Sulfonamide Formation from Sodium Sulfinates and Amines or Ammonia under Metal-Free Conditions at Ambient Temperature

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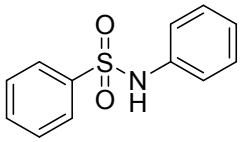
General information

All experiments were conducted with a round-bottom flask. Flash column chromatography was performed over silica gel (200-300 mesh). ^{1}H NMR spectra were recorded on a Bruker AVIII-500M spectrometers, Chemical shifts (in ppm) were referenced to $CDCl_3$ (δ = 7.26 ppm), DMSO-d_6 (δ = 2.50 ppm) as an internal standard. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.0 ppm), DMSO-d_6 (δ = 39.6 ppm). Unless otherwise noted, materials obtained from commercial suppliers were used without further purification, and the most starting materials were purchased from Adamas.

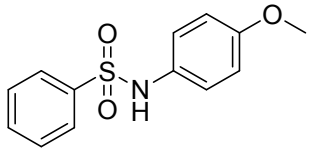
General Procedure for the Preparation of Sodium Sulfinates (2c-2e, 2f-2o)[1]:

4-Methoxybenzenesulfinic acid sodium salt (**2c**) was prepared by heating 2.5 g (20 mmol) of sodium sulfite, 2.06 g (10 mmol) of 4-methoxybenzenesulphonyl chloride, and 1.68 (20 mmol) g of sodium bicarbonate in 10 mL of water at 80 ^{o}C for 4 h. After cooling to room temperature, water was removed under vacuumand the residue was extracted by ethanol, recrystallization as a white solid, the yield was 62% (1.2 g). Similarly, other sodium arenesulfinates (**2d**, **2e**, **2f-2o**) was prepared from their corresponding sulphonyl chlorides.

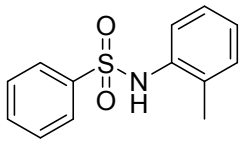
General procedure A for preparation of sulfonamides from amines and sodium sulfinates: To a round-bottom flask was added the corresponding sodium sulfinates (1 mmol) and I_2 (0.5 mmol), the mixture was stirred at room temperature for 20 min, then EtOH (2 mL) and amine (0.5 mmol) was added. The resulting solution was continued to stir at room temperature for 3 h. Upon completion of the reaction, 10% sodium thiosulfate solution (20 mL) was added, aqueous layers was extracted with ethyl acetate (20 mL) thrice. The combine organic layers were dried over anhydrous Na_2SO_4. The solvents were removed via rotary evaporator and the residue was purified with flash chromatography (silica gel).



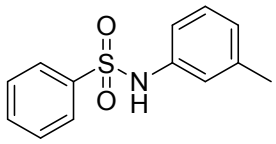
N-phenylbenzenesulfonamide (3aa),[2] the same procedure was used for aniline and sodium benzenesulfinate. The reaction gave 88.5 mg of N-phenylbenzenesulfonamide (3aa) in 76% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.82-7.80 (m, 2H), 7.54-7.51 (m, 1H), 7.44-7.41 (m, 2H), 7.24-7.21 (m, 2H), 7.11-7.09 (m, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 138.9, 136.4, 133.0, 129.3, 129.0, 127.2, 125.4, 121.6.



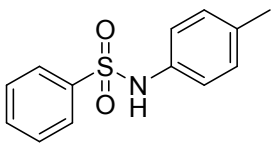
N-(4-methoxyphenyl)benzenesulfonamide (3ba),[3] the same procedure was used for 4-methoxyaniline and sodium benzenesulfinate. The reaction gave 105.2 mg of N-(4-methoxyphenyl)benzenesulfonamide (3ba) in 80 % isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.74-7.72 (m, 2H), 7.53-7.50 (m, 1H), 7.43-7.40 (m, 2H), 7.03 (s, 1H), 6.99 (d, J = 9.0 Hz, 2H), 6.74 (d, J = 9.0 Hz, 2H), 3.73 (s, 3H).^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 157.9, 138.8, 132.8, 128,9, 128.7, 127.2, 125.3, 114.4, 55.4.



N-(o-tolyl)benzenesulfonamide (3ca),[3] the same procedure was used for o-toluidine and sodium benzenesulfinate. The reaction gave 56.8 mg of N-(o-tolyl)benzenesulfonamide (3ca) in 46% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.74 (d, J = 7.0 Hz, 2H), 7.54 (t, J = 7.5 Hz, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.31 (t, J = 7.5 Hz, 1H), 7.15-7.07 (m, 2H), 6.74 (s, 1H), 1.99 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 139.6, 134.3, 132.9, 131.7, 130.7, 128.9, 127.0, 126.9, 126.3, 124.6, 17.5.

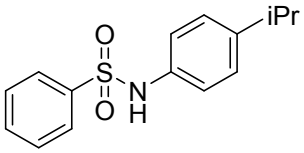


N-(m-tolyl)benzenesulfonamide (3da),[4] The same procedure was used for m-toluidine and sodium benzenesulfinate. The reaction gave 67.9 mg of N-(m-tolyl)benzenesulfonamide (3da) in 55 % isolated yield as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (dd, J = 8.0, 1.0 Hz, 2H), 7.54-7.51 (m, 1H), 7.45-7.42 (m, 2H), 7.23 (s, 1H), 7.11-7.08 (m, 1H), 6.91– 6.89 (m, 3H), 2.25 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 139.3, 139.0, 136.3, 132.9, 129.0, 127.2, 126.1, 122.2, 118.4, 21.3.

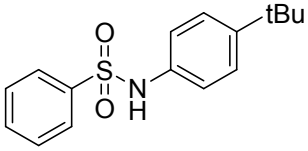


N-(p-tolyl)benzenesulfonamide (3ea),[2] the same procedure was used for p-toluidine and

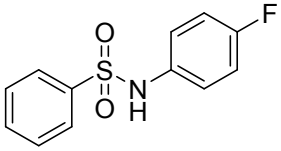
sodium benzenesulfinate. The reaction gave 98.8 mg of N-(p-tolyl)benzenesulfonamide (3ea) in 80 % isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.79-7.77 (m, 2H), 7.52 (t, J = 7.5 Hz, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.09 (s, 1H), 7.02 (d, J = 8.0 Hz, 2H), 6.97 (d, J = 8.0 Hz, 2H), 2.26 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 138.9, 135.4, 133.6, 132.8, 129.8, 127.2, 122.3, 20.8.



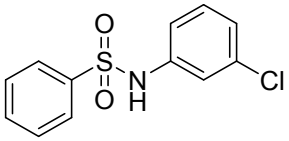
N-(4-isopropylphenyl)benzenesulfonamide (3fa, 116752-56-8), the same procedure was used for 4-isopropylaniline and sodium benzenesulfinate. The reaction gave 151.1 mg of N-(4-isopropylphenyl)benzenesulfonamide (3fa) in 91% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.81-7.79 (m, 2H), 7.52 (t, J = 7.5 Hz, 1H), 7.42 (t, J = 8.0 Hz, 2H), 7.16 (s, 1H), 7.07 (d, J = 8.5 Hz, 2H), 7.00 (d, J = 8.5 Hz, 2H), 2.85-2.79 (m, 1H), 1.18 (d, J = 6.5 Hz, 6H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 146.3, 139.1, 133.8, 132.8, 128.9, 127.2, 127.1, 122.2, 33.4, 23.9



N-(4-(tert-butyl)phenyl)benzenesulfonamide (3ga),[4] the same procedure was used for 4-(tert-butyl)aniline and sodium benzenesulfinate. The reaction gave 119.9 mg of N-(4-(tert-butyl)phenyl)benzenesulfonamide (3ga) in 83 % isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.79 (d, J = 7.5 Hz, 2H), 7.55-7.51 (m, 1H), 7.45-7.42 (m, 2H), 7.24 (d, J = 8.5 Hz, 2H), 6.99 (d, J = 8.5 Hz, 2H), 6.92 (s, 1H), 1.25 (s, 9H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 148.6, 139.2, 133.5, 132.9, 128.9, 127.2, 126.1, 121.7, 34.3, 31.2.

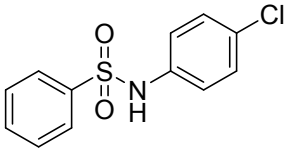


N-(4-fluorophenyl)benzenesulfonamide (3ha),[5] the same procedure was used for 4-fluoroaniline and sodium benzenesulfinate. The reaction gave 79.0 mg of N-(4-fluorophenyl)benzenesulfonamide (3ha) in 63% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.77-7.75 (m, 2H), 7.56-7.53 (m, 1H), 7.45-7.42 (m, 2H), 7.09 (s, 1H), 7.20 (s, 1H), 7.07-7.04 (m, 2H), 6.93-6.90 (m, 2H), 2.26 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 161.6, 159.7, 138.6, 133.1, 132.1, 129.1, 127.2, 124.7, 124.6, 116.2, 116.0.

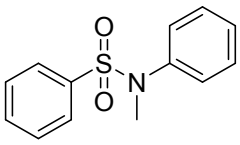


N-(3-chlorophenyl)benzenesulfonamide (3ia),[4] The same procedure was used for 3-chloroaniline and sodium benzenesulfinate. The reaction gave 66.8 mg of N-(3-chlorophenyl)benzenesulfonamide (3ia) in 50% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.84-7.82 (m, 2H), 7.5-7.54 (m, 1H), 7.48-7.45 (m, 2H), 7.38 (s, 1H),

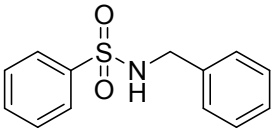
7.15-7.13 (m, 2H), 7.07-7.05 (m, 1H), 6.99-6.97 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 138.6, 137.7, 134.9, 133.3, 130.3, 129.2, 127.2, 125.3, 121.1, 119.1.



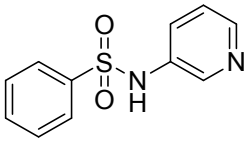
N-(4-chlorophenyl)benzenesulfonamide (3ja),[3] the same procedure was used for 4-chloroaniline and sodium benzenesulfinate. The reaction gave 86.8 mg of N-(4-chlorophenyl)benzenesulfonamide (3ja) in 65% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.79 (d, J = 7.0 Hz, 2H), 7.55 (t, J = 7.0 Hz, 1H), 7.45 (t, J = 7.0 Hz, 3H), 7.18 (d, J = 8.5 Hz, 2H), 7.04 (d, J = 9.0 Hz, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 138.5, 134.9, 133.2, 130.9, 129.4, 129.1, 127.2, 123.0.



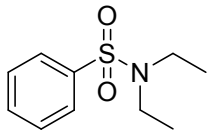
N-methyl-N-phenylbenzenesulfonamide (3ka),[6] the same procedure was used for N-methylaniline and sodium benzenesulfinate. The reaction gave 56.8 mg of N-methyl-N-phenylbenzenesulfonamide (3ka) in 46% isolated yield as a white solid.^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.57-7.53 (m, 3H), 7.46-7.43 (m, 2H), 7.31-7.24 (m, 3H), 7.09-7.07 (m, 2H), 3.17 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 141.4, 136.4, 132.7, 128.8, 128.7, 127.8, 127.3, 126.6, 38.1.



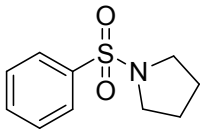
N-benzylbenzenesulfonamide (3la),[2] the same procedure was used for phenylmethanamine and sodium benzenesulfinate. The reaction gave 79.0 mg of N-benzylbenzenesulfonamide (3la) in 64% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.87-7.85 (m, 2H), 7.59-7.56 (m, 1H), 7.51-7.48 (m, 2H), 7.26-7.24 (m, 3H), 7.19-7.17 (m, 2H), 5.00 (s, 1H), 4.14 (d, J = 6.0 Hz, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 139.9, 136.2, 132.6, 129.1, 128.6, 127.9, 127.8, 127.0, 47.2.



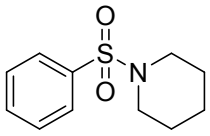
N-(pyridin-3-yl)benzenesulfonamide (3ma),[3] The same procedure was used for pyridin-3-amine and sodium benzenesulfinate. The reaction gave 19.9 mg of N-(pyridin-3-yl)benzenesulfonamide (3na) in 17% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 10.58 (s, 1H), 8.27-8.24 (m, 2H), 7.77-7.75 (m, 2H), 7.62 (d, J = 7.5 Hz, 1H), 7.56 (d, J = 7.5 Hz, 2H), 7.51-7.49 (m, 1H), 7.28 (dd, J_1 = 8.0 Hz, J_2 = 5.0 Hz, 1H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 145.5, 141.9, 139.1, 134.5, 133.3, 129.5, 127.5, 126.7, 124.1.



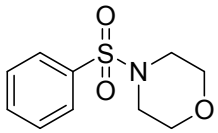
N,N-diethylbenzenesulfonamide (3na),[2] the same procedure was used for diethylamine and sodium benzenesulfinate. The reaction gave 87.3 mg of N,N-diethylbenzenesulfonamide (3ma) in 82% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.82-7.80 (m, 2H), 7.56-7.53 (m, 1H), 7.50-7.47 (m, 2H), 3.24 (q, J = 7.0 Hz, 4H), 1.12 (t, J = 7.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 140.4, 132.2, 129.0, 126.9, 42.0, 14.1.



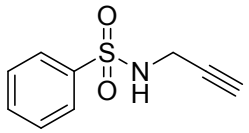
1-(phenylsulfonyl)pyrrolidine (3oa),[7] the same procedure was used for pyrrolidine and sodium benzenesulfinate. The reaction gave 66.5 mg of 1-(phenylsulfonyl)pyrrolidine (3oa) in 63% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.82 (d, J = 8.0 Hz, 2H), 7.58 (t, J = 7.5 Hz, 1H), 7.52 (t, J = 7.5 Hz, 2H), 3.23 (t, J = 6.0 Hz, 4H), 1.75-1.72 (m, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 136.9, 132.5, 128.9, 127.4, 47.9, 25.2.



1-(phenylsulfonyl)piperidine (3pa),[7] the same procedure was used for piperidine and sodium benzenesulfinate. The reaction gave 79.8 mg of 1-(phenylsulfonyl)piperidine (3pa) in 71% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.76-7.74 (m, 2H), 7.60-7.57 (m, 1H), 7.54-7.51 (m, 2H), 2.98 (t, J = 5.0 Hz, 4H), 1.65-1.61 (m, 4H), 1.43-1.39 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 136.3, 132.5, 128.9, 127.6, 46.9, 25.1, 23.5.

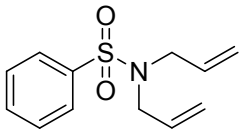


4-(phenylsulfonyl)morpholine (3qa),[8] the same procedure was used for morpholine and sodium benzenesulfinate. The reaction gave 107.8 mg of 4-(phenylsulfonyl)morpholine (3qa) in 95% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.77-7.75 (m, 2H), 7.64-7.61 (m, 1H), 7.57-7.54 (m, 2H), 3.74 (t, J = 5.0 Hz, 4H), 3.00 (t, J = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 135.1, 133.0, 129.1, 127.8, 66.1, 46.0.

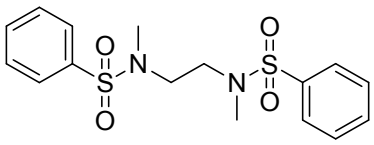


N-(prop-2-yn-1-yl)benzenesulfonamide (3ra),[9] The same procedure was used for prop-2-yn-1-amine and sodium benzenesulfinate at 0 ^{o}C-rt. The reaction gave 57.5 mg of N-(prop-2-yn-1-yl)benzenesulfonamide (3ra) in 59% isolated yield as a light yellow oil. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.90 (d, J = 8.0 Hz, 2H), 7.60 – 7.57 (m, 1H), 7.52 (t, J = 7.5 Hz, 2H),

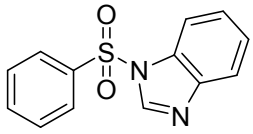
5.04 (s, 1H), 3.84 (s, 2H), 2.07-2.06 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 139.5, 132.9, 129.0, 127.3, 77.8, 73.0, 32.8.



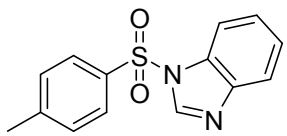
N,N-diallylbenzenesulfonamide (3sa),[10] the same procedure was used for diallylamine and sodium benzenesulfinate at 0 ^{o}C-rt. The reaction gave 99.5 mg of N,N-diallylbenzenesulfonamide (3sa) in 42% isolated yield as a light yellow oil. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.82 (d, J = 7.5 Hz, 2H), 7.58-7.55 (m, 1H), 7.50 (t, J = 7.5 Hz, 2H), 5.63-5.55 (m, 2H), 5.15 – 5.12 (m, 4H), 3.81 (d, J = 6.5 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 140.4, 134.5, 129.0, 127.1, 119.0, 49.3.



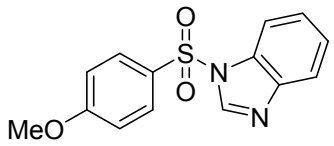
N,N'-(ethane-1,2-diyl)bis(N-methylbenzenesulfonamide) (3ta, 60395-33-7), the same procedure was used for N,N'-dimethyl-1,2-ethanediamine (0.25 mmol) and sodium benzenesulfinate. The reaction gave 60.7 mg of N,N'-(ethane-1,2-diyl)bis(N-methylbenzenesulfonamide) (3ta) in 66% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.79-7.77 (m, 4H), 7.61-7.59 (m, 2H), 7.53 (t, J = 8.0 Hz, 4H), 3.23 (s, 4H), 2.83 (s, 6H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 137.3, 132.9, 129.2, 127.3, 48.8, 35.8.



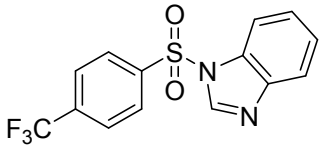
1-(phenylsulfonyl)-1H-benzo[d]imidazole (3ua),[11] the same procedure was used for benzimidazole and sodium benzenesulfinate. The reaction gave 51.6 mg of 1-(phenylsulfonyl)-1H-benzo[d]imidazole (3ua) in 40% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.39 (s, 1H), 7.99 (d, J = 8.0 Hz, 2H), 7.87 (d, J = 8.0 Hz, 1H), 7.77 (d, J = 7.5 Hz, 1H), 7.61 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 8.0 Hz, 2H), 7.41-7.34 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 144.0, 141.1, 137.5, 134.7, 130.7, 129.7, 127.1, 125.6, 124.8, 121.1, 112.4.



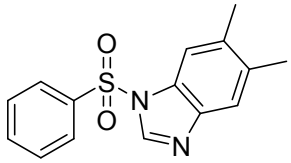
1-tosyl-1H-benzo[d]imidazole (3ub),[12] the same procedure was used for benzimidazole and sodium 4-methylbenzenesulfinate. The reaction gave 65.3 mg of 1-tosyl-1H-benzo[d]imidazole (3ub) in 48% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.38 (s, 1H), 7.88-7.85 (m, 3H), 7.76 (d, J = 7.5 Hz, 1H), 7.40-7.33 (m, 2H), 7.29 (d, J = 7.5 Hz, 2H), 2.37 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 146.2, 144.0, 141.2, 134.5, 130.7, 130.3, 127.2, 125.5, 124.7, 121.0, 112.4, 21.6.



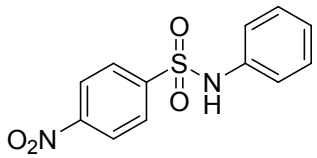
1-((4-methoxyphenyl)sulfonyl)-1H-benzo[d]imidazole (3uc),[12] the same procedure was used for benzimidazole and sodium 4-methoxybenzenesulfinate. The reaction gave 46.1 mg of 1-((4-methoxyphenyl)sulfonyl)-1H-benzo[d]imidazole (3uc) in 32% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.38 (s, 1H), 7.92 (d, J = 9.0 Hz, 2H), 7.84 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.39-7.33 (m, 2H), 6.93 (d, J = 9.0 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 164.5, 143.9, 141.2, 130.7, 129.6, 128.6, 125.4, 124.6, 121.0, 114.9, 112.4, 55.8.



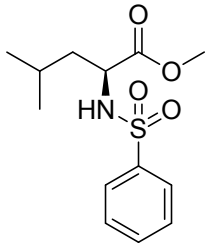
1-((4-(trifluoromethyl)phenyl)sulfonyl)-1H-benzo[d]imidazole (3ud, 1030365-08-2), the same procedure was used for benzimidazole and sodium 4-(trifluoromethyl)benzenesulfinate. The reaction gave 70.1 mg of 1-((4-(trifluoromethyl)phenyl)sulfonyl)-1H-benzo[d]imidazole (3ud) in 43% isolated yield as a white solid.^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.38 (s, 1H), 8.13 (d, J = 8.0 Hz, 2H), 7.88-7.86 (m, 1H), 7.79 (d, J = 8.0 Hz, 3H), 7.45-7.38 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 144.0, 140.9, 136.3 (q, J = 33.8 Hz), 130.6, 127.7, 126.9 (q, J = 3.8 Hz), 126.0, 125.3, 122.7 (q, J = 271.3 Hz), 121.4, 112.3.



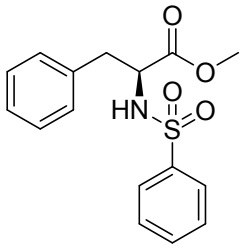
5,6-dimethyl-1-(phenylsulfonyl)-1H-benzo[d]imidazole (3va, 325810-41-1), the same procedure was used for5,6-dimethyl-1H-benzo[d]imidazole and sodium benzenesulfinate. The reaction gave 100.1 mg of 5,6-dimethyl-1-(phenylsulfonyl)-1H-benzo[d]imidazole (3va) in 70% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.27 (s, 1H), 7.98-7.96 (m, 2H), 7.63 (s, 1H), 7.63-7.58 (m, 1H), 7.51-7.48 (m, 3H), 2.37 (m, 3H), 2.32 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 142.4, 140.4, 137.7, 135.0, 134.5, 133.9, 129.6, 129.1, 126.9, 121.0, 112.5, 20.6, 20.1



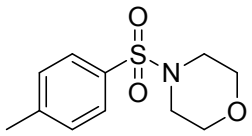
4-nitro-N-phenylbenzenesulfonamide (3ae),[13] the same procedure was used for aniline and sodium 4-nitrobenzenesulfinate. The reaction gave 44.5 mg of 4-nitro-N-phenylbenzenesulfonamide (3ae) in 32% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 10.61 (s, 1H), 8.36 (t, J = 9.0 Hz, 2H), 7.99 (t, J = 9.0 Hz, 2H), 7.27-7.23 (m, 2H), 7.11-7.05 (m, 3H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 149.9, 144.9, 137.0, 129.5, 128.4, 124.9, 124.7, 120.8.



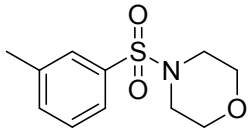
(S)-methyl 4-methyl-2-(phenylsulfonamido)pentanoate (3wa, 68305-82-8), the same procedure was used for methyl L-leucinate hydrochloride, sodium benzenesulfinate and Et_3N (1 eq). The reaction gave 52.7 mg of (S)-methyl 4-methyl-2-(phenylsulfonamido)pentanoate (3wa) in 37% isolated yield as a colorless oil. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.84-7.83 (m, 2H), 7.58-7.55 (m, 1H), 7.51-7.48 (m, 2H), 5.14 (d, J = 10 Hz, 1H), 3.96-3.92 (m, 1H), 3.41 (s, 3H), 1.80- 1.73 (m, 1H), 1.51-1.44 (m, 2H), 0.89 (d, J = 6.5 Hz, 3H), 0.86 (d, J = 6.5 Hz, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 172.6, 139.6, 132.8, 128.9, 127.3, 54.3, 52.3, 42.3, 24.2, 22.7, 21.3.



(S)-methyl 3-phenyl-2-(phenylsulfonamido)propanoate (3xa),[14] the same procedure was used for methyl L-phenylalaninate hydrochloride, sodium benzenesulfinate and Et_3N (1 eq). The reaction gave 57.4 mg of (S)-methyl 3-phenyl-2-(phenylsulfonamido)propanoate (3xa) in 36% isolated yield as a colorless oil. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.76-7.40 (m, 2H), 7.56-7.53 (m, 1H), 7.46-7.43 (m, 2H), 7.25-7.23 (m, 3H), 7.07-7.05 (m, 2H), 5.15 (s, 1H), 4.25-4.20 (m, 1H), 3.47 (s, 3H), 3.04-3.03 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 171.1, 139.5, 134.8, 132.7, 129.3, 129.0, 128.6, 127.3, 127.1, 56.6, 52.4, 39.3.

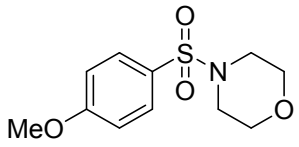


4-tosylmorpholine (3qb),[15] the same procedure was used for morpholine and sodium 4-methylbenzenesulfinate. The reaction gave 85.5 mg of 4-tosylmorpholine (3qb) in 71% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.63 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 3.73 (t, J = 4.5 Hz, 4H), 2.97 (t, J = 4.5 Hz, 4H), 2.43 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 143.9, 132.0, 129.7, 127.8, 66.1, 45.9, 21.5.

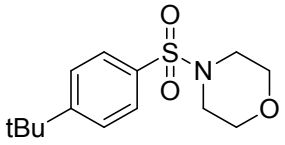


4-(m-tolylsulfonyl)morpholine (3qf),[16] the same procedure was used for morpholine and sodium 3-methylbenzenesulfinate. The reaction gave 98.8 mg of 4-(m-

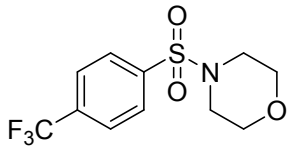
tolylsulfonyl)morpholine (3qf) in 82% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.55-7.53 (m, 2H), 7.45-7.42 (m, 2H), 3.73 (t, J = 5.0 Hz, 4H), 2.99 (t, J = 5.0 Hz, 4H), 2.44 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 139.3, 134.9, 133.8, 128.9, 128.1, 124.9, 66.1, 46.0, 21.4.



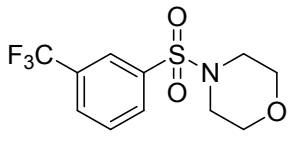
4-((4-methoxyphenyl)sulfonyl)morpholine (3qc),[17] the same procedure was used for morpholine and sodium 4-methoxybenzenesulfinate. The reaction gave 113.1 mg of 4-((4-methoxyphenyl)sulfonyl)morpholine (3qc) in 88% isolated yield as a white solid.^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.68 (d, J = 9.0 Hz, 2H), 7.01 (d, J = 9.0 Hz, 2H), 3.87 (s, 3H), 3.72 (t, J = 5.0 Hz, 4H), 2.96 (t, J = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 163.2, 129.9, 126.6, 114.3, 66.0, 55.6, 46.0.



4-((4-(tert-butyl)phenyl)sulfonyl)morpholine (3qg, 324526-64-9), the same procedure was used for morpholine and sodium 4-(tert-butyl)benzenesulfinate. The reaction gave 103.4 mg of 4-((4-(tert-butyl)phenyl)sulfonyl)morpholine (3qg) in 73% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.67 (d, J = 8.0 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 3.74 (t, J = 4.5 Hz, 4H), 2.99 (t, J = 4.5 Hz, 4H), 1.34 (s, 9H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 156.8, 131.9, 127.7, 126.0, 66.1, 45.9, 35.2, 31.4.

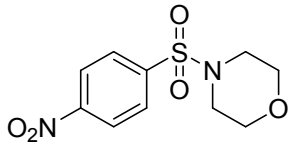


4-((4-(trifluoromethyl)phenyl)sulfonyl)morpholine (3qd, 457960-71-3), the same procedure was used for morpholine and sodium 4-(trifluoromethyl)benzenesulfinate. The reaction gave 57.5 mg of 4-((4-(trifluoromethyl)phenyl)sulfonyl)morpholine (3qd) in 39% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.88 (d, J = 8.0 Hz, 2H), 7.82 (d, J = 8.0 Hz, 2H), 3.74 (t, J = 4.5 Hz, 4H), 3.03 (t, J = 4.5 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 138.9, 134.7 (q, J = 32.5 Hz), 128.3, 126.3 (q, J = 3.8 Hz), 123.1 (q, J = 271.3 Hz), 66.0, 45.9.

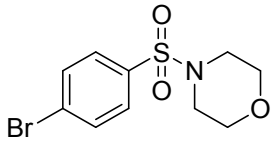


4-((3-(trifluoromethyl)phenyl)sulfonyl)morpholine (3qh, 613657-76-4), the same procedure was used for morpholine and sodium 3-(trifluoromethyl)benzenesulfinate. The reaction gave 67.8 mg of4-((3-(trifluoromethyl)phenyl)sulfonyl)morpholine (3qh) in 46% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.00 (s, 1H), 7.95(d, J = 7.5 Hz, 1H), 7.89 (d, J = 7.5 Hz, `H), 7.73 (t, J = 7.5 Hz, 1H), 3.76 (t, J = 4.5 Hz, 4H), 3.02 (t,

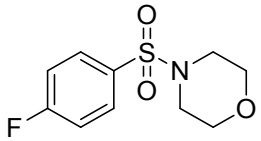
J = 4.5 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 136.6, 131.9 (q, *J* = 33.8 Hz), 130.0, 129.7 (q, *J* = 3.8 Hz), 124.7 (q, *J* = 3.8 Hz), 123.1 (q, *J* = 271.3 Hz), 66.0, 45.9.



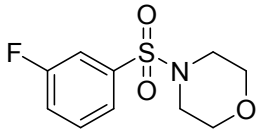
4-((4-nitrophenyl)sulfonyl)morpholine (3qe),[18] the same procedure was used for morpholine and sodium 4-nitrobenzenesulfinate. The reaction gave 70.7 mg of 4-((4-nitrophenyl)sulfonyl)morpholine (3qe) in 52% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.40 (d, *J* = 8.5 Hz, 2H), 7.94 (d, *J* = 8.5 Hz, 2H), 3.75 (t, *J* = 4.5 Hz, 4H), 3.05 (t, *J* = 4.5 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 150.3, 141.3, 128.9, 124.4, 66.0, 45.8.



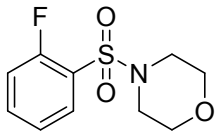
4-((4-bromophenyl)sulfonyl)morpholine (3qi),[19] the same procedure was used for morpholine and sodium 4-bromobenzenesulfinate. The reaction gave 59.4 mg of 4-((4-bromophenyl)sulfonyl)morpholine (3qi) in 39% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.69 (d, *J* = 8.5 Hz, 2H), 7.61 (d, *J* = 8.5 Hz, 2H), 3.74 (t, *J* = 5.0 Hz, 4H), 2.99 (t, *J* = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 134.2, 132.4, 129.3, 128.2, 66.0, 45.9.



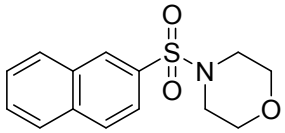
4-((4-fluorophenyl)sulfonyl)morpholine (3qj),[15] the same procedure was used for morpholine and sodium 4-fluorobenzenesulfinate. The reaction gave 91.8 mg of 4-((4-fluorophenyl)sulfonyl)morpholine (3qj) in 75% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.78-7.75 (m, 2H), 7.25-7.21 (m, 2H), 3.73 (t, *J* = 5.0 Hz, 4H), 2.98 (t, *J* = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 165.3 (d, *J* = 253.8 Hz), 131.1 (d, *J* = 3.8 Hz), 130.5 (d, *J* = 8.8 Hz), 116.4 (d, *J* = 22.5 Hz), 66.0, 45.9.



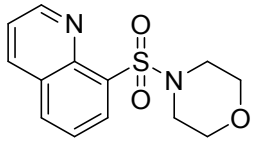
4-((3-fluorophenyl)sulfonyl)morpholine (3qk),[17] the same procedure was used for morpholine and sodium 3-fluorobenzenesulfinate. The reaction gave 77.2 mg of 4-((3-fluorophenyl)sulfonyl)morpholine (3qk) in 63% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ7.56-7.54 (m, 2H), 7.47-7.45 (m, 1H), 7.35-7.31 (m, 1H), 3.74 (t, *J* = 5.0 Hz, 4H), 3.02 (t, *J* = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 162.4 (d, *J* = 255.6 Hz), 137.3 (d, *J* = 6.5 Hz), 130.9 (d, *J* = 7.6 Hz), 123.5 (d, *J* = 3.4 Hz), 120.2 (d, *J* = 21.1 Hz), 115.0 (d, *J* = 24.1 Hz), 66.0, 45.9.



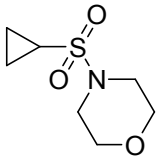
4-((2-fluorophenyl)sulfonyl)morpholine (3ql, 613657-01-5), the same procedure was used for morpholine and sodium 2-fluorobenzenesulfinate. The reaction gave 38.0 mg of 4-((2-fluorophenyl)sulfonyl)morpholine (3ql) in 31% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 7.84-7.81 (m, 1H), 7.62-7.58 (m, 1H), 7.31-7.28 (m, 1H), 7.25-7.22 (m, 1H), 3.74 (t, J = 5.0 Hz, 4H), 3.18 (t, J = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 159.0 (d, J = 255.0 Hz), 135.3 (d, J = 8.7 Hz), 131.3, 124.5 (q, J = 5.0 Hz), 117.3 (d, J = 10 Hz), 66.3, 45.7 (d, J = 1.3 Hz).



4-(naphthalen-2-ylsulfonyl)morpholine (3qm),[20] the same procedure was used for morpholine and sodium naphthalene-2-sulfinate. The reaction gave 84.5 mg of 4-(naphthalen-2-ylsulfonyl)morpholine (3qm) in 61% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 8.33 (s, 1H), 8.00-7.98 (m, 2H), 7.93 (d, J = 8.0 Hz, 1H) 7.75-7.73 (m, 1H), 7.69-7.61 (m, 2H), 3.74 (t, J = 5.0 Hz, 4H), 3.07 (t, J = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 134.9, 132.2, 132.1, 129.3, 129.2, 129.2, 129.0, 127.9, 127.6, 122.9, 66.1, 46.1.



4-(quinolin-8-ylsulfonyl)morpholine (3qn),[20] the same procedure was used for morpholine and sodium quinoline-8-sulfinate. The reaction gave 83.4 mg of 4-(quinolin-8-ylsulfonyl)morpholine (3qn) in 60% isolated yield as a white solid.. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 9.05-9.04 (m, 1H), 8.23 (dd, J_1 = 7.5 Hz, J_2 = 1.5 Hz, 1H), 8.24-8.22 (m, 1H), 8.04-8.02 (m, 1H), 7.60 (t, J = 7.5 Hz, 1H), 7.52 (q, J = 4.0 Hz, 1H) 3.69 (t, J = 5.0 Hz, 4H), 3.43 (t, J = 5.0 Hz, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ13C NMR (126 MHz, CDCl3) δ 151.1, 144.1, 136.4, 136.3, 133.6, 133.2, 128.9, 125.4, 122.0, 66.8, 46.3.

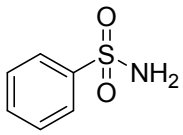


4-(cyclopropylsulfonyl)morpholine (3qo),[15] the same procedure was used for morpholine and sodium cyclopropanesulfinate. The reaction gave 77.4 mg of 4-(cyclopropylsulfonyl)morpholine (3qo) in 81% isolated yield as a white solid. ^{1}H NMR: (500 MHz, $CDCl_3$, ppm) δ 3.75 (t, J = 5.0 Hz, 4H), 2.27 (t, J = 5.0 Hz, 4H), 2.27-2.24 (m, 1H), 1.16-1.15 (m, 2H), 1.00-0.98 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$, ppm) δ 66.4, 46.1, 25.0, 4.2.

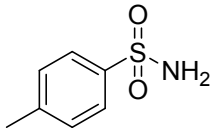
General procedure B_1 for preparation of sulfonamides from ammonia in water and sodium sulfinates: To a round-bottom flask was added the corresponding sodium sulfinates

(0.5 mmol) and I_2 (0.5 mmol), the mixture was stirred at room temperature for 20 min, thenammonium hydroxide (1 mL) was added. The resulting solution was continued to stir at room temperature for 3 h. Upon completion of the reaction, 10% sodium thiosulfate solution (20 mL) was added, aqueous layers was extracted with ethyl acetate (20 mL) thrice. The combine organic layers were dried over anhydrous Na_2SO_4. The solvents were removed via rotary evaporator and the residue was purified with flash chromatography (silica gel).

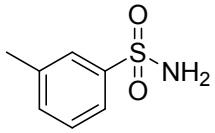
General procedure B_2 for preparation of sulfonamides from ammonia in water and sodium sulfinates: To a round-bottom flask was added the corresponding sodium sulfinates (0.5 mmol) and I_2 (0.5 mmol), the mixture was stirred at room temperature for 20 min, then EtOH (1 mL) and ammonium hydroxide (1 mL) was added. The resulting solution was continued to stir at room temperature for 3 h. Upon completion of the reaction, 10% sodium thiosulfate solution (20 mL) was added, aqueous layers was extracted with ethyl acetate (20 mL) thrice. The combine organic layers were dried over anhydrous Na_2SO_4. The solvents were removed via rotary evaporator and the residue was purified with flash chromatography (silica gel).



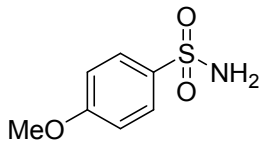
benzenesulfonamide (4a),[21] the procedure B_1 was used for sodium benzenesulfinate. The reaction gave 58.9 mg of benzenesulfonamide (4a) in 75% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.85-7.83 (m, 2H), 7.60-7.57 (m, 3H), 7.37 (s, 2H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 144.2, 131.9, 129.0, 125.7.



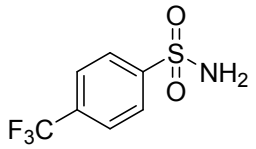
4-methylbenzenesulfonamide (4b),[2] the procedure B_2 was used for sodium 4-methylbenzenesulfinate. The reaction gave 61.2 mg of benzenesulfonamide (4a) in 72% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.71 (d, J = 8.0 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 7.27 (s, 2H), 2.37 (s, 3H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 142.0, 141.5, 129.4, 125.7, 21.0.



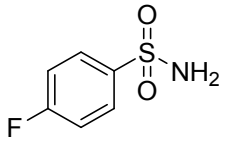
3-methylbenzenesulfonamide (4f),[22] the procedure B_1 was used for odium 3-methylbenzenesulfinate. The reaction gave 42.7 mg of 3-methylbenzenesulfonamide (4f) in 50% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.65-7.62 (m, 2H), 7.45-7.41 (m, 2H), 7.40 (s, 2H), 2.38 (s, 3H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 144.2, 138.6, 132.5, 128.9, 126.0, 122.9, 21.0.



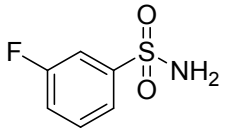
4-methoxybenzenesulfonamide (4c),[23] the procedure B_2 was used for sodium 4-methoxybenzenesulfinate. The reaction gave 58.9 mg of 4-methoxybenzenesulfonamide (4c) in 63% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.76 (d, J = 9.0 Hz, 2H), 7.21 (s, 2H), 7.08 (d, J = 9.0 Hz, 2H), 3.82 (s, 3H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 161.8, 136.3, 127.8, 114.1, 55.7.



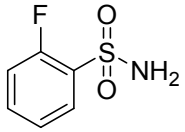
4-(trifluoromethyl)benzenesulfonamide (4d),[24] the procedure B_2 was used for sodium 4-(trifluoromethyl)benzenesulfinate. The reaction gave 56.2 mg of 4-(trifluoromethyl)benzenesulfonamide (4d) in 58% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 8.04 (d, J = 8.5 Hz, 2H), 7.97 (d, J = 8.5Hz, 2H), 7.62 (s, 2H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 147.9, 131.8 (q, J = 31.3 Hz), 126.7, 126.3 (q, J = 3.8 Hz), 123.7 (q, J = 271.3 Hz),



4-fluorobenzenesulfonamide (4j),[23] the procedure B_2 was used for sodium 4-fluorobenzenesulfinate. The reaction gave 669.1 mg of 4-fluorobenzenesulfonamide (4j) in 79% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.89-7.87 (m, 2H), 7.43-7.39 (m, 4H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 163.8 (d, J = 248.8 Hz), 140.7 (d, J = 3.8 Hz), 128.7 (d, J = 8.8 Hz), 116.1 (d, J = 21.3 Hz).

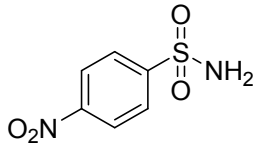


3-fluorobenzenesulfonamide (4k),[25] the procedure B_2 was used for sodium 3-fluorobenzenesulfinate. The reaction gave 66.5 mg of 3-fluorobenzenesulfonamide (4k) in 76% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.69-7.60 (m, 3H), 7.52 (s, 2H), 7.49-7.45 (m, 1H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 161.7 (d, J = 246.3 Hz), 134.7 (d, J = 6.3 Hz), 131.5 (d, J = 7.5 Hz), 121.9 (d, J = 3.8 Hz), 119.0 (d, J = 20.0 Hz), 112.8 (d, J = 23.8 Hz).

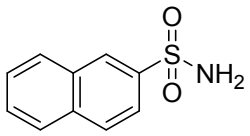


2-fluorobenzenesulfonamide (4l),[26] the procedure B_1 was used for - sodium 2-fluorobenzenesulfinate. The reaction gave 31.5 mg of 2-fluorobenzenesulfonamide (4l) in 36%

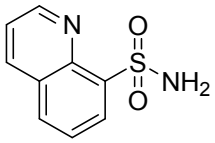
isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 7.82-7.79 (m, 1H), 7.67-7.64 (m, 3H), 7.43-7.40 (m, 1H), 7.38-7.35 (m, 1H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 158.2 (d, J = 251.3 Hz), 134.7 (d, J = 8.8 Hz), 131.7 (d, J = 8.8 Hz), 128.5, 124.7 (d, J = 3.8 Hz), 117.1 (d, J = 20.0 Hz).



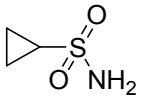
4-nitrobenzenesulfonamide (4e),[27] the procedure B_1 was used for sodium 4-nitrobenzenesulfinate. The reaction gave 82.8 mg of 4-nitrobenzenesulfonamide (4e) in 82% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 8.41 (d, J = 9.0 Hz, 2H), 8.07(d, J = 9.0 Hz, 2H), 7.74 (s, 2H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 149.5, 149.3, 127.4, 124.6.



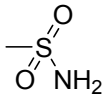
naphthalene-2-sulfonamide (4m),[28] the procedure B_1 was used for sodium naphthalene-2-sulfinate. The reaction gave 65.2 mg of naphthalene-2-sulfonamide (4m) in 63% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 8.44(s, 1H), 8.12 (t, J = 8.5 Hz, 2H), 8.02 (t, J = 8.0 Hz, 1H), 7.90 (dd, J_1 = 8.5 Hz, J_2 = 1.5 Hz, 1H), 7.70-7.64 (m, 2H), 7.47 (s, 2H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 141.3, 134.0, 131.8, 129.2, 129.1, 128.5, 127.9, 127.6, 125.8, 122.2.



quinoline-8-sulfonamide (4n),[29] the procedure B_1 was used for sodium quinoline-8-sulfinate. The reaction gave 72.8 mg of quinoline-8-sulfonamide (4n) in 70% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 9.08 (dd, J_1 = 4.5 Hz, J_2 = 1.5 Hz, 1H), 8.55 (dd, J_1 = 4.5 Hz, J_2 = 1.5 Hz, 1H), 8.32-8.30 (m, 1H), 8.27-8.26 (m, 1H), 7.76-7.70 (m, 2H), 7.27 (s, 2H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 151.3, 142.7, 139.4, 137.2, 133.1, 128.6, 128.5, 125.6, 122.5.



cyclopropanesulfonamide (4o),[30] the procedure B_1 was used for sodium cyclopropanesulfinate. The reaction gave 19.4 mg of cyclopropanesulfonamide (4o) in 32% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 6.78 (s, 2H), 2.51-2.46 (m, 1H), 0.90-0.88 (m, 4H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 32.2, 5.1.



methanesulfonamide (4p),[31] the procedure B_1 was used for sodium methanesulfinate. The

reaction gave 10.9 mg of methanesulfonamide (4p) in 23% isolated yield as a white solid. ^{1}H NMR: (500 MHz, DMSO-d_6, ppm) δ 6.81 (s, 2H), 2.91 (s, 3H). ^{13}C NMR: (125 MHz, DMSO-d_6, ppm) δ 43.2.

General procedure for preparation of 4-tosylmorpholine (3qb) from *N*-iodomorpholine hydroiodide and sodium *p*-toluenesulfinate :

***N*-iodomorpholine hydroiodide (1q')** was prepared according to the previous report[32] by addition of morpholine (2 mmol) to a solution of iodine (2 mmol) in methanol. The orange precipitate was filtered, washed, dried in vacuo and used without further purification or characterization.

To a round-bottom flask was added the corresponding sodium *p*-toluenesulfinate (1 mmol) and *N*-iodomorpholine hydroiodide (0.5 mmol), then EtOH (2 mL) was added. The resulting solution was stirred at room temperature for 3 h. Upon completion of the reaction, 10% sodium thiosulfate solution (20 mL) was added, aqueous layers was extracted with ethyl acetate (20 mL) thrice. The combine organic layers were dried over anhydrous Na_2SO_4. The solvents were removed via rotary evaporator and the residue was purified with flash chromatography (silica gel). The reaction gave 102.4 mg of 4-tosylmorpholine (3qb) in 85% isolated yield as a white solid.

General procedure for preparation of 4-tosylmorpholine (3qb) from morpholine and tosyl iodide:

Tosyl iodide (2b') was prepared according to the previous report[33] by adding an equivalent amount of a ethanol solution of iodine (2 mmol) to a aqueous solution of sodium *p*-toluenesulfinate (2 mmol). The yellow tosyl iodide precipitated and was collected and recrystallized from carbon tetrachloride.

To a round-bottom flask was added the corresponding tosyl iodide (0.5 mmol) and morpholine (0.5 mmol), then EtOH (2 mL) was added. The resulting solution was stirred at room temperature for 3 h. Upon completion of the reaction, 10% sodium thiosulfate solution (20 mL) was added, aqueous layers was extracted with ethyl acetate (20 mL) thrice. The combine organic layers were dried over anhydrous Na_2SO_4. The solvents were removed via rotary evaporator and the residue was purified with flash chromatography (silica gel). The reaction gave 64.0 mg of 4-tosylmorpholine (3qb) in 53% isolated yield as a white solid.

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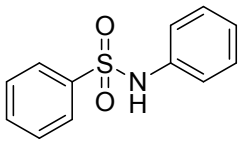
Spectroscopic data



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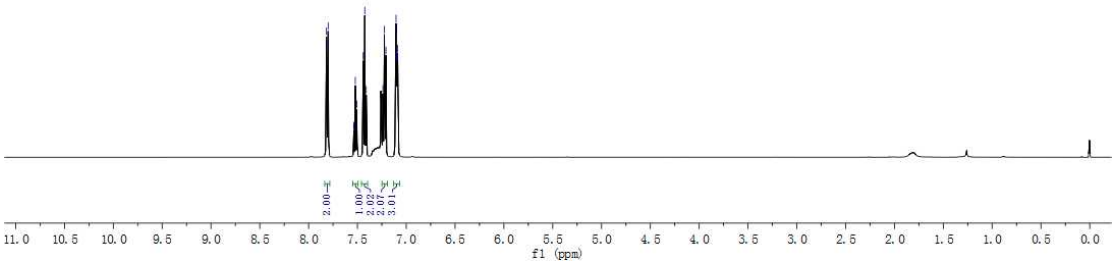
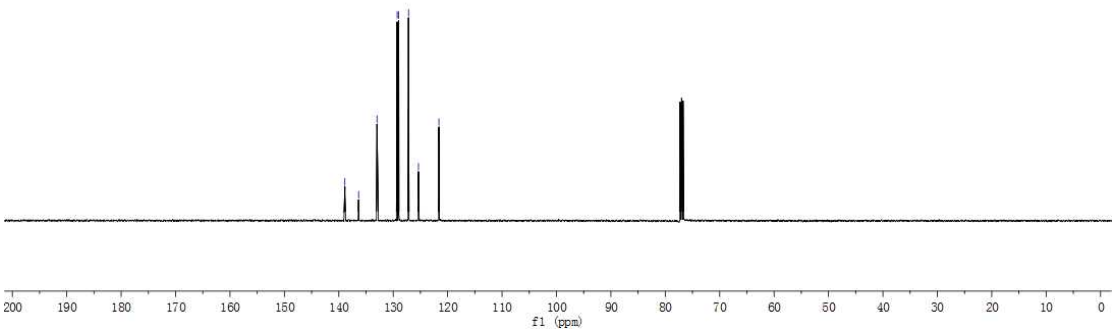
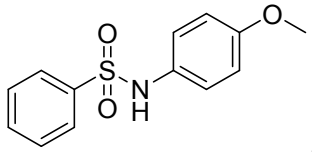
7.090
7.088


2.00
1.00
2.02
2.01
3.01
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 1.5 1.0 0.5 0.0
f1 (ppm)

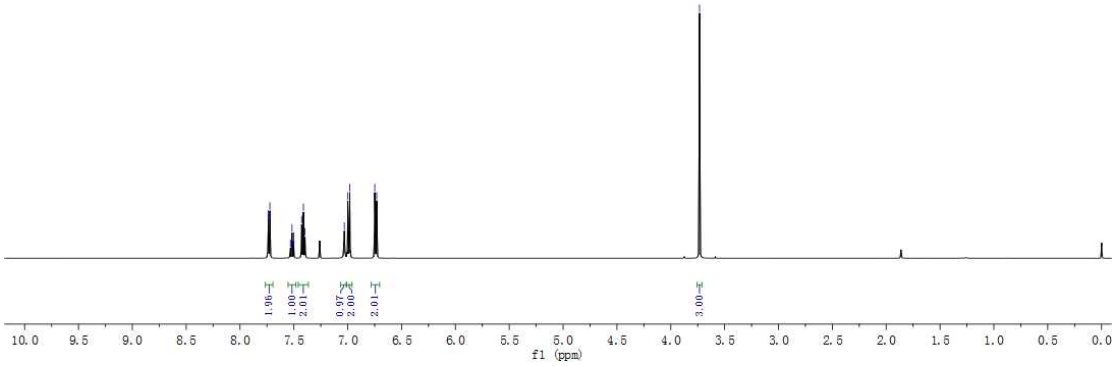

138.890
136.359
132.978
129.269
128.992
127.187
125.362
121.601

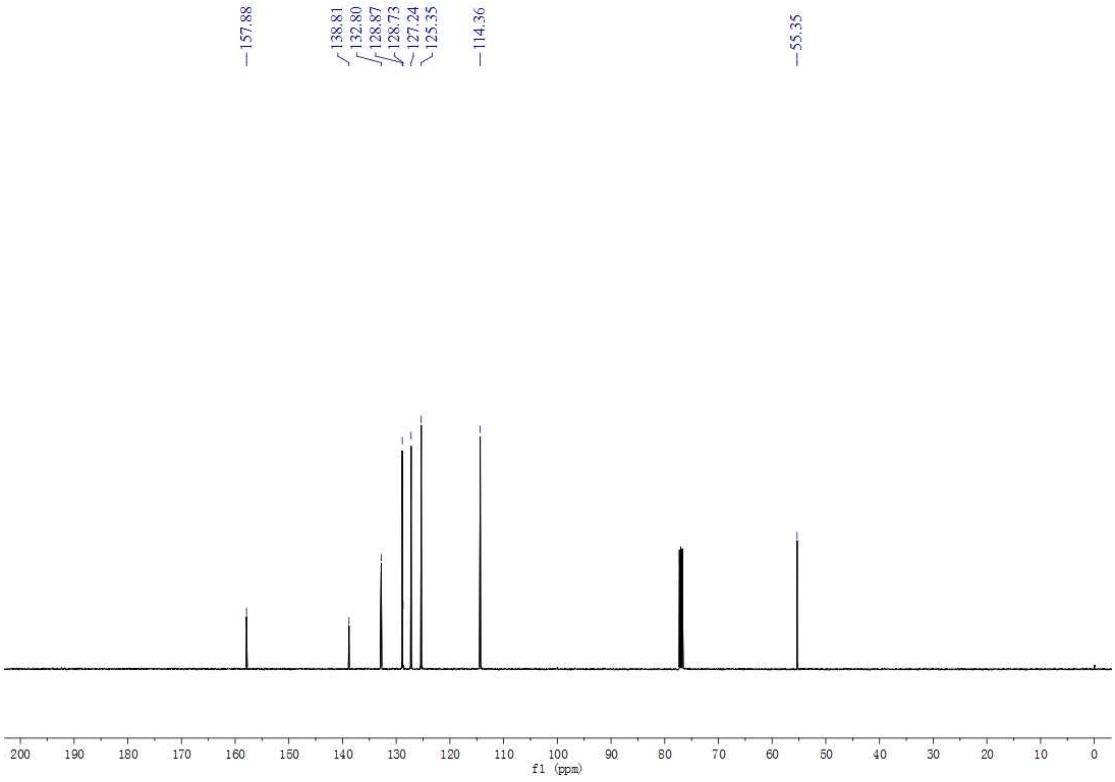

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

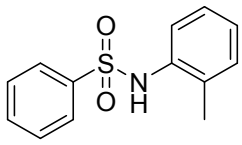


3ba

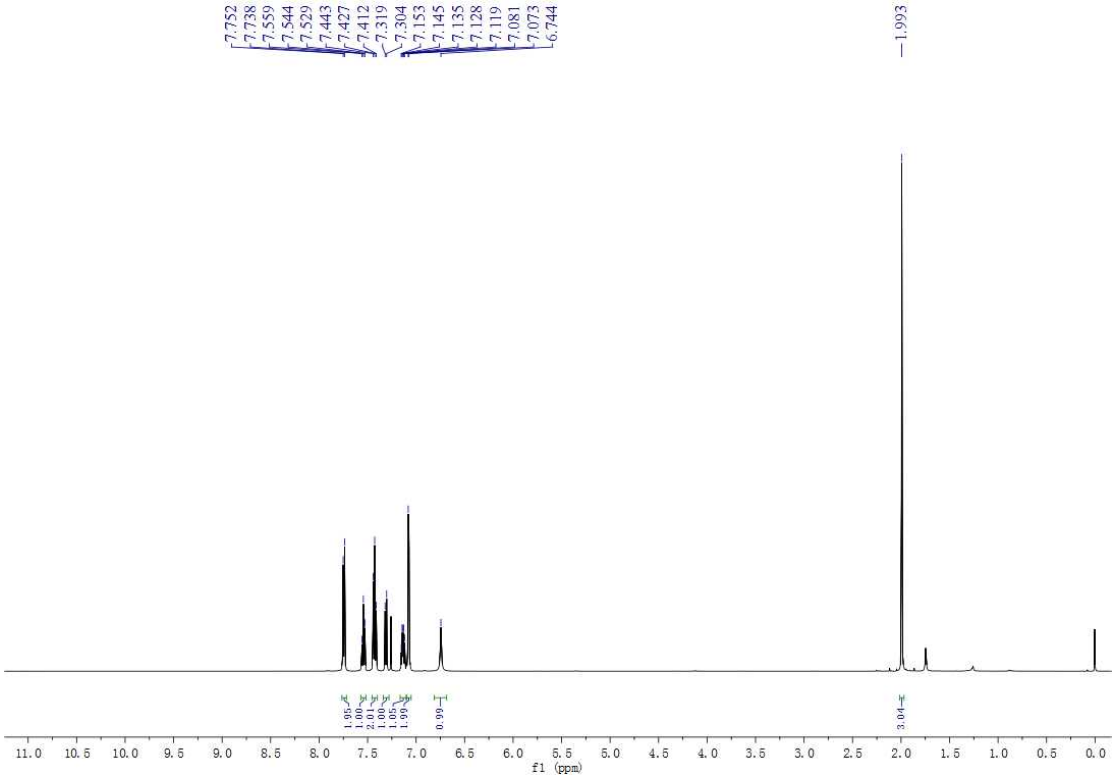

7.739
7.737
7.723
7.720
7.532
7.517
7.514
7.505
7.502
7.500
7.428
7.411
7.396
7.032
6.999
6.981
6.749
6.731
3.733

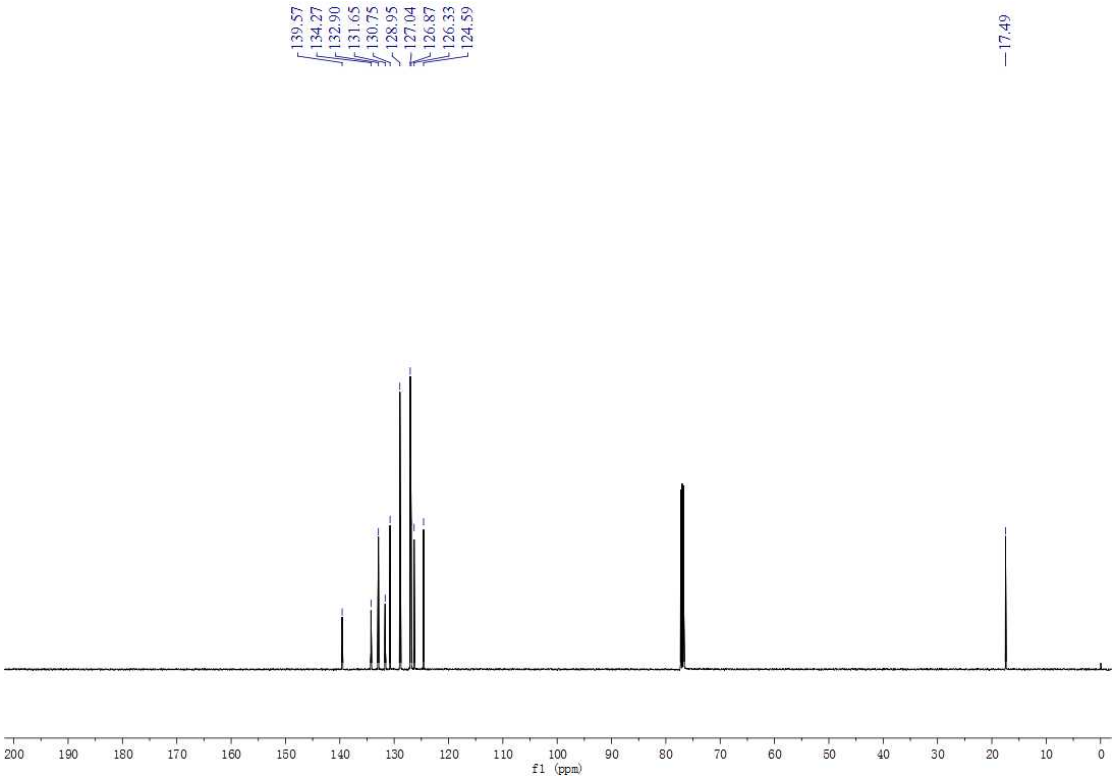

1.98
1.00
2.01
0.97
2.02
2.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

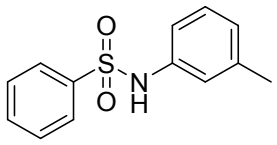

157.88
138.81
132.80
128.87
128.73
127.24
125.35
114.36
55.35
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



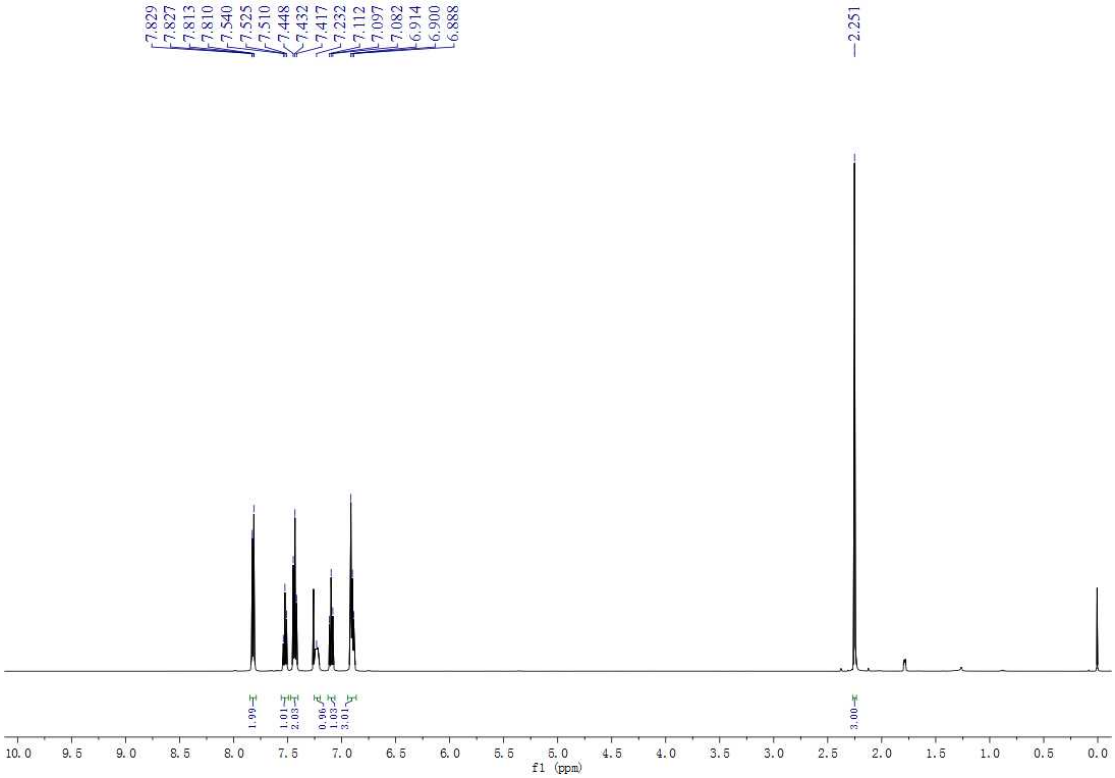
3ca

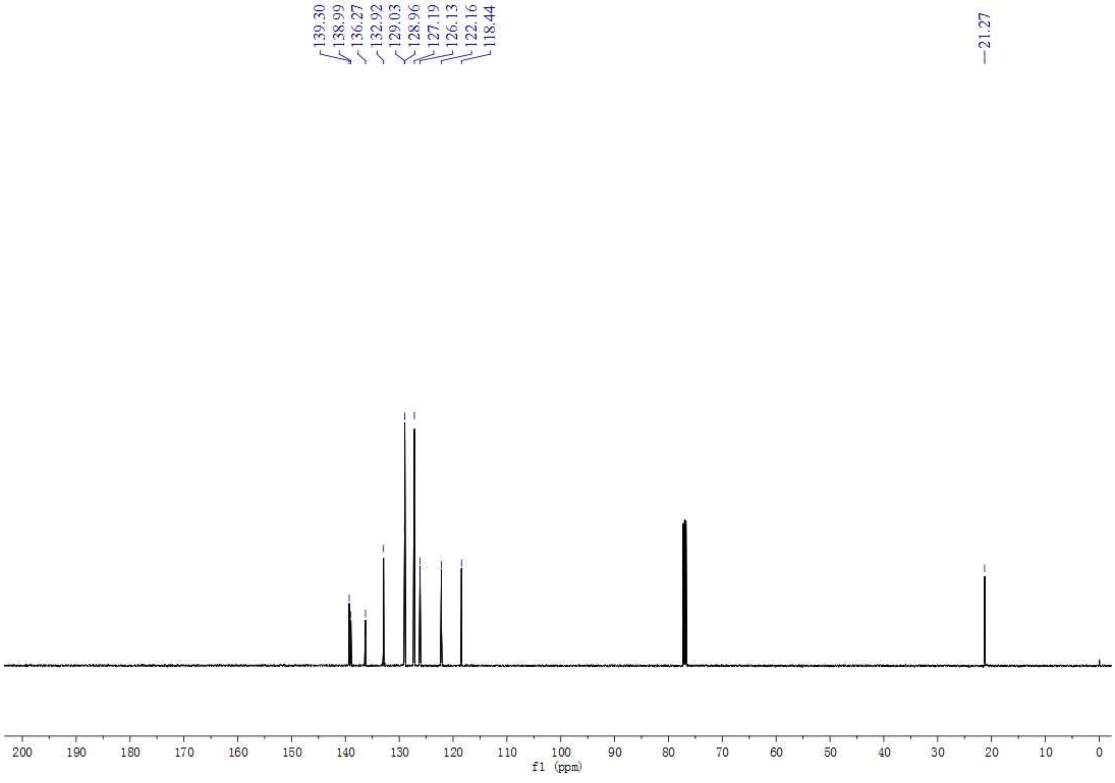

7.752
7.738
7.559
7.544
7.529
7.443
7.427
7.412
7.319
7.304
7.153
7.145
7.135
7.128
7.119
7.081
7.073
6.744
1.993
1.95
1.00
2.01
1.08
1.05
1.99
0.99
3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

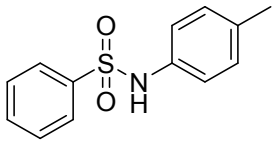

139.57
134.27
132.90
131.65
130.75
128.95
127.04
126.87
126.33
124.59
17.49
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



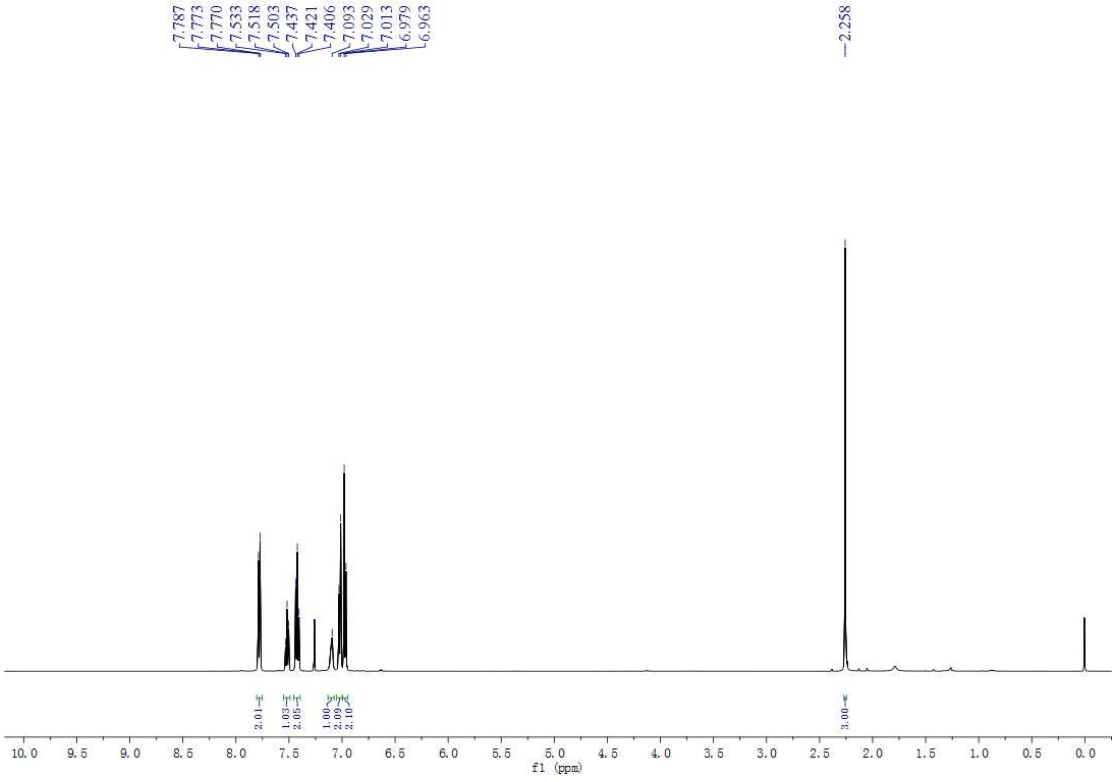
3da

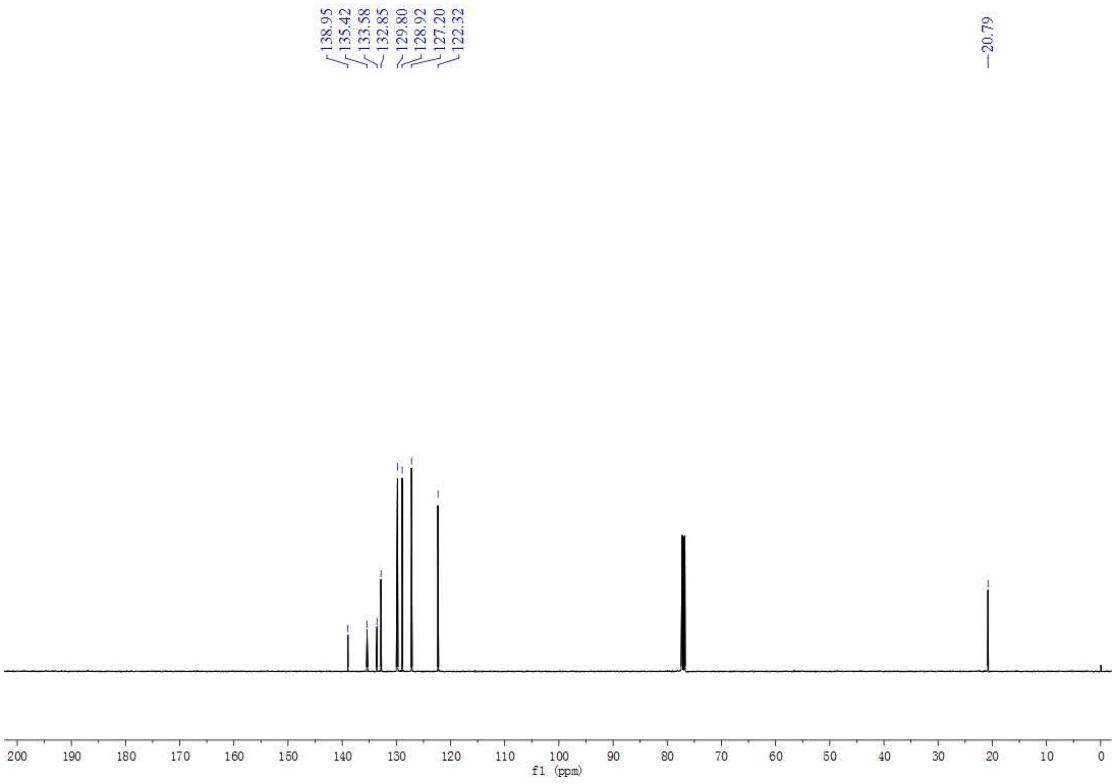

7.829
7.827
7.813
7.810
7.540
7.525
7.510
7.448
7.432
7.417
7.232
7.112
7.097
7.082
6.914
6.900
6.888
2.251
1.99
1.01
2.03
0.96
1.03
3.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

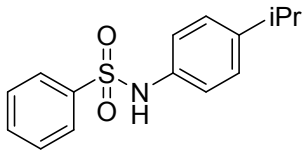

139.30
138.99
136.27
132.92
129.03
128.96
127.19
126.13
122.16
118.44
21.27
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



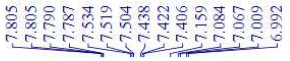
3ea

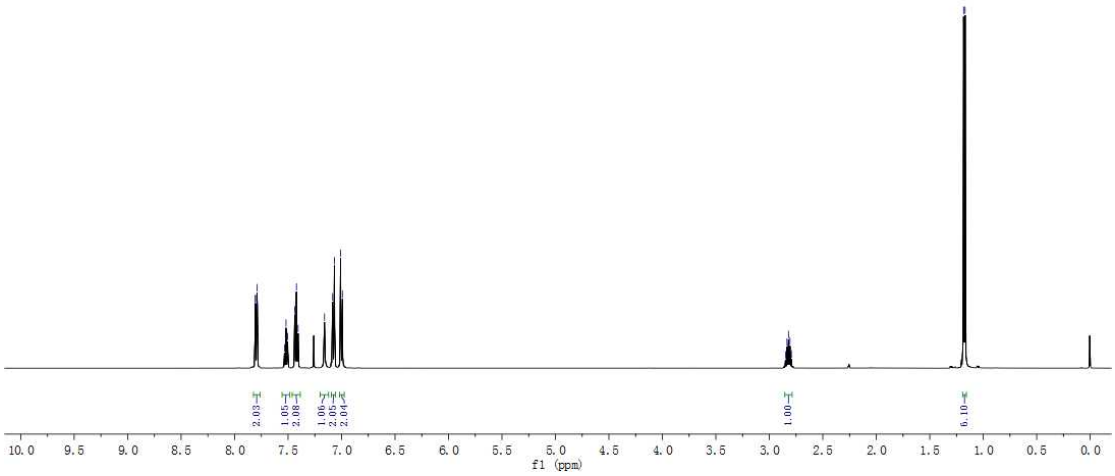

7.787
7.773
7.770
7.533
7.518
7.503
7.437
7.421
7.406
7.093
7.029
7.013
6.979
6.963
2.258
2.01
1.03
2.05
1.08
1.00
2.18
3.00
f1 (ppm)

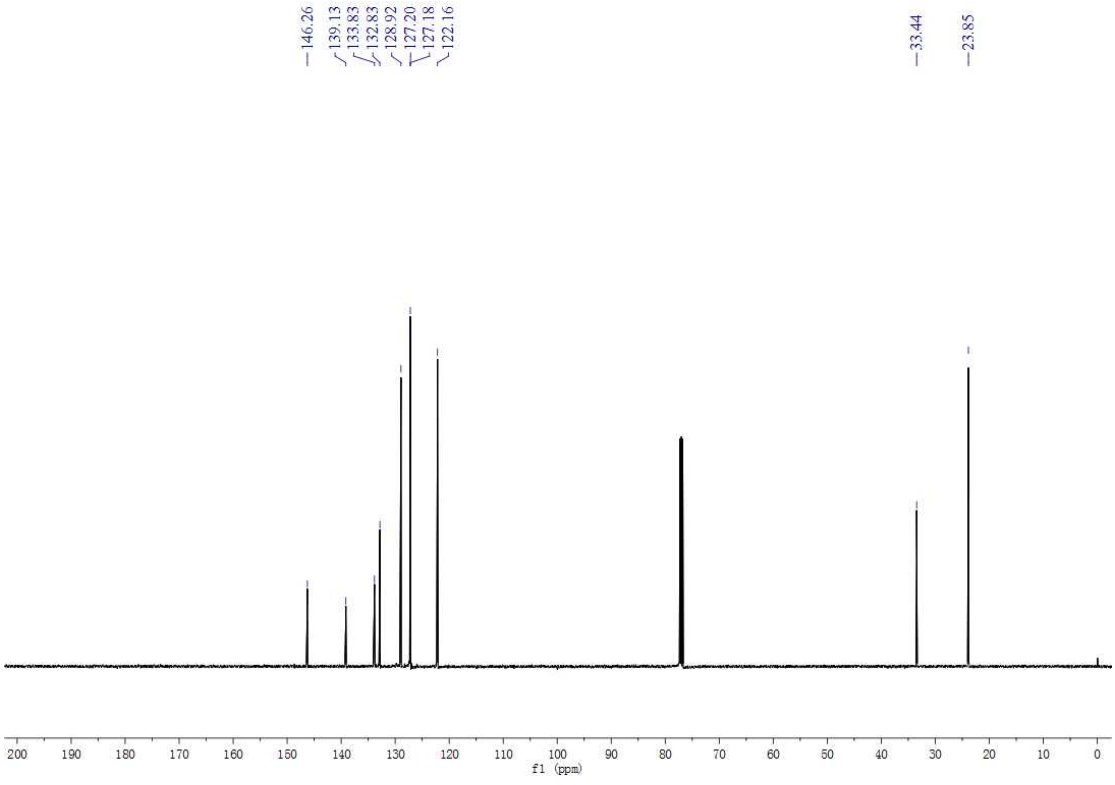

138.95
135.42
133.58
132.85
129.80
128.92
127.20
122.32
20.79
f1 (ppm)

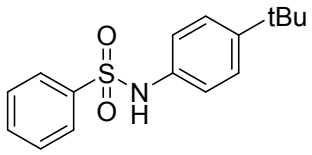

iPr
O
S
O
N
H

3fa

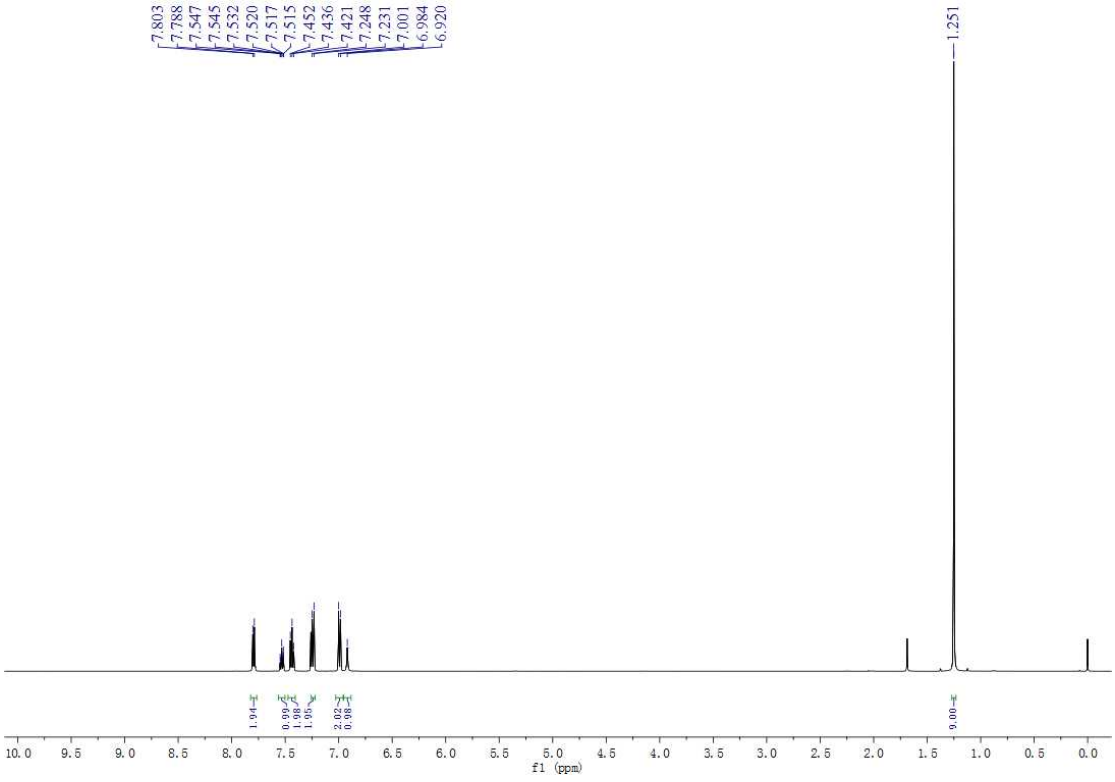

7.805
7.805
7.790
7.787
7.534
7.519
7.504
7.438
7.422
7.406
7.159
7.084
7.067
7.009
6.992
2.849
2.835
2.821
2.807
2.793
1.183
1.170

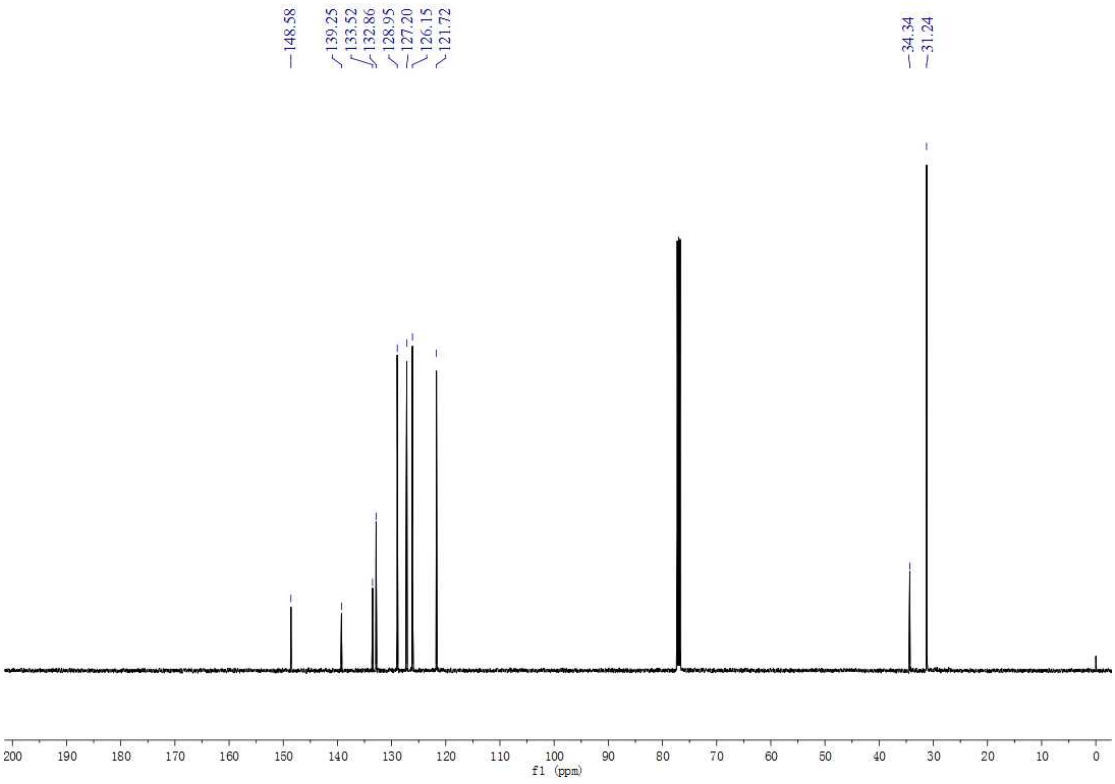

2.03
1.05
2.08
1.06
2.05
2.04
1.00
6.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

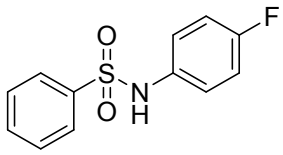

146.26
139.13
133.83
132.83
128.92
127.20
127.18
122.16
33.44
23.85
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


tBu
O
S
N
H
O

3ga

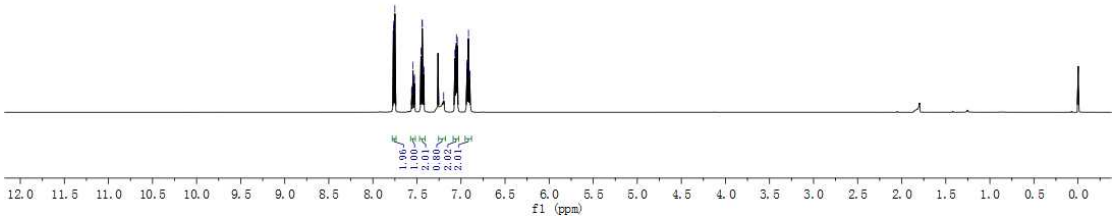

7.803
7.788
7.547
7.545
7.532
7.520
7.517
7.515
7.452
7.436
7.421
7.248
7.231
7.001
6.984
6.920
1.251
1.94
0.99
1.98
1.00
2.02
0.98
9.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

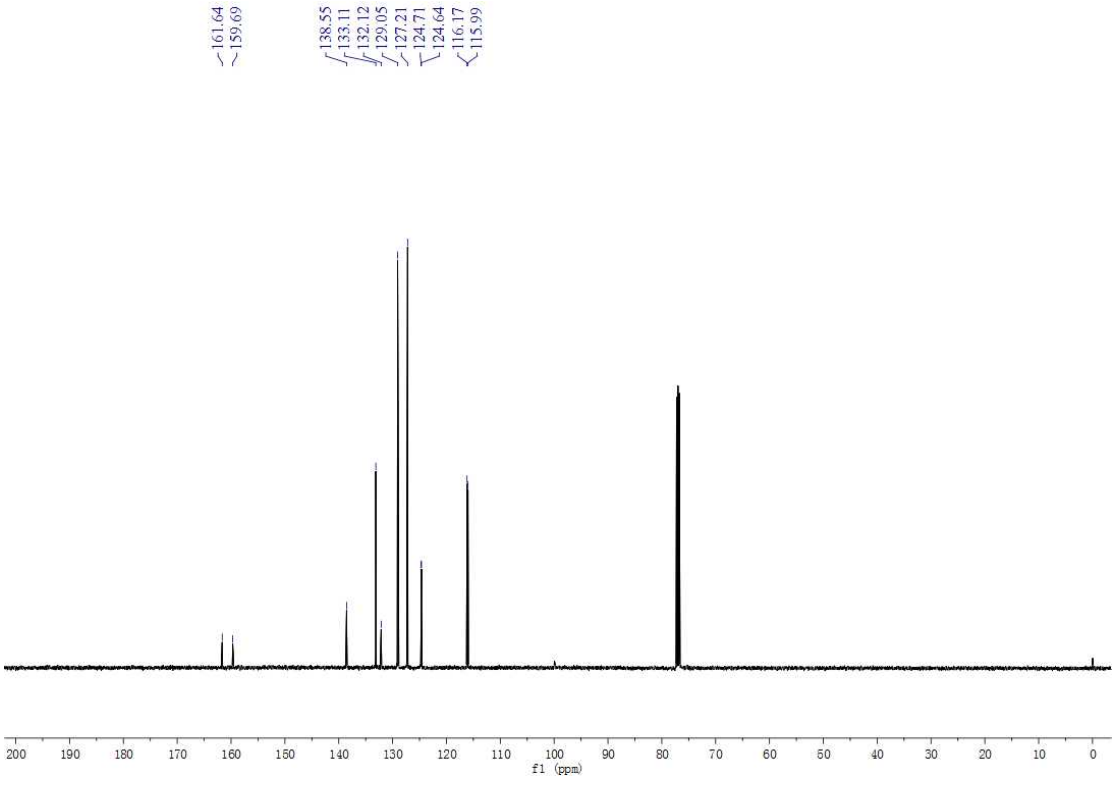

148.58
139.25
133.52
132.86
128.95
127.20
126.15
121.72
34.34
31.24
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

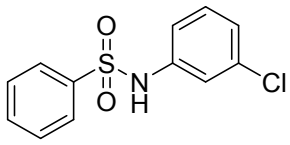

O
S
O
N
H
F

3ha


7.767
7.765
7.750
7.748
7.561
7.558
7.544
7.531
7.528
7.455
7.440
7.439
7.424
7.423
7.197
7.069
7.060
7.055
7.051
7.042
6.933
6.932
6.915
6.900
6.898

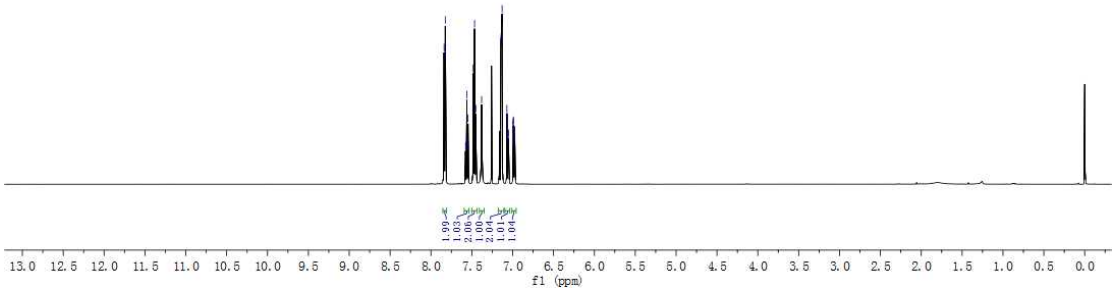

1.96
1.00
2.01
2.00
2.02
2.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

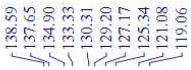

161.64
159.69
138.55
133.11
132.12
129.05
127.21
124.71
124.64
116.17
115.99
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

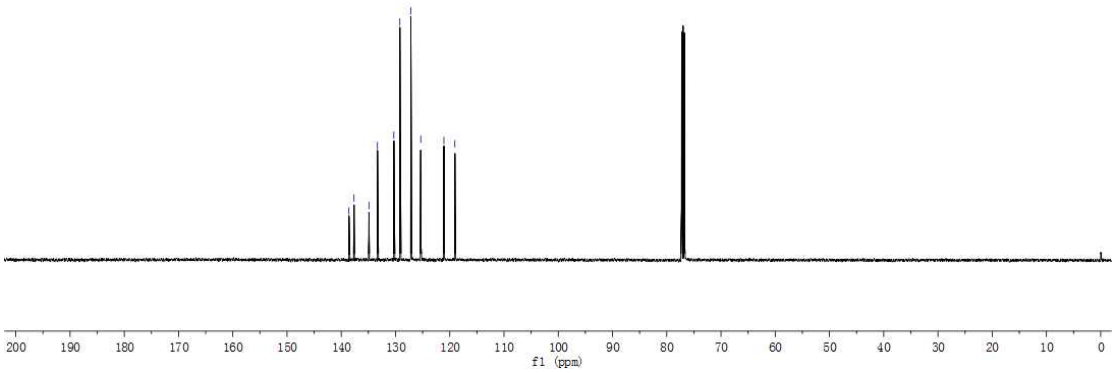

O
S
O
N
H
Cl

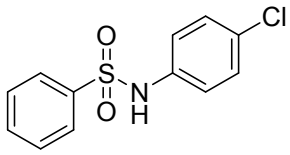
3ia


7.841
7.839
7.825
7.822
7.576
7.561
7.549
7.546
7.544
7.483
7.466
7.451
7.380
7.145
7.134
7.130
7.072
7.071
7.069
7.067
7.056
7.053
7.051
6.995
6.994
6.991
6.979
6.977
6.975
6.973


1.99
1.03
2.08
2.04
2.00
1.01
1.04
13.0 12.5 12.0 11.5 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 1.5 1.0 0.5 0.0
f1 (ppm)


138.59
137.65
134.90
133.33
130.31
129.20
127.17
125.34
121.08
119.06


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Cl
O
S
N
H
O

3ja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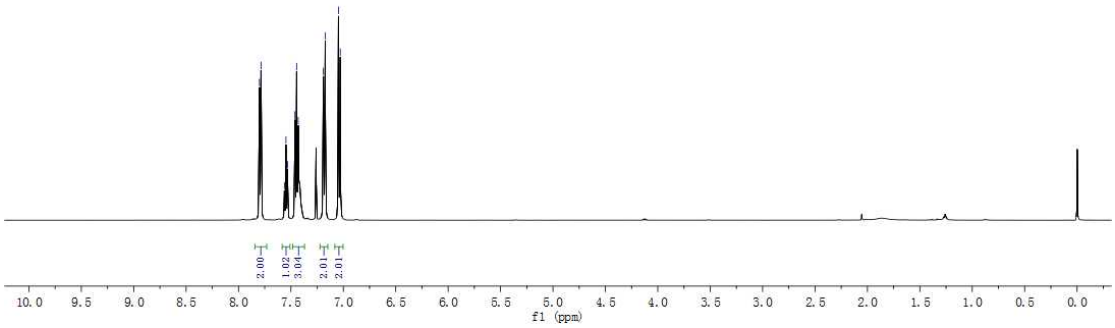
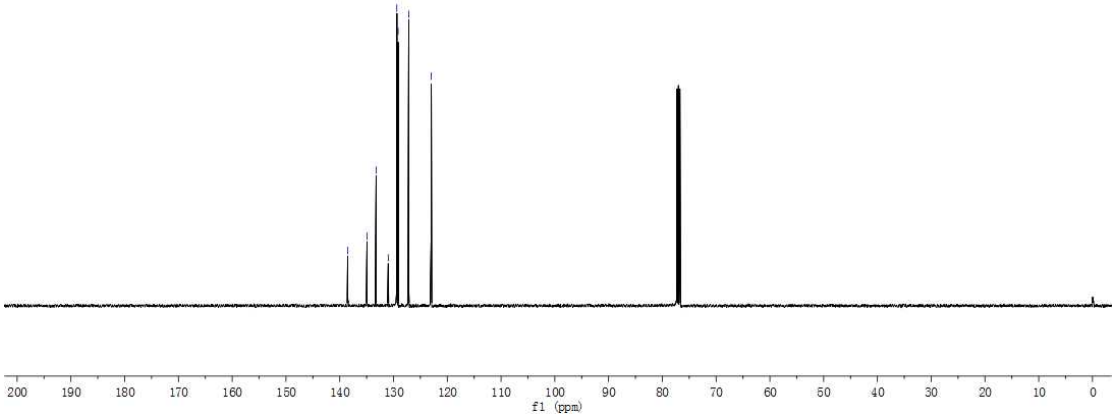
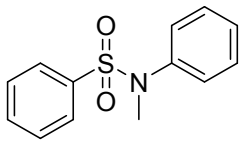
7.786
7.562
7.548
7.533
7.462
7.446
7.430
7.190
7.173
7.047
7.029

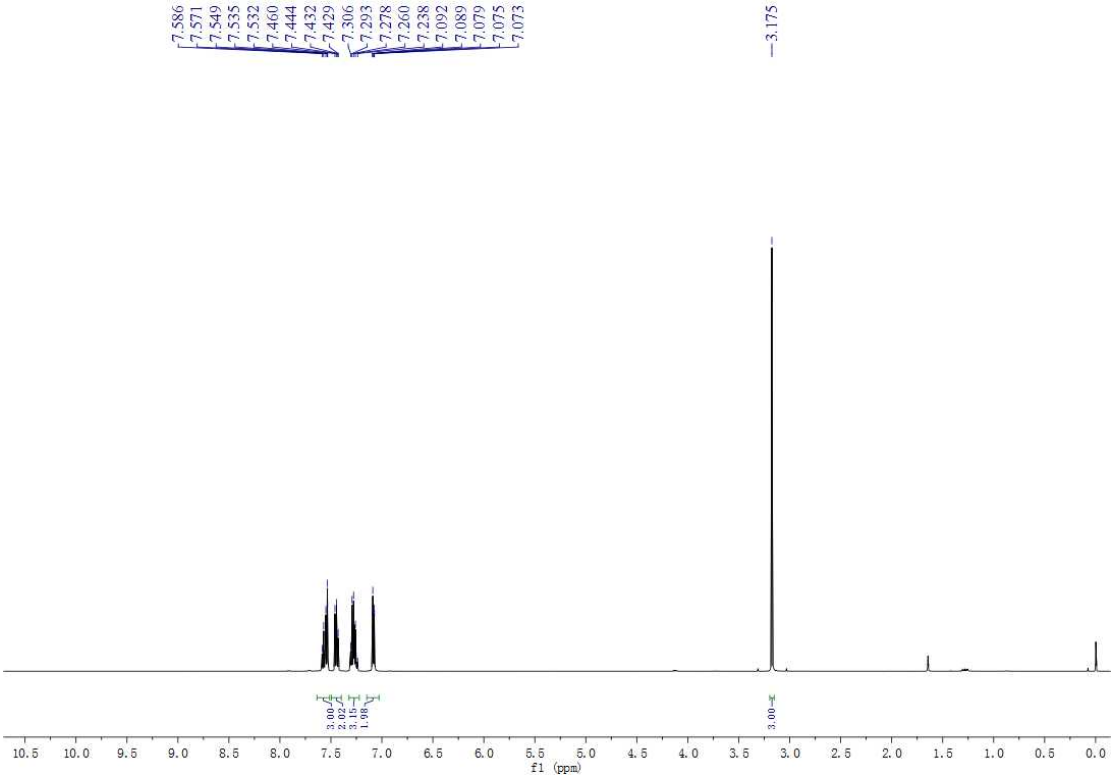

2.00
1.02
3.04
2.01
2.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


138.53
134.93
133.23
130.98
129.38
129.14
127.16
122.97


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



3ka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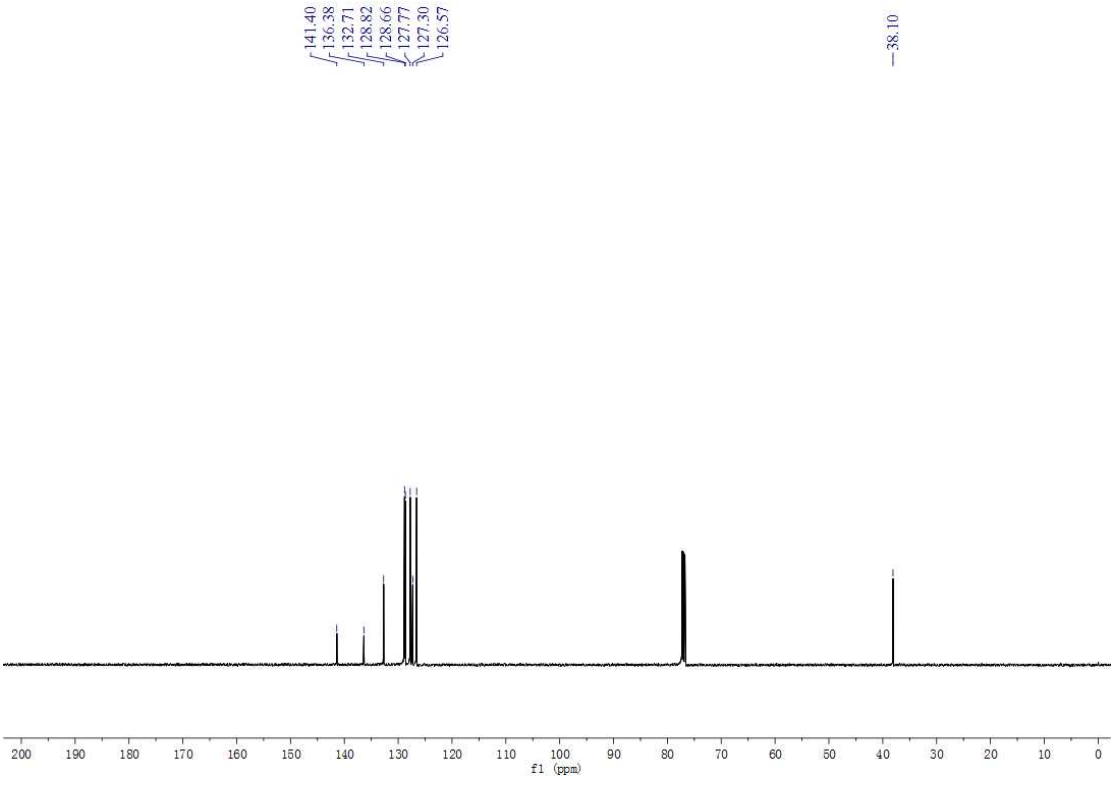
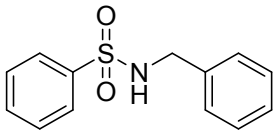
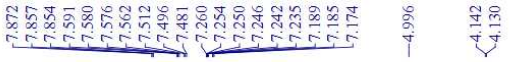
7.532
7.460
7.444
7.432
7.429
7.306
7.293
7.278
7.260
7.238
7.092
7.089
7.079
7.075
7.073
3.175
3.00
2.02
3.18
1.98
3.00
f1 (ppm)


141.40
136.38
132.71
128.82
128.66
127.77
127.30
126.57
38.10
f1 (ppm)



3la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7.512
7.496
7.481
7.260
7.254
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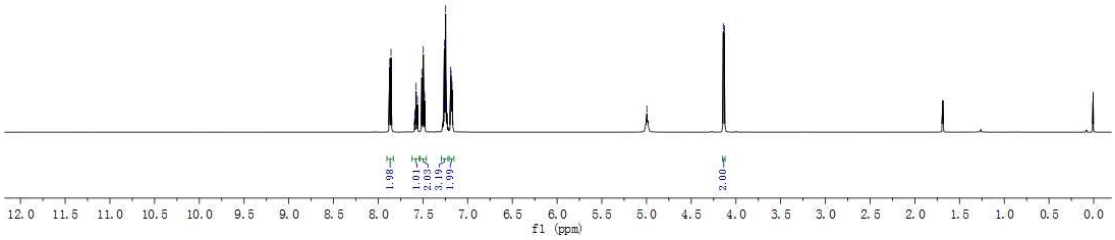
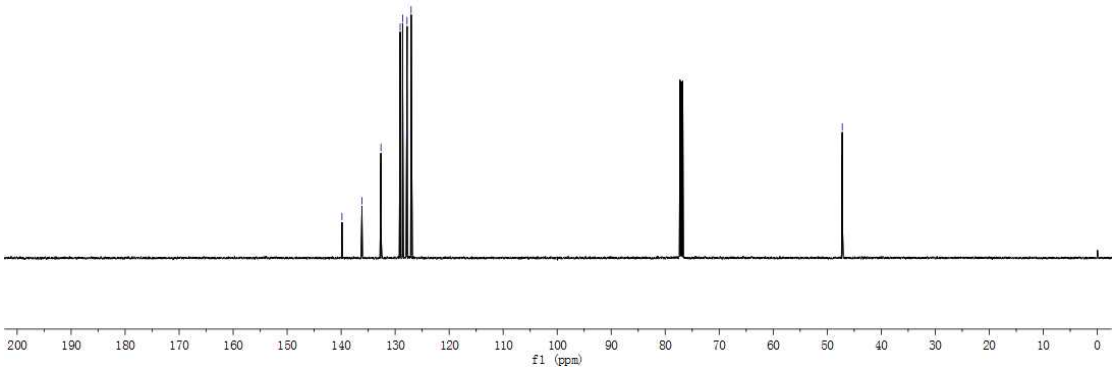
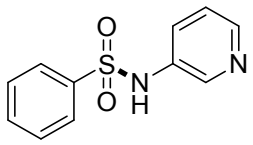
7.189
7.185
7.174
4.996
4.142
4.130

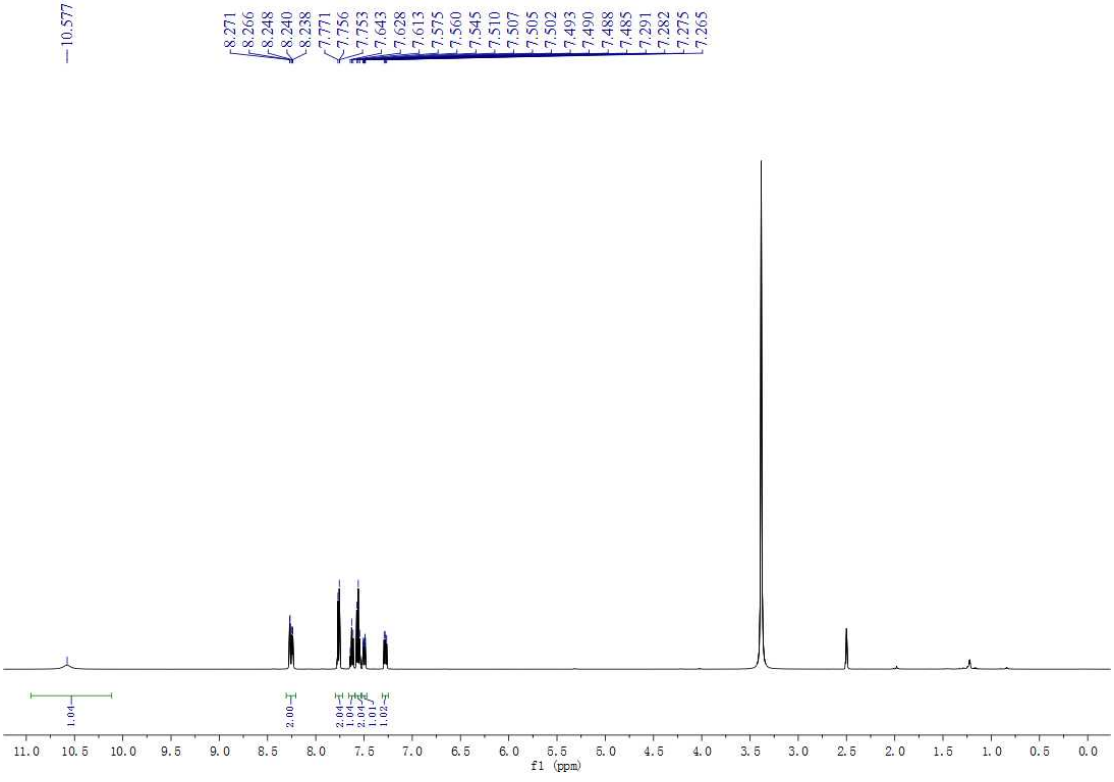

1.98
1.01
2.03
3.19
1.99
2.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

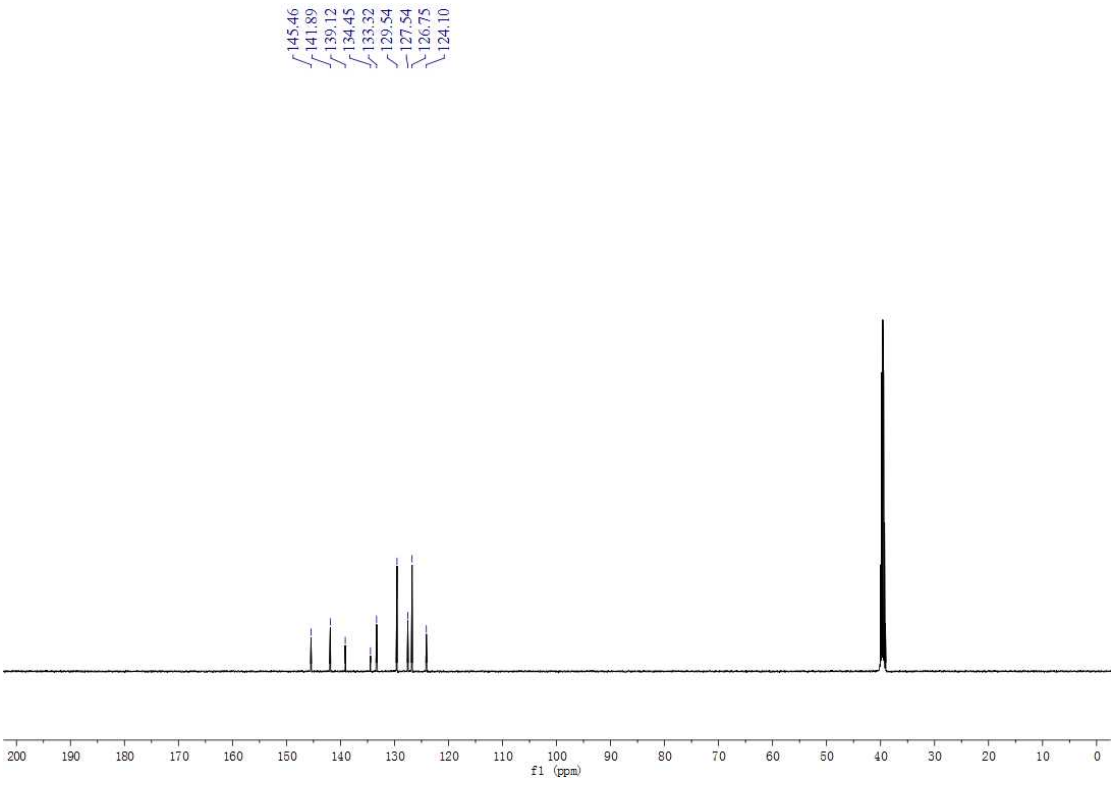

139.85
136.15
132.63
129.07
128.63
127.85
127.80
127.04
47.21

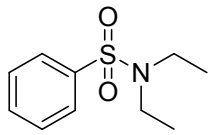

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



3ma


10.577
8.271
8.266
8.248
8.240
8.238
7.771
7.756
7.753
7.643
7.628
7.613
7.575
7.560
7.545
7.510
7.507
7.505
7.502
7.493
7.490
7.488
7.485
7.291
7.282
7.275
7.265
1.04
2.00
2.04
1.04
2.04
1.01
1.02
f1 (ppm)


145.46
141.89
139.12
134.45
133.32
129.54
127.54
126.75
124.10
f1 (ppm)

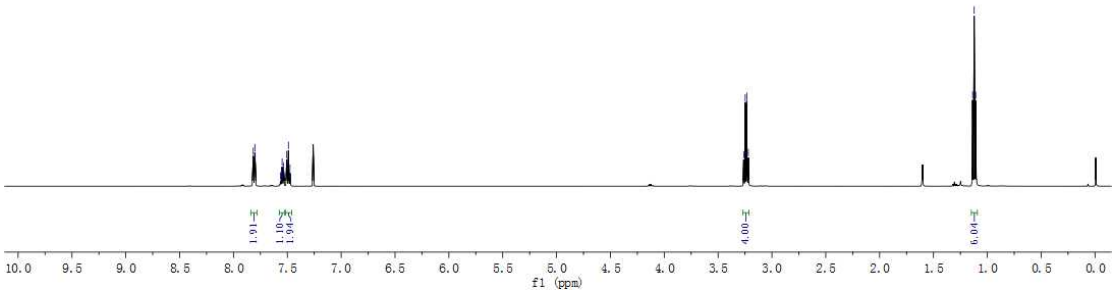


3na

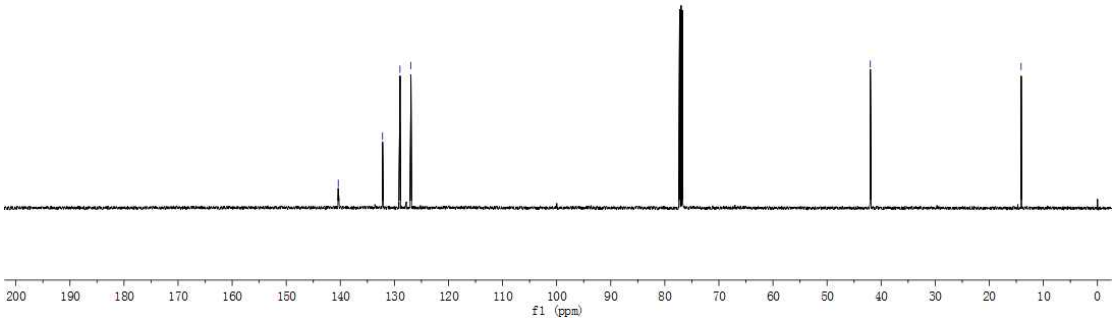

7.816
7.802
7.799
7.563
7.554
7.548
7.544
7.536
7.534
7.505
7.490
7.479
7.475

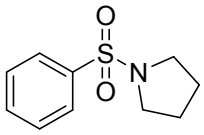

3.262
3.248
3.234
3.220


1.136
1.122
1.108

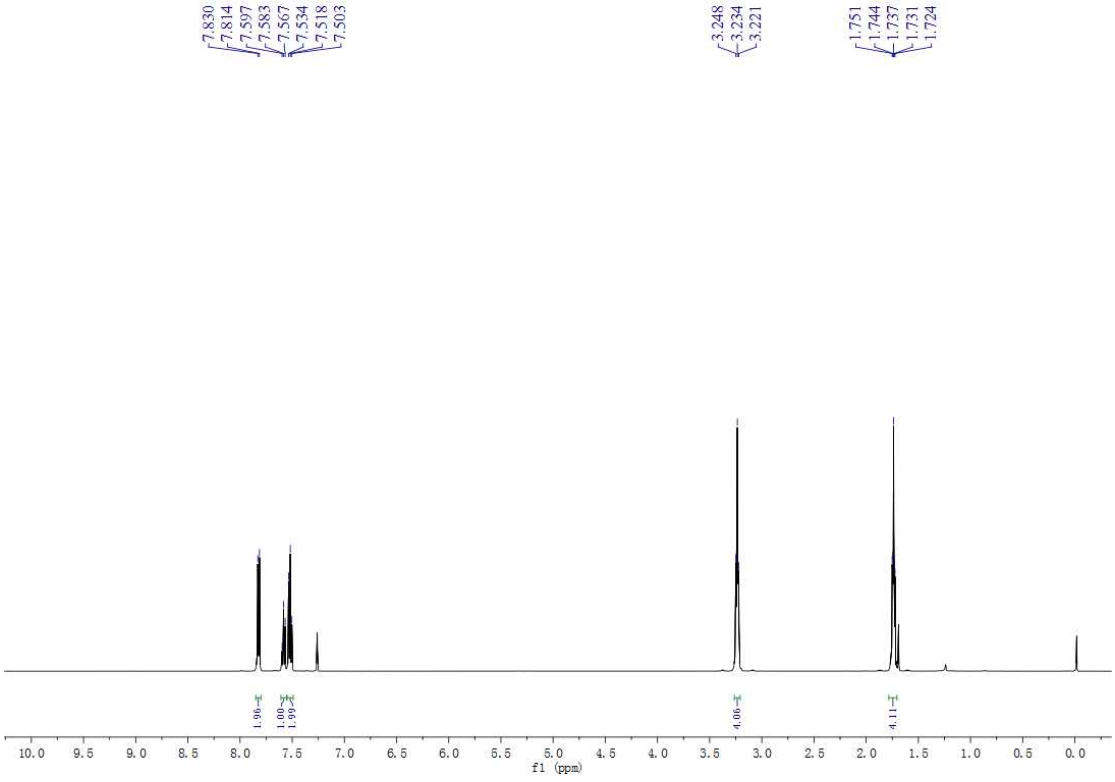

1.91
1.10
1.94
4.00
6.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

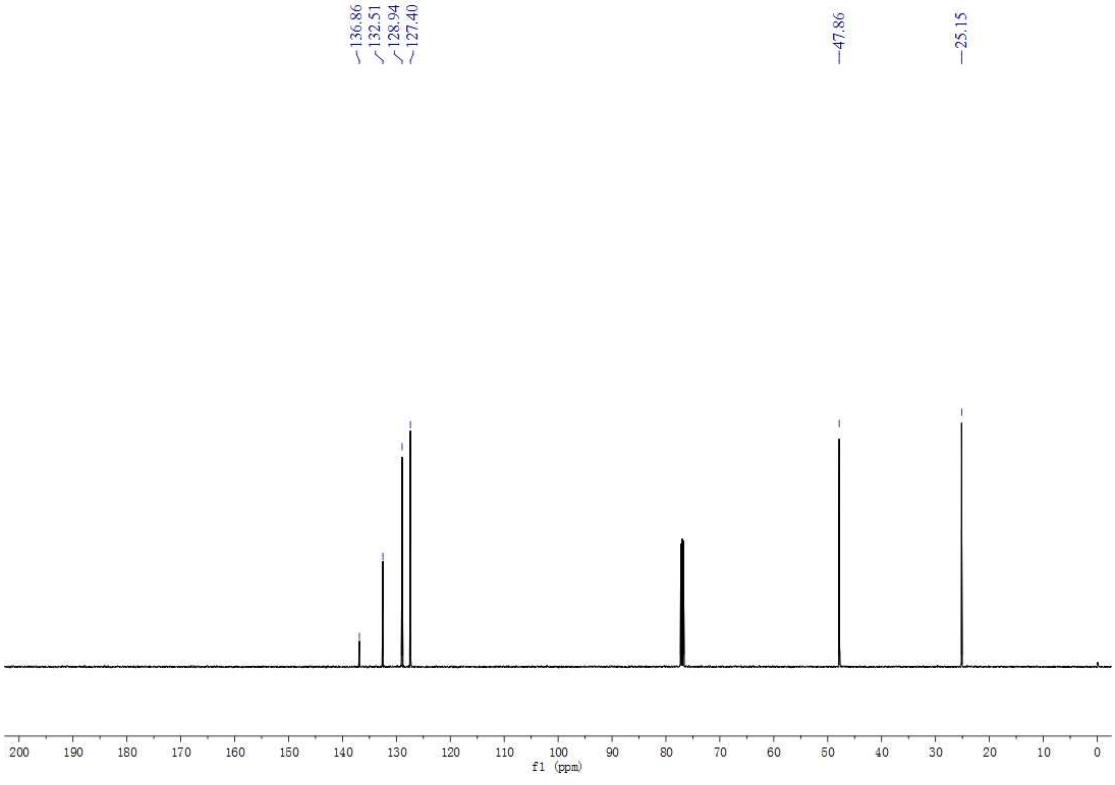

140.39
132.20
128.96
126.94
41.99
14.11

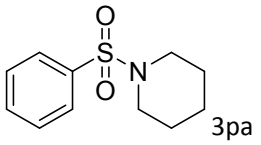

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



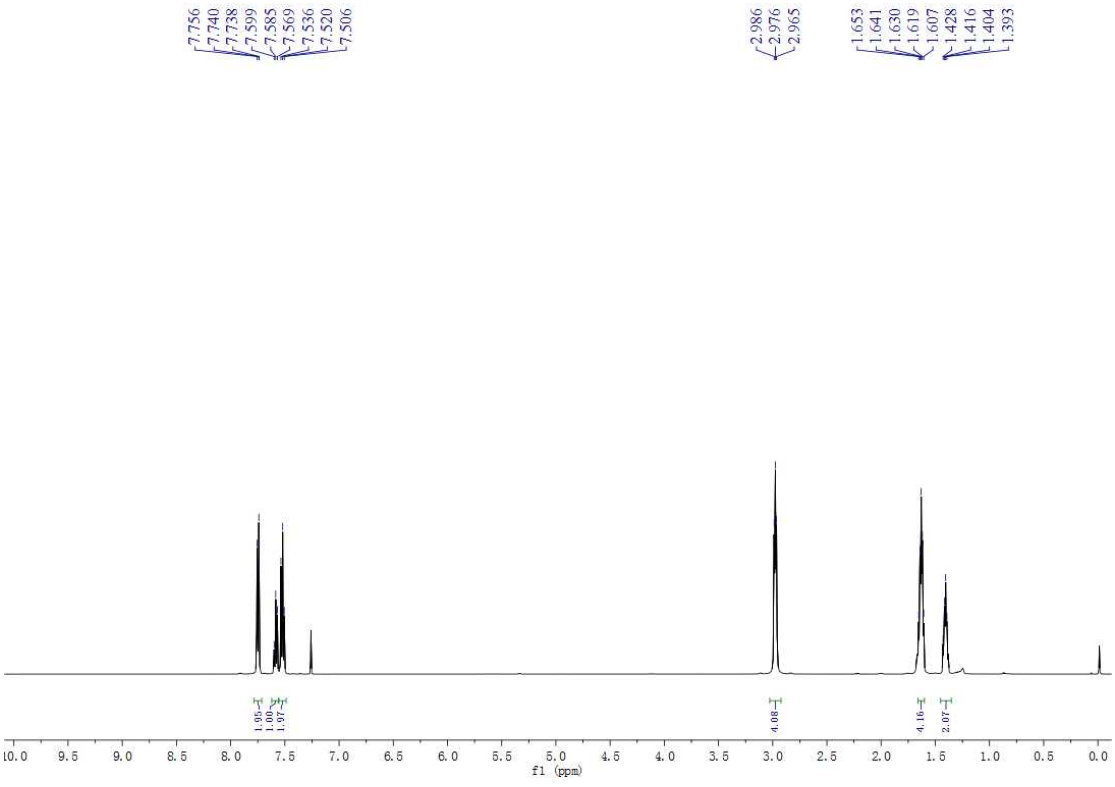
3oa

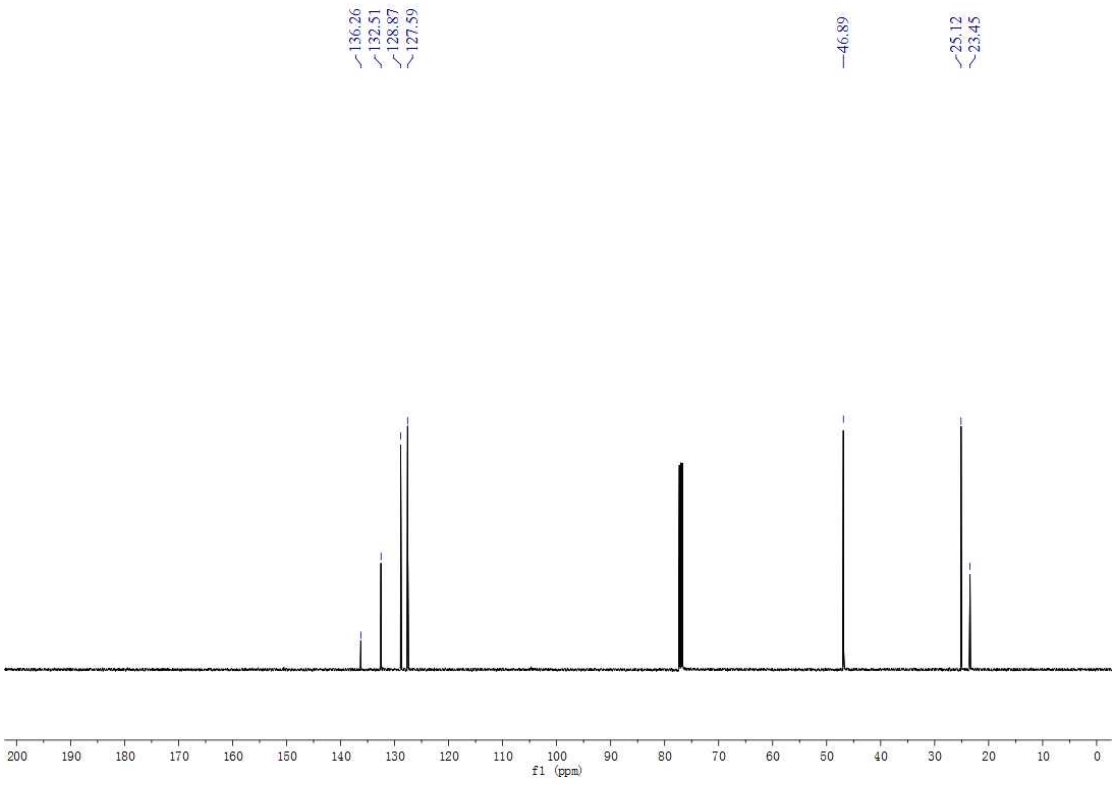

7.830
7.814
7.597
7.583
7.567
7.534
7.518
7.503
3.248
3.234
3.221
1.751
1.744
1.737
1.731
1.724
1.96
1.09
1.99
4.06
4.11
f1 (ppm)

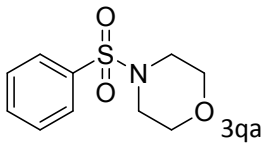

136.86
132.51
128.94
127.40
47.86
25.15
f1 (ppm)

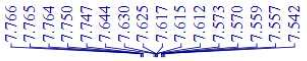


3pa

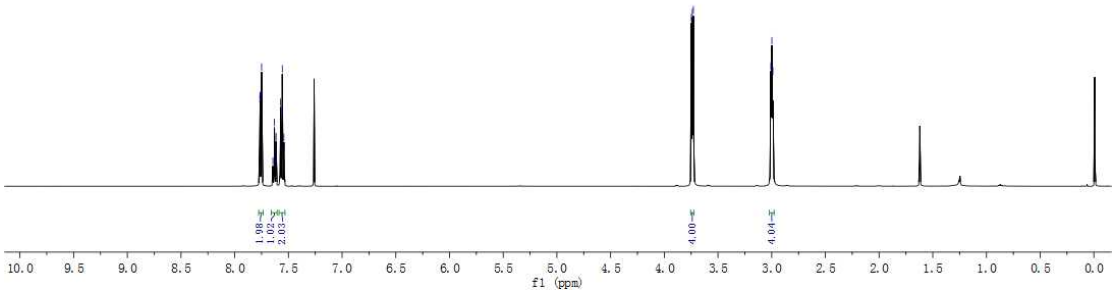

7.756
7.740
7.738
7.599
7.585
7.569
7.536
7.520
7.506
2.986
2.976
2.965
1.653
1.641
1.630
1.619
1.607
1.428
1.416
1.404
1.393
1.96
1.09
1.97
4.08
4.16
2.07
f1 (ppm)


136.26
132.51
128.87
127.59
46.89
25.12
23.45
f1 (ppm)

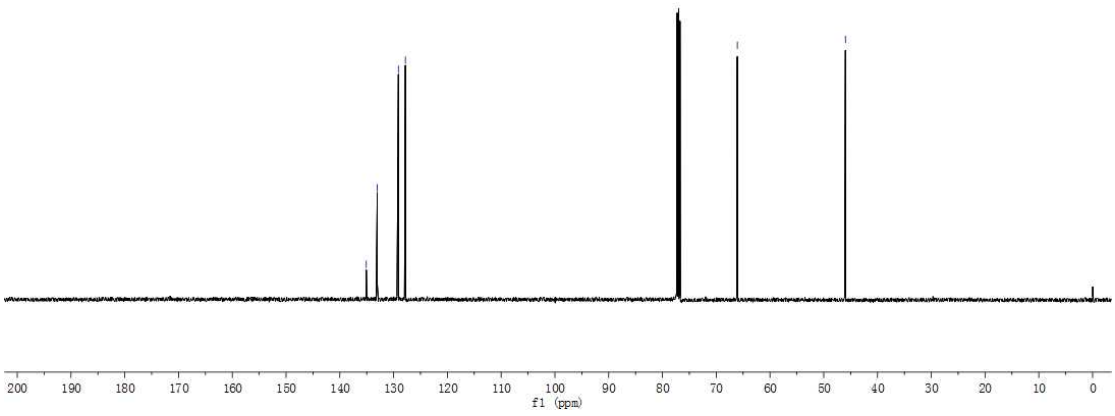

O
S
O
N
O
3qa

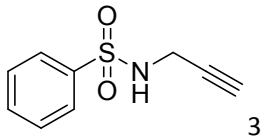

7.766
7.765
7.764
7.750
7.747
7.644
7.630
7.625
7.617
7.615
7.612
7.573
7.570
7.559
7.557
7.542


3.748
3.739
3.729
3.008
2.998
2.989


1.98
1.03
2.03
4.00
4.04
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
f1 (ppm)

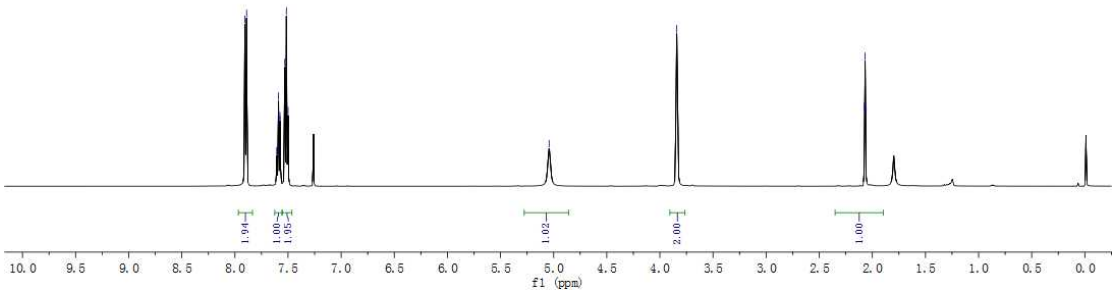

135.082
133.044
129.104
127.804
66.070
45.961


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

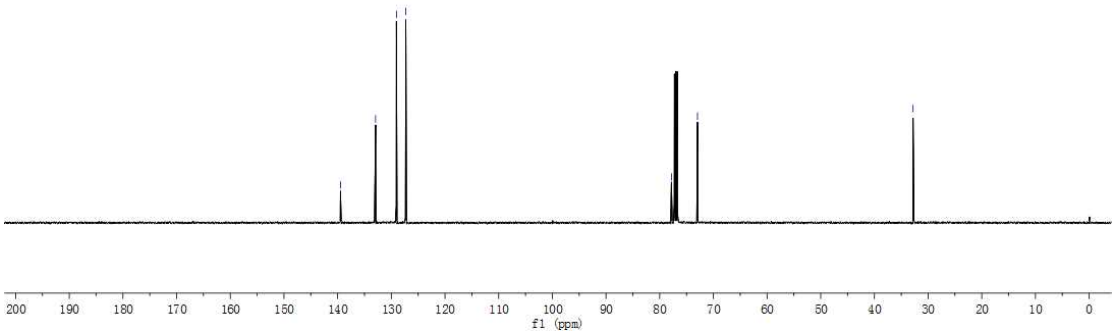


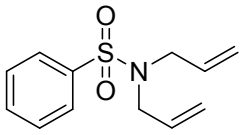
3ra


7.906
7.890
7.605
7.604
7.590
7.576
7.574
7.531
7.516
7.500
5.042
3.841
2.073
2.072
2.068
2.064

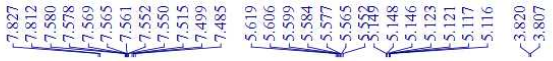

1.94
1.00
1.95
1.02
2.00
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

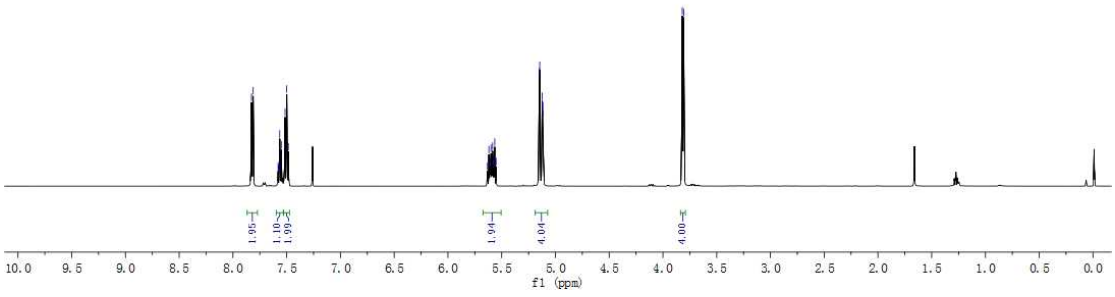

139.47
132.94
129.05
127.30
77.82
72.96
32.80

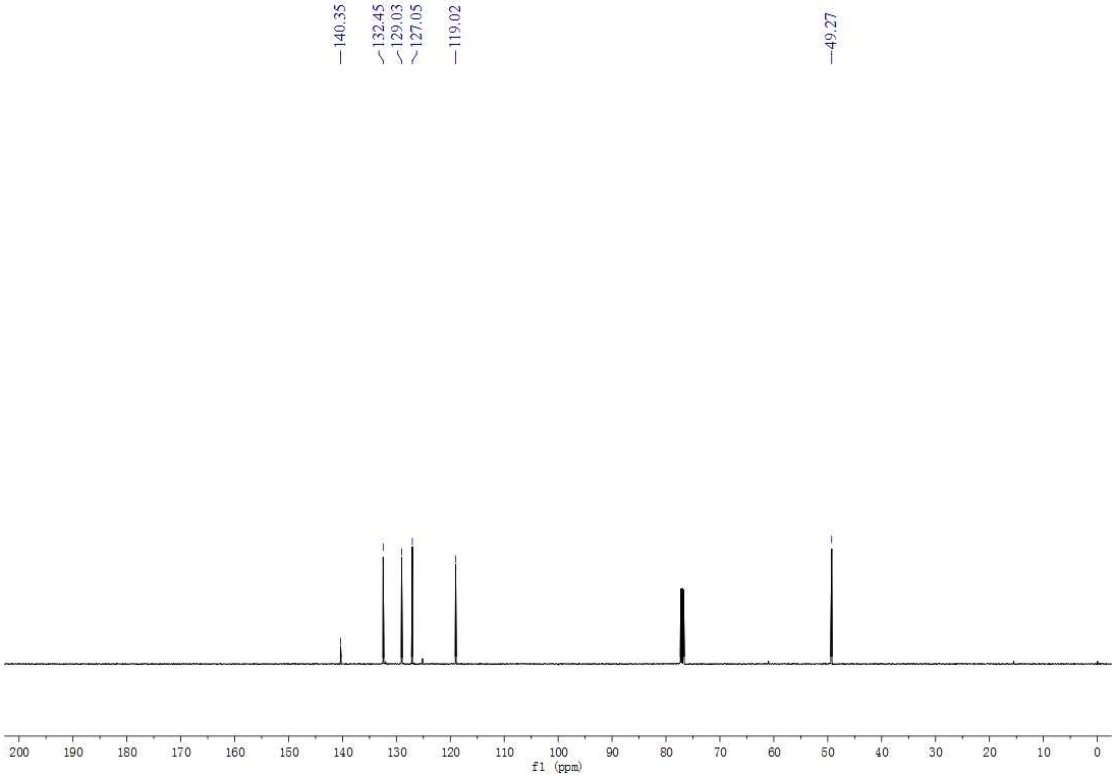

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

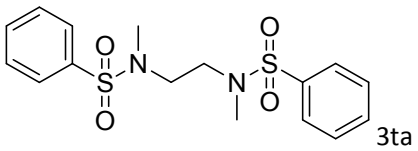


3sa


7.827
7.812
7.580
7.578
7.569
7.565
7.561
7.552
7.550
7.515
7.499
7.485
5.619
5.606
5.599
5.584
5.577
5.565
5.148
5.146
5.123
5.121
5.117
5.116
3.820
3.807


1.95
1.10
1.99
1.94
4.04
4.00
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
f1 (ppm)

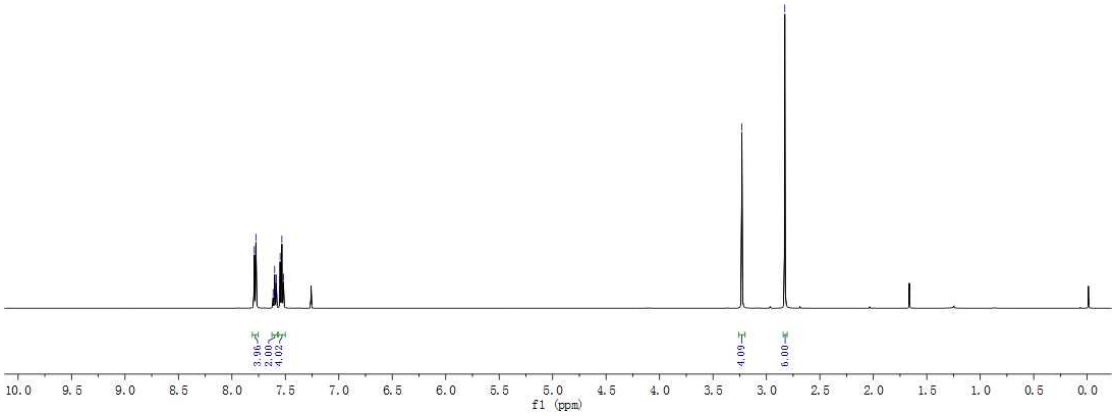

140.35
132.45
129.03
127.05
119.02
49.27
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



3ta

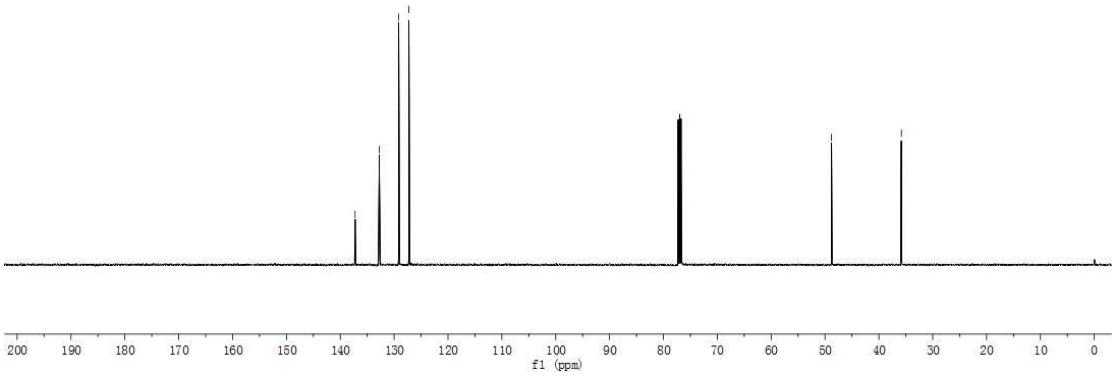

7.790
7.776
7.773
7.615
7.604
7.600
7.596
7.587
7.585
7.549
7.533
7.518

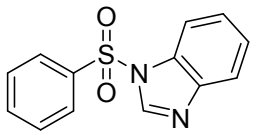

3.230
2.828


3.96
2.00
4.02
4.09
6.00
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
f1 (ppm)


137.25
132.78
129.15
127.26

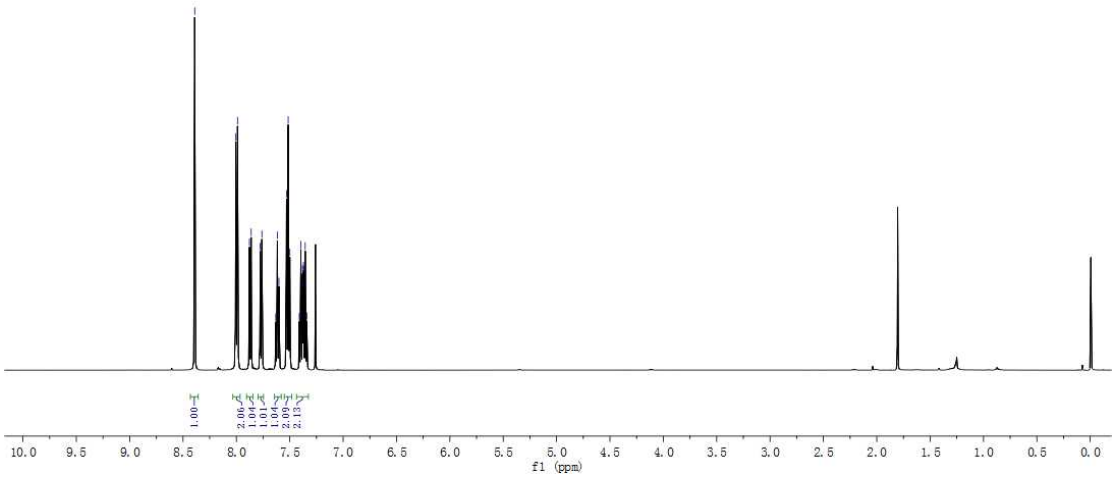

48.79
35.82

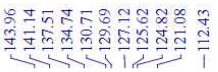

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

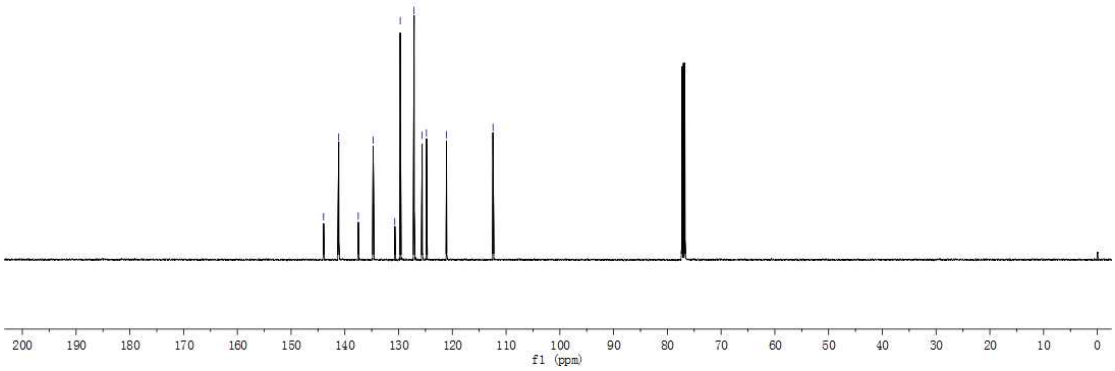


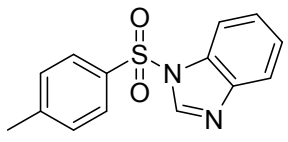
3ua


8.391
8.005
7.989
7.878
7.862
7.776
7.761
7.631
7.617
7.601
7.531
7.515
7.499
7.411
7.396
7.381
7.378
7.371
7.369
7.355
7.341


1.00
2.04
1.04
1.01
1.04
2.09
2.15
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
f1 (ppm)

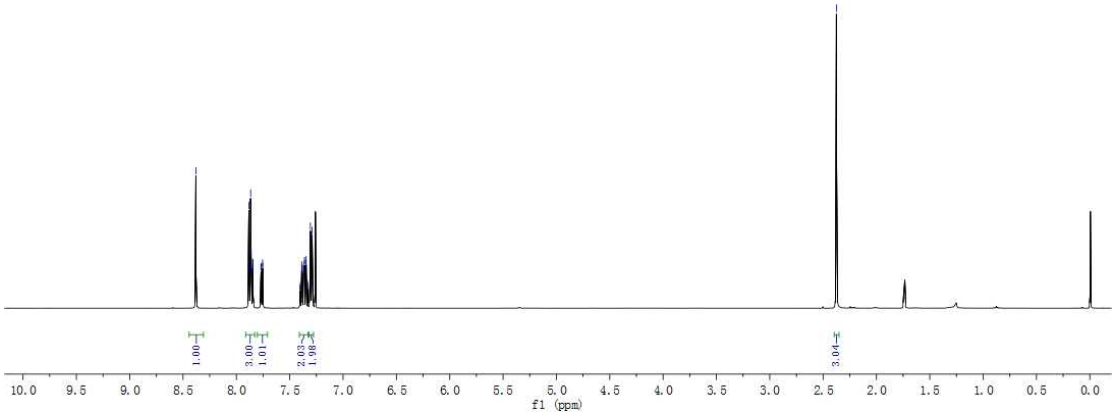

143.96
141.14
137.51
134.74
130.71
129.69
127.12
125.62
124.82
121.08
112.43


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

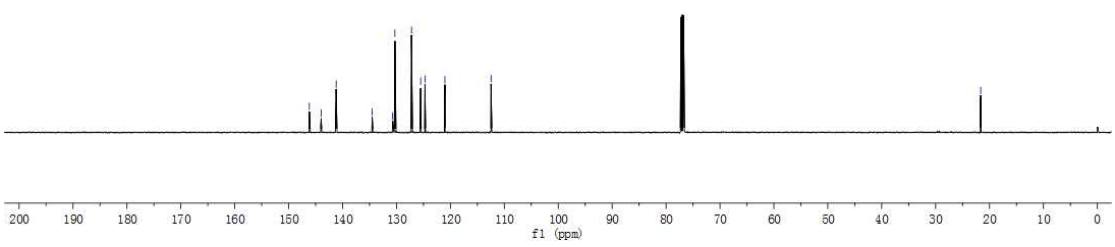


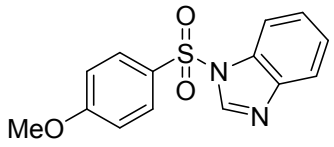
3ub


8.380
7.883
7.880
7.870
7.866
7.863
7.862
7.849
7.847
7.846
7.845
7.770
7.769
7.755
7.753
7.402
7.400
7.388
7.385
7.372
7.369
7.364
7.361
7.348
7.346
7.334
7.331
7.306
7.290
7.289
2.375


1.00
3.00
1.01
2.03
1.98
3.04
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
f1 (ppm)

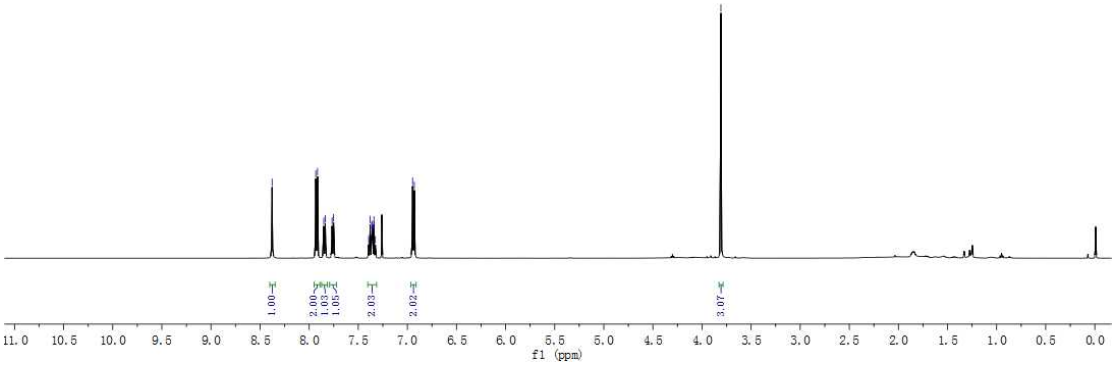

146.17
143.98
141.19
134.51
130.72
130.30
127.21
125.51
124.72
121.03
112.46
21.63


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

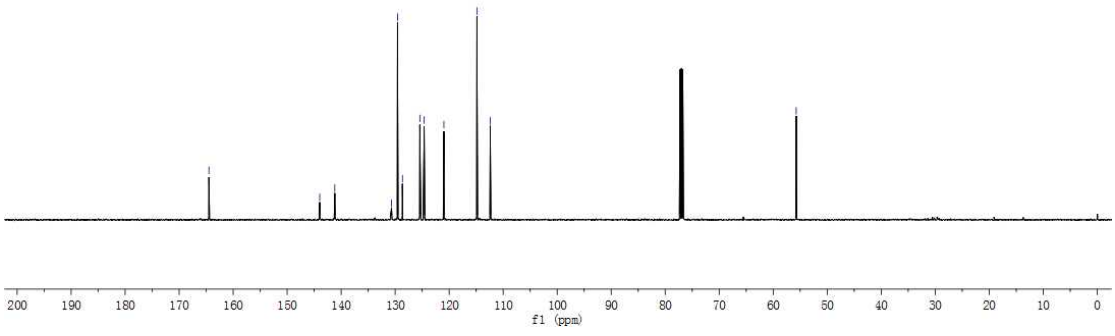

O
S
O
N
N
MeO

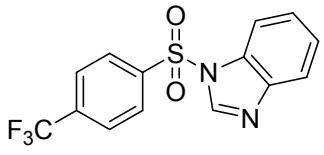
3uc


8.377
7.933
7.915
7.851
7.835
7.767
7.751
7.394
7.379
7.364
7.361
7.356
7.354
7.341
7.339
7.326
6.947
6.929
3.807

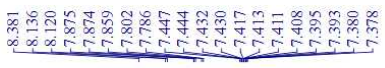

1.00
2.09
1.03
1.03
2.03
2.02
3.07
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

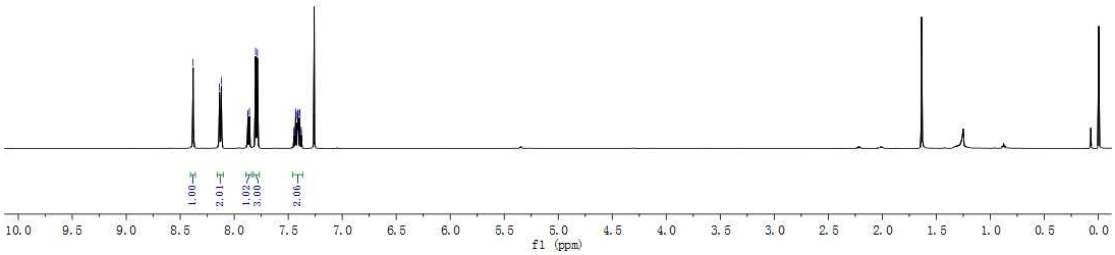

164.48
143.97
141.19
130.68
129.57
128.65
125.43
124.63
120.98
114.86
112.39
55.75


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

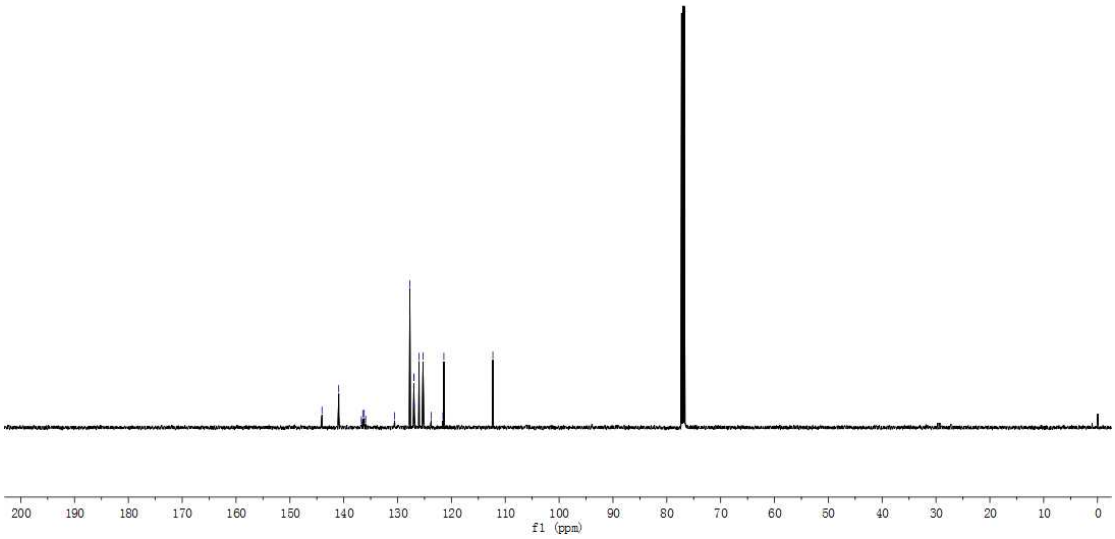

O
S
O
N
N
F3C

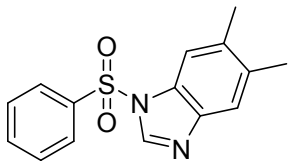
3ud


8.381
8.136
8.120
7.875
7.874
7.859
7.802
7.786
7.447
7.444
7.432
7.430
7.417
7.413
7.411
7.408
7.395
7.393
7.380
7.378

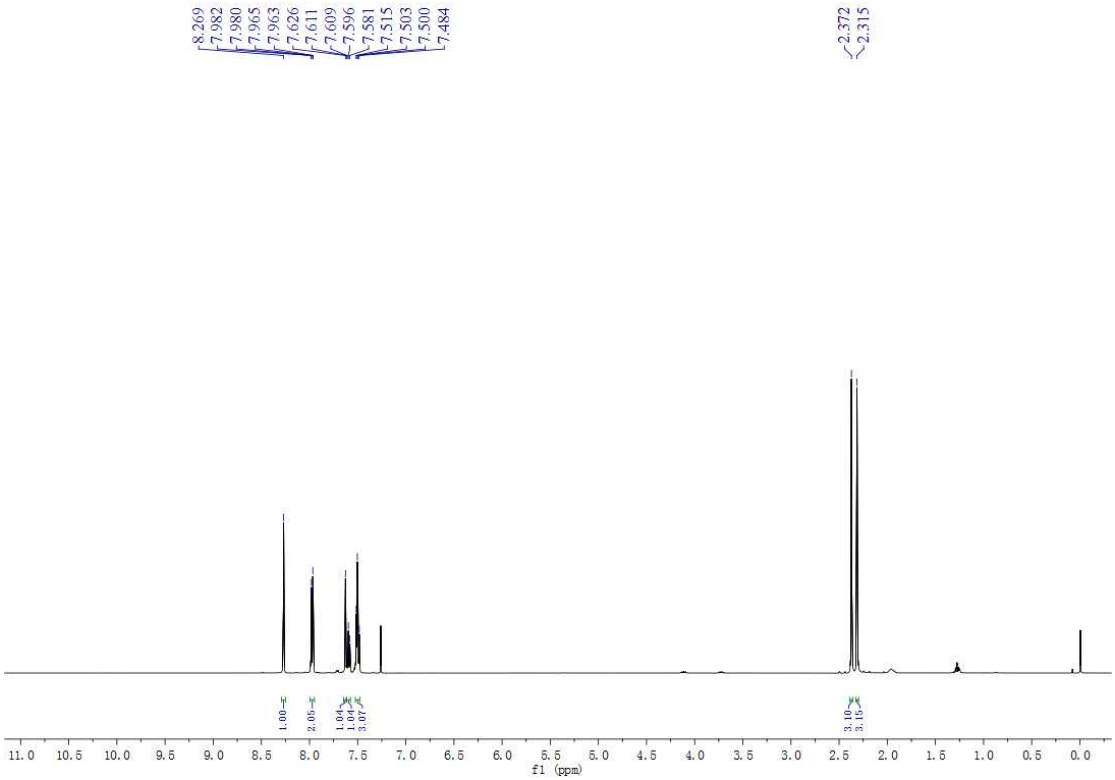

1.00
2.01
1.02
3.00
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

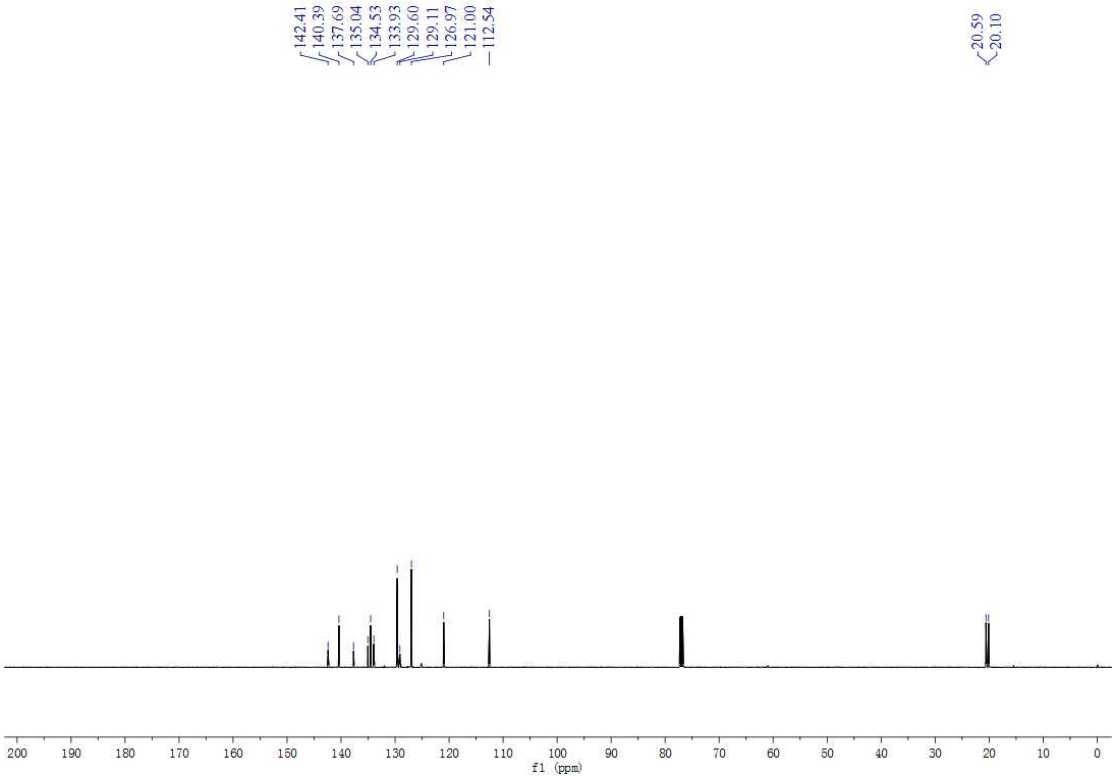

144.04
140.94
136.73
136.47
136.20
135.93
130.55
127.72
127.00
126.97
126.94
126.91
126.02
125.27
123.76
121.59
121.40
112.30

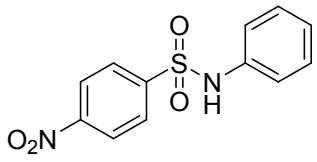

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



3va

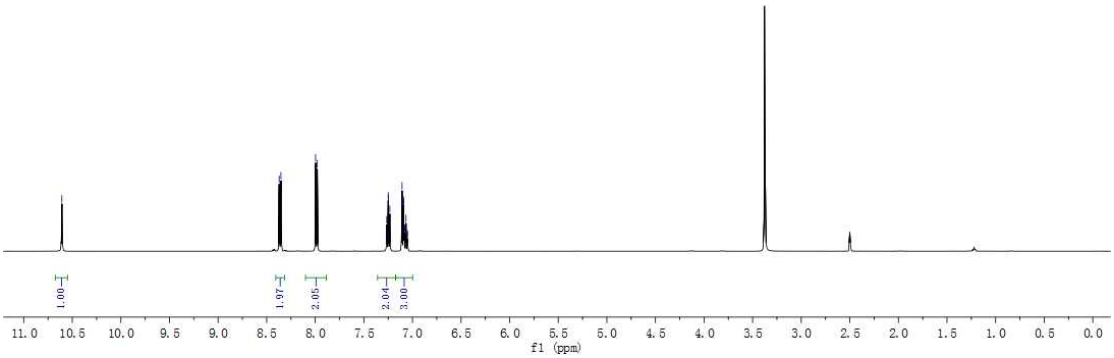

8.269
7.982
7.980
7.965
7.963
7.626
7.611
7.609
7.596
7.581
7.515
7.503
7.500
7.484
2.372
2.315
1.00
2.05
1.04
1.04
3.07
3.10
3.16
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


142.41
140.39
137.69
135.04
134.53
133.93
129.60
129.11
126.97
121.00
112.54
20.59
20.10
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

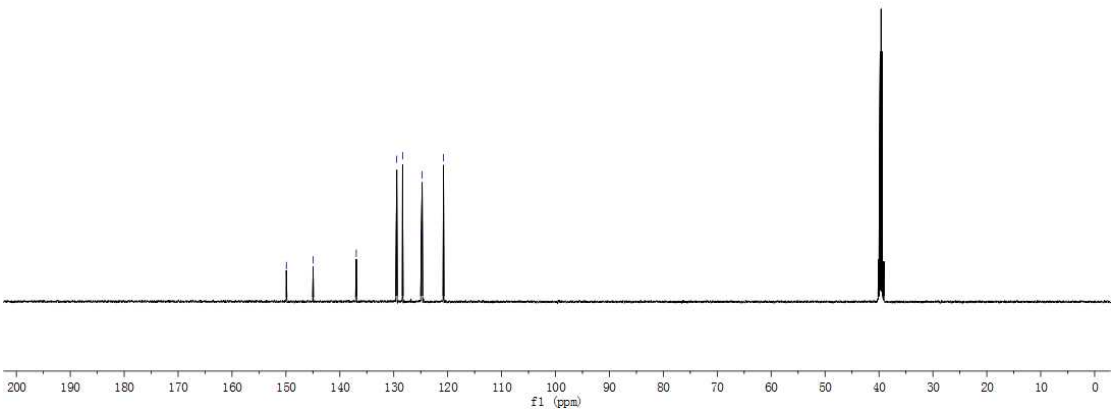

O
S
O
N
H
O2N

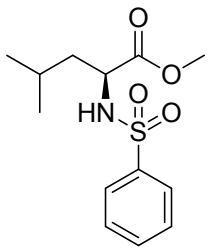
3ae


10.608
8.371
8.353
7.997
7.979
7.265
7.261
7.250
7.248
7.233
7.109
7.107
7.092
7.090
7.084
7.081
7.067
7.054
7.052

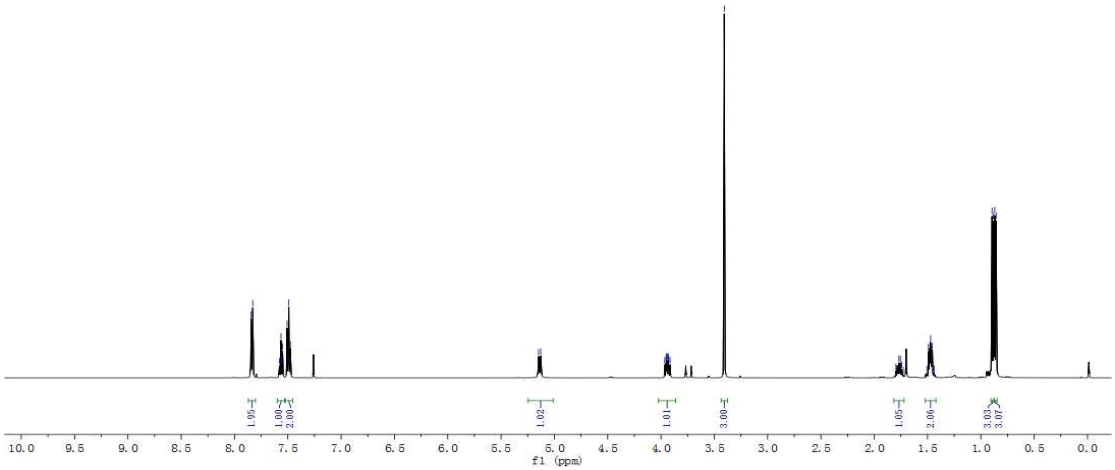

1.00
1.97
2.05
2.04
3.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

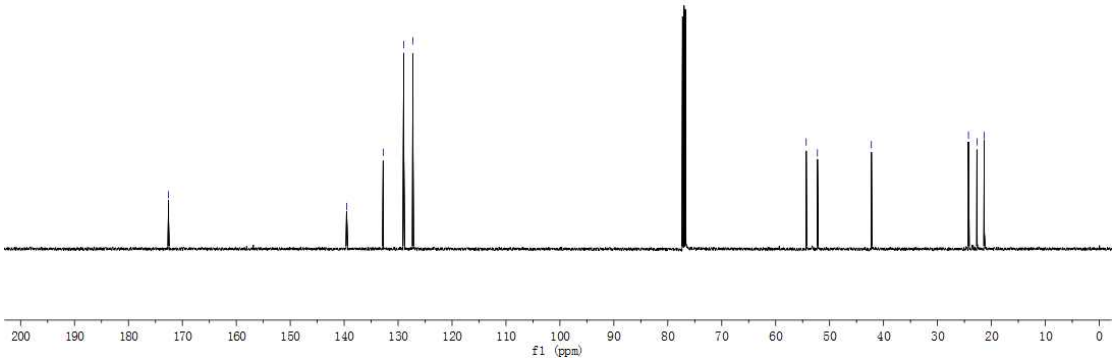

149.92
144.96
136.96
129.45
128.36
124.89
124.73
120.78

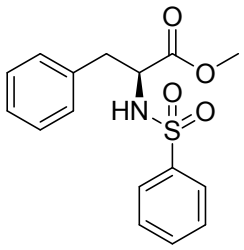

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



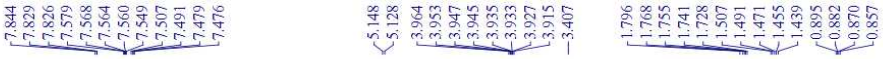
3wa

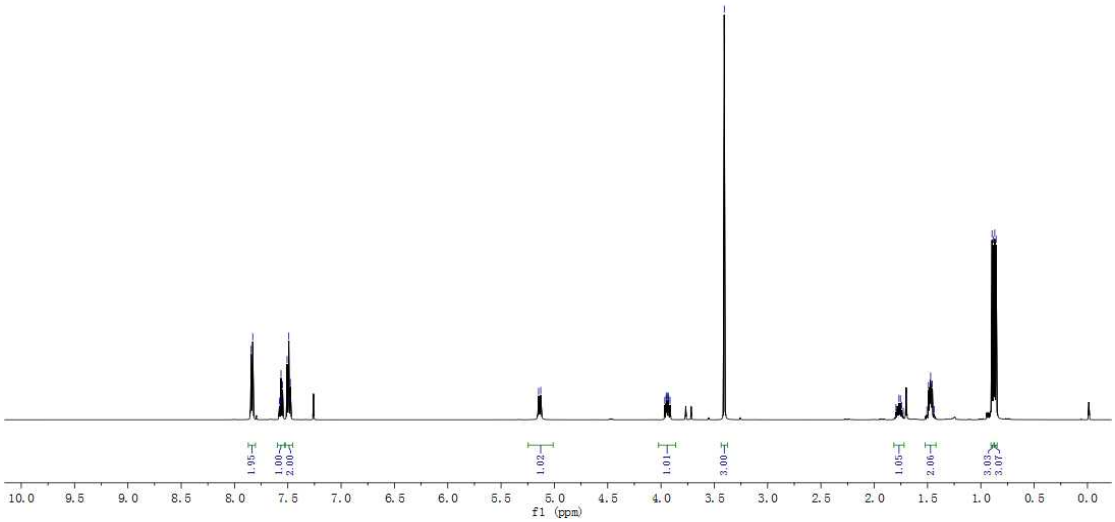




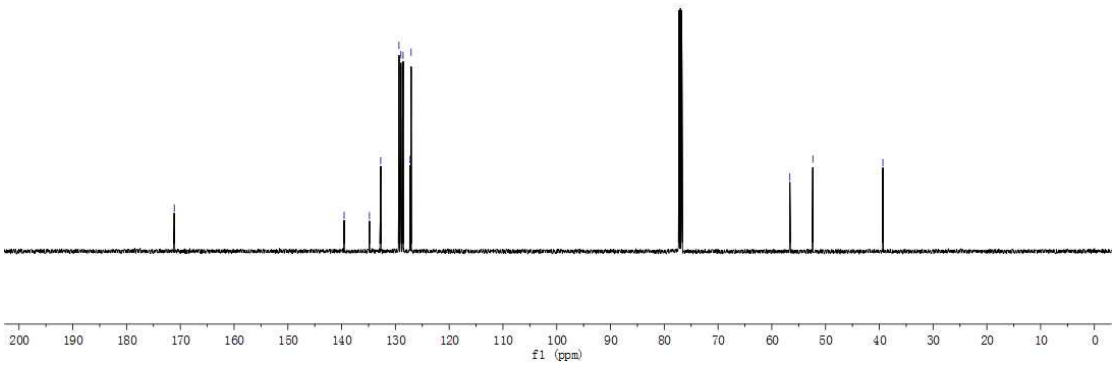


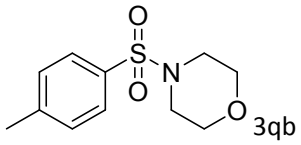
3xa


7.844
7.829
7.826
7.579
7.568
7.564
7.560
7.549
7.507
7.491
7.479
7.476
5.148
5.128
3.964
3.953
3.947
3.945
3.935
3.933
3.927
3.915
3.407
1.796
1.768
1.755
1.741
1.728
1.507
1.491
1.471
1.455
1.439
0.895
0.882
0.870
0.857

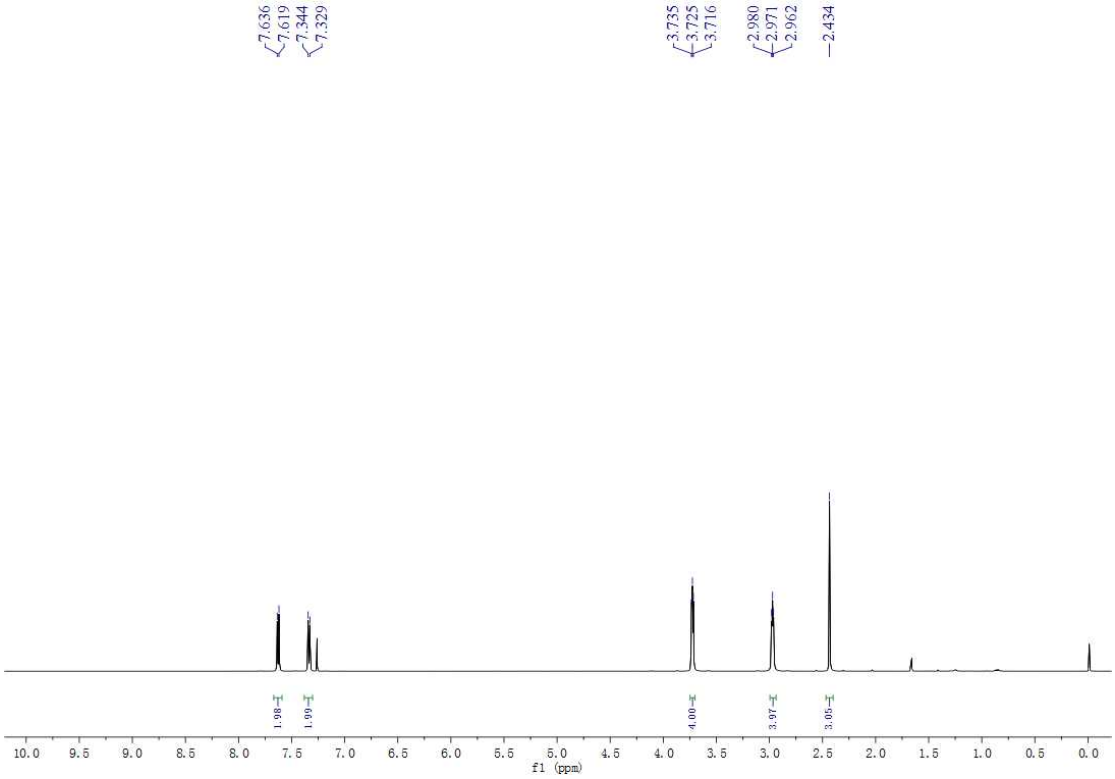

1.95
1.00
2.00
1.02
1.01
3.00
1.05
2.06
3.03
3.07
f1 (ppm)

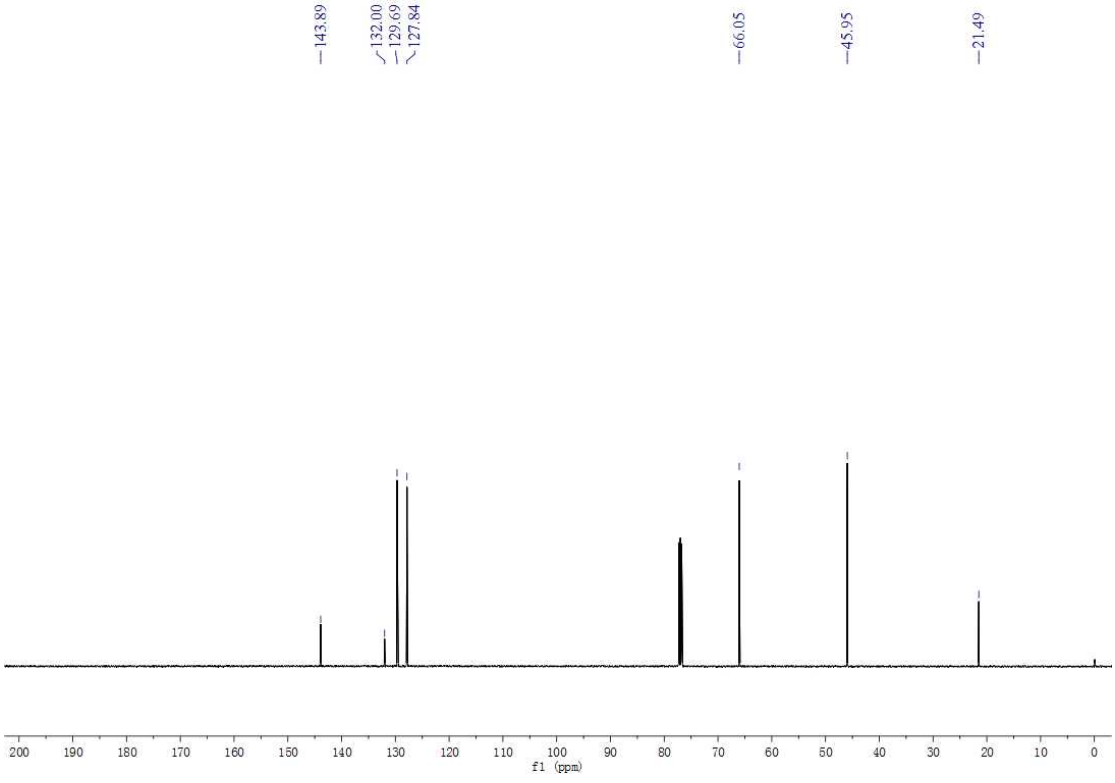

171.12
139.54
134.83
132.73
129.33
128.97
128.57
127.27
127.07
56.64
52.36
39.33

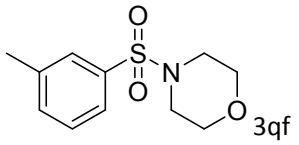

f1 (ppm)



3qb

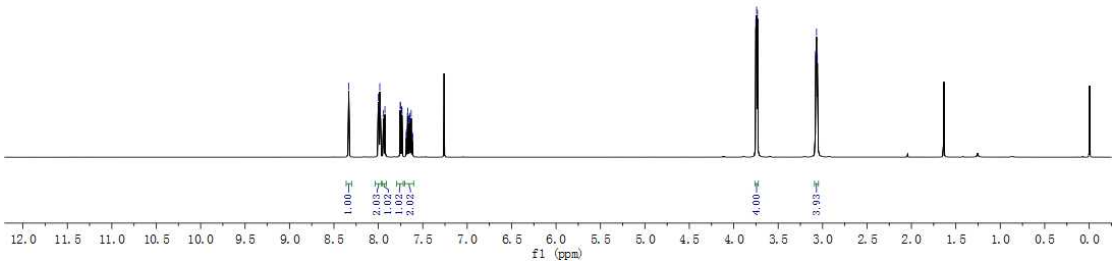

7.636
7.619
7.344
7.329
3.735
3.725
3.716
2.980
2.971
2.962
2.434
1.98
1.99
4.00
3.97
3.05
f1 (ppm)


143.89
132.00
129.69
127.84
66.05
45.95
21.49
f1 (ppm)

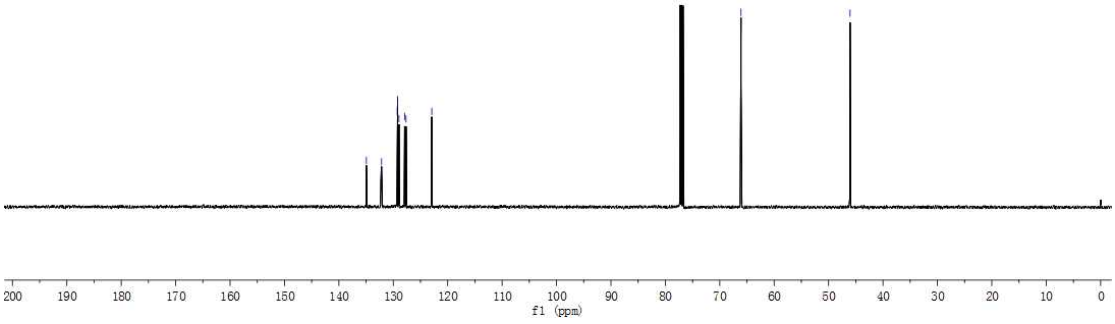

O
S
O
N
O
3qf

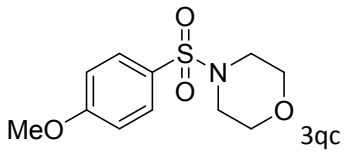

8.333
8.001
7.994
7.984
7.978
7.942
7.926
7.754
7.751
7.737
7.733
7.686
7.684
7.672
7.670
7.657
7.654
7.647
7.644
7.630
7.617
7.615


3.752
3.743
3.733
3.078
3.068
3.059

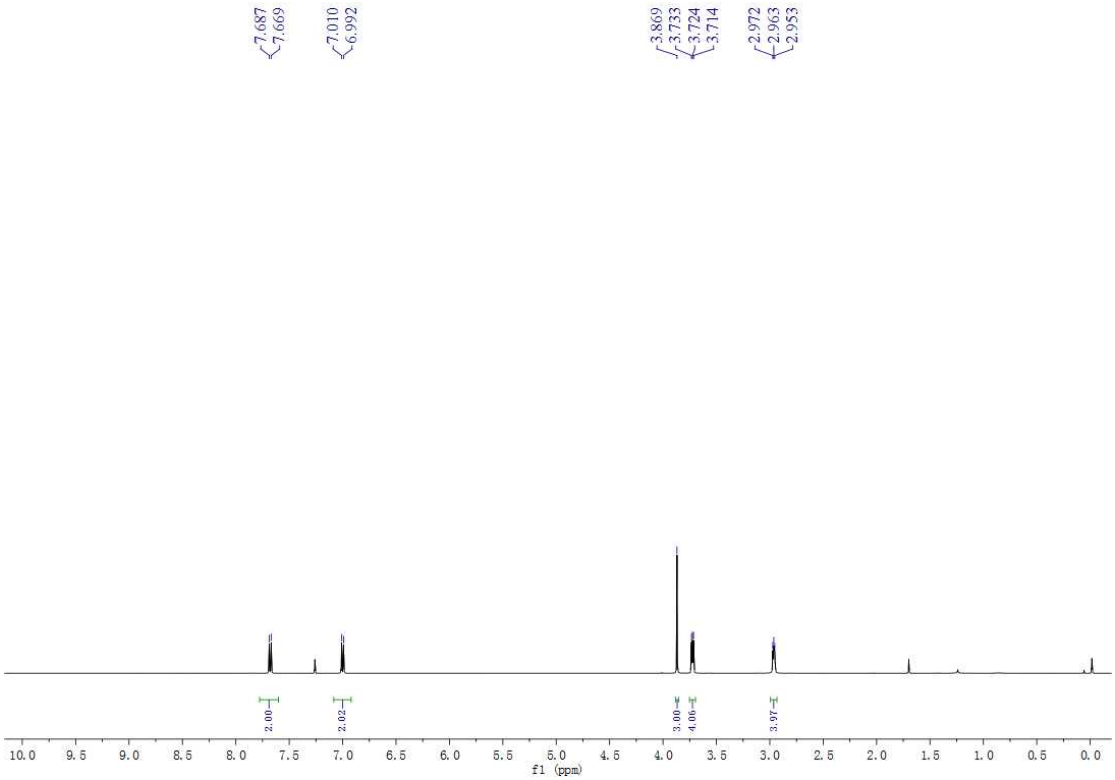

1.00
2.03
1.02
1.02
2.02
4.00
3.93
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

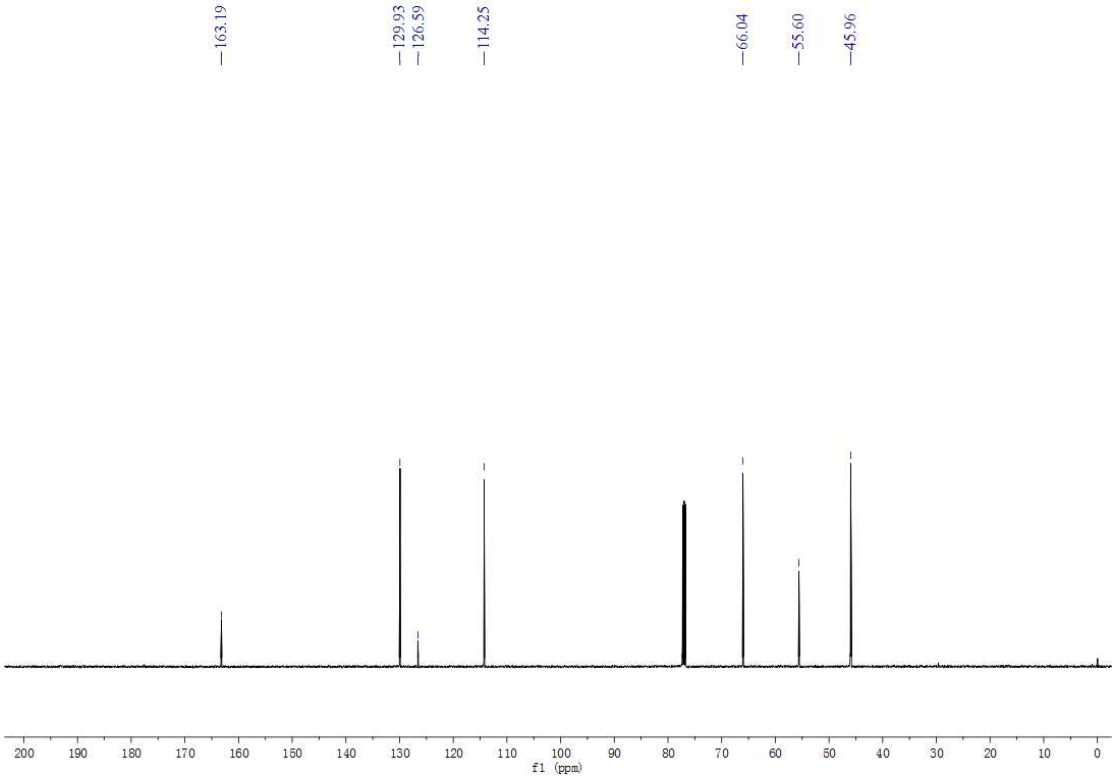

134.94
132.27
132.15
129.29
129.21
129.20
128.96
127.92
127.65
122.92
66.09
46.05

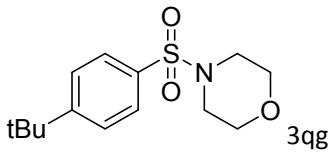

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
S
O
N
O
MeO

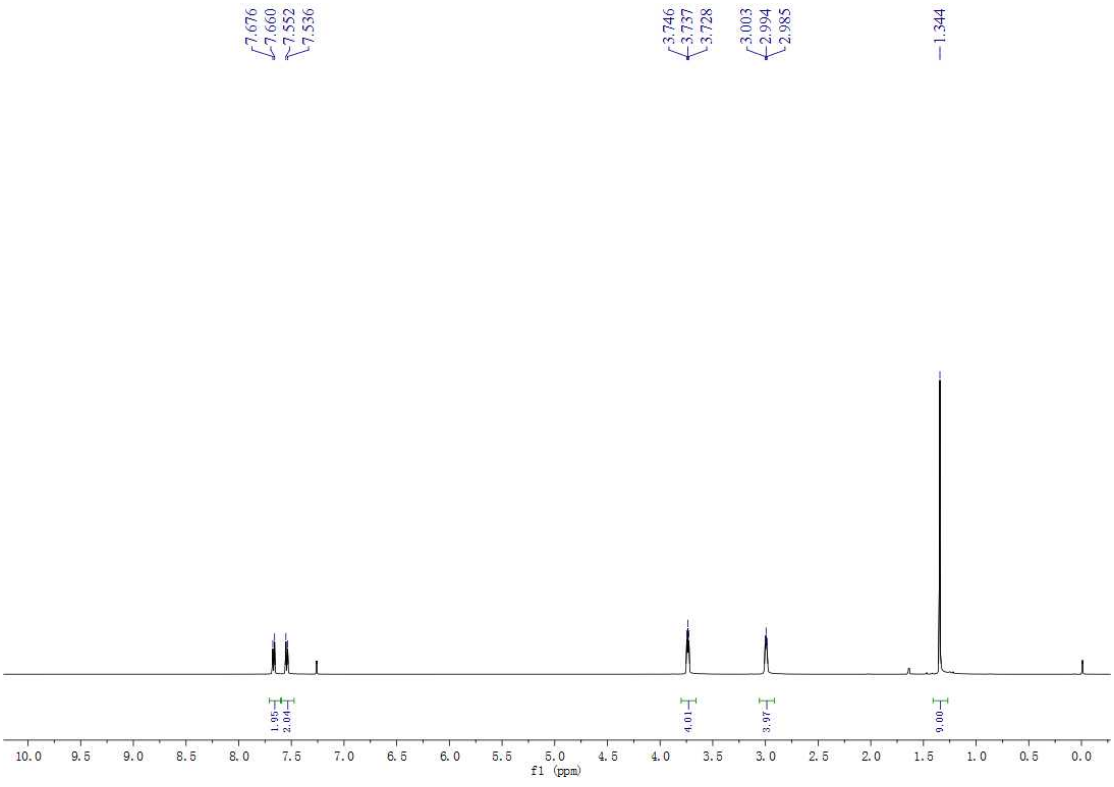
3qc

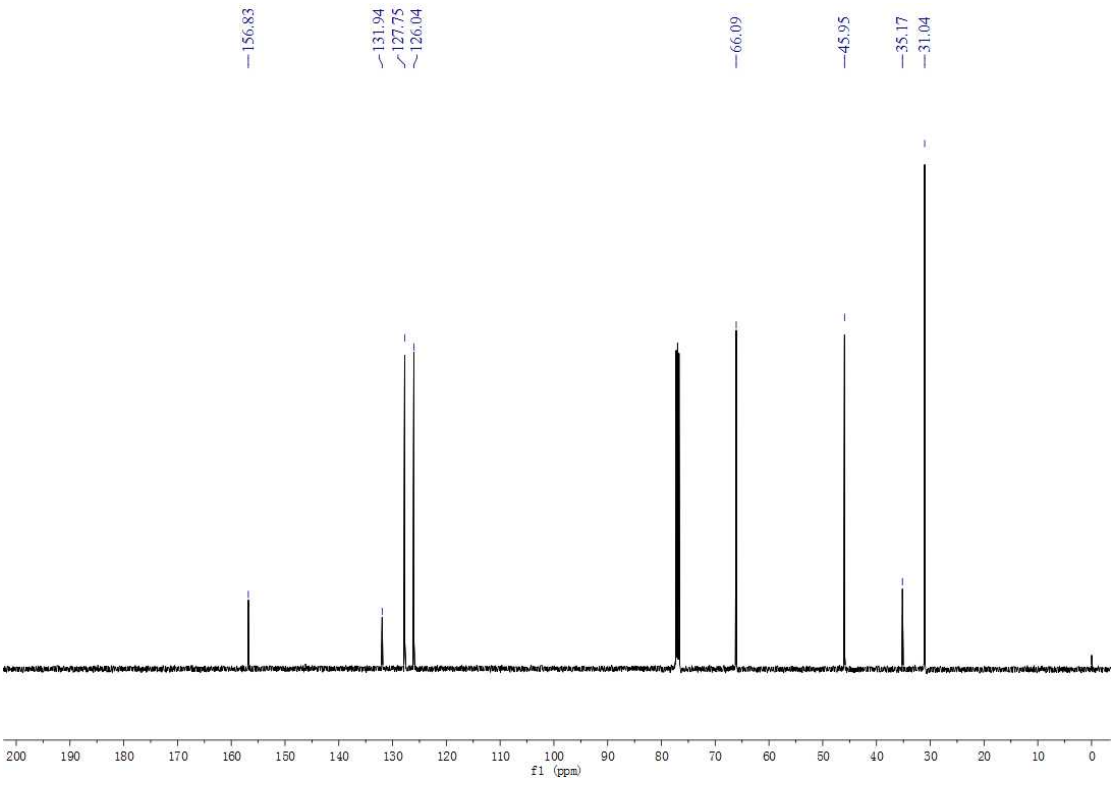

7.687
7.669
7.010
6.992
3.869
3.733
3.724
3.714
2.972
2.963
2.953
2.00
2.02
3.00
4.06
3.97
f1 (ppm)

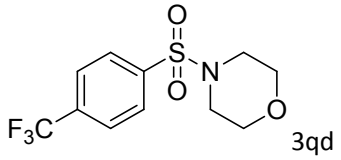

163.19
129.93
126.59
114.25
66.04
55.60
45.96
f1 (ppm)


O
S
O
N
O
tBu

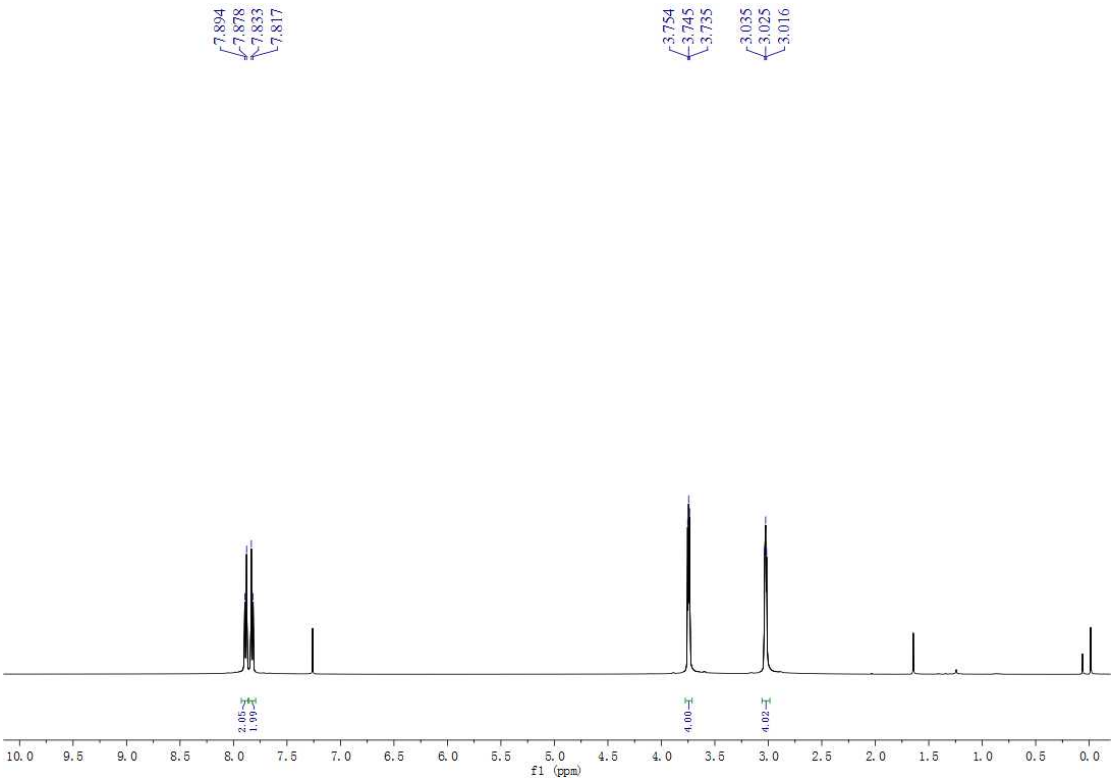
3qg

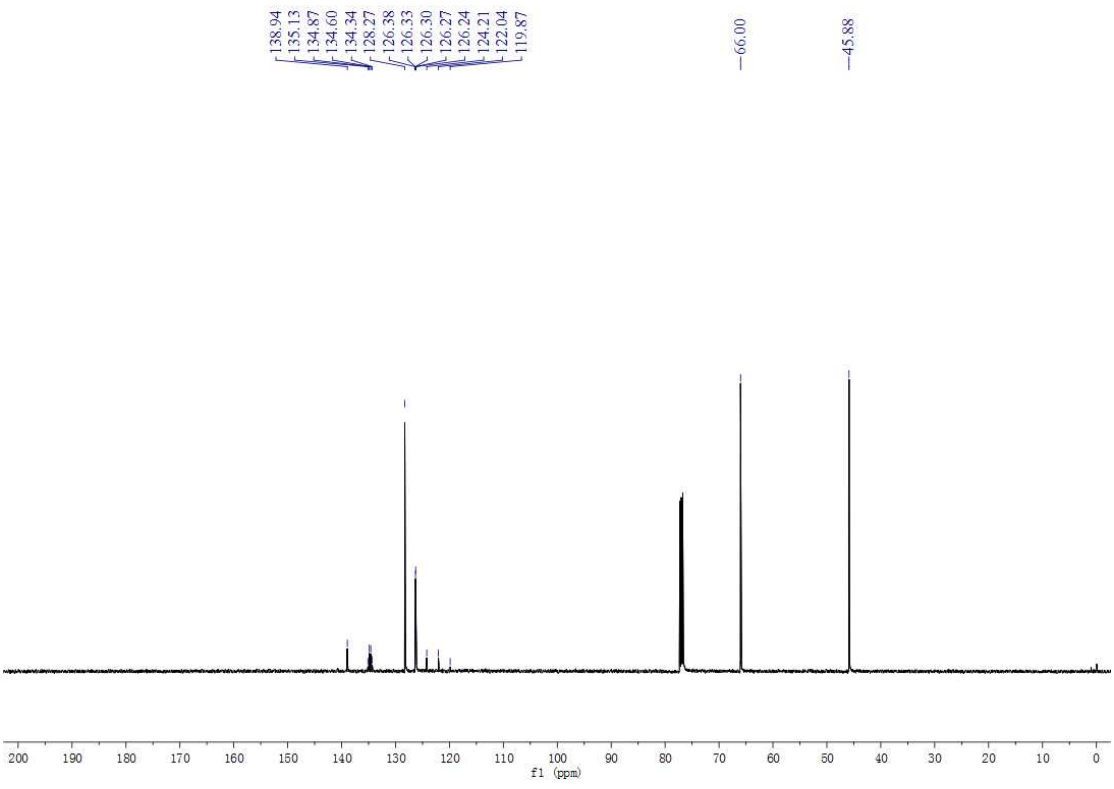

7.676
7.660
7.552
7.536
3.746
3.737
3.728
3.003
2.994
2.985
1.344
1.95
2.04
4.01
3.97
9.00
f1 (ppm)

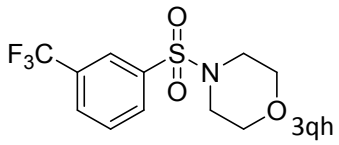

156.83
131.94
127.75
126.04
66.09
45.95
35.17
31.04
f1 (ppm)

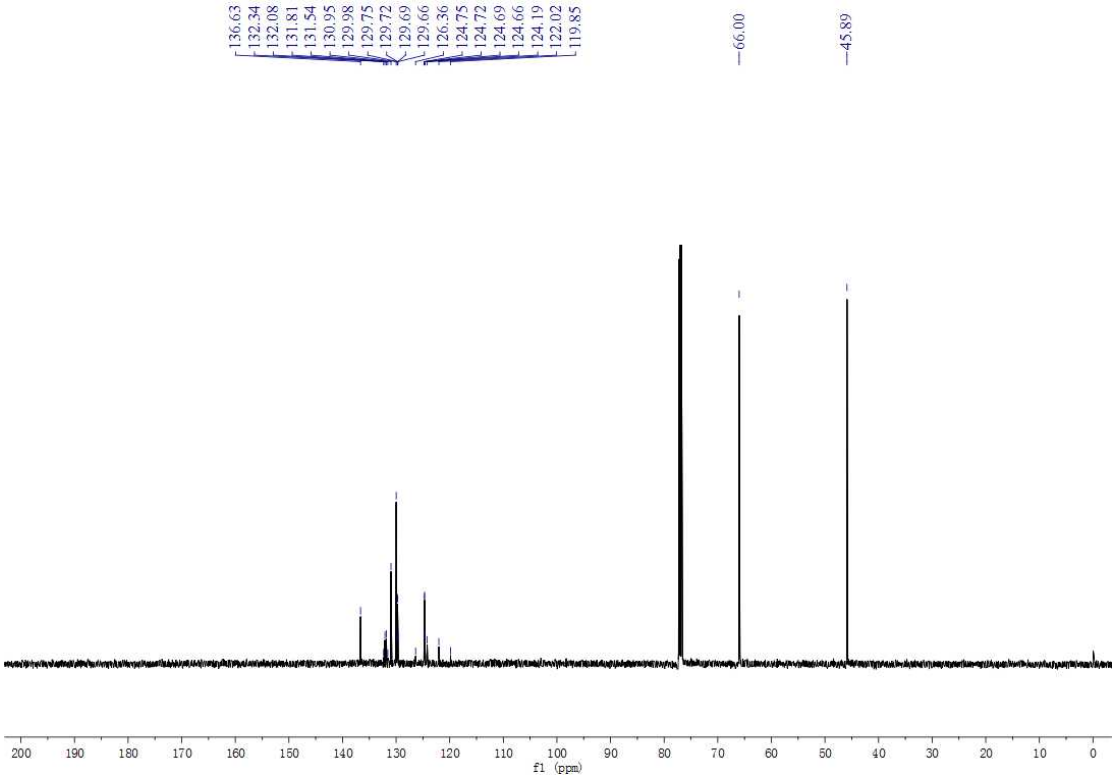

O
S
O
N
O
F3C

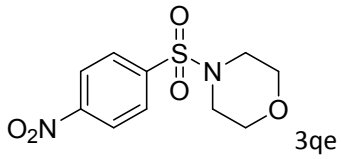
3qd


7.894
7.878
7.833
7.817
3.754
3.745
3.735
3.035
3.025
3.016
2.05
1.99
4.00
4.02
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
f1 (ppm)


138.94
135.13
134.87
134.60
134.34
128.27
126.38
126.33
126.30
126.27
126.24
124.21
122.04
119.87
66.00
45.88
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

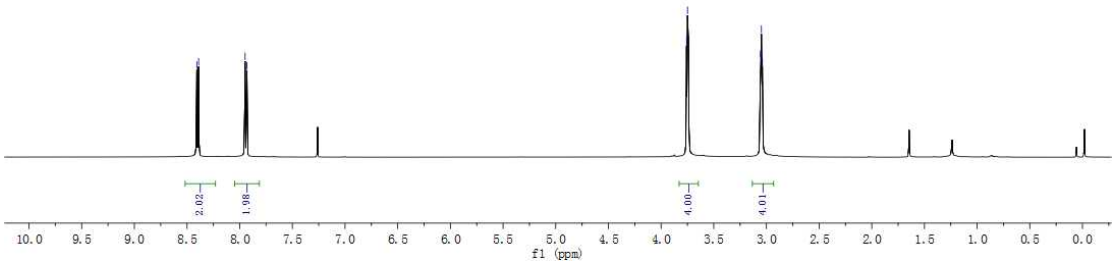

F3C
O
S
O
N
O
3qh


136.63
132.34
132.08
131.81
131.54
130.95
129.98
129.75
129.72
129.69
129.66
126.36
124.75
124.72
124.69
124.66
124.19
122.02
119.85
66.00
45.89
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

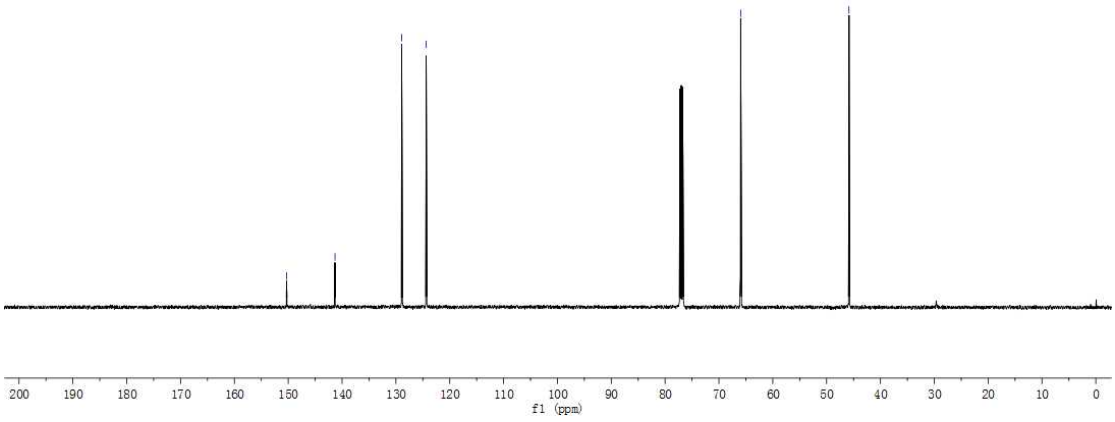

O
S
O
N
O
O2N

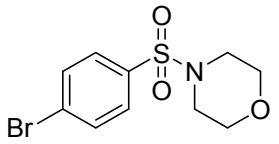
3qe

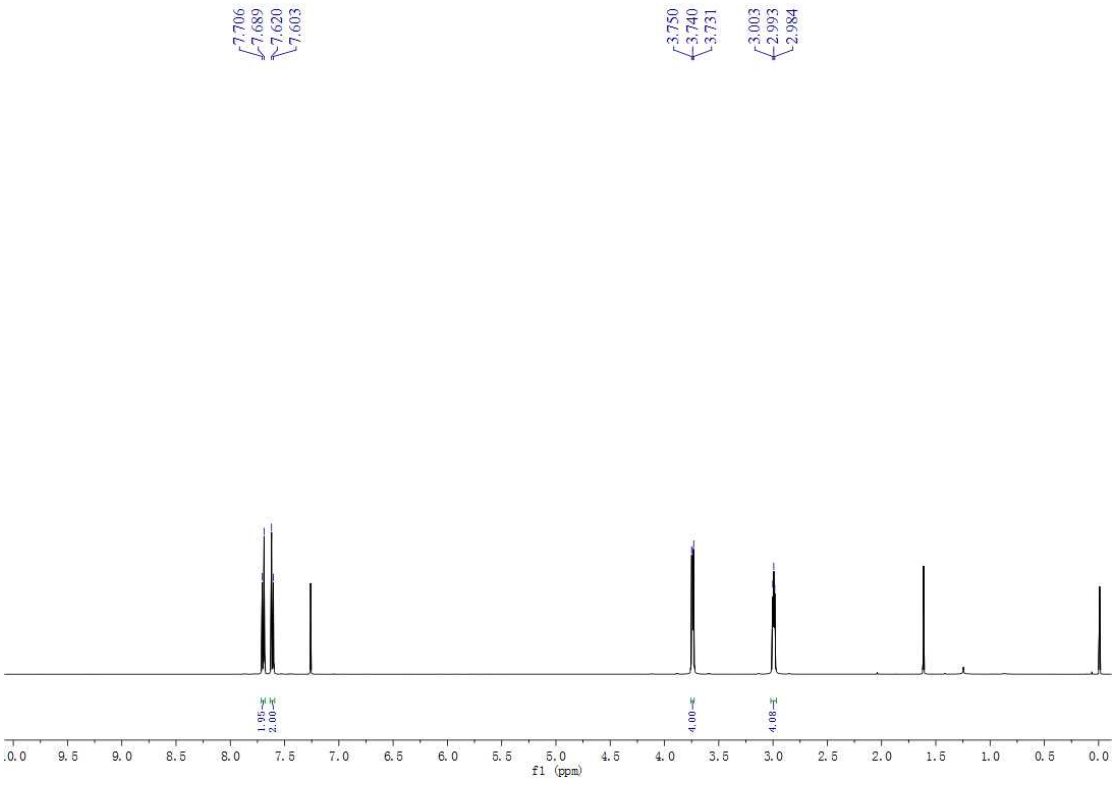

8.407
8.390
7.949
7.932
3.758
3.749
3.739
3.057
3.048
3.038

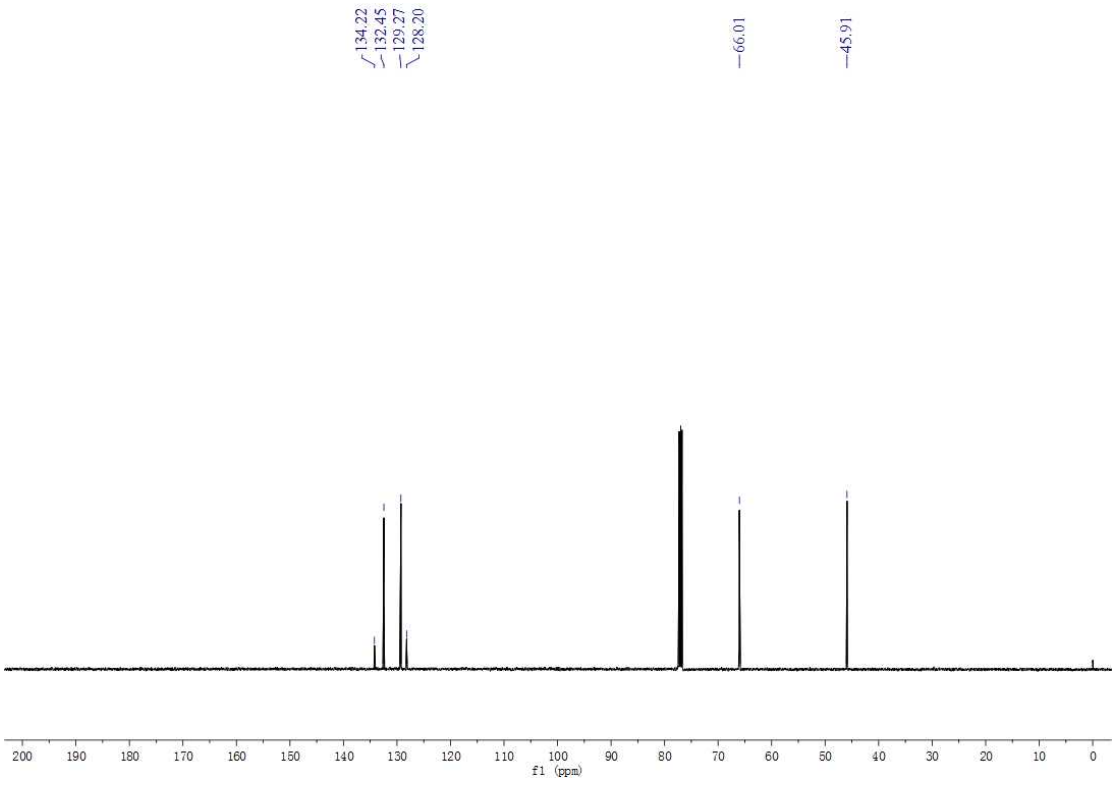

2.02
1.98
4.00
4.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

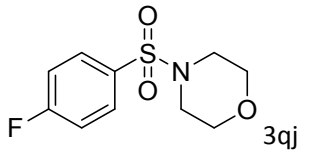

150.31
141.31
128.93
124.37
65.96
45.85


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
S
N
O
O
Br

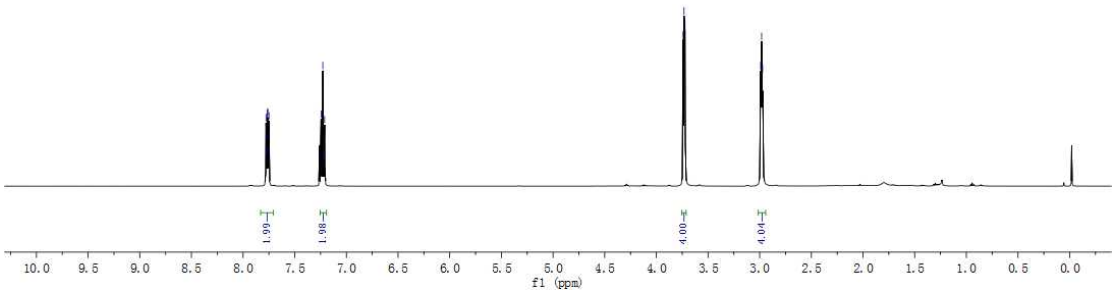

7.706
7.689
7.620
7.603
3.750
3.740
3.731
3.003
2.993
2.984
1.95
2.00
4.00
4.08
f1 (ppm)


134.22
132.45
129.27
128.20
66.01
45.91
f1 (ppm)

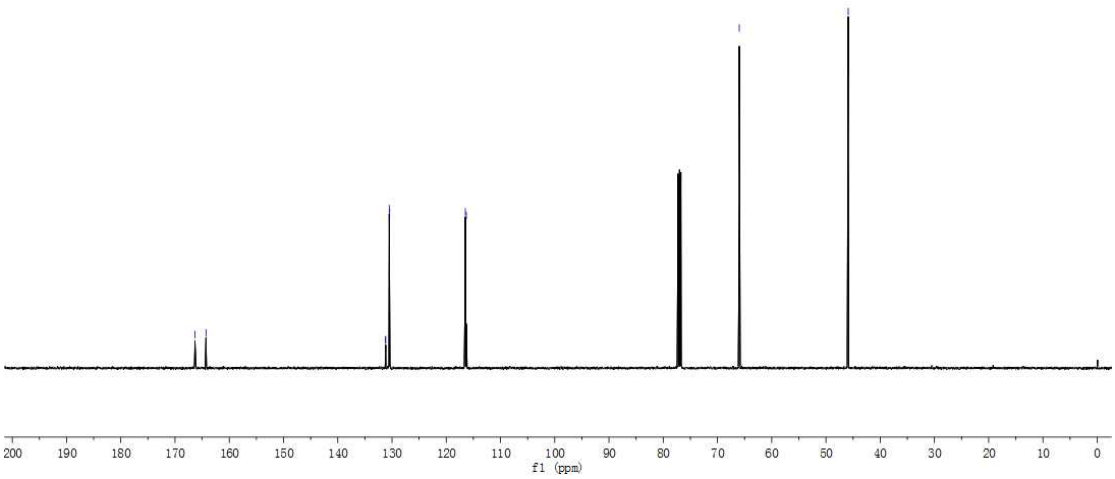

O
S
O
N
O
F

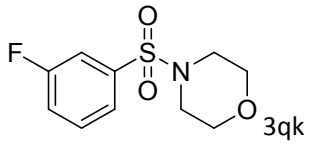
3qj


7.778
7.774
7.768
7.764
7.760
7.754
7.750
7.245
7.241
7.228
7.215
7.211
3.742
3.732
3.723
2.991
2.981
2.972

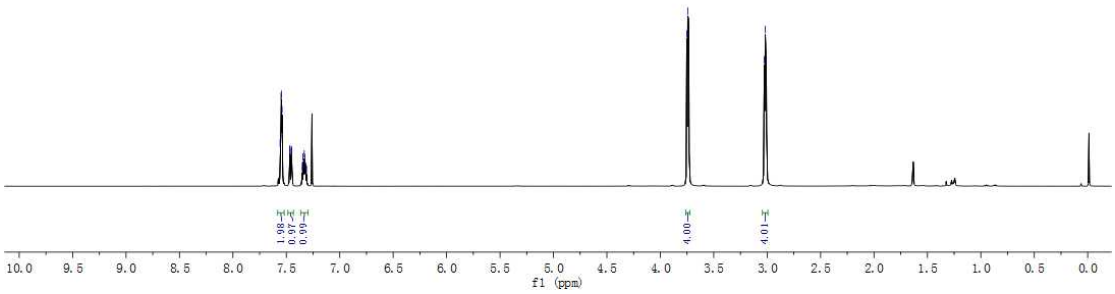

1.99
1.98
4.00
4.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.32
164.29
131.19
131.16
130.50
130.43
116.48
116.30
65.98
45.90

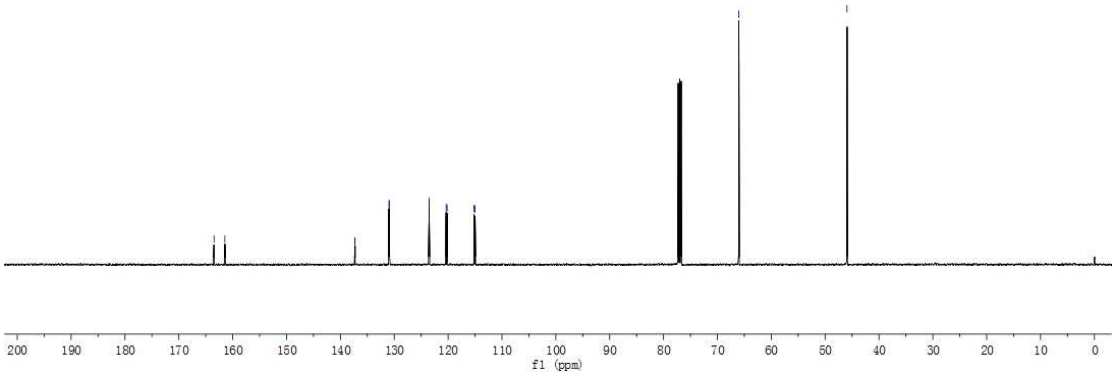

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

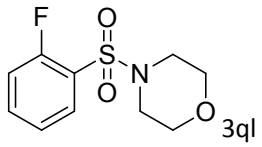

O
S
O
N
O
F
3qk


7.559
7.550
7.546
7.540
7.468
7.466
7.455
7.452
7.451
7.450
7.351
7.346
7.333
7.327
7.316
7.311
3.752
3.743
3.733
3.027
3.018
3.008


1.98
0.97
0.99
4.00
4.01
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
f1 (ppm)

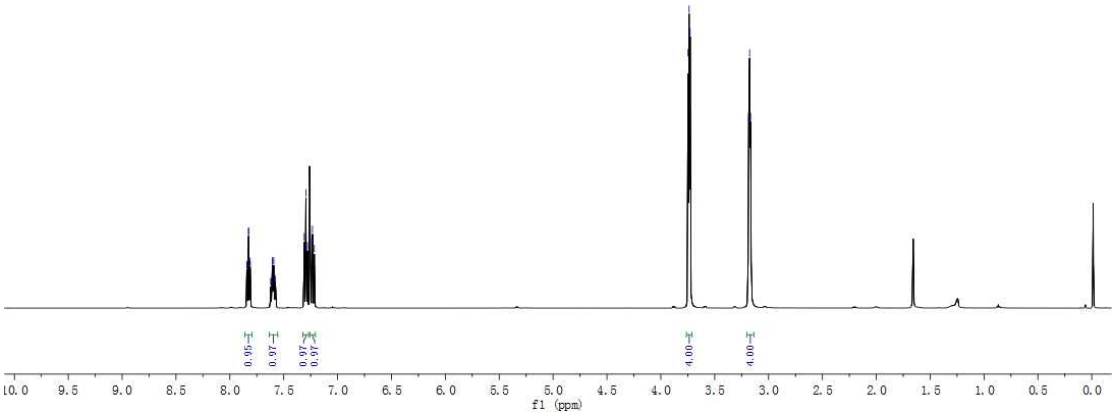

163.46
161.45
137.33
137.28
130.98
130.91
123.53
123.50
120.36
120.19
115.19
114.99
66.02
45.94


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

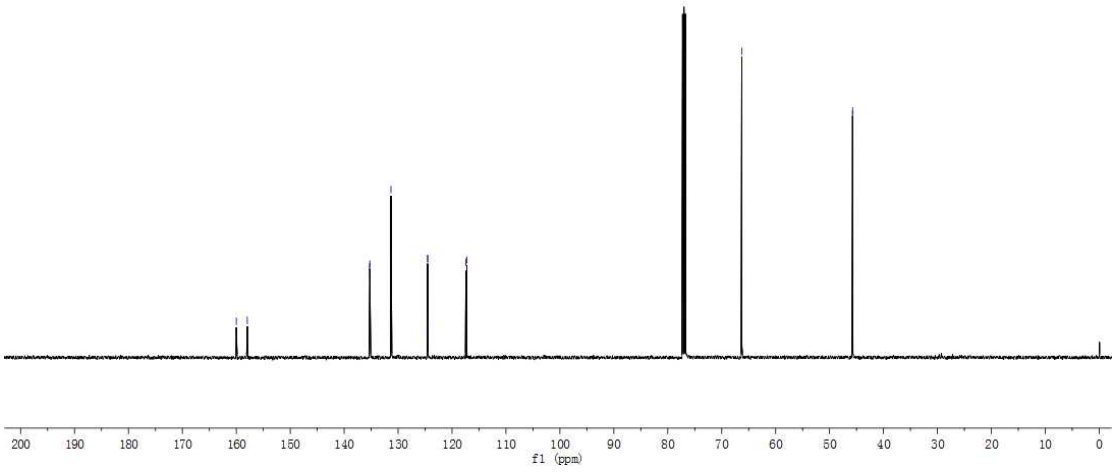

F
O
S
O
N
O
3ql

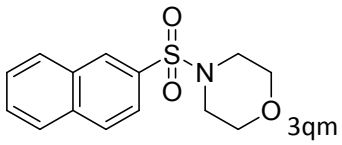

7.843
7.839
7.827
7.826
7.813
7.810
7.621
7.617
7.606
7.602
7.592
7.586
7.576
7.310
7.308
7.295
7.279
7.253
7.236
7.233
7.216

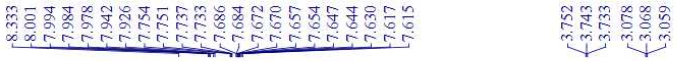

3.747
3.737
3.728
3.185
3.176
3.166

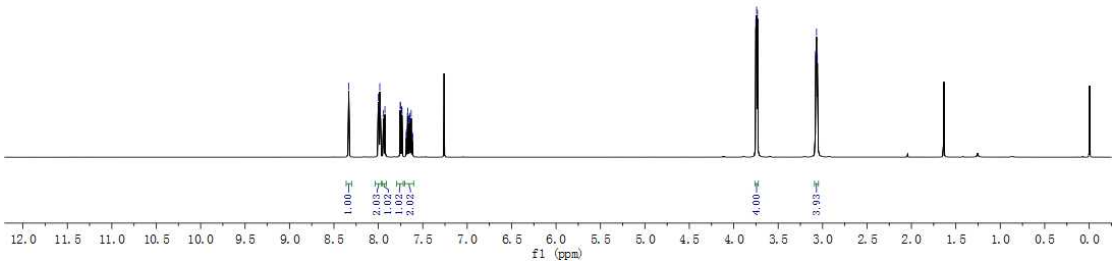

0.95
0.97
0.97
0.97
4.00
4.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


160.00
157.96
135.31
135.24
131.33
124.58
124.54
124.51
124.47
117.44
117.26
66.28
45.74
45.73

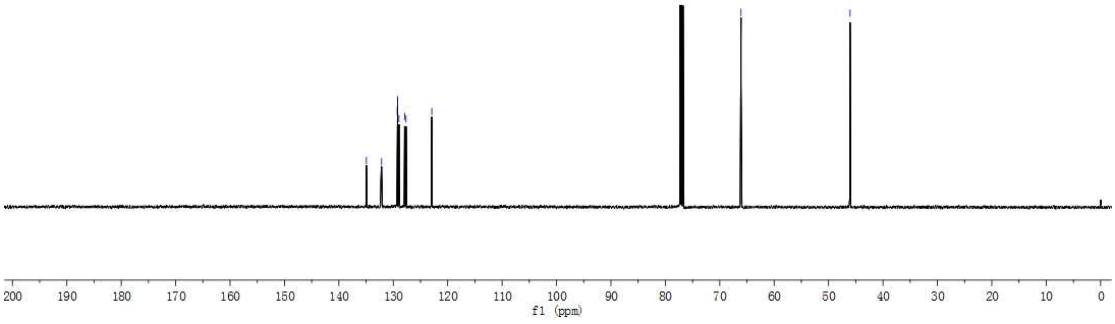

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

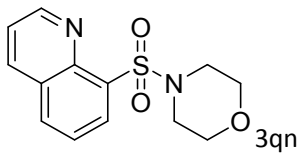

O
S
N
O
O
3qm


8.333
8.001
7.994
7.984
7.978
7.942
7.926
7.754
7.751
7.737
7.733
7.686
7.684
7.672
7.670
7.657
7.654
7.647
7.644
7.630
7.617
7.615
3.752
3.743
3.733
3.078
3.068
3.059

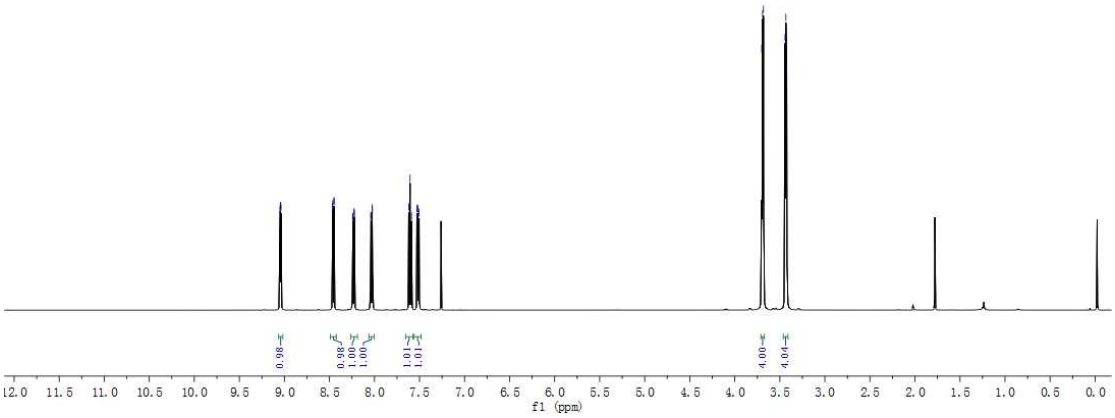

1.00
2.03
1.02
1.02
2.02
4.00
3.93
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


134.94
132.27
132.15
129.29
129.21
129.20
128.96
127.92
127.65
122.92
66.09
46.05

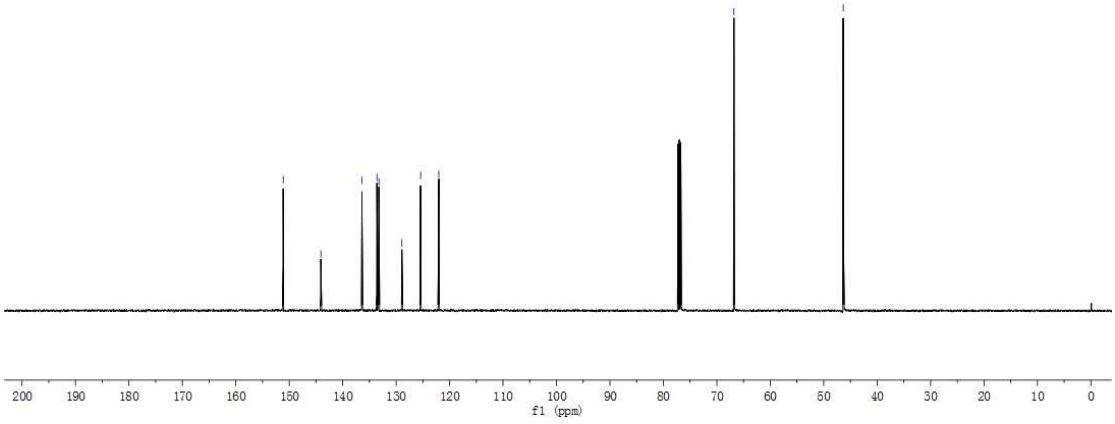

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

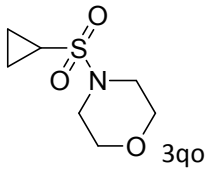

N
O
S
O
N
O
3qn


9.050
9.046
9.041
9.038
8.466
8.463
8.451
8.448
8.243
8.240
8.227
8.223
8.042
8.039
8.025
8.023
7.620
7.605
7.589
7.529
7.521
7.513
7.504
3.701
3.692
3.682
3.442
3.433
3.423


0.98
0.98
1.00
1.00
1.01
1.01
4.00
4.04
12.0 11.5 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 1.5 1.0 0.5 0.0
f1 (ppm)

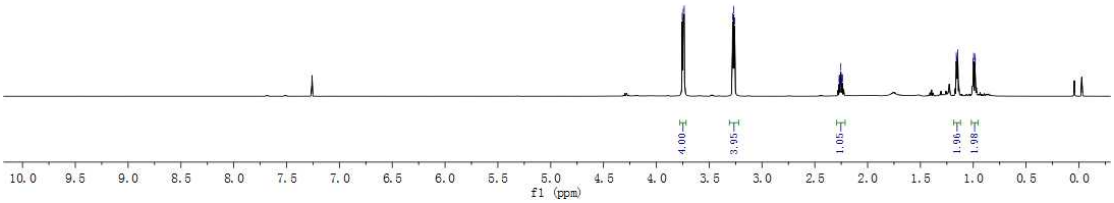

151.12
144.08
136.41
136.34
133.59
133.22
128.93
125.42
122.02
66.80
46.34

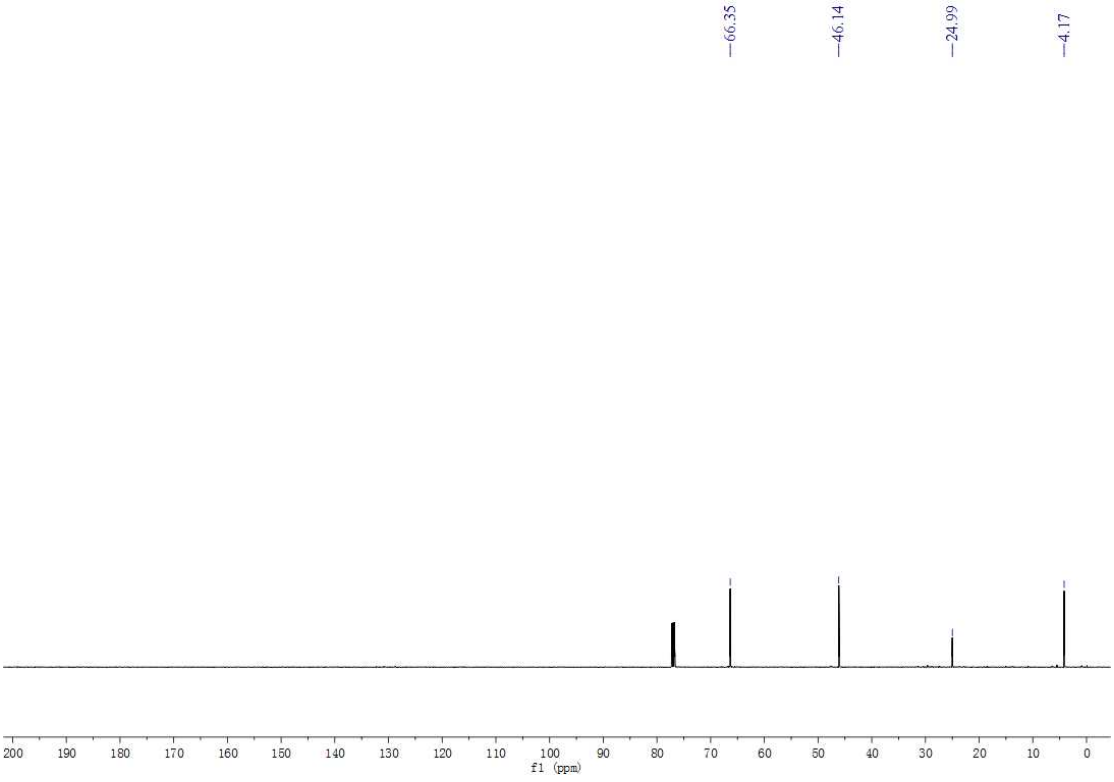

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

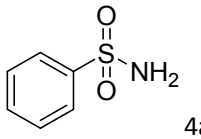


3qo

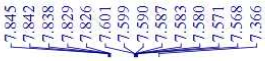

3.755
3.746
3.736
3.278
3.268
3.259
2.270
2.264
2.260
2.254
2.248
2.244
2.238
1.160
1.156
1.151
1.147
1.001
0.996
0.985
0.981

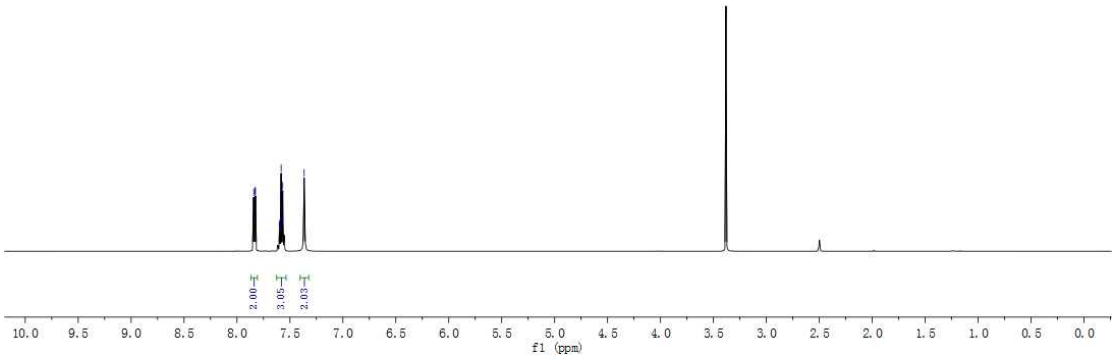

4.00
3.95
1.05
1.96
1.98
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

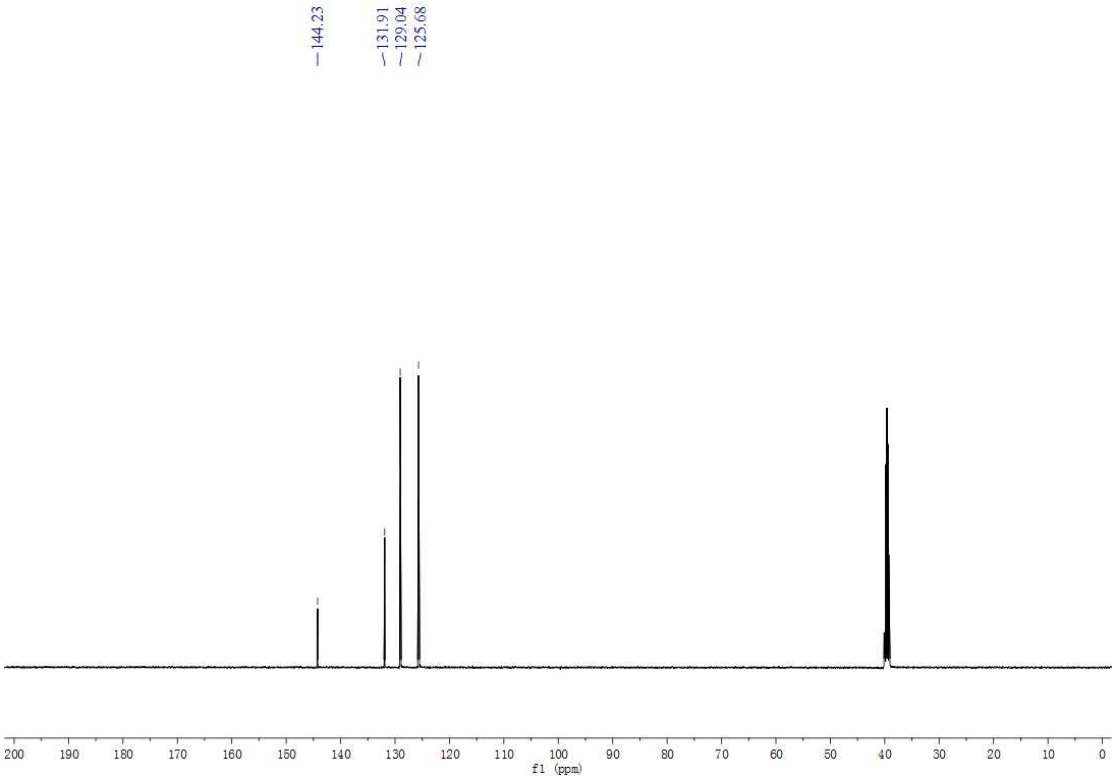

66.35
46.14
24.99
4.17
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

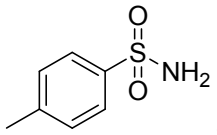


4a

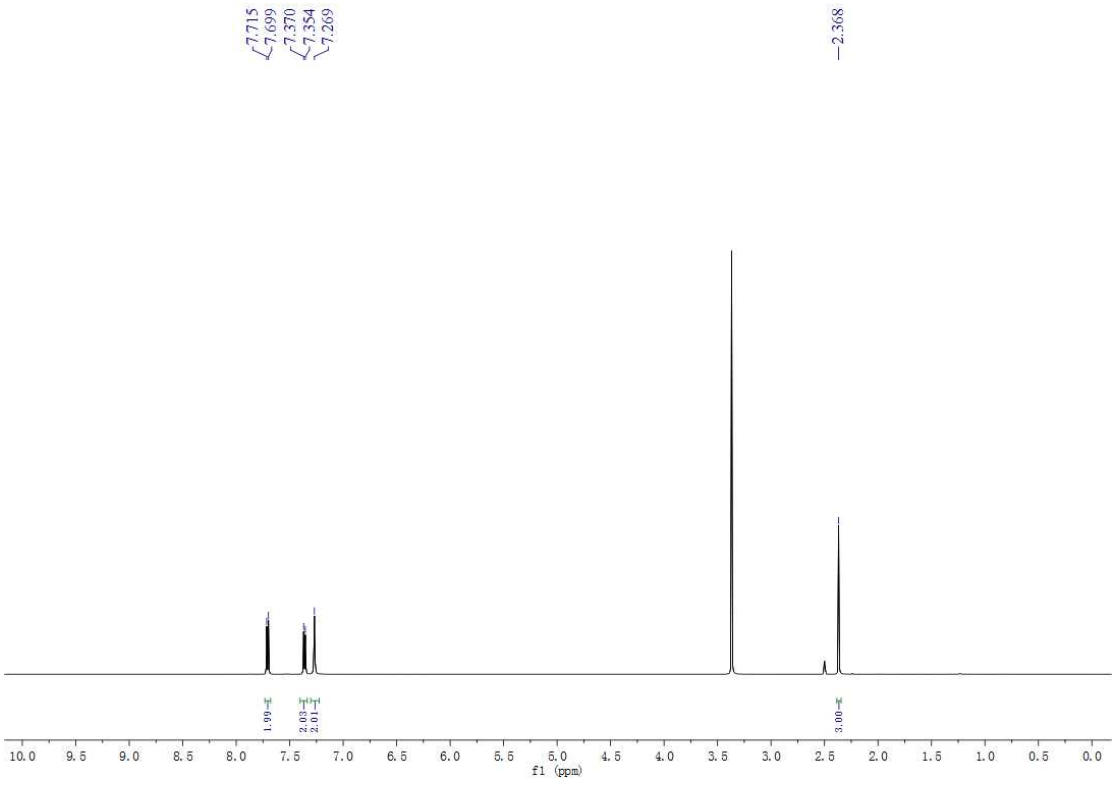

7.845
7.842
7.838
7.829
7.826
7.601
7.599
7.590
7.587
7.583
7.580
7.571
7.568
7.366

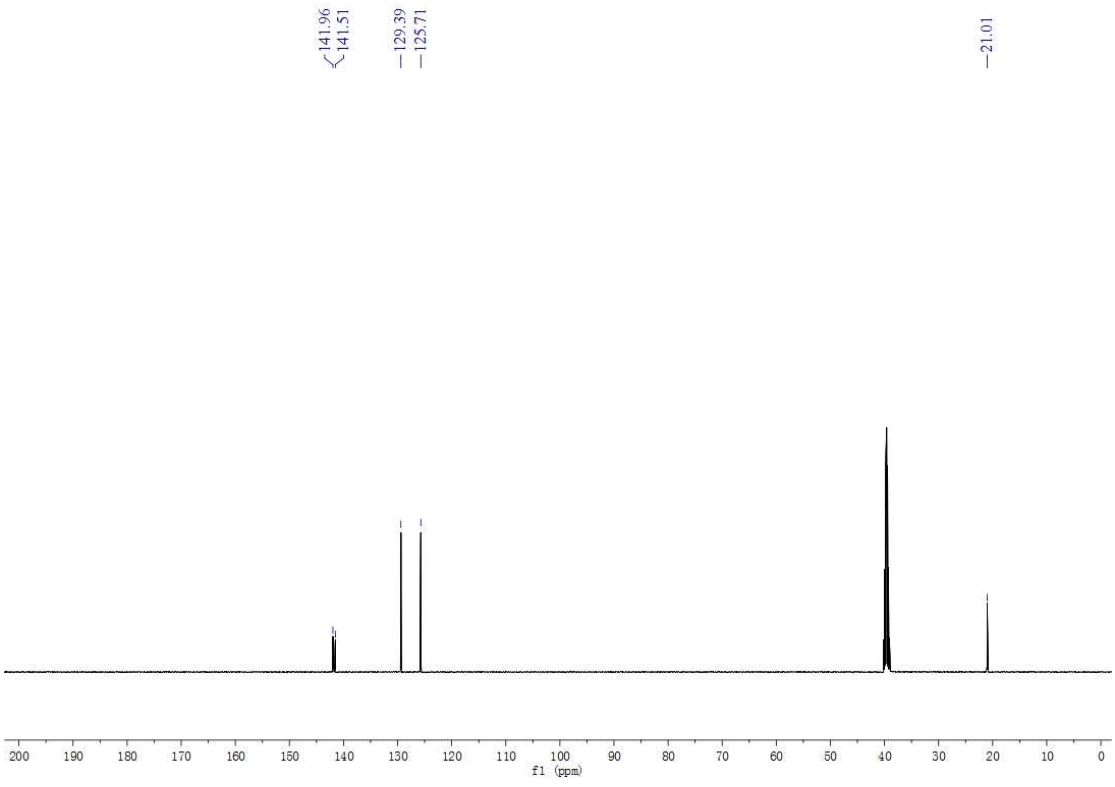

2.00
3.05
2.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

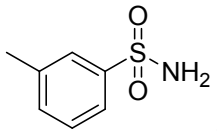

144.23
131.91
129.04
125.68
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



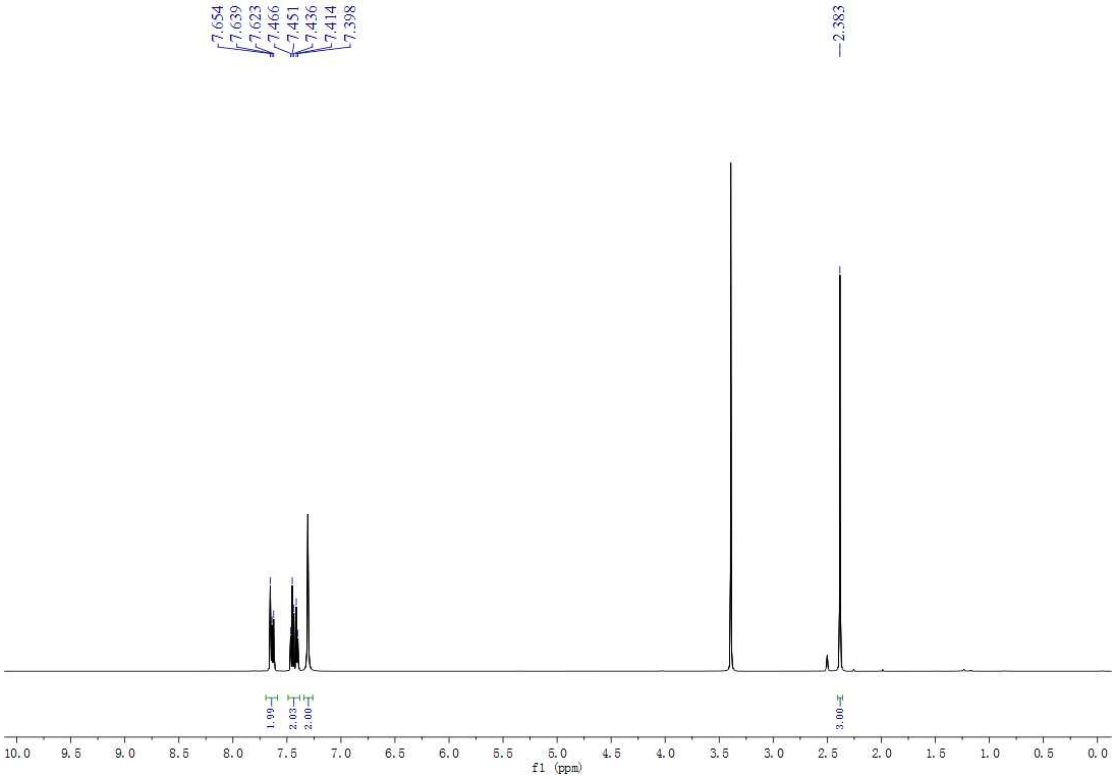
4b

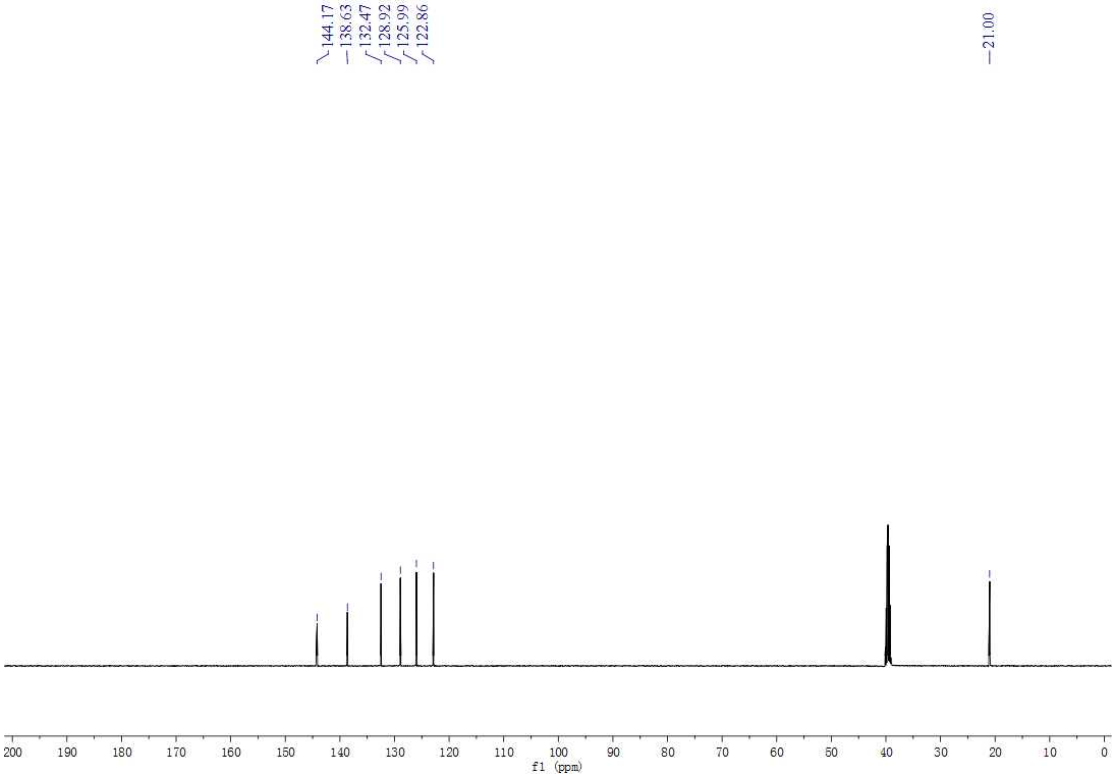

7.715
7.699
7.370
7.354
7.269
2.368
1.99
2.03
2.01
3.00
f1 (ppm)

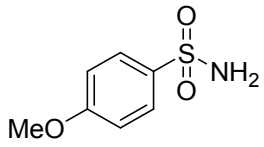

141.96
141.51
129.39
125.71
21.01
f1 (ppm)



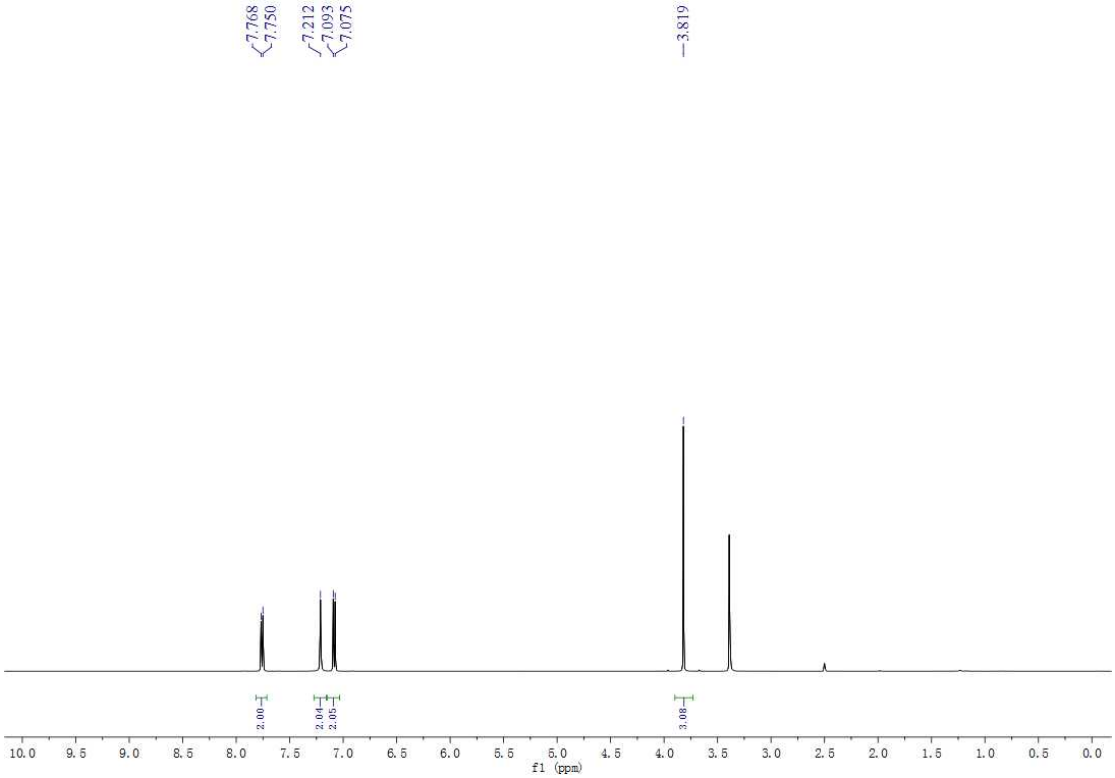
4f

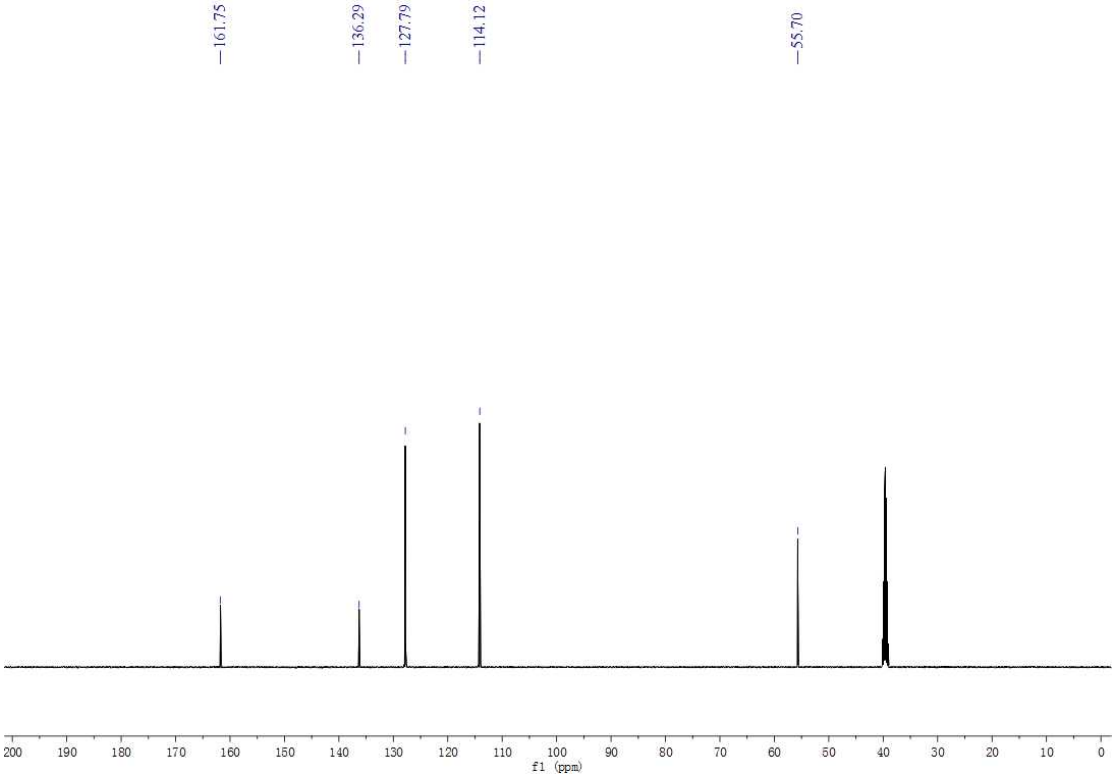

7.654
7.639
7.623
7.466
7.451
7.436
7.414
7.398
2.383
1.99
2.03
2.00
3.00
f1 (ppm)

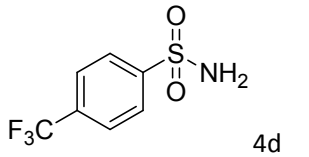

144.17
138.63
132.47
128.92
125.99
122.86
21.00
f1 (ppm)


O
S
NH2
O
MeO

4c

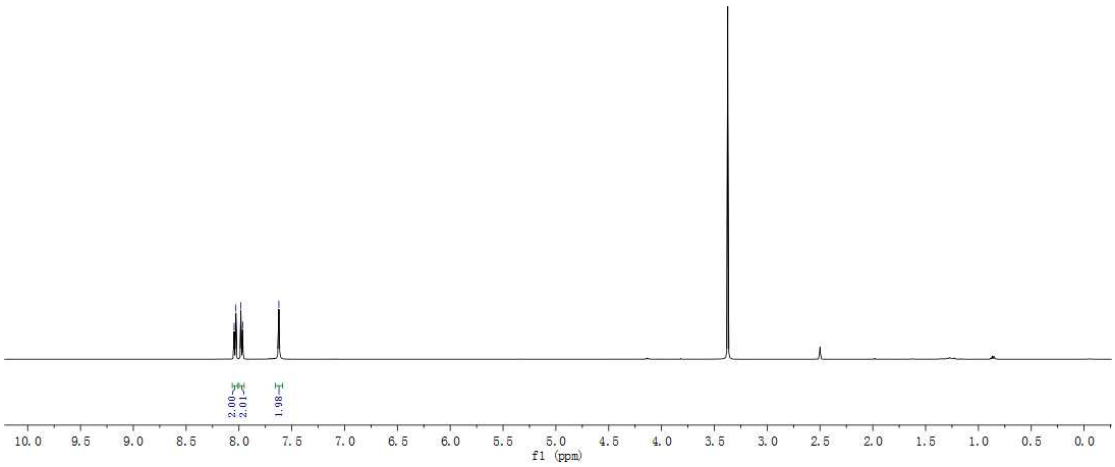

7.768
7.750
7.212
7.093
7.075
3.819
2.00
2.04
2.05
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.75
136.29
127.79
114.12
55.70
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

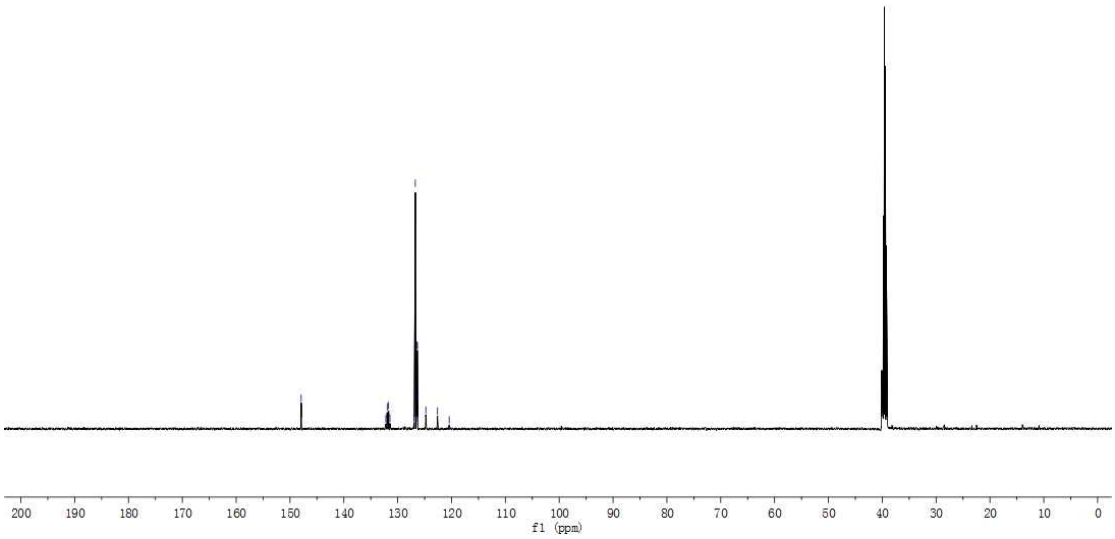

O
S
O
NH2
F3C

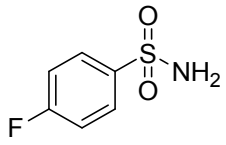
4d


8.046
8.029
7.982
7.965
7.622


2.00
2.01
1.98
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
f1 (ppm)

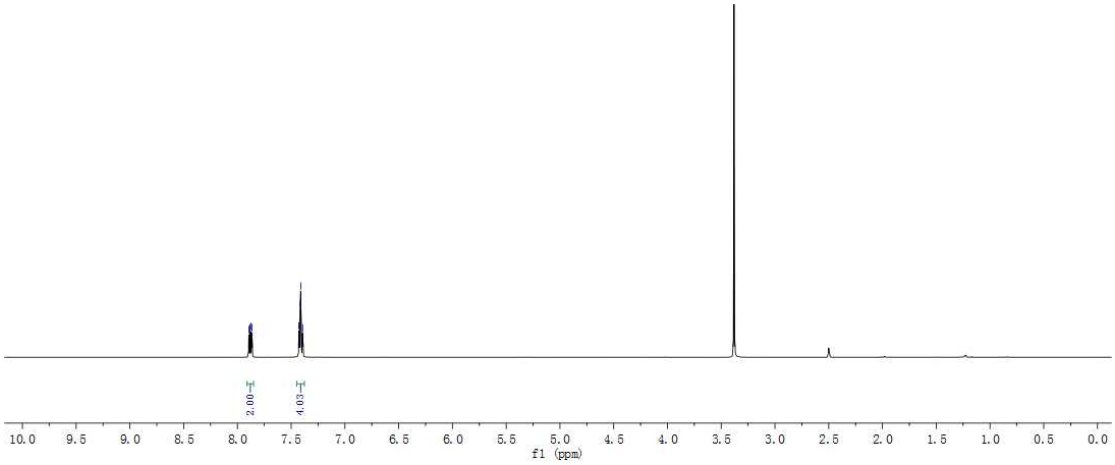

147.93
132.20
131.95
131.69
131.43
126.94
126.73
126.40
126.37
126.34
126.31
124.77
122.60
120.44


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

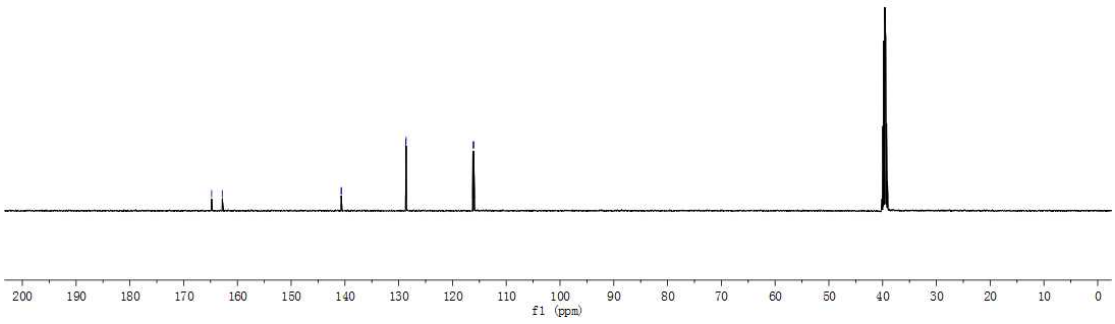

O
S
NH2
O
F

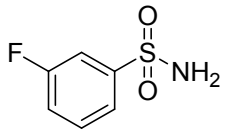
4j


7.894
7.883
7.876
7.866
7.429
7.425
7.415
7.411
7.393


2.00
4.03
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
f1 (ppm)

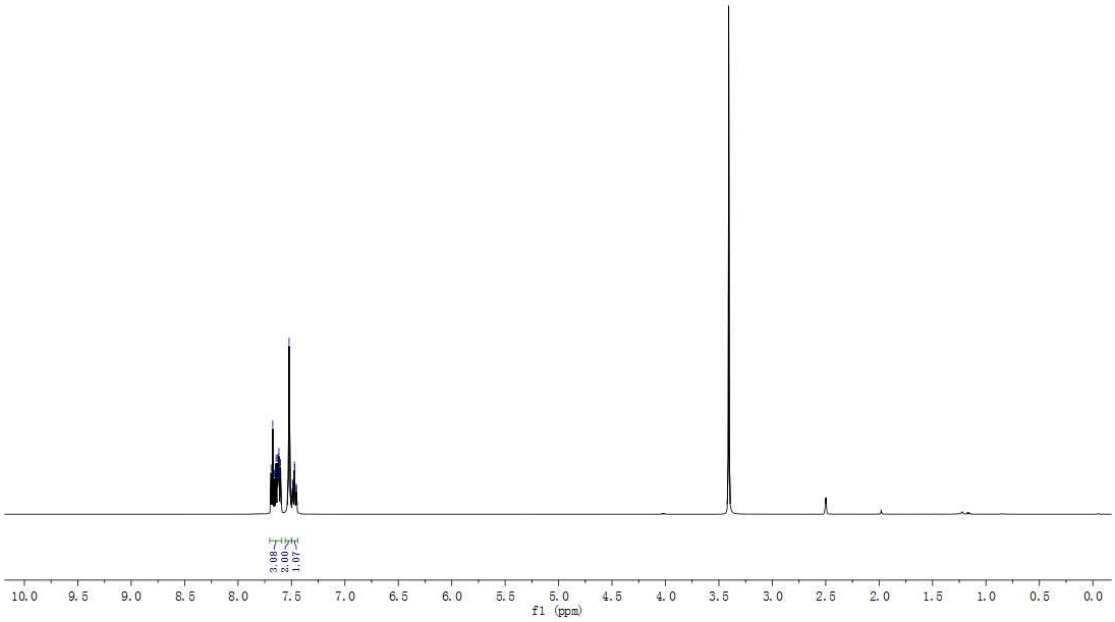

164.80
162.81
140.70
140.67
128.70
128.63
116.21
116.04

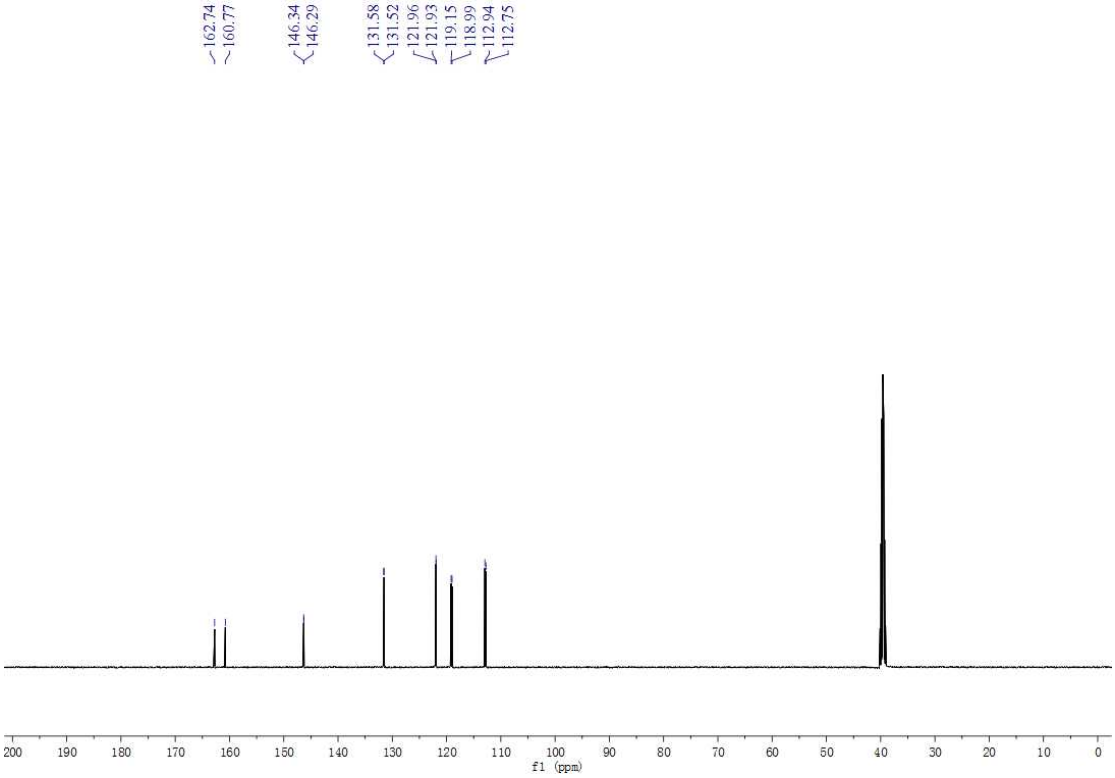

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

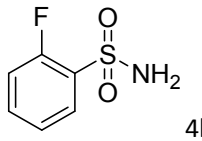

O
S
NH2
O
F

4k


7.691
7.675
7.660
7.649
7.644
7.633
7.628
7.625
7.620
7.618
7.607
7.603
7.600
7.522
7.490
7.487
7.473
7.469
7.456
7.452

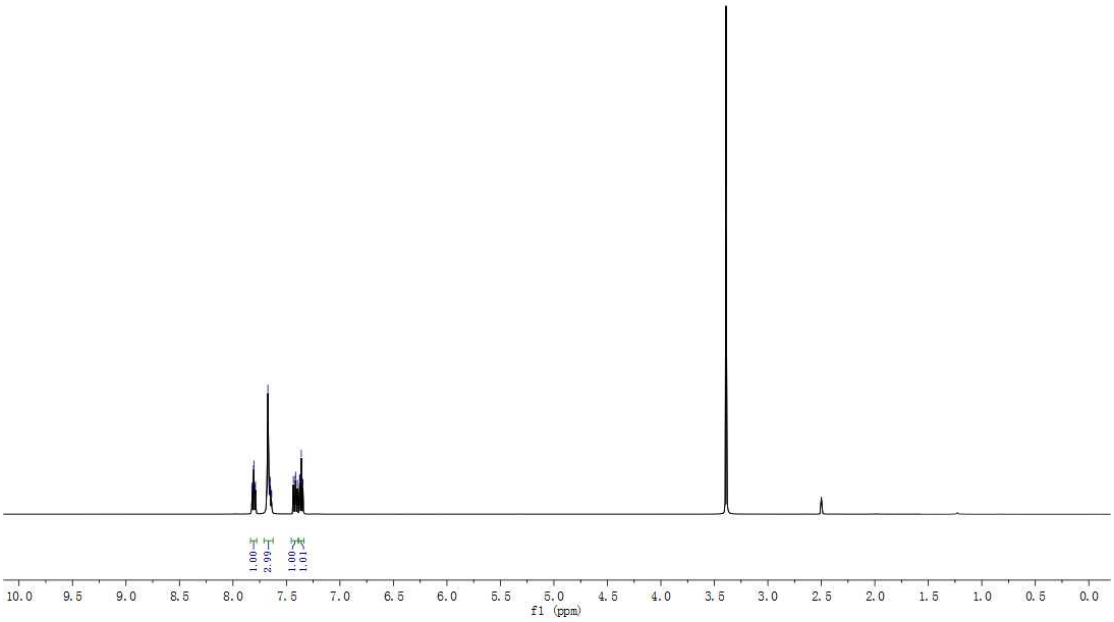

3.08
2.00
1.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

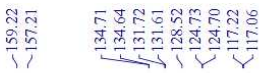

162.74
160.77
146.34
146.29
131.58
131.52
121.96
121.93
119.15
118.99
112.94
112.75
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

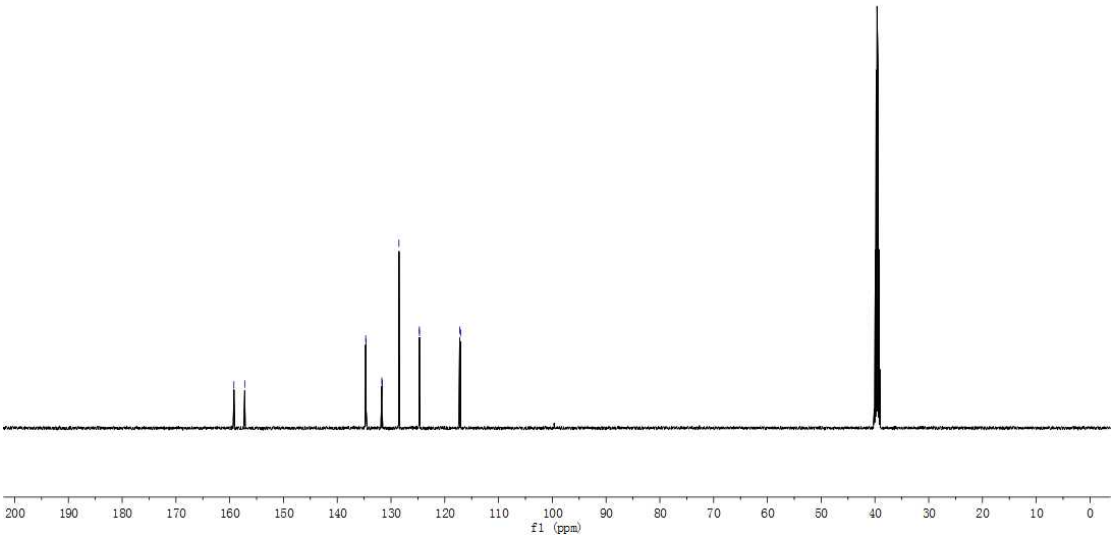

F
O
S
NH2
O

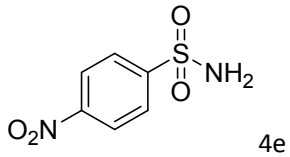
4l


7.823
7.820
7.808
7.805
7.793
7.789
7.673
7.657
7.653
7.648
7.641
7.638
7.435
7.418
7.414
7.398
7.377
7.376
7.361
7.347
7.346

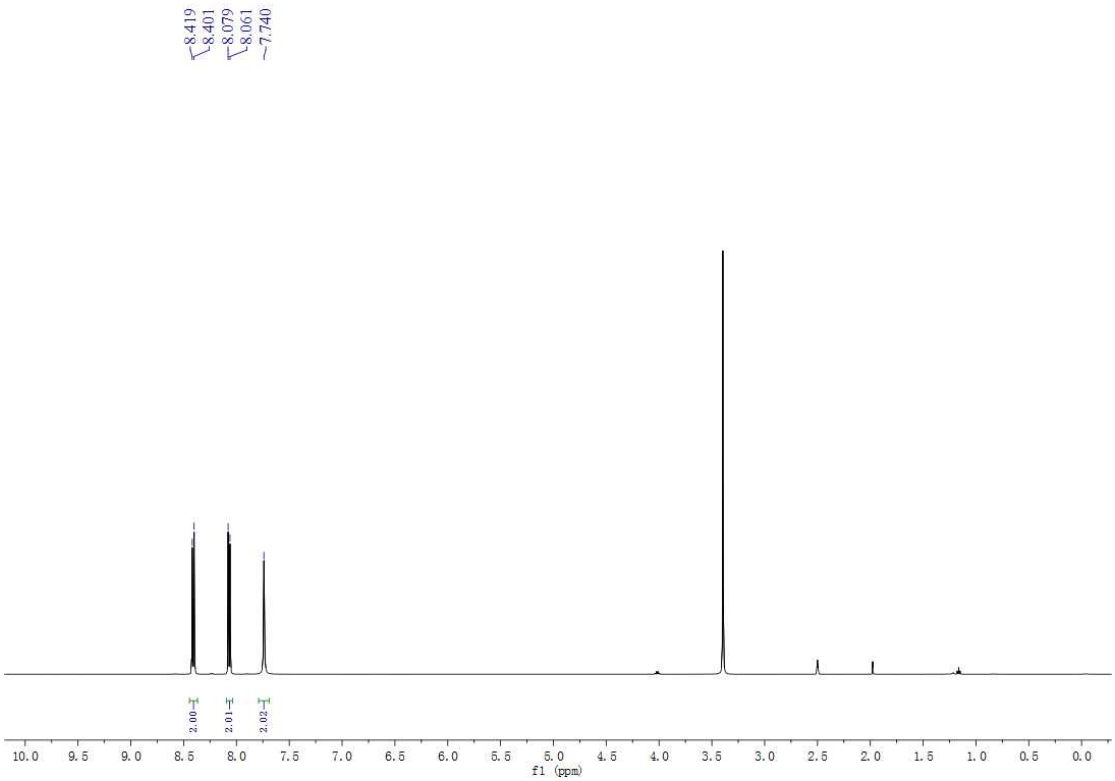

1.00
2.99
1.00
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

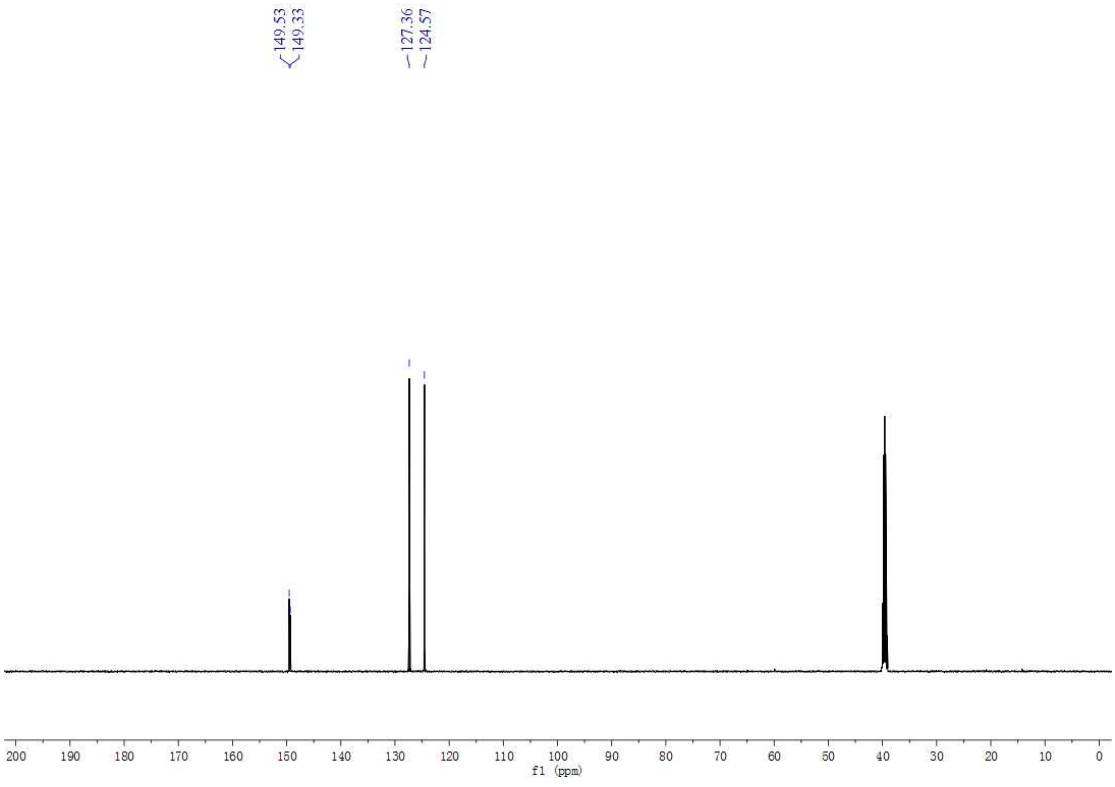

159.22
157.21
134.71
134.64
131.72
131.61
128.52
124.73
124.70
117.22
117.06

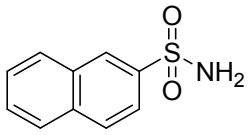

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
S
O
NH2
O2N

4e

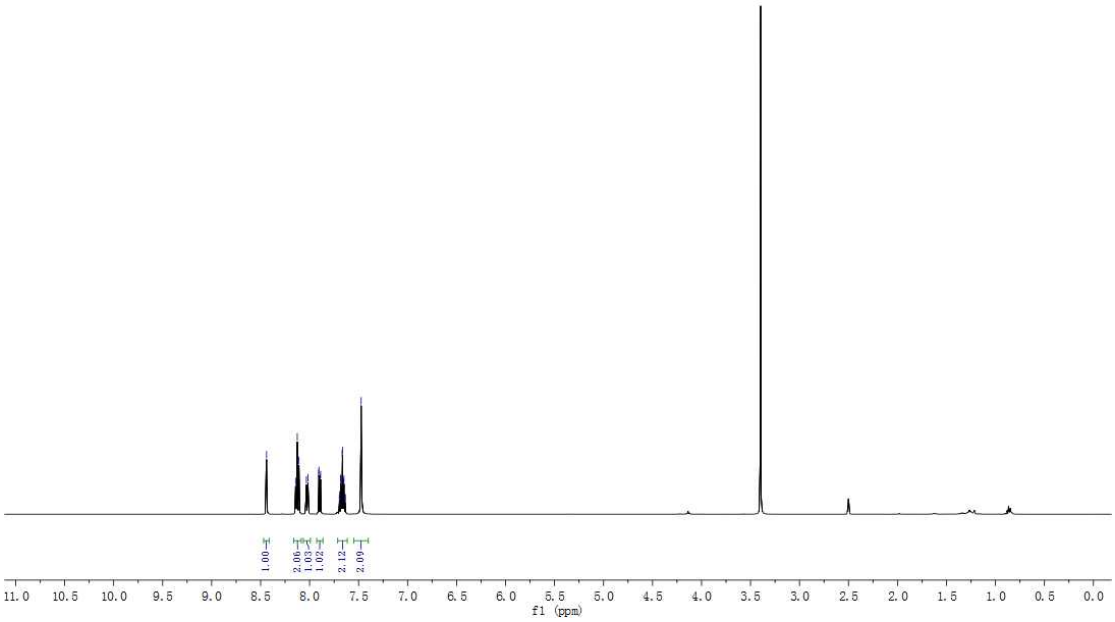

8.419
8.401
8.079
8.061
7.740
2.00
2.01
2.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


149.53
149.33
127.36
124.57
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

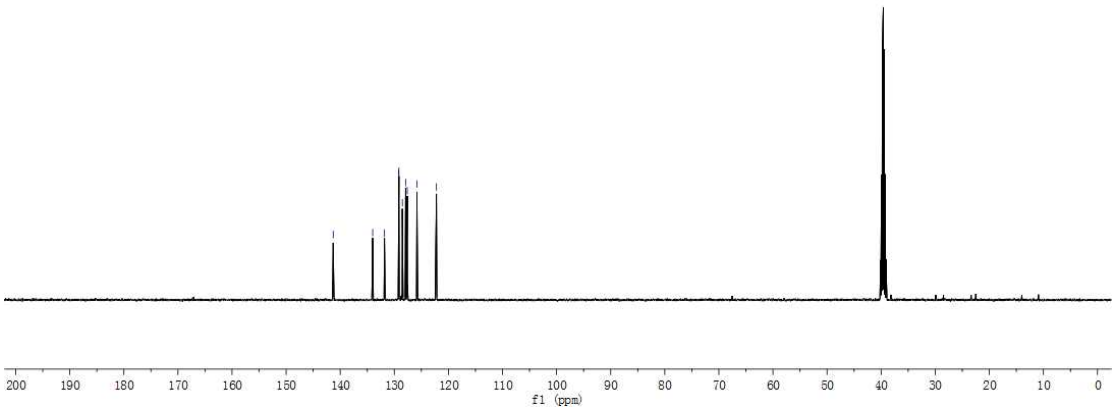


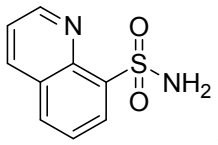
4m


8.439
8.143
8.126
8.109
8.033
8.017
7.908
7.905
7.891
7.888
7.697
7.694
7.683
7.680
7.668
7.664
7.652
7.649
7.638
7.636
7.474


1.00
2.08
2.03
1.02
2.12
2.09
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 1.5 1.0 0.5 0.0
f1 (ppm)

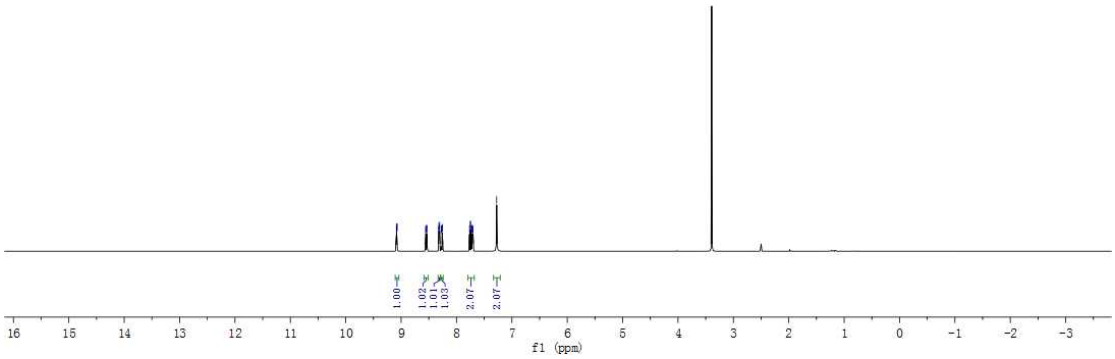

141.29
133.99
131.81
129.17
129.16
128.51
127.91
127.61
125.82
122.21


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

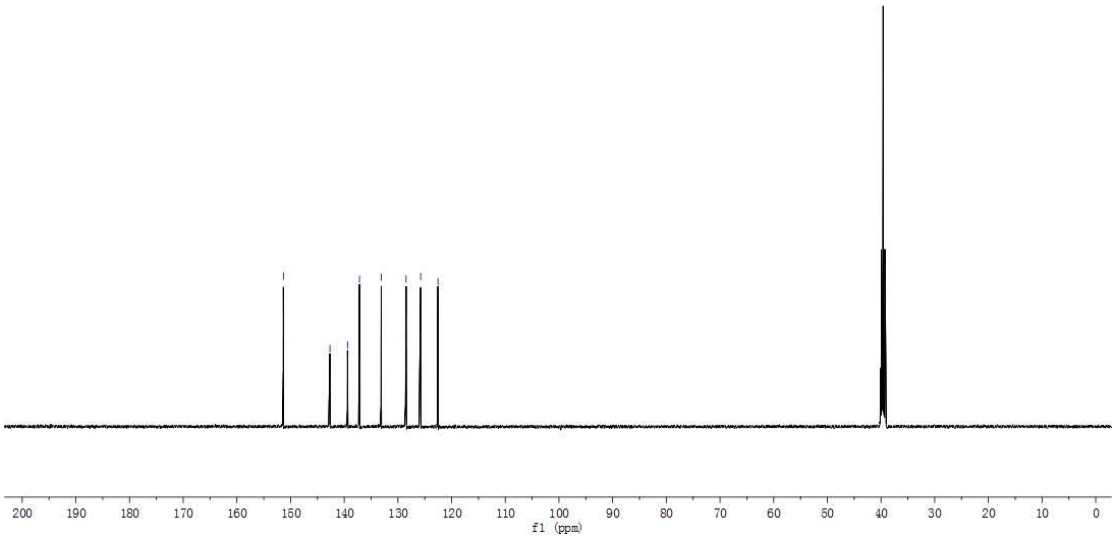

N
O
S
O
NH2

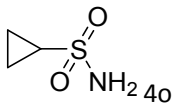
4n


9.085
9.082
9.077
9.073
8.556
8.553
8.539
8.536
8.322
8.319
8.307
8.304
8.274
8.272
8.258
8.255
7.764
7.749
7.747
7.733
7.726
7.718
7.710
7.701
7.275

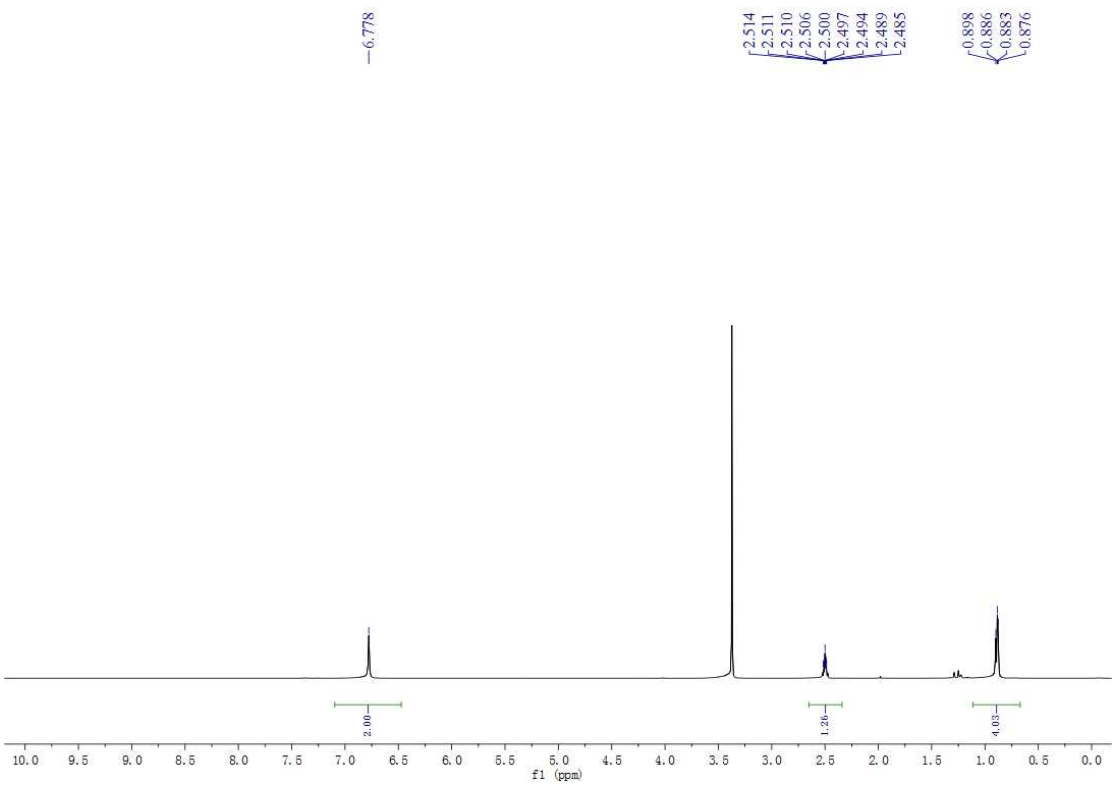

1.00
1.02
1.01
1.03
2.07
2.07
f1 (ppm)

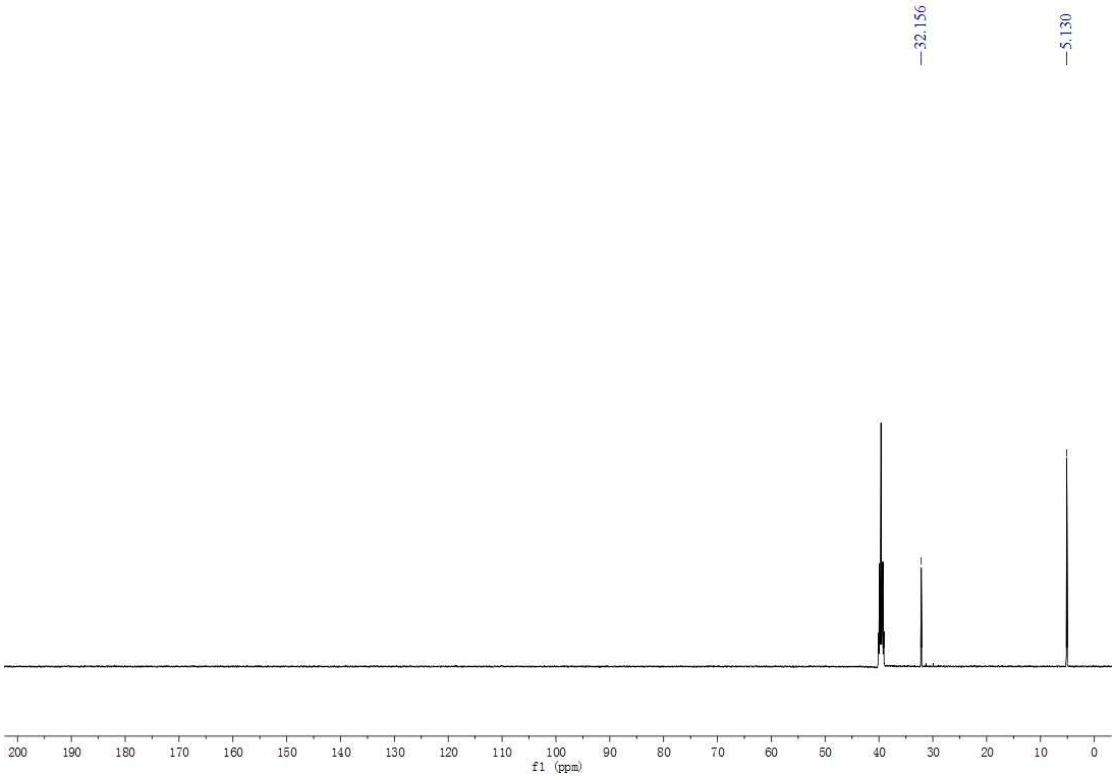

151.34
142.66
139.39
137.16
133.12
128.57
128.48
125.77
122.54

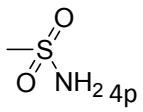

f1 (ppm)


O
S
O
NH2

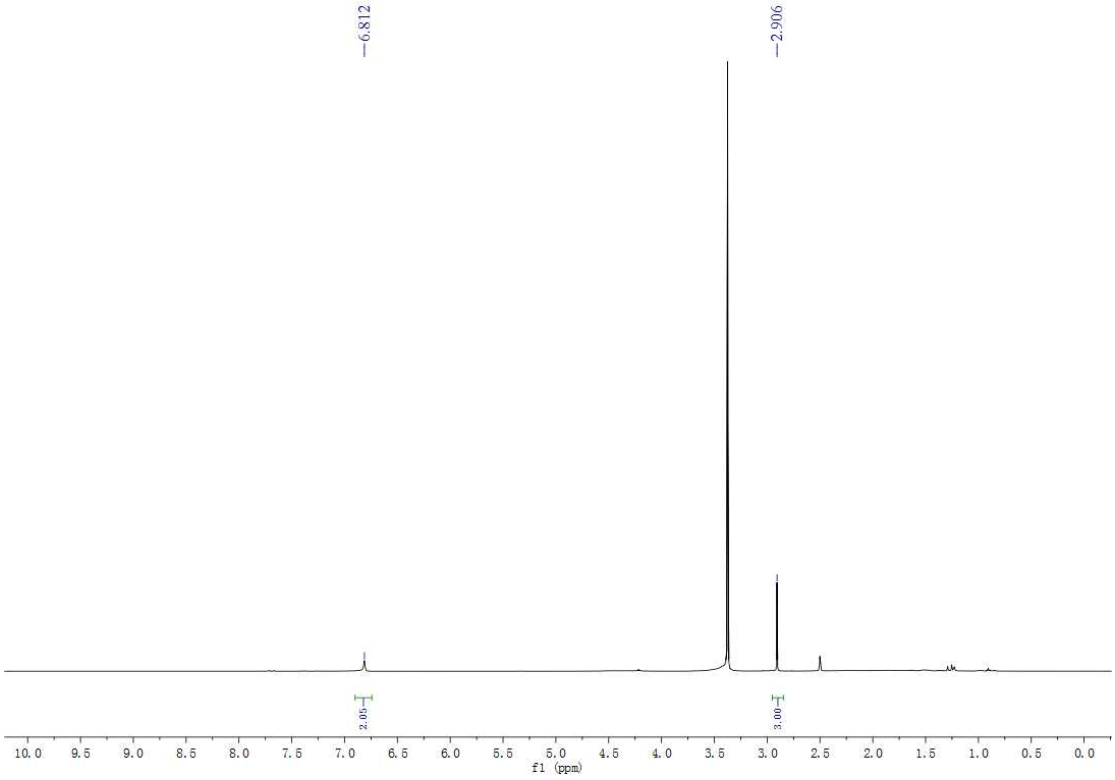
4o

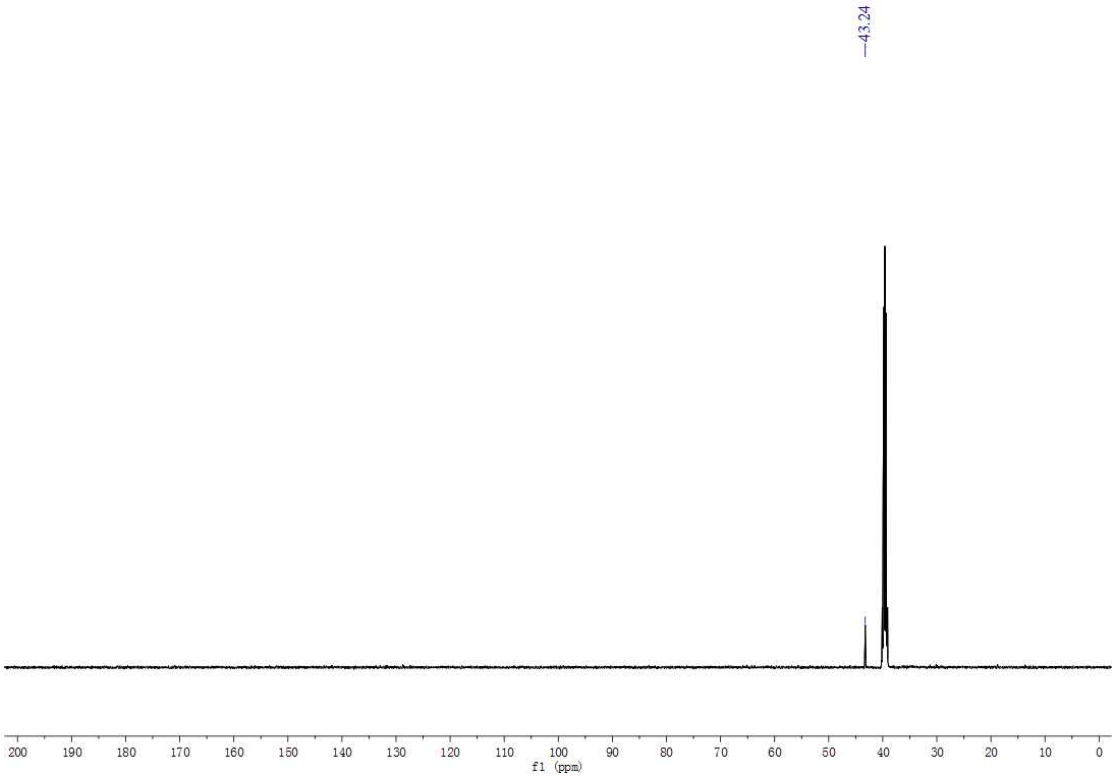

6.778
2.514
2.511
2.510
2.506
2.500
2.497
2.494
2.489
2.485
0.898
0.886
0.883
0.876
2.00
1.26
4.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


32.156
5.130
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
S
O
NH2

4p


6.812
2.906
2.05
3.00
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
f1 (ppm)


43.24
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)