Copper-catalyzed synthesis of 2-aminobenzothiazoles from 2-iodophenyl isocyanides, Potassium sulfide and amines

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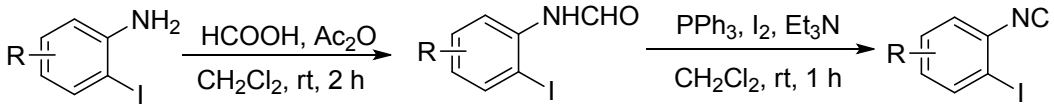
Supporting Information

1) General Information

NMR spectra of the products **3a-3v** were obtained using Bruker Avance-500 instruments, calibrated to TMS (^{1}H NMR spectra) and CD(H)Cl_3 (^{13}C NMR spectra) as the internal reference (0.00 ppm for ^{1}H NMR spectra and 77.00 ppm for ^{13}C NMR spectra). High-resolution massspectra. (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). Reactions were monitored by thin-layer chromatography. Column chromatography (petroleum ether/ethyl acetate) was performed on silica gel(200-300 mesh).

2) Synthesis of Starting Materials Preparation of 1a-1j:[1,2]



General Procedure for the Synthesis of o-iodoformanilides[1]

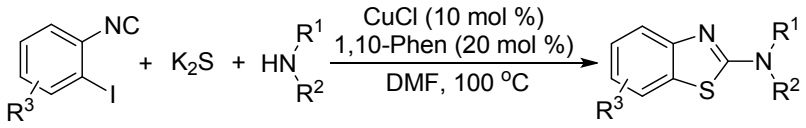
To formic acid (2.41 mL, 63.9 mmol) was added acetic anhydride (2.16 mL, 22.8 mmol) and the mixure was stirred at room temperature. After stirring for 10 min, to the mixture was added the solution of *o*-iodoaniline (3.99 g, 18.3mmol) in CH_2Cl_2 (60 mL) and the mixture was stirred at room temperature. After stirring for 2 h, the mixture was concentrated under reduced pressure to give *o*-iodoformanilides (4.42 g, 98%) as a white solid

General Procedure for the Synthesis of 2-iodophenyl isocyanides[2]

To a stirred solution of formamide **1** (1.0 mmol) and I_2 (381 mg, 1.5 mmol) in CH_2Cl_2 (3 mL) was added Ph_3P (394 mg, 1.5 mmol), followed by the dropwise addition of Et_3N (415 μL, 301 mg, 3 mmol). The mixture was stirred at r.t. until the consumption of the formamide was complete (TLC monitoring, typically 1 h), and then diluted with CH_2Cl_2 (10 mL) and washed with ice-cold sat. aq $Na_2S_2O_3$ solution (10 mL). The aq phase was extracted with CH_2Cl_2 (2 × 10 mL). Each portion of the organic phase was sequentially washed with deionized H_2O (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4, and filtered. The combined organic phase was concentrated under reduced pressure and purified by column chromatography (silica

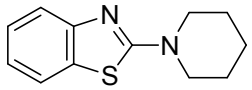
gel, typically PE as the eluent) to give the corresponding 2-iodophenyl isocyanides

3) Typical Procedures

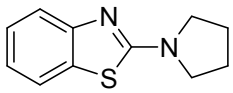


To a schlenk tube were added 2-iodophenyl isocyanides (0.3 mmol), K_2S (3equiv), amines (0.3 mmol), CuCl (10 mol%), 1,10-Phen (20 mol%), and DMF (2 mL). the mixture was stirred at 100 °C (oil bath temperature) for the indicated time until complete consumption of starting material as monitored by TLC. After the reaction was finished, the reaction mixture was cooled to room temperature, diluted in ethyl acetate, and washed with water. The aqueous phase was re-extracted with ethylacetate. The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate as eluent to afford the desired product.

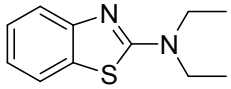
4) Characterization Data



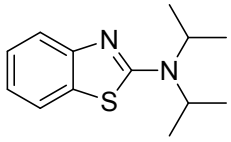
2-(piperidin-1-yl)benzo[d]thiazole(3a):[3] The product was purified by flash chromatography to give 62 mg (95%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.56 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 3.58 (s, 4H), 1.66 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.8, 152.8, 130.5, 125.8, 121.0, 120.5, 118.7, 49.5, 25.2, 24.1.



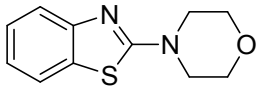
2-(pyrrolidin-1-yl)benzo[d]thiazole(3b):[3] The product was purified by flash chromatography to give 60 mg (98%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.58 (t, *J* = 8.0 Hz, 2H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 3.54 (t, *J* = 6.5 Hz, 4H), 2.03-2.01 (m, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 165.2, 153.0, 130.5, 125.8, 120.5(2C), 118.4, 49.4, 25.5.



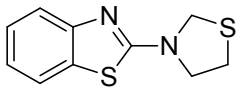
N,N-diethylbenzo[d]thiazol-2-amine(3c):[4] The product was purified by flash chromatography to give 58 mg (94%) as a colourless oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.55 (t, *J* = 8.0 Hz, 2H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 3.57-3.52 (m, 4H), 1.26 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 167.2, 153.2, 130.5, 125.7, 120.6, 120.4 118.4, 45.3, 12.8.



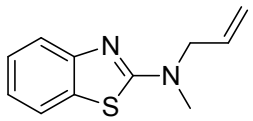
N,N-diisopropylbenzo[d]thiazol-2-amine(3d):[4] The product was purified by flash chromatography to give 48 mg (68%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.56 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 4.01-3.93 (m, 2H), 1.41 (d, *J* = 7.0 Hz, 12H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 165.4, 153.0, 129.7, 125.5, 120.4, 120.0, 118.5, 50.8, 20.3.



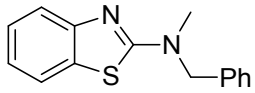
4-(benzo[d]thiazol-2-yl)morpholine(3e):[3] The product was purified by flash chromatography to give 62 mg (94%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.61 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 3.81 (t, *J* = 5 Hz, 4H), 3.60 (t, *J* = 5 Hz, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.9, 152.4, 130.5, 126.0, 121.6, 120.7, 119.2, 66.2, 48.4.



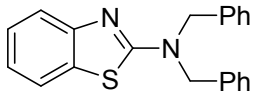
2-(thiazolidin-3-yl)benzo[d]thiazole(3f): The product was purified by flash chromatography to give 55 mg (82%) as a yellow solid. mp: 105.3-107.1 ^{o}C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.61 (t, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 4.69 (s, 2H) 3.88 (t, *J* = 6.0 Hz, 2H), 3.15 (t, *J* = 6.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 165.0, 152.1, 130.6, 126.1, 121.7, 120.8, 119.2, 52.6, 51.8, 30.6.



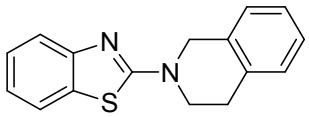
N-allyl-N-methylbenzo[d]thiazol-2-amine(3g): The product was purified by flash chromatography to give 58 mg (95%) as a colourless oil. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.57 (t, *J* = 7.5 Hz, 2H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 5.90-5.82 (m, 1H), 5.26 (d, *J* = 7.5 Hz, 1H), 5.23 (s, 1H), 4.12 (d, *J* = 5.5 Hz, 2H), 3.14 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.4, 152.8, 131.8, 130.6, 125.9, 121.0, 120.5, 118.7, 117.9, 55.4, 37.6. HRMS (ESI) m/z calcd for $C_{11}H_{13}N_2S^+$(M+H)$^+$205.07940, found 205.07928.



N-benzyl-N-methylbenzo[d]thiazol-2-amine(3h):[5] The product was purified by flash chromatography to give 74 mg (97%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.58 (t, *J* = 8.5 Hz, 2H), 7.33-7.27 (m, 6H), 7.05 (t, *J* = 7.5 Hz, 1H), 4.74 (s, 2H), 3.11 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.8, 153.0, 136.3, 130.8, 128.7, 127.6, 127.5, 125.9, 121.0, 120.6, 118.8, 56.4, 37.7.

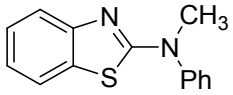


N,N-dibenzylbenzo[d]thiazol-2-amine(3i):[6] The product was purified by flash chromatography to give 84 mg (85%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.59 (t, *J* = 8.0 Hz, 2H), 7.34-7.27 (m, 11H), 7.08 (t, *J* = 7.5 Hz, 1H), 4.74 (s, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 169.0, 153.0, 136.2, 131.0, 128.7, 127.7, 127.6, 126.0, 121.2, 120.6, 119.0, 53.2.

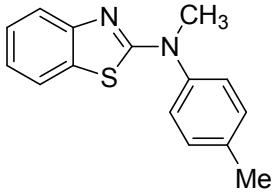


2-(3,4-dihydroisoquinolin-2(1H)-yl)benzo[d]thiazole(3j):[7] The product was purified by flash chromatography to give 78 mg (98%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.60 (d, *J* = 8.0 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.24-7.16 (m, 4H), 7.07 (t, *J* = 7.5 Hz, 1H), 4.80 (s, 2H), 3.85 (t, *J* = 6.0 Hz, 2H), 3.00 (t, *J* = 6.0 Hz,

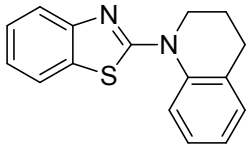
2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.0, 152.7, 134.2, 132.4, 130.4, 128.5, 126.8, 126.5, 126.3, 125.9, 121.1, 120.6, 118.9, 49.5, 46.1, 28.7.



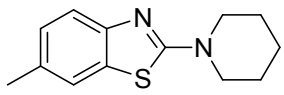
N-methyl-N-phenylbenzo[d]thiazol-2-amine(3k):[8] The product was purified by flash chromatography to give 40 mg (56%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.62 (d, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.45-7.40 (m, 4H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H) 7.06 (t, *J* = 7.5 Hz, 1H), 3.63 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.2, 152.5, 145.7, 131.1, 129.9, 127.4, 125.9, 125.8, 121.7, 120.4, 119.1, 40.4.



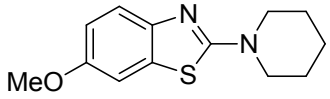
N-methyl-N-(p-tolyl)benzo[d]thiazol-2-amine(3l):[9] The product was purified by flash chromatography to give 52 mg (68%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.61 (d, *J* = 8.0 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.29-7.23 (m, 5H), 7.04 (t, *J* = 7.0 Hz, 1H), 3.60 (s, 3H), 2.39 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.5, 152.6, 143.2, 137.5, 131.1, 130.5, 125.9, 125.8, 121.5, 120.4, 119.0, 40.4, 21.1.



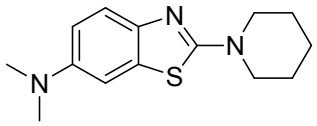
2-(3,4-dihydroquinolin-1(2H)-yl)benzo[d]thiazole(3m):[7] The product was purified by flash chromatography to give 54 mg (68%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.87 (d, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.22 (t, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 7.0 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 7.07 (t, *J* = 7.5 Hz, 1H), 4.07 (t, *J* = 6.0 Hz, 2H), 2.78 (t, *J* = 6.0 Hz, 2H), 2.06-2.01 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 166.6, 151.6, 140.3, 130.8, 130.2, 129.0, 126.6, 125.9, 124.2, 122.3, 121.1, 120.5, 119.5, 49.3, 27.3, 23.4.



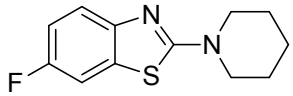
6-methyl-2-(piperidin-1-yl)benzo[d]thiazole(3n):[3] The product was purified by flash chromatography to give 56 mg (80%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.42 (d, *J* = 8.5 Hz, 1H), 7.37 (s, 1H), 7.07 (d, *J* = 8.5 Hz, 1H), 3.57 (s, 4H), 2.37 (s, 3H), 1.67 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.3, 150.7, 130.6, 130.6, 126.9, 120.6, 118.3, 49.5, 25.2, 24.2, 21.1.



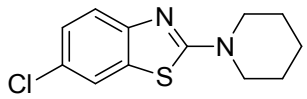
6-methoxy-2-(piperidin-1-yl)benzo[d]thiazole(3o):[3] The product was purified by flash chromatography to give 66 mg (89%) as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.44 (d, *J* = 8.5 Hz, 1H), 7.13 (d, *J* = 2.5 Hz, 1H), 7.07 (dd, *J* = 9.0 Hz, 2.5 Hz, 1H), 3.80 (s, 3H), 3.55 (s, 4H), 1.67 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 167.6, 154.7, 147.1, 131.6, 119.2, 113.3, 105.2, 55.8, 49.6, 25.2, 24.2.



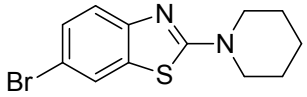
N,N-dimethyl-2-(piperidin-1-yl)benzo[d]thiazol-6-amine(3p): The product was purified by flash chromatography to give 68 mg (87%) as a green solid. mp: 113.6-114.2 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.42 (d, *J* = 8.5 Hz, 1H), 6.98 (s, 1H), 6.79 (d, *J* = 8.5 Hz, 1H), 3.52 (s, 4H), 2.90 (s, 6H), 1.65 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 166.7, 146.5, 144.8, 132.1, 118.9, 113.0, 104.8, 49.5, 41.6, 25.2, 24.2. HRMS (ESI) m/z calcd for $C_{14}H_{20}N_3S^+$ $(M+H)^+$ 262.13724, found 262.13724.



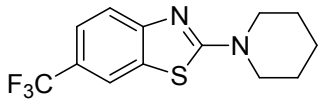
6-fluoro-2-(piperidin-1-yl)benzo[d]thiazole(3q):[10] The product was purified by flash chromatography to give 57 mg (81%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.44 (dd, *J* = 9.0 Hz, 4.5 Hz, 1H), 7.28 (dd, *J* = 8.0 Hz, 2.5Hz, 1H), 6.98 (td, *J* = 9.0 Hz, 2.5 Hz, 1H), 3.56 (s, 4H), 1.68 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 168.4 (d, *J* = 1.6 Hz), 157.9 (d, *J* = 238.3 Hz), 149.3, 131.3 (d, *J* = 10.5 Hz), 119.0 (d, *J* = 8.5 Hz), 113.4 (d, *J* = 23.5 Hz), 107.2 (d, *J* = 27.0 Hz), 49.5, 25.2, 24.1.



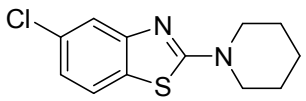
6-chloro-2-(piperidin-1-yl)benzo[d]thiazole(3r):[10] The product was purified by flash chromatography to give 63 mg (83%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.52 (s, 1H), 7.40 (d, *J* = 8.5 Hz, 1H), 7.21 (dd, *J* = 9.0 Hz, 2.0 Hz, 1H), 3.57 (s, 4H), 1.68 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.8, 151.5, 131.8, 126.2, 125.9, 120.2, 119.3, 49.6, 25.2, 24.1. HRMS (ESI) m/z calcd for $C_{12}H_{14}ClN_2S^+$ $(M+H)^+$ 253.05607, found 253.05609.



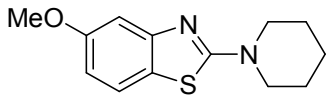
6-bromo-2-(piperidin-1-yl)benzo[d]thiazole(3s):[11] The product was purified by flash chromatography to give 76 mg (85%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.67 (s, 1H), 7.36 (s, 2H), 3.58 (s, 4H), 1.69 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.8, 151.9, 132.3, 129.0, 123.0, 119.8, 113.2, 49.6, 25.3, 24.1.



2-(piperidin-1-yl)-6-(trifluoromethyl)benzo[d]thiazole(3t):[10] The product was purified by flash chromatography to give 40 mg (47%) as a yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.82 (s, 1H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.51 (d, *J* = 8.5 Hz, 1H), 3.63 (s, 4H), 1.71 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ = 170.2, 155.5, 130.7, 125.7, 123.1 (q, *J* = 3.6 Hz), 122.6 (q, *J* = 32.3 Hz), 118.3, 117.9 (q, *J* = 3.9 Hz), 49.7, 25.3, 24.1.



5-chloro-2-(piperidin-1-yl)benzo[d]thiazole(3u): The product was purified by flash chromatography to give 45 mg (60%) as a yellow solid. mp: 88.2-89.3 ^{o}C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.49 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 6.99 (dd, *J* = 8.5 Hz, 2.0 Hz, 1H), 3.58 (s, 4H), 1.68 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 169.7, 154.0, 131.6, 128.8, 121.1, 121.0, 118.6, 49.6, 25.2, 24.1. HRMS (ESI) m/z calcd for $C_{12}H_{14}ClN_2S^+$ $(M+H)^+$ 253.05607, found 253.05609.



5-methoxy-2-(piperidin-1-yl)benzo[d]thiazole(3v): The product was purified by flash chromatography to give 58 mg (78%) as a yellow solid. mp: 102.6-103.6 ^{o}C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 7.40 (d, *J* = 8.5 Hz, 1H), 7.10 (s, 1H), 6.67 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 3.81 (s, 3H), 3.57 (s, 4H), 1.67 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 170.1, 158.9, 154.1, 122.0, 120.7, 109.6, 103.0, 55.4, 49.5, 25.2, 24.2. HRMS (ESI) m/z calcd for $C_{13}H_{17}N_2OS^+$ $(M+H)^+$ 249.10561, found 249.10562.

5) References

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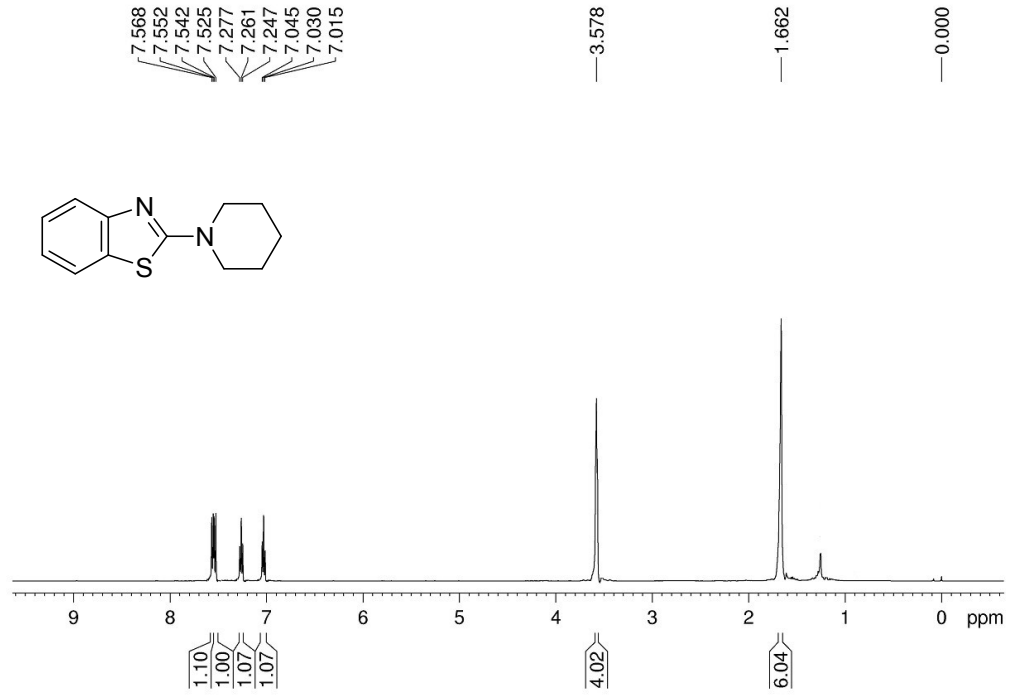
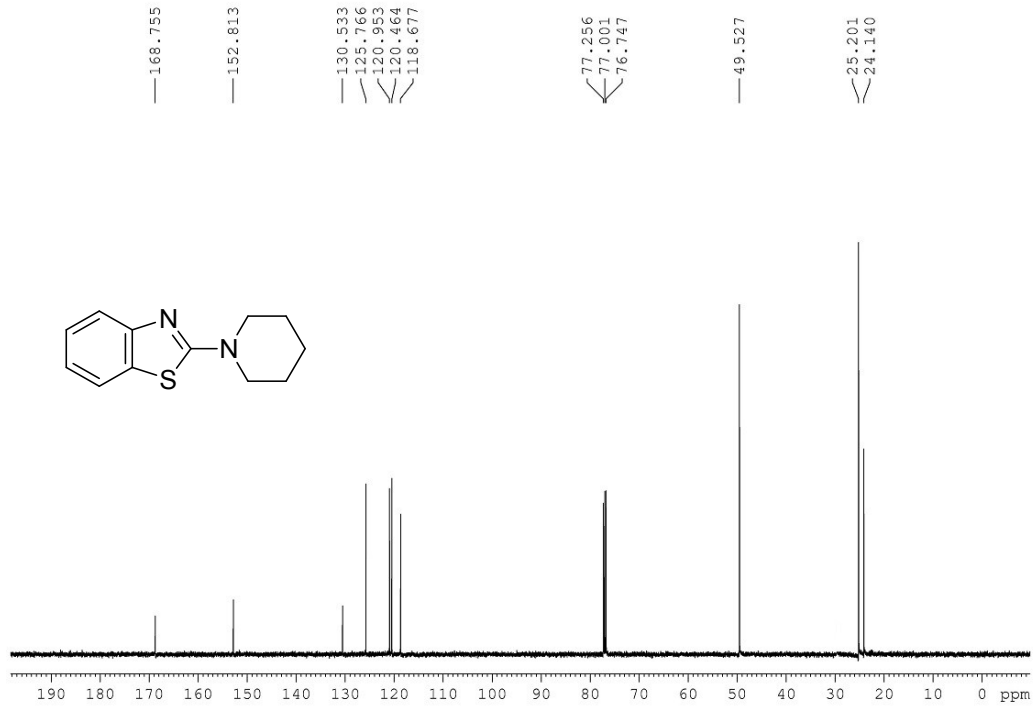
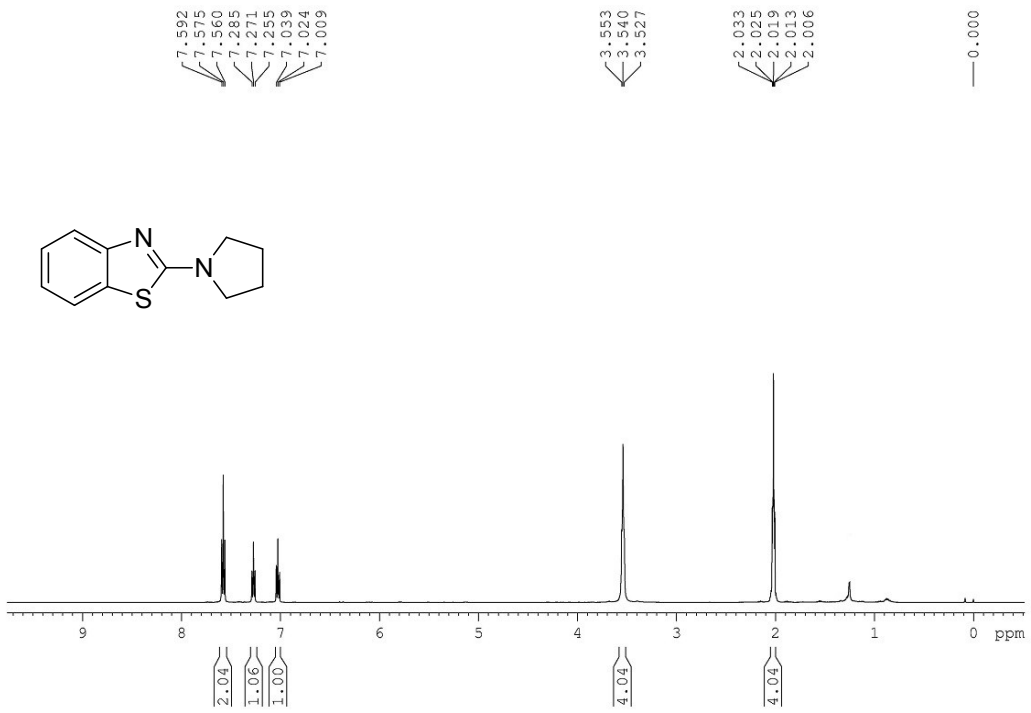
6) Scanned ^{1}H NMR and ^{13}C NMR Spectra of All New Compounds



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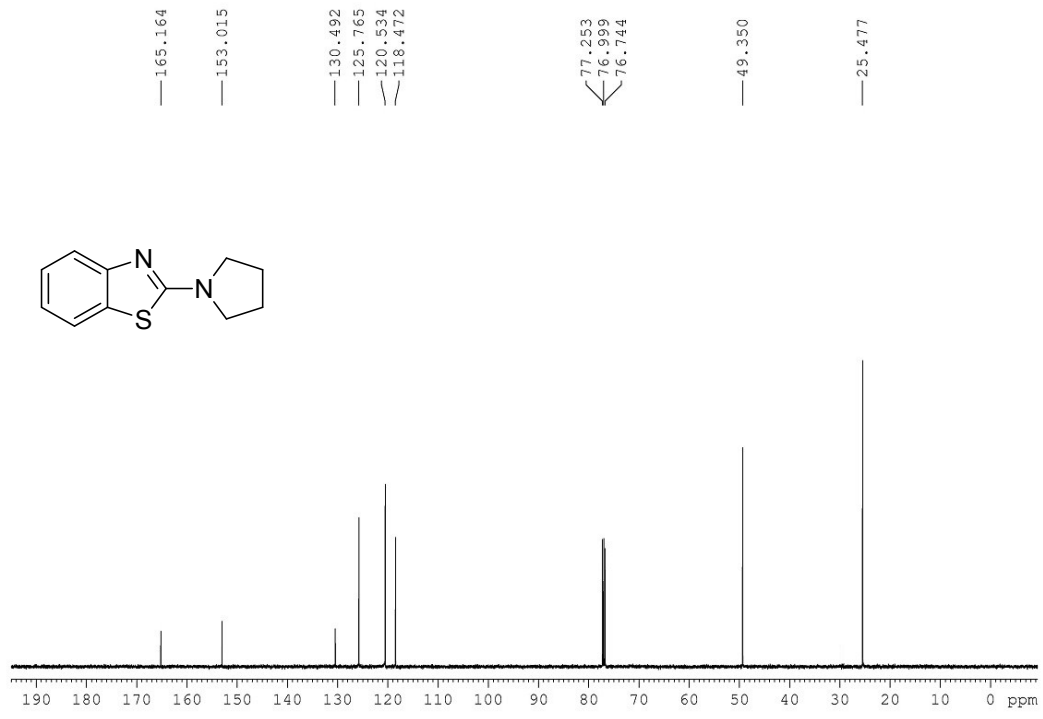
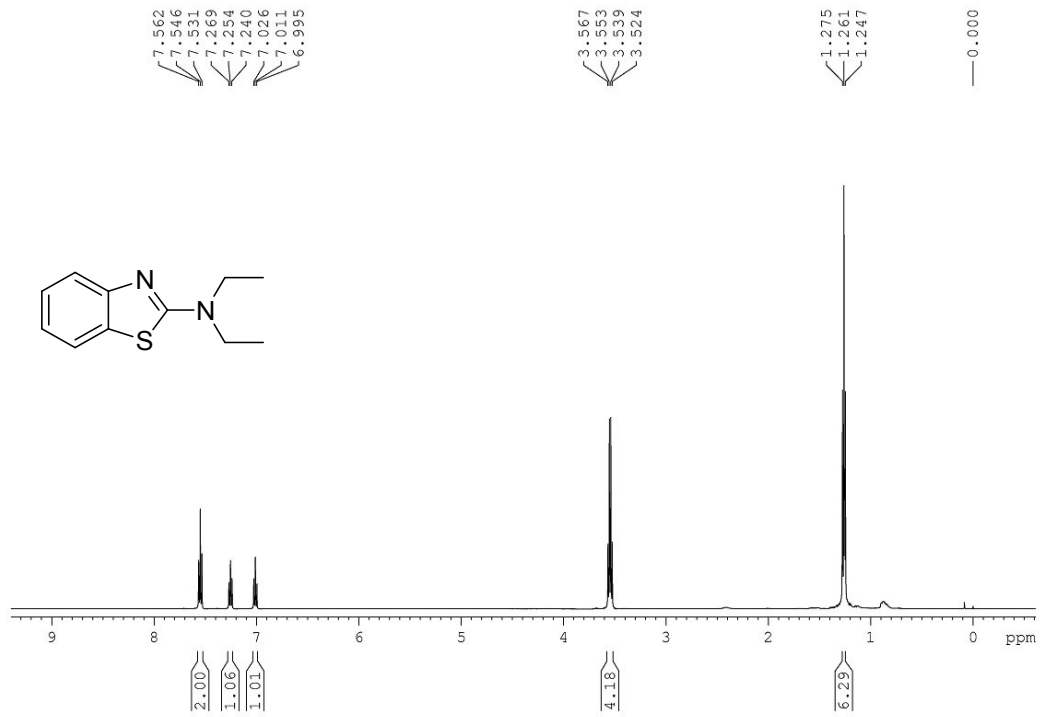
7.575
7.560
7.285
7.271
7.255
7.039
7.024
7.009
3.553
3.540
3.527
2.033
2.025
2.019
2.013
2.006
0.000
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
2.04
1.06
1.00
4.04
4.04


165.164
153.015
130.492
125.765
120.534
118.472
77.253
76.999
76.744
49.350
25.477
N
S
N
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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7.546
7.531
7.269
7.254
7.240
7.026
7.011
6.995
3.567
3.553
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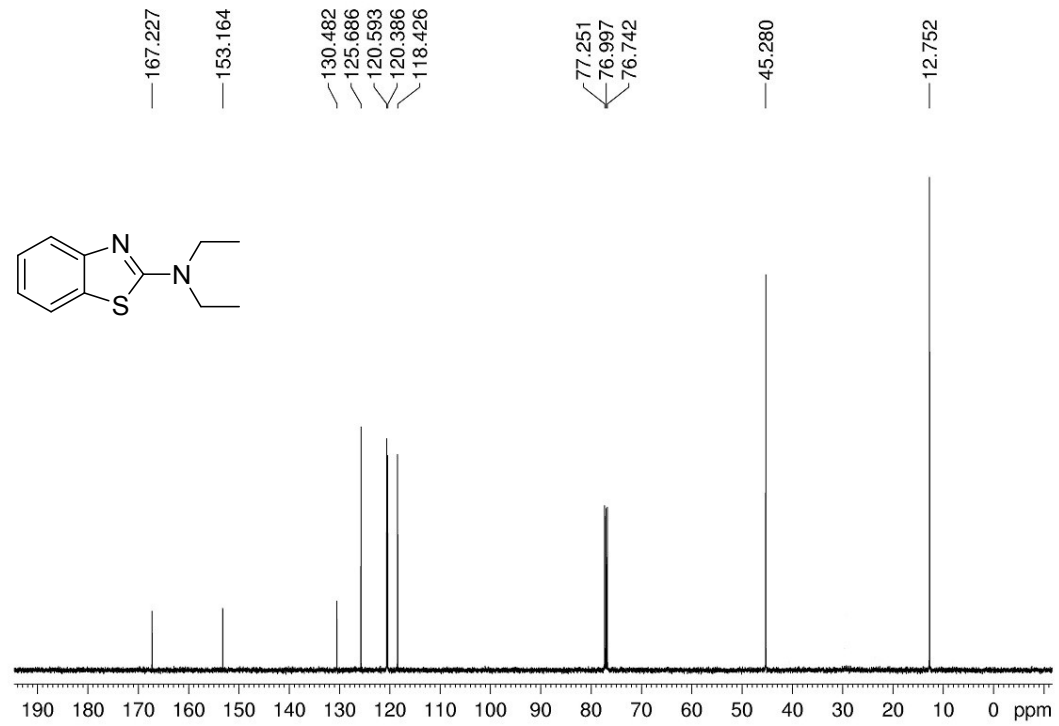
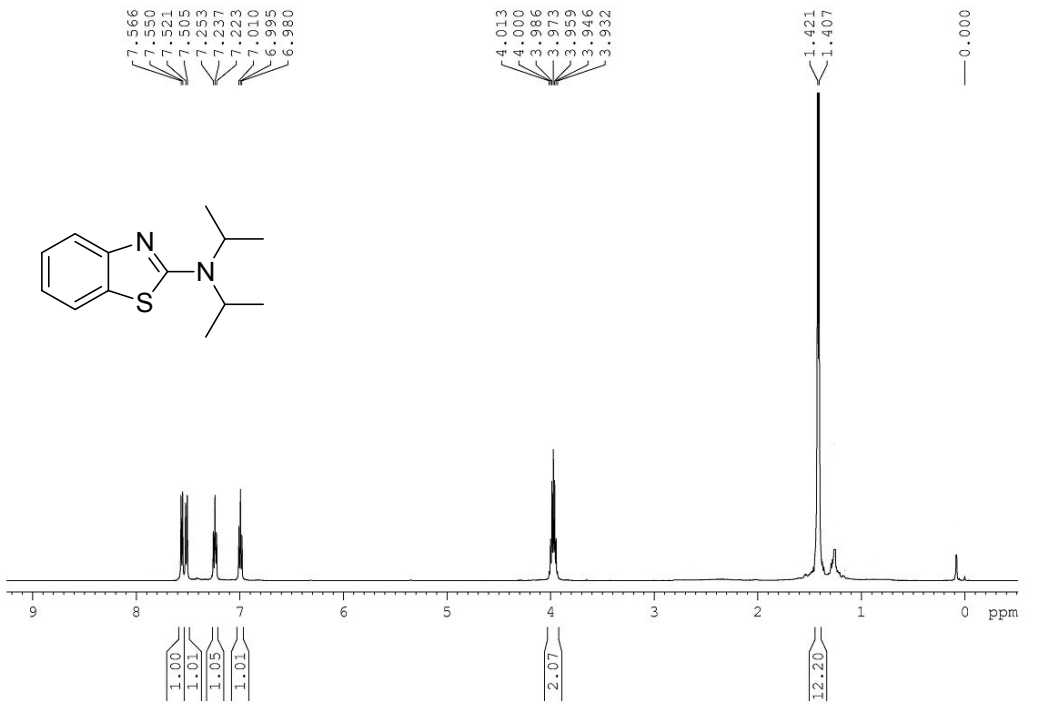
3.524
1.275
1.261
1.247
0.000
N
S
N
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.06
1.01
4.18
6.29


167.227
153.164
130.482
125.686
120.593
120.386
118.426
77.251
76.997
76.742
45.280
12.752
N
S
N
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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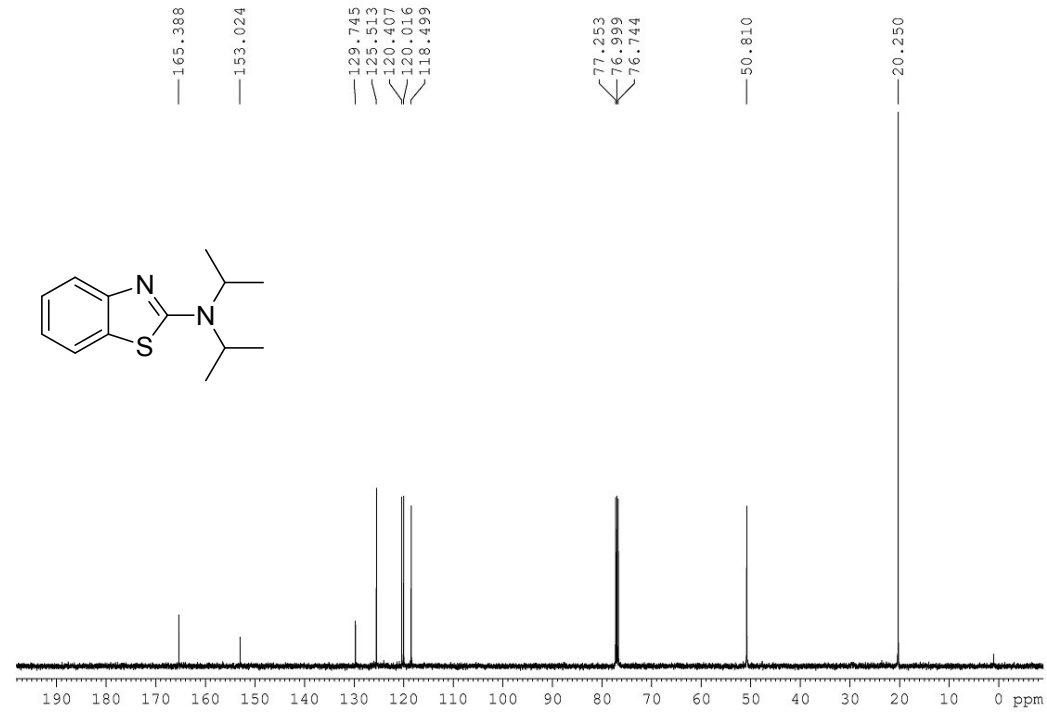
7.550
7.521
7.505
7.253
7.237
7.223
7.010
6.995
6.980
4.013
4.000
3.986
3.973
3.959
3.946
3.932
1.421
1.407
0.000
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.01
1.05
1.01
2.07
12.20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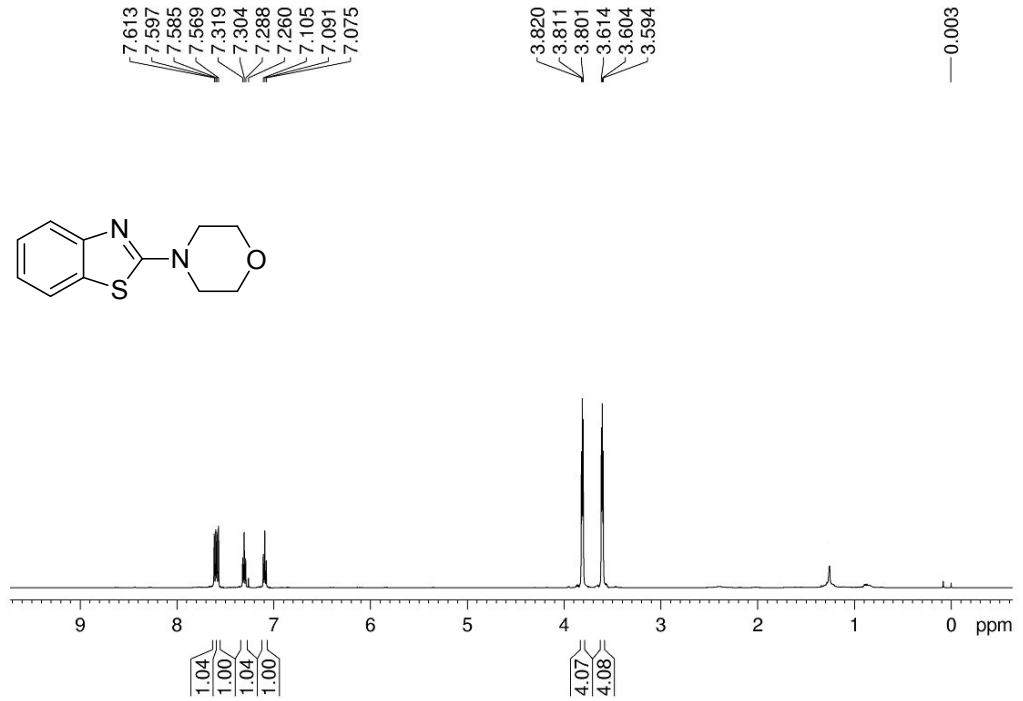
120.016
118.499
77.253
76.999
76.744
50.810
20.250
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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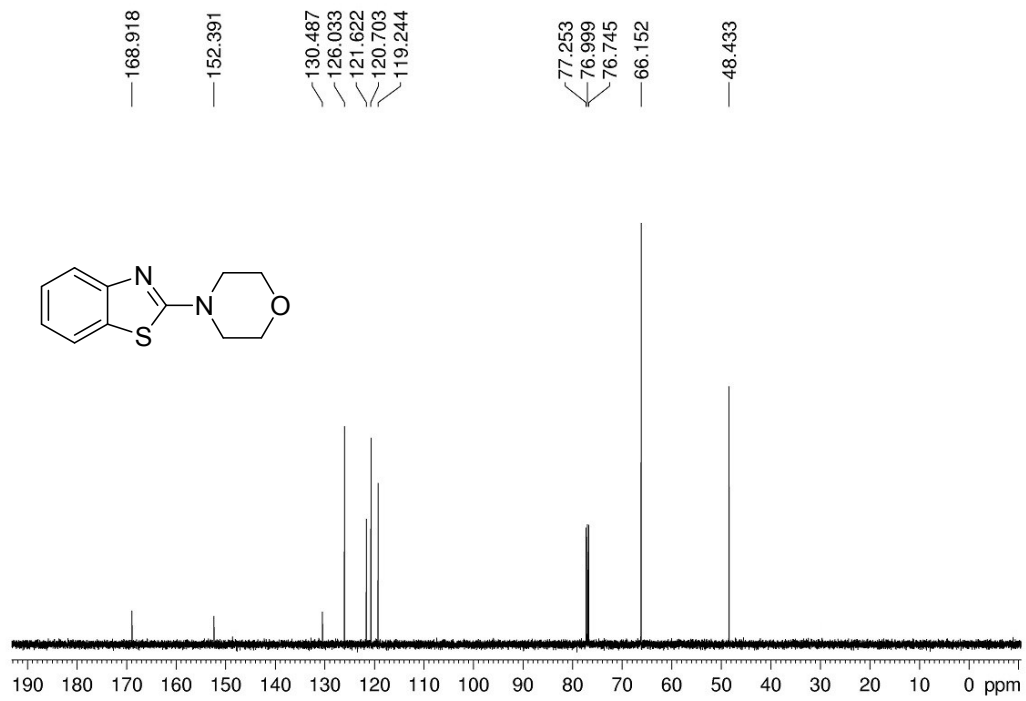
7.597
7.585
7.569
7.319
7.304
7.288
7.260
7.105
7.091
7.075
3.820
3.811
3.801
3.614
3.604
3.594
0.003
N
S
N
O
9
8
7
6
5
4
3
2
1
0 ppm
1.04
1.00
1.04
1.00
4.07
4.08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119.244
77.253
76.999
76.745
66.152
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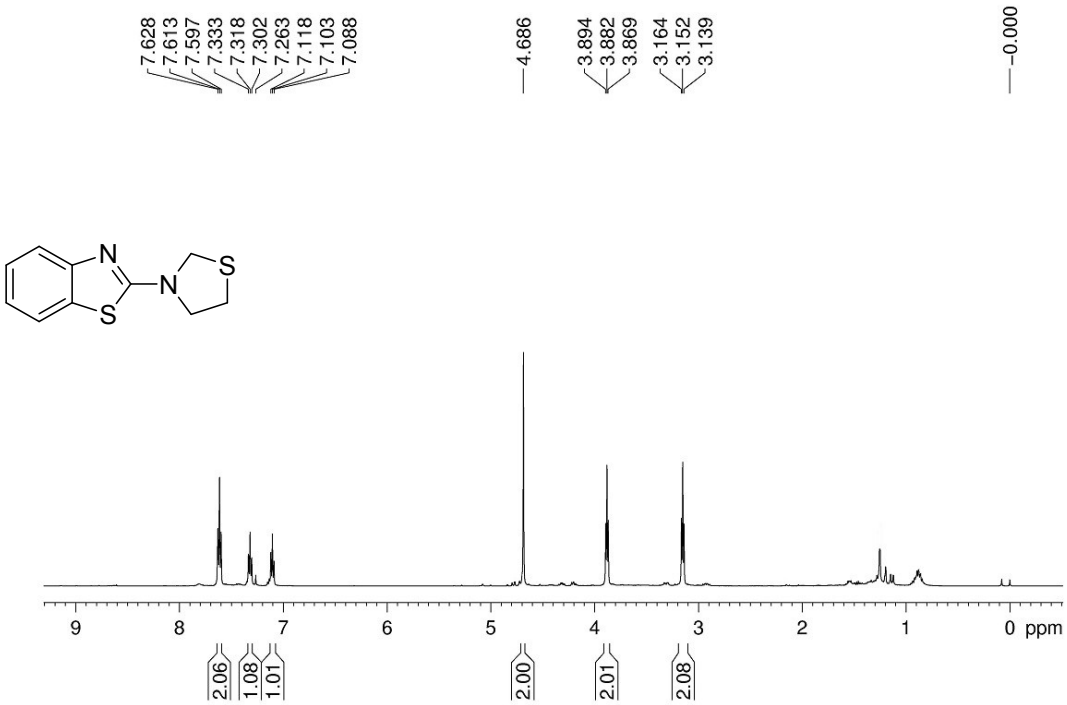
N
S
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.318
7.302
7.263
7.118
7.103
7.088
4.686
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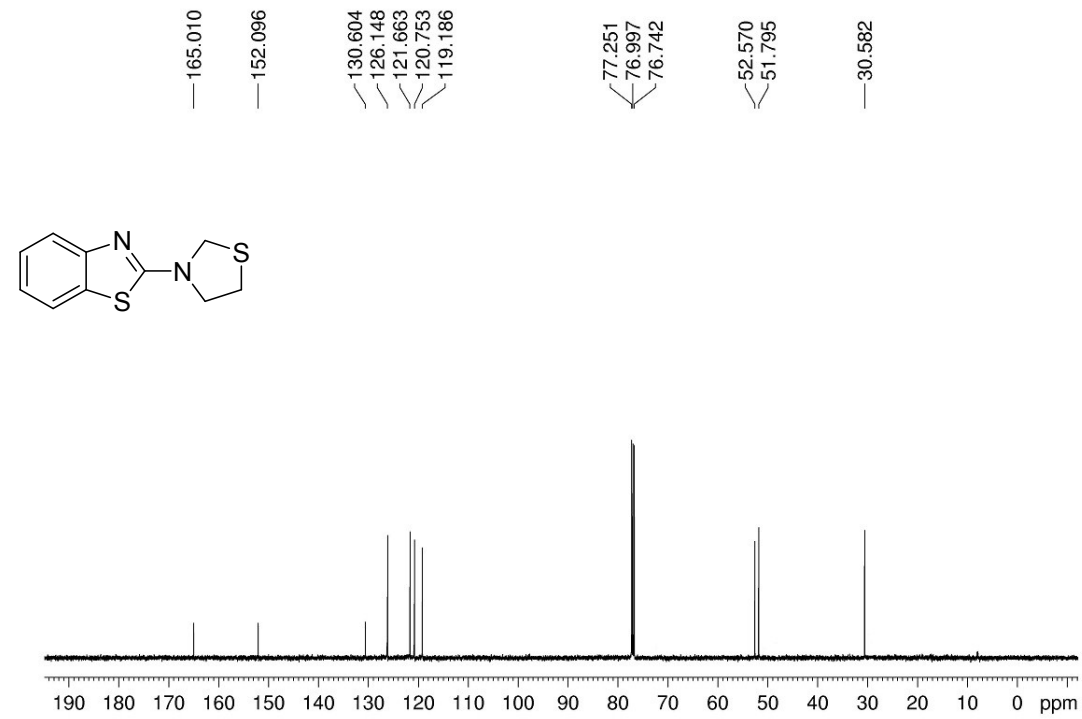
3.882
3.869
3.164
3.152
3.139
0.000
N
S
N
S
9
8
7
6
5
4
3
2
1
0 ppm
2.06
1.08
1.01
2.00
2.01
2.08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120.753
119.186
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76.742
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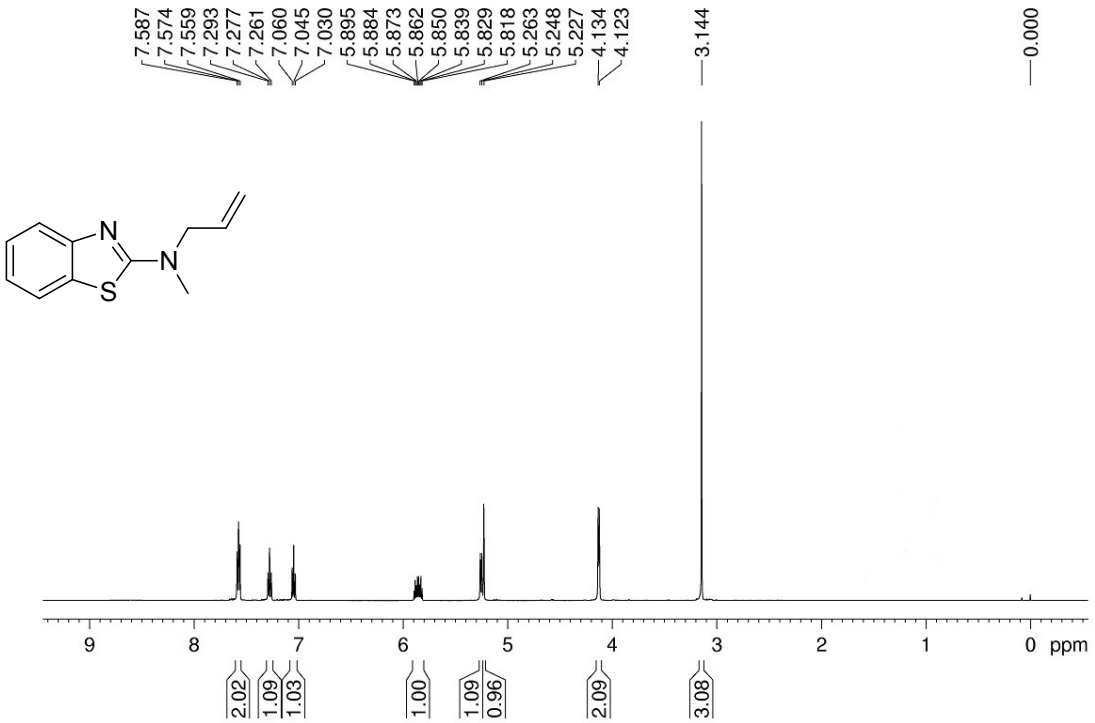
51.795
30.582
N
S
N
S
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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5.862
5.850
5.839
5.829
5.818
5.263
5.248
5.227
4.134
4.123
3.144
0.000
N
S
N
9
8
7
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4
3
2
1
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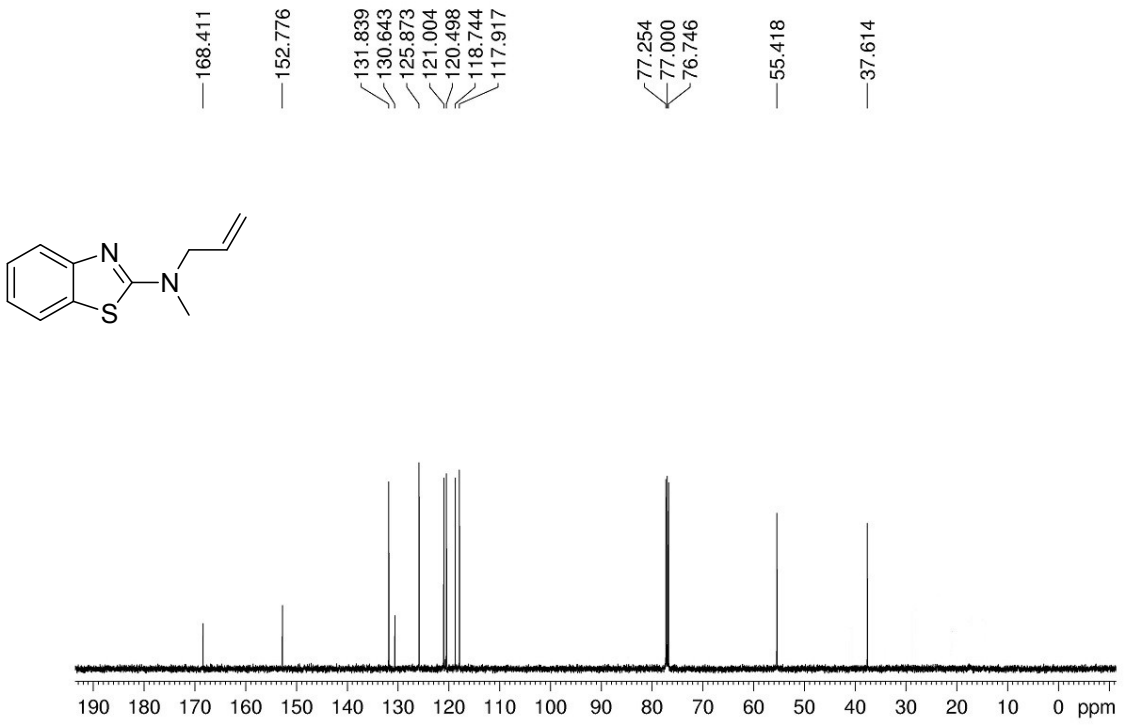
3.08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76.746
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37.614
N
S
N
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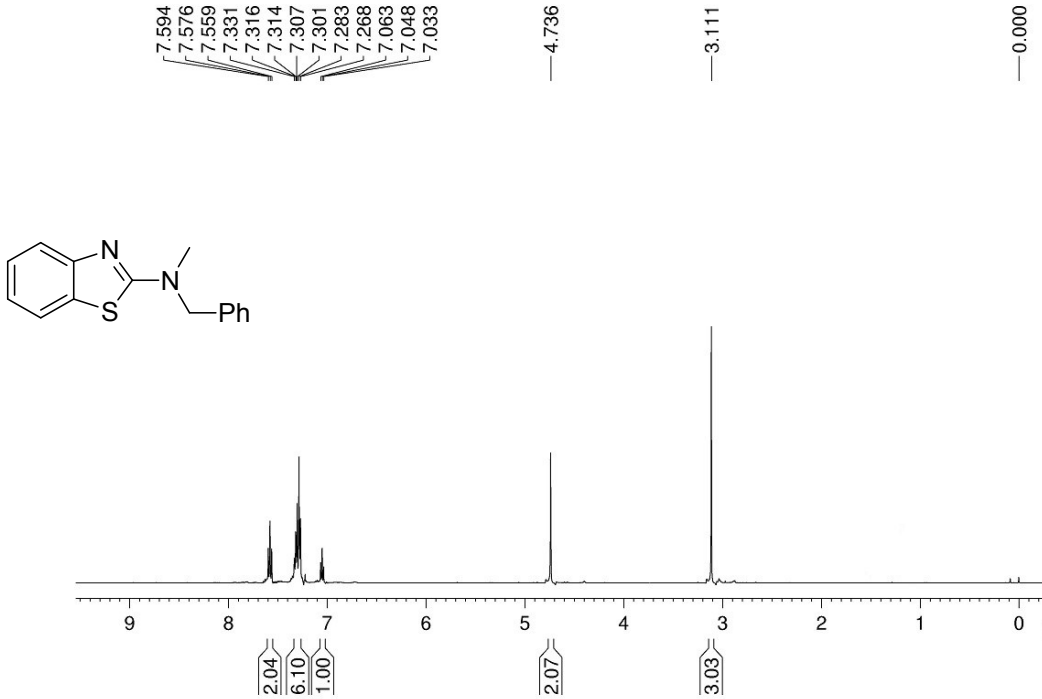
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7.033
4.736
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0.000
N
S
N
Ph
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6
5
4
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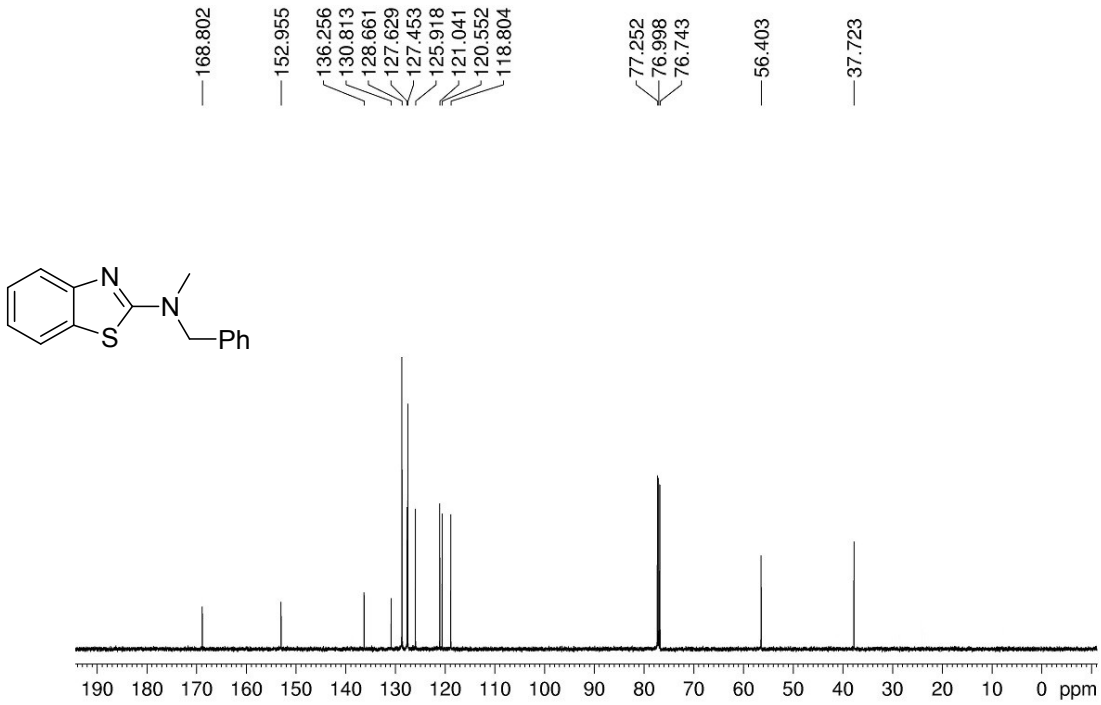
6.10
1.00
2.07
3.03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76.743
56.403
37.723
N
S
N
Ph
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140
130
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110
100
90
80
70
60
50
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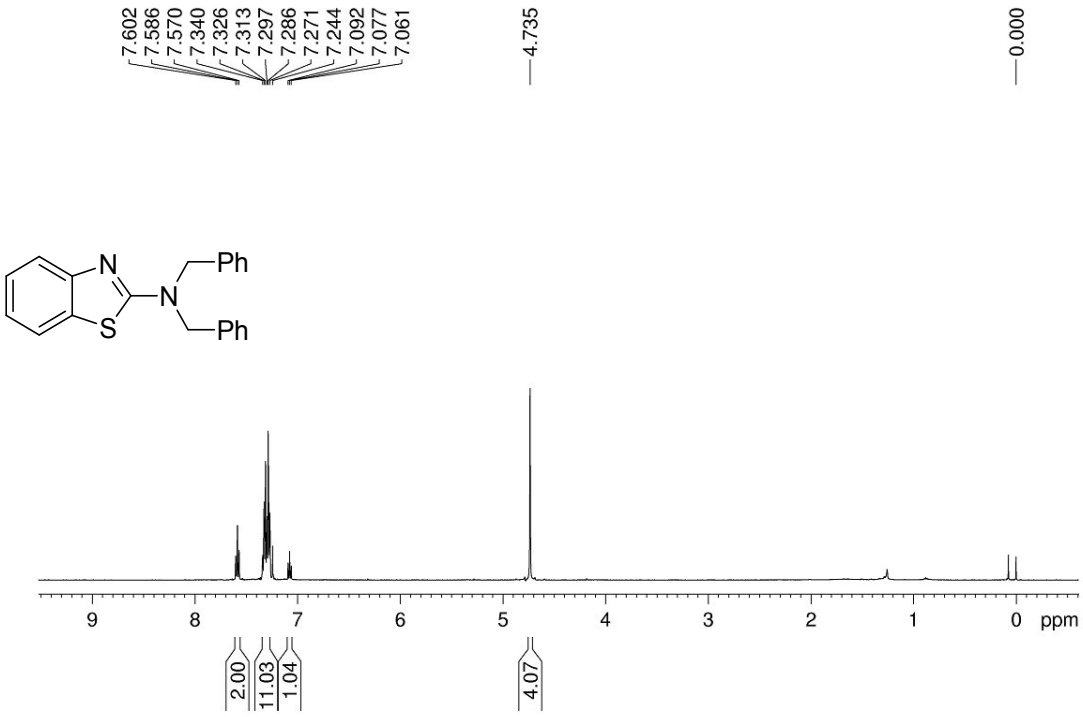
30
20
10
0
ppm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7.061
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0.000
N
Ph
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S
Ph
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8
7
6
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4
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ppm
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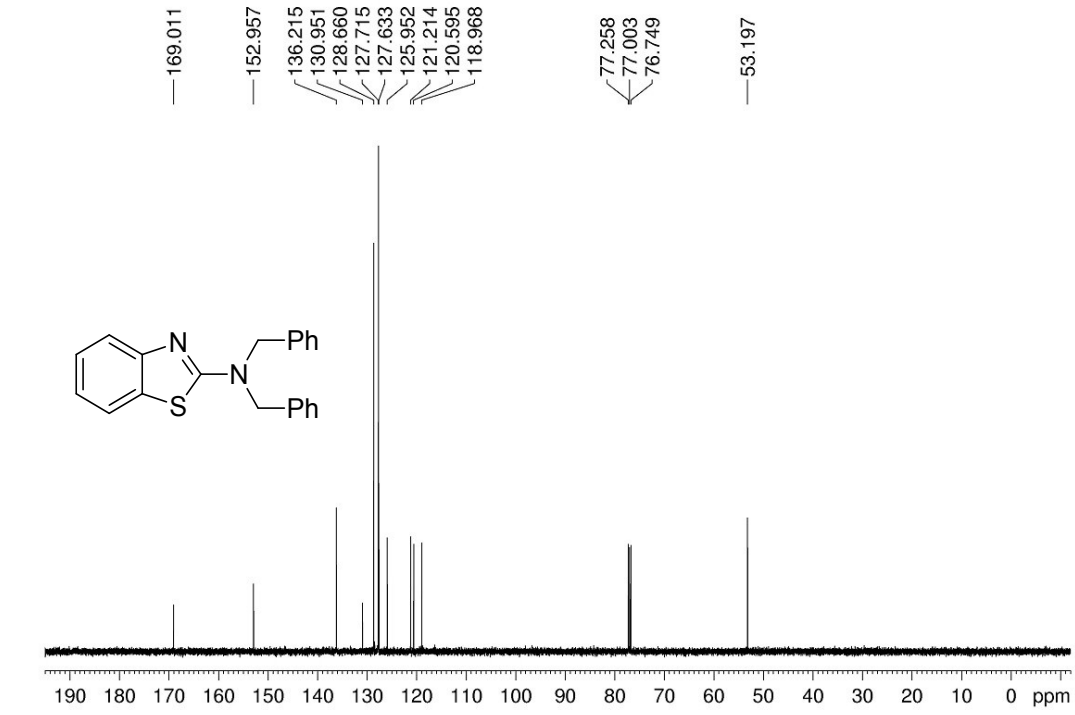
11.03
1.04
4.07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77.258
77.003
76.749
53.197
N
Ph
N
S
Ph
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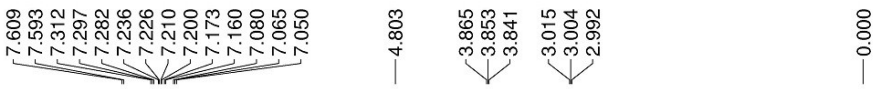
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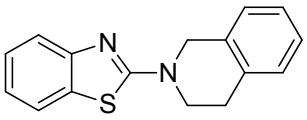
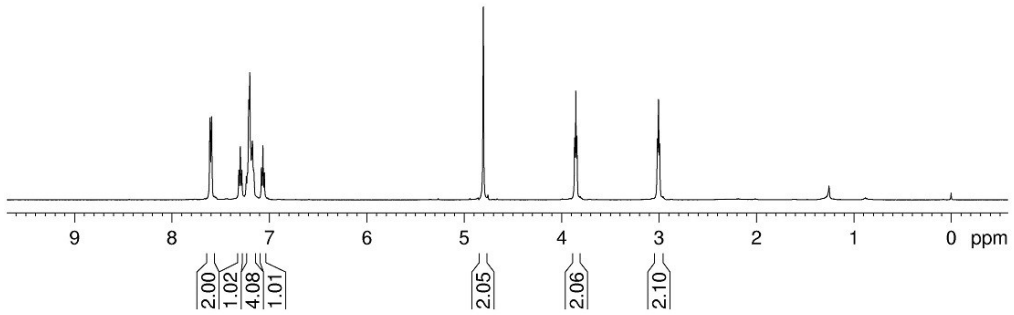

N
S
N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8
7
6
5
4
3
2
1
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1.02
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1.01
2.05
2.06
2.10


168.005
152.654
134.201
132.435
130.362
128.464
126.759
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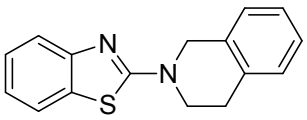
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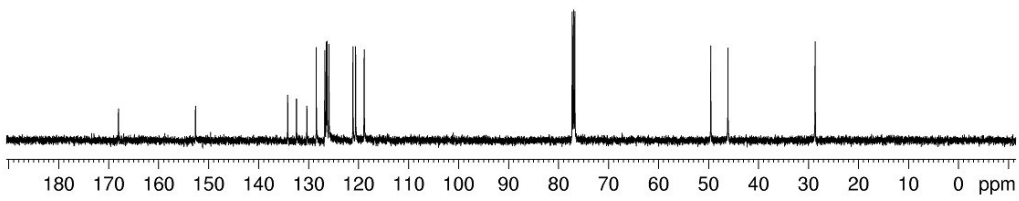
S
N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120
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60
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20
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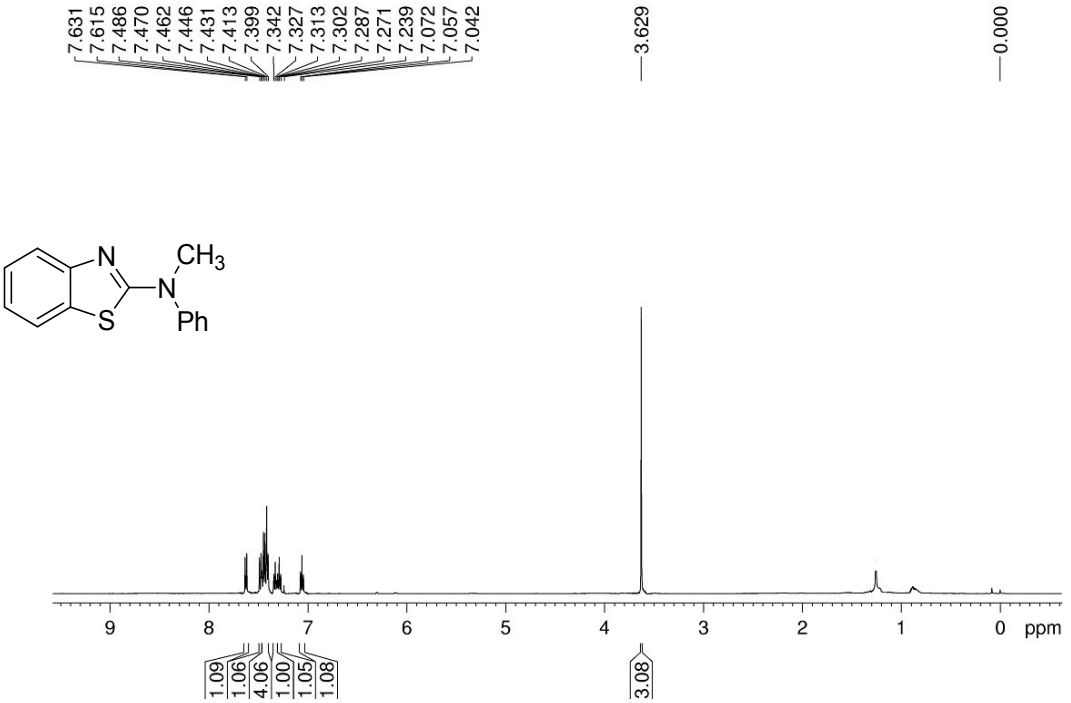
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7.342
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7.302
7.287
7.271
7.239
7.072
7.057
7.042
3.629
0.000
N
CH3
N
S
Ph
9
8
7
6
5
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3
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1
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1.06
4.06
1.00
1.05
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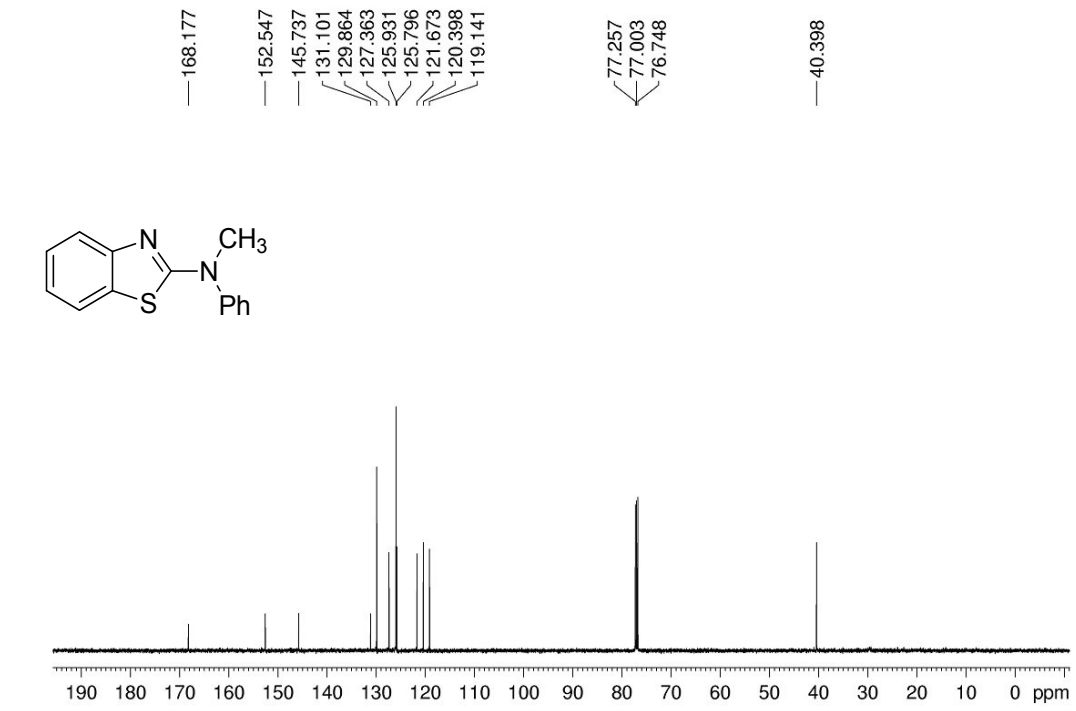
3.08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119.141
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Ph
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90
80
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60
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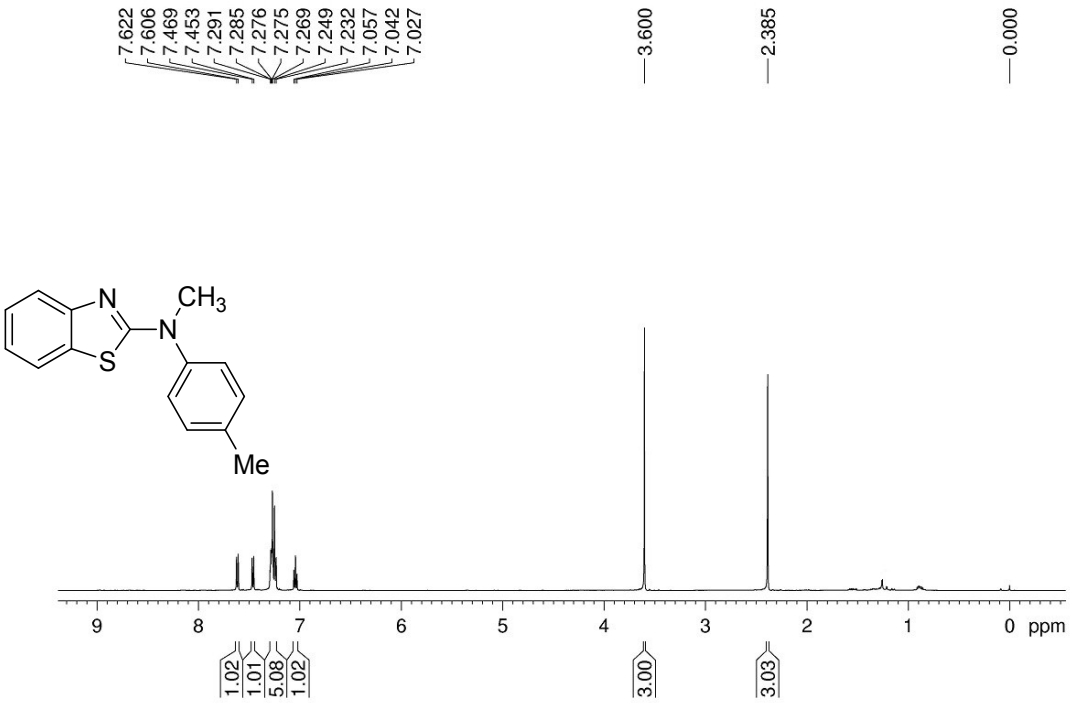
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7.276
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7.249
7.232
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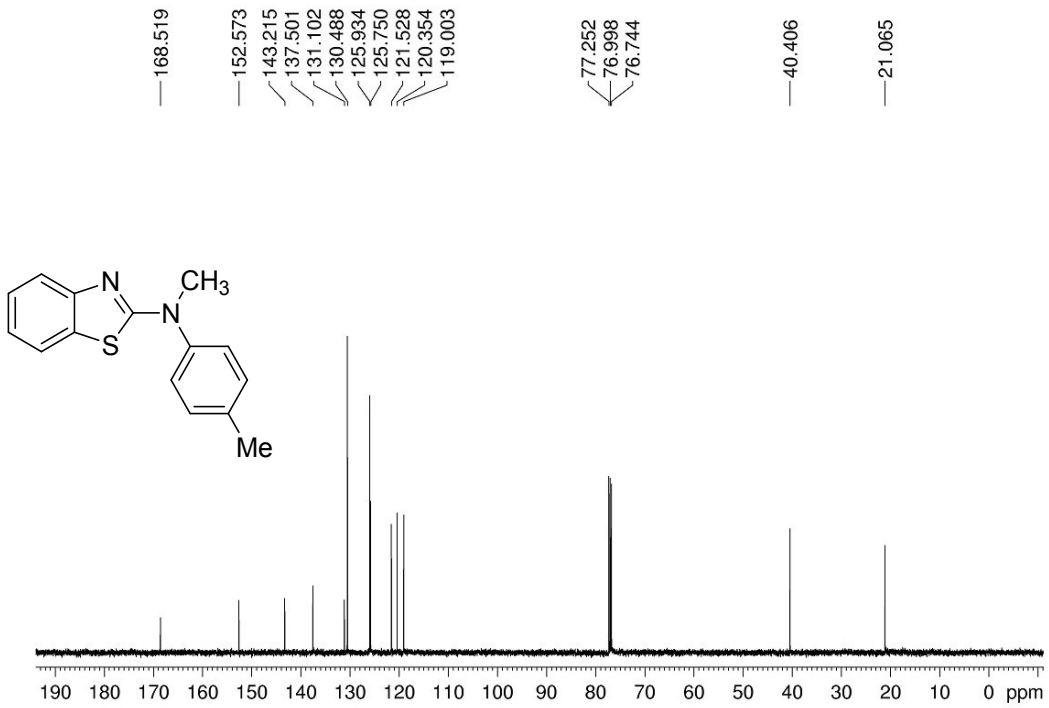
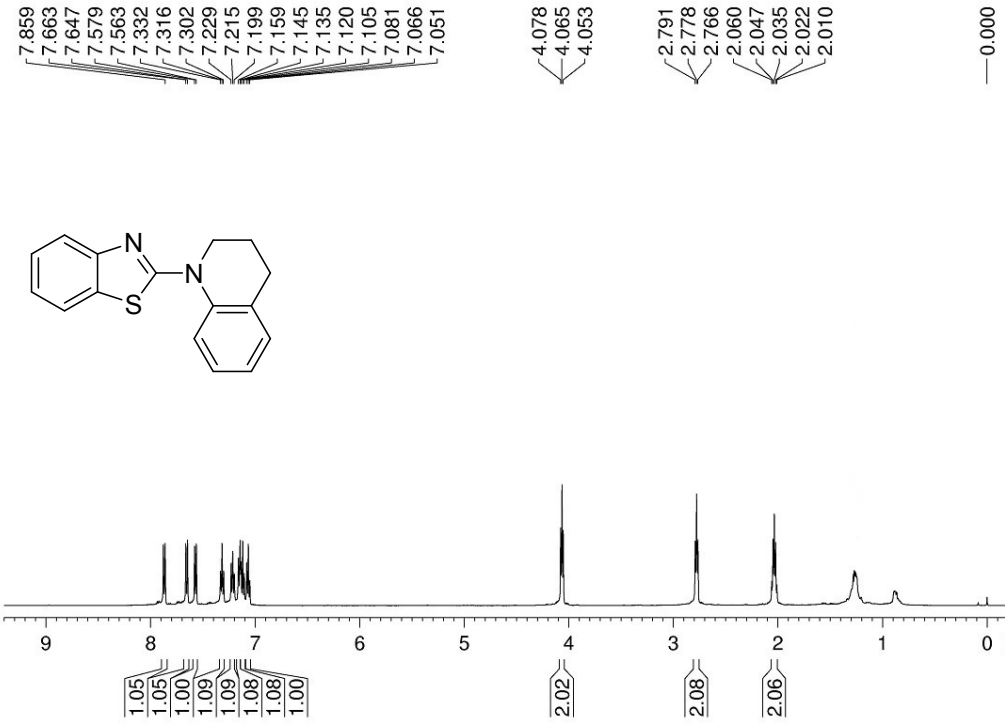
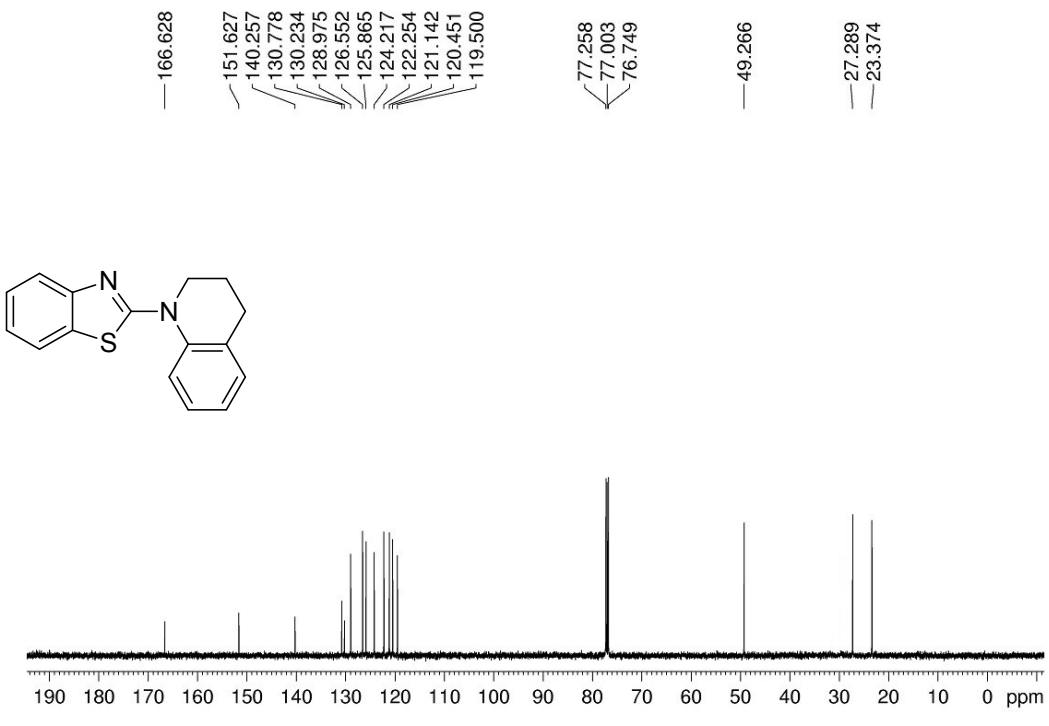
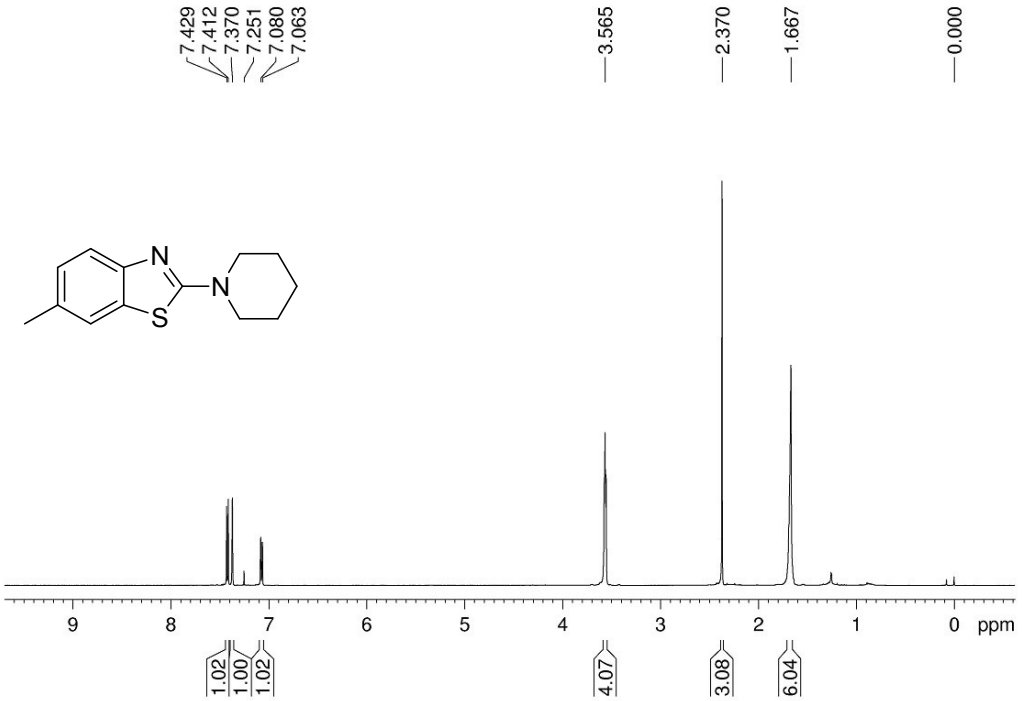
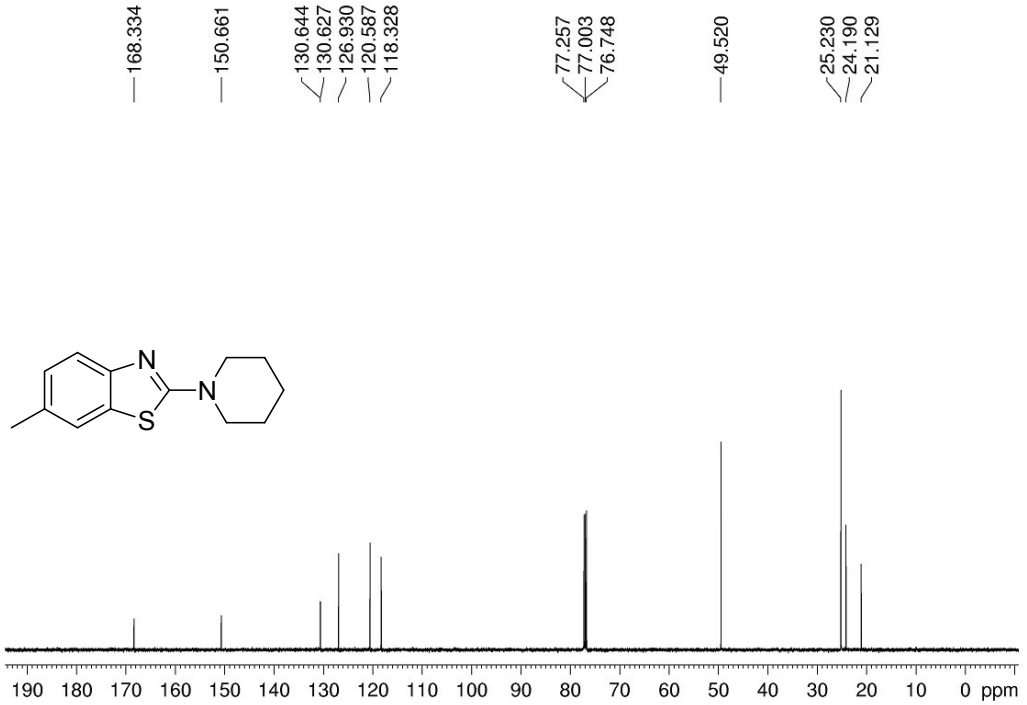
7.042
7.027
3.600
2.385
0.000
N
CH3
N
S
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.02
1.01
5.08
1.02
3.00
3.03

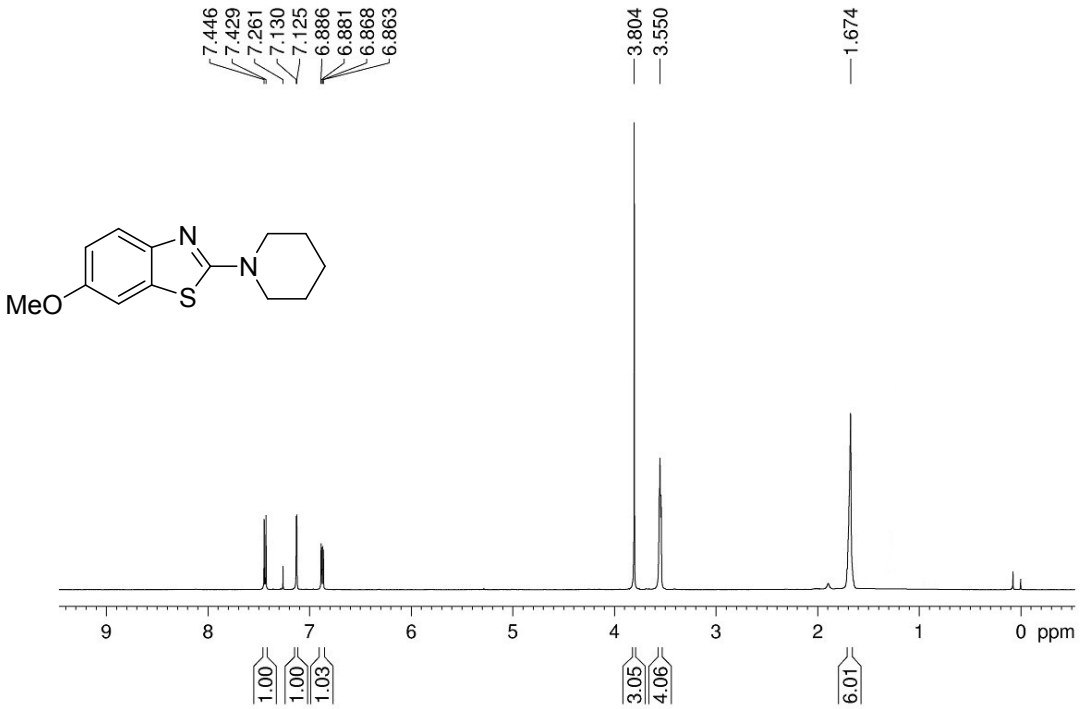

168.519
152.573
143.215
137.501
131.102
130.488
125.934
125.750
121.528
120.354
119.003
77.252
76.998
76.744
40.406
21.065
N
CH3
N
S
Me
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

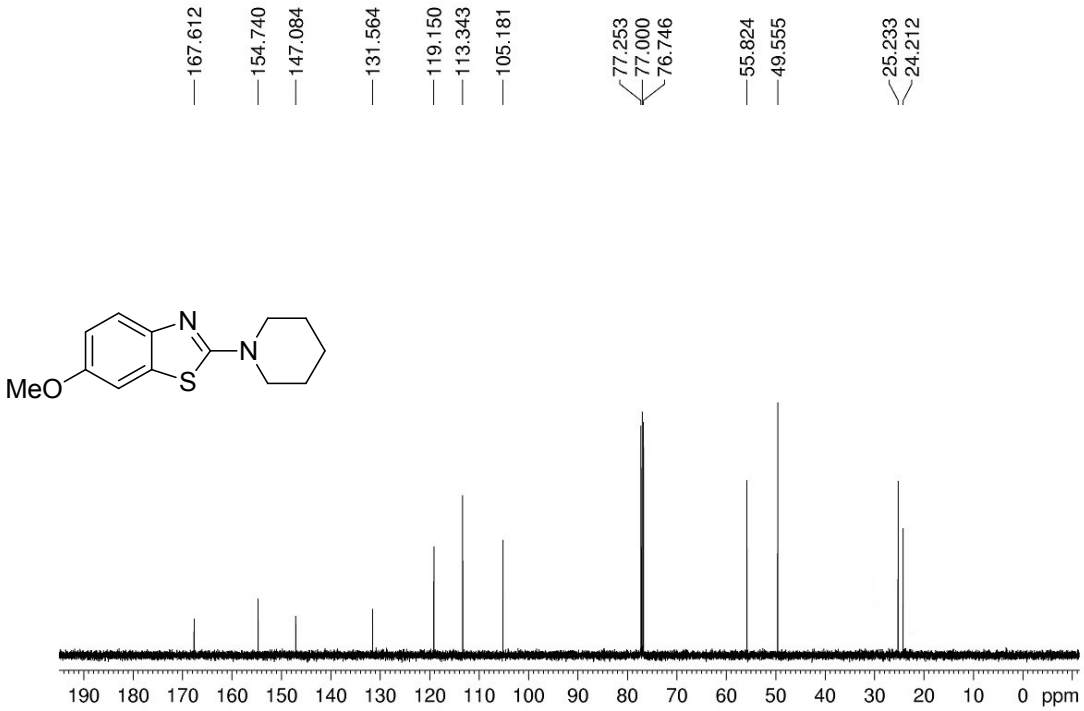

7.859
7.663
7.647
7.579
7.563
7.332
7.316
7.302
7.229
7.215
7.199
7.159
7.145
7.135
7.120
7.105
7.081
7.066
7.051
4.078
4.065
4.053
2.791
2.778
2.766
2.060
2.047
2.035
2.022
2.010
0.000
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.05
1.05
1.00
1.09
1.09
1.08
1.08
1.00
2.02
2.08
2.06

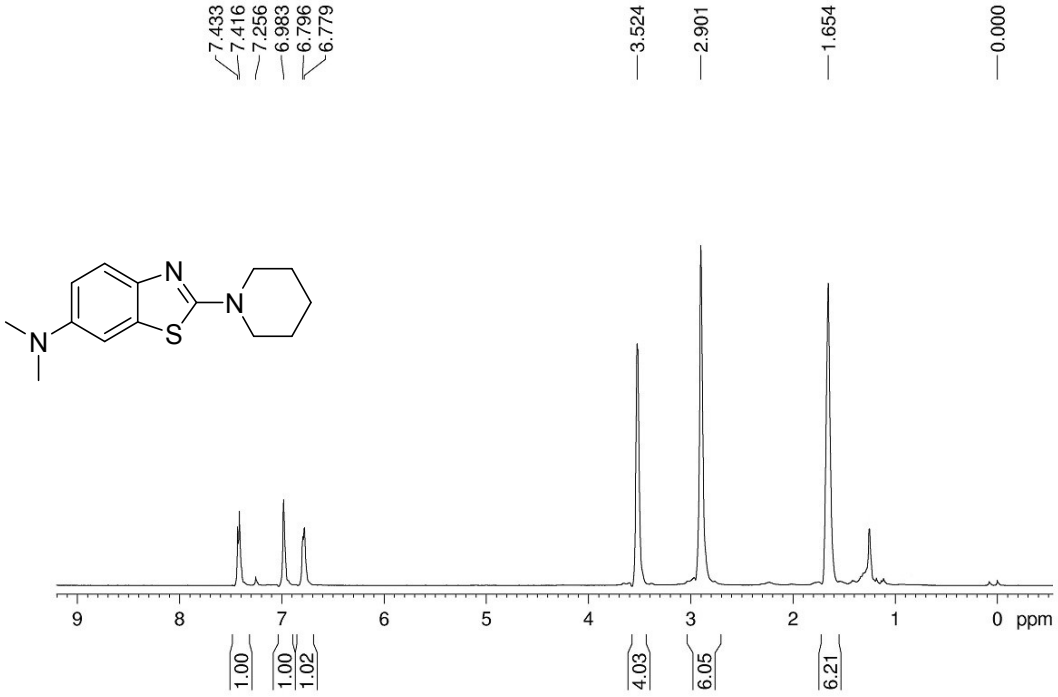

166.628
151.627
140.257
130.778
130.234
128.975
126.552
125.865
124.217
122.254
121.142
120.451
119.500
77.258
77.003
76.749
49.266
27.289
23.374
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

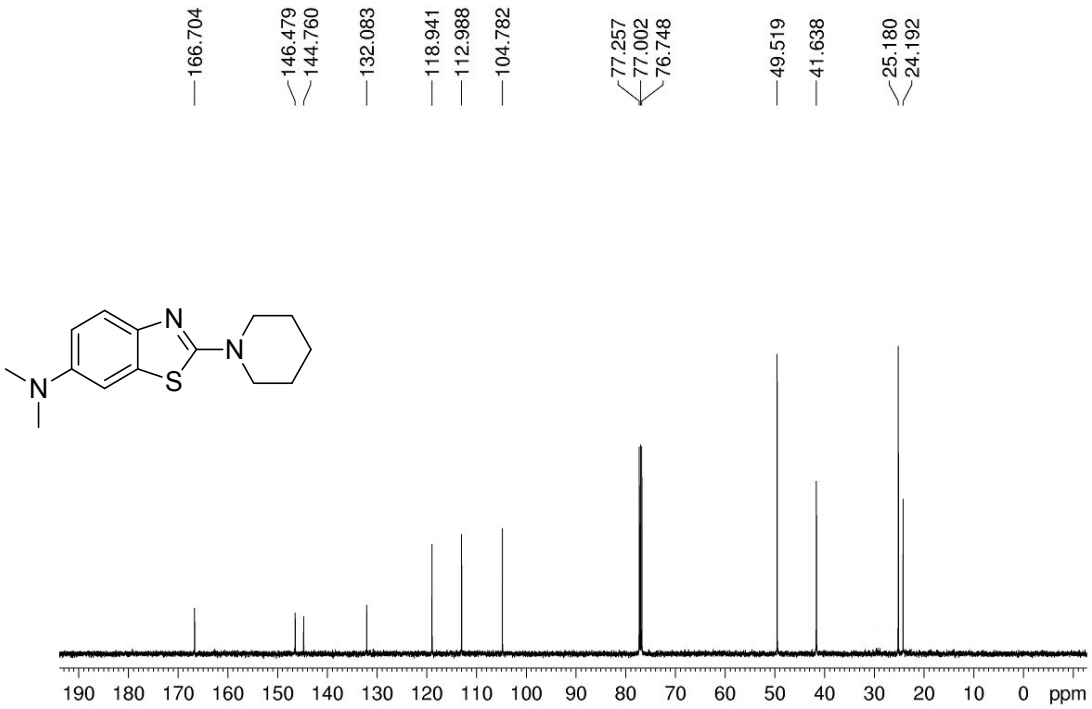

7.429
7.412
7.370
7.251
7.080
7.063
3.565
2.370
1.667
0.000
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.02
1.00
1.02
4.07
3.08
6.04

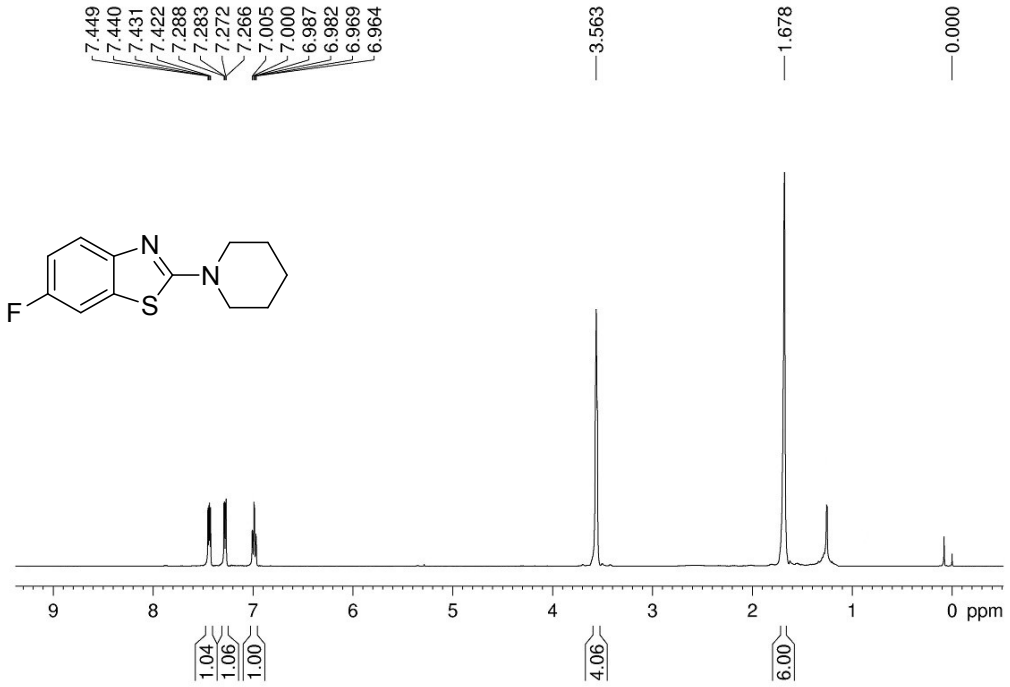

168.334
150.661
130.644
130.627
126.930
120.587
118.328
77.257
77.003
76.748
49.520
25.230
24.190
21.129
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

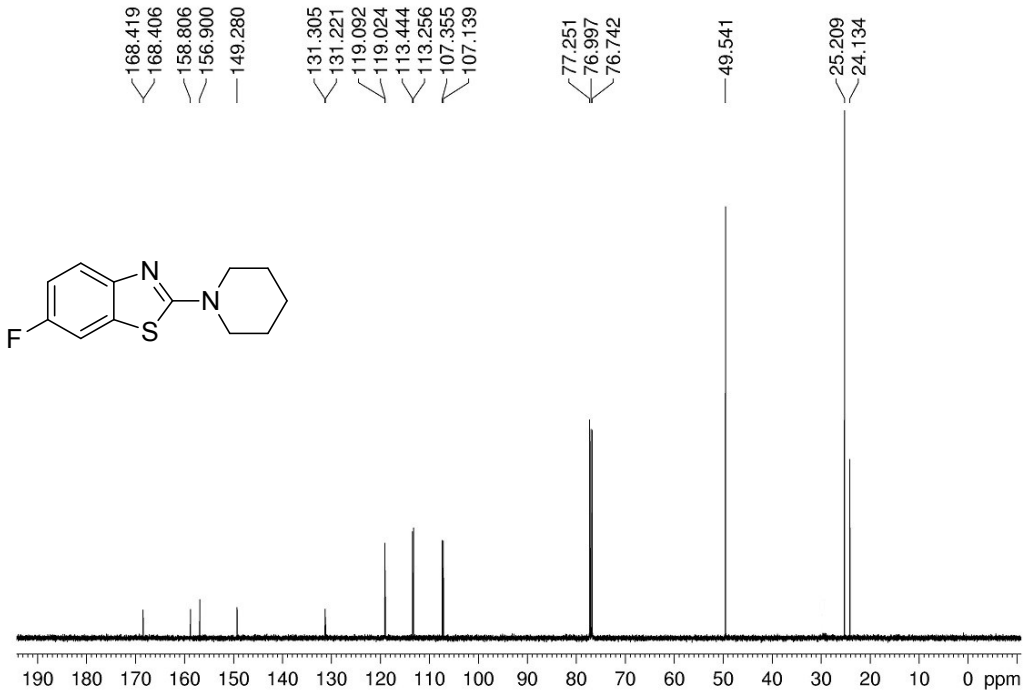

7.446
7.429
7.261
7.130
7.125
6.886
6.881
6.868
6.863
3.804
3.550
1.674
N
S
N
MeO
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.00
1.03
3.05
4.06
6.01

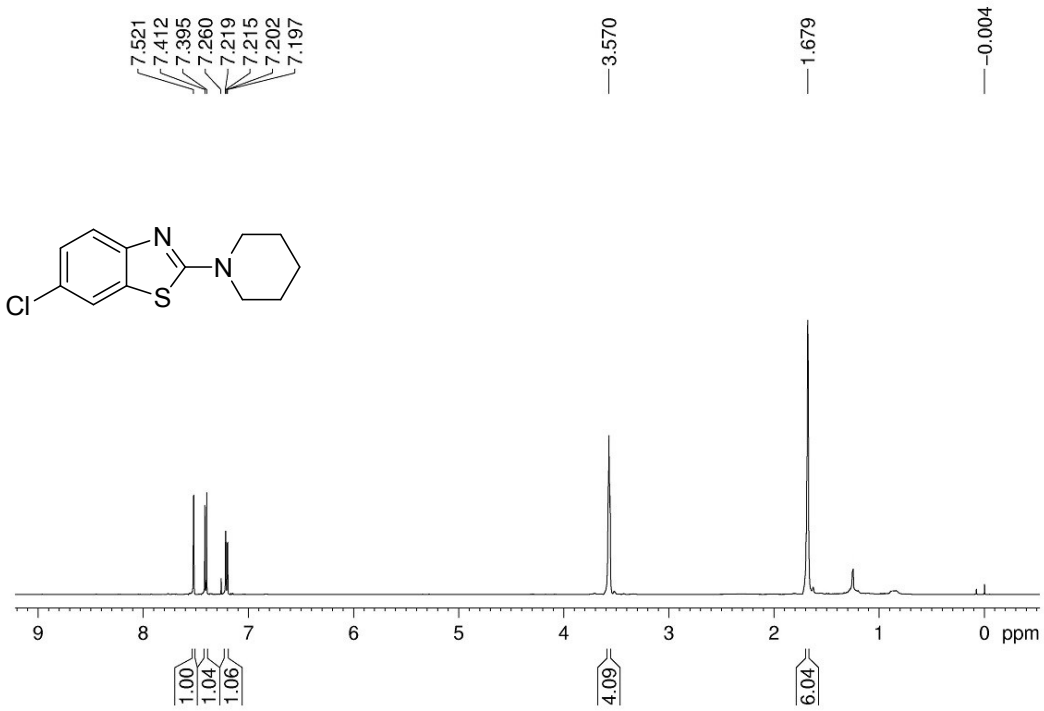

167.612
154.740
147.084
131.564
119.150
113.343
105.181
77.253
77.000
76.746
55.824
49.555
25.233
24.212
N
S
N
MeO
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

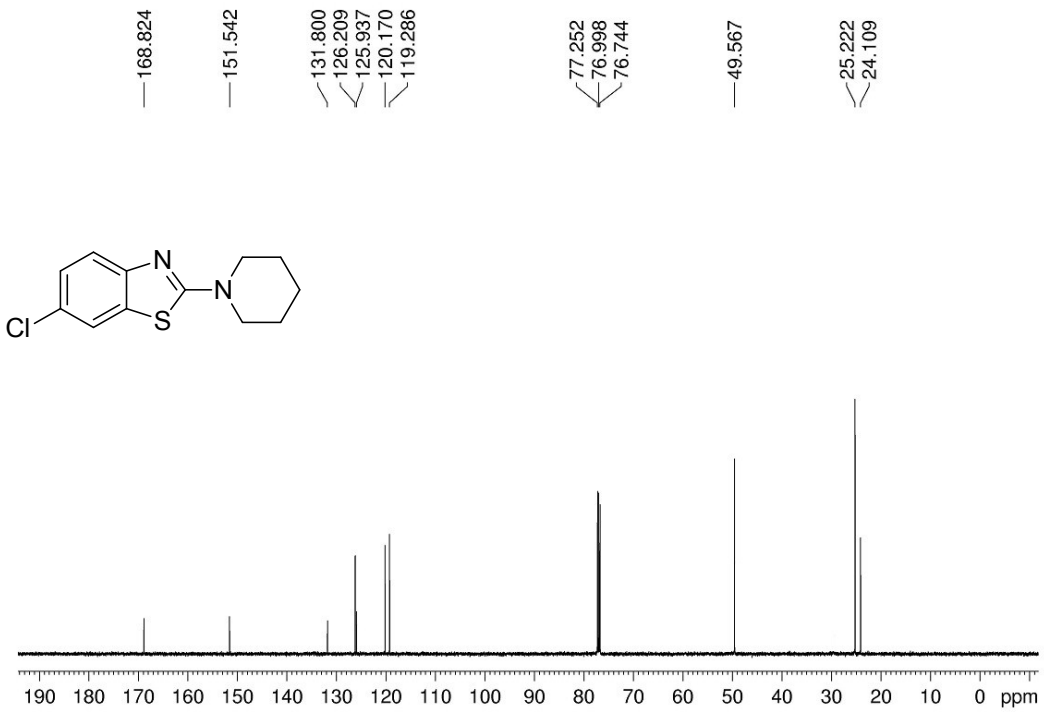

7.433
7.416
7.256
6.983
6.796
6.779
3.524
2.901
1.654
0.000
N
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.00
1.02
4.03
6.05
6.21

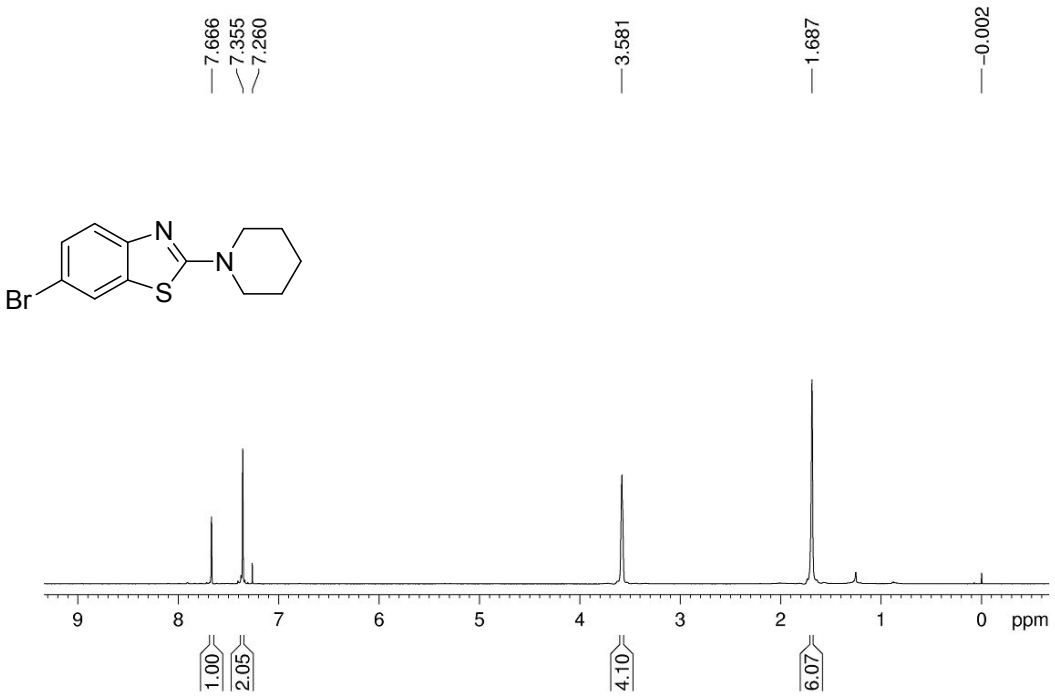

166.704
146.479
144.760
132.083
118.941
112.988
104.782
77.257
77.002
76.748
49.519
41.638
25.180
24.192
N
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

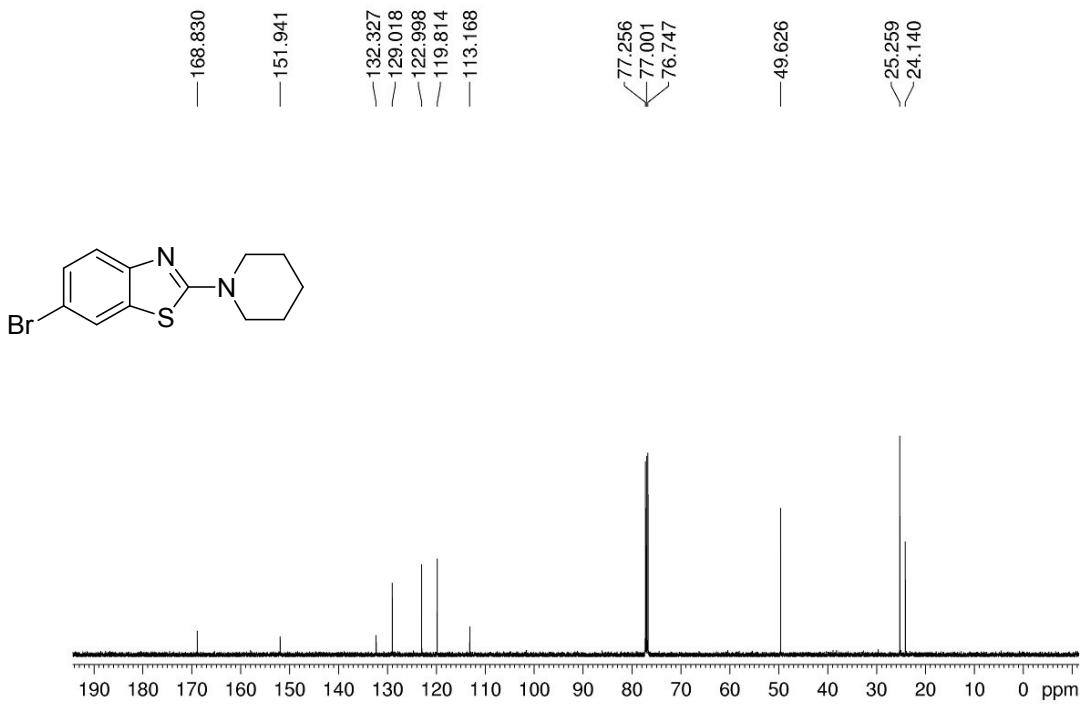

7.449
7.440
7.431
7.422
7.288
7.283
7.272
7.266
7.005
7.000
6.987
6.982
6.969
6.964
3.563
1.678
0.000
N
N
S
F
9
8
7
6
5
4
3
2
1
0 ppm
1.04
1.06
1.00
4.06
6.00

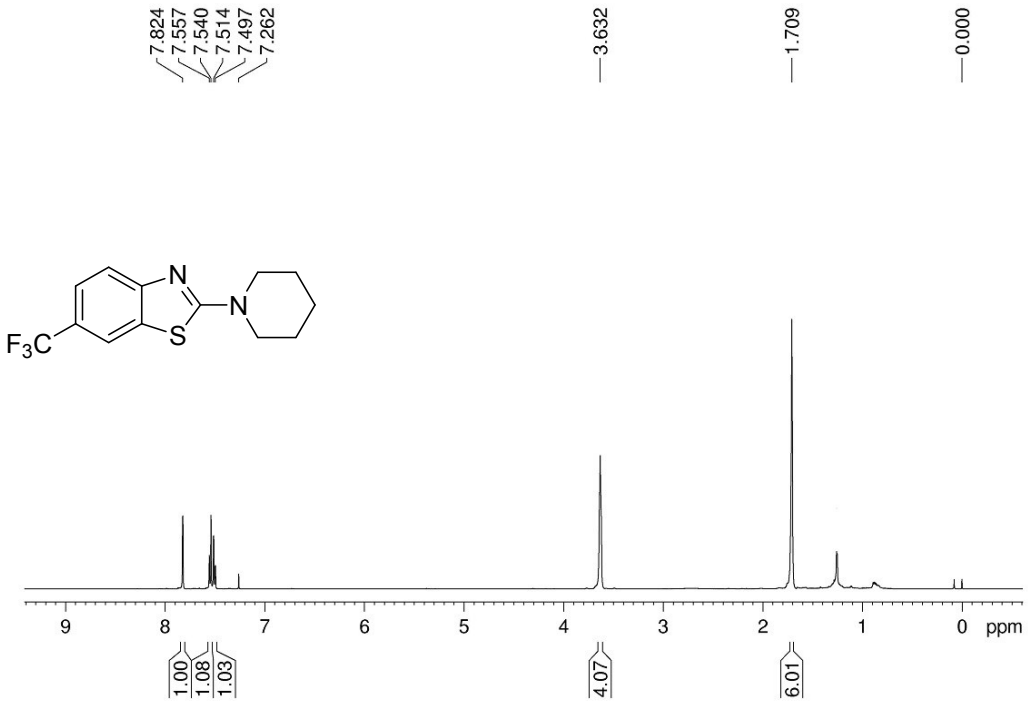

168.419
168.406
158.806
156.900
149.280
131.305
131.221
119.092
119.024
113.444
113.256
107.355
107.139
77.251
76.997
76.742
49.541
25.209
24.134
N
N
S
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

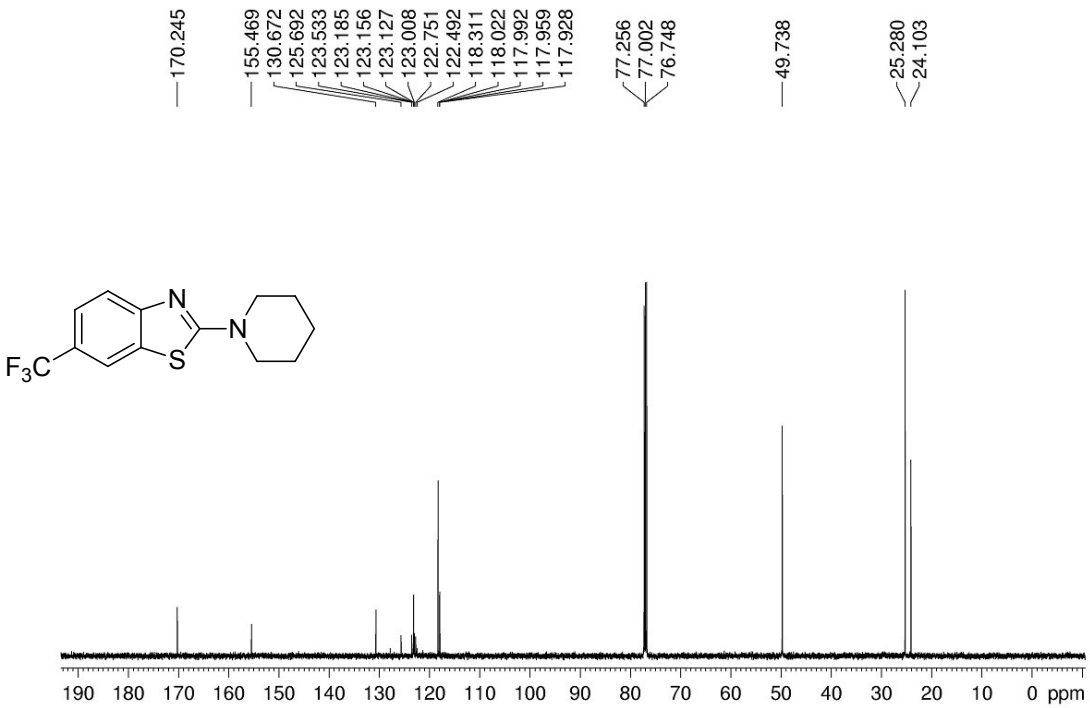

7.521
7.412
7.395
7.260
7.219
7.215
7.202
7.197
3.570
1.679
0.004
Cl
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.04
1.06
4.09
6.04

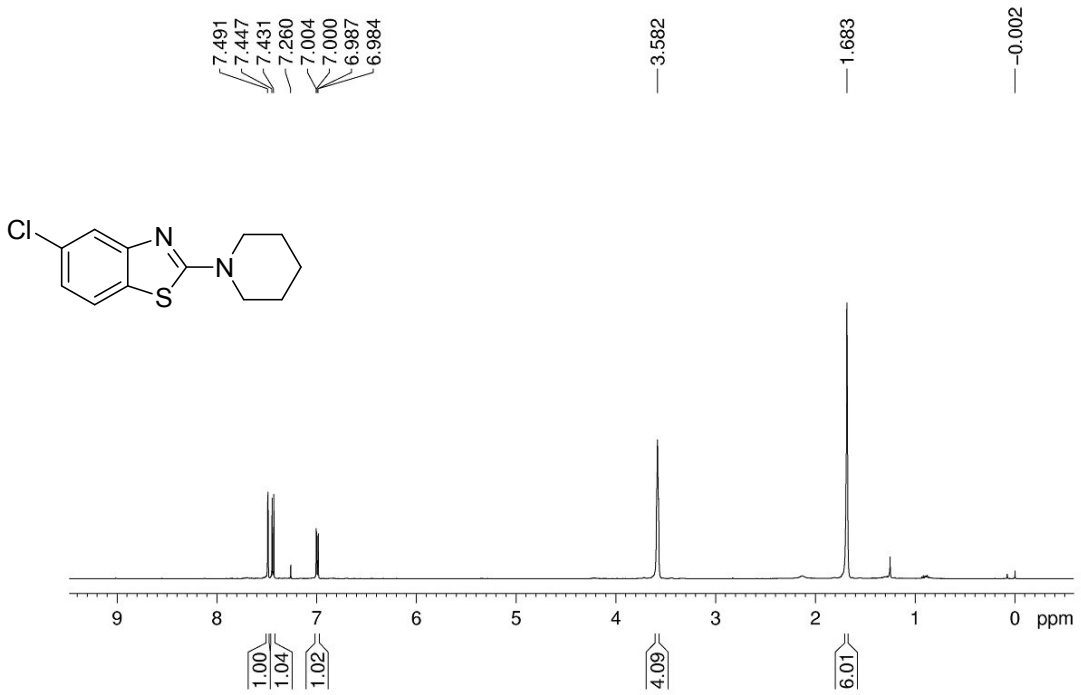

168.824
151.542
131.800
126.209
125.937
120.170
119.286
77.252
76.998
76.744
49.567
25.222
24.109
Cl
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

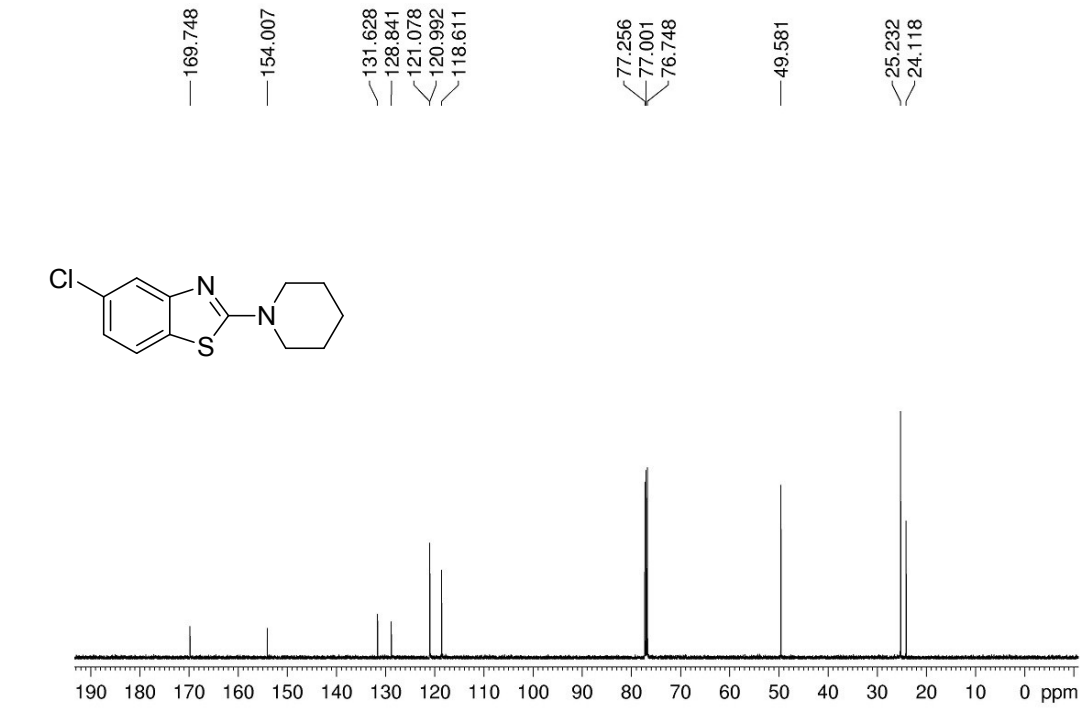

7.666
7.355
7.260
3.581
1.687
0.002
Br
N
S
N
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.05
4.10
6.07

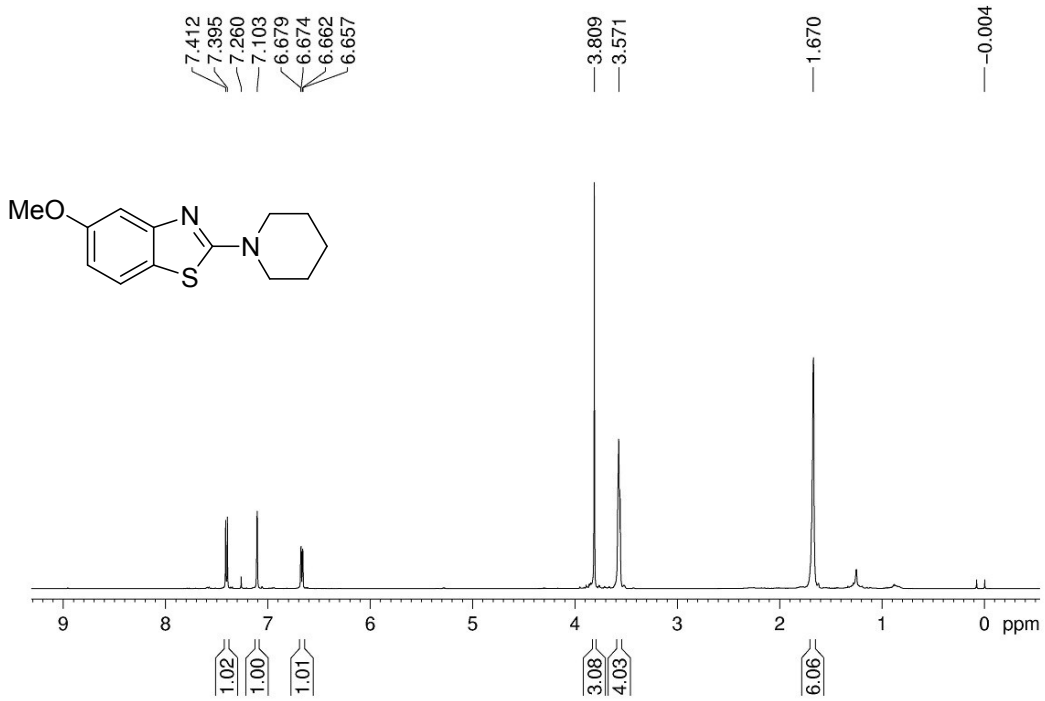

168.830
151.941
132.327
129.018
122.998
119.814
113.168
77.256
77.001
76.747
49.626
25.259
24.140
Br
N
S
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

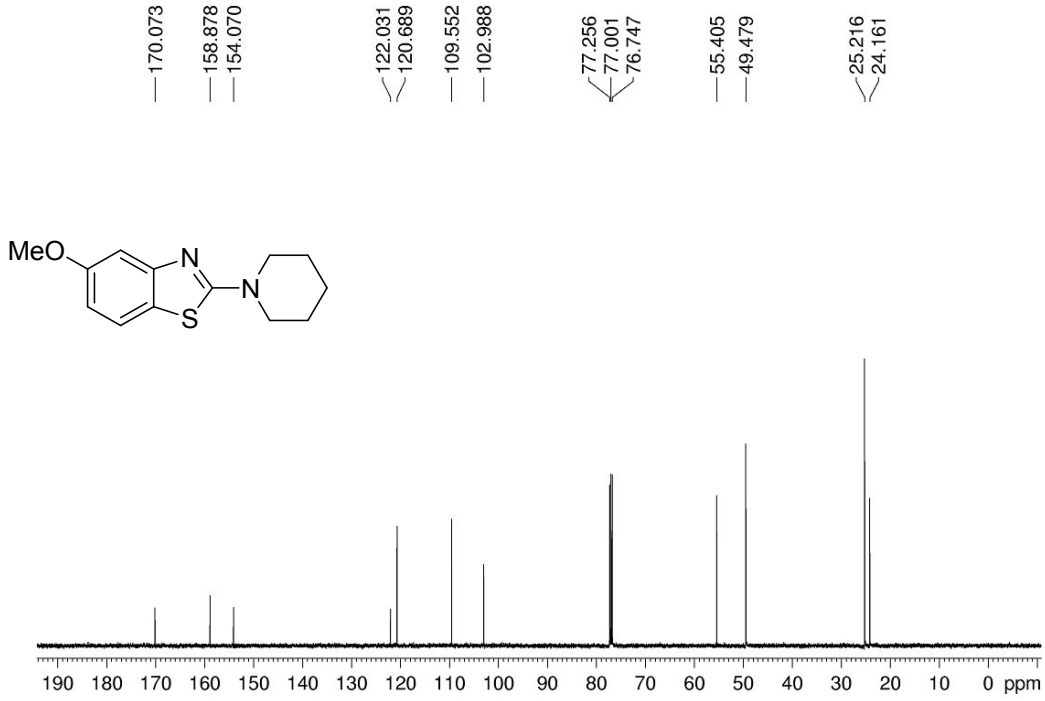

7.824
7.557
7.540
7.514
7.497
7.262
3.632
1.709
0.000
N
N
S
F3C
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.08
1.03
4.07
6.01


170.245
155.469
130.672
125.692
123.533
123.185
123.156
123.127
123.008
122.751
122.492
118.311
118.022
117.992
117.959
117.928
77.256
77.002
76.748
49.738
25.280
24.103
N
N
S
F3C
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.491
7.447
7.431
7.260
7.004
7.000
6.987
6.984
3.582
1.683
0.002
Cl
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.04
1.02
4.09
6.01


169.748
154.007
131.628
128.841
121.078
120.992
118.611
77.256
77.001
76.748
49.581
25.232
24.118
Cl
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.412
7.395
7.260
7.103
6.679
6.674
6.662
6.657
3.809
3.571
1.670
0.004
MeO
N
S
N
9
8
7
6
5
4
3
2
1
0 ppm
1.02
1.00
1.01
3.08
4.03
6.06


170.073
158.878
154.070
122.031
120.689
109.552
102.988
77.256
77.001
76.747
55.405
49.479
25.216
24.161
MeO
N
S
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm