Electronic supplementary materials *Mendeleev Commun.*, 2023, **33**, 118–120

Efficient thiocyanation of phenols and anilines in the $CeBr_3/H_2O_2$ system

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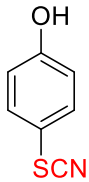
Contents

General experimental

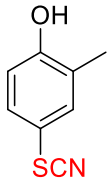
All reagents were obtained from local commercial suppliers and used without further purification. The progress of the reaction was monitored by TLC using analytical-grade silica gel plates (GF254) under UV light. Fourier transform Infrared data were recorded on an AVATAR-370 (Nicolet) spectrometer by transmission through the sample deposited on a KBr pellet. Melting points (m.p.) are determined with an Optimelt MPA 100 apparatus and are not corrected. ^1H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 at 300 MHz instrument respectively, using TMS as internal standard. Chemical shifts are given in parts per million (δ, ppm) and the coupling constants J are given in Hz. Mass spectrometry was performed on an LCMS-2010 EV (Shimadzu) instrument with an ESI source. The reaction temperature of the water bath was controlled at room temperature (25 ^oC).

*General procedure for the synthesis of **2a-n***. A solution of phenol **1a-g** or aniline **1h-n** (0.5 mmol, 1 equiv.), $CeBr_3$ (0.1 mmol, 0.2 equiv.) and ammonium thiocyanate (1.5 mmol, 3 equiv.) in EtOAc (2 ml) was cooled to 0 ^oC. Then 30% H_2O_2 (3.5 equiv.) was added dropwise in 30 min. After completion, the mixture was stirred for more 2 h at room temperature (TLC control). The residue was extracted with ethyl acetate (3×5 ml) and washed with water. The combined extracts were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel with petroleum ether/ethyl acetate to afford the pure desired product **2a-n**. Compounds **5,6** were synthesized using the same procedure.

Characterization data of products 2, 5, 6

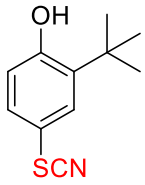


*4-Thiocyanatophenol (**2a**)*.[S1] Brown solid (74.5 mg, 99%). M.p. 48~50 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.0, 134.2, 117.5, 113.4, 112.1. MS (ESI): 152.0 $[M + H]^+$.

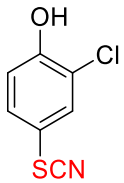


*2-Methyl-4-thiocyanatophenol (**2b**)*.[S2] Yellow solid (78 mg, 95%). M.p. 73~75 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 2.4 Hz, 1H), 7.25 (d, *J* = 2.5 Hz, 1H), 6.79 (d, *J* = 8.4 Hz, 1H), 6.45 (s, 1H), 2.22 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.6, 135.1, 131.3, 127.5, 116.9, 112.8, 112.2, 15.9. MS (ESI): 166.0 $[M + H]^+$.

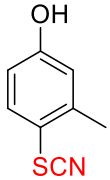
*2-Methoxy-4-thiocyanatophenol (**2c**)*.[S1] White solid (85 mg, 94%). M.p. 95~97 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.01 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.96 (d, *J* = 2.1 Hz, 1H), 6.88 – 6.82 (m, 1H), 6.02 (s, 1H), 3.82 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.9, 147.6, 126.3, 116.0, 114.5, 112.9, 111.9, 56.3. MS (ESI): 182.0 $[M + H]^+$.



*2-(tert-Butyl)-4-thiocyanatophenol (**2d**)*.[S3] Yellow oil (94 mg, 91%).^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (d, *J* = 2.4 Hz, 1H), 7.28 (dd, *J* = 8.4, 2.5 Hz, 1H), 6.74 (d, *J* = 8.3 Hz, 1H), 6.30 (s, 1H), 1.39 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.0, 138.9, 131.9, 131.6, 118.3, 112.60, 112.58, 35.0, 29.6. MS (ESI): 208.2 $[M + H]^+$.

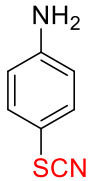


*2-Chloro-4-thiocyanatophenol (**2e**)*.[S4] White solid (79 mg, 85%). M.p. 98~100 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (d, *J* = 2.3 Hz, 1H), 7.41 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.08 (d, *J* = 8.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 132.7, 132.5, 121.5, 118.0, 114.8, 110.9. MS (ESI): 186.1 $[M + H]^+$.

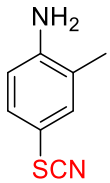


*3-Methyl-4-thiocyanatophenol (**2f**)*.[S2] Yellow solid (79 mg, 96%). M.p. 77~79 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.5 Hz, 1H), 6.77 (t, *J* = 6.4 Hz, 1H), 6.69 (dt, *J* = 18.0, 9.0 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 143.4, 136.2, 118.7, 115.1, 112.22, 112.20, 21.0. MS (ESI): 166.0 $[M + H]^+$.

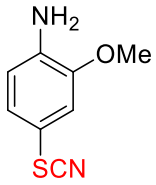
*3-Methoxy-4-thiocyanatophenol (**2g**)*.[S1] White solid (86 mg, 95%). M.p. 138~140 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.27 (s, 1H), 7.39 (t, J = 6.7 Hz, 1H), 6.58 (t, J = 4.3 Hz, 1H), 6.49 (dd, J = 8.5, 2.4 Hz, 1H), 3.87 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 162.3, 159.7, 135.4, 111.9, 109.4, 101.0, 99.0, 56.8. MS (ESI): 182.2 $[M + H]^+$.



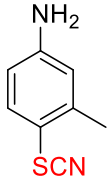
*4-Thiocyanatoaniline (**2h**)*.[S1] Brown solid (69 mg, 97%). M.p. 52~54 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.41 (d, J = 8.6 Hz, 2H), 6.74 (d, J = 8.6 Hz, 2H), 5.88 (s, 2H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 151.9, 135.6, 115.6, 113.8, 105.2. MS (ESI) *m/z* 151.0 $[M + H]^+$.



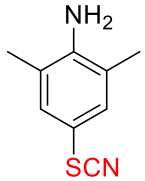
*2-Methyl-4-thiocyanatoaniline (**2i**)*.[S3] Brownish solid (69 mg, 92%). M.p. 67~69 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.23 – 7.19 (m, 2H), 6.70 (d, J = 8.2 Hz, 1H), 5.54 (s, 2H), 2.07 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 150.0, 135.1, 132.8, 123.4, 115.3, 113.5, 105.3, 17.6. MS (ESI) *m/z* 165.0 $[M + H]^+$.



*2-Methoxy-4-thiocyanatoaniline (**2j**)*.[S5]Brown solid (84 mg, 93%). M.p. 51~53 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.07 – 7.03 (m, 2H), 6.73-6.70 (d, J = 8.1 Hz, 1H), 5.42 (s, 2H), 3.81 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 147.1, 141.4, 127.8, 115.5, 114.4, 113.6, 105.5, 56.1. MS (ESI) *m/z* 181.0 $[M + H]^+$.

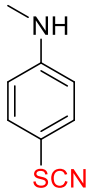


*3-Methyl-4-thiocyanatoaniline (**2k**)*.[S1] Brown solid (75 mg, 91%). M.p. 81~83 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.29 (d, J = 8.4 Hz, 1H), 6.58 (dd, J = 2.5, 0.5 Hz, 1H), 6.49 (dd, J = 8.4, 2.6 Hz, 1H), 5.72 (s, 2H), 2.35 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 150.0, 135.5, 132.8, 123.4, 115.3, 113.6, 105.3, 17.6. MS (ESI) *m/z* 165.0 $[M + H]^+$.

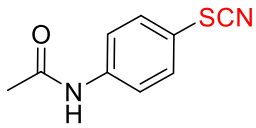


*2,6-Dimethyl-4-thiocyanatoaniline (**2l**)*.[S6] Brown solid (81 mg, 91%). M.p. 88~90 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.15 (s, 2H), 5.23 (s, 2H), 2.11 (s, 6H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 147.9, 133.5, 122.9,

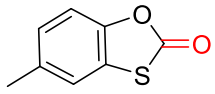
113.5, 105.3, 18.1. MS (ESI) *m/z* 179.0 $[M + H]^+$.



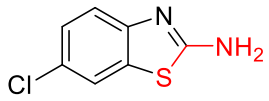
*N-methyl-4-thiocyanatoaniline (**2m**)*.[S5] Brownish solid (74 mg, 90%). M.p. 48~50 °C. ^{1}H NMR (300 MHz, DMSO-d_6) 7.42 – 7.37 (m, 2H), 6.64 – 6.59 (m, 2H), 6.37 (d, J = 4.6 Hz, 1H), 2.69 (d, J = 4.9 Hz, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 152.3, 135.3, 113.6, 113.3, 105.1, 29.7. MS (ESI) *m/z* 165.0 $[M + H]^+$.



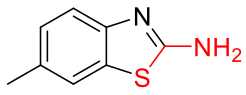
*N-(4-thiocyanatophenyl)acetamide (**2n**)*.[S7] Brown solid (85 mg, 88%). M.p. 190~192 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.22 (s, 1H), 7.73-7.70 (d, J = 8.8 Hz, 2H), 7.60-7.57 (d, J = 8.8 Hz, 2H), 2.06(s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 169.4, 141.5, 132.9, 120.8, 116.9, 112.5, 24.5. MS (ESI) *m/z* 193.0 $[M + H]^+$.



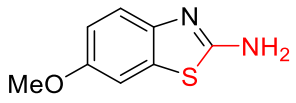
*5-Methylbenzo[d][1,3]oxathiol-2-one (**5**)*.[S1] White solid (74 mg, 89%). M.p. 80~82 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.07 (t, J = 3.0 Hz, 1H), 7.00 (d, J = 1.0 Hz, 2H), 2.33 (d, J = 0.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 149.1, 134.7, 127.5, 123.8, 121.9, 110.7, 20.8. MS (ESI) *m/z* 167.1 $[M + H]^+$.



*6-Chlorobenzo[d]thiazol-2-amine (**6a**)*.[S8]White solid (75 mg, 82%). M.p. 196~198 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, J = 2.1 Hz, 1H), 7.62 (s, 2H), 7.30 (d, J = 8.5 Hz, 1H), 7.20 (dd, J = 8.6, 2.2 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 152.3, 132.8, 126.3, 125.0, 121.0, 119.4. MS (ESI) *m/z* 185.6 $[M + H]^+$.



*6-Methylbenzo[d]thiazol-2-amine (**6b**)*.[S9] White solid (76 mg, 93%). M.p. 133~135 °C. ^{1}H NMR (100 MHz, DMSO-d_6) 7.42 (s, 1H), 7.35 (s, 2H), 7.21 (d, J = 8.1 Hz, 1H), 7.01 – 6.99 (m, 1H), 2.29 (s, 3H). ^{13}C NMR (75 MHz, DMSO) δ 166.2, 151.1, 131.5, 130.4, 126.9, 121.3, 117.9, 21.2. MS (ESI) *m/z* 165.0 $[M + H]^+$.



*6-Methoxybenzo[d]thiazol-2-amine (**6c**)*.[S1] White solid (82 mg, 91%). M.p. 130~132 °C. ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.29 – 7.26 (m, 4H), 6.82-6.78 (dd, J = 8.7, 2.7 Hz, 1H), 3.72 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 165.3, 154.8, 147.3, 132.4, 118.6, 113.4, 106.0, 56.0. MS (ESI) *m/z* 181.0 $[M + H]^+$.

References

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S6. D. S. Bhalerao and K. G. Akamanchi, *Synlett*, 2007, 2952.

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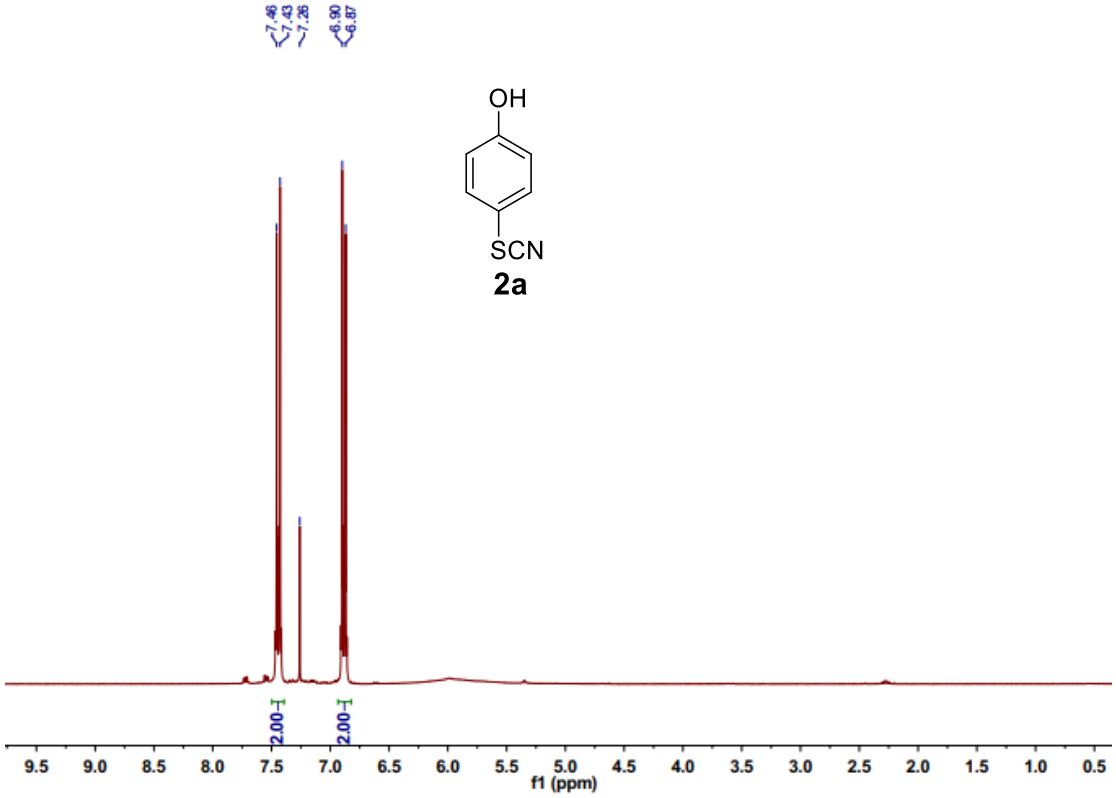
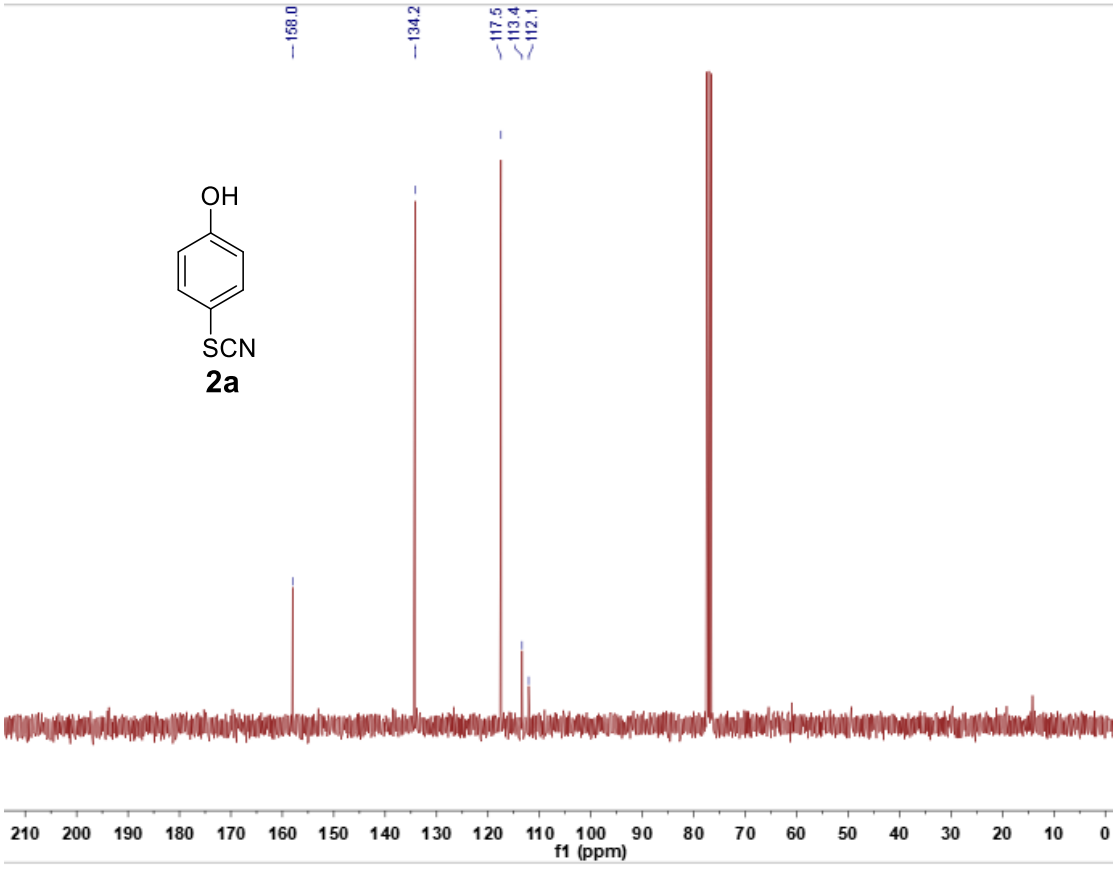
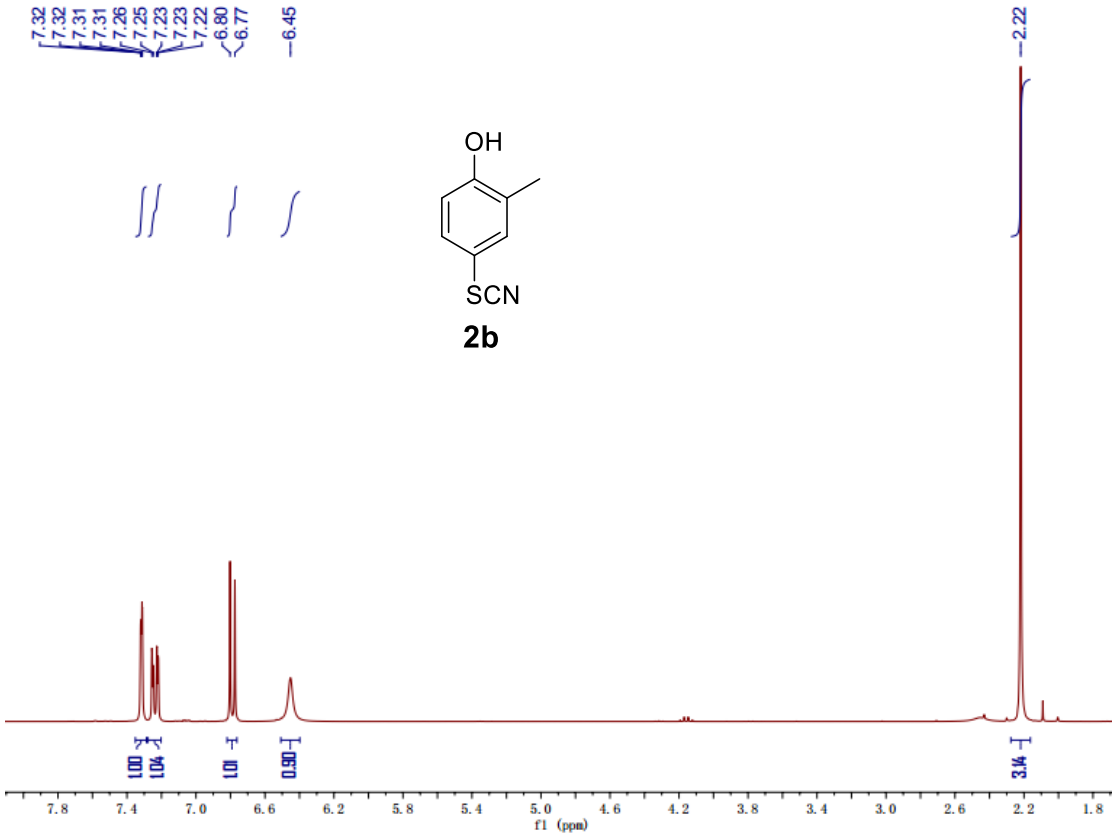
Copies of ^{1}H and ^{13}C NMR Spectra



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SCN
2b
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7.8
7.4
7.0
6.6
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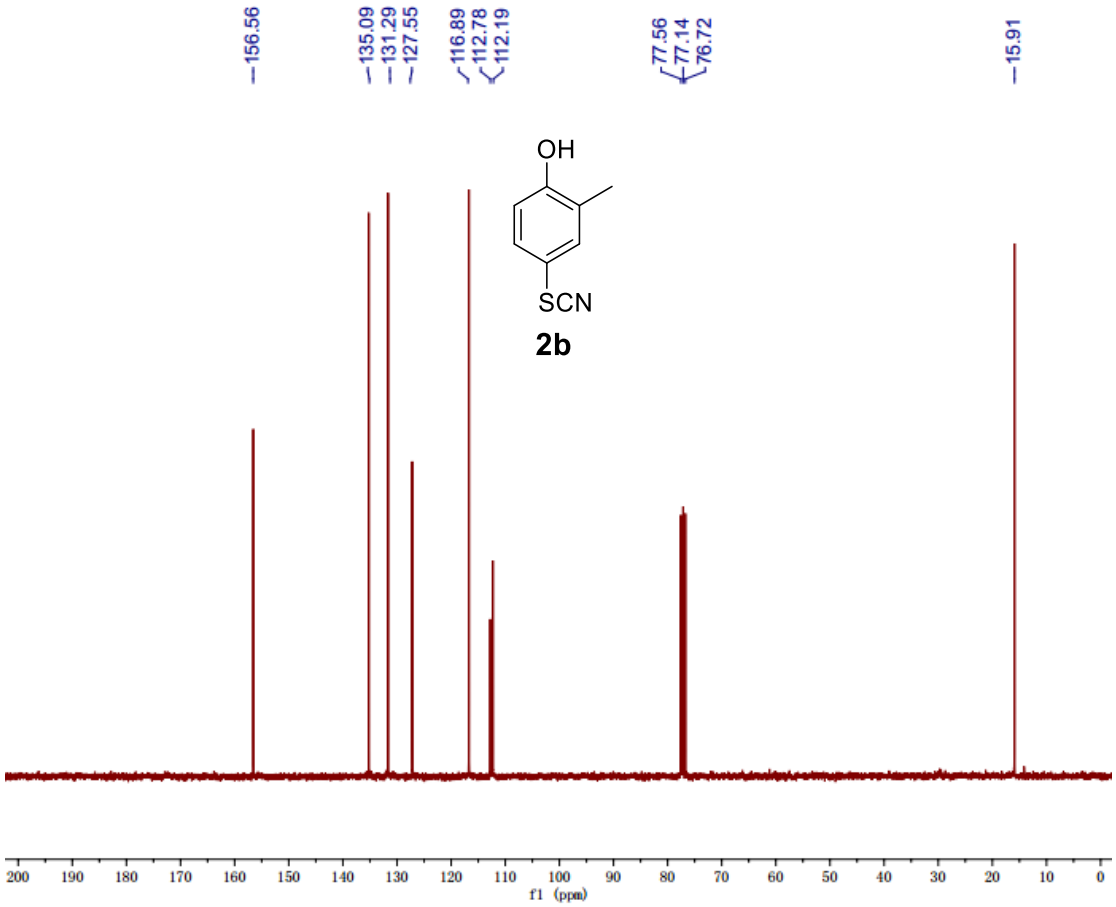
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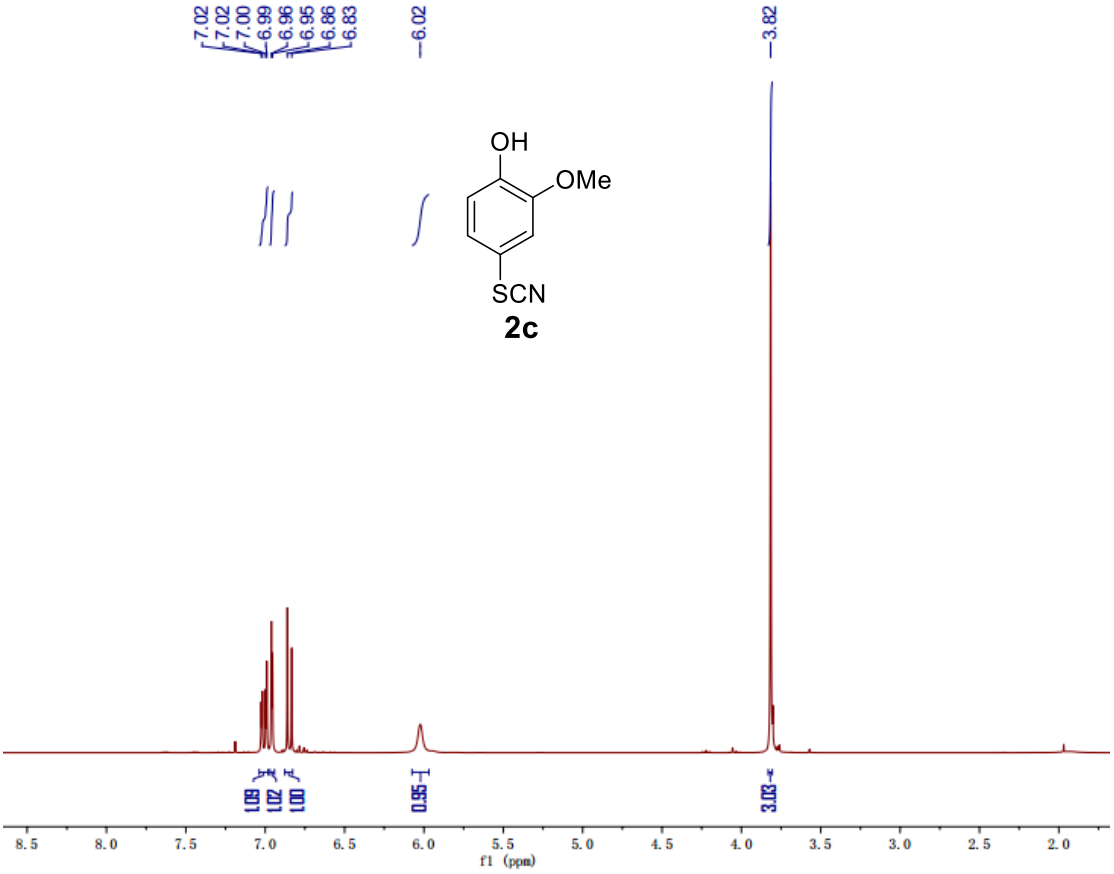
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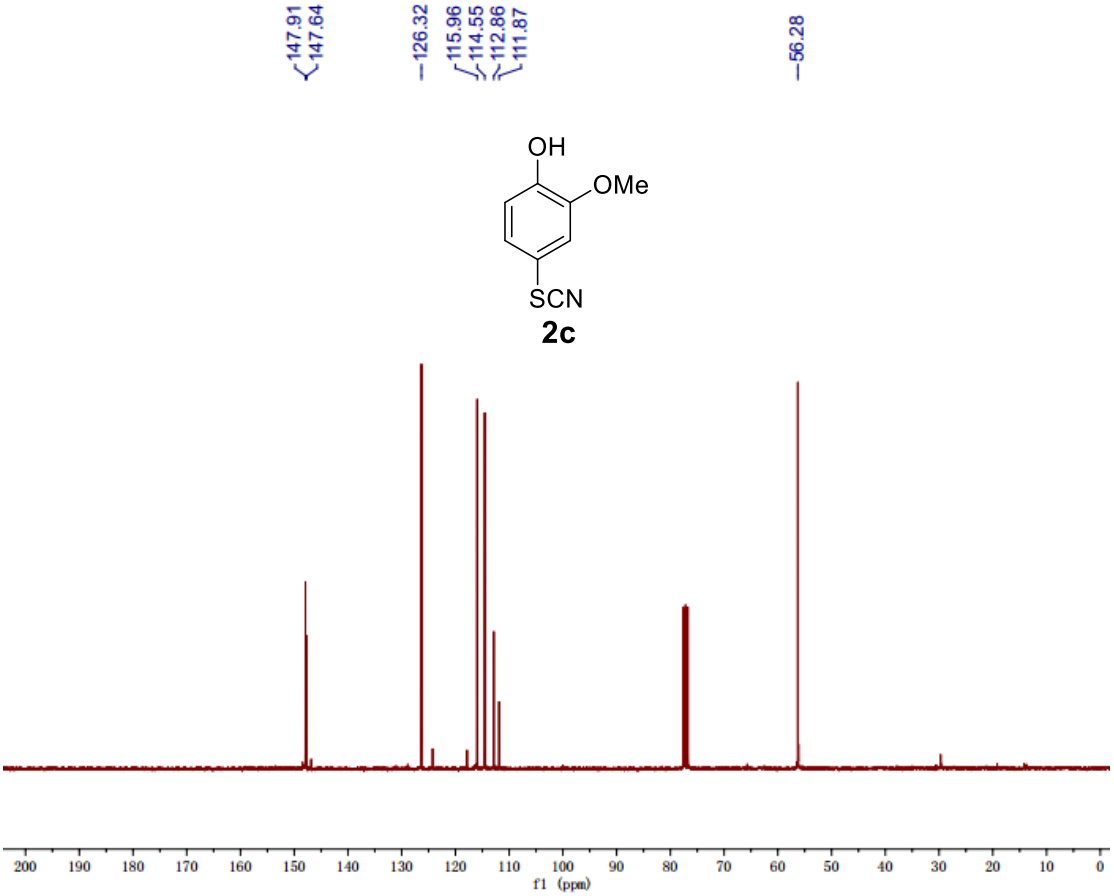
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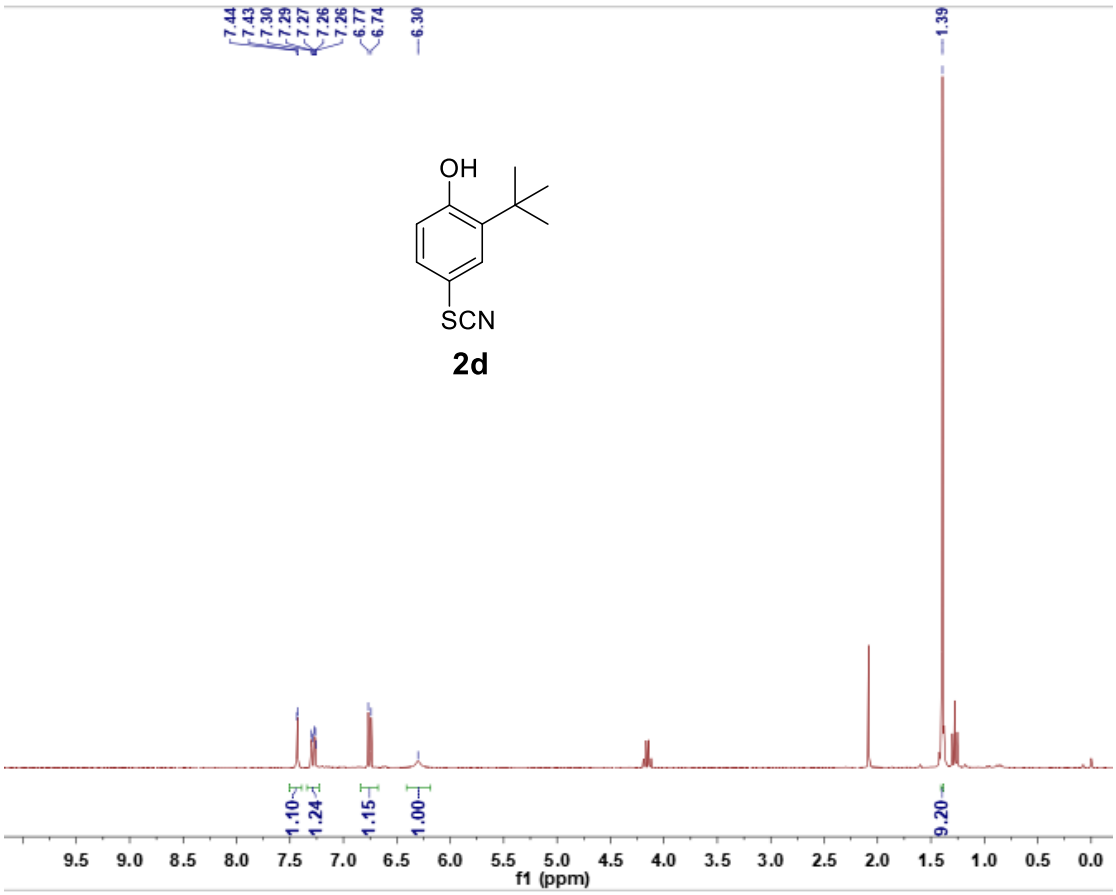
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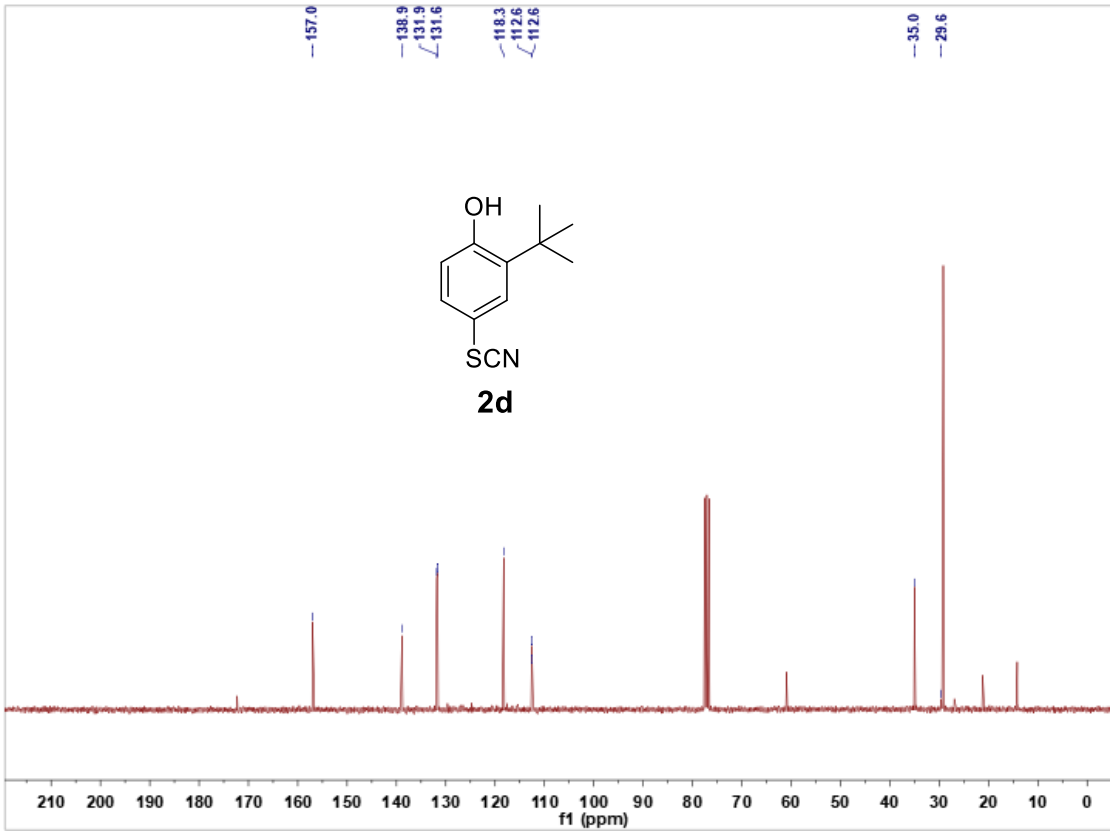
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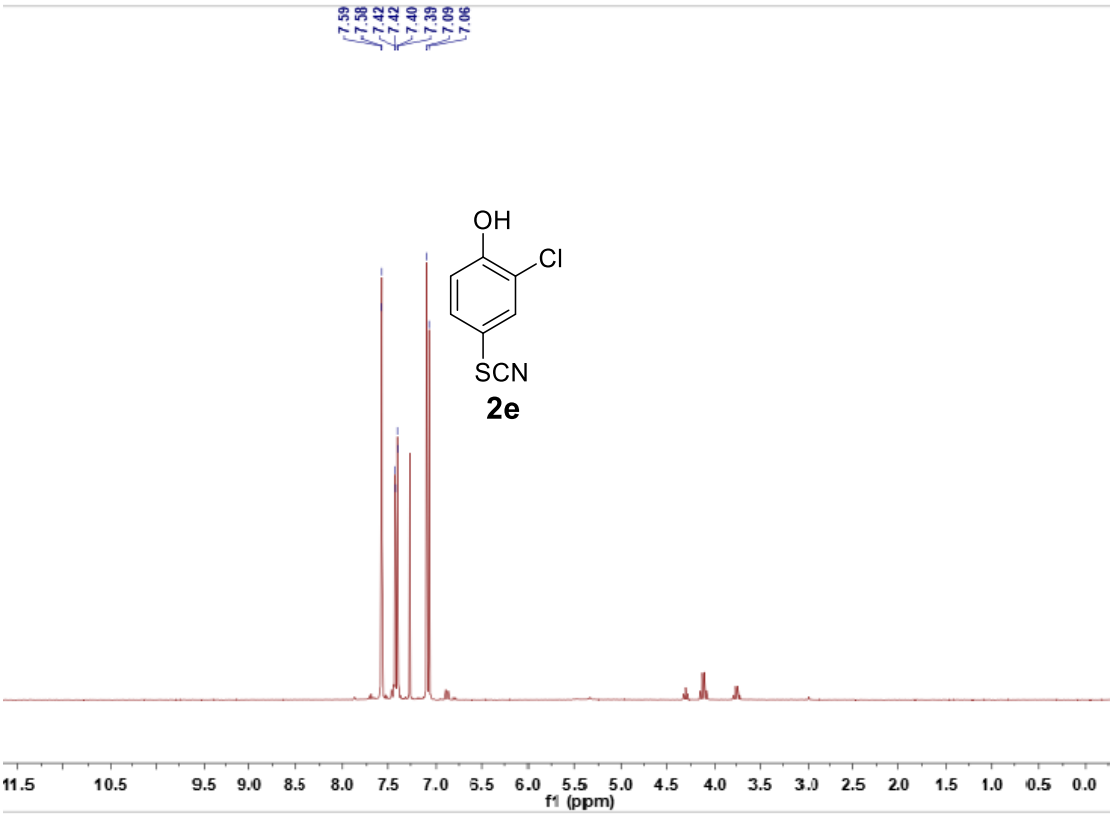
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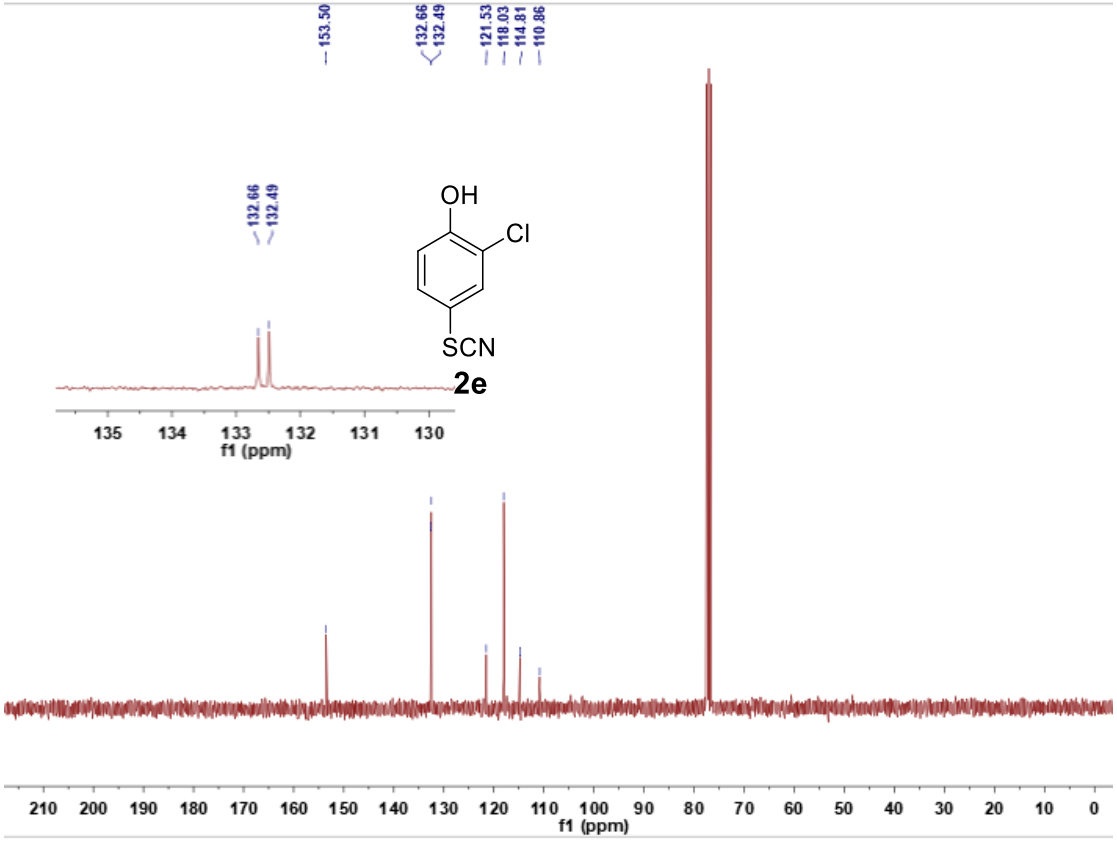
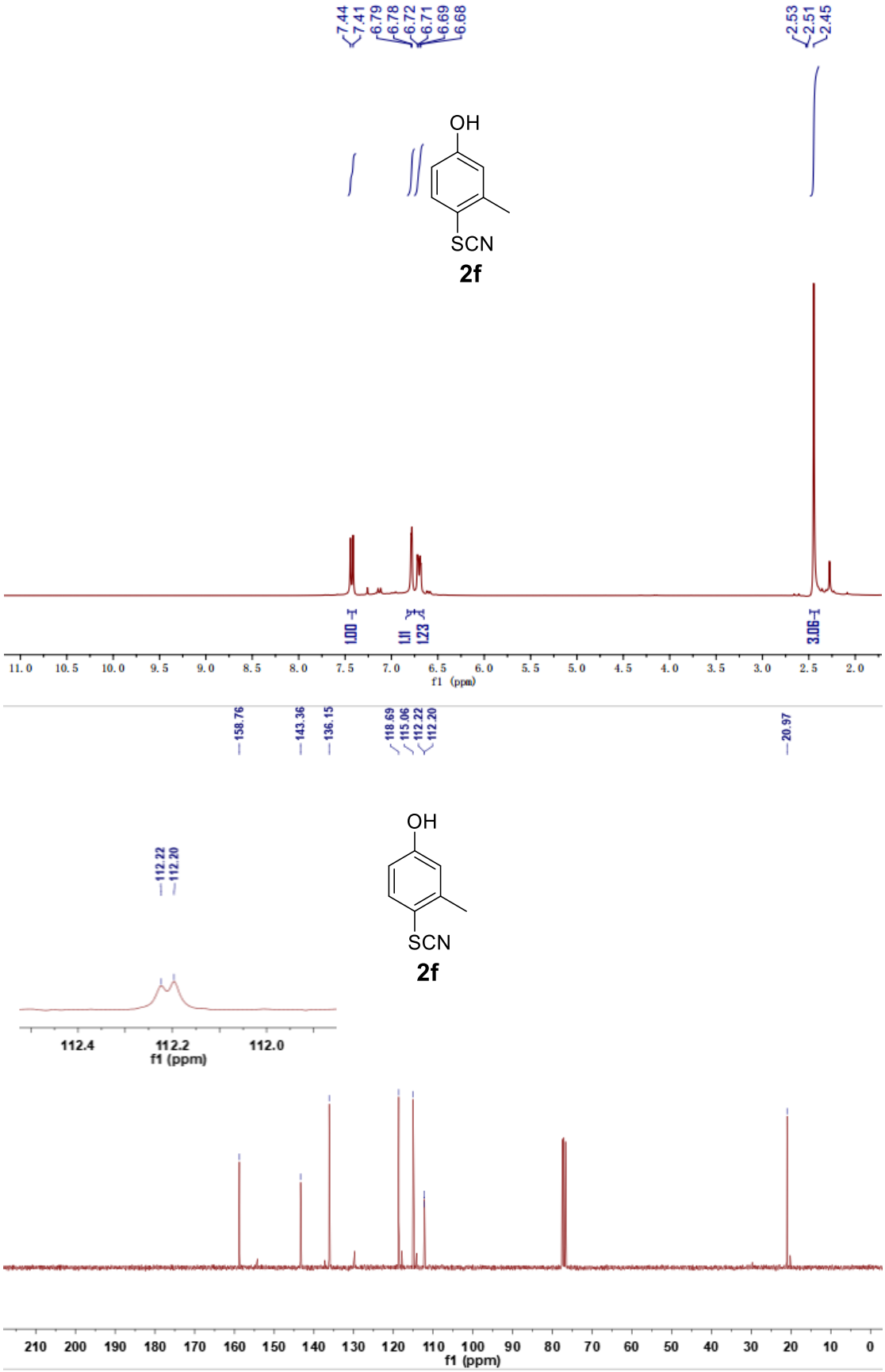
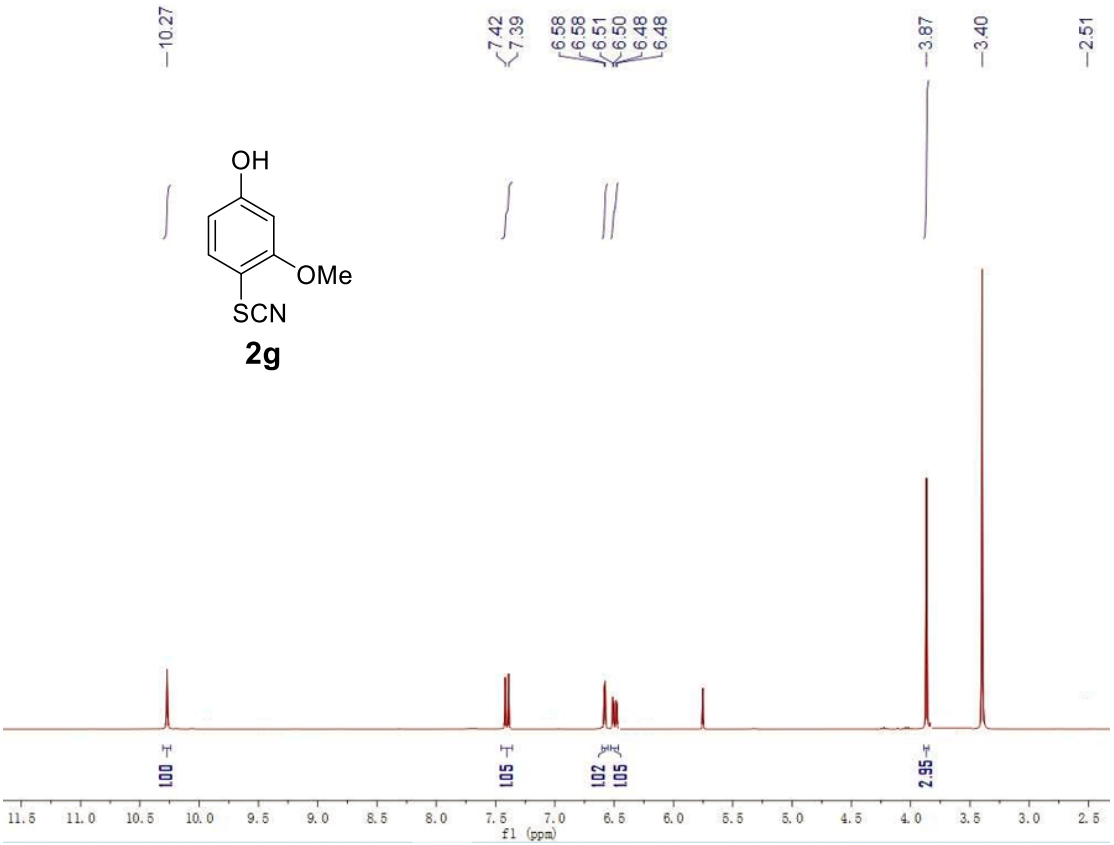
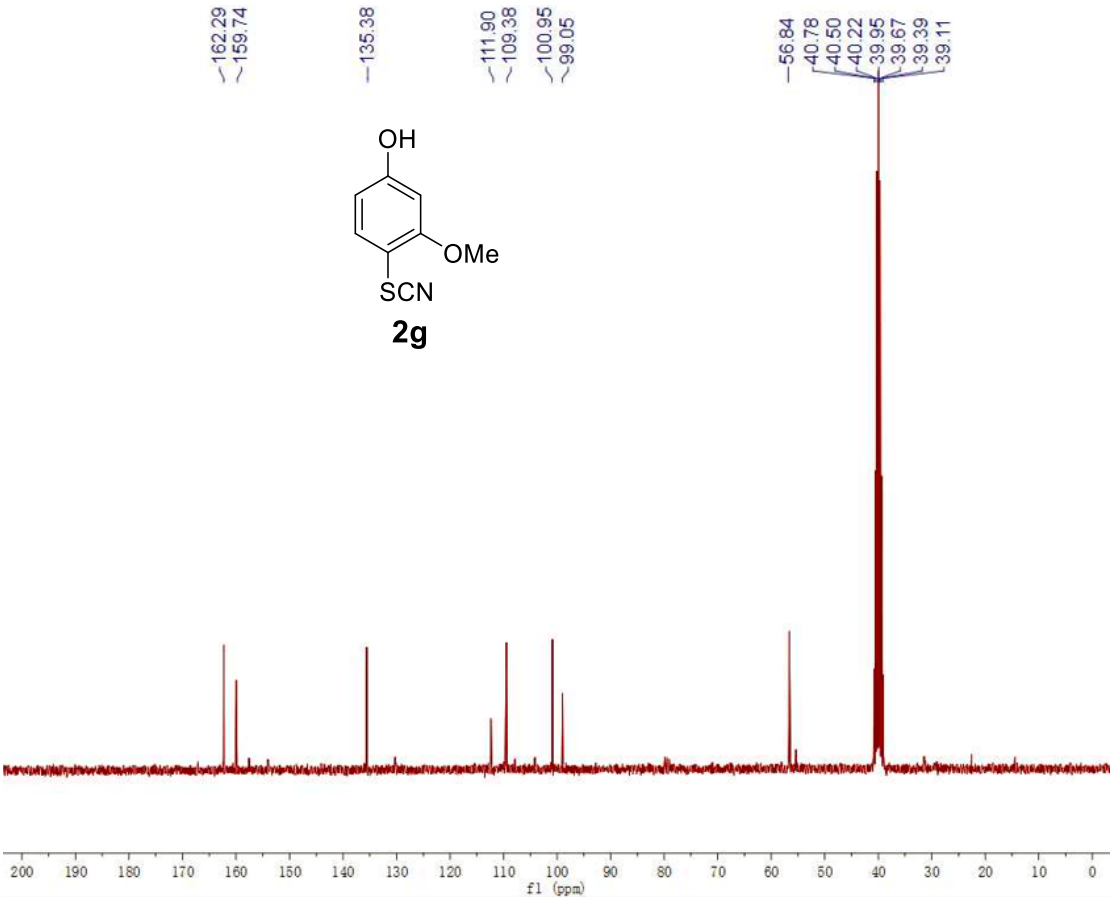
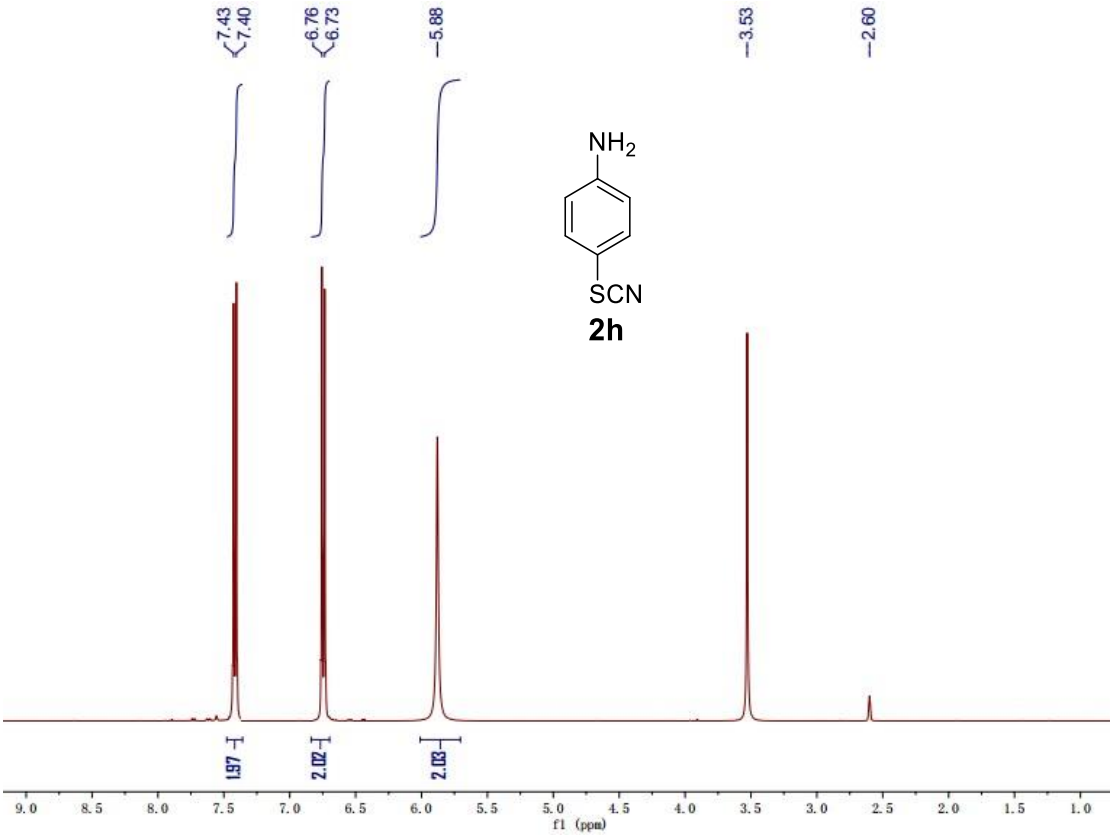
2e
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

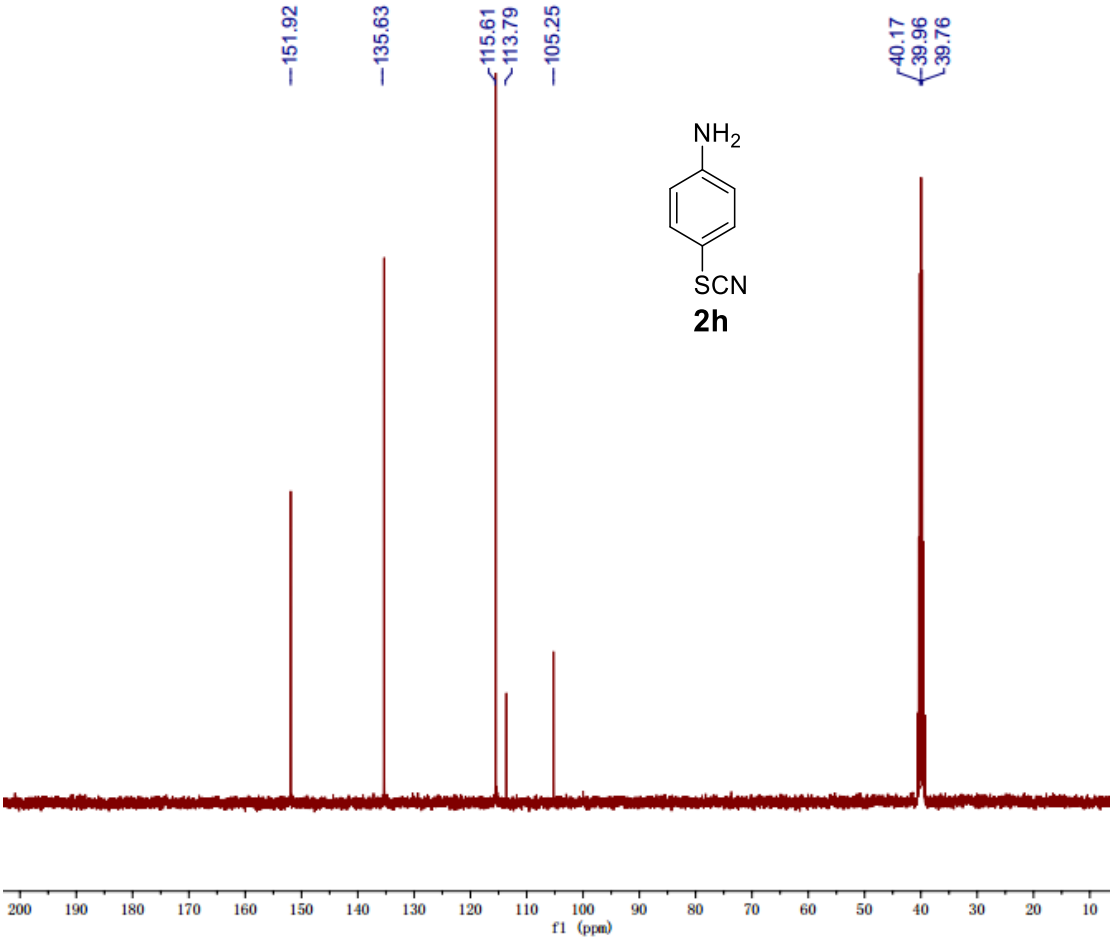

153.50
132.66
132.49
121.53
118.03
114.81
110.86
132.66
132.49
OH
Cl
SCN
2e
135 134 133 132 131 130
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

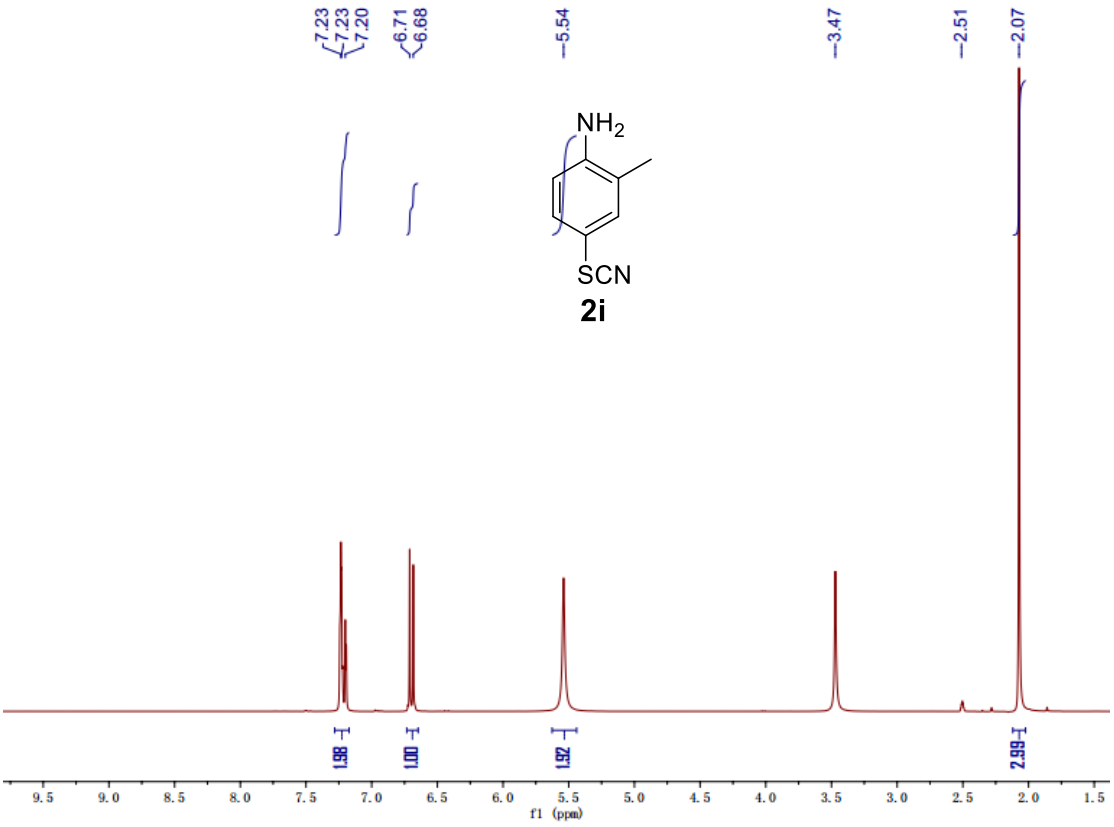

7.44
7.41
6.79
6.78
6.72
6.71
6.69
6.68
2.53
2.51
2.45
OH
SCN
2f
1.00
1.11
1.23
3.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)
158.76
143.36
136.15
118.69
115.06
112.22
112.20
20.97
112.22
112.20
OH
SCN
2f
112.4 112.2 112.0
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

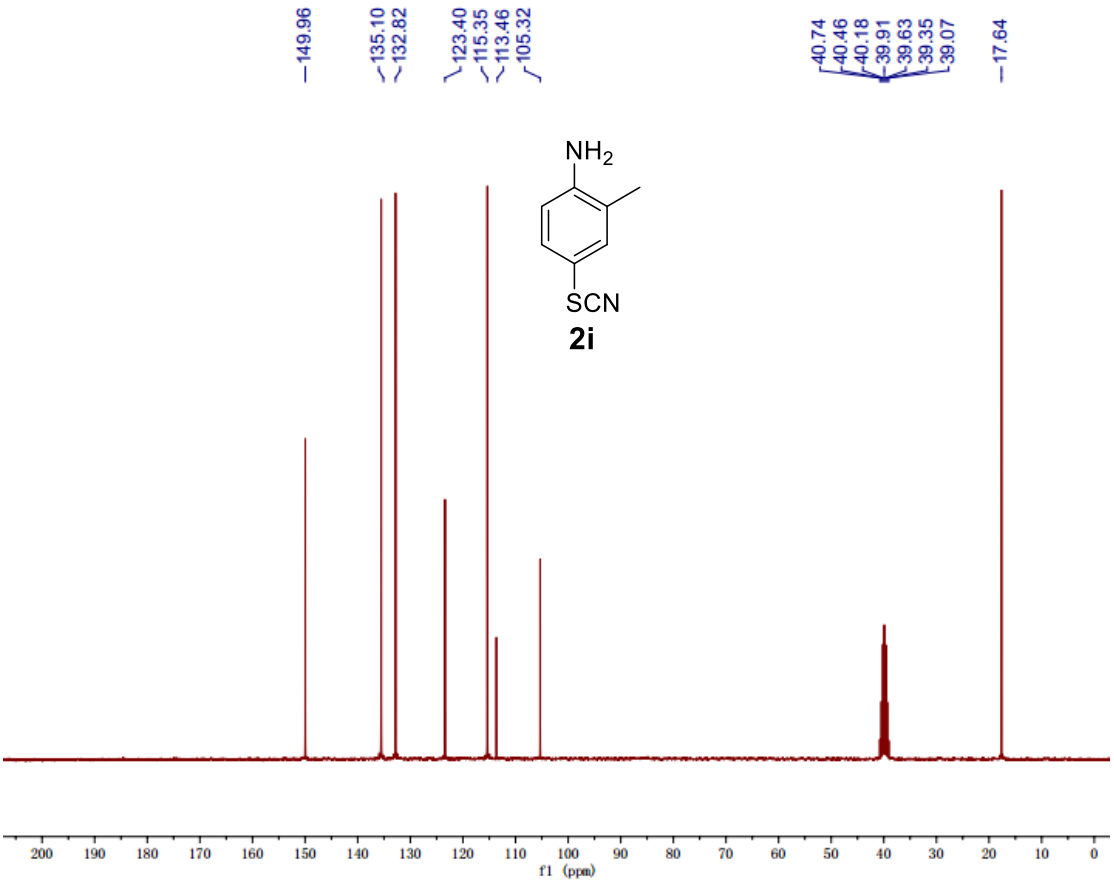

10.27
7.42
7.39
6.58
6.58
6.51
6.50
6.48
6.48
3.87
3.40
2.51
OH
OMe
SCN
2g
1.00
1.05
1.02
1.05
2.95
f1 (ppm)

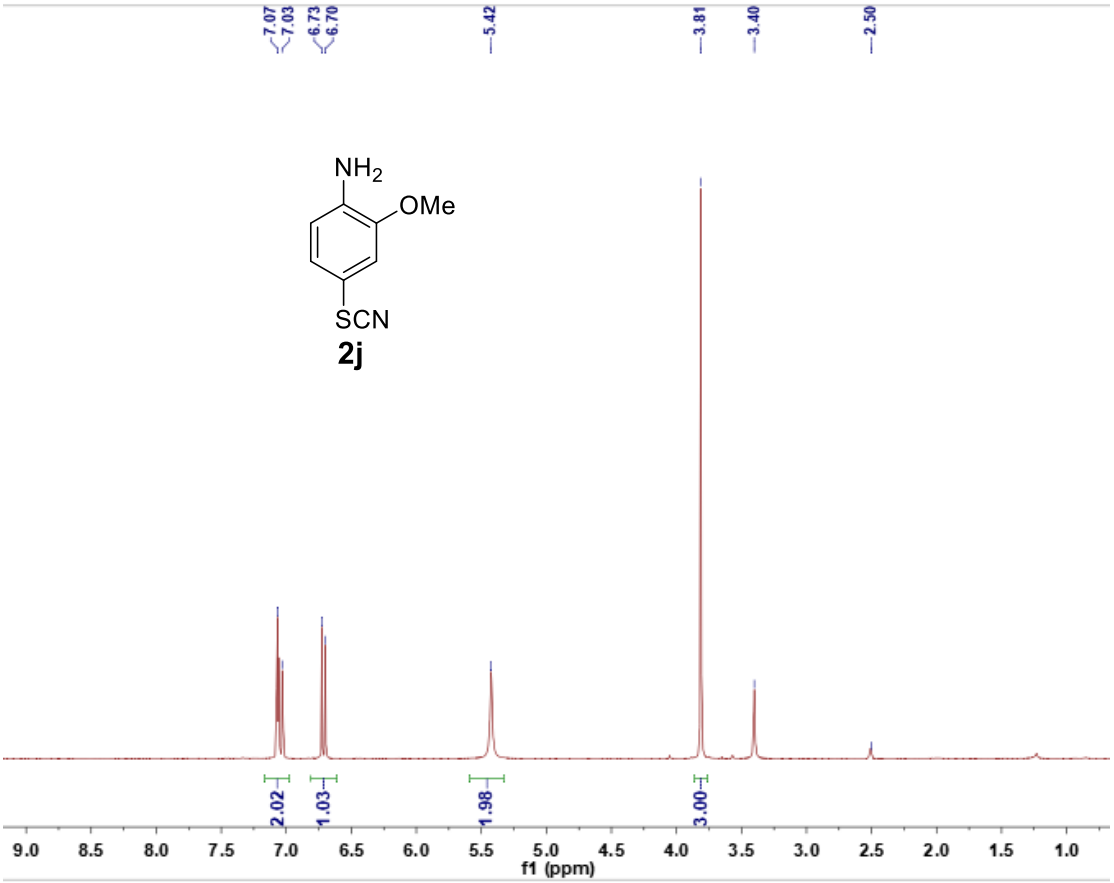

162.29
159.74
135.38
111.90
109.38
100.95
99.05
56.84
40.78
40.50
40.22
39.95
39.67
39.39
39.11
OH
OMe
SCN
2g
f1 (ppm)

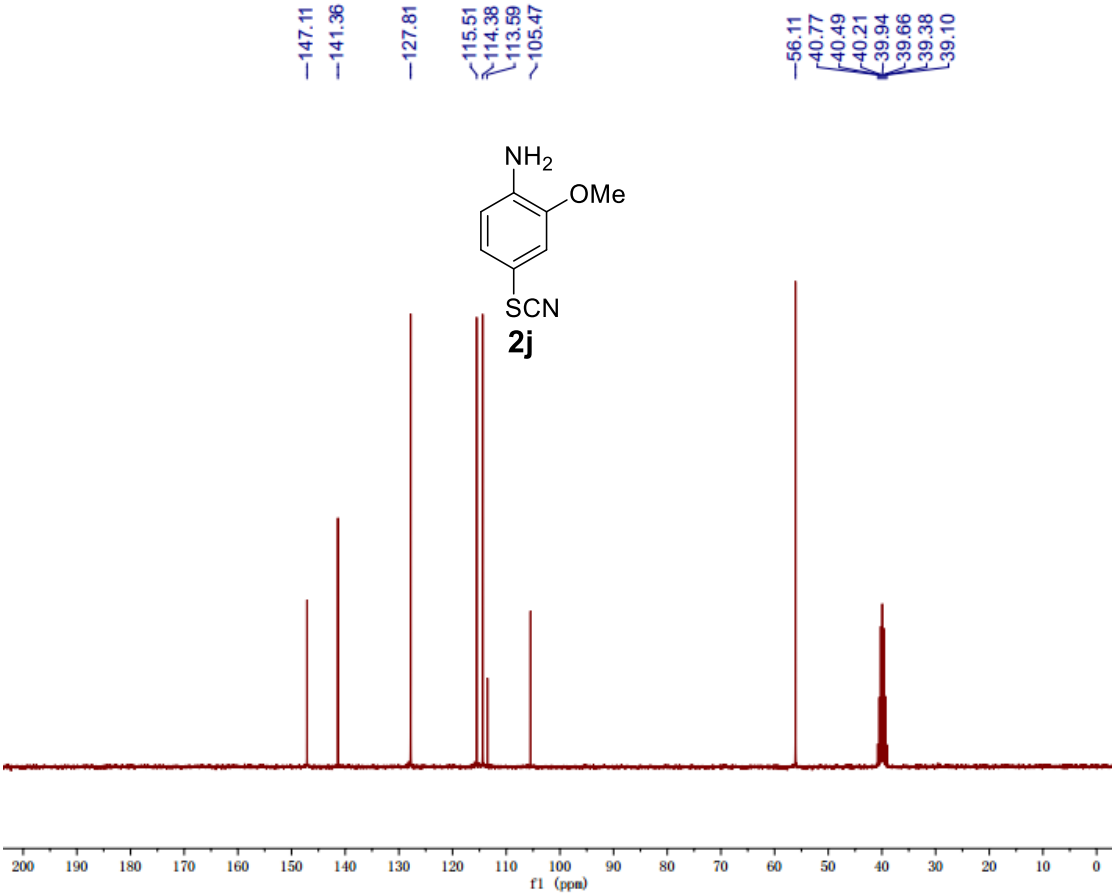

7.43
7.40
6.76
6.73
5.88
3.53
2.60
NH2
SCN
2h
1.97
2.02
2.03
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

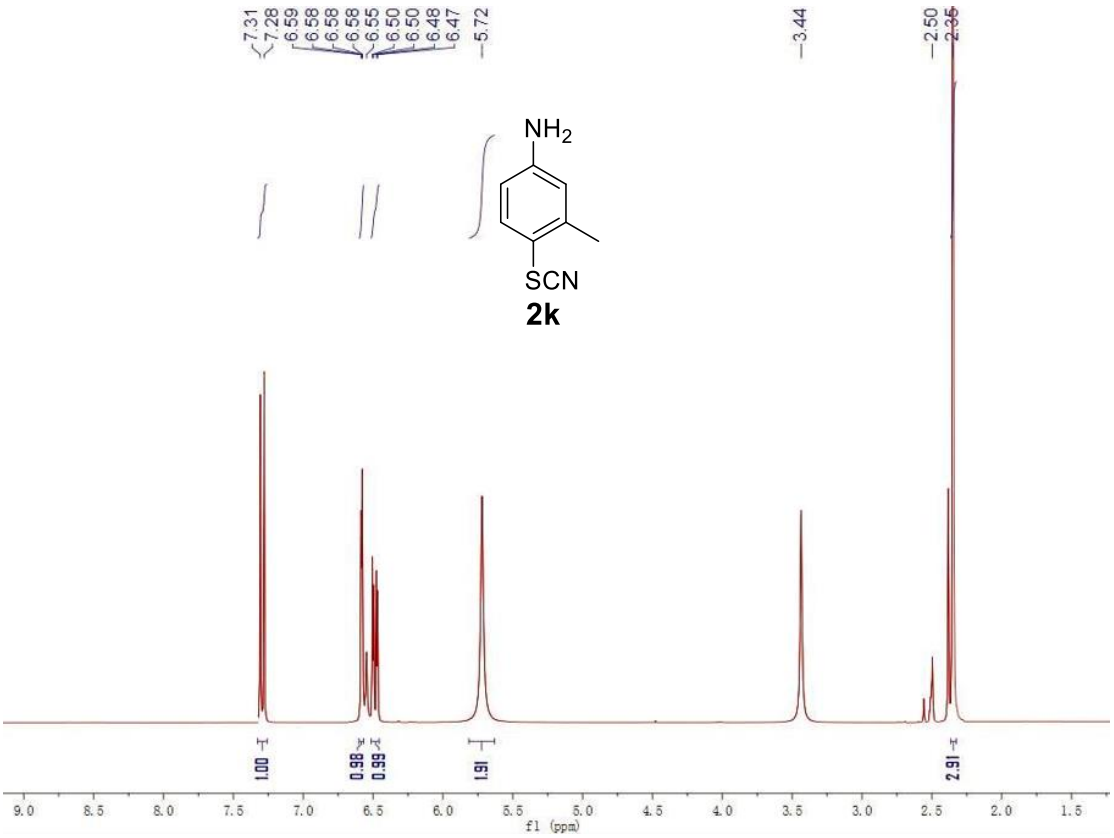

151.92
135.63
115.61
113.79
105.25
40.17
39.96
39.76
NH2
SCN
2h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

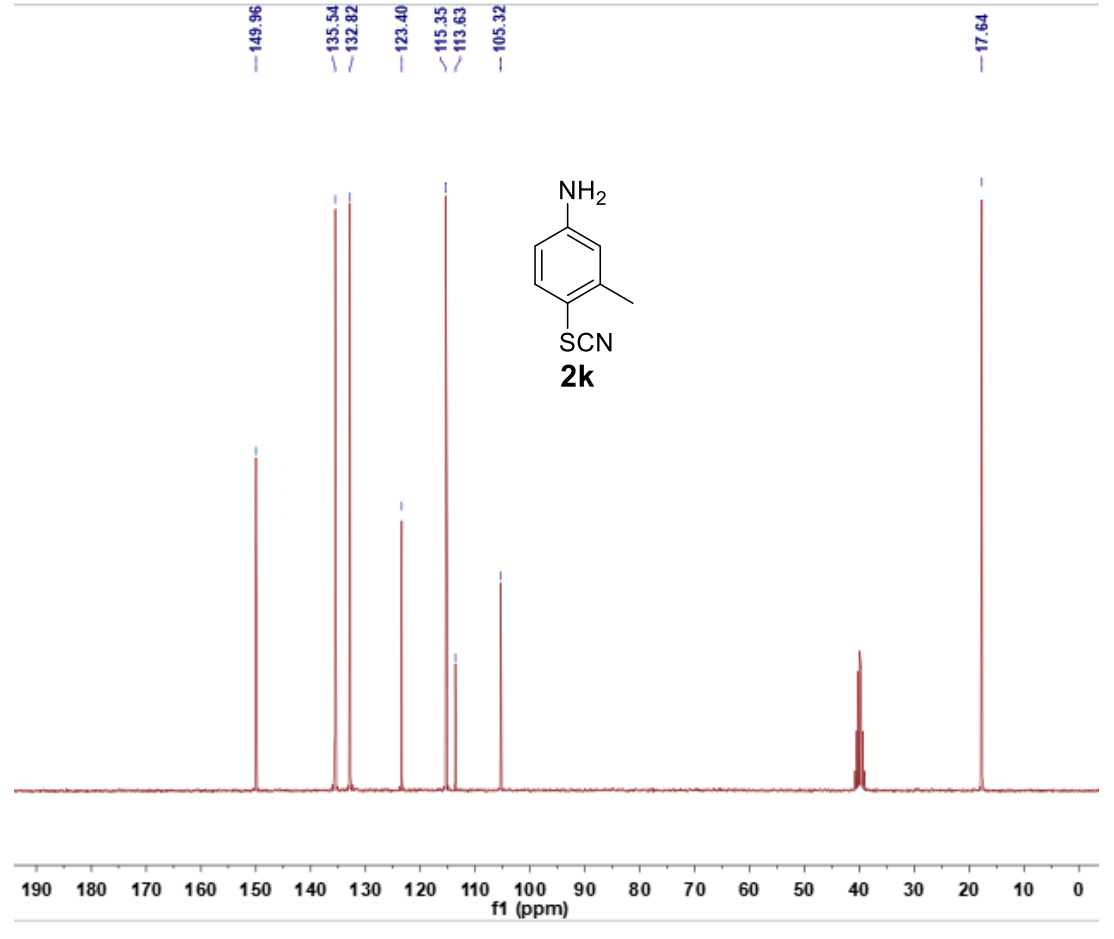

7.23
7.23
7.20
6.71
6.68
5.54
3.47
2.51
2.07
NH2
SCN
2i
1.98
1.00
1.92
2.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

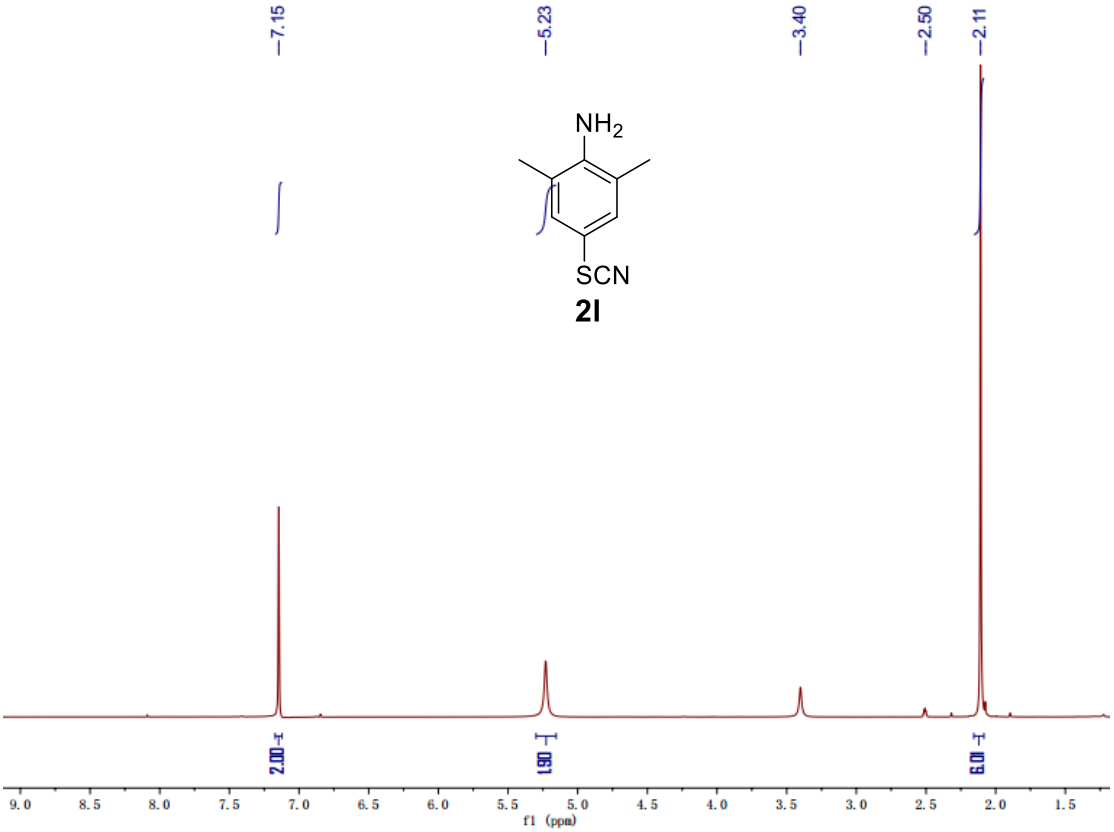

149.96
135.10
132.82
123.40
115.35
113.46
105.32
40.74
40.46
40.18
39.91
39.63
39.35
39.07
17.64
NH2
SCN
2i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

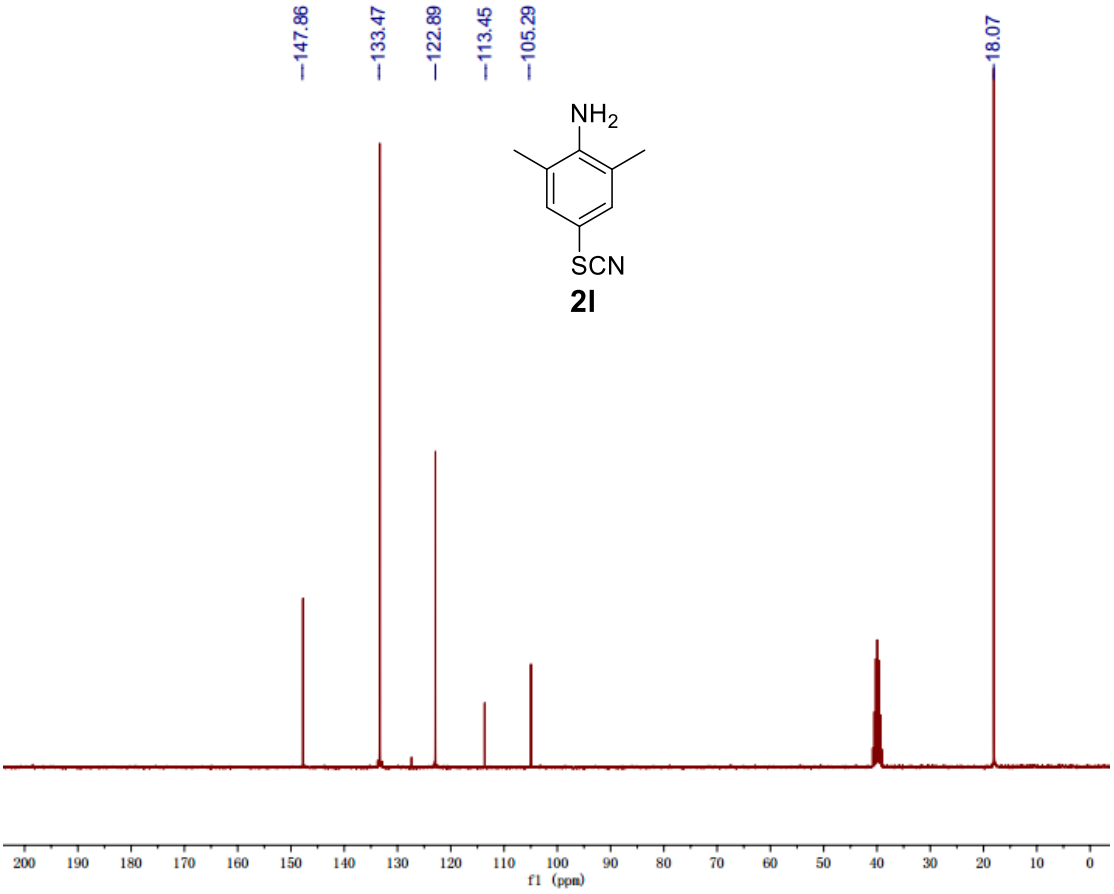

7.07
7.03
6.73
6.70
5.42
3.81
3.40
2.50
NH2
OMe
SCN
2j
2.02
1.03
1.98
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

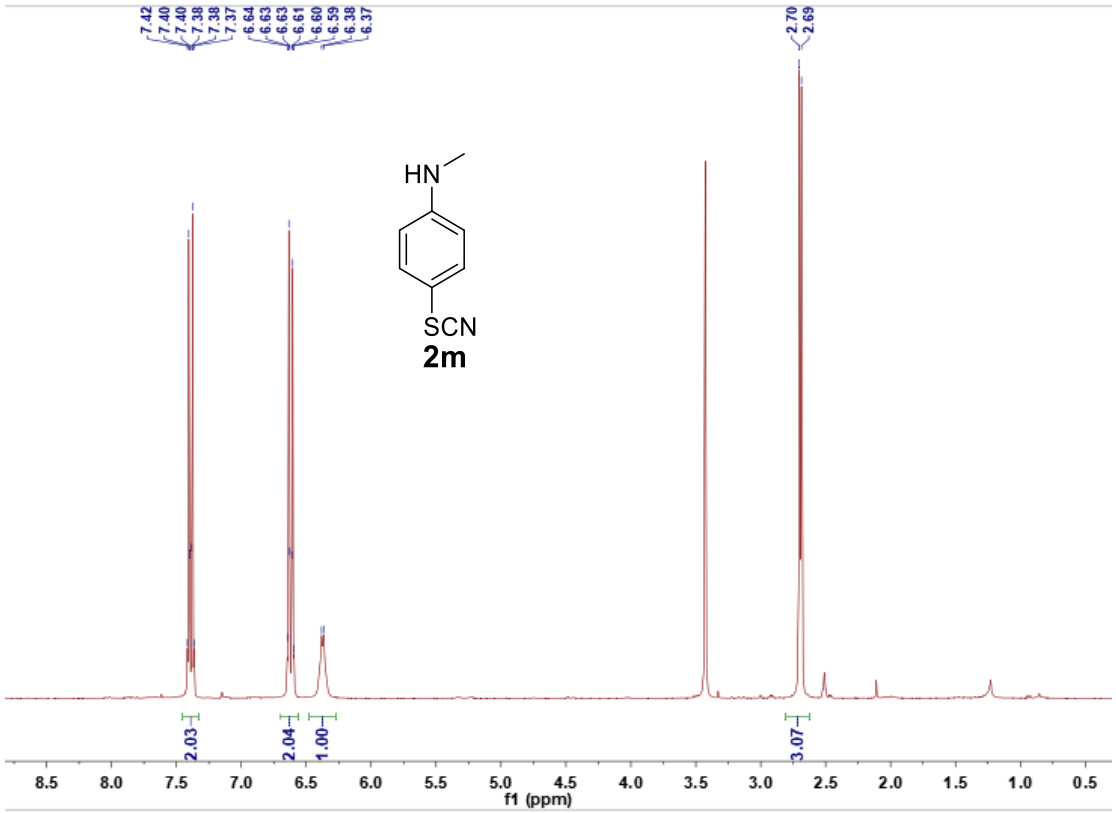

147.11
141.36
127.81
115.51
114.38
113.59
105.47
56.11
40.77
40.49
40.21
39.94
39.66
39.38
39.10
NH2
OMe
SCN
2j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

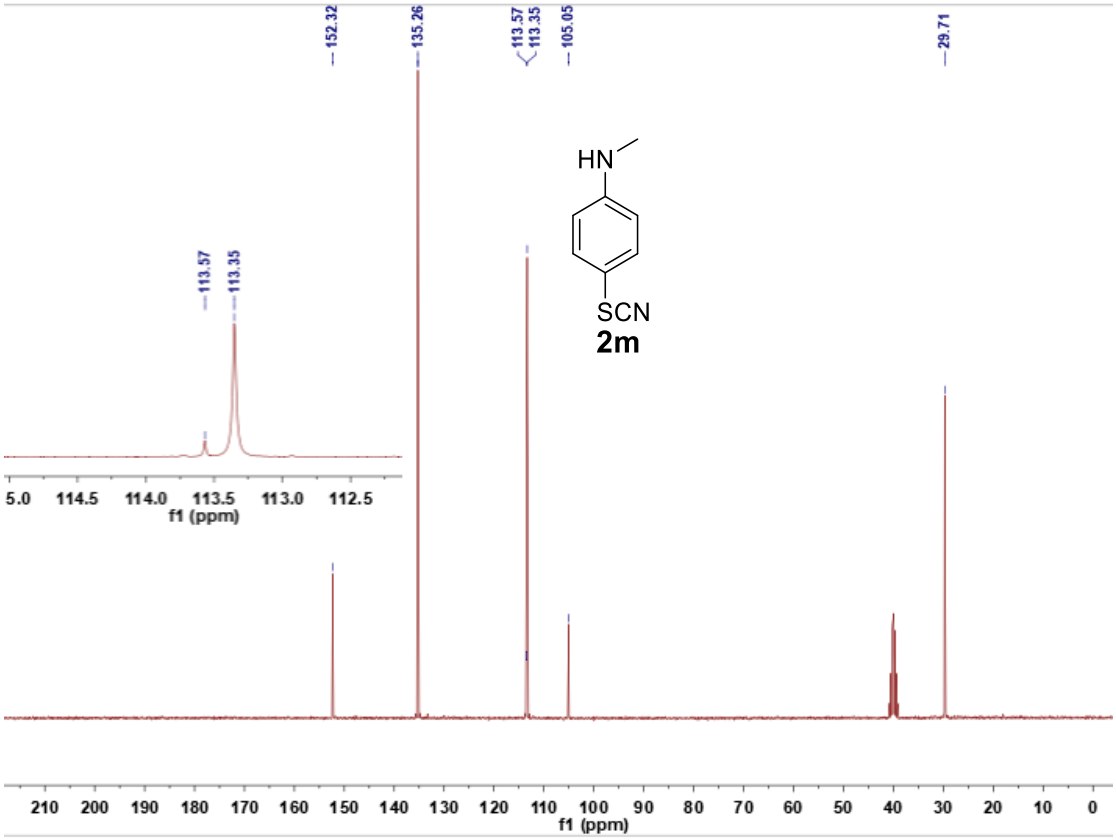

7.31
7.28
6.59
6.58
6.58
6.58
6.55
6.50
6.50
6.48
6.47
5.72
3.44
2.50
2.35
NH2
SCN
2k
1.00
0.98
0.99
1.91
2.91
f1 (ppm)

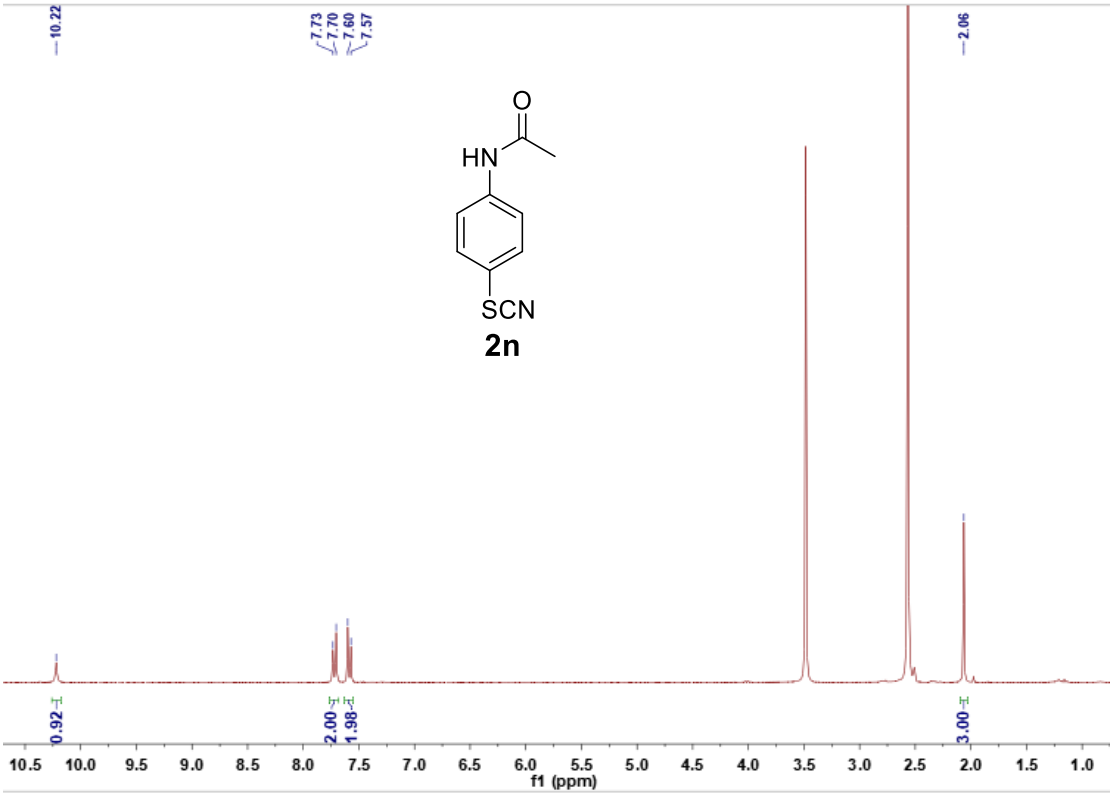

149.96
135.54
132.82
123.40
115.35
113.63
105.32
17.64
NH2
SCN
2k
f1 (ppm)

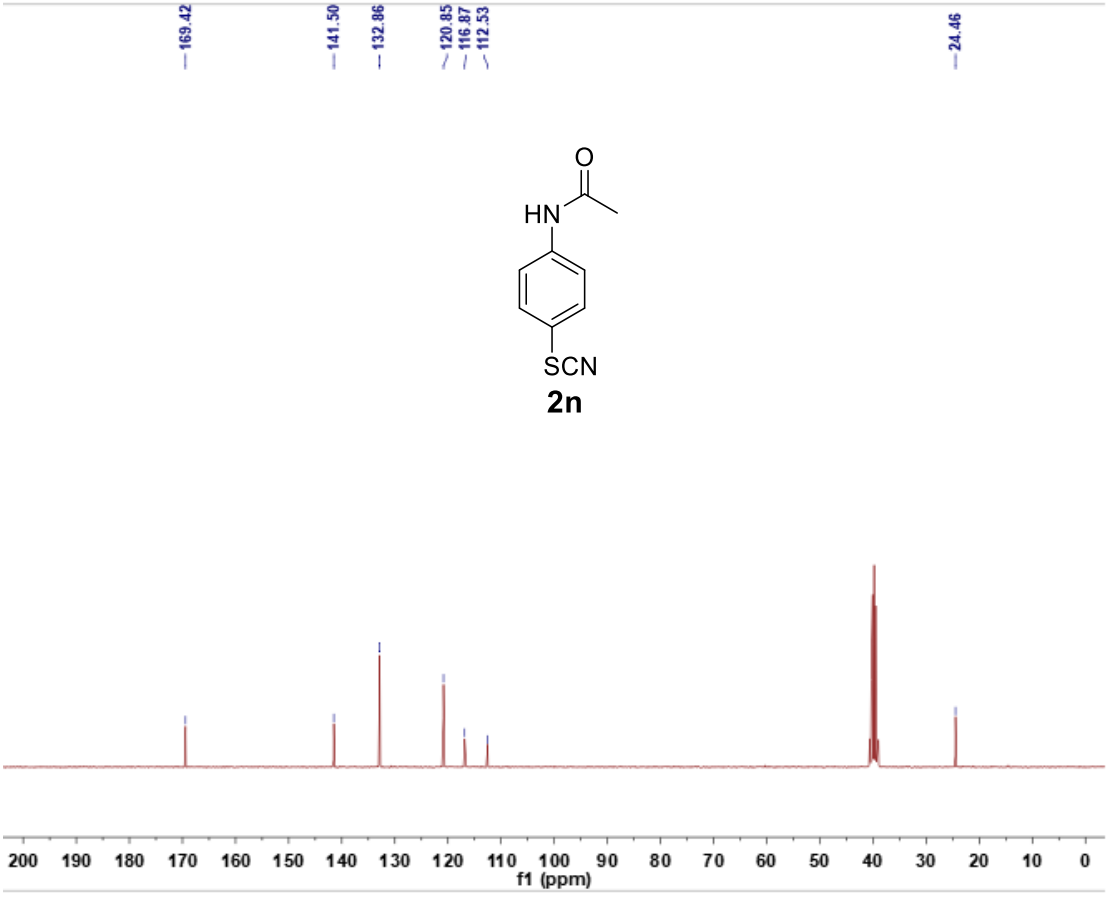

7.15
5.23
3.40
2.50
2.11
NH2
SCN
2l
2.00
1.90
6.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

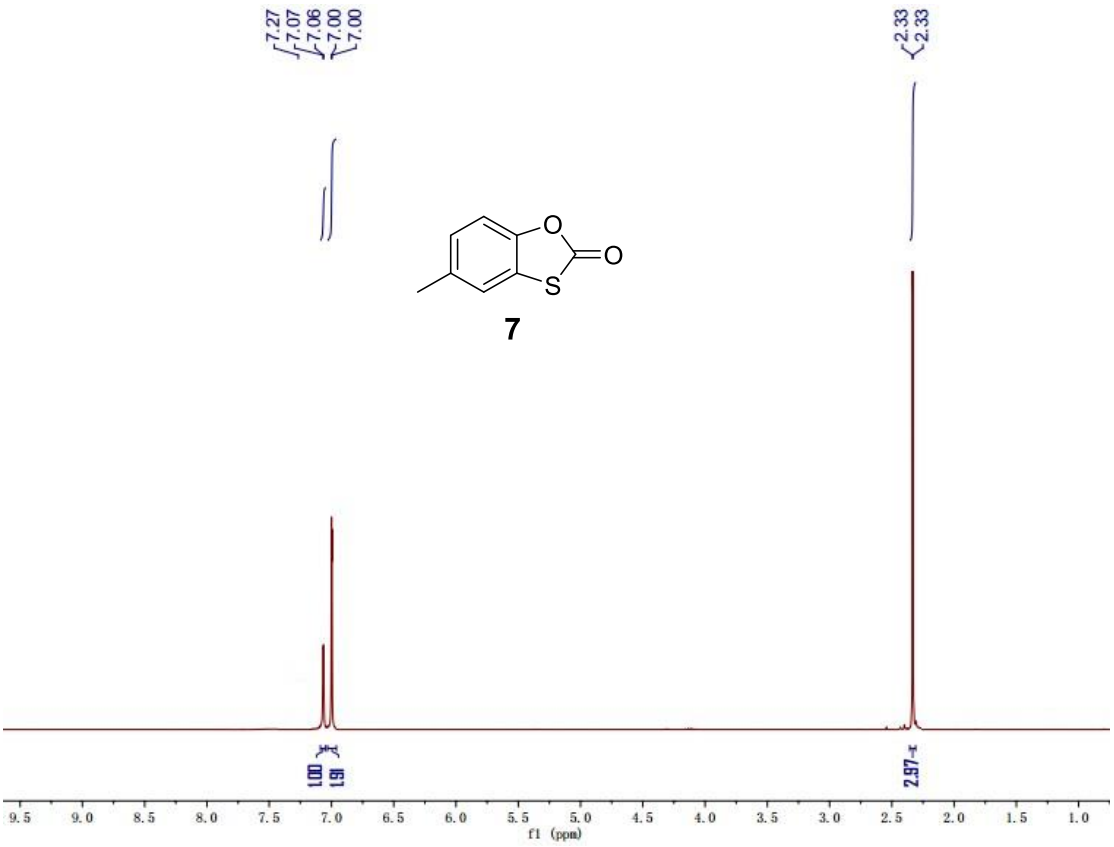

147.86
133.47
122.89
113.45
105.29
18.07
NH2
SCN
2l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

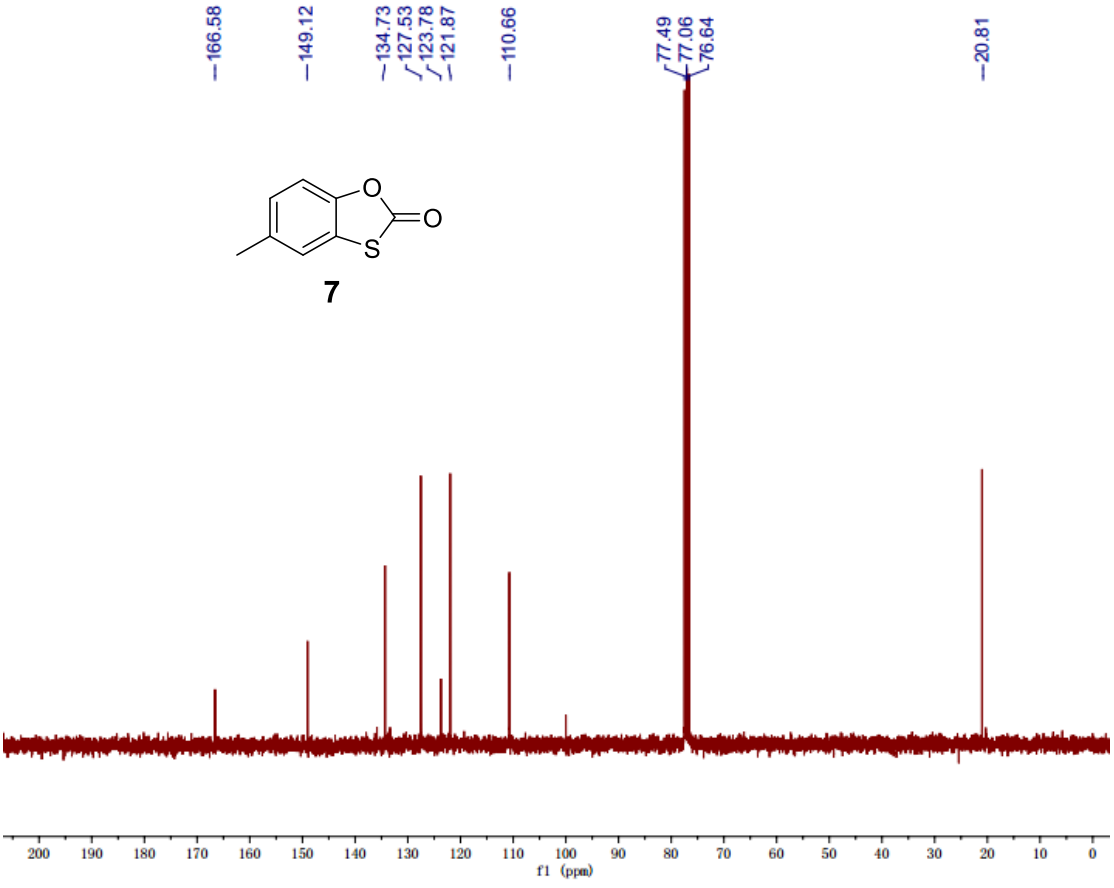

7.42
7.40
7.40
7.38
7.38
7.37
6.64
6.63
6.63
6.61
6.60
6.59
6.38
6.37
2.70
2.69
HN
SCN
2m
2.03
2.04
1.00
3.07
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

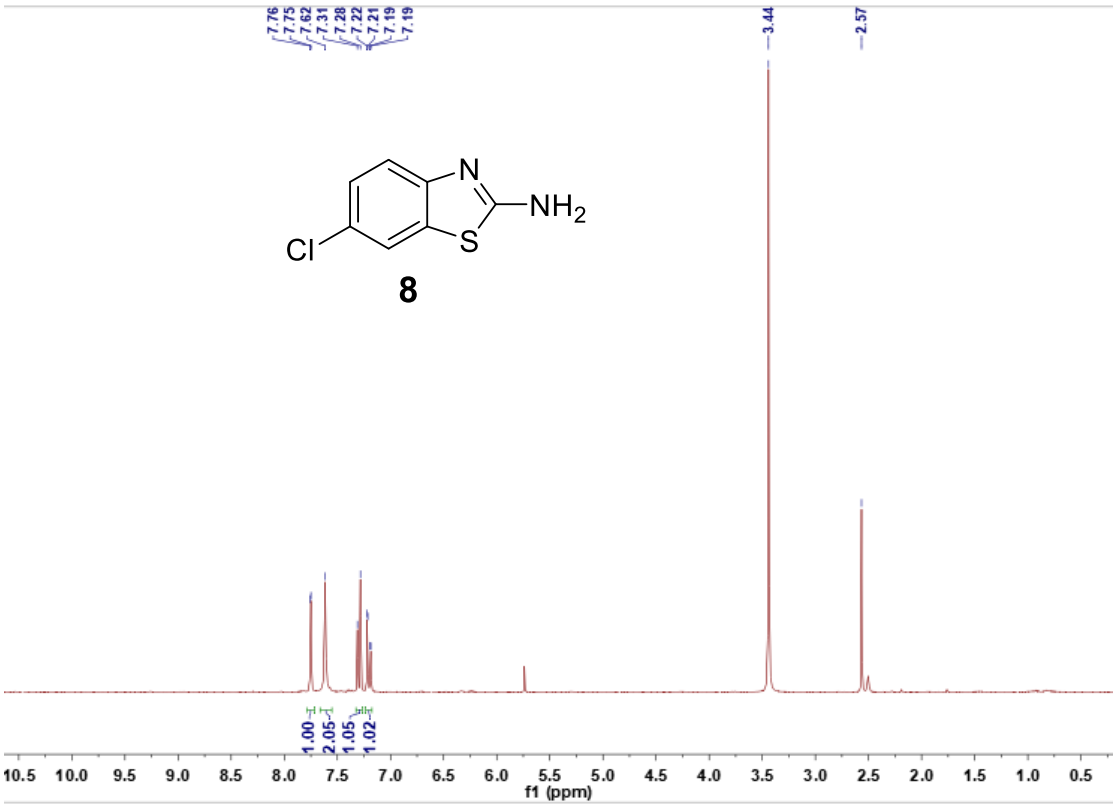

152.32
135.26
113.57
113.35
105.05
29.71
HN
SCN
2m
113.57
113.35
5.0 114.5 114.0 113.5 113.0 112.5
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

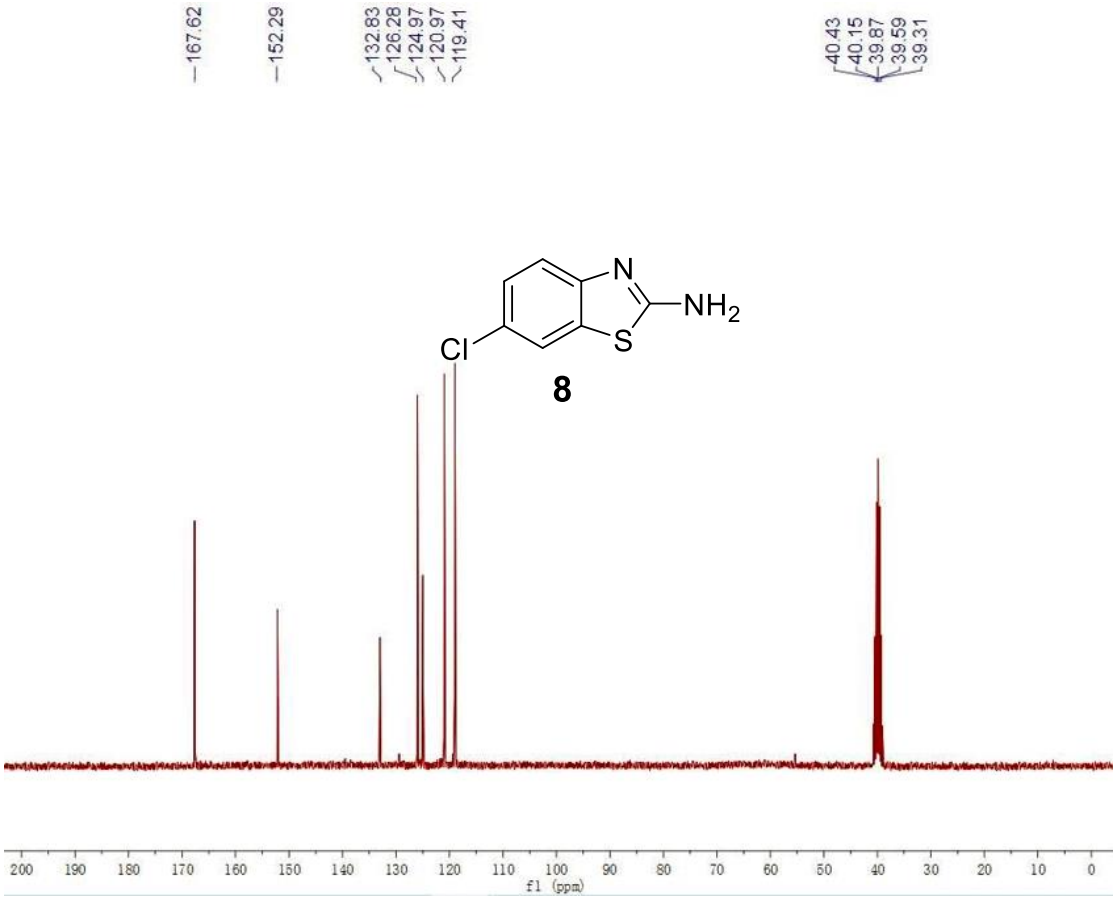

10.22
7.73
7.70
7.60
7.57
2.06
O
HN
SCN
2n
0.92
2.00
1.98
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

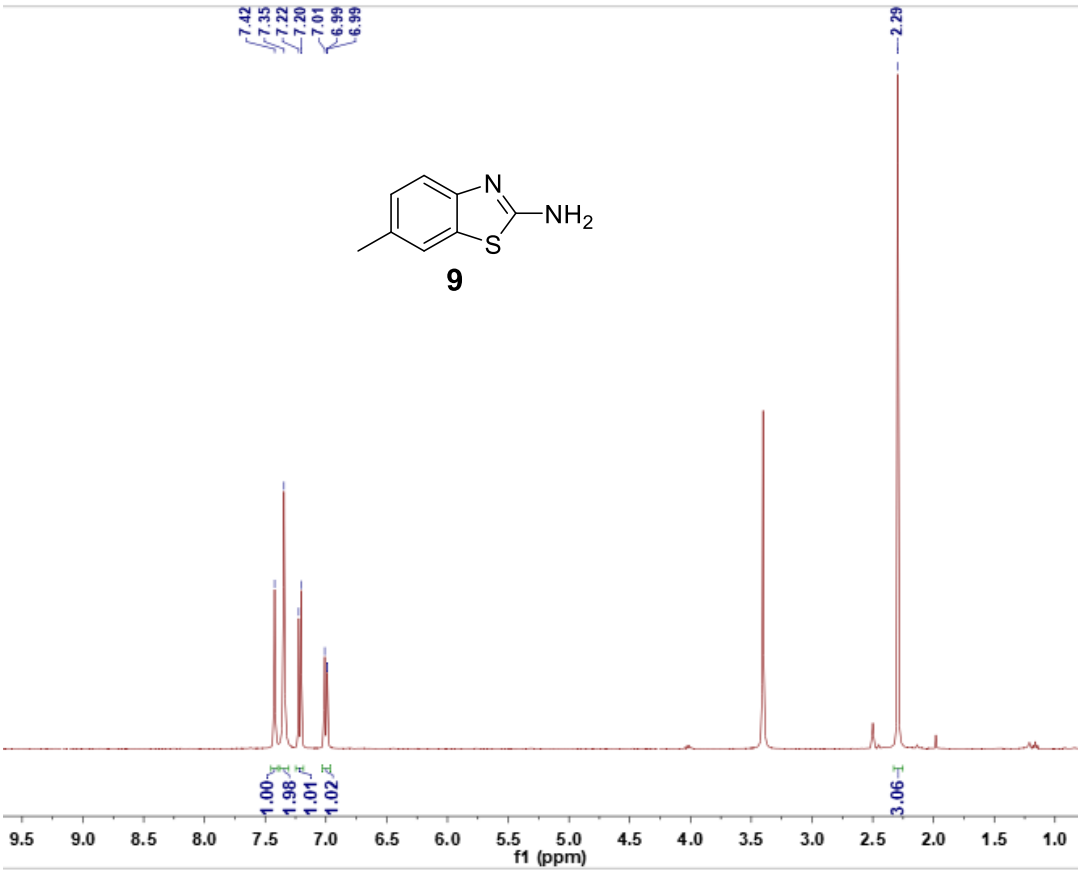

169.42
141.50
132.86
120.85
116.87
112.53
24.46
O
HN
SCN
2n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

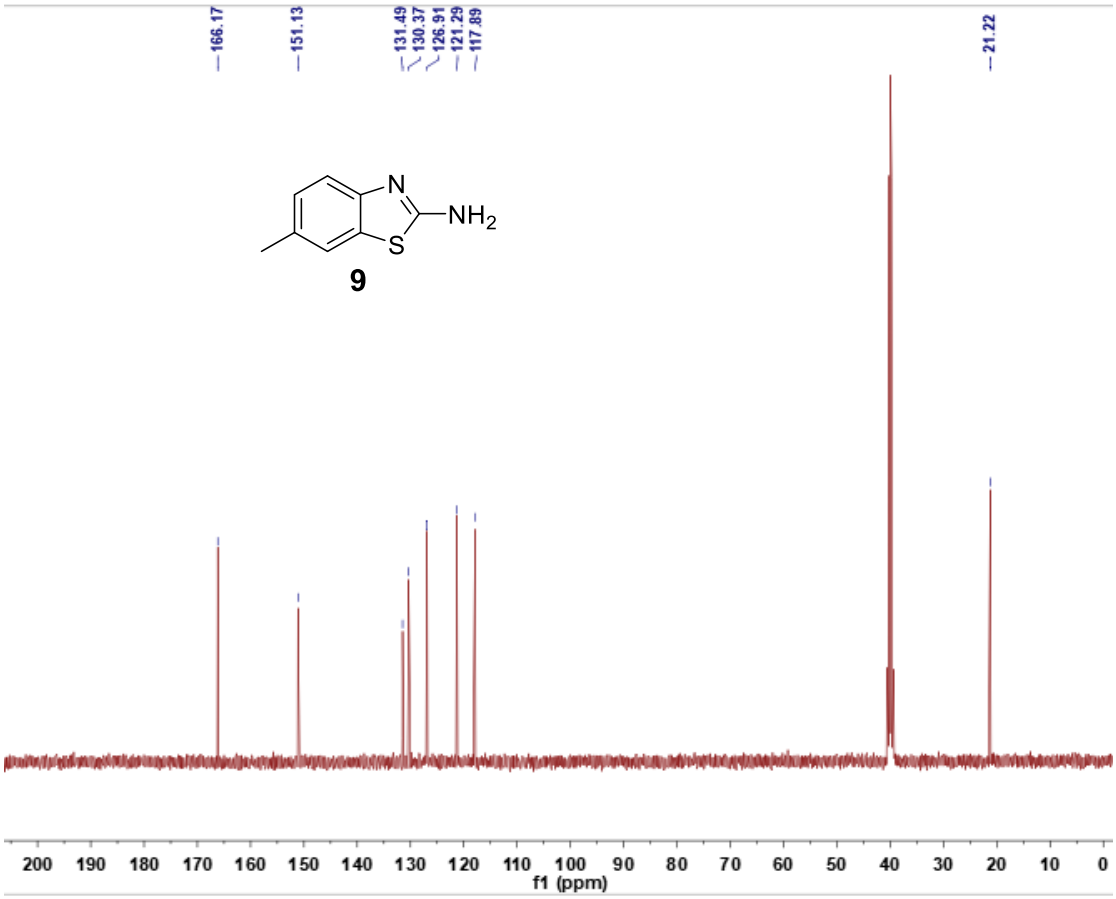

7.27
7.07
7.06
7.00
7.00
2.33
2.33
O
O
S
7
1.00
1.61
2.97
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

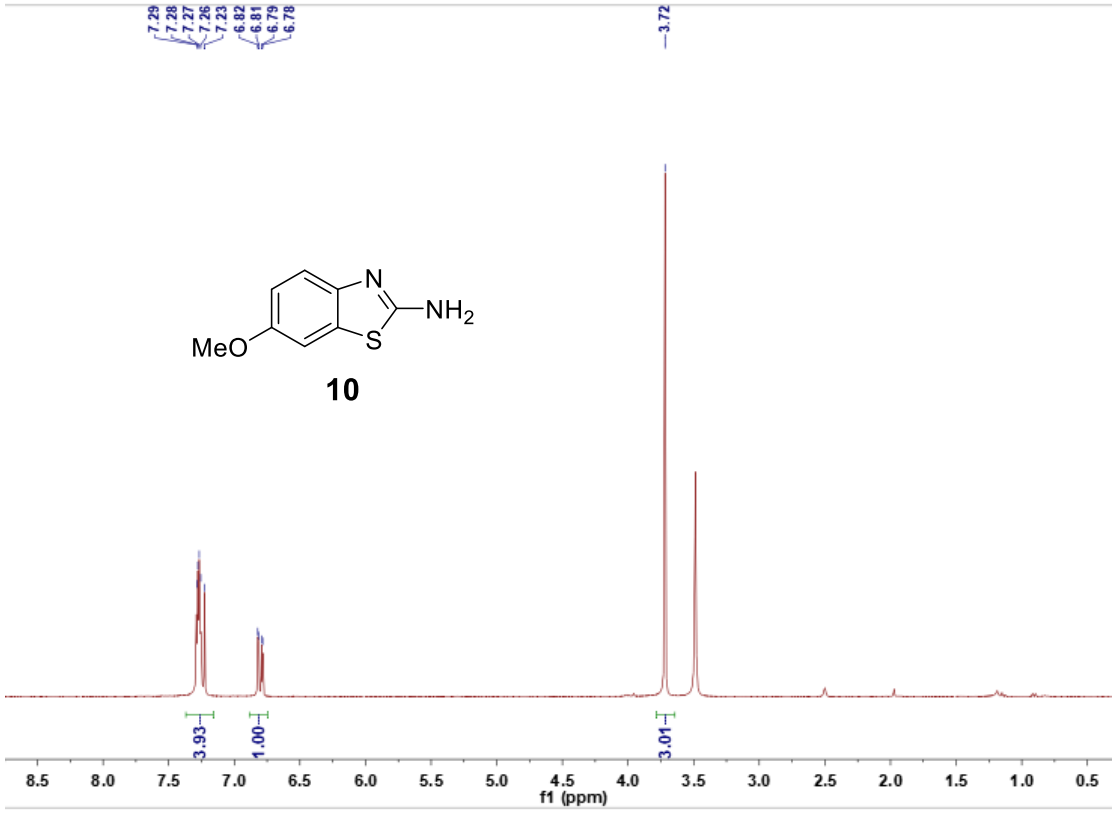

166.58
149.12
134.73
127.53
123.78
121.87
110.66
77.49
77.06
76.64
20.81
O
O
S
7
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

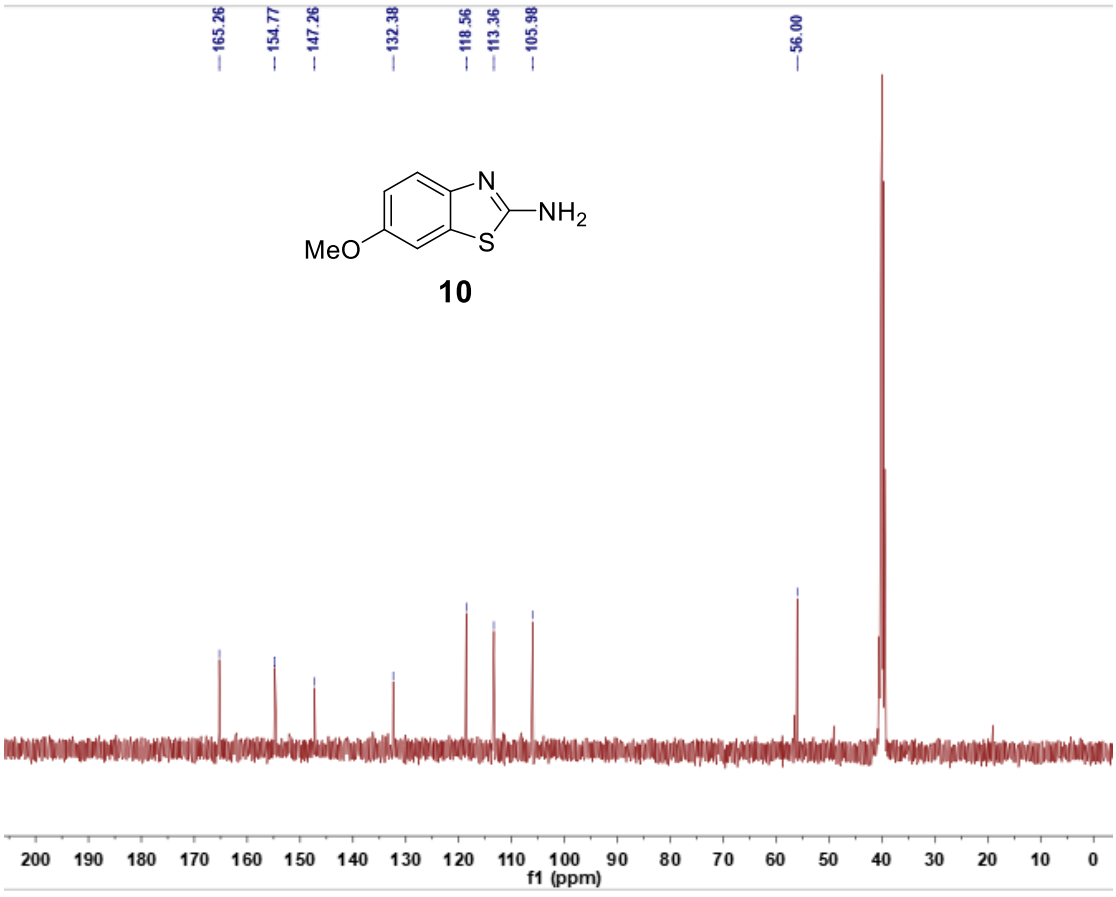

7.76
7.75
7.62
7.31
7.28
7.22
7.21
7.19
7.19
3.44
2.57
N
NH2
Cl
S
8
1.00
2.05
1.05
1.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


167.62
152.29
132.83
126.28
124.97
120.97
119.41
40.43
40.15
39.87
39.59
39.31
N
NH2
Cl
S
8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.42
7.35
7.22
7.20
7.01
6.99
6.99
2.29
N
NH2
S
9
1.00
1.98
1.01
1.02
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
f1 (ppm)


166.17
151.13
131.49
130.37
126.91
121.29
117.89
21.22
N
NH2
S
9
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.29
7.28
7.27
7.26
7.23
6.82
6.81
6.79
6.78
3.72
N
NH2
MeO
S
10
3.93
1.00
3.01
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
f1 (ppm)


165.26
154.77
147.26
132.38
118.56
113.36
105.98
56.00
N
NH2
MeO
S
10
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)