Silica-zinc chloride (SiO_2-$ZnCl_2$) catalyzed Michael addition reaction of active methylene compounds to β-nitrostyrenes: Synthesis of functionalized pyrazole derivatives

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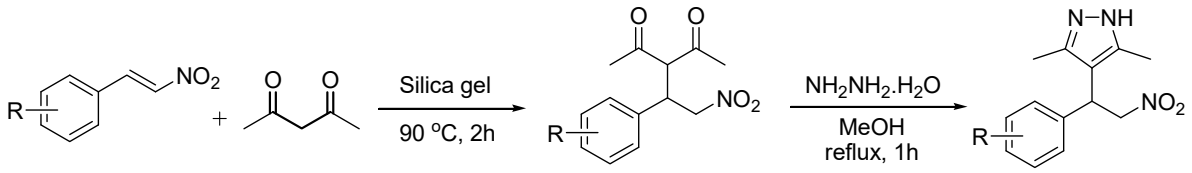
A B S T R A C T

Under solvent-free conditions, a simple and efficient procedure has been developed for the Michael addition reaction of active methylene compounds to β-nitrostyrenes. The resulting Michael adducts are efficiently converted into functionalized pyrazole derivatives with hydrazine hydrate in excellent yields. All the compounds were well characterized by spectroscopic techniques.

Silica gel mediated Michael addition reaction of active methylene compounds to β-nitrostyrenes: Synthesis of functionalized pyrazolederivatives

Abstract:



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2
3
4
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6
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8
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3.00
2.95
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4.233
4.239
4.245
4.248
4.254
4.260
4.270
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4.609
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4.633
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7.197
7.266
7.290
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7.307
7.318
7.321
7.329
7.332
7.346
0.000
O
O
NO2

201.76
201.08
136.03
129.31
128.52
127.95
78.15
77.40
77.08
76.77
70.61
42.80
30.45
29.65
O
O
NO2
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

ppm
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1
2
3
4
5
6
7
8
9
3.09
3.02
3.04
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0.97
2.00
2.07
2.05
0.000
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2.293
2.582
2.597
2.612
2.628
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4.213
4.216
4.223
4.229
4.238
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4.597
4.607
4.622
7.078
7.094
7.139
7.155
7.266
NO2
O
O

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201.26
144.51
133.08
128.73
127.85
78.31
77.42
77.10
76.78
70.72
42.48
30.43
29.57
28.36
15.17
NO2
O
O
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4
5
6
7
8
9
2.01
2.05
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3.02
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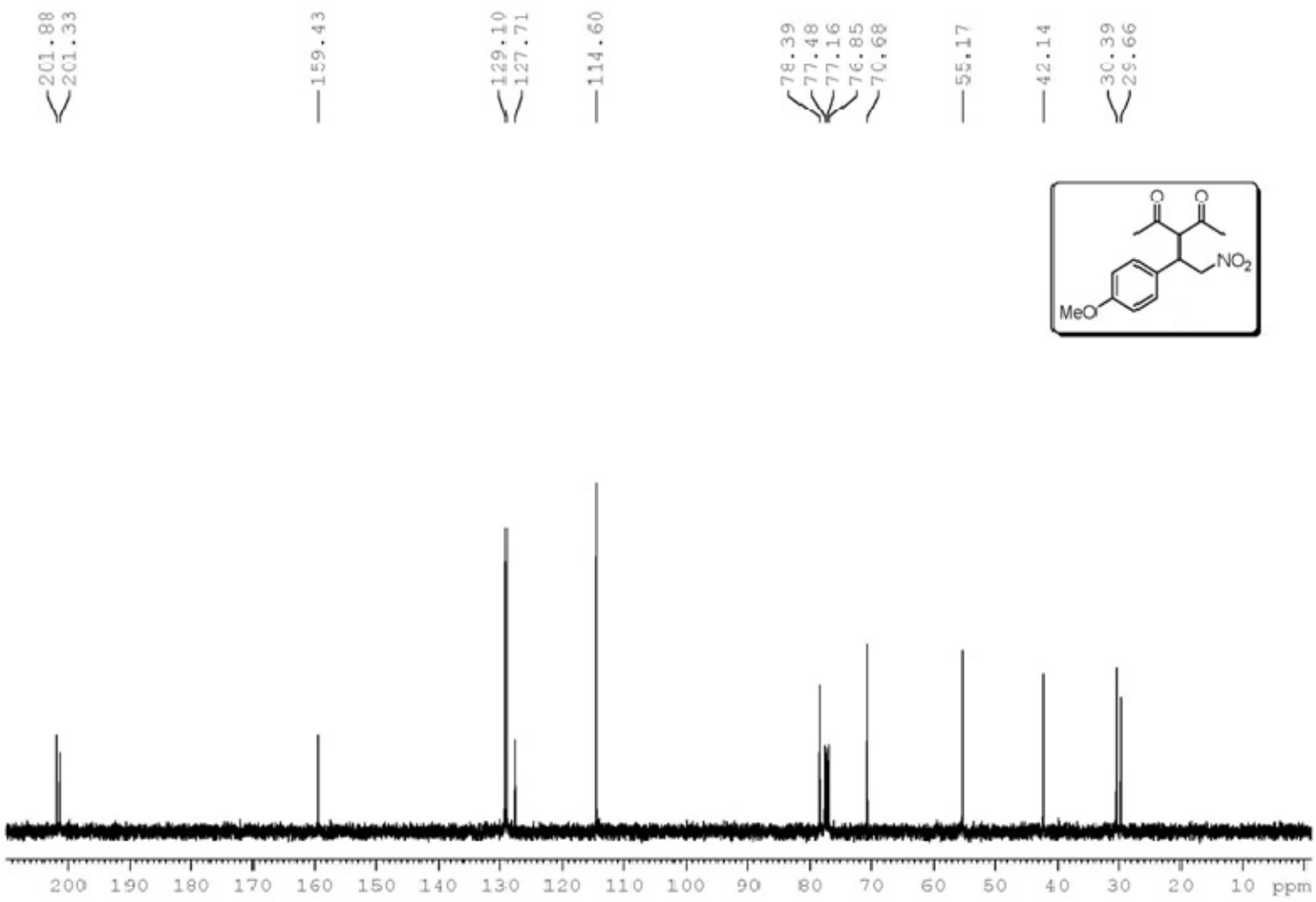
MeO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42.14
30.39
29.66
O
O
NO2
MeO
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ppm
0
1
2
3
4
5
6
7
8
9
3.00
3.01
3.06
1.01
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1.01
1.05
0.99
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4.504
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4.576
4.591
4.600
4.611
4.765
4.781
4.789
4.805
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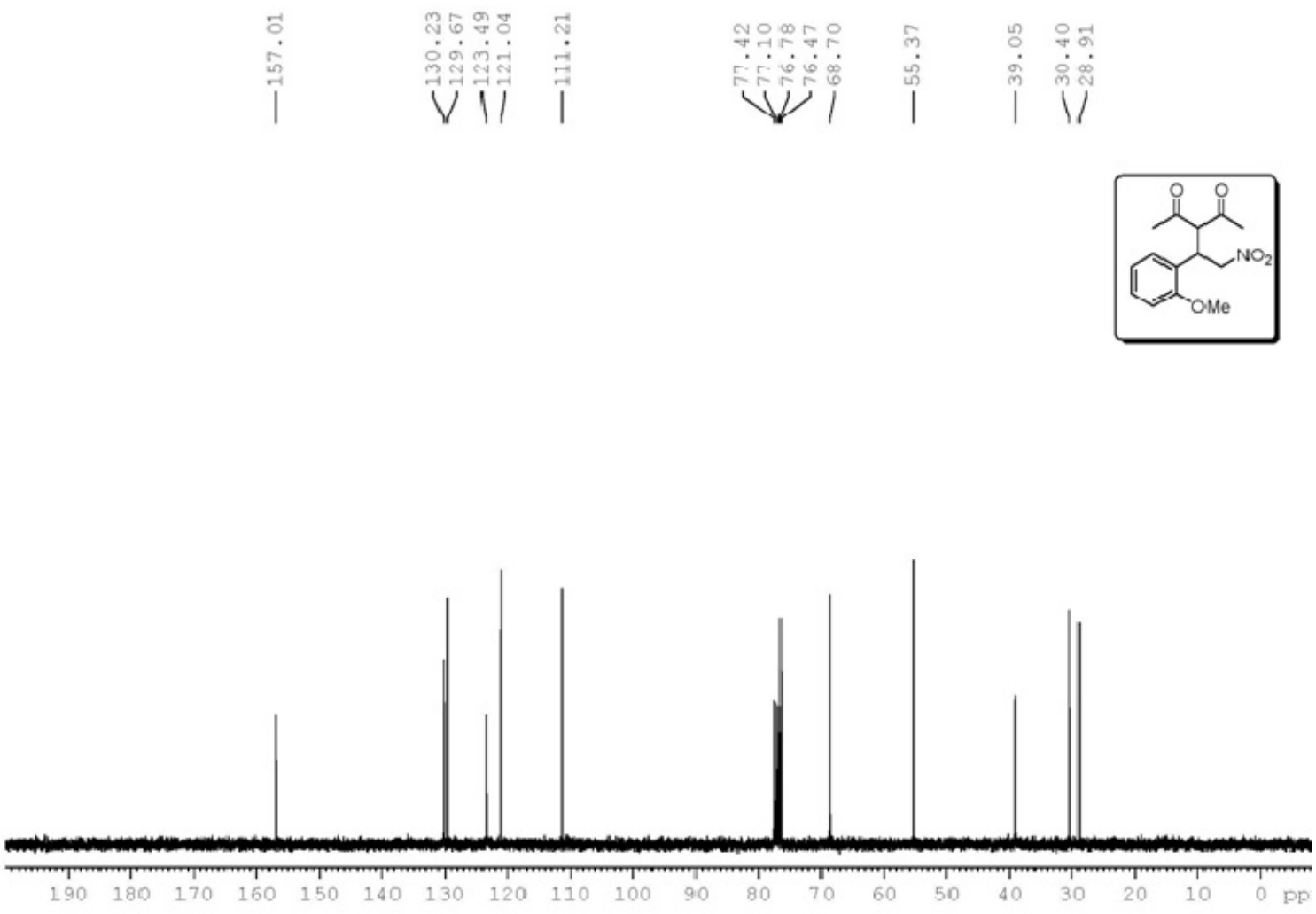
OMe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76.47
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39.05
30.40
28.91
O
O
NO2
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

ppm
0
1
2
3
4
5
6
7
8
9
1.93
1.94
2.15
4.22
5.28
1.00
1.98
1.01

Ph
NO2
Ph
O
O

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1.254

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4.617
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5.004
5.833
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7.226
7.228
7.242
7.345
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7.361
7.377
7.392
7.409
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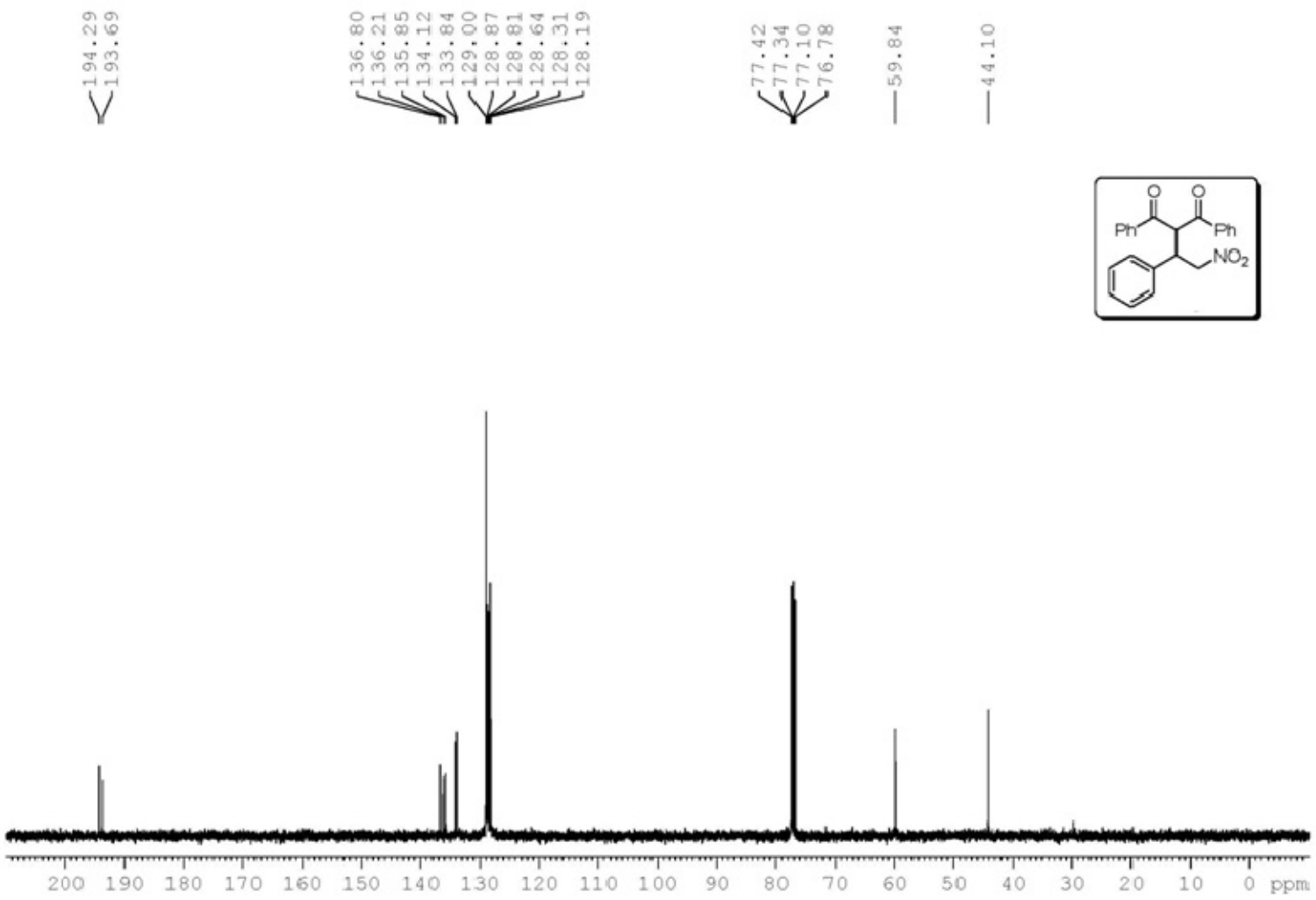
7.775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128.81
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128.19
77.42
77.34
77.10
76.78
59.84
44.10
Ph
Ph
NO2
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ppm
0
1
2
3
4
5
6
7
8
9
3.05
2.06
1.01
1.96
1.00
2.01
2.21
4.22
2.24
1.99
2.03
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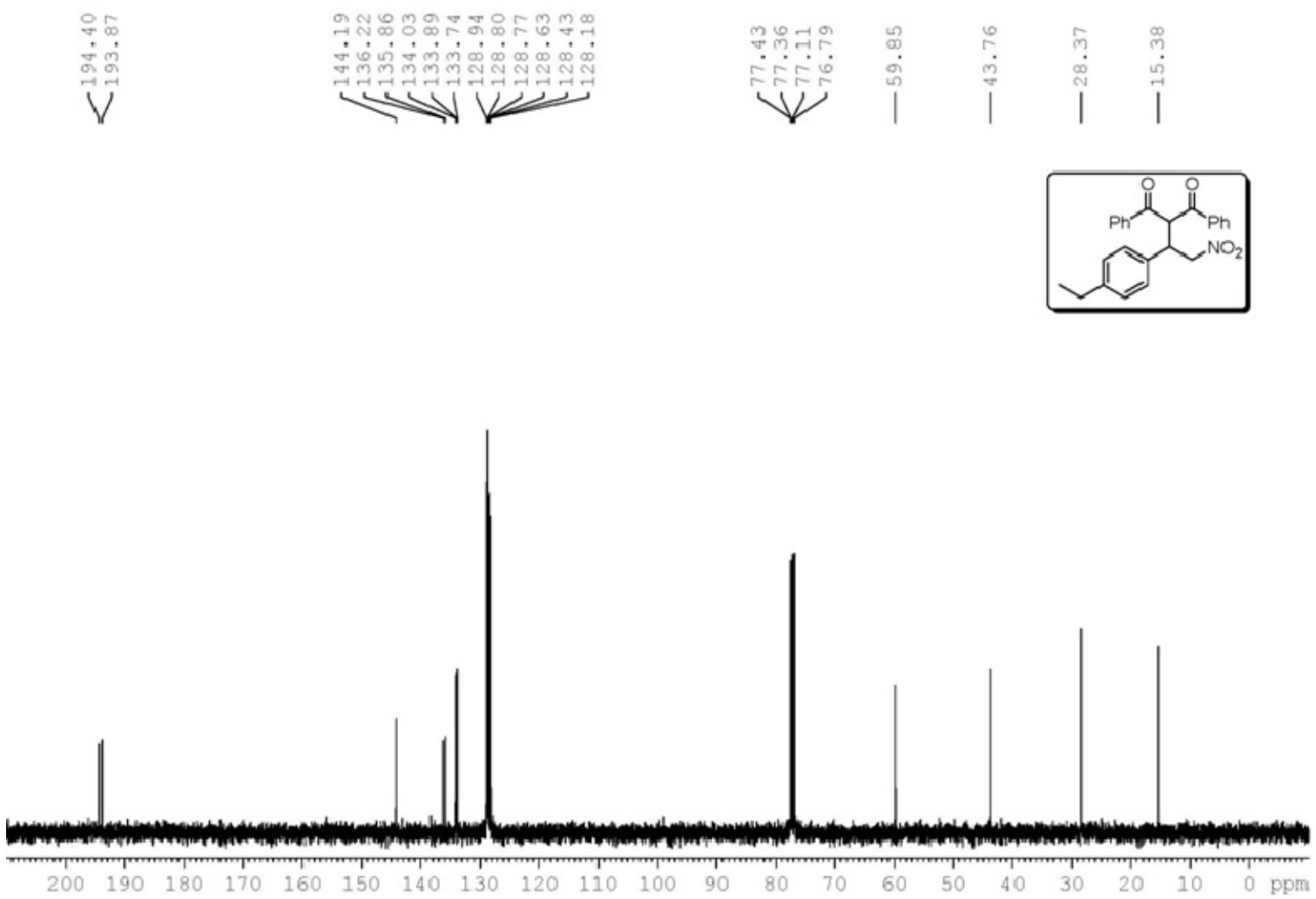
Ph
NO2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77.36
77.11
76.79
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Ph
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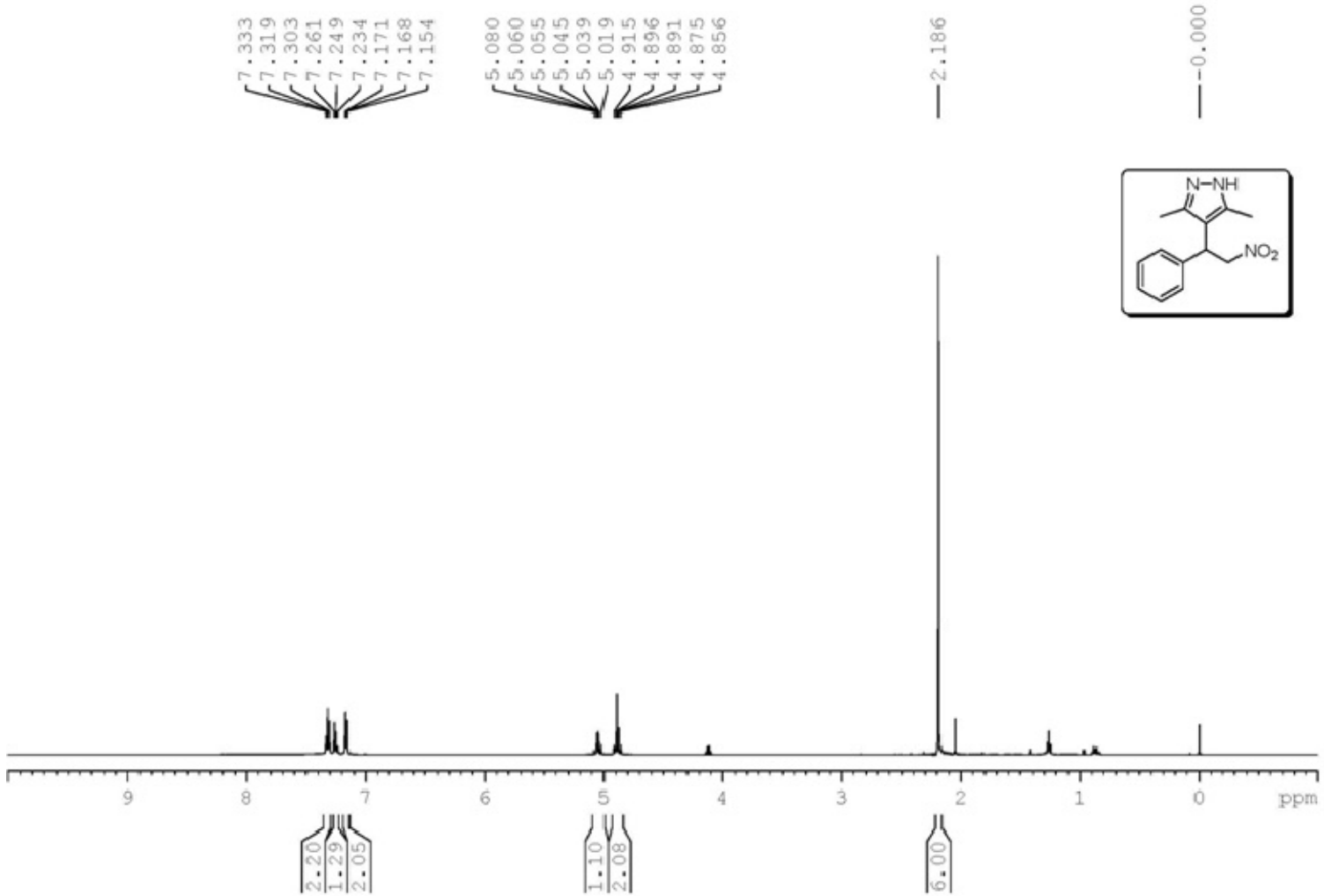
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5.039
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4.896
4.891
4.875
4.856
2.186
-0.000
N-NH
NO2
9
8
7
6
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4
3
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ppm
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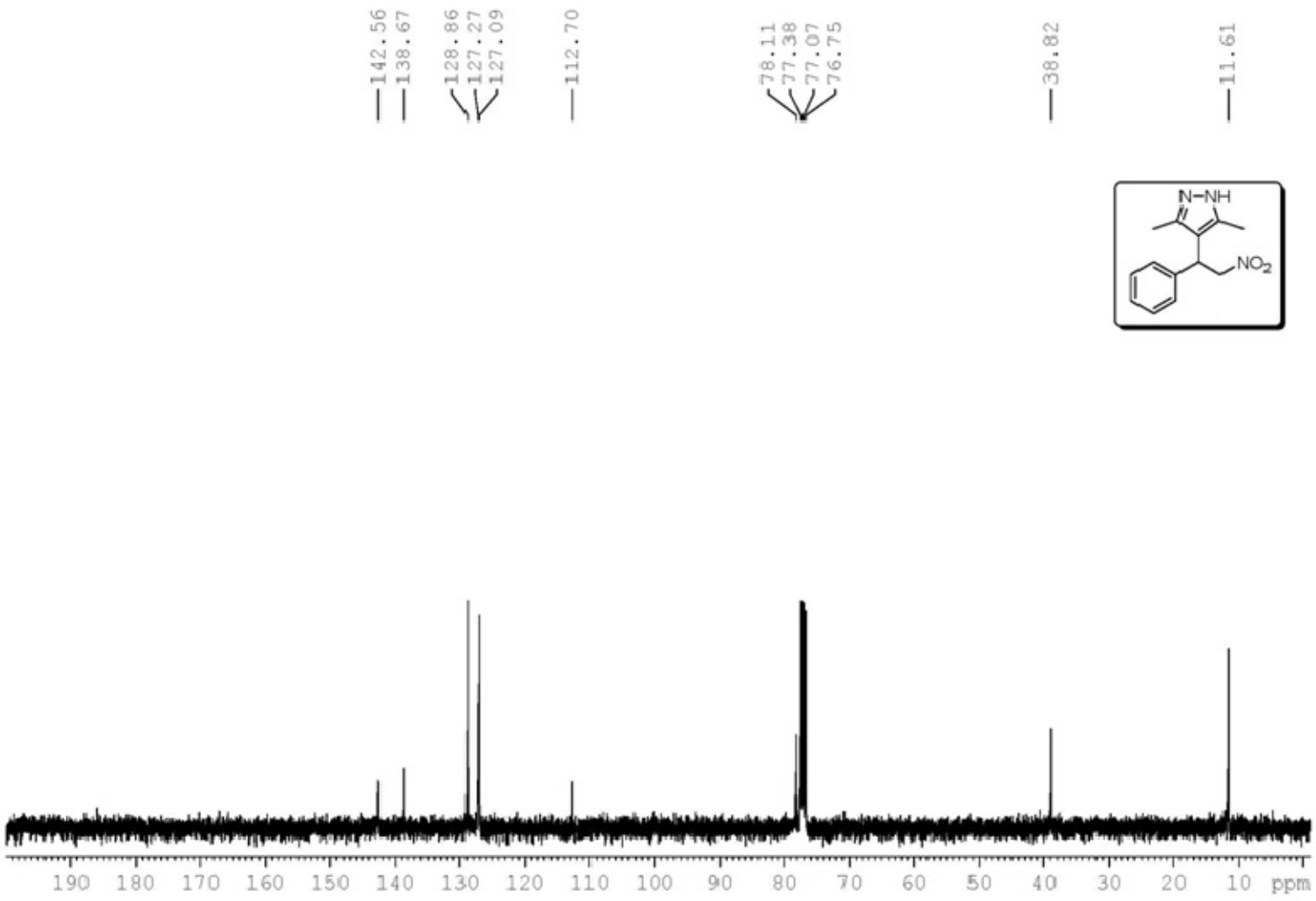
1.29
2.05
1.10
2.08
6.00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78.11
77.38
77.07
76.75
38.82
11.61
N–NH
NO2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

ppm
0
1
2
3
4
5
6
7
8
9
3.07
6.00
2.07
1.93
0.97
2.12
2.11
-0.000
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1.226
2.195
2.593
2.608
2.623
2.639
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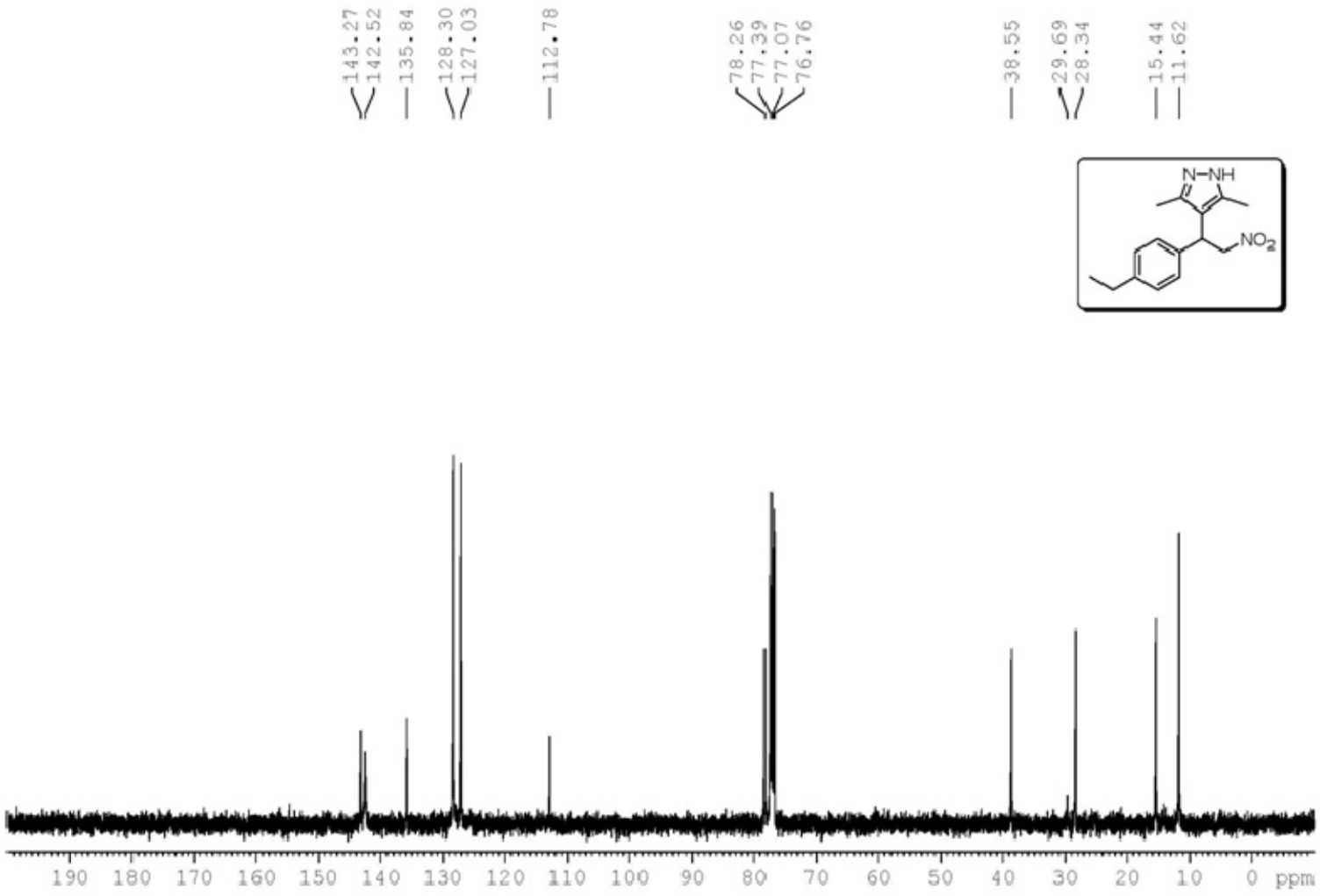
NO2
HN
N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127.03
112.78
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77.07
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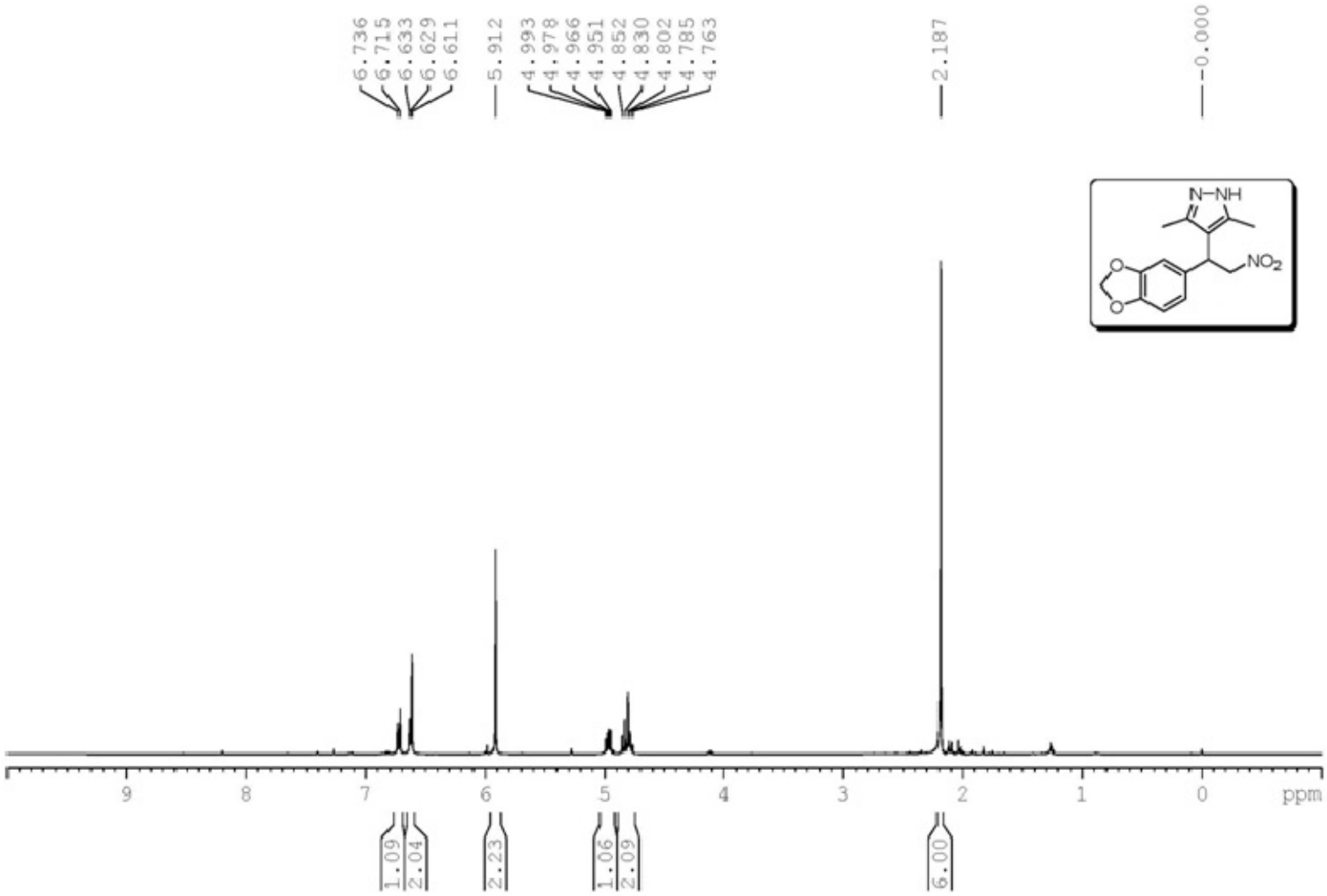
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4.802
4.785
4.763
2.187
0.000
N–NH
NO2
9
8
7
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3
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ppm
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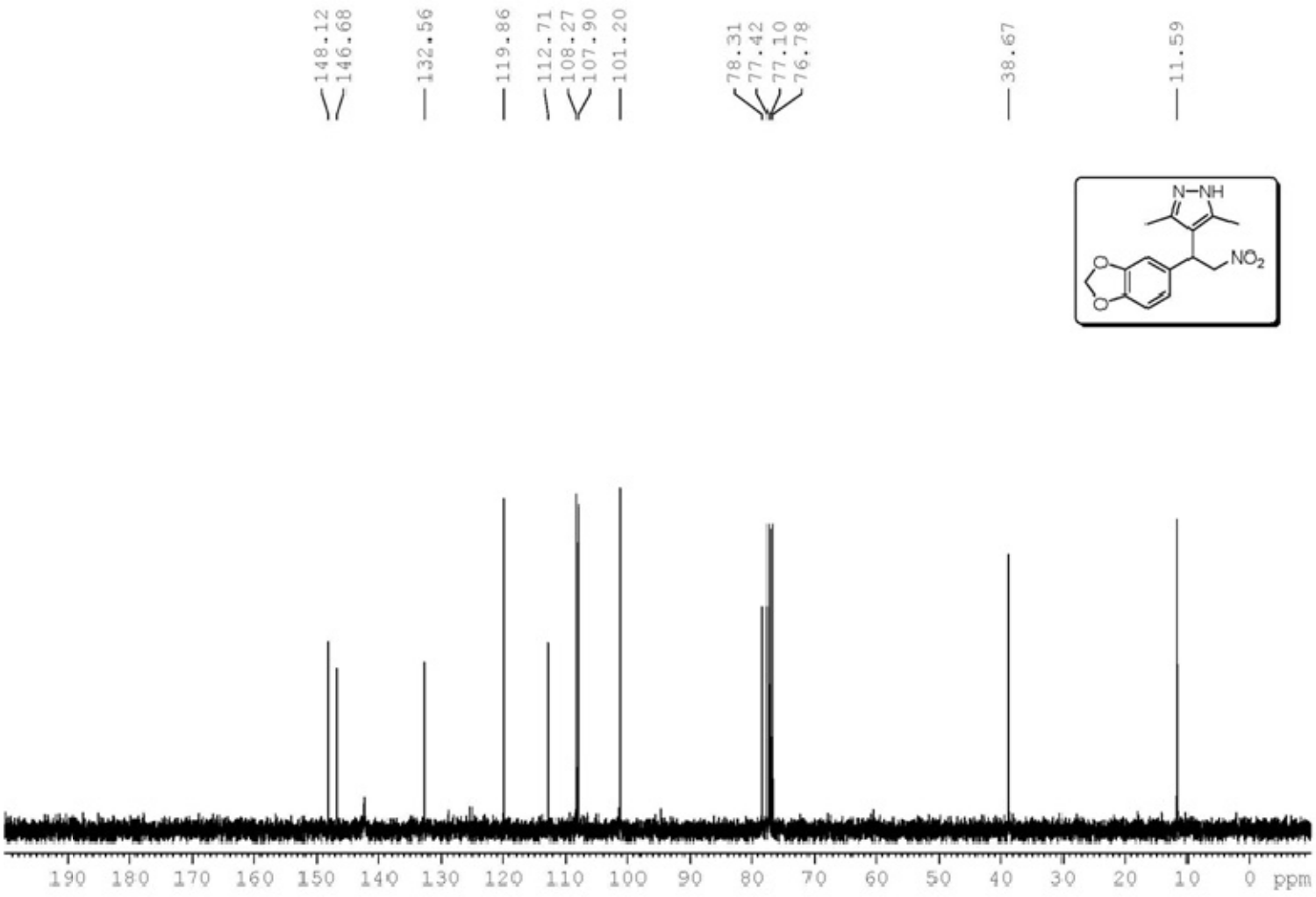
1.06
2.09
6.00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77.42
77.10
76.78
38.67
11.59
N–NH
NO2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

7.079
7.057
6.837
6.815
5.024
4.997
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4.957
4.859
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4.836
4.819
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3.745
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-0.001
6.06
3.14
1.97
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ppm
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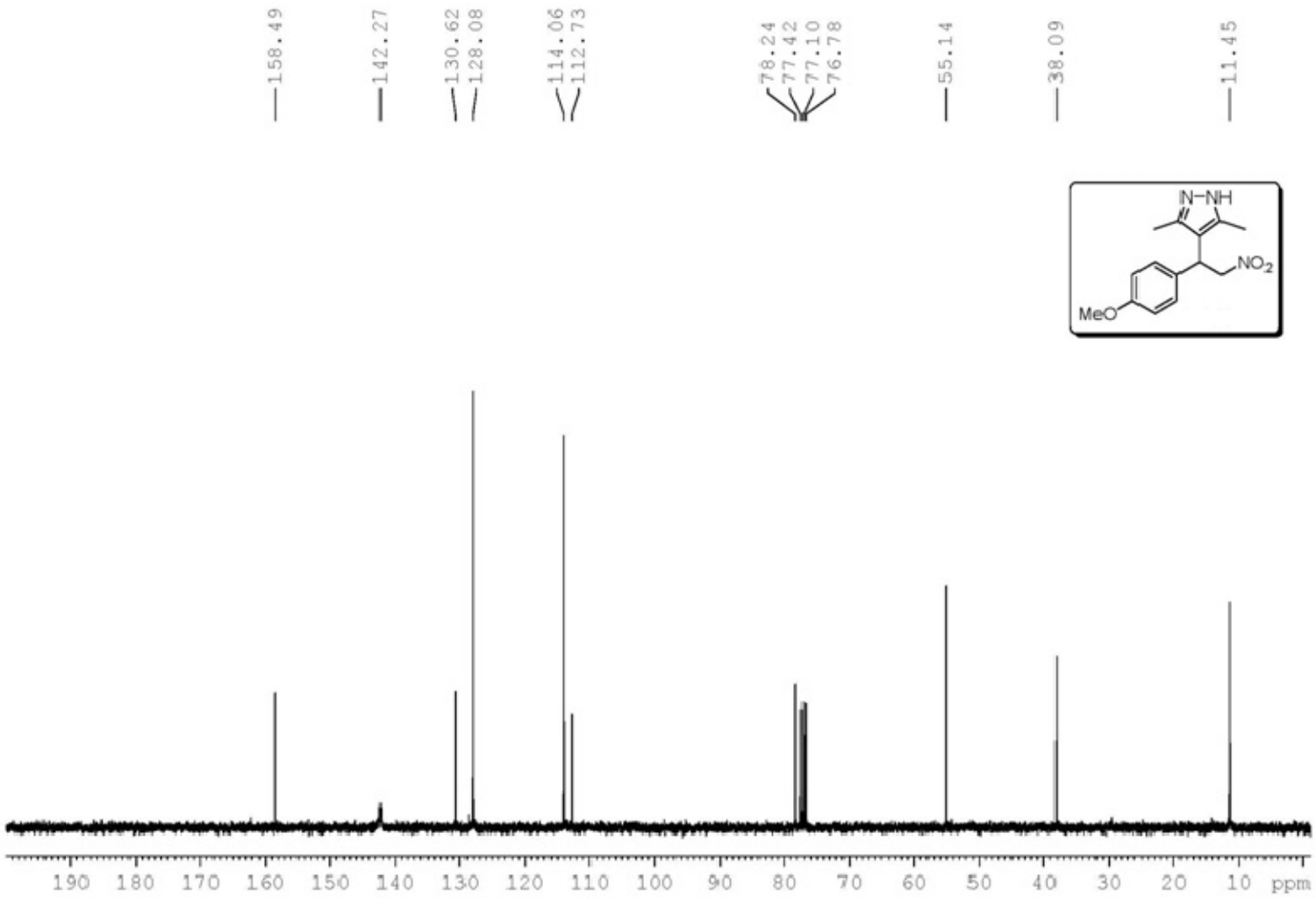
7
8
9
HN–N
NO2
MeO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114.06
112.73
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55.14
38.09
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NO2
MeO
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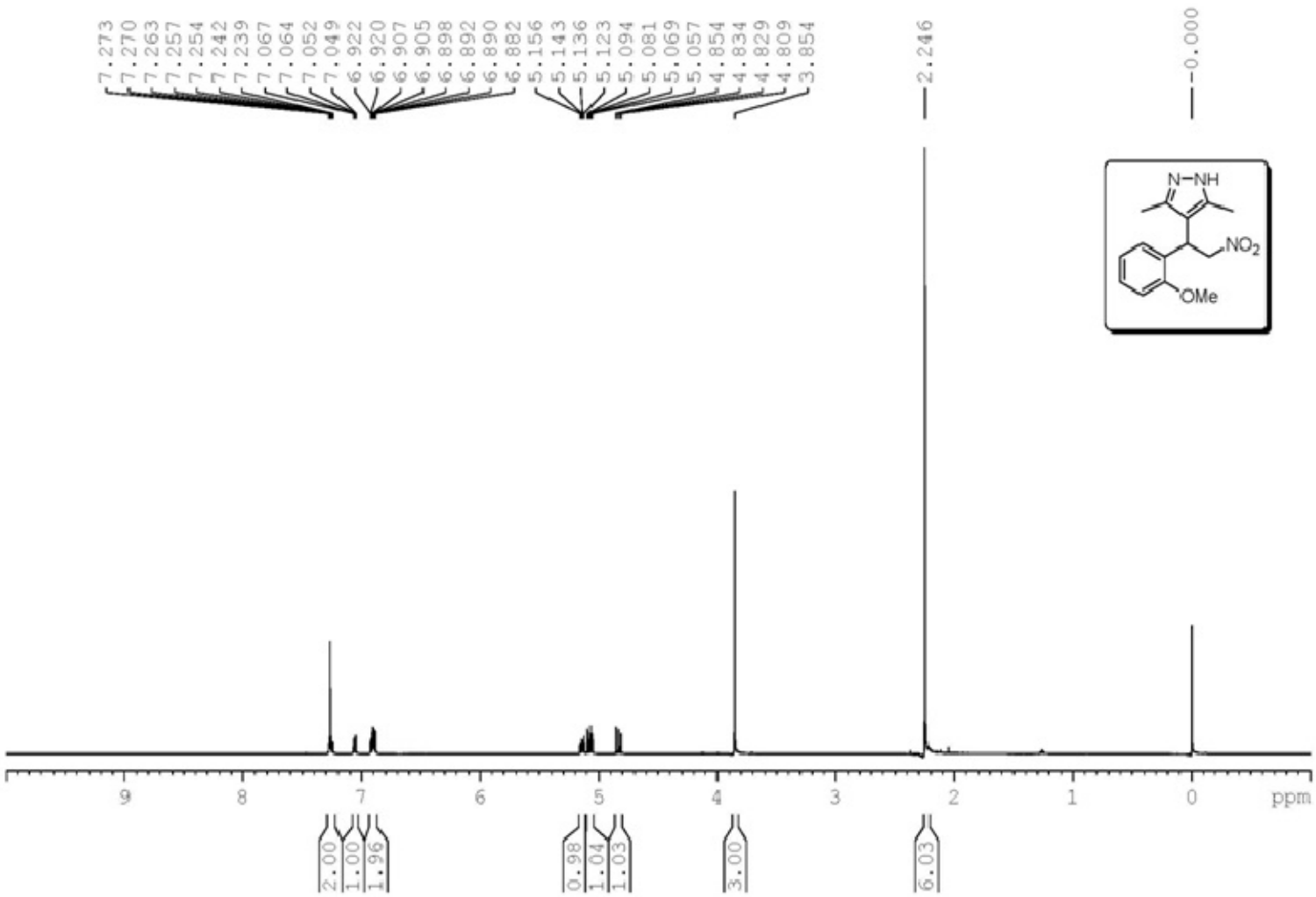
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6.907
6.905
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6.890
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5.156
5.143
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5.123
5.094
5.081
5.069
5.057
4.854
4.834
4.829
4.809
3.854
2.246
0.000
N–NH
NO2
OMe
9
8
7
6
5
4
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ppm
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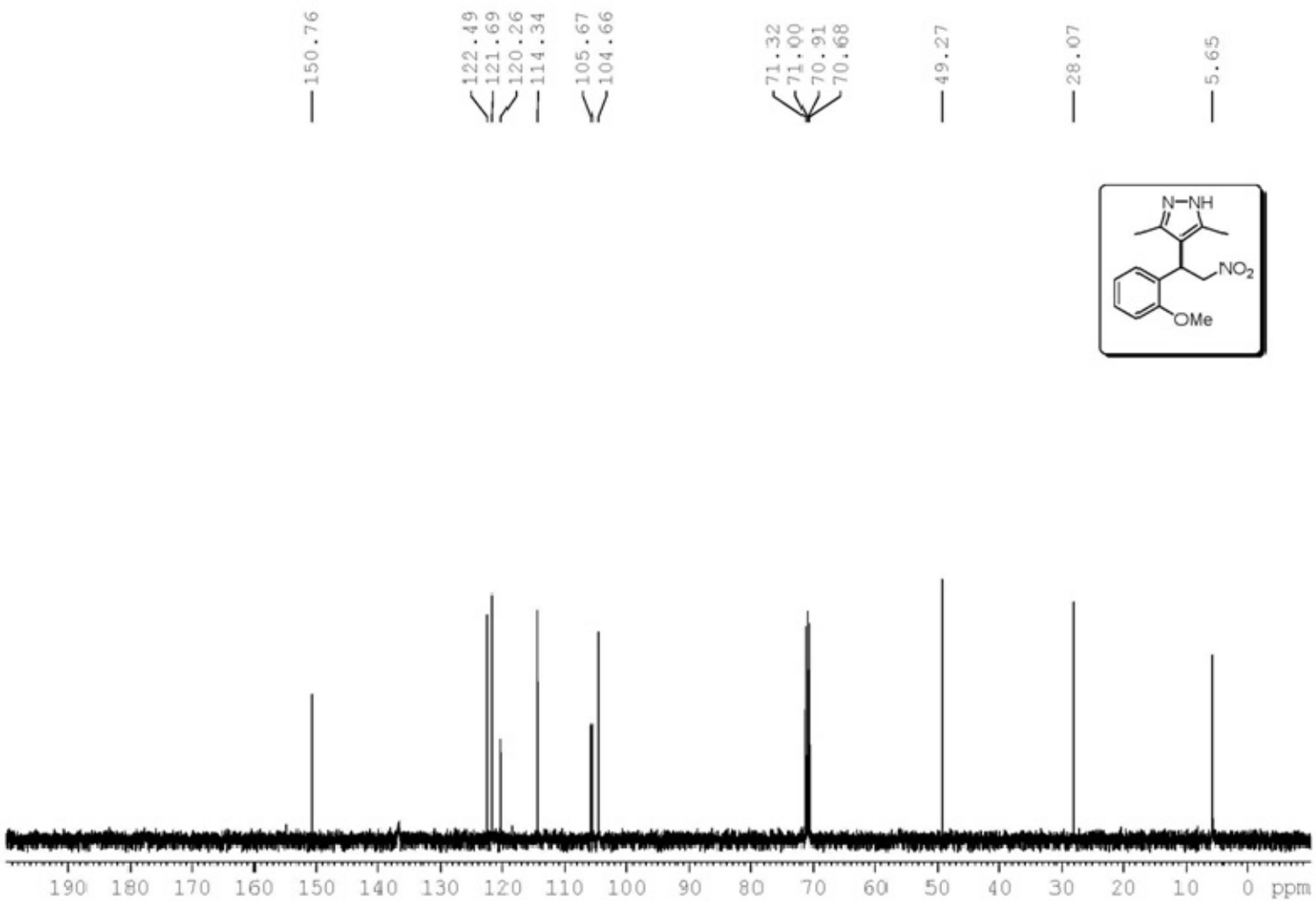
6.03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121.69
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105.67
104.66
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70.91
70.68
49.27
28.07
5.65
N–NH
NO2
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm