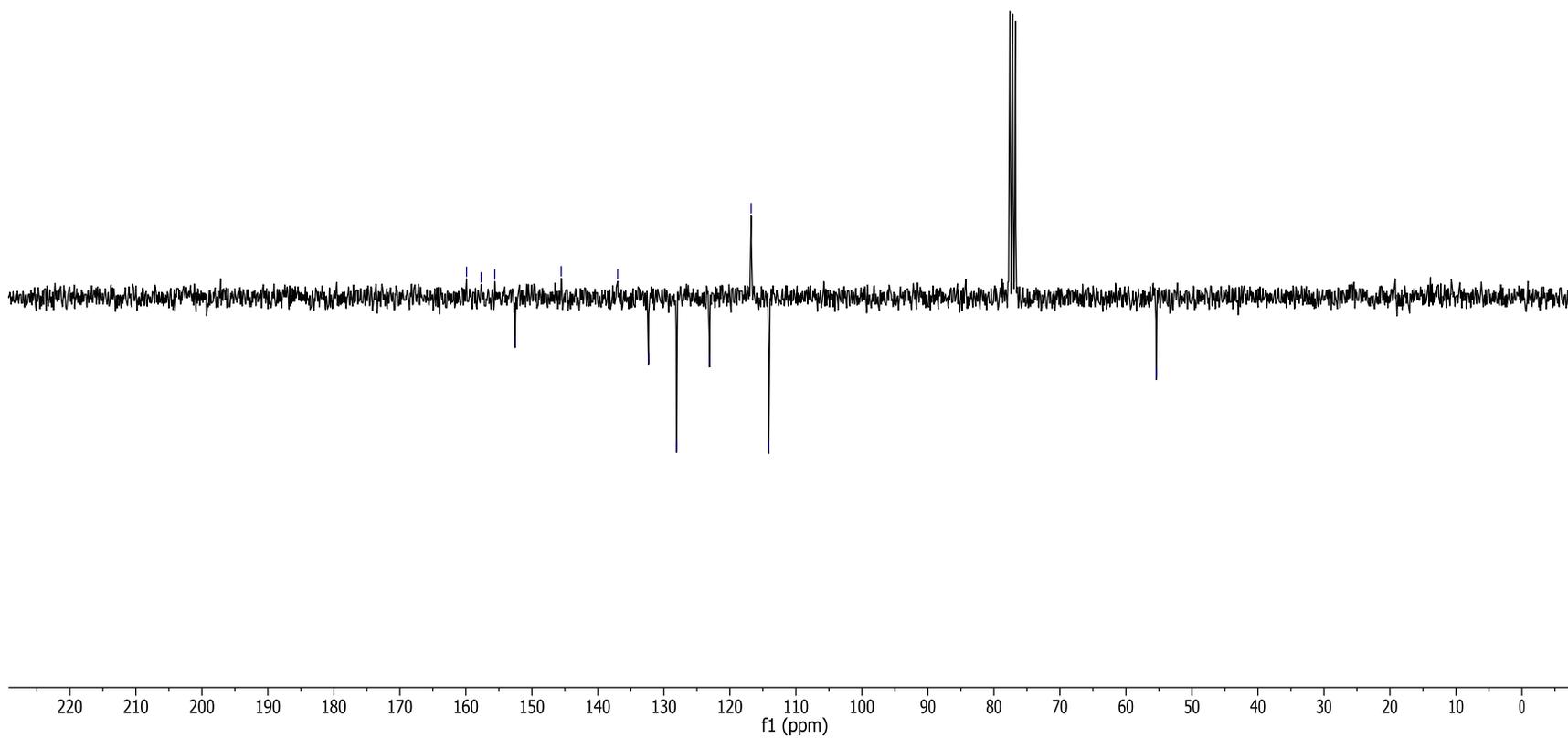
2012-03-27

ML 111

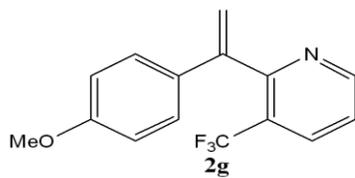
JMOD CDCl3 D:\ chit 53



159.90
157.70
155.62
152.53
145.55
137.00
132.34
128.10
123.09
116.77
114.11
55.41



2012.04.24
ML 128 F1
PROTON CDCl3 v biocis 14



8.83
8.83
8.81
8.80

8.08
8.07
8.04
8.03

7.40
7.21

7.20
7.18

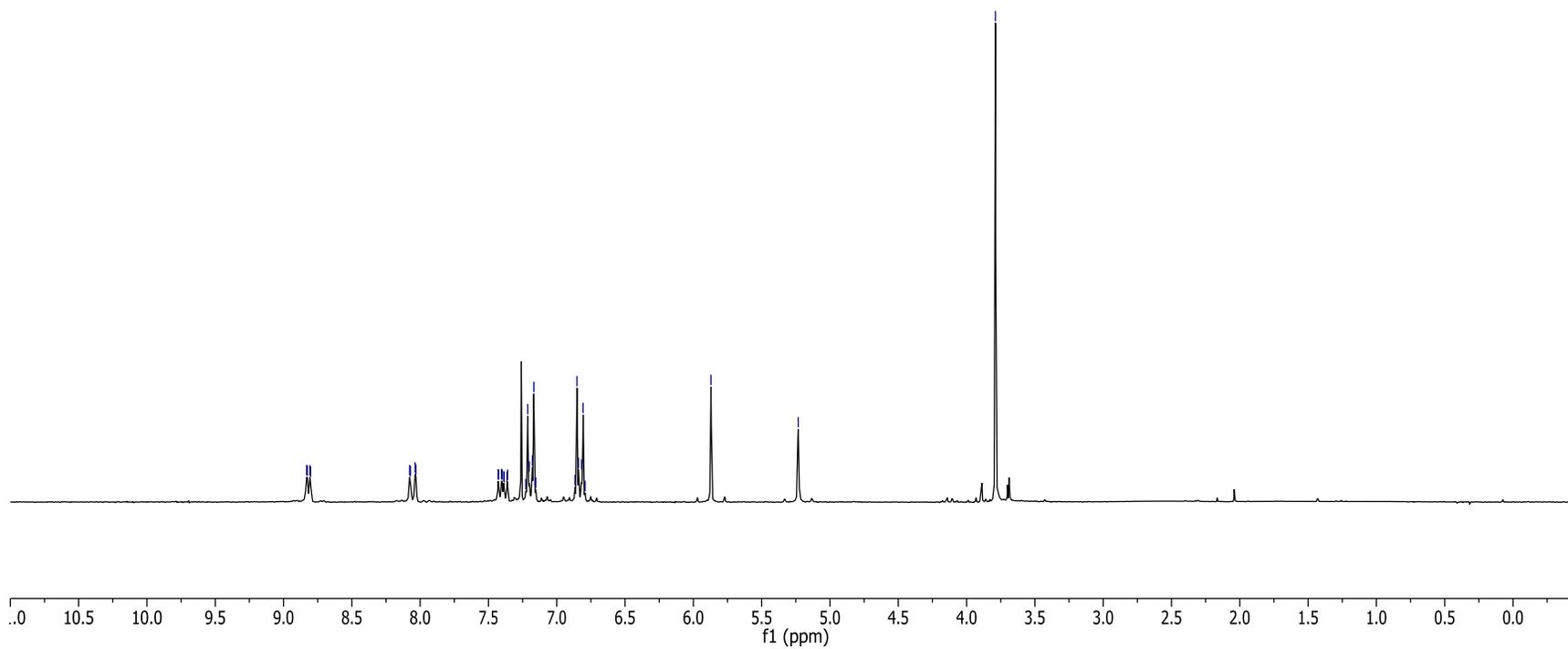
7.17
6.87

6.85
6.84
6.82
6.81
6.79

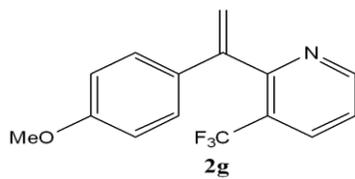
5.87

5.23

3.79

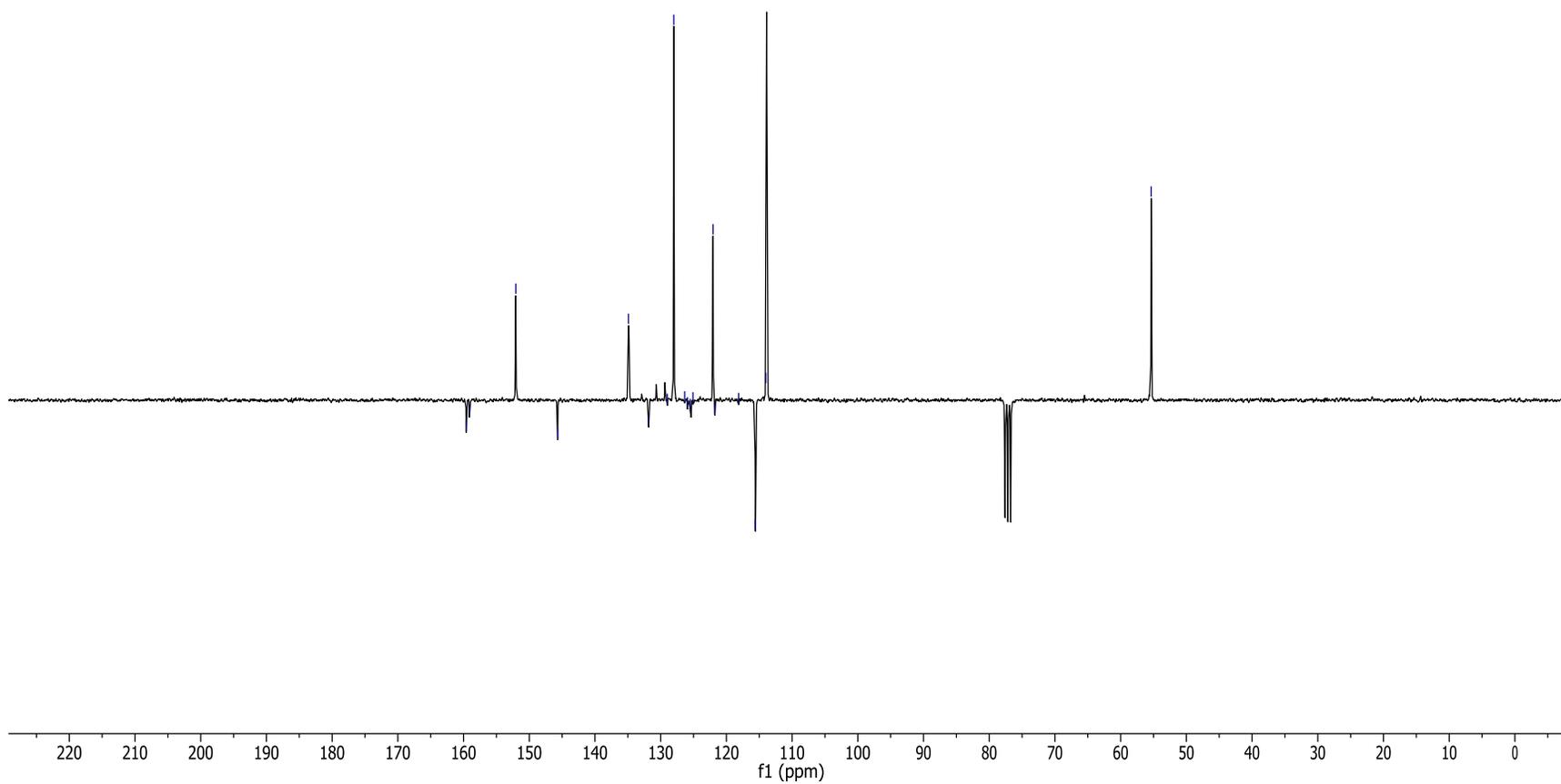


2012-05-25
JA 128
JMOD CDCl3 D:\chit 17

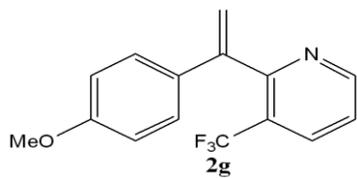


159.59
159.11
152.06
145.68
134.90
131.83
128.99
128.01
126.35
125.93
125.51
125.09
122.03
121.75
118.13
115.58
114.00

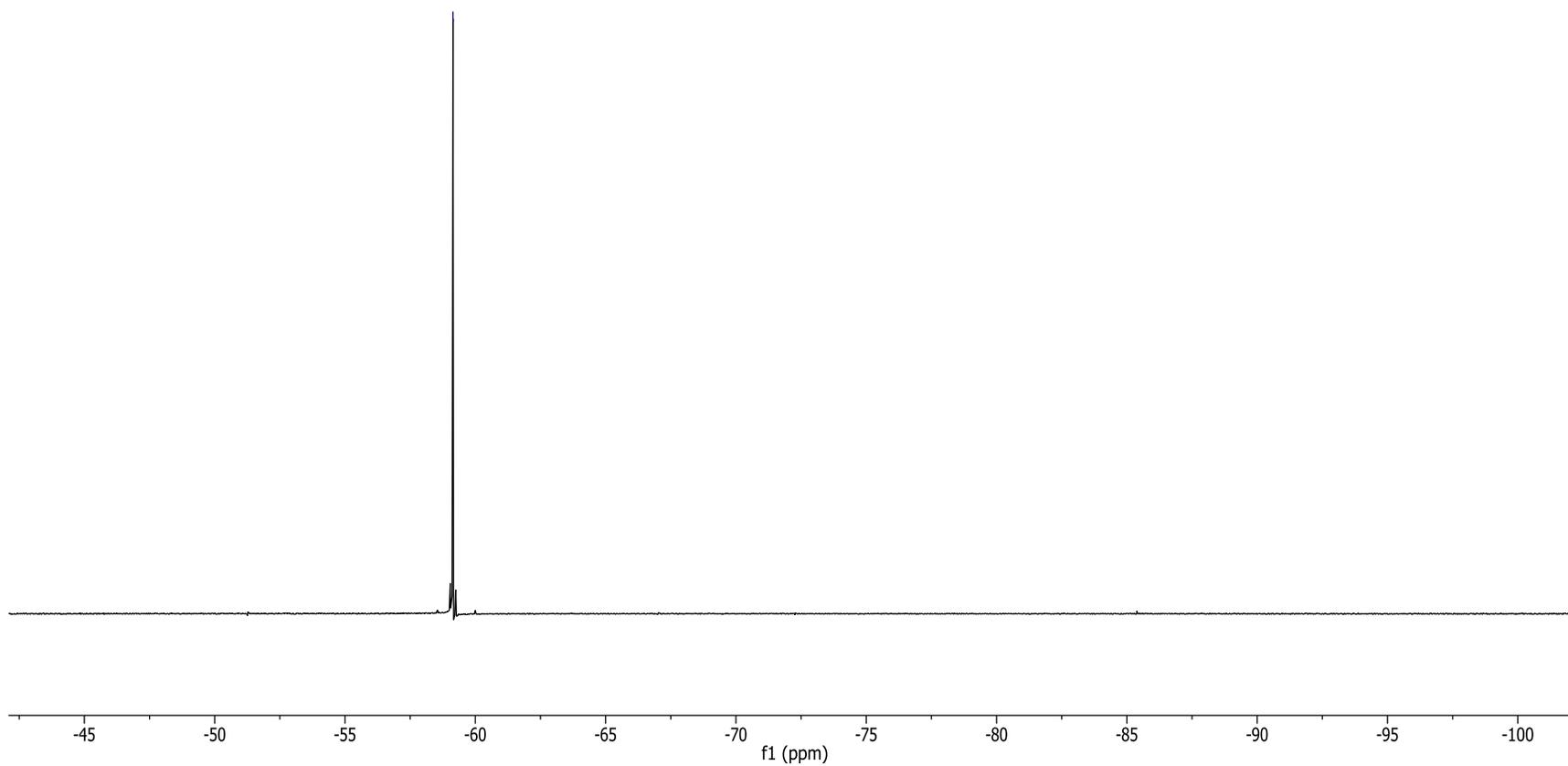
55.36



2012.04.25
ML 128
F19_CF3 CDCl3 v biocis 22

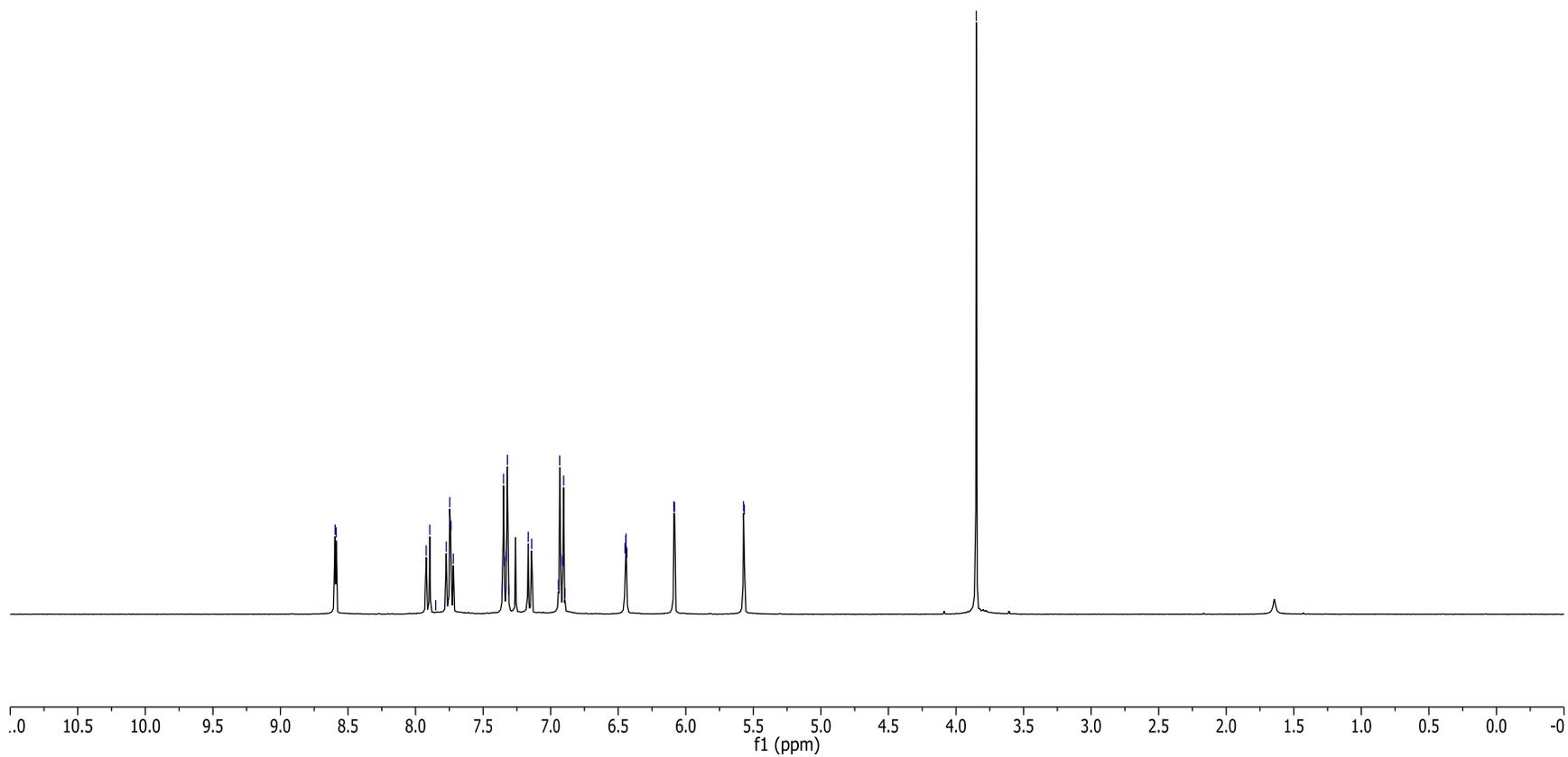
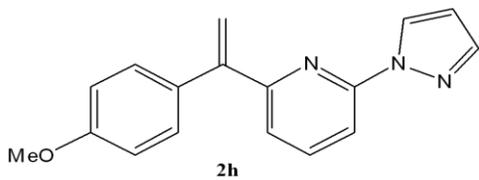


-59.14



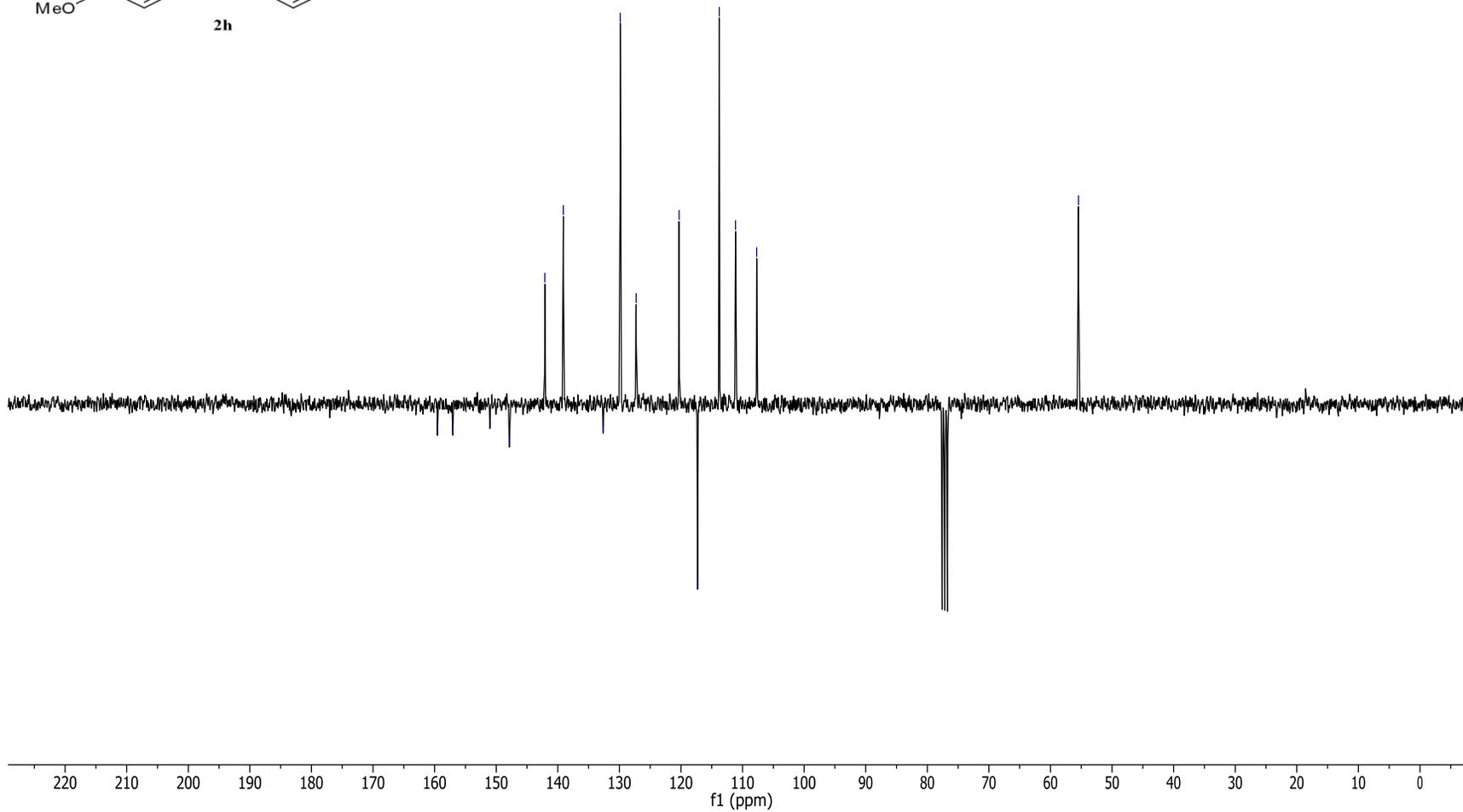
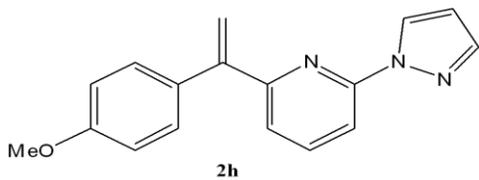
2012-03-30
ML 116
Proton.4 CDCl3 D:\ chit 2

8.60
8.59
7.92
7.89
7.77
7.75
7.74
7.72
7.35
7.32
7.17
7.14
6.93
6.90
6.45
6.44
6.09
6.08
5.57
5.57
3.85



2012-03-30
ML 114
JMOD CDCl3 D:\ chit 2

159.55
157.03
151.04
147.89
142.10
139.09
132.64
129.86
127.28
120.30
117.31
113.78
111.14
107.70
55.46



2012-03-30

ML 114 F1

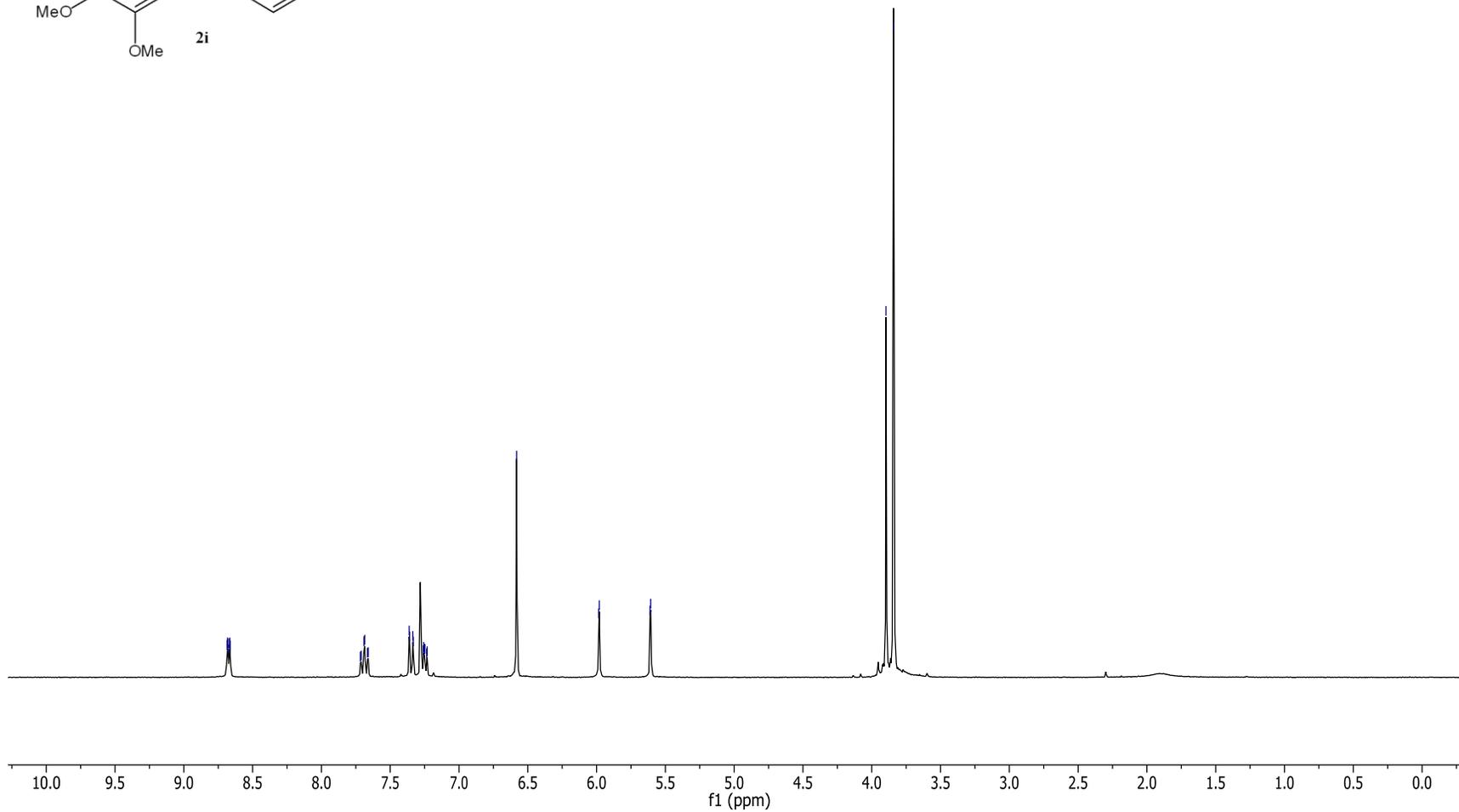
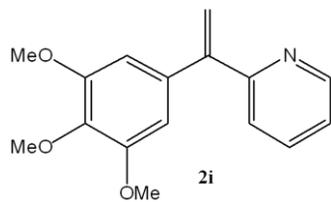
Proton.4 CDCl3 D:\chit ZZ

8.69
8.68
8.68
8.67
8.67
8.66
7.72
7.71
7.69
7.68
7.67
7.66
7.36
7.36
7.34
7.33
7.26
7.25
6.58

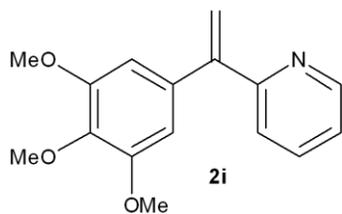
5.99
5.98

5.61
5.61

3.90
3.84



2012-03-30
ML 114
JMOD CDCI3 D:\\ chit 8



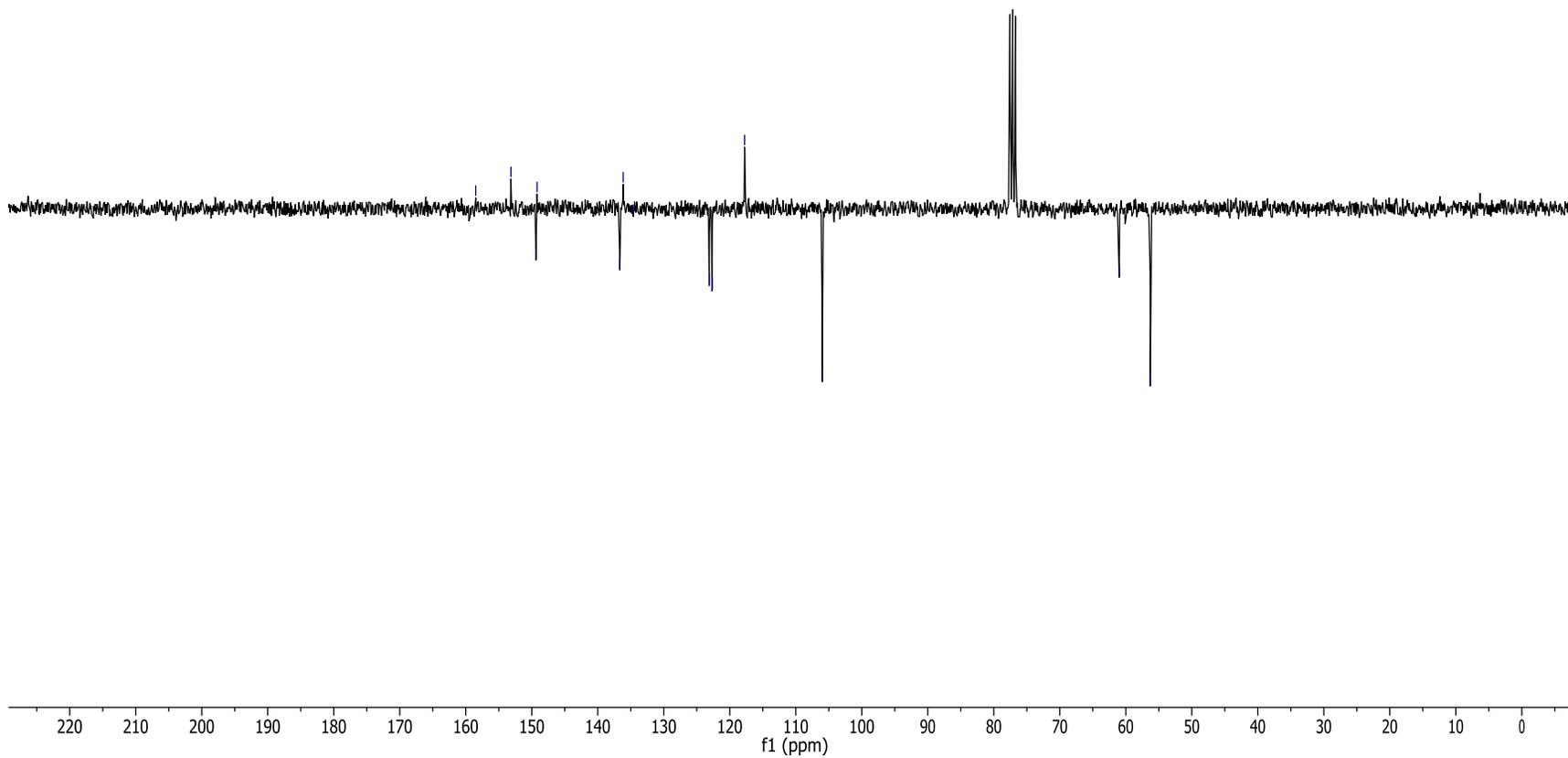
158.49
153.16
149.37
149.20

136.65
136.15
134.64

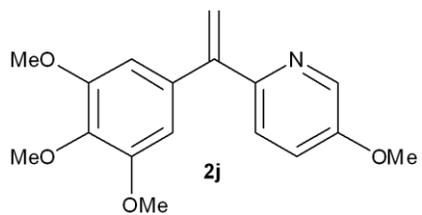
123.12
122.70
117.75

105.99

61.00
56.28



2012-05-03
ML 132
Proton.4 CDCl3 D:\ chit 28

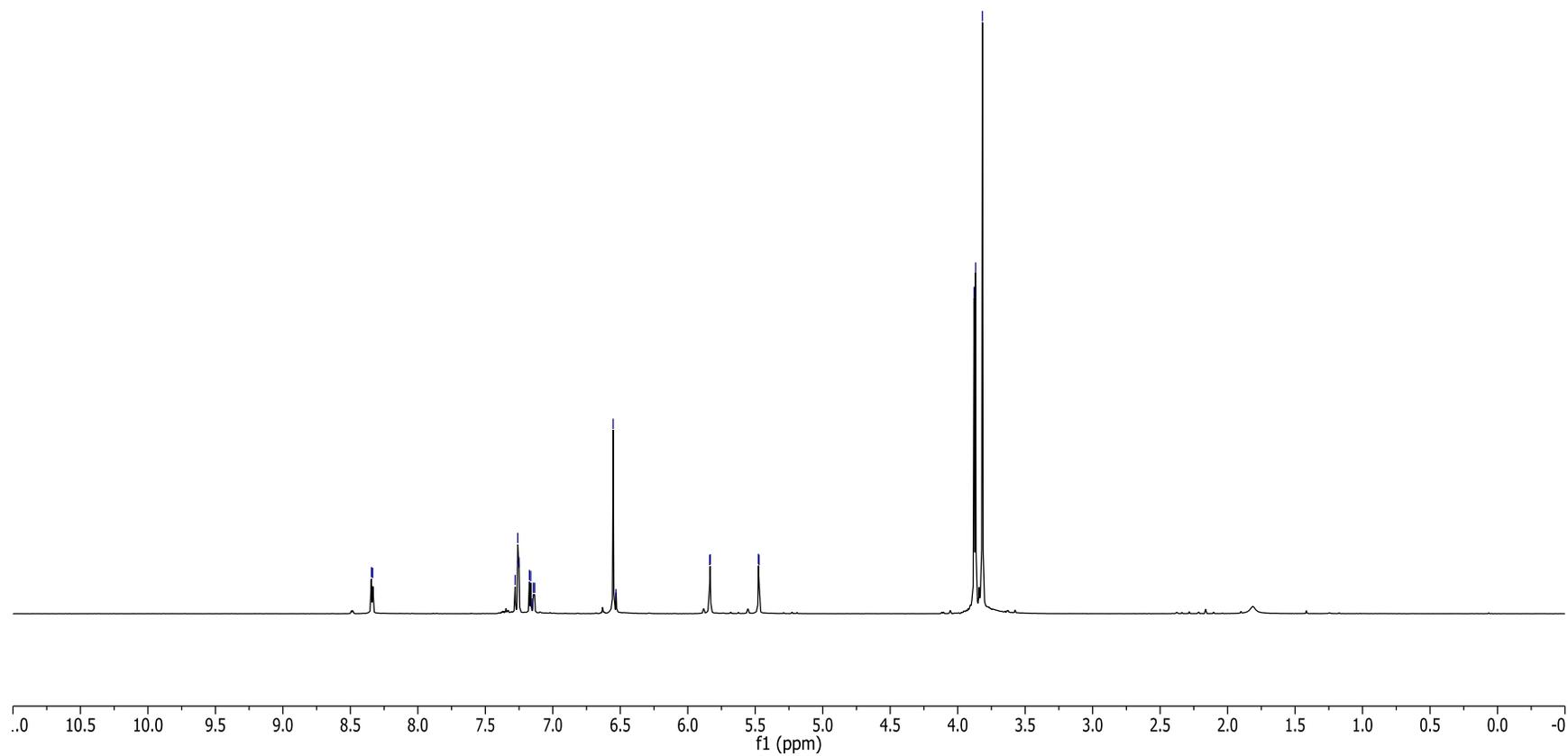


8.34
8.33

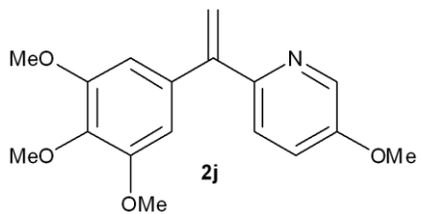
7.28
7.26
7.25
7.17
7.16
7.15
7.14
7.13
6.55
6.53

5.84
5.83
5.48
5.47

3.88
3.87
3.82



2012-05-03
ML 132
JMOD CDCl3 D:\chit 28



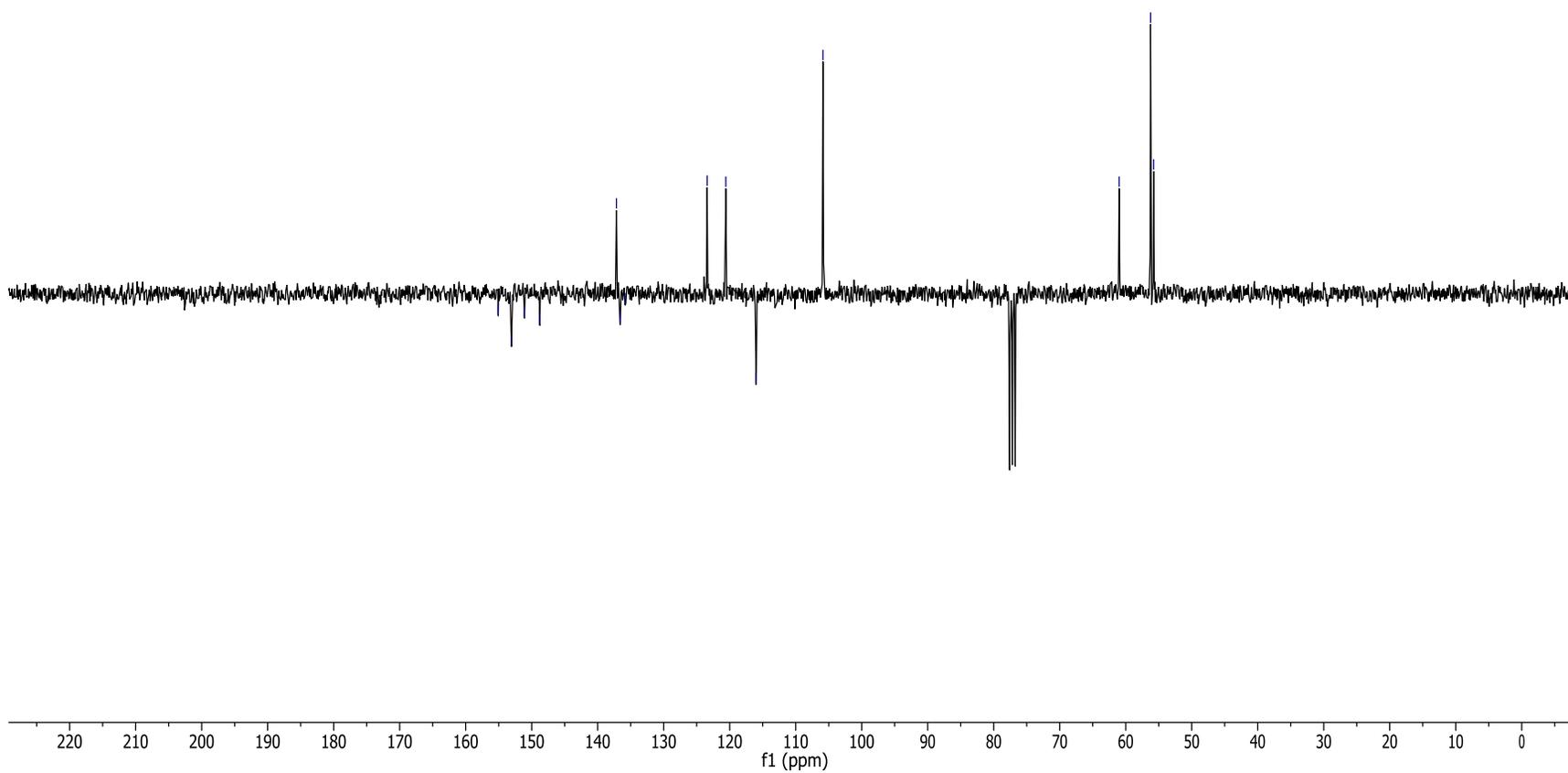
155.07
153.07
151.09
148.77

137.16
136.60
135.85

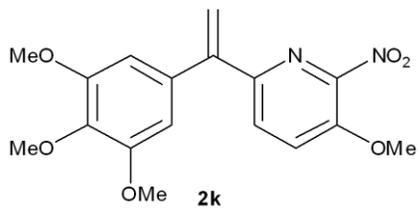
123.42
120.58
115.98

105.87

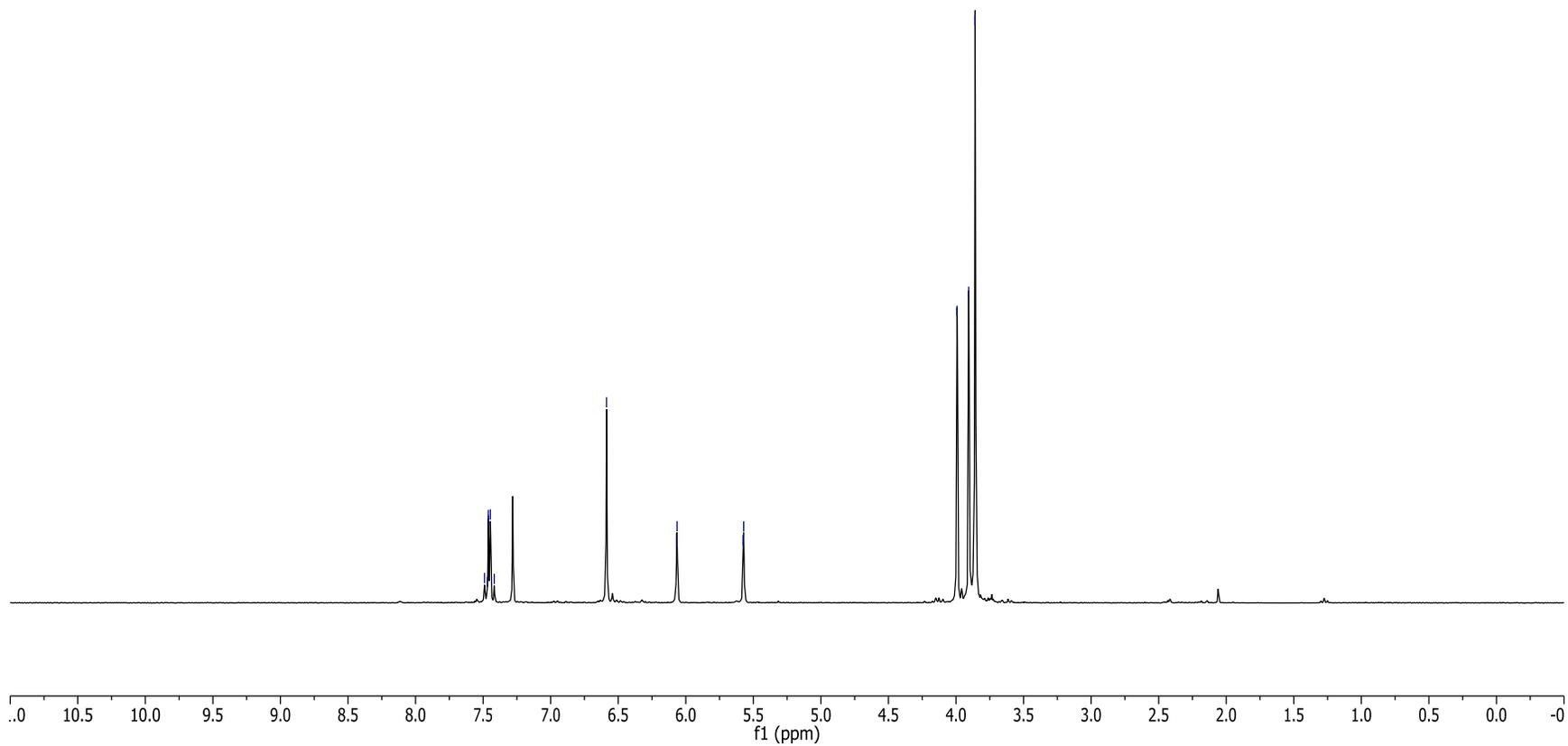
61.00
56.24
55.78



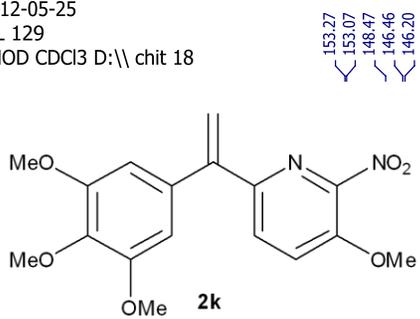
2012-04-26
ML 129
Proton.4 CDCl3 D:\\ chit 50



7.49
7.47
7.46
7.45
7.42
6.59
6.07
6.06
5.58
5.57
3.99
3.91
3.86



2012-05-25
ML 129
JMOD CDCl3 D:\\ chit 18



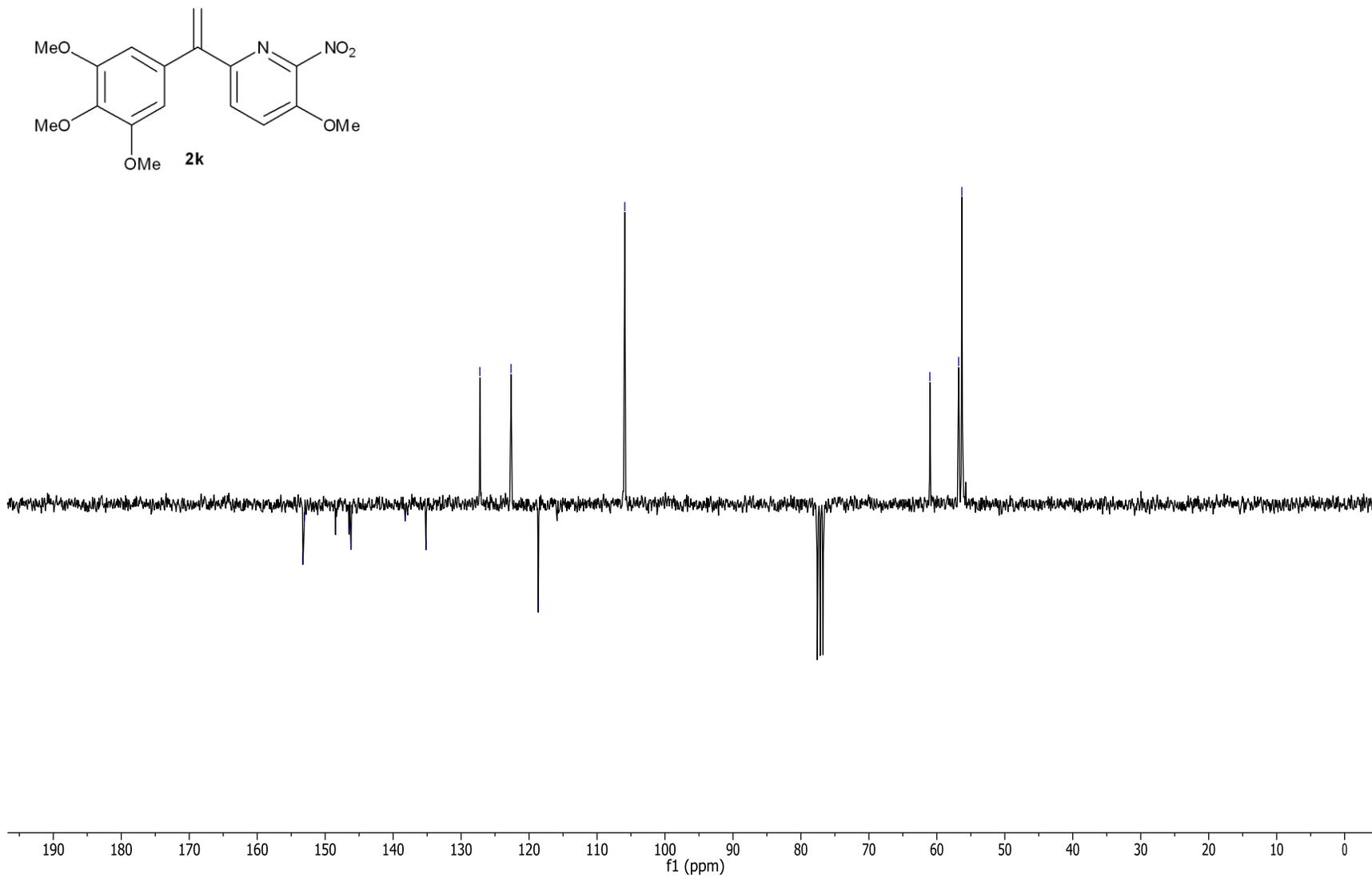
153.27
153.07
148.47
146.46
146.20

138.23
135.18

127.23
122.64
118.64

105.92

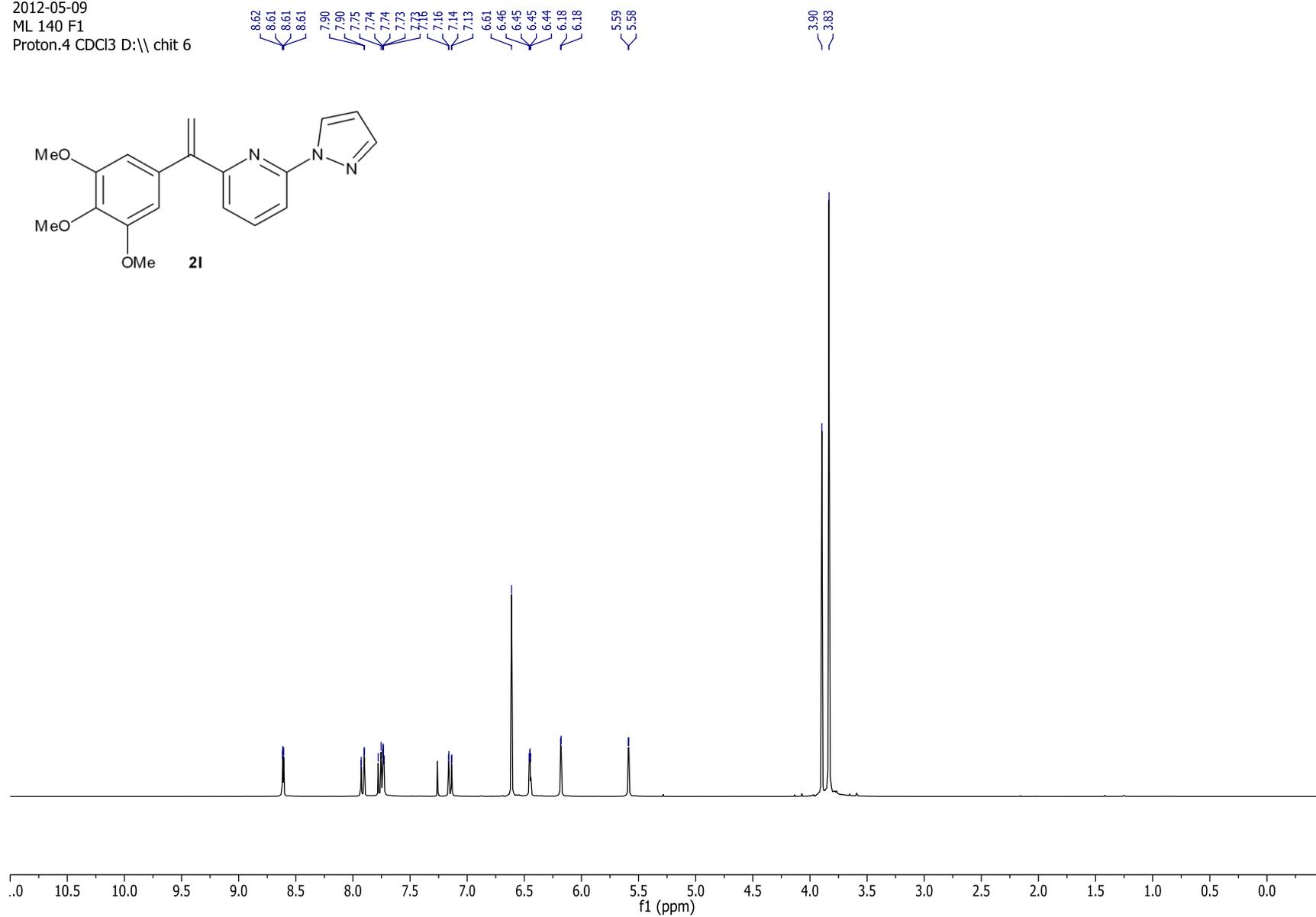
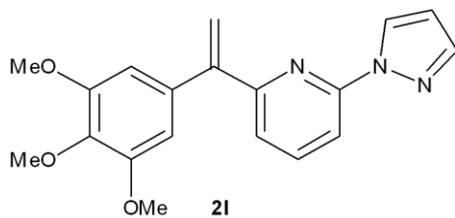
61.02
56.79
56.32



2012-05-09
ML 140 F1
Proton.4 CDCl3 D:\ chit 6

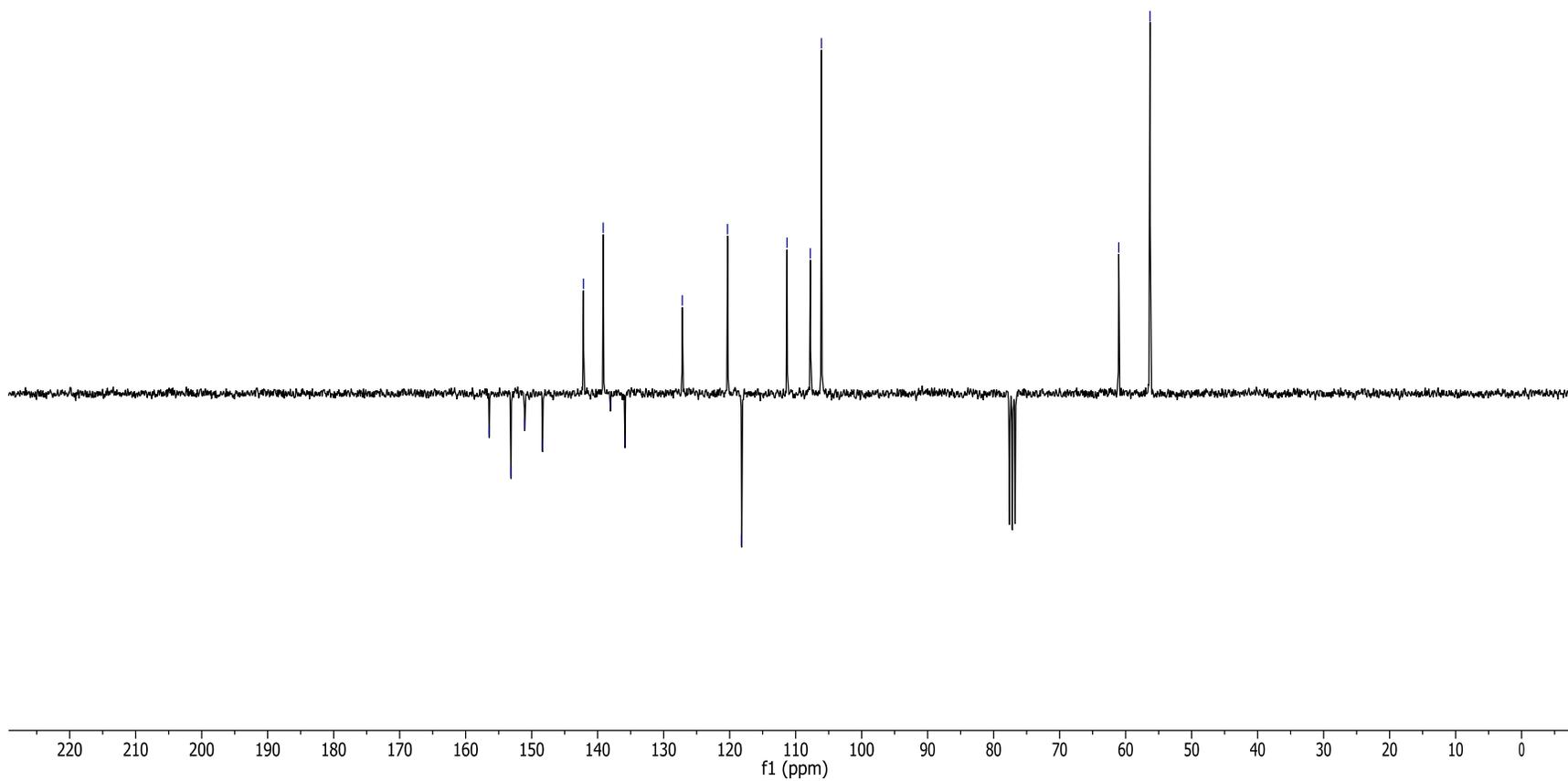
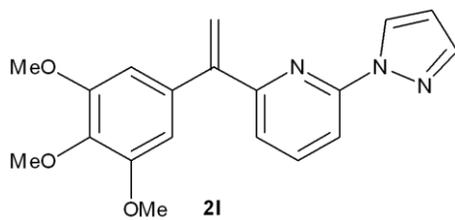
8.62
8.61
8.61
7.90
7.90
7.75
7.74
7.74
7.73
7.73
7.16
7.16
7.14
7.13
6.61
6.46
6.45
6.45
6.44
6.18
6.18
5.59
5.58

3.90
3.83



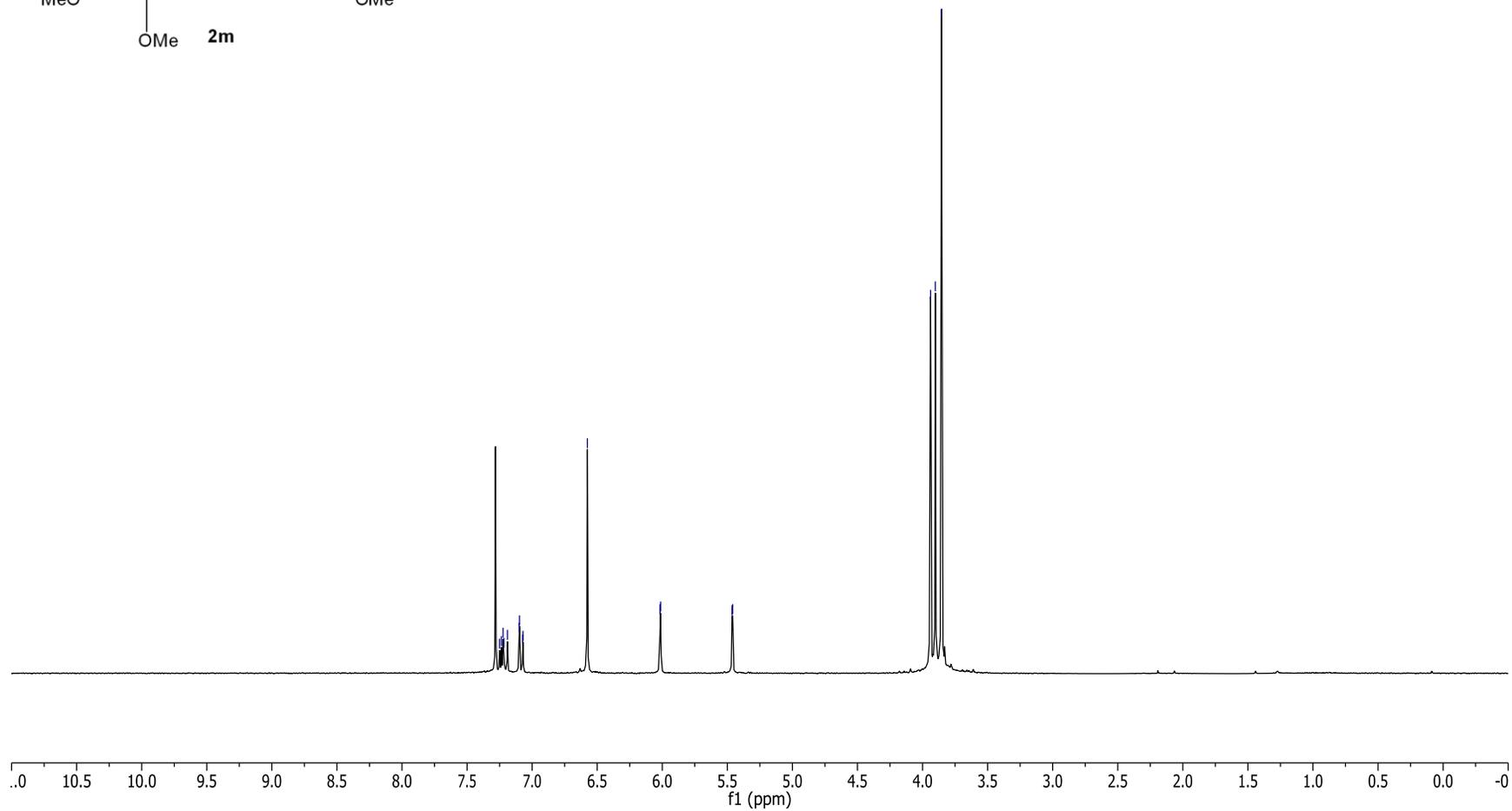
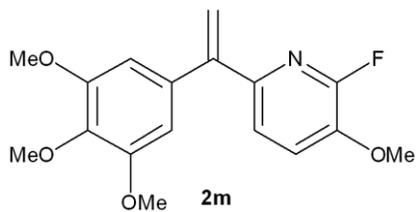
2012-05-10
ML 140
JMOD CDCl3 D:\\ chit 21

156.43
153.13
151.07
148.34
142.15
139.16
138.06
135.85
127.18
120.32
118.17
111.31
107.78
106.10
61.04
56.31



2012-05-04
ML 137 F2
Proton.4 CDCl3 D:\ chit 2

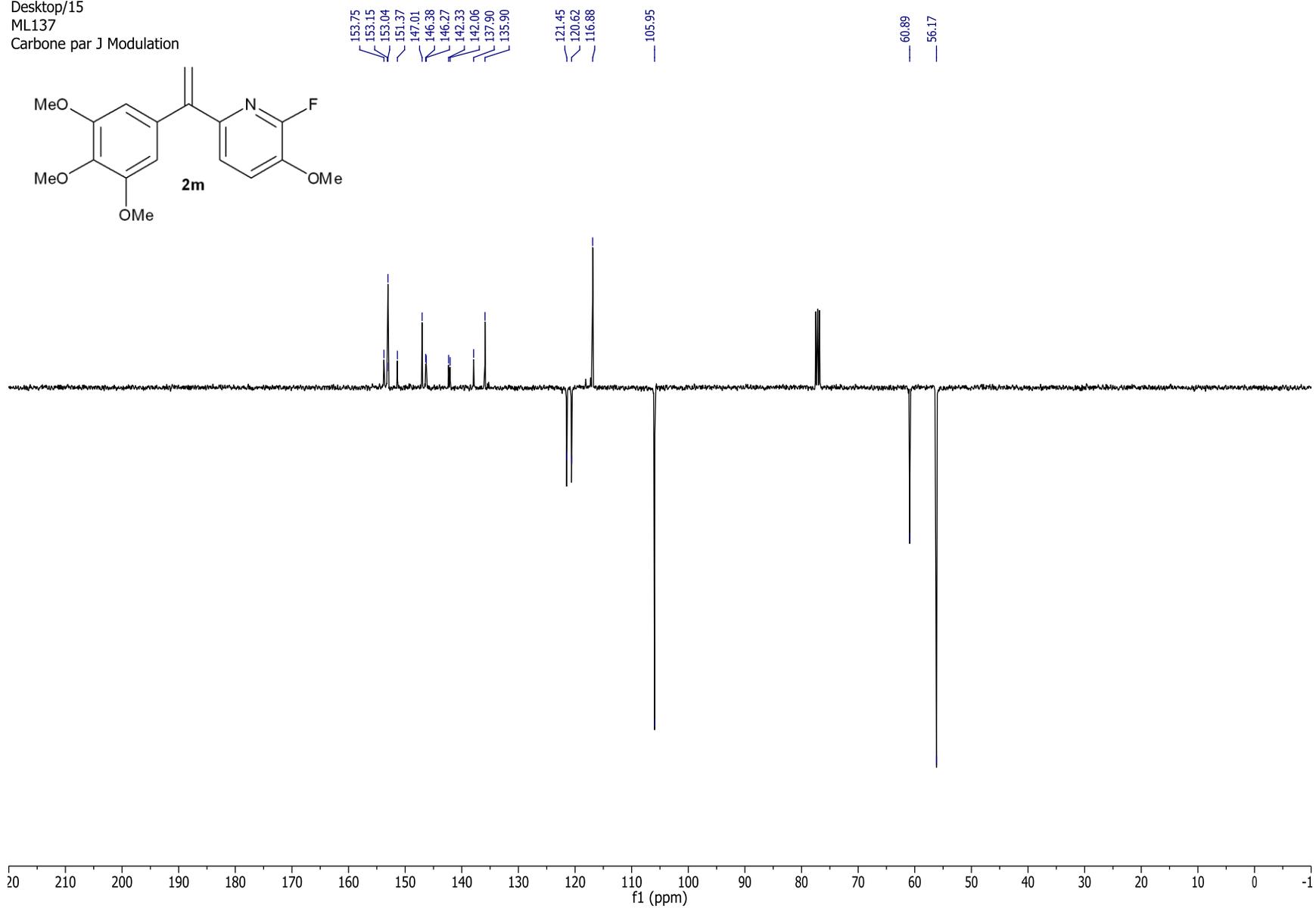
7.25
7.24
7.22
7.22
7.19
7.10
7.10
7.07
7.07
6.58
6.02
6.01
5.46
5.46
3.94
3.90
3.85



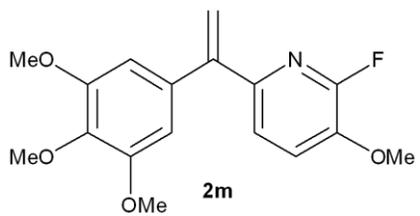
Desktop/15

ML137

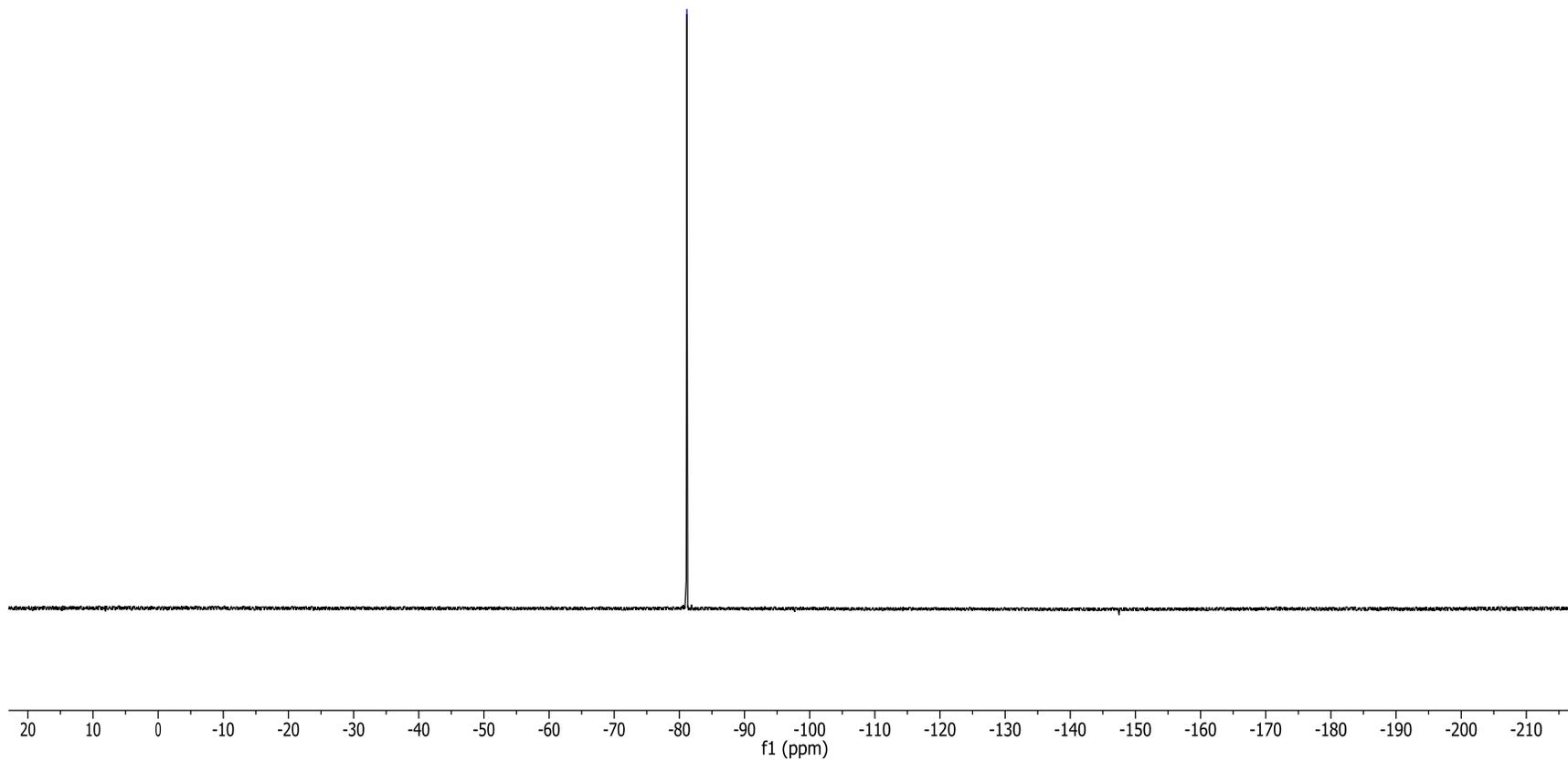
Carbone par J Modulation



2012.05.04
ml 137
F19CPD CDCl3 v biocis 10

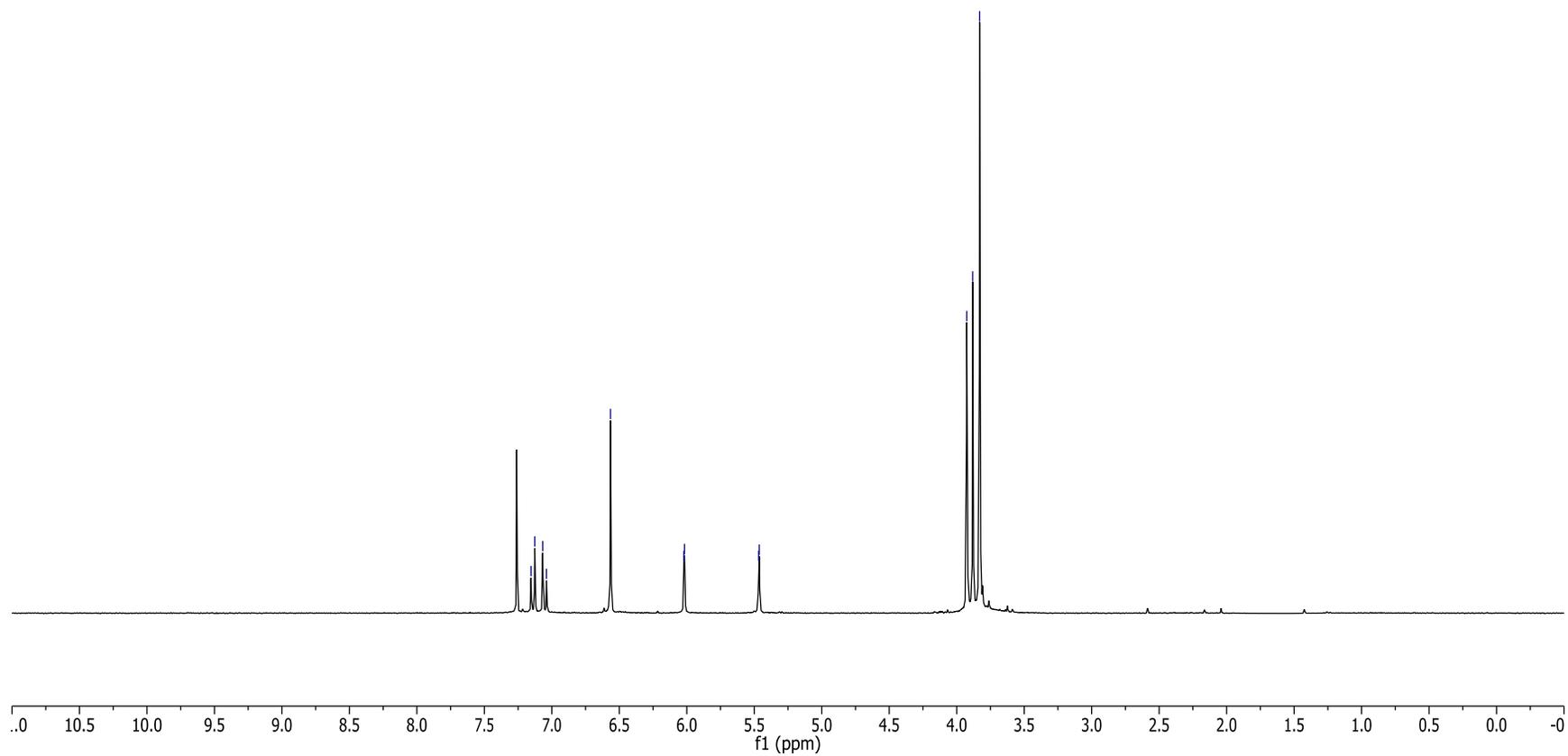
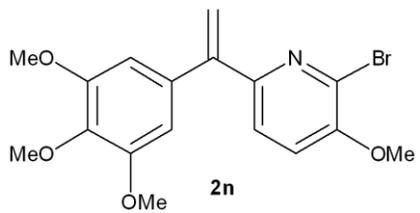


-81.16



2012-04-30
ML 134 F1
Proton.4 CDCl3 D:\ chit 13

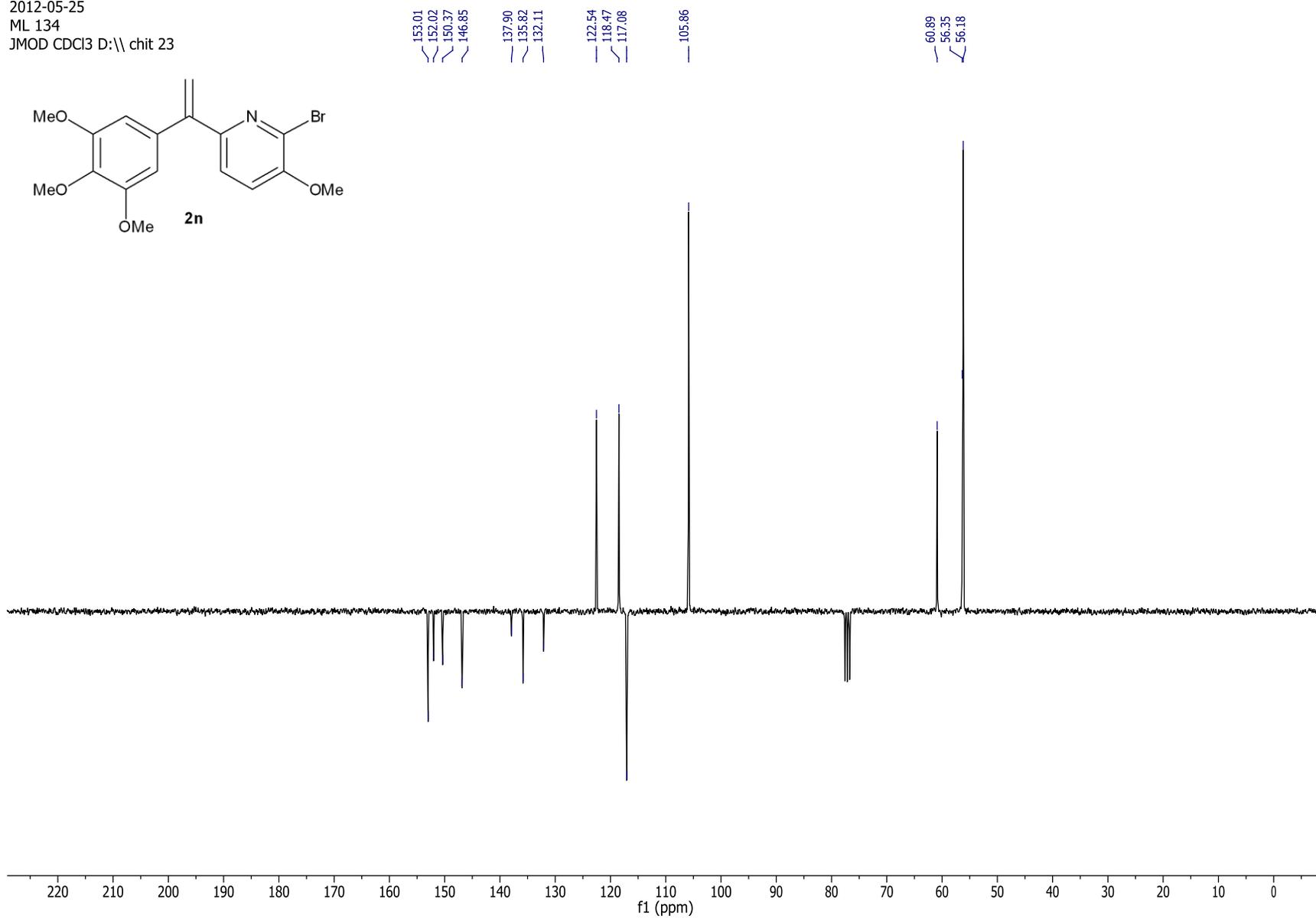
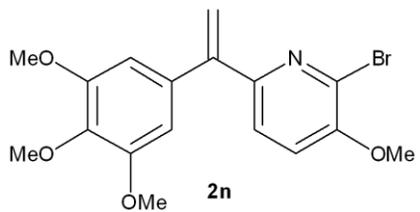
7.15
7.13
7.07
7.04
6.57
6.02
6.02
5.47
5.46
3.93
3.88
3.83



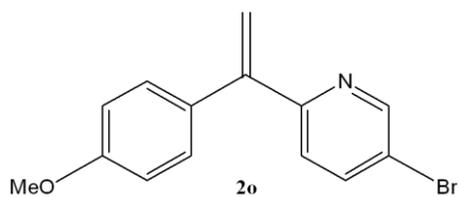
2012-05-25

ML 134

JMOD CDCl3 D:\chit 23



2012-03-21
ML 100
Proton.4 CDCl3 D:\ chit 45



8.70
8.69

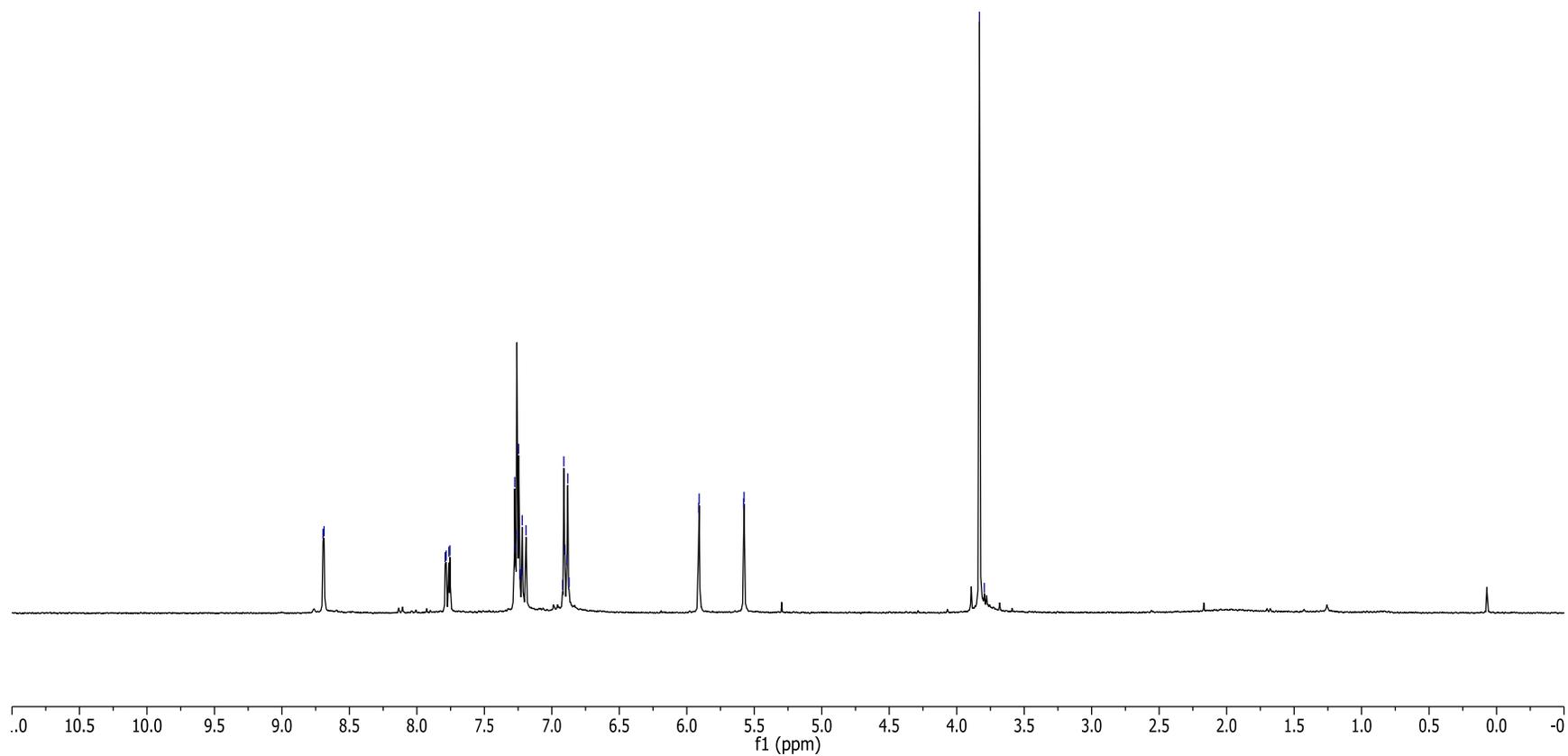
7.79
7.78
7.76
7.75

7.28
7.25
7.22

6.91
6.90
6.89
6.88
6.87

5.91
5.91
5.58
5.58

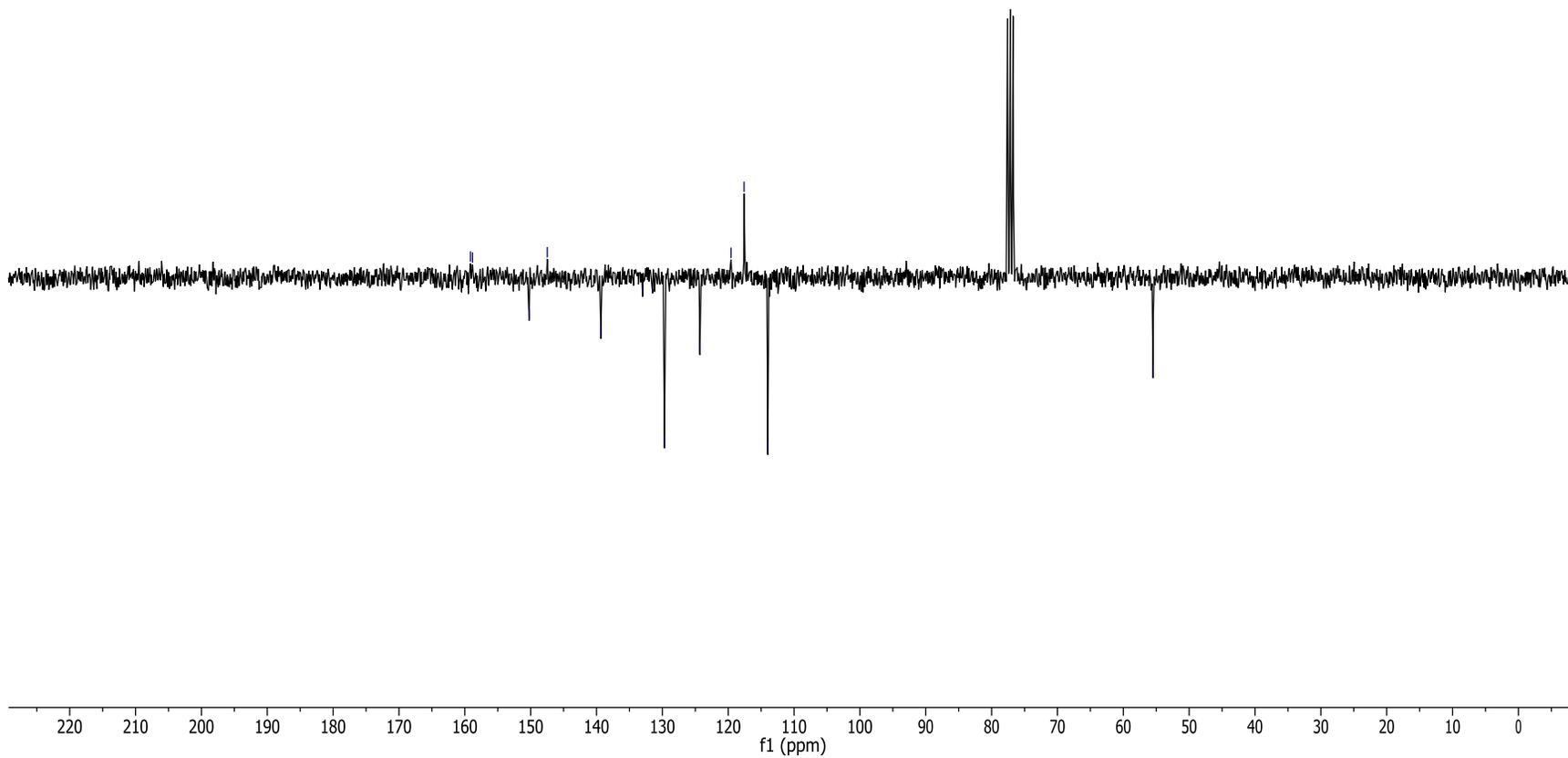
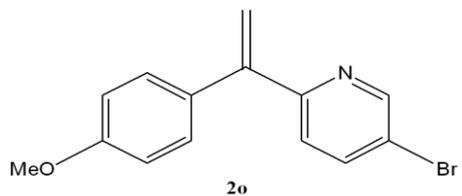
3.83
3.80



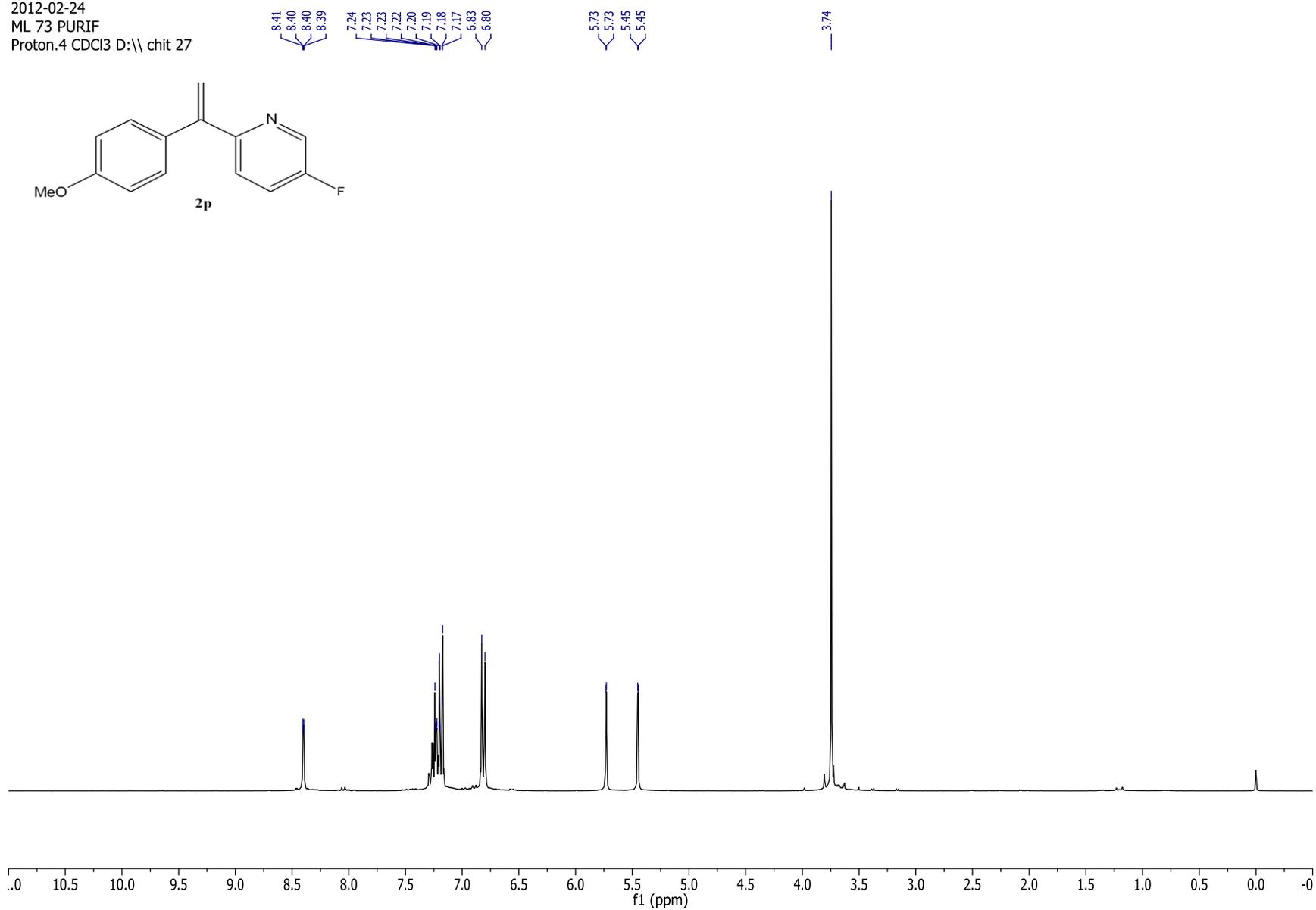
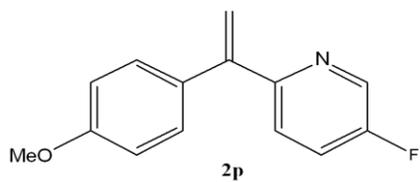
2012-03-21
ML 100
JMOD CDCl3 D:\ chit 44

159.14
158.83
150.25
147.47
139.33
133.01
129.68
124.31
119.57
117.59
113.99

55.48



2012-02-24
ML 73 PURIF
Proton.4 CDCl3 D:\\ chit 27



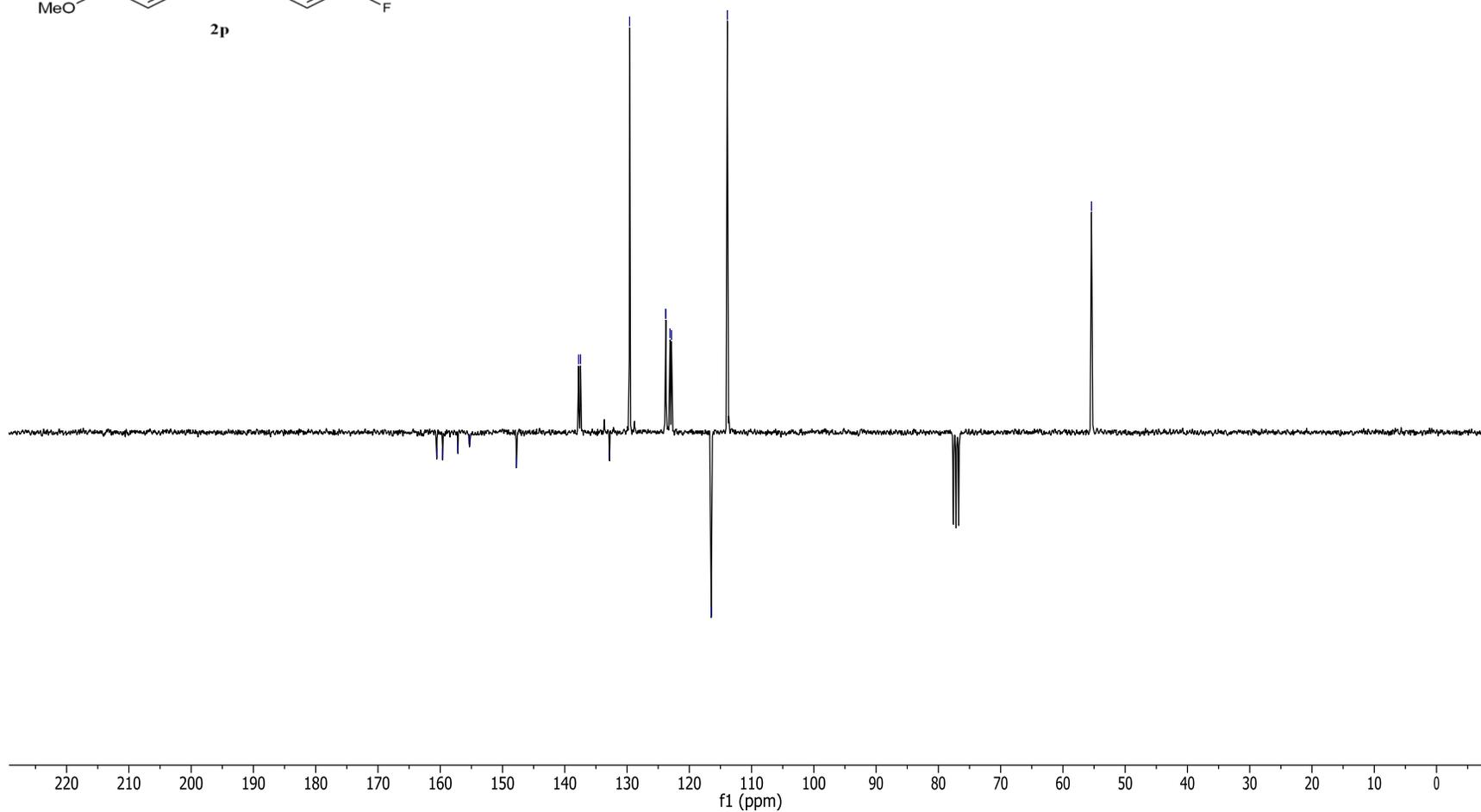
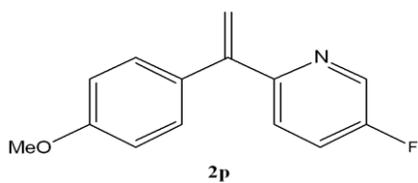
2012-02-27
ML73
JMOD CDCl3 D:\ chit 7

160.55
159.61
157.15
155.36
155.31
147.77

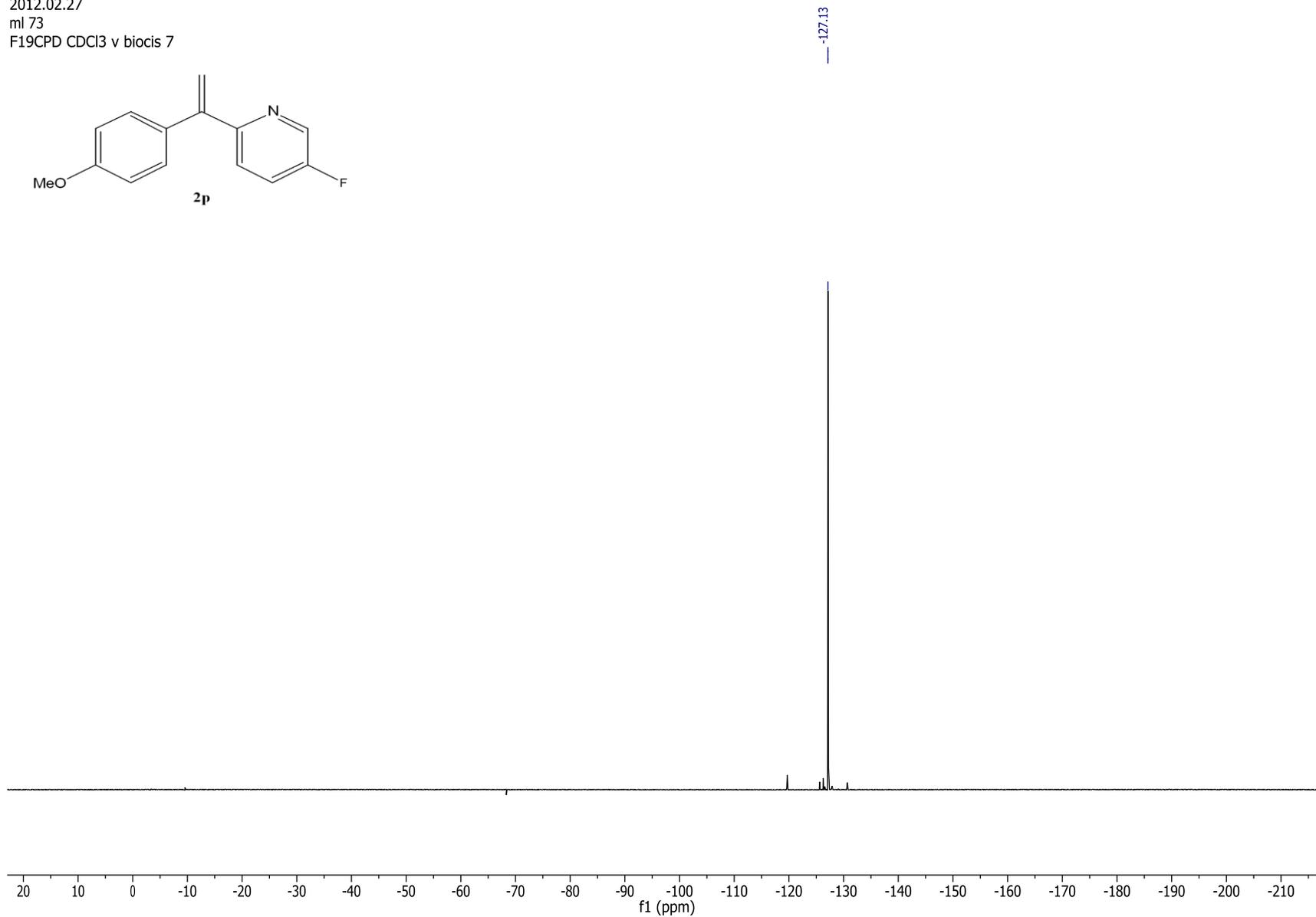
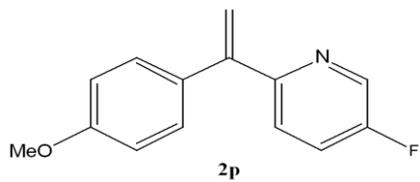
137.78
137.46
132.80
129.59

123.83
123.77
116.49
113.88

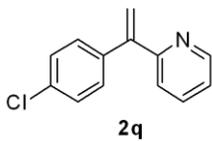
55.42



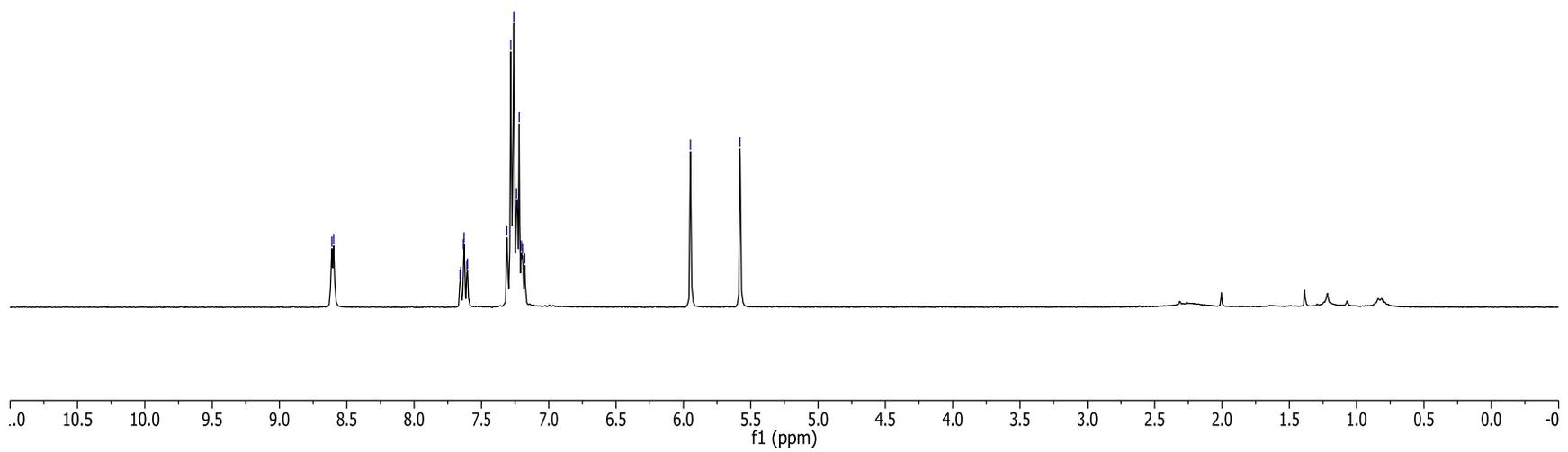
2012.02.27
ml 73
F19CPD CDCl3 v biosis 7

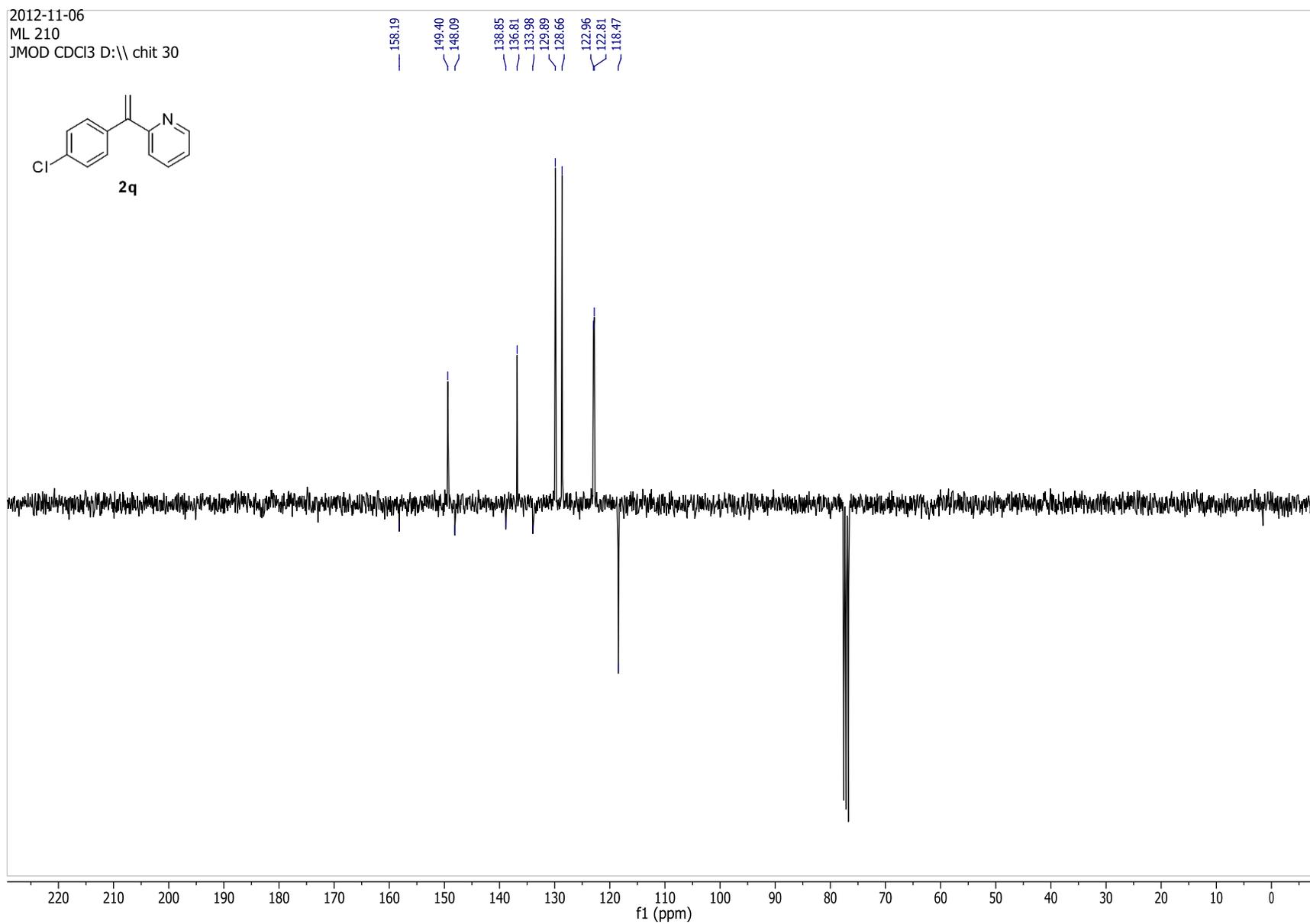


2012-11-06
ML 210
Proton.4 CDCl3 D:\ chit 30

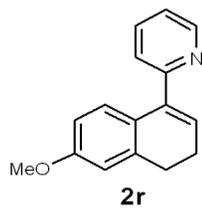


8.61
8.60
7.66
7.65
7.63
7.63
7.61
7.60
7.31
7.28
7.26
7.24
7.23
7.22
7.20
7.20
7.18
5.95
5.58





2012-11-05
ML 204
Proton.4 CDCl3 D:\\ chit 49

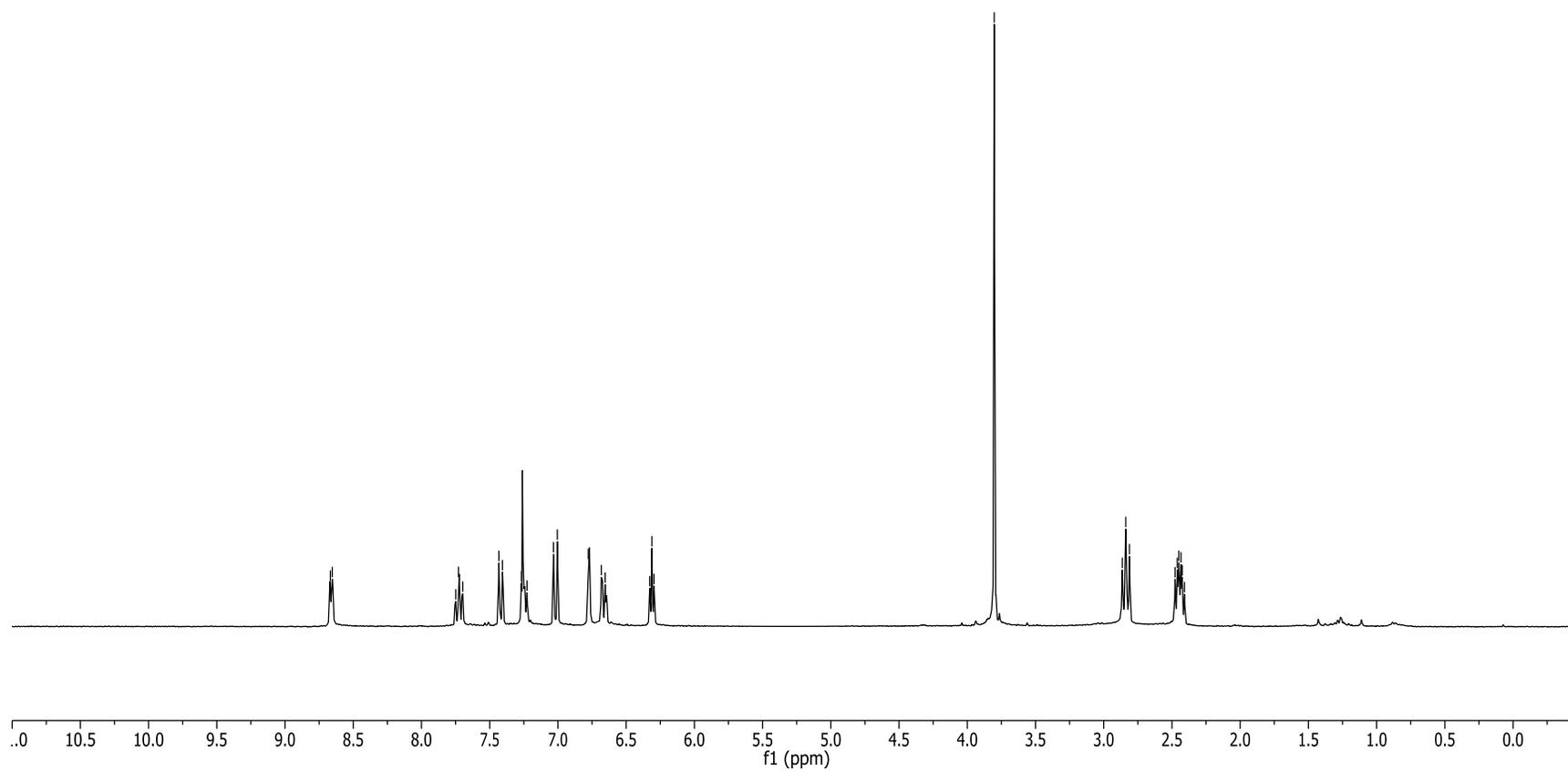


8.67
8.65

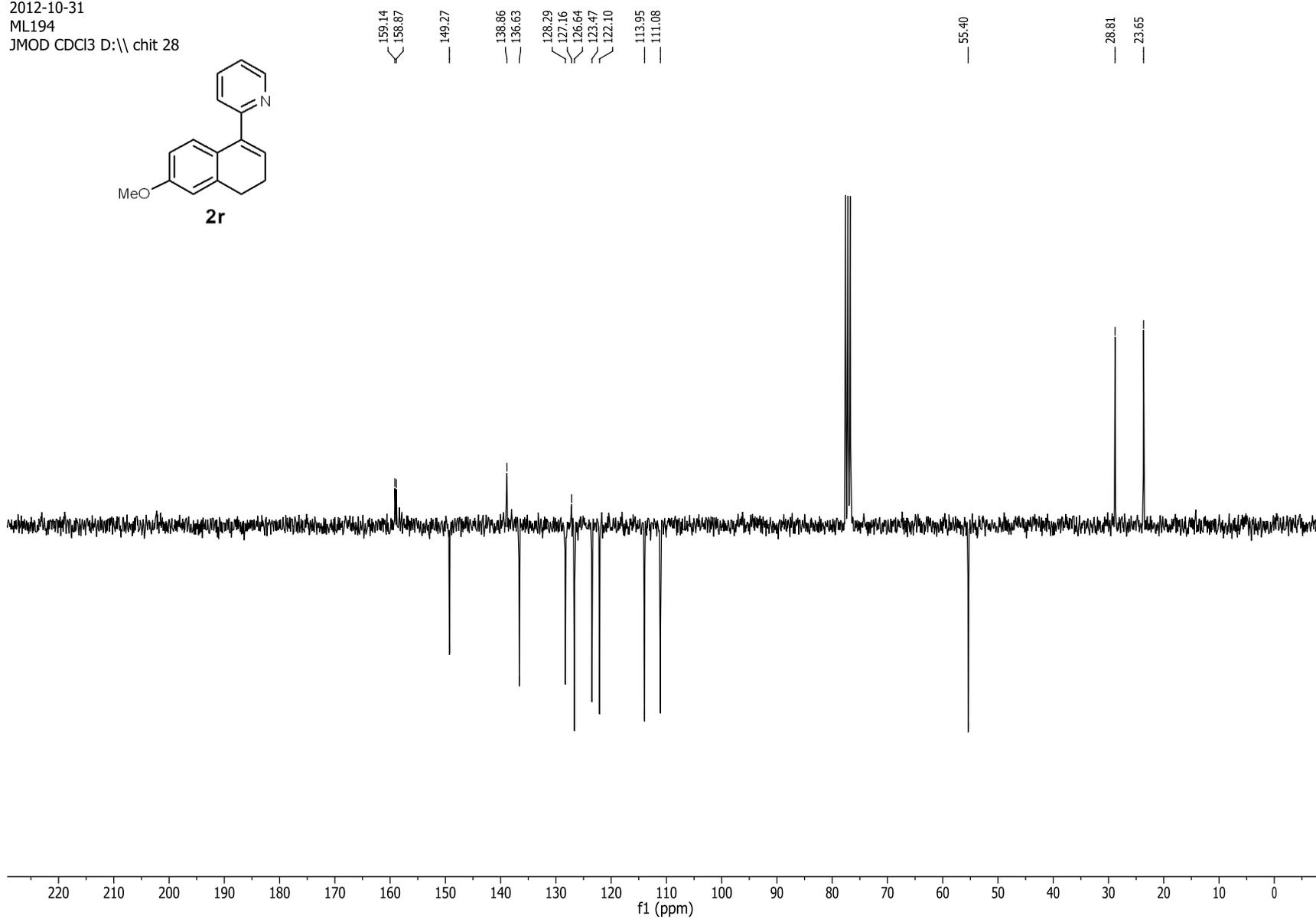
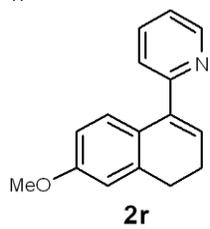
7.75
7.73
7.70
7.43
7.41
7.27
7.03
7.00
6.78
6.68
6.55
6.33
6.31
6.30

3.80

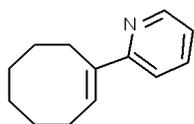
2.86
2.84
2.81
2.48
2.46
2.45
2.43
2.42
2.41



2012-10-31
ML194
JMOD CDCl3 D:\\ chit 28



2012-11-06
ML 205
Proton.4 CDCl3 D:\\ chit 29



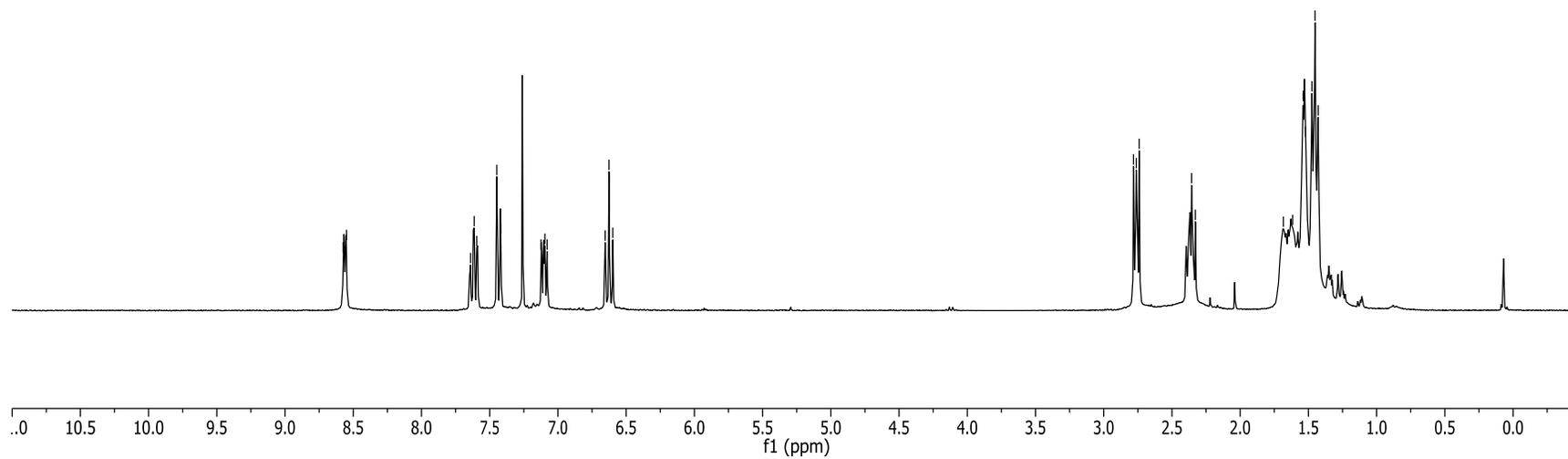
2s

8.57
8.55

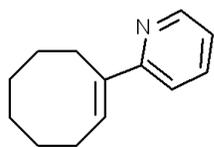
7.64
7.61
7.59
7.45
7.42
7.42
7.12
7.10
7.08
6.65
6.63
6.60

2.78
2.76
2.74
2.36
2.33

1.68
1.62
1.54
1.52
1.47
1.45
1.43



2012-11-07
ML 205
JMOD CDCl3 D:\\ chit 36



2s

159.24

148.79

139.84

136.61

131.99

121.37

119.86

29.95

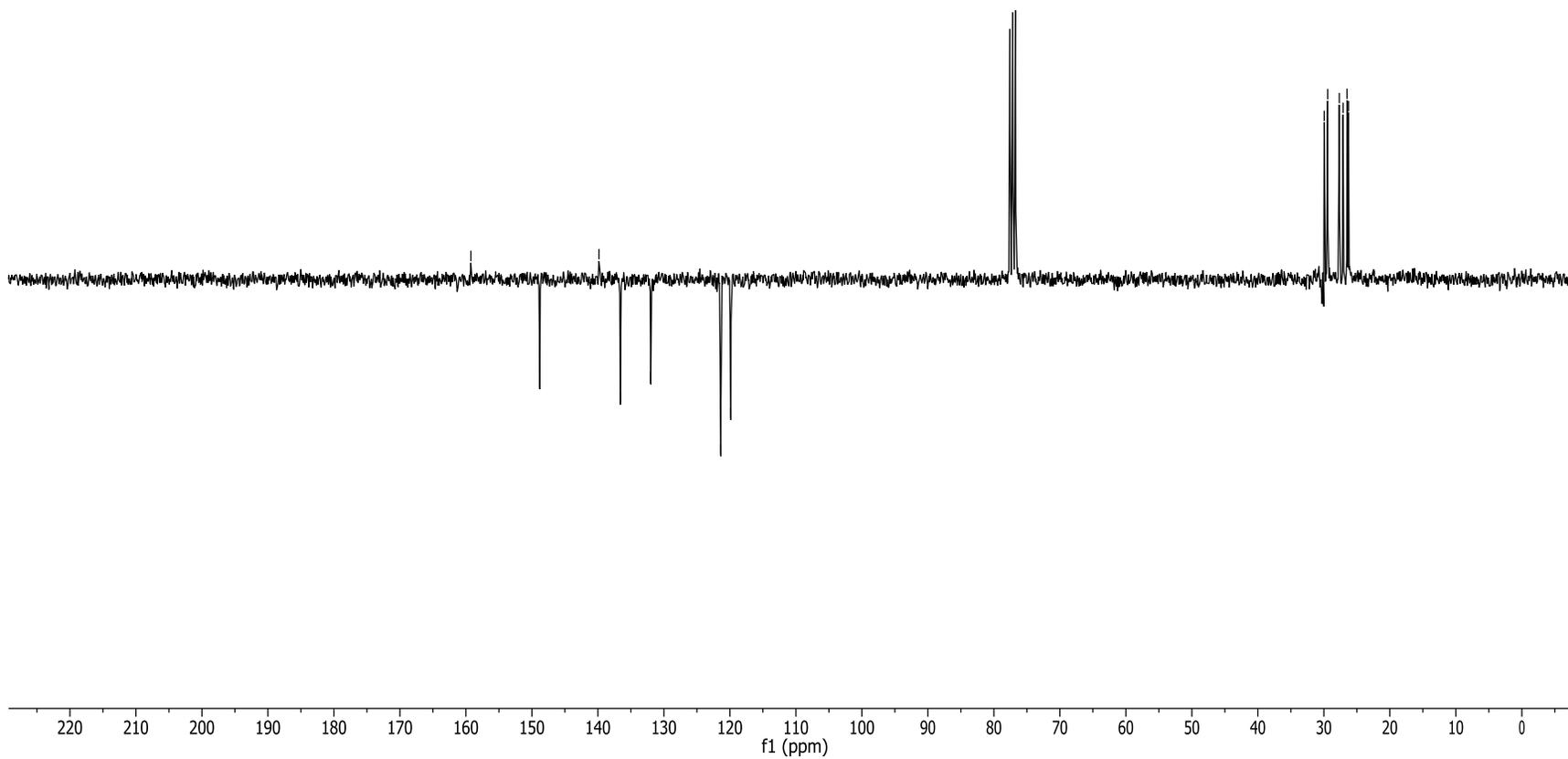
29.43

27.66

27.09

26.49

26.27

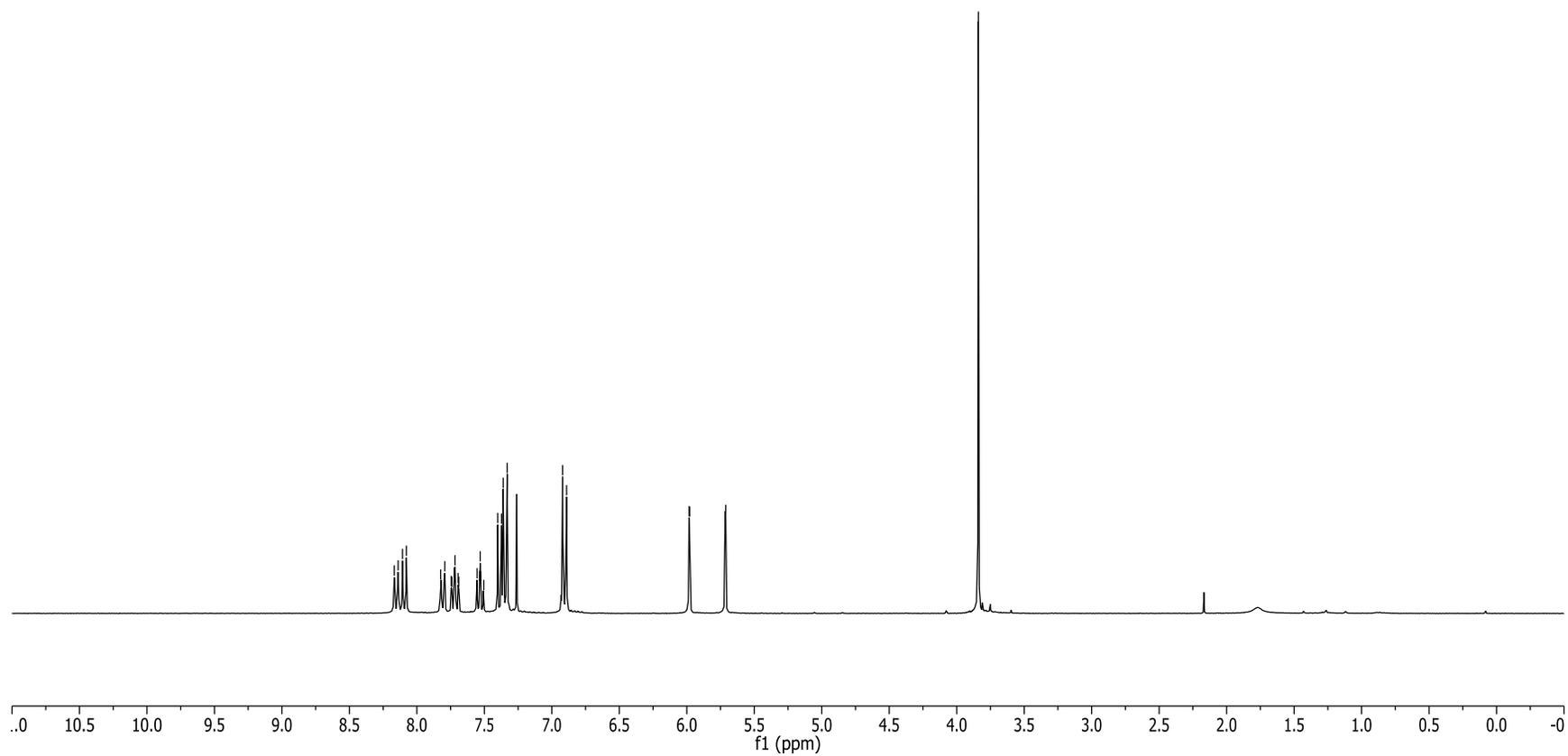
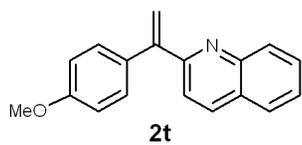


2012-11-02
ML 201 F1
Proton.4 CDCl3 D:\

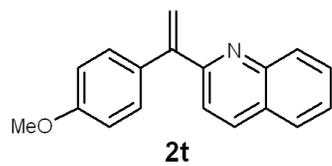
8.17
8.14
8.11
8.08
7.82
7.79
7.75
7.74
7.72
7.72
7.69
7.69
7.55
7.53
7.53
7.50
7.40
7.37
7.36
7.33
6.92
6.89

5.98
5.98
5.71

3.84



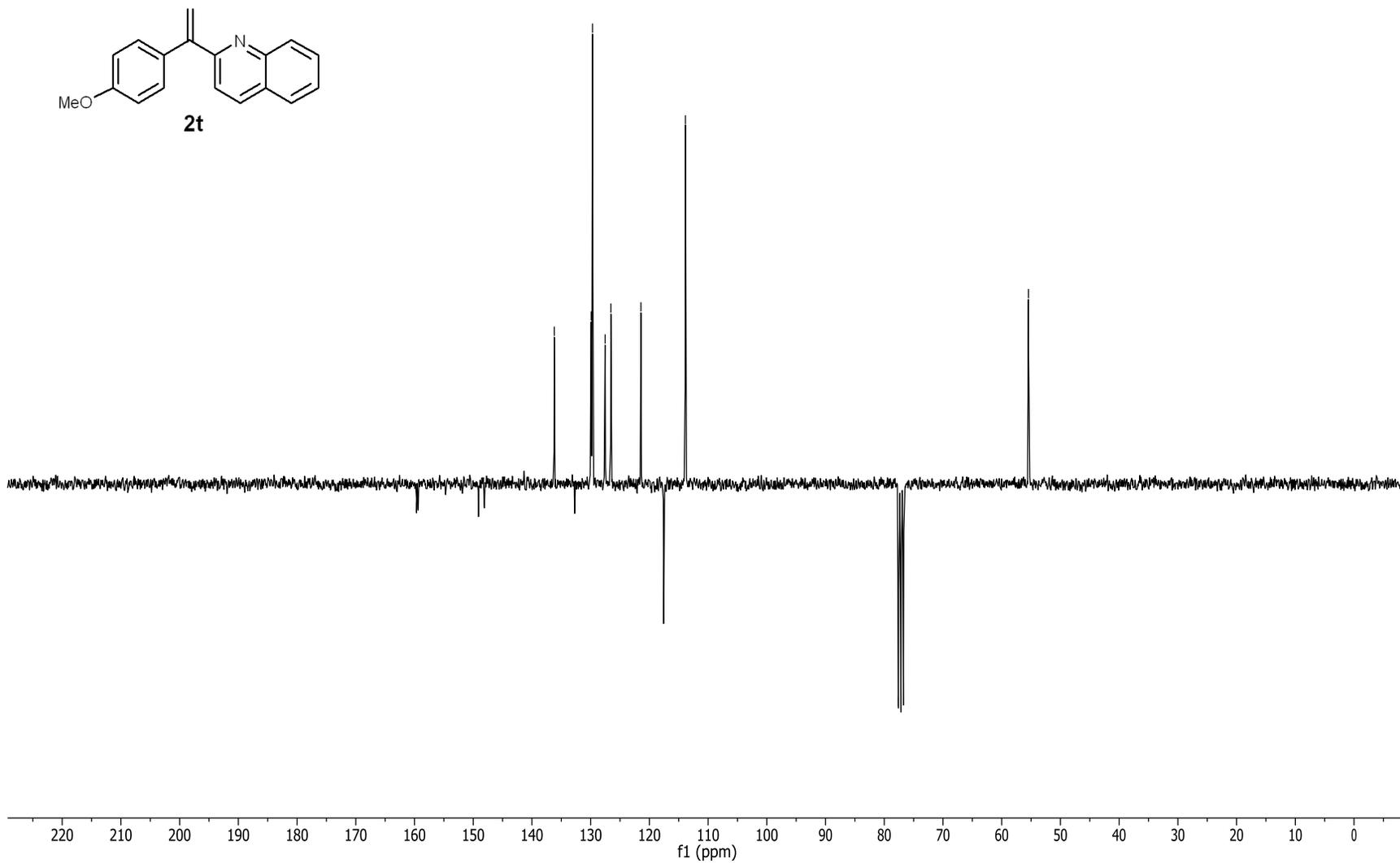
2012-11-02
ML 201
JMOD CDCl3 D:\\ chit 45



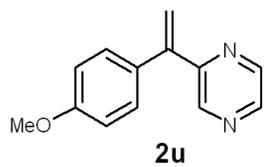
159.65
159.37
151.87
149.11
148.08
136.19
132.74
129.92
129.69
127.54
126.56
121.44
117.55
113.86

77.58

55.45



2012-10-18
ML 176
Proton.4 CDCl3 D:\ chit 32



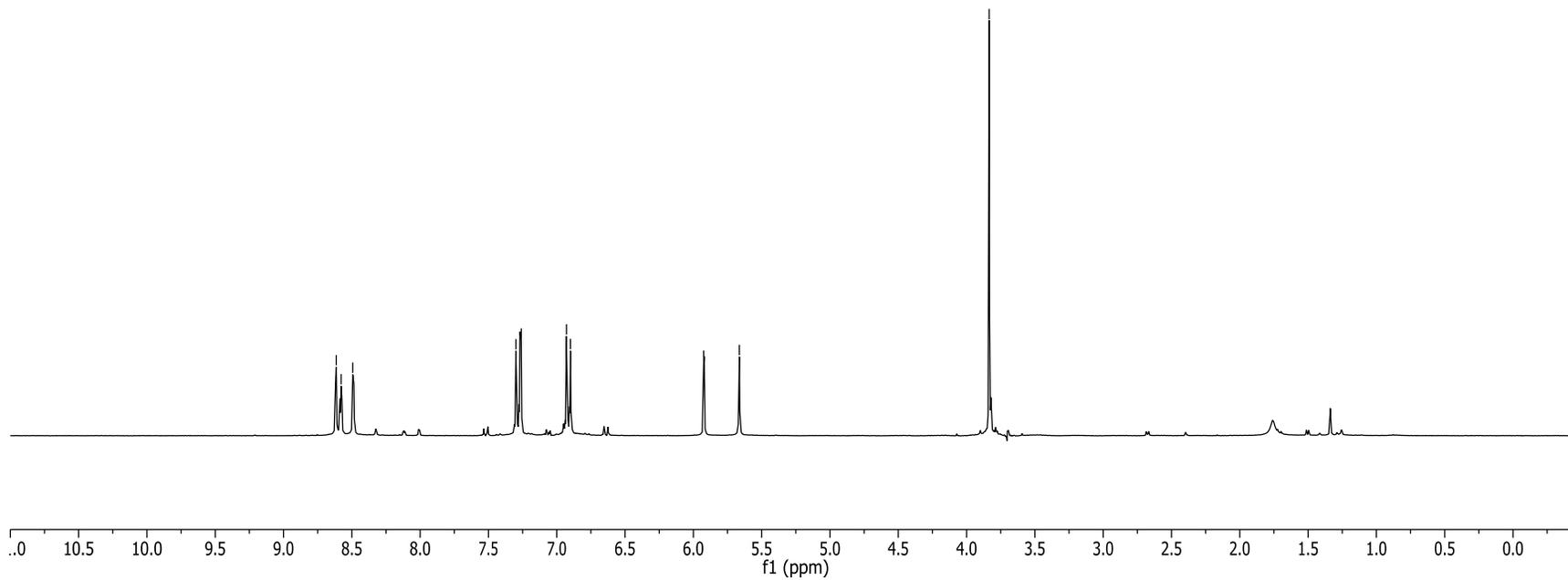
8.61
8.58
8.49

7.30
7.26

6.93
6.90

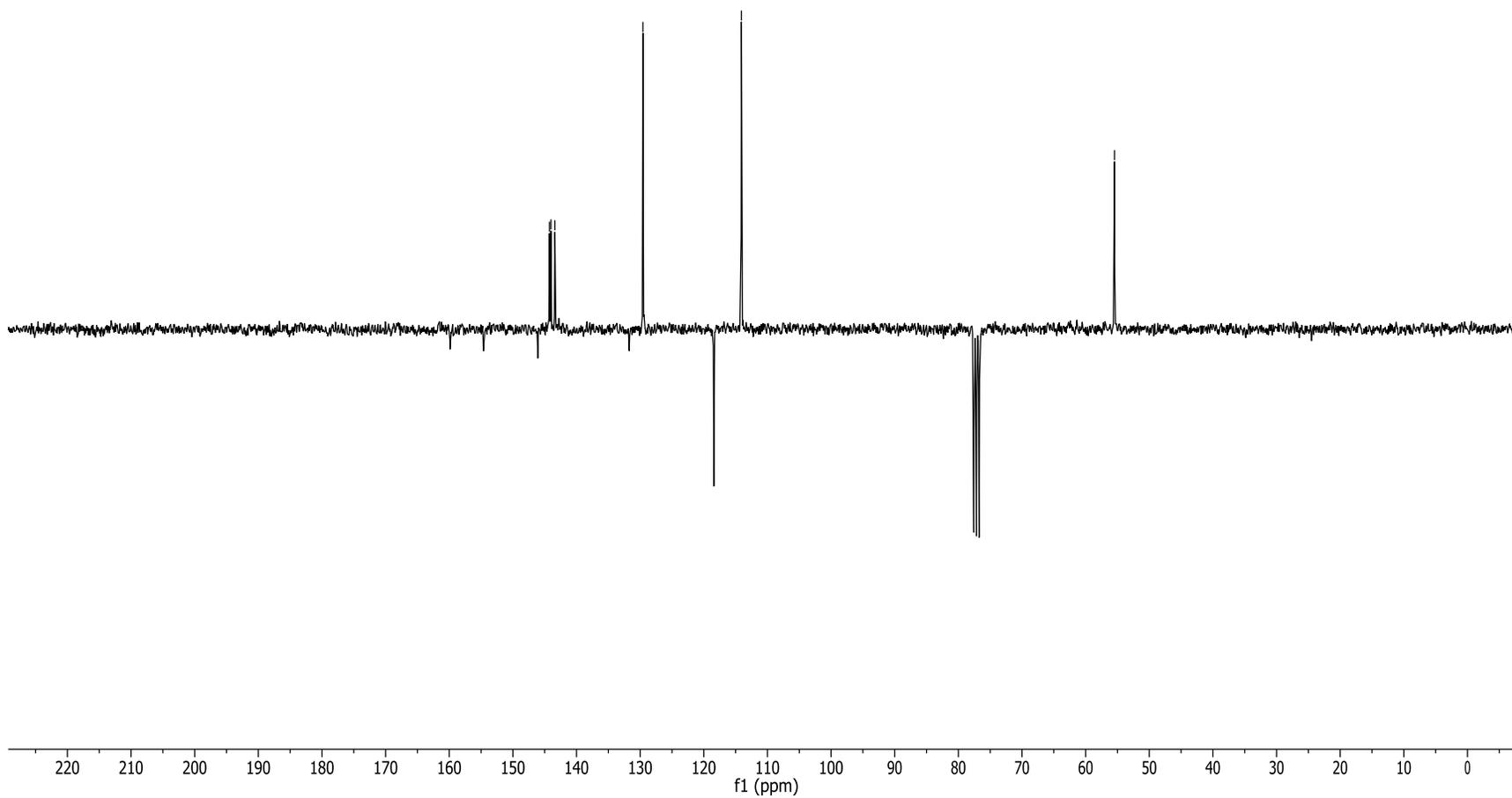
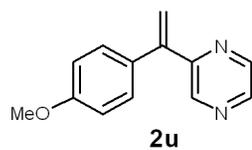
5.92
5.66

3.83

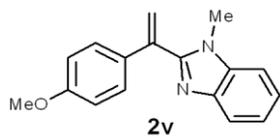


2012-12-05
ML 224
JMOD CDCl3 D:\\ chit 55

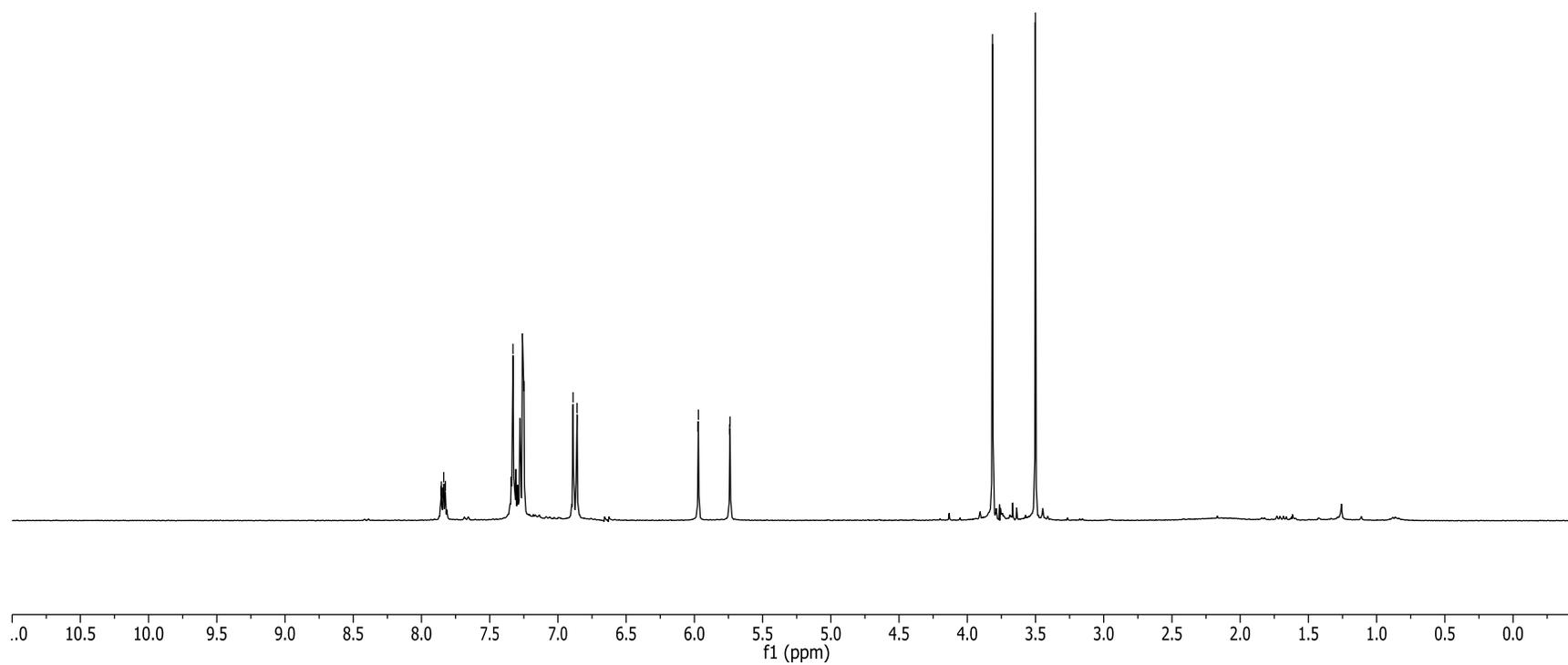
159.87
154.63
146.08
144.25
144.01
143.40
131.73
129.57
118.43
114.09
55.47



2012-11-13
ML 217
Proton.4 CDCl3 D:\\ chit 37

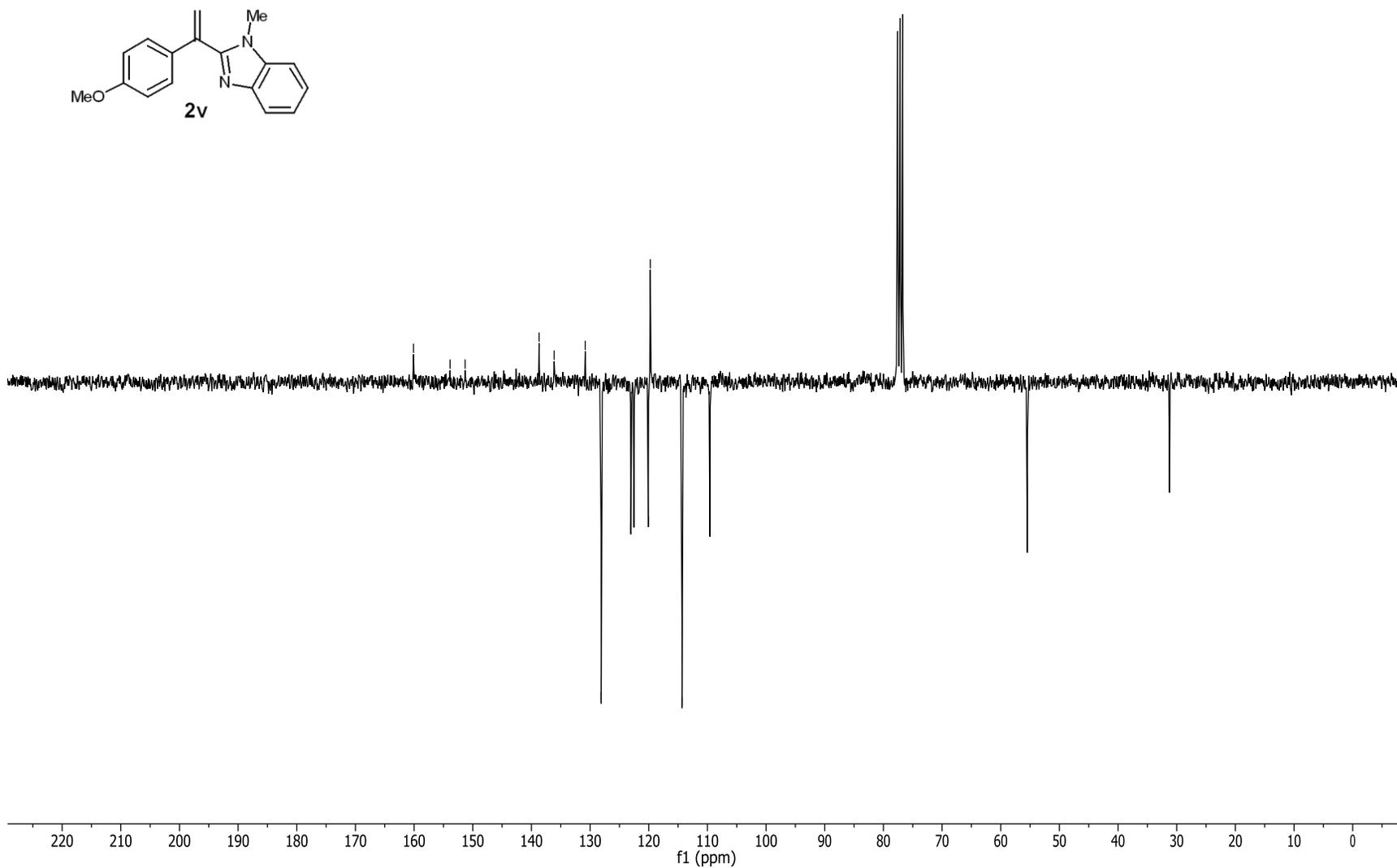
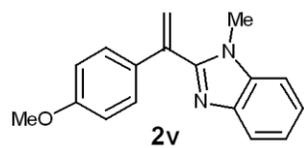


7.86
7.85
7.85
7.84
7.84
7.83
7.82
7.33
7.27
7.25
6.89
6.86
5.97
5.97
5.74
5.74
3.82
3.50

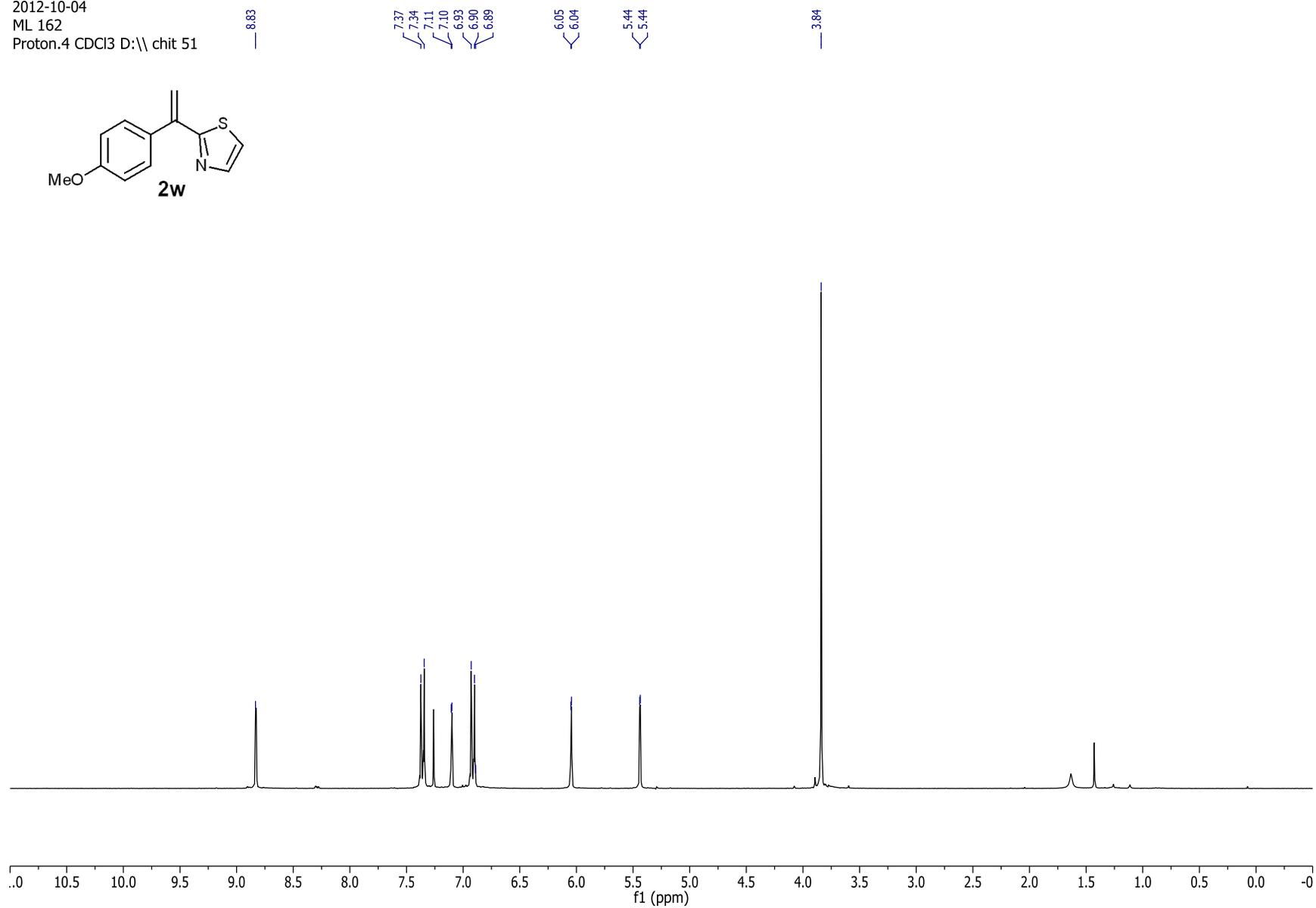
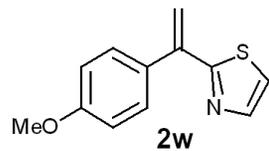


2012-11-13
ML 217
JMOD CDCl3 D:\\ chit 47

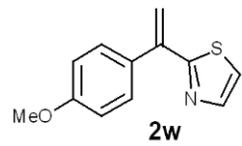
160.10
153.88
151.31
138.69
136.13
130.83
128.11
123.04
122.52
120.10
119.74
114.33
109.58
55.48
31.22



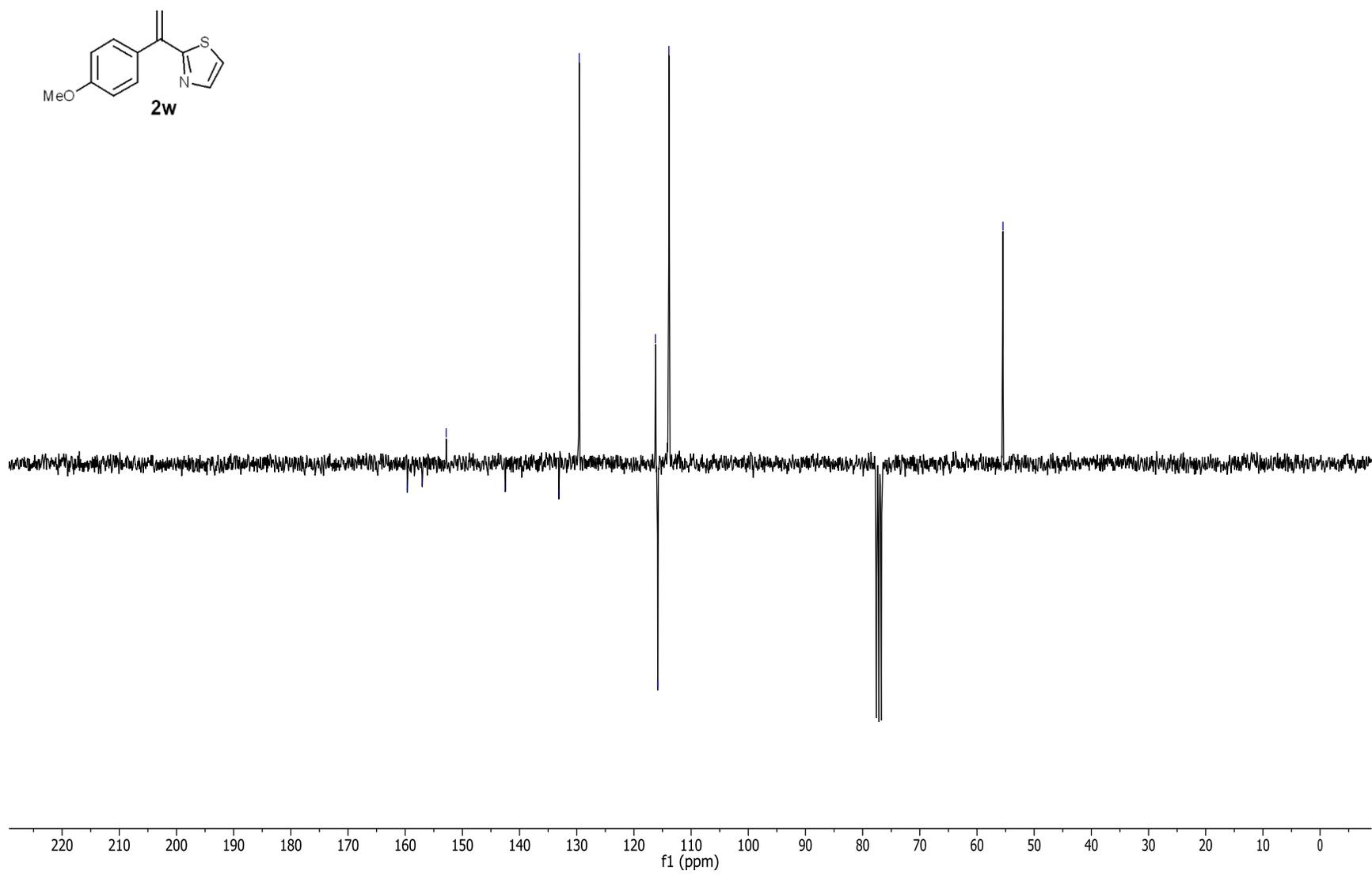
2012-10-04
ML 162
Proton.4 CDCl3 D:\ chit 51



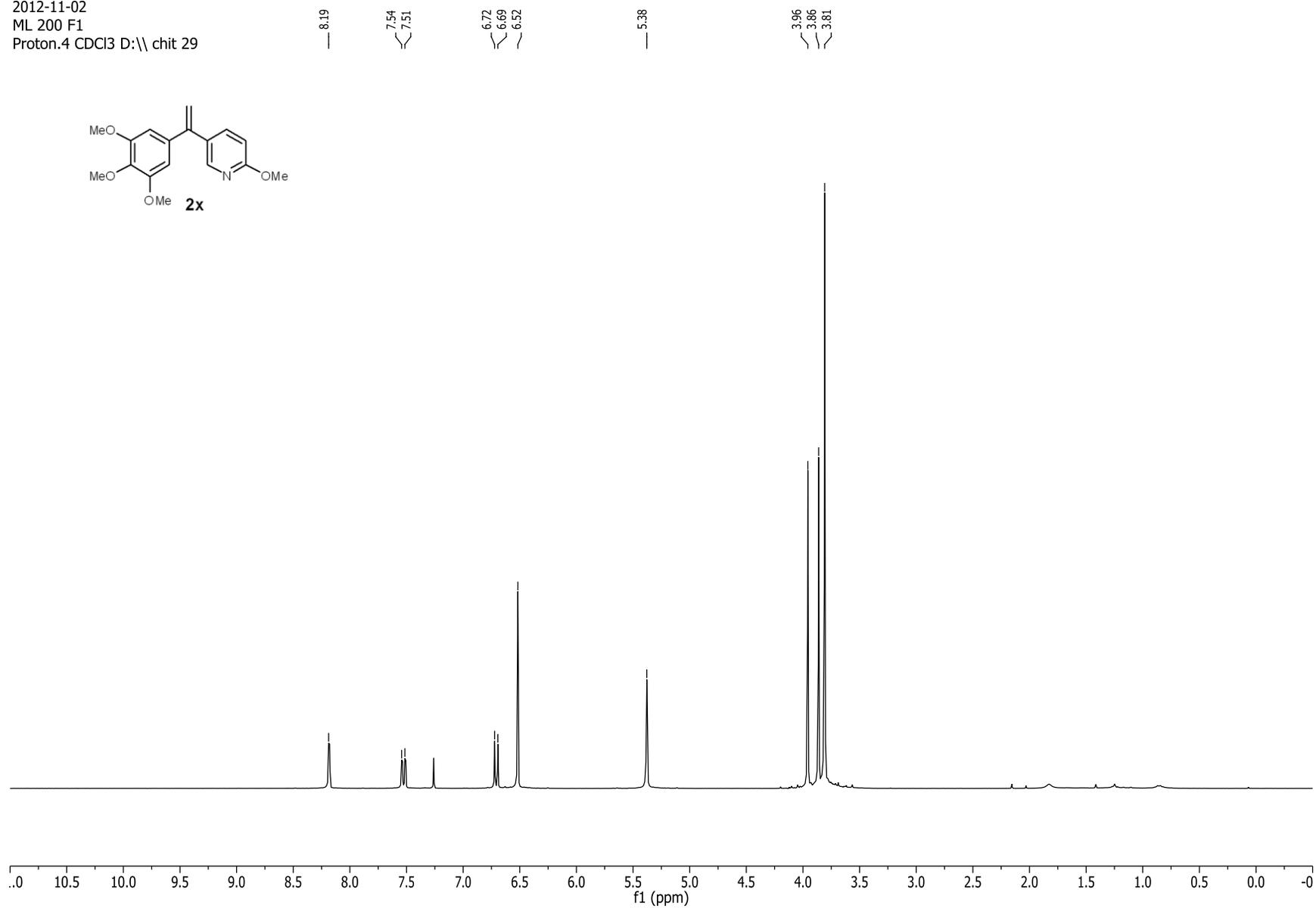
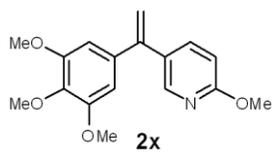
2012-10-08
ML 162
JMOD CDCl3 D:\ chit 47



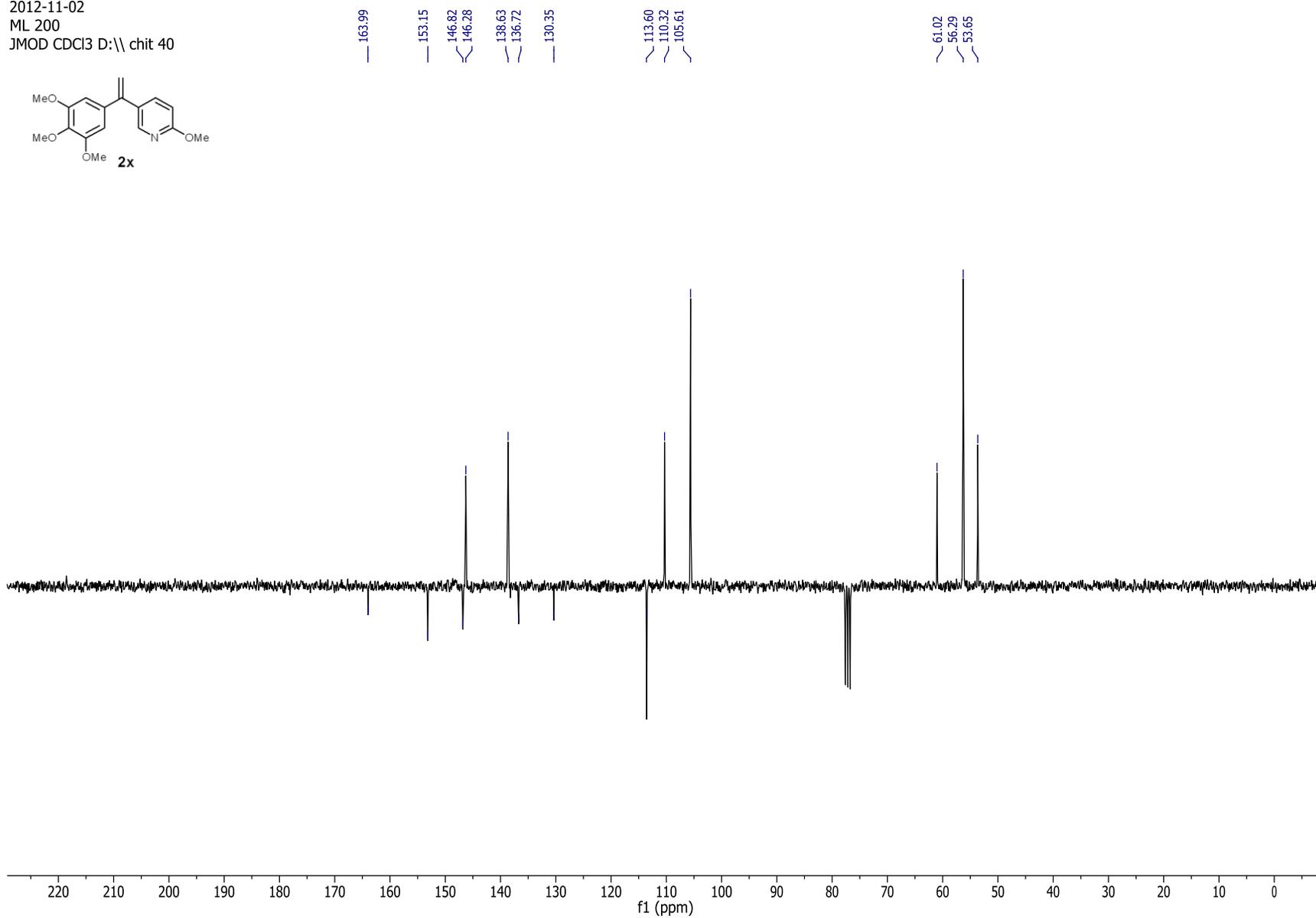
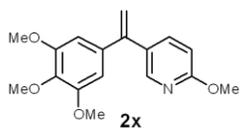
159.63
157.02
152.83
142.51
133.14
129.57
116.23
115.83
113.88
55.46



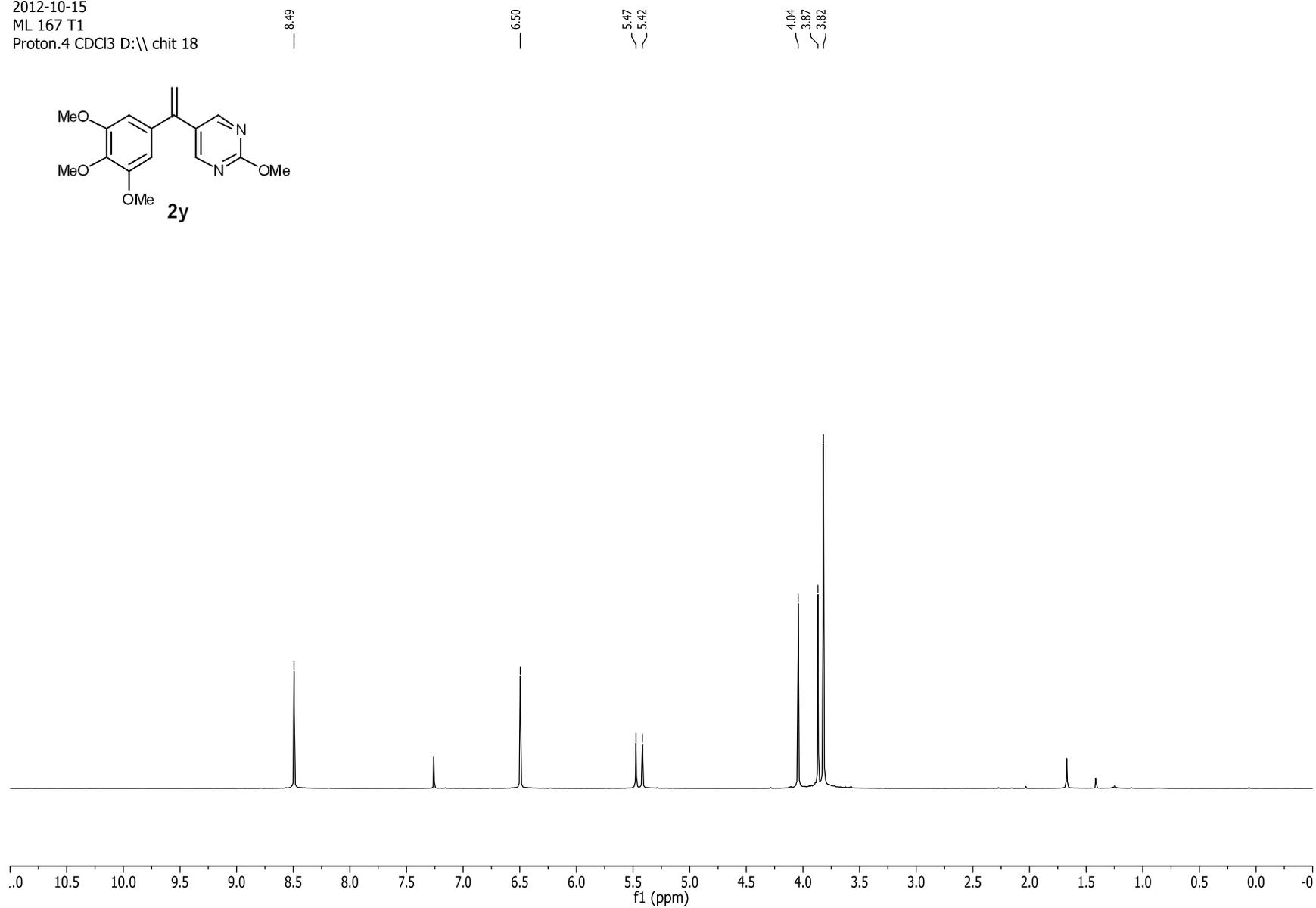
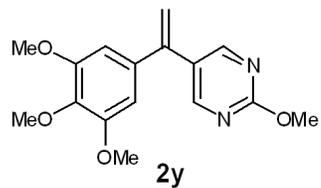
2012-11-02
ML 200 F1
Proton.4 CDCl3 D:\ chit 29



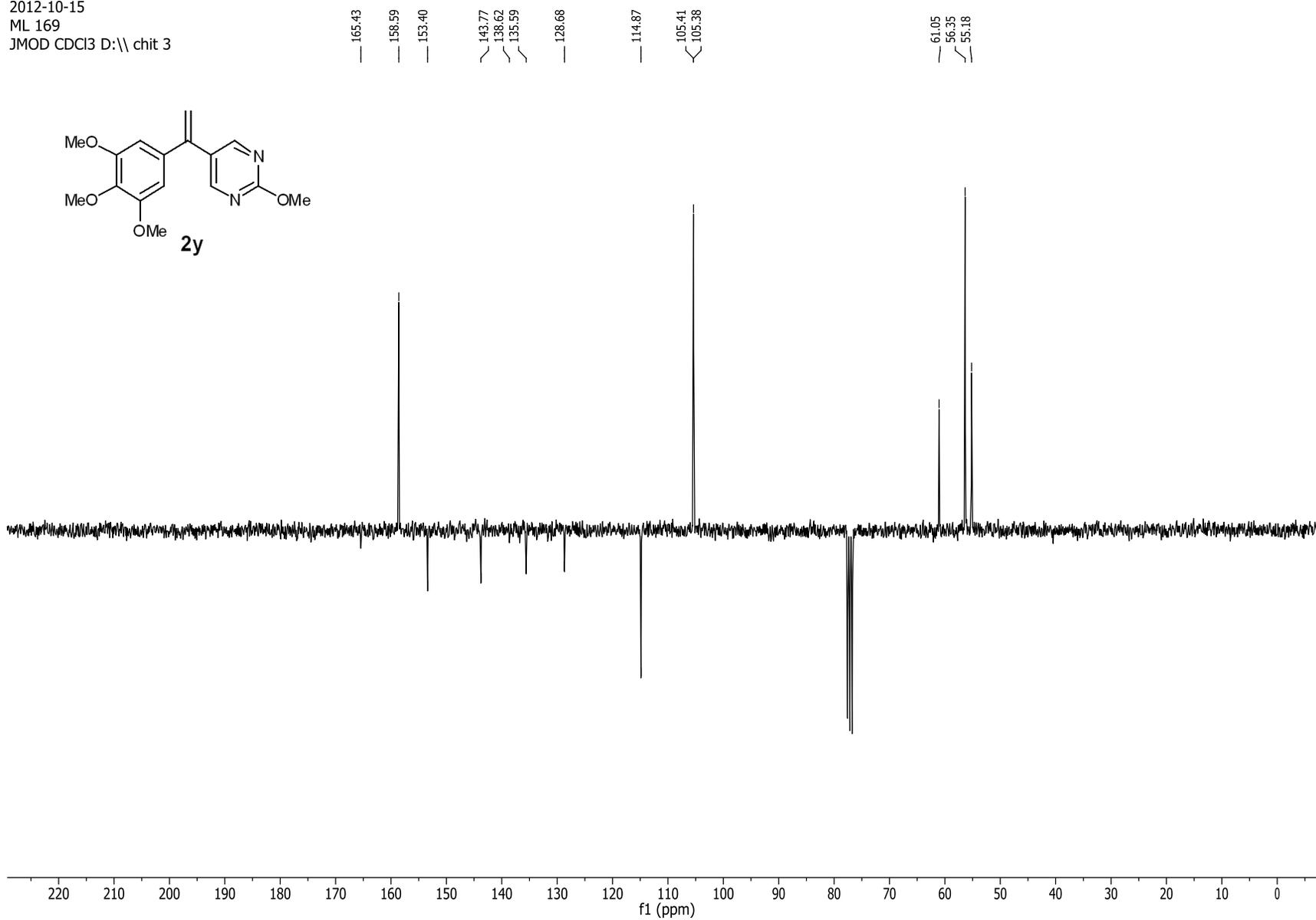
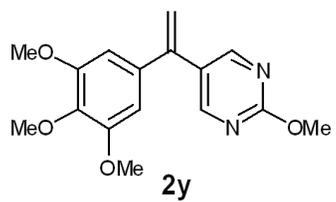
2012-11-02
ML 200
JMOD CDCl3 D:\ chit 40



2012-10-15
ML 167 T1
Proton.4 CDCl3 D:\ chit 18



2012-10-15
ML 169
JMOD CDCl3 D:\\ chit 3



2012-05-14
ML 144
Proton.4 Acetone D:\ chit 1

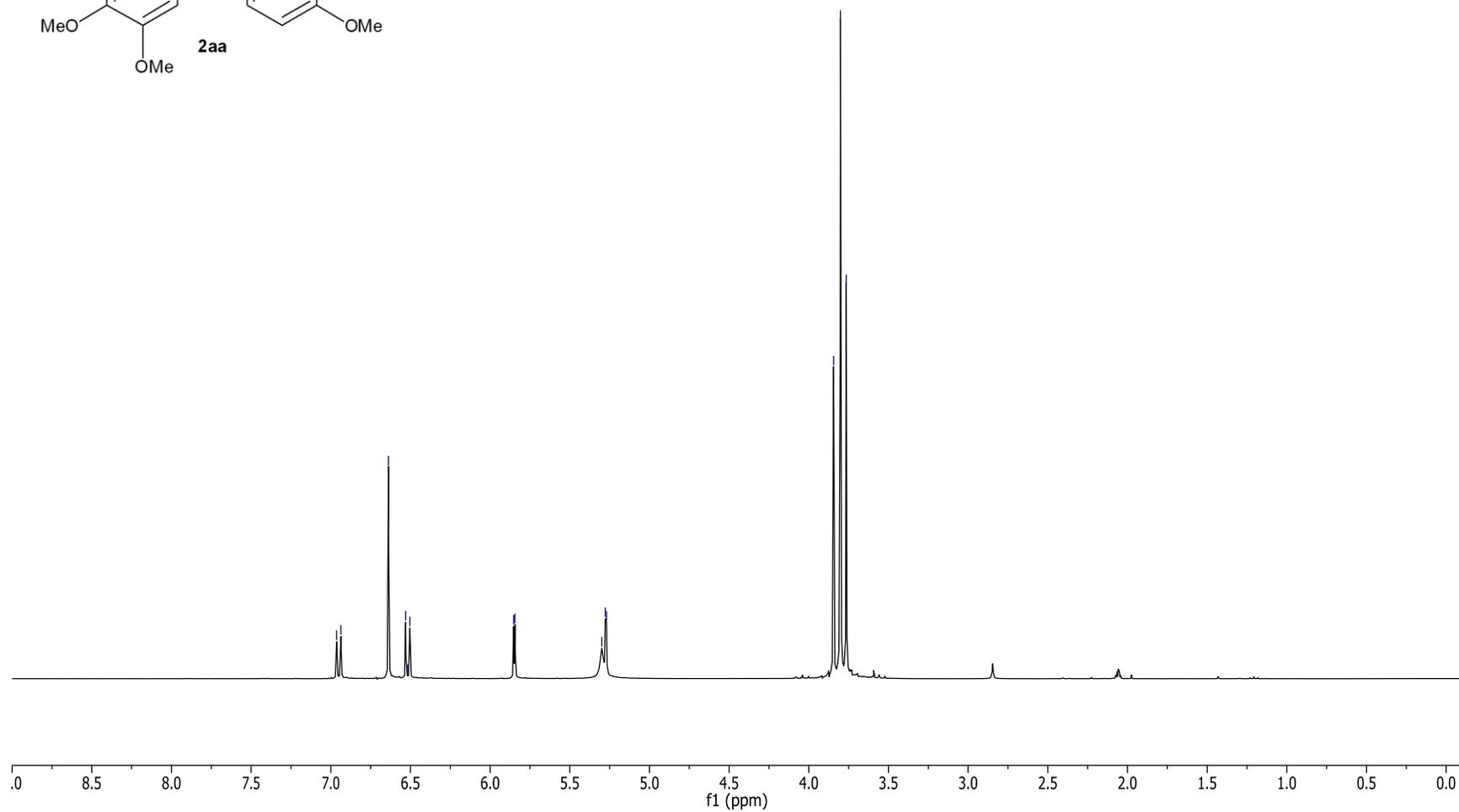
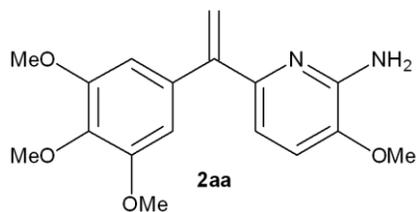
6.96
6.94

6.64
6.53
6.52
6.50

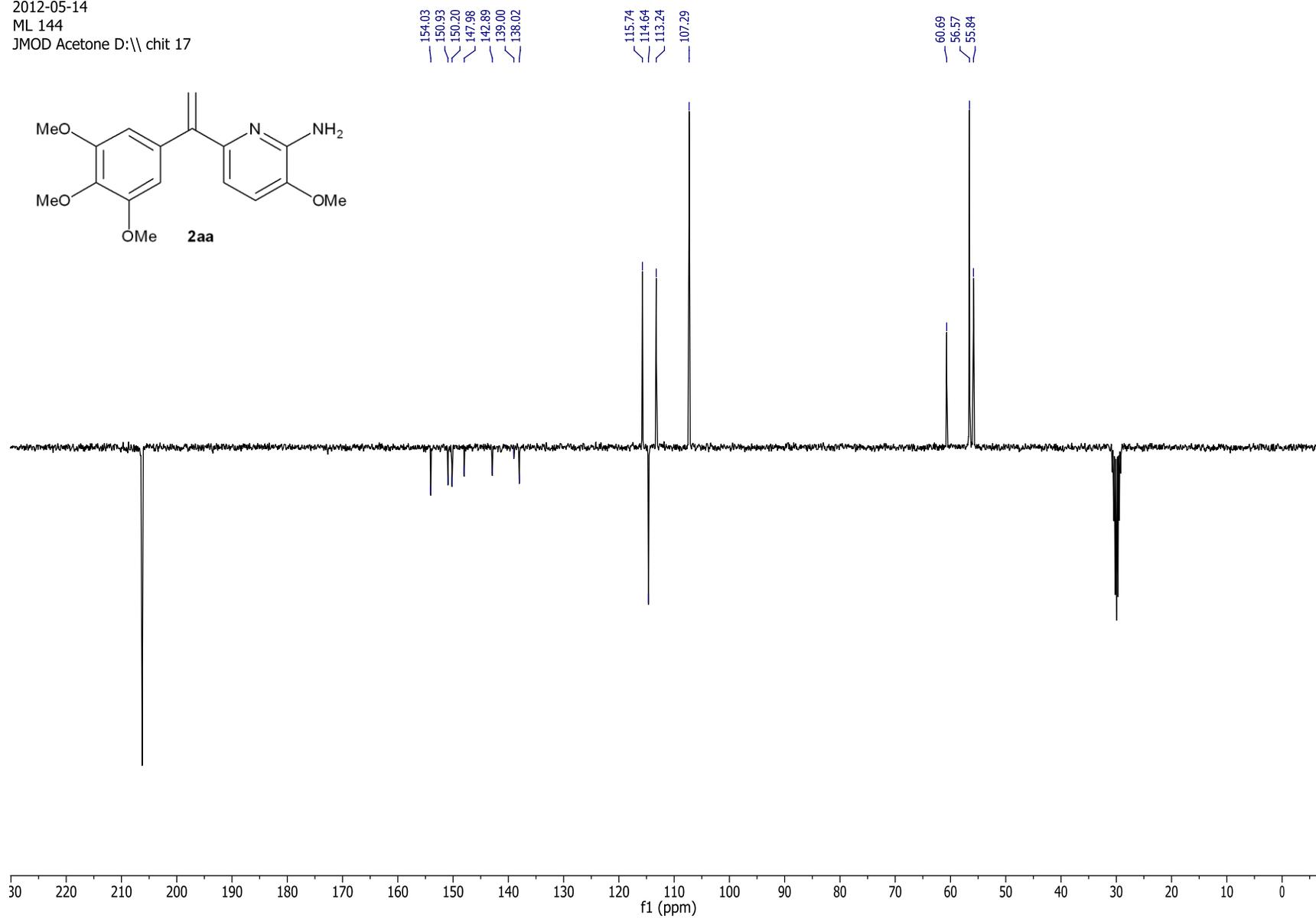
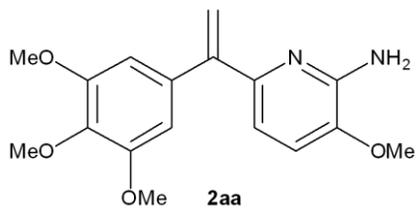
5.85
5.84

5.30
5.28
5.27

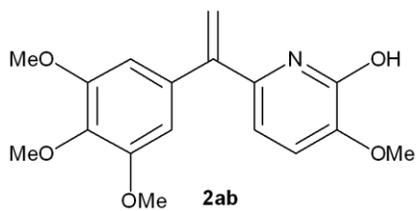
3.84
3.80
3.76



2012-05-14
ML 144
JMOD Acetone D:\\ chit 17

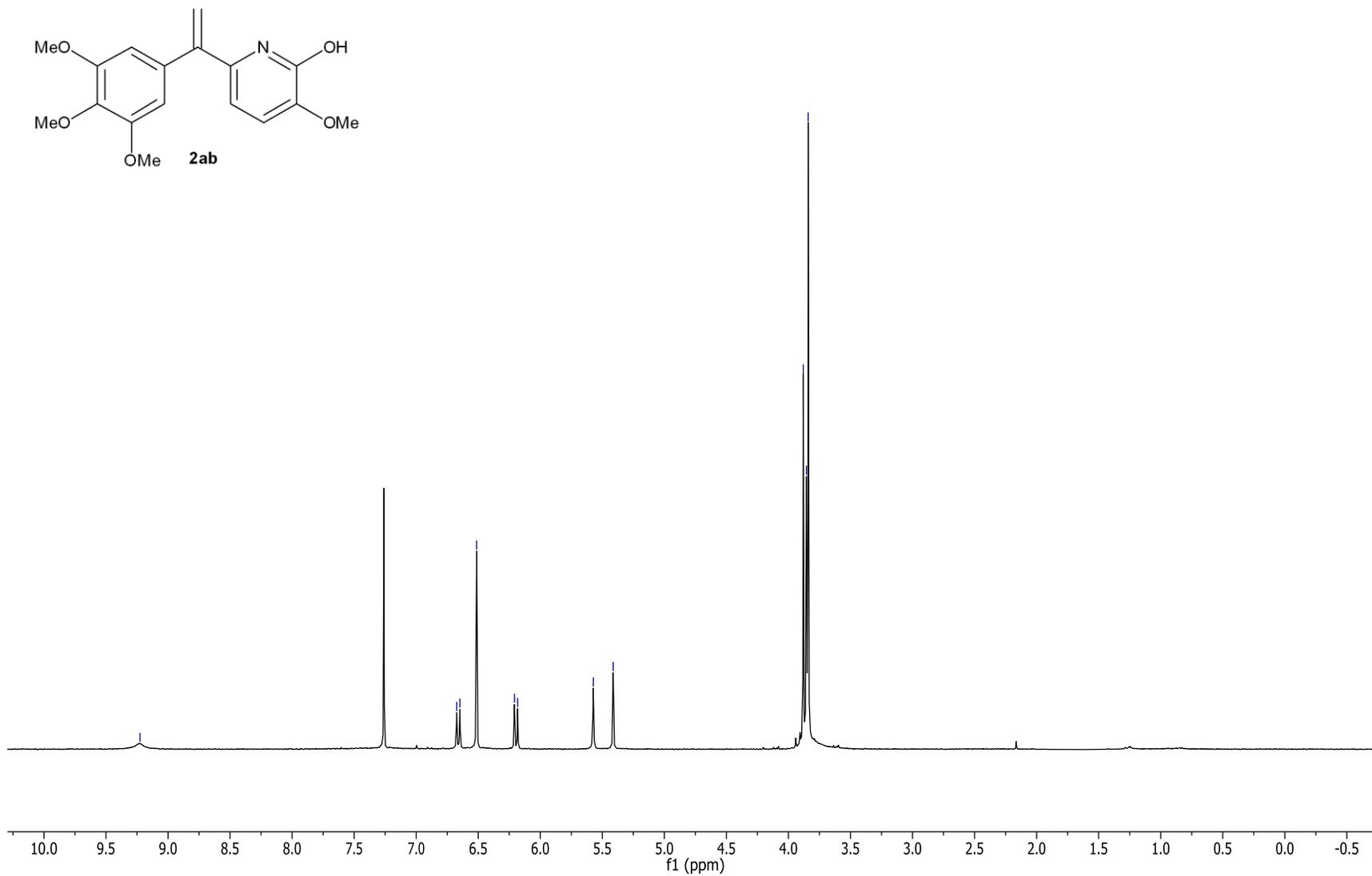


2012-05-21
ML 148
Proton.4 CDCl3 D:\ chit 12



6.67
6.65
6.51
6.21
6.18
5.57
5.41

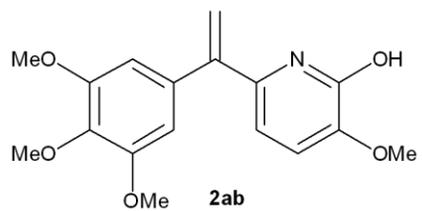
3.88
3.85
3.84



2012-05-22

ML 148

JMOD CDCl3 D:\chit 43



158.64
153.31
150.02

143.02
138.64
136.31
134.09

115.65
113.87

106.39
106.14

61.02
56.03

