

Supporting Information

Electrosynthesis of Sulfonamides from DMSO and Amines under Mild Conditions

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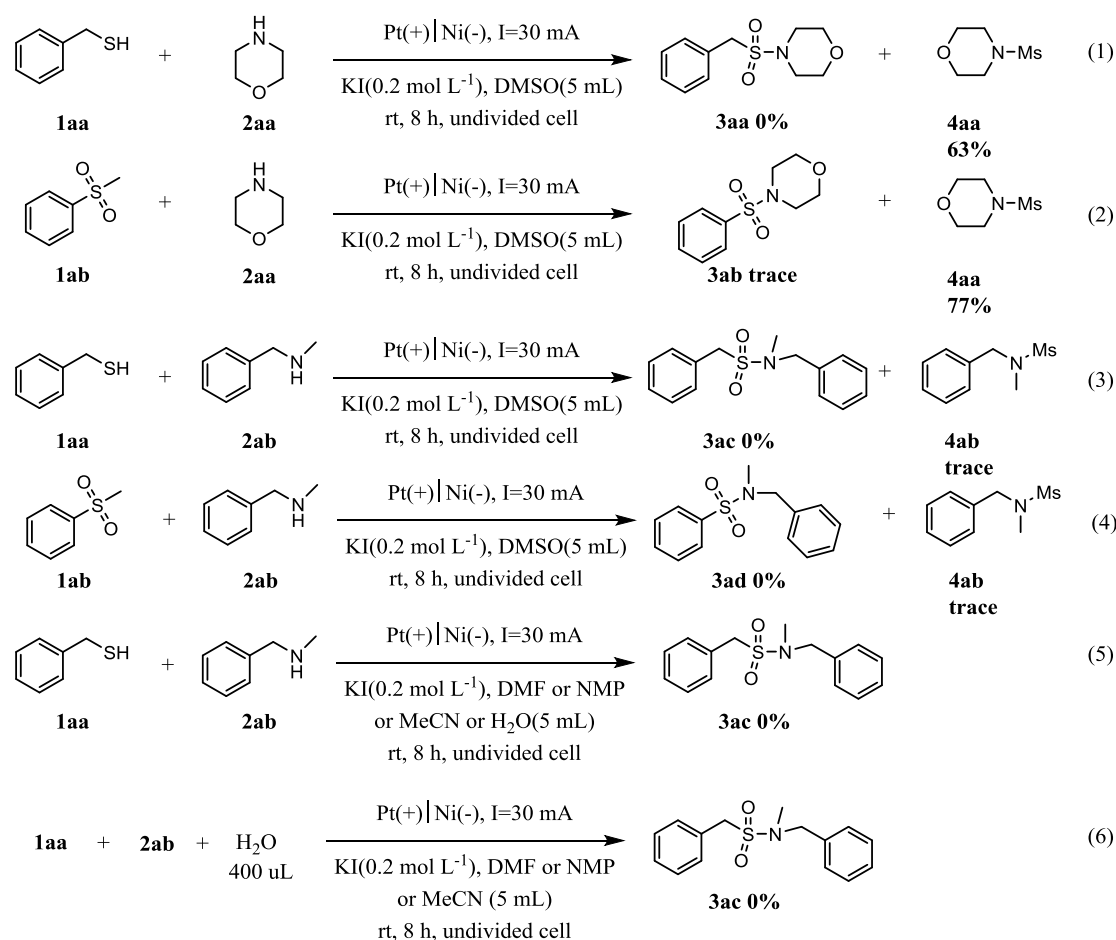
1. General information

Unless otherwise stated, analytical grade commercial reagents and solvents were used as the received. Amines were bought from Alfa Aesar, Acros and Aldrich. Analytical thin layer chromatography (TLC) was performed by using commercially prepared 100–400 mesh silica gel plates (GF254) and visualization was effected at 254 nm. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz NMR spectrometer (400 MHz for ^1H , 100 MHz for ^{13}C), CDCl_3 was used as the solvent with TMS as the internal standard. Mass spectra were recorded on a Shimadzu GCMS-QP5050A spectrometer at an ionization voltage of 70 eV equipped with a DB-WAX capillary column (internal diameter: 0.25 mm, length: 30 m). HRMS analysis was performed in an Agilent 1290/Bruker maXis Impact high resolution mass spectrometer. Melting points were determined with a TA DSC250 instrument.

2. General procedure for the synthesis of Sulfonamides

In a typical procedure, amine (0.5 mmol), KI (0.2 mol L^{-1}), and DMSO (5 mL) were added to the undivided cell which was equipped with a Pt plate anode (10 mm \times 10 mm \times 0.1 mm) and Ni plate cathode (10 mm \times 10 mm \times 0.1 mm). The anode and cathode were connecting to a DC regulated power supply. The electrosynthesis was carried out with current 30 mA at room temperature for 8 h under magnetic stirring. After the electrolysis, the reaction mixture was decolorized with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ solution, and then washed with distilled water (50 mL) and extracted with ethyl acetate (15 mL \times 3). The organic layer was dried over anhydrous MgSO_4 and concentrated in vacuum. The crude product was purified by column chromatography on silica gel with petroleum ether-ethyl acetate (v/v=15:1) as eluent to give pure desired product.

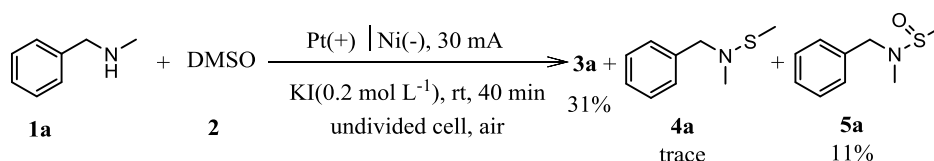
3. Experiments of phenylmethanethiol and (methylsulfinyl)benzene



Pt plate (10 mm × 10 mm × 0.1 mm) anode, Ni plate (10 mm × 10 mm × 0.1 mm) cathode, constant current = 30 mA, **1aa** (0.5 mmol), **2aa** (0.5 mmol), **1ab** (0.5 mmol), **2ab** (0.5 mmol), KI (0.2 mol L⁻¹), DMSO (5.0 mL), room temperature, 8 h, under air and an undivided cell. Yield was analyzed by GC-MS with *n*-dodecane as an internal standard.

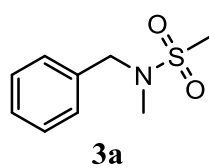
The corresponding product could not be detected when phenylmethanethiol was used as a raw material both in the presence of DMSO and in the absence of DMSO (eq 1, eq 3, eq 5, eq 6). The corresponding product just can be detected by GC-MS when (methylsulfinyl)benzene was used as the raw material (eq 2).

4. The preparing of 5a

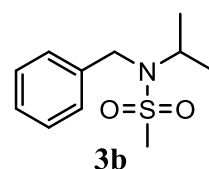


In a typical procedure, **1a** (0.5 mmol), KI (0.2 mol L⁻¹), and DMSO (5 mL) were added to the undivided cell which was equipped with a Pt plate anode and Ni plate cathode. The electrolysis was carried out with current 30 mA at room temperature for 40 minutes under magnetic stirring. The reaction mixture was decolorized with saturated aqueous Na₂S₂O₃ solution, and then washed with distilled water (50 mL) and extracted with ethyl acetate (15 mL × 3). The organic layer was dried over anhydrous MgSO₄ and concentrated in vacuum. The crude product was purified by column chromatography on silica gel with petroleum ether-ethyl acetate (v/v=15:1) as eluent to give pure desired product.

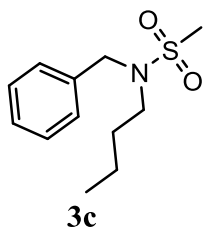
5. Characterization data of products



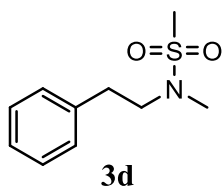
3a *N*-Methyl-*N*-benzylmethanesulfonamide (**3a**);^[1] 81 mg as brown liquid. ¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.28 (m, 5H), 4.31 (s, 2H), 2.83 (s, 3H), 2.77 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 135.58, 128.78, 128.40, 128.08, 53.92, 35.98, 34.33.



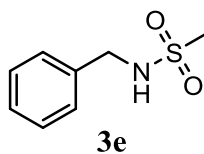
3b *N*-Benzyl-*N*-isopropylmethanesulfonamide (**3b**);^[2] 90 mg as white solid mp 75-76 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, *J* = 7.4 Hz, 2H), 7.33 (t, *J* = 7.3 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 1H), 4.37 (s, 2H), 4.14 (dt, *J* = 13.4, 6.7 Hz, 1H), 2.80 (s, 3H), 1.18 (d, *J* = 6.8 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 138.38, 128.51, 127.92, 127.49, 50.24, 46.70, 40.47, 21.63.



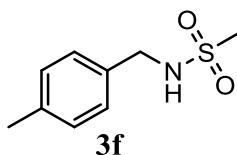
***N*-Benzyl-*N*-butylmethanesulfonamide (3c)**; 106 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.30 (m, 5H), 4.39 (s, 2H), 3.19 – 3.13 (m, 2H), 2.83 (s, 3H), 1.55 – 1.46 (m, 2H), 1.26 (t, $J = 7.4$ Hz, 2H), 0.86 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.23, 128.71, 128.41, 127.95, 50.92, 47.11, 39.16, 30.04, 19.87, 13.68. HRMS (ESI, m/z): Calcd. for $\text{C}_{12}\text{H}_{19}\text{NNaO}_2\text{S}$ [$\text{M}+\text{Na}$] $^+$ 236.0722, found 236.0721.



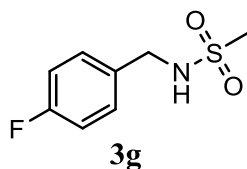
***N*-Methyl-*N*-phenethylmethanesulfonamide (3d)**; 91 mg as white solid, mp 64–65 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.37 – 7.31 (m, 2H), 7.25 (d, $J = 7.4$ Hz, 3H), 3.43 (t, $J = 7.5$ Hz, 2H), 2.92 (t, $J = 7.5$ Hz, 2H), 2.88 (s, 3H), 2.66 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.31, 128.89, 128.68, 126.73, 51.67, 36.17, 34.87. HRMS (ESI, m/z): Calcd. for $\text{C}_{10}\text{H}_{15}\text{NNaO}_2\text{S}$ [$\text{M}+\text{Na}$] $^+$ 236.0722, found 236.0721.



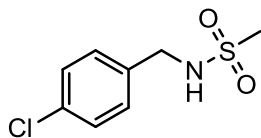
***N*-Benzylmethanesulfonamide (3e)**; ^[3] 67 mg as white solid, mp 62–63 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.28 (m, 5H), 5.07 (s, 1H), 4.29 (d, $J = 6.0$ Hz, 2H), 2.82 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.69, 128.94, 128.14, 127.93, 47.22, 41.13.



***N*-(4-Methylbenzyl) methanesulfonamide (3f)**; ^[3] 78 mg as white solid, mp 97–98 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.23 (d, $J = 7.7$ Hz, 2H), 7.17 (d, $J = 7.7$ Hz, 2H), 4.81 (s, 1H), 4.27 (d, $J = 6.0$ Hz, 2H), 2.84 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.92, 133.64, 129.58, 127.92, 46.99, 41.09, 21.15.

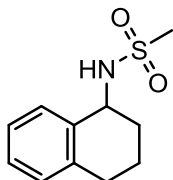


***N*-(4-Fluorobenzyl) methanesulfonamide (3g)**; 77 mg as white solid, mp 52–53 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.30 (m, 2H), 7.05 (t, $J = 8.3$ Hz, 2H), 5.02 (s, 1H), 4.27 (d, $J = 5.9$ Hz, 2H), 2.85 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 163.69, 161.24, 132.58, 129.72, 115.80, 46.45, 41.12. HRMS (ESI, m/z): Calcd. for $\text{C}_{10}\text{H}_{10}\text{FNNaO}_2\text{S}$ [$\text{M}+\text{Na}$] $^+$ 226.0308, found 226.0314.



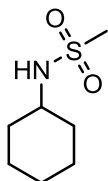
3h

N-(4-Chlorobenzyl)methanesulfonamide (**3h**);^[3] 80 mg as white solid, mp 133-134 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 4.87 (s, 1H), 4.28 (d, *J* = 6.0 Hz, 2H), 2.88 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 135.28, 134.01, 129.17, 46.48, 41.24.



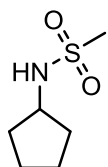
3i

N-(1,2,3,4-Tetrahydronaphthalen-1-yl)methanesulfonamide (**3i**);^[4] 99 mg as white solid, mp 109-110 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.48 – 7.40 (m, 1H), 7.24 – 7.16 (m, 2H), 7.13 – 7.07 (m, 1H), 4.73 (s, 1H), 4.66 (s, 1H), 3.05 (s, 3H), 2.80 (d, *J* = 18.6 Hz, 2H), 2.11 (s, 1H), 1.91 (d, *J* = 17.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 137.54, 135.72, 129.34, 128.81, 127.79, 126.44, 52.23, 42.16, 31.51, 28.88, 19.37.



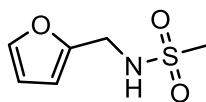
3j

N-Cyclohexylmethanesulfonamide (**3j**);^[5] 77 mg as white solid, mp 106-107 °C. ¹H NMR (400 MHz, CDCl₃) δ 4.58 (s, 1H), 3.31 (s, 1H), 2.99 (s, 3H), 1.99 (d, *J* = 11.8 Hz, 2H), 1.78 – 1.72 (m, 2H), 1.60 (d, *J* = 13.0 Hz, 1H), 1.36 – 1.20 (m, 5H). ¹³C NMR (100 MHz, CDCl₃) δ 52.84, 42.05, 34.38, 25.14, 24.78.



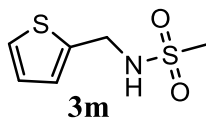
3k

N-Cyclopentylmethanesulfonamide (**3k**);^[6] 73 mg as brown liquid. ¹H NMR (400 MHz, CDCl₃) δ 4.59 (s, 1H), 3.80 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.99 (s, 3H), 2.02 (dt, *J* = 12.0, 6.3 Hz, 2H), 1.75 – 1.49 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 55.26, 41.34, 33.84, 23.20.



3l

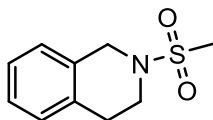
N-(Furan-2-ylmethyl)methanesulfonamide (**3l**);^[7] 72 mg as brown liquid. ¹H NMR (400 MHz, CDCl₃) δ 7.43 (s, 1H), 6.36 (d, *J* = 9.9 Hz, 2H), 4.83 (s, 1H), 4.36 (d, *J* = 5.8 Hz, 2H), 2.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 150.05, 142.92, 110.64, 108.65, 41.22, 39.99.



3m

N-(Thiophen-2-ylmethyl)methanesulfonamide (3m);^[7] 83 mg as brown liquid.

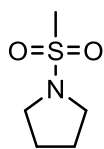
¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 5.1 Hz, 1H), 7.05 (s, 1H), 6.99 – 6.95 (m, 1H), 4.77 (s, 1H), 4.52 (d, *J* = 5.7 Hz, 1H), 2.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.55, 127.16, 126.84, 126.05, 41.97, 41.53.



3n

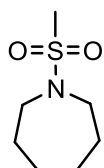
2-(Methylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (3n);^[8] 101 mg as white solid,

mp 129-130 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.19 (dd, *J* = 26.2, 14.5 Hz, 4H), 4.49 (s, 2H), 3.59 (t, *J* = 5.8 Hz, 2H), 3.01 (t, *J* = 5.7 Hz, 2H), 2.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 133.16, 129.07, 127.04, 126.59, 126.39, 47.30, 43.46, 35.88, 28.70



3o

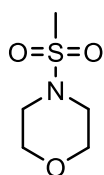
1-(Methylsulfonyl)pyrrolidine (3o);^[2] 65 mg as brown solid, mp 70-71 °C. ¹H NMR (400 MHz, CDCl₃) δ 3.32 (t, *J* = 6.1 Hz, 4H), 2.81 (s, 3H), 1.98 – 1.91 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 47.88, 34.44, 25.72.



3p

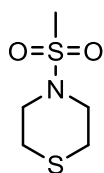
1-(Methylsulfonyl)azepane (3p); 82 mg as brown solid, mp 45-46 °C. ¹H NMR (400

MHz, CDCl₃) δ 3.36 – 3.32 (m, 4H), 2.81 (s, 3H), 1.76 (s, 4H), 1.64 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 48.06, 36.88, 29.33, 26.94. HRMS (ESI, *m/z*): Calcd. for C₇H₁₅NNaO₂S [M+Na]⁺ 200.0713, found 200.0716.



3q

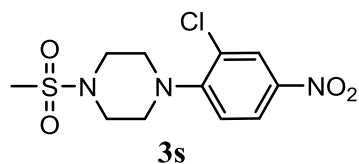
4-(Methylsulfonyl)morpholine (3q);^[9] 82 mg as white solid, mp 91-92 °C. ¹H NMR (400 MHz, CDCl₃) δ 3.74 – 3.70 (m, 4H), 3.16 – 3.12 (m, 3H), 2.73 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 66.30, 45.86, 33.95.



3r

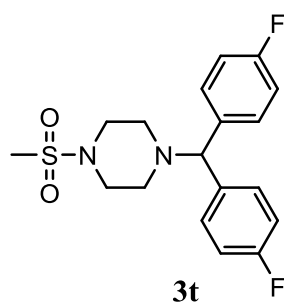
4-(Methylsulfonyl)thiomorpholine (3r);^[9] 82 mg as white solid, mp 131-132 °C. ¹H NMR

(400 MHz, CDCl₃) δ 3.54 (s, 4H), 2.81 (s, 3H), 2.76 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 47.67, 35.85, 27.53.



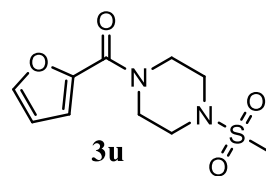
1-(2-Chloro-4-nitrophenyl)-4-(methylsulfonyl)piperazine (3s); 129

mg as brown solid, mp 178-179 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, J = 2.5 Hz, 1H), 8.13 (dd, J = 8.9, 2.5 Hz, 1H), 7.09 (d, J = 8.9 Hz, 1H), 3.49 – 3.43 (m, 4H), 3.33 – 3.27 (m, 4H), 2.87 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 154.01, 142.96, 128.15, 126.63, 123.53, 119.97, 50.33, 45.83, 34.66, 29.71. HRMS (ESI, m/z): Calcd. for C₁₁H₁₄ClN₃NaO₄S [M+Na]⁺ 342.0280, found 342.0286.



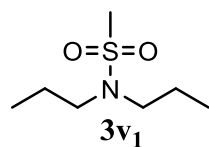
1-(Bis(4-fluorophenyl)methyl)-4-(methylsulfonyl)piperazine (3t); 118 mg

as brown solid, mp 199-200 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.37 – 7.29 (m, 4H), 6.98 (t, J = 8.5 Hz, 4H), 4.28 (s, 1H), 3.23 (s, 4H), 2.79 (s, 3H), 2.47 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 163.19, 160.74, 137.50, 129.19, 115.77, 115.56, 73.97, 50.99, 46.08, 34.12. HRMS (ESI, m/z): Calcd. for C₁₈H₂₁F₂N₂O₂S [M+Na]⁺ 367.1284, found 367.1286.



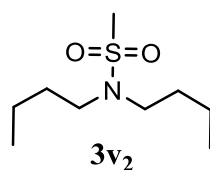
Furan-2-yl(4-(methylsulfonyl)piperazin-1-yl)methanone (3u); 112 mg as

white solid, mp 155-156 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.51 (s, 1H), 7.08 (d, J = 3.3 Hz, 1H), 6.52 (s, 1H), 3.94 (s, 4H), 3.33 – 3.29 (m, 4H), 2.81 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.09, 147.49, 144.06, 117.48, 111.63, 45.99, 34.70. HRMS (ESI, m/z): Calcd. for C₁₀H₁₅N₂O₄S [M+H]⁺ 259.0752; found 259.0749.



N,N-Dipropylmethanesulfonamide(3v1); ^[10] 72 mg as brown liquid. ¹H NMR (400

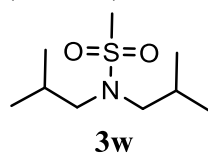
MHz, CDCl₃) δ 3.16 – 3.10 (m, 4H), 2.83 (s, 3H), 1.63 (dd, J = 15.2, 7.3 Hz, 4H), 0.92 (t, J = 7.4 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 49.58, 38.28, 22.02, 11.17.



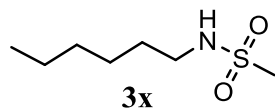
N,N-Dibutylmethanesulfonamide (3v2); 78 mg as brown liquid. ¹H NMR (400

MHz, CDCl₃) δ 3.16 (t, J = 7.6 Hz, 4H), 2.82 (s, 3H), 1.63 – 1.53 (m, 4H), 1.35 (dt, J = 14.8, 7.3 Hz,

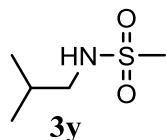
4H), 0.94 (t, $J = 7.3$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 47.54, 38.26, 30.81, 19.94, 13.74. HRMS (ESI, m/z): Calcd. for $\text{C}_9\text{H}_{21}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$ 230.1181, found 230.1185.



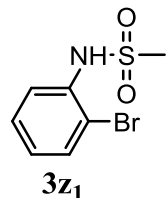
***N,N*-Diisobutylmethanesulfonamide (3w)**; 83 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 3.16 (t, $J = 7.6$ Hz, 4H), 2.82 (s, 3H), 1.63 – 1.53 (m, 4H), 1.35 (dt, $J = 14.8, 7.3$ Hz, 4H), 0.94 (t, $J = 7.3$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 47.54, 38.26, 30.81, 19.94, 13.74. HRMS (ESI, m/z): Calcd. for $\text{C}_9\text{H}_{21}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$ 230.1185, found 230.1185.



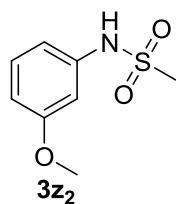
***N*-Hexylmethanesulfonamide (3x)**,^[11] 67 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 4.64 (s, 1H), 3.15 – 3.09 (m, 2H), 2.96 (s, 3H), 1.59 – 1.53 (m, 2H), 1.31 (s, 6H), 0.89 (t, $J = 6.0$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 43.36, 40.12, 31.32, 30.04, 26.24, 22.51, 13.99.



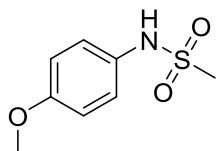
***N*-Isobutylmethanesulfonamide (3y)**,^[12] 59 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 4.79 (s, 1H), 2.99 – 2.91 (m, 5H), 1.82 (dt, $J = 13.3, 6.7$ Hz, 1H), 0.96 (d, $J = 6.7$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 50.64, 40.08, 28.77, 19.86.



***N*-(2-Bromophenyl)methanesulfonamide (3z₁)**,^[13] 106 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 8.0$ Hz, 1H), 7.59 (d, $J = 8.0$ Hz, 1H), 7.35 (t, $J = 7.7$ Hz, 1H), 7.08 (d, $J = 15.2$ Hz, 1H), 6.80 (s, 1H), 3.01 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 134.83, 132.97, 129.02, 126.74, 122.65, 115.62, 39.91.



***N*-(3-Methoxyphenyl)methanesulfonamide (3z₂)**,^[14] 106 mg as brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.27 – 7.22 (m, 1H), 6.83 – 6.71 (m, 4H), 3.81 (s, 3H), 3.02 (s, 3H). ^{13}C NMR (400 MHz, CDCl_3) δ 160.69, 137.97, 130.49, 112.62, 110.86, 106.47, 55.42, 39.30.

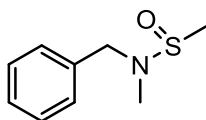


3z₃

N-(4-Methoxyphenyl)methanesulfonamide (**3z₃**);^[14] 46 mg as brown liquid.

¹H NMR (400 MHz, CDCl₃) δ 7.27 – 7.18 (m, 3H), 6.89 (d, *J* = 8.9 Hz, 2H), 3.80 (s, 3H), 2.95 (s, 3H).

¹³C NMR (400 MHz, CDCl₃) δ 158.20, 128.97, 124.90, 114.86, 55.54, 38.97.



5a

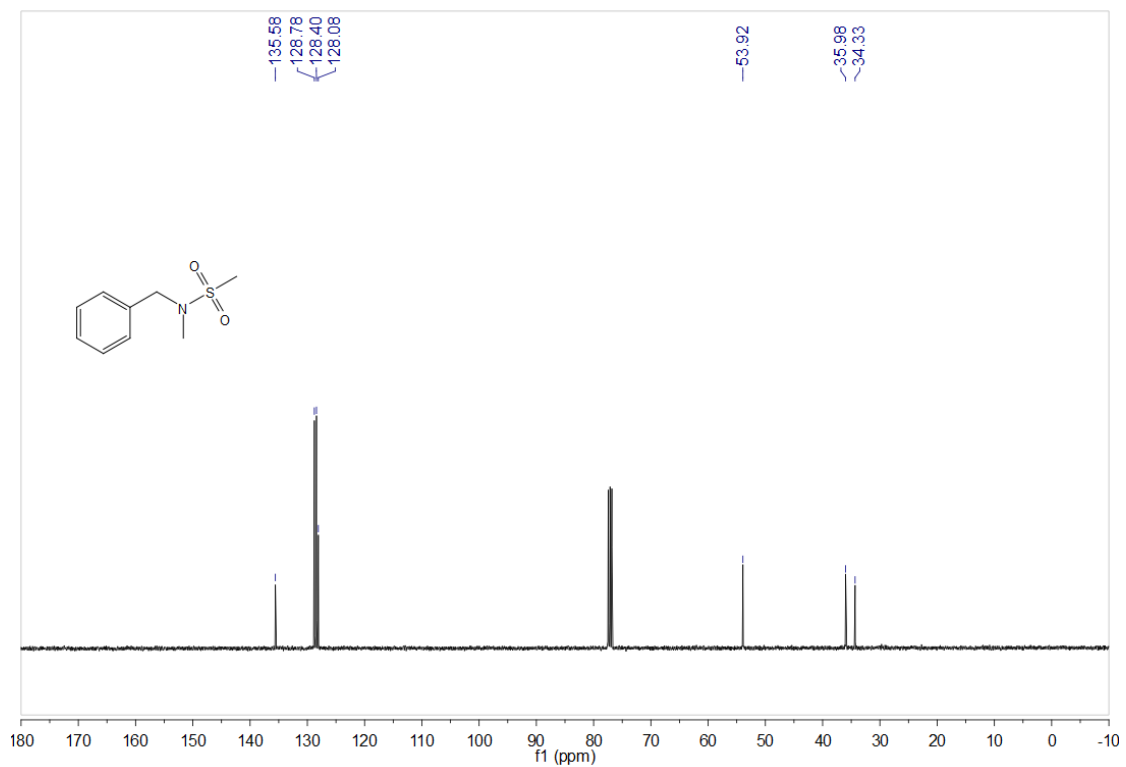
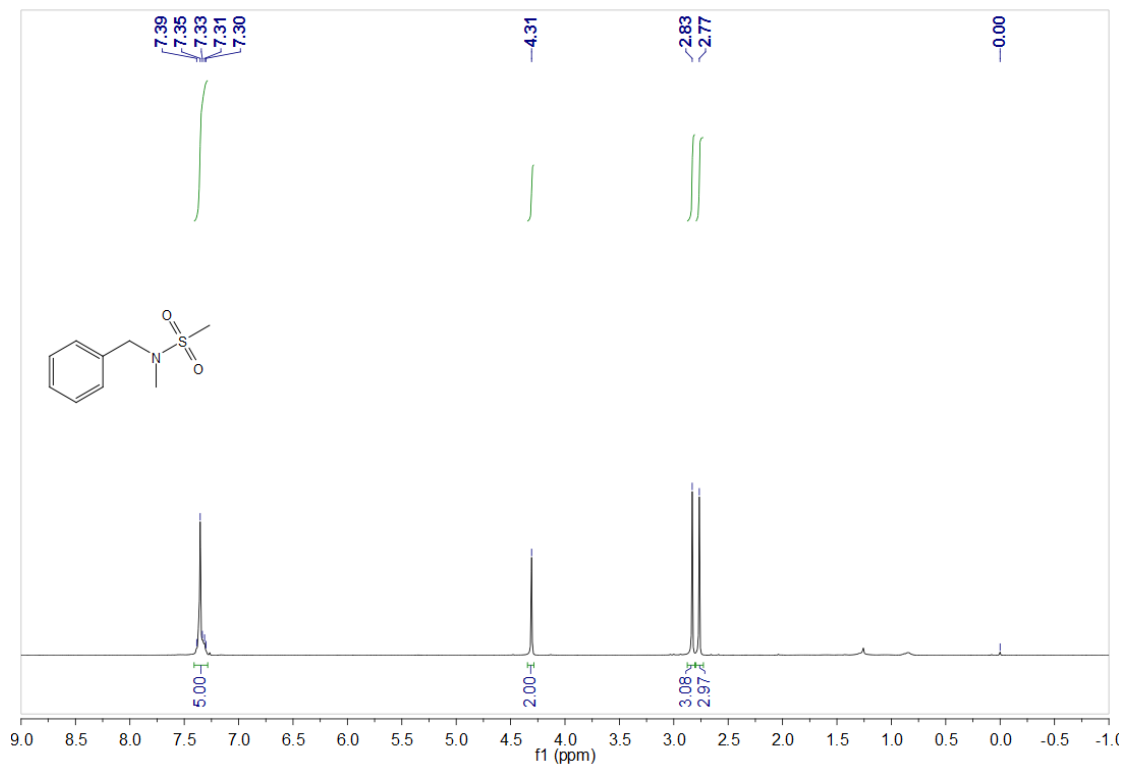
N-Benzyl-*N*-methylmethanesulfonamide (**5a**), yellow liquid. ¹H NMR (400 MHz, CDCl₃) δ 7.37 – 7.27 (m, 5H), 4.25 (d, *J* = 14.3 Hz, 2H), 2.65 (d, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 136.84, 128.64, 128.17, 127.72, 55.89, 40.23, 31.80. MS (EI) *m/z*: 183, 168, 118, 91, 77, 65.

6. References

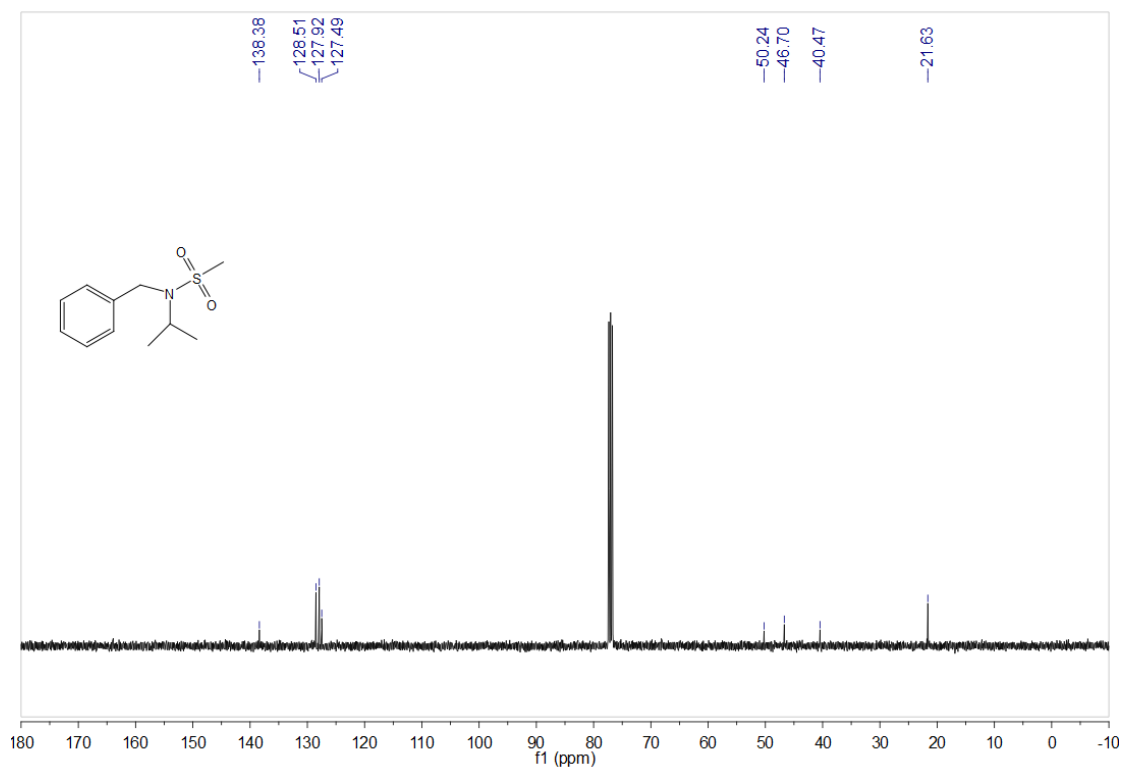
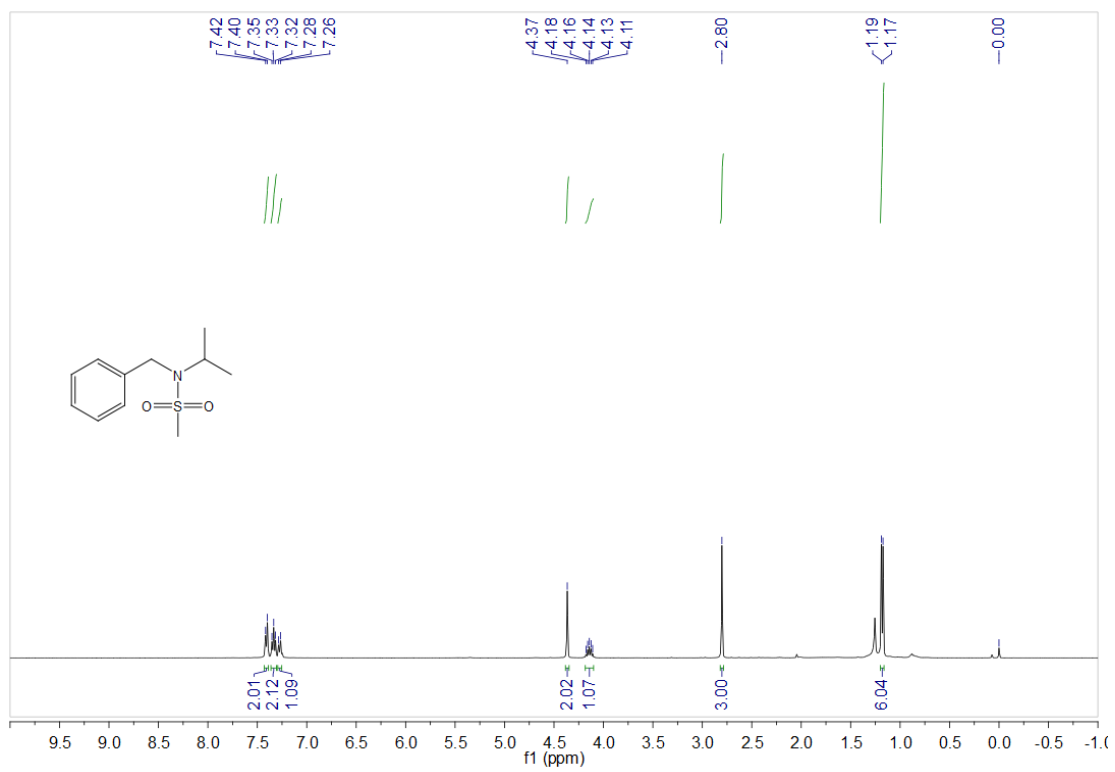
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7. NMR spectra of products

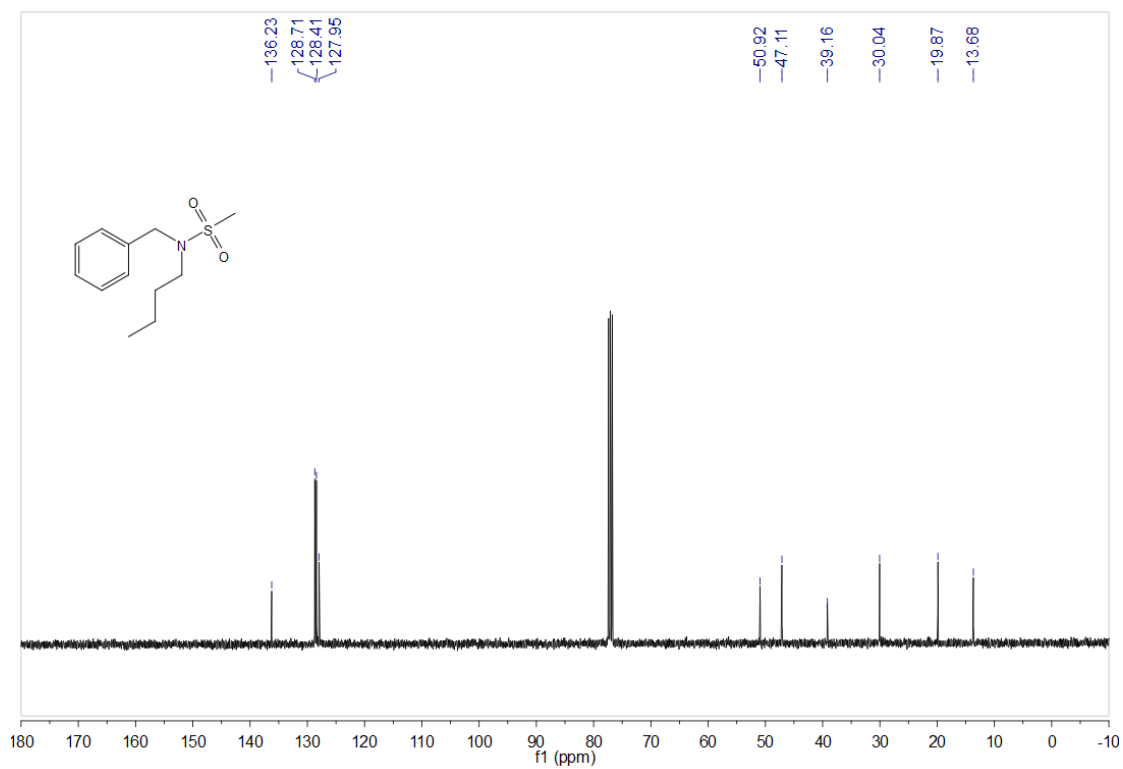
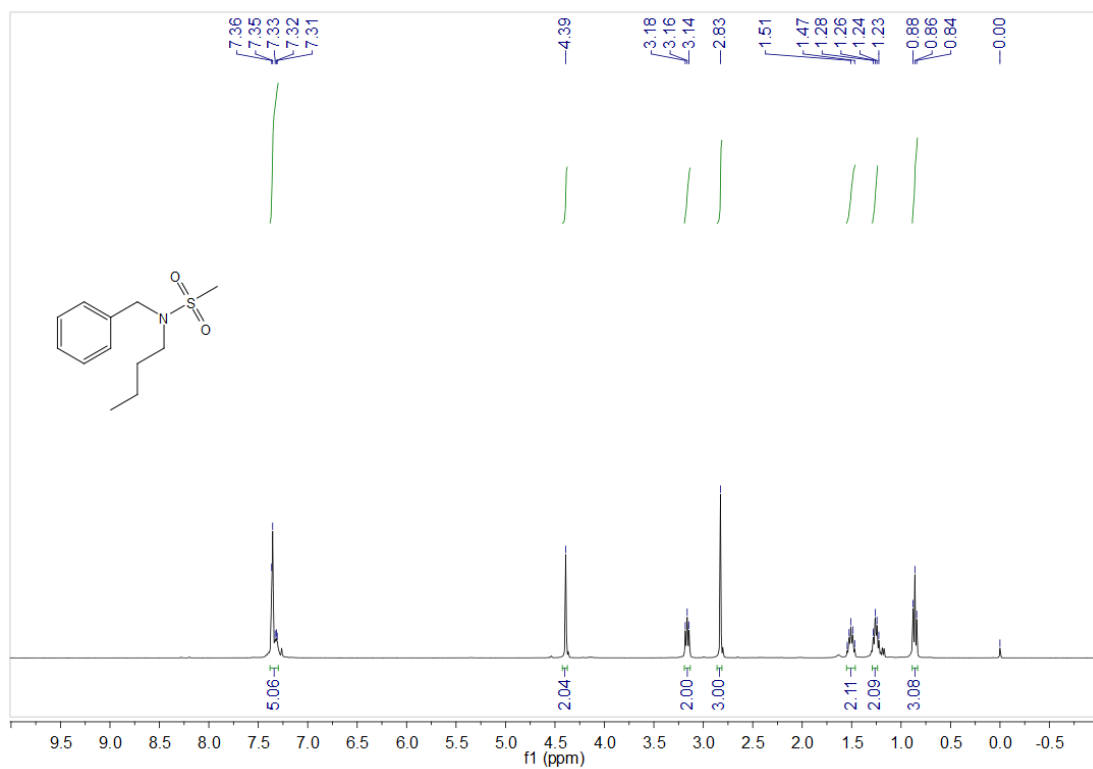
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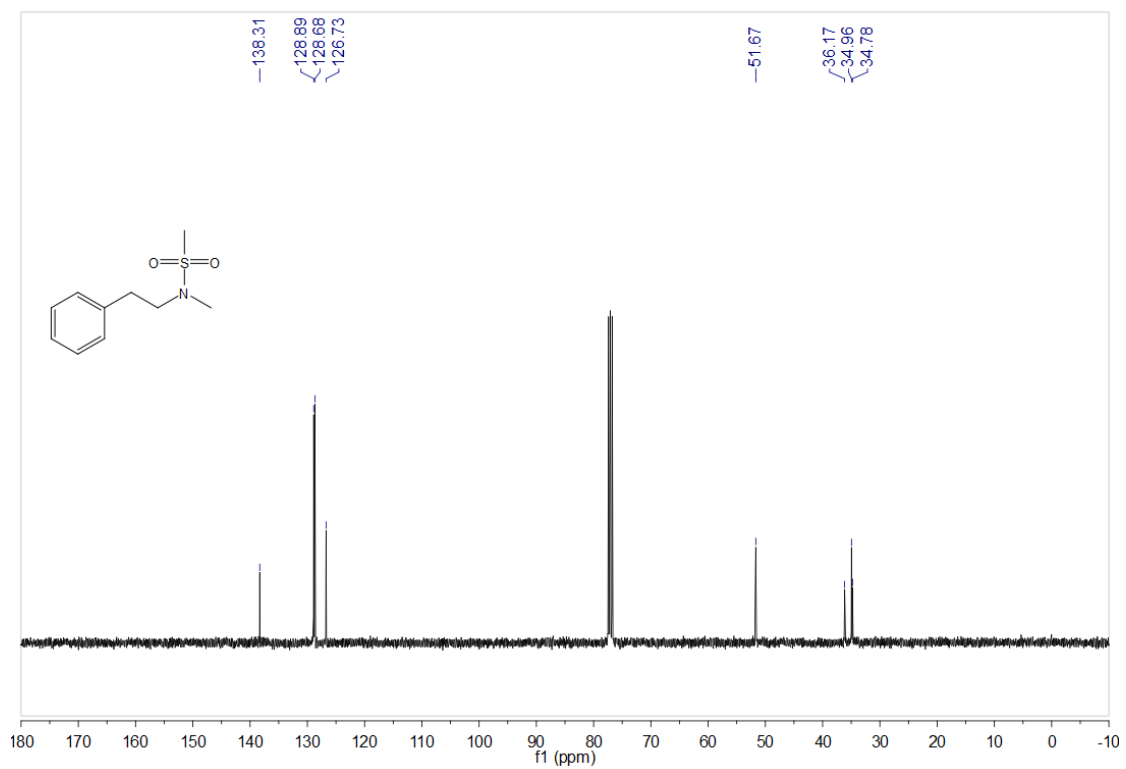
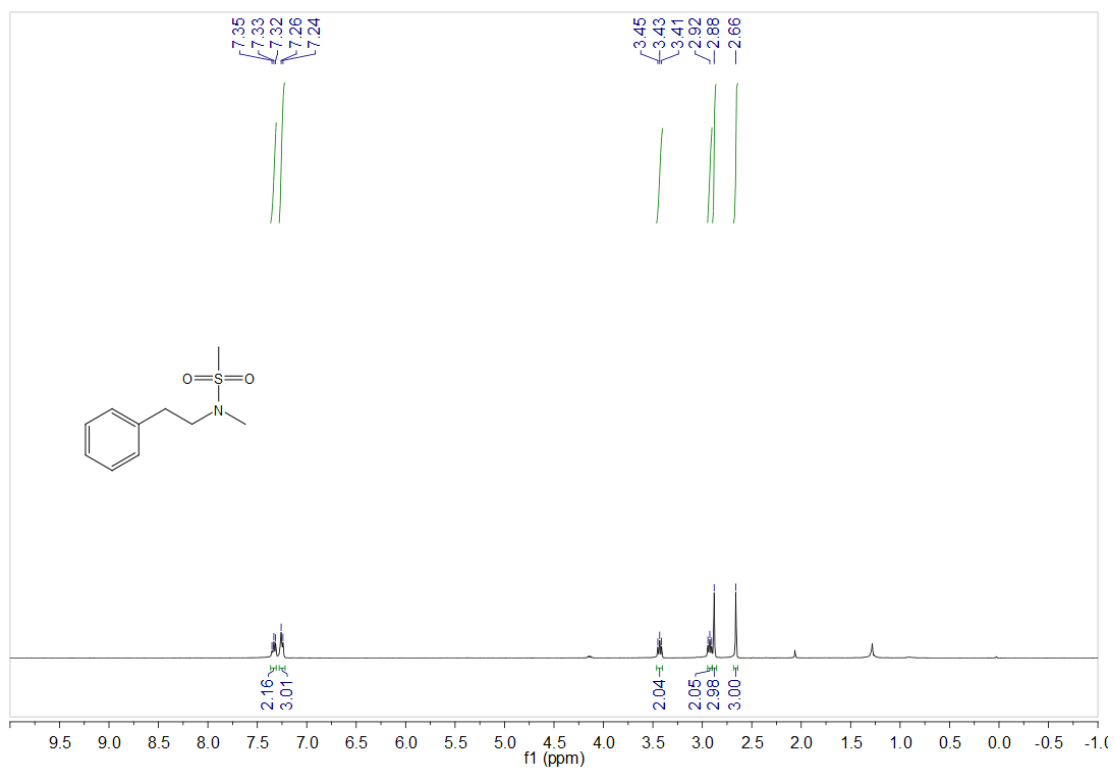
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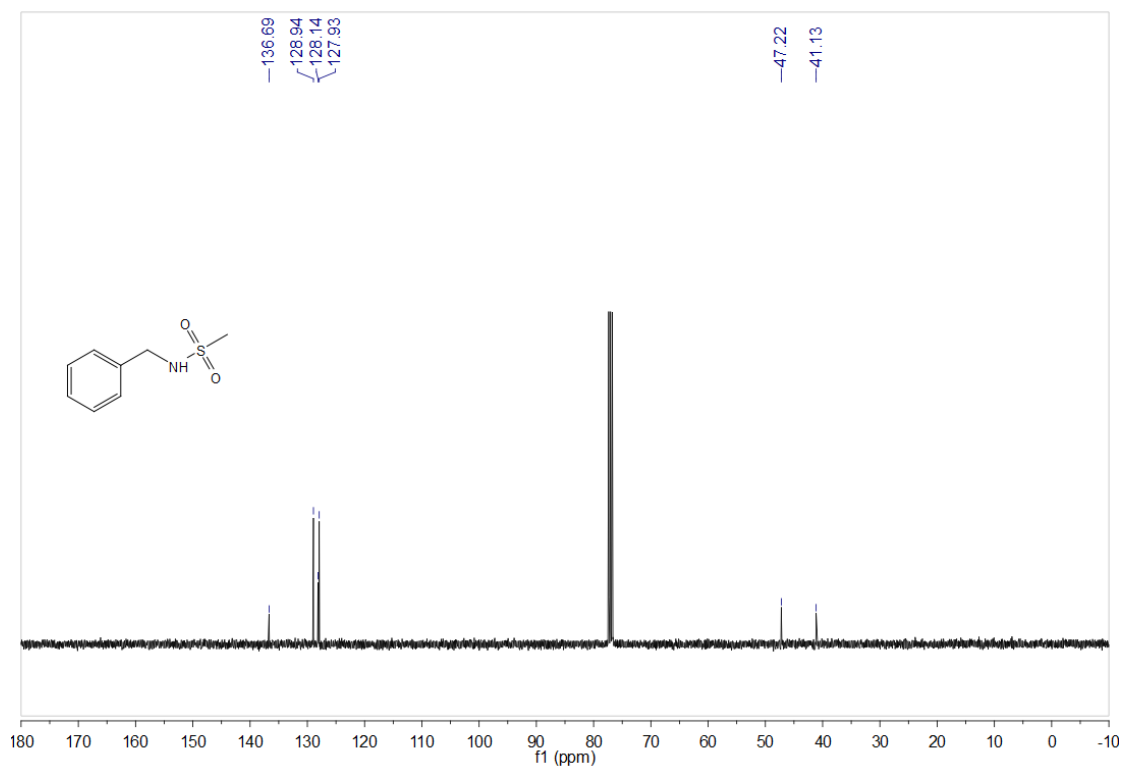
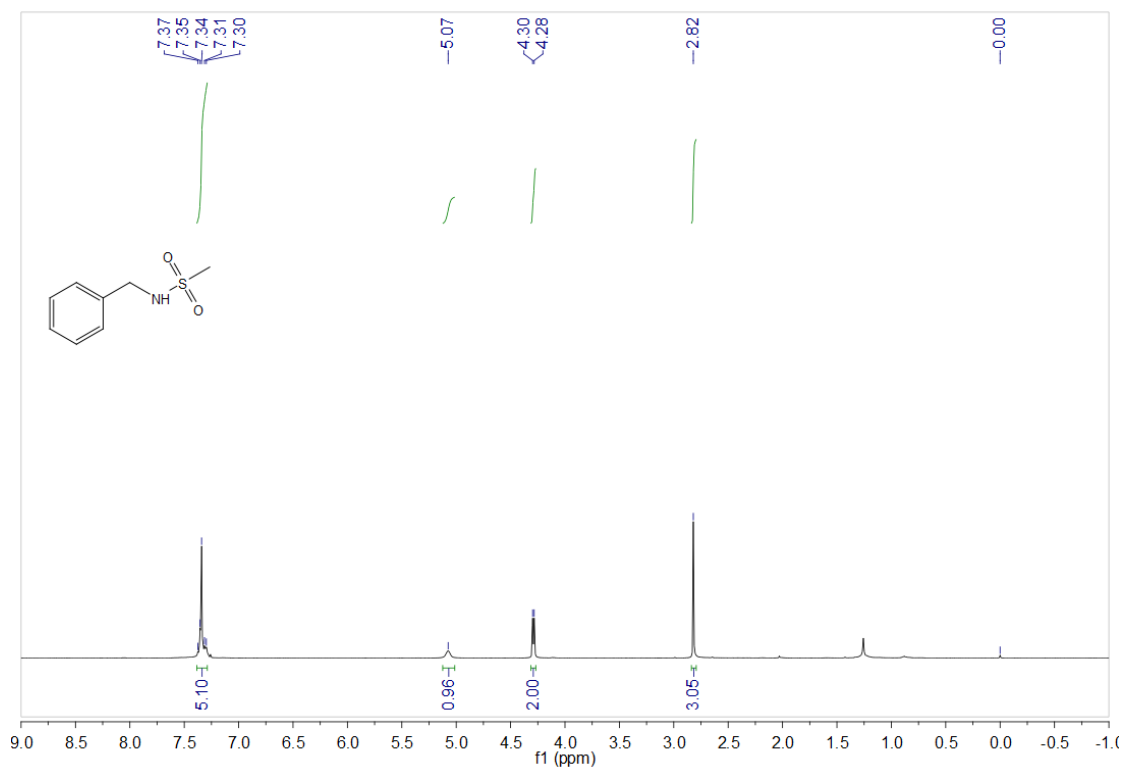
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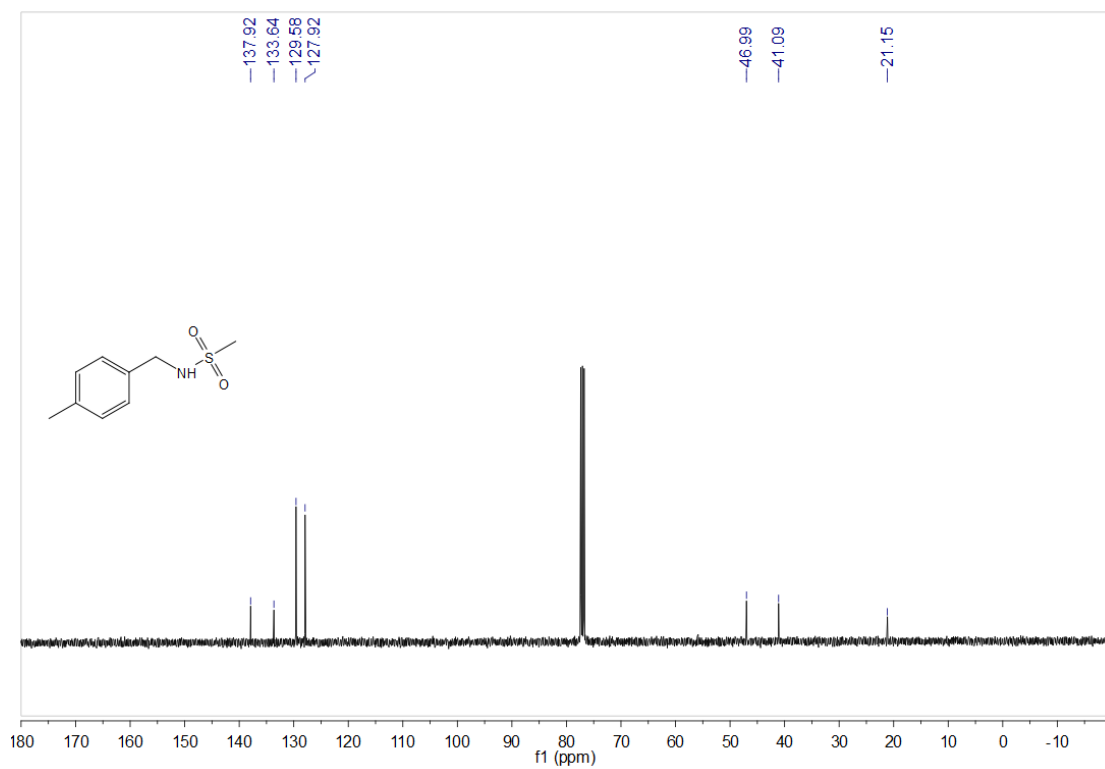
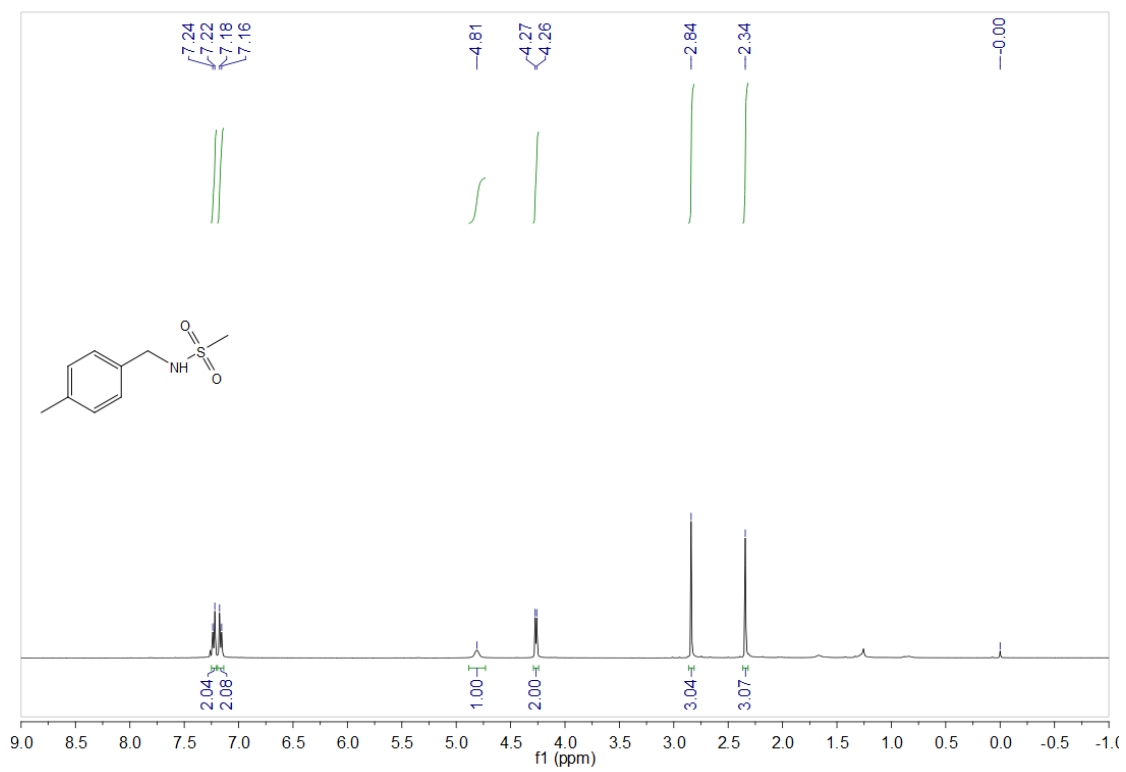
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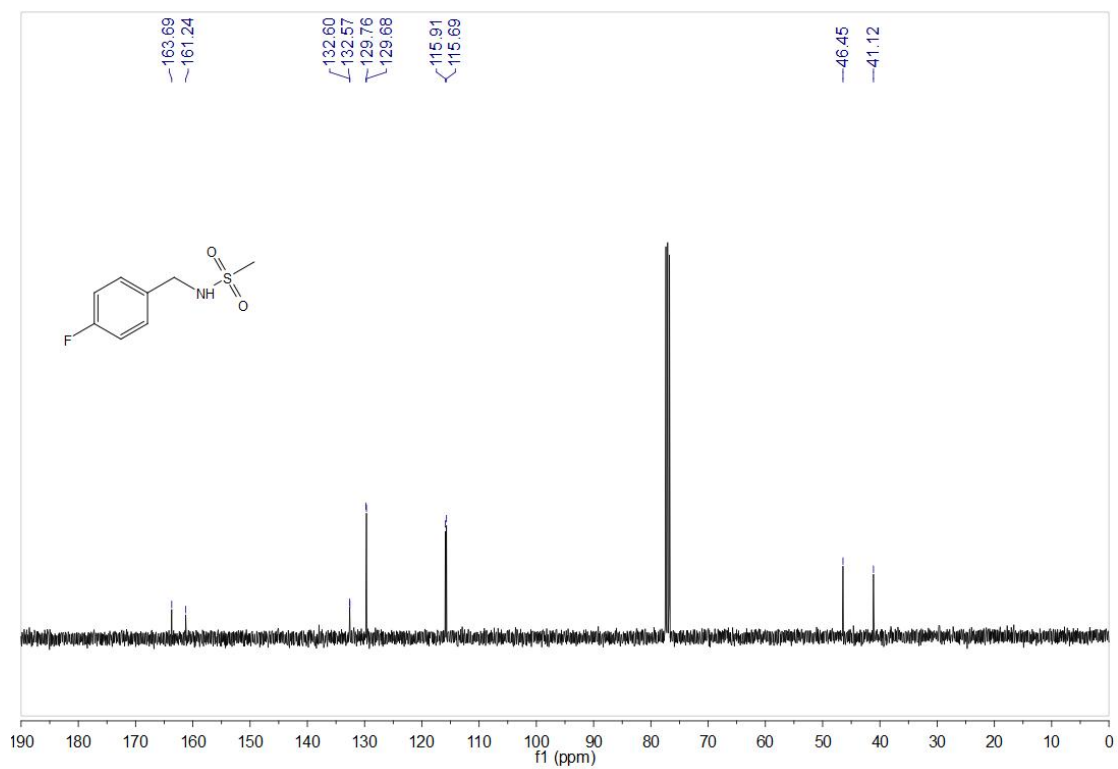
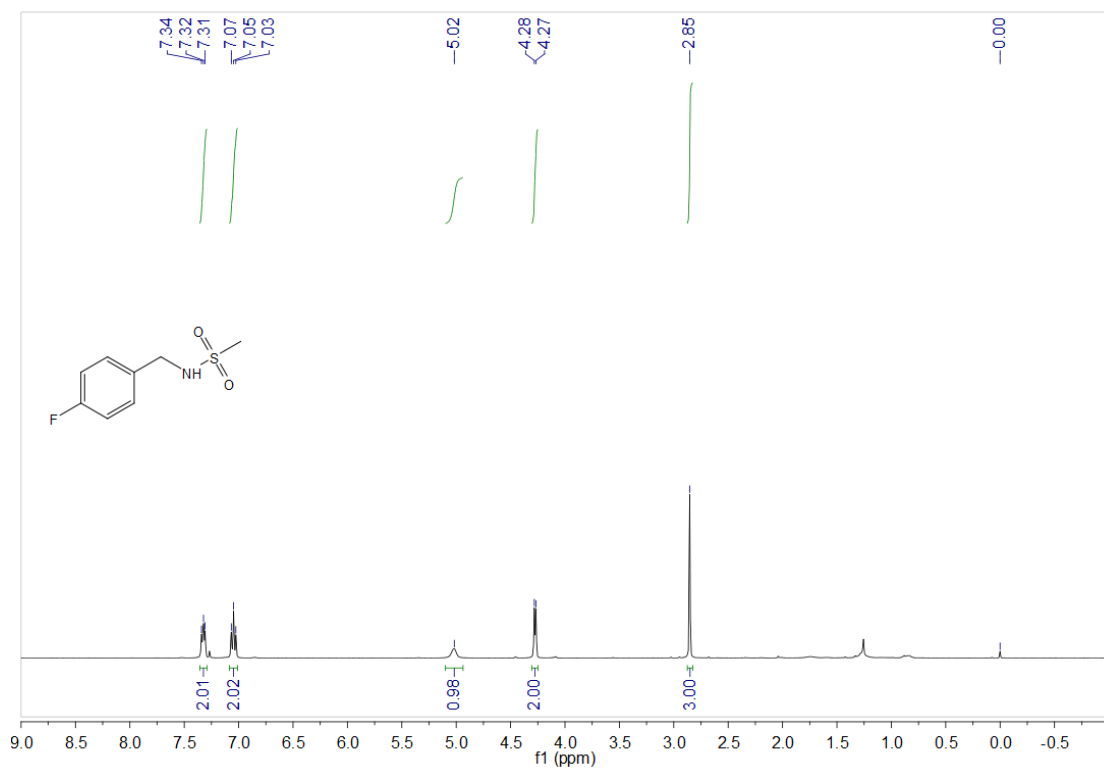
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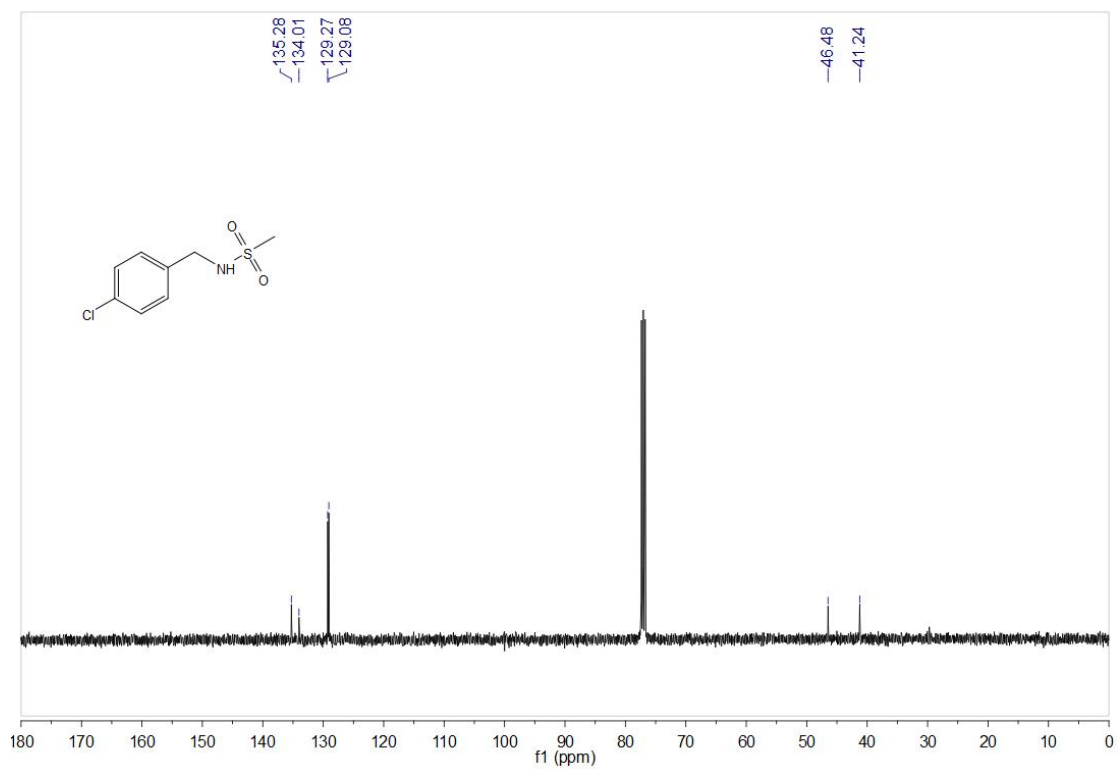
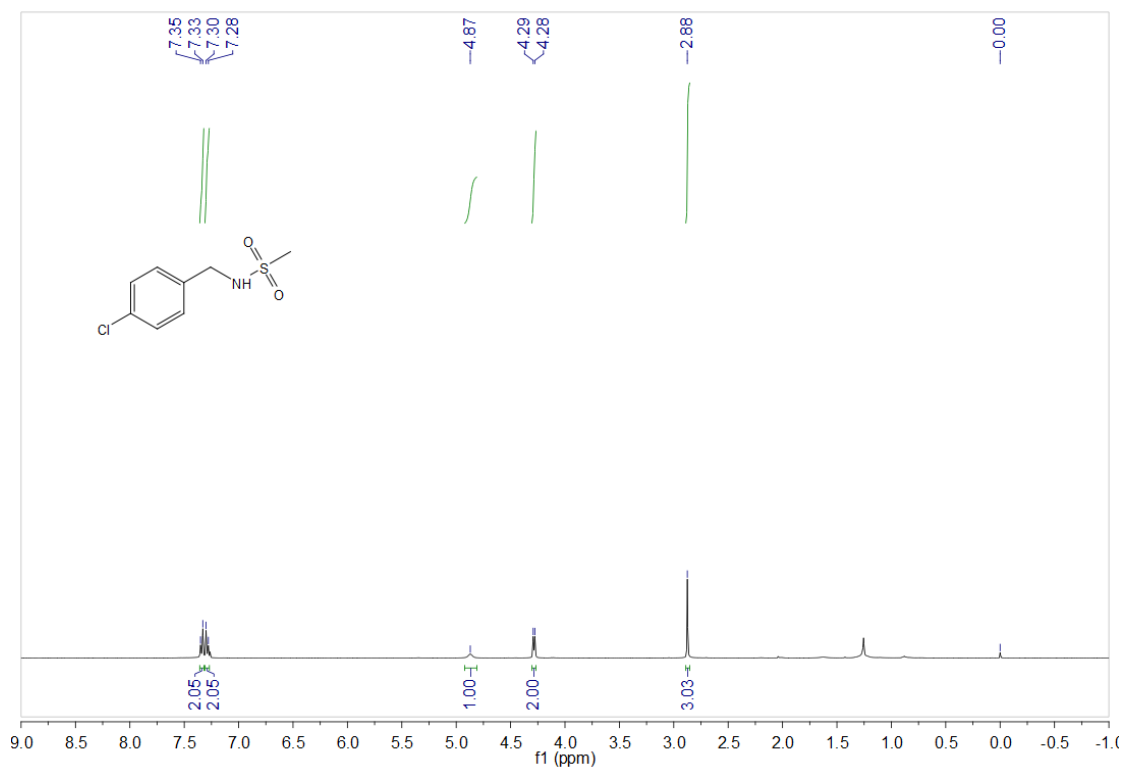
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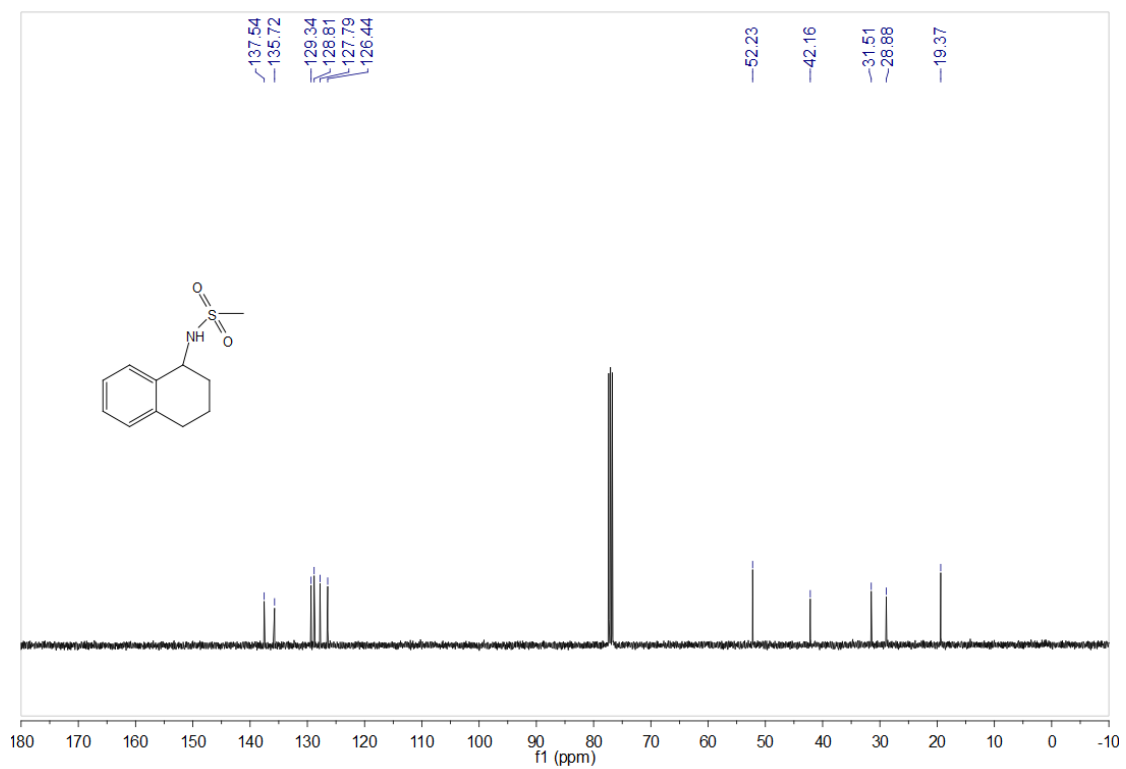
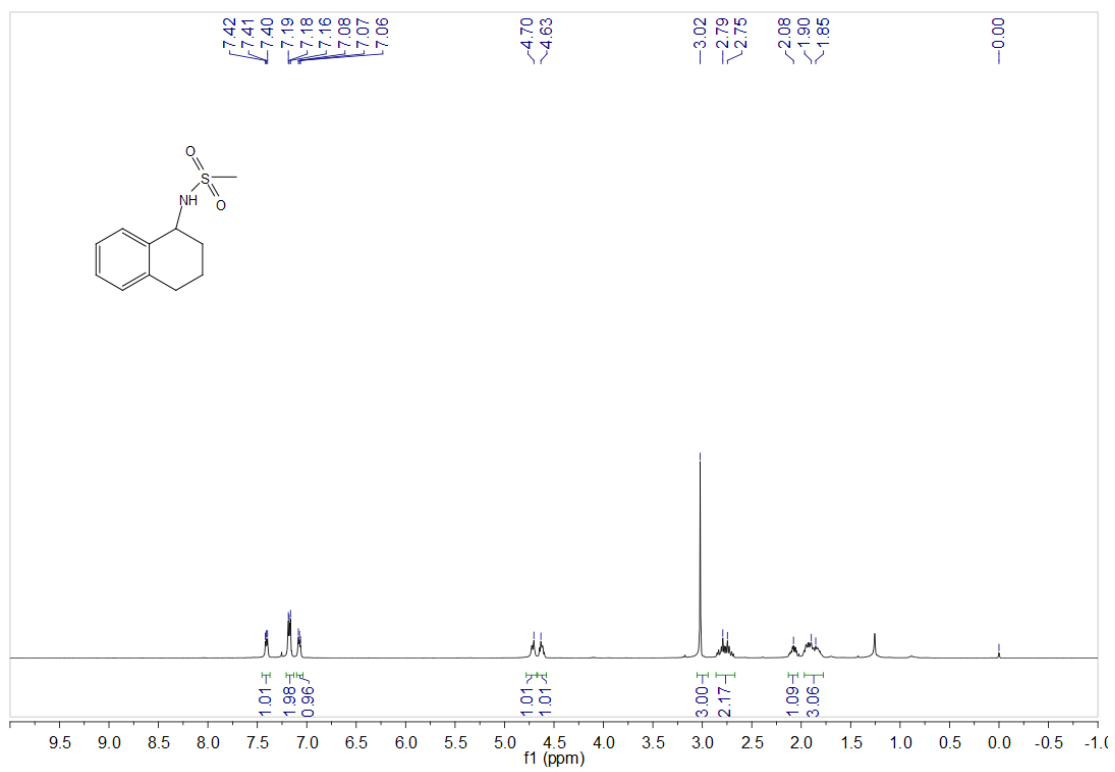
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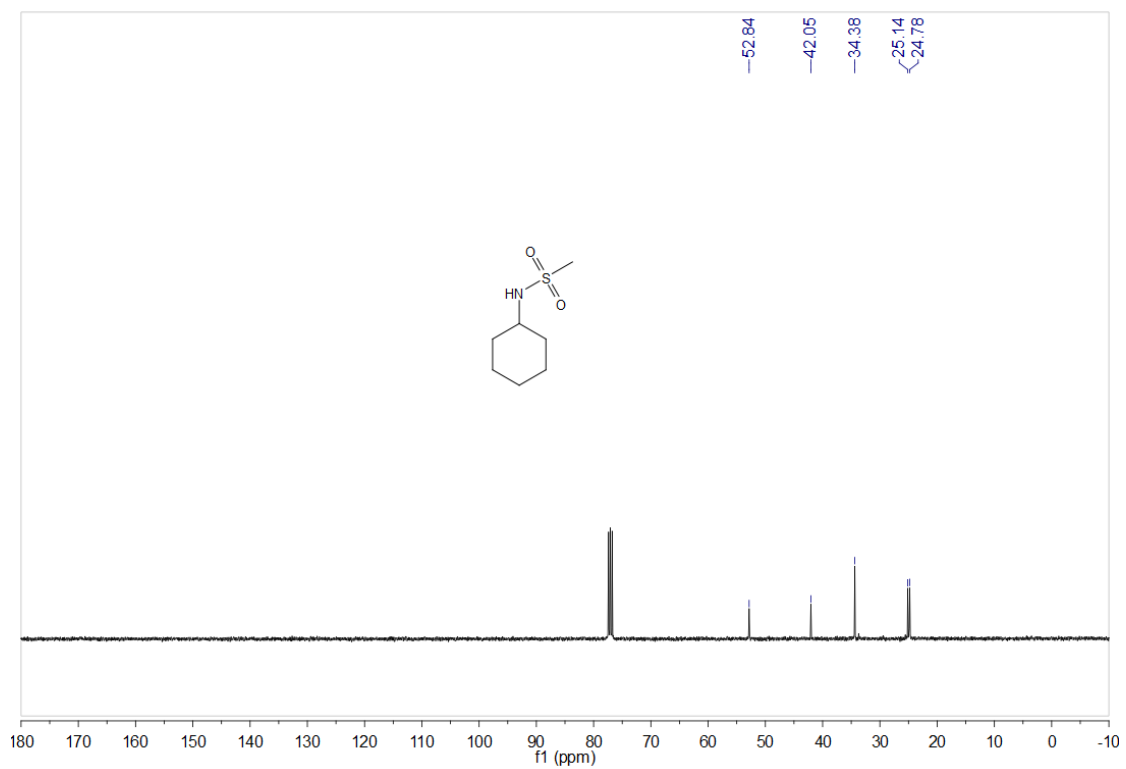
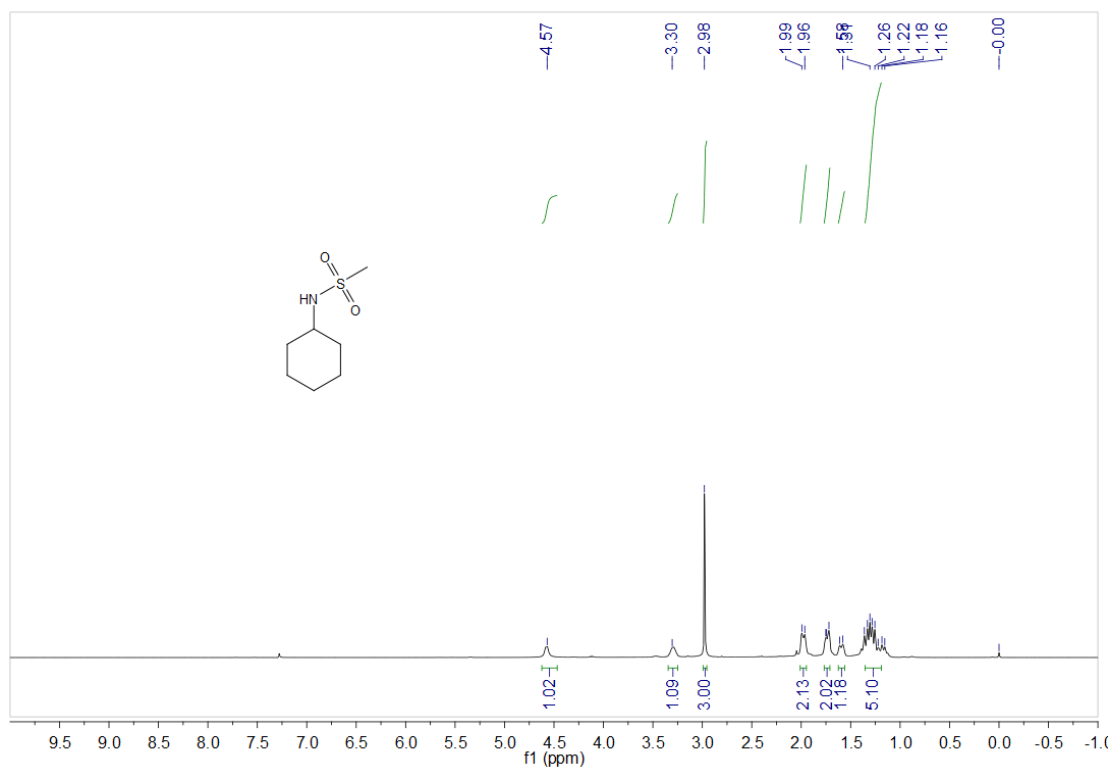
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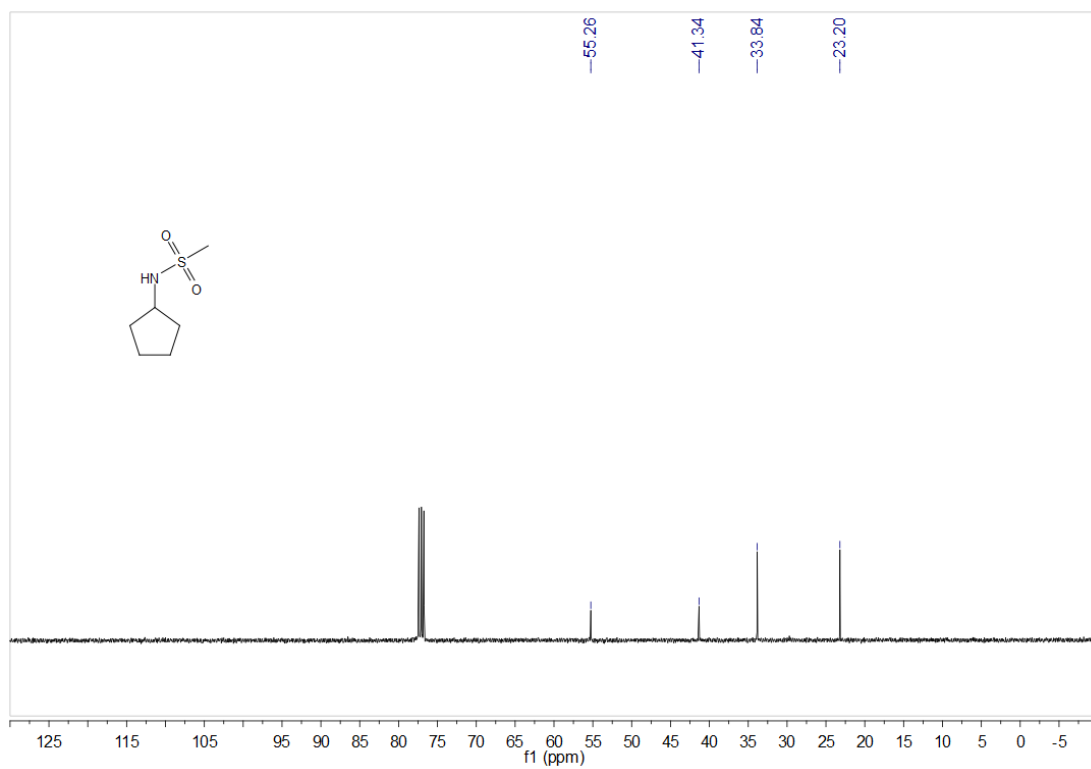
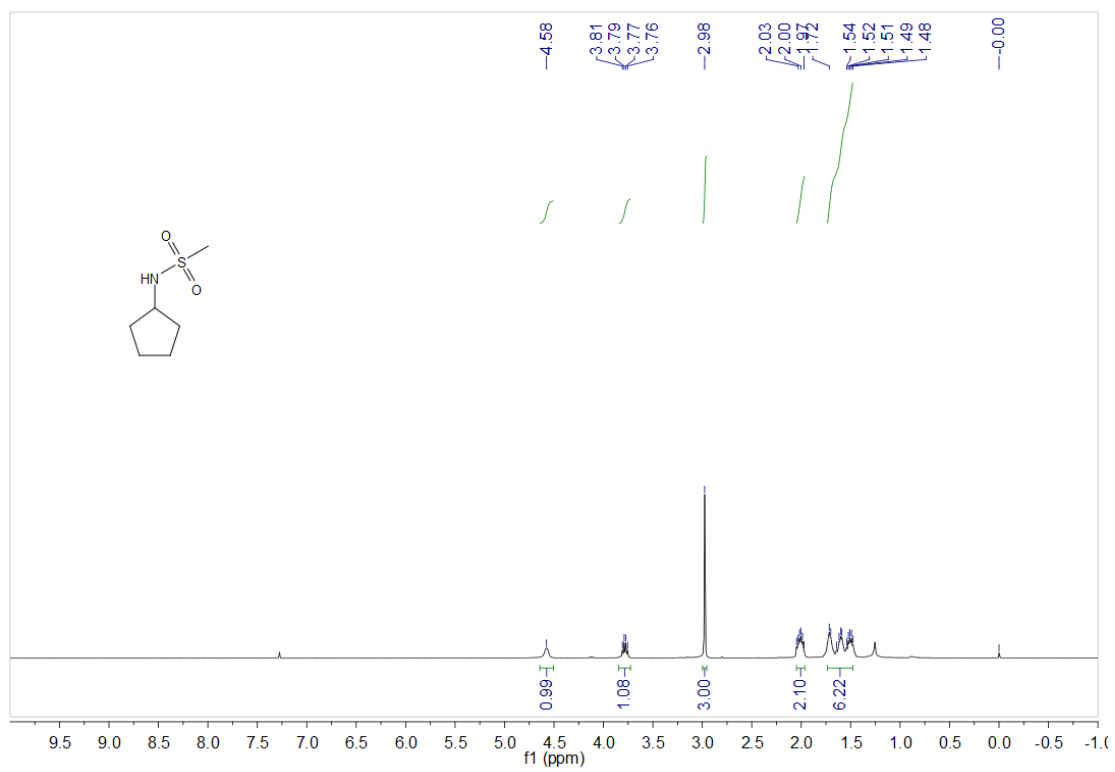
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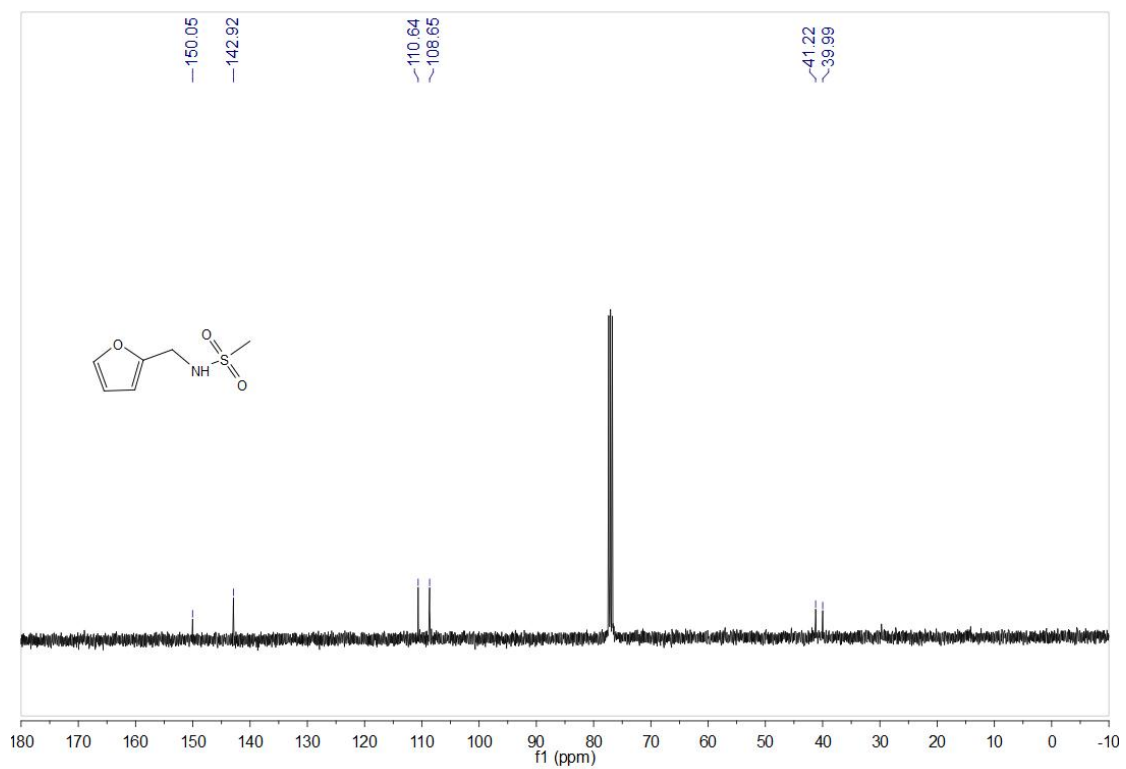
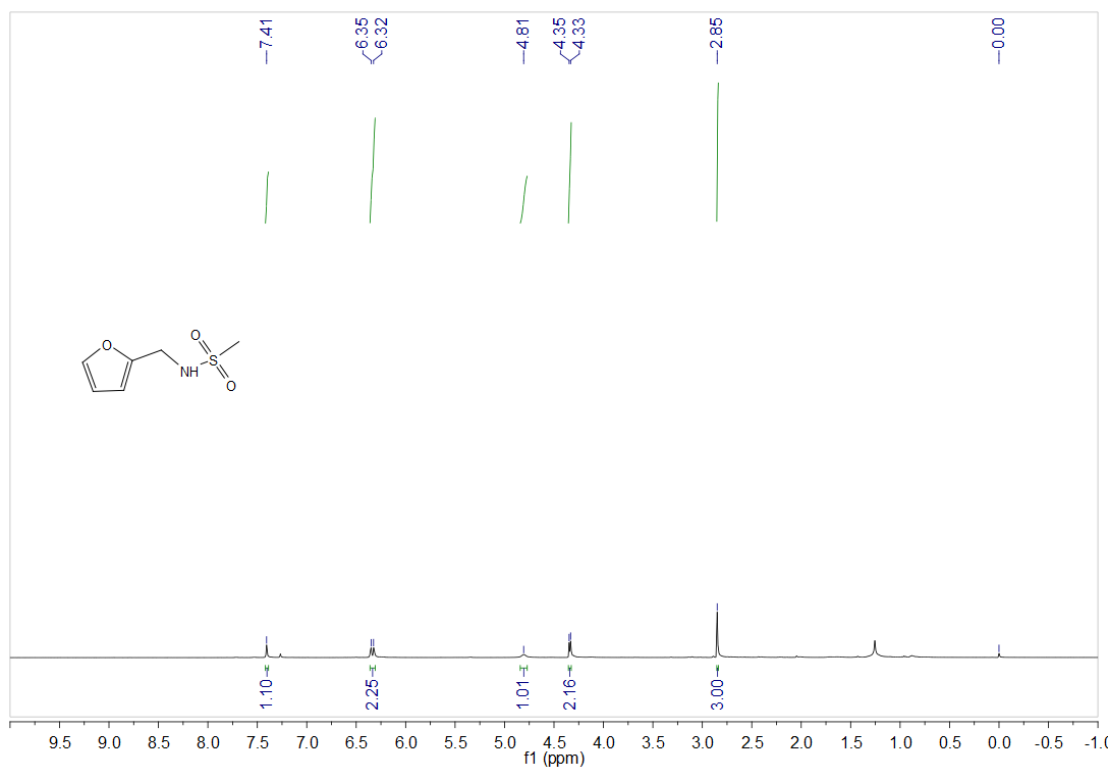
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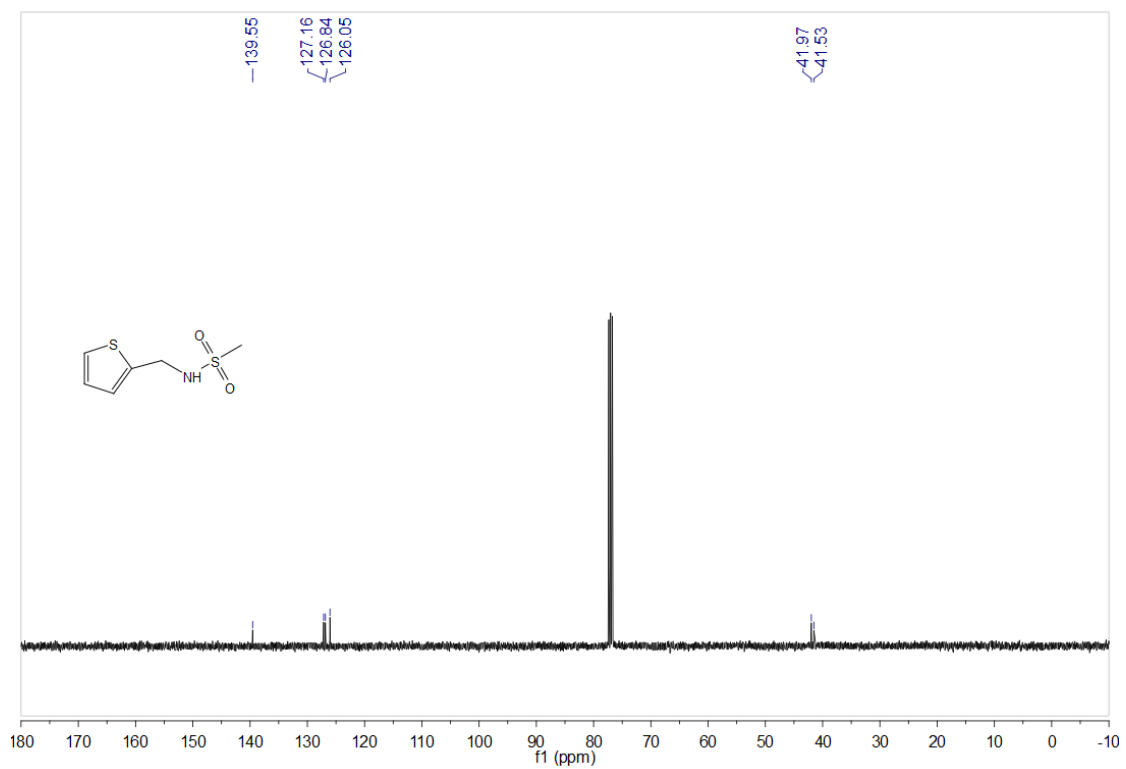
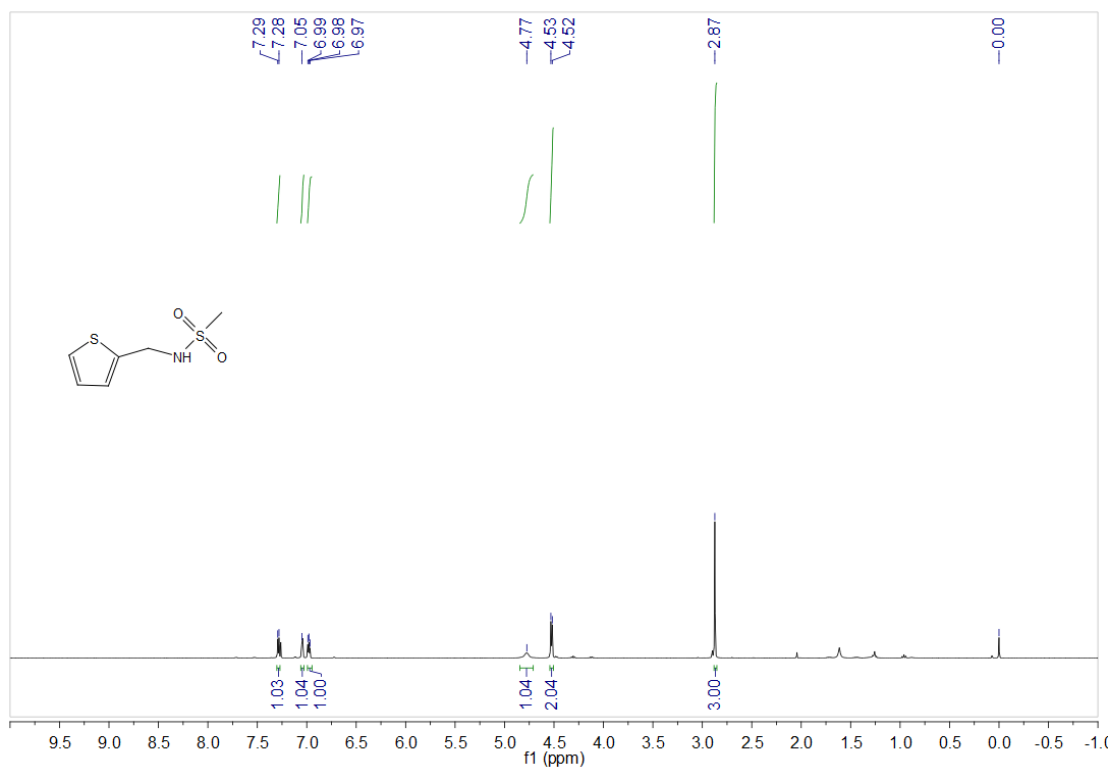
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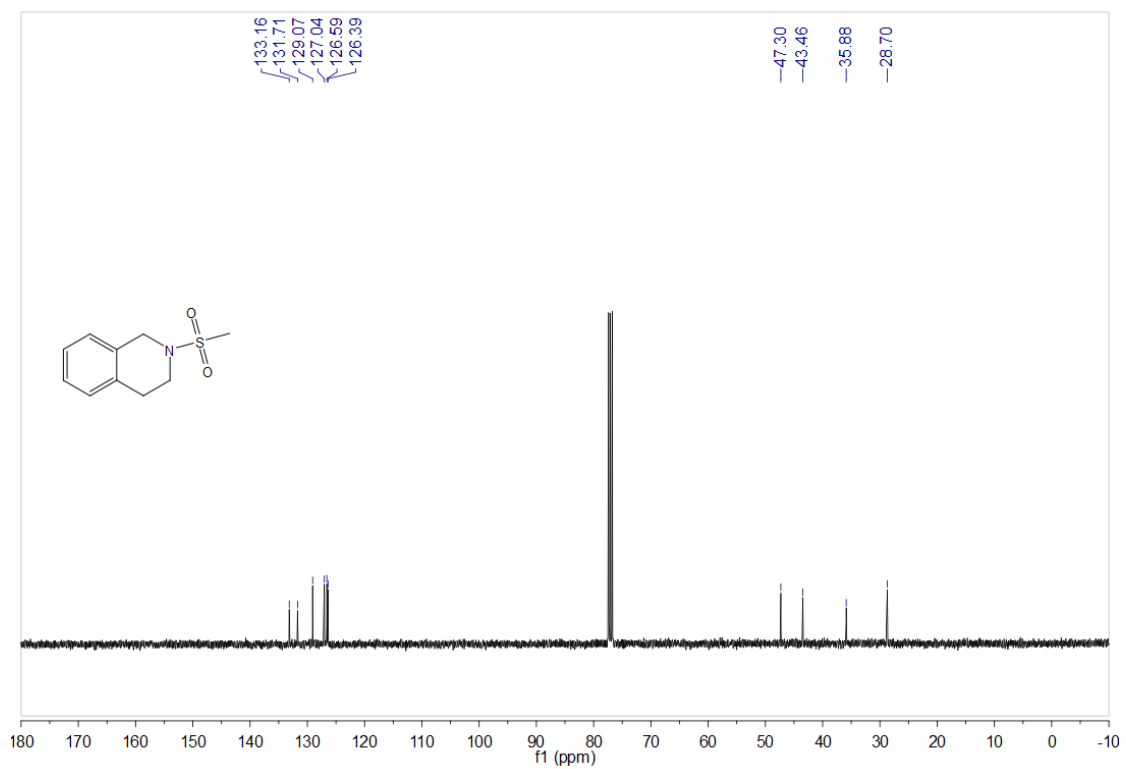
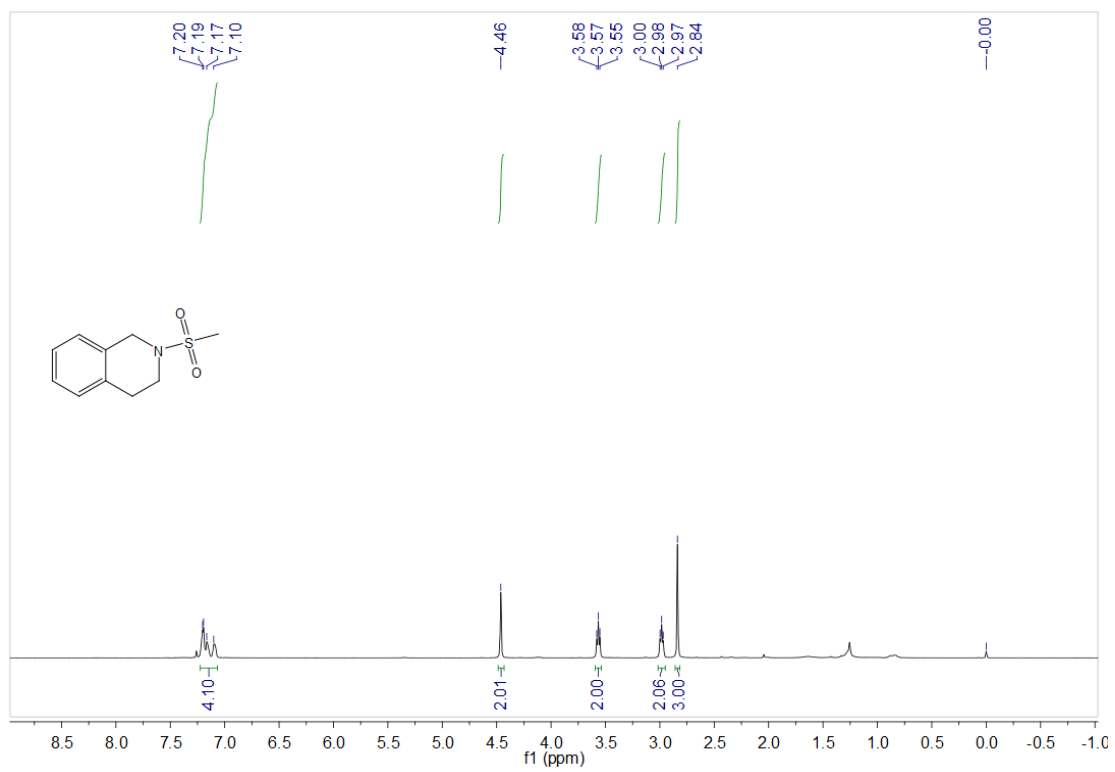
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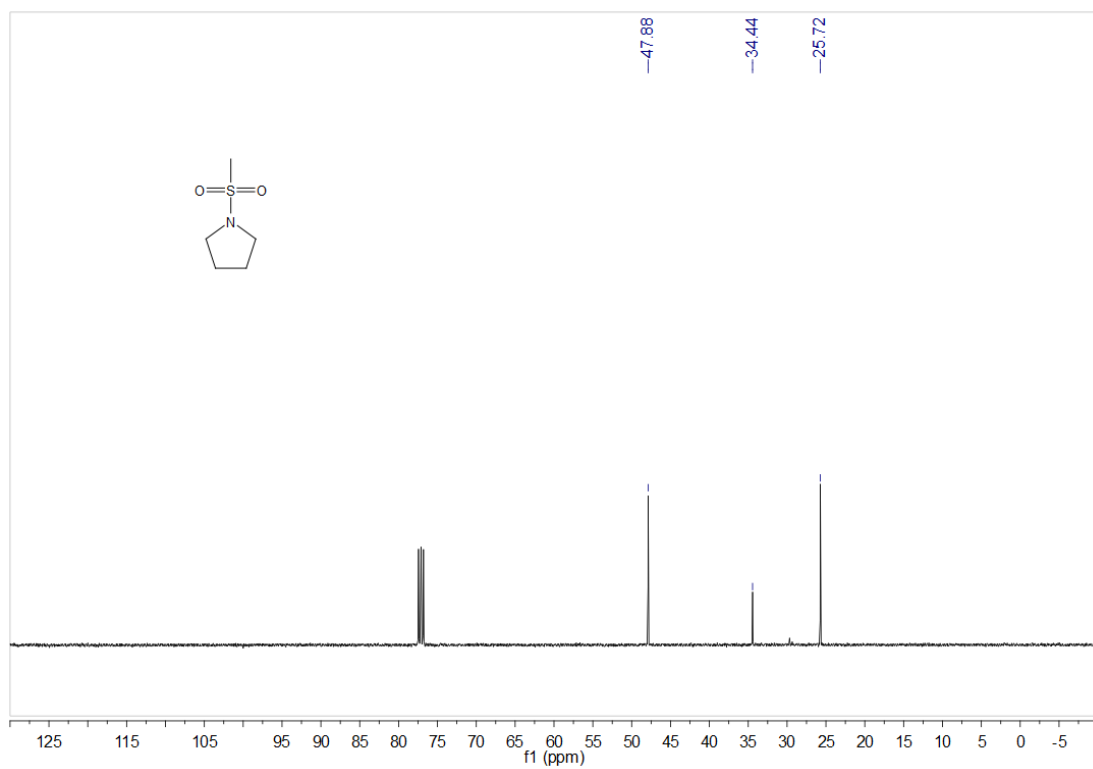
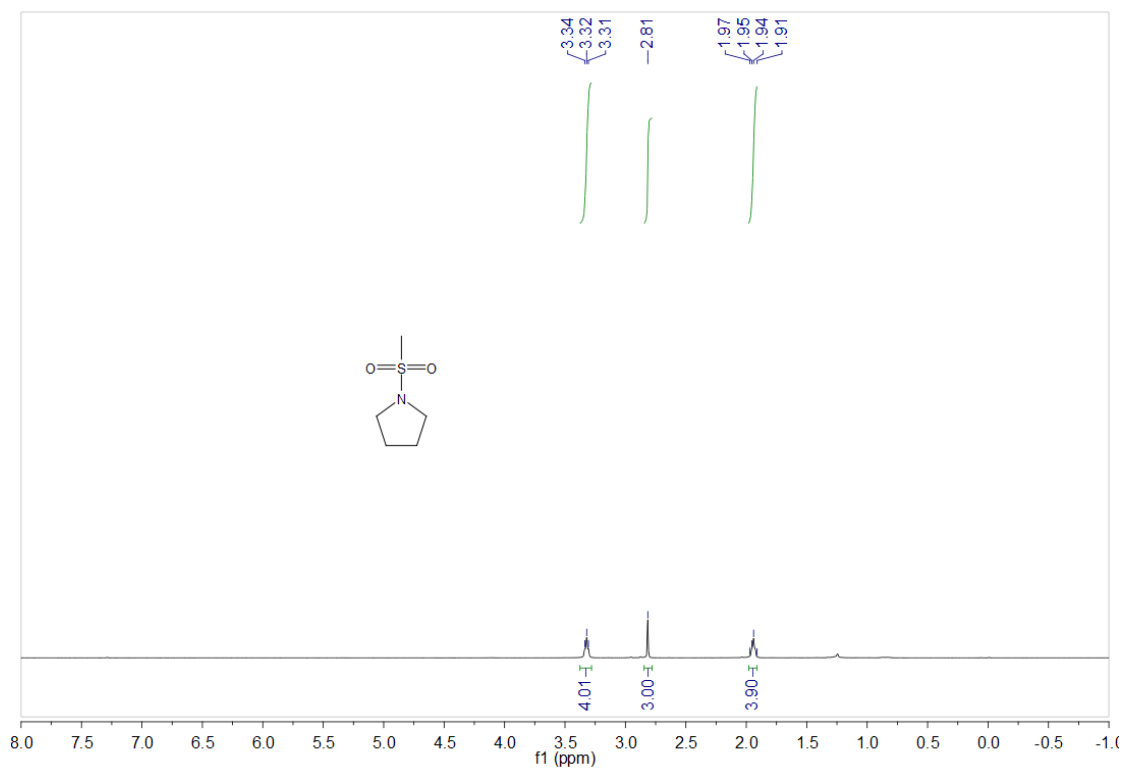
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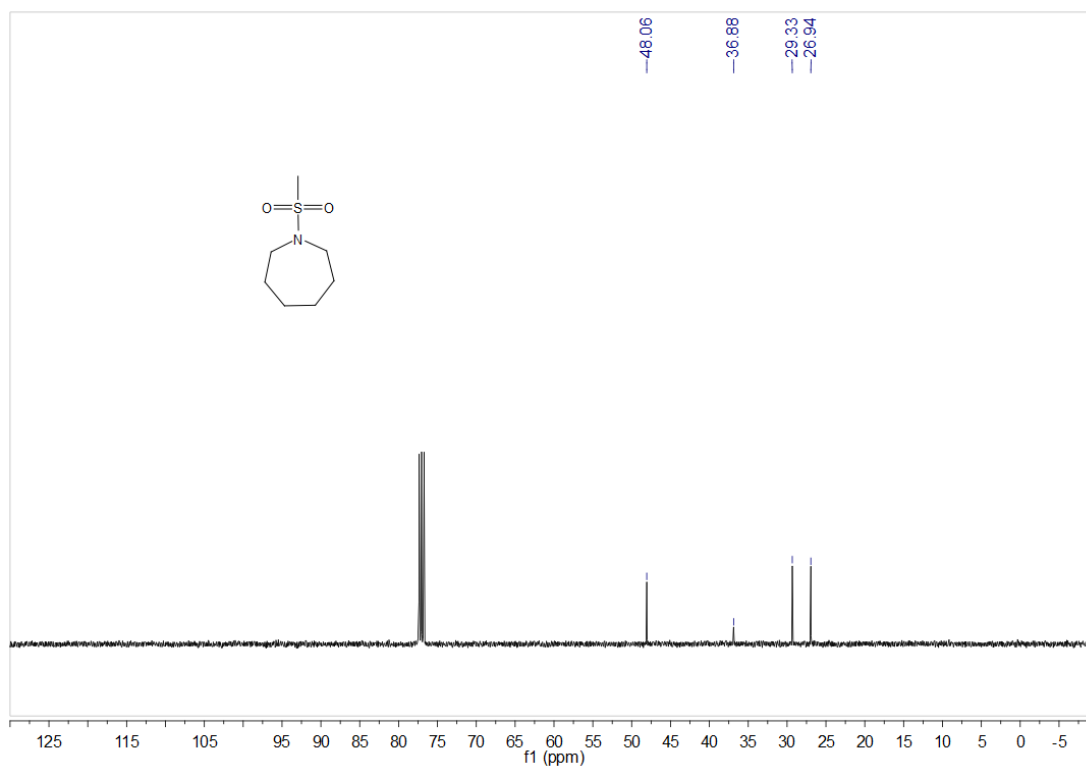
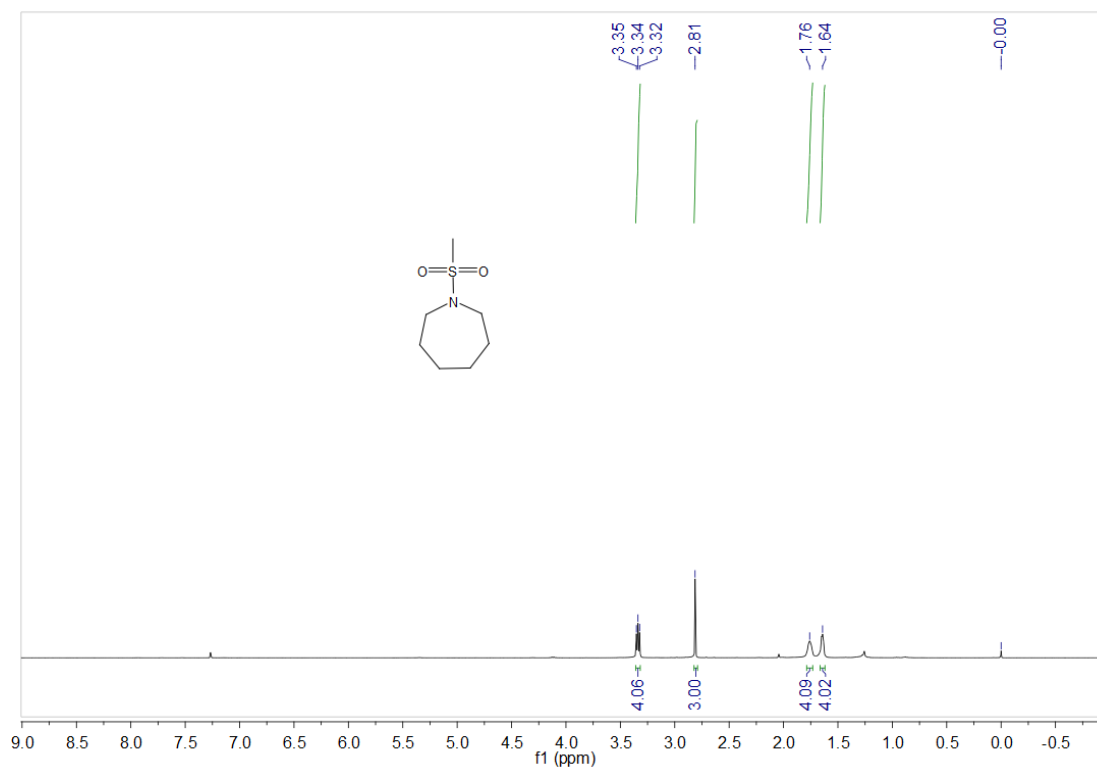
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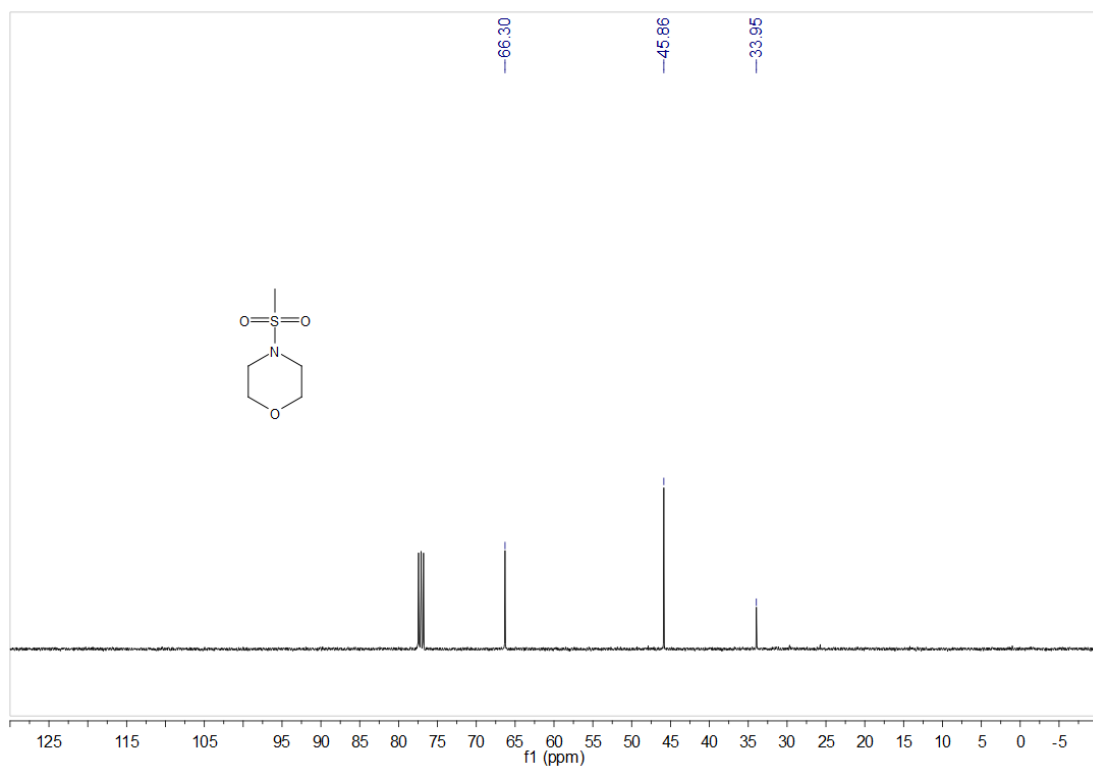
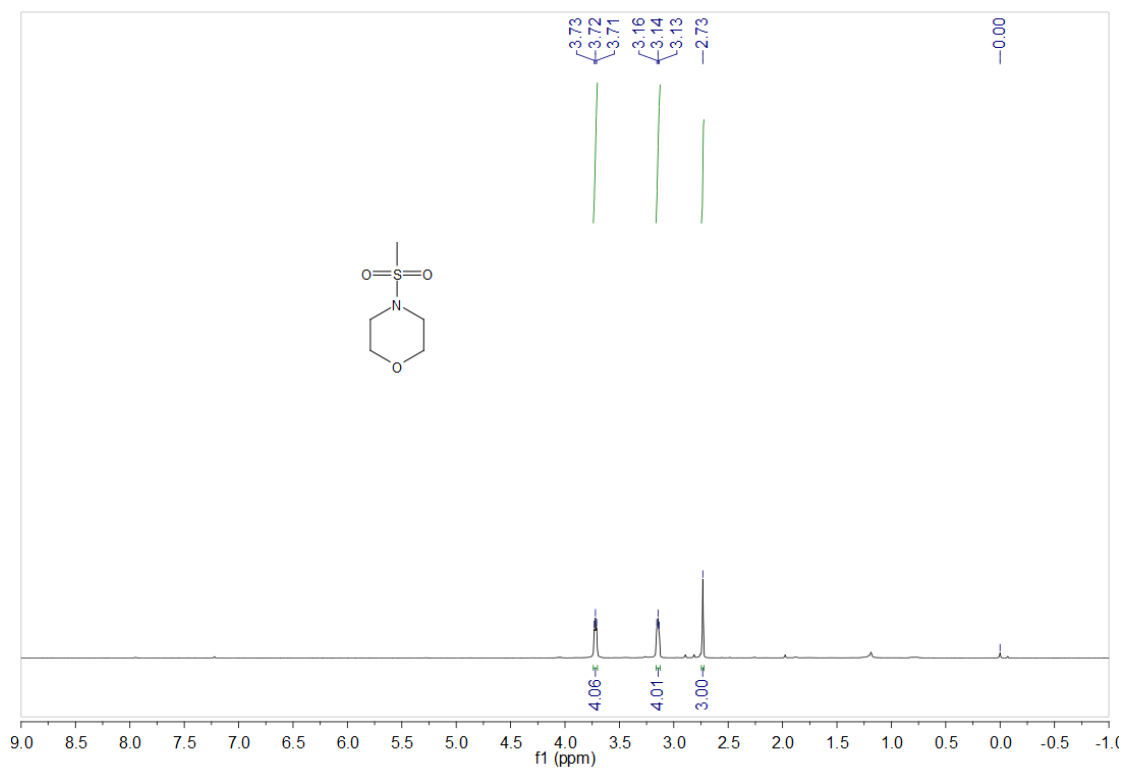
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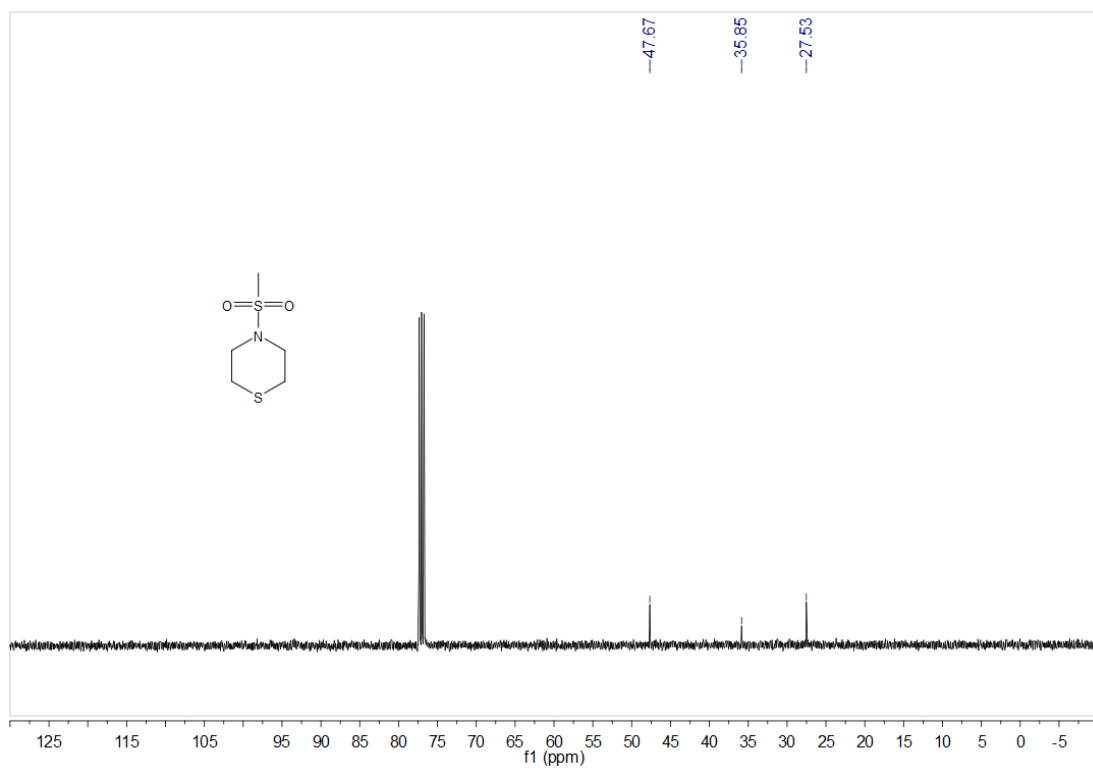
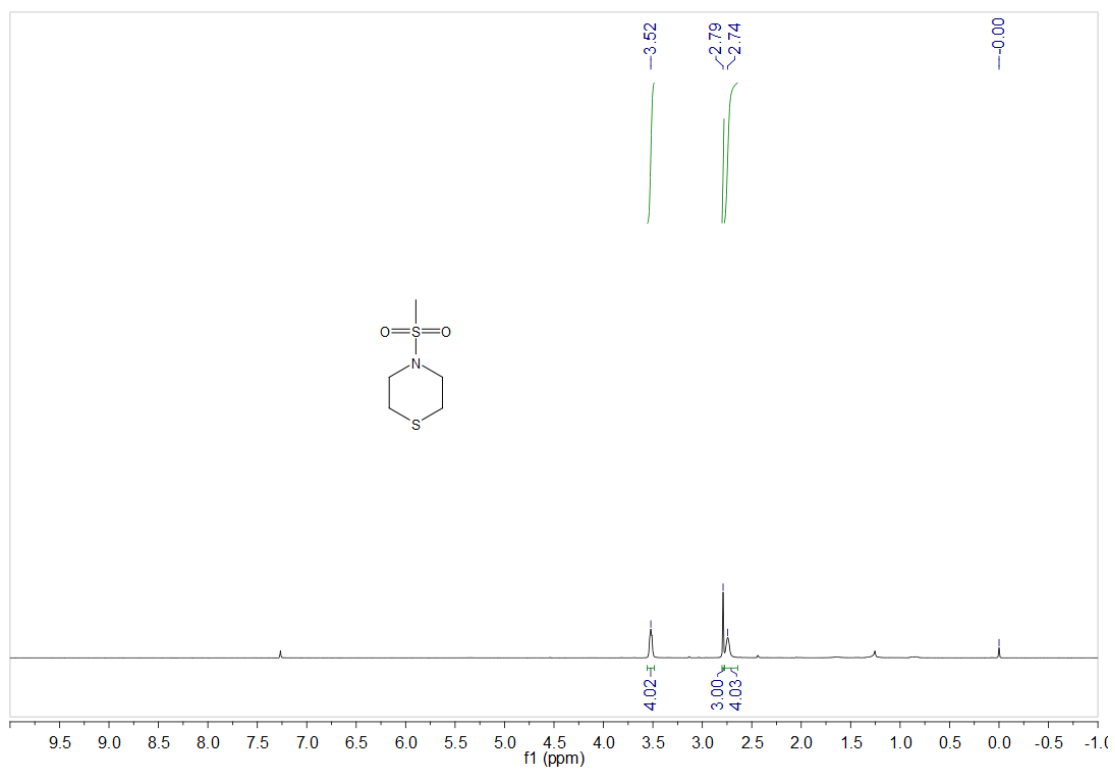
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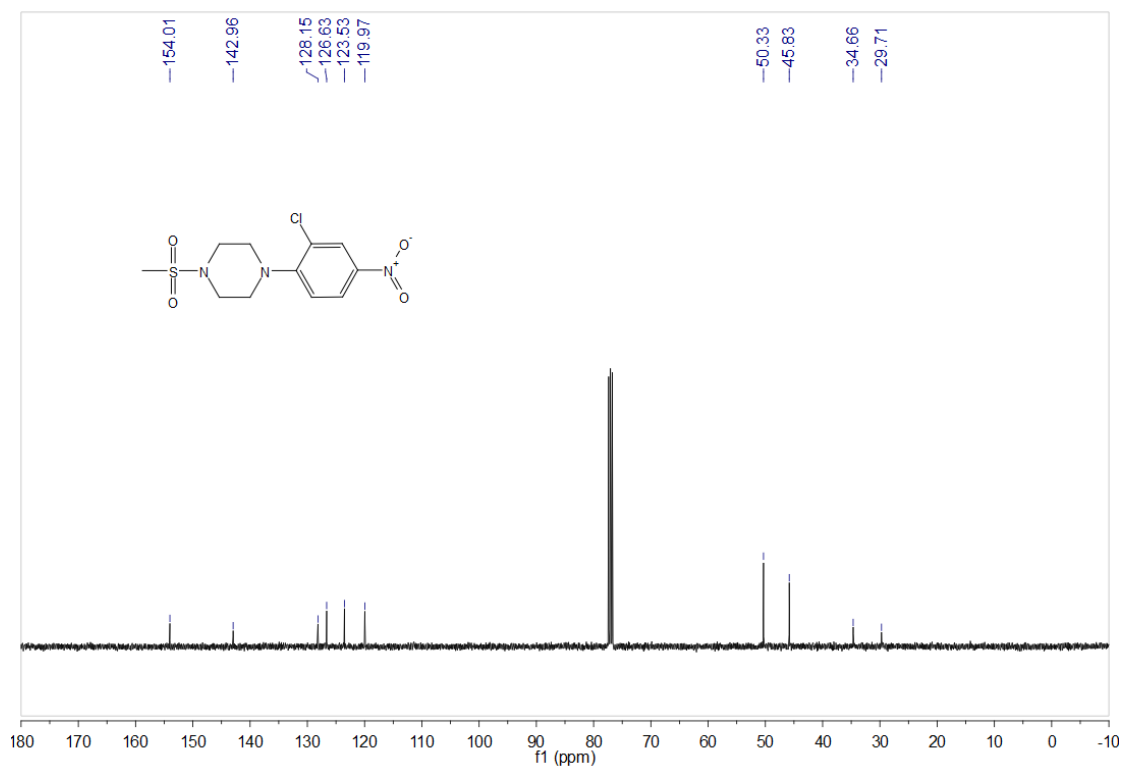
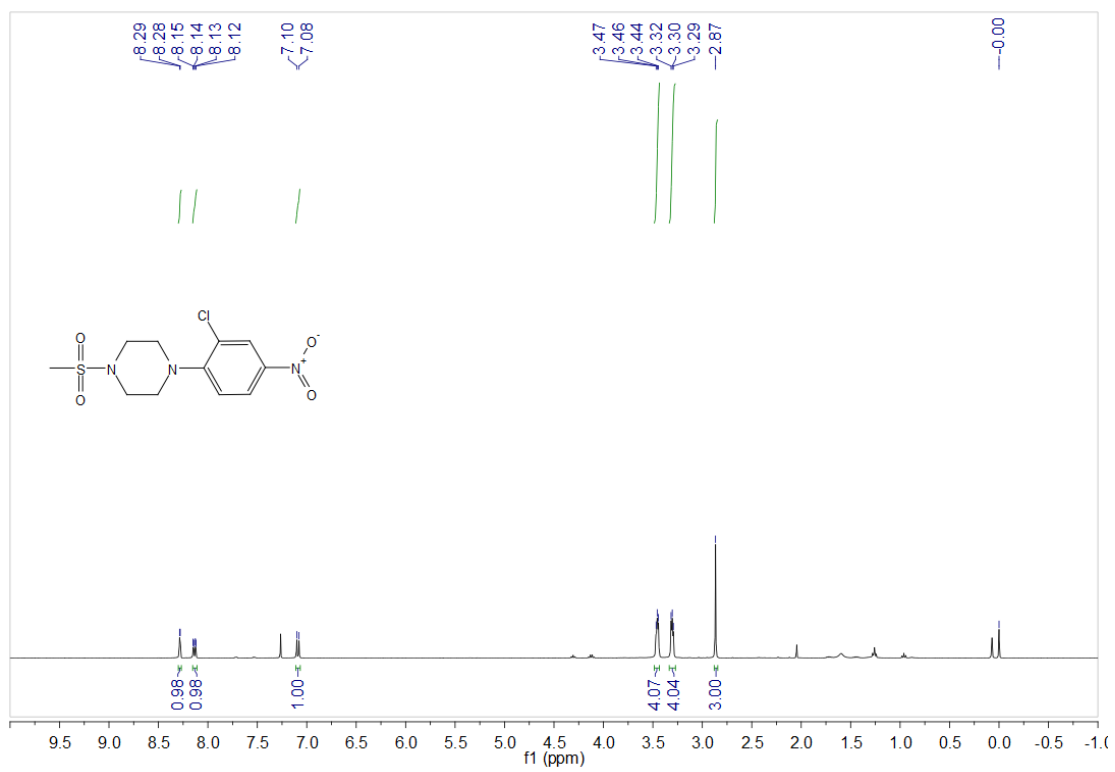
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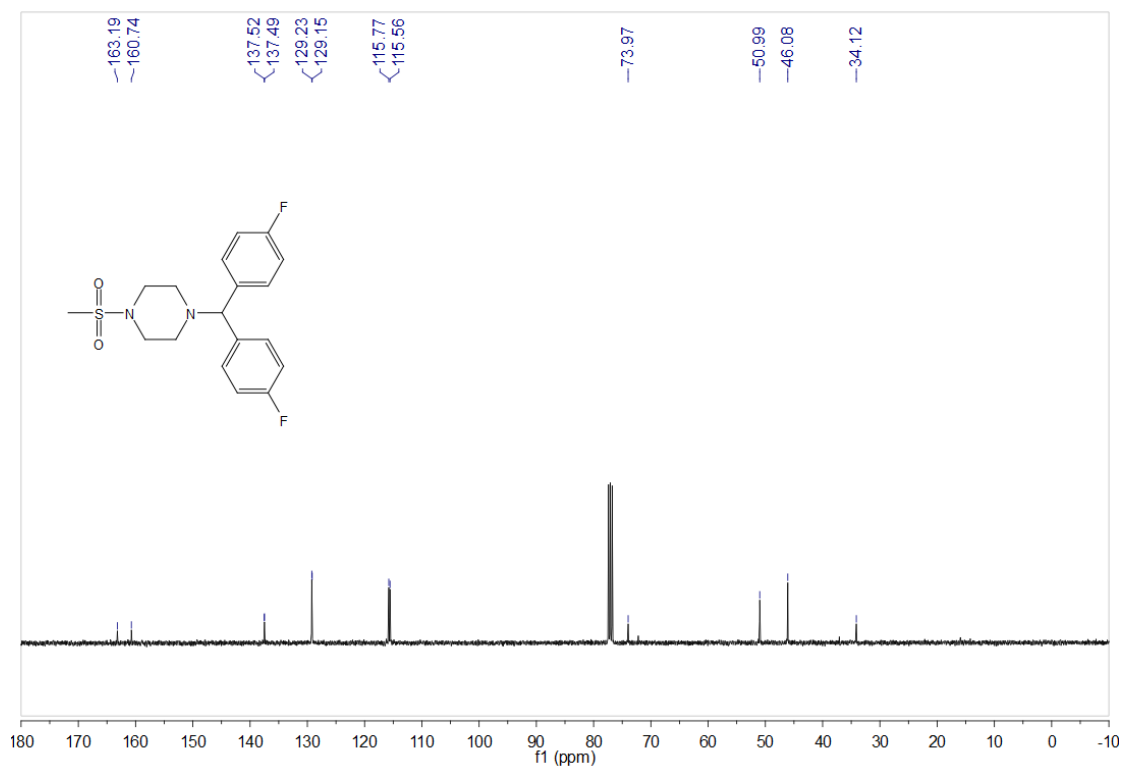
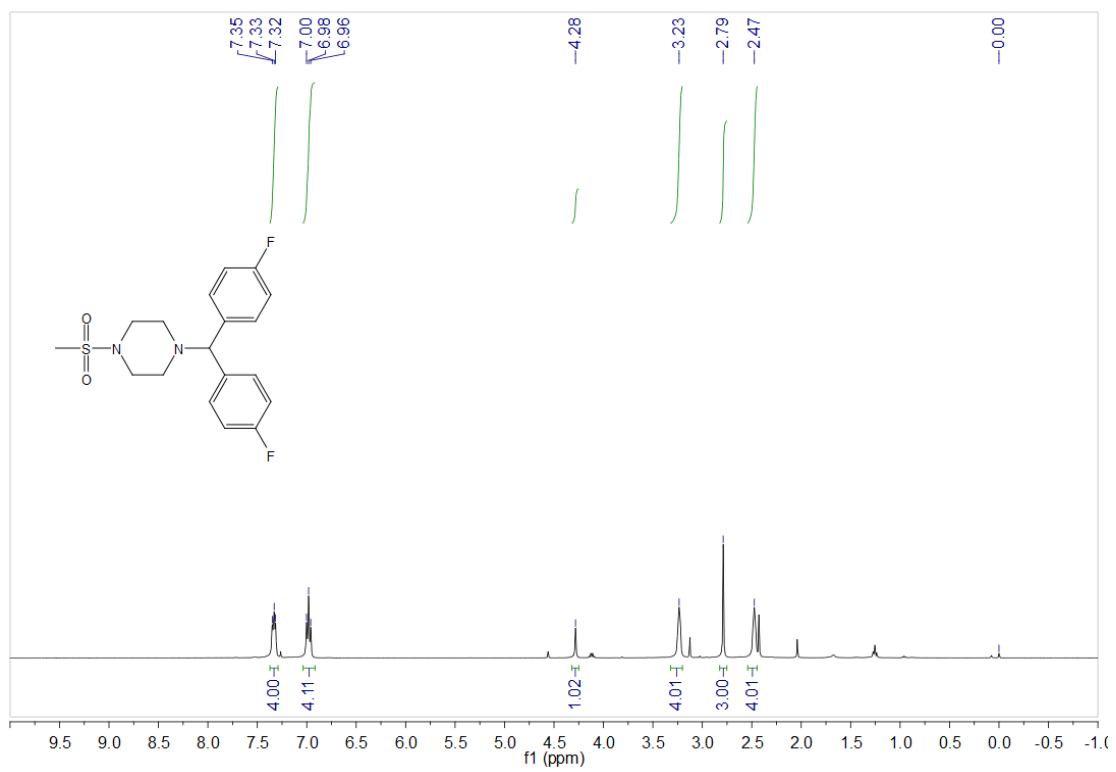
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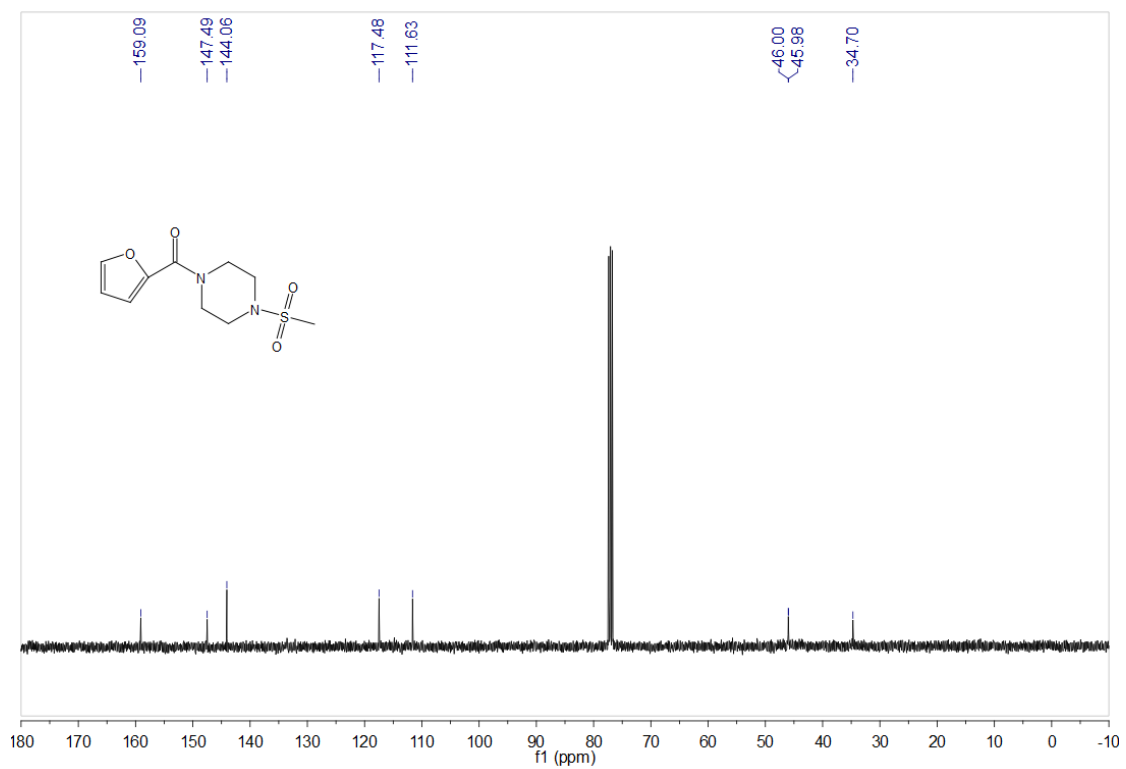
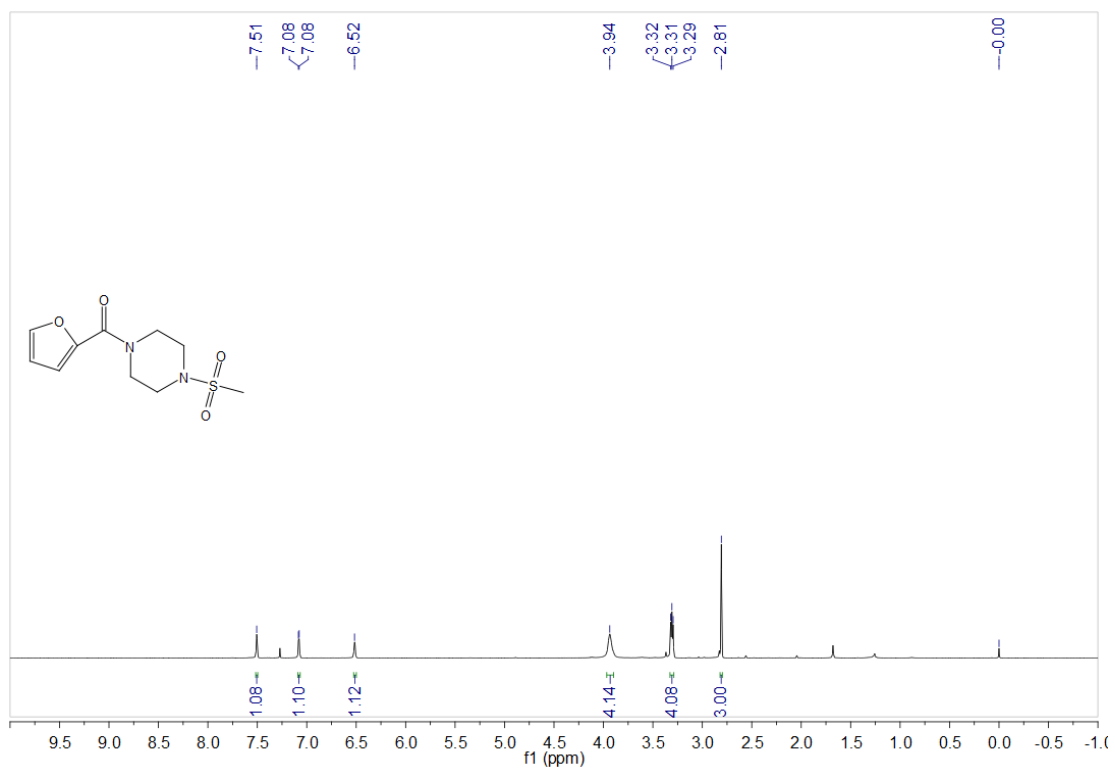
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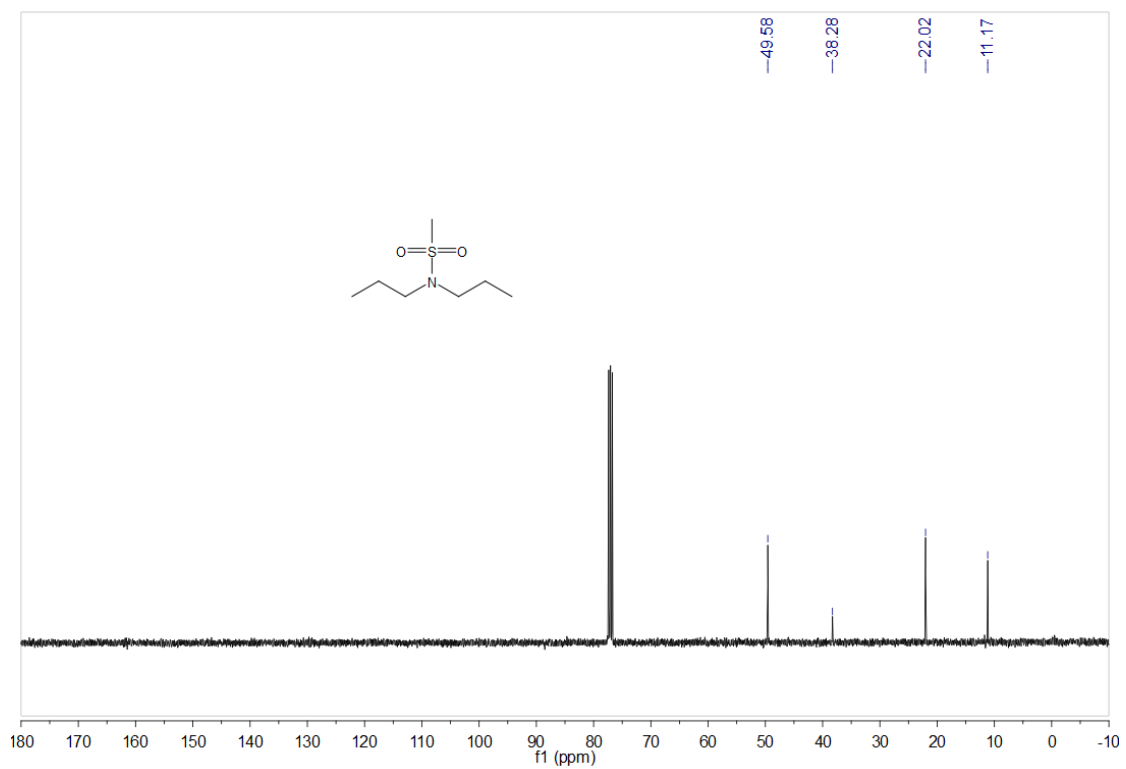
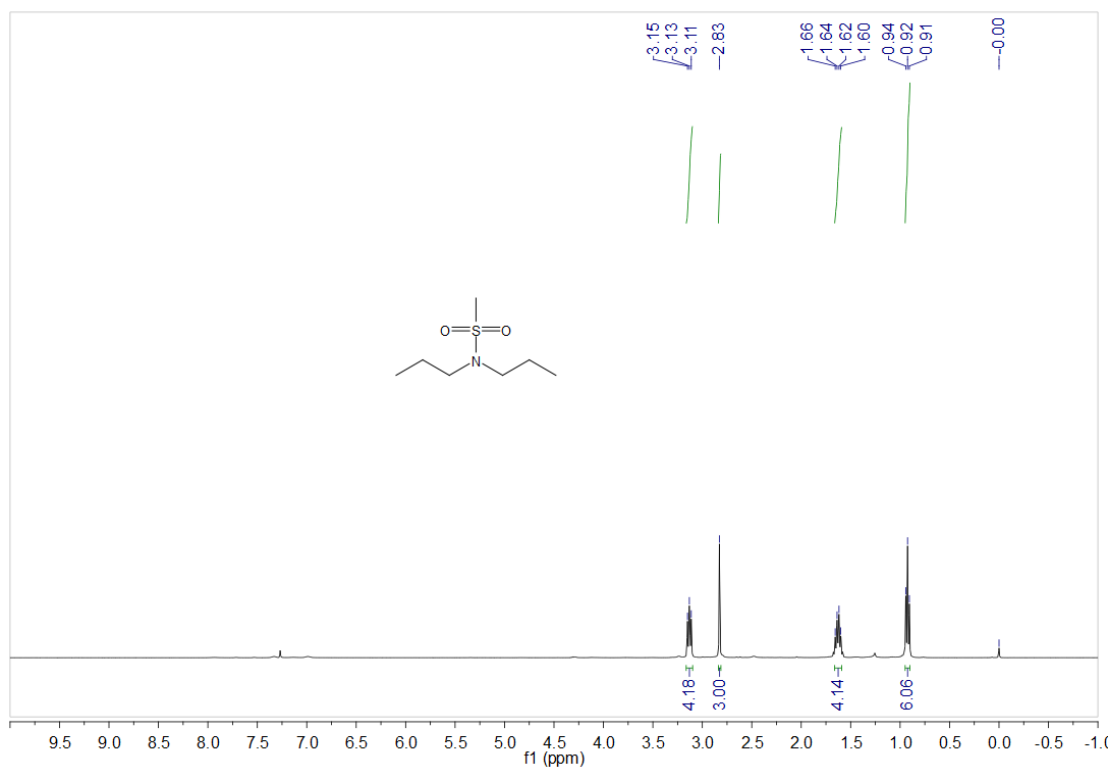
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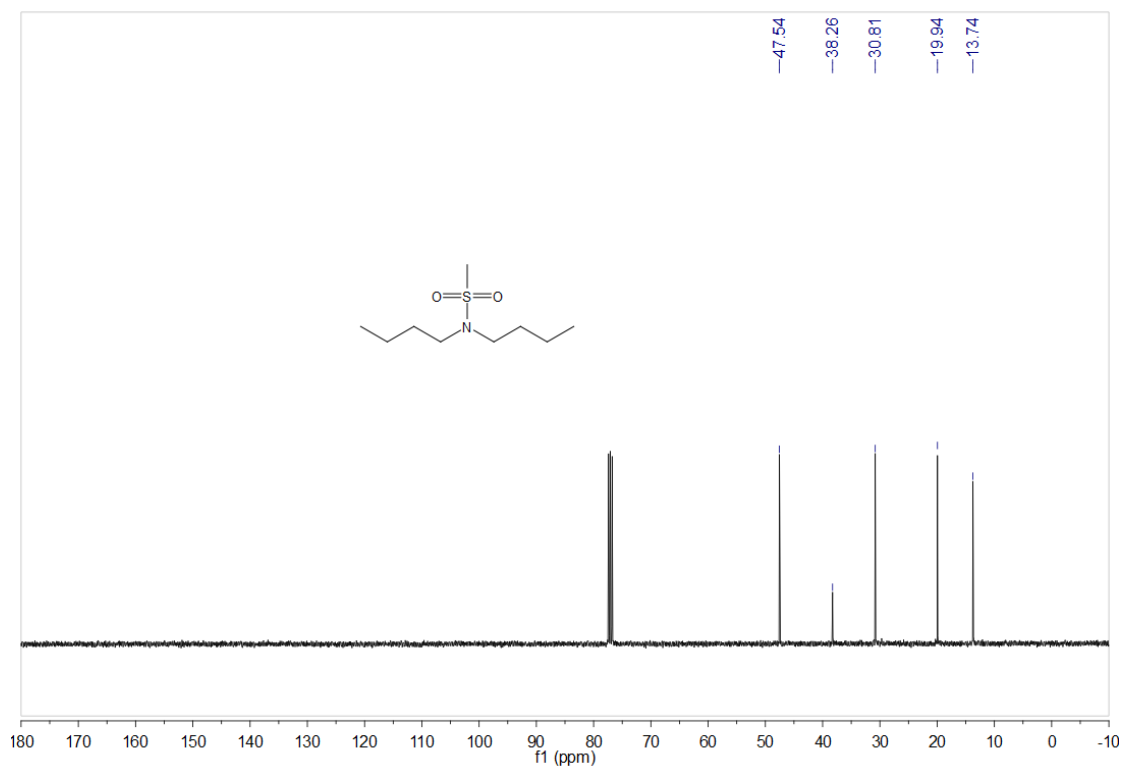
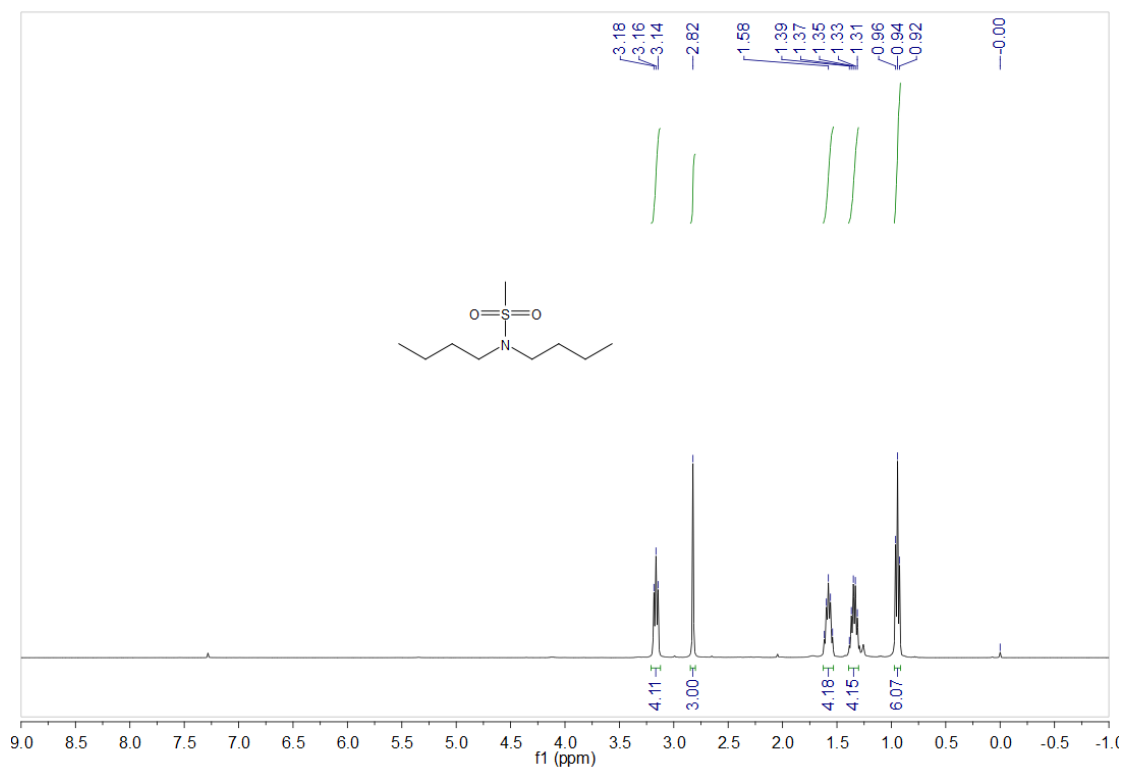
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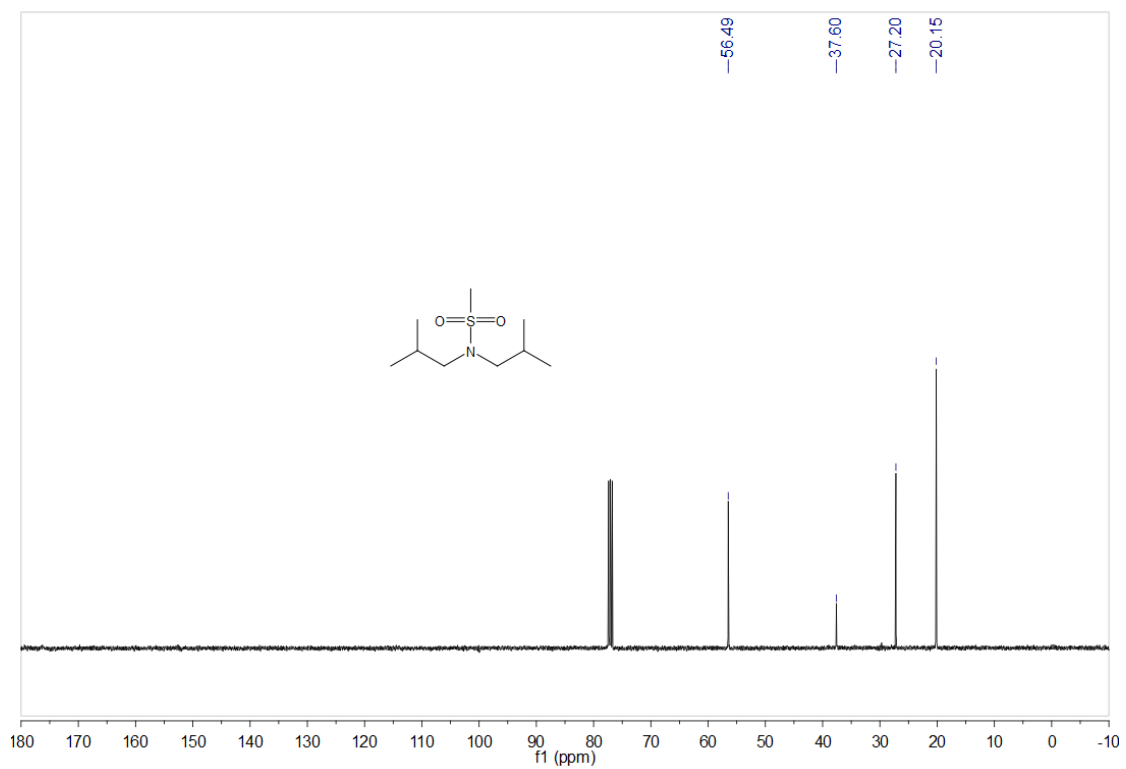
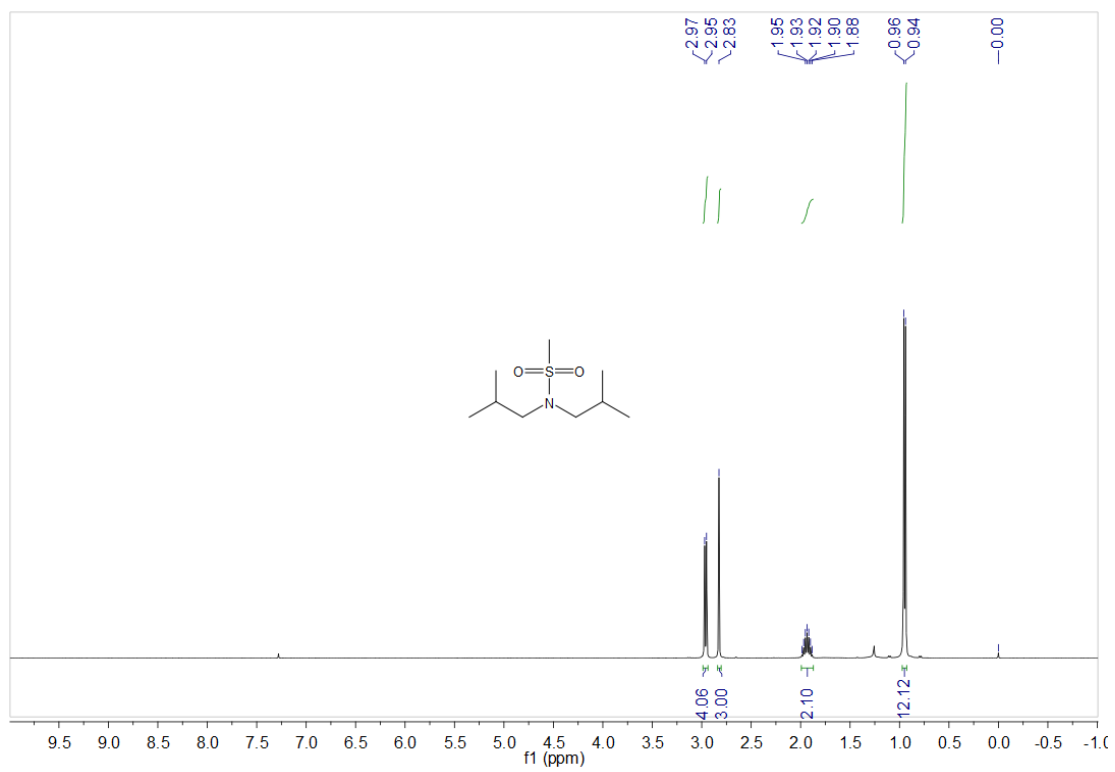
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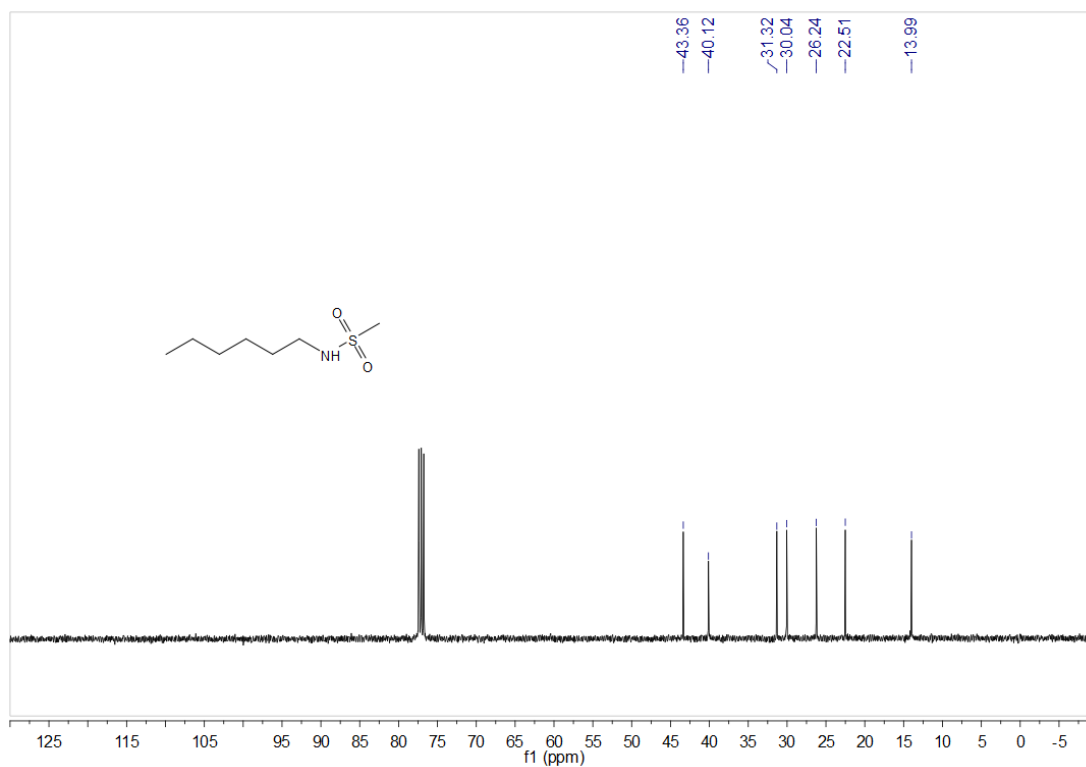
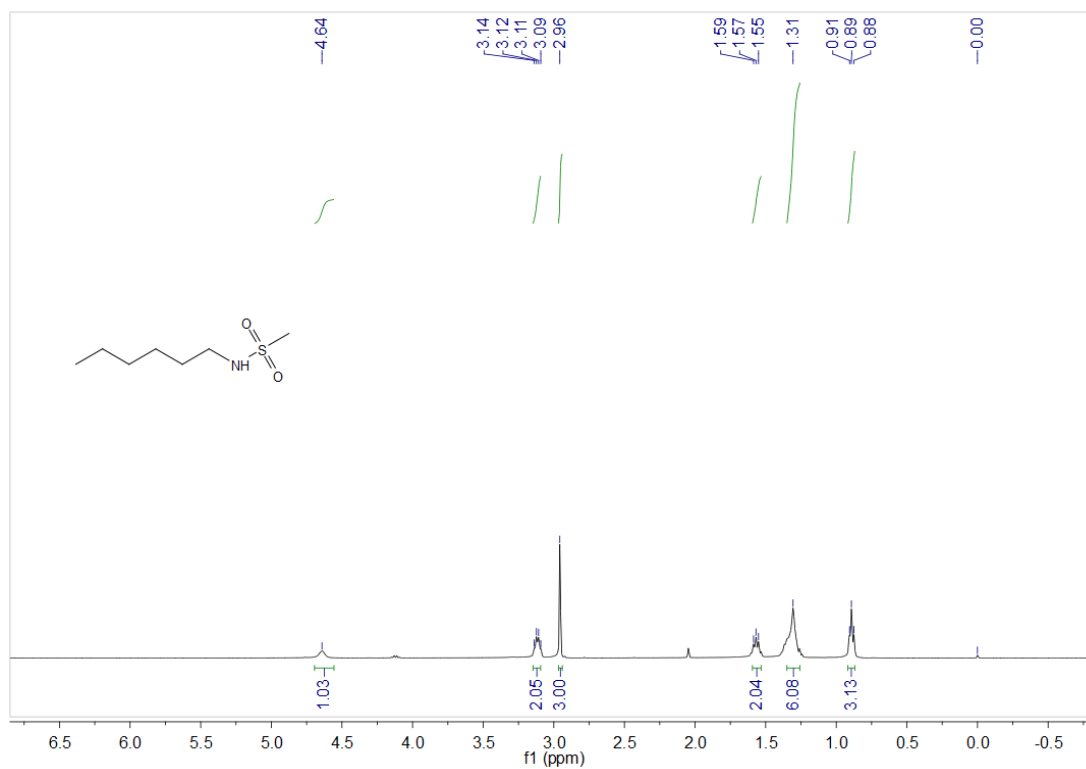
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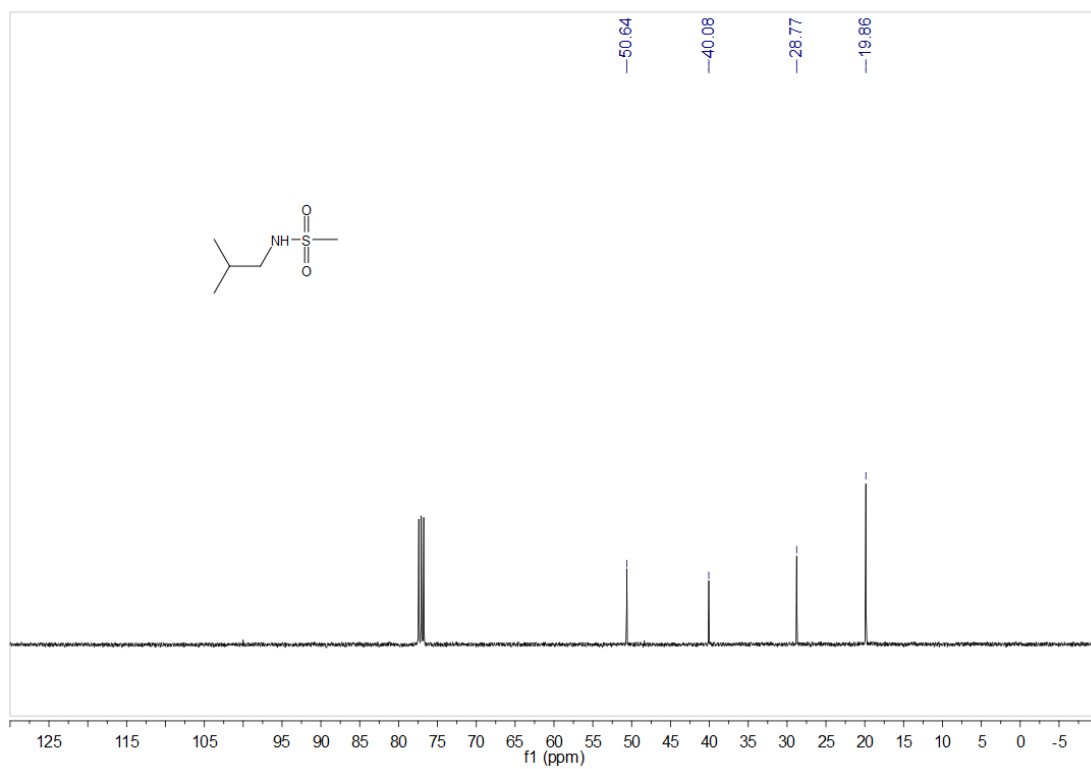
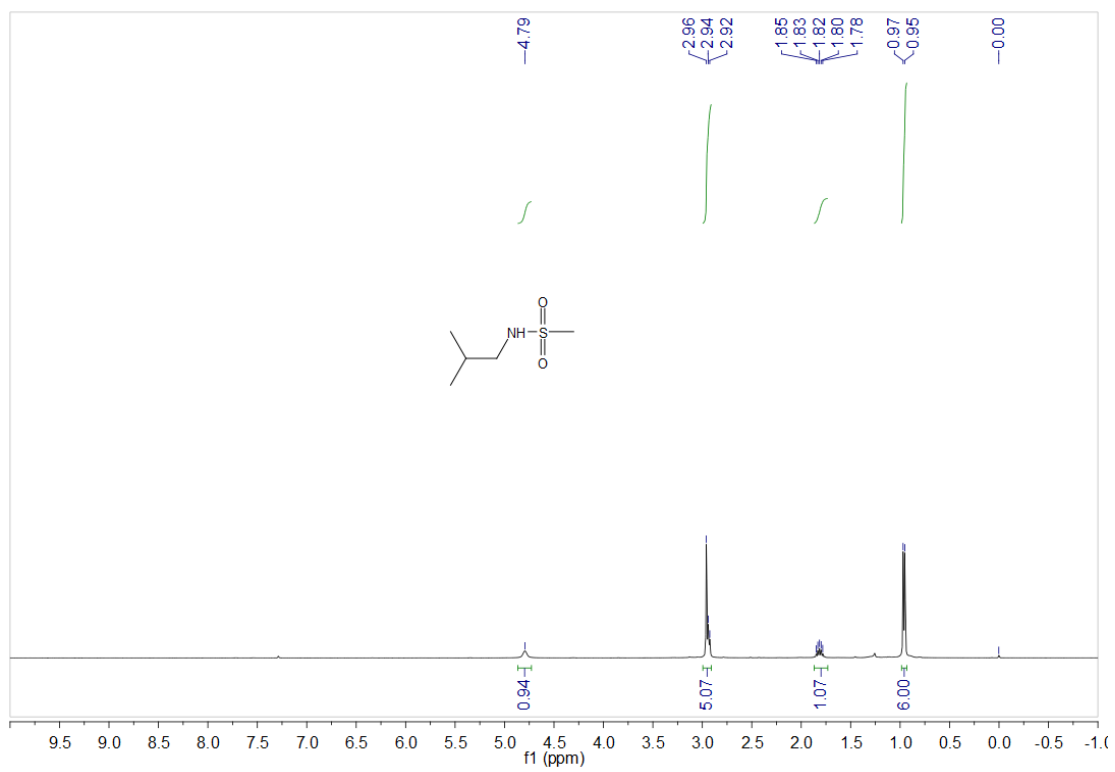
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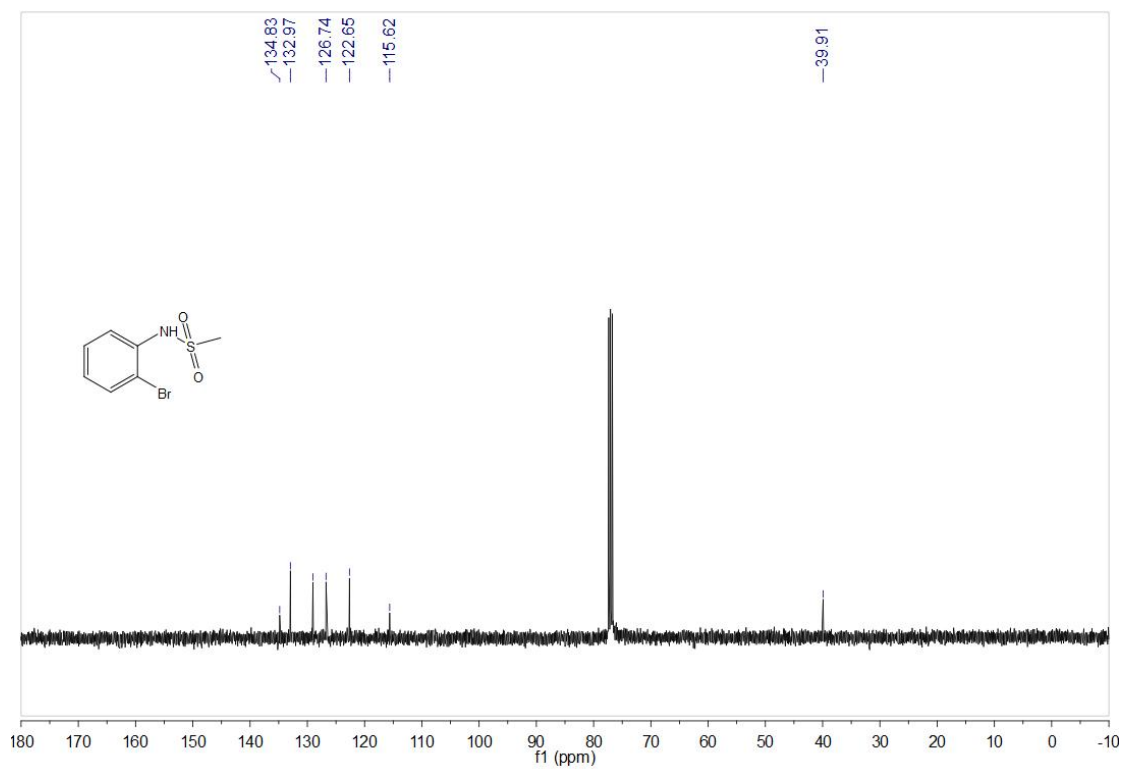
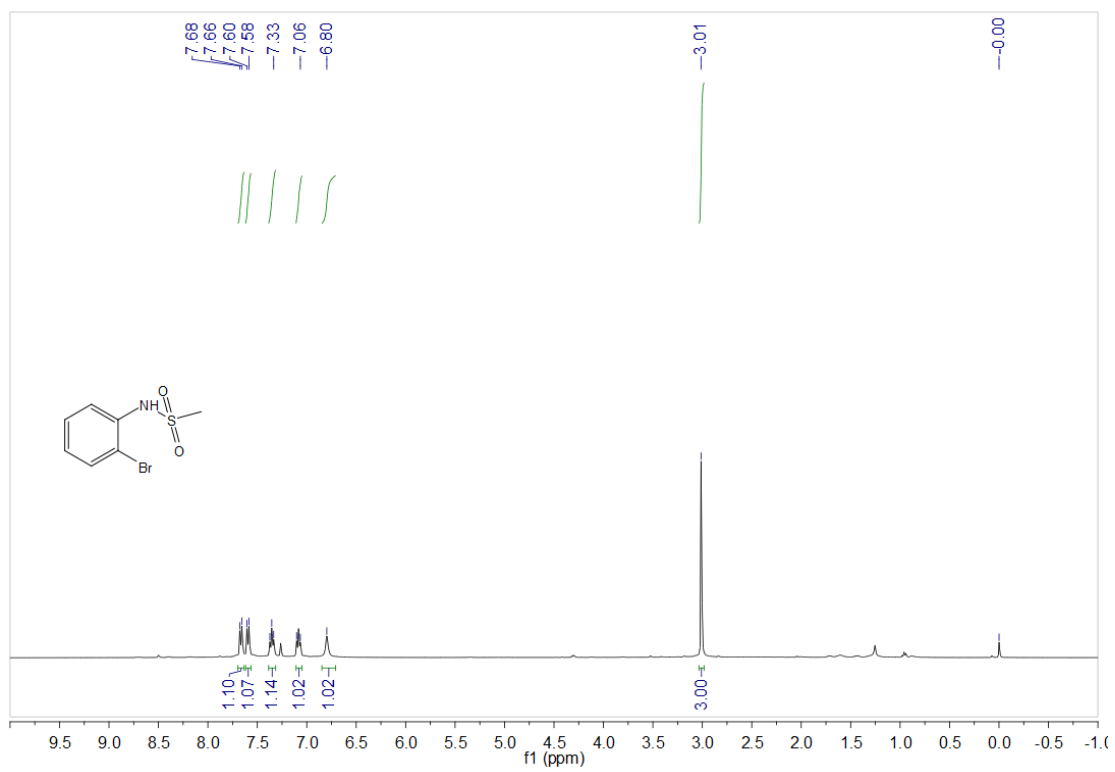
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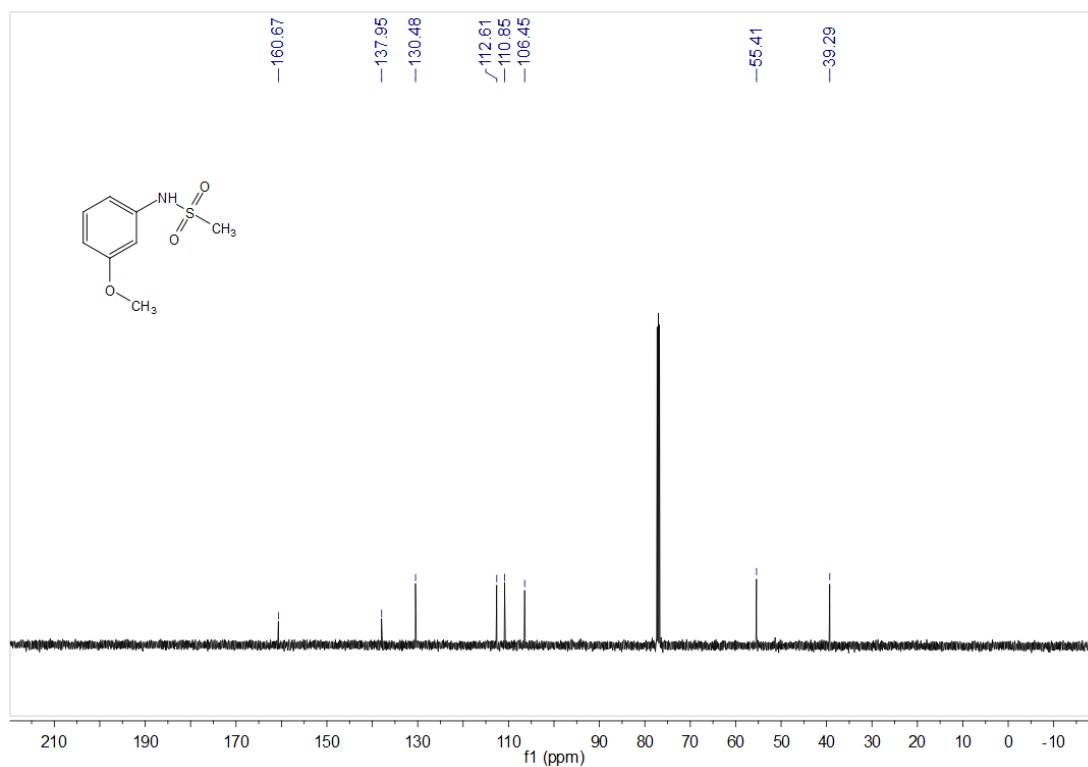
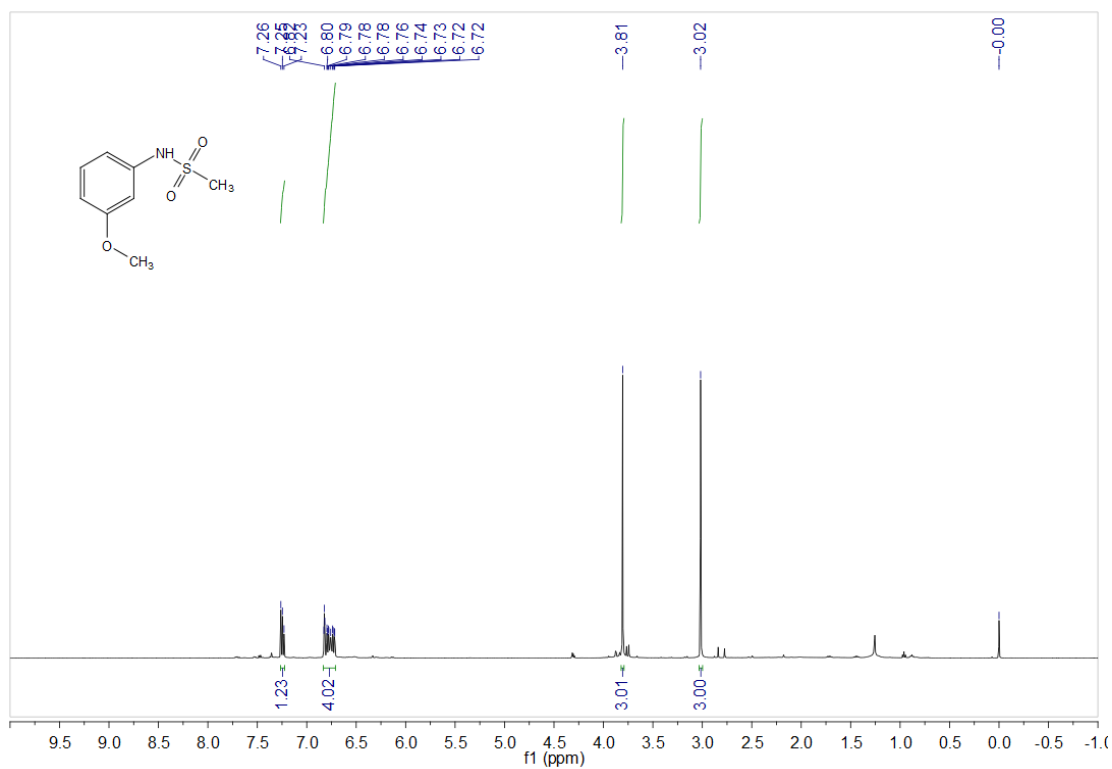
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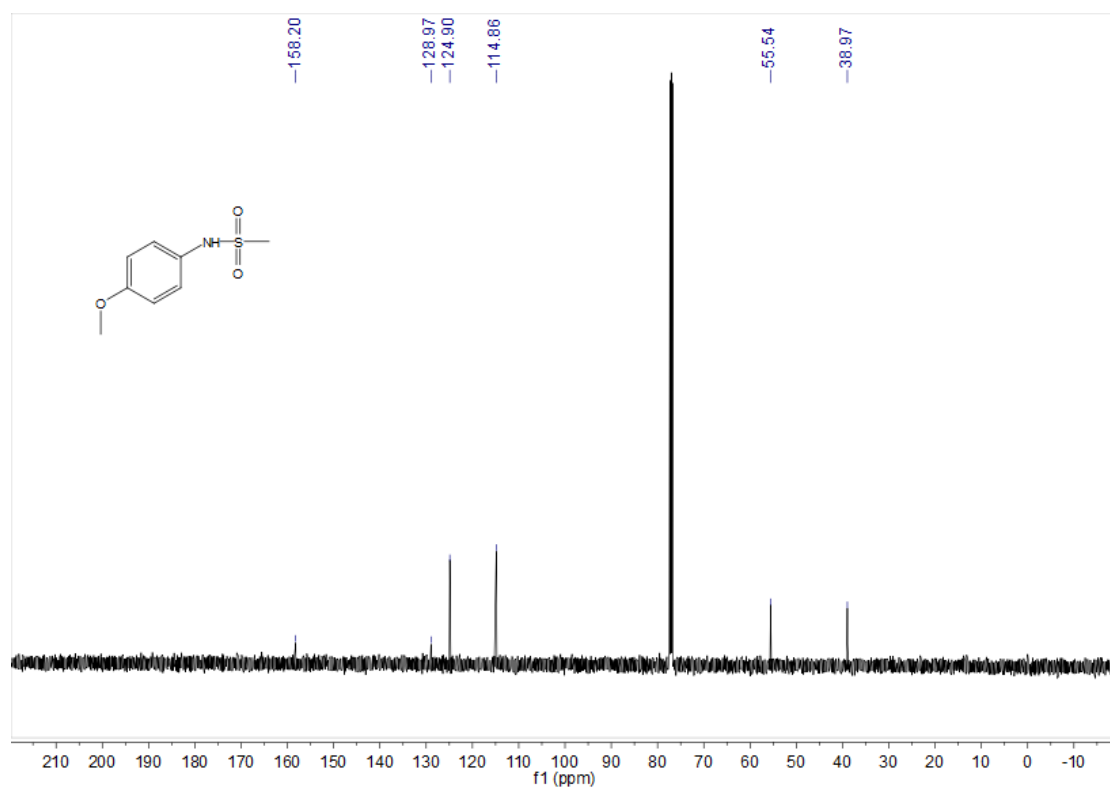
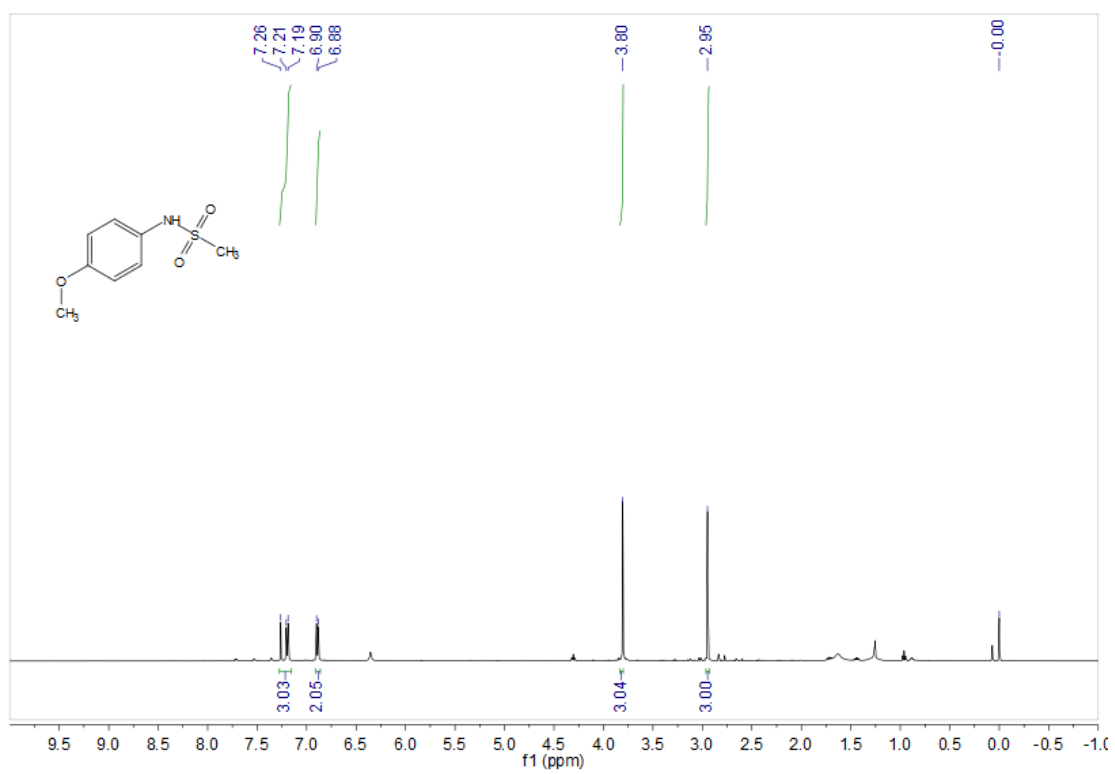
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3z₂



3z₃



5a

