Supporting Information for

Post-sulfonated One-pot Synthesized Magnetic Cellulose Nanocomposite for Knoevenagel and Thorpe-Ziegler Reactions

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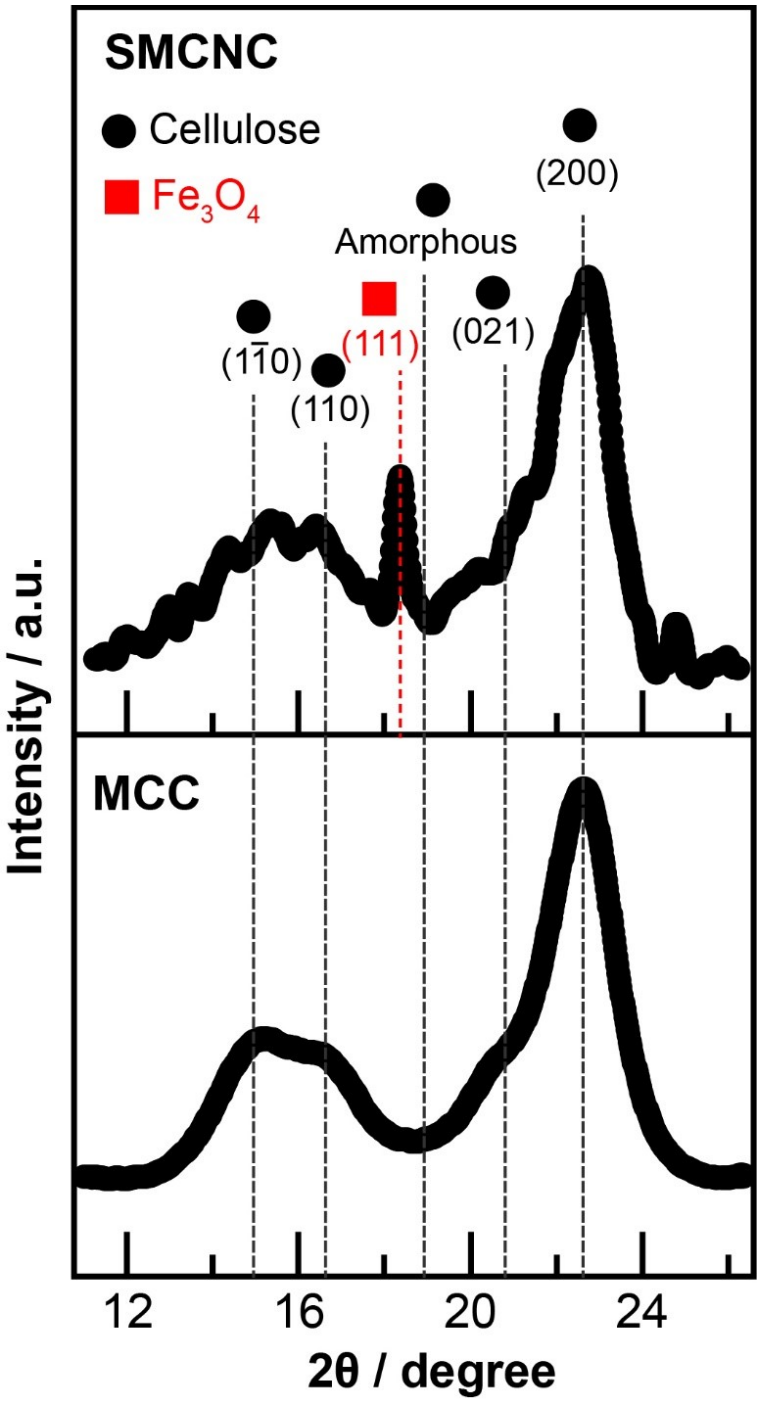


Fig. S1. Deconvoluted XRD spectra of MCC and SMCNC in the range of 11° to 26°.

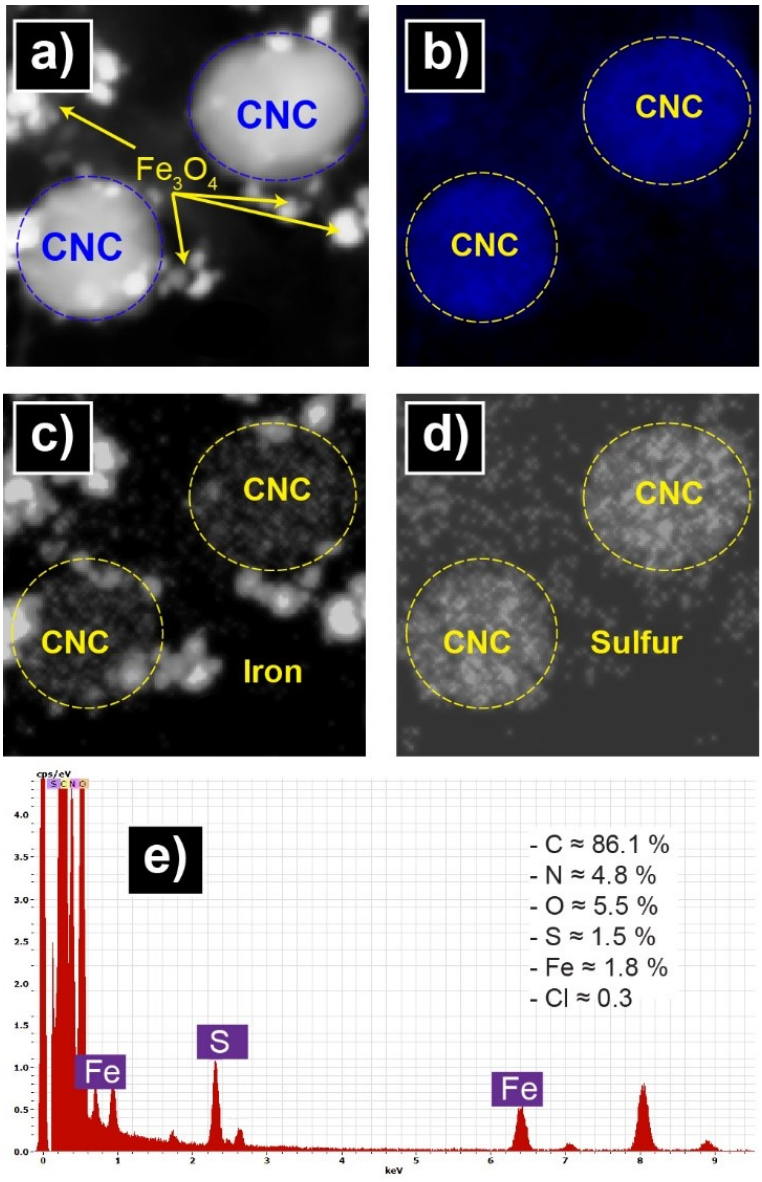


Fig. S2. (a) High-angle annular dark-field (HAADF) image, EDX-mapping of (b) carbon, (c) iron, and (d) sulfur, and (e) EDX-spectrum of SMCNC.

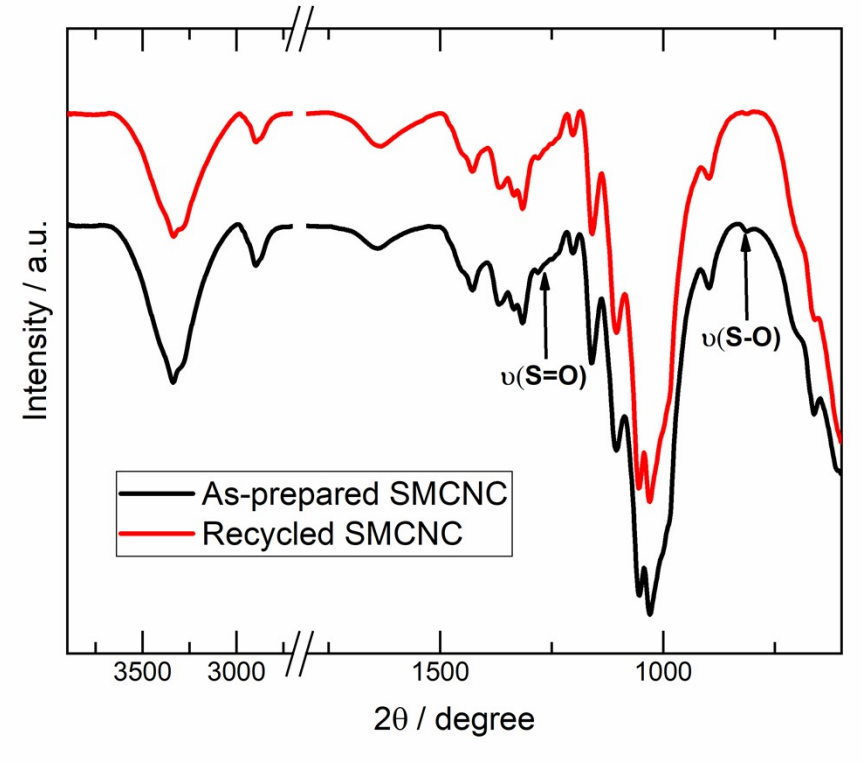


Fig. S3. FTIR spectrum of the as-prepared and recycled SMCNC samples.

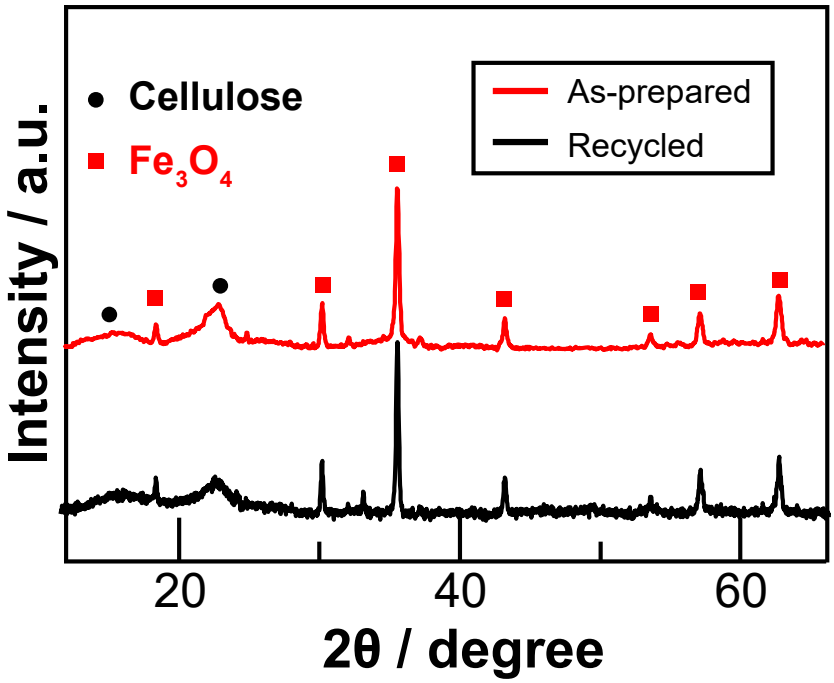


Fig. S4. XRD spectrum of the as prepared and recycled SMCNC.

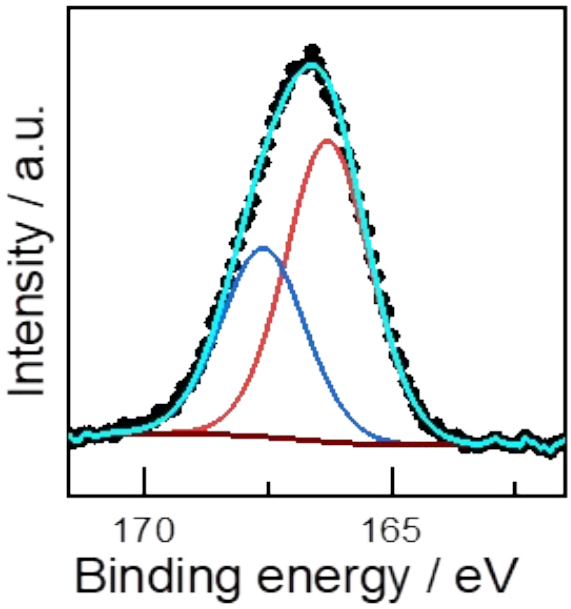


Fig. S5. XPS S2p spectrum of recycled SMCNC.

General method for Knoevenagel condensation

In a representative experimental procedure, benzaldehyde **1a** (0.21 gm, 2 mmol) was transferred into a 25 mL reaction flask charged with malononitrile **2a** (0.20 gm, 3 mmol), 5 mL of the water, SMCNC (10 % wt.) were mixed. The reaction was performed under stirring at ambient temperature for the mentioned time. The reaction completion was followed using TLC. After the reaction completion, the SMCNC was collected using a Neodymium magnet.

Table S1. Gutmann's AN, DN, dipole moment, and dielectric constant of different solvents with the isolated yield (%).

Solvent	AN	DN	Dipole moment	Dielectric constant	yield, (%)
H_2O	54.8	18.0	1.87	80.1	97
EtOH	37.9	19.3	3.69	24.3	95
DMF	16.0	26.6	3.82	38.2	90
Toluene	0	0.1	0.31	2.38	50
Hexane	0	0	0.08	1.88	60

Table S2. The melting point of different synthesized compounds: Reported versus obtained.

Compound	Melting point (°C)	
	Obtained	**Reported**
3aa	81-83	82-84[1]
3ba	138-140	137 - 138[3]
3ca	140-142	138 - 140[5]
3da	163-164	162 - 164[7]
3ea	113-115	114-115[9]
3fa	138-140	137 - 139[10]
3ga	80-82	81 - 82[11]
3ha	130-132	131 - 133[12]
3ia	167-169	168-169[14]
3ja	124-126	125 - 127[16]
3ka	165-166	164 - 166[18]
3la	220-222	221 - 223[19]
3ma	64-66	63-65[21]

Compound	Melting point (°C)	
	Obtained	
3ab	47-48	47-48[2]
3bb	167-168	168-169[4]
3cb	96-97	97-98[6]
3db	83-85	89-90[8]
3eb	159-160	160-161[8]
3fb	91-92	89-91[4]
3gb	73-74	70-72[2]
3hb	54-55	53-54[13]
3ib	153-154	152-153[15]
3jb	112-113	112−114[17]
3kb	66-67	66-67[15]
3lb	187-189	188-189[20]

Analytical Data for Some Knoevenagel Products

2-(2-Nitrobenzylidene)malononitrile **(3ca)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 0.7 Hz, 1H, C<u>H</u>=C), 8.38 (dd, *J* = 8.5, 1.3 Hz, 1H, Ar-H), 7.94 – 7.88 (m, 1H, Ar-H), 7.86 – 7.78 (m, 2H, Ar-H).

2-(4-Chlorobenzylidene)malononitrile **(3da)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.73 (s, 1H, C<u>H</u>=C), 7.51 (d, *J* = 8.5 Hz, 2H, Ar-H).

2-(3-Chlorobenzylidene)malononitrile **(3ea)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 1H, C<u>H</u>=C), 8.20 (dt, *J* = 8.1, 0.9 Hz, 1H, Ar-H), 7.60 – 7.54 (m, 2H, Ar-H), 7.51 – 7.41 (m, 1H, Ar-H).

2-(4-Methylbenzylidene)malononitrile **(3fa)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.79 (m, 2H, Ar-H), 7.74 (s, 1H, CH=C), 7.36 (d, *J* = 8.0 Hz, 2H, Ar-H), 2.48 (s, 3H, CH_3).

2-(2,4,5-Trimethoxybenzylidene)malononitrile **(3ia)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H, C<u>H</u>=C), 7.82 (s, 1H, Ar-H), 6.45 (s, 1H, Ar-H), 3.99 (s, 3H, CH_3), 3.91 (s, 3H, CH_3), 3.87 (s, 3H, CH_3).

2-(3-Indolyl) malononitrile **(3la)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H, NH), 7.98 (s, 1H, C<u>H</u>=C), 7.79 (d, *J* = 8.2 Hz, 1H, Ar-H), 7.63 – 7.56 (m, 1H, CH-pyrrole), 7.53 (d, *J* = 8.5 Hz, 1H, Ar-H), 7.36 (dd, *J* = 12.7, 5.4 Hz, 2H, Ar-H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.63, 138.52, 130.24, 126.72, 125.17, 124.78, 122.81, 120.57, 114.65, 113.72, 112.61.

Ethyl (Z)-2-cyano-3-(4-nitrophenyl)acrylate **(3bb)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, *J* = 8.4 Hz, 2H, Ar-H), 8.32 (s, 1H, C<u>H</u>=C), 8.15 (d, *J* = 8.4 Hz, 2H, Ar-H), 4.44 (q, *J* = 7.2 *Hz*, 2H, CH_2), 1.44 (t, *J* = 7.1 *Hz*, 3H, CH_3).

Ethyl (Z)-2-cyano-3-(2-nitrophenyl)acrylate **(3cb)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (t, *J* = 2.0 Hz, 1H, C<u>H</u>=C), 8.46 – 8.34 (m, 2H, Ar-H), 8.30 (s, 1H, Ar-H), 7.73 (t, *J* = 8.1 Hz, 1H, Ar-H), 4.40 (q, *J* = 7.1 Hz, 2H, CH_2), 1.40 (t, *J* = 7.2 Hz, 3H, CH_3).

Ethyl (Z)-2-cyano-3-(p-tolyl)acrylate **(3fb)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H, C<u>H</u>=C), 7.96 – 7.82 (m, 2H, Ar-H), 7.29 (d, *J* = 8.0 Hz, 2H, Ar-H), 4.37 (q, *J* = 7.1 Hz, 2H, $CH_3C\underline{H}_2$), 2.42 (s, 3H, CH_3), 1.39 (t, *J* = 7.1 Hz, 3H, $C\underline{H}_3CH_2$).

Ethyl (Z)-2-cyano-3-(2-methoxyphenyl)acrylate **(3gb)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (s, 1H, C<u>H</u>=C), 8.24 (d, *J* = 7.9 Hz, 1H, Ar-H), 7.47 (t, *J* = 7.9 Hz, 1H, Ar-H), 7.08 – 6.86 (m, 2H, Ar-H), 4.34 (q, *J* = 7.1 Hz, 2H, $CH_3C\underline{H}_2$), 3.86 (s, 3H, CH_3), 1.36 (t, *J* = 7.2 Hz, 3H, $C\underline{H}_3CH_2$).

Ethyl-2-cyano-3-(naphthalen-1-yl) acrylate **(3kb)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.21 (s, 1H, C<u>H</u>=C), 8.42 (d, *J* = 7.3 Hz, 1H, Ar-H), 8.14 (t, *J* = 7.9 Hz, 2H, Ar-H), 8.02 (dd, *J* = 8.0, 1.0 Hz, 1H, Ar-H), 7.79 – 7.65 (m, 3H, Ar-H), 4.55 (q, *J* = 7.1 Hz, 2H, $CH_3C\underline{H}_2$), 1.55 (t, *J* = 7.2 Hz, 3H, $C\underline{H}_3CH_2$). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.43, 152.89, 133.60, 133.53, 131.76, 129.25, 128.41, 128.33, 127.91, 126.92, 125.52, 122.95, 115.52, 105.86, 14.31.

Ethyl 2-cyano-3-anthracenylacrylate **(3lb)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.08 (s, 1H, Ar-H), 8.25 (s, 1H, C<u>H</u>=C), 7.80 – 7.75 (m, 2H, Ar-H), 7.60 – 7.54 (m, 2H, Ar-H), 7.49 – 7.41 (m, 4H, Ar-H), 7.34 (t, *J* = 7.4 Hz, 1H, Ar-H), 4.29 (q, *J* = 7.1 Hz, $CH_3C\underline{H}_2$), 1.32 (t, *J* = 7.1 Hz, $C\underline{H}_3CH_2$). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.22,

155.92, 145.80, 138.40, 130.38, 129.22, 128.91, 128.81, 128.70, 128.55, 127.69, 119.47, 116.24, 114.48, 99.37, 61.93, 13.74.

General method for Thorpe-Ziggler condensation

In a typical experimental procedure, dimethylpyridine 4 (0.33 gm, 2 mmol) was transferred to a 25 mL reaction vessel charged with α-halogenated carbonyl compound 5 (2.5 mmol), ethanol (5 mL), and SMCNC (10% wt.) were mixed. The reaction was stirred at reflux temperature in an oil bath for the proper time. The reaction completion was followed using TLC. After the reaction completion, the SMCNC was collected using Neodymium magnet.

Table S3. The melting point of different synthesized compounds: Reported versus obtained.

Compound	Melting point (°C)	
	Obtained	Reported
6a	193-195	192-193[22]
6b	88-90	87-88[24]
6c	131-133	132-133[26]
6d	142-143	140-141[28]
6e	177-178	176-178[30]

Compound	Melting point (°C)	
	Obtained	Reported
7a	235-236	236-237[23]
7b	156-157	154-155[25]
7c	206-208	206-207[27]
7d	203-204	204-205[29]
7e	221-223	219-220[31]

Analytical Data for Some Alkylated and Thorpe Products.

2-((3-Cyano-4,6-dimethylpyridin-2-yl)thio)-N-(4-methoxyphenyl)acetamide **(6f)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.15 (s, 1H, NH), 7.43 – 7.36 (m, 2H, Ar-H), 7.30 (s, 1H, Ar-H), 6.91 – 6.84 (m, 2H, Ar-H), 4.01 (s, 2H, CH_2), 3.81 (s, 3H, OCH_3), 2.65 (s, 3H, CH_3), 2.53 (s, 3H, CH_3). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.64, 161.33, 160.85, 156.36, 153.01, 130.92, 121.41, 121.10, 114.37, 114.15, 105.62, 59.49, 55.46, 38.11, 34.65, 31.21, 24.76, 20.27.

2-((3-Cyano-4,6-dimethylpyridin-2-yl)thio)-N-(naphthalen-1-yl)acetamide **(6g)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.37 (s, 1H, NH), 7.89 (dd, *J* = 28.1, 7.8 Hz, 2H, Ar-H), 7.69 (dd, *J* = 19.8, 8.3 Hz, 2H, Ar-H), 7.49 (td, *J* = 7.6, 4.1 Hz, 2H, Ar-H), 7.42 (t, *J* = 7.3 Hz, 1H, Ar-H), 6.95 (s, 1H, Ar-H), 4.17 (s, 2H, CH_2), 2.56 (s, 3H, CH_3), 2.53 (s, 3H, CH_3). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.83, 159.56, 148.68, 143.98, 134.24, 132.27, 128.71, 128.15, 126.55, 126.34, 126.14, 125.66, 123.49, 122.41, 121.98, 121.21, 97.79, 31.26, 24.34, 20.27.

N-(Benzo[d]thiazol-2-yl)-2-((3-cyano-4,6-dimethylpyridin-2-yl)thio)acetamide **(6h)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 31.2, 8.0 Hz, 2H, Ar-H), 7.48 – 7.45 (m, 1H, Ar-H), 7.36 (dd, *J* = 11.2, 4.0 Hz, 1H, Ar-H), 4.10 (s, 2H, CH_2), 2.74 (s, 3H, CH_3), 2.53 (s, 3H, CH_3). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.36, 161.98, 160.11, 158.00, 153.25, 146.84, 131.43, 126.40, 124.15, 121.51, 121.42, 120.40, 114.05, 105.52, 38.01, 34.26, 31.10, 24.47, 20.22.

2-((3-Cyano-4,6-dimethylpyridin-2-yl)thio)-N-(thiazol-2-yl)acetamide **(6i)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 11.85 (s, 1H, NH), 7.85 (d, *J* = 7.3 Hz, 1H, CH thiazole), 7.44 (d, *J* = 7.6 Hz, 1H, CH thiazole), 7.05 (s, 1H, CH pyridine), 4.07 (s, 2H), 2.86 (s, 3H), 2.56 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.75, 161.94, 160.60, 157.35, 153.43, 149.69, 134.09, 128.63, 127.95, 125.83, 121.59, 114.13, 107.65, 105.66, 34.10, 24.40, 20.33.

3-Amino-N-(4-methoxyphenyl)-4,6-dimethylthieno[2,3-b]pyridine-2-carboxamide **(7f)**

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.44 (m, 2H, Ar-H), 7.11 (s, 1H, NH), 6.92 (dd, *J* = 6.6, 2.3 Hz, 3H, Ar-H+CH pyridine), 6.44 (s, 2H), 3.83 (s, 3H, OCH_3), 2.79 (s, 3H, CH_3), 2.63 (s, 3H, CH_3). **^{13}C NMR** (126 MHz $CDCl_3$) δ 164.13, 156.69, 148.25, 130.52, 122.96, 122.33, 114.19, 55.50, 38.13, 31.22, 24.21, 20.22.

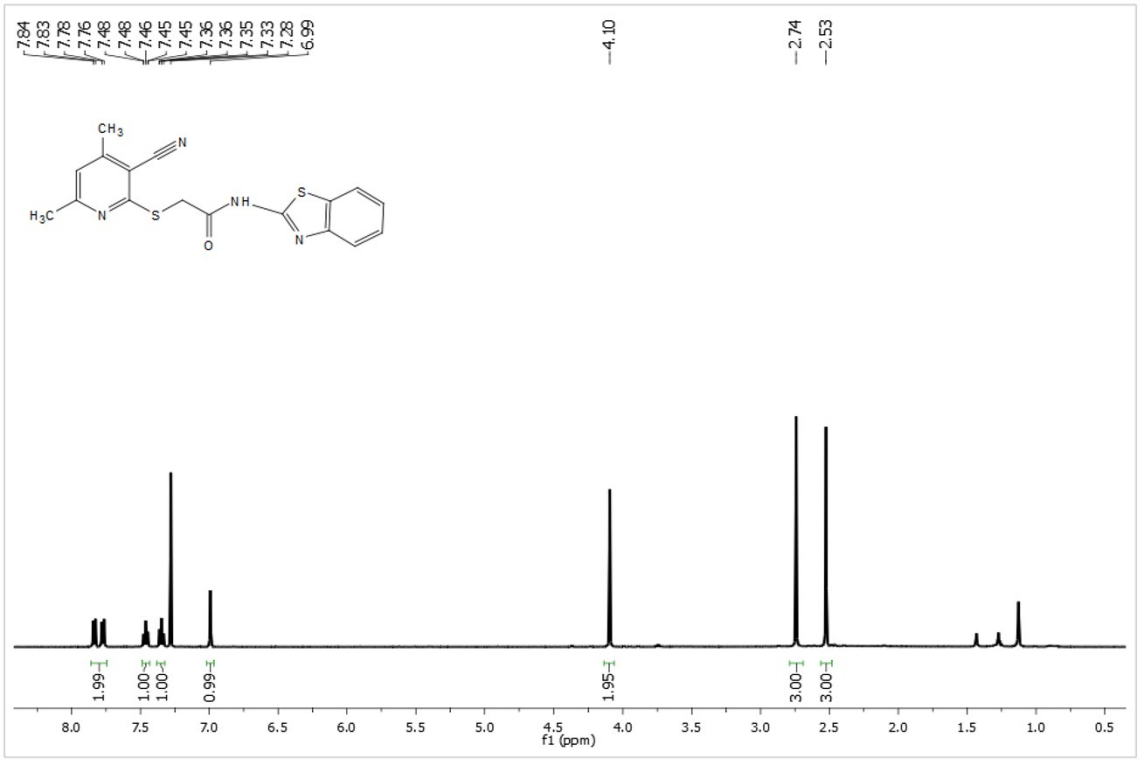
3-Amino-4,6-dimethyl-N-(naphthalen-1-yl)thieno[2,3-b]pyridine-2-carboxamide **(7g)**

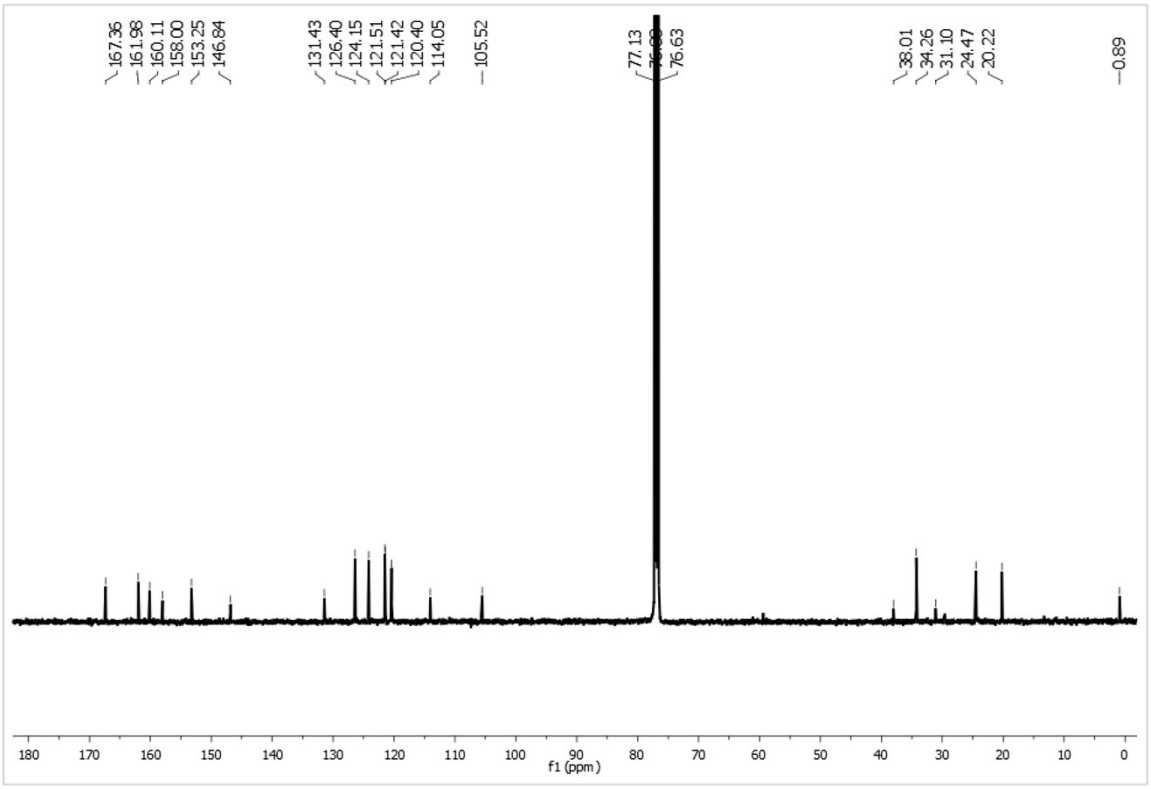
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H, Ar-H), 7.93 – 7.89 (m, 2H, Ar-H+NH), 7.78 (d, *J* = 8.2 Hz, 1H), 7.60 – 7.51 (m, 4H, Ar-H), 6.95 (s, 1H, CH pyridine), 6.49 (s, 2H, NH_2) 2.80 (s, 3H, CH_3), 2.65 (s, 3H, CH_3). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.76, 159.49, 148.62, 143.91, 134.18, 132.20, 128.65, 128.08, 126.48, 126.28, 126.07, 125.59, 123.43, 122.35, 121.92, 121.15, 97.73, 24.28, 20.20.

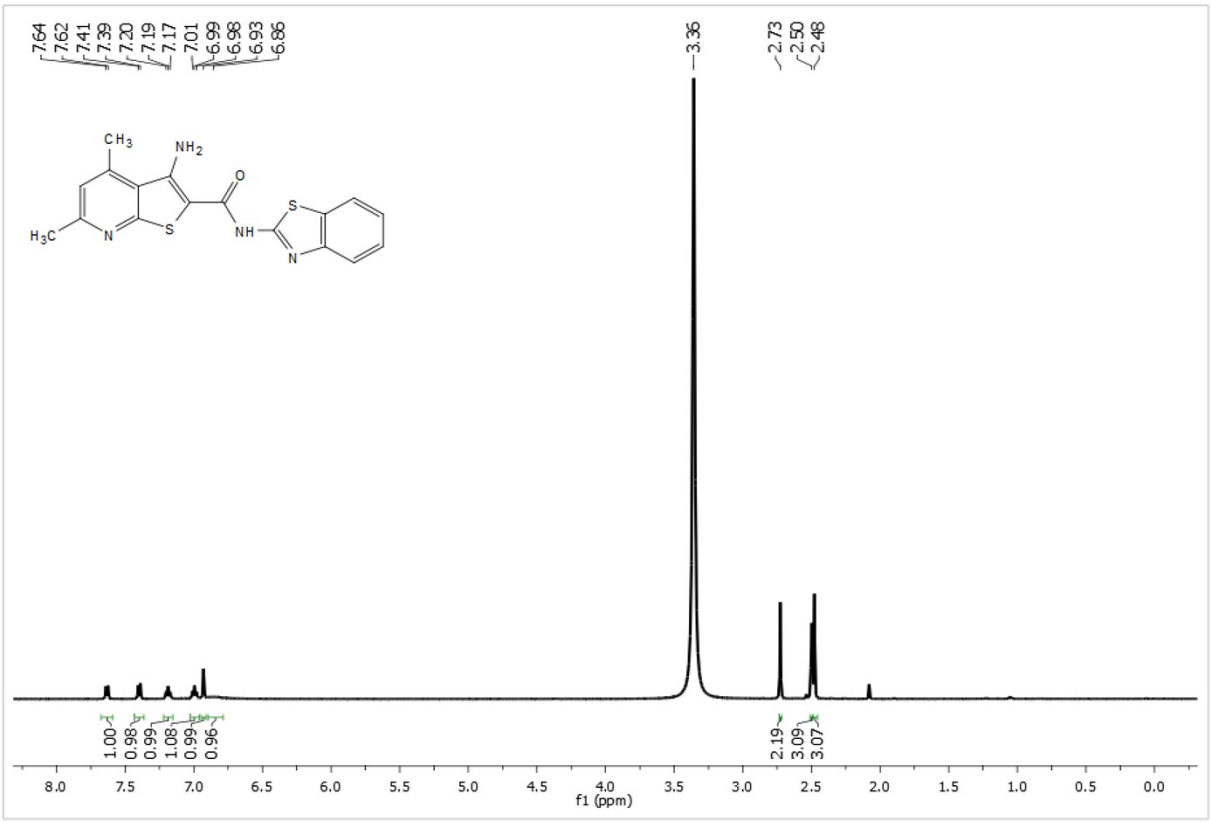
3-Amino-N-(benzo[d]thiazol-2-yl)-4,6-dimethylthieno[2,3-b]pyridine-2-carboxamide **(7h)**

^{1}H NMR (500 MHz, DMSO-d6) δ 7.63 (d, *J* = 7.6 Hz, 1H, Ar-H), 7.40 (d, *J* = 7.9 Hz, 1H, NH), 7.19 (t, *J* = 7.4 Hz, 1H, Ar-H), 6.99 (t, *J* = 7.3 Hz, 1H, Ar-H), 6.93 (s, 1H), 6.86 (s, 1H, CH pyridine), 2.73 (s, 2H, NH_2), 2.50 (s, 3H, CH_3), 2.48 (s, 3H, CH_3). **^{13}C NMR** (126 MHz, DMSO-d6) δ 170.14, 167.36, 159.45, 156.49, 143.60, 132.47, 124.42, 120.91, 120.65, 120.20, 117.53, 17, 23.77, 19.69.

NMR Spectra of the Products



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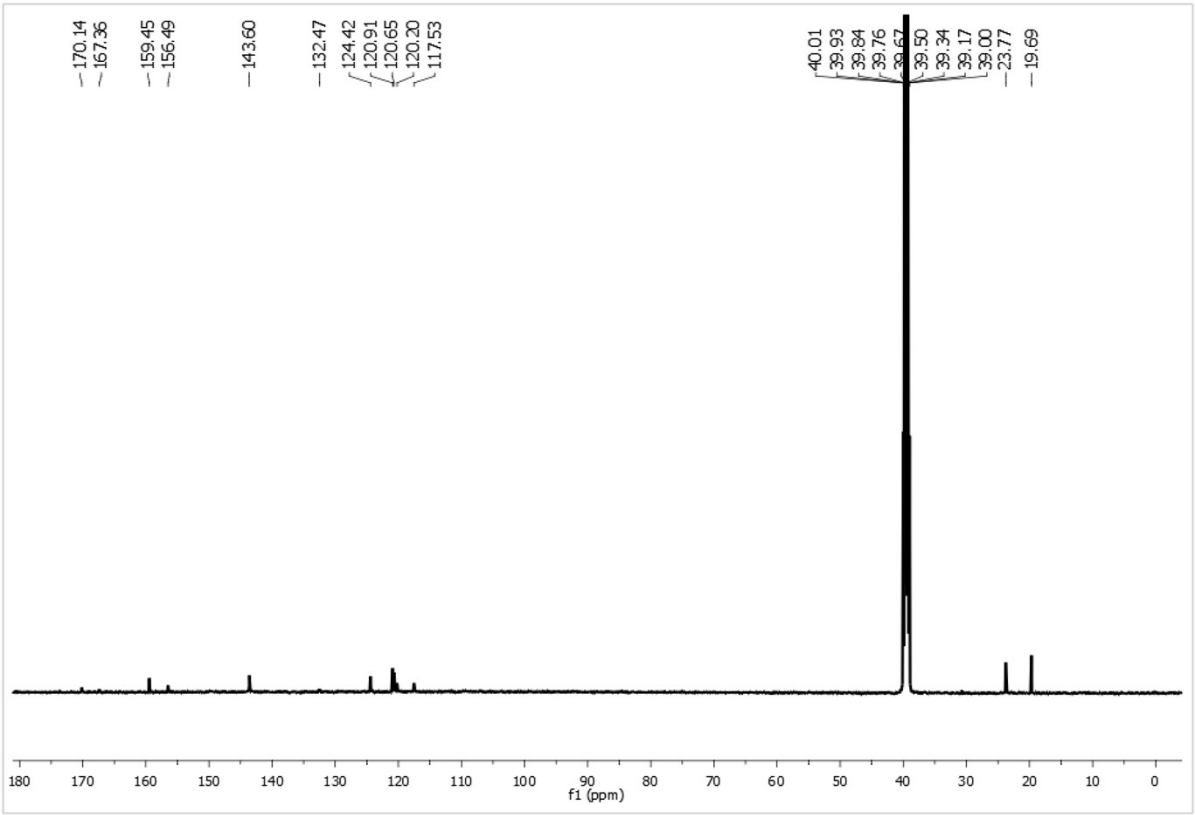
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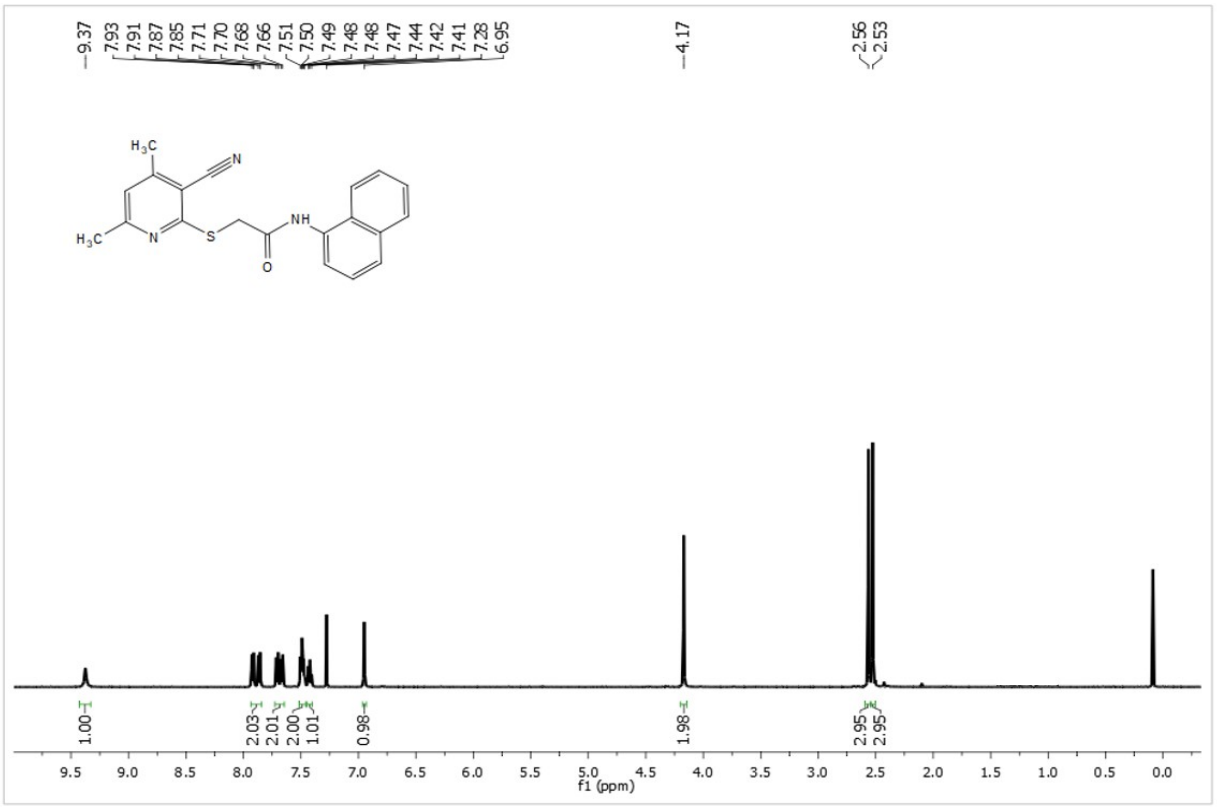
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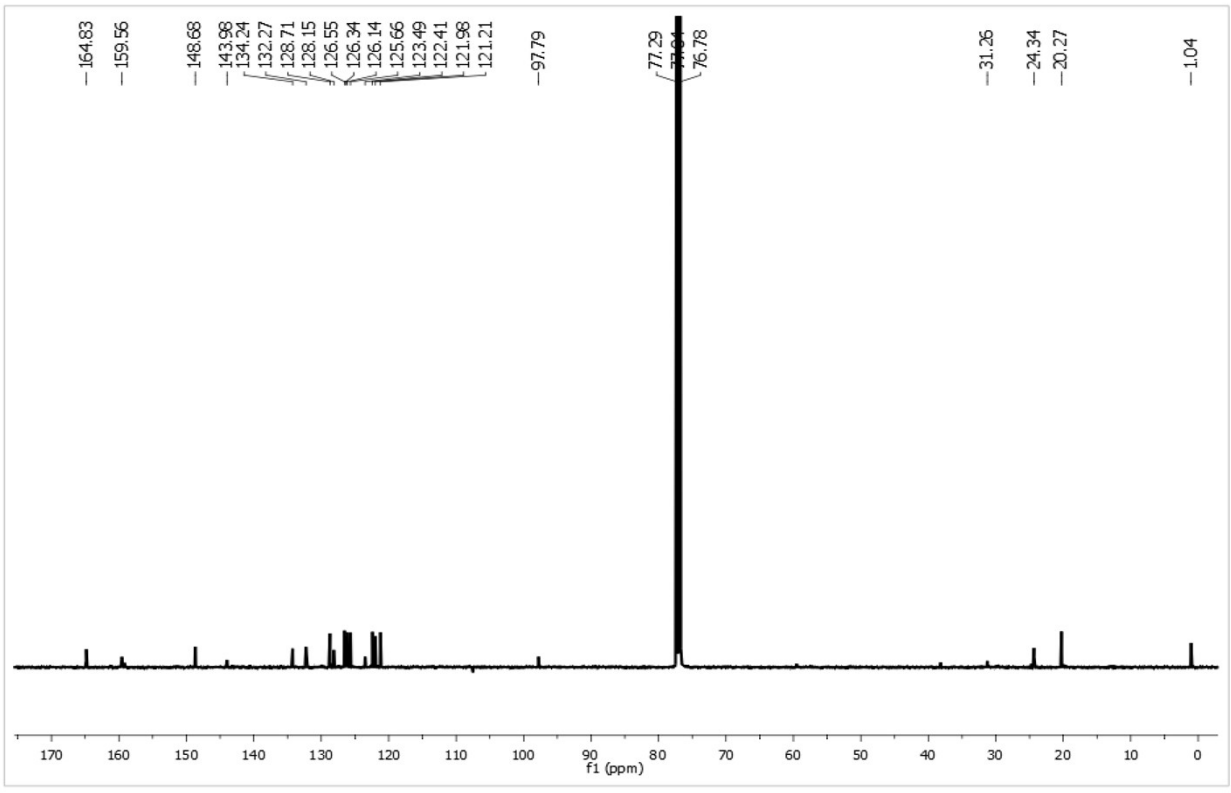
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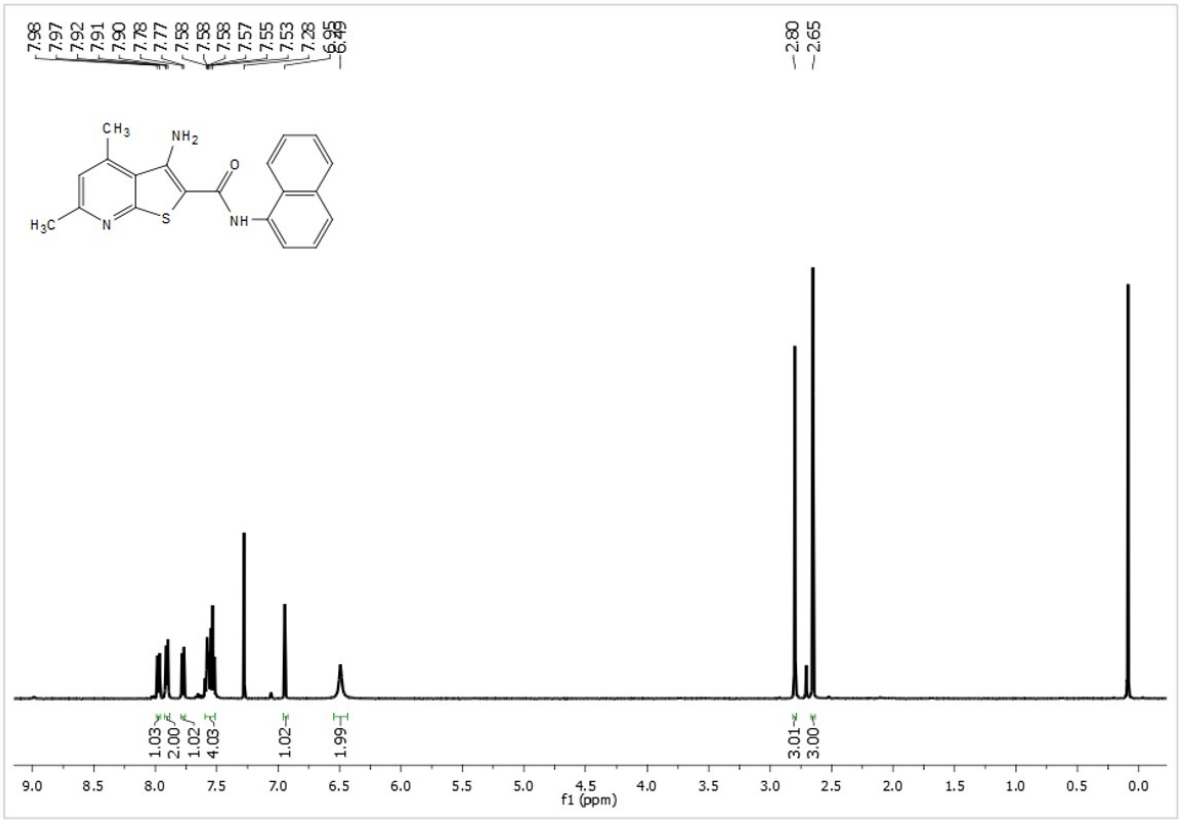
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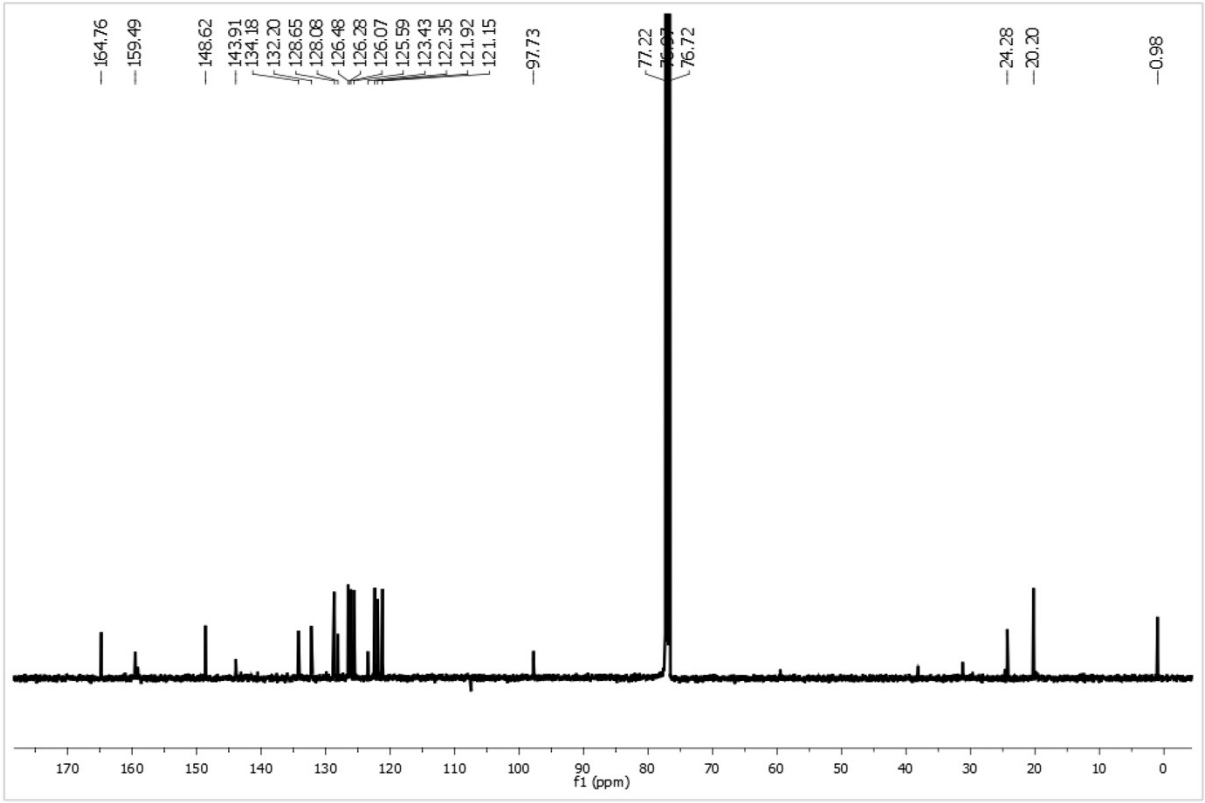
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110
100
90
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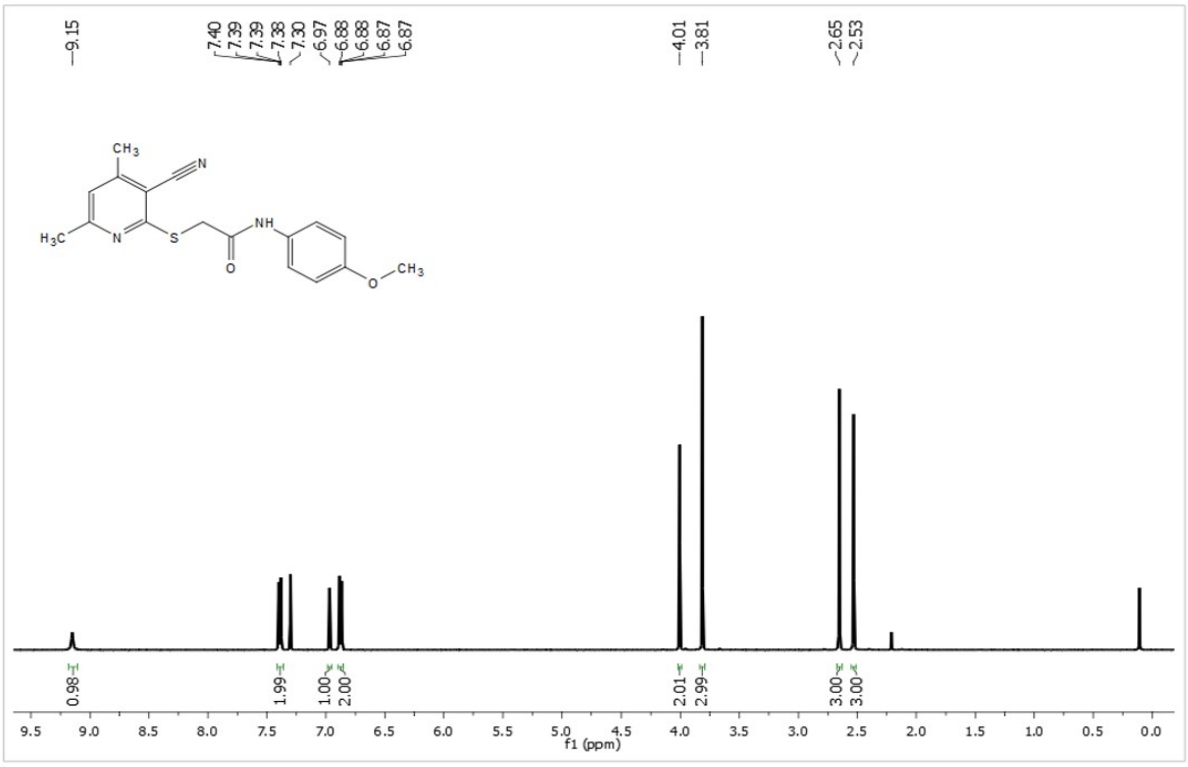
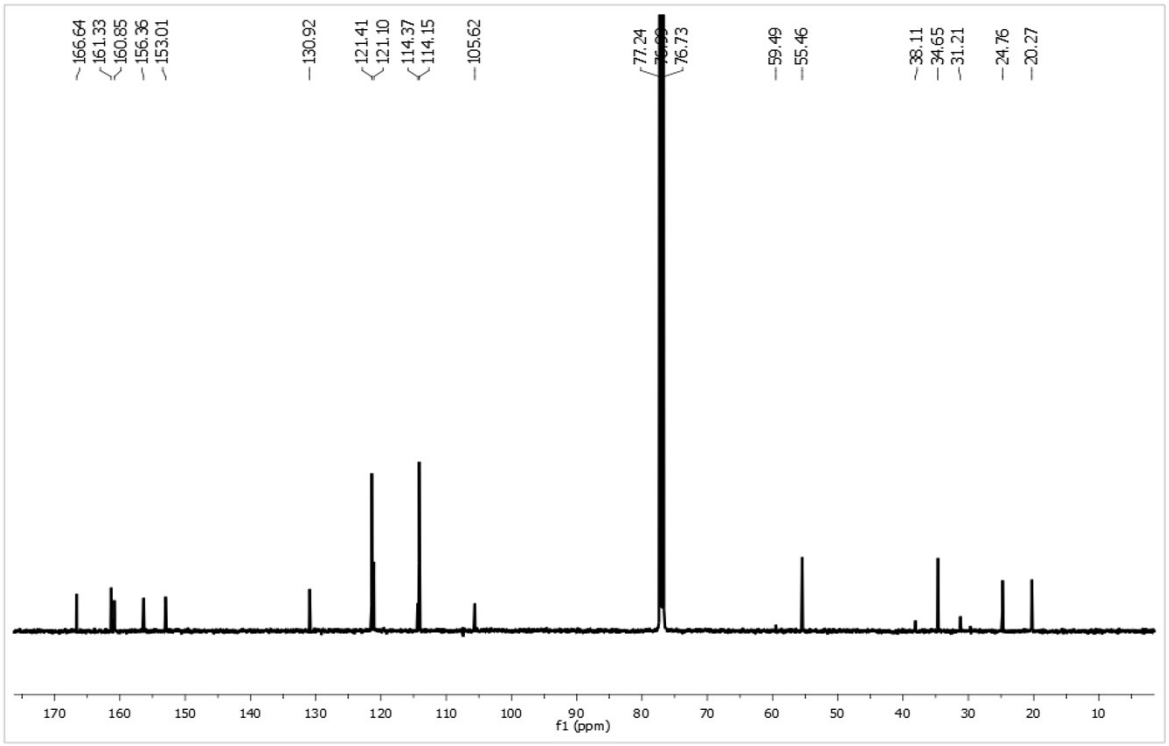
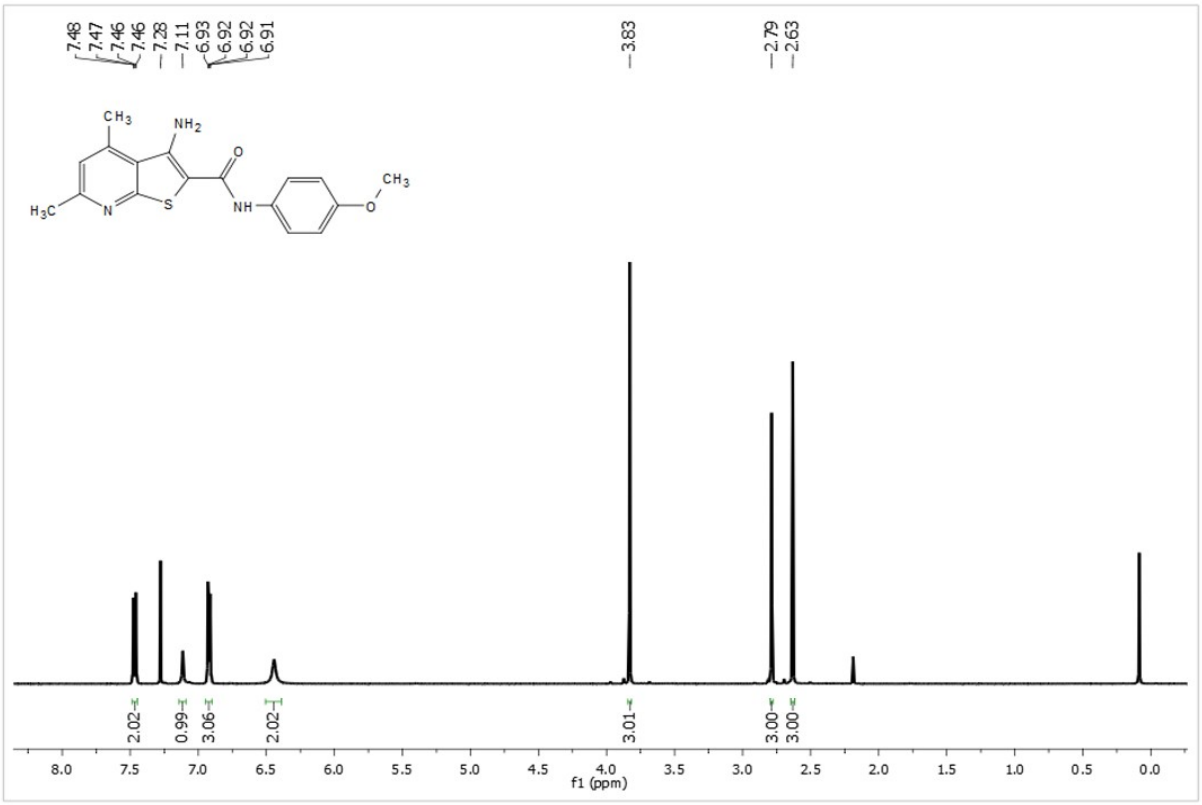
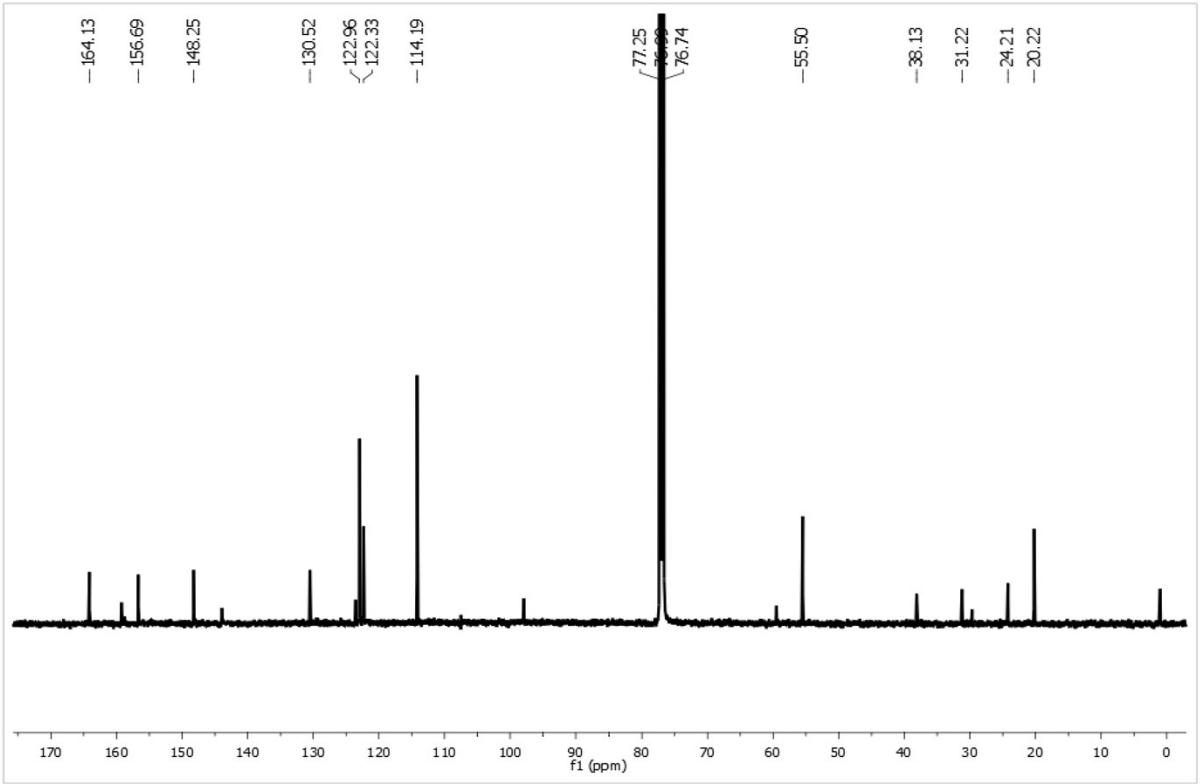
70
60
50
40
30
20
10
0
f1 (ppm)


9.15
7.40
7.39
7.39
7.38
7.30
6.97
6.88
6.88
6.87
6.87
4.01
3.81
2.65
2.53
CH3
N
H3C
N
S
NH
O
O
CH3
0.98
1.99
1.00
2.00
2.01
2.99
3.00
3.00
f1 (ppm)


166.64
161.33
160.85
156.36
153.01
130.92
121.41
121.10
114.37
114.15
105.62
77.24
76.99
76.73
59.49
55.46
38.11
34.65
31.21
24.76
20.27
f1 (ppm)


7.48
7.47
7.46
7.46
7.28
7.11
6.93
6.92
6.92
6.91
3.83
2.79
2.63
CH3
NH2
O
CH3
H3C
N
S
NH
O
2.02
0.99
3.06
2.02
3.01
3.00
3.00
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)


164.13
156.69
148.25
130.52
122.96
122.33
114.19
77.25
76.74
55.50
38.13
31.22
24.21
20.22
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

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