Supporting Information

A domino N_2-extrusion-cyclization of alkynylarylketone derivatives for the synthesis of indoloquinolines and carbocycle-fused quinolines

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Charnsak Thongsornkleeb[†‡] and Somsak Ruchirawat[†‡]

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[‡]Laboratory of Medicinal Chemistry, Chulabhorn Research Institute, 54 Kamphaeng Phet 6, Laksi, Bangkok 10210, Thailand

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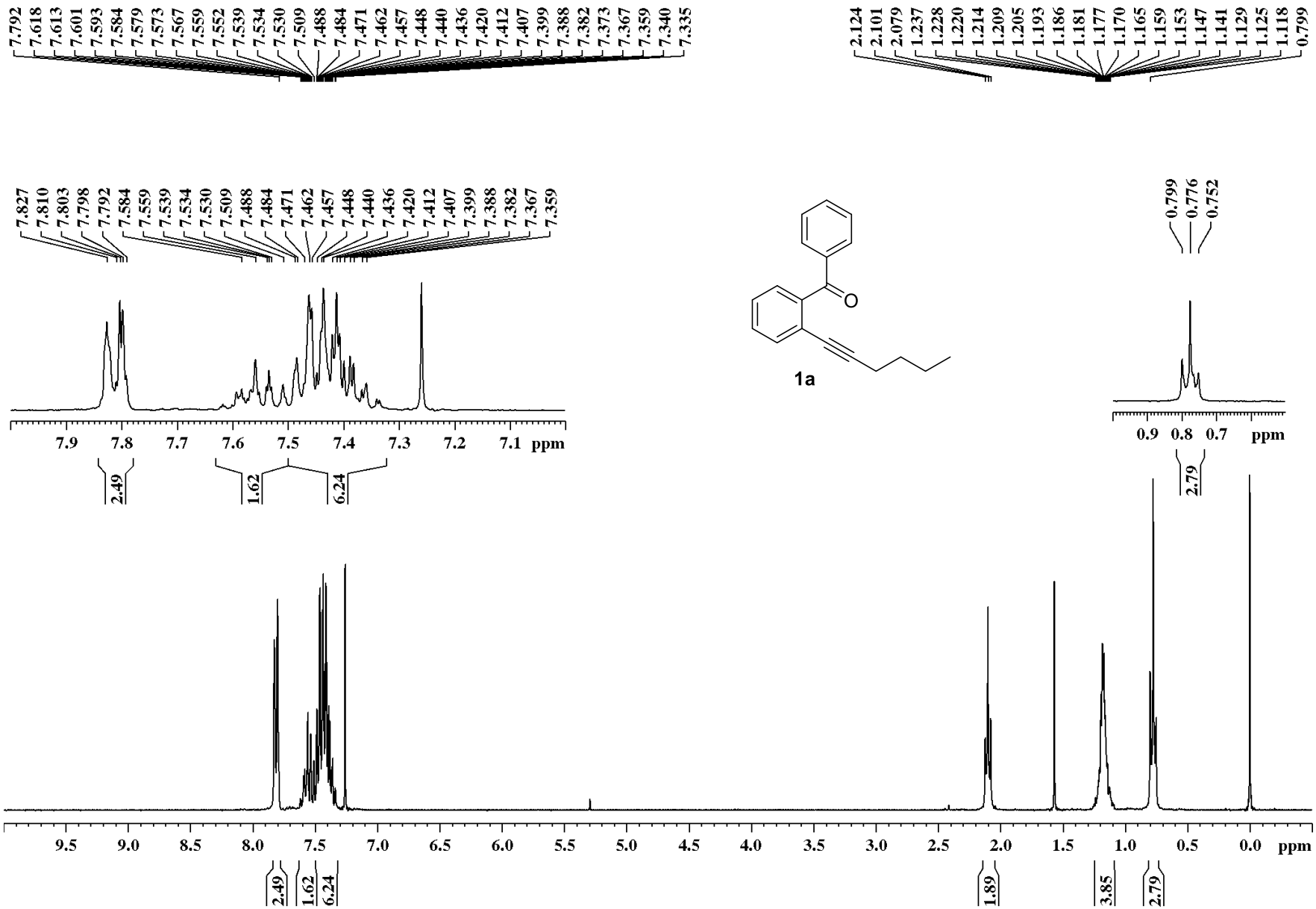
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2.124 2.101 2.079 1.237 1.228 1.220 1.214 1.209 1.205 1.193 1.186 1.181 1.177 1.170 1.165 1.159 1.153 1.147 1.141 1.129 1.125 1.118 0.799
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0.799 0.776 0.752
O
1a
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2.49 1.62 6.24
0.9 0.8 0.7 ppm
2.79
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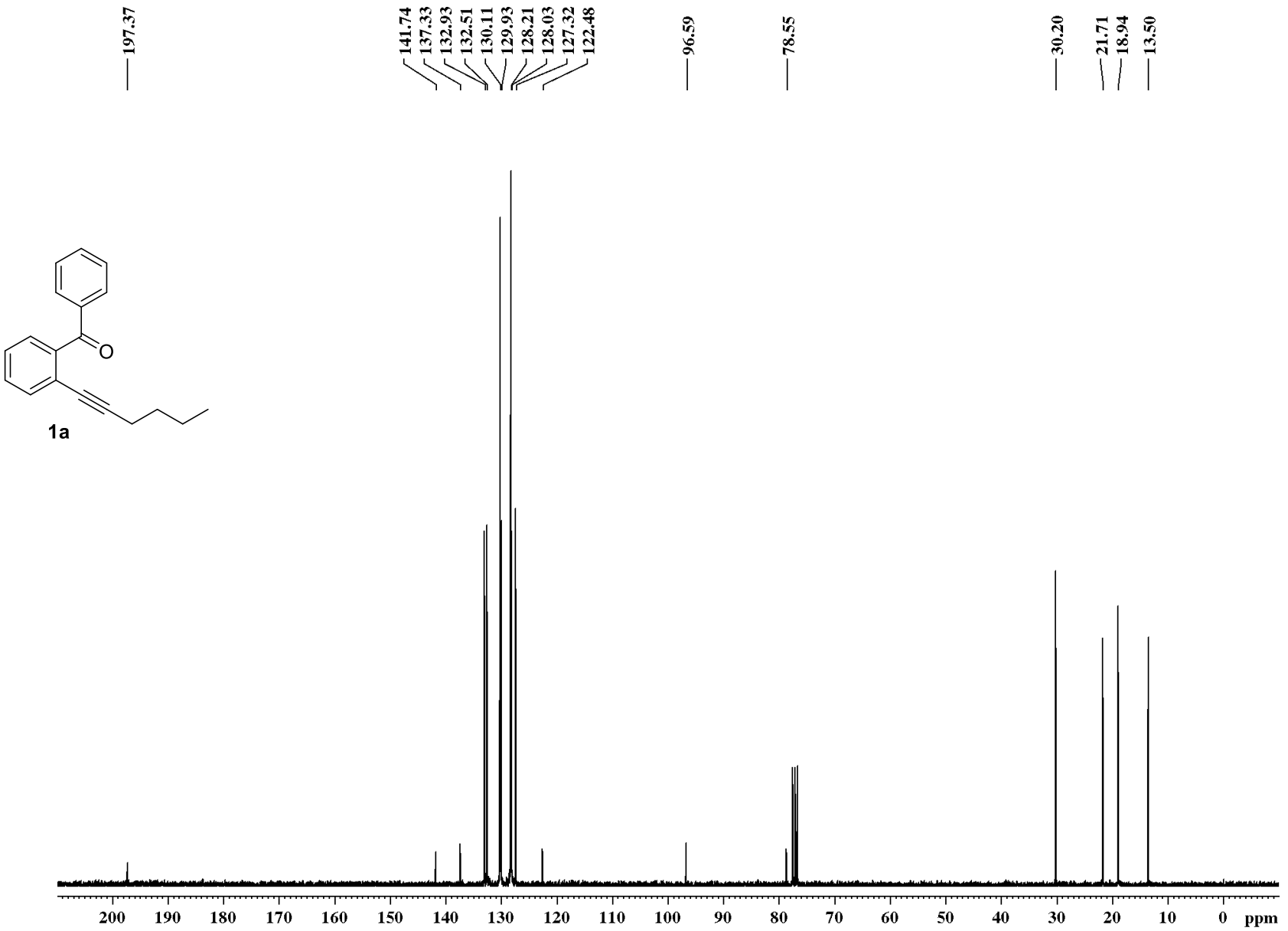
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13.50
O
1a
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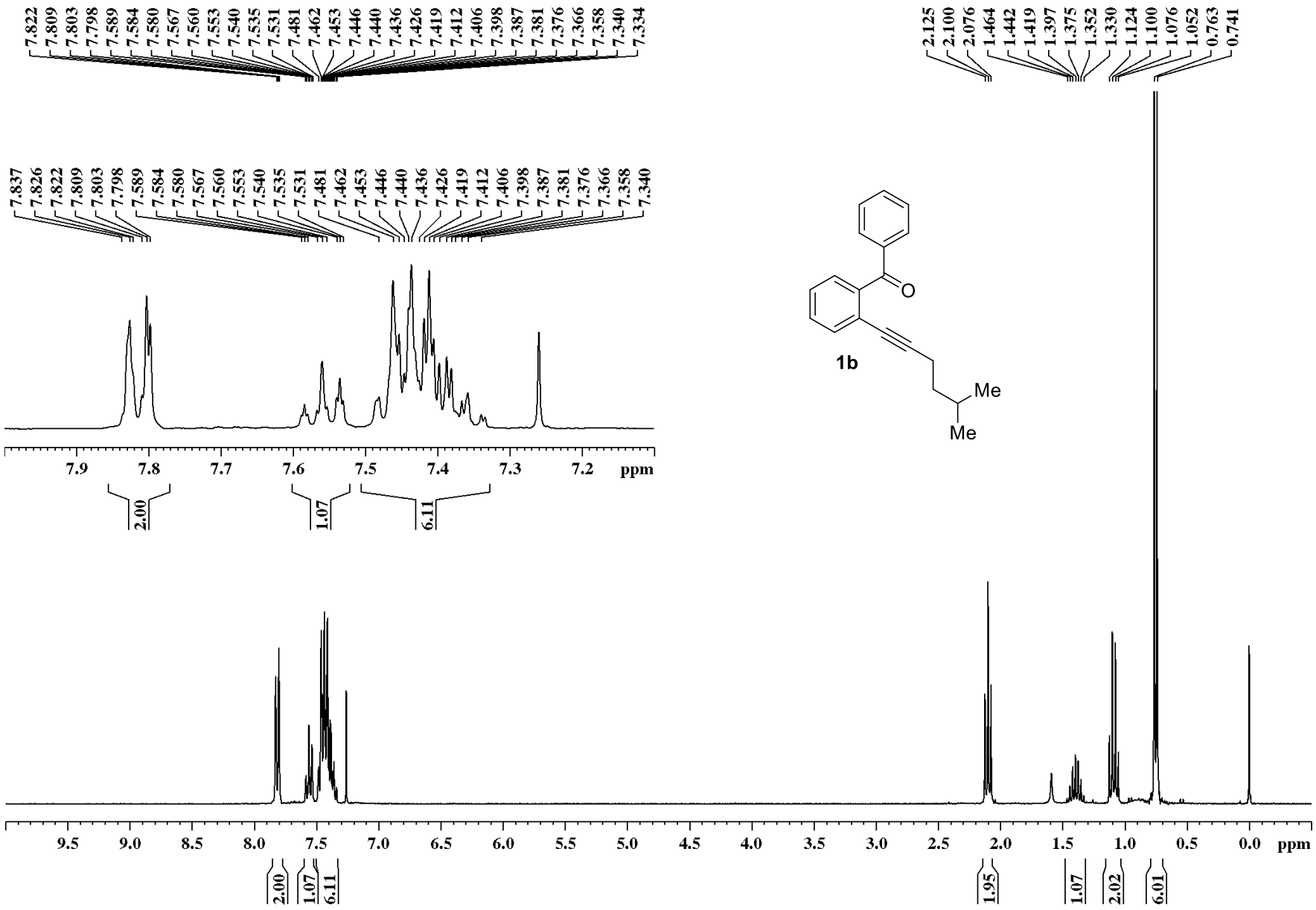
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7.376
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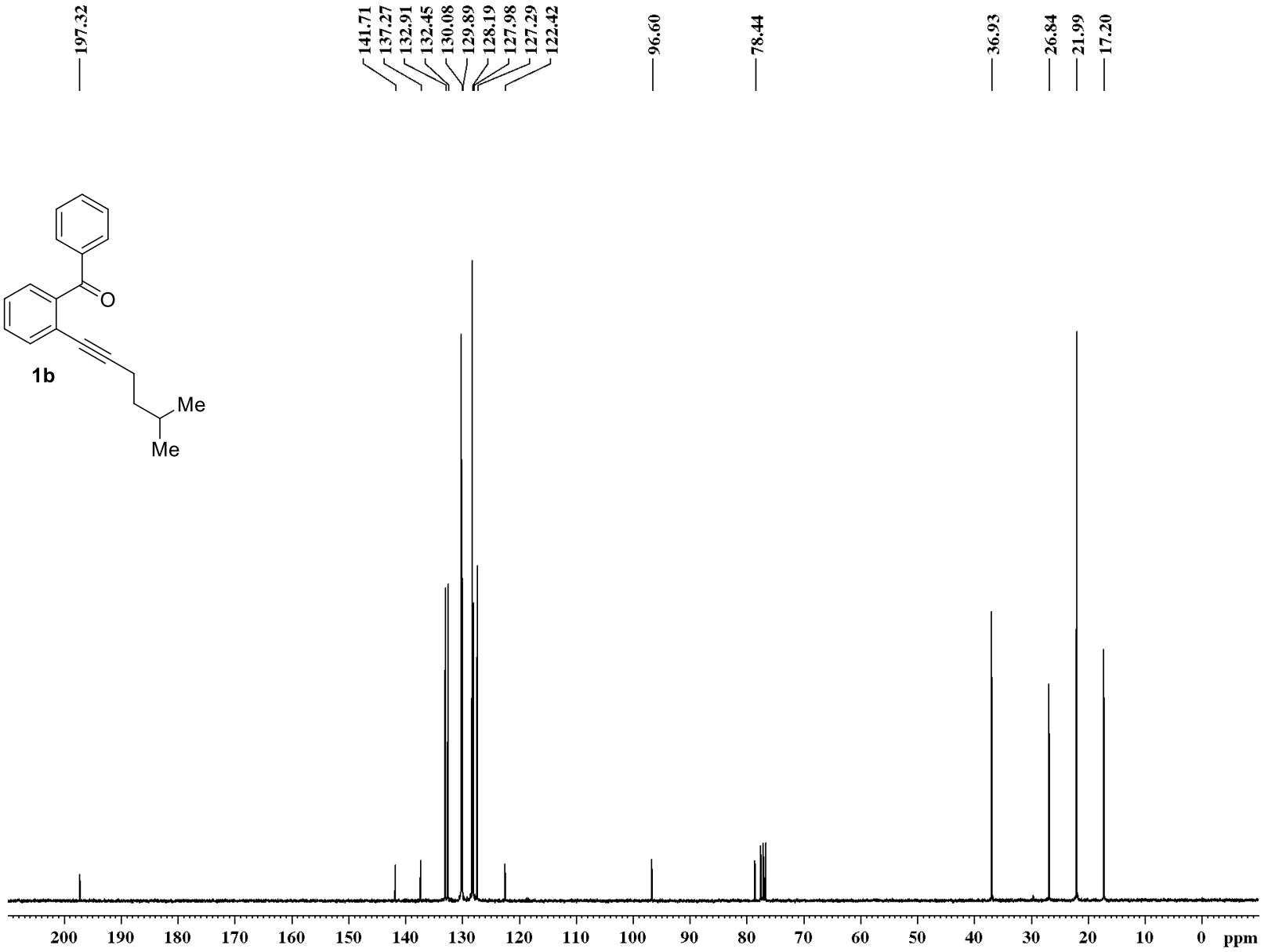
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Me
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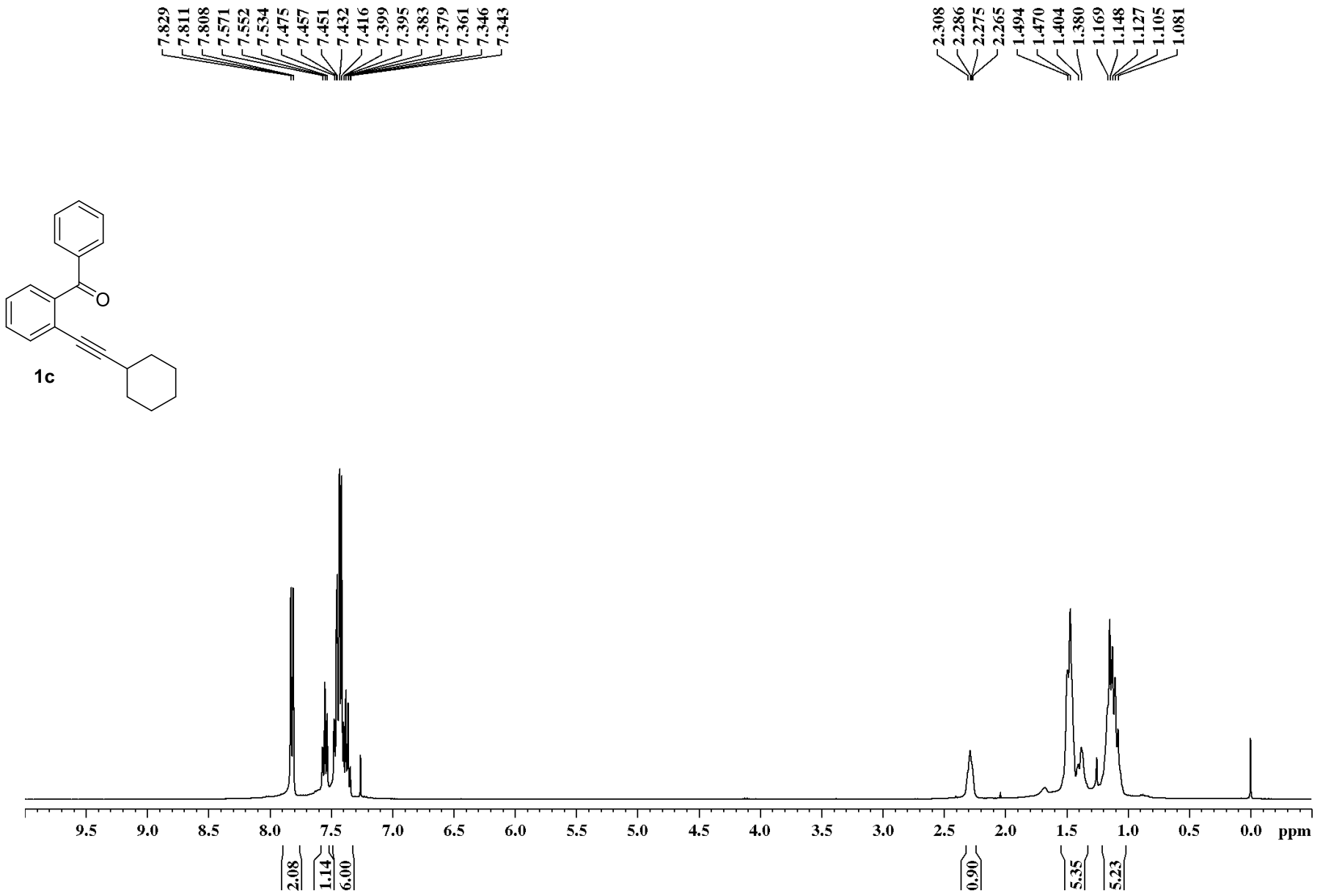
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6.0
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5.0
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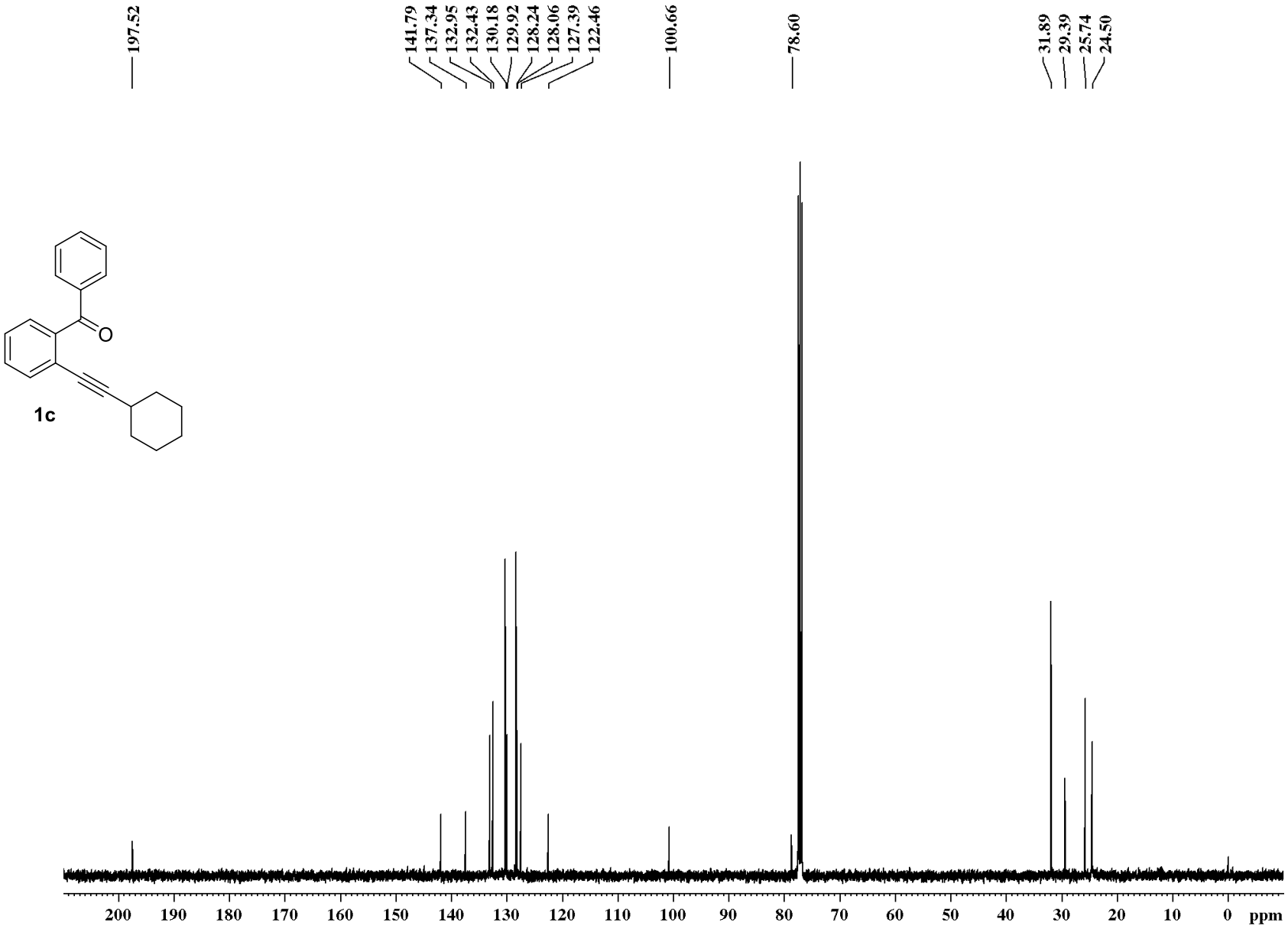
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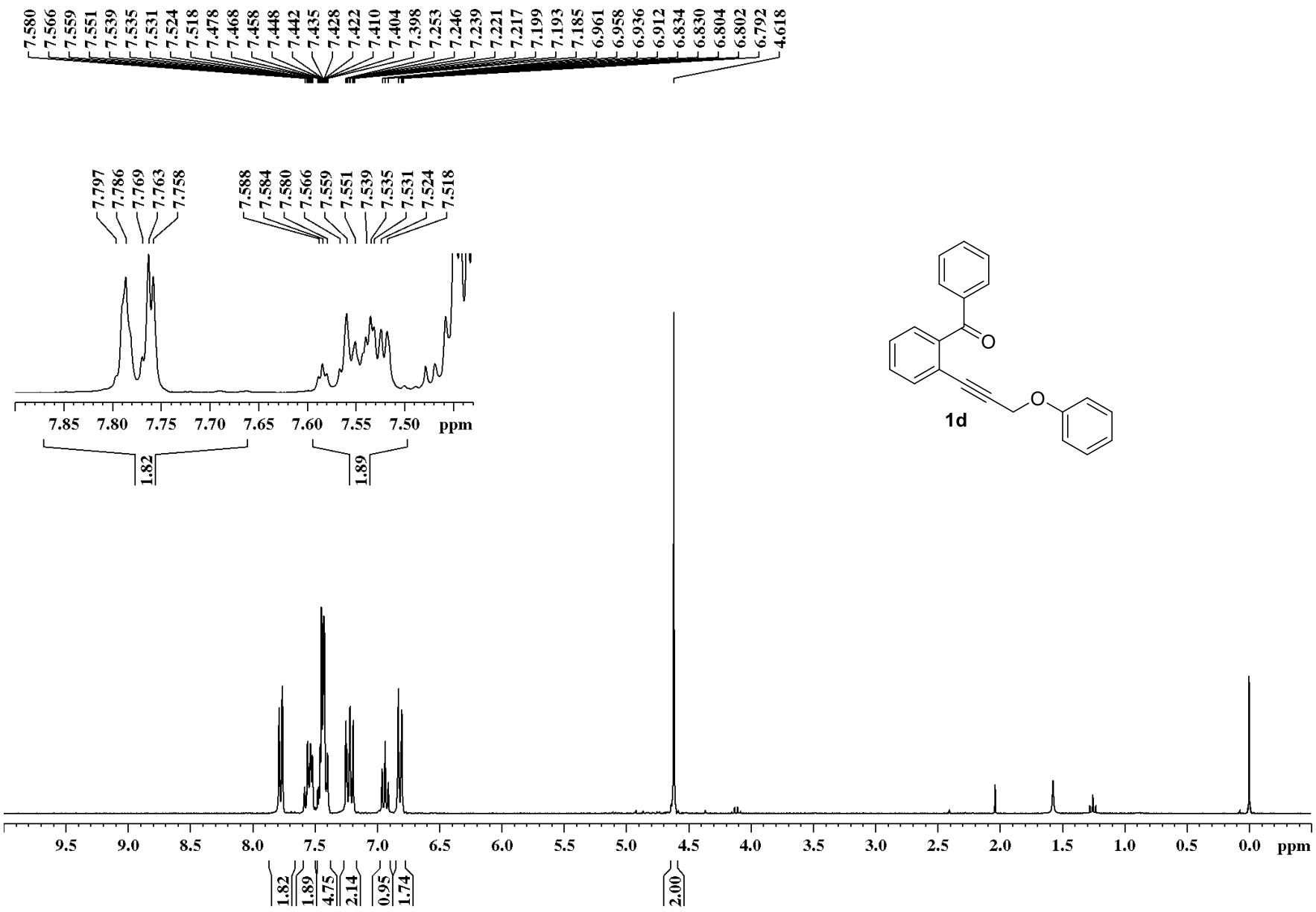
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7.566
7.559
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7.539
7.535
7.531
7.524
7.518
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O
O
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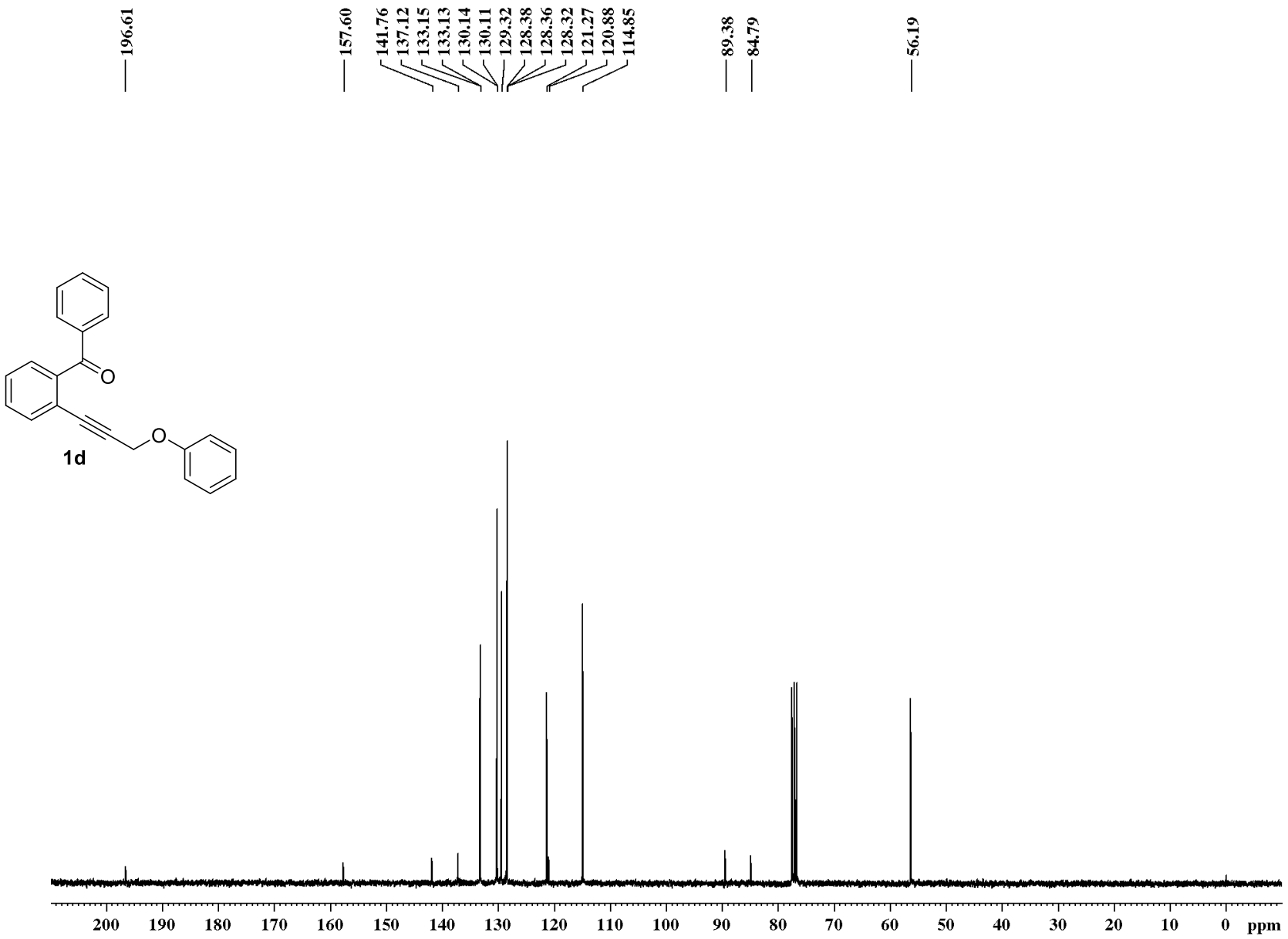
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O
O
1d
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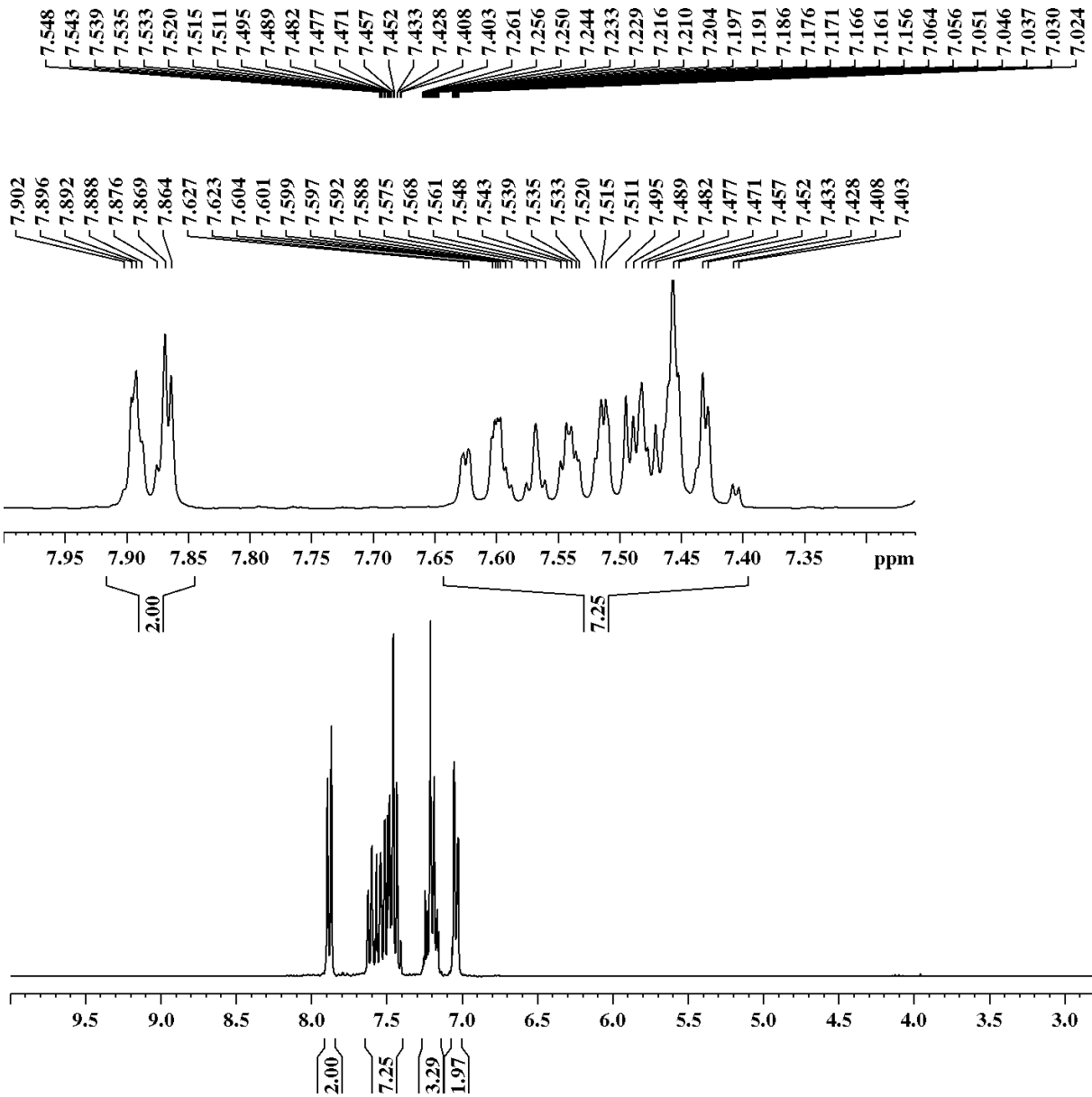
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7.902 7.896 7.892 7.888 7.876 7.869 7.864 7.627 7.623 7.604 7.601 7.599 7.597 7.592 7.588 7.575 7.568 7.561 7.548 7.543 7.539 7.535 7.533 7.520 7.515 7.511 7.495 7.489 7.482 7.477 7.471 7.457 7.452 7.433 7.428 7.408 7.403
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 ppm
2.00
7.25
O
1e
9.5 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 0.0 ppm
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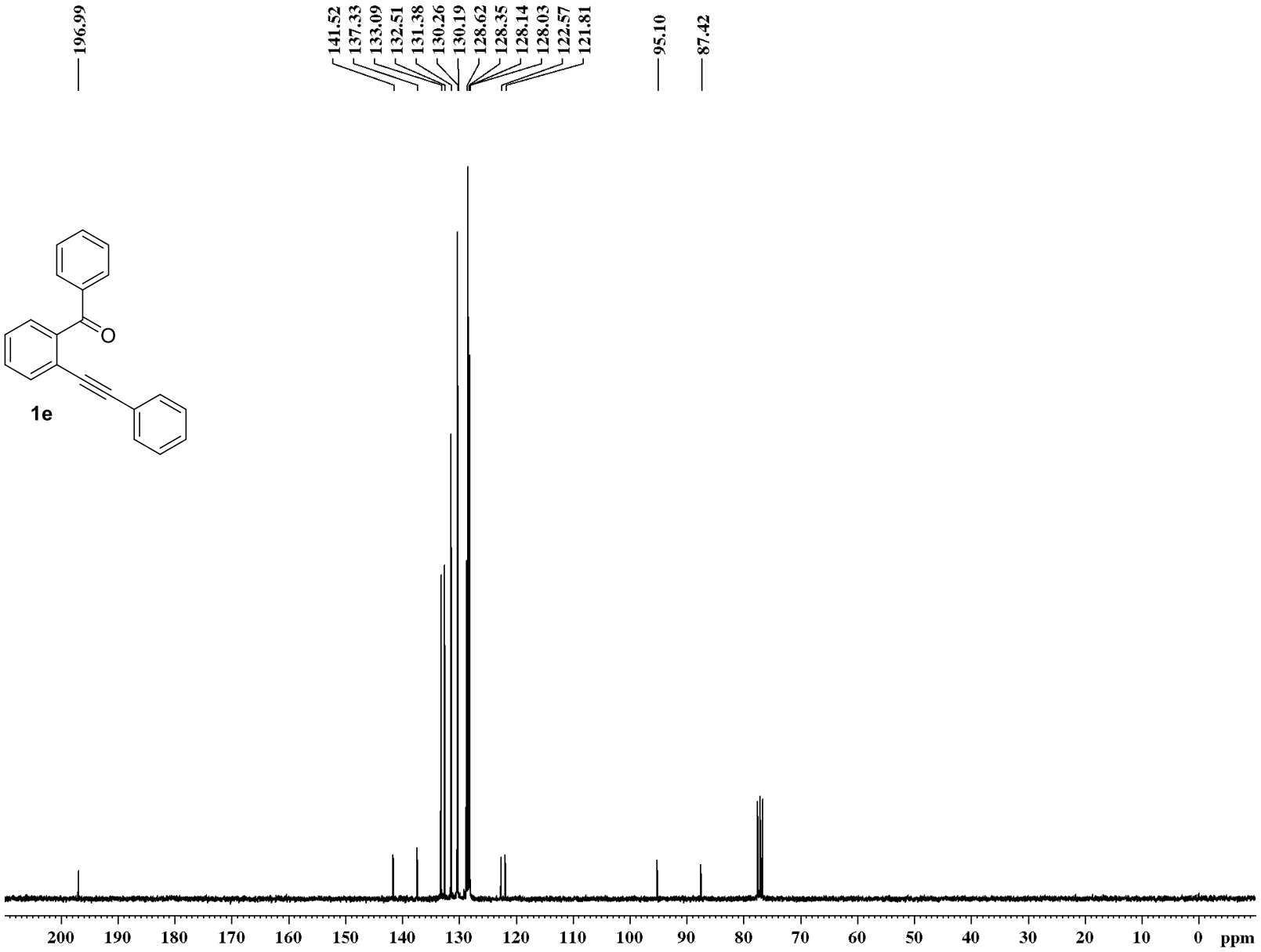
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87.42
O
1e
200
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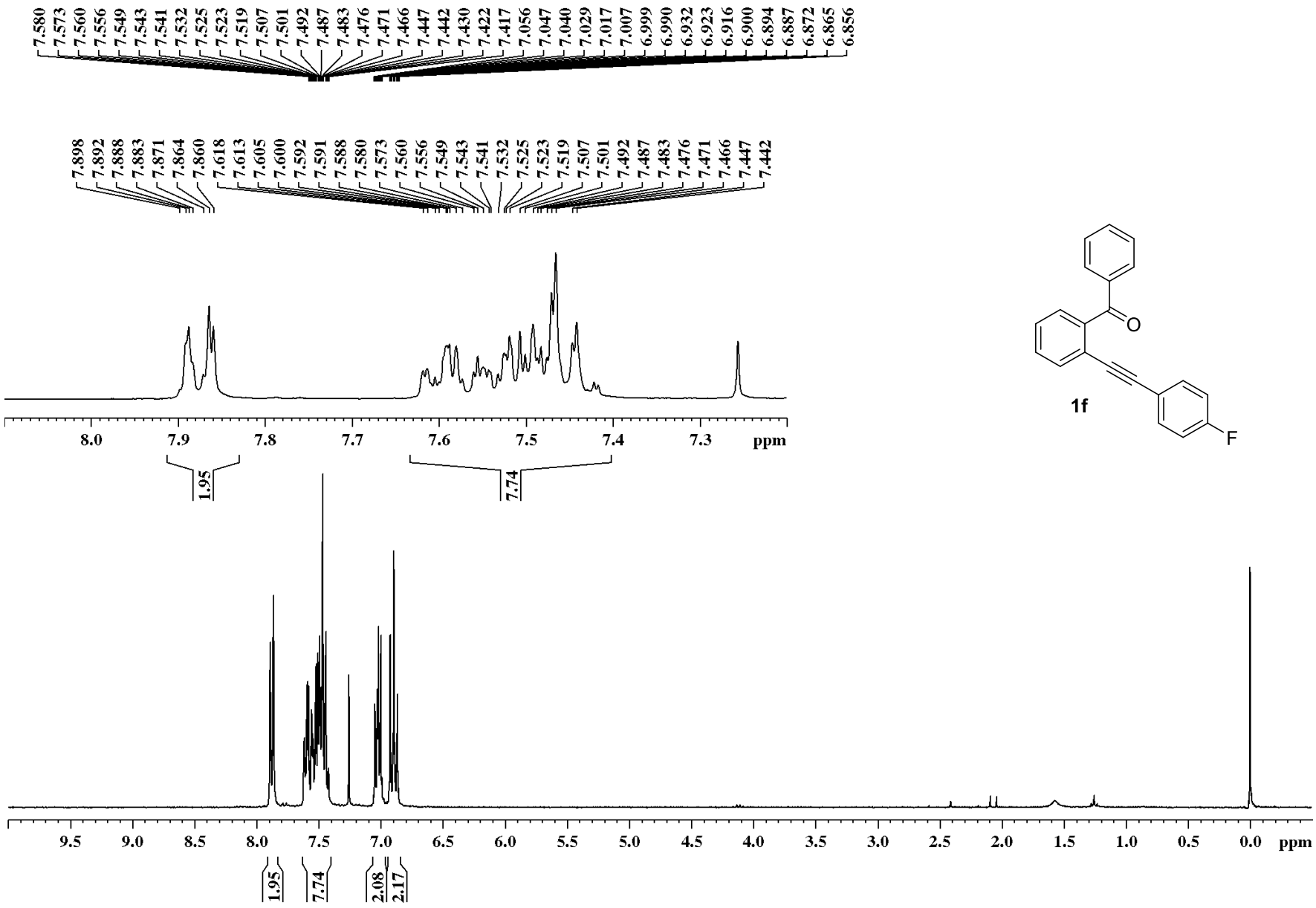
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7.898 7.892 7.888 7.883 7.871 7.864 7.860 7.618 7.613 7.605 7.600 7.592 7.591 7.588 7.580 7.573 7.560 7.556 7.549 7.543 7.541 7.532 7.525 7.523 7.519 7.507 7.501 7.492 7.487 7.483 7.476 7.471 7.466 7.447 7.442
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
1.95
7.74
O
F
1f
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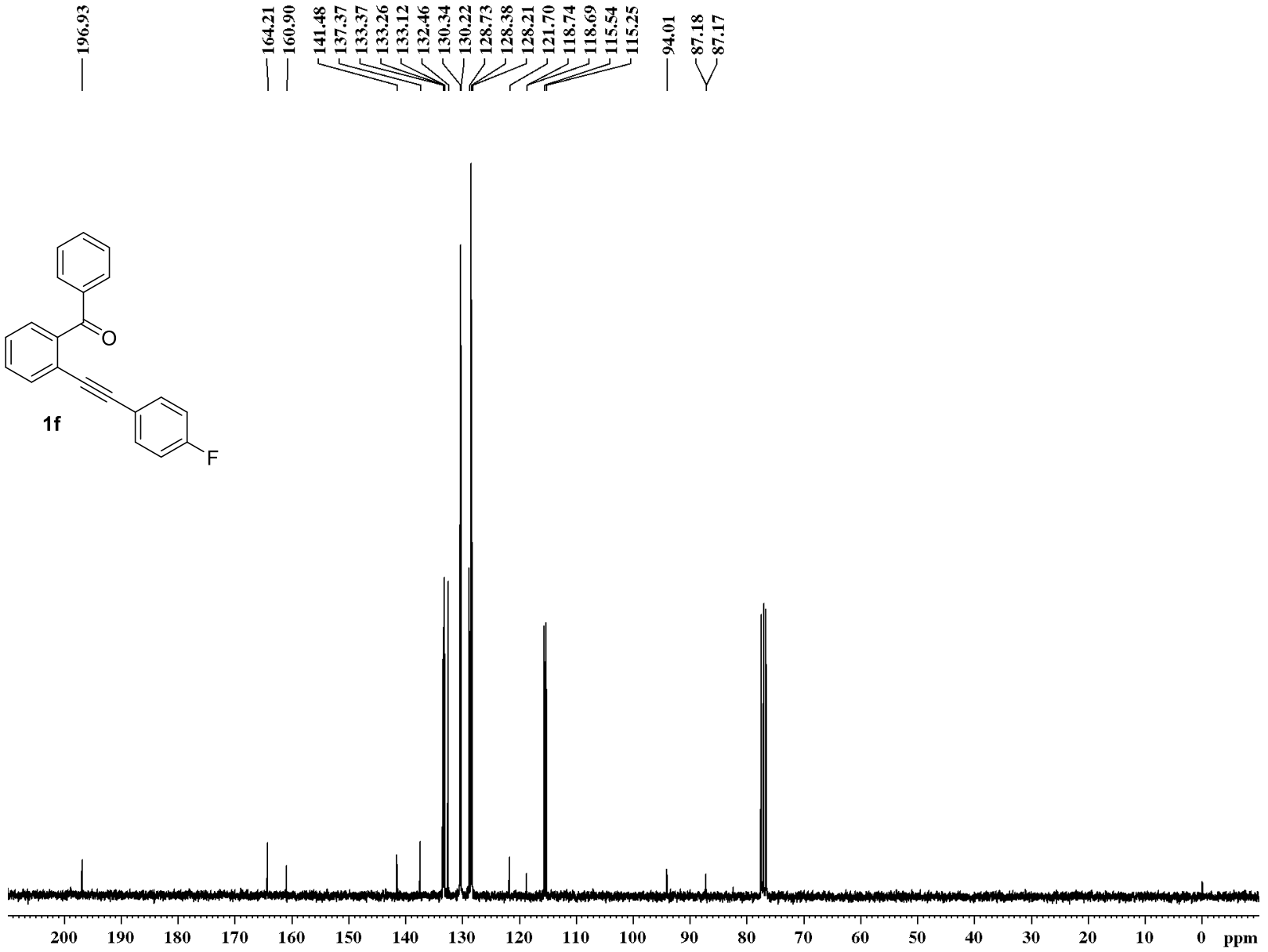
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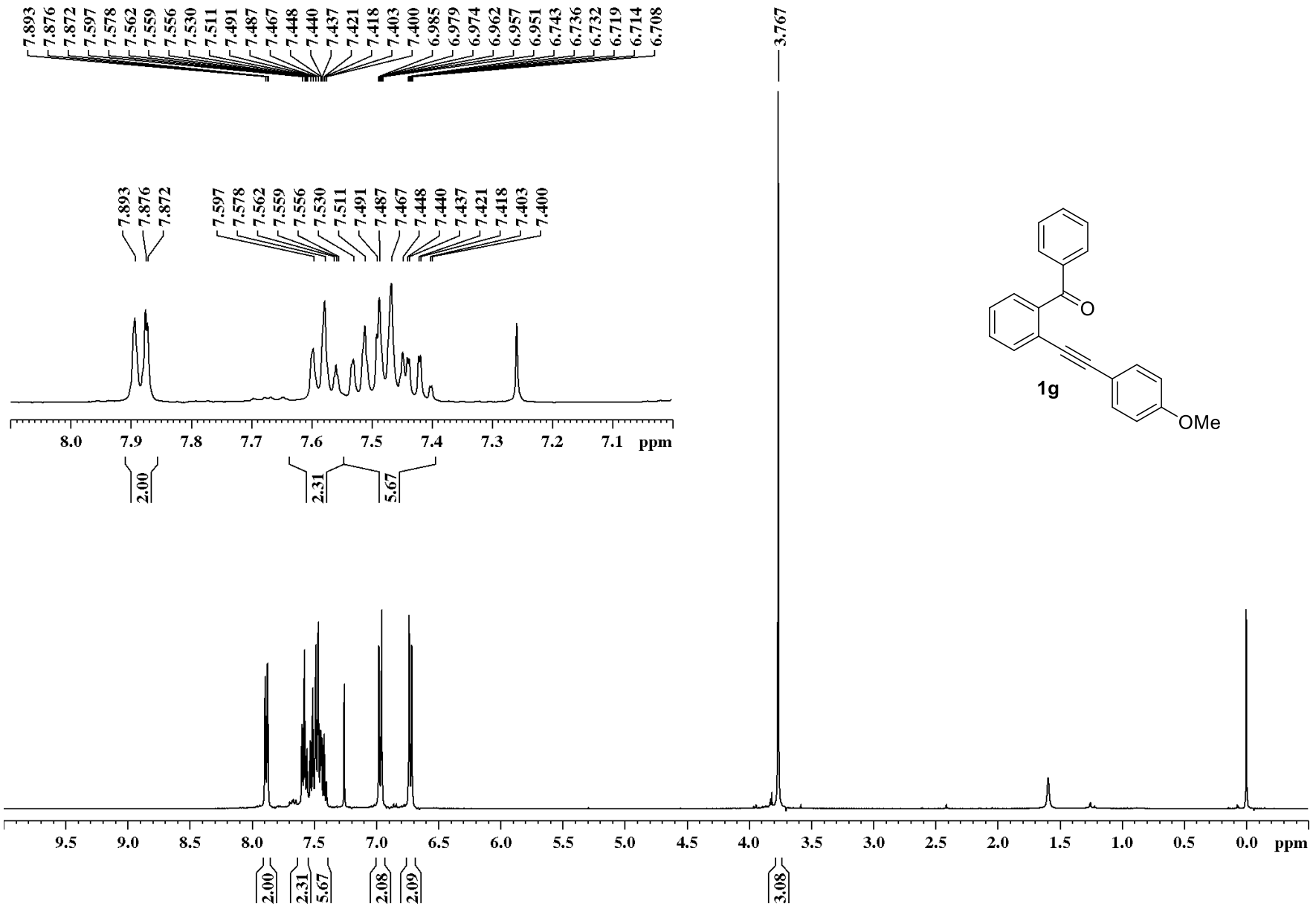
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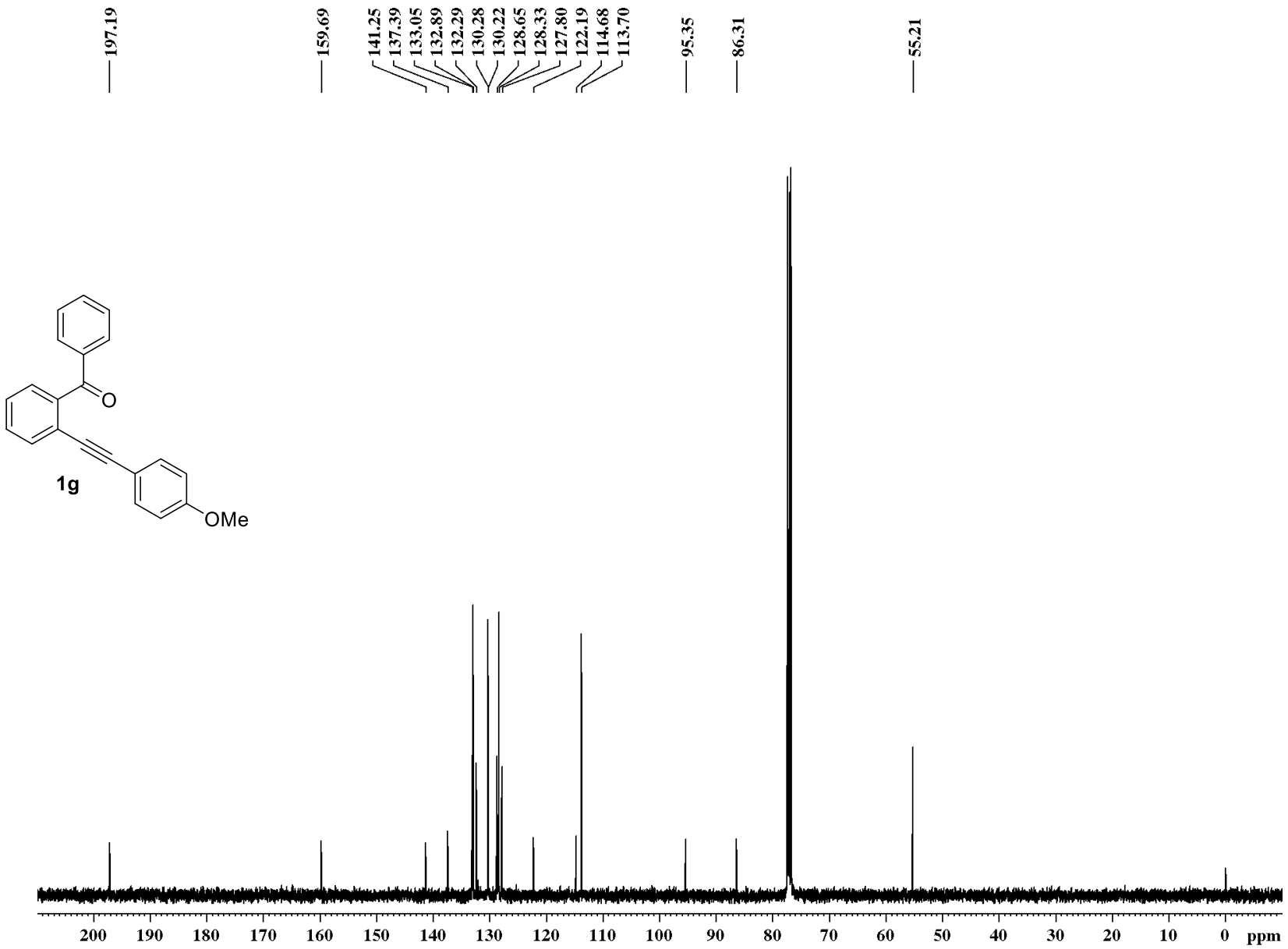
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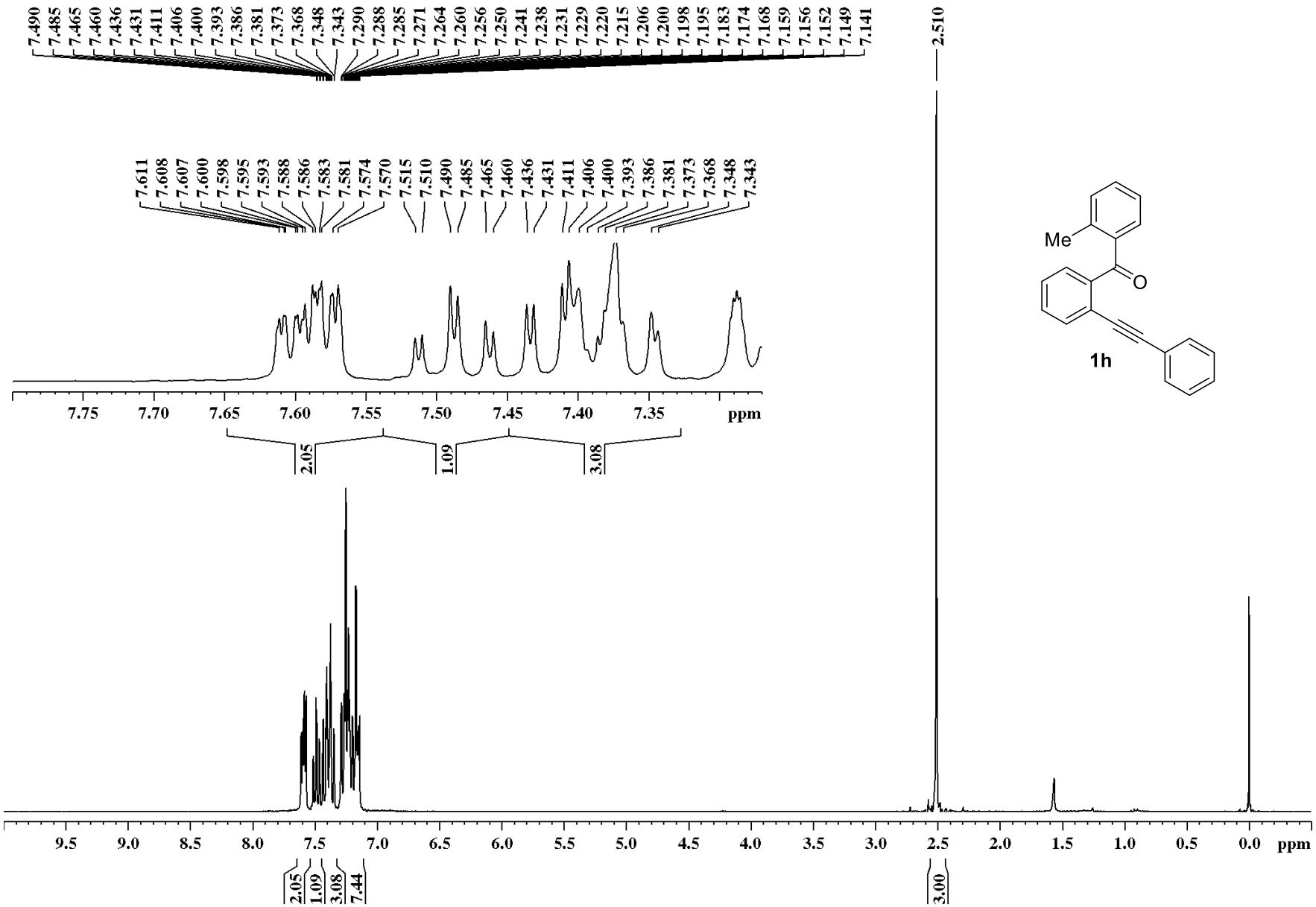
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7.238
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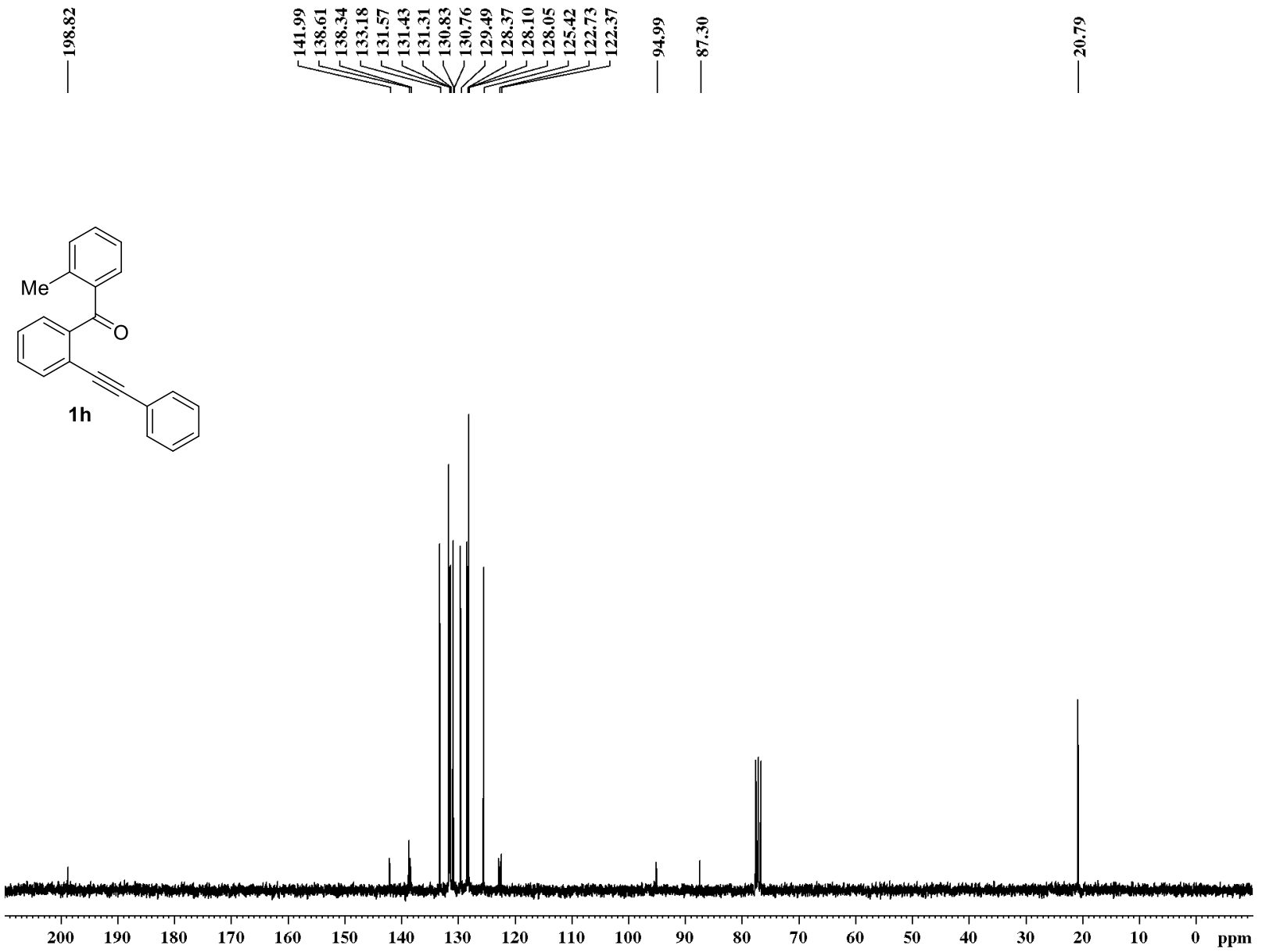
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128.10
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122.73
122.37
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20.79
Me
O
1h
200
190
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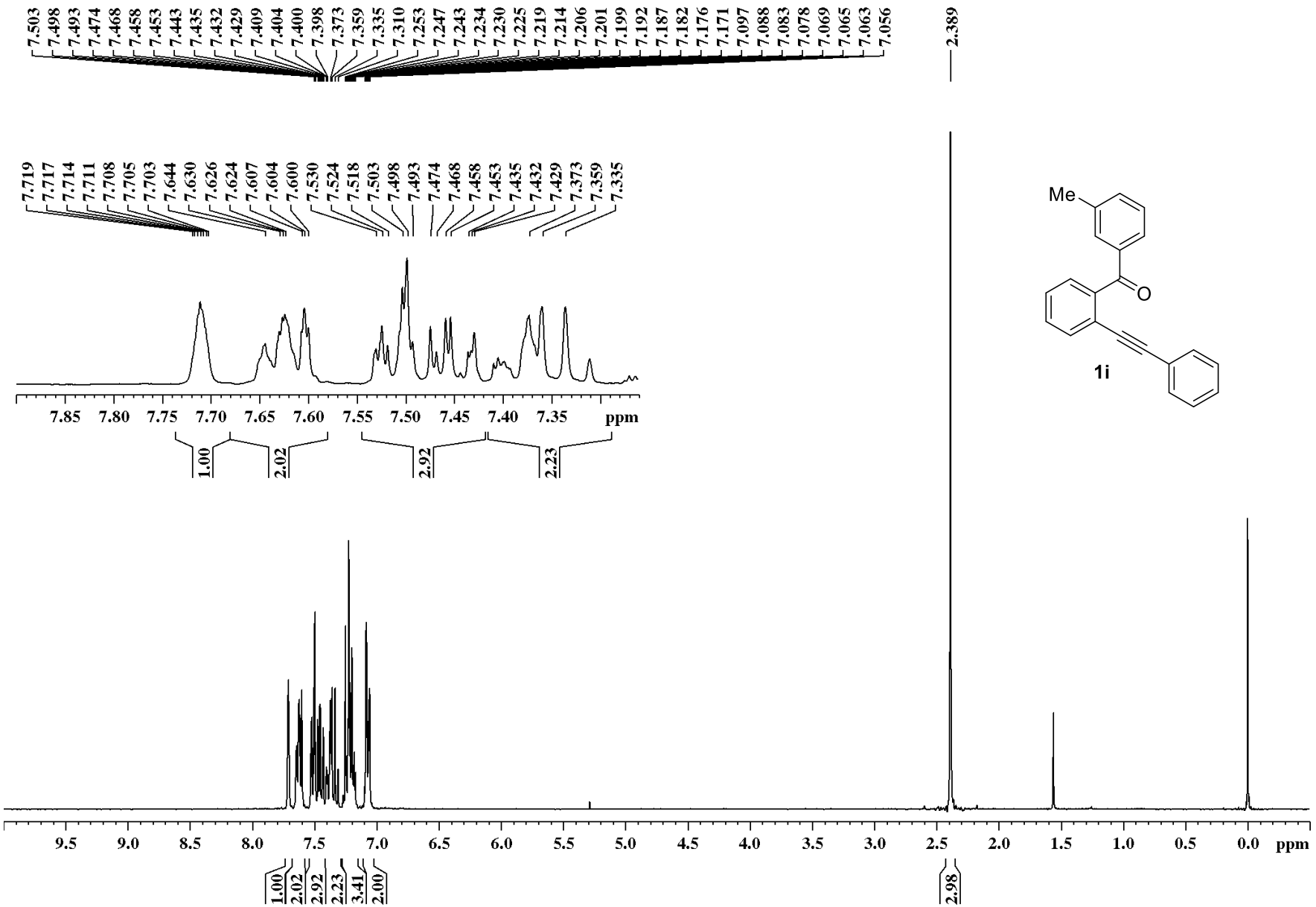
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7.474
7.468
7.458
7.453
7.435
7.432
7.429
7.373
7.359
7.335
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 ppm
1.00
2.02
2.92
2.23
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
2.02
2.92
2.23
3.41
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Me
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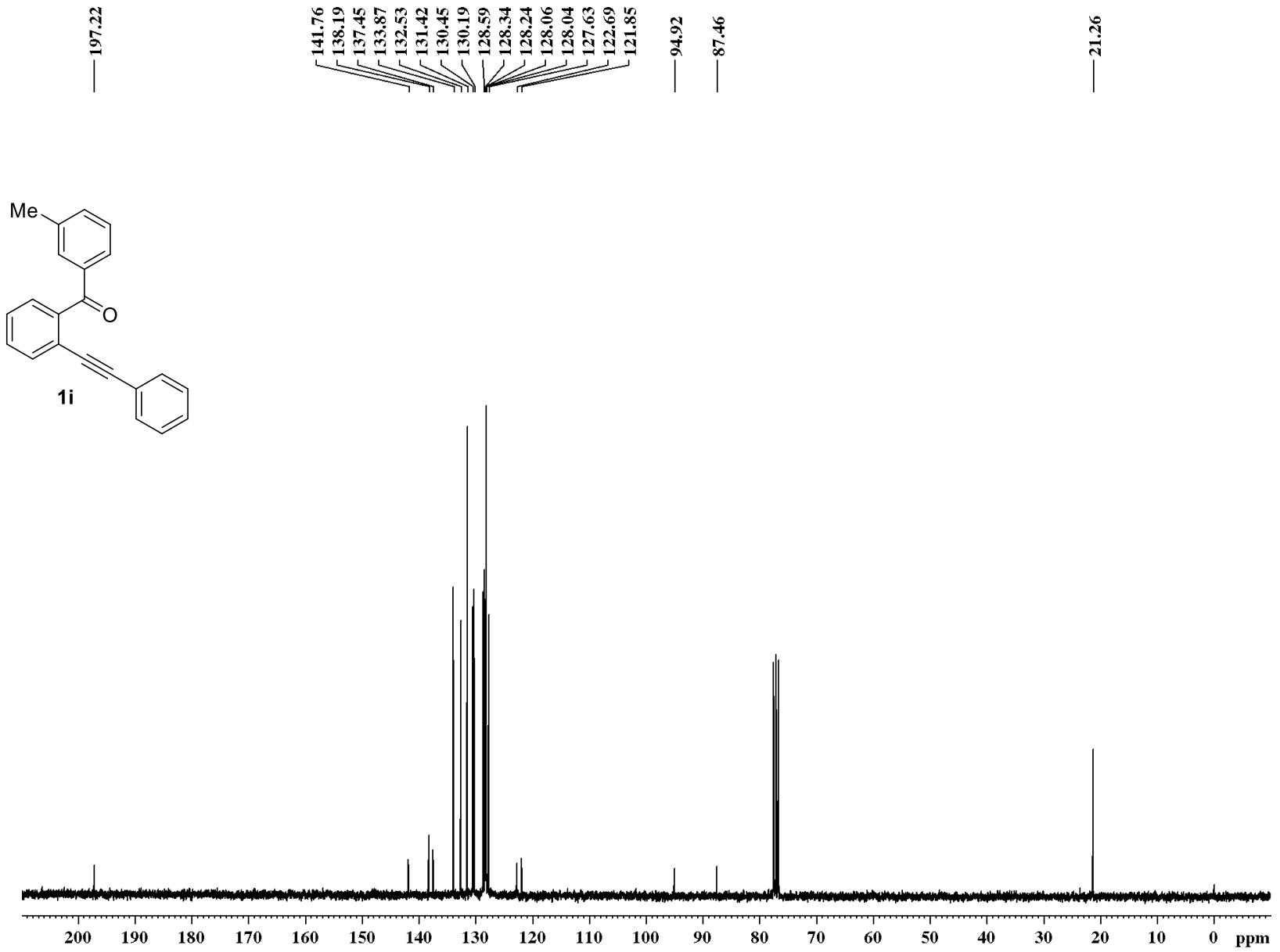
1i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87.46
21.26
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O
1i
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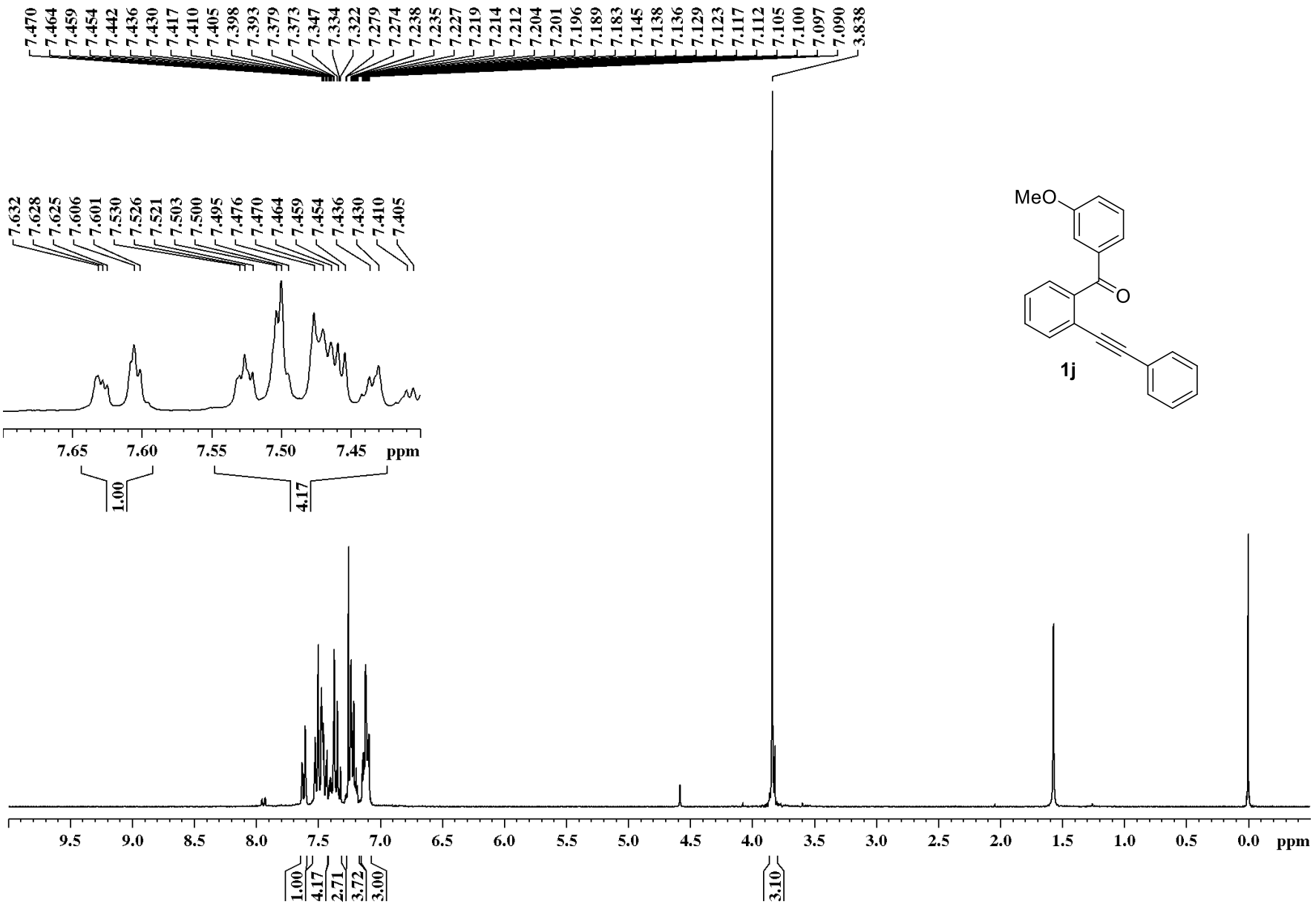
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O
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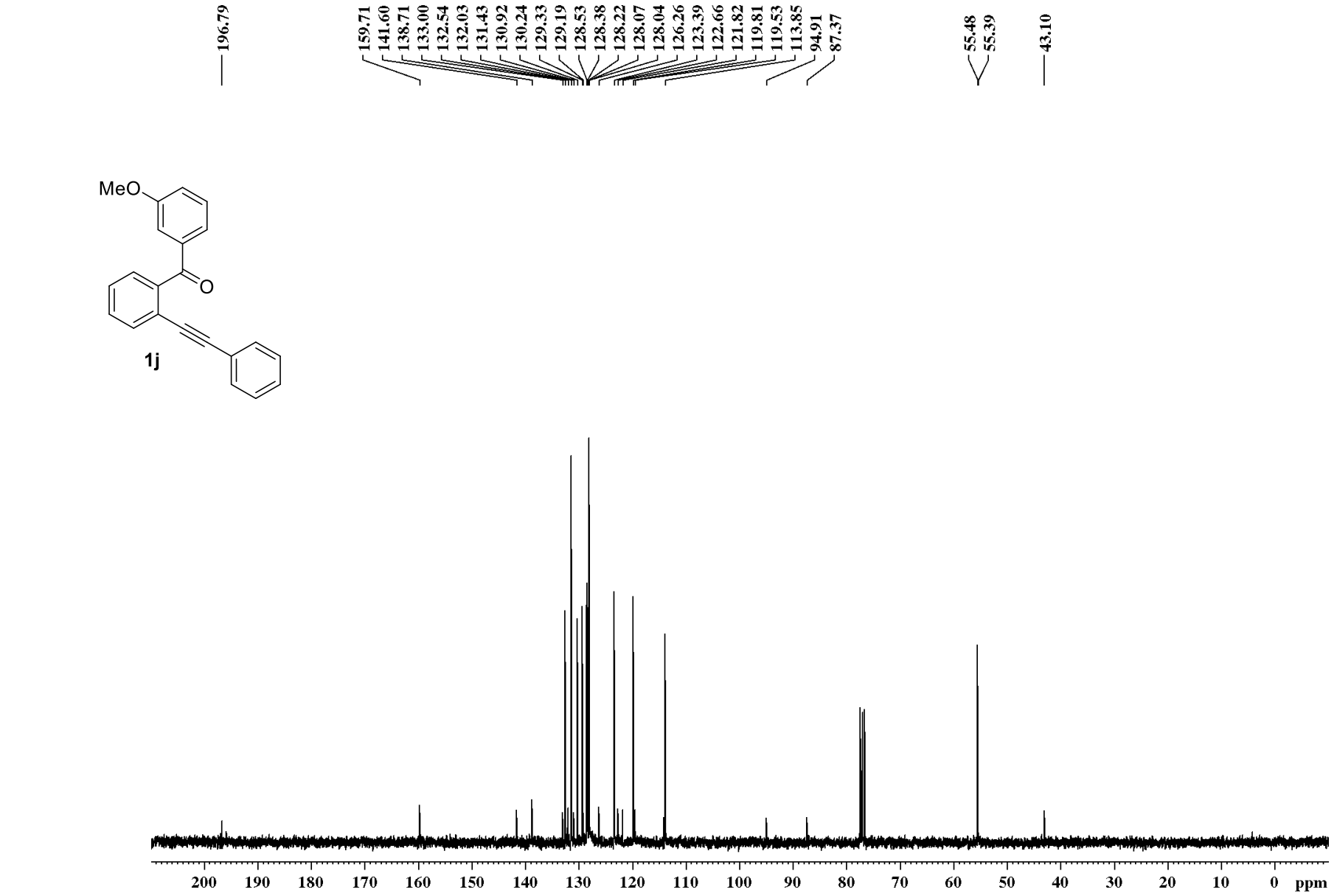
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O
1j
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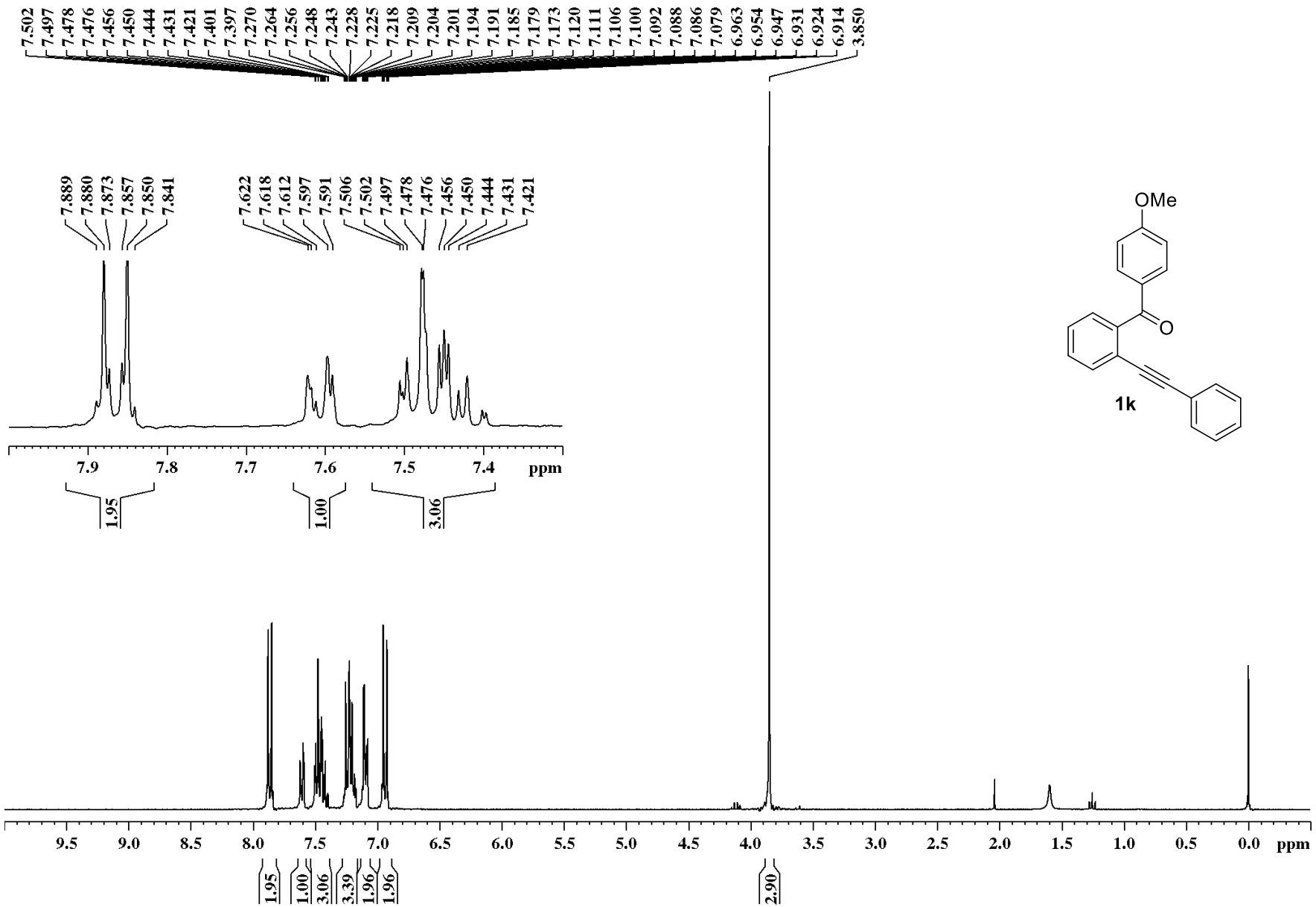
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OMe
O
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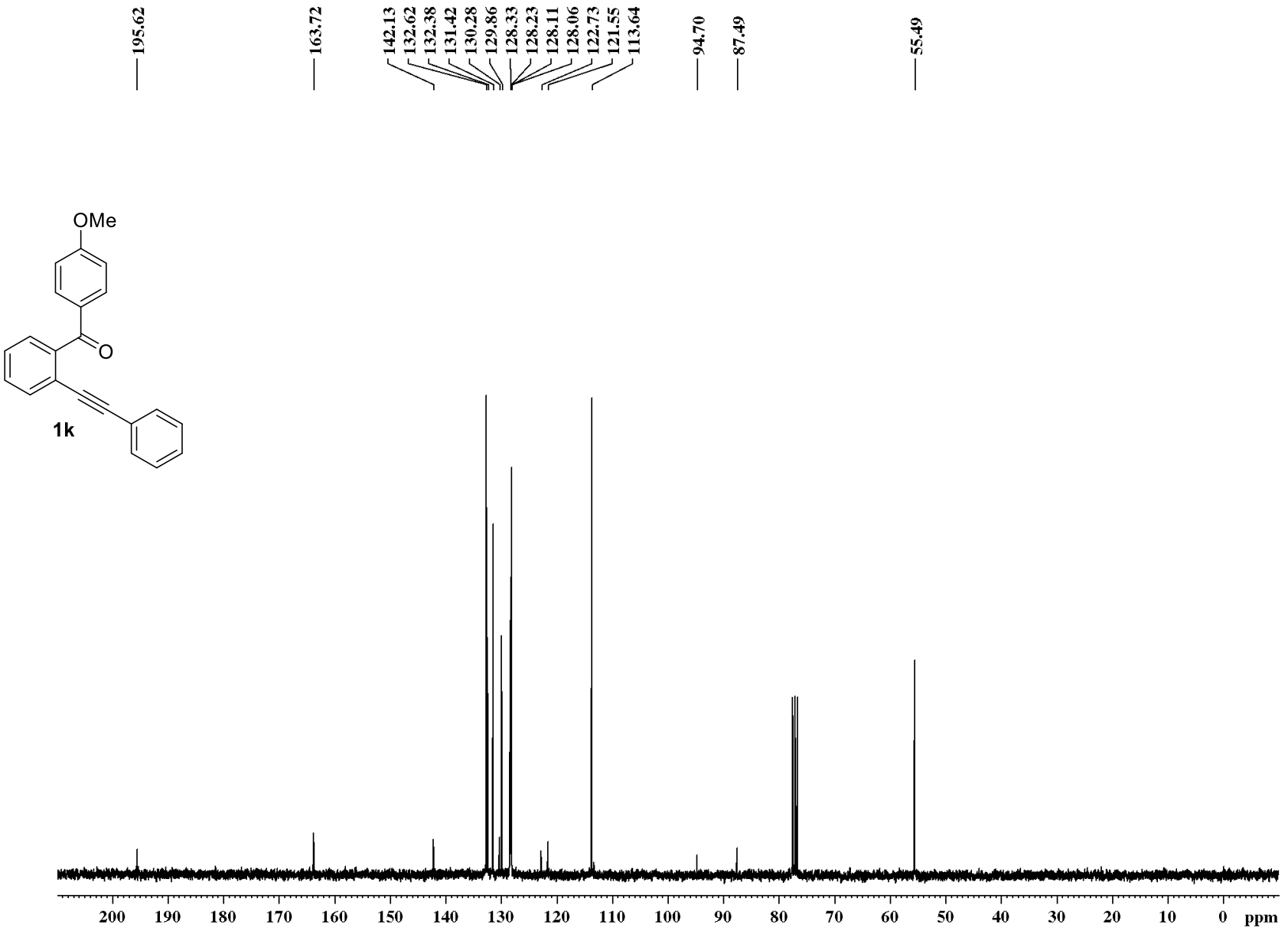
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87.49
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O
1k
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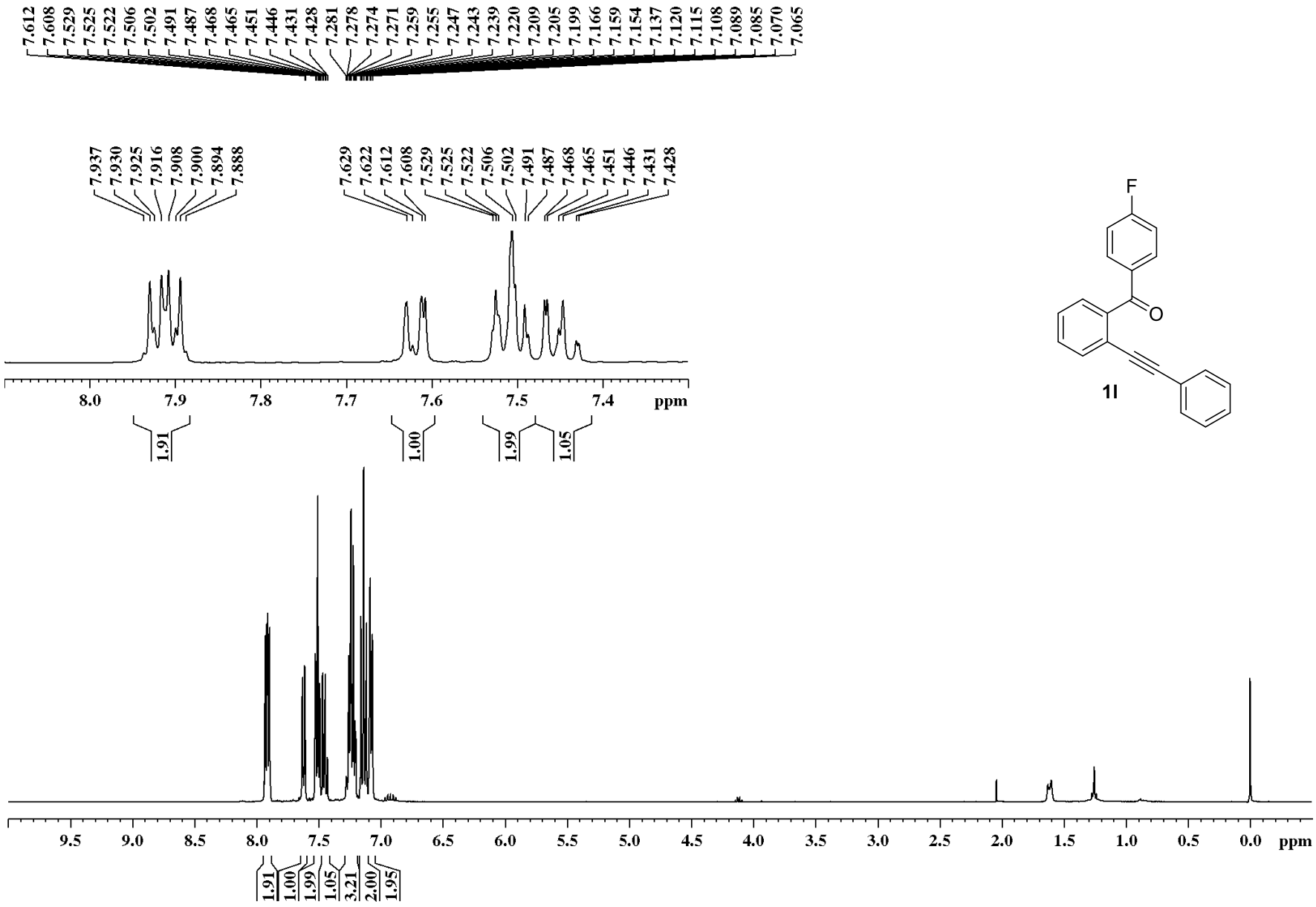
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7.205
7.199
7.166
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7.8
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7.6
7.5
7.4
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1.05
F
O
1l
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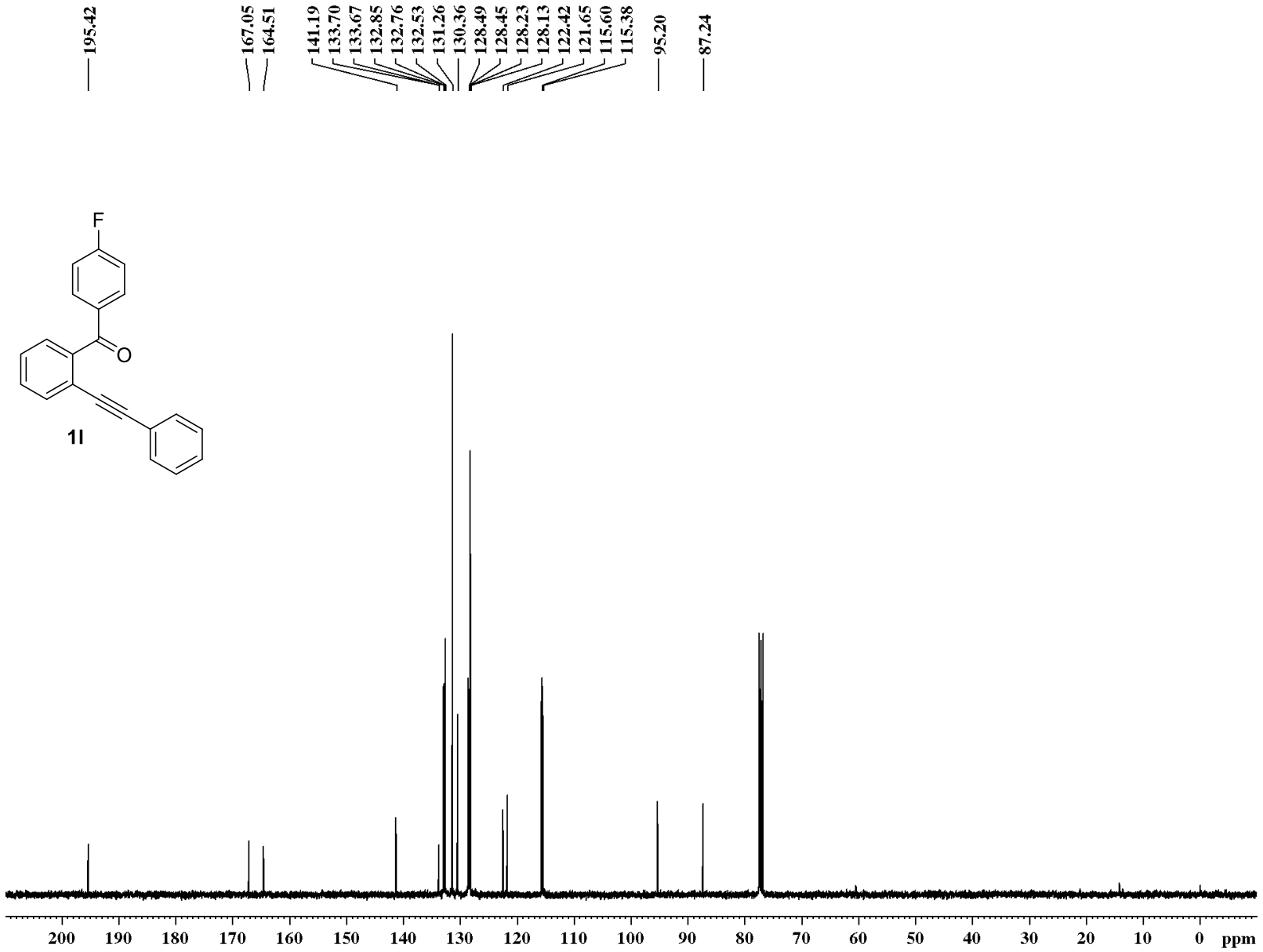
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122.42
121.65
115.60
115.38
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87.24
F
O
1l
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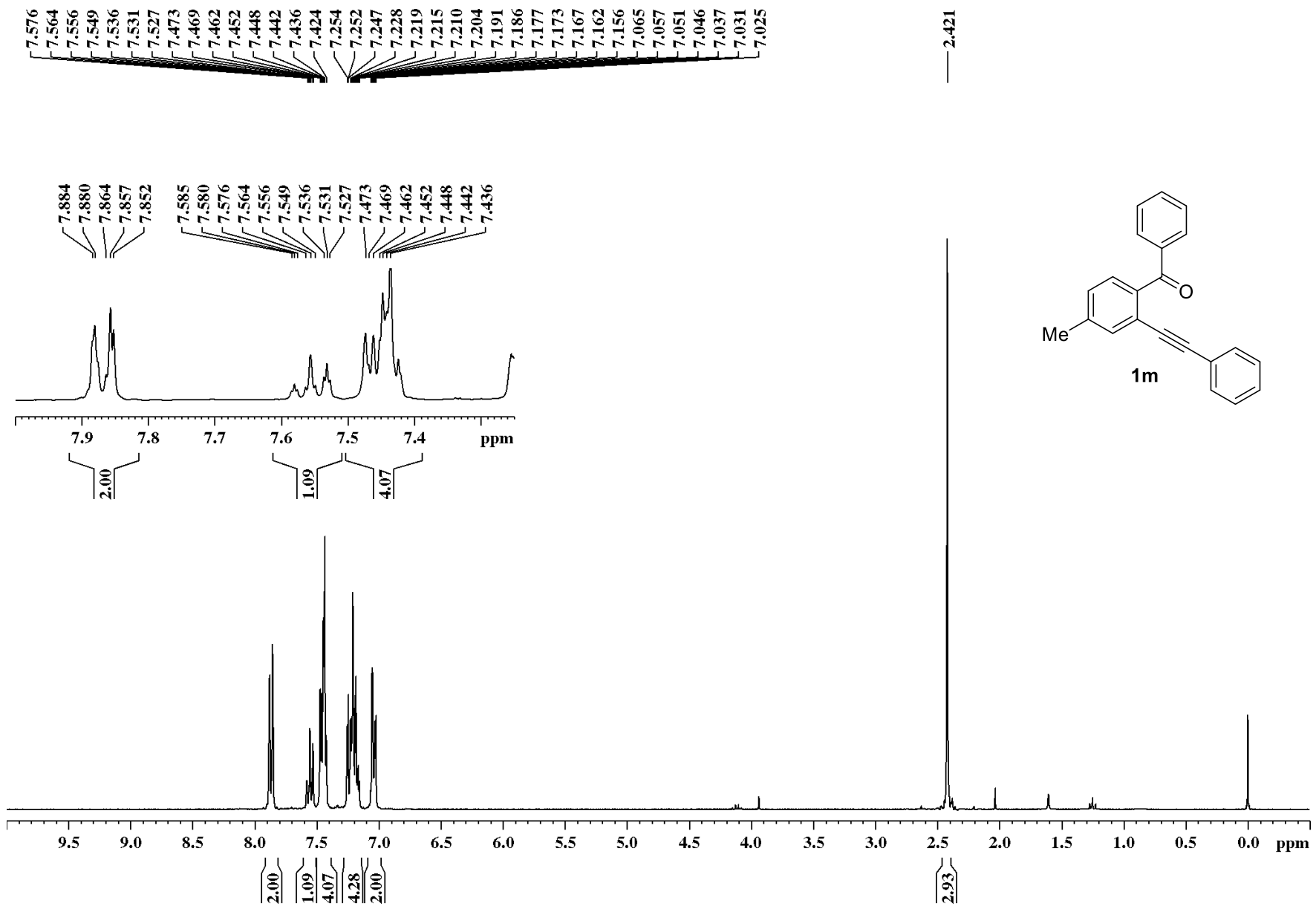
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7.173
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7.7
7.6
7.5
7.4
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4.07
Me
O
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4.0
3.5
3.0
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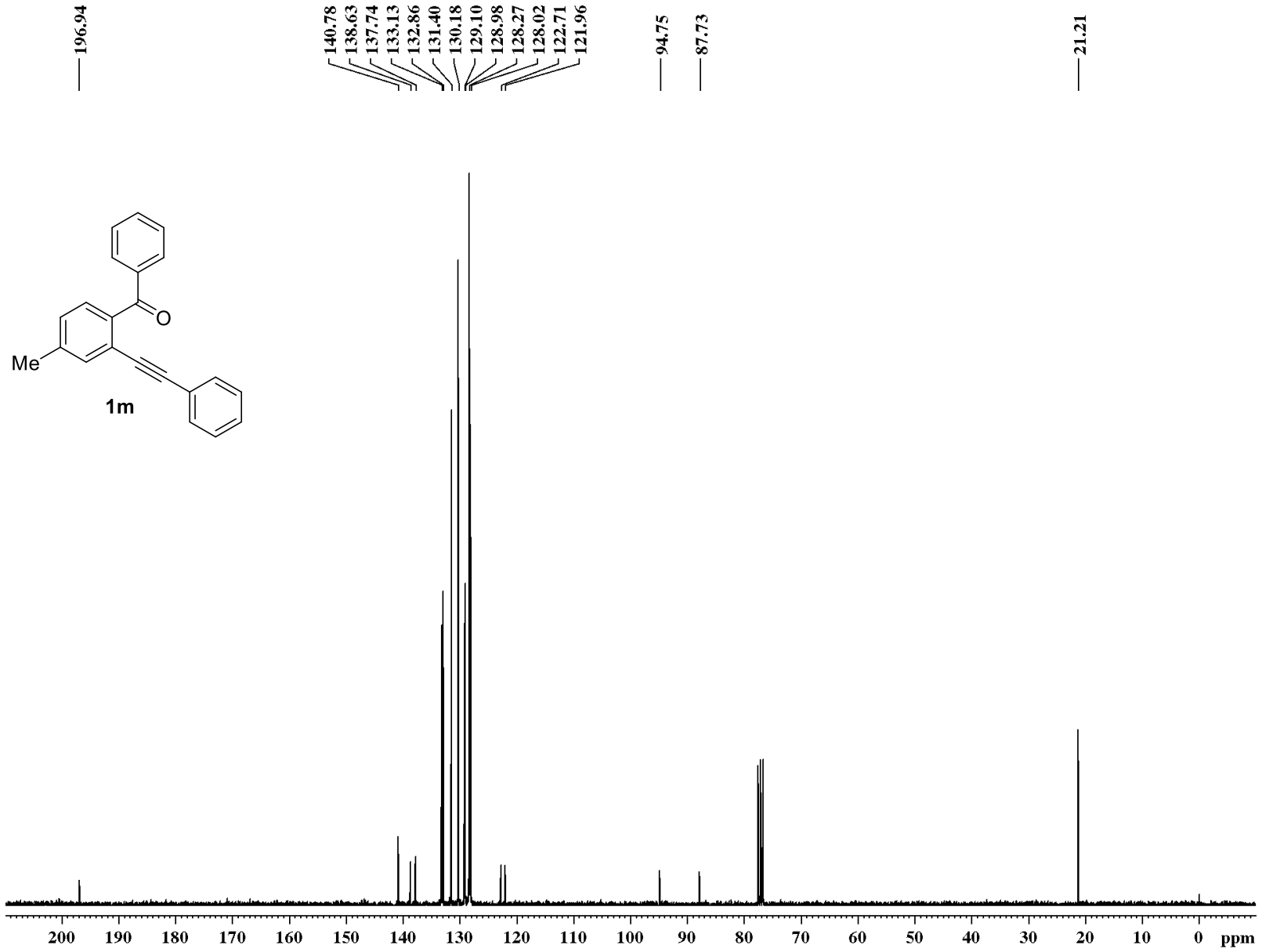
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87.73
21.21
O
Me
1m
200
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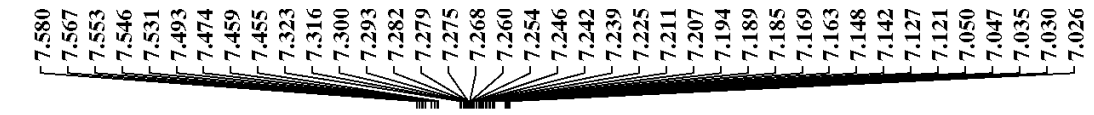
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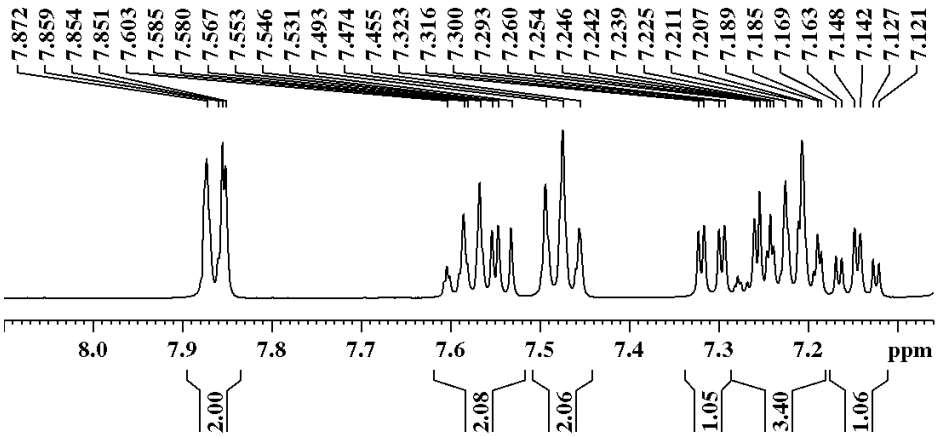
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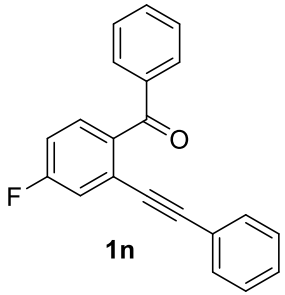
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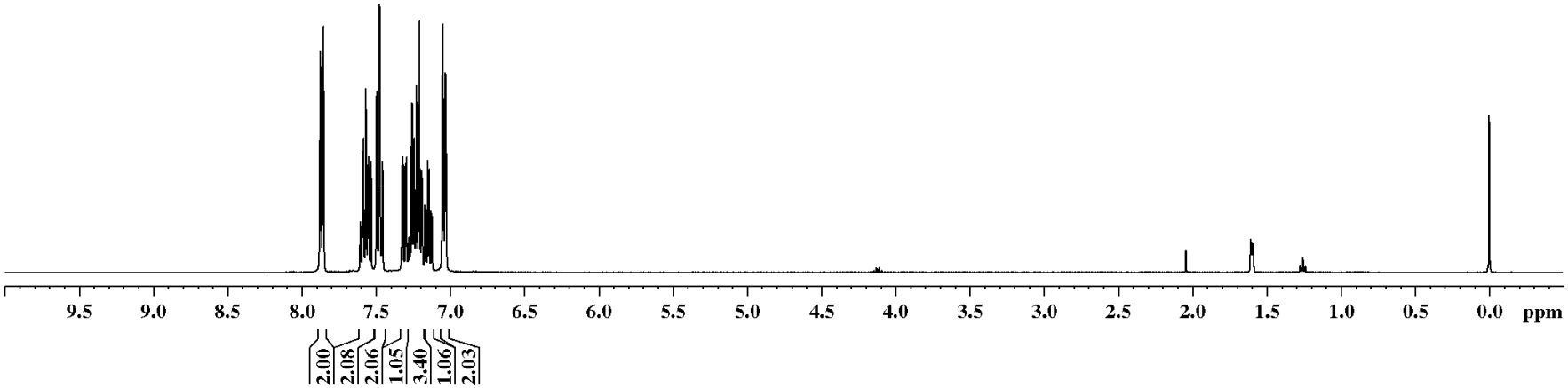
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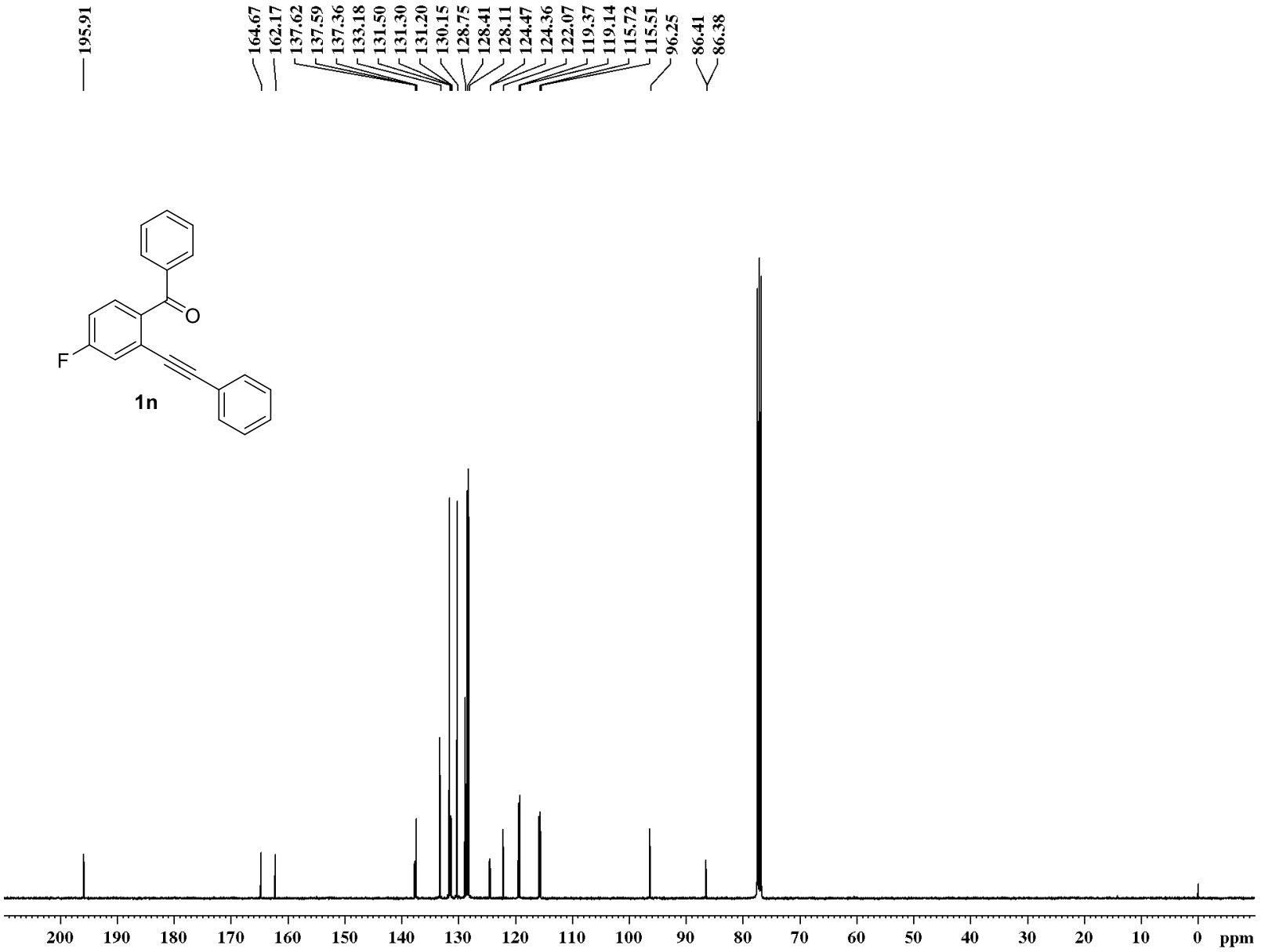
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119.14
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115.51
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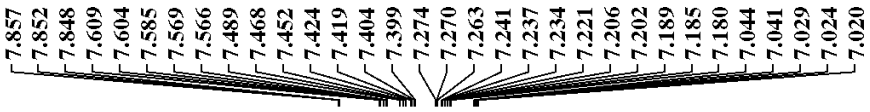
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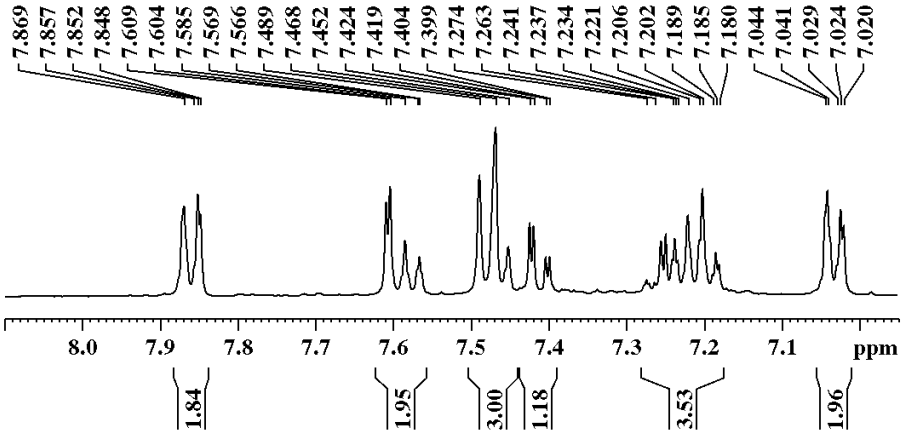
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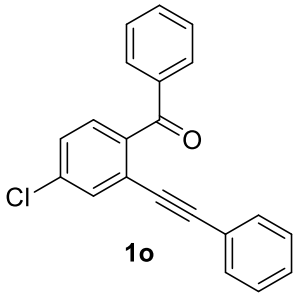
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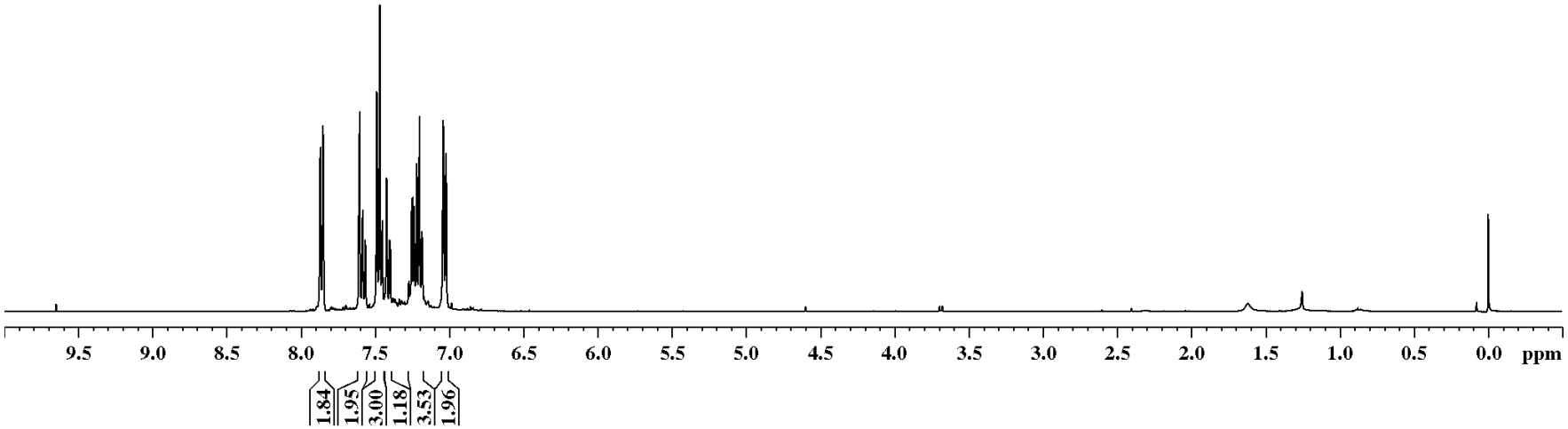
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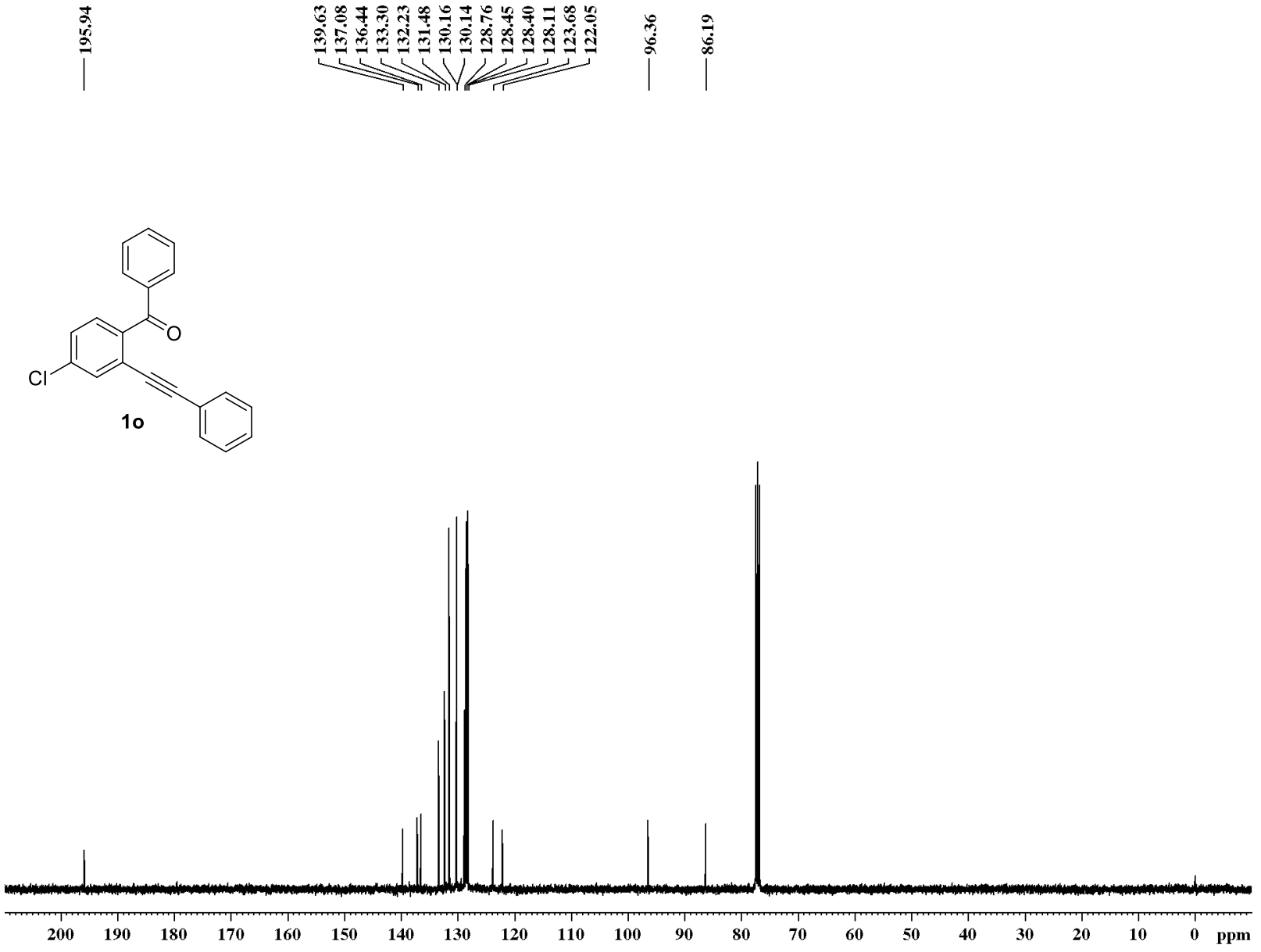
1.96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O
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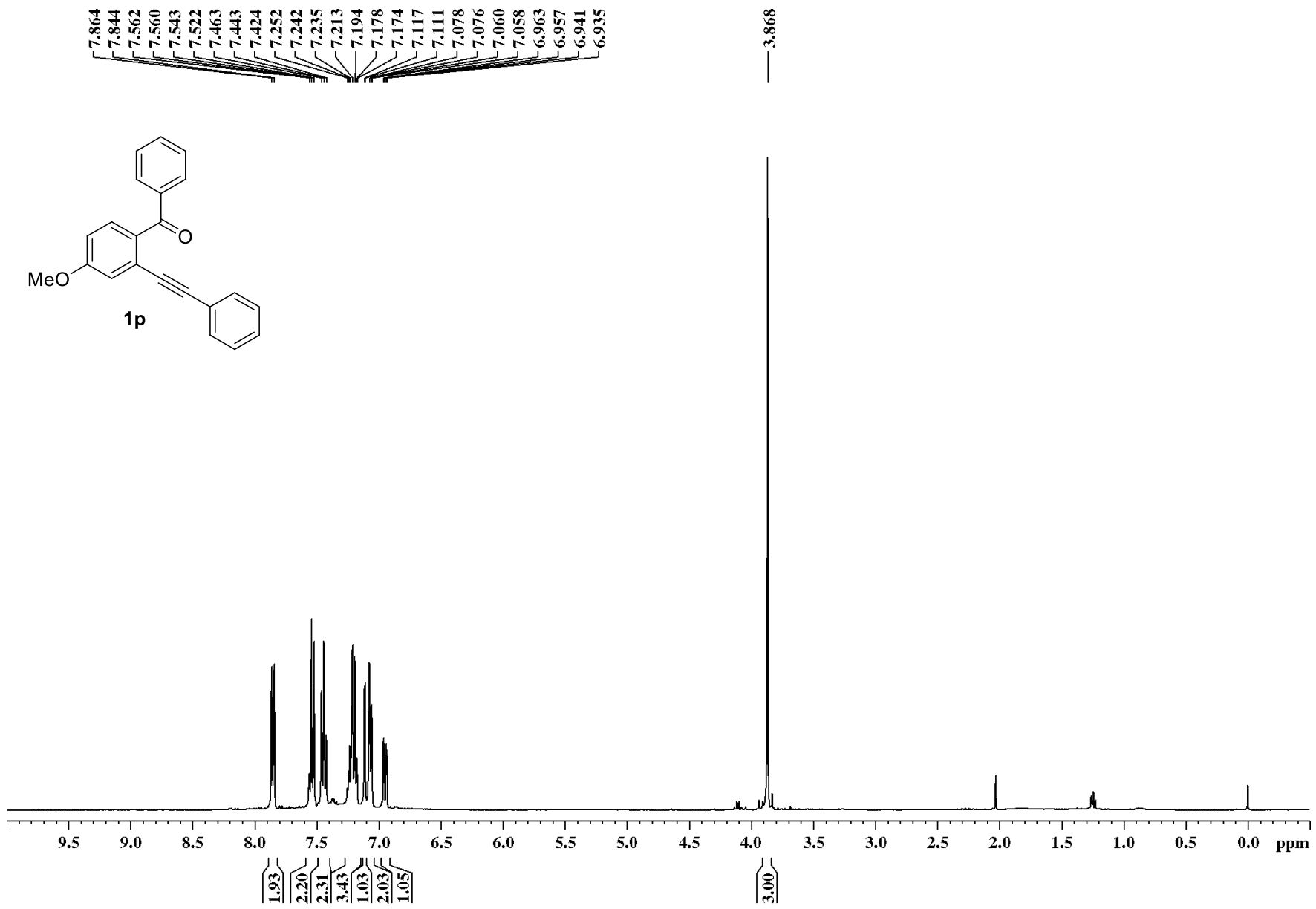
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7.060
7.058
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O
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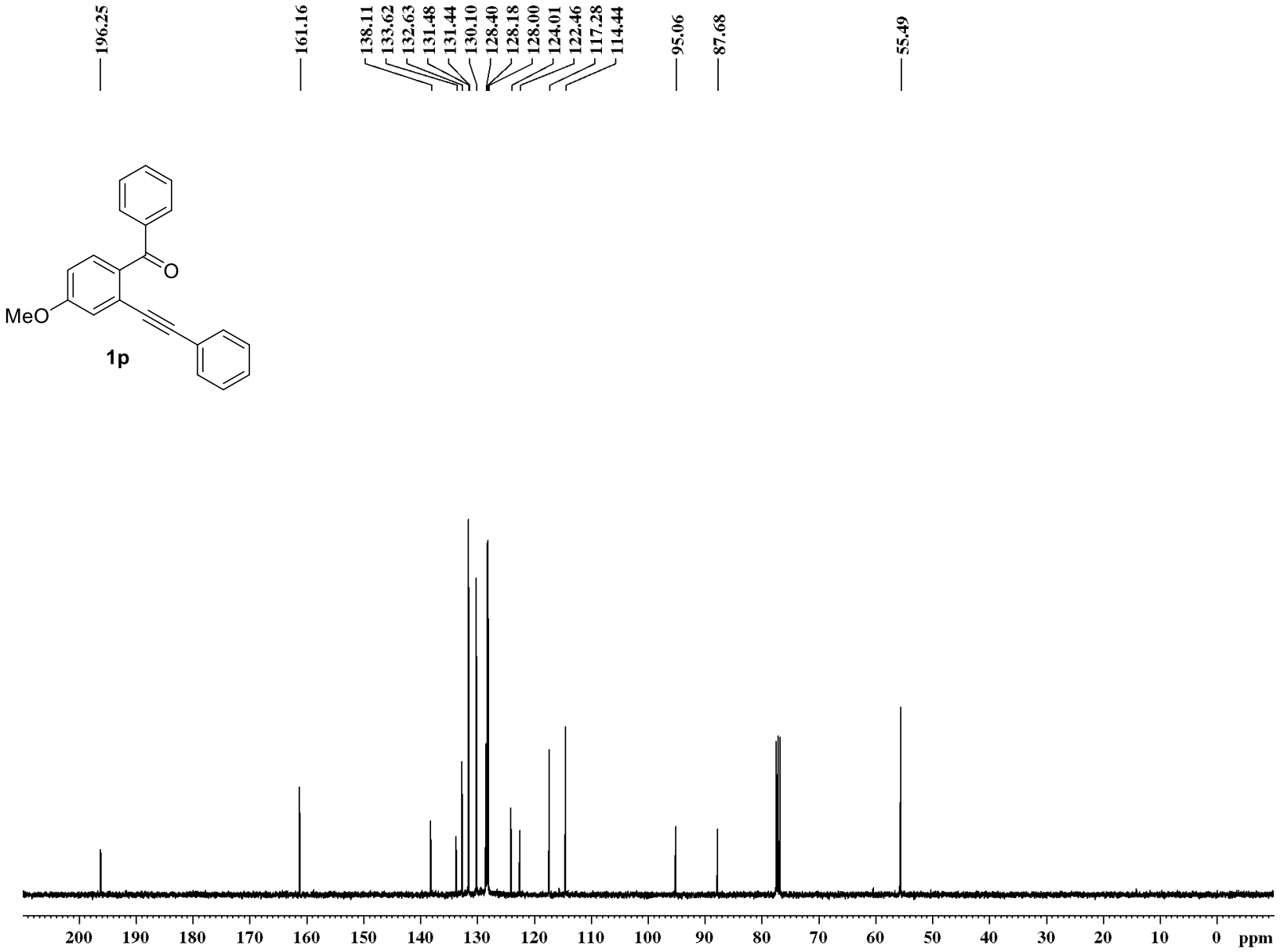
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MeO
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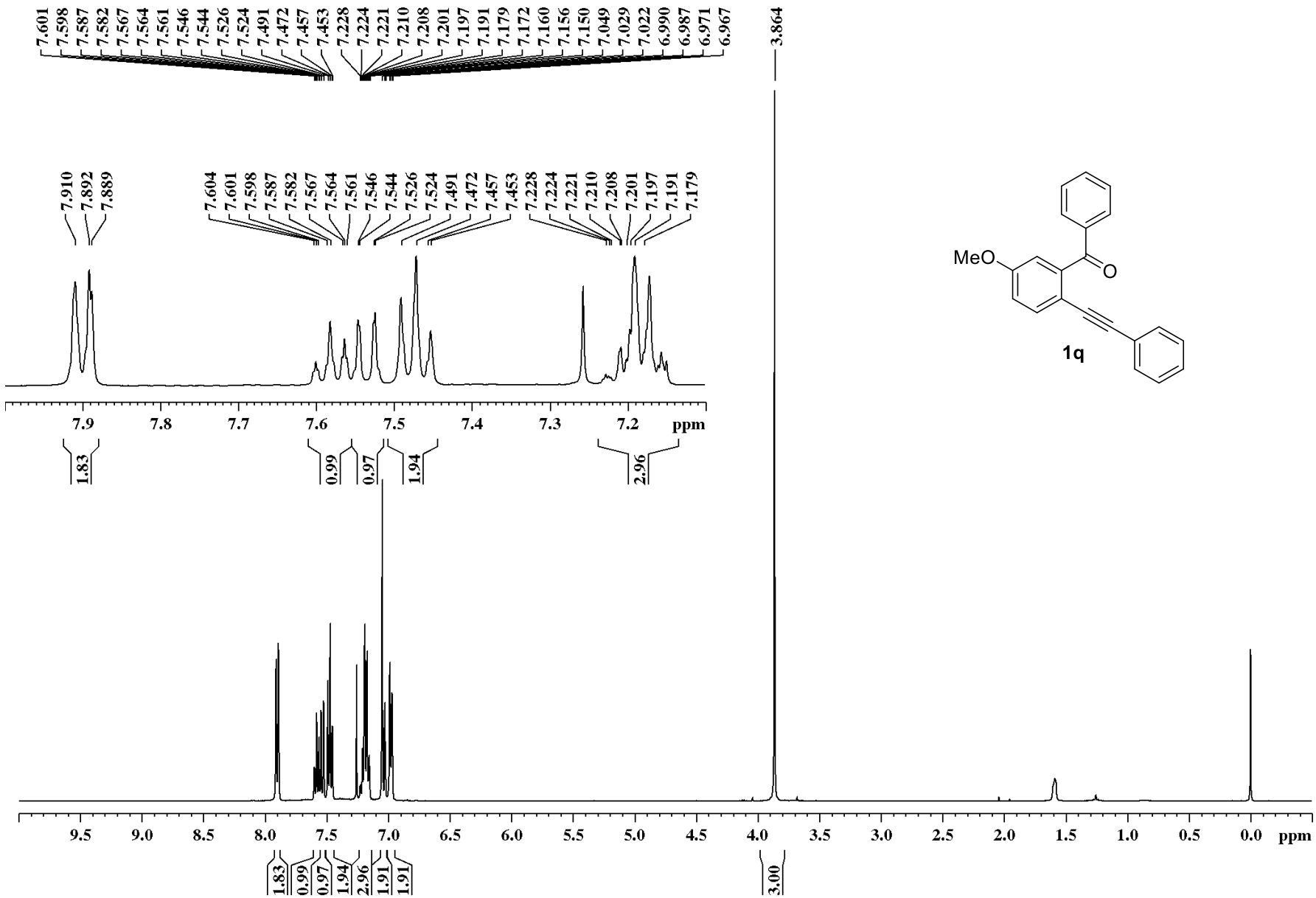
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7.561
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7.526
7.524
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7.453
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7.224
7.221
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7.208
7.201
7.197
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7.8
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7.6
7.5
7.4
7.3
7.2
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O
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6.0
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4.0
3.5
3.0
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2.0
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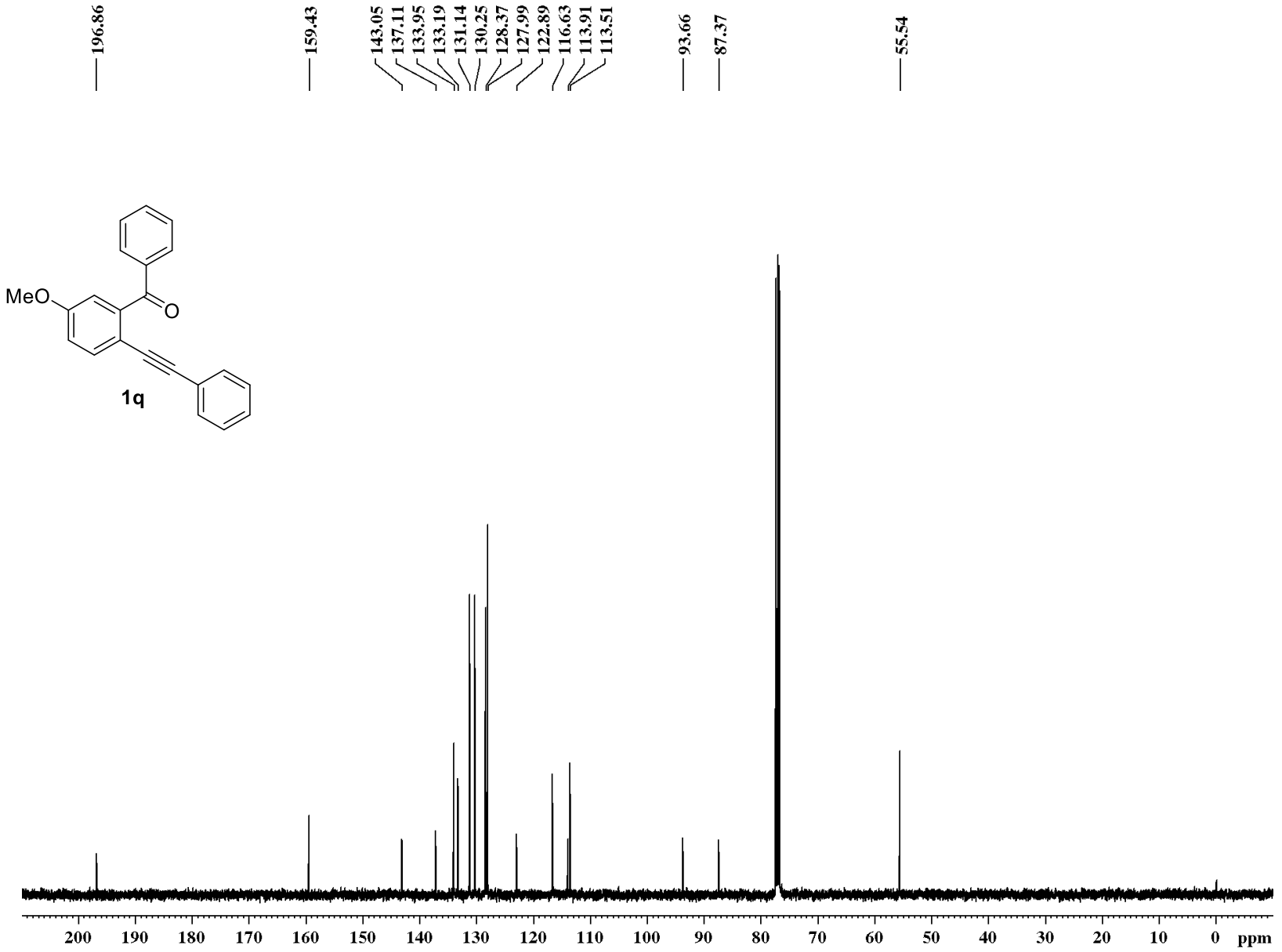
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113.51
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87.37
55.54
MeO
O
1q
200
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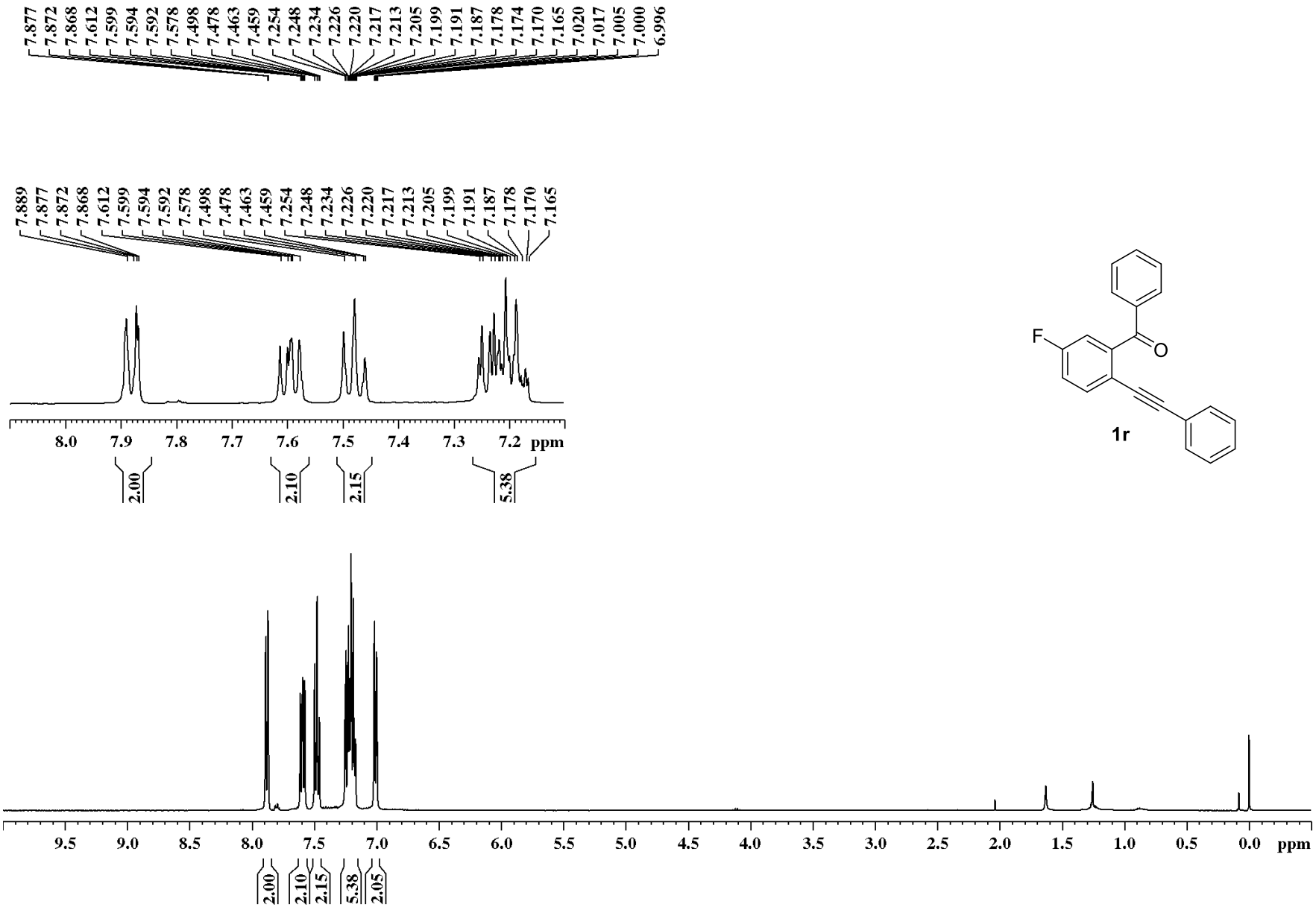
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7.234
7.226
7.220
7.217
7.213
7.205
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7.191
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7.174
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7.165
7.020
7.017
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7.594
7.592
7.578
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7.220
7.217
7.213
7.205
7.199
7.191
7.187
7.178
7.170
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7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm
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2.10
2.15
5.38
F
O
1r
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
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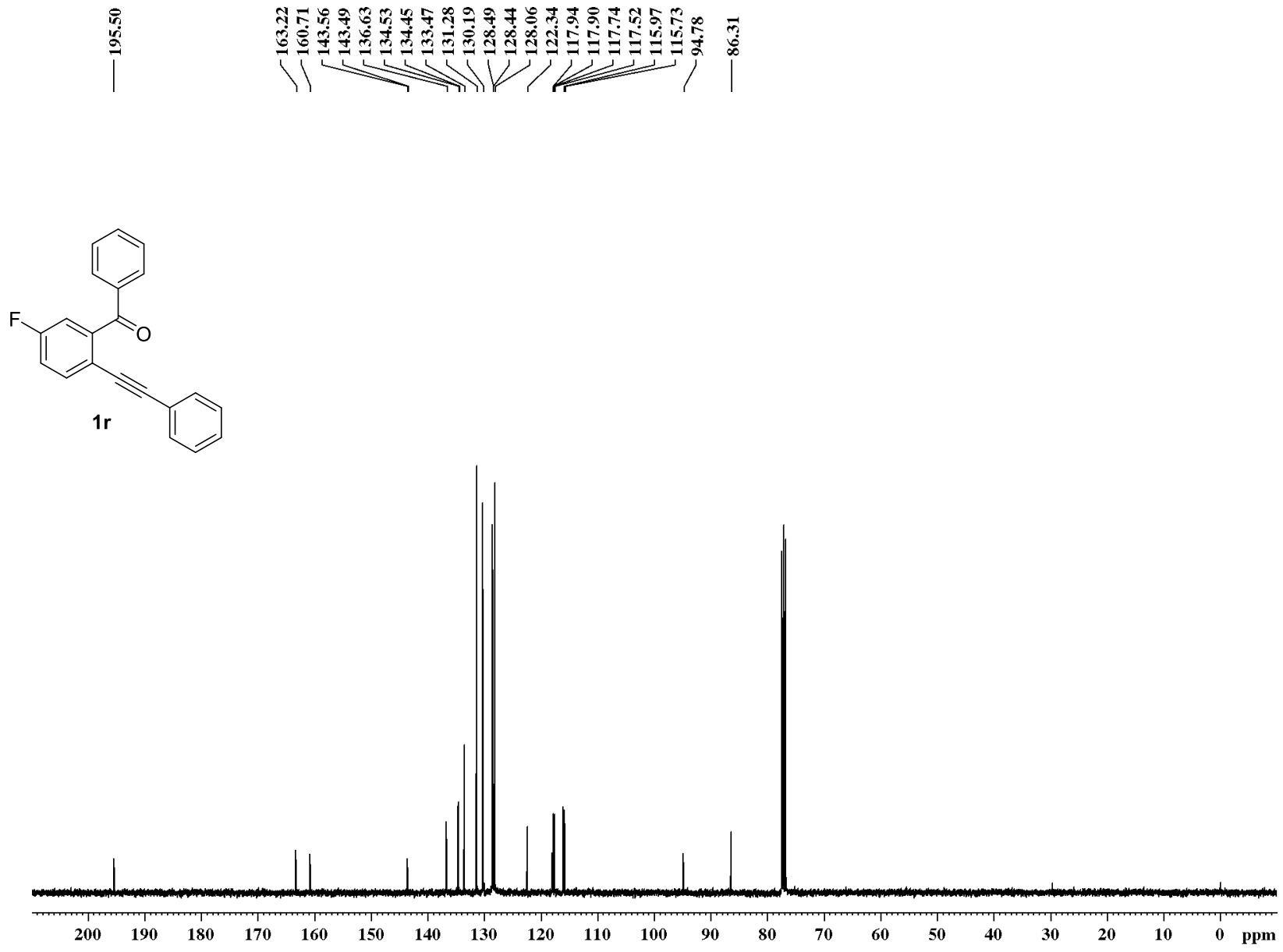
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128.06
122.34
117.94
117.90
117.74
117.52
115.97
115.73
94.78
86.31
F
O
1r
200
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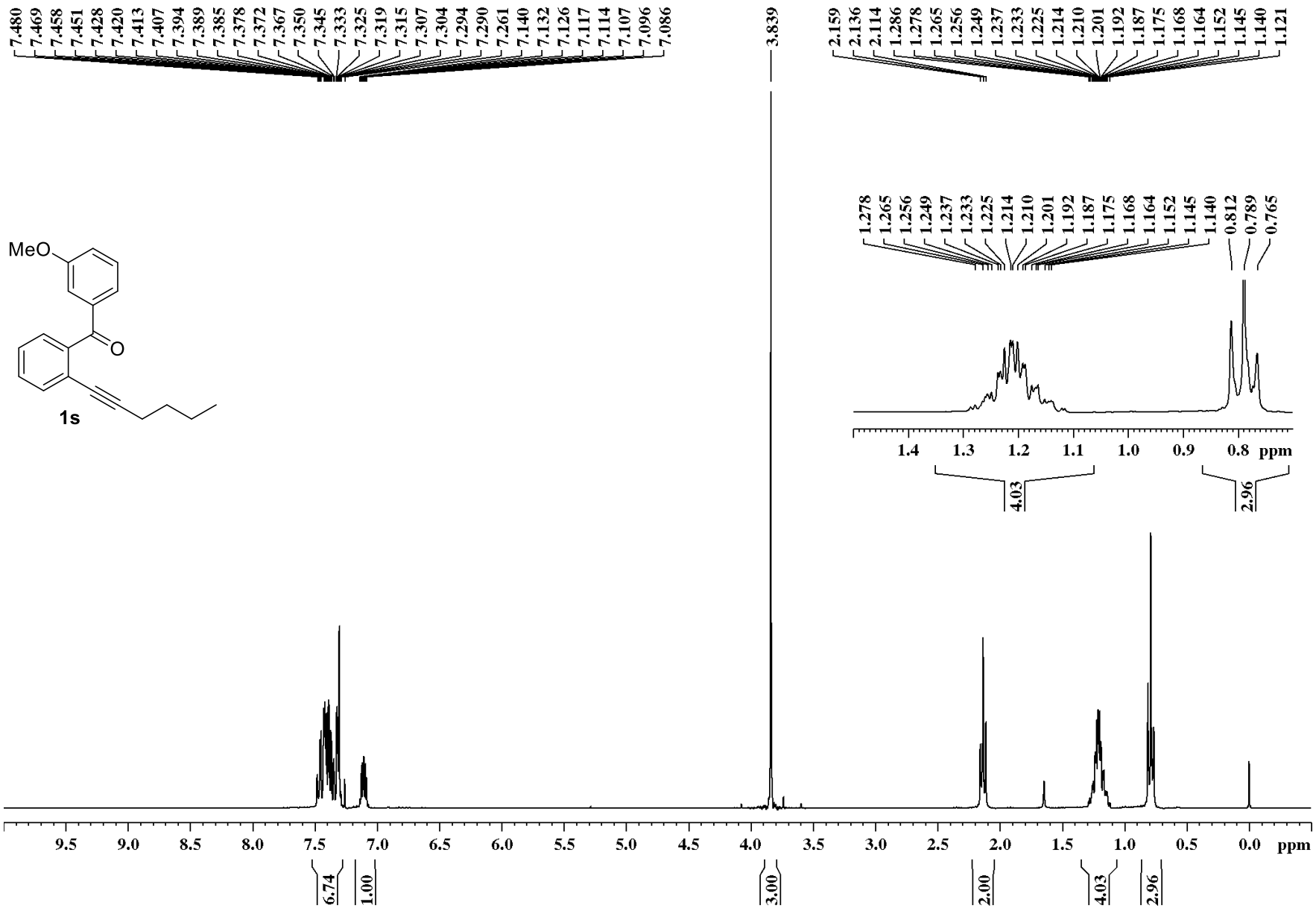
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7.367
7.350
7.345
7.333
7.325
7.319
7.315
7.307
7.304
7.294
7.290
7.261
7.140
7.132
7.126
7.117
7.114
7.107
7.096
7.086
3.839
2.159
2.136
2.114
1.286
1.278
1.265
1.256
1.249
1.237
1.233
1.225
1.214
1.210
1.201
1.192
1.187
1.175
1.168
1.164
1.152
1.145
1.140
1.121
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0.789
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O
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ppm
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2.00
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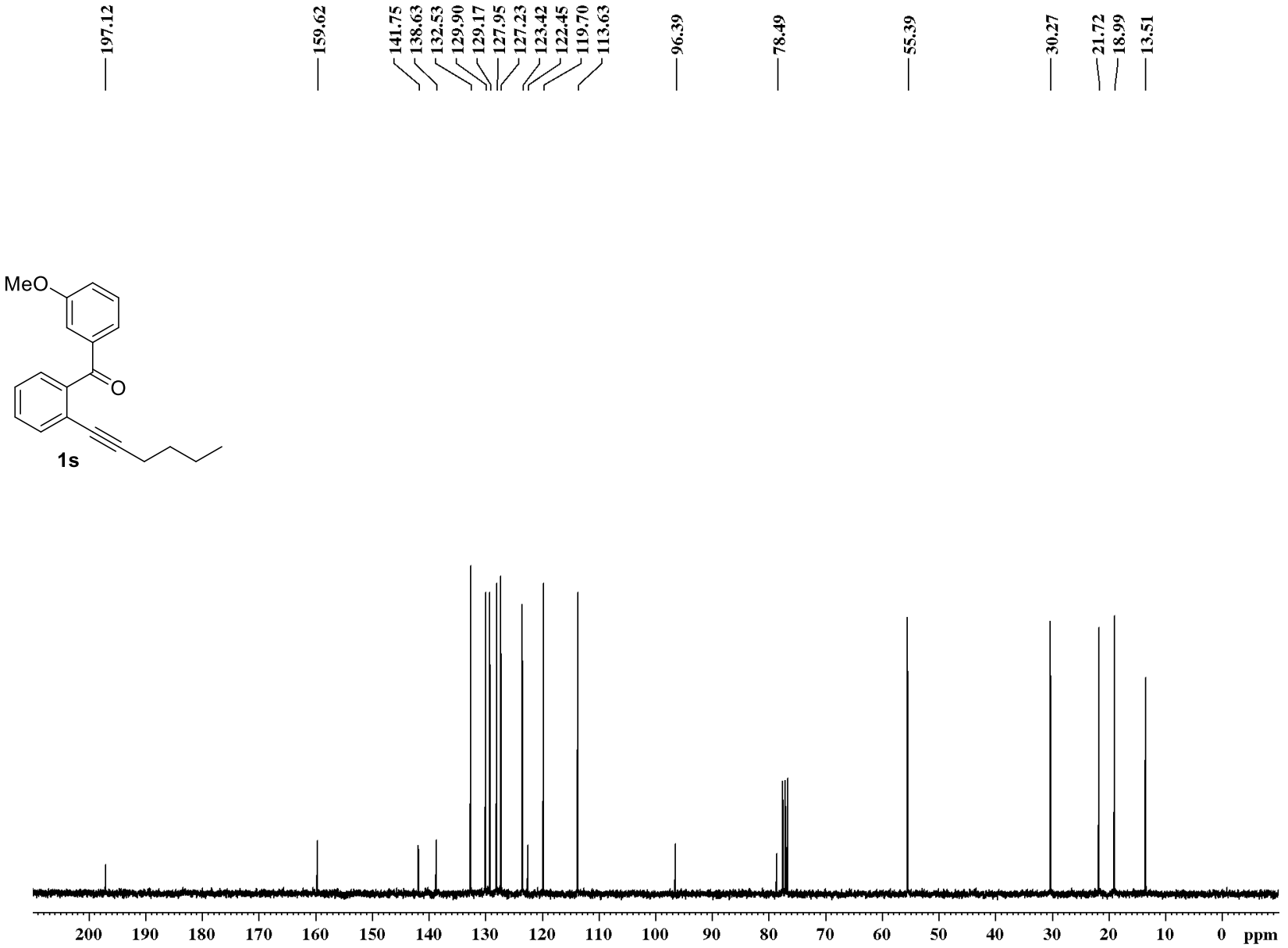
2.96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123.42
122.45
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113.63
96.39
78.49
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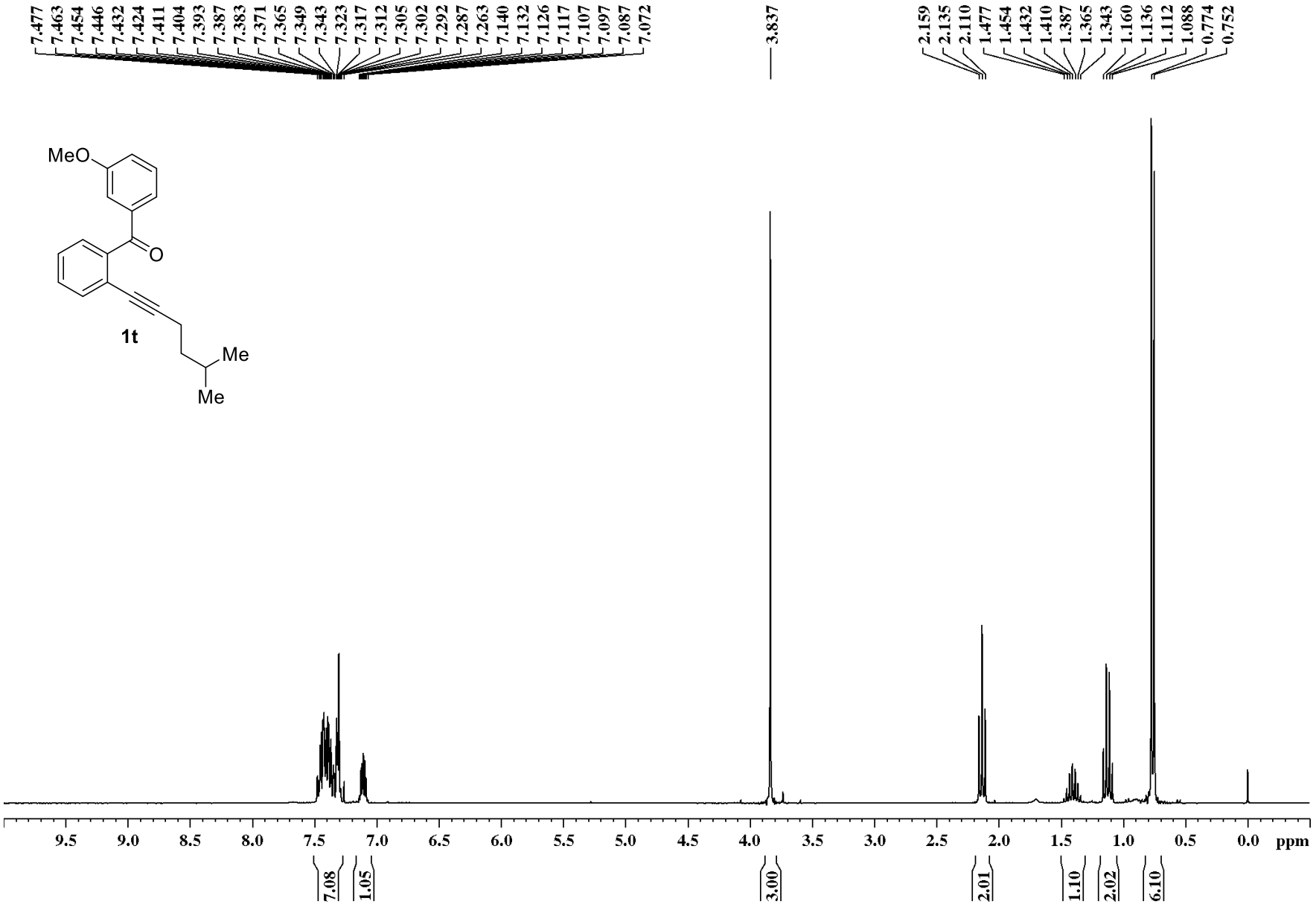
18.99
13.51
MeO
O
1s
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.387
7.383
7.371
7.365
7.349
7.343
7.323
7.317
7.312
7.305
7.302
7.292
7.287
7.263
7.140
7.132
7.126
7.117
7.107
7.097
7.087
7.072
3.837
2.159
2.135
2.110
1.477
1.454
1.432
1.410
1.387
1.365
1.343
1.160
1.136
1.112
1.088
0.774
0.752
MeO
O
1t
Me
Me
9.5
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
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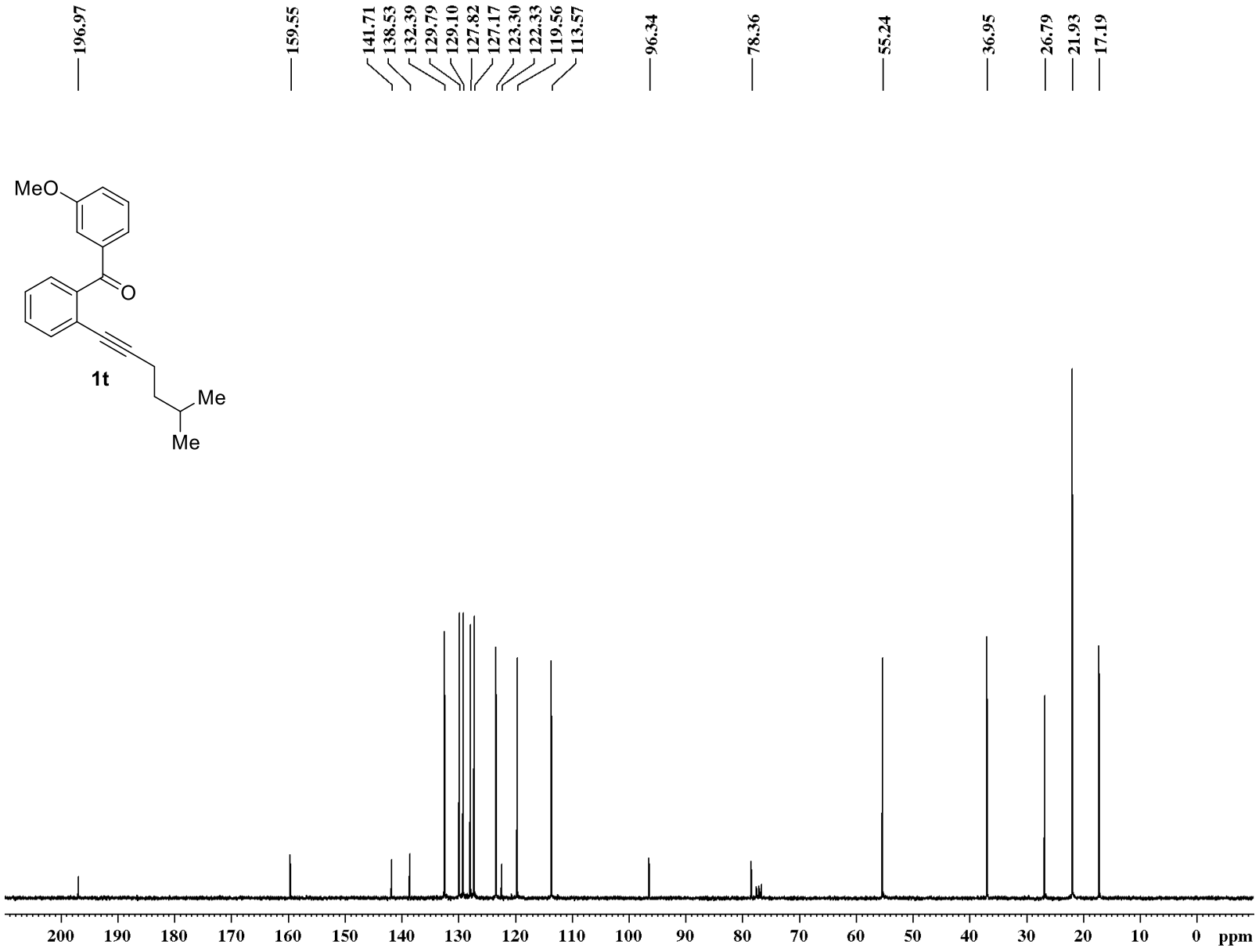
1.10
2.02
6.10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96.34
78.36
55.24
36.95
26.79
21.93
17.19
MeO
O
1t
Me
Me
200
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20
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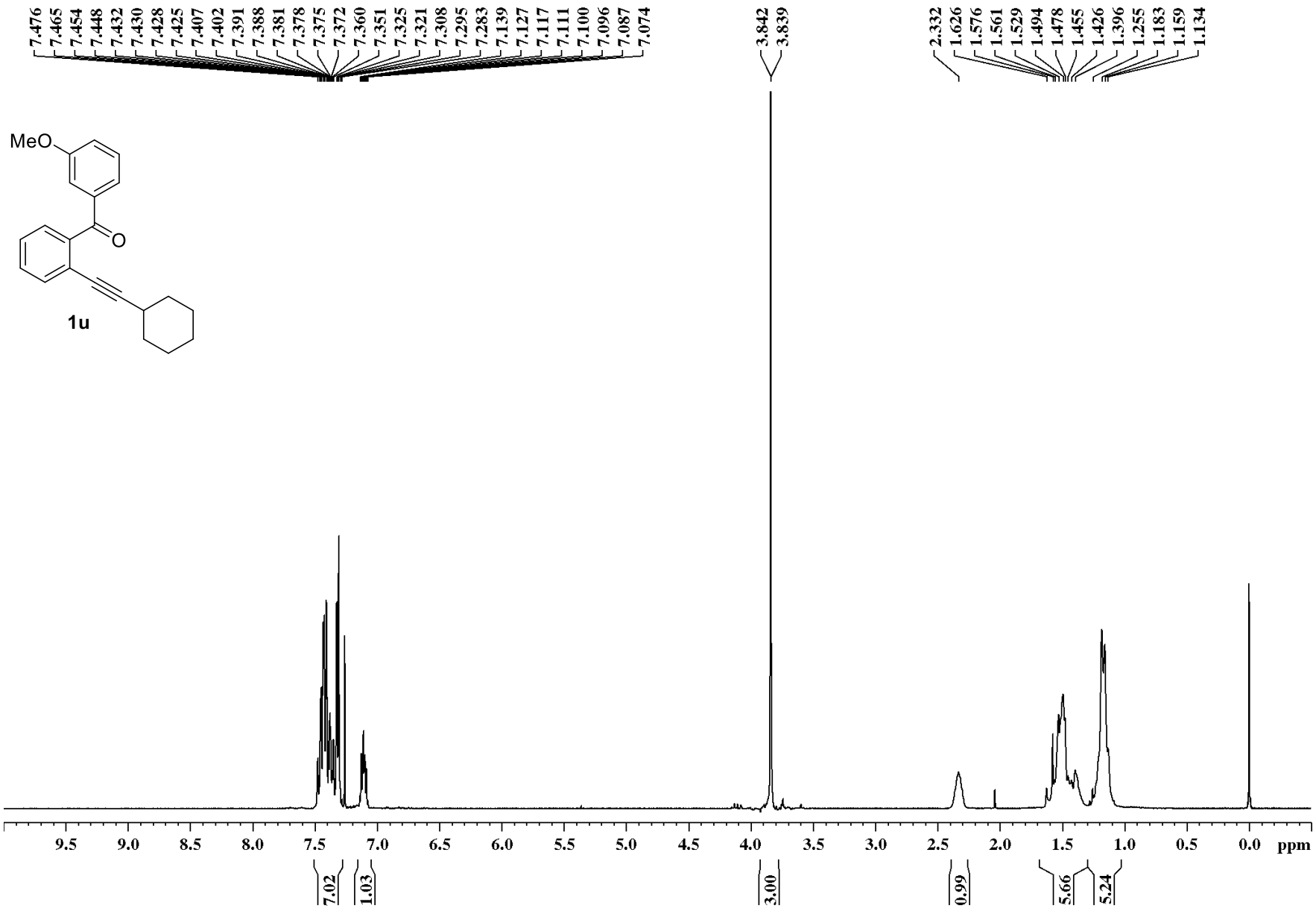
ppm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7.378
7.375
7.372
7.360
7.351
7.325
7.321
7.308
7.295
7.283
7.139
7.127
7.117
7.111
7.100
7.096
7.087
7.074
3.842
3.839
2.332
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1.576
1.561
1.529
1.494
1.478
1.455
1.426
1.396
1.255
1.183
1.159
1.134
MeO
O
1u
9.5
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
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1.0
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1.03
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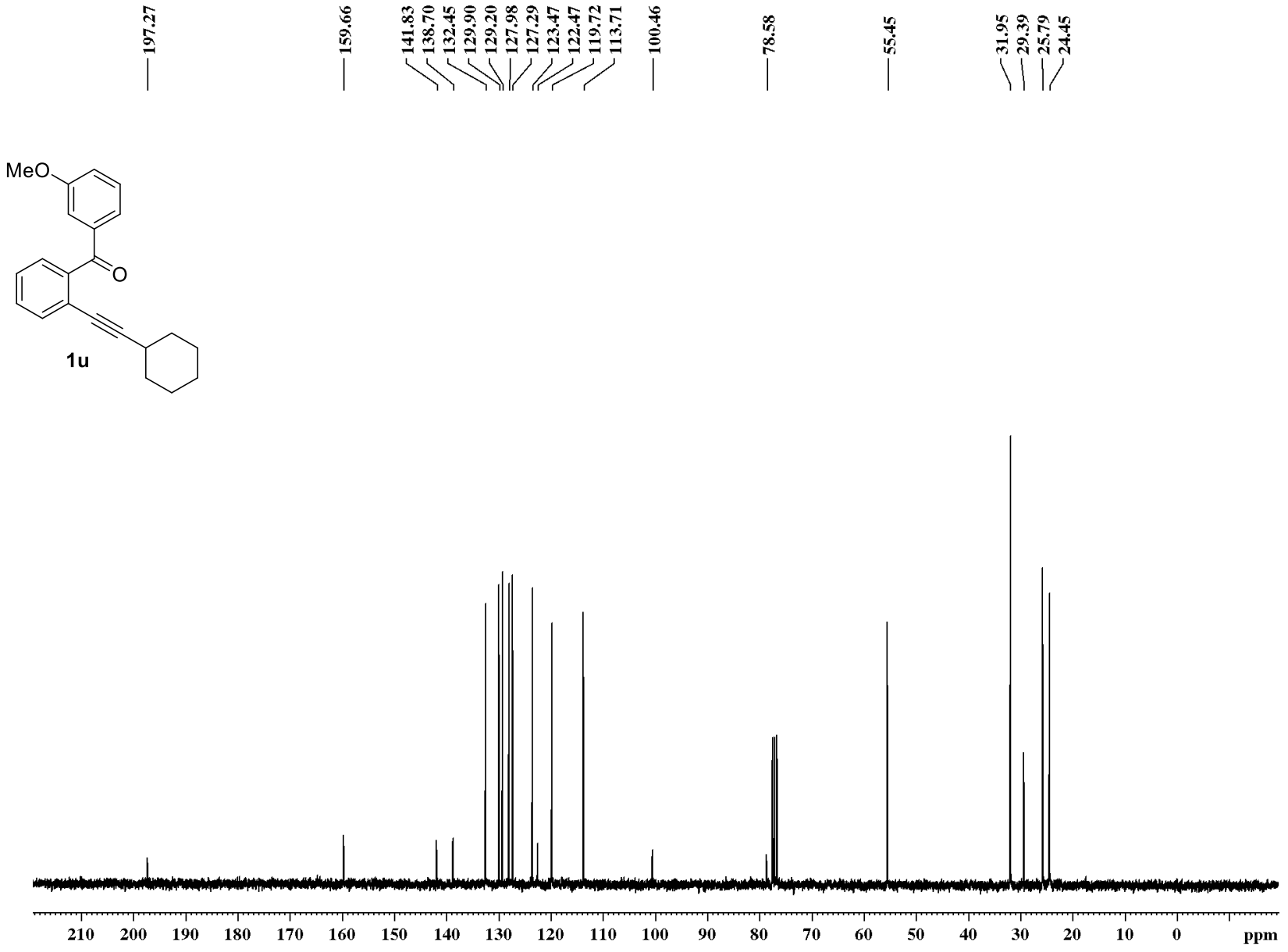
5.24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100.46
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55.45
31.95
29.39
25.79
24.45
MeO
O
1u
210
200
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140
130
120
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60
50
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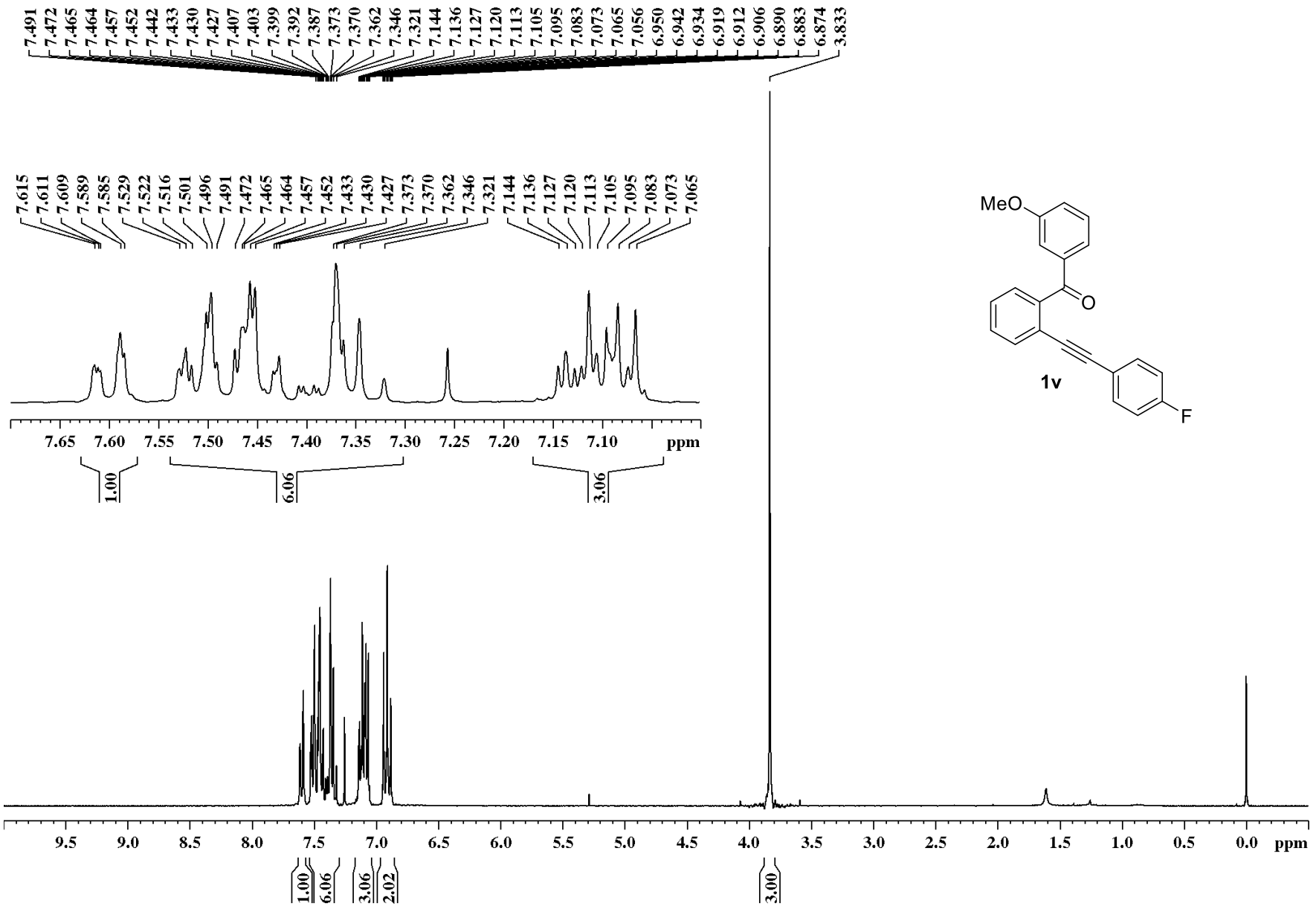
ppm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7.392
7.387
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7.370
7.362
7.346
7.321
7.144
7.136
7.127
7.120
7.113
7.105
7.095
7.083
7.073
7.065
7.056
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6.942
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6.912
6.906
6.890
6.883
6.874
3.833
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7.611
7.609
7.589
7.585
7.529
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7.516
7.501
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7.491
7.472
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7.464
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7.433
7.430
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7.370
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7.346
7.321
7.144
7.136
7.127
7.120
7.113
7.105
7.095
7.083
7.073
7.065
7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 ppm
1.00
6.06
3.06
9.5 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 0.0 ppm
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2.02
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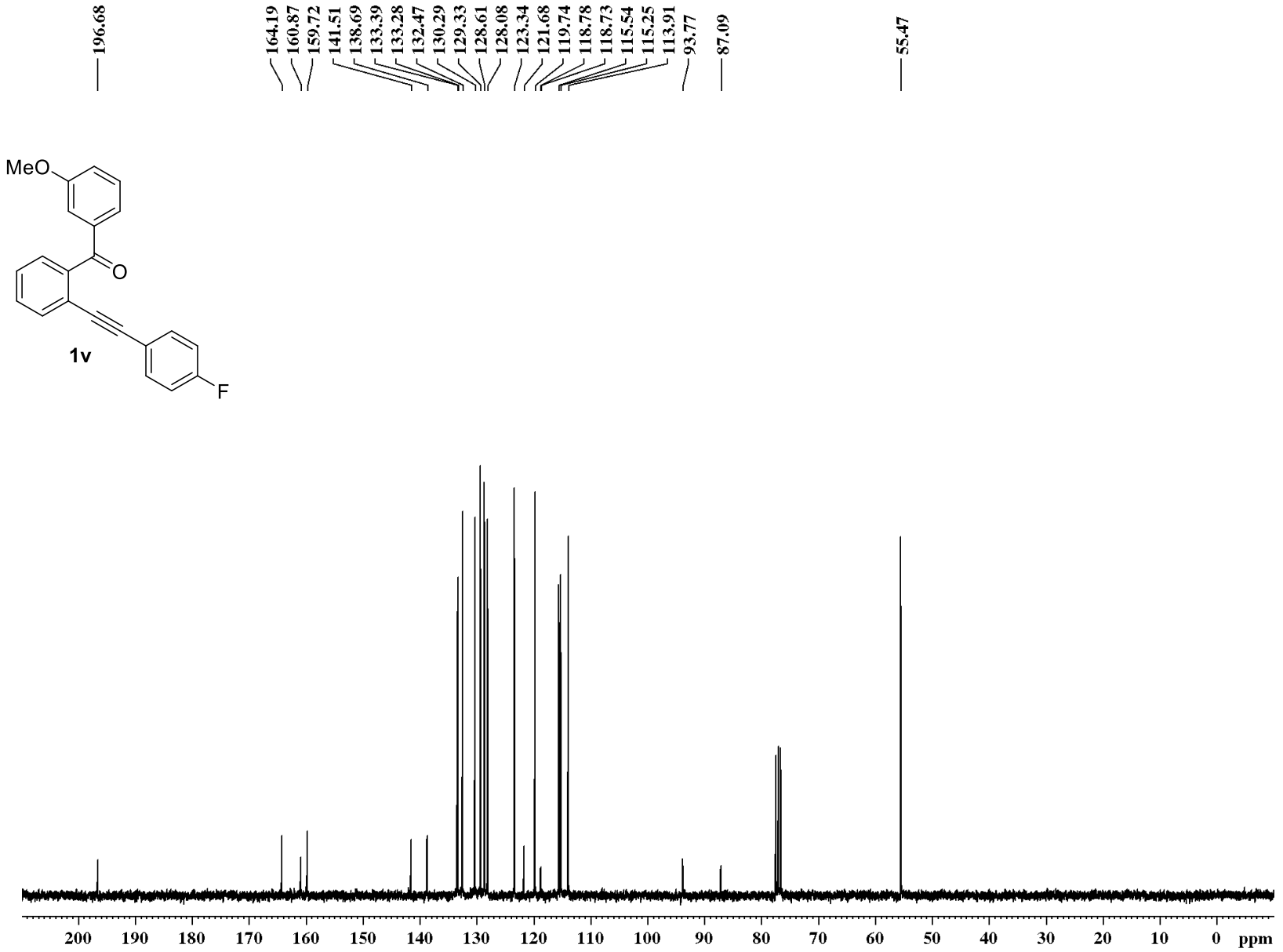
MeO
O
F
1v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121.68
119.74
118.78
118.73
115.54
115.25
113.91
93.77
87.09
55.47
MeO
O
F
1v
200
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140
130
120
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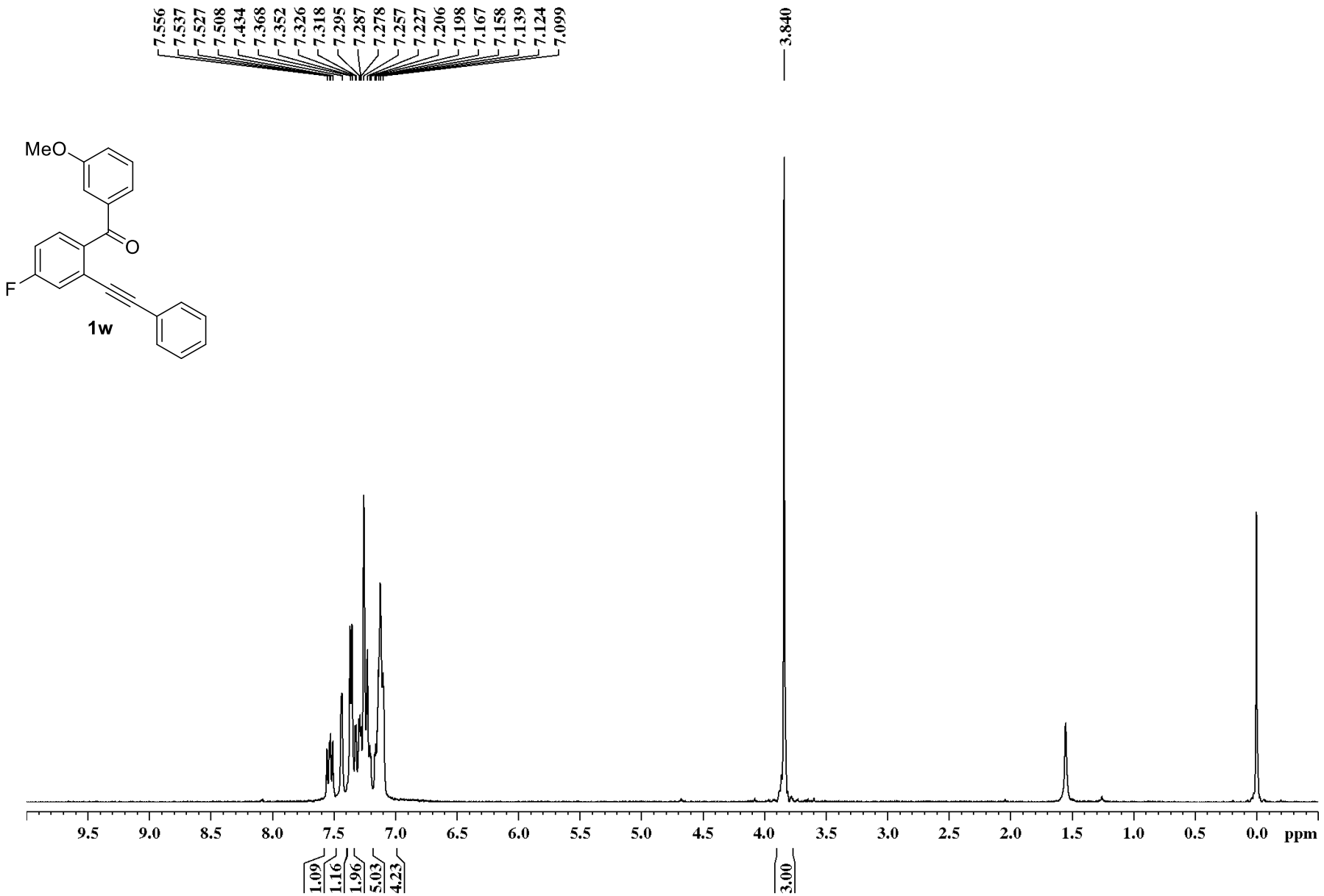
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7.167
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7.139
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F
1w
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6.5
6.0
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4.5
4.0
3.5
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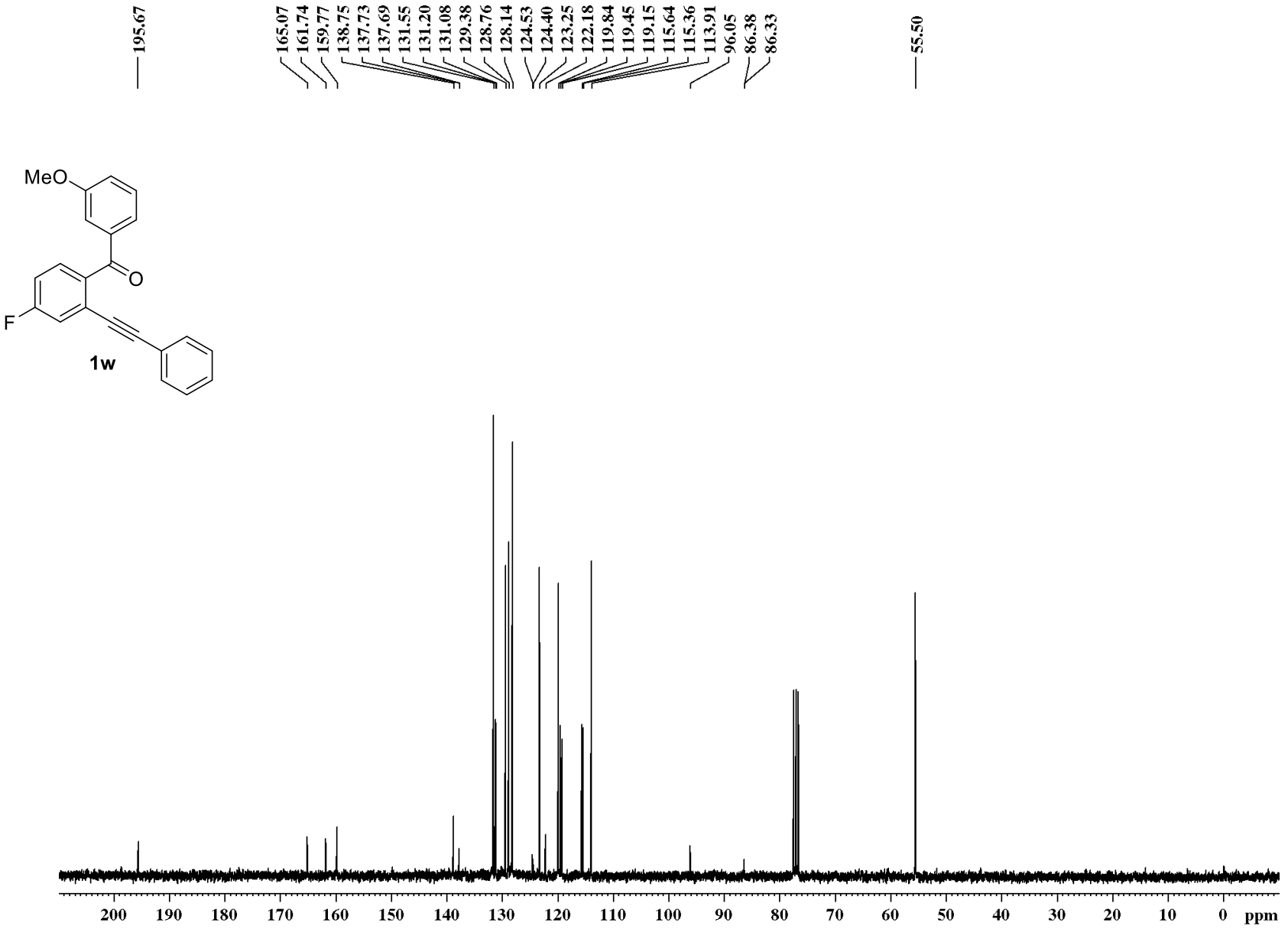
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123.25
122.18
119.84
119.45
119.15
115.64
115.36
113.91
96.05
86.38
86.33
55.50
MeO
O
F
1w
200
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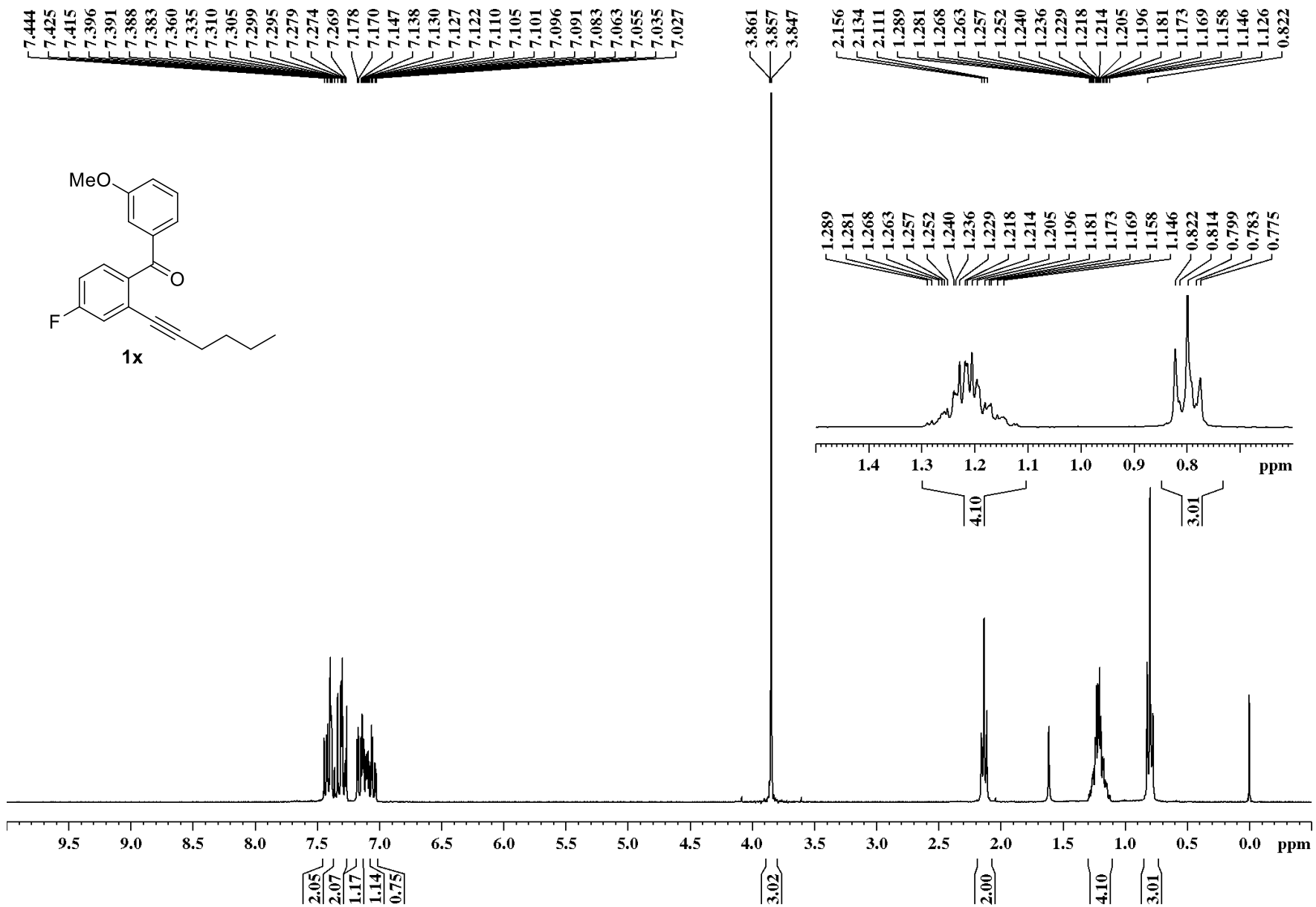
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7.127
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MeO
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F
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1.1
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0.9
0.8
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6.0
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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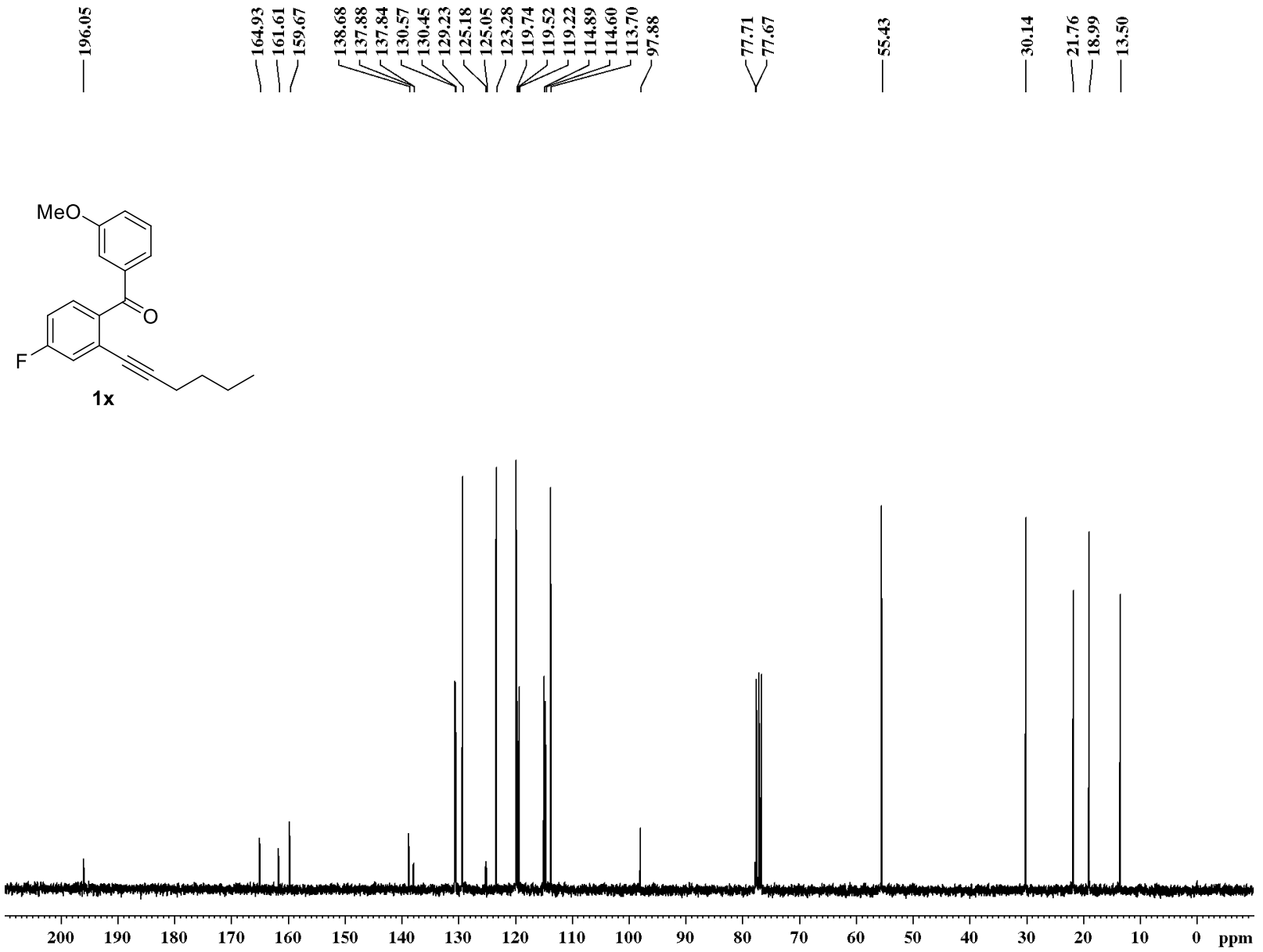
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123.28
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119.52
119.22
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114.60
113.70
97.88
77.71
77.67
55.43
30.14
21.76
18.99
13.50
MeO
O
F
1x
200
190
180
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140
130
120
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100
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80
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60
50
40
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20
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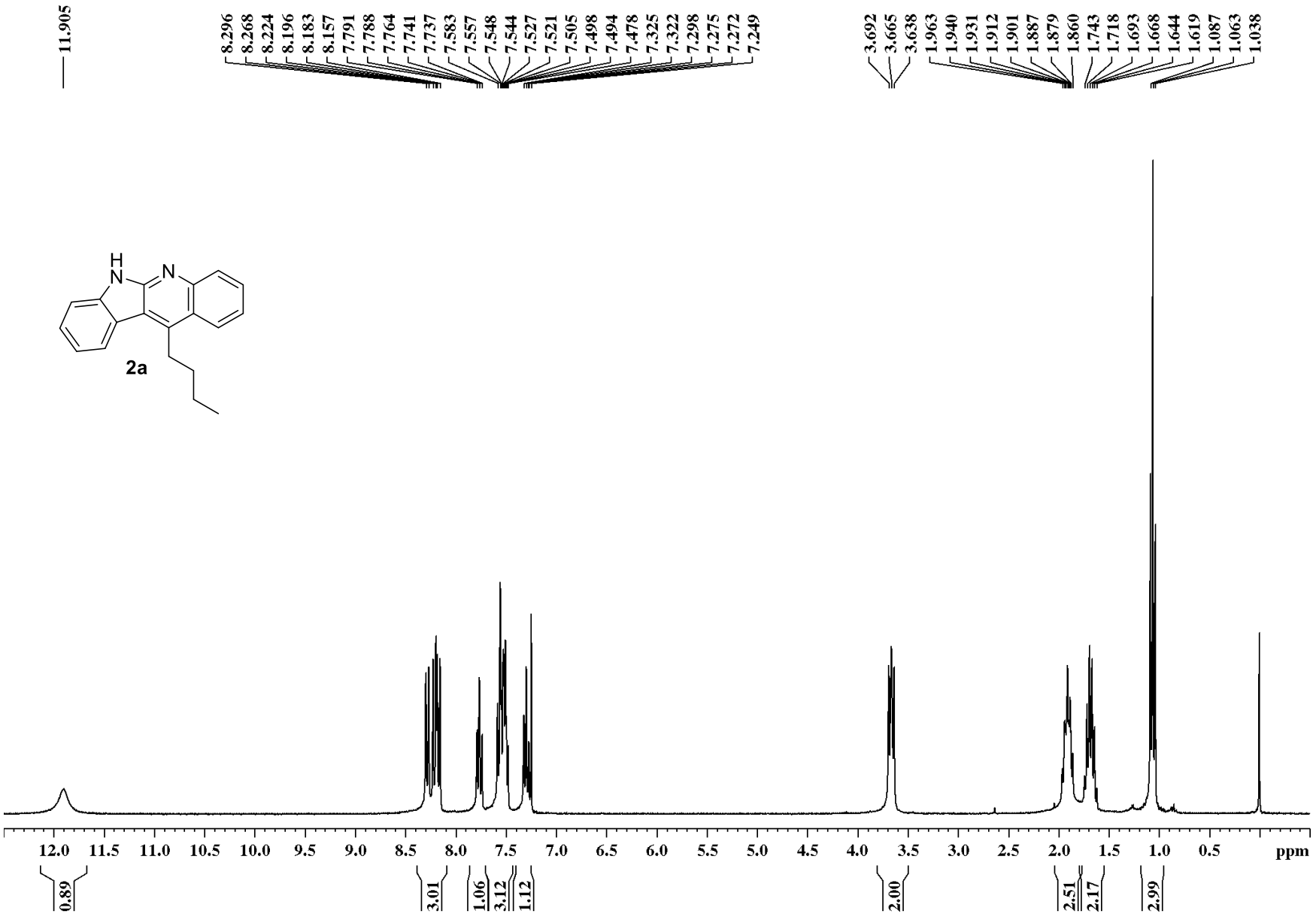
0
ppm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7.737
7.583
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7.527
7.521
7.505
7.498
7.494
7.478
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7.322
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1.940
1.931
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1.901
1.887
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1.668
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1.619
1.087
1.063
1.038
H
N
N
2a
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10.5
10.0
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9.0
8.5
8.0
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6.5
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5.5
5.0
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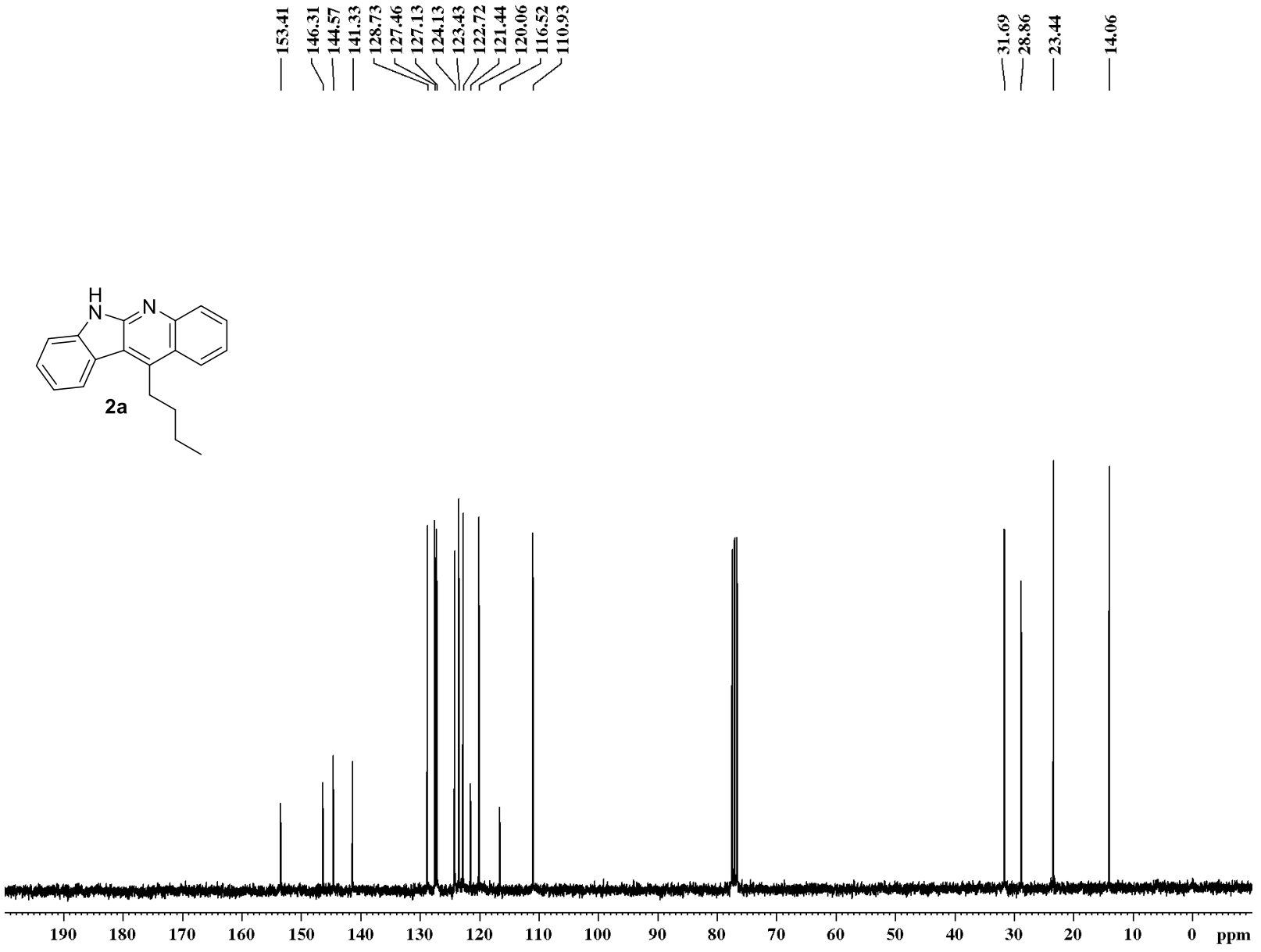
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31.69
28.86
23.44
14.06
H
N
N
2a
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60
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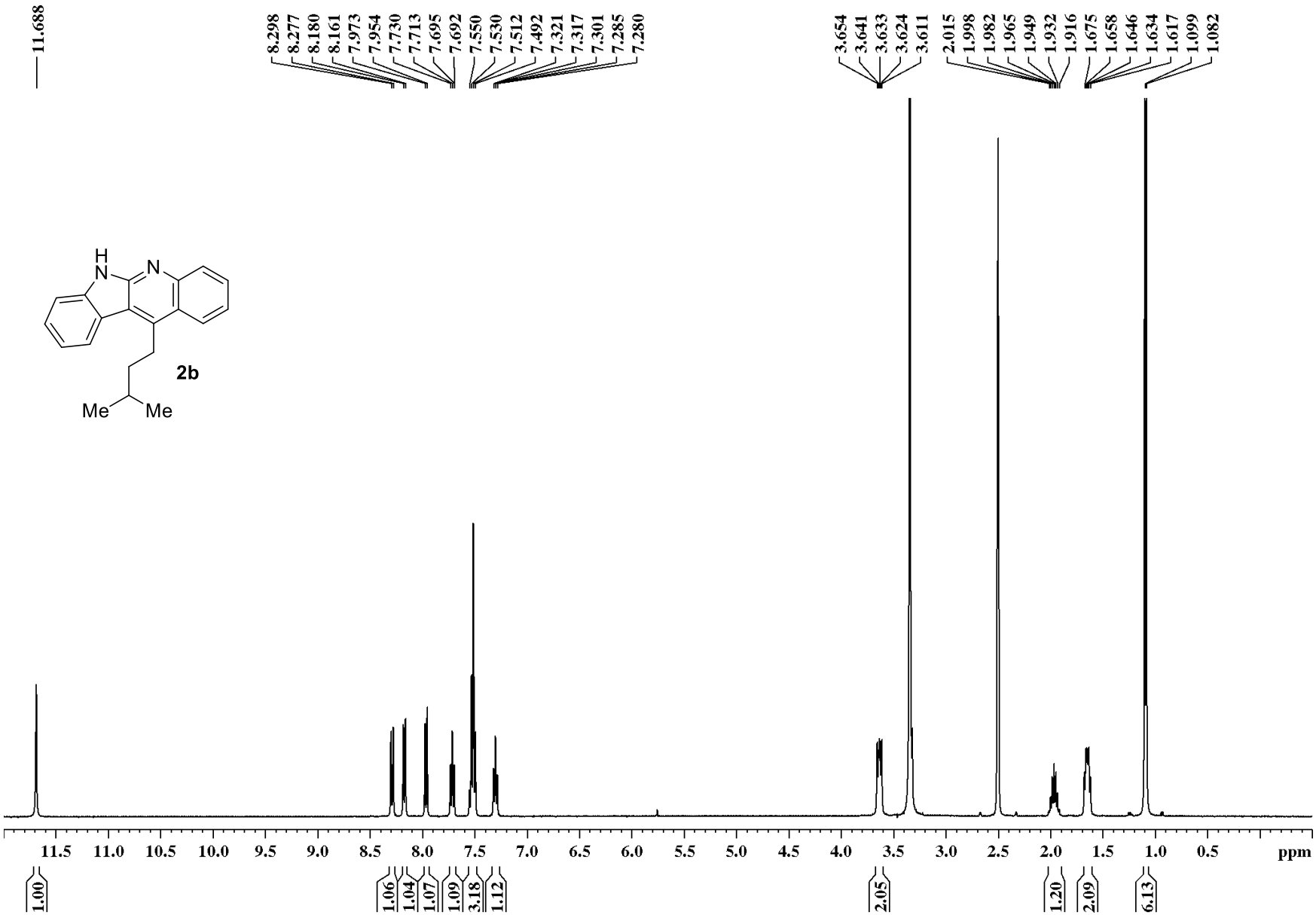
ppm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7.301
7.285
7.280
3.654
3.641
3.633
3.624
3.611
2.015
1.998
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1.965
1.949
1.932
1.916
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1.646
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1.617
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1.082
H
N
N
2b
Me
Me
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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
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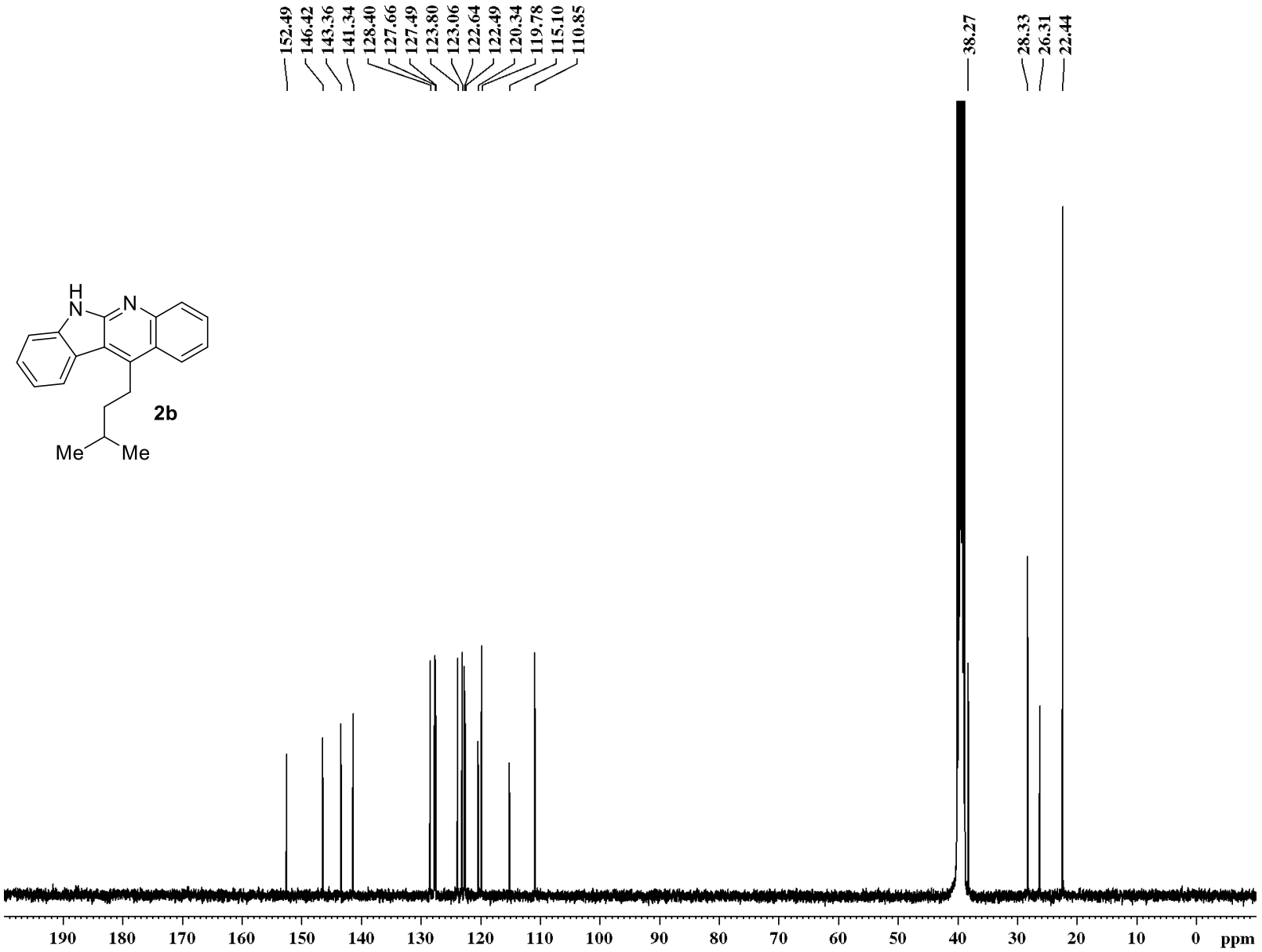
1.04
1.07
1.09
3.18
1.12
2.05
1.20
2.09
6.13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119.78
115.10
110.85
38.27
28.33
26.31
22.44
H
N
N
2b
Me
Me
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140
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100
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80
70
60
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20
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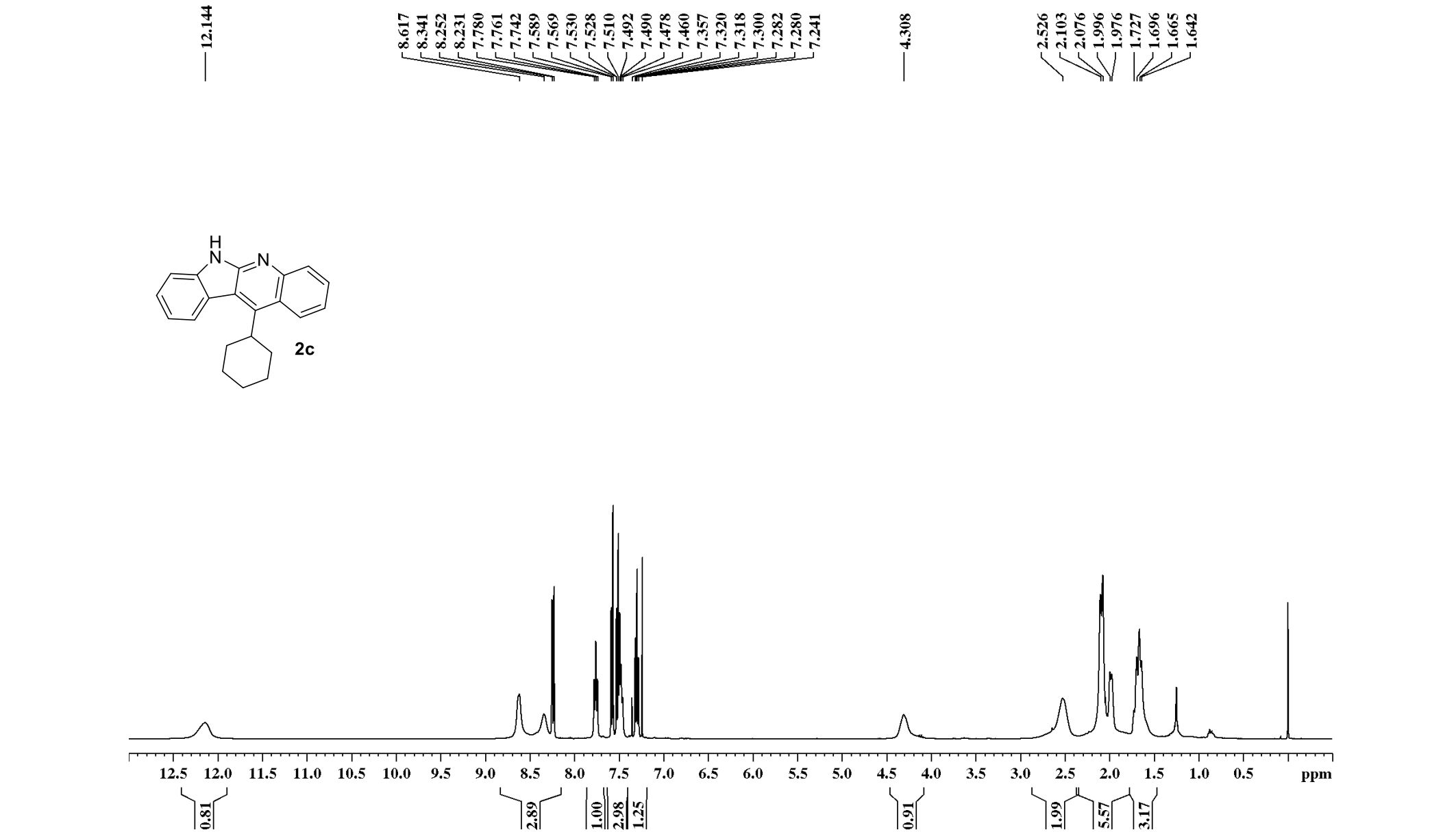
0
ppm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7.569
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7.528
7.510
7.492
7.490
7.478
7.460
7.357
7.320
7.318
7.300
7.282
7.280
7.241
4.308
2.526
2.103
2.076
1.996
1.976
1.727
1.696
1.665
1.642
H
N
N
2c
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 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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1.25
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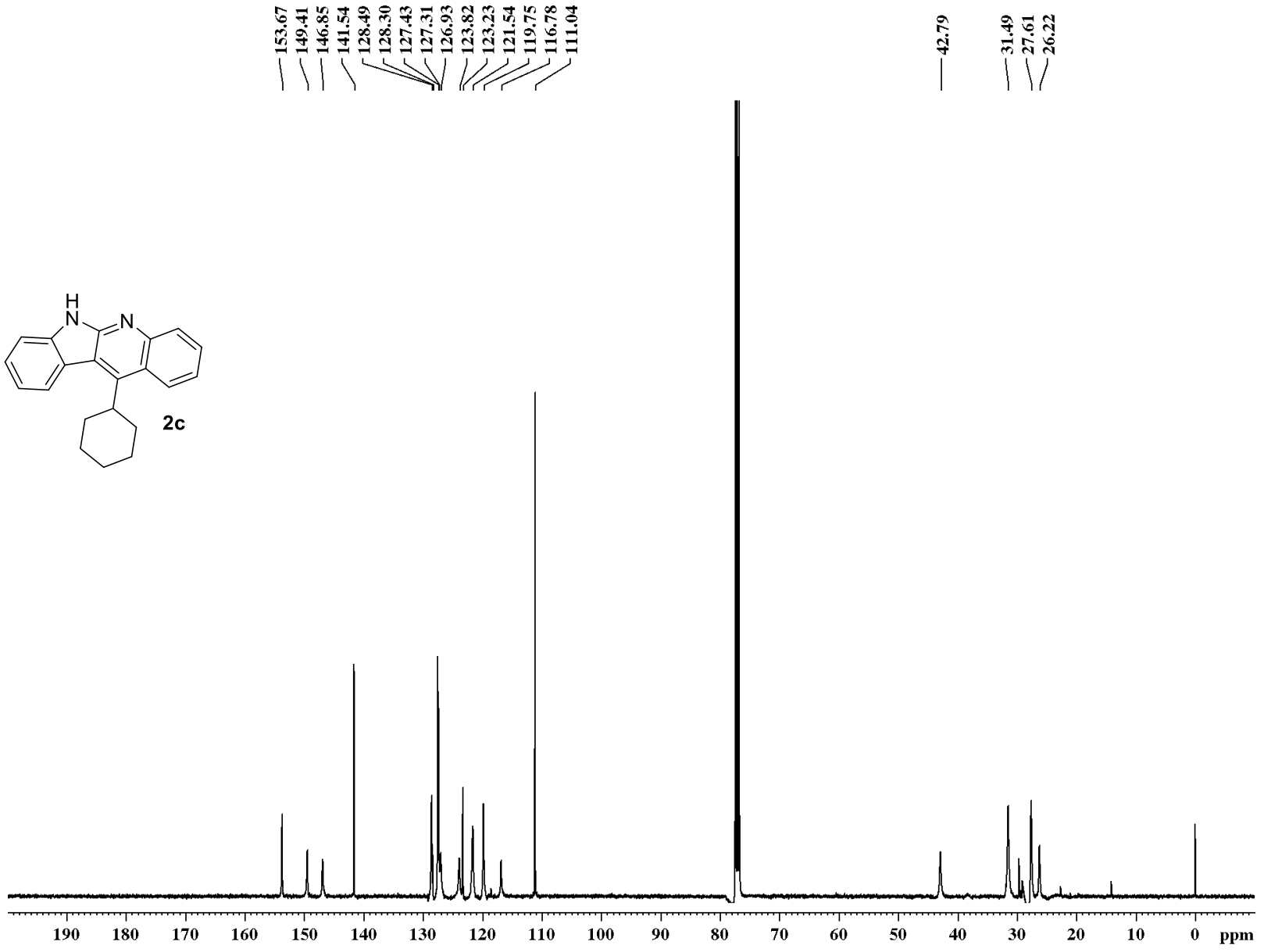
1.99
5.57
3.17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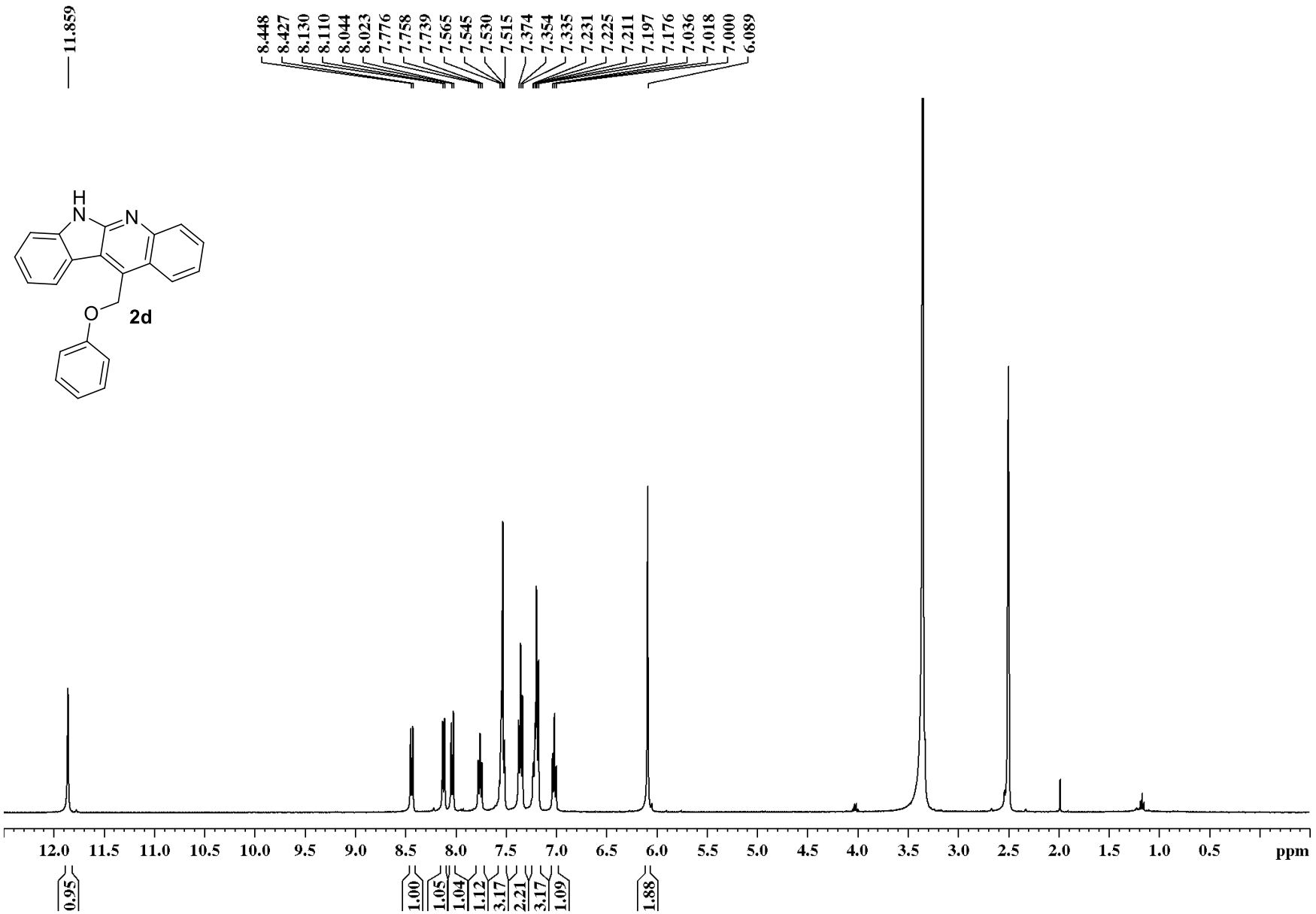
123.23
121.54
119.75
116.78
111.04
42.79
31.49
27.61
26.22
H
N
N
2c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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8.023
7.776
7.758
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7.565
7.545
7.530
7.515
7.374
7.354
7.335
7.231
7.225
7.211
7.197
7.176
7.036
7.018
7.000
6.089
H
N
N
O
2d
12.0
11.5
11.0
10.5
10.0
9.5
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
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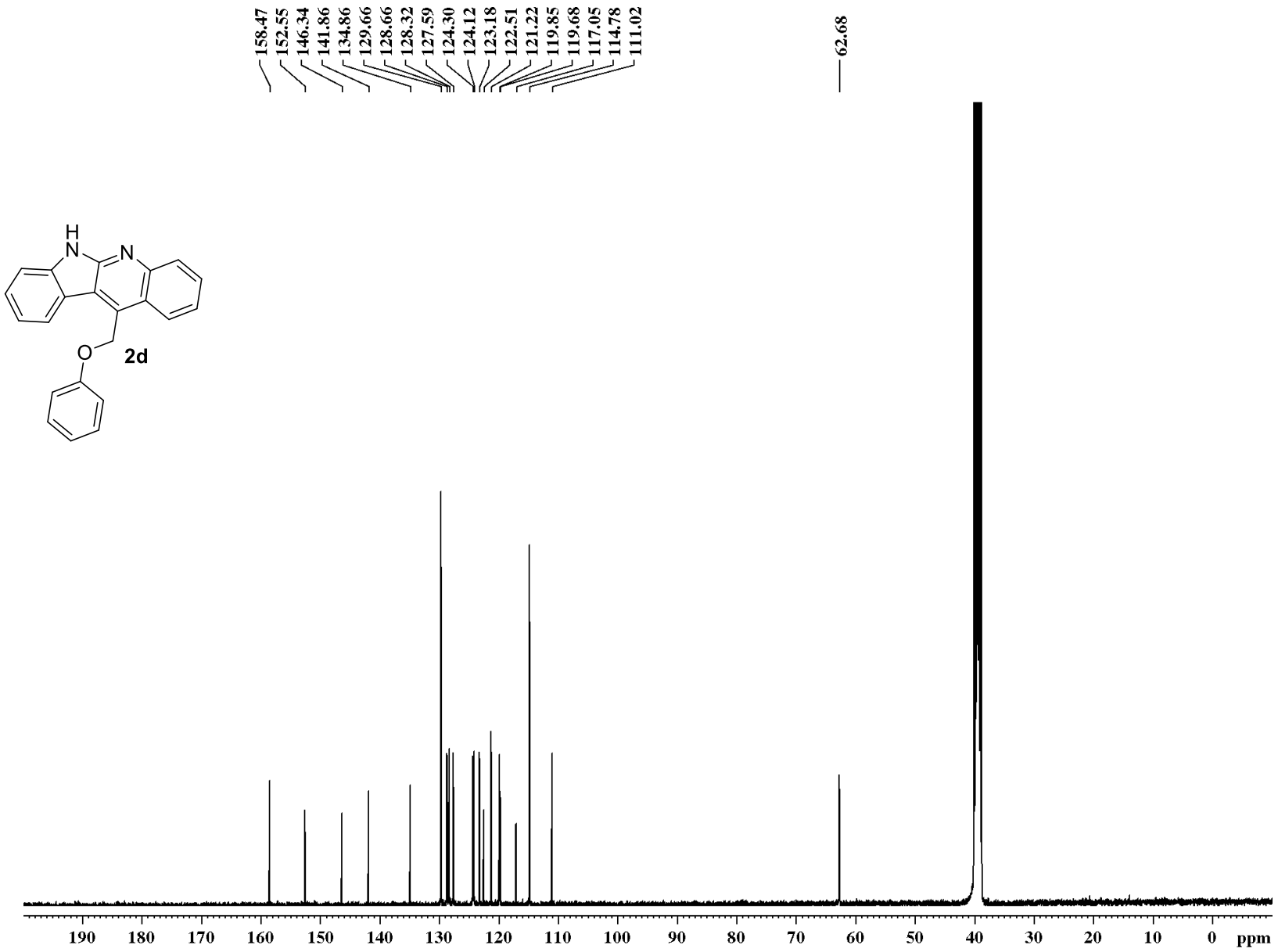
3.17
1.09
1.88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128.32
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124.12
123.18
122.51
121.22
119.85
119.68
117.05
114.78
111.02
62.68
H
N
N
O
2d
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140
130
120
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100
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50
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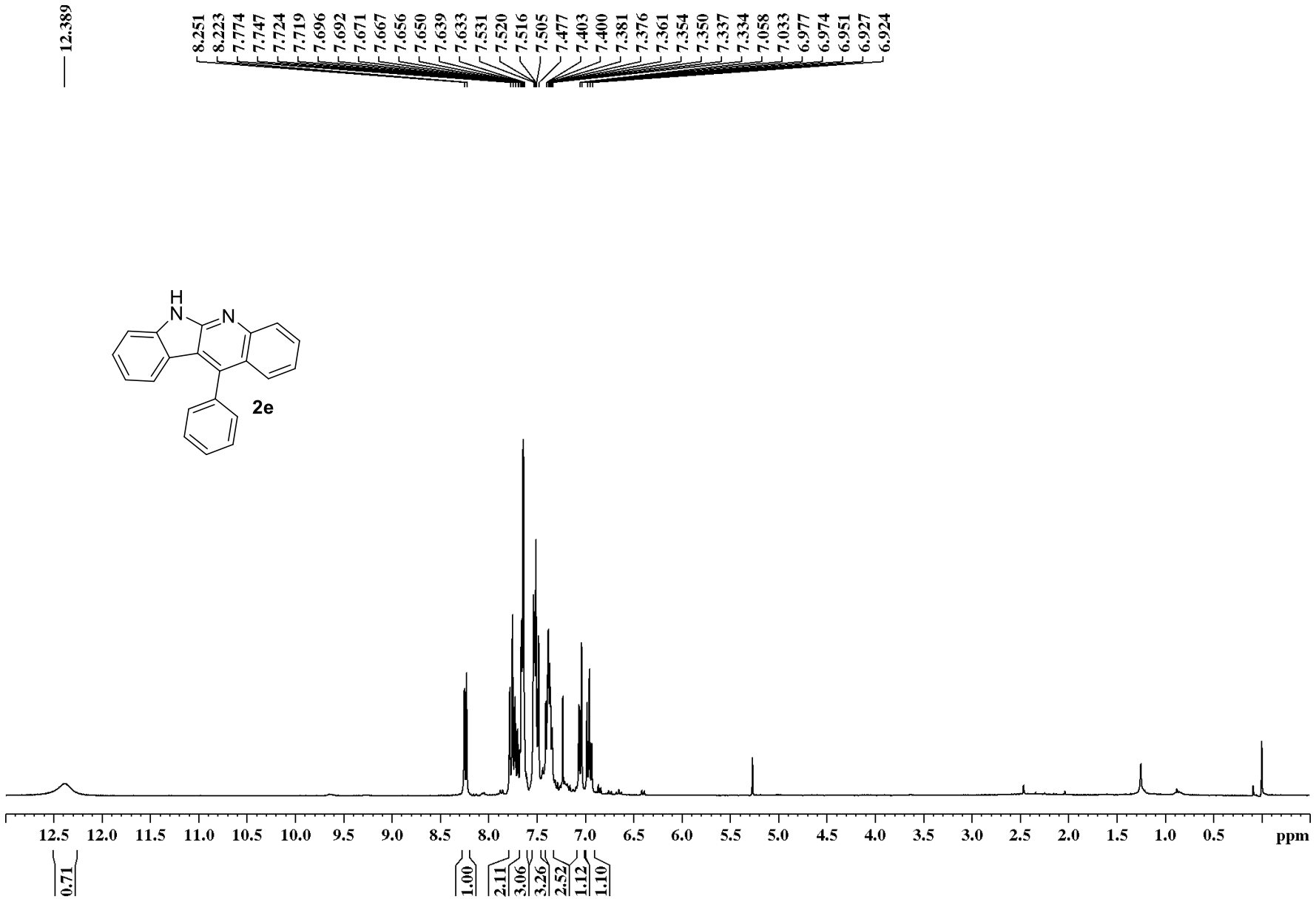
20
10
0
ppm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7.724
7.719
7.696
7.692
7.671
7.667
7.656
7.650
7.639
7.633
7.531
7.520
7.516
7.505
7.477
7.403
7.400
7.381
7.376
7.361
7.354
7.350
7.337
7.334
7.058
7.033
6.977
6.974
6.951
6.927
6.924
H
N
N
2e
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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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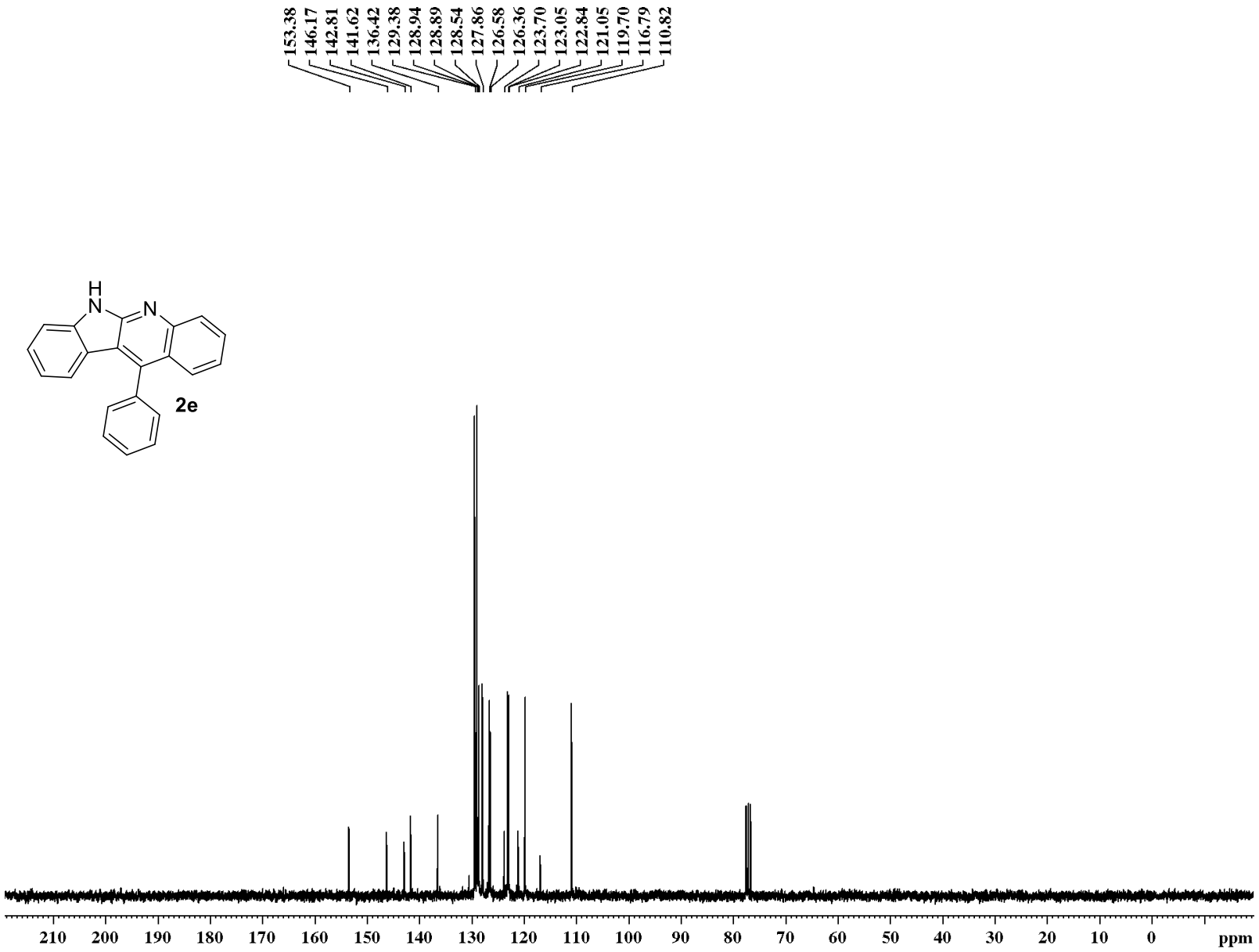
2.11
3.06
3.26
2.52
1.12
1.10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142.81
141.62
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129.38
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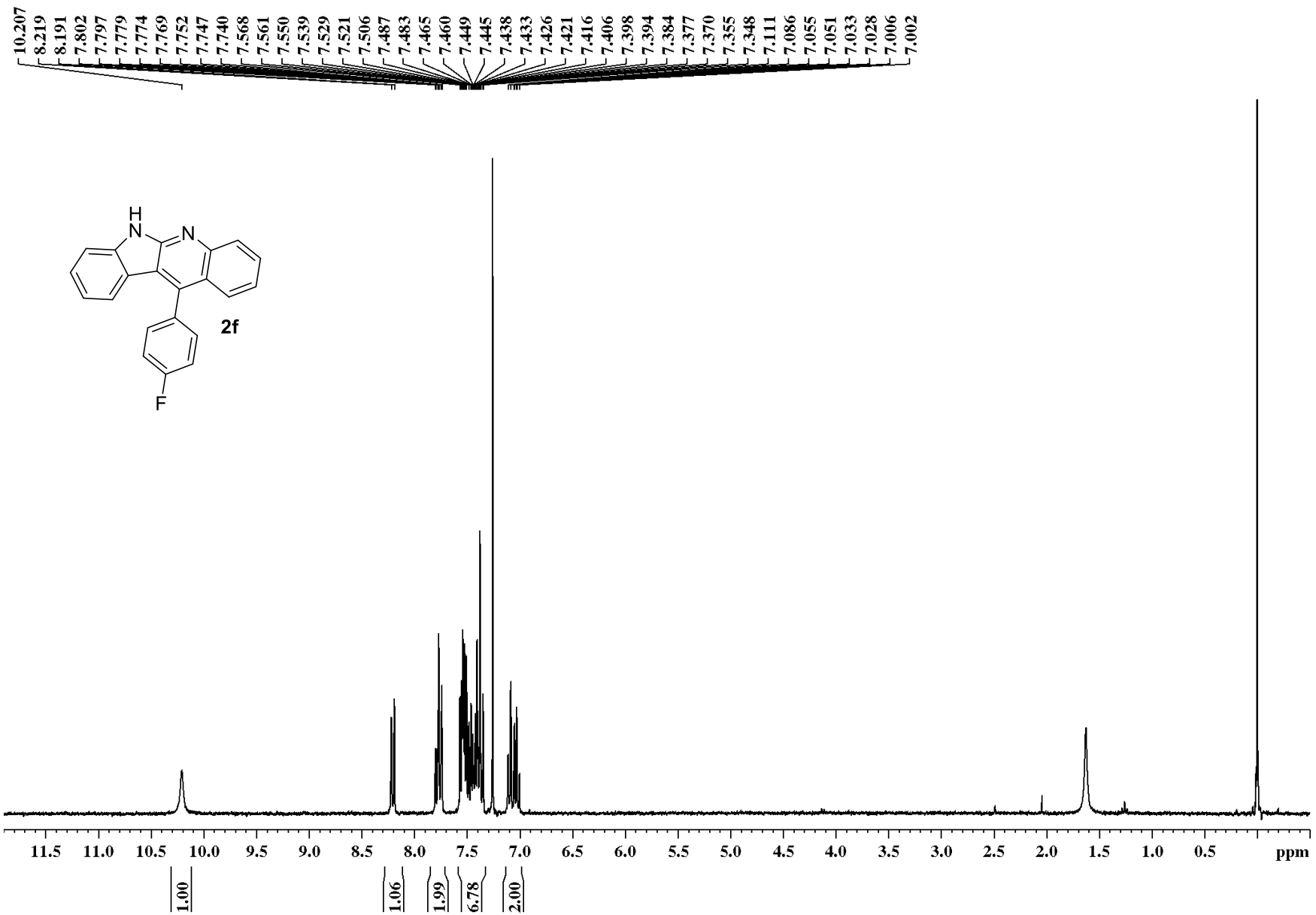
128.89
128.54
127.86
126.58
126.36
123.70
123.05
122.84
121.05
119.70
116.79
110.82
H
N
N
2e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.779
7.774
7.769
7.752
7.747
7.740
7.568
7.561
7.550
7.539
7.529
7.521
7.506
7.487
7.483
7.465
7.460
7.449
7.445
7.438
7.433
7.426
7.421
7.416
7.406
7.398
7.394
7.384
7.377
7.370
7.355
7.348
7.111
7.086
7.055
7.051
7.033
7.028
7.006
7.002
H
N
N
F
2f
11.5 11.0 10.5 10.0 9.5 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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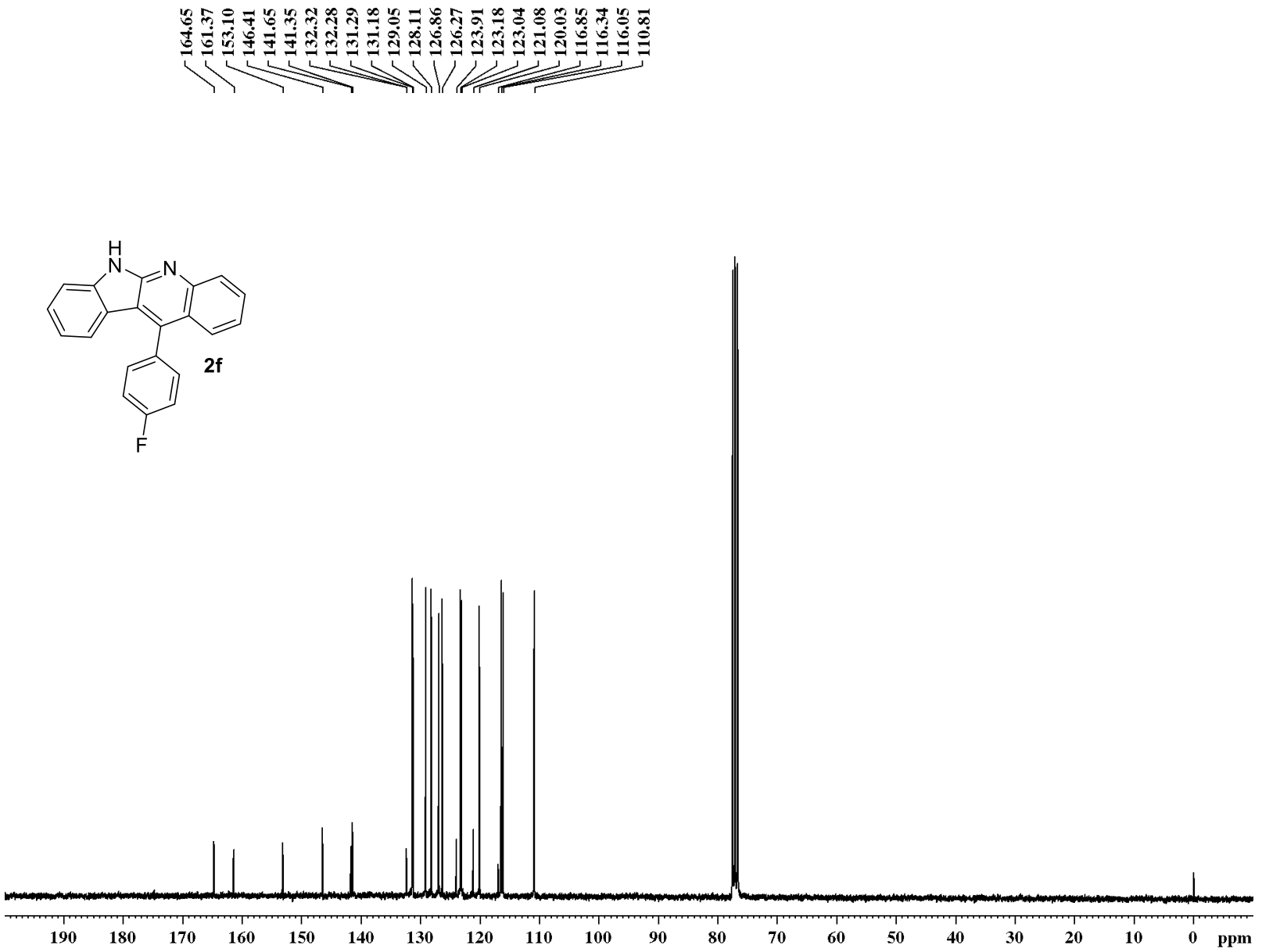
1.06
1.99
6.78
2.00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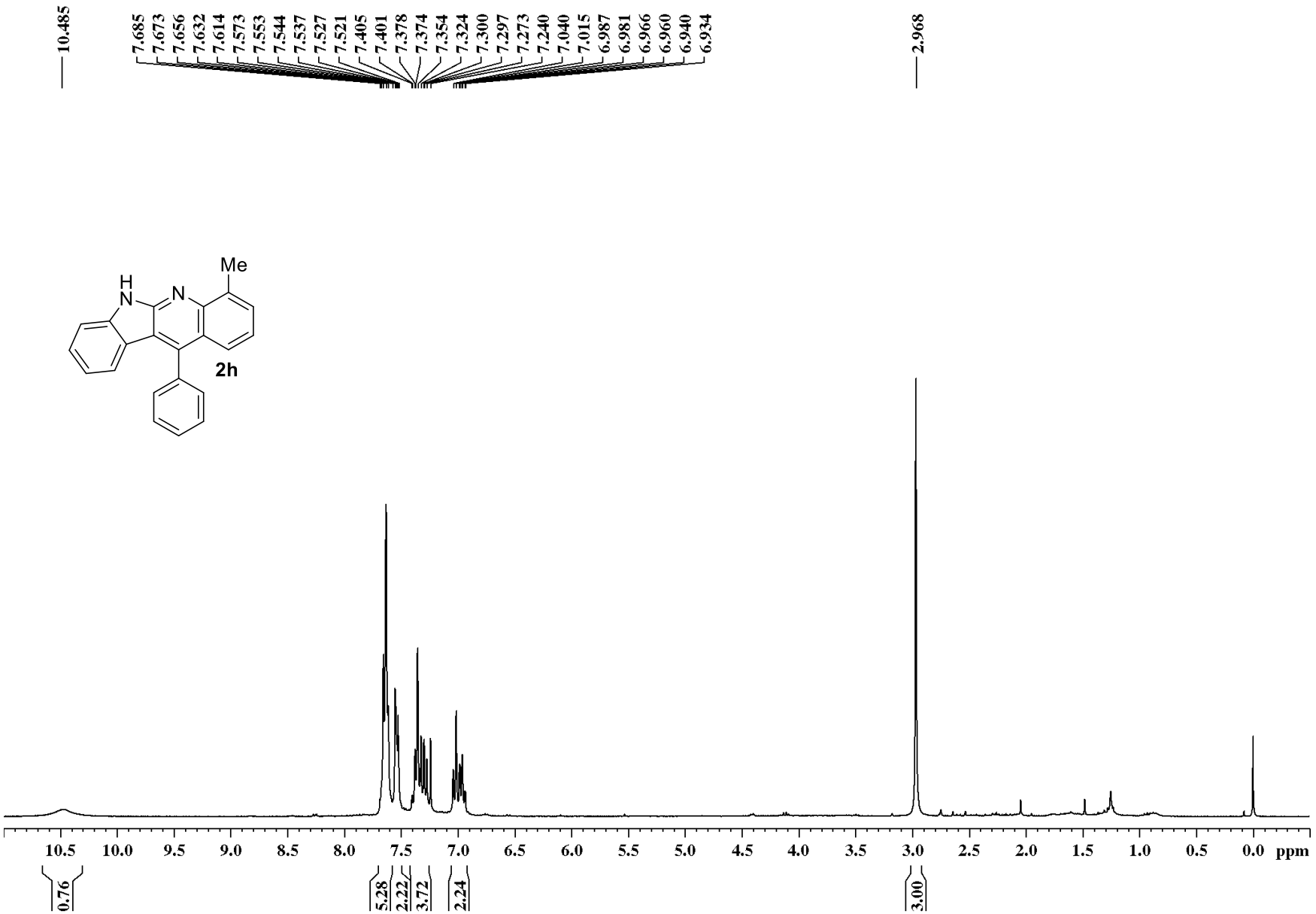
161.37
153.10
146.41
141.65
141.35
132.32
132.28
131.29
131.18
129.05
128.11
126.86
126.27
123.91
123.18
123.04
121.08
120.03
116.85
116.34
116.05
110.81
H
N
N
2f
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.614
7.573
7.553
7.544
7.537
7.527
7.521
7.405
7.401
7.378
7.374
7.354
7.324
7.300
7.297
7.273
7.240
7.040
7.015
6.987
6.981
6.966
6.960
6.940
6.934
2.968
Me
H
N
N
2h
10.5
10.0
9.5
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
0.0
ppm
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5.28
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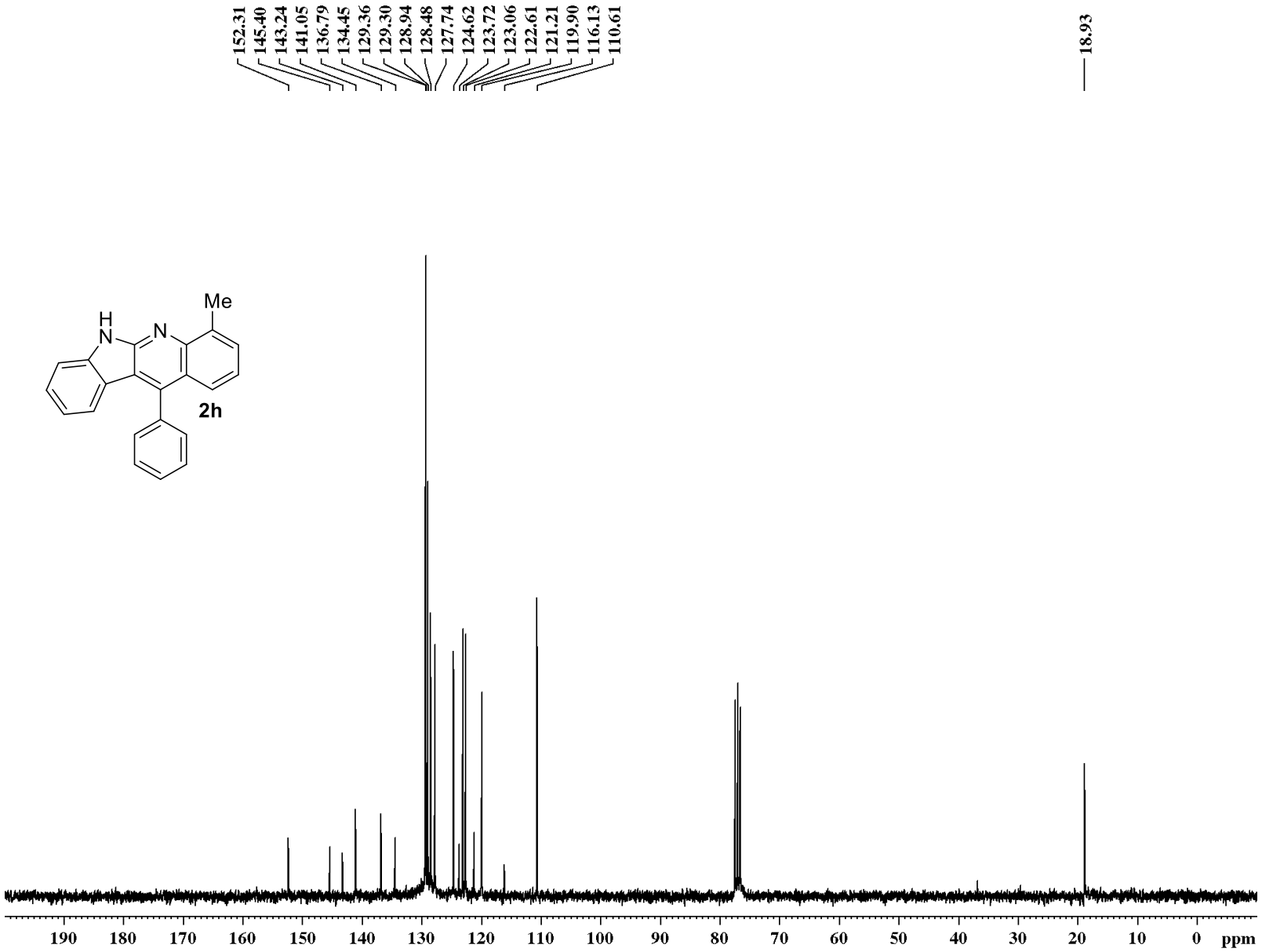
3.72
2.24
3.00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143.24
141.05
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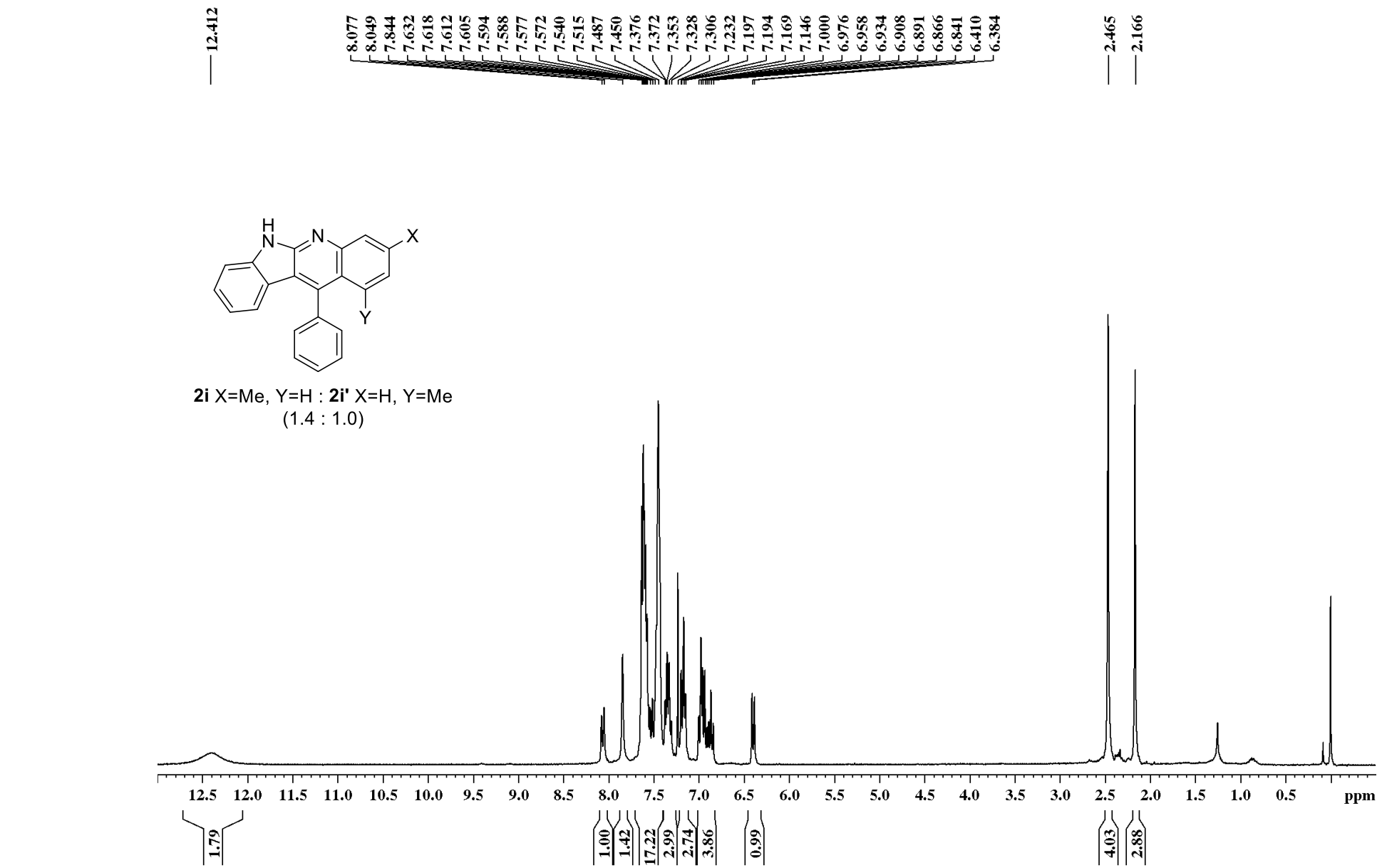
129.30
128.94
128.48
127.74
124.62
123.72
123.06
122.61
121.21
119.90
116.13
110.61
18.93
Me
H
N
N
2h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.618
7.612
7.605
7.594
7.588
7.577
7.572
7.540
7.515
7.487
7.450
7.376
7.372
7.353
7.328
7.306
7.232
7.197
7.194
7.169
7.146
7.000
6.976
6.958
6.934
6.908
6.891
6.866
6.841
6.410
6.384
2.465
2.166
H
N
N
X
Y
2i X=Me, Y=H : 2i' X=H, Y=Me
(1.4 : 1.0)
12.5 12.0 11.5 11.0 10.5 10.0 9.5 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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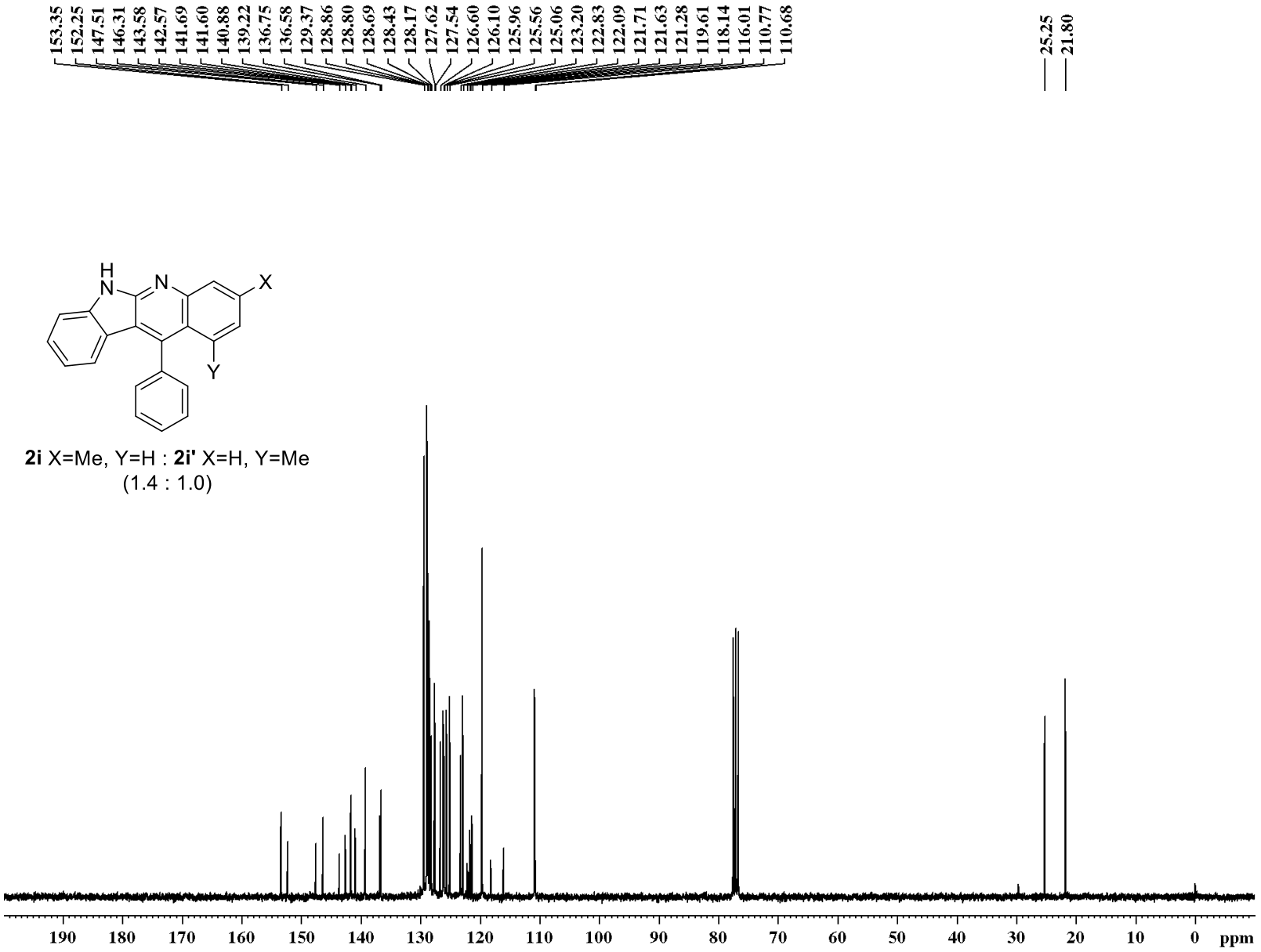
1.00
1.42
17.22
2.99
2.74
3.86
0.99
4.03
2.88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142.57
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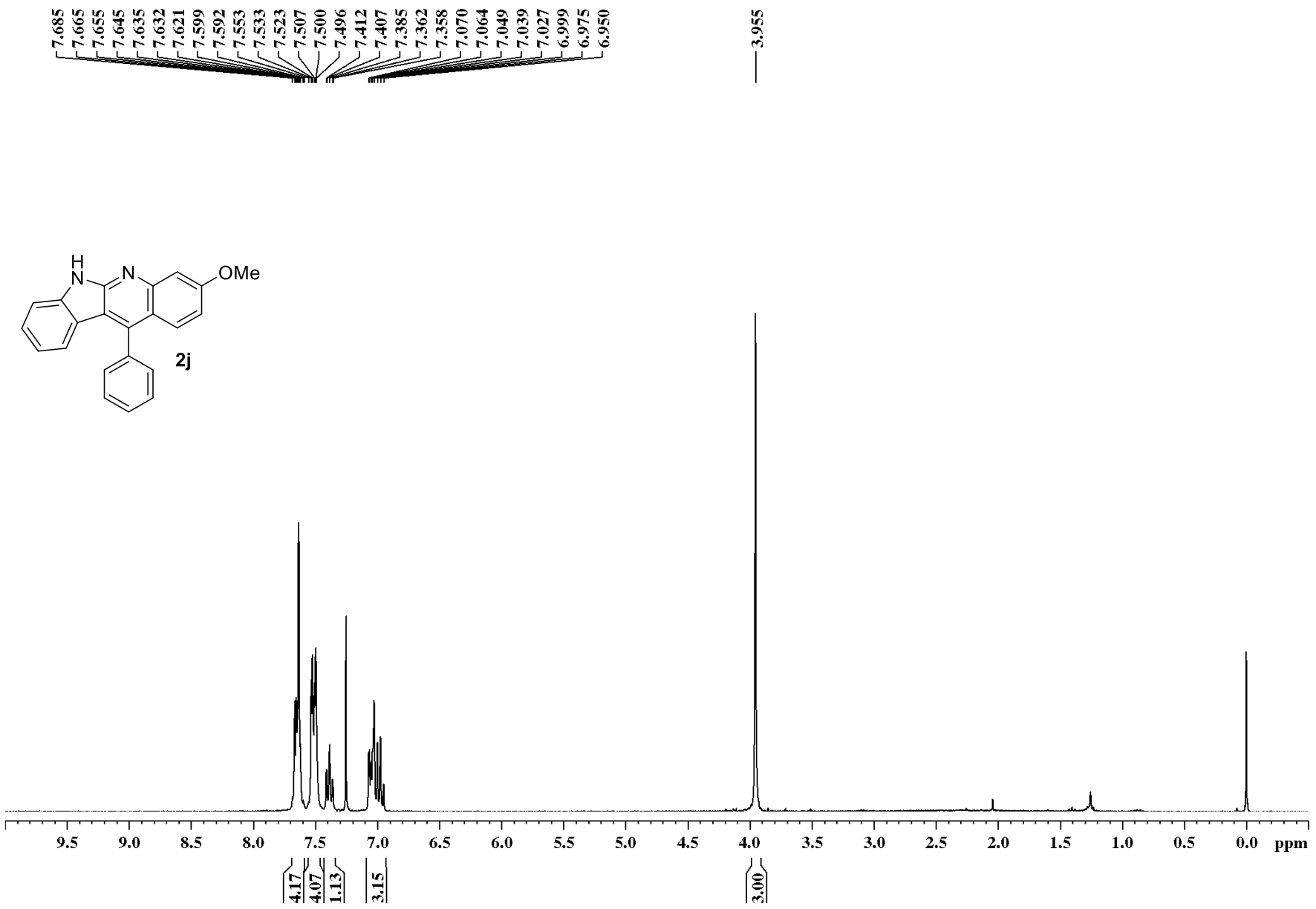
141.60
140.88
139.22
136.75
136.58
129.37
128.86
128.80
128.69
128.43
128.17
127.62
127.54
126.60
126.10
125.96
125.56
125.06
123.20
122.83
122.09
121.71
121.63
121.28
119.61
118.14
116.01
110.77
110.68
25.25
21.80
H
N
N
X
Y
2i X=Me, Y=H : 2i' X=H, Y=Me
(1.4 : 1.0)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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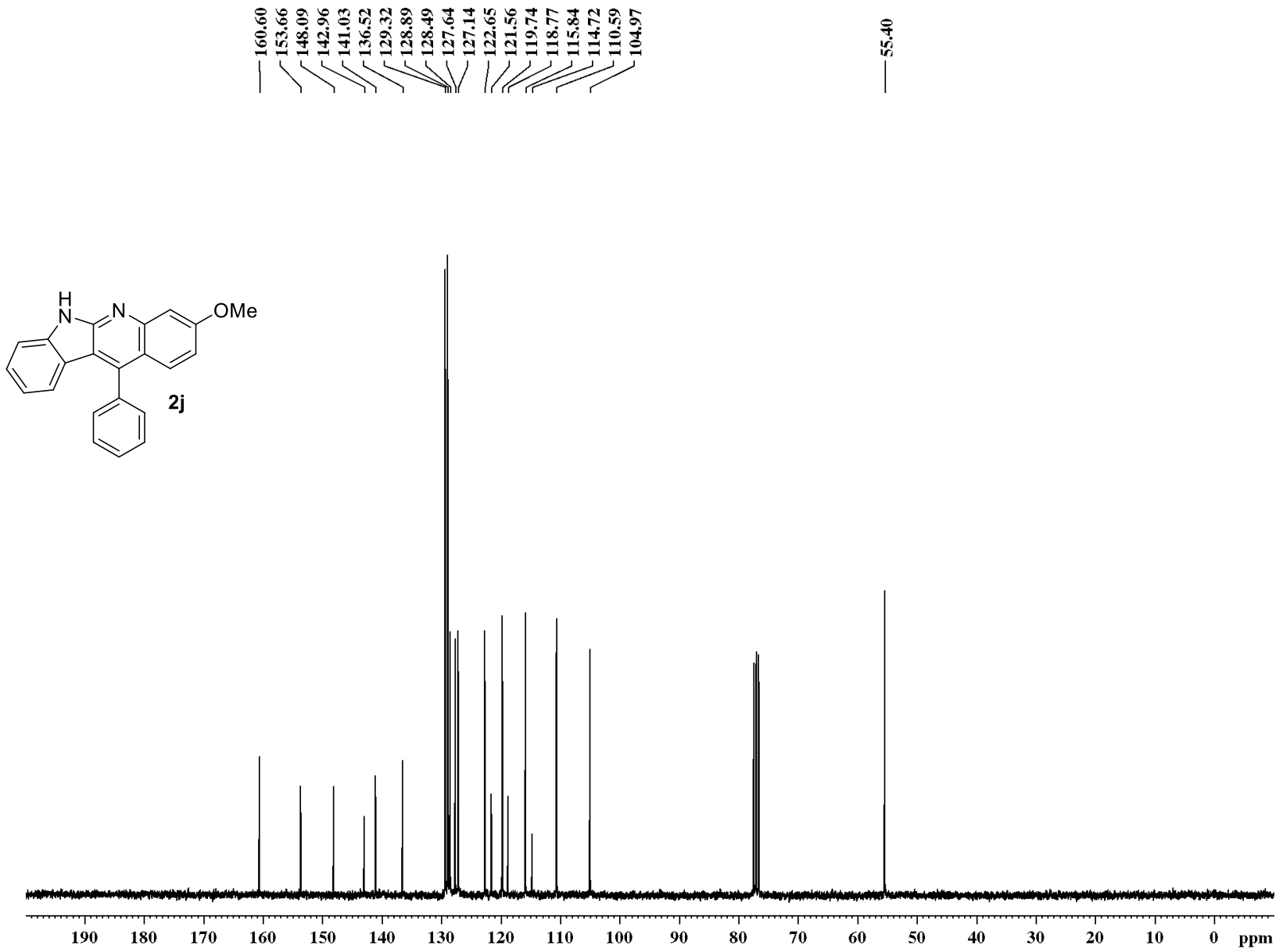
7.645
7.635
7.632
7.621
7.599
7.592
7.553
7.533
7.523
7.507
7.500
7.496
7.412
7.407
7.385
7.362
7.358
7.070
7.064
7.049
7.039
7.027
6.999
6.975
6.950
3.955
H
N
N
OMe
2j
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.17
4.07
1.13
3.15
3.00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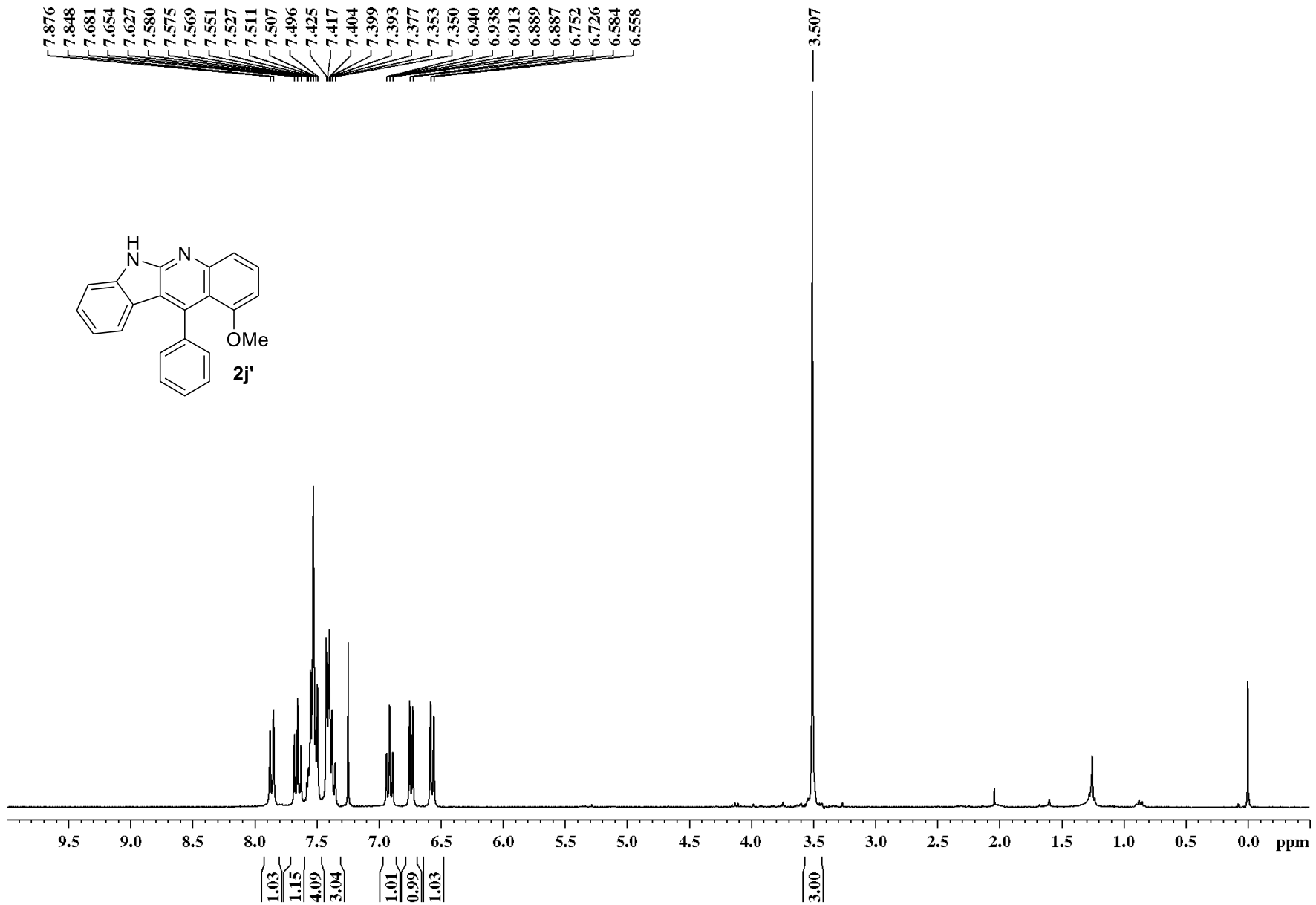
148.09
142.96
141.03
136.52
129.32
128.89
128.49
127.64
127.14
122.65
121.56
119.74
118.77
115.84
114.72
110.59
104.97
55.40
H
N
N
OMe
2j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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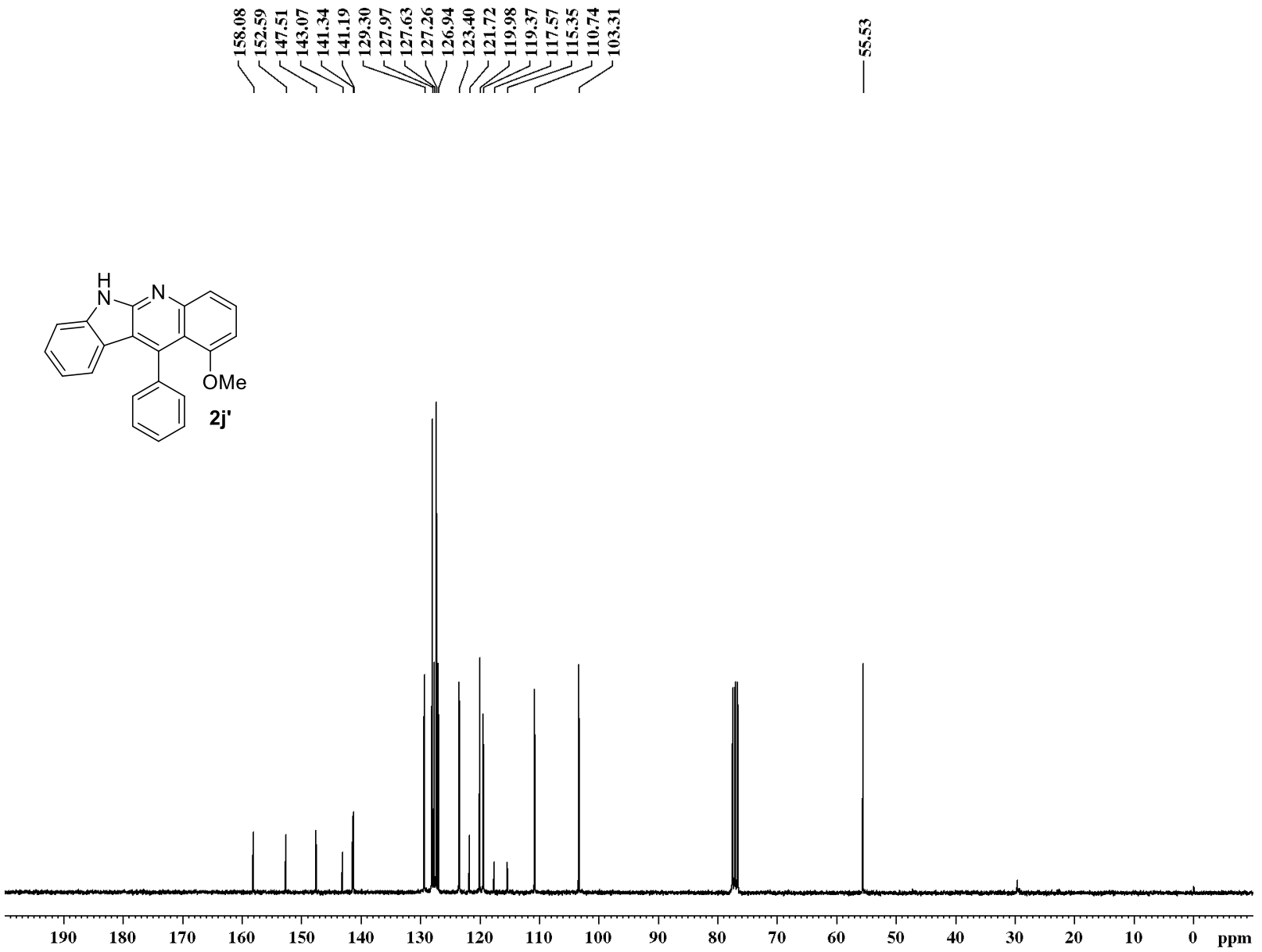
7.580
7.575
7.569
7.551
7.527
7.511
7.507
7.496
7.425
7.417
7.404
7.399
7.393
7.377
7.353
7.350
6.940
6.938
6.913
6.889
6.887
6.752
6.726
6.584
6.558
3.507
H
N
N
OMe
2j'
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.03
1.15
4.09
3.04
1.01
0.99
1.03
3.00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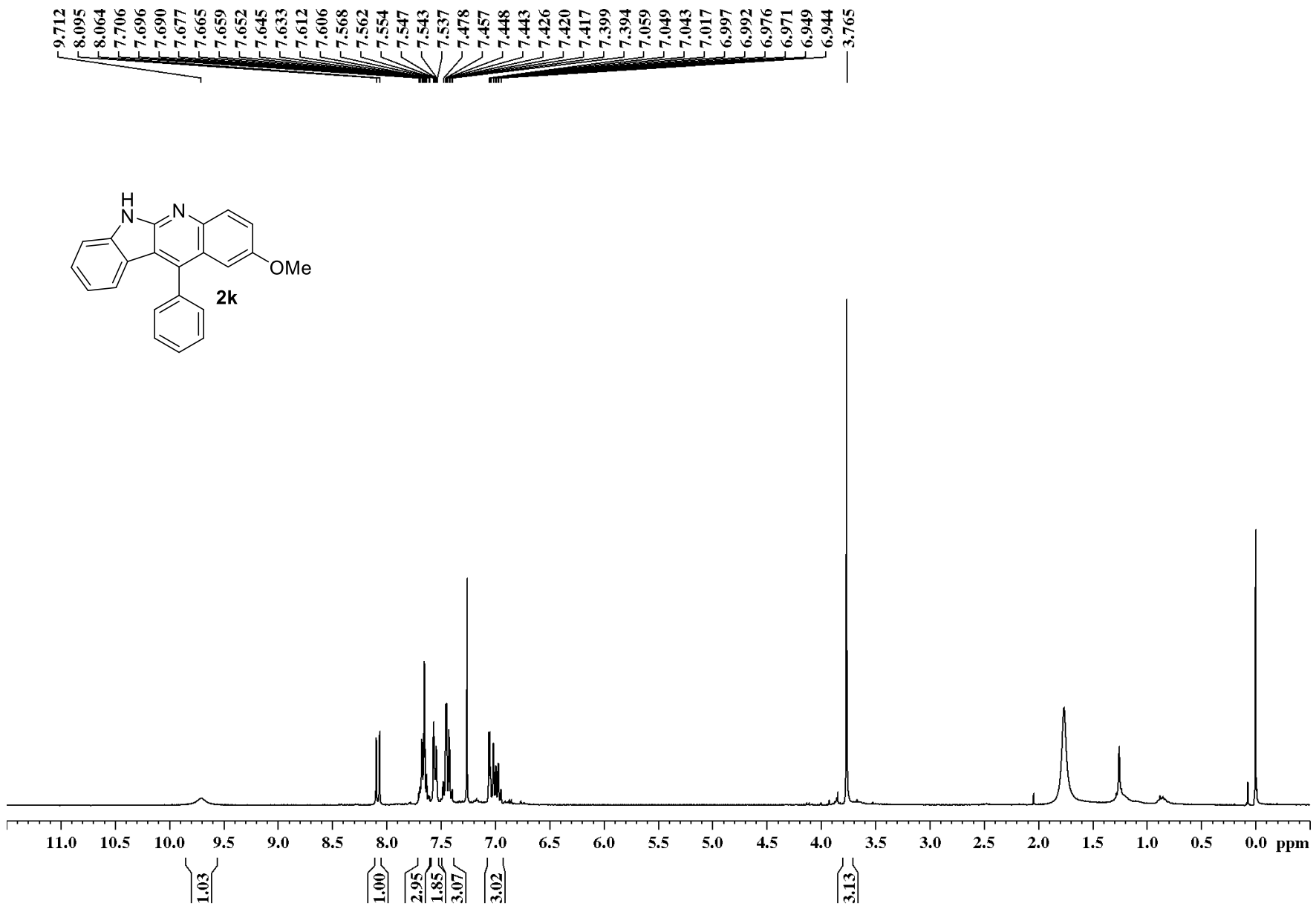
127.97
127.63
127.26
126.94
123.40
121.72
119.98
119.37
117.57
115.35
110.74
103.31
55.53
H
N
N
OMe
2j'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.665
7.659
7.652
7.645
7.633
7.612
7.606
7.568
7.562
7.554
7.547
7.543
7.537
7.478
7.457
7.448
7.443
7.426
7.420
7.417
7.399
7.394
7.059
7.049
7.043
7.017
6.997
6.992
6.976
6.971
6.949
6.944
3.765
H
N
N
OMe
2k
11.0 10.5 10.0 9.5 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 0.0 ppm
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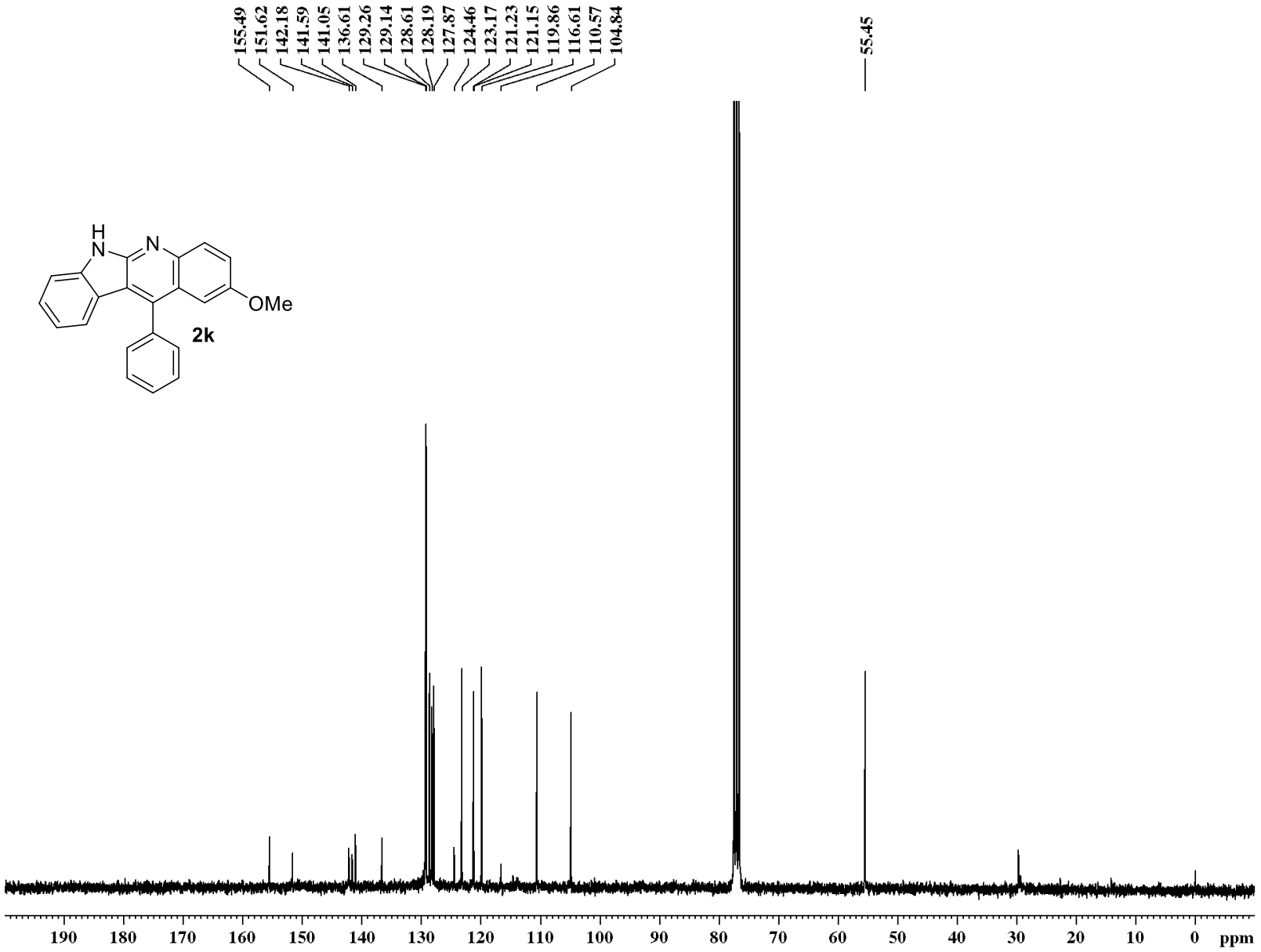
1.00
2.95
1.85
3.07
3.02
3.13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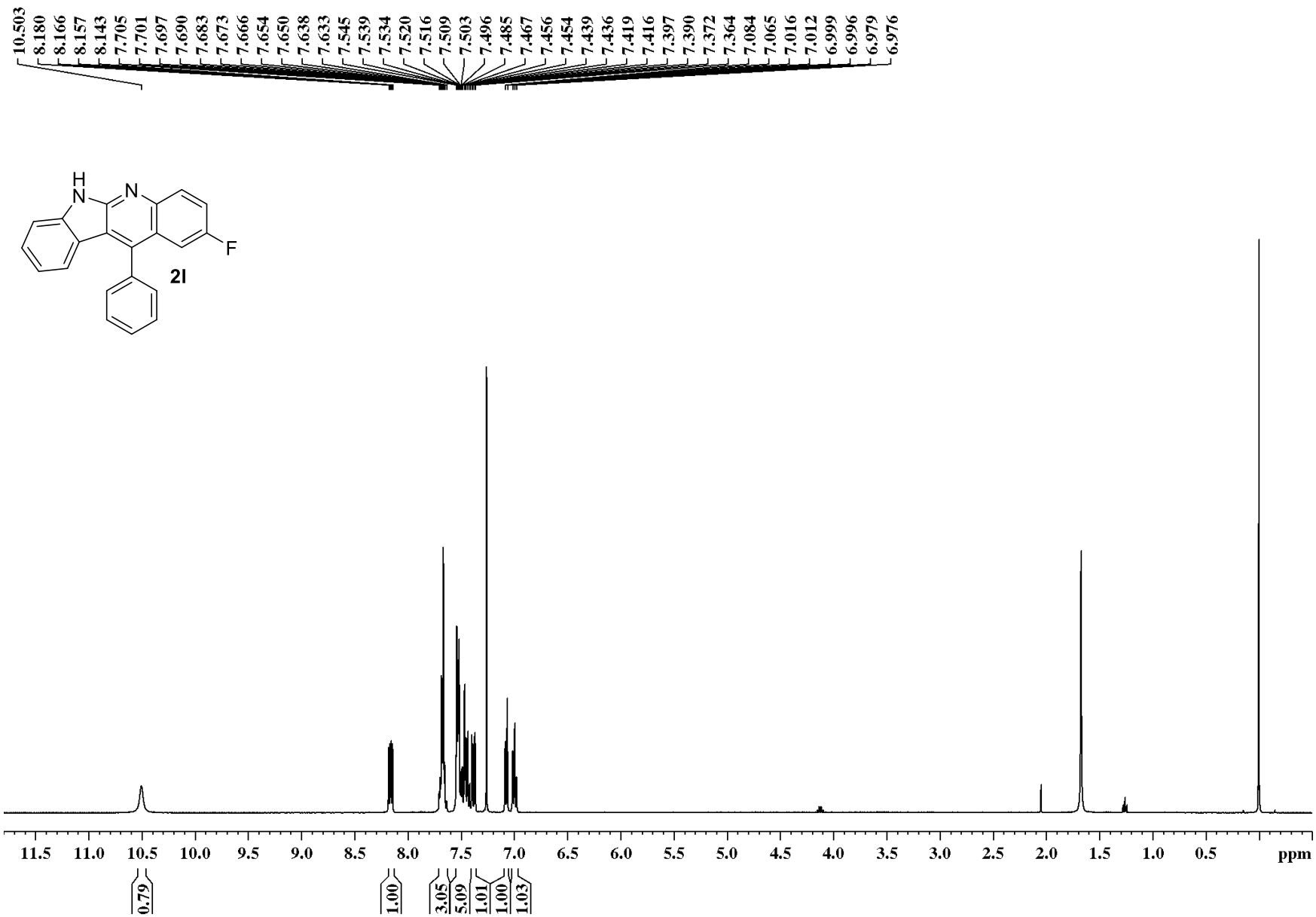
141.05
136.61
129.26
129.14
128.61
128.19
127.87
124.46
123.17
121.23
121.15
119.86
116.61
110.57
104.84
55.45
H
N
N
OMe
2k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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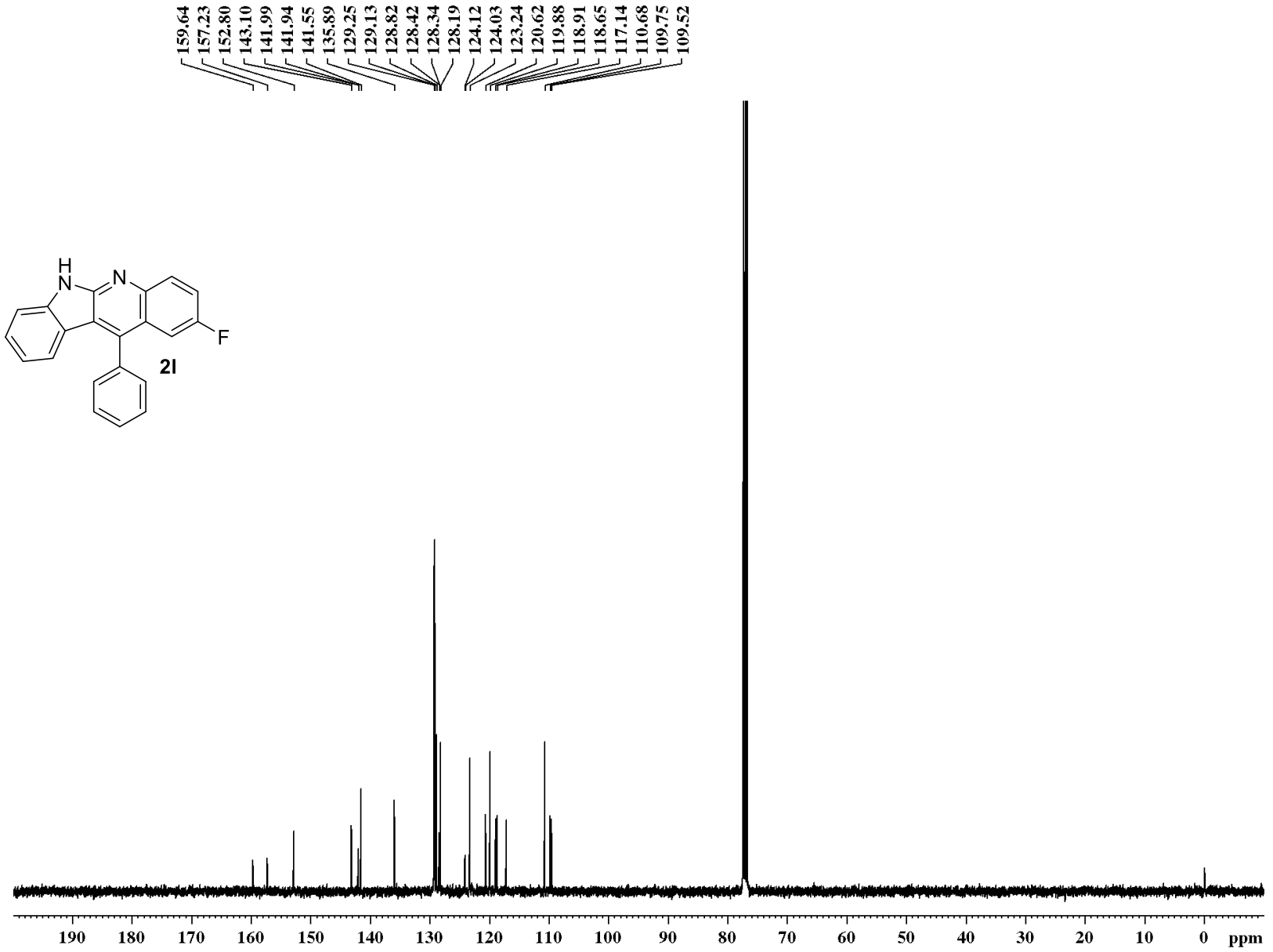
8.166
8.157
8.143
7.705
7.701
7.697
7.690
7.683
7.673
7.666
7.654
7.650
7.638
7.633
7.545
7.539
7.534
7.520
7.516
7.509
7.503
7.496
7.485
7.467
7.456
7.454
7.439
7.436
7.419
7.416
7.397
7.390
7.372
7.364
7.084
7.065
7.016
7.012
6.999
6.996
6.979
6.976
H
N
N
F
2l
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 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
0.79
1.00
3.05
5.09
1.01
1.00
1.03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109.75
109.52
H
N
N
F
2l
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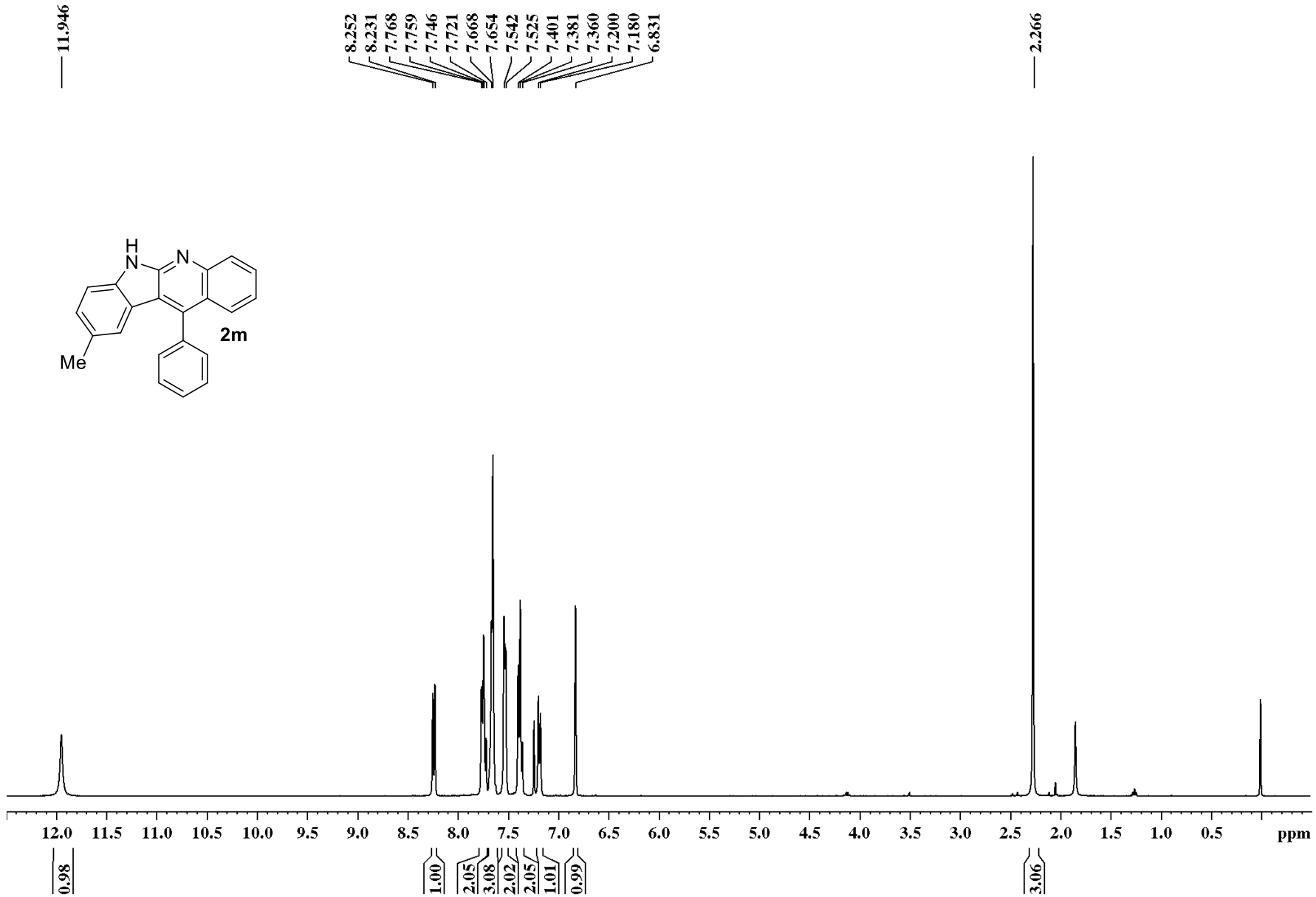
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7.360
7.200
7.180
6.831
2.266
H
N
N
Me
2m
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11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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2.5
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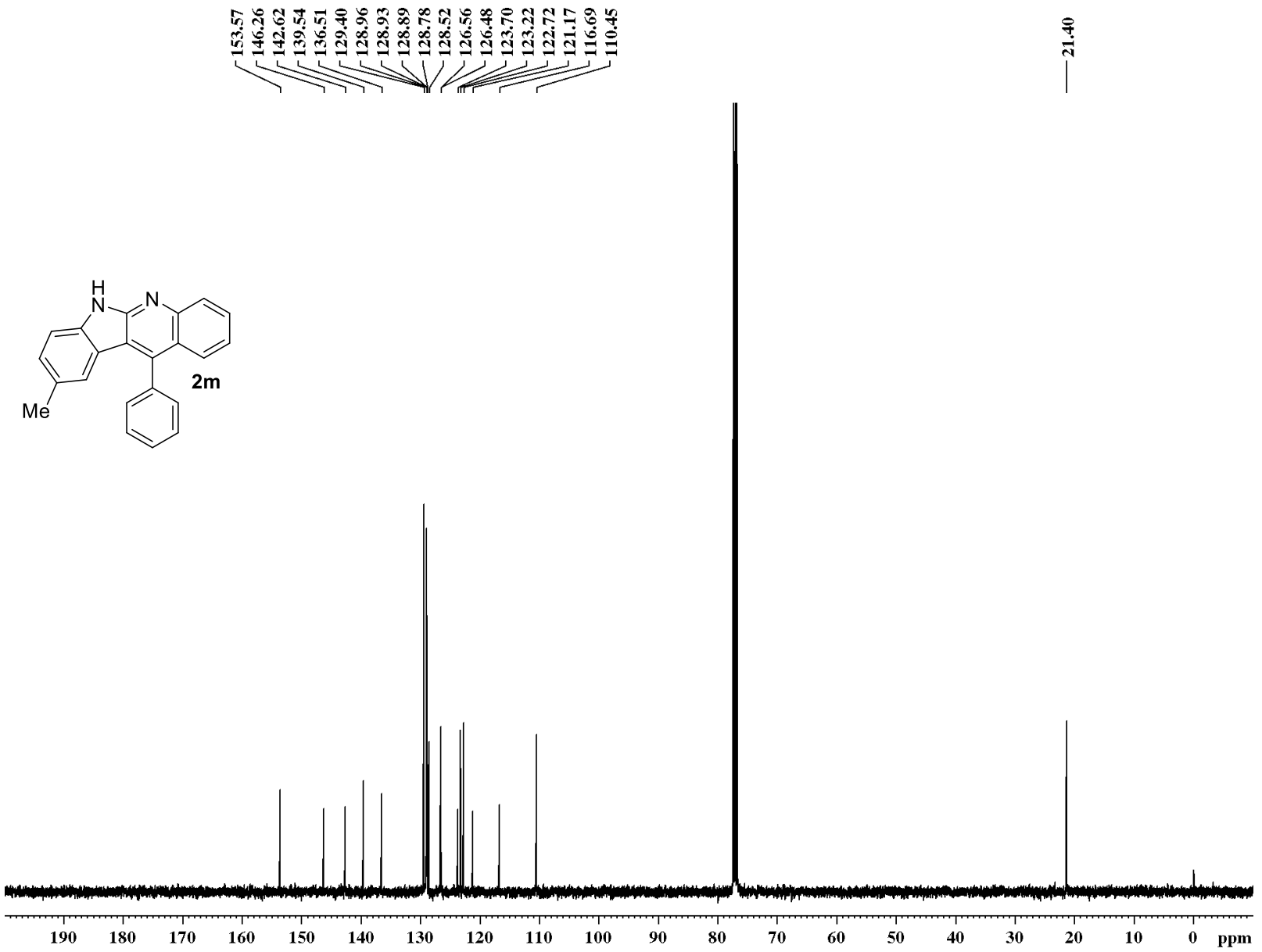
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Me
2m
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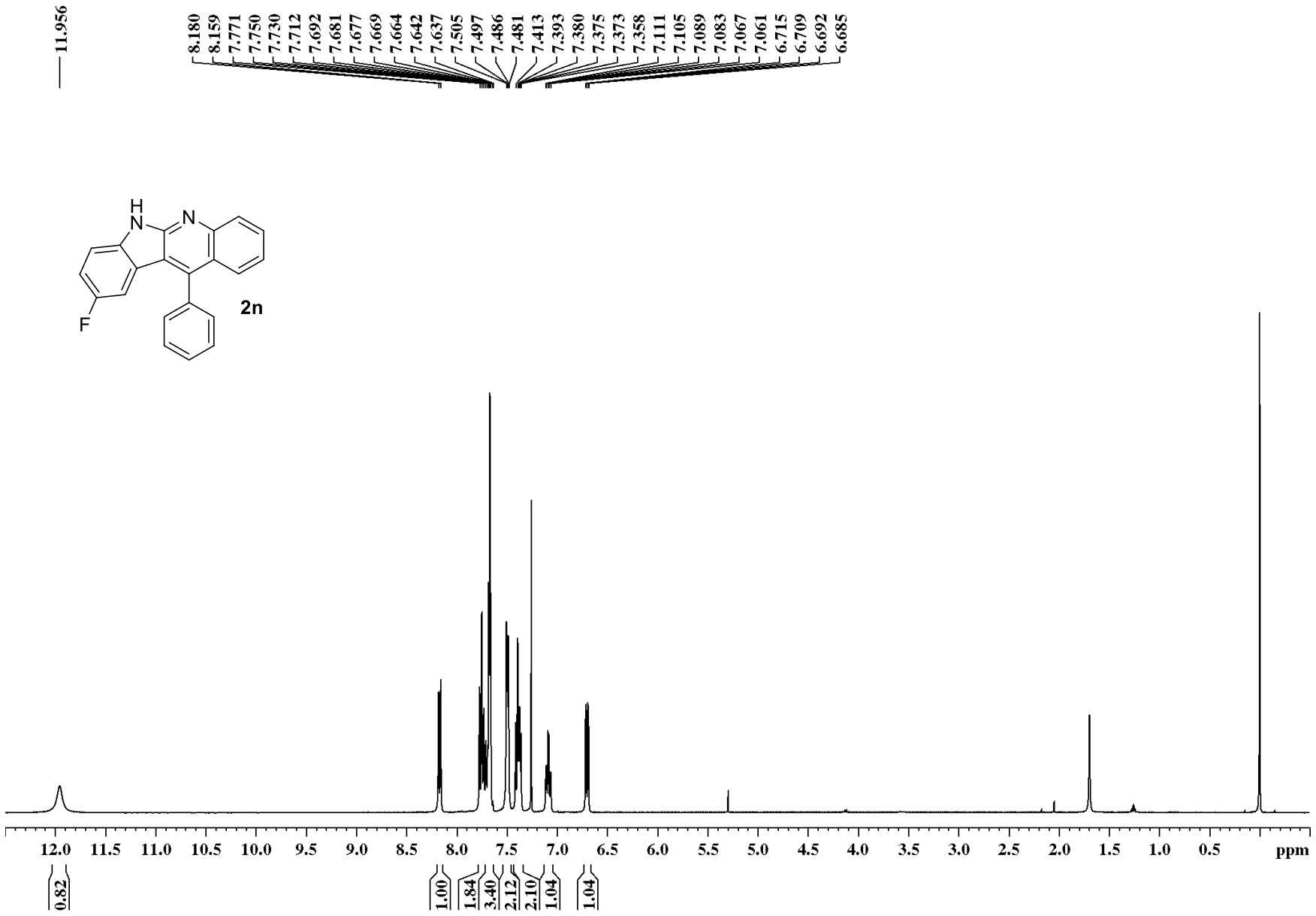
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7.642
7.637
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7.497
7.486
7.481
7.413
7.393
7.380
7.375
7.373
7.358
7.111
7.105
7.089
7.083
7.067
7.061
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6.692
6.685
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N
N
F
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
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5.0
4.5
4.0
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3.0
2.5
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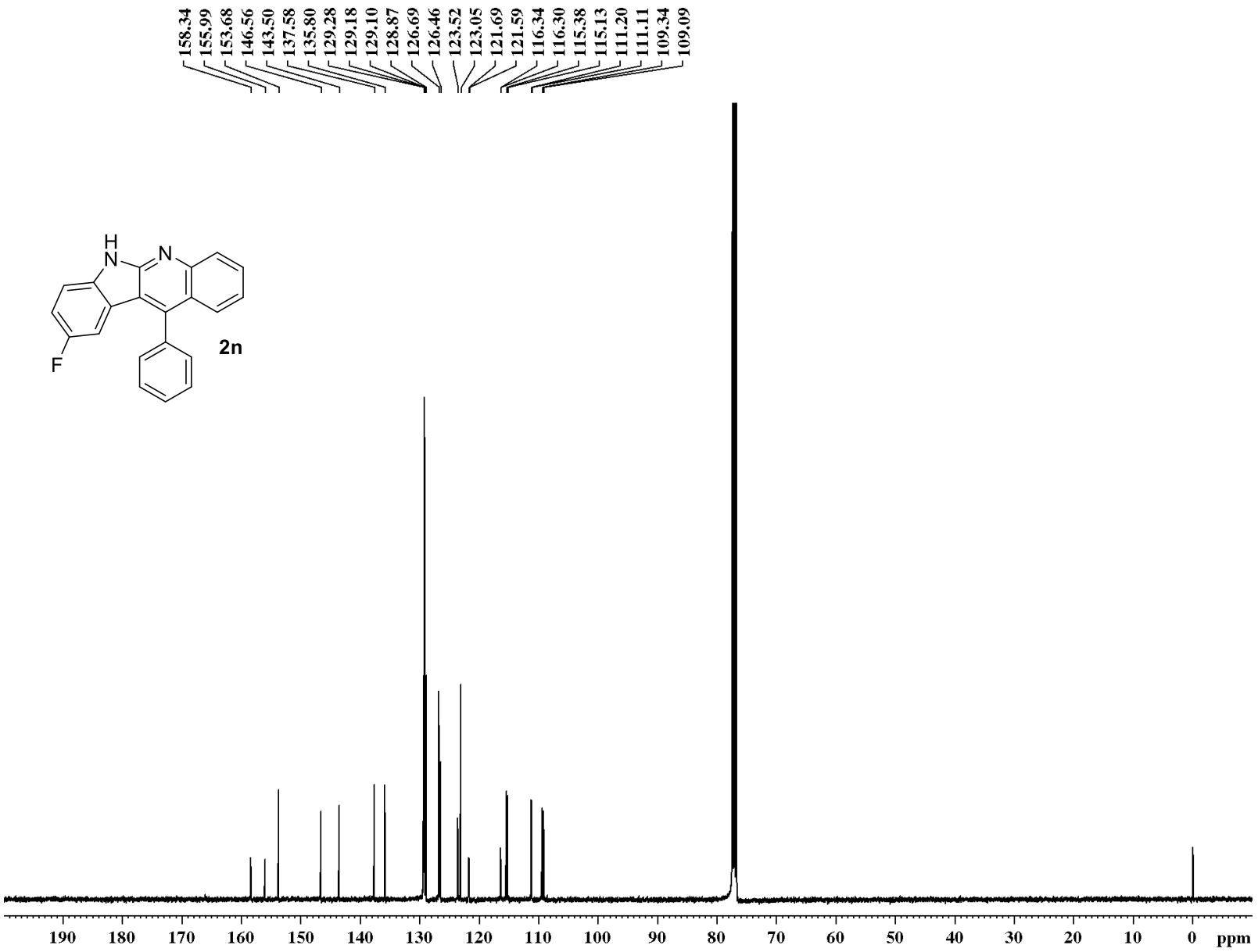
1.04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111.11
109.34
109.09
H
N
N
F
2n
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140
130
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100
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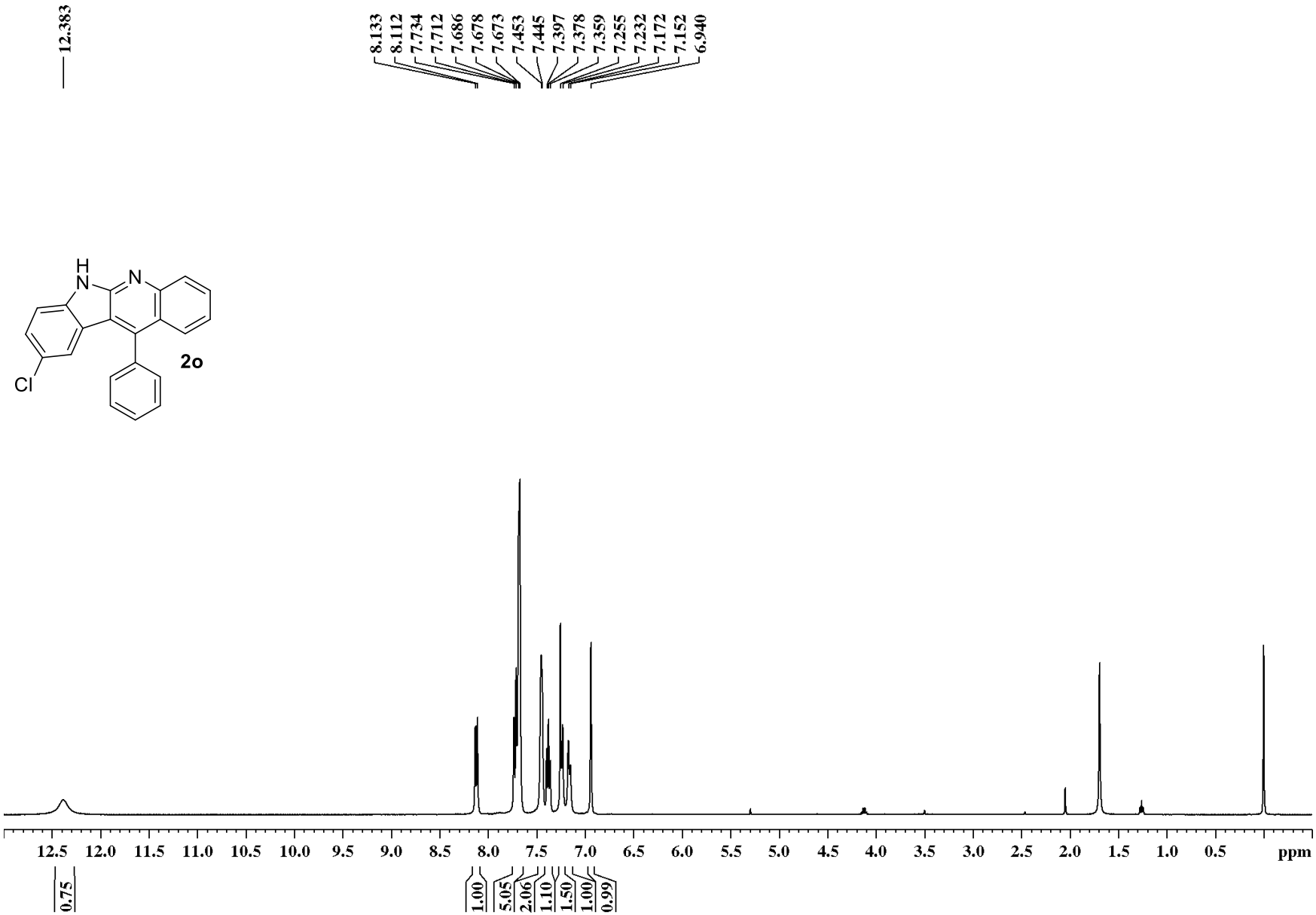
ppm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7.378
7.359
7.255
7.232
7.172
7.152
6.940
H
N
N
Cl
2o
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12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
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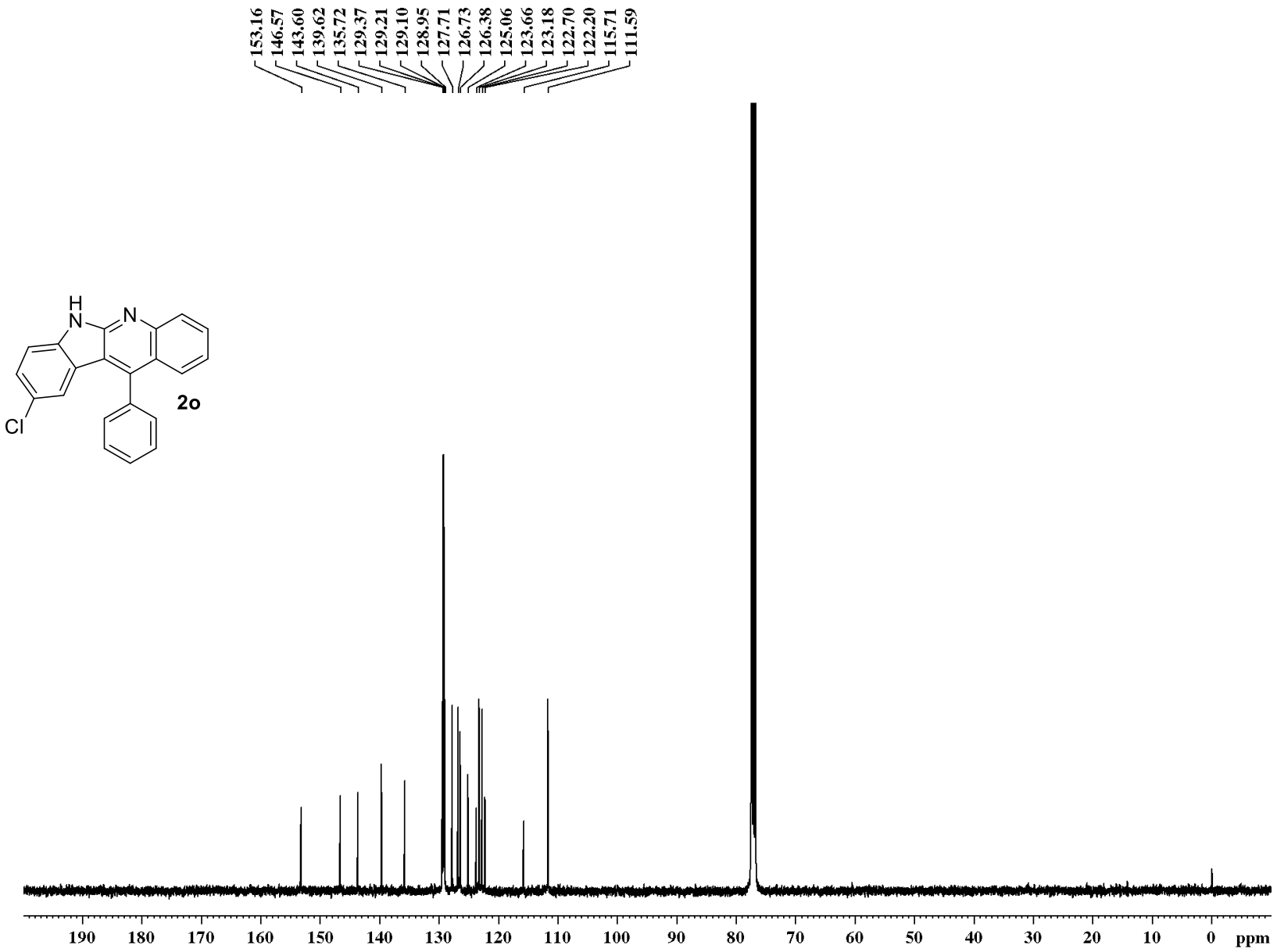
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123.18
122.70
122.20
115.71
111.59
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N
N
Cl
2o
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180
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140
130
120
110
100
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60
50
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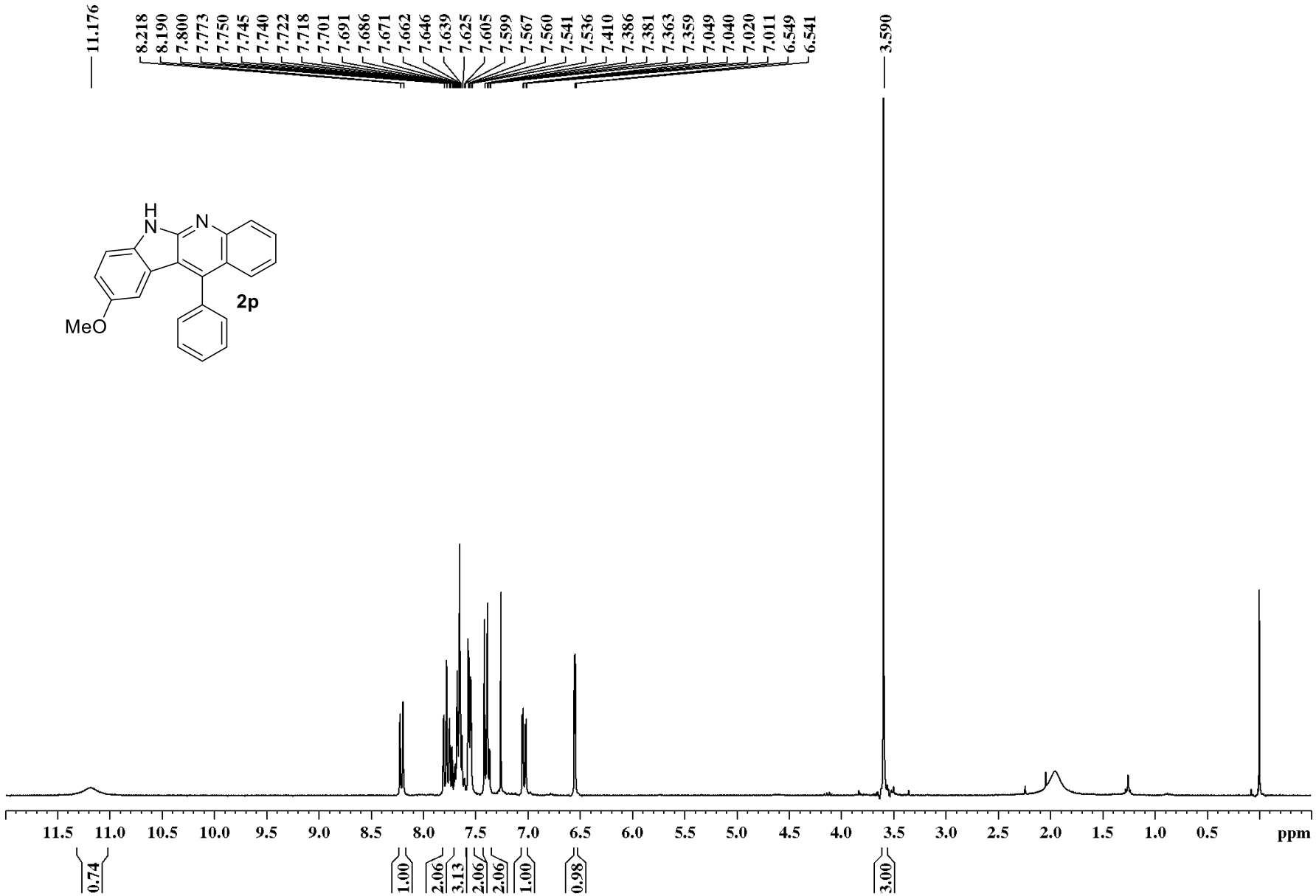
30
20
10
0
ppm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7.671
7.662
7.646
7.639
7.625
7.605
7.599
7.567
7.560
7.541
7.536
7.410
7.386
7.381
7.363
7.359
7.049
7.040
7.020
7.011
6.549
6.541
3.590
H
N
N
MeO
2p
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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2.06
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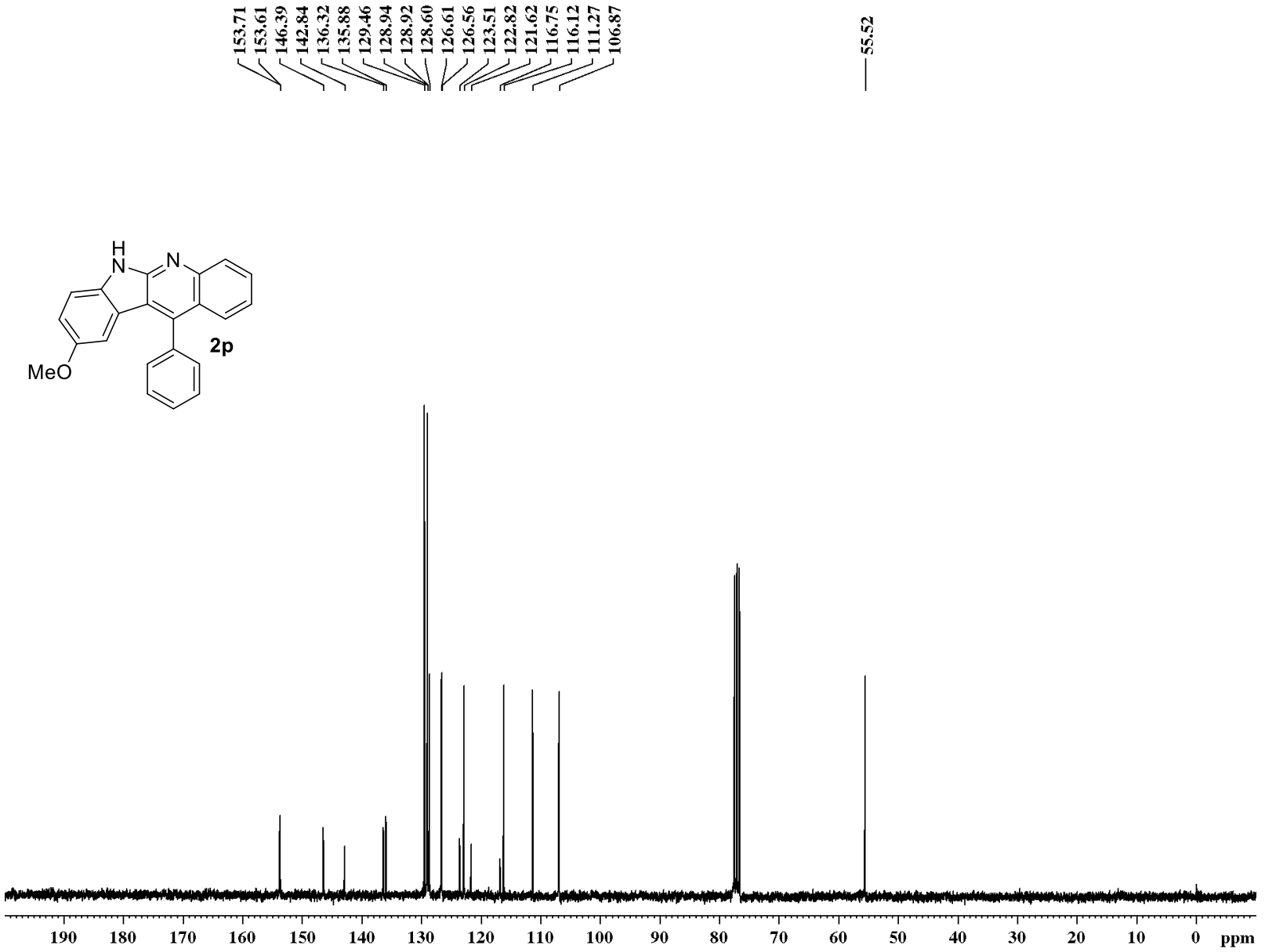
3.00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123.51
122.82
121.62
116.75
116.12
111.27
106.87
55.52
H
N
N
MeO
2p
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180
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140
130
120
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100
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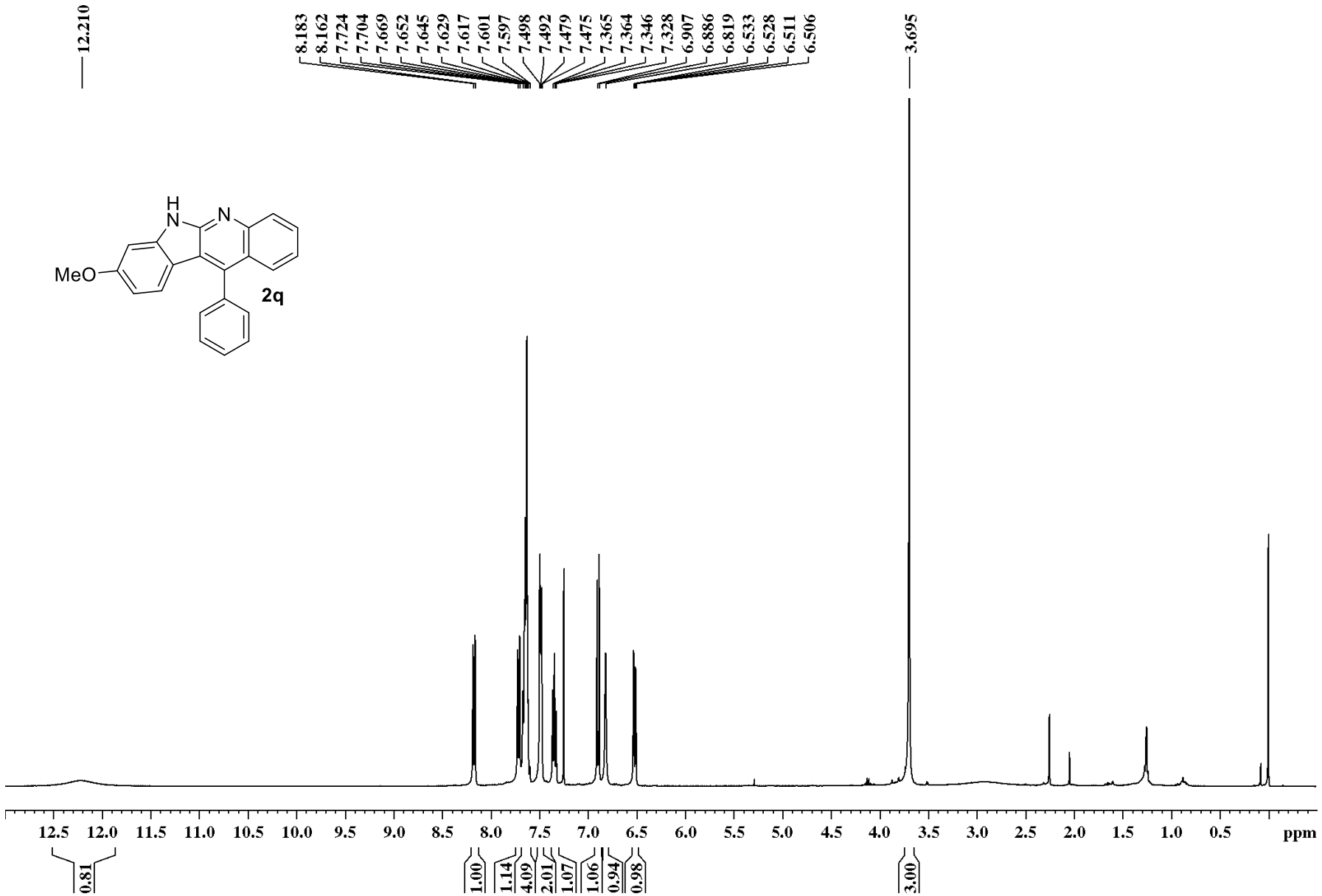
20
10
0
ppm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7.492
7.479
7.475
7.365
7.364
7.346
7.328
6.907
6.886
6.819
6.533
6.528
6.511
6.506
3.695
MeO
2q
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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1.07
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0.94
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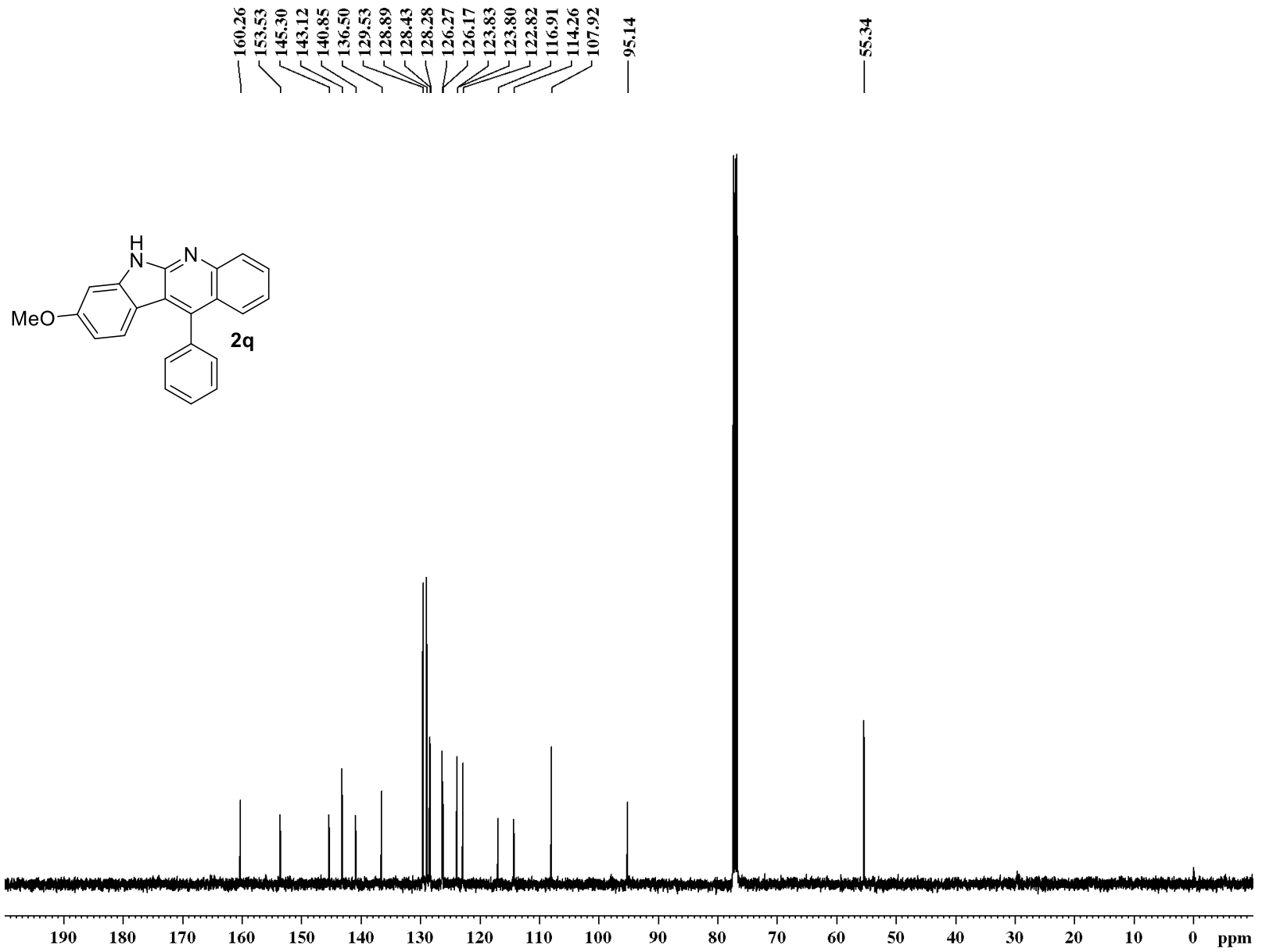
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123.83
123.80
122.82
116.91
114.26
107.92
95.14
55.34
H
N
N
MeO
2q
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180
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140
130
120
110
100
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60
50
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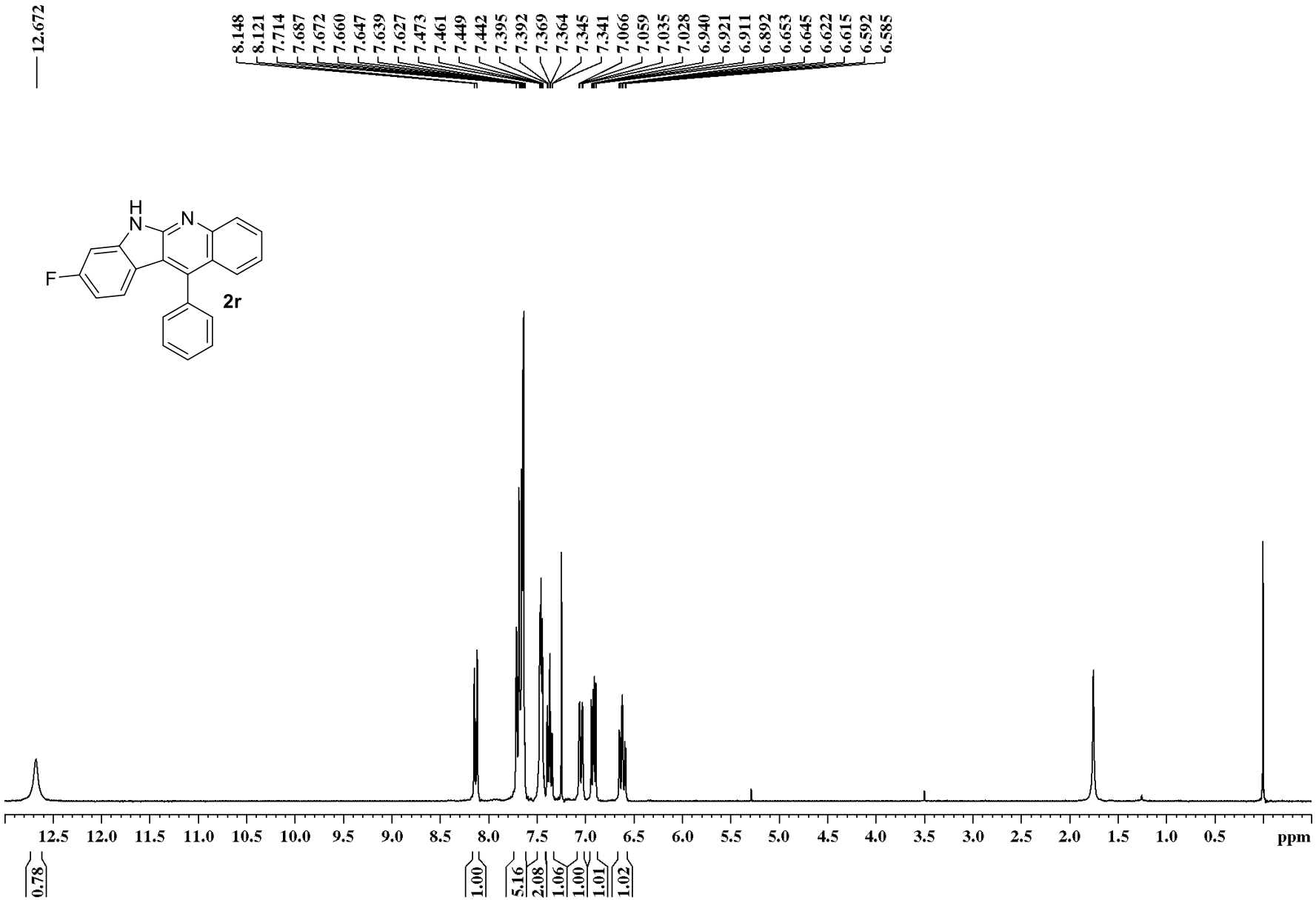
30
20
10
0
ppm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7.449
7.442
7.395
7.392
7.369
7.364
7.345
7.341
7.066
7.059
7.035
7.028
6.940
6.921
6.911
6.892
6.653
6.645
6.622
6.615
6.592
6.585
H
N
N
F
2r
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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2.08
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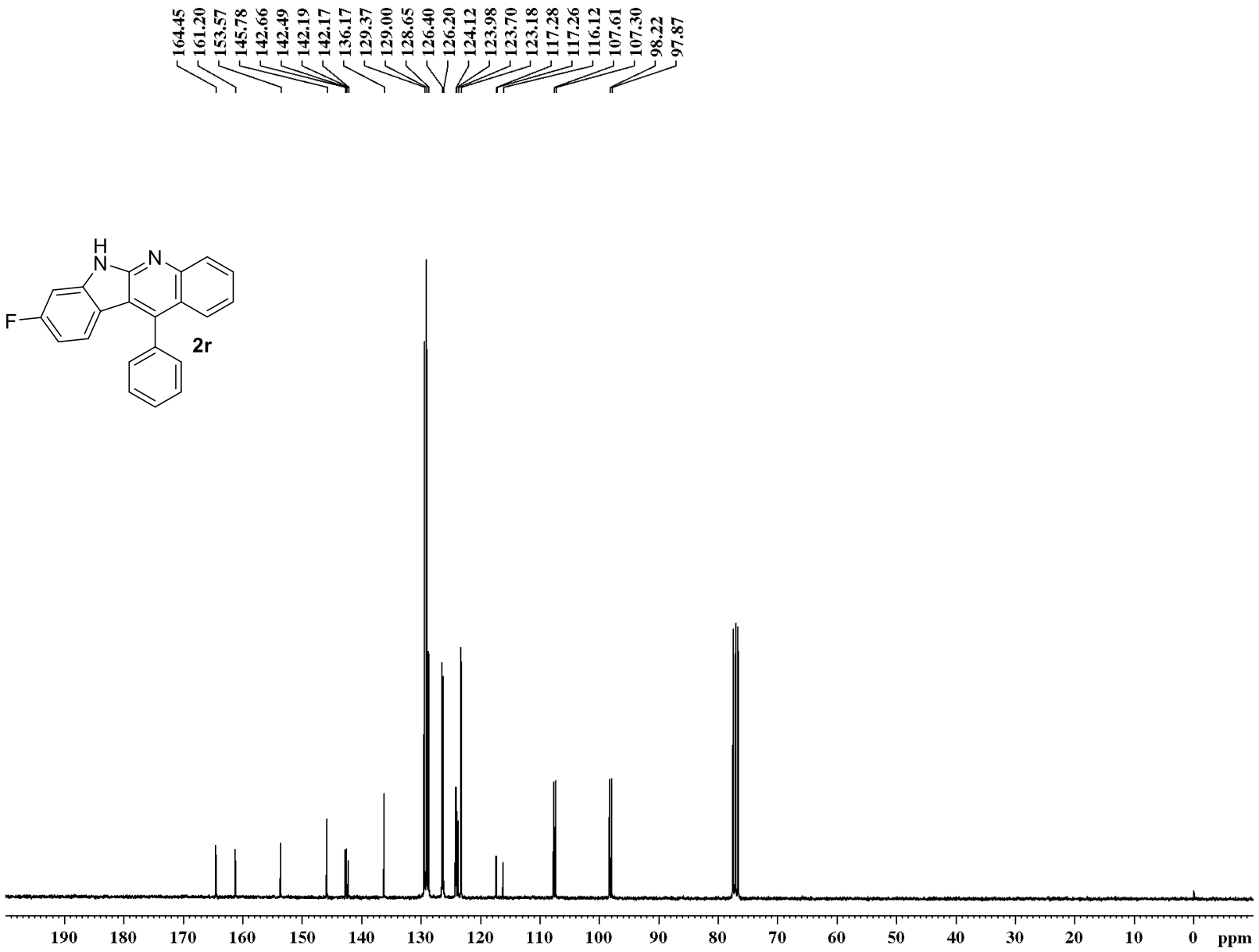
1.00
1.01
1.02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123.18
117.28
117.26
116.12
107.61
107.30
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97.87
H
N
N
F
2r
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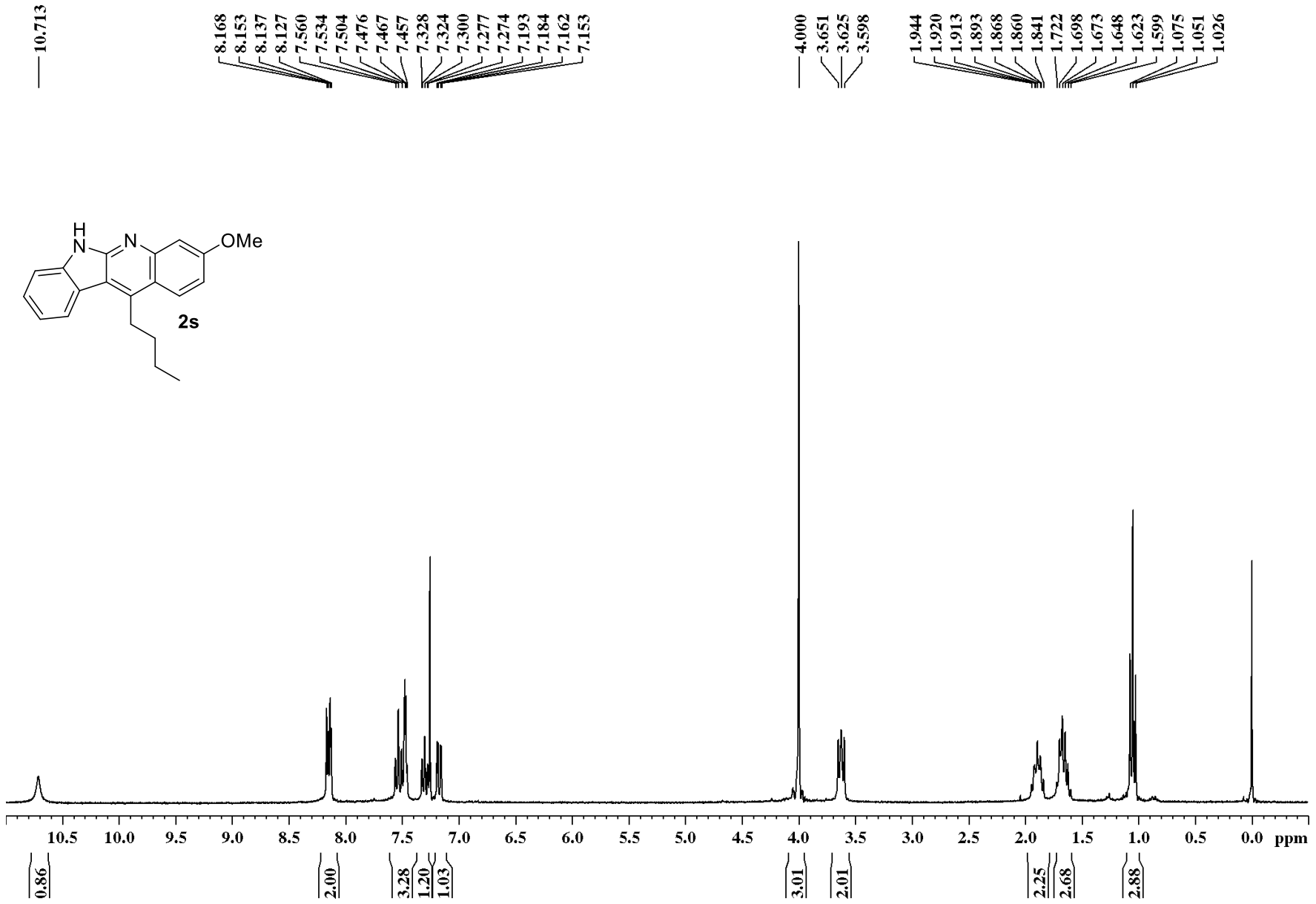
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7.274
7.193
7.184
7.162
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3.625
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1.920
1.913
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1.868
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1.841
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1.698
1.673
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1.623
1.599
1.075
1.051
1.026
H
N
N
OMe
2s
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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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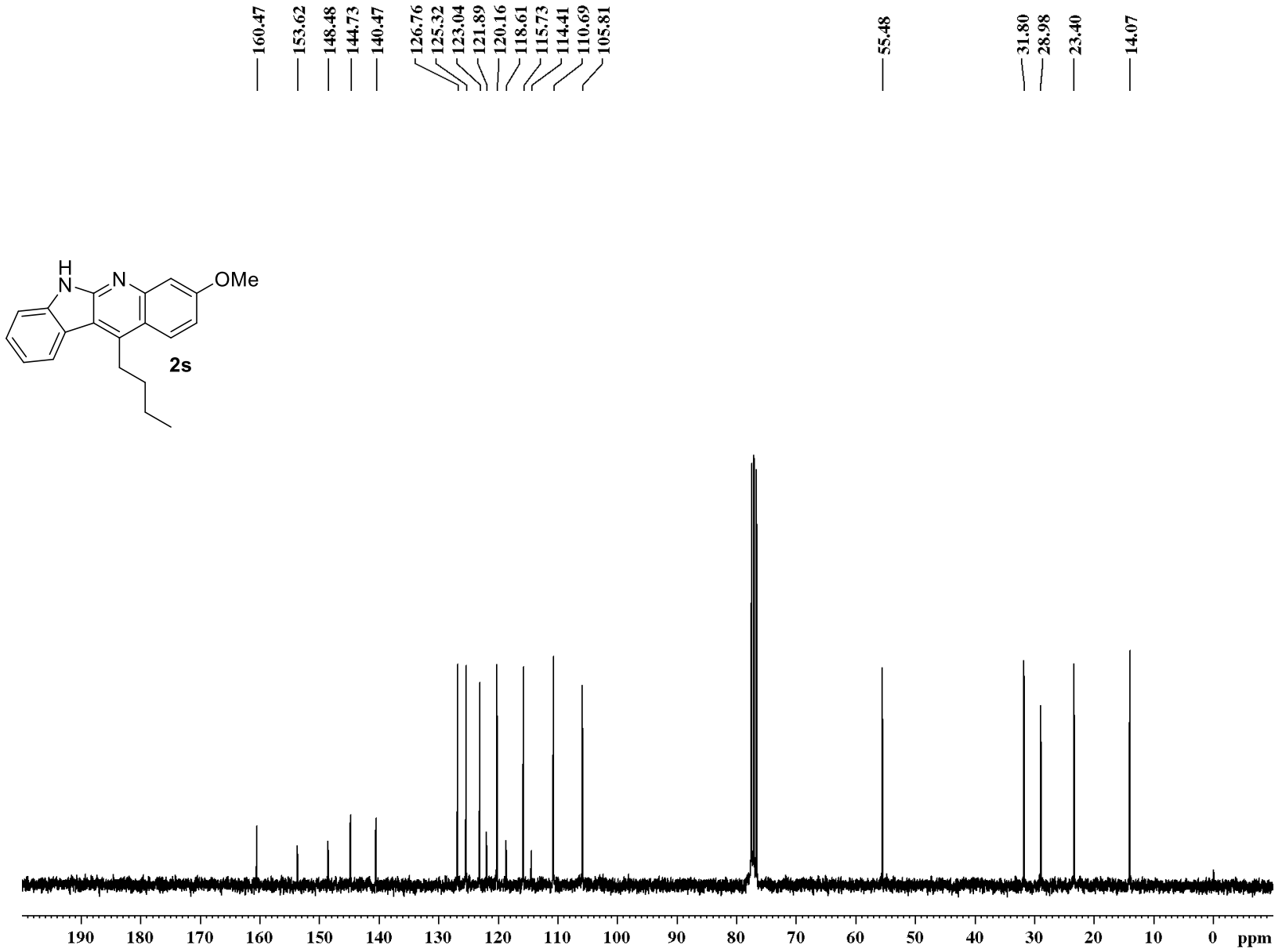
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55.48
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28.98
23.40
14.07
H
N
N
OMe
2s
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130
120
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20
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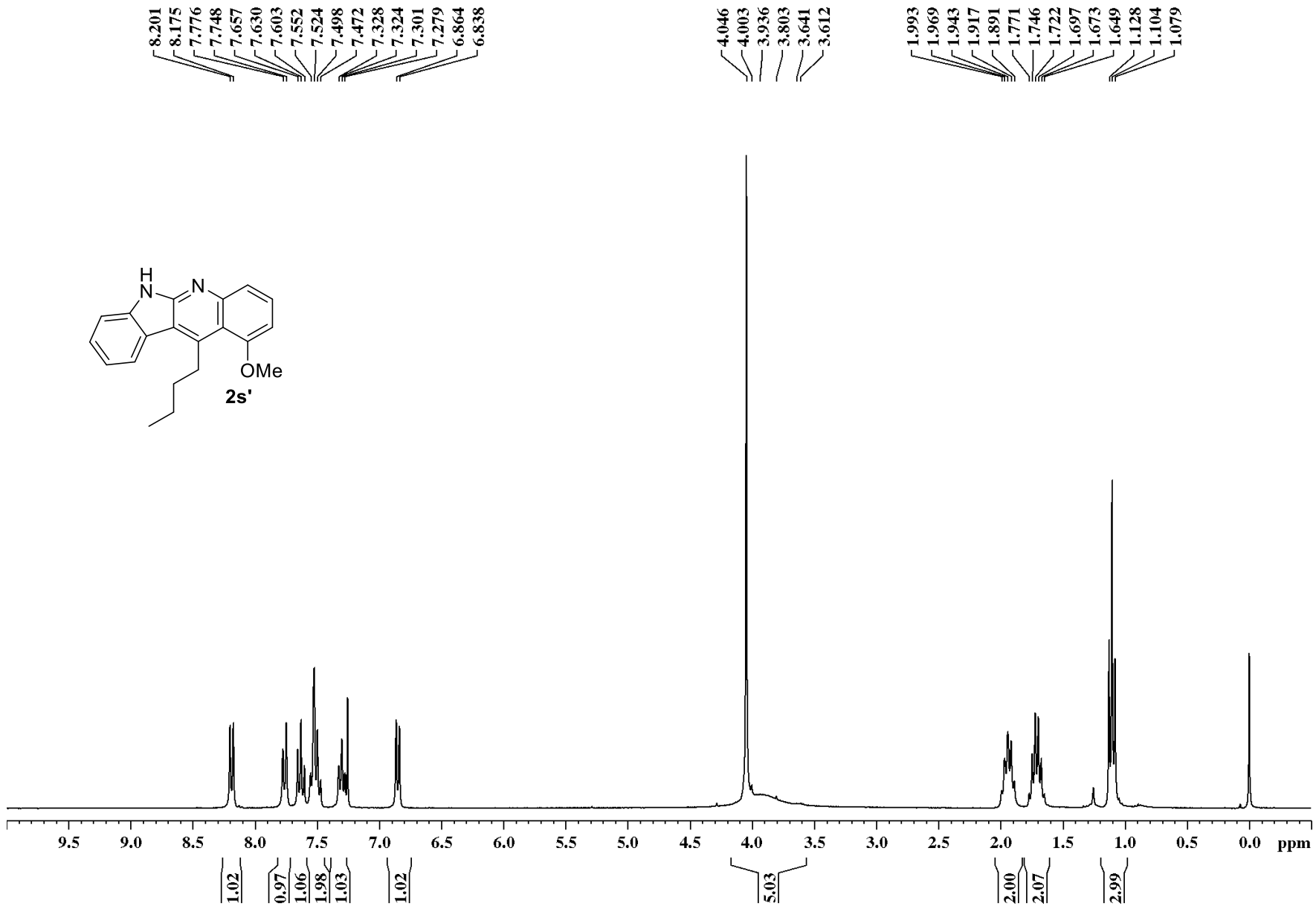
0
ppm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4.003
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3.641
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1.993
1.969
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1.891
1.771
1.746
1.722
1.697
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1.128
1.104
1.079
H
N
N
OMe
2s'
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8.5
8.0
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7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
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1.0
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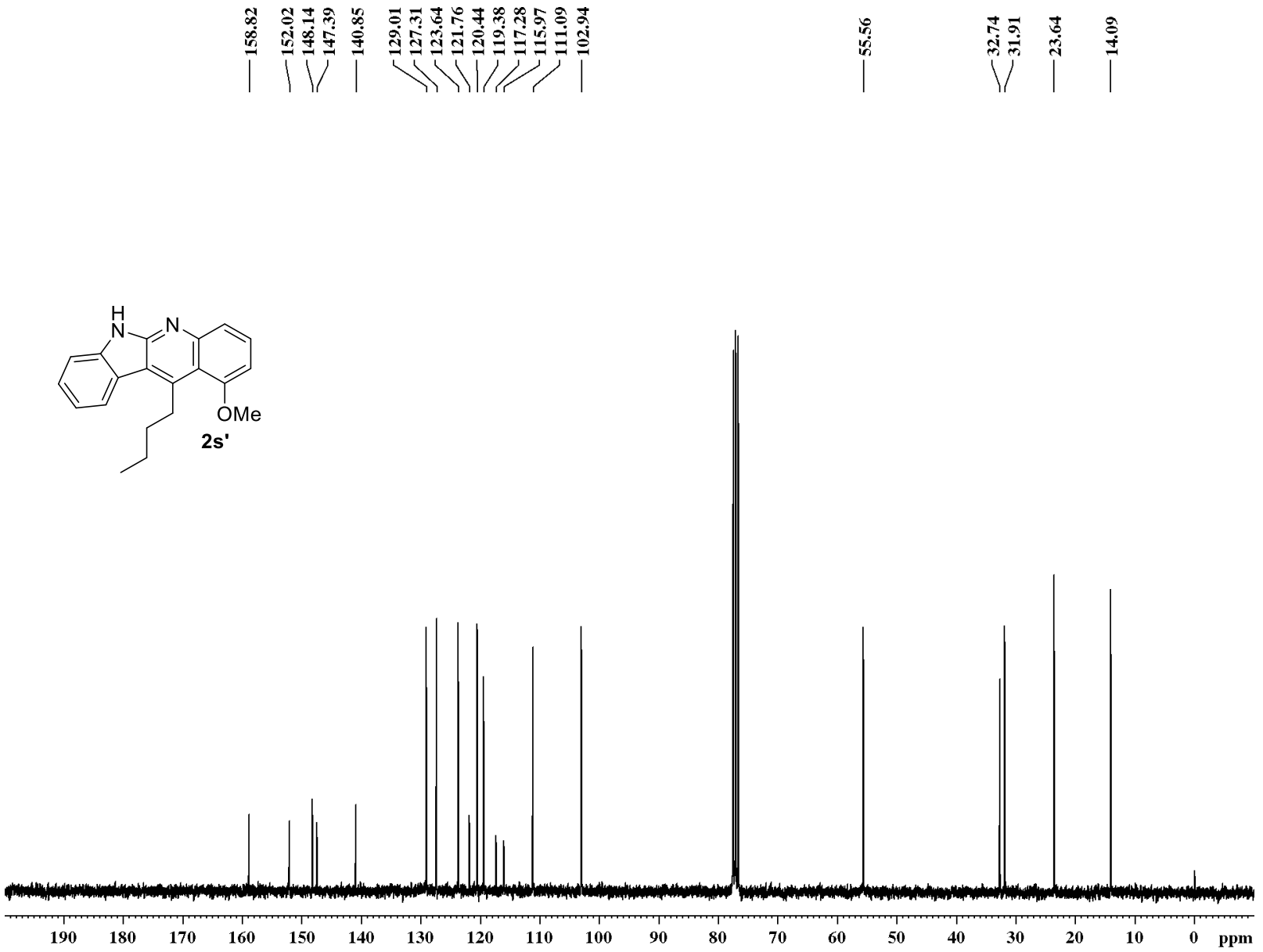
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H
N
N
OMe
2s'
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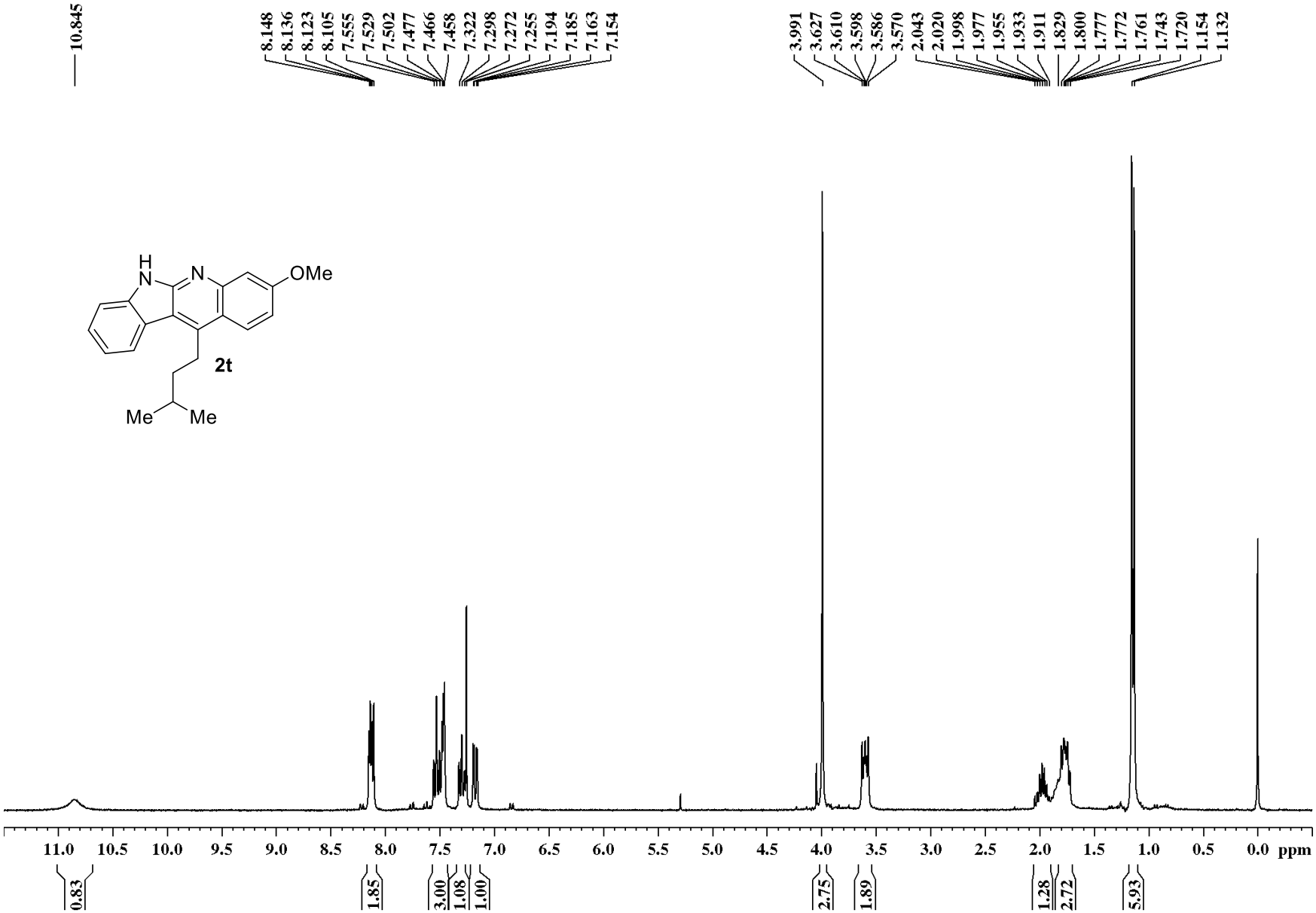
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7.255
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3.570
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1.911
1.829
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N
OMe
2t
Me
Me
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10.0
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6.0
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2.0
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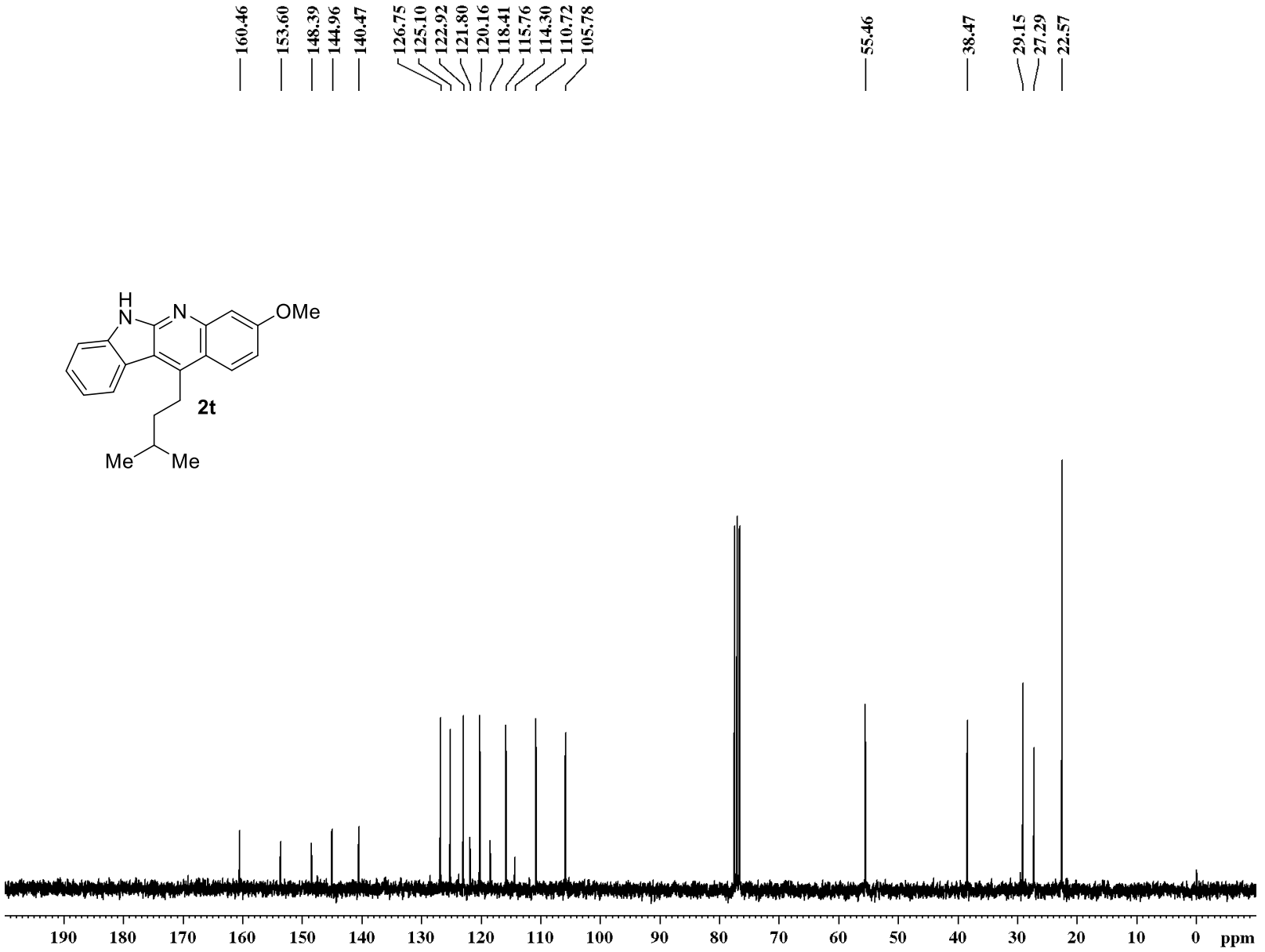
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55.46
38.47
29.15
27.29
22.57
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OMe
2t
Me
Me
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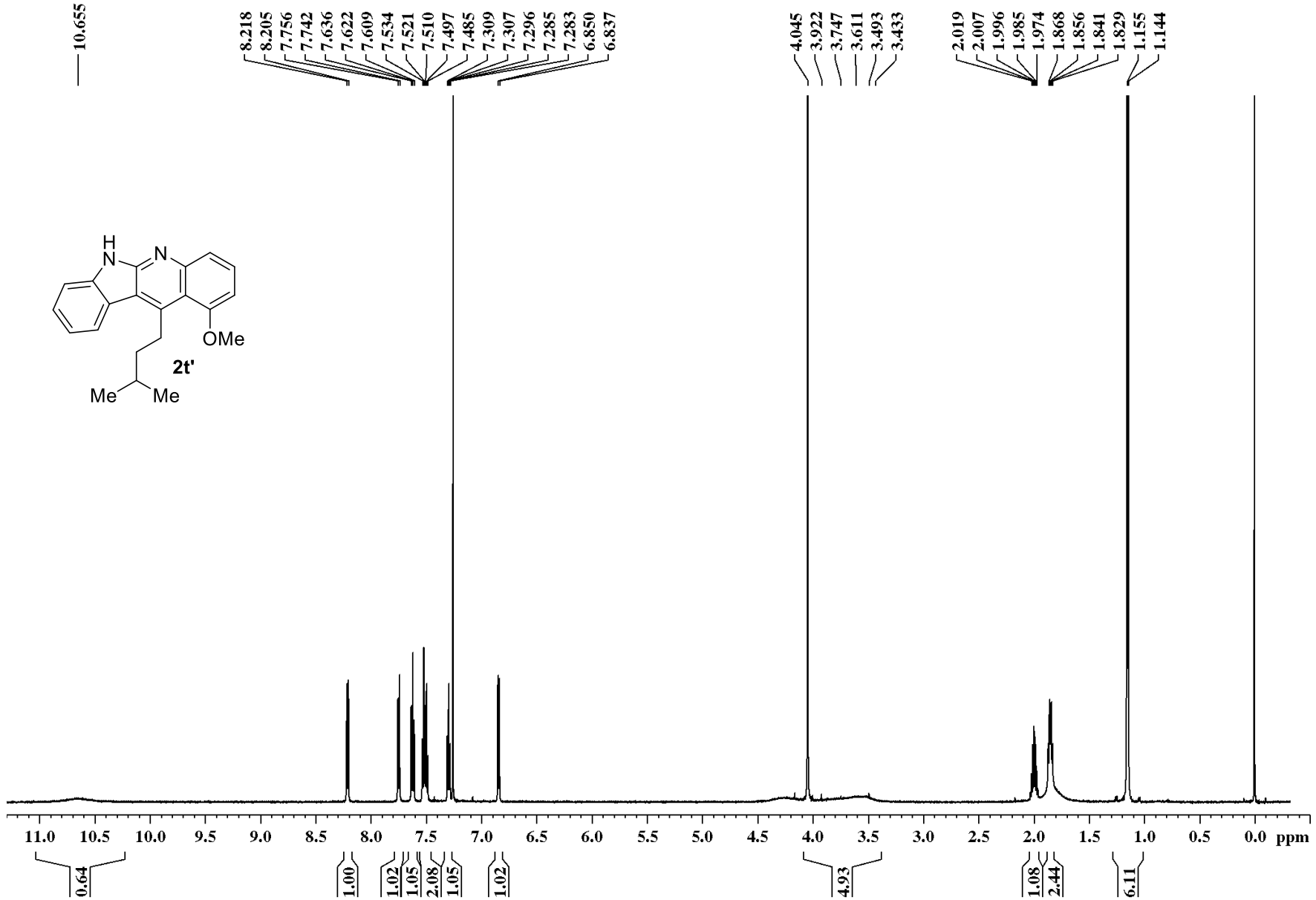
0
ppm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7.485
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7.307
7.296
7.285
7.283
6.850
6.837
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3.922
3.747
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3.493
3.433
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N
N
OMe
2t'
Me
Me
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9.0
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4.0
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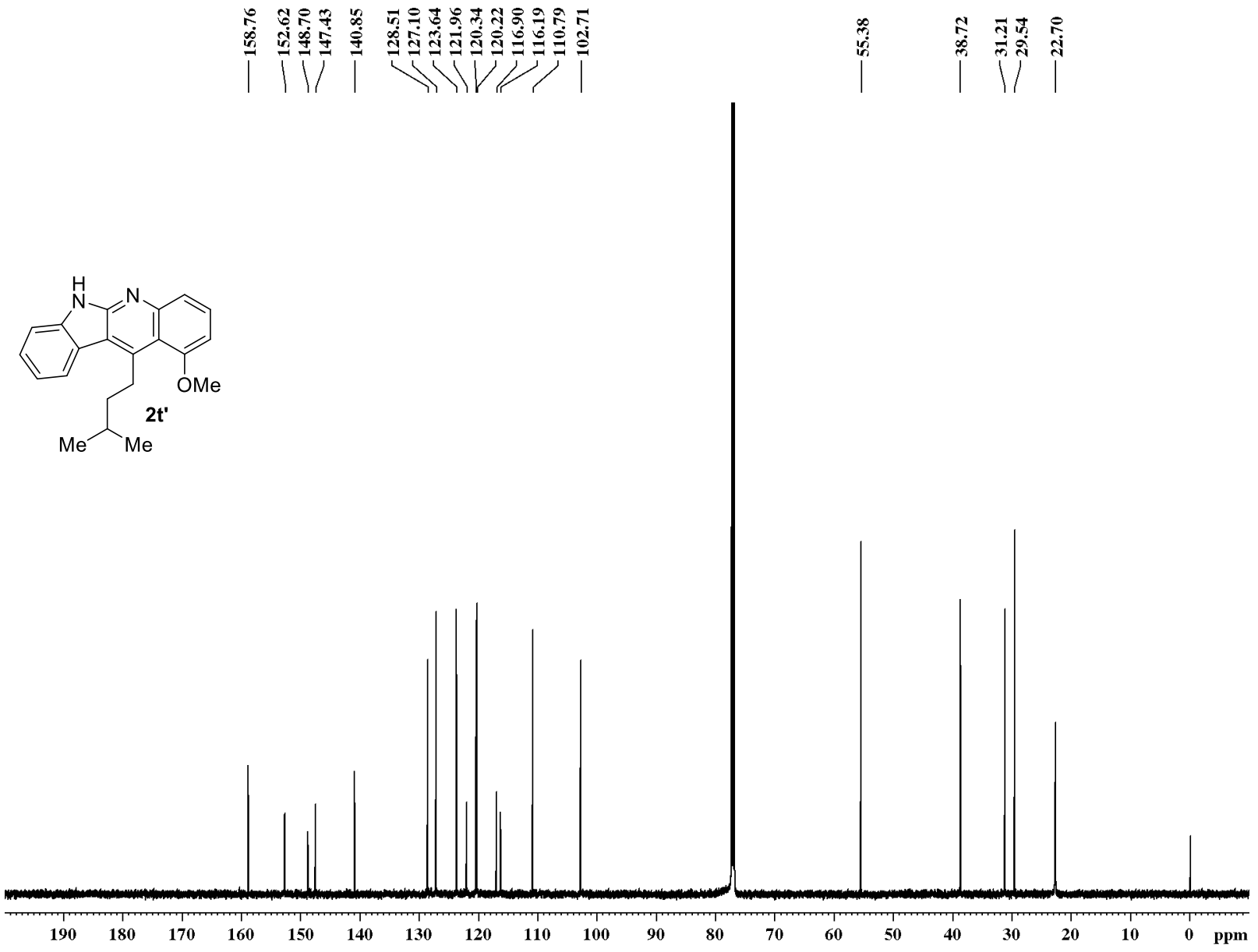
6.11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123.64
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120.34
120.22
116.90
116.19
110.79
102.71
55.38
38.72
31.21
29.54
22.70
H
N
N
OMe
2t'
Me
Me
190
180
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140
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120
110
100
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80
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60
50
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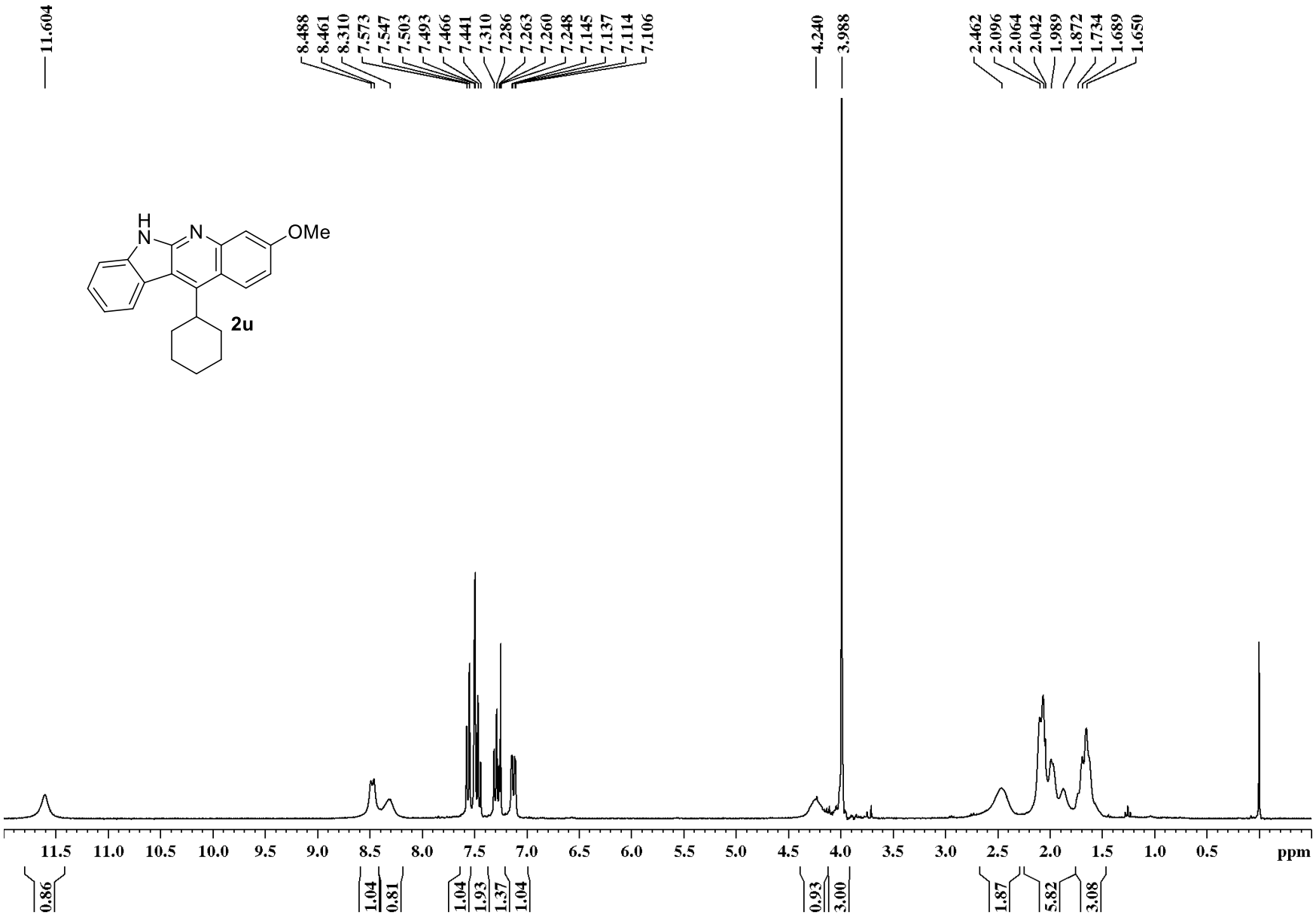
10
0
ppm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7.493
7.466
7.441
7.310
7.286
7.263
7.260
7.248
7.145
7.137
7.114
7.106
4.240
3.988
2.462
2.096
2.064
2.042
1.989
1.872
1.734
1.689
1.650
H
N
N
OMe
2u
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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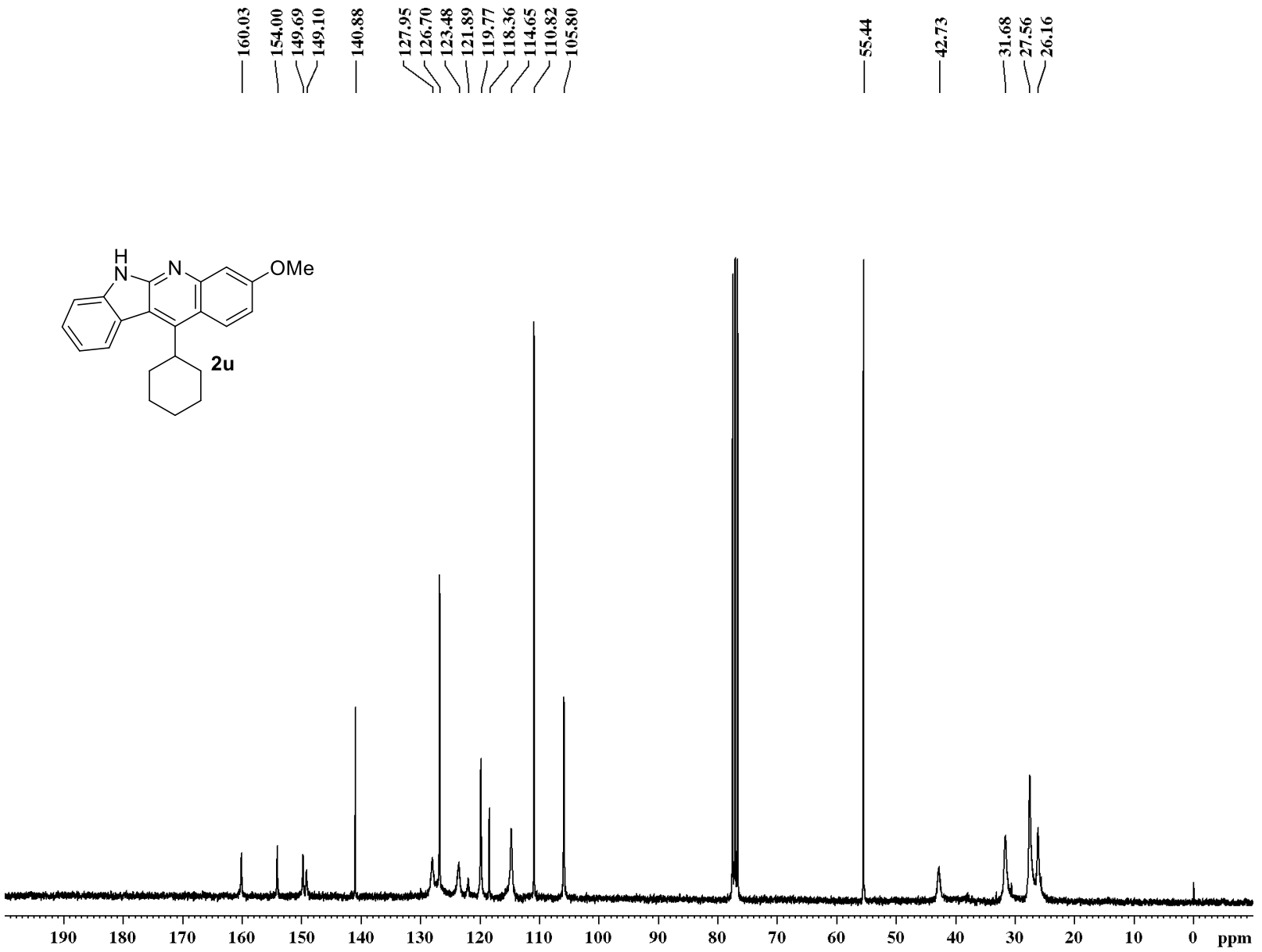
0.81
1.04
1.93
1.37
1.04
0.93
3.00
1.87
5.82
3.08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114.65
110.82
105.80
55.44
42.73
31.68
27.56
26.16
H
N
N
OMe
2u
190
180
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160
150
140
130
120
110
100
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80
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60
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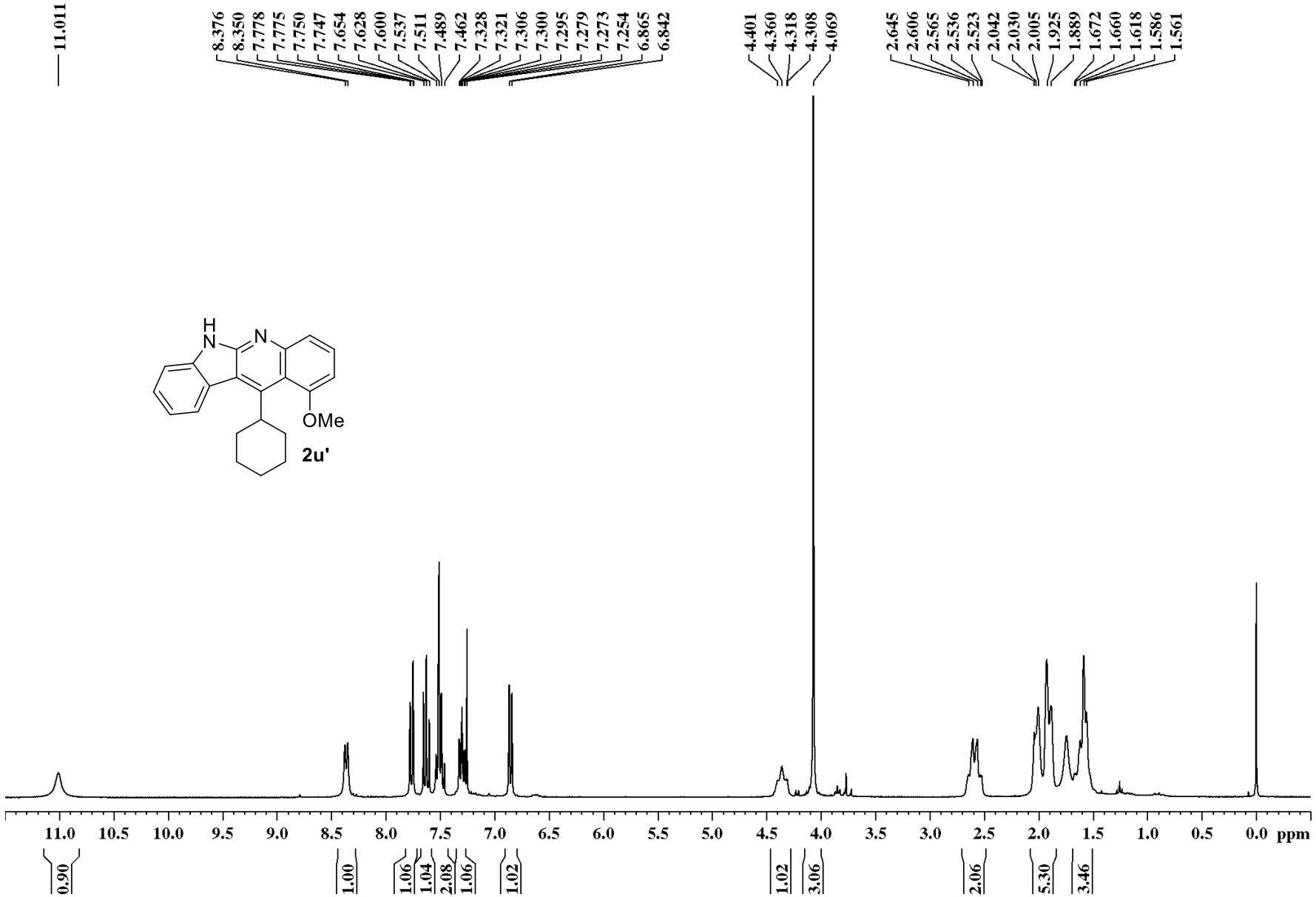
40
30
20
10
0
ppm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7.654
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7.600
7.537
7.511
7.489
7.462
7.328
7.321
7.306
7.300
7.295
7.279
7.273
7.254
6.865
6.842
4.401
4.360
4.318
4.308
4.069
2.645
2.606
2.565
2.536
2.523
2.042
2.030
2.005
1.925
1.889
1.672
1.660
1.618
1.586
1.561
H
N
N
OMe
2u'
11.0
10.5
10.0
9.5
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
0.0 ppm
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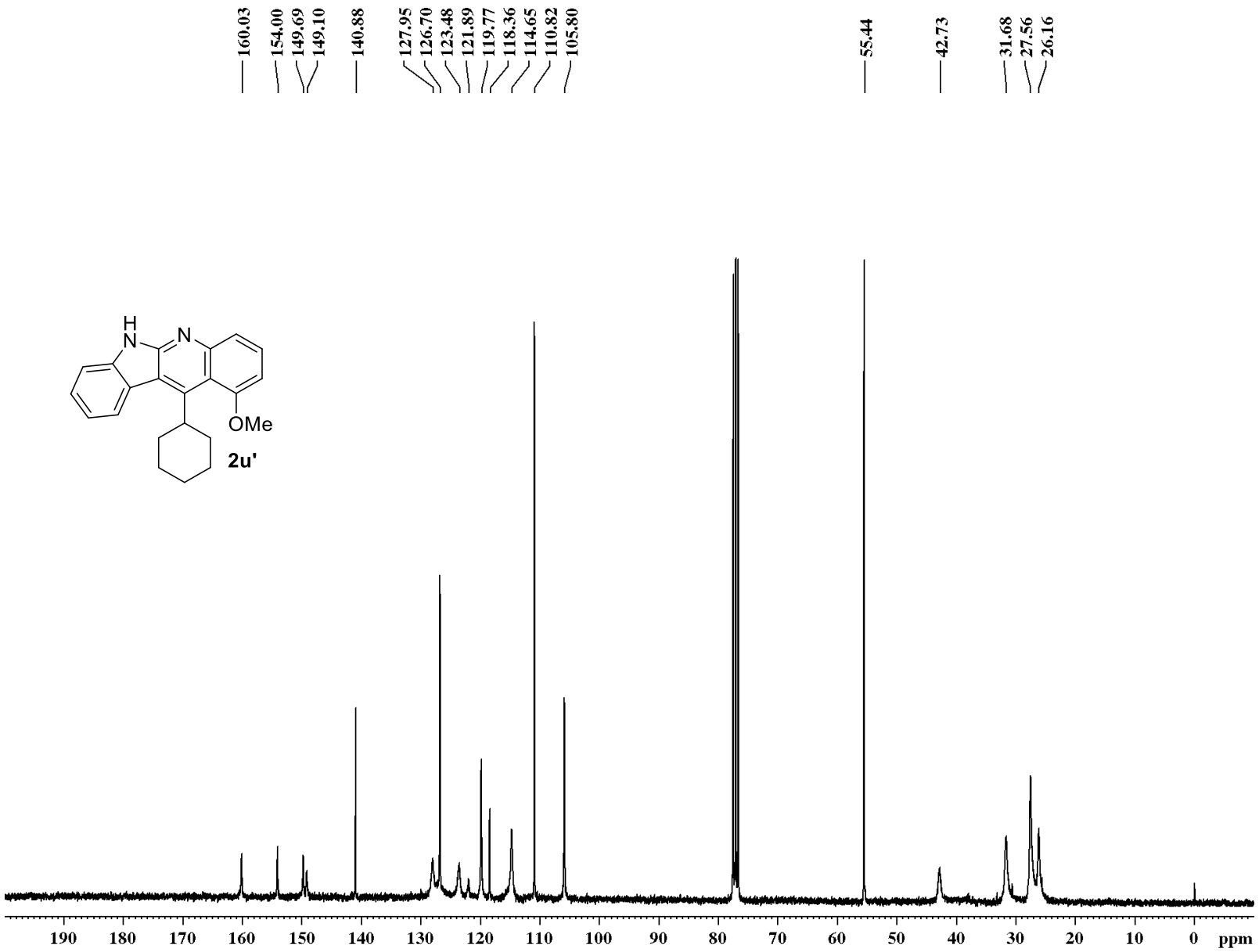
1.06
1.04
2.08
1.06
1.02
1.02
3.06
2.06
5.30
3.46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149.69
149.10
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127.95
126.70
123.48
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119.77
118.36
114.65
110.82
105.80
55.44
42.73
31.68
27.56
26.16
H
N
N
OMe
2u'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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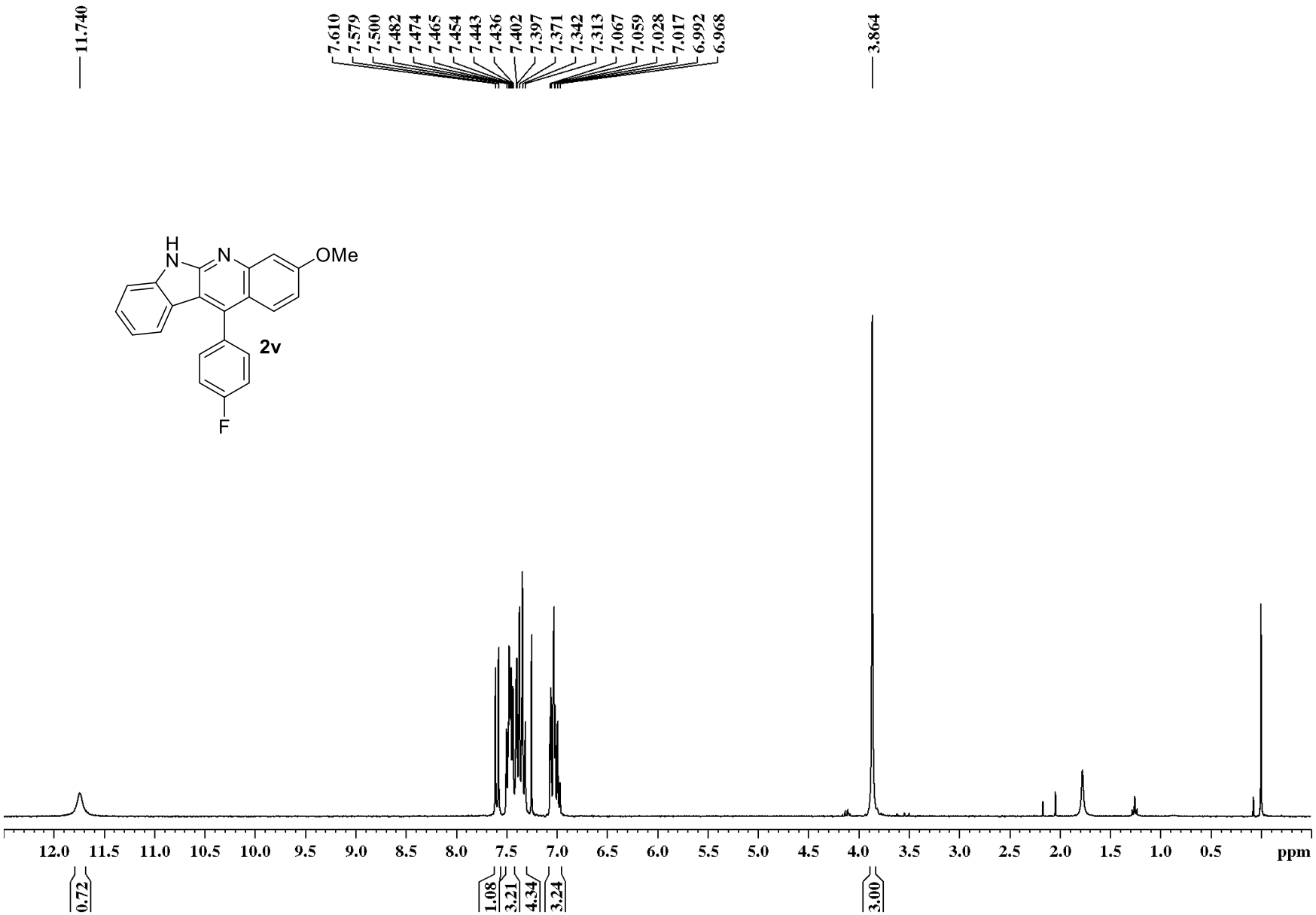
30
20
10
0
ppm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7.500
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7.474
7.465
7.454
7.443
7.436
7.402
7.397
7.371
7.342
7.313
7.067
7.059
7.028
7.017
6.992
6.968
3.864
H
N
N
OMe
F
2v
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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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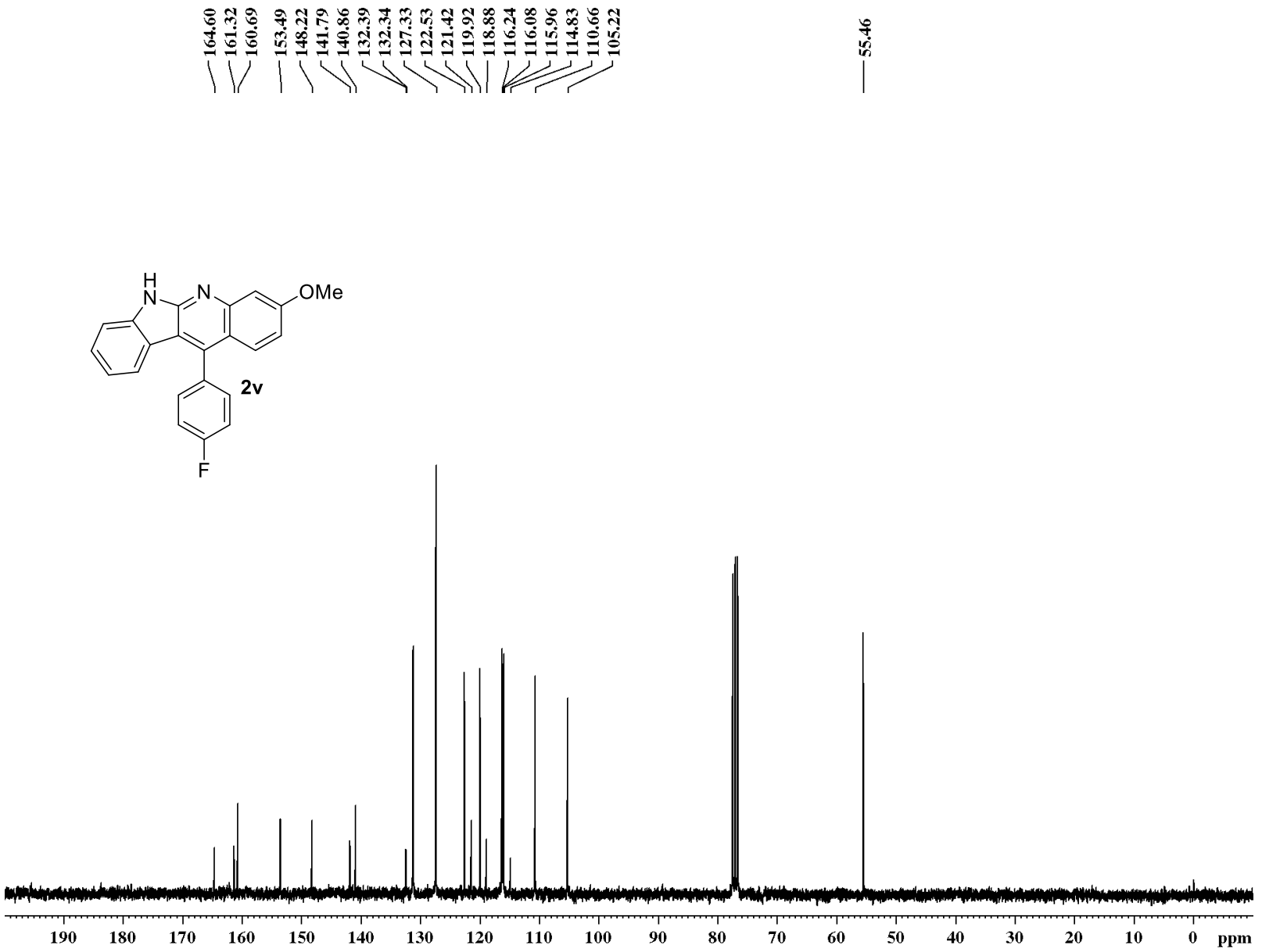
1.08
3.21
4.34
3.24
3.00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153.49
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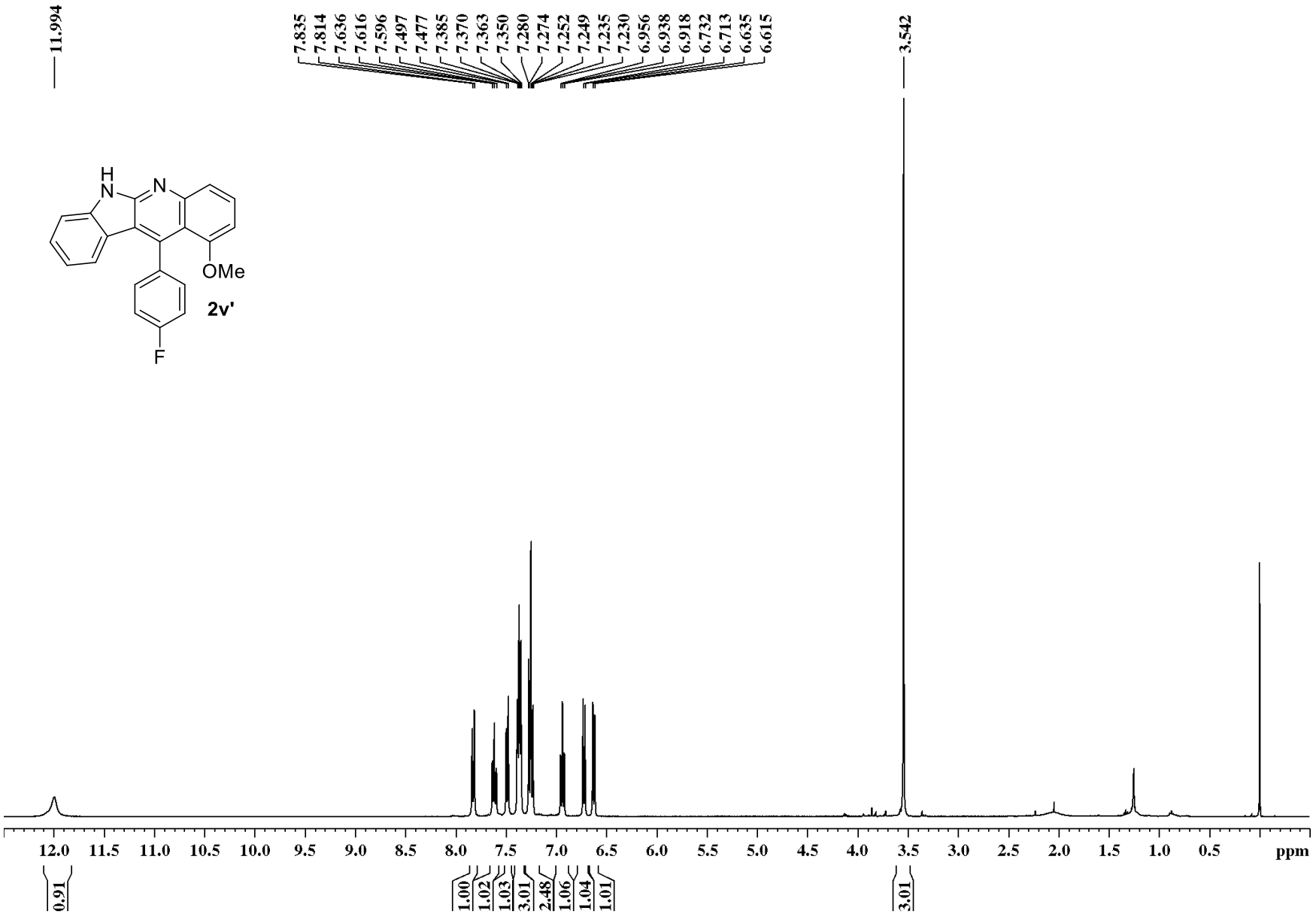
141.79
140.86
132.39
132.34
127.33
122.53
121.42
119.92
118.88
116.24
116.08
115.96
114.83
110.66
105.22
55.46
H
N
N
OMe
2v
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.616
7.596
7.497
7.477
7.385
7.370
7.363
7.350
7.280
7.274
7.252
7.249
7.235
7.230
6.956
6.938
6.918
6.732
6.713
6.635
6.615
3.542
H
N
N
OMe
2v'
F
12.0
11.5
11.0
10.5
10.0
9.5
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
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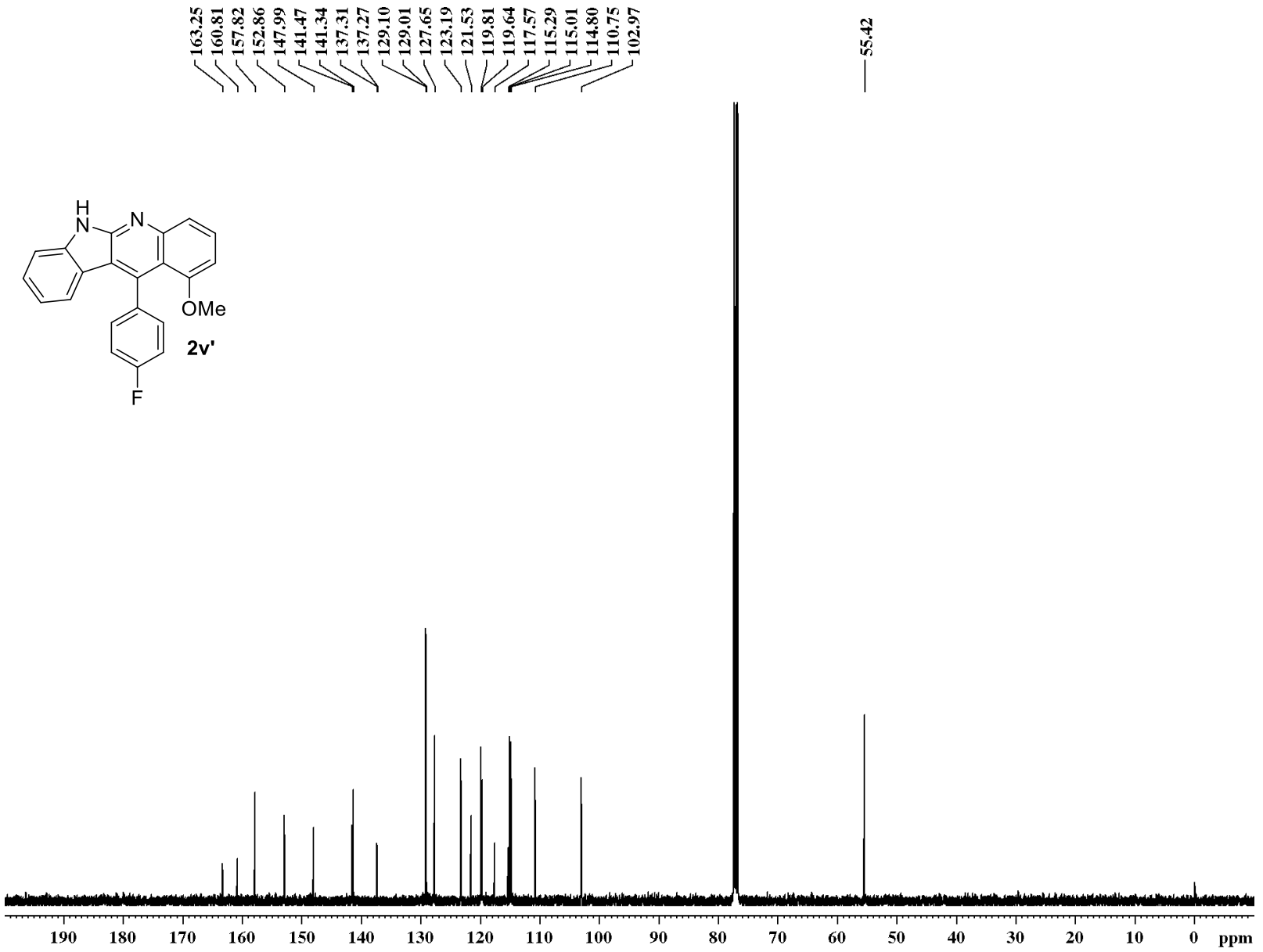
1.00
1.02
1.03
3.01
2.48
1.06
1.04
1.01
3.01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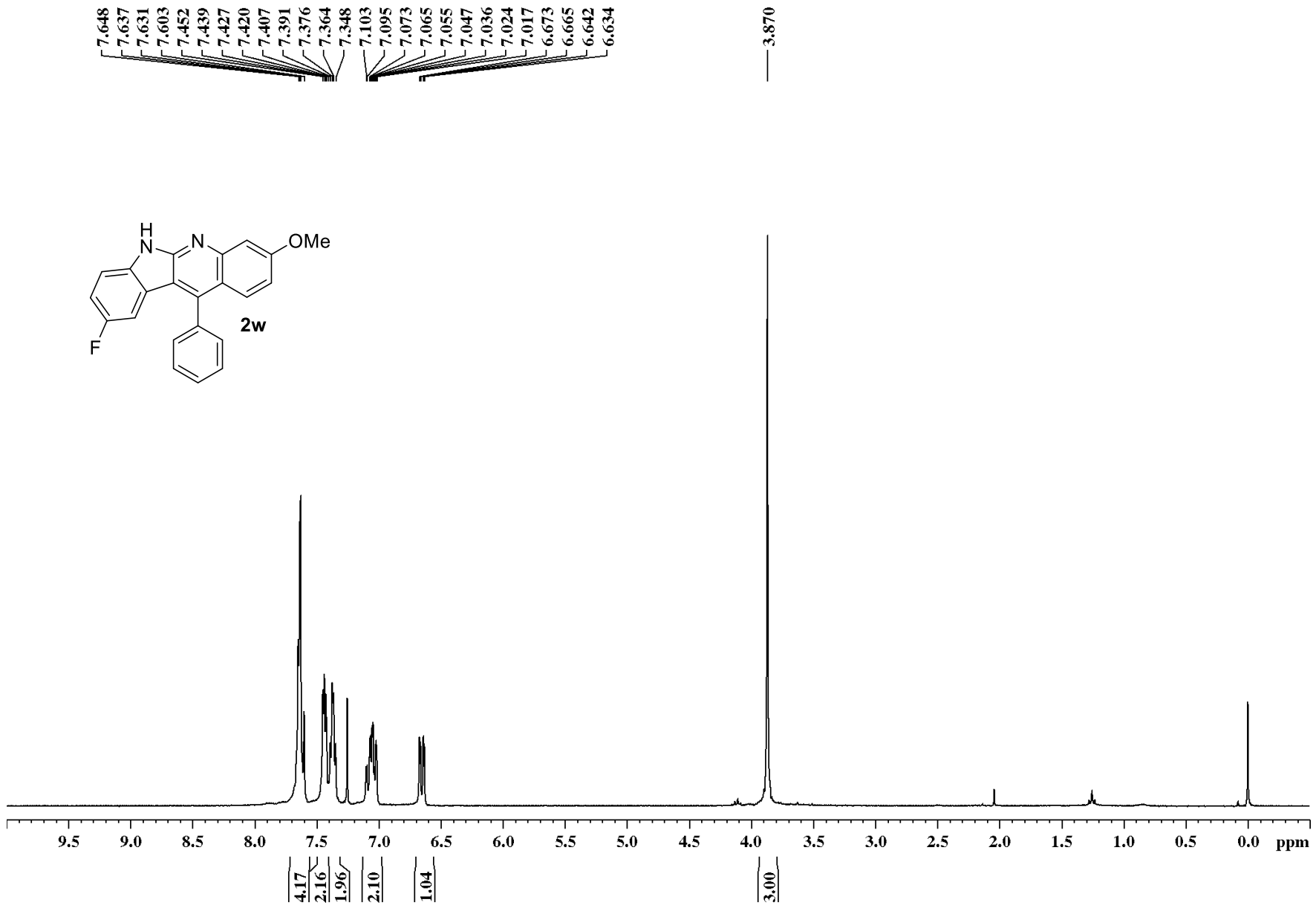
141.34
137.31
137.27
129.10
129.01
127.65
123.19
121.53
119.81
119.64
117.57
115.29
115.01
114.80
110.75
102.97
55.42
H
N
N
OMe
2v'
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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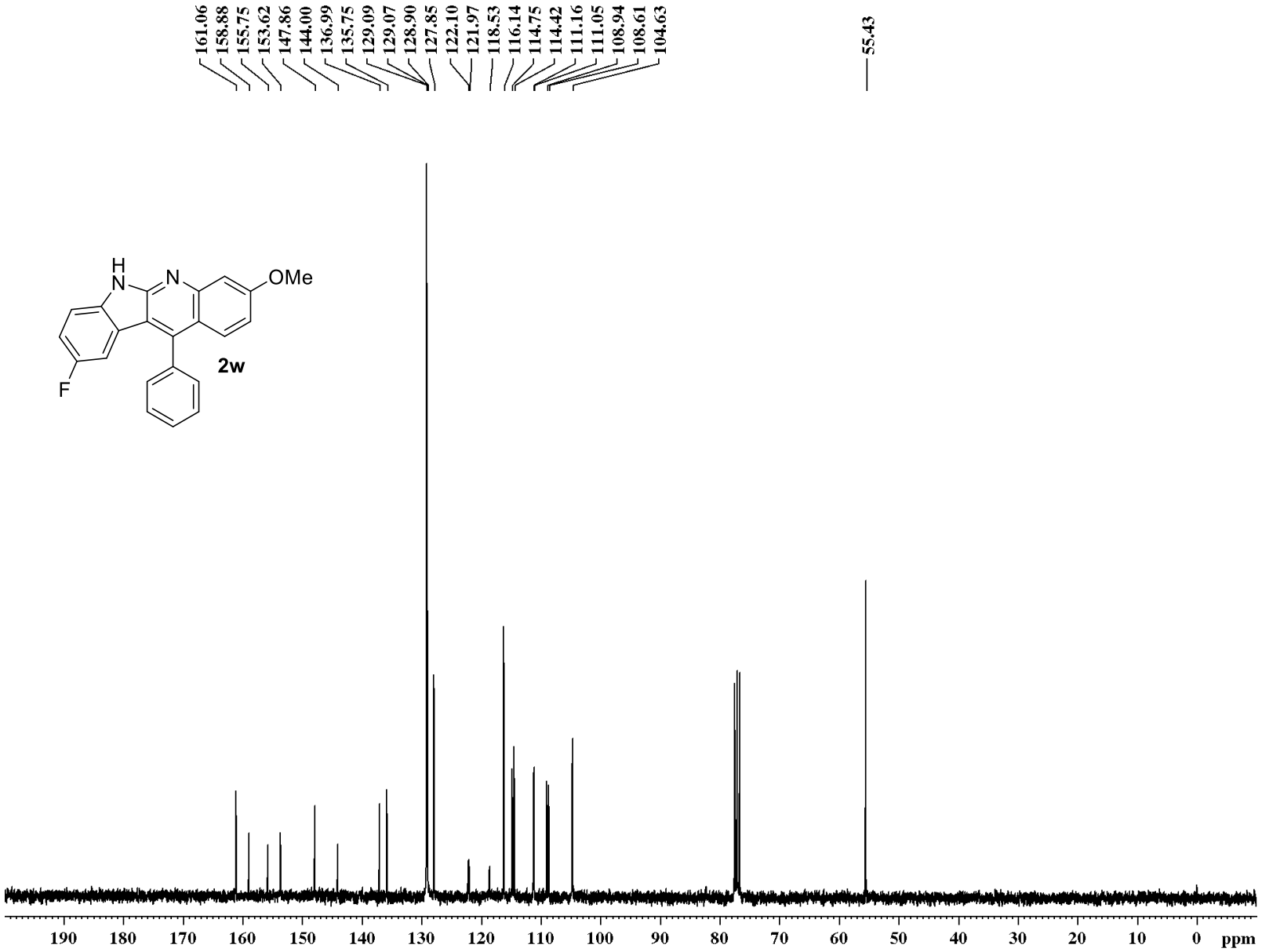
7.439
7.427
7.420
7.407
7.391
7.376
7.364
7.348
7.103
7.095
7.073
7.065
7.055
7.047
7.036
7.024
7.017
6.673
6.665
6.642
6.634
3.870
H
N
N
OMe
F
2w
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.17
2.16
1.96
2.10
1.04
3.00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144.00
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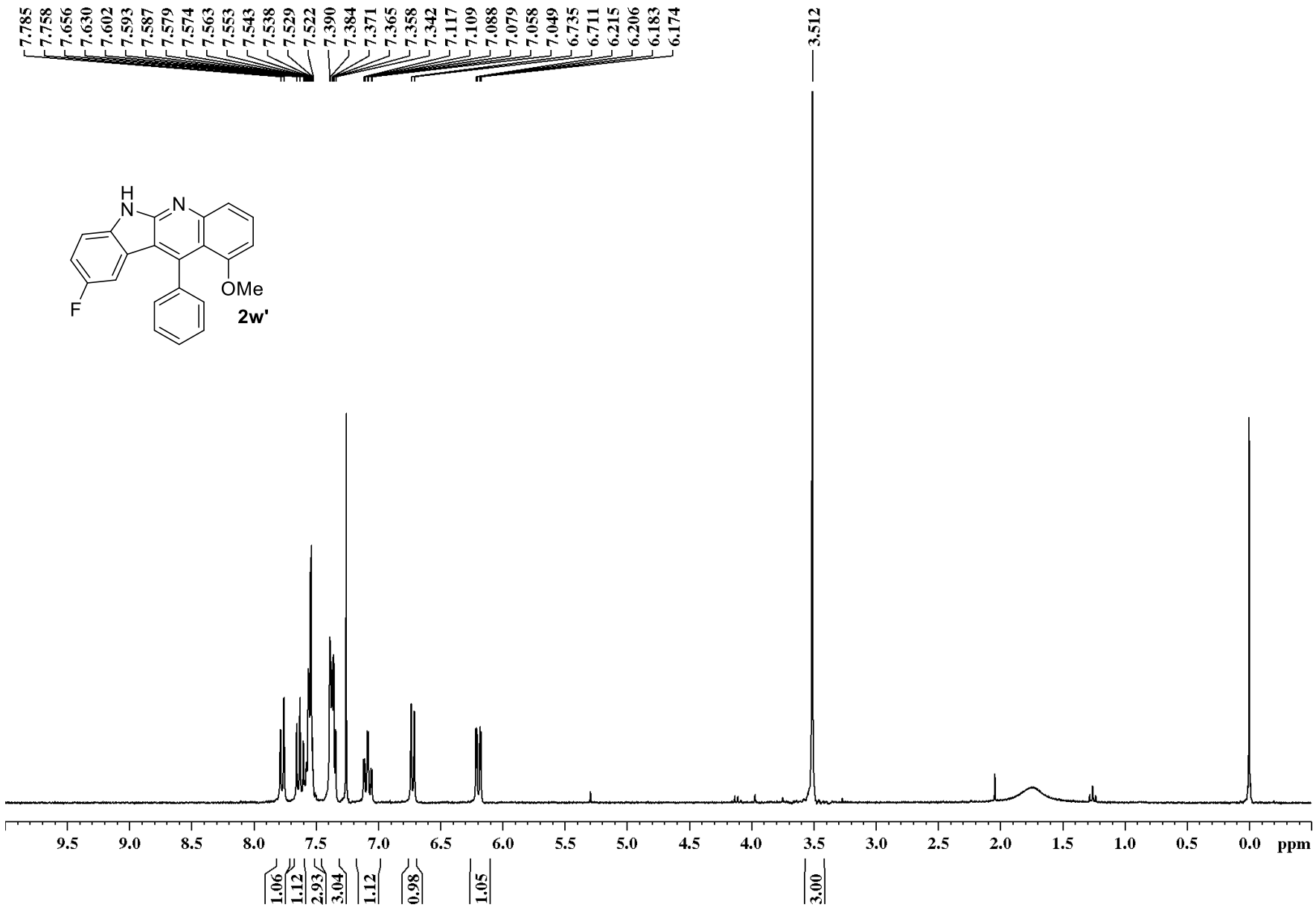
129.07
128.90
127.85
122.10
121.97
118.53
116.14
114.75
114.42
111.16
111.05
108.94
108.61
104.63
55.43
H
N
N
OMe
F
2w
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.593
7.587
7.579
7.574
7.563
7.553
7.543
7.538
7.529
7.522
7.390
7.384
7.371
7.365
7.358
7.342
7.117
7.109
7.088
7.079
7.058
7.049
6.735
6.711
6.215
6.206
6.183
6.174
3.512
H
N
N
OMe
F
2w'
9.5
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
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0.0
ppm
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3.04
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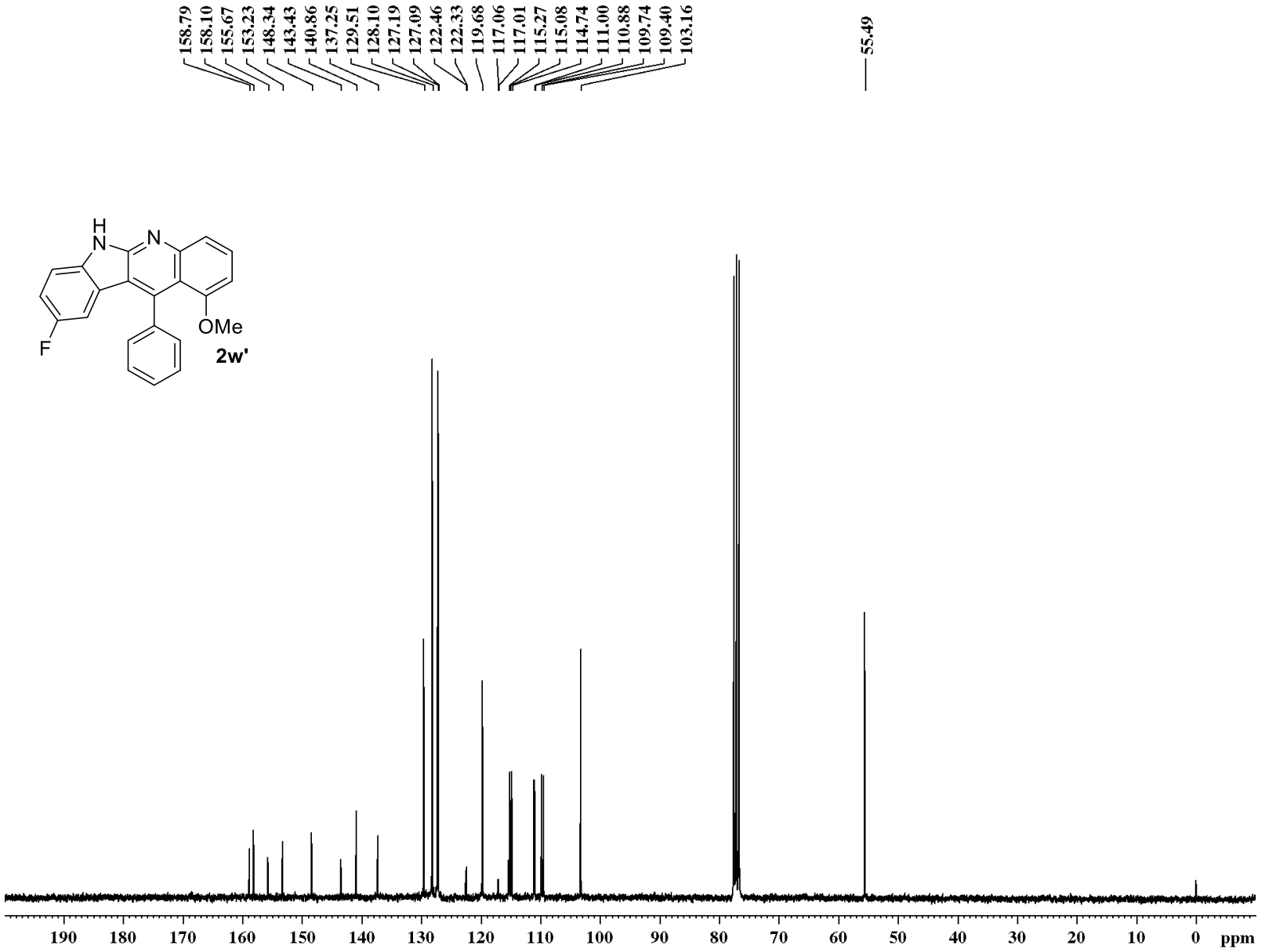
0.98
1.05
3.00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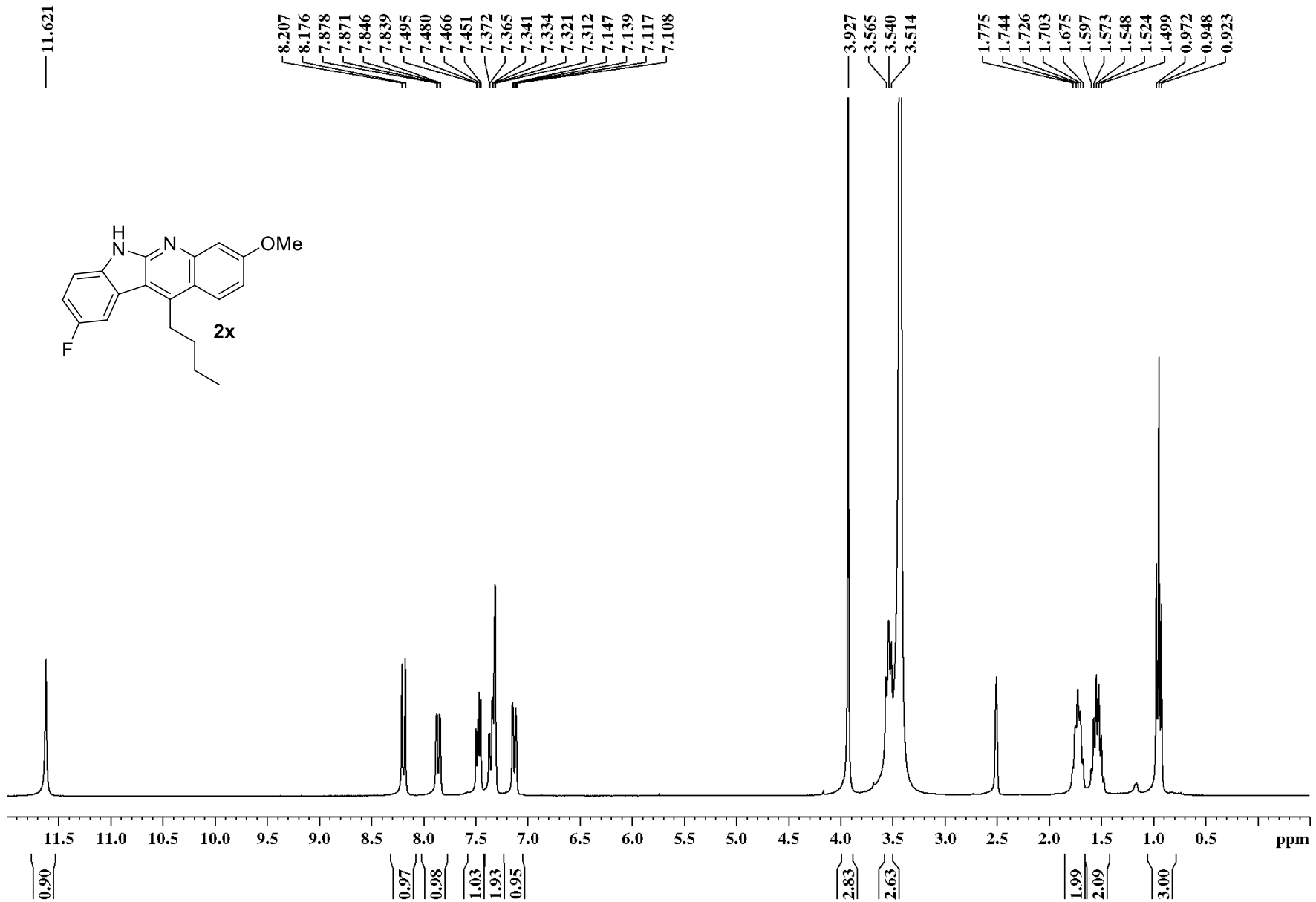
127.19
127.09
122.46
122.33
119.68
117.06
117.01
115.27
115.08
114.74
111.00
110.88
109.74
109.40
103.16
55.49
H
N
N
F
OMe
2w'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.466
7.451
7.372
7.365
7.341
7.334
7.321
7.312
7.147
7.139
7.117
7.108
3.927
3.565
3.540
3.514
1.775
1.744
1.726
1.703
1.675
1.597
1.573
1.548
1.524
1.499
0.972
0.948
0.923
H
N
N
OMe
F
2x
11.5 11.0 10.5 10.0 9.5 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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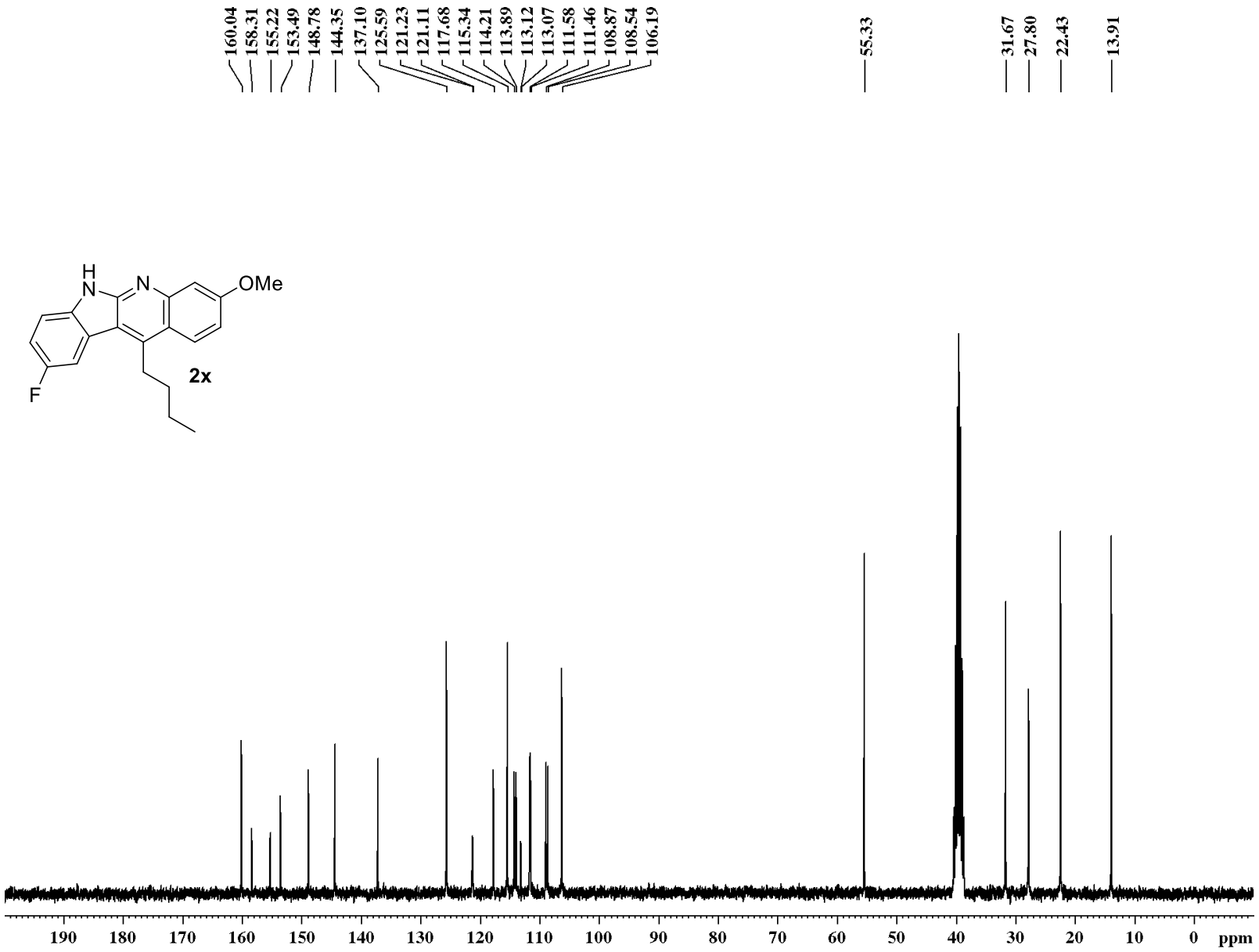
0.97
0.98
1.03
1.93
0.95
2.83
2.63
1.99
2.09
3.00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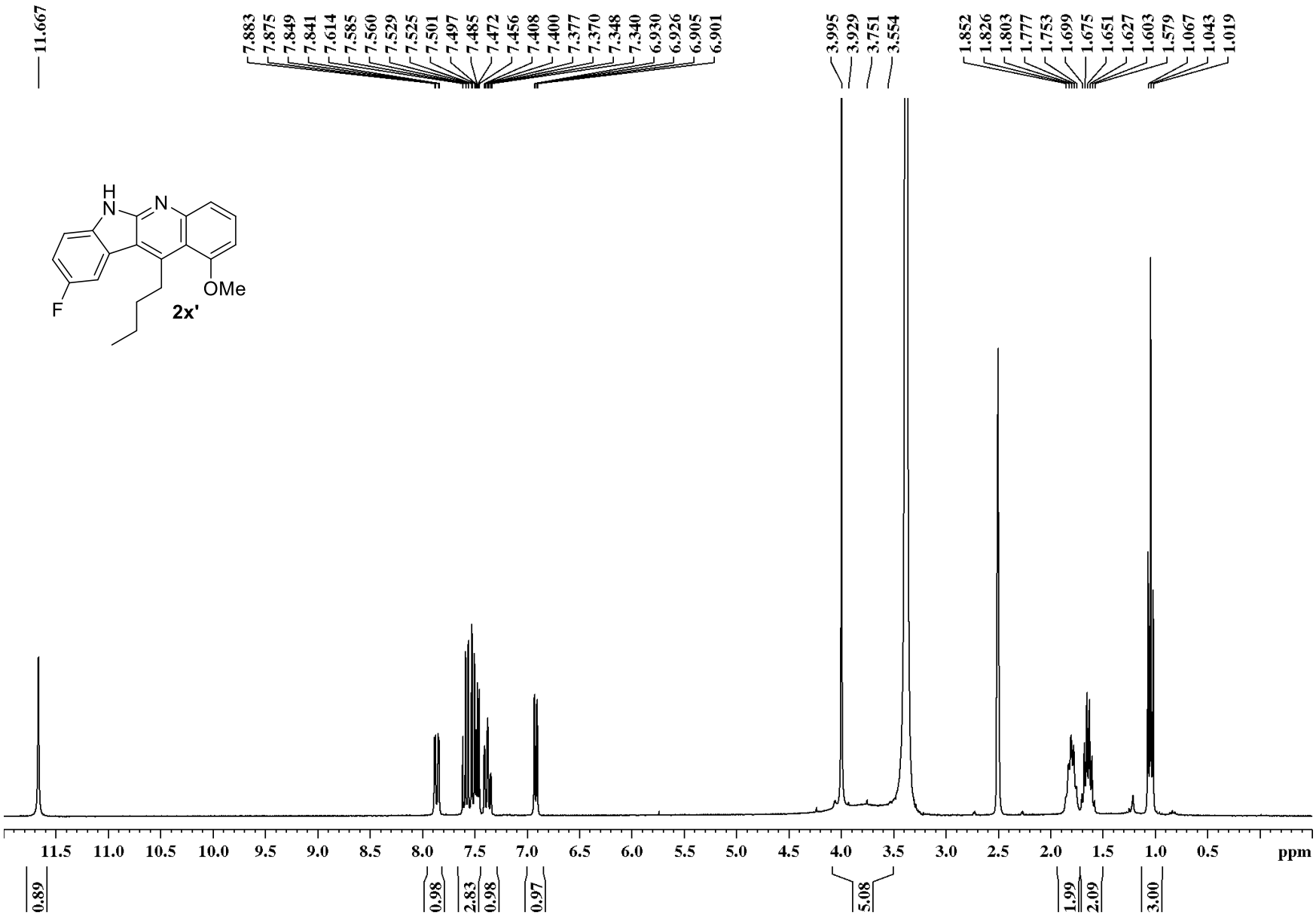
137.10
125.59
121.23
121.11
117.68
115.34
114.21
113.89
113.12
113.07
111.58
111.46
108.87
108.54
106.19
55.33
31.67
27.80
22.43
13.91
H
N
N
OMe
F
2x
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.614
7.585
7.560
7.529
7.525
7.501
7.497
7.485
7.472
7.456
7.408
7.400
7.377
7.370
7.348
7.340
6.930
6.926
6.905
6.901
3.995
3.929
3.751
3.554
1.852
1.826
1.803
1.777
1.753
1.699
1.675
1.651
1.627
1.603
1.579
1.067
1.043
1.019
H
N
N
OMe
F
2x'
11.5
11.0
10.5
10.0
9.5
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
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ppm
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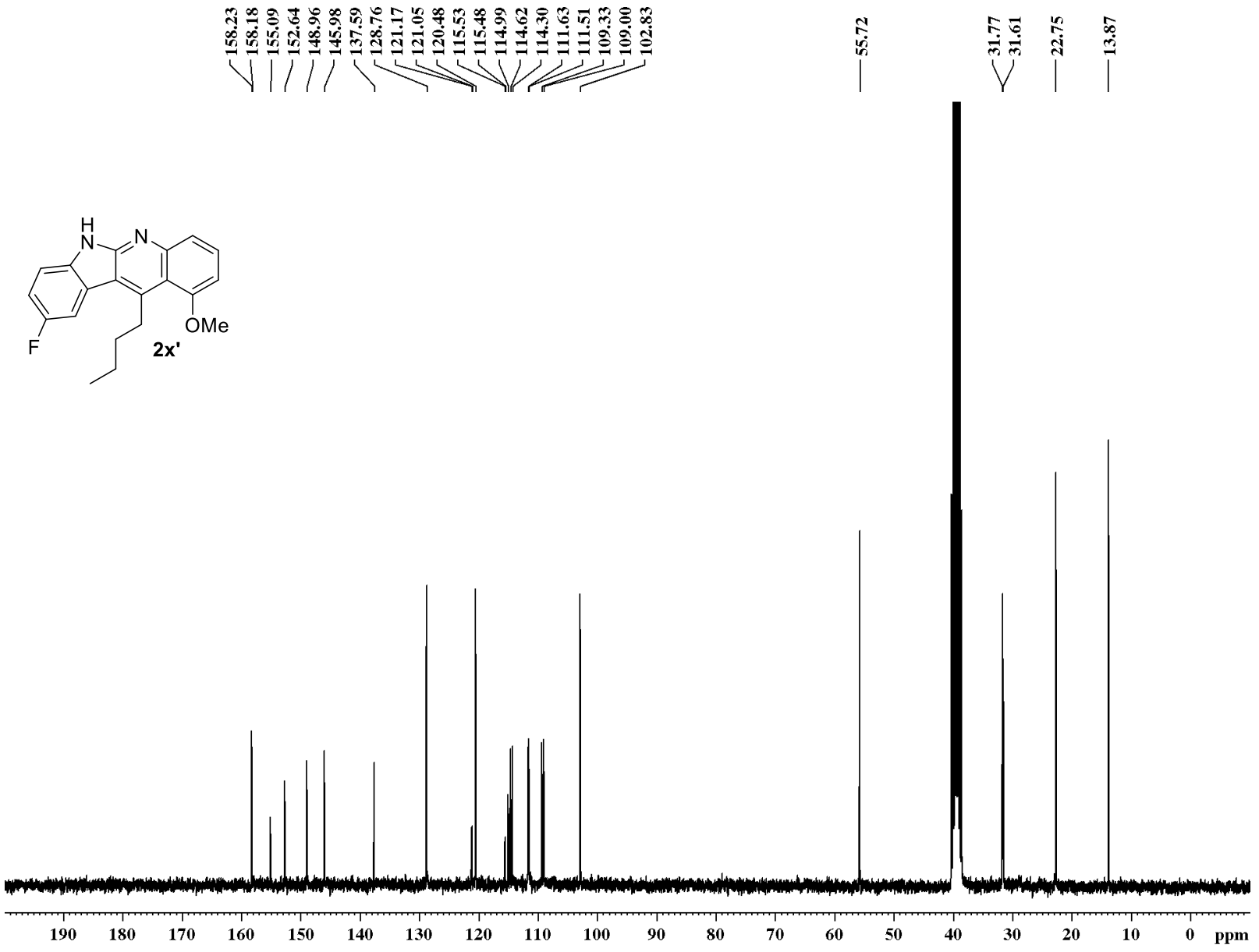
0.98
0.97
5.08
1.99
2.09
3.00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152.64
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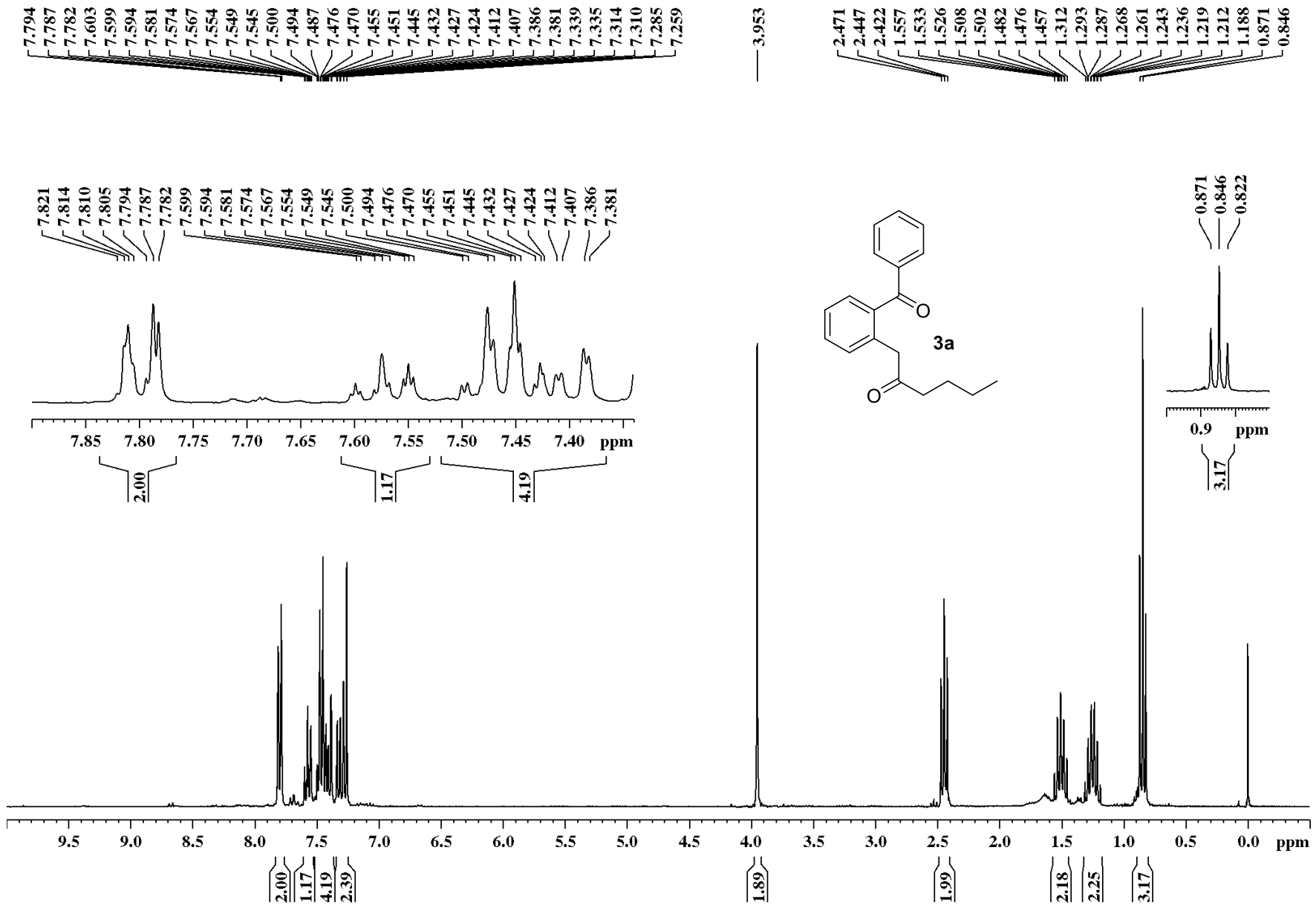
145.98
137.59
128.76
121.17
121.05
120.48
115.53
115.48
114.99
114.62
114.30
111.63
111.51
109.33
109.00
102.83
55.72
31.77
31.61
22.75
13.87
H
N
N
F
OMe
2x'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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3.953
2.471 2.447 2.422 1.557 1.533 1.526 1.508 1.502 1.482 1.476 1.457 1.312 1.293 1.287 1.268 1.261 1.243 1.236 1.219 1.212 1.188 0.871 0.846
7.821 7.814 7.810 7.805 7.794 7.787 7.782 7.599 7.594 7.581 7.574 7.567 7.554 7.549 7.545 7.500 7.494 7.476 7.470 7.455 7.451 7.445 7.432 7.427 7.424 7.412 7.407 7.386 7.381
0.871 0.846 0.822
3a
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 ppm
2.00 1.17 4.19
0.9 ppm
3.17
9.5 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 0.0 ppm
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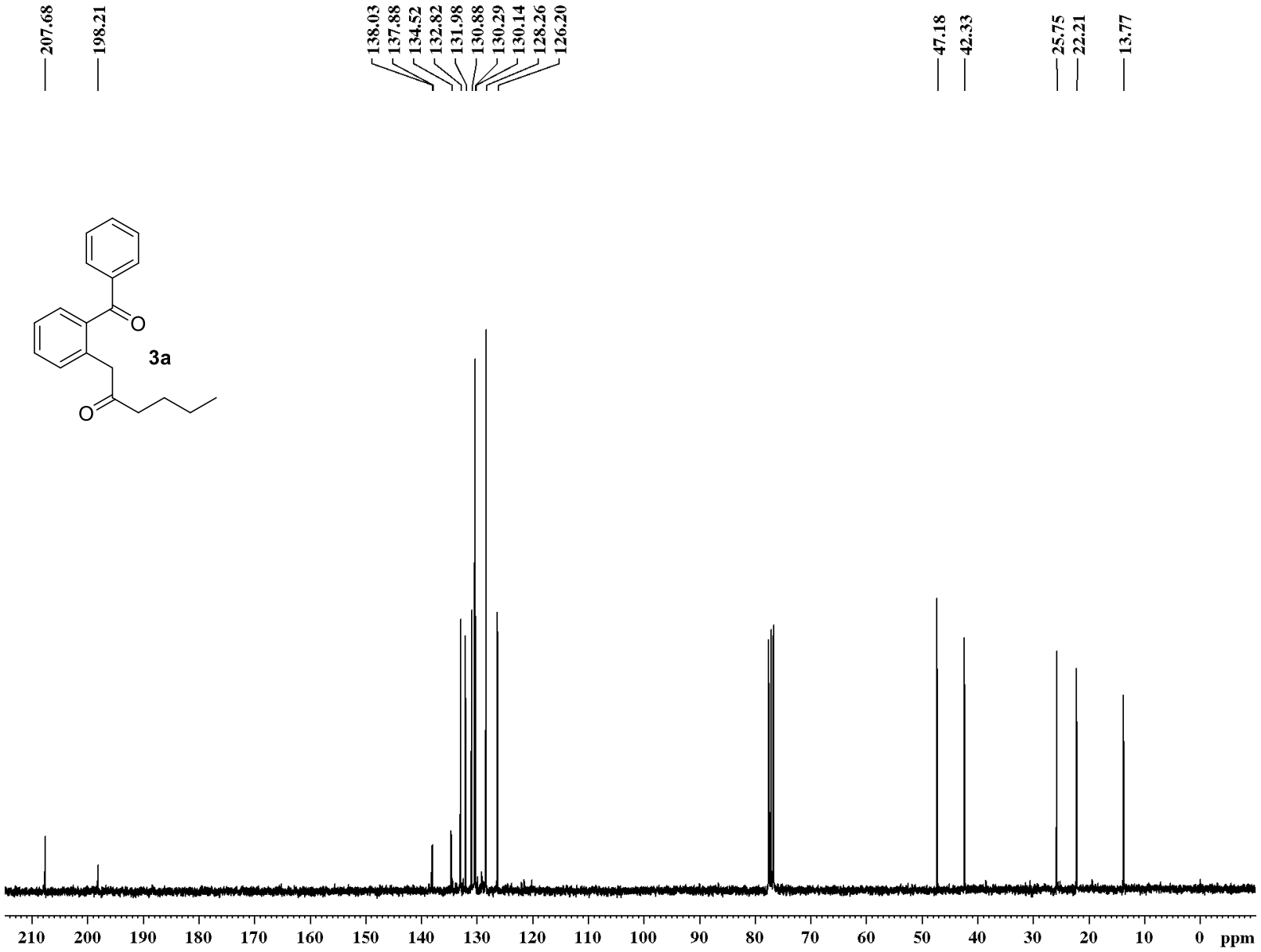
1.89
1.99
2.18 2.25
3.17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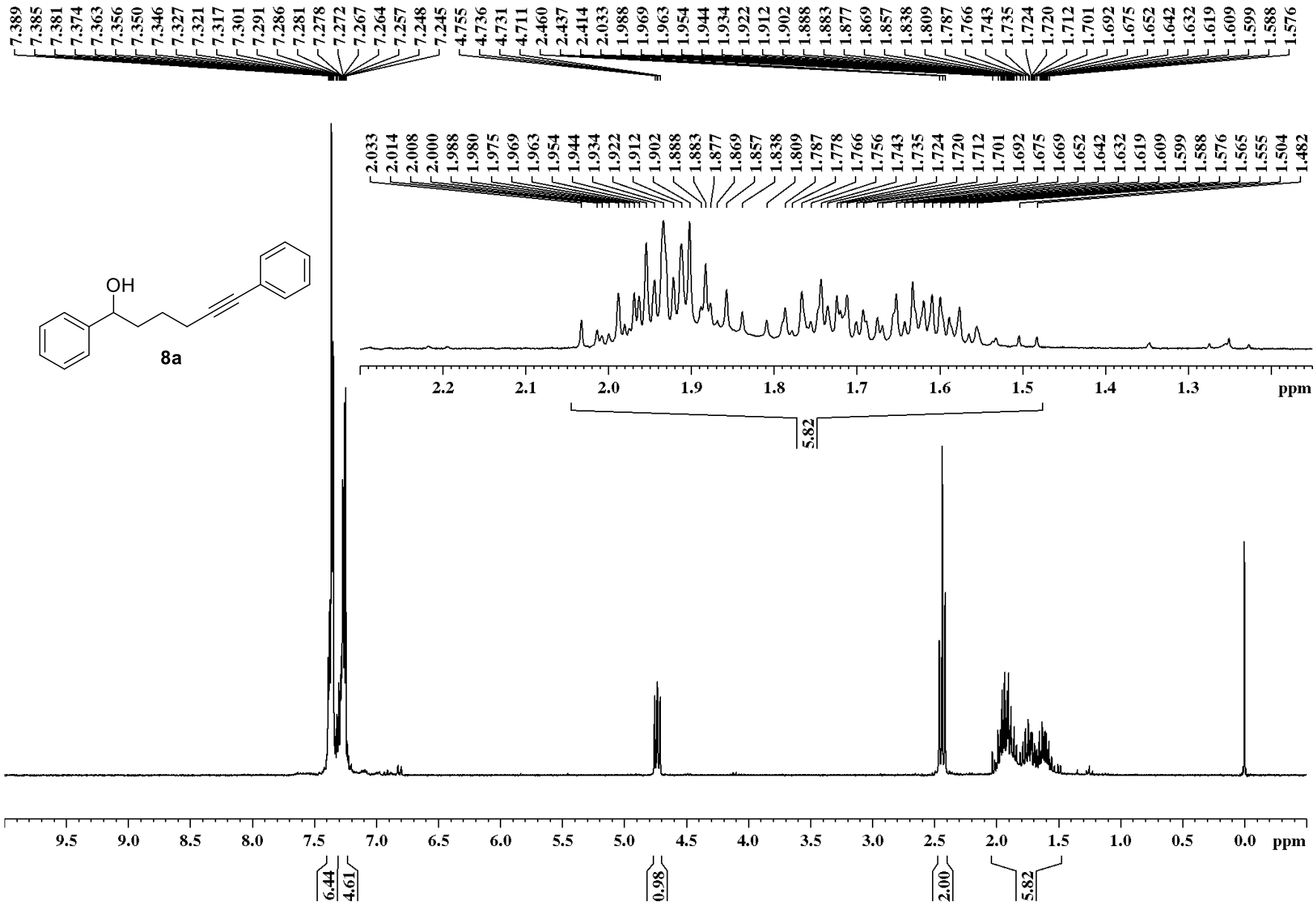
138.03
137.88
134.52
132.82
131.98
130.88
130.29
130.14
128.26
126.20
47.18
42.33
25.75
22.21
13.77
O
O
3a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.389 7.385 7.381 7.374 7.363 7.356 7.350 7.346 7.327 7.321 7.317 7.301 7.291 7.286 7.281 7.278 7.272 7.267 7.264 7.257 7.248 7.245
4.755 4.736 4.731 4.711 2.460 2.437 2.414 2.033 1.988 1.969 1.963 1.954 1.944 1.934 1.922 1.912 1.902 1.888 1.883 1.877 1.869 1.857 1.838 1.809 1.787 1.766 1.743 1.735 1.724 1.720 1.712 1.701 1.692 1.675 1.652 1.642 1.632 1.619 1.609 1.599 1.588 1.576
2.033 2.014 2.008 2.000 1.988 1.980 1.975 1.969 1.963 1.954 1.944 1.934 1.922 1.912 1.902 1.888 1.883 1.877 1.869 1.857 1.838 1.809 1.787 1.778 1.766 1.756 1.743 1.735 1.724 1.720 1.712 1.701 1.692 1.675 1.669 1.652 1.642 1.632 1.619 1.609 1.599 1.588 1.576 1.565 1.555 1.504 1.482
OH
8a
2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 ppm
5.82
9.5 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 0.0 ppm
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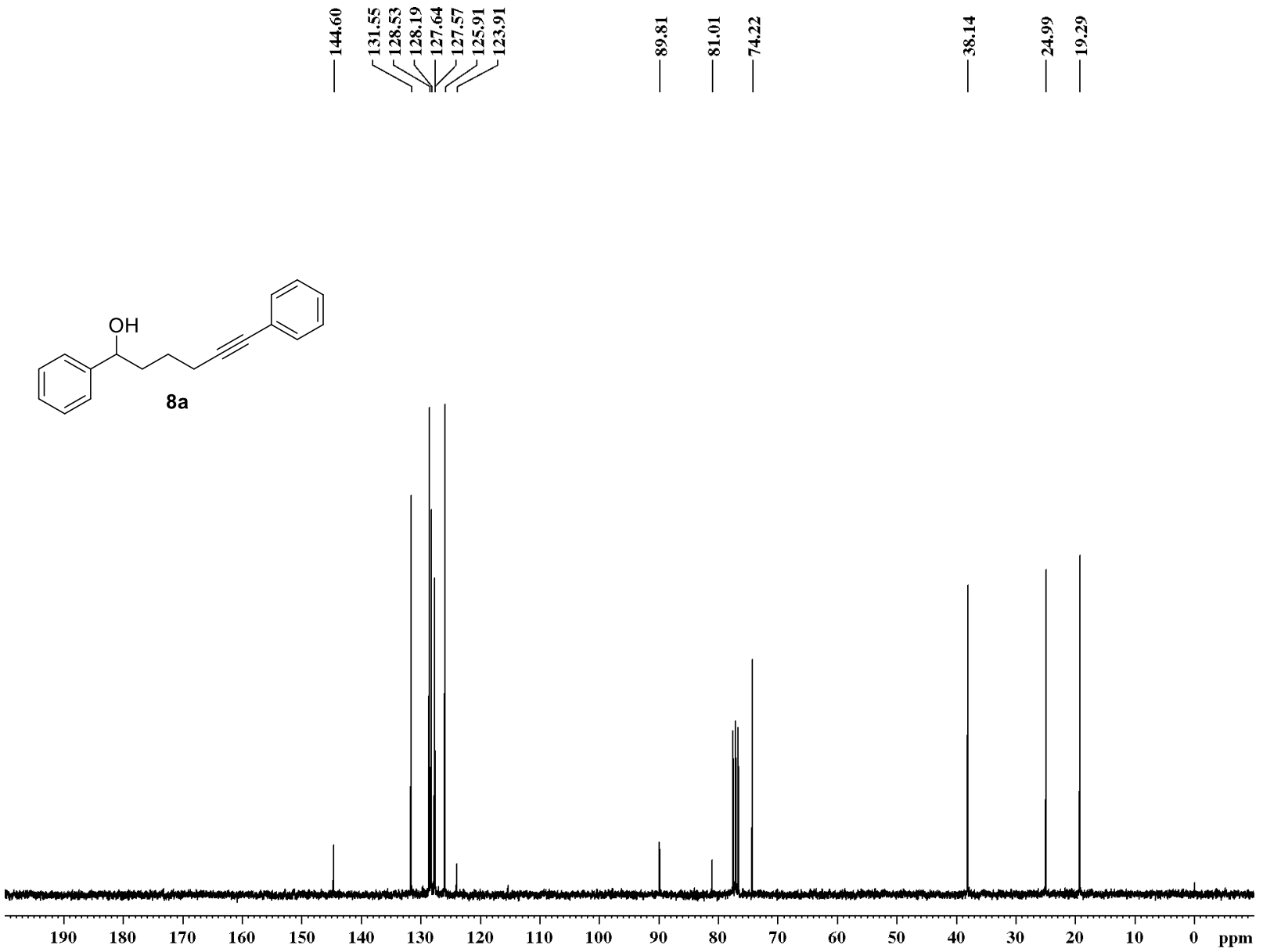
2.00
5.82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24.99
19.29
OH
8a
190
180
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140
130
120
110
100
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80
70
60
50
40
30
20
10
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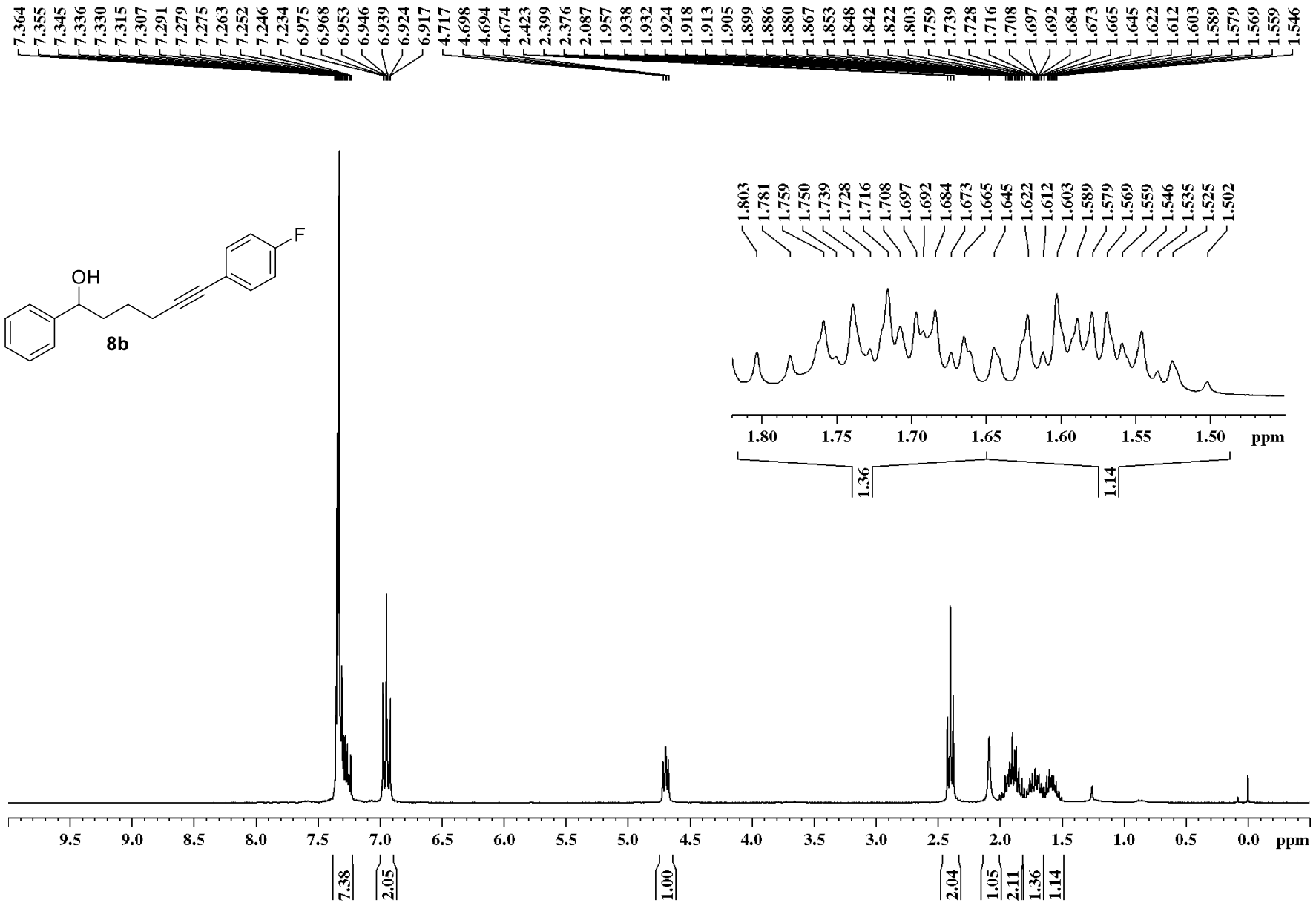
ppm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6.975
6.968
6.953
6.946
6.939
6.924
6.917
4.717
4.698
4.694
4.674
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2.399
2.376
2.087
1.957
1.938
1.932
1.924
1.918
1.913
1.905
1.899
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1.880
1.867
1.853
1.848
1.842
1.822
1.803
1.759
1.739
1.728
1.716
1.708
1.697
1.692
1.684
1.673
1.665
1.645
1.622
1.612
1.603
1.589
1.579
1.569
1.559
1.546
OH
F
8b
1.803
1.781
1.759
1.750
1.739
1.728
1.716
1.708
1.697
1.692
1.684
1.673
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1.645
1.622
1.612
1.603
1.589
1.579
1.569
1.559
1.546
1.535
1.525
1.502
1.80
1.75
1.70
1.65
1.60
1.55
1.50
ppm
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1.14
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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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2.04
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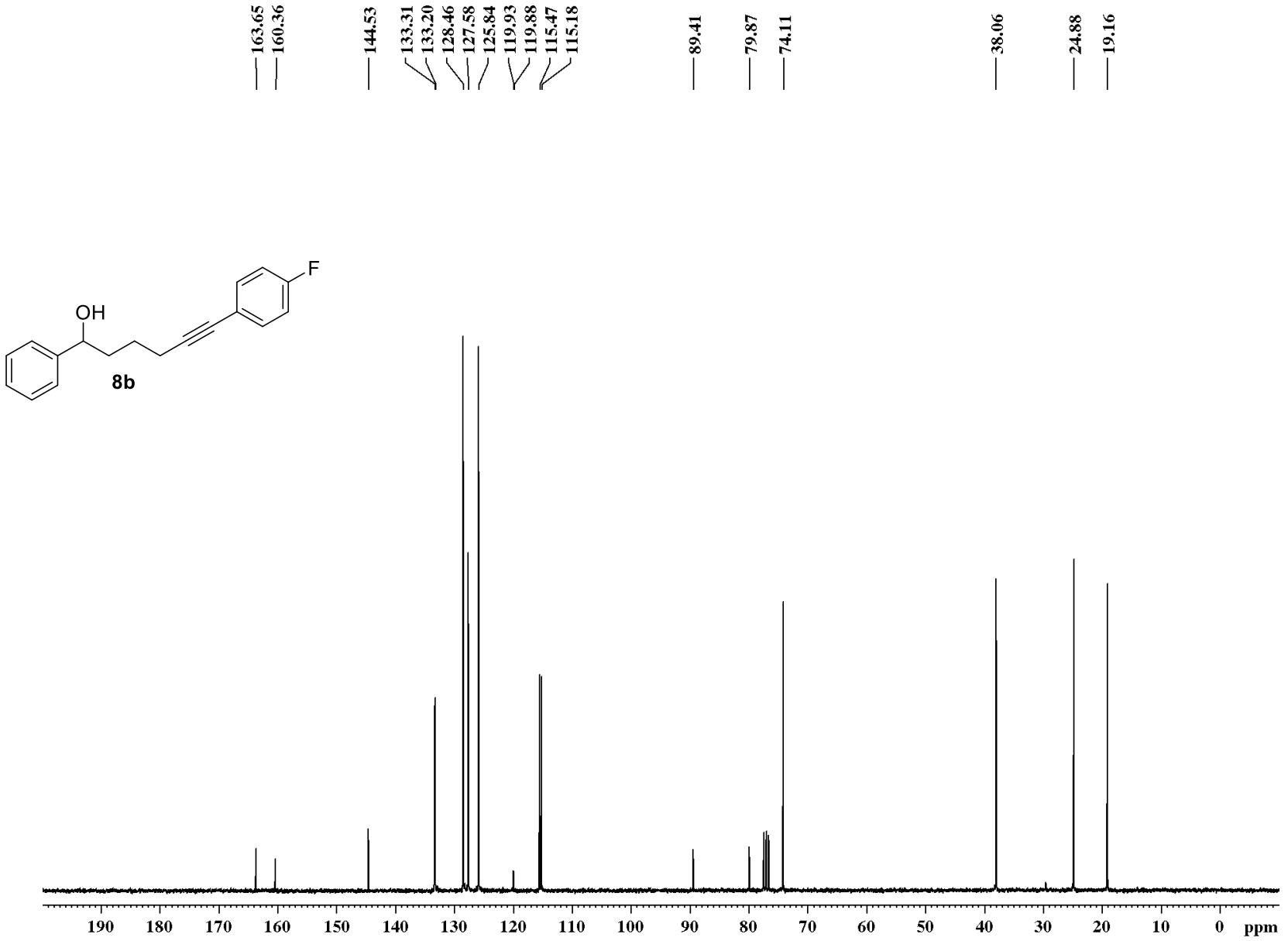
1.36
1.14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89.41
79.87
74.11
38.06
24.88
19.16
OH
F
8b
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140
130
120
110
100
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80
70
60
50
40
30
20
10
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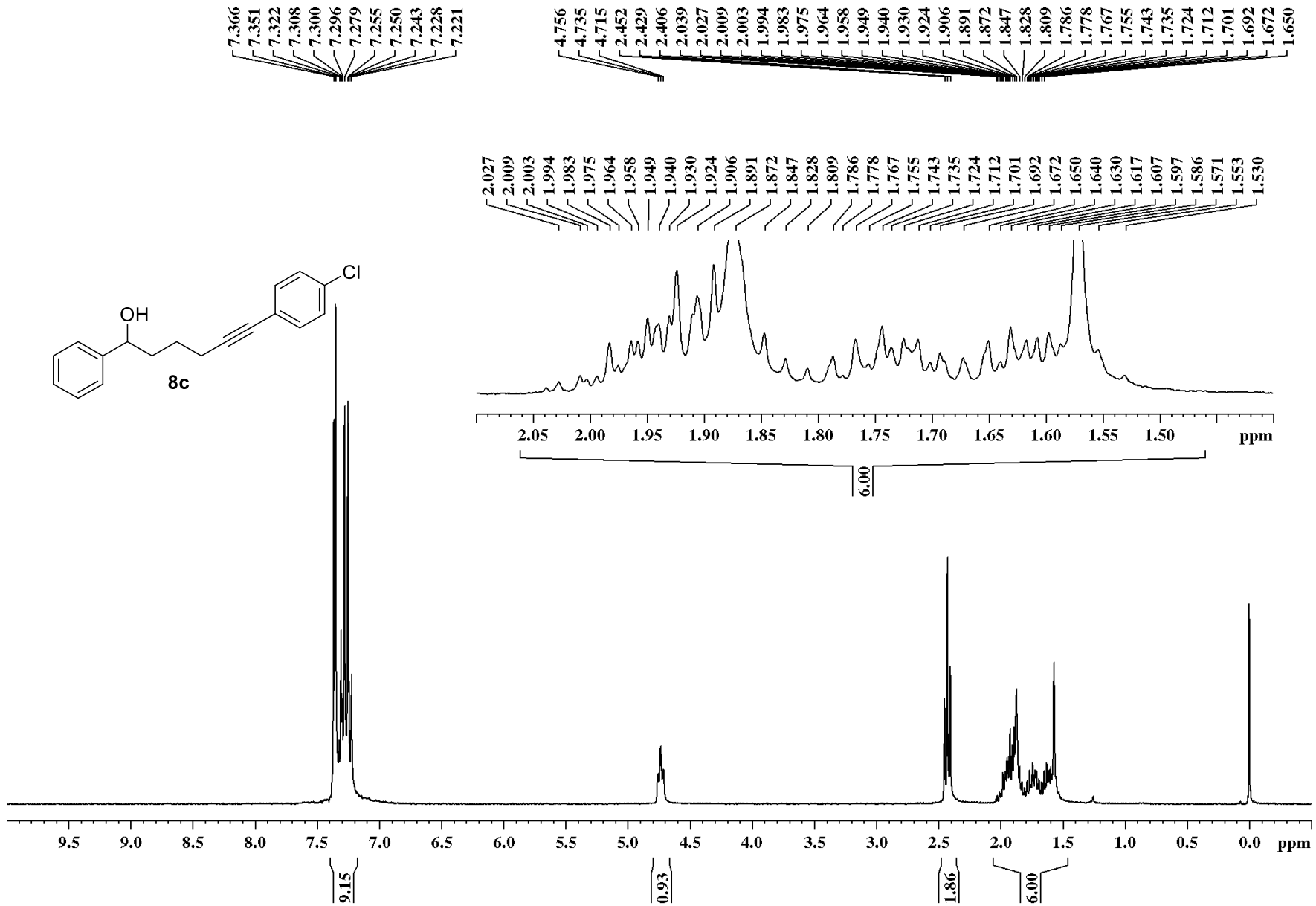
ppm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4.735
4.715
2.452
2.429
2.406
2.039
2.027
2.009
2.003
1.994
1.983
1.975
1.964
1.958
1.949
1.940
1.930
1.924
1.906
1.891
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1.828
1.809
1.786
1.778
1.767
1.755
1.743
1.735
1.724
1.712
1.701
1.692
1.672
1.650
Cl
OH
8c
2.05
2.00
1.95
1.90
1.85
1.80
1.75
1.70
1.65
1.60
1.55
1.50
ppm
6.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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1.0
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ppm
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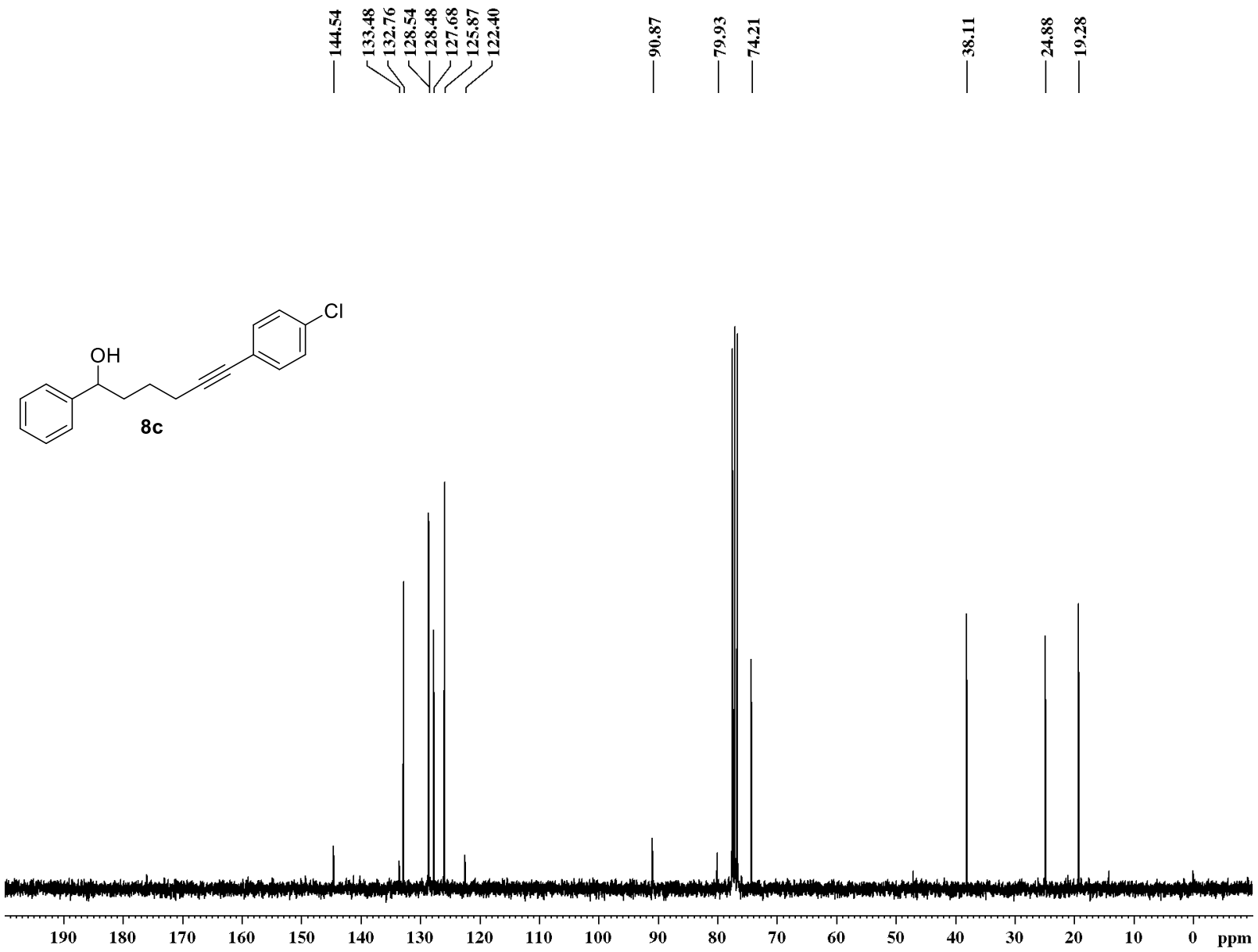
1.86
6.00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127.68
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122.40
90.87
79.93
74.21
38.11
24.88
19.28
Cl
OH
8c
190
180
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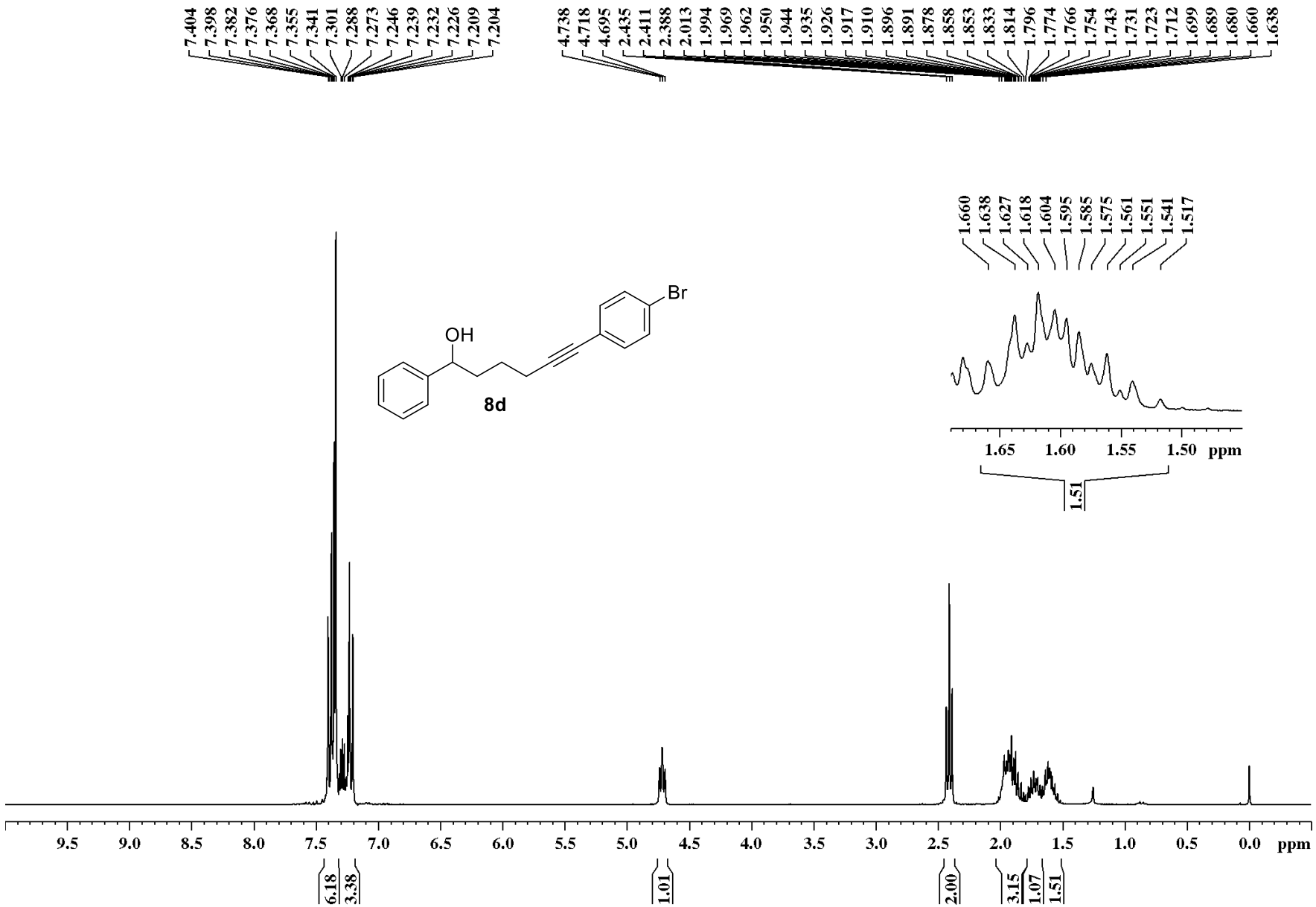
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.232
7.226
7.209
7.204
4.738
4.718
4.695
2.435
2.411
2.388
2.013
1.994
1.969
1.962
1.950
1.944
1.935
1.926
1.917
1.910
1.896
1.891
1.878
1.858
1.853
1.833
1.814
1.796
1.774
1.766
1.754
1.743
1.731
1.723
1.712
1.699
1.689
1.680
1.660
1.638
Br
OH
8d
1.660
1.638
1.627
1.618
1.604
1.595
1.585
1.575
1.561
1.551
1.541
1.517
1.65
1.60
1.55
1.50
ppm
1.51
9.5
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
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0.5
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ppm
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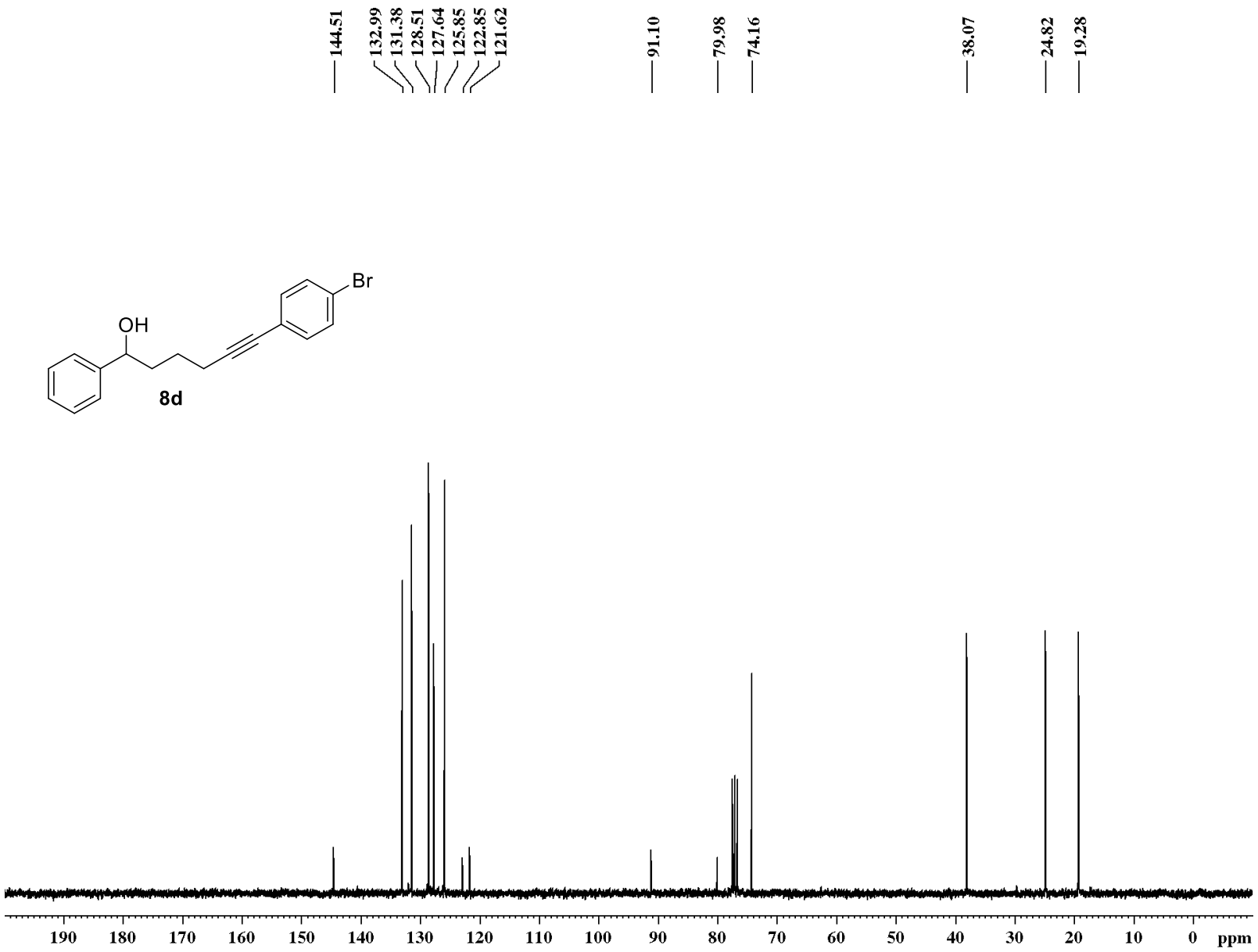
2.00
3.15
1.07
1.51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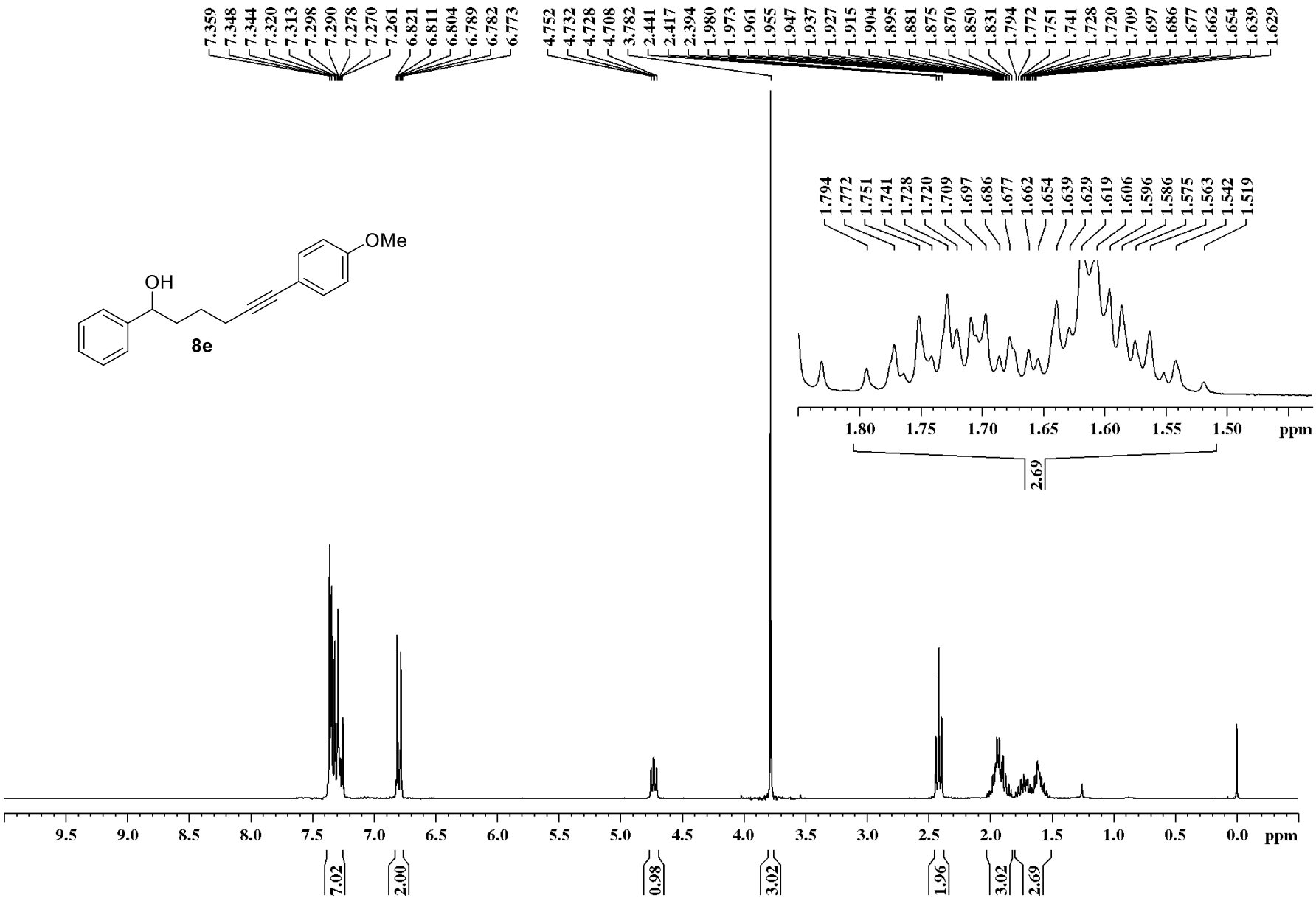
122.85
121.62
91.10
79.98
74.16
38.07
24.82
19.28
Br
OH
8d
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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6.782
6.773
4.752
4.732
4.728
4.708
3.782
2.441
2.417
2.394
1.980
1.973
1.961
1.955
1.947
1.937
1.927
1.915
1.904
1.895
1.881
1.875
1.870
1.850
1.831
1.794
1.772
1.751
1.741
1.728
1.720
1.709
1.697
1.686
1.677
1.662
1.654
1.639
1.629
1.619
1.606
1.596
1.586
1.575
1.563
1.542
1.519
OMe
OH
8e
1.80
1.75
1.70
1.65
1.60
1.55
1.50
ppm
2.69
9.5
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
0.0
ppm
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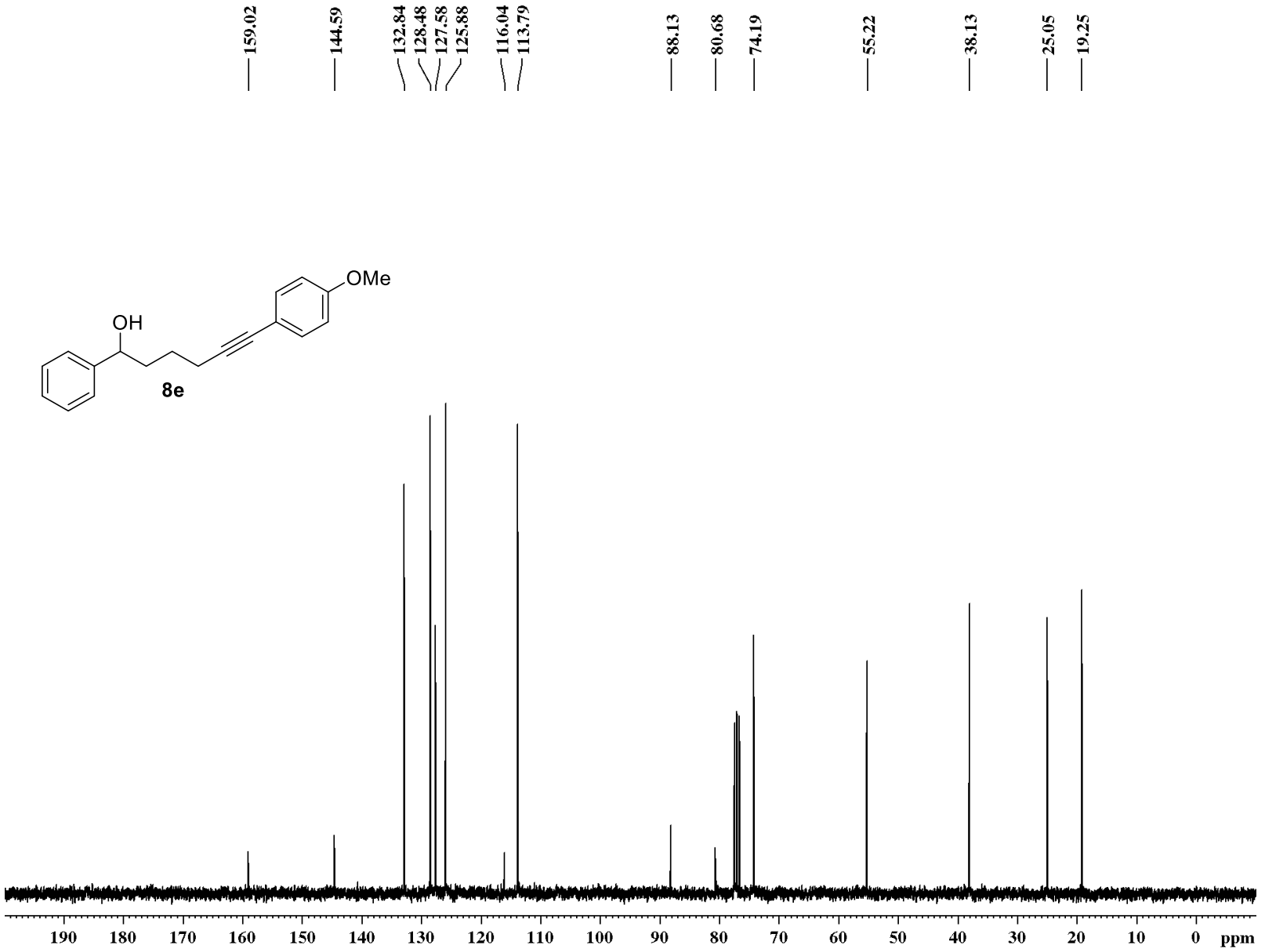
0.98
3.02
1.96
3.02
2.69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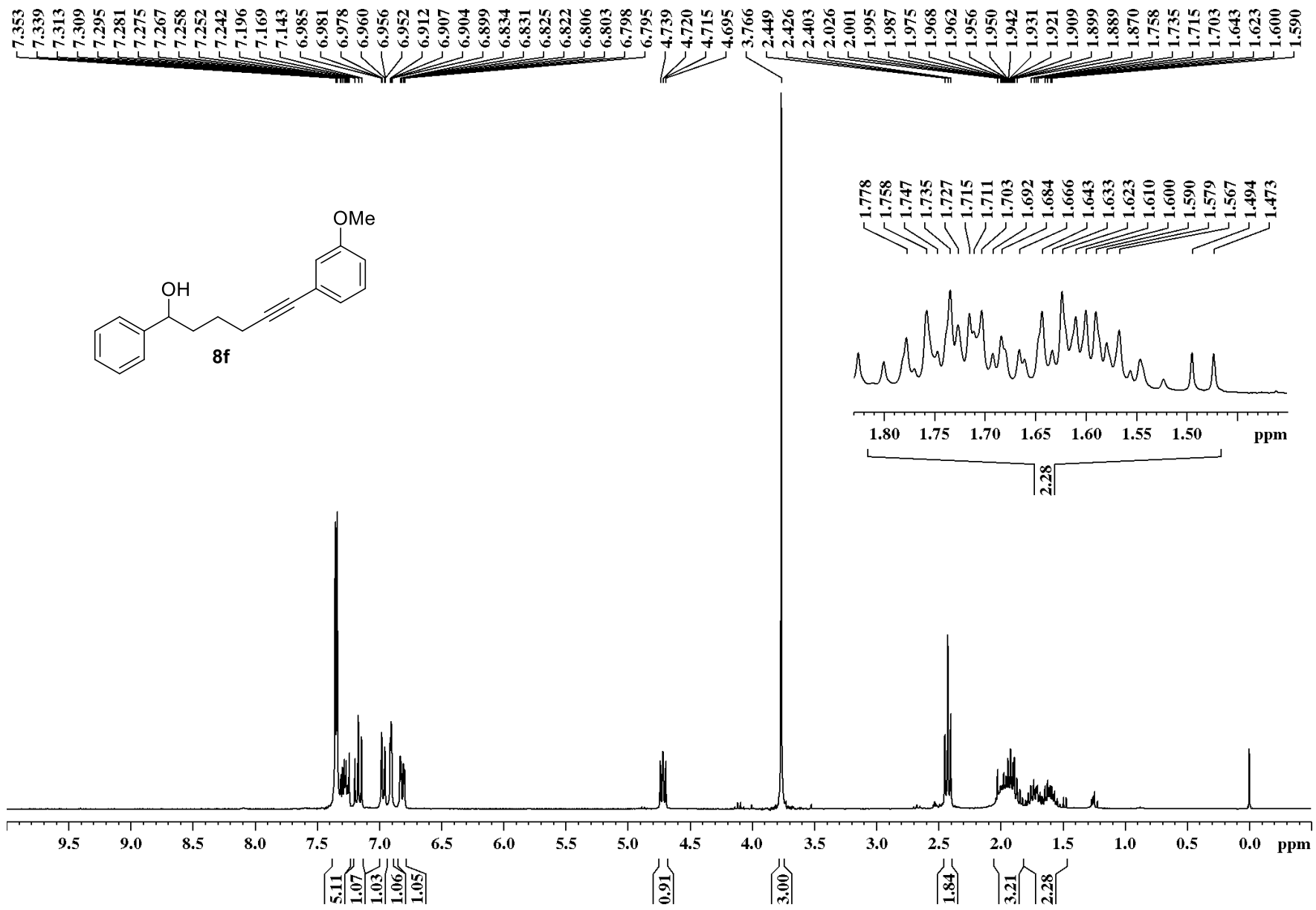
113.79
88.13
80.68
74.19
55.22
38.13
25.05
19.25
OMe
OH
8e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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OMe
OH
8f
1.778 1.758 1.747 1.735 1.727 1.715 1.711 1.703 1.692 1.684 1.666 1.643 1.633 1.623 1.610 1.600 1.590 1.579 1.567 1.494 1.473
1.80 1.75 1.70 1.65 1.60 1.55 1.50 ppm
2.28
9.5 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 0.0 ppm
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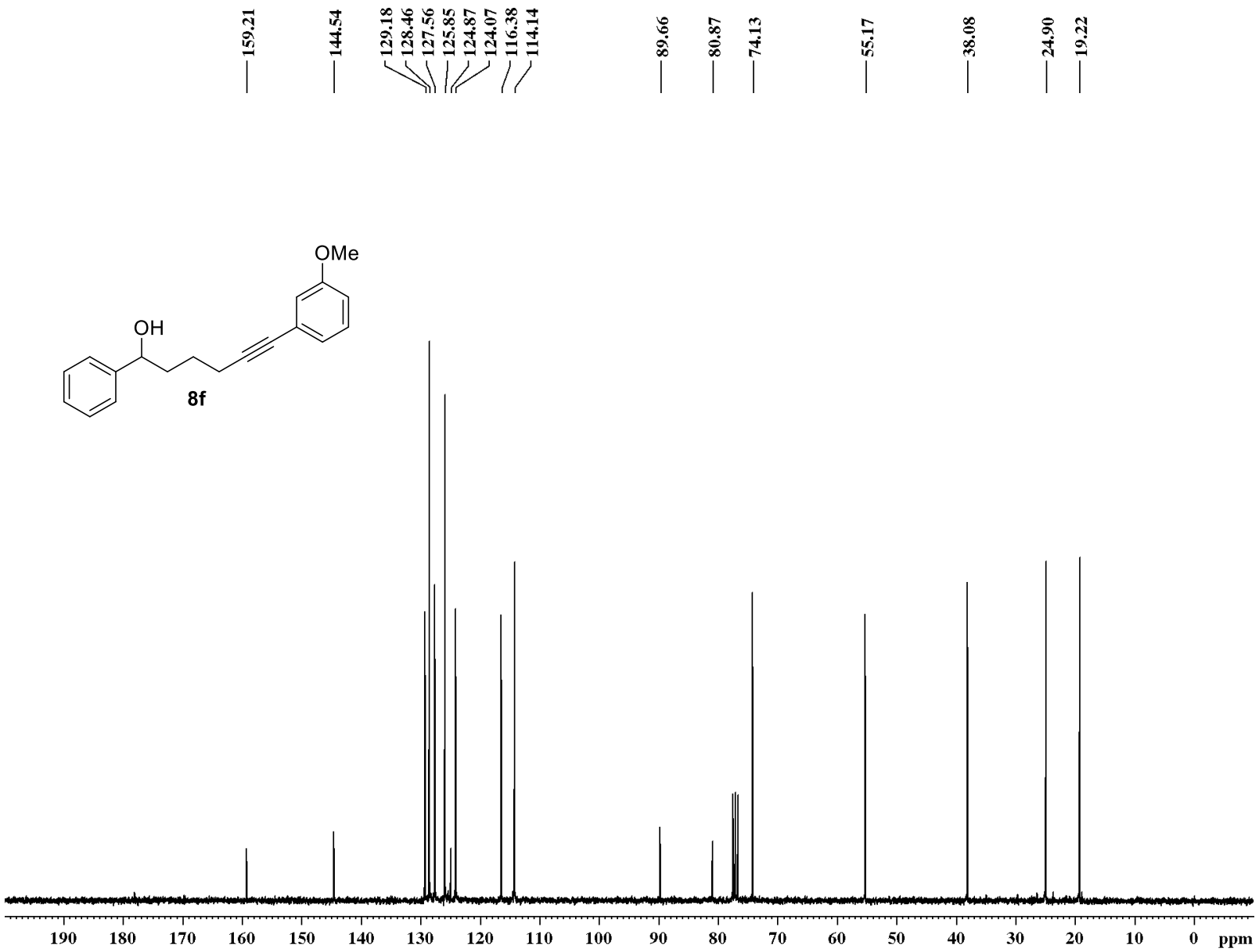
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124.07
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114.14
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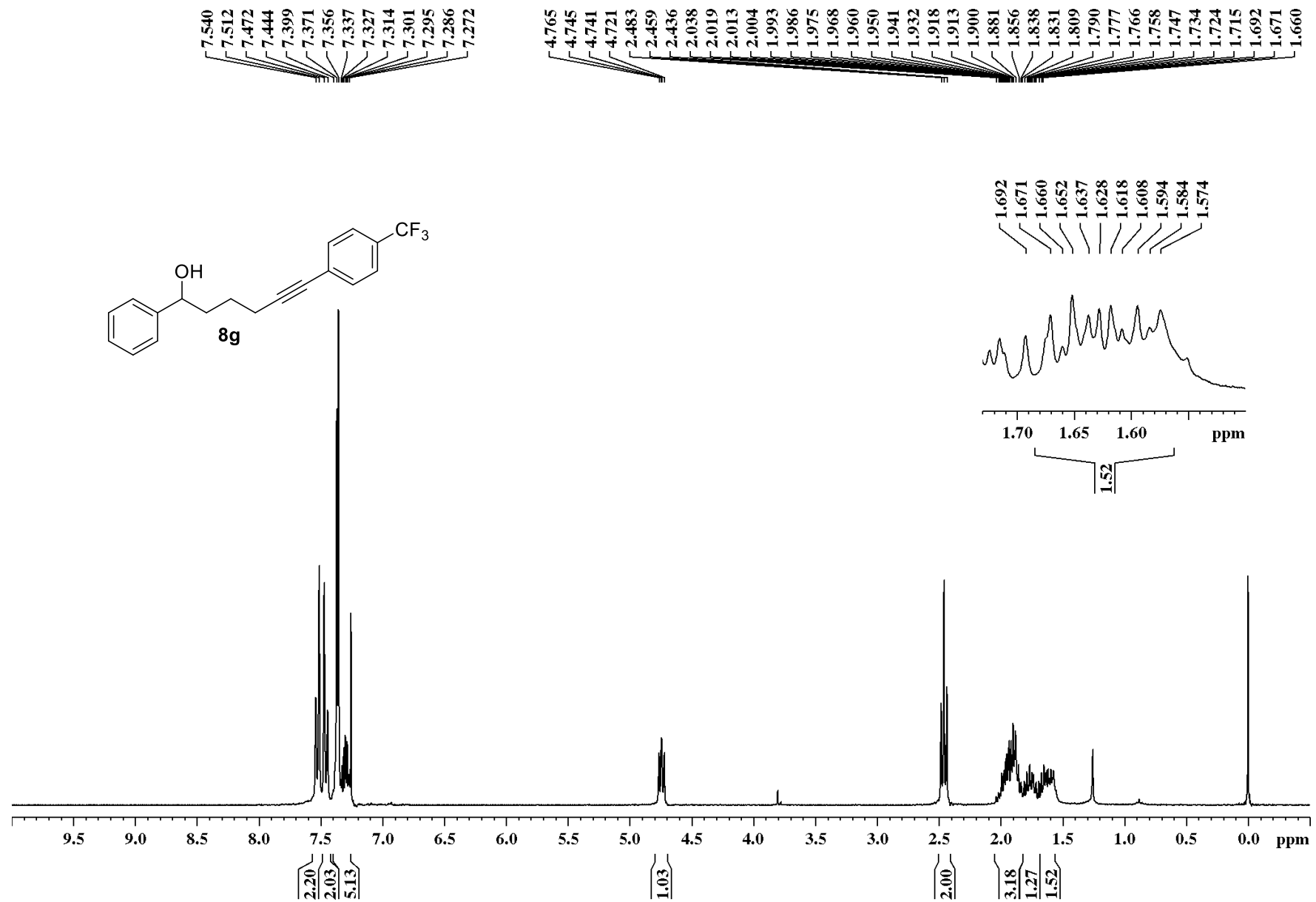
80.87
74.13
55.17
38.08
24.90
19.22
OMe
OH
8f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.272
4.765
4.745
4.741
4.721
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2.436
2.038
2.019
2.013
2.004
1.993
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1.975
1.968
1.960
1.950
1.941
1.932
1.918
1.913
1.900
1.881
1.856
1.838
1.831
1.809
1.790
1.777
1.766
1.758
1.747
1.734
1.724
1.715
1.692
1.671
1.660
1.692
1.671
1.660
1.652
1.637
1.628
1.618
1.608
1.594
1.584
1.574
OH
CF3
8g
1.70
1.65
1.60
ppm
1.52
9.5
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
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ppm
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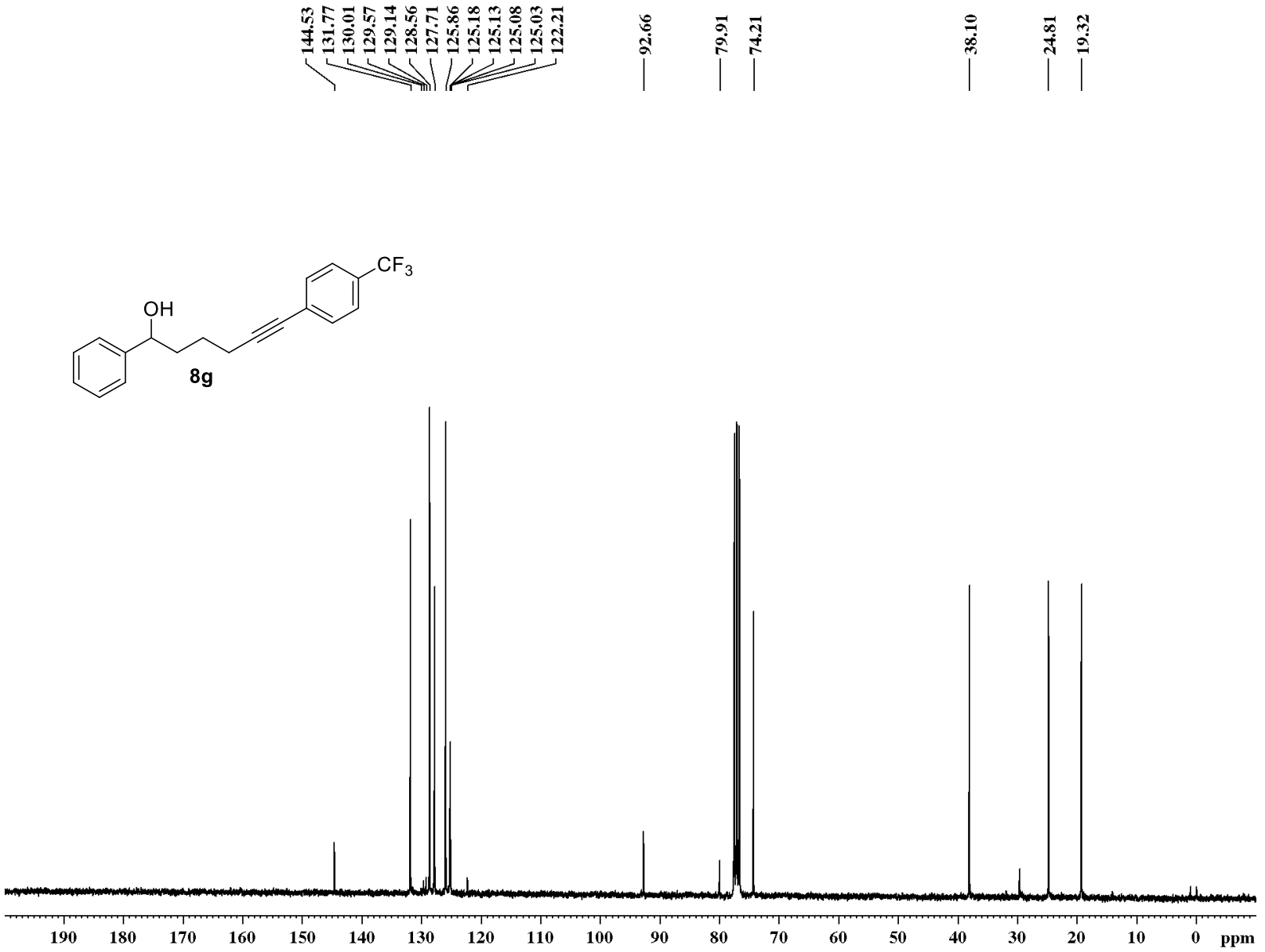
1.52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125.13
125.08
125.03
122.21
92.66
79.91
74.21
38.10
24.81
19.32
CF3
OH
8g
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180
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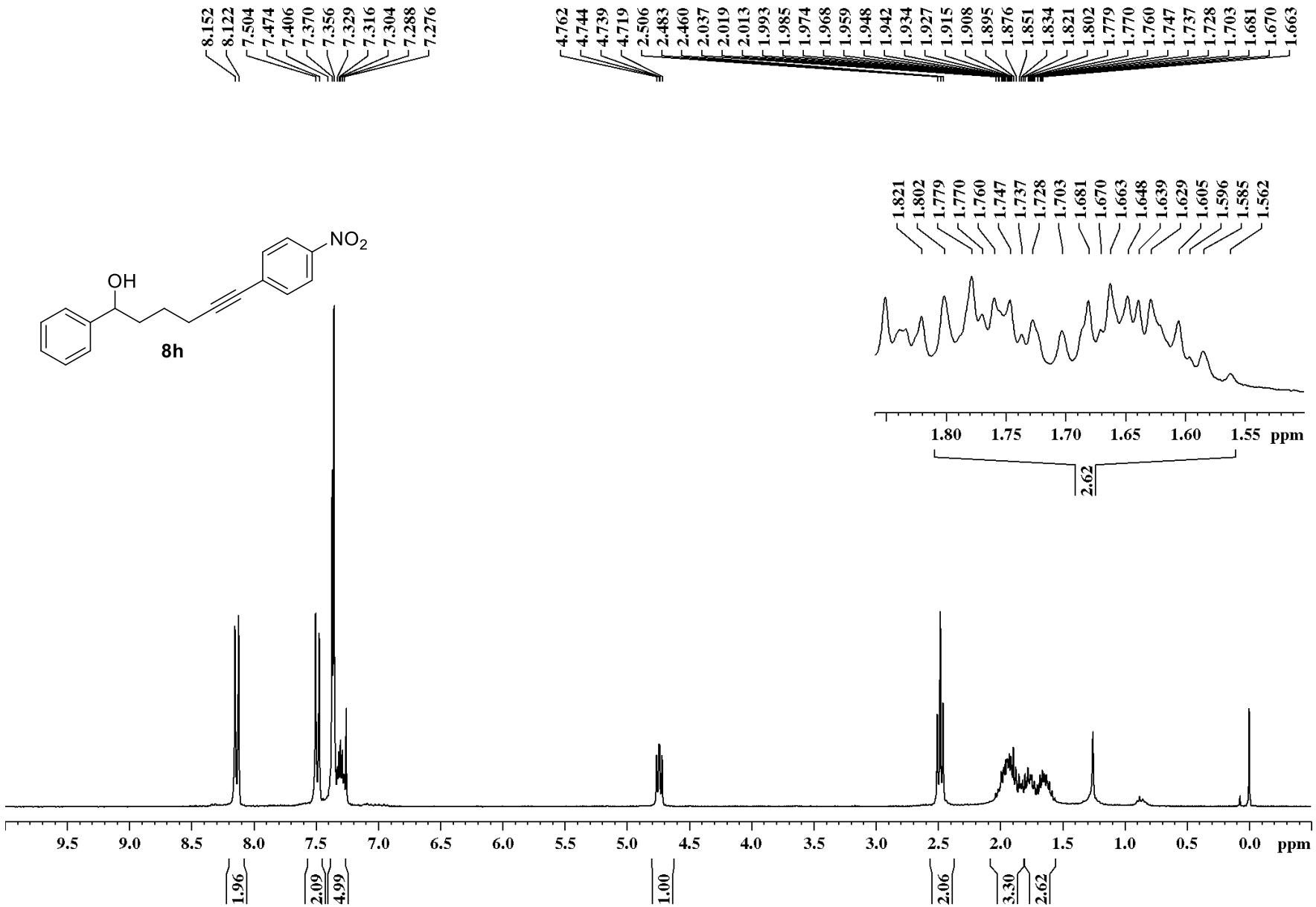
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.276
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4.744
4.739
4.719
2.506
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1.968
1.959
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1.942
1.934
1.927
1.915
1.908
1.895
1.876
1.851
1.834
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1.802
1.779
1.770
1.760
1.747
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1.728
1.703
1.681
1.670
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1.648
1.639
1.629
1.605
1.596
1.585
1.562
NO2
OH
8h
1.80
1.75
1.70
1.65
1.60
1.55 ppm
2.62
9.5
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
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4.99
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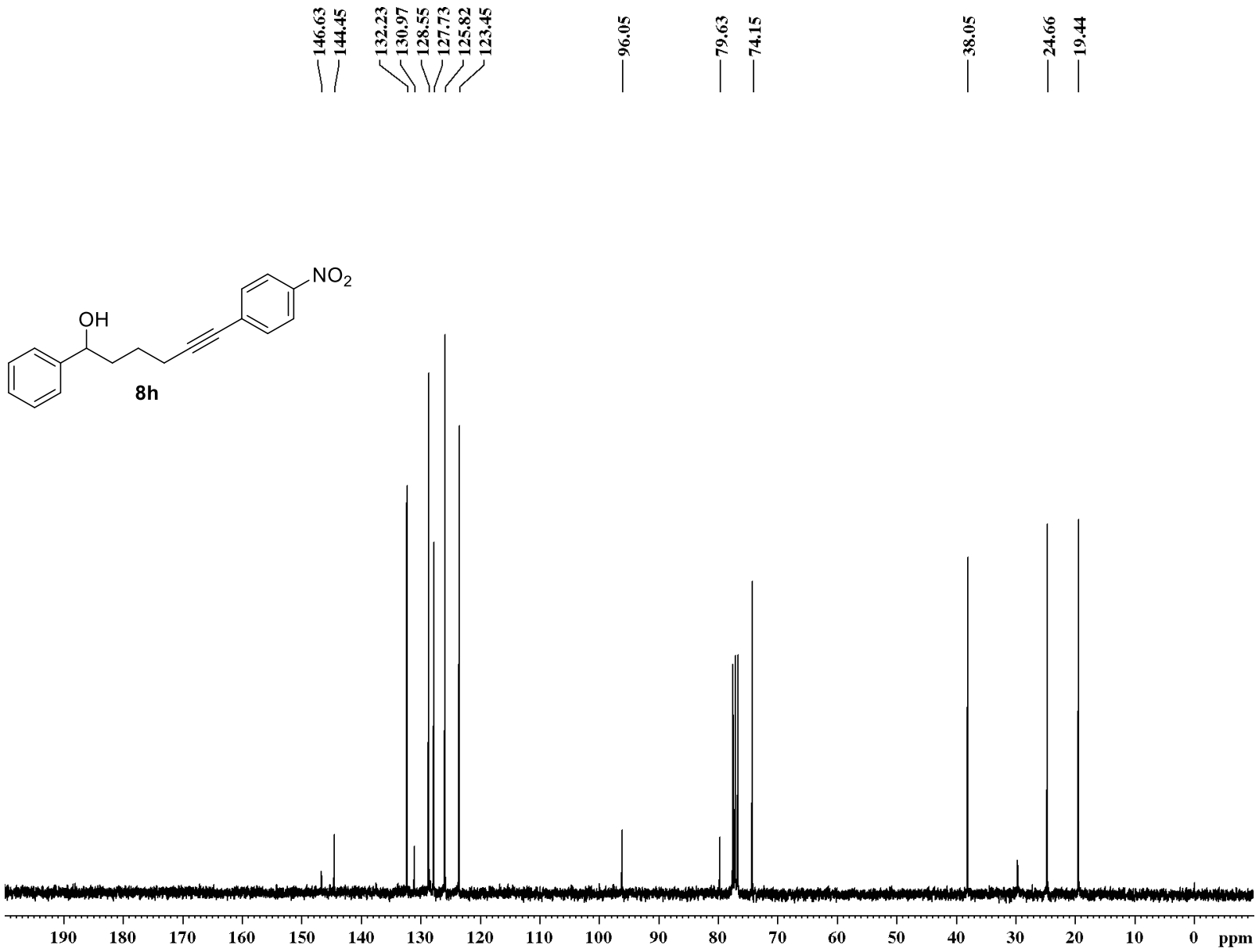
2.62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74.15
38.05
24.66
19.44
NO2
OH
8h
190
180
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140
130
120
110
100
90
80
70
60
50
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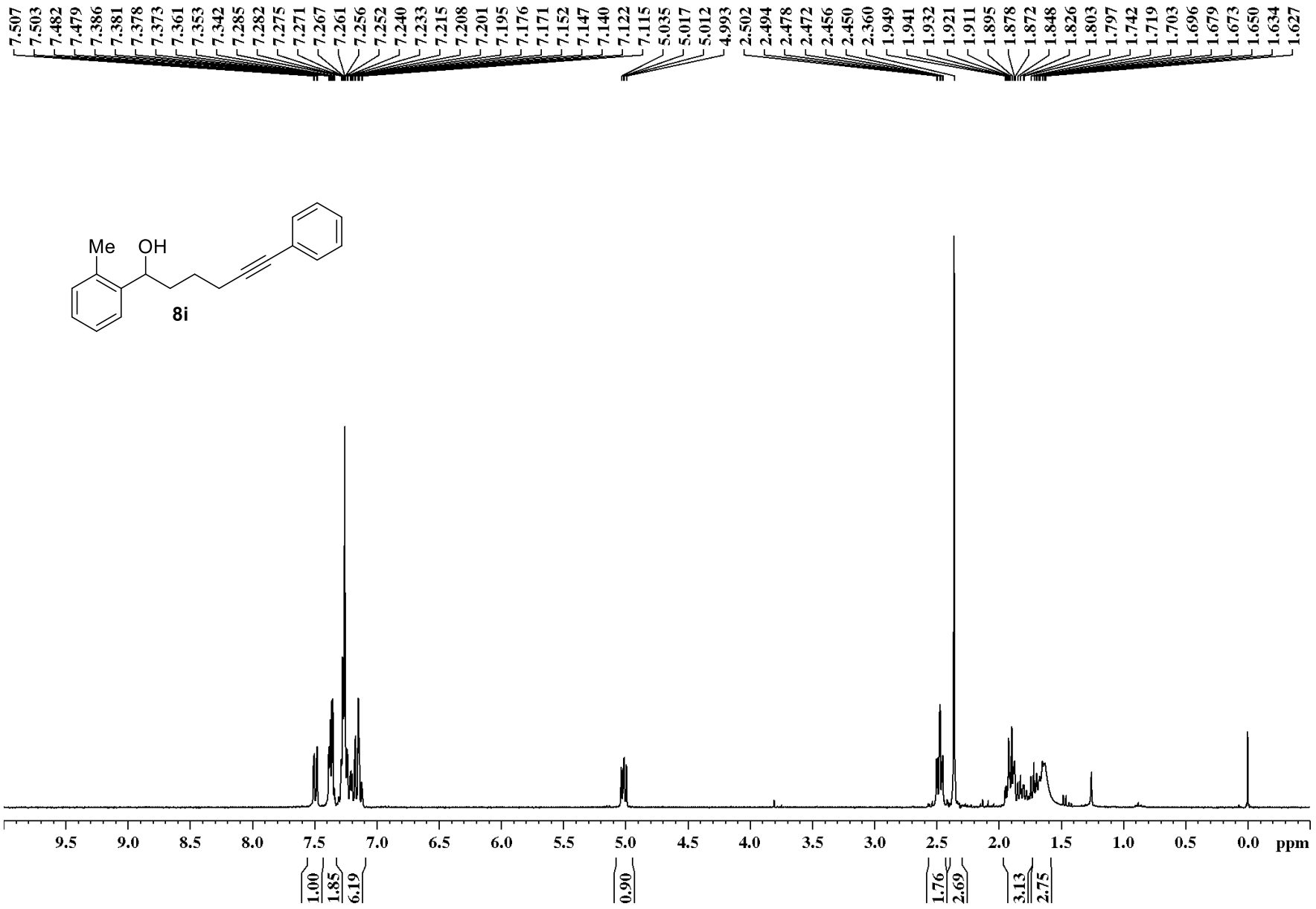
30
20
10
0
ppm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7.275
7.271
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7.256
7.252
7.240
7.233
7.215
7.208
7.201
7.195
7.176
7.171
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7.140
7.122
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5.035
5.017
5.012
4.993
2.502
2.494
2.478
2.472
2.456
2.450
2.360
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1.941
1.932
1.921
1.911
1.895
1.878
1.872
1.848
1.826
1.803
1.797
1.742
1.719
1.703
1.696
1.679
1.673
1.650
1.634
1.627
Me
OH
8i
9.5
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
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ppm
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0.90
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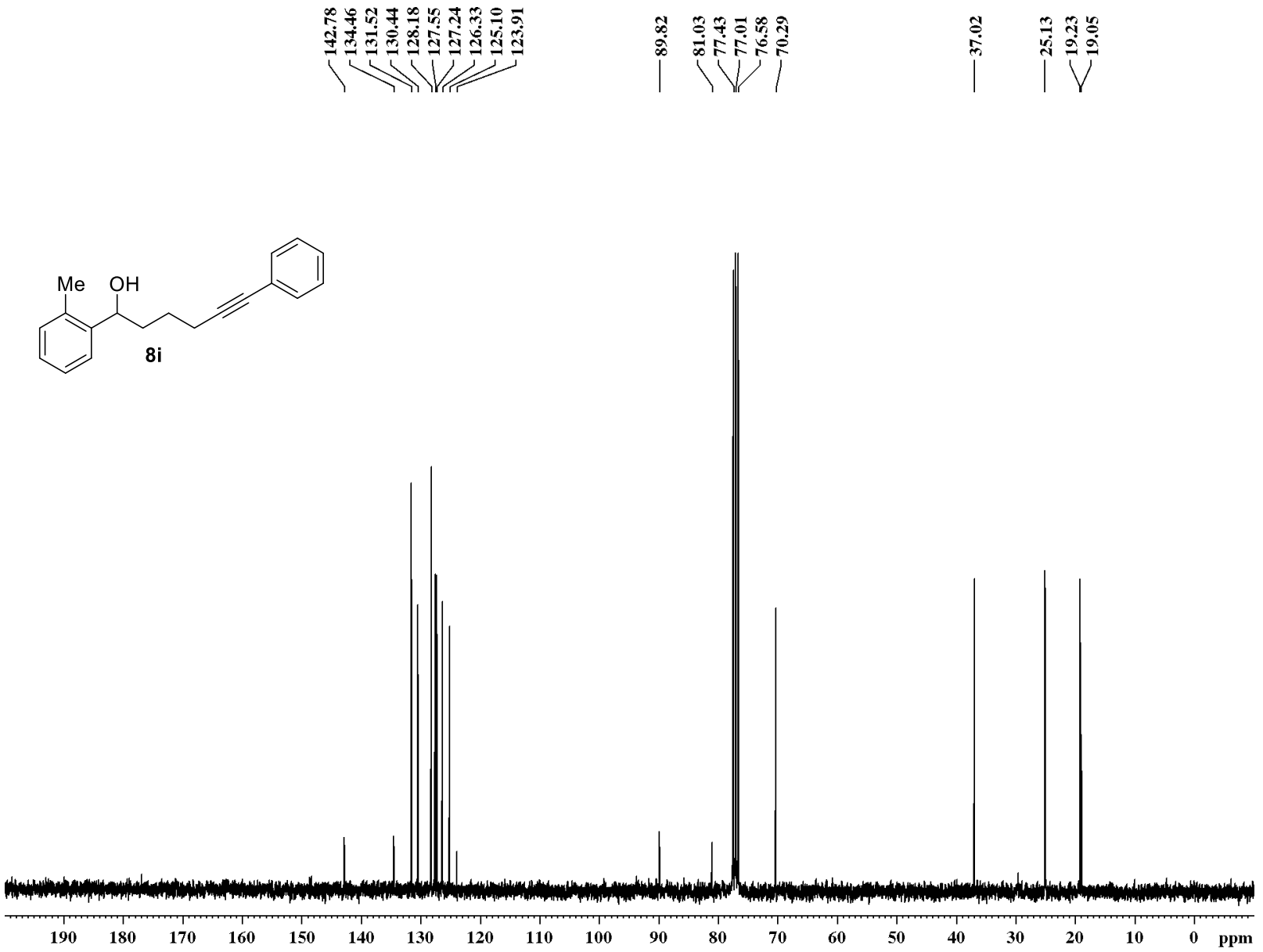
2.75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77.01
76.58
70.29
37.02
25.13
19.23
19.05
Me
OH
8i
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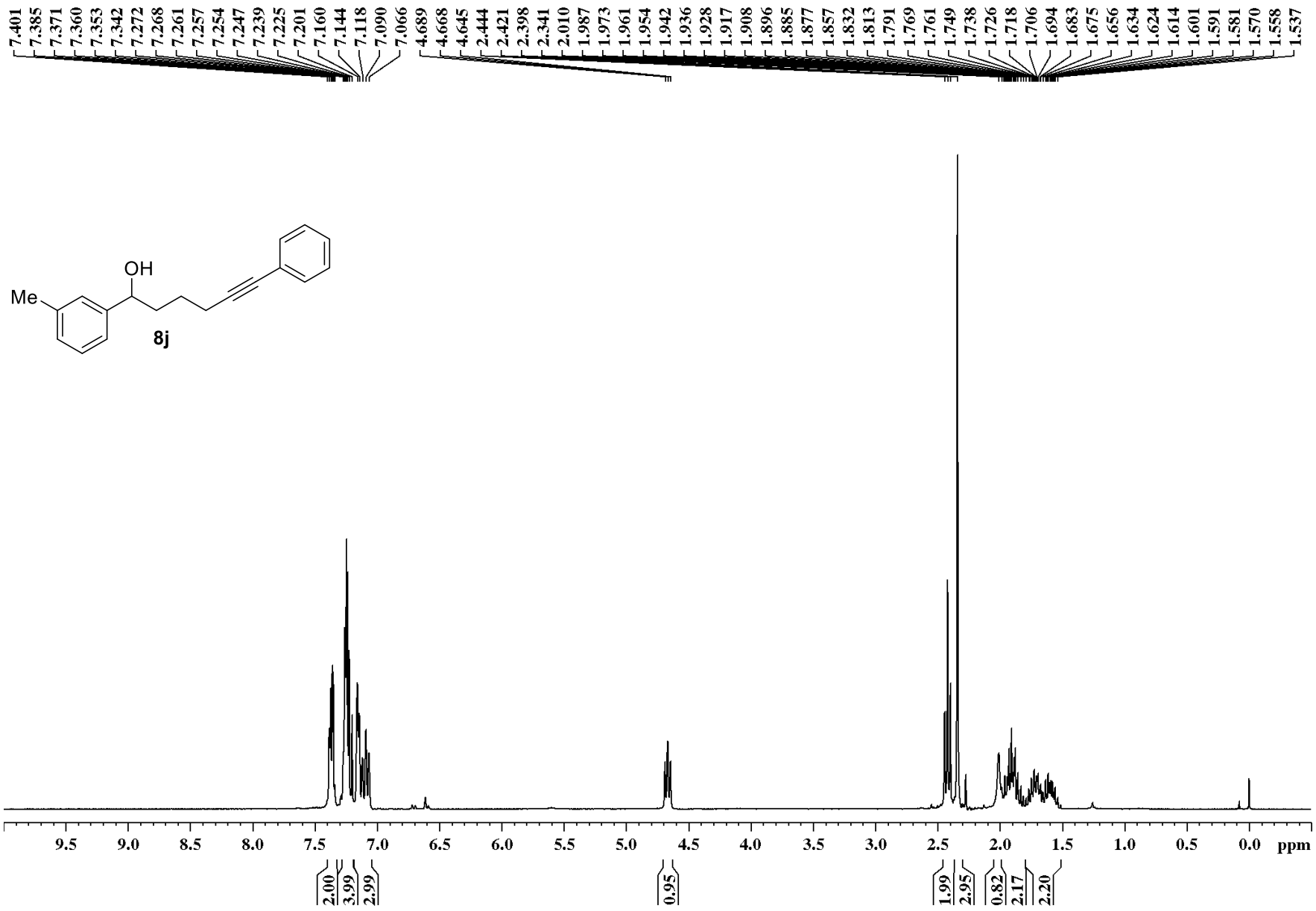
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7.225
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4.689
4.668
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2.421
2.398
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1.942
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1.928
1.917
1.908
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1.857
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1.694
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1.614
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1.591
1.581
1.570
1.558
1.537
OH
Me
8j
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8.5
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6.0
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1.5
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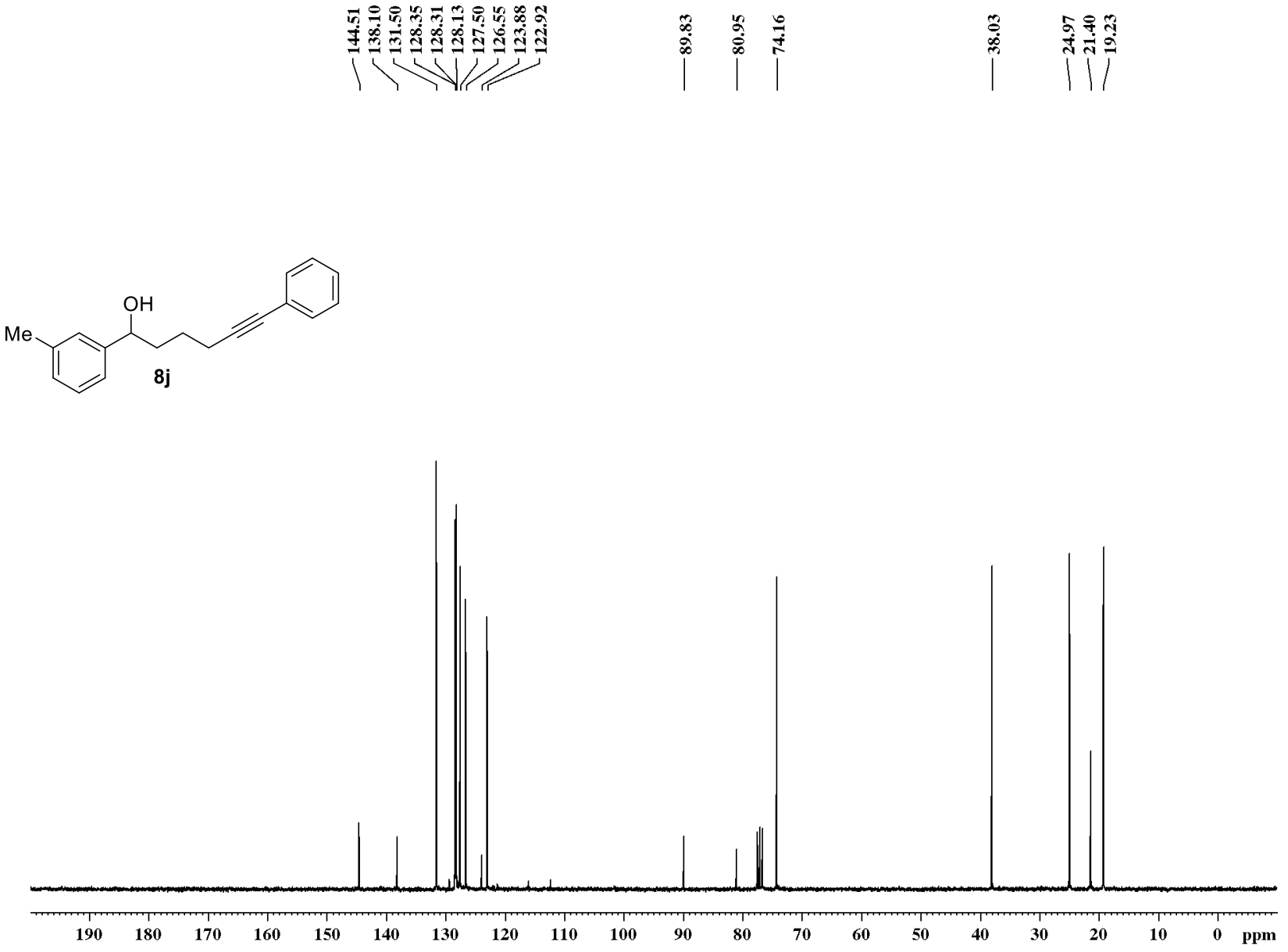
2.20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38.03
24.97
21.40
19.23
OH
Me
8j
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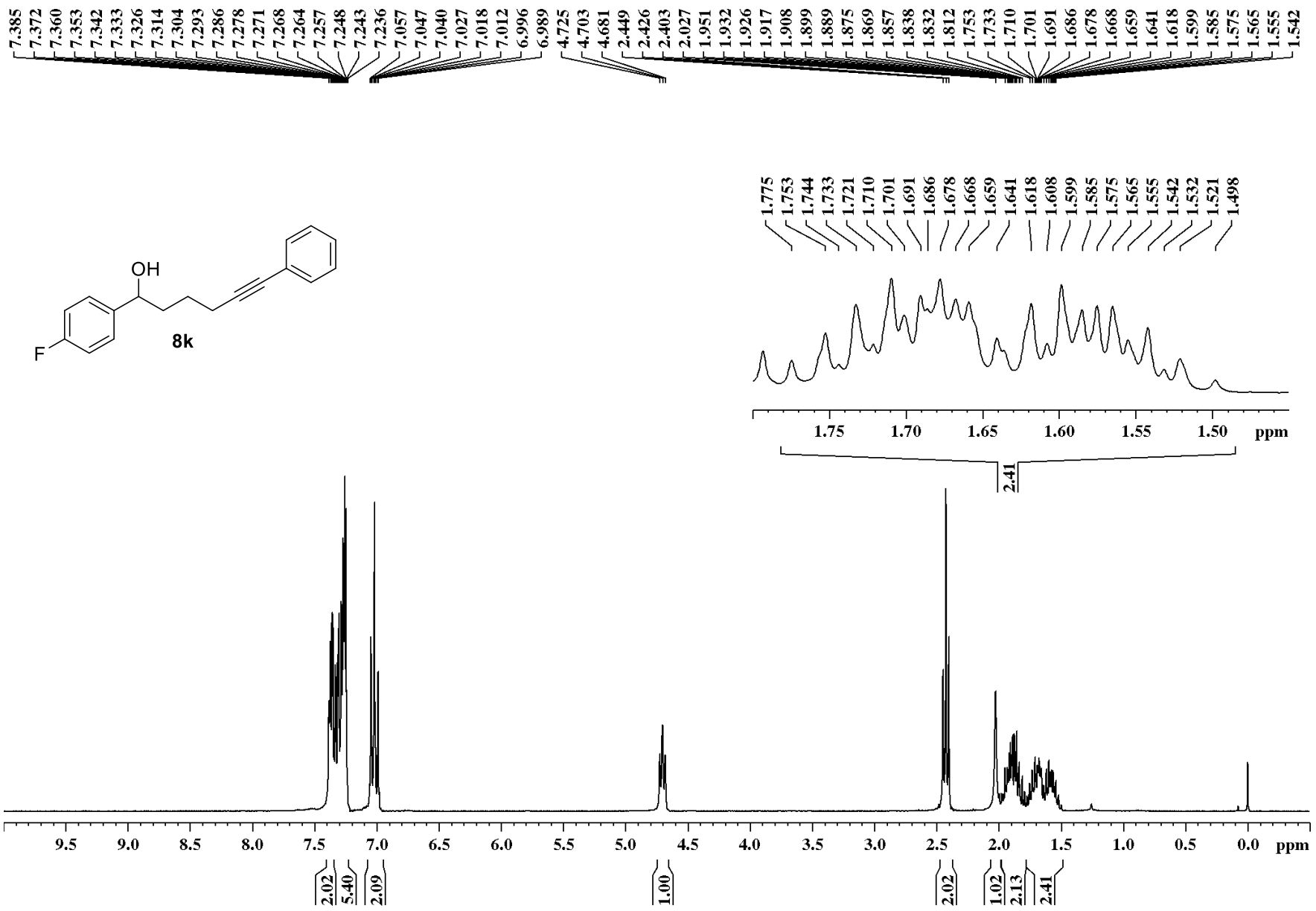
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4.725 4.703 4.681 2.449 2.426 2.403 2.027 1.951 1.932 1.926 1.917 1.908 1.899 1.889 1.875 1.869 1.857 1.838 1.832 1.812 1.753 1.733 1.710 1.701 1.691 1.686 1.678 1.668 1.659 1.641 1.618 1.599 1.585 1.542
OH
F
8k
1.775 1.753 1.744 1.733 1.721 1.710 1.701 1.691 1.686 1.678 1.668 1.659 1.641 1.618 1.608 1.599 1.585 1.575 1.565 1.555 1.542 1.532 1.521 1.498
1.75 1.70 1.65 1.60 1.55 1.50 ppm
2.41
9.5 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 0.0 ppm
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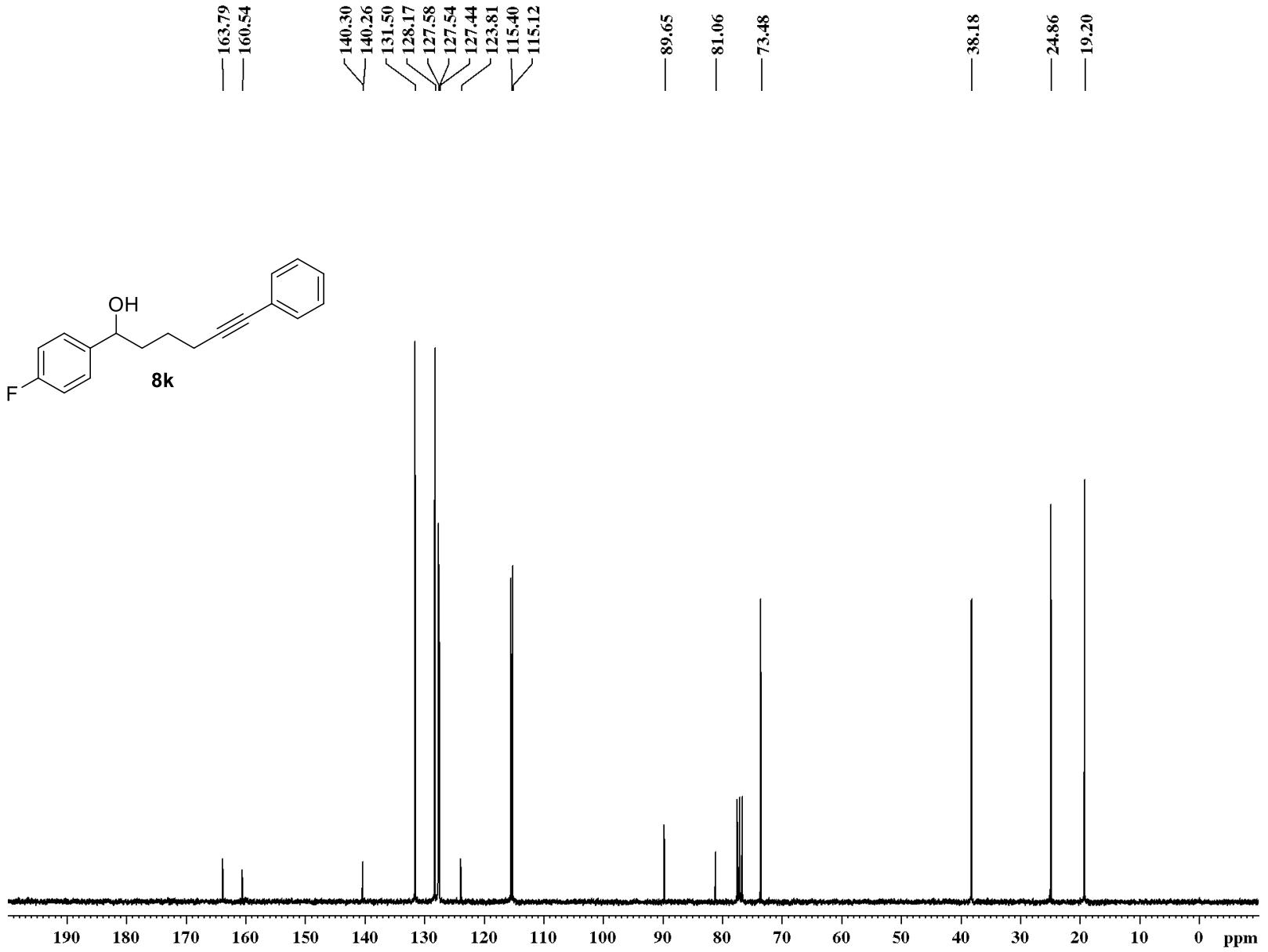
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81.06
73.48
38.18
24.86
19.20
OH
F
8k
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140
130
120
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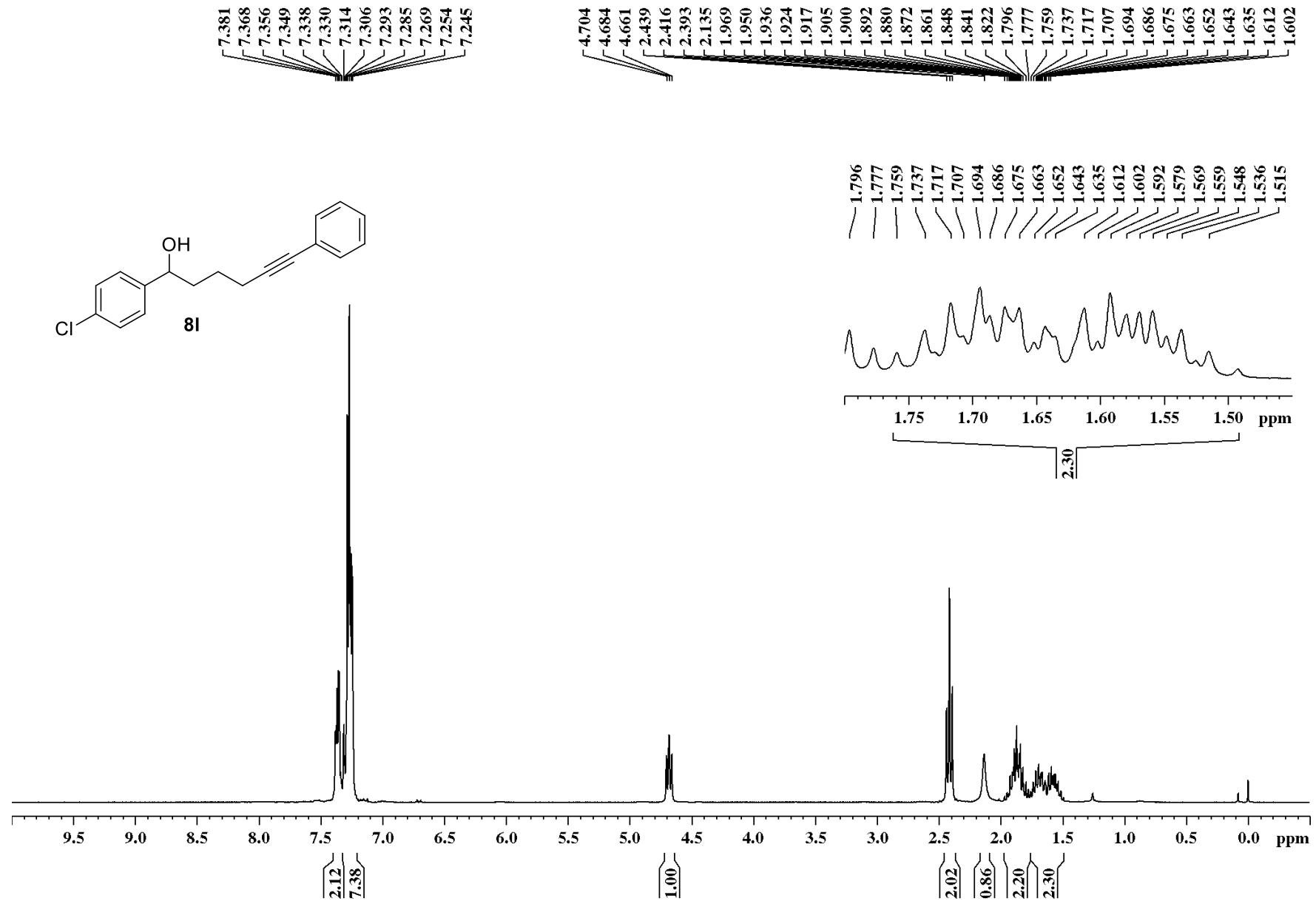
60
50
40
30
20
10
0
ppm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1.917
1.905
1.900
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1.872
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1.841
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1.796
1.777
1.759
1.737
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1.707
1.694
1.686
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1.663
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1.643
1.635
1.612
1.602
1.592
1.579
1.569
1.559
1.548
1.536
1.515
OH
Cl
8l
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1.70
1.65
1.60
1.55
1.50
ppm
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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
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0.0
ppm
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1.00
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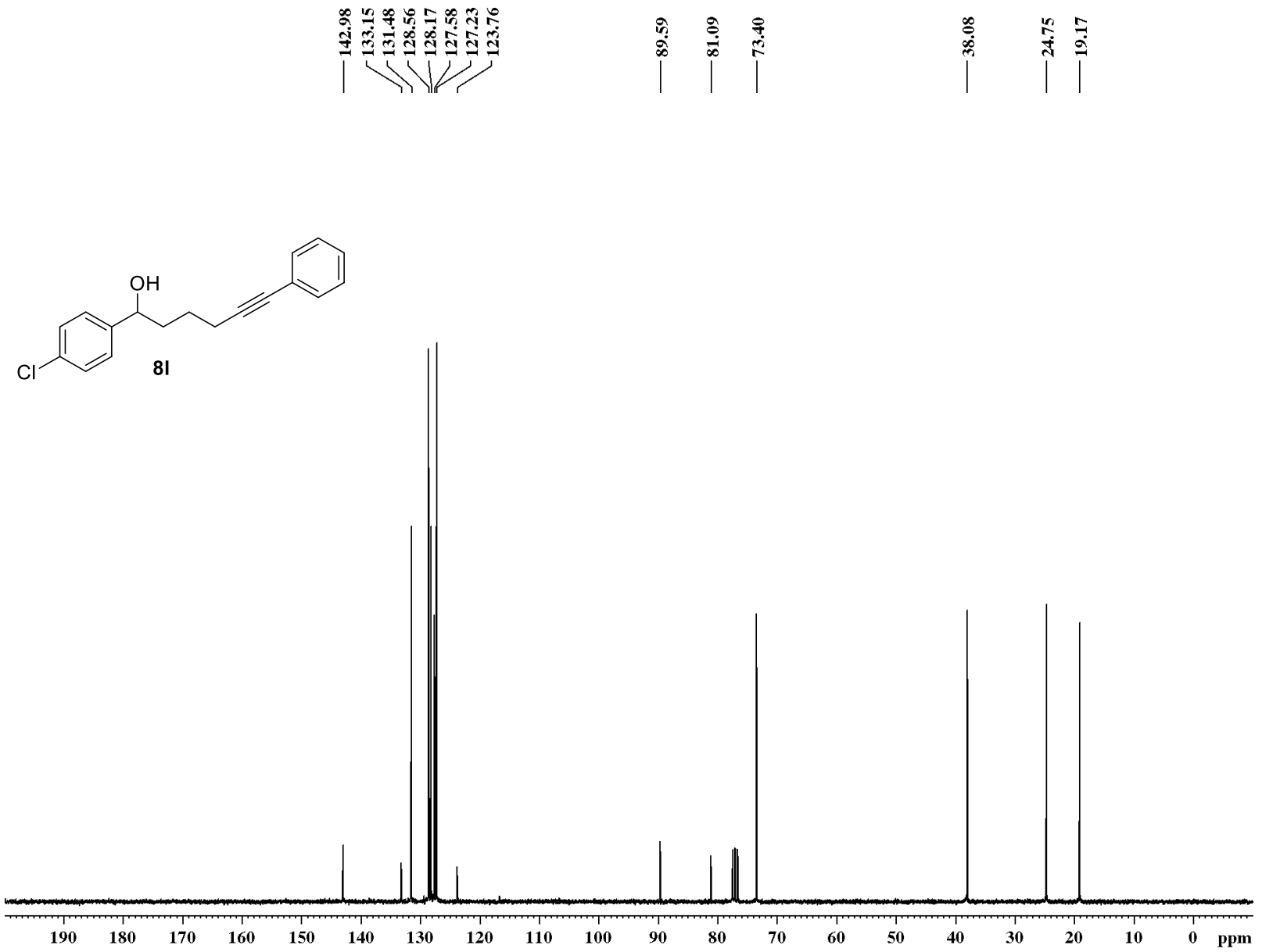
2.30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73.40
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24.75
19.17
OH
Cl
8l
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180
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140
130
120
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100
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60
50
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30
20
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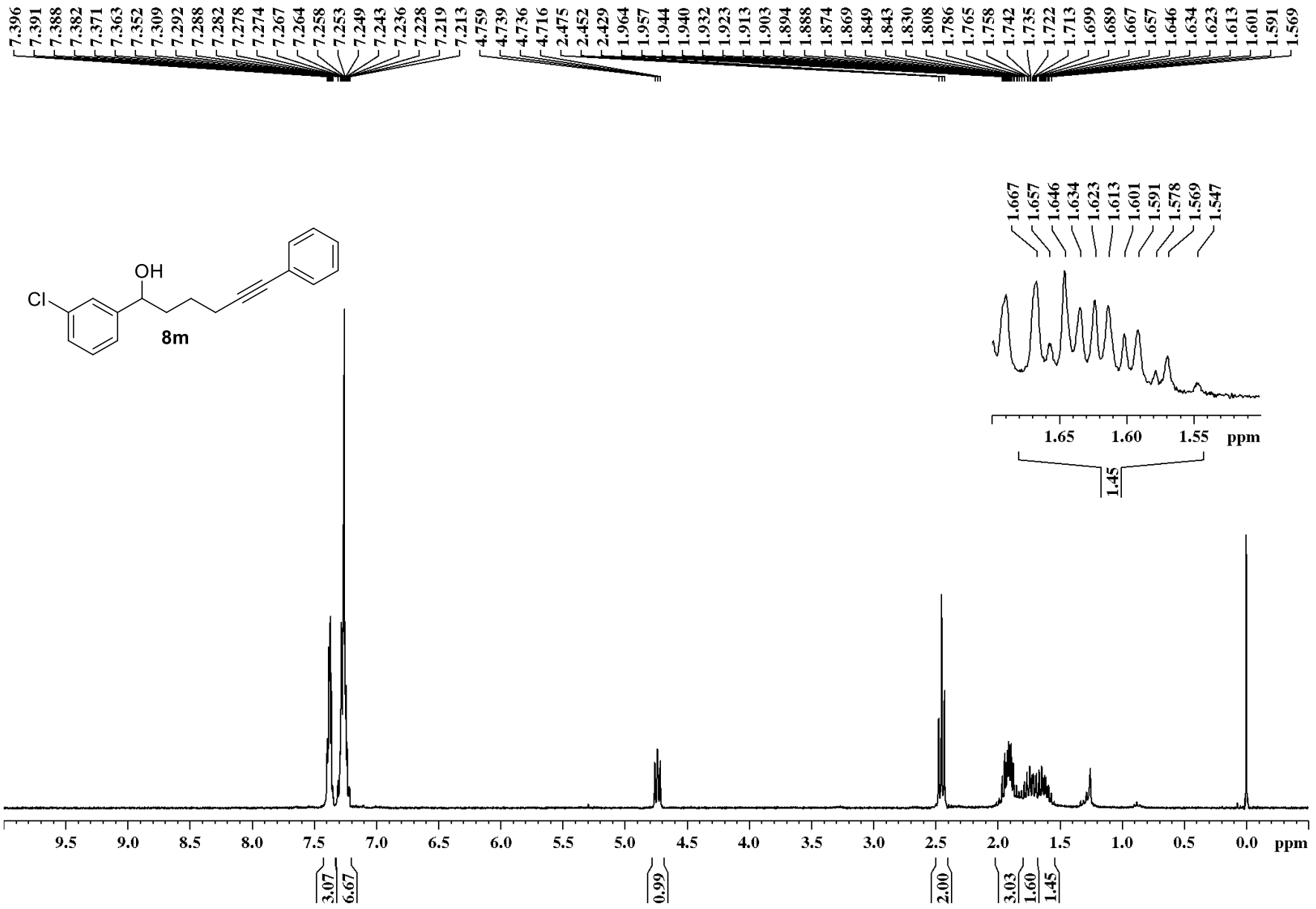
0
ppm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7.253
7.249
7.243
7.236
7.228
7.219
7.213
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4.739
4.736
4.716
2.475
2.452
2.429
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1.957
1.944
1.940
1.932
1.923
1.913
1.903
1.894
1.888
1.874
1.869
1.849
1.843
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1.808
1.786
1.765
1.758
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1.735
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1.591
1.578
1.569
1.547
OH
Cl
8m
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1.60
1.55
ppm
1.45
9.5
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
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0.5
0.0
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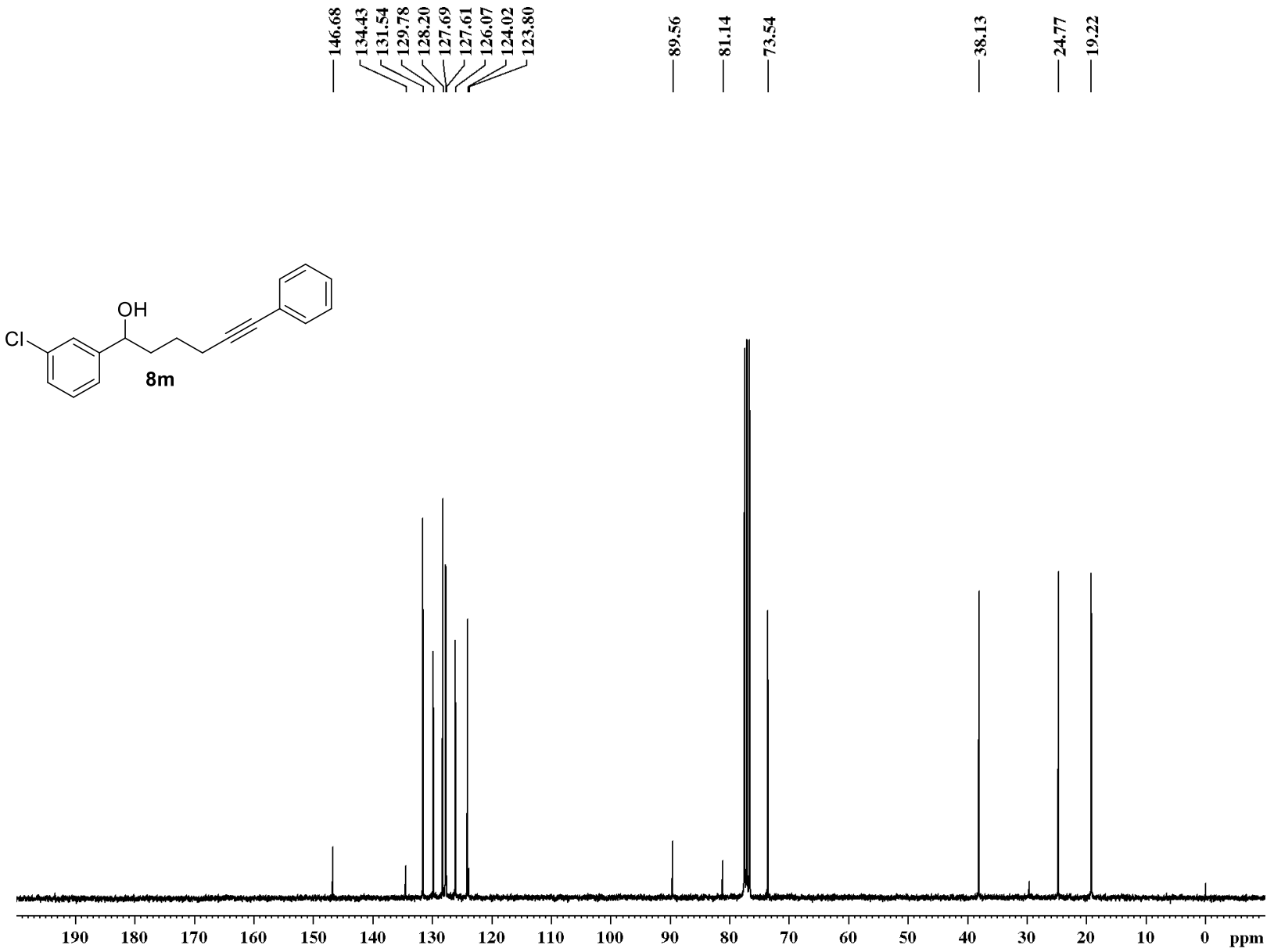
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127.69
127.61
126.07
124.02
123.80
89.56
81.14
73.54
38.13
24.77
19.22
OH
Cl
8m
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150
140
130
120
110
100
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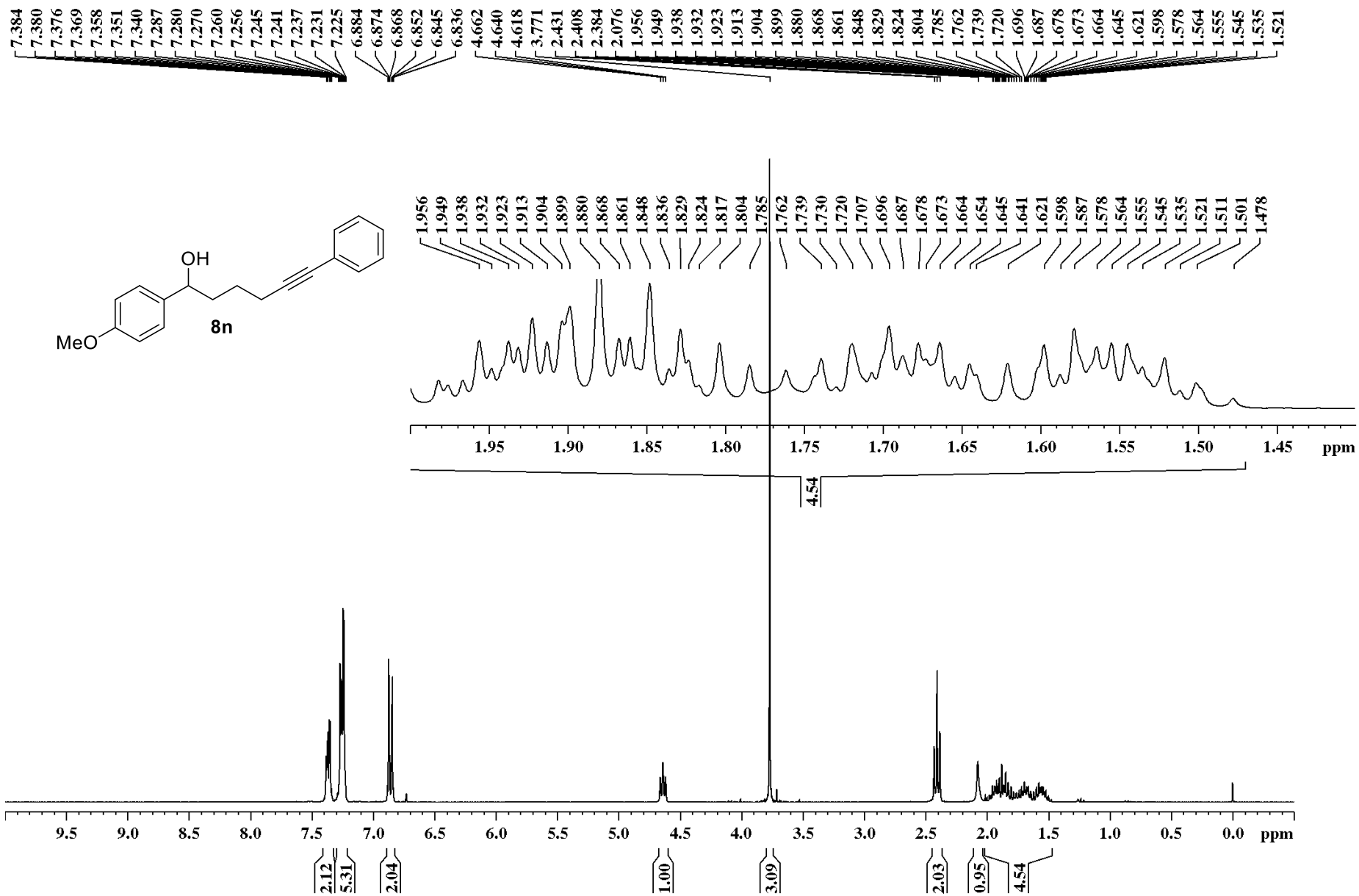
60
50
40
30
20
10
0
ppm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4.662 4.640 4.618 3.771 2.431 2.408 2.384 2.076 1.956 1.949 1.938 1.932 1.923 1.913 1.904 1.899 1.880 1.868 1.861 1.848 1.829 1.824 1.804 1.785 1.762 1.739 1.720 1.696 1.687 1.678 1.673 1.664 1.645 1.621 1.598 1.578 1.564 1.555 1.545 1.535 1.521
OH
MeO
8n
1.956 1.949 1.938 1.932 1.923 1.913 1.904 1.899 1.880 1.868 1.861 1.848 1.836 1.829 1.824 1.817 1.804 1.785 1.762 1.739 1.730 1.720 1.707 1.696 1.687 1.678 1.673 1.664 1.654 1.645 1.641 1.621 1.598 1.587 1.578 1.564 1.555 1.545 1.535 1.521 1.511 1.501 1.478
1.95 1.90 1.85 1.80 1.75 1.70 1.65 1.60 1.55 1.50 1.45 ppm
4.54
9.5 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 0.0 ppm
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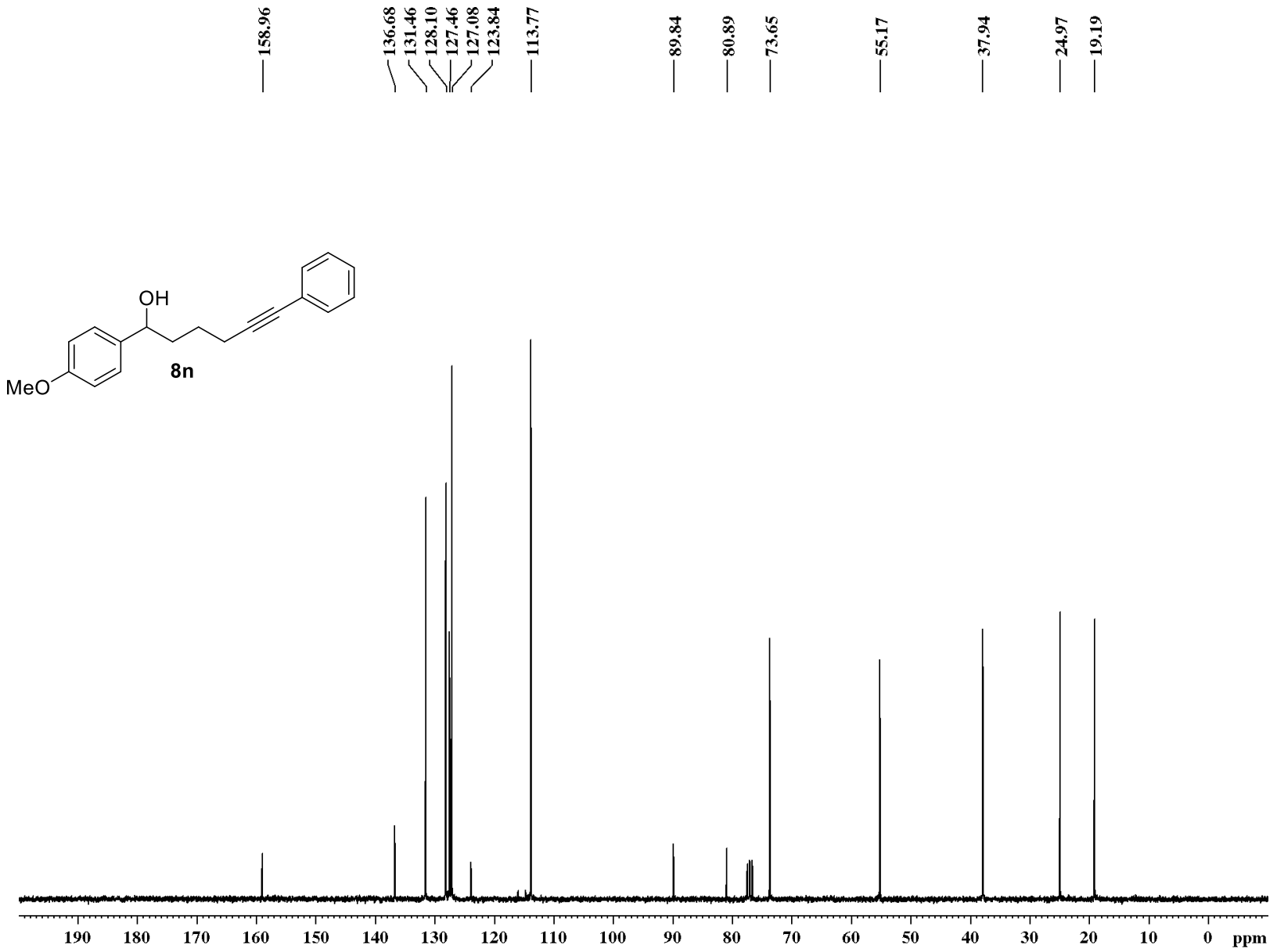
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37.94
24.97
19.19
OH
MeO
8n
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140
130
120
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100
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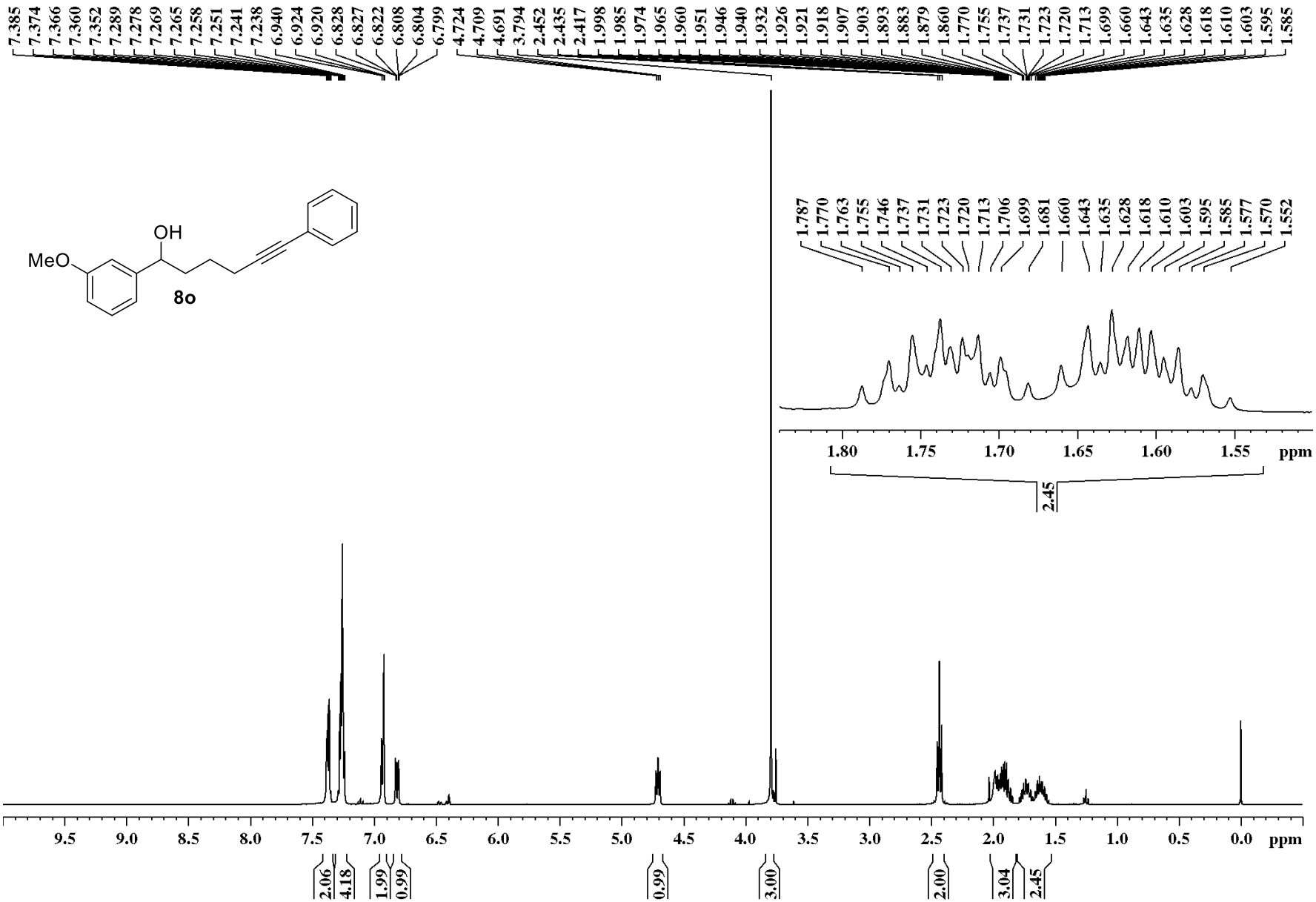
60
50
40
30
20
10
0
ppm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6.827
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1.737
1.731
1.723
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1.713
1.699
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1.643
1.635
1.628
1.618
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1.595
1.585
OH
MeO
8o
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1.595
1.585
1.577
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3.5
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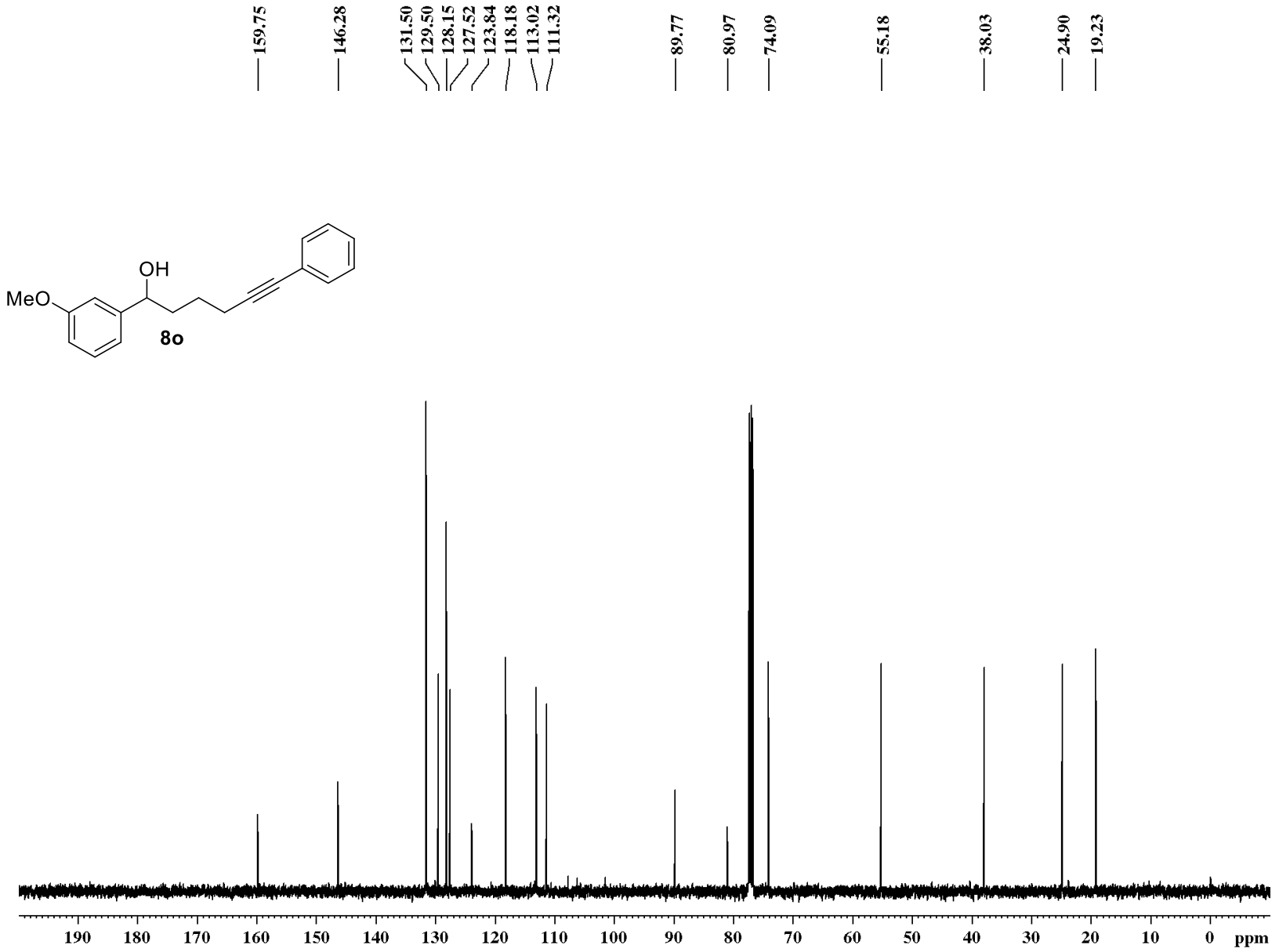
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74.09
55.18
38.03
24.90
19.23
OH
MeO
8o
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140
130
120
110
100
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60
50
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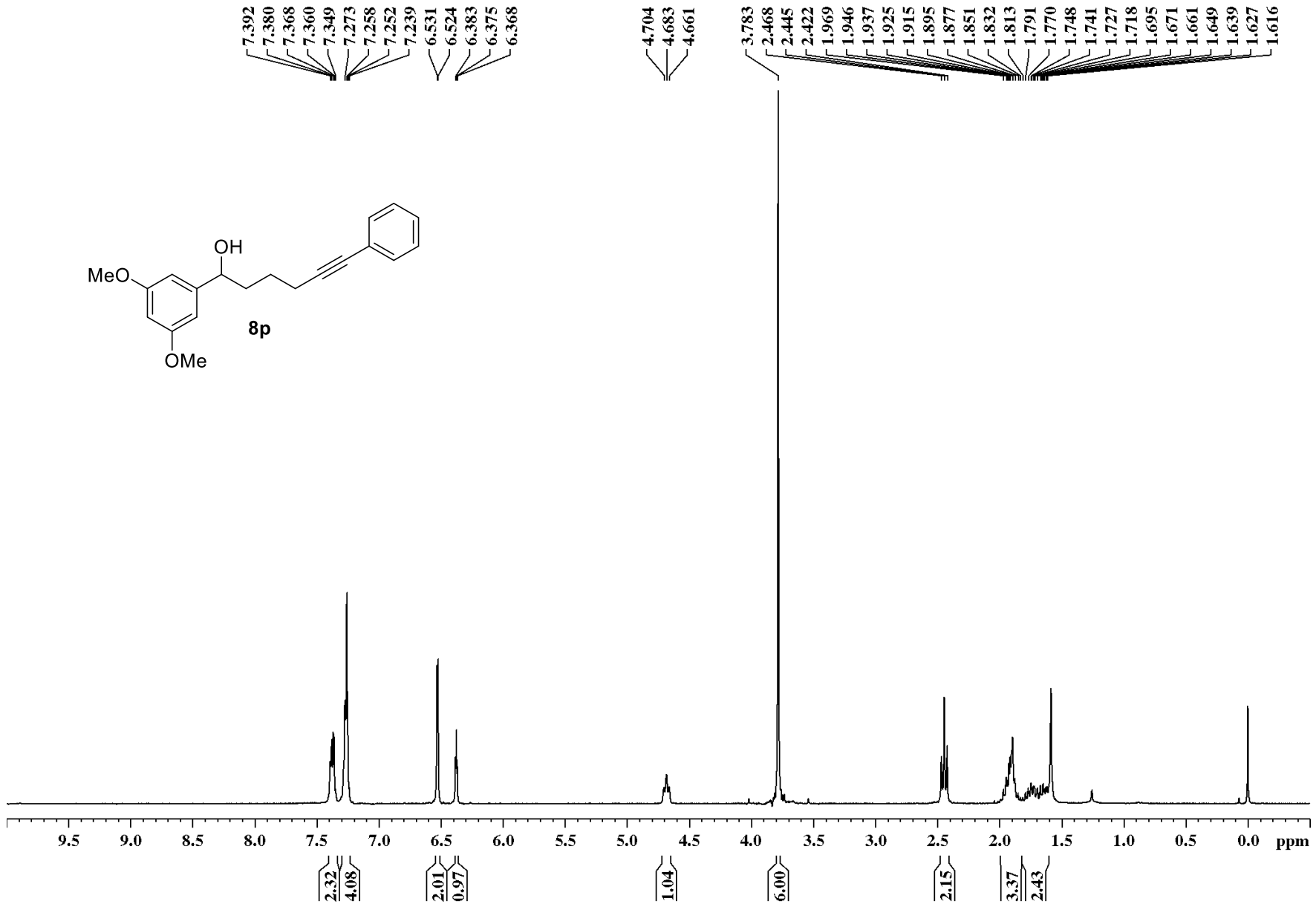
30
20
10
0
ppm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4.683
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3.783
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2.445
2.422
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1.925
1.915
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1.851
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1.639
1.627
1.616
OH
MeO
OMe
8p
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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1.5
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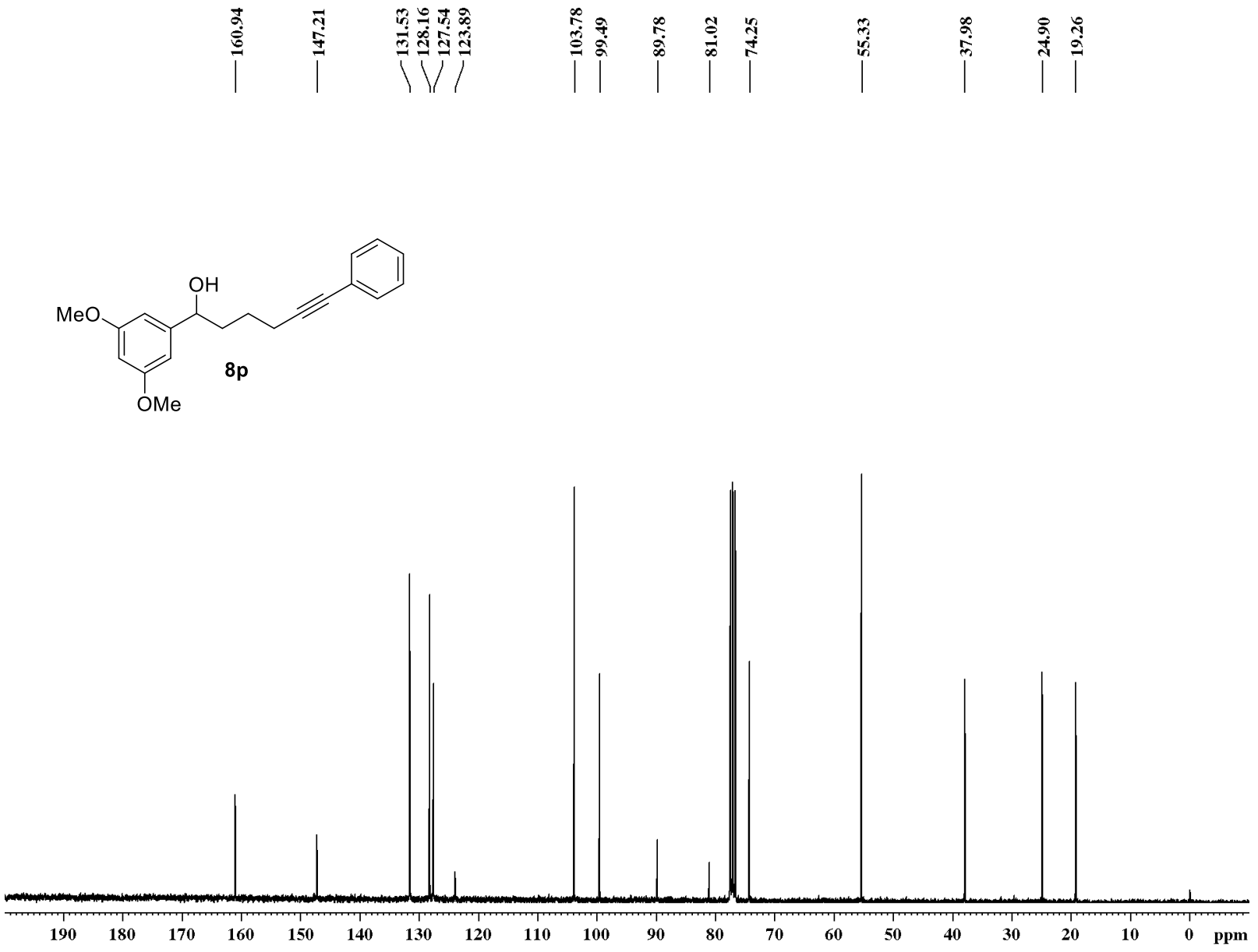
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37.98
24.90
19.26
OH
MeO
OMe
8p
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60
50
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30
20
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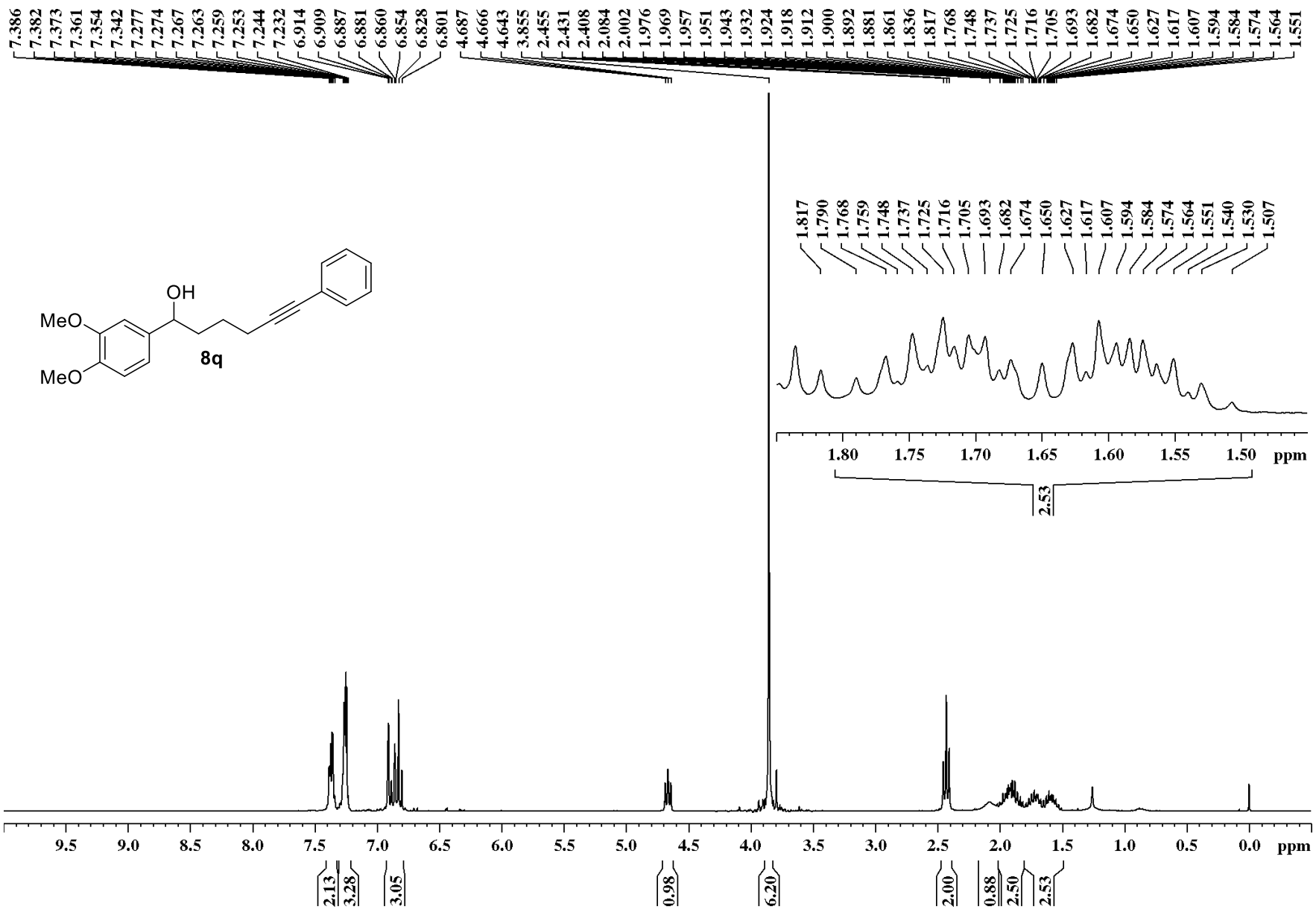
0
ppm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1.881
1.861
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1.748
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OH
MeO
MeO
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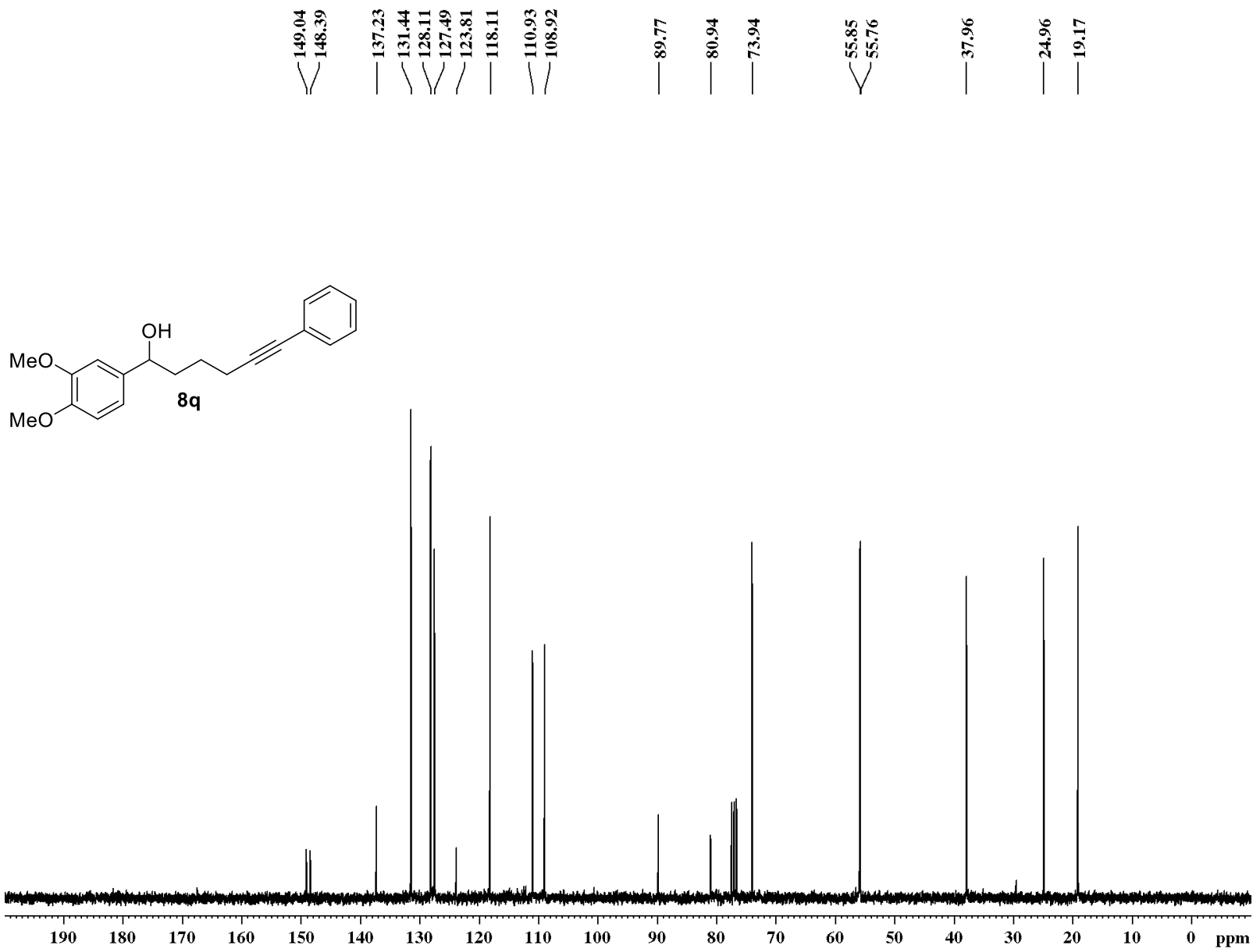
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55.76
37.96
24.96
19.17
OH
MeO
MeO
8q
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120
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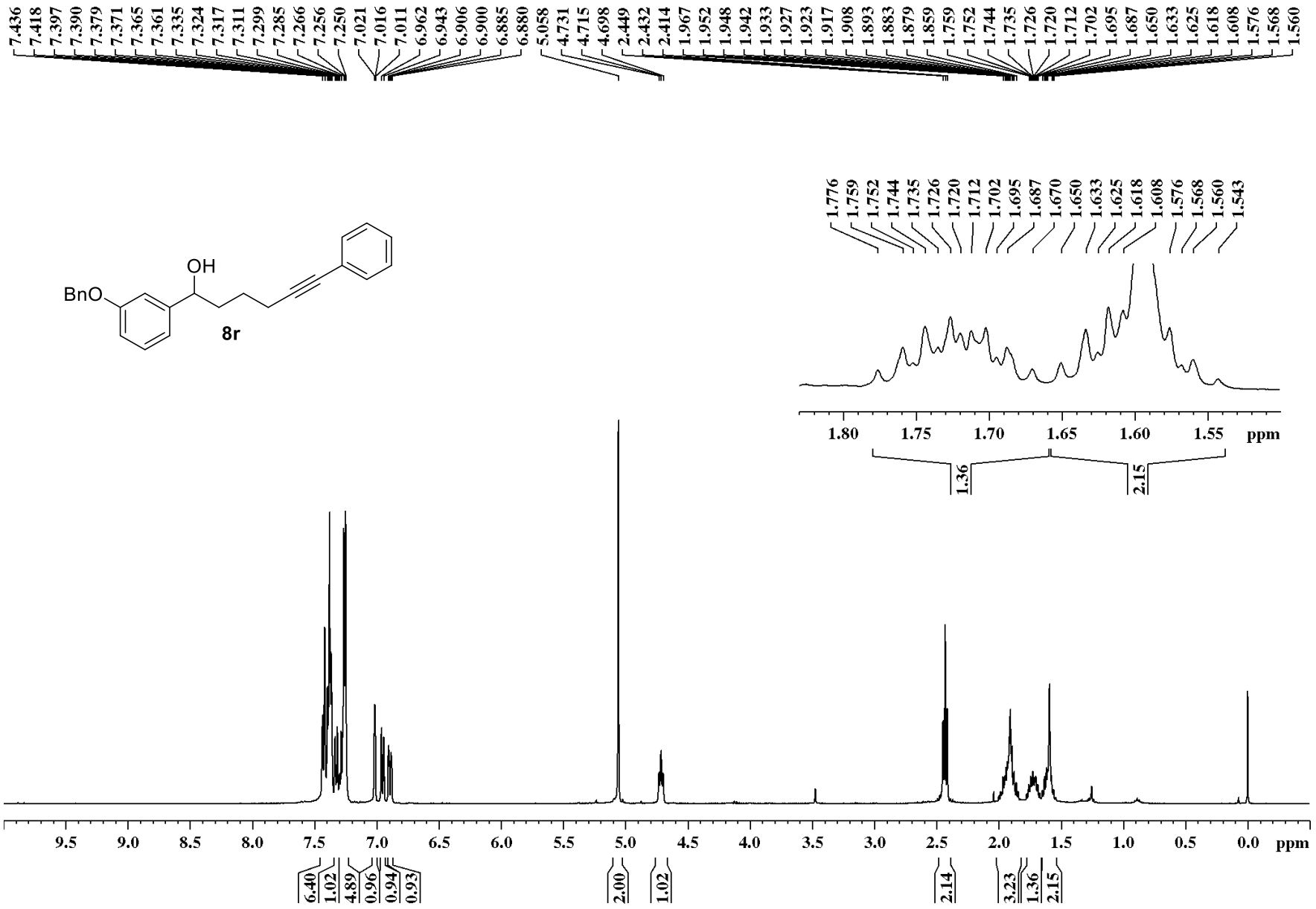
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1.720
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1.695
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ppm
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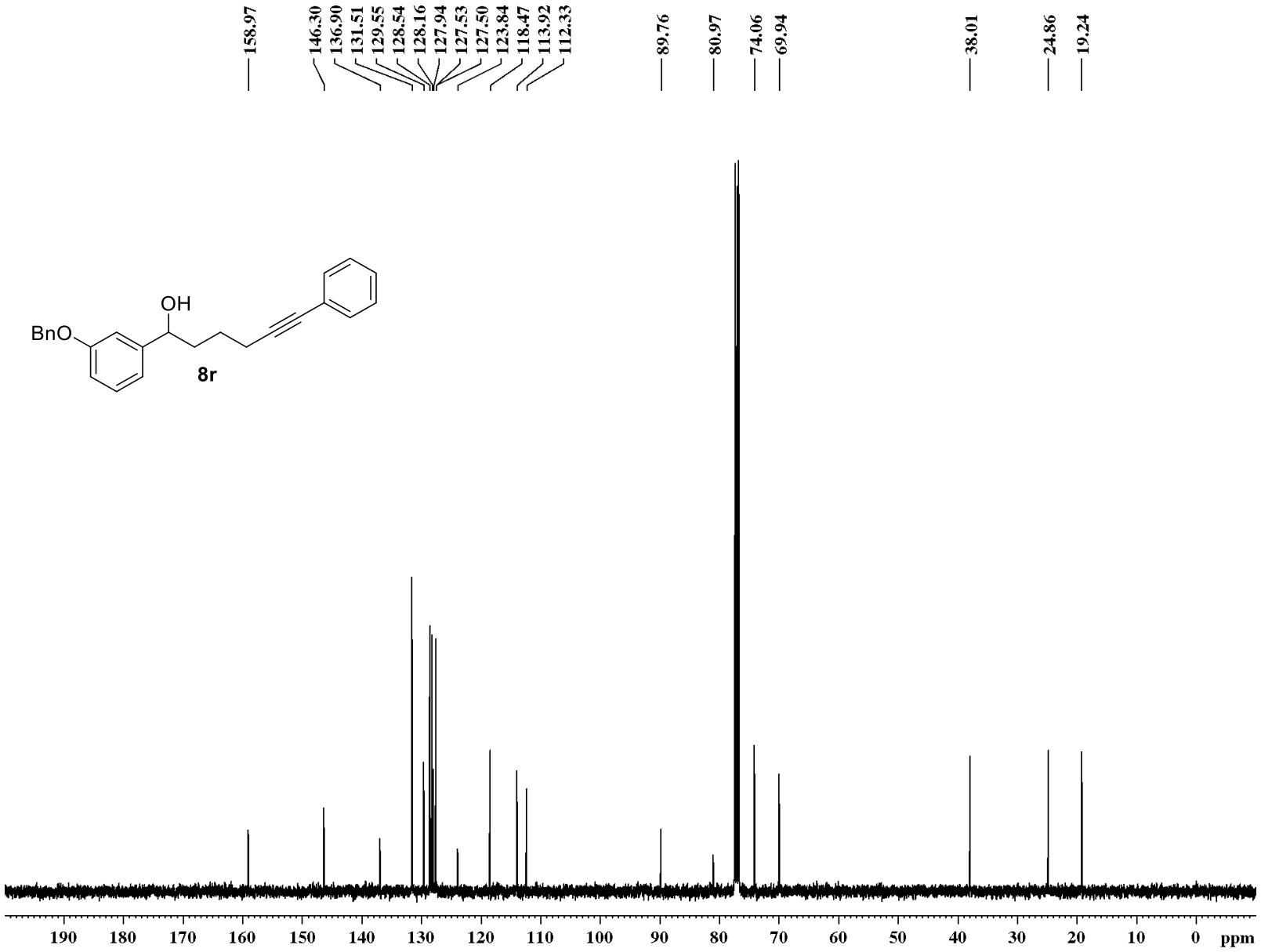
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80.97
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38.01
24.86
19.24
OH
BnO
8r
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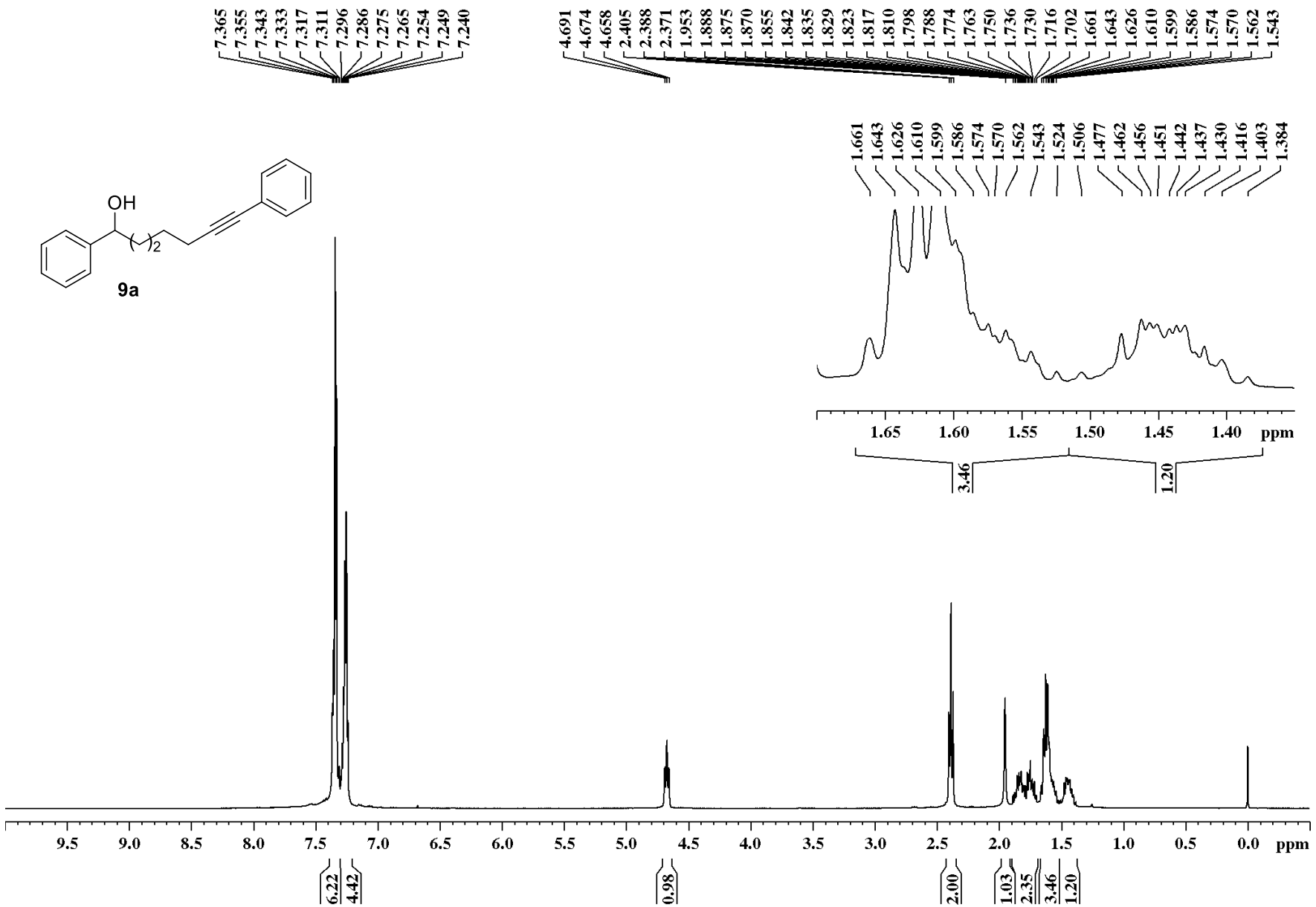
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1.661
1.643
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1.610
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1.586
1.574
1.570
1.562
1.543
OH
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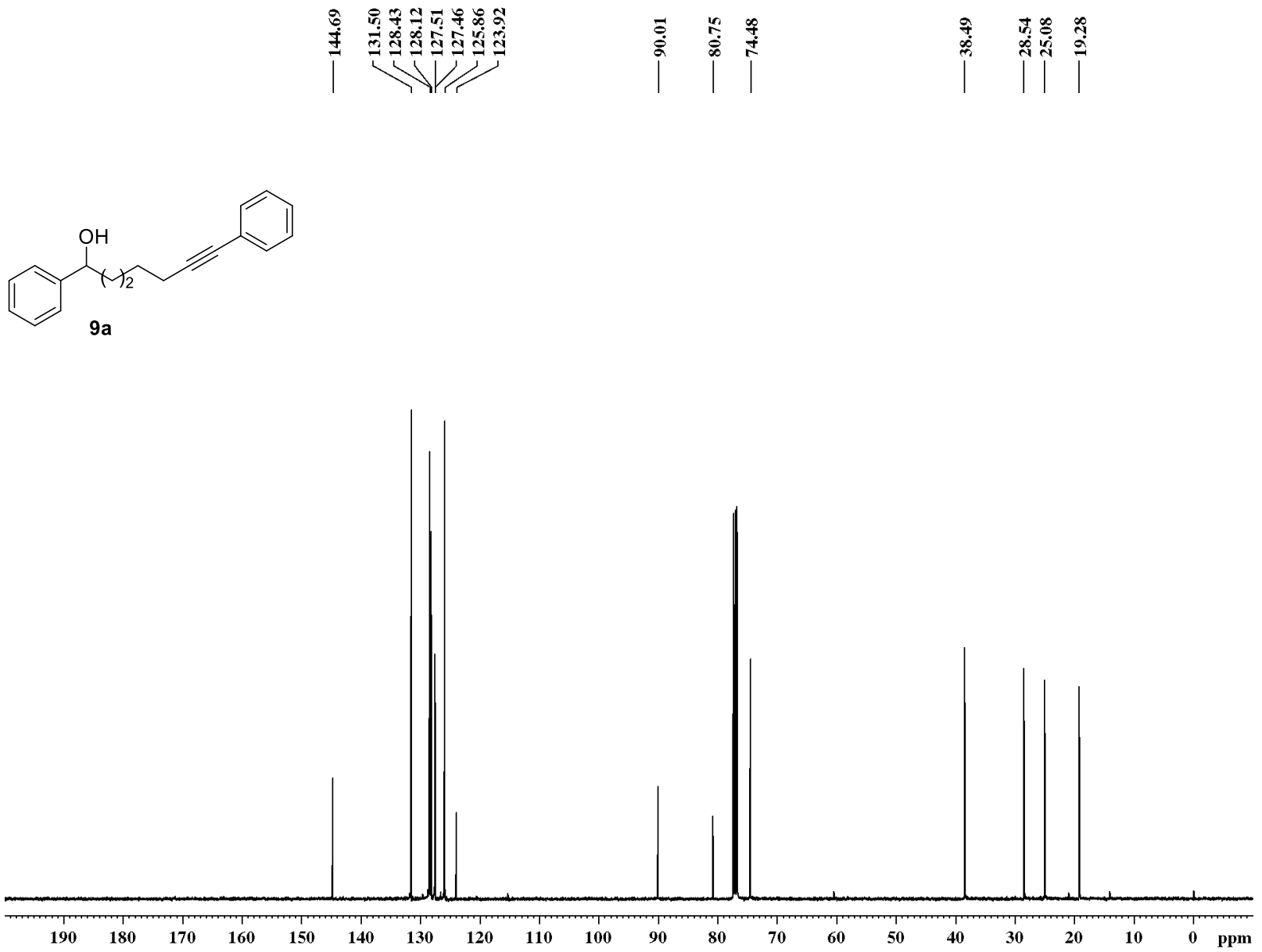
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19.28
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2
9a
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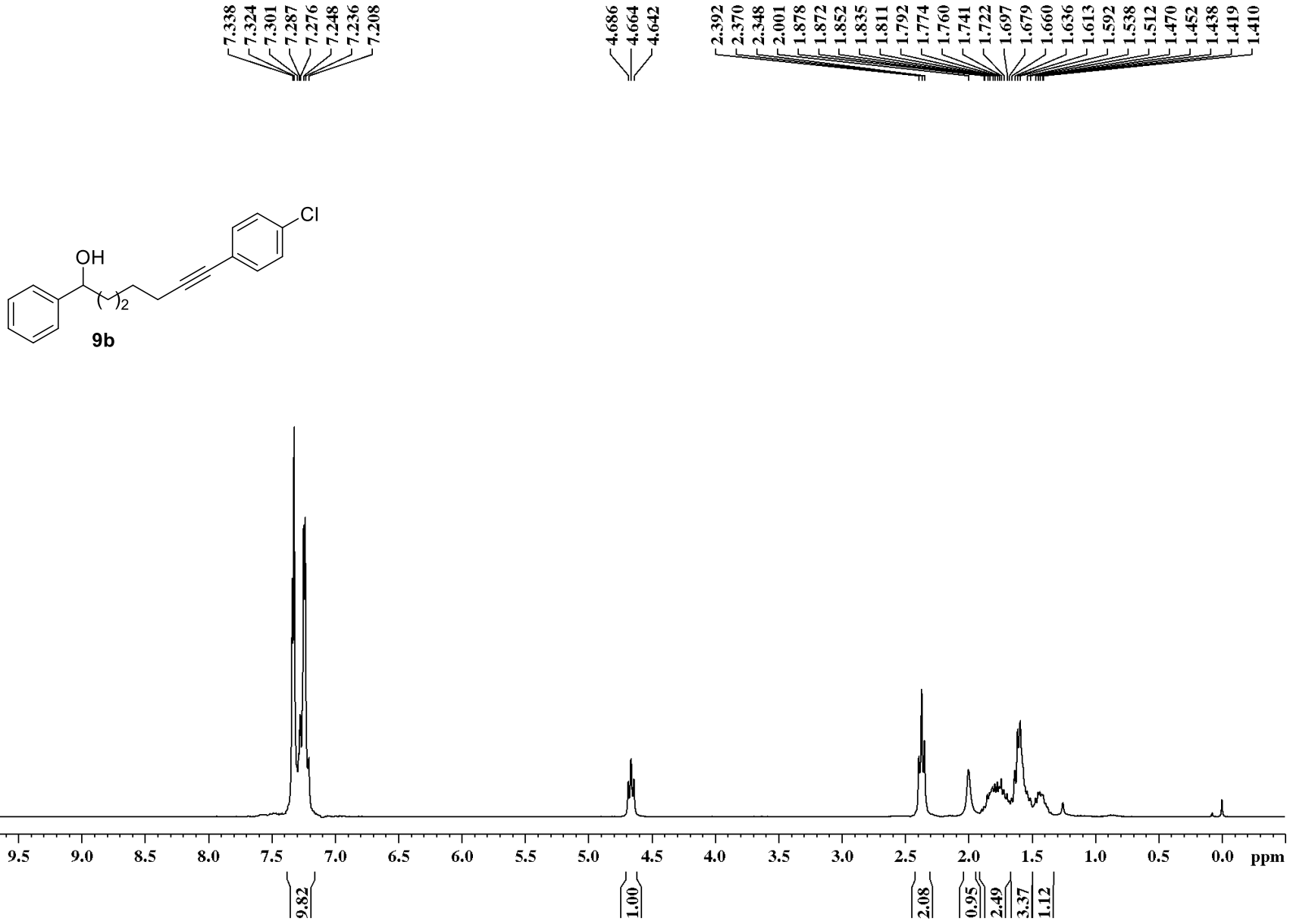
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1.722
1.697
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1.613
1.592
1.538
1.512
1.470
1.452
1.438
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1.410
Cl
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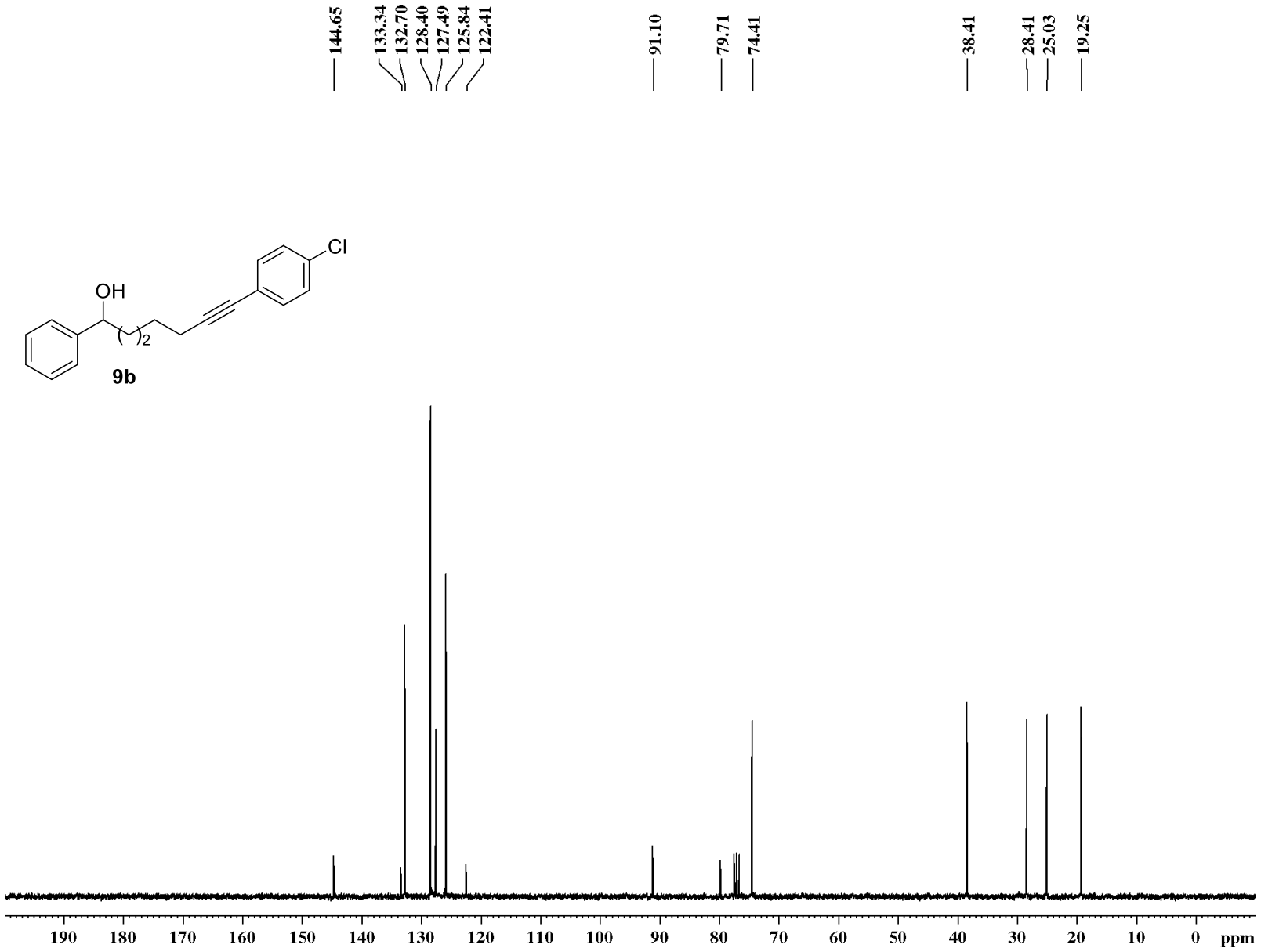
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Cl
OH
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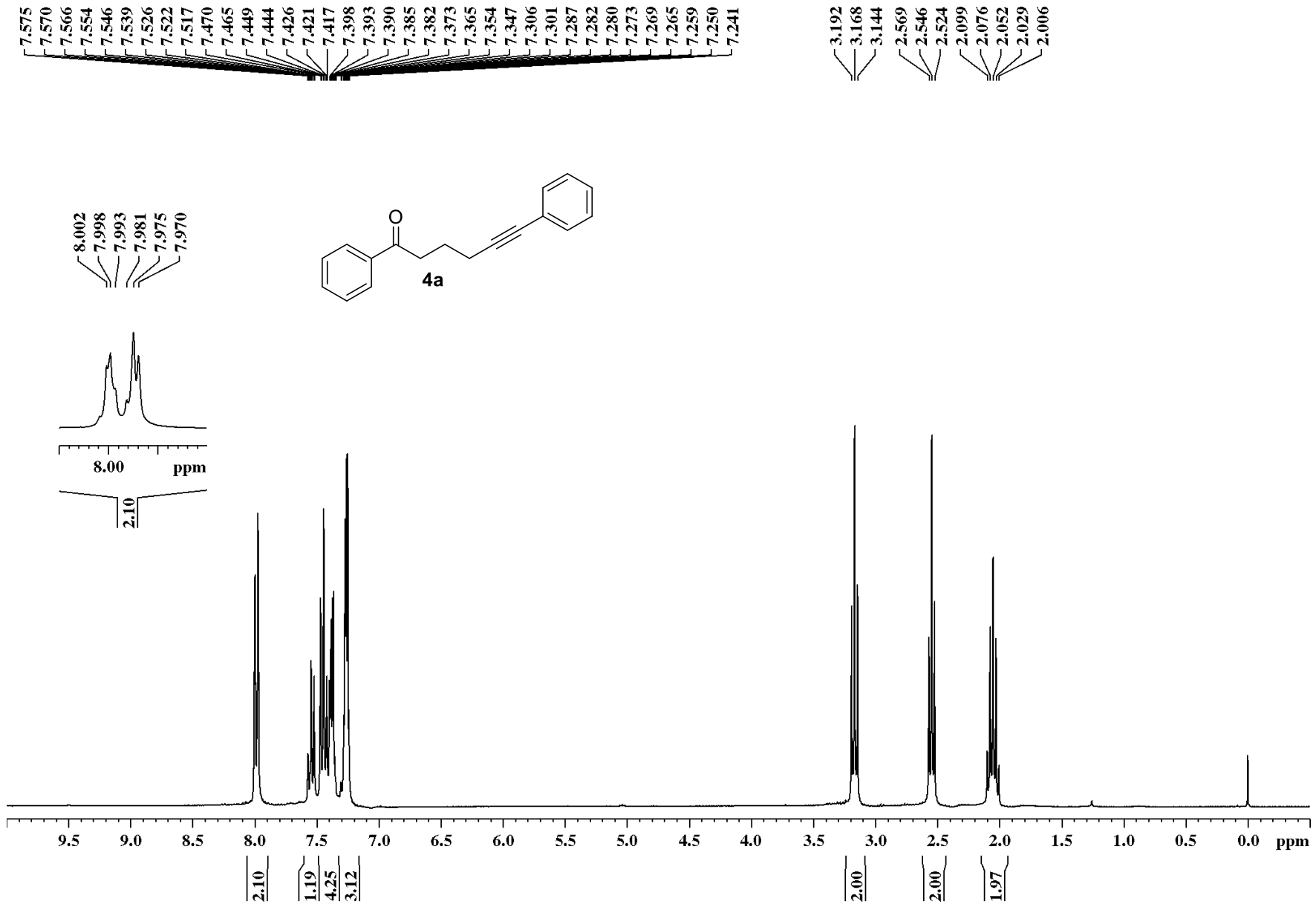
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7.444
7.426
7.421
7.417
7.398
7.393
7.390
7.385
7.382
7.373
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7.301
7.287
7.282
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7.250
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3.144
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2.524
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2.006
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7.981
7.975
7.970
8.00
ppm
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O
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8.5
8.0
7.5
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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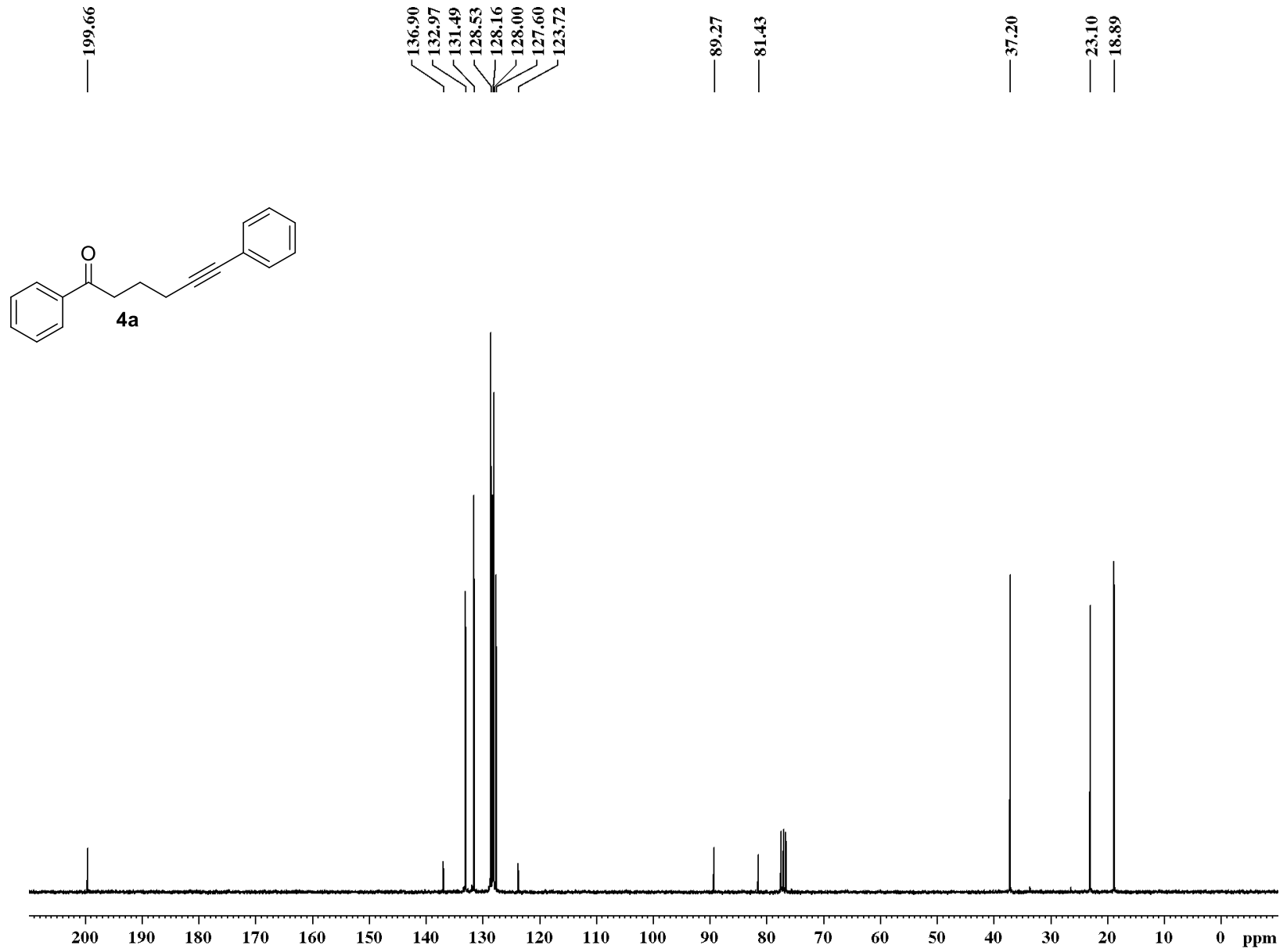
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18.89
O
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120
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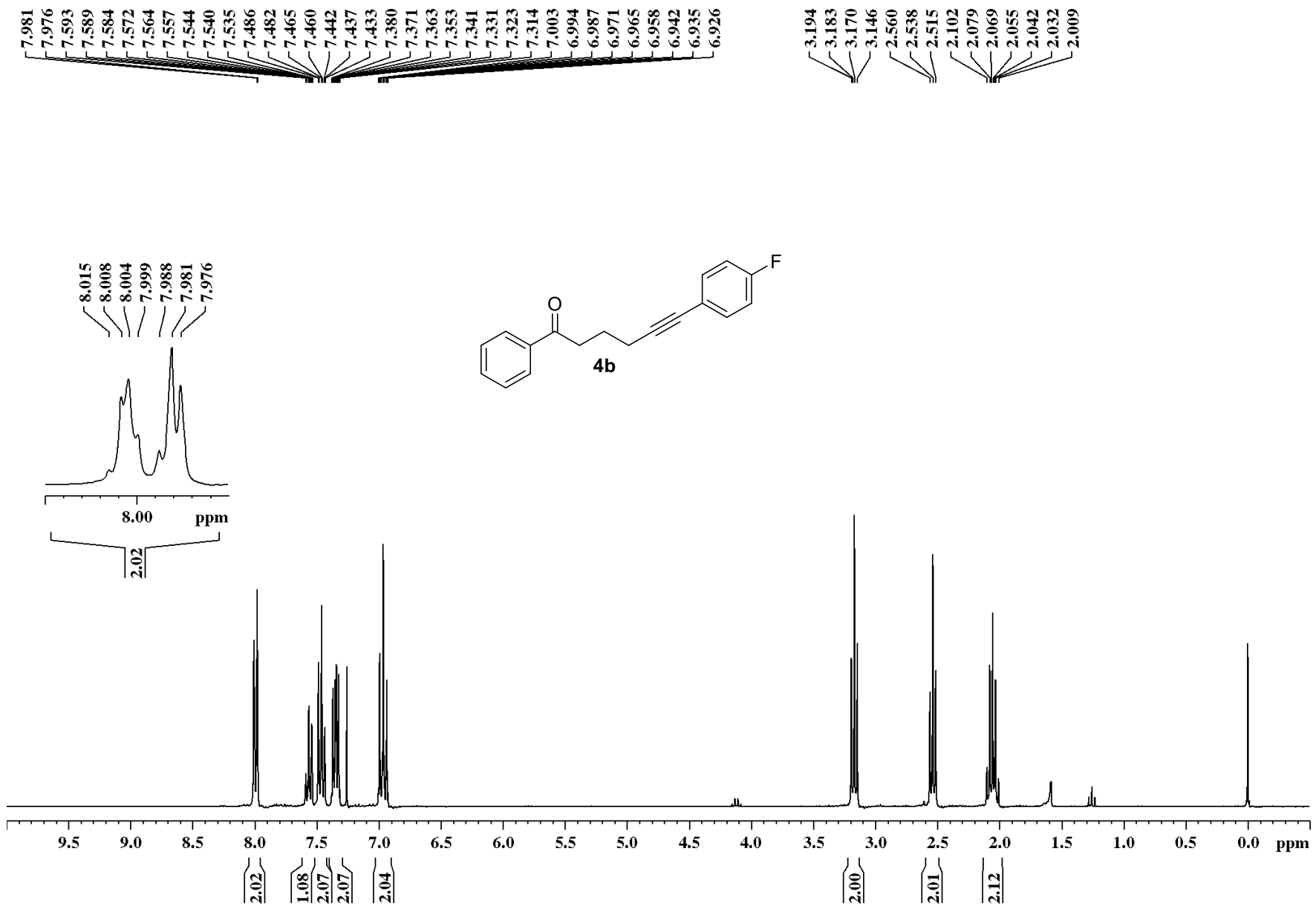
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7.460
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7.437
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7.353
7.341
7.331
7.323
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6.987
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6.965
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6.935
6.926
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3.183
3.170
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2.079
2.069
2.055
2.042
2.032
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8.008
8.004
7.999
7.988
7.981
7.976
8.00
ppm
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O
F
4b
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6.0
5.5
5.0
4.5
4.0
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2.0
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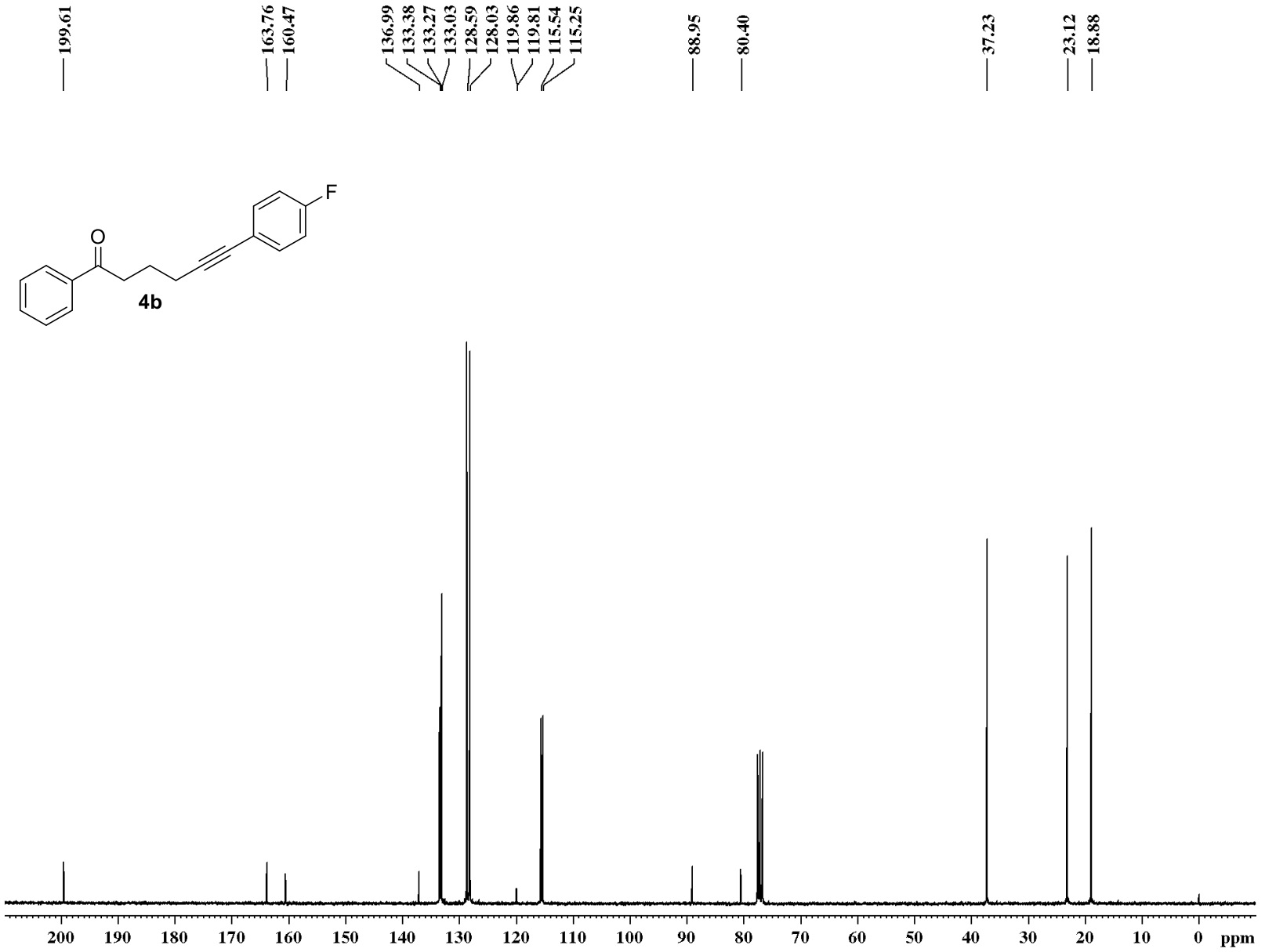
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80.40
37.23
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18.88
F
O
4b
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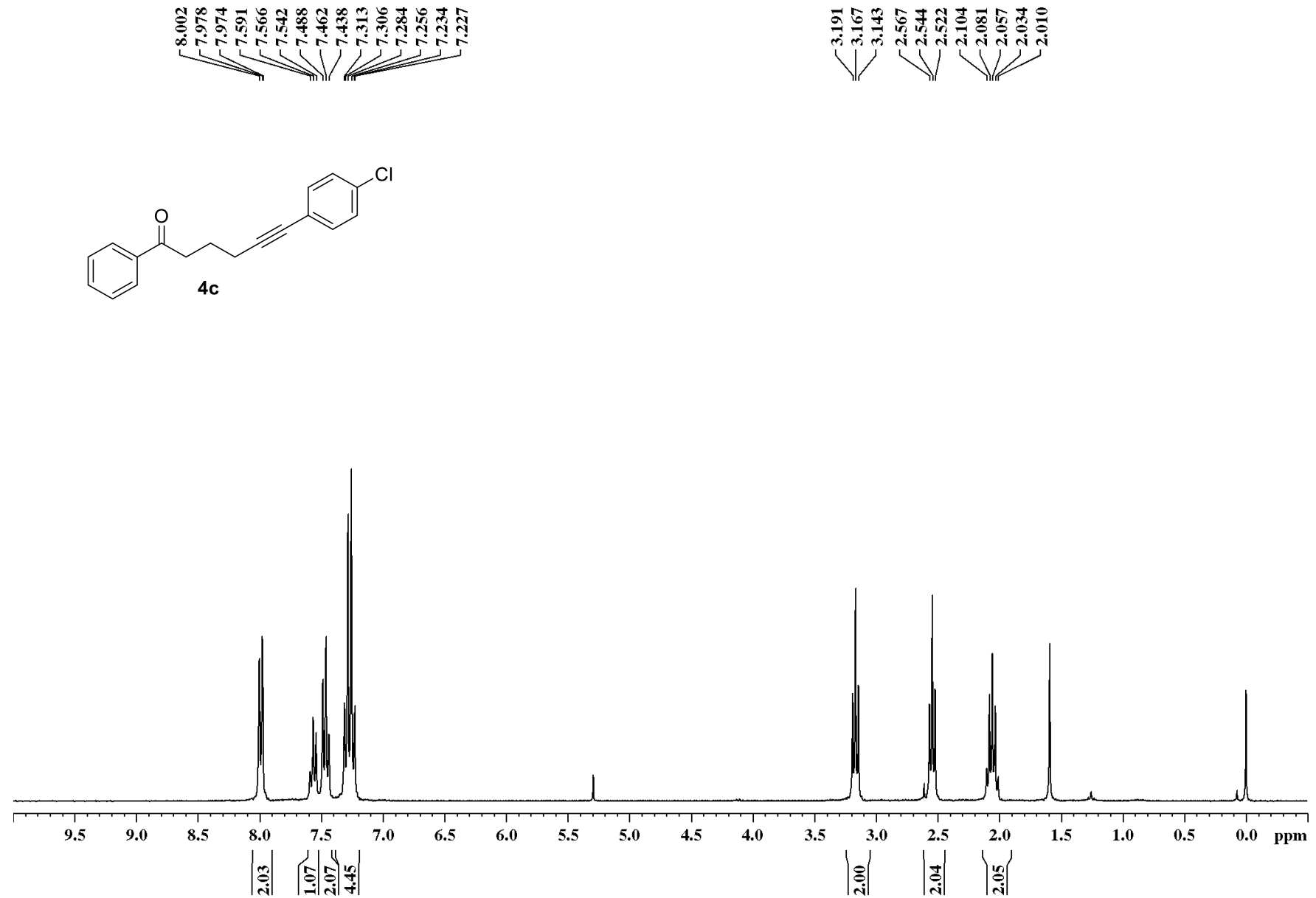
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2.544
2.522
2.104
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2.057
2.034
2.010
Cl
O
4c
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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2.07
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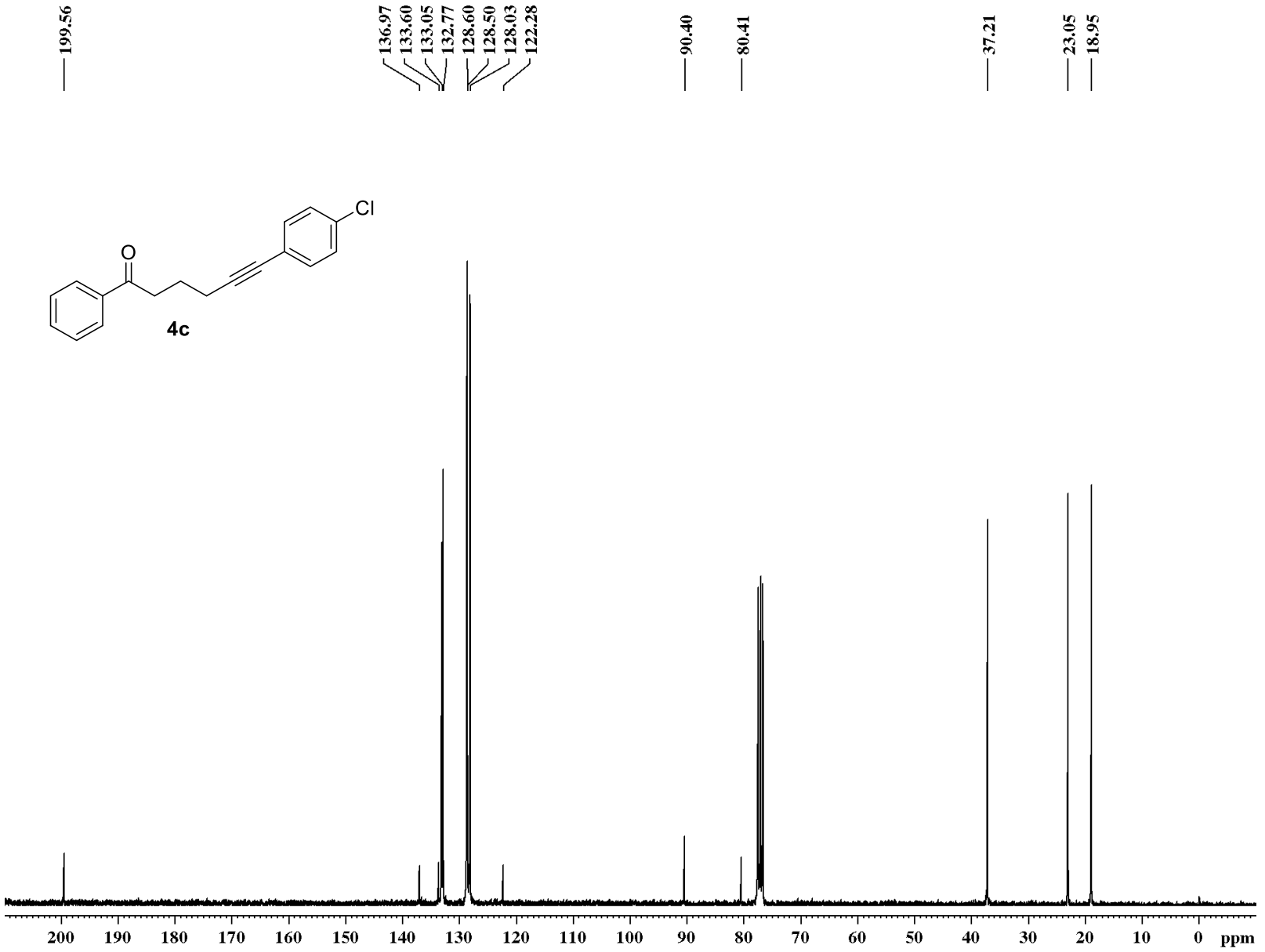
2.05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23.05
18.95
Cl
O
4c
200
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140
130
120
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50
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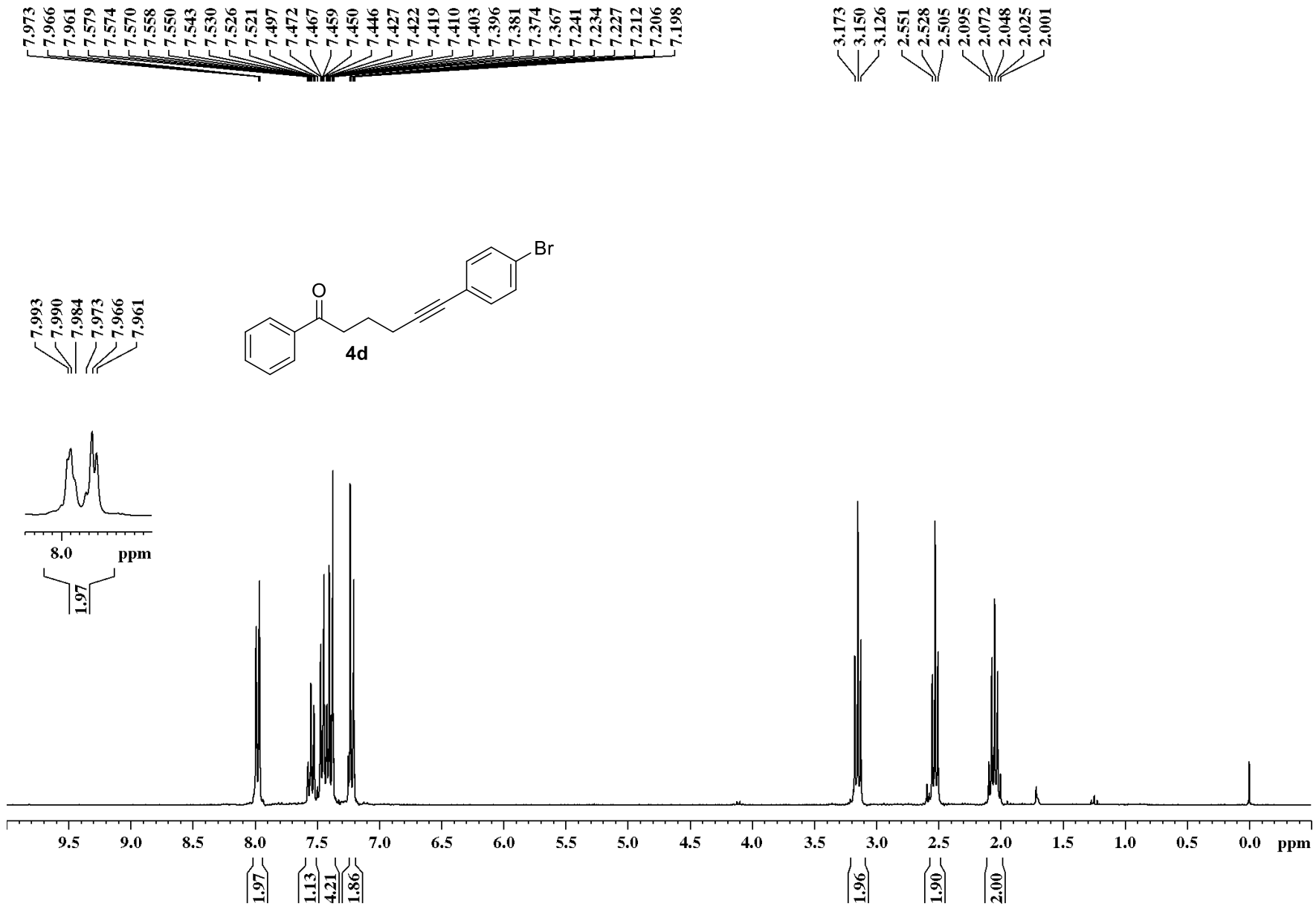
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7.0
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4.5
4.0
3.5
3.0
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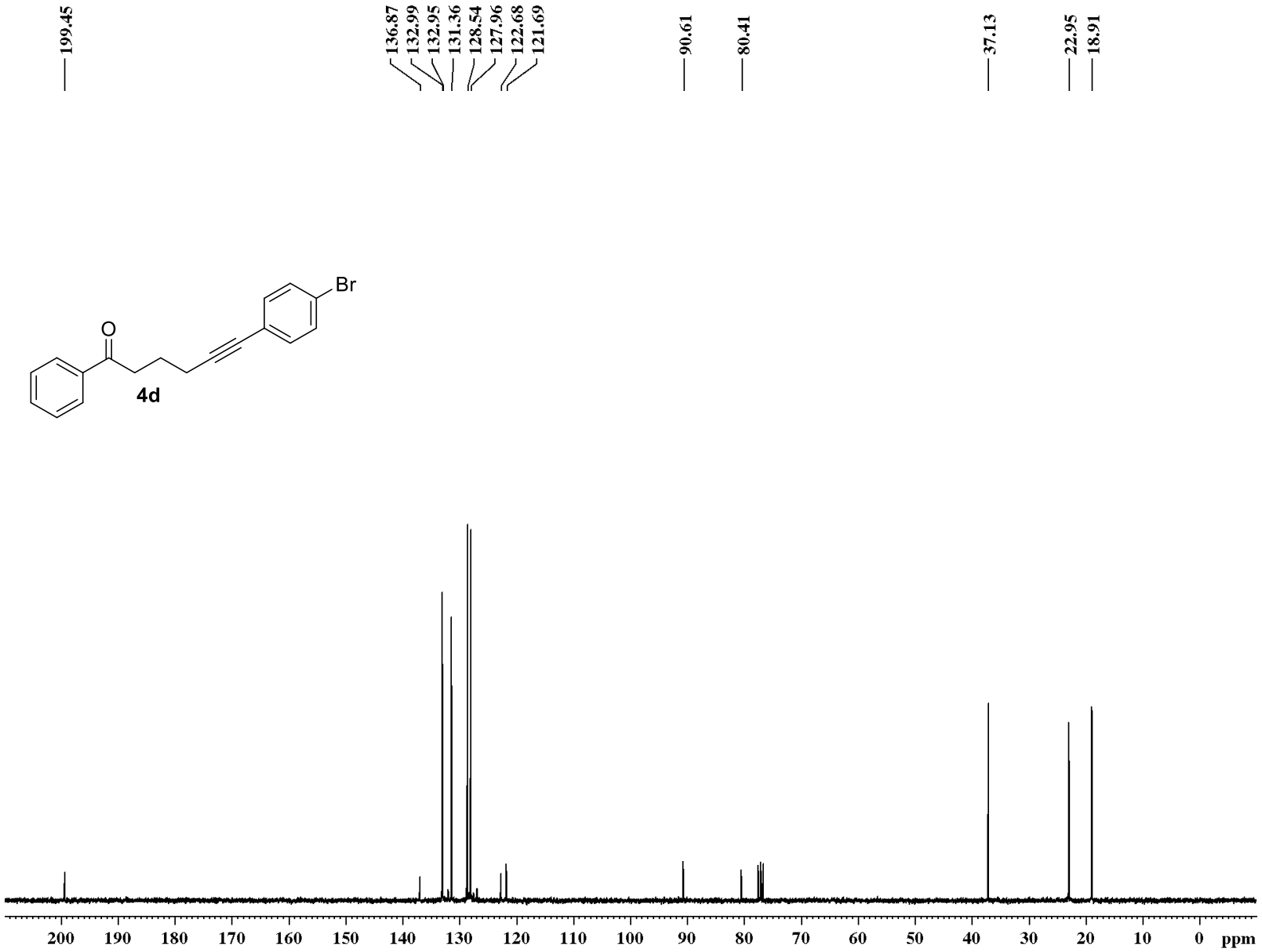
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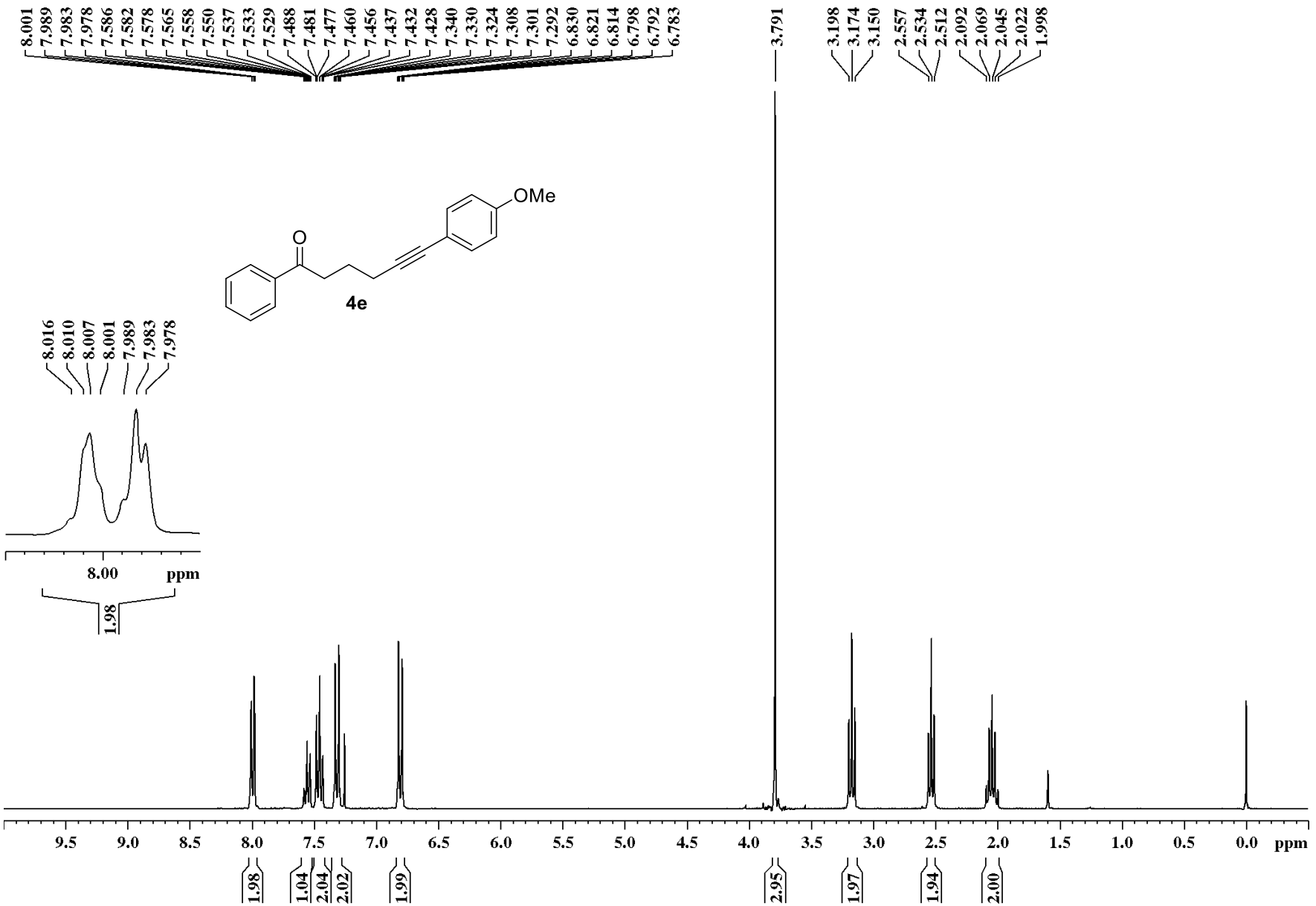
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7.301
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6.814
6.798
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3.150
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2.045
2.022
1.998
OMe
O
4e
8.016
8.010
8.007
8.001
7.989
7.983
7.978
8.00
ppm
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8.0
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6.0
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5.0
4.5
4.0
3.5
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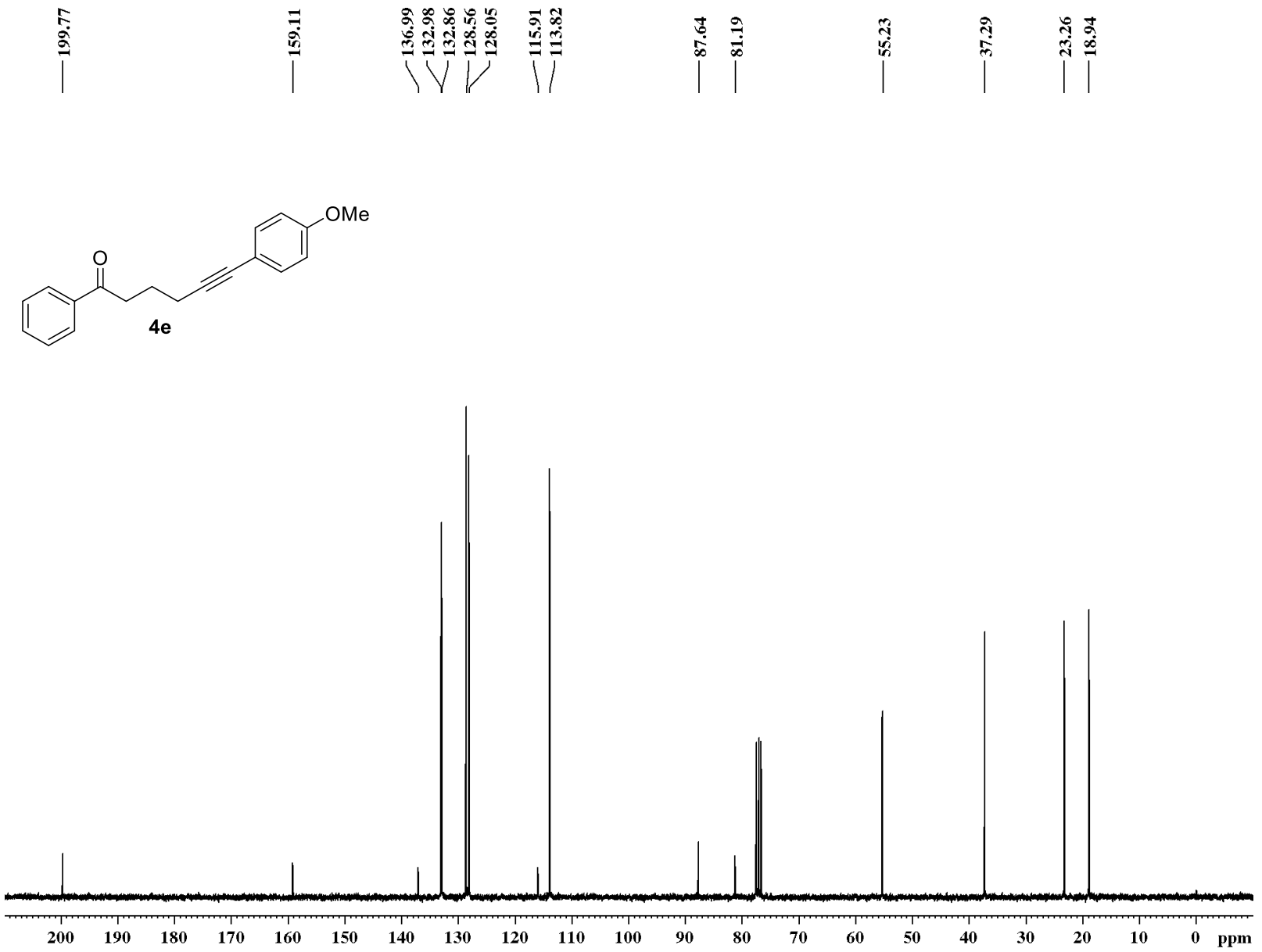
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O
4e
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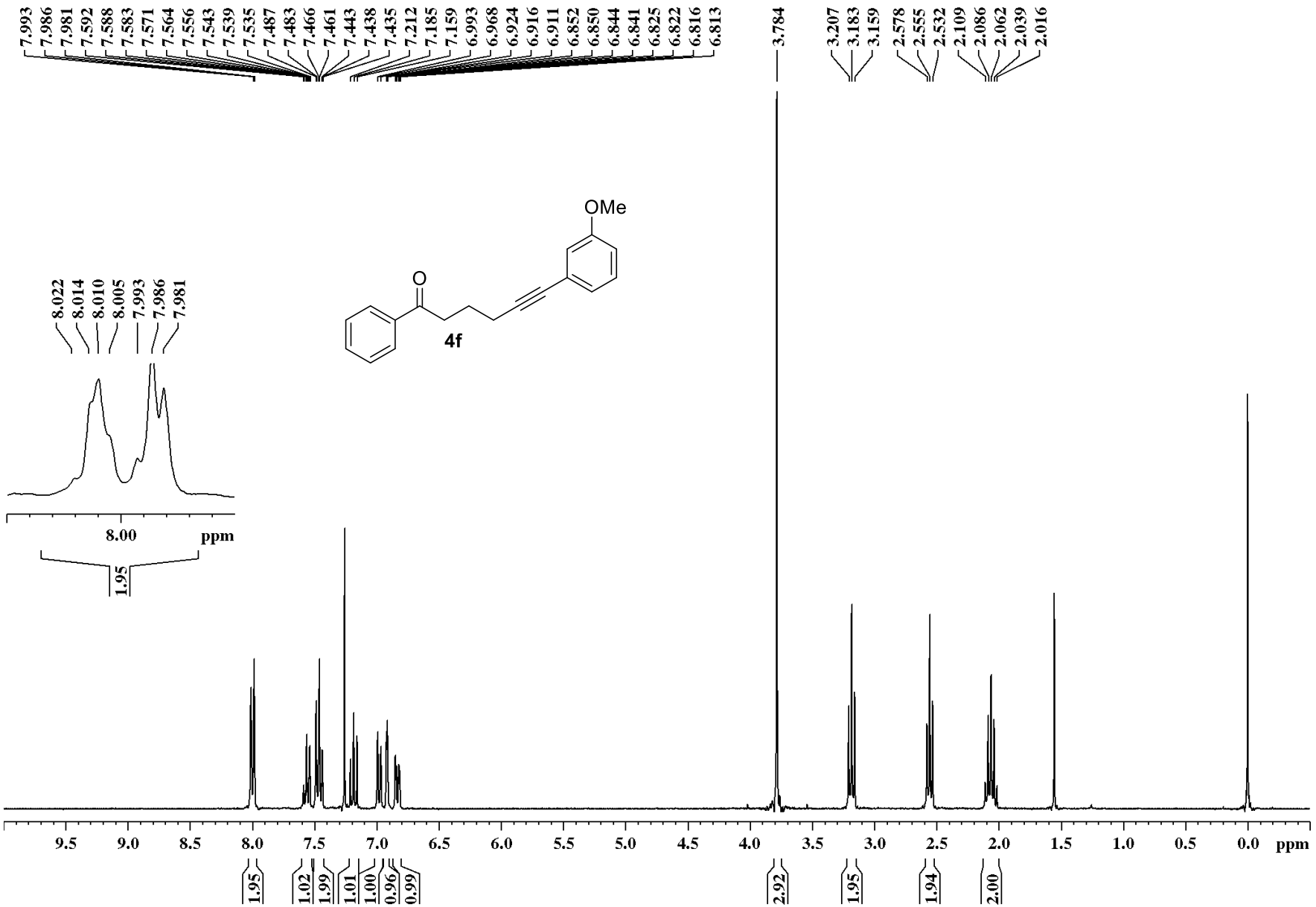
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O
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ppm
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6.0
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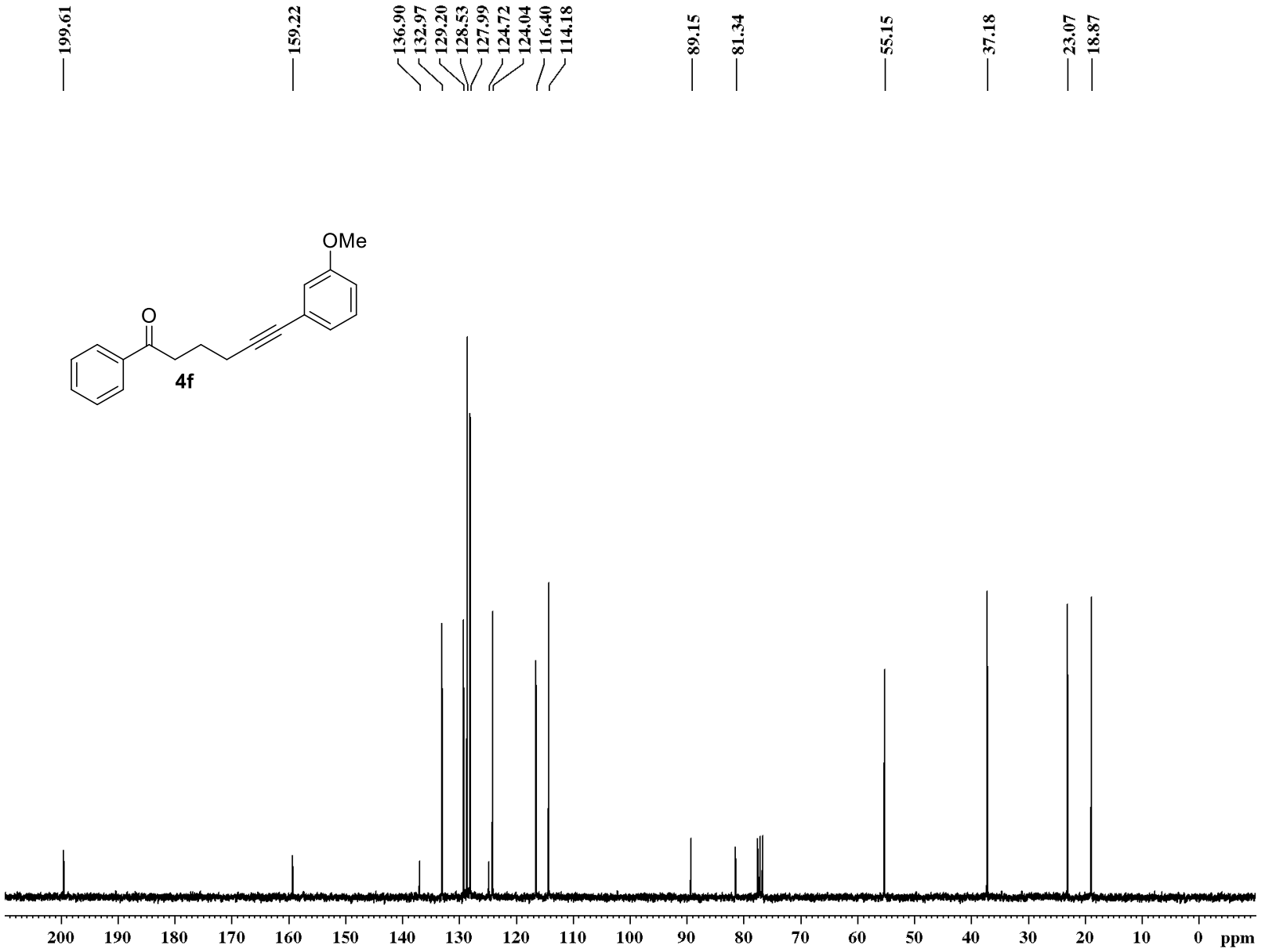
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55.15
37.18
23.07
18.87
OMe
O
4f
200
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110
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80
70
60
50
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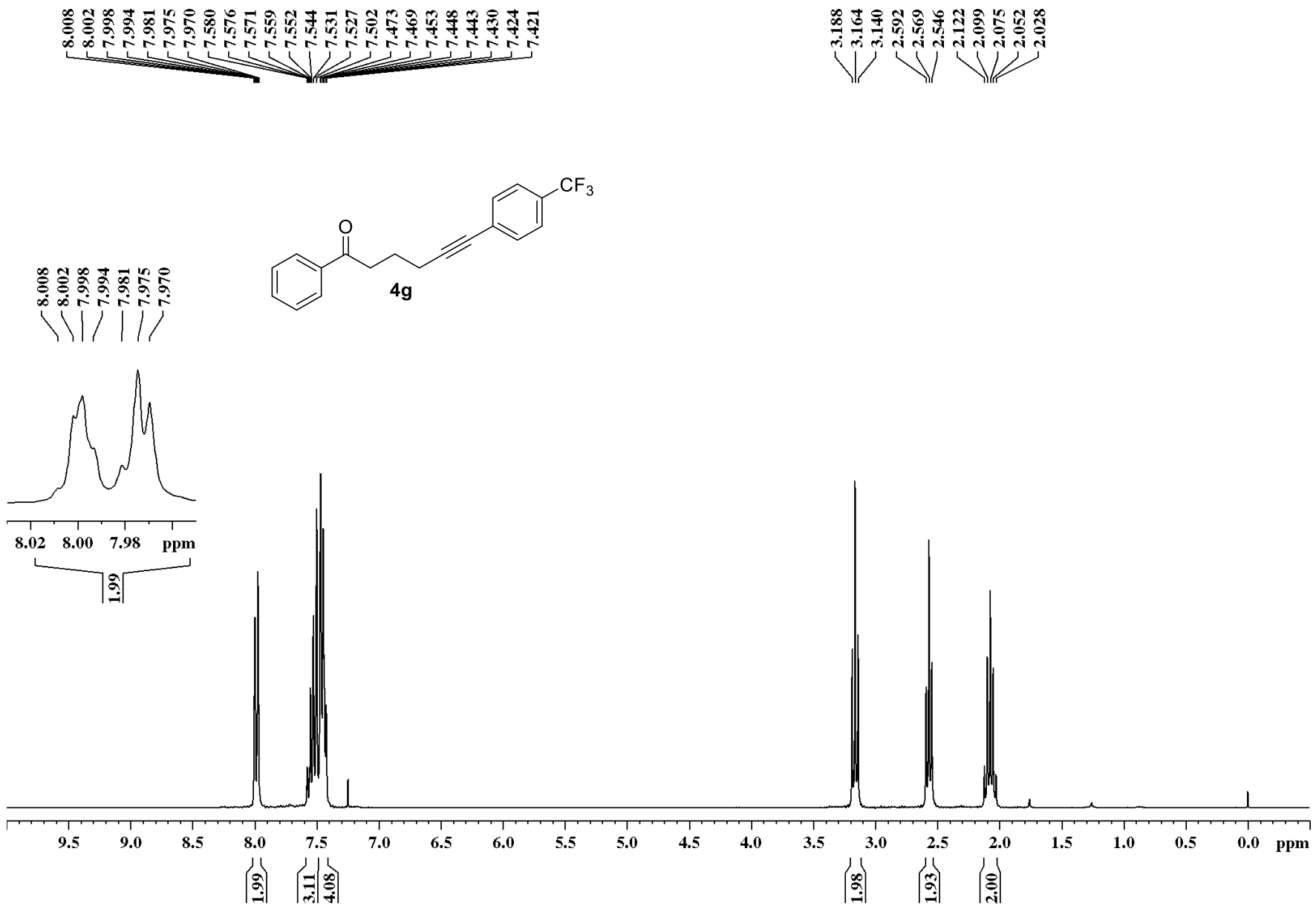
30
20
10
0
ppm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3.140
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2.052
2.028
CF3
O
4g
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8.00
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6.5
6.0
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4.0
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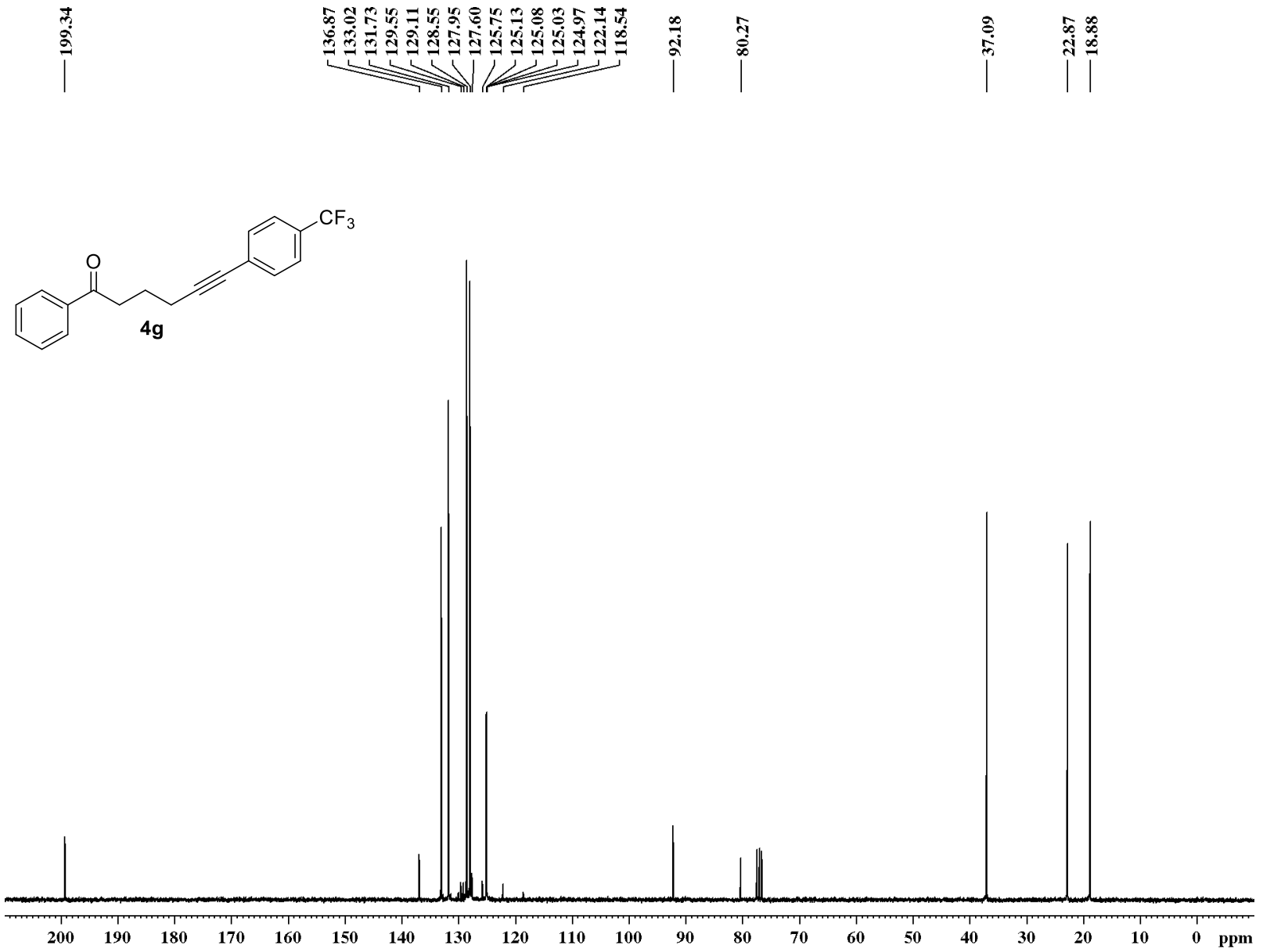
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122.14
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CF3
O
4g
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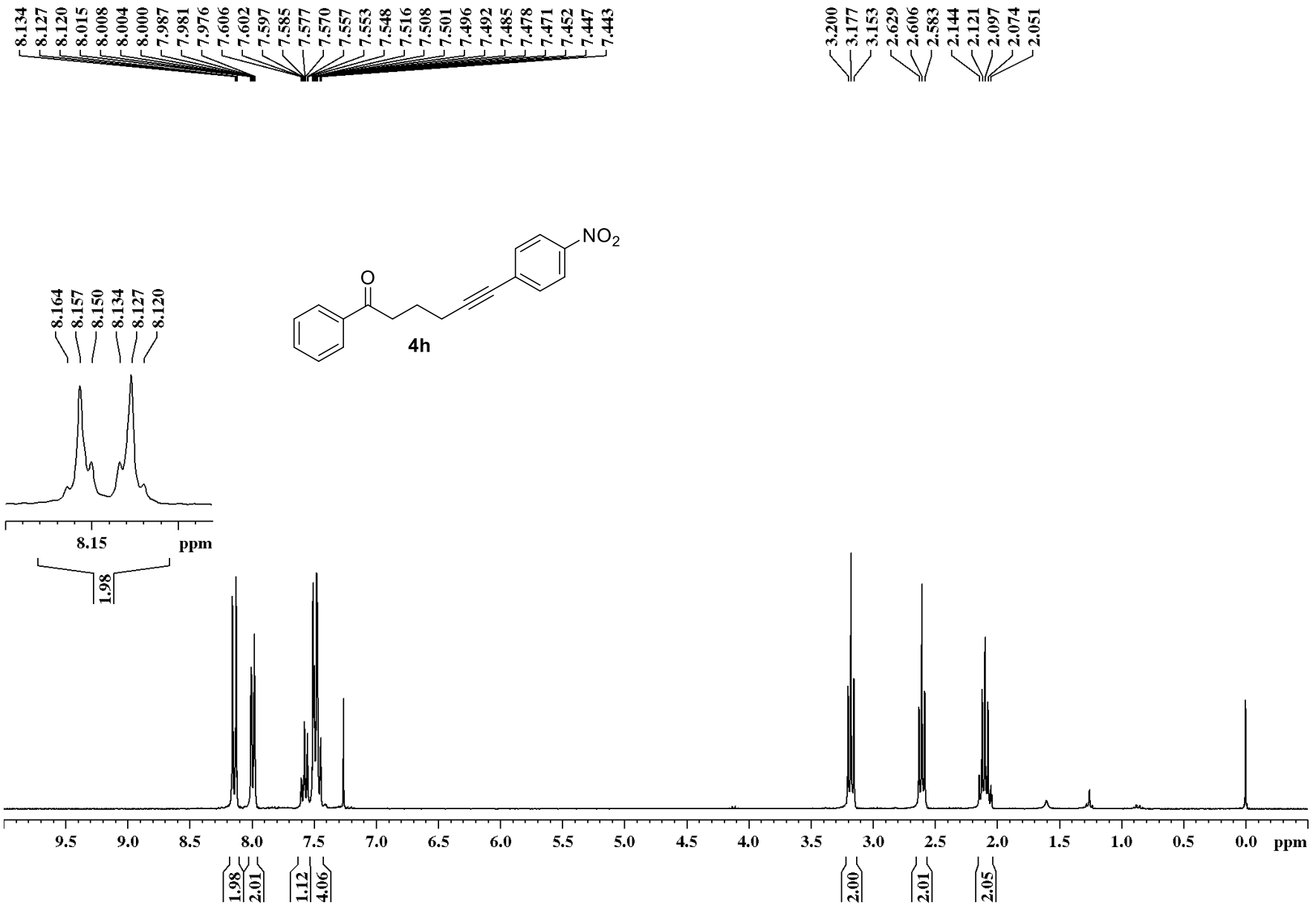
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7.577
7.570
7.557
7.553
7.548
7.516
7.508
7.501
7.496
7.492
7.485
7.478
7.471
7.452
7.447
7.443
3.200
3.177
3.153
2.629
2.606
2.583
2.144
2.121
2.097
2.074
2.051
NO2
O
4h
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8.157
8.150
8.134
8.127
8.120
8.15
ppm
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8.0
7.5
7.0
6.5
6.0
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5.0
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4.0
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3.0
2.5
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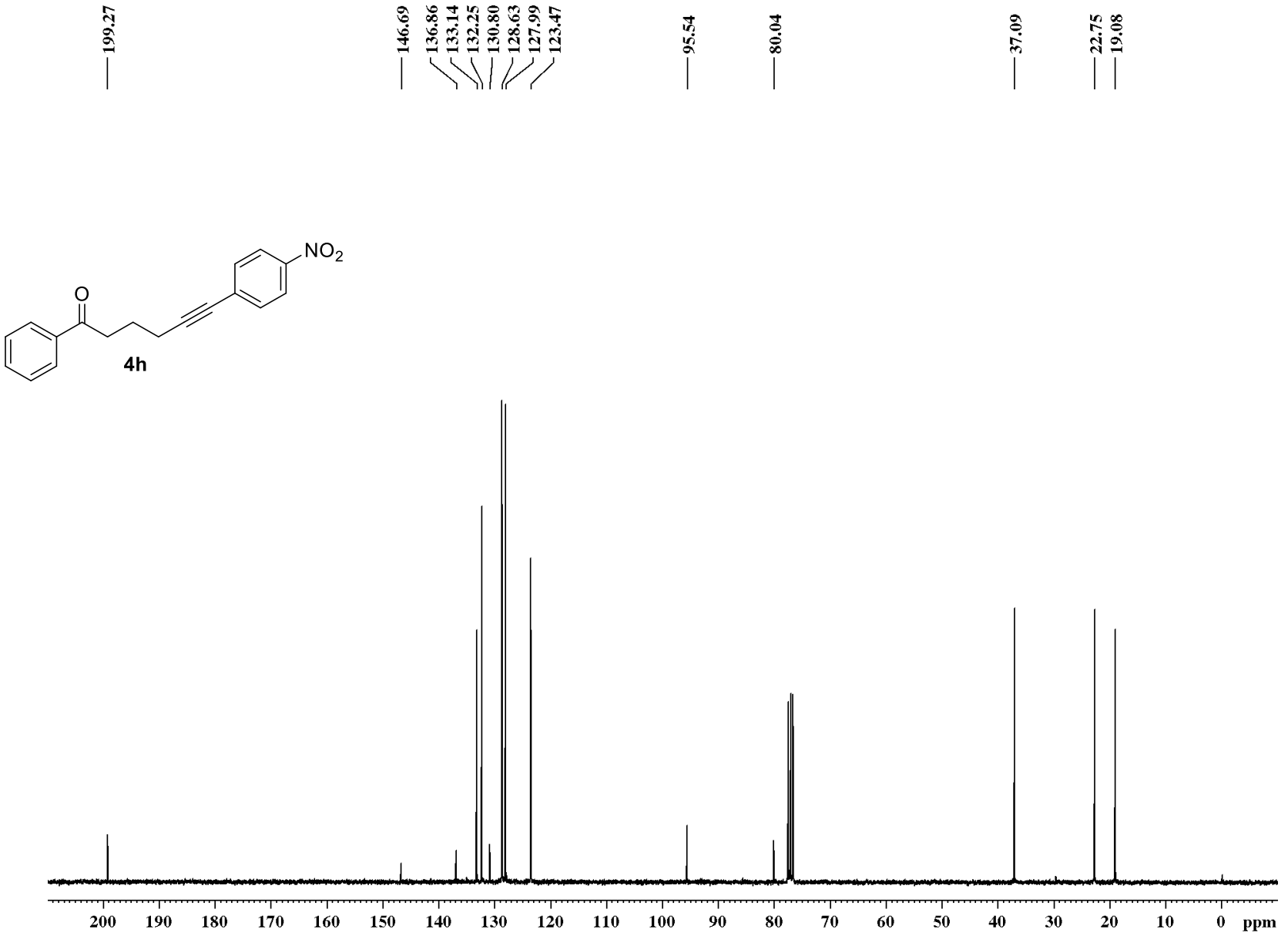
2.05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19.08
NO2
O
4h
200
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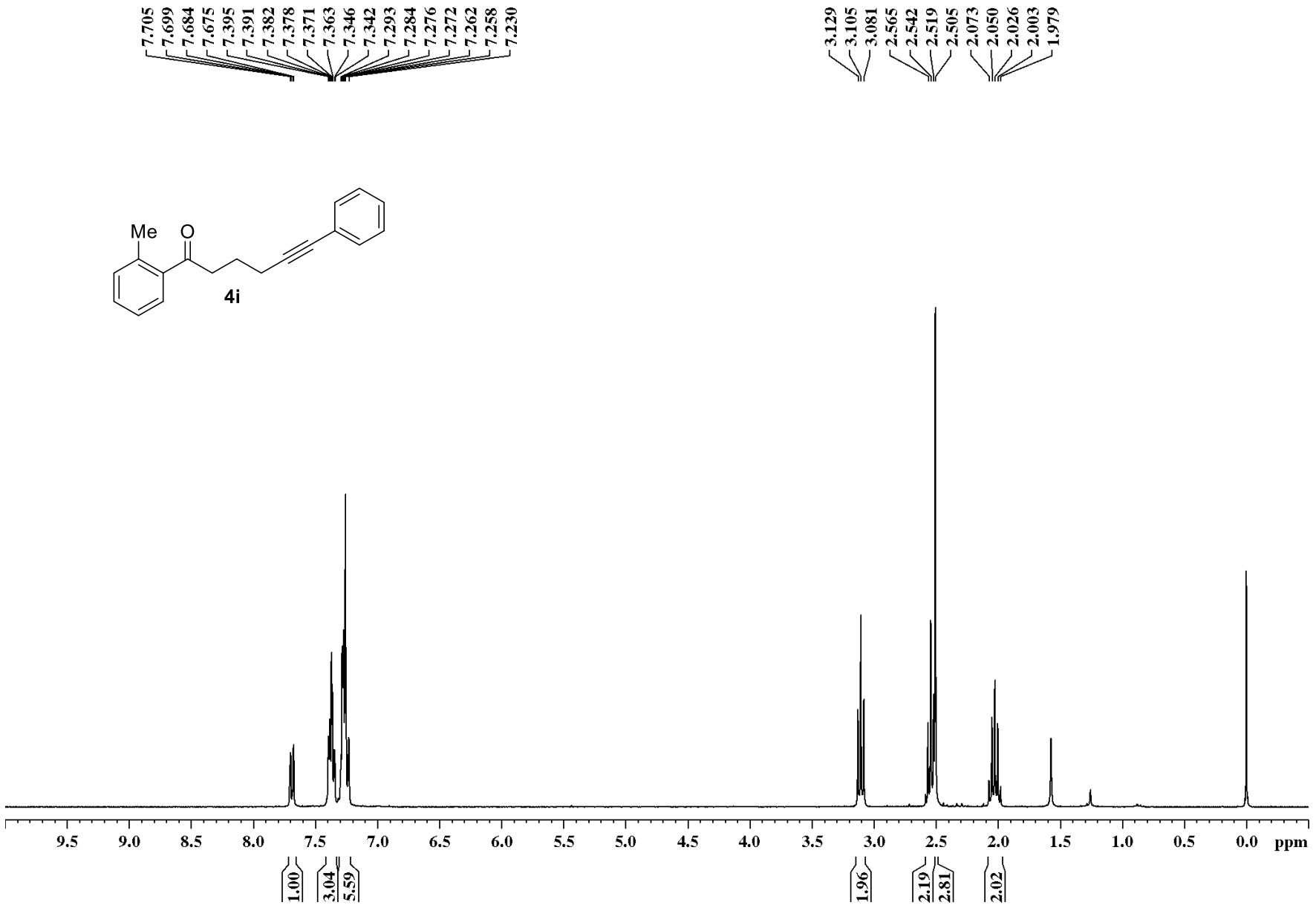
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7.272
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7.258
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3.081
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2.542
2.519
2.505
2.073
2.050
2.026
2.003
1.979
Me
O
4i
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8.0
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7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
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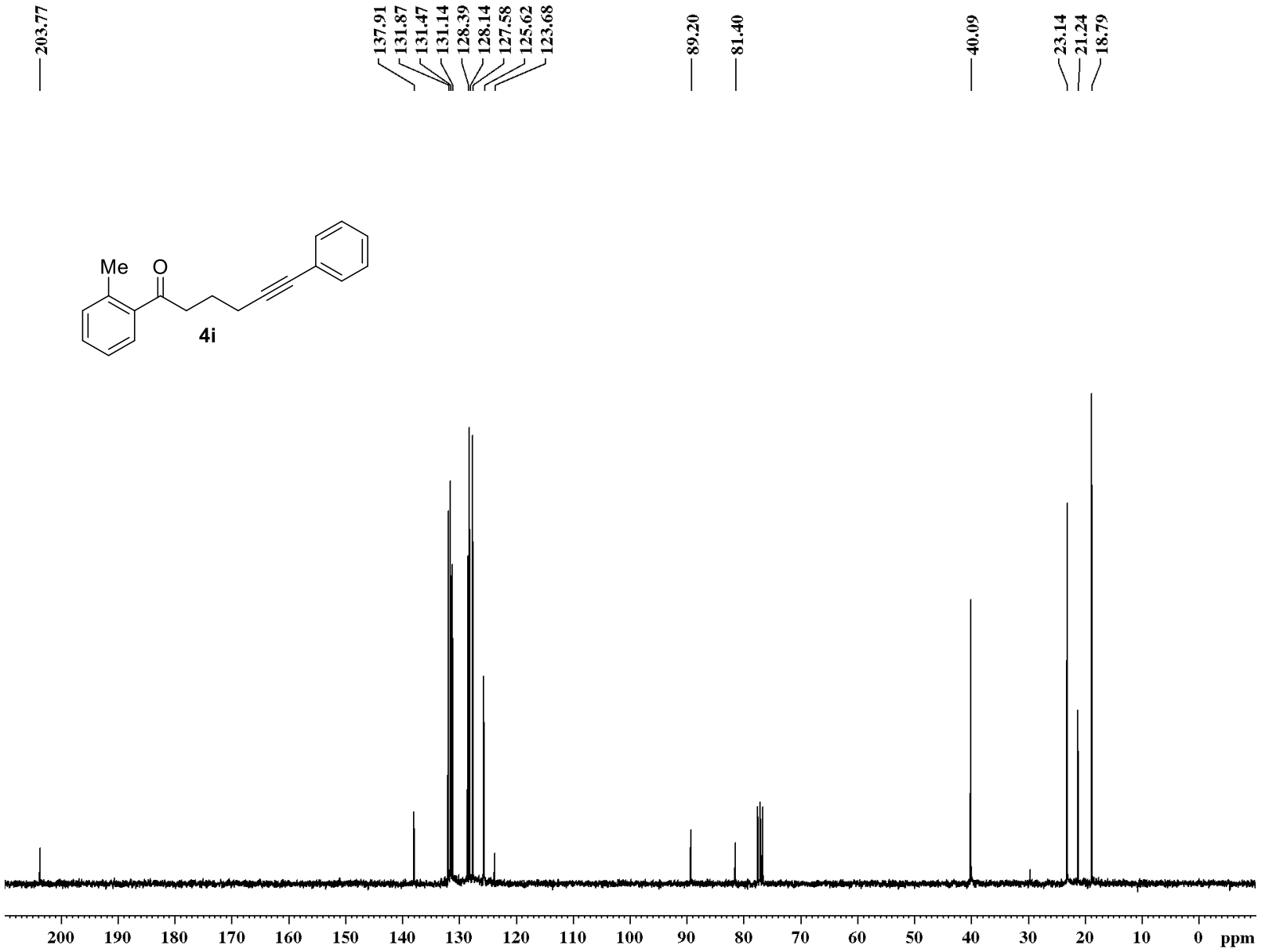
2.02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127.58
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123.68
89.20
81.40
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23.14
21.24
18.79
Me
O
4i
200
190
180
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140
130
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60
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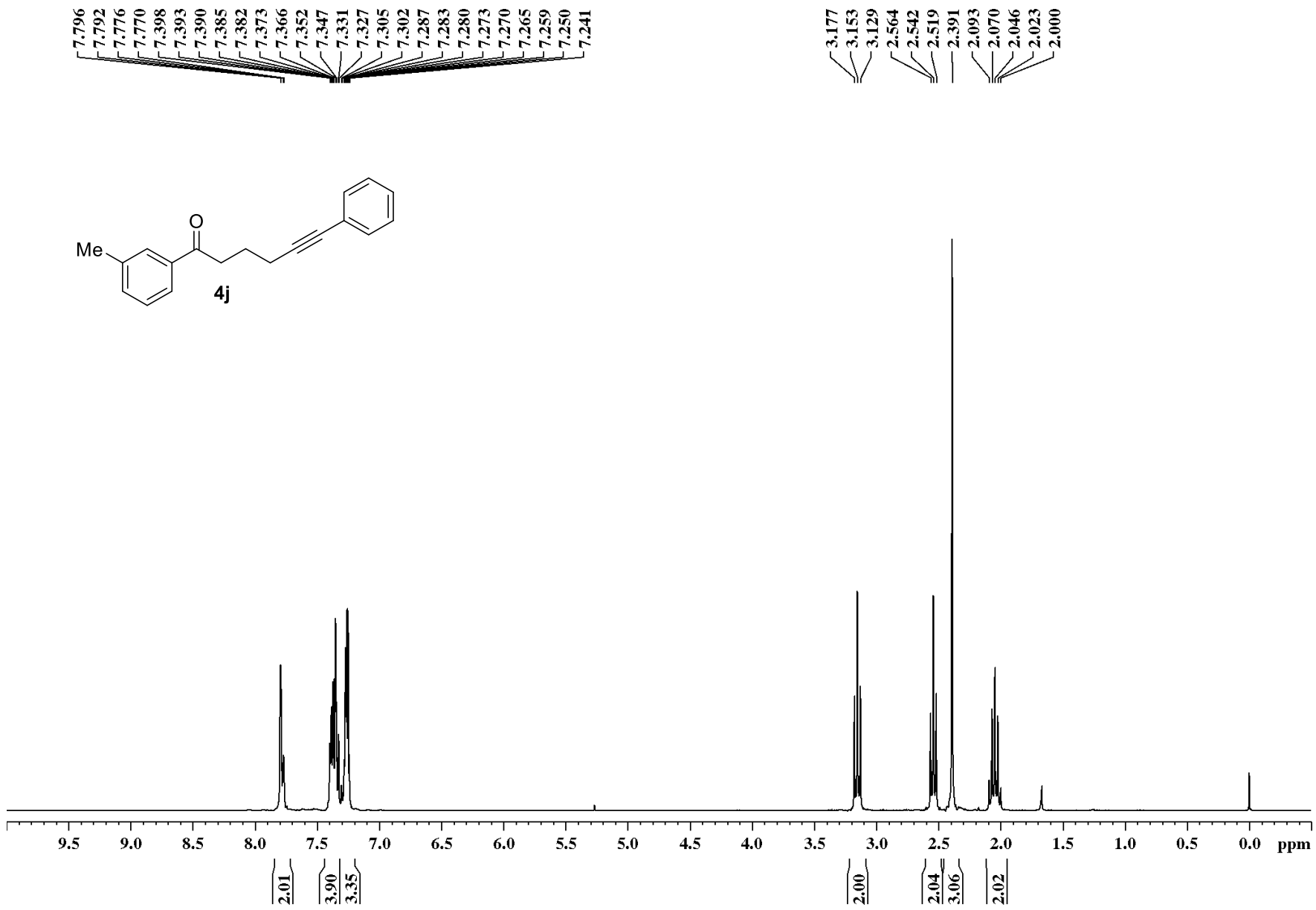
ppm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7.327
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7.287
7.283
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7.259
7.250
7.241
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3.129
2.564
2.542
2.519
2.391
2.093
2.070
2.046
2.023
2.000
O
Me
4j
9.5
9.0
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8.0
7.5
7.0
6.5
6.0
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5.0
4.5
4.0
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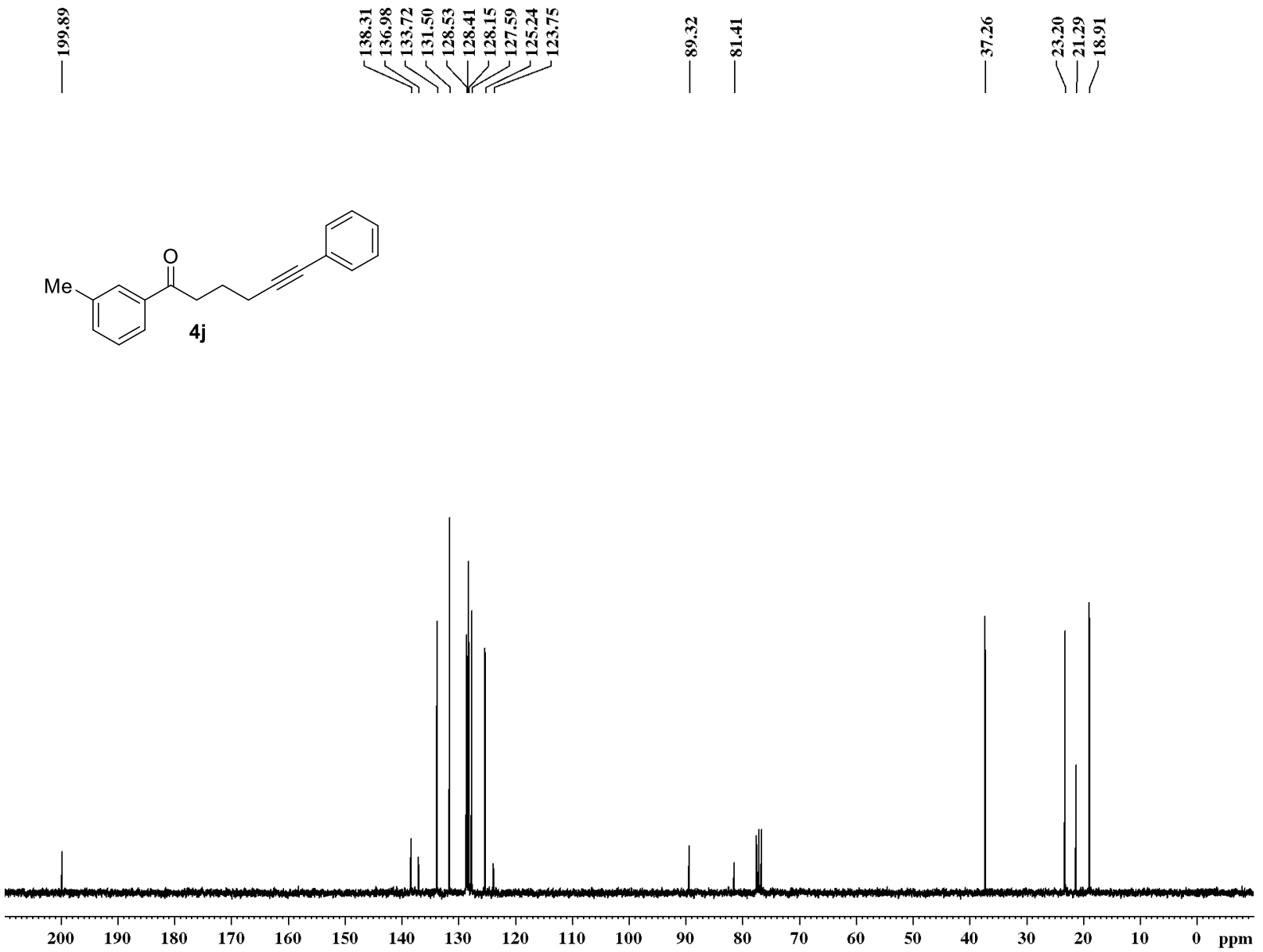
2.02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128.41
128.15
127.59
125.24
123.75
89.32
81.41
37.26
23.20
21.29
18.91
O
Me
4j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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30
20
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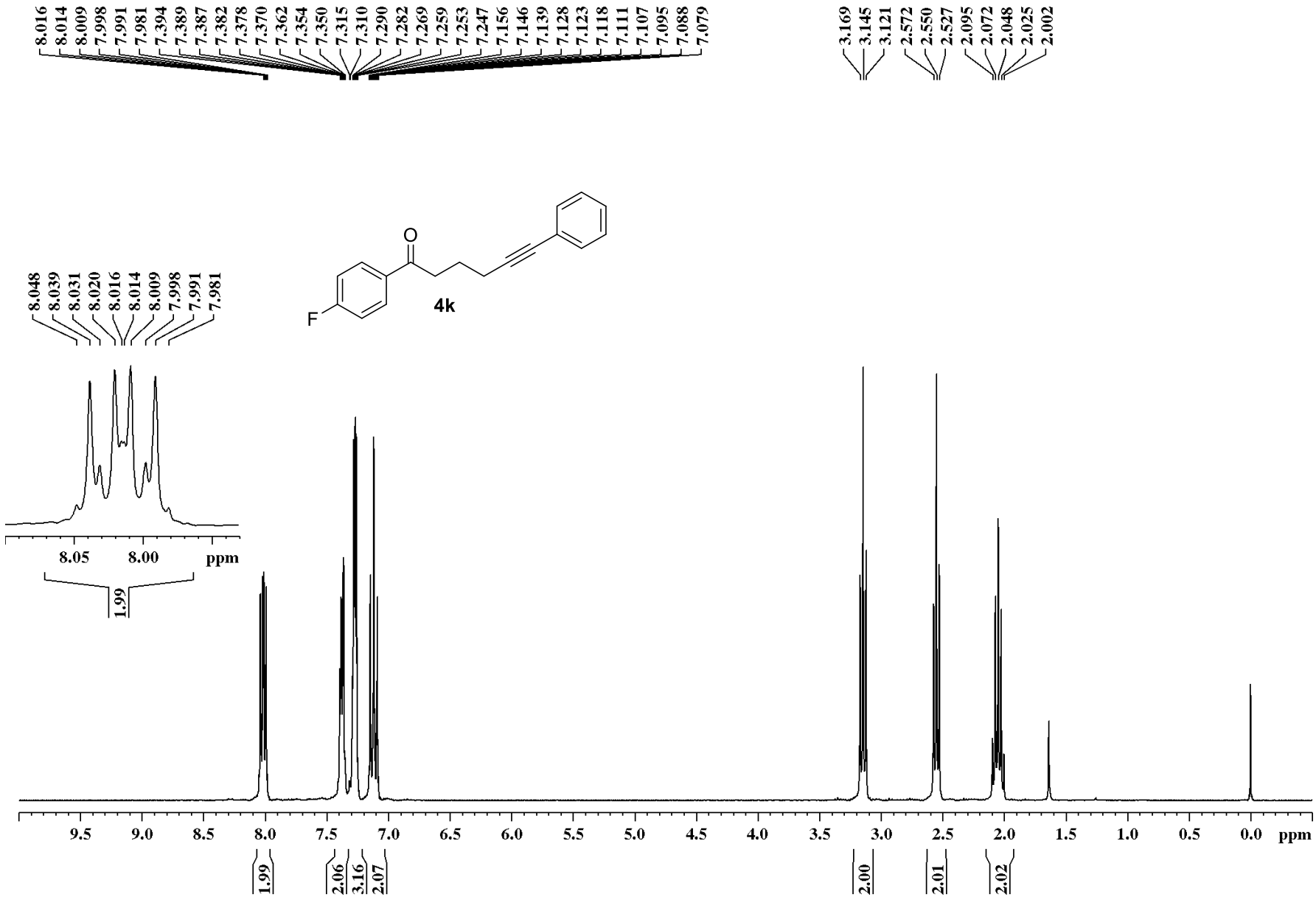
ppm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7.389
7.387
7.382
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7.370
7.362
7.354
7.350
7.315
7.310
7.290
7.282
7.269
7.259
7.253
7.247
7.156
7.146
7.139
7.128
7.123
7.118
7.111
7.107
7.095
7.088
7.079
3.169
3.145
3.121
2.572
2.550
2.527
2.095
2.072
2.048
2.025
2.002
O
F
4k
8.048
8.039
8.031
8.020
8.016
8.014
8.009
7.998
7.991
7.981
8.05
8.00
ppm
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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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0.5
0.0
ppm
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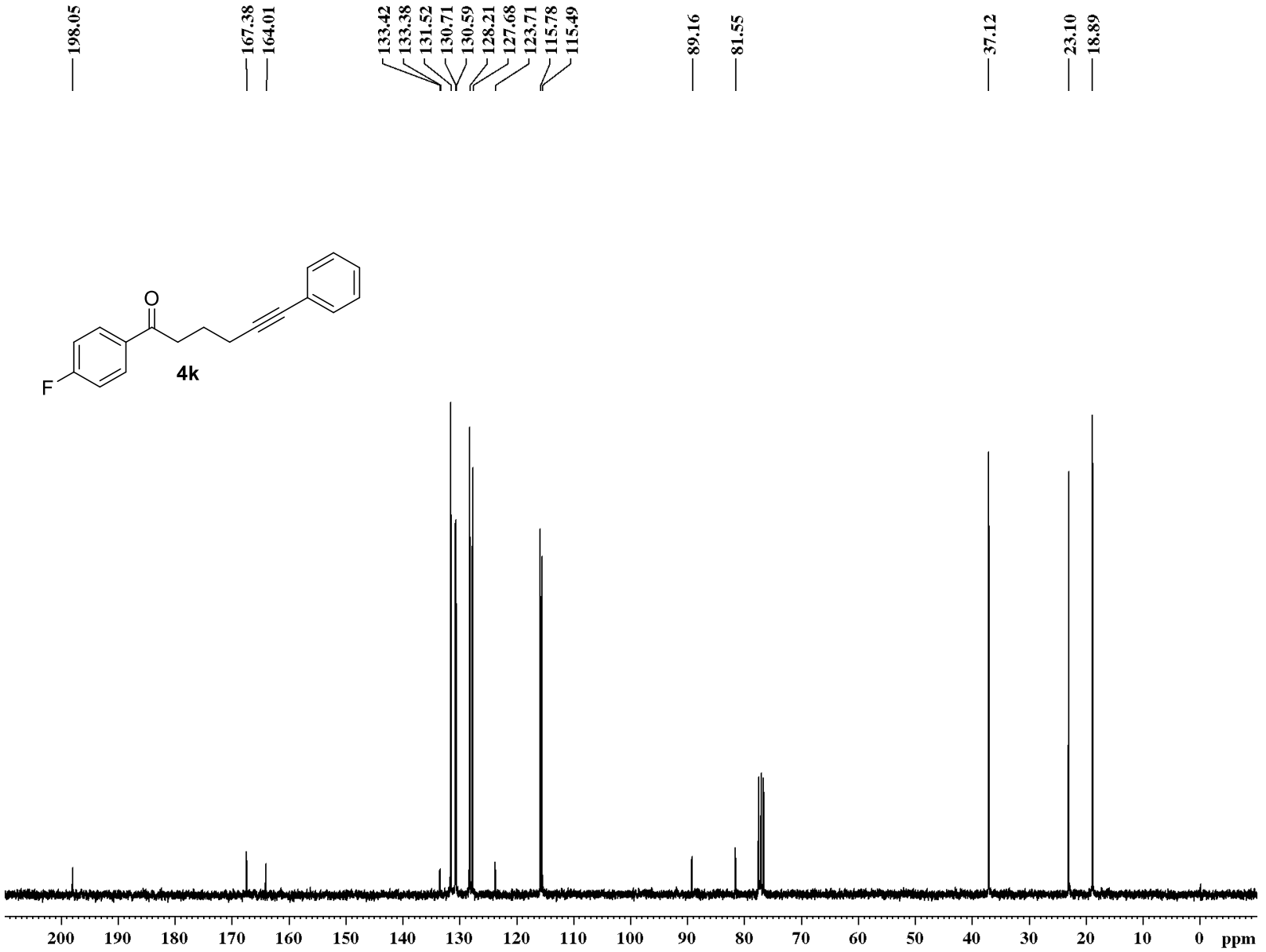
2.06
3.16
2.07
2.00
2.01
2.02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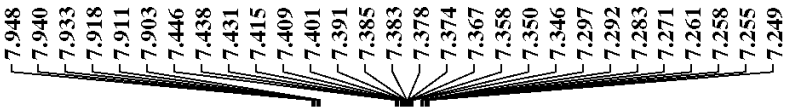
133.38
131.52
130.71
130.59
128.21
127.68
123.71
115.78
115.49
89.16
81.55
37.12
23.10
18.89
O
F
4k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.401
7.391
7.385
7.383
7.378
7.374
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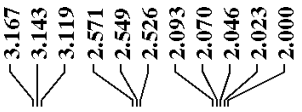
7.255
7.249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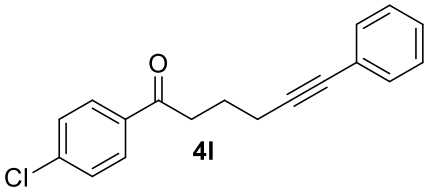
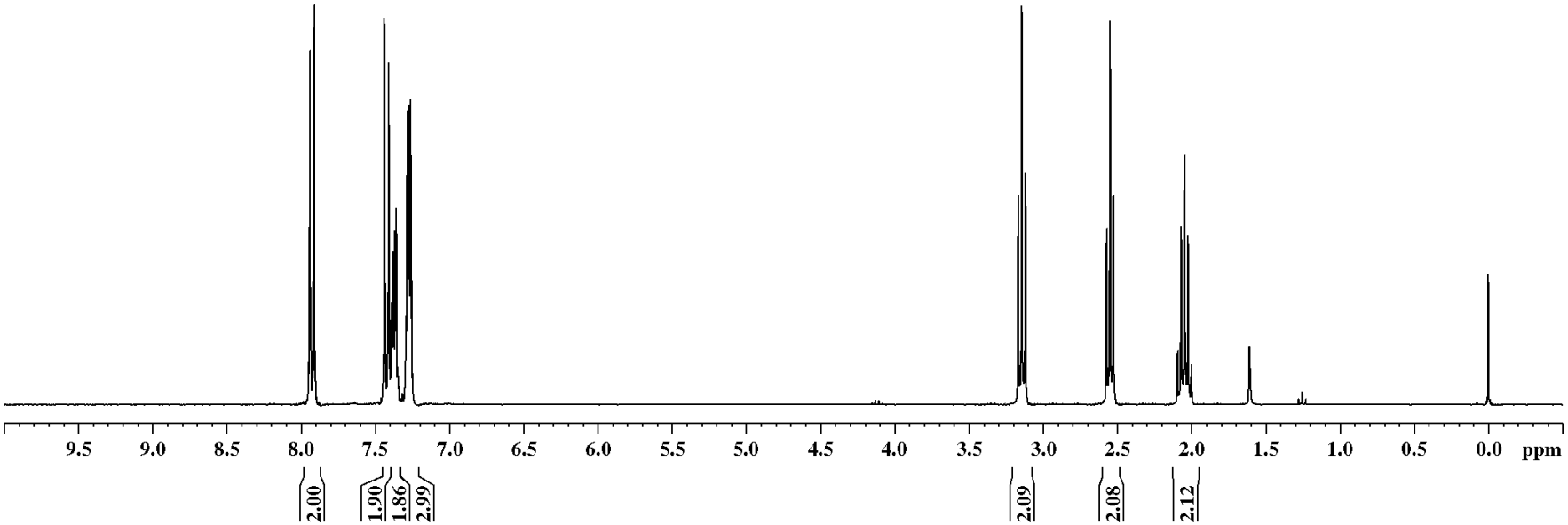
2.000


O
Cl

4l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6.0
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5.0
4.5
4.0
3.5
3.0
2.5
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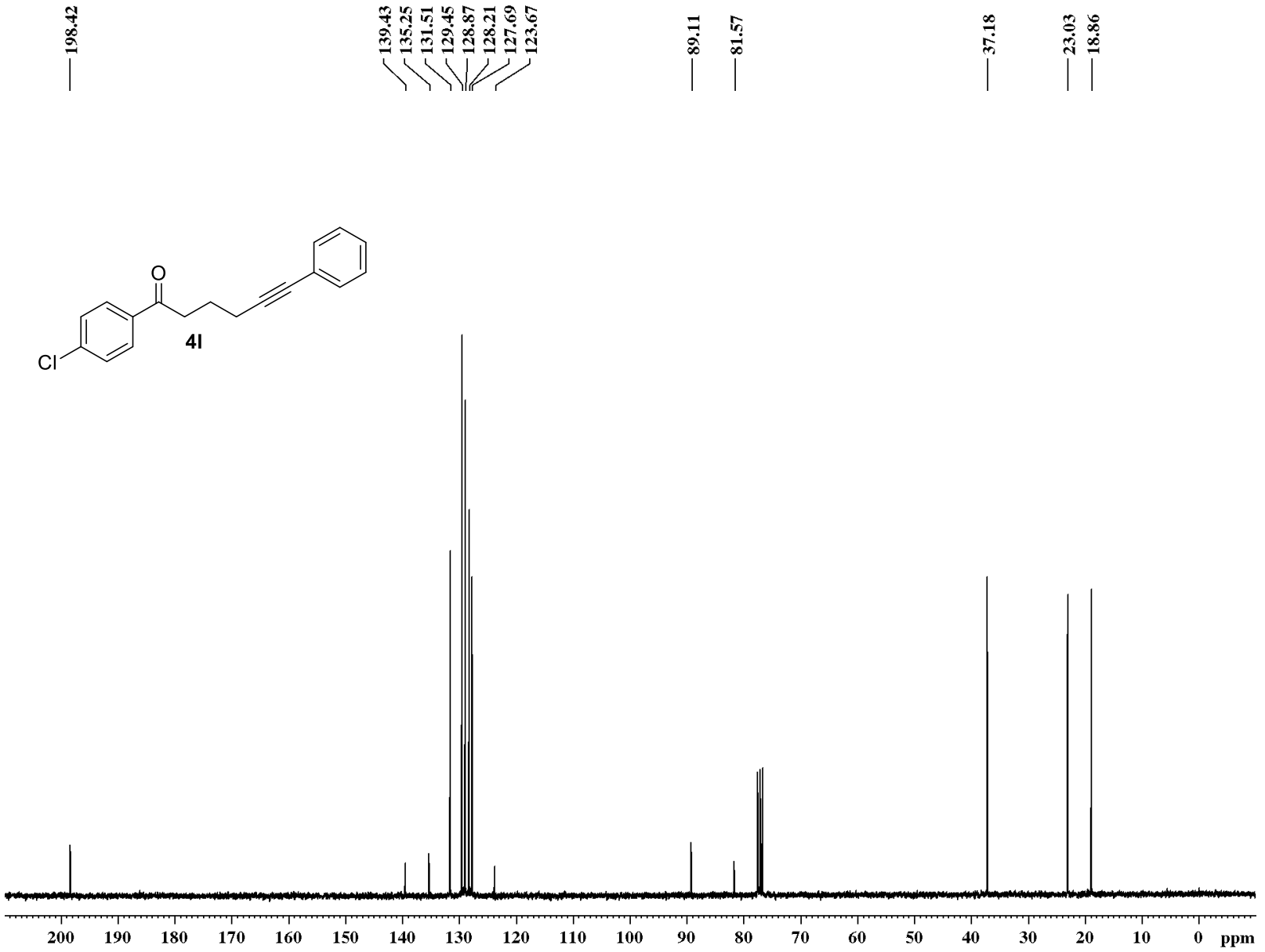
2.08
2.12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127.69
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89.11
81.57
37.18
23.03
18.86
O
Cl
4l
200
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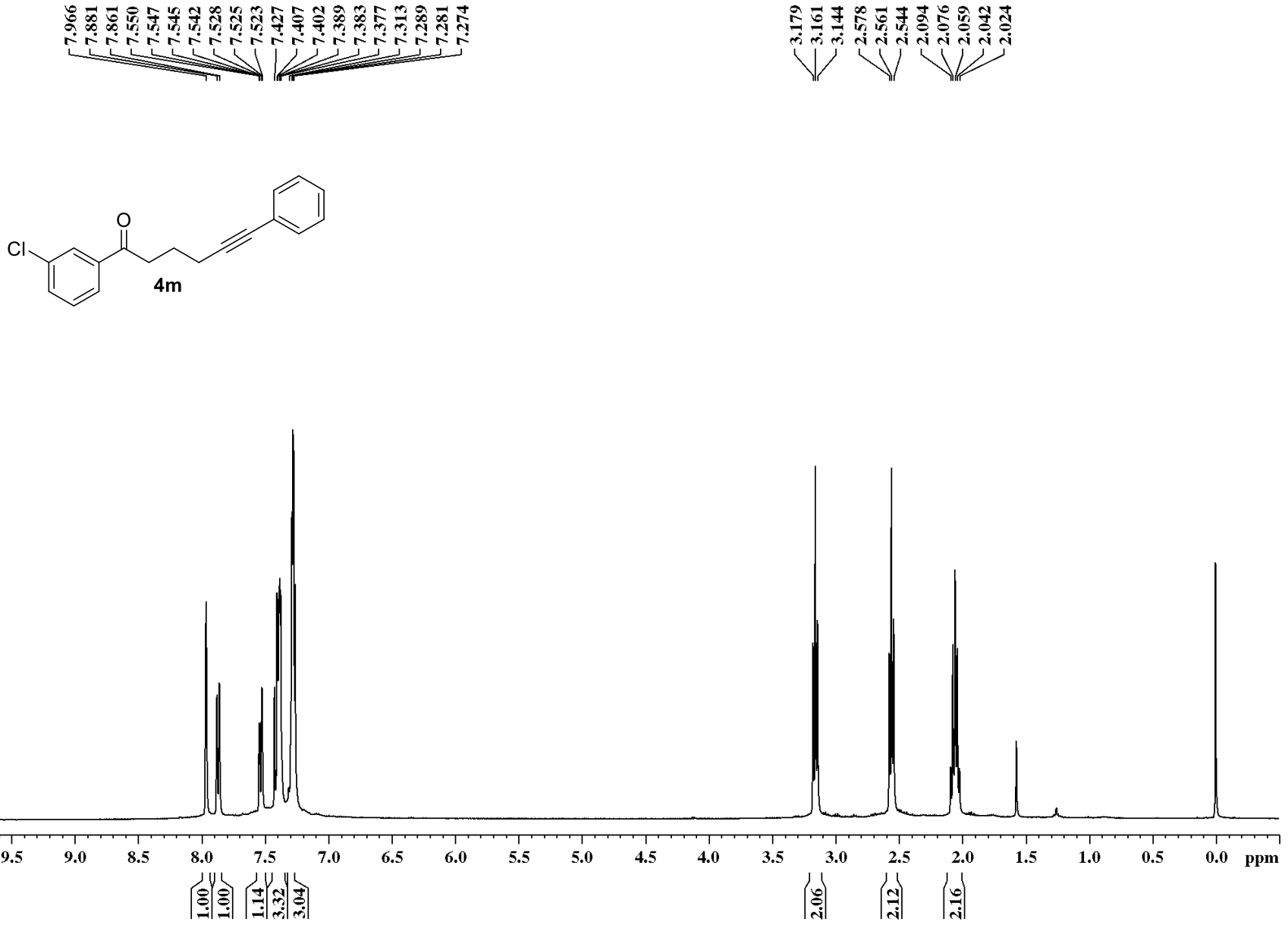
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.525
7.523
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7.402
7.389
7.383
7.377
7.313
7.289
7.281
7.274
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3.161
3.144
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2.561
2.544
2.094
2.076
2.059
2.042
2.024
O
Cl
4m
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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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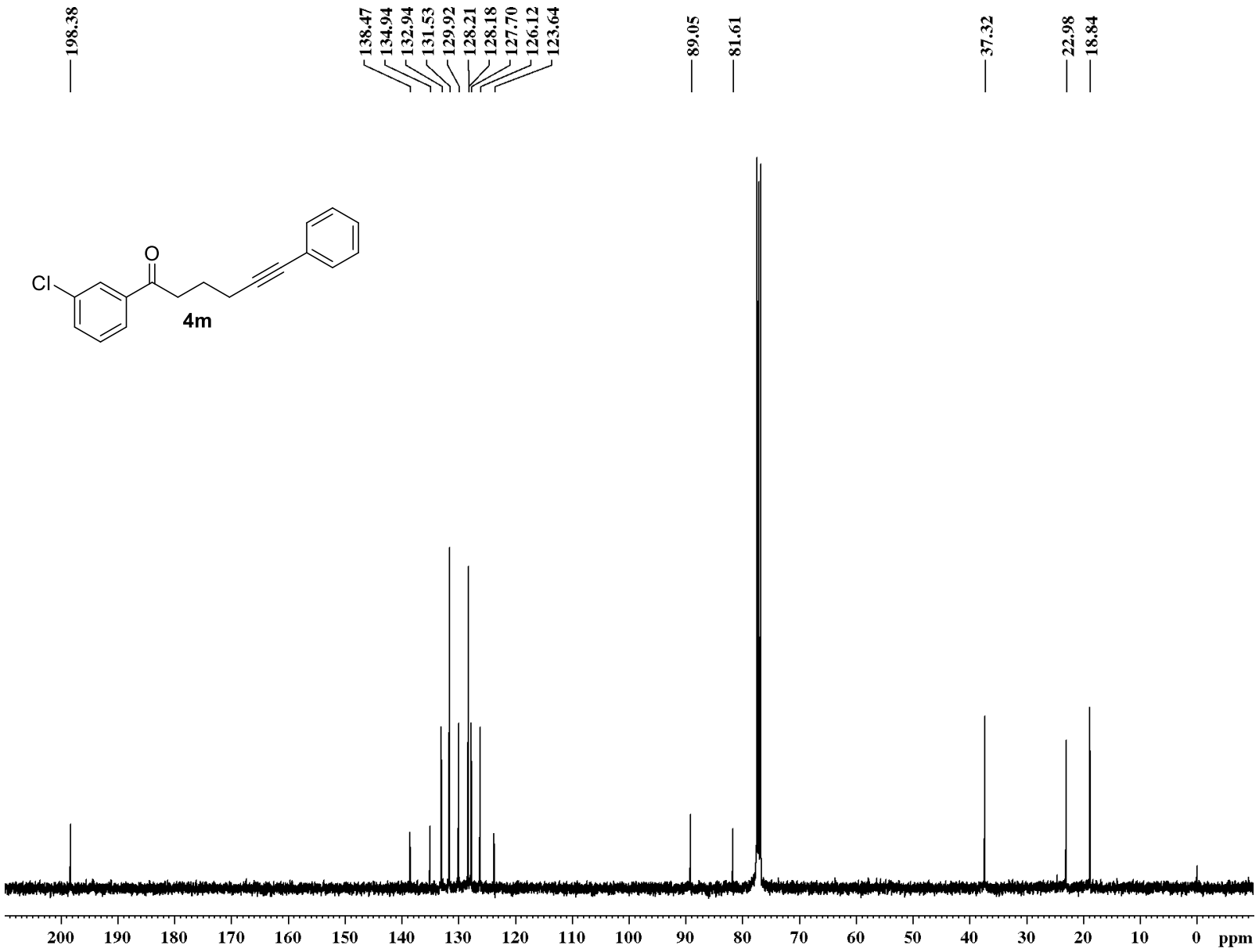
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81.61
37.32
22.98
18.84
Cl
O
4m
200
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120
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100
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10
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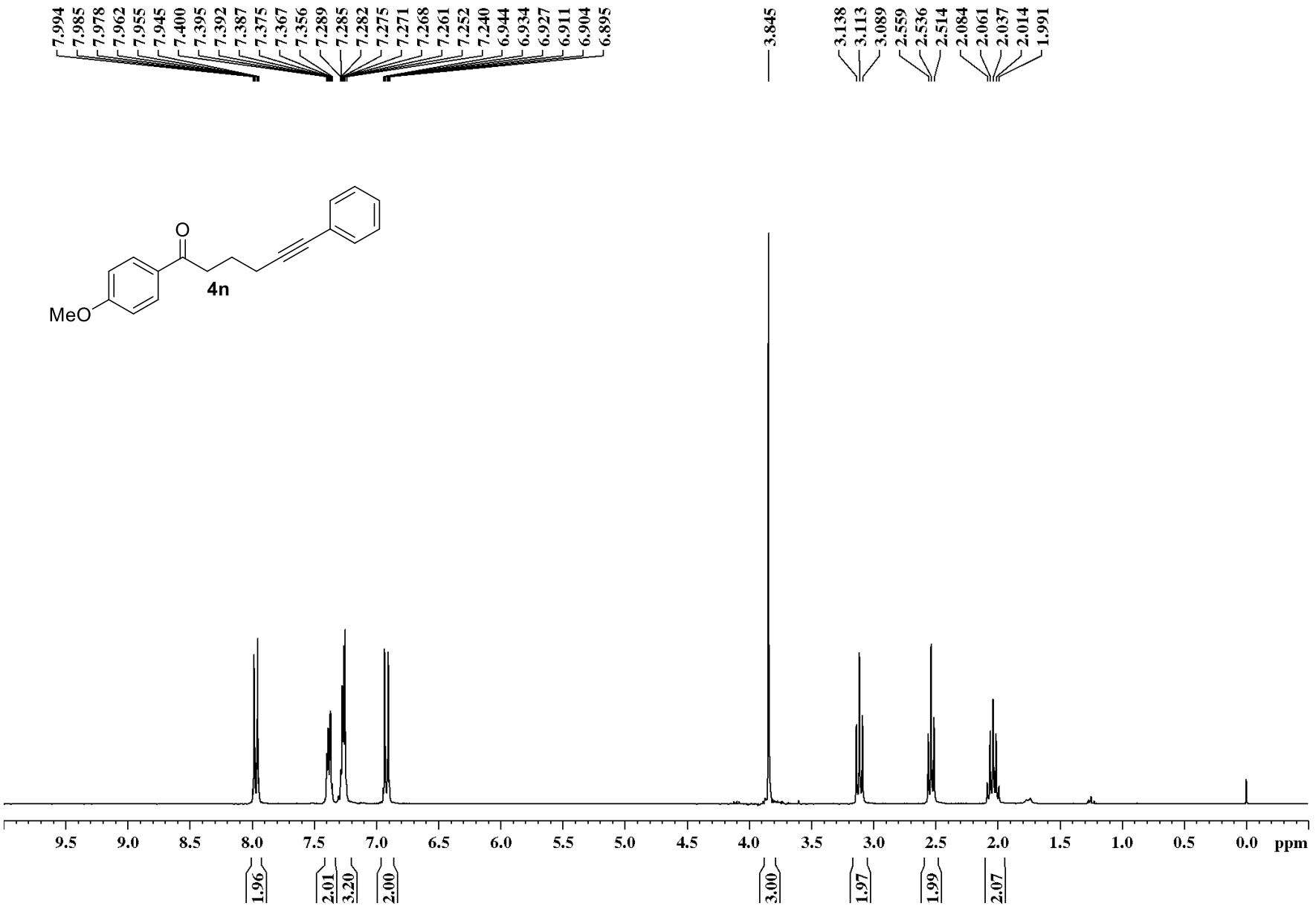
ppm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2.536
2.514
2.084
2.061
2.037
2.014
1.991
O
MeO
4n
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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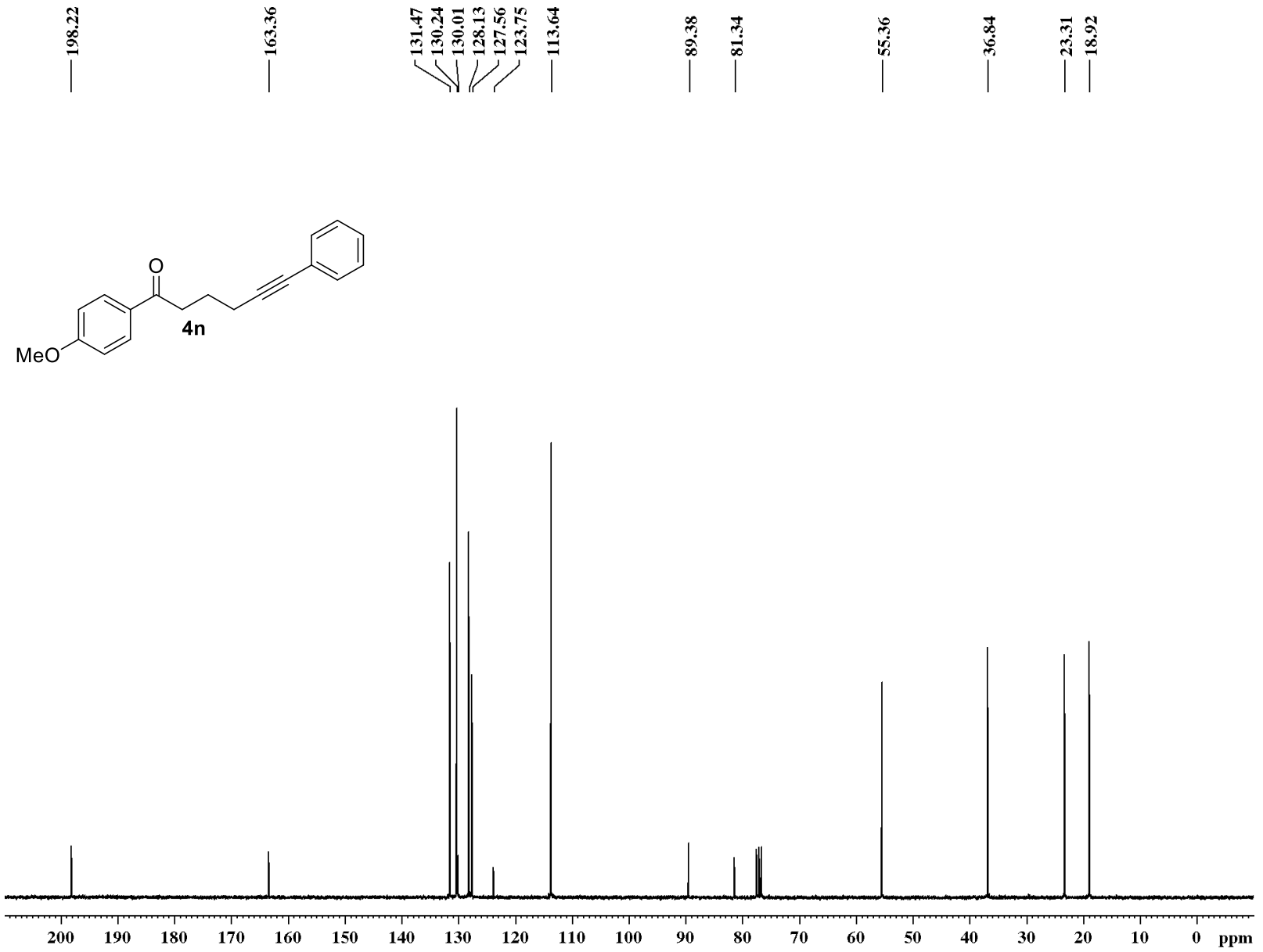
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MeO
4n
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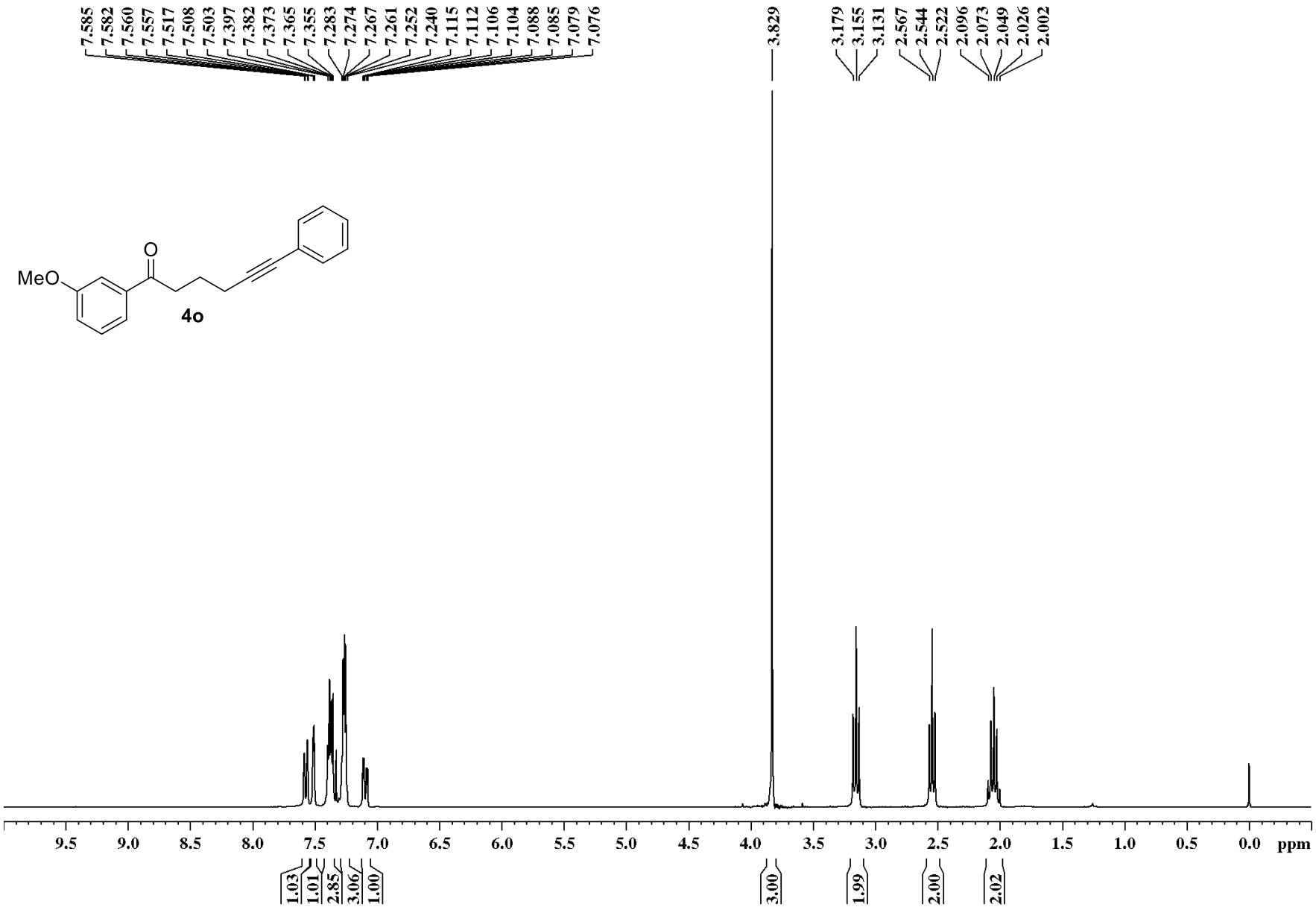
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7.252
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7.104
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7.085
7.079
7.076
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3.155
3.131
2.567
2.544
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2.096
2.073
2.049
2.026
2.002
MeO
O
4o
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9.0
8.5
8.0
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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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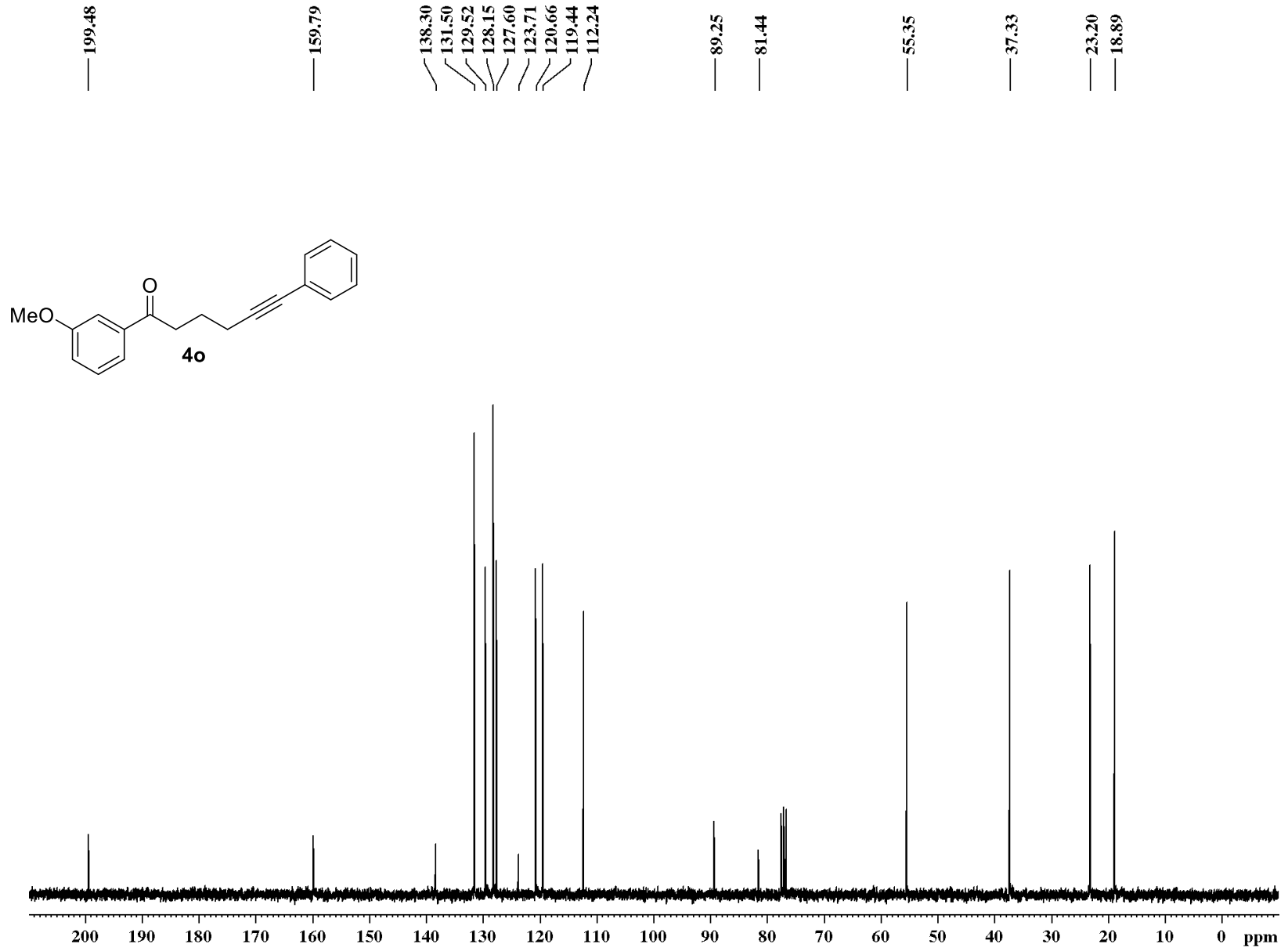
2.02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55.35
37.33
23.20
18.89
MeO
O
4o
200
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180
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140
130
120
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90
80
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60
50
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30
20
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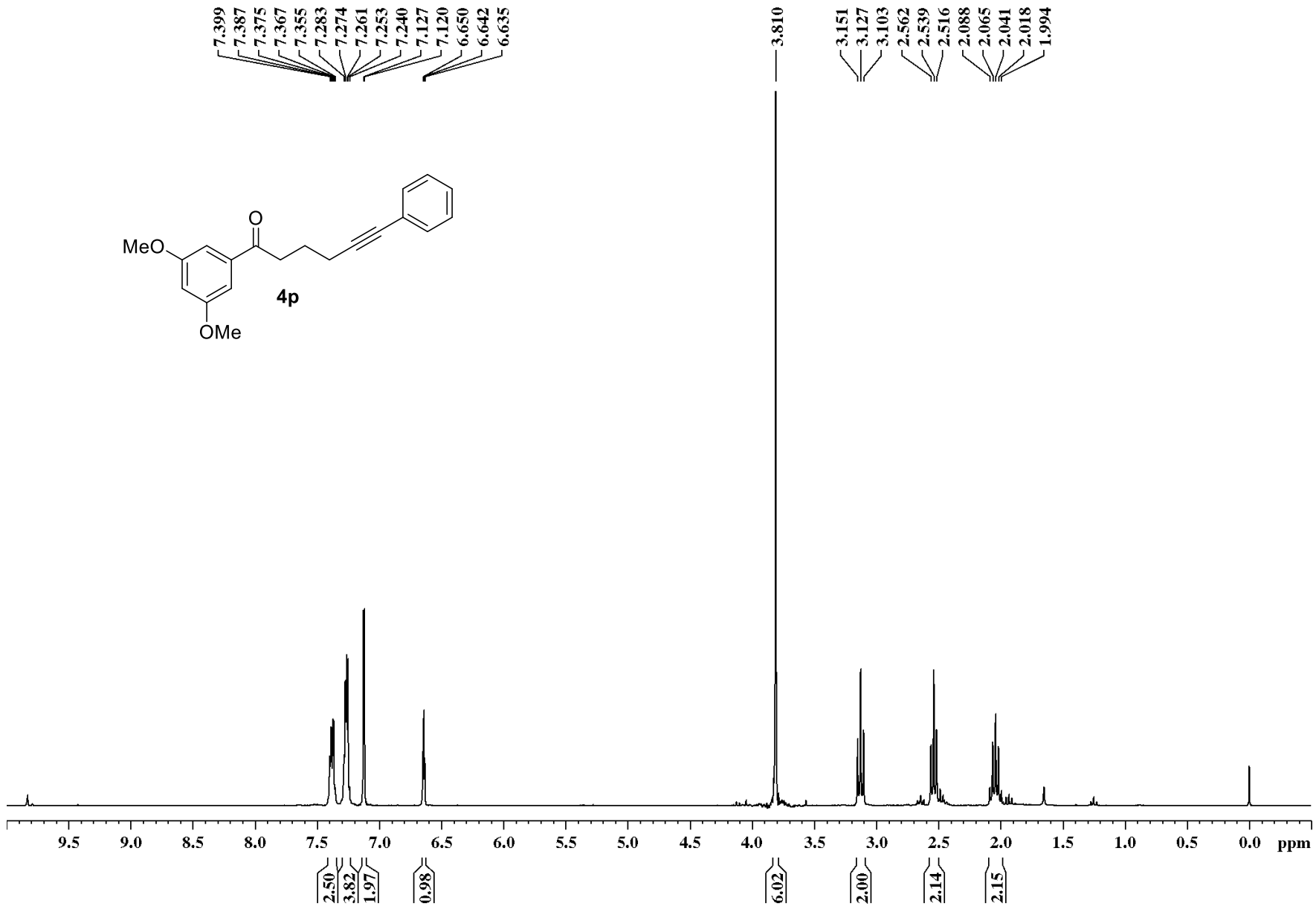
0
ppm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3.127
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2.018
1.994
MeO
O
OMe
4p
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8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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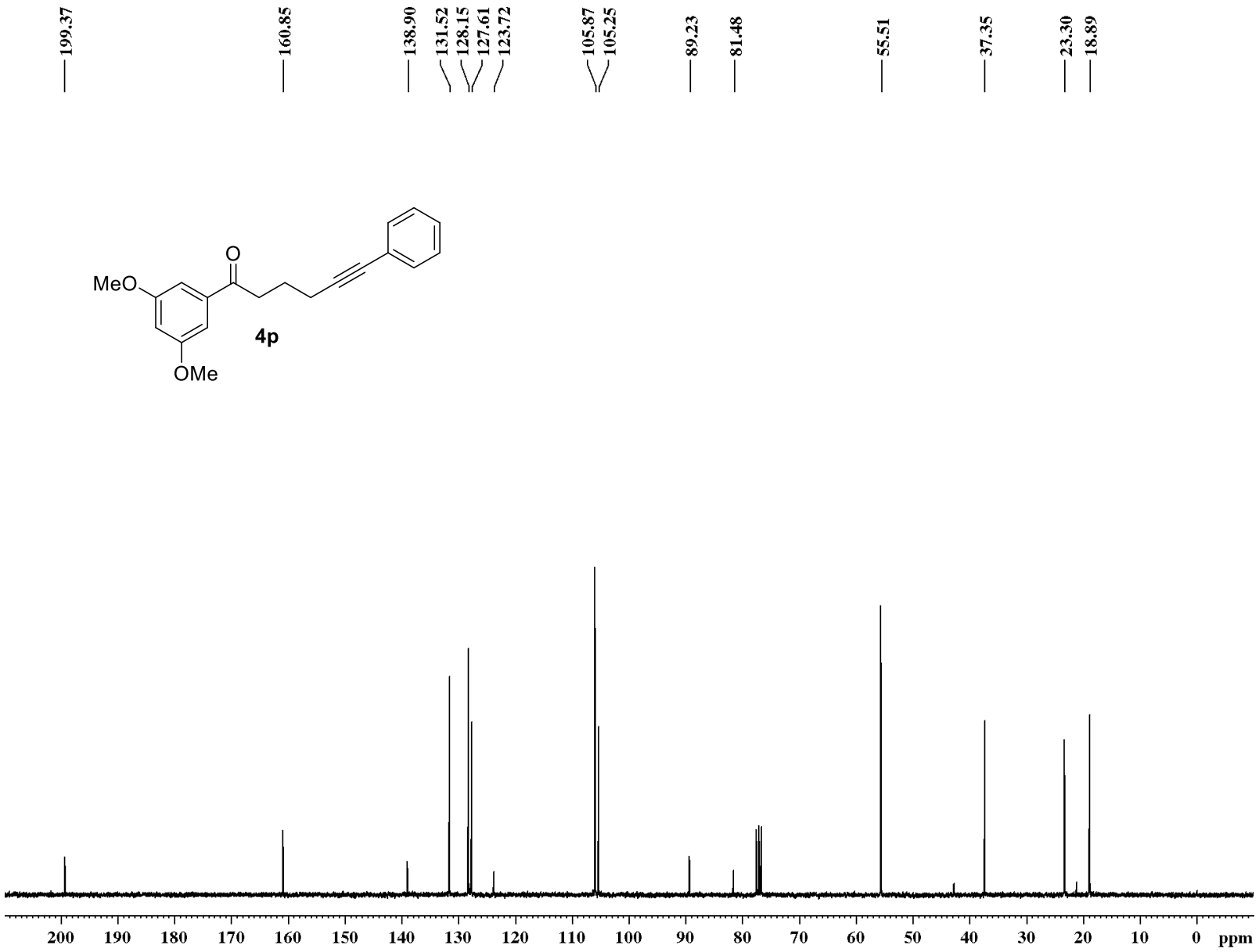
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89.23
81.48
55.51
37.35
23.30
18.89
MeO
O
OMe
4p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
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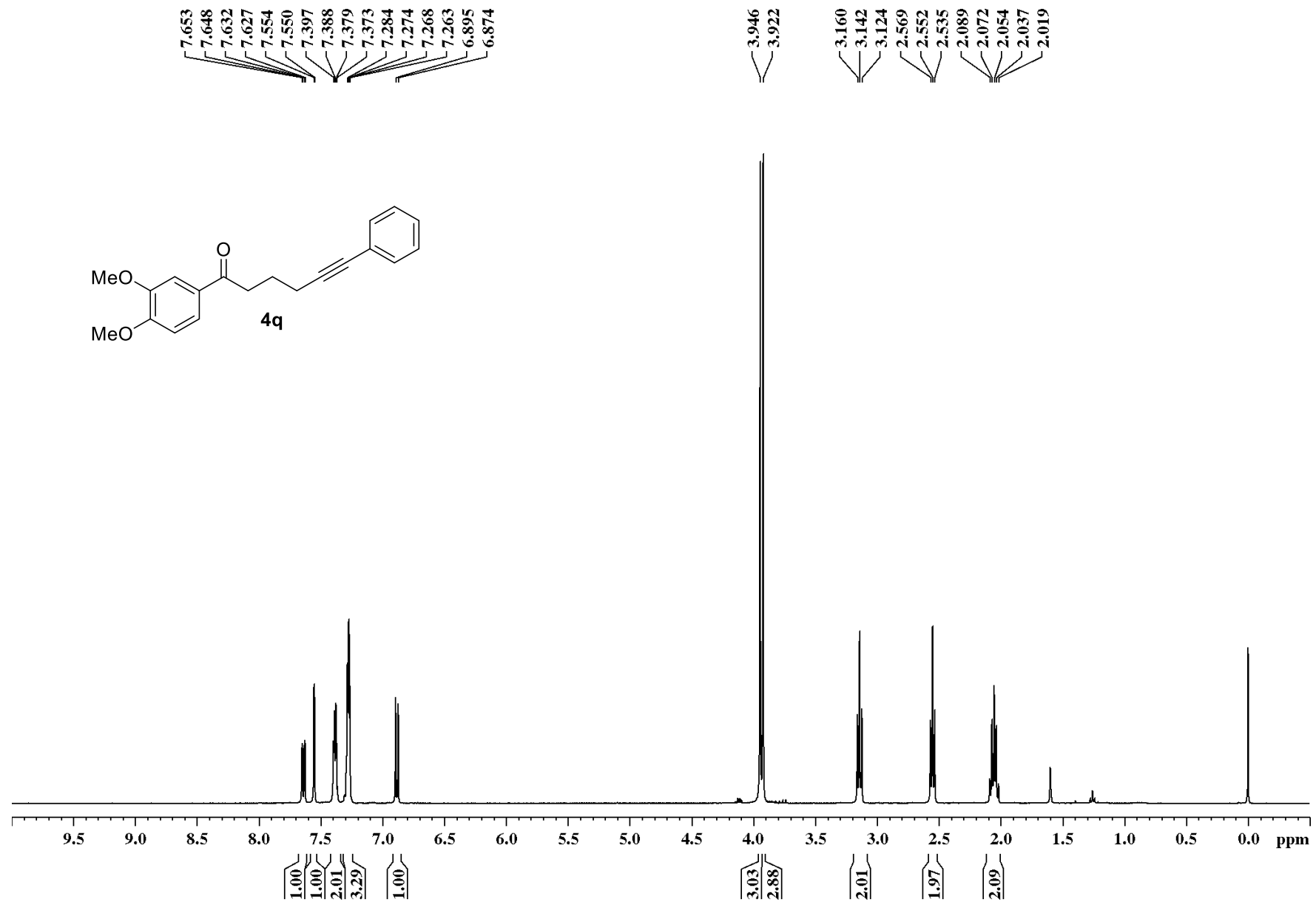
0
ppm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6.874
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3.922
3.160
3.142
3.124
2.569
2.552
2.535
2.089
2.072
2.054
2.037
2.019
MeO
MeO
O
4q
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9.0
8.5
8.0
7.5
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6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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2.0
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1.0
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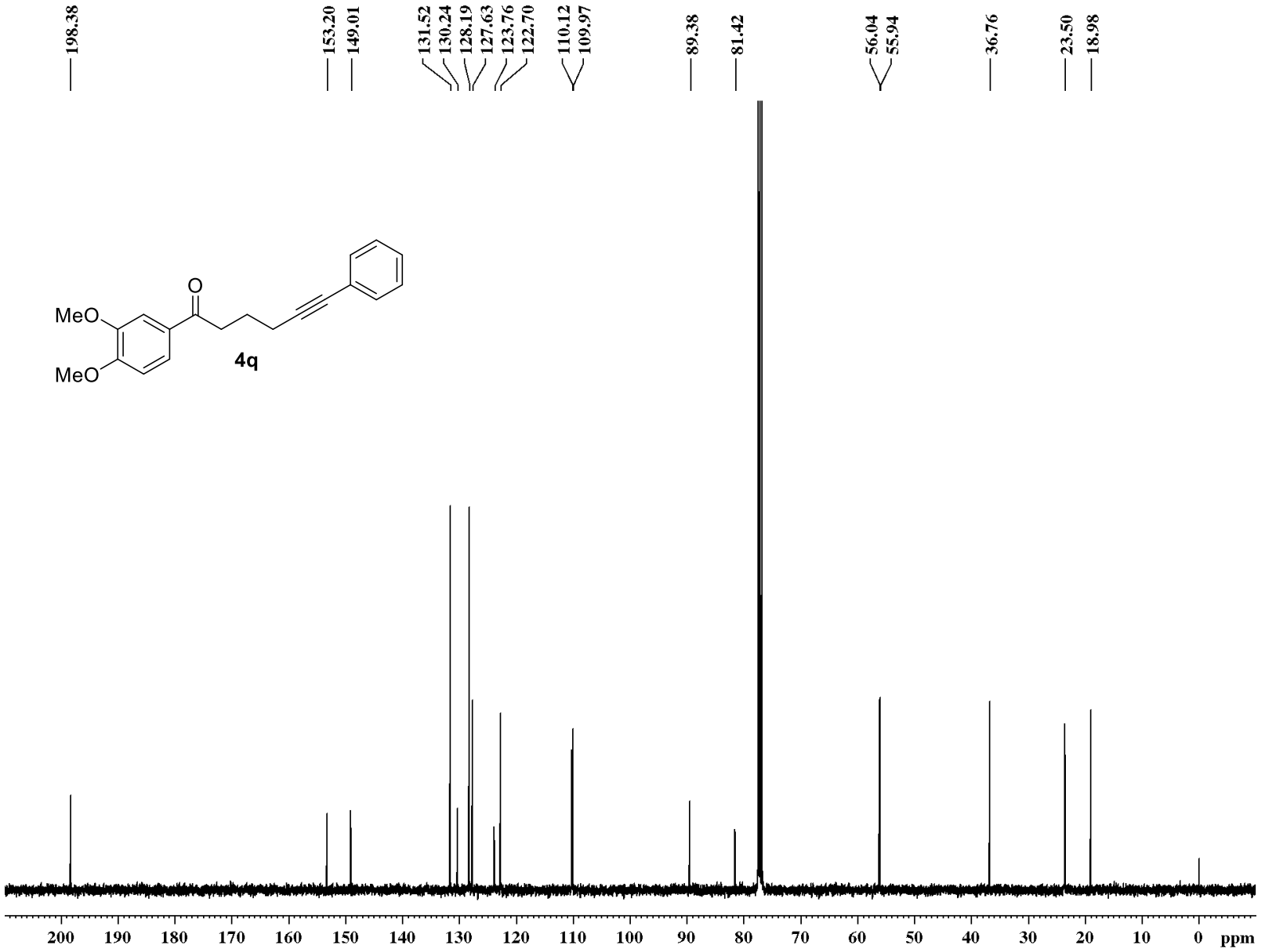
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81.42
56.04
55.94
36.76
23.50
18.98
MeO
MeO
O
4q
200
190
180
170
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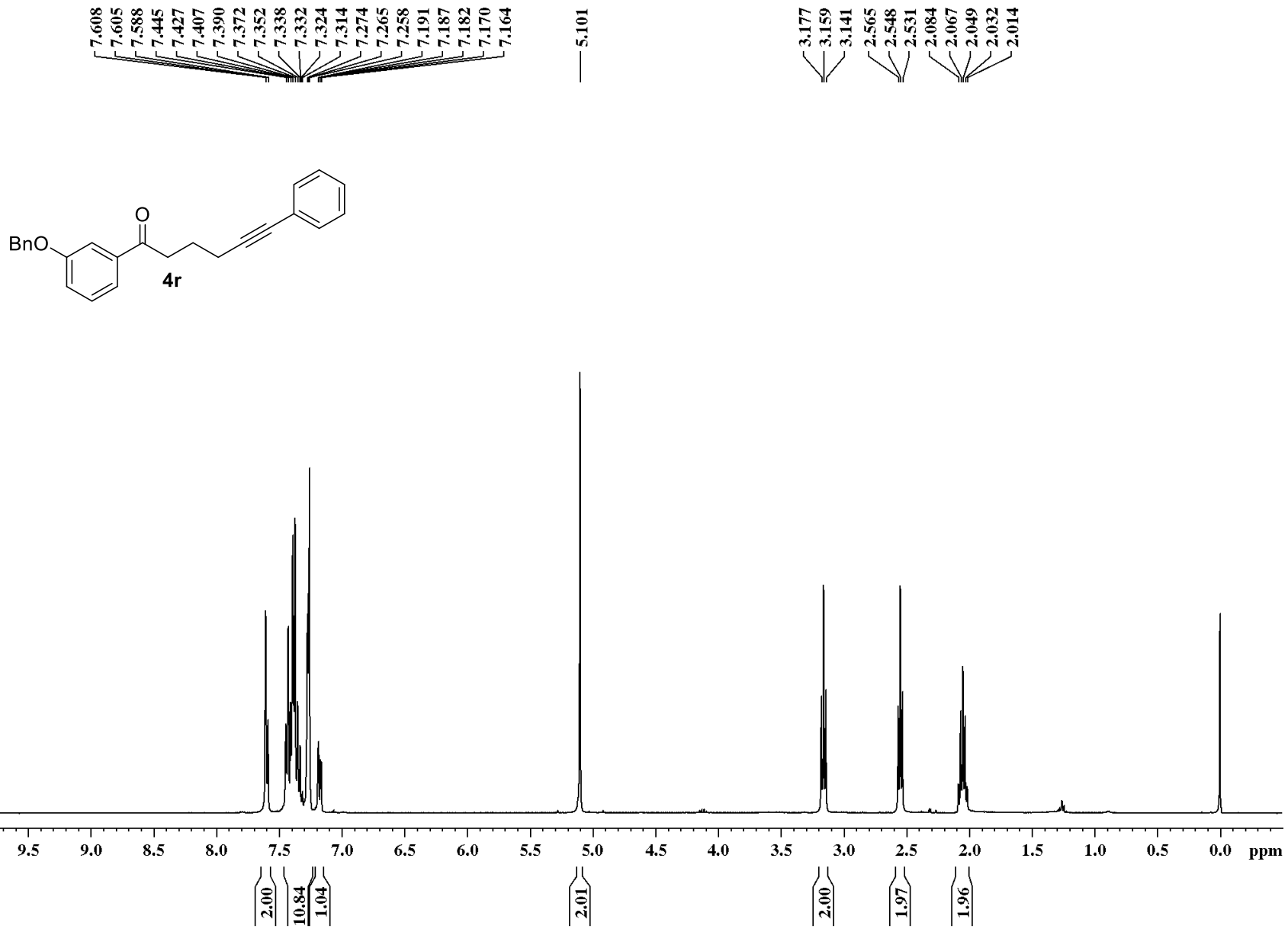
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2.032
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BnO
O
4r
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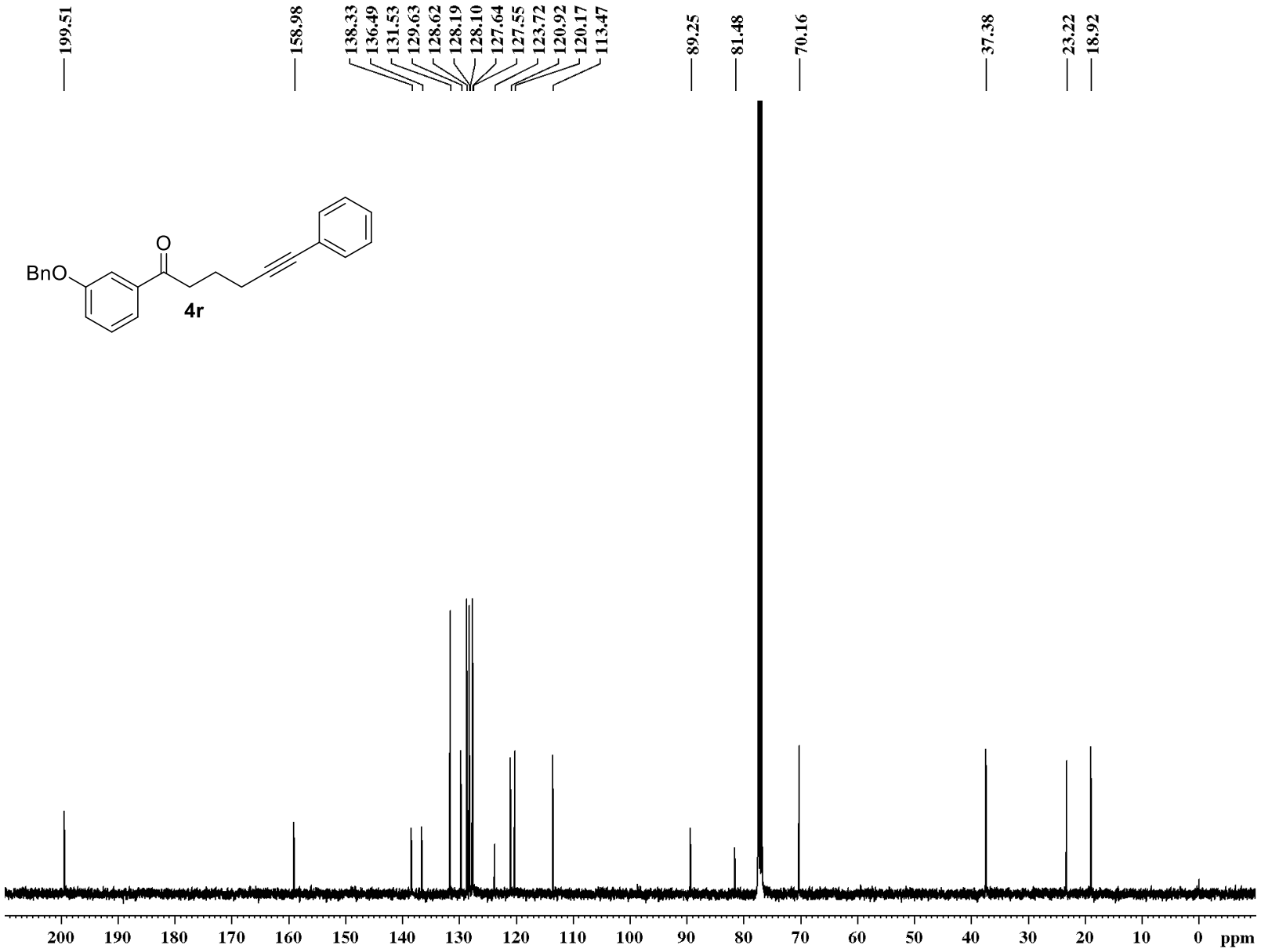
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23.22
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BnO
O
4r
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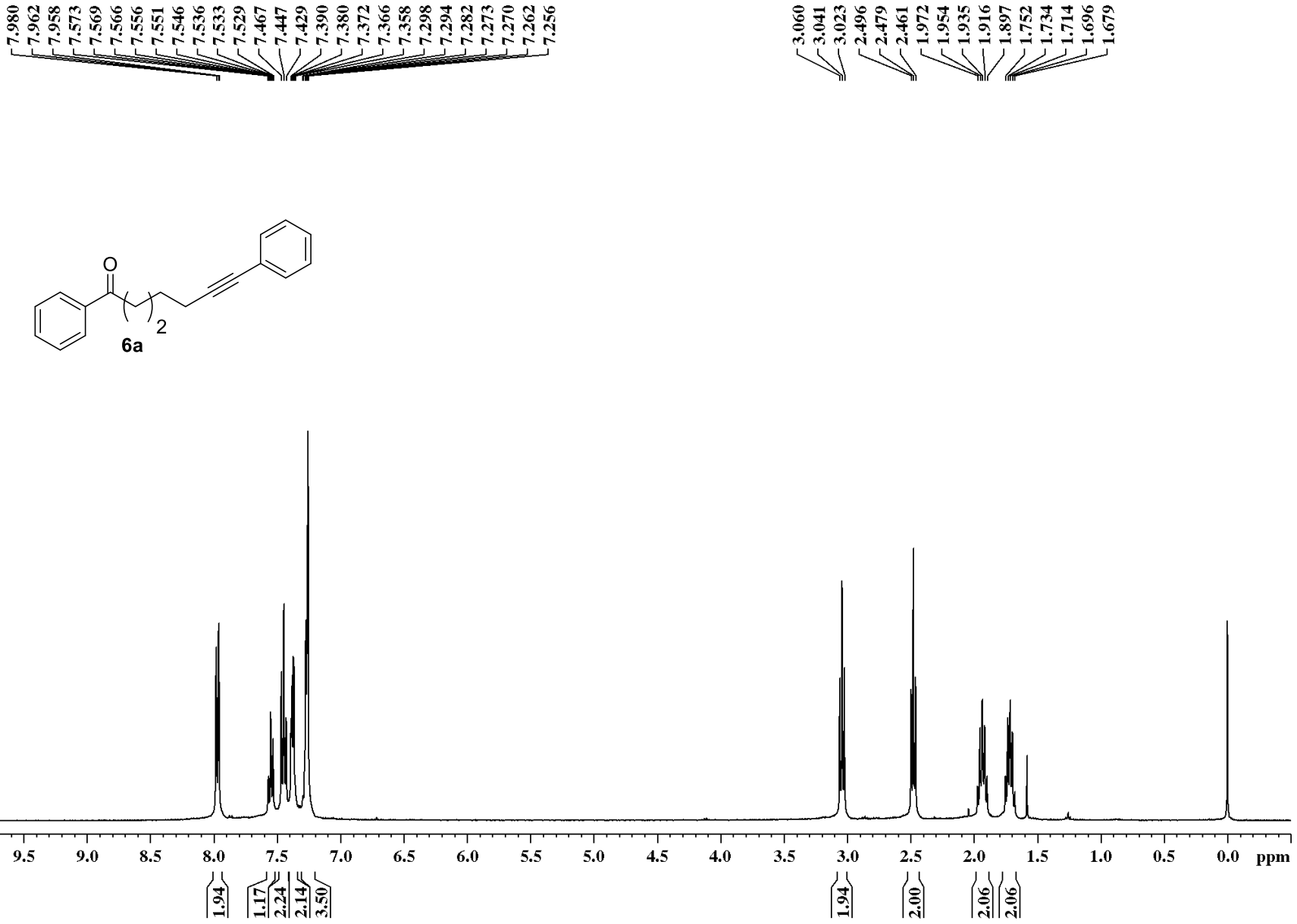
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O
2
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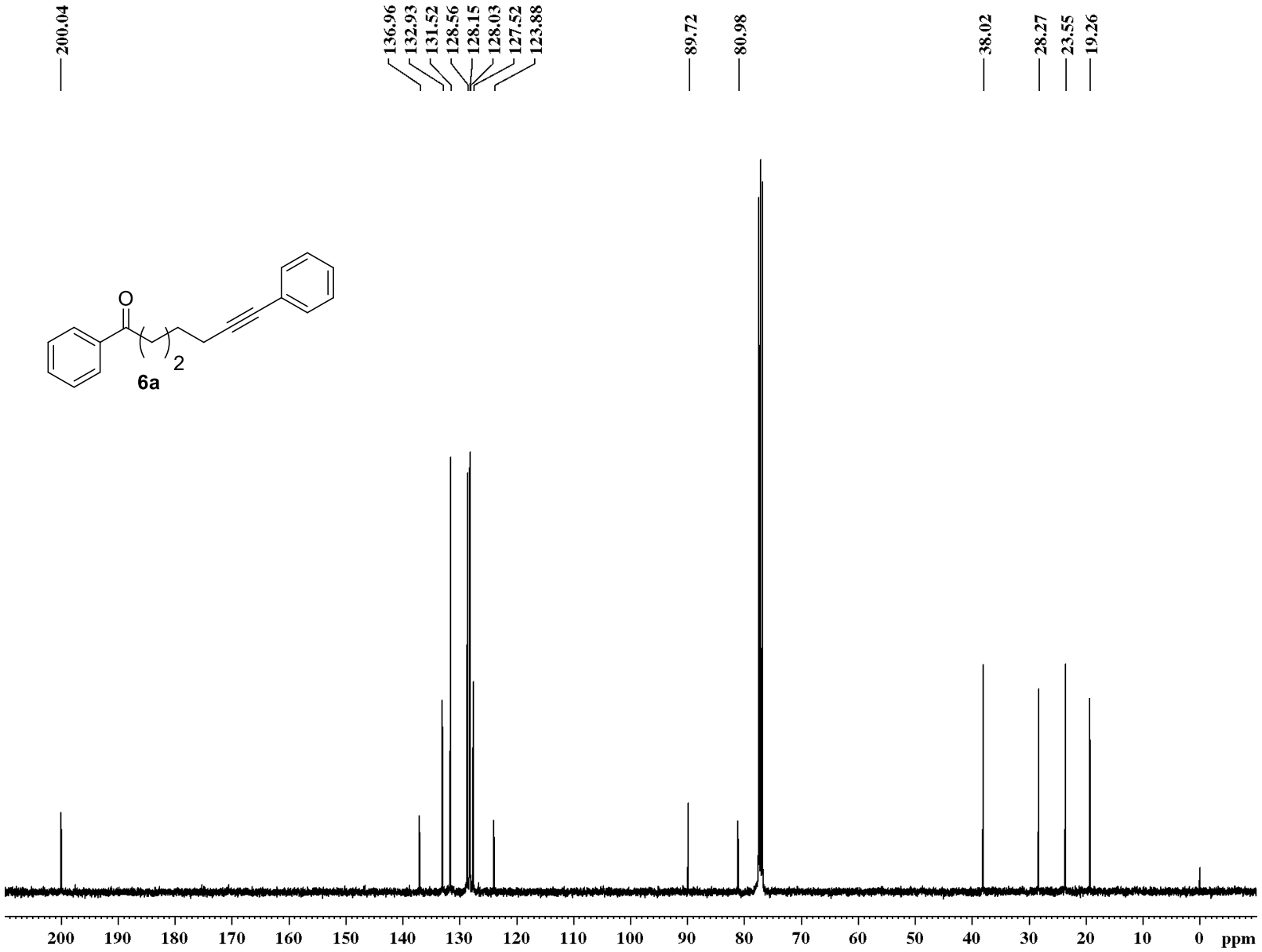
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O
2
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200
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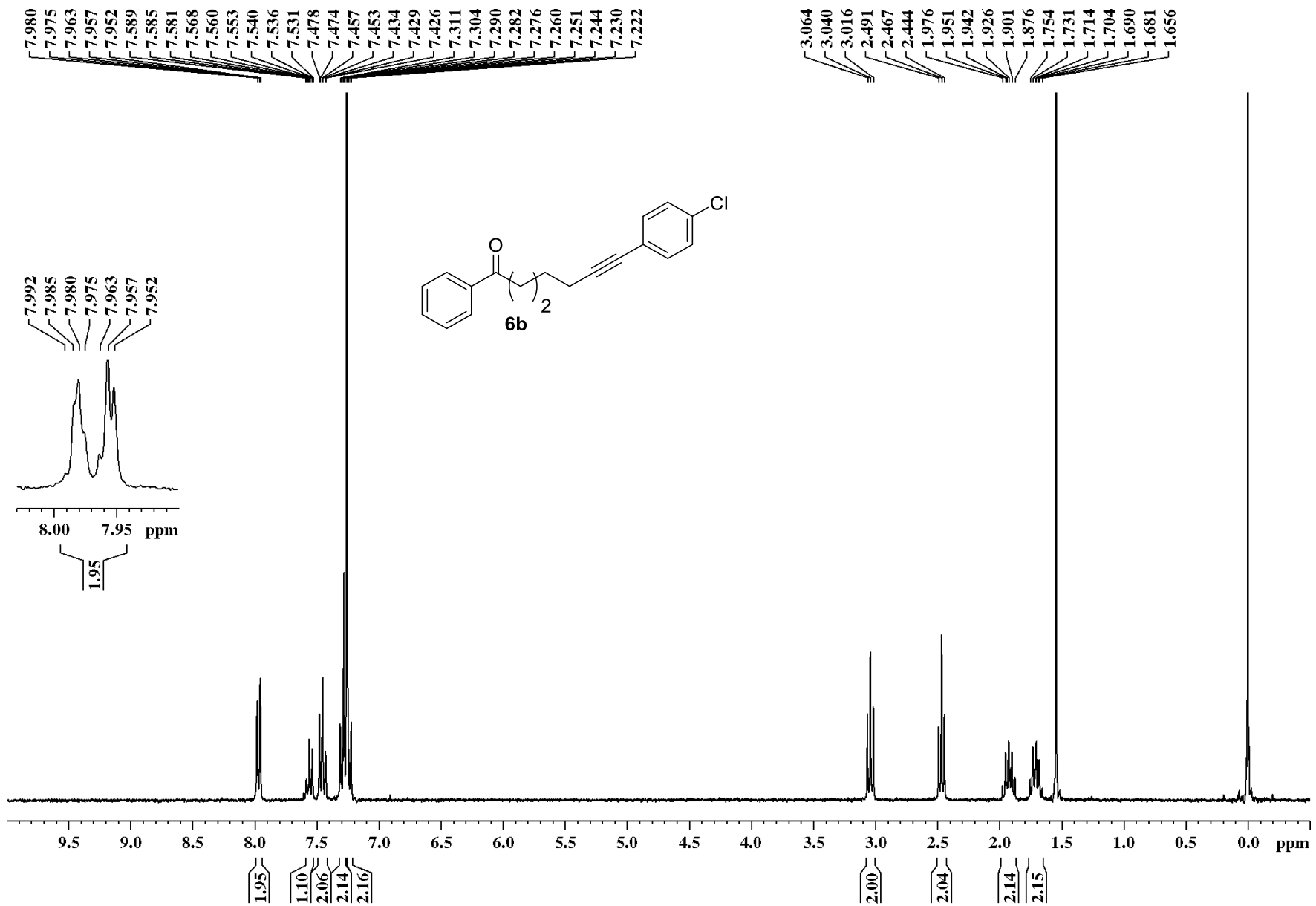
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O
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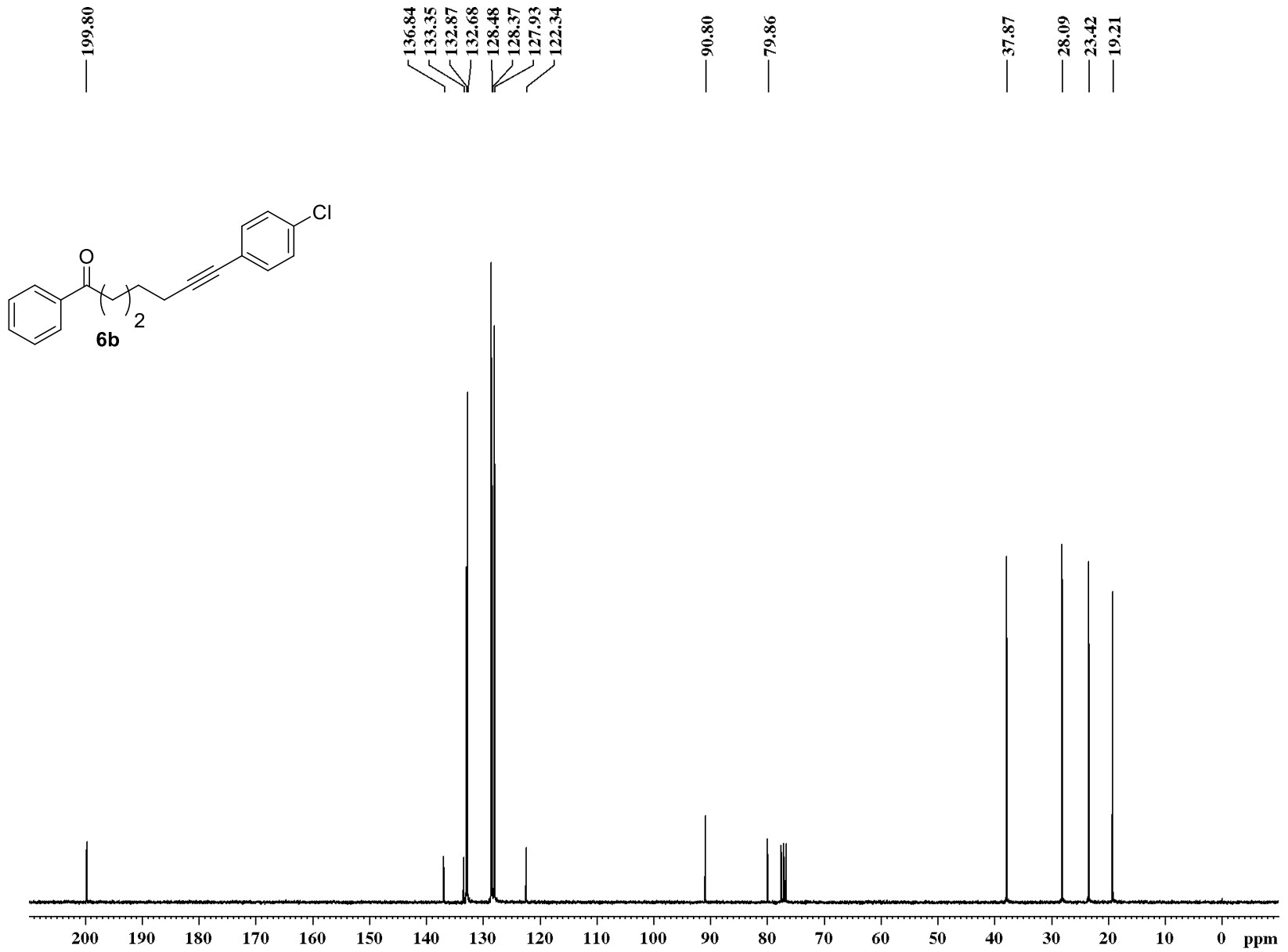
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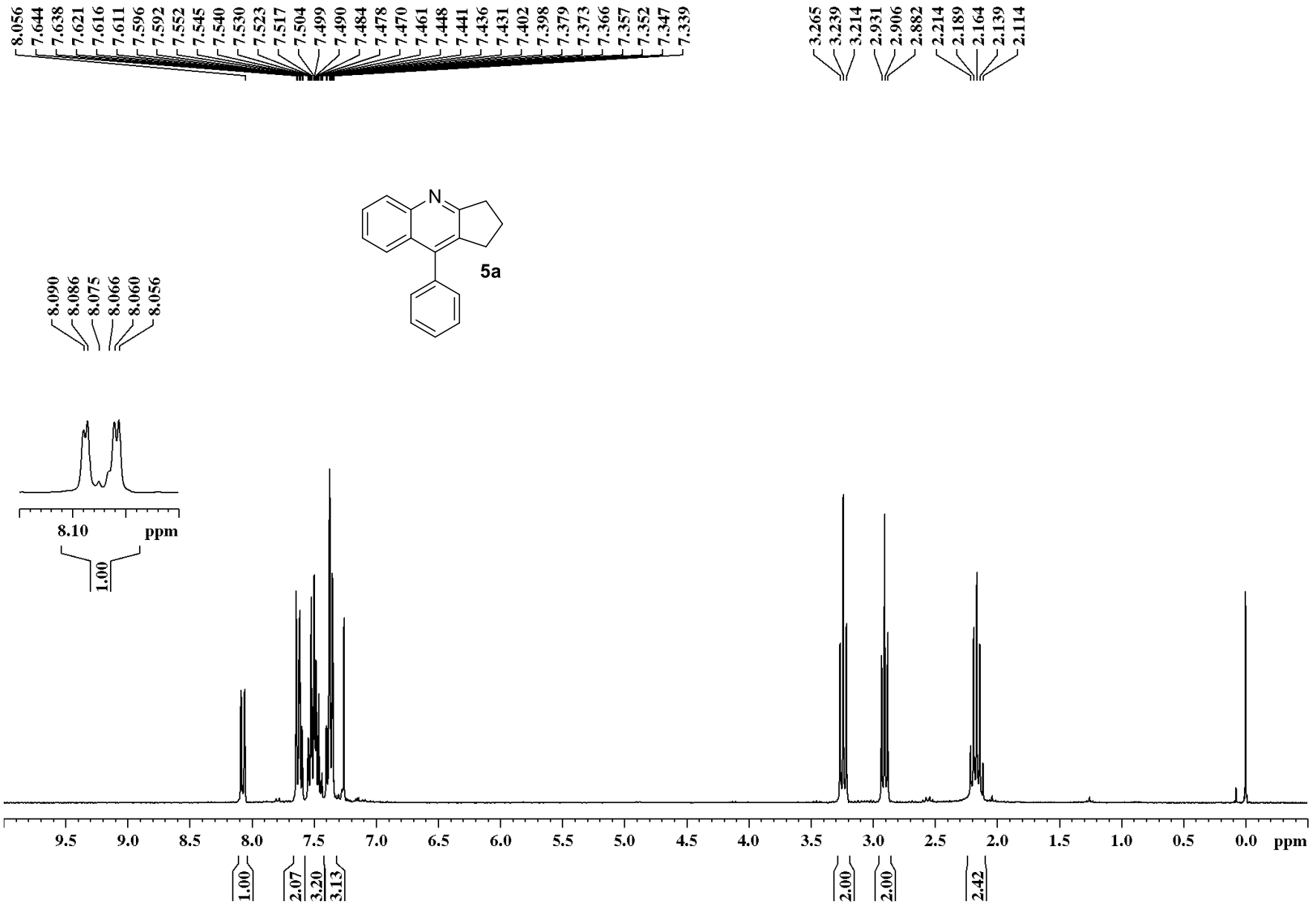
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7.504
7.499
7.490
7.484
7.478
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7.431
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7.347
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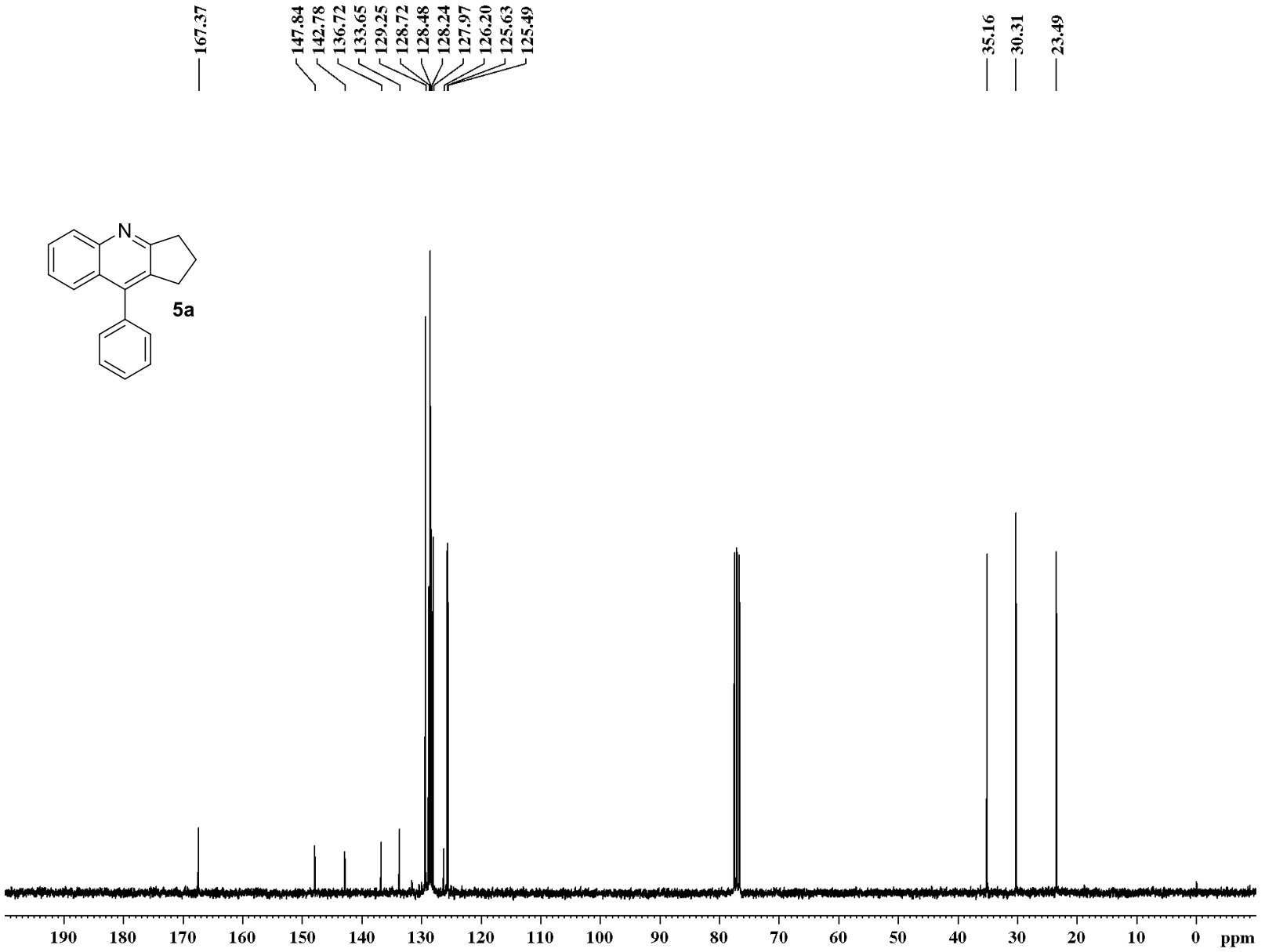
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23.49
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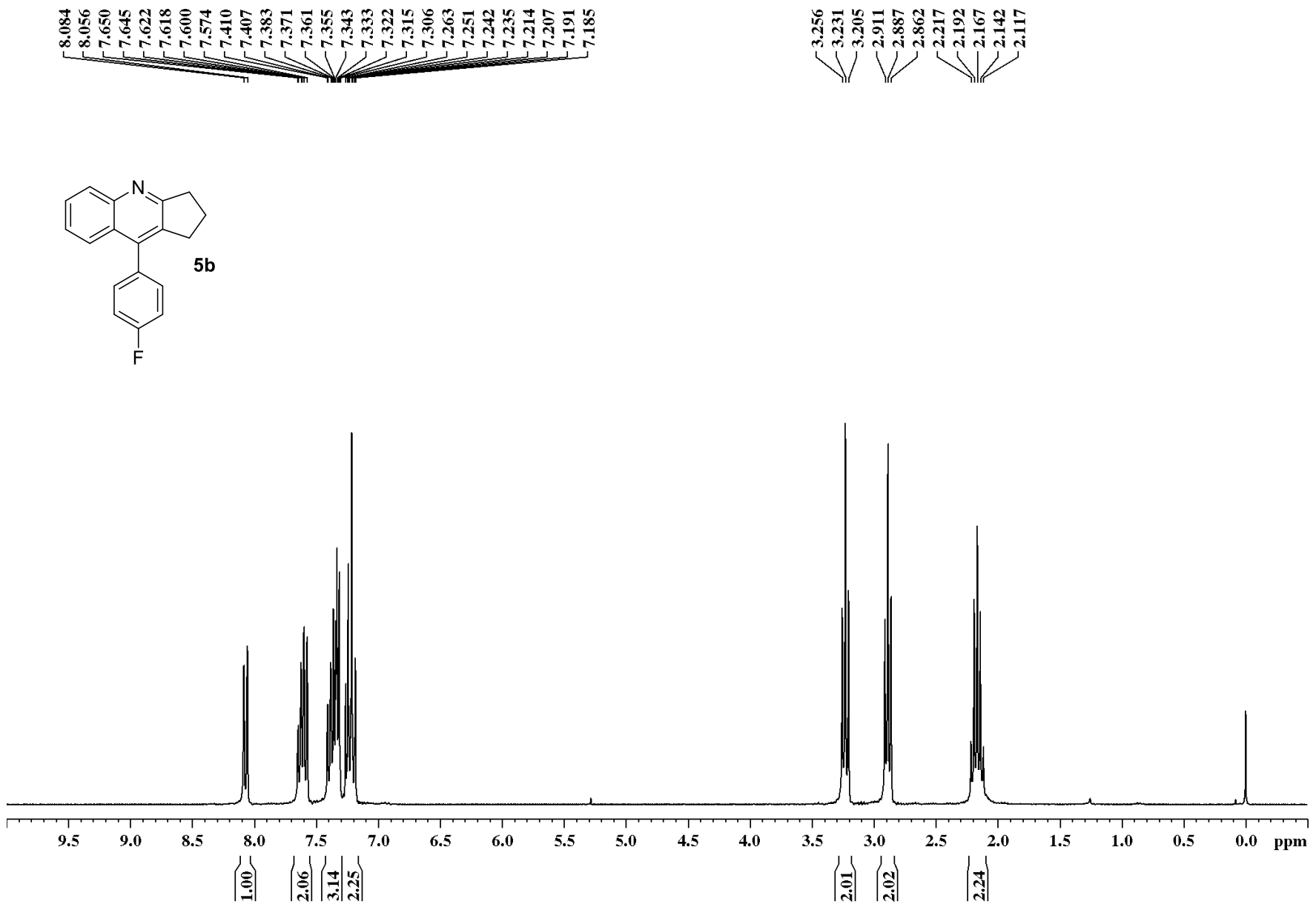
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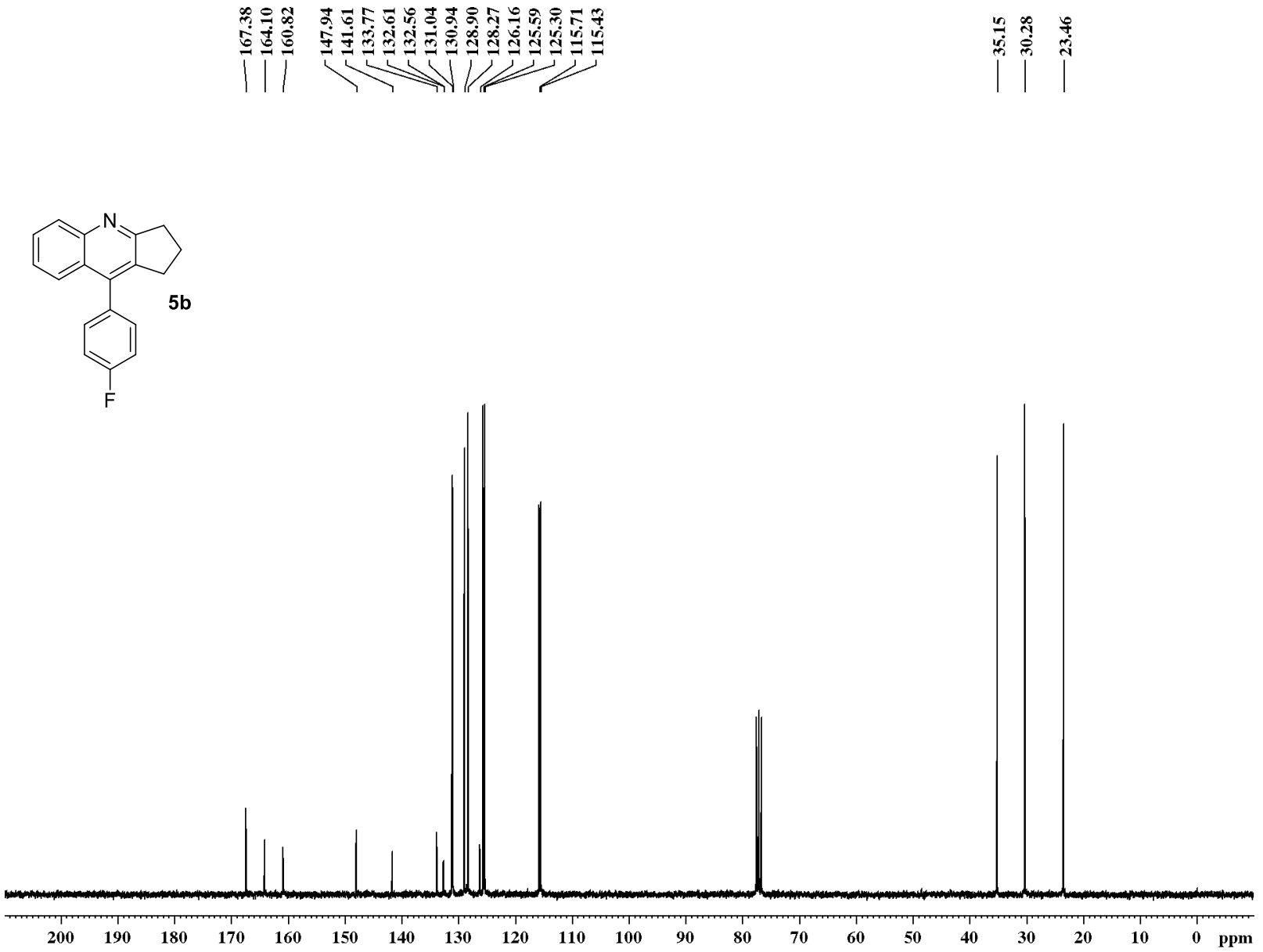
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F
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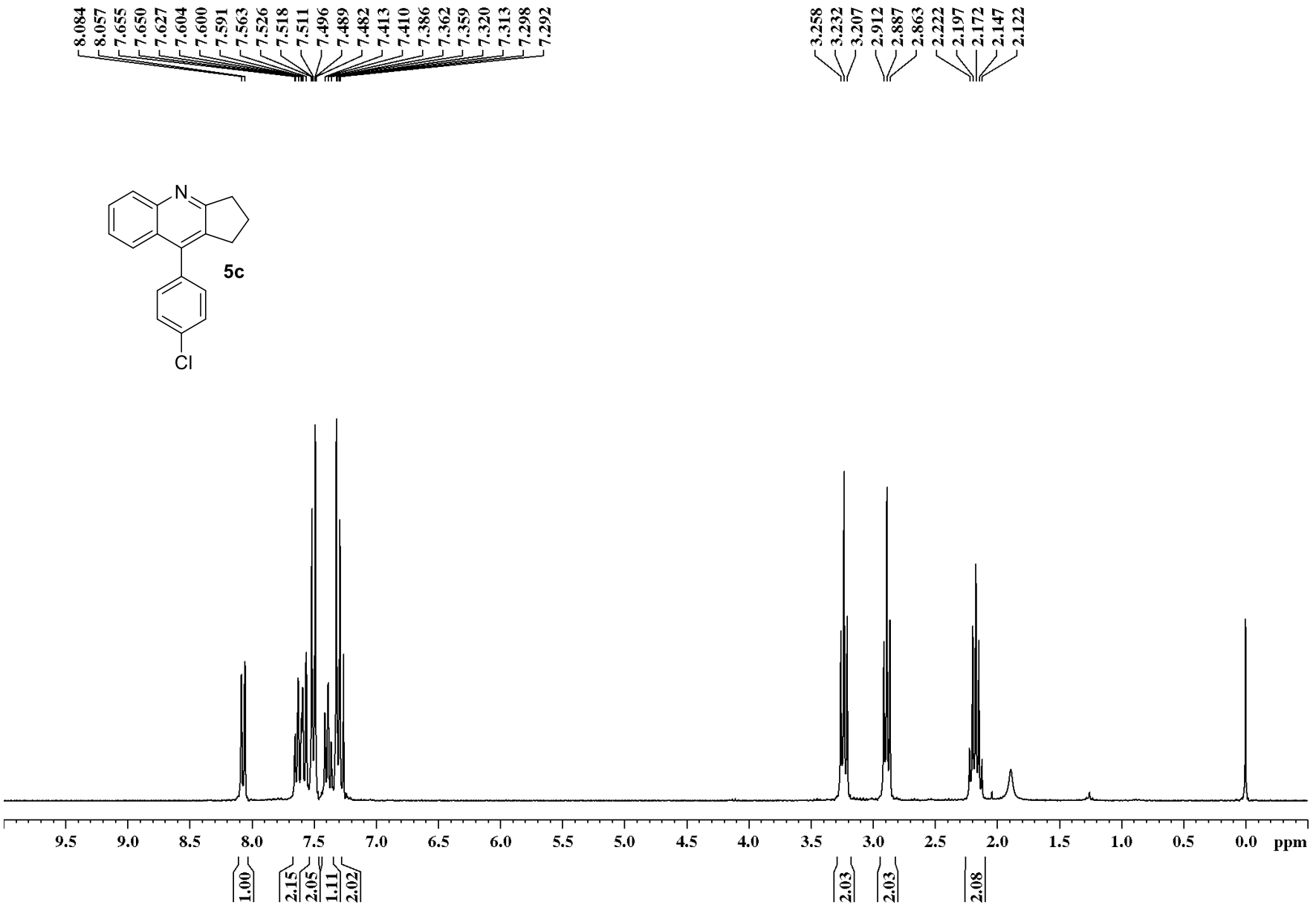
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3.232
3.207
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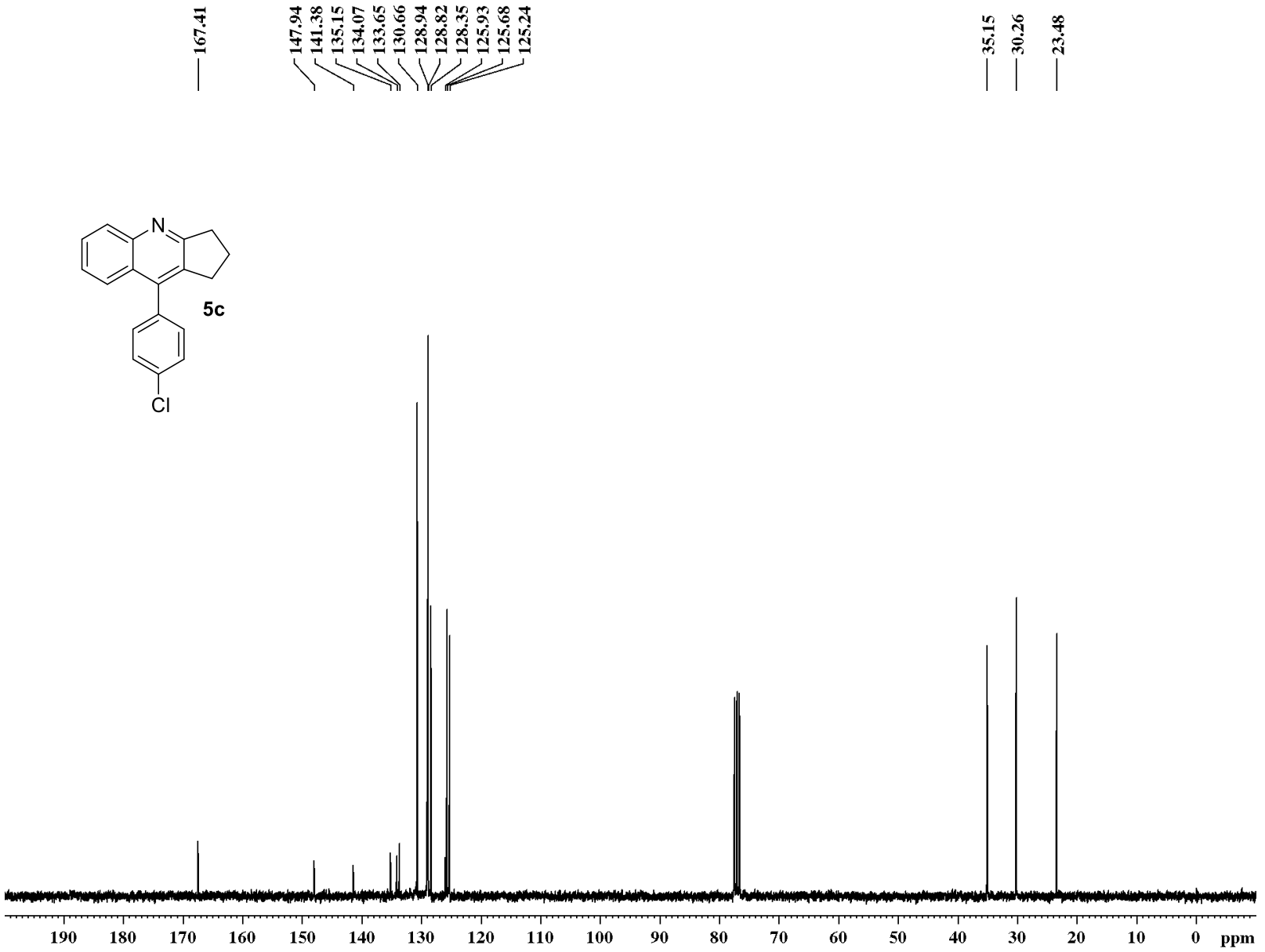
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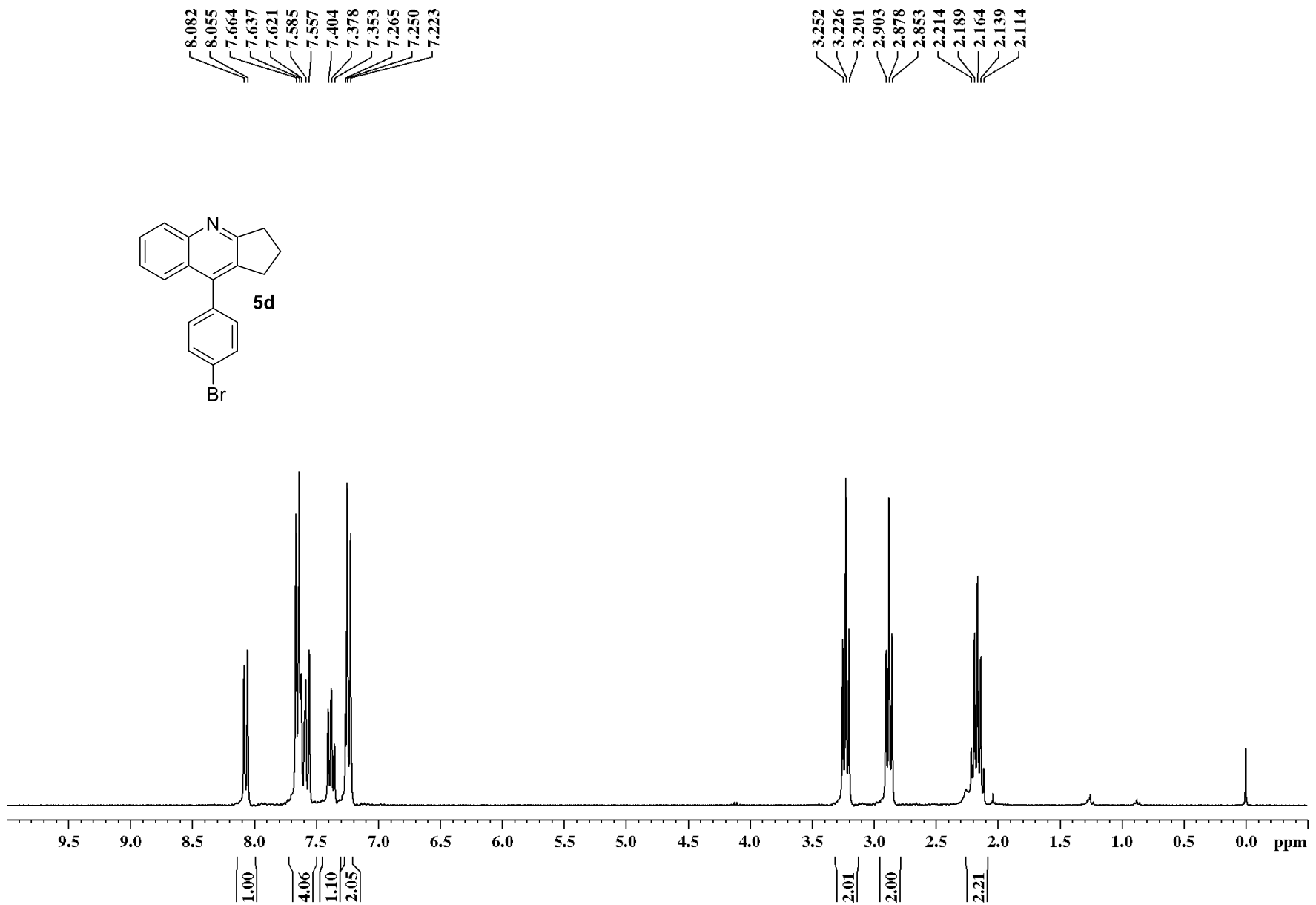
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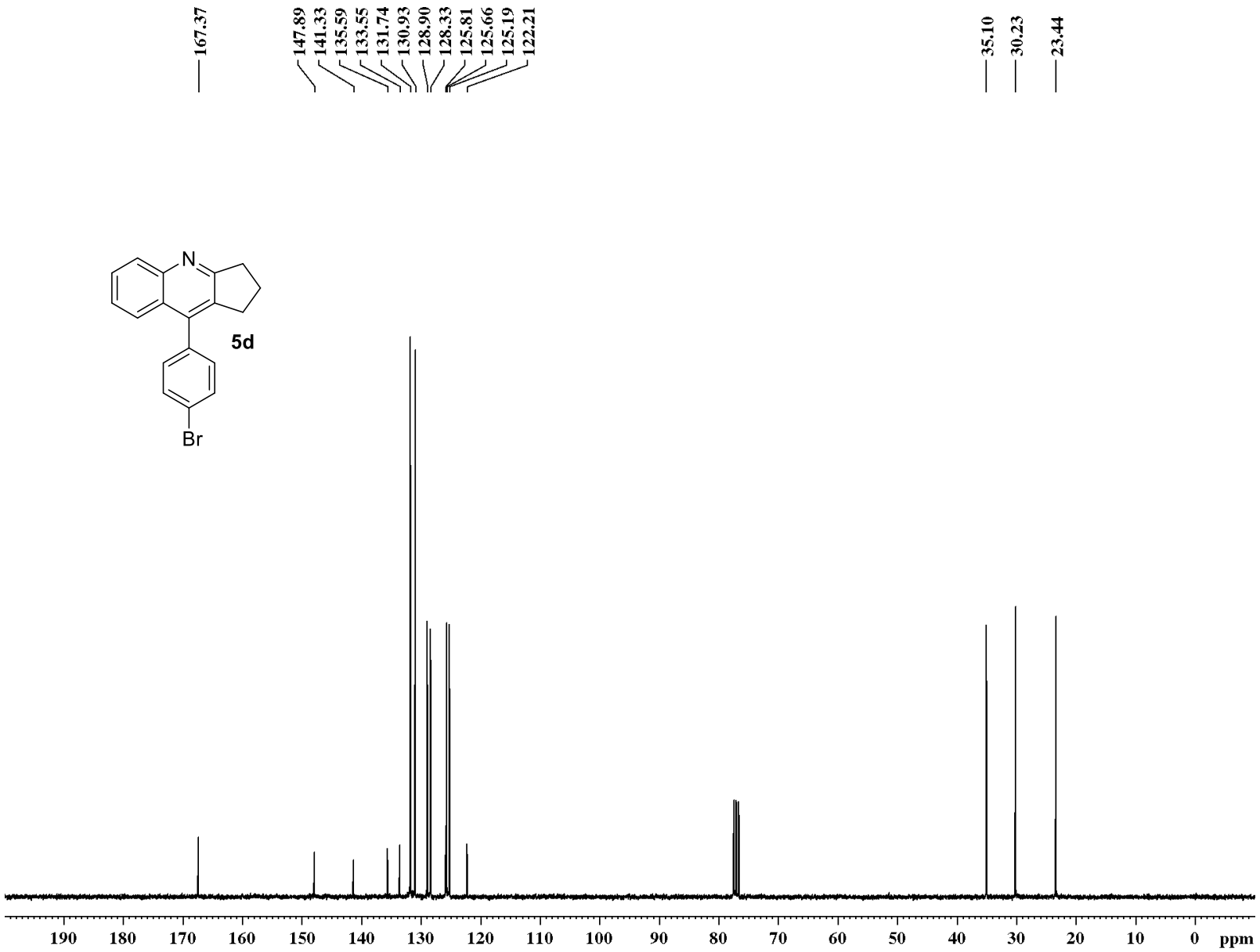
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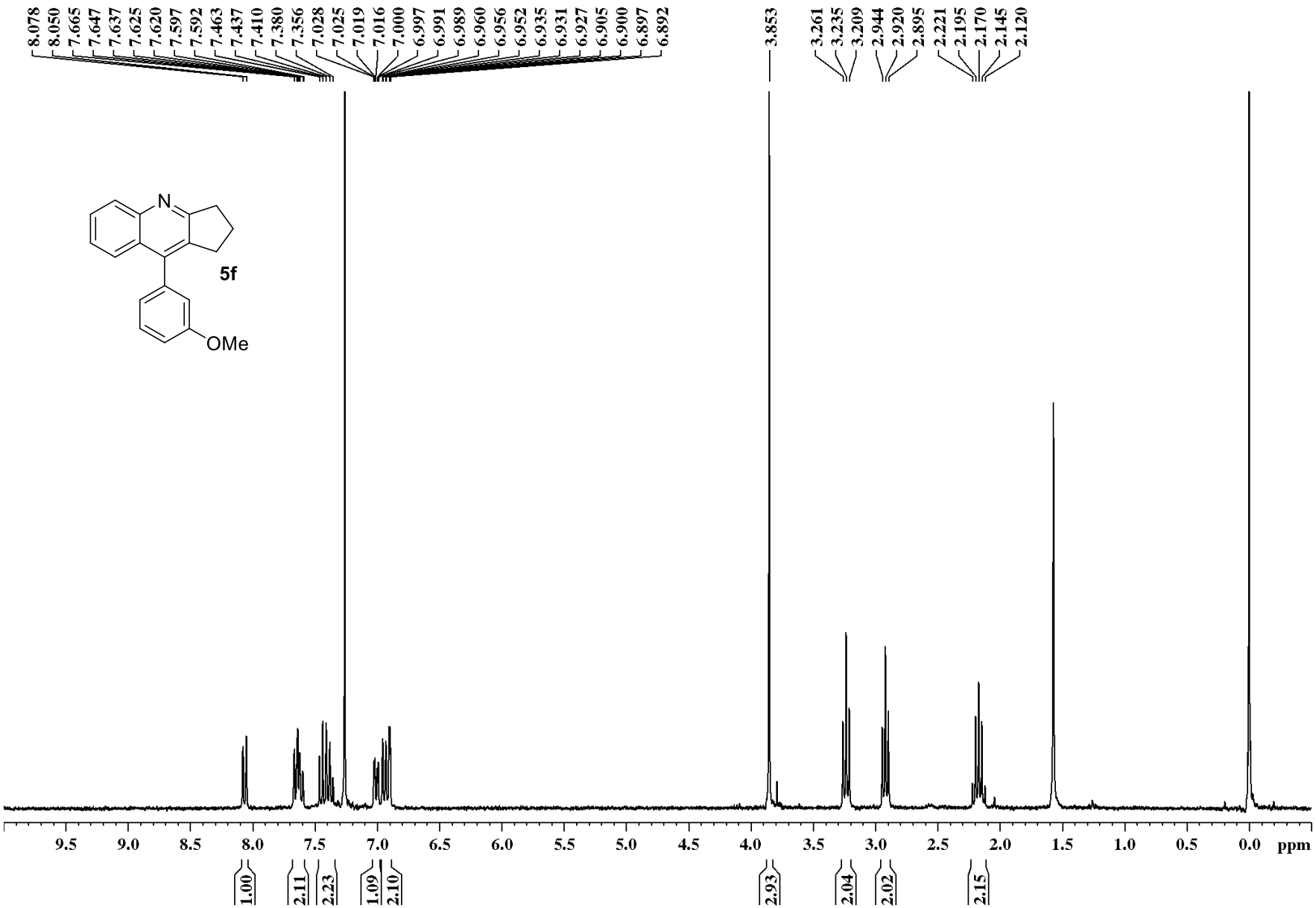
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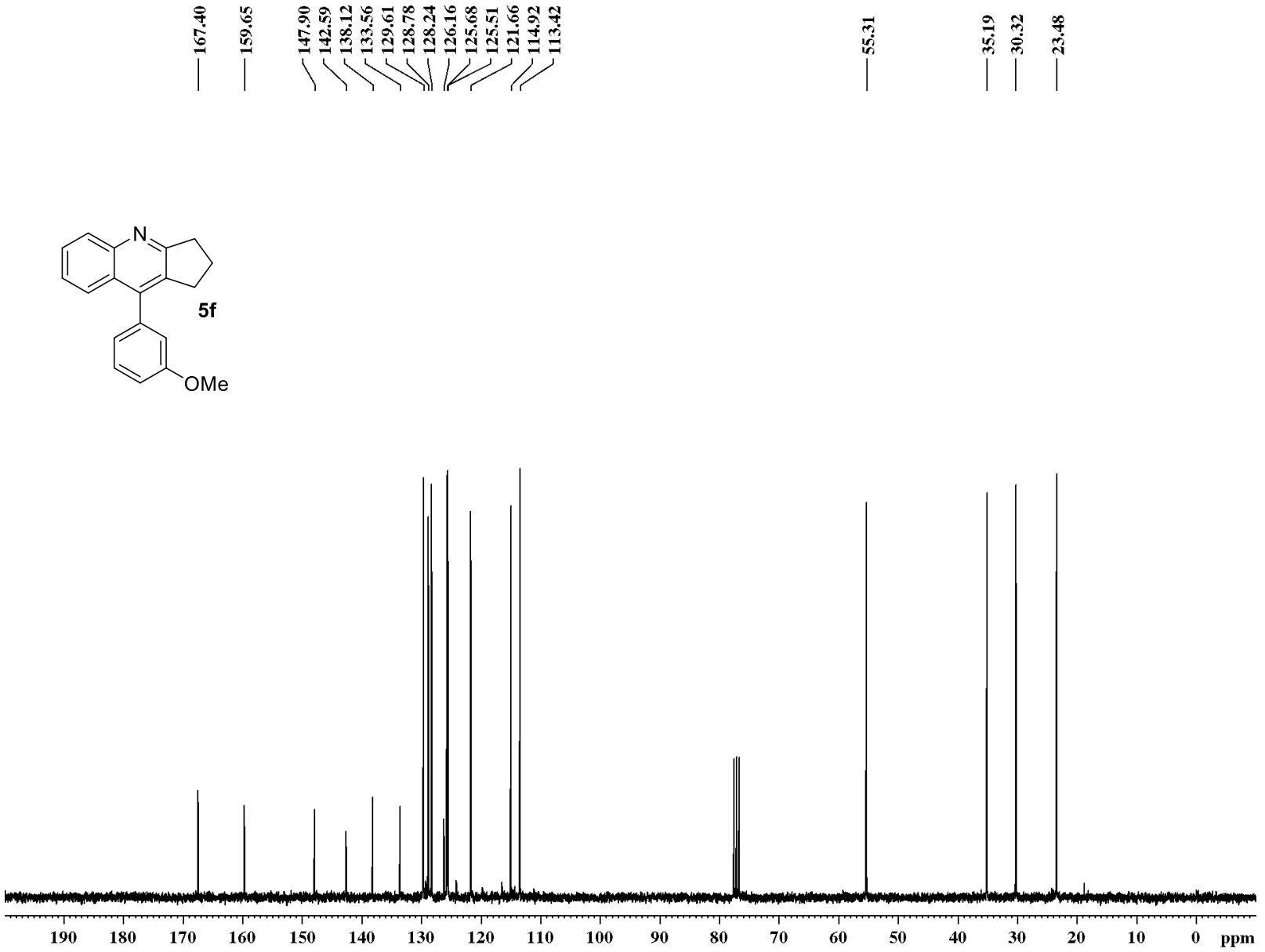
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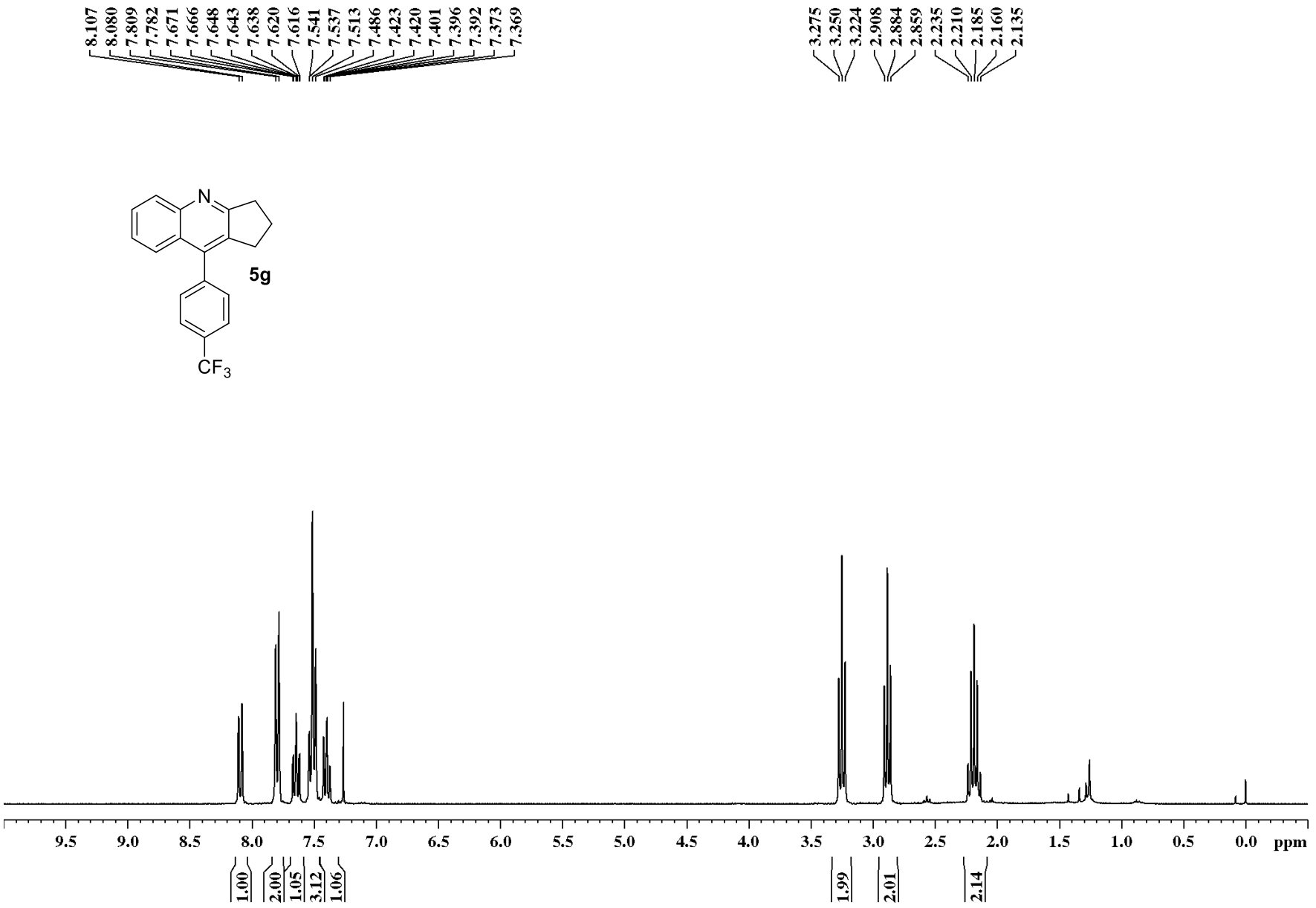
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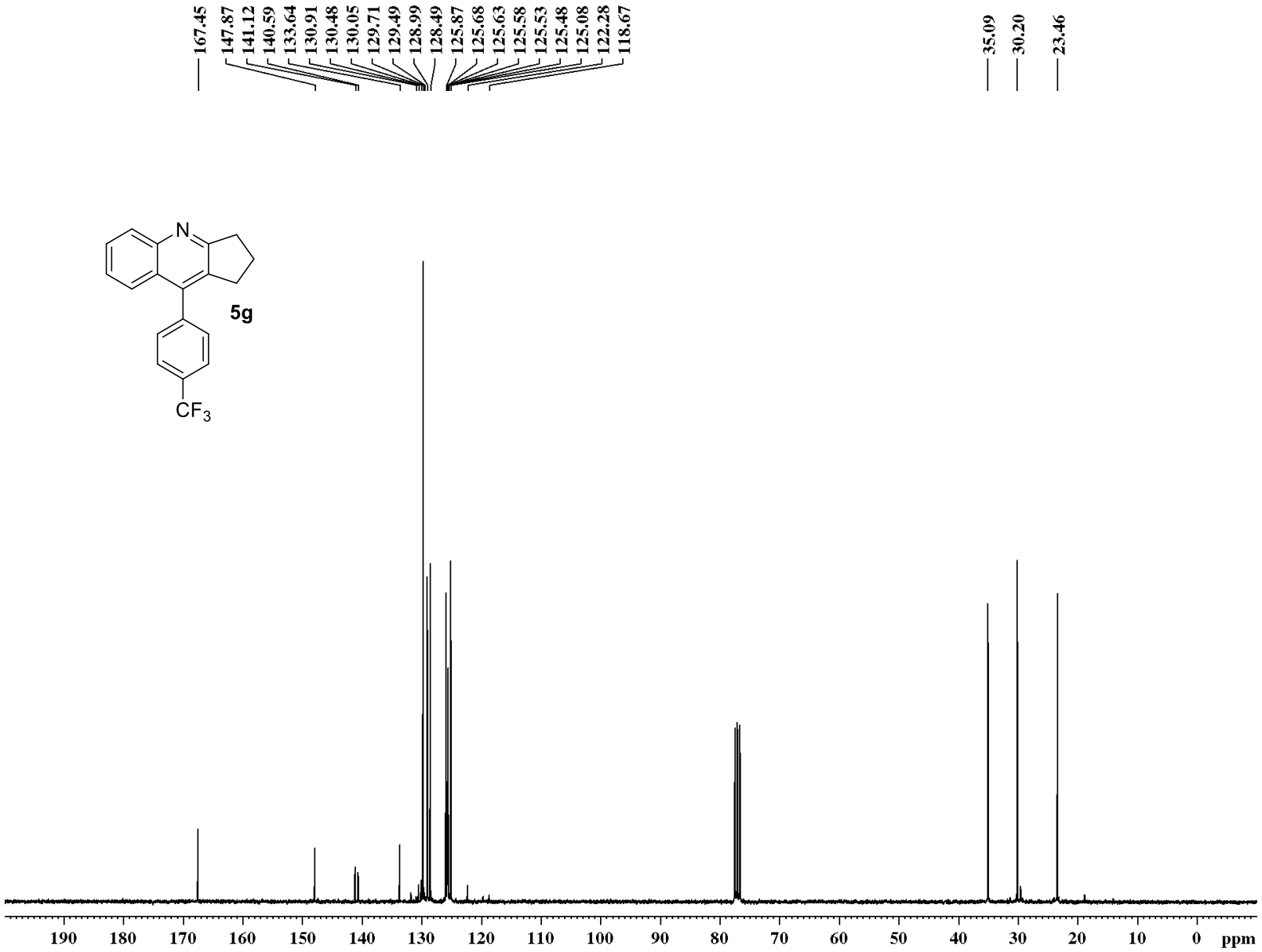
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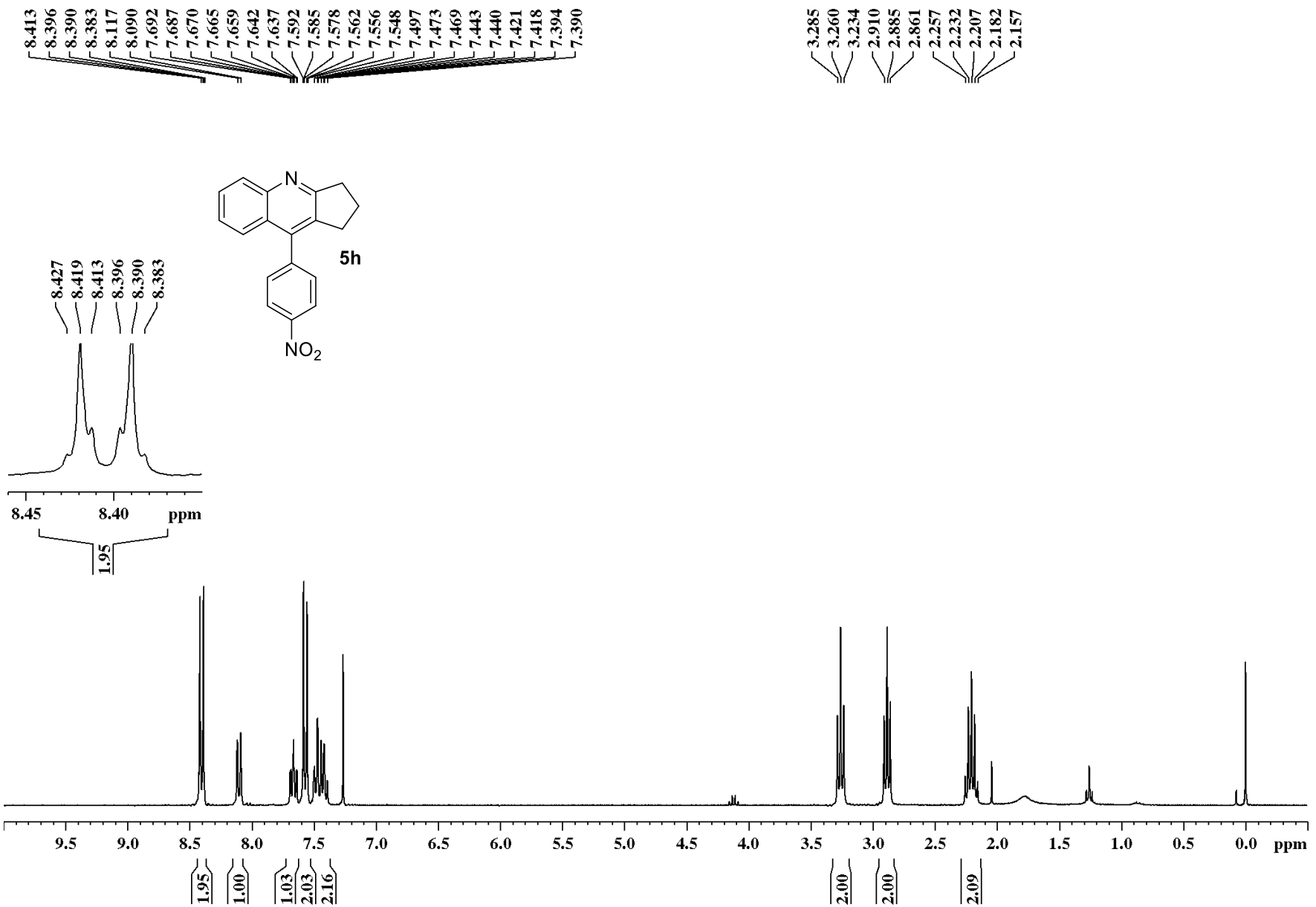
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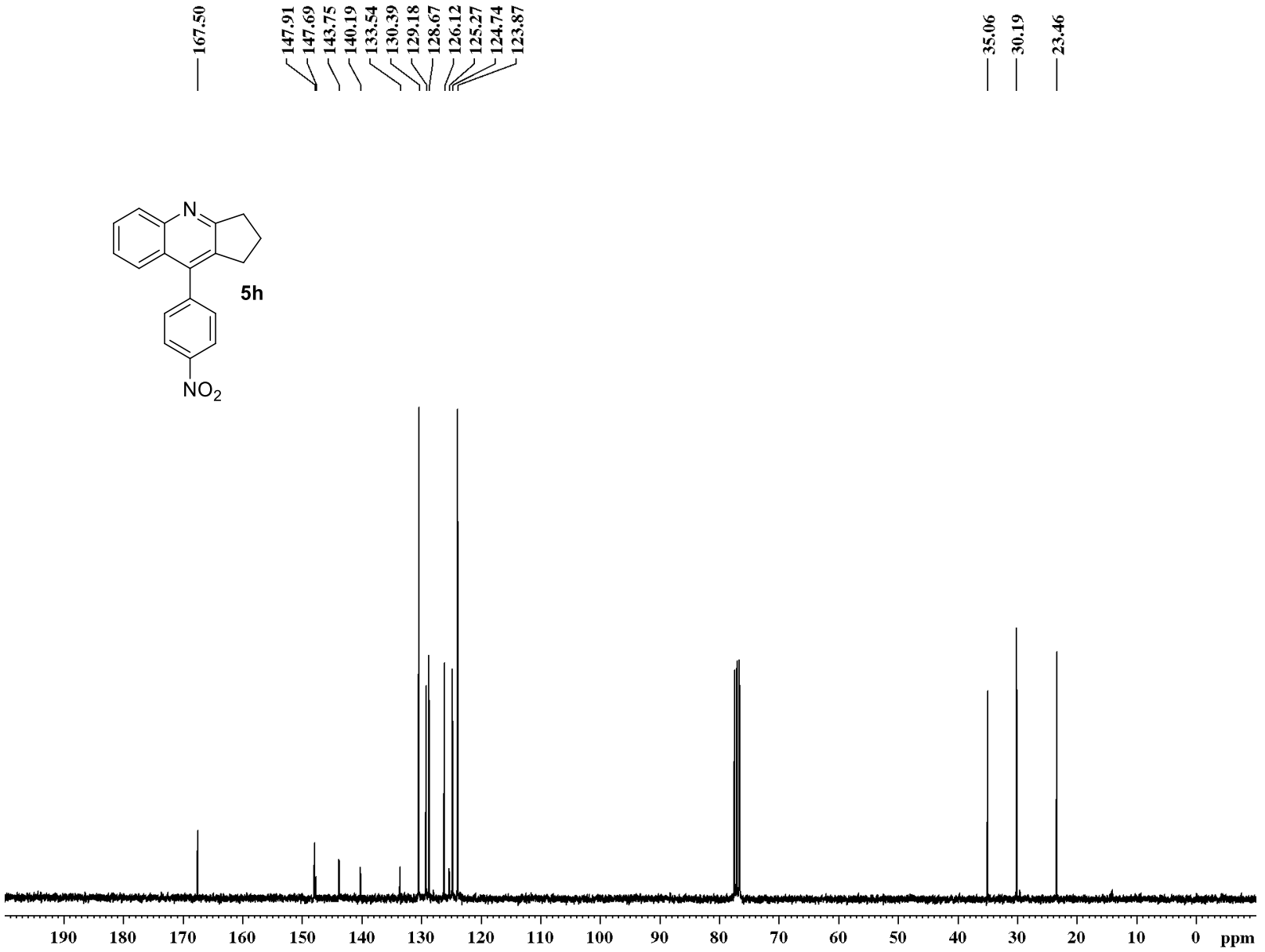
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NO2
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50
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10
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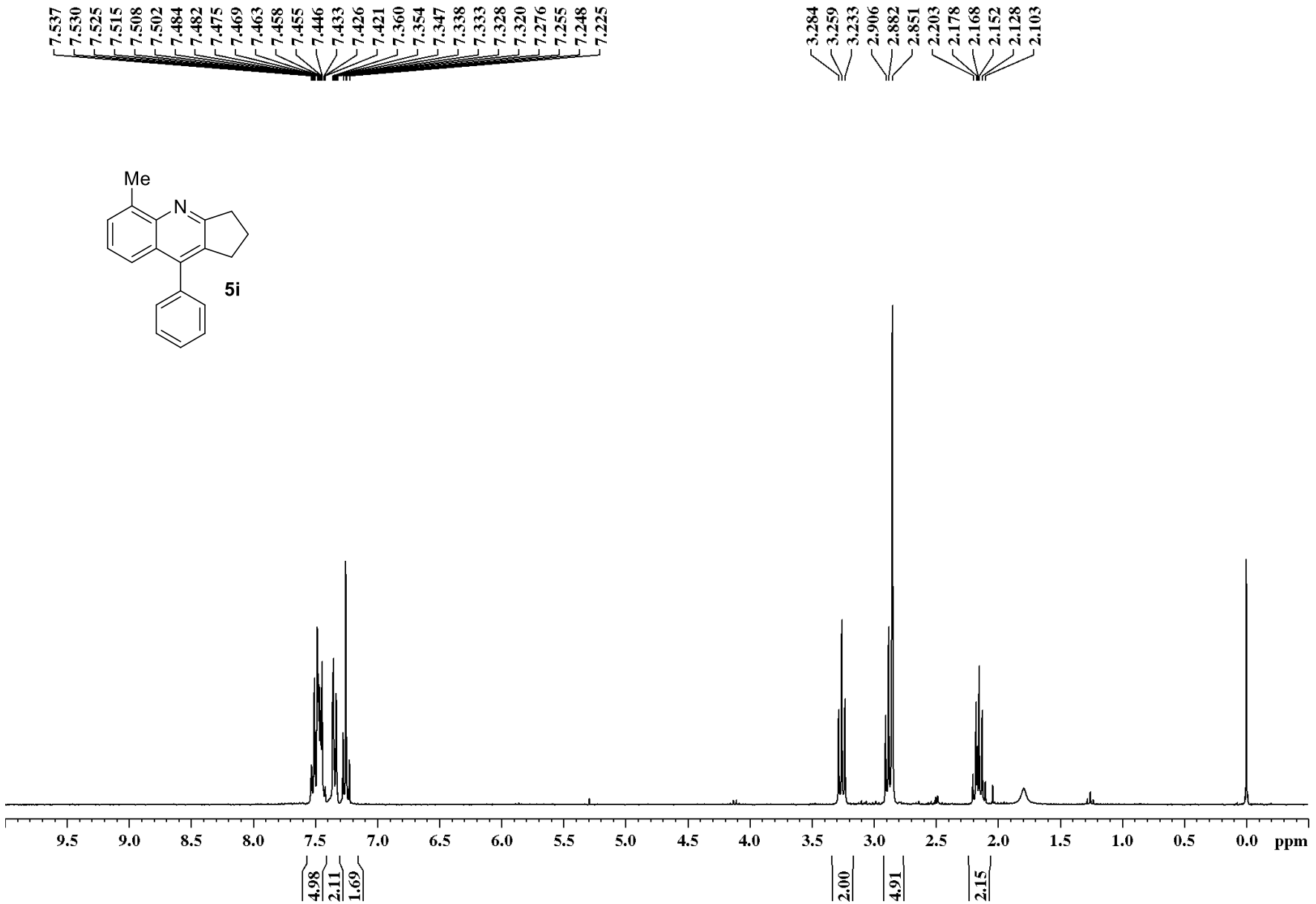
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7.502
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7.455
7.446
7.433
7.426
7.421
7.360
7.354
7.347
7.338
7.333
7.328
7.320
7.276
7.255
7.248
7.225
3.284
3.259
3.233
2.906
2.882
2.851
2.203
2.178
2.168
2.152
2.128
2.103
Me
N
5i
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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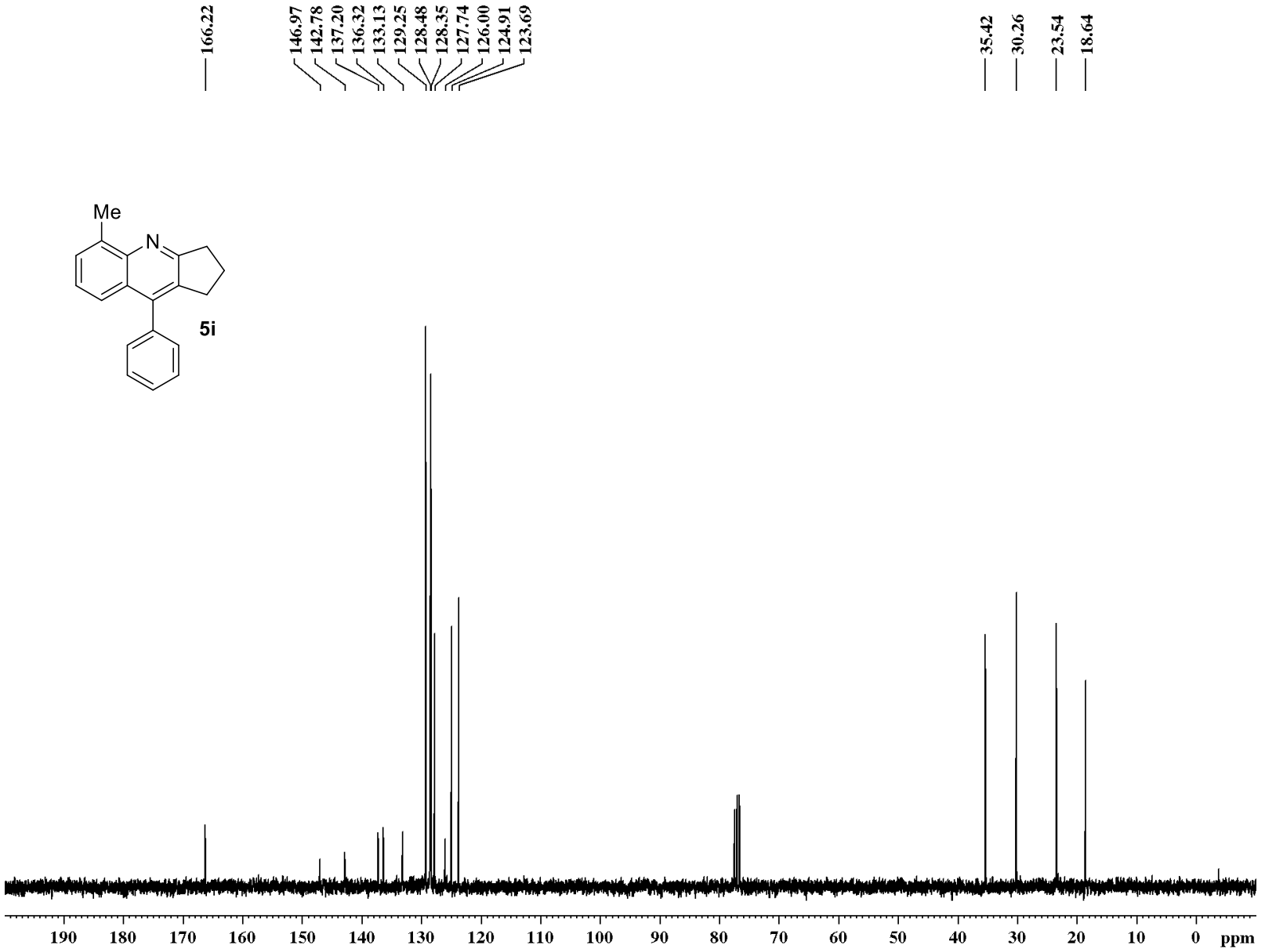
2.15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133.13
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128.48
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127.74
126.00
124.91
123.69
35.42
30.26
23.54
18.64
Me
N
5i
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140
130
120
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100
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60
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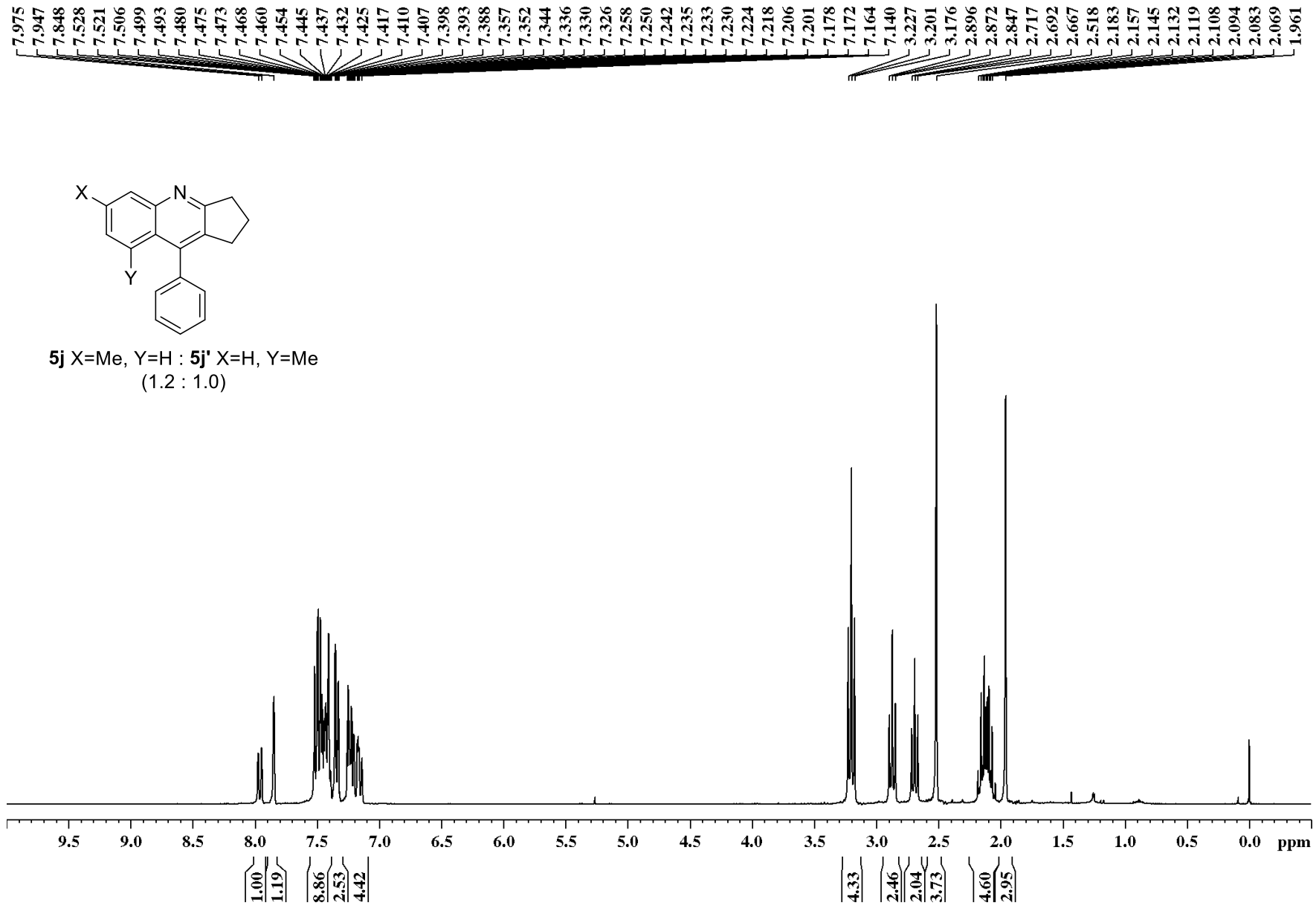
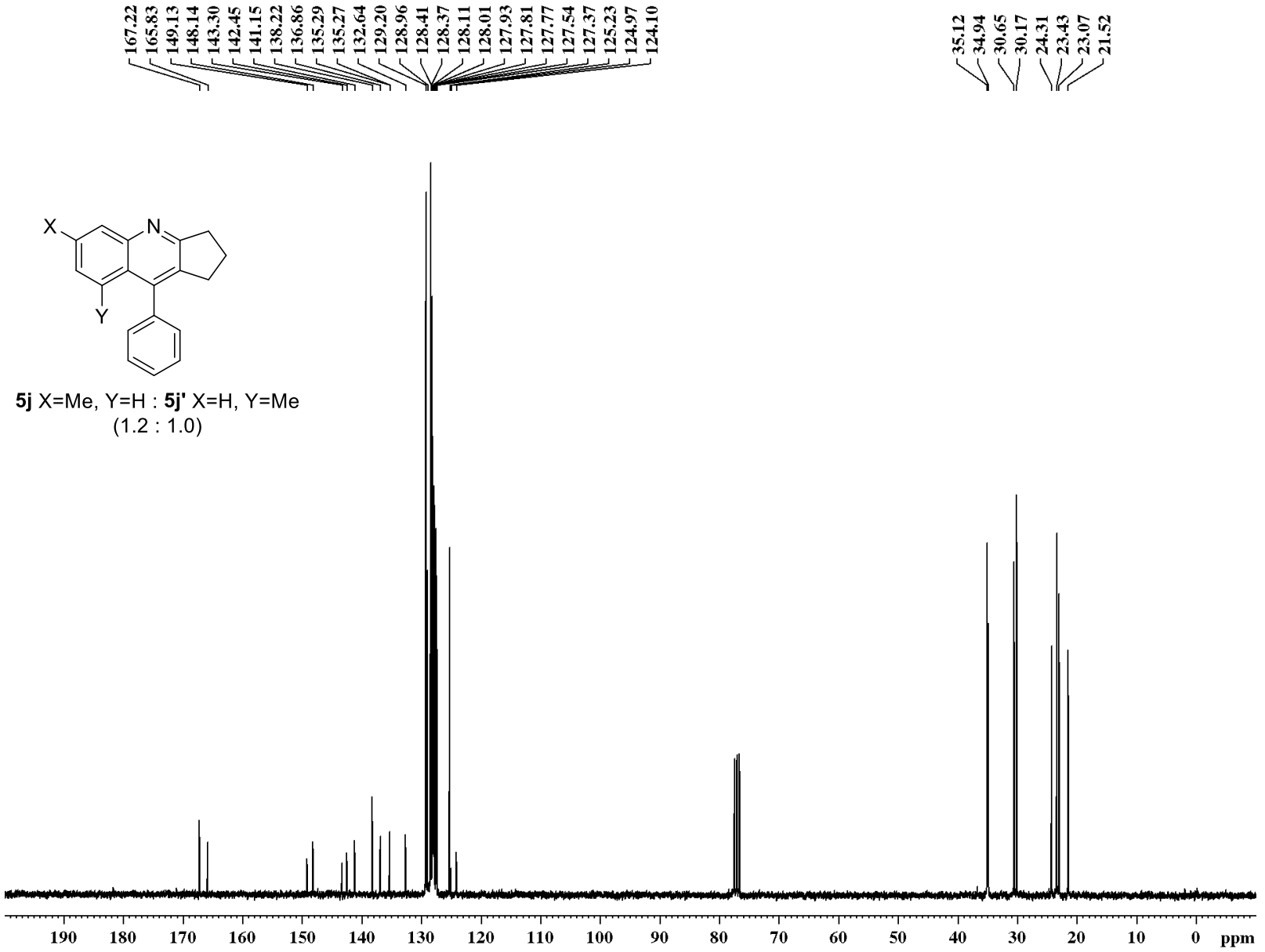


5j X=Me, Y=H : **5j'** X=H, Y=Me
(1.2 : 1.0)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142.45
141.15
138.22
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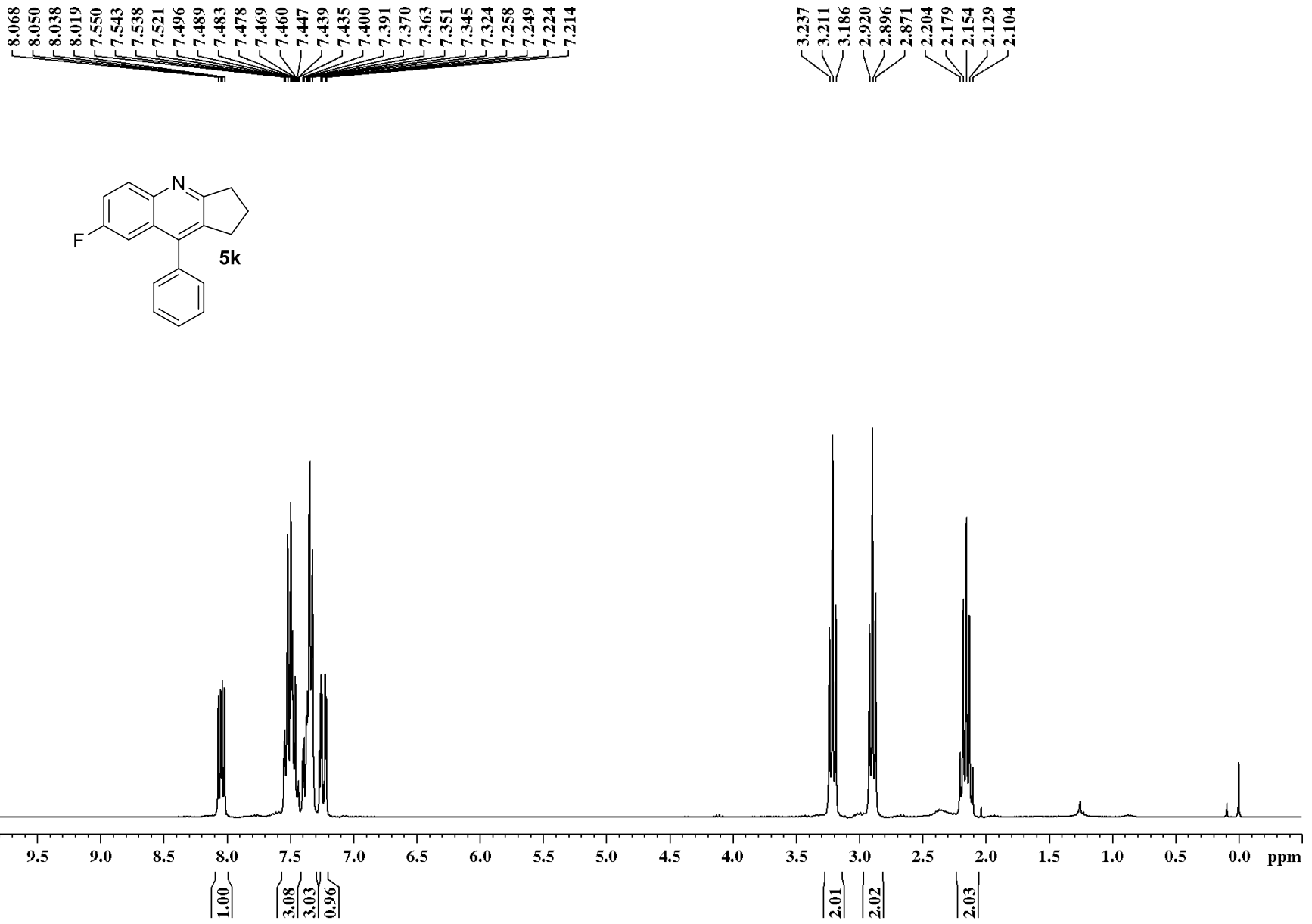
135.29
135.27
132.64
129.20
128.96
128.41
128.37
128.11
128.01
127.93
127.81
127.77
127.54
127.37
125.23
124.97
124.10
35.12
34.94
30.65
30.17
24.31
23.43
23.07
21.52
X
N
Y
5j X=Me, Y=H : **5j'** X=H, Y=Me
(1.2 : 1.0)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.478
7.469
7.460
7.447
7.439
7.435
7.400
7.391
7.370
7.363
7.351
7.345
7.324
7.258
7.249
7.224
7.214
3.237
3.211
3.186
2.920
2.896
2.871
2.204
2.179
2.154
2.129
2.104
N
F
5k
9.5
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
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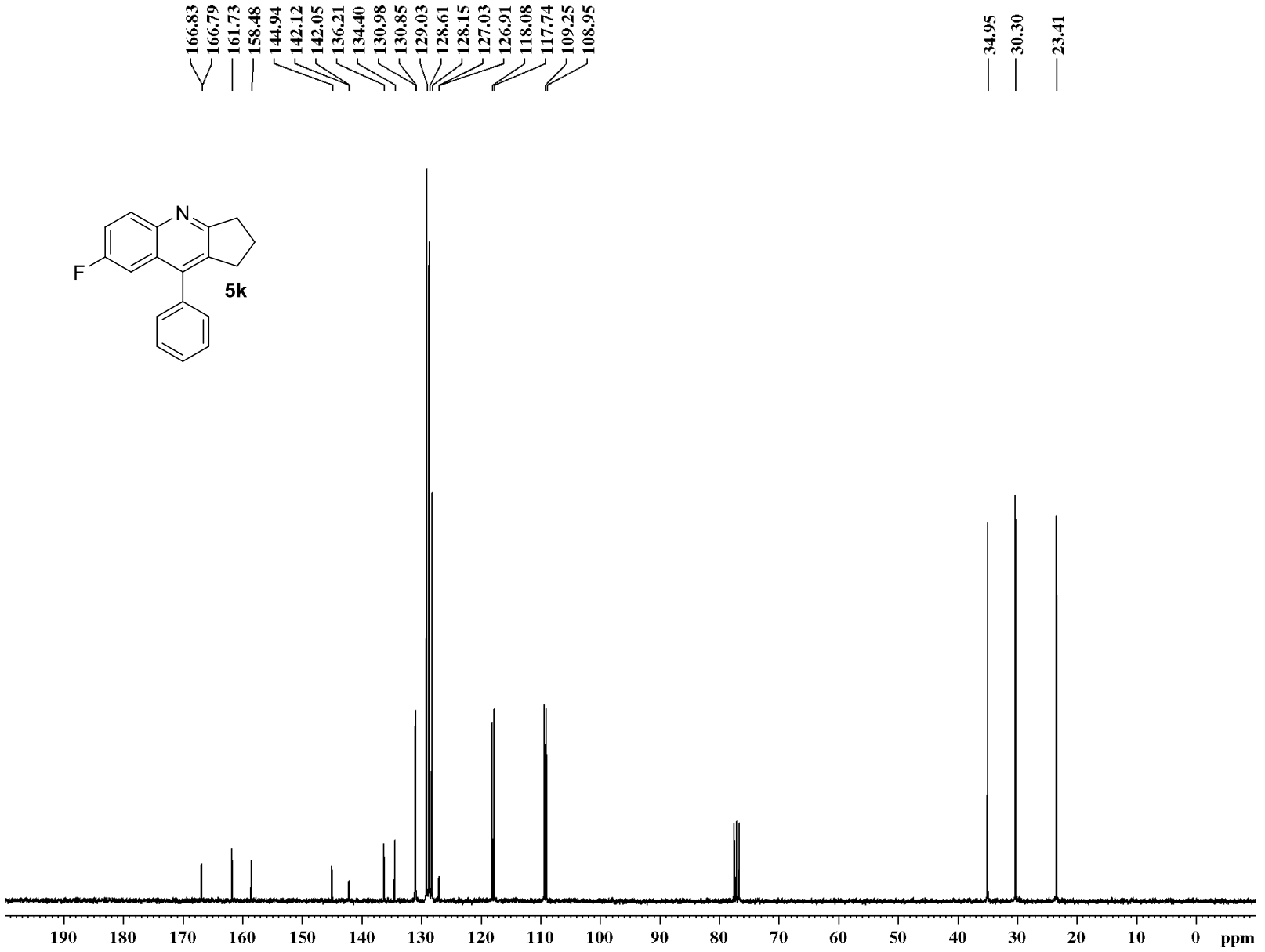
2.02
2.03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142.05
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130.98
130.85
129.03
128.61
128.15
127.03
126.91
118.08
117.74
109.25
108.95
34.95
30.30
23.41
N
F
5k
190
180
170
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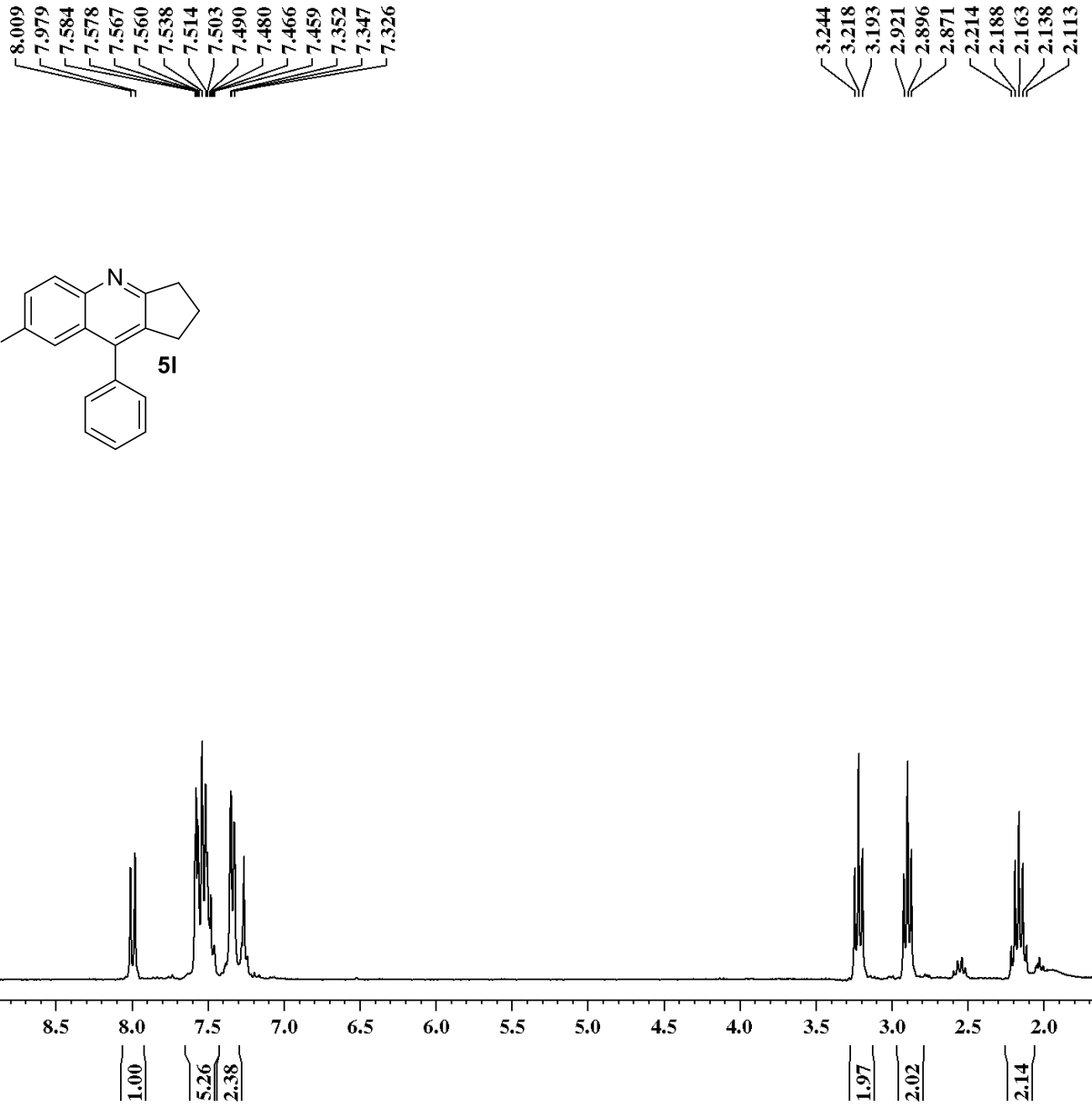
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.584
7.578
7.567
7.560
7.538
7.514
7.503
7.490
7.480
7.466
7.459
7.352
7.347
7.326
3.244
3.218
3.193
2.921
2.896
2.871
2.214
2.188
2.163
2.138
2.113
N
Cl
5l
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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0.0
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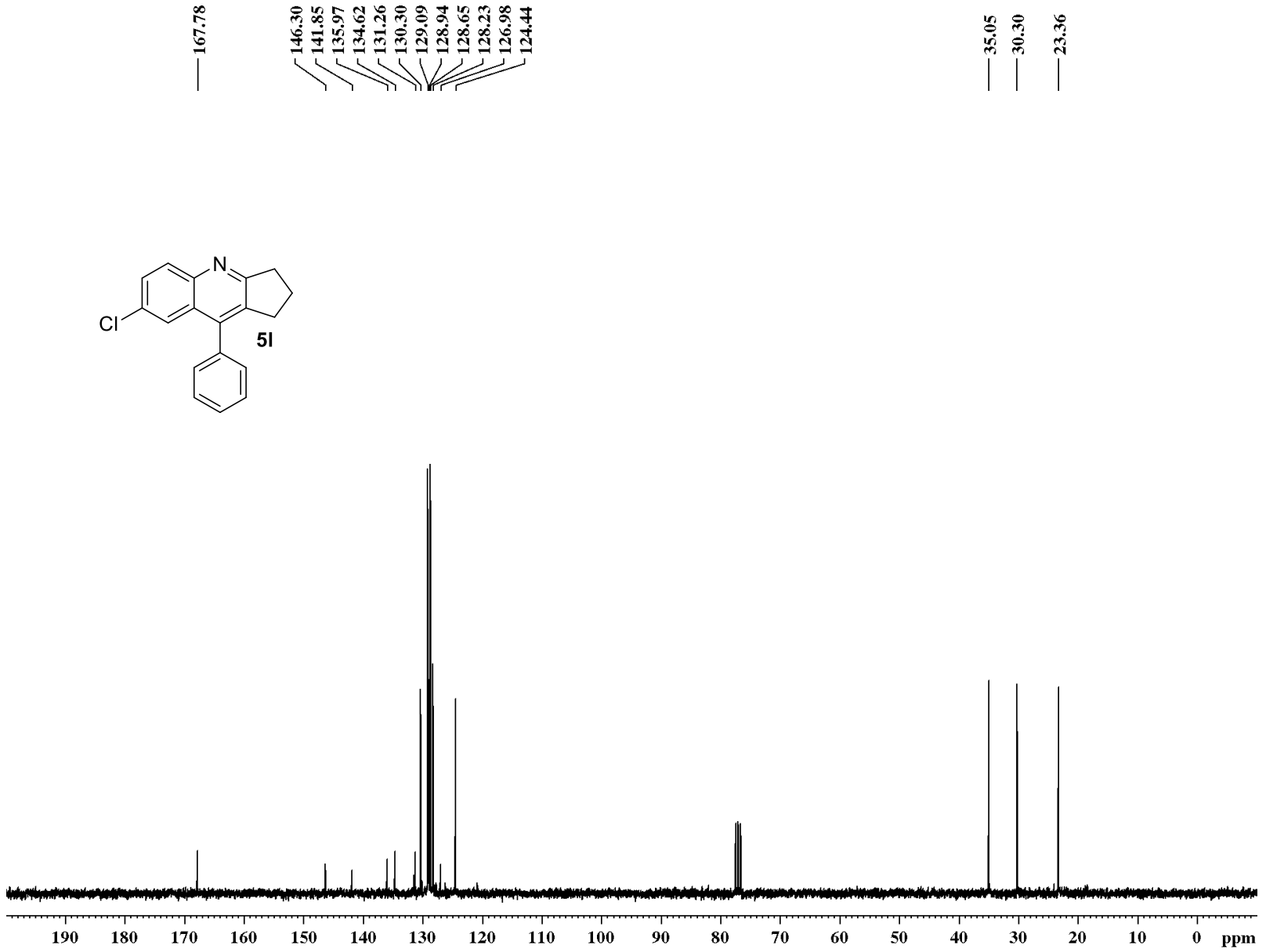
2.14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131.26
130.30
129.09
128.94
128.65
128.23
126.98
124.44
35.05
30.30
23.36
N
Cl
5l
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180
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150
140
130
120
110
100
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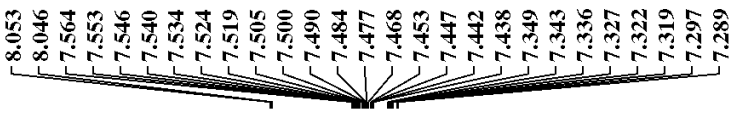
80
70
60
50
40
30
20
10
0
ppm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7.524
7.519
7.505
7.500
7.490
7.484
7.477
7.468
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7.442
7.438
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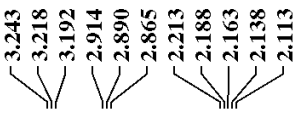
7.343
7.336
7.327
7.322
7.319
7.297
7.289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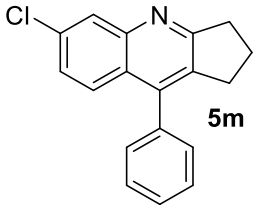
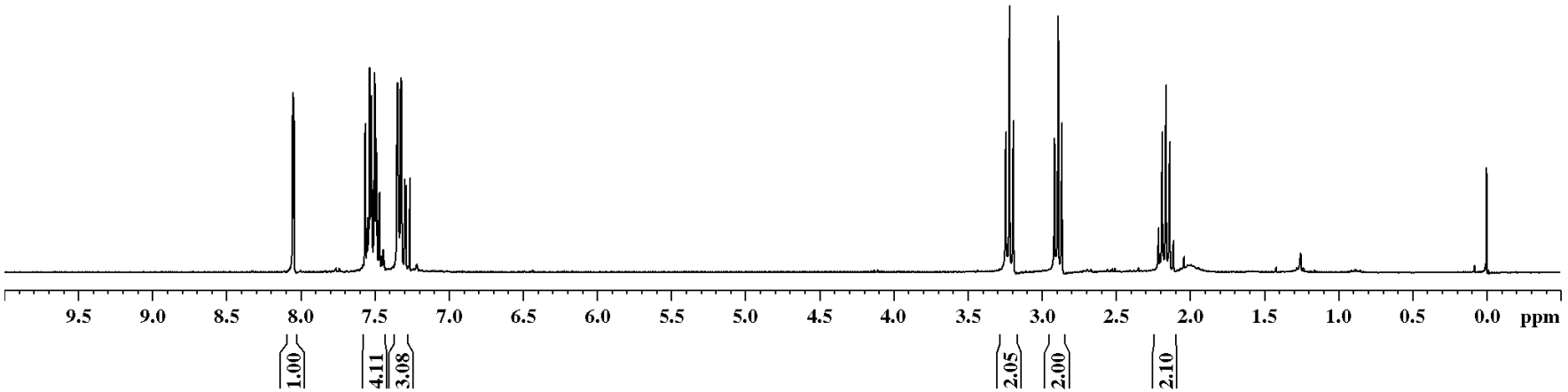
2.213
2.188
2.163
2.138
2.113


Cl
N
5m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4.5
4.0
3.5
3.0
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1.0
0.5
0.0
ppm
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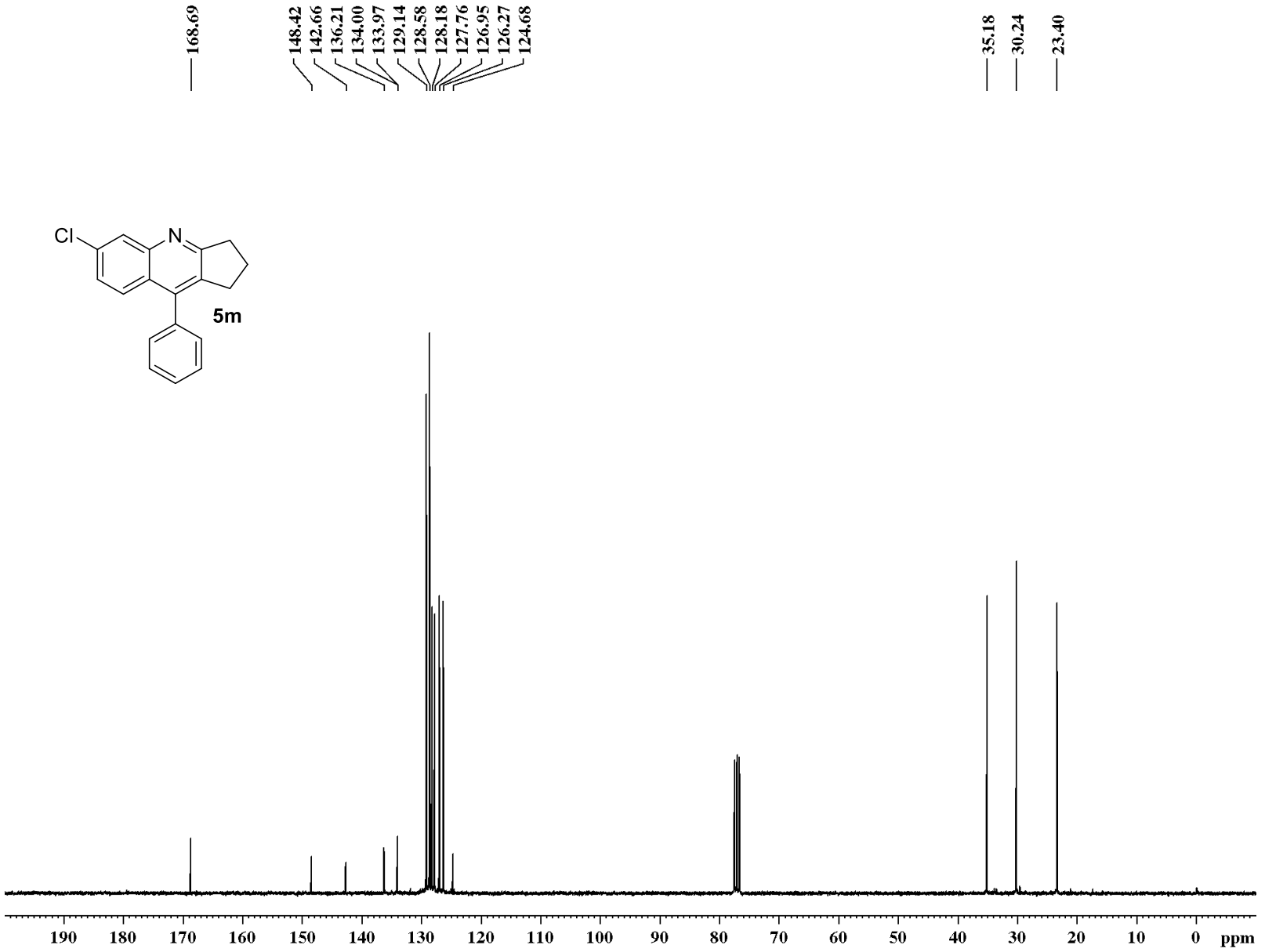
4.11
3.08
2.05
2.00
2.10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126.27
124.68
35.18
30.24
23.40
Cl
N
5m
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180
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160
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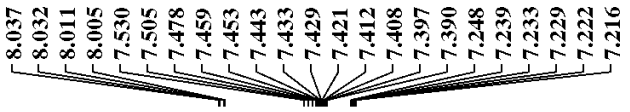
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.429
7.421
7.412
7.408
7.397
7.390
7.248
7.239
7.233
7.229
7.222
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3.200
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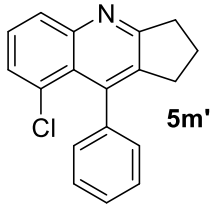
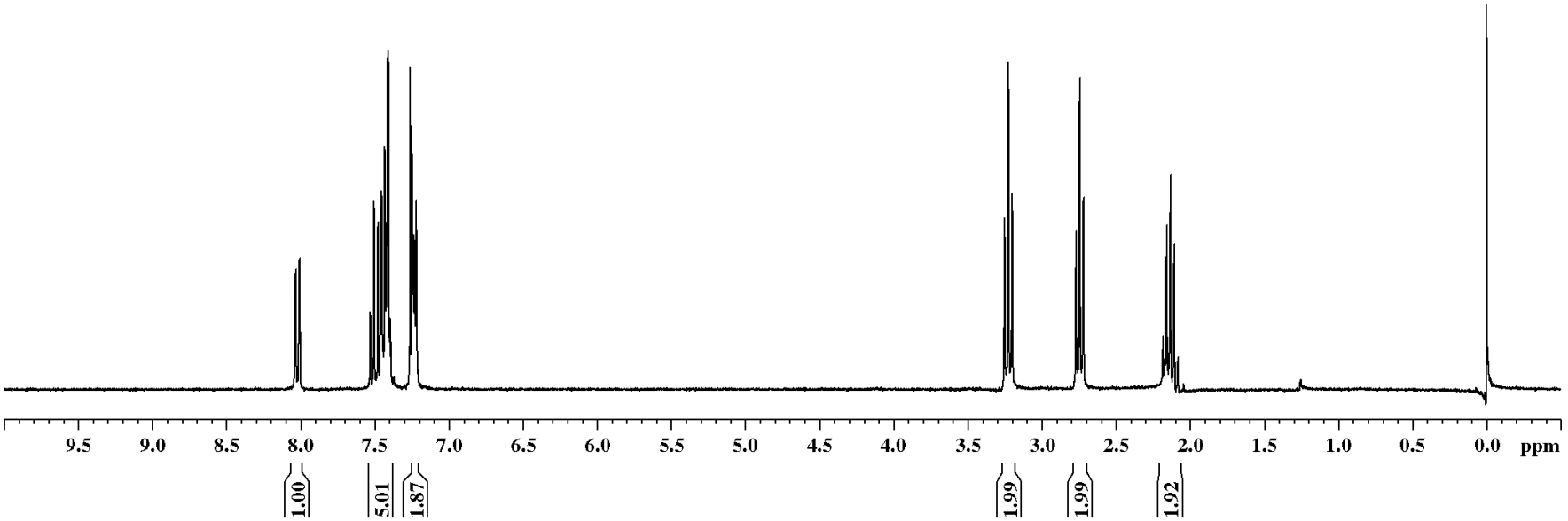
2.082


N
Cl

5m'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3.5
3.0
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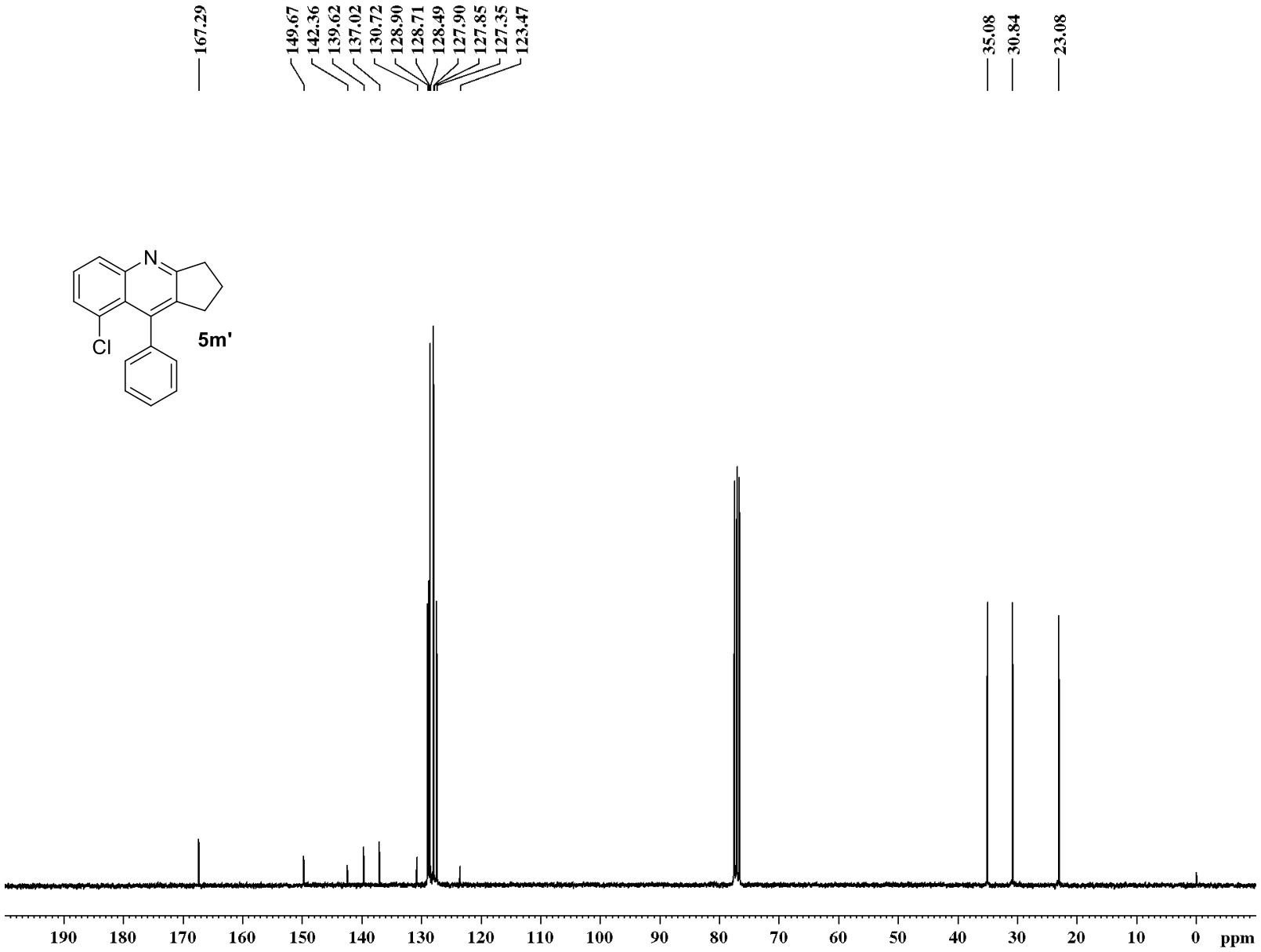
1.99
1.92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127.90
127.85
127.35
123.47
35.08
30.84
23.08
N
Cl
5m'
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140
130
120
110
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90
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60
50
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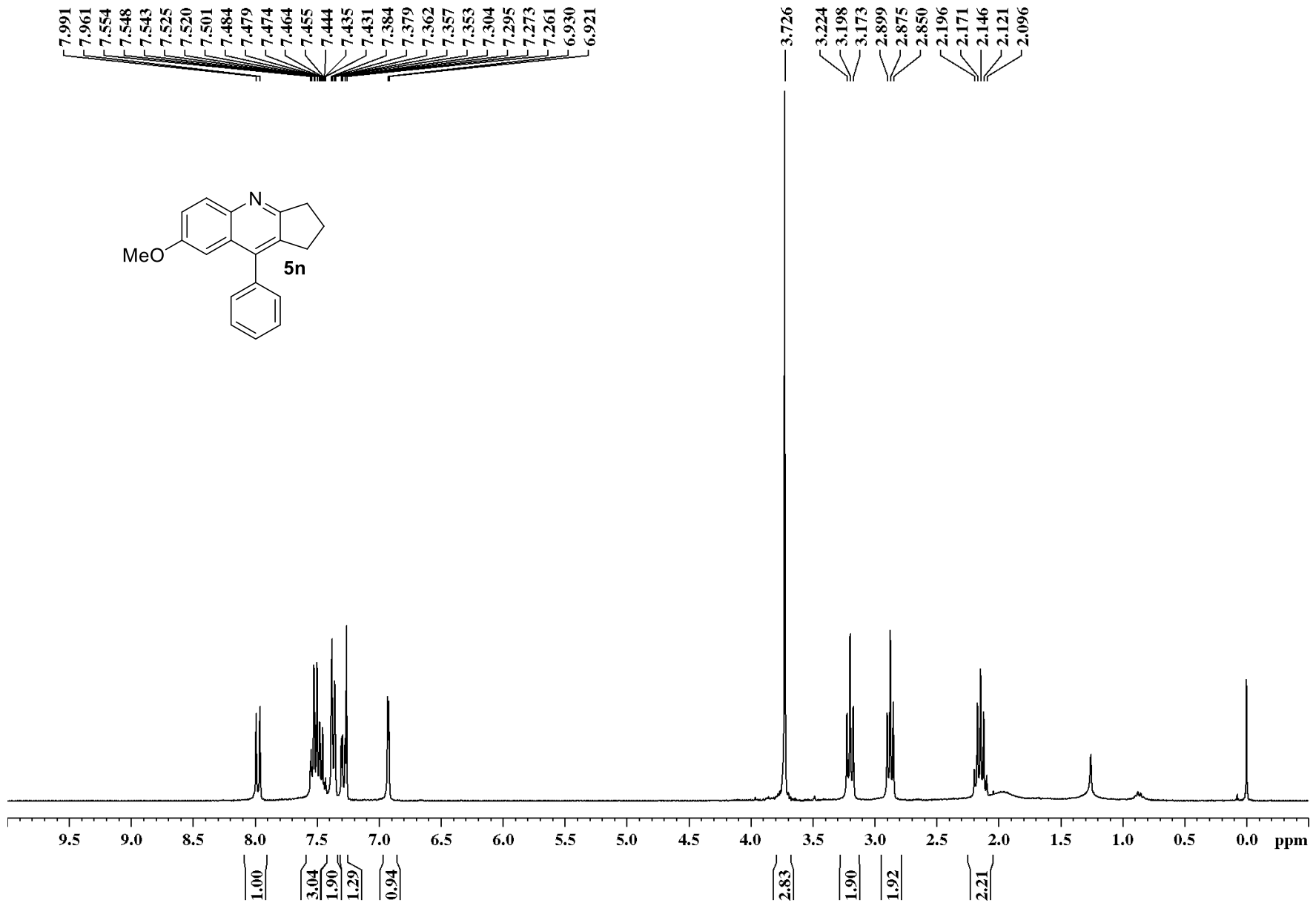
20
10
0
ppm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7.444
7.435
7.431
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7.379
7.362
7.357
7.353
7.304
7.295
7.273
7.261
6.930
6.921
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3.198
3.173
2.899
2.875
2.850
2.196
2.171
2.146
2.121
2.096
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MeO
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8.0
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6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
2.0
1.5
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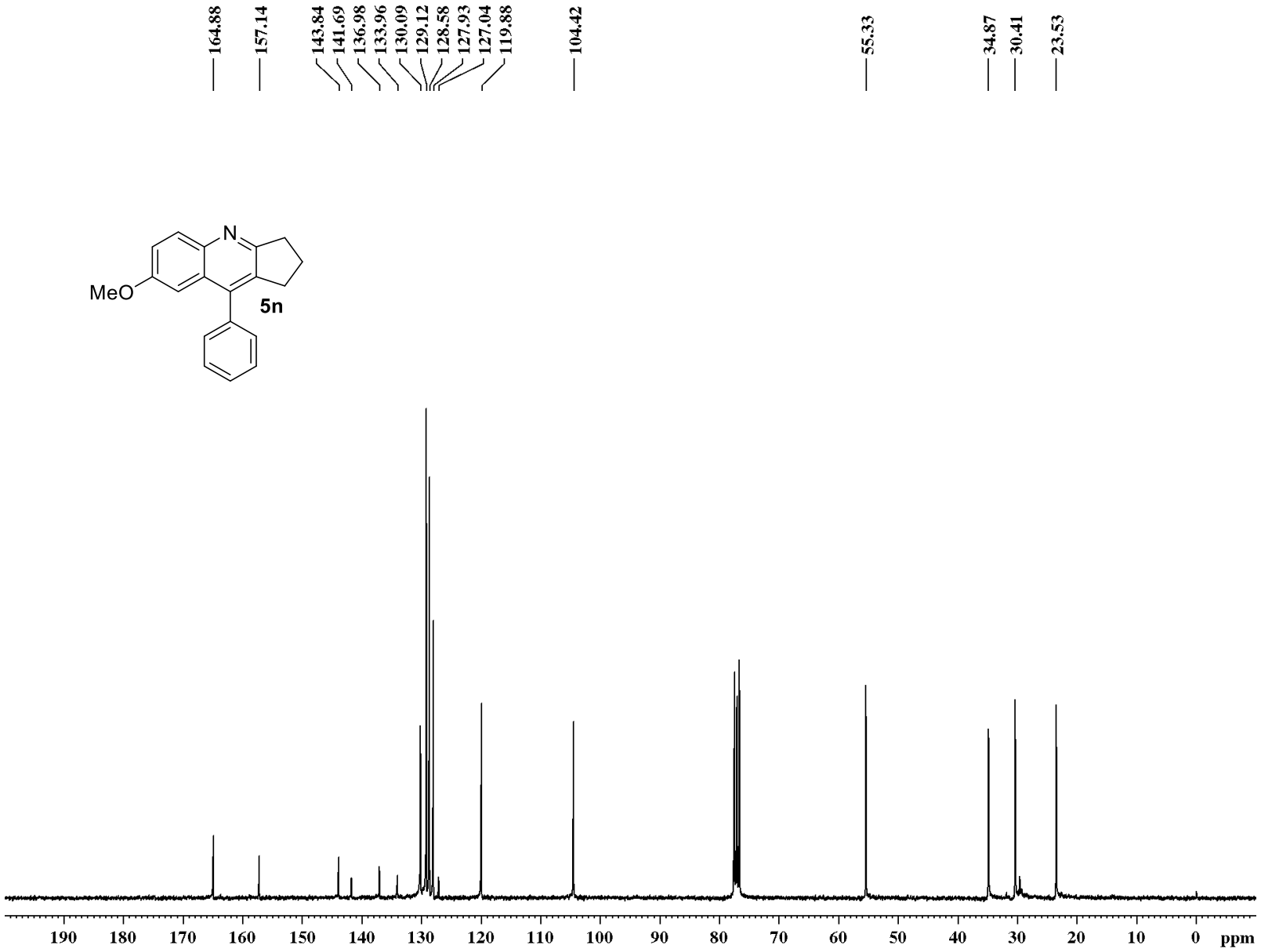
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127.93
127.04
119.88
104.42
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34.87
30.41
23.53
N
MeO
5n
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60
50
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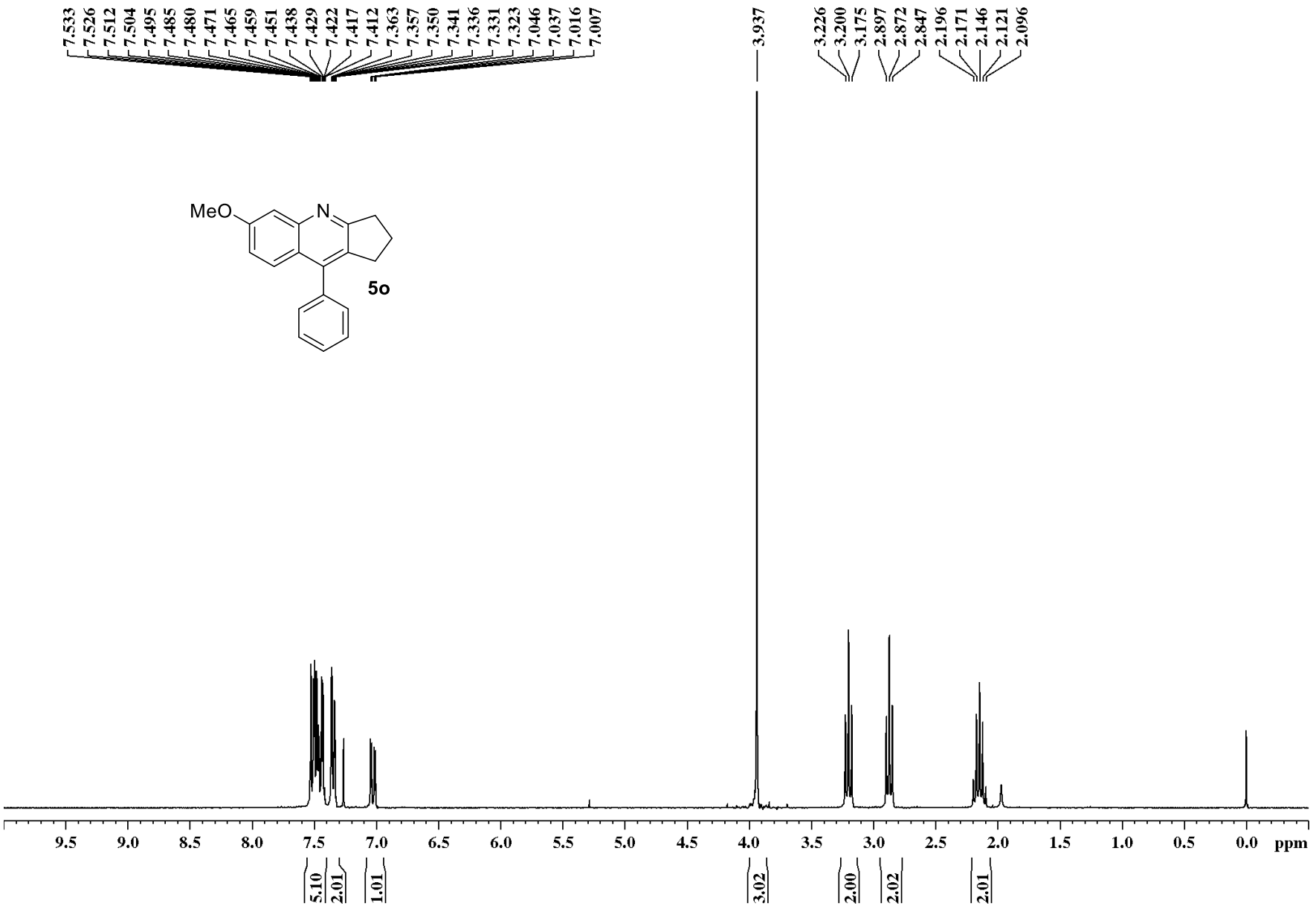
ppm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7.429
7.422
7.417
7.412
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7.350
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7.016
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3.175
2.897
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2.171
2.146
2.121
2.096
MeO
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6.0
5.5
5.0
4.5
4.0
3.5
3.0
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1.01
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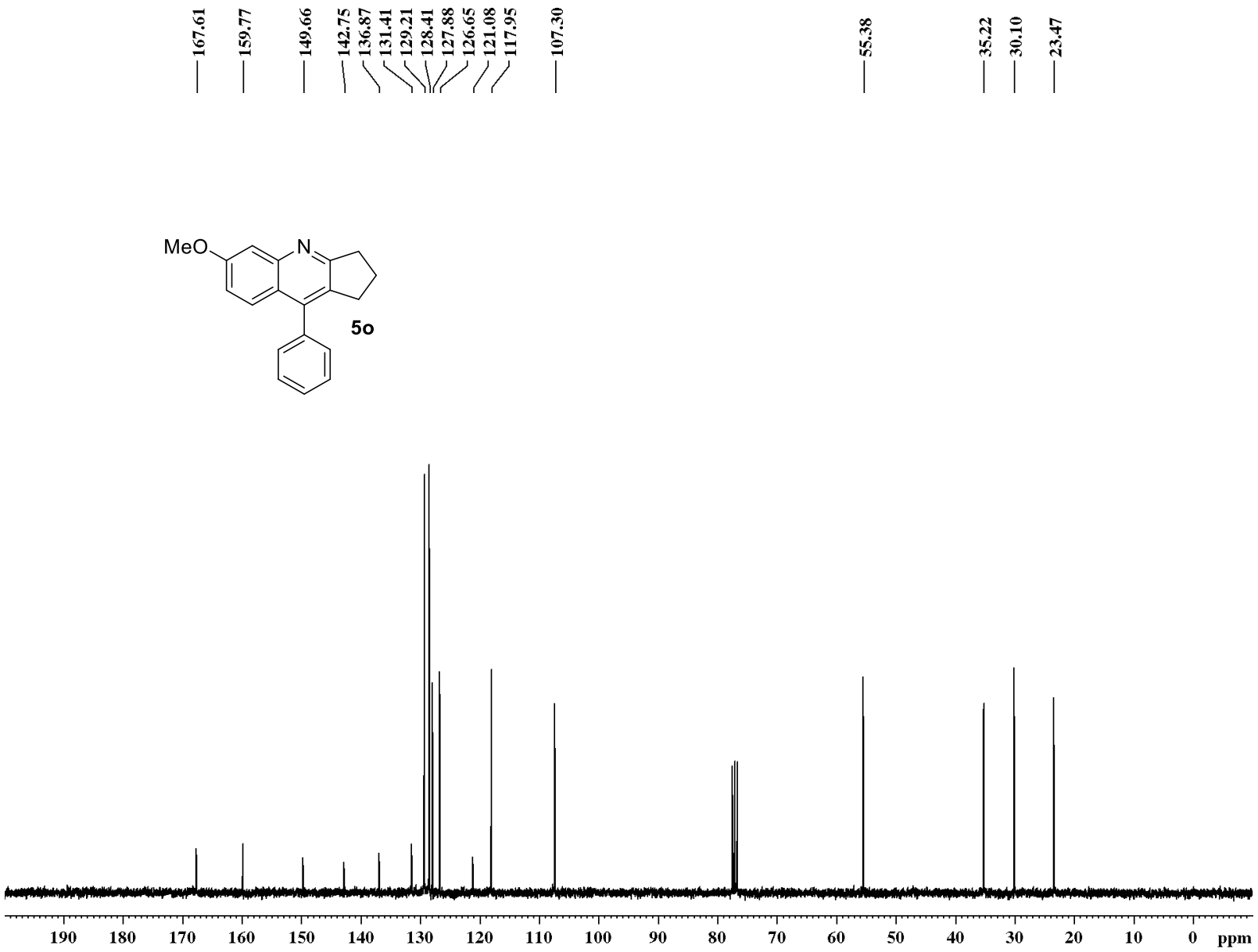
2.02
2.01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128.41
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126.65
121.08
117.95
107.30
55.38
35.22
30.10
23.47
MeO
N
5o
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140
130
120
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100
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60
50
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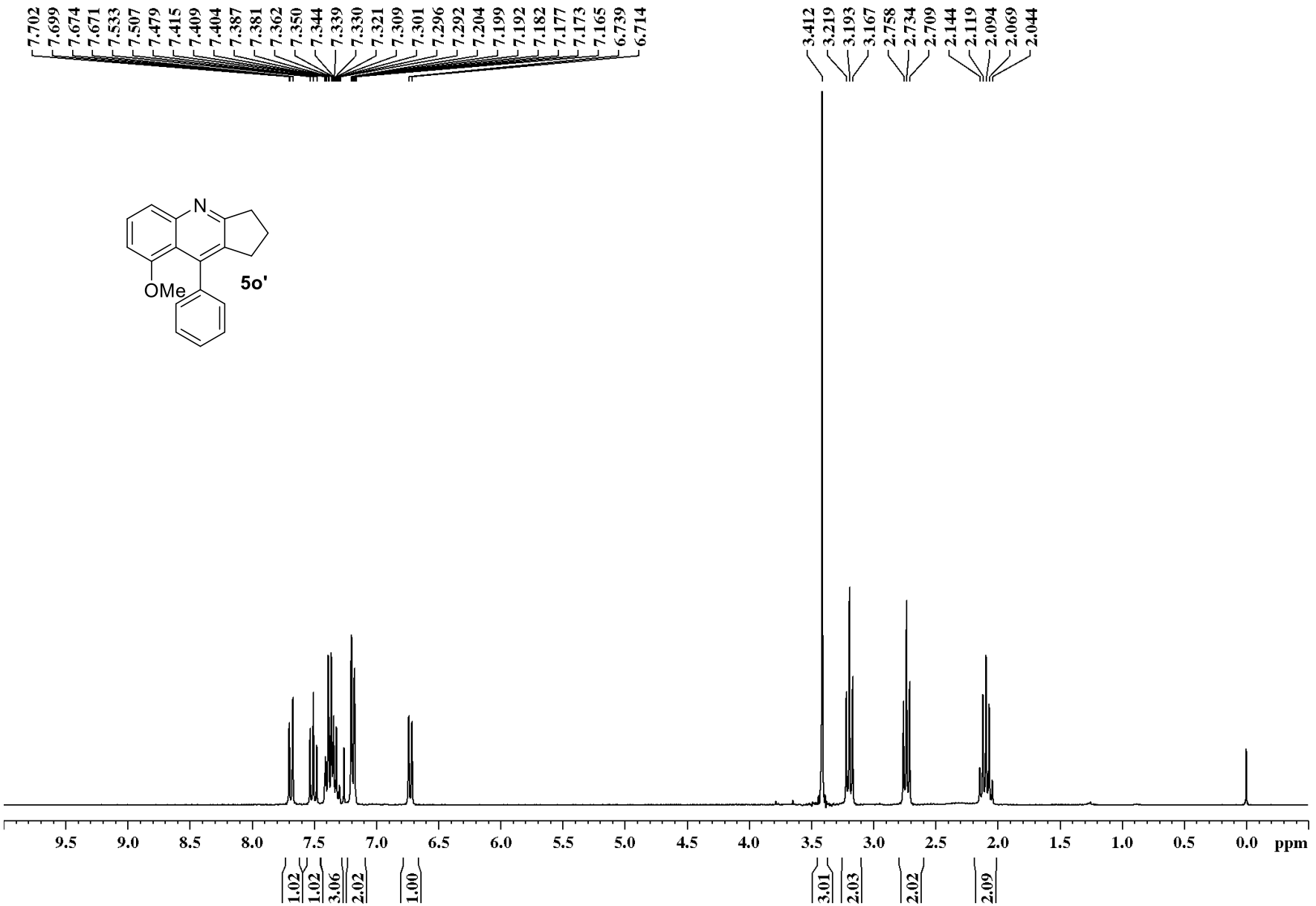
30
20
10
0
ppm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7.321
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7.301
7.296
7.292
7.204
7.199
7.192
7.182
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7.173
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3.219
3.193
3.167
2.758
2.734
2.709
2.144
2.119
2.094
2.069
2.044
N
OMe
5o'
9.5 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 0.0 ppm
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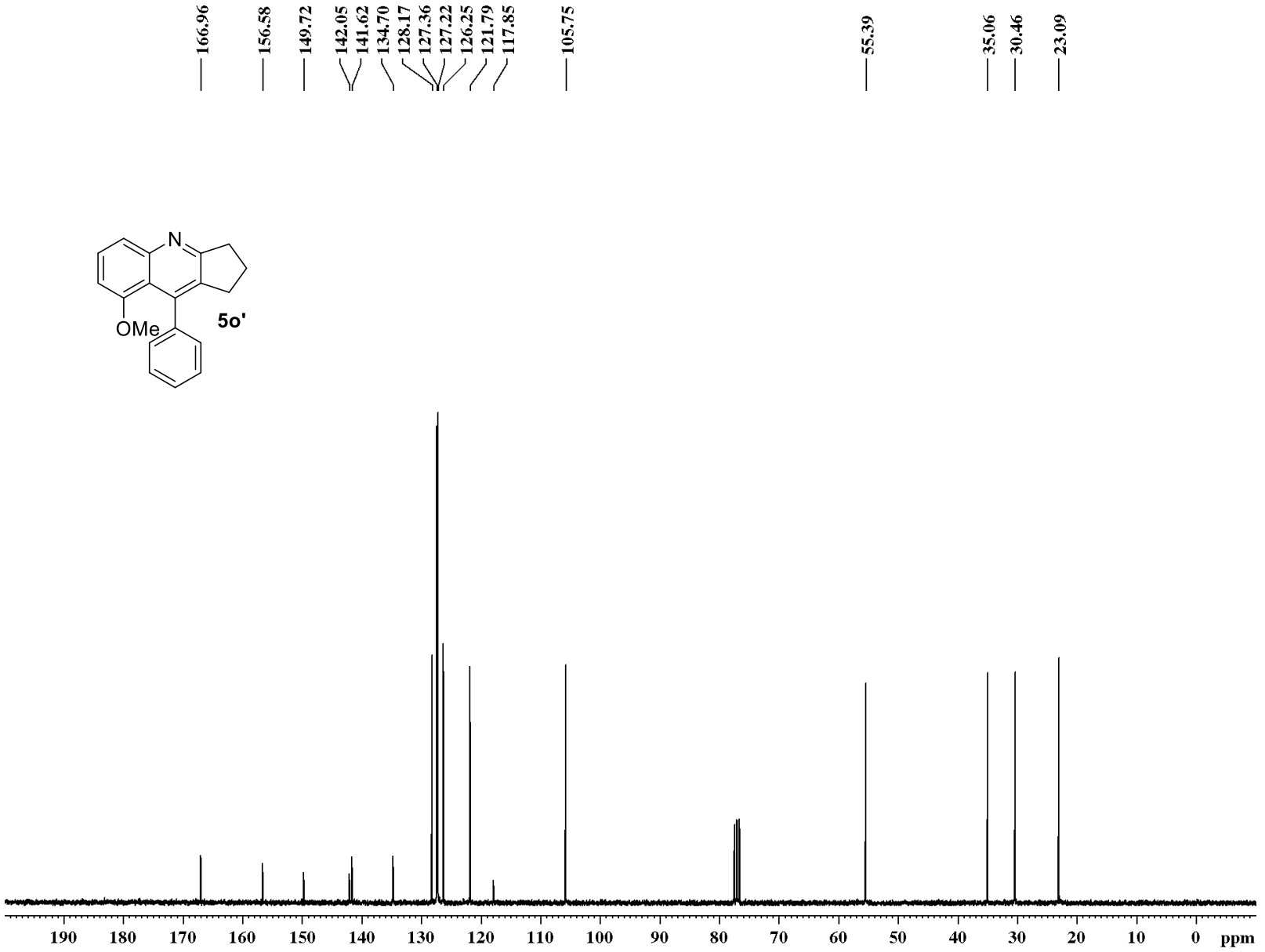
1.02
3.06
2.02
1.00
3.01
2.03
2.02
2.09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127.36
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126.25
121.79
117.85
105.75
55.39
35.06
30.46
23.09
N
OMe
5o'
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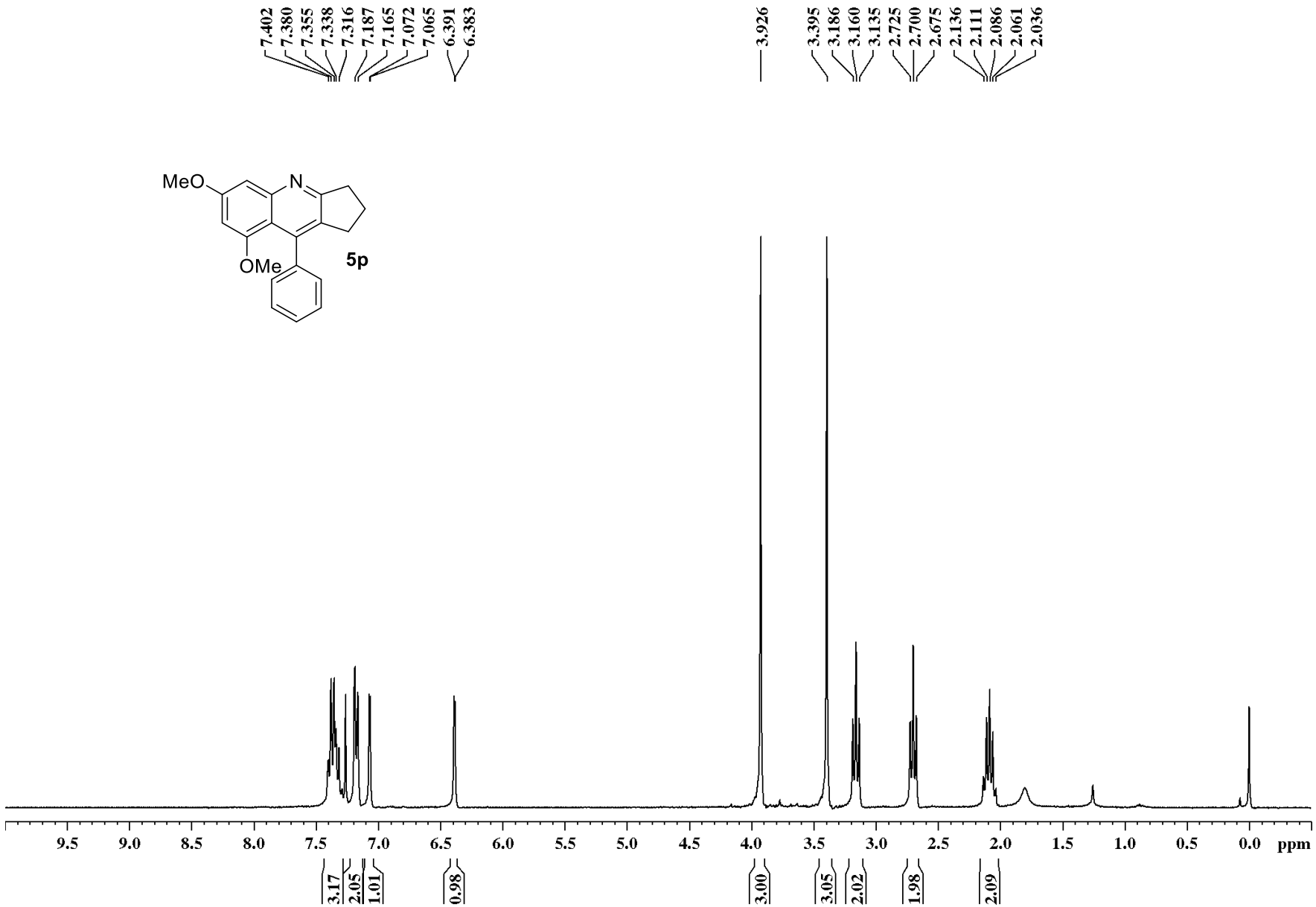
0
ppm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3.395
3.186
3.160
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2.675
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2.111
2.086
2.061
2.036
MeO
N
OMe
5p
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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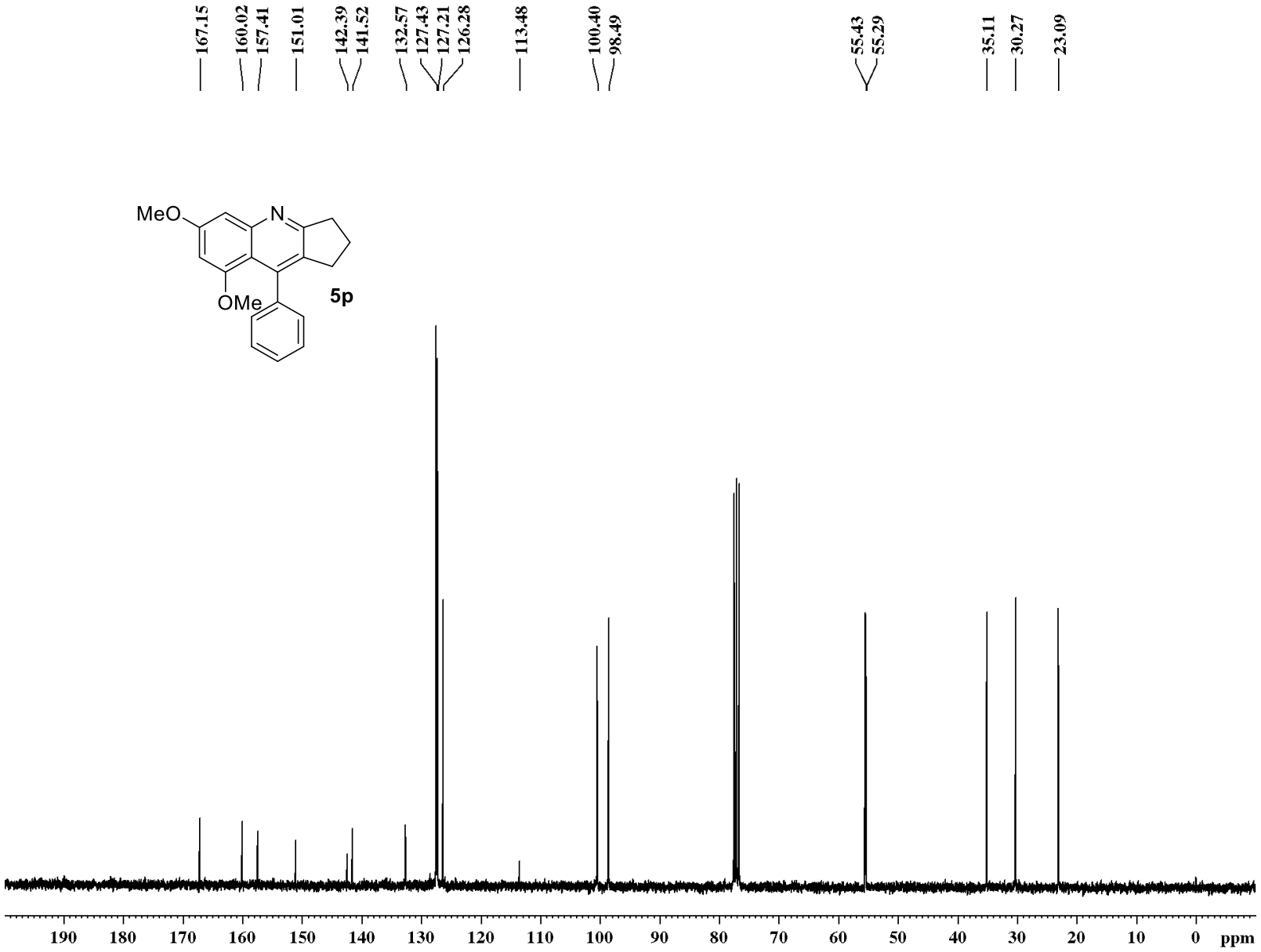
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126.28
113.48
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55.29
35.11
30.27
23.09
MeO
N
OMe
5p
190
180
170
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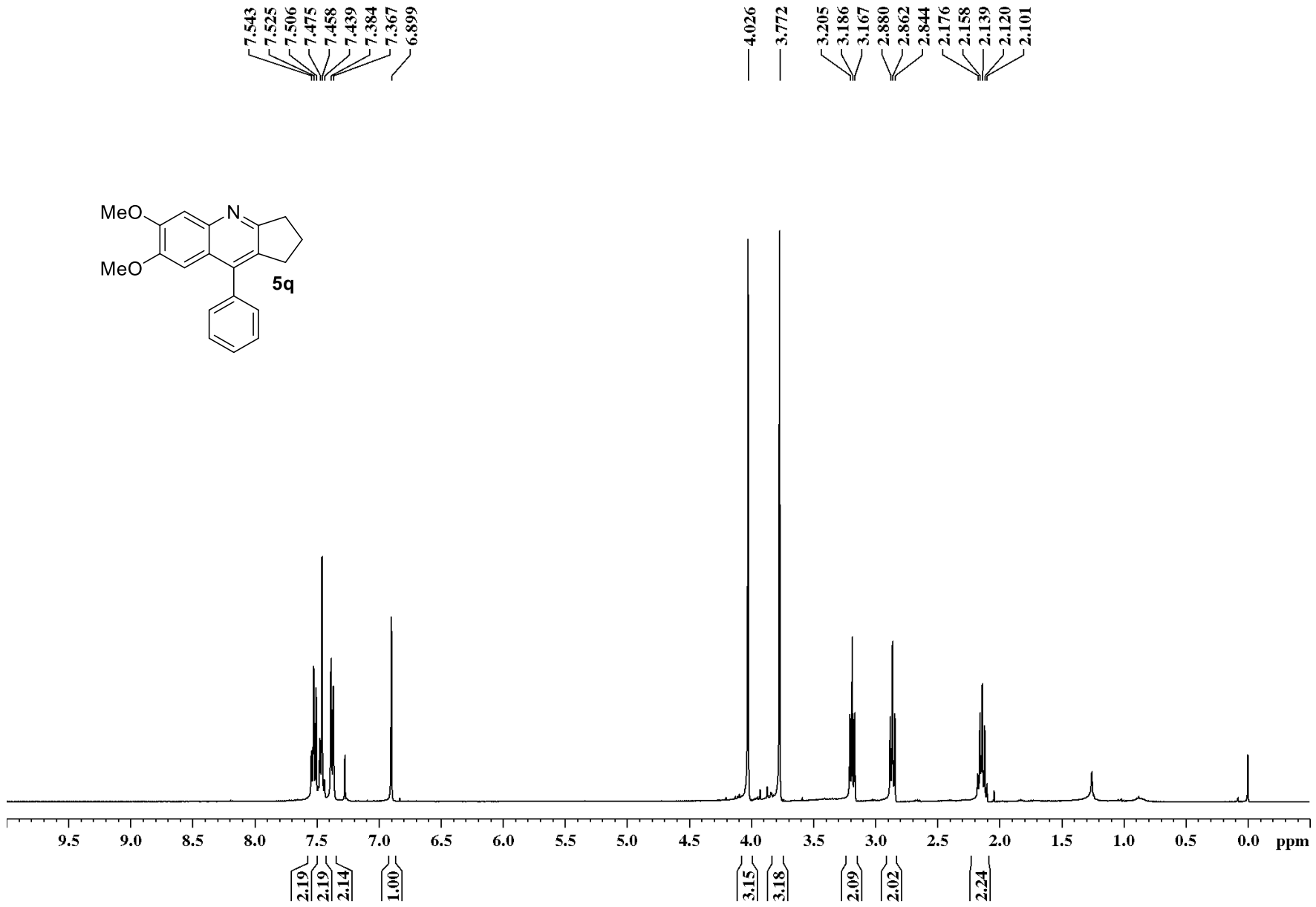
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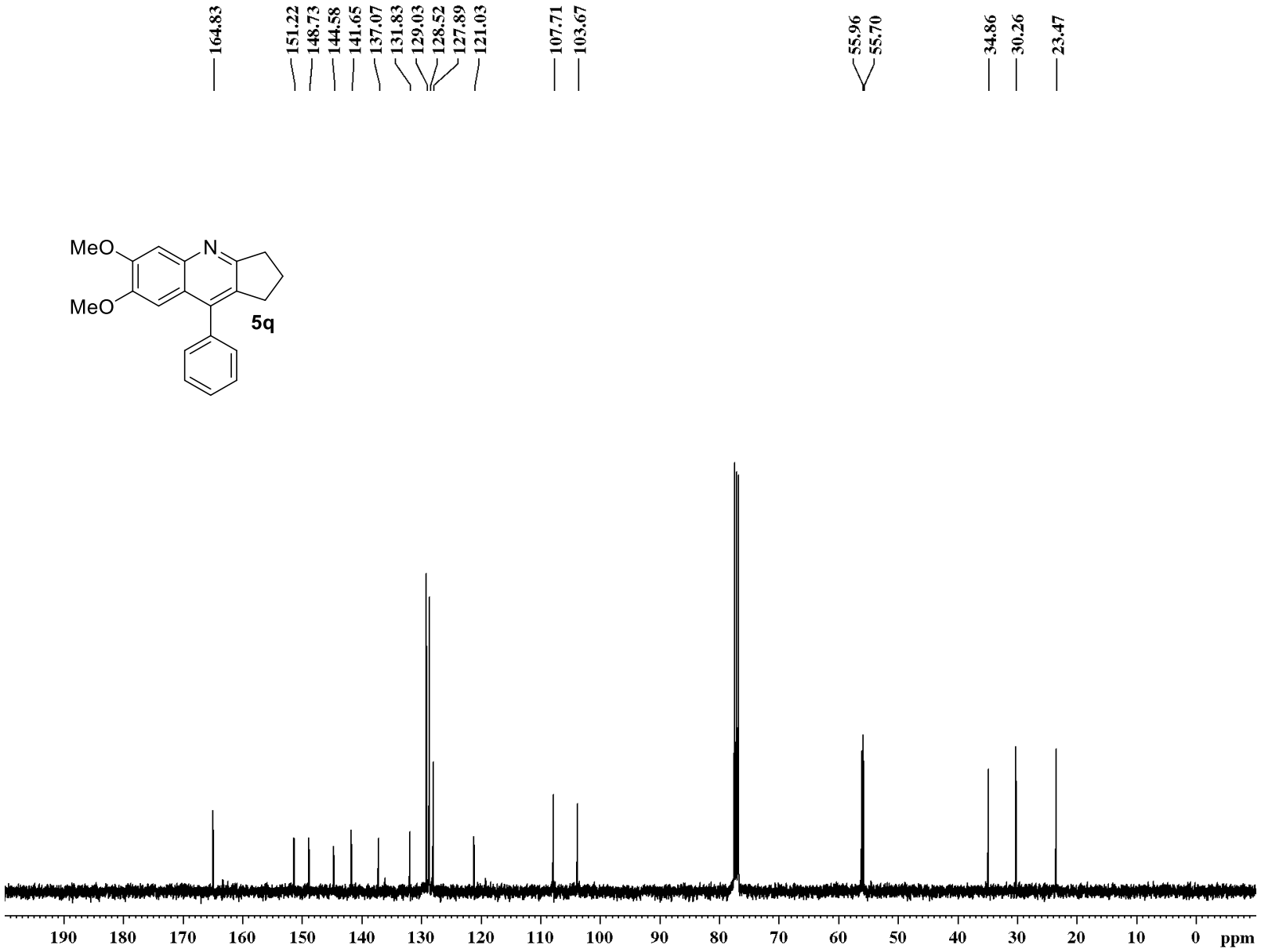
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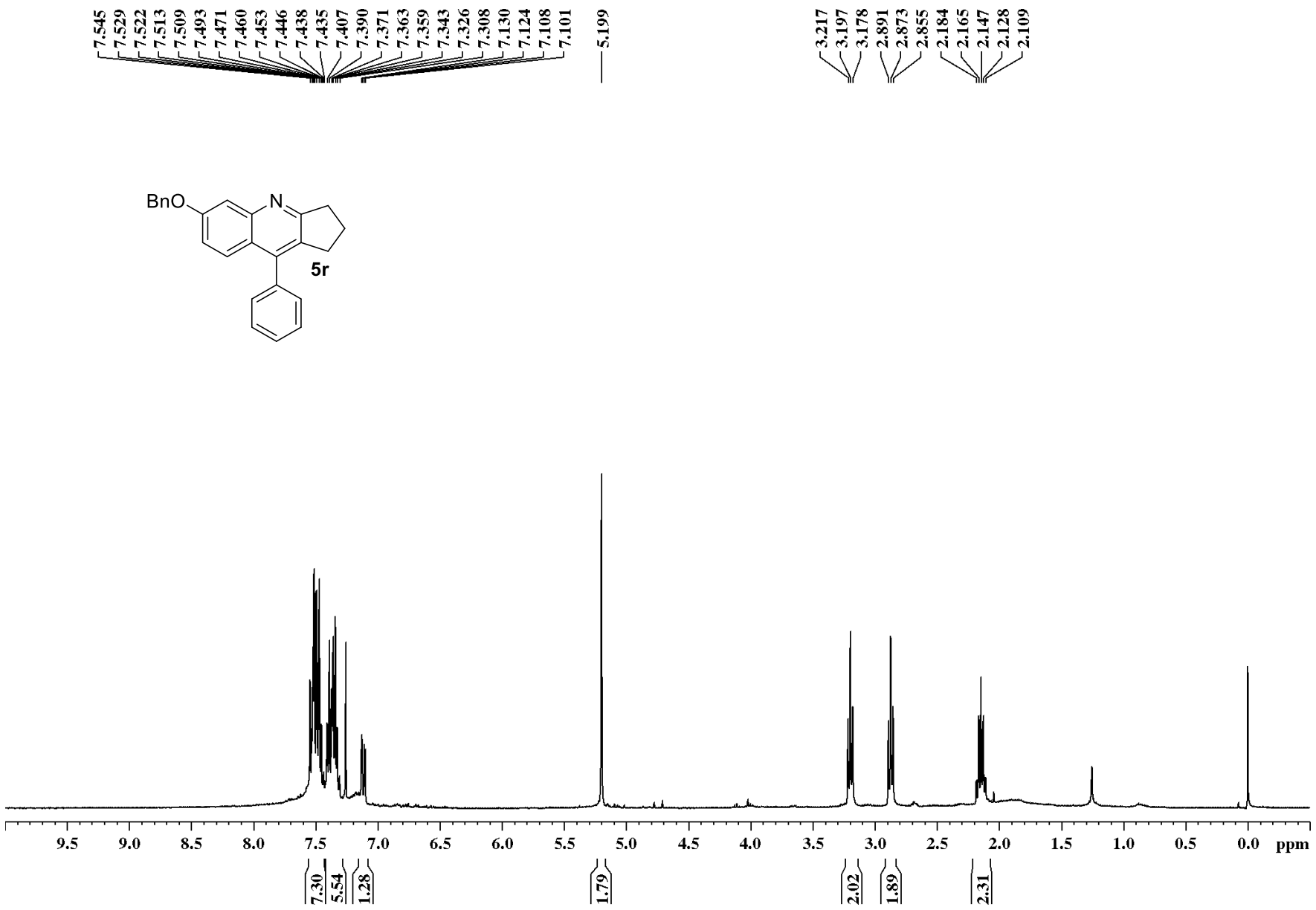
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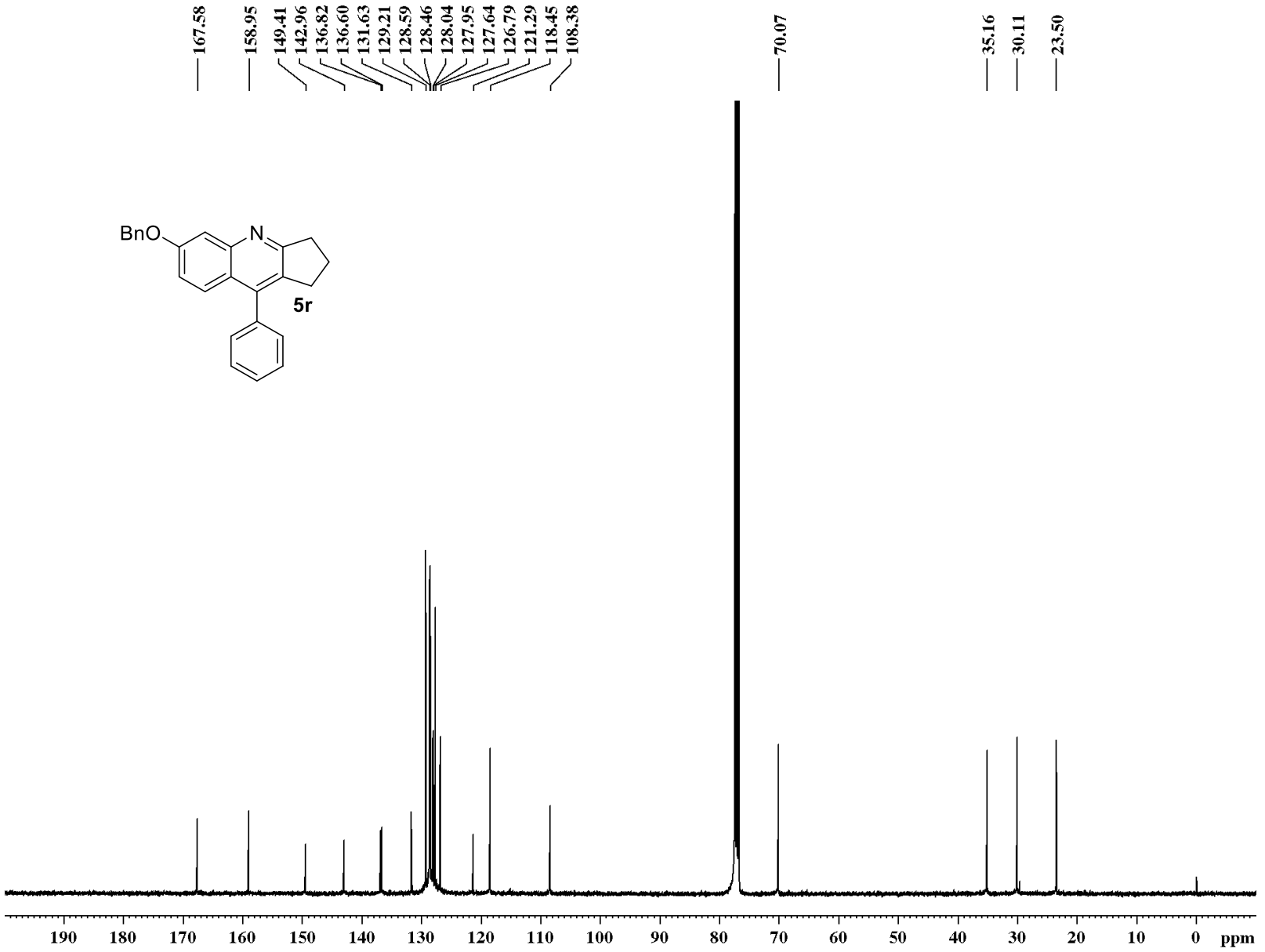
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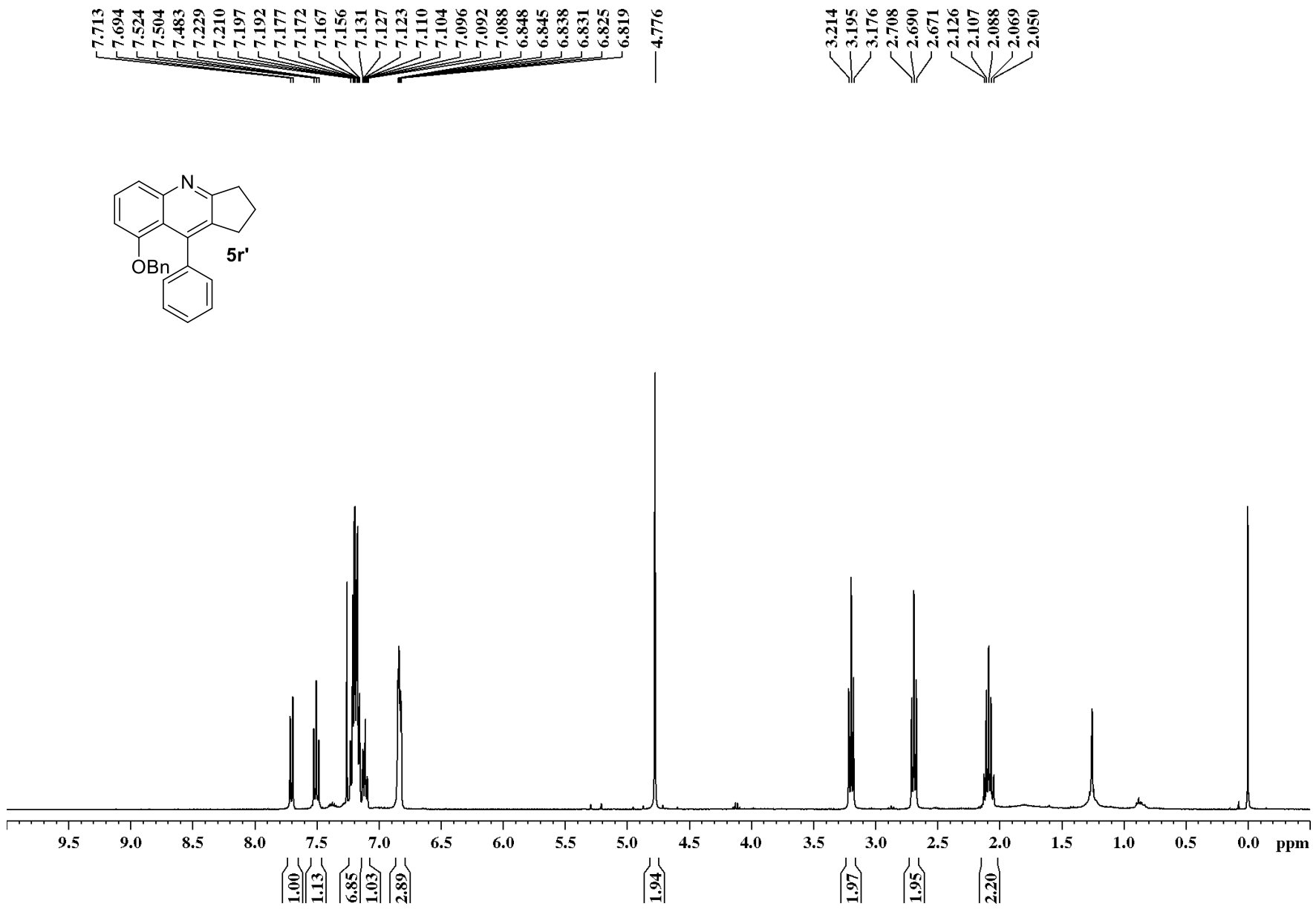
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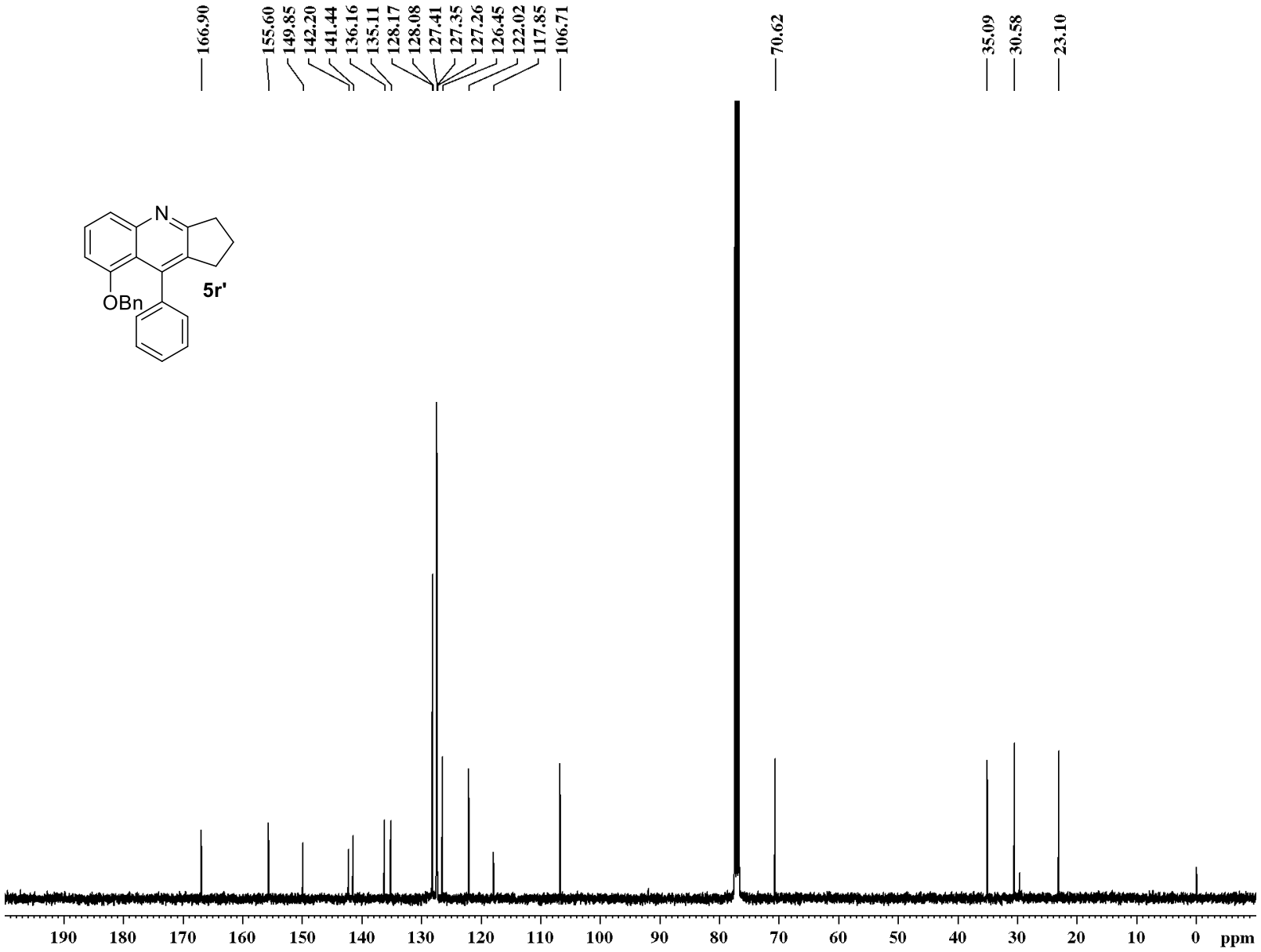
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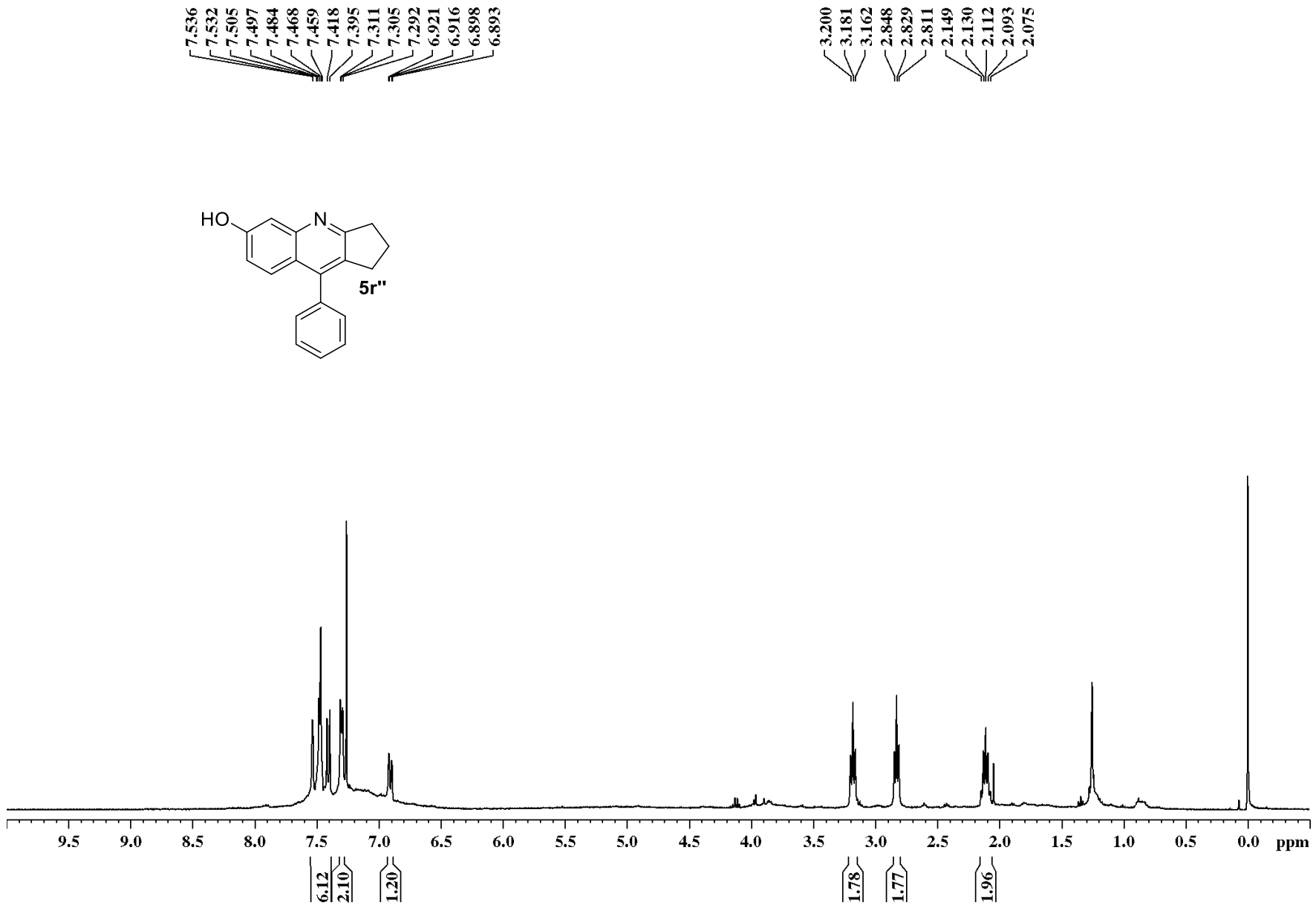
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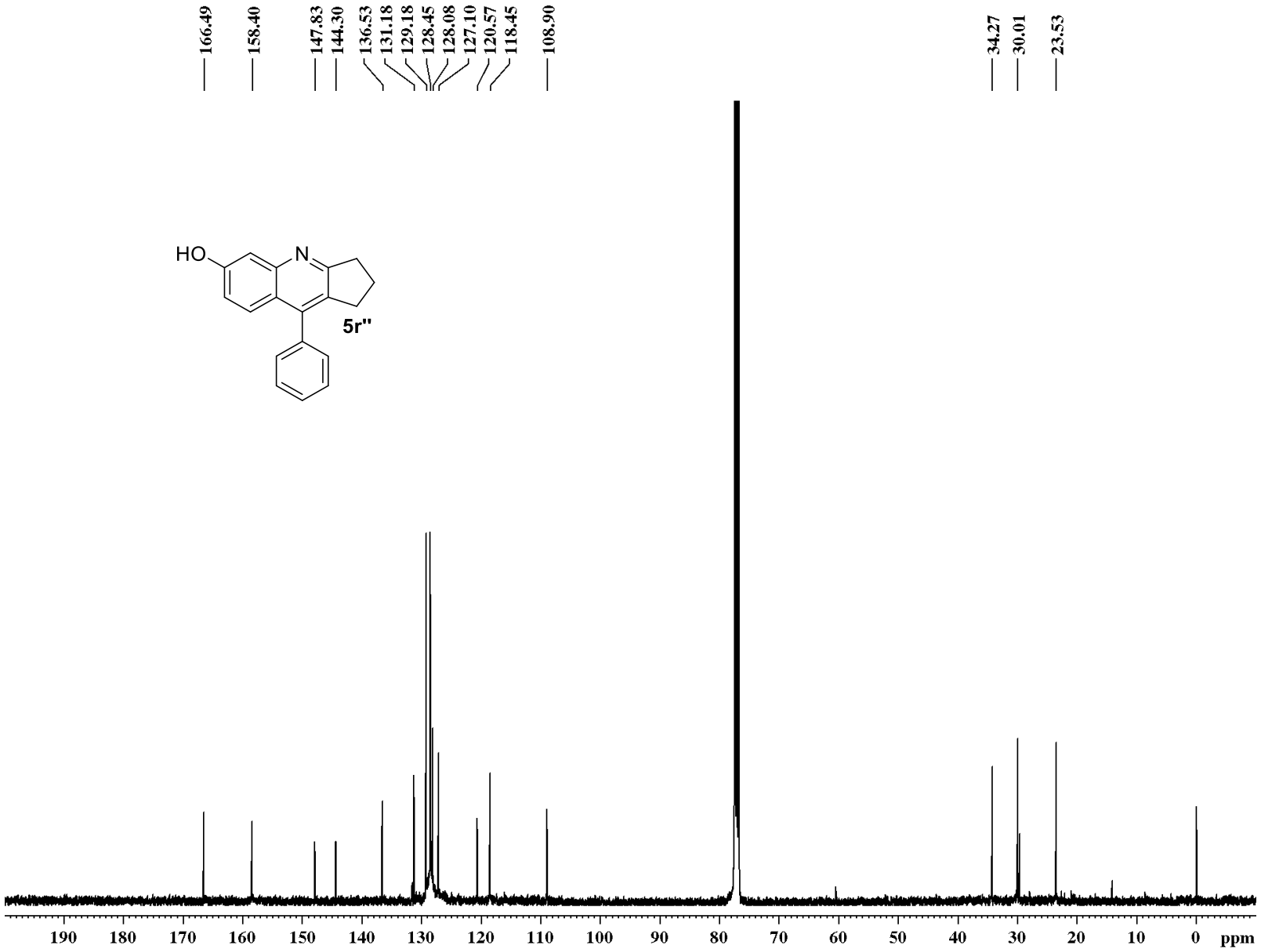
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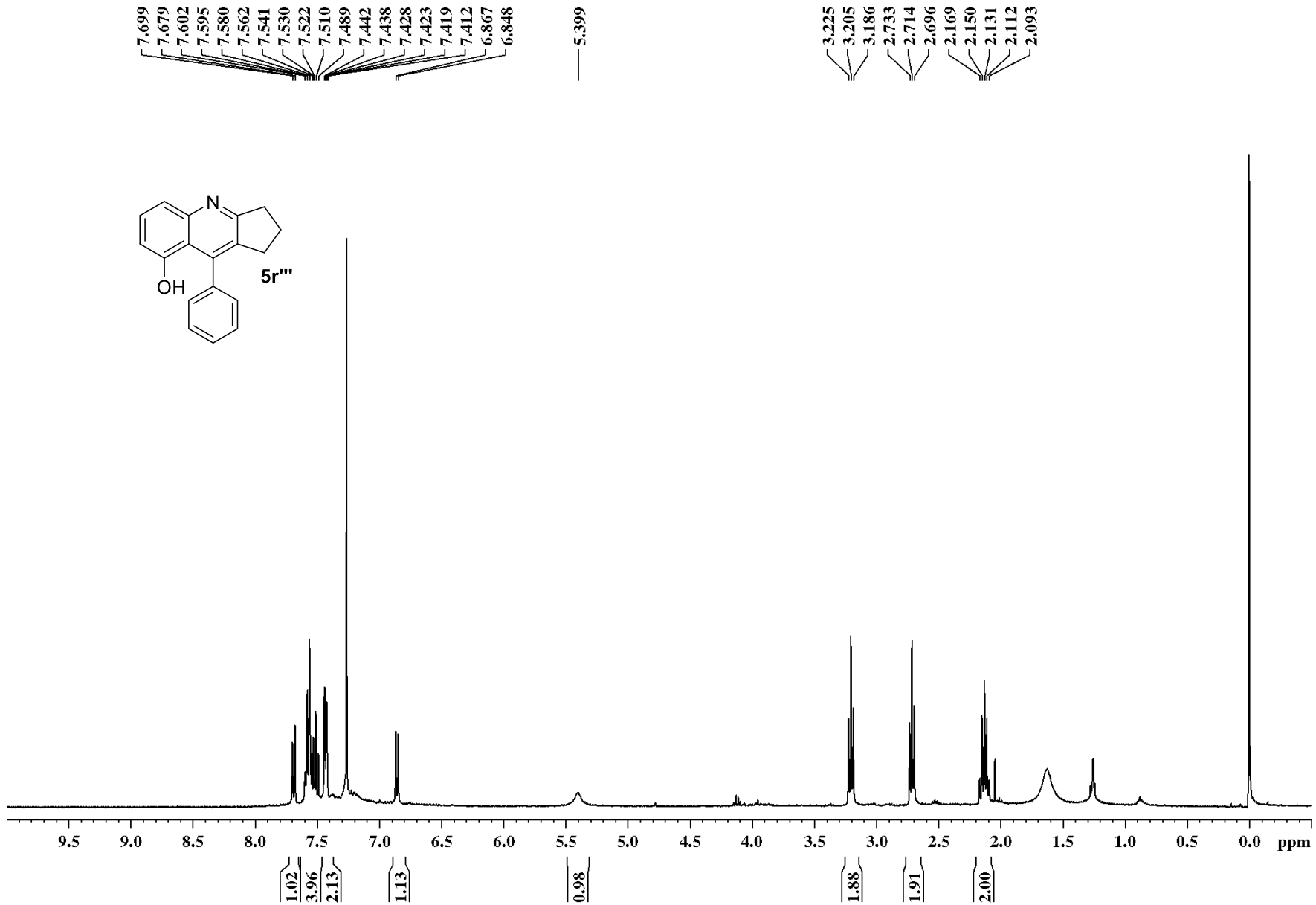
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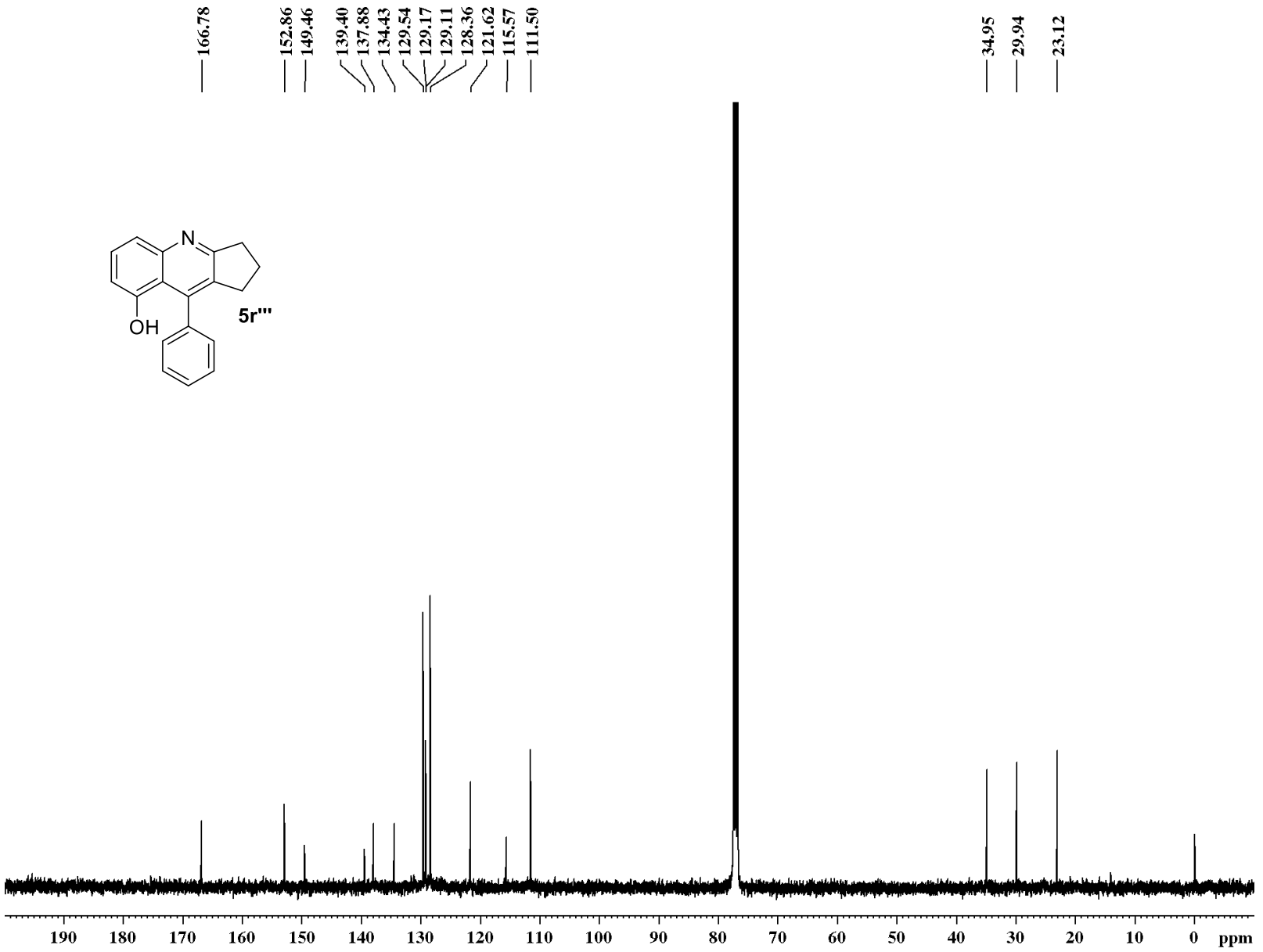
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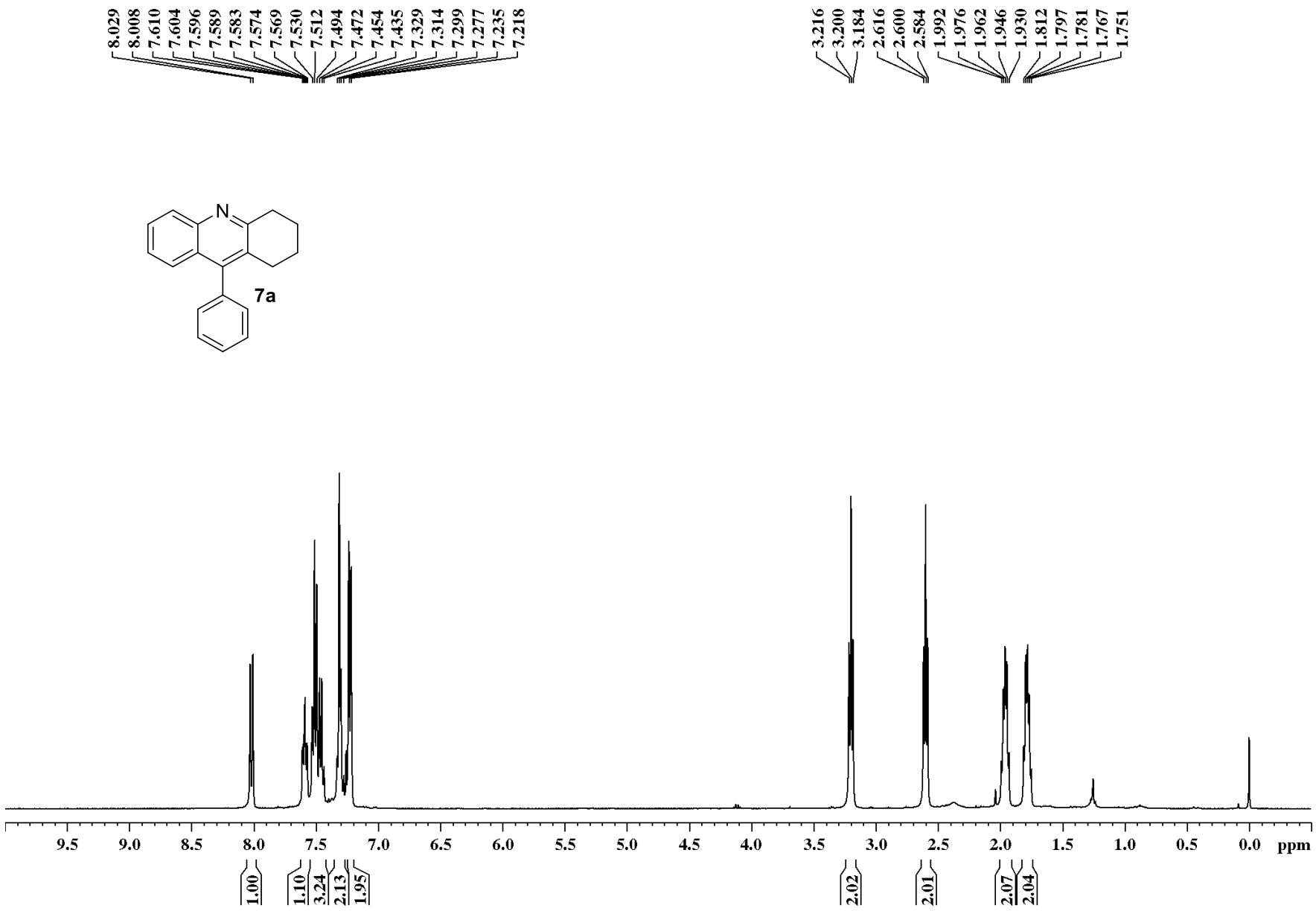
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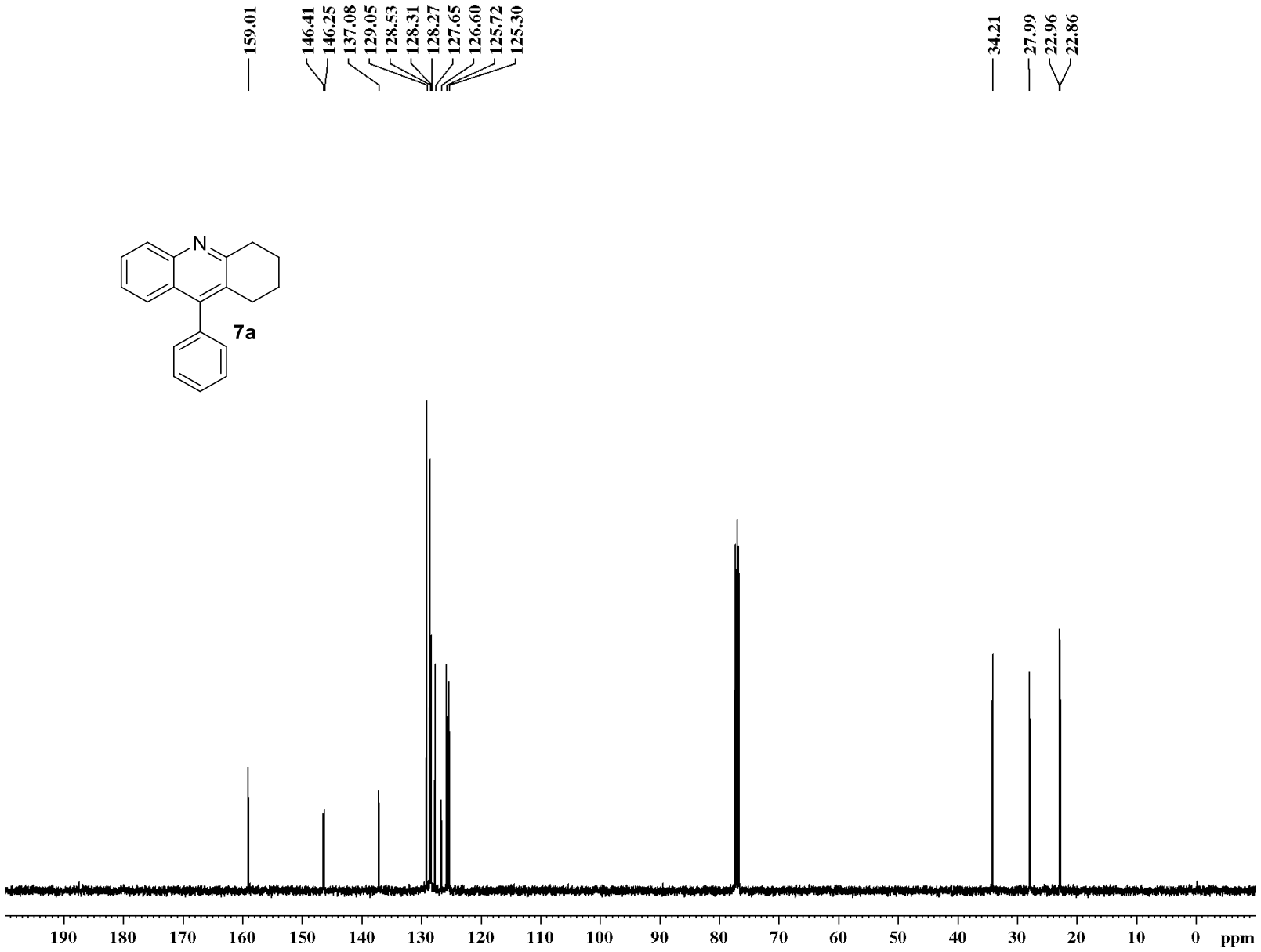
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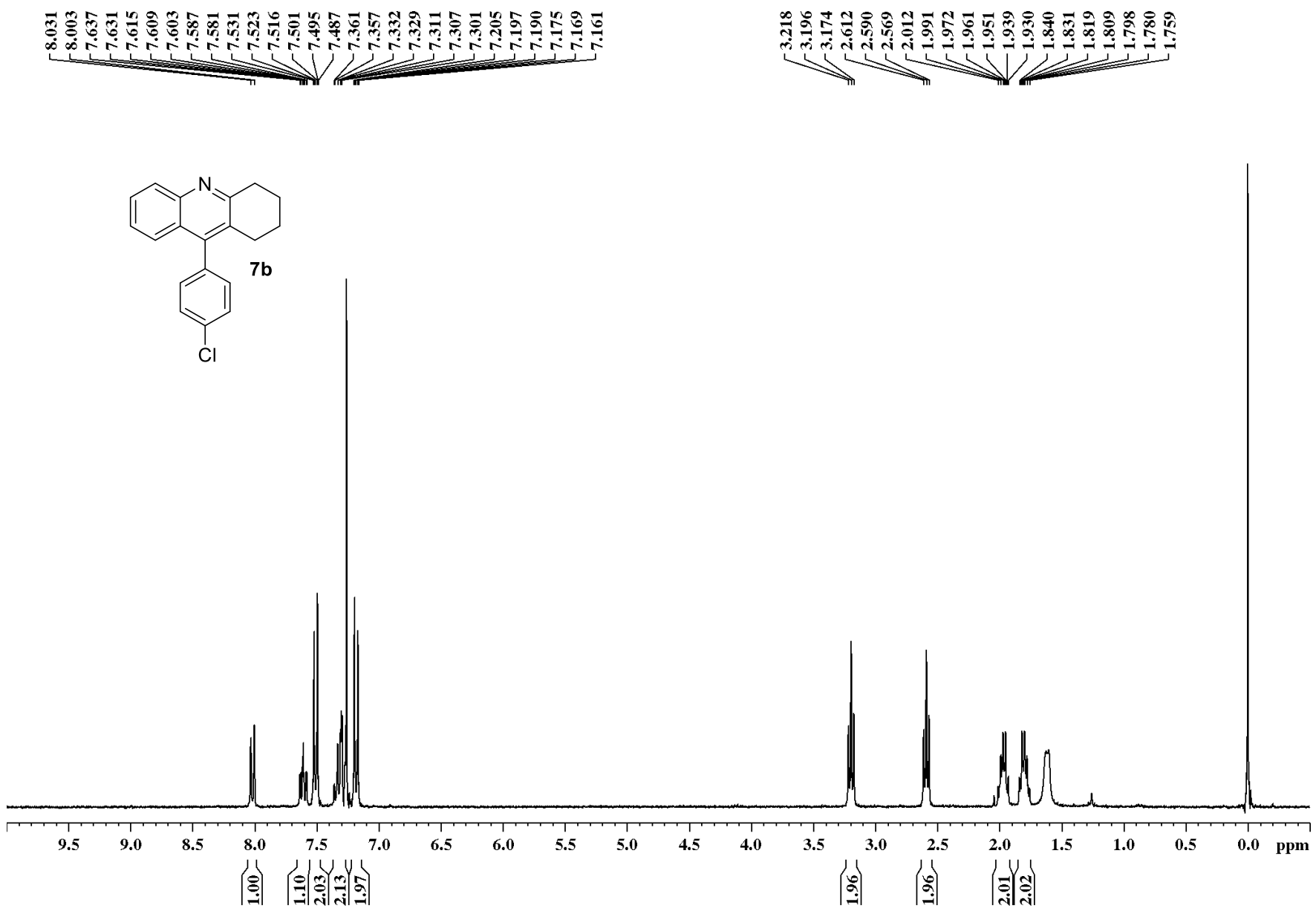
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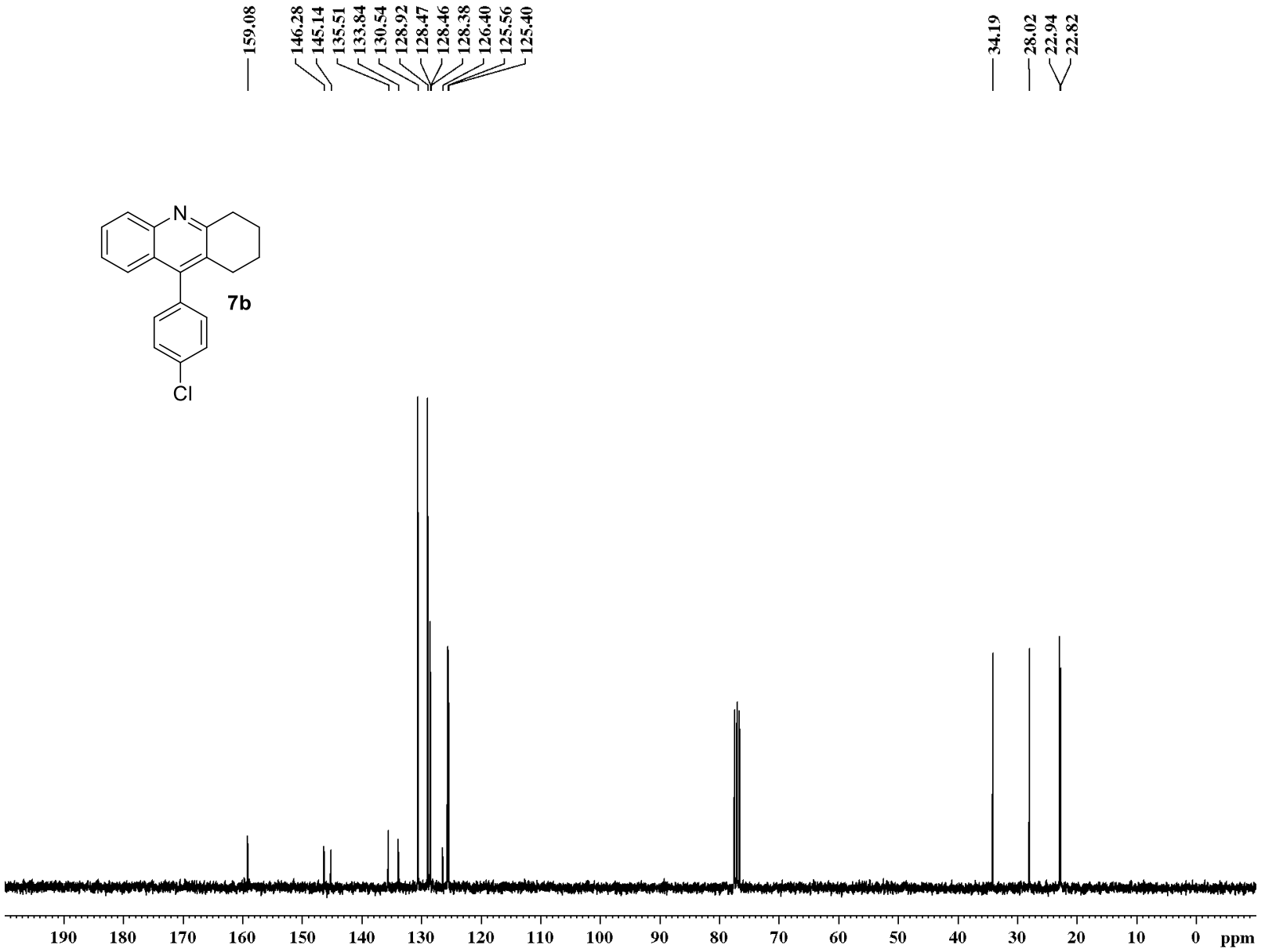
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20
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Optimization of Method for the synthesis of indoloquinolines

Ortho-alkynylarylketone **1a** was employed as the substrate to screen for the optimal condition. To determine the ratio between indoloquinoline product and 1, 5-diketone by-product, we varied equivalent of $TMSN_3$ and TfOH as well as solvent and concentration to improve the yield of the desired product and reduce the formation of by-product. The isolated yield of compound **2a** and **3a** in each entry is summarized in the table below.

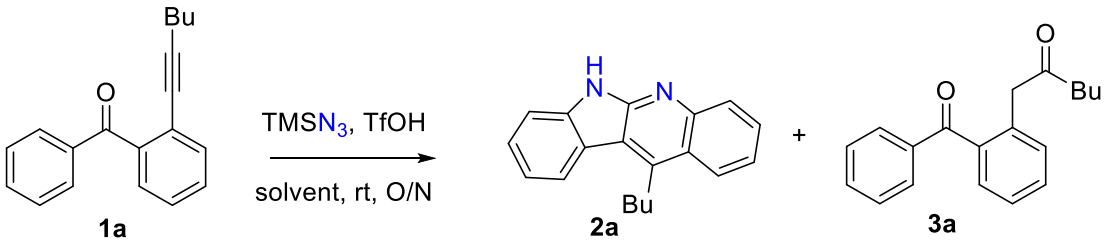

Table S1. Optimization for the synthesis of indoloquinoline **2a** from alkynylarylketone **1a**. [a]Isolated yields.

Entry	Compound 1a (equiv)	$TMSN_3$ (equiv)	TfOH (equiv)	Solvent (dry)	%yield[a]	
					2a	3a
1	1.0	2.5	2.5	0.1 M DCM	48	22
2	1.0	3.0	3.0	0.1 M DCM	47	16
3	1.0	4.0	4.0	0.1 M DCM	50	19
4	1.0	3.5	3.5	0.1 M DCM	49	23
5	1.0	3.5	3.5	0.2 M DCM	54	13
6	1.0	3.5	3.5	0.2 M Toluene	46	14
7	1.0	3.5	3.5	0.3 M DCM	58	8

Optimization of Method for the synthesis of carbocycle-fused quinolines

To screen for the optimal condition, α-alkynylarylketone **4a** was used as the precursor. Compound **4a** was treated under the optimal condition of indoloquinoline method, resulting moderate yield of desired product **5a**. In order to improve the yield of product, we varied various variables ($TMSN_3$, TfOH, solvents and concentrations, time and temperature. The result of the optimizations is shown in Table 2.

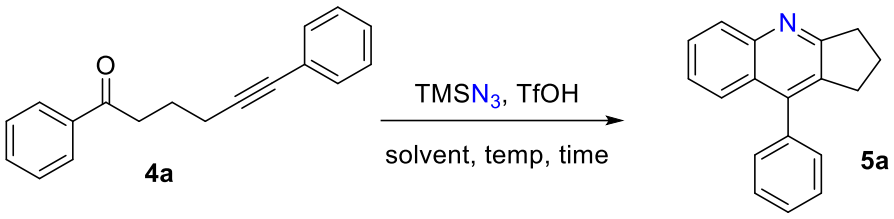

Table S2. Optimization for the carbocycle-fused quinoline **5a** from α-alkynylarylketone **4a**. [a]Isolated yields.

Entry	Compound 4a (equiv)	$TMSN_3$ (equiv)	TfOH (equiv)	Solvent (dry)	Time	%yield[a]
1	1.0	3.5	3.5	0.3 M DCM	O/N, rt	58
2	1.0	1.2	1.2	0.1 M DCM	1 h, rt	77
2	1.0	1.2	1.1	0.1 M Tolulene	1 h, rt	54
3	1.0	1.2	1.2	0.05 M DCM	1 h, rt	71
4	1.0	1.2	1.2	0.2 M DCM	1 h, rt	75
5	1.0	1.2	1.1	0.1 M DCM	1 h, rt	65
6	1.0	1.0	1.0	0.1 M DCM	1 h, rt	63
7	1.0	1.2	1.2	0.1 M DCM	3 h, rt	72
8	1.0	1.4	1.4	0.1 M DCM	1 h, rt	75
9	1.0	2.0	2.0	0.1 M DCM	1 h, 0 °C	67