Electronic Supplementary Material (ESI) for RSC Advances.

Microwave-Assisted Green Construction of Imidazole-Fused Hybrid Scaffolds Using 2-Aminobenzimidazoles as Building Blocks

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1. **Synthesis and characterization of 2-(2-bromoaryl)- and 2-(2-bromovinyl)imidazoles 1.**

To a 25 mL round-bottom flask was added 2-bromobenzaldehyde or β-bromo-α,β-unsaturated aldehyde (5 mmol), 1,2-diarylethane-1,2-dione (5 mmol), ammonium acetate (20 mmol), acetic acid (1.3 mL), and ethanol (12.5 mL). After the reaction mixture was stirred for 4 h under reflux condition and cooled down to room temperature, solvents were removed under reduced pressure. The residual solid was purified by column chromatography (dichloromethane/MeOH = 99/1) to give **1**. All new starting compounds were characterized spectroscopically as shown below.[1]

2-(2-Bromophenyl)-4,5-di-*o*-tolyl-1*H*-imidazole (1e): Yield 58% (1.17 g), white solid. mp 193-195 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.51 (s, 1H), 7.77 (dd, J = 8.0 and 0.9 Hz, 1H), 7.75 (dd, J = 7.7 and 1.6 Hz, 1H), 7.52-7.49 (m, 1H), 7.38-7.35 (m, 1H), 7.25-7.22 (m, 2H), 7.19-7.11 (m, 4H), 7.05-7.01 (m, 2H), 2.24 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 143.8, 138.3, 136.5, 136.3, 134.8, 133.5, 132.3, 131.6, 130.8, 130.6, 130.3, 130.2 (130.21), 130.2 (130.18), 130.0, 128.0 (128.02), 128.0 (128.00), 127.7, 126.8, 125.7, 125.2, 121.3, 20.2, 19.8. HRMS (EI): m/z [M^+] calcd for $C_{23}H_{19}BrN_2$: 402.0732, Found 402.0732.

2-(2-Bromophenyl)-4,5-di(furan-2-yl)-1*H*-imidazole (1j): Yield 40% (0.71 g), brown solid. mp 102-104 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.87 (s, 1H) , 7.78 (dd, J = 6.5 and 0.6 Hz, 1H), 7.72 (s, 1H), 7.66 (dd, J = 7.6 and 1.6 Hz, 1H), 7.53-7.49 (m, 1H), 7.44-7.40 (m, 1H), 6.94 (d, J = 2.7 Hz, 1H), 6.73 (d, J = 2.5 Hz, 1H), 6.65 (s, 1H), 6.58 (s, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 149.3, 145.4, 144.4, 142.6, 141.8, 133.1, 132.0, 131.9, 130.9, 128.9, 127.6, 122.0, 119.0, 111.8, 111.4, 108.1, 106.8. HRMS (EI): m/z [M^+] calcd for $C_{17}H_{11}BrN_2O_2$: 354.0004, Found 354.0004.

2-(1-Bromonaphthalen-2-yl)-4,5-diphenyl-1*H*-imidazole (1k): Yield 51% (1.08 g), yellow solid. mp 179-181 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.78 (s, 1H), 8.37 (d, J = 8.5 Hz, 1H), 8.10-8.07 (m, 2H), 7.84 (d, J = 5.7 Hz, 1H), 7.79-7.75 (m, 1H), 7.71-7.67 (m, 1H), 7.60-7.53 (m, 4H), 7.46-7.43 (m, 2H), 7.38-7.31 (m, 3H), 7.25 (d, J = 6.7 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 145.3, 136.9, 135.2, 133.8, 131.8, 130.9, 130.7, 128.7, 128.5 (128.49), 128.5 (128.47), 128.4, 128.2 (128.23), 128.2 (128.17), 128.0, 127.9, 127.7, 127.5, 127.3, 127.2, 126.6, 121.9. HRMS (EI): m/z [M^+] calcd for $C_{25}H_{17}BrN_2$: 424.0575, Found 424.0573.

2-(2-Bromocyclohex-1-en-1-yl)-4,5-diphenyl-1*H*-imidazole (1l): Yield 61% (1.16 g), white solid. mp 227-229 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.23 (s, 1H), 7.49-7.18 (m, 10H), 2.65-2.63 (m, 2H), 2.57-2.54 (m, 2H), 1.76-1.74 (m, 4H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 145.7, 136.0, 135.3, 131.0, 128.9, 128.6, 128.1, 128.0, 127.5, 127.1, 126.7, 126.4, 122.5, 36.9, 31.3, 24.0, 21.6. HRMS (EI): m/z [M^+] calcd for $C_{21}H_{19}BrN_2$: 378.0732, Found 378.0730.

(Z)-2-(1-Bromo-1-phenylprop-1-en-2-yl)-4,5-diphenyl-1*H*-imidazole (1m): Yield 67% (1.39 g), white

solid. mp 214-217 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.73 (s, 1H), 7. 50-7.15 (m, 15H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 144.4, 140.4, 136.1, 134.9, 130.9, 129.4, 128.8, 128.6, 128.2, 128.0 (128.04), 128.0 (128.00), 127.8, 127.6, 127.0, 126.8, 126.4, 124.9, 24.0. HRMS (EI): *m*/*z* [M^+] calcd for $C_{24}H_{19}BrN_2$: 414.0732, Found 414.0729.

(Z)-2-(1-Bromo-3-methyl-1-phenylbut-1-en-2-yl)-4,5-diphenyl-1*H*-imidazole (1n): Yield 23% (0.51 g), white solid. mp 164-167 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.81 (s, 1H), 7.43-7.41 (m, 2H), 7.33-7.16 (m, 11H), 7.10-7.08 (m, 2H), 3.41 (sept, 1H), 1.18 (d, J = 6.9 Hz, 6H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 141.7, 140.1, 139.1, 135.9, 135.2, 131.0, 129.0, 128.5, 128.1, 127.9, 127.8, 127.6, 127.4, 126.9, 126.5, 126.3, 124.4, 35.4, 20.3. HRMS (EI): *m*/*z* [M^+] calcd for $C_{26}H_{23}BrN_2$: 442.1045, Found 442.1043.

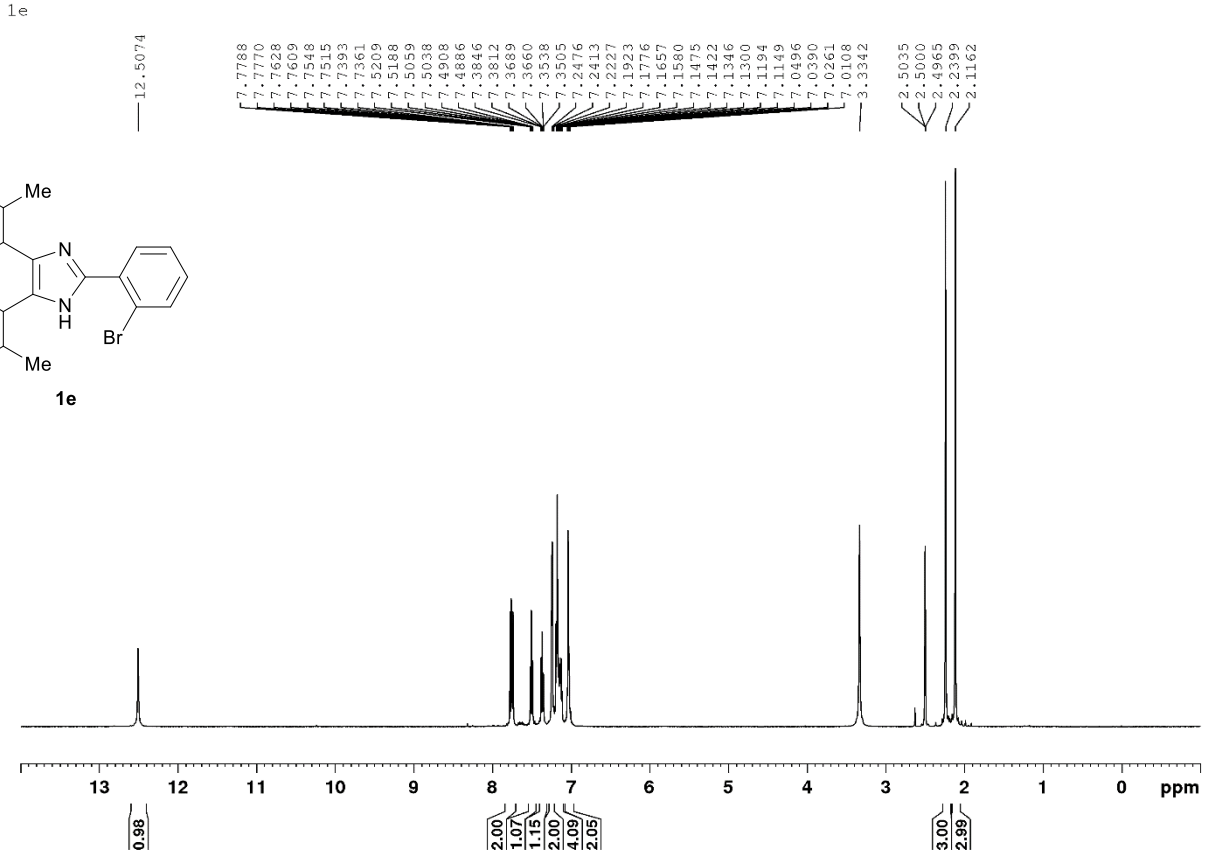
(Z)-2-(1-Bromo-3-methyl-1-phenylhex-1-en-2-yl)-4,5-diphenyl-1*H*-imidazole (1o): Yield 18% (0.41 g), pale yellow solid. mp 140-143 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.74 (s, 1H), 7.44-7.41 (m, 1H), 7.37-7.33 (m, 2H), 7.32-7.25 (m, 6H), 7.24-7.21 (m, 3H), 7.18-7.14 (m, 1H), 7.12-7.10 (m, 2H), 2.88-2.85 (m, 2H), 1.55-1.49 (m, 2H), 1.44-1.37 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 143.8, 140.4, 136.1, 135.0, 133.7, 130.9, 129.4, 128.6, 128.5, 128.0, 127.9, 127.7, 127.6, 126.9, 126.8, 126.3, 124.6, 36.9, 29.3, 21.8, 13.8. HRMS (EI): *m*/*z* [M^+] calcd for $C_{27}H_{25}BrN_2$: 456.1201, Found 456.1197.

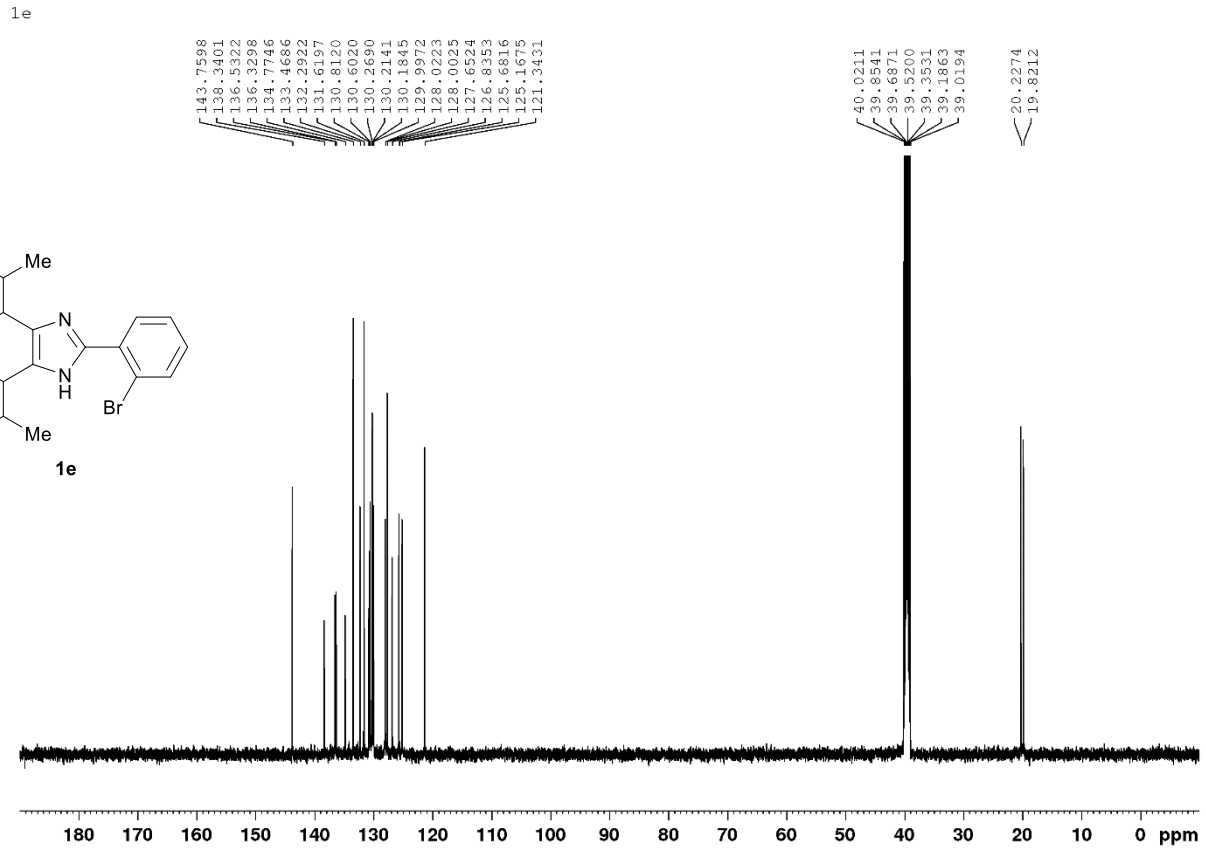
(Z)-2-(2-Bromo-1,2-diphenylvinyl)-4,5-diphenyl-1*H*-imidazole (1p): Yield 16% (0.38 g), white solid. mp 220-222 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.71 (s, 1H), 7.51-7.47 (m, 4H), 7.43-7.40 (m, 2H), 7.36-7.25 (m, 8H), 7.22-7.15 (m, 4H), 7.07-7.06 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 146.6, 139.9, 138.5, 136.4, 135.1, 134.7, 130.9, 129.6 (129.63), 129.6 (129.55), 128.6 (128.63), 128.6 (128.55), 128.3, 128.1, 128.0 (128.03), 128.0 (127.98), 127.6, 127.5, 127.1, 126.9, 126.5, 125.6. HRMS (EI): *m*/*z* [M^+] calcd for $C_{29}H_{21}BrN_2$: 476.0888, Found 476.0885.

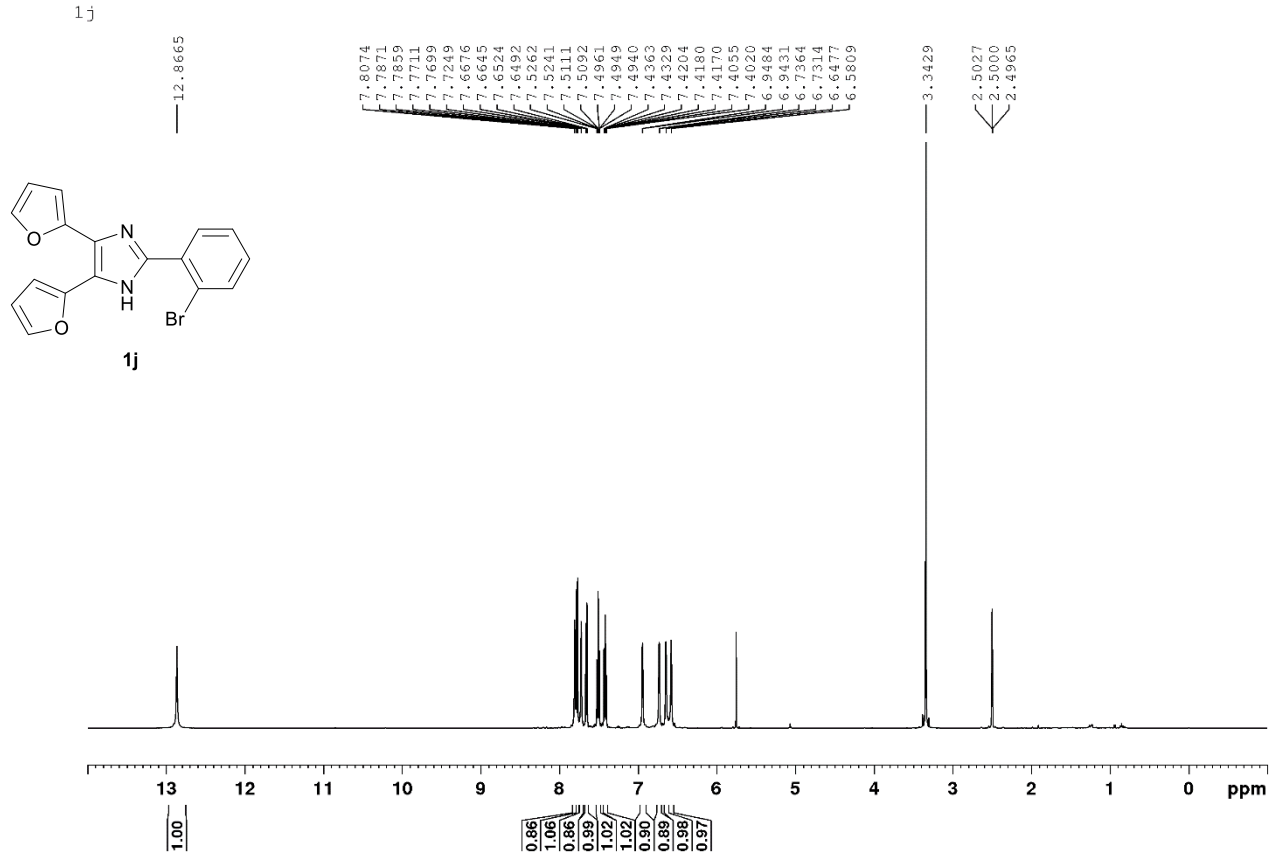
References

1. M.-Y. Gang, J.-Q. Liu and X.-S. Wang, *Tetrahedron*, 2017, **73**, 469

2. Copies of ^{1}H and ^{13}C NMR Spectra



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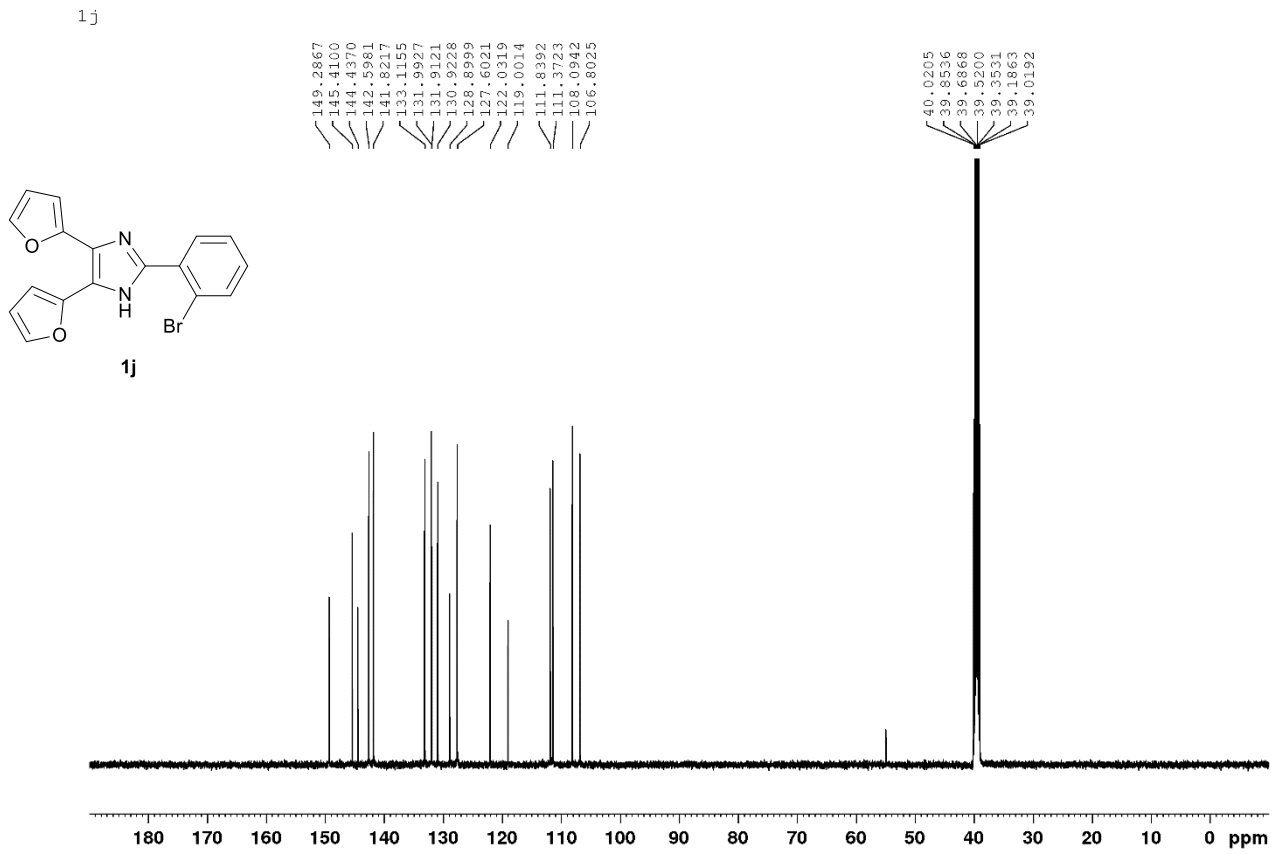
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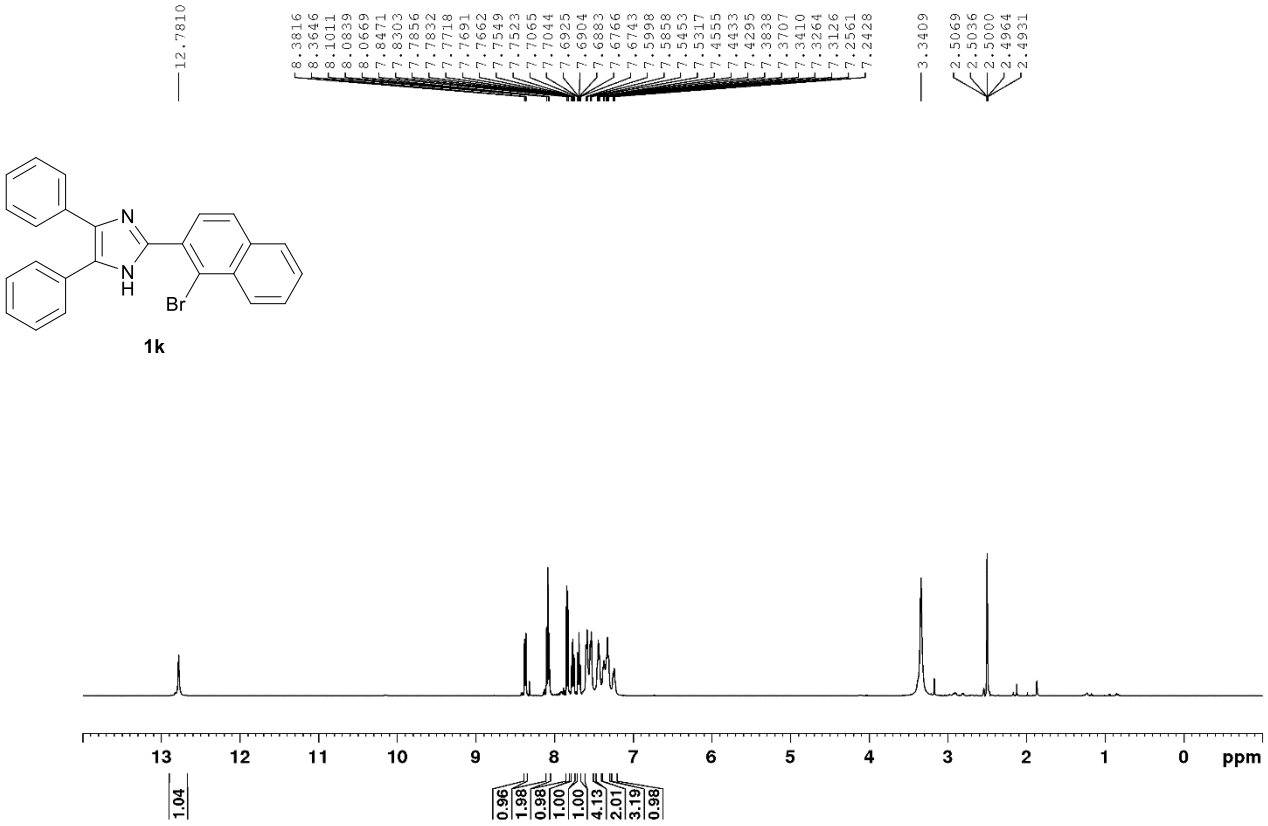
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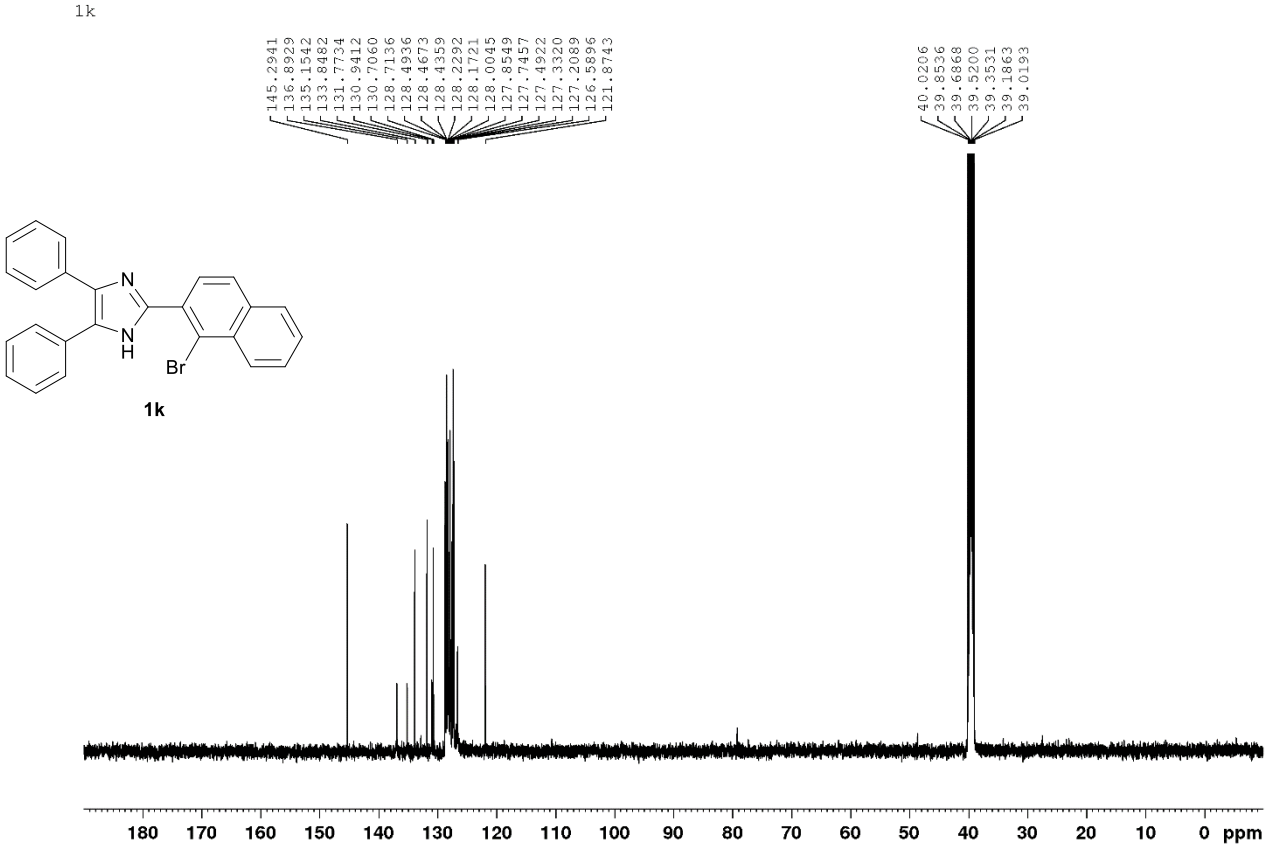
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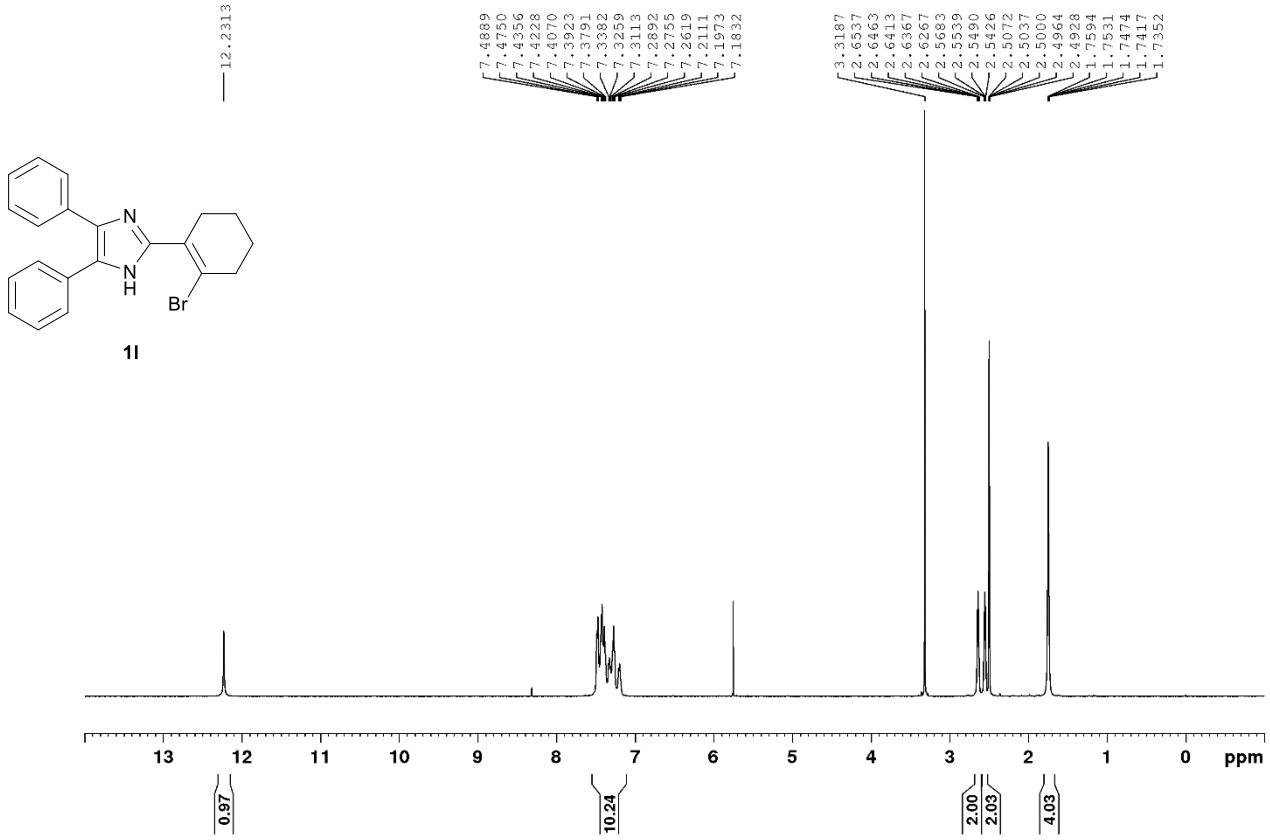
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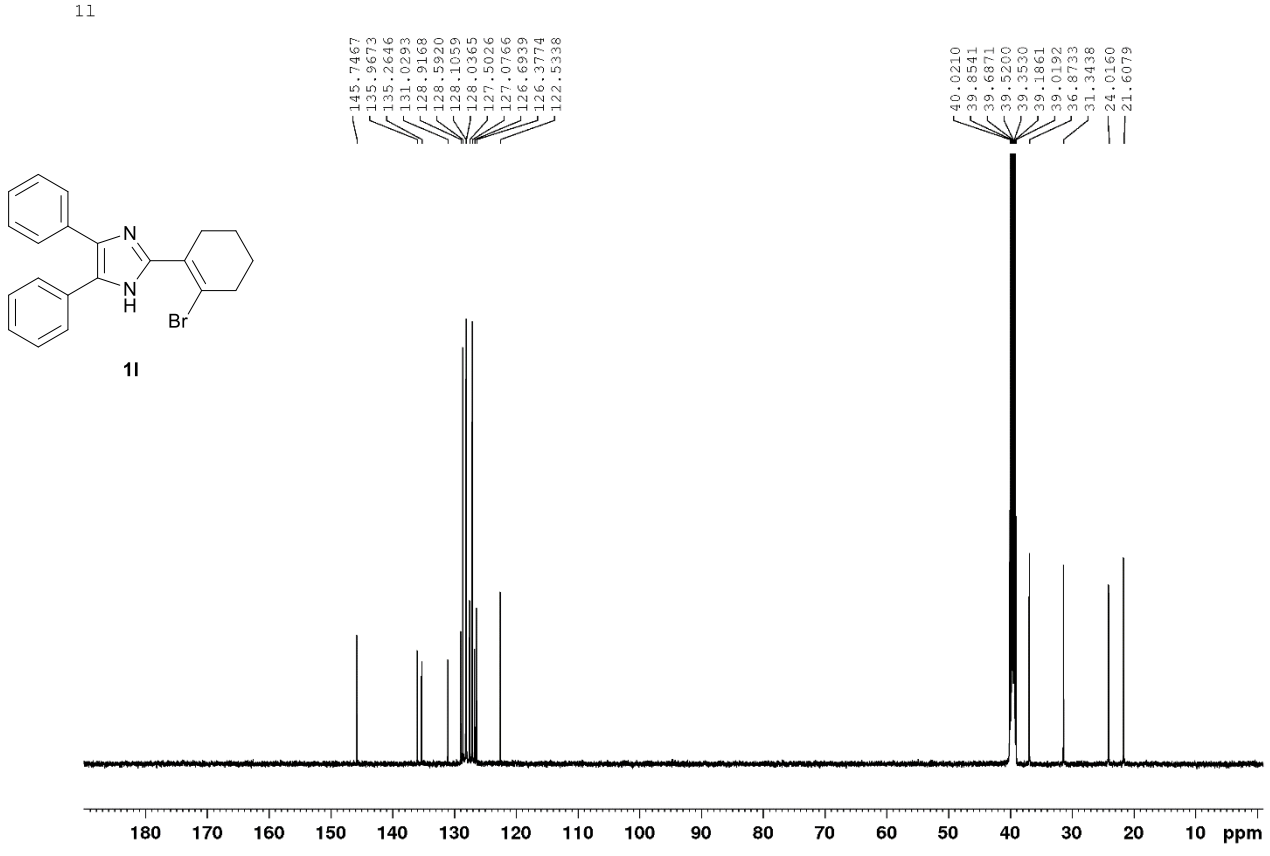
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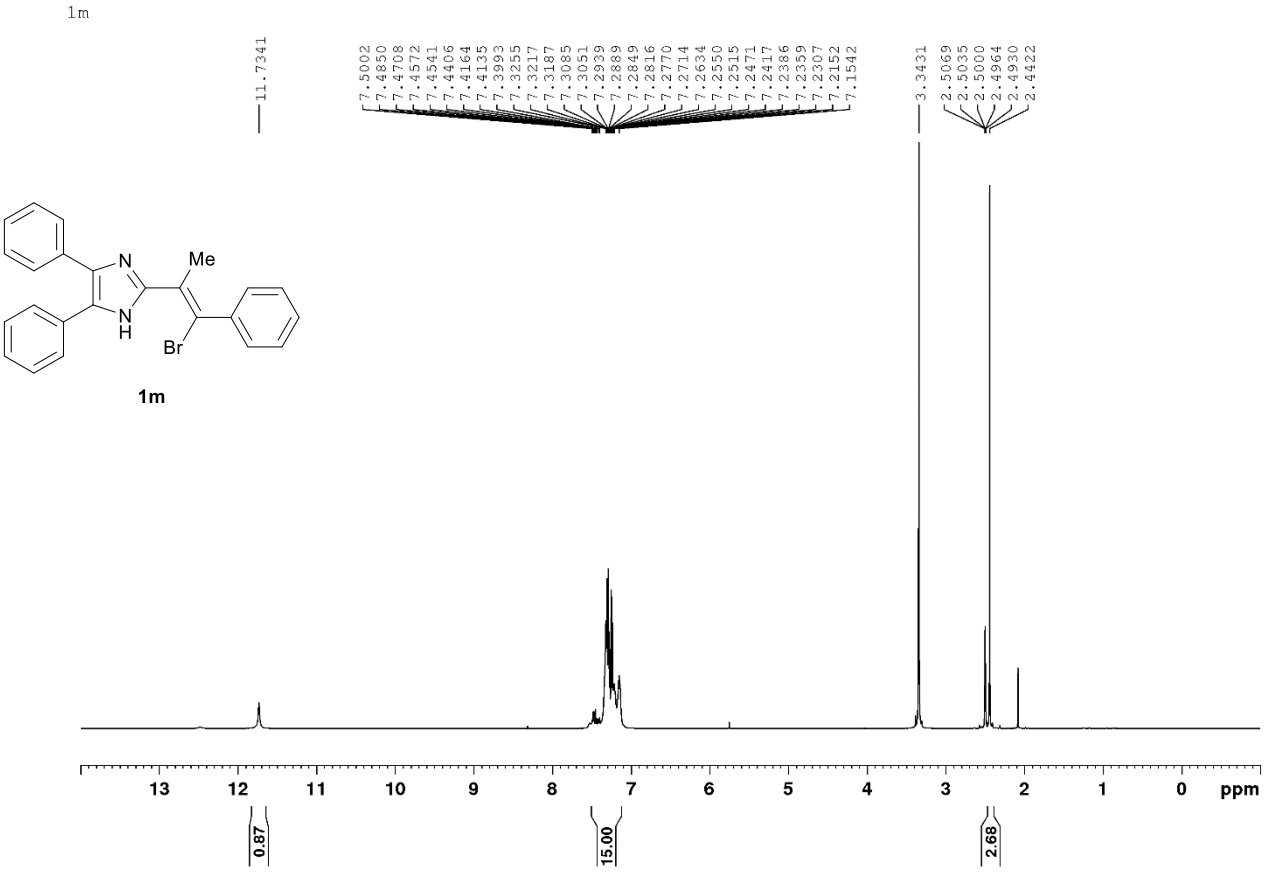
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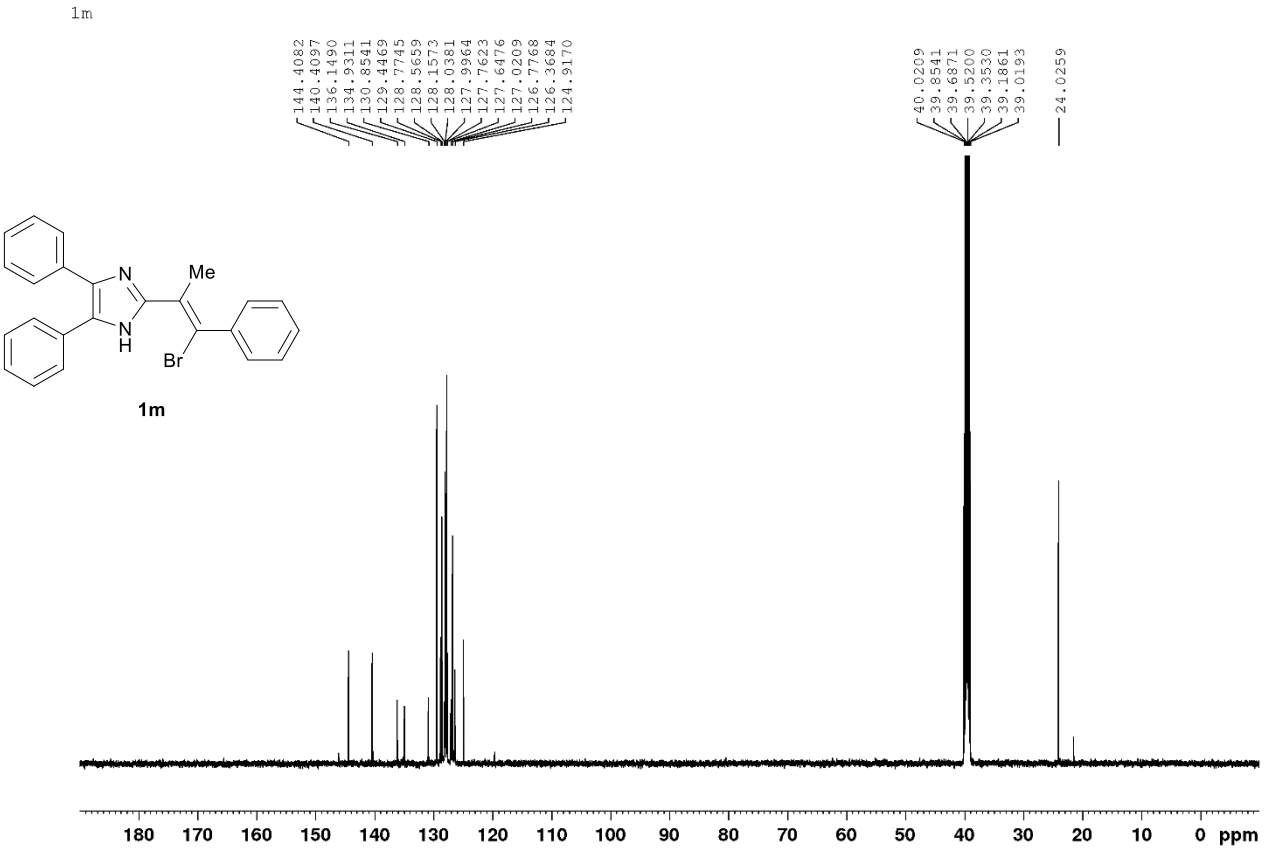
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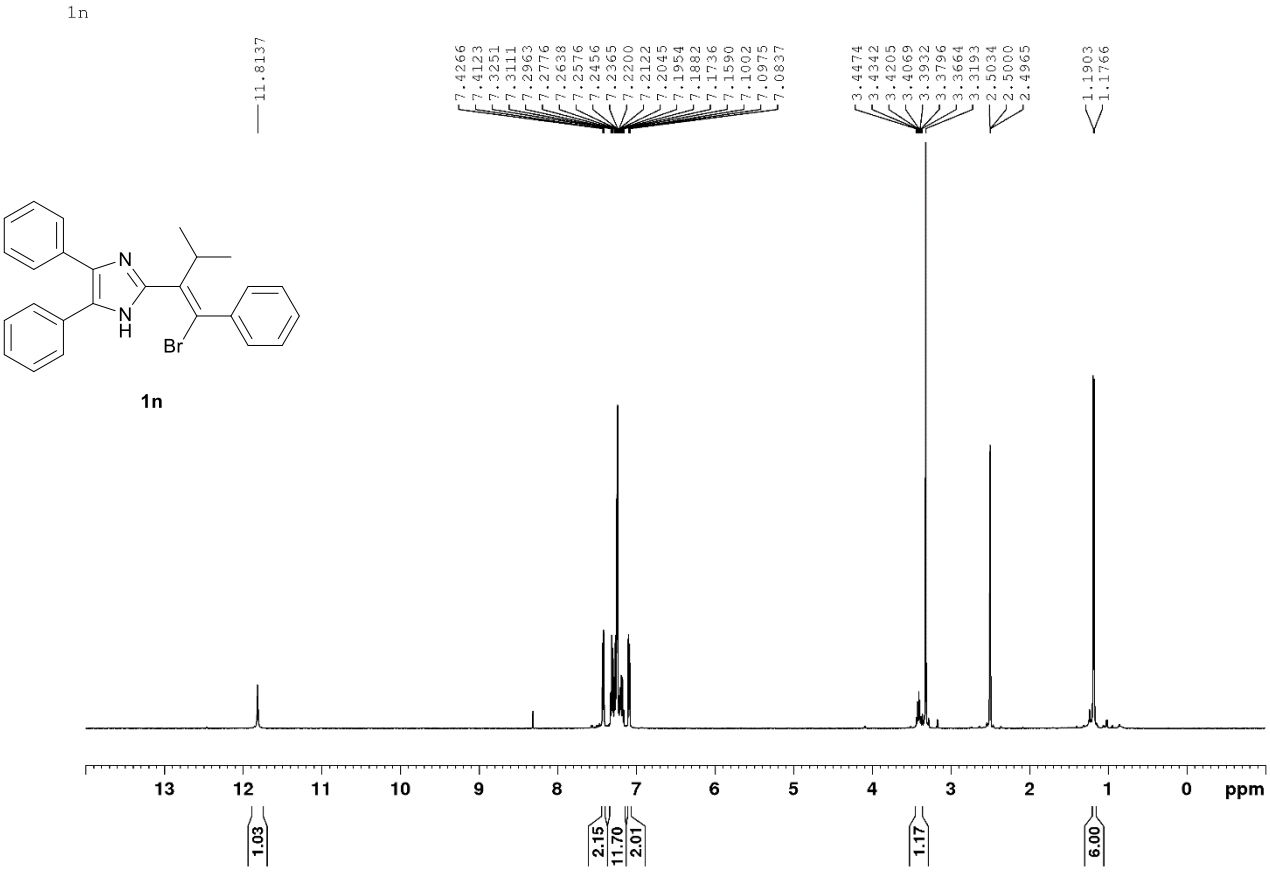
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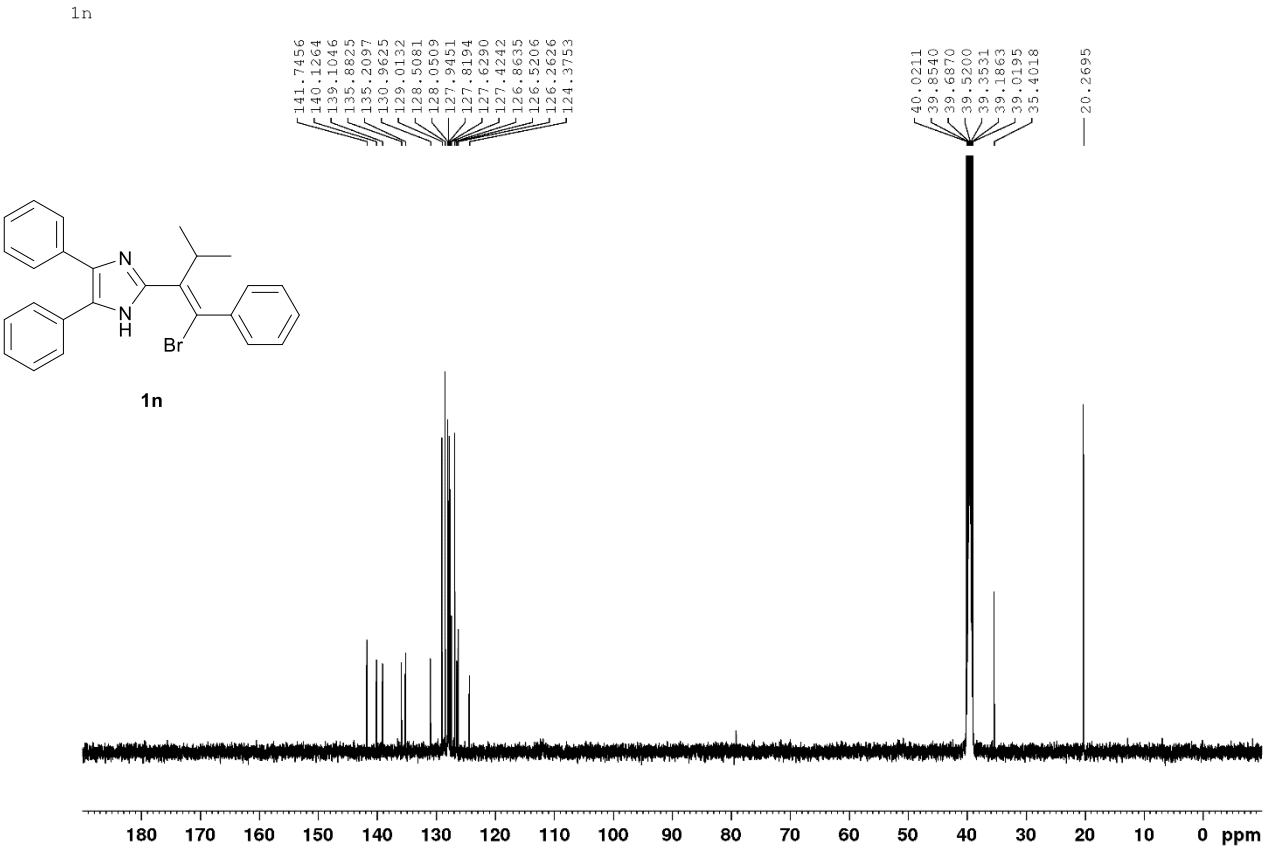
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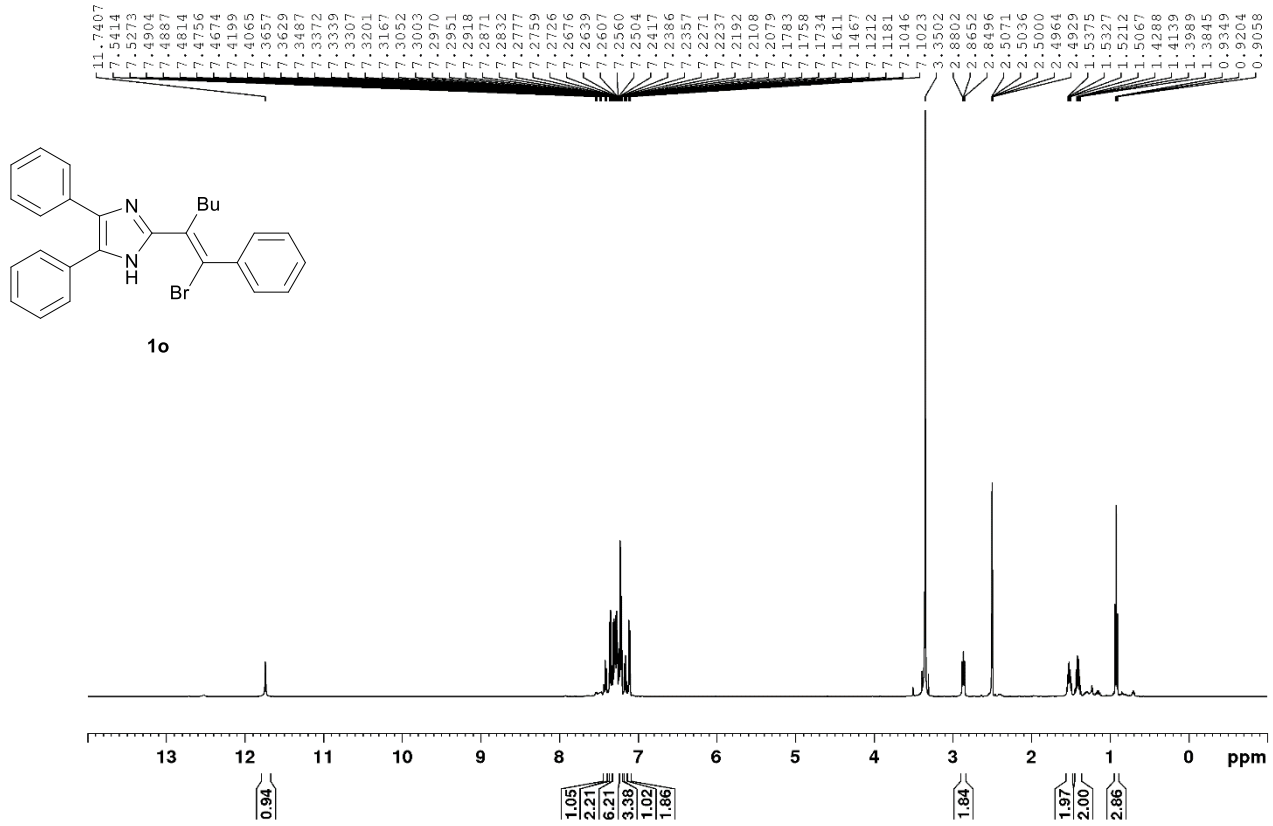
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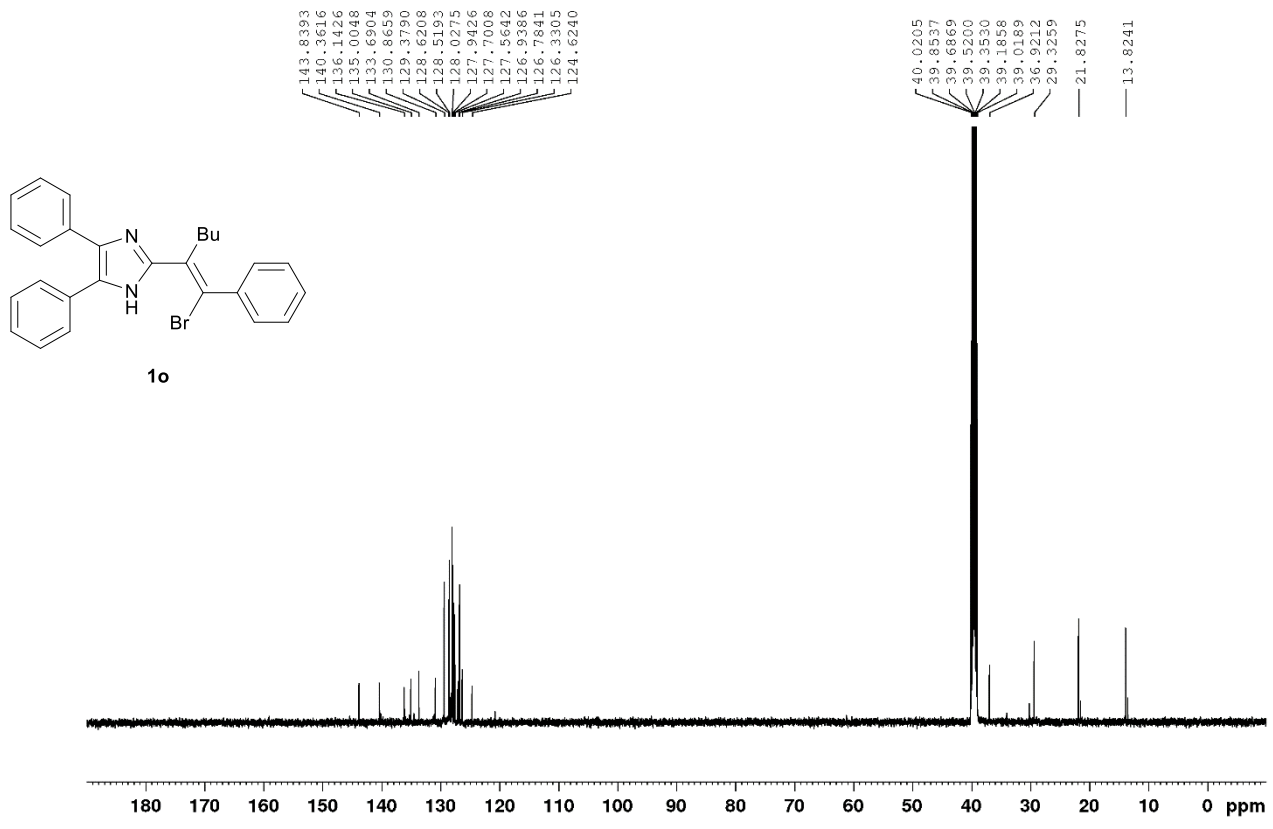
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126.7841
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124.6240
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39.5200
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39.1858
39.0189
36.9212
29.3259
21.8275
13.8241
N
Bu
N
H
Br
1o
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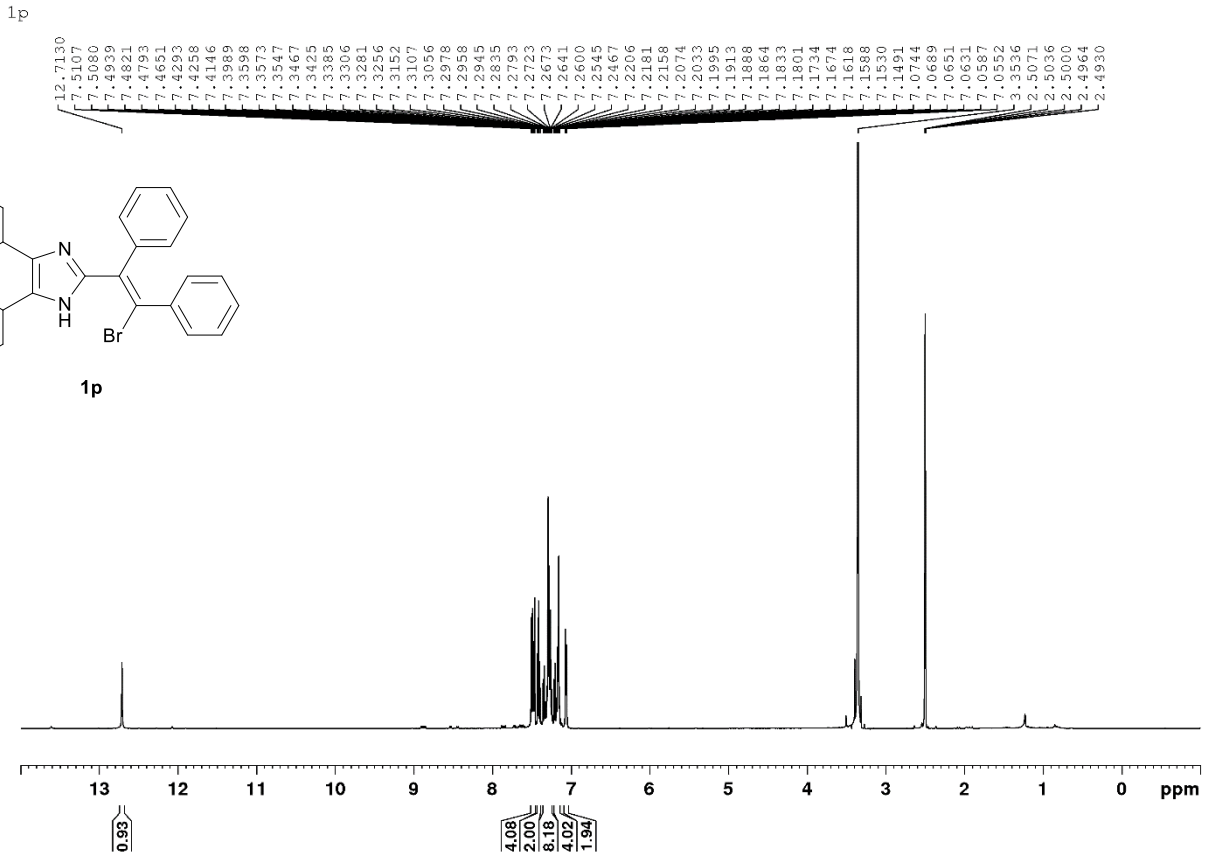
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7.4258
7.4146
7.3989
7.3598
7.3573
7.3547
7.3467
7.3425
7.3385
7.3306
7.3281
7.3256
7.3152
7.3107
7.3056
7.2978
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7.2945
7.2835
7.2793
7.2723
7.2673
7.2641
7.2600
7.2545
7.2467
7.2206
7.2181
7.2158
7.2074
7.2033
7.1995
7.1913
7.1888
7.1864
7.1833
7.1801
7.1734
7.1674
7.1618
7.1588
7.1530
7.1491
7.0744
7.0689
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7.0587
7.0552
3.3536
2.5071
2.5036
2.5000
2.4964
2.4930
N
N
H
Br
1p
13
12
11
10
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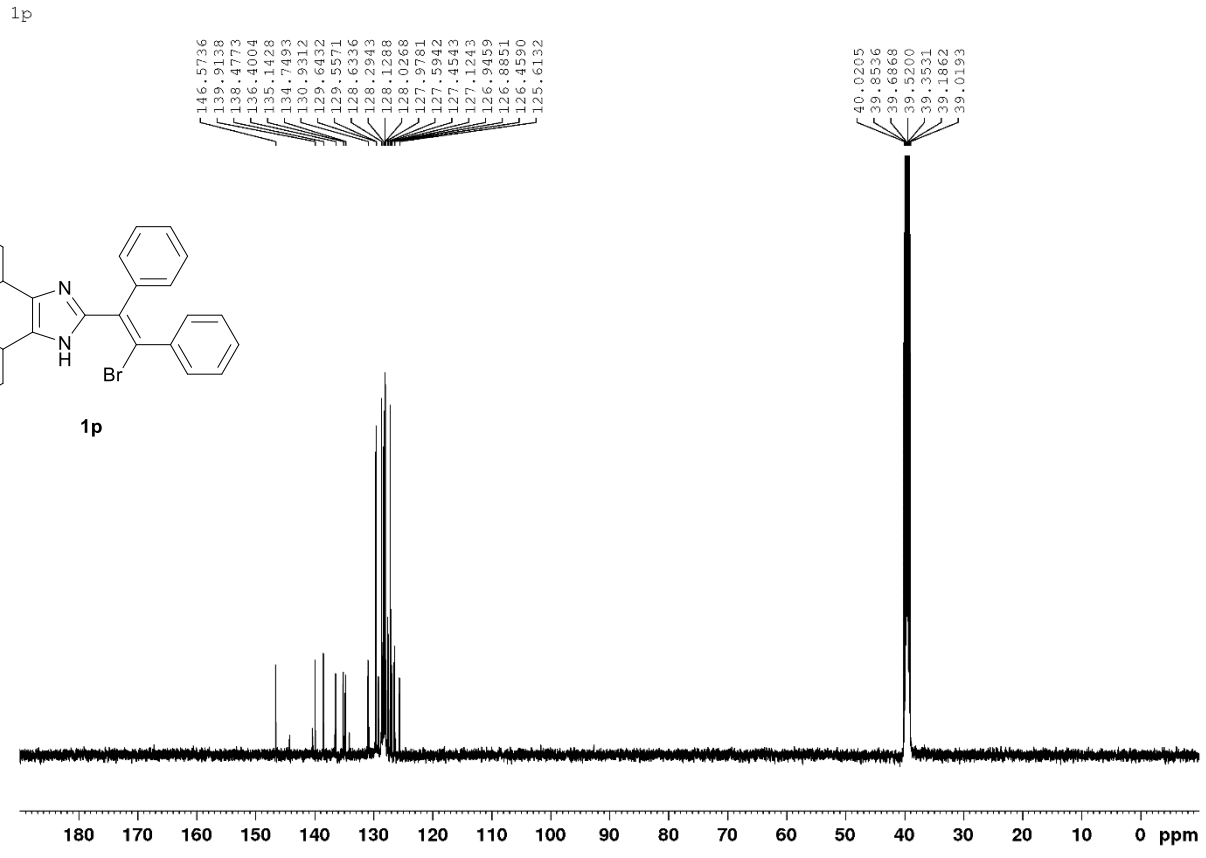
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127.4543
127.1243
126.9459
126.8851
126.4590
125.6132
40.0205
39.8536
39.6868
39.5200
39.3531
39.1862
39.0193
N
N
H
Br
1p
180
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10
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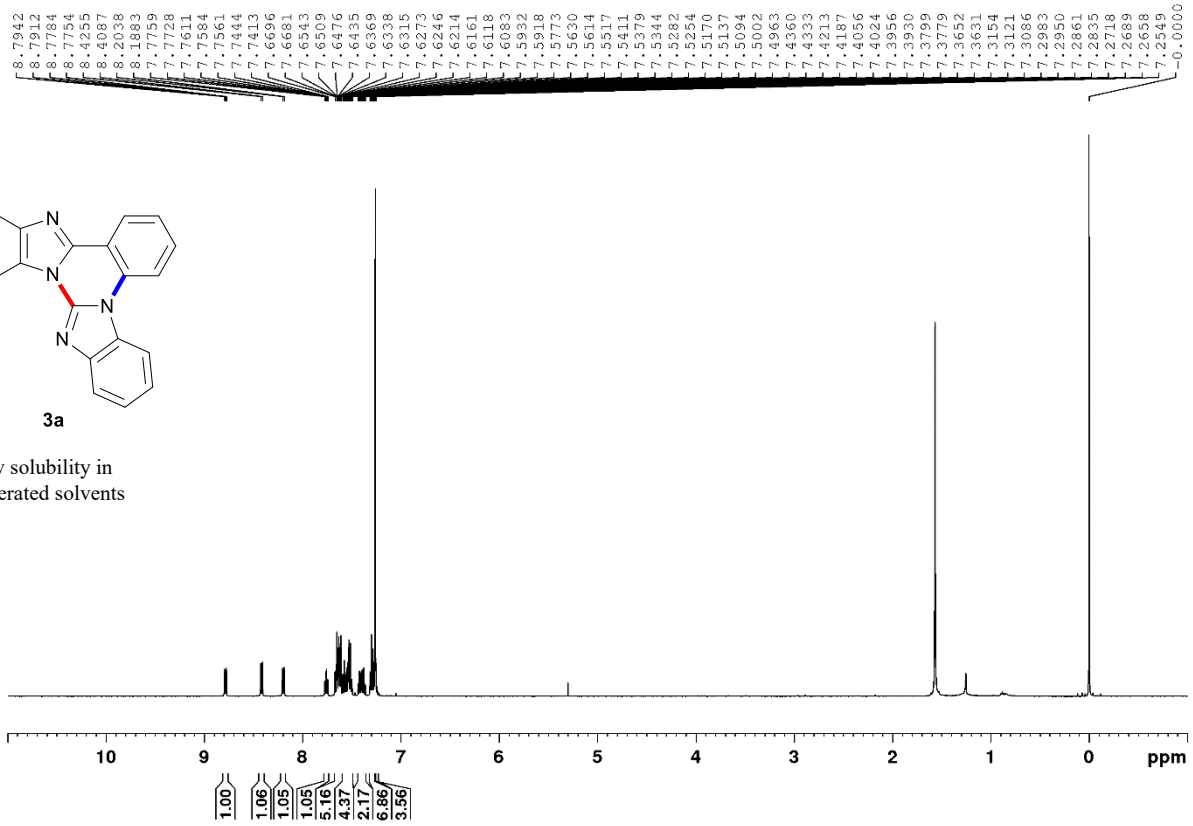
ppm

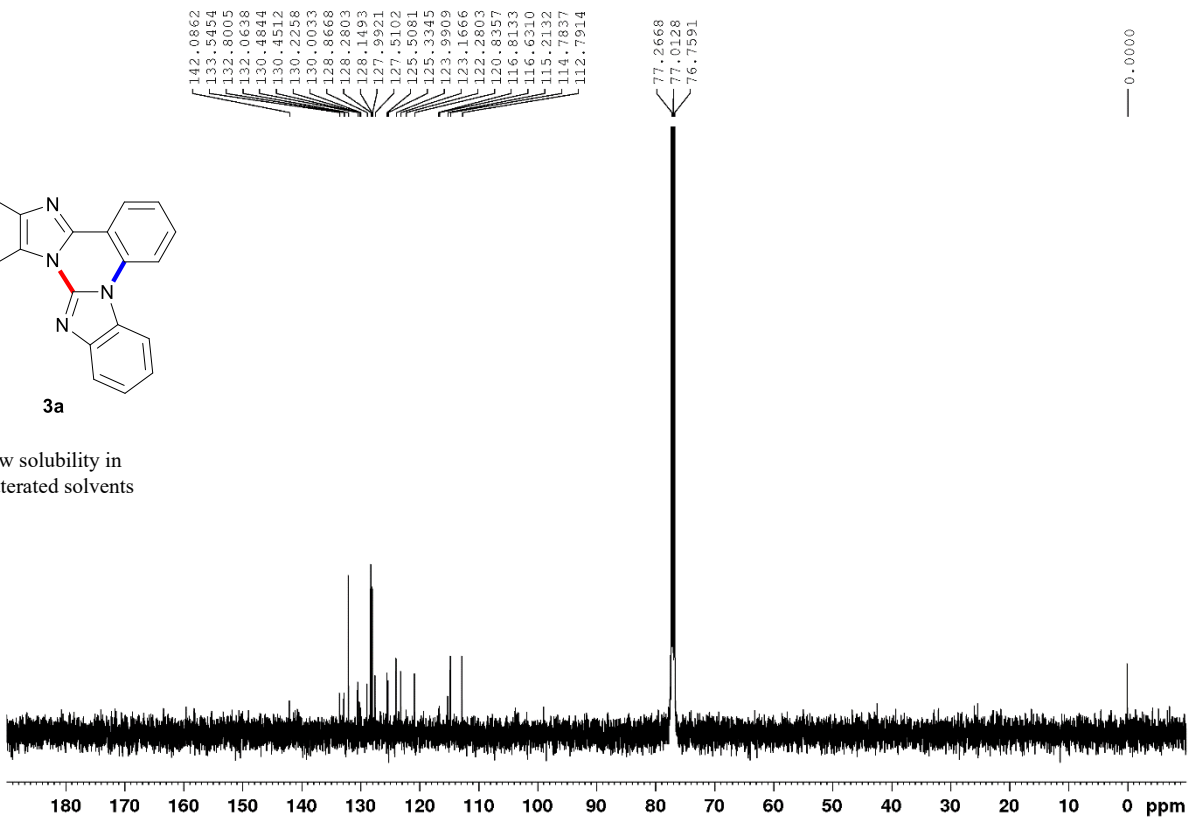
3a



3a

low solubility in
deuterated solvents

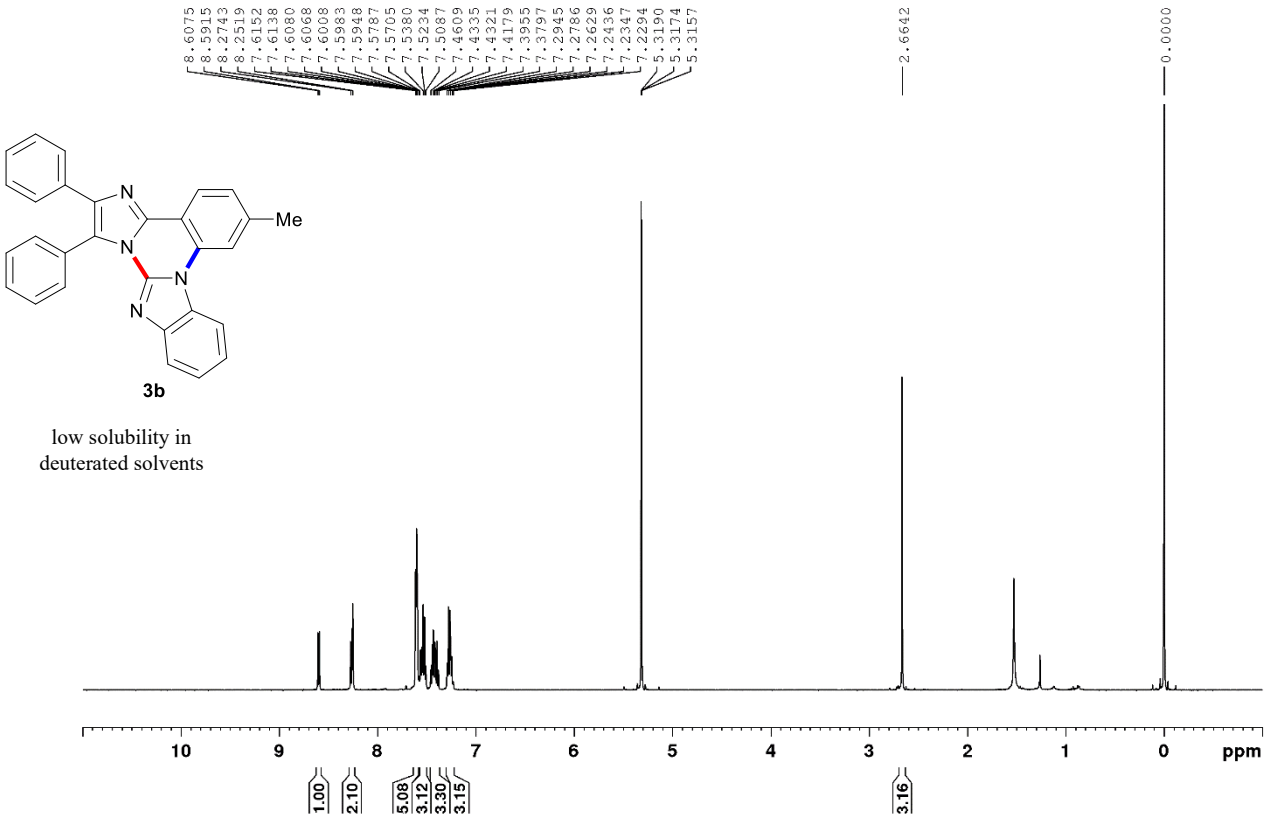
3a



3a

low solubility in
deuterated solvents

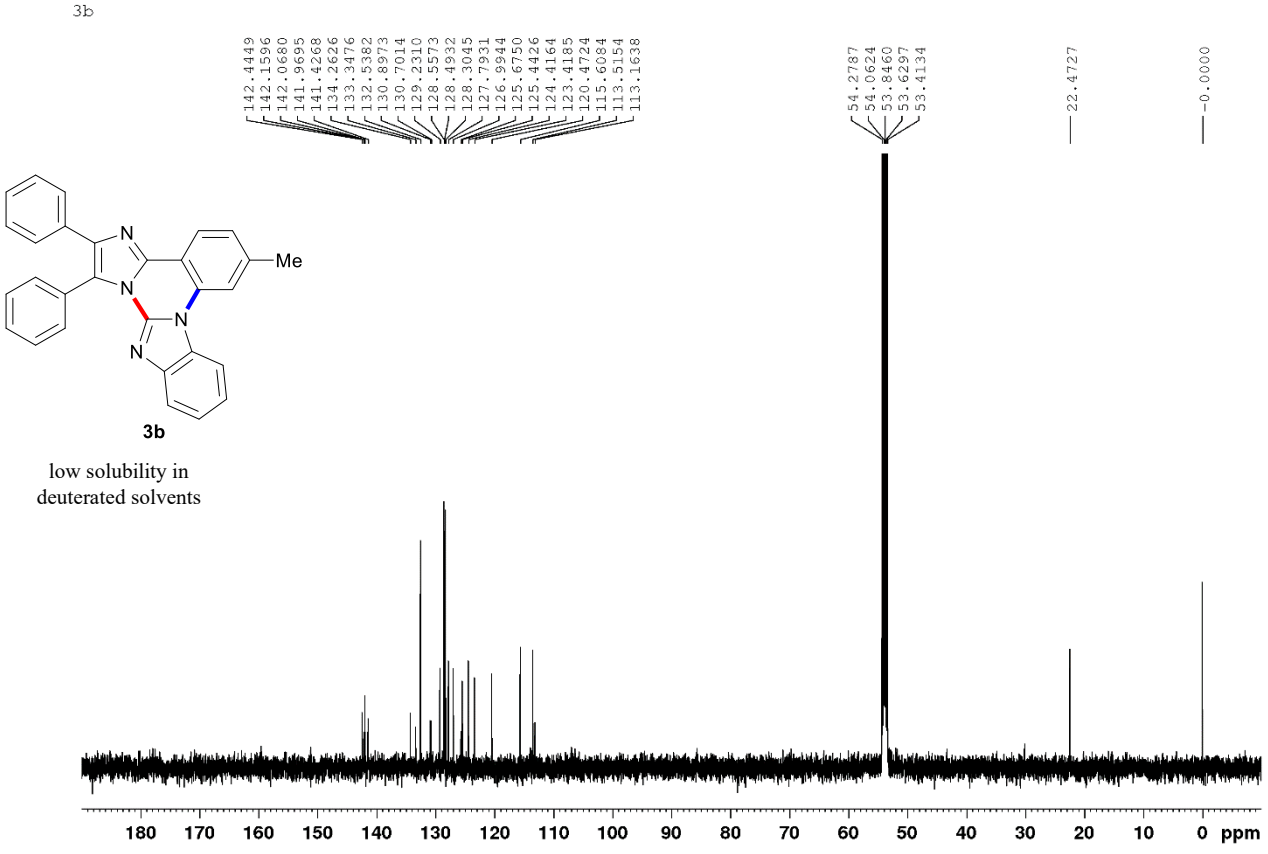
3b



3b

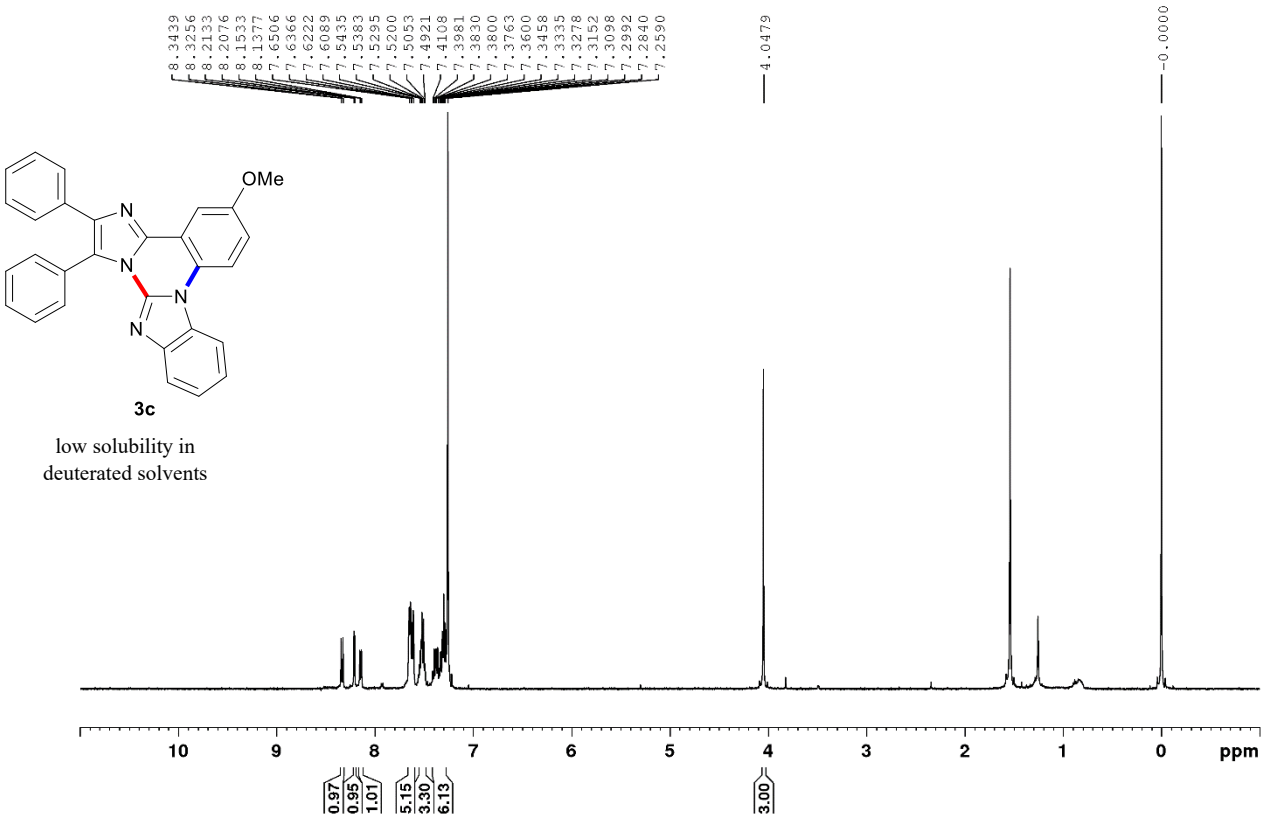
low solubility in deuterated solvents

3b



3b

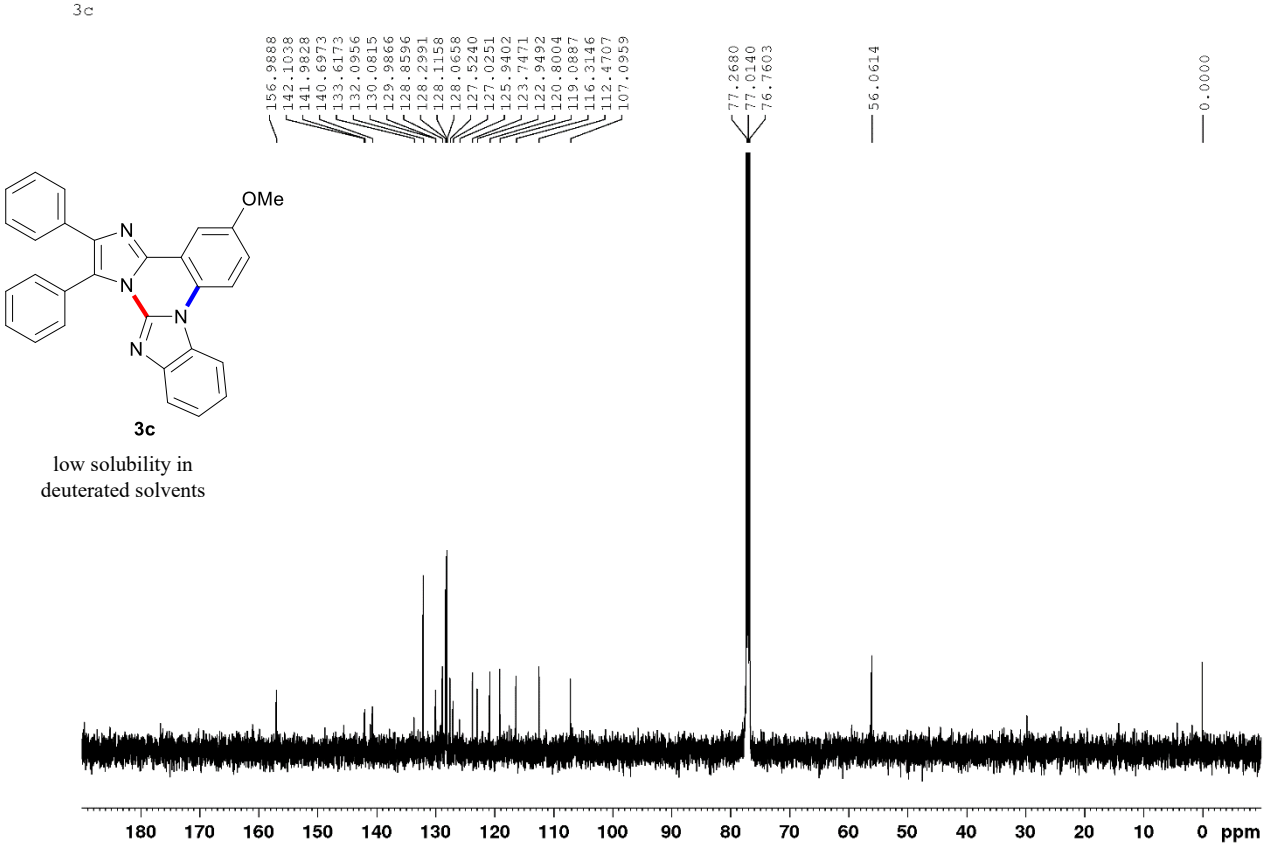
low solubility in deuterated solvents



3c

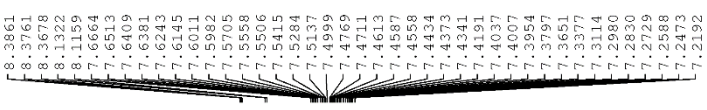
low solubility in deuterated solvents

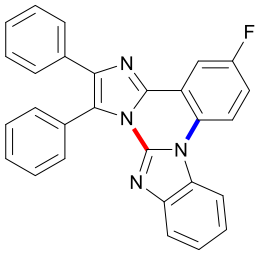
3c



3c

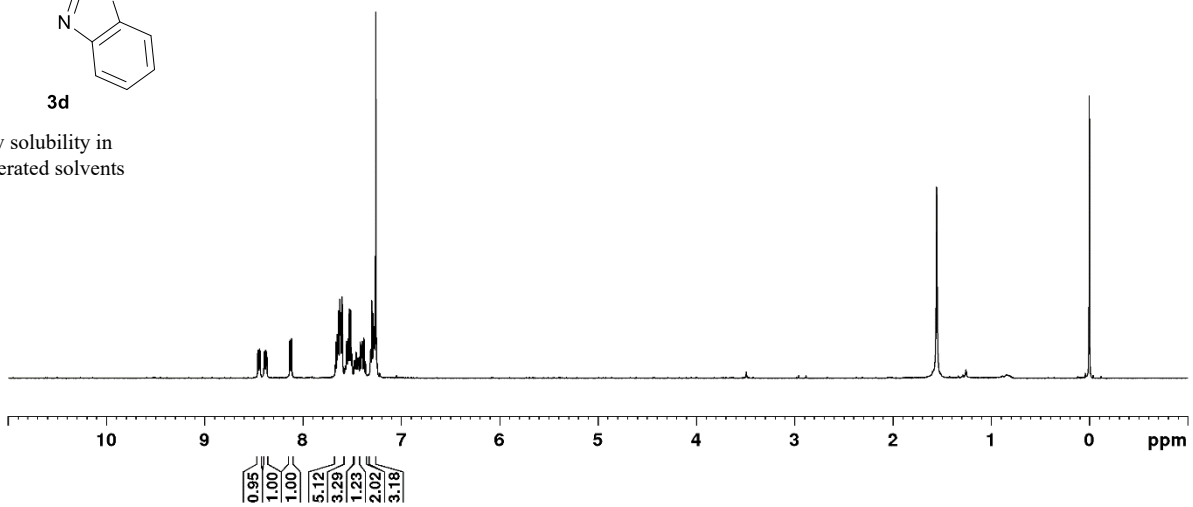
low solubility in deuterated solvents

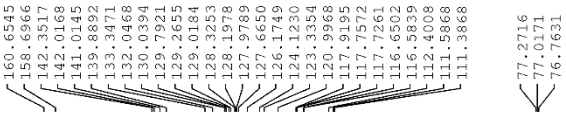


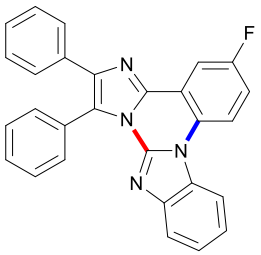


3d

low solubility in deuterated solvents

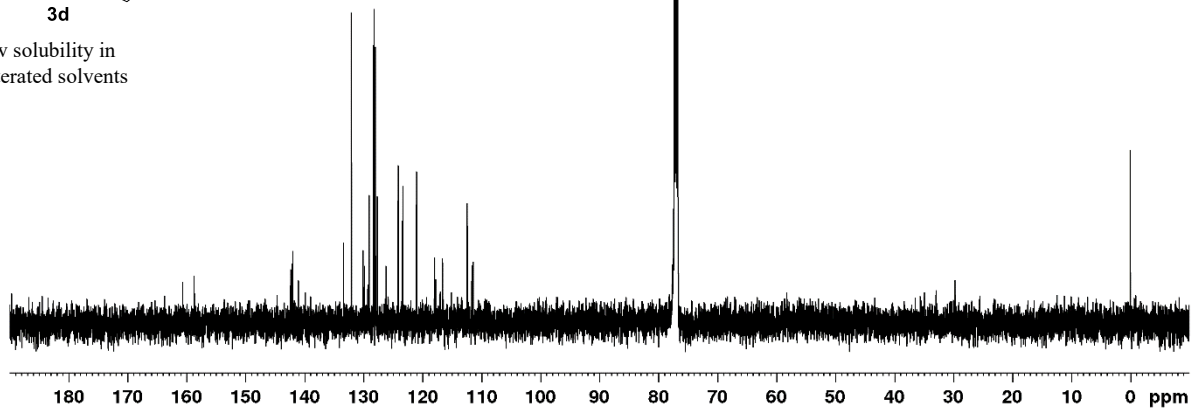


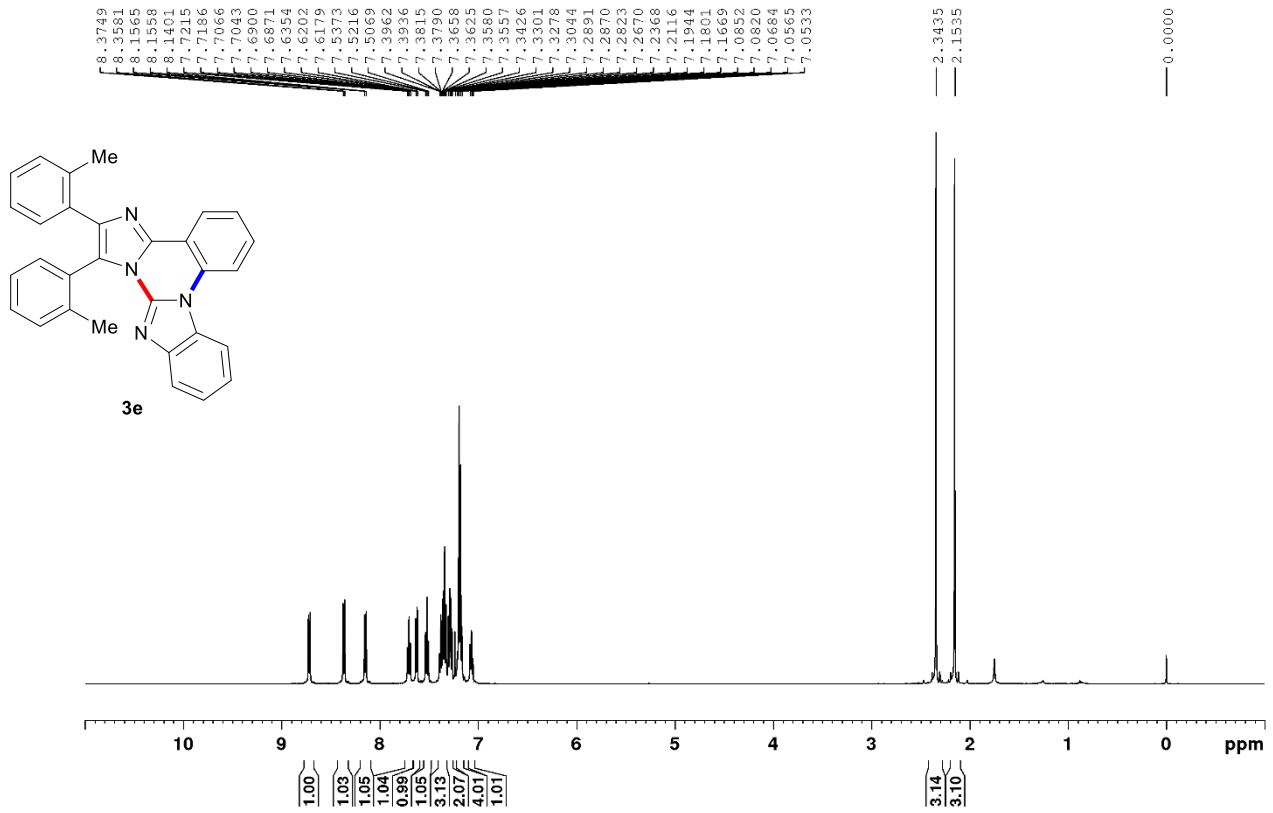




3d

low solubility in deuterated solvents

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7.6900
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7.6354
7.6202
7.6179
7.5373
7.5216
7.5069
7.3962
7.3936
7.3815
7.3790
7.3658
7.3625
7.3580
7.3557
7.3426
7.3301
7.3278
7.3044
7.2891
7.2870
7.2823
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7.1944
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7.0820
7.0684
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7.0533
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2.1535
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Me
N
N
N
N
Me
3e
10
9
8
7
6
5
4
3
2
1
0
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1.04
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2.07
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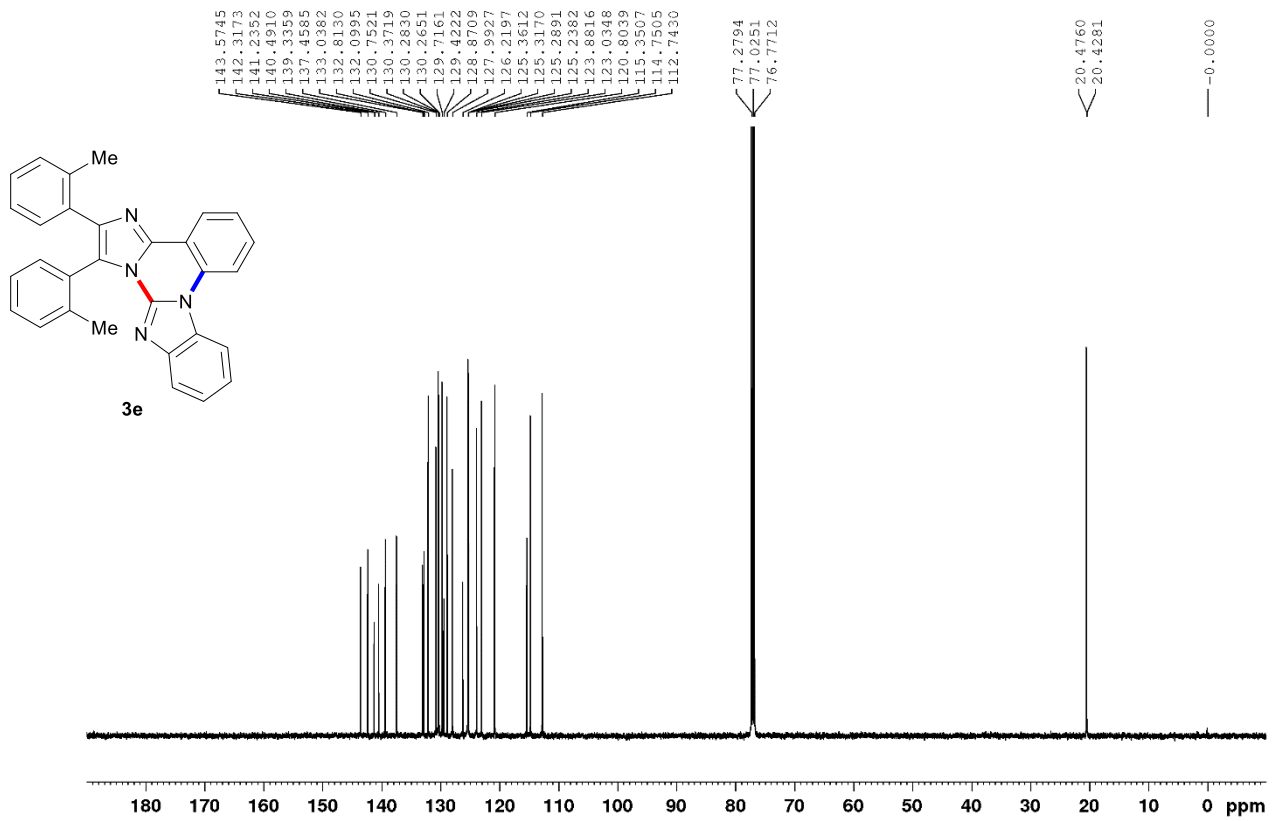
3.10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125.3612
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125.2891
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123.8816
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120.8039
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112.7430
77.2794
77.0251
76.7712
20.4760
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Me
N
N
N
N
Me
3e
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110
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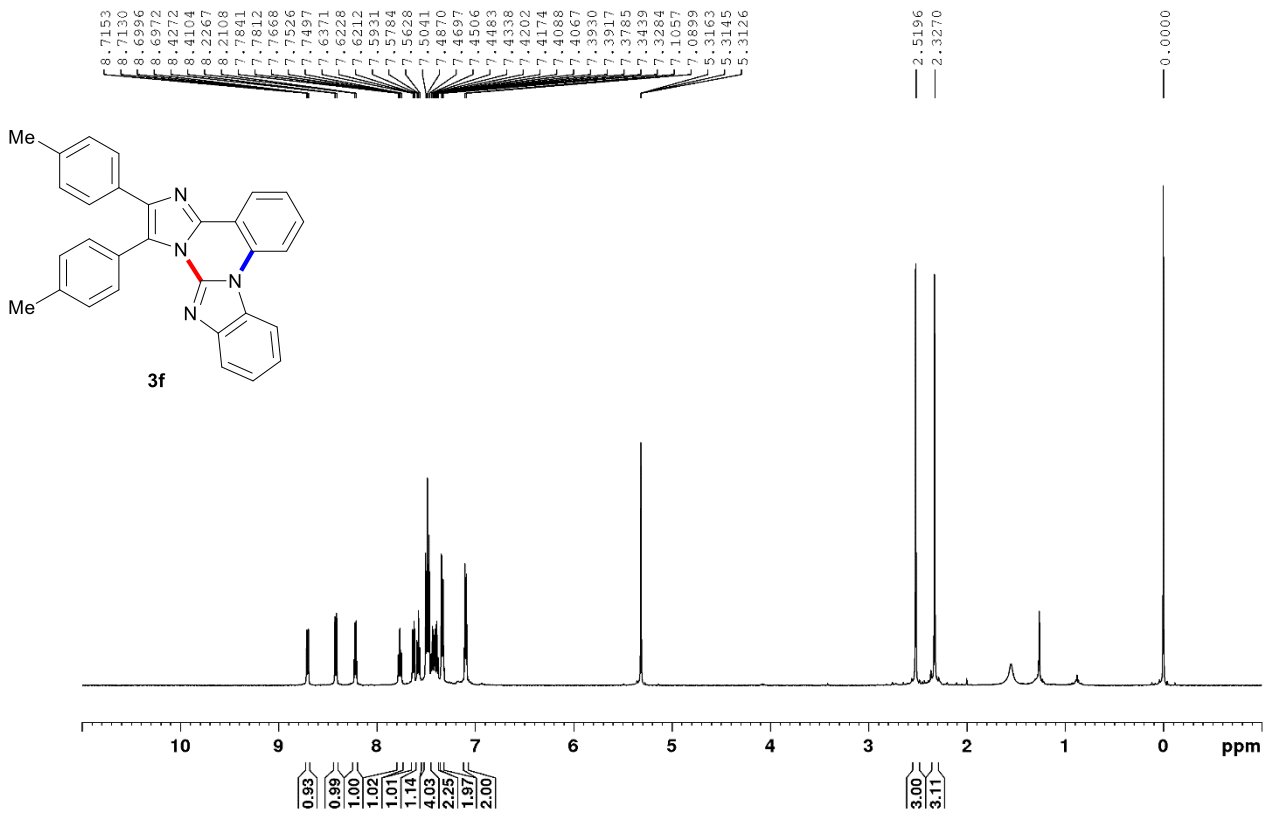
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7.7812
7.7668
7.7526
7.7497
7.6371
7.6228
7.6212
7.5931
7.5784
7.5628
7.5041
7.4870
7.4697
7.4506
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7.4338
7.4202
7.4174
7.4088
7.4067
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7.3917
7.3785
7.3439
7.3284
7.1057
7.0899
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5.3145
5.3126
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Me
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N
N
N
3f
10
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8
7
6
5
4
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1
0
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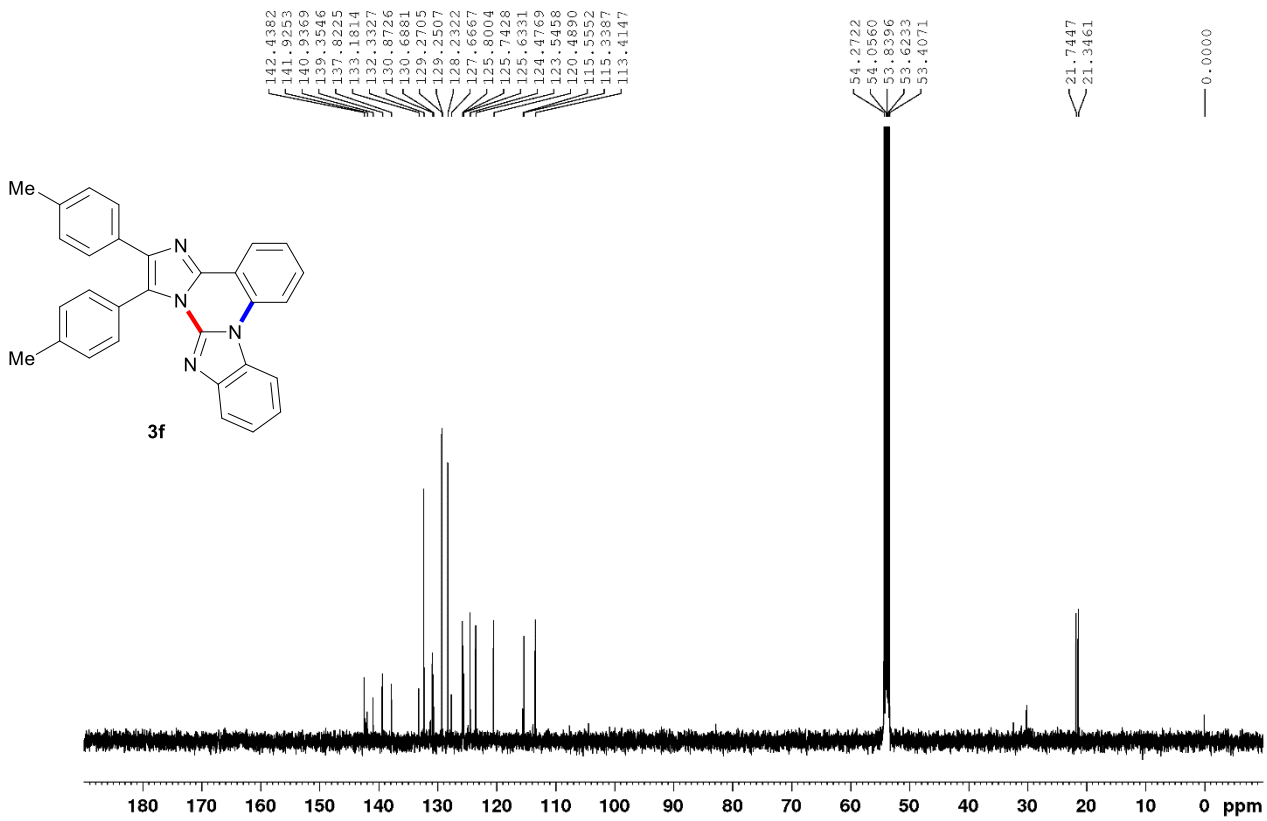
3.00
3.11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127.6667
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125.7428
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123.5458
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115.3387
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53.8396
53.6233
53.4071
21.7447
21.3461
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Me
Me
N
N
N
N
3f
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140
130
120
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100
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80
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60
50
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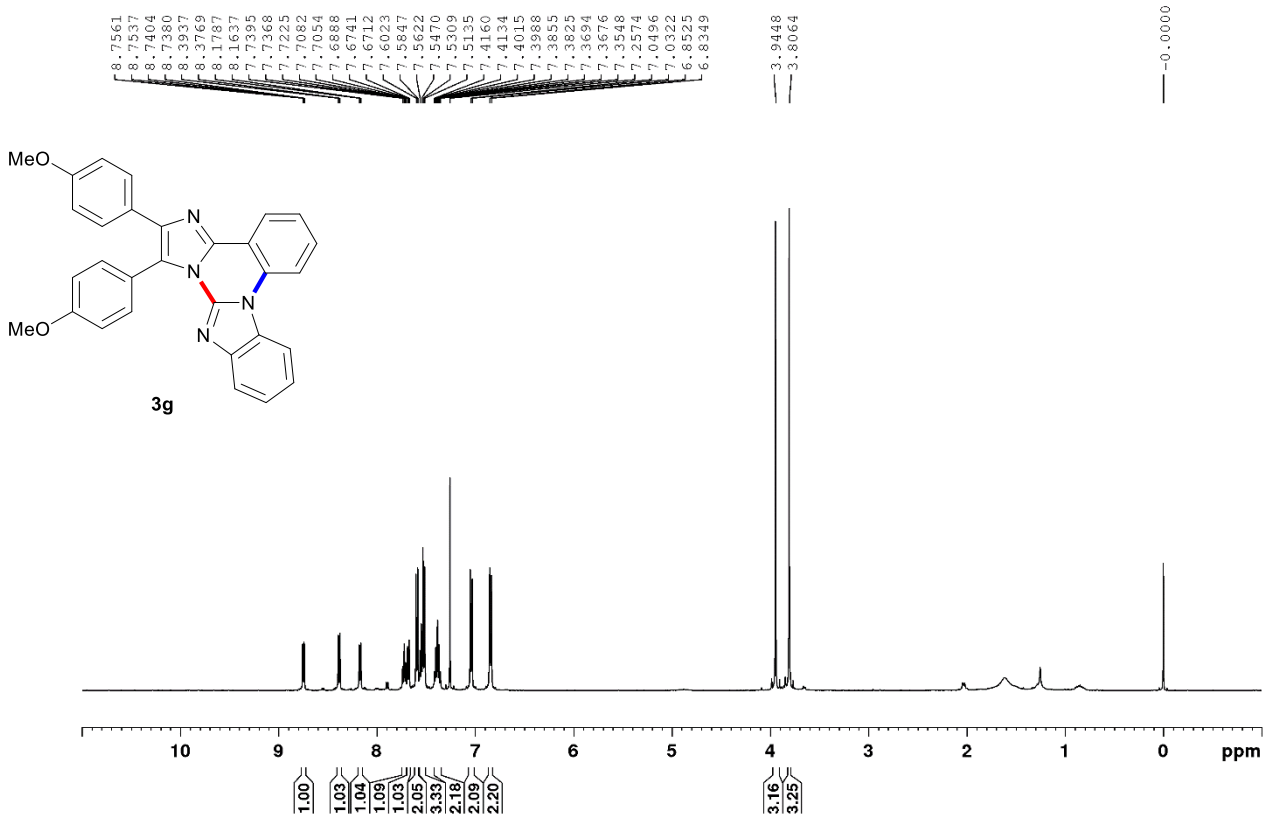
30
20
10
0
ppm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8.1787
8.1637
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7.7082
7.7054
7.6888
7.6741
7.6712
7.6023
7.5847
7.5622
7.5470
7.5309
7.5135
7.4160
7.4134
7.4015
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7.3855
7.3825
7.3694
7.3676
7.3548
7.2574
7.0496
7.0322
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6.8349
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3.8064
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MeO
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N
N
N
3g
10
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8
7
6
5
4
3
2
1
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1.09
1.03
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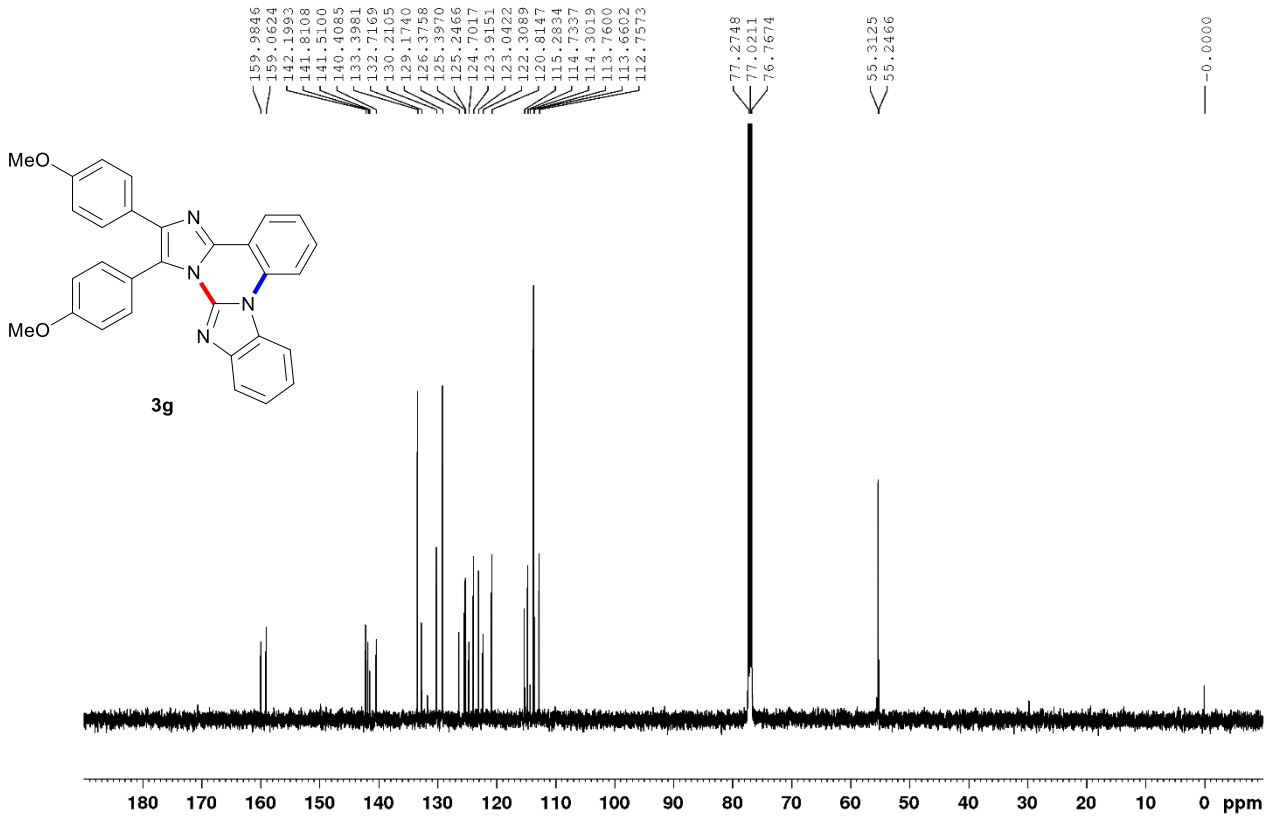
2.20
3.16
3.25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113.7600
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112.7573
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MeO
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N
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N
3g
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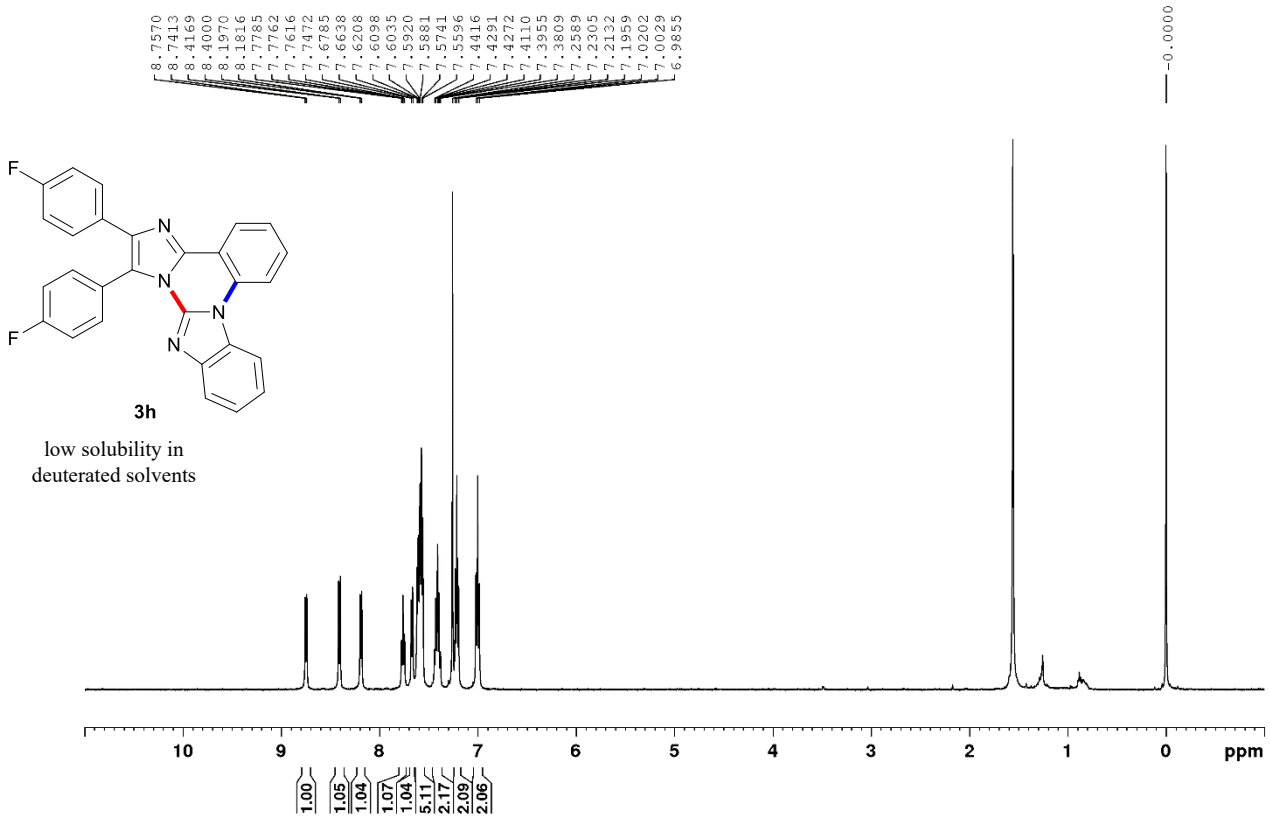
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7.6098
7.6035
7.5920
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7.5741
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7.2305
7.2132
7.1959
7.0202
7.0029
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F
F
N
N
N
N
3h
low solubility in
deuterated solvents
10
9
8
7
6
5
4
3
2
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ppm
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1.05
1.04
1.07
1.04
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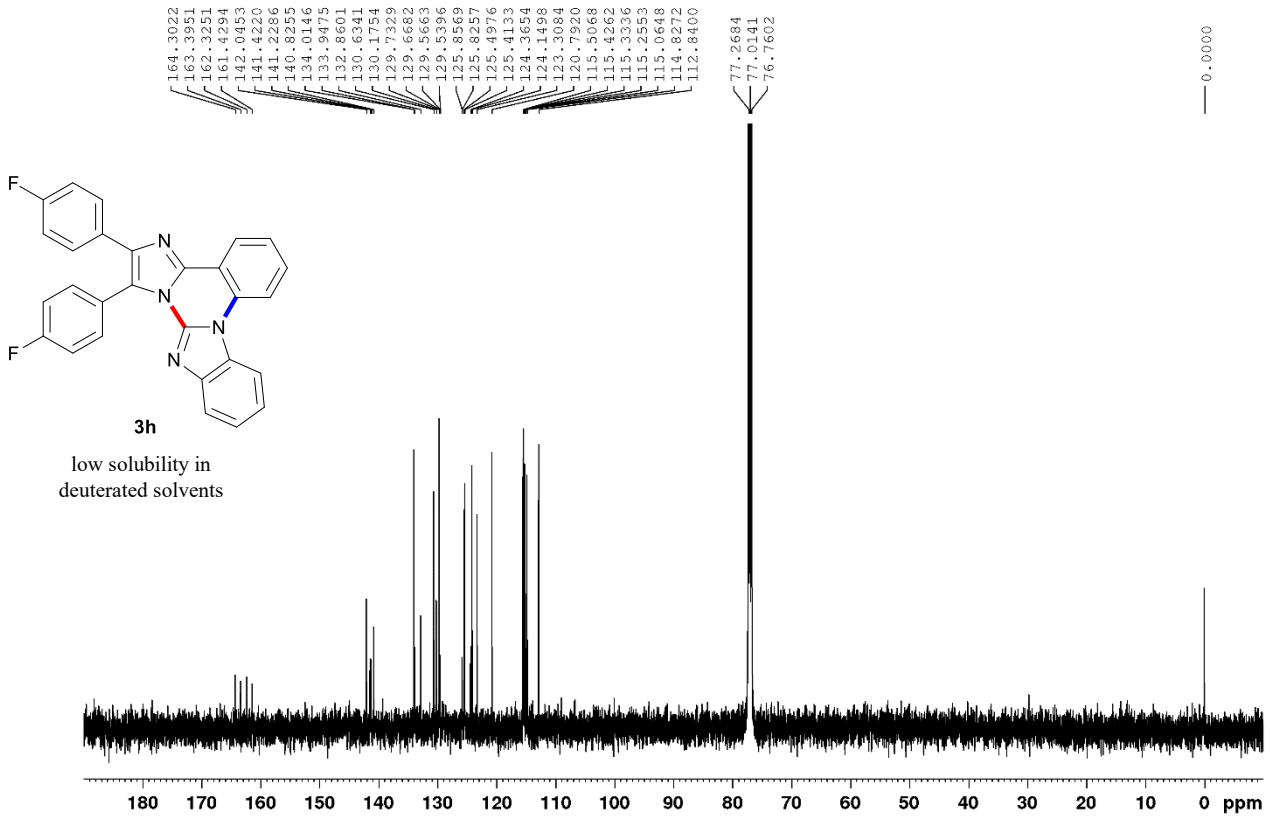
2.06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F
N
N
N
N
3h
low solubility in
deuterated solvents
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140
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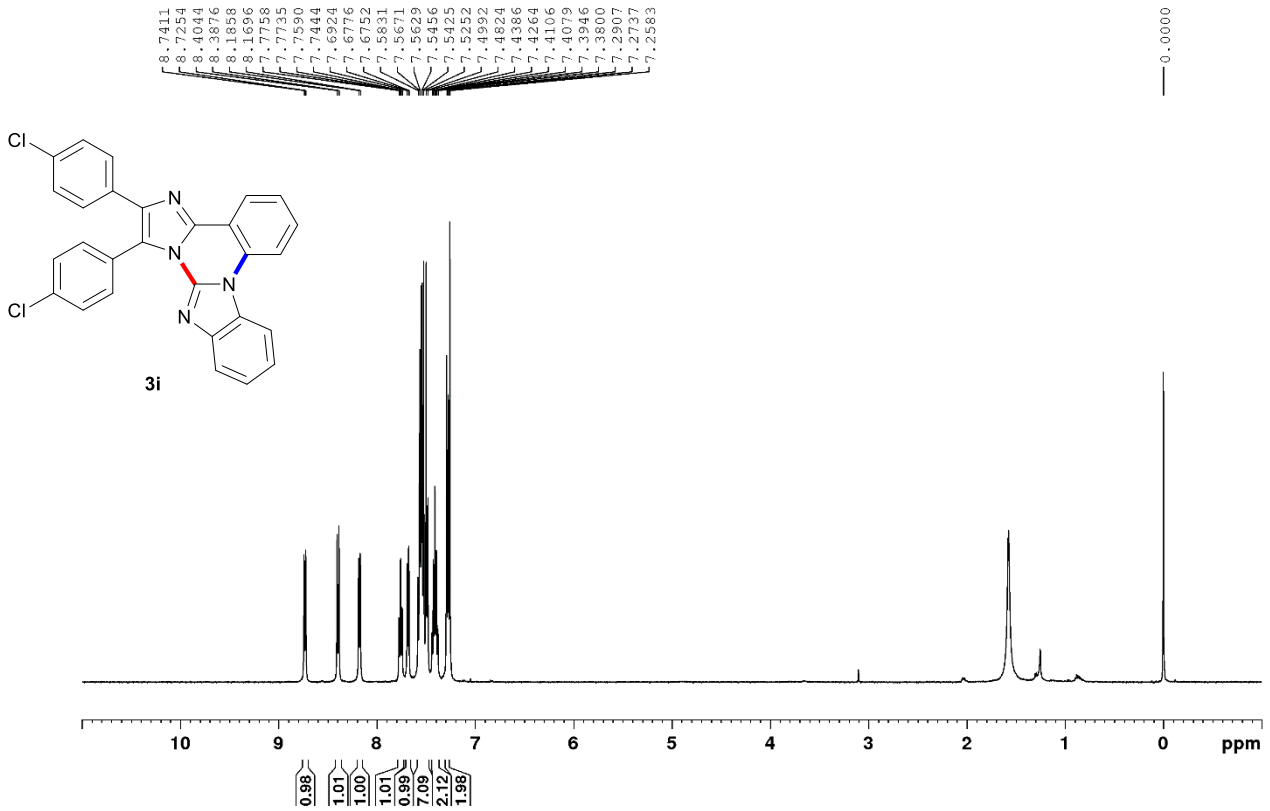
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7.2583
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Cl
Cl
N
N
N
N
3i
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9
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7
6
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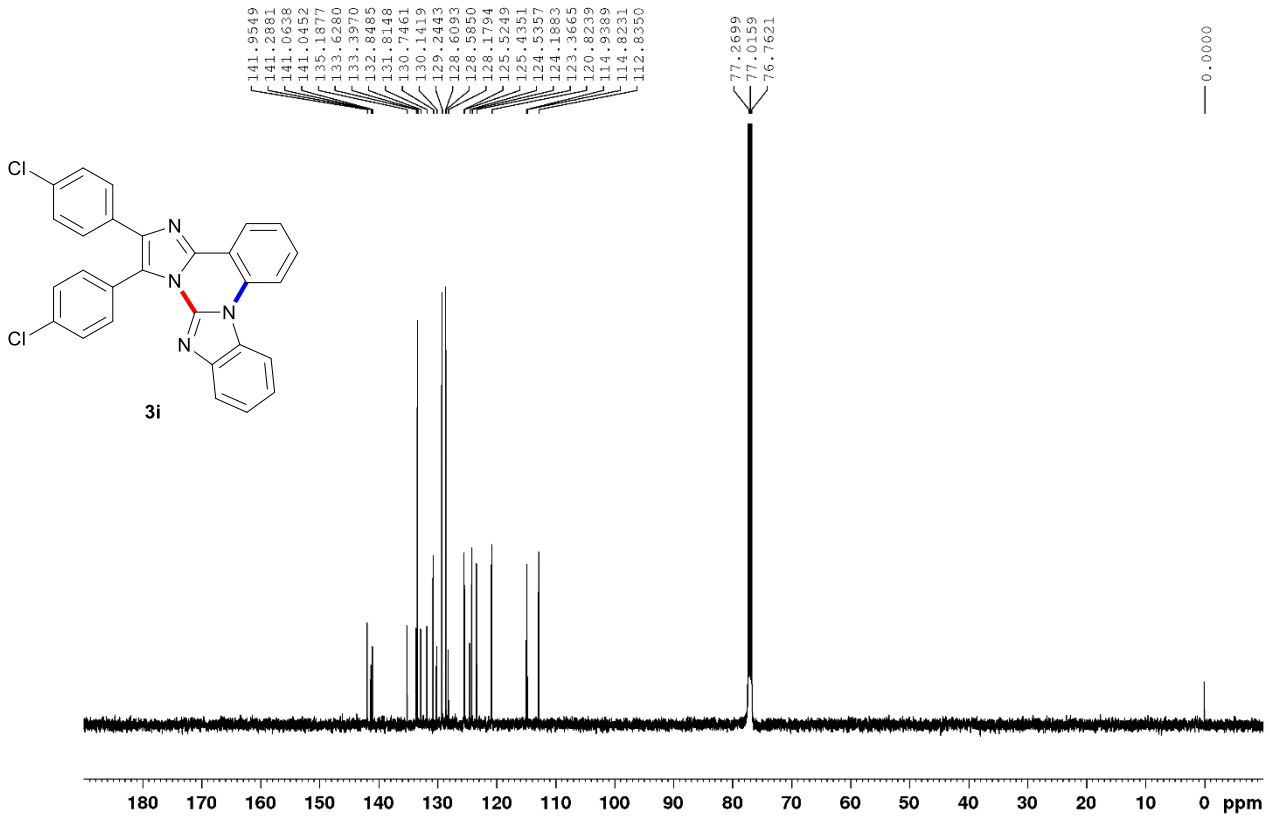
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Cl
N
N
N
N
3i
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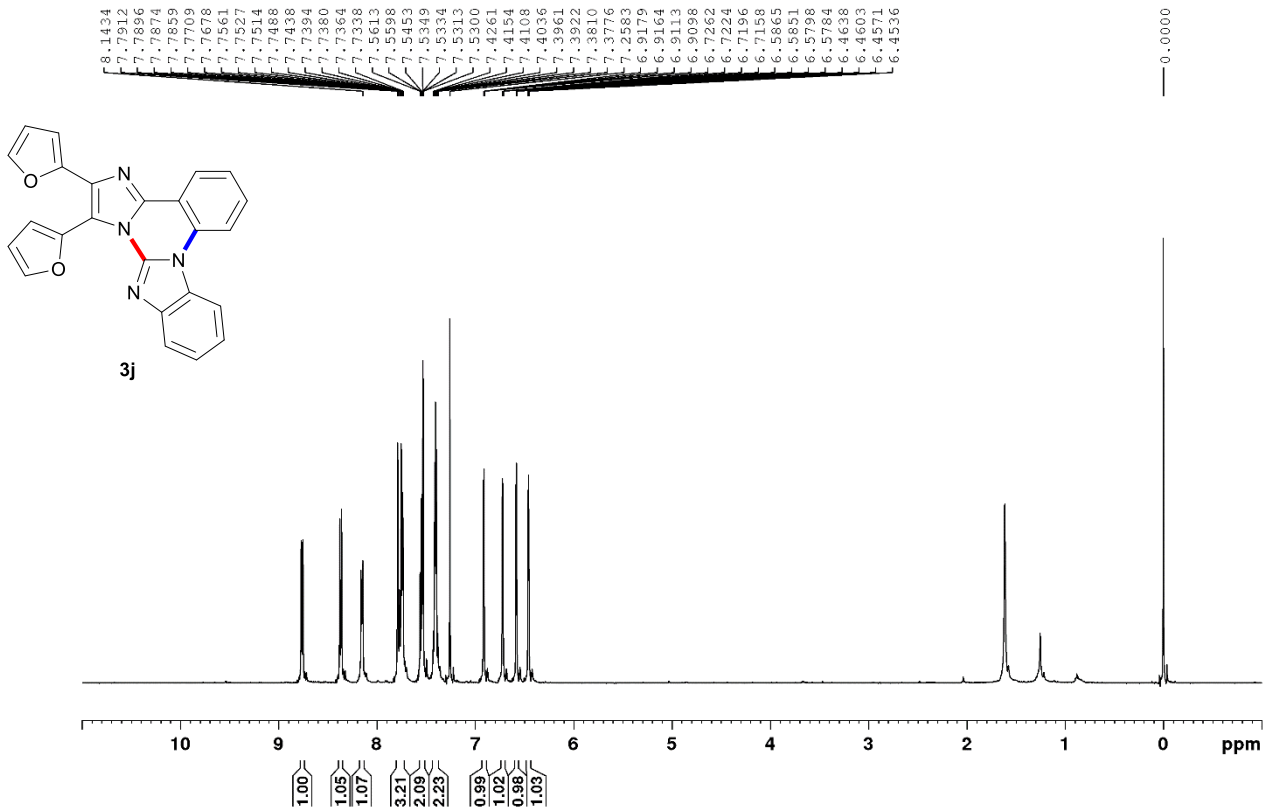
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3j
10
9
8
7
6
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4
3
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2.23
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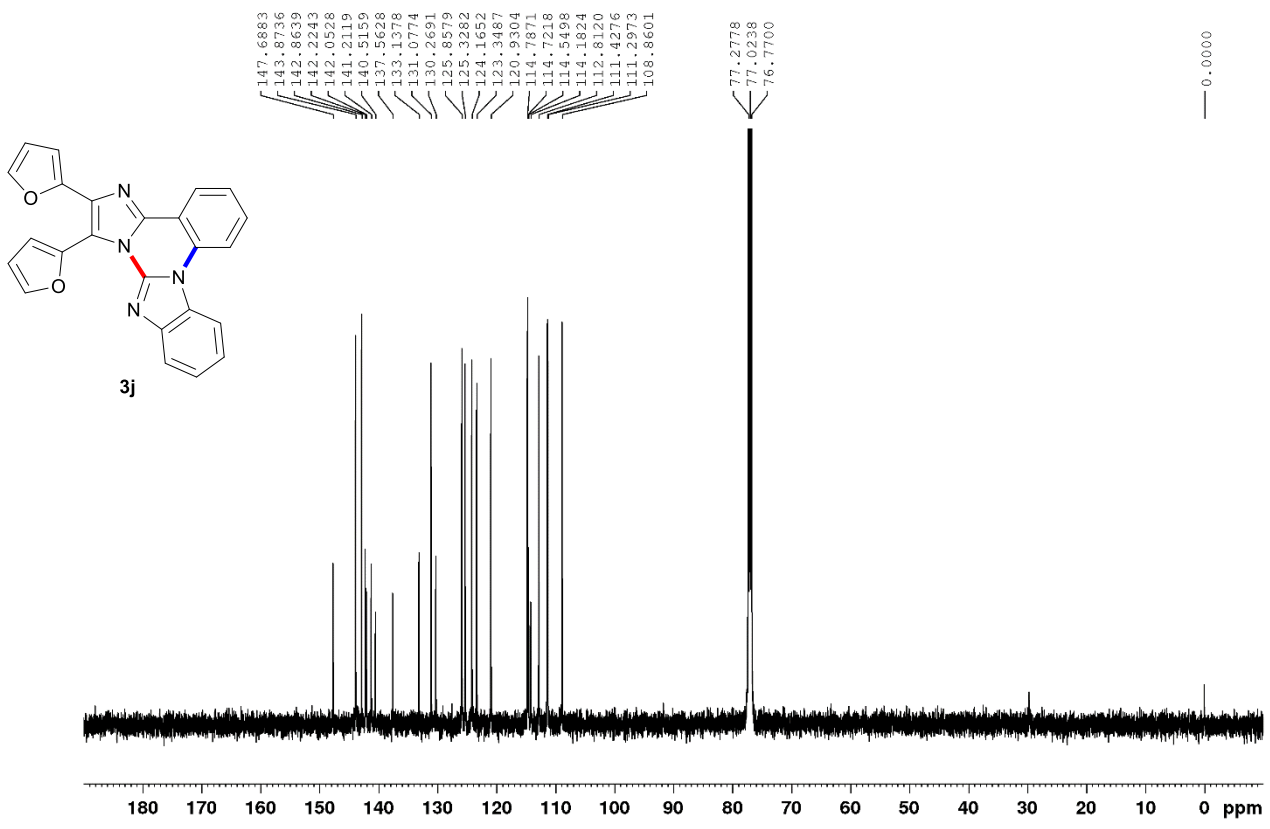
1.03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114.5498
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111.4276
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77.0238
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0.0000
3j
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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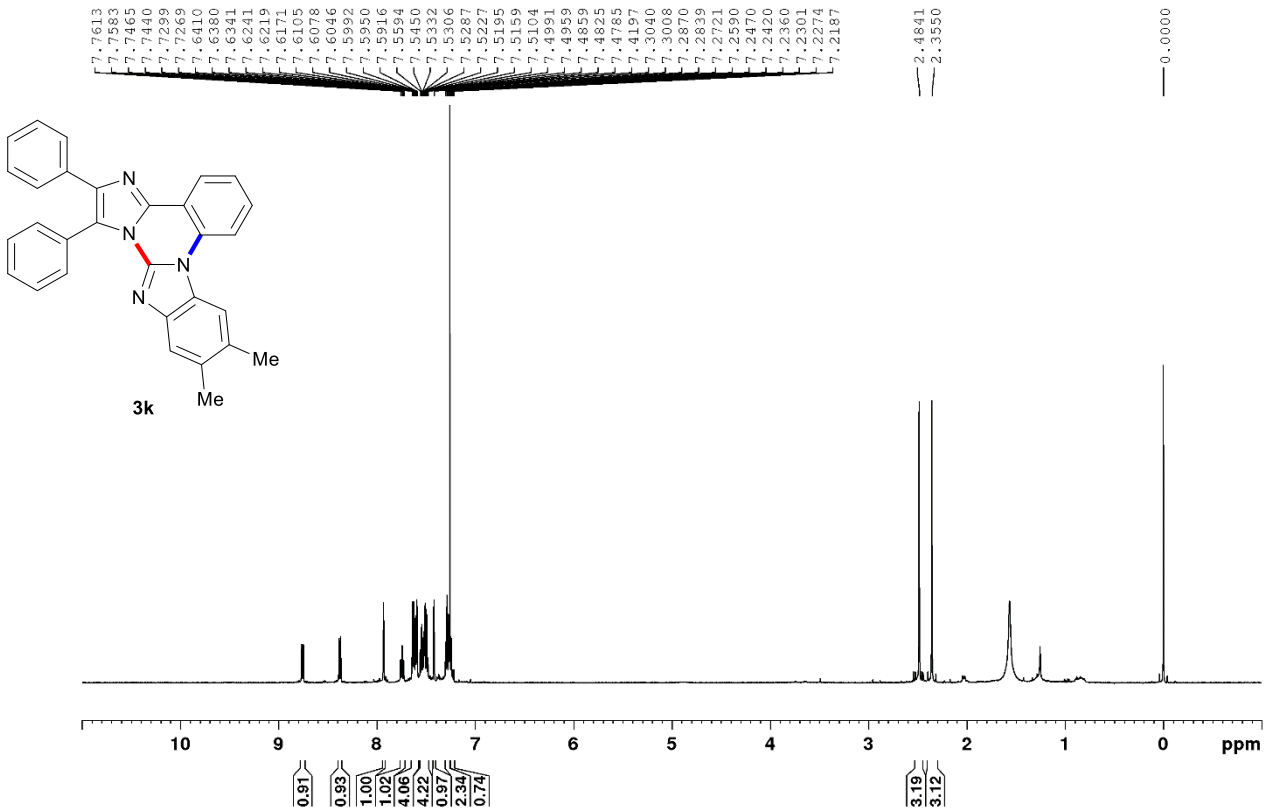
ppm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7.6241
7.6219
7.6171
7.6105
7.6078
7.6046
7.5992
7.5950
7.5916
7.5594
7.5450
7.5332
7.5306
7.5287
7.5227
7.5195
7.5159
7.5104
7.4991
7.4959
7.4859
7.4825
7.4785
7.4197
7.3040
7.3008
7.2870
7.2839
7.2721
7.2590
7.2470
7.2420
7.2360
7.2301
7.2274
7.2187
2.4841
2.3550
0.0000
N
N
N
N
Me
Me
3k
10
9
8
7
6
5
4
3
2
1
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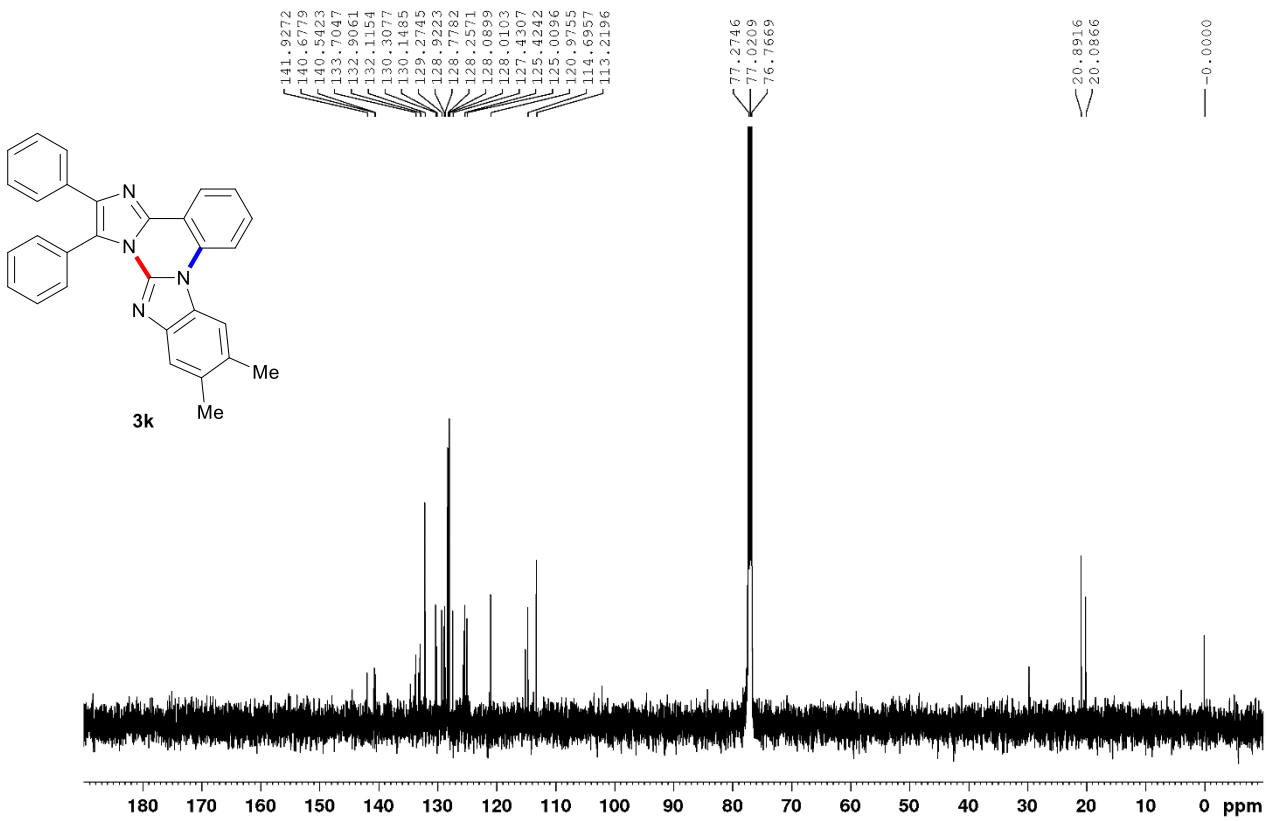
3.12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128.2571
128.0899
128.0103
127.4307
125.4242
125.0096
120.9755
114.6957
113.2196
77.2746
77.0209
76.7669
20.8916
20.0866
0.0000
N
N
N
N
Me
Me
3k
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170
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140
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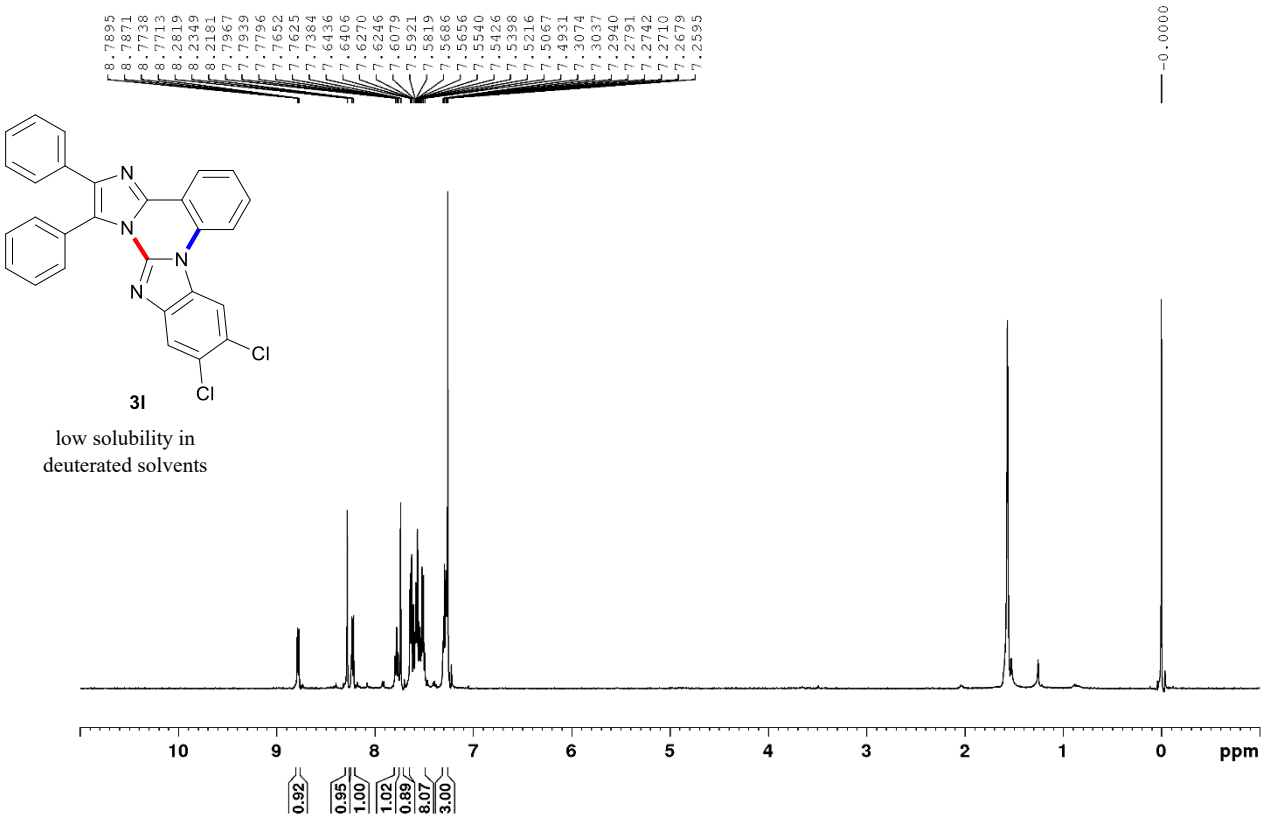
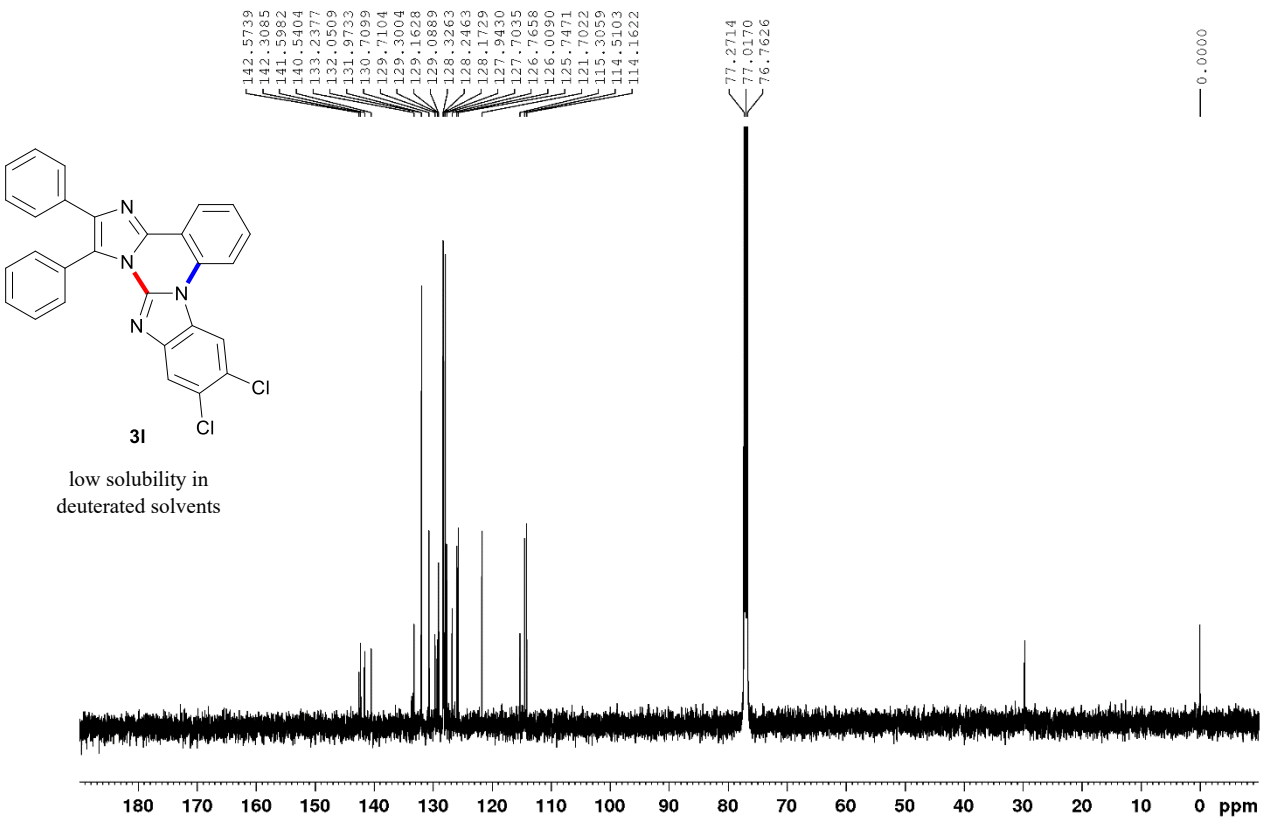
ppm



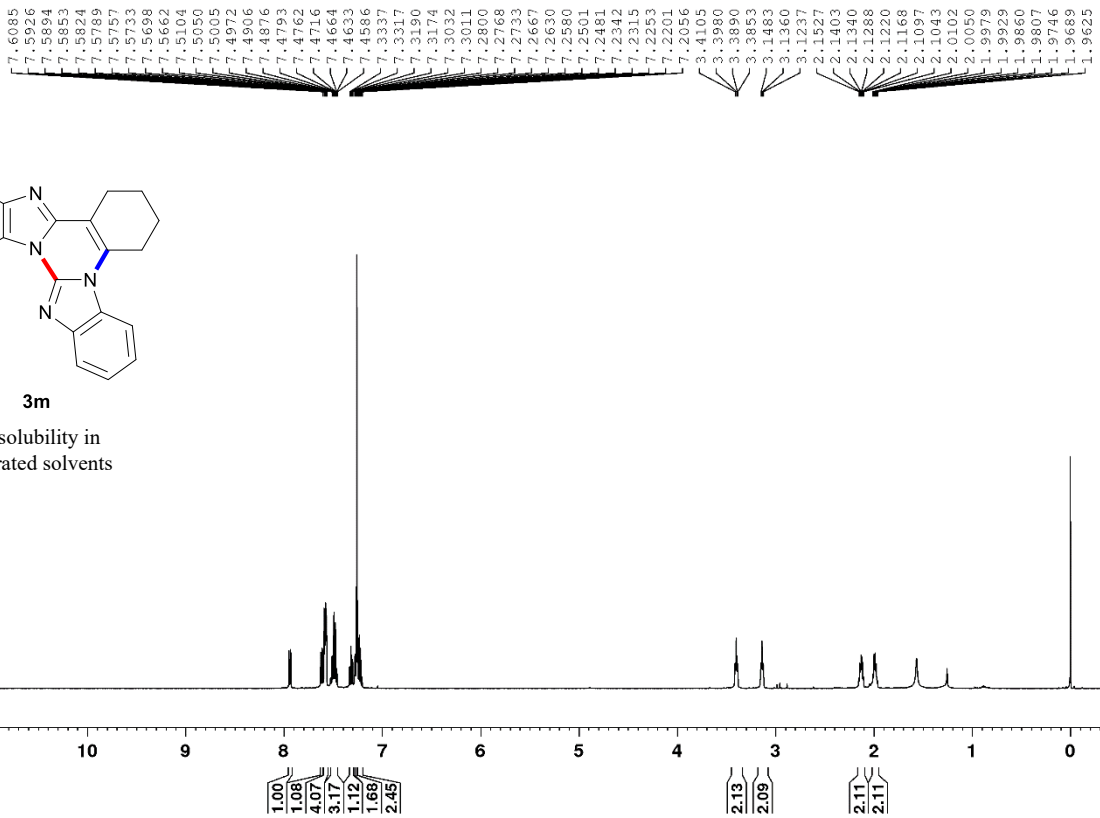
3l

low solubility in deuterated solvents



3l

low solubility in deuterated solvents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7.5824
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7.5757
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7.5662
7.5104
7.5050
7.5005
7.4972
7.4906
7.4876
7.4793
7.4762
7.4716
7.4664
7.4633
7.4586
7.3337
7.3317
7.3190
7.3174
7.3032
7.3011
7.2800
7.2768
7.2733
7.2667
7.2630
7.2580
7.2501
7.2481
7.2342
7.2315
7.2253
7.2201
7.2056
3.4105
3.3980
3.3890
3.3853
3.1483
3.1360
3.1237
2.1527
2.1403
2.1340
2.1288
2.1220
2.1168
2.1097
2.1043
2.0102
2.0050
1.9979
1.9929
1.9860
1.9807
1.9746
1.9689
1.9625
N
N
N
N
3m
low solubility in
deuterated solvents
10
9
8
7
6
5
4
3
2
1
0
ppm
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4.07
3.17
1.12
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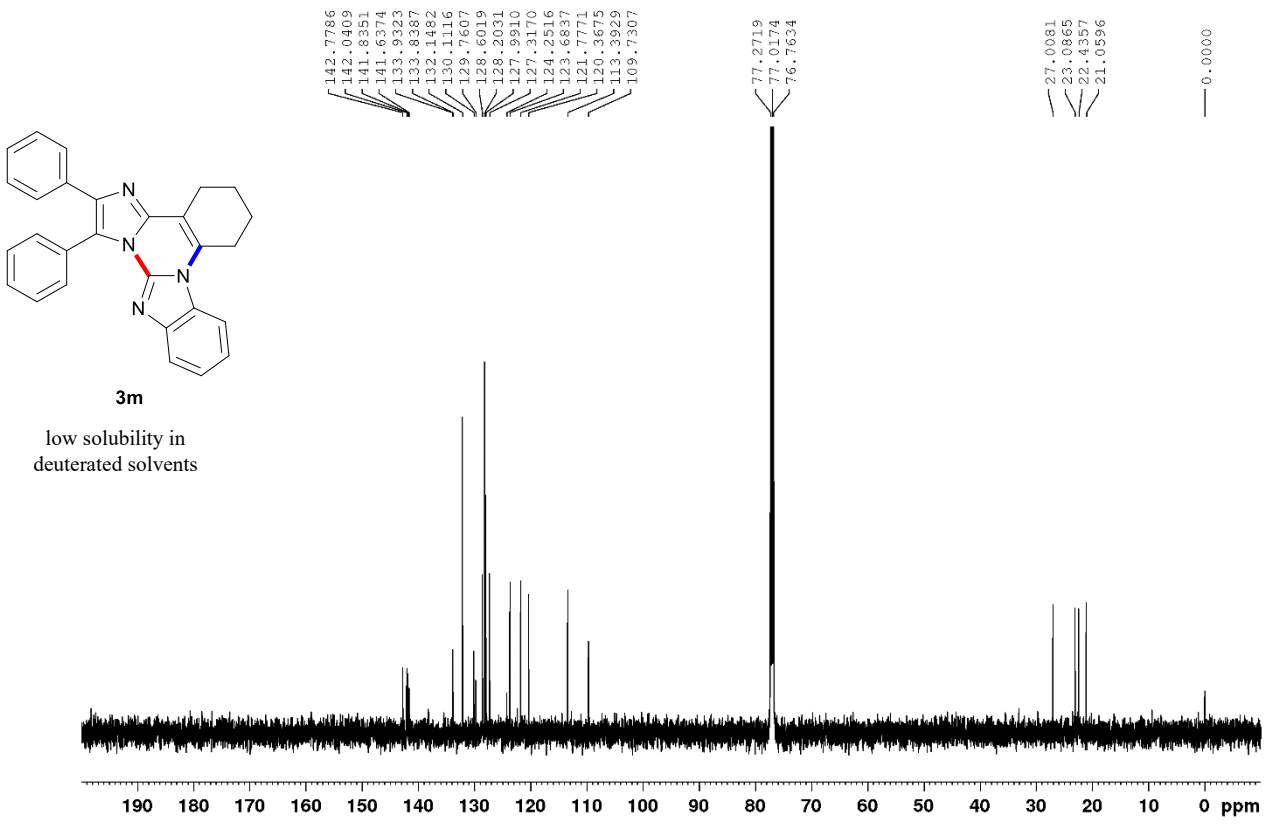
2.13
2.09
2.11
2.11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132.1482
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129.7607
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128.2031
127.9910
127.3170
124.2516
123.6837
121.7771
120.3675
113.3929
109.7307
77.2719
77.0174
76.7634
27.0081
23.0865
22.4357
21.0596
0.0000
N
N
N
N
3m
low solubility in
deuterated solvents
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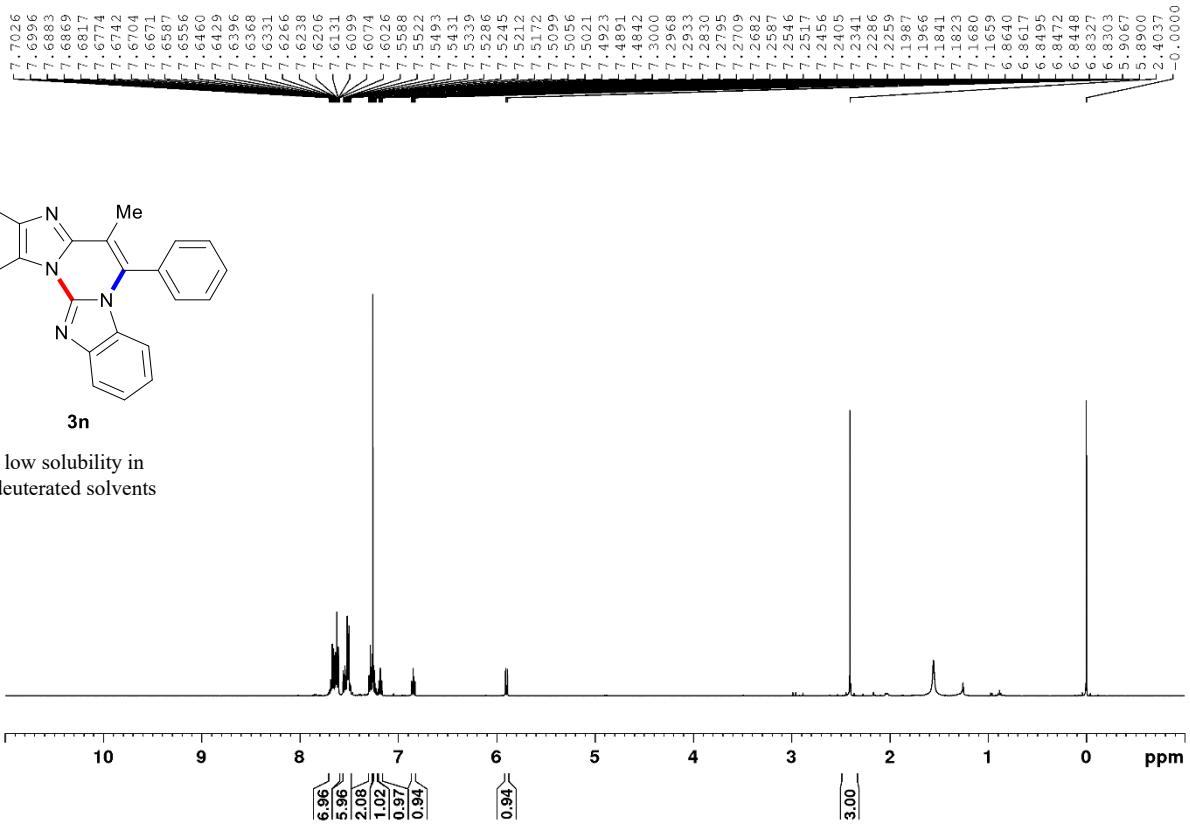
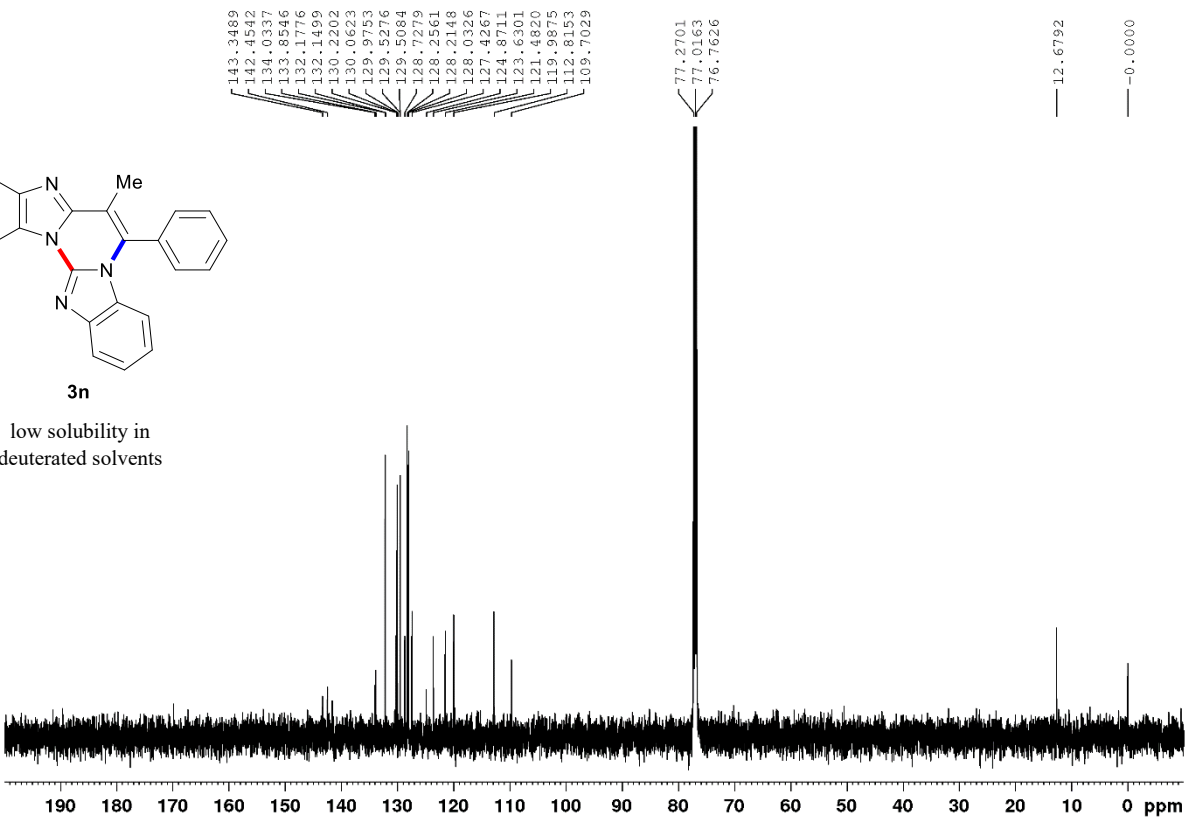
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



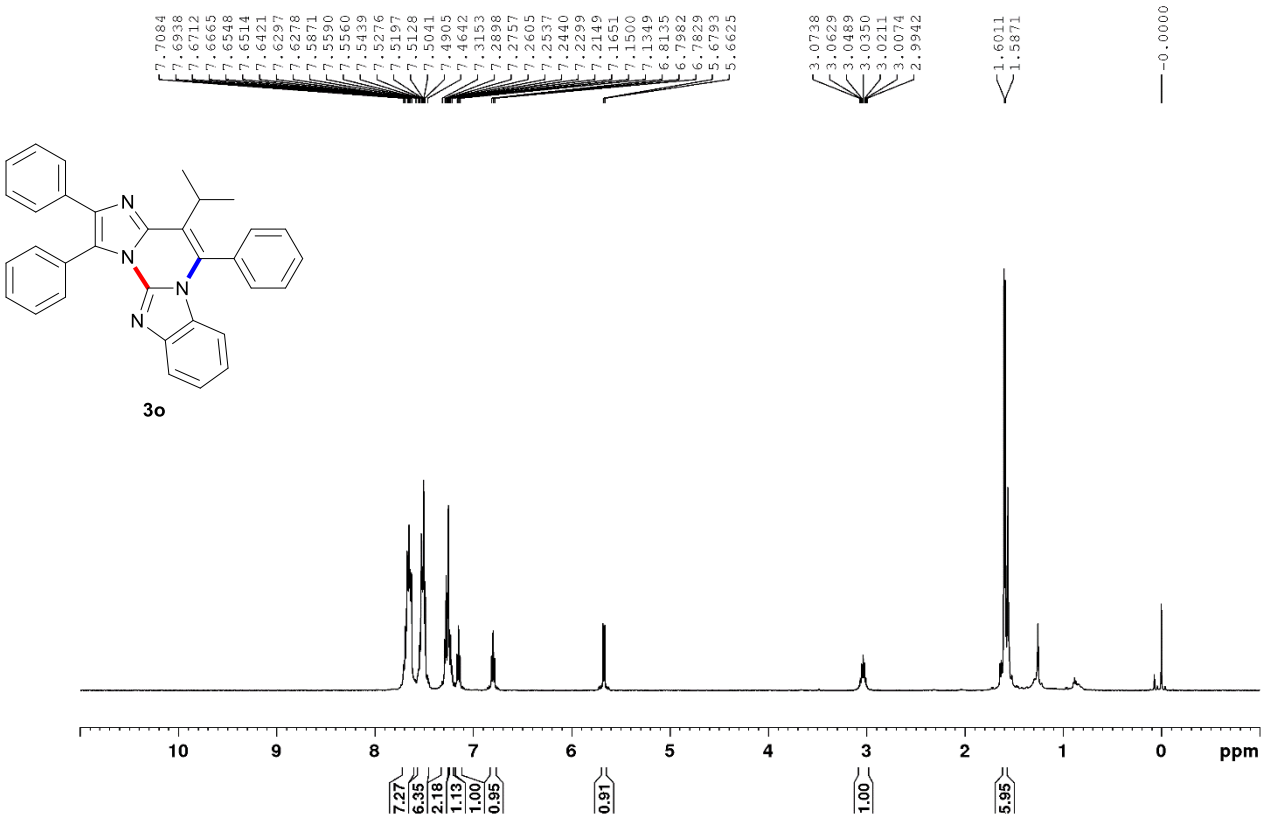
3n

low solubility in deuterated solvents



3n

low solubility in deuterated solvents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7.6665
7.6548
7.6514
7.6421
7.6297
7.6278
7.5871
7.5590
7.5560
7.5439
7.5276
7.5197
7.5128
7.5041
7.4905
7.4642
7.3153
7.2898
7.2757
7.2605
7.2537
7.2440
7.2299
7.2149
7.1651
7.1500
7.1349
6.8135
6.7982
6.7829
5.6793
5.6625
3.0738
3.0629
3.0489
3.0350
3.0211
3.0074
2.9942
1.6011
1.5871
-0.0000
3o
10
9
8
7
6
5
4
3
2
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ppm
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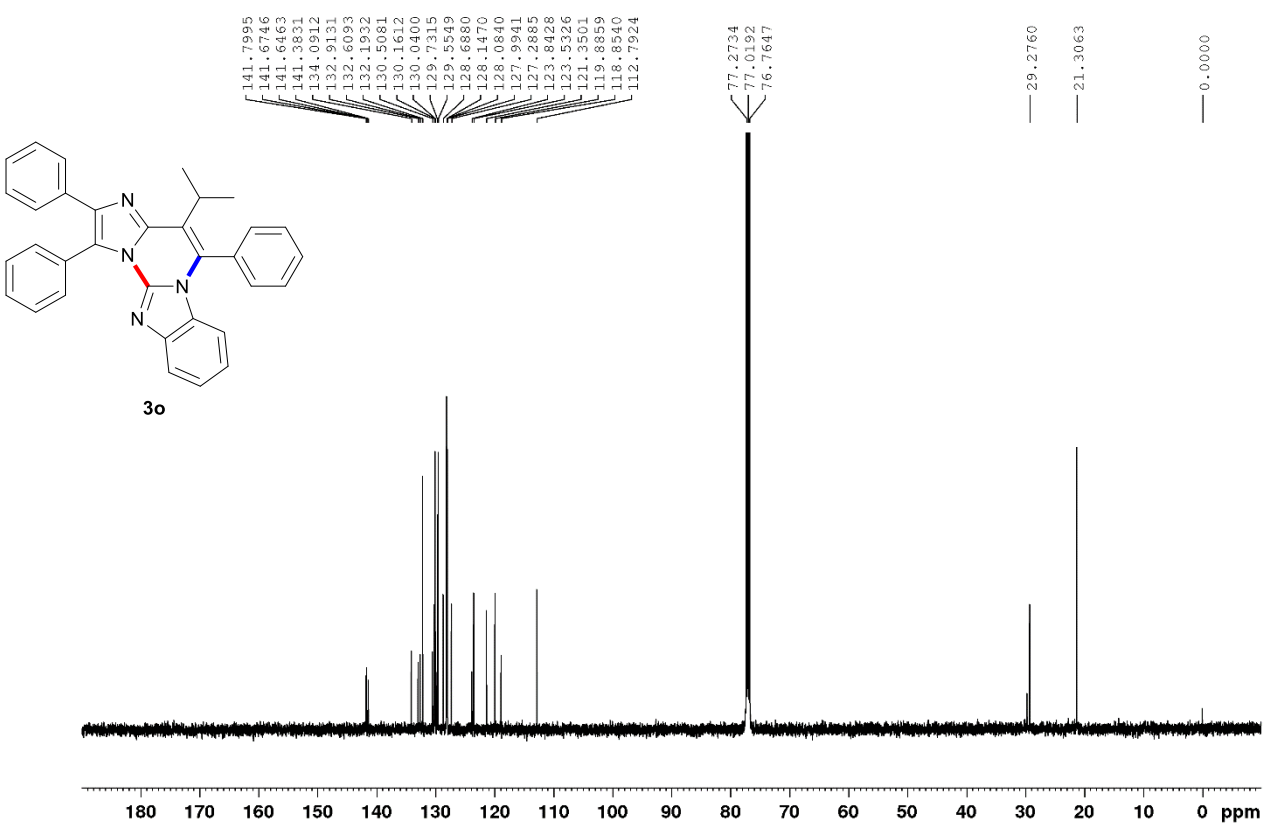
2.18
1.13
1.00
0.95
0.91
1.00
5.95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141.6463
141.3831
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132.6093
132.1932
130.5081
130.1612
130.0400
129.7315
129.5549
128.6880
128.1470
128.0840
127.9941
127.2885
123.8428
123.5326
121.3501
119.8859
118.8540
112.7924
77.2734
77.0192
76.7647
29.2760
21.3063
0.0000
3o
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170
160
150
140
130
120
110
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60
50
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30
20
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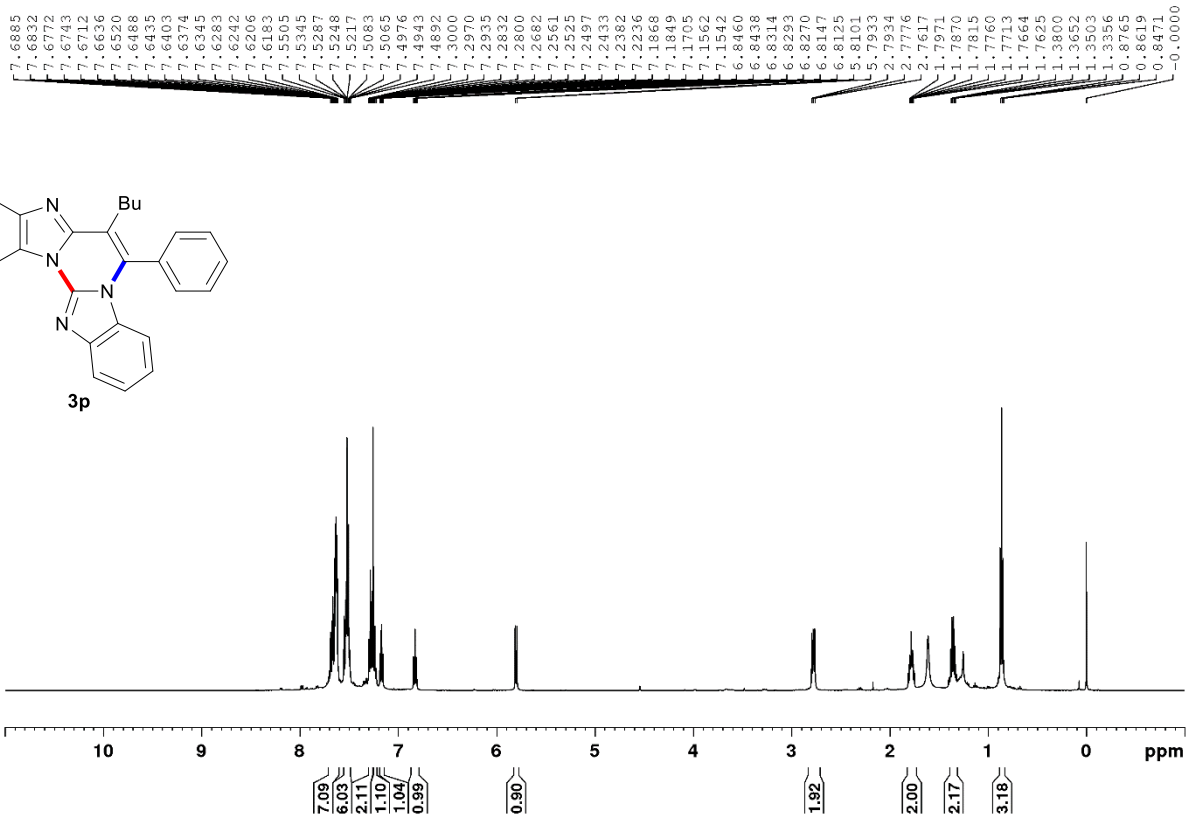
0
ppm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7.6374
7.6345
7.6283
7.6242
7.6206
7.6183
7.5505
7.5345
7.5287
7.5248
7.5217
7.5083
7.5065
7.4976
7.4943
7.4892
7.3000
7.2970
7.2935
7.2832
7.2800
7.2682
7.2561
7.2525
7.2497
7.2433
7.2382
7.2236
7.1868
7.1849
7.1705
7.1562
7.1542
6.8460
6.8438
6.8314
6.8293
6.8270
6.8147
6.8125
5.8101
5.7933
2.7934
2.7776
2.7617
1.7971
1.7870
1.7815
1.7760
1.7713
1.7664
1.7625
1.3800
1.3652
1.3503
1.3356
0.8765
0.8619
0.8471
-0.0000
N
N
N
N
Bu
3p
10
9
8
7
6
5
4
3
2
1
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ppm
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2.11
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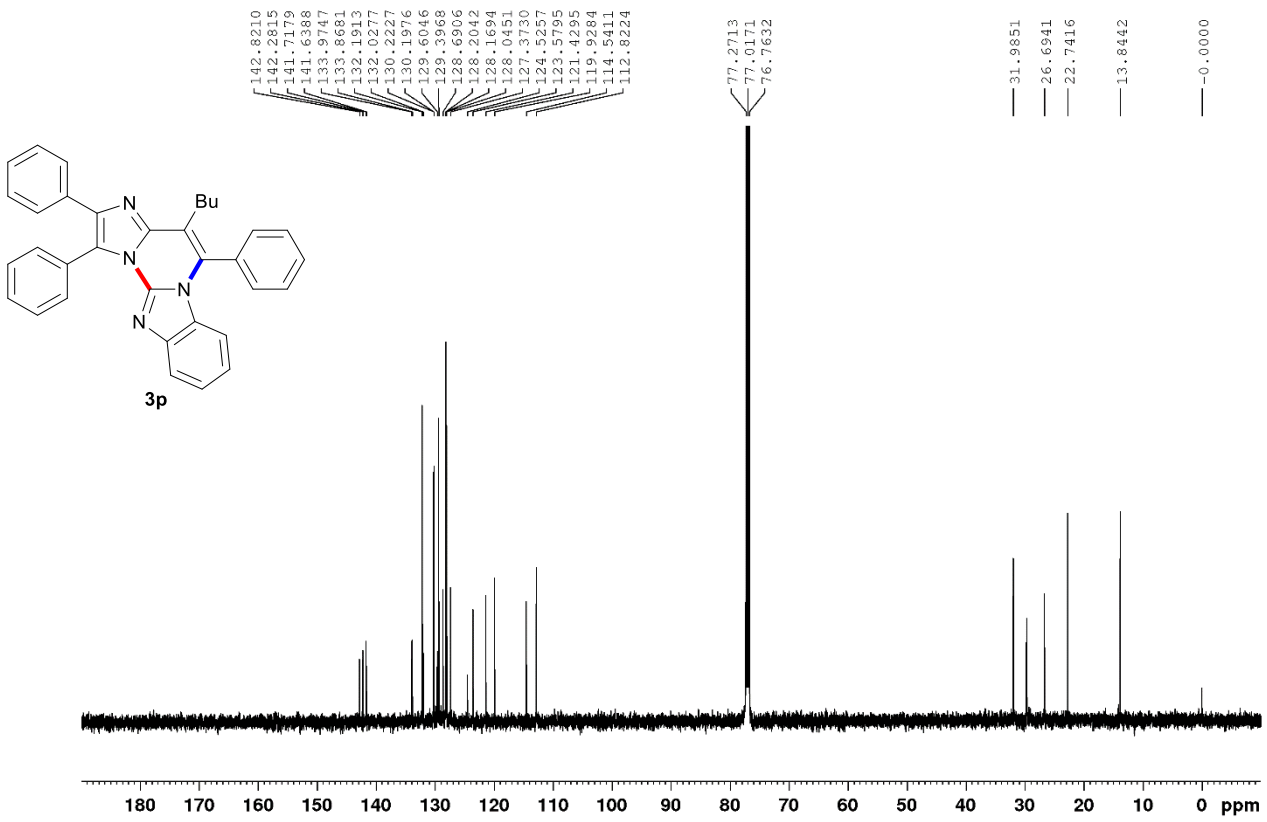
3.18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129.6046
129.3968
128.6906
128.2042
128.1694
128.0451
127.3730
124.5257
123.5795
121.4295
119.9284
114.5411
112.8224
77.2713
77.0171
76.7632
31.9851
26.6941
22.7416
13.8442
-0.0000
N
N
N
N
Bu
3p
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170
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140
130
120
110
100
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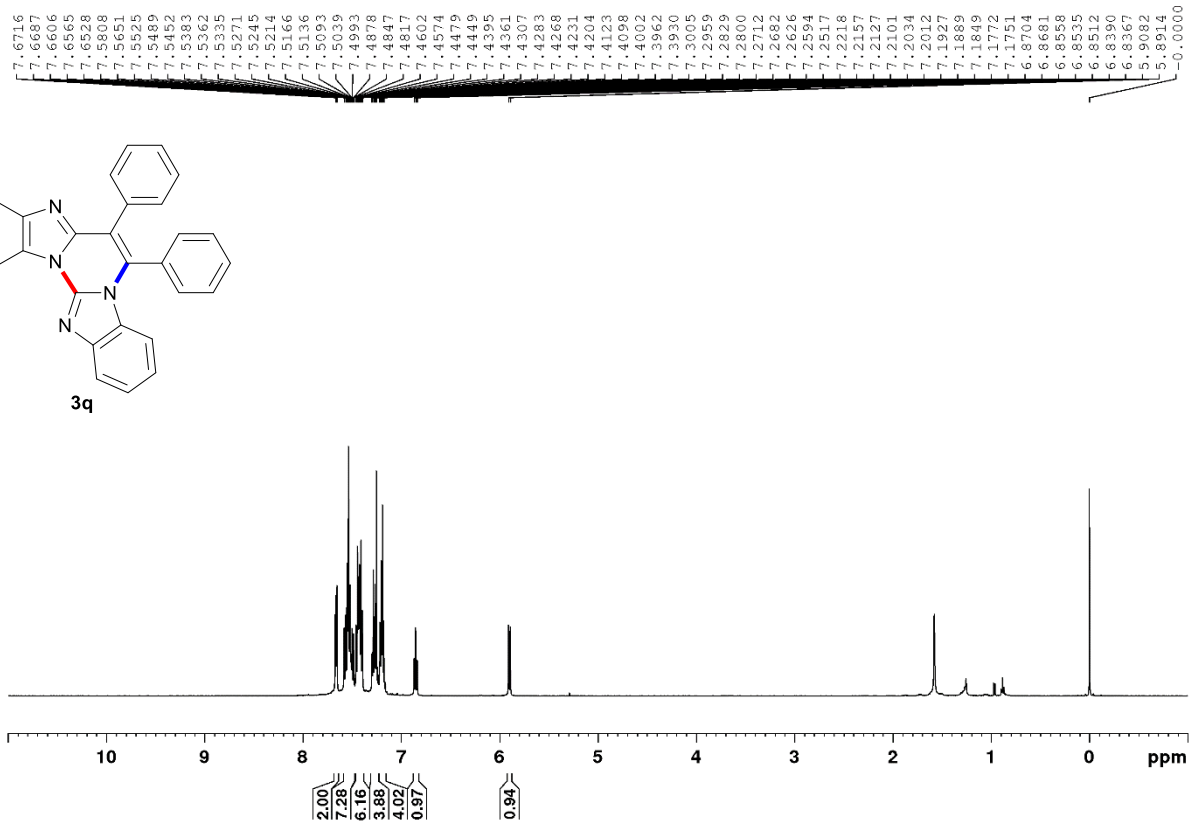
80
70
60
50
40
30
20
10
0
ppm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7.5651
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7.5383
7.5362
7.5335
7.5271
7.5245
7.5214
7.5166
7.5136
7.5093
7.5039
7.4993
7.4878
7.4847
7.4817
7.4602
7.4574
7.4479
7.4449
7.4395
7.4361
7.4307
7.4283
7.4268
7.4231
7.4204
7.4123
7.4098
7.4002
7.3962
7.3930
7.3005
7.2959
7.2829
7.2800
7.2712
7.2682
7.2626
7.2594
7.2517
7.2218
7.2157
7.2127
7.2101
7.2034
7.2012
7.1927
7.1889
7.1849
7.1772
7.1751
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6.8681
6.8558
6.8535
6.8512
6.8390
6.8367
5.9082
5.8914
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3q
10
9
8
7
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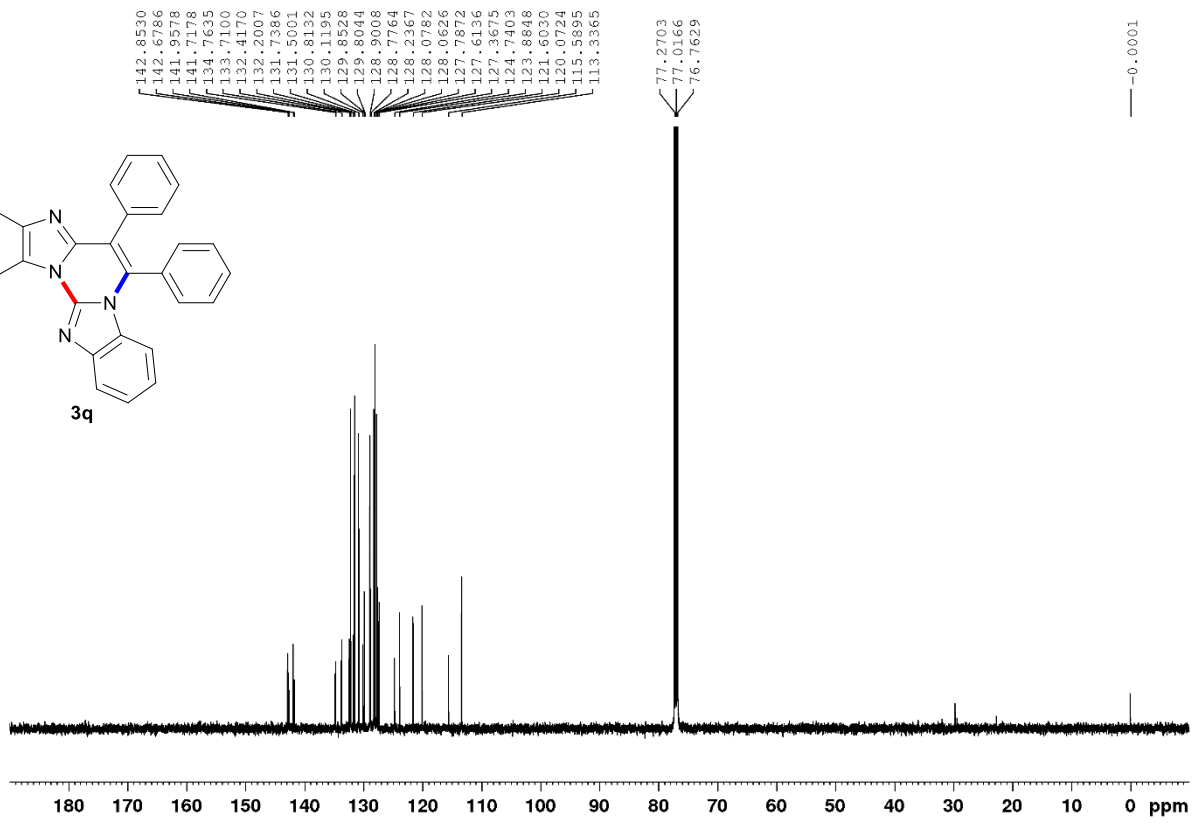
0.94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131.5001
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130.1195
129.8528
129.8044
128.9008
128.7764
128.2367
128.0782
128.0626
127.7872
127.6136
127.3675
124.7403
123.8848
121.6030
120.0724
115.5895
113.3365
77.2703
77.0166
76.7629
-0.0001
3q
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170
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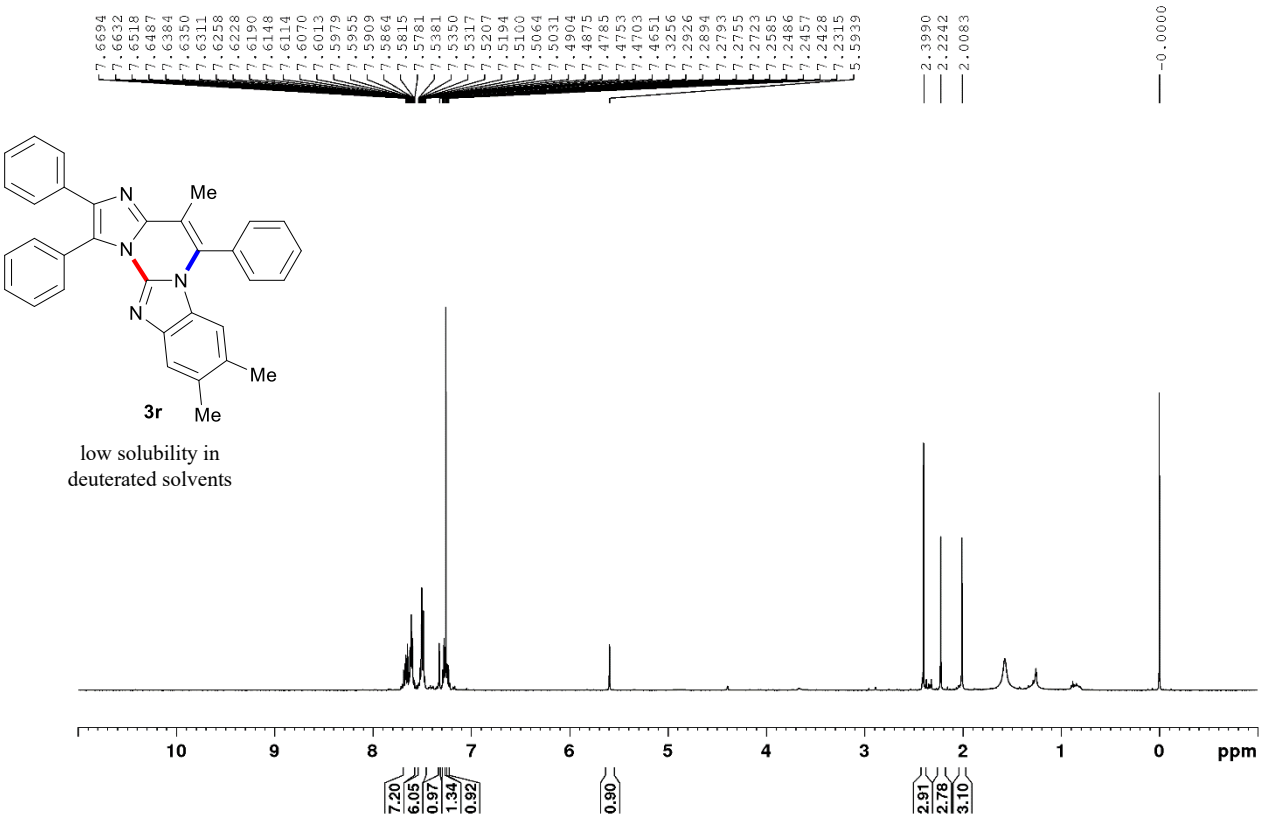
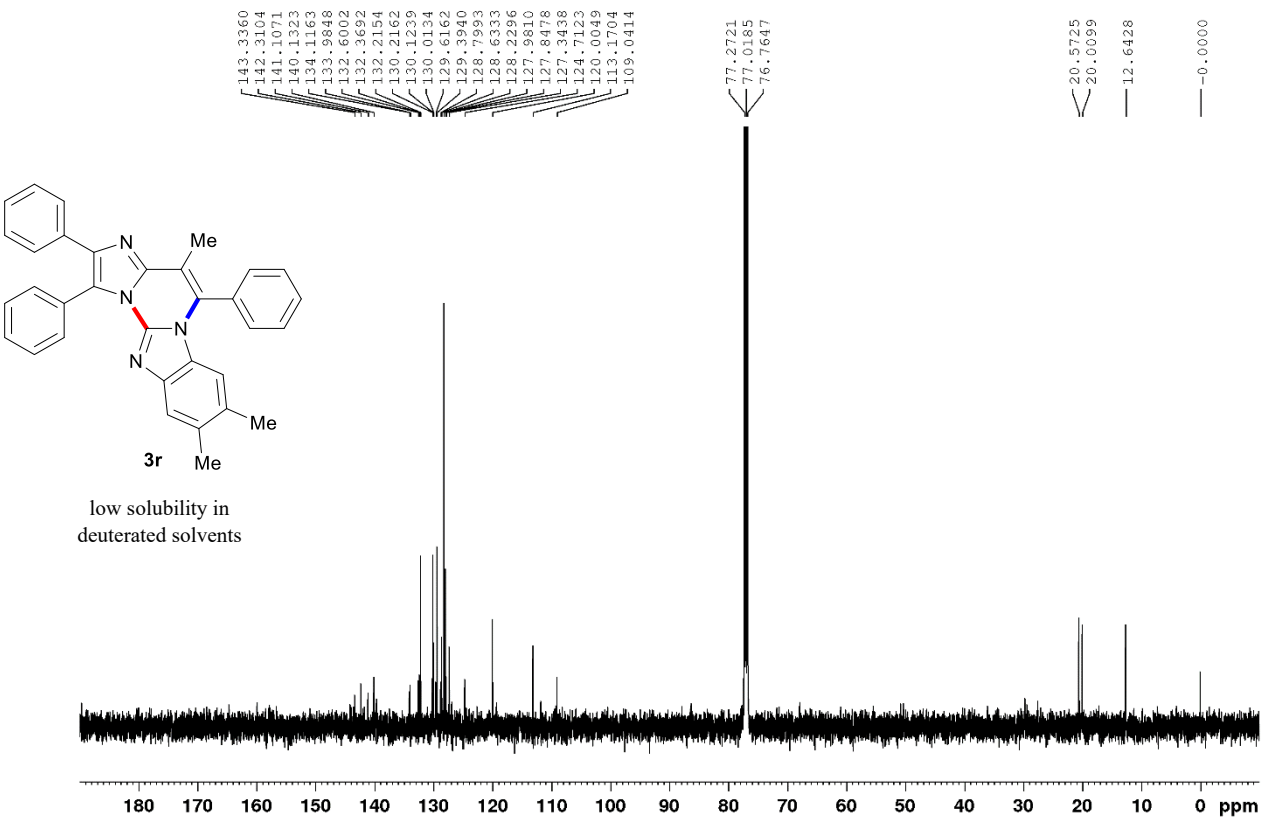
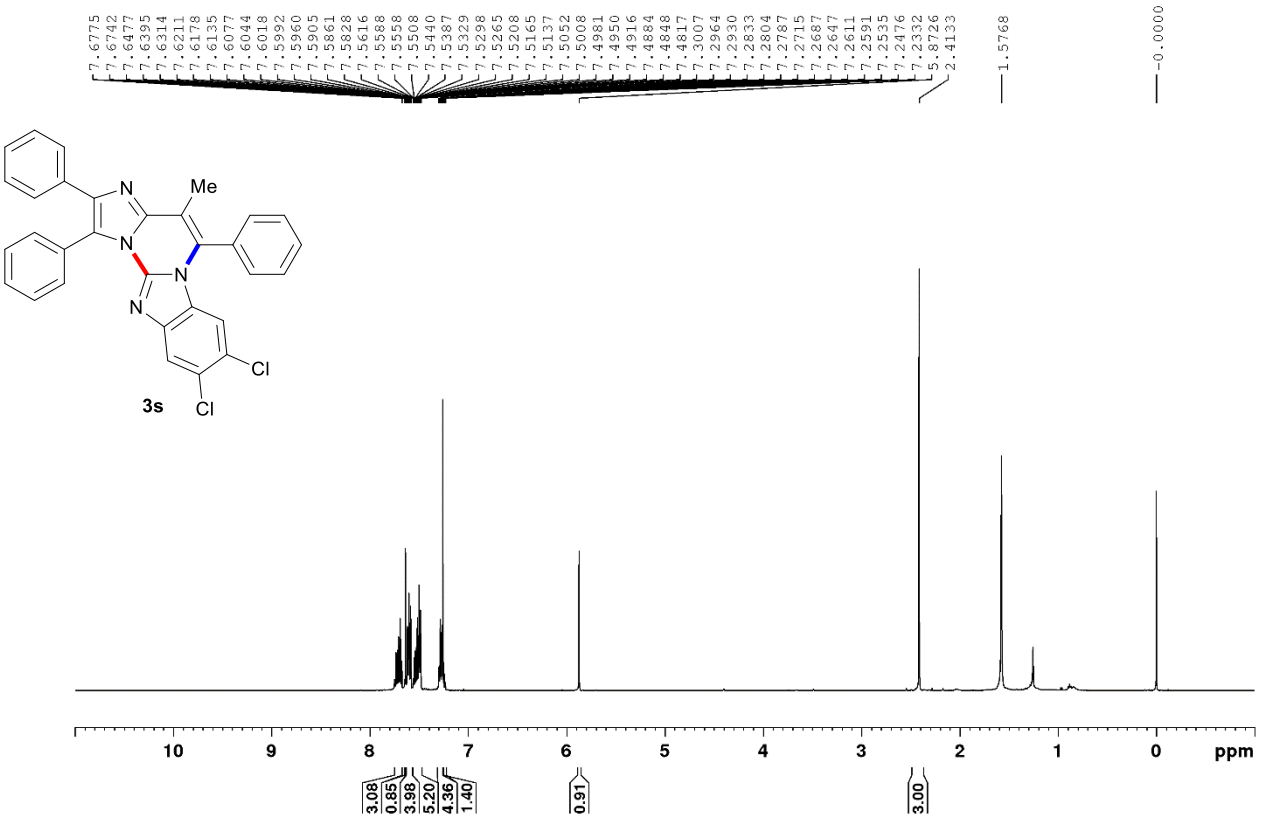
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


3r
low solubility in
deuterated solvents
ppm


3r
low solubility in
deuterated solvents
ppm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7.6077
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7.5960
7.5905
7.5861
7.5828
7.5616
7.5588
7.5558
7.5508
7.5440
7.5387
7.5329
7.5298
7.5265
7.5208
7.5165
7.5137
7.5052
7.5008
7.4981
7.4950
7.4916
7.4884
7.4848
7.4817
7.3007
7.2964
7.2930
7.2833
7.2804
7.2787
7.2715
7.2687
7.2647
7.2611
7.2591
7.2535
7.2476
7.2332
5.8726
2.4133
1.5768
0.0000
Me
N
N
N
N
Cl
Cl
3s
10
9
8
7
6
5
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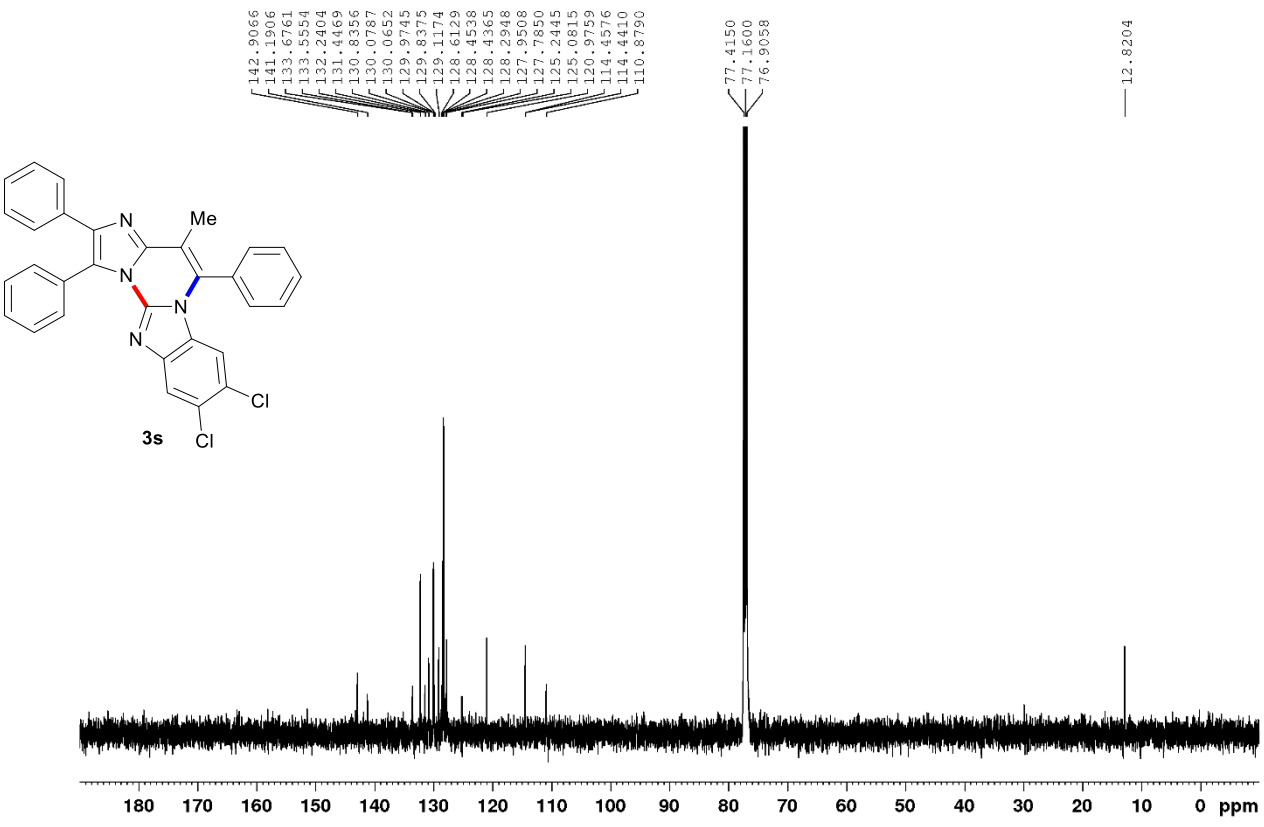
3.00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129.1174
128.6129
128.4538
128.4365
128.2948
127.9508
127.7850
125.2445
125.0815
120.9759
114.4576
114.4410
110.8790
77.4150
77.1600
76.9058
12.8204
Me
N
N
N
N
Cl
Cl
3s
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140
130
120
110
100
90
80
70
60
50
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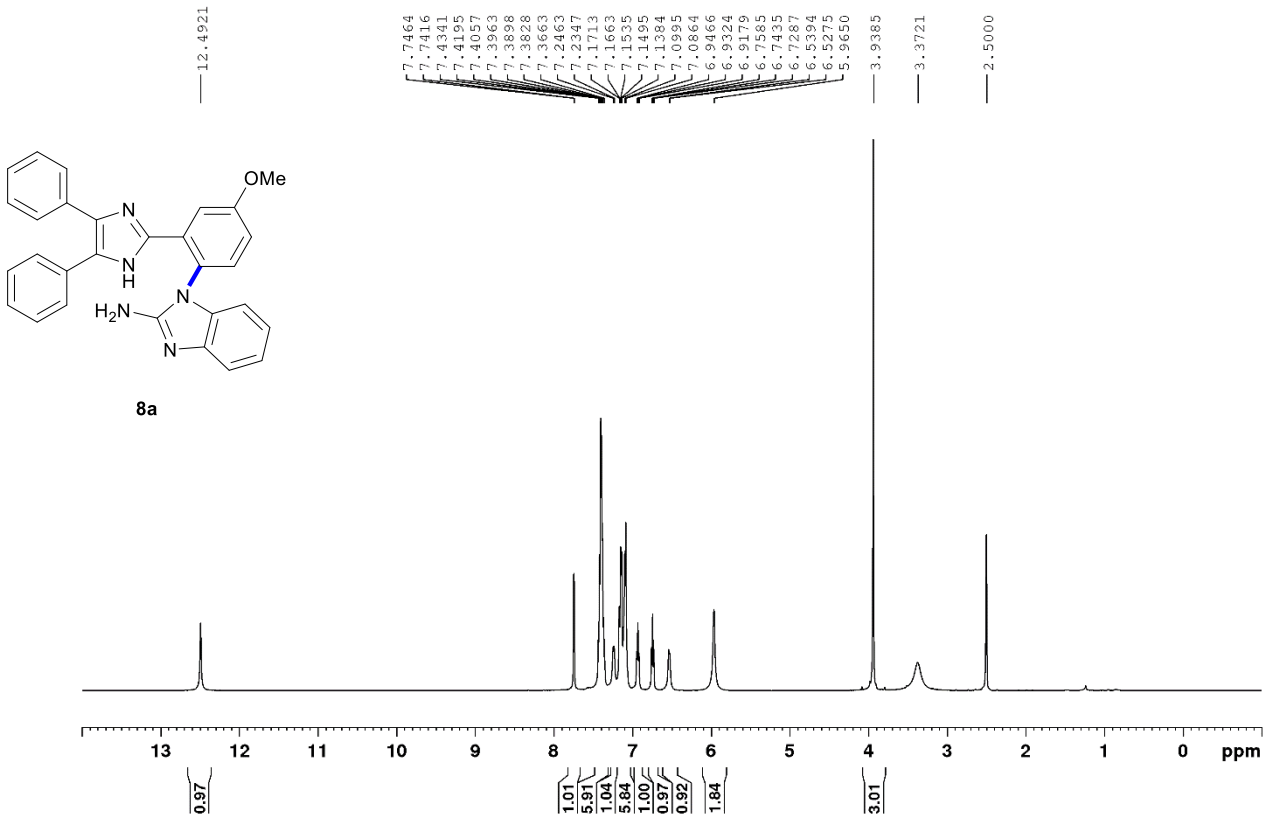
30
20
10
0
ppm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7.3828
7.3663
7.2463
7.2347
7.1713
7.1663
7.1535
7.1495
7.1384
7.0995
7.0864
6.9466
6.9324
6.9179
6.7585
6.7435
6.7287
6.5394
6.5275
5.9650
3.9385
3.3721
2.5000
OMe
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N
H
N
H2N
N
8a
13
12
11
10
9
8
7
6
5
4
3
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ppm
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5.91
1.04
5.84
1.00
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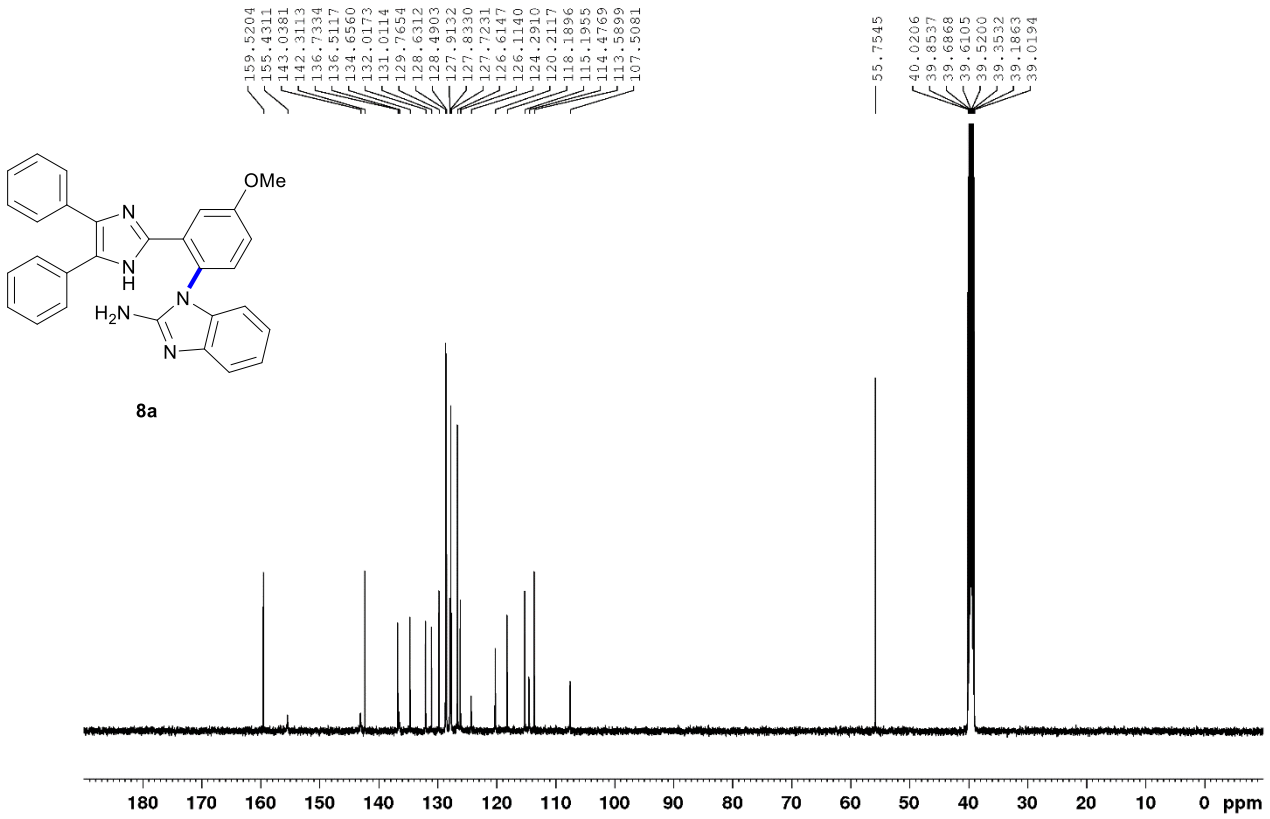
0.92
1.84
3.01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131.0114
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128.4903
127.9132
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127.7231
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126.1140
124.2910
120.2117
118.1896
115.1955
114.4769
113.5899
107.5081
55.7545
40.0206
39.8537
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39.6105
39.5200
39.3532
39.1863
39.0194
OMe
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H
N
H2N
N
8a
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120
110
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80
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50
40
30
20
10
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ppm