Electronic Supplementary Material (ESI) for RSC Advances

Supporting Information

A new ferrocene-based bulky pyridine as an efficient reusable homogeneous catalyst

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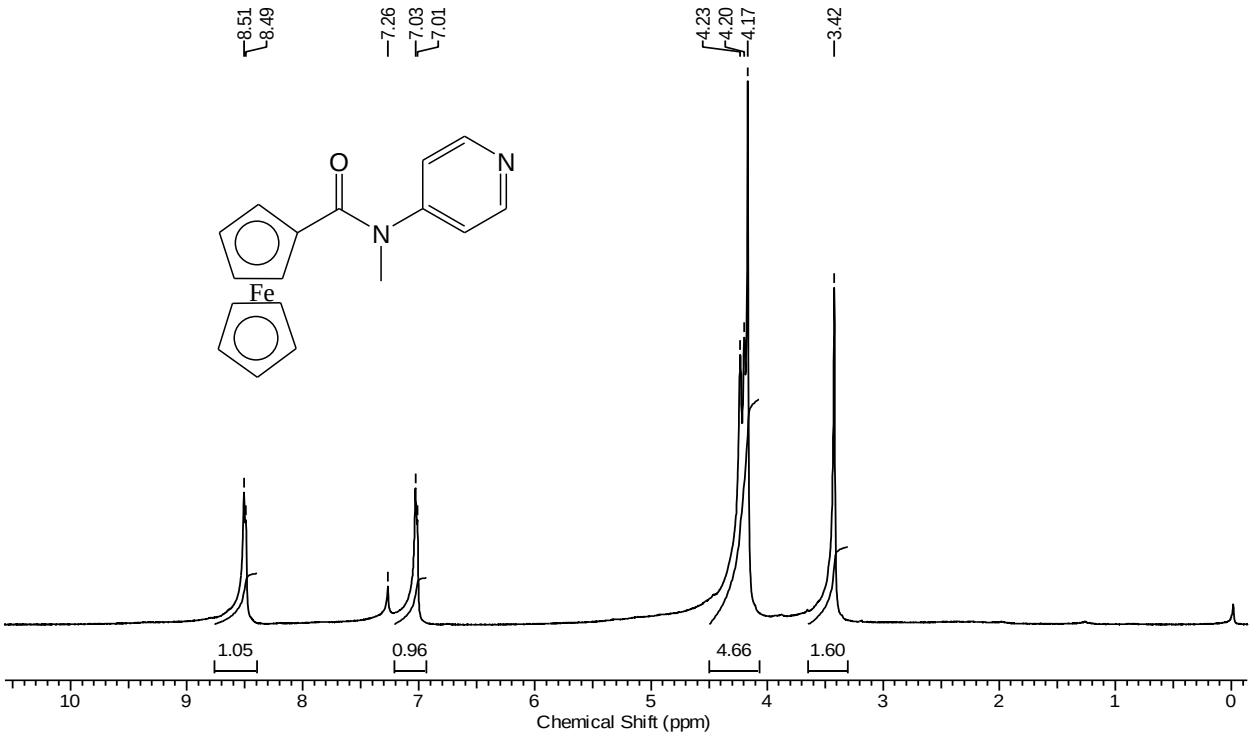
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4
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2
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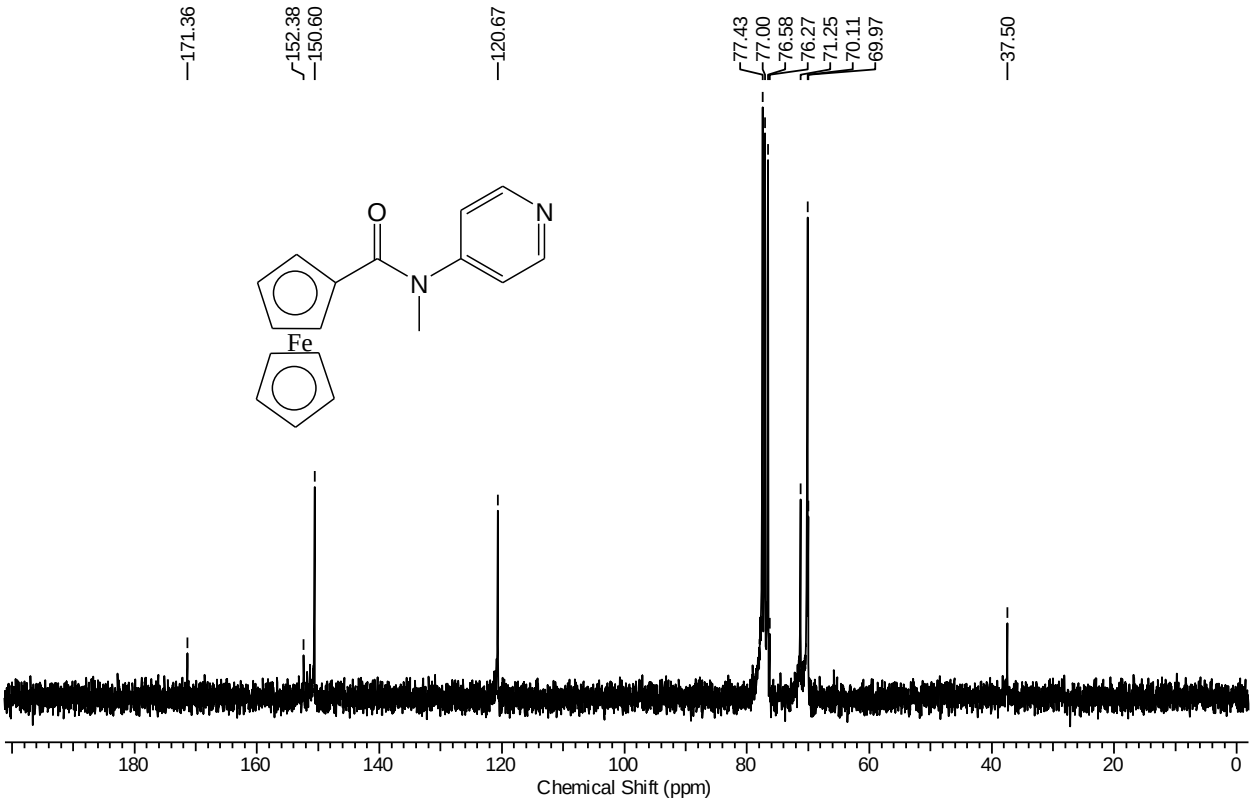
Chemical Shift (ppm)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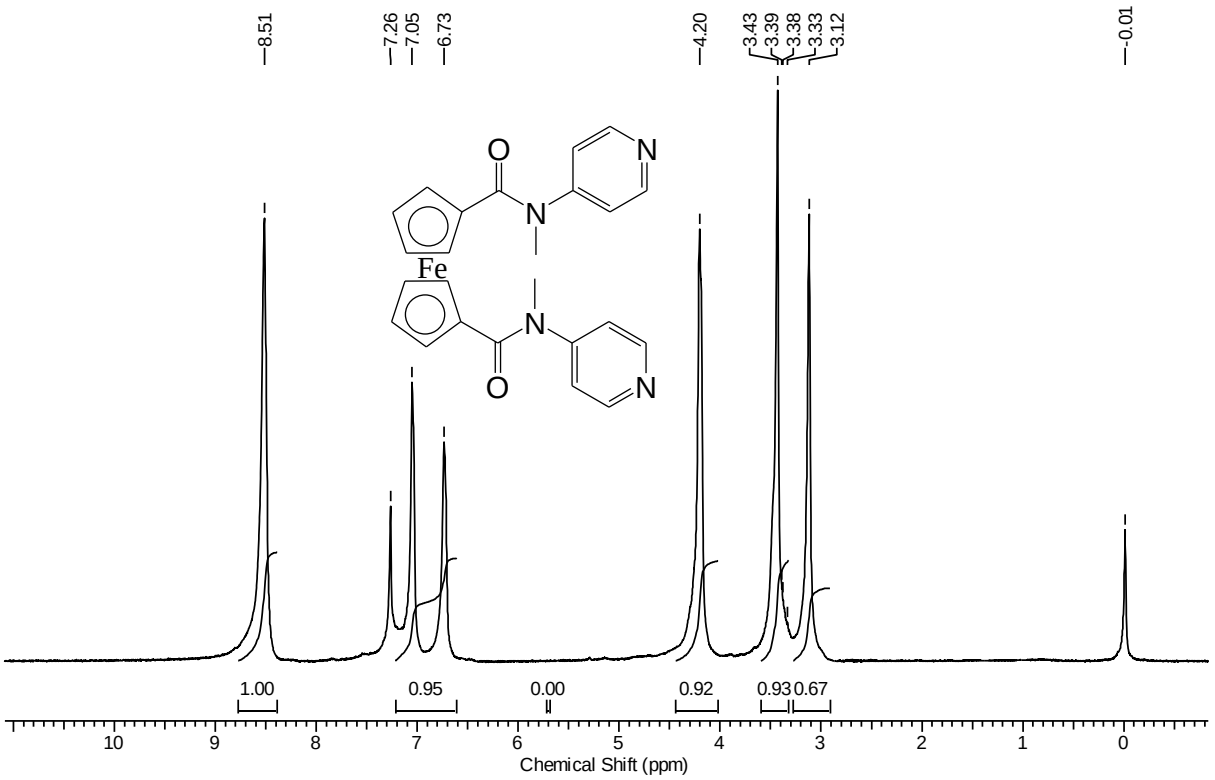
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Fe
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O
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0.95
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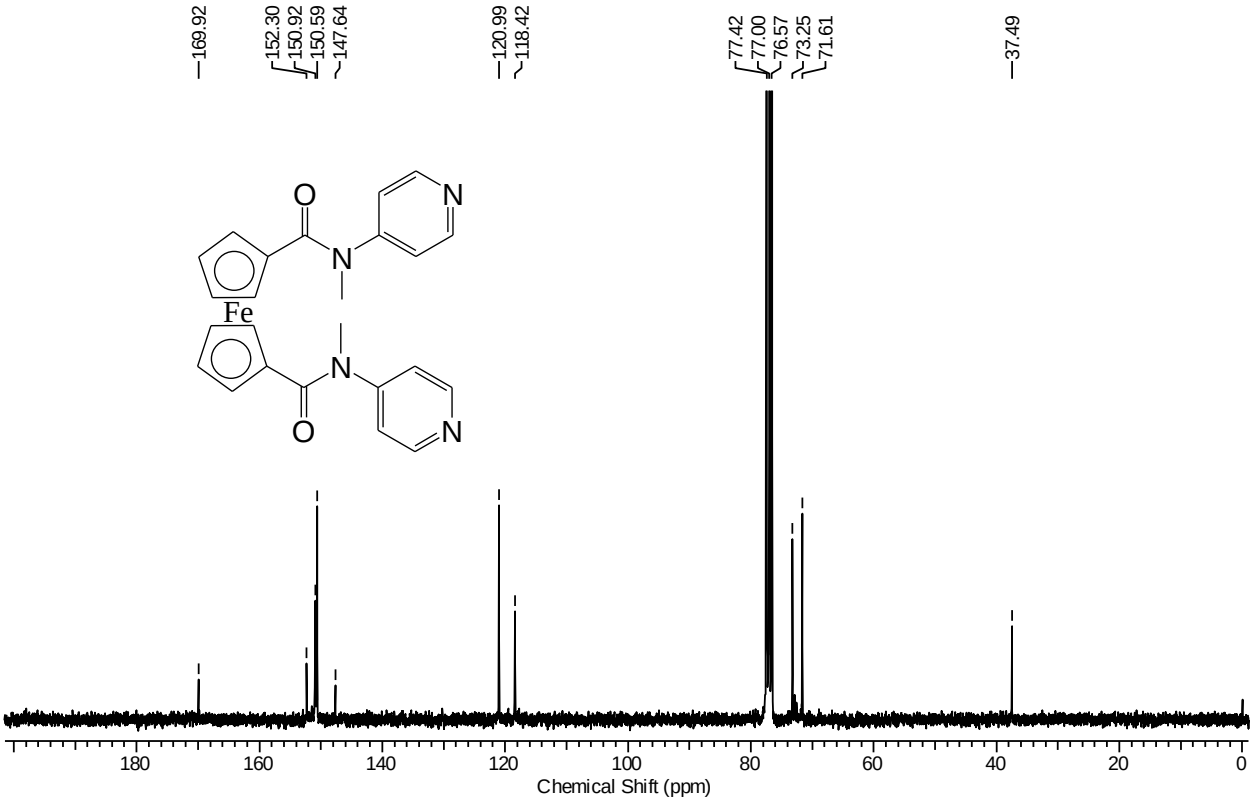
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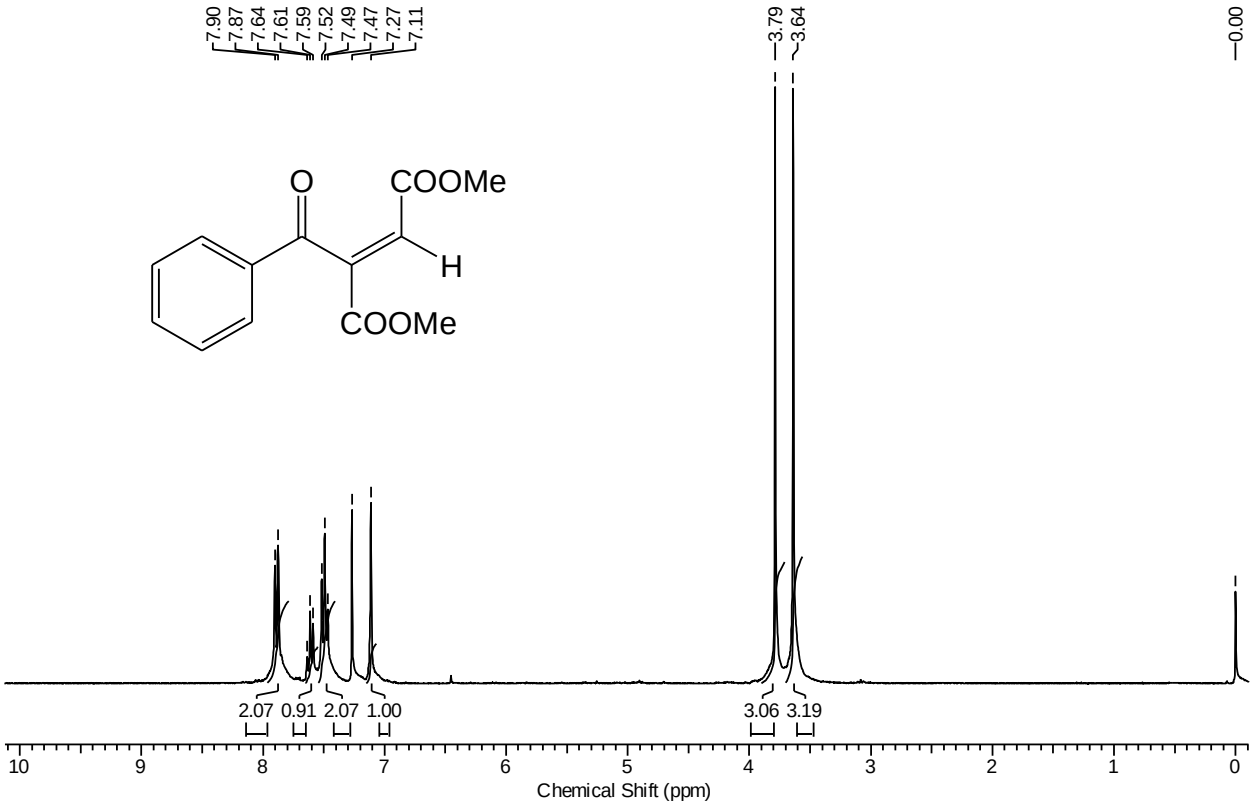
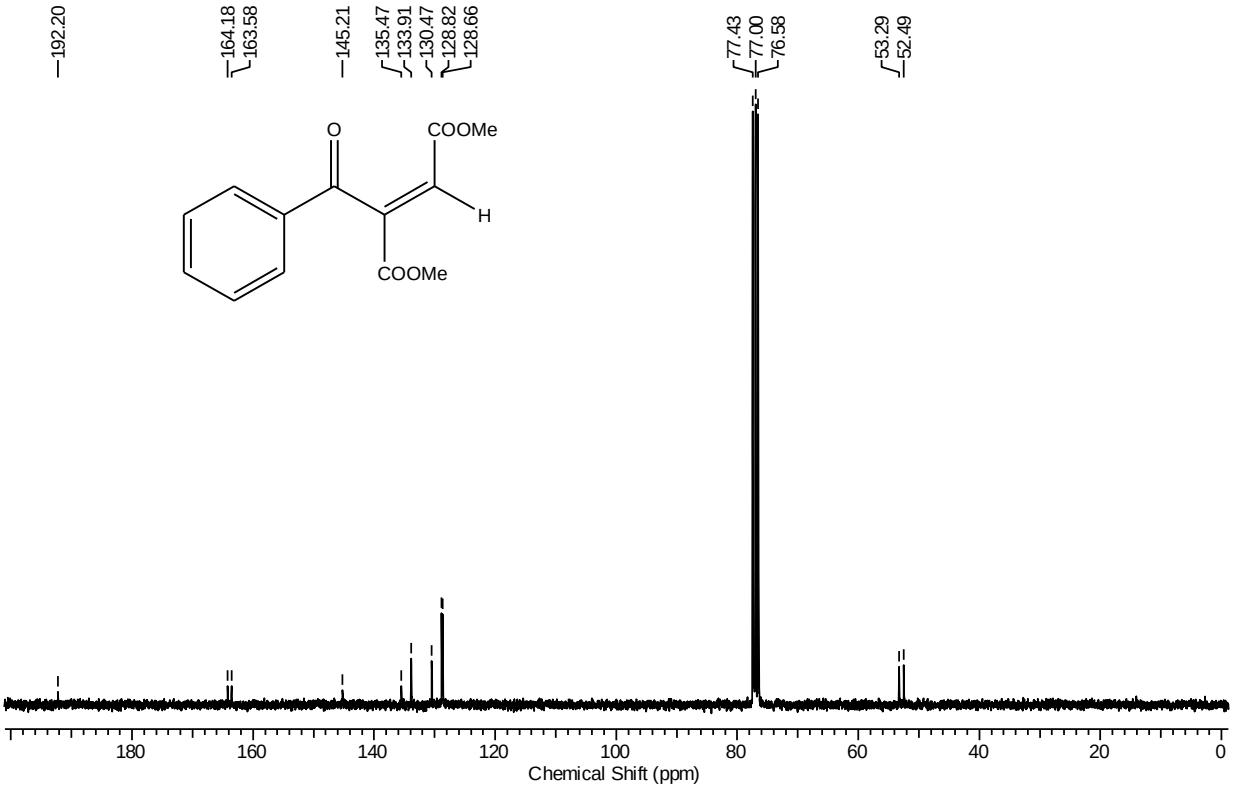
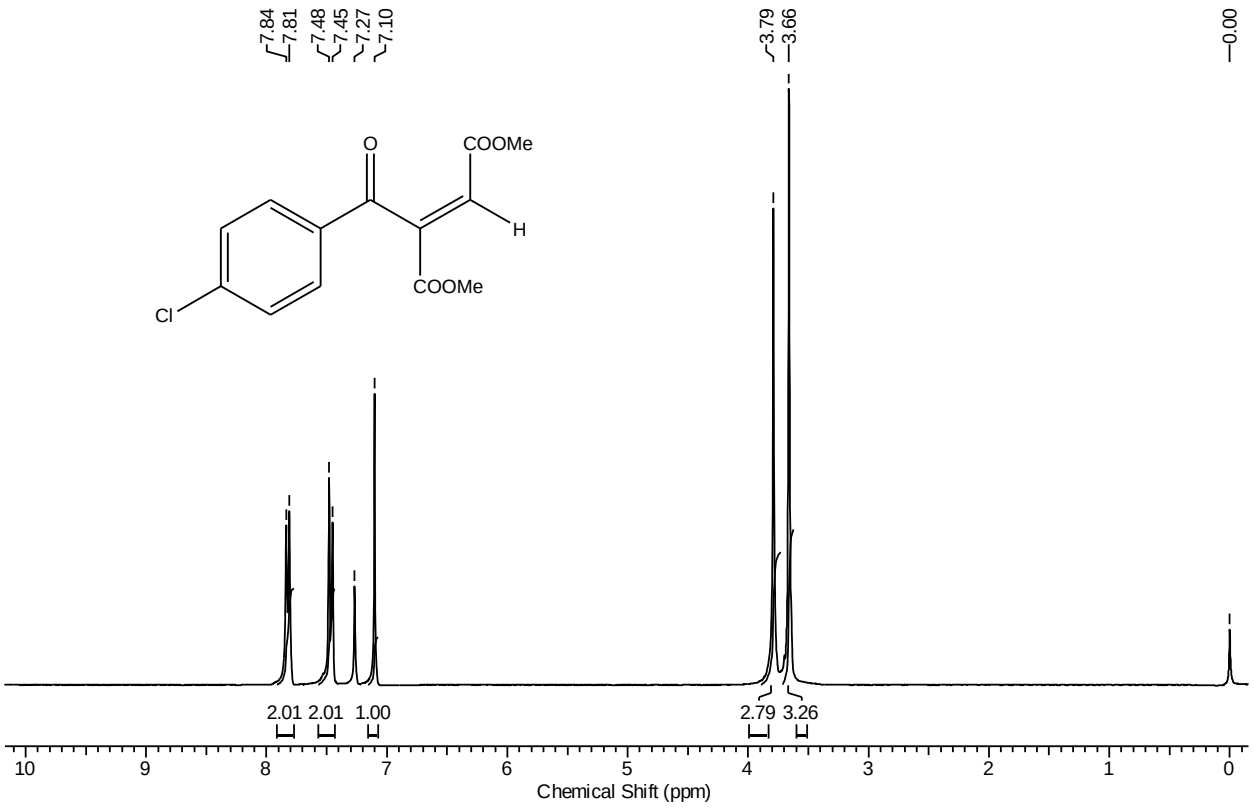
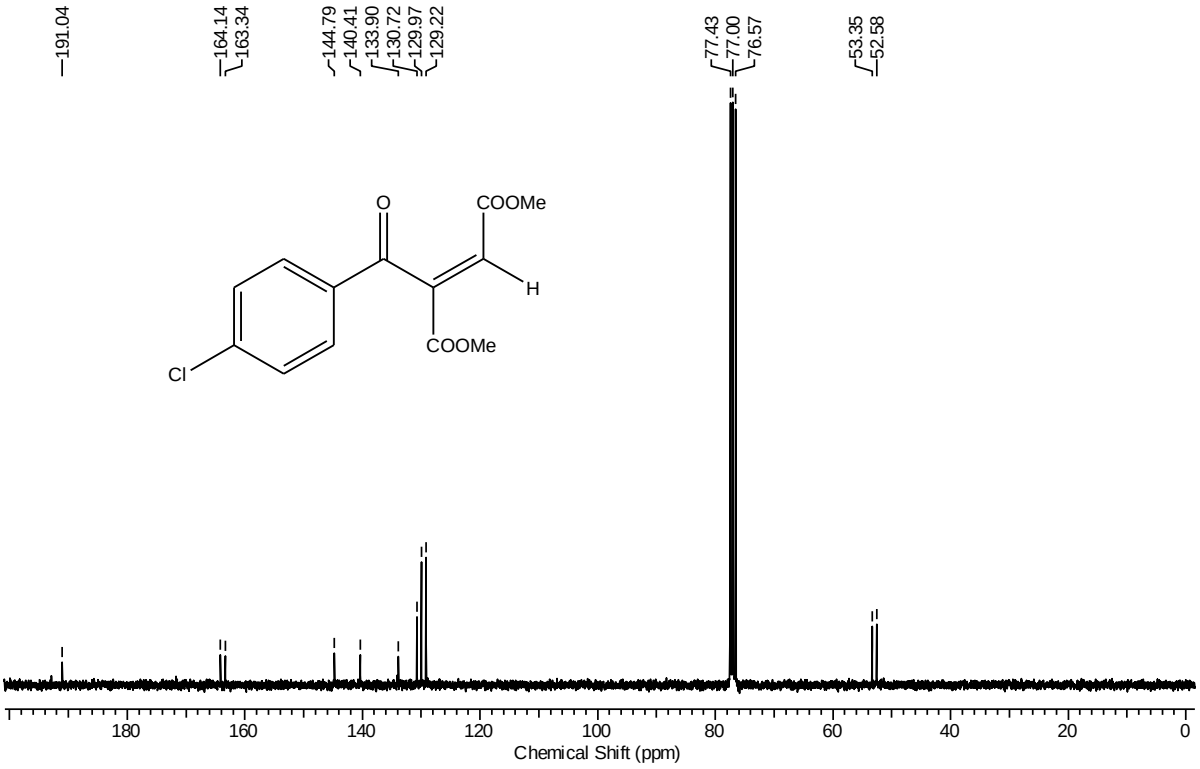
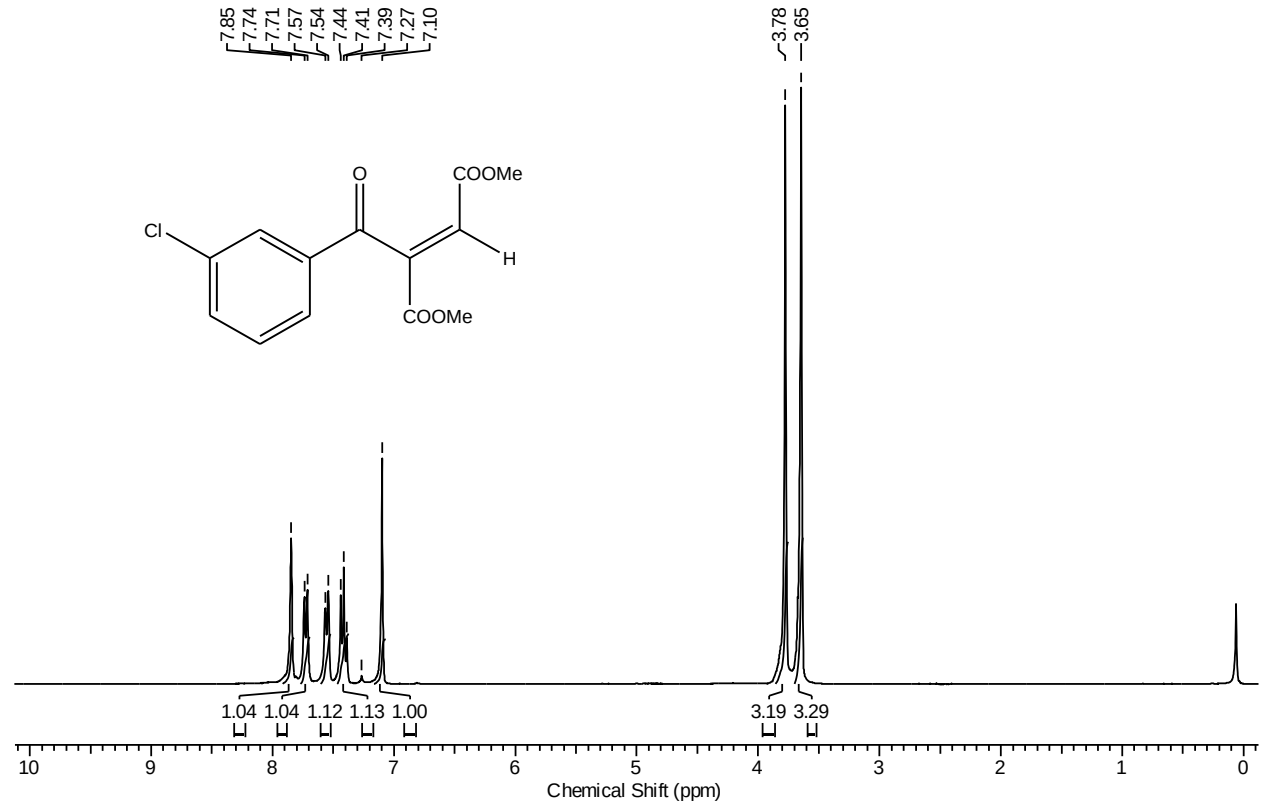
Chemical Shift (ppm)

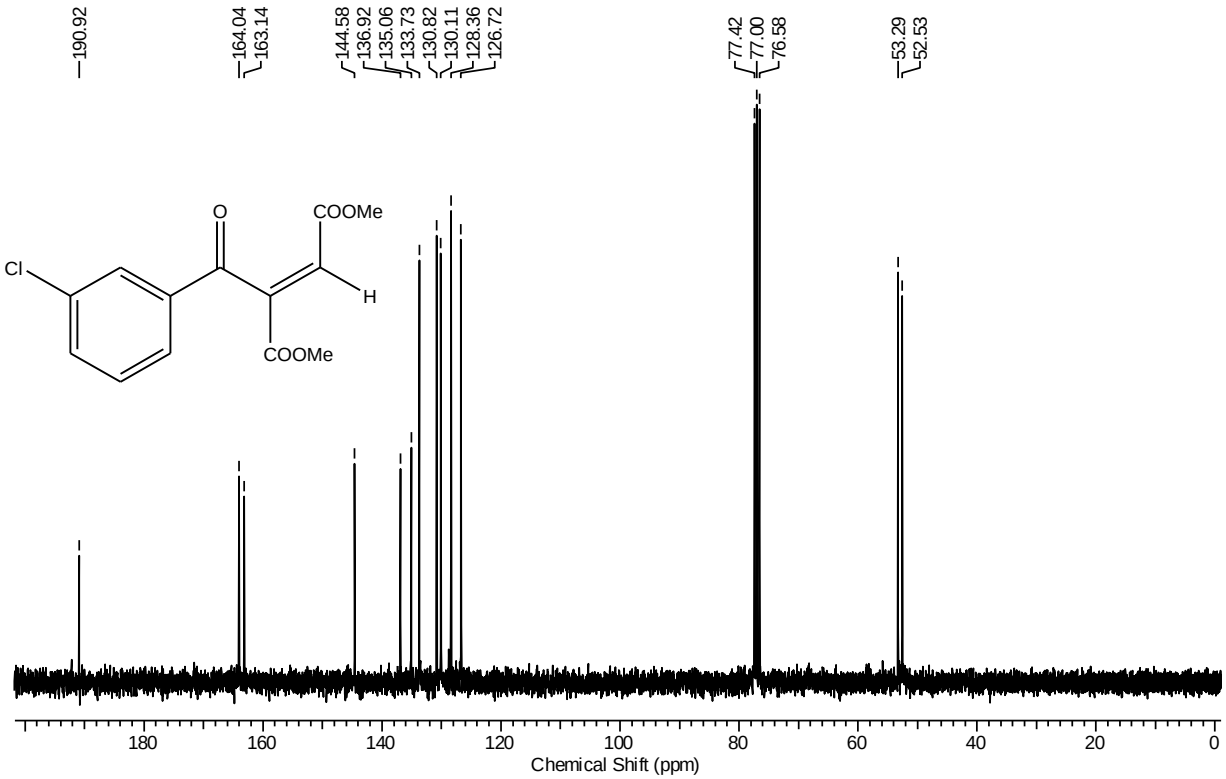


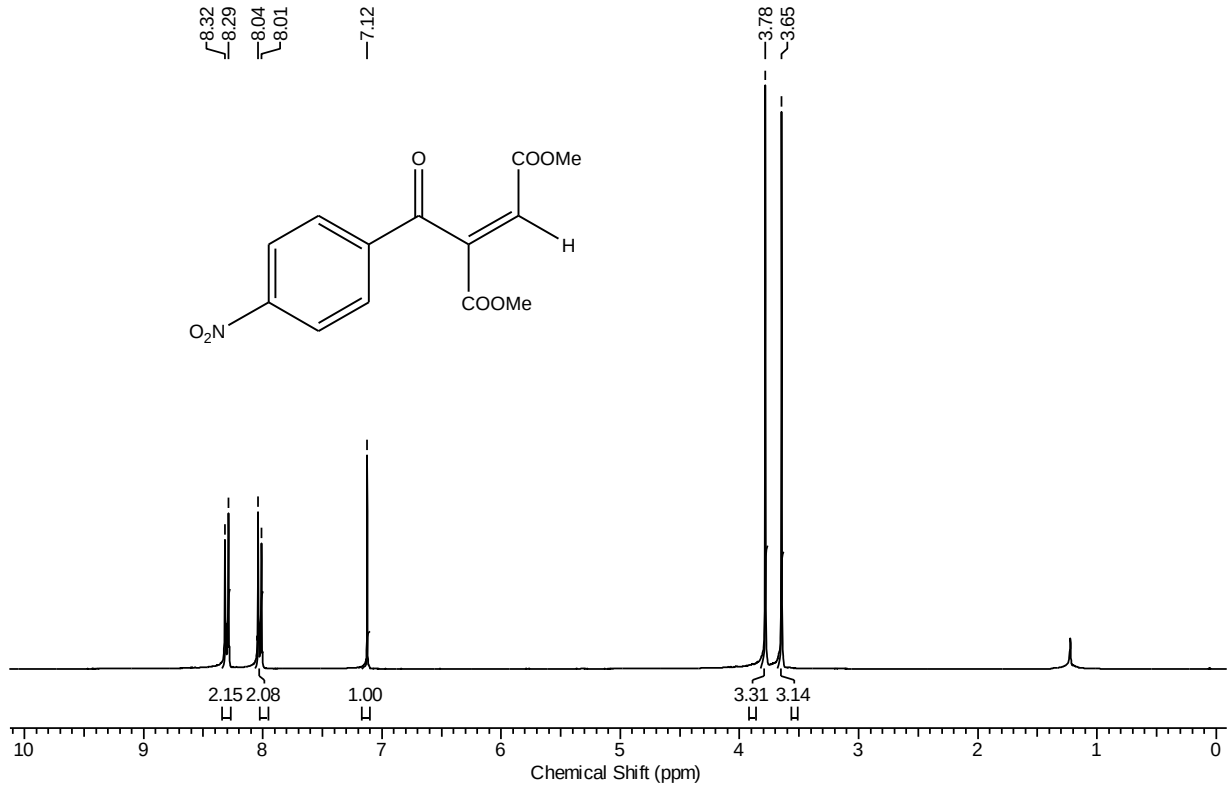


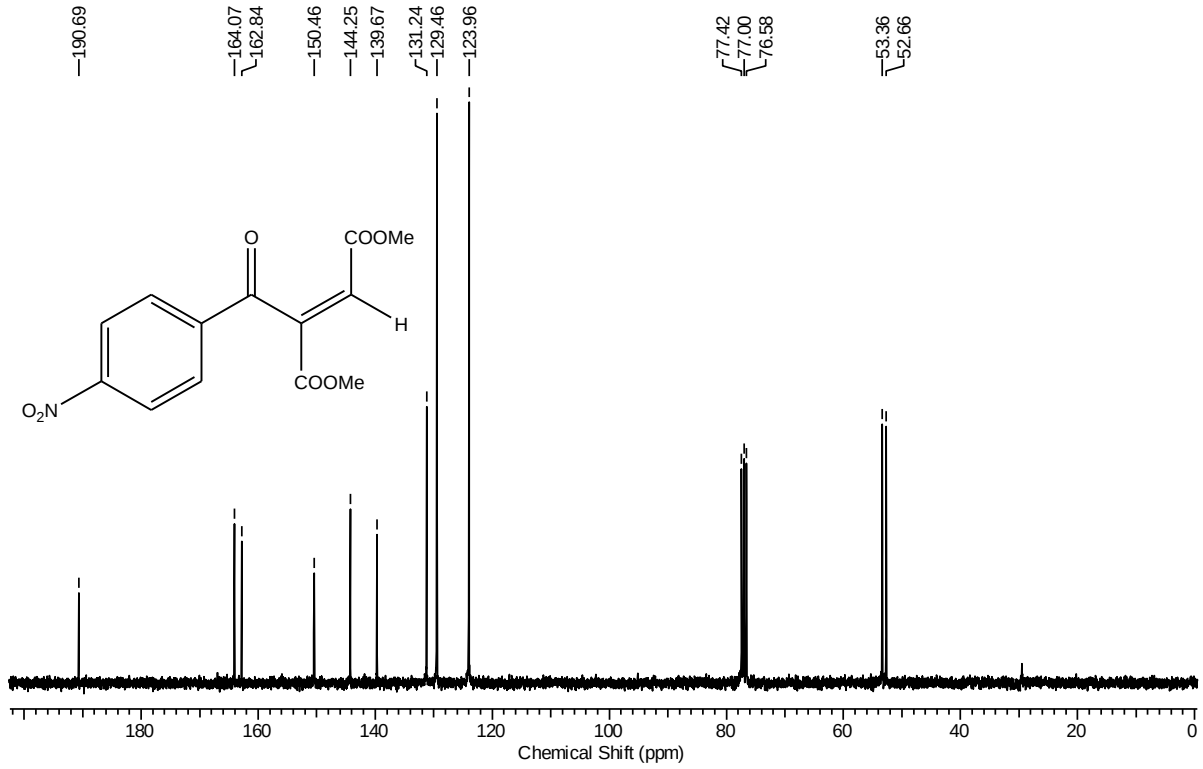


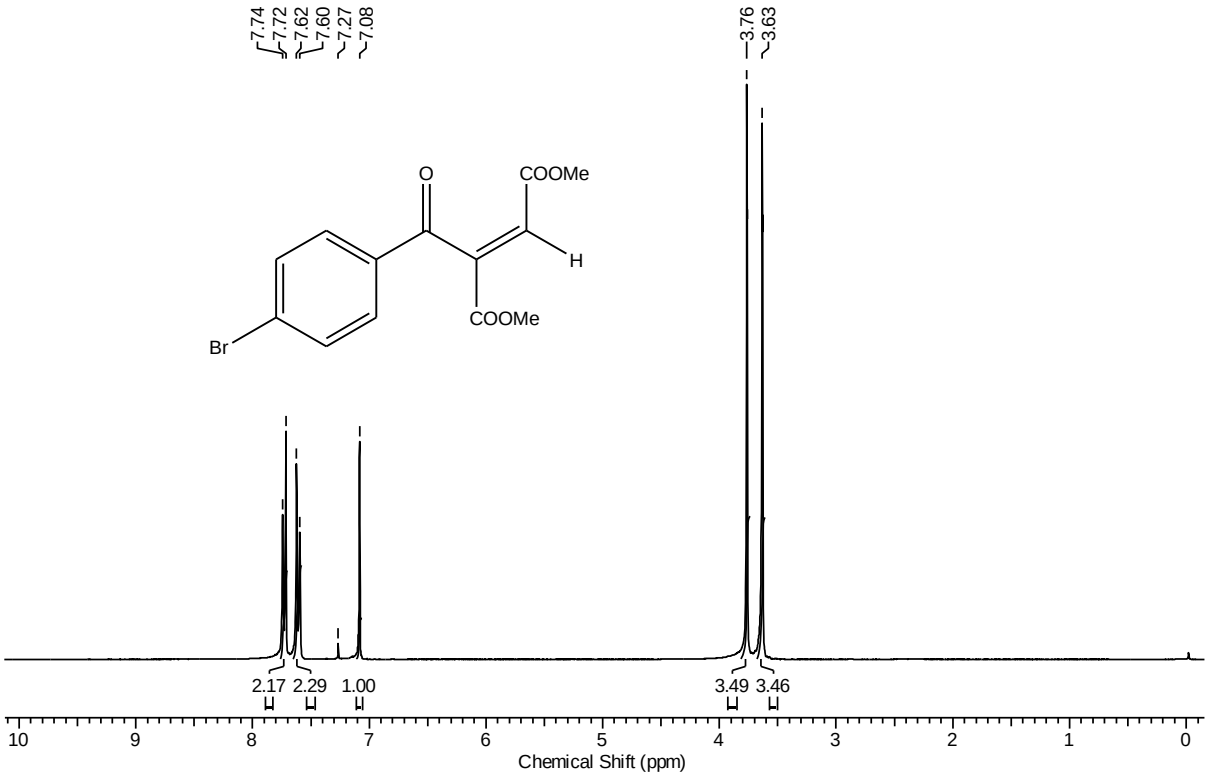


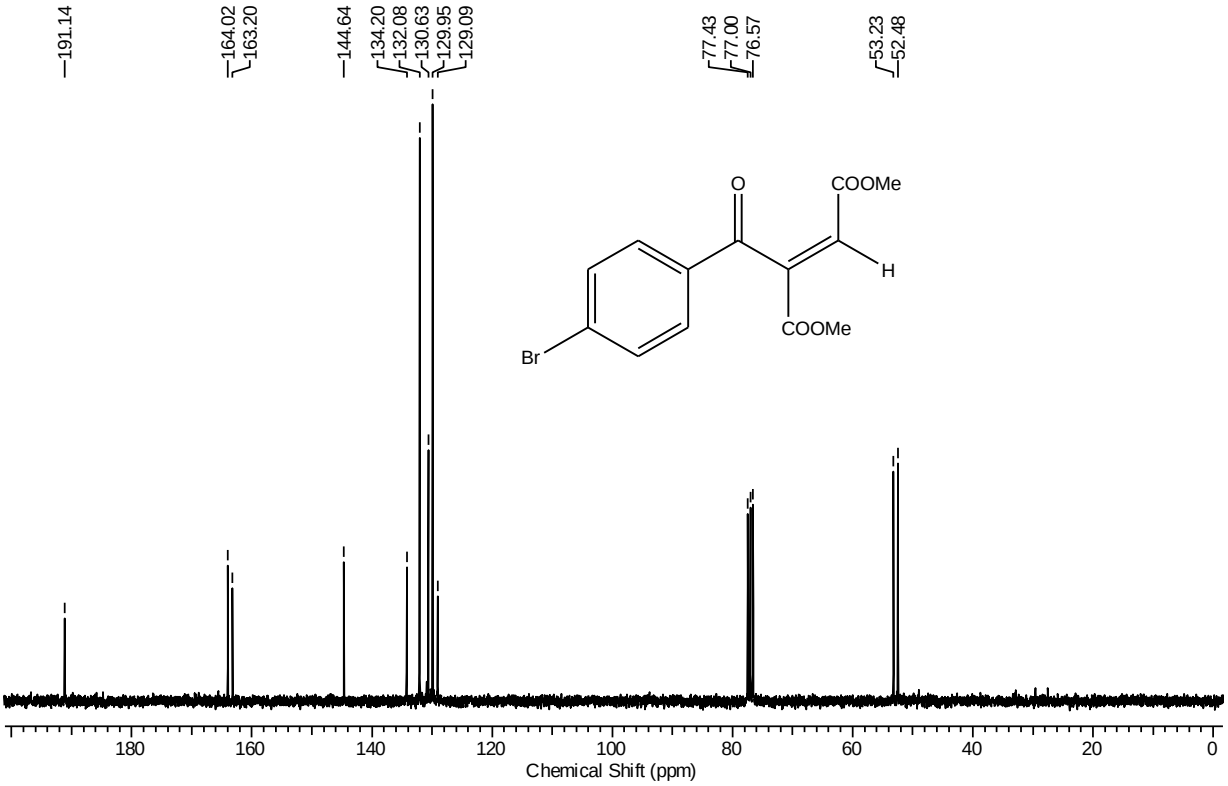


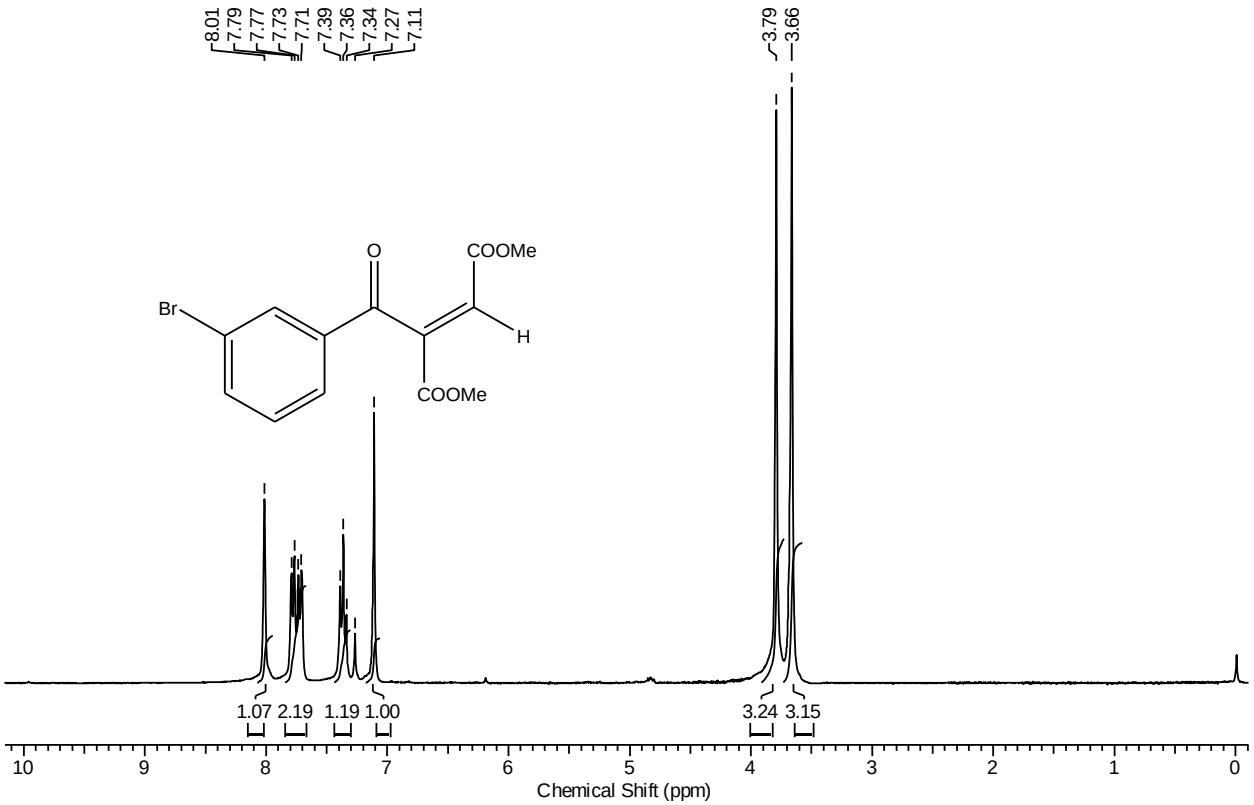


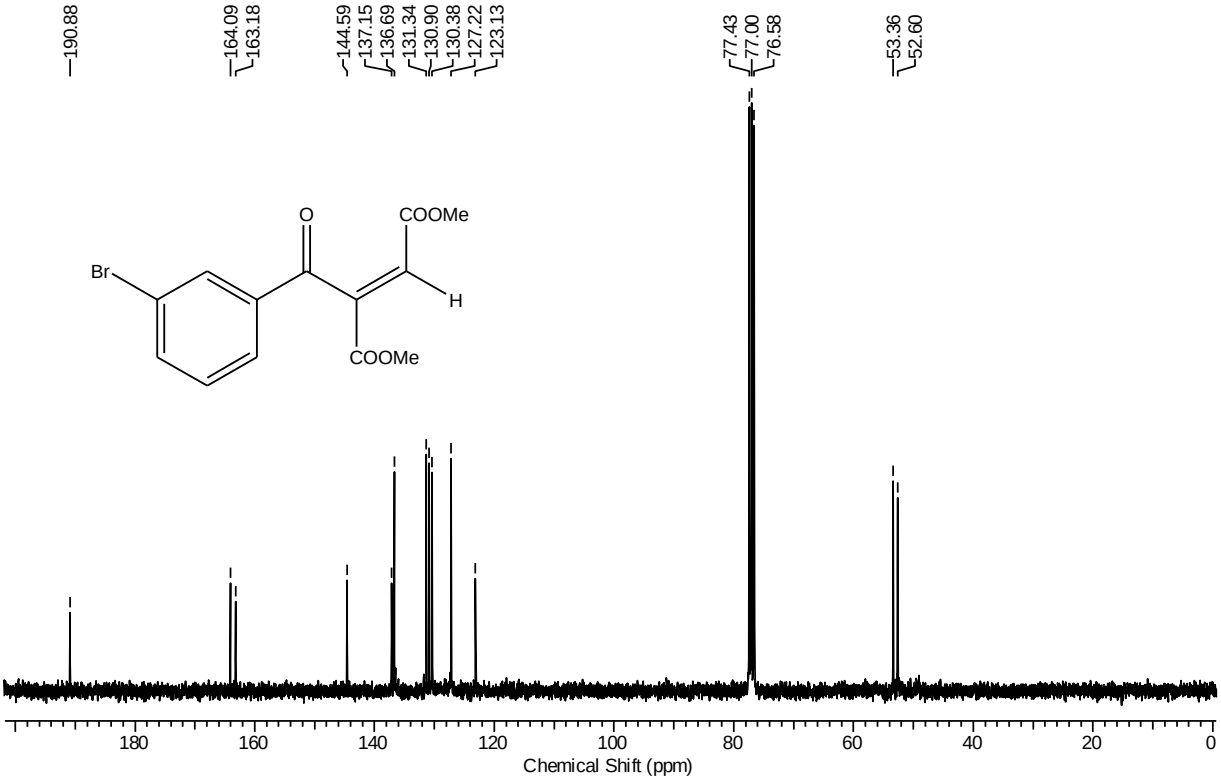


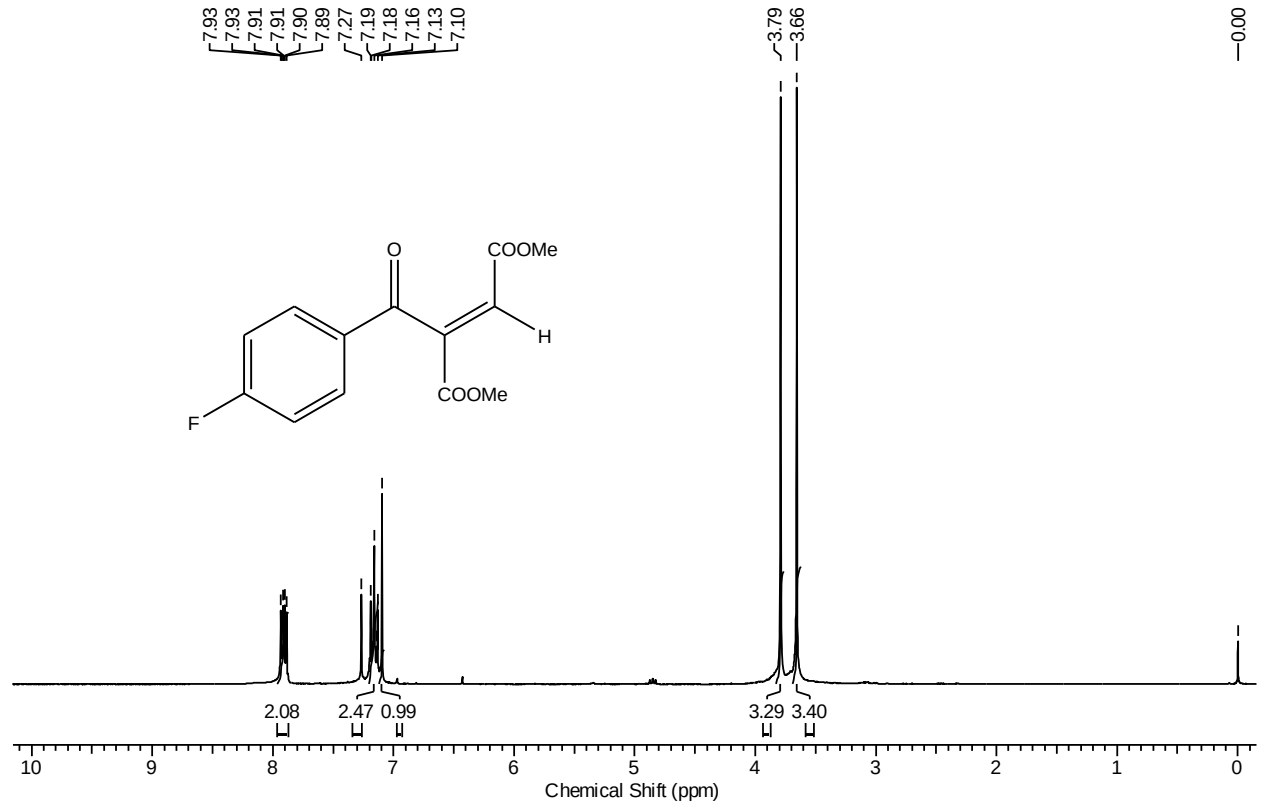


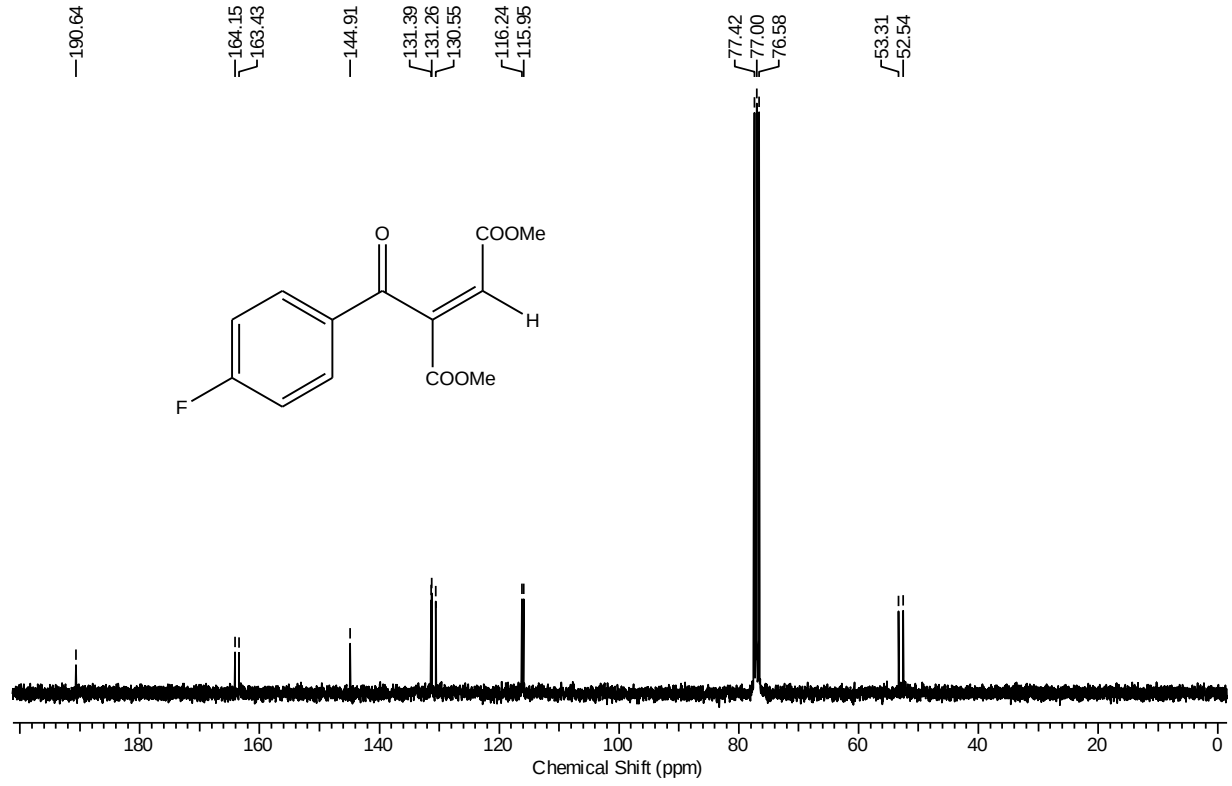


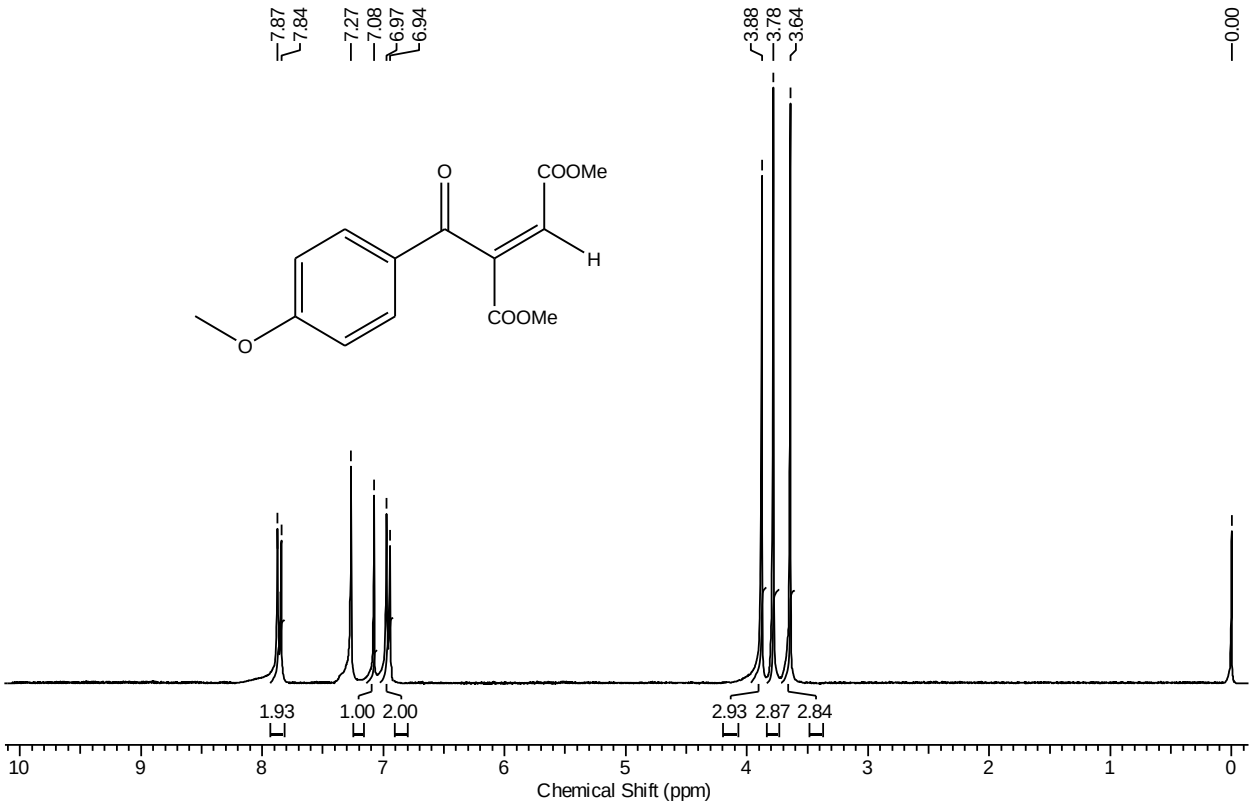


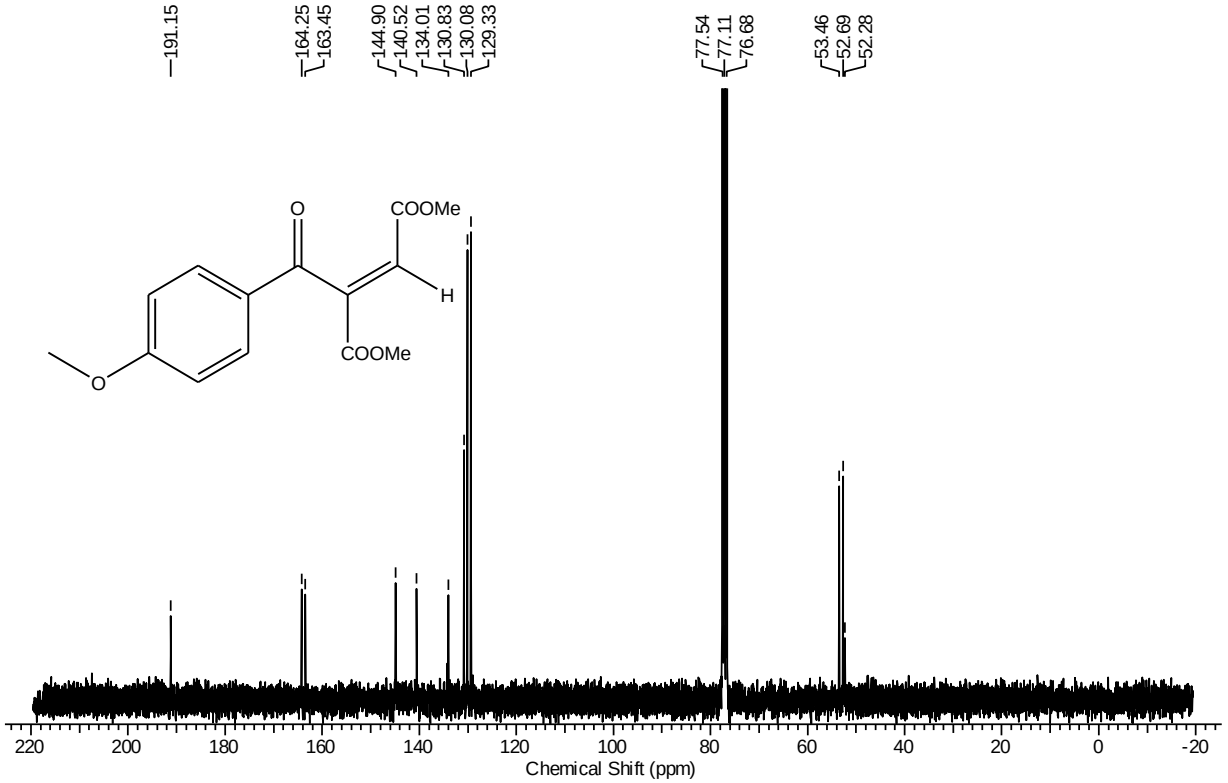


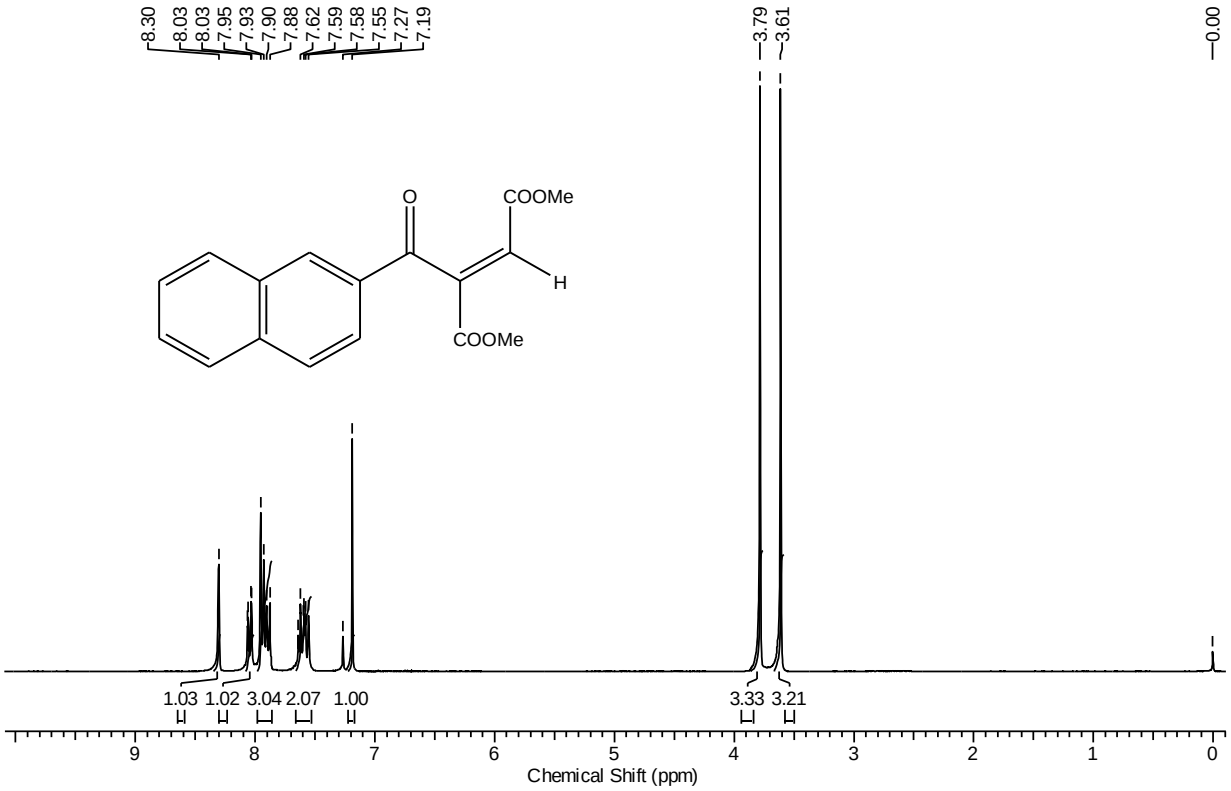


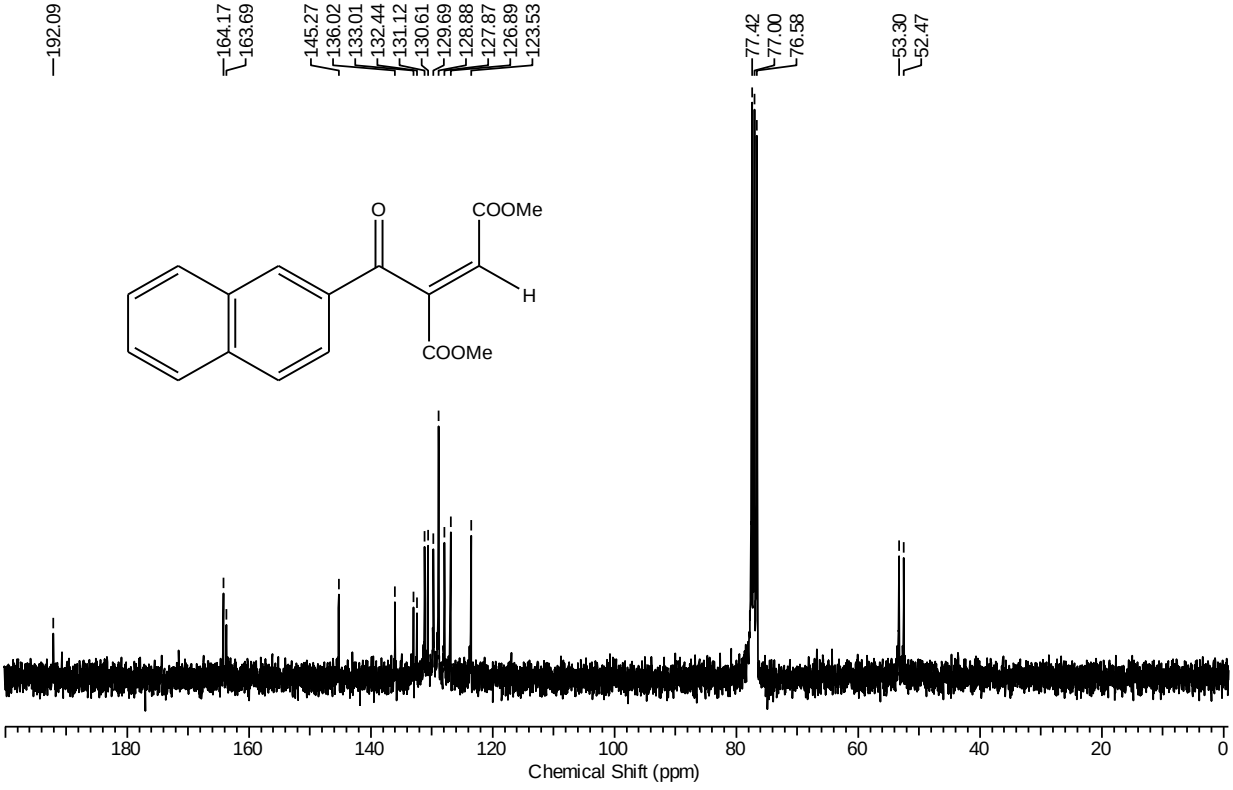


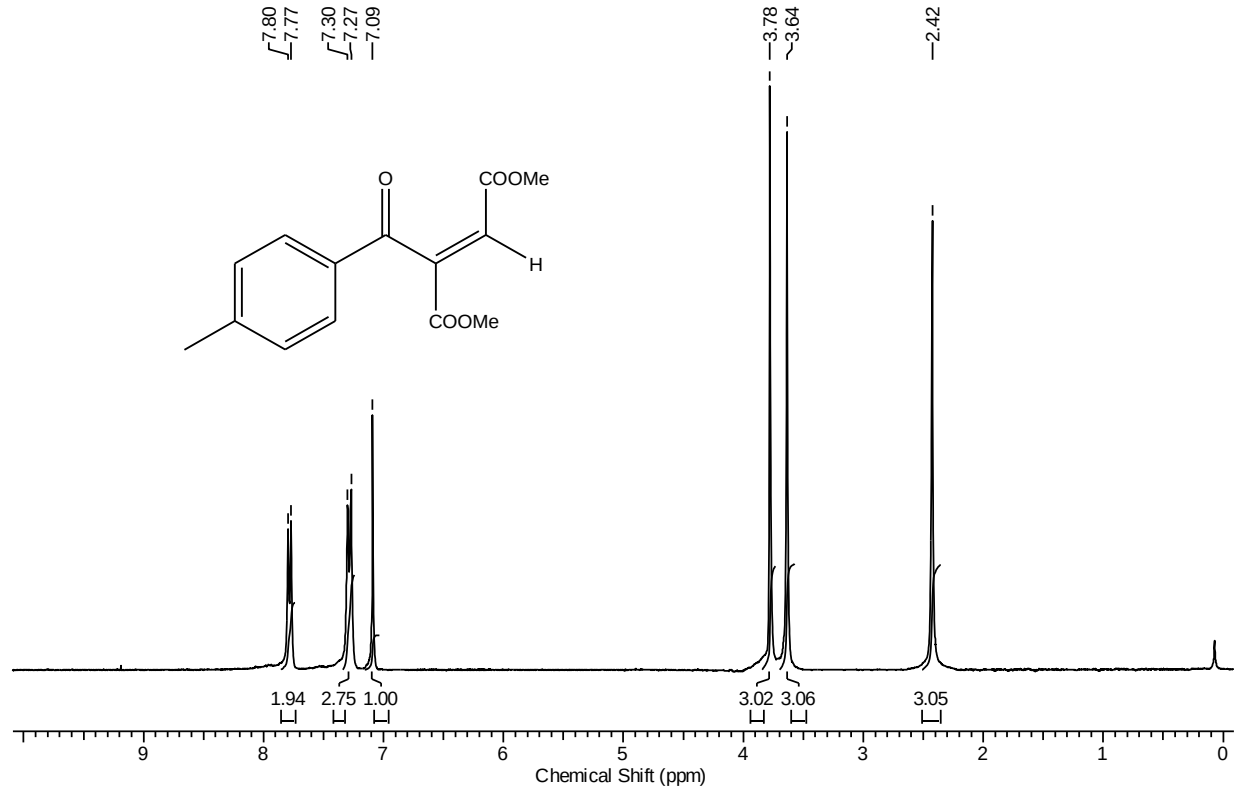


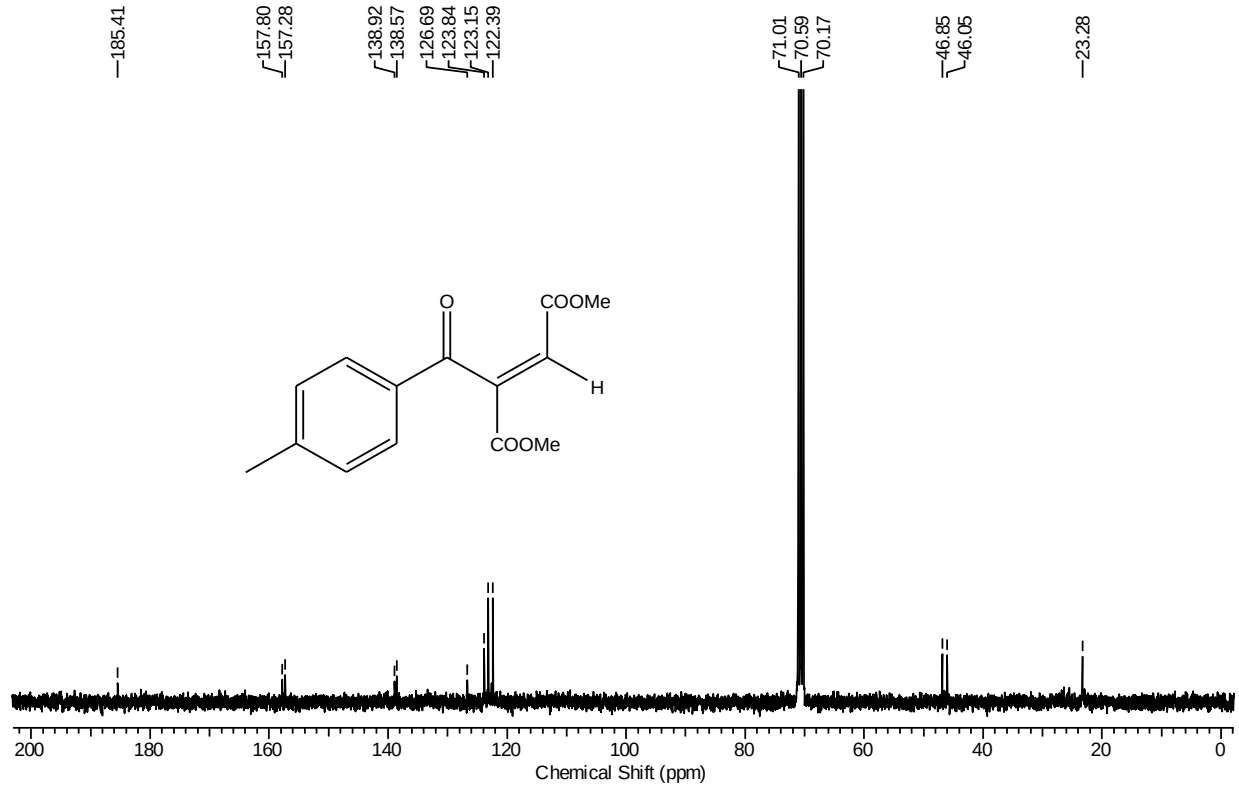


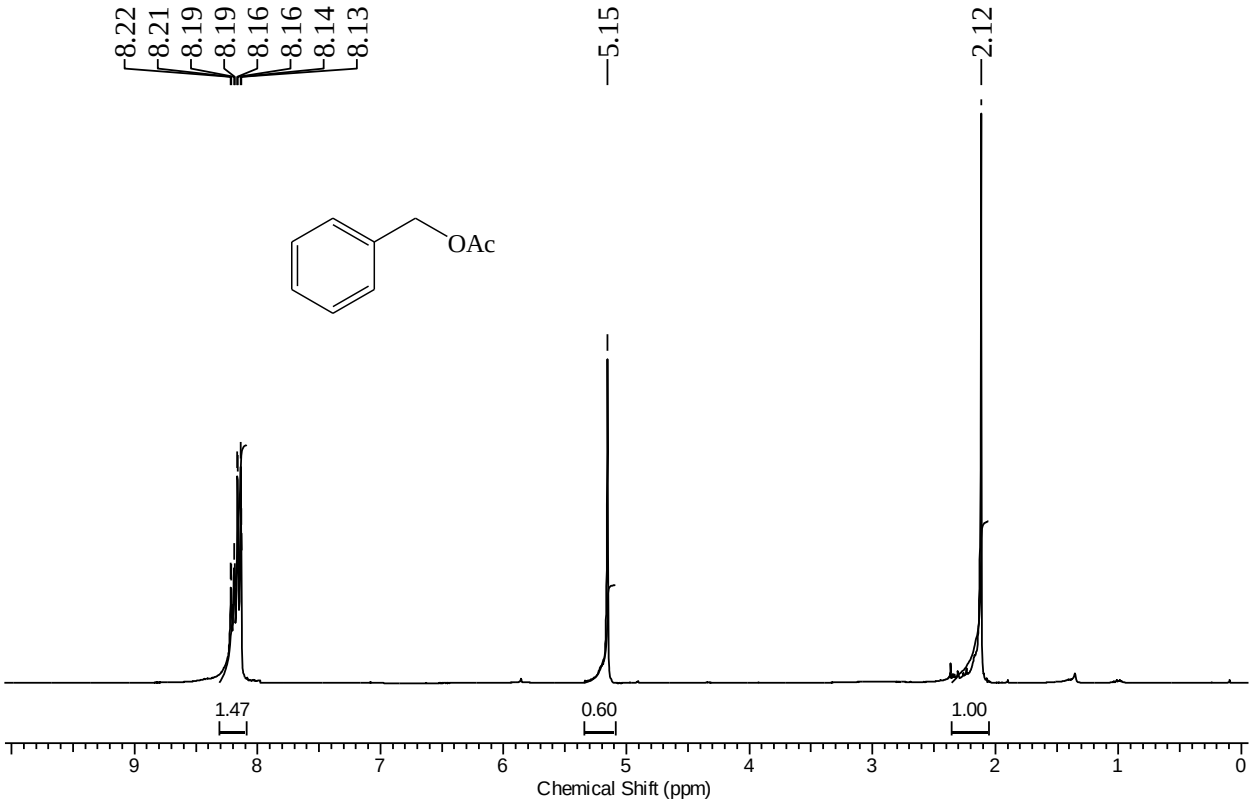


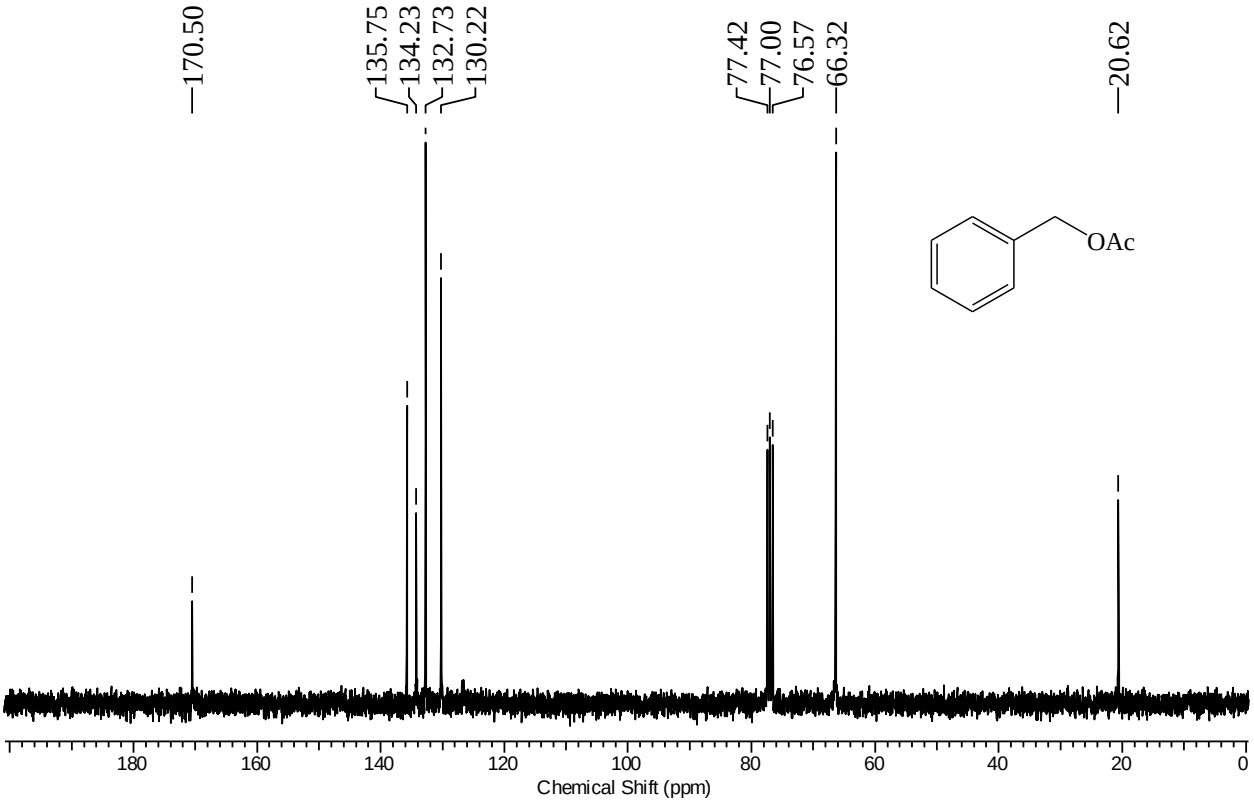


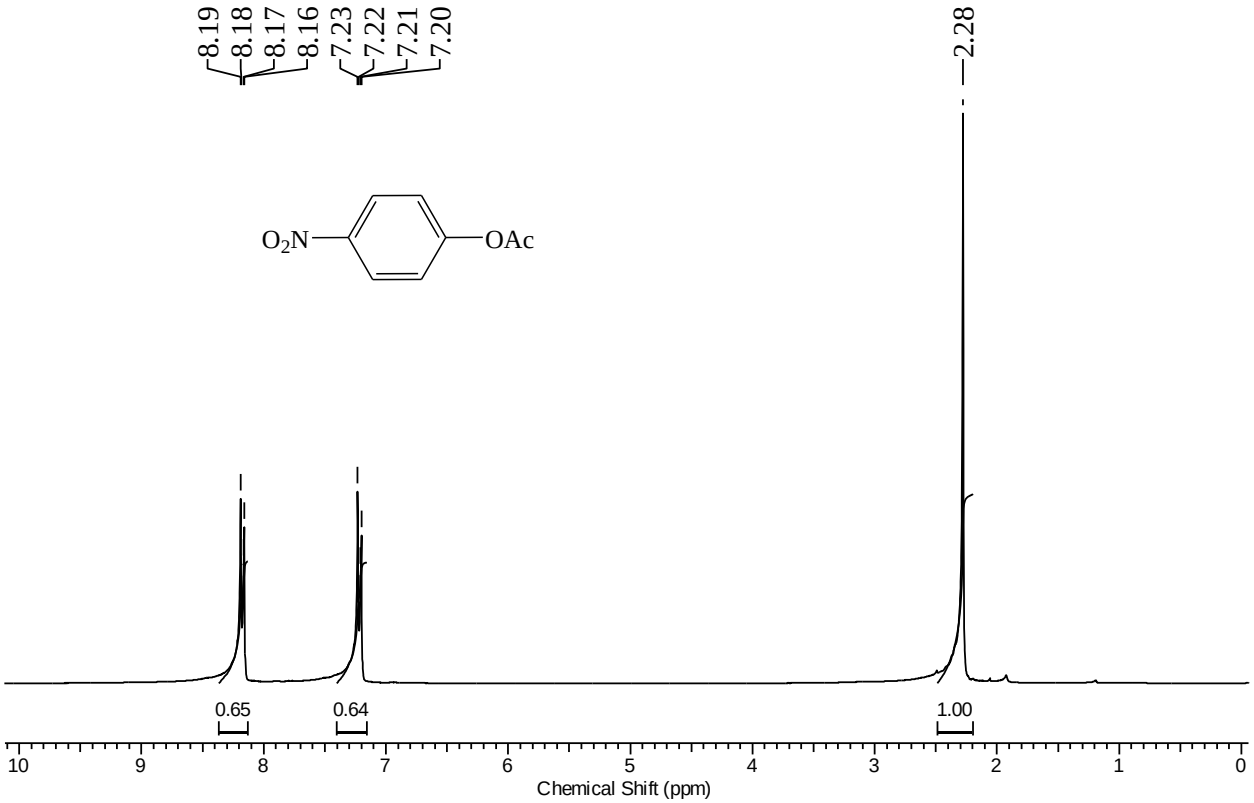


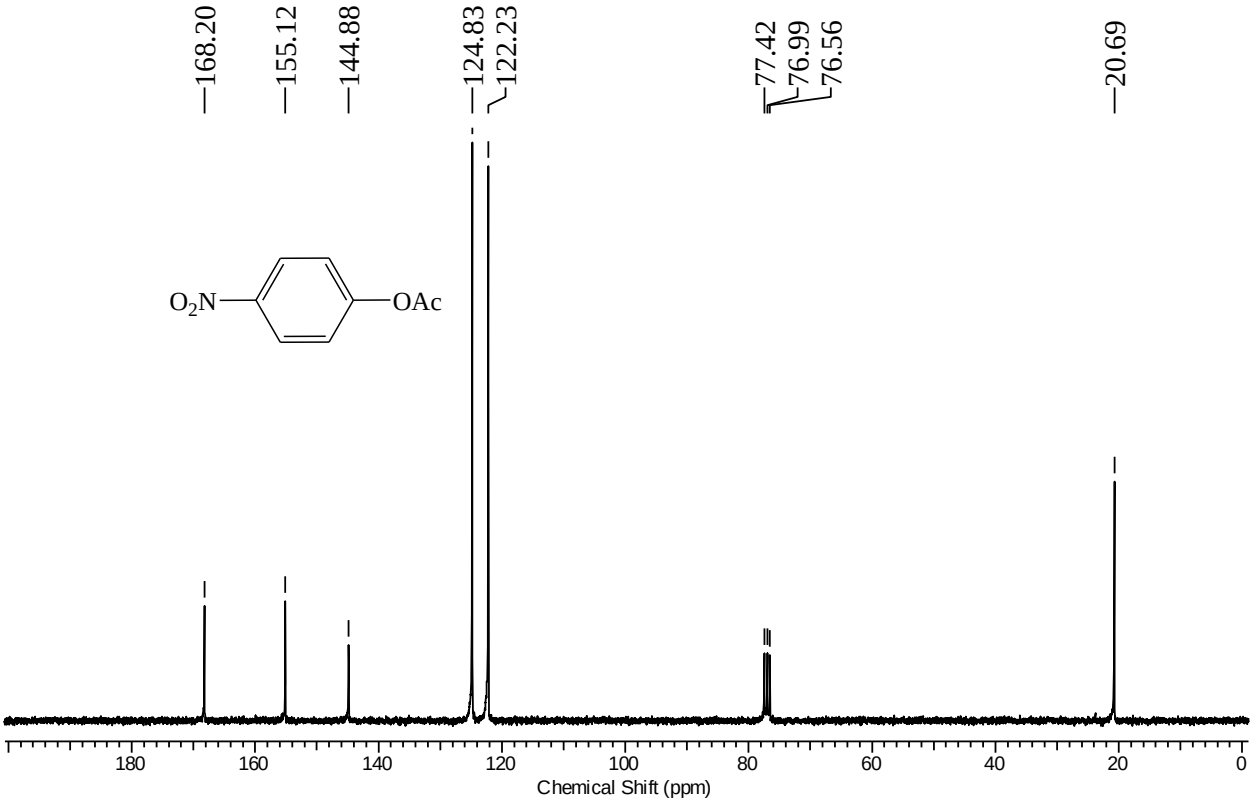


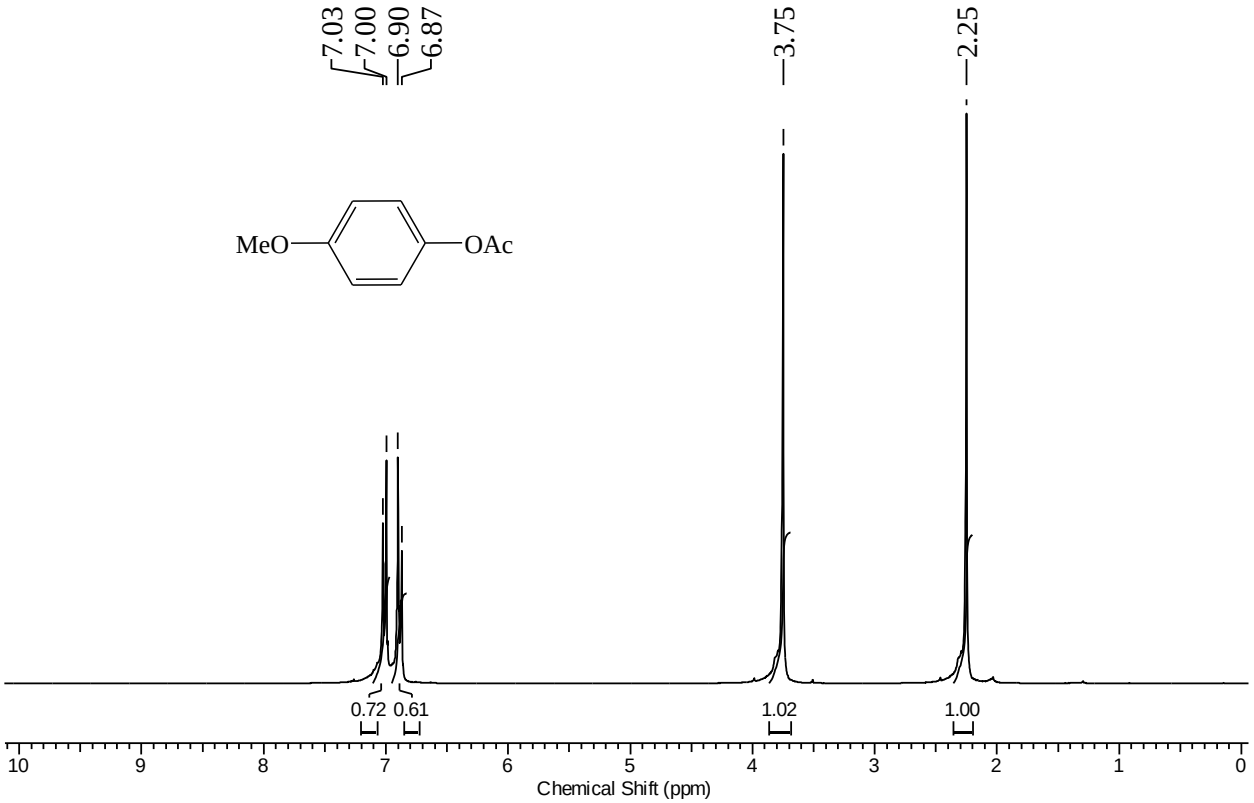


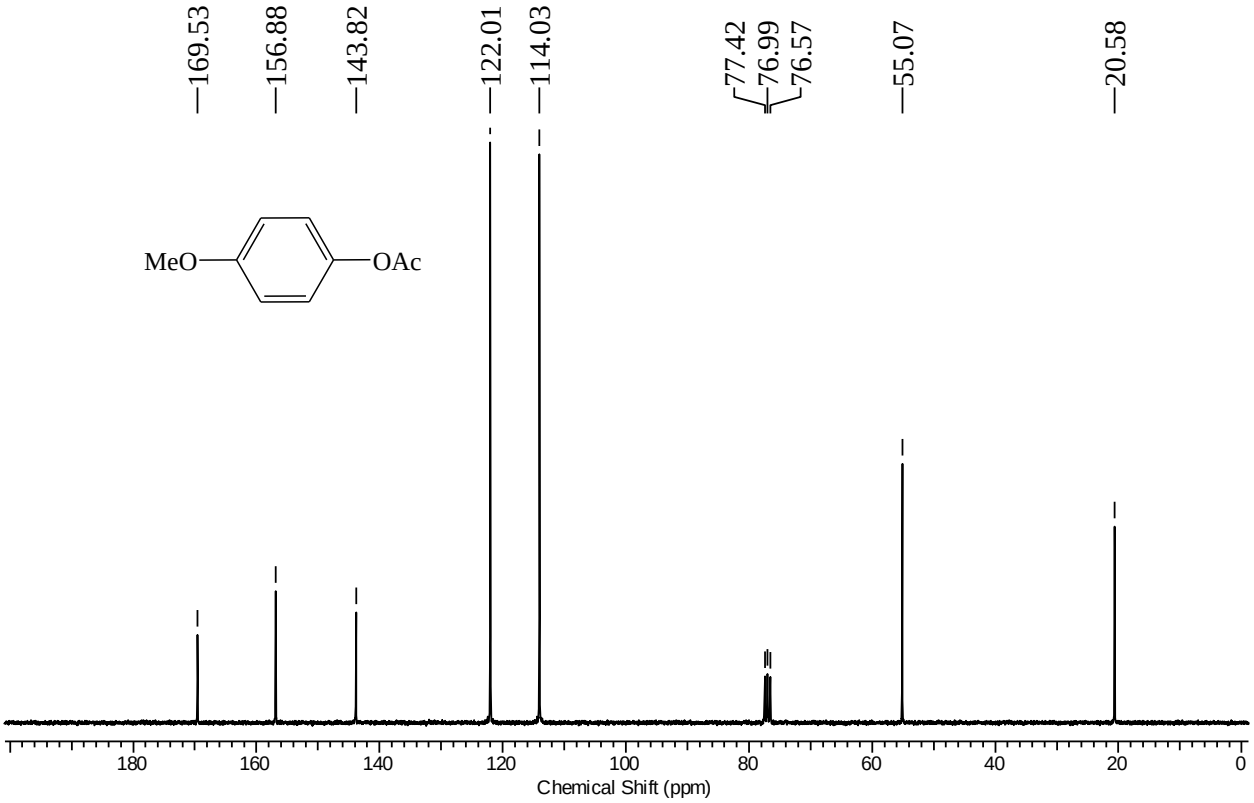


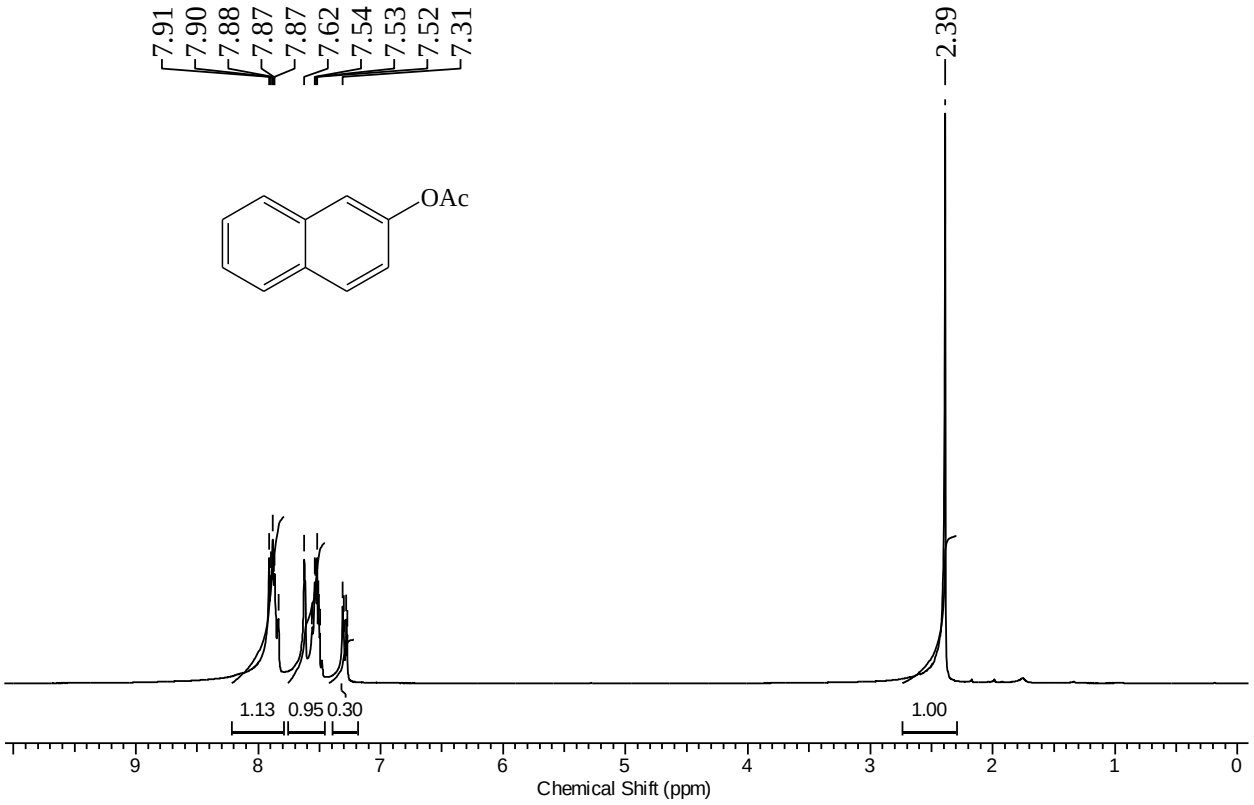


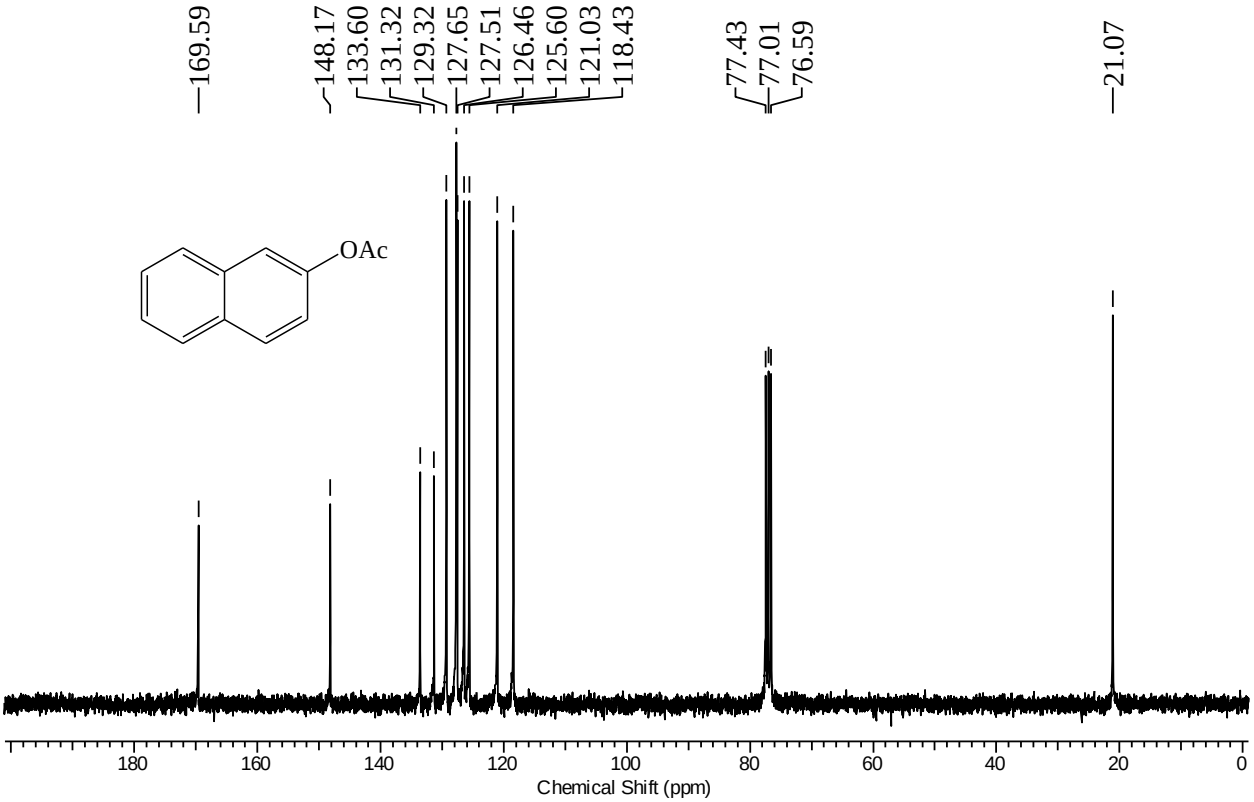


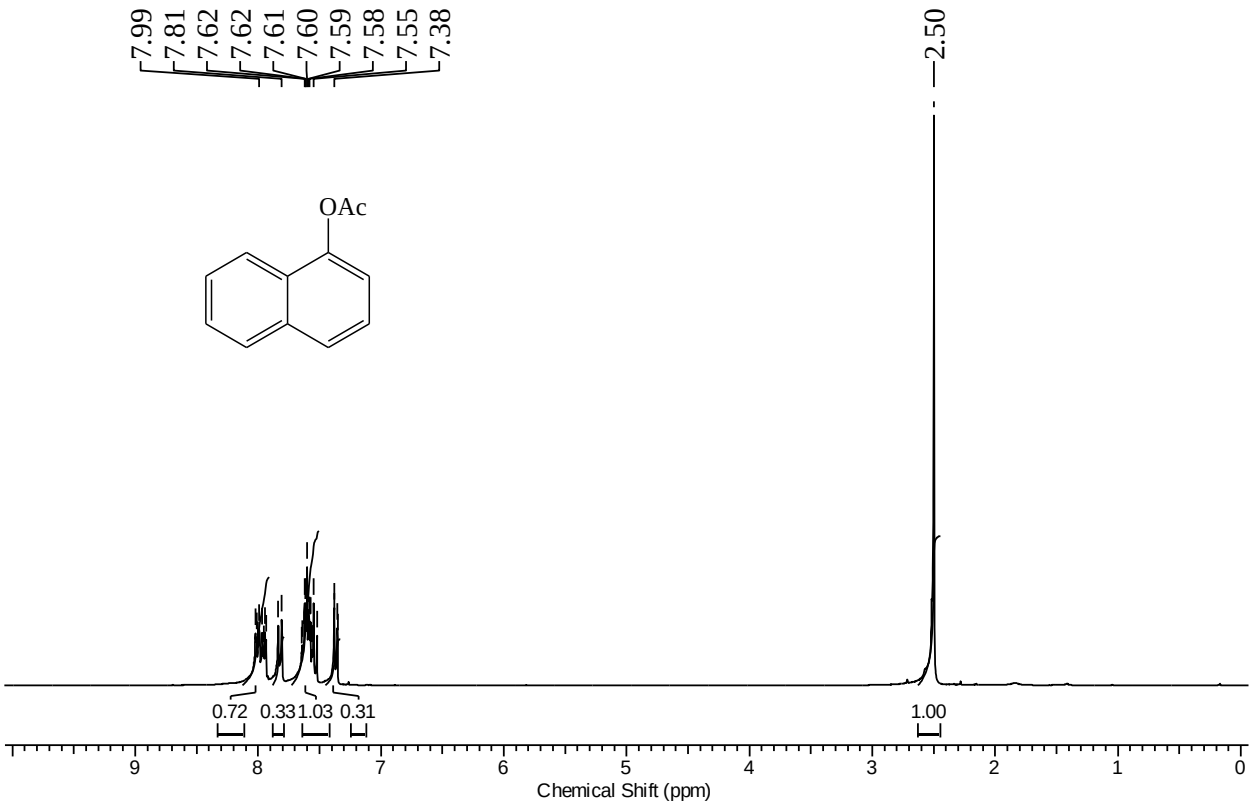


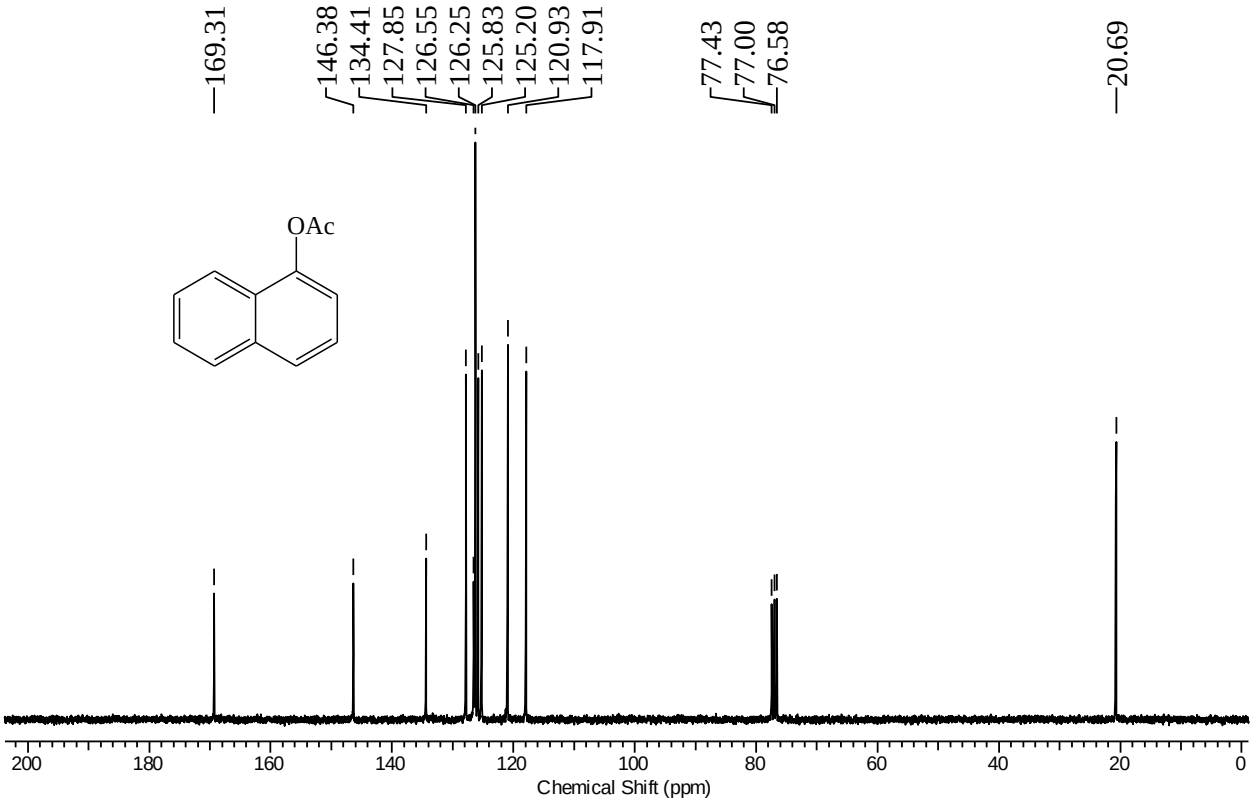


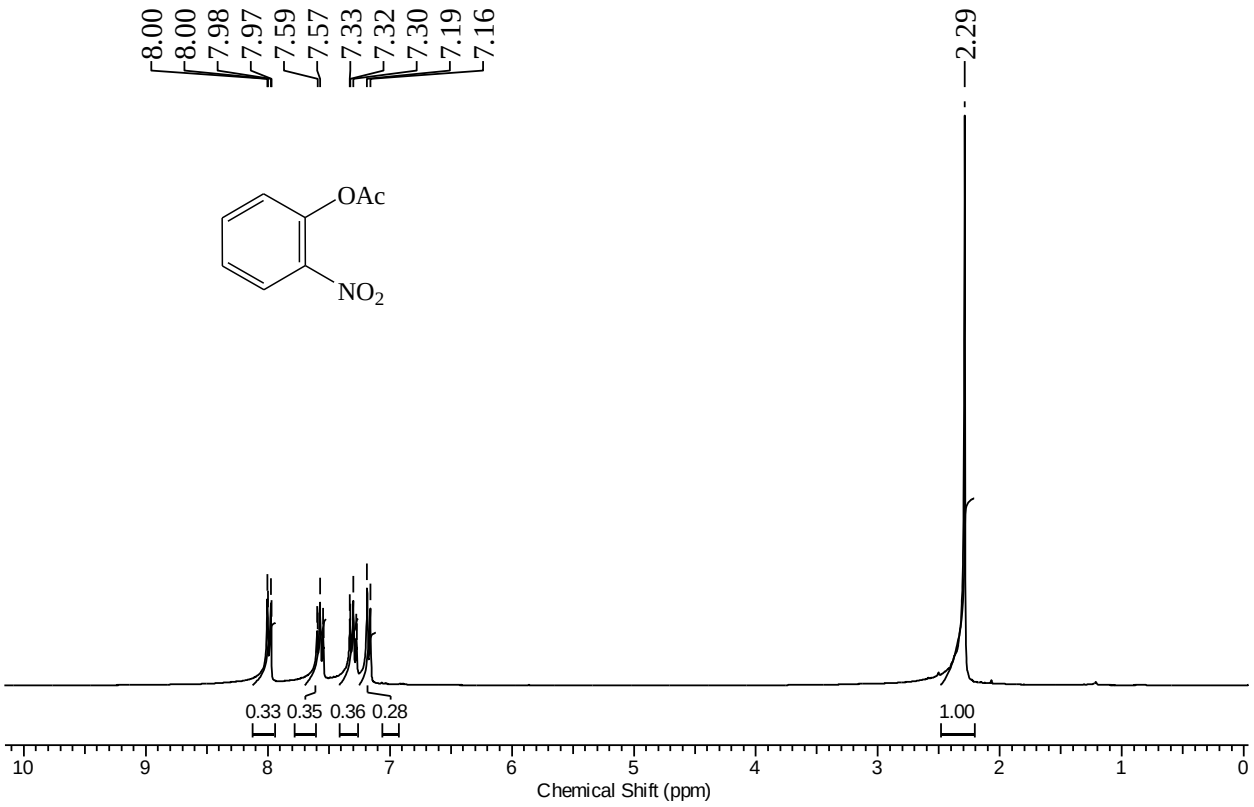


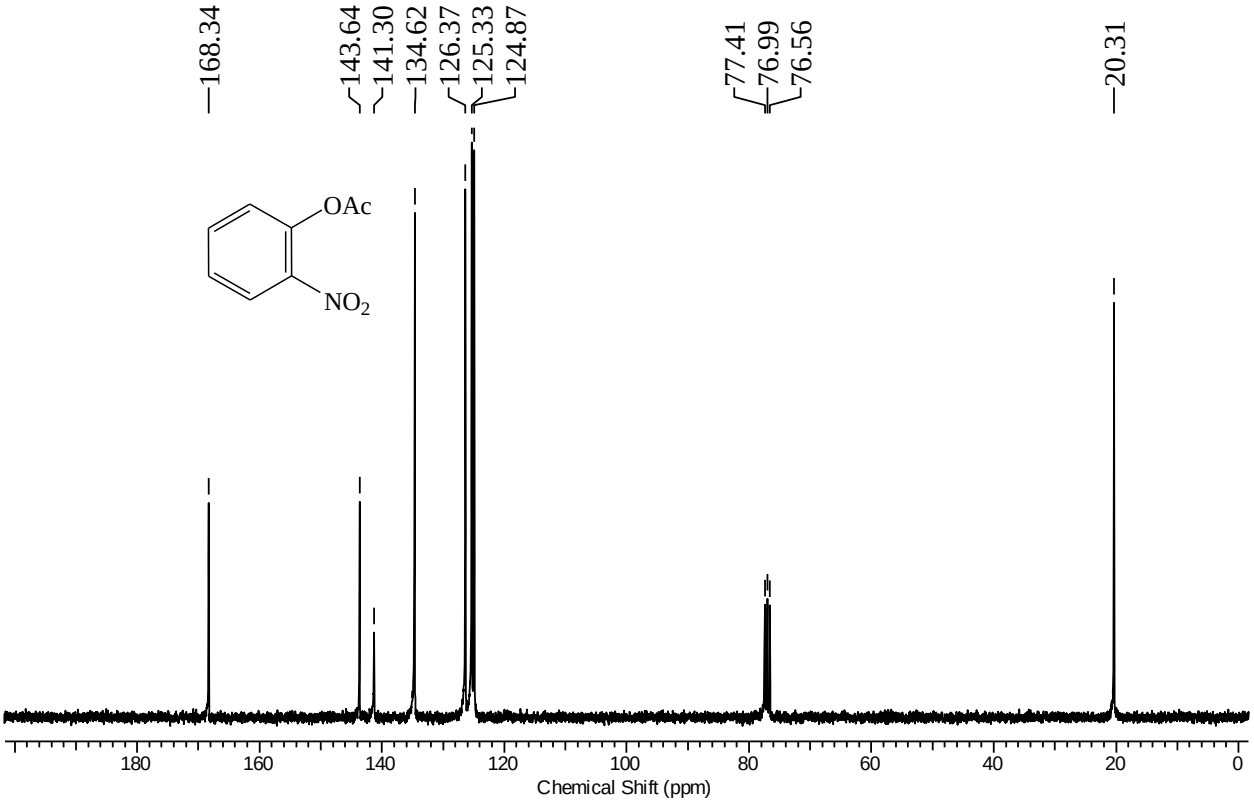


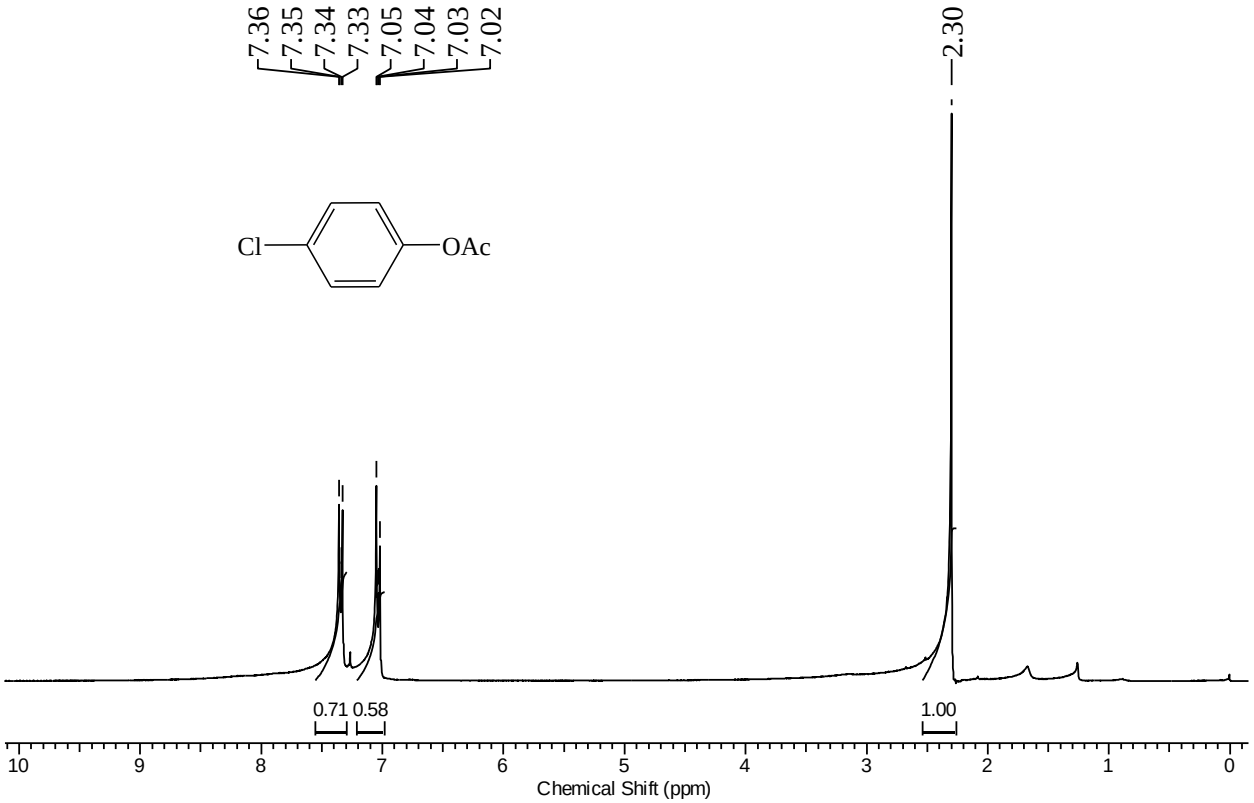


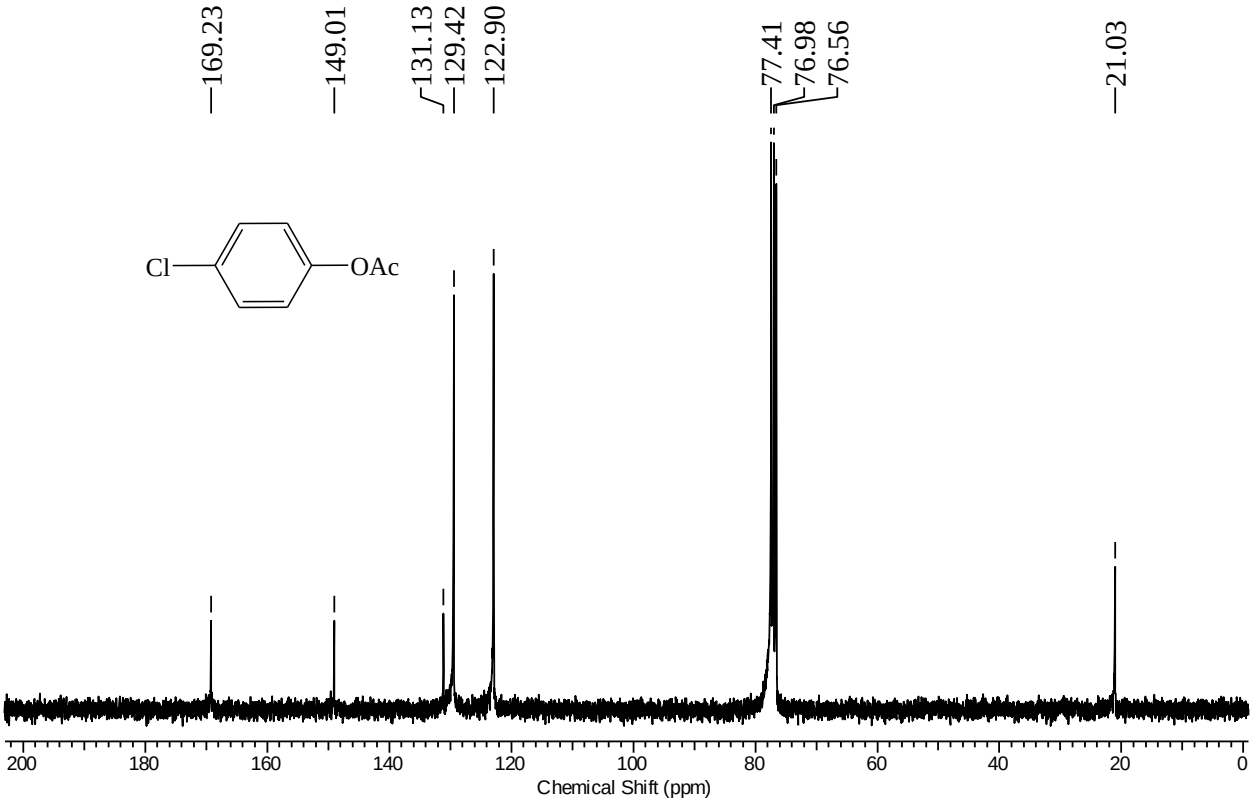


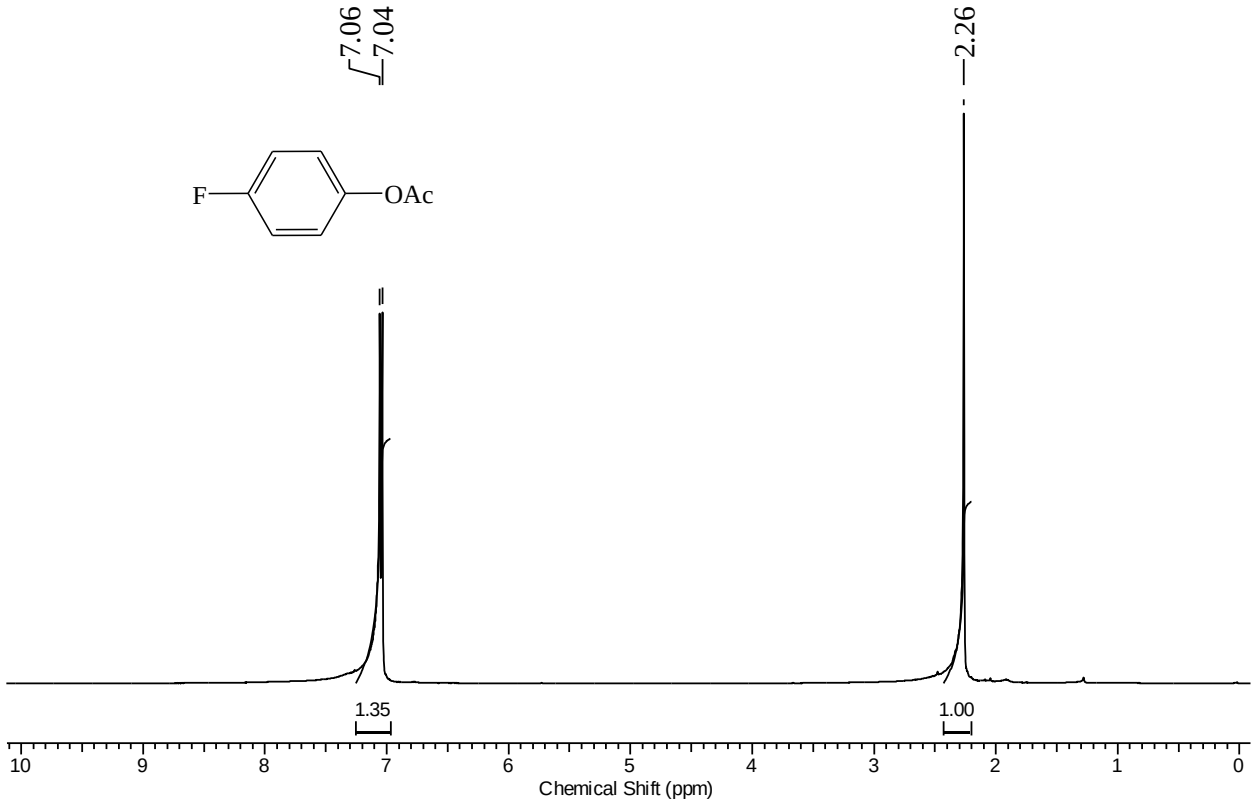


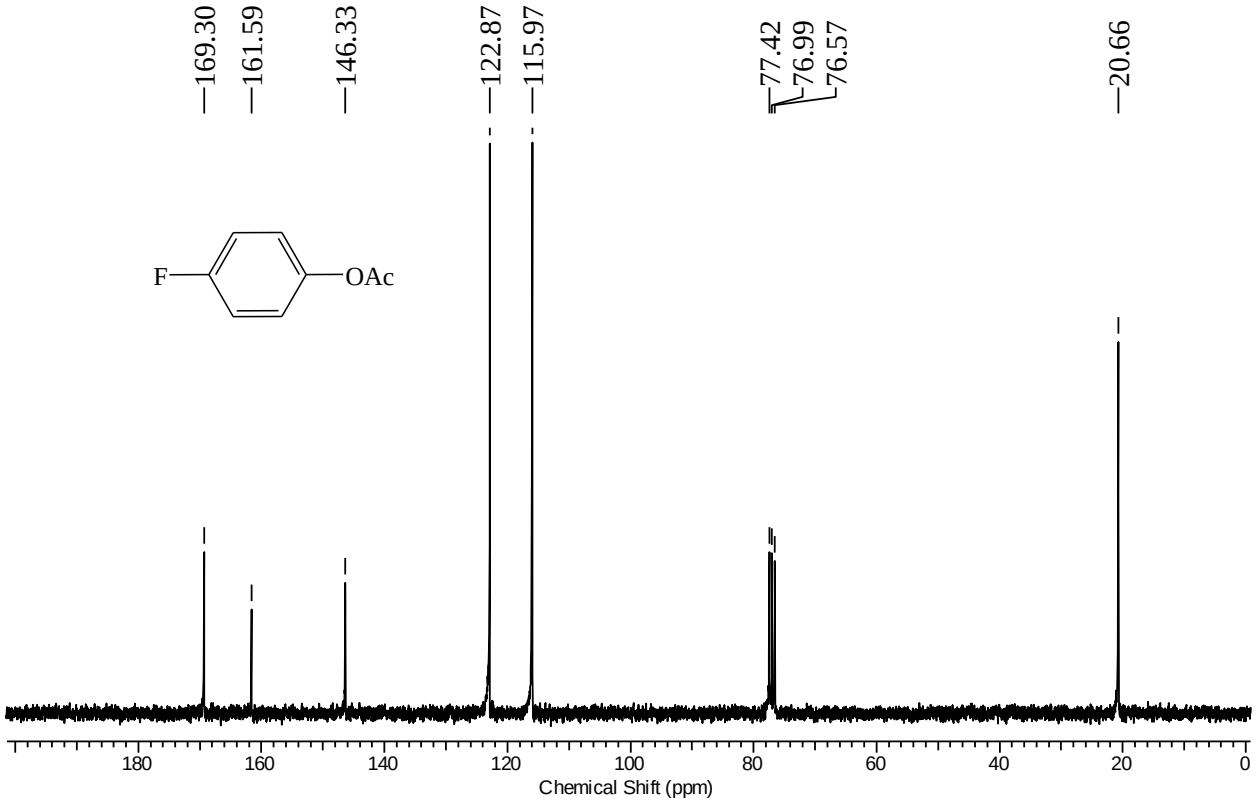


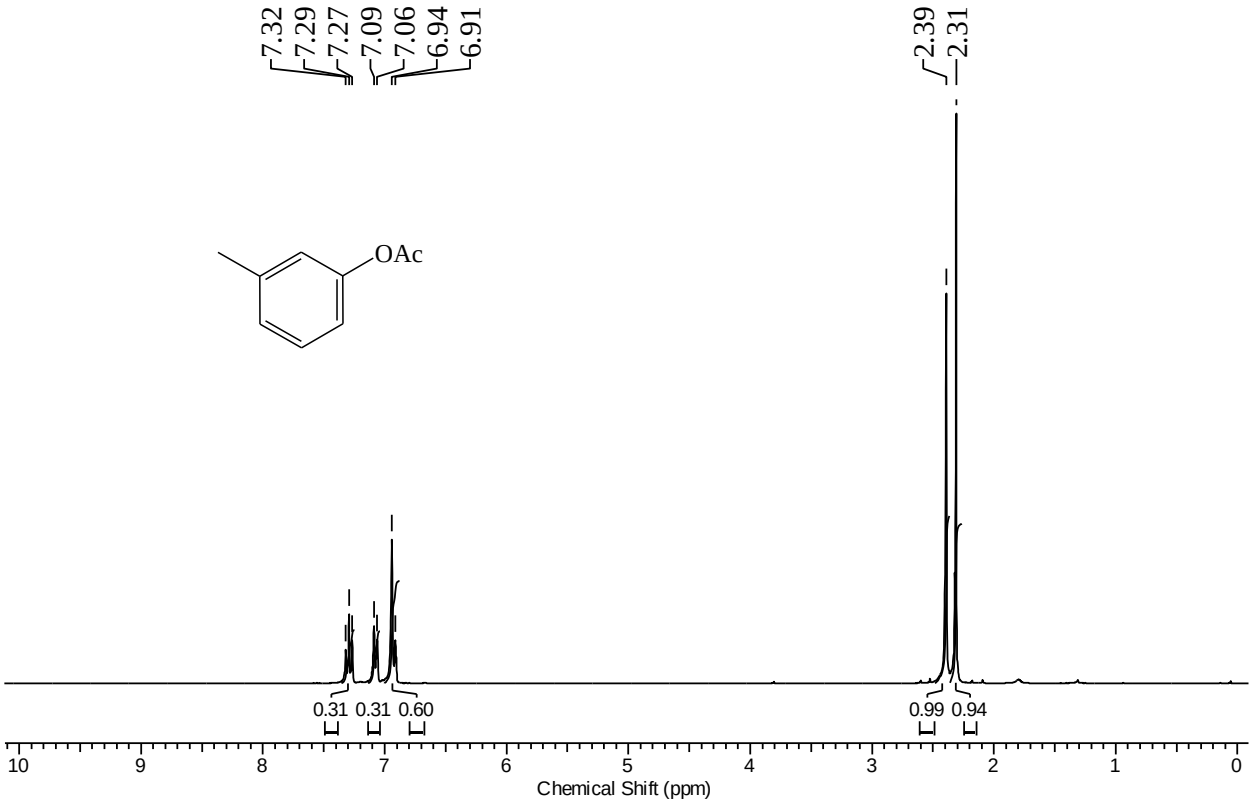


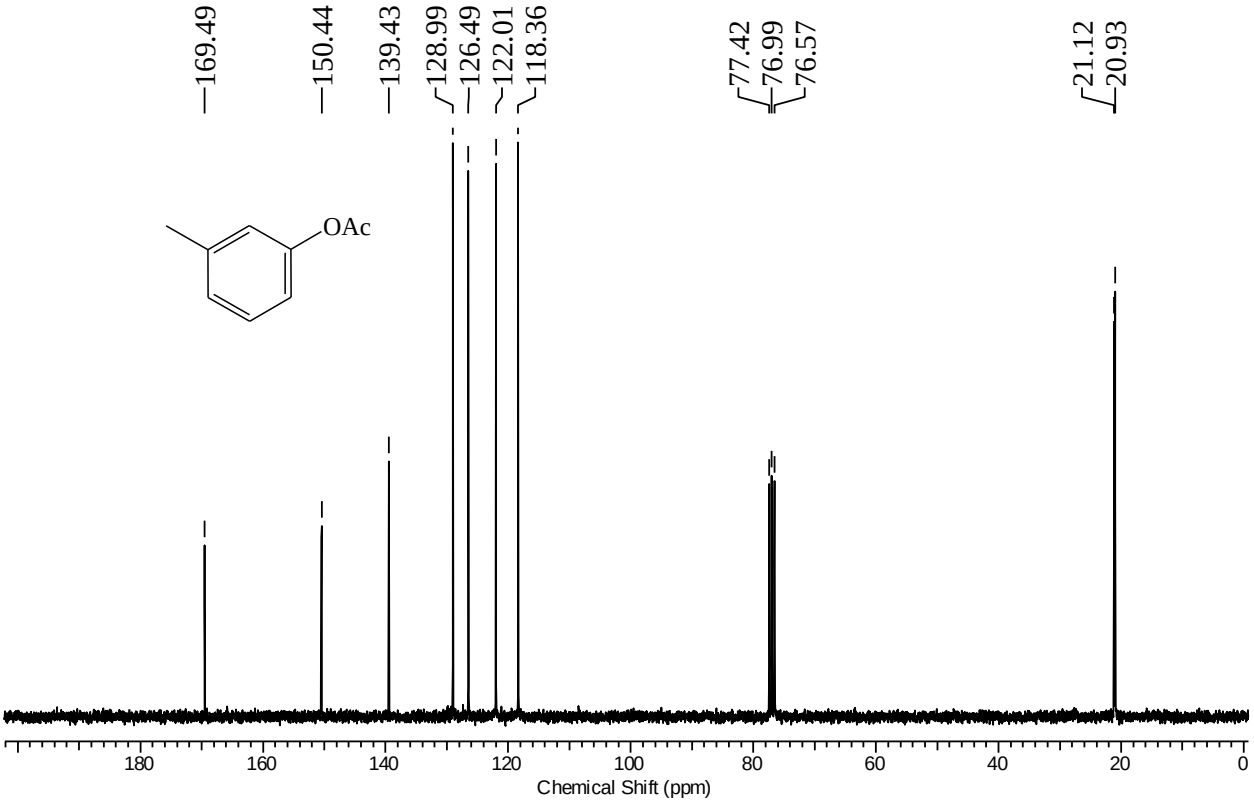


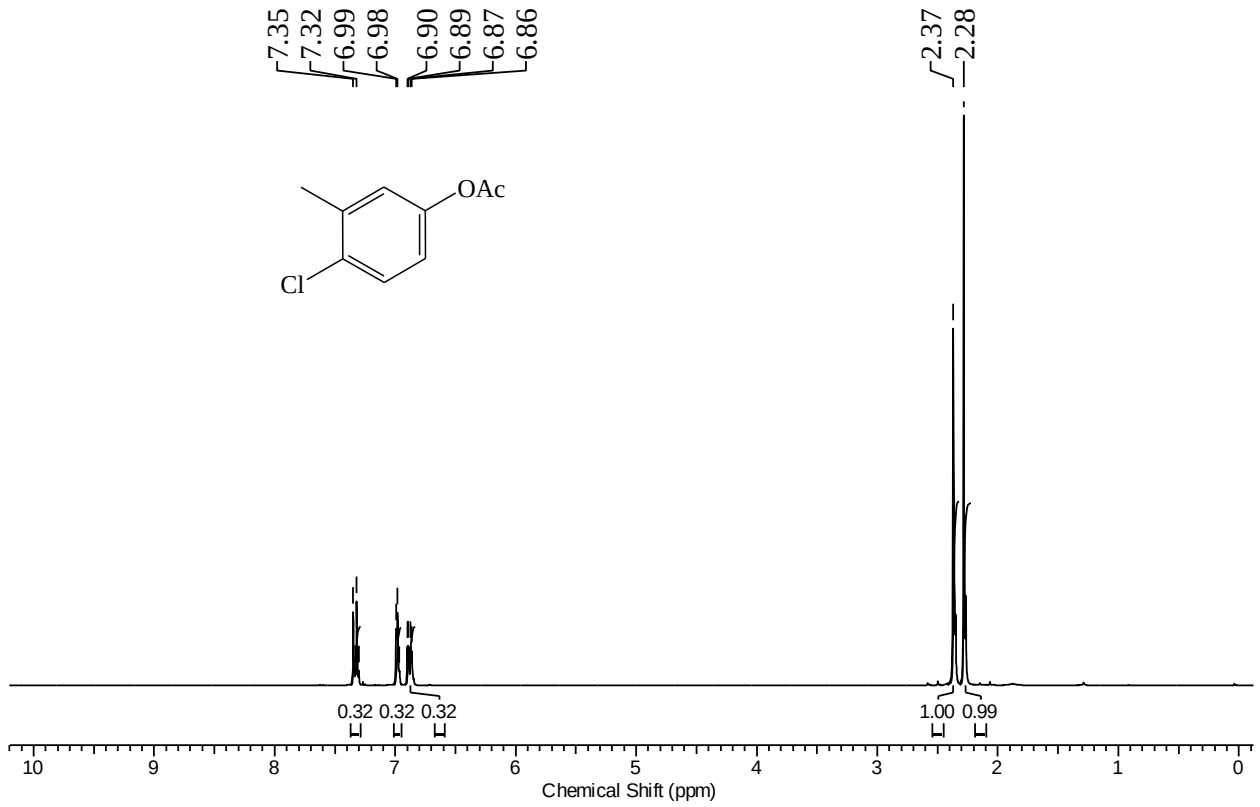


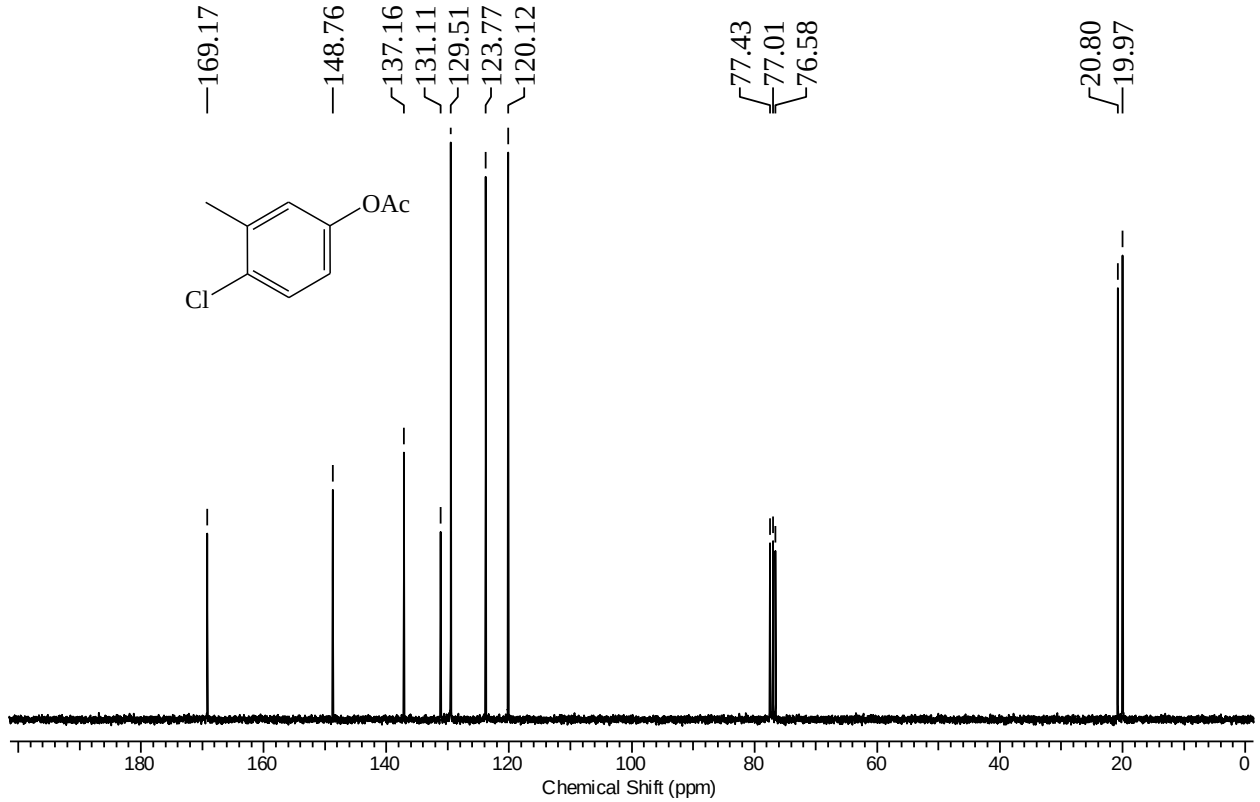


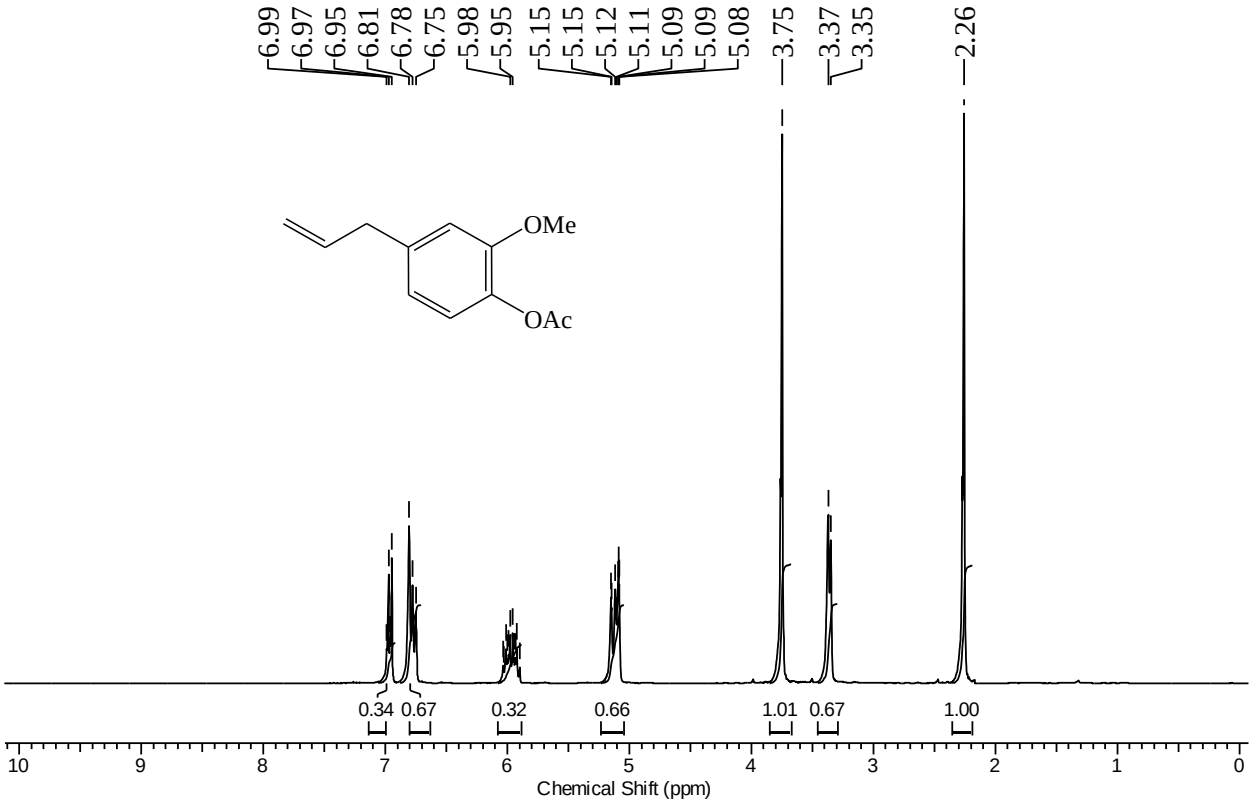


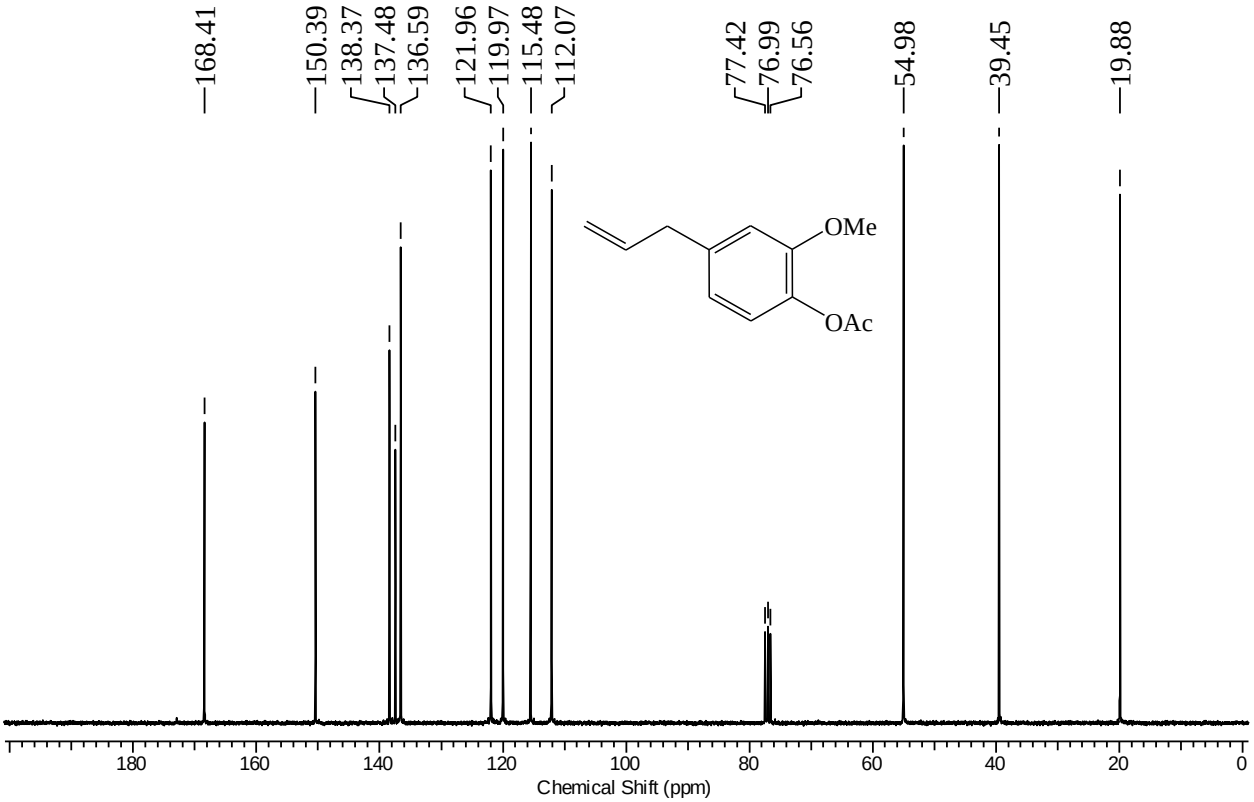


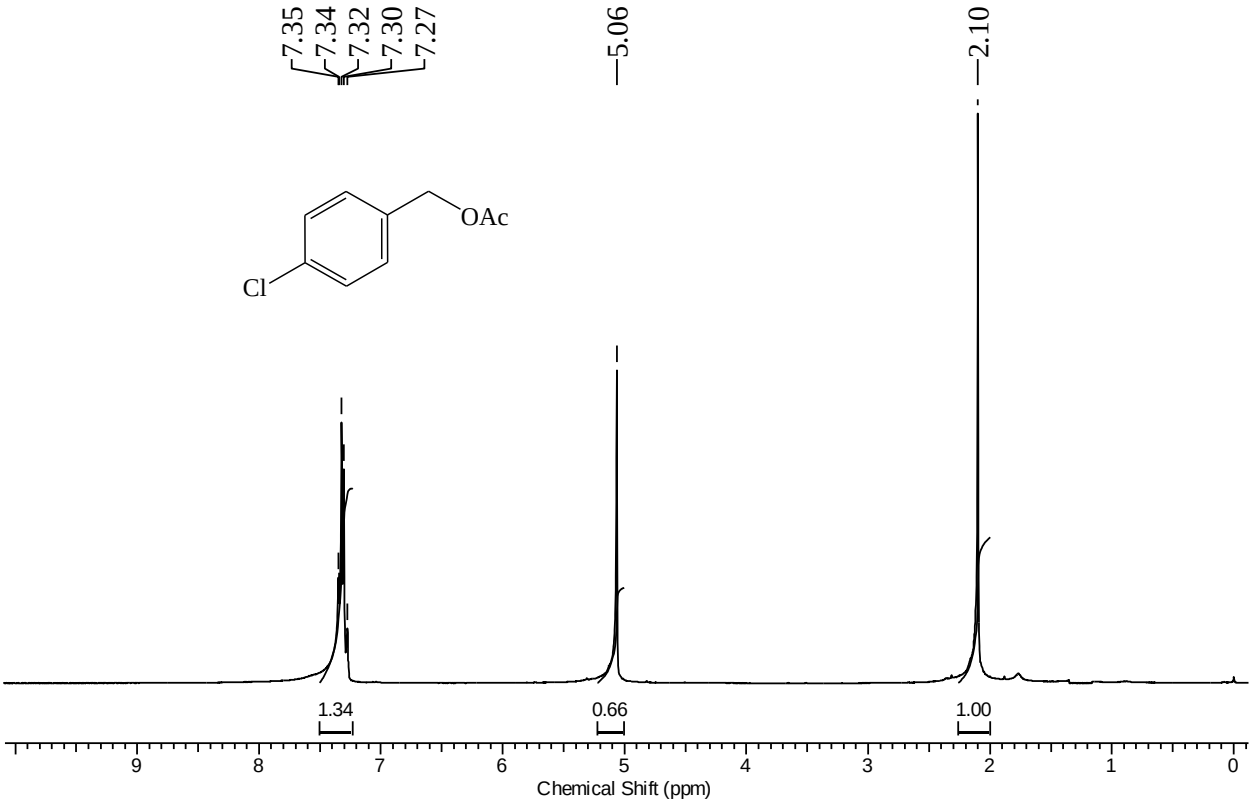


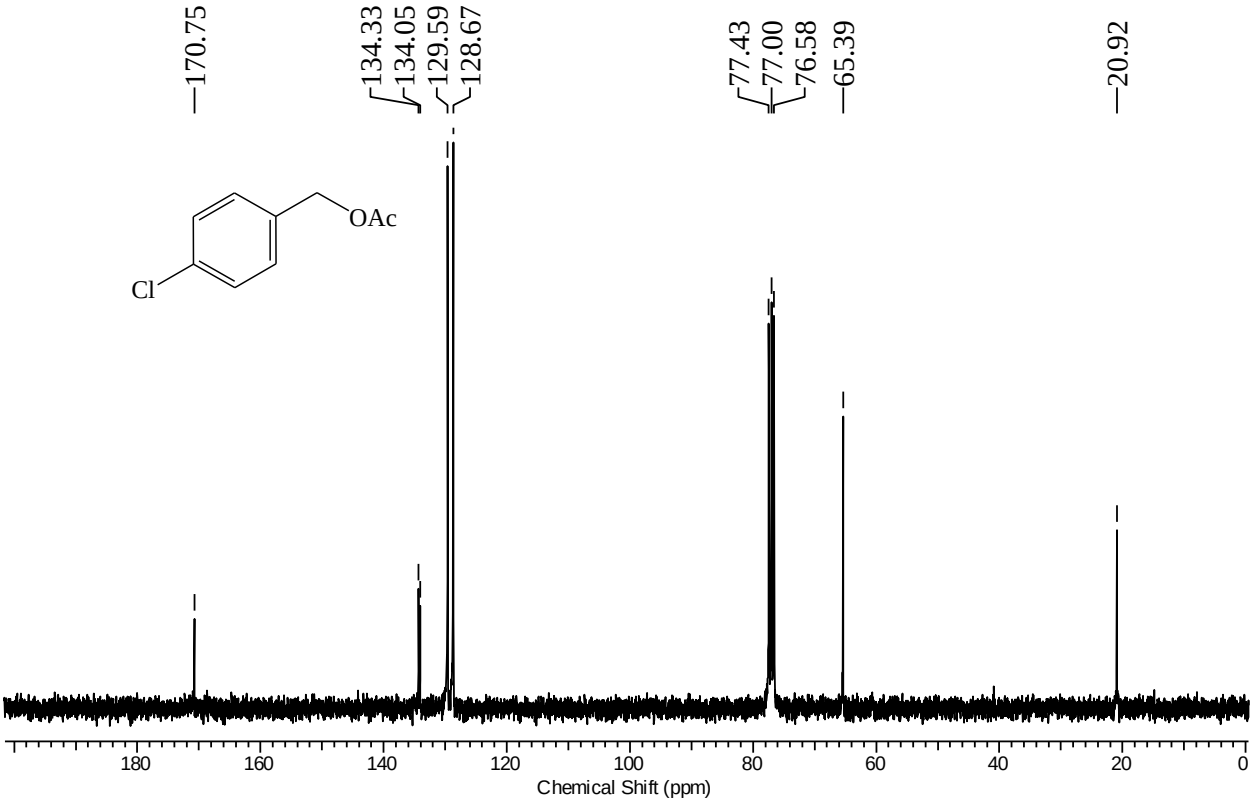


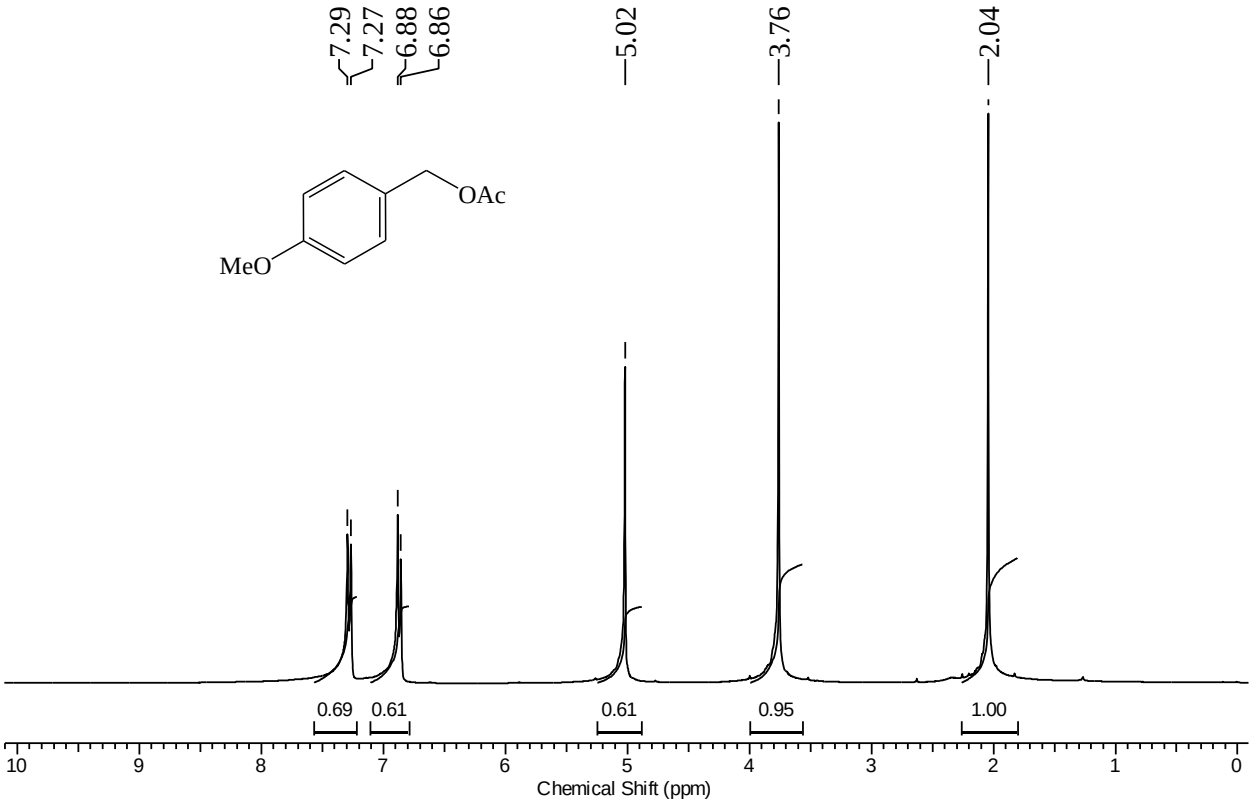


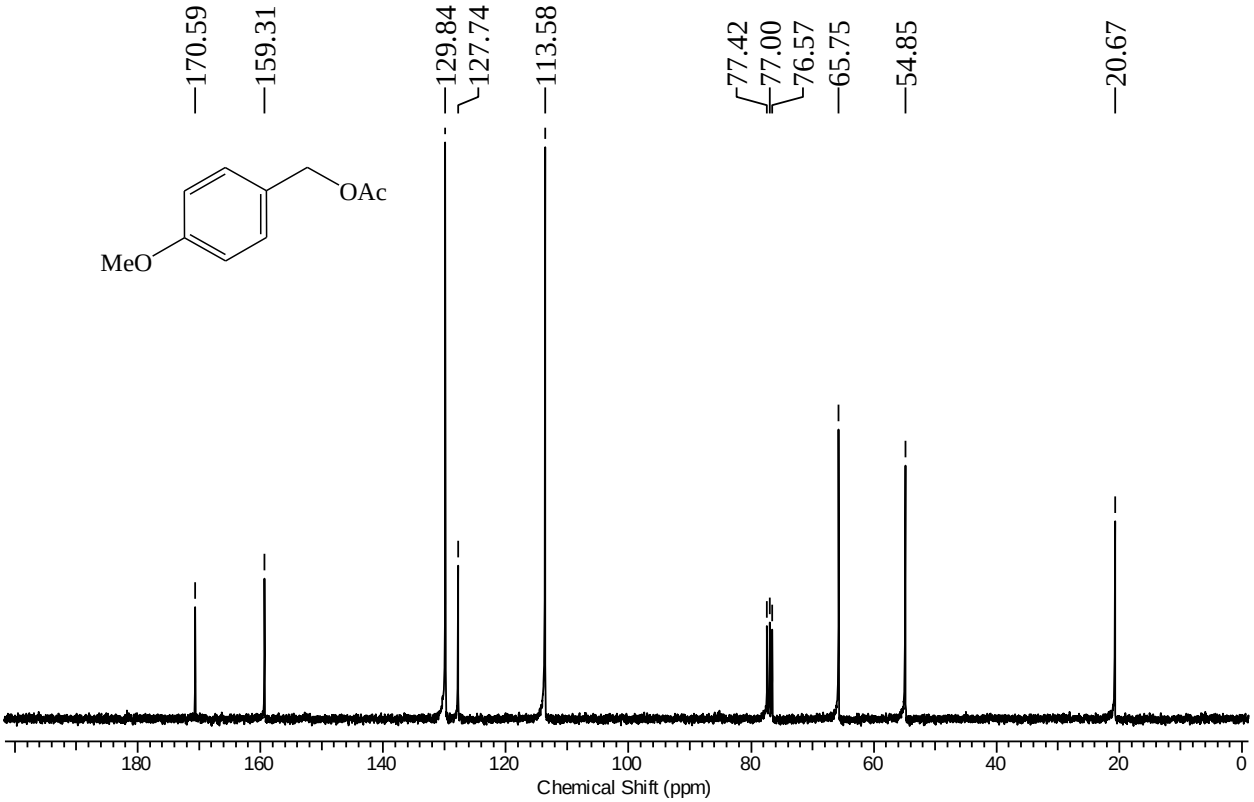


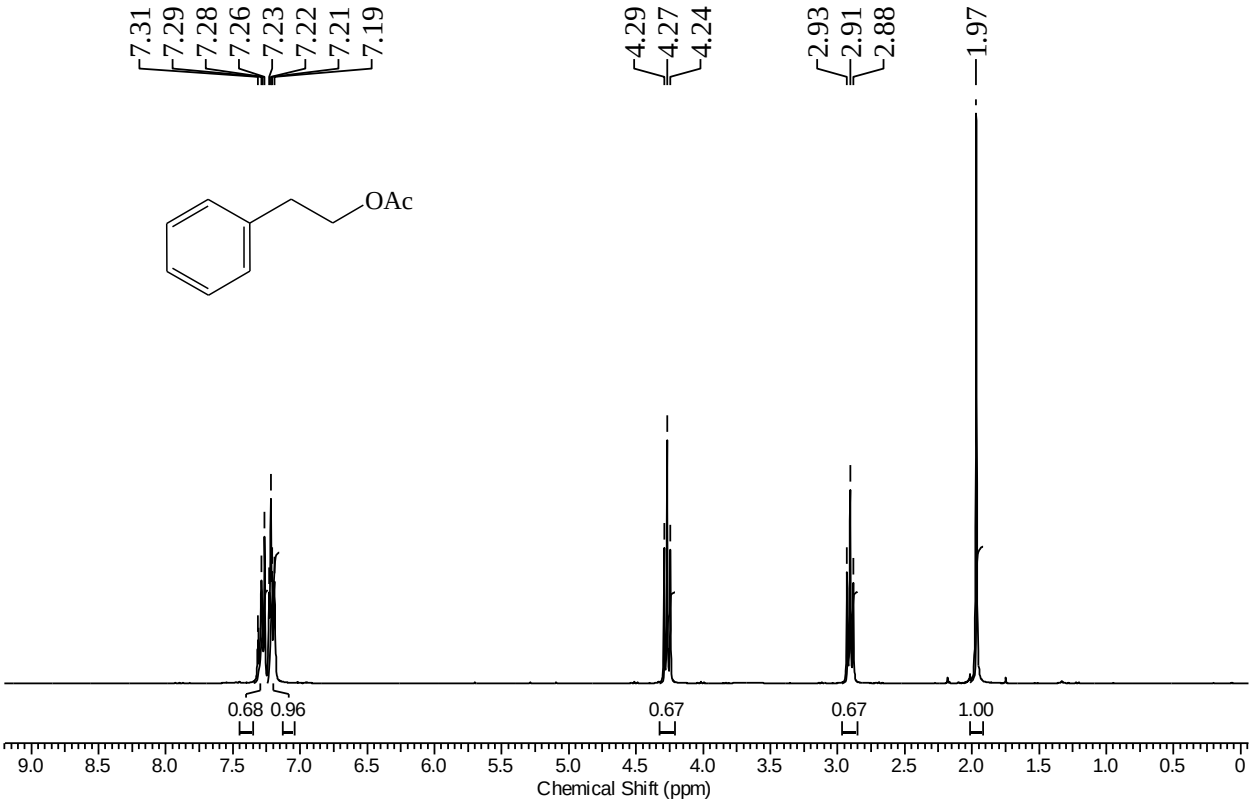


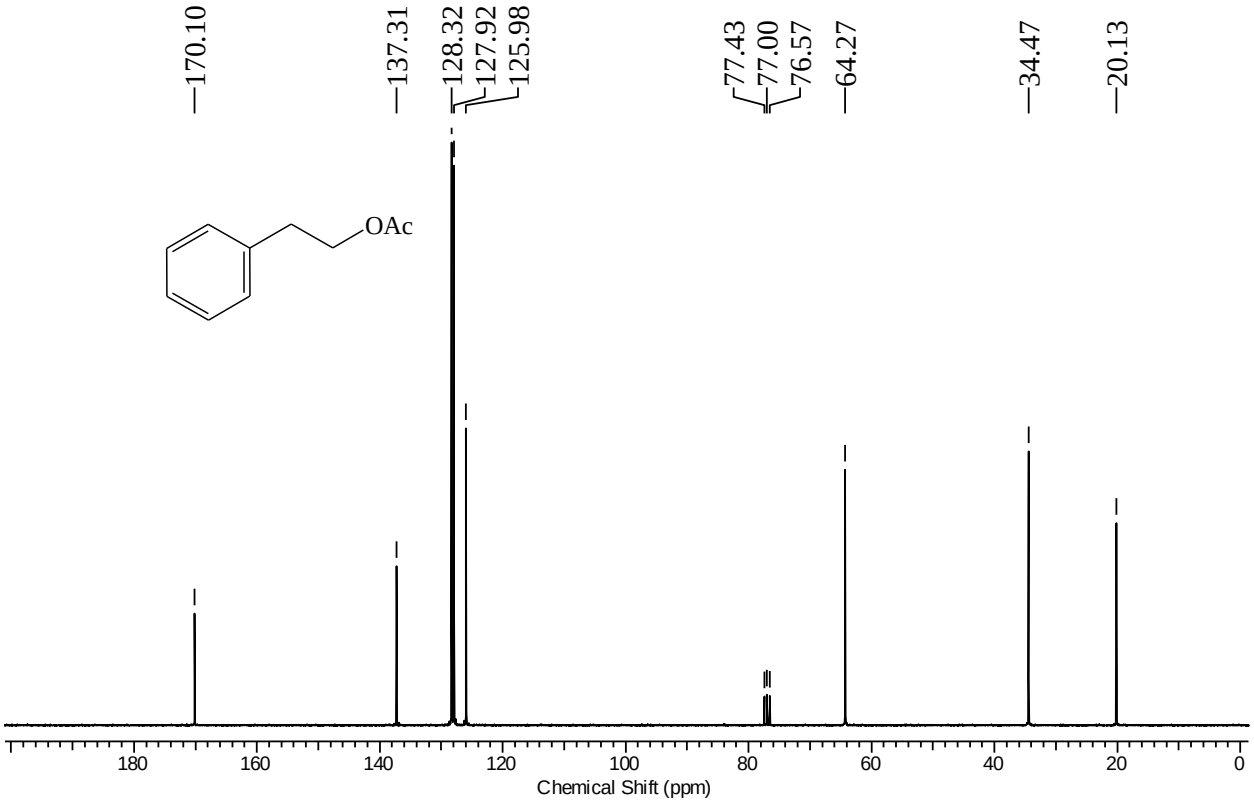


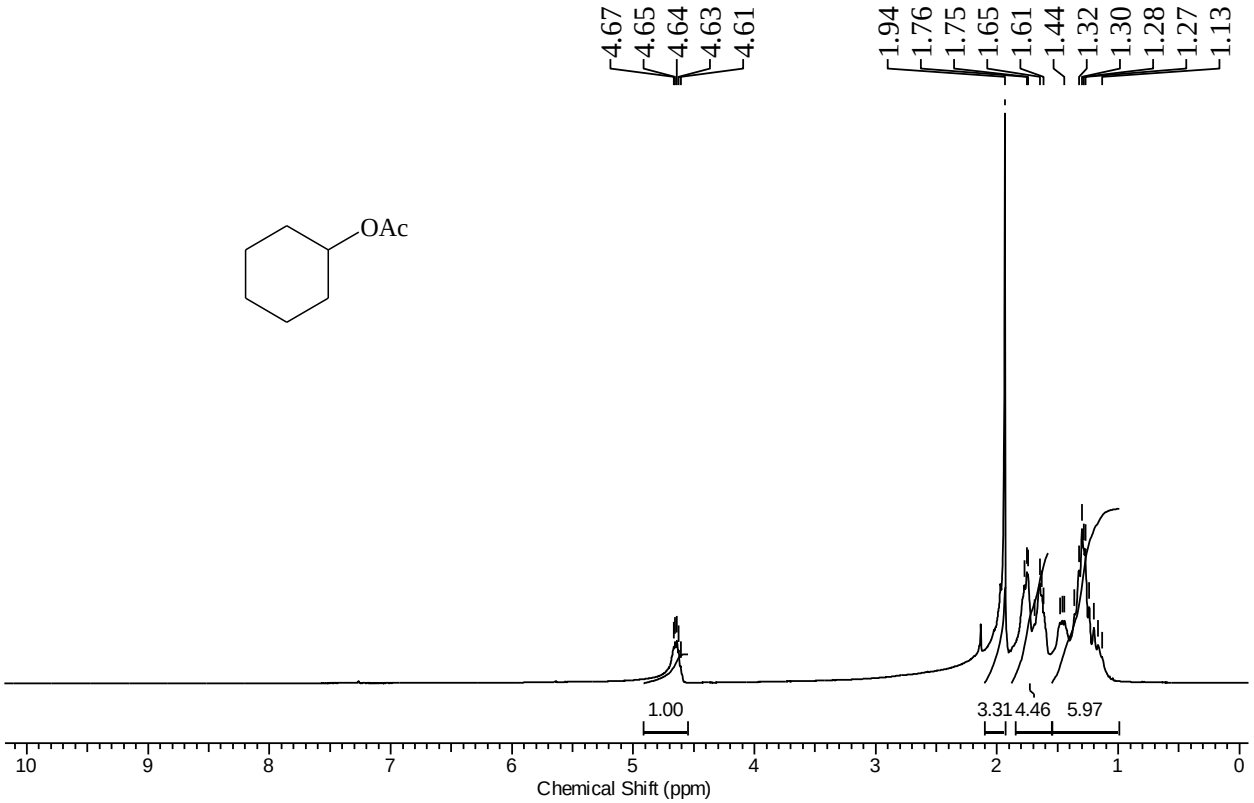


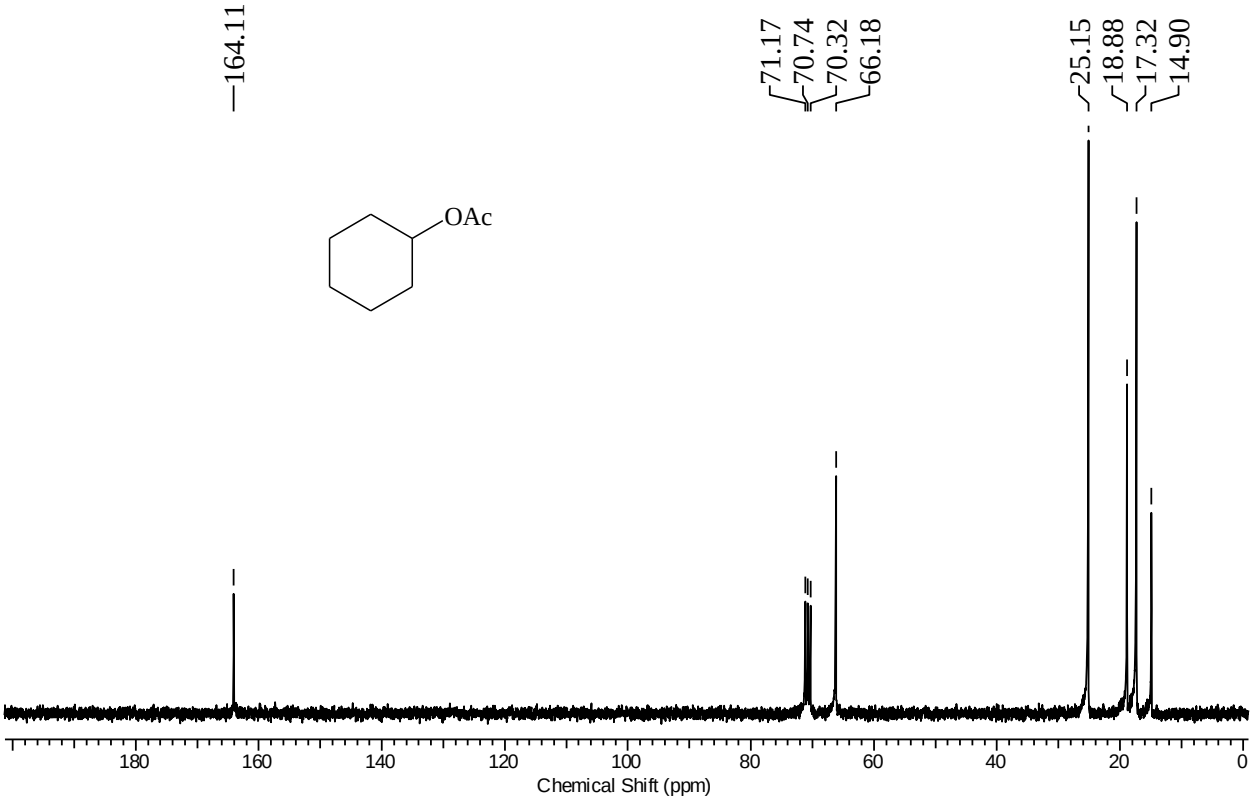


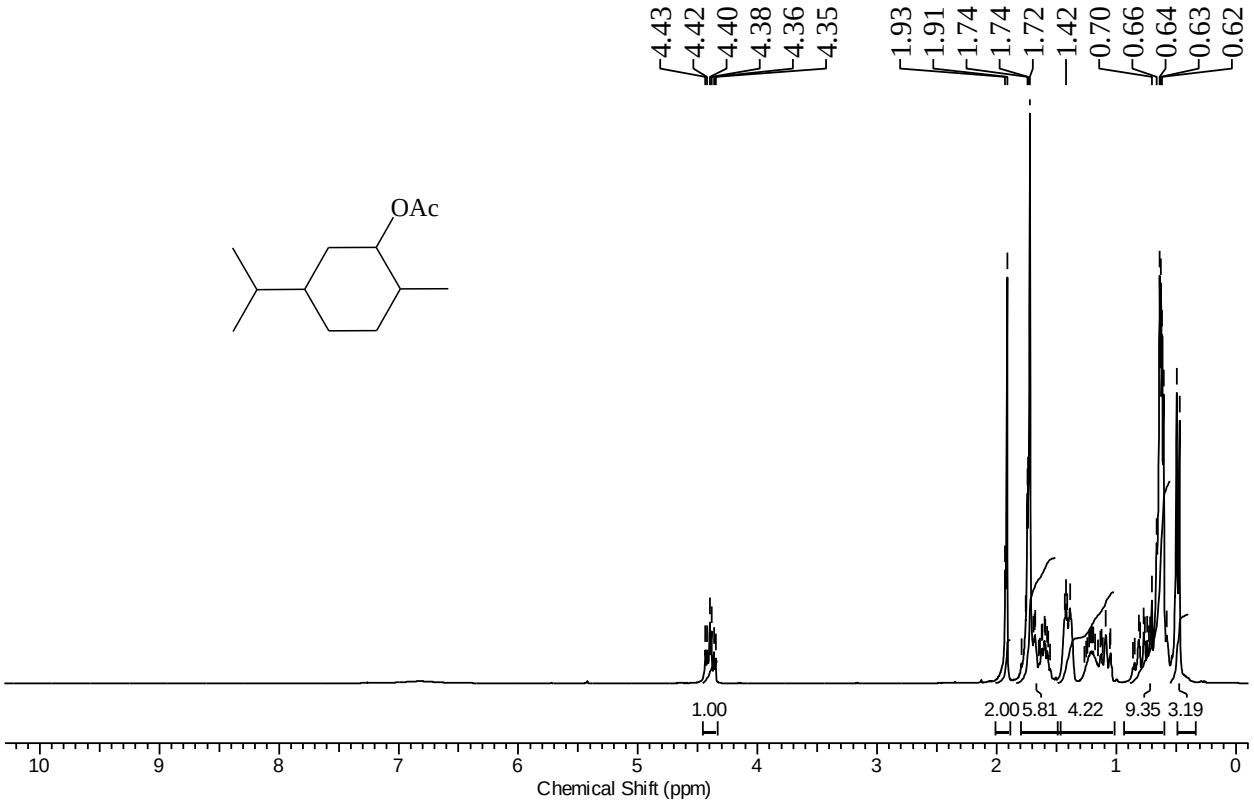


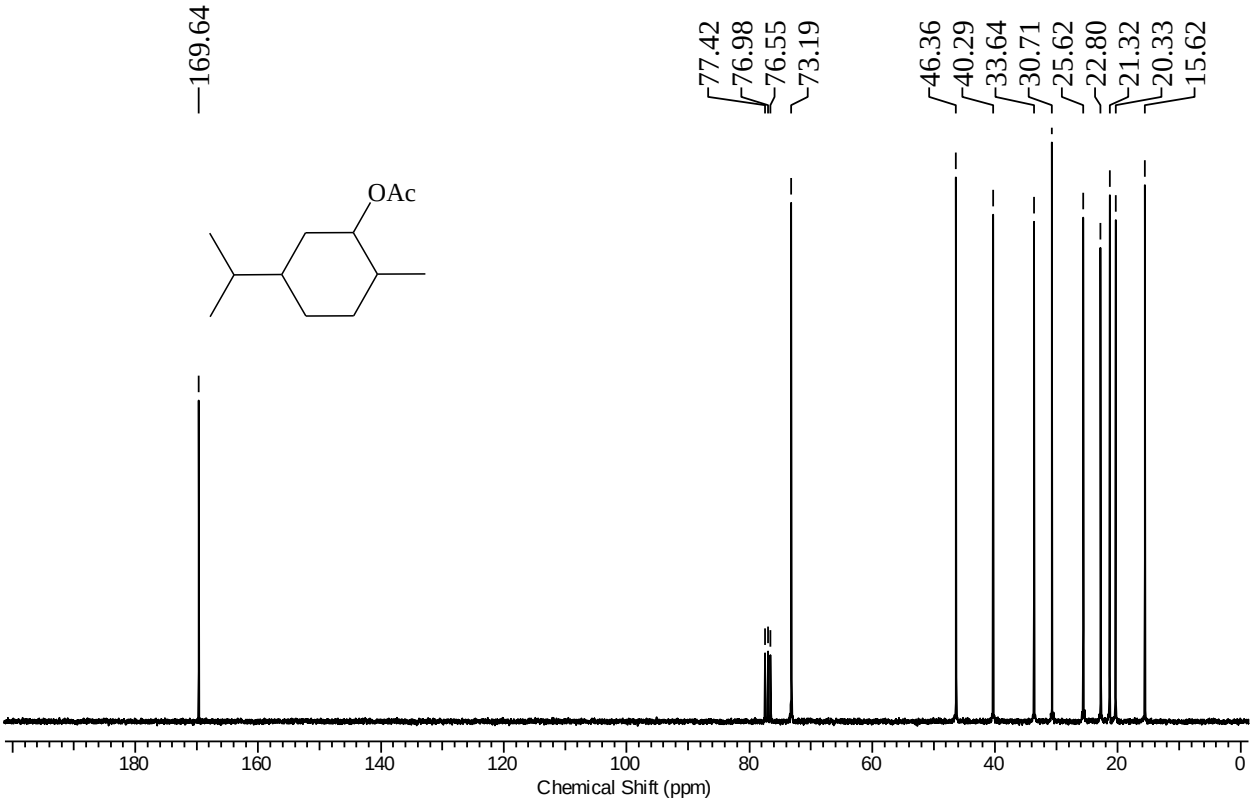


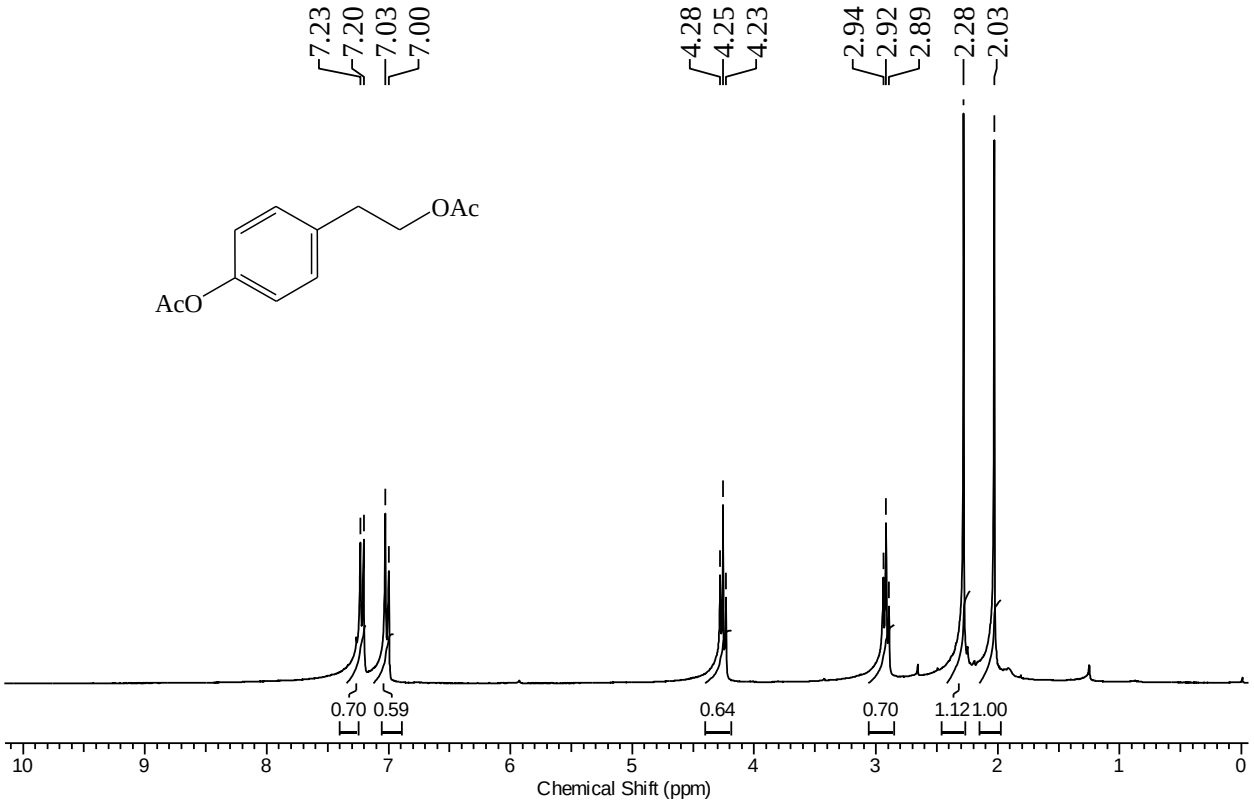


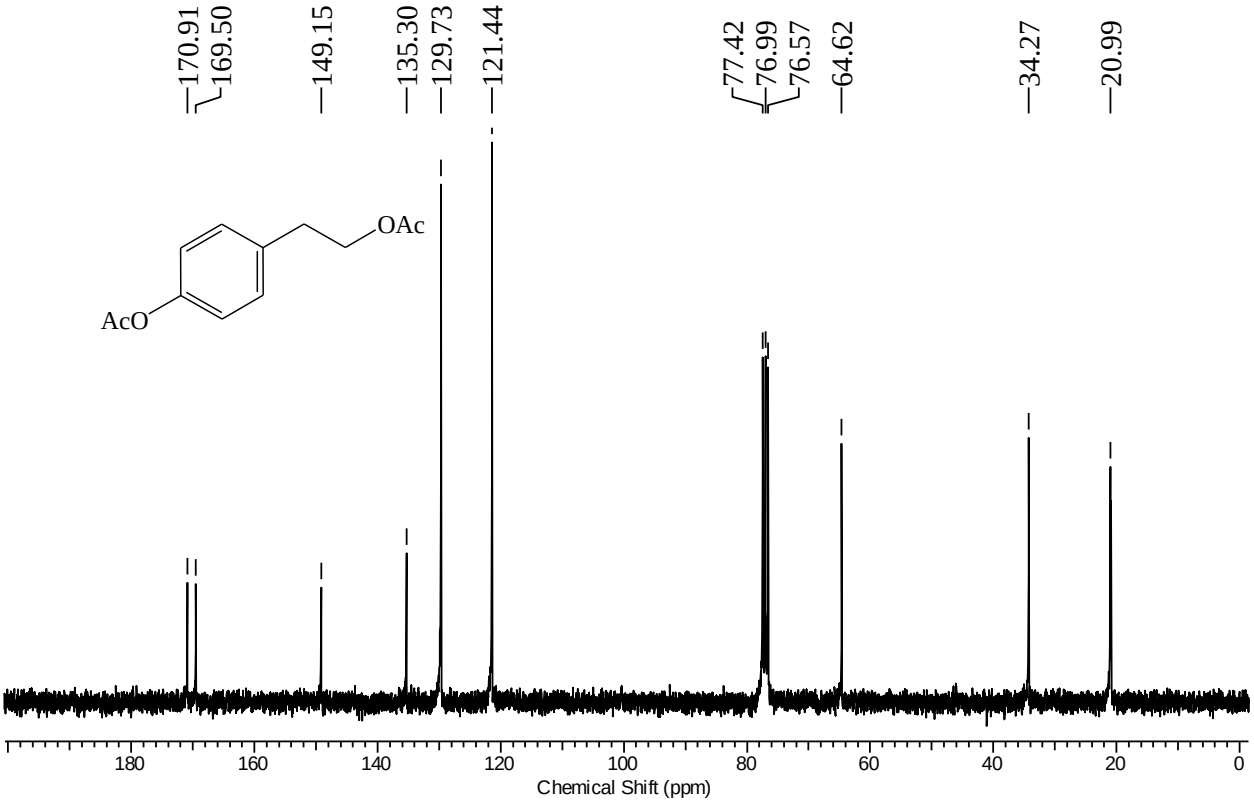


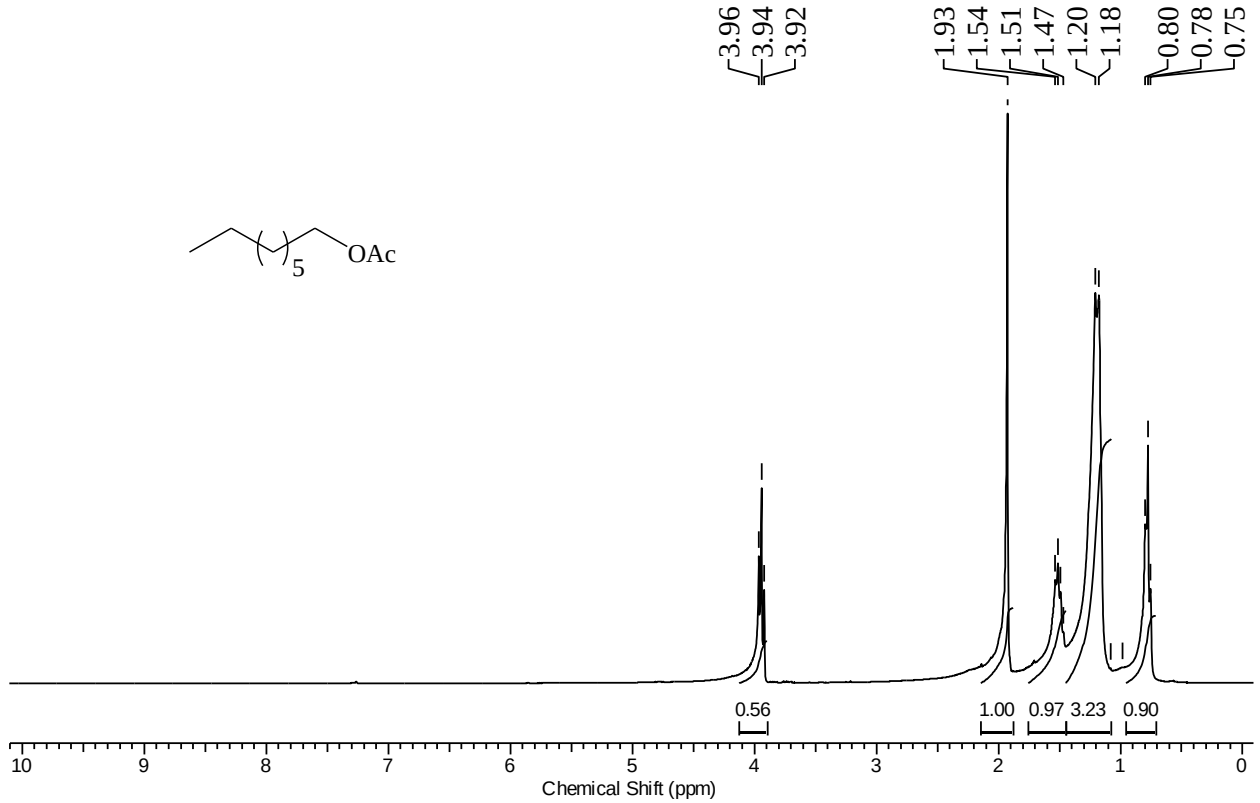


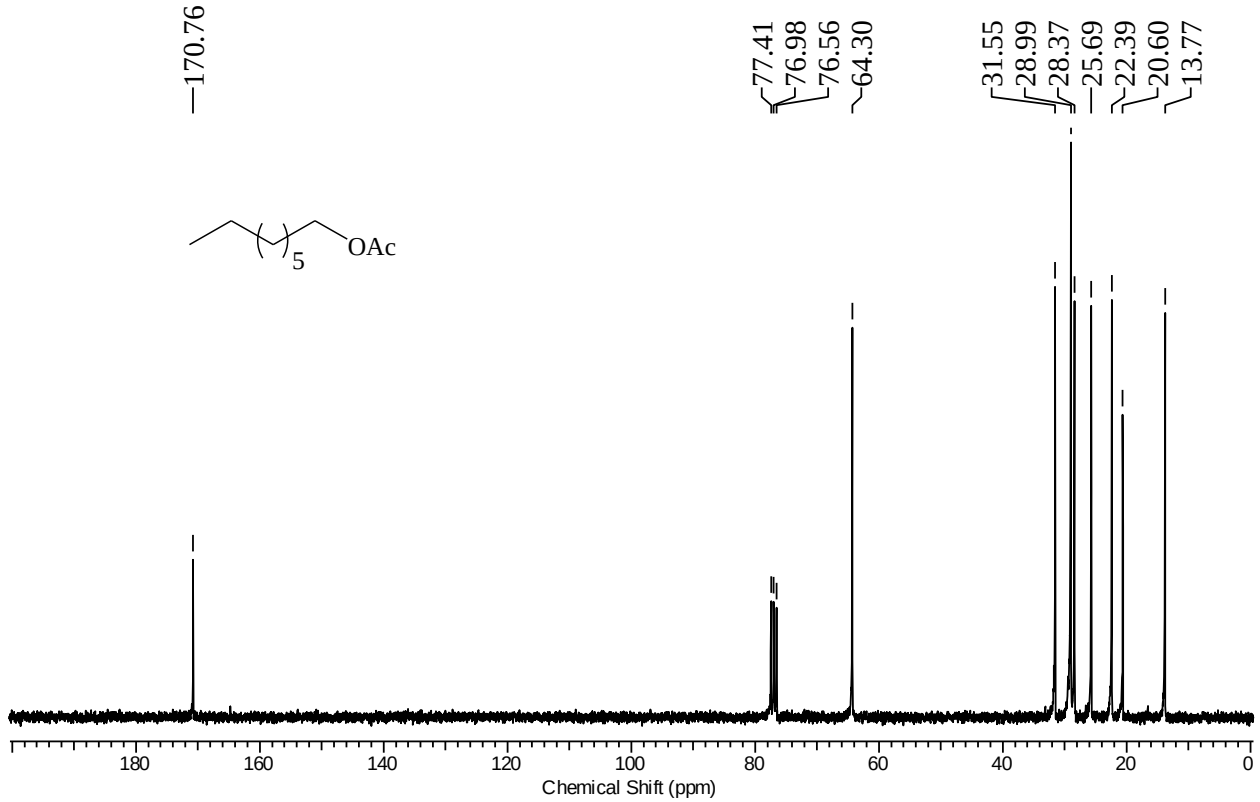


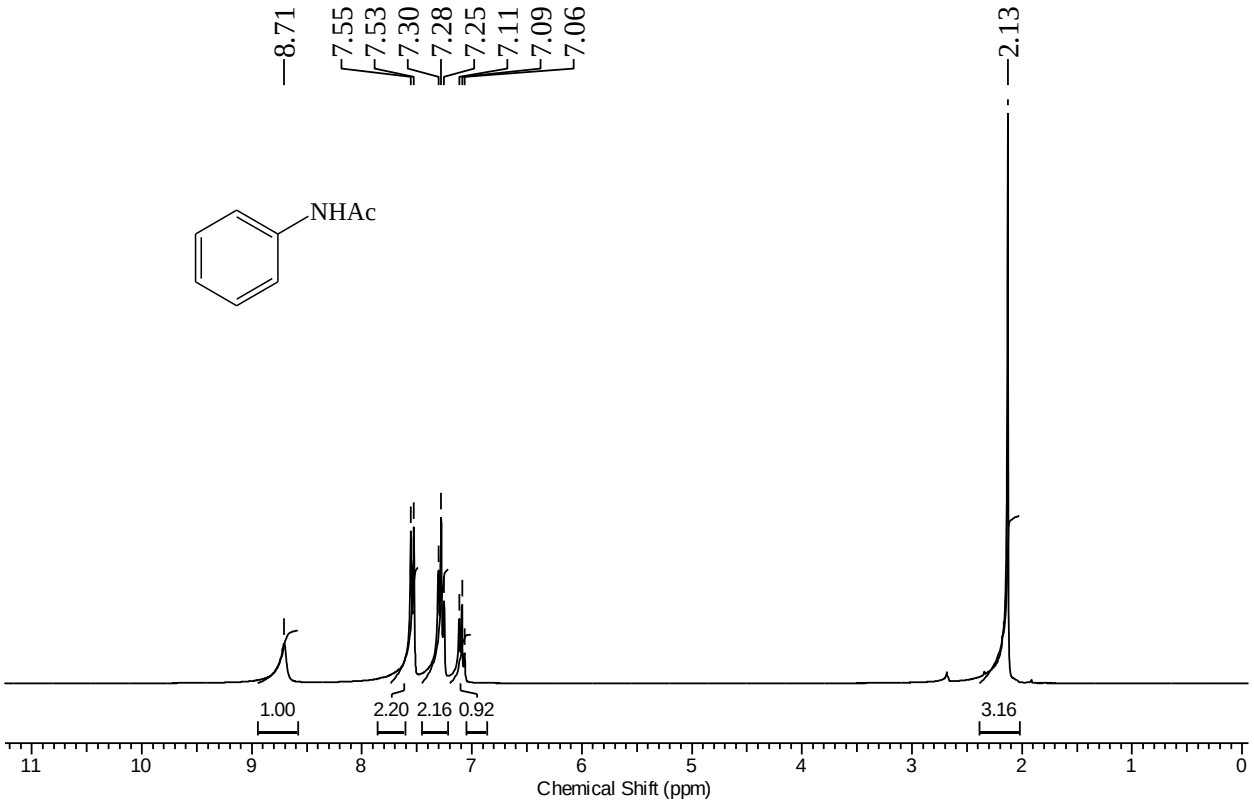


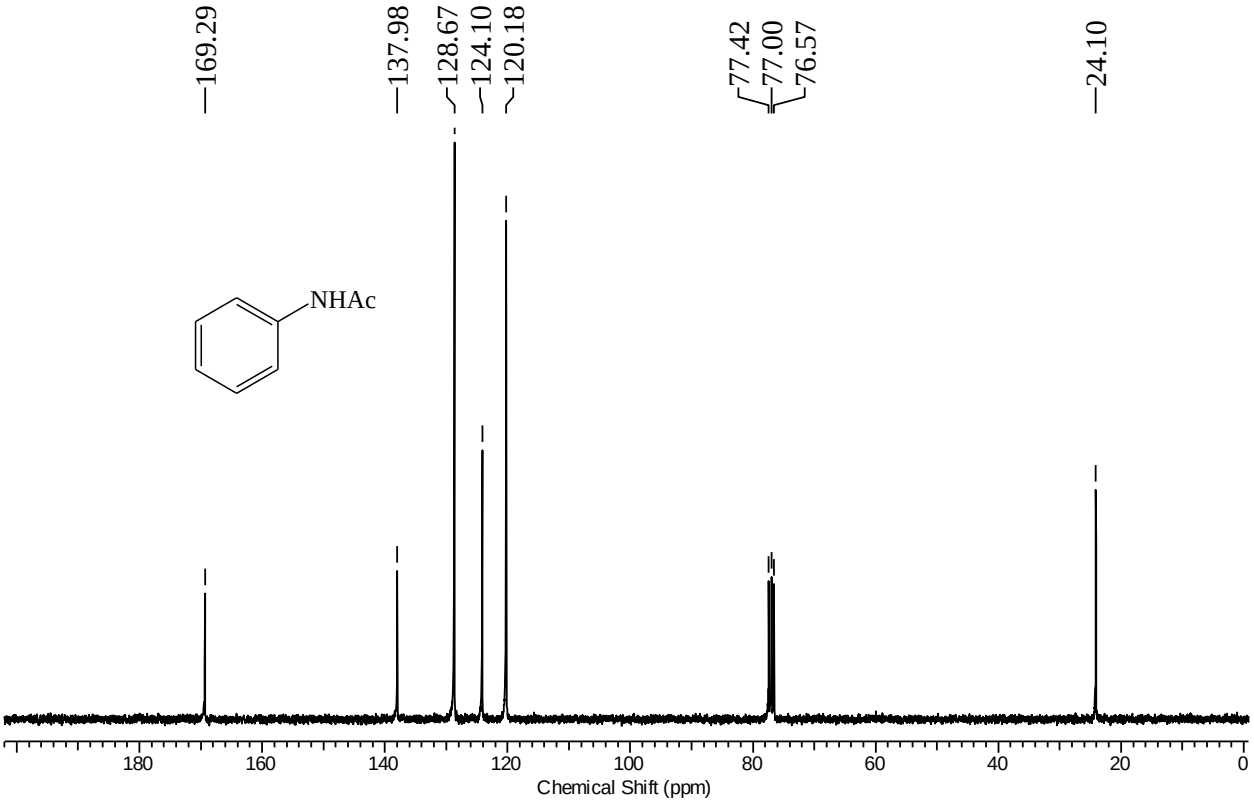


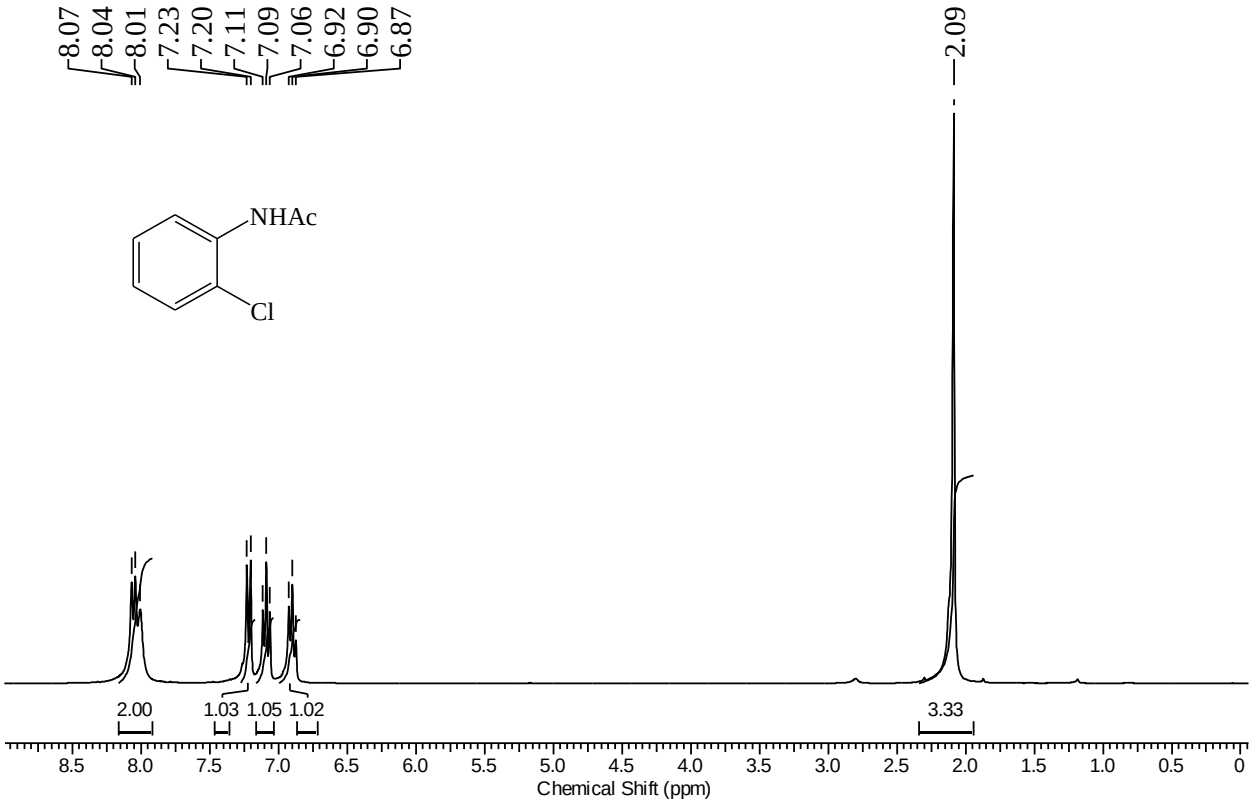


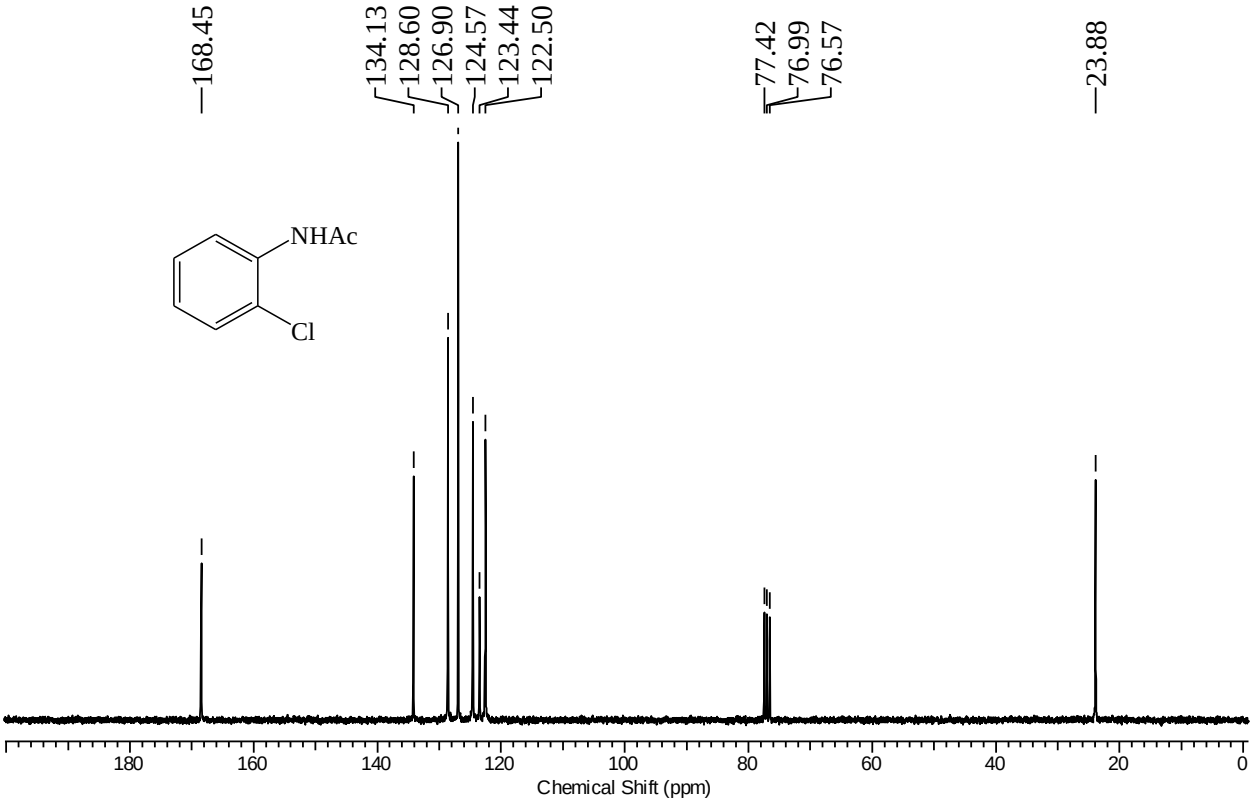


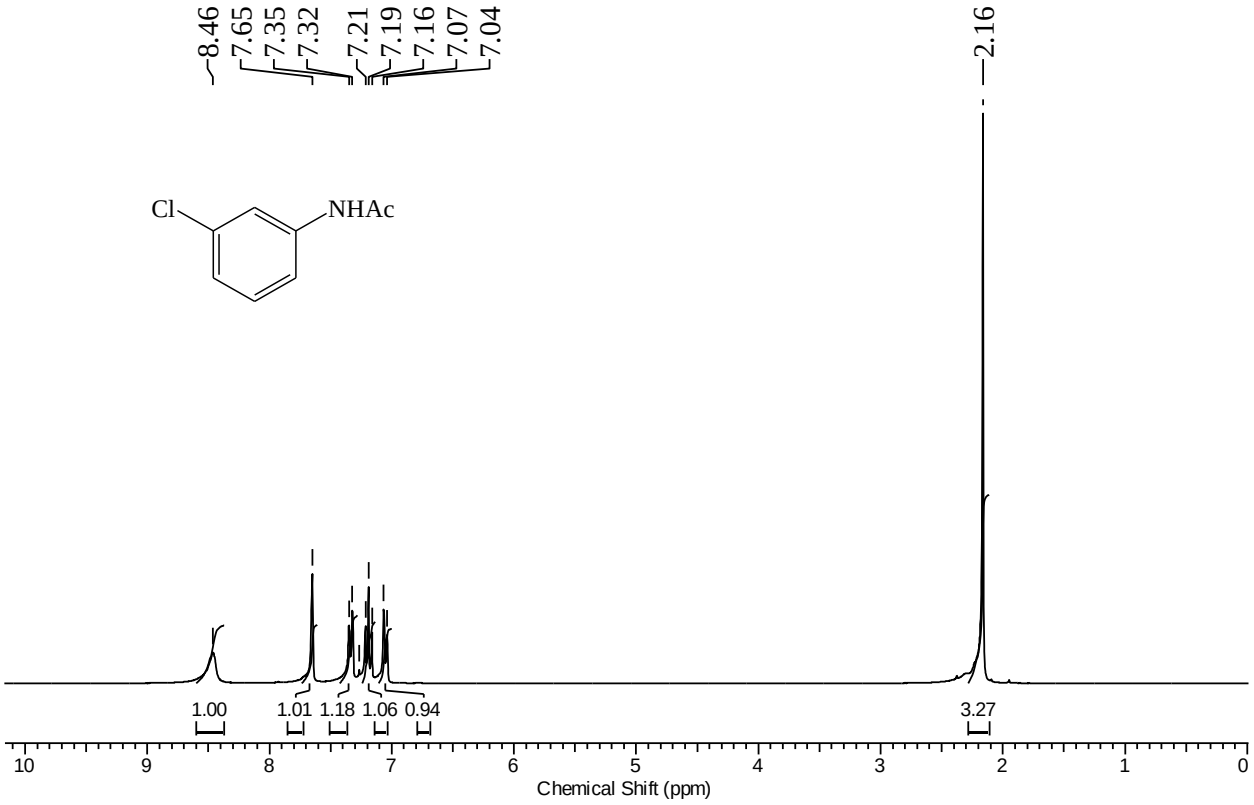


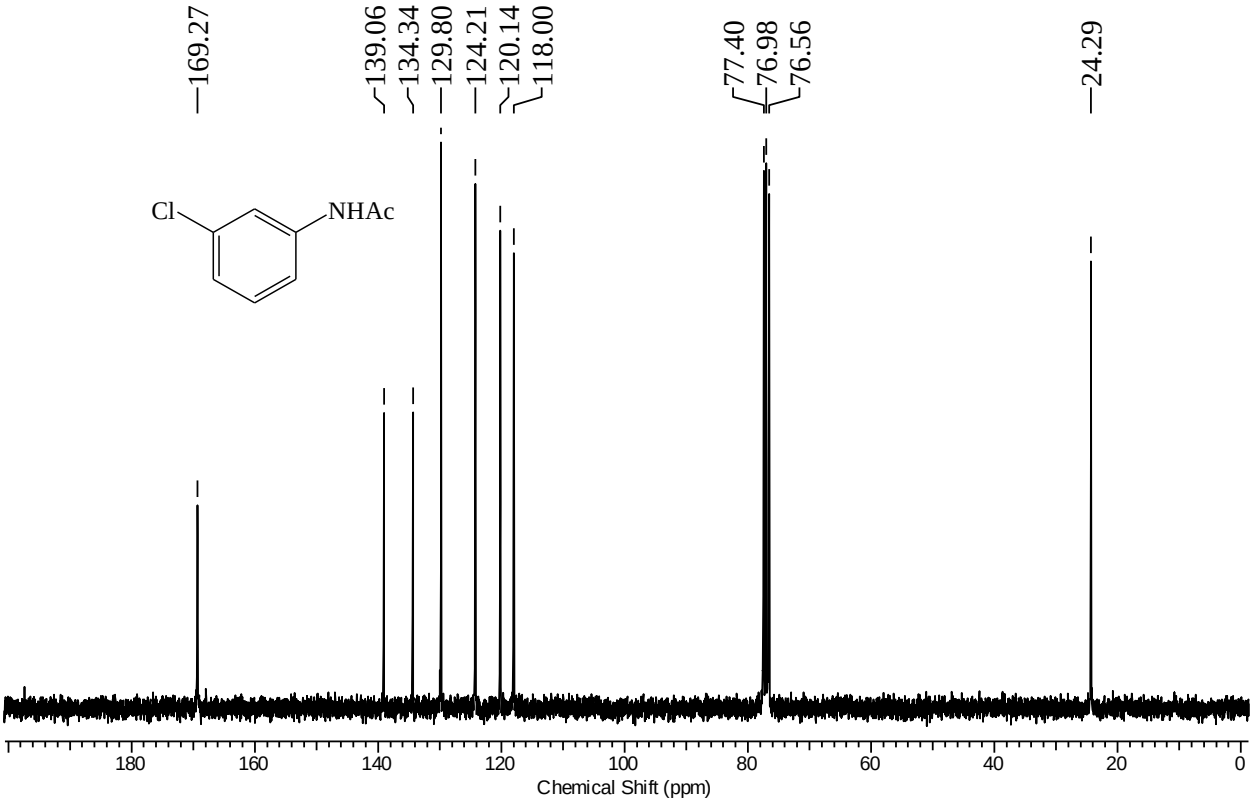


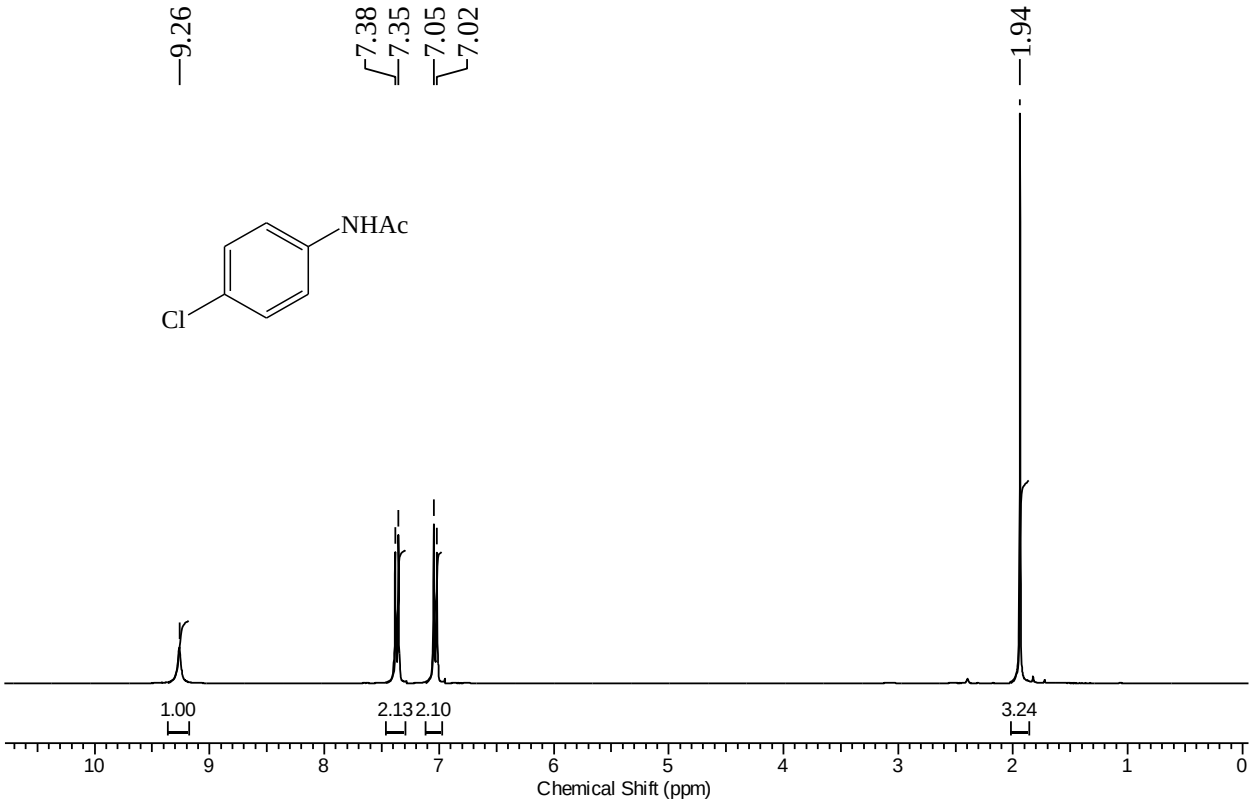


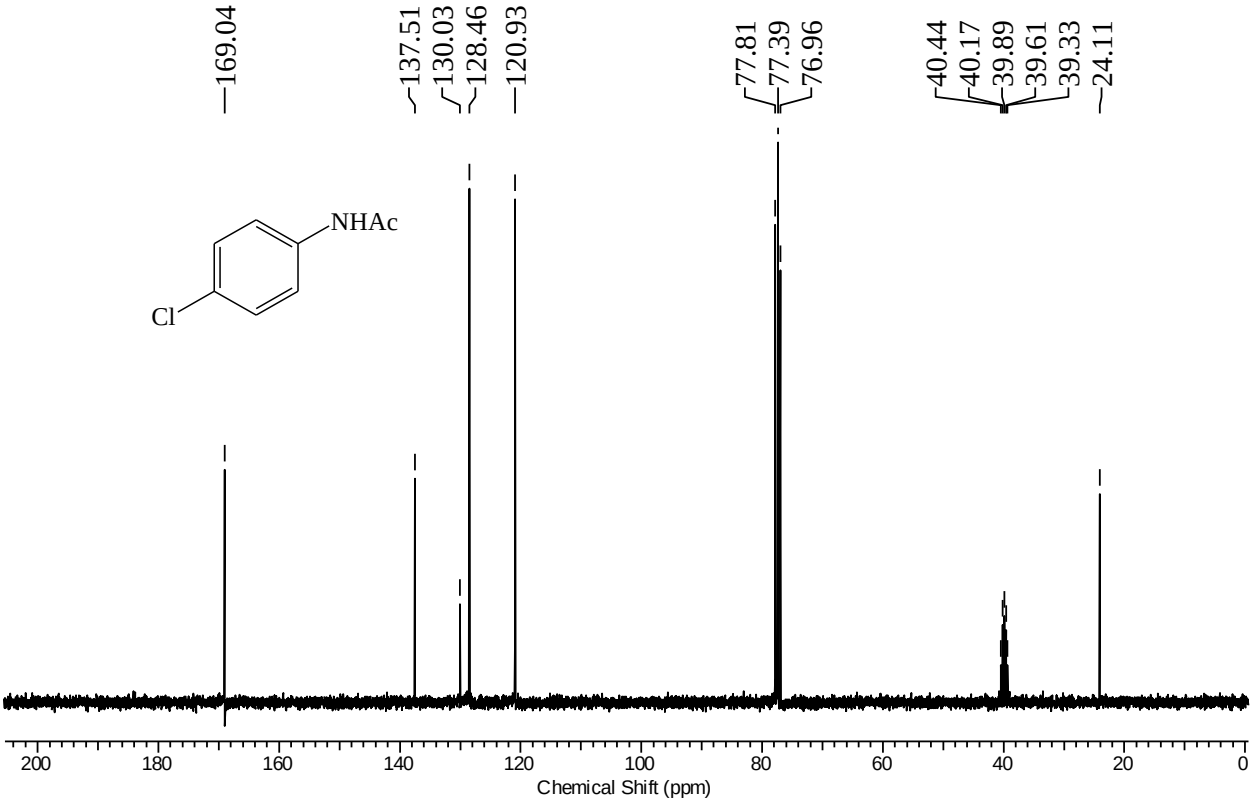


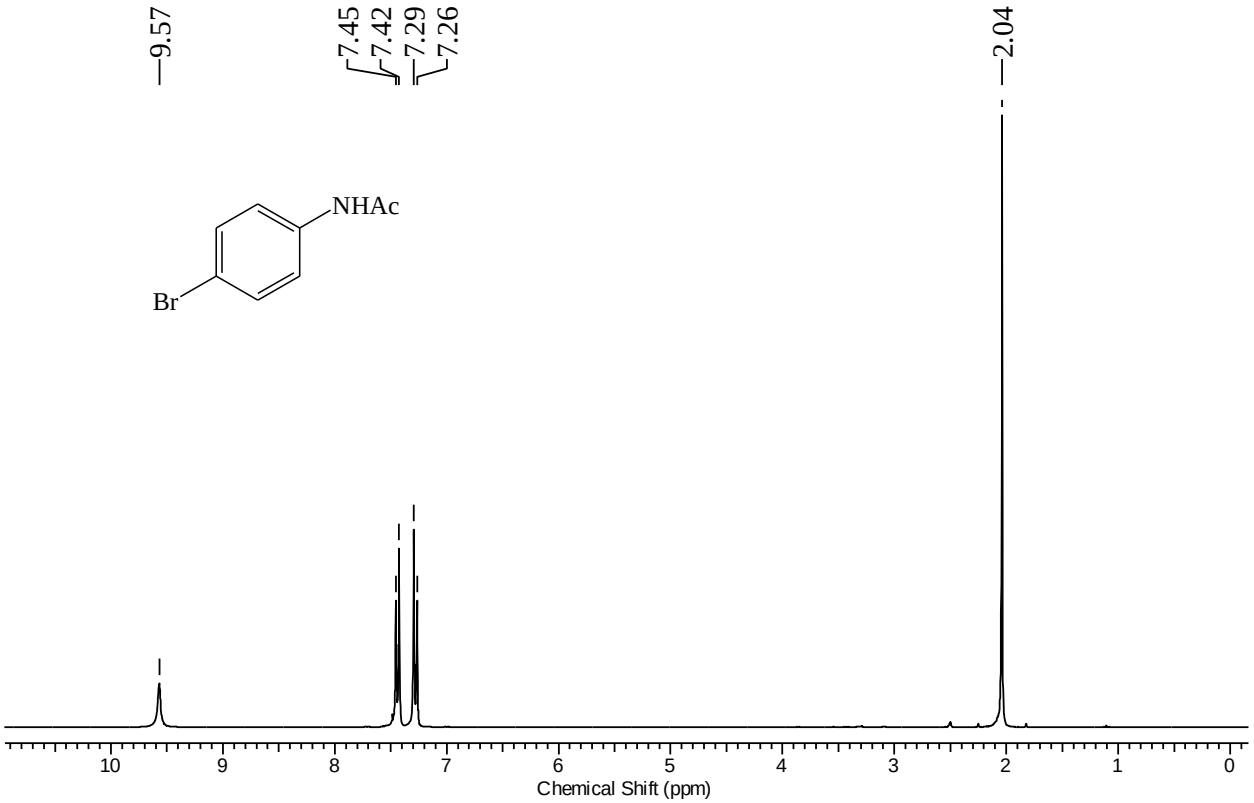


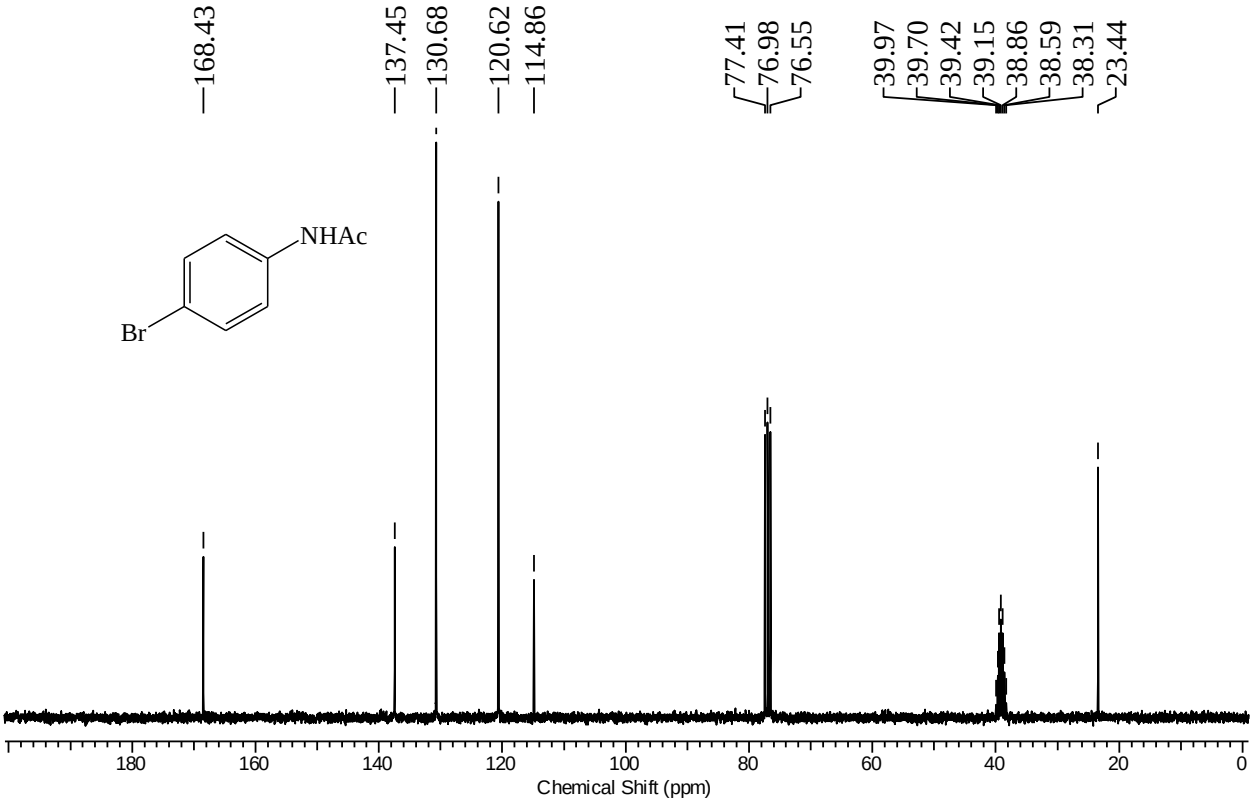


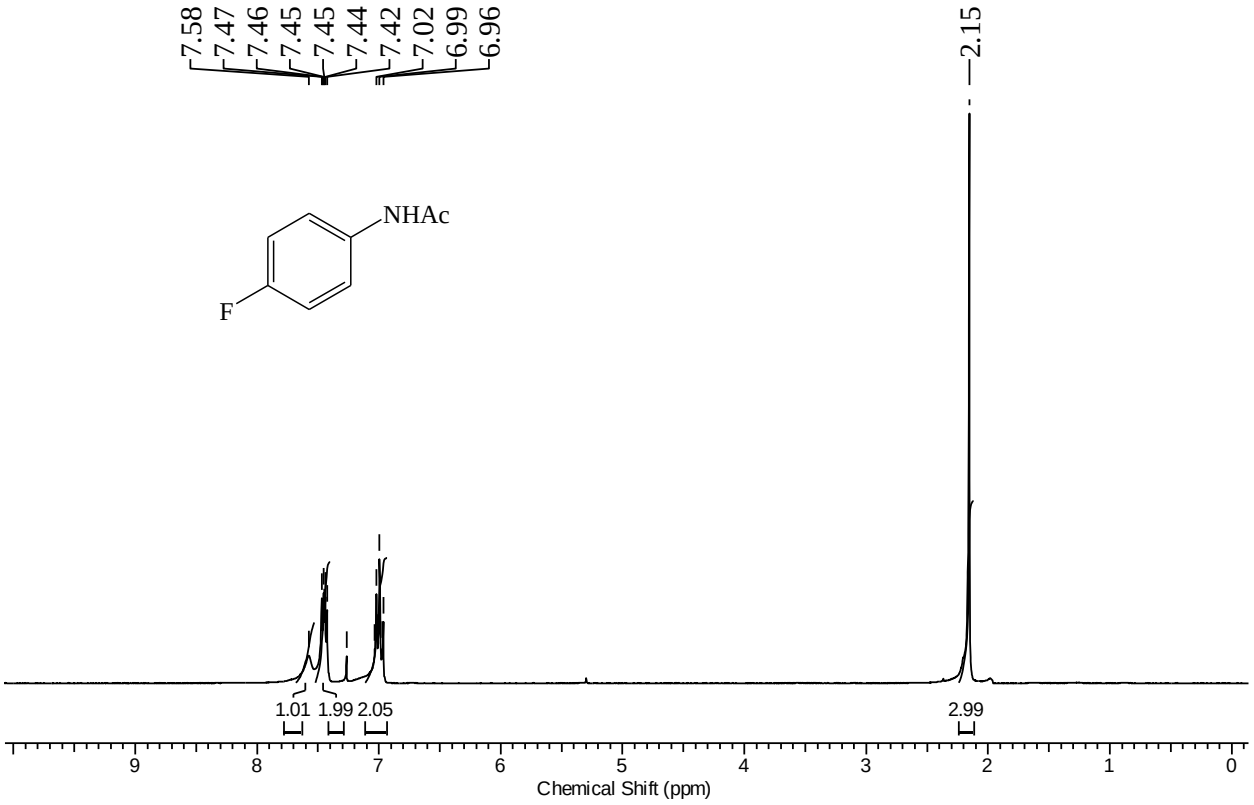


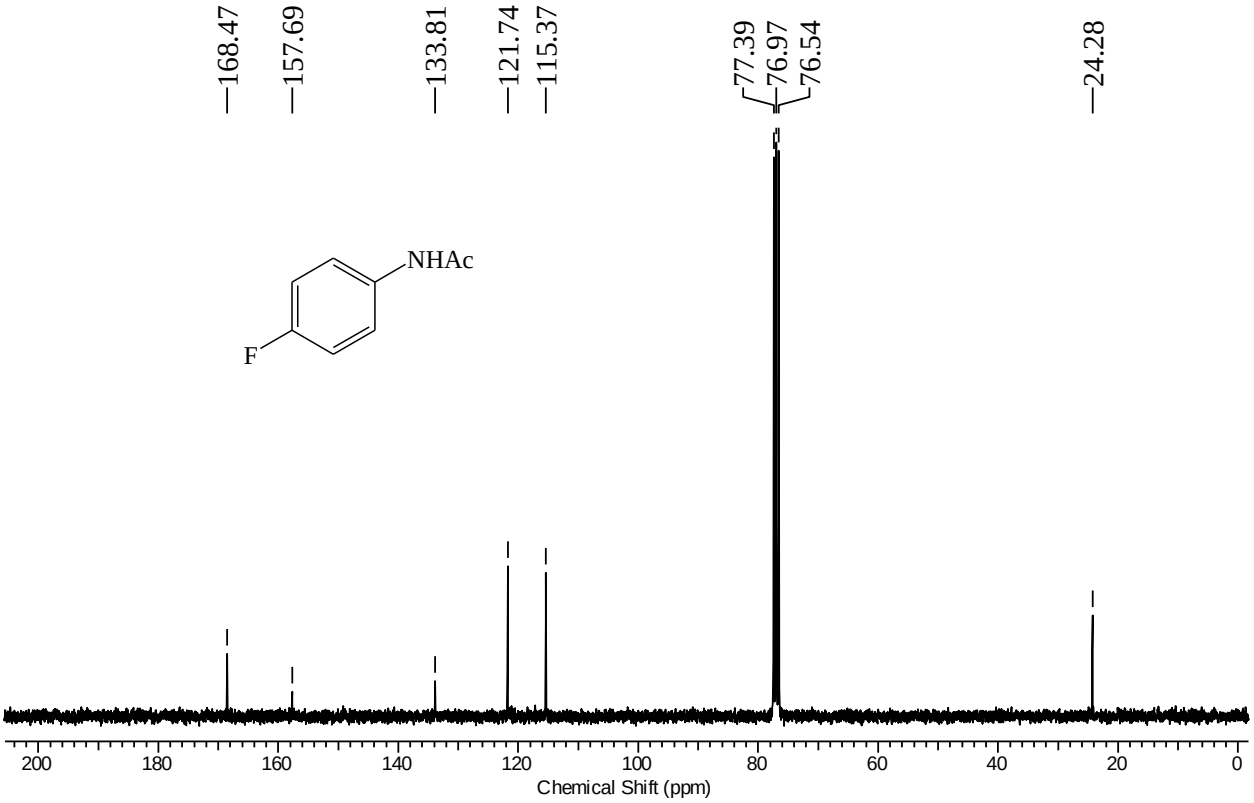


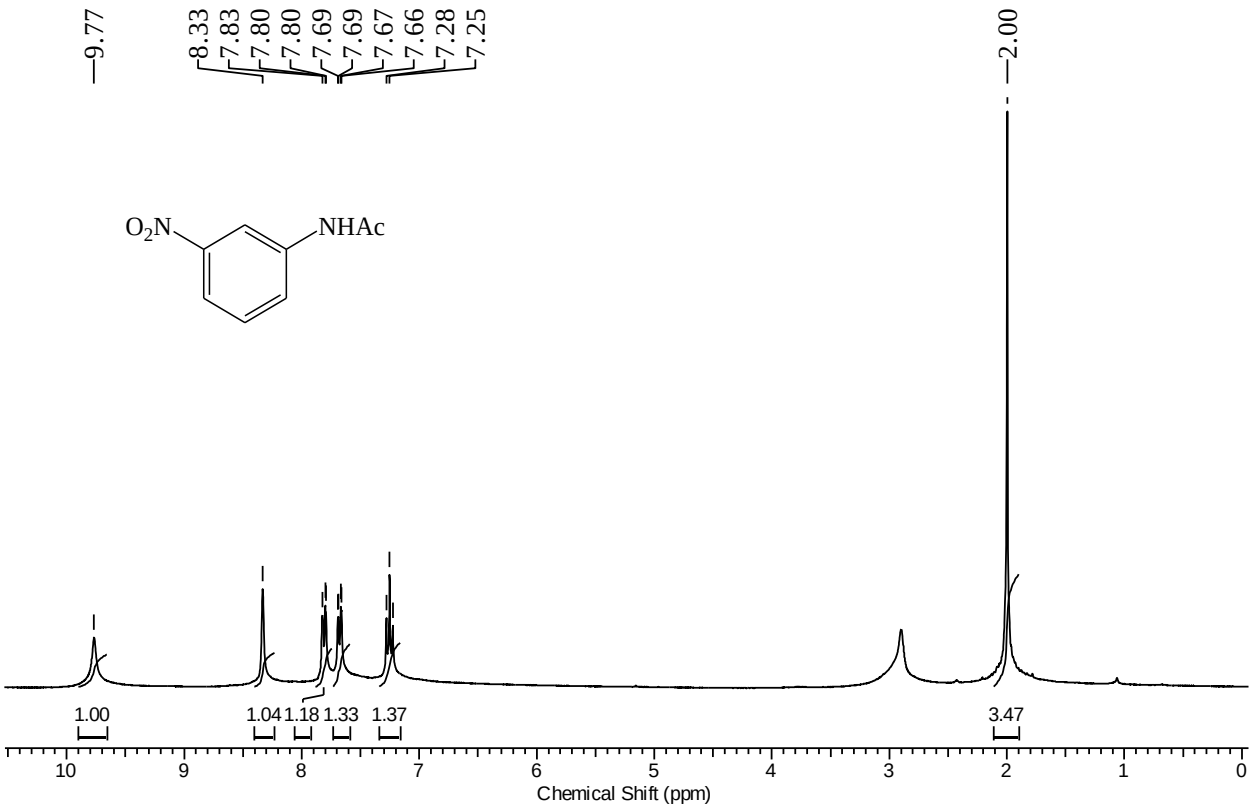


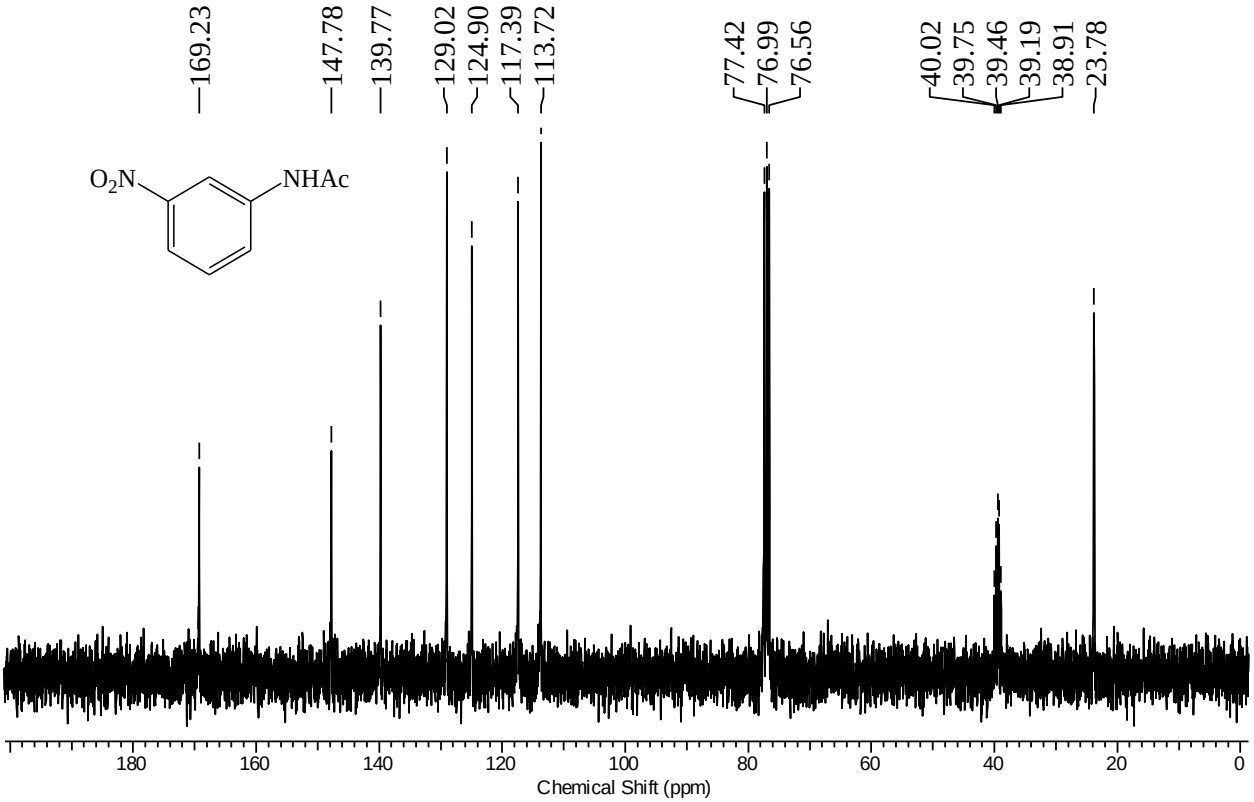


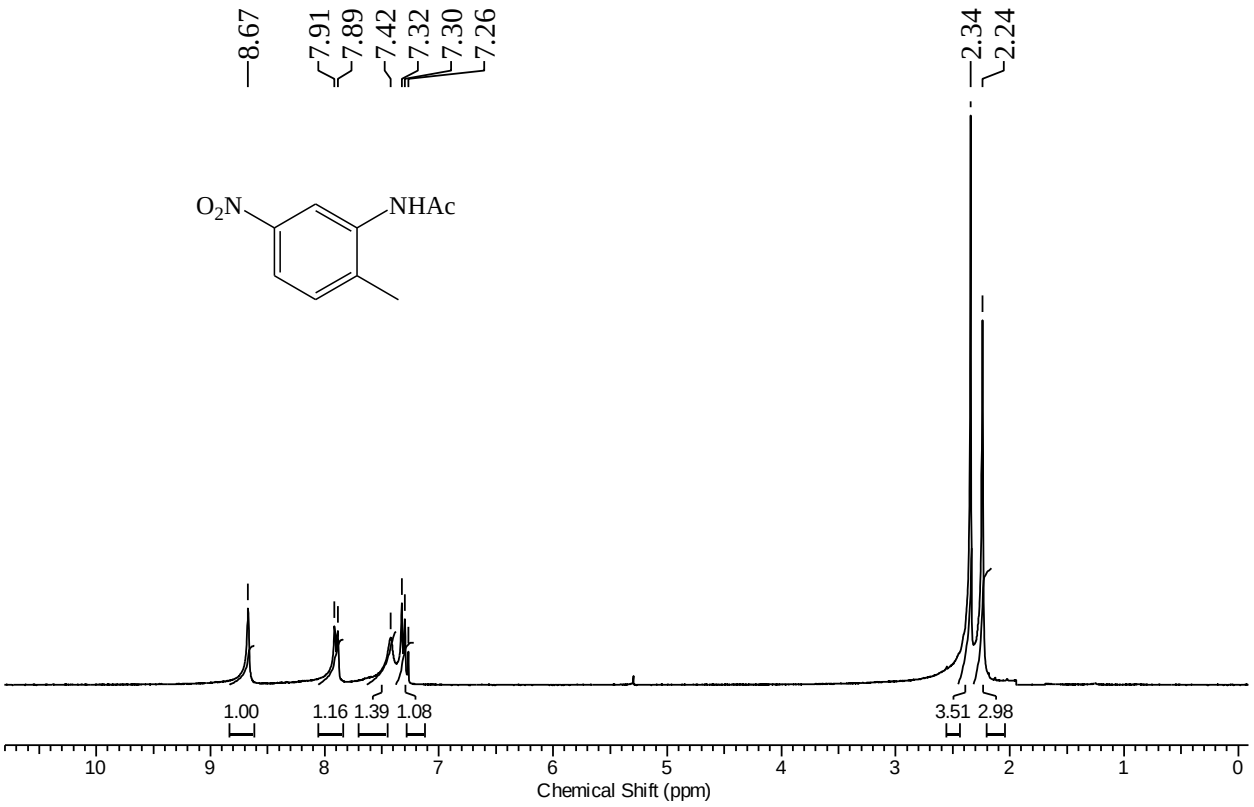


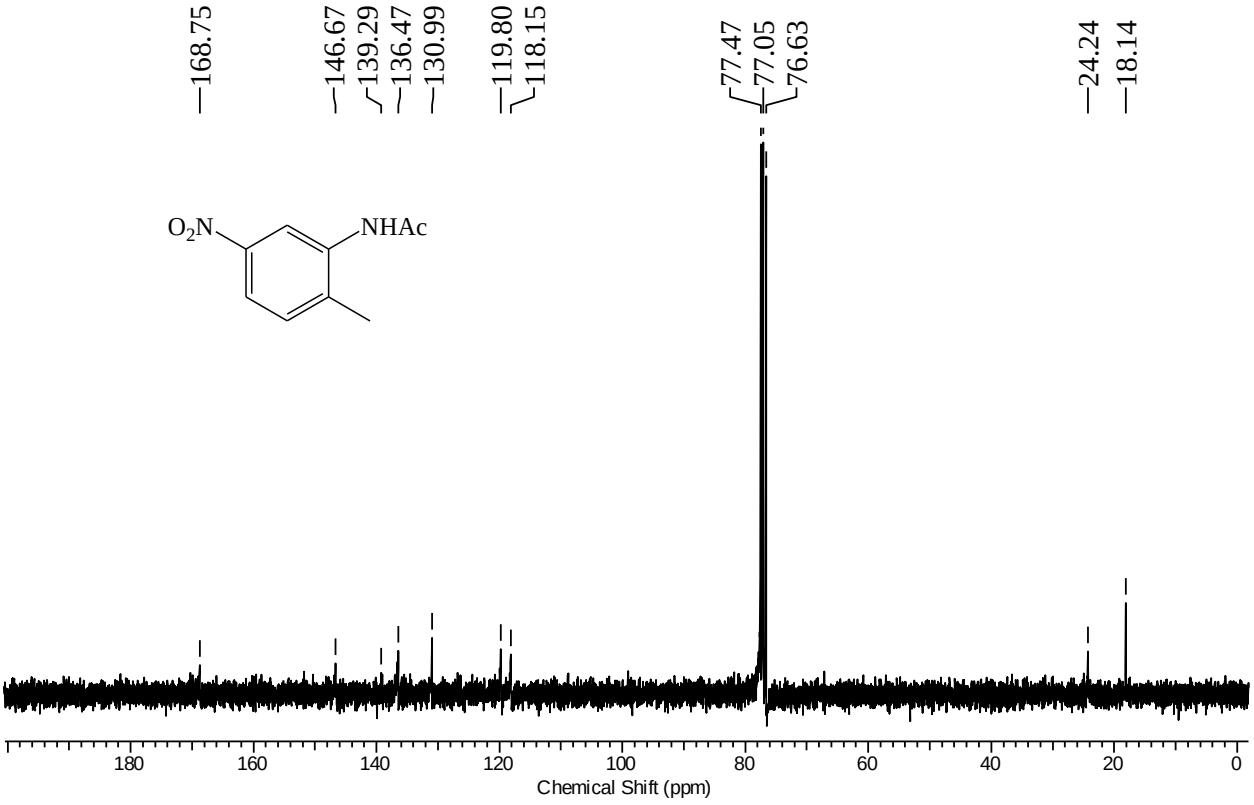


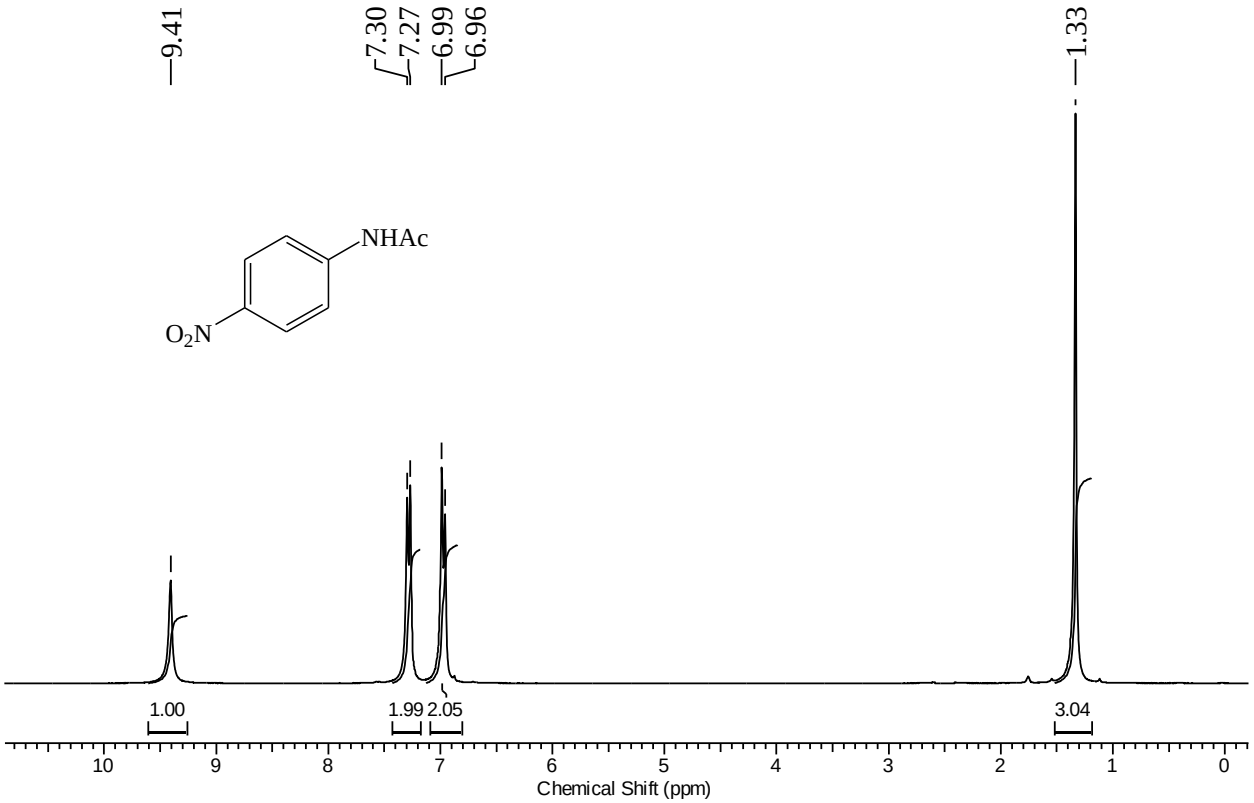


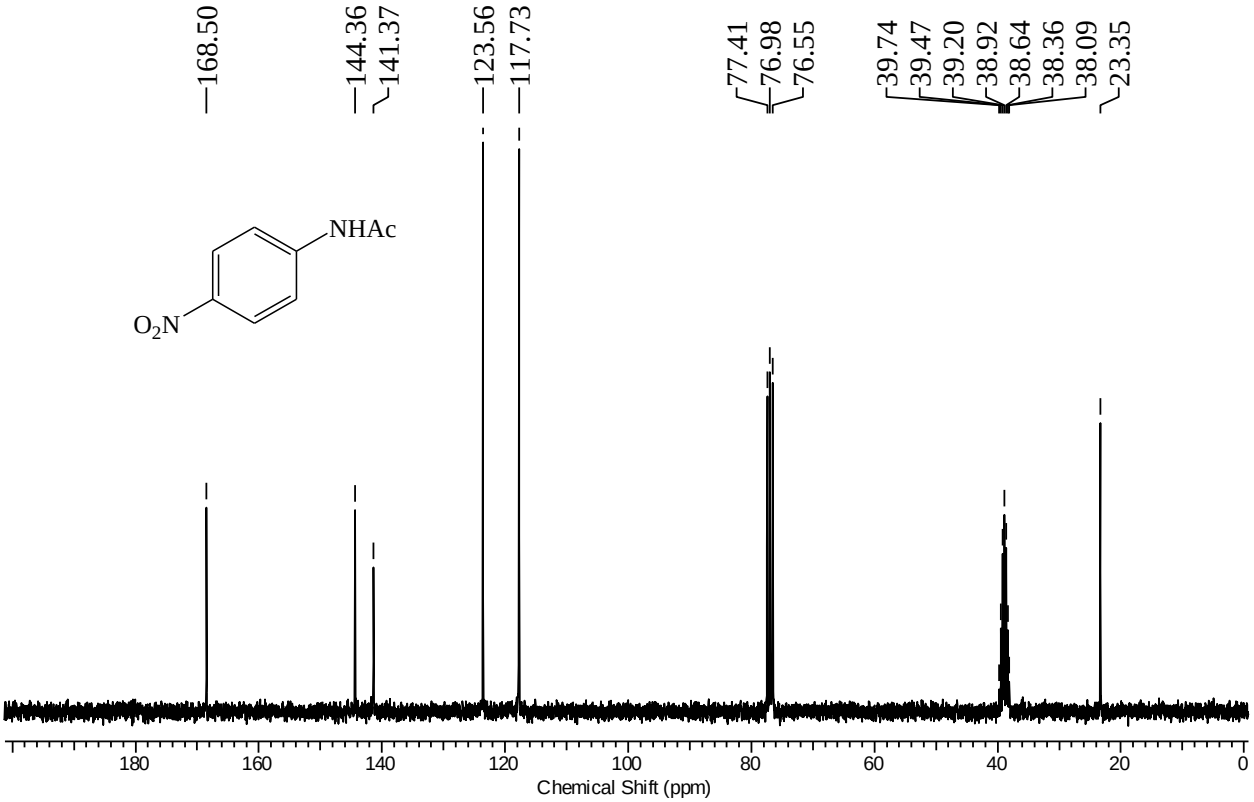


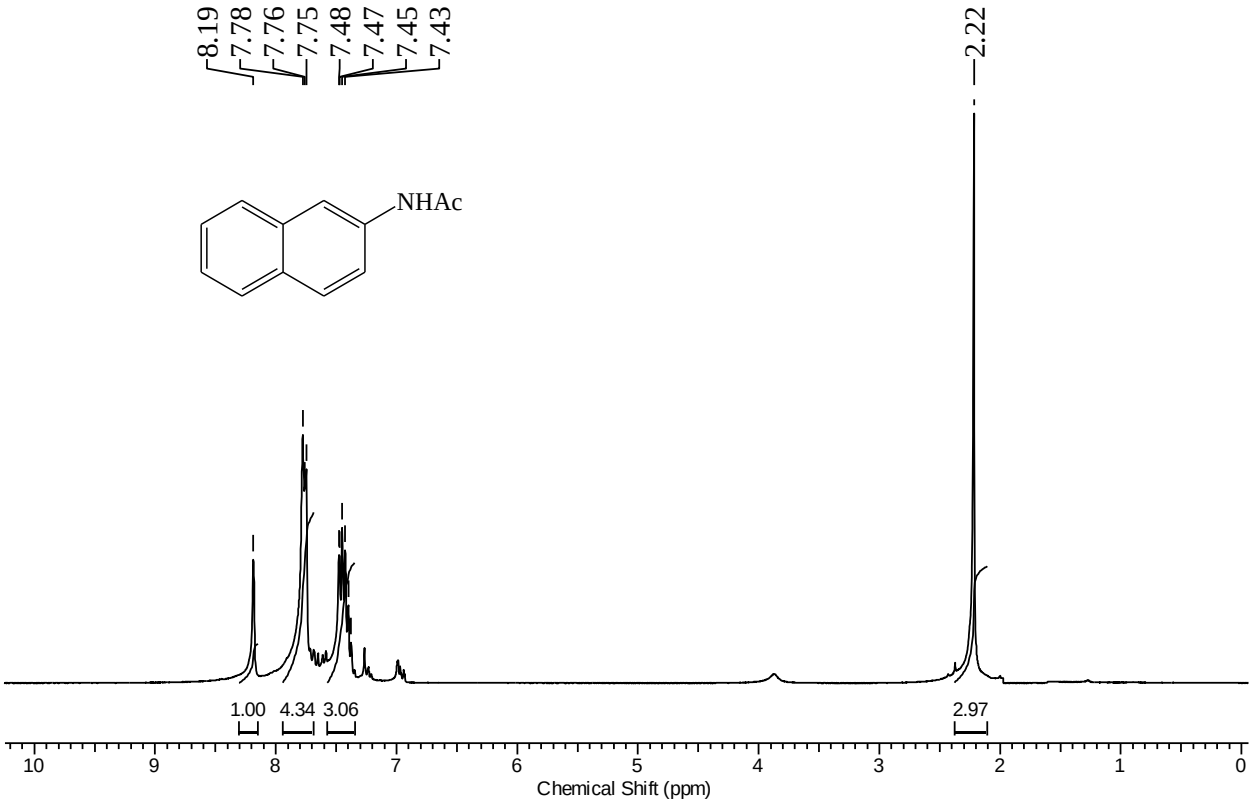


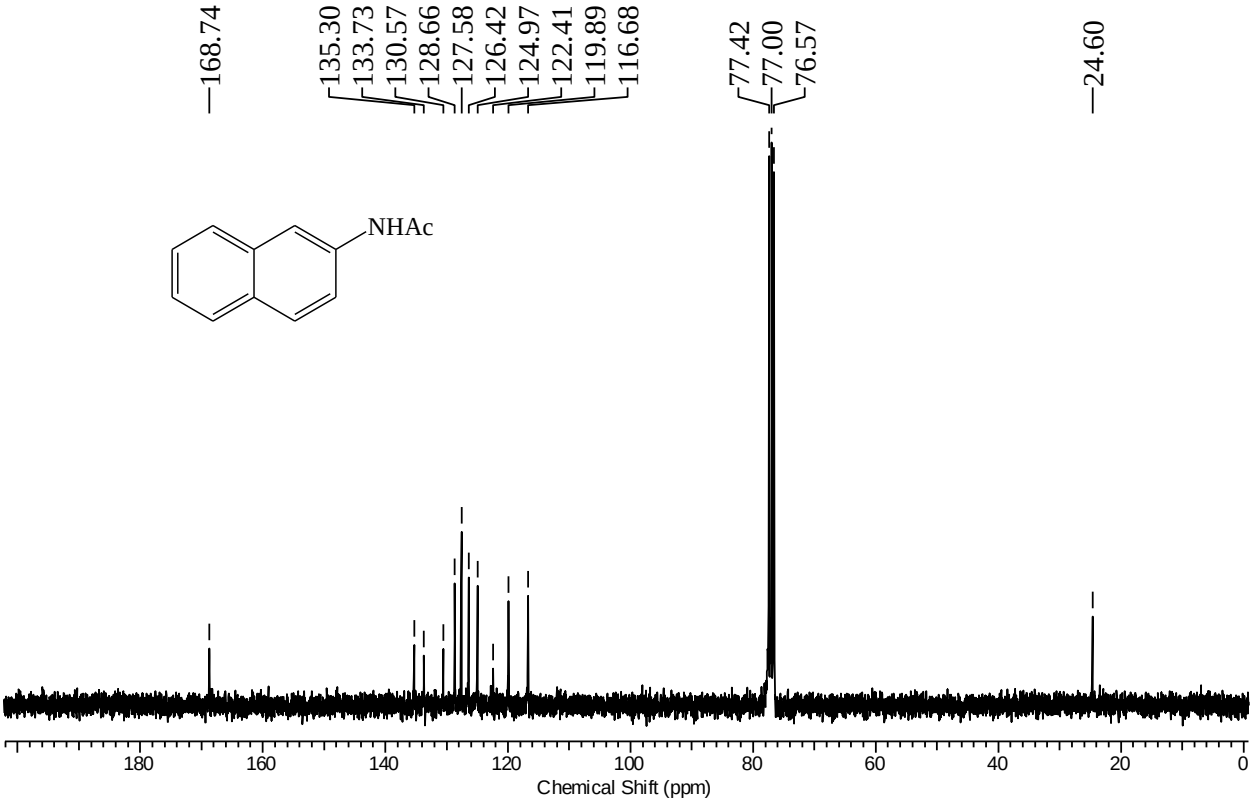


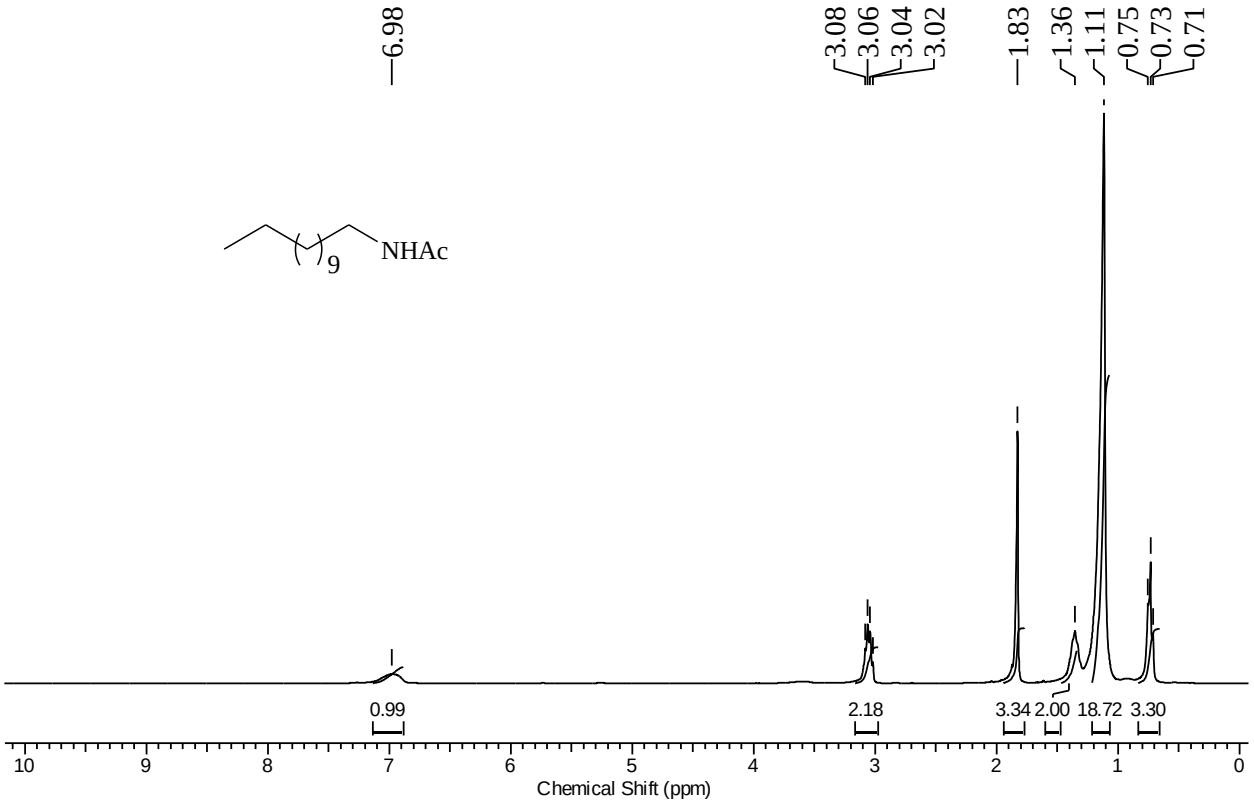


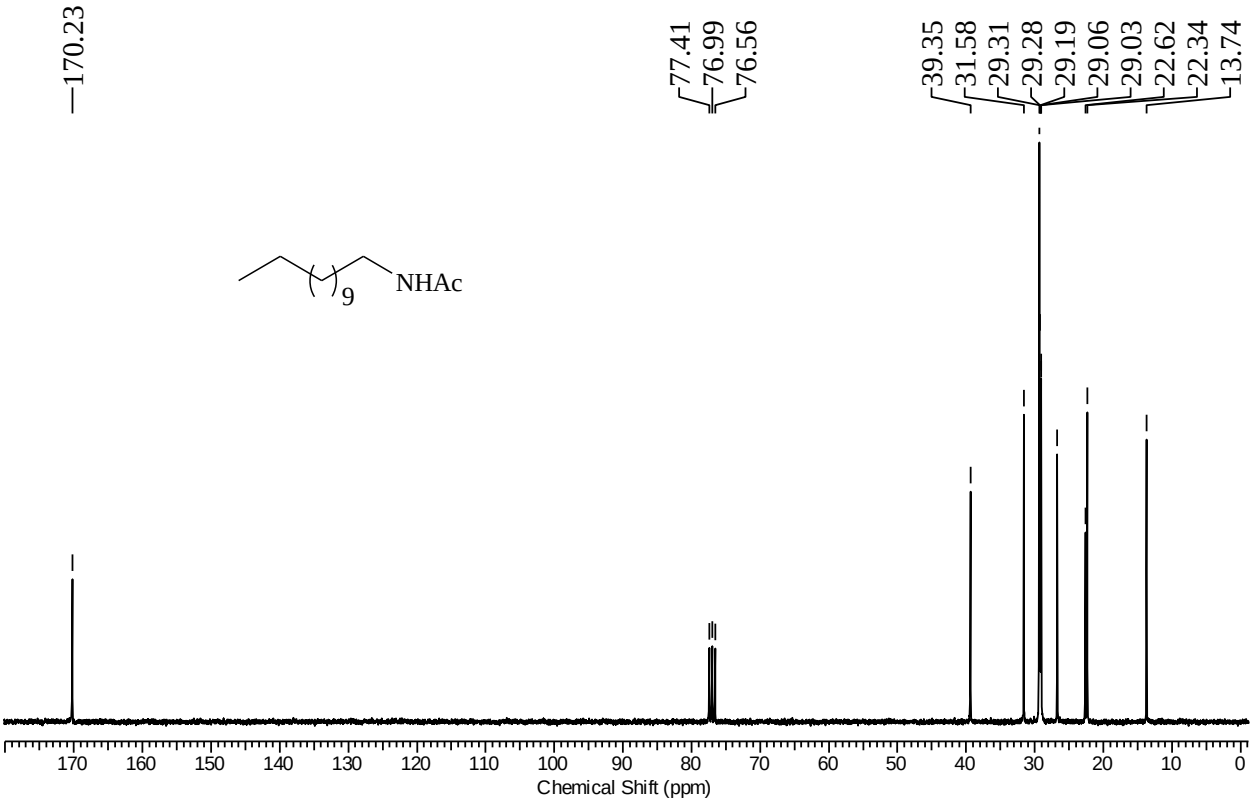


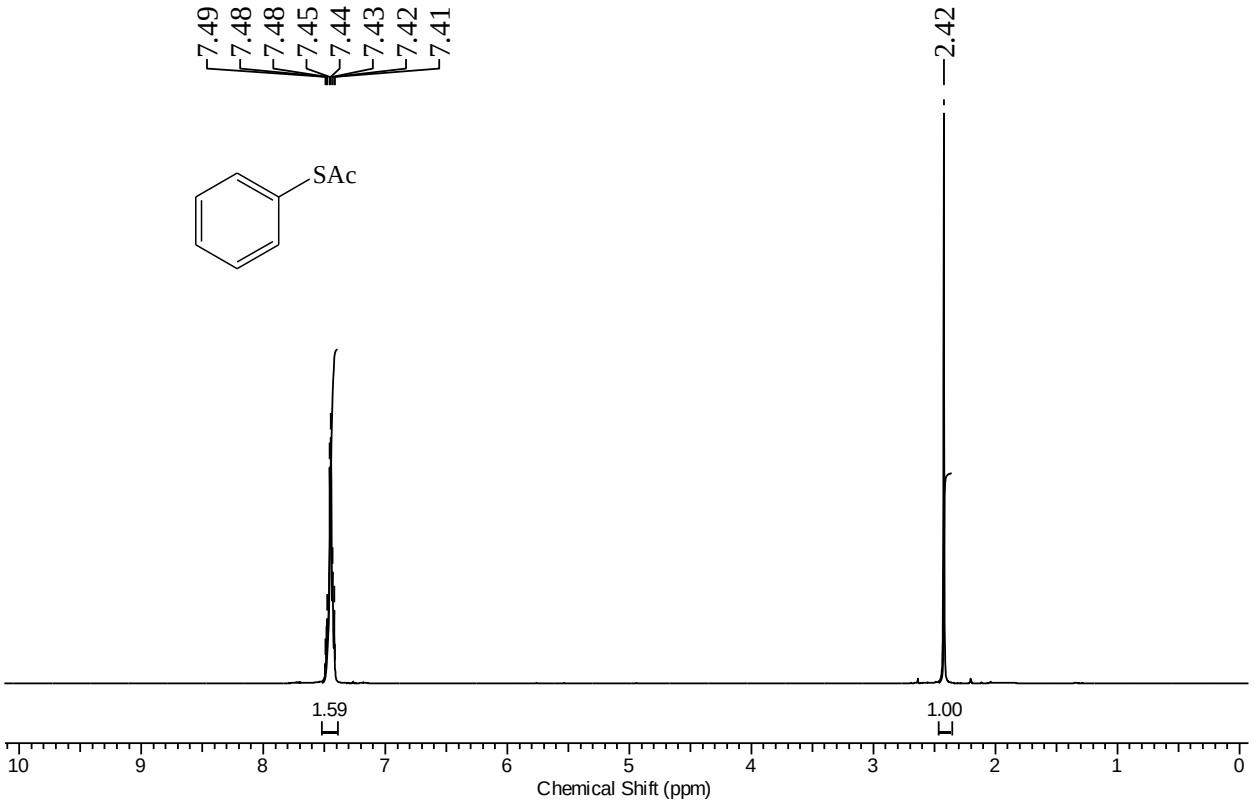


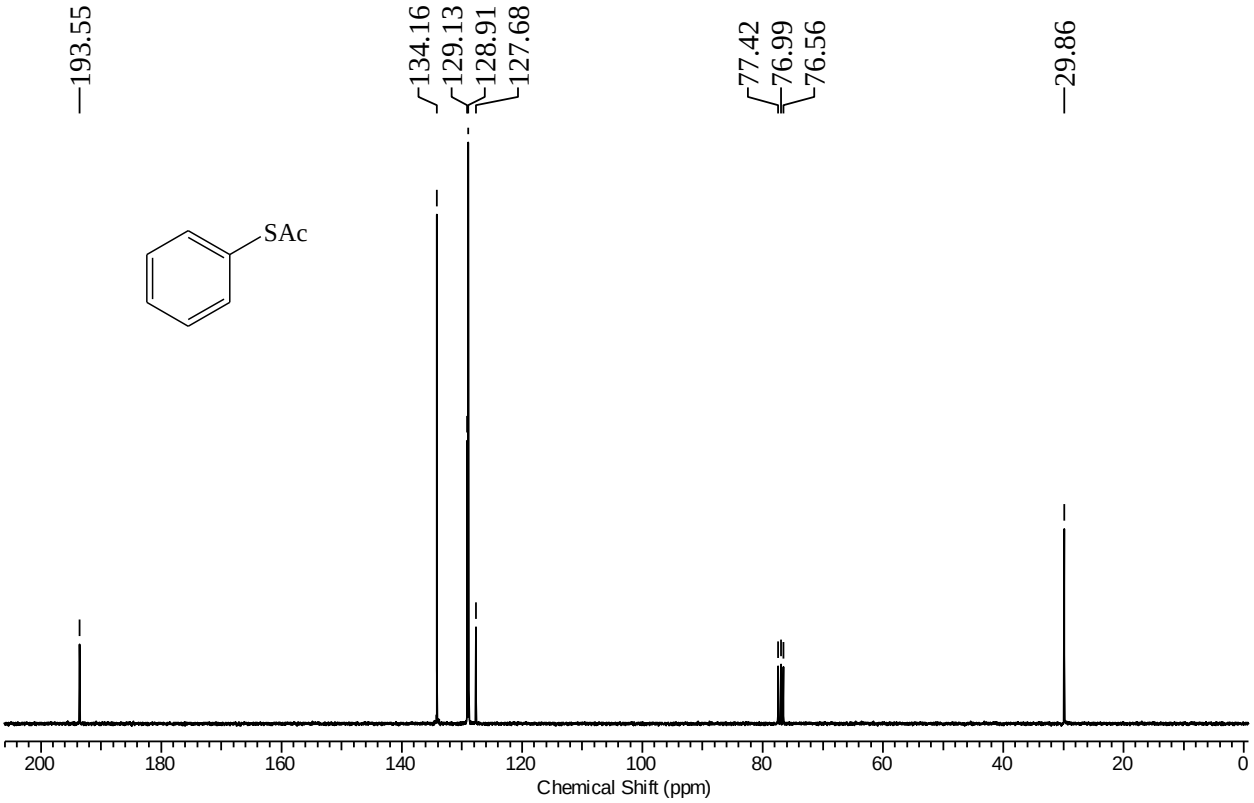


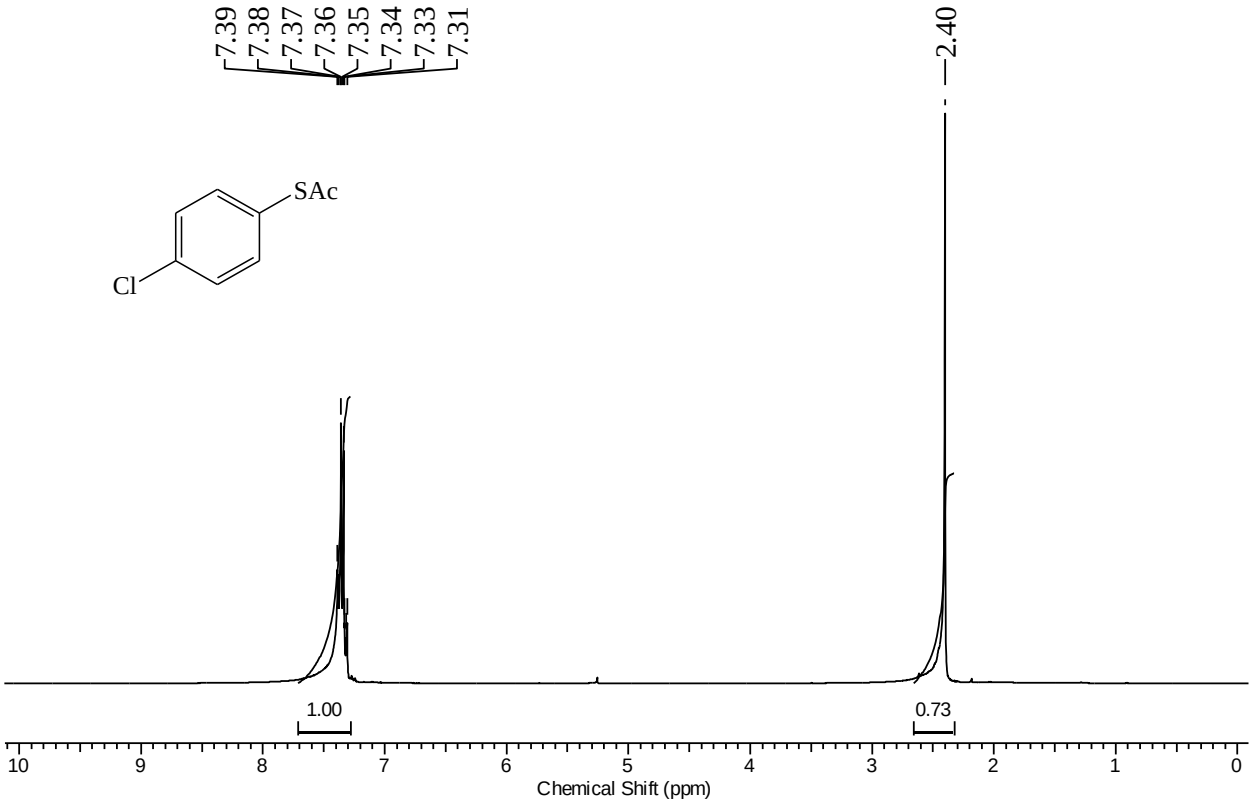


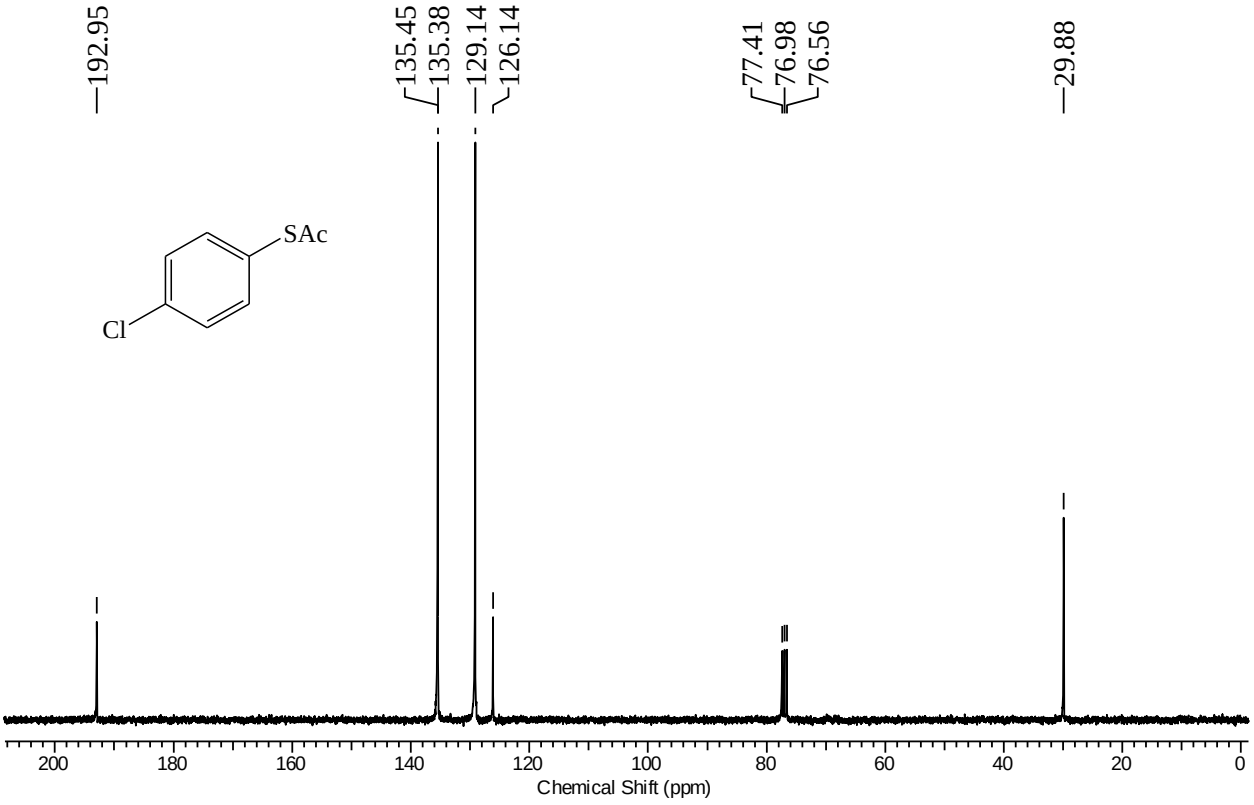


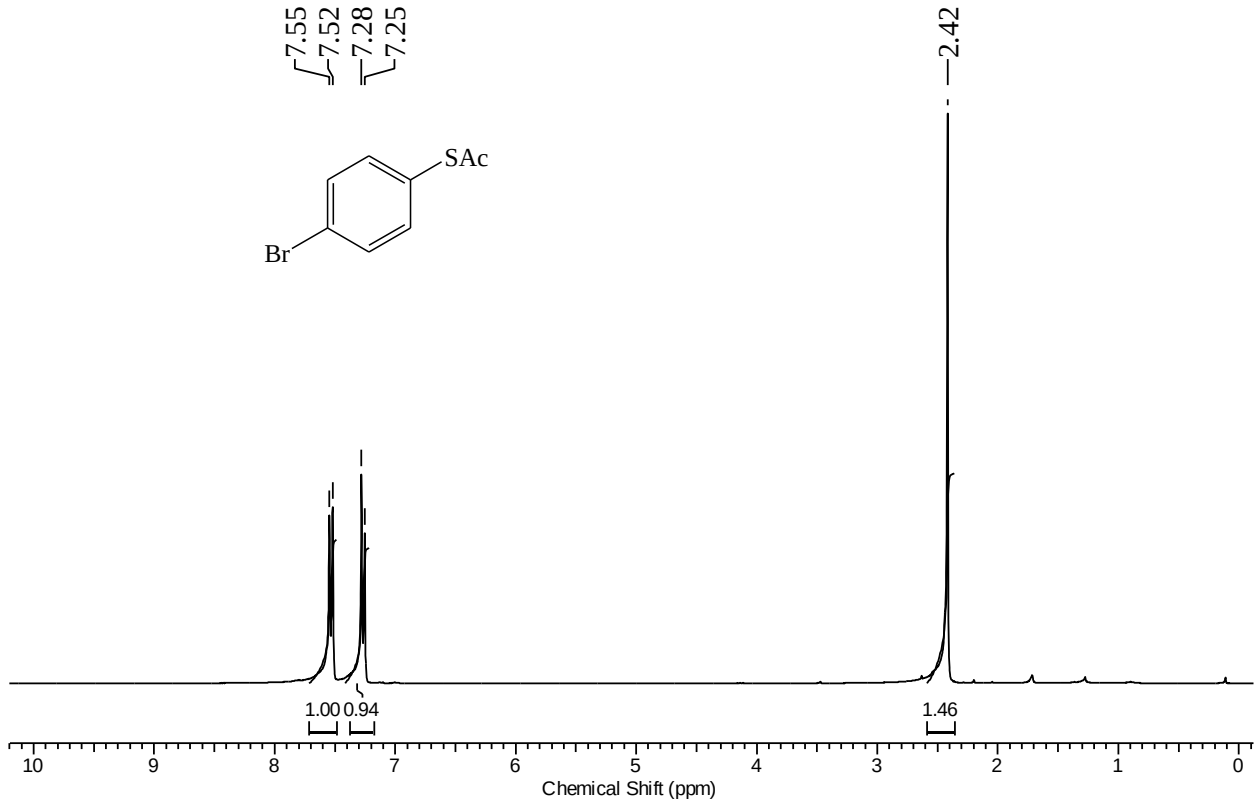


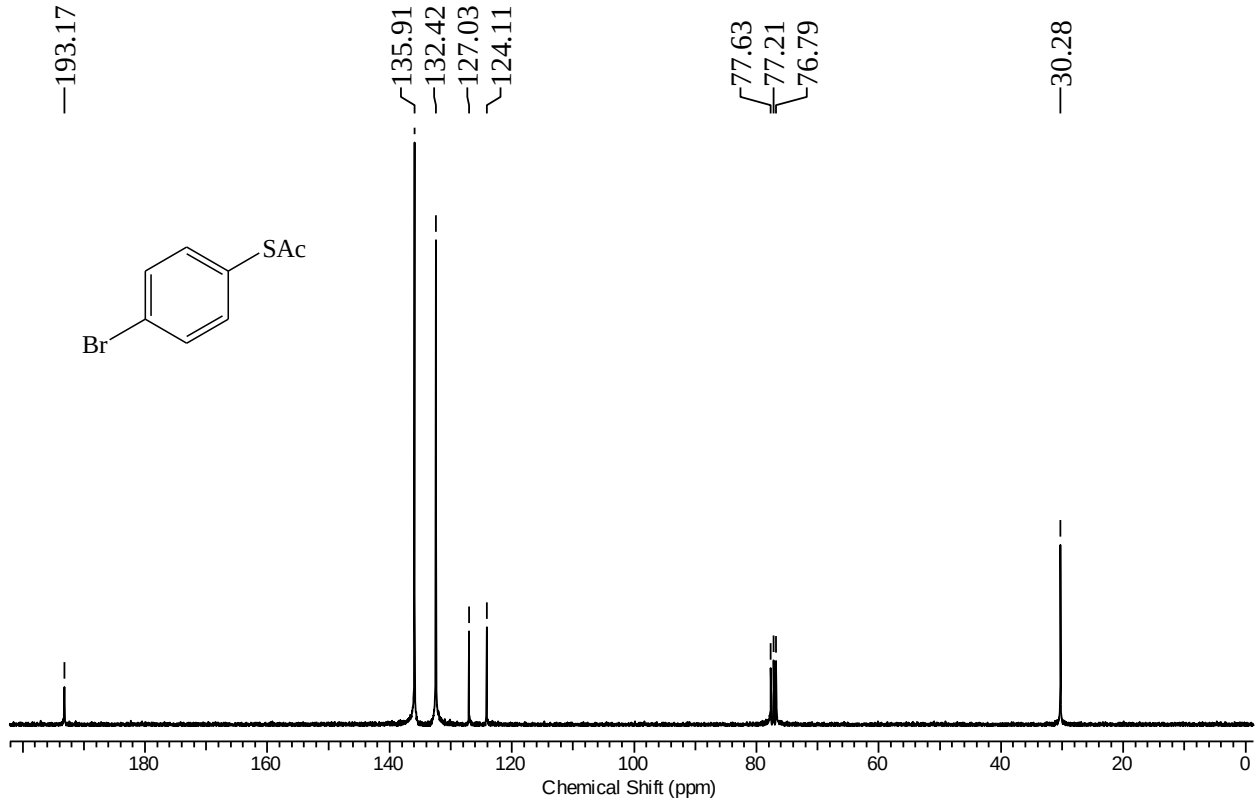


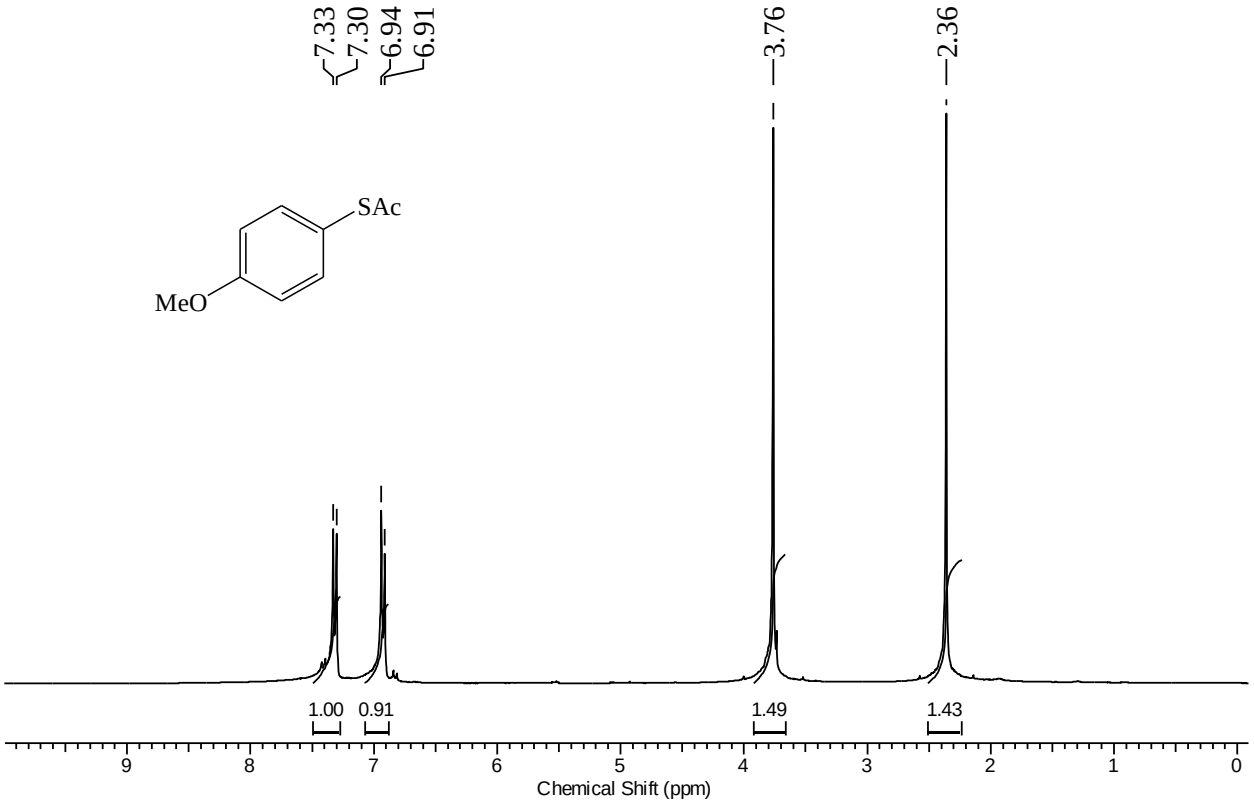


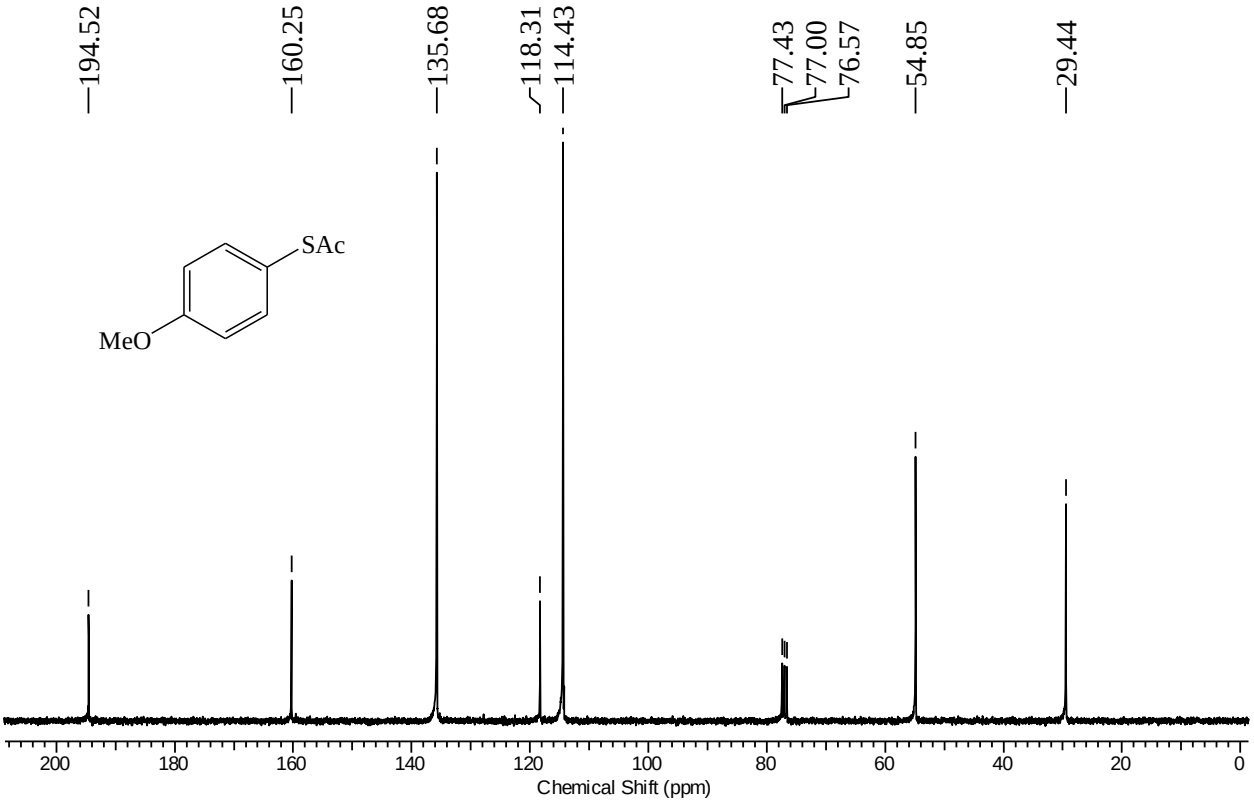


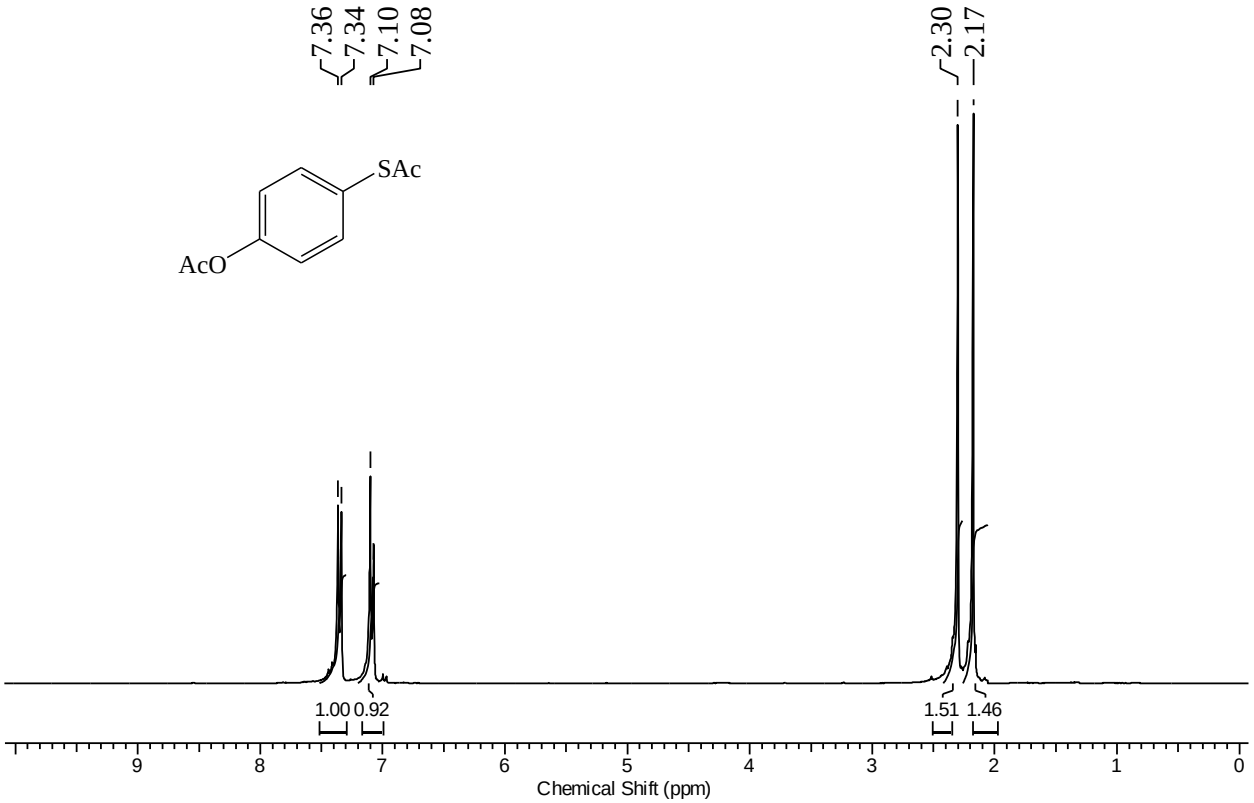


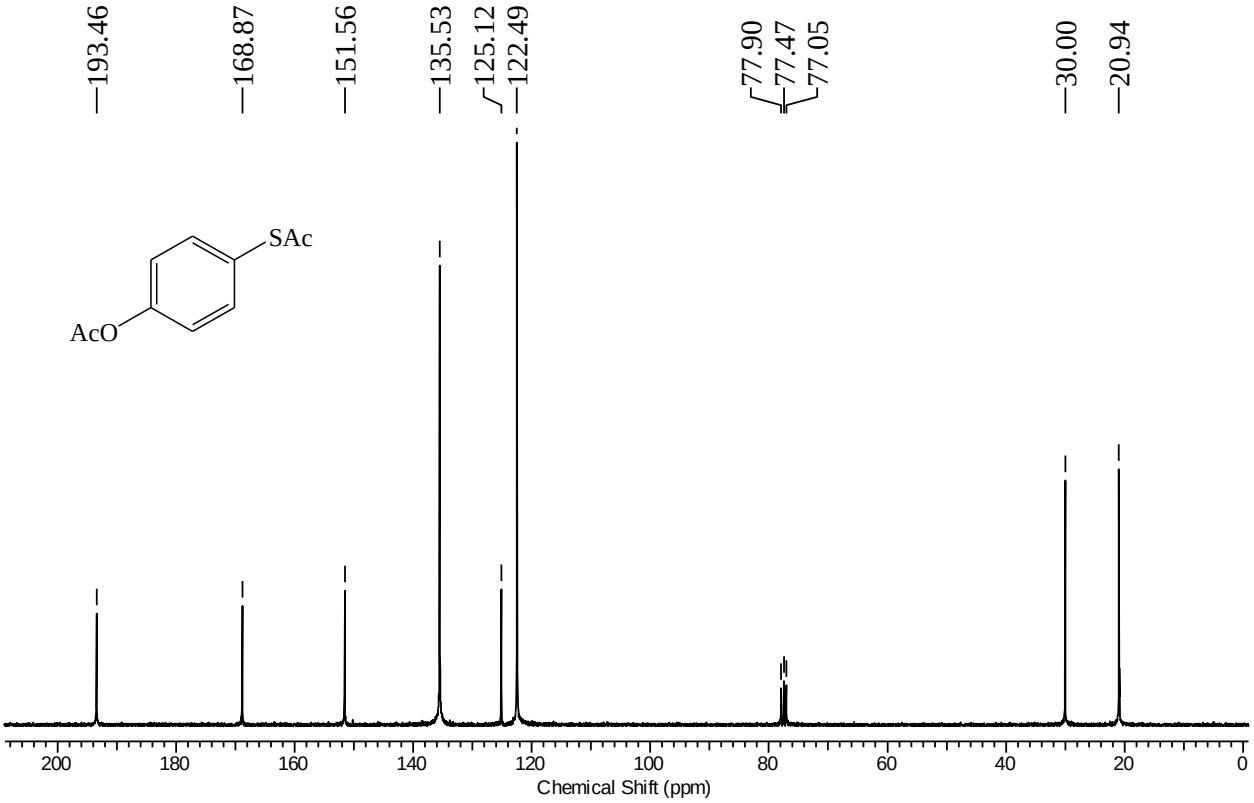


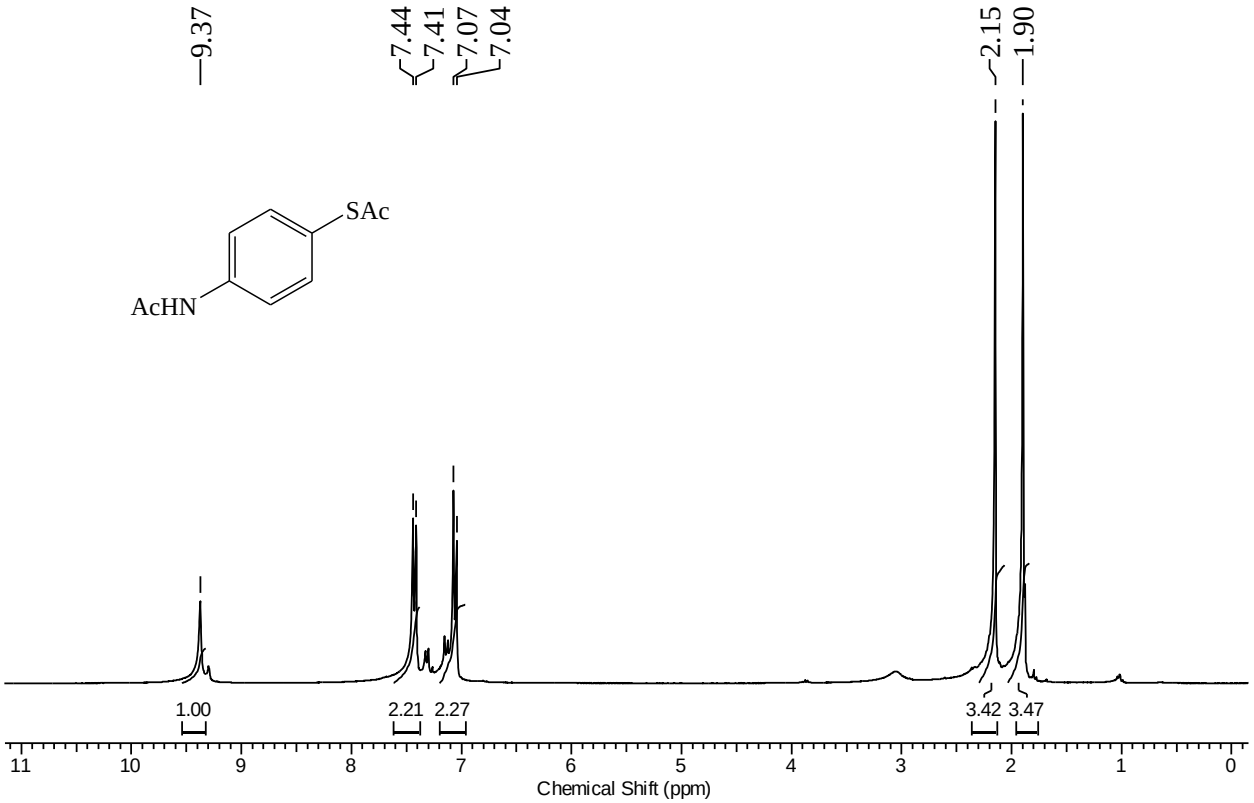


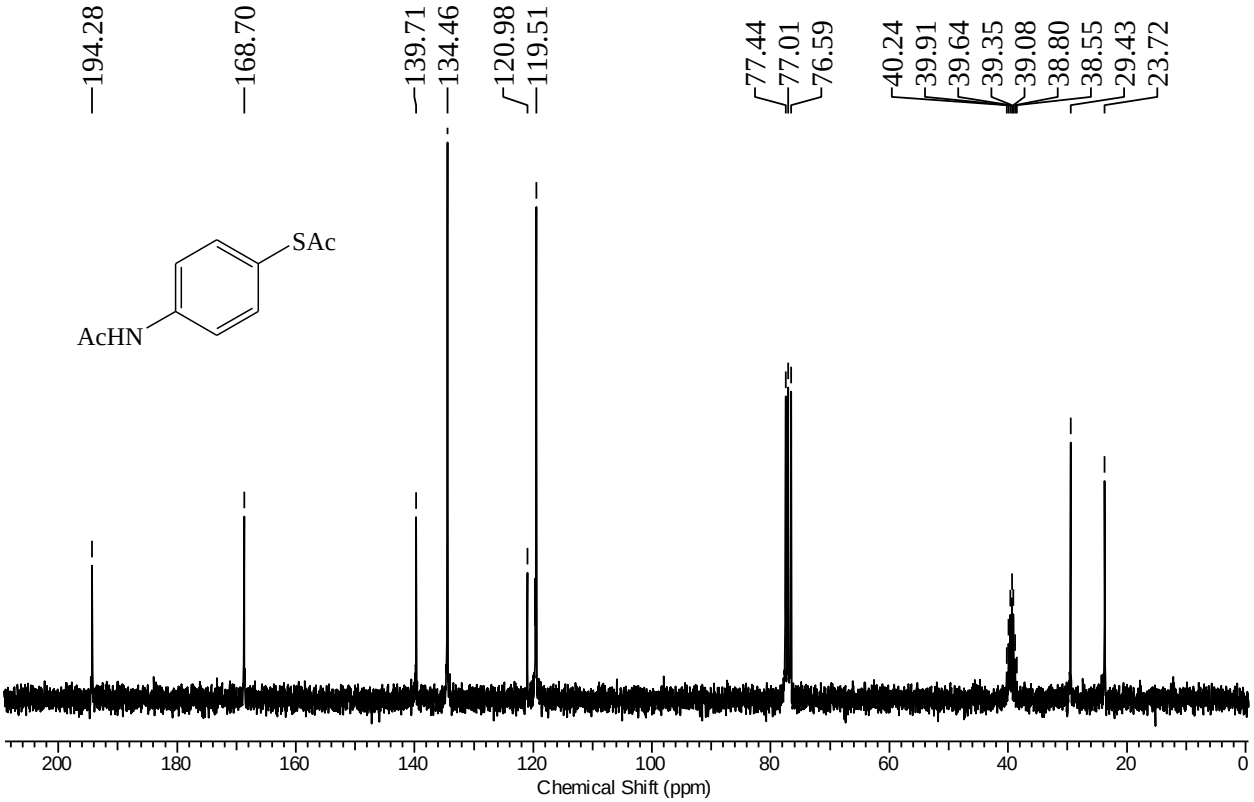


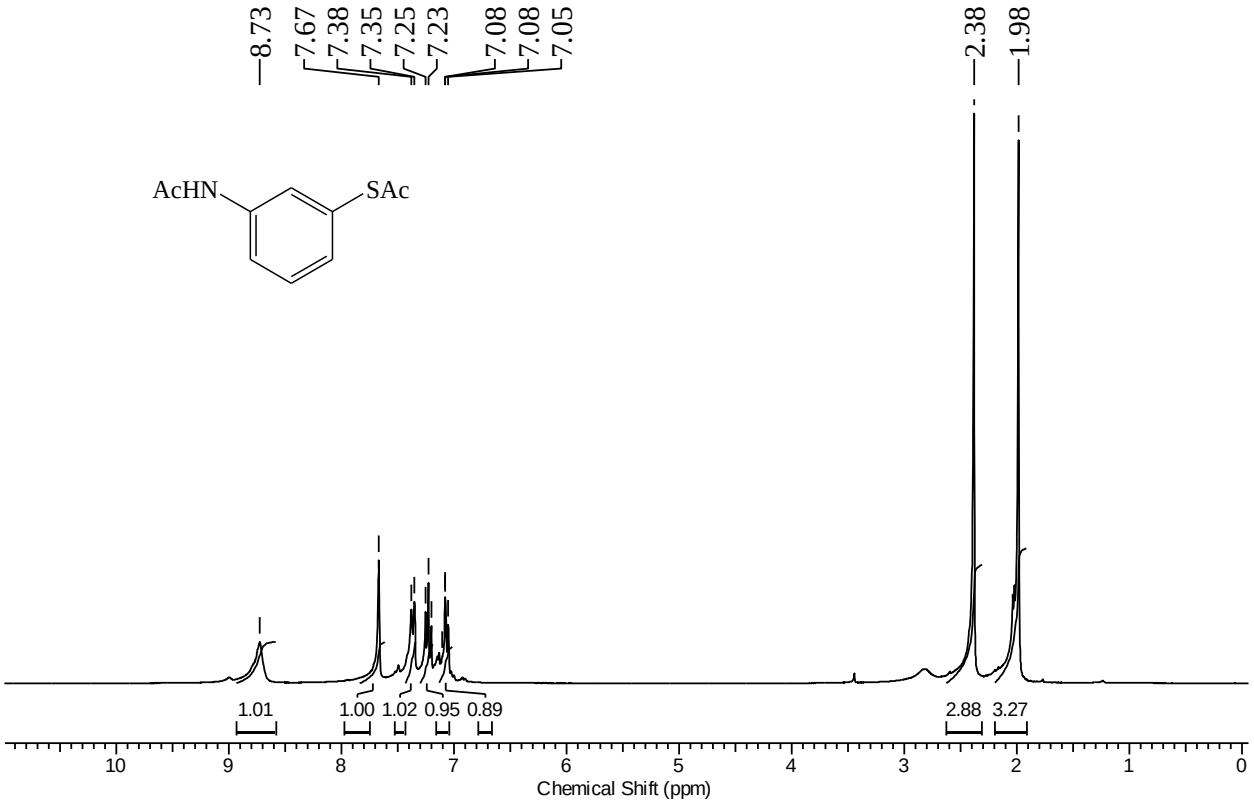


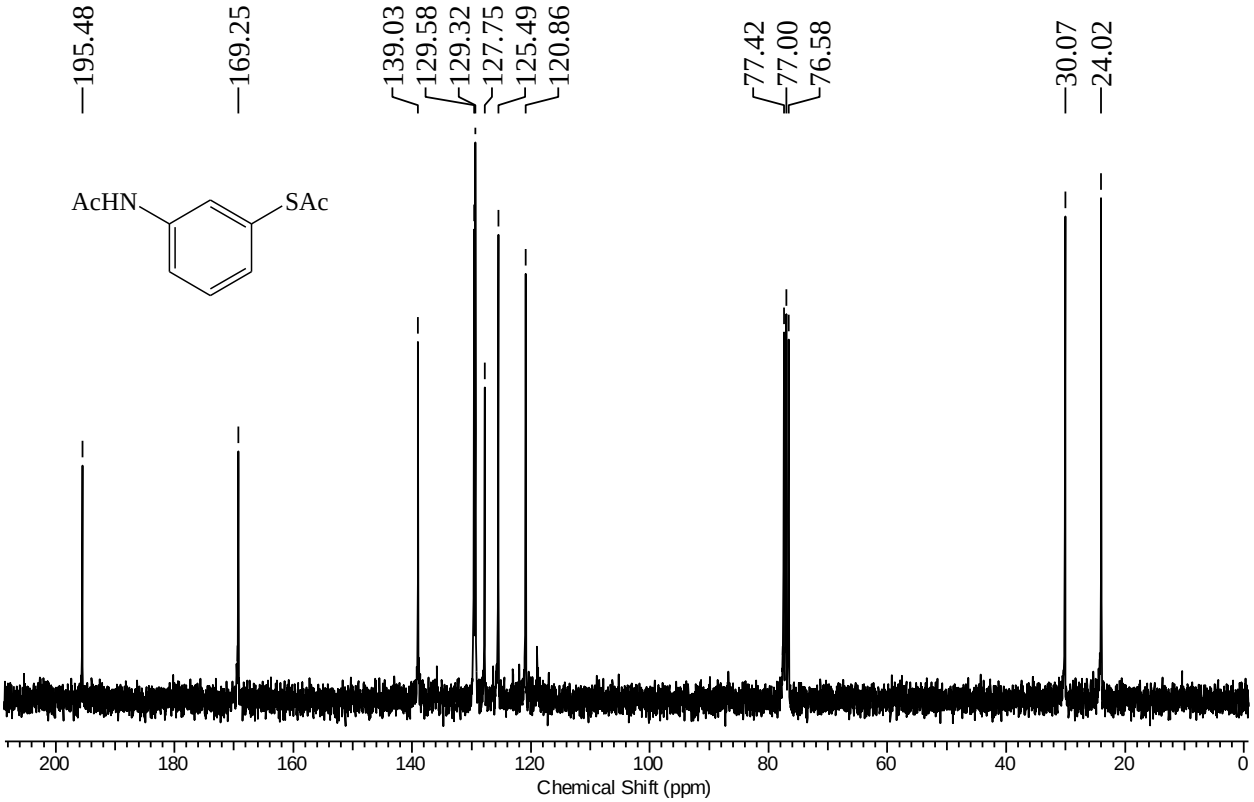


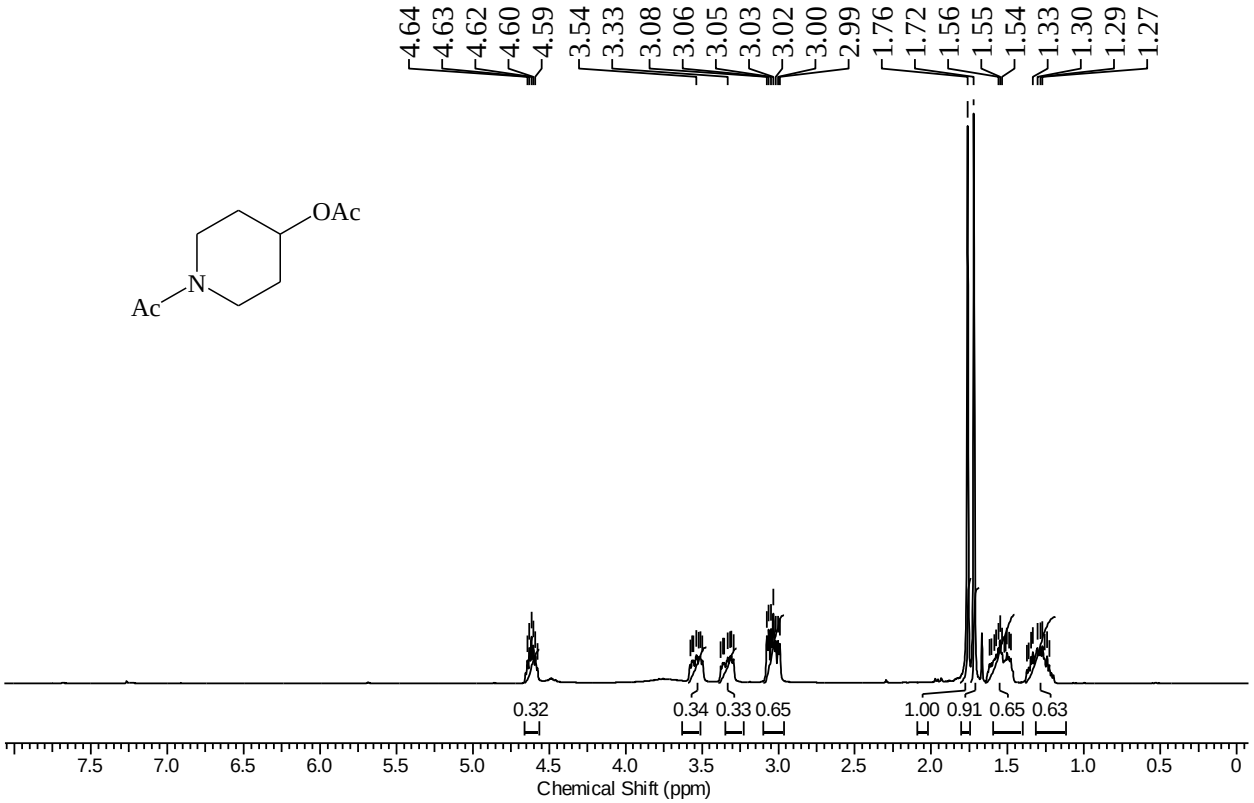


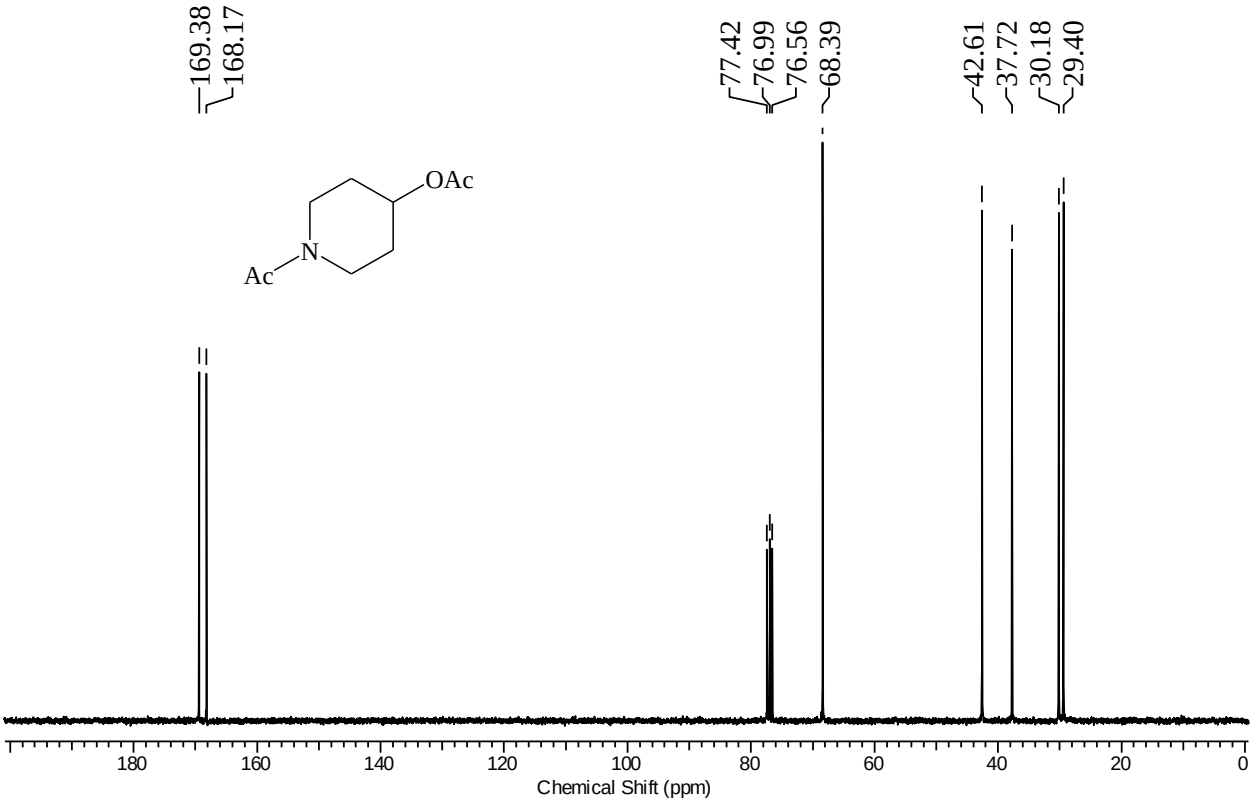




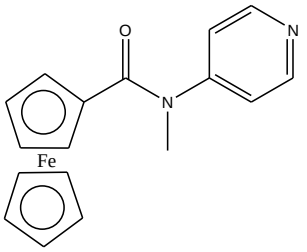






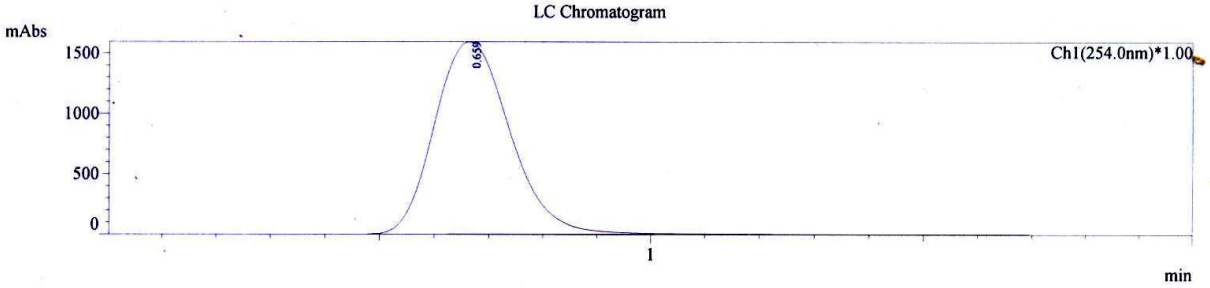


LC-MS of Compound I



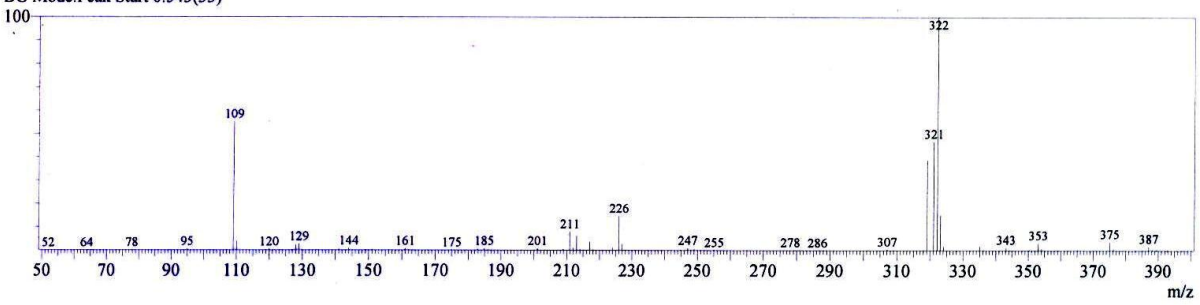
User	: Admin
Sample	: BMAP1
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\BMAP1-APCI-POS1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

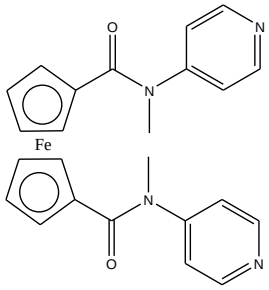
Line#:1 R.Time:0.827(Scan#:50) Positive
MassPeaks:192 BasePeak:322.10(10279617)
RawMode:Single 0.827(50)
BG Mode:Peak Start 0.543(33)



MS Peak Table

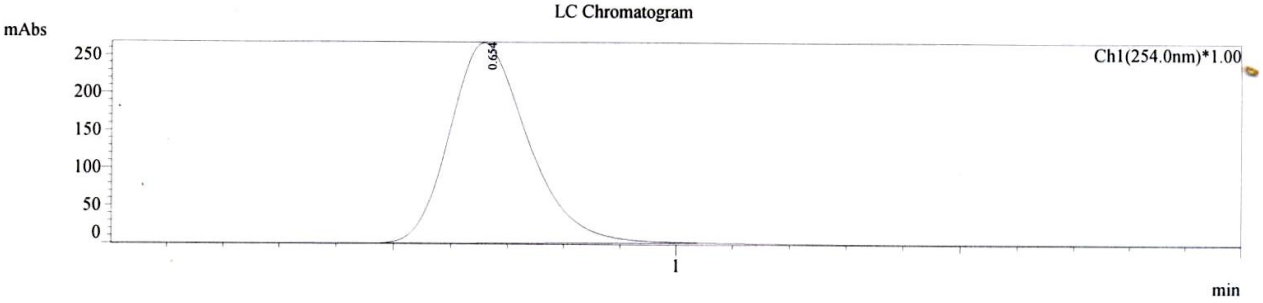
Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.827	0.543	1.143	722595345	36528405	19.78		100.00		322.10	10279617
				722595345	36528405			100.00			

LC-MS of Compound II



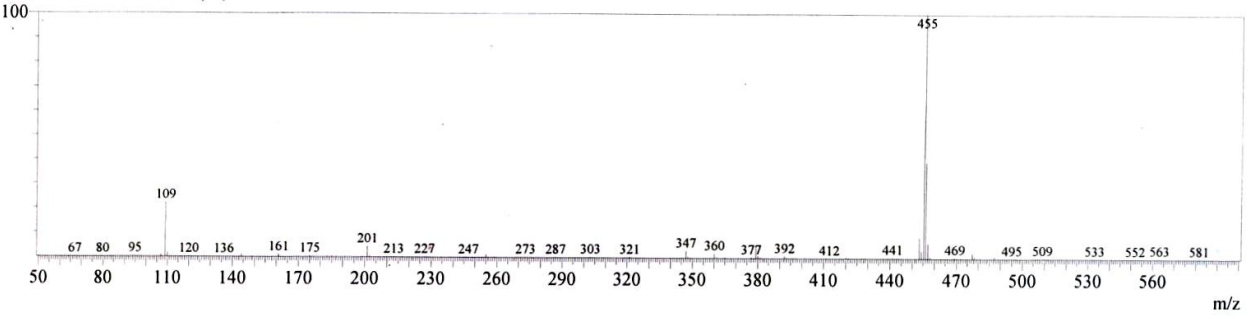
User	: Admin
Sample	: BMAP2
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\BMAP2-APCI-POS1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



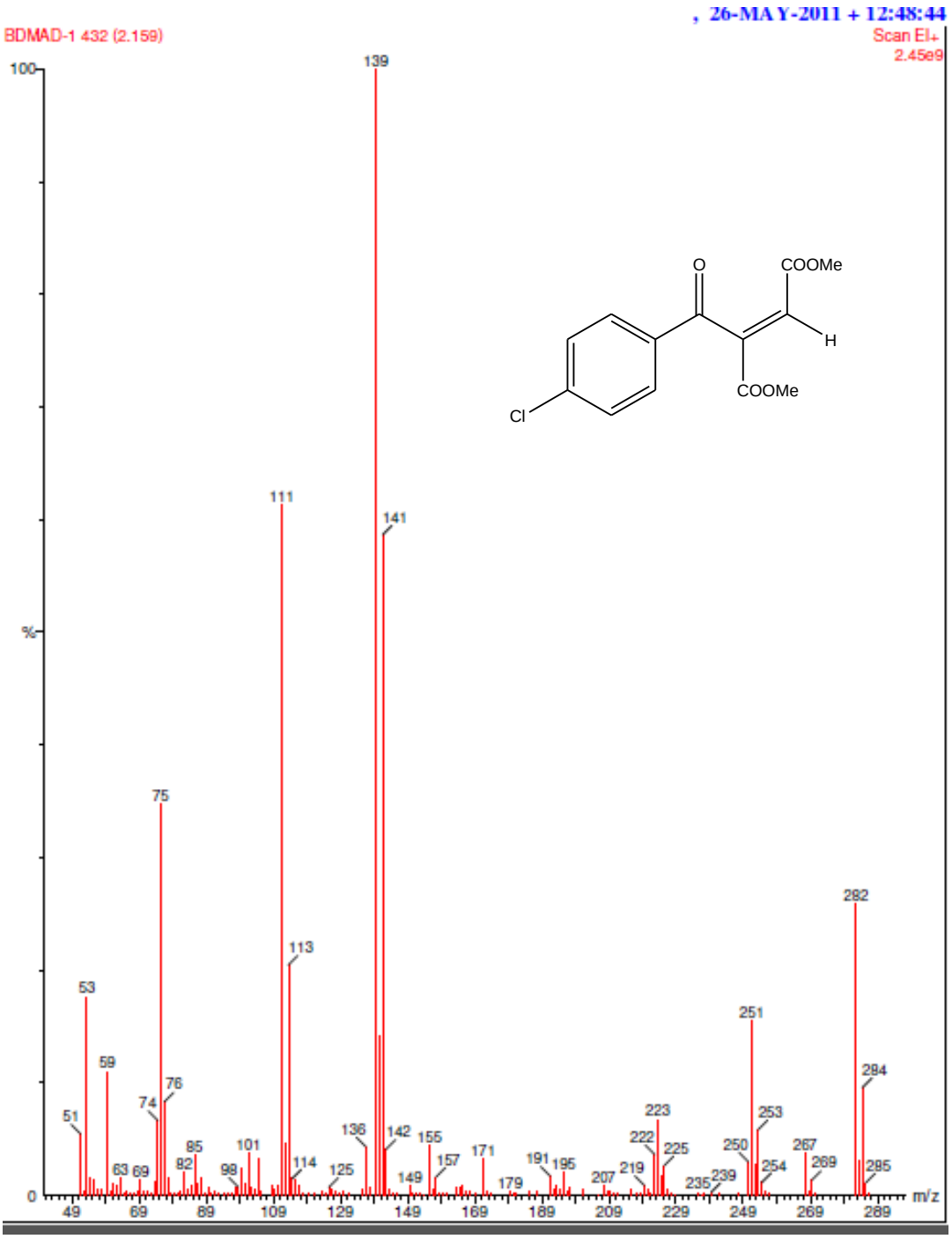
MS Spectrum

Line#:1 R.Time:0.758(Scan#:46) Positive
MassPeaks:378 BasePeak:455.15(10984034)
RawMode:Single 0.758(46)
BG Mode:Peak Start 0.560(34)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.758	0.560	1.043	294824113	21849024	13.49		100.00		455.15	10984034
				294824113	21849024			100.00			


, 26-MAY-2011 + 12:48:44
BDMAD-1 432 (2.159)
Scan EI+
2.45e9
100
%
0
O
COOMe
H
COOMe
Cl
m/z
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109
129
149
169
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209
229
249
269
289
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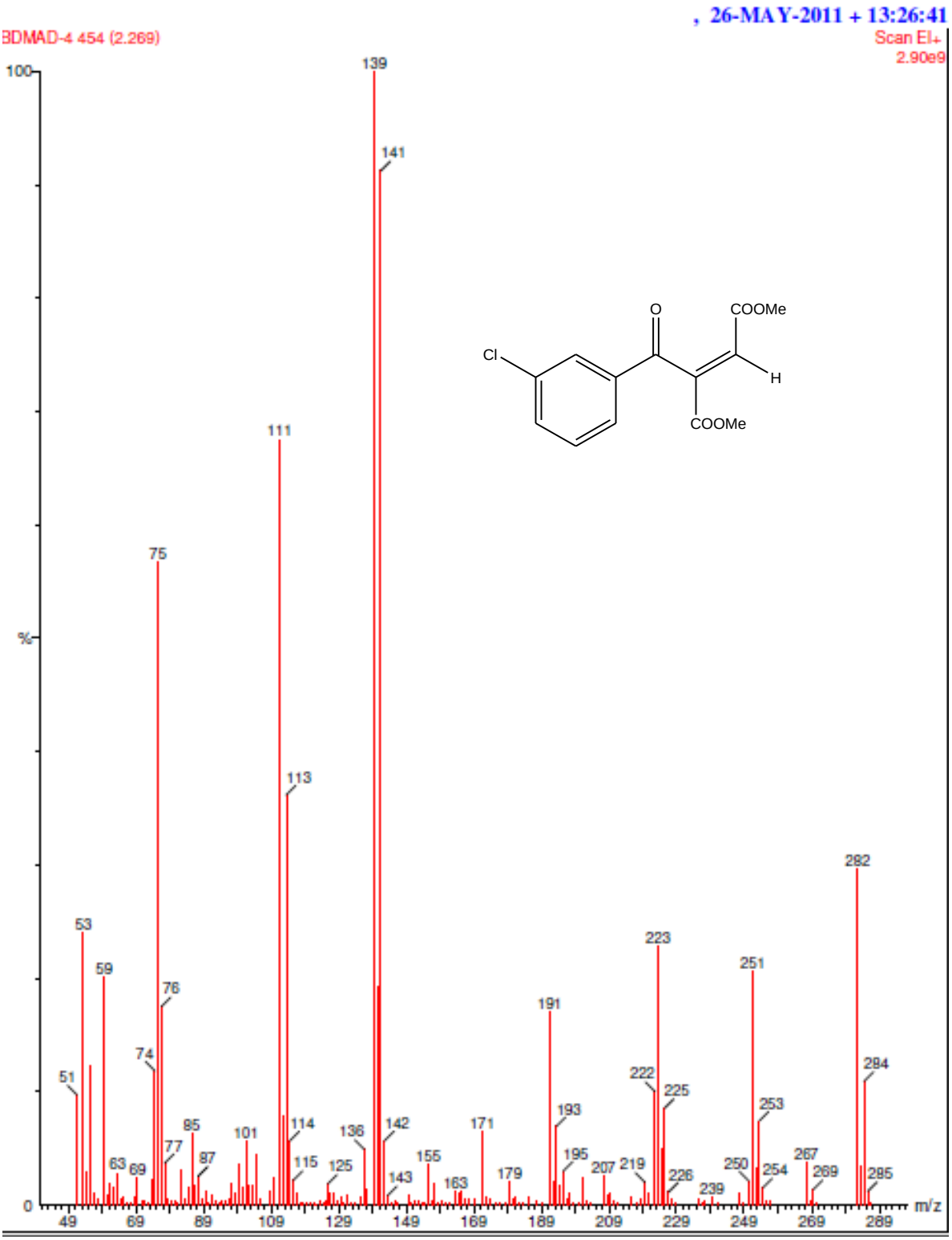

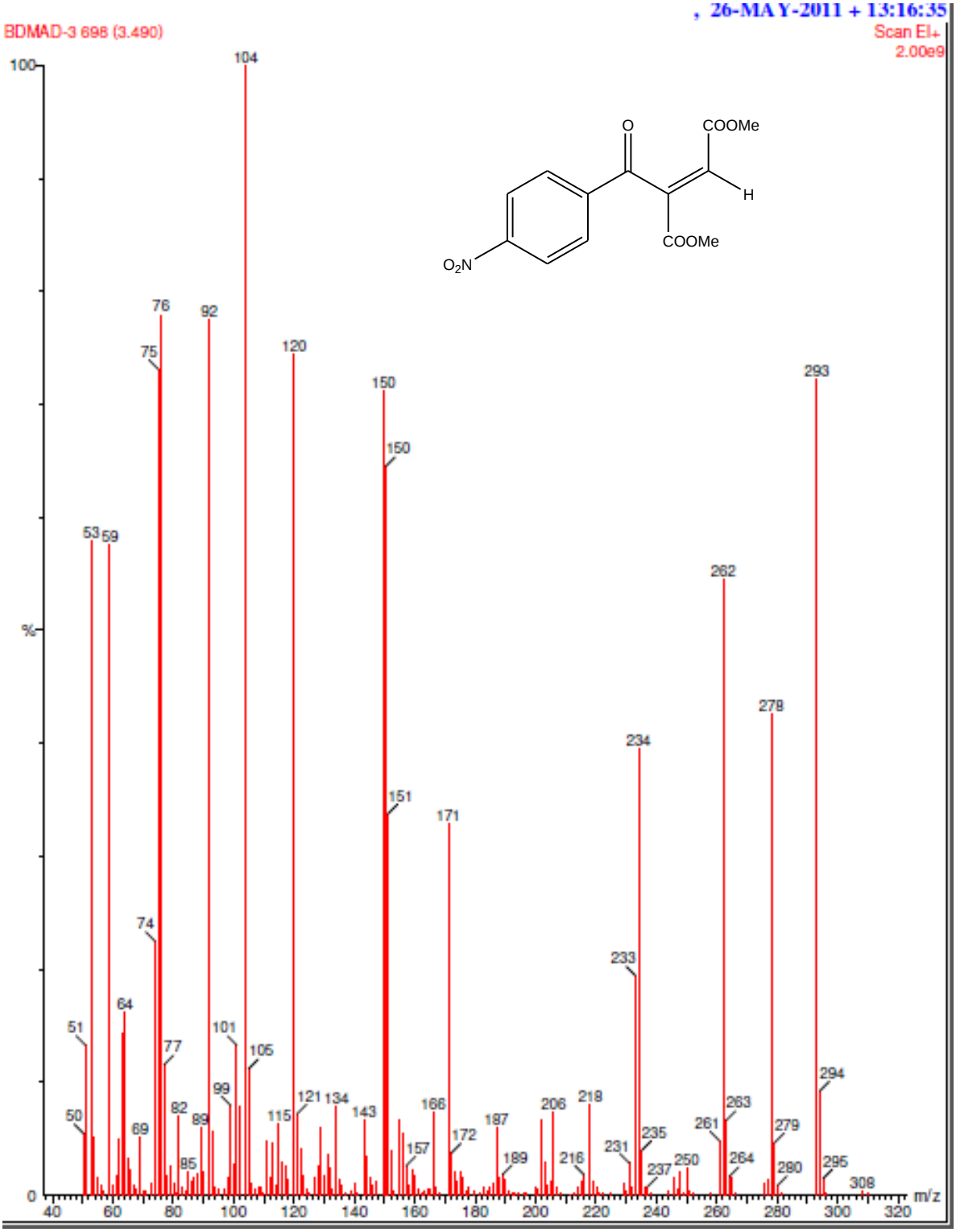

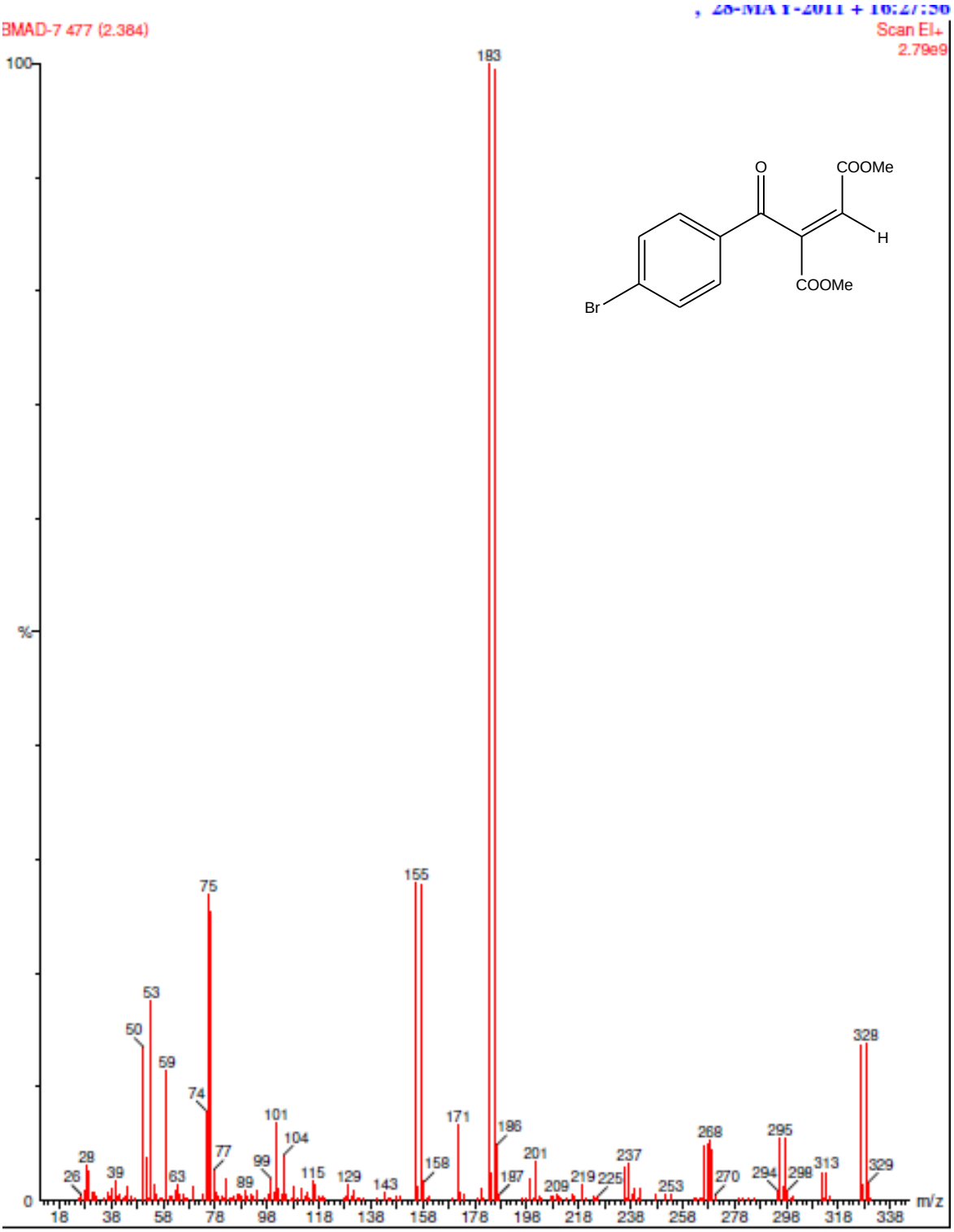
BMAD-7 477 (2.384)
Scan EI+
2.79e9
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m/z
O
COOMe
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COOMe
Br
183
155
75
53
50
59
74
101
104
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99
89
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158
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201
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268
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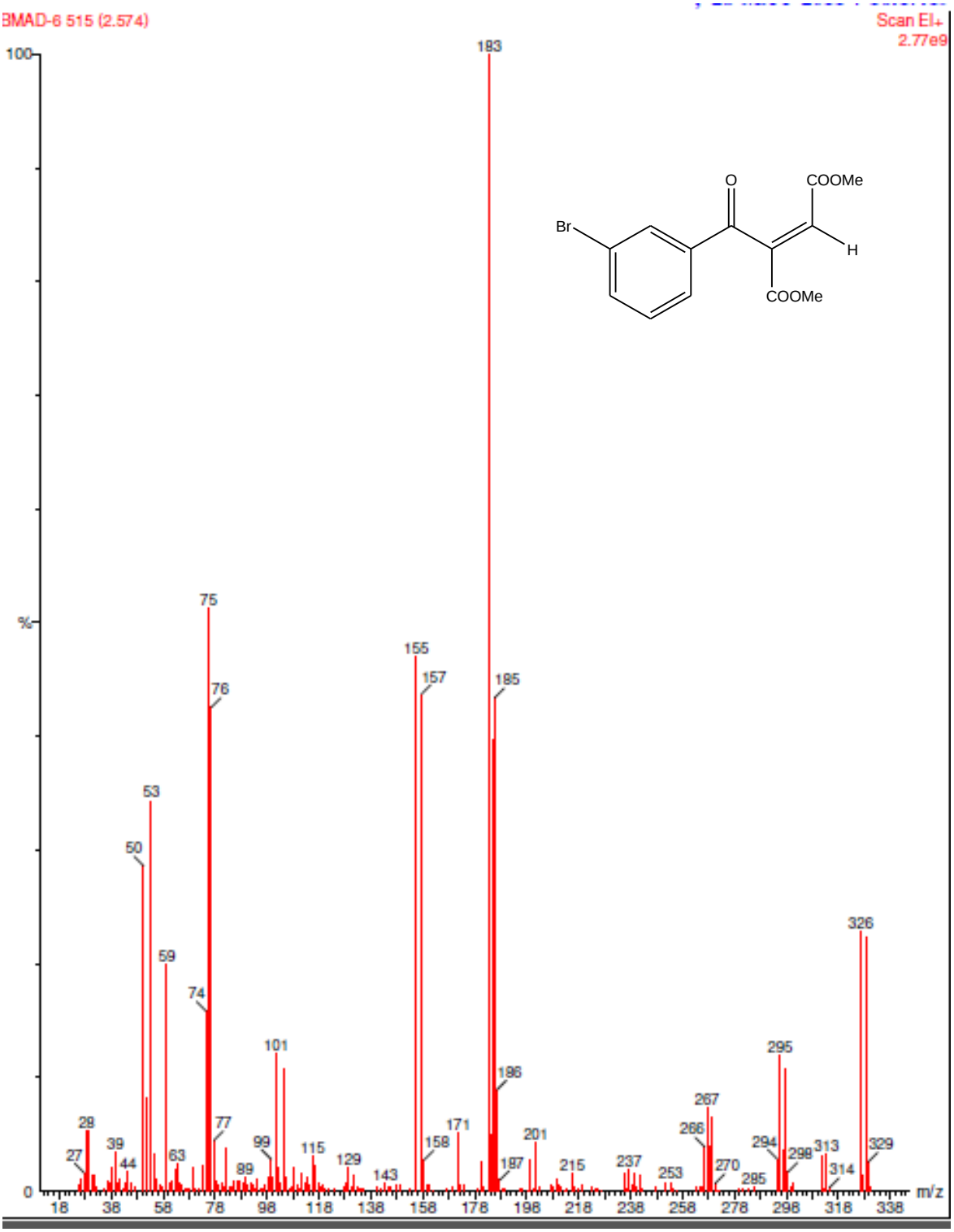

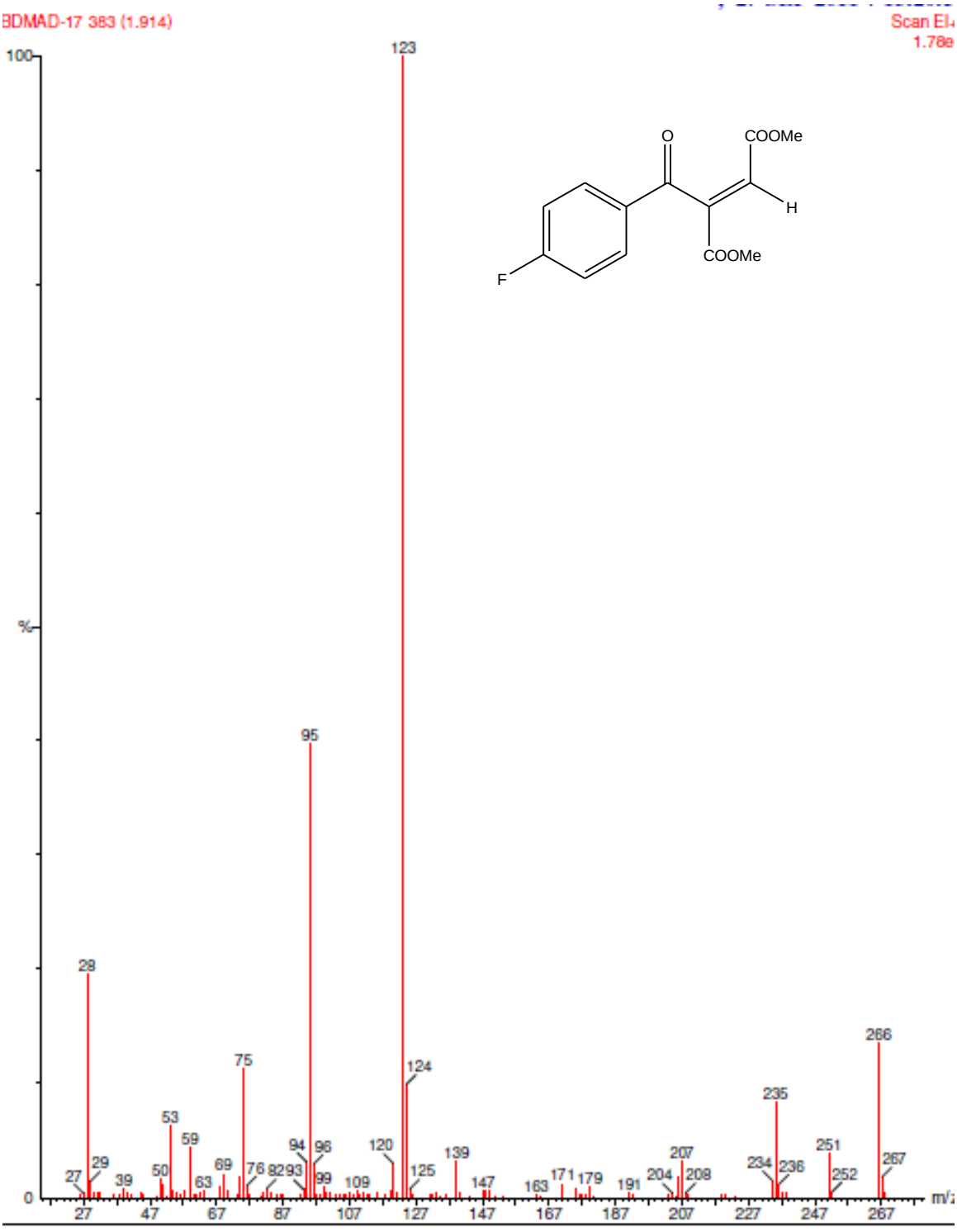

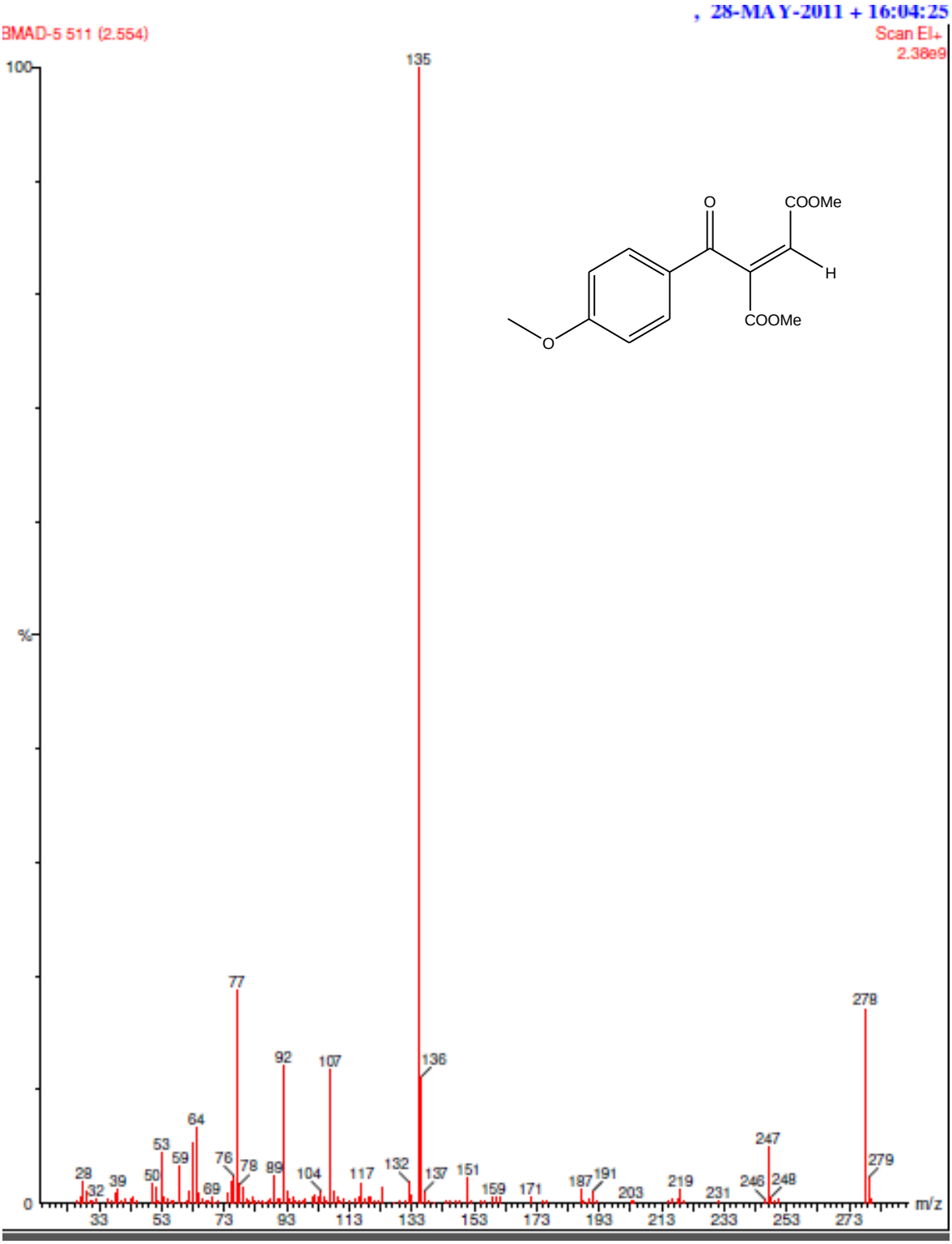
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%
0
135
O
COOMe
H
COOMe
O
77
92
107
136
278
64
53
59
76
78
89
104
117
132
137
151
159
171
187
191
203
219
231
246
247
248
279
28
32
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m/z
33
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253
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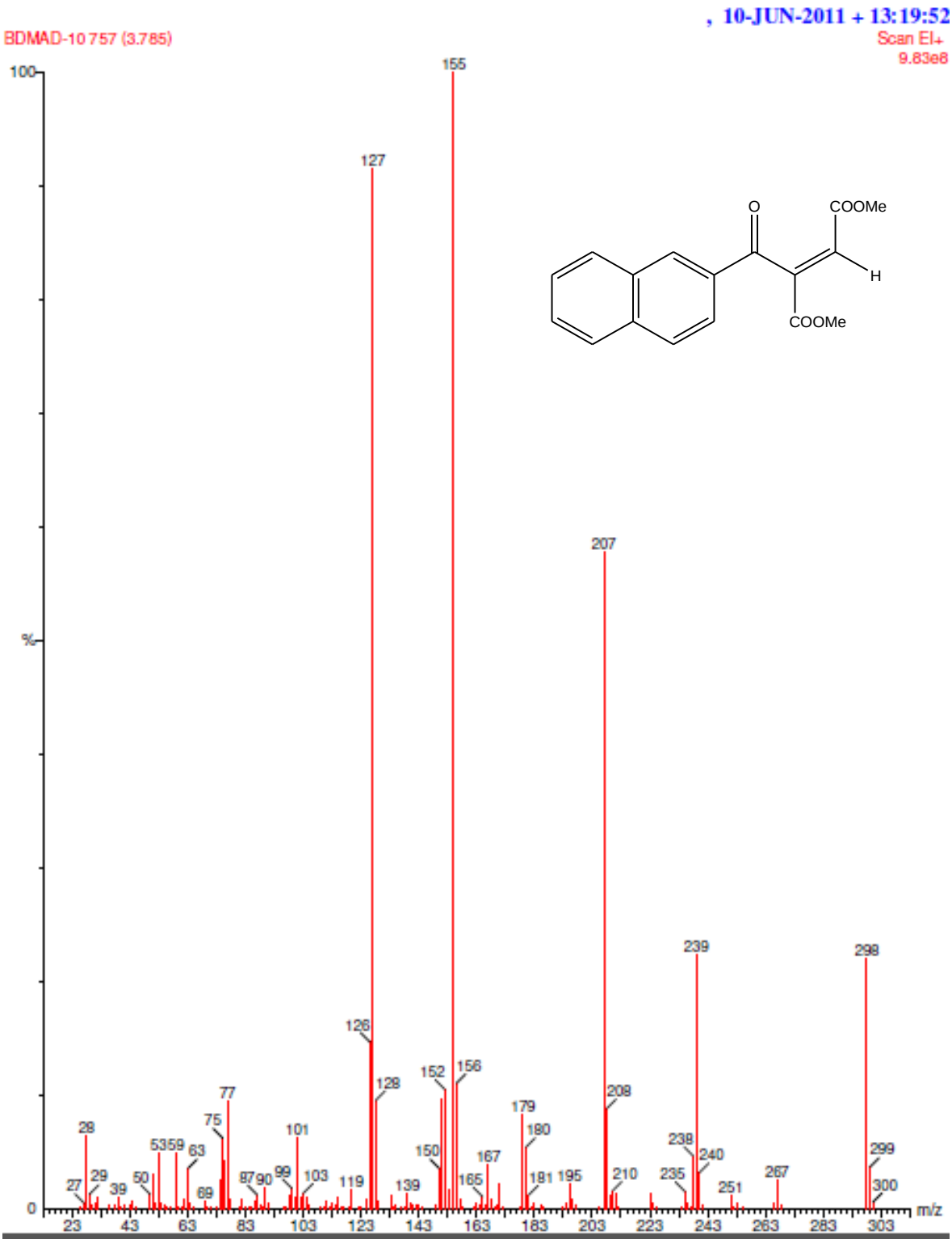

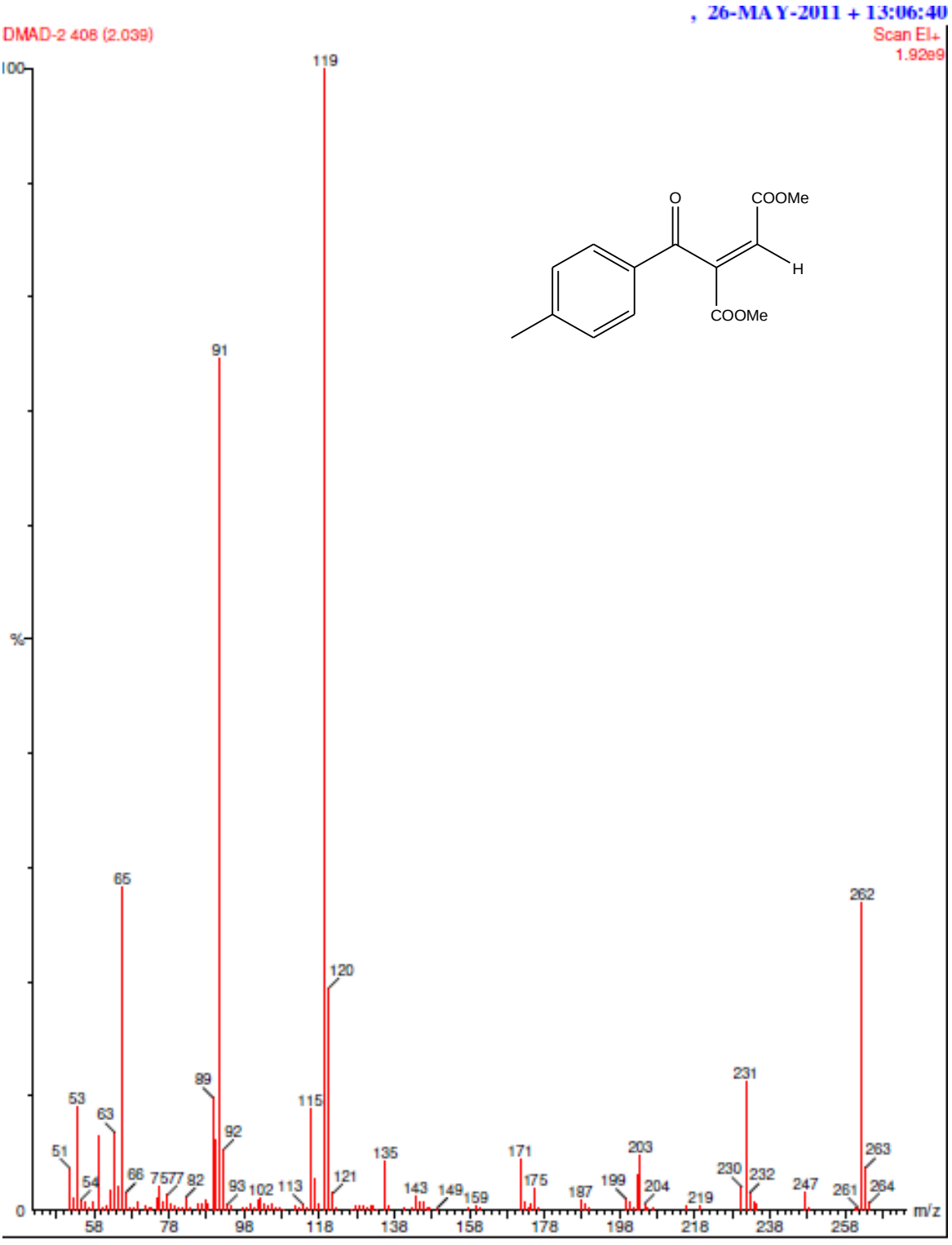
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COOMe
H
COOMe
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91
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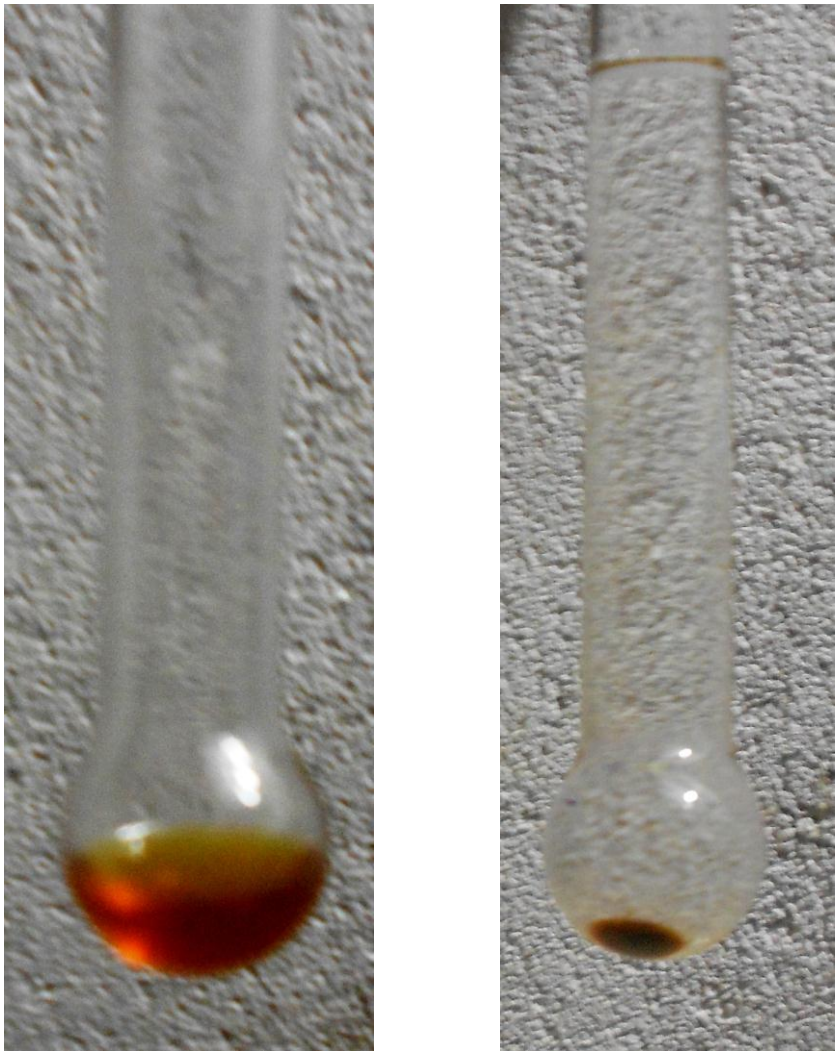
120
262
89
231
53
115
63
92
135
171
203
263
51
230
232
66
75
77
82
121
143
149
159
175
187
199
204
219
247
261
264
93
102
113
m/z
58
78
98
118
138
158
178
198
218
238
258

Table S1: Crystallographic Data of *N*-methyl-*N*-(pyridin-4-yl)ferroceneamide (**I**) and *N,N´*-dimethyl-*N,N´*-di(pyridin-4-yl)ferrocene-1,1´-dicarboxamide (**II**)

empirical formula	$C_{17}H_{16}N_2OFe$	$C_{24}H_{22}N_4O_2Fe$
formula weight	320.17	454.31
crystal system	Orthorhombic	Monoclinic
space group	*P*na2(1)	*C*2/c
T (K)	298	298
a/Å	20.791(11)	28.447(9)
b/Å	5.874(3)	6.2758(19)
c/Å	11.646(6)	12.914(4)
α/deg	90.00	90.00
β/deg	90.00	116.647(7)
γ/deg	90.00	90.00
V/Å^3	1422.2(13)	2060.6(11)
D_{calcd} (g cm^{-3})	1.495	1.464
μ (mm^{-1})	1.060	0.762
Z	4	4
reflns collected	13592	10015
unique reflns	2766	1994
observed reflns	2636	1878
R_1 [I > 2 σ(I)]	0.0335	0.0366
wR2 (all)	0.0733	0.0946
goodness-of-fit	1.100	1.118
diffractometer	SMART Bruker Apex -II	SMART Bruker Apex -II

(a) (b)

Figure S1: (a) catalyst **I** in DCM (b) precipitate of catalyst **I** after addition of petroleum ether

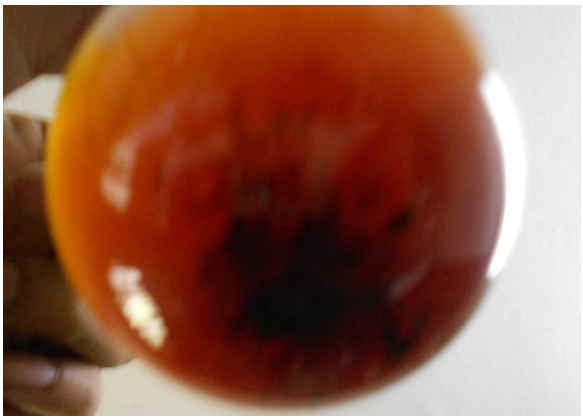


(a) (b)

Figure S2: (a) catalyst **II** in DCM (b) precipitate of catalyst **II** after addition of petroleum ether

(a)

(b)



(c)

Figure S3: (a) reaction before the addition of the catalyst **II** (b) reaction after the addition of the catalyst **II** (c) Precipitation of the catalyst from the reaction mixture.

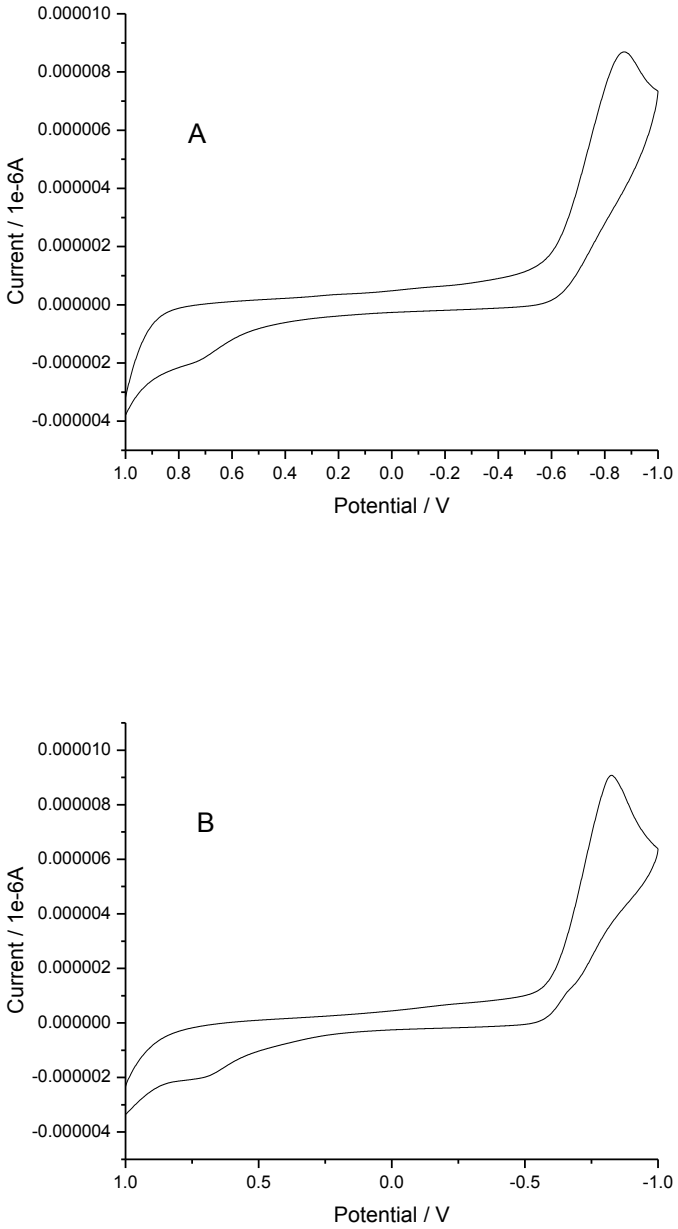


Figure S4: Cyclic voltammetry in DMSO of (A) Dimethyl (2E)-2-(4-bromobenzoyl)but-2-enedioate (5b) (B) Reaction mixture after removal of catalyst II. Condition for CV: Glassy Carbon working electrode, Ag-AgCl (3M solution) as reference and Pt wire grid as counter electrode. Scan rate: 100 mV/sec.

(Figure S4 (B) indicates the absence of catalyst II in the reaction mixture after catalyst separation.)

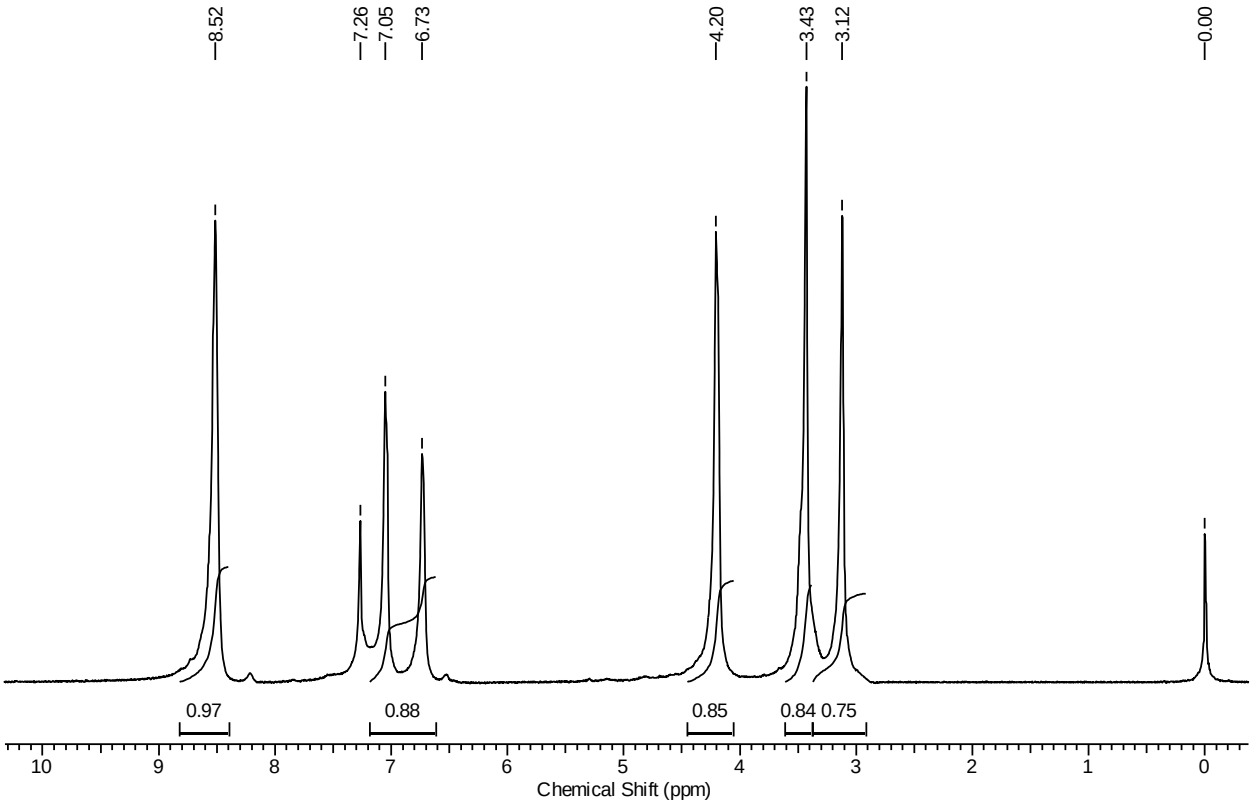


Figure S5: 1H NMR of recovered catalyst II after reaction.