SUPPLEMENTARY MATERIAL

Advantageous Use of $^{t}Bu_2P$-N=P($^{i}BuPCH_2CH_2)_3N$ in the Hiyama Coupling of Aryl Bromides and Chlorides

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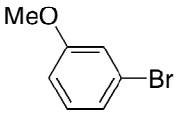
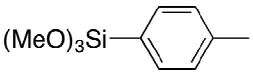
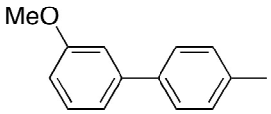
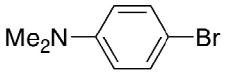
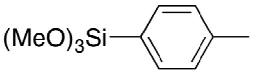
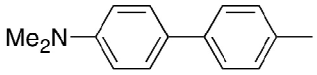
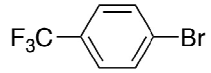
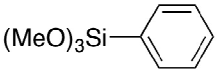
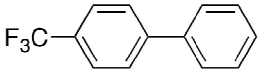
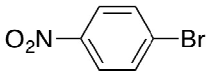
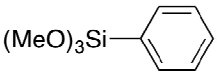
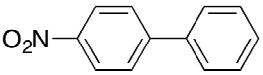
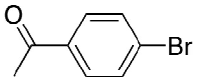
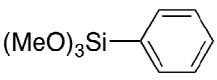
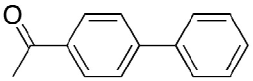
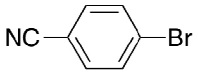
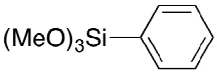
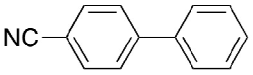
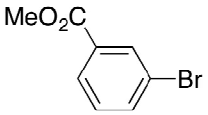
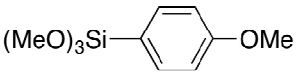
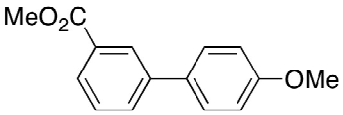
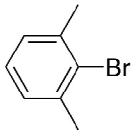
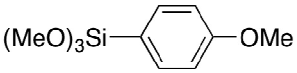
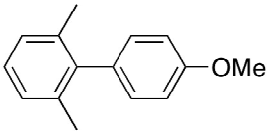
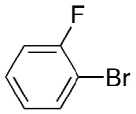
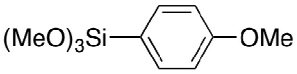
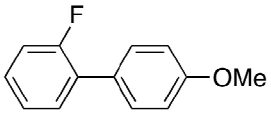
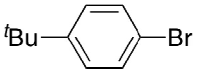
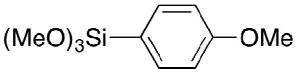
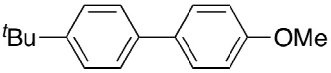
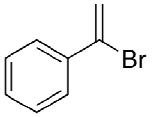
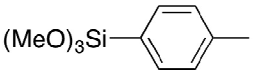
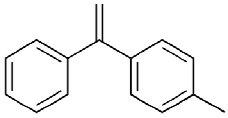
Experimental Section:

General Considerations. All reactions were carried out under inert atmosphere using standard Schlenk procedures. Dioxane was refluxed at 130 °C over activated molecular sieves and distilled, and the distillate was stored over activated molecular sieves under argon. Aryl bromides and chlorides were purchased from Aldrich chemical company and used without further purification. Phenylsiloxanes were purchased from Gelest and used without further purification. Palladium, ligand, and tetrabutylammonium fluoride (TBAF•xH_2O) were stored in a glove box. Ligands **1**, **2**, and **3** were prepared according to literature procedures.[1] Ligand **4** is available from Aldrich chemical company. ^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz Bruker DRX in $CDCl_3$. Thin layer chromatography (TLC) was performed using commercial 60 mesh silica gel plates visualized with short-wavelength UV (254 nm) light.

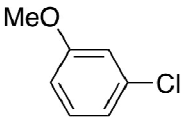
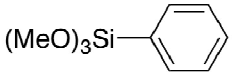
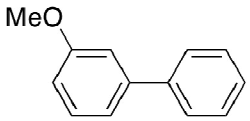
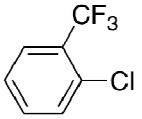
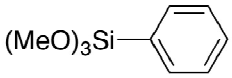
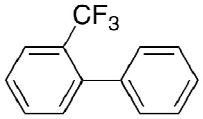
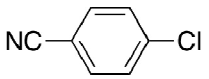
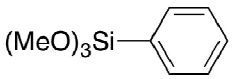
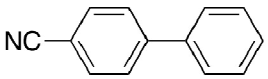
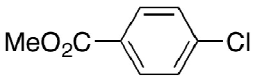
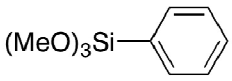
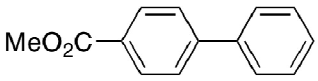
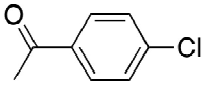
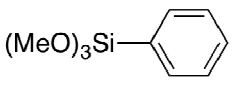
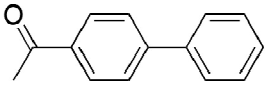
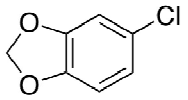
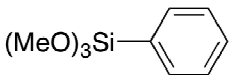
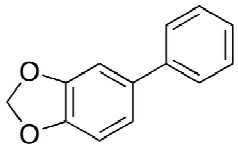
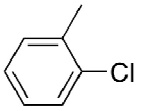
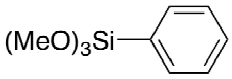
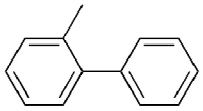
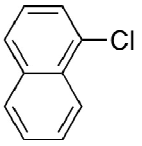
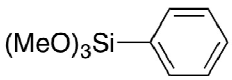
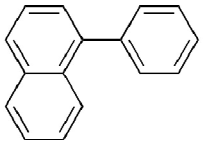
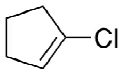
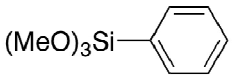
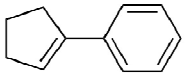
General Procedure for the Hiyama Coupling of Aryl Bromides and Chlorides. A 10 mL vial was charged with 3 mmol TBAF•xH_2O (794 mg), 0.5 mole % $Pd(OAc)_2$ (2.24 mg) and 1.0 mole % ligand **1** (10.03 mg) in a glove box. The vial was capped and brought outside the glove box where 4 mmol of arylsiloxane and 2 mmol of aryl halide were added under an argon flow. The reaction vessel was heated to 80 °C for the specified times given in the tables. The mixtures were then purified by column chromatography (0-5% EtOAc) to obtain pure biaryls.

Table 2. Scope of Aryl Bromides

entry	aryl bromide	siloxane	time	product	yield (%)[a,b]
1			3 h		91[c] (Lit: 80)[h]
2			5 h		71[d]
3			2 h		93[e] (Lit: 87-99)[i]
4			2 h		91[e] (Lit: 51-93)[j]
5			2 h		91[e] (Lit: 55-99)[k]
6			2 h		84[e] (Lit: 85)[l]
7			1.5 h		87[f]
8			2 h		89[g]
9			0.5 h		83[c]
10			0.5 h		85[c]
11			1 h		78[c]

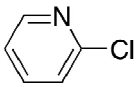
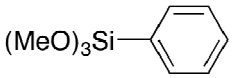
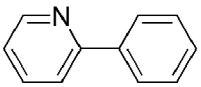
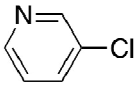
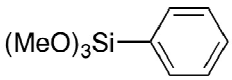
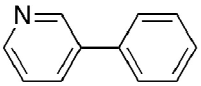
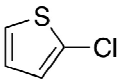
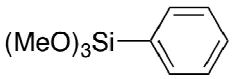
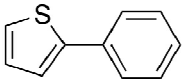
[a]Average of two runs. Yields in correspond to isolated yields. [b]Reaction conditions: 2 mmol aryl bromide, 4 mmol siloxane, 3 mmol TBAF•xH_2O. [c]0.5 mole % $Pd(OAc)_2$, 1 mole % **1**, neat. [d]1 mole % $Pd(OAc)_2$, 2 mole % 1, 5 mL dioxane. [e]0.5 mole % $Pd(OAc)_2$, 1 mole % 1, 5 mL dioxane. [f]0.25 mole % $Pd(OAc)_2$, 0.5 mole % 1, 5 mL dioxane. [g]1 mole % $Pd(OAc)_2$, 2 mole % 1, neat. [h]Ref 2a. [i]Refs 2b-2e. [j]Refs 2e-2j. [k]Refs 2a-2d, 2f-2i, 2k, 2l-2p. [l]Ref 2c.

Table 3. Scope of Aryl Chlorides

entry	aryl chloride	siloxane	time	product	yield (%)[a,b]
1			1.5 h		87[c] (Lit: 25)[f]
2			3 h		82[d]
3			2 h		93[e]
4			2 h		95[e] (Lit: 68)[g]
5			1.5 h		95[e] (Lit: 62-99)[h]
6			2 h		90[c]
7			2 h		87[d]
8			1 h		83[d]
9			0.5 h		91[c]

[a]Average of two runs. Yields correspond to isolated yields. [b]Reaction conditions: 2 mmol aryl chloride, 4 mmol siloxane, 3 mmol TBAF•xH_2O, neat, time: 0.5-2 h. [c]0.5 mole % $Pd(OAc)_2$, 1 mole % **1**, neat. [d]1 mole % $Pd(OAc)_2$, 2 mole % **1**, neat. [e]0.5 mole % $Pd(OAc)_2$, 1.0 mole % **1**, 5 mL dioxane. [f]Ref 2g. [g]Ref 3a. [h]Refs 2g-2i, 2m-2n, 3b.

Table 4. Scope of Heterocyclic Aryl Chlorides

entry	aryl chloride	siloxane	time	product	yield (%)[a,b]
1			2 h		81[c]
2			1.5 h		82[d] (Lit: 63-92)[e]
3			1 h		95[c]

[a]Average of two runs. Yields correspond to isolated yields. [b]Reaction conditions: 2 mmol aryl chloride, 4 mmol siloxane, 0.5 mole % $Pd(OAc)_2$, 1 mole % **1**, 3 mmol TBAF•xH_2O, time: 0.5-2 h. [c]Neat condition. [d]5 mL dioxane. [e]Refs 3a-3b.

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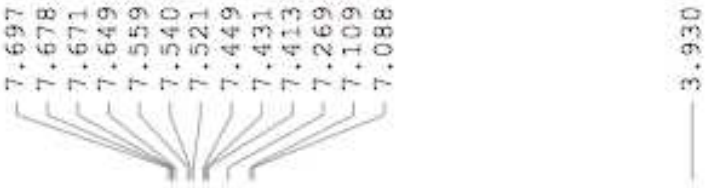

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White solid
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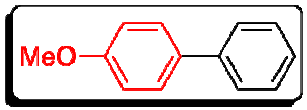
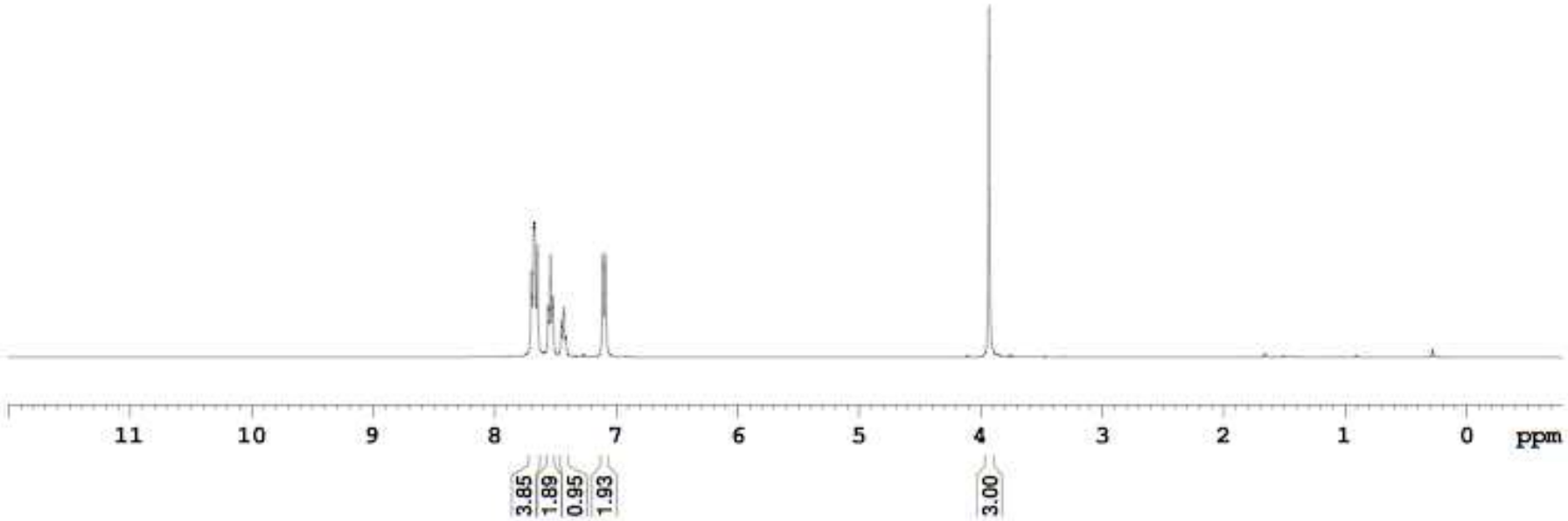
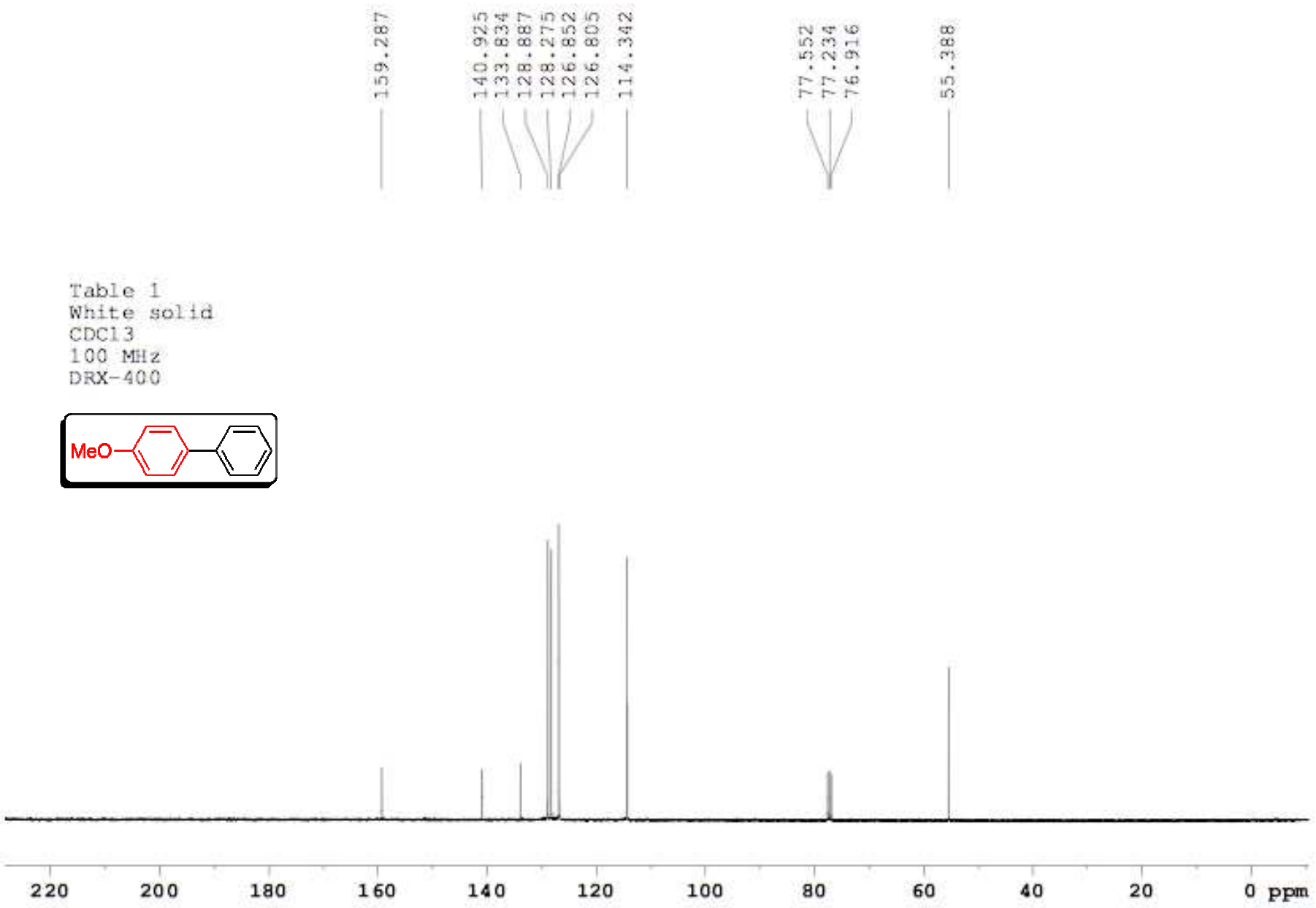
DRX-400



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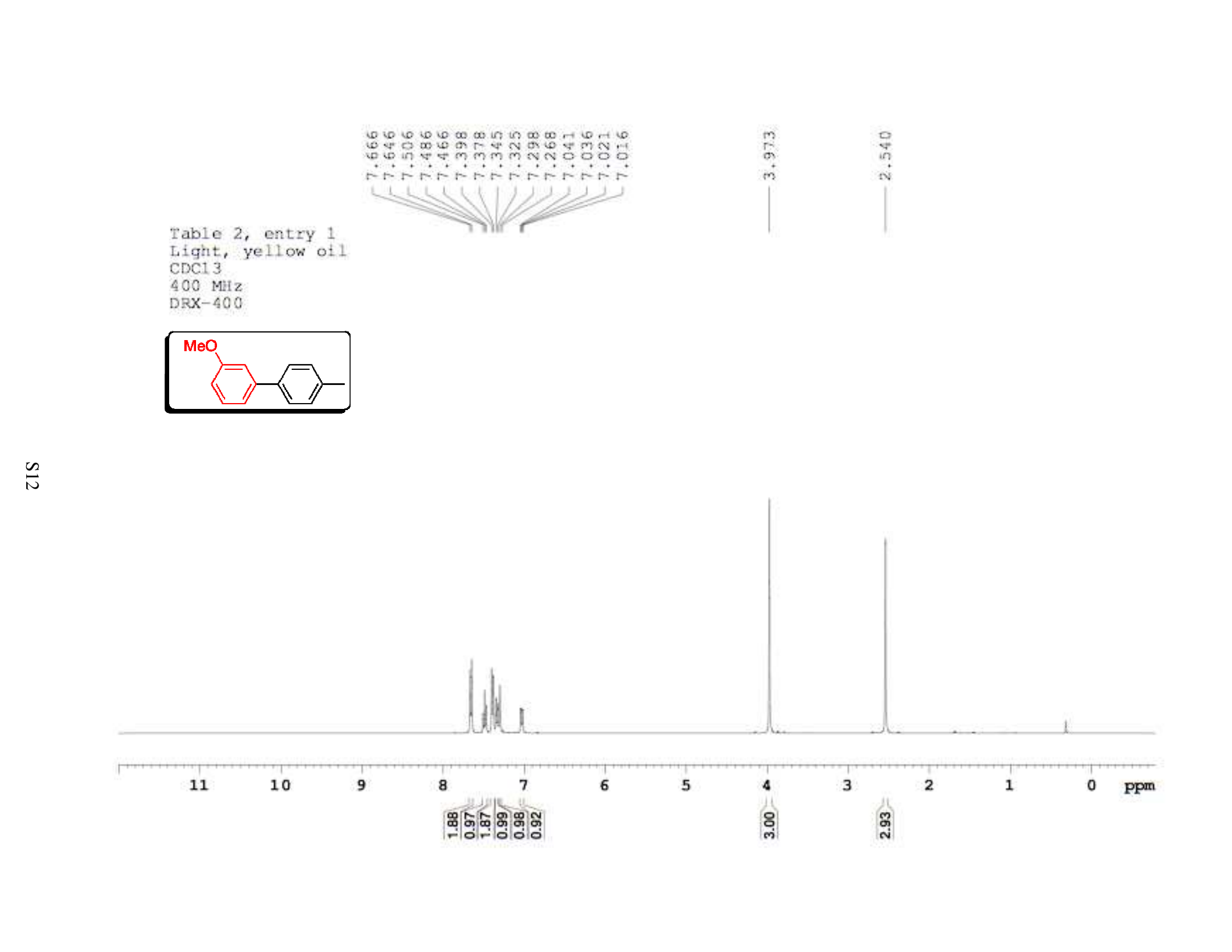
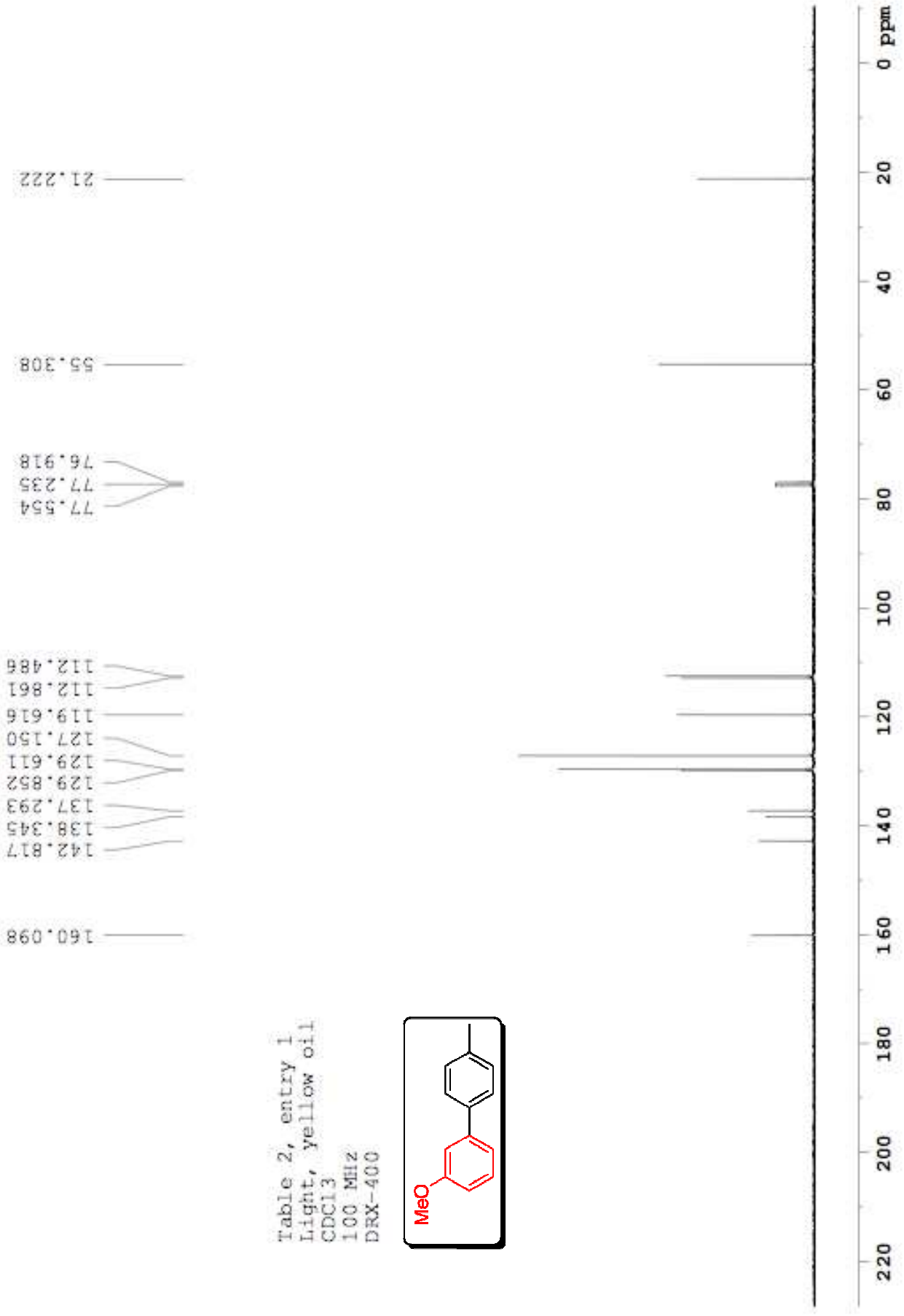
140.925
133.834
128.887
128.275
126.852
126.805
114.342
77.552
77.234
76.916
55.388
Table 1
White solid
CDCl3
100 MHz
DRX-400
MeO
220
200
180
160
140
120
100
80
60
40
20
0 ppm


7.666
7.646
7.506
7.486
7.466
7.398
7.378
7.345
7.325
7.298
7.268
7.041
7.036
7.021
7.016
3.973
2.540
Table 2, entry 1
Light, yellow oil
CDCl3
400 MHz
DRX-400
MeO
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.88
0.97
1.87
0.99
0.98
0.92
3.00
2.93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55.308
76.918
77.235
77.554
112.486
112.861
119.616
127.150
129.611
129.852
137.293
138.345
142.817
160.098
Table 2, entry 1
Light, yellow oil
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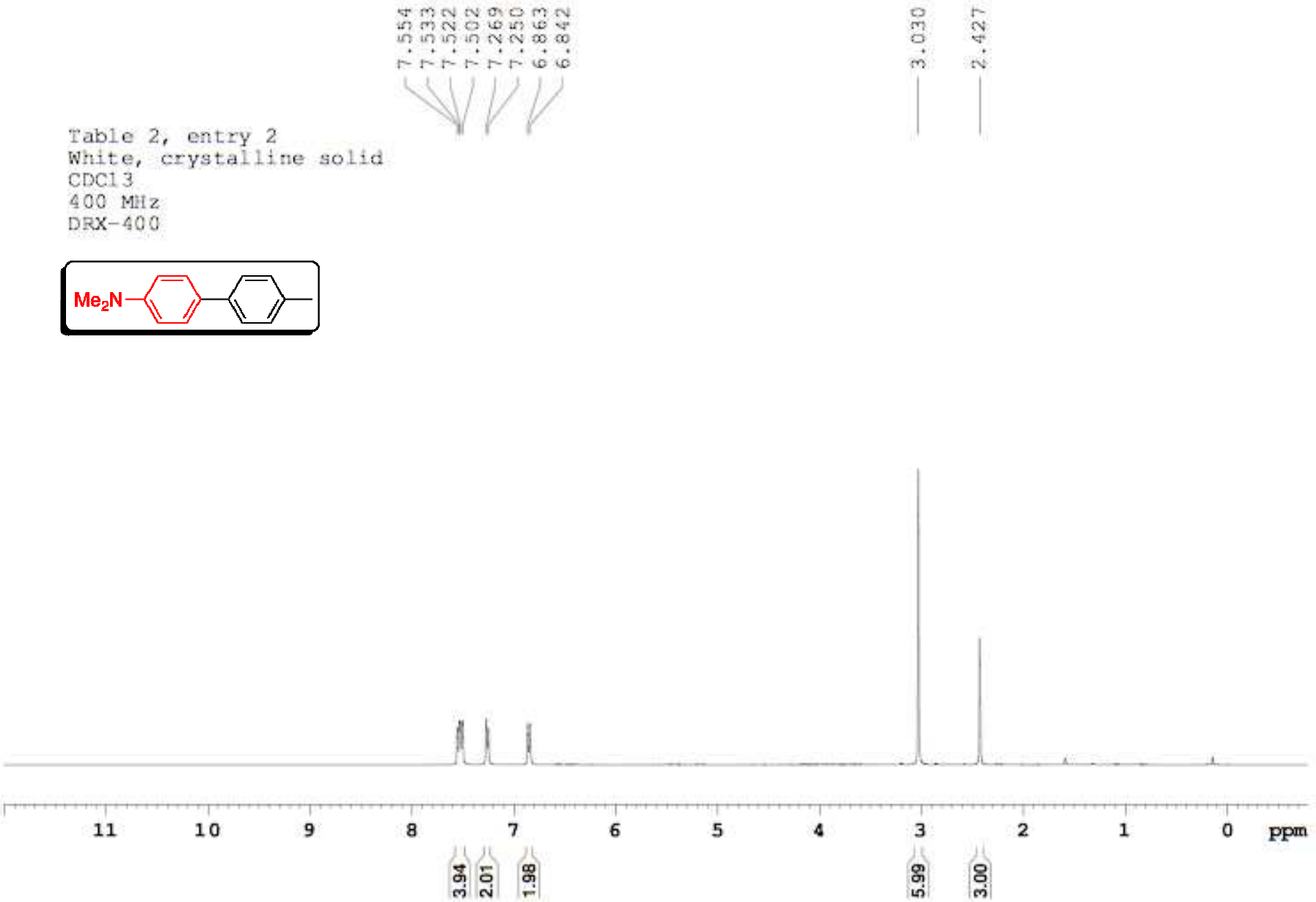
100 MHz
DRX-400
MeO
0 ppm
20
40
60
80
100
120
140
160
180
200
220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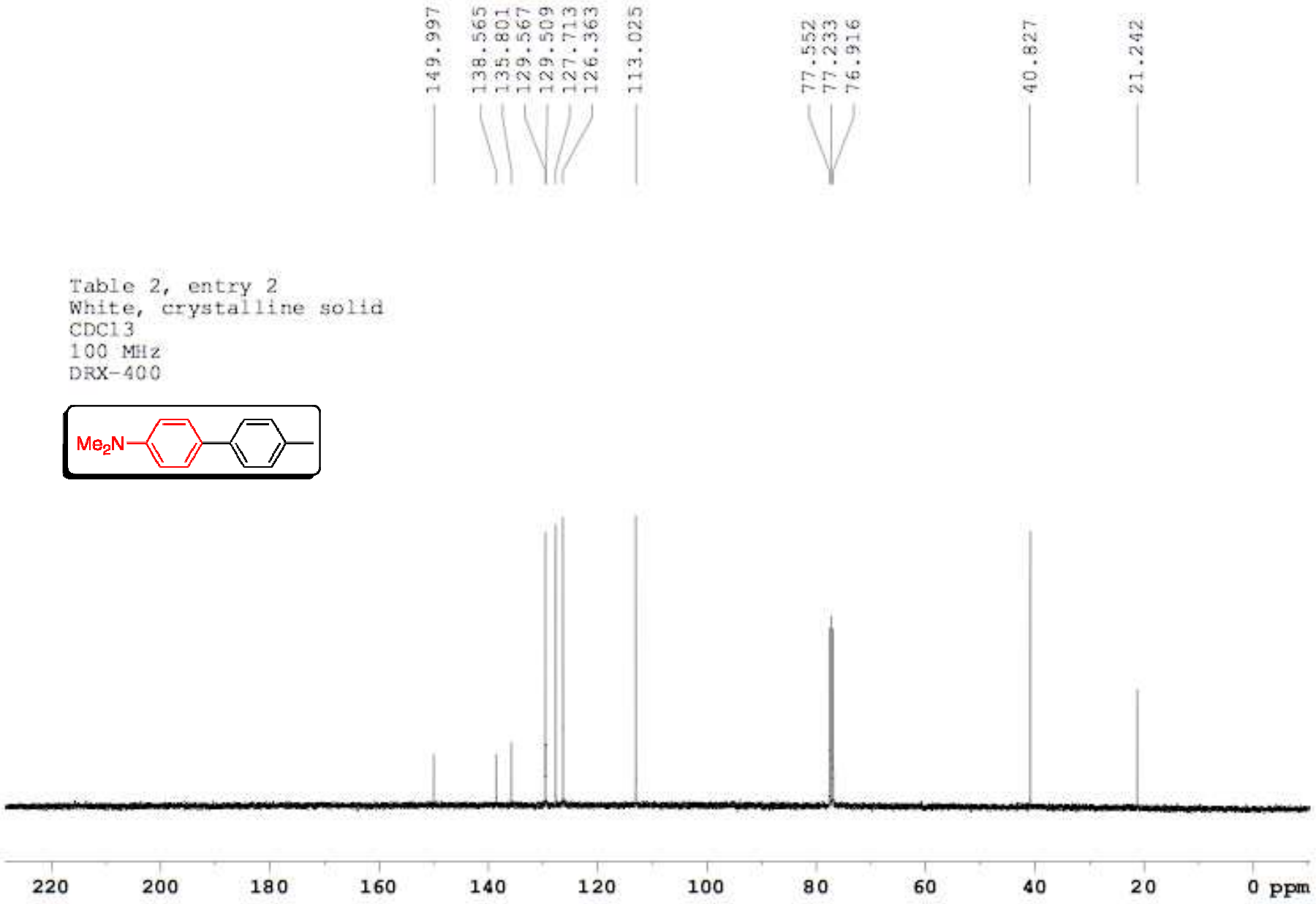
7.533
7.522
7.502
7.269
7.250
6.863
6.842
3.030
2.427
Table 2, entry 2
White, crystalline solid
CDCl3
400 MHz
DRX-400
Me2N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.94
2.01
1.98
5.99
3.00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138.565
135.801
129.567
129.509
127.713
126.363
113.025
77.552
77.233
76.916
40.827
21.242
Table 2, entry 2
White, crystalline solid
CDCl3
100 MHz
DRX-400
Me2N
220
200
180
160
140
120
100
80
60
40
20
0 ppm

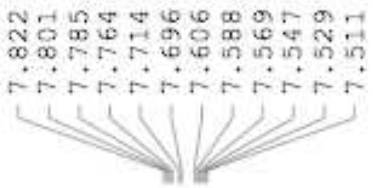

Table 2, entry 3
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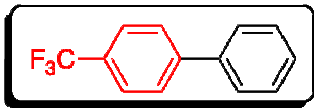
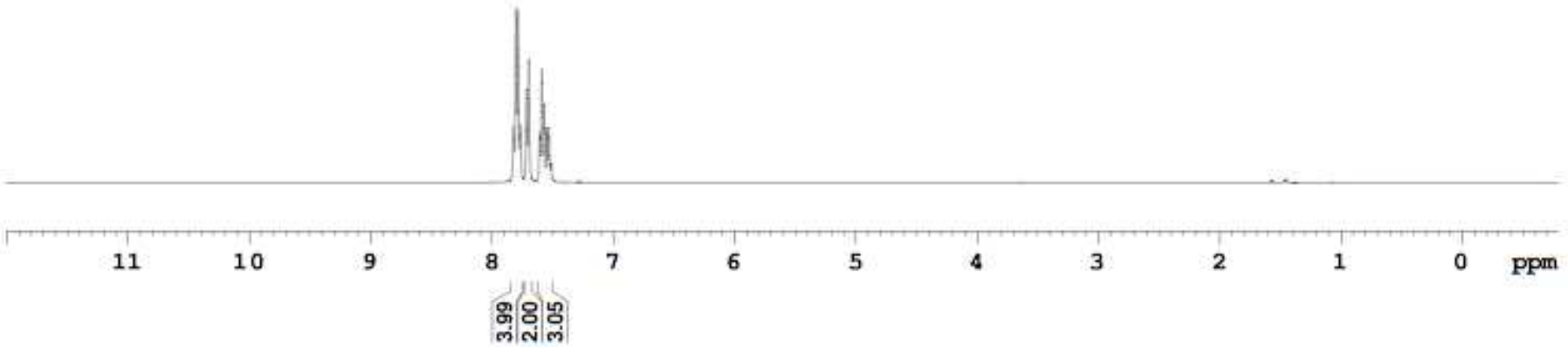
400 MHz
DRX-400

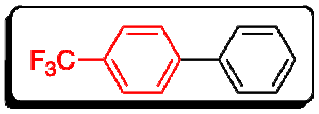


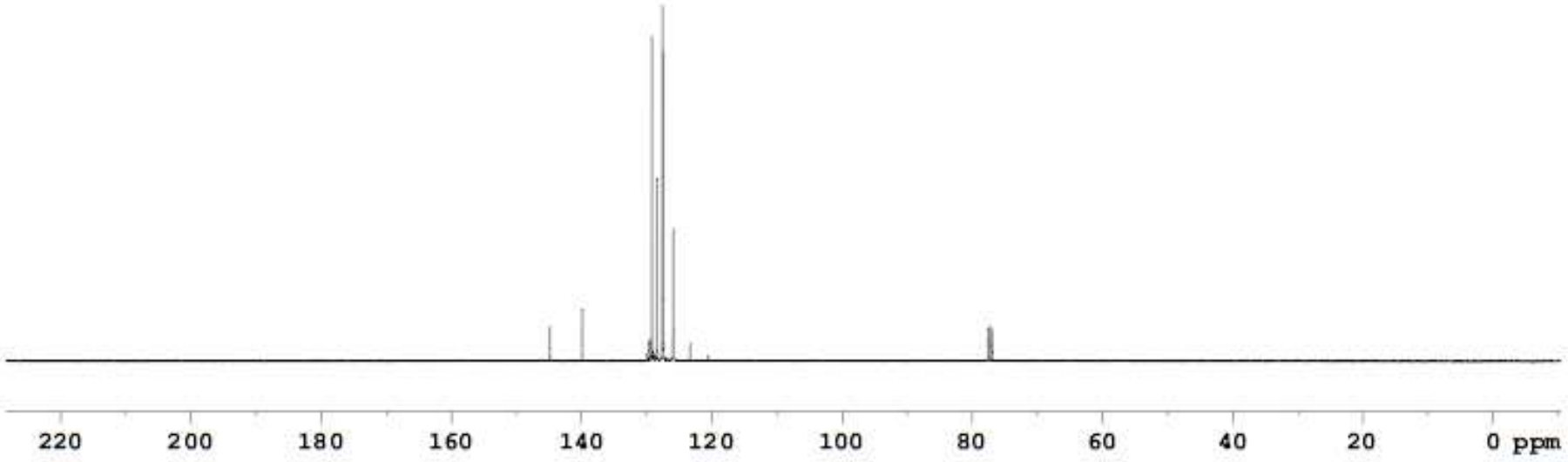


144.906
139.914
129.711
129.389
129.201
128.417
127.586
127.451
125.995
125.965
125.927
125.889
125.853
123.293

77.552
77.234
76.916

Table 2, entry 3
White solid
CDCl3
100 MHz
DRX-400





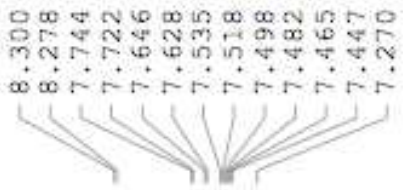
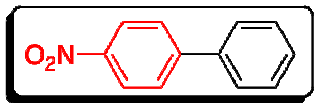
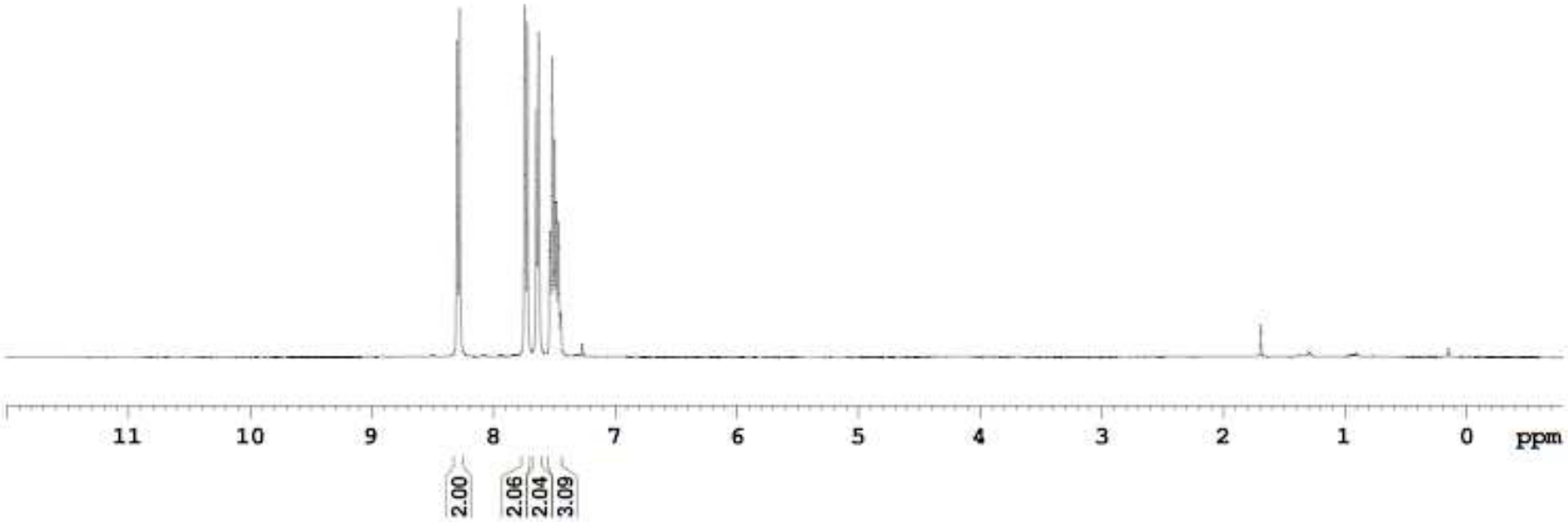


Table 2, entry 4
Light yellow solid
CDCl3
400 MHz
DRX-400

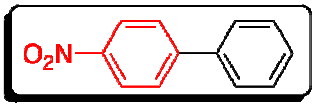




147.682
147.147
138.805
129.278
129.057
127.868
127.479
124.189

77.555
77.236
76.919

Table 2, entry 4
Light, yellow solid
CDCl3
100 MHz
DRX-400



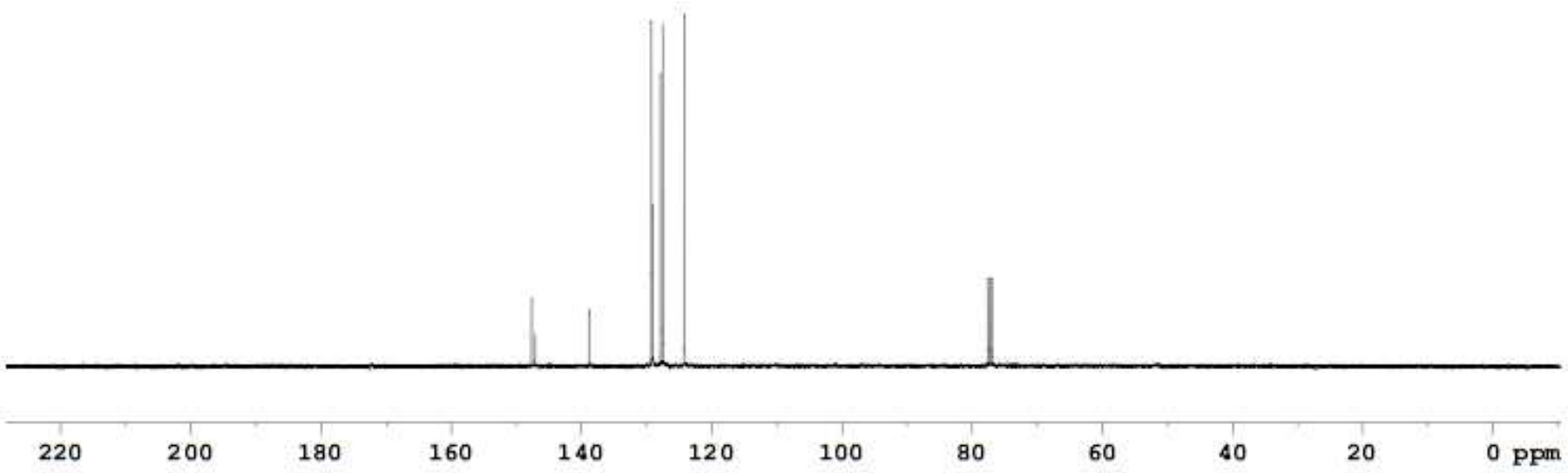
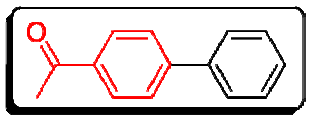


Table 2, entry 5 &
Table 3, entry 5
White solid
CDCl3
400 MHz
DRX-400



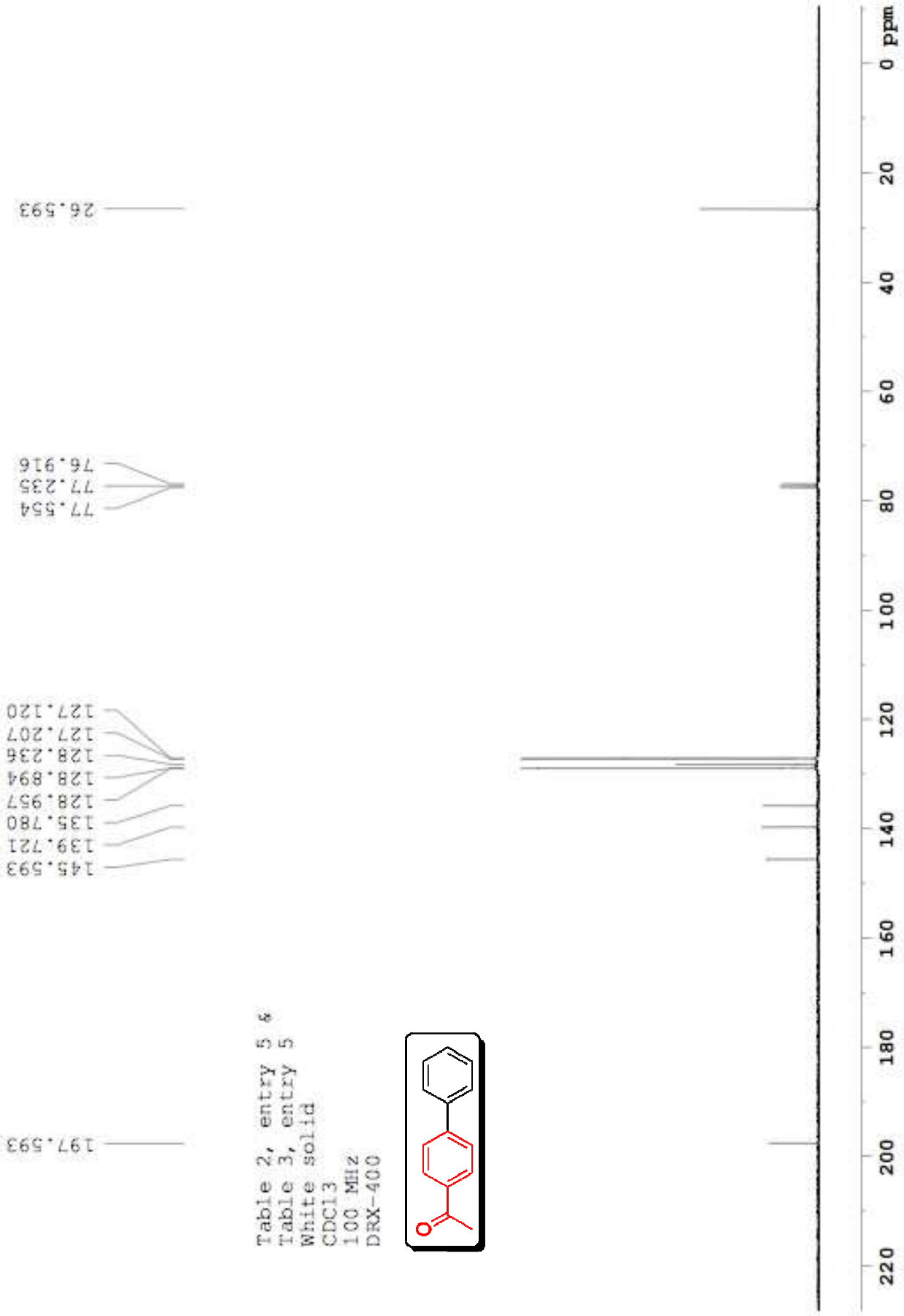


Table 2, entry 5 &
Table 3, entry 5
White solid
CDCl3
100 MHz
DRX-400
O
197.593
145.593
139.721
135.780
128.957
128.894
128.236
127.207
127.120
77.554
77.235
76.916
26.593
0 ppm
20
40
60
80
100
120
140
160
180
200
220

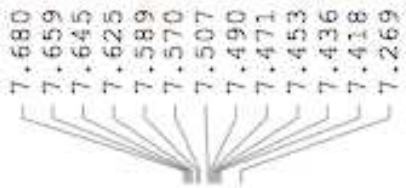
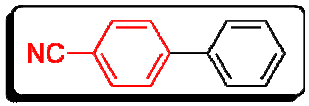
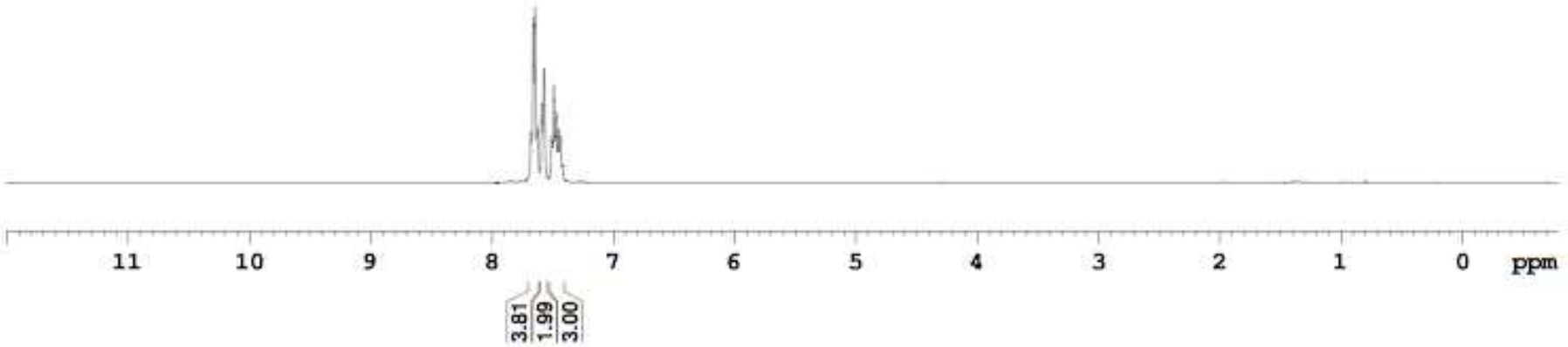


Table 2, entry 6
Table 3, entry 3
White solid
CDCl3
400 MHz
DRX-400





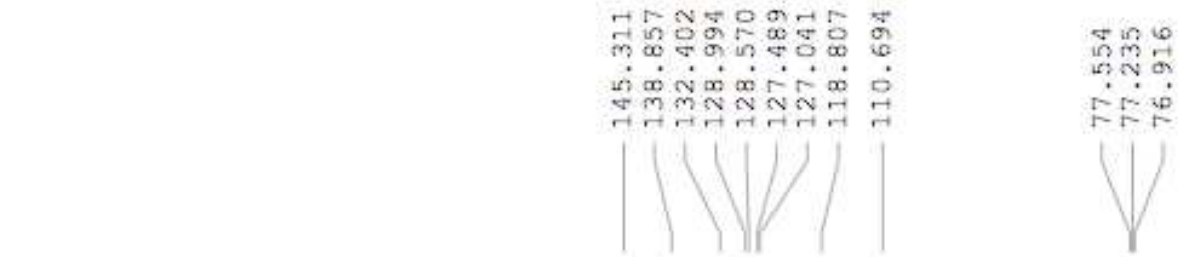
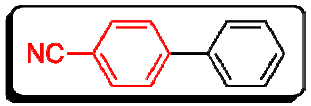
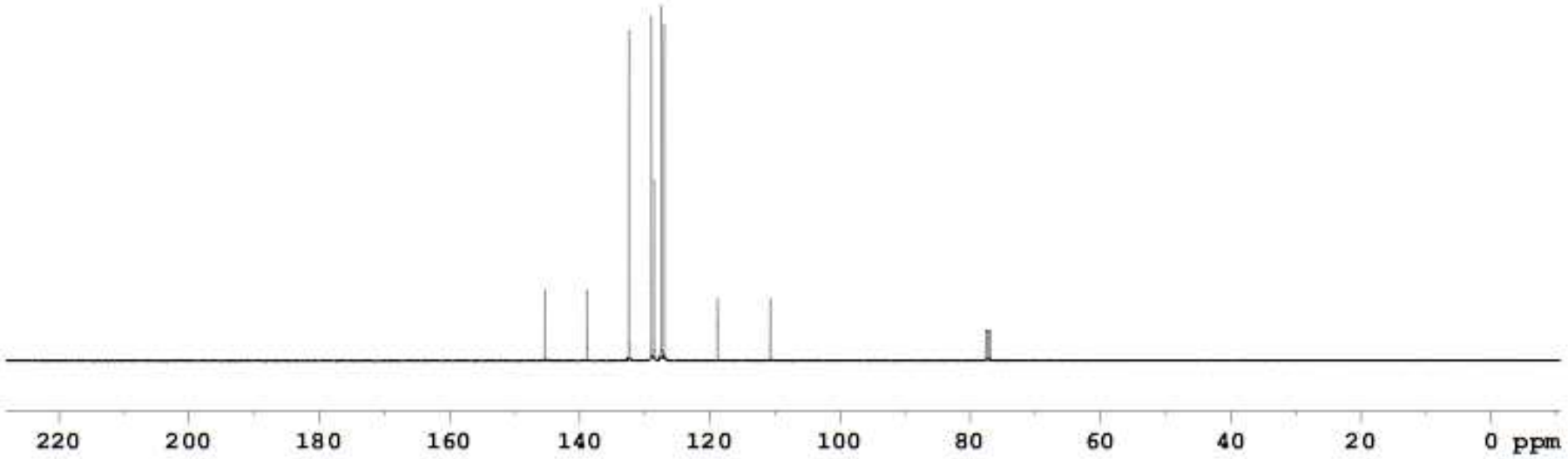


Table 2, entry 6 &
Table 3, entry 3
White solid
CDCl3
100 MHz
DRX-400





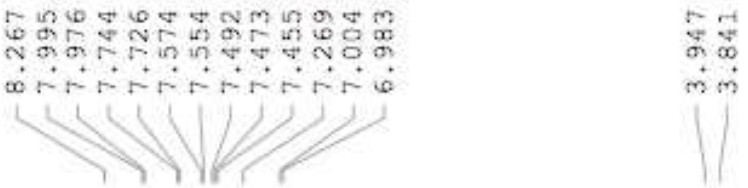
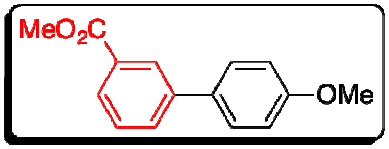
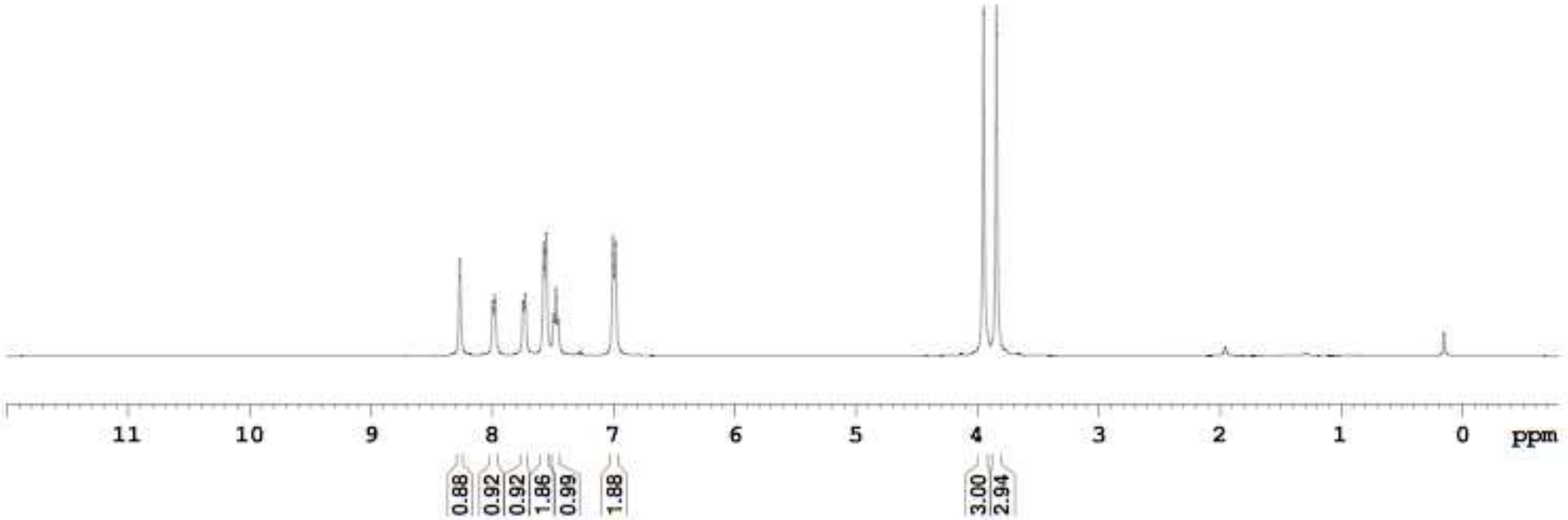
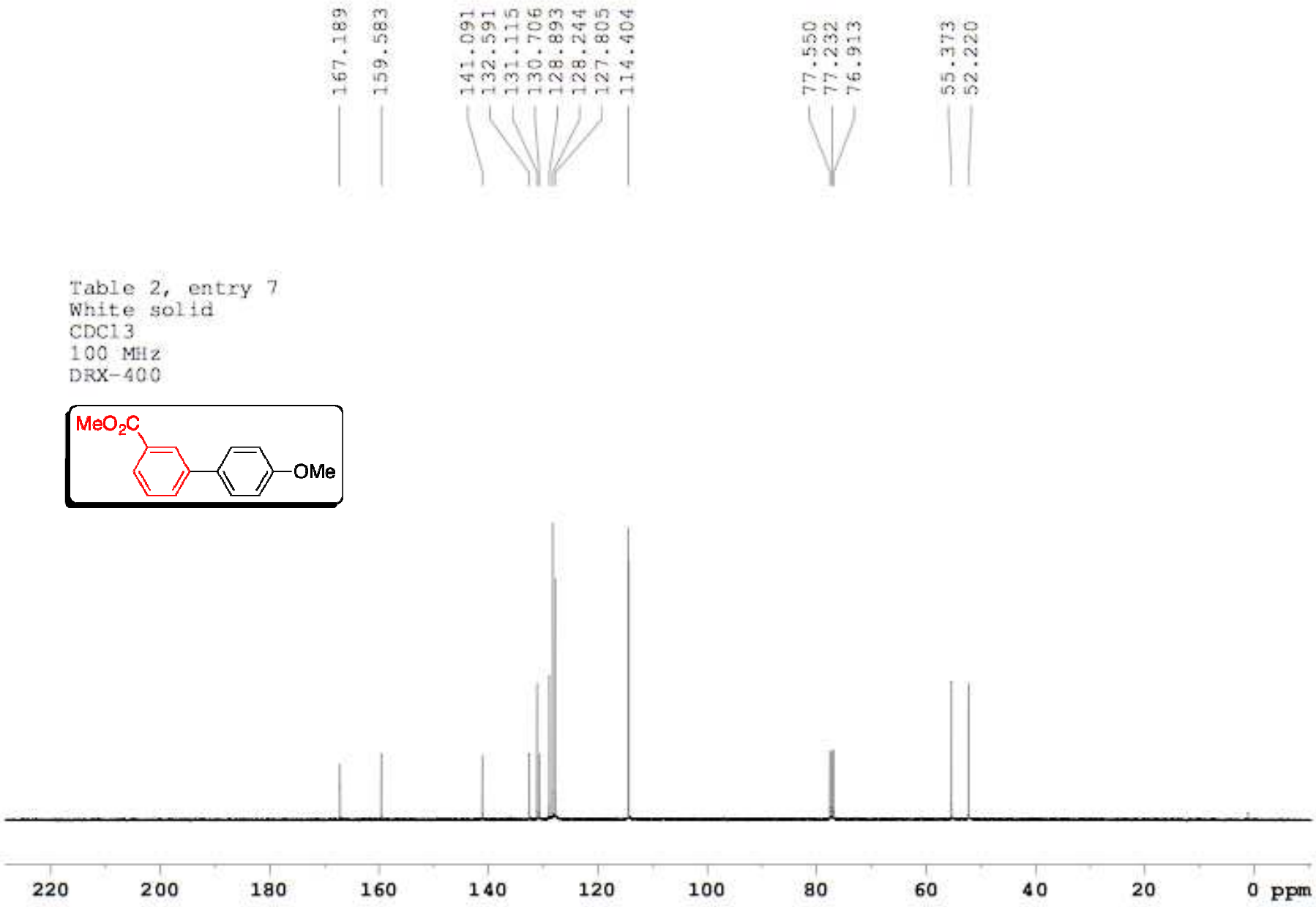
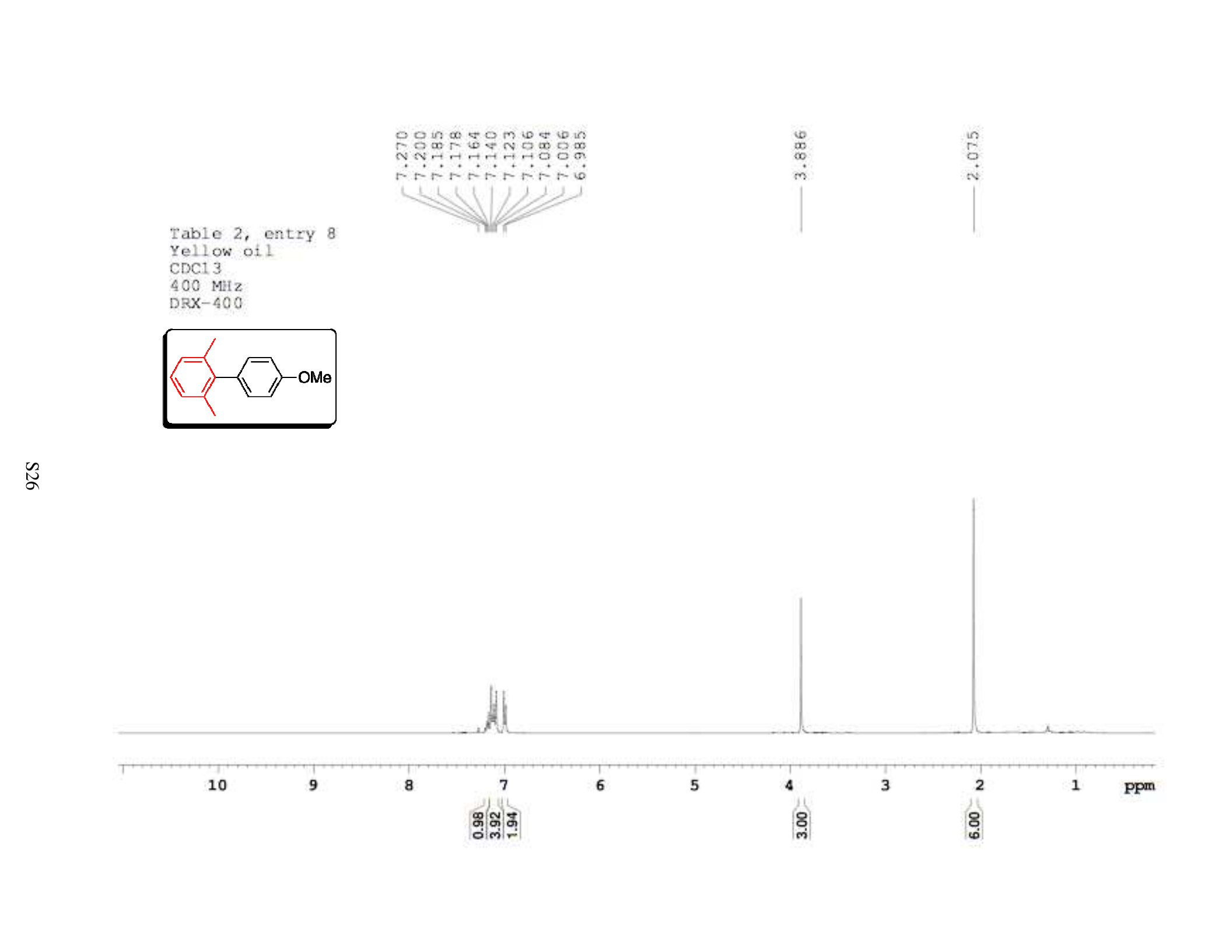


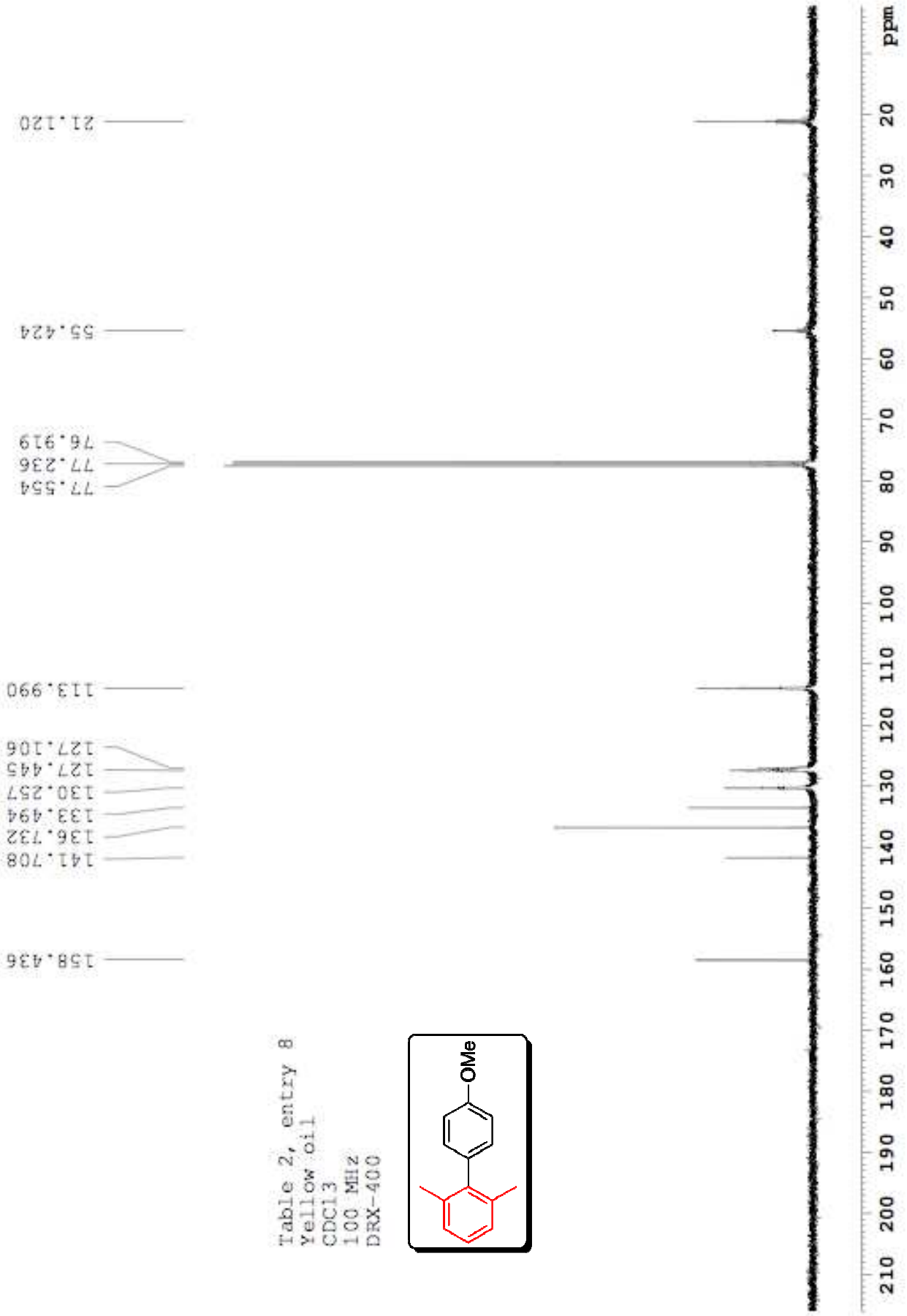
Table 2, entry 7
White solid
CDCl3
400 MHz
DRX-400






167.189
159.583
141.091
132.591
131.115
130.706
128.893
128.244
127.805
114.404
77.550
77.232
76.913
55.373
52.220
Table 2, entry 7
White solid
CDCl3
100 MHz
DRX-400
MeO2C
OMe
220
200
180
160
140
120
100
80
60
40
20
0 ppm


7.270
7.200
7.185
7.178
7.164
7.140
7.123
7.106
7.084
7.006
6.985
3.886
2.075
Table 2, entry 8
Yellow oil
CDCl3
400 MHz
DRX-400
OMe
10
9
8
7
6
5
4
3
2
1
ppm
0.98
3.92
1.94
3.00
6.00


21.120
55.424
76.919
77.236
77.554
113.990
127.106
127.445
130.257
133.494
136.732
141.708
158.436
Table 2, entry 8
Yellow oil
CDCl3
100 MHz
DRX-400
OMe
ppm
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

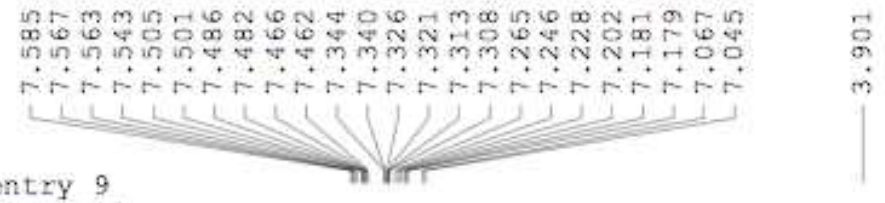
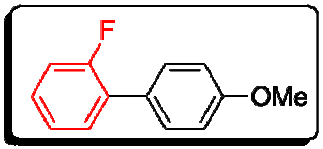
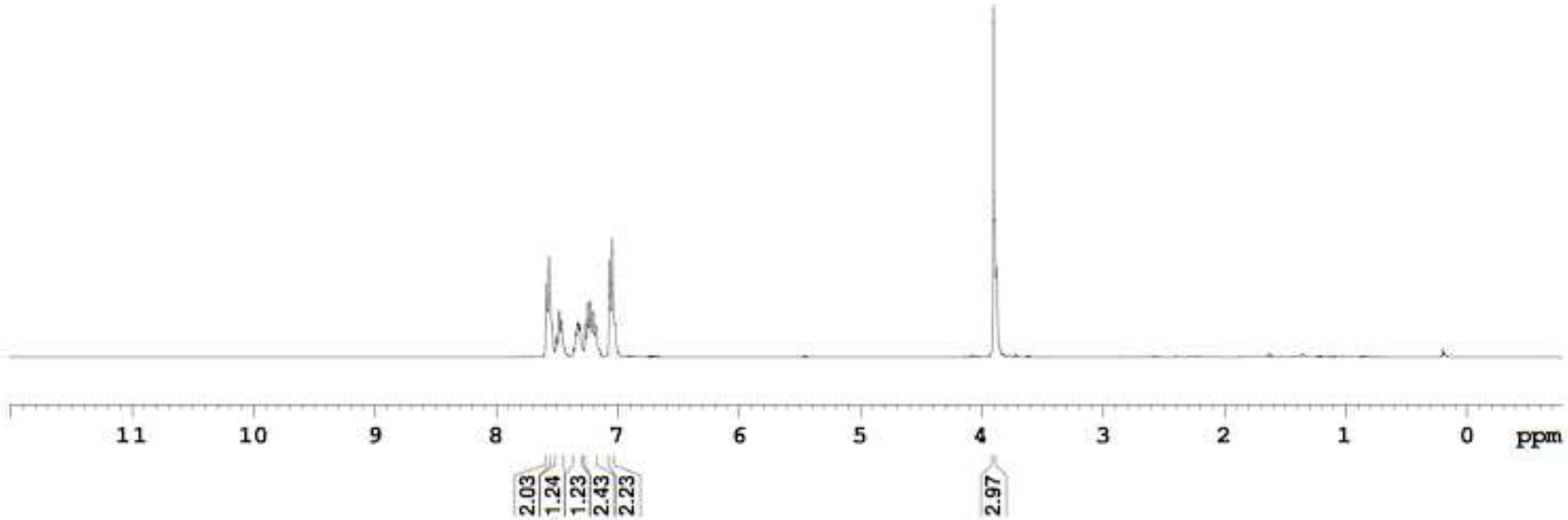
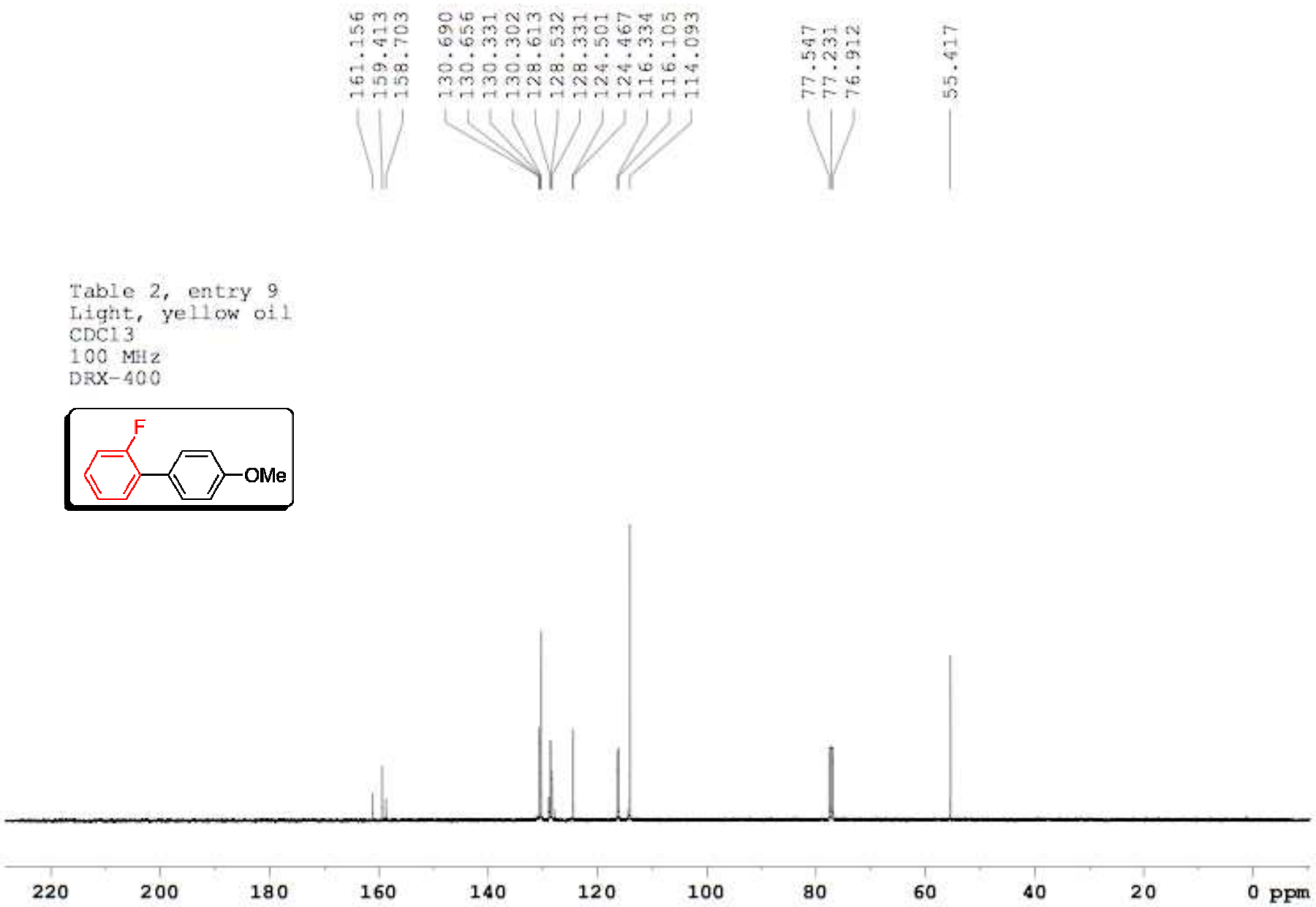
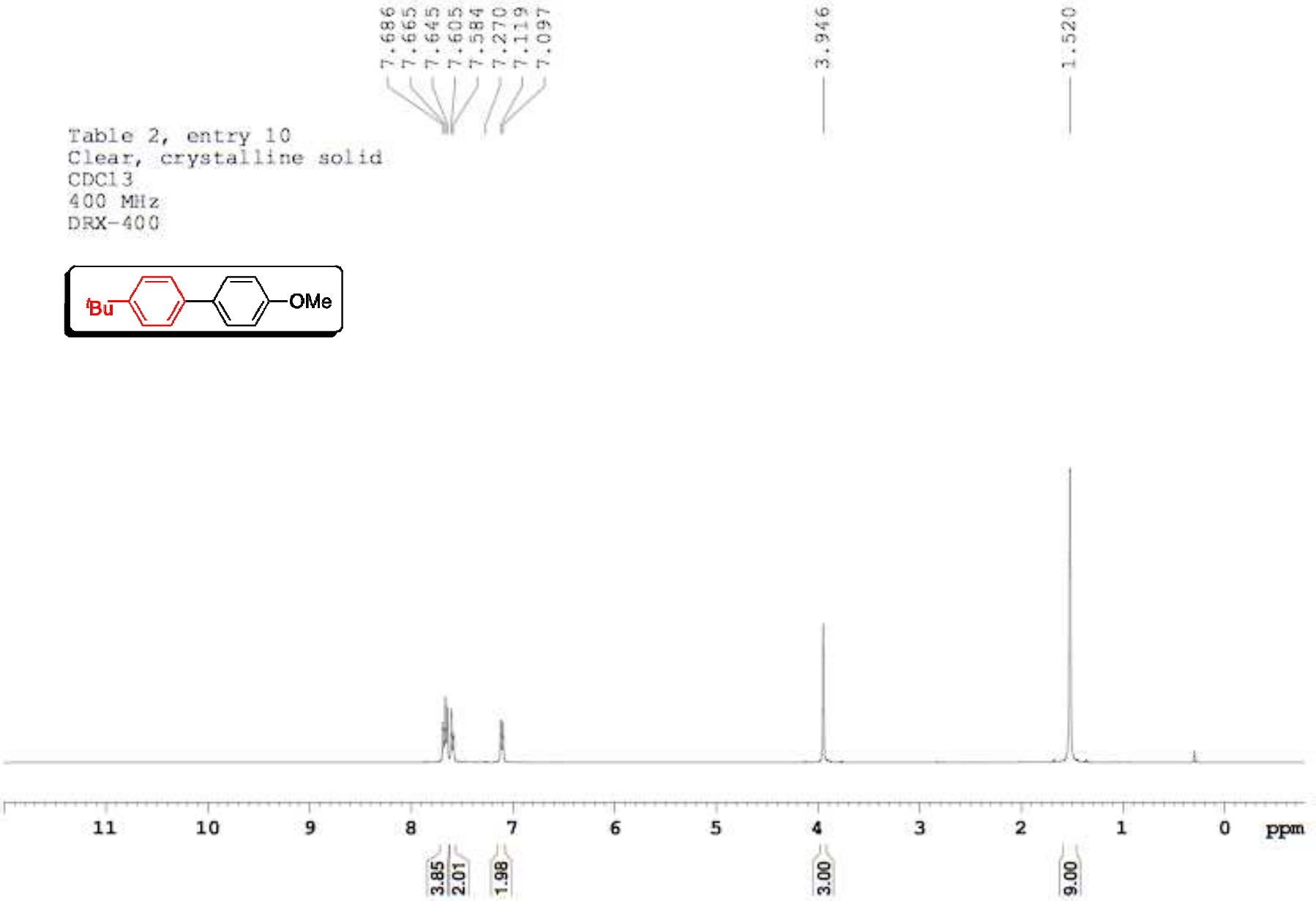


Table 2, entry 9
Light, yellow oil
CDCl3
400 MHz
DRX-400






161.156
159.413
158.703
130.690
130.656
130.331
130.302
128.613
128.532
128.331
124.501
124.467
116.334
116.105
114.093
77.547
77.231
76.912
55.417
Table 2, entry 9
Light, yellow oil
CDCl3
100 MHz
DRX-400
F
OMe
220
200
180
160
140
120
100
80
60
40
20
0 ppm


7.686
7.665
7.645
7.605
7.584
7.270
7.119
7.097
3.946
1.520
Table 2, entry 10
Clear, crystalline solid
CDCl3
400 MHz
DRX-400
tBu
OMe
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.85
2.01
1.98
3.00
9.00

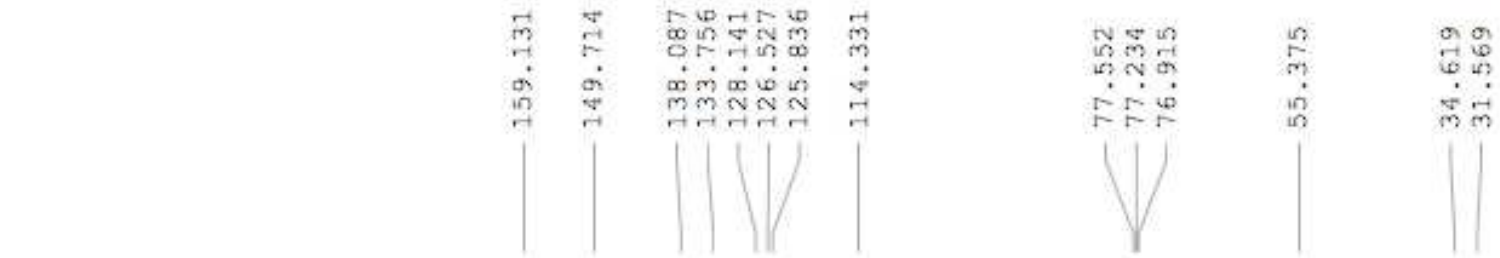
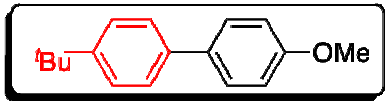
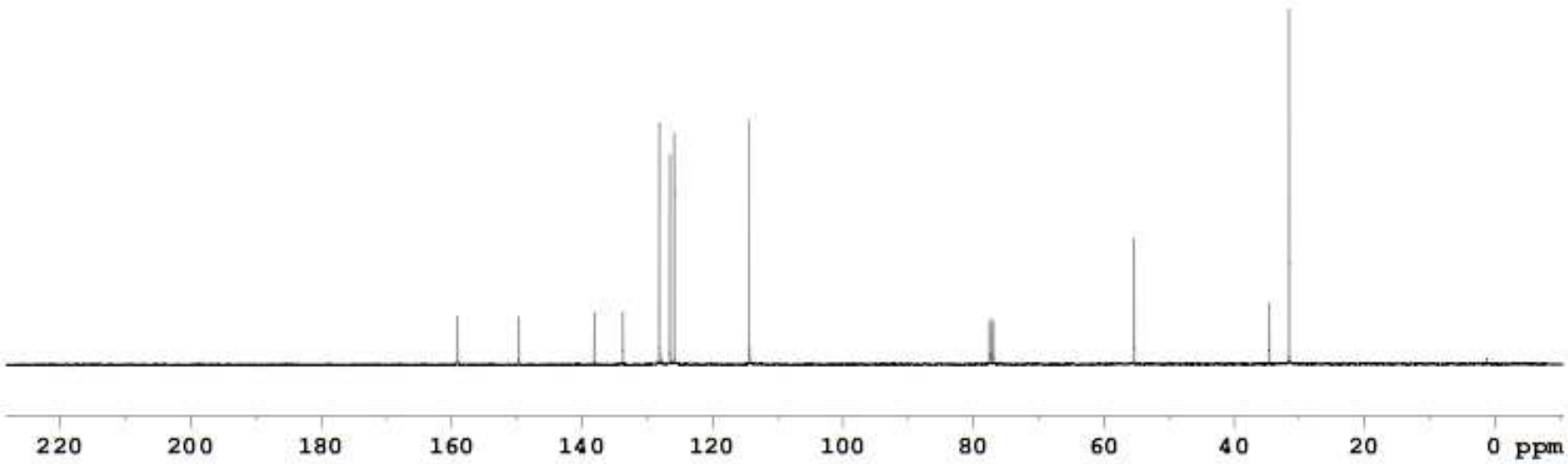
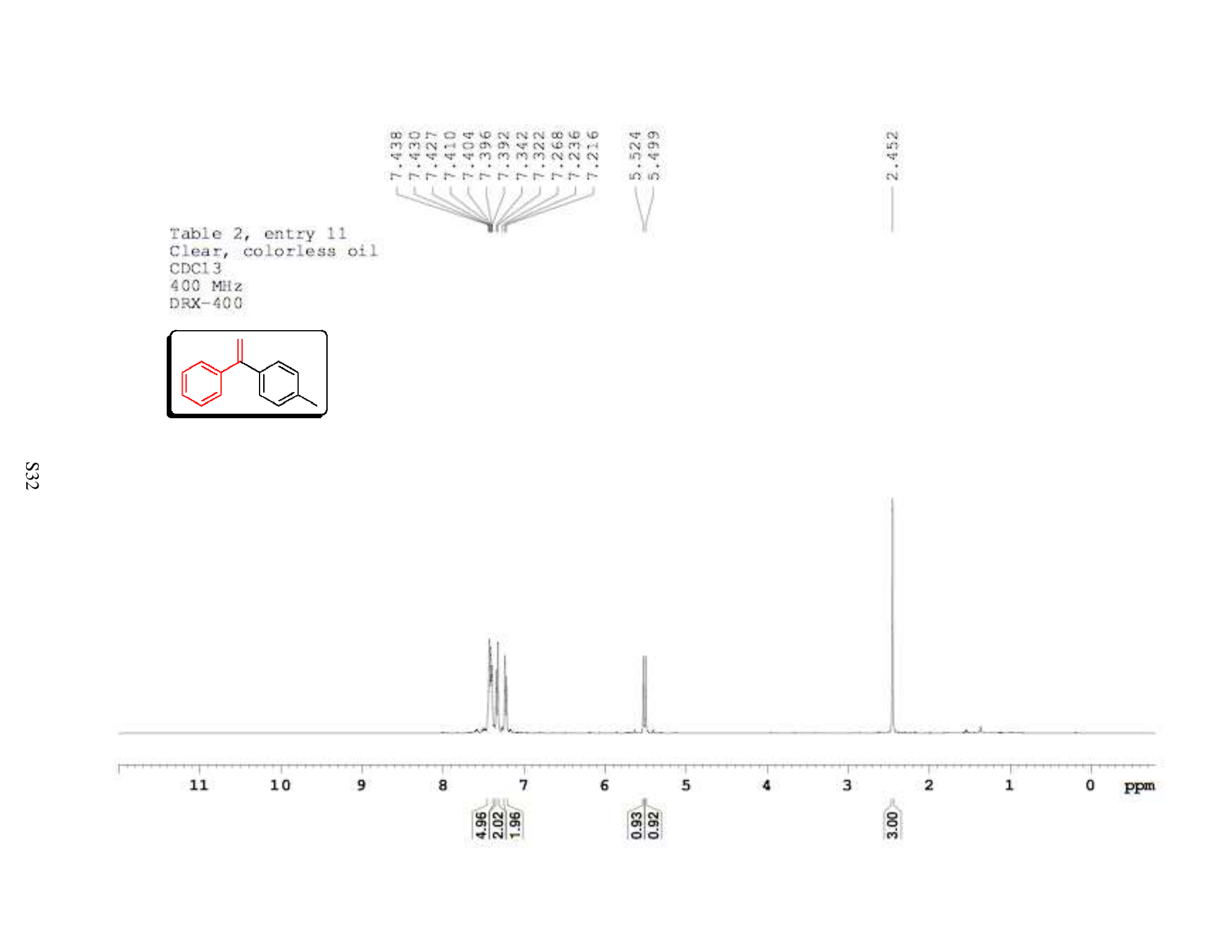
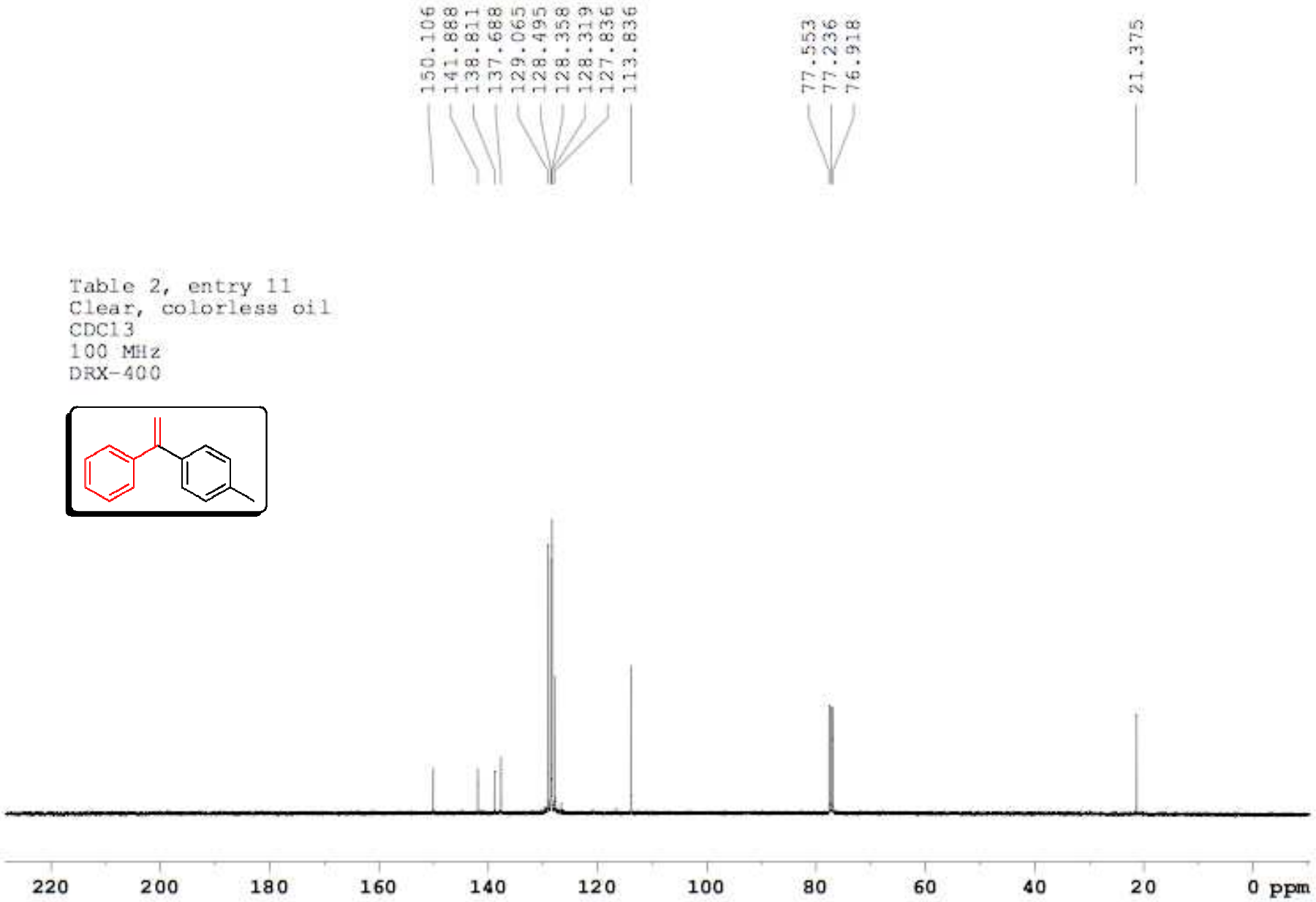


Table 2, entry 10
Clear, crystalline solid
CDCl3
100 MHz
DRX-400






7.438
7.430
7.427
7.410
7.404
7.396
7.392
7.342
7.322
7.268
7.236
7.216
5.524
5.499
2.452
Table 2, entry 11
Clear, colorless oil
CDCl3
400 MHz
DRX-400
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.96
2.02
1.96
0.93
0.92
3.00


150.106
141.888
138.811
137.688
129.065
128.495
128.358
128.319
127.836
113.836
77.553
77.236
76.918
21.375
Table 2, entry 11
Clear, colorless oil
CDCl3
100 MHz
DRX-400
220
200
180
160
140
120
100
80
60
40
20
0 ppm

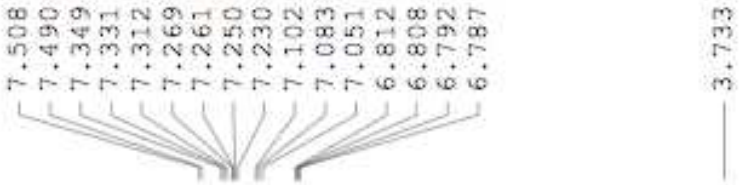
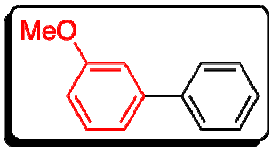
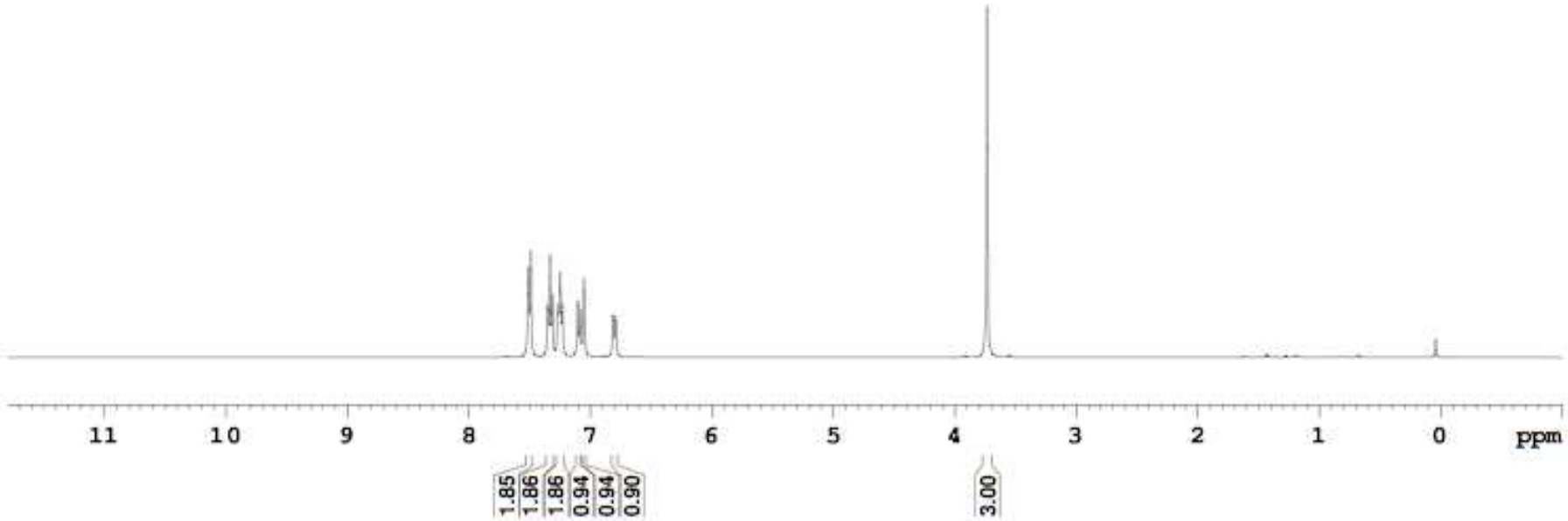
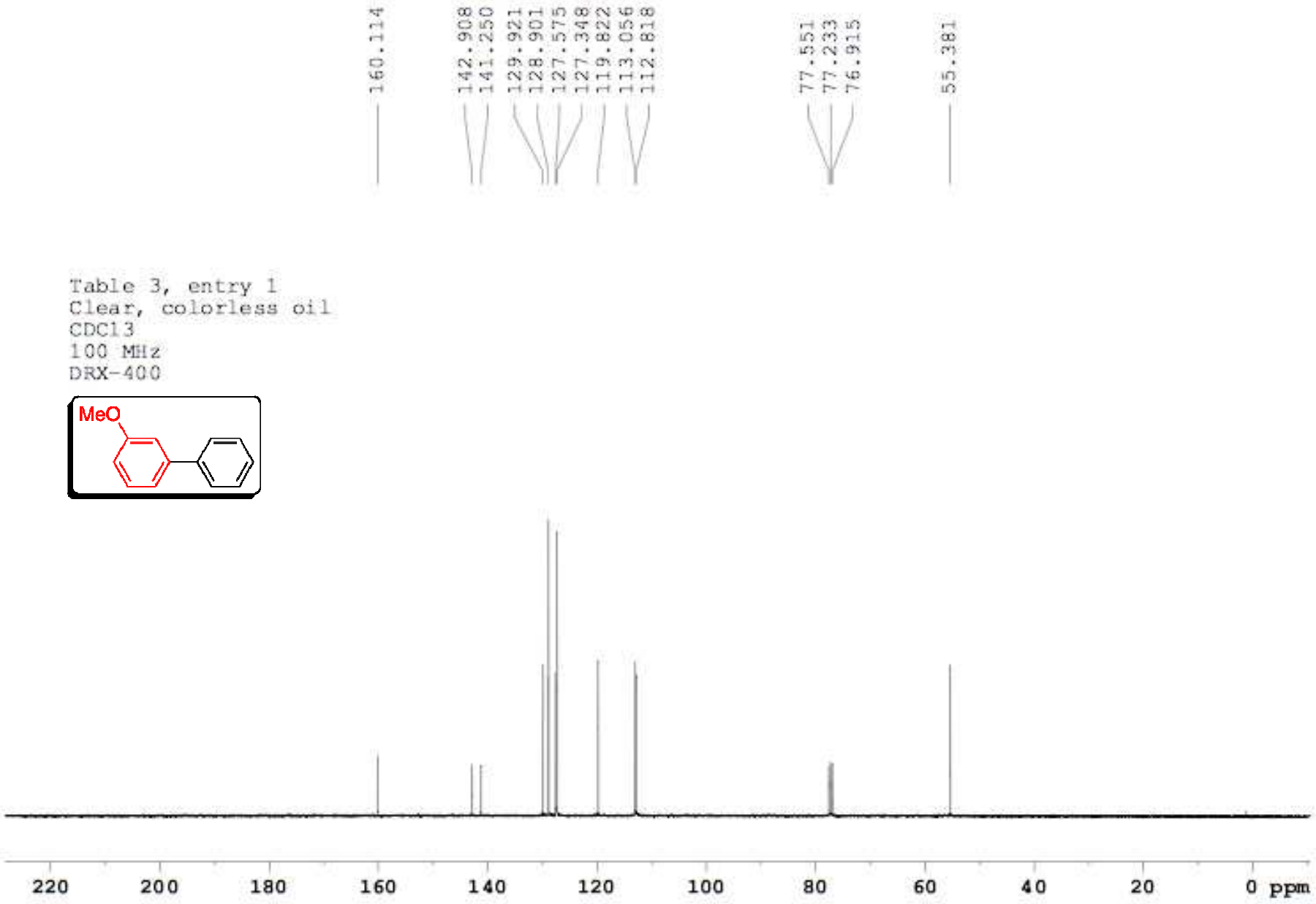


Table 3, entry 1
Clear, colorless oil
CDCl3
400 MHz
DRX-400






160.114
142.908
141.250
129.921
128.901
127.575
127.348
119.822
113.056
112.818
77.551
77.233
76.915
55.381
Table 3, entry 1
Clear, colorless oil
CDCl3
100 MHz
DRX-400
MeO
220
200
180
160
140
120
100
80
60
40
20
0 ppm

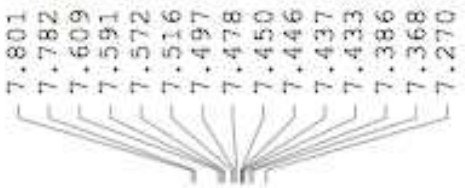
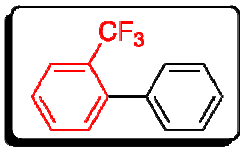
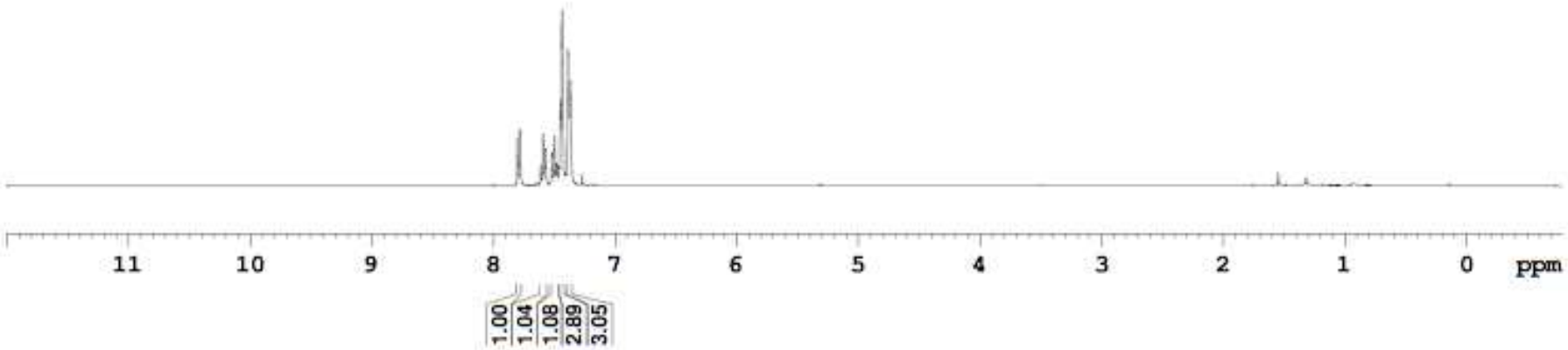


Table 3, entry 2
Clear, colorless oil
CDCl3
400 MHz
DRX-400





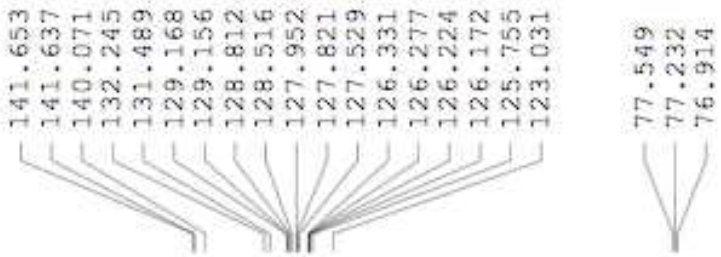
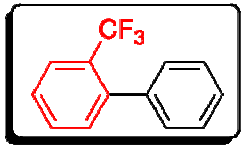
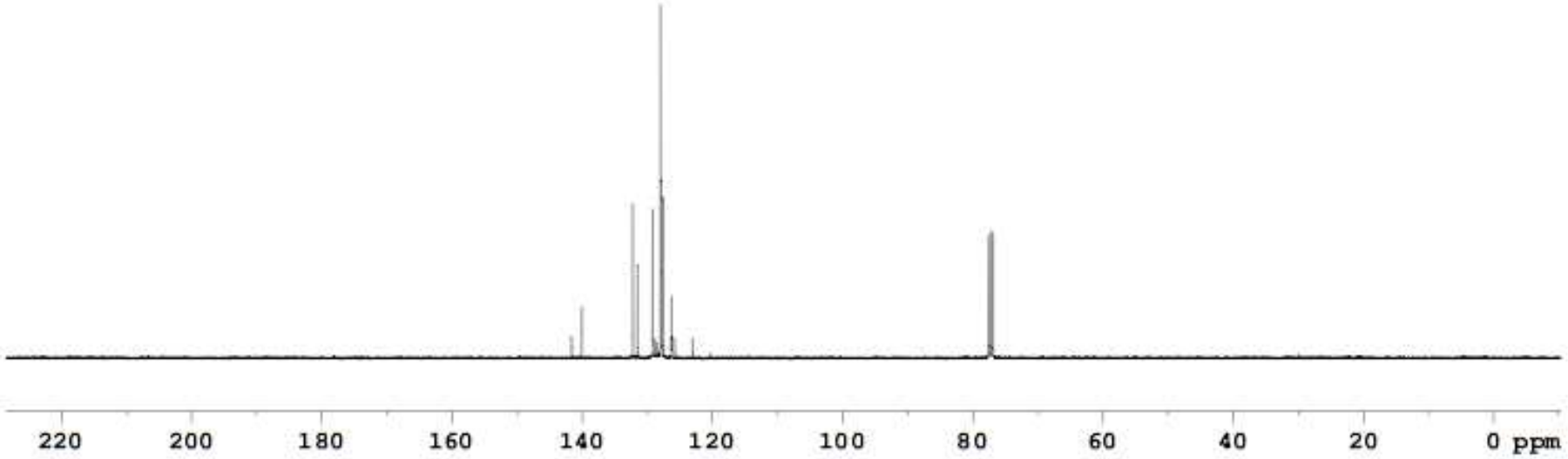
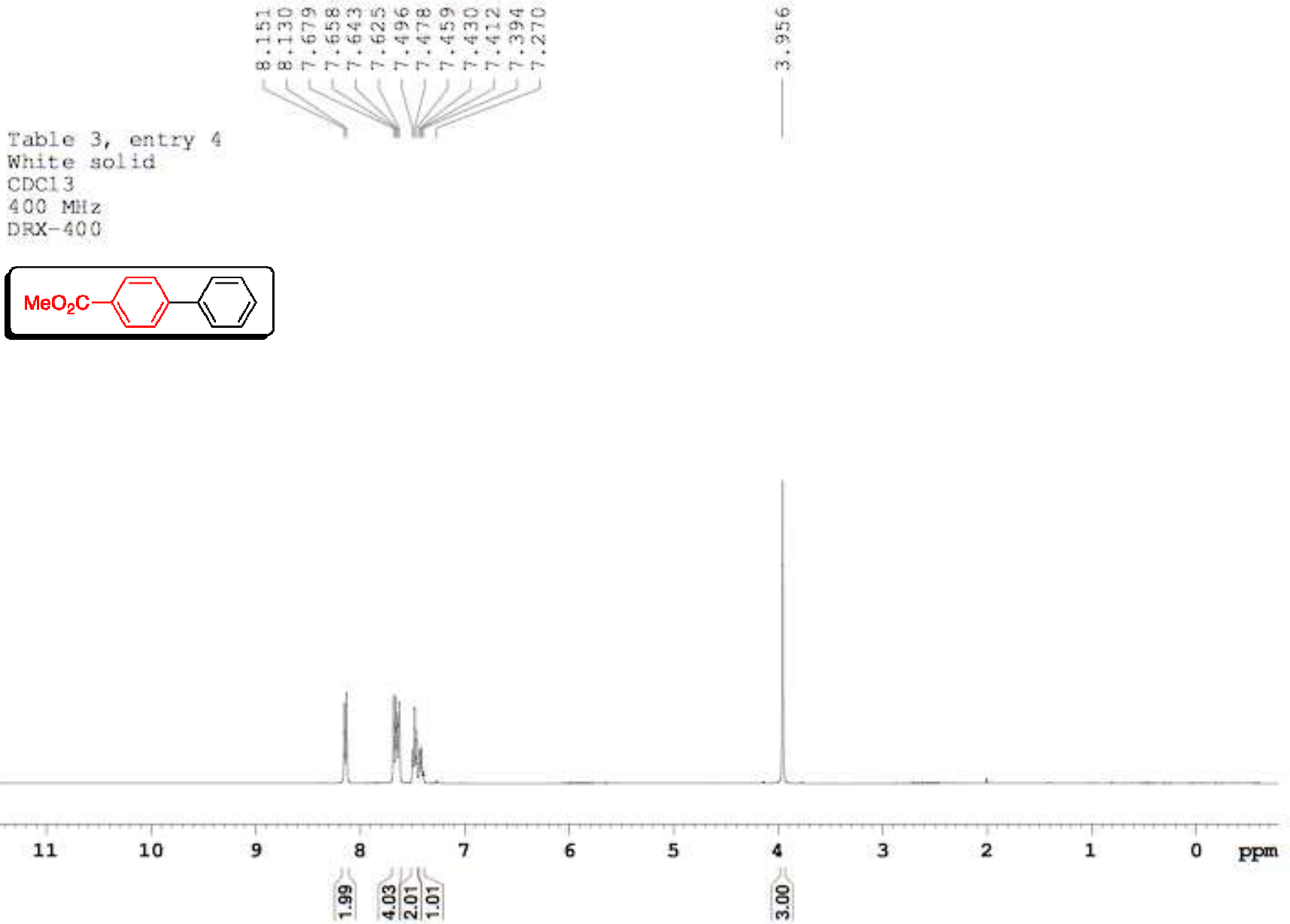


Table 3, entry 2
Clear, colorless oil
CDCl3
100 MHz
DRX-400






8.151
8.130
7.679
7.658
7.643
7.625
7.496
7.478
7.459
7.430
7.412
7.394
7.270
3.956
Table 3, entry 4
White solid
CDCl3
400 MHz
DRX-400
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.99
4.03
2.01
1.01
3.00

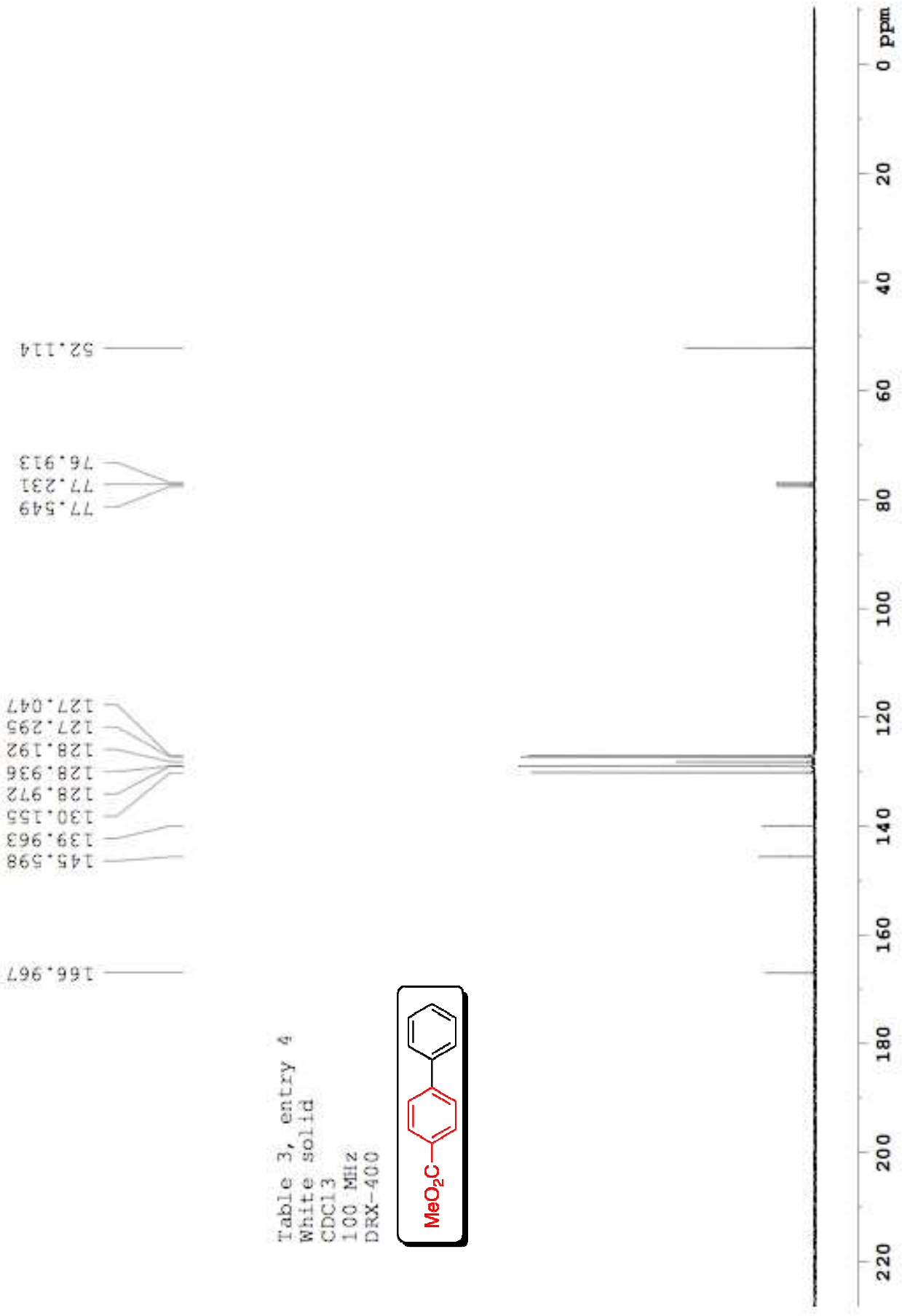


Table 3, entry 4
White solid
CDCl3
100 MHz
DRX-400
MeO2C
166.967
145.598
139.963
130.155
128.972
128.936
128.192
127.295
127.047
77.549
77.231
76.913
52.114
0 ppm
20
40
60
80
100
120
140
160
180
200
220

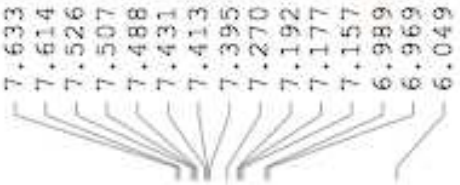
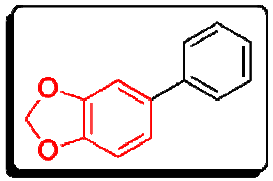
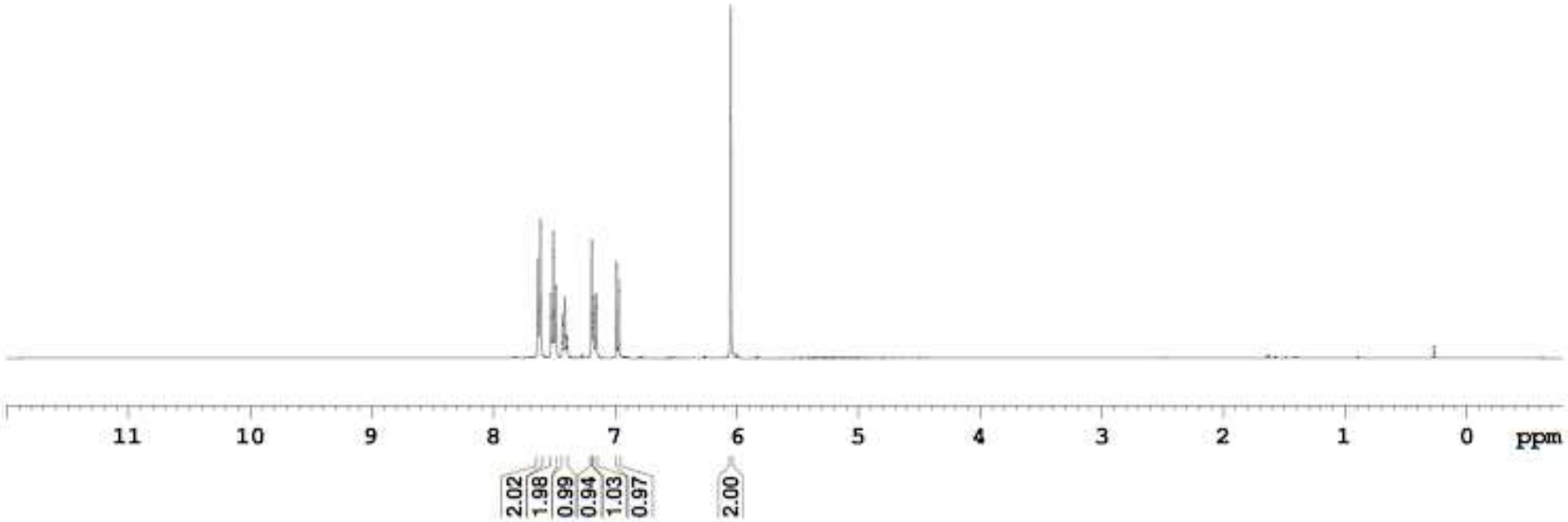
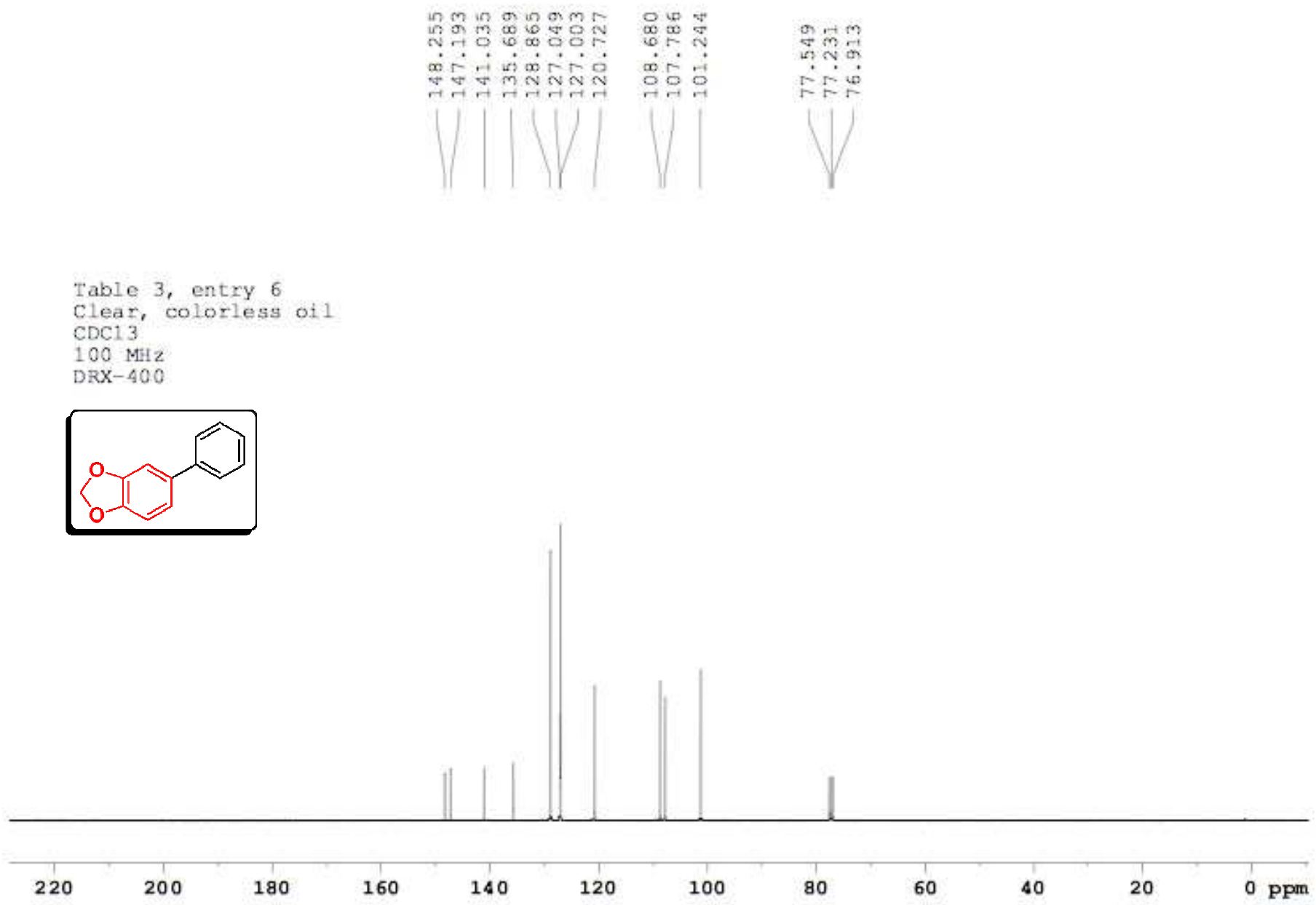
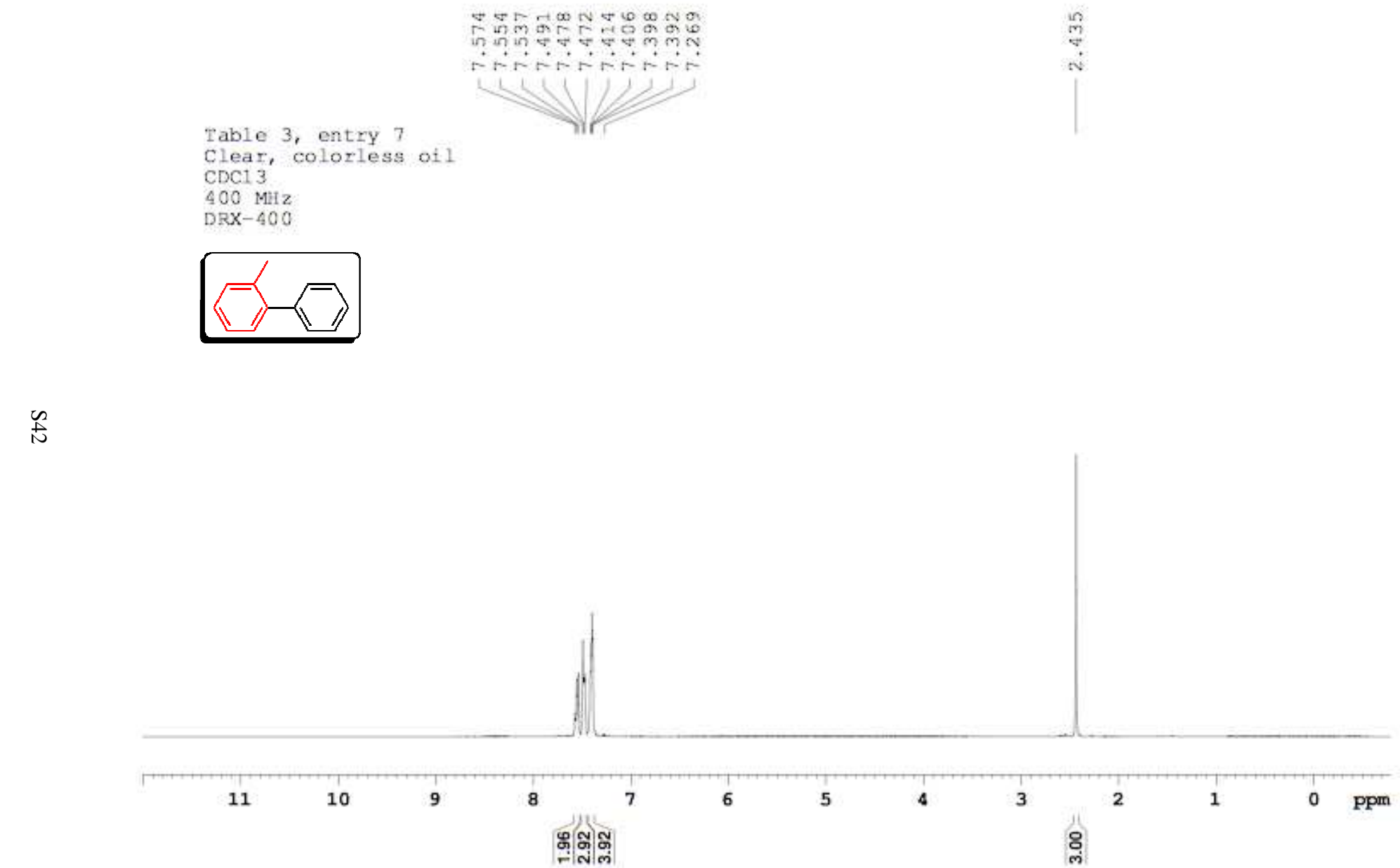


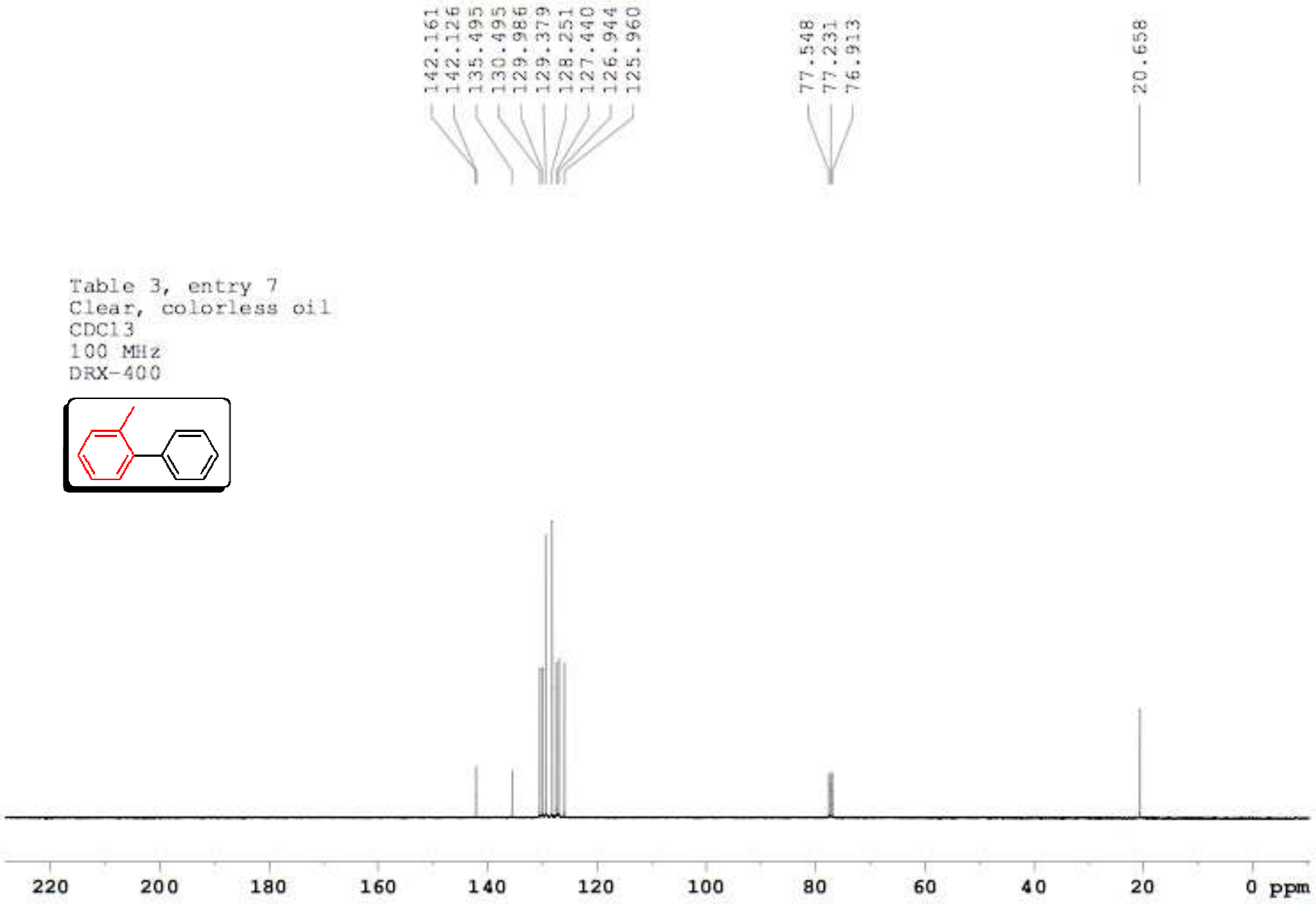
Table 3, entry 6
Clear, colorless oil
CDCl3
400 MHz
DRX-400






148.255
147.193
141.035
135.689
128.865
127.049
127.003
120.727
108.680
107.786
101.244
77.549
77.231
76.913
Table 3, entry 6
Clear, colorless oil
CDCl3
100 MHz
DRX-400
O
O
220
200
180
160
140
120
100
80
60
40
20
0 ppm


7.574
7.554
7.537
7.491
7.478
7.472
7.414
7.406
7.398
7.392
7.269
2.435
Table 3, entry 7
Clear, colorless oil
CDCl3
400 MHz
DRX-400
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
2.92
3.92
3.00


142.161
142.126
135.495
130.495
129.986
129.379
128.251
127.440
126.944
125.960
77.548
77.231
76.913
20.658
Table 3, entry 7
Clear, colorless oil
CDCl3
100 MHz
DRX-400
220
200
180
160
140
120
100
80
60
40
20
0 ppm

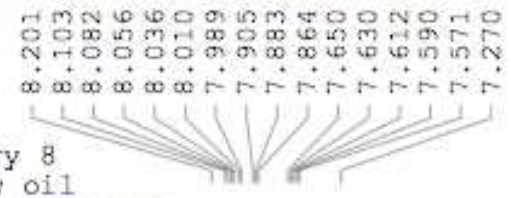
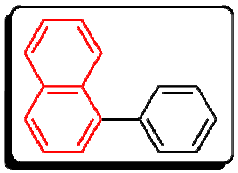
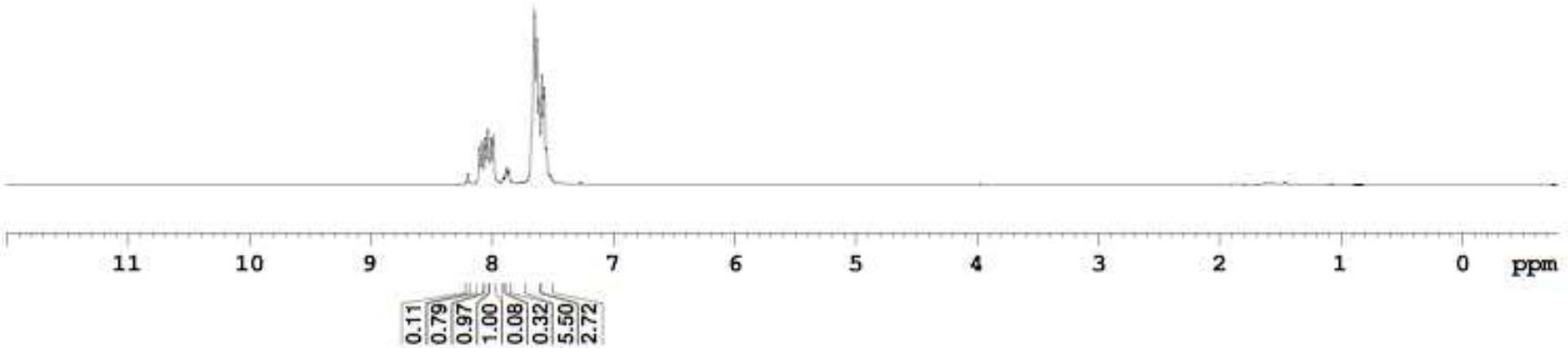


Table 3, entry 8
Light, yellow oil
10% 2-phenylnaphthalene
CDCl3
400 MHz
DRX-400





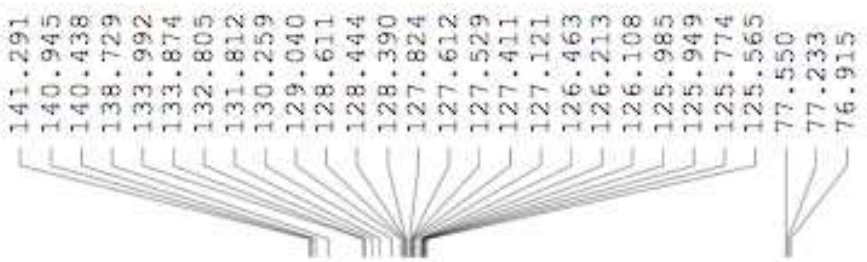
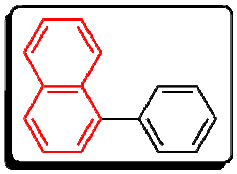
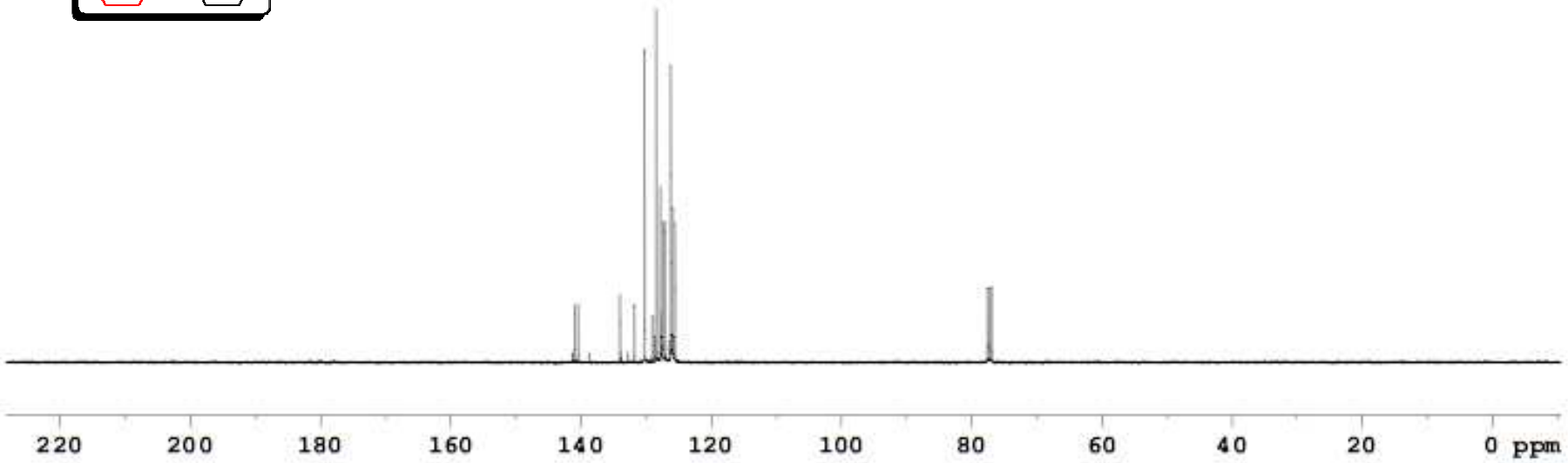
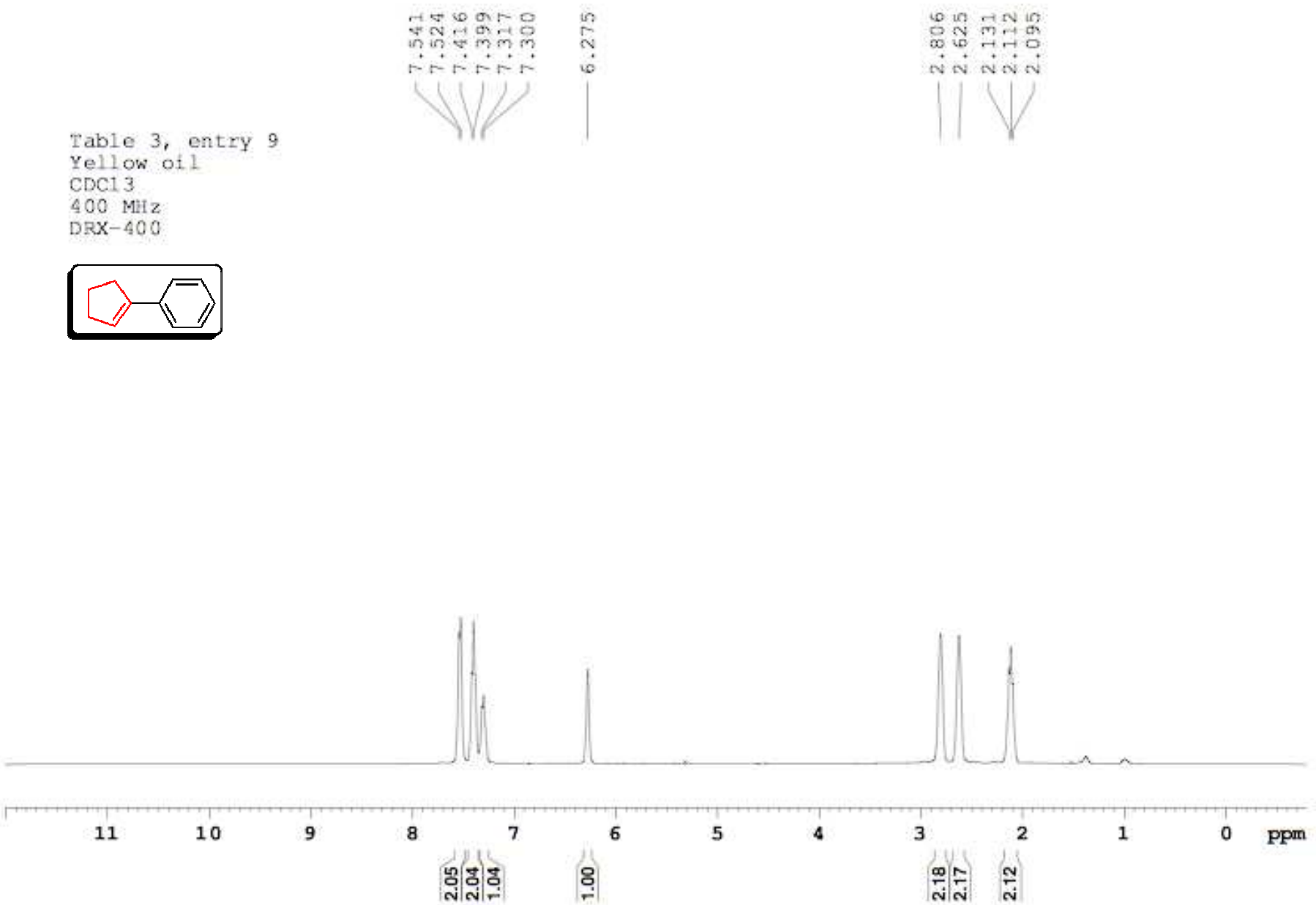
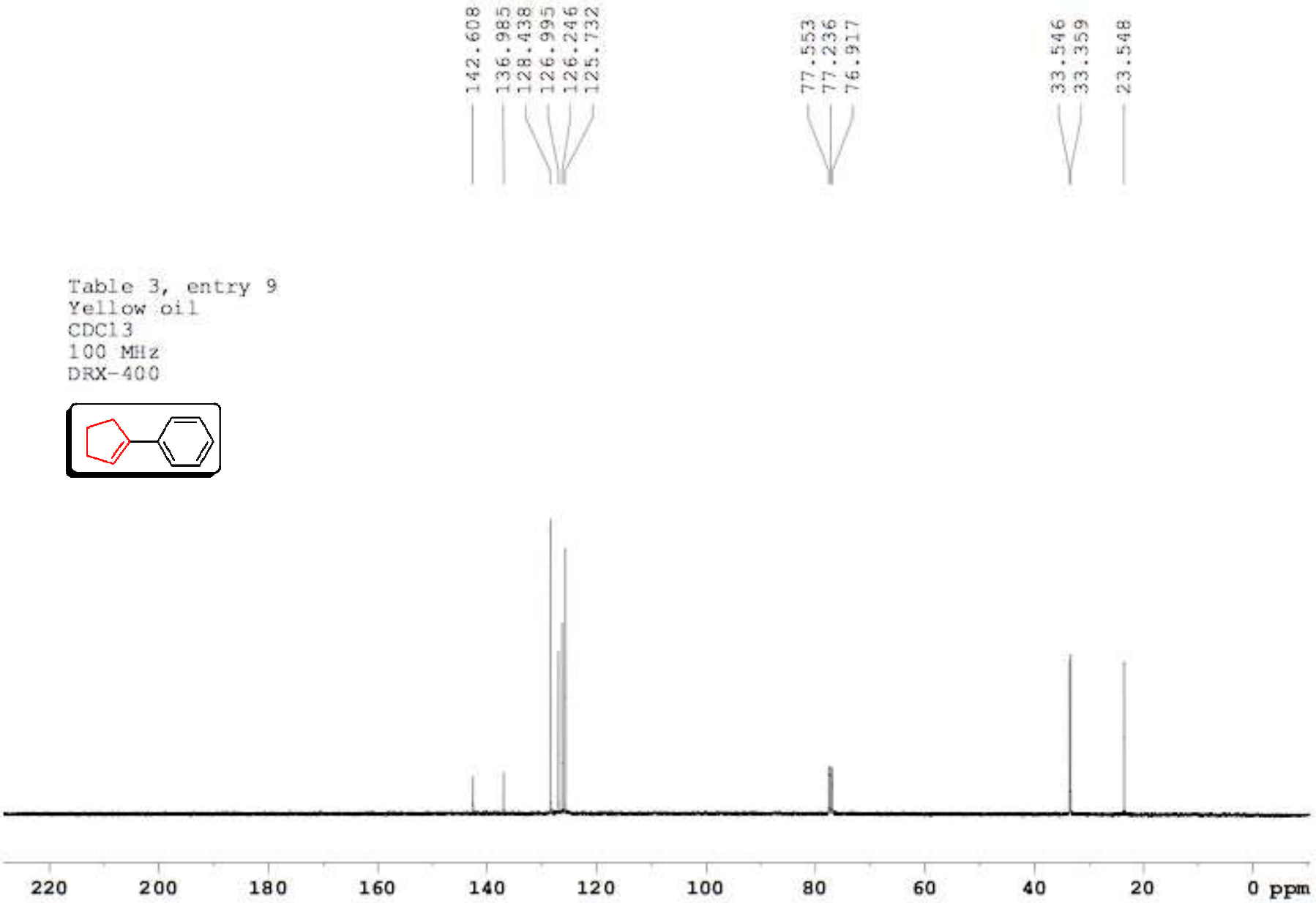


Table 3, entry 8
Light, yellow oil
10% 2-phenylnaphthalene
CDCl3
100 MHz
DRX-400






7.541
7.524
7.416
7.399
7.317
7.300
6.275
2.806
2.625
2.131
2.112
2.095
Table 3, entry 9
Yellow oil
CDCl3
400 MHz
DRX-400
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
2.04
1.04
1.00
2.18
2.17
2.12


142.608
136.985
128.438
126.995
126.246
125.732
77.553
77.236
76.917
33.546
33.359
23.548
Table 3, entry 9
Yellow oil
CDCl3
100 MHz
DRX-400
220
200
180
160
140
120
100
80
60
40
20
0 ppm

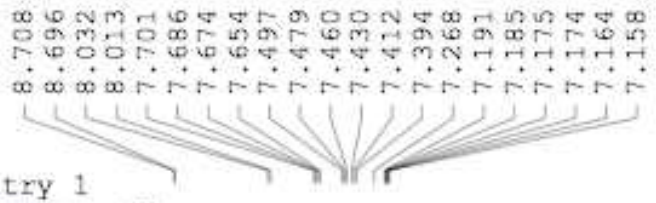
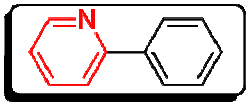
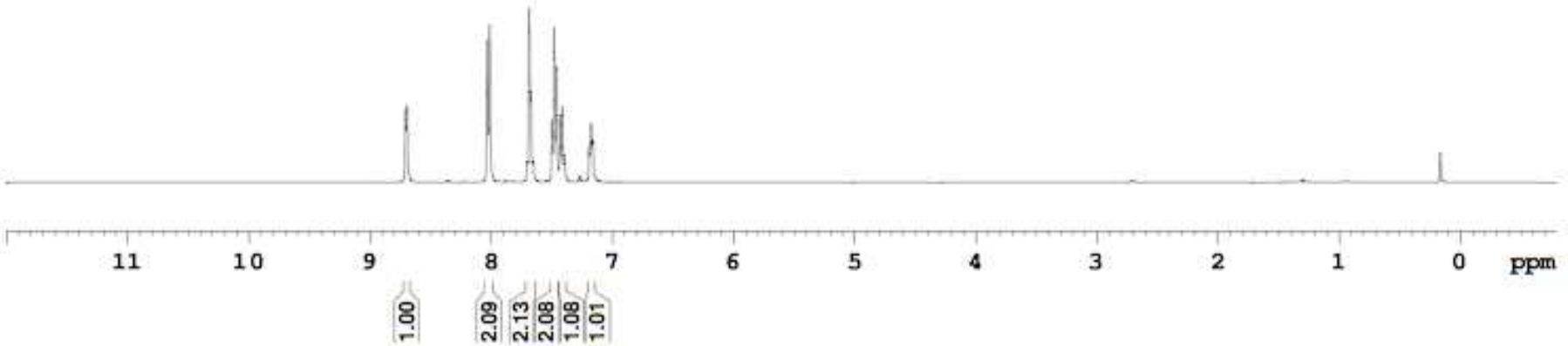
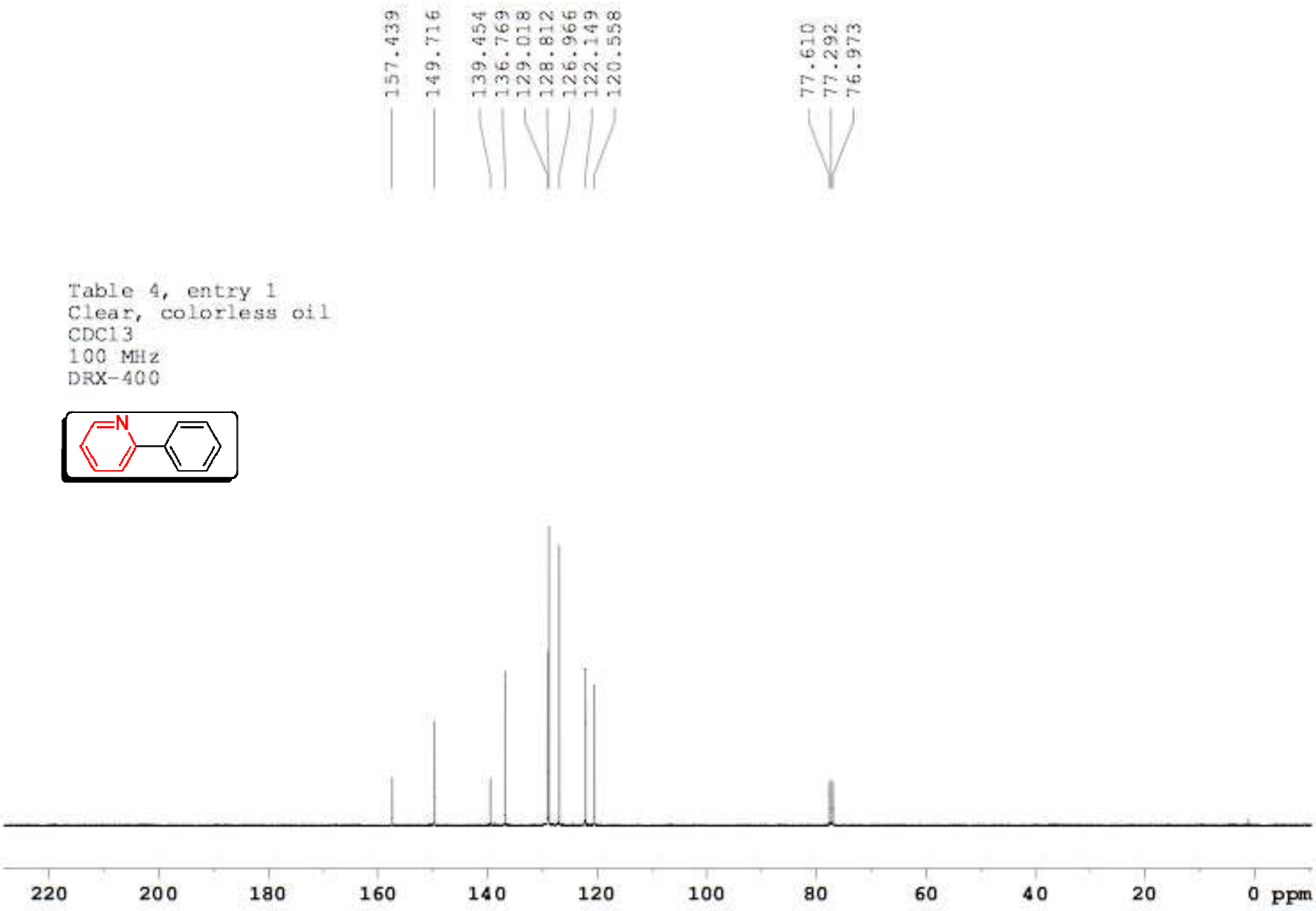


Table 4, entry 1
Clear, colorless oil
CDCl3
400 MHz
DRX-400






157.439
149.716
139.454
136.769
129.018
128.812
126.966
122.149
120.558
77.610
77.292
76.973
Table 4, entry 1
Clear, colorless oil
CDCl3
100 MHz
DRX-400
N
220
200
180
160
140
120
100
80
60
40
20
0 ppm

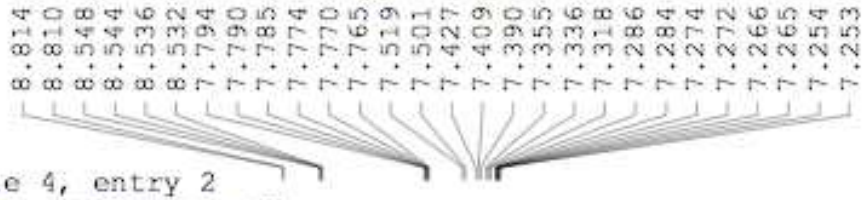
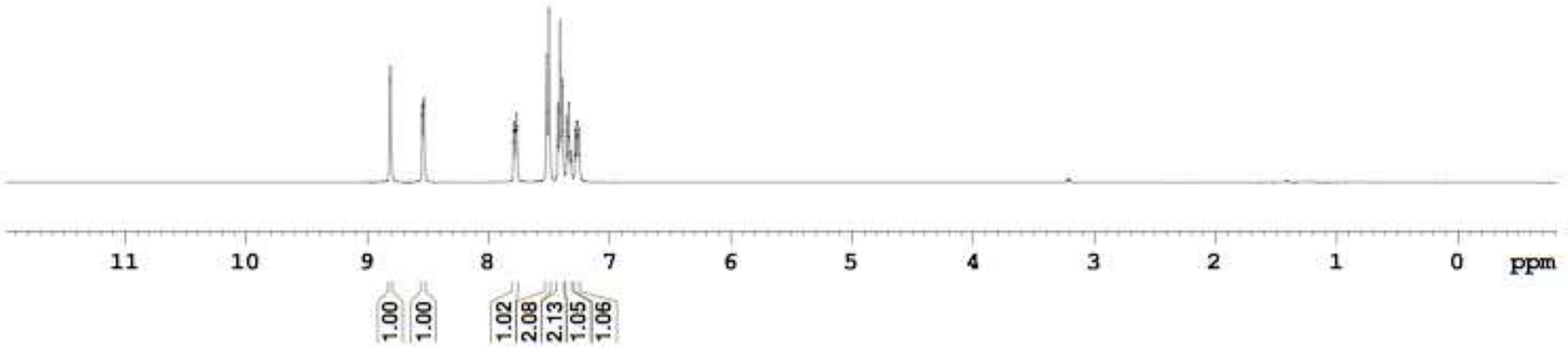
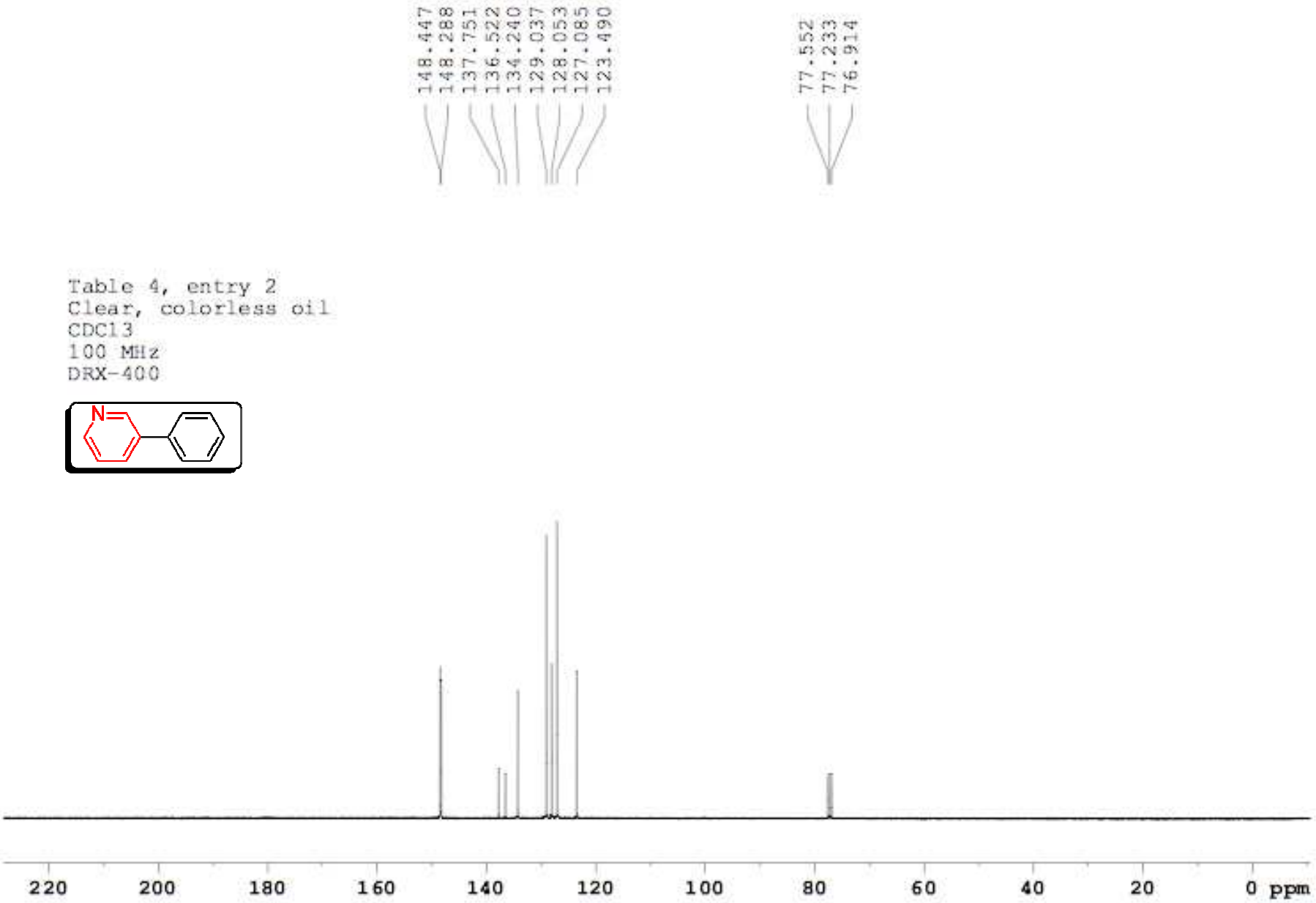


Table 4, entry 2
Clear, colorless oil
CDCl3
400 MHz
DRX-400




148.447
148.288
137.751
136.522
134.240
129.037
128.053
127.085
123.490
77.552
77.233
76.914
Table 4, entry 2
Clear, colorless oil
CDCl3
100 MHz
DRX-400
N
220
200
180
160
140
120
100
80
60
40
20
0 ppm

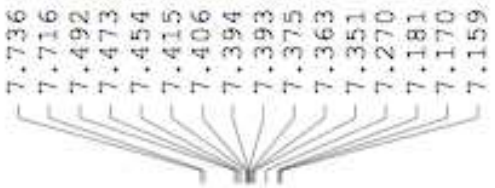
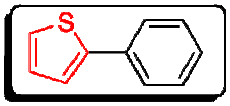
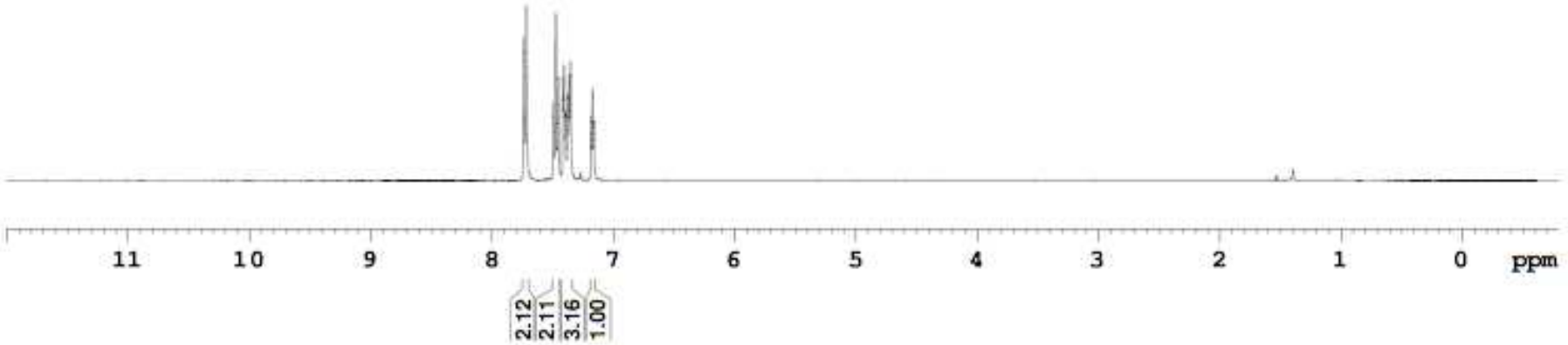


Table 4, entry 3
White solid
CDCl3
400 MHz
DRX-400





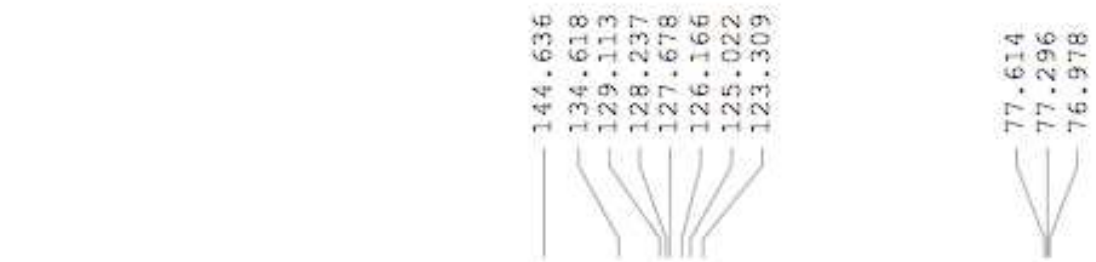
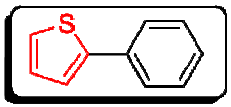
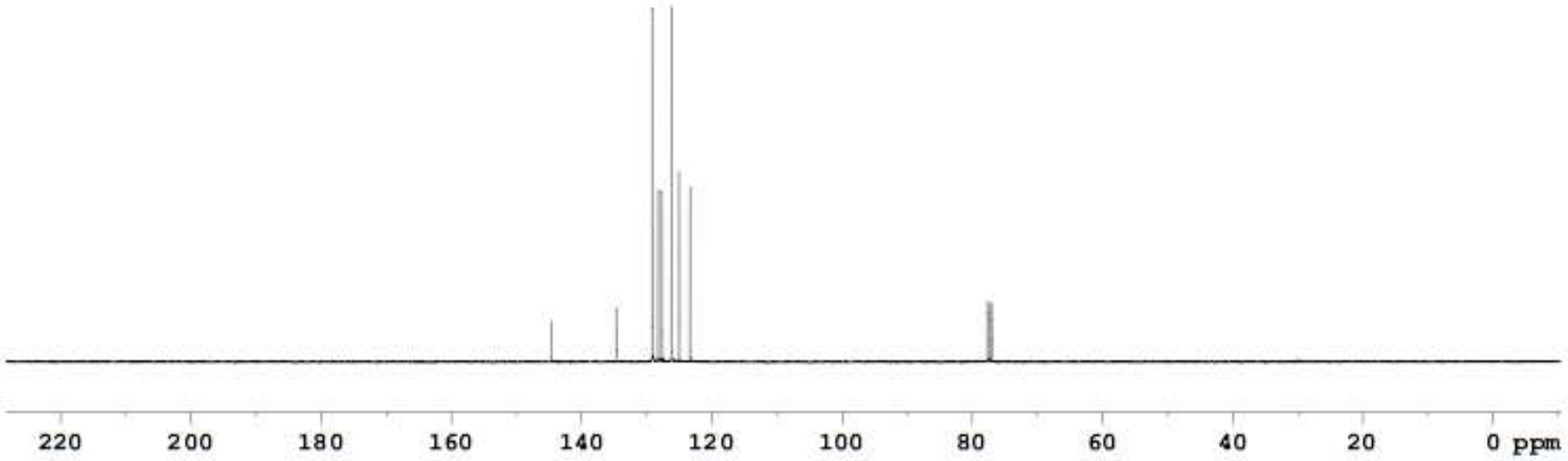
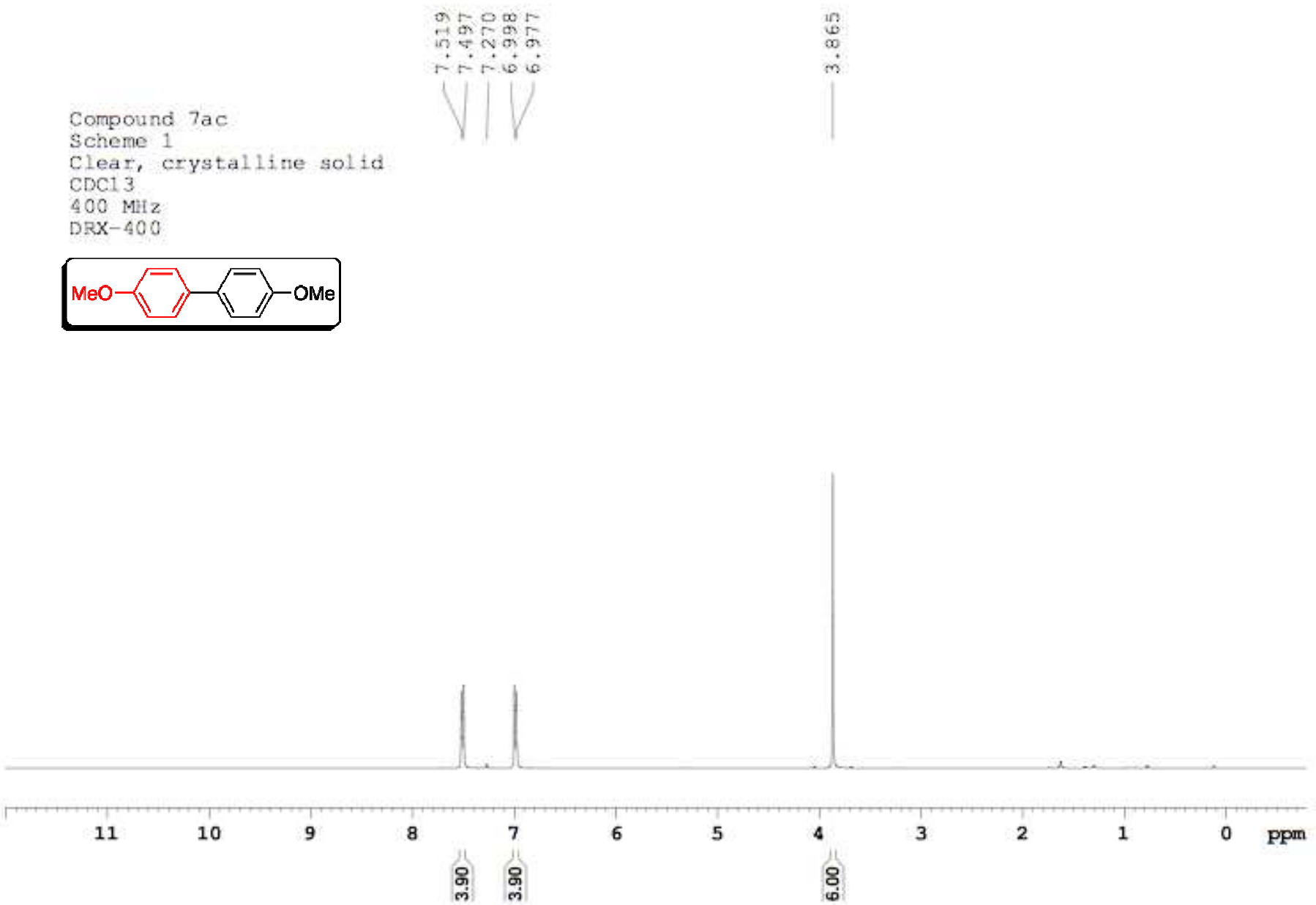
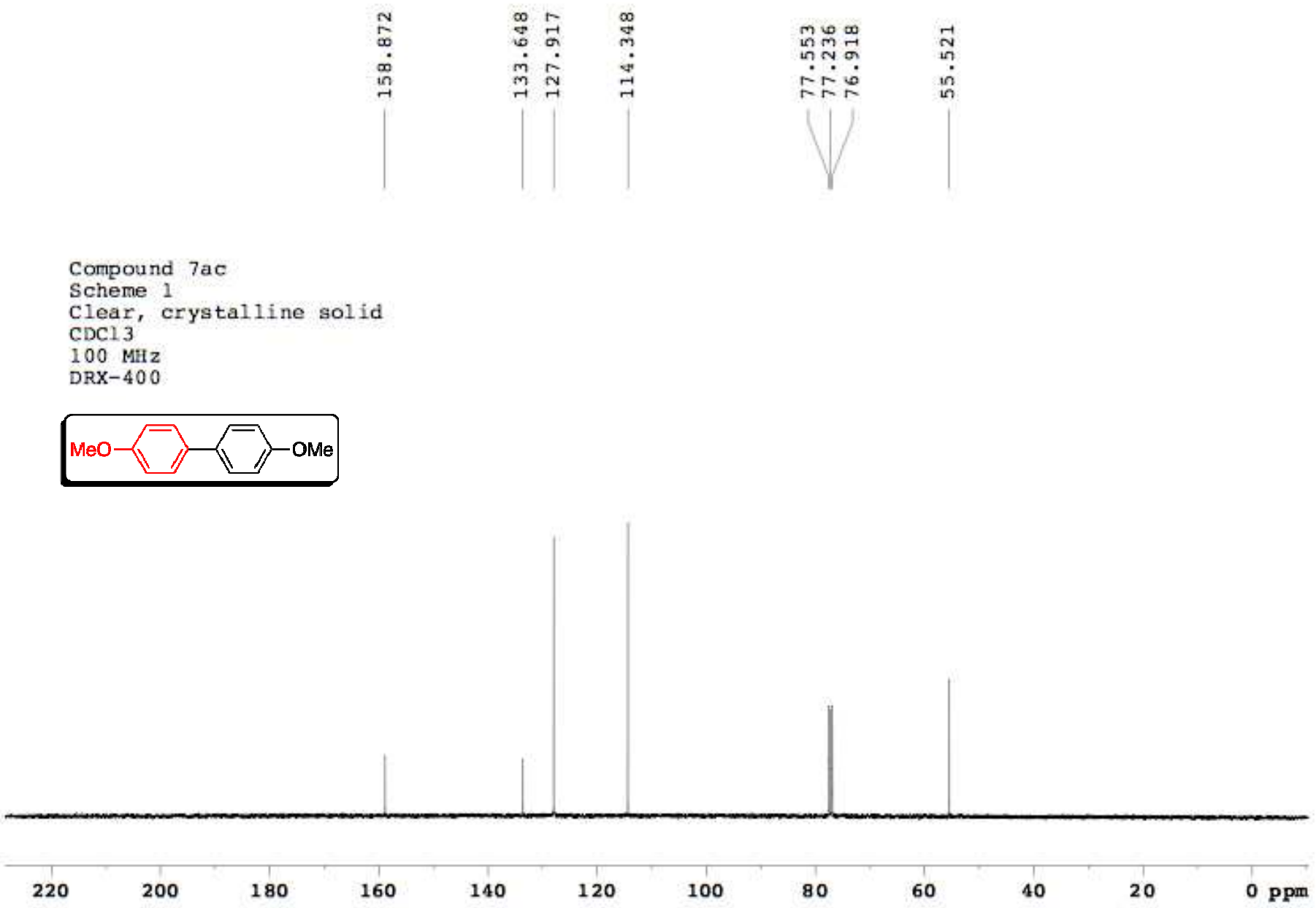


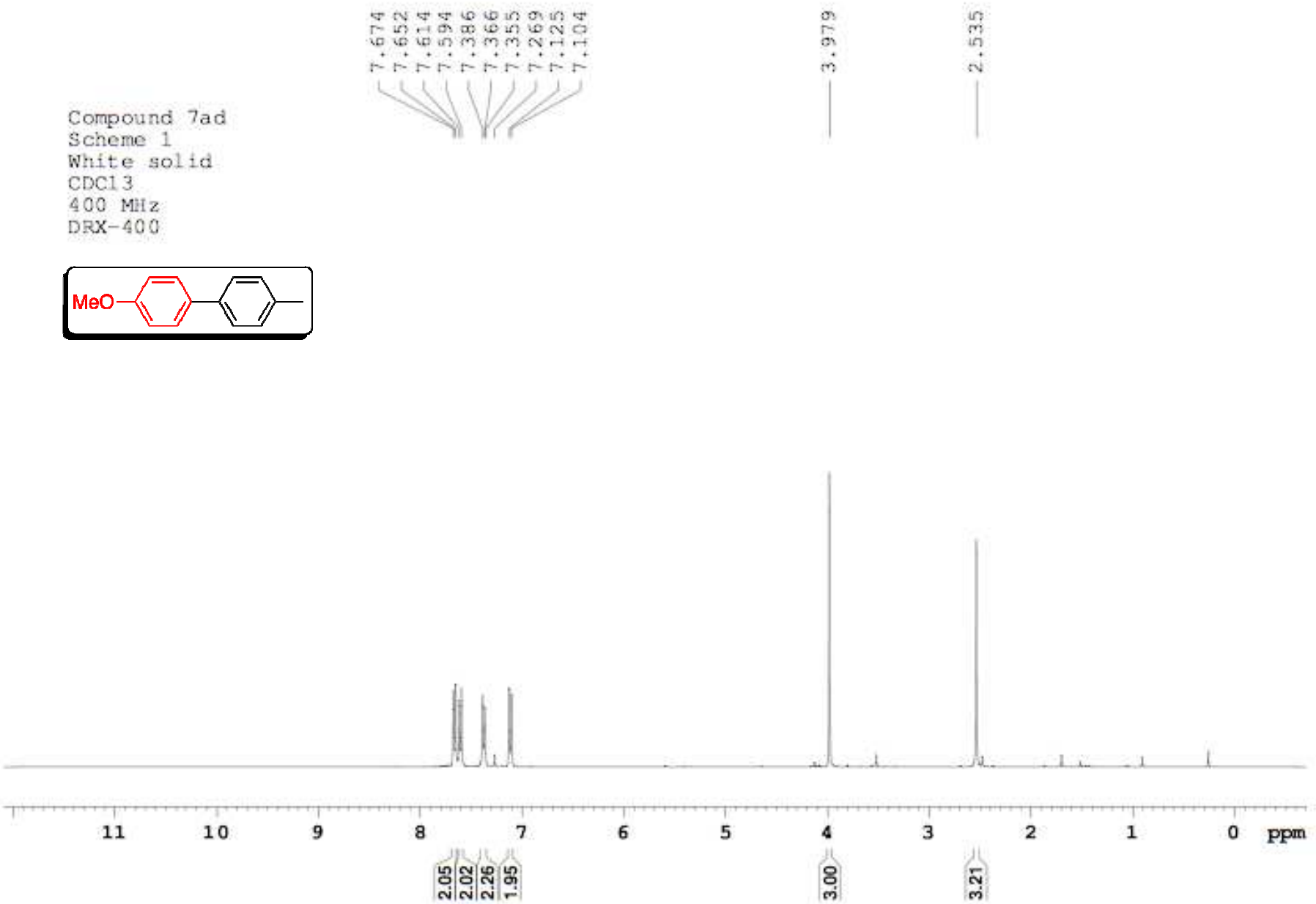
Table 4, entry 3
White solid
CDCl3
100 MHz
DRX-400

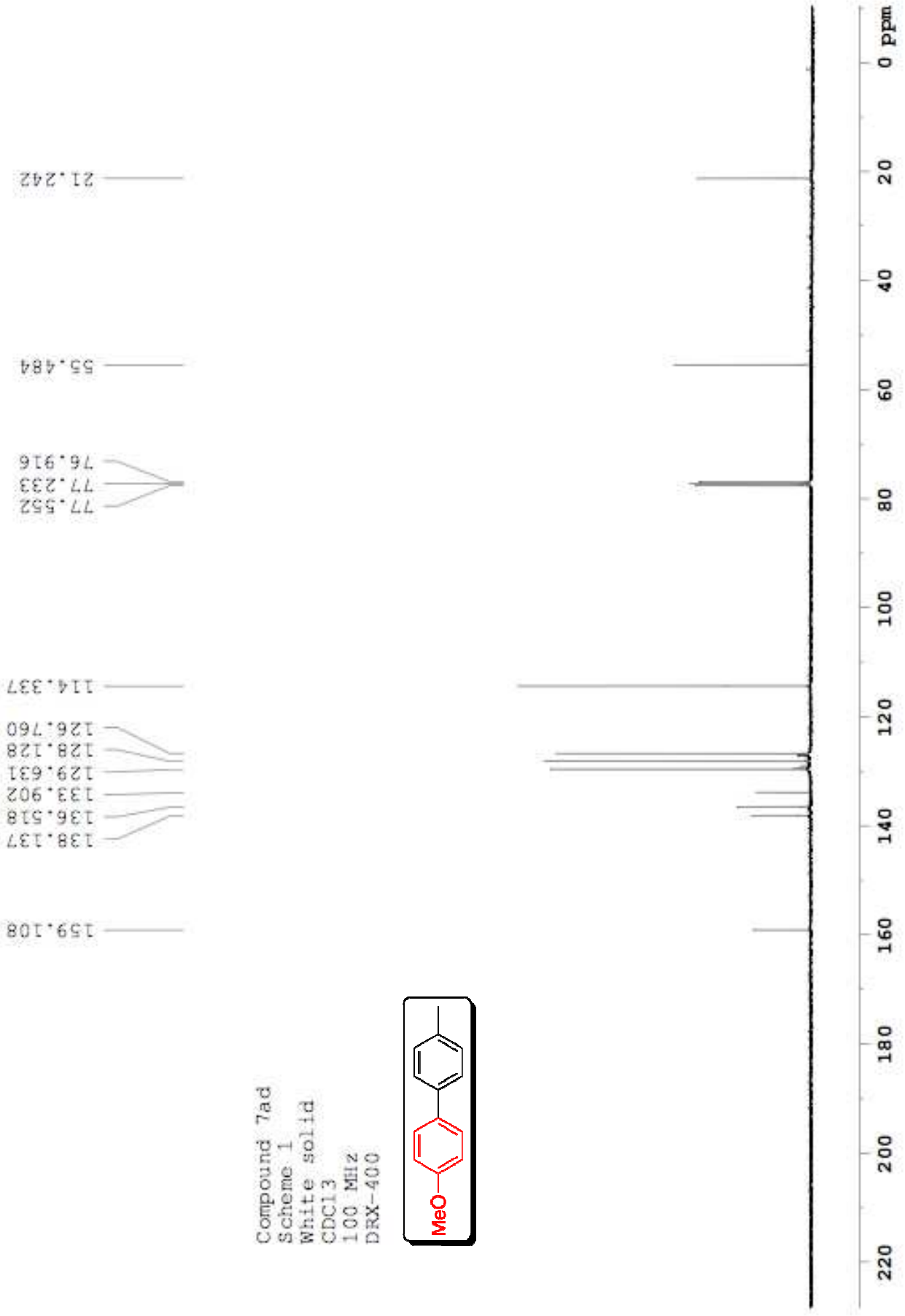


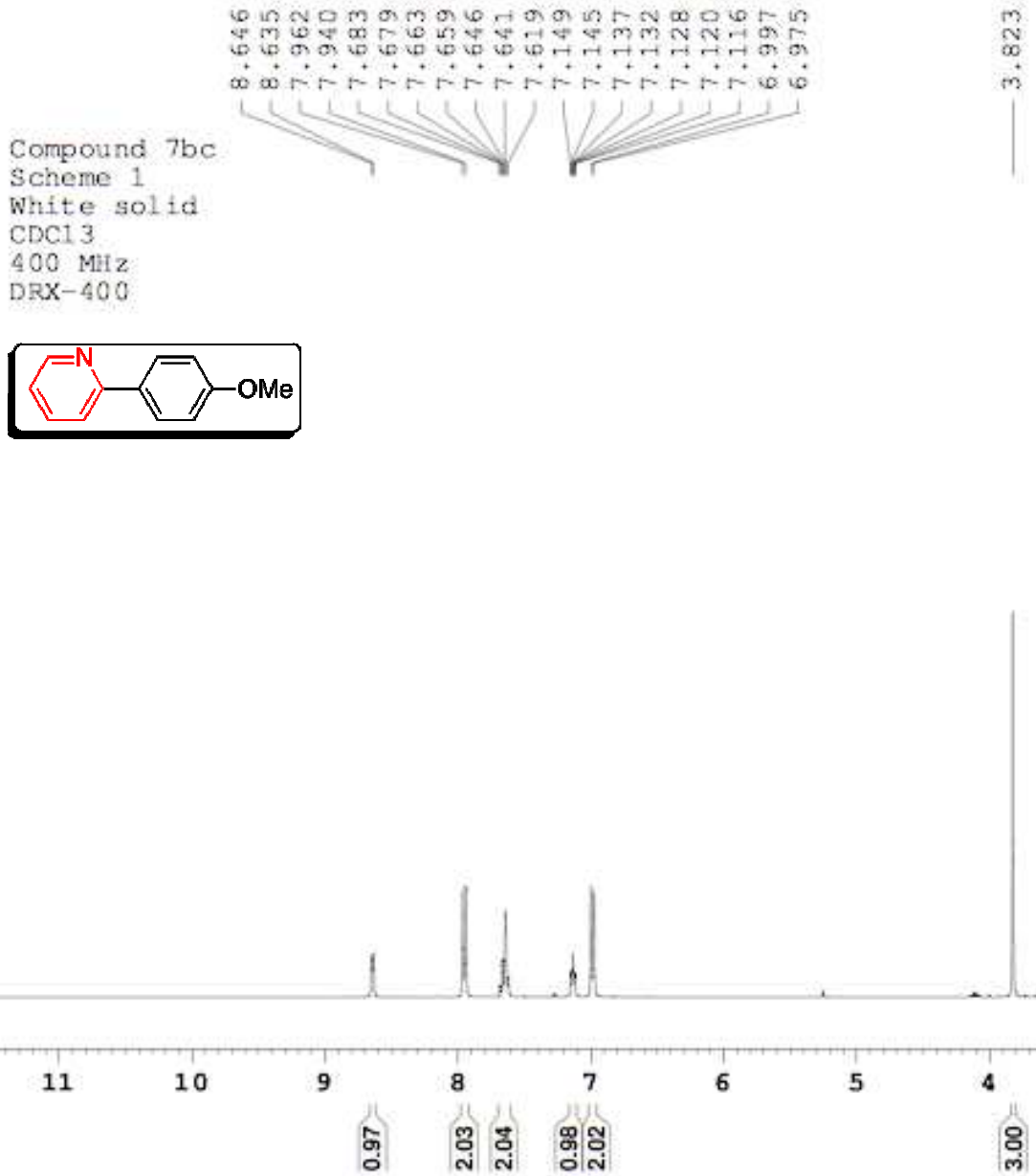


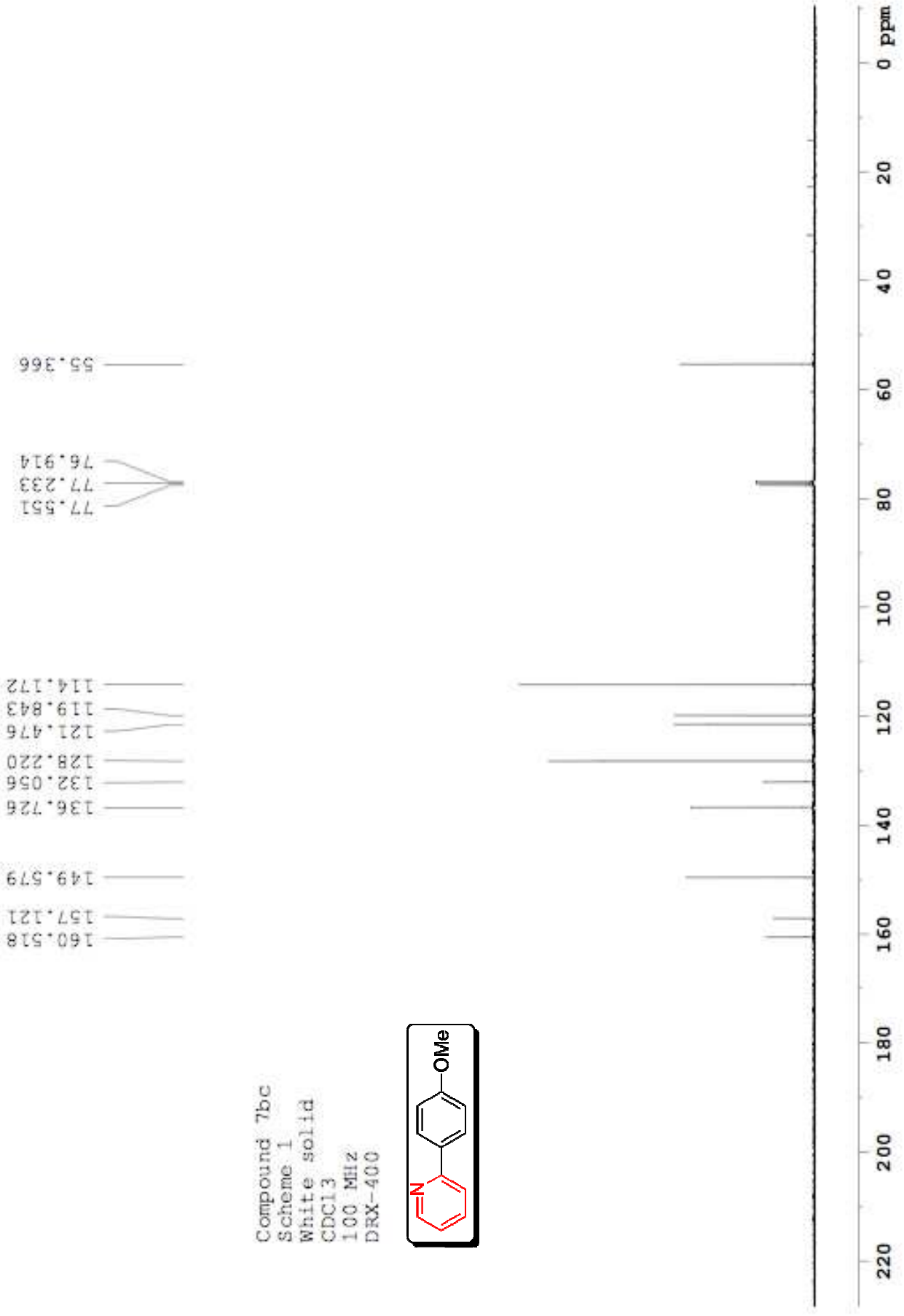

7.519
7.497
7.270
6.998
6.977
3.865
Compound 7ac
Scheme 1
Clear, crystalline solid
CDCl3
400 MHz
DRX-400
MeO
OMe
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.90
3.90
6.00

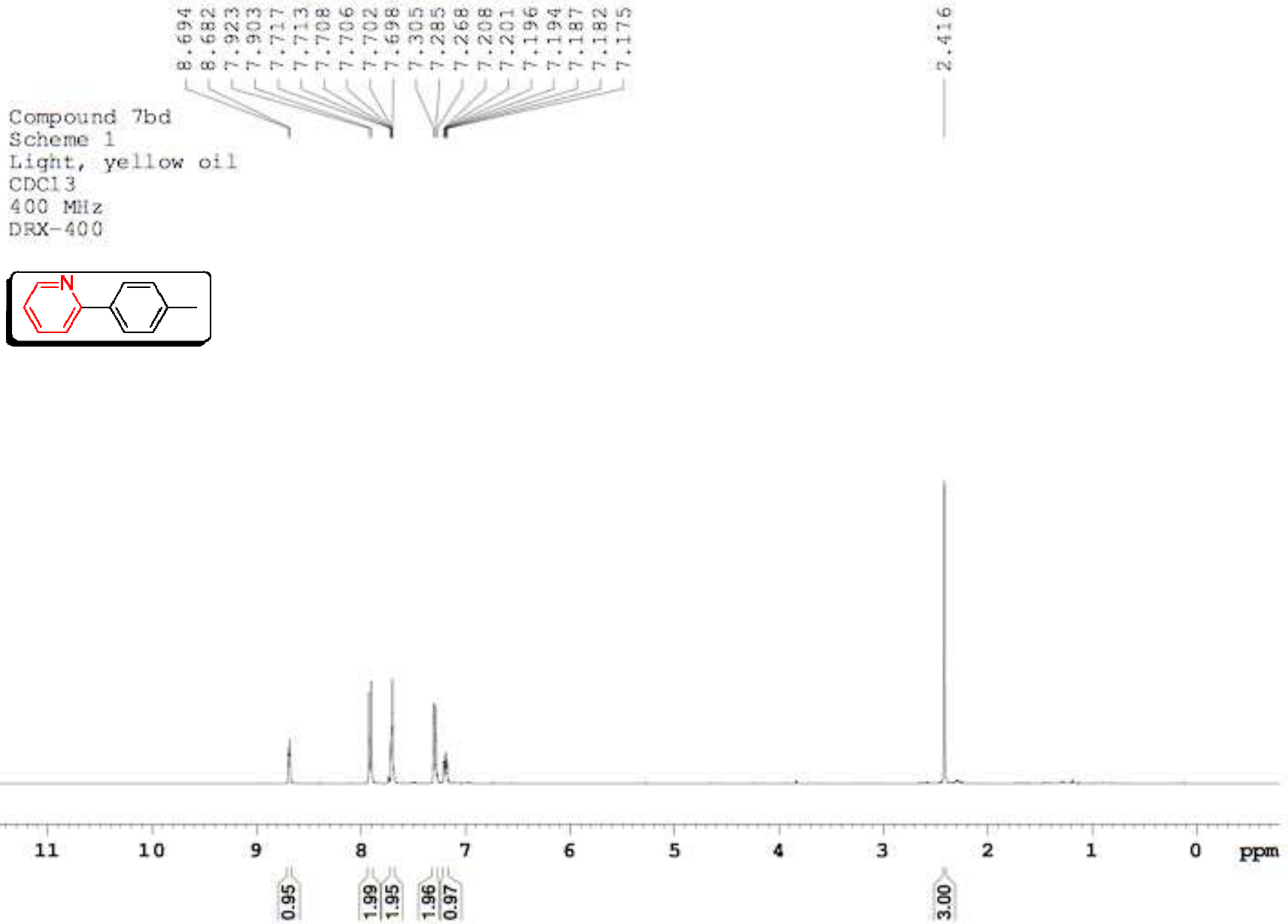

158.872
133.648
127.917
114.348
77.553
77.236
76.918
55.521
Compound 7ac
Scheme 1
Clear, crystalline solid
CDCl3
100 MHz
DRX-400
MeO
OMe
220
200
180
160
140
120
100
80
60
40
20
0 ppm

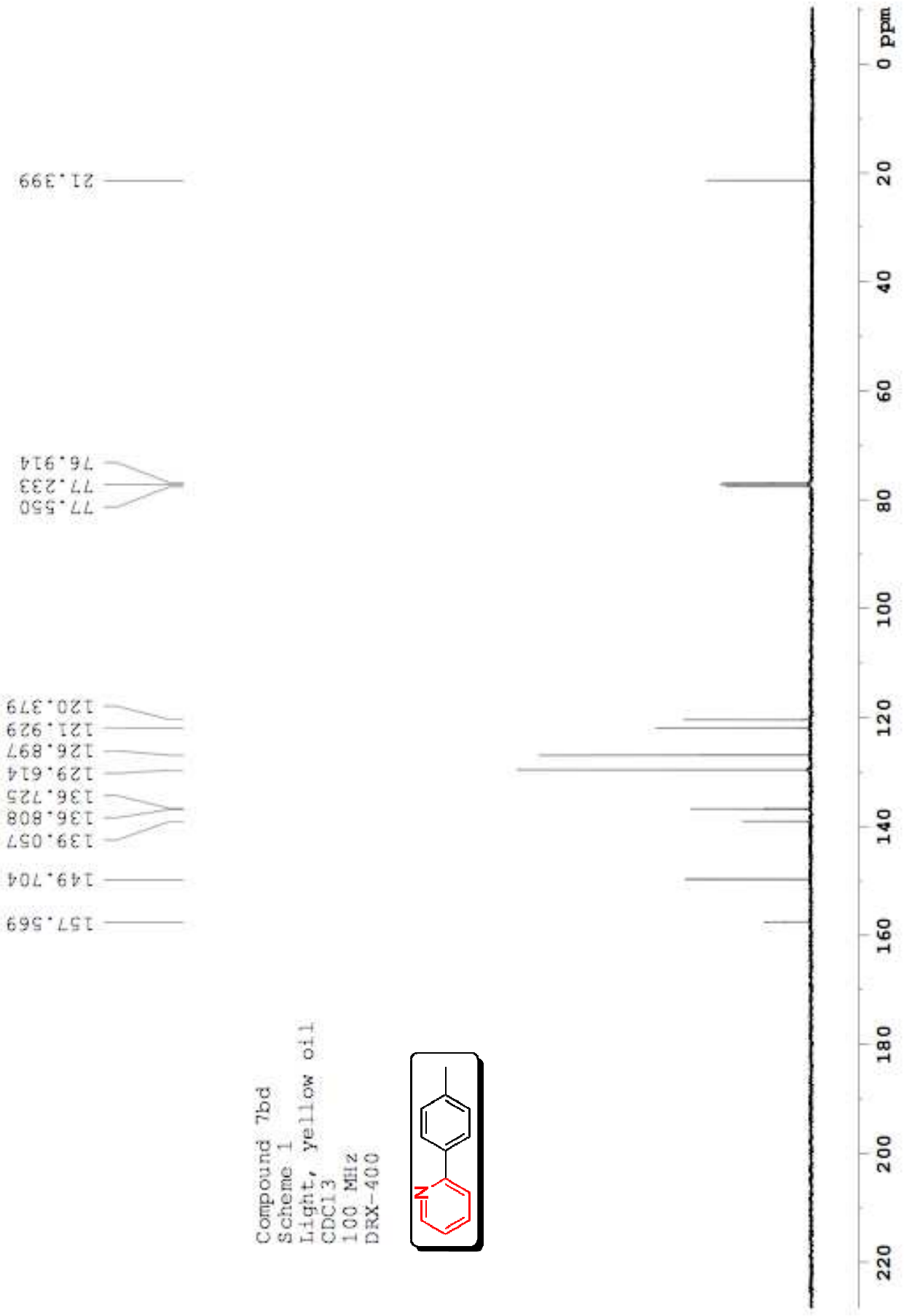

7.674
7.652
7.614
7.594
7.386
7.366
7.355
7.269
7.125
7.104
3.979
2.535
Compound 7ad
Scheme 1
White solid
CDCl3
400 MHz
DRX-400
MeO
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
2.02
2.26
1.95
3.00
3.21


Compound 7ad
Scheme 1
White solid
CDCl3
100 MHz
DRX-400
MeO
21.242
55.484
76.916
77.233
77.552
114.337
126.760
128.128
129.631
133.902
136.518
138.137
159.108
0 ppm
20
40
60
80
100
120
140
160
180
200
220


8.646
8.635
7.962
7.940
7.683
7.679
7.663
7.659
7.646
7.641
7.619
7.149
7.145
7.137
7.132
7.128
7.120
7.116
6.997
6.975
3.823
Compound 7bc
Scheme 1
White solid
CDCl3
400 MHz
DRX-400
N
OMe
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
2.03
2.04
0.98
2.02
3.00


55.366
76.914
77.233
77.551
114.172
119.843
121.476
128.220
132.056
136.726
149.579
157.121
160.518
Compound 7bc
Scheme 1
White solid
CDCl3
100 MHz
DRX-400
OMe
N
0 ppm
20
40
60
80
100
120
140
160
180
200
220


8.694
8.682
7.923
7.903
7.717
7.713
7.708
7.706
7.702
7.698
7.305
7.285
7.268
7.208
7.201
7.196
7.194
7.187
7.182
7.175
2.416
Compound 7bd
Scheme 1
Light, yellow oil
CDCl3
400 MHz
DRX-400
N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.99
1.95
1.96
0.97
3.00


Compound 7bd
Scheme 1
Light, yellow oil
CDCl3
100 MHz
DRX-400
N
157.569
149.704
139.057
136.808
136.725
129.614
126.897
121.929
120.379
77.550
77.233
76.914
21.399
0 ppm
20
40
60
80
100
120
140
160
180
200
220