Supporting Information

Interfacing Sugar-based Surfactant Micelles and Cu Nanoparticles: A Nanoreactor for C-S Coupling Reaction in Water

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Experimental Section

Chemicals and Characterization

The M2070 was purchased from Hengyu Trading (Guangzhou, China) Co., Ltd. All reagents and solvents were general reagent grade unless otherwise stated. ^{1}H NMR spectra were recorded on Bruker DRX (400 MHz) and ^{13}C NMR spectra on Bruker DRX (100 MHz) spectrometer. Melting points were determined on an X4-Data microscopic melting point apparatus and were uncorrected. Mass spectra were recorded on a Finnigan TSQ Quantum-MS instrument in the electrospray ionization (ESI) mode. X-ray diffraction (XRD) pattern was recorded with a diffractometer (Bruker D8 Advance) using Cu Ka radiation at a range of 5-85° on 2θ. X-ray photoelectron spectroscopy (XPS) measurements were carried out with an AXIS Supra by Kratos Analytical Inc. The transmission electron microscopy (TEM) was performed on JEM-2100plus at an acceleration voltage of 200 kV. High Resolution Transmission electron microscopy (HRTEM) imaging was conducted using JEM-2100F instrument. The surface tension (γ) was measured at 25.0 ± 0.1 °C by the platinum plate method using an BZY-2A automatic surface tensiometer (Shanghai Balance Instrument Factory Co., Ltd). For the React-IR experiments, the reaction spectra were recorded using a React-IR 15 from Mettler-Toledo Auto-Chem. Data manipulation was carried out using the iC IR software, version 4.3.

On a chemisorption instrument (BELCAT II, Japan) with both TCD and Mass detectors (BELMASS, Japan), H_2-TPR characterization of samples was conducted. For H_2-TPR measurement, the sample was primarily treated at 573 K for 1 h in a He stream, then cooled down to 353 K and heated in a 10% H_2/N_2 stream to 873 K to obtain the

H_2-TPR profile. The mass signals of m/z = 18 and 20 were recorded.

The synthesis of sugar-based surfactants (GluM, LacM, GluLM)

Preparation of GluM： A solution of glucose (1.8 g, 10 mmol), and amine terminated polyether M2070 (20 g, 10 mmol) in methanol (100 mL) was mechanical stirred for 24 h at room temperature. Then the final mixture was placed at -15 °C for 5 times of recrystallization, and the supernatant liquid was removed the solvent by vacuum distillation to give yellow liquid in 85% yield.

Preparation of LacM： Lactose monohydrate (10 mmol, 3.80 g) was dissolved in 60 mL of ultrapure water, and amine terminated polyether M2070 (20 g, 10 mmol) was dissolved in 100 mL of isopropanol. The two solutions were mixed and stirred at 80°C for 24 h. The final mixture was removed the solvent by vacuum distillation. Then recrystallized with ethanol at -15 °C for 5 times to give brown liquid in 80% yield.

Preparation of GluLM： To a solution of the gluconolactone (1.78 g, 10 mmol) in methanol (100 mL) was slowly added amine terminated polyether M2070 (20 g, 10 mmol). The resulting mixture was heated at 80°C for 24 h Then the final mixture was placed at -15 °C for 5 times of recrystallization, and the supernatant liquid was removed the solvent by vacuum distillation to give yellow liquid in 82% yield.

The measurement of DLS

Dynamic light scattering (DLS) was used to study the size changes of the GluM micelles after dissolving iodobenzene at 25 ^{o}C. Before measurement, 0.22 μm hydrophilic polyvinyl fluoride (PVDF) membrane was used to remove the present impurities. The size and distribution of the micelle were measured at 90^{o} by an AlV/DLS/LS-5022F laser scattering system (He-Ne laser (λ= 632.8 nm)). ALV-V3.0 software was used to analyze and process the experimental data.

General procedure of Ullmann C-S coupling reaction in water

The copper salt catalyst, aryl halide (1.0 mmol), sodium benzoesulfonate or its derivatives (1.2 mmol) were added to a micellar solution of glycosyl polyether surfactant and H_2O (10 mL). The micelle solution mixture was then heated and stirred at 100 ^{o}C for 7 h. At the end of the reaction, the organic phase was extracted with ethyl acetate and dried with anhydrous Na_2SO_4, and the crude product was obtained by vacuum concentration. Crude products were purified by silica gel column chromatography (eluent: ethyl acetate/petroleum ether) to obtain the corresponding pure products, and characterized by ^{1}H NMR and ^{13}C NMR.

Recycling of GluM/H_2O system

Once the reaction was finished, the reaction mixture was extracted with ethyl acetate to obtain the product at 50 °C. After removal of the organic phase, the substrate (aryl halide (1.0 mmol) and sodium benzoesulfonate (1.2 mmol)) were re-added to the obtained aqueous solution containing GluM and NPs Cu for the next catalytic cycle.

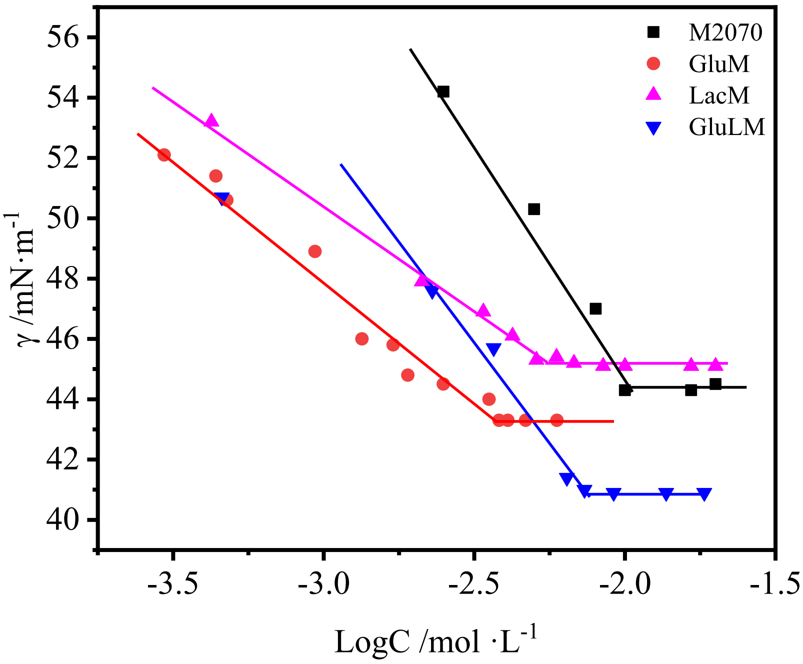


Fig. S1 Surface tension (*γ*) of sugar-based surfactants aqueous solution as a function of surfactant molarity (c) at 25°C

Table S1. CMC of four characteristic surfactants at 25°C

Surfactant	CMC/mol·L^{-1}
M2070	0.01
GluM	0.0038
LacM	0.0051
GluLM	0.0074

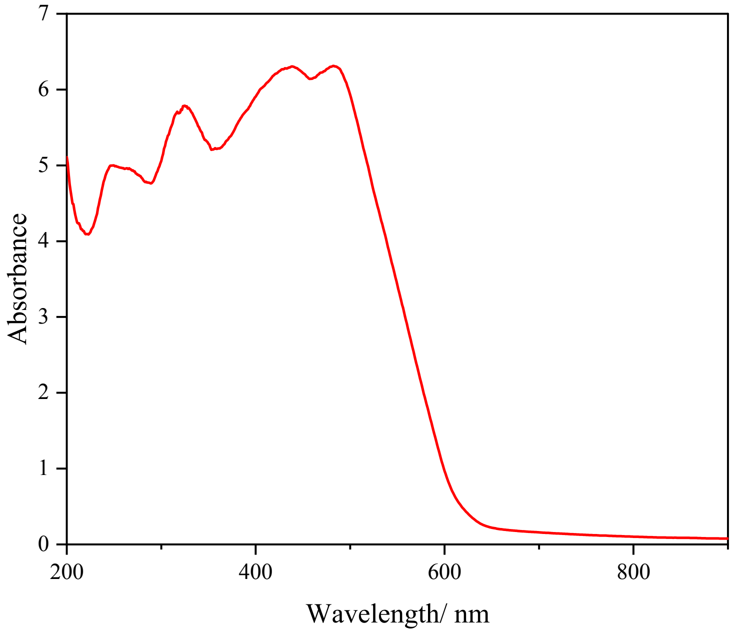


Fig.S2 DRUV absorption spectra of Cu_2O NPs

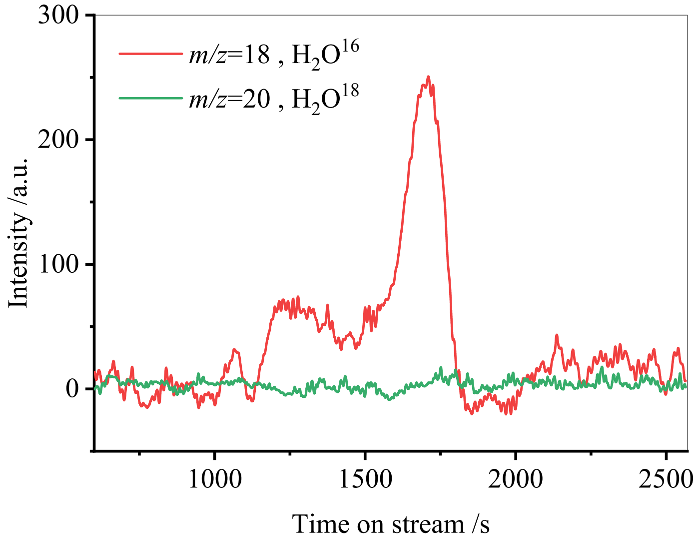


Fig.S3 The tracing of oxygen source for Cu_2O.

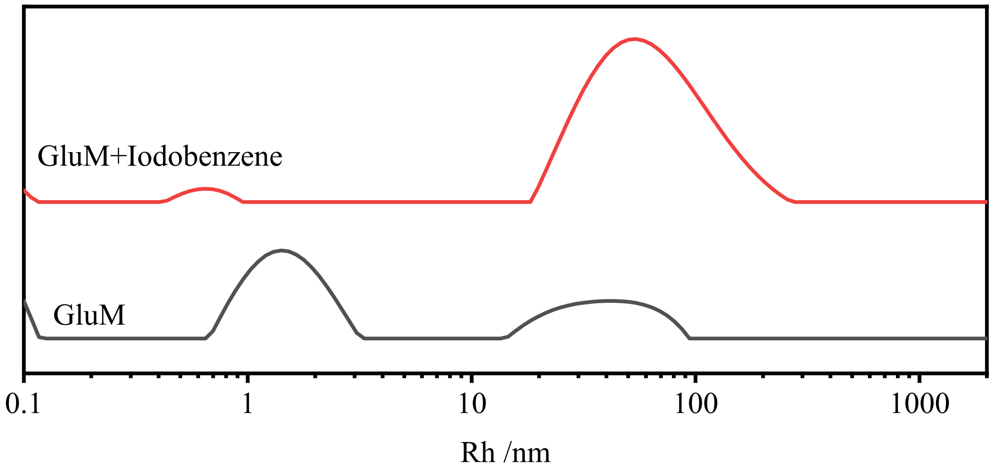


Fig. S4 The particle size distribution of GluM aqueous solution before and after adding iodobenzene at 25 °C

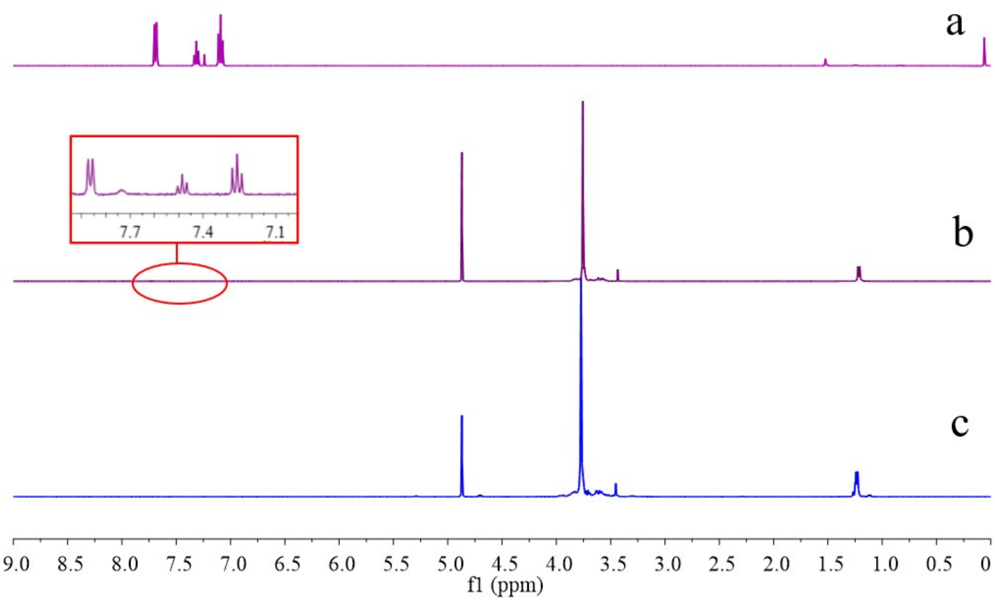


Fig. S5 ^{1}H NMR spectra of (a) Iodobenzene/$CDCl_3$, (b) GluM+Iodobenzene/D_2O, (c) GluM/D_2O

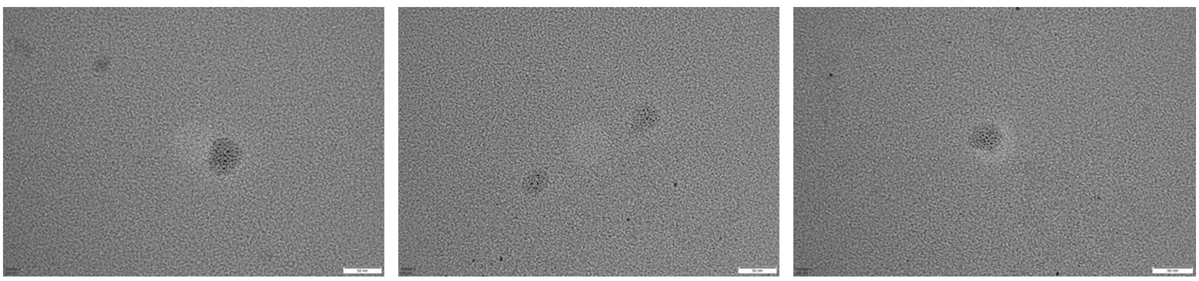
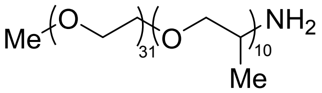
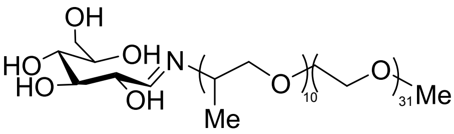


Fig. S6 TEM image of recycled GluM/water system.

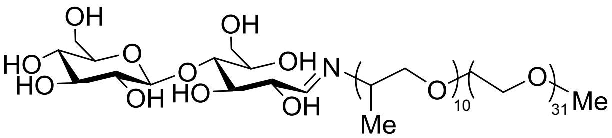
The Data of Characterization



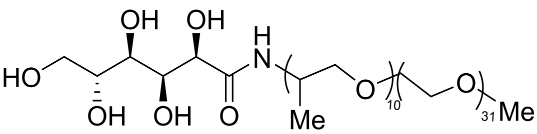
M2070. ^{1}H NMR (400 MHz, D_2O) δ 3.86 – 3.80 (m, 13H), 3.75 (s, 124H), 3.65 – 3.52 (m, 19H), 3.43 (s, 3H), 1.21 (d, J = 6.3 Hz, 30H). ^{13}C NMR (100 MHz, D_2O) δ 178.48, 178.42, 75.16, 75.03, 74.85, 74.07, 74.05, 72.60, 72.21, 71.86, 71.25, 71.04, 70.97, 70.27, 70.01, 69.92, 69.64, 69.49, 67.55, 62.68, 58.11, 41.12, 15.96, 15.87, 15.77. MS (MALDI-TOF): $[M+H]^+$ 1977.0.



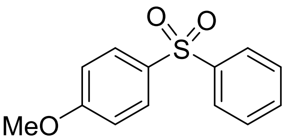
GluM. ^{1}H NMR (400 MHz, D_2O) δ 5.25 (d, J = 2.6 Hz, 1H), 4.67 (dd, J = 7.9, 0.9 Hz, 1H), 3.81 (dd, J = 8.5, 3.8 Hz, 13H), 3.74 (s, 124H), 3.58 (dd, J = 17.2, 7.0 Hz, 23H), 3.42 (s, 4H), 1.20 (d, J = 6.1 Hz, 30H), 1.09 (d, J = 6.1 Hz, 2H). FT-IR (neat) v_{max}= 3396, 2864, 1968, 1636, 1455, 1354, 1103 cm^{-1}. MS (MALDI-TOF): $[M+H]^+$ 2139.4. Anal. Found: C, 54.81; H, 8.96; N, 0.94.



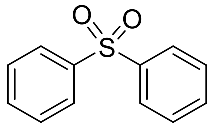
LacM. ^{1}H NMR (400 MHz, DMSO) δ 3.56 – 3.53 (m, 16H), 3.52 – 3.49 (m, 124H), 3.48 – 3.46 (m, 10H), 3.44 (d, J = 7.0 Hz, 8H), 3.38 (d, J = 5.8 Hz`, 6H), 3.31 (d, J = 4.8 Hz, 6H), 3.29 (d, J = 4.8 Hz, 4H), 3.24 (s, 2H), 1.05 – 1.03 (m, 30H), 0.96 (d, J = 6.5 Hz, 2H). FT-IR (neat) v_{max}= 3396, 2864, 1968, 1636, 1455, 1354, 1103 cm^{-1}. MS (MALDI-TOF): $[M+H]^+$ 2301.1. Anal. Found: C, 55.01; H, 9.42; N, 0.85.



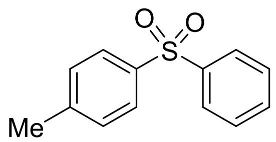
GluLM. ^{1}H NMR (400 MHz, DMSO) δ 3.55 (dd, J = 5.4, 3.5 Hz, 16H), 3.50 (d, J = 6.5 Hz, 124H), 3.48 – 3.46 (m, 9H), 3.40 (d, J = 5.8 Hz, 4H), 3.38 (d, J = 5.7 Hz, 4H), 3.31 (d, J = 4.6 Hz, 10H), 3.24 (s, 2H), 1.04 (d, J = 6.3 Hz, 30H). FT-IR (neat) v_{max}= 3396, 2864, 1978, 1787, 1656, 1653, 1445, 1063 cm^{-1}. MS (MALDI-TOF): $[M+H]^+$ 2155.2. Anal. Found: C, 55.74; H, 9.48; N, 0.87.



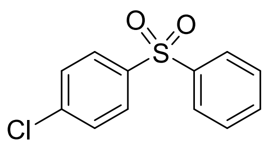
4-Methoxyphenyl phenyl sulfone 3a. White solid, m.p.: 91-92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, J = 14.2, 7.6 Hz, 4H), 7.44 (dd, J = 14.8, 7.0 Hz, 3H), 6.89 (d, J = 7.6 Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 141.4, 132.1, 131.8, 128.9, 128.2, 126.3, 113.5, 54.6.



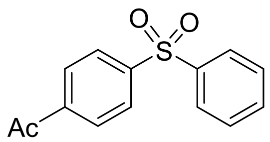
Diphenyl sulphone 3b. White solid, m.p.:123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.6 Hz, 4H), 7.58 (t, *J* = 7.4 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 133.2, 129.3, 127.7.



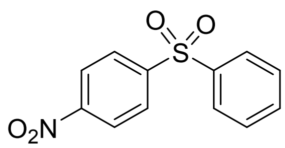
Phenyltolyl sulfone 3c. White solid, m.p.: 125-126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2 Hz, 2H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 7.0 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 140.9, 137.6, 131.9, 128.9, 128.2, 126.7, 126.5, 20.6.



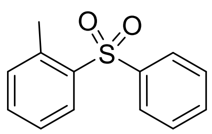
4-Chlorophenyl phenyl sulfone 3d. White solid, m.p.: 93-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.4 Hz, 2H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.55 – 7.44 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.1, 140.1, 139.9, 133.5, 129.6, 129.4, 129.1, 127.6.



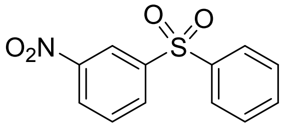
4-Acetylphenyl phenyl sulfone 3e. White solid, m.p.: 130-131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 8.03 (m, 4H), 8.01 – 7.94 (m, 2H), 7.62 (dd, *J* = 8.0, 6.4 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 2H), 2.65 (s, 3H). ^{13}C NMR (100 MHz, CDCl3) δ 196.8, 145.4, 140.8, 140.4, 133.7, 129.5, 129.1, 127.9, 127.8, 26.9.



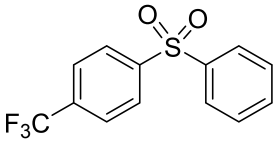
4-Nitrophenyl phenyl sulfone 3f. Yellow solid, m.p.: 141-143 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 8.8 Hz, 2H), 8.14 (d, *J* = 8.8 Hz, 2H), 7.98 (d, *J* = 7.6 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.4, 147.4, 140.0, 134.2, 129.7, 128.9, 128.0, 124.5.



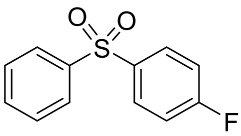
1-methoxy-2-(phenylsulfonyl)benzene 3g. White solid, m.p.: 74-76 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.93 – 7.86 (m, 2H), 7.59 (ddd, *J* = 6.6, 3.8, 1.2 Hz, 1H), 7.55 – 7.48 (m, 3H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.26 (d, *J* = 7.6 Hz, 1H), 2.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.4, 138.9, 138.0, 133.6, 132.9, 132.7, 129.5, 129.0, 127.7, 126.5, 20.2.



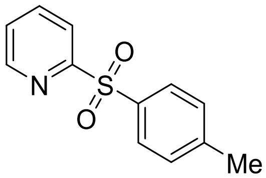
3-Nitro-(phenylsulfonyl)benzene 3h. Yellow solid, m.p.: 78-80 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (s, 1H), 8.35 (d, *J* = 8.2 Hz, 1H), 8.22 (d, *J* = 7.6 Hz, 1H), 7.93 (d, *J* = 7.4 Hz, 2H), 7.67 (t, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.49 (dd, *J* = 15.2, 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 142.9, 139.1, 133.1, 132.1, 129.7, 128.7, 126.9, 126.7, 121.9.



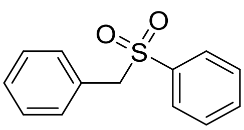
1-(Phenylsulfonyl)-4-(trifluoromethyl)benzene 3i. White solid, m.p.: 91-92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.2 Hz, 2H), 8.04 – 7.94 (m, 2H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.69 – 7.60 (m, 1H), 7.56 (dd, *J* = 10.4, 4.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 140.6, 134.6 (J = 160 Hz), 129.6, 128.2, 127.9, 126.5 (J = 8.5 Hz), 124.5, 121.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ 63.21.



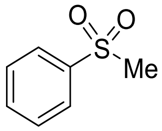
4-Fluorophenyl phenyl sulfone 3j. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.88 (m, 4H), 7.58 – 7.46 (m, 3H), 7.18 – 7.12 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.90, 164.36, 141.67, 137.91, 137.88, 133.51, 130.72, 130.63, 129.57, 127.76, 116.90, 116.67, 77.23. ^{19}F NMR (376 MHz, $CDCl_3$) δ 104.21.



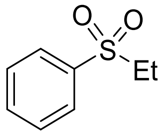
2-(4-methylphenyl)sulfonylpyridine 3k. ^{1}H NMR (400 MHz, DMSO) δ 8.68 (ddd, *J* = 4.7, 1.6, 0.8 Hz, 1H), 8.20 (dt, *J* = 7.9, 1.0 Hz, 1H), 8.12 (td, *J* = 7.7, 1.7 Hz, 1H), 7.89 – 7.82 (m, 2H), 7.68 – 7.62 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 158.12, 150.52, 144.85, 139.08, 135.59, 129.97, 128.50, 127.66, 121.78, 21.04.



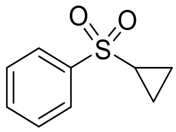
Benzyl phenyl sulfone 3l. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.45 (m, 3H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.18 (dt, *J* = 14.5, 7.1 Hz, 1H), 6.99 (d, *J* = 7.1 Hz, 1H), 4.22 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.99, 133.88, 130.98, 128.87, 128.27, 63.03.



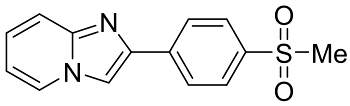
1-(Methylsulfonyl)benzene 3m. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.89 (m, 2H), 7.63 (d, J = 7.5 Hz, 1H), 7.56 (t, J = 7.7 Hz, 2H), 3.04 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.65, 133.68, 129.36, 127.28, 44.45.



(Ethylsulfonyl)benzene 3n. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.83 (m, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.55 (t, J = 7.5 Hz, 2H), 3.10 (q, J = 7.4 Hz, 2H), 1.31 – 1.25 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.69 , 133.88 , 129.46 , 128.42, 50.79, 7.65.



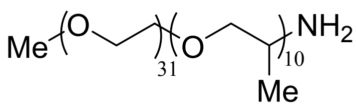
(Cyclopropylsulfonyl)benzene 3o. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.80 (m, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.5 Hz, 2H), 2.42 (tt, J = 8.0, 4.8 Hz, 1H), 1.28 (qd, J = 5.9, 1.2 Hz, 2H), 0.98 (qd, J = 5.9, 1.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.67, 133.41, 129.26, 127.51, 32.89, 5.97.

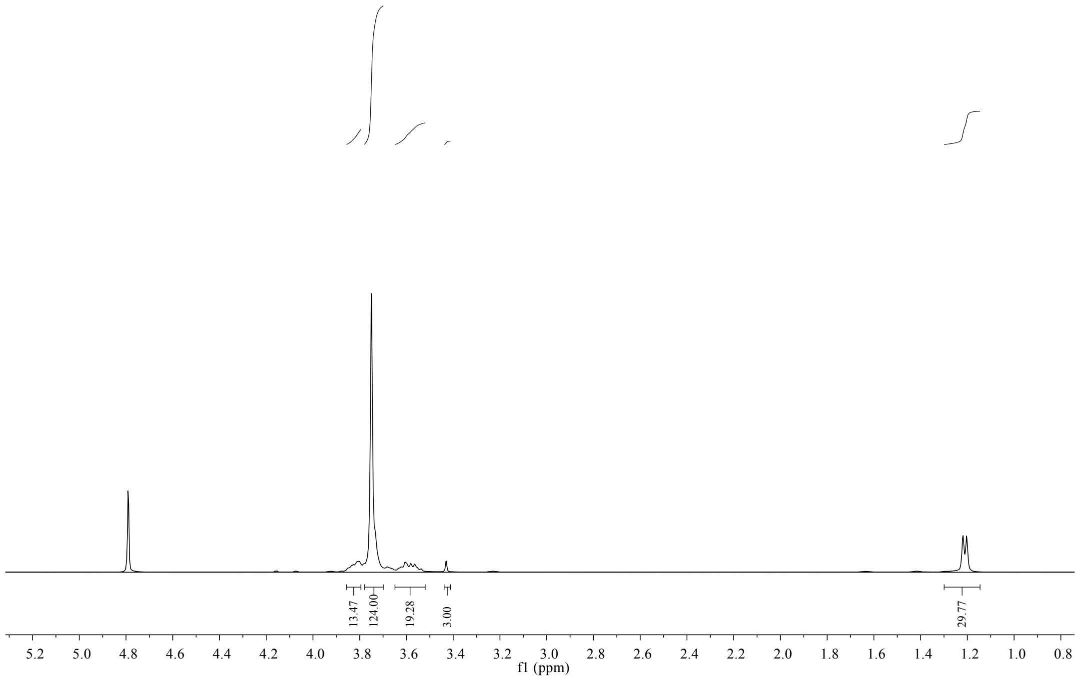


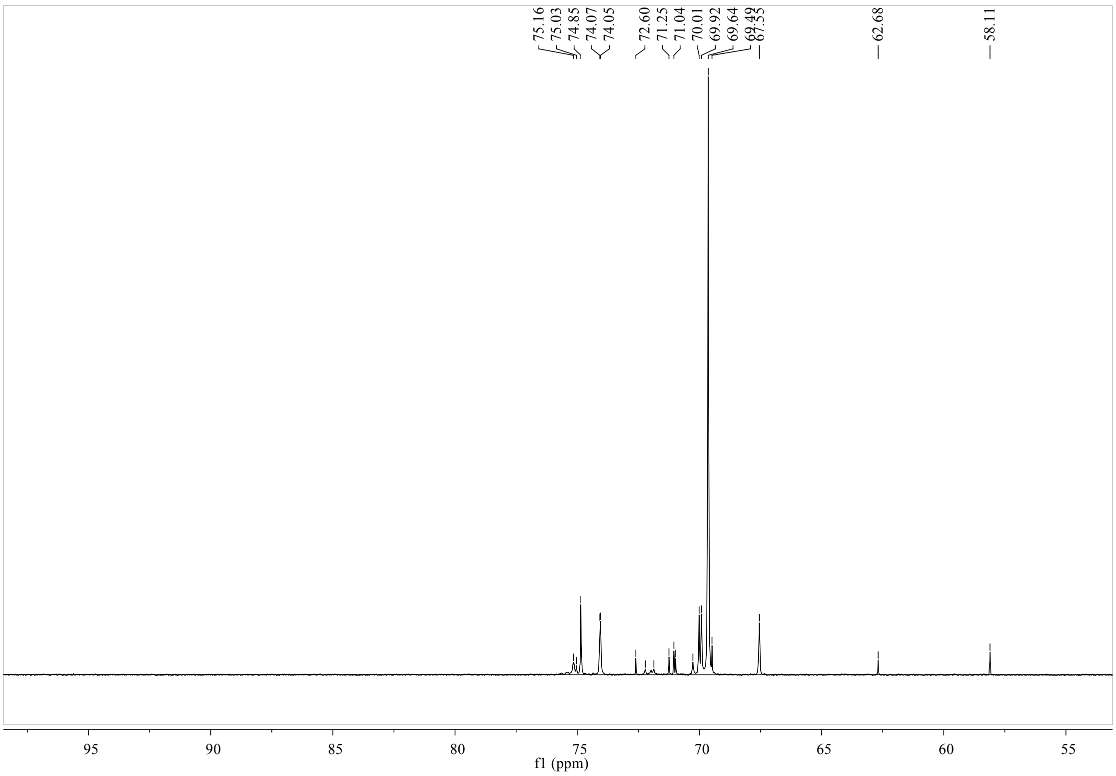
Zolimidine 3p[1]. Yellowish white solid ;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, J = 6.7, 5.5 Hz, 3H), 7.96 (d, J = 8.4 Hz, 3H), 7.63 (d, J = 9.6 Hz, 1H), 7.20 (dd, J = 10.3, 6.8 Hz, 1H), 6.81 (t, J = 7.3 Hz, 1H), 3.06 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.47, 144.07, 139.78, 128.41, 127.12, 126.36, 126.07, 118.37, 113.61, 110.18, 45.12.

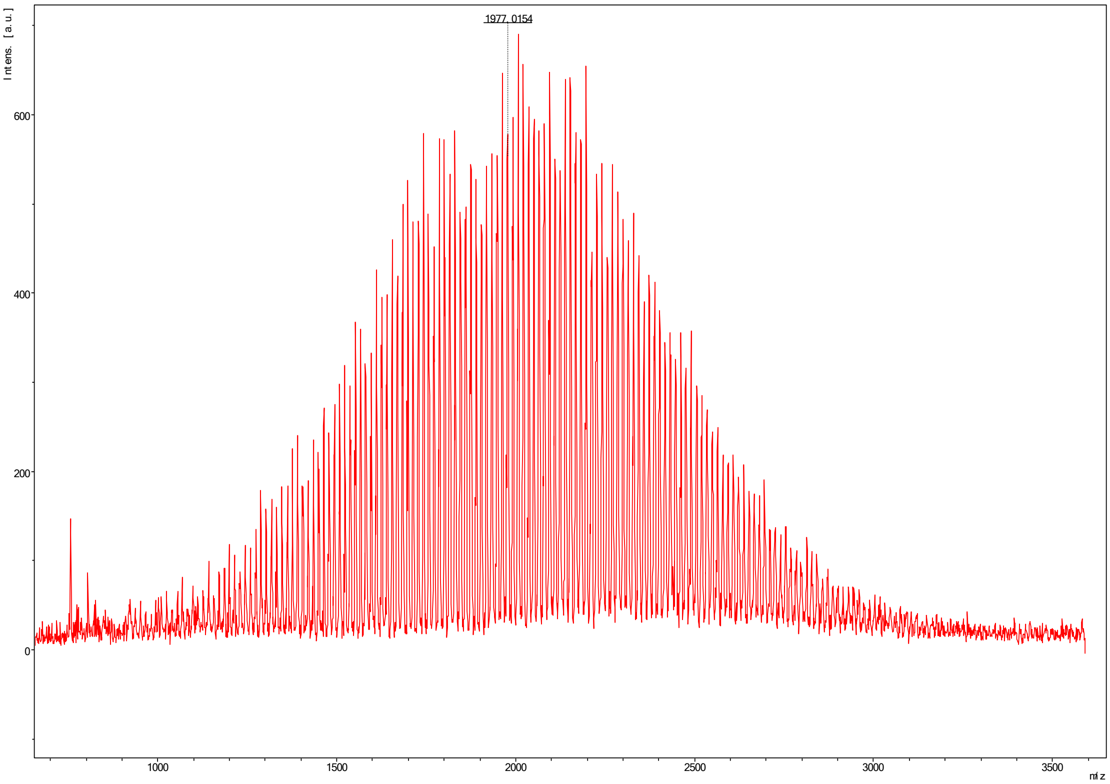
The Spectra

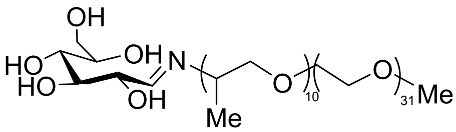
M2070

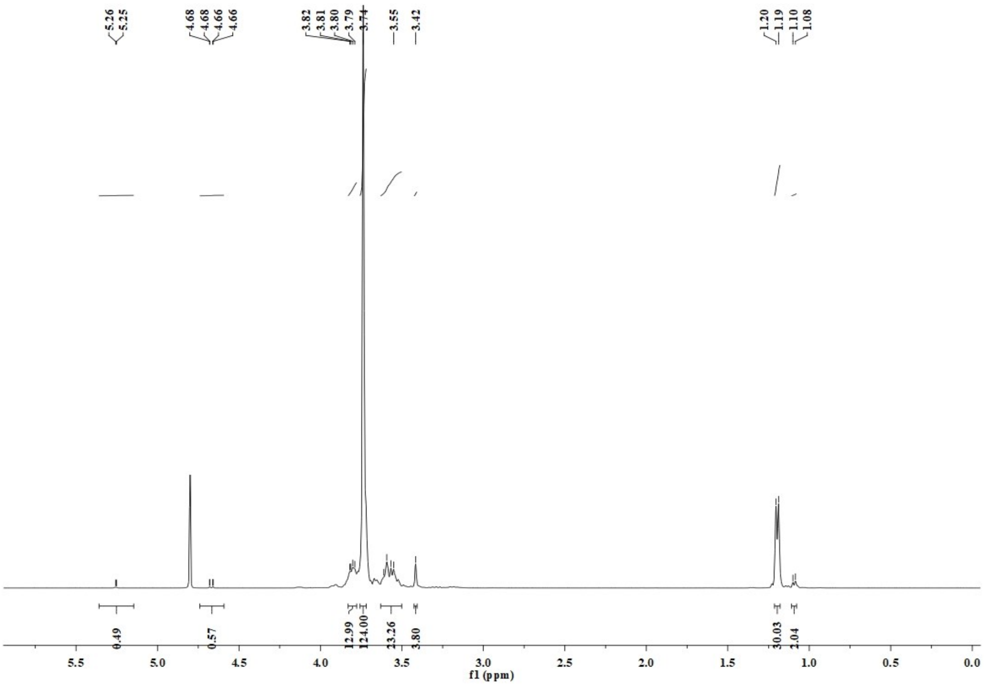


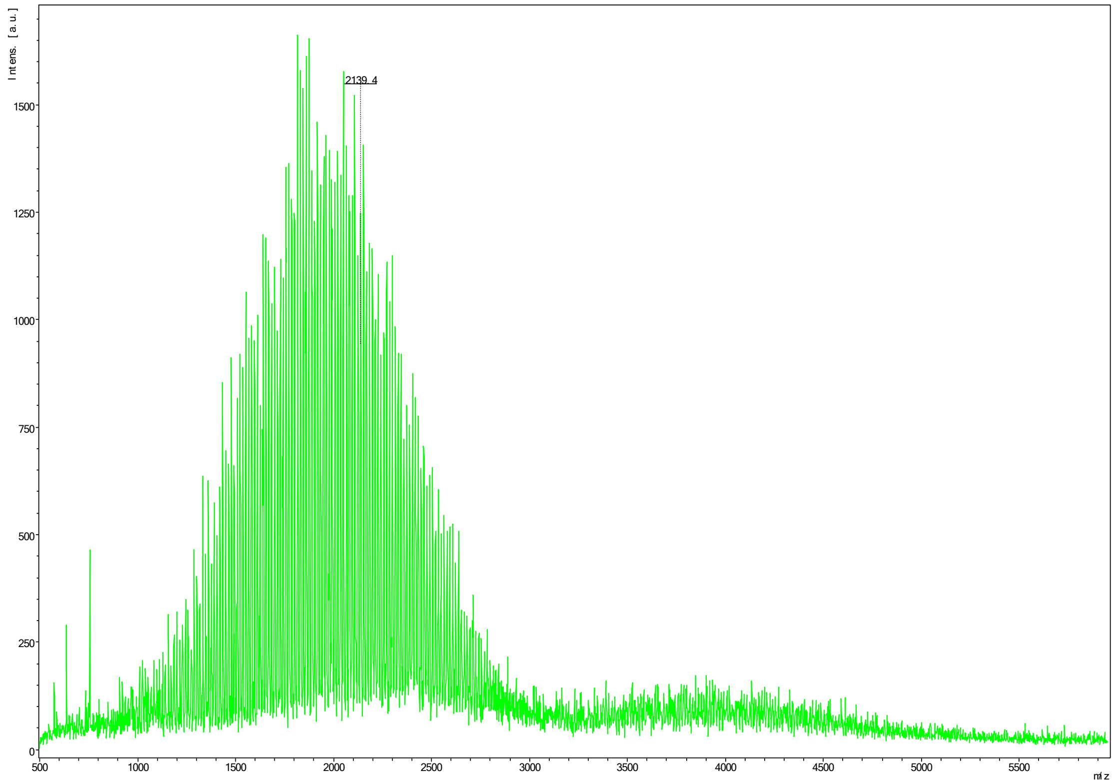




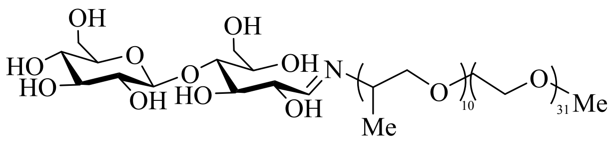

1977.0154
Intens. [a.u.]
600
400
200
0
1000
1500
2000
2500
3000
3500
m/z

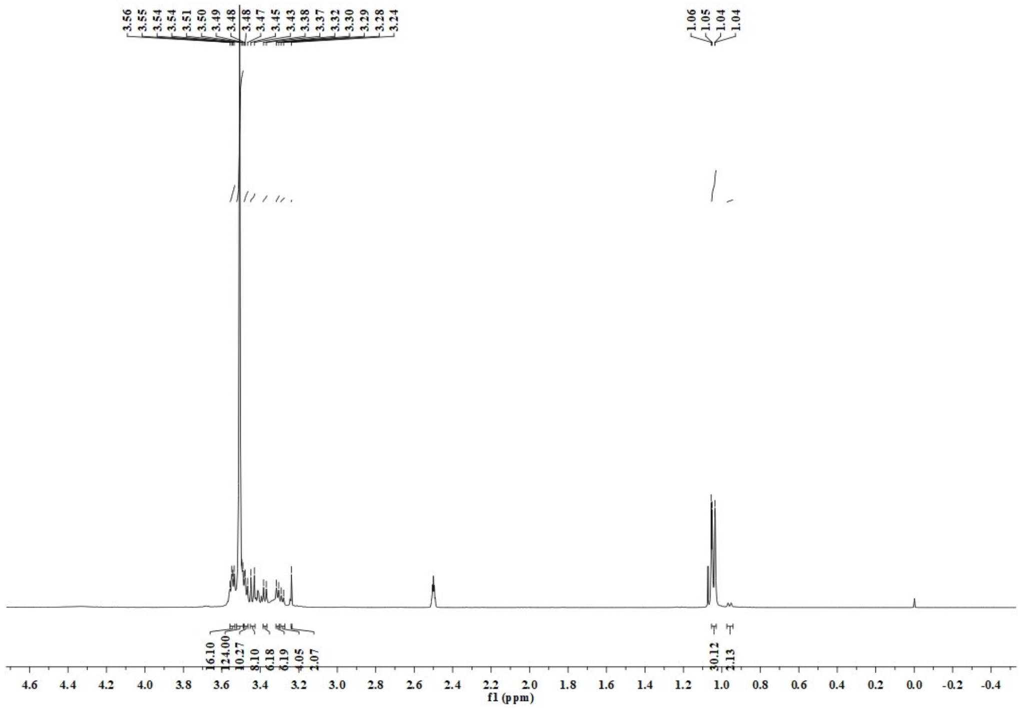


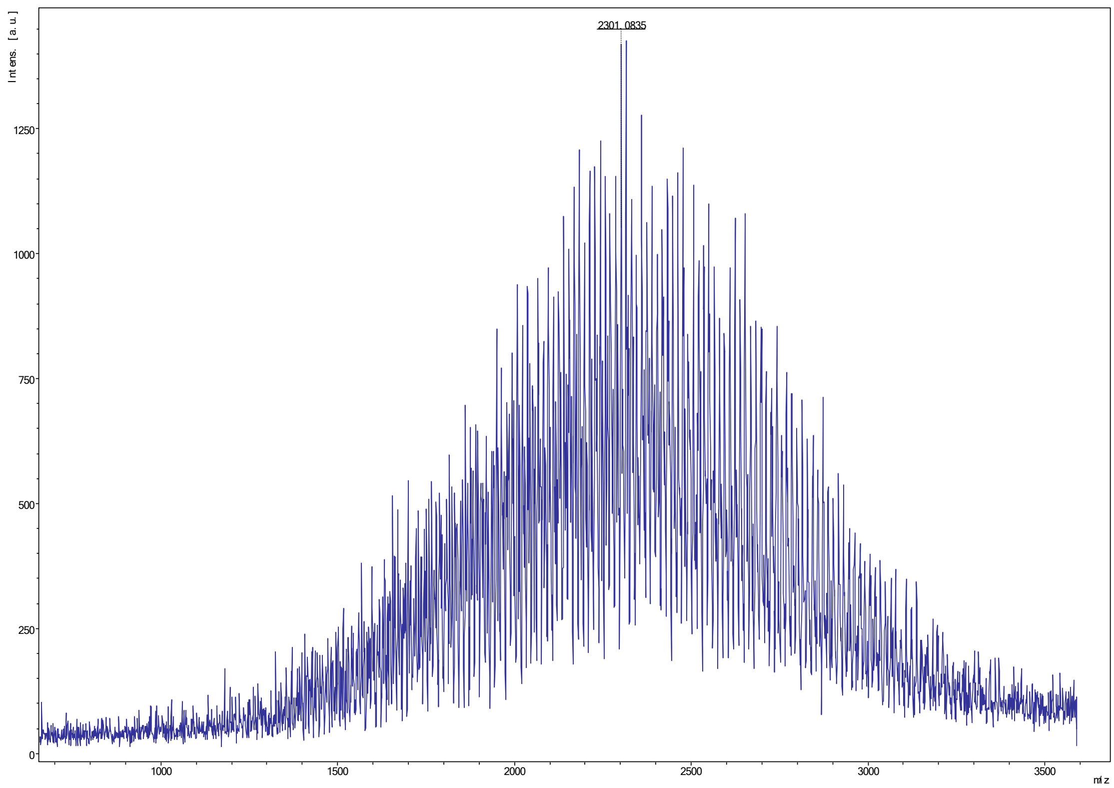

5.26
5.25
4.68
4.68
4.66
4.66
3.82
3.81
3.80
3.79
3.74
3.55
3.42
1.20
1.19
1.10
1.08
0.49
0.57
12.99
124.00
23.26
3.80
30.03
2.04
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

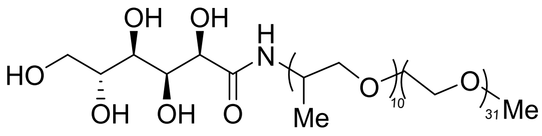


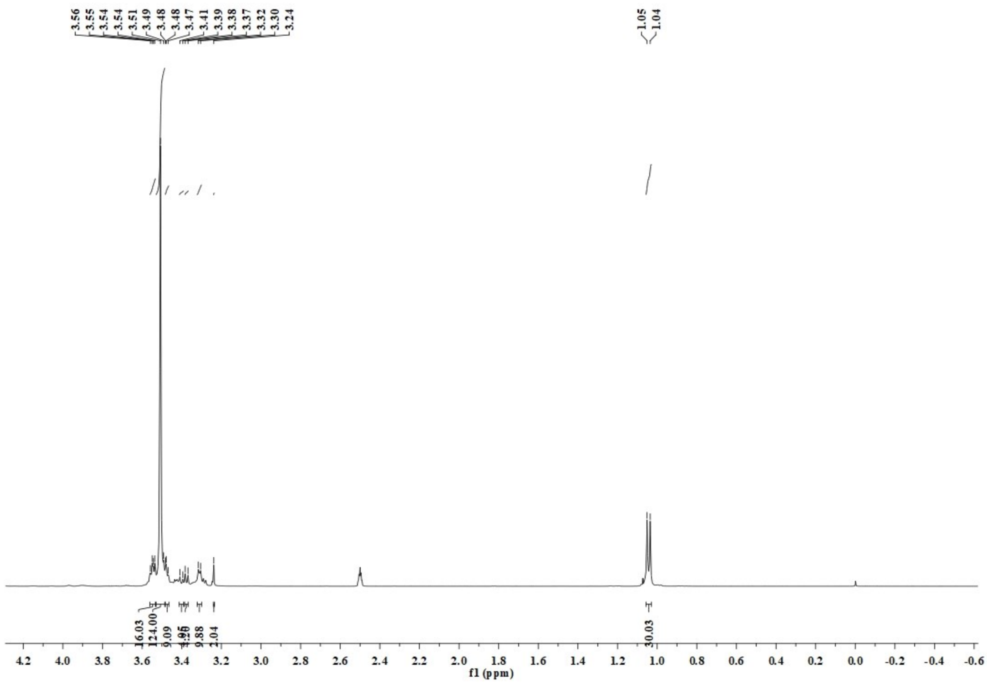
Intens. [a.u.]
2139.4
m/z

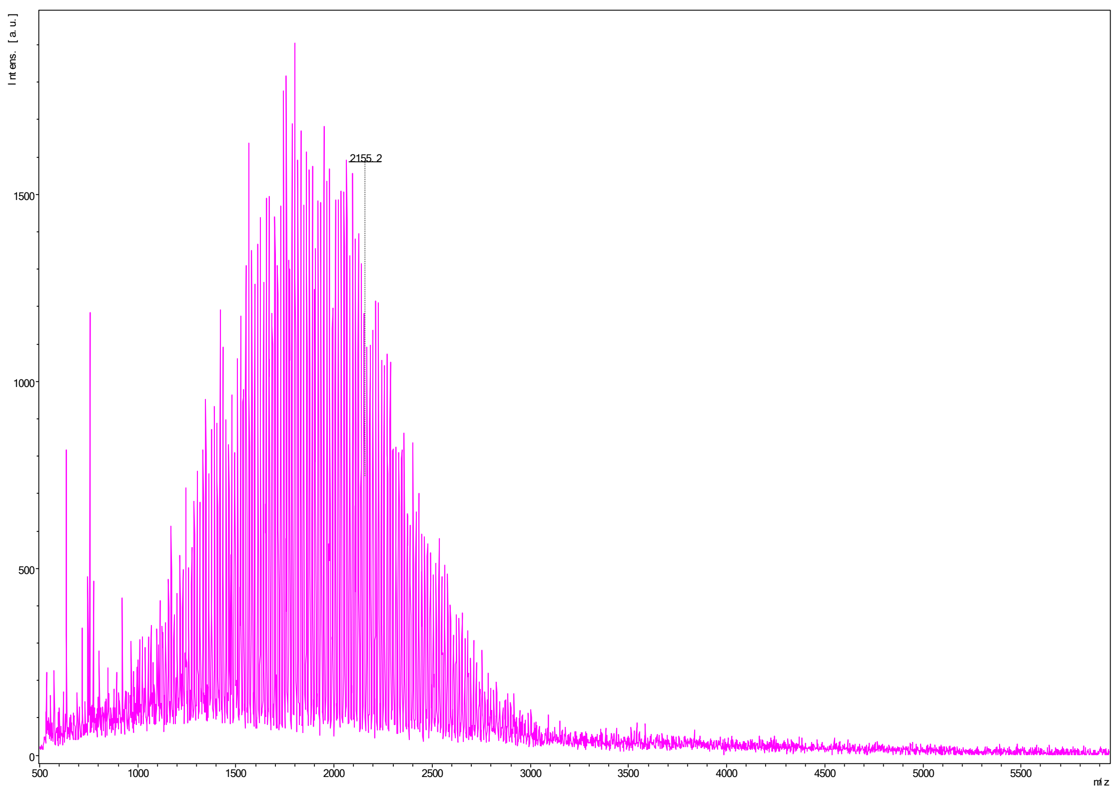


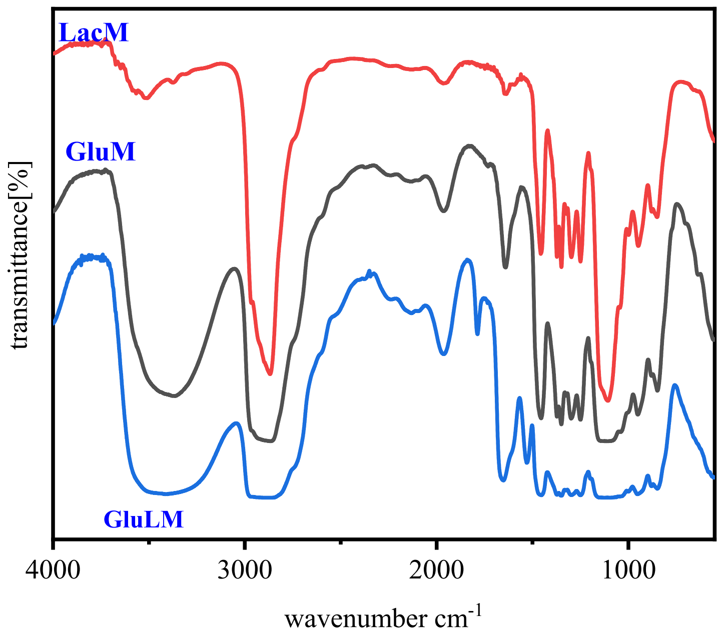

3.56 3.55 3.54 3.54 3.51 3.50 3.49 3.48 3.48 3.47 3.45 3.43 3.38 3.37 3.32 3.30 3.29 3.28 3.24
1.06 1.05 1.04 1.04
16.10 124.00 10.27 8.10 6.18 6.19 4.05 2.07
30.12 2.13
4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 0.0 -0.2 -0.4
f1 (ppm)


Intens. [a.u.]
2301.0835
1250
1000
750
500
250
0
1000
1500
2000
2500
3000
3500
m/z

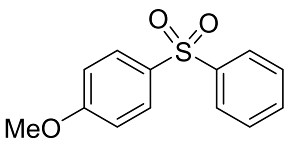


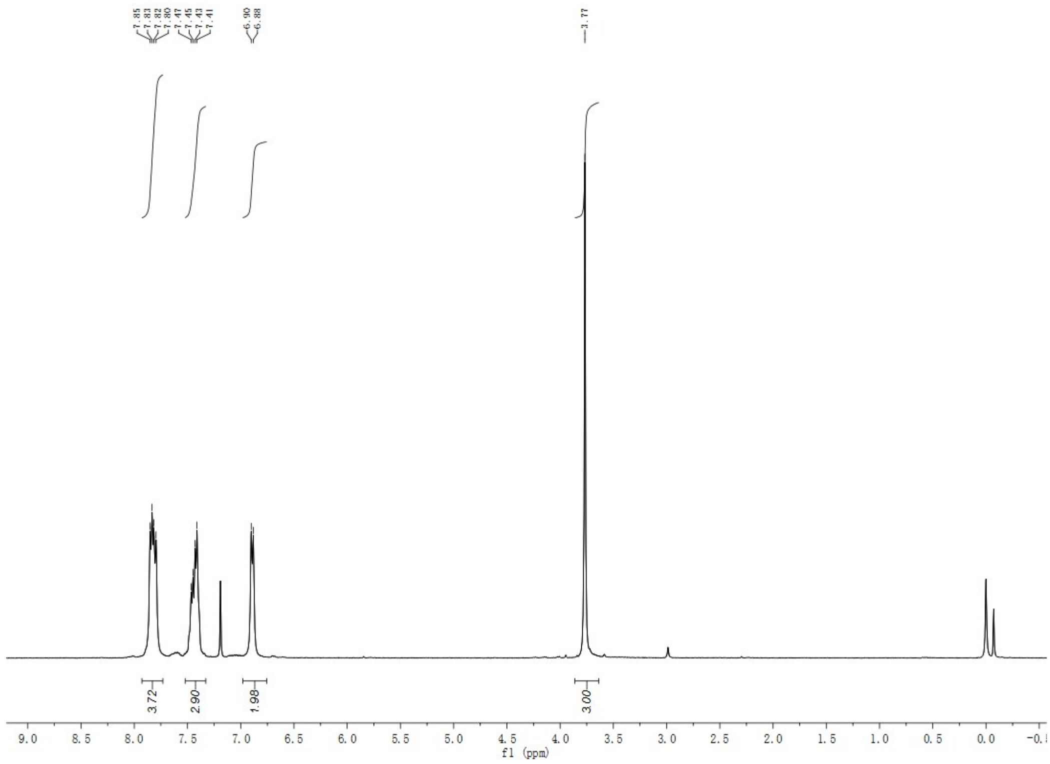


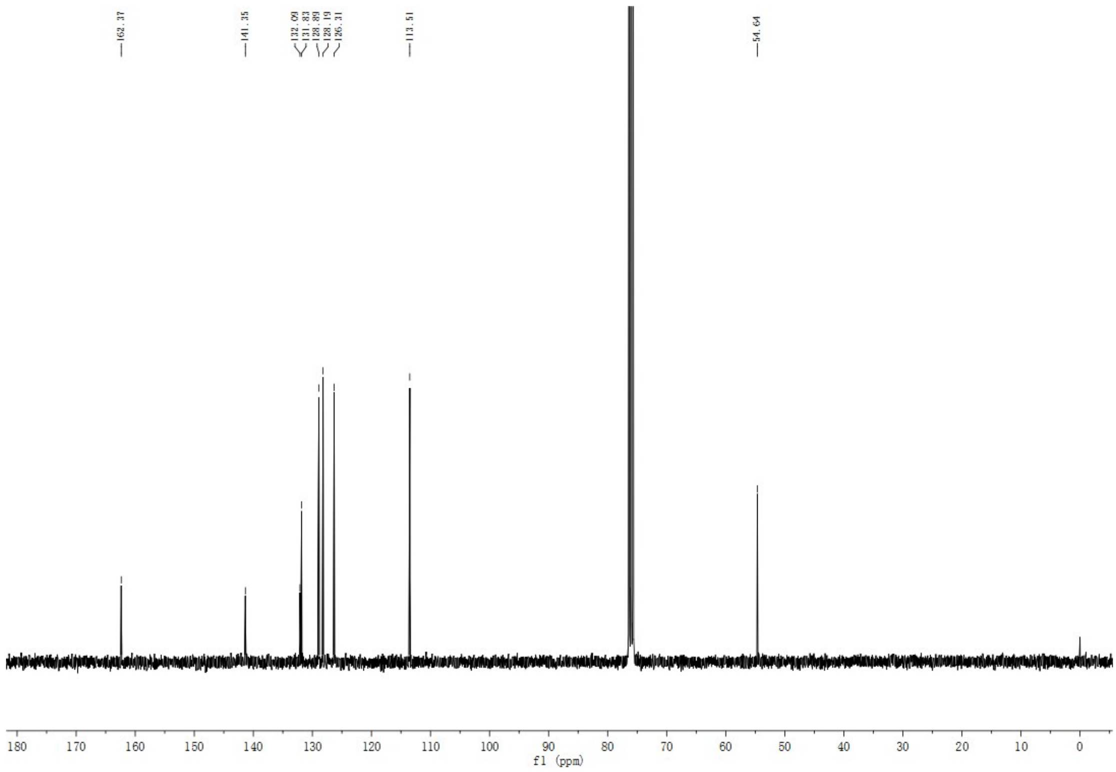

Intens. [a.u.]
2155.2
m/z

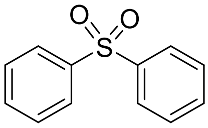


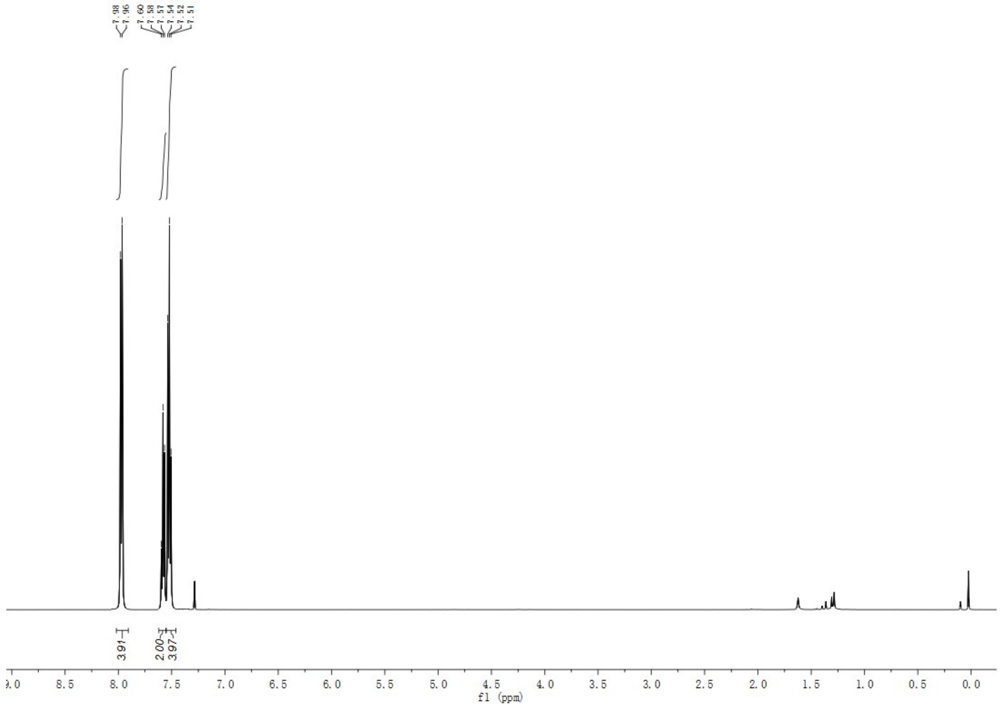
LacM
GluM
GluLM
transmittance[%]
4000
3000
2000
1000
wavenumber cm-1

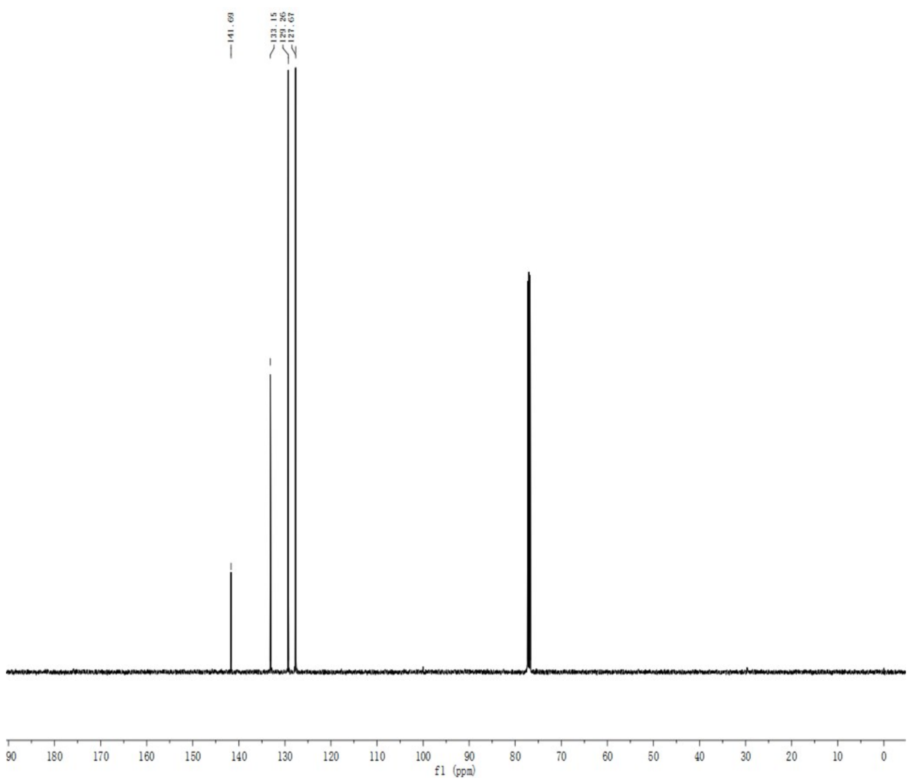


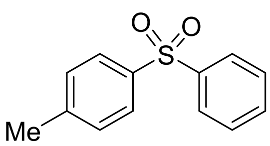


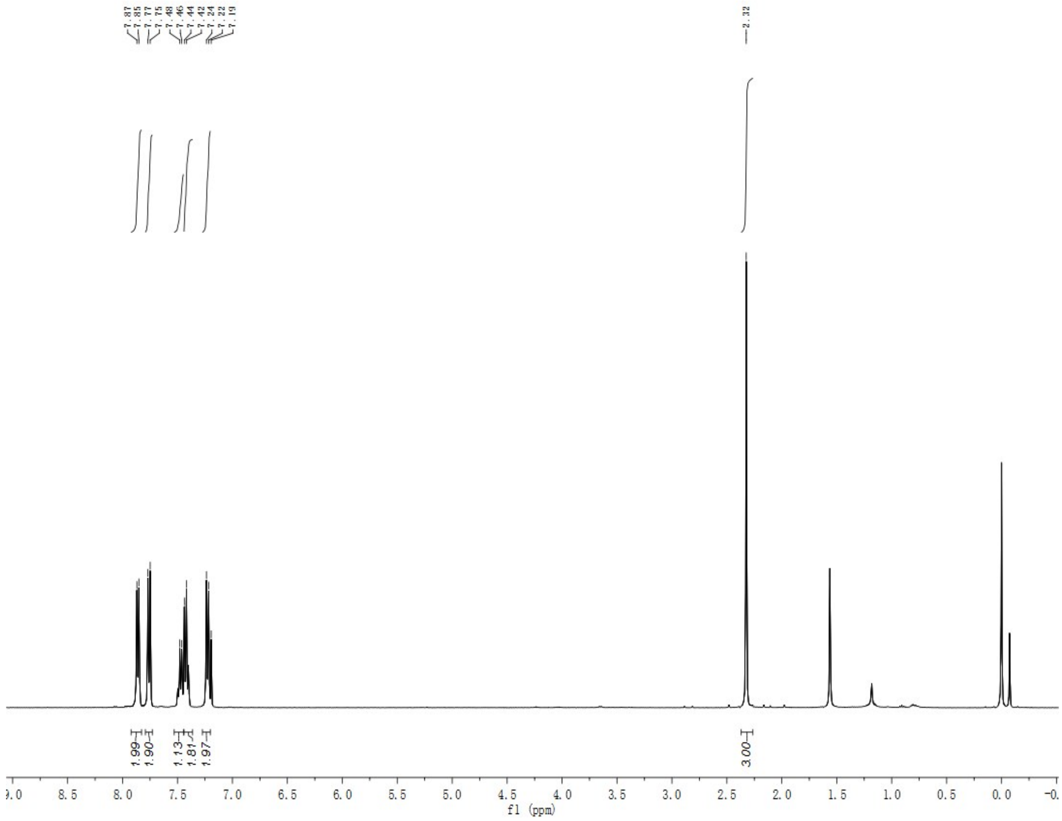


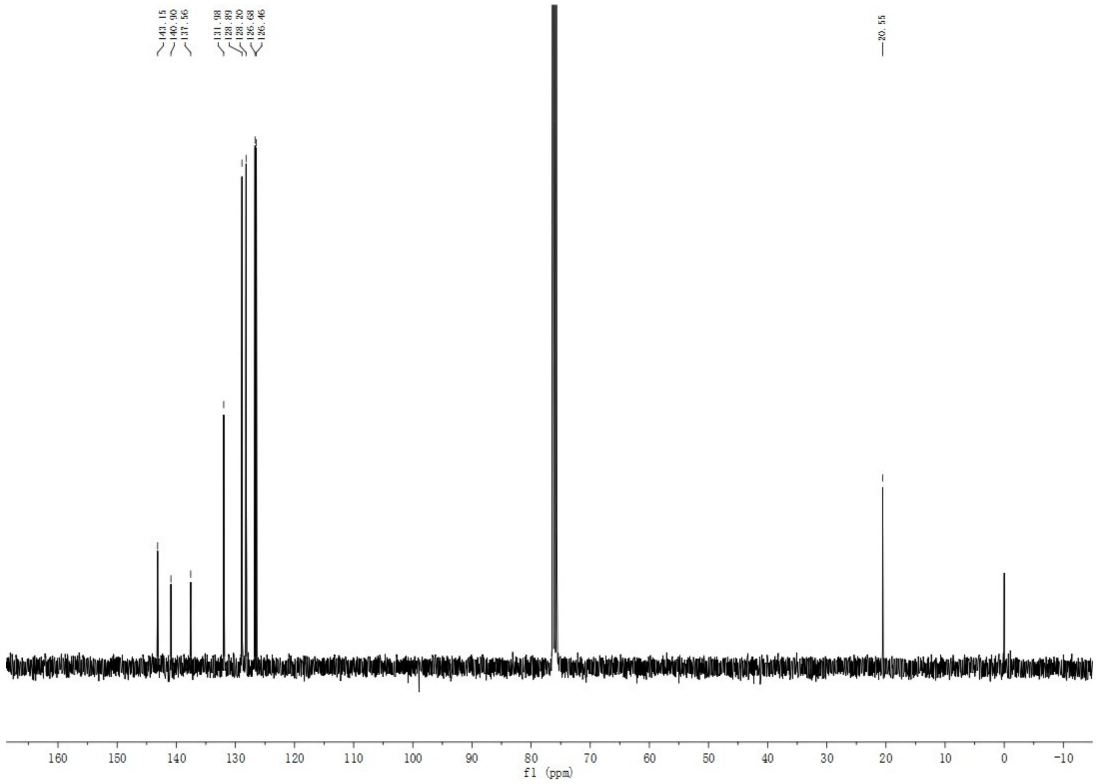


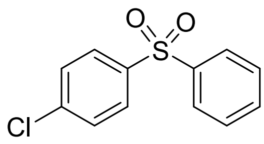

7.98
7.96
7.60
7.58
7.57
7.54
7.52
7.51
3.91
2.00
3.97
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


141.69
133.15
129.26
127.67
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

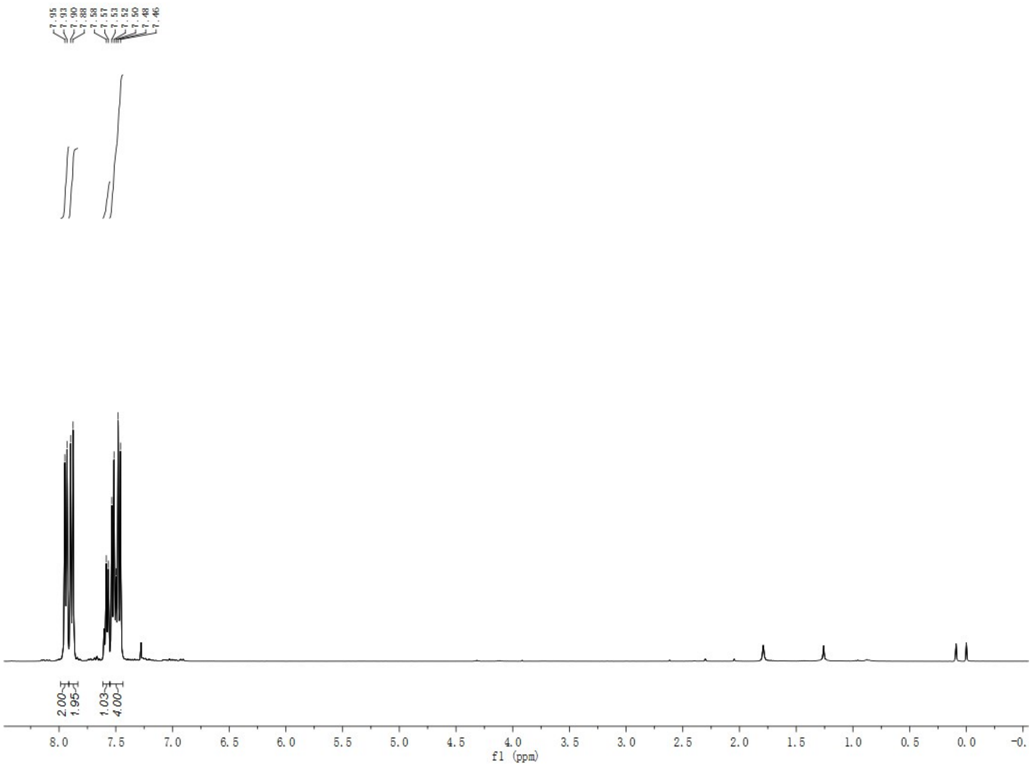

O
S
O
Me


7.87
7.85
7.77
7.75
7.48
7.46
7.44
7.42
7.24
7.22
7.19
2.32
1.99
1.90
1.13
1.81
1.97
3.00
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)

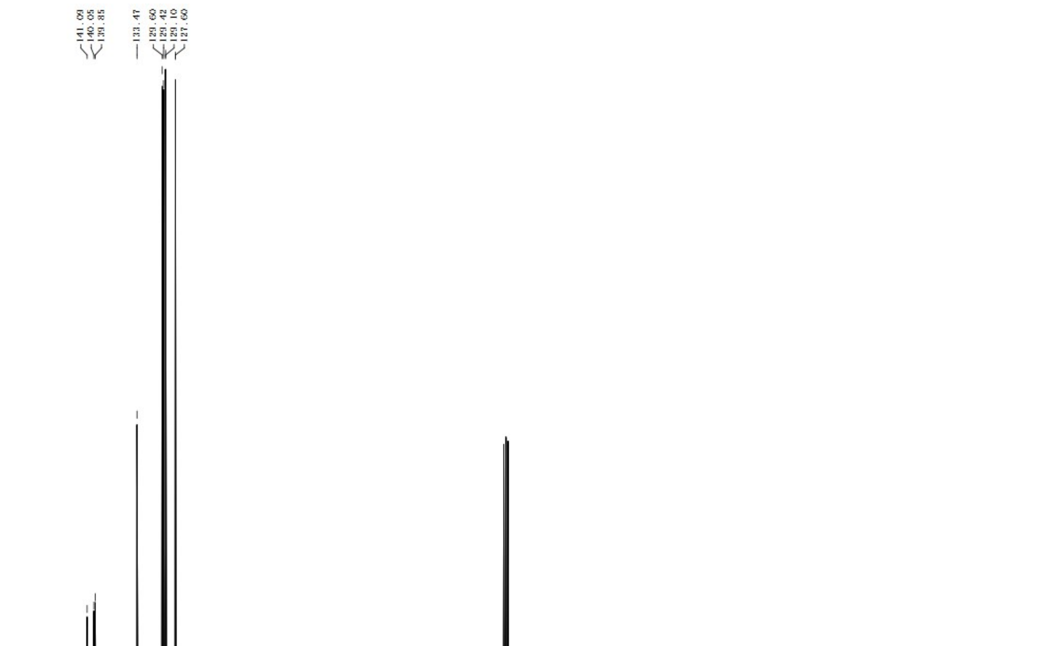

143.15
140.90
137.56
131.98
128.89
128.20
126.68
126.46
20.55
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



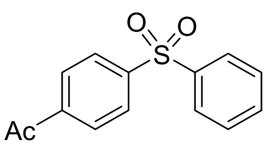
O
O
S
Cl

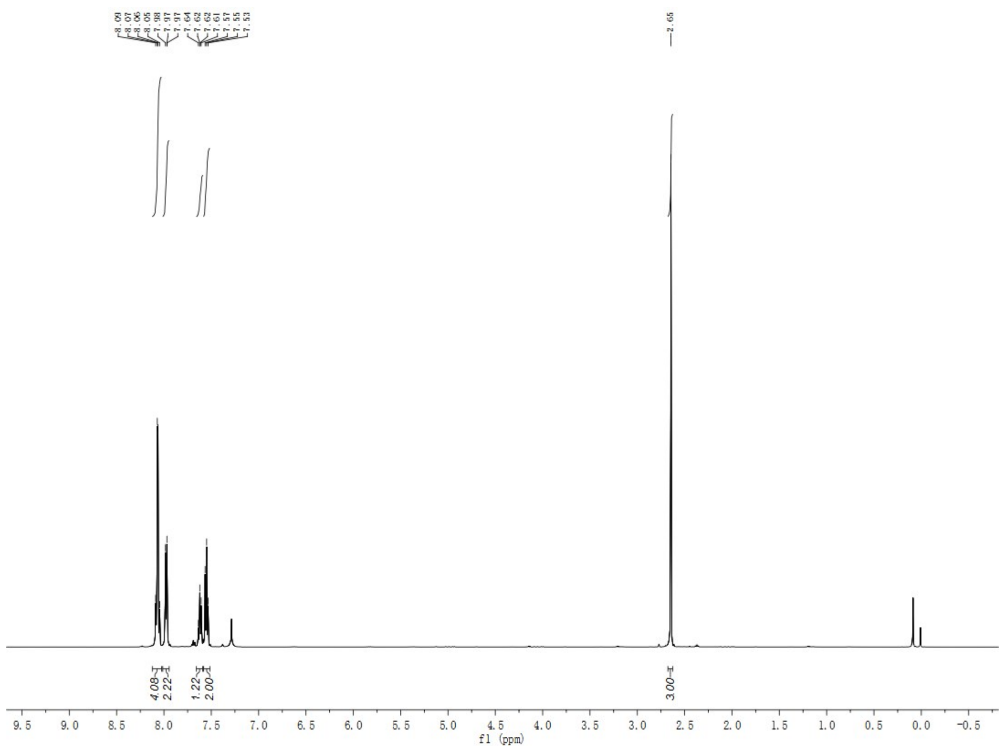


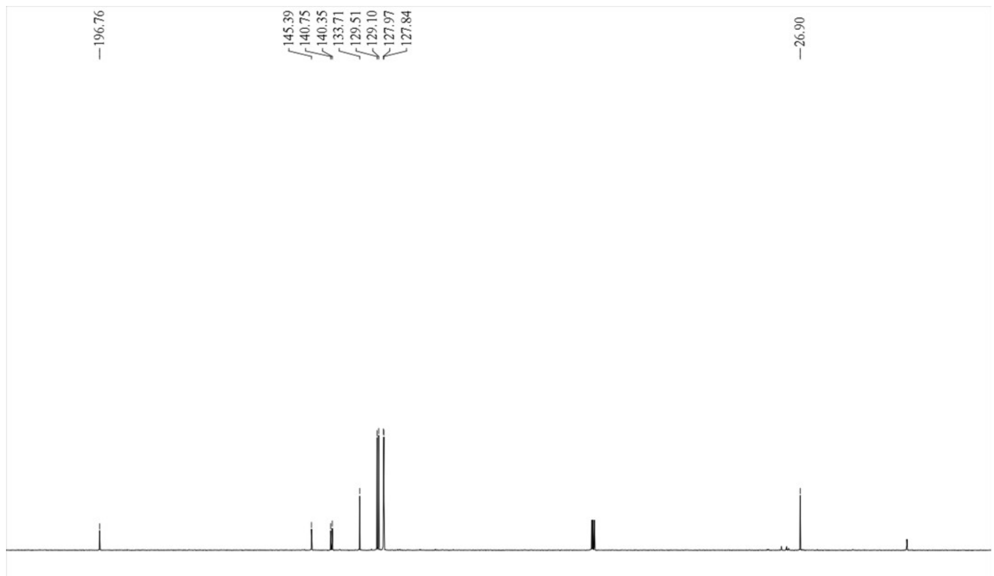
7.95
7.93
7.90
7.88
7.58
7.57
7.53
7.52
7.50
7.48
7.46
2.00
1.95
1.03
4.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

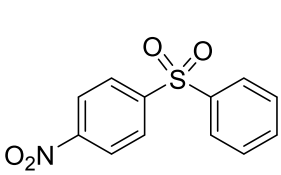


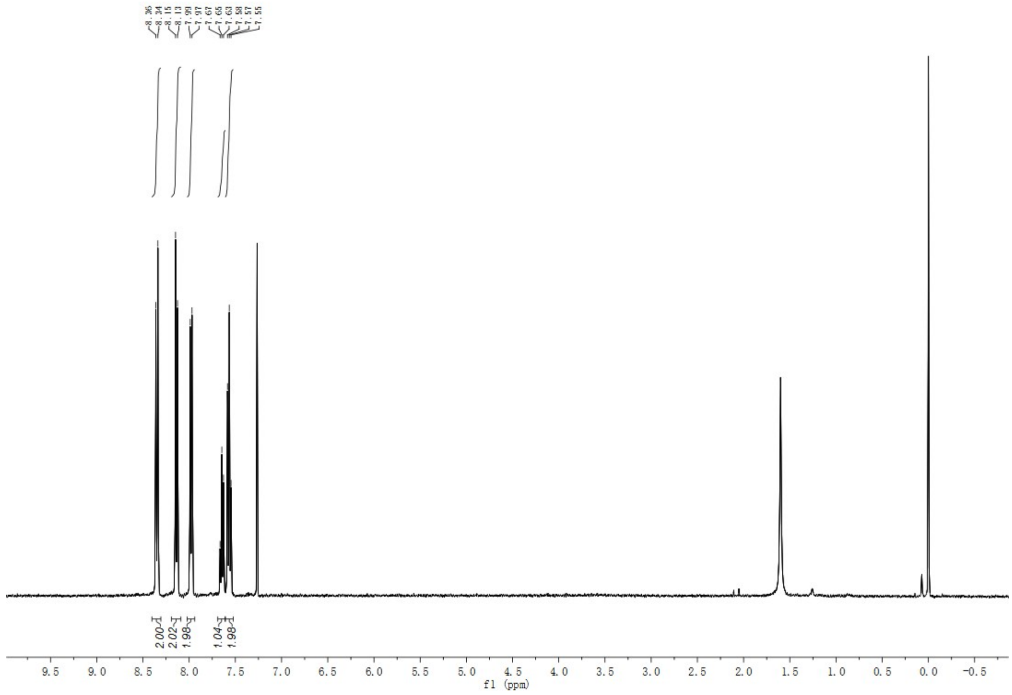
141.09
140.05
139.85
133.47
129.60
129.42
129.10
127.60
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

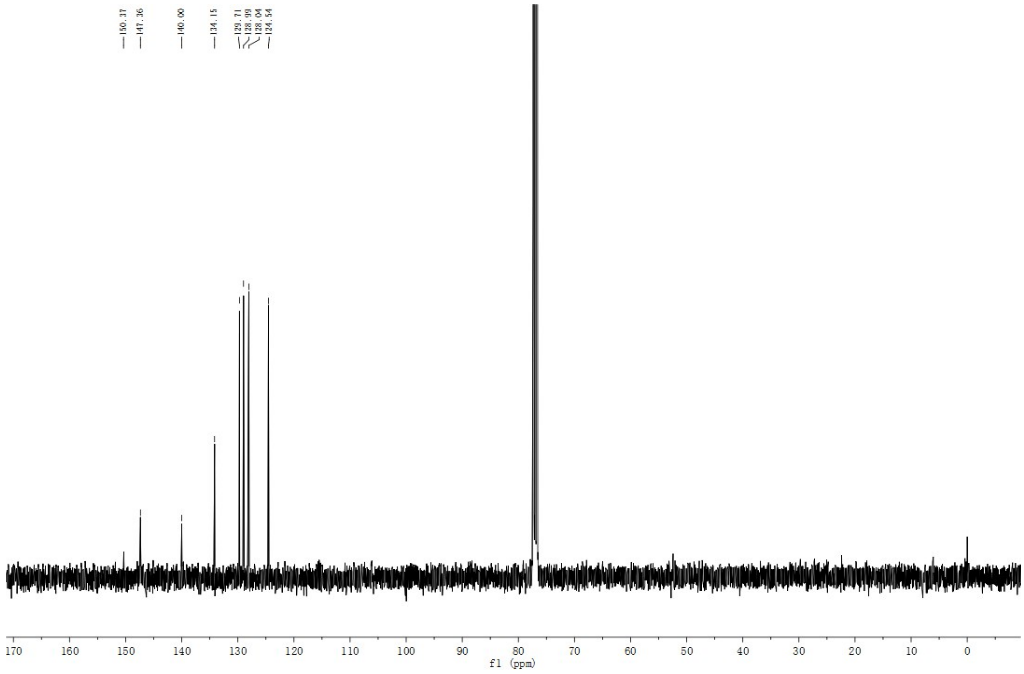

O
O
S
Ac

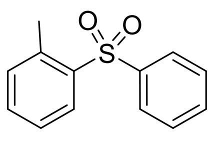

8.09
8.07
8.06
8.05
7.98
7.97
7.97
7.64
7.62
7.62
7.61
7.57
7.55
7.53
2.65
4.08
2.22
1.22
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

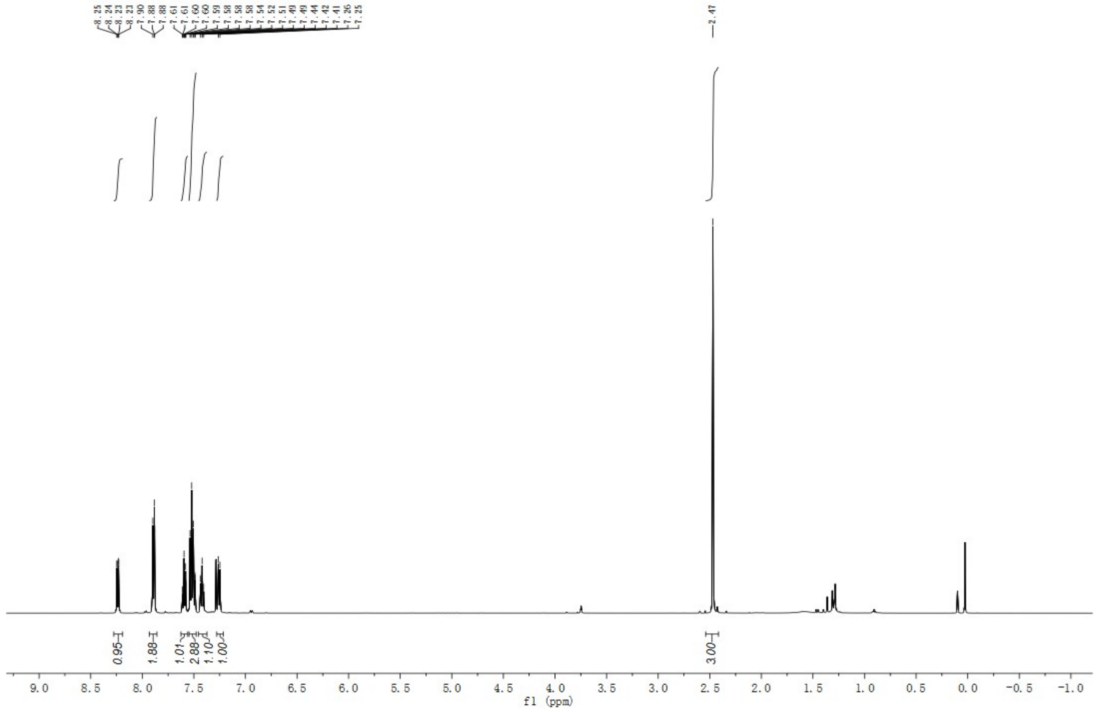

196.76
145.39
140.75
140.35
133.71
129.51
129.10
127.97
127.84
26.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
O
S
O_2N

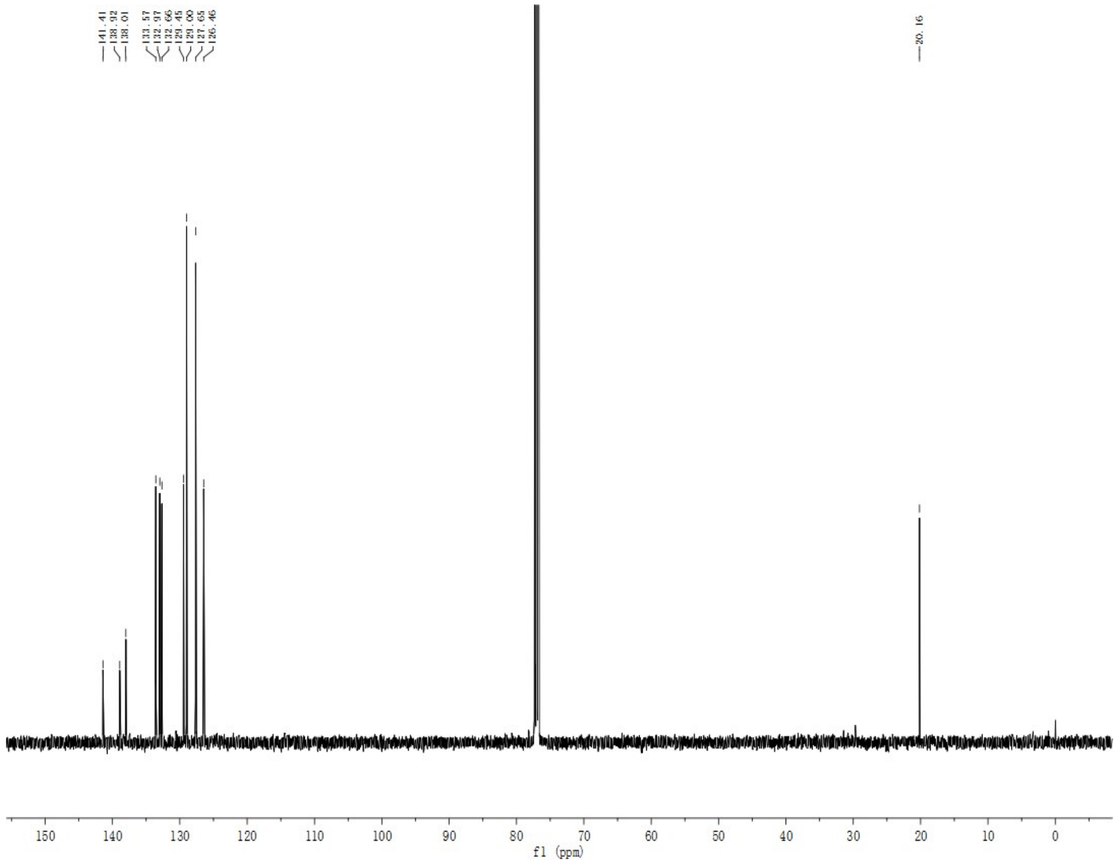

8.36
8.34
8.15
8.13
7.99
7.97
7.67
7.65
7.63
7.58
7.57
7.55
2.00
2.02
1.98
1.04
1.98
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


150.37
147.36
140.00
134.15
129.71
128.99
128.04
124.54
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

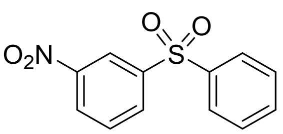

O
S
O



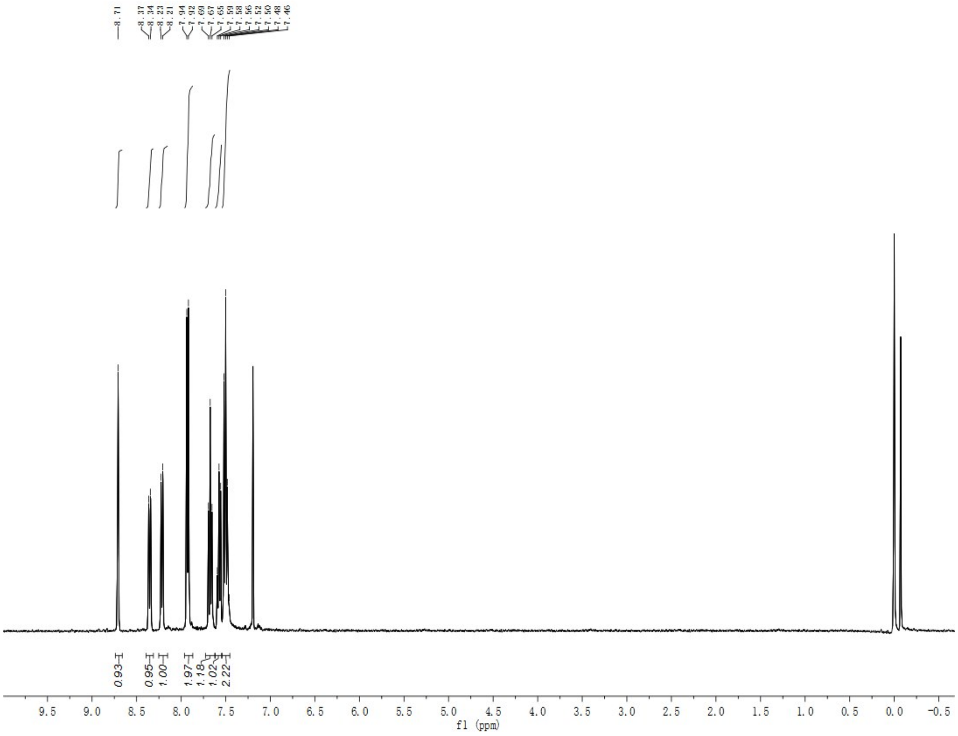
2.47
0.95
1.88
1.01
2.88
1.10
1.00
3.00
f1 (ppm)



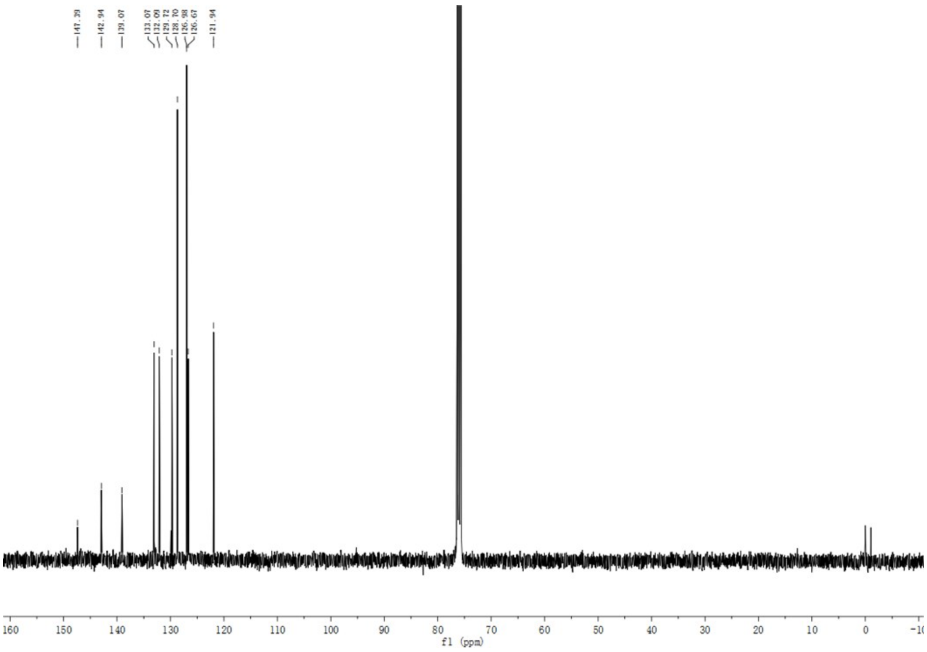
141.41
138.92
138.01
133.57
132.97
132.66
129.45
129.00
127.65
126.46
20.16
f1 (ppm)



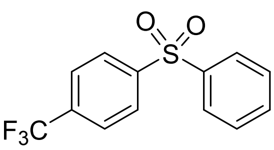
O
O
S
O2N



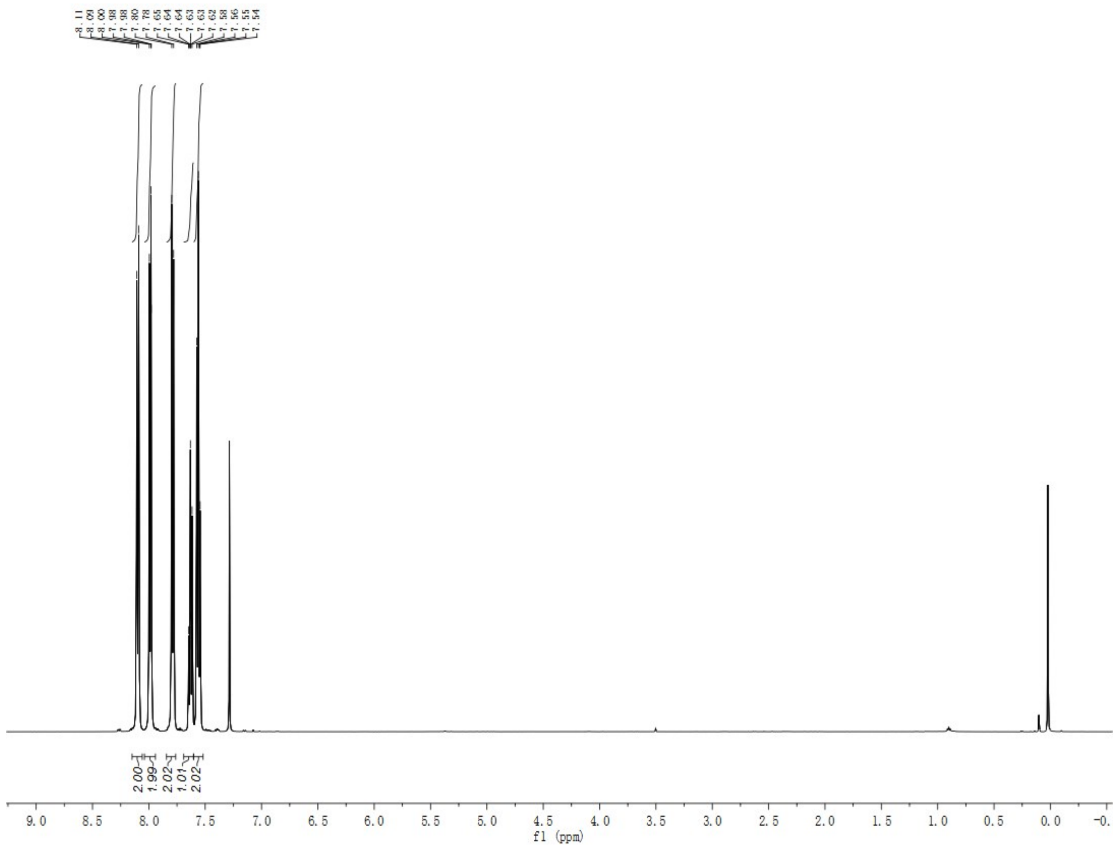
8.71
8.37
8.34
8.23
8.21
7.94
7.92
7.69
7.67
7.65
7.59
7.58
7.56
7.52
7.50
7.48
7.46
0.93
0.95
1.00
1.97
1.18
1.02
2.22
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)



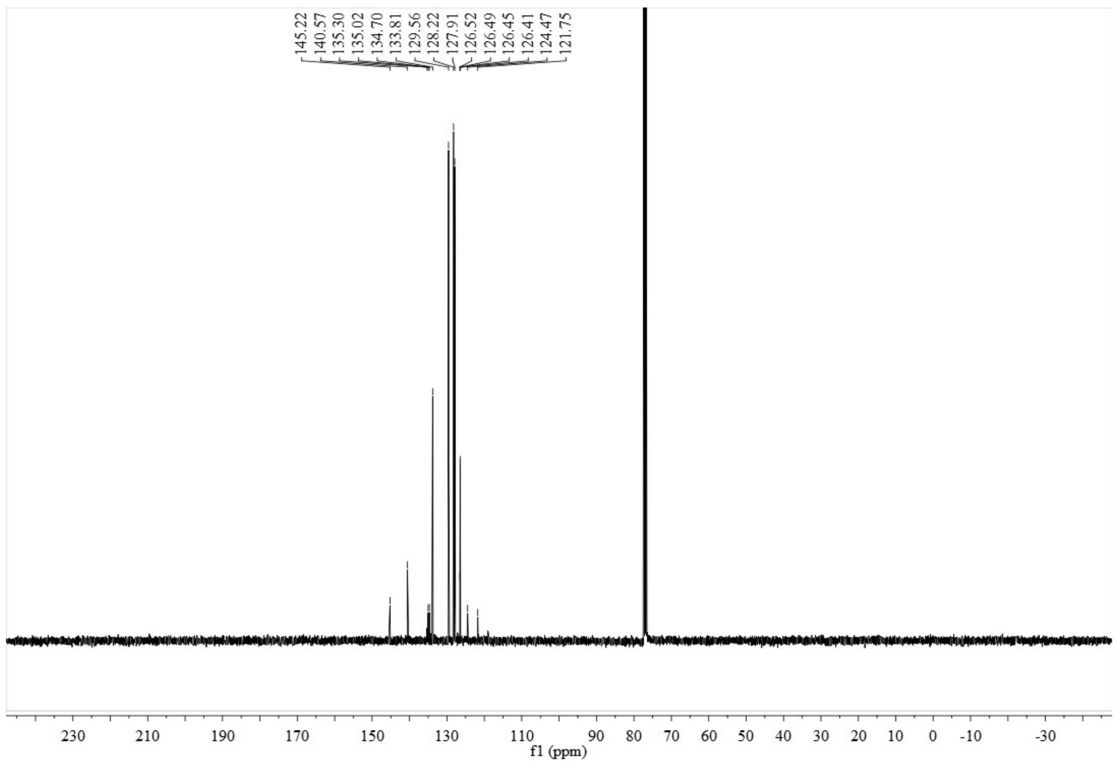
147.39
142.94
139.07
133.07
132.09
129.72
128.70
126.98
126.67
121.94
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



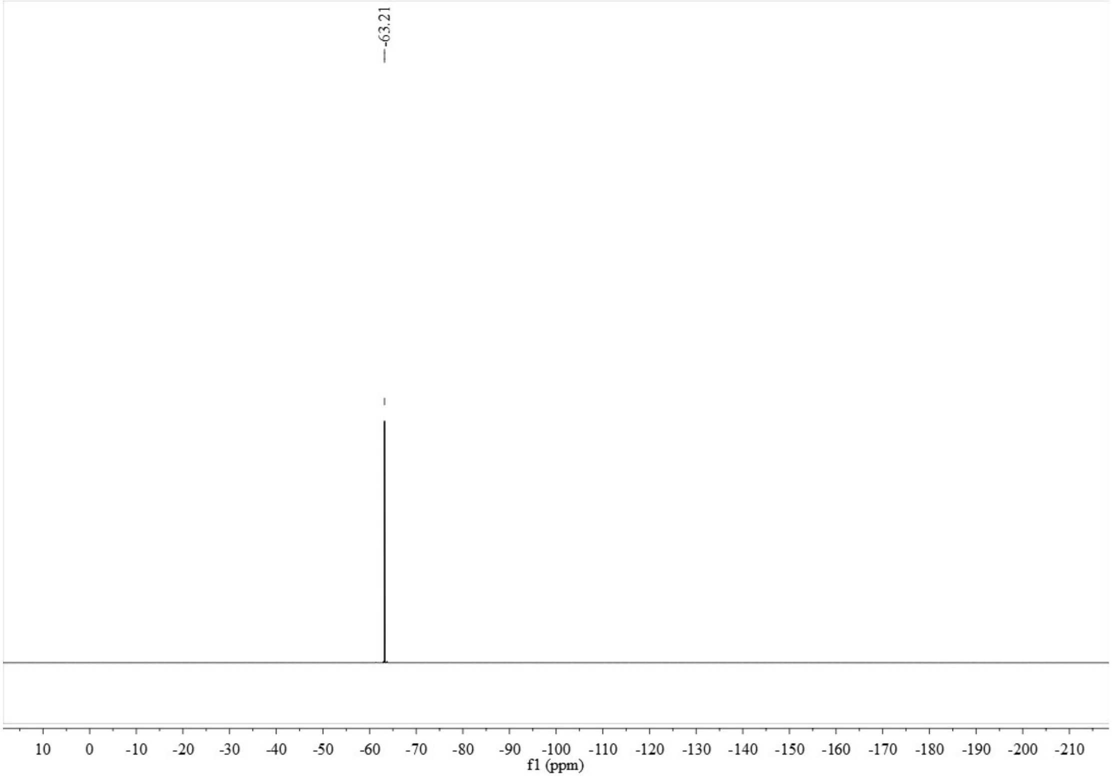
O
O
S
F3C



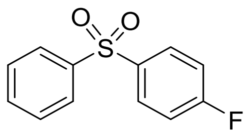
8.11
8.09
8.00
7.98
7.98
7.80
7.78
7.65
7.64
7.64
7.63
7.63
7.62
7.58
7.56
7.55
7.54
2.00
1.99
2.02
1.01
2.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



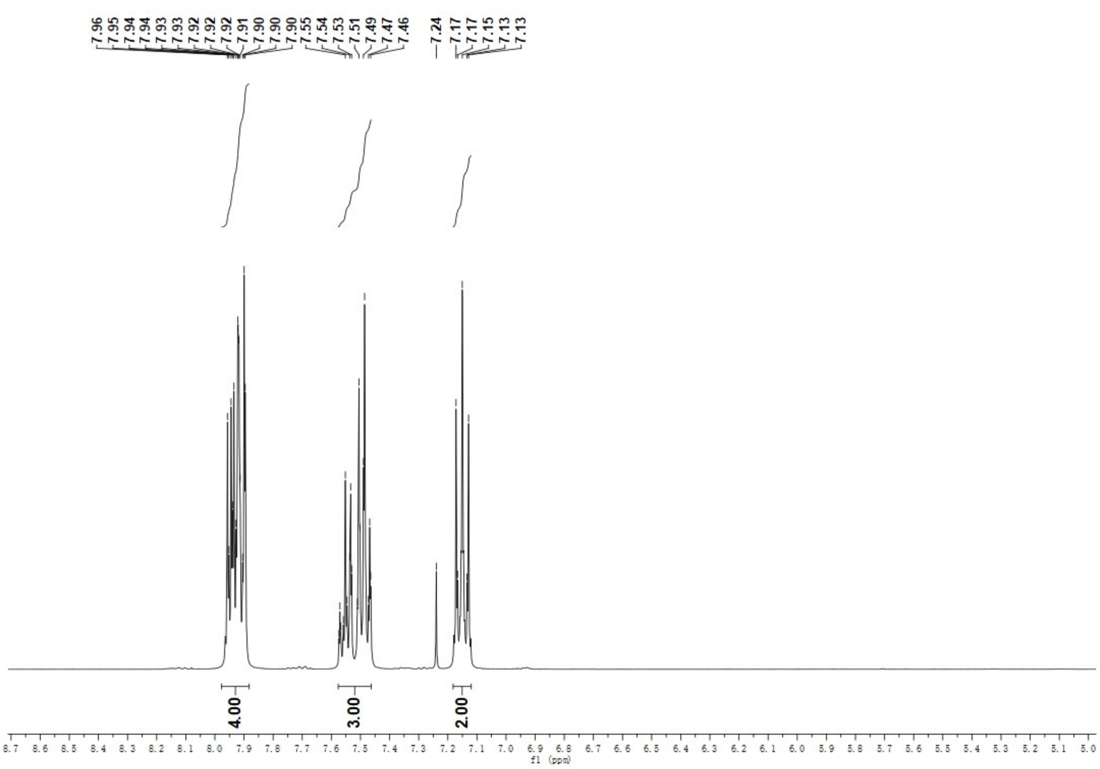
145.22
140.57
135.30
135.02
134.70
133.81
129.56
128.22
127.91
126.52
126.49
126.45
126.41
124.47
121.75
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -30
f1 (ppm)



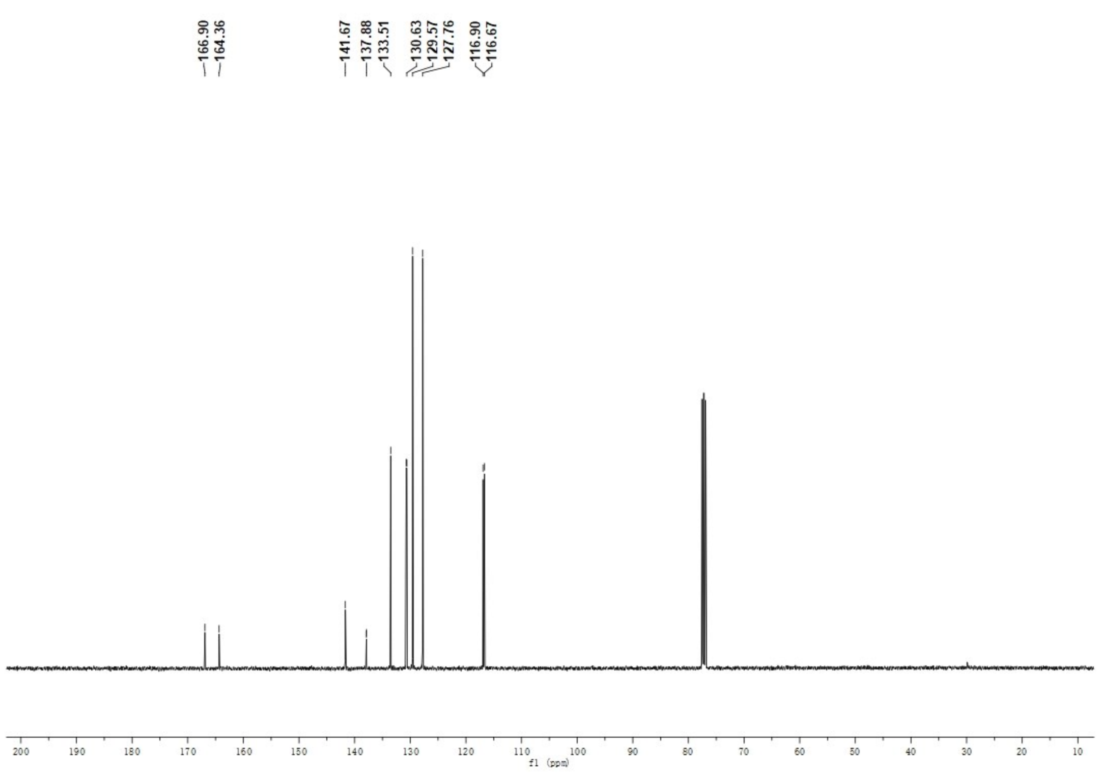
-63.21
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)



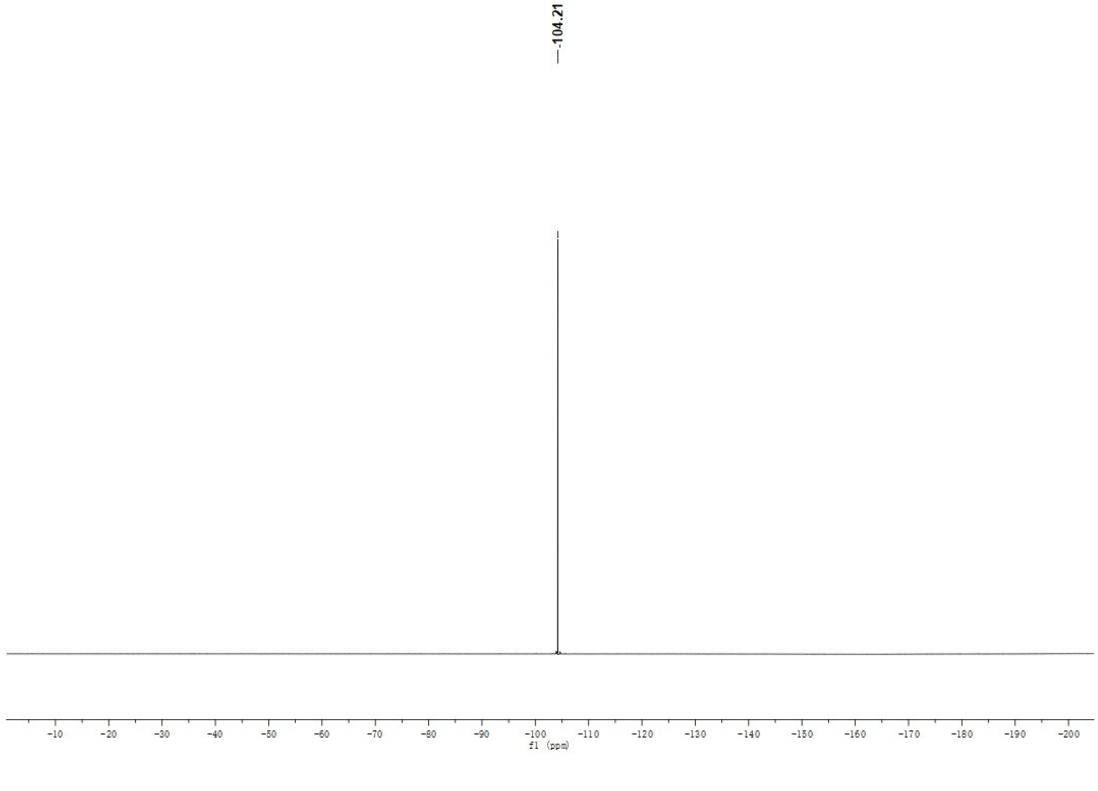
O
S
O
F



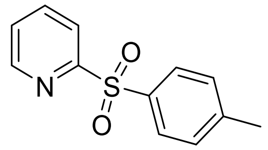
7.96 7.95 7.94 7.94 7.93 7.93 7.92 7.92 7.92 7.91 7.90 7.90 7.90 7.55 7.54 7.53 7.51 7.49 7.47 7.46
7.24
7.17 7.17 7.15 7.13 7.13
4.00
3.00
2.00
8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0
f1 (ppm)



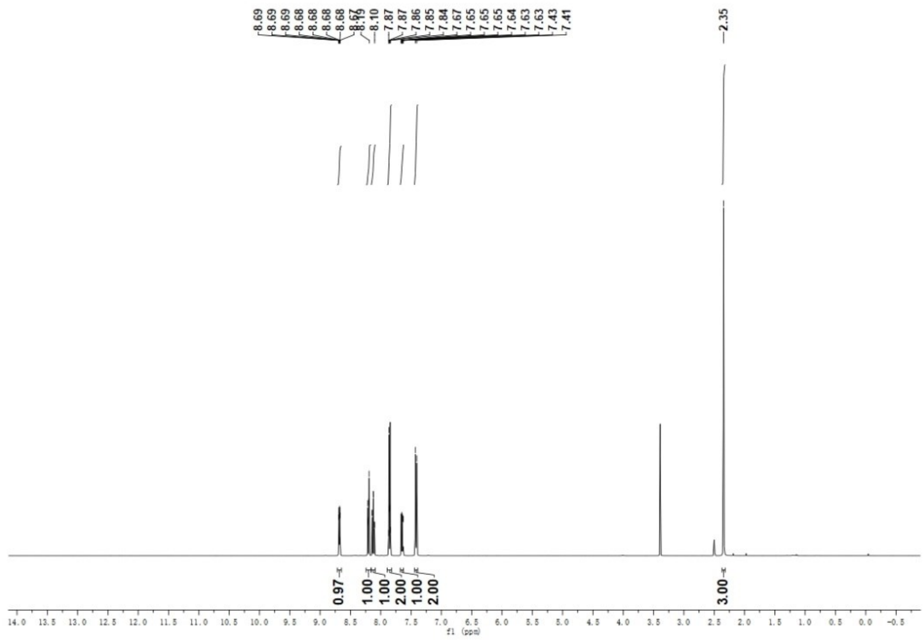
166.90
164.36
141.67
137.88
133.51
130.63
129.57
127.76
116.90
116.67
f1 (ppm)

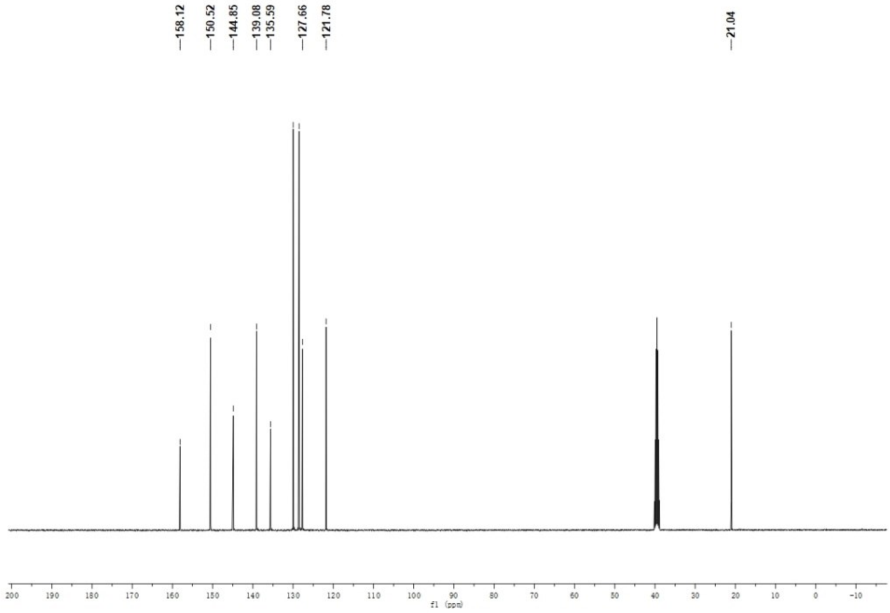


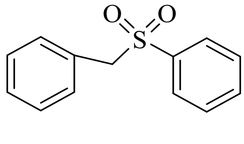
-104.21
f1 (ppm)

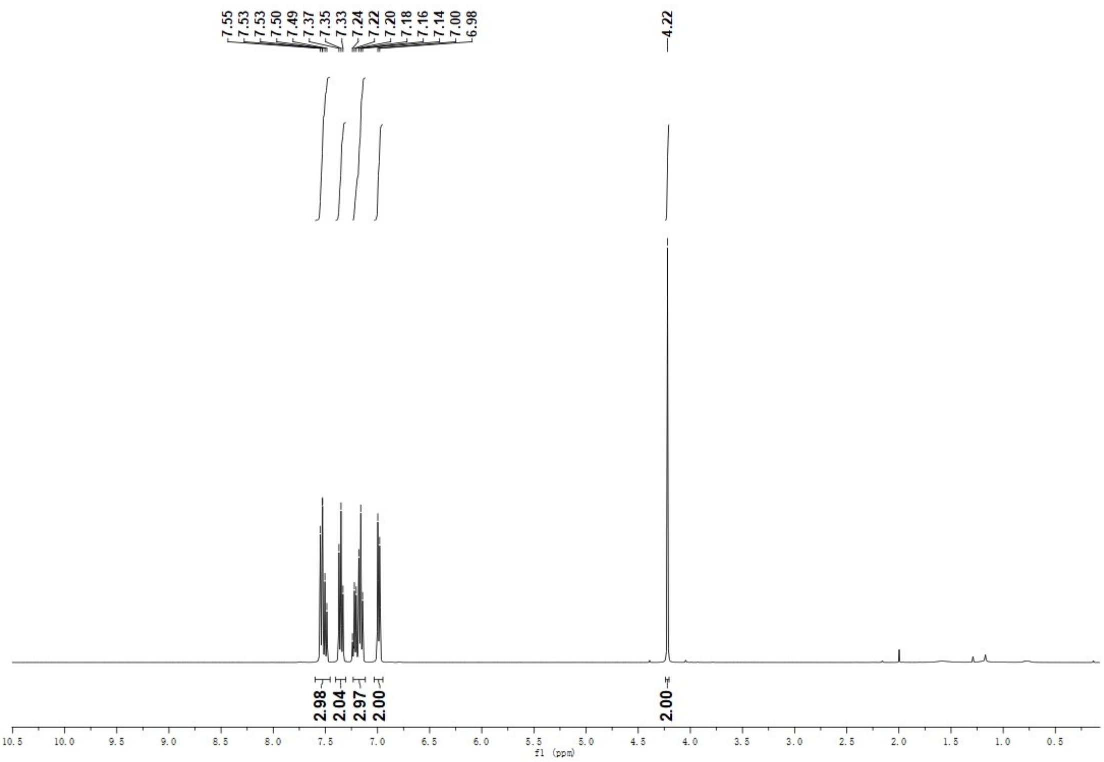


N
S
O
O


8.69
8.69
8.69
8.68
8.68
8.68
8.68
8.10
7.87
7.87
7.86
7.85
7.84
7.67
7.65
7.65
7.65
7.64
7.63
7.63
7.43
7.41
2.35
0.97
1.00
1.00
2.00
1.00
2.00
3.00
f1 (ppm)


158.12
150.52
144.85
139.08
135.59
127.66
121.78
21.04
f1 (ppm)

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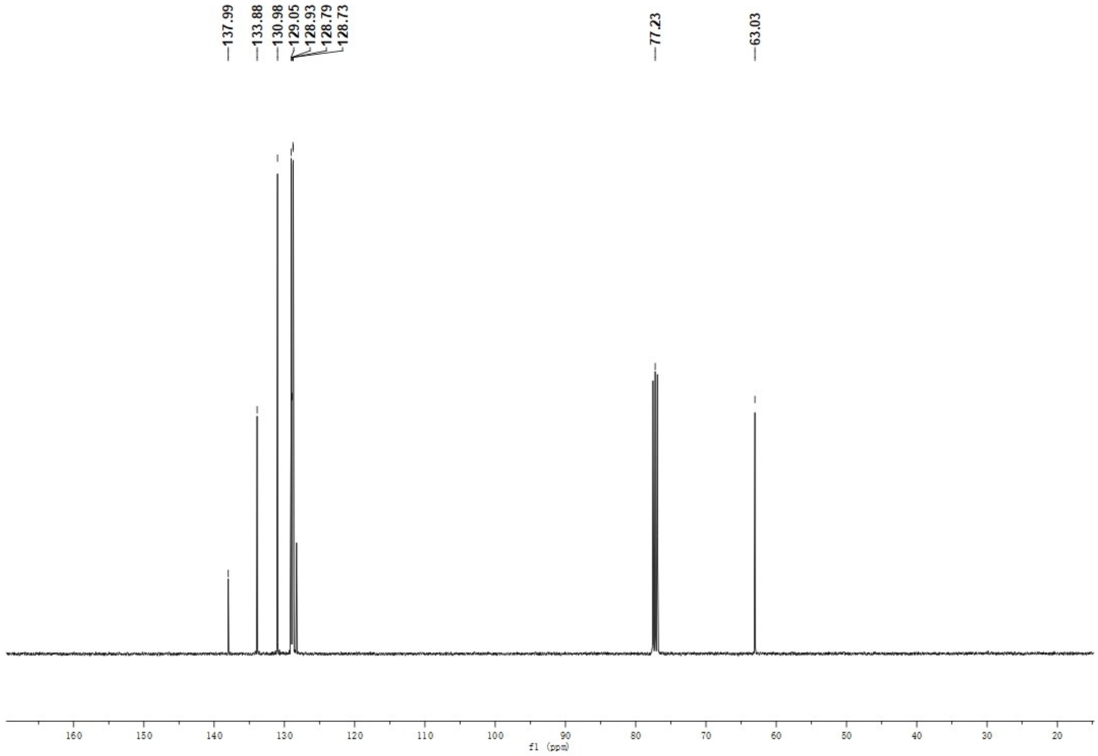
7.53
7.53
7.50
7.49
7.37
7.35
7.33
7.24
7.22
7.20
7.18
7.16
7.14
7.00
6.98
4.22
2.98
2.04
2.97
2.00
2.00
f1 (ppm)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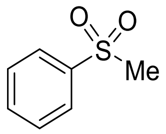
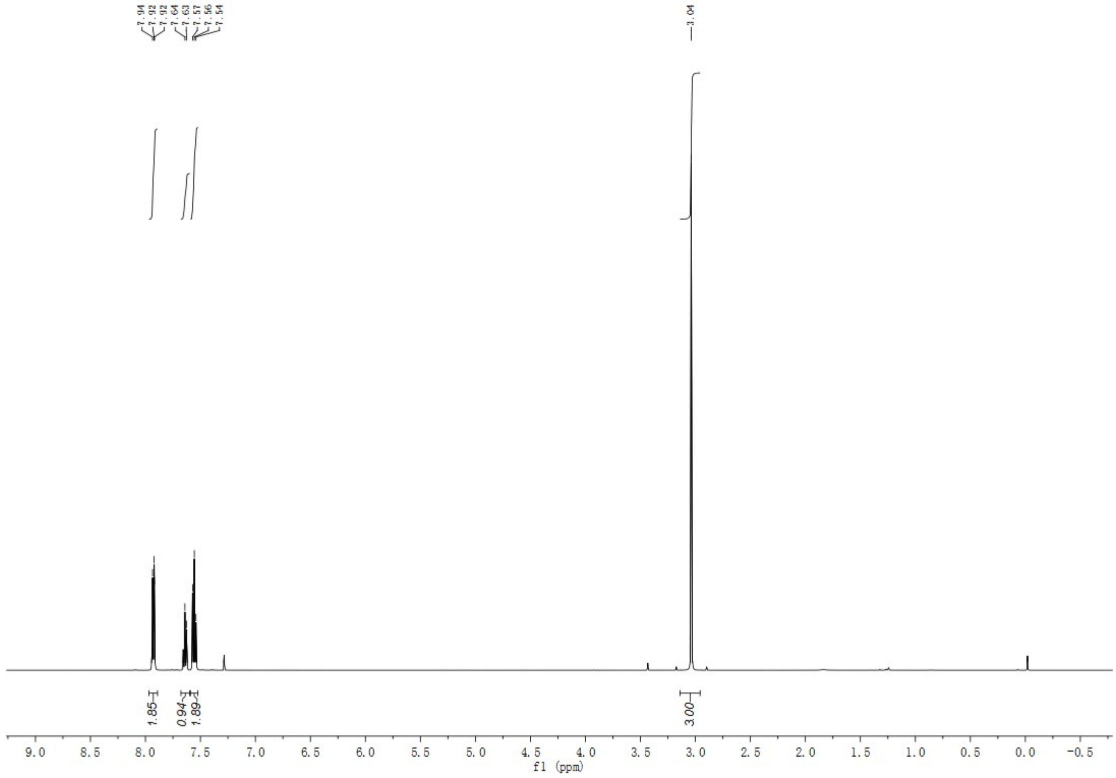
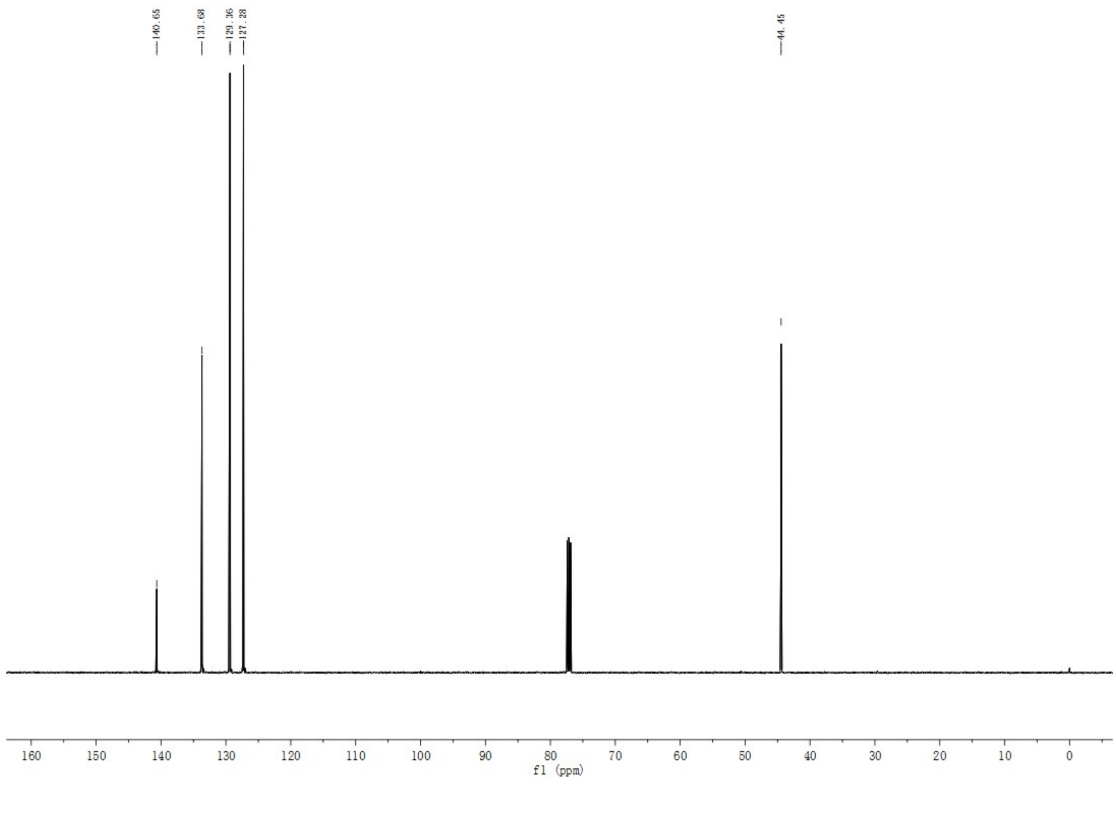
133.88
130.98
129.05
128.93
128.79
128.73
77.23
63.03
f1 (ppm)


O
O
S
Me


7.94
7.92
7.92
7.64
7.63
7.57
7.56
7.54
3.04
1.85
0.94
1.89
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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133.68
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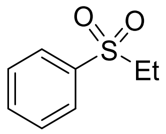
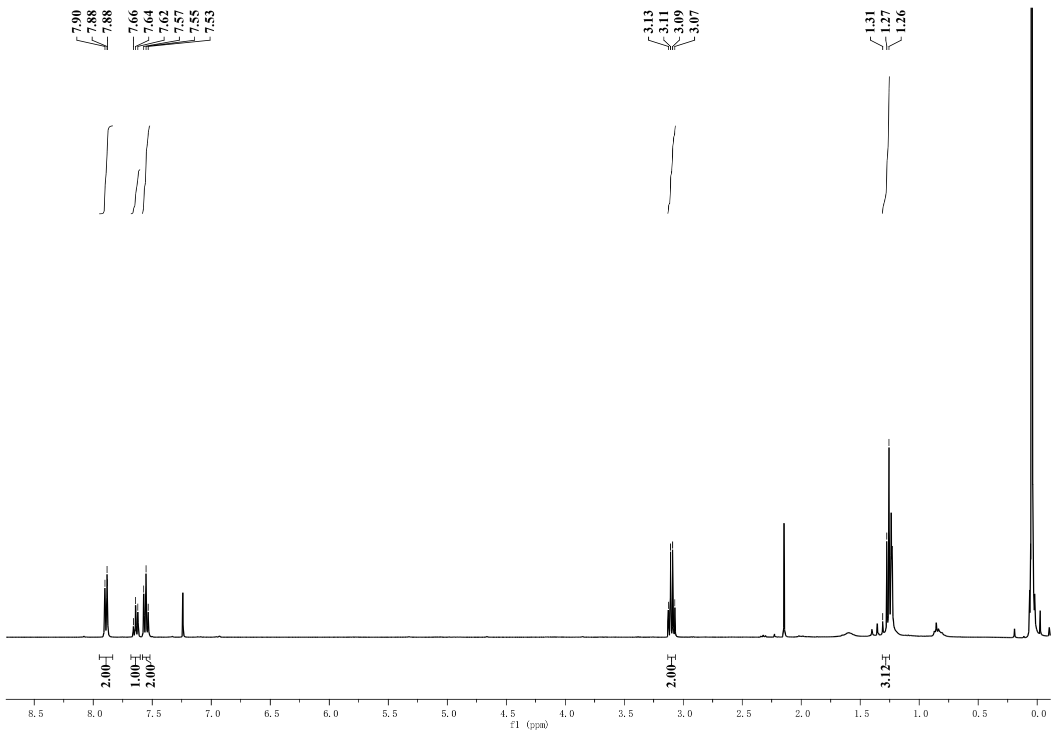
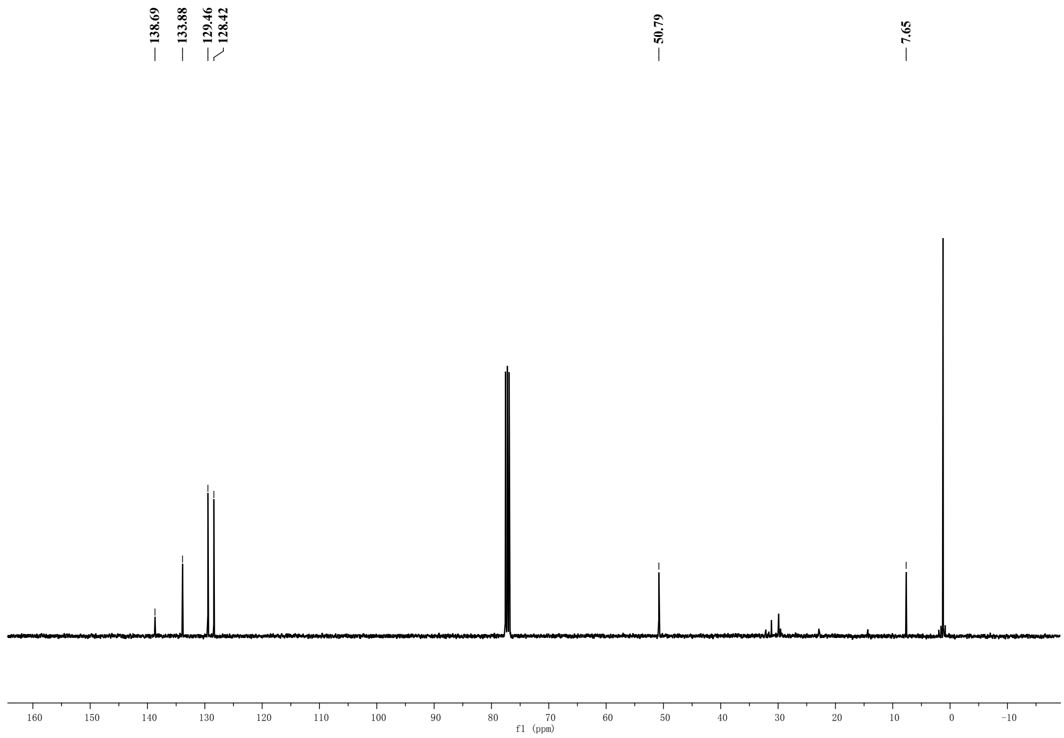
127.28
44.45
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O
S
Et


7.90
7.88
7.88
7.66
7.64
7.62
7.57
7.55
7.53
3.13
3.11
3.09
3.07
1.31
1.27
1.26
2.00
1.00
2.00
2.00
3.12
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)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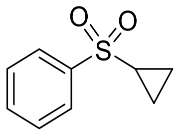
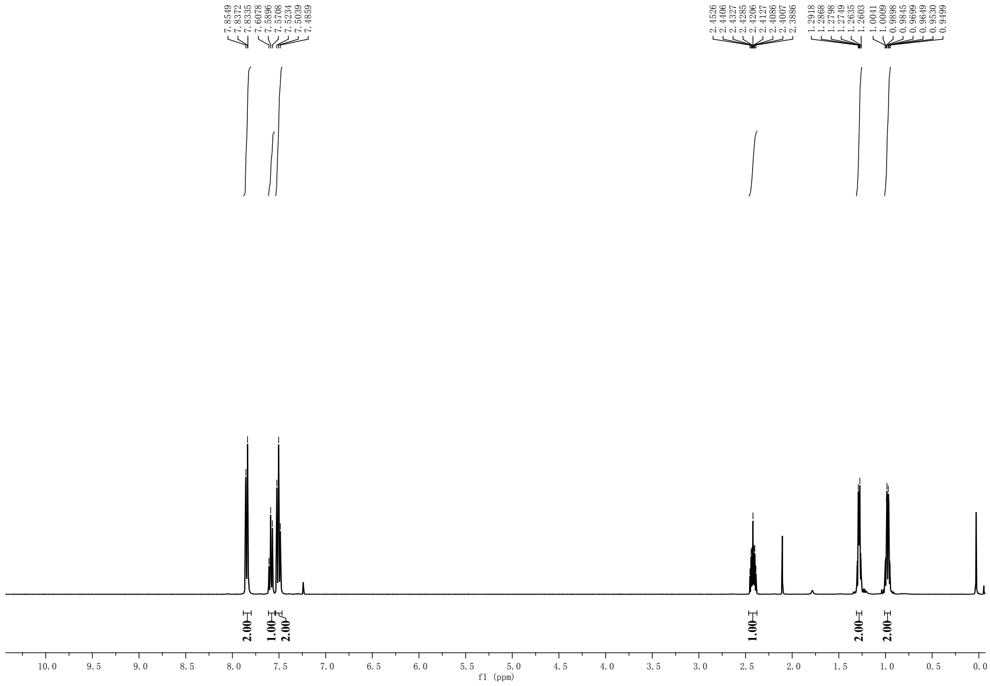
129.46
128.42
50.79
7.65
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
O
S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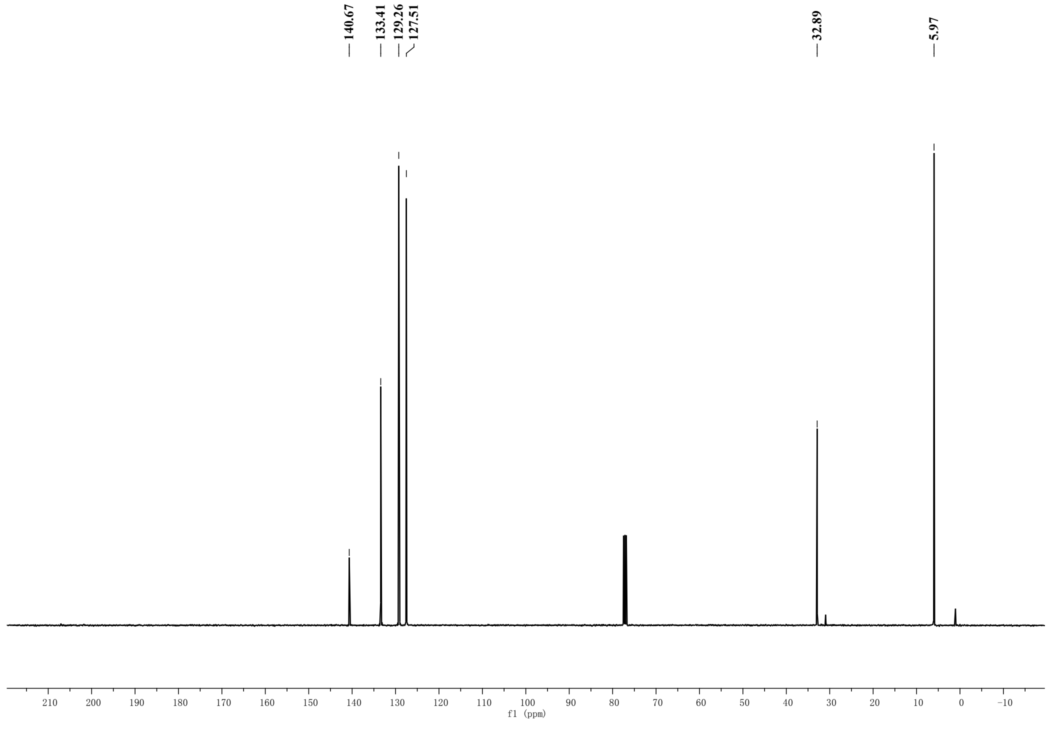
7.8372
7.8335
7.6078
7.5896
7.5708
7.5234
7.5039
7.4859
2.4526
2.4406
2.4327
2.4285
2.4206
2.4127
2.4086
2.4007
2.3886
1.2918
1.2868
1.2798
1.2749
1.2635
1.2603
1.0041
1.0009
0.9898
0.9845
0.9699
0.9649
0.9530
0.9499
2.00
1.00
2.00
1.00
2.00
2.00
f1 (ppm)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133.41
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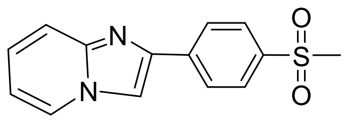
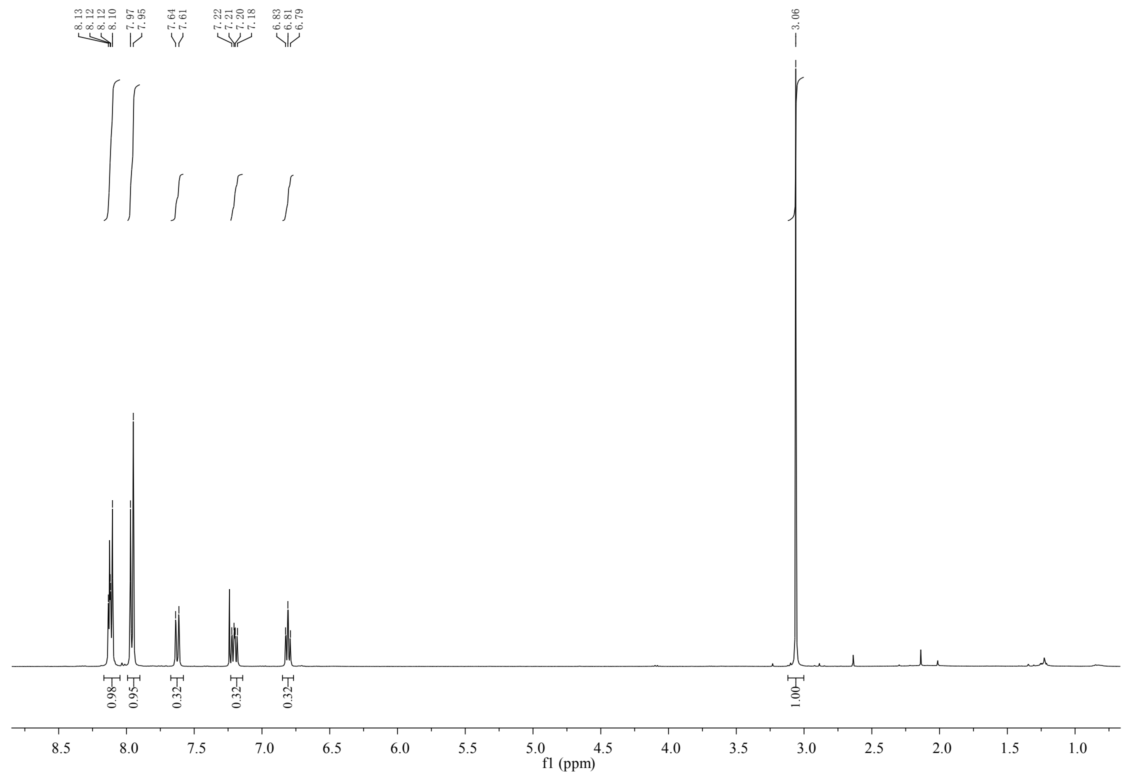
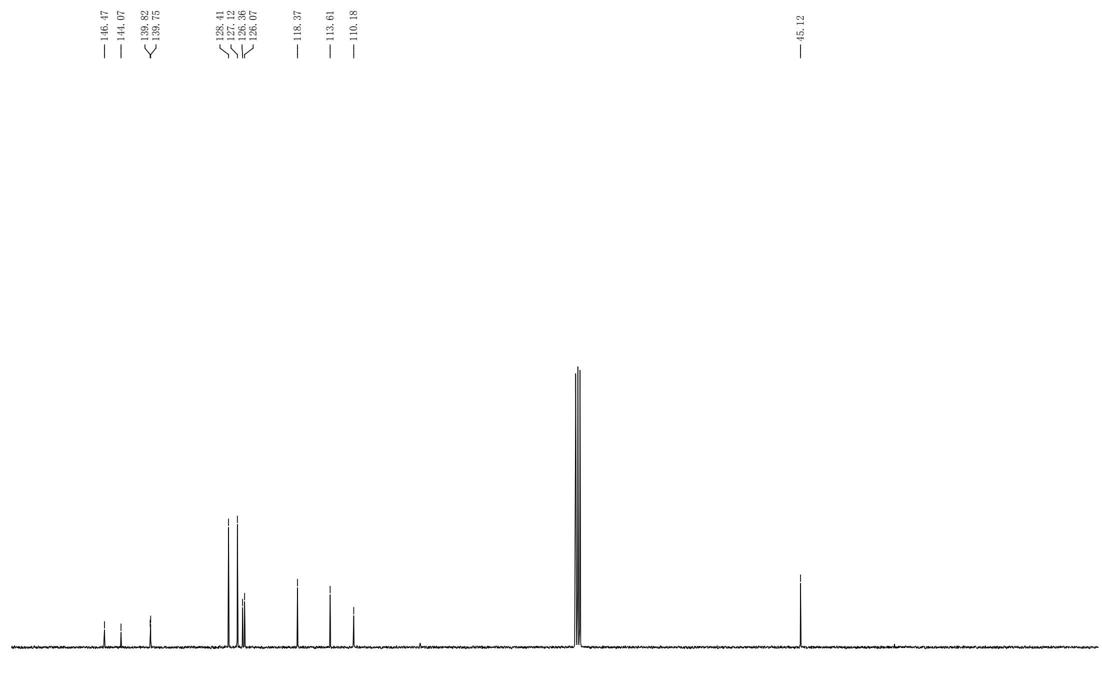
127.51
32.89
5.97
f1 (ppm)


N
N
S
O
O


8.13
8.12
8.12
8.10
7.97
7.95
7.64
7.61
7.22
7.21
7.20
7.18
6.83
6.81
6.79
3.06
0.98
0.95
0.32
0.32
0.32
1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)


146.47
144.07
139.82
139.75
128.41
127.12
126.36
126.07
118.37
113.61
110.18
45.12
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

References

1. C. Shen, J. Xu, W. Yu and P. Zhang, *Green Chem.,* 2014, **16**, 3007-3012.