Supplementary Material (ESI) for Organic & Biomolecular Chemistry

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Supporting Information

Palladium-Catalyzed Direct *ortho*-Ethoxycarbonylation of Azobenzenes and Azoxybenzenes with Diethyl Azodicarboxylate

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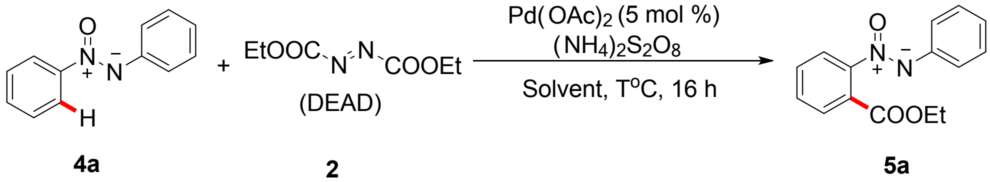
Table of Contents for Supporting Information

1. Optimization of solvents and temperature on the ethoxycarbonylation of azoxybenzenes with diethyl azodicarboxylate (Table S1)

Table S1 Optimization of the solvents and temperature[a]



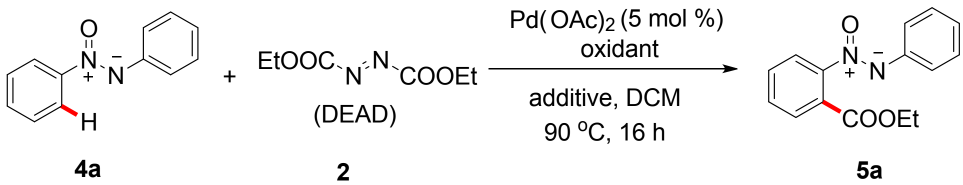
Entry	Solvent	T (°C)	Yield (%)[b]
1	DMF	90	0
2	DMSO	90	0
3	DMA	90	0
4	DCE	90	0
5	HOAc	90	38
6	THF	90	0
7	Toluene	90	0
8	CH_3CN	90	0
9	NMP	90	0
10	1,4-Dioxane	90	0
11	DME	90	0
12	DCM	90	59
13	DCM	110	46
14	DCM	60	41
15	DCM	40	38
16	DCM	rt	36

[a] *Reaction conditions:* azoxybenzene (**4a**, 0.20 mmol), diethyl azodicarboxylate (**2**, 0.50 mmol), $Pd(OAc)_2$ (5 mol%), $(NH_4)_2S_2O_8$ (0.30 mmol) under air at the temperature indicated in TS1 for 16 h.

[b] Isolated yield.

2. Optimization of oxidants and additives on the ethoxycarbonylation of azoxybenzenes with diethyl azodicarboxylate (Table S2)

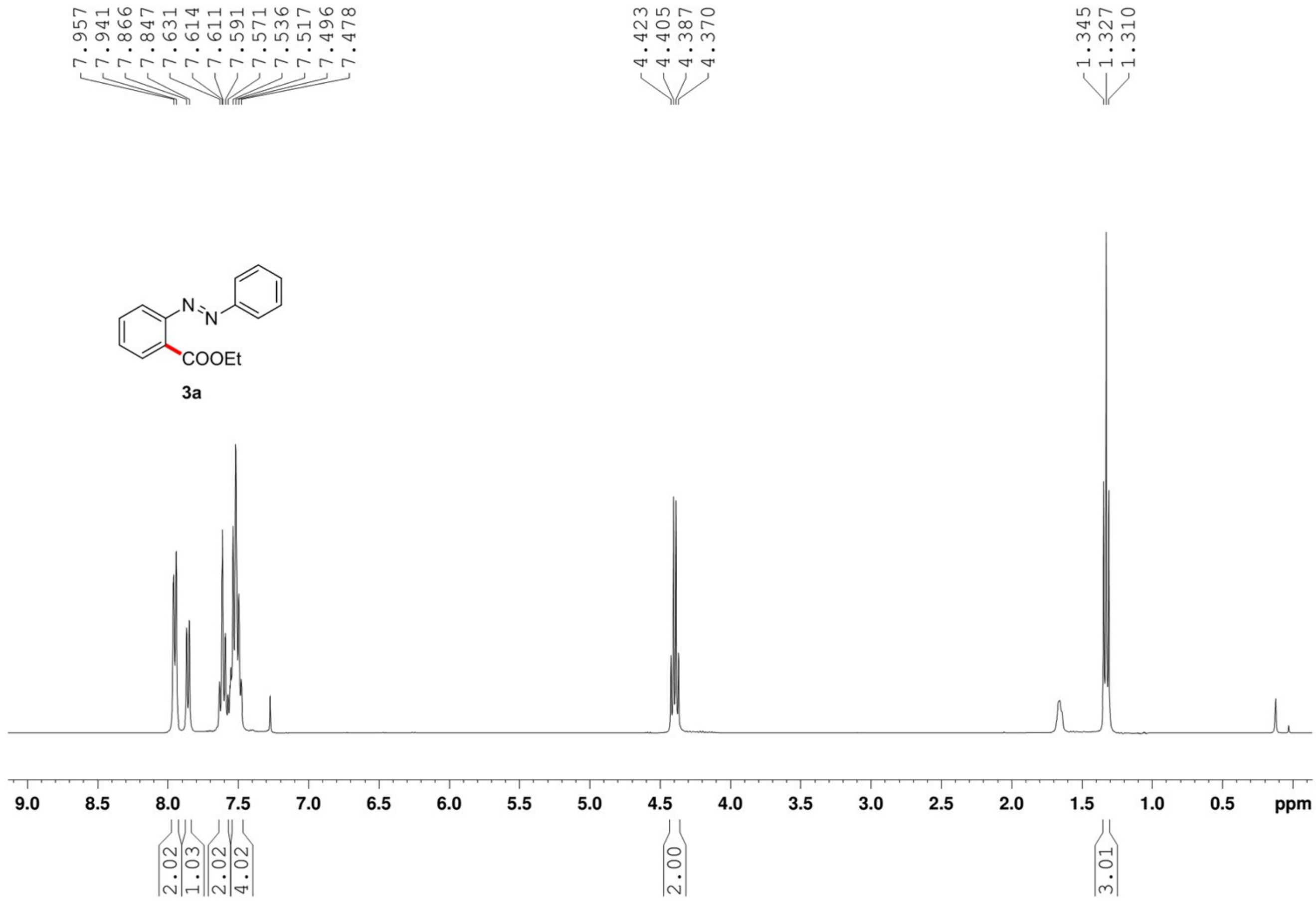
Table S2 Optimization of oxidants and additives[a]

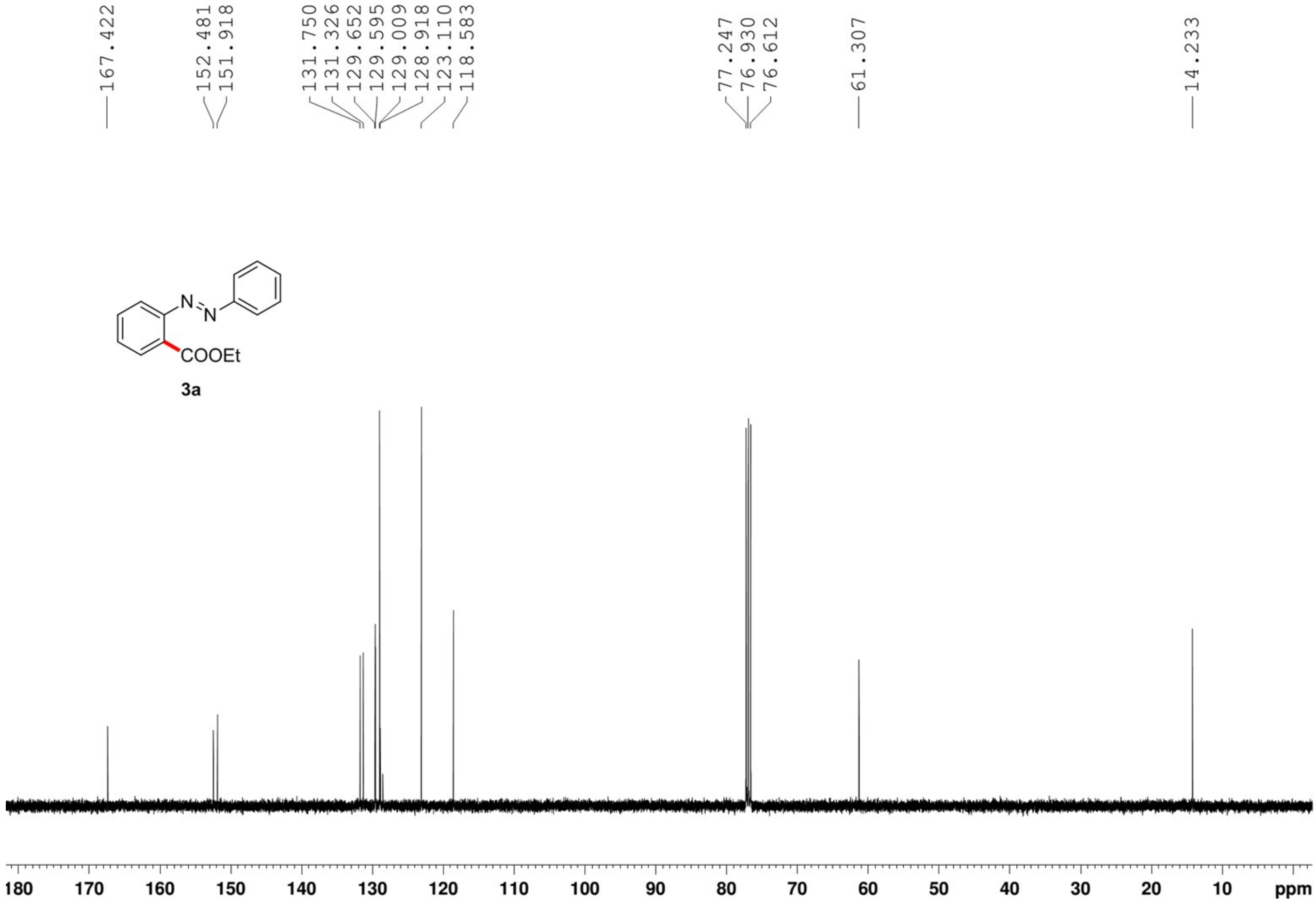


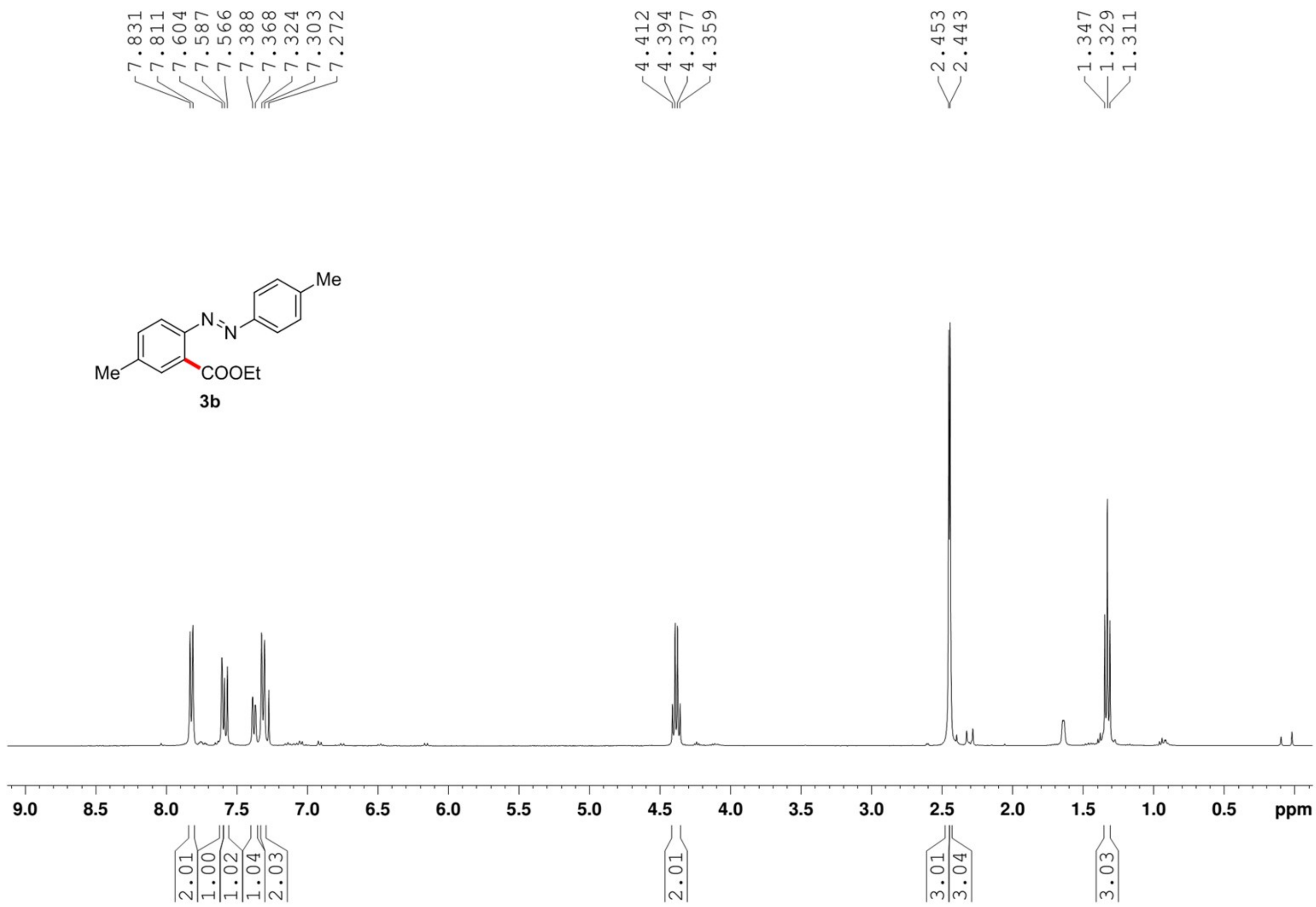
Entry	Oxidant	Additive	Yield (%) [b]
1	$Cu(OAc)_2$	–	0
2	$(NH_4)_2S_2O_8$	–	59
3	$K_2S_2O_8$	–	trace
4	BQ	–	trace
5	TBHP	–	trace
6	Ag_2O	–	0
7	$PhI(OAc)_2$	–	trace
8	$Na_2S_2O_8$	–	38
9	DDQ	–	trace
10	DTBP	–	0
11	$(NH_4)_2S_2O_8$	K_2CO_3	0
12	$(NH_4)_2S_2O_8$	Et_3N	0
13	$(NH_4)_2S_2O_8$	HOAc	31
14	$(NH_4)_2S_2O_8$	CF_3SO_3H	trace
15	$(NH_4)_2S_2O_8$	CH_3COOH	24
16	$(NH_4)_2S_2O_8$	CH_3SO_3H	35
17	$(NH_4)_2S_2O_8$	PivOH	trace
18	$(NH_4)_2S_2O_8$	*p*-TsOH	71
19 [c]	$(NH_4)_2S_2O_8$	*p*-TsOH	61
20 [d]	$(NH_4)_2S_2O_8$	*p*-TsOH	69
21 [e]	$(NH_4)_2S_2O_8$	*p*-TsOH	49
22 [f]	$(NH_4)_2S_2O_8$	*p*-TsOH	70

[a] *Reaction conditions:* azoxybenzene (**4a**, 0.20 mmol), diethyl azodicarboxylate (**2**, 0.50 mmol), $Pd(OAc)_2$ (5 mol%), oxidant (0.30 mmol), additive (0.40 mmol) in DCM (2.0 mL) at 90 °C under air for 16 h. [b] Isolated yield. [c] 12 h. [d] 24 h. [e] **4a**/**2** = 1:1. [f] **4a**/**2** = 1:3.

3. ^{1}H and ^{13}C spectra of the products



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2.443
1.347
1.329
1.311
Me
N
N
Me
COOEt
3b
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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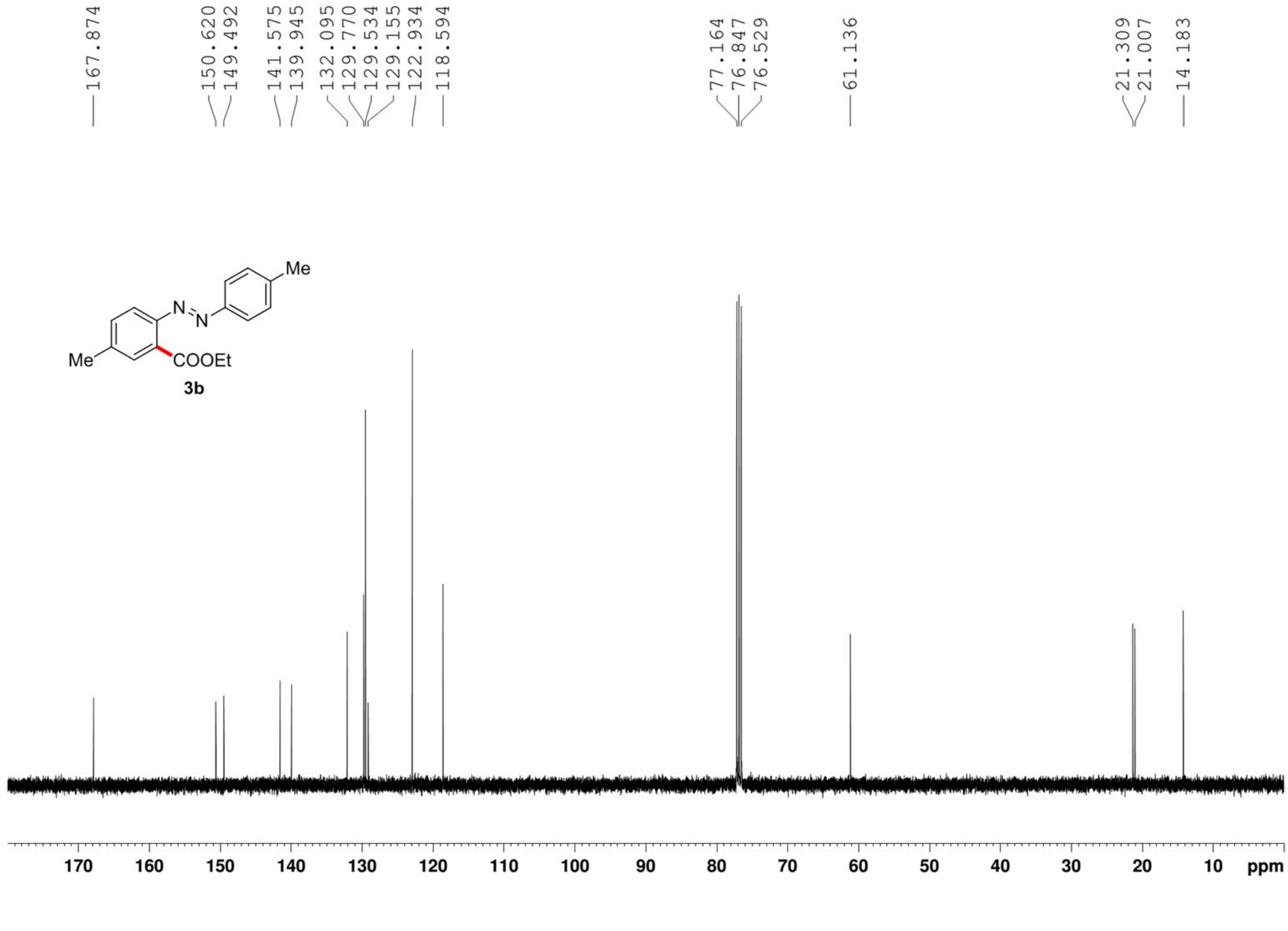
3.03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76.529
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21.007
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N
Me
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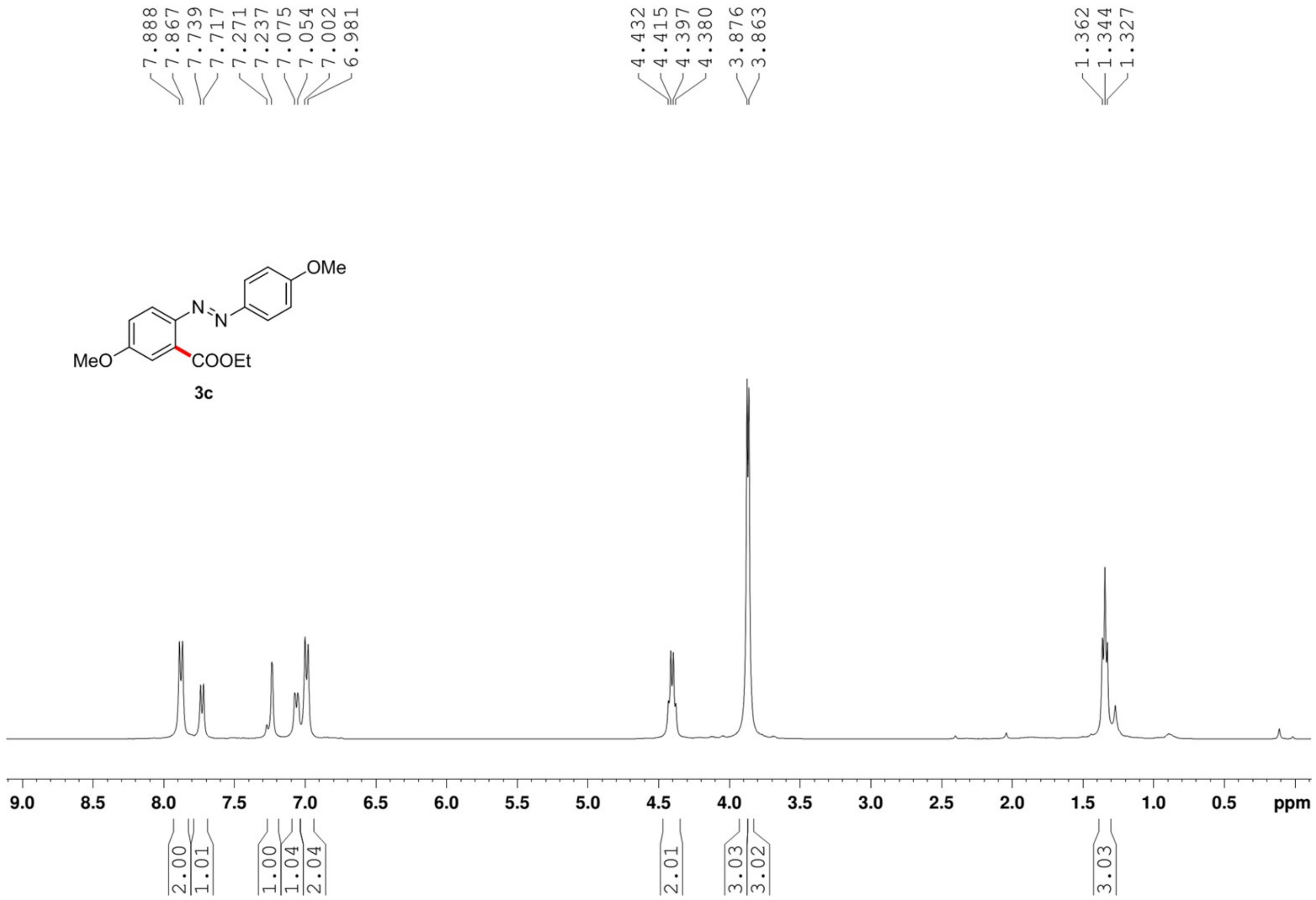
10
ppm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4.397
4.380
3.876
3.863
1.362
1.344
1.327
OMe
N
N
MeO
COOEt
3c
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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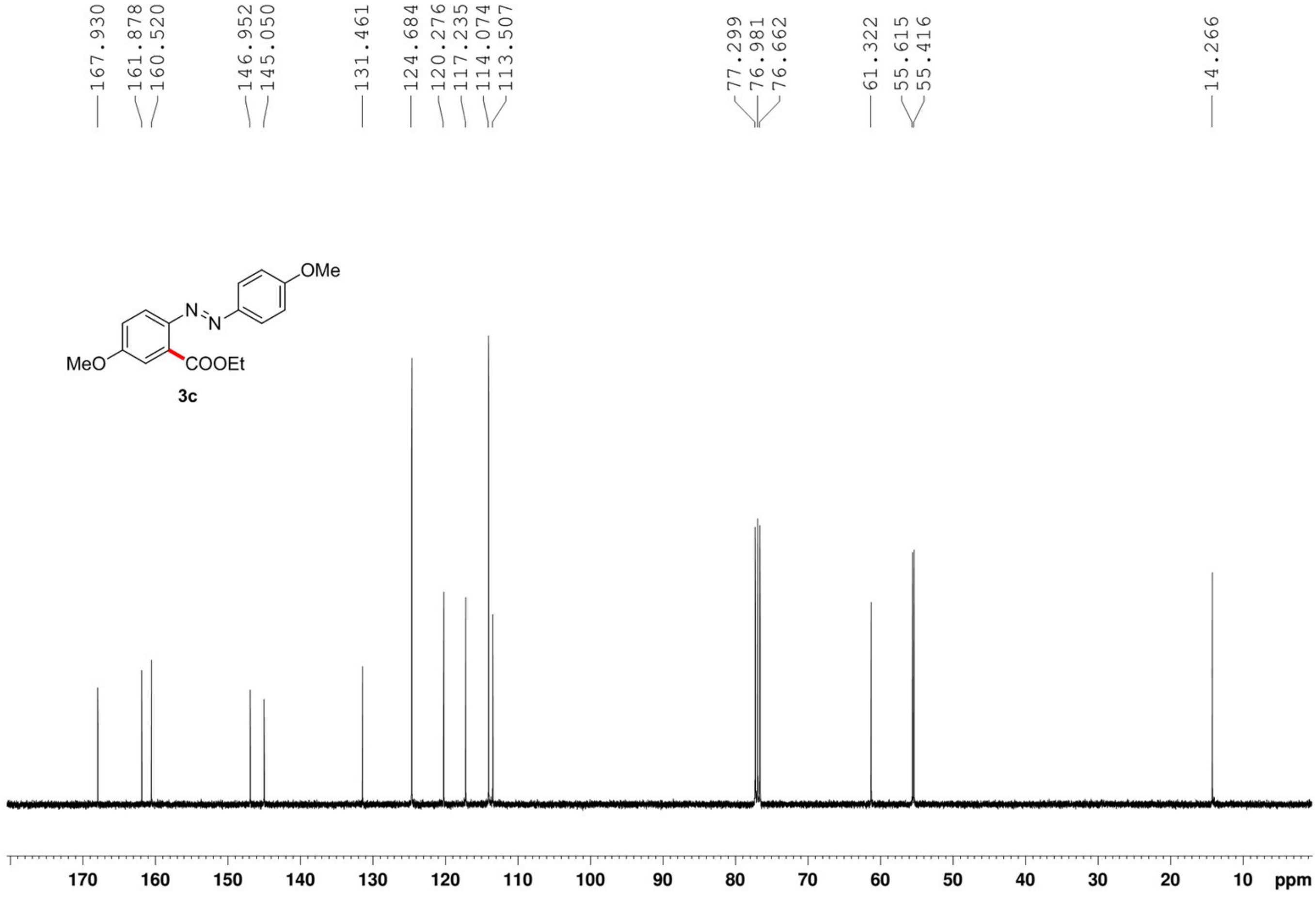
3.03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76.662
61.322
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14.266
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N
MeO
COOEt
3c
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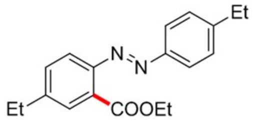

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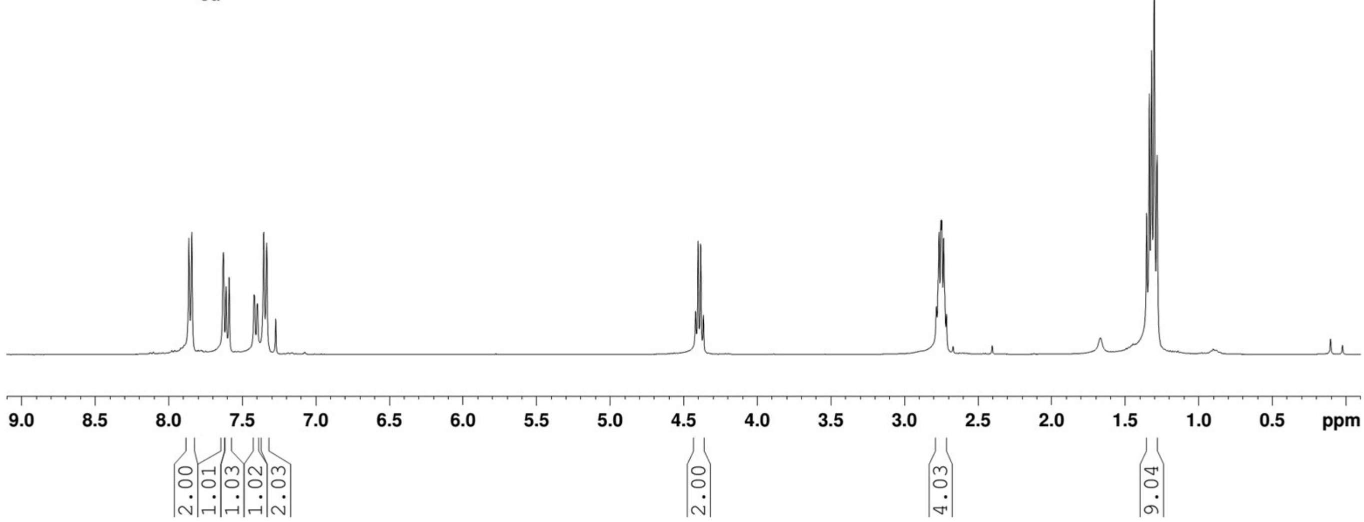
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4.367
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2.765
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2.734
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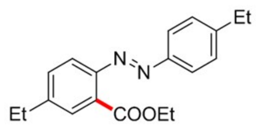

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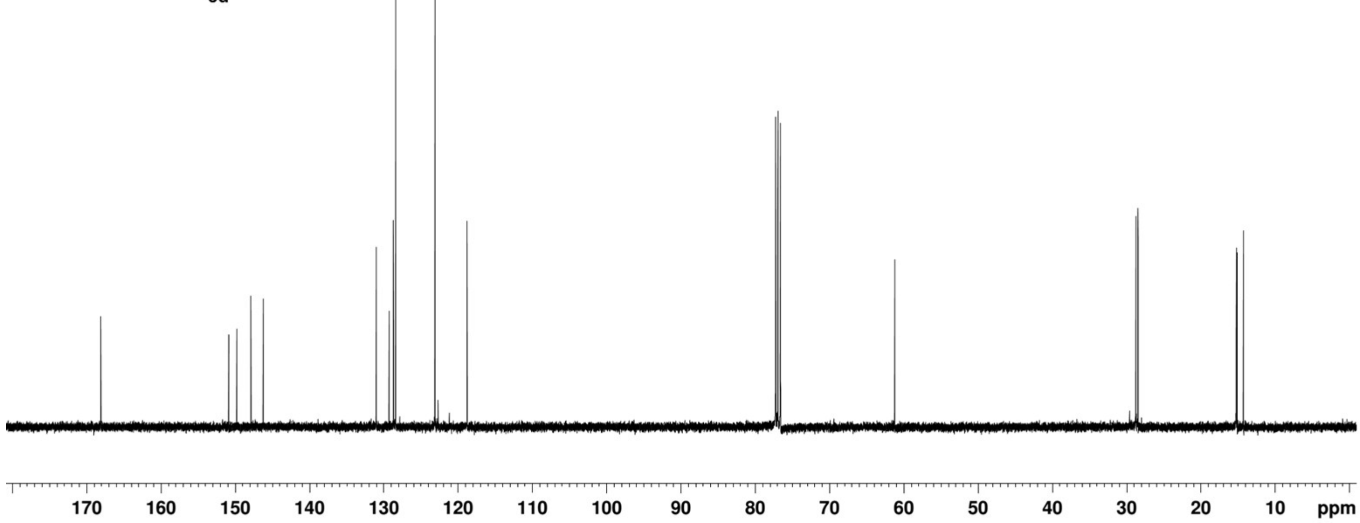
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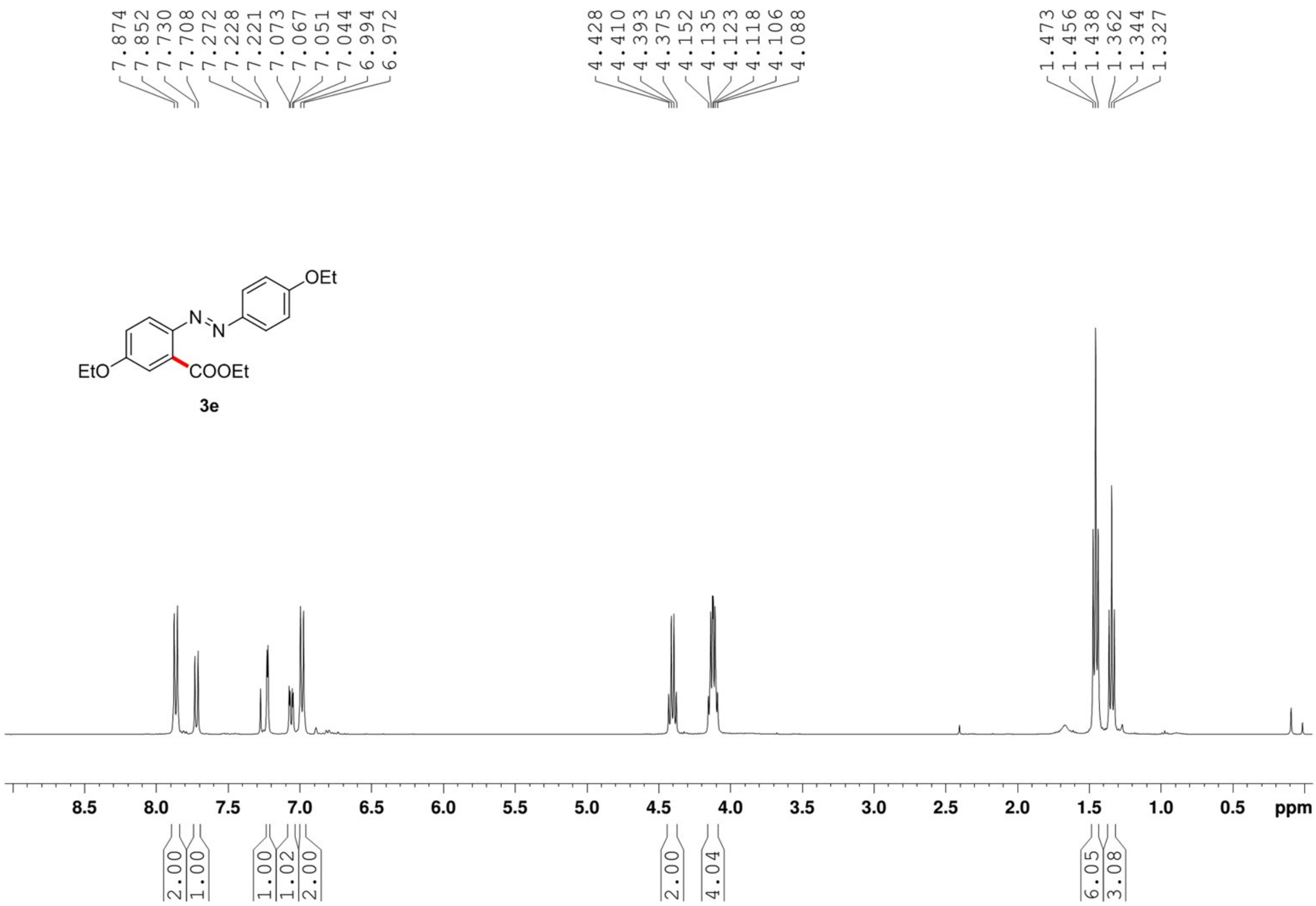
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1.344
1.327
OEt
N
N
EtO
COOEt
3e
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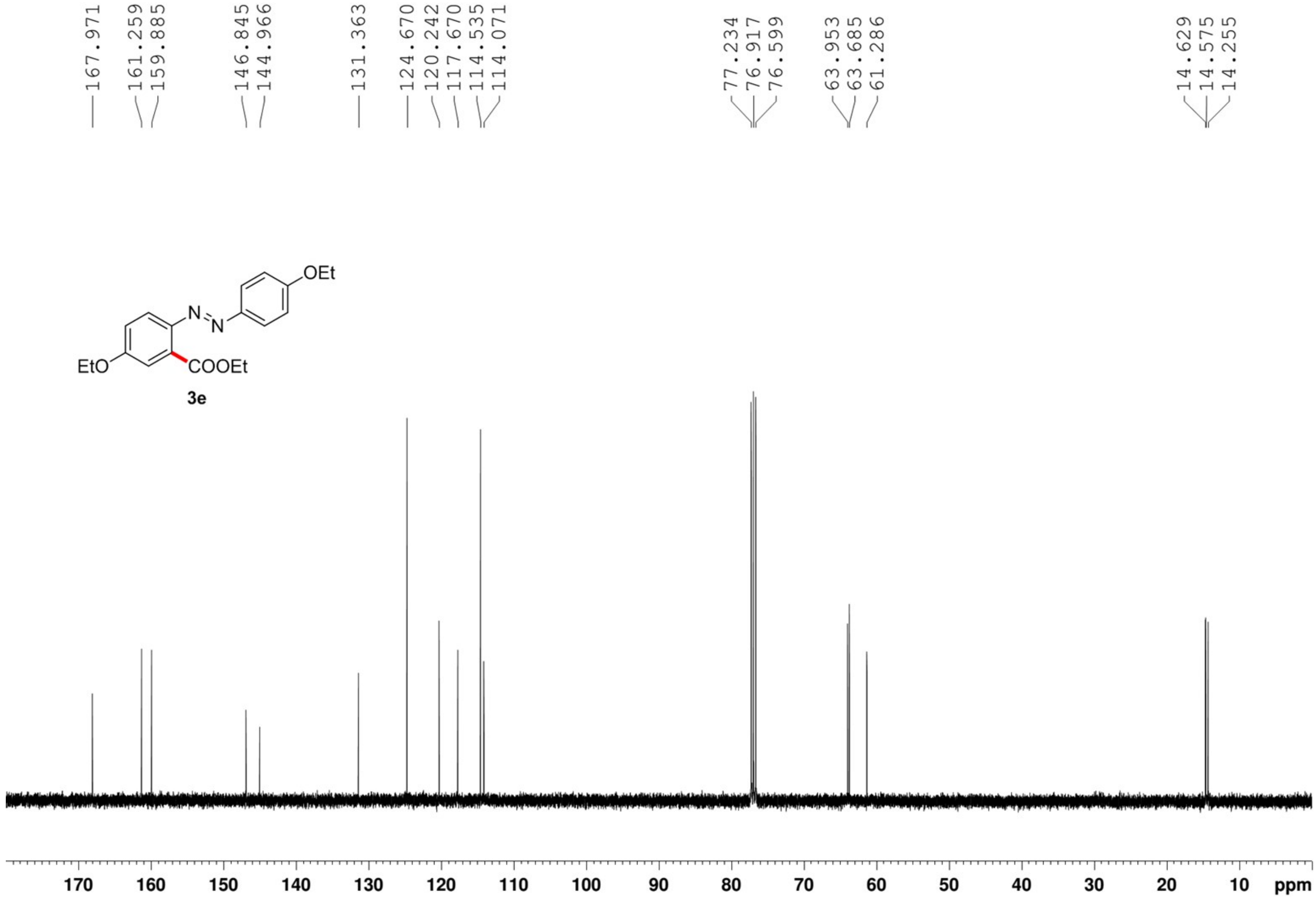
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63.685
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N
EtO
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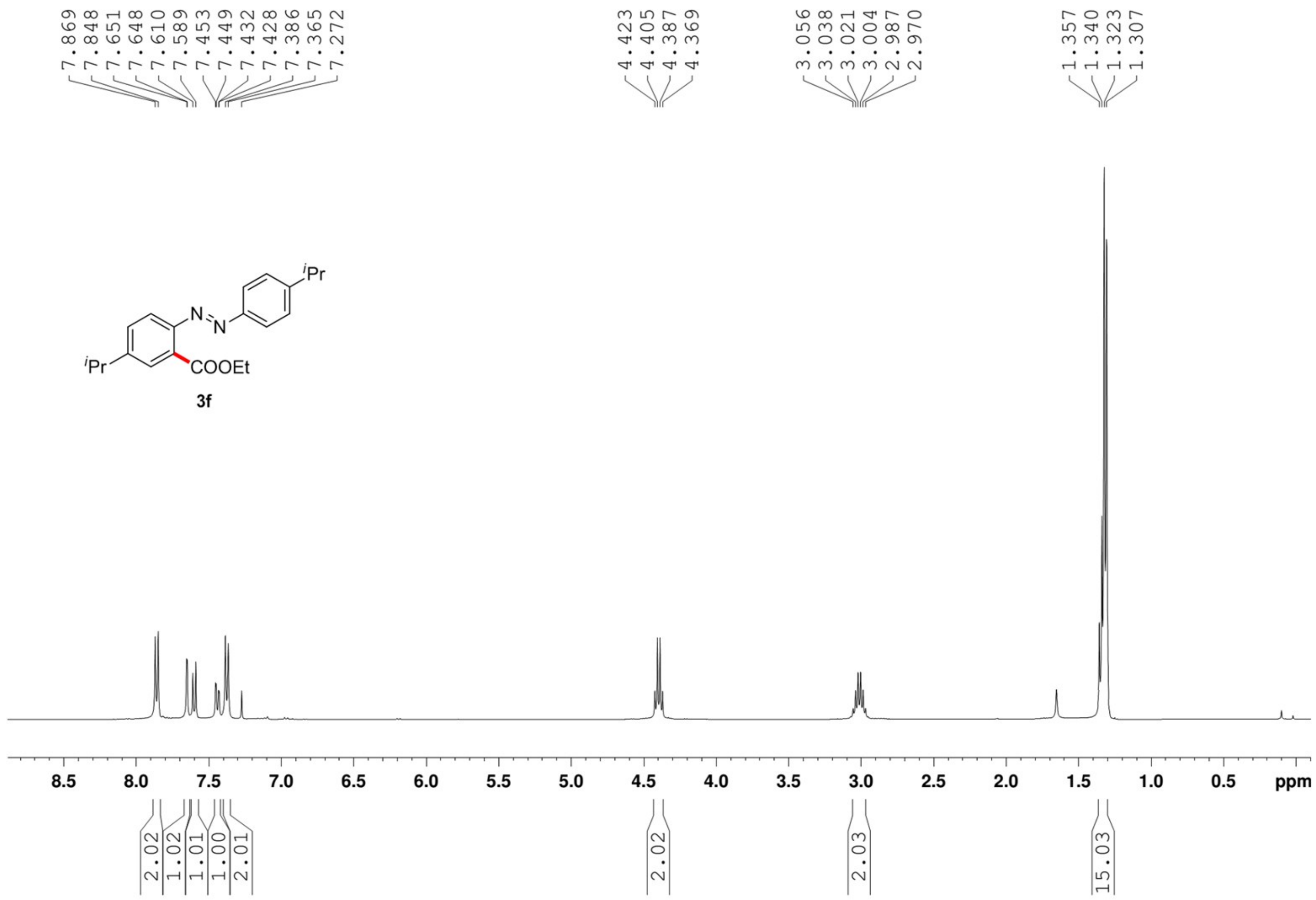
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3.004
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1.340
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4.0
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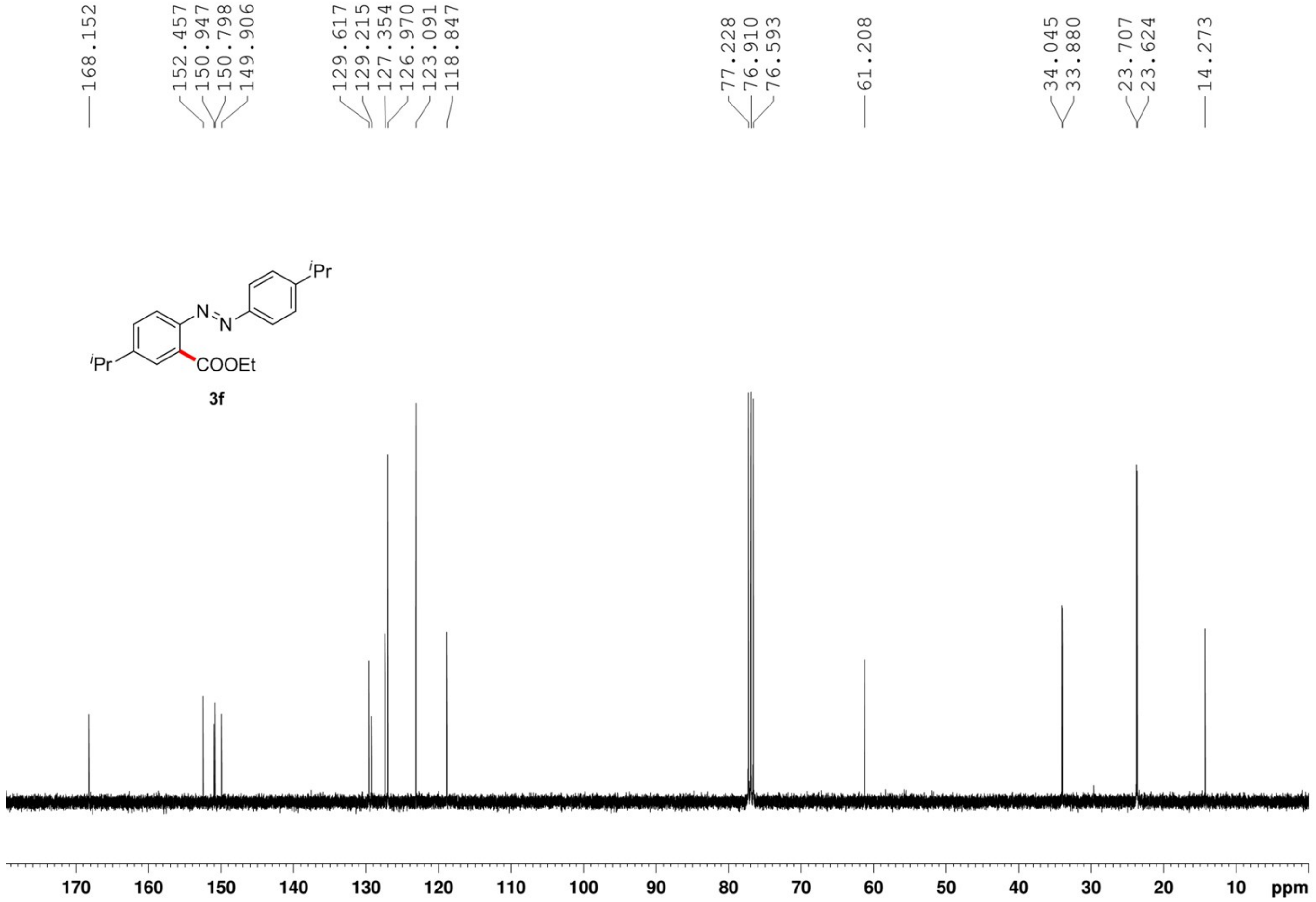
15.03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23.624
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N
iPr
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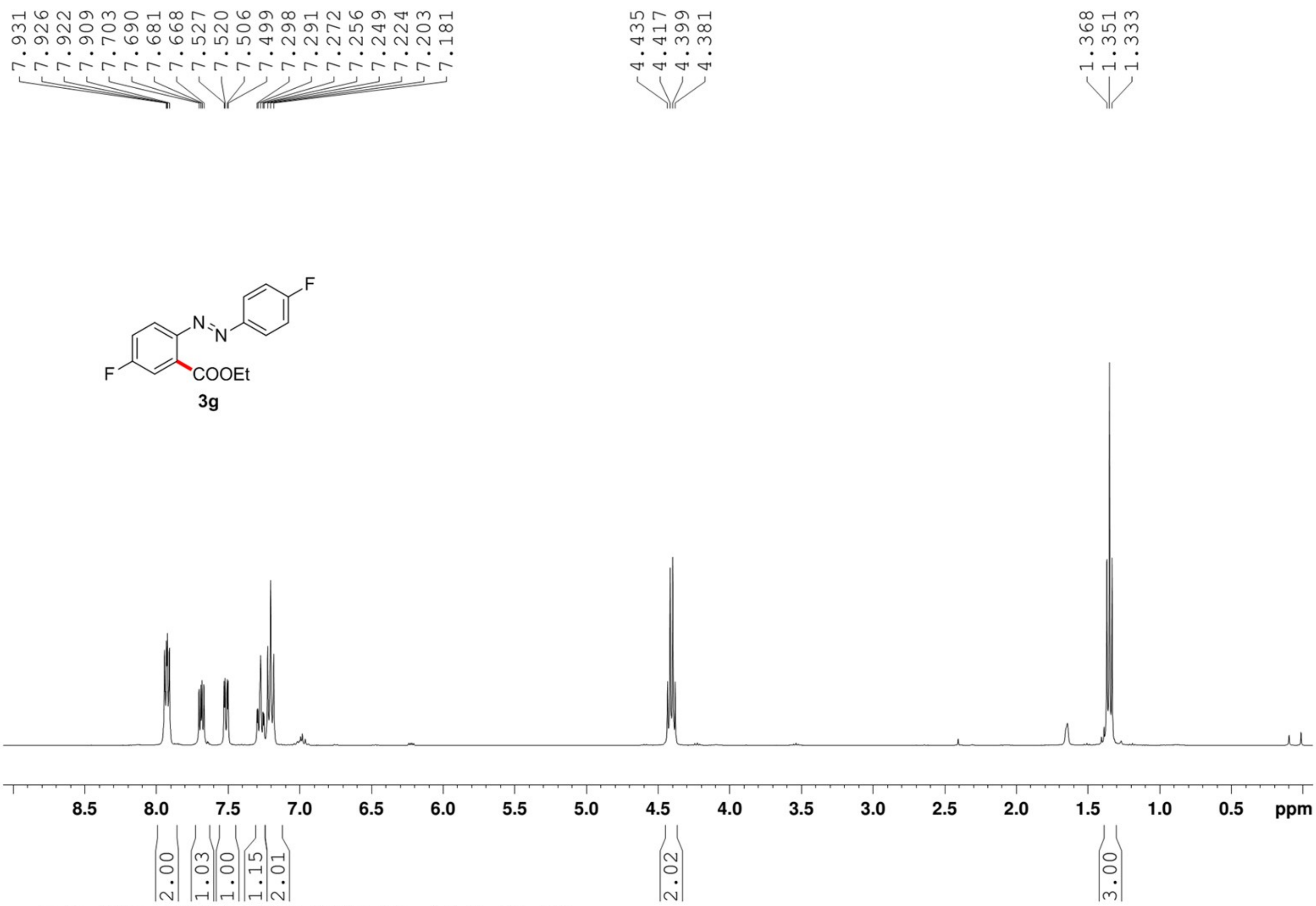

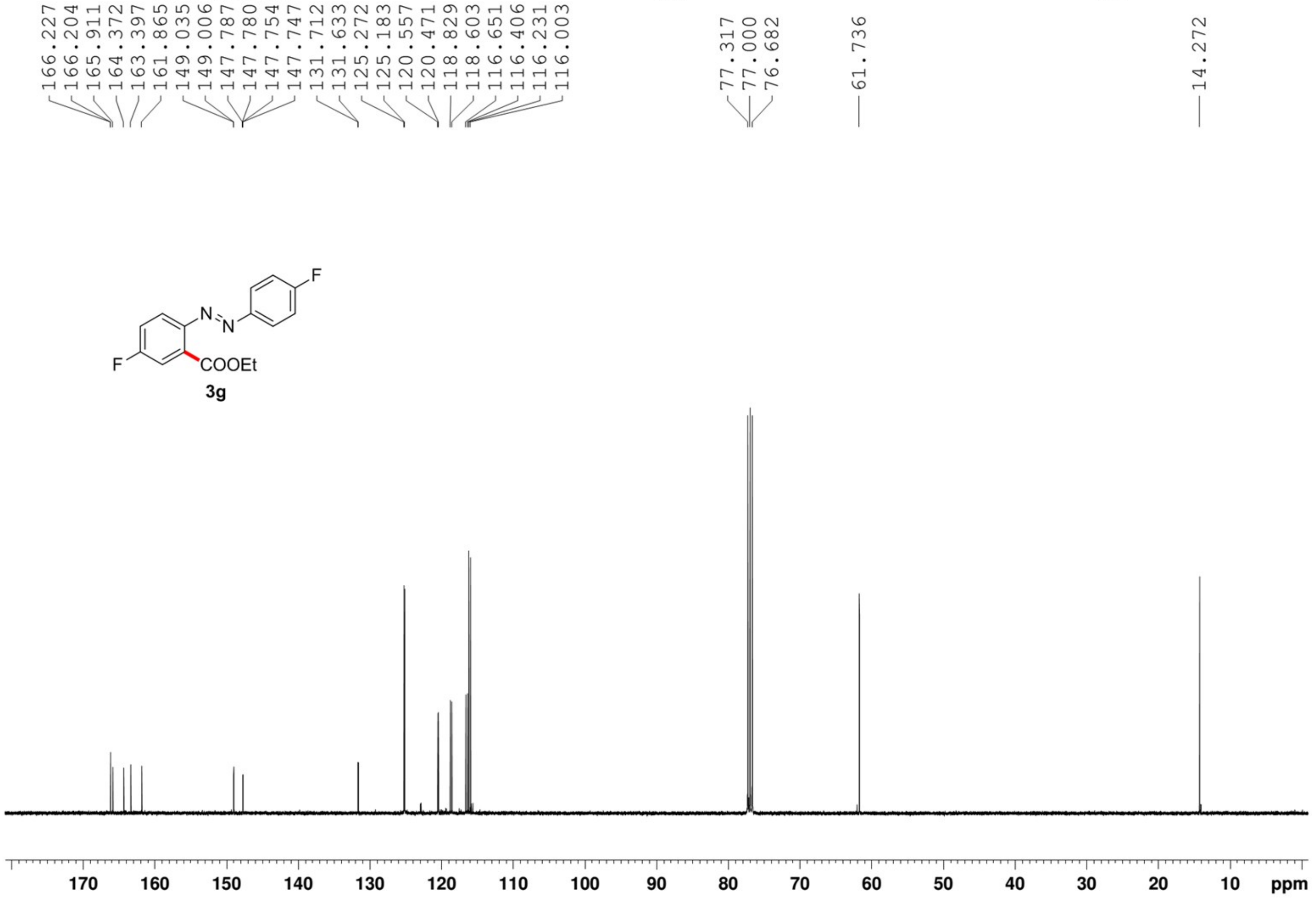

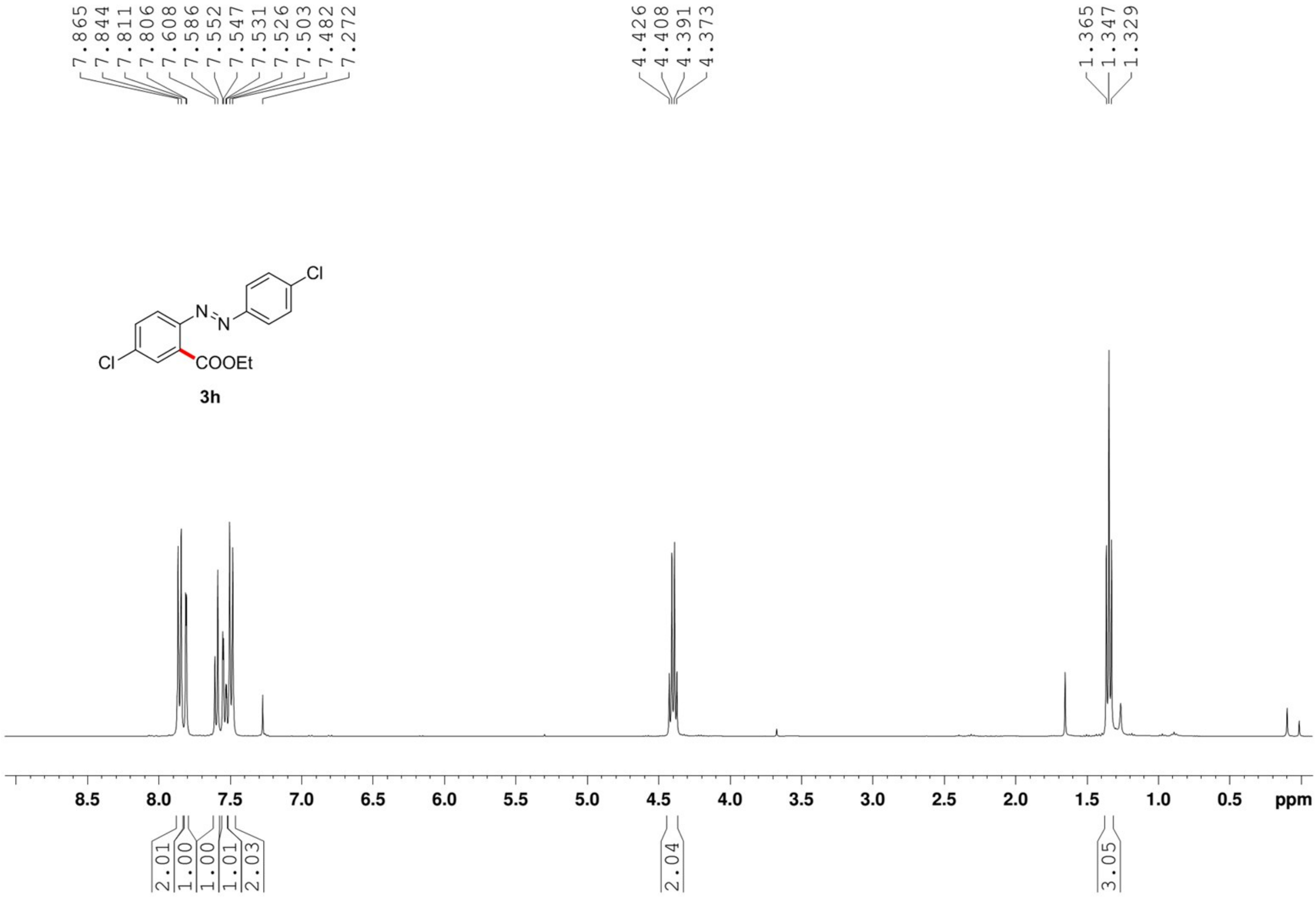
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N
Cl
COOEt
3h
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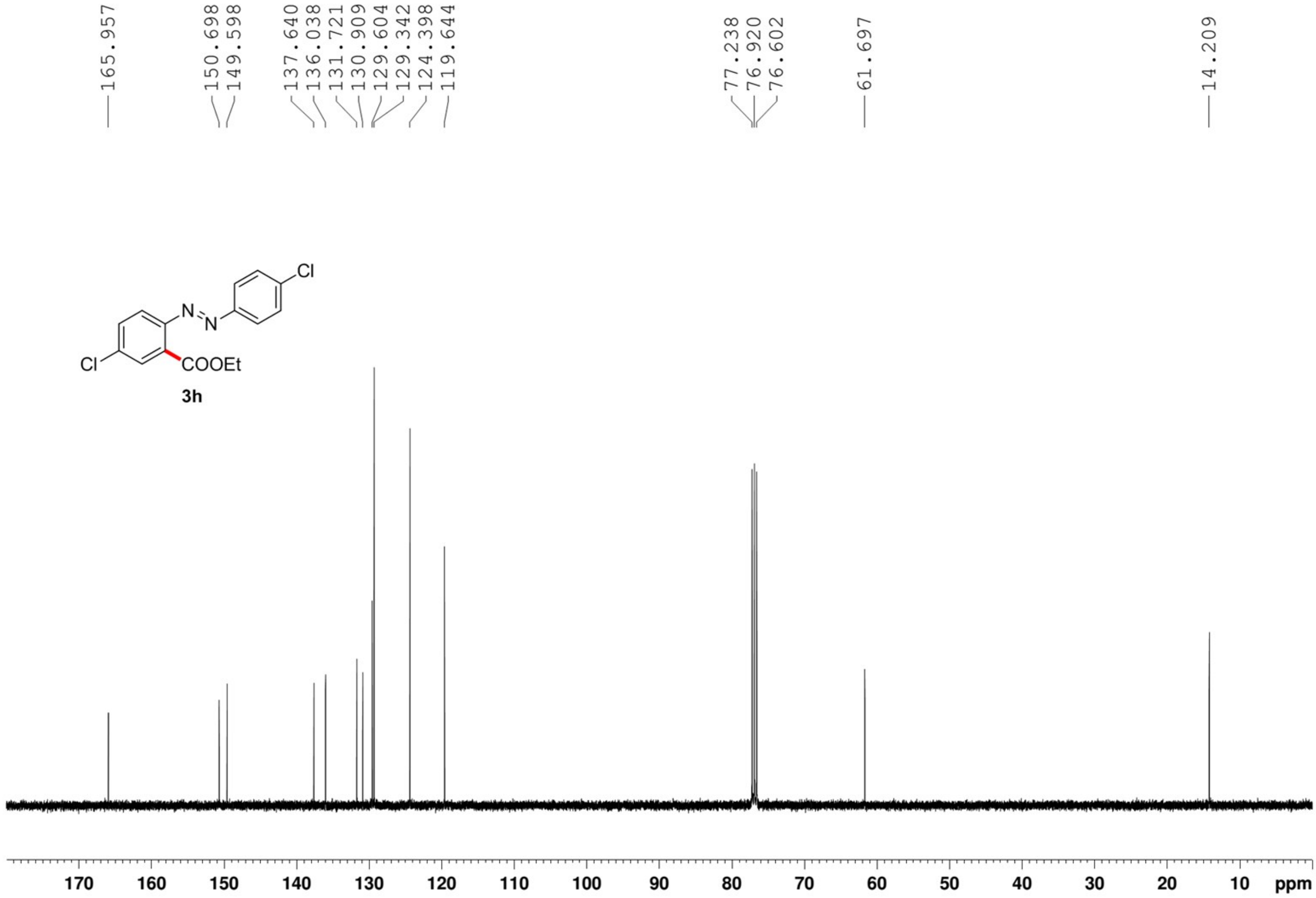
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Cl
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3h
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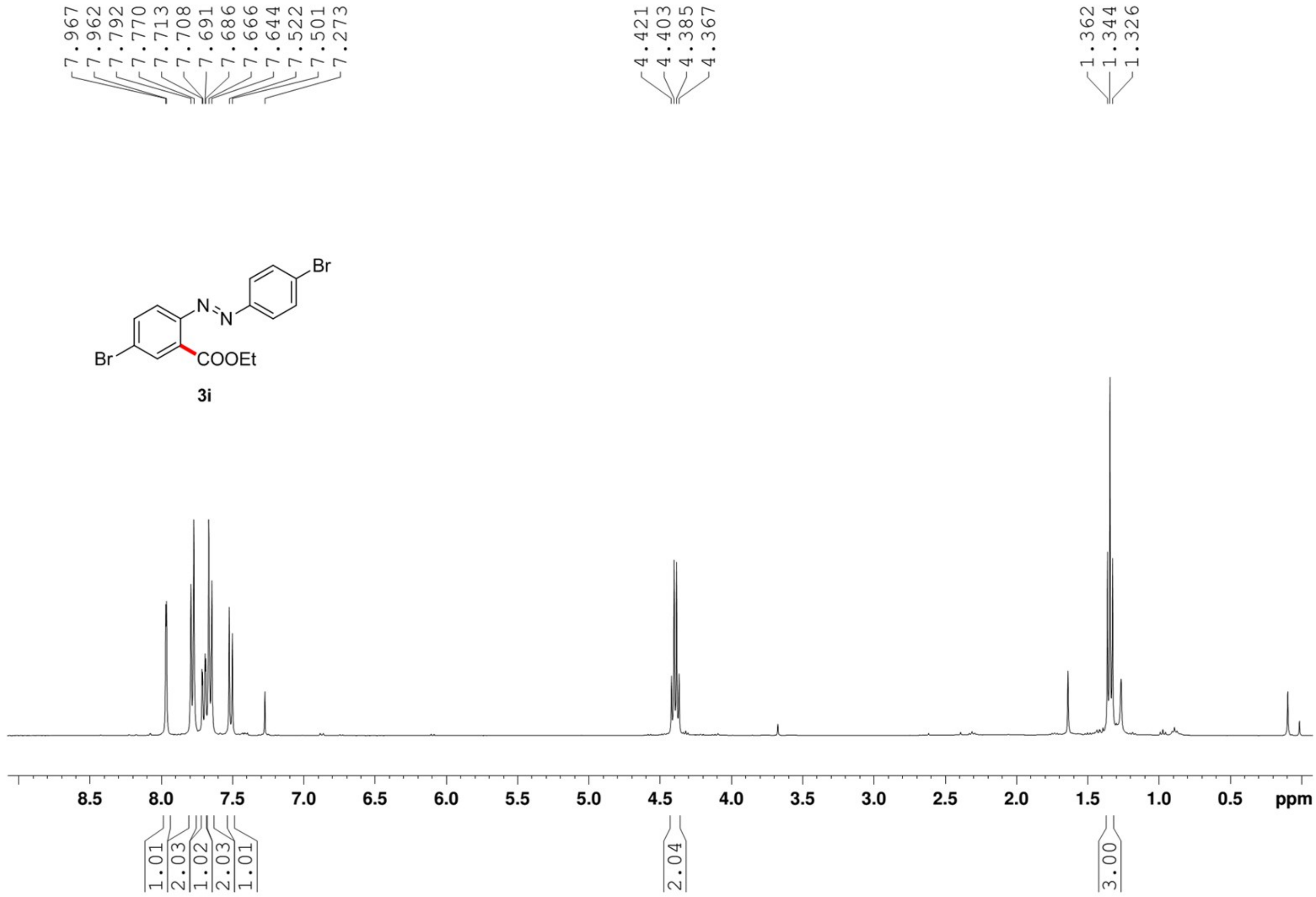

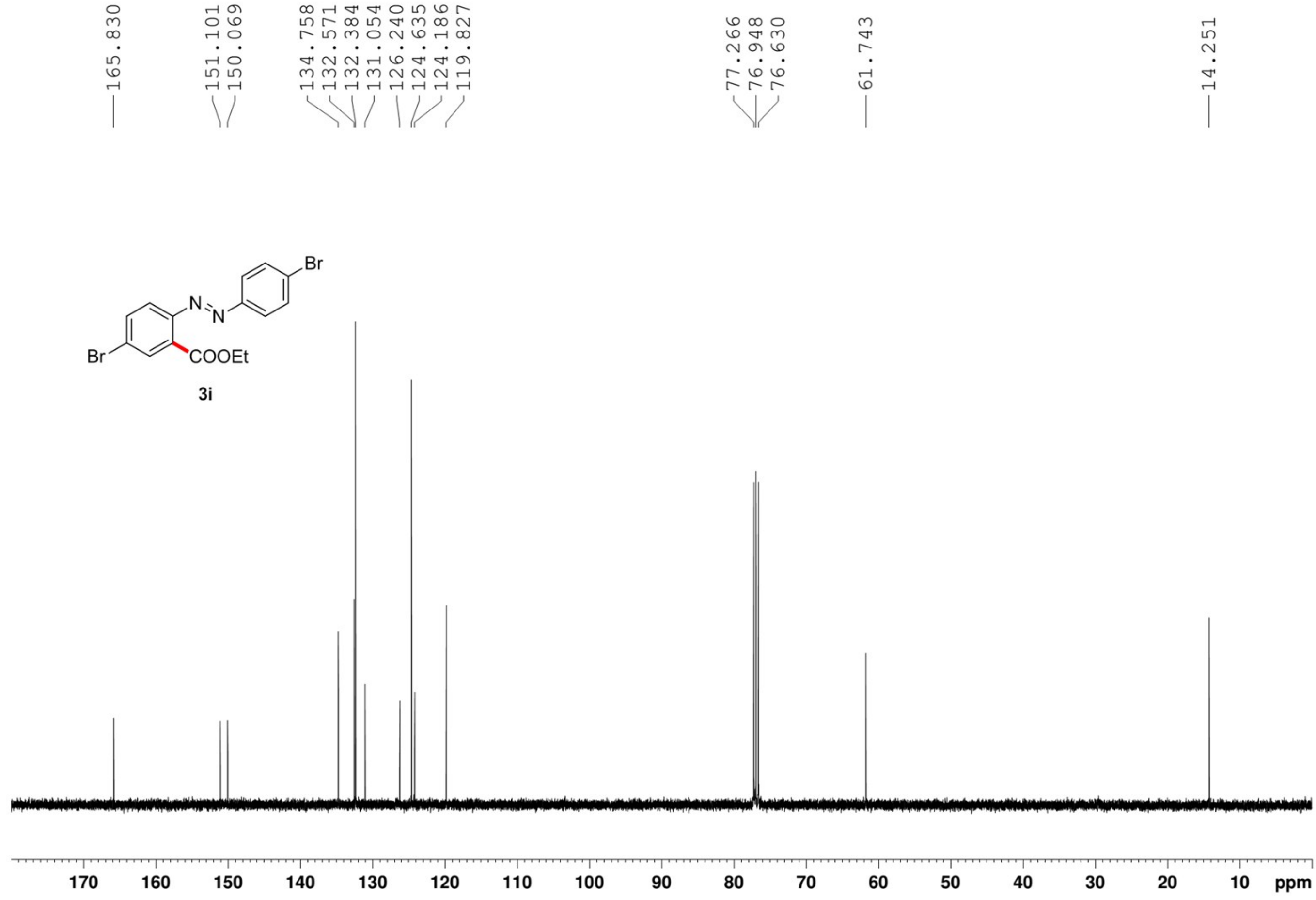

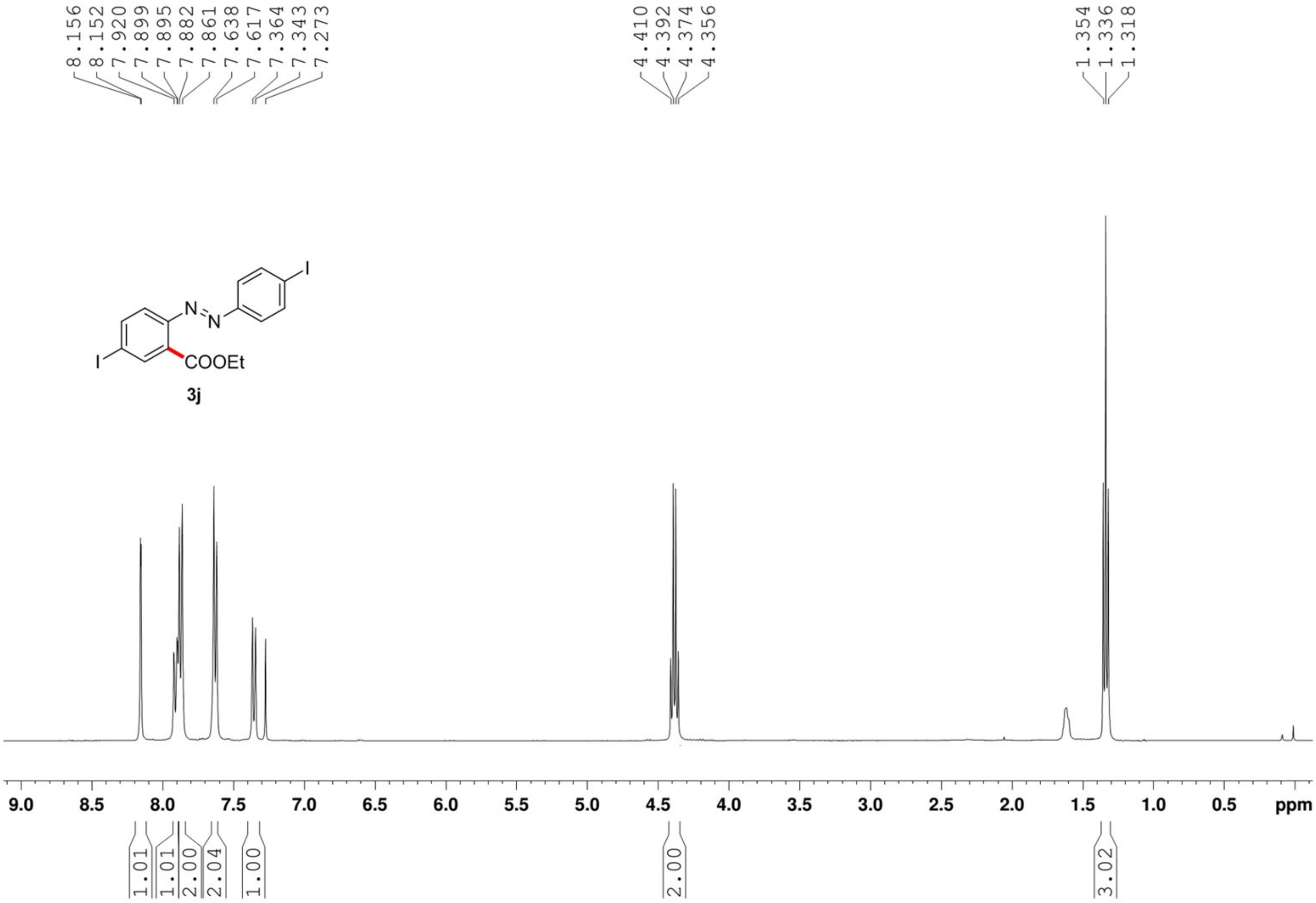
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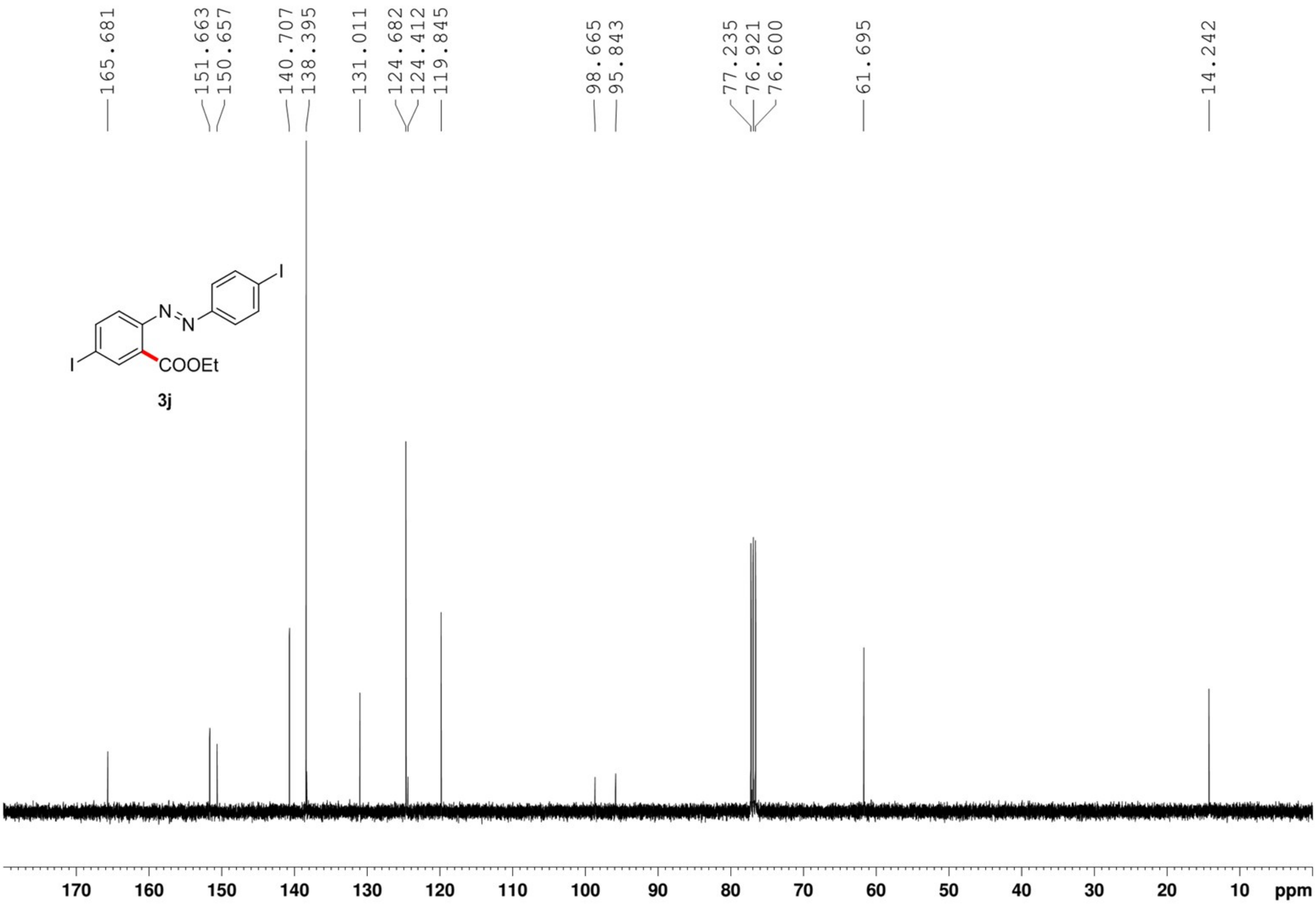
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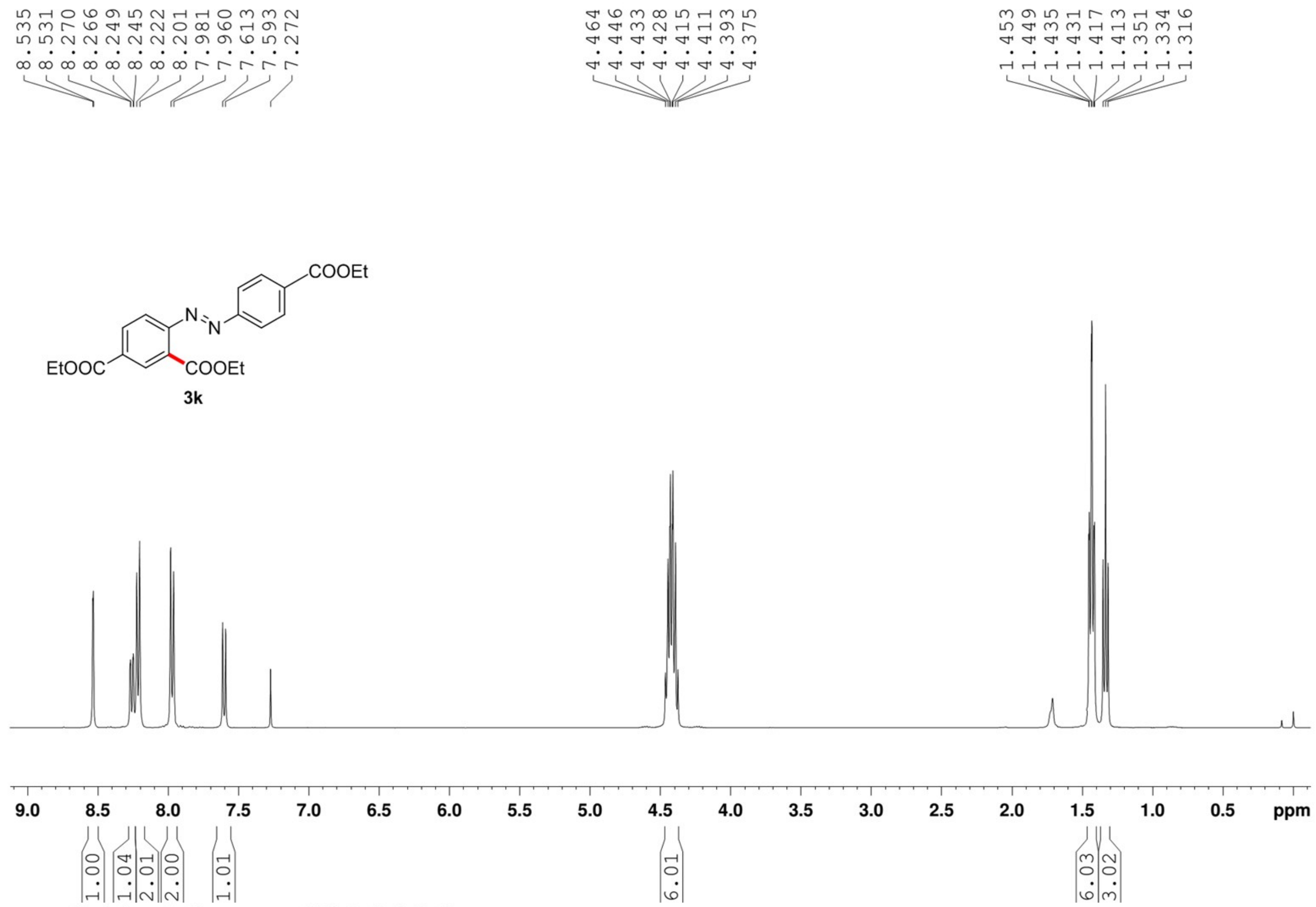
ppm



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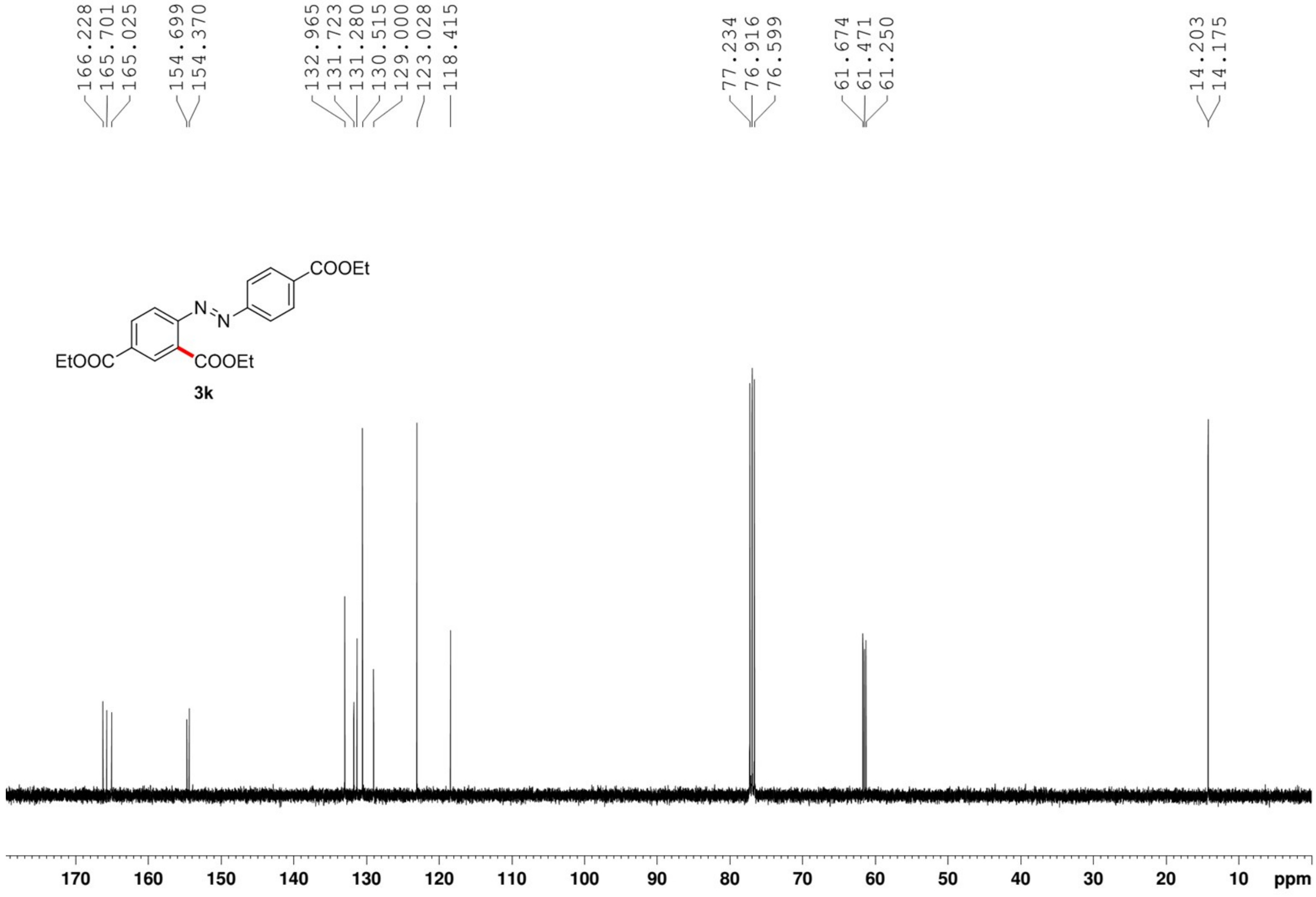

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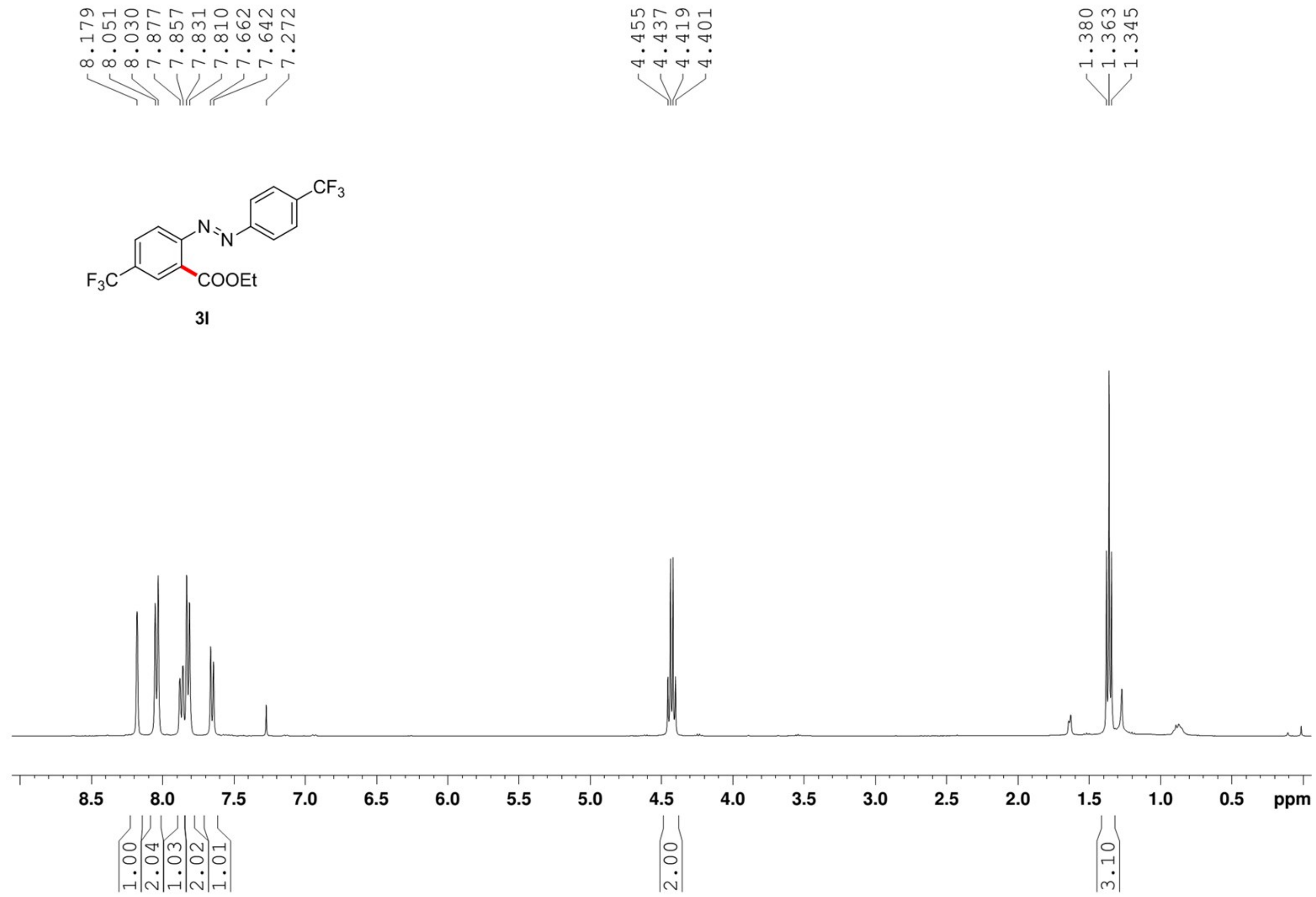
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1.363
1.345
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3l
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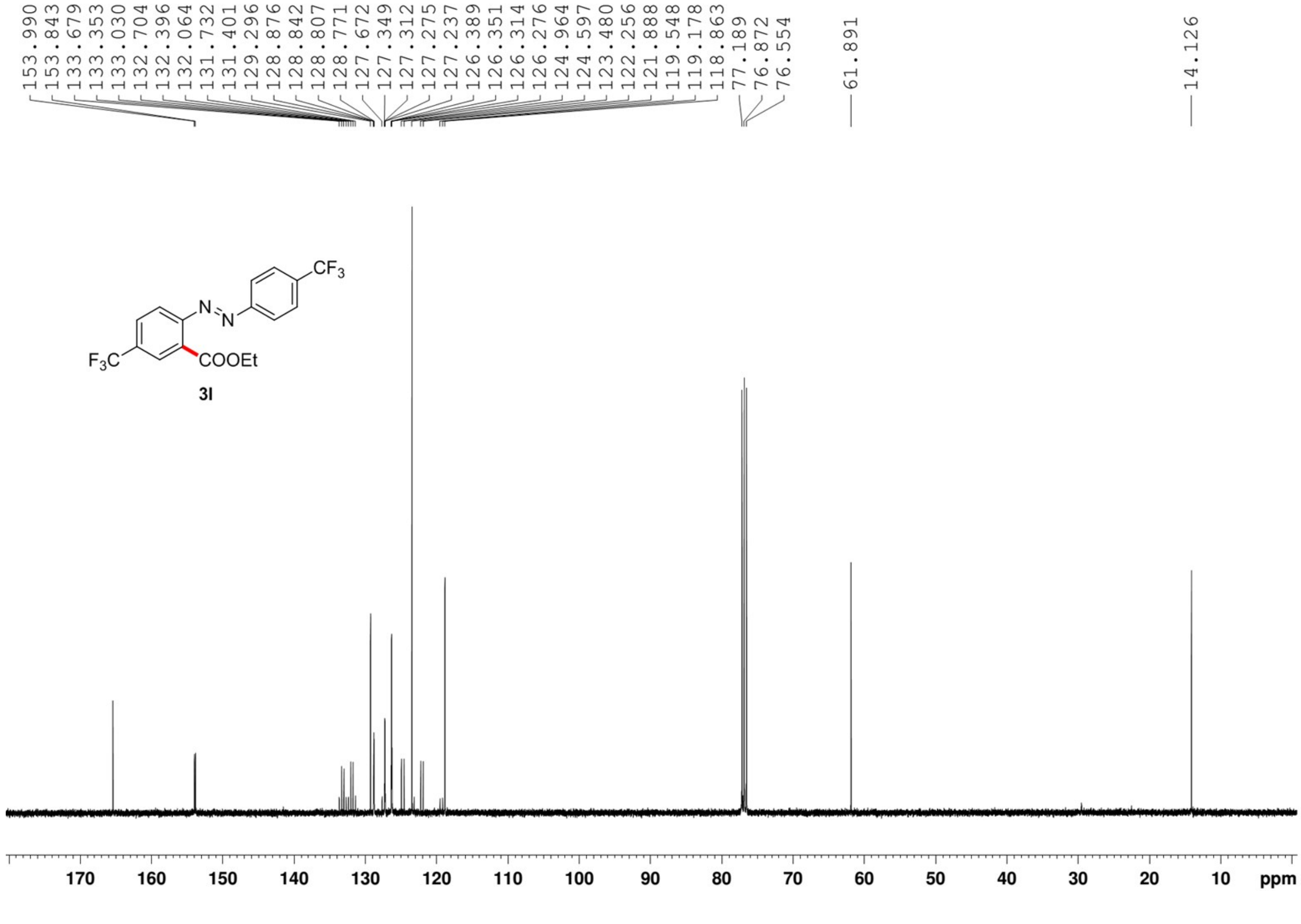
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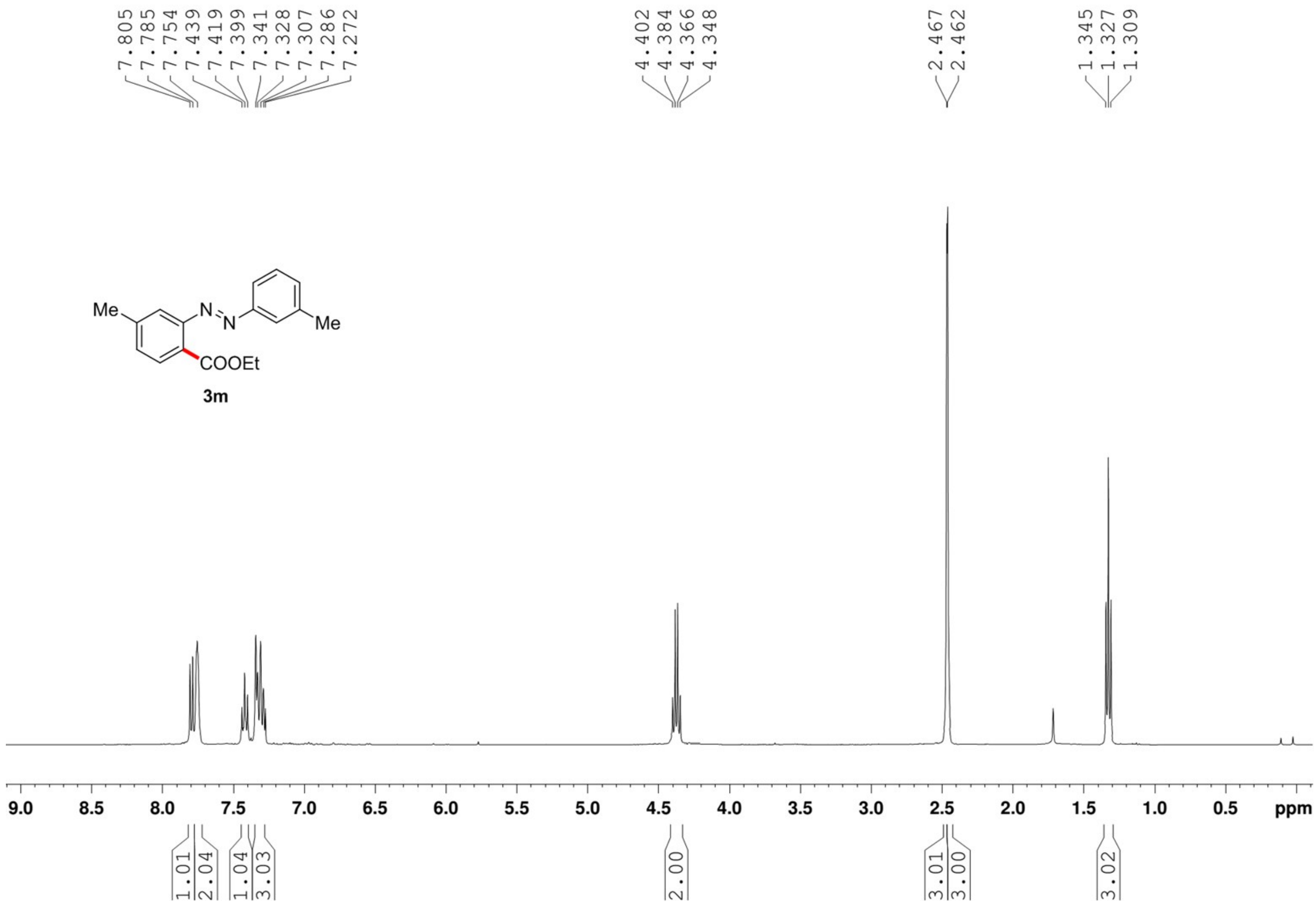
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4.366
4.348
2.467
2.462
1.345
1.327
1.309
Me
N
N
Me
COOEt
3m
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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2.04
1.04
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2.00
3.01
3.00
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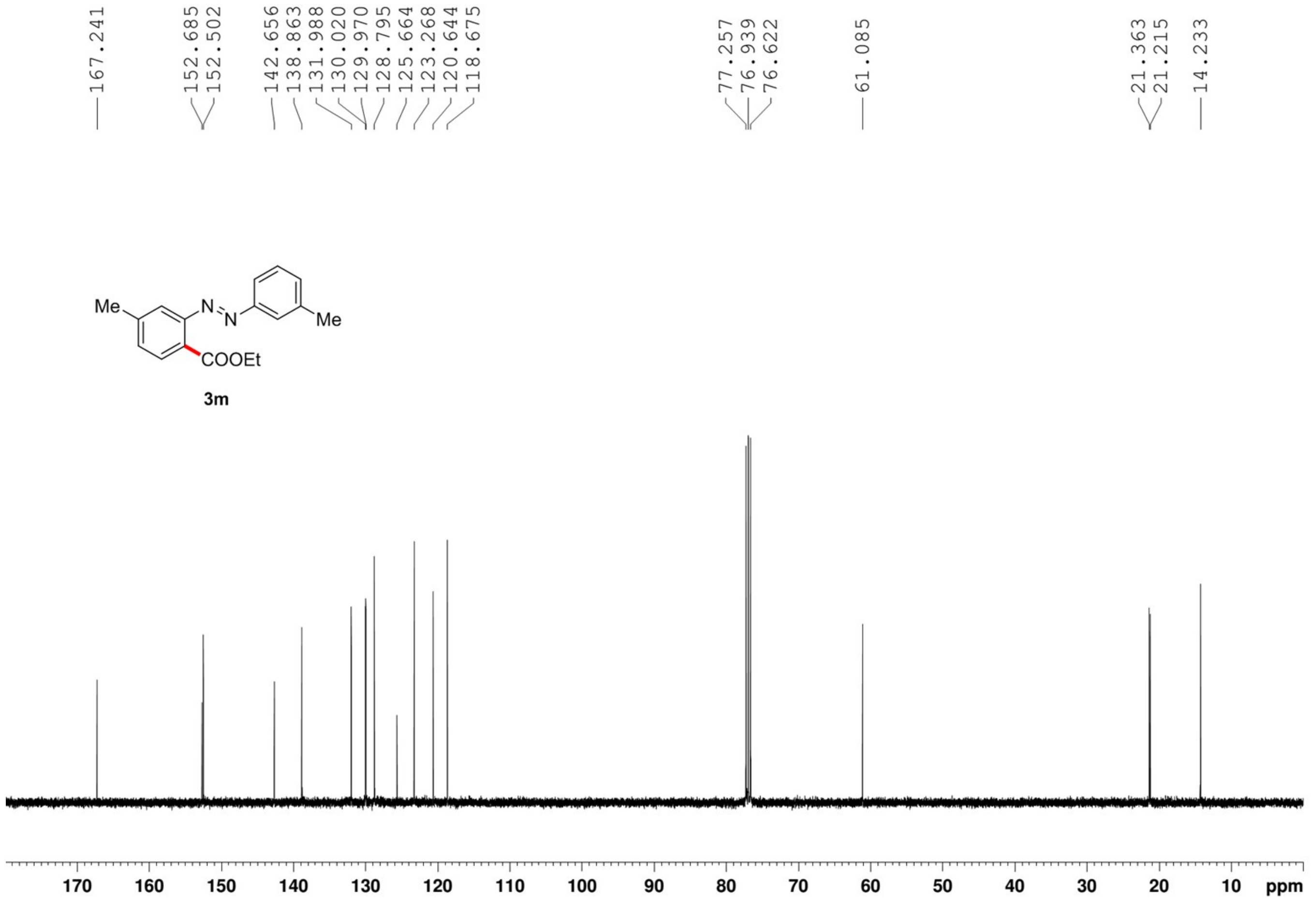
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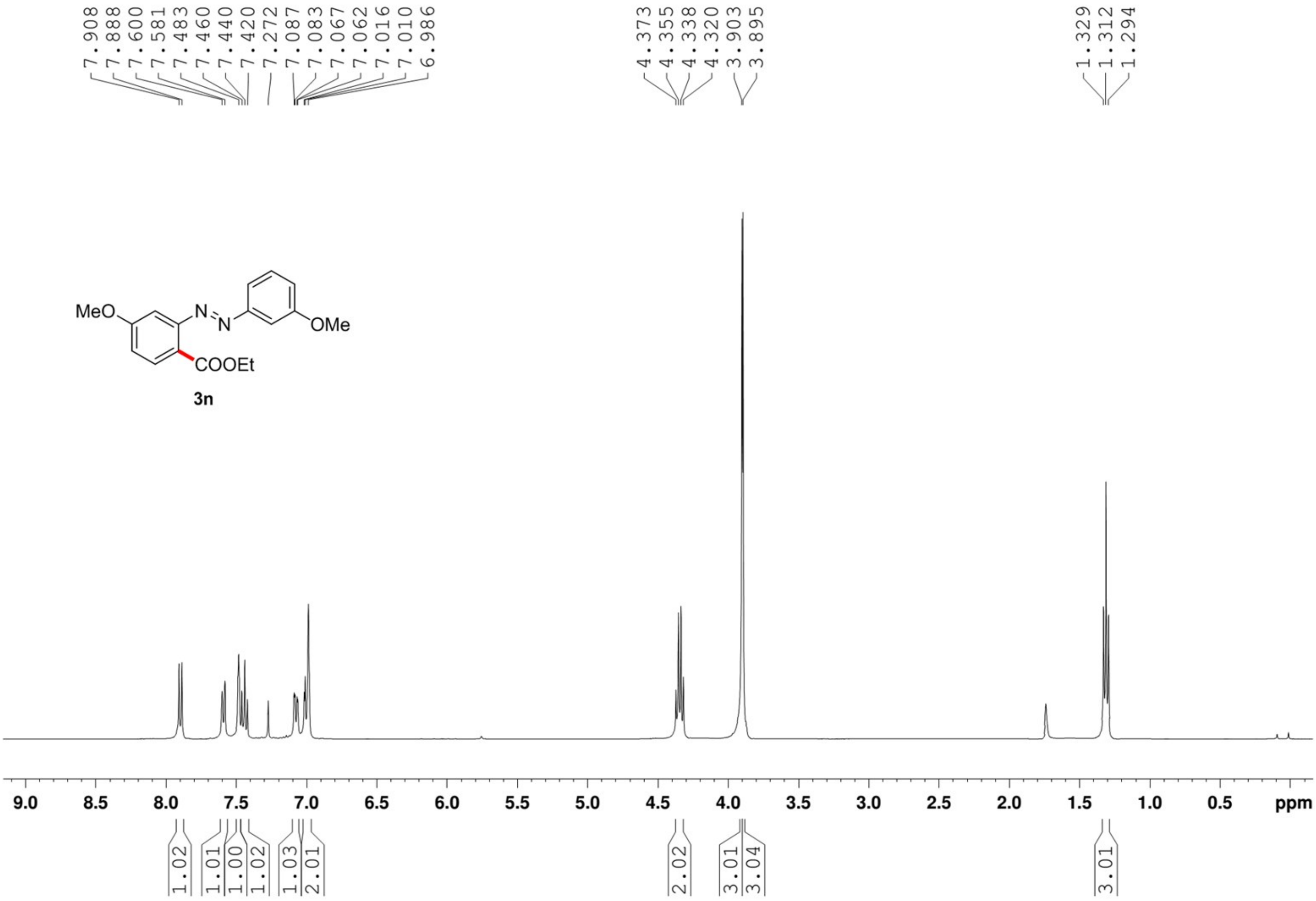
77.257
76.939
76.622
61.085
21.363
21.215
14.233
Me
N
N
Me
COOEt
3m
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.483
7.460
7.440
7.420
7.272
7.087
7.083
7.067
7.062
7.016
7.010
6.986
4.373
4.355
4.338
4.320
3.903
3.895
1.329
1.312
1.294
MeO
N
N
OMe
COOEt
3n
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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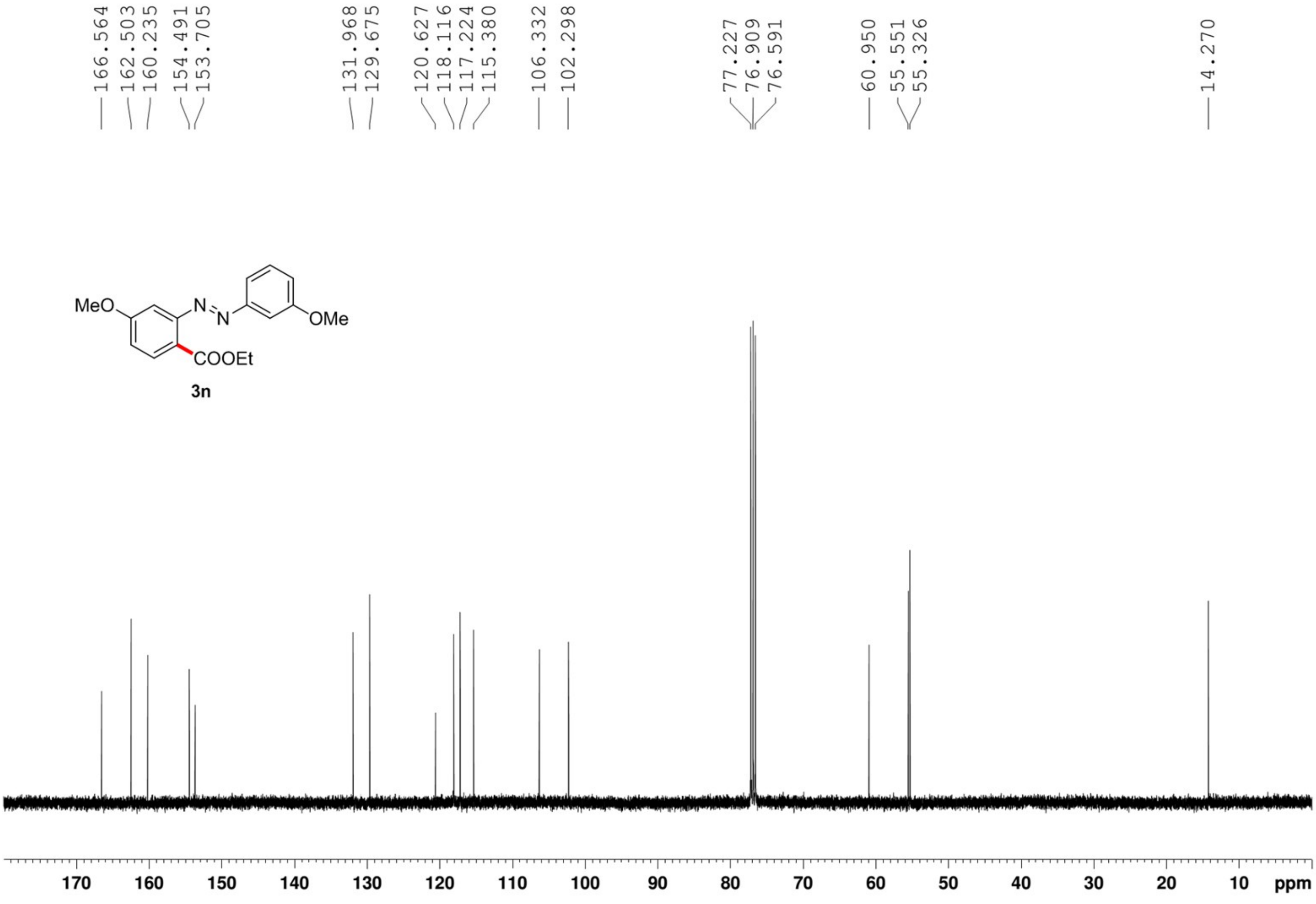
1.01
1.00
1.02
1.03
2.01
2.02
3.01
3.04
3.01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129.675
120.627
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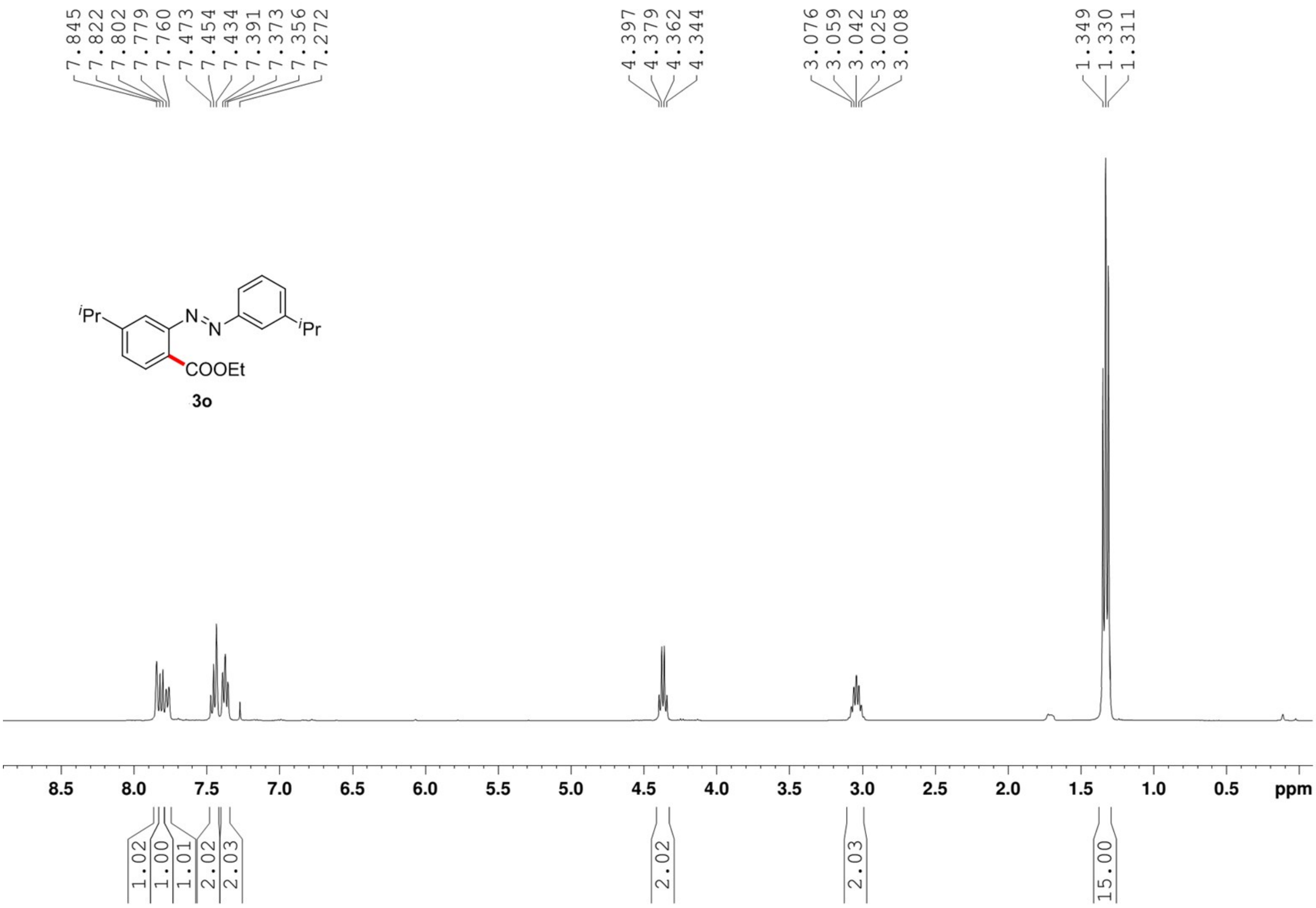
117.224
115.380
106.332
102.298
77.227
76.909
76.591
60.950
55.551
55.326
14.270
MeO
N
N
OMe
COOEt
3n
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.779
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7.434
7.391
7.373
7.356
7.272
4.397
4.379
4.362
4.344
3.076
3.059
3.042
3.025
3.008
1.349
1.330
1.311
iPr
N
N
iPr
COOEt
3o
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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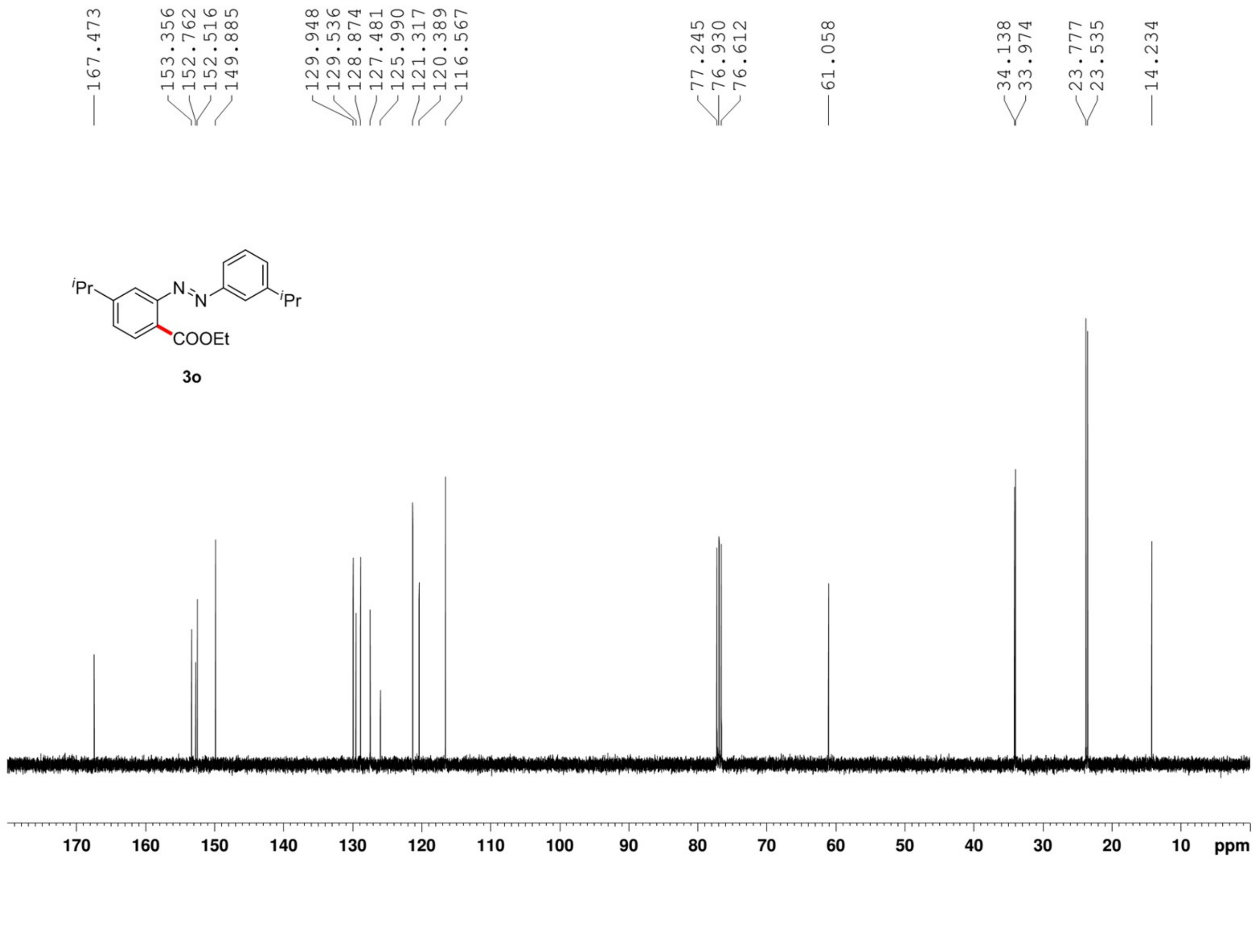
1.00
1.01
2.02
2.03
2.03
2.02
2.03
15.00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125.990
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120.389
116.567
77.245
76.930
76.612
61.058
34.138
33.974
23.777
23.535
14.234
iPr
N
N
iPr
COOEt
3o
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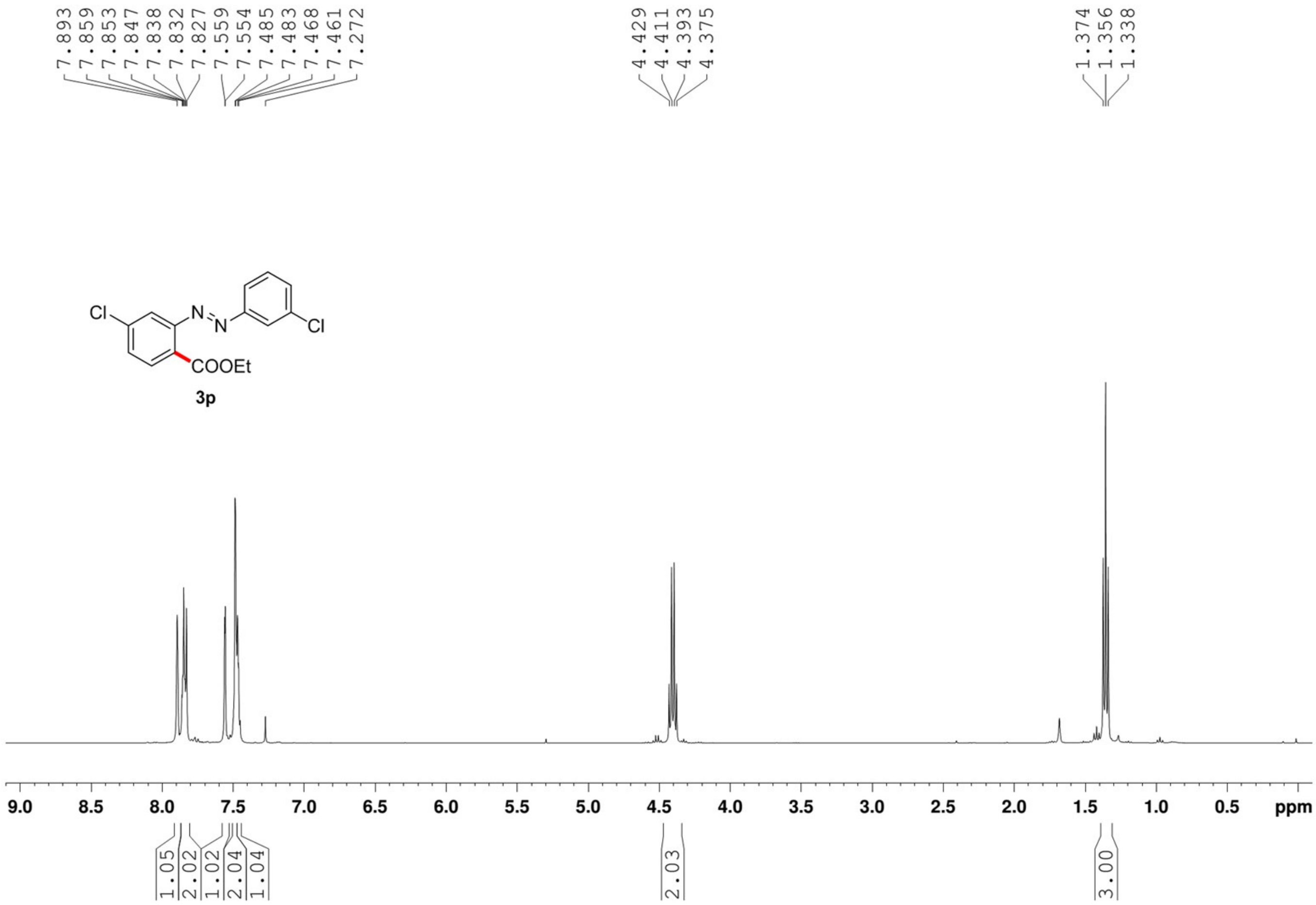
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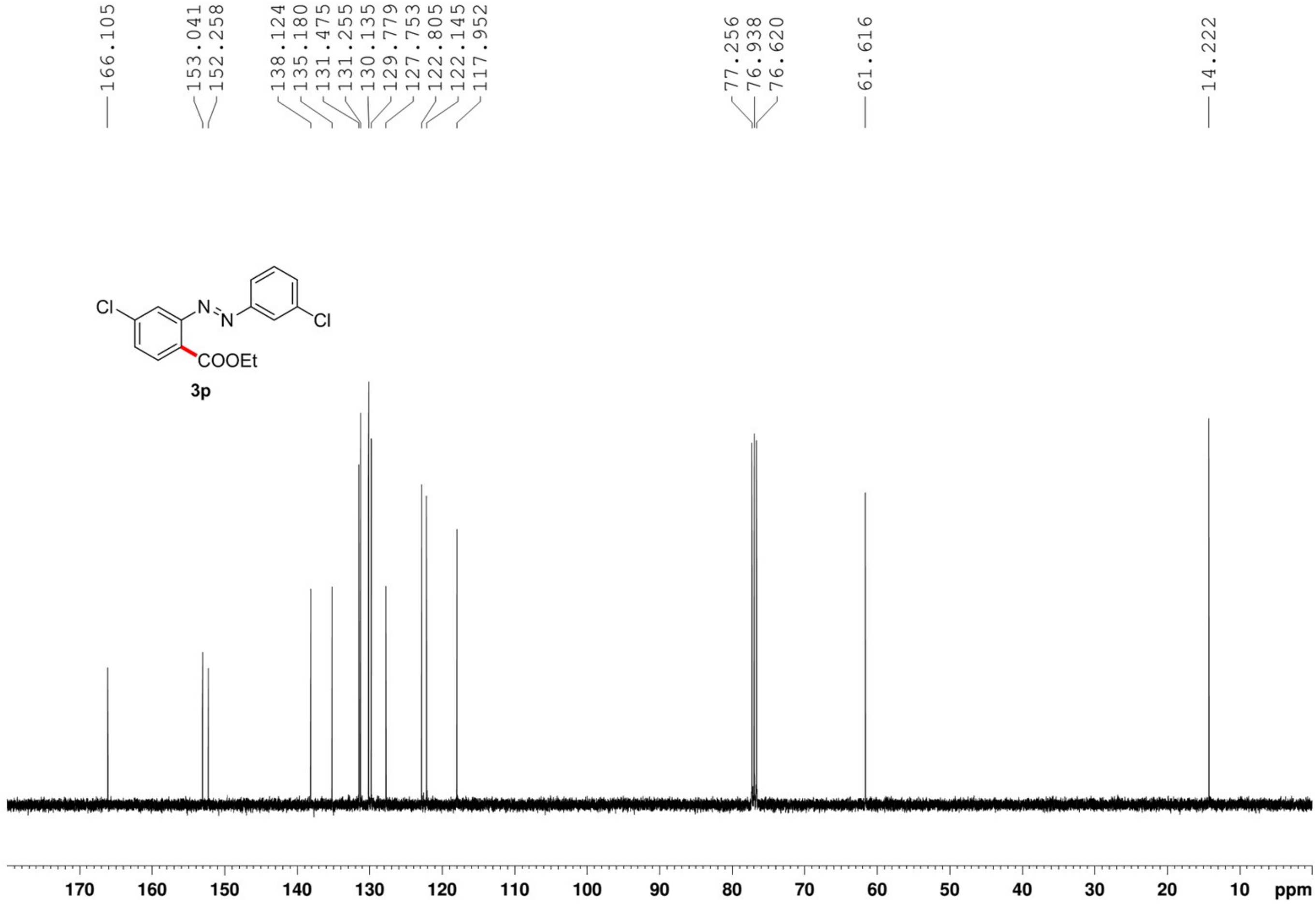
7.853
7.847
7.838
7.832
7.827
7.559
7.554
7.485
7.483
7.468
7.461
7.272
4.429
4.411
4.393
4.375
1.374
1.356
1.338
Cl
N
N
Cl
COOEt
3p
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.05
2.02
1.02
2.04
1.04
2.03
3.00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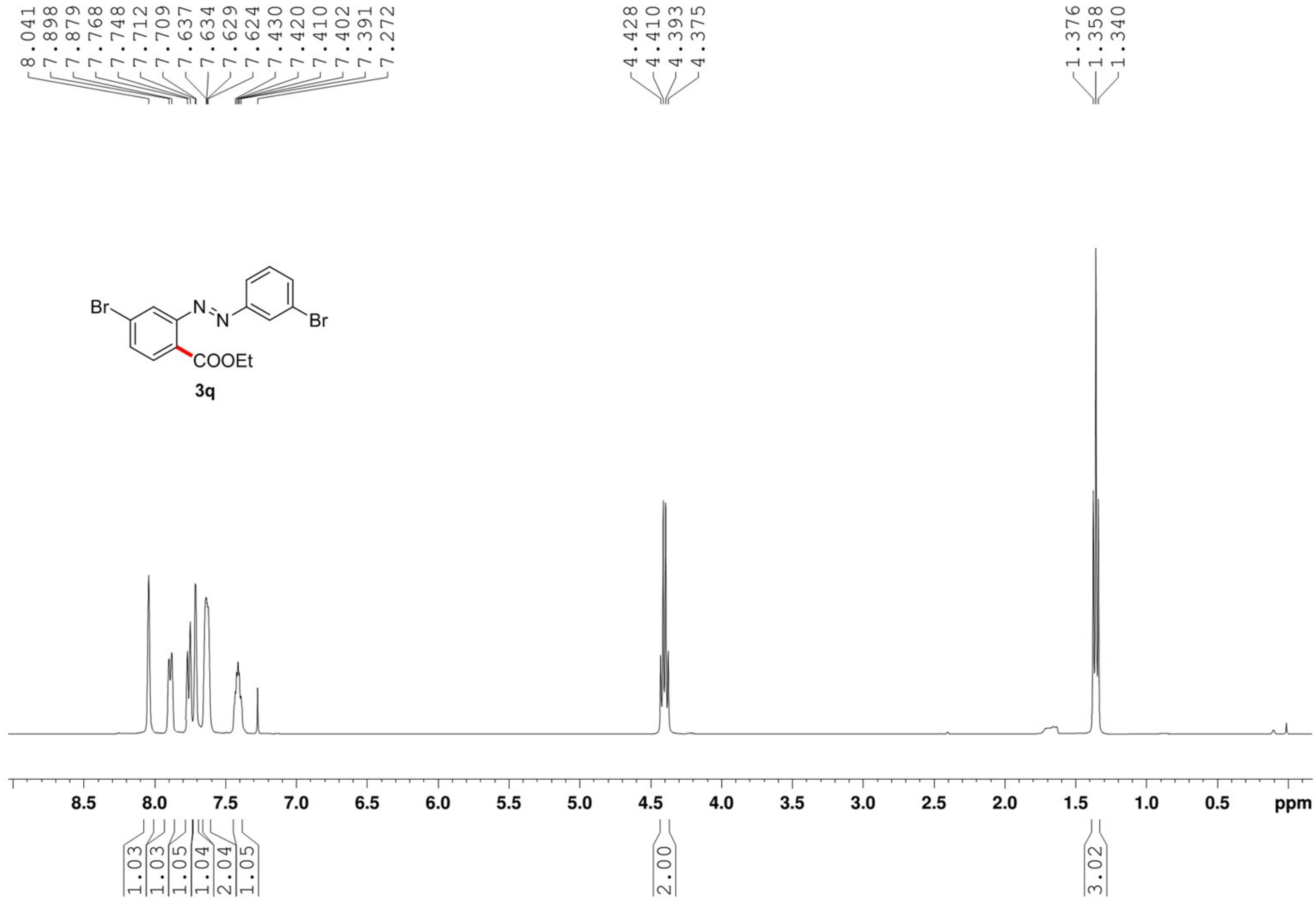
152.258
138.124
135.180
131.475
131.255
130.135
129.779
127.753
122.805
122.145
117.952
77.256
76.938
76.620
61.616
14.222
Cl
N
N
Cl
COOEt
3p
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.879
7.768
7.748
7.712
7.709
7.637
7.634
7.629
7.624
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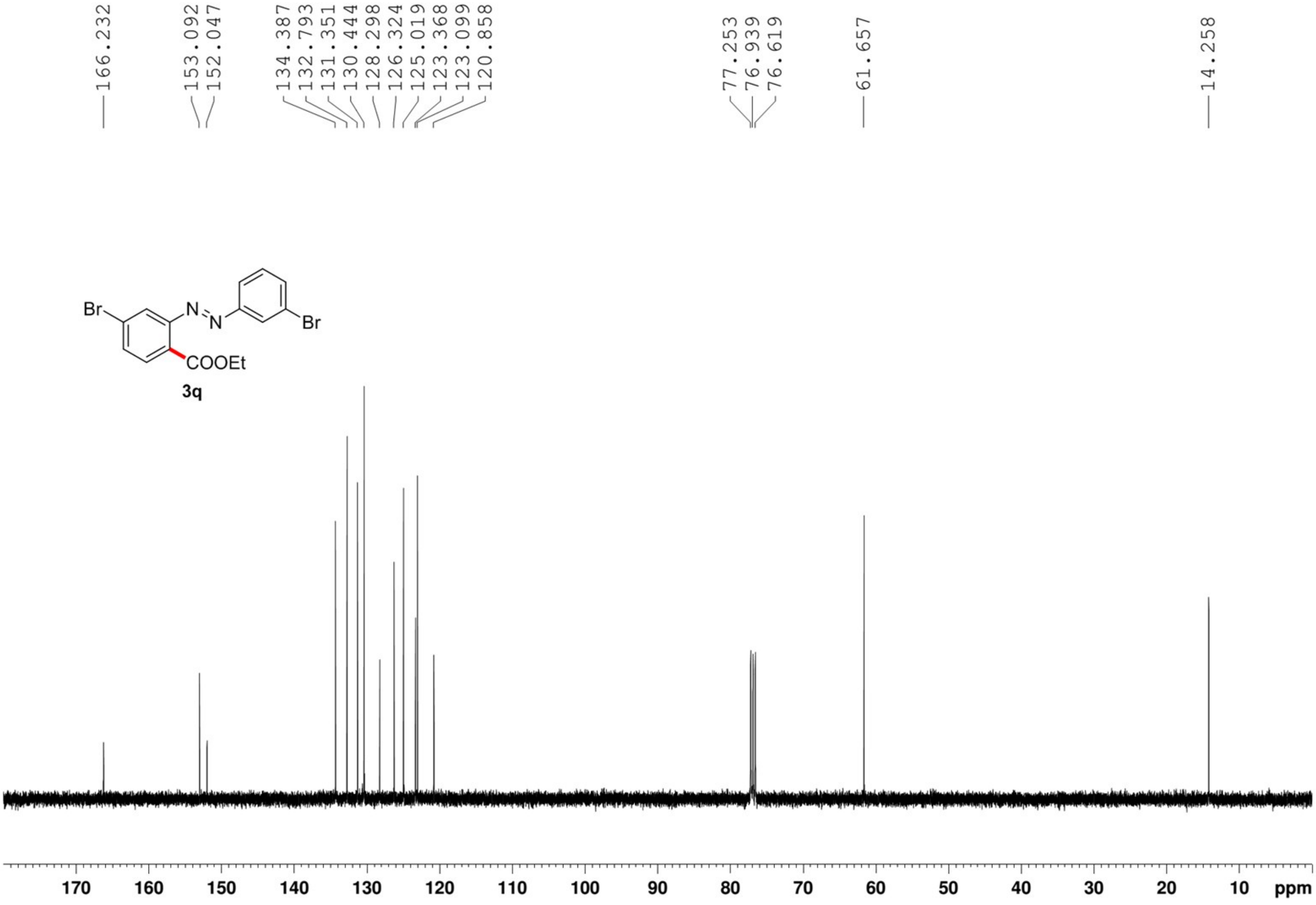
7.420
7.410
7.402
7.391
7.272
4.428
4.410
4.393
4.375
1.376
1.358
1.340
Br
N
N
Br
COOEt
3q
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.03
1.03
1.05
1.04
2.04
1.05
2.00
3.02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130.444
128.298
126.324
125.019
123.368
123.099
120.858
77.253
76.939
76.619
61.657
14.258
Br
N
N
Br
COOEt
3q
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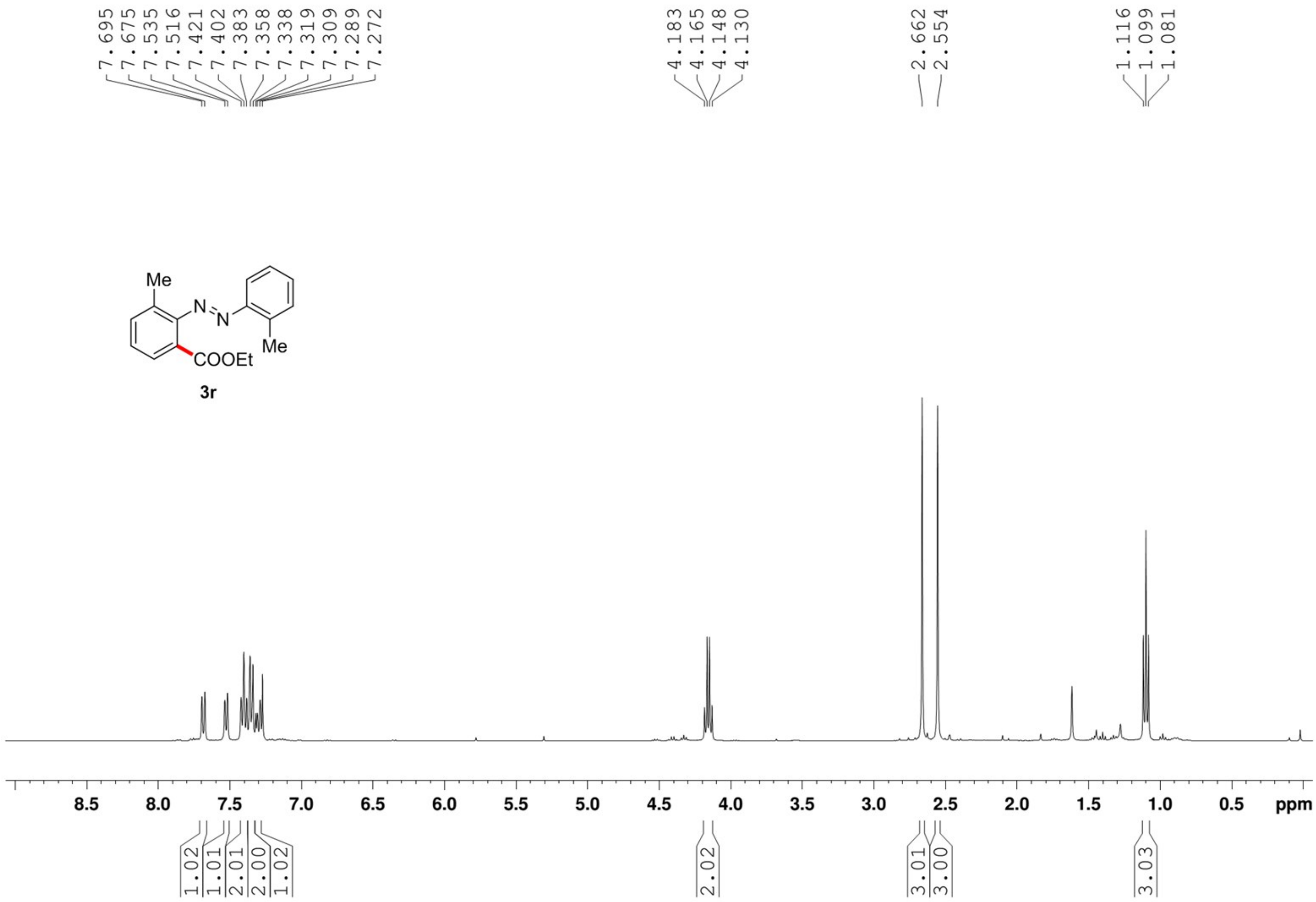
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.535
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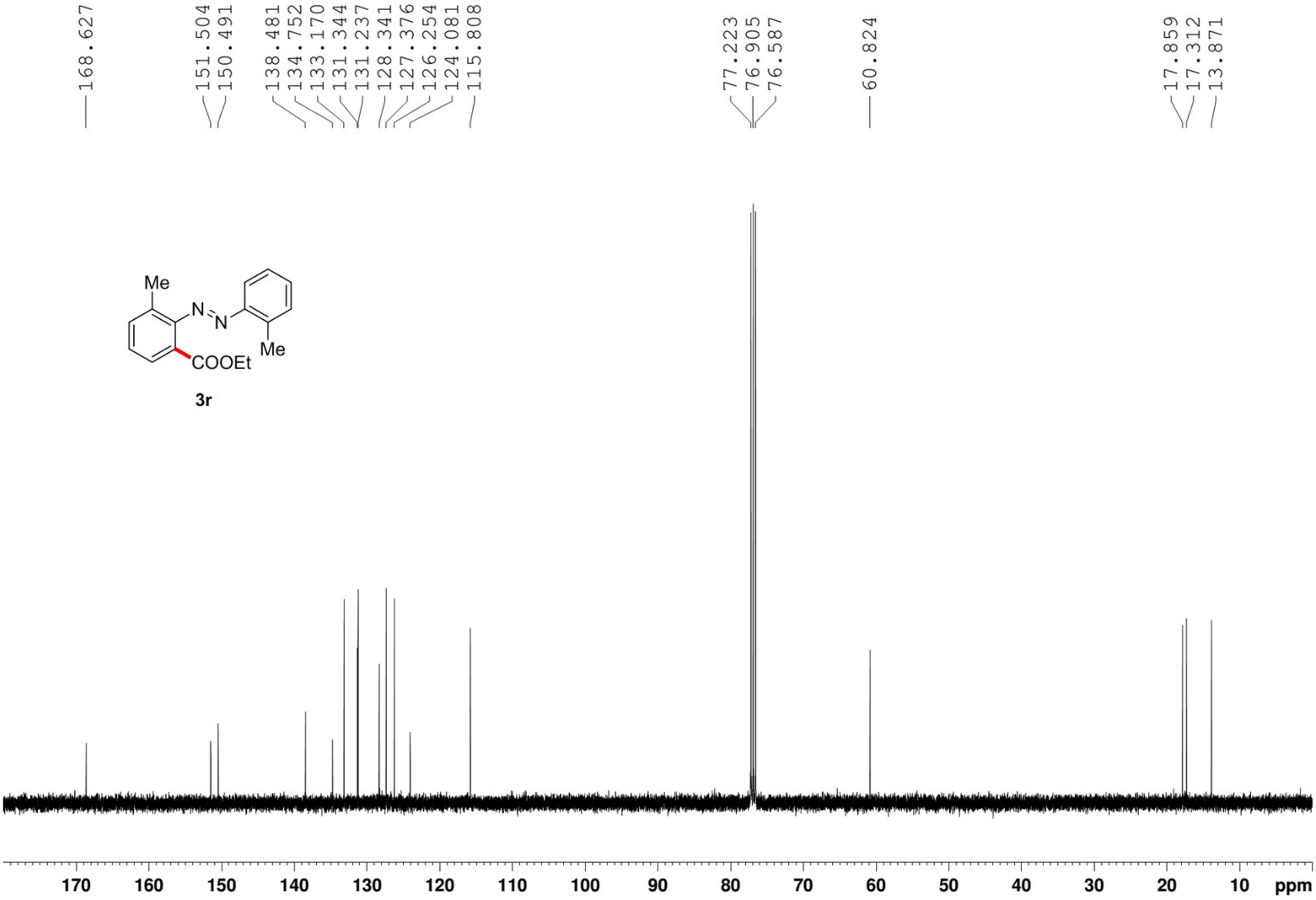
7.421
7.402
7.383
7.358
7.338
7.319
7.309
7.289
7.272
4.183
4.165
4.148
4.130
2.662
2.554
1.116
1.099
1.081
Me
N
N
Me
COOEt
3r
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.02
1.01
2.01
2.00
1.02
2.02
3.01
3.00
3.03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150.491
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134.752
133.170
131.344
131.237
128.341
127.376
126.254
124.081
115.808
77.223
76.905
76.587
60.824
17.859
17.312
13.871
Me
N
N
Me
COOEt
3r
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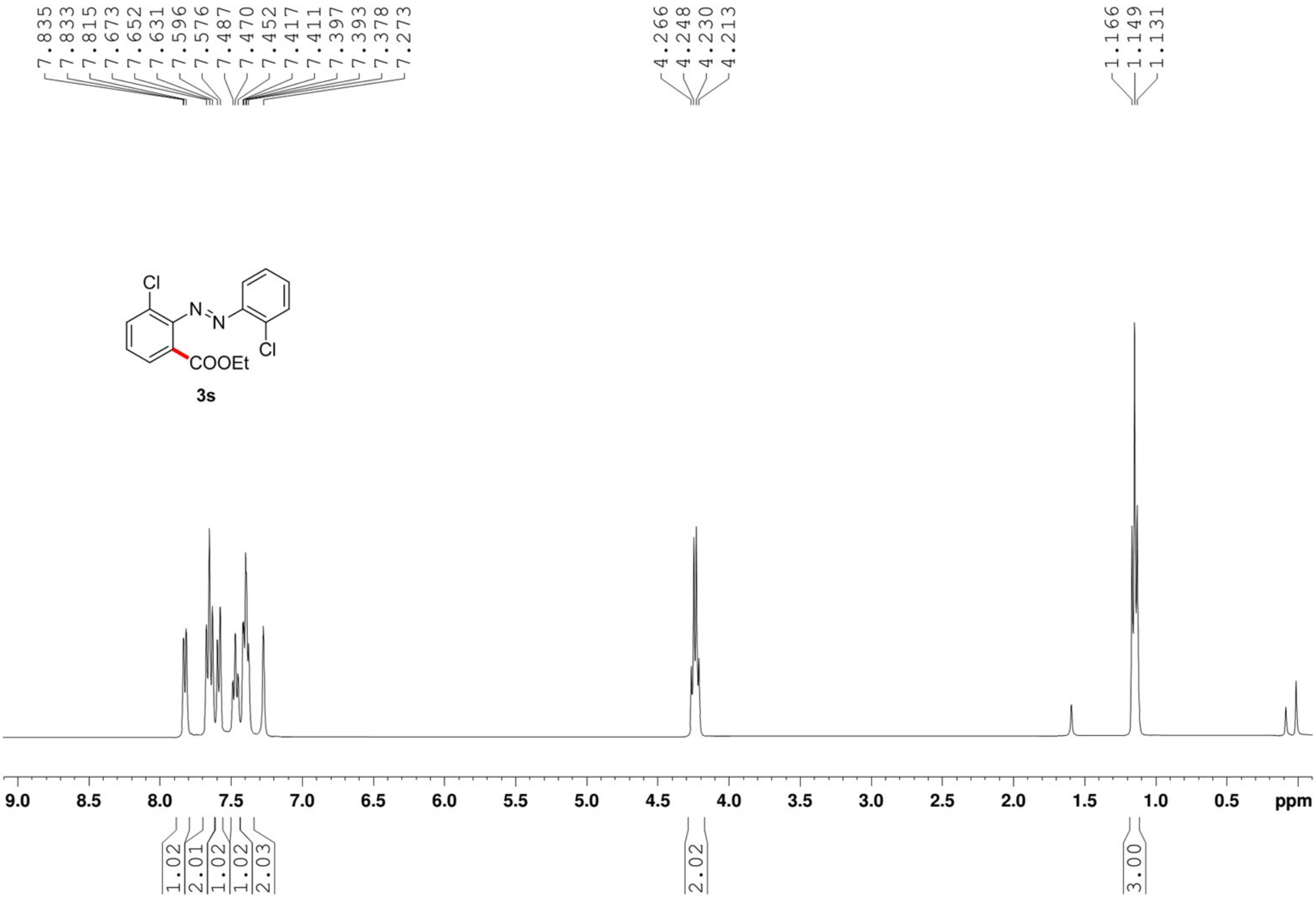
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.673
7.652
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7.576
7.487
7.470
7.452
7.417
7.411
7.397
7.393
7.378
7.273
4.266
4.248
4.230
4.213
1.166
1.149
1.131
Cl
N
N
Cl
COOEt
3s
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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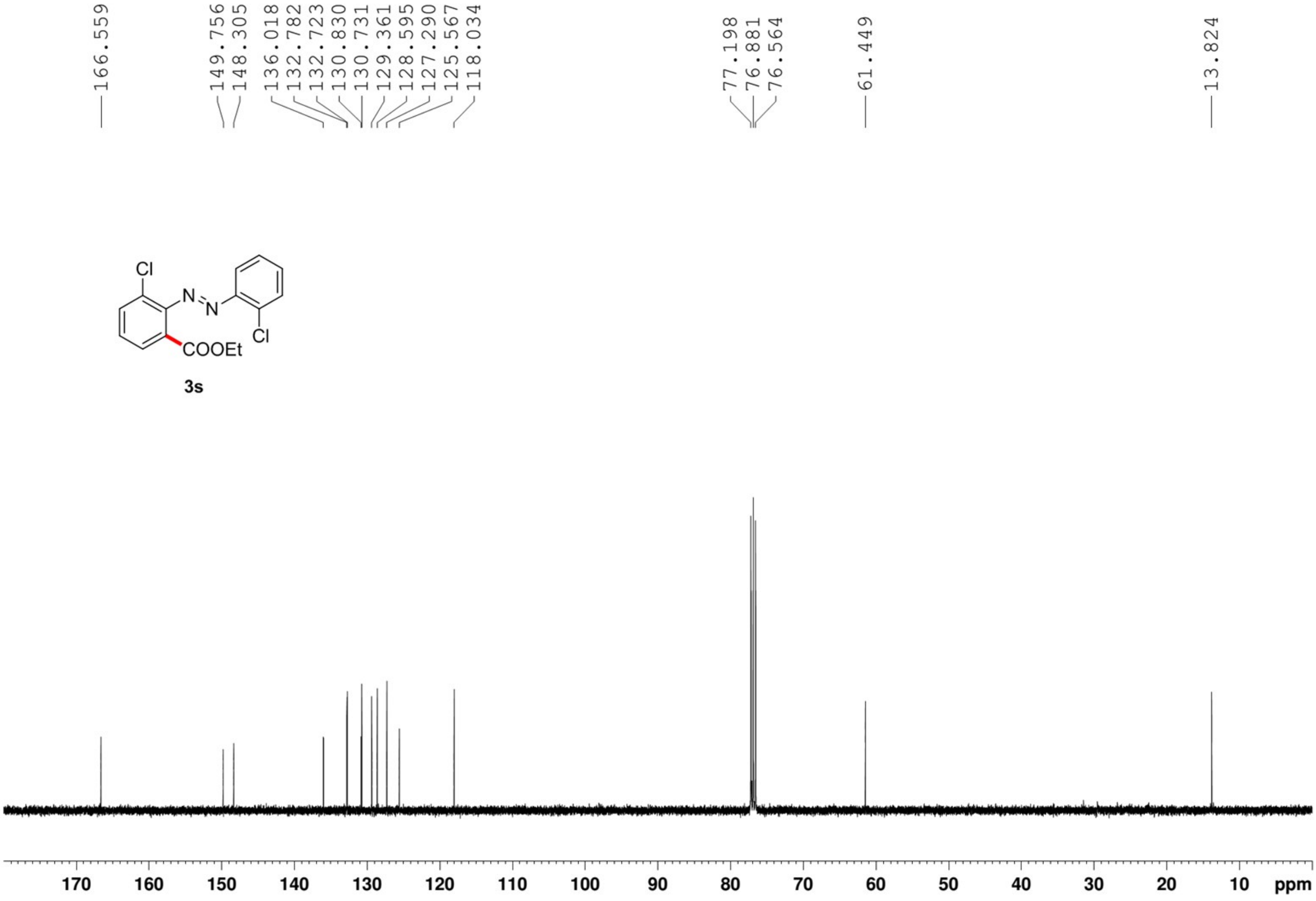
2.01
1.02
1.02
2.03
2.02
3.00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130.731
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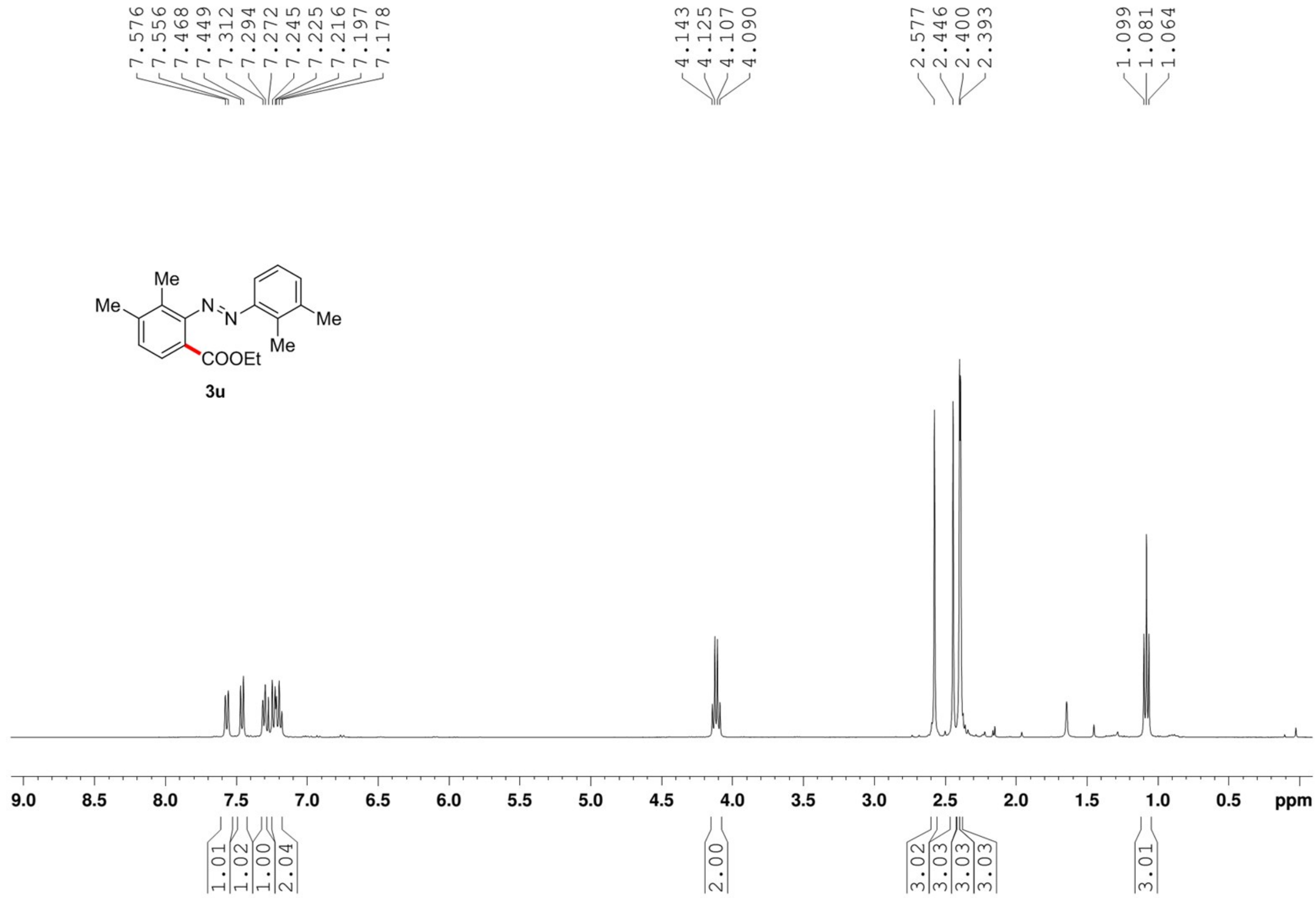
128.595
127.290
125.567
118.034
77.198
76.881
76.564
61.449
13.824
Cl
N
N
Cl
COOEt
3s
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.468
7.449
7.312
7.294
7.272
7.245
7.225
7.216
7.197
7.178
4.143
4.125
4.107
4.090
2.577
2.446
2.400
2.393
1.099
1.081
1.064
Me
Me
N
N
Me
Me
COOEt
3u
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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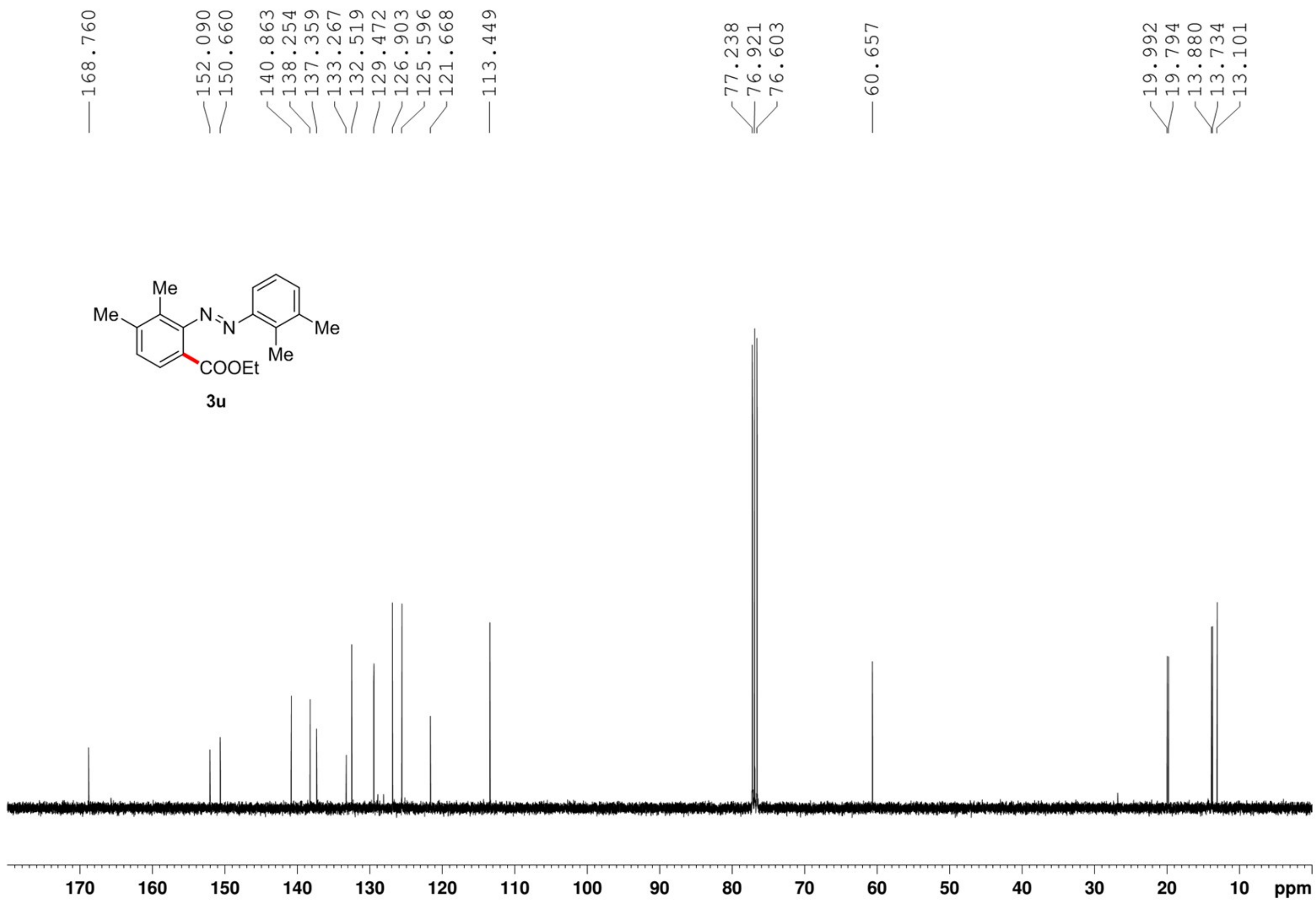
1.02
1.00
2.04
2.00
3.02
3.03
3.03
3.03
3.01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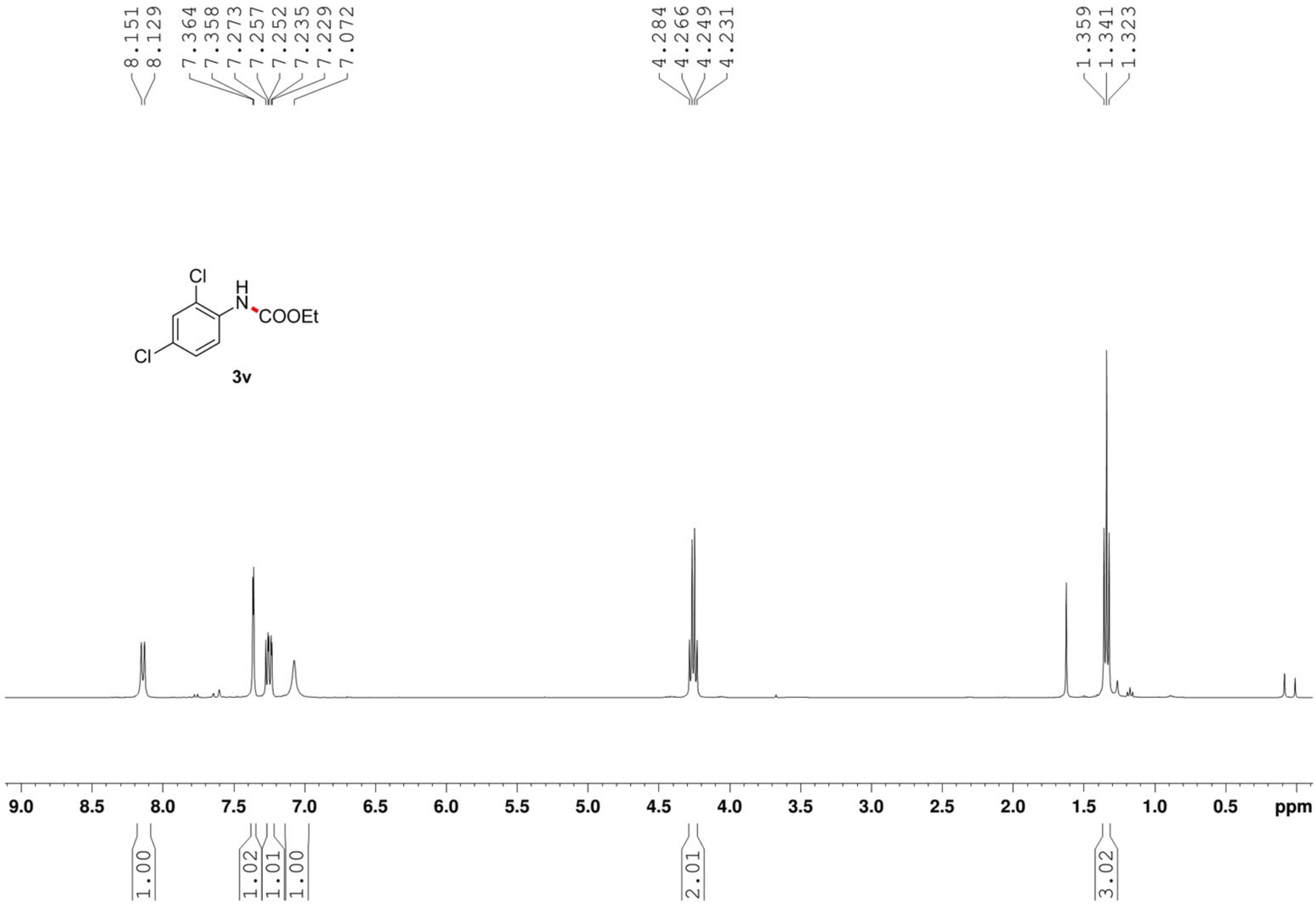
137.359
133.267
132.519
129.472
126.903
125.596
121.668
113.449
77.238
76.921
76.603
60.657
19.992
19.794
13.880
13.734
13.101
Me
Me
N
N
Me
Me
COOEt
3u
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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8.129
7.364
7.358
7.273
7.257
7.252
7.235
7.229
7.072
4.284
4.266
4.249
4.231
1.359
1.341
1.323
Cl
H
N
COOEt
Cl
3v
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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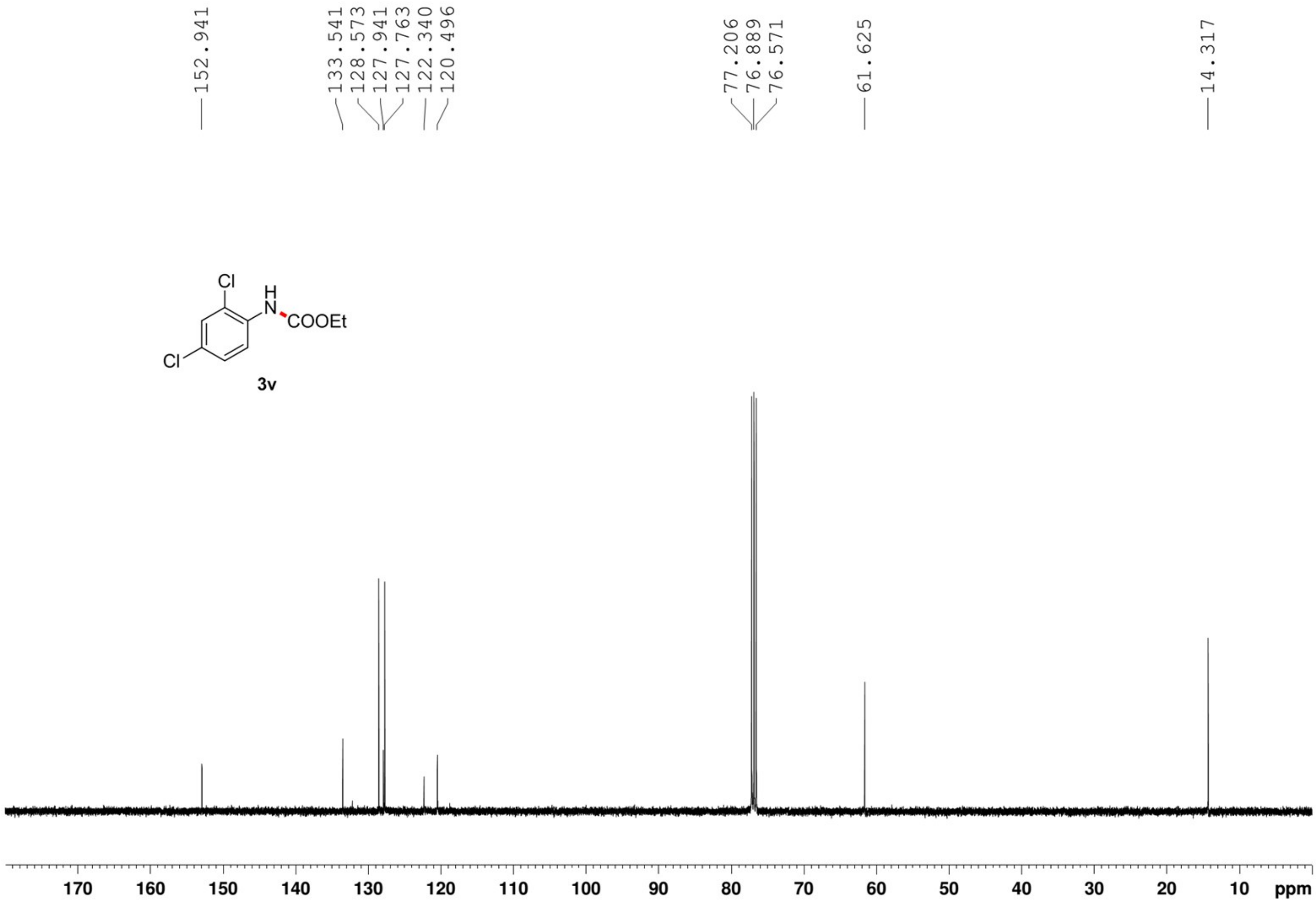
1.02
1.01
1.00
2.01
3.02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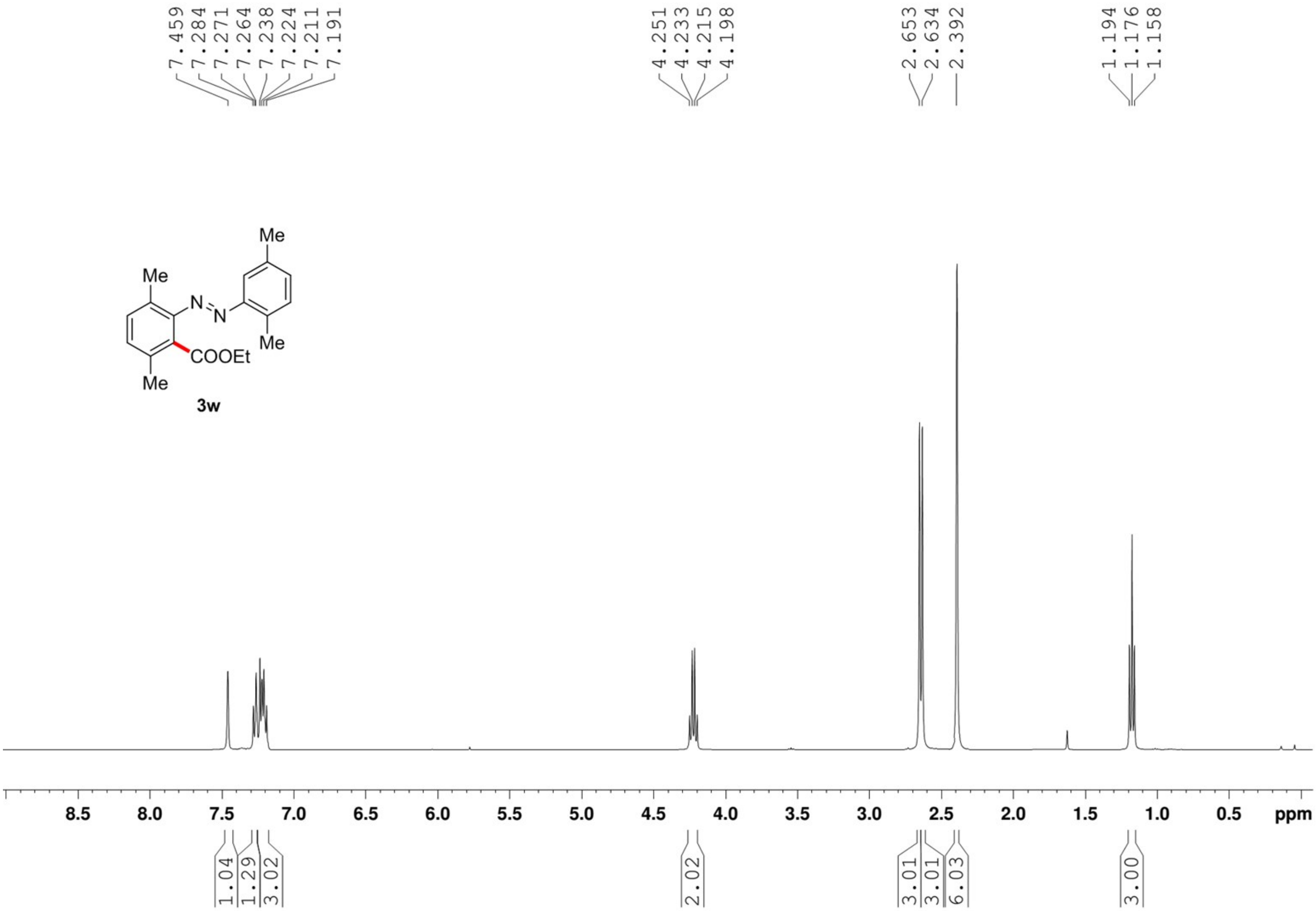
128.573
127.941
127.763
122.340
120.496
77.206
76.889
76.571
61.625
14.317
Cl
H
N
COOEt
Cl
3v
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.264
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7.224
7.211
7.191
4.251
4.233
4.215
4.198
2.653
2.634
2.392
1.194
1.176
1.158
Me
Me
N
N
Me
COOEt
Me
3w
1.04
1.29
3.02
2.02
3.01
3.01
6.03
3.00
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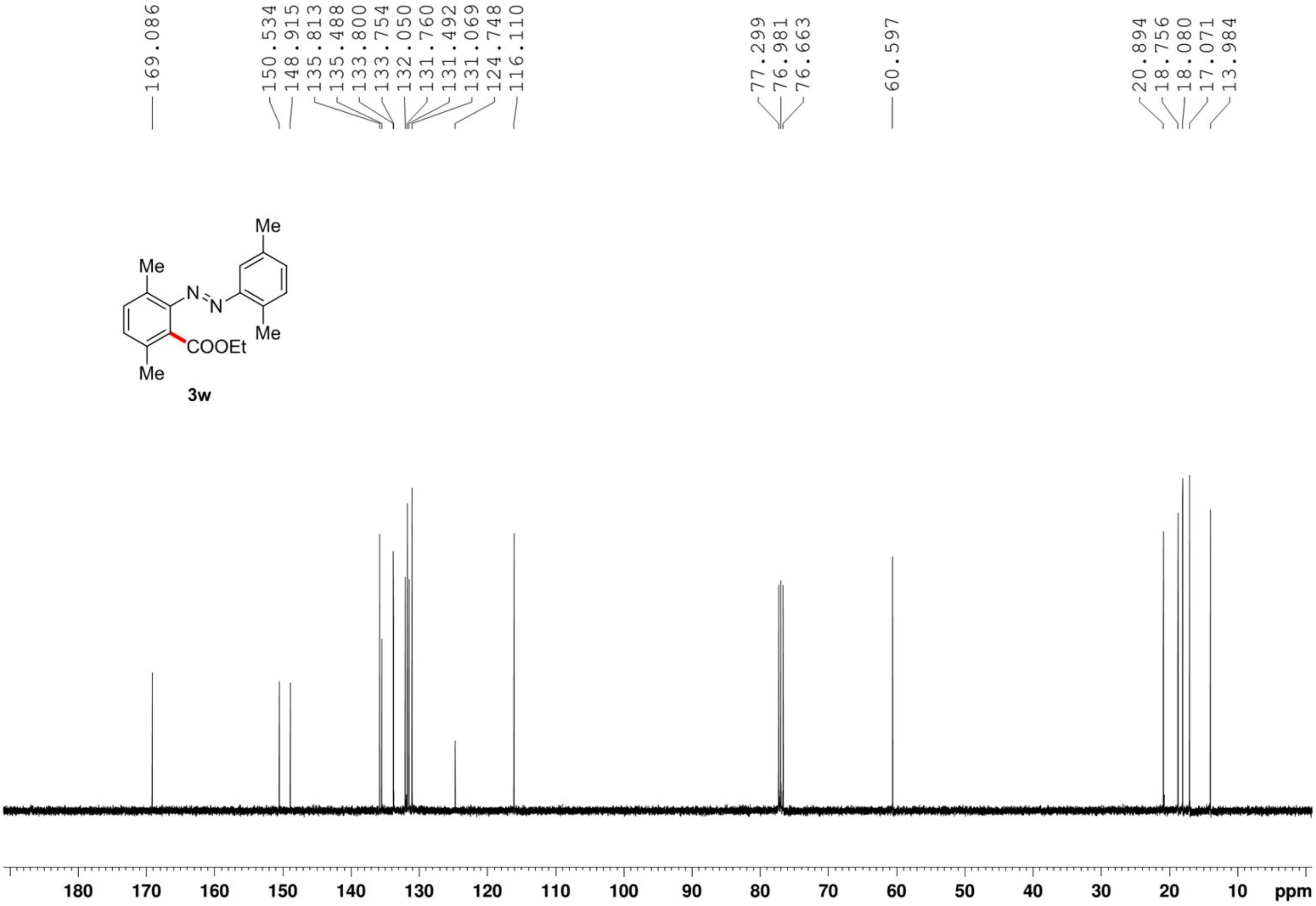
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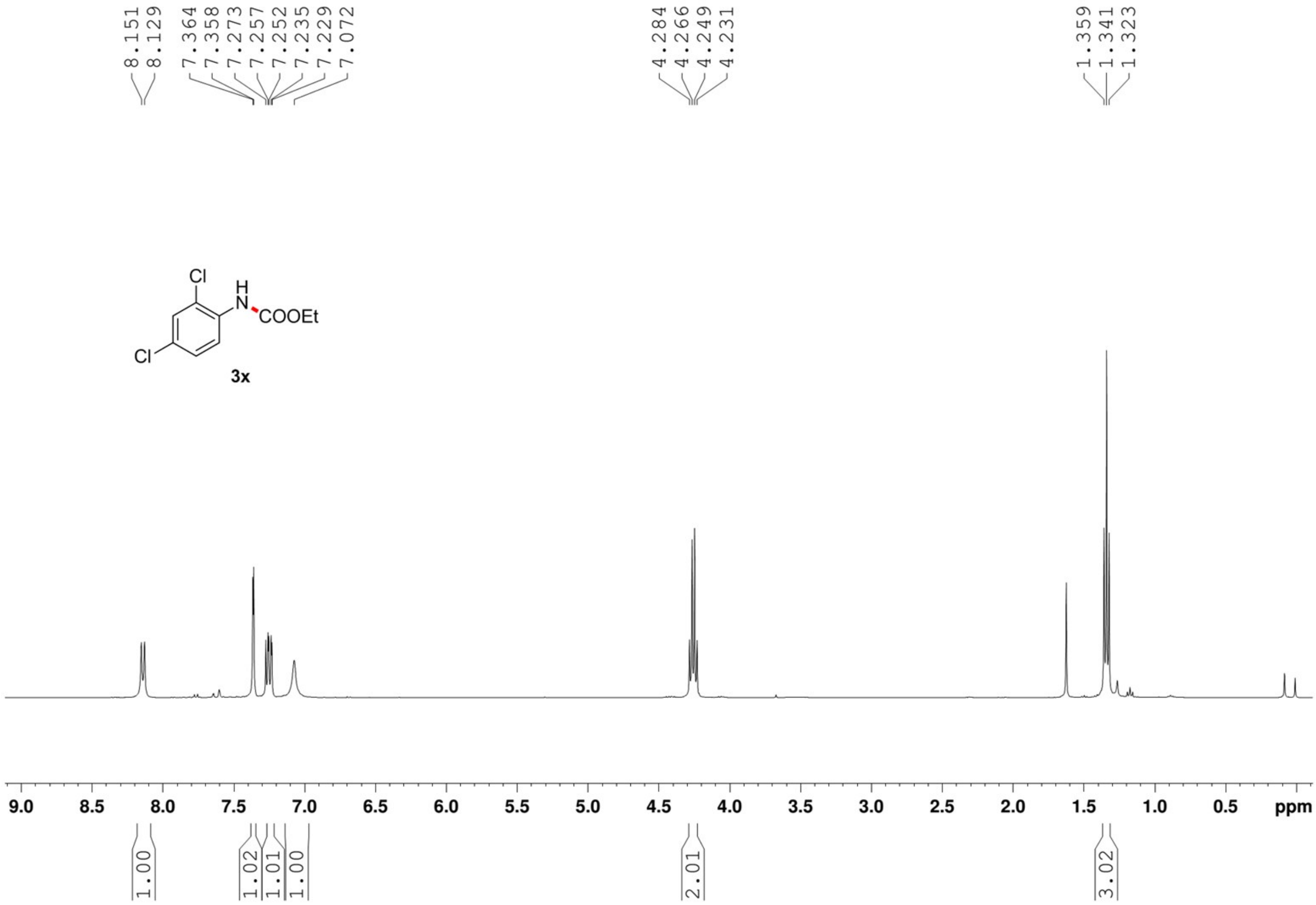
132.050
131.760
131.492
131.069
124.748
116.110
77.299
76.981
76.663
60.597
20.894
18.756
18.080
17.071
13.984
Me
Me
N
N
Me
COOEt
Me
3w
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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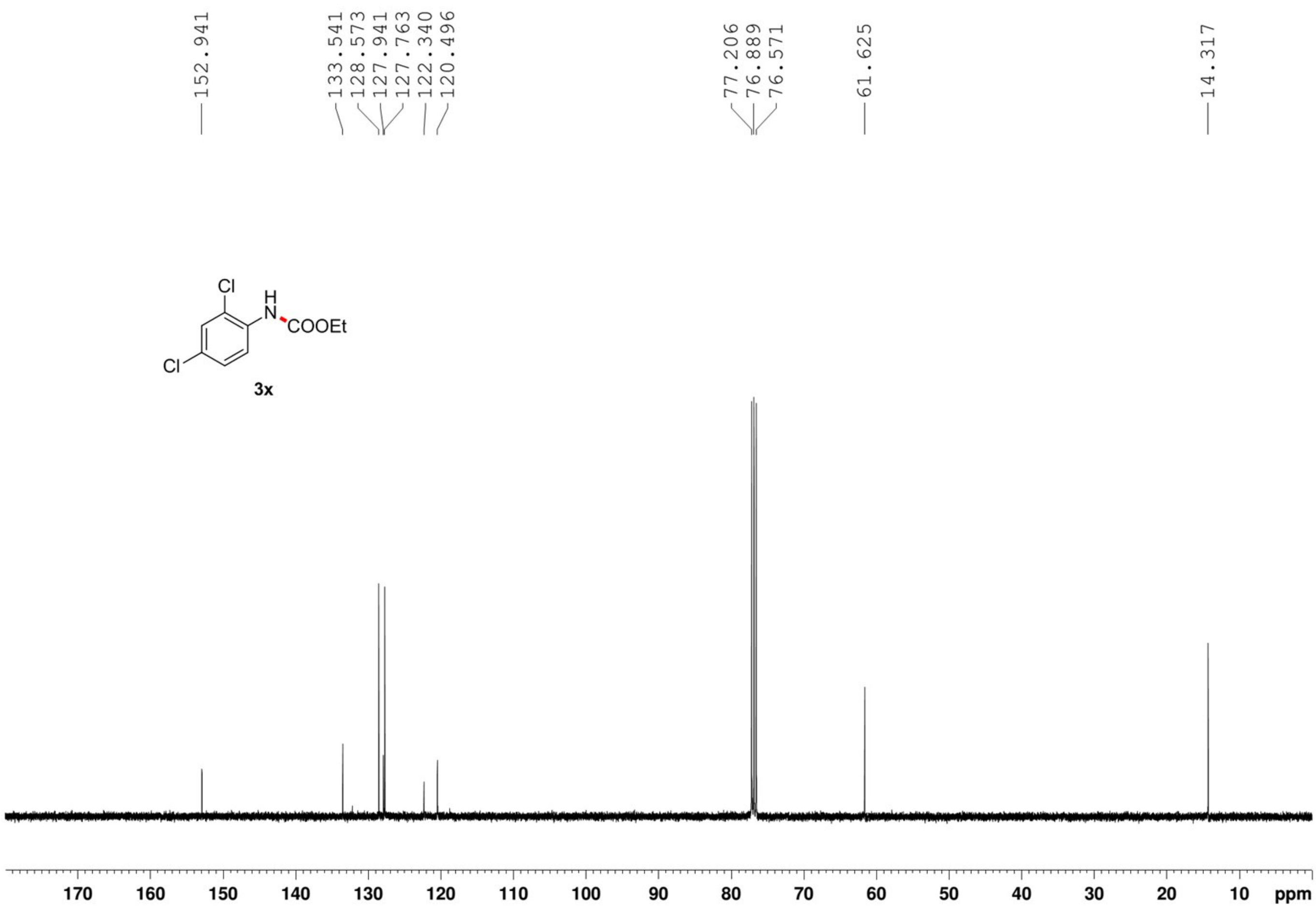
8.129
7.364
7.358
7.273
7.257
7.252
7.235
7.229
7.072
4.284
4.266
4.249
4.231
1.359
1.341
1.323
Cl
H
N
COOEt
Cl
3x
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.02
1.01
1.00
2.01
3.02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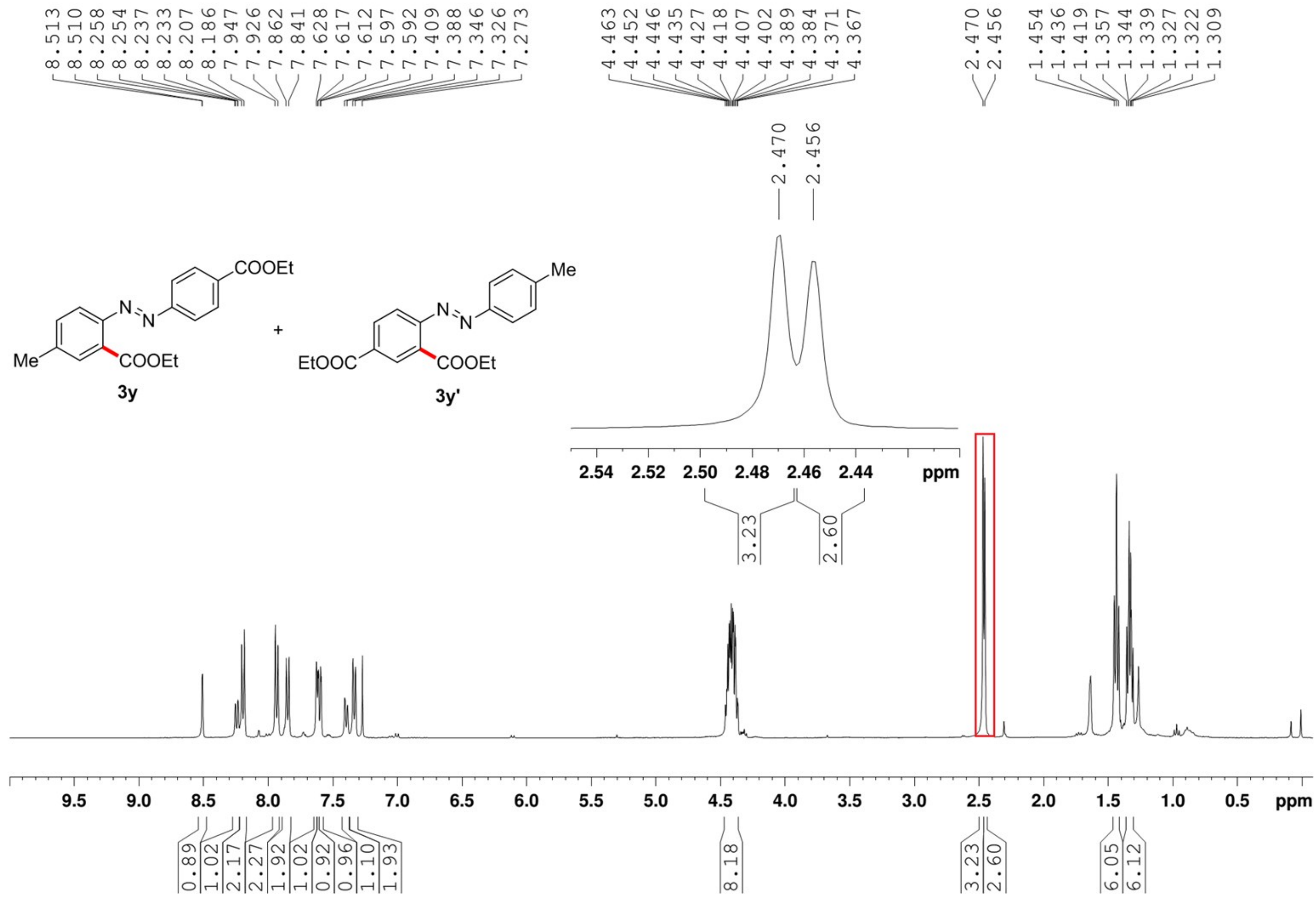
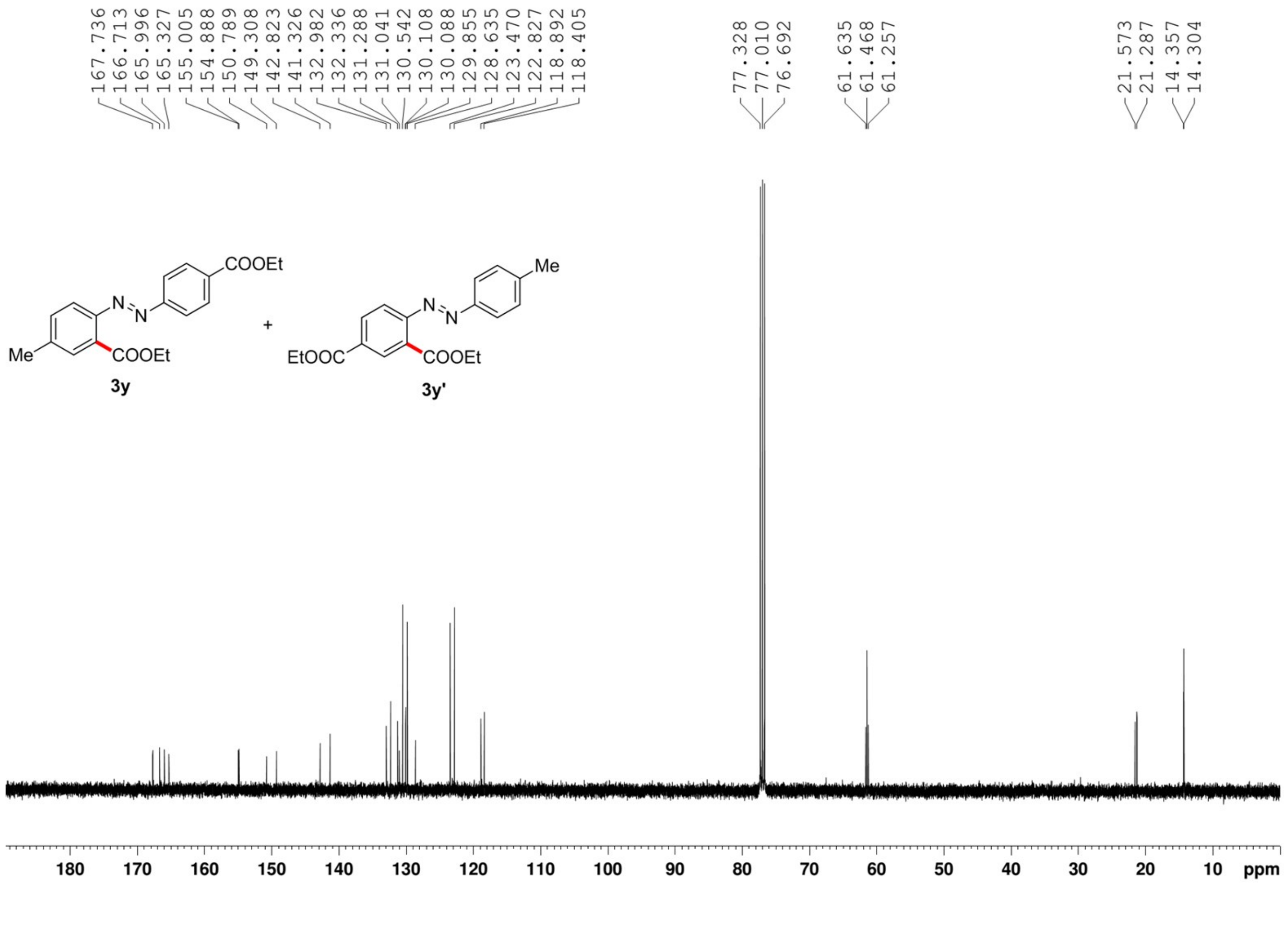
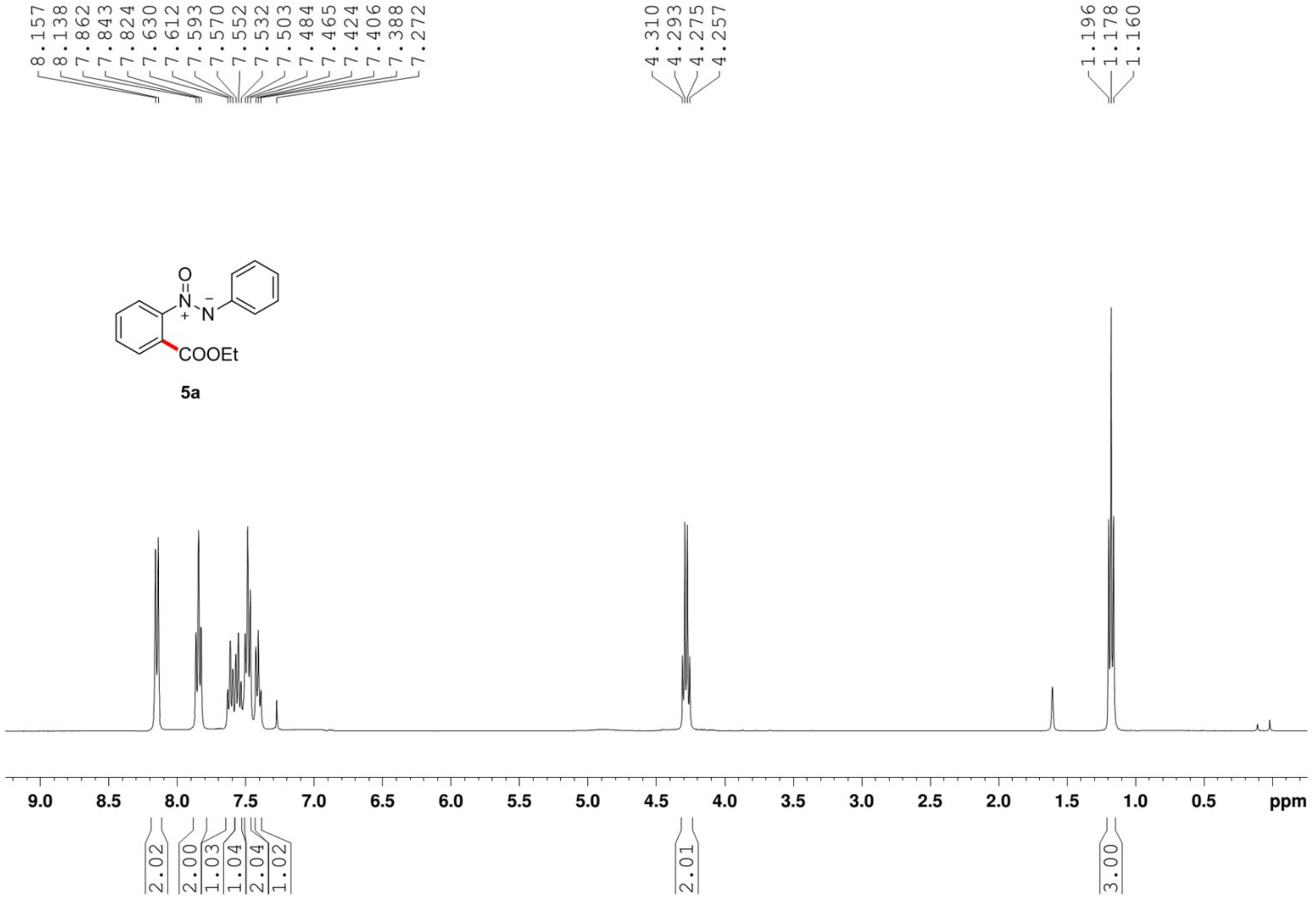
127.941
127.763
122.340
120.496
77.206
76.889
76.571
61.625
14.317
Cl
H
N
COOEt
Cl
3x
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



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7.593
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7.552
7.532
7.503
7.484
7.465
7.424
7.406
7.388
7.272
4.310
4.293
4.275
4.257
1.196
1.178
1.160
COOEt
5a
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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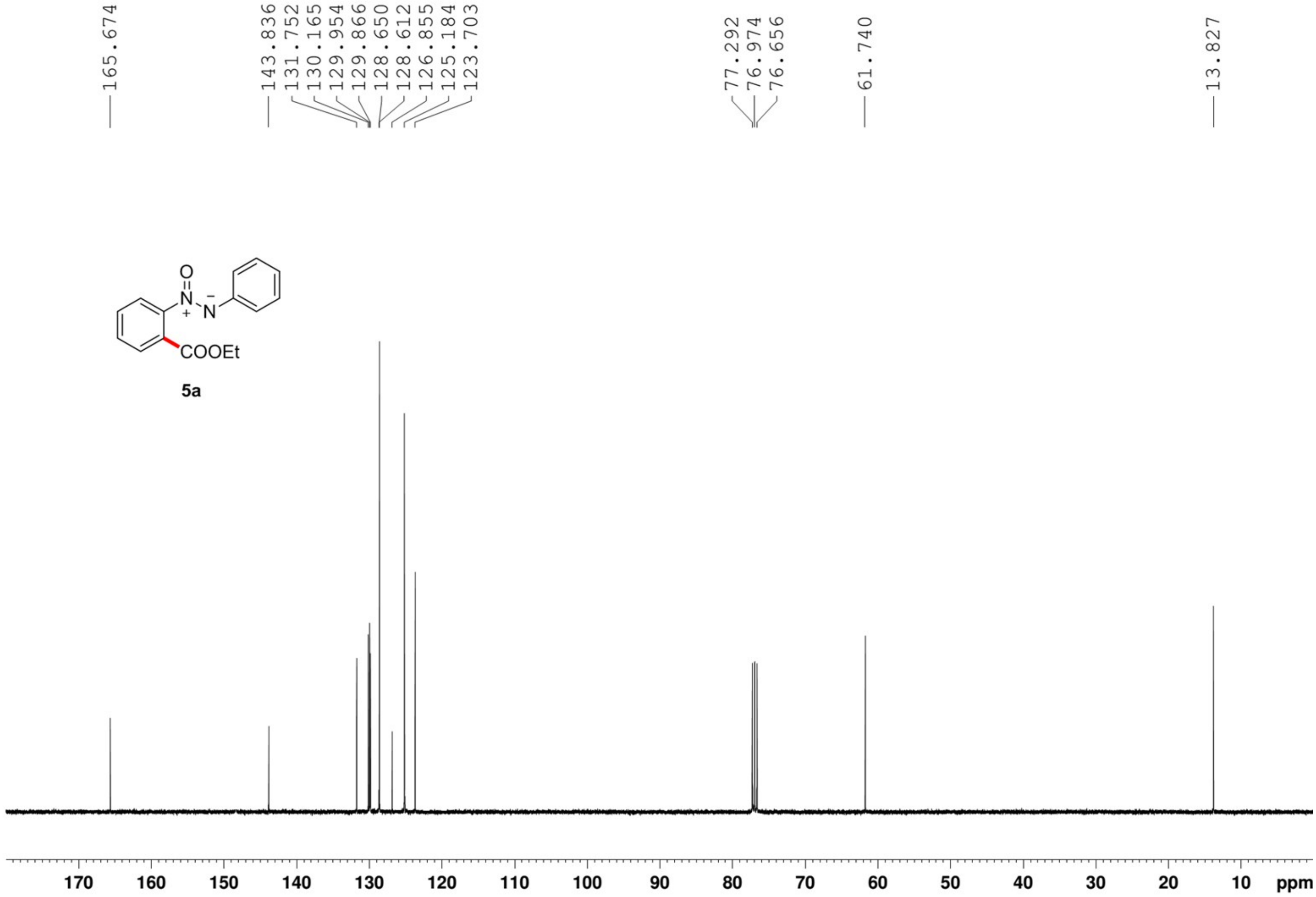
2.00
1.03
1.04
2.04
1.02
2.01
3.00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128.612
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125.184
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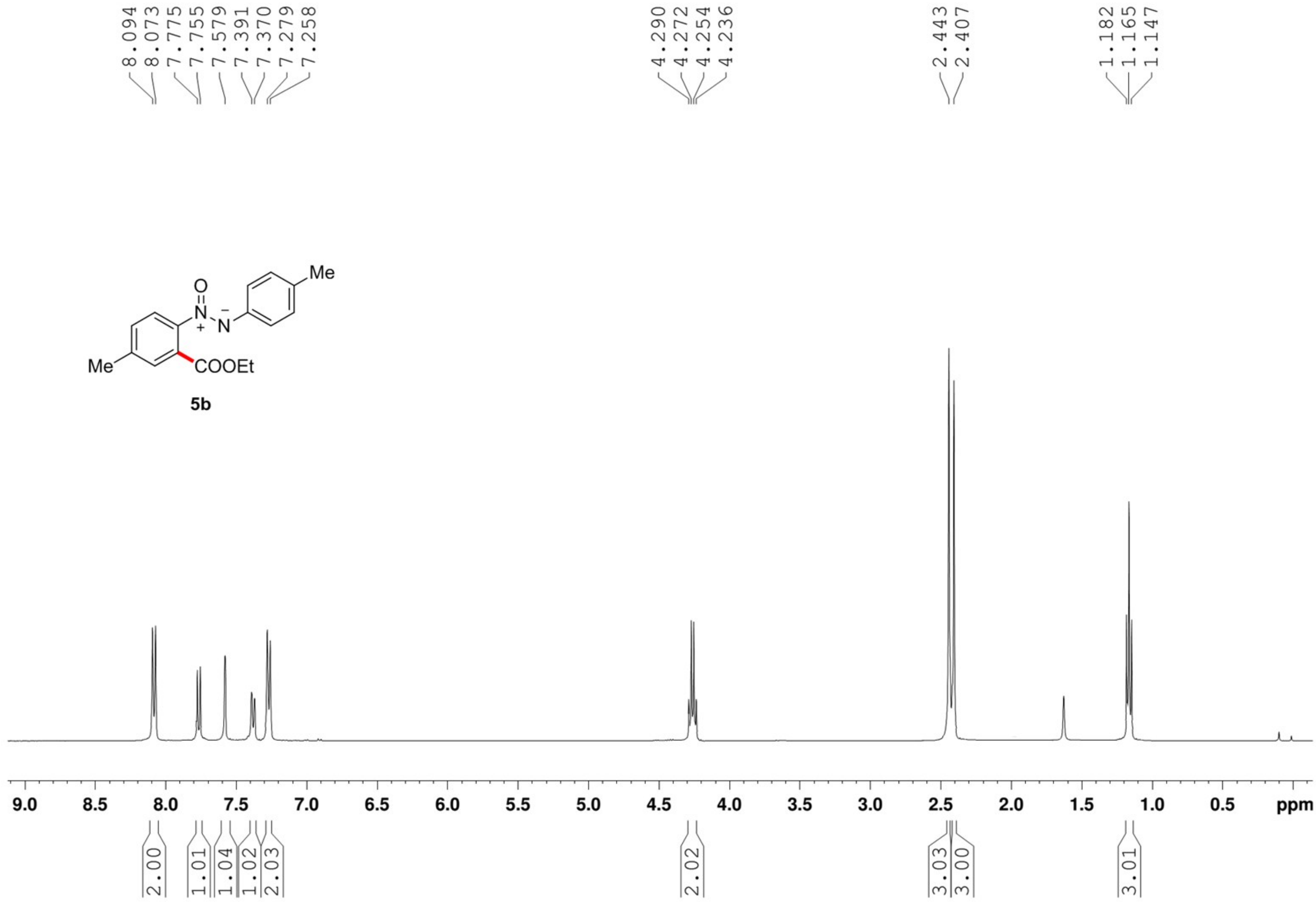
76.974
76.656
61.740
13.827
COOEt
5a
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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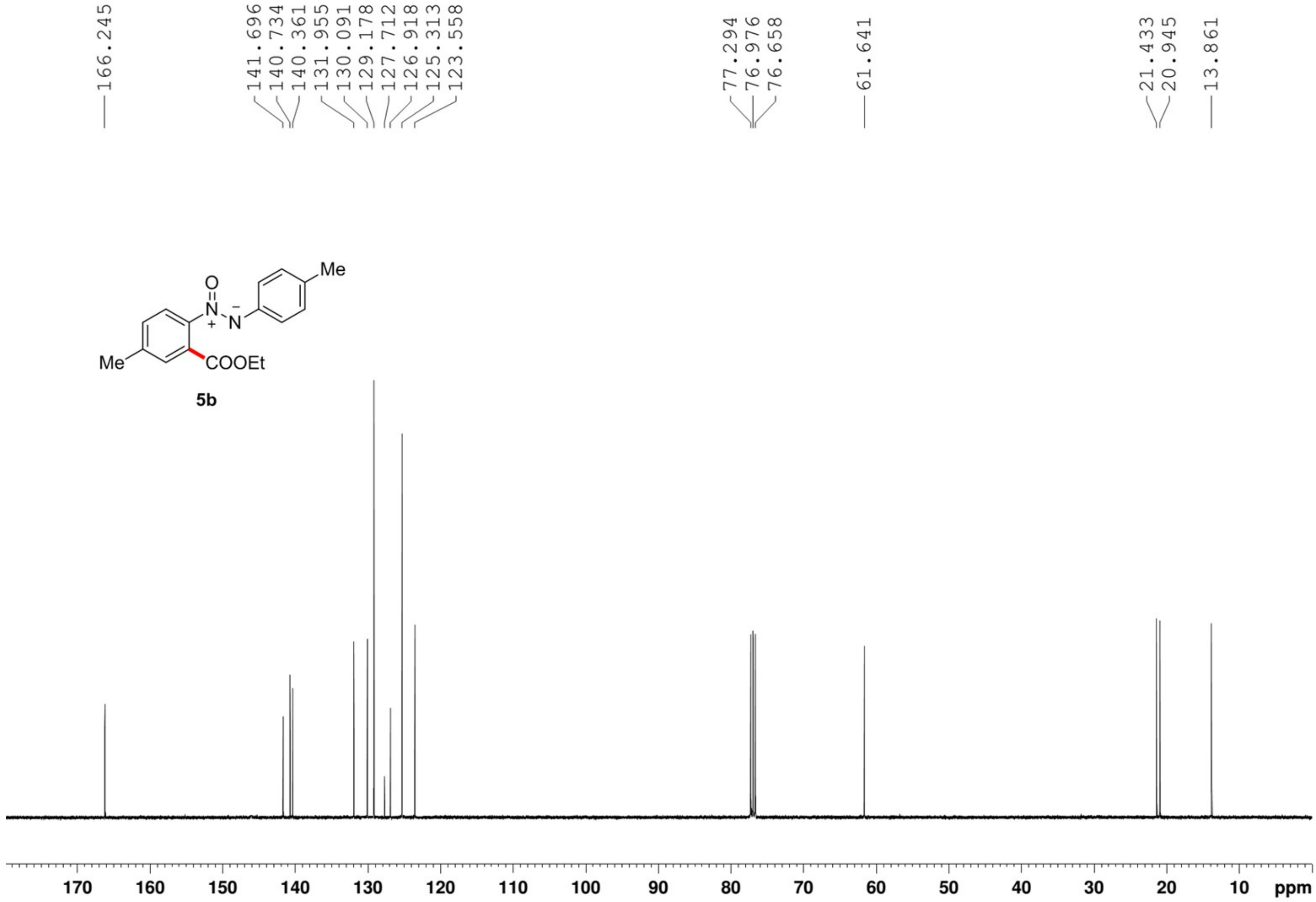
7.370
7.279
7.258
4.290
4.272
4.254
4.236
2.443
2.407
1.182
1.165
1.147
Me
O
N
+
–
N
Me
COOEt
5b
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
1.01
1.04
1.02
2.03
2.02
3.03
3.00
3.01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127.712
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125.313
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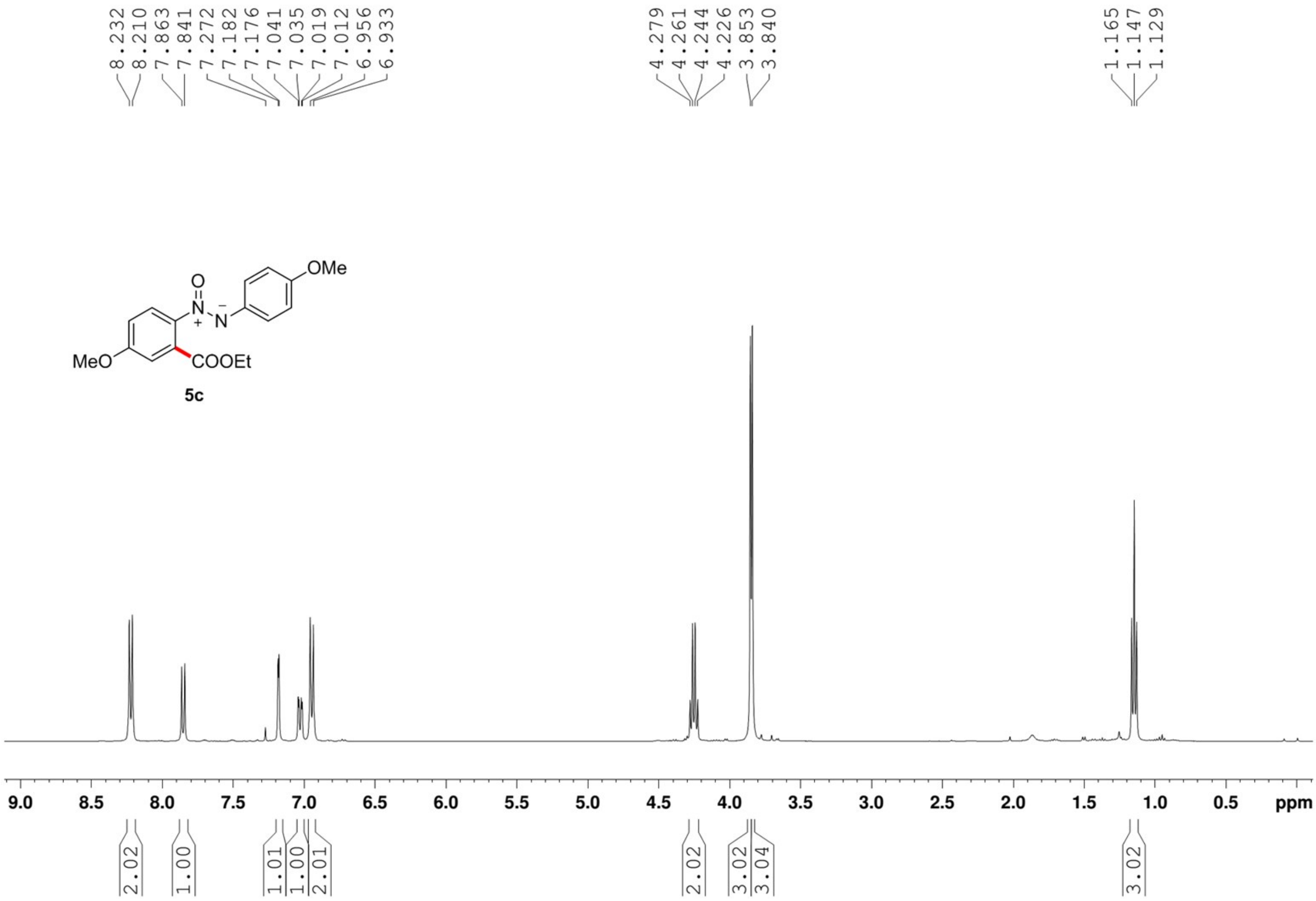
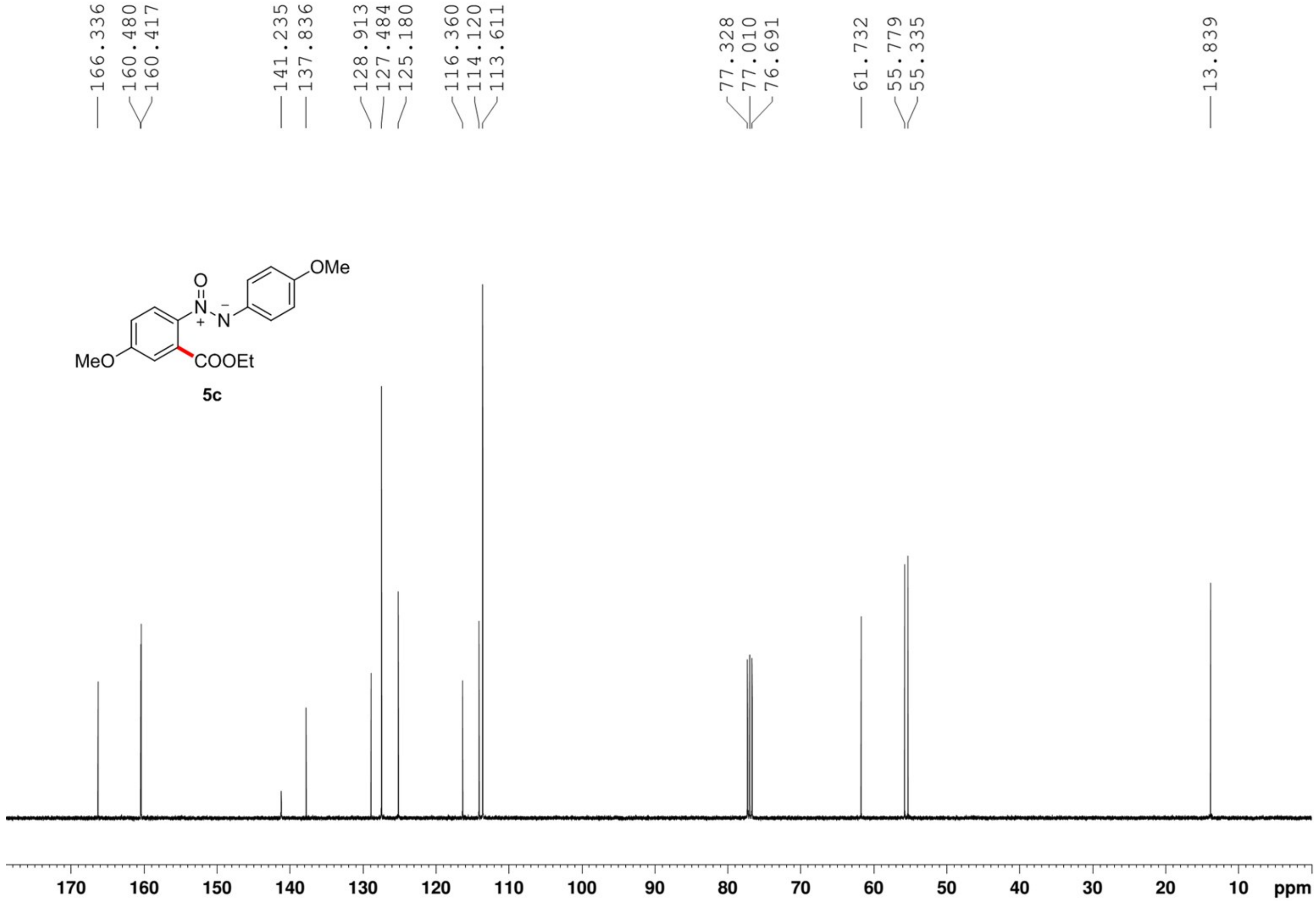
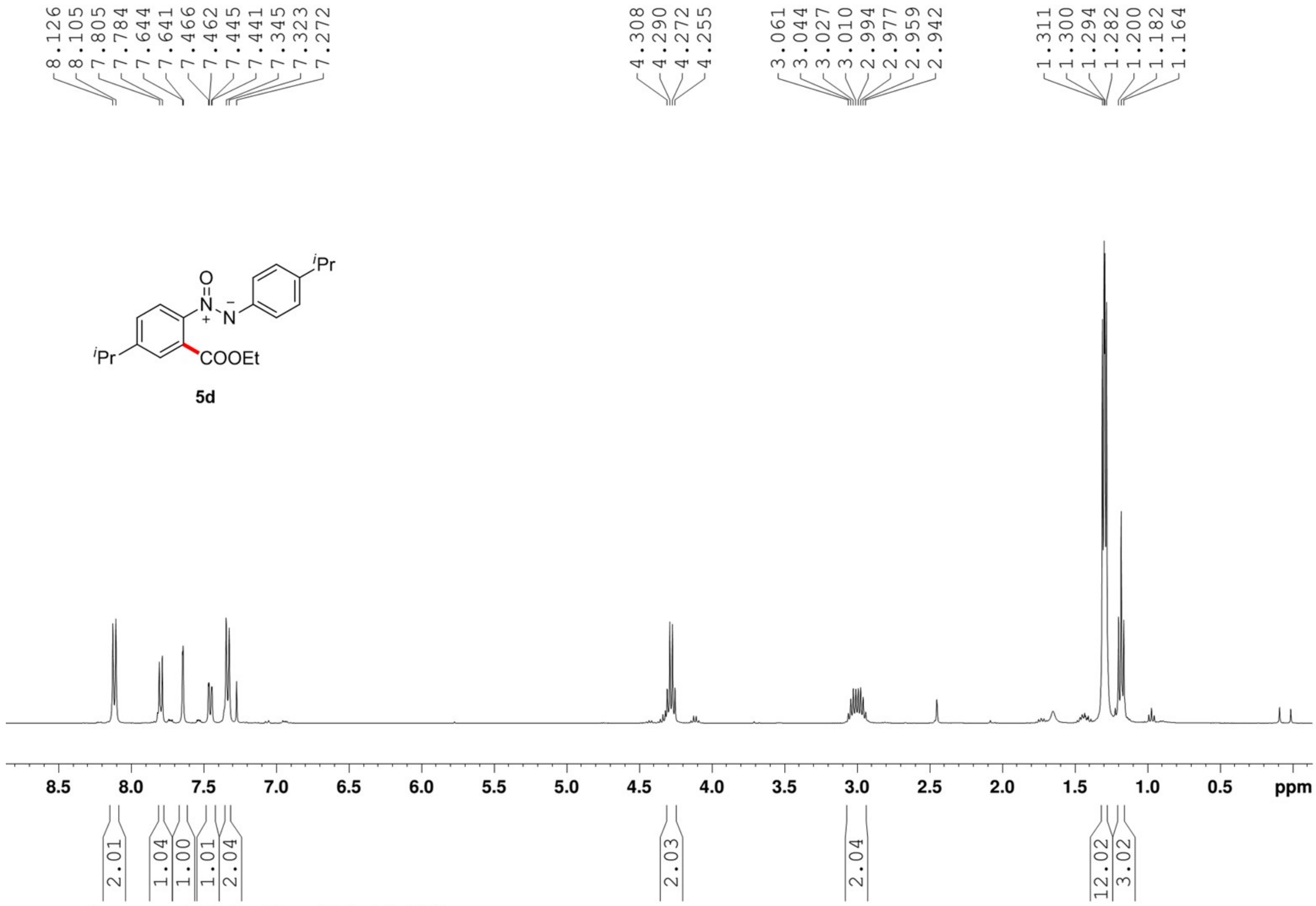
77.294
76.976
76.658
61.641
21.433
20.945
13.861
Me
O
N
+
–
N
Me
COOEt
5b
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



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8.105
7.805
7.784
7.644
7.641
7.466
7.462
7.445
7.441
7.345
7.323
7.272
4.308
4.290
4.272
4.255
3.061
3.044
3.027
3.010
2.994
2.977
2.959
2.942
1.311
1.300
1.294
1.282
1.200
1.182
1.164
O
N
N
+
–
iPr
iPr
COOEt
5d
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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ppm
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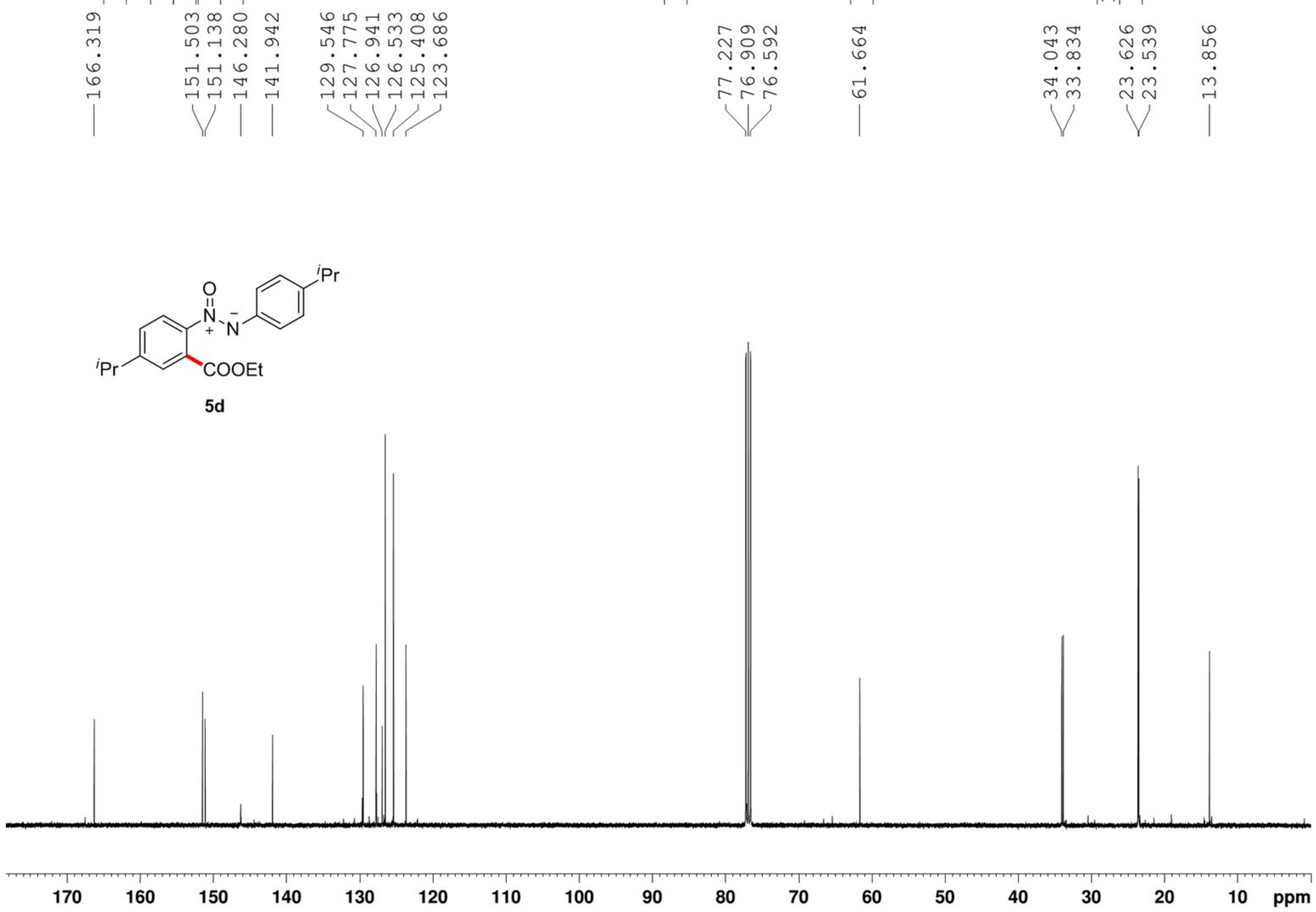
1.00
1.01
2.04
2.03
2.04
12.02
3.02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126.941
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76.909
76.592
61.664
34.043
33.834
23.626
23.539
13.856
O
N
N
+
–
iPr
iPr
COOEt
5d
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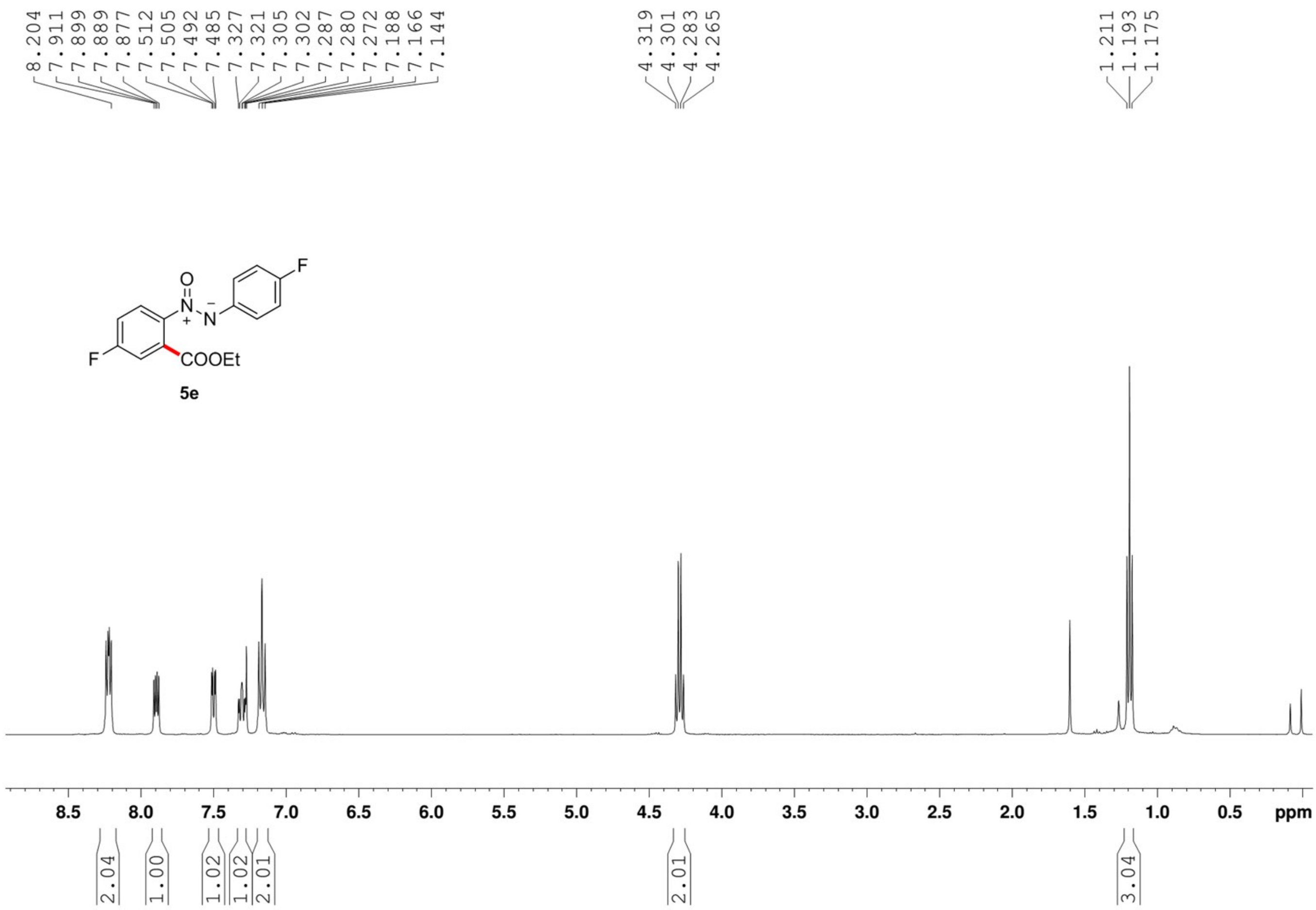
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.302
7.287
7.280
7.272
7.188
7.166
7.144
4.319
4.301
4.283
4.265
1.211
1.193
1.175
F
O
N
+
–
N
F
COOEt
5e
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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1.0
0.5
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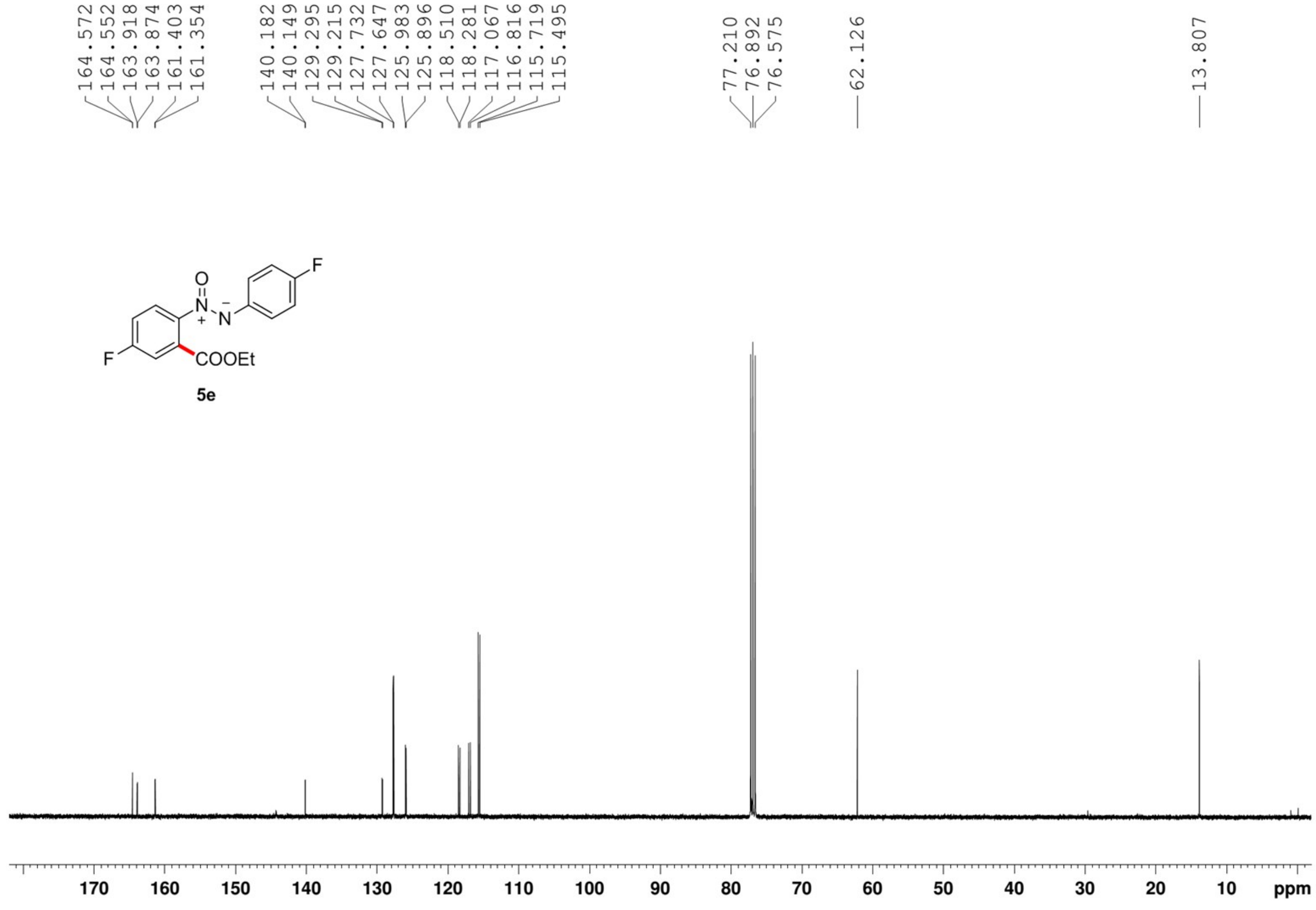
1.02
1.02
2.01
2.01
3.04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161.354
140.182
140.149
129.295
129.215
127.732
127.647
125.983
125.896
118.510
118.281
117.067
116.816
115.719
115.495
77.210
76.892
76.575
62.126
13.807
F
O
N
+
–
N
F
COOEt
5e
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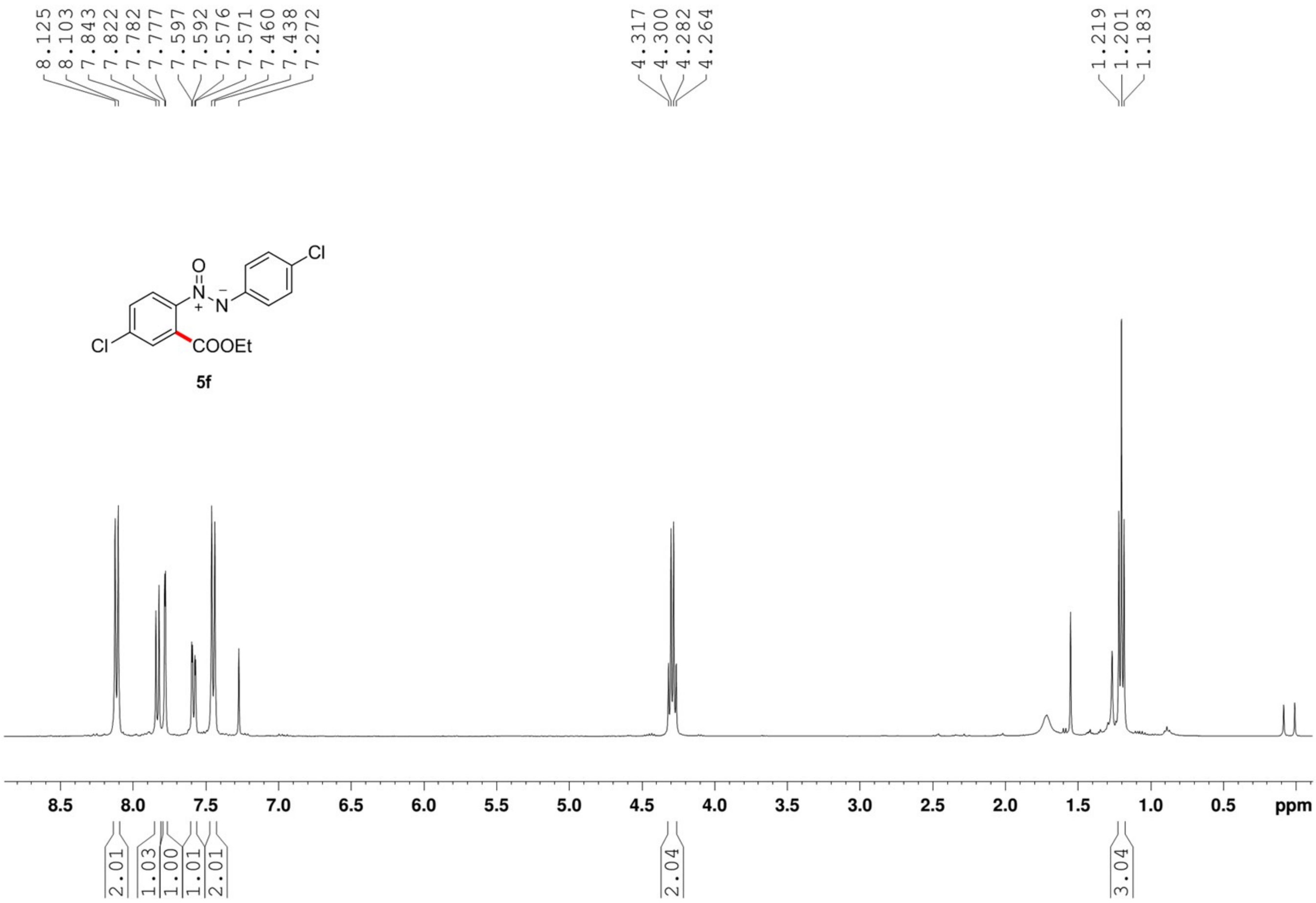
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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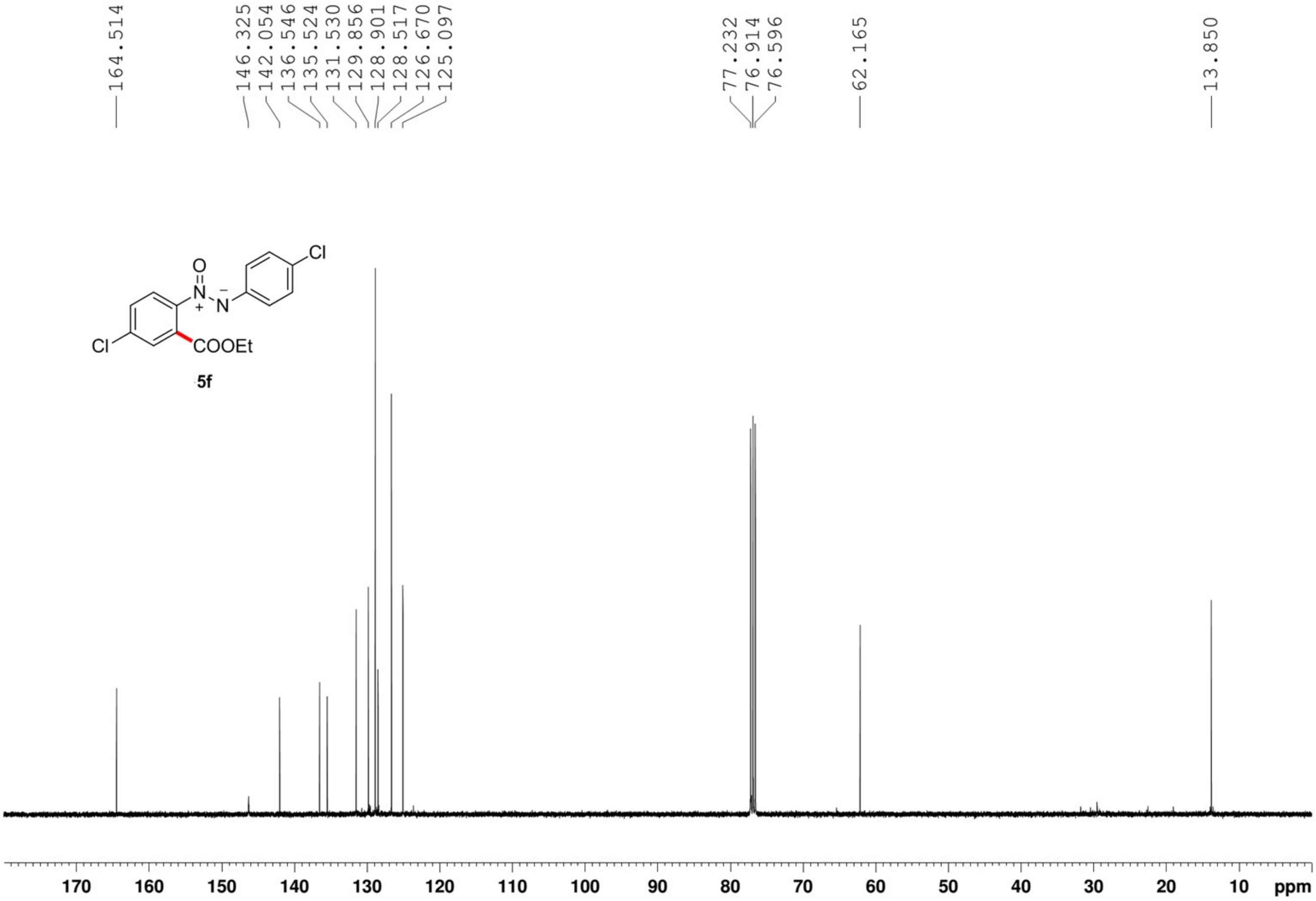
7.822
7.782
7.777
7.597
7.592
7.576
7.571
7.460
7.438
7.272
4.317
4.300
4.282
4.264
1.219
1.201
1.183
Cl
O
N
N
Cl
COOEt
5f
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.01
1.03
1.00
1.01
2.01
2.04
3.04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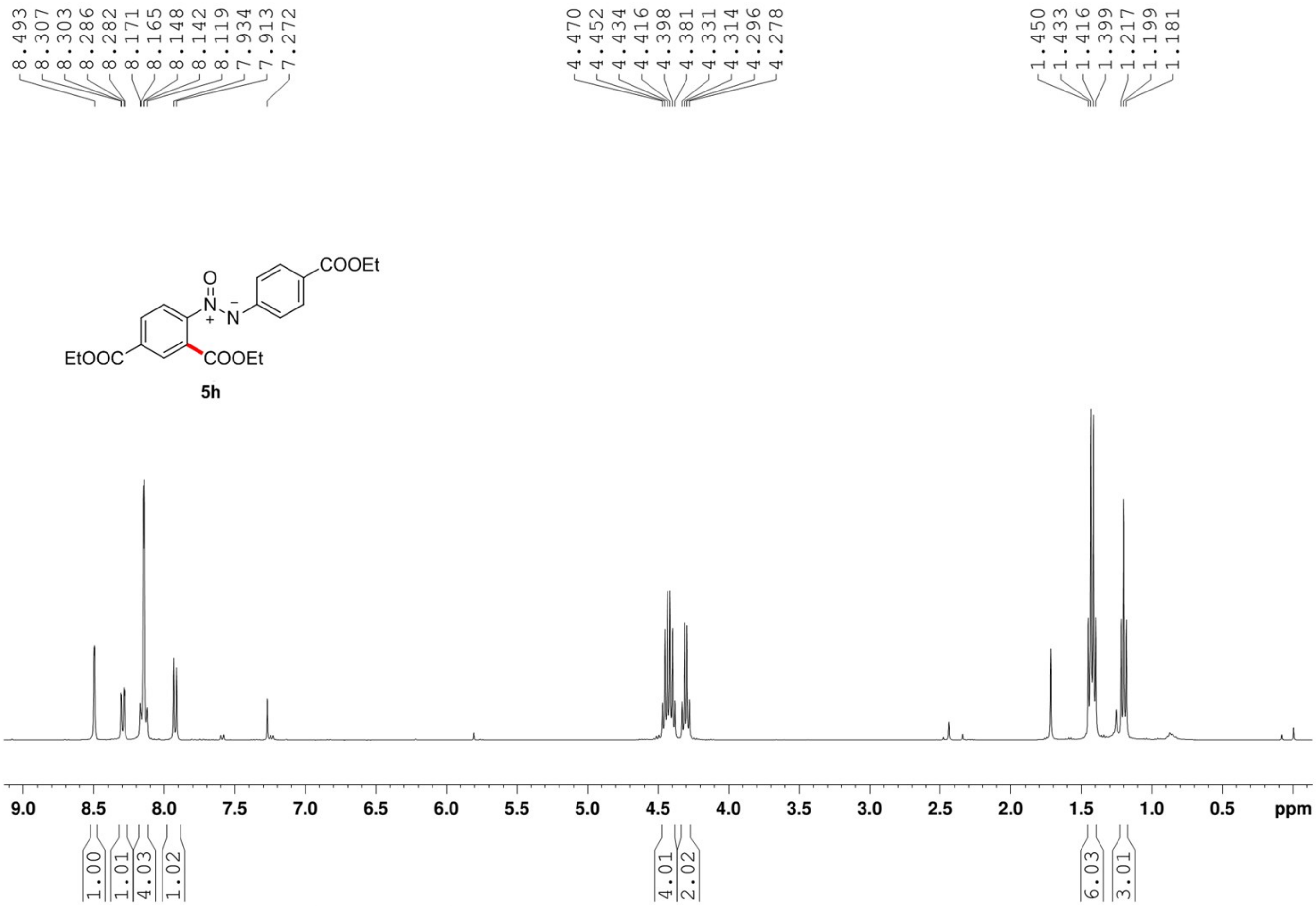
142.054
136.546
135.524
131.530
129.856
128.901
128.517
126.670
125.097
77.232
76.914
76.596
62.165
13.850
Cl
O
N
N
Cl
COOEt
5f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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8.307
8.303
8.286
8.282
8.171
8.165
8.148
8.142
8.119
7.934
7.913
7.272
4.470
4.452
4.434
4.416
4.398
4.381
4.331
4.314
4.296
4.278
1.450
1.433
1.416
1.399
1.217
1.199
1.181
COOEt
EtOOC
COOEt
5h
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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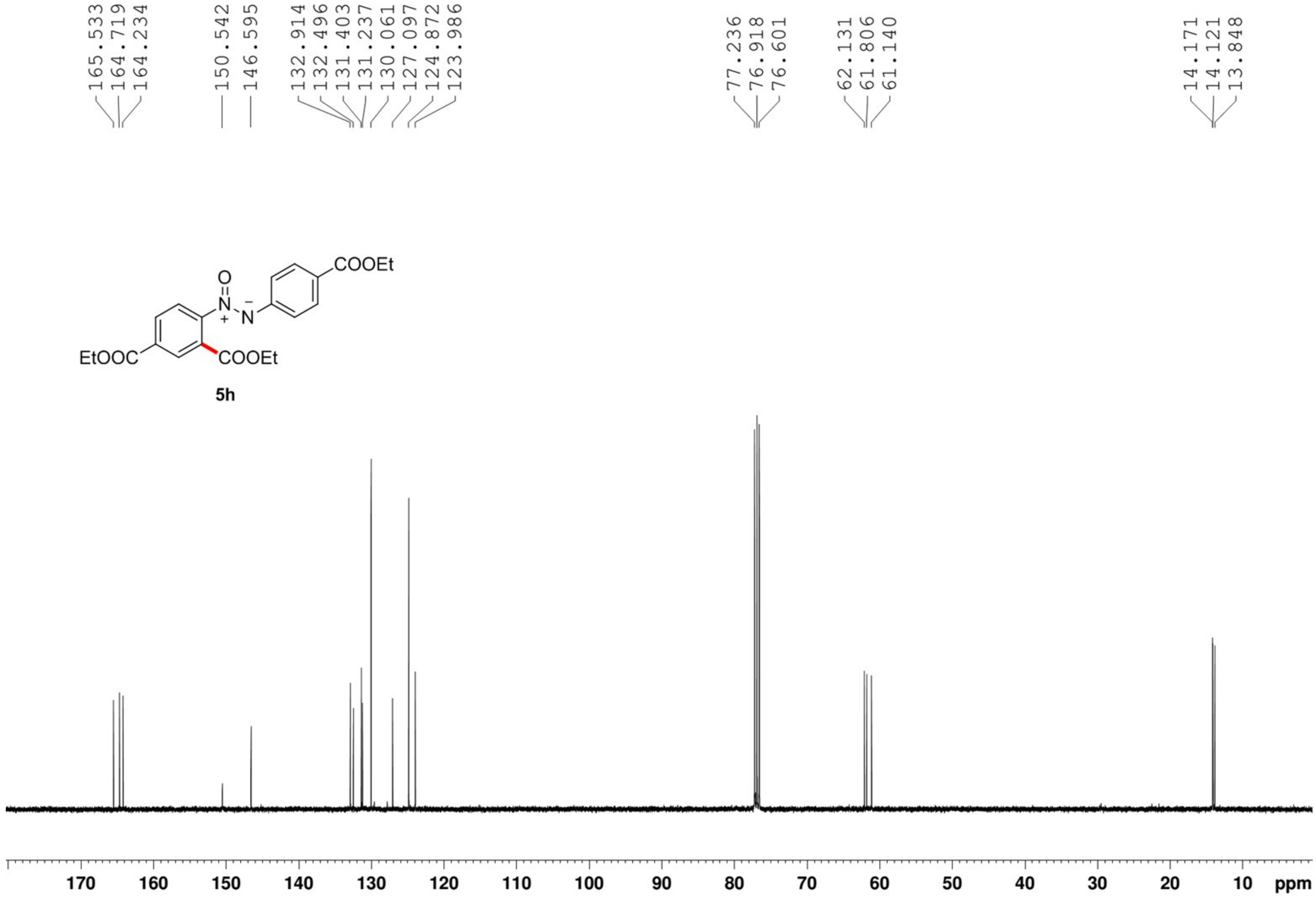
1.01
4.03
1.02
4.01
2.02
6.03
3.01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150.542
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132.496
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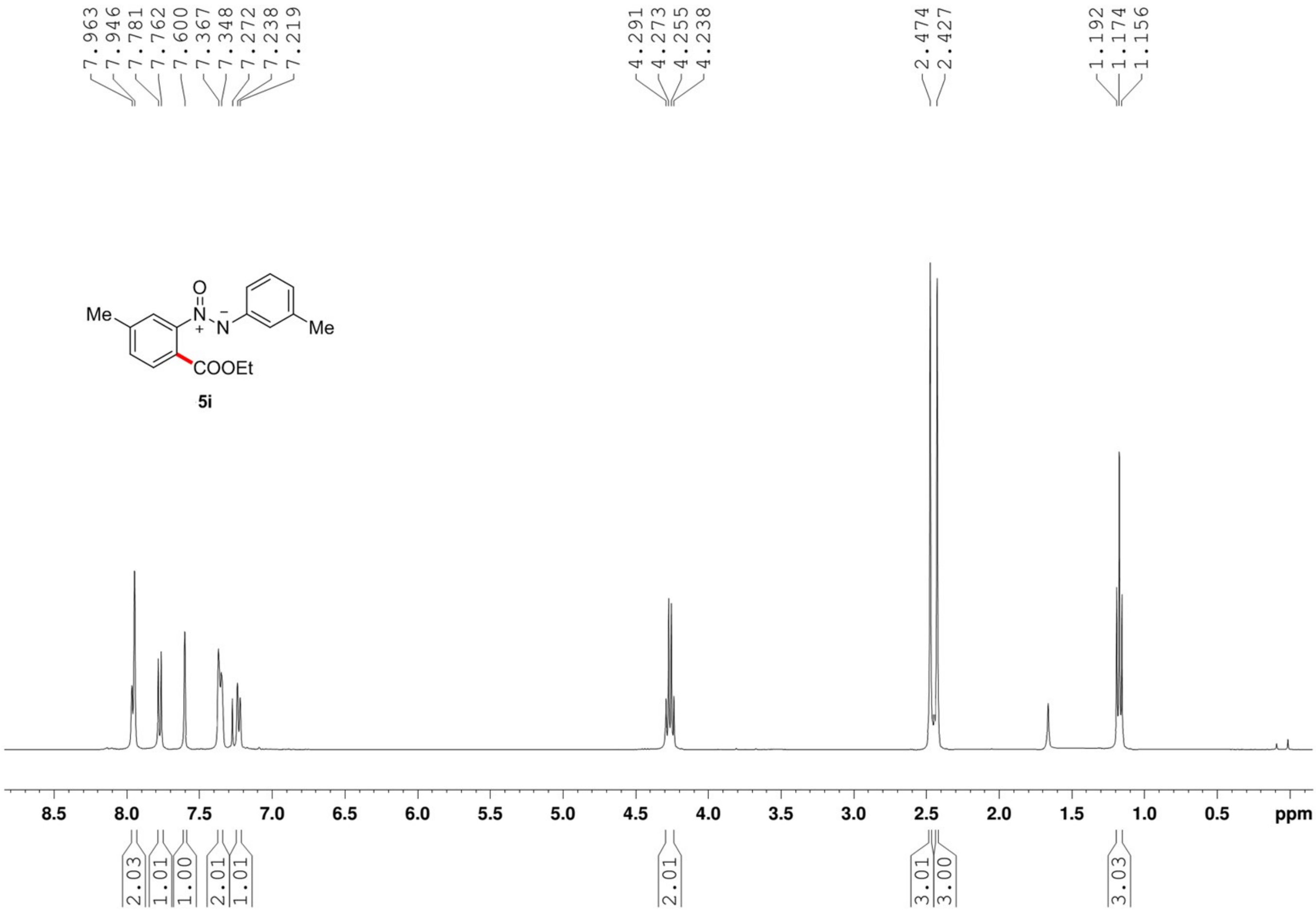
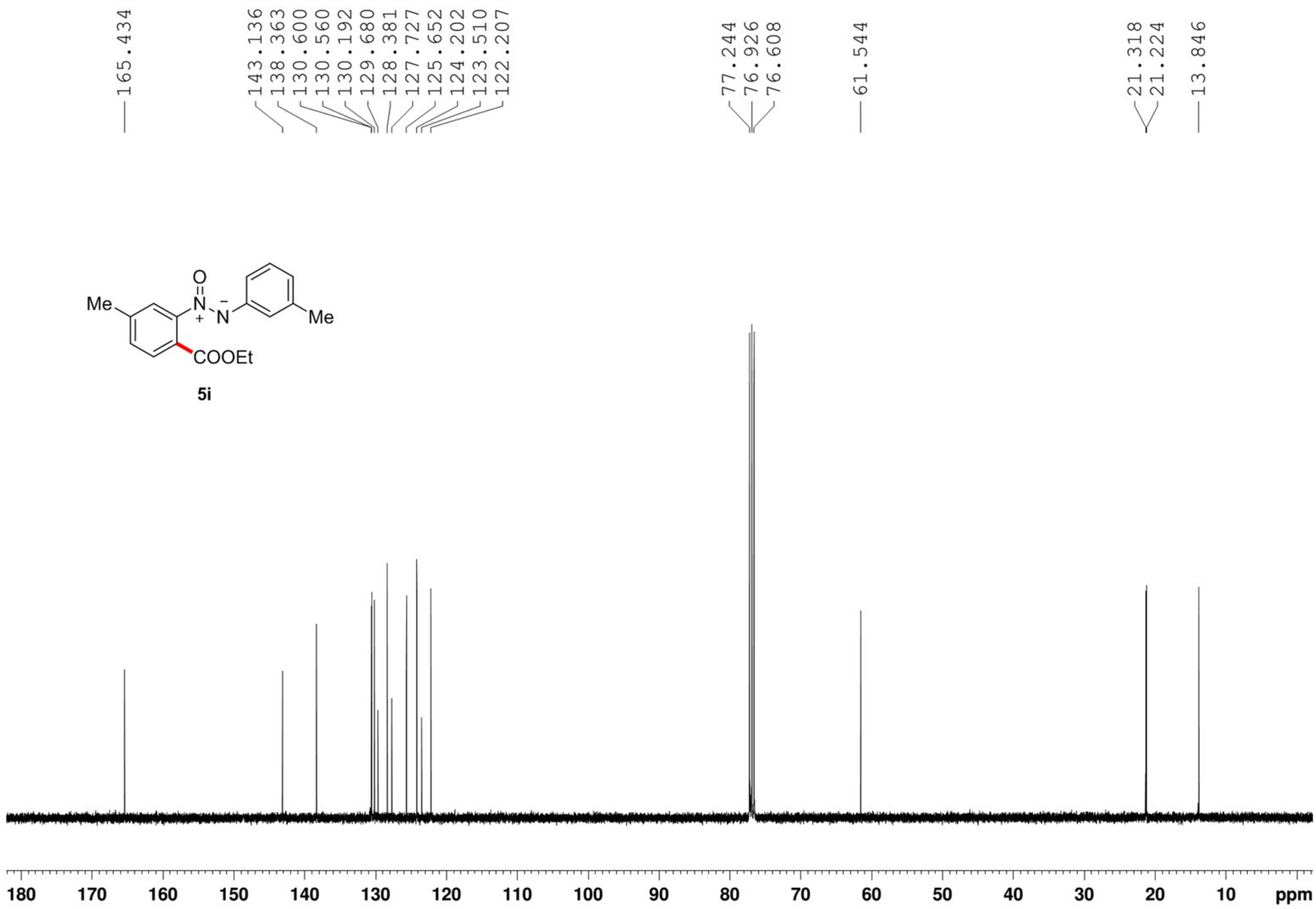
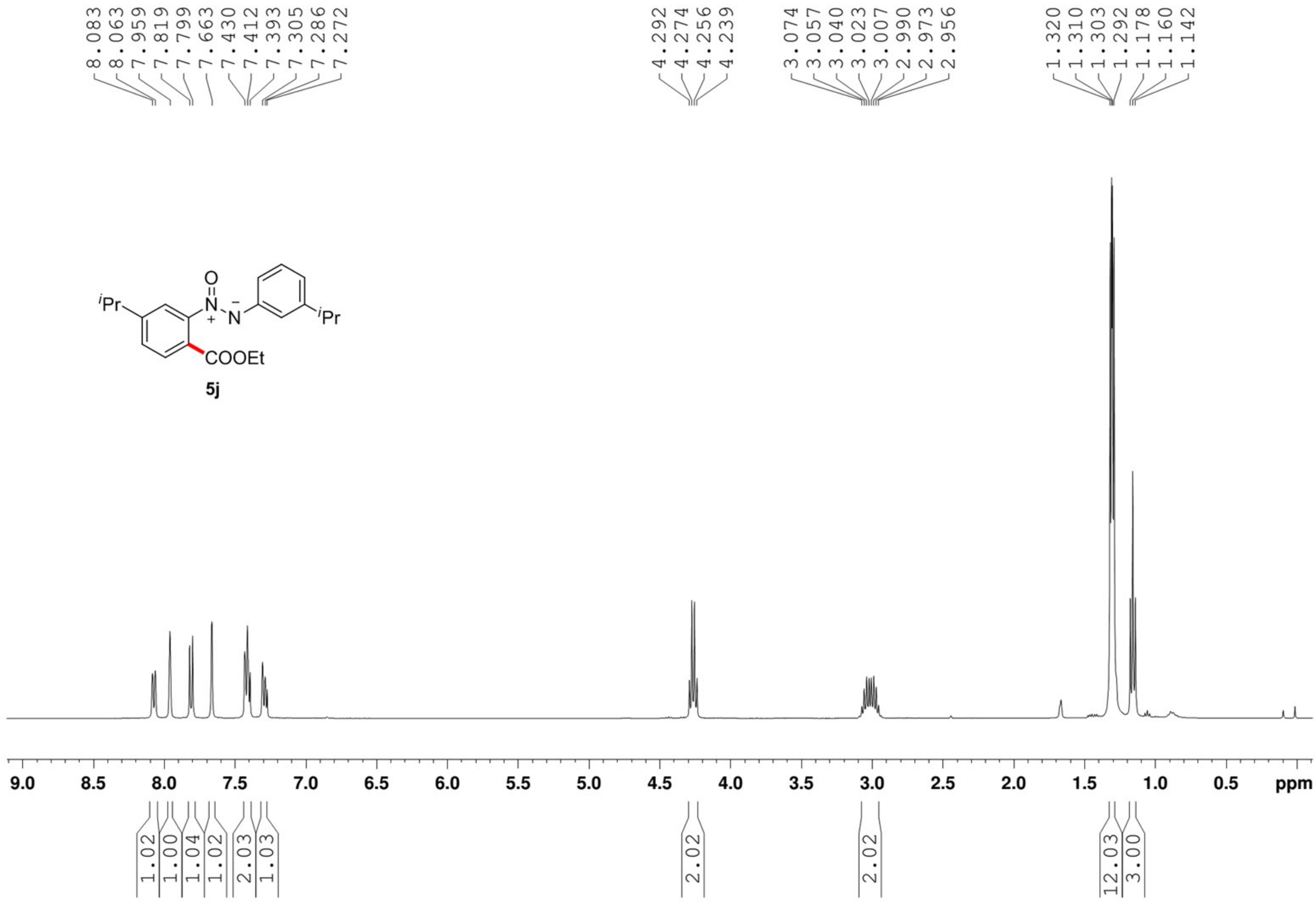
131.237
130.061
127.097
124.872
123.986
77.236
76.918
76.601
62.131
61.806
61.140
14.171
14.121
13.848
COOEt
EtOOC
COOEt
5h
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



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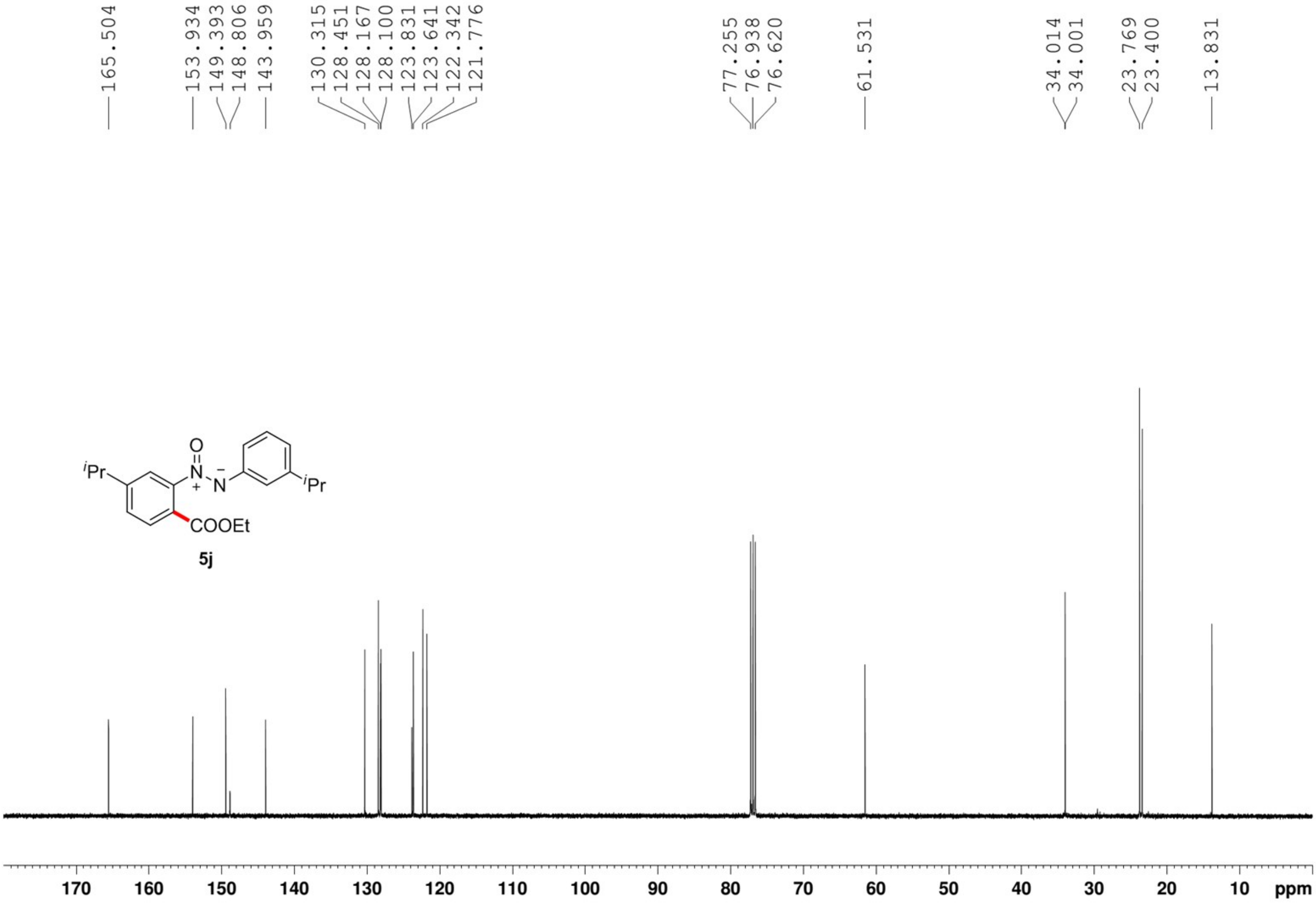
8.063
7.959
7.819
7.799
7.663
7.430
7.412
7.393
7.305
7.286
7.272
4.292
4.274
4.256
4.239
3.074
3.057
3.040
3.023
3.007
2.990
2.973
2.956
1.320
1.310
1.303
1.292
1.178
1.160
1.142
iPr
O
N
+
N
–
iPr
COOEt
5j
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02
1.00
1.04
1.02
2.03
1.03
2.02
2.02
12.03
3.00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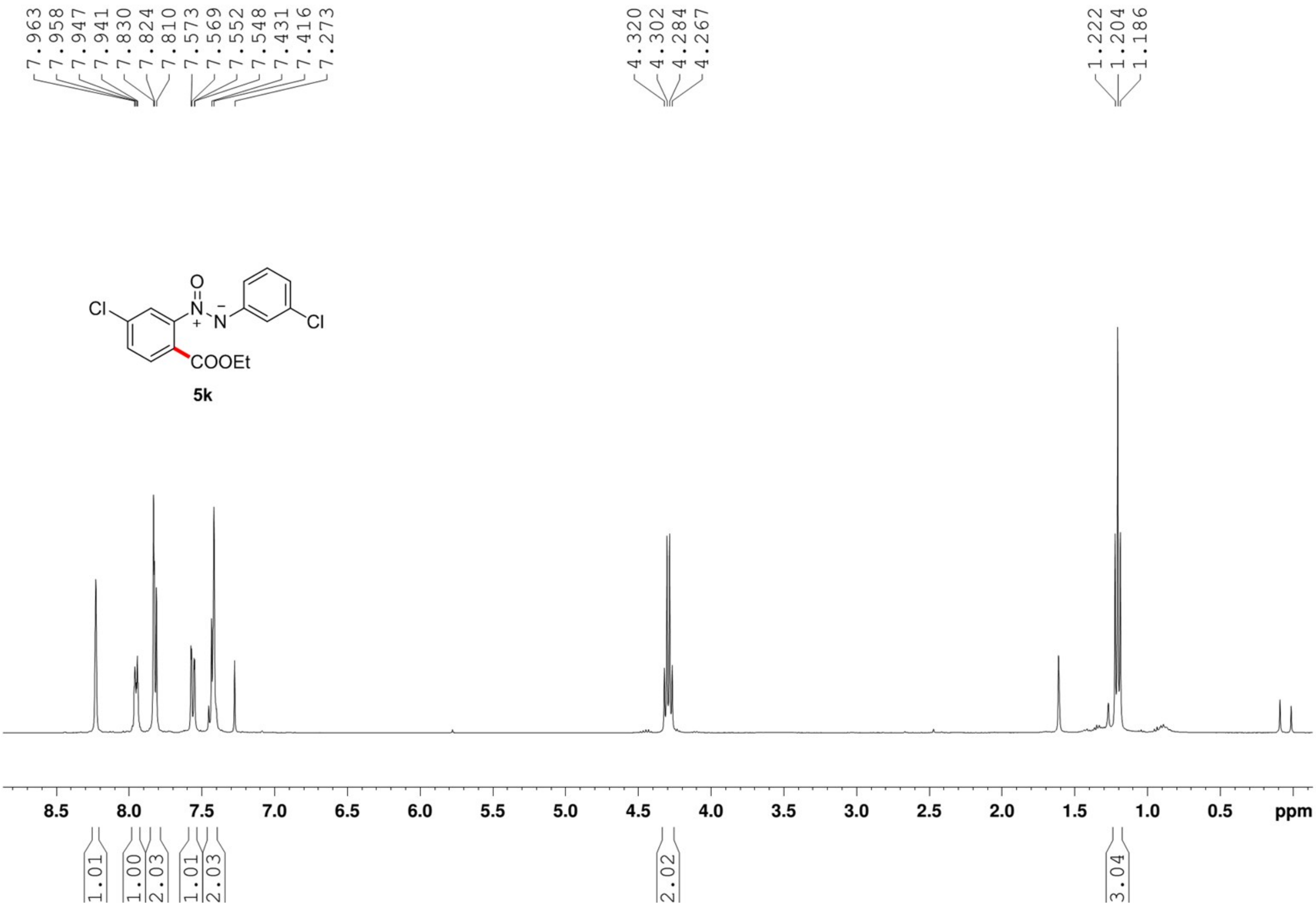
153.934
149.393
148.806
143.959
130.315
128.451
128.167
128.100
123.831
123.641
122.342
121.776
77.255
76.938
76.620
61.531
34.014
34.001
23.769
23.400
13.831
iPr
O
N
+
N
–
iPr
COOEt
5j
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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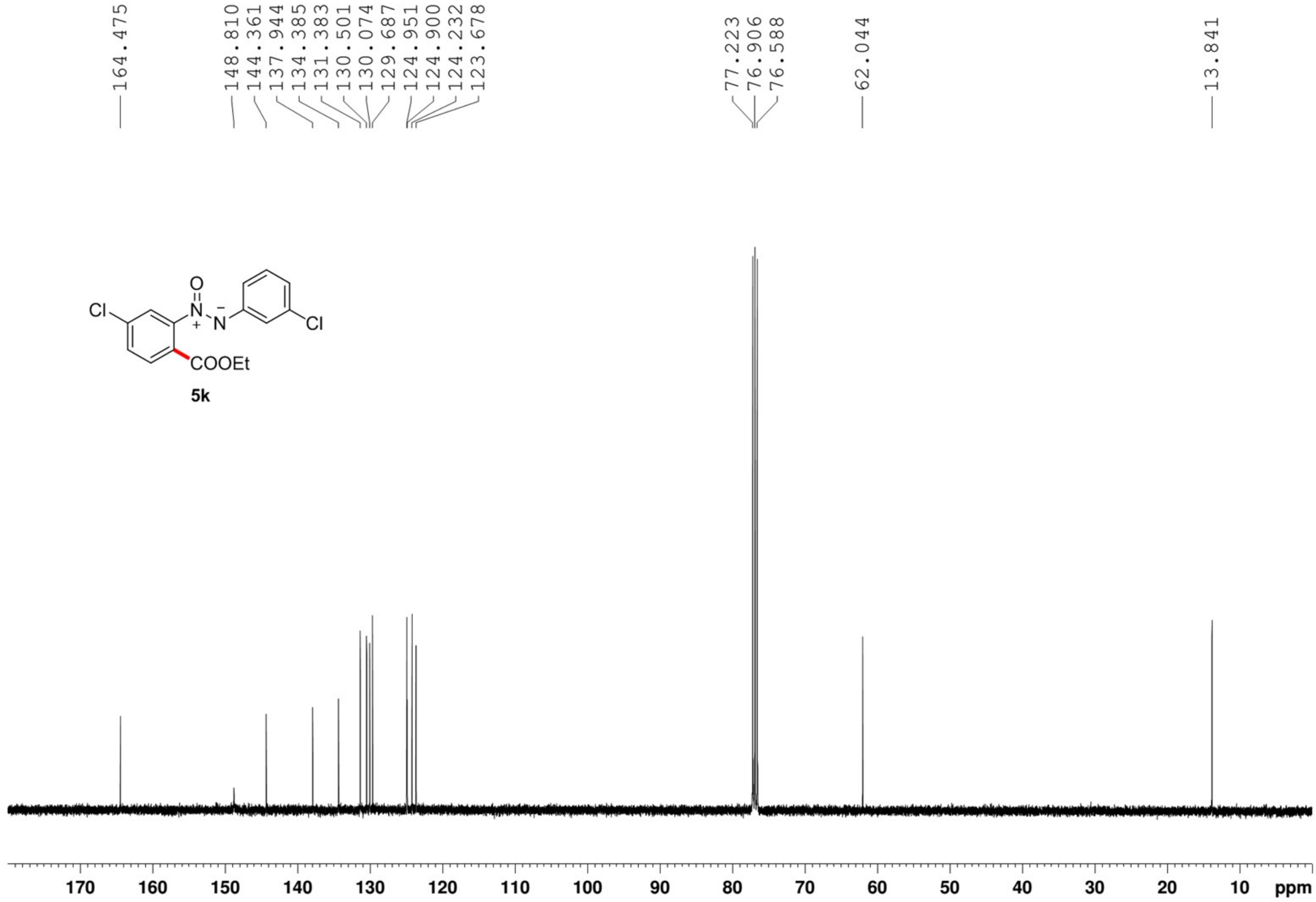
7.958
7.947
7.941
7.830
7.824
7.810
7.573
7.569
7.552
7.548
7.431
7.416
7.273
4.320
4.302
4.284
4.267
1.222
1.204
1.186
Cl
O
N
N
Cl
COOEt
5k
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.01
1.00
2.03
1.01
2.03
2.02
3.04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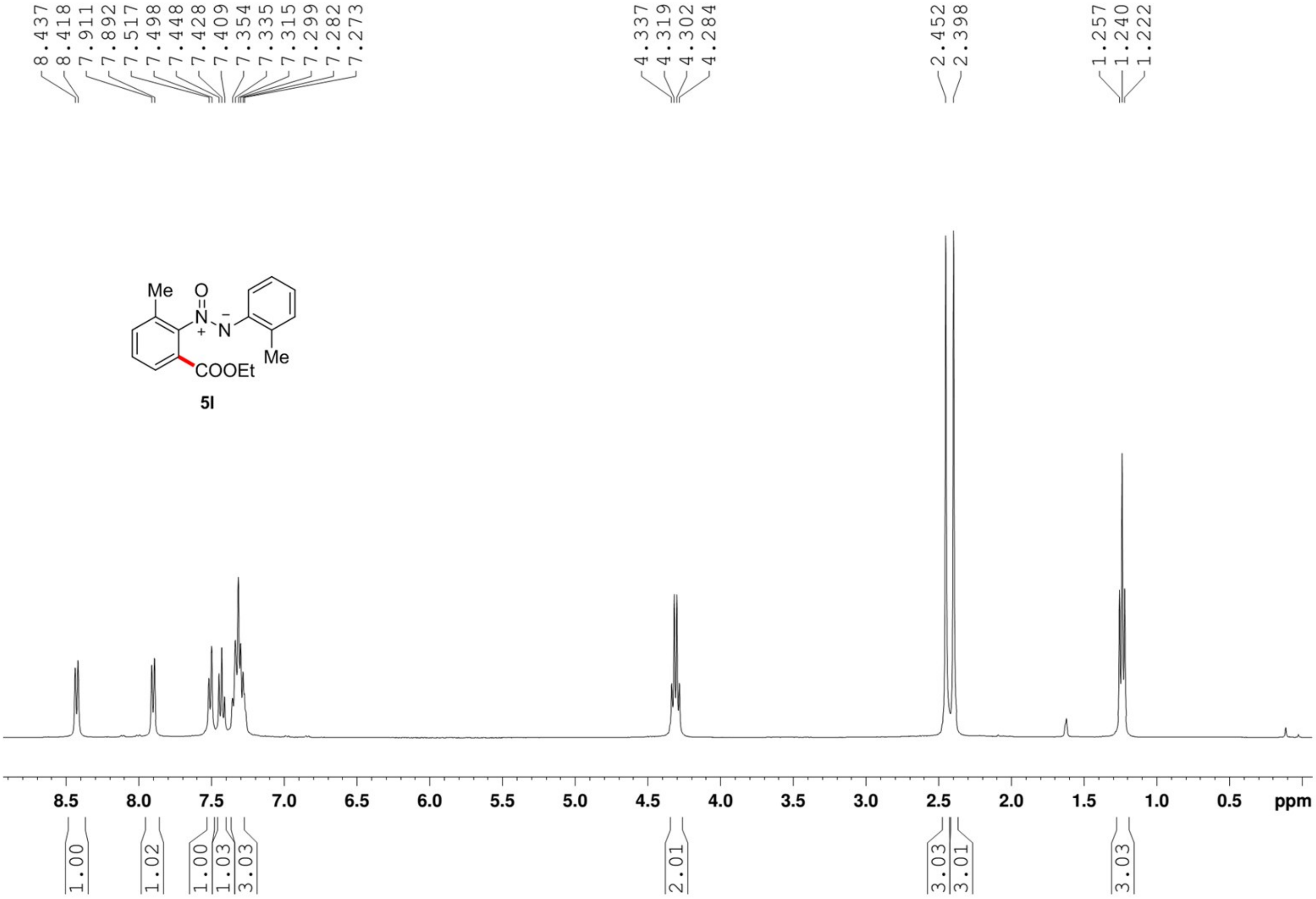
144.361
137.944
134.385
131.383
130.501
130.074
129.687
124.951
124.900
124.232
123.678
77.223
76.906
76.588
62.044
13.841
Cl
O
N
N
Cl
COOEt
5k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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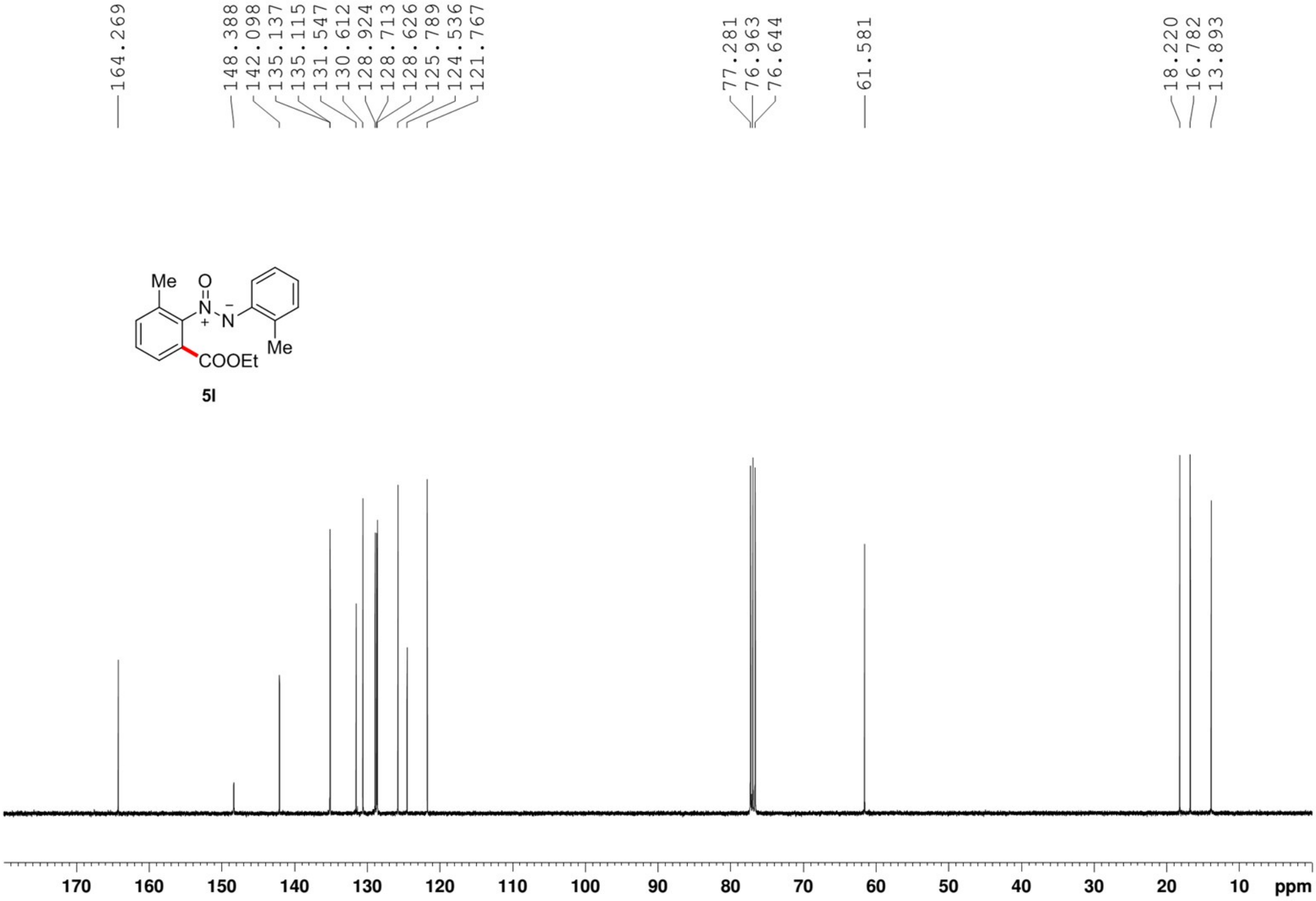
8.418
7.911
7.892
7.517
7.498
7.448
7.428
7.409
7.354
7.335
7.315
7.299
7.282
7.273
4.337
4.319
4.302
4.284
2.452
2.398
1.257
1.240
1.222
Me
O
N
N
Me
COOEt
5l
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.02
1.00
1.03
3.03
2.01
3.03
3.01
3.03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135.137
135.115
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130.612
128.924
128.713
128.626
125.789
124.536
121.767
77.281
76.963
76.644
61.581
18.220
16.782
13.893
Me
O
N
N
Me
COOEt
5l
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm