Supplementary Information

Metal-free I_2O_5-mediated direct construction of sulfonamides from thiols and amines

Minghui Zhu, Wei Wei,* Daoshan Yang, Huanhuan Cui, Leilei Wang, Hua Wang*

The Key Laboratory of Life-Organic Analysis and Key Laboratory of Pharmaceutical Intermediates and Analysis of Natural Medicine, School of Chemistry and Chemical Engineering, Qufu Normal University, Qufu 273165, Shandong, China

**Phone:+86 537 4458317; Fax:+86 537 4458317.*

E-mail: weiweiqfnu@163.com; huawang_qfnu@126.com

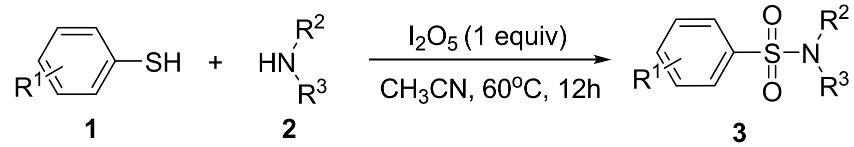
Contents

1. General information

All commercially available reagent grade chemicals were purchased from Aldrich, Acros, Alfa Aesar and Beijing Ouhe Chemical Company and used as received without further purification unless otherwise stated. ^{1}H NMR, and ^{13}C NMR were recorded in $CDCl_3$ on a Bruker Avance 500 spectrometer with TMS as internal standard (500 MHz ^{1}H, 125 MHz ^{13}C) at room temperature, the chemical shifts (δ) were expressed in ppm and J values were given in Hz. The following abbreviations are used to indicate the multiplicity: singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of triplets (dt), and multiplet (m). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m). Mass analyses and HRMS were obtained on a Finnigan-LCQDECA mass spectrometer and a Bruker Daltonics Bio-TOF-Q mass spectrometer by the ESI method, respectively. Column chromatography was performed on silica gel (200-300 mesh).

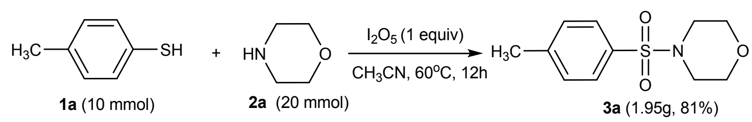
2. General procedure for I_2O_5-mediated direct construction of sulfonamides from thiols and amines.

2.1



In a tube (25ml), arylthiol **1** (0.2 mmol), amine **2** (0.4 mmol), I_2O_5 (0.2 mmol), and CH_3CN (2 mL) were added. Subsequently, the tube was sealed and the reaction vessel was allowed to stir at 60 °C for 12 h. After completion of the reaction, the reaction mixture was concentrated in vaccum. The resulting mixture purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3**.

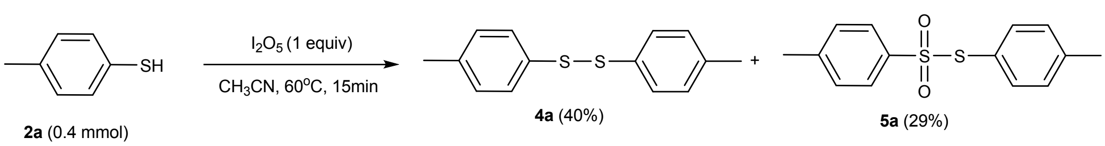
2.2. Gram-scale reaction



In a tube (100 ml), 4-methylbenzenethiol **1a** (10 mmol), morpholine **2a** (20 mmol) I_2O_5 (10 mmol), and CH_3CN (20 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 60 °C for 12h. After completion of the reaction, the resulting mixture was concentrated under vacuum and the residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3a** in 81% yield (1.95g).

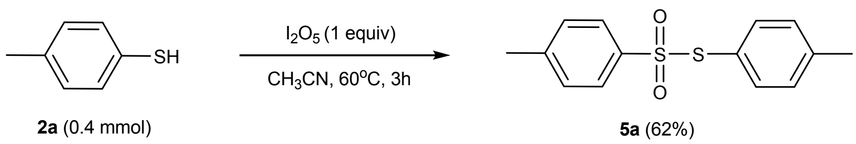
3. Preliminary mechanistic studies

3.1 The reaction of 4-methylbenzenethiol (**2a**) with I_2O_5 (15 min).



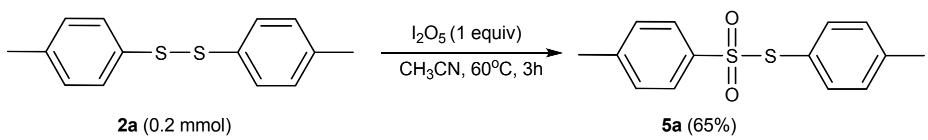
In a seal tube (15 mL), 4-methylbenzenethiol **2a** (0.4 mmol), I_2O_5 (0.2 mmol), and CH_3CN (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 60 °C for 15 min. After completion of the reaction, the solution was concentrated in vacuum, the product **4a** and **5a** were isolated in 40% and 29% yields, respectively.

3.2 The reaction of 4-methylbenzenethiol (**2a**) with I_2O_5 (3h).



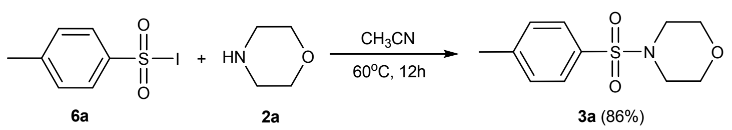
In a seal tube (15 mL), 4-methylbenzenethiol **2a** (0.4 mmol), I_2O_5 (0.2 mmol), and CH_3CN (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 60 °C for 3h. After completion of the reaction, the solution was concentrated in vacuum, the product **5a** was isolated in 62% yield.

3.3 The reaction of 1,2-dip-tolyldisulfane (**4a**) with I_2O_5.



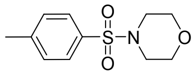
In a seal tube (15 mL), 1,2-dip-tolyldisulfane (**4a**) (0.2 mmol), I_2O_5 (0.2 mmol), and CH_3CN (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 60 °C for 3h. After completion of the reaction, the solution was concentrated in vacuum, the product **5a** were isolated in 65% yield.

3.4 The reaction of sulfonyl iodide **6a** with morpholine **2a**.

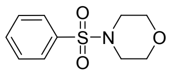


In a seal tube (15 mL), 4-methylbenzene-1-sulfonyl iodide (0.2 mmol), morpholine **2a** (0.4 mmol) and CH_3CN (2 mL) were added. The reaction vessel was allowed to stir at 60 °C for 12h. After completion of the reaction, the resulting mixture was concentrated under vacuum and the residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3a** in 86% yield.

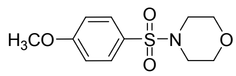
4. Characterization data of products

4-tosylmorpholine 

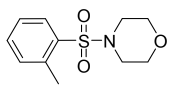
Compound **3a** was obtained in 82% yield according to the general procedure. ^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 7.66 (d, J = 8.3 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 3.76 (t, J = 4.7 Hz, 4H), 3.00 (t, J = 4.8 Hz, 4H), 2.47 (s, 3H); ^{13}C NMR (CDCl$_3$, 125MHz, ppm): δ 143.9, 132.1, 129.7, 127.9, 66.1, 46.0, 21.6; HRMS calc. for $C_{11}H_{15}NO_3SNa$ (M+Na)$^+$, 264.0670; found, 264.0675.

4-(phenylsulfonyl)morpholine 

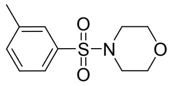
Compound **3b** was obtained in 81% yield according to the general procedure. ^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 7.77-7.75 (m, 2H), 7.65-7.62 (m, 1H), 7.58-7.55 (m, 2H), 3.75 (t, J = 4.7 Hz, 4H), 3.01 (t, J = 4.8 Hz, 4H); ^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 135.1, 133.1, 129.1, 127.8, 66.1, 46.0; HRMS calc. for $C_{10}H_{13}NO_3SNa$ (M+Na)$^+$, 250.0514; found, 250.0517.

4-(4-methoxyphenylsulfonyl)morpholine 

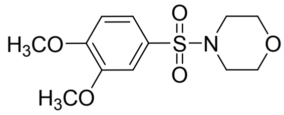
Compound **3c** was obtained in 85% yield according to the general procedure. 1H NMR (CDCl3, 500 MHz, ppm): δ 7.70-7.69 (m, 2H), 7.02-7.01 (m, 2H), 3.89 (s, 3H), 3.74 (t, J = 4.7 Hz, 4H), 2.98 (t, J = 4.8 Hz, 4H); ^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 163.2, 130.0, 126.7, 114.3, 66.1, 55.7, 46.0; HRMS calc. for $C_{11}H_{15}NO_4SNa$ (M+Na)+, 280.0619; found, 280.0622.

4-(o-tolylsulfonyl)morpholine 

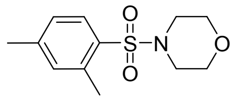
Compound **3d** was obtained in 88% yield according to the general procedure. ^{1}H NMR (CDCl$_3$, 500 MHz, ppm): δ 7.91-7.89 (m, 1H), 7.50-7.47 (m, 1H), 7.35-7.32 (m, 2H), 3.72 (t, J = 4.7 Hz, 4H), 3.15 (t, J = 4.9 Hz, 4H), 2.65 (s, 3H); ^{13}C NMR (CDCl$_3$, 125 MHz, ppm): δ 138.2, 135.0, 133.1, 132.9, 130.4, 126.2, 66.3, 45.3, 20.9; HRMS calc. for $C_{11}H_{15}NO_3SNa$ (M+Na)$^+$, 264.0670; found, 264.0671.

4-(m-tolylsulfonyl)morpholine 

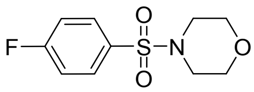
Compound **3e** was obtained in 85% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.56-7.55 (m, 2H), 7.46-7.43 (m, 2H), 3.75 (t, J = 4.8 Hz, 4H), 3.00 (t, J = 4.8 Hz, 4H), 2.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 125MHz, ppm): δ 139.4, 134.9, 133.9, 129.0, 128.1, 125.0, 66.1, 46.0, 21.4; HRMS calc. for $C_{11}H_{15}NO_3SNa$ $(M+Na)^+$, 264.0670; found, 264.0673.

4-(3,4-dimethoxyphenylsulfonyl)morpholine 

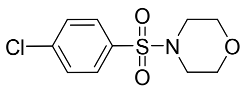
Compound **3f** was obtained in 81% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.39-7.36 (m, 1H), 7.21 (d, J = 2.1 Hz, 1H), 6.98 (d, J = 8.5 Hz, 1H), 3.96 (s, 3H), 3.94 (s, 3H), 3.75 (t, J = 4.7 Hz, 4H), 3.00 (t, J = 4.7 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 152.9, 152.9, 149.2, 149.1, 126.8, 121.8, 110.7, 110.7, 110.4, 110.3, 66.1, 56.3, 56.2, 46.0; HRMS calc. for $C_{12}H_{17}NO_5SNa$ $(M+Na)^+$, 310.0725; found, 310.0728.

4-(2,4-dimethylphenylsulfonyl)morpholine 

Compound **3g** was obtained in 67% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.77 (d, J = 7.8 Hz, 1H), 7.13 (d, J = 8.7 Hz, 2H), 3.72-3.71 (m, 4H), 3.14-3.12 (m, 4H), 2.60 (s, 3H), 2.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 143.9, 138.0, 133.6, 131.8, 130.7, 126.8, 66.3, 45.3, 21.3, 20.8; HRMS calc. for $C_{12}H_{17}NO_3SNa$ $(M+Na)^+$, 278.0827; found, 278.0831.

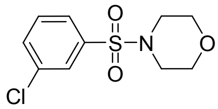
4-(4-fluorophenylsulfonyl)morpholine 

Compound **3h** was obtained in 81% yield according to the general procedure. 1H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.80-7.77 (m, 2H), 7.26-7.23 (m, 2H), 3.75 (t, J = 4.7 Hz, 4H), 3.00 (t, J = 4.8 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 165.4 (d, J = 254 Hz), 131.3 (d, J = 3.3 Hz), 130.5 (d, J = 9.2 Hz), 116.4 (d, J = 22.5 Hz), 66.1, 46.0; HRMS calc. for $C_{10}H_{12}FNO_3SNa$ (M+Na)+, 268.0420; found, 268.0423.

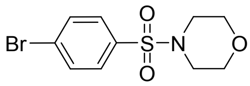
4-(4-chlorophenylsulfonyl)morpholine 

Compound **3i** was obtained in 75% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.70 (d, J = 8.6 Hz, 2H), 7.54 (d, J = 8.6 Hz, 2H), 3.75 (t,

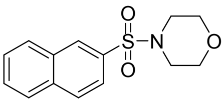
J = 4.7 Hz, 4H), 3.01 (t, J = 4.7 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 139.7, 133.7, 129.5, 129.2, 66.1, 45.9; HRMS calc. for $C_{10}H_{12}ClNO_3SNa$ $(M+Na)^+$, 284.0124; found, 284.0126.

4-(3-chlorophenylsulfonyl)morpholine 

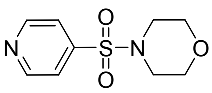
Compound **3j** was obtained in 86% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.68 (t, J = 1.8 Hz, 1H), 7.59-7.53 (m, 2H), 7.44 (t, J = 8.0 Hz, 1H), 3.69 (t, J = 4.6 Hz, 4H), 2.96 (t, J = 4.8 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 136.0, 134.5, 132.2, 129.4, 126.8, 124.9, 65.0, 44.9; HRMS calc. for $C_{10}H_{12}ClNO_3SNa$ $(M+Na)^+$, 284.0124; found, 284.0119.

4-(4-bromophenylsulfonyl)morpholine 

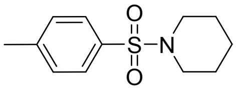
Compound **3k** was obtained in 62% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.70 (d, J = 8.6 Hz, 2H), 7.62 (d, J = 8.6 Hz, 2H), 3.75 (t, J = 4.8 Hz, 4H), 3.00 (t, J = 4.8, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 134.2, 132.5, 129.3, 128.2, 66.0, 45.9; HRMS calc. for $C_{10}H_{12}BrNO_3SNa$ $(M+Na)^+$, 327.9619; found, 327.9621.

4-(naphthalen-2-ylsulfonyl)morpholine 

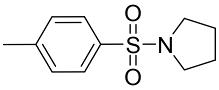
Compound **3l** was obtained in 75% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 8.34 (d, J = 1.8 Hz, 1H), 8.01-7.98 (m, 2H), 7.94 (d, J = 8.1 Hz, 1H), 7.76-7.74 (m, 1H), 7.69-7.62 (m, 2H), 3.75 (t, J = 4.8 Hz, 4H), 3.07 (t, J = 4.8 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 135.0, 132.3, 132.2, 129.3, 129.3, 129.2, 129.0, 128.0, 127.7, 123.0, 66.1, 46.1; HRMS calc. for $C_{14}H_{15}NO_3SNa$ $(M+Na)^+$, 300.0670; found, 300.0673.

4-(pyridin-4-ylsulfonyl)morpholine 

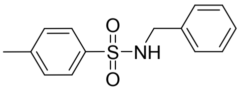
Compound **3m** was obtained in 60% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 8.90 (d, J = 4.1 Hz, 2H), 7.60 (d, J = 5.8 Hz, 2H), 3.76 (t, J = 4.8 Hz, 4H), 3.06 (t, J = 4.7 Hz, 4H); ^{13}C NMR ($CDCl_3$, 125MHz, ppm): δ 151.2, 143.6, 120.9, 66.0, 45.9; HRMS calc. for $C_9H_{12}N_2O_3SNa$ (M+Na)+, 251.0466; found, 251.0467.

1-tosylpiperidine 

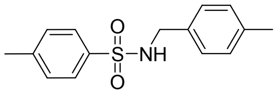
Compound **3n** was obtained in 64% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.66 (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 2.99 (t, J = 5.5 Hz, 4H), 2.45 (s, 3H), 1.68-1.63 (m, 4H), 1.45-1.41 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 143.3, 133.3, 129.5, 127.7, 46.9, 25.2, 23.5, 21.5; HRMS calc. for $C_{12}H_{17}NO_2SNa$ $(M+Na)^+$, 262.0878; found, 262.0879.

1-tosylpyrrolidine 

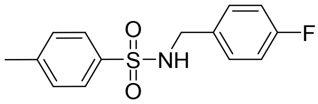
Compound **3o** was obtained in 56% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.74 (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 3.26-3.24 (m, 4H), 2.45 (s, 3H), 1.78-1.75 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 143.3, 133.9, 129.6, 127.6, 47.9, 25.2, 21.5; HRMS calc. for $C_{11}H_{15}NO_2SNa$ $(M+Na)^+$, 248.0721; found, 248.0727.

N-benzyl-4-methylbenzenesulfonamide 

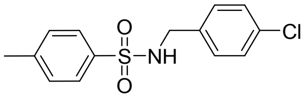
Compound **3p** was obtained in 63% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.76 (d, J = 8.3 Hz, 2H), 7.32-7.25 (m, 5H), 7.20-7.19 (m, 2H), 4.67 (d, J = 5.7 Hz, 1H), 4.12 (d, J = 6.2 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 143.5, 136.9, 136.3, 129.8, 128.7, 127.9, 127.9, 127.2, 47.3, 21.6; HRMS calc. for $C_{14}H_{15}NO_2SNa$ $(M+Na)^+$, 284.0721; found, 284.0723.

4-methyl-N-(4-methylbenzyl)benzenesulfonamide 

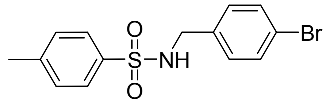
Compound **3q** was obtained in 60% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.64 (d, J = 8.2 Hz, 2H), 7.32 (d, J = 7.9 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 4.24 (brs, 1H), 4.22-4.19 (m, 1H), 3.87-3.83 (m, 1H), 2.42 (s, 3H), 2.32 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.3, 140.9, 137.4, 134.7, 129.6, 129.3, 128.3, 126.0, 44.4, 21.4, 21.1; HRMS calc. for $C_{15}H_{17}NO_2SNa$ $(M+Na)^+$, 298.0878, found, 298.0881.

N-(4-fluorobenzyl)-4-methylbenzenesulfonamide 

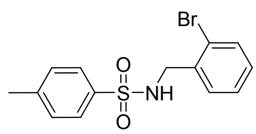
Compound **3r** was obtained in 61% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.63 (d, J = 8.3 Hz, 2H), 7.33-7.31 (m, 2H), 7.25-7.22 (m, 2H), 6.99 (t, J = 8.7 Hz, 2H), 4.30 (t, J = 1.6 Hz, 1H), 4.23-4.19 (m, 1H), 3.90-3.86 (m, 1H) 2.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 162.2 (d, J = 244.7 Hz), 141.5, 140.7, 133.6 (d, J = 3.2 Hz), 130.0 (d, J = 8.1 Hz), 129.7, 126.4, 126.0, 115.6 (d, J =21.6 Hz), 44.0, 21.4; HRMS calc. for $C_{14}H_{14}FNO_2SNa$ $(M+Na)^+$, 302.0627; found, 302.0625.

N-(4-chlorobenzyl)-4-methylbenzenesulfonamide 

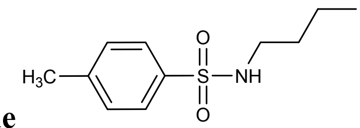
Compound **3s** was obtained in 67% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.63 (d, J = 7.9 Hz, 2H), 7.32(d, J = 8.0 Hz, 2H) 7.27 (d, J = 8.3 Hz, 2H), 7.20 (d, J = 8.2 Hz, 2H), 4.32-4.28 (m, 1H), 4.22-4.18 (m, 1H), 3.91-3.87 (m, 1H), 2.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.5, 140.7, 136.4, 133.5, 129.7, 128.8, 125.9, 44.0, 21.4; HRMS calc. for $C_{14}H_{14}ClNO_2SNa$ $(M+Na)^+$, 318.0331; found, 318.0333.

N-(4-bromobenzyl)-4-methylbenzenesulfonamide 

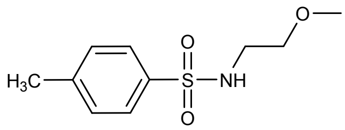
Compound **3t** was obtained in 53% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.62 (d, J = 8.3 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 7.14 (d, J = 8.4 Hz, 2H), 4.31 (t, J = 6.2 Hz, 1H), 4.20-4.16 (m, 1H), 3.90-3.85 (m, 1H), 2.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 141.6, 140.7, 136.9, 131.7, 130.0, 129.7, 125.9, 121.6, 44.0, 21.4; HRMS calc. for $C_{14}H_{14}BrNO_2SNa$ $(M+Na)^+$, 361.9826; found, 361.9829.

N-(2-bromobenzyl)-4-methylbenzenesulfonamide 

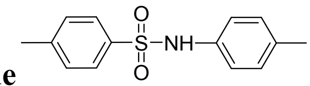
Compound **3u** was obtained in 60% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.63 (d, J = 8.3 Hz, 2H), 7.53 (d, J = 7.6 Hz, 1H), 7.30 (d, J = 7.9 Hz, 2H), 7.25-7.24 (m, 2H), 7.15-7.11 (m, 1H), 4.46 (t, J = 6.7 Hz, 1H), 4.37-4.33 (m, 1H), 4.09-4.04 (m, 1H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): δ 140.4, 139.8, 136.3, 131.9, 129.5, 128.5, 128.3, 126.6, 125.0, 123.0, 44.3, 20.3; HRMS calc. for $C_{14}H_{14}BrNO_2SNa$ $(M+Na)^+$, 361.9826; found, 361.9831.

N-butyl-4-methylbenzenesulfonamide 

Compound **3v** was obtained in 56% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.75 (t, *J* = 8.3 Hz, 2H), 7.30 (t, *J* = 8.0 Hz, 2H), 4.70 (brs, 1H), 2.94-2.90 (m, 2H), 2.43 (s, 3H), 1.46-1.41 (m, 2H), 1.31-1.26 (m, 2H), 0.84 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125MHz, ppm): δ 143.3, 137.0, 129.7, 127.1, 42.9, 31.6, 21.5, 19.7, 13.5; HRMS calc. for $C_{11}H_{17}NO_2SNa$ (M+Na)+, 250.0878; found, 250.0877.

N-(ethoxymethyl)-4-methylbenzenesulfonamide 

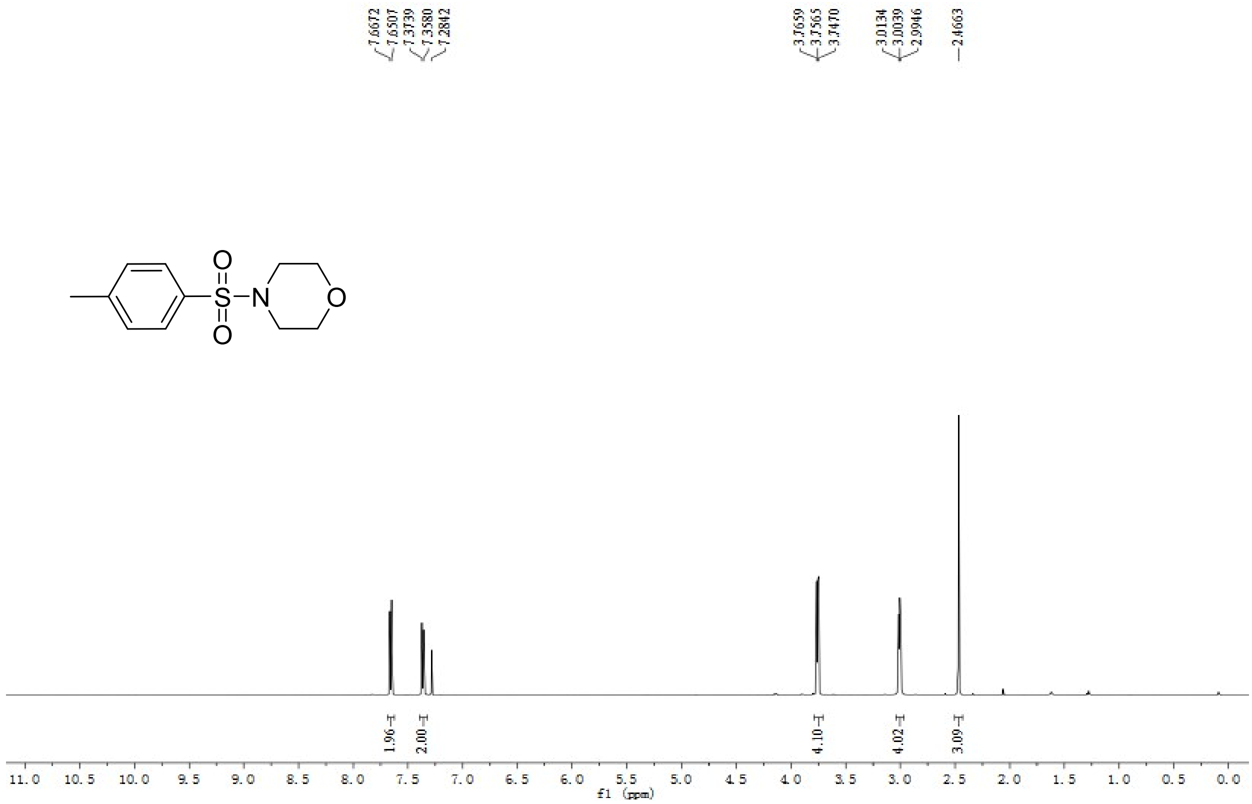
Compound **3w** was obtained in 57% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm): δ 7.59 (d , *J* = 8.2 Hz ,2H), 7.29 (t, *J* = 8.0 Hz, 2H), 4.51 (s, 1H), 3.49-3.42 (m, 2H), 3.33 (s, 3H), 3.29-3.24 (m, 1H), 2.96-2.90 (m, 1H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 125MHz, ppm): δ 141.2, 136.5, 129.6, 126.0, 71.9, 58.7, 40.3, 21.3; HRMS calc. for $C_{10}H_{15}NO_3SNa$ (M+Na)+, 252.0670; found, 252.0673.

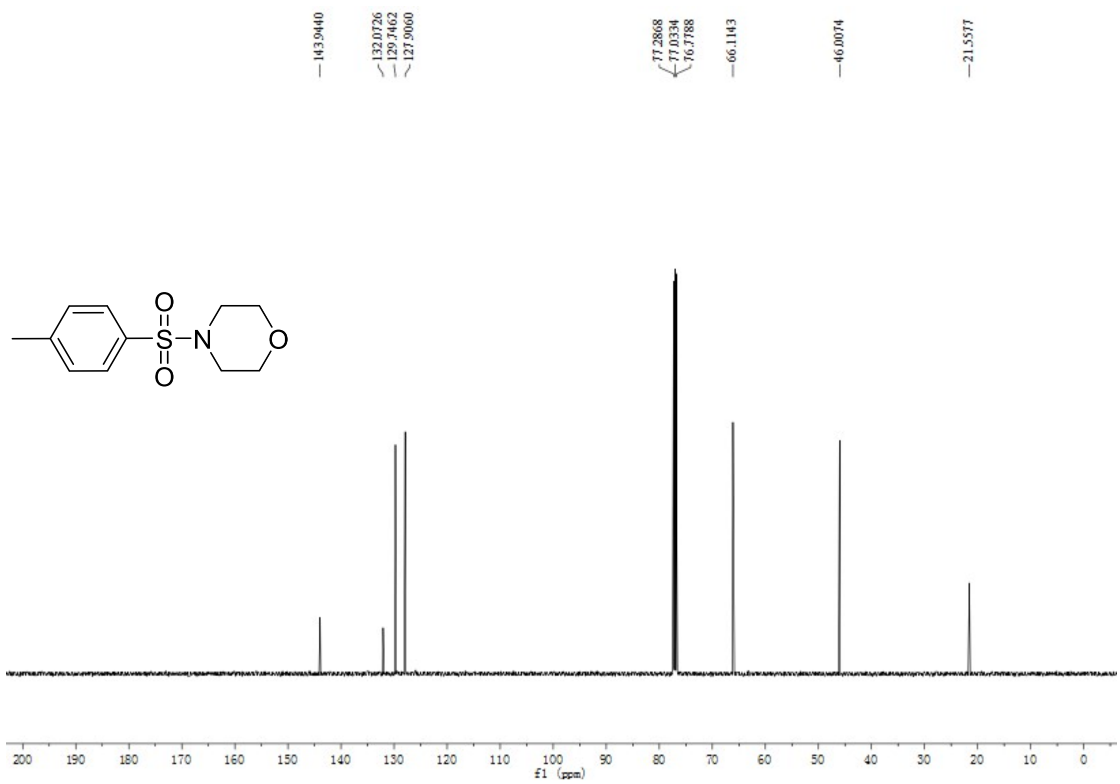
4-methyl-N-p-tolylbenzenesulfonamide 

Compound **3x** was obtained in 42% yield according to the general procedure. ^{1}H NMR ($CDCl_3$, 500 MHz, ppm):*δ* 7.66 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.73 (s, 1H), 2.40 (s, 3H), 2.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, ppm): *δ* 143.7, 136.1, 135.4, 133.7, 129.8, 129.6, 127.3, 122.3, 21.5, 20.8; HRMS calc. for $C_{14}H_{15}NO_2SNa$ $(M+Na)^{+}$, 284.0721; found, 284.0723.

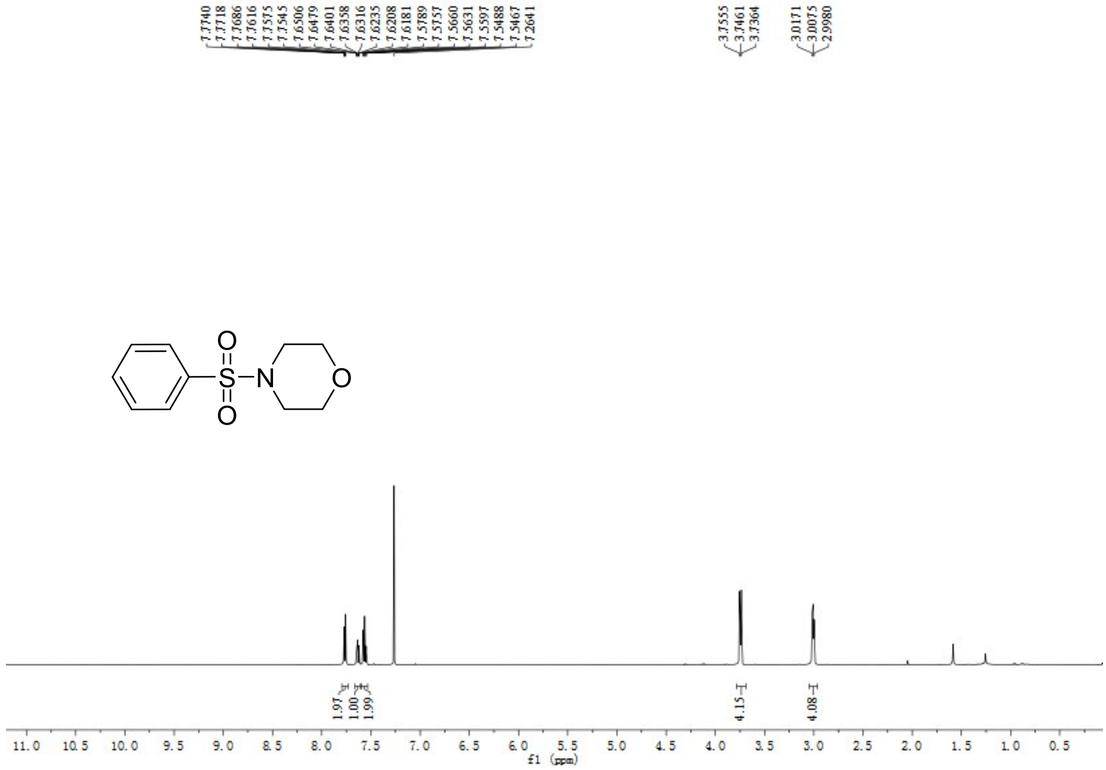
5. Copies of NMR spectra for 3a-3x

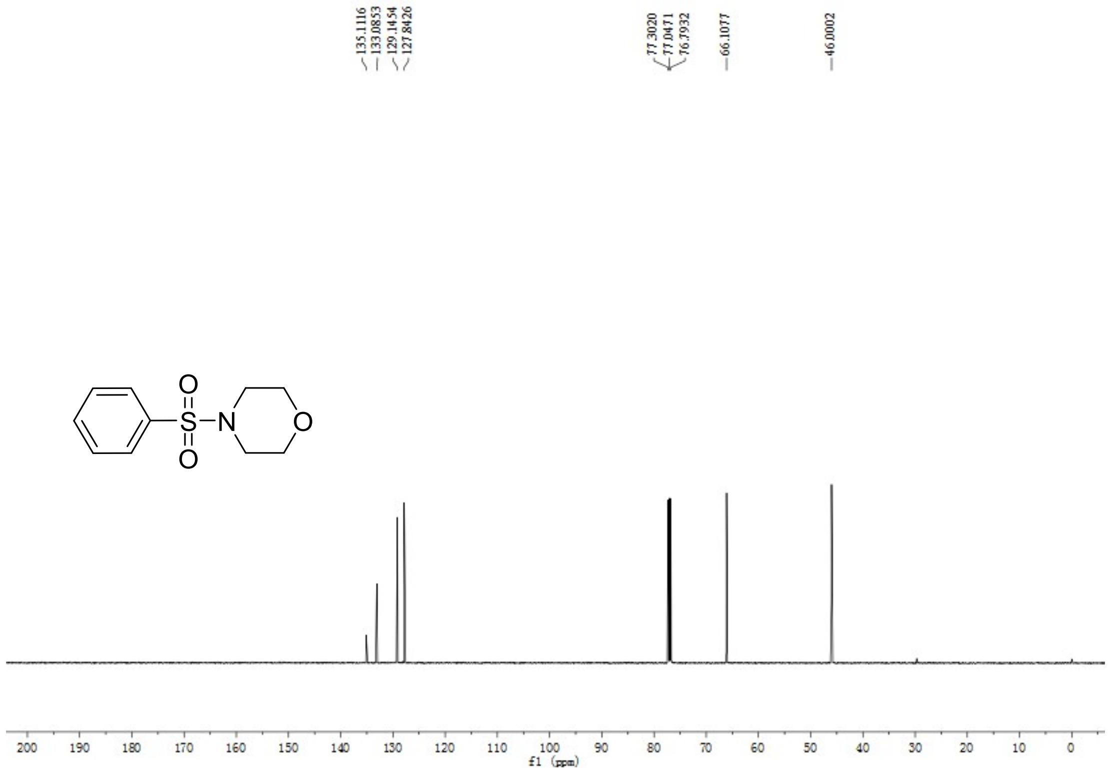
3a



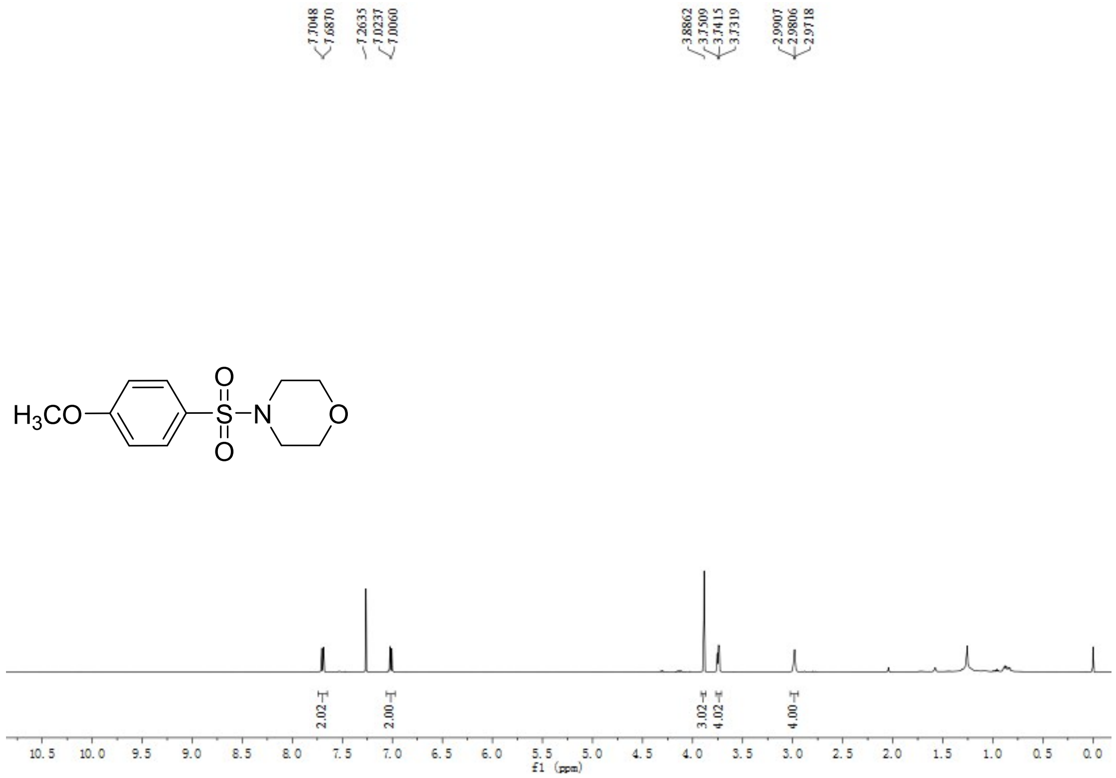


3b





3c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3.7319
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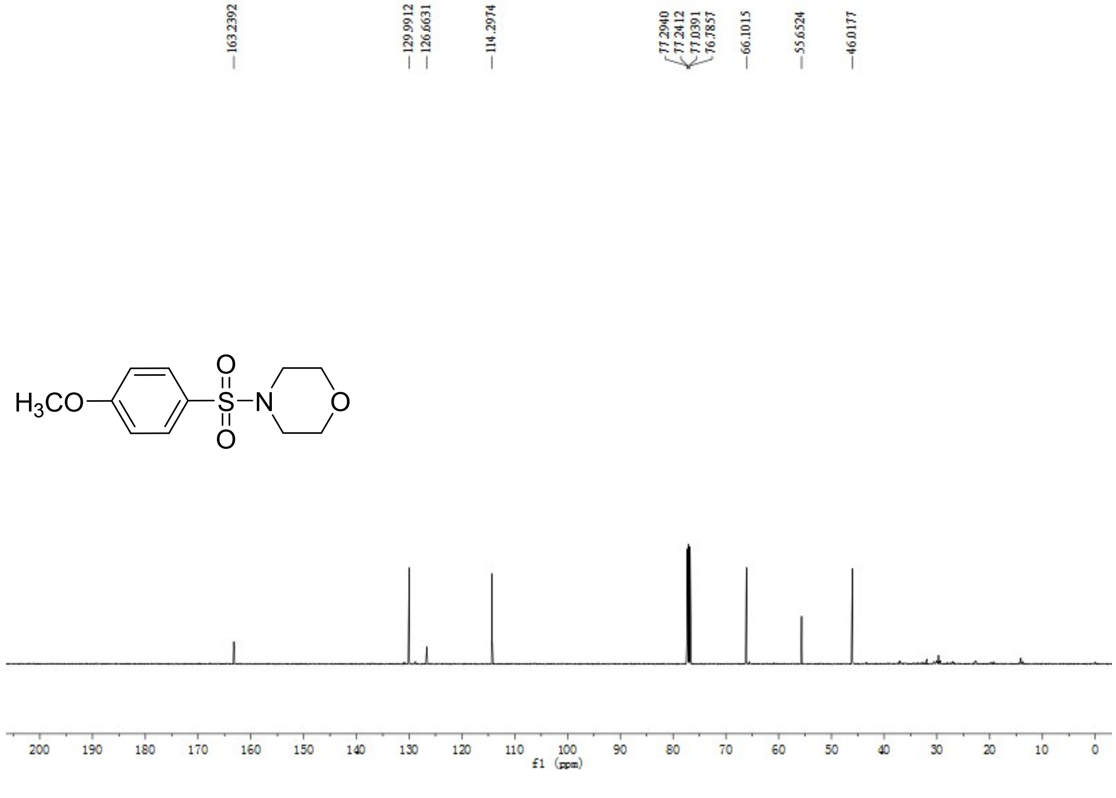
2.9806
2.9718
H3CO
S
O
O
N
O
2.02
2.00
3.02
4.02
4.00
f1 (ppm)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126.6631
114.2974
77.2940
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77.0391
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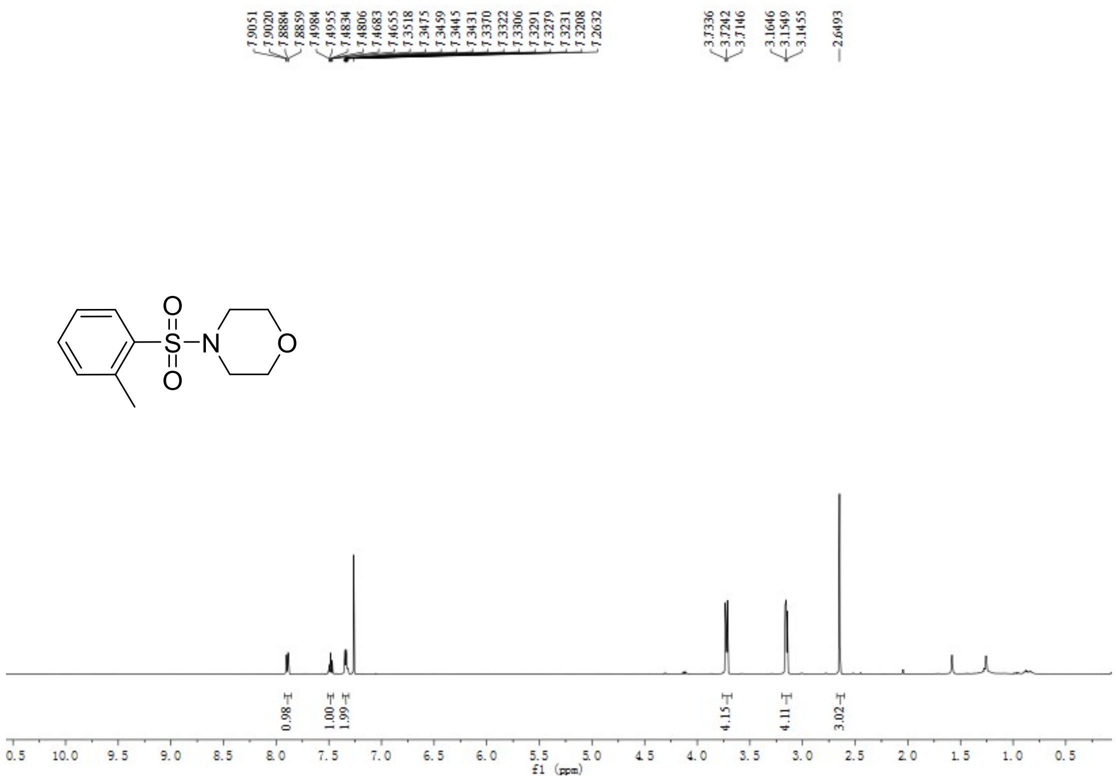
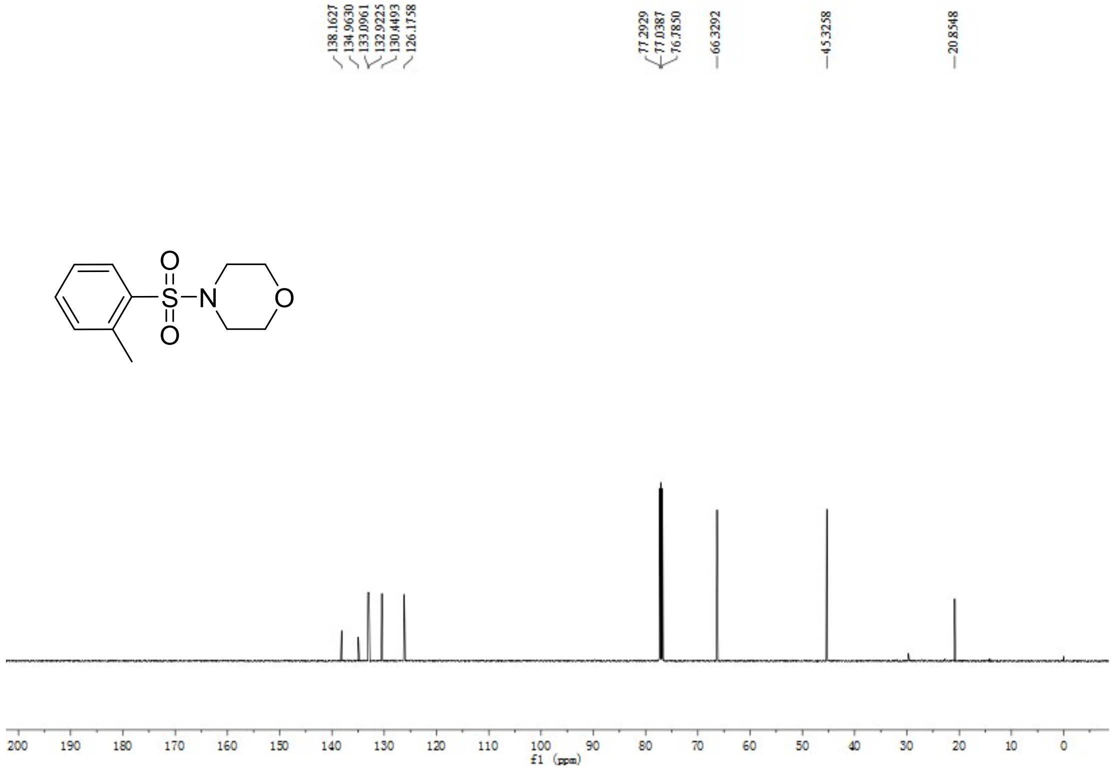
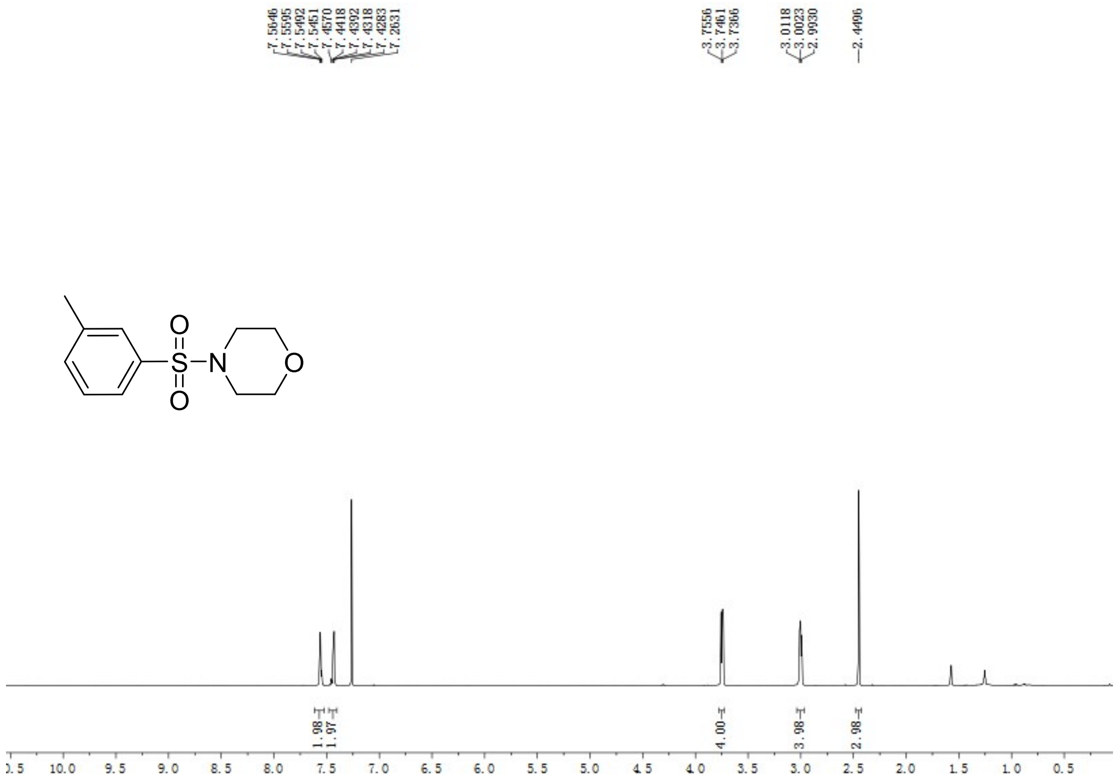
46.0177
H3CO
S
O
O
N
O
f1 (ppm)

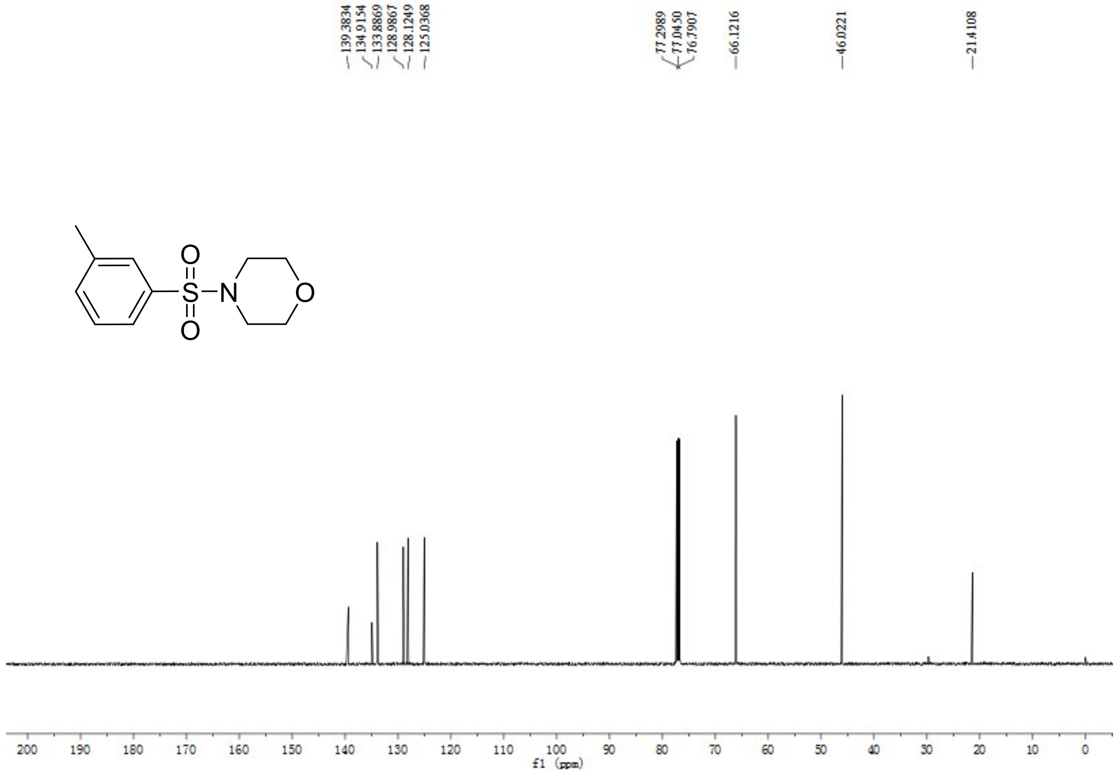
3d



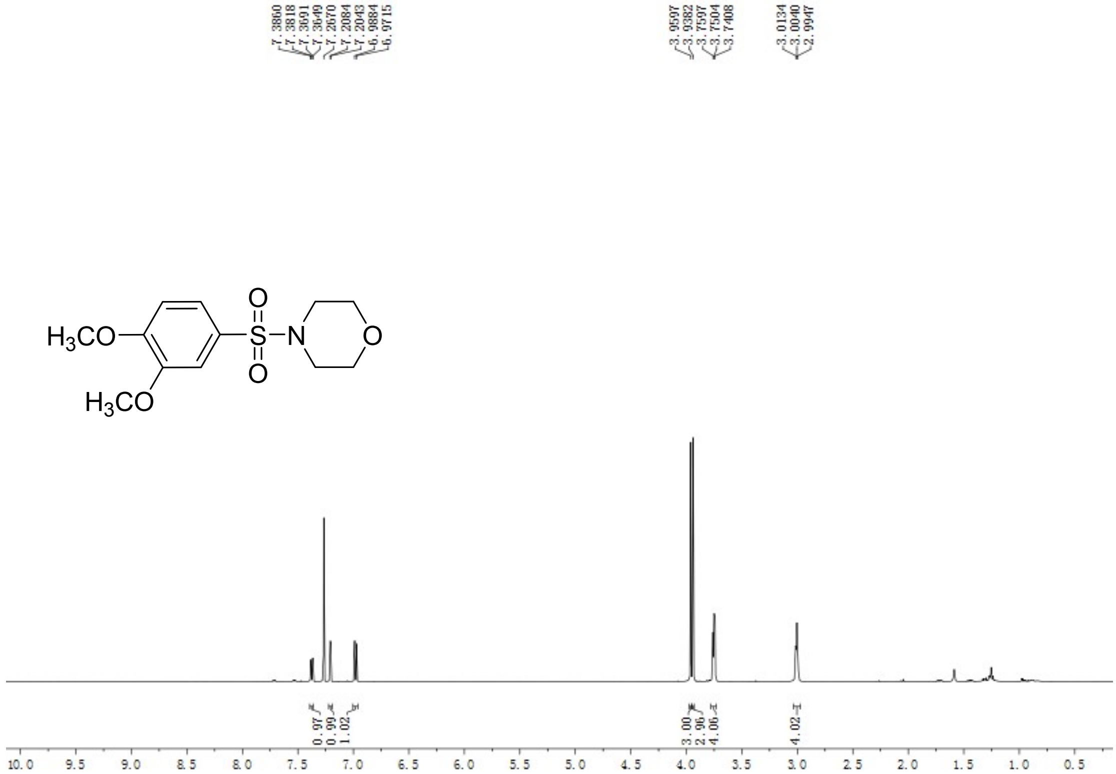


3e





3f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7.2670
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7.2043
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3.9382
3.7597
3.7504
3.7408
3.0134
3.0040
2.9947
H_3CO
H_3CO
S
N
O
O
O
0.97
0.99
1.02
3.00
2.96
4.06
4.02
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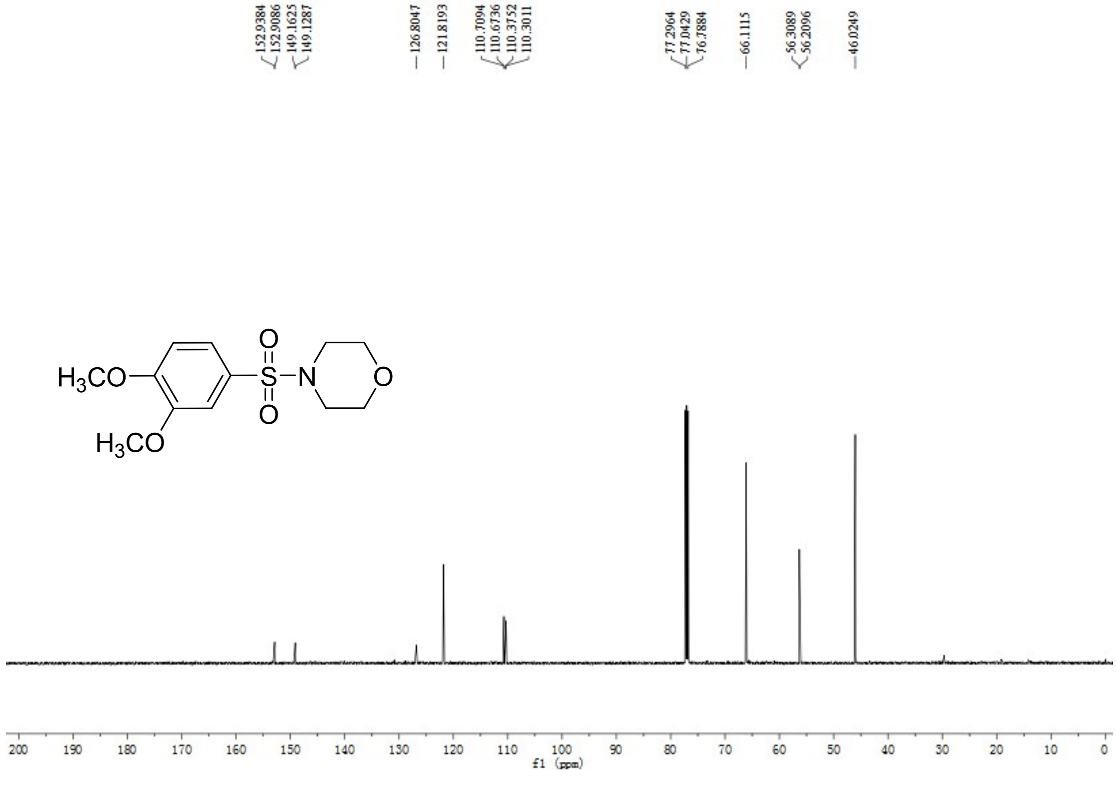
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110.3011
77.2964
77.0429
76.7884
66.1115
56.3089
56.2096
46.0249
H_3CO
H_3CO
S
N
O
O
O
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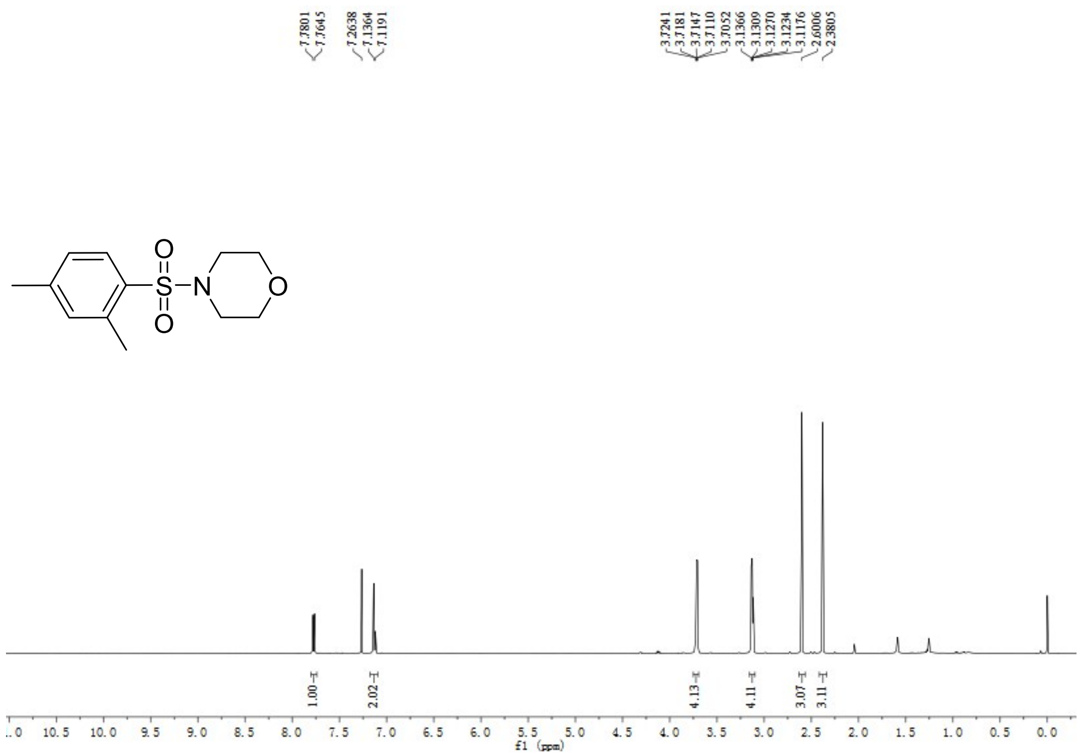
f1 (ppm)

3g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3.1366
3.1309
3.1270
3.1234
3.1176
2.6006
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S
N
O
O
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2.02
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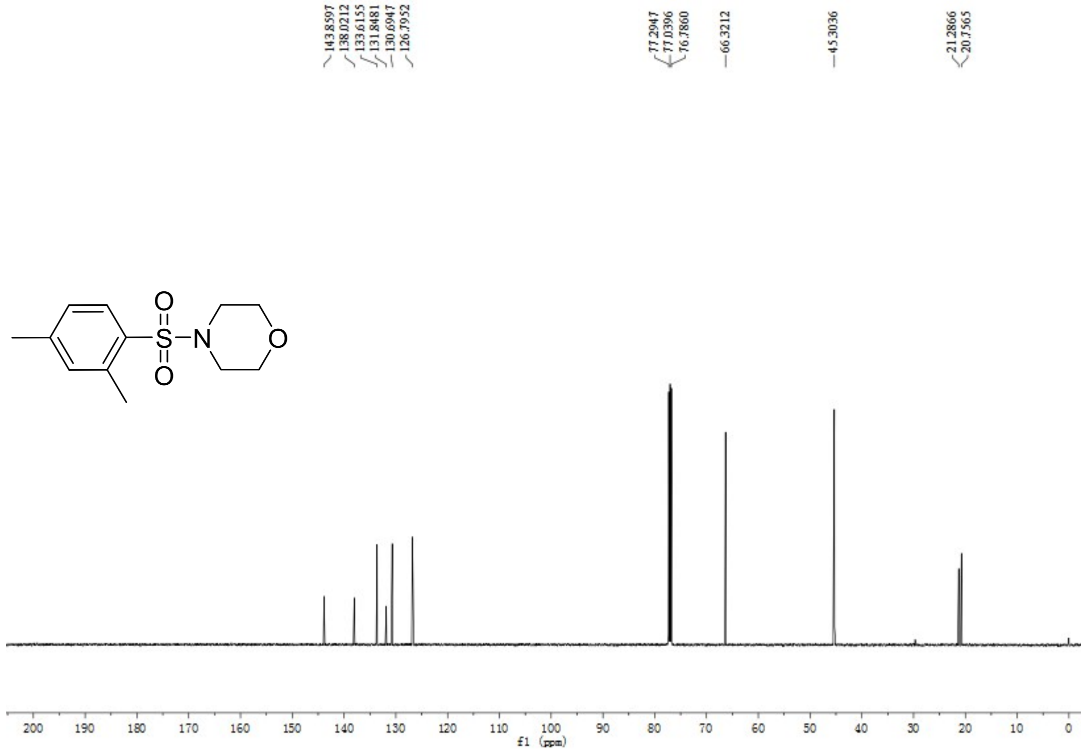
4.11
3.07
3.11
f1 (ppm)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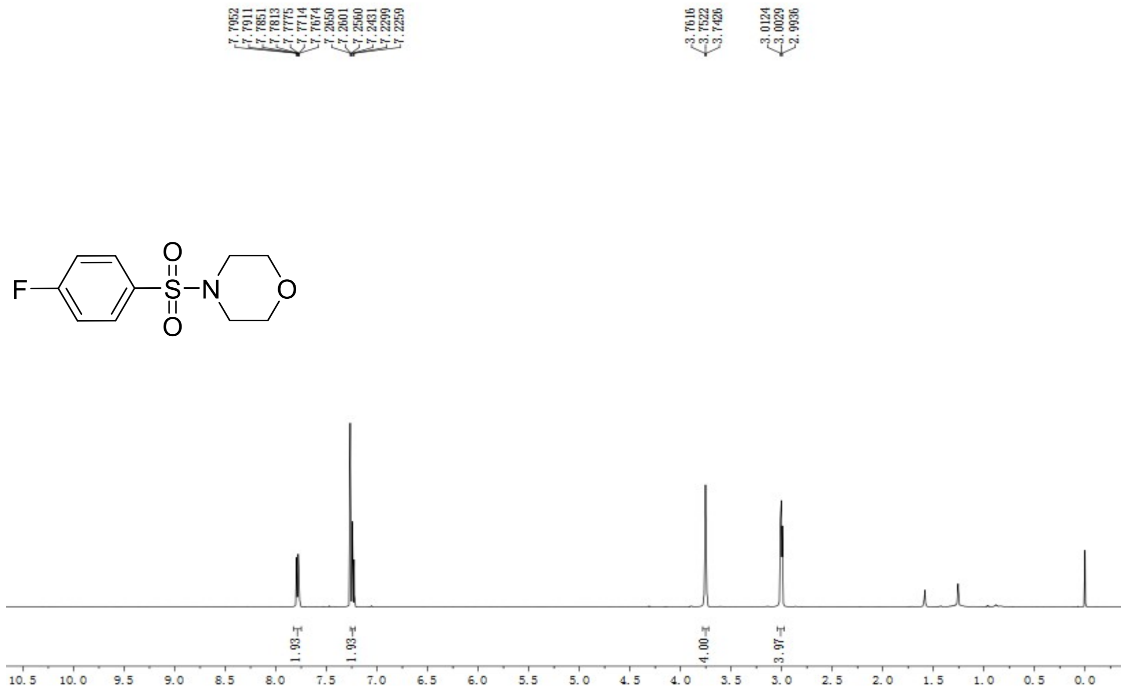
21.2866
20.7565
O
S
N
O
O
f1 (ppm)

3h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7.2431
7.2299
7.2259
3.7616
3.7522
3.7426
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3.0029
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F
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S
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O
O
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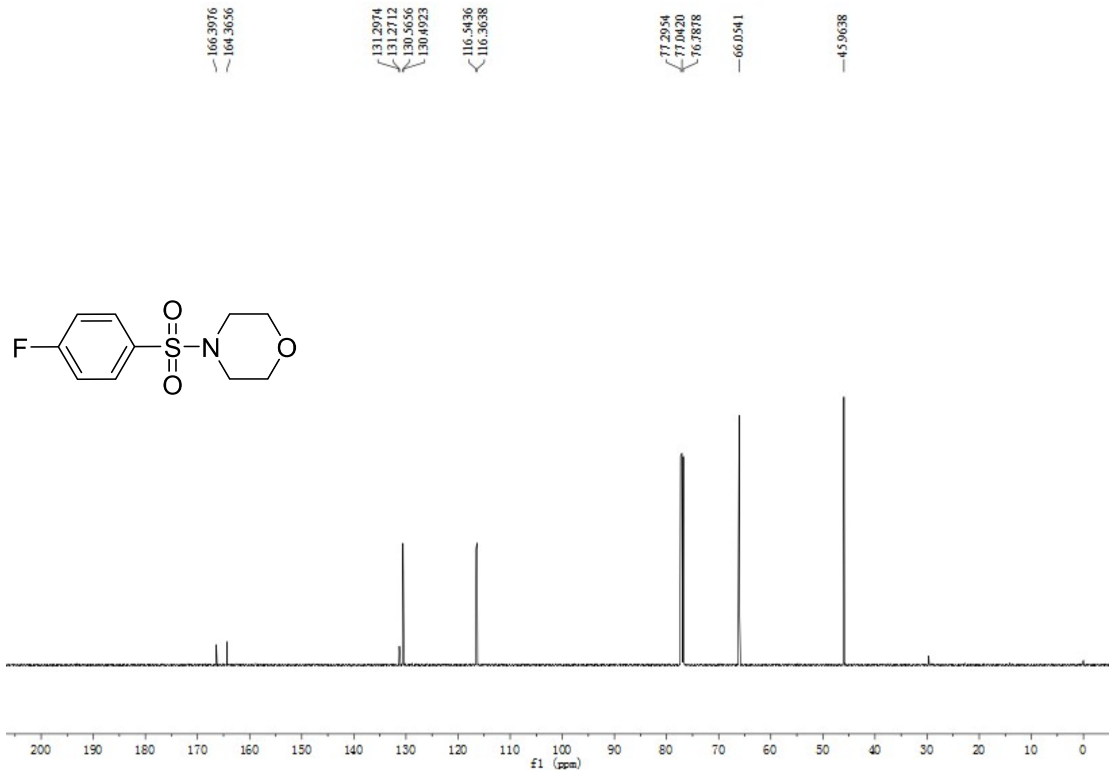
1.93
4.00
3.97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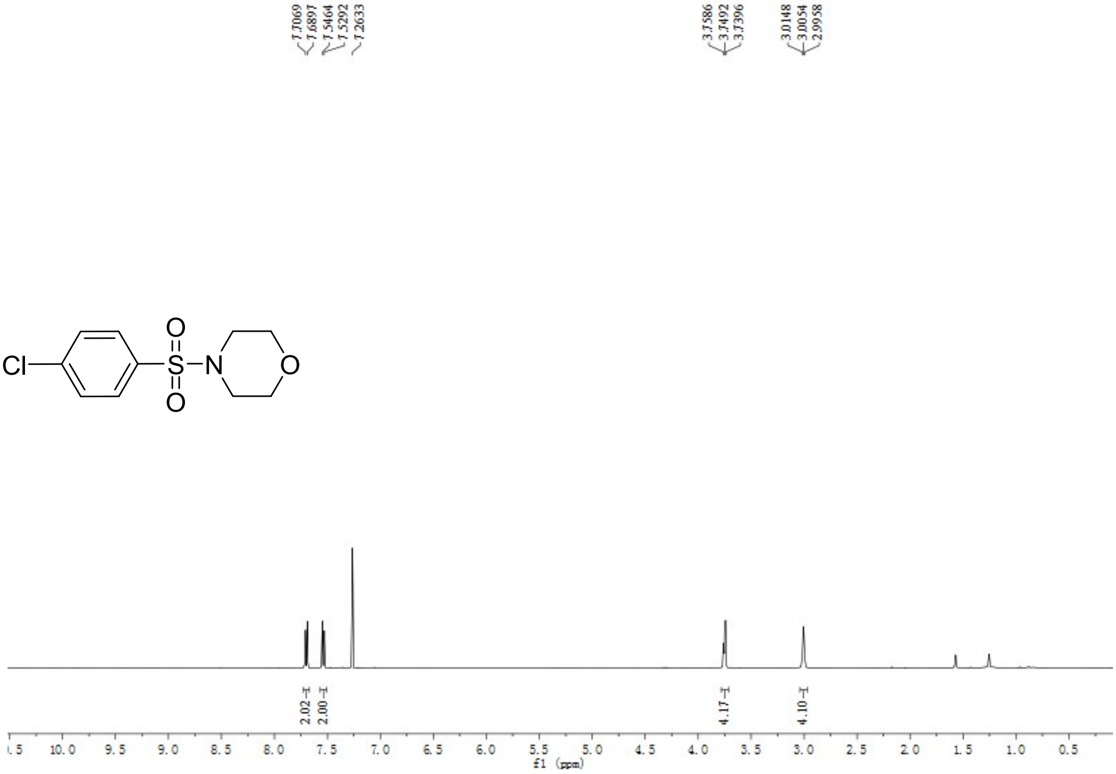
F
O
S
N
O
O
f1 (ppm)

3i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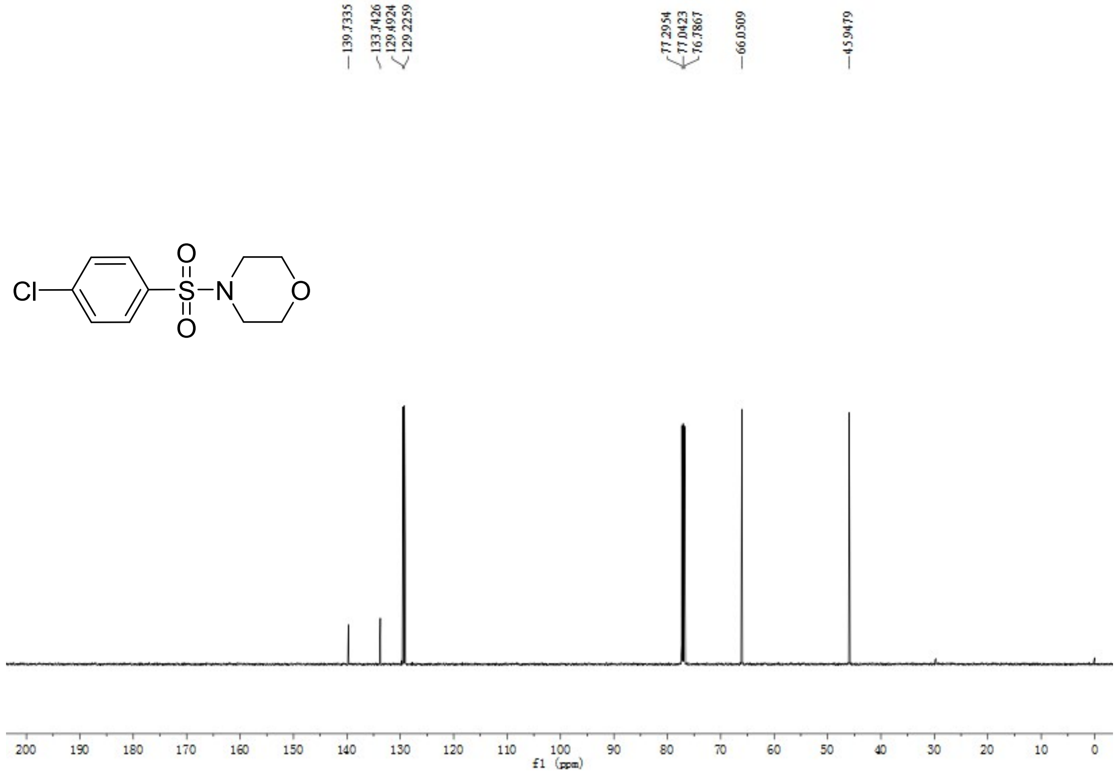
2.9958
Cl
S
O
O
N
O
2.02
2.00
4.17
4.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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O
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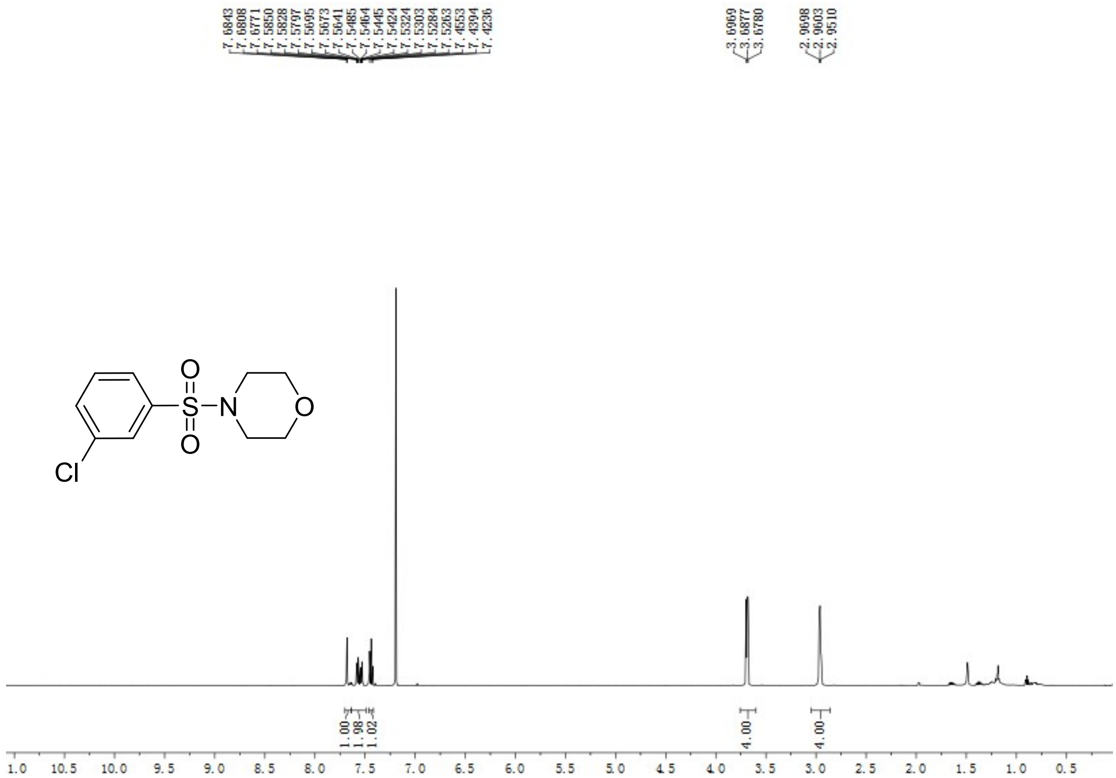
f1 (ppm)

3j



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7.5445
7.5424
7.5324
7.5303
7.5284
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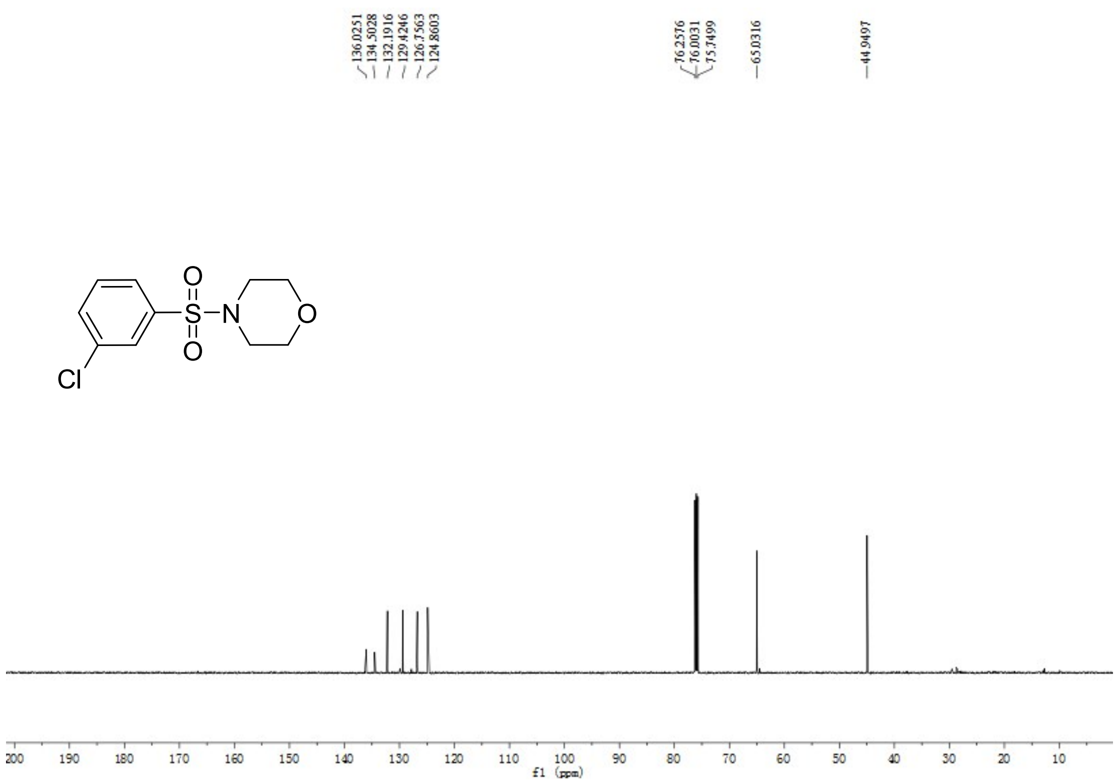
4.00
4.00
Cl



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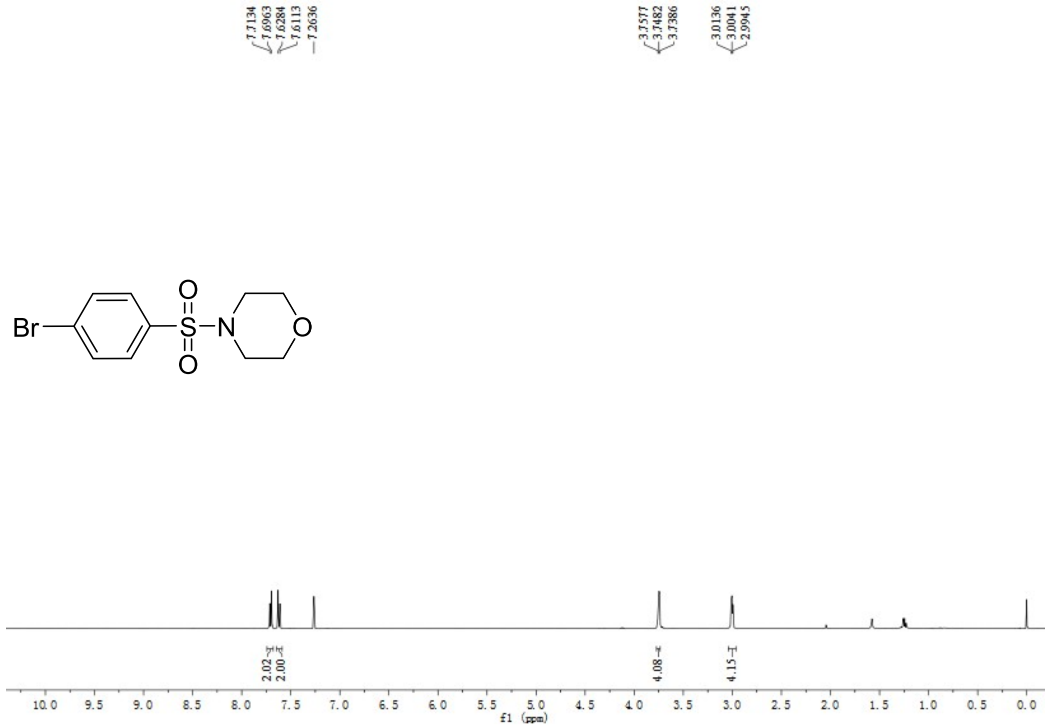
Cl
f1 (ppm)

3k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N
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2.00
4.08
4.15
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 0.0
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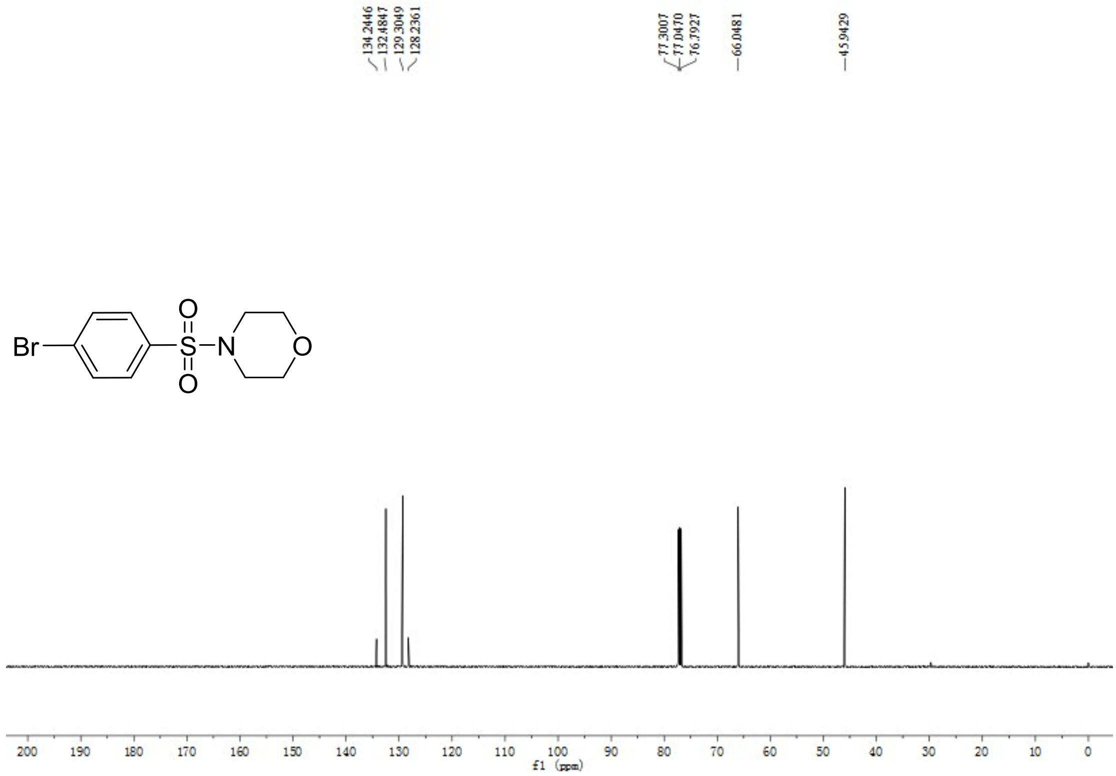
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O
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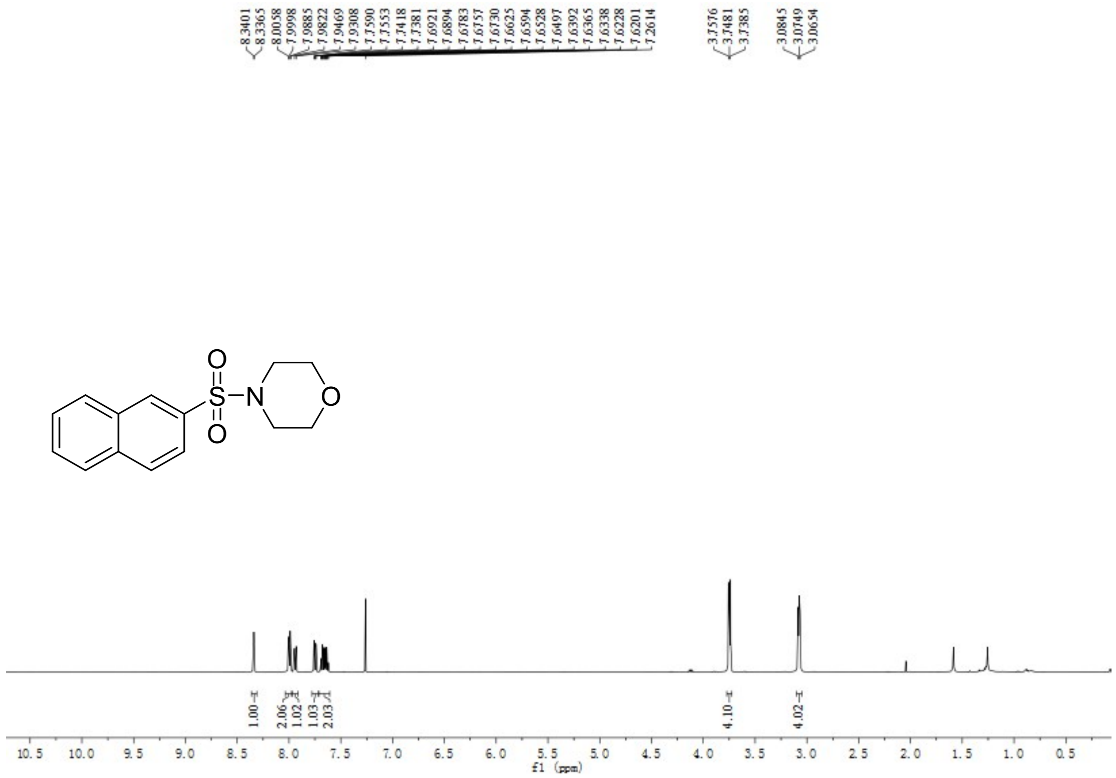
f1 (ppm)

3l



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7.6894
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7.6730
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N
O
O
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1.02
1.03
2.03
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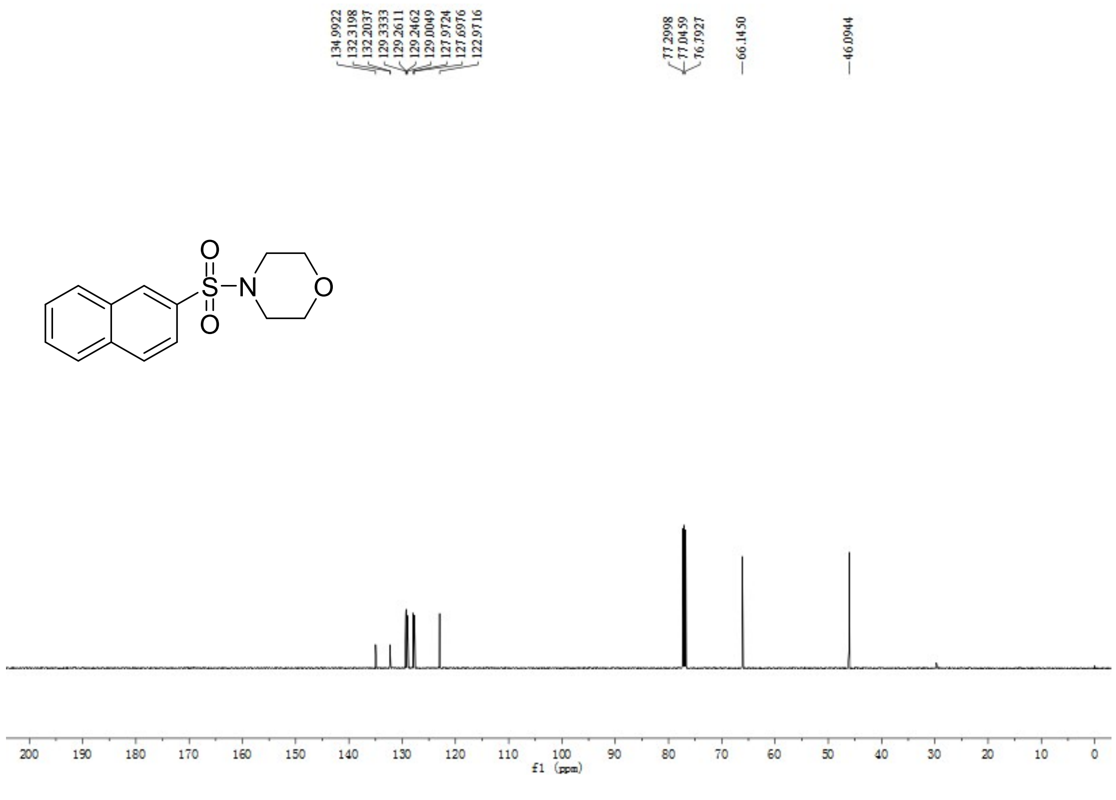
4.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



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46.0944
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N
O
O
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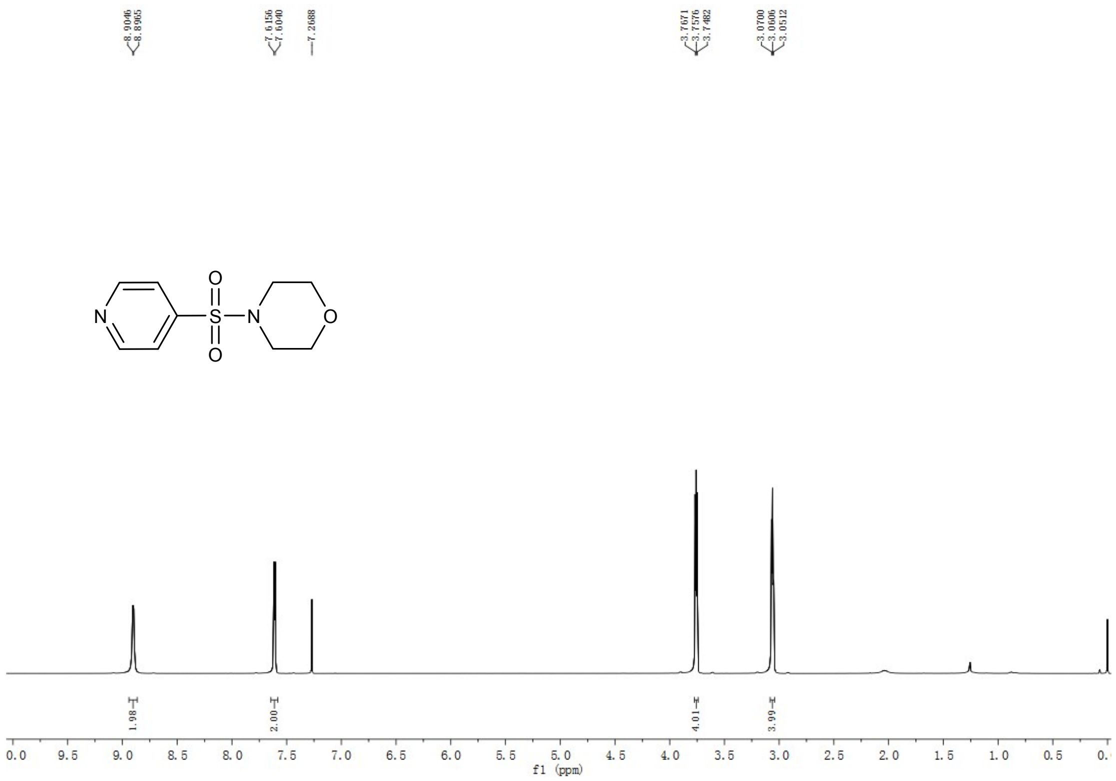
f1 (ppm)

3m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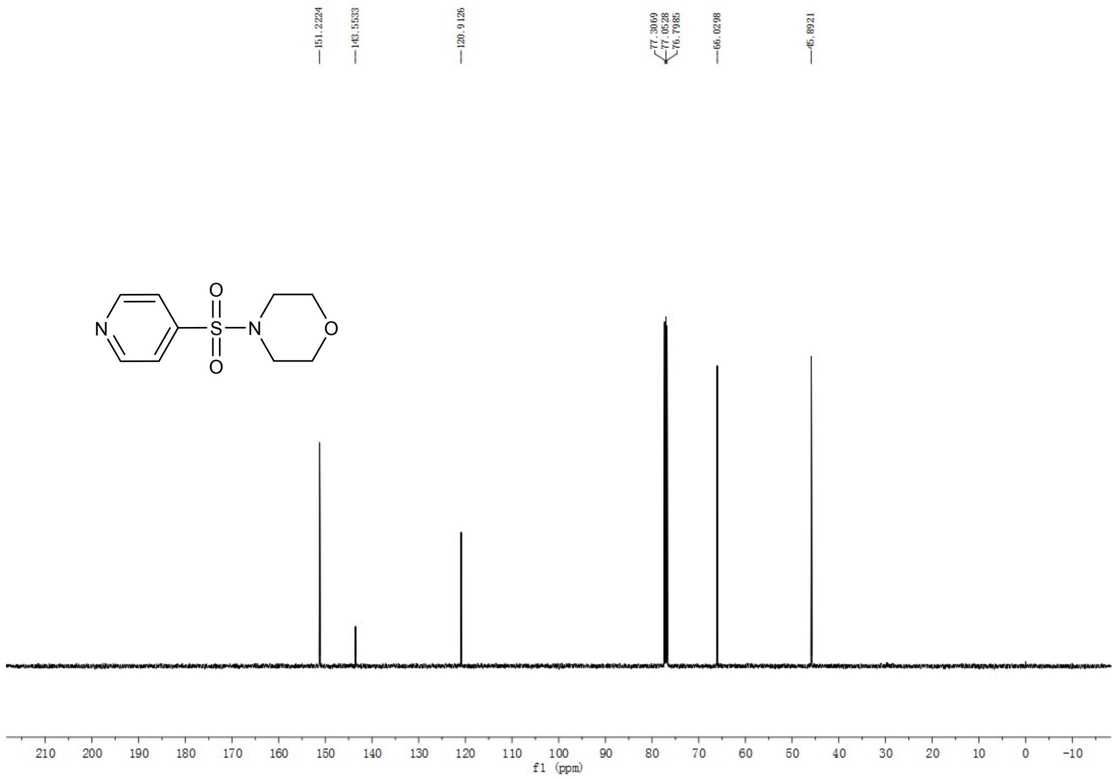
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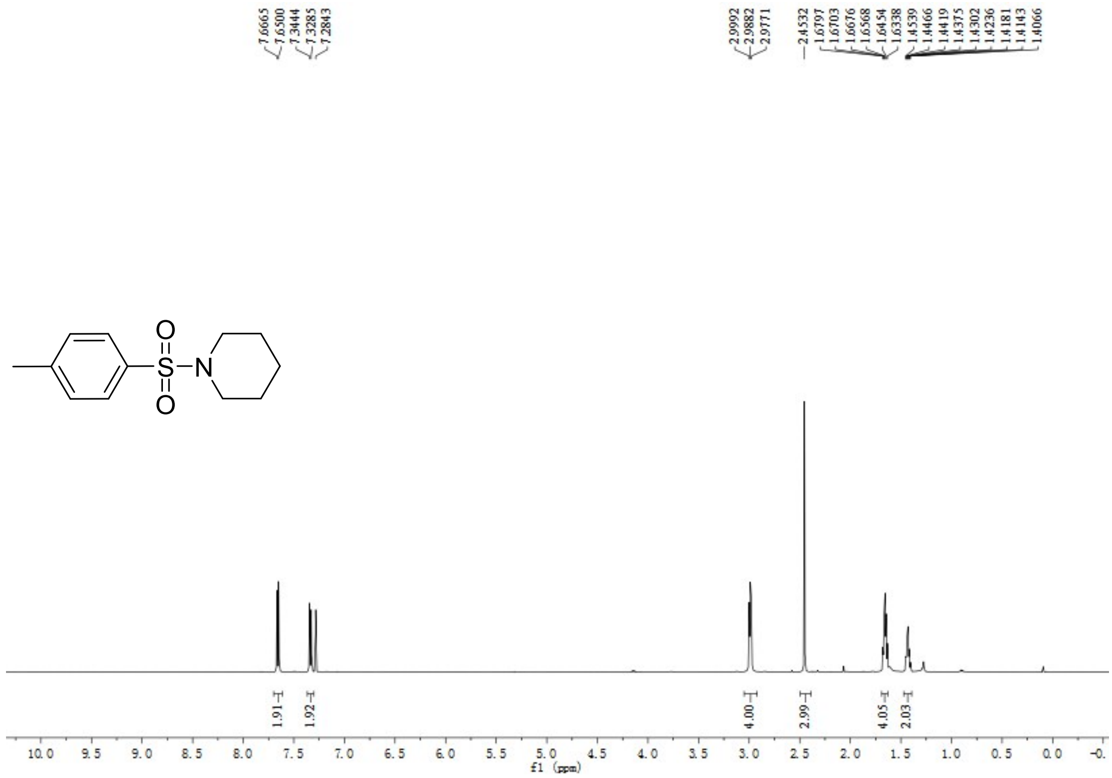
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1.4419
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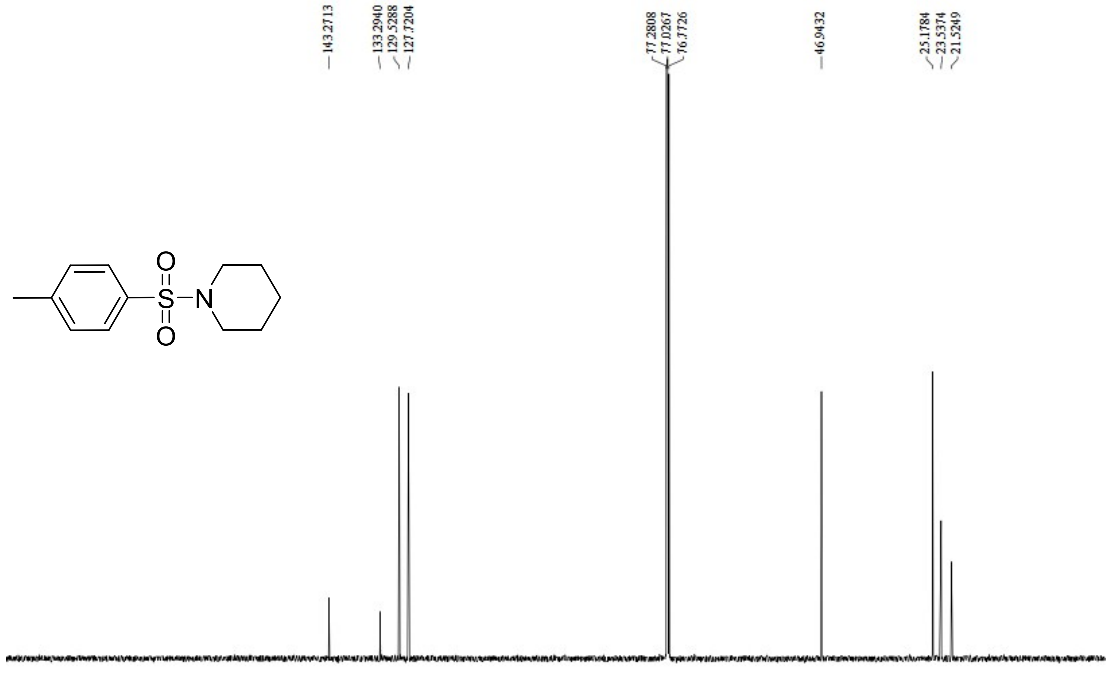
2.03
f1 (ppm)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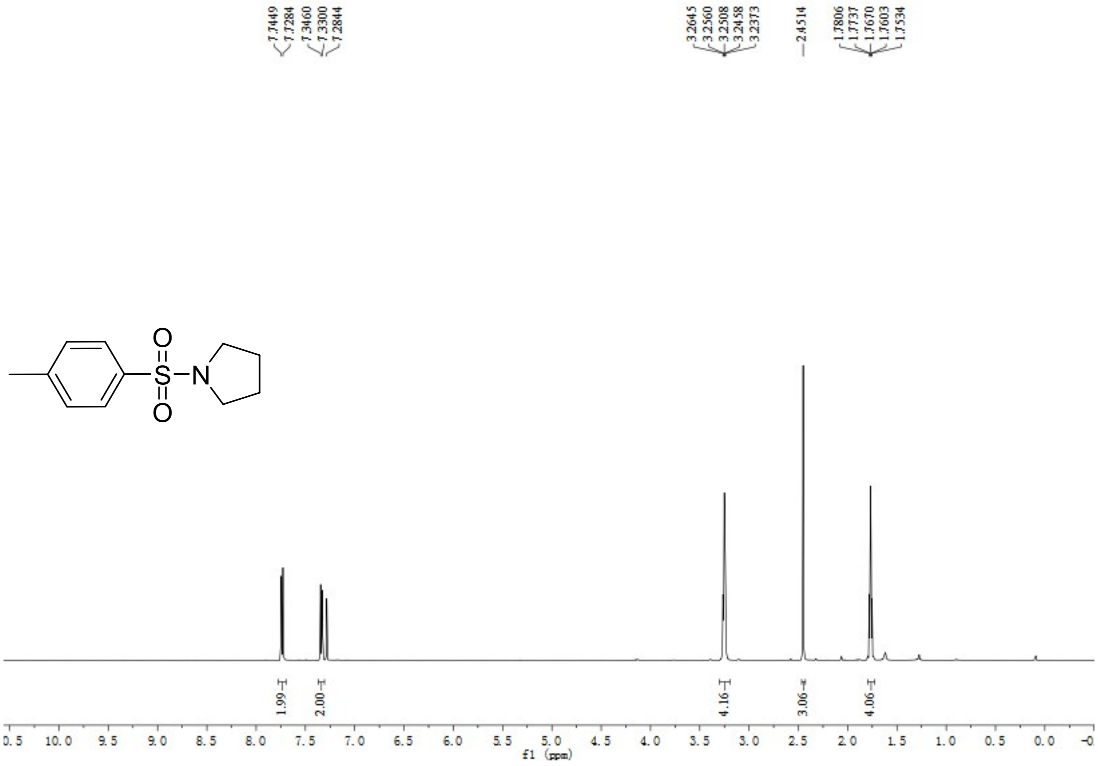
N
O
f1 (ppm)

3o



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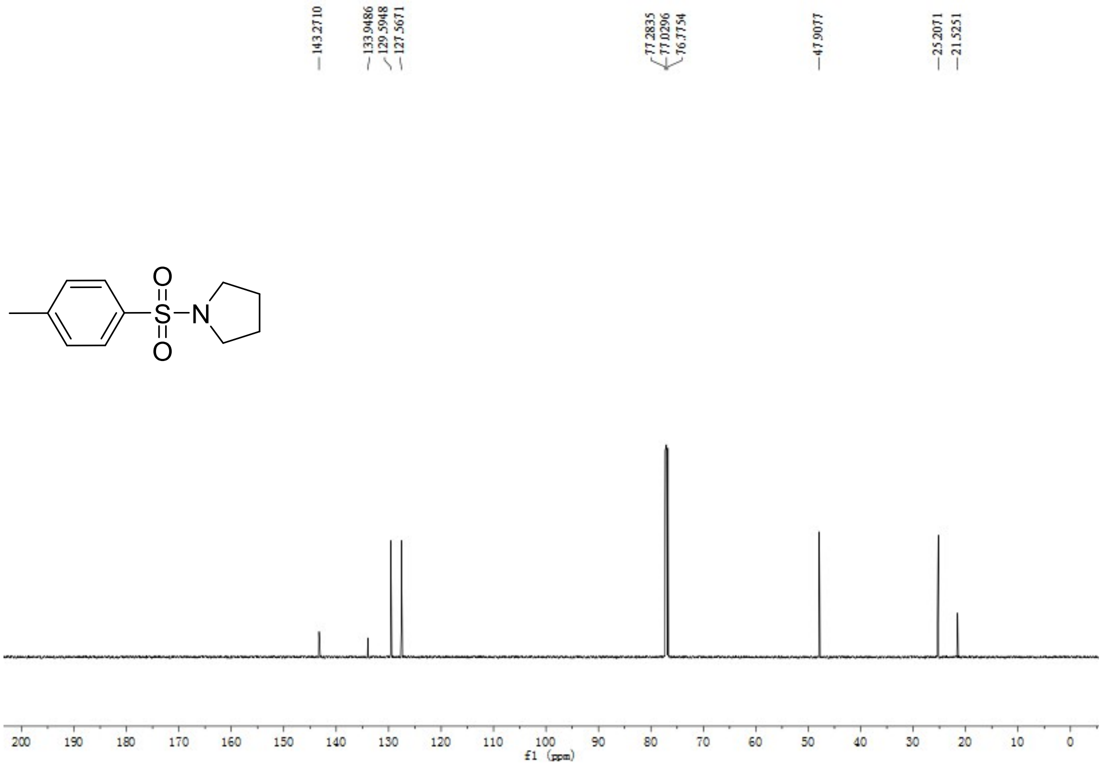
1.7737
1.7670
1.7603
1.7534
O
S
N
O
1.99
2.00
4.16
3.06
4.06
f1 (ppm)



143.2710
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129.5948
127.5671
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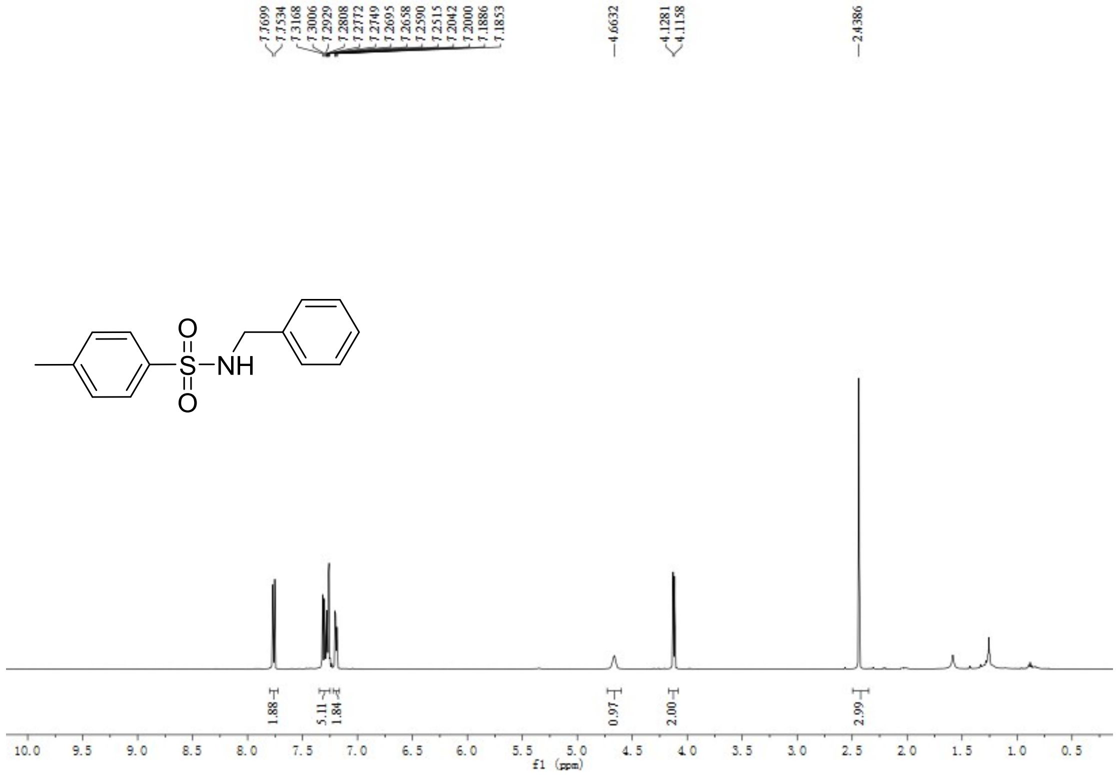
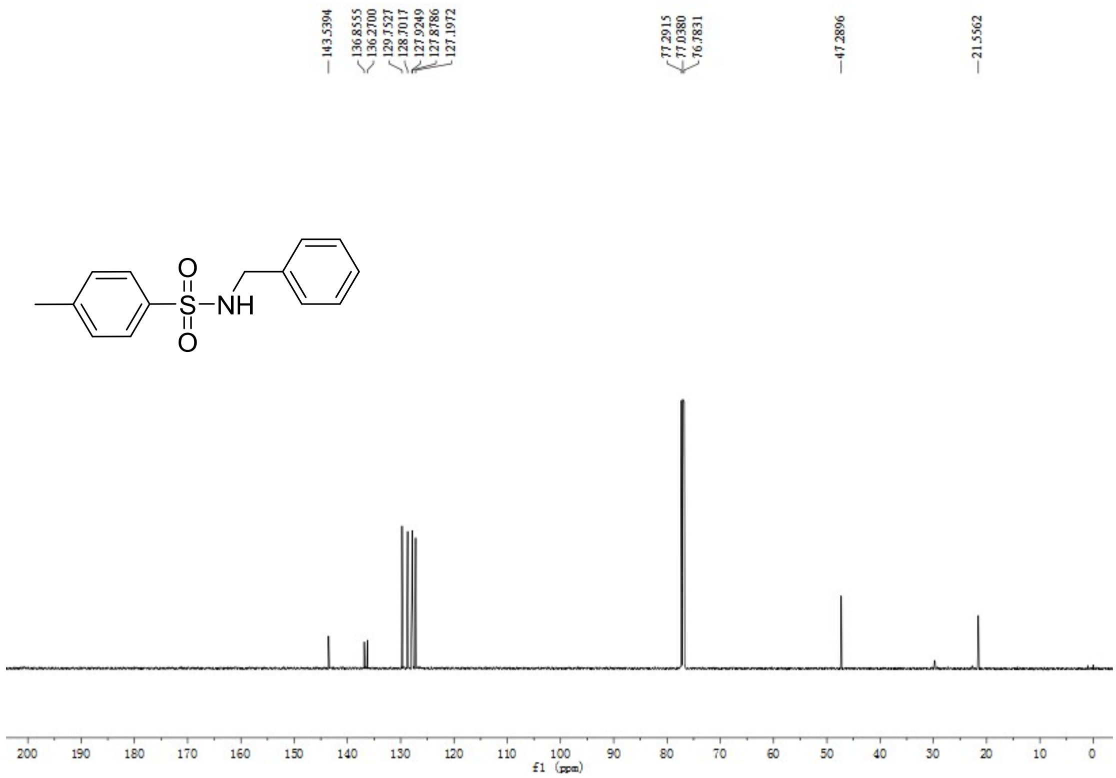
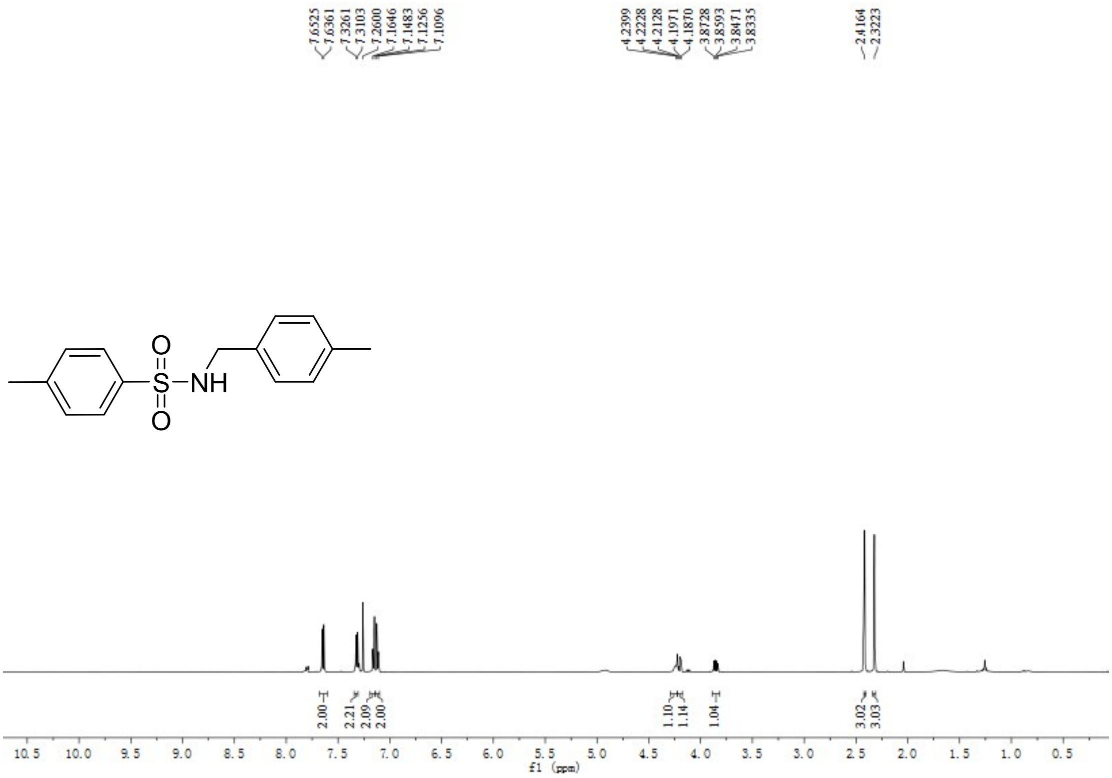
77.0296
76.7754
47.9077
25.2071
21.5251
O
S
N
O
f1 (ppm)

3p





3q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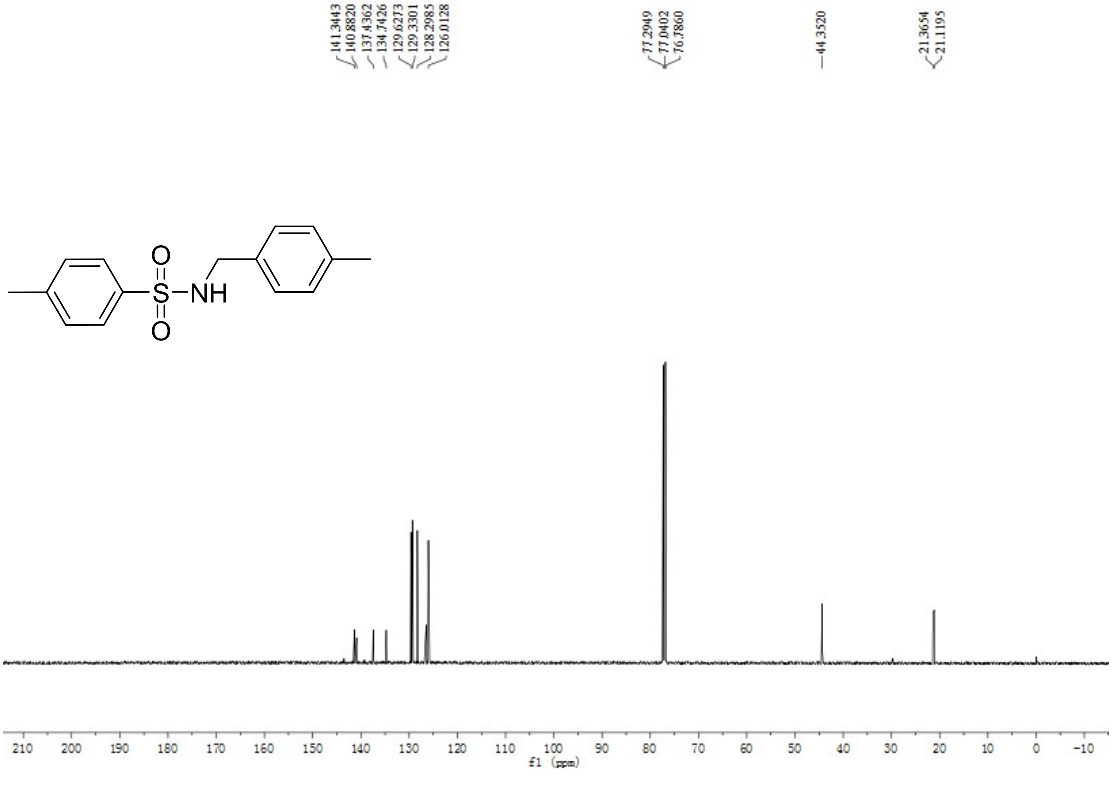
7.3261
7.3103
7.2600
7.1646
7.1483
7.1256
7.1096
4.2399
4.2228
4.2128
4.1971
4.1870
3.8728
3.8593
3.8471
3.8335
2.4164
2.3223
O
S
NH
O
2.00
2.21
2.09
2.00
1.10
1.14
1.04
3.02
3.03
f1 (ppm)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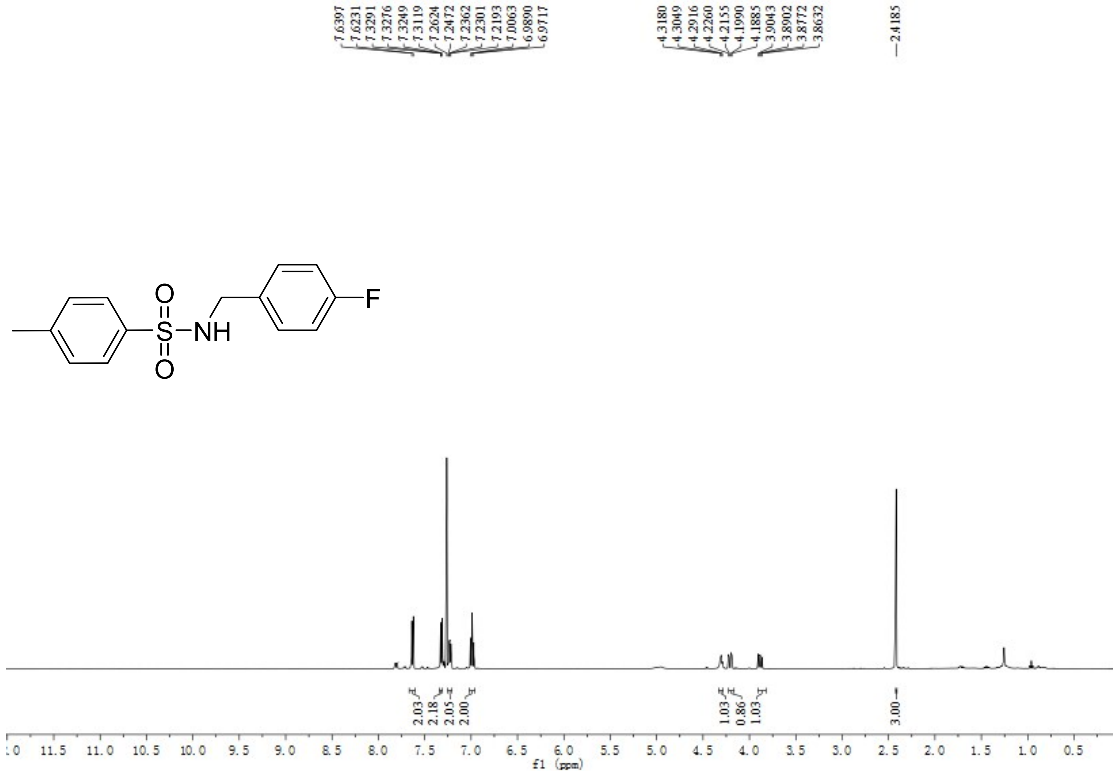
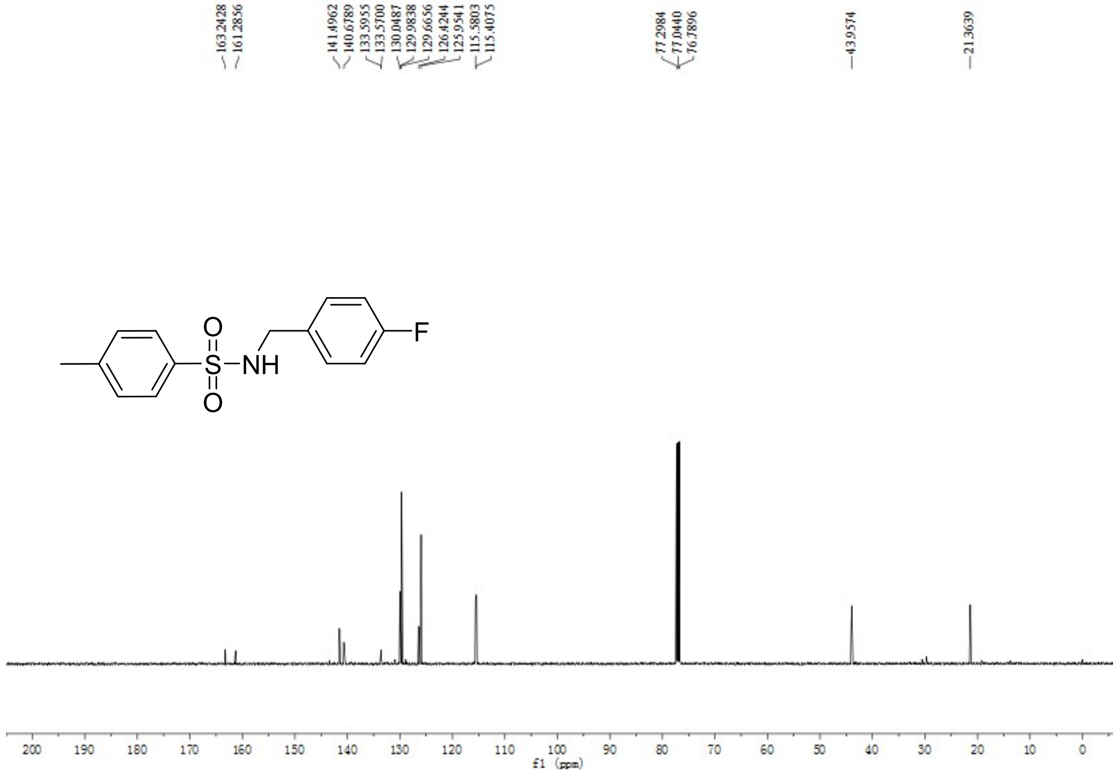
140.8820
137.4362
134.7426
129.6273
129.3301
128.2985
126.0128
77.2949
77.0402
76.7860
44.3520
21.3654
21.1195
O
S
NH
O
f1 (ppm)

3r



7.6397
7.6231
7.3291
7.3276
7.3249
7.3119
7.2624
7.2472
7.2362
7.2301
7.2193
7.0063
6.9890
6.9717
4.3180
4.3049
4.2916
4.2260
4.2155
4.1990
4.1885
3.9043
3.8902
3.8772
3.8632
2.4185
O
S
O
NH
F
2.03
2.18
2.05
2.00
1.03
0.86
1.03
3.00
f1 (ppm)



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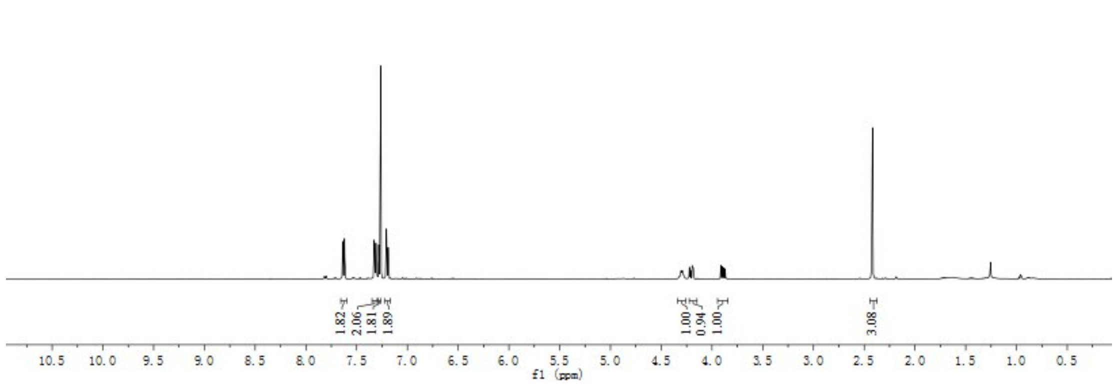
161.2856
141.4962
140.6789
133.5955
133.5700
130.0487
129.9838
129.6656
126.4244
125.9541
115.5803
115.4075
77.2984
77.0440
76.7896
43.9574
21.3639
O
S
O
NH
F
f1 (ppm)

3s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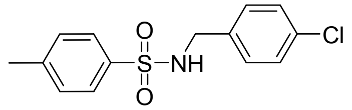
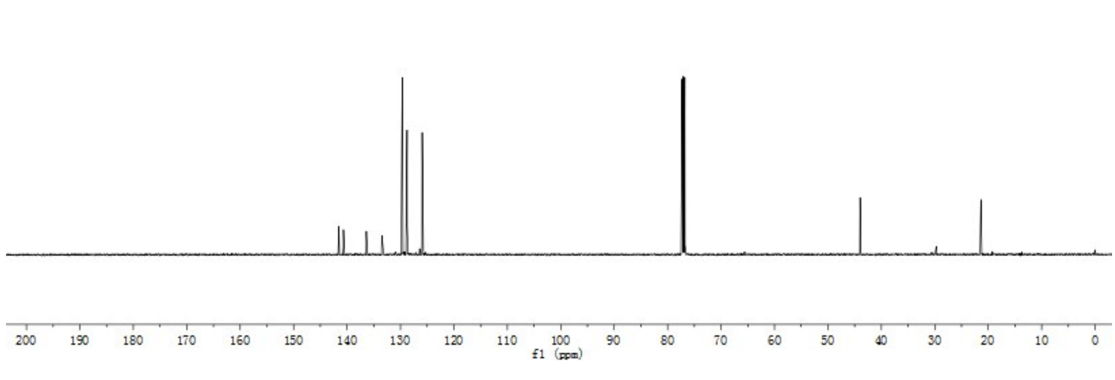
7.6201
7.3267
7.3108
7.2823
7.2657
7.2616
7.2068
7.1904
4.3189
4.3055
4.3012
4.2919
4.2873
4.2779
4.2220
4.2113
4.1945
4.1839
3.9113
3.8972
3.8838
3.8697
2.4181
1.82
2.06
1.81
1.89
1.00
0.94
1.00
3.08
f1 (ppm)


S
O
O
NH
Cl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136.3713
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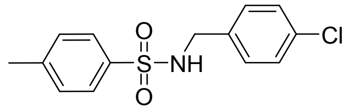
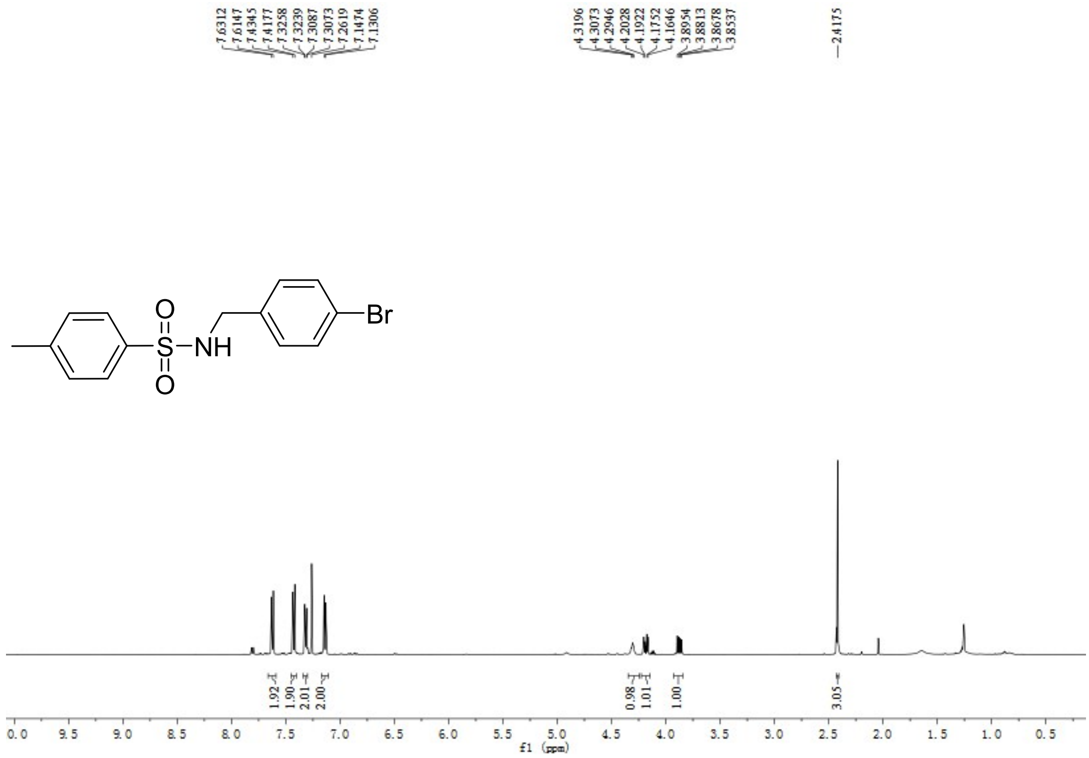
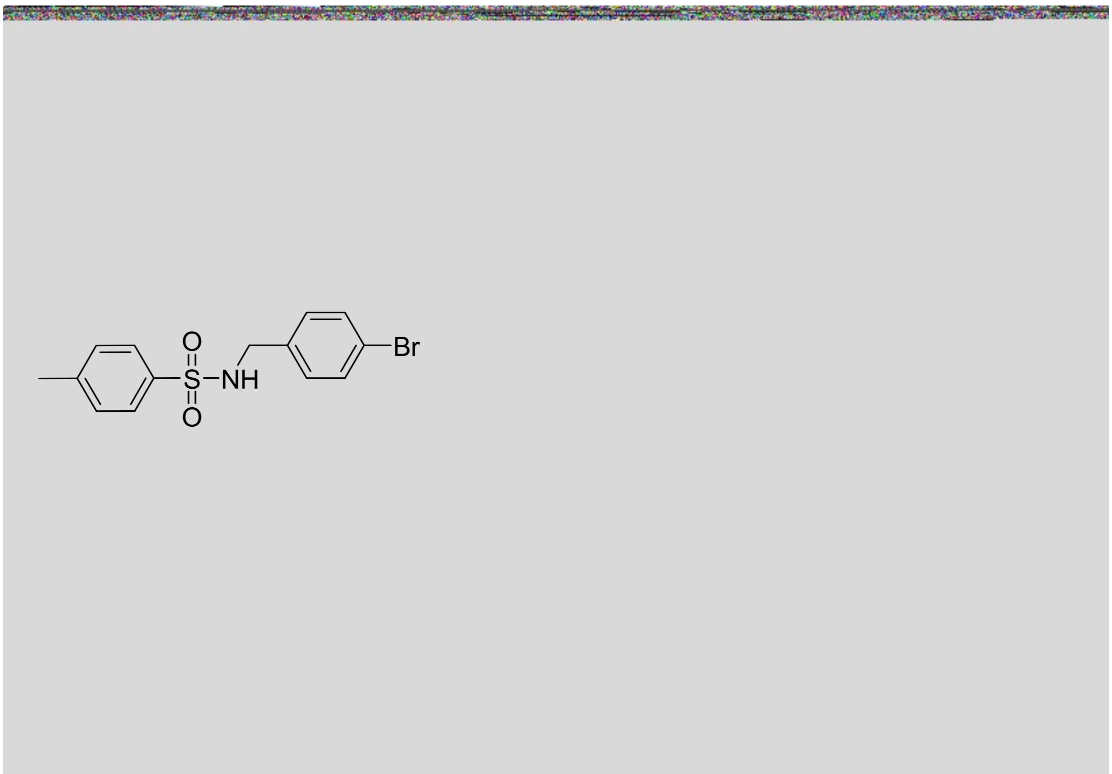
129.6676
128.7668
125.9414
77.3023
77.0467
76.7933
43.9826
21.3684
f1 (ppm)


S
O
O
NH
Cl

3t

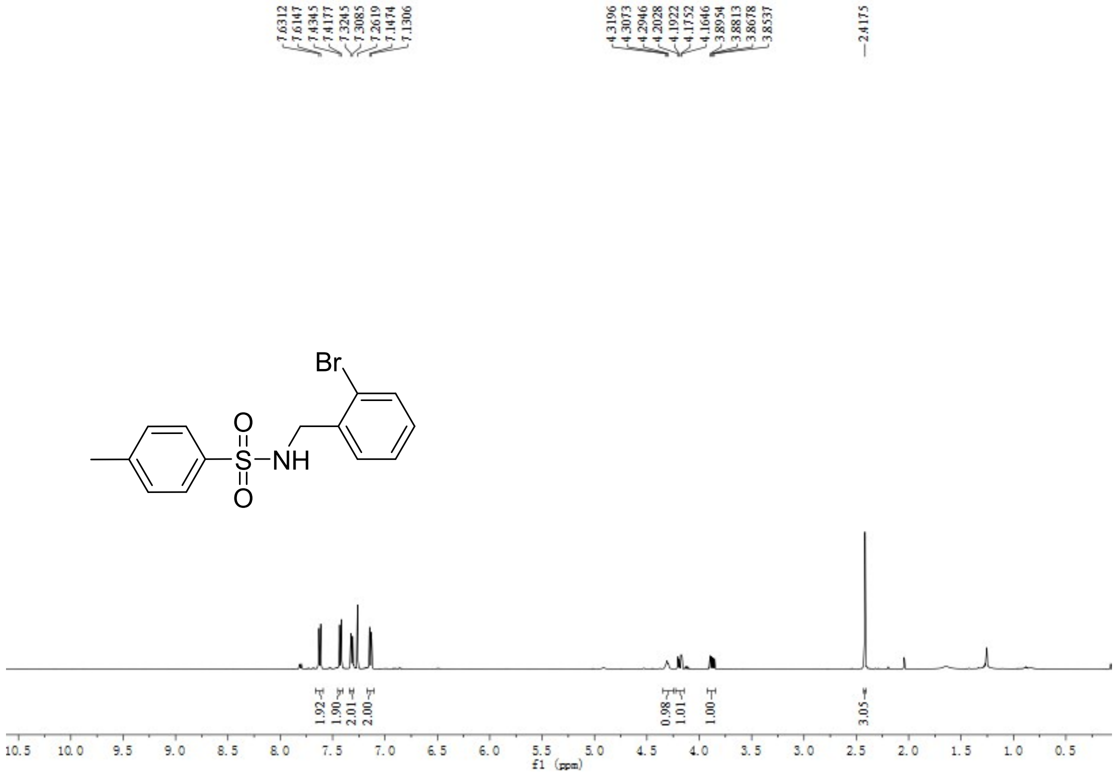


7.6312
7.6147
7.4345
7.4177
7.3258
7.3239
7.3087
7.3073
7.2619
7.1474
7.1306
4.3196
4.3073
4.2946
4.2028
4.1922
4.1752
4.1646
3.8954
3.8813
3.8678
3.8537
2.4175
O
S
NH
O
Br
1.92
1.90
2.01
2.00
0.98
1.01
1.00
3.05
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



O
S
NH
O
Br

3u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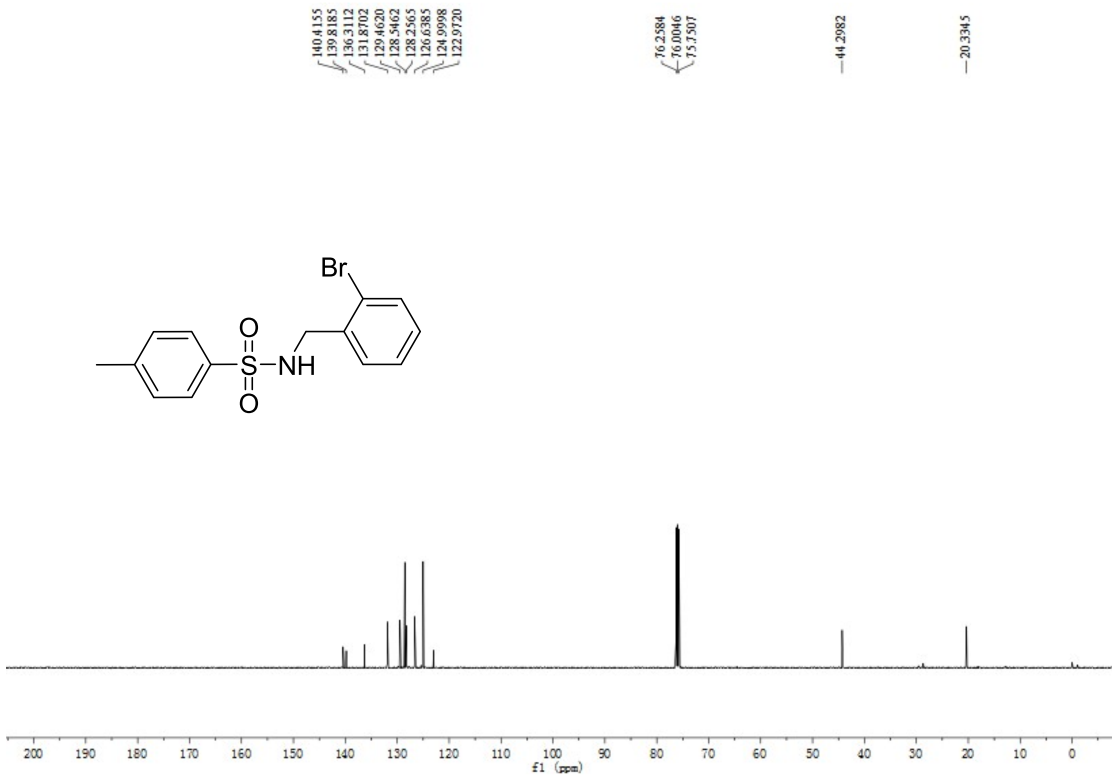
7.6147
7.4345
7.4177
7.3245
7.3085
7.2619
7.1474
7.1306
4.3196
4.3073
4.2946
4.2028
4.1922
4.1752
4.1646
3.8954
3.8813
3.8678
3.8537
2.4175
Br
O
S
NH
O
1.92
1.90
2.01
2.00
0.98
1.01
1.00
3.05
f1 (ppm)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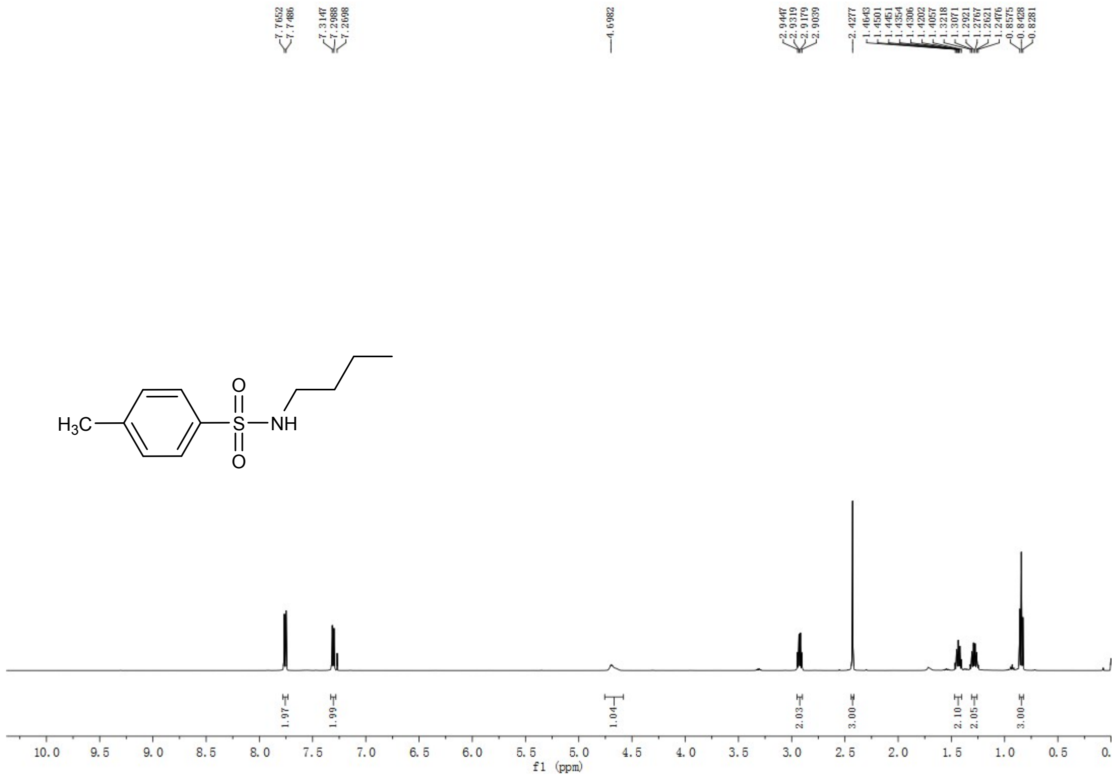
139.8185
136.3112
131.8702
129.4620
128.5462
128.2565
126.6385
124.9998
122.9720
76.2584
76.0046
75.7507
44.2982
20.3345
Br
O
S
NH
O
f1 (ppm)

3v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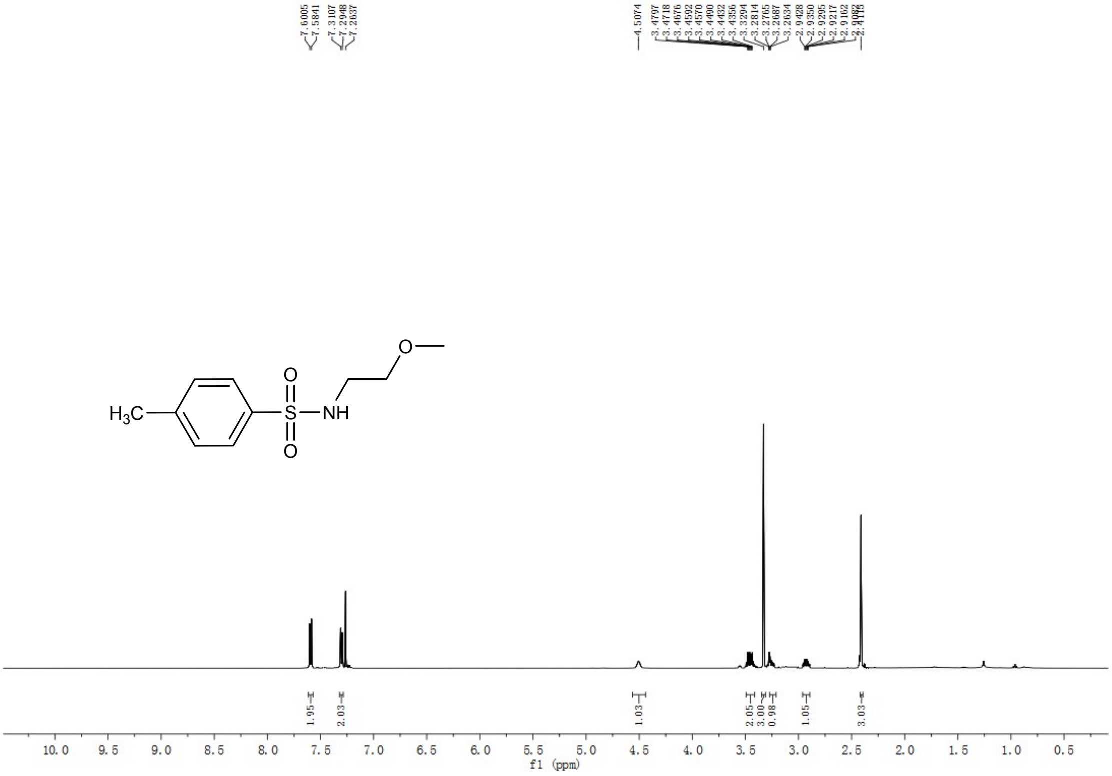
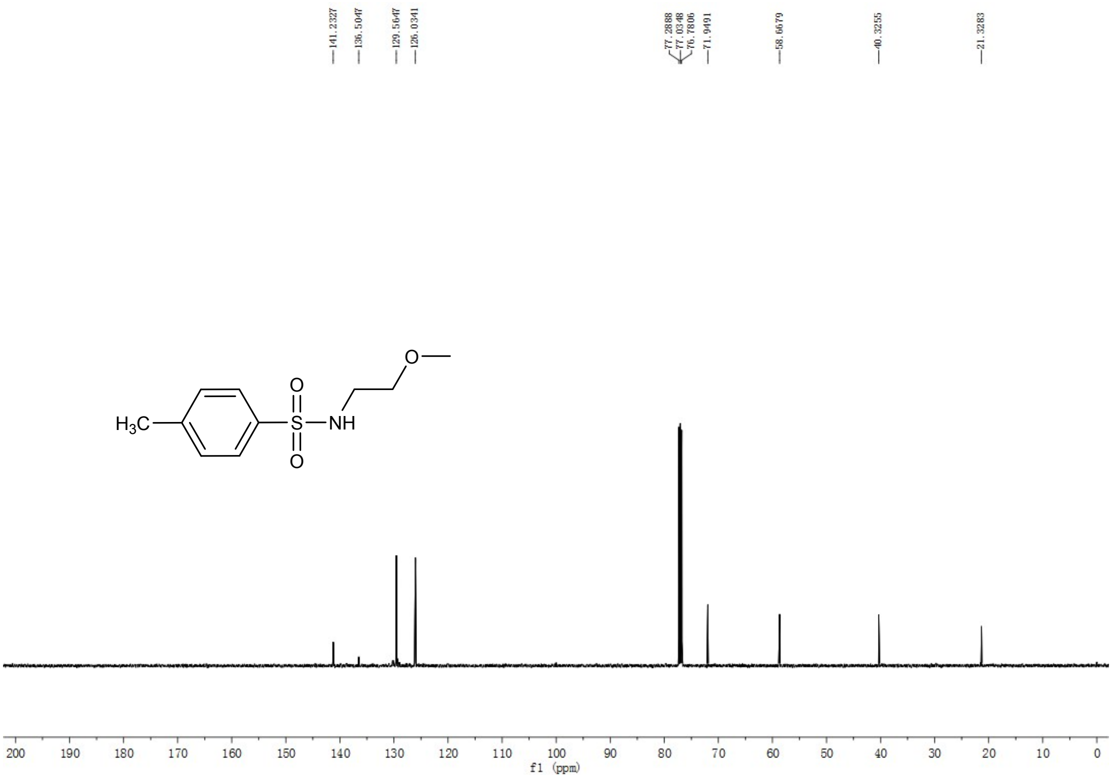
7.3147
7.2988
7.2698
4.6982
2.9447
2.9319
2.9179
2.9039
2.4277
H_3C
S
O
O
NH
1.97
1.99
1.04
2.03
3.00
2.10
2.05
3.00
f1 (ppm)

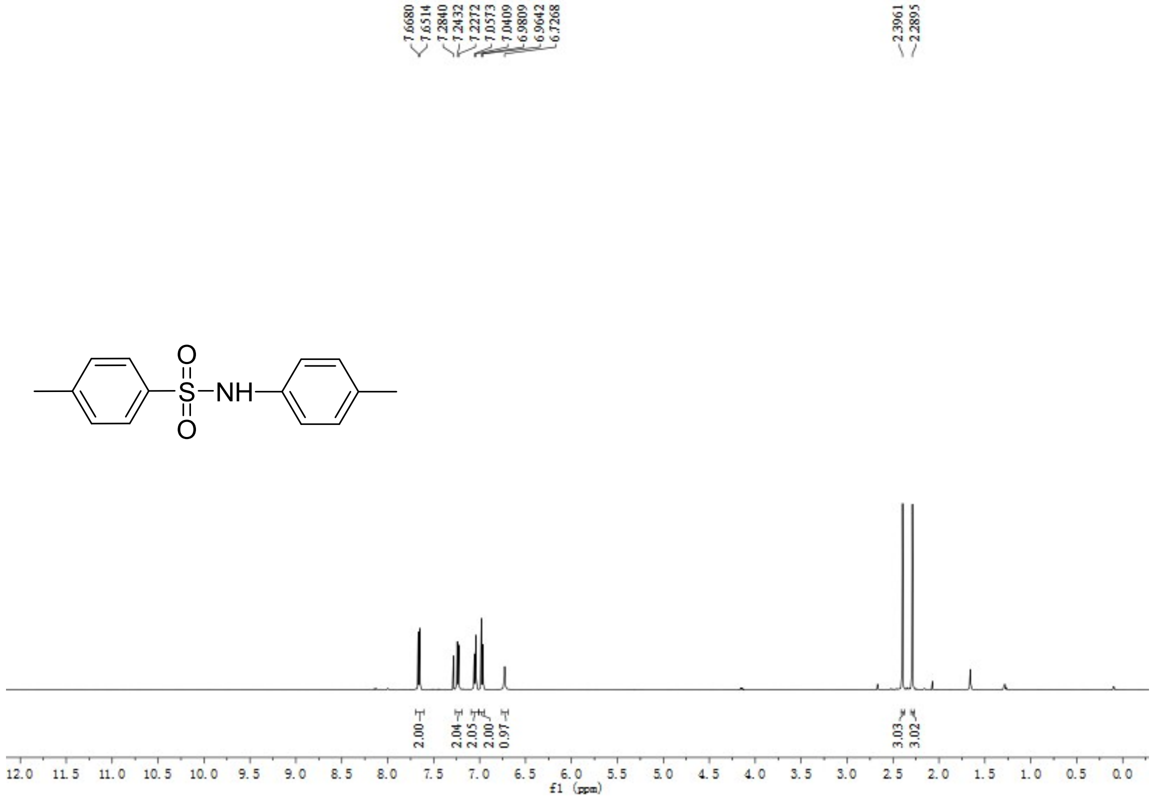

143.2973
137.0245
129.6736
127.1091
77.3073
77.0531
76.7989
42.9198
31.5674
21.5058
19.6905
13.5192
H_3C
S
O
O
NH
f1 (ppm)

3w





3x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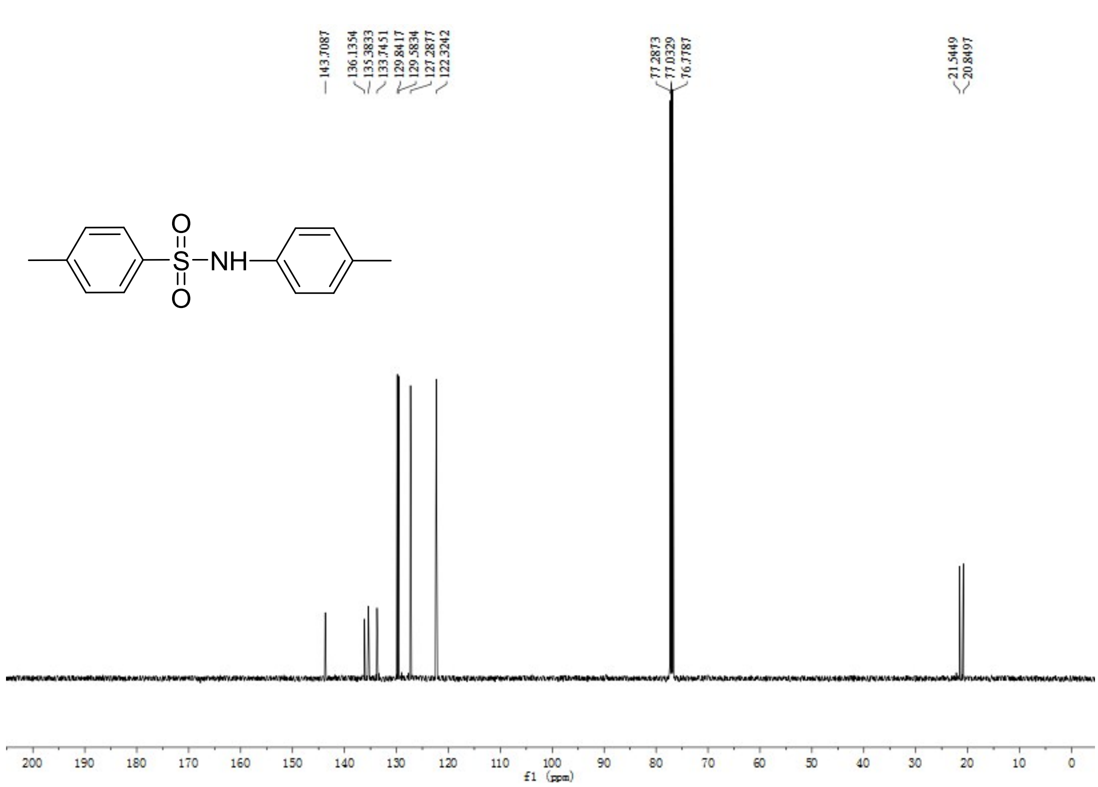
7.2272
7.0573
7.0409
6.9809
6.9642
6.7268
2.3961
2.2895
NH
2.00
2.04
2.05
2.00
0.97
3.03
3.02
f1 (ppm)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129.5834
127.2877
122.3242
77.2873
77.0329
76.7787
21.5449
20.8497
NH
f1 (ppm)