Electronic Supplementary Material (ESI) for New Journal of Chemistry.

The recoverable Isatin resin as a ligand for copper-type Ullmann *C*-*N* coupling reaction

Changxu Zhao, Xu Shi, Zhen Zhang, Jifan Wang, Guoliang Chen*

Key Laboratory of Structure-Based Drug Design & Discovery of Ministry of Education, Shenyang Pharmaceutical University, Shenyang 110016, Liaoning, P. R. of China.

Table of Contents

1. General information

Unless otherwise noted, all chemical reagents and solvents were obtained from commercial sources and were used without further purification. Chloromethyl Polystyrene Resin cross-linked with 1 % DVB (100-200 mesh, 1-1.24 mmol/g). TLC was performed on silicagel 60 GF254 and monitored under UV light. Melting points (m.p.) were measured with Hanon MP420 melting point apparatus and was uncorrected. Column chromatography was performed on 200-400 mesh silica gel under pressure. NMR spectra were obtained on a Bruker AVIII-HD-400 spectrometer (^{1}H), TMS as internal standard and chemical shifts (δ) were given ppm. The following abbreviations were applied in reporting NMR data: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), dt (doublets of triplet), td (triplets of doublet) and m (multiplet). The mass spectral date were obtained on Waters ACQUITY QDa Mass Detector. The infrared spectral data were measured on FTIR-650 Fourier transform infrared spectrometer.

2. Synthesis of polymer ligand L

2.1 General procedure for the preparation indole-2,3-dione ligand L

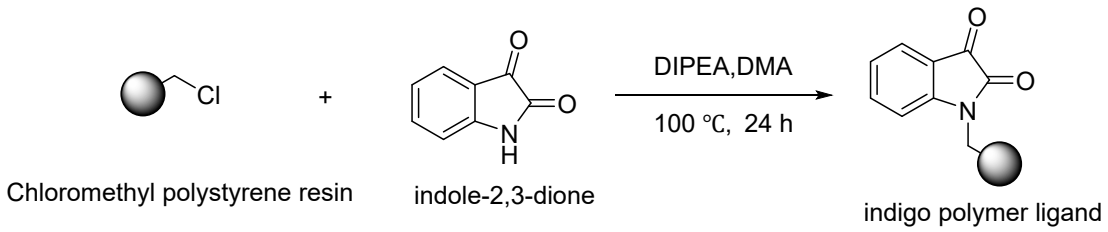


Figure S1. Synthesis of L

Chloromethyl polystyrene resin 5.000 g (5-6.20 mmol -Cl) swelled in 50 mL DMA for 12 h. Then indole-2,3-dione (7.35 g, 50 mmol) , DIPEA (6.46 g, 50 mmol) were added to the mixture. The mixture was stirred at 100 ℃ for 24 h. After cooling to room temperature, the mixture was filtered, and the filter cake was washed with acetonitrile and 70 ℃ deionized water, and then dried under vacuum to give ligand L (5.906 g) as a red solid.

2.2 Fourier transform infrared (FTIR) spectra of ligand L

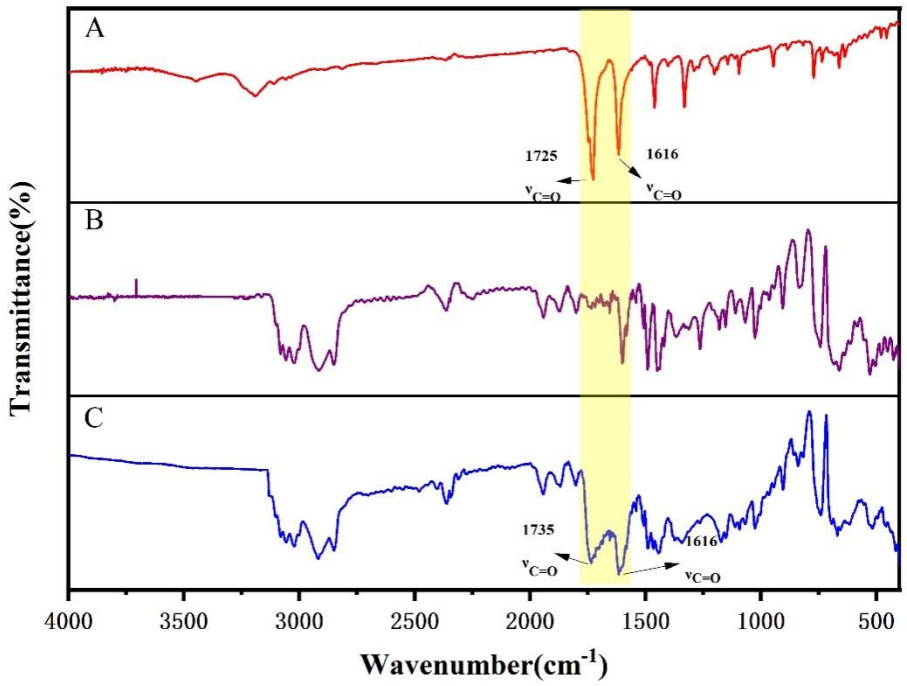
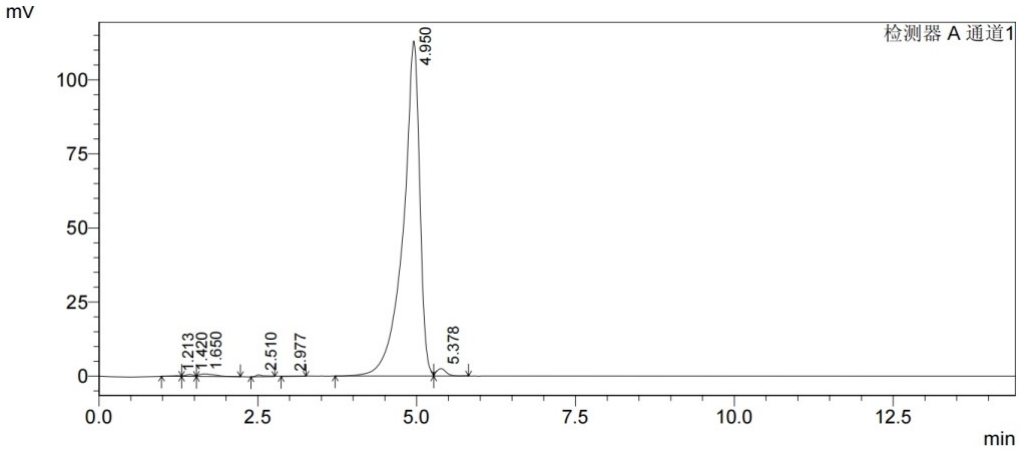


Figure S2. FTIR spectra of A (indole-2,3-dione), B (Chloromethyl polystyrene resin) and C (L)

Compared with the FTIR spectrum of Chloromethyl polystyrene resin, the most significant change in the infrared spectrum of L is the addition of a C=O stretching vibration peak at 1735 cm^{-1}. The strong absorption peaks at 1727 cm^{-1} and 1625 cm^{-1} in the FTIR spectrum of L correspond to the stretching vibration peaks of the carbonyl in the ketone carbonyl group.

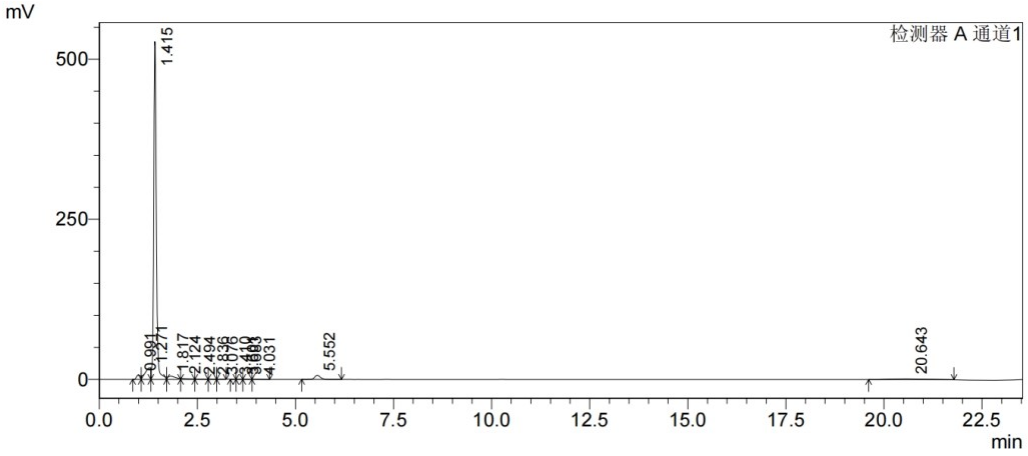
2.3 Determination of Isatin in reaction wastewater by high performance liquid chromatography (HPLC)

A: HPLC conditions: Agilent ZORBAX Extend-C18, 4.6×150 mm, 20 um, 25 % acetonitrile/75 % H_2O eluent, 1 mL/min, 254 nm.



峰#	保留时间	面积	高度	面积 %	高度 %
1	1.213	1720	162	0.082	0.138
2	1.420	5444	615	0.259	0.521
3	1.650	15305	872	0.729	0.738
4	2.510	5755	656	0.274	0.556
5	2.977	2783	152	0.132	0.129
6	4.950	2042851	113073	97.243	95.795
7	5.378	26904	2506	1.281	2.123
总计		2100762	118036	100.000	100.000

B: HPLC conditions: Agilent ZORBAX Extend-C18, 4.6×150 mm, 20 um, 25 % acetonitrile/75 % H_2O eluent, 1 mL/min, 254 nm.

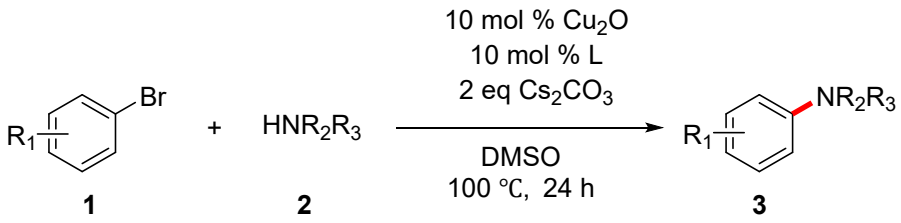


峰#	保留时间	面积	高度	面积 %
1	0.991	48179	6790	1.673
2	1.271	182018	18170	6.321
3	1.415	2402143	527628	83.418
4	1.817	71195	5026	2.472
5	2.124	18474	1364	0.642
6	2.494	8233	508	0.286
7	2.836	3534	316	0.123
8	3.076	3307	289	0.115
9	3.410	1261	160	0.044
10	3.601	2616	311	0.091
11	3.683	2788	296	0.097
12	4.031	2424	220	0.084
13	5.552	68722	6530	2.386
14	20.643	64756	1247	2.249
总计		2879650	568856	100.000

Figure S3 HPLC of reaction wastewater and Isatin. (**A**) Isatin; (**B**) wastewater (Reaction conditions: 4-Bromotoluene (3 mmol), pyrazole (3.75 mmol), Cu_2O (0.3 mmol), **L9** (0.3 g, 0.3 mmol), Cs_2CO_3 (6 mmol) and 4 mL DMSO)

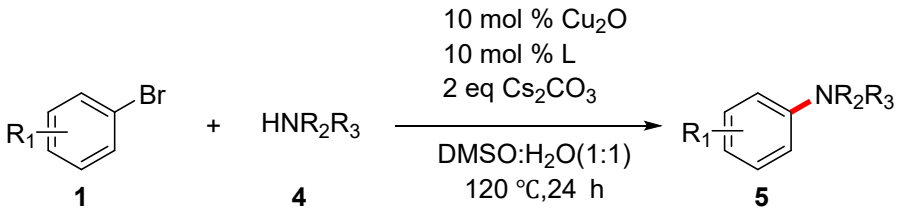
3. General procedure for amination

3.1 General procedure for the Cu_2O-catalyzed coupling of aryl bromides with nitrogen aromatic heterocycles



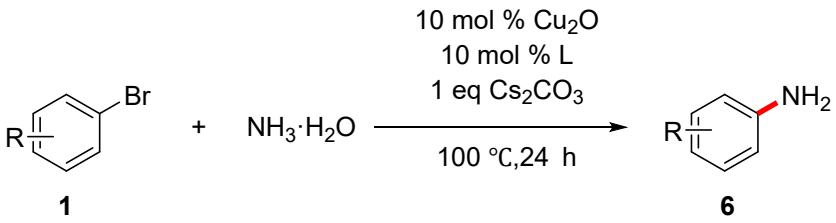
A 25 mL of seal tube with a magnetic stirring bar was charged with aryl bromides (3 mmol), amines (3.6 mmol), Cu_2O (0.04 g, 0.3 mmol), ligand L (0.47 g, 0.3 mmol), Cs_2CO_3 (1.95 g, 6 mmol), 5 mL DMSO. The mixture was stirred at 100 ℃ for 24 h. The reaction mixture was cooled to ambient temperature, suction filtration to recover the ligand L. The filtrate was diluted by 20 mL water and extracted with ethyl acetate (3×30 mL). The combined extracts was washed with brine and dried with $MgSO_4$. Filtered and evaporated under vacuum to give the crude product, which was purified with column chromatography on silica gel and eluting with petroleum ether /ethyl acetate or dichloromethane/methanol to give pure desired products.

3.2 General procedure for the Cu_2O-catalyzed coupling of aryl bromides with cyclic secondary amines



To a 25 mL seal tube with a magnetic stirring bar was charged with aryl bromides (3 mmol), amines (3.6 mmol), Cu_2O (0.3 mmol), ligand L (0.47 g, 0.3 mmol), Cs_2CO_3 (1.95 g, 6 mmol), 4 mL DMSO. The mixture was stirred at 105 ℃ for 24 h. After cooling to room temperature, the mixture was filtered to recover the ligand L. The filtrate was diluted by 20 mL water and extracted with ethyl acetate (3×30 mL). The combined extracts was washed with brine and dried with $MgSO_4$. Filtered and evaporated under vacuum to give the crude product, which was purified with column chromatography on silica gel and eluting with petroleum ether /ethyl acetate or dichloromethane/methanol to give pure desired products.

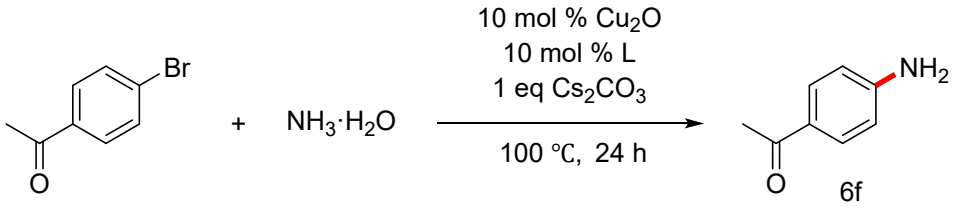
3.3 General procedure for the Cu_2O-catalyzed coupling of aryl bromides with ammonia solution



1 **6**

A 25 mL of seal tube with a magnetic stirring bar was charged with aryl bromides (3 mmol), 25 % ammonia aqueous solution (4 mL), Cu_2O (0.04 g, 0.3 mmol) and ligand L (0.47 g, 0.3 mmol). The resulting solution was stirred at 100 ℃ for 24 h. The reaction mixture was cooled to ambient temperature, suction filtration to recover the ligand L. The filtrate was diluted by 20 mL water and extracted with ethyl acetate (3×30 mL). The combined extracts was dried over $MgSO_4$, filtered and evaporated under vacuum to give the crude product, which was purified with petroleum ether /ethyl acetate or dichloromethane/methanol to give pure desired products.

3.4 Procedure for 4-Aminoacetophenone (6f)



6f

A 100 ml of flask with a magnetic stirring bar was charged with *p*-Bromoacetophenone (2.99 g, 15 mmol), Cu_2O (0.21 g, 1.50 mmol), ligand L (1.50 g, 1.5 mmol), Cs_2CO_3 (4.89 g, 15 mmol), 50 ml $NH_3 \cdot H_2O$ (25 %). The resulting solution was stirred at 100 ℃ for 24 h. The reaction mixture was cooled to room temperature and 50 ml H_2O was added, suction filtration to recover the ligand L. The filtrate was extracted with ethyl acetate (3×30 mL). The combined extracts was dried over $MgSO_4$, filtered and evaporated under vacuum to give the crude product, which was purified with petroleum ether /ethyl acetate to give compound **6f** as a white solid (1.82 g; yield: 90 %). The ligand L was washed with water and ethyl acetate, and reusing under as is condition for the next catalytic cycle.

4. A possible reaction mechanism

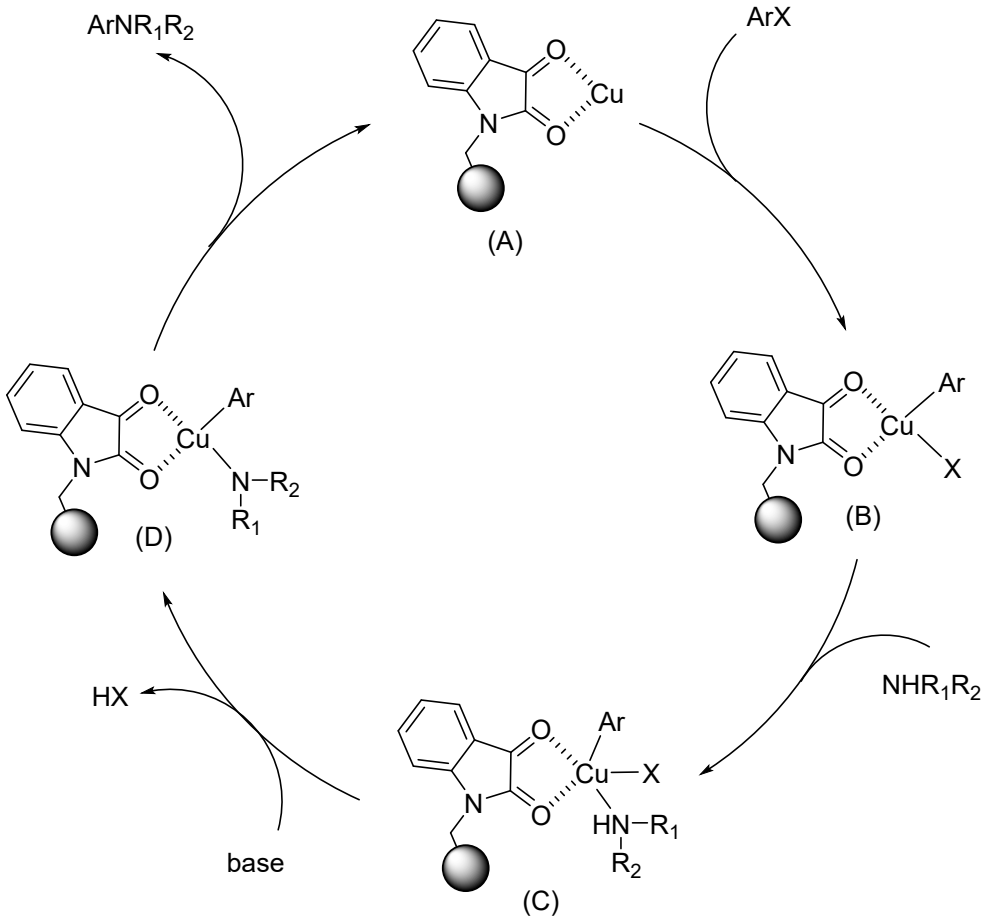
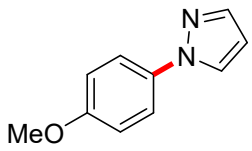


Figure S4. Possible Cu(I)/Cu(III) catalytic cycle mechanism for the Ullmann coupling reaction

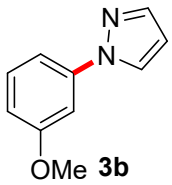
Furthermore, we proposed a possible reaction mechanism to explain the copper-catalyzed coupling reaction. As described in **Figure S3**, the cross-coupling reaction might go through a prototypical Cu(I)/Cu(III) catalytic cycle *via* an oxidative addition and reductive elimination pathway. Firstly, the coordination of the Ketones carbonyl on the isatin with the Cu(I) center formed the reactive species A, and then the chelating aryl halides to afford complexes B via oxidative addition process. Secondly, the complexes B react with amine to afford intermediate C under the action of base. Finally, the desired product was obtained by reductive elimination process and release the reactive species A ready to re-enter the next catalytic cycle.

5. Characterization



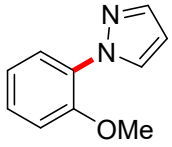
3a

1-(4-Methoxyphenyl)-1*H*-pyrazole (3a): Colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *j* = 2.4 Hz, 1H), 7.70 (d, *j* = 1.8 Hz, 1H), 7.62 – 7.57 (m, 2H), 6.98 (d, *j* = 9.0 Hz, 2H), 6.47 – 6.41 (m, 1H), 3.85 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [1]



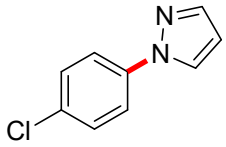
3b

1-(3-Methoxyphenyl)-1*H*-pyrazole (3b): Colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *j* = 2.5 Hz, 1H), 7.72 (d, *j* = 1.8 Hz, 1H), 7.36 – 7.30 (m, 2H), 7.27 – 7.21 (m, 1H), 6.83 (dd, *j* = 8.7, 3.0 Hz, 1H), 6.48 – 6.43 (m, 1H), 3.87 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [2]



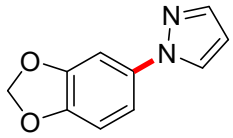
3c

1-(2-Methoxyphenyl)-1*H*-pyrazole (3c): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *j* = 2.4 Hz, 1H), 7.74 – 7.68 (m, 2H), 7.32 – 7.24 (m, 1H), 7.09 – 7.01 (m, 2H), 6.42 (d, *j* = 2.1 Hz, 1H), 3.87 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [3]



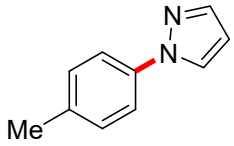
3d

1-(4-Chlorophenyl)-1*H*-imidazole (3d): white solid; m. p. 85-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *j* = 2.5 Hz, 1H), 7.72 (d, *j* = 1.9 Hz, 1H), 7.69 – 7.60 (m, 2H), 7.46 – 7.39 (m, 2H), 6.50 – 6.45 (m, 1H); ESI-MS (*m/z*): 179.1 $[M+H]^+$. [4]



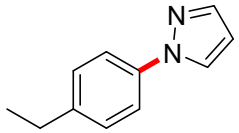
3e

1-(Benzo[d][1,3]dioxol-5-yl)-1*H*-imidazole (3e): white solid; m. p. 78-80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *j* = 2.4 Hz, 1H), 7.68 (d, *j* = 2.3 Hz, 1H), 7.22 (d, *j* = 2.2 Hz, 1H), 7.10 (dd, *j* = 8.4, 2.2 Hz, 1H), 6.85 (d, *j* = 8.3 Hz, 1H), 6.45 – 6.41 (m, 1H), 6.02 (s, 2H); ESI-MS (*m/z*): 189.0 $[M+H]^+$. [2]



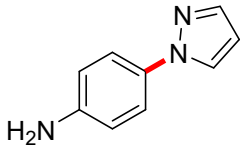
3f

1-*p*-Tolyl-1*H*-pyrazole (3f): white oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *j* = 2.5 Hz, 1H), 7.70 (d, *j* = 1.8 Hz, 1H), 7.56 (d, *j* = 8.5 Hz, 2H), 7.23 (d, *j* = 7.8 Hz, 2H), 6.44 – 6.41 (m, 1H), 2.36 (s, 3H); ESI-MS (*m/z*): 159.1 $[M+H]^+$. [4]



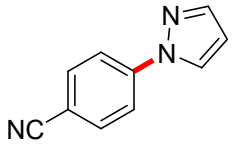
3g

1-(4-Ethylphenyl)-1*H*-pyrazole (3g):White solid;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *j* = 2.4 Hz, 1H), 7.71 (d, *j* = 1.7 Hz, 1H), 7.60 (d, *j* = 8.5 Hz, 2H), 7.28 (d, *j* = 8.5 Hz, 2H), 6.45 (t, *j* = 2.1 Hz, 1H), 2.69 (q, *j* = 7.6 Hz, 2H), 1.26 (t, *j* = 7.6 Hz, 3H);ESI-MS (*m/z*): 173.1 $[M+H]^+$;m. p. 37-40 °C. [5]



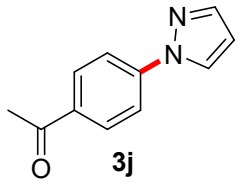
3h

4-(1*H*-Pyrazol-1-yl)aniline (3h): White solid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.17 (d, *j* = 2.4 Hz, 1H), 7.59 (d, *j* = 1.8 Hz, 1H), 7.47 – 7.38 (m, 2H), 6.69 – 6.59 (m, 2H), 6.46 – 6.36 (m, 1H), 5.15 (s, 2H);ESI-MS (*m/z*): 160.1 $[M+H]^+$;m. p. 41-42 °C. [6]



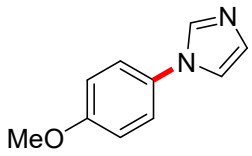
3i

(4-Cyanophenyl)-1*H*-pyrazole (3i):White solid; m. p. 89-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *j* = 2.5 Hz, 1H), 7.87 – 7.82 (m, 2H), 7.78 (d, *j* = 1.8 Hz, 1H), 7.77 – 7.73 (m, 2H), 6.54 (dd, *j* = 2.6, 1.7 Hz, 1H);ESI-MS (*m/z*): 170.1 $[M+H]^+$.[4]



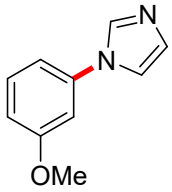
3j

1-[4-(1*H*-Pyrazol-1-yl)phenyl]ethanone (3j): White solid; m. p. 107-108 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *j* = 8.8 Hz, 2H), 8.02 (d, *j* = 2.6 Hz, 1H), 7.81 (s, 2H), 7.77 (d, *j* = 1.7 Hz, 1H), 6.54 – 6.50 (m, 1H), 2.63 (s, 3H); ESI-MS (*m/z*): 187.1 $[M + H]^+$,209.0 $[M+ Na]^+$.[4]



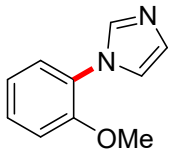
3k

1-(4-Methoxyphenyl)-1*H*-imidazole (3k): White solid; m. p. 60-61 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.33 – 7.27 (m, 2H), 7.19 (d, *j* = 7.3 Hz, 2H), 7.01 – 6.95 (m, 2H), 3.85 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [7]



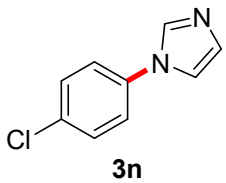
3l

1-(3-Methoxyphenyl)-1*H*-imidazole (3l): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.38 (t, *j* = 8.4 Hz, 1H), 7.27 (s, 1H), 7.19 (s, 1H), 6.97 (d, *j* = 8.2 Hz, 1H), 6.93 – 6.88 (m, 2H), 3.86 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [8]



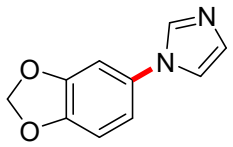
3m

1-(2-Methoxy-phenyl)-1*H*-imidazole(3m): Colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 1H), 7.38 – 7.32 (m, 1H), 7.29 – 7.26 (m, 1H), 7.19 (s, 1H), 7.16 (s, 1H), 7.07 – 7.00 (m, 2H), 3.84 (s, 3H); ESI-MS (*m/z*): 175.1 $[M+H]^+$. [9]



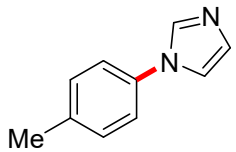
3n

1-(4-Chlorophenyl)-1*H*-imidazole (3n): White solid; m. p. 84-85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.46 (d, *j* = 8.8 Hz, 2H), 7.38 – 7.31 (m, 2H), 7.26 (s, 1H), 7.23 (s, 1H); ESI-MS (*m/z*): 179.0 $[M+H]^+$. [4]



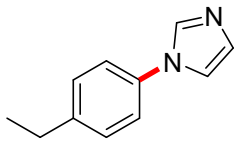
3o

1-(1,3-Benzodioxol-5-yl)-1*H*-imidazole (3o): Brown solid; m. p. 81-83 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.18 (s, 2H), 6.89 – 6.82 (m, 3H), 6.05 (s, 2H); ESI - MS (*m/z*): 189.0 $[M+H]^+$. [2]



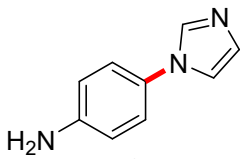
3p

1-*p*-Tolyl-1*H*-imidazole (3p): White solid; m. p. 45-46 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.20 (s, 1H), 7.68 (t, *j* = 1.4 Hz, 1H), 7.51 (d, *j* = 8.4 Hz, 2H), 7.30 (d, *j* = 8.3 Hz, 2H), 7.10 (s, 1H), 2.33 (s, 3H); ESI-MS (*m/z*): 159.1 $[M+H]^+$. [4]



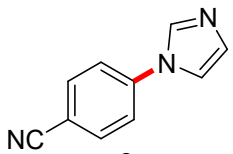
3q

1-(4-Ethylphenyl)-1*H*-imidazole (3q): Yellowish oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.20 (s, 1H), 7.69 (t, *j* = 1.3 Hz, 1H), 7.57 – 7.50 (m, 2H), 7.33 (d, *j* = 8.5 Hz, 2H), 7.09 (s, 1H), 2.63 (q, *j* = 7.6 Hz, 2H), 1.19 (t, *j* = 7.6 Hz, 3H); ESI-MS (*m/z*): 173.1 $[M+H]^+$. [5]



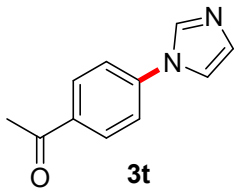
3r

4-(1*H*-Imidazol-1-yl)aniline (3r): White solid; m. p. 142-143 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.95 (s, 1H), 7.48 (s, 1H), 7.21 (d, *j* = 8.7 Hz, 2H), 7.01 (s, 1H), 6.64 (d, *j* = 8.7 Hz, 2H), 5.26 (s, 2H); ESI-MS (*m/z*): 160.1 $[M+H]^+$. [10]



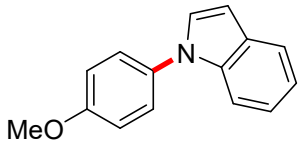
3s

4-(1*H*-Imidazol-1-yl)benzonitrile (3s): White solid; m. p. 152-154 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.46 (s, 1H), 8.05 – 7.99 (m, 2H), 7.95 – 7.90 (m, 3H), 7.16 (s, 1H);ESI-MS (*m/z*): 170.1 $[M+H]^+$. [4]



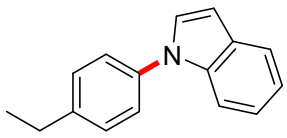
3t

1-[4-(1*H*-Imidazol-1-yl)phenyl]ethanone (3t) :White solid; m. p. 112-114 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.43 (s, 1H), 8.08 (d, *j* = 8.7 Hz, 2H), 7.89 (t, *j* = 1.4 Hz, 1H), 7.87 – 7.82 (m, 2H), 7.15 (s, 1H), 2.61 (s, 3H); ESI-MS (*m/z*): 187.1 $[M+H]^+$. [7]



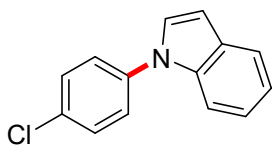
3u

1-(4-Methoxyphenyl)-1*H*-indole (3u) :White solid; m. p. 59-61 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *j* = 7.8 Hz, 1H), 7.46 (d, *j* = 8.1 Hz, 1H), 7.41 (d, *j* = 8.9 Hz, 2H), 7.29 (d, *j* = 3.2 Hz, 1H), 7.24 – 7.14 (m, 2H), 7.04 (d, *j* = 8.9 Hz, 2H), 6.66 (d, *j* = 4.0 Hz, 1H), 3.89 (s, 3H). [11]



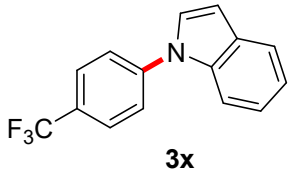
3v

1-(4-Ethylphenyl)-1*H*-indole (3v) : Colorless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.65 (d, *j* = 7.6 Hz, 1H), 7.61 (d, *j* = 3.2 Hz, 1H), 7.52 (dd, *j* = 8.2, 1.0 Hz, 1H), 7.51 – 7.46 (m, 2H), 7.41 (d, *j* = 8.5 Hz, 2H), 7.20 – 7.16 (m, 1H), 7.11 (ddd, *j* = 8.0, 7.0, 1.1 Hz, 1H), 6.68 (dd, *j* = 3.2, 0.8 Hz, 1H), 2.69 (q, *j* = 7.6 Hz, 2H), 1.25 (d, *j* = 7.6 Hz, 3H). [12]



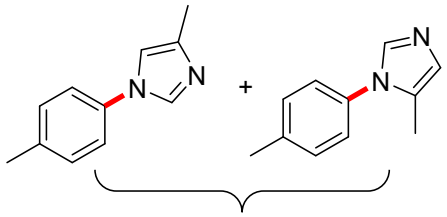
3w

1-(4-Chlorophenyl)-1*H*-indole (3w):White solid; m. p. 64-66 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.68 – 7.65 (m, 2H), 7.63 (s, 4H), 7.56 (d, *j* = 9.2 Hz, 1H), 7.24 – 7.19 (m, 1H), 7.14 (t, *j* = 6.9 Hz, 1H), 6.72 (d, *j* = 3.3 Hz, 1H). [13]



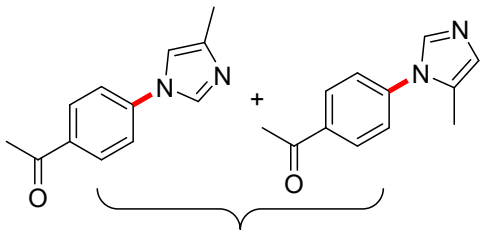
3x

1-[4-(Trifluoromethyl)phenyl]-1*H*-indole (3x): White solid; m. p. 45-47 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.94 (d, *j* = 8.6 Hz, 2H), 7.86 (d, *j* = 8.5 Hz, 2H), 7.77 (d, *j* = 3.3 Hz, 1H), 7.68 (ddd, *j* = 8.3, 2.6, 1.2 Hz, 2H), 7.25 (ddd, *j* = 8.4, 7.0, 1.3 Hz, 1H), 7.21 – 7.15 (m, 1H), 6.78 (dd, *j* = 3.4, 0.8 Hz, 1H). [14]



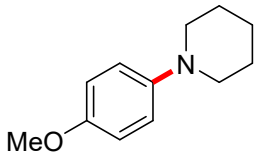
3y (14:1)

4-Methyl-1-(4-methylphenyl)-1*H*-imidazole and 5-Methyl-1-(4-methylphenyl)-1*H*-imidazole(3y): Colorless oil; ^{1}H NMR (400 MHz, DMSO-*d6*) δ 8.06 (s, 1H), 7.46 (d, J = 8.5 Hz, 2H), 7.37 (s, 1H), 7.28 (d, J = 6.4 Hz, 2H), 2.33 (s, 3H), 2.15 (s, 3H); ESI-MS (*m/z*): 173.16 $[M+H]^+$. [15]



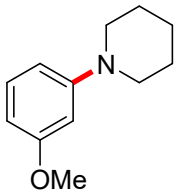
3Z (6:1)

1-[4-(4-Methyl-1*H*-imidazol-1-yl)phenyl]ethanone and 1-(4-(5-methyl-1*H*-imidazol-1-yl)phenyl)ethan-1-one(3y): Brown solid;1*H* NMR (400 MHz, DMSO-*d6*) δ 8.30 (d, J = 1.5 Hz, 1H), 8.05 (d, J = 8.7 Hz, 2H), 7.77 (d, J = 8.8 Hz, 2H), 7.57 (s, 1H), 2.59 (s, 3H), 2.17 (s, 3H); ESI-MS (m/z): 201.15 $[M+H]^+$. [16]



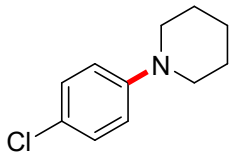
5a

1-(4-Methoxyphenyl)piperidine (5a) : Colorless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.89 – 6.84 (m, 2H), 6.82 – 6.77 (m, 2H), 3.67 (s, 3H), 3.00 – 2.92 (m, 4H), 1.66 – 1.57 (m, 4H), 1.49 (q, j = 5.5 Hz, 2H); ESI-MS (*m/z*): 192.1 $[M+H]^+$.[1]



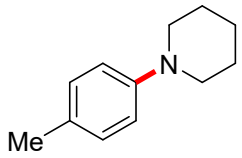
5b

1-(3-Methoxyphenyl)piperidine (5b): Colorless oil;^{1}H NMR (400 MHz, DMSO-d_6) δ 6.95 – 6.82 (m, 4H), 3.76 (s, 3H), 2.91 – 2.85 (m, 4H), 1.62 (p, j = 5.8 Hz, 4H), 1.54 – 1.46 (m, 2H); ESI-MS (*m/z*): 192.1 $[M+H]^+$. [17]



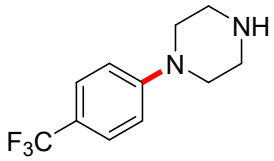
5c

1-(4-Chlorophenyl)piperidine (5c): White solid; m. p. 208-209 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, j = 8.3 Hz, 2H), 6.89 (s, 2H), 3.13 (s, 4H), 1.58 (s, 6H); ESI-MS (*m/z*): 196.1 $[M+H]^+$. [18]



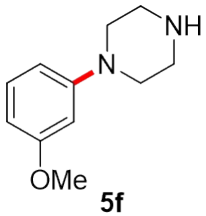
5d

1-(4-Methylphenyl)piperidine (5d): White solid; m. p. 267-268 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.99 (d, j = 7.9 Hz, 2H), 6.83 – 6.77 (m, 2H), 3.08 – 2.99 (m, 4H), 2.18 (s, 3H), 1.60 (p, j = 5.5 Hz, 4H), 1.53 – 1.47 (m, 2H); ESI-MS (*m/z*): 176.1 $[M+H]^+$. [19]



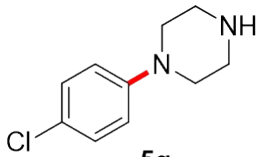
5e

1-[4-(Trifluoromethyl)phenyl]piperazine (5e): White solid; m. p. >300 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.48 (d, j = 8.8 Hz, 2H), 7.03 (d, j = 8.7 Hz, 2H), 3.20 – 3.15 (m, 4H), 2.86 – 2.79 (m, 4H); ESI-MS (m/z): 176.1 $[M+H]^+$. [20]



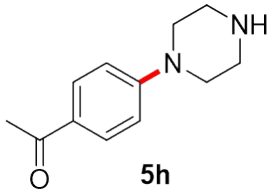
5f

1-(3-Methoxyphenyl)piperazine (5f): White solid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.09 (t, j = 8.2 Hz, 1H), 6.49 (dd, j = 8.2, 2.3 Hz, 1H), 6.41 (t, j = 2.4 Hz, 1H), 6.34 (dd, j = 8.0, 2.4 Hz, 1H), 3.70 (s, 3H), 3.02 (t, j = 3.6 Hz, 4H), 2.84 – 2.79 (m, 4H), 1.23 (s, 1H); ESI-MS (m/z): 173.1 $[M+H]^+$. [21]



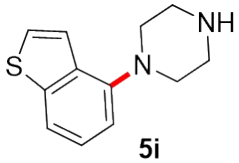
5g

1-(4-Chlorophenyl)piperazine (5g): White solid; m. p. 71-73 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.22 (d, j = 9.0 Hz, 2H), 6.92 (d, j = 8.8 Hz, 2H), 3.05 (t, j = 5.1 Hz, 4H), 2.84 (s, 4H), 1.23 (d, j = 3.0 Hz, 1H);ESI-MS (m/z): 197.1 $[M+H]^+$. [21]



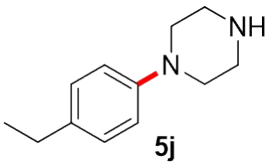
5h

1-(4-Acetylphenyl)piperazine (5h): White solid; m. p. 95-96 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.82 – 7.76 (m, 2H), 6.96 – 6.91 (m, 2H), 3.25 – 3.21 (m, 4H), 2.83 – 2.78 (m, 4H), 2.44 (s, 3H); ESI-MS (m/z): 205.2 $[M+H]^+$. [22]



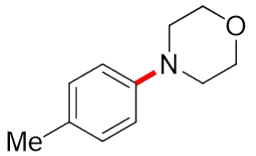
5i

1-Benzo[b]thien-4-ylpiperazine (5i): yellow solid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.68 (d, j = 5.5 Hz, 1H), 7.60 (d, j = 8.0 Hz, 1H), 7.41 (d, j = 5.5 Hz, 1H), 7.27 (t, j = 7.9 Hz, 1H), 6.88 (d, j = 7.6 Hz, 1H), 2.97 (d, j = 11.2 Hz, 8H), 1.23 (s, 1H); ESI-MS (m/z): 219.1 $[M+H]^+$. [23]



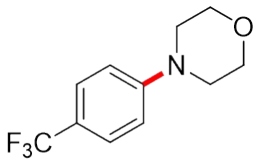
5j

1-(4-Ethylphenyl)piperazine (5j): Pale yellow solid; m. p. 74-76 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, j = 8.6 Hz, 2H), 6.87 (d, j = 8.6 Hz, 2H), 3.18 – 3.11 (m, 4H), 3.07 (d, j = 3.9 Hz, 4H), 2.58 (q, j = 7.6 Hz, 2H), 1.21 (d, j = 15.0 Hz, 3H); ESI-MS (m/z): 191.2 $[M+H]^+$. [24]



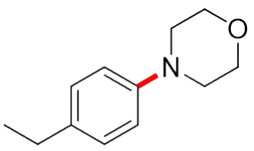
5k

4-(4-Methoxyphenyl)morpholine (5k): White solid; m. p. 73-74 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.03 (d, j = 8.4 Hz, 2H), 6.83 (d, j = 8.6 Hz, 2H), 3.74 – 3.70 (m, 4H), 3.05 – 3.00 (m, 4H), 2.20 (s, 3H); ESI-MS (m/z): 178.1 $[M+H]^+$. [17]



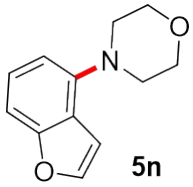
5l

4-(4-Trifluoromethylphenyl)morpholine (5i): White solid; m. p. 57-58 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.52 (d, j = 8.7 Hz, 2H), 7.07 (d, j = 8.7 Hz, 2H), 3.76 – 3.71 (m, 4H), 3.25 – 3.20 (m, 4H); ESI-MS (m/z): 219.1 $[M+H]^+$. [25]



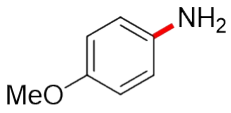
5m

4-(4-Ethylphenyl)morpholine (5m): White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *j* = 8.1 Hz, 2H), 6.92 (s, 2H), 3.89 (s, 4H), 3.15 (s, 4H), 2.59 (q, *j* = 6.6, 5.7 Hz, 2H), 1.22 (d, *j* = 7.6 Hz, 3H); ESI-MS (*m/z*): 192.2 $[M+H]^+$. [26]



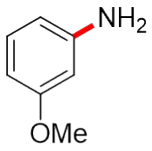
5n

4-(4-Benzofuranyl)morpholine (5n): m. p. 98-100 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (d, *j* = 2.2 Hz, 1H), 7.45 (d, *j* = 9.0 Hz, 1H), 7.12 (d, *j* = 2.5 Hz, 1H), 7.02 (dd, *j* = 9.0, 2.5 Hz, 1H), 6.83 (d, *j* = 2.2 Hz, 1H), 3.81 – 3.71 (m, 4H), 3.12 – 3.03 (m, 4H); ESI-MS (*m/z*): 204.1 $[M+H]^+$. [27]



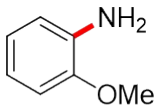
6a

4-Methoxybenzenamine (6a): Brown solid; m. p. 57-58 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.66 – 6.61 (m, 2H), 6.53 – 6.48 (m, 2H), 4.57 (s, 2H), 3.61 (s, 3H); ESI-MS (*m/z*): 124.2 $[M+H]^+$. [8]



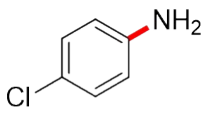
6b

3-Methoxybenzenamine (6a): Brown oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.89 (t, *j* = 8.1 Hz, 1H), 6.14 (dd, *j* = 7.4, 1.1 Hz, 2H), 6.07 (dd, *j* = 8.6, 2.8 Hz, 1H), 5.02 (s, 2H), 3.64 (s, 3H); ESI-MS (*m/z*): 124.1 $[M+H]^+$. [28]



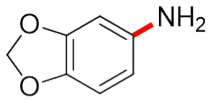
6c

2-Methoxybenzenamine (6c): Brown oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.77 (dd, *j* = 8.0, 1.3 Hz, 1H), 6.69 – 6.60 (m, 2H), 6.54 – 6.49 (m, 1H), 4.64 (s, 2H), 3.74 (s, 3H); ESI-MS (*m/z*): 124.1$[M+H]^+$. [28]



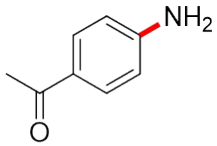
6d

4-Chloroaniline (6d): White solid; m.p. 72-73 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.03 – 6.98 (m, 2H), 6.57 – 6.52 (m, 2H), 5.24 (s, 2H); ESI-MS (*m/z*): 128.1 $[M+H]^+$. [8]



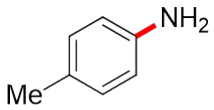
6e

1,3-Benzodioxol-5-amine (6e): Grey solid; m. p. 44-46 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ 6.57 (d, *j* = 8.2 Hz, 1H), 6.22 (d, *j* = 2.2 Hz, 1H), 5.99 (dd, *j* = 8.2, 2.2 Hz, 1H), 5.80 (s, 2H), 4.73 (s, 2H); ESI-MS (*m/z*): 138.1 $[M+H]^+$. [28]



6f

4-Aminoacetophenone (6f): White solid; m. p. 106-107 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.78 (m, 2H), 6.69 – 6.61 (m, 2H), 4.10 (s, 2H), 2.51 (s, 3H); ESI-MS (*m/z*): 136.1 $[M+H]^+$. [29]



6g

4-Methylaniline (6g): White solid; m. p. 44-45 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.97 (d, *j* = 7.9 Hz, 2H), 6.62 (d, *j* = 8.4 Hz, 2H), 3.49 (s, 2H), 2.24 (s, 3H); ESI-MS (*m/z*): 108.2 $[M+H]^+$. [29]

6. References

[1] P. Hong, X. Zhu, X. Lai, Z. Gong, M. Huang and Y. Wan, *The Journal of Organic Chemistry*, 2024, **89**, 57-67.

[2] D. Song, Y. Qin, Y. Liu, J. Wang, H. Yang, Z. Zheng, F. Xu, X. Bao and G. Chen, *New Journal of Chemistry*, 2022, **46**, 19100-19103.

[3] S. Sinha, R. Sikari, V. Sinha, U. Jash, S. Das, P. Brandão, S. Demeshko, F. Meyer, B. de Bruin and N. D. Paul, *Inorganic Chemistry*, 2019, **58**, 1935-1948.

[4] G. Sarkar, N. Bandopadhyay, K. Paramanik, S. Saha, S. J. Panda, C. S. Purohit, B. Biswas and H. S. Das, *Molecular Catalysis*, 2023, **545**, 113212.

[5] S. N. Murthy, B. Madhav, V. P. Reddy and Y. V. D. Nageswar, *Advanced Synthesis & Catalysis*, 2010, **352**, 3241-3245.

[6] M. Taillefer, N. Xia and A. Ouali, *Angewandte Chemie International Edition*, 2007, **46**, 934-936.

[7] R. Lv, Y. Wang, C. Zhou, L. Li and R. Wang, *ChemCatChem*, 2013, **5**, 2978-2982.

[8] C. T. Yang, Y. Fu, Y. B. Huang, J. Yi, Q. X. Guo and L. Liu, *Angewandte Chemie International Edition*, 2009, **48**, 7398-7401.

[9] M. Guo, B. Chen, K. Chen, S. Guo, F.-S. Liu, C. Xu and H.-G. Yao, *Tetrahedron Letters*, 2022, **107**, 154074.

[10] J. Engel Andreasen, B. Shimpukade and T. Ulven, *Green Chemistry*, 2013, **15**, 336-340.

[11] C. A. Echeverry Gonzalez, M. C. Ortiz Villamizar and V. V. Kouznetsov, *New Journal of Chemistry*, 2021, **45**, 243-250.

[12] R. Saikia, P. K. Boruah, S. M. Ahmed, M. R. Das, A. J. Thakur and U. Bora, *Applied Catalysis A: General*, 2022, **643**, 118767.

[13] H. Sharghi, A. A. Saei and M. Aberi, *ChemistrySelect*, 2019, **4**, 13228-13234.

[14] K. Swapna, S. N. Murthy and Y. V. D. Nageswar, *European Journal of Organic Chemistry*, 2010, **2010**, 6678-6684.

[15] X. Q. Yu, Y. Yamamoto and N. Miyaura, *Chemistry – An Asian Journal*, 2008, **3**, 1517-1522.

[16] K. D. Rynearson, R. N. Buckle, R. J. Herr, N. J. Mayhew, X. Chen, W. D. Paquette, S. A. Sakwa, J. Yang, K. D. Barnes, P. Nguyen, W. C. Mobley, G. Johnson, J. H. Lin, R. E. Tanzi and S. L. Wagner, *Bioorganic & Medicinal Chemistry*, 2020, **28**, 115734.

[17] M. Kaloğlu and İ. Özdemir, *Catalysis Letters*, 2024, **154**, 1479-1494.

[18] J. C. Vantourout, H. N. Miras, A. Isidro-Llobet, S. Sproules and A. J. B. Watson, *Journal of the American Chemical Society*, 2017, **139**, 4769-4779.

[19] J. Tappen, I. Rodstein, K. McGuire, A. Großjohann, J. Löffler, T. Scherpf and V. H. Gessner, *Chemistry – A European Journal*, 2020, **26**, 4281-4288.

[20] S. W. Reilly and R. H. Mach, *Organic Letters*, 2016, **18**, 5272-5275.

[21] K. G. Liu and A. J. Robichaud, *Tetrahedron Letters*, 2005, **46**, 7921-7922.

[22] S. Bhunia, S. V. Kumar and D. Ma, *The Journal of Organic Chemistry*, 2017, **82**, 12603-12612.

[23] F. Y. Zhang, X. B. Lan, C. Xu, H. G. Yao, T. Li and F.-S. Liu, *Organic Chemistry Frontiers*, 2019, **6**, 3292-3299.

[24] Y. Huang, L. S. Kegeles, S. A. Bae, D.-r. Hwang, B. L. Roth, J. E. Savage and M. Laruelle, *Bioorganic & Medicinal Chemistry Letters*, 2001, **11**, 1375-1377.

[25] A. R. Martin, D. J. Nelson, S. Meiries, A. M. Z. Slawin and S. P. Nolan, *European Journal of Organic Chemistry*, 2014, **2014**, 3127-3131.

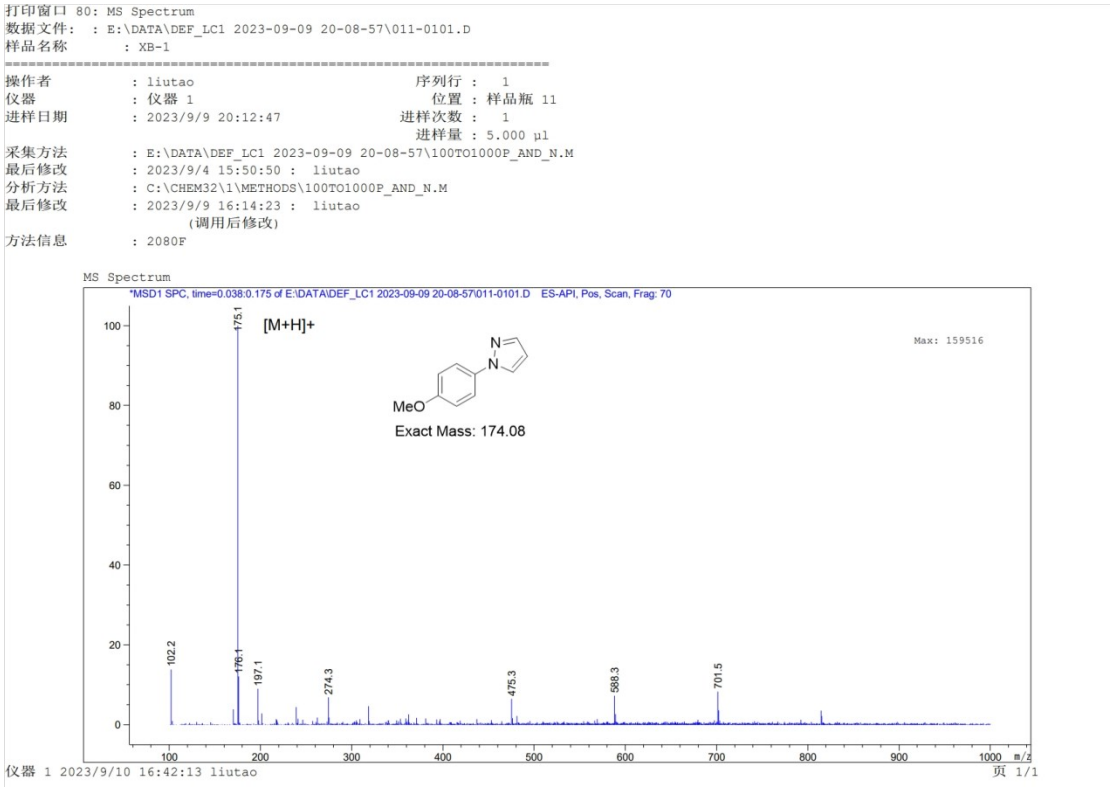
[26] H. Talukdar and P. Phukan, *ChemistrySelect*, 2024, **9**, e202304163.

[27] S. U. Dighe, F. Juliá, A. Luridiana, J. J. Douglas and D. Leonori, Nature, 2020, **584**, 75-81.

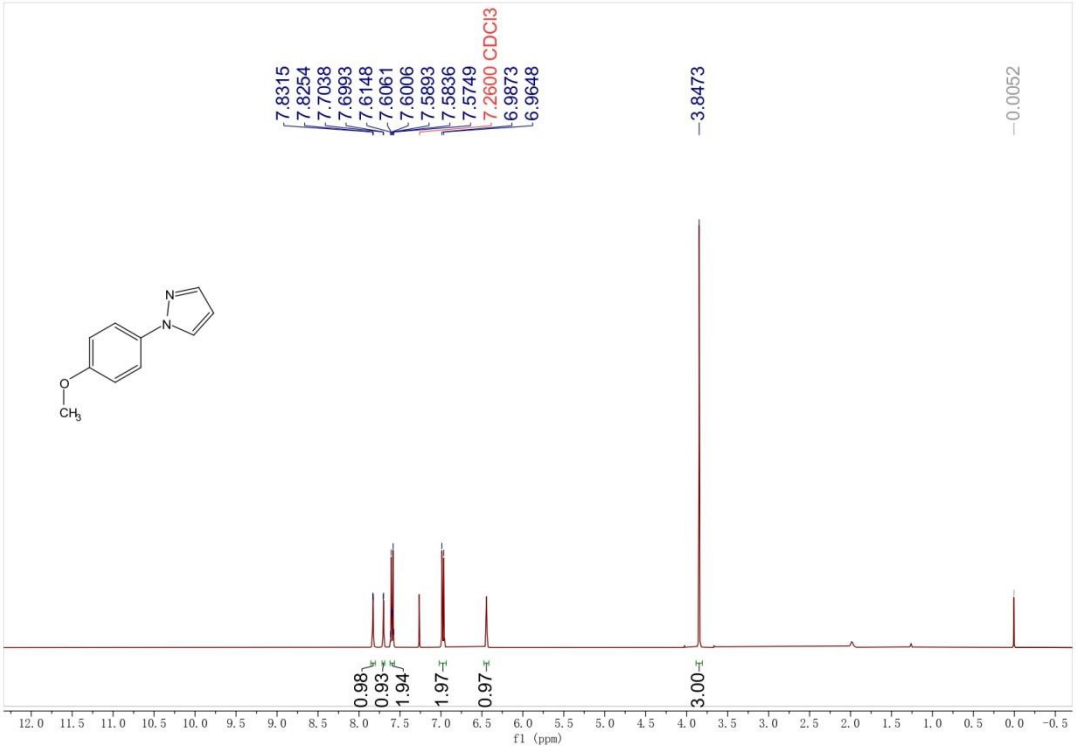
[28] B. Xue, J. Shen, S. Manna, A. Doppiu and L. J. Gooßen, Advanced Synthesis & Catalysis, 2023, **365**, 3473-3477.

[29] G. Zhang, L. Liu, Q. Zhu and X. Kong, *Catalysis Letters*, 2023, **153**, 1536-1542.

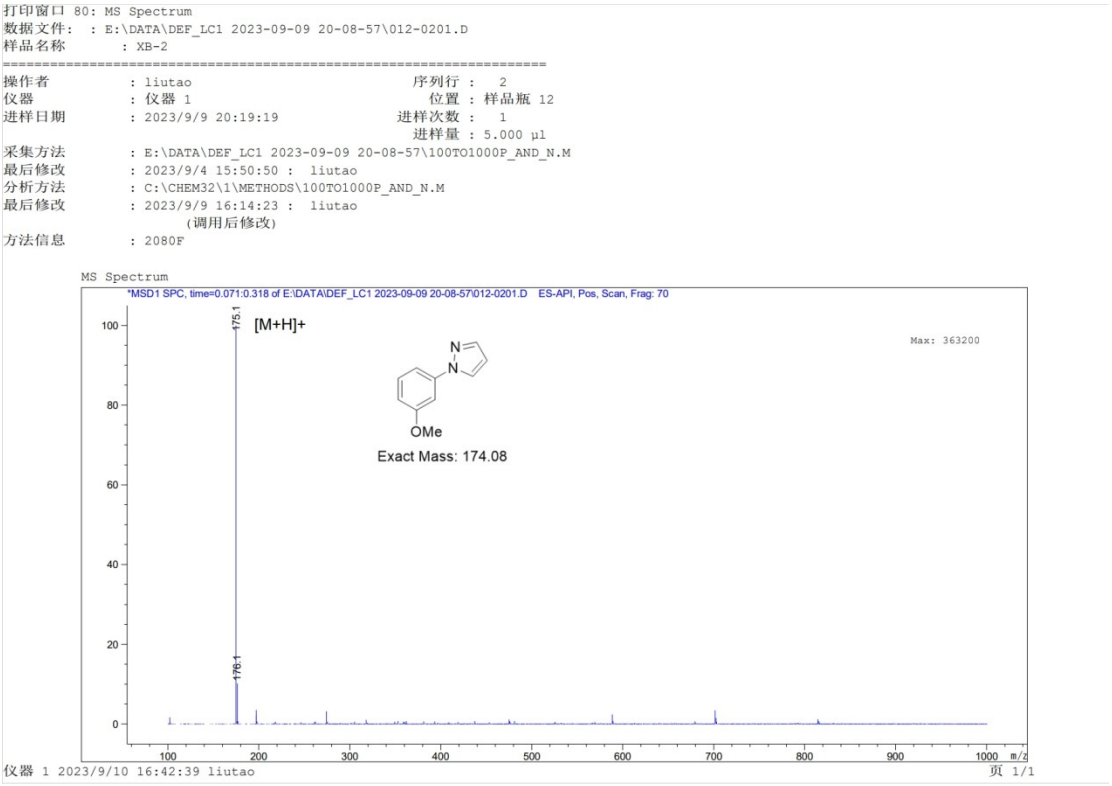
7. Copies of MS and ^{1}H spectrum of products



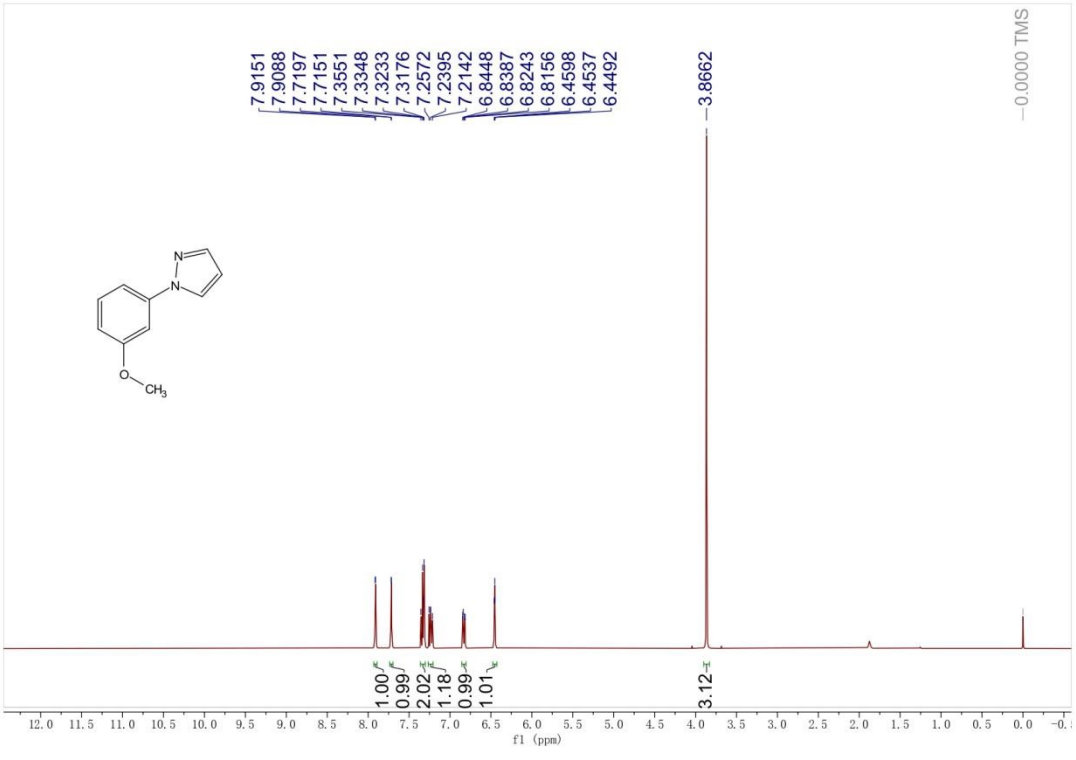
The MS spectrum of **3a**



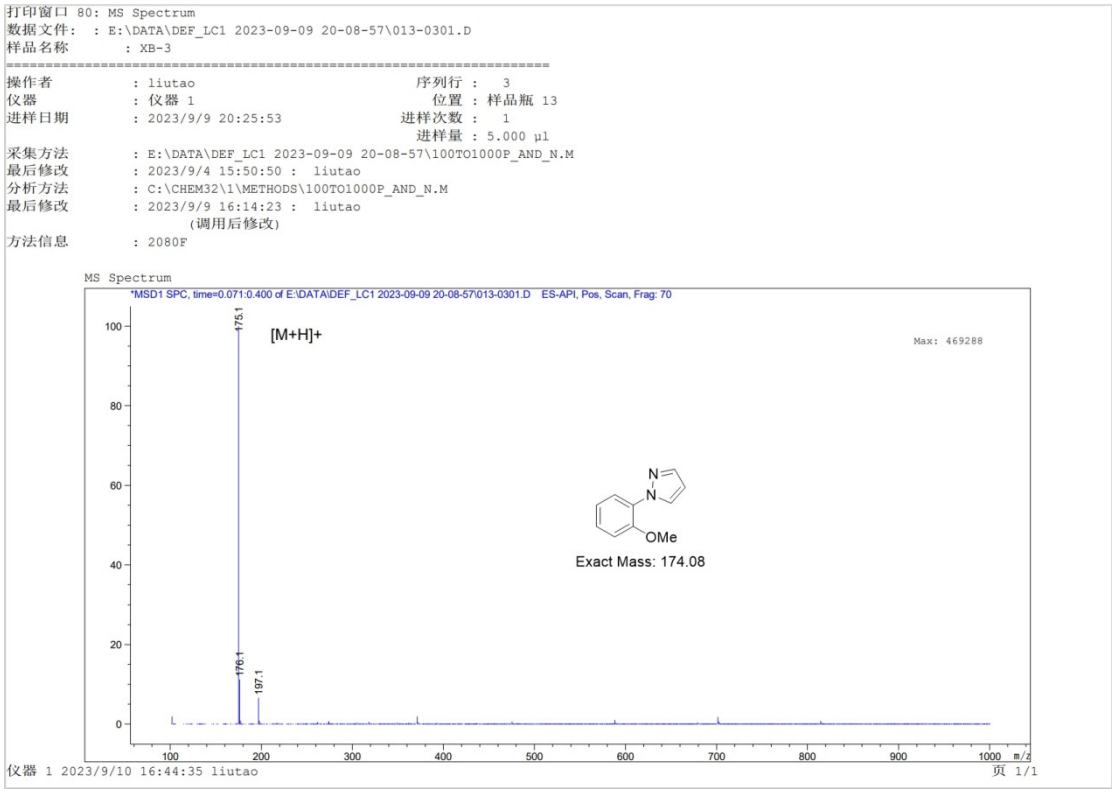
^{1}H NMR spectrum of **3a** (400 MHz, $CDCl_3$)



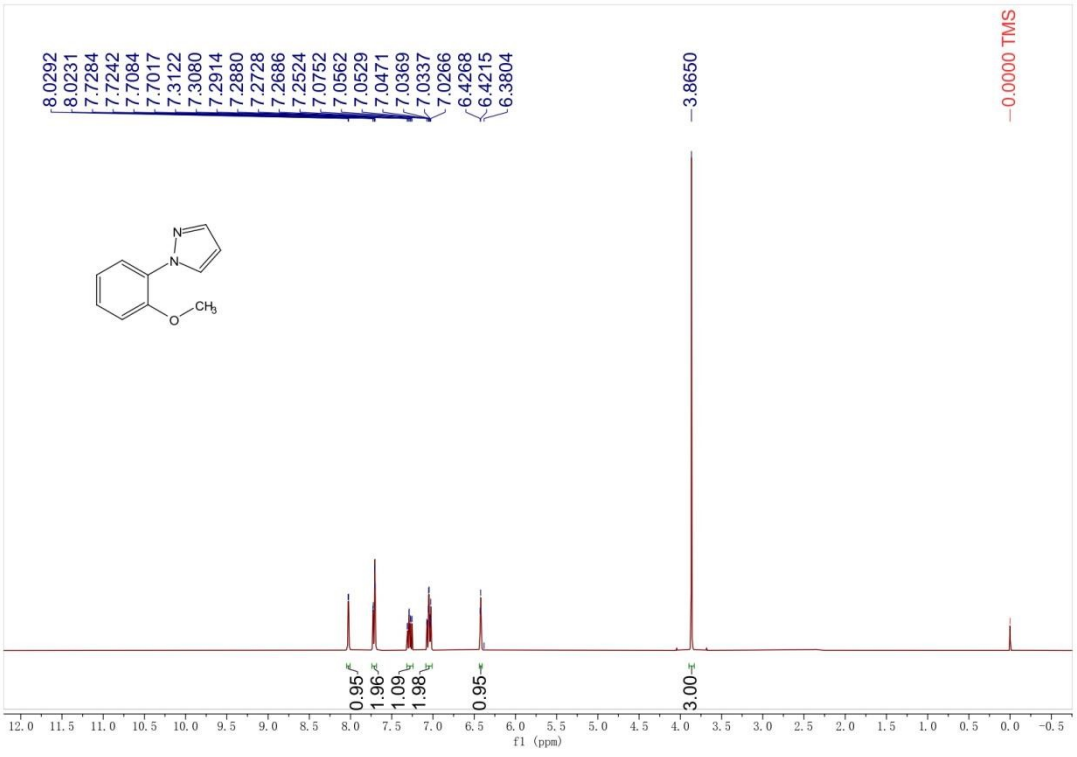
The MS spectrum of **3b**



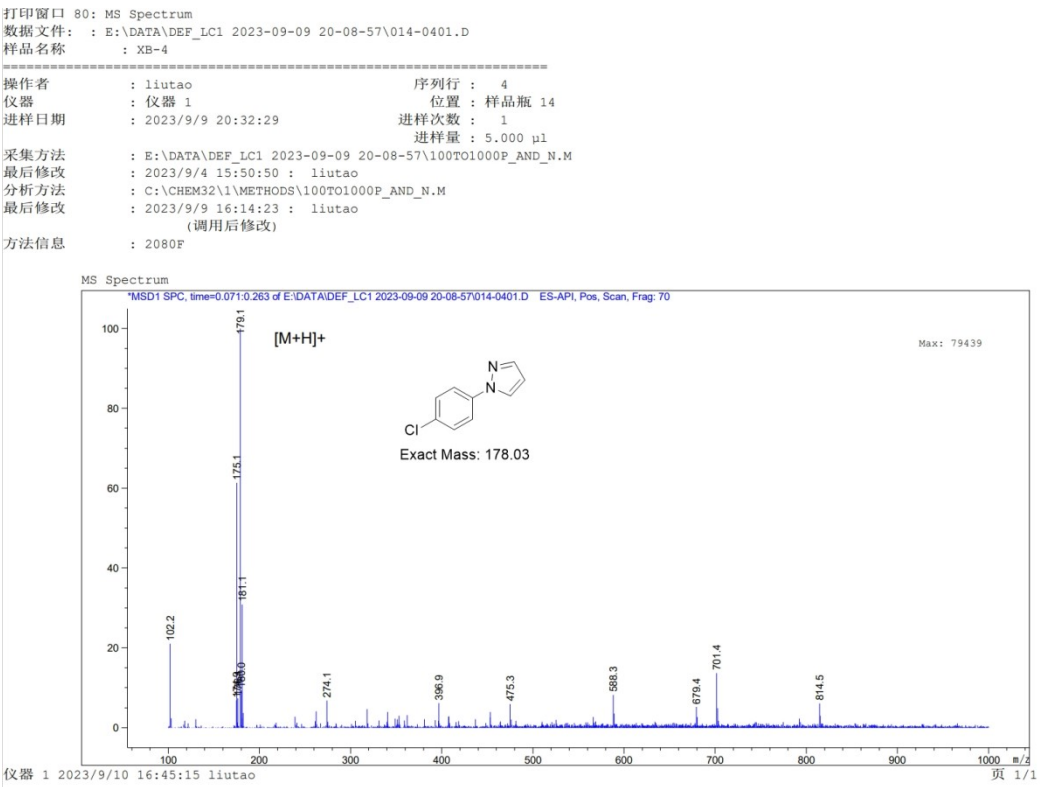
^{1}H NMR spectrum of **3b** (400 MHz, $CDCl_3$)



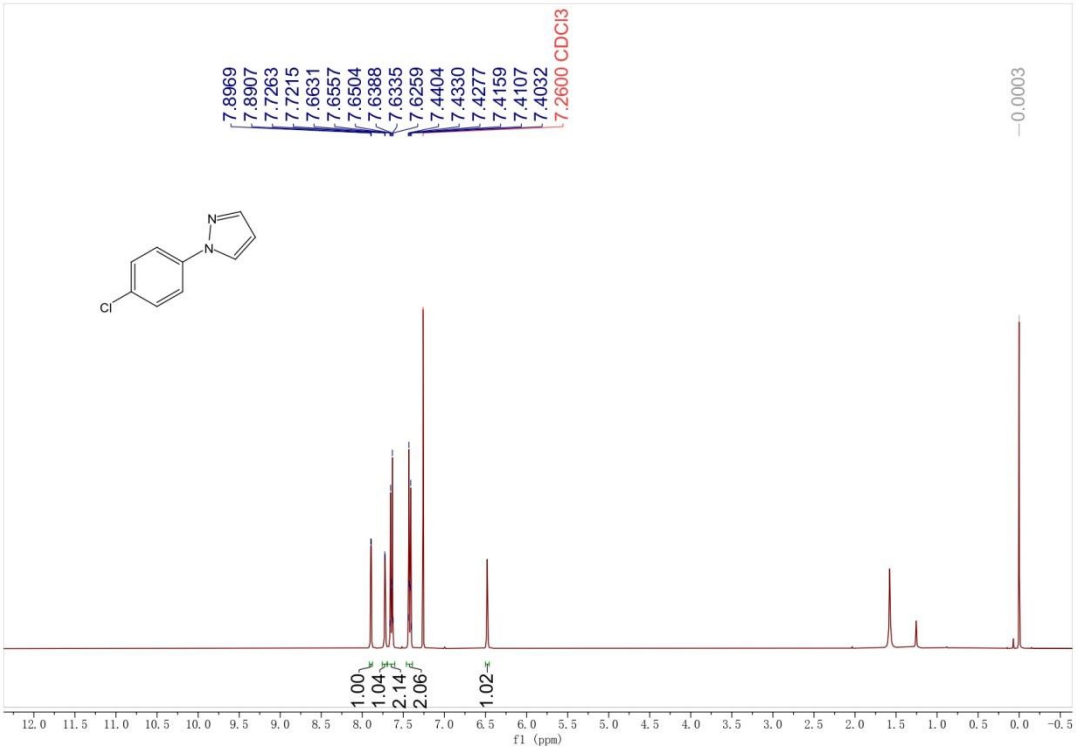
The MS spectrum of **3c**



^{1}H NMR spectrum of **3c** (400 MHz, $CDCl_3$)



The MS spectrum of **3d**



^{1}H NMR spectrum of **3d** (400 MHz, $CDCl_3$)

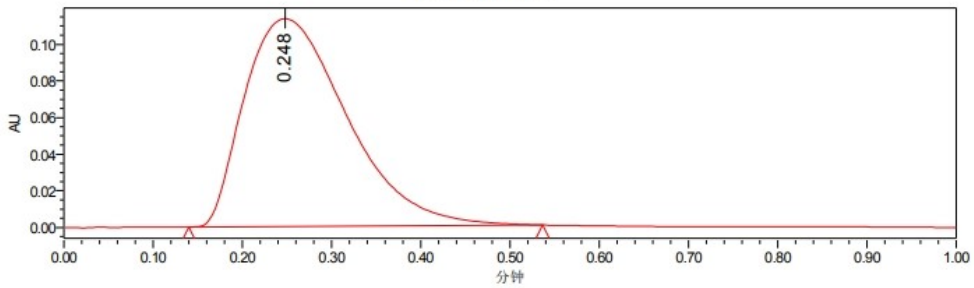
Empower 3 SOFTWARE

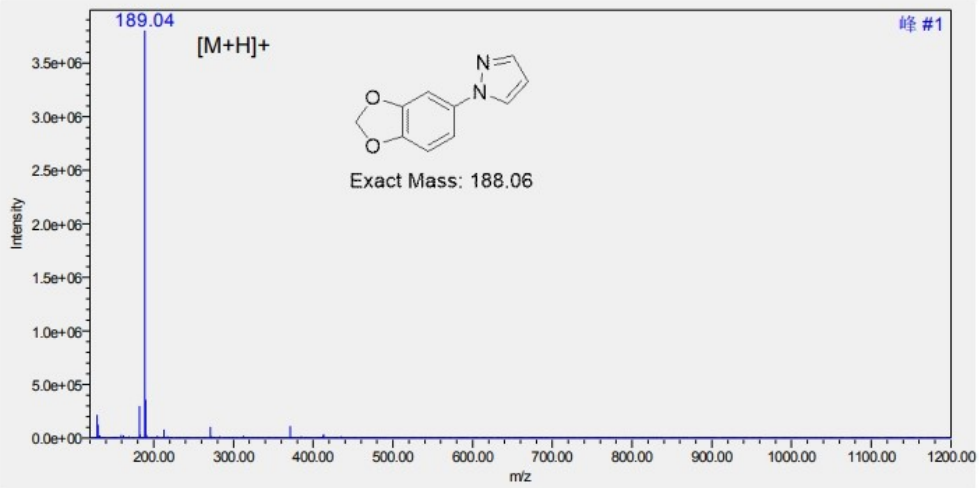
MS

样品信息

样品名称:	XB-5	采集者:	test1
样品类型:	标准样	样品组名称:	2023062802
瓶号:	2:D,6	采集方法组:	MS_ZHENG
进样次数:	1	处理方法:	MS_YY
进样体积:	15.00 ul	通道名称:	W2489 ChA
运行时间:	1.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2023/6/28 15:20:05 CST		
处理时间:	2023/6/28 15:21:12 CST		

自动缩放色谱图

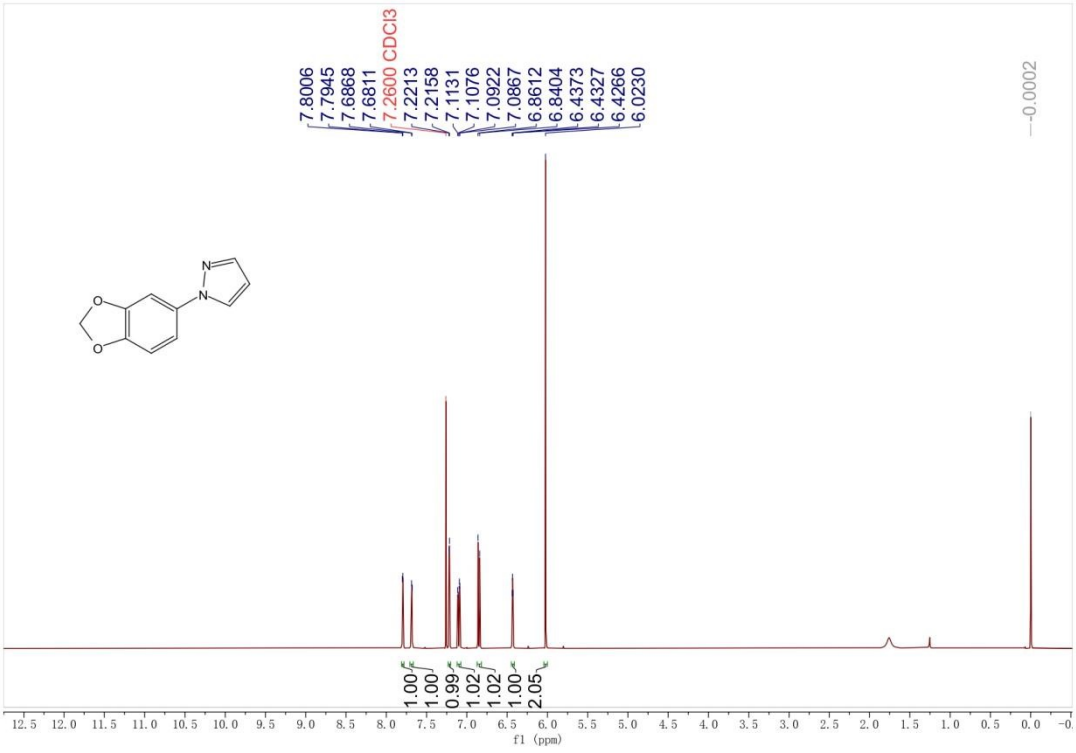




报告用户: test1
报告方法: MS
报告方法 ID: 358(358050
页码: 1 (共计 1)

项目名称: TEST1
打印日期:
2023/6/28
15:34:08 PRC

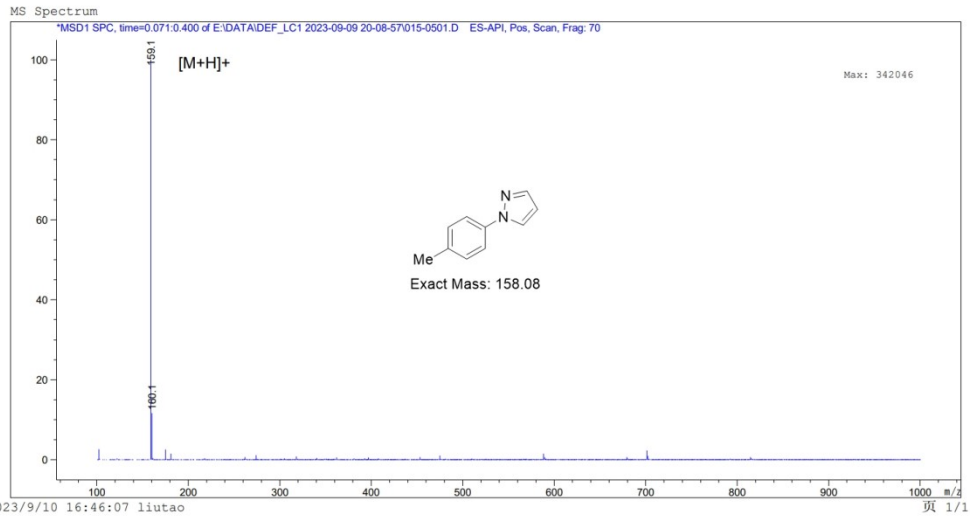
The MS spectrum of **3e**



^{1}H NMR spectrum of **3e** (400 MHz, $CDCl_3$)

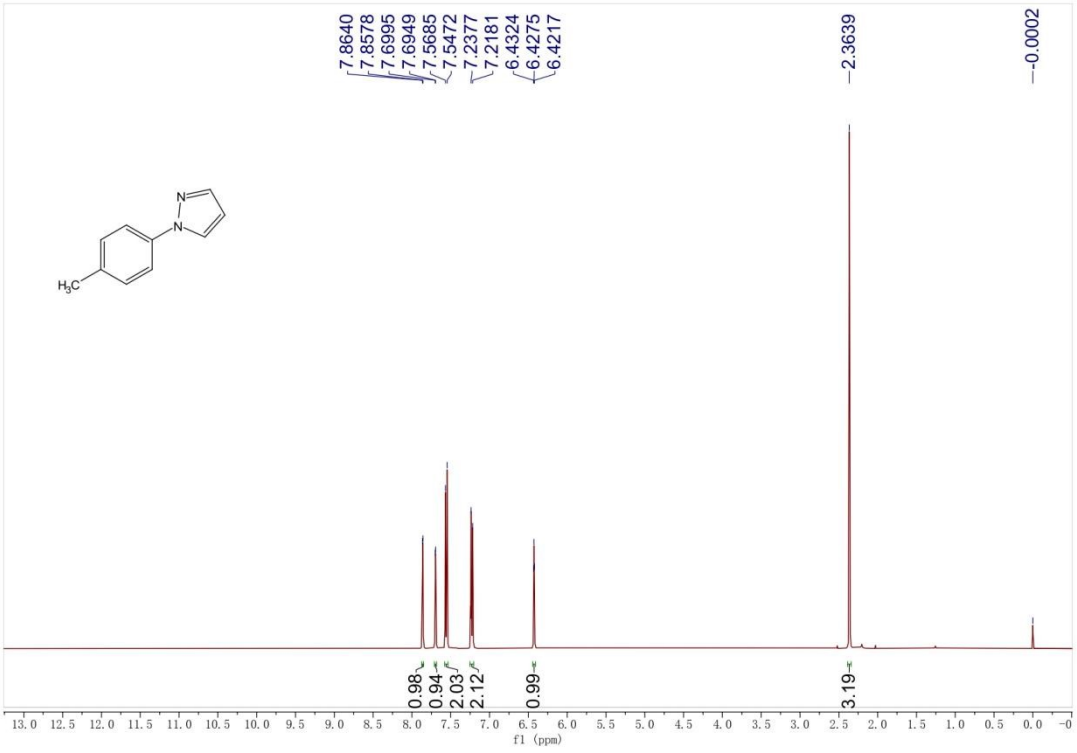
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-09-09 20-08-57\015-0501.D
样品名称 : XB-6

操作者 : liutao 序列行 : 5
仪器 : 仪器 1 位置 : 样品瓶 15
进样日期 : 2023/9/9 20:39:05 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-09-09 20-08-57\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/9/9 16:14:23 : liutao
(调用后修改)
方法信息 : 2080F

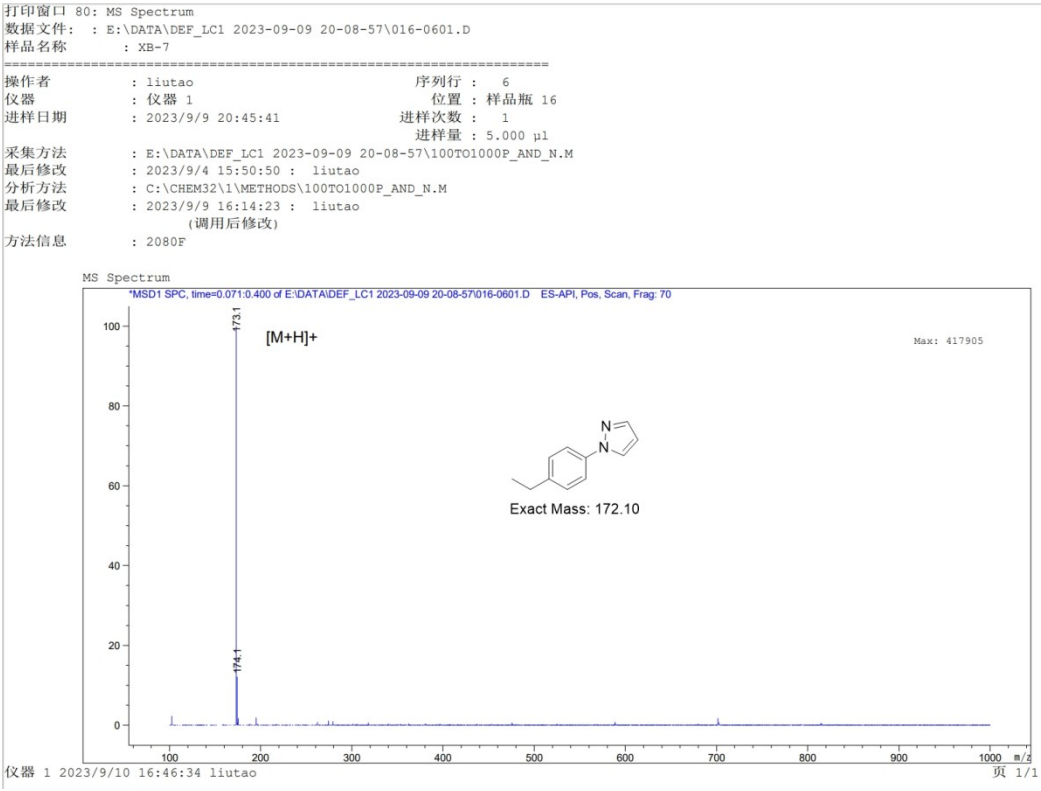


仪器 1 2023/9/10 16:46:07 liutao 页 1/1

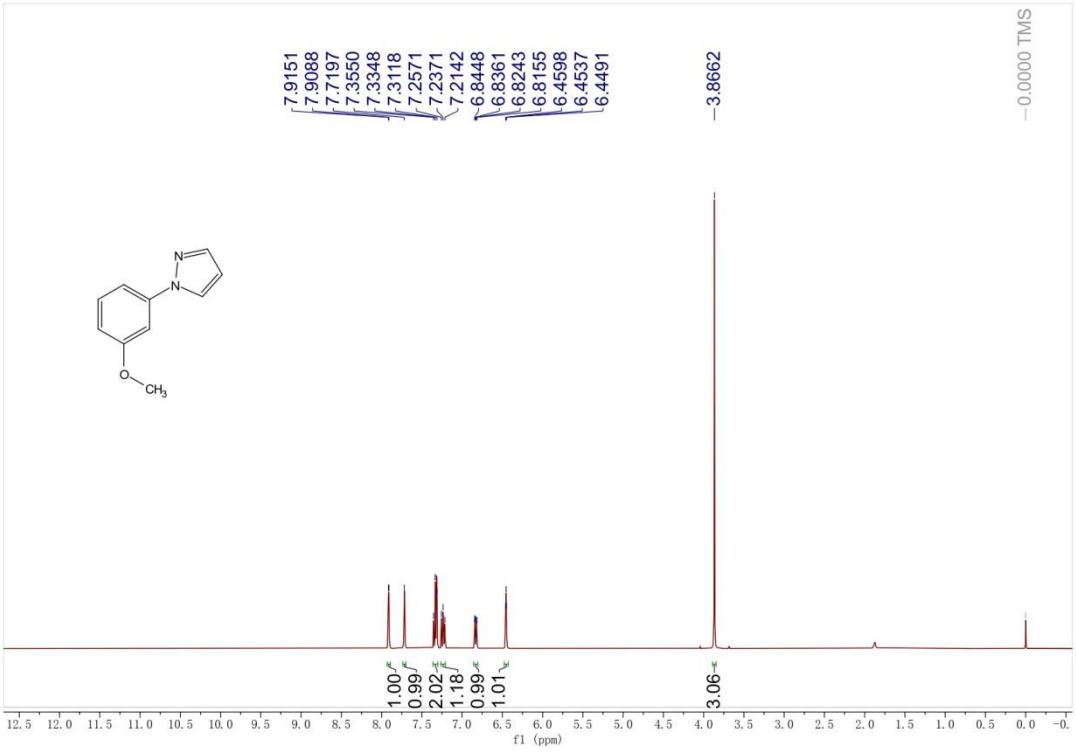
The MS spectrum of **3f**



^{1}H NMR spectrum of **3f** (400 MHz, $CDCl_3$)



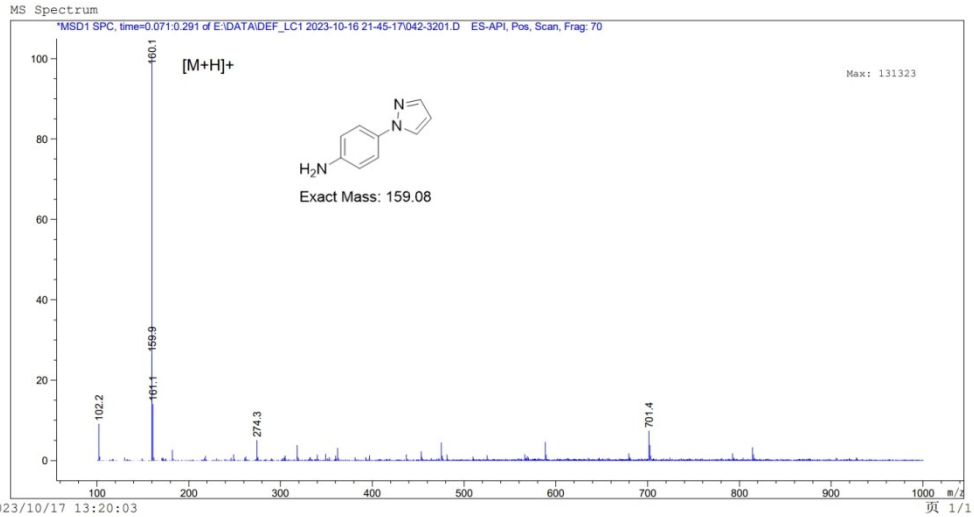
The MS spectrum of **3g**



^{1}H NMR spectrum of **3g** (400 MHz, $CDCl_3$)

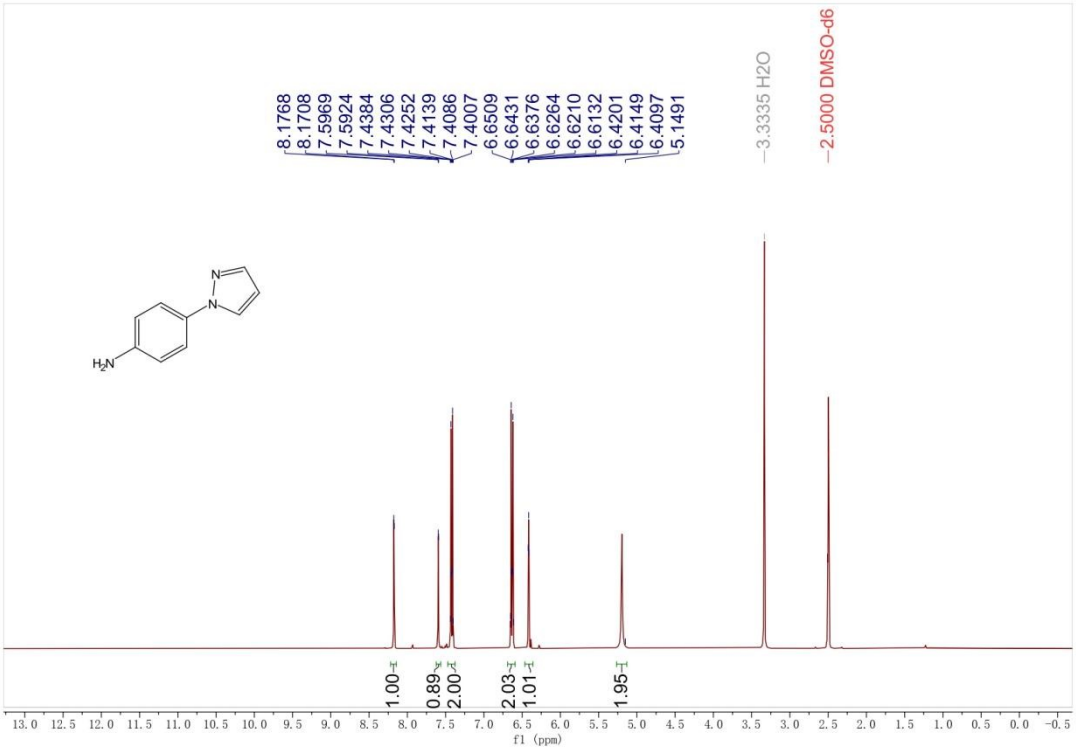
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\042-3201.D
样品名称 : XB-8

操作者 : 序列行 : 32
仪器 : 仪器 1 位置 : 样品瓶 42
进样日期 : 2023/10/17 1:15:07 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F

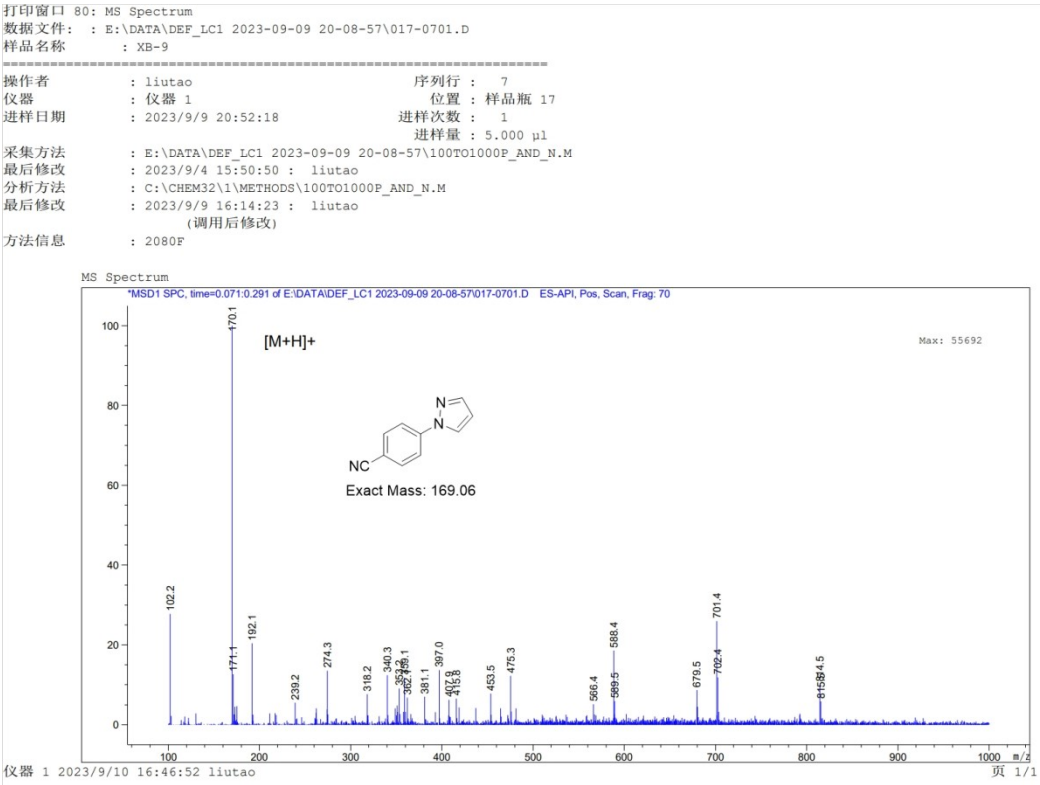


仪器 1 2023/10/17 13:20:03 页 1/1

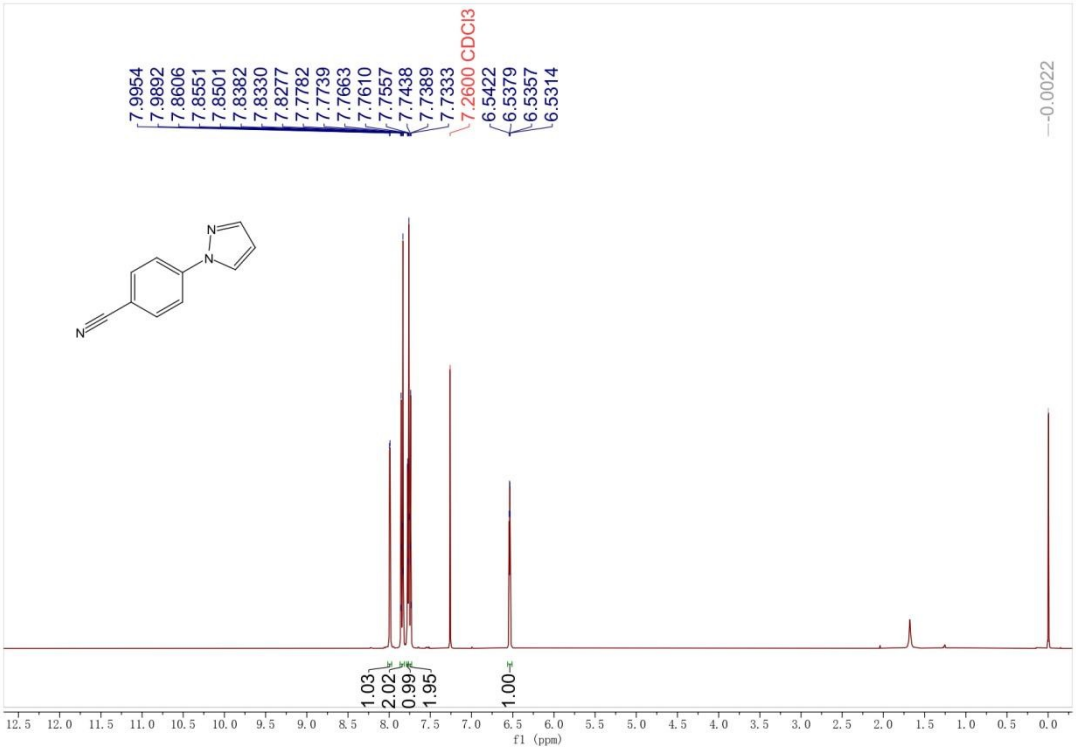
The MS spectrum of **3h**



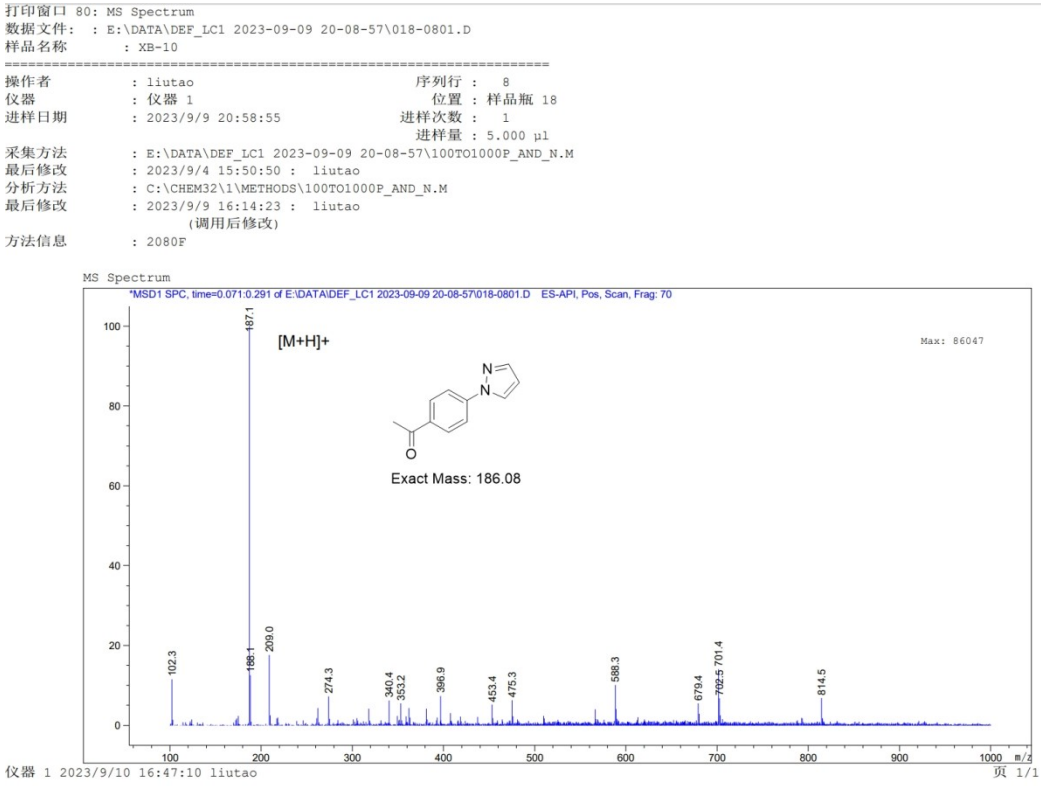
^{1}H NMR spectrum of **3h** (400 MHz, DMSO-*d6*)



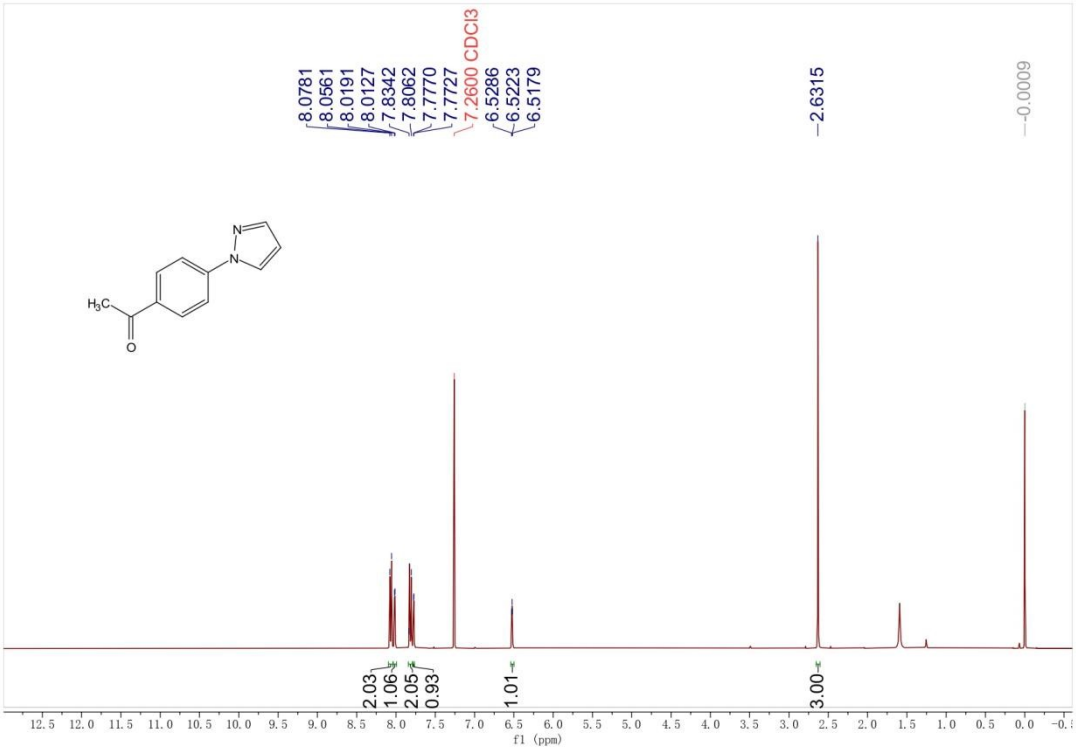
The MS spectrum of **3i**



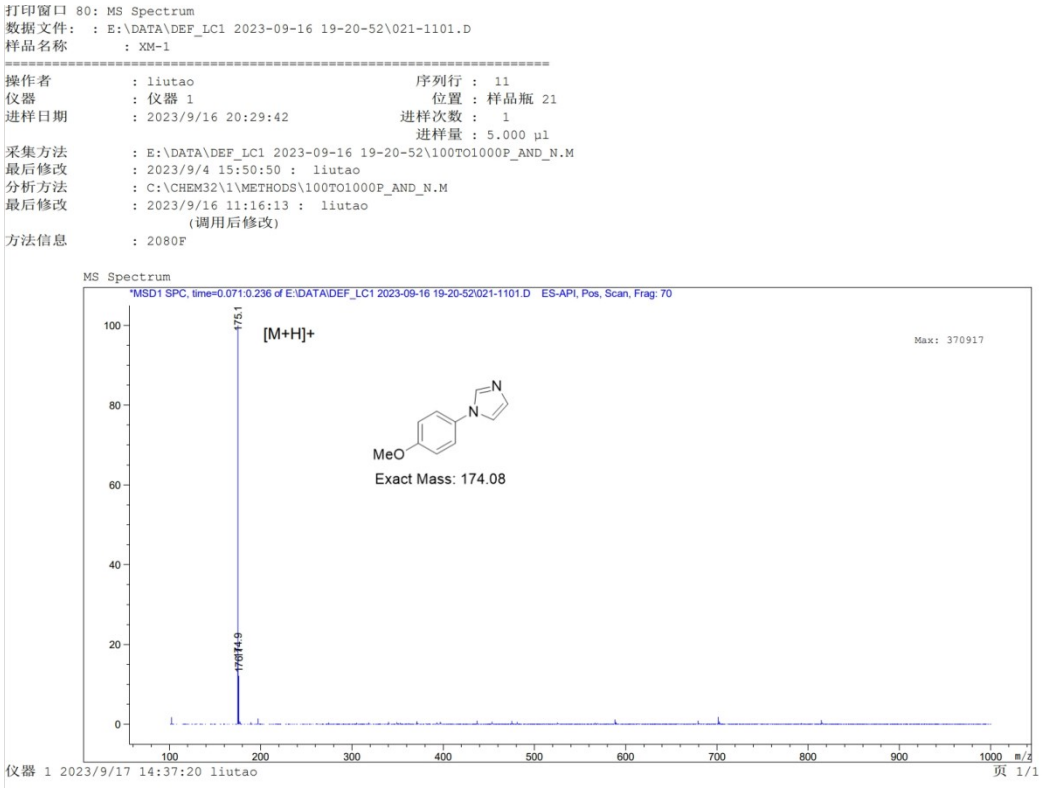
^{1}H NMR spectrum of **3i** (400 MHz, $CDCl_3$)



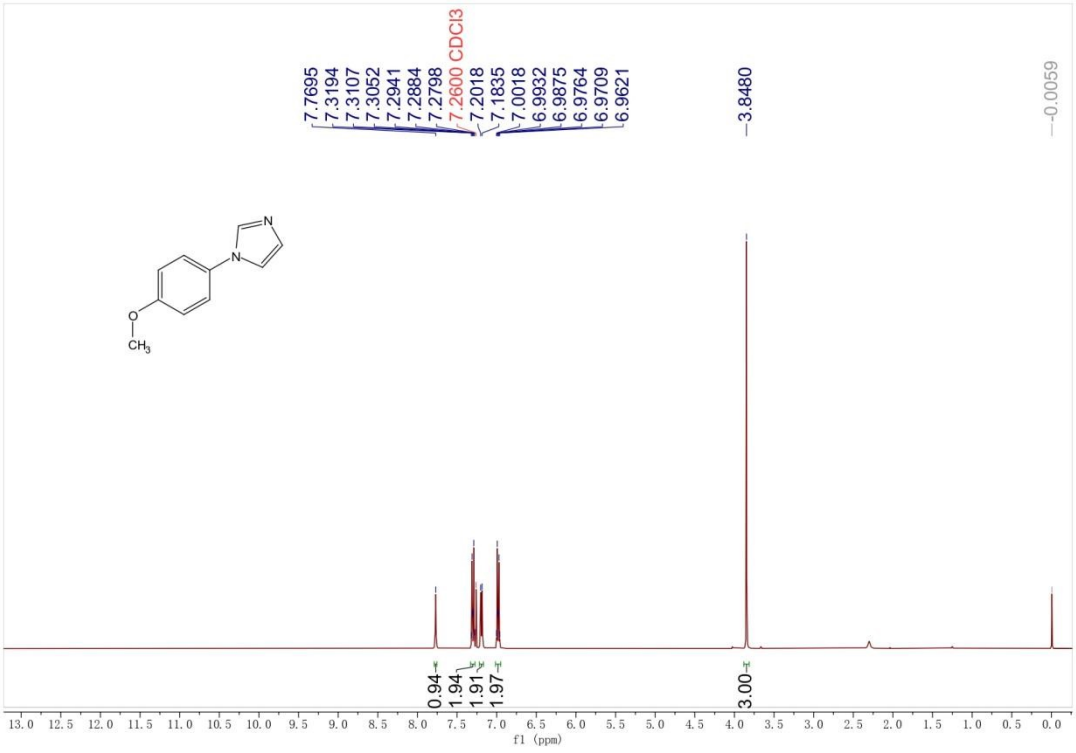
The MS spectrum of **3j**



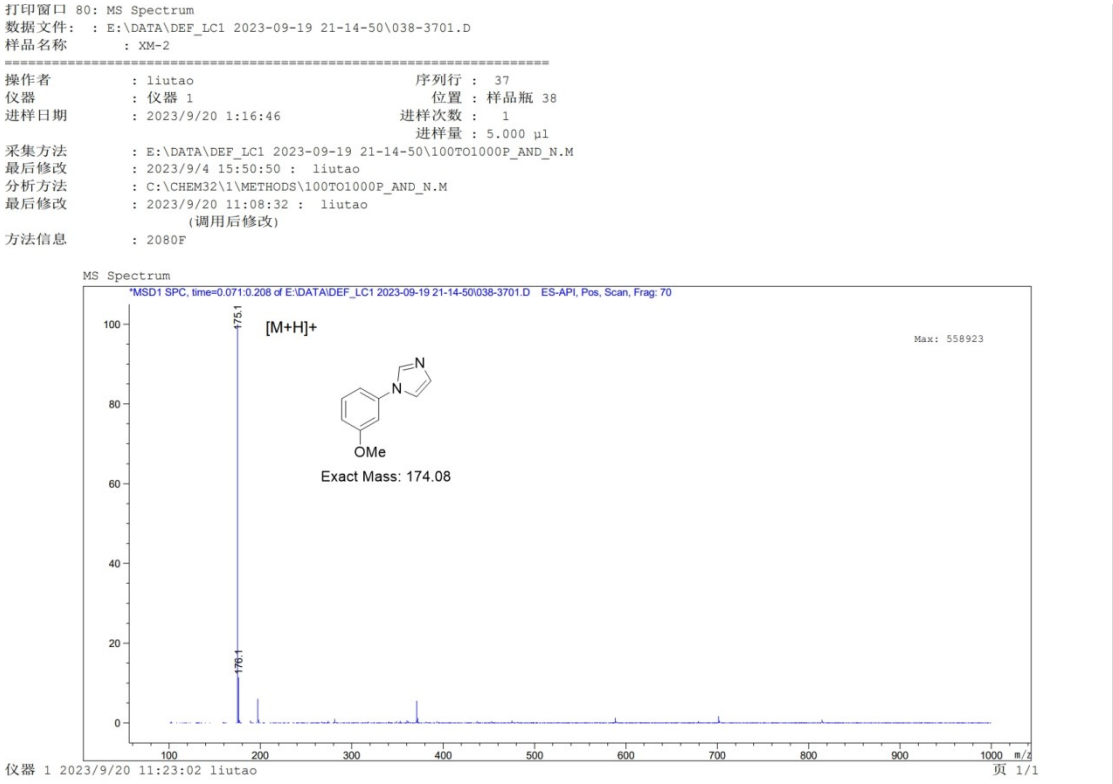
^{1}H NMR spectrum of **3j** (400 MHz, $CDCl_3$)



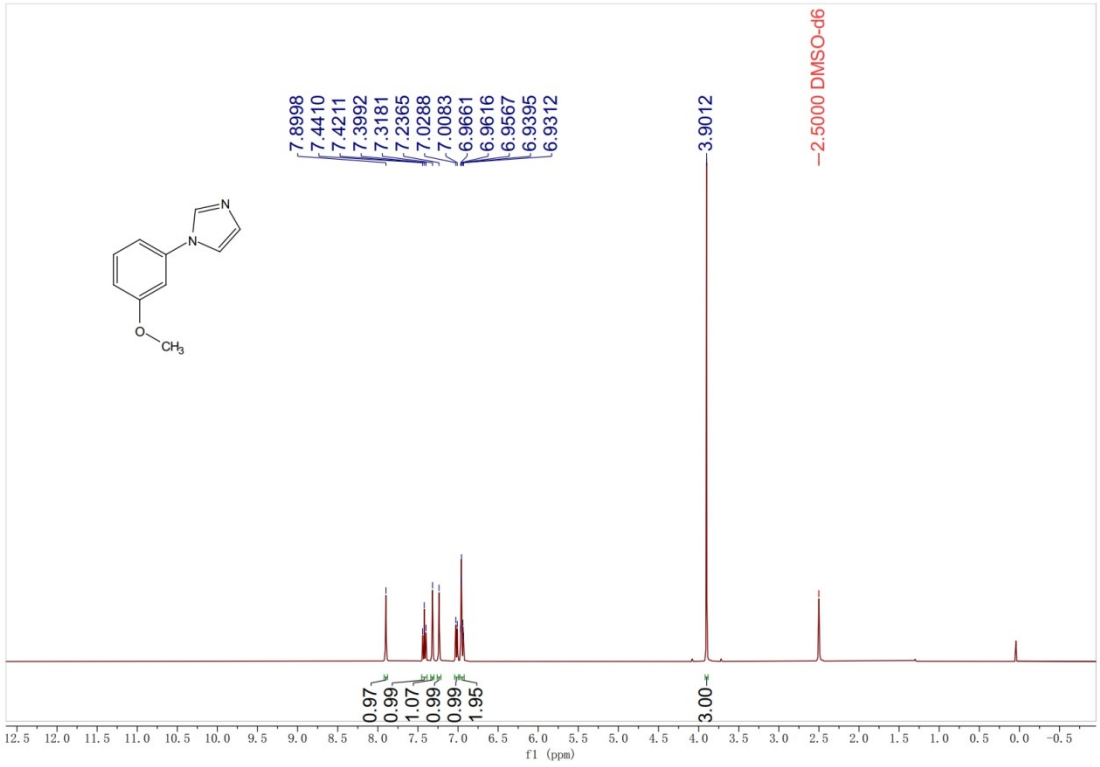
The MS spectrum of **3k**



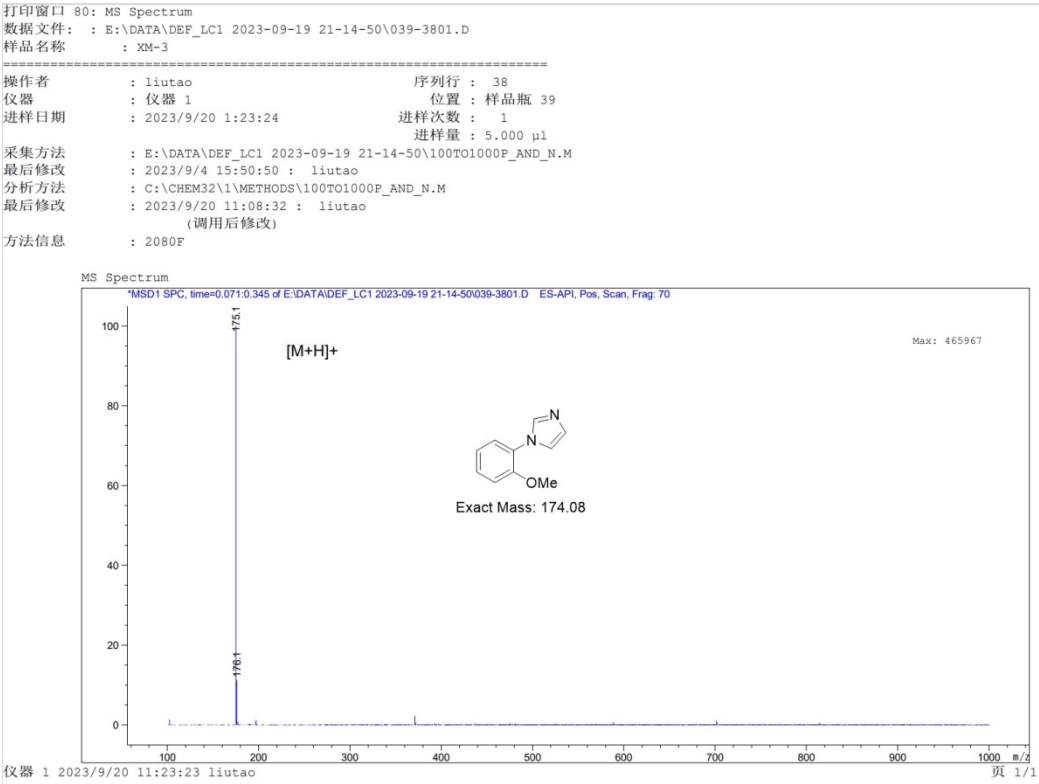
^{1}H NMR spectrum of **3k** (400 MHz, $CDCl_3$)



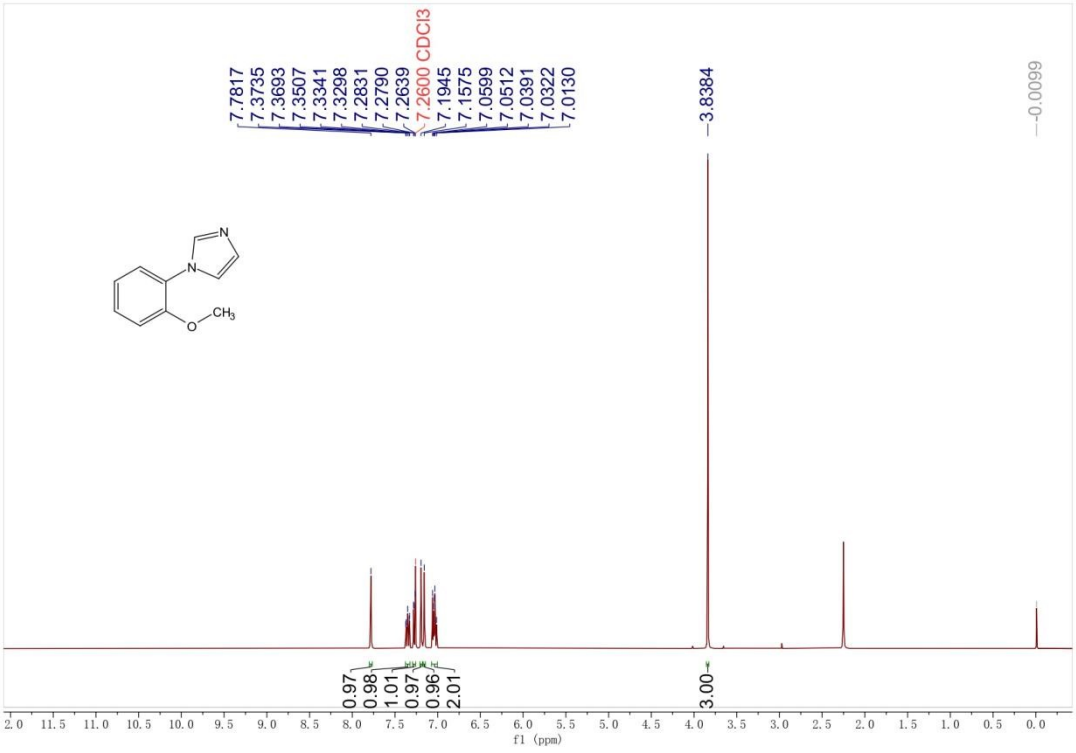
The MS spectrum of **3l**



^{1}H NMR spectrum of **3m** (400 MHz, DMSO-d6)



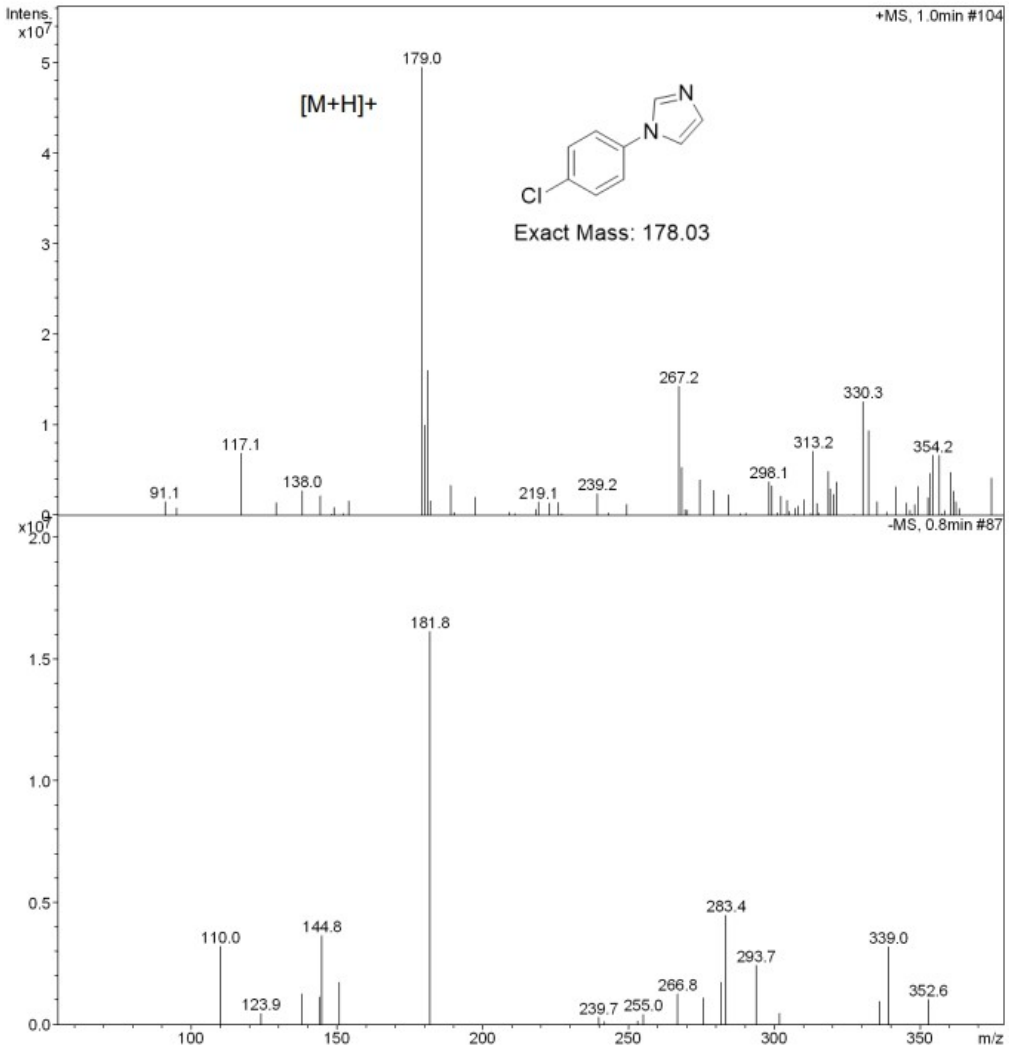
The MS spectrum of **3m**



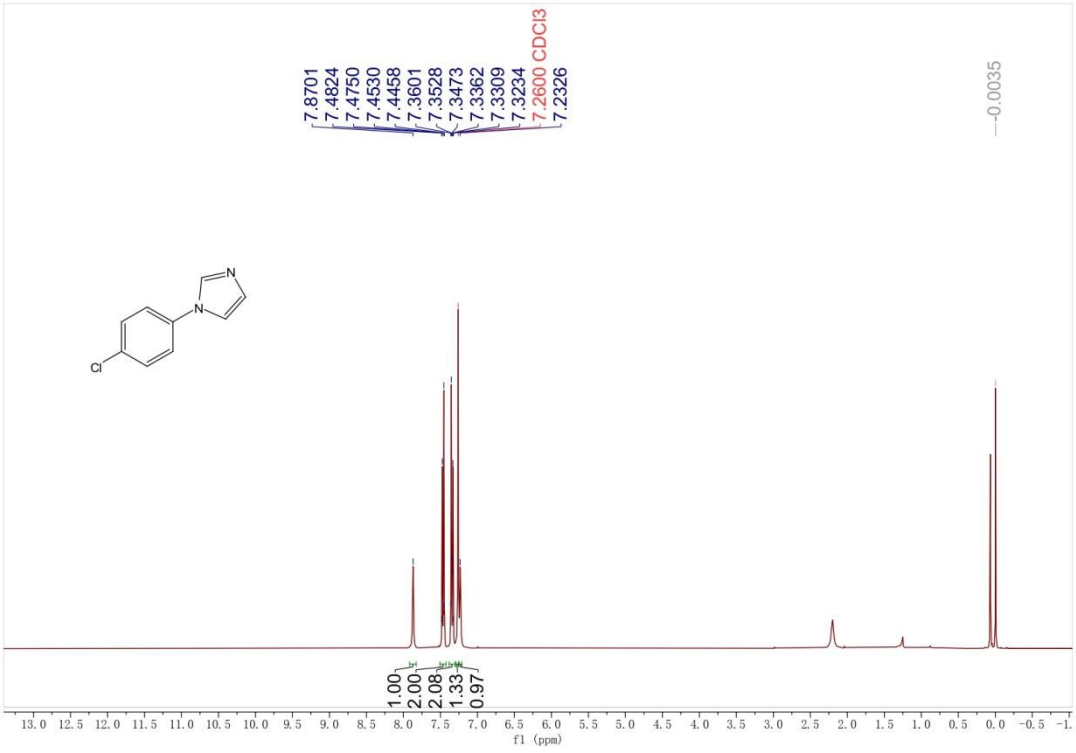
^{1}H NMR spectrum of **3m** (400 MHz, $CDCl_3$)

Direct Mass Spectrometry Analysis

Analysis Name: 23072915.d
Sample Name: XM-4
Instrument: LC-MSD-Trap-SL
Operator: 21953830E000H12U
Print Date: 7/29/2023 10:07:29 AM
Acq. Date: 7/29/2023 10:05:26 AM



The MS spectrum of **3n**



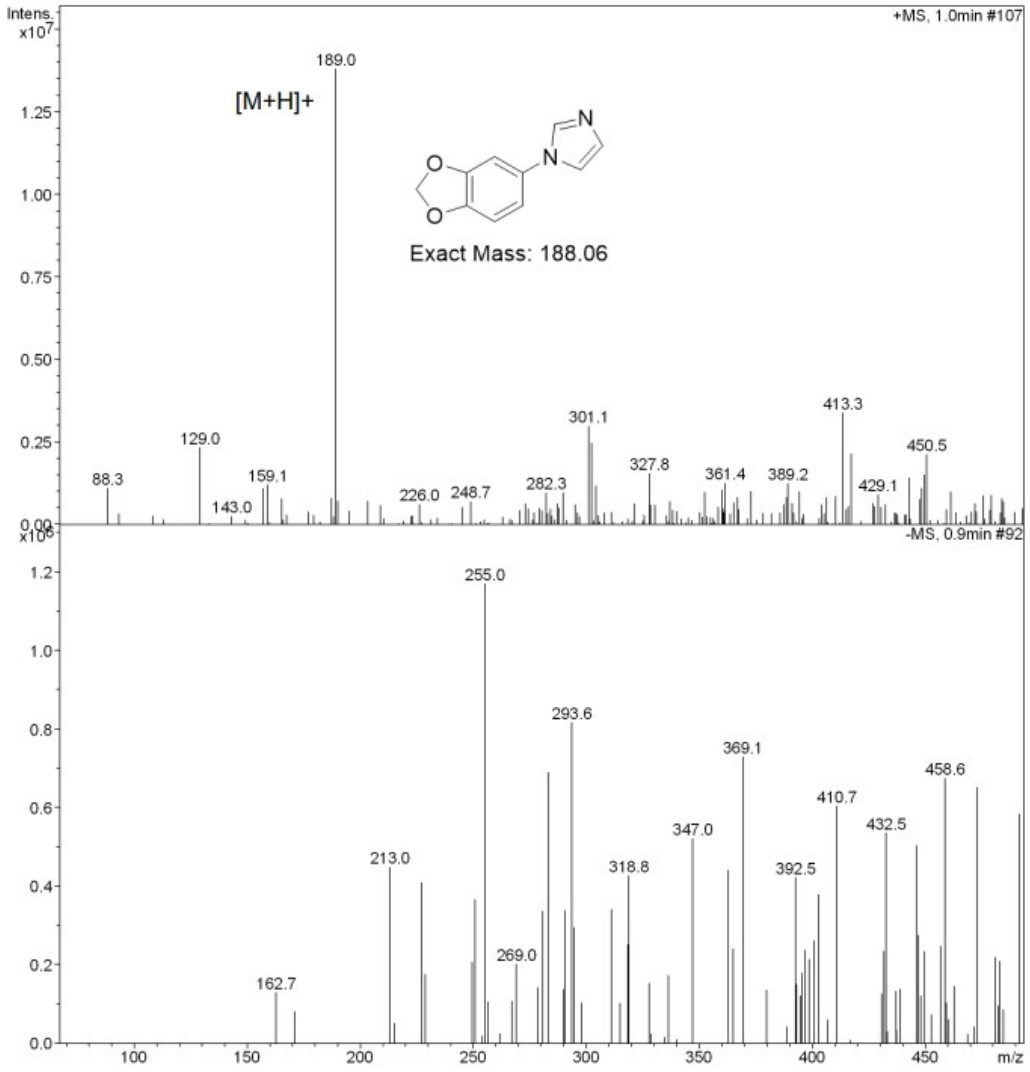
^{1}H NMR spectrum of **3n** (400 MHz, $CDCl_3$)

Direct Mass Spectrometry Analysis

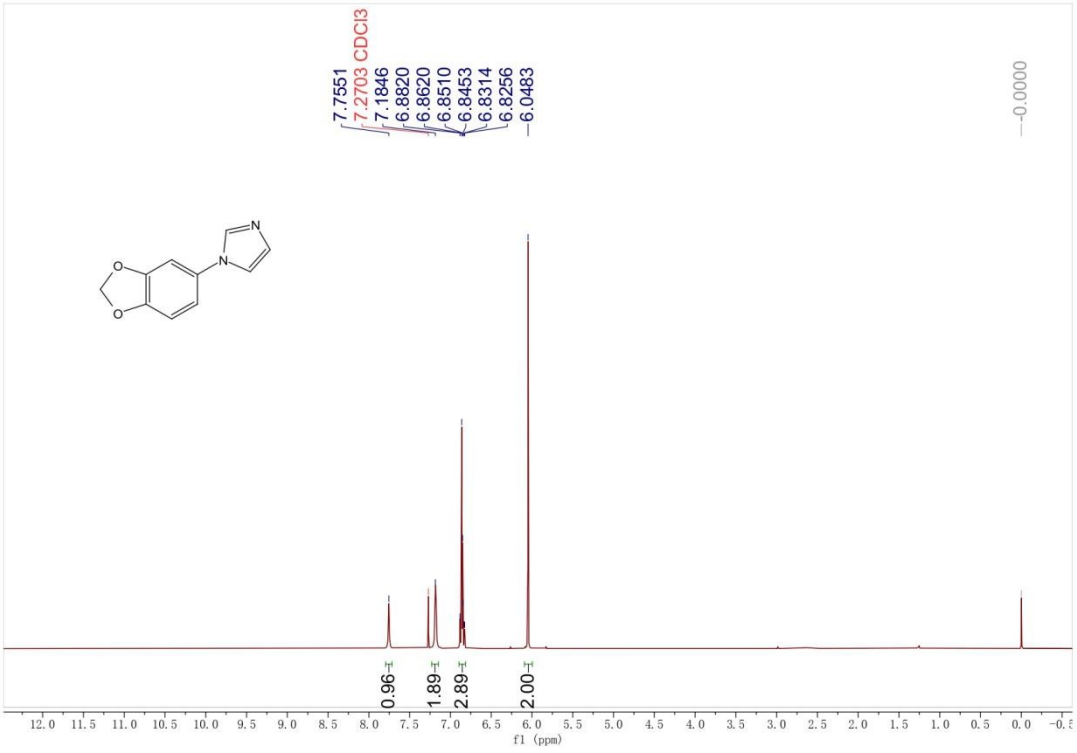
Analysis Name: 23072902.d
Sample Name: XM-5

Instrument: LC-MSD-Trap-SL
Operator: 21953830E000H12U

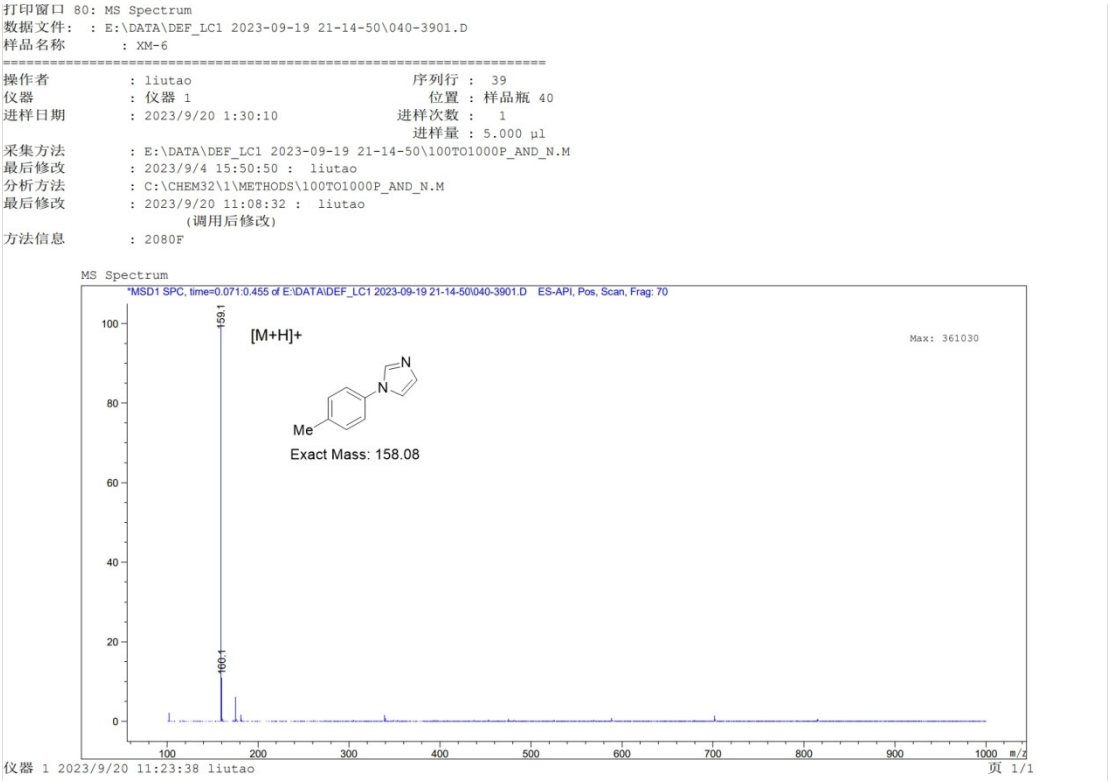
Print Date: 7/29/2023 9:11:43 AM
Acq. Date: 7/29/2023 9:09:33 AM



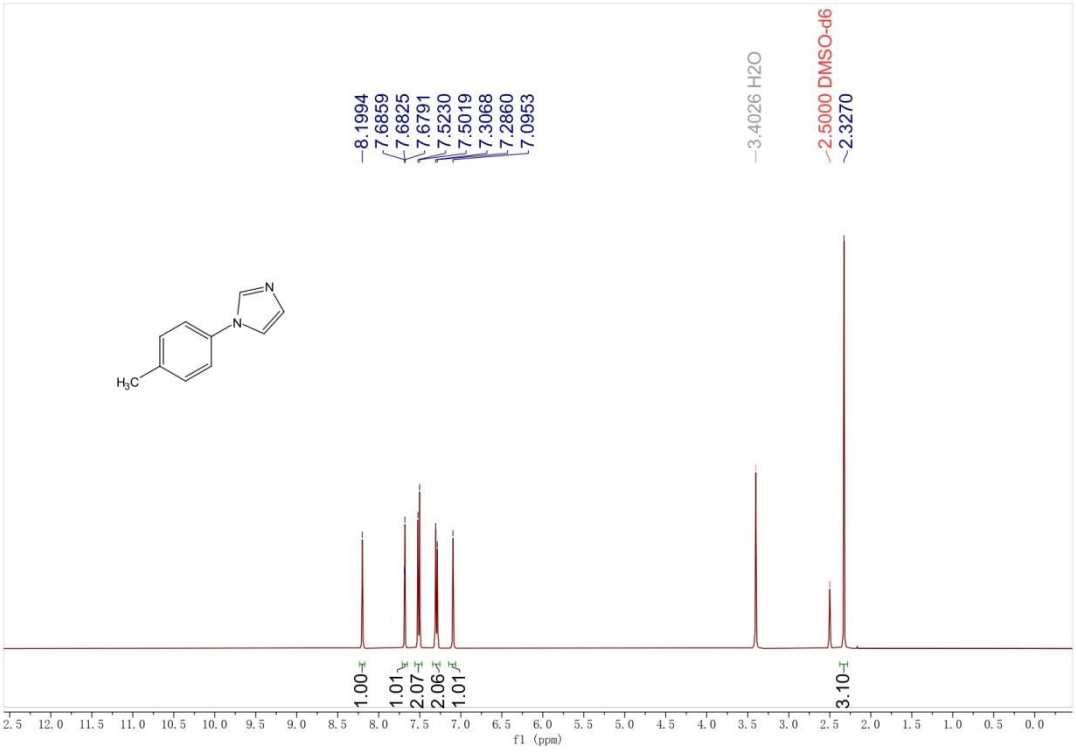
The MS spectrum of **3o**



^{1}H NMR spectrum of **3o** (400 MHz, $CDCl_3$)



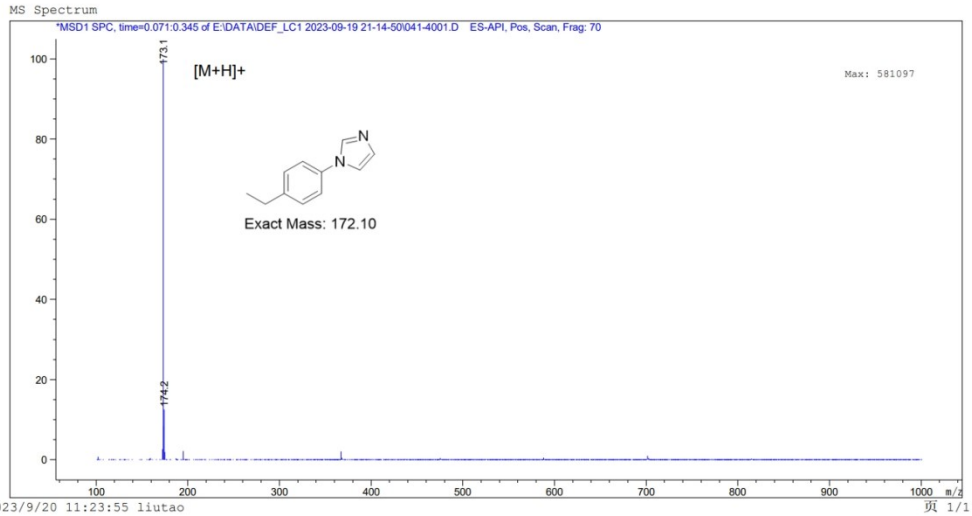
The MS spectrum of **3p**



^{1}H NMR spectrum of **3p** (400 MHz, DMSO-d6)

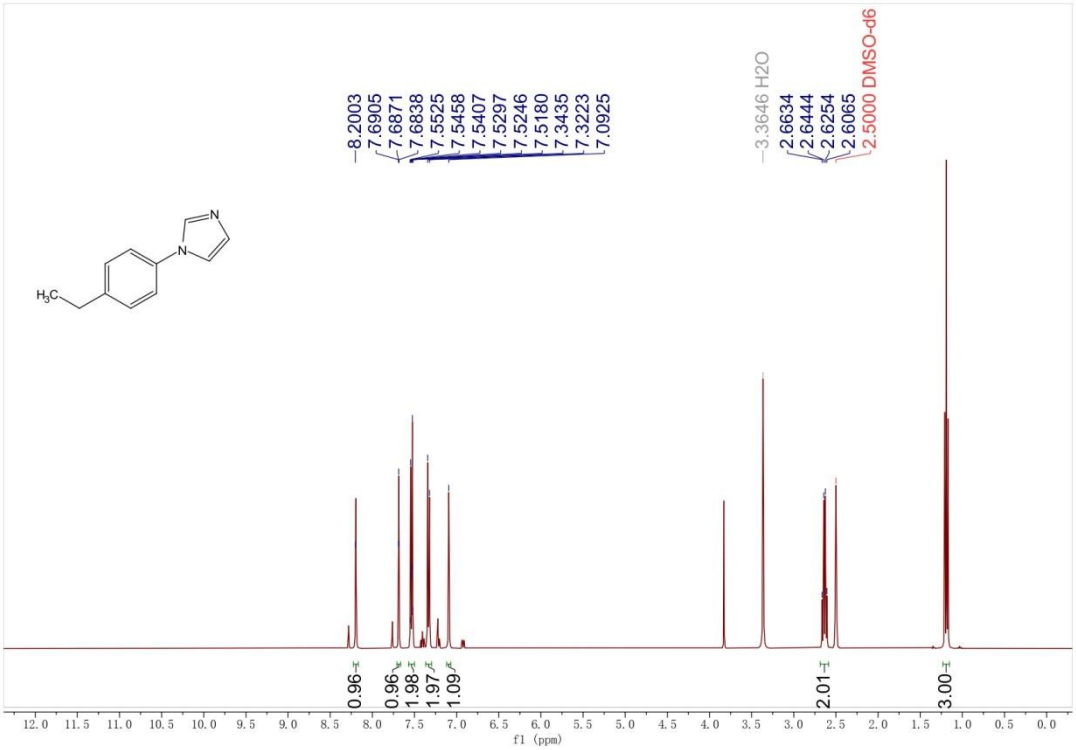
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-09-19 21-14-50\041-4001.D
样品名称 : XM-7

操作者 : liutao 序列行 : 40
仪器 : 仪器 1 位置 : 样品瓶 41
进样日期 : 2023/9/20 1:36:46 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-09-19 21-14-50\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/9/20 11:08:32 : liutao
(调用后修改)
方法信息 : 2080F

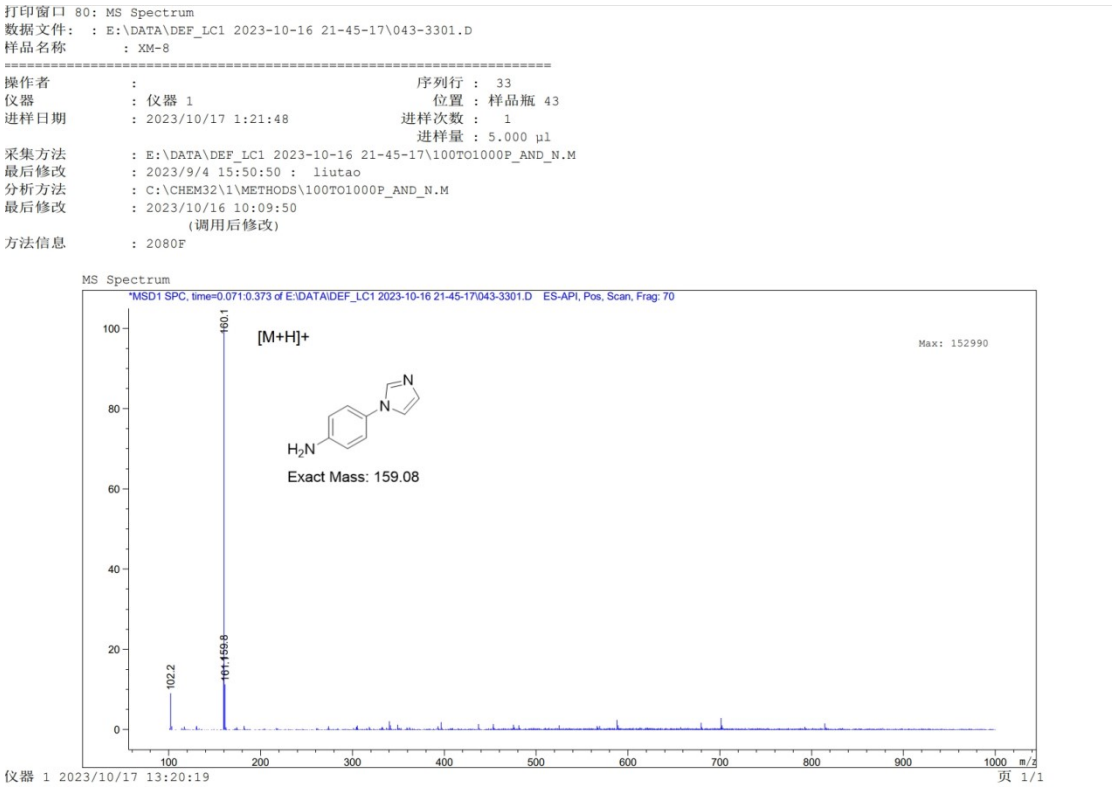


仪器 1 2023/9/20 11:23:55 liutao 页 1/1

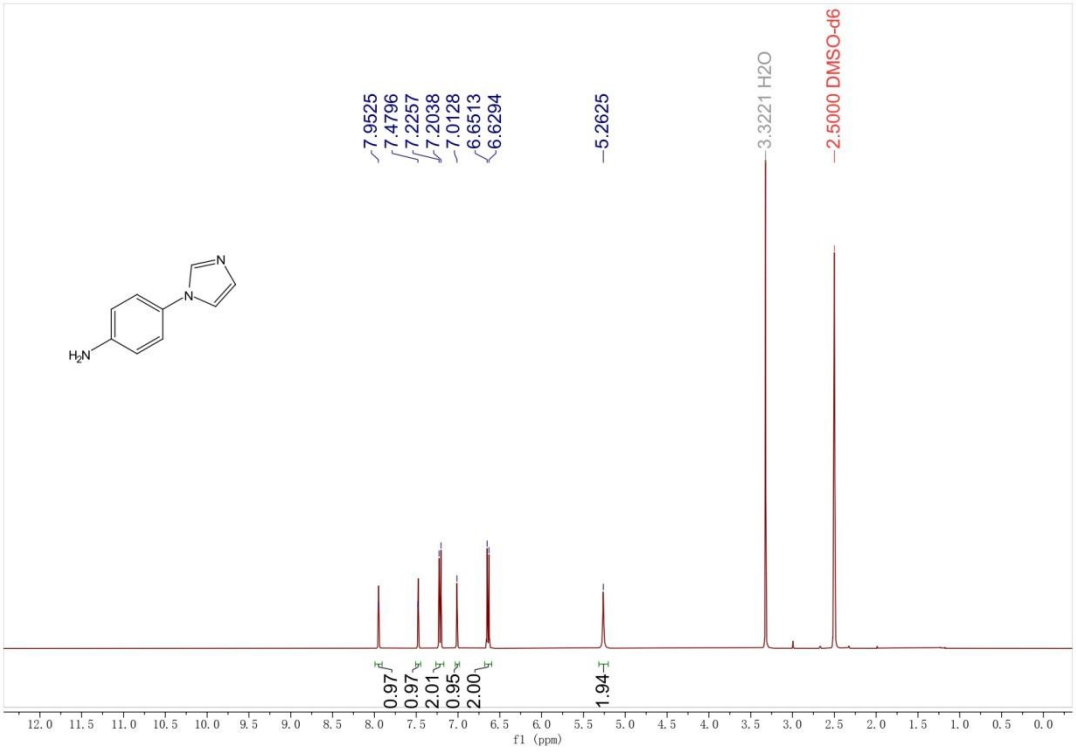
The MS spectrum of **3q**



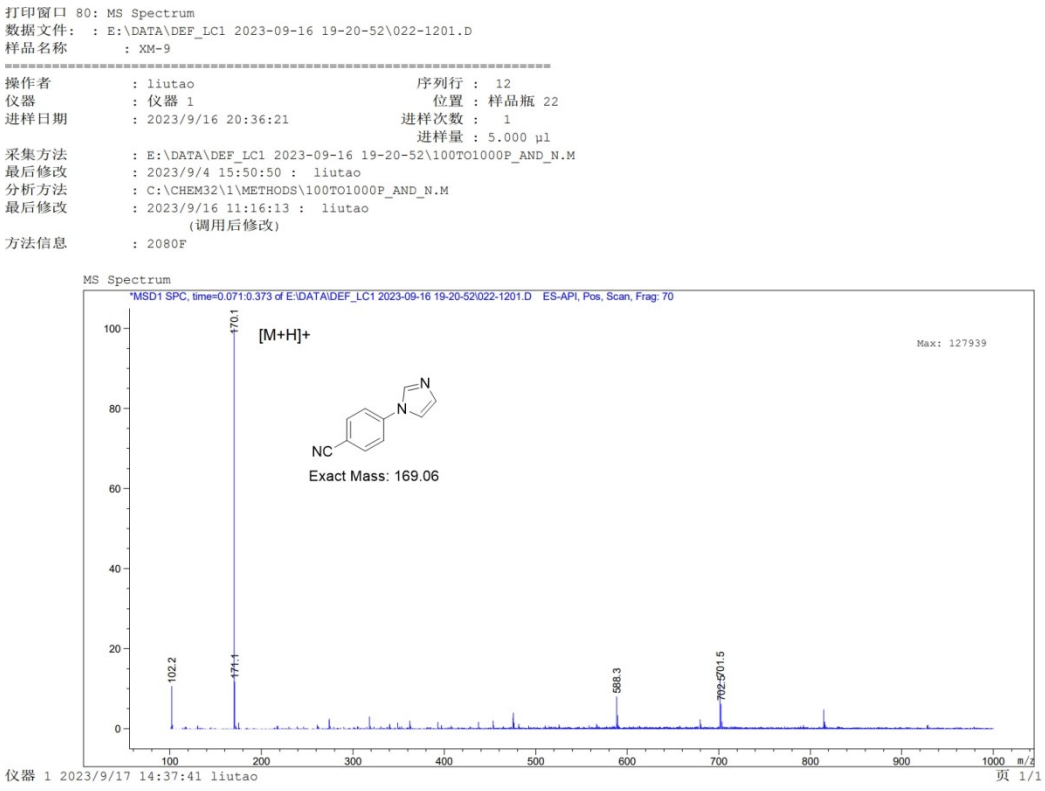
^{1}H NMR spectrum of **3q** (400 MHz, DMSO-d6)



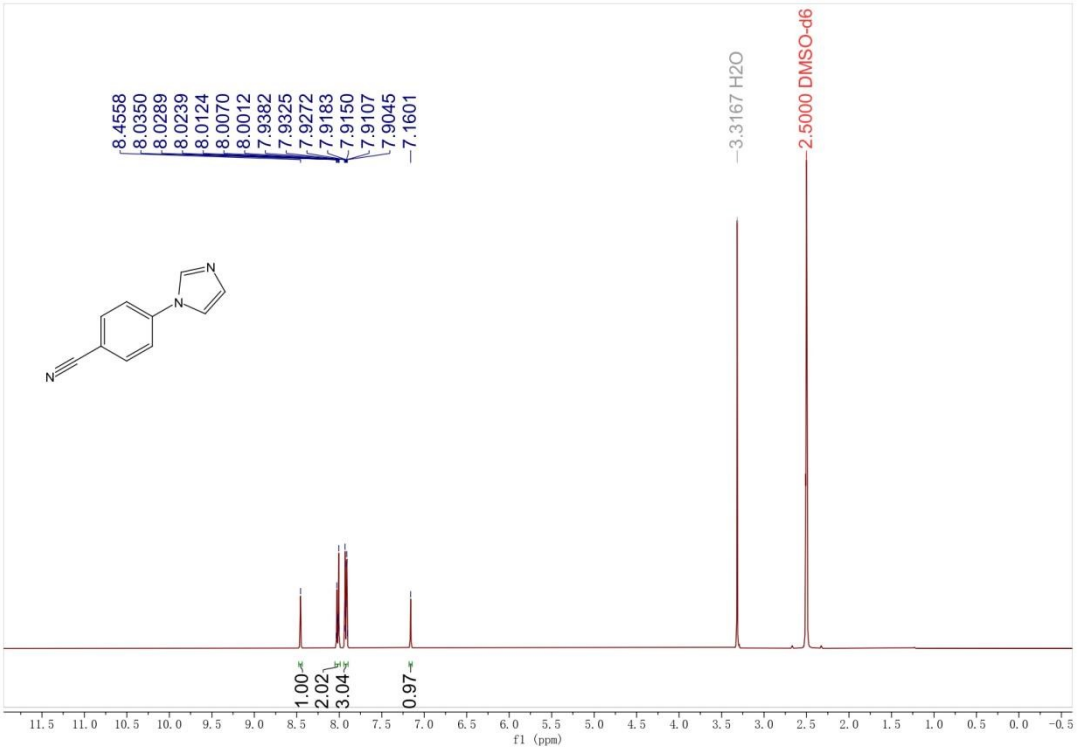
The MS spectrum of **3r**



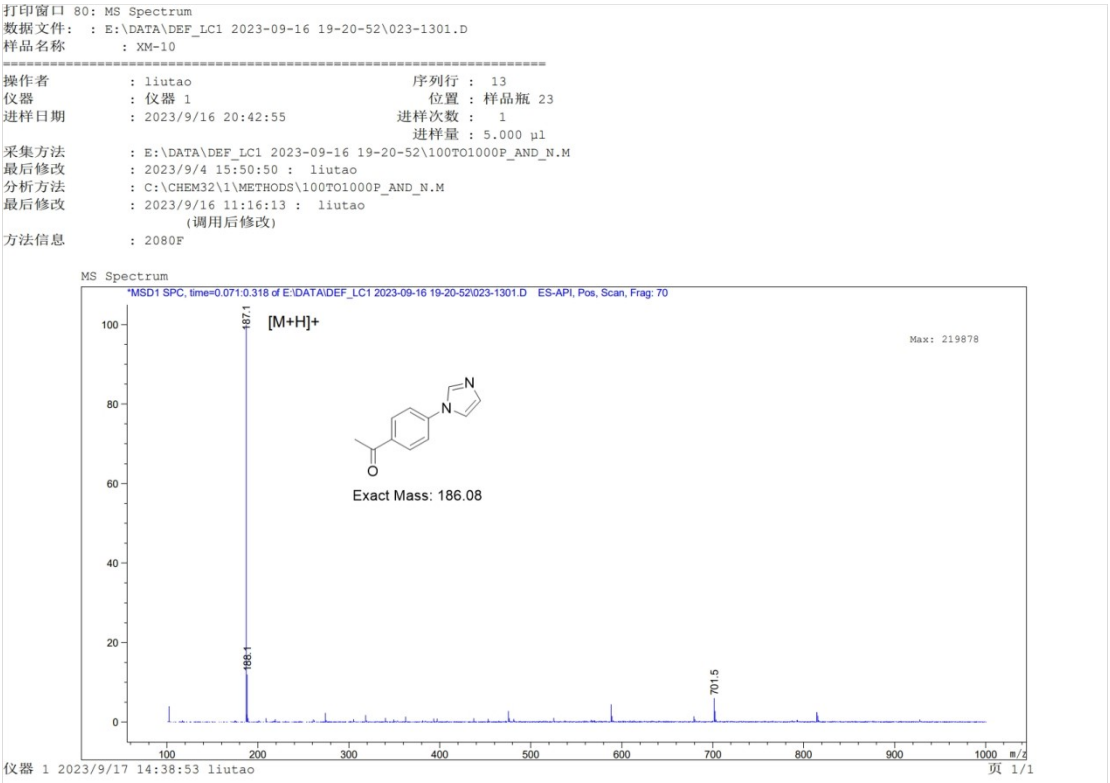
^{1}H NMR spectrum of **3r** (400 MHz, DMSO-d6)



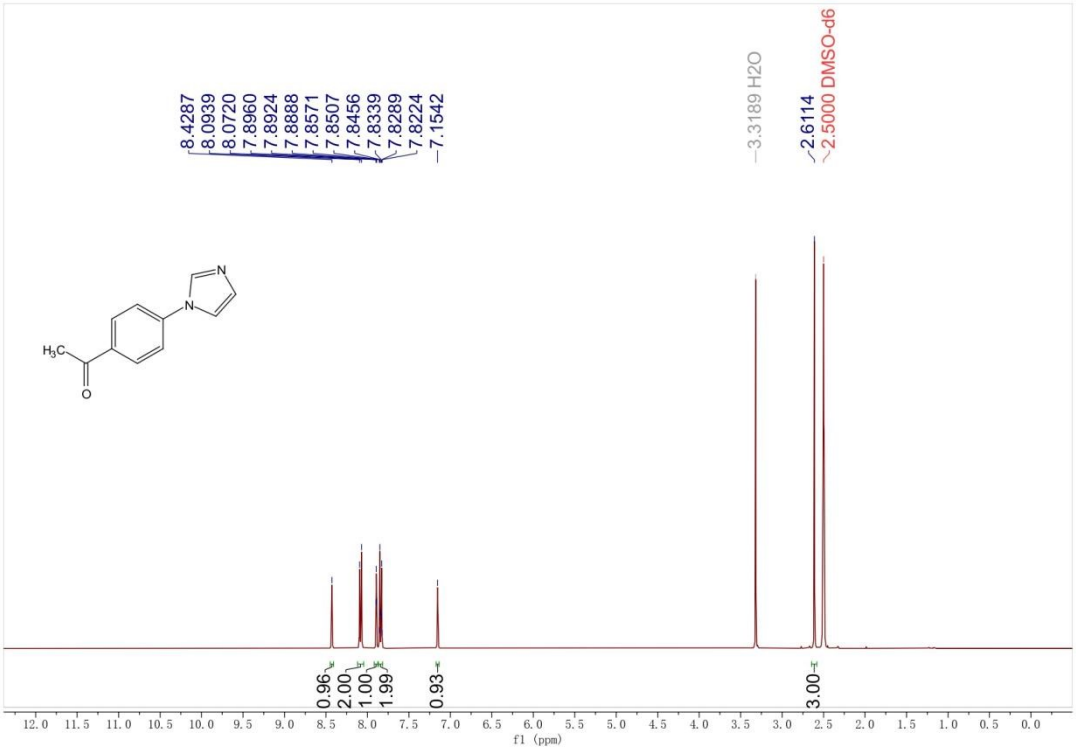
The MS spectrum of **3S**



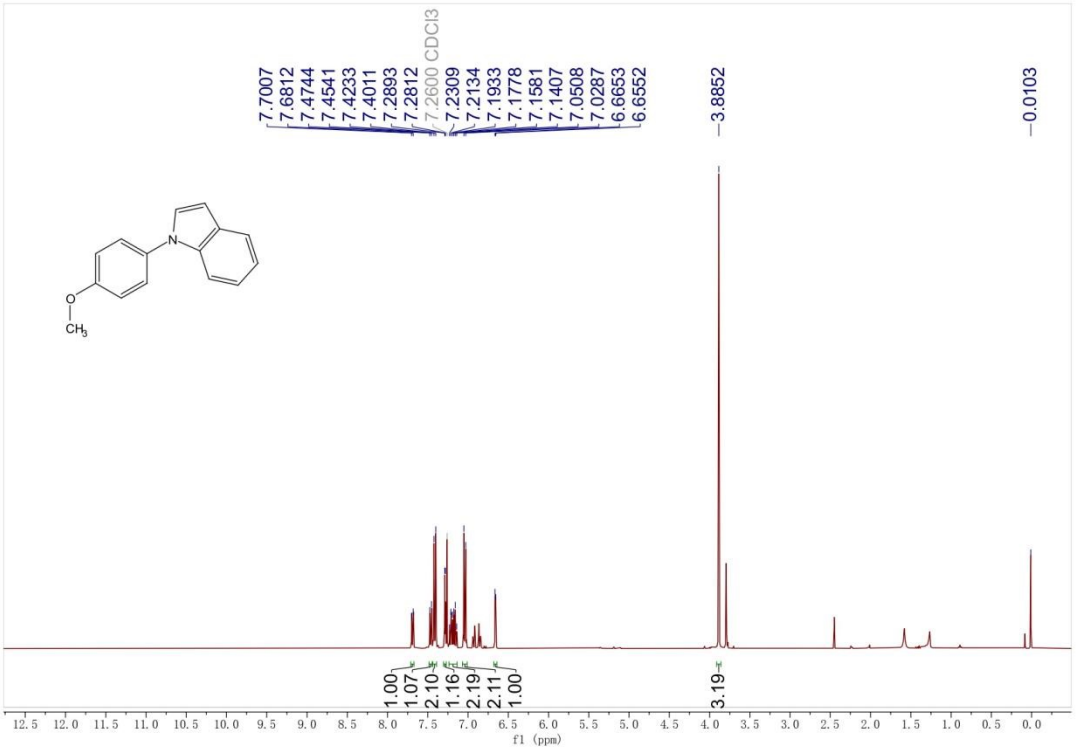
^{1}H NMR spectrum of **3S** (400 MHz, DMSO-d6)



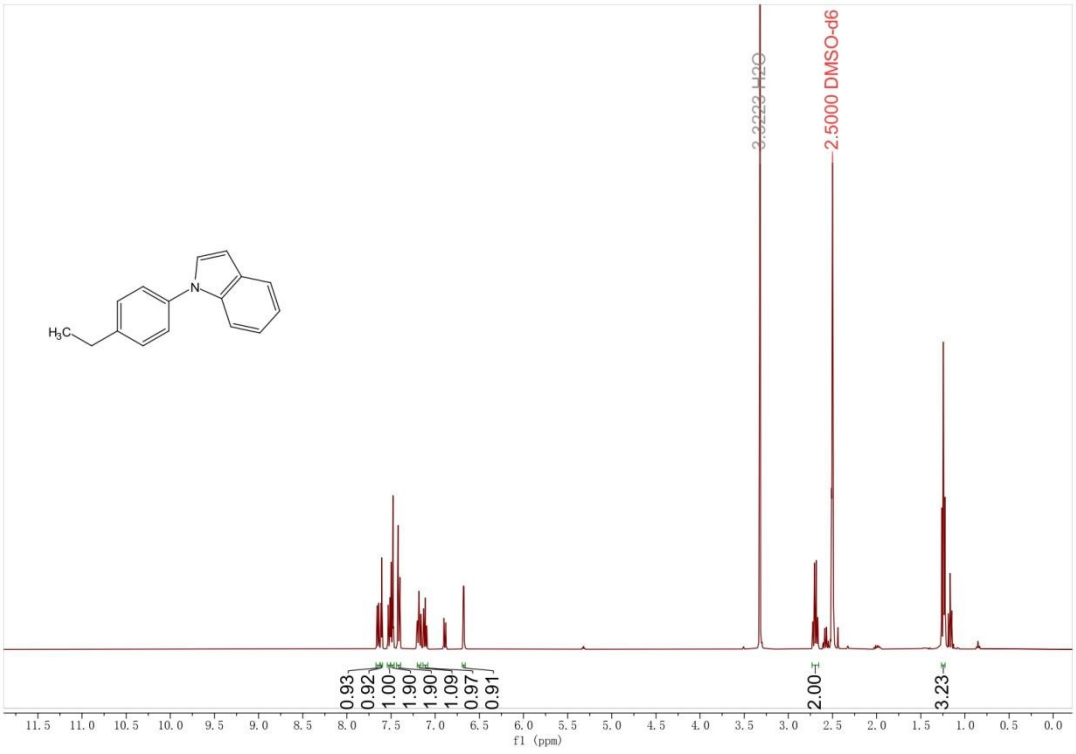
The MS spectrum of **3t**



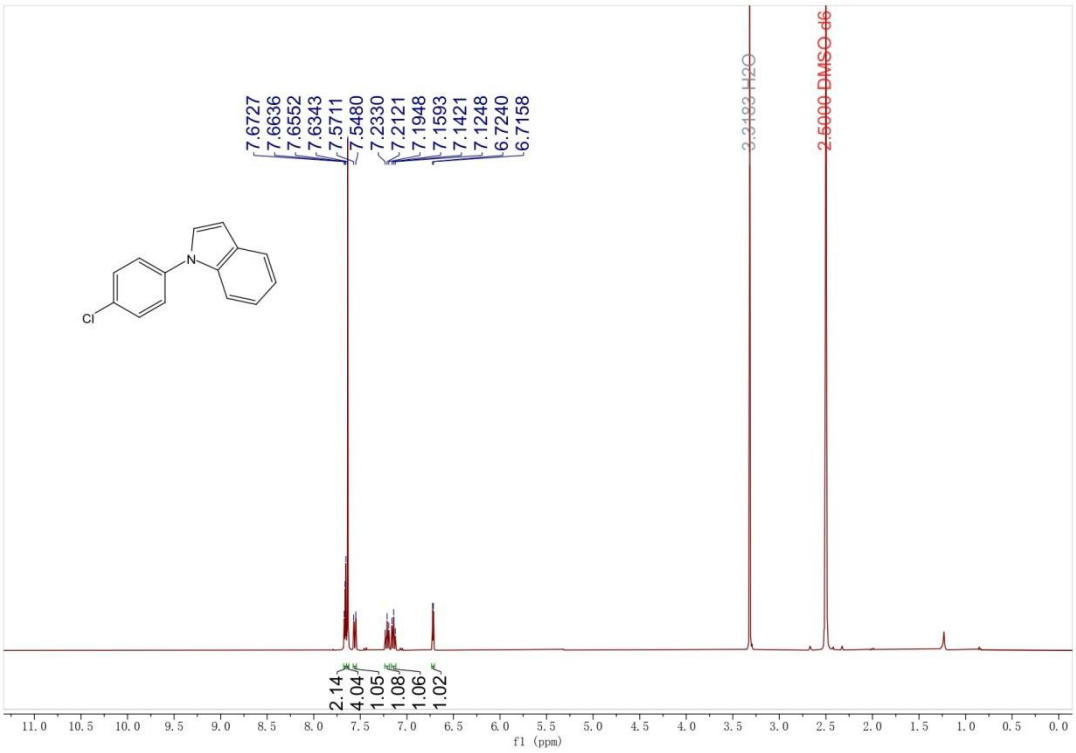
^{1}H NMR spectrum of **3t** (400 MHz, DMSO-d6)



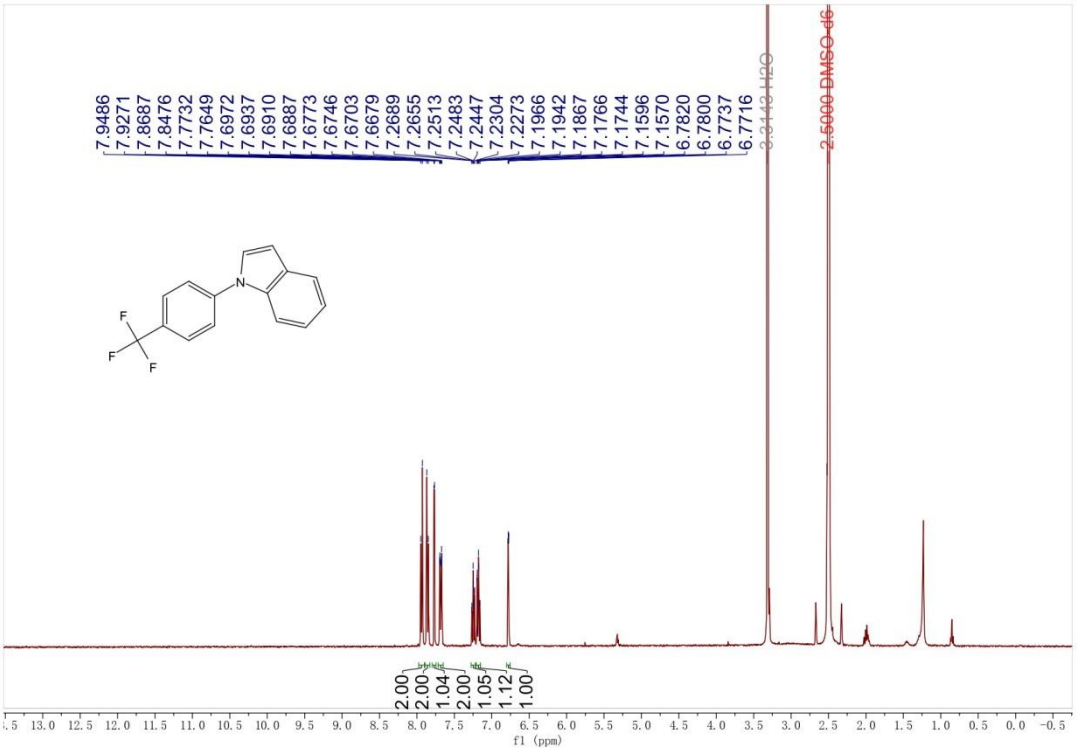
^{1}H NMR spectrum of **3u** (400 MHz, $CDCl_3$)



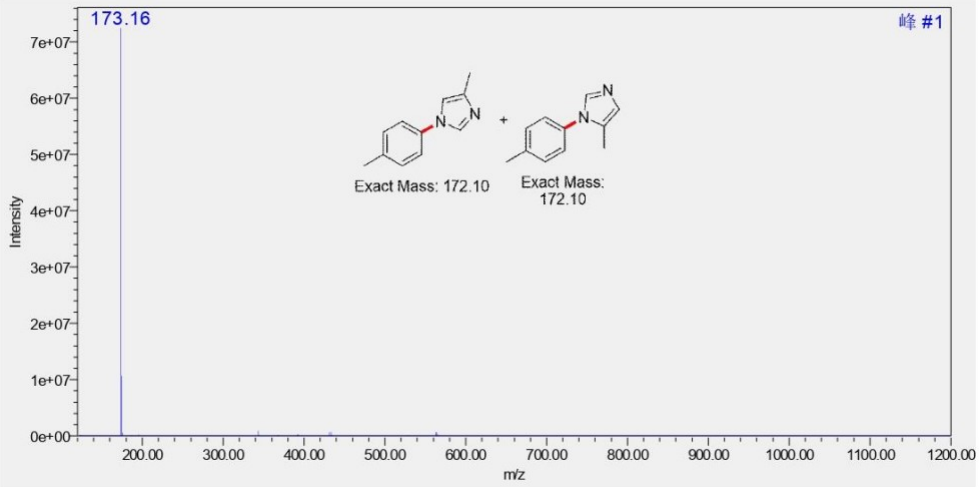
^{1}H NMR spectrum of **3v** (400 MHz, DMSO-d6)



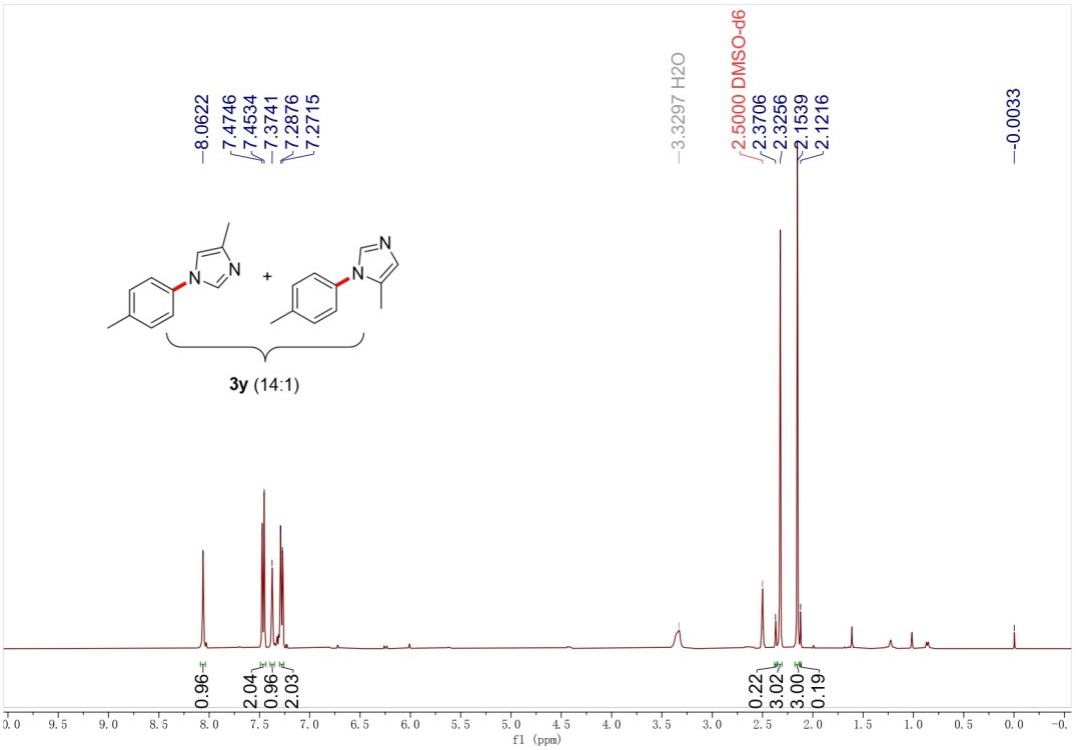
^{1}H NMR spectrum of **3v** (400 MHz, DMSO-d6)



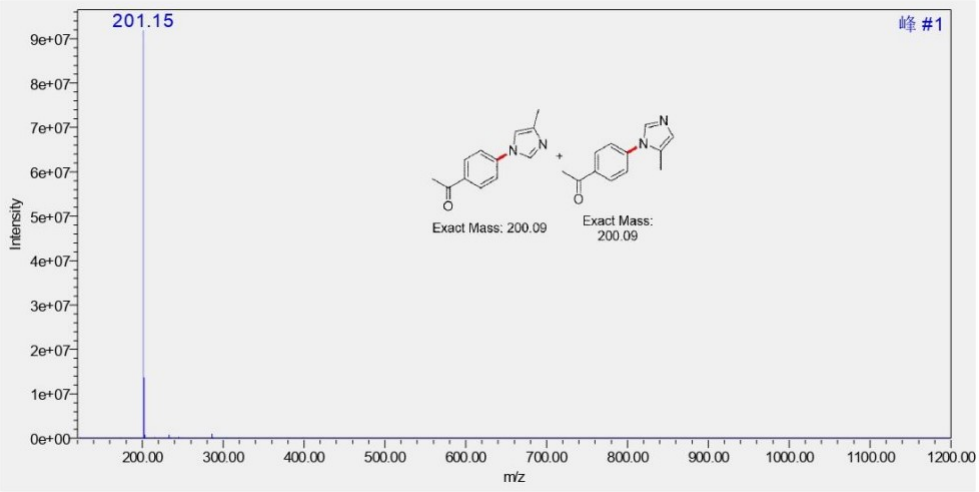
^{1}H NMR spectrum of **3v** (400 MHz, DMSO-d6)



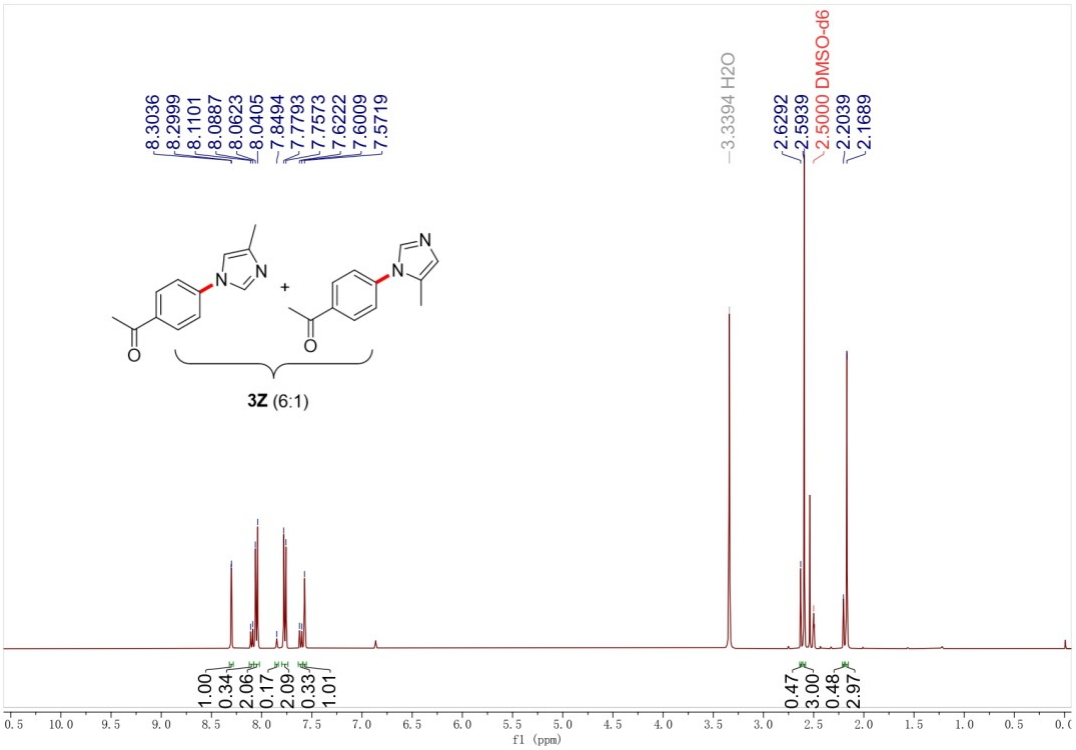
The MS spectrum of **3y**



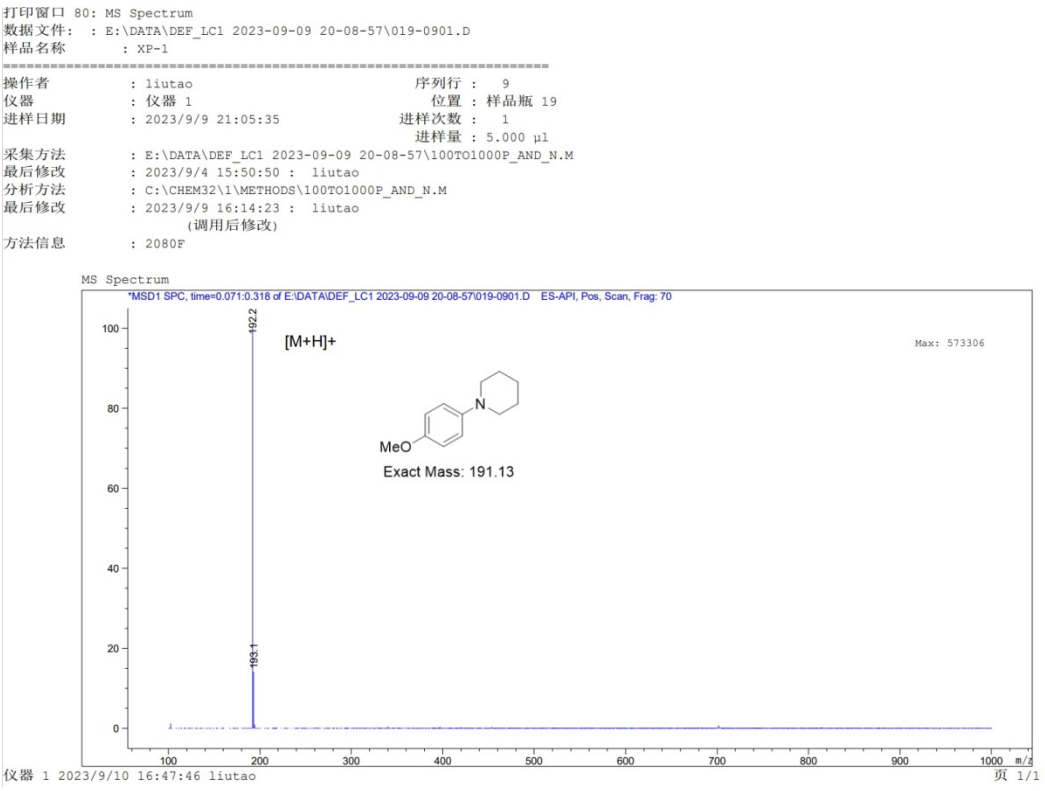
^{1}H NMR spectrum of **3y** (400 MHz, DMSO-*d6*)



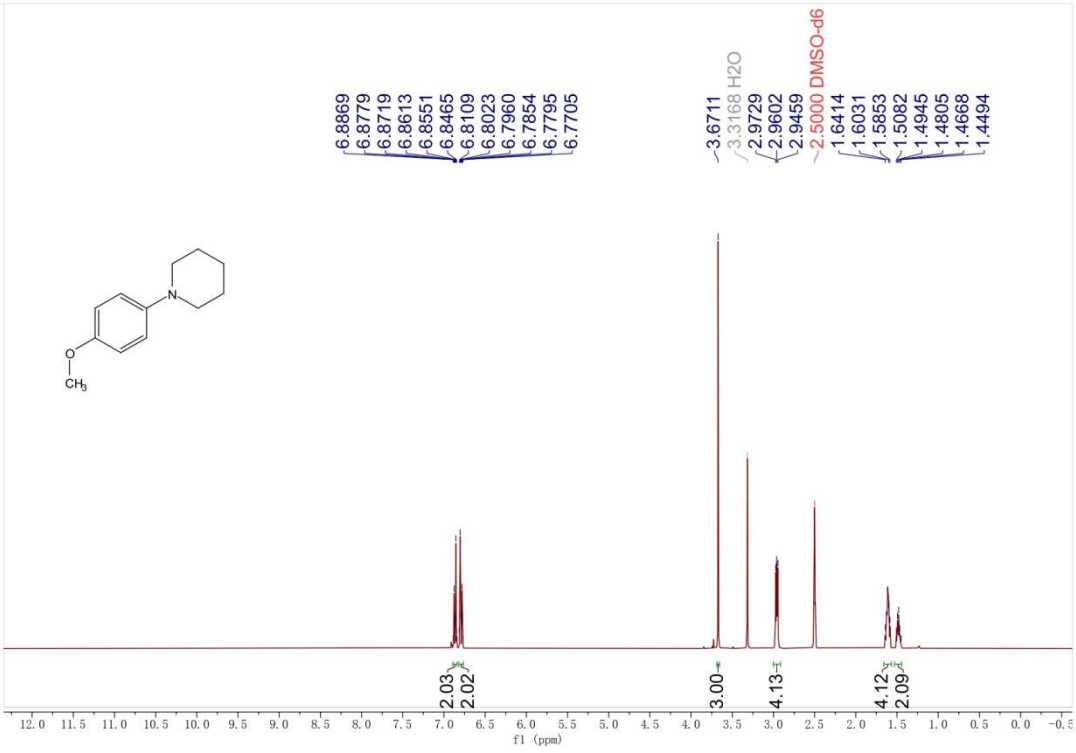
The MS spectrum of **3z**



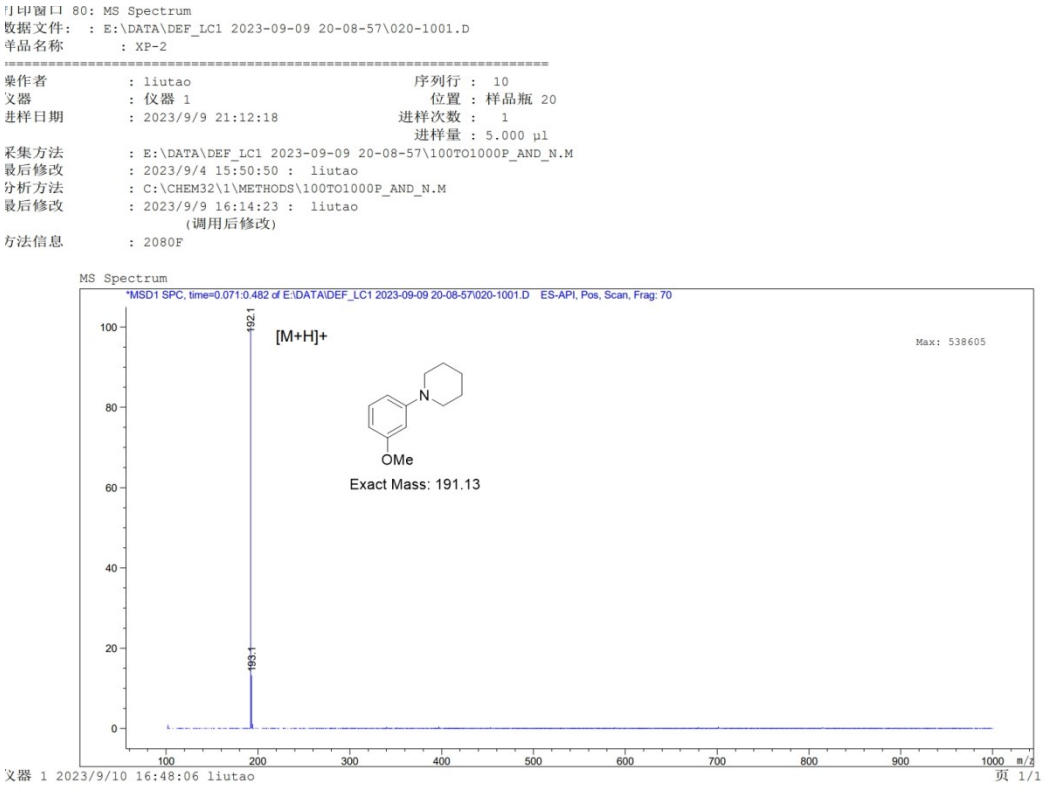
^{1}H NMR spectrum of **3z** (400 MHz, DMSO-*d6*)



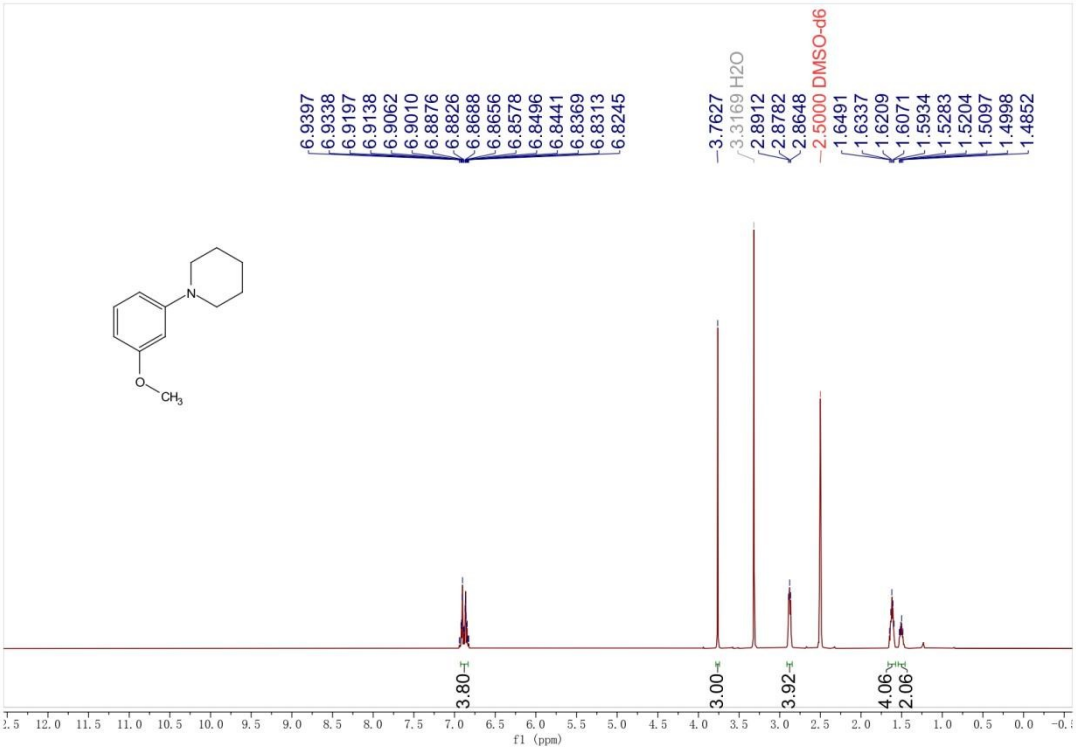
The MS spectrum of **5a**



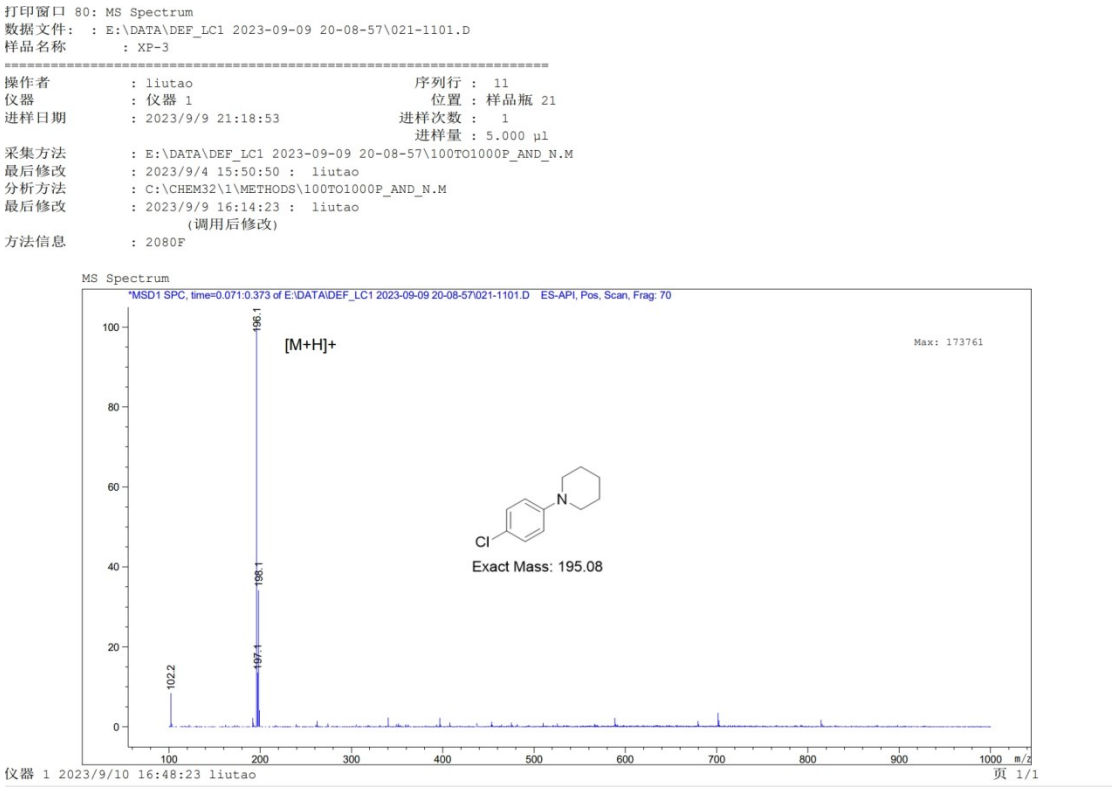
^{1}H NMR spectrum of **5a** (400 MHz, DMSO-d6)



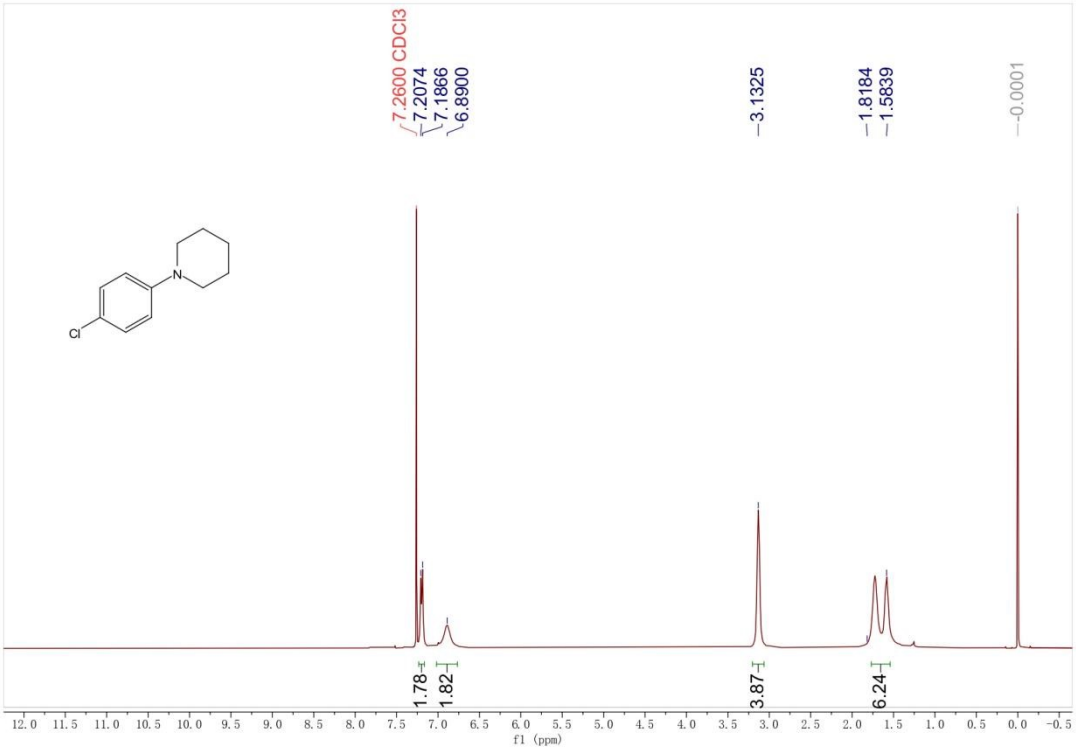
The MS spectrum of **5b**



^{1}H NMR spectrum of **5b** (400 MHz, DMSO-d6)



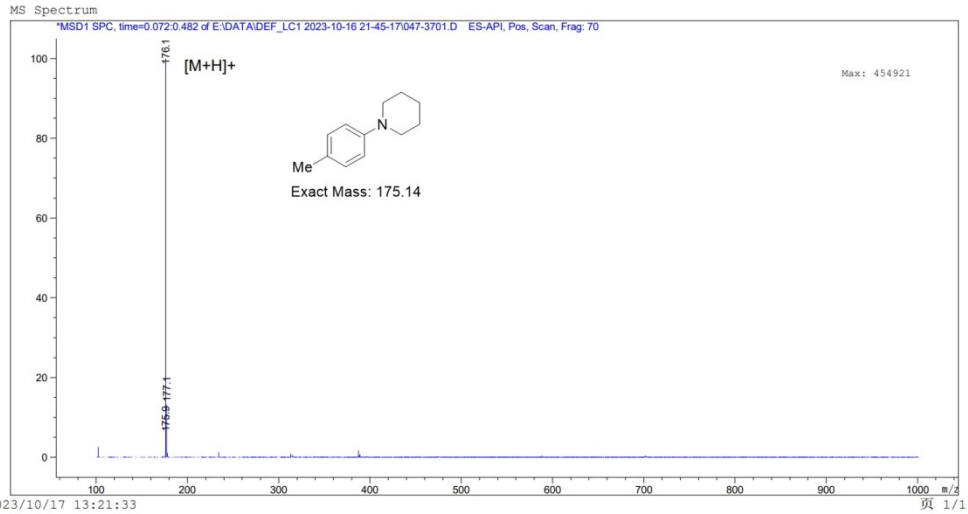
The MS spectrum of **5c**



^{1}H NMR spectrum of **5c** (400 MHz, $CDCl_3$)

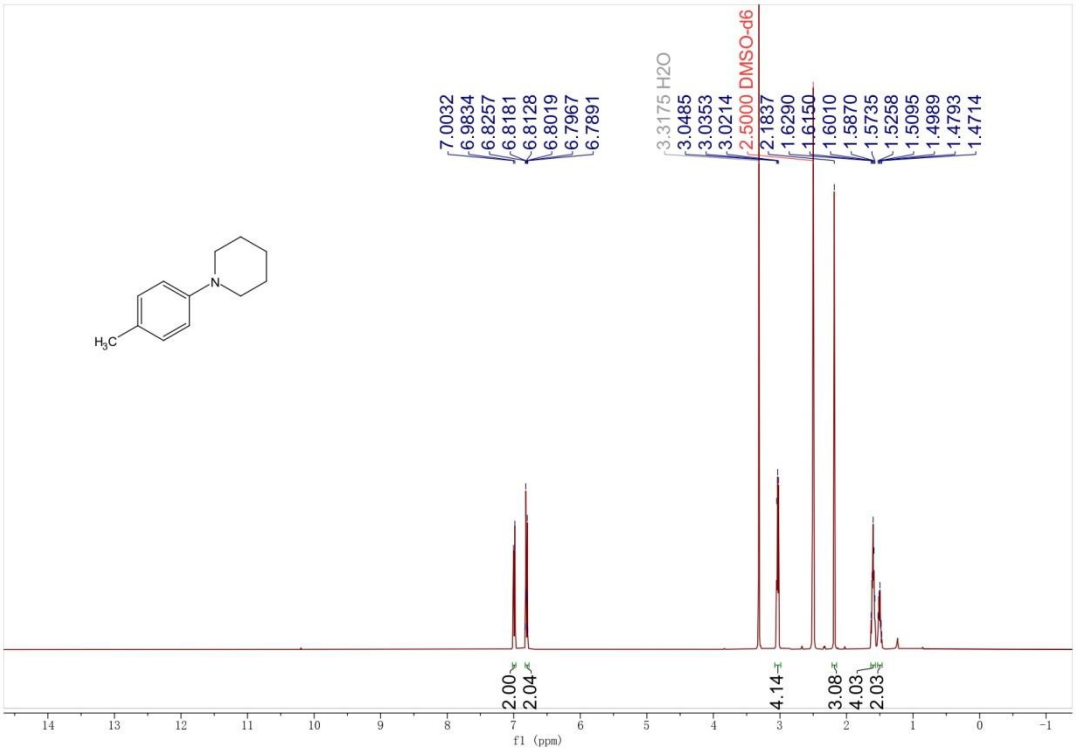
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\047-3701.D
样品名称 : XP-4

操作者 : 序列行 : 37
仪器 : 仪器 1 位置 : 样品瓶 47
进样日期 : 2023/10/17 1:48:31 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F

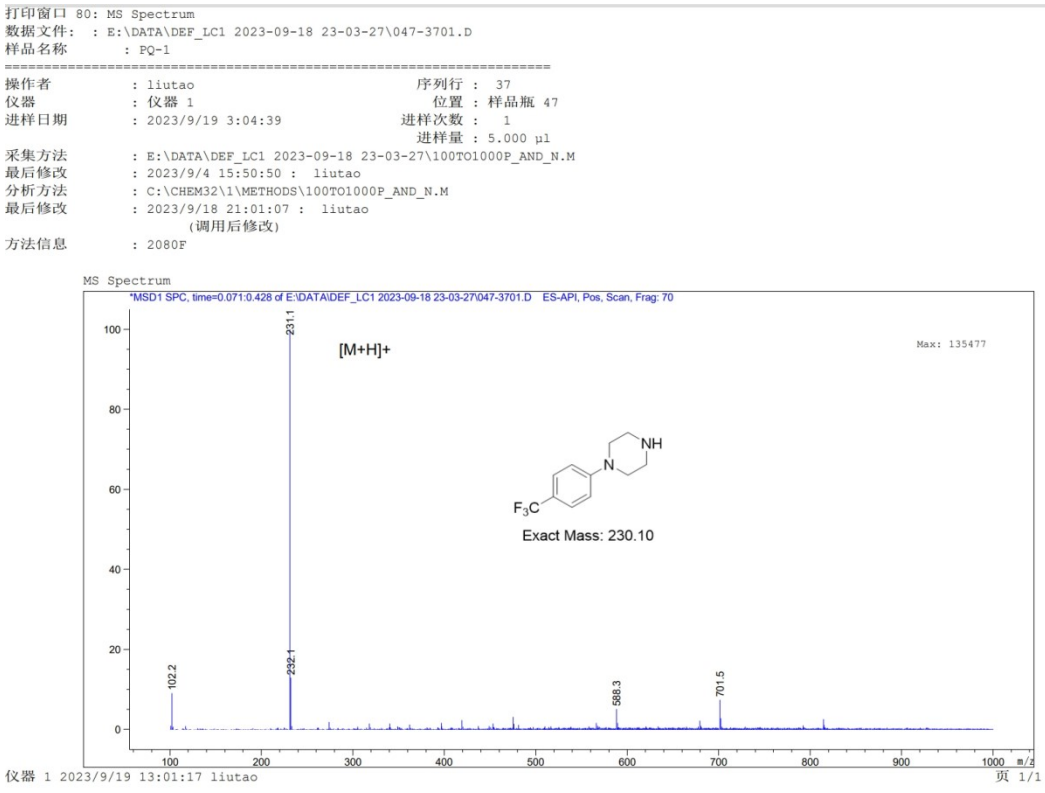


仪器 1 2023/10/17 13:21:33 页 1/1

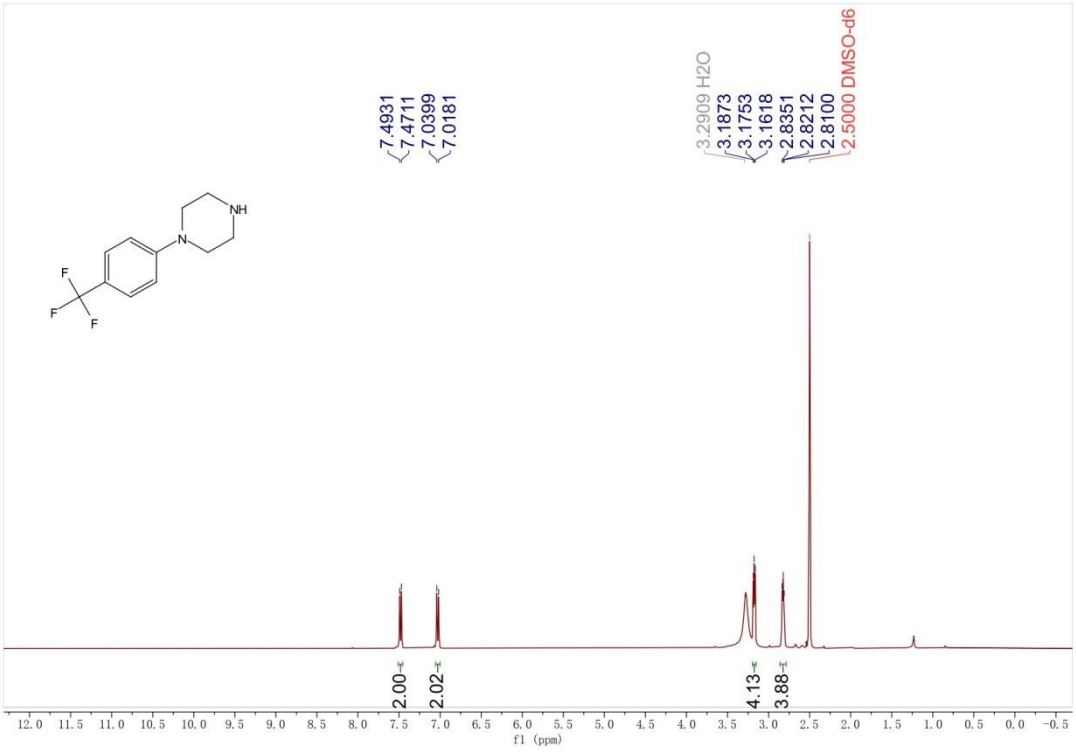
The MS spectrum of **5d**



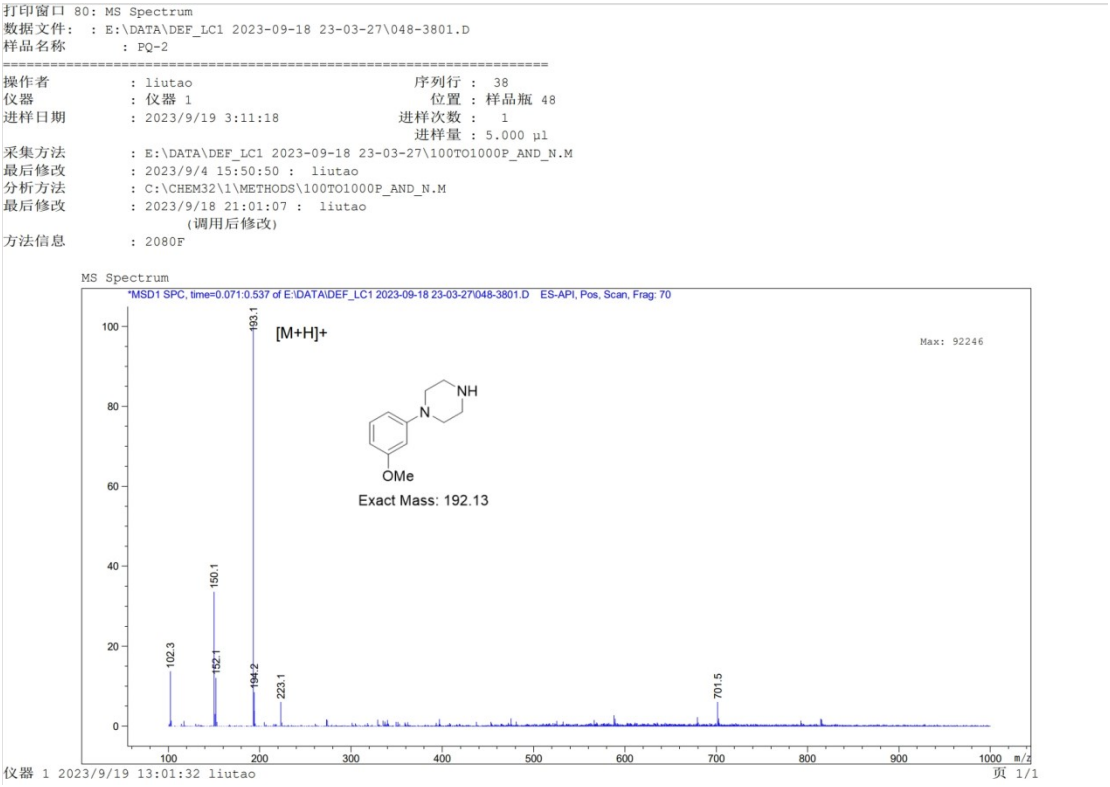
^{1}H NMR spectrum of **5d** (400 MHz, DMSO-d6)



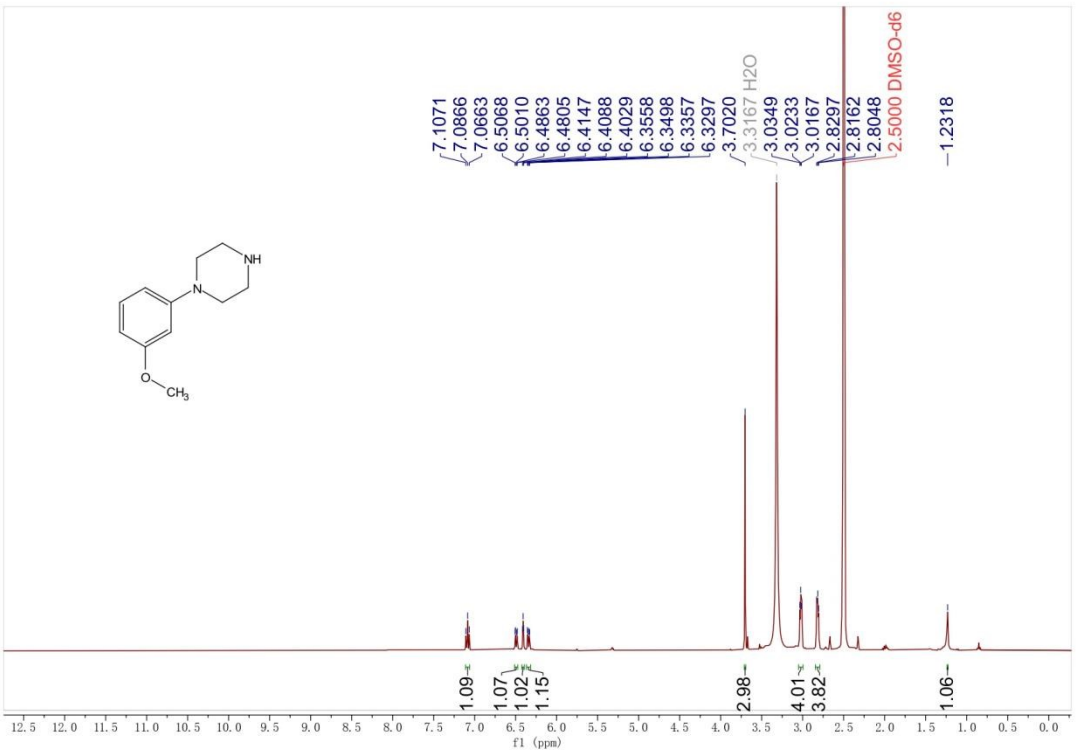
The MS spectrum of **5e**



^{1}H NMR spectrum of **5e** (400 MHz, DMSO-d6)



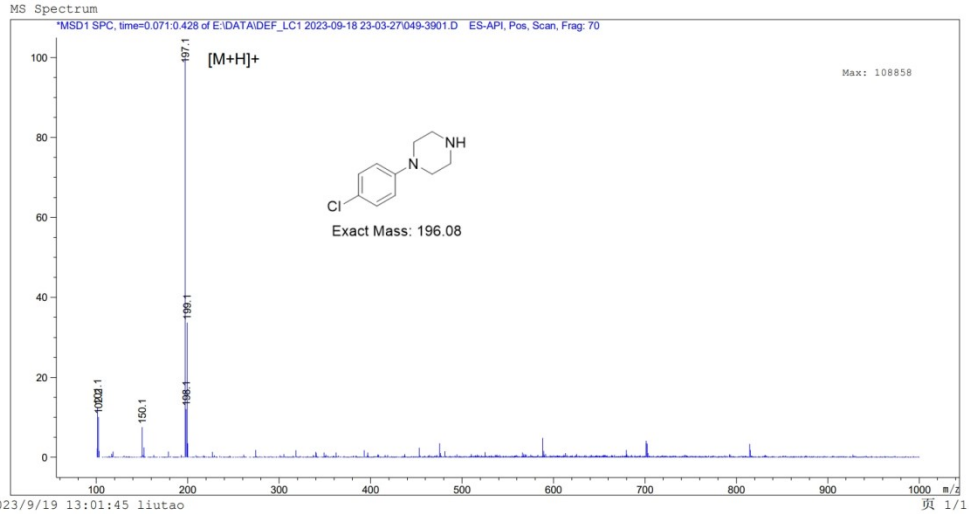
The MS spectrum of **5f**



^{1}H NMR spectrum of **5f** (400 MHz, DMSO-d6)

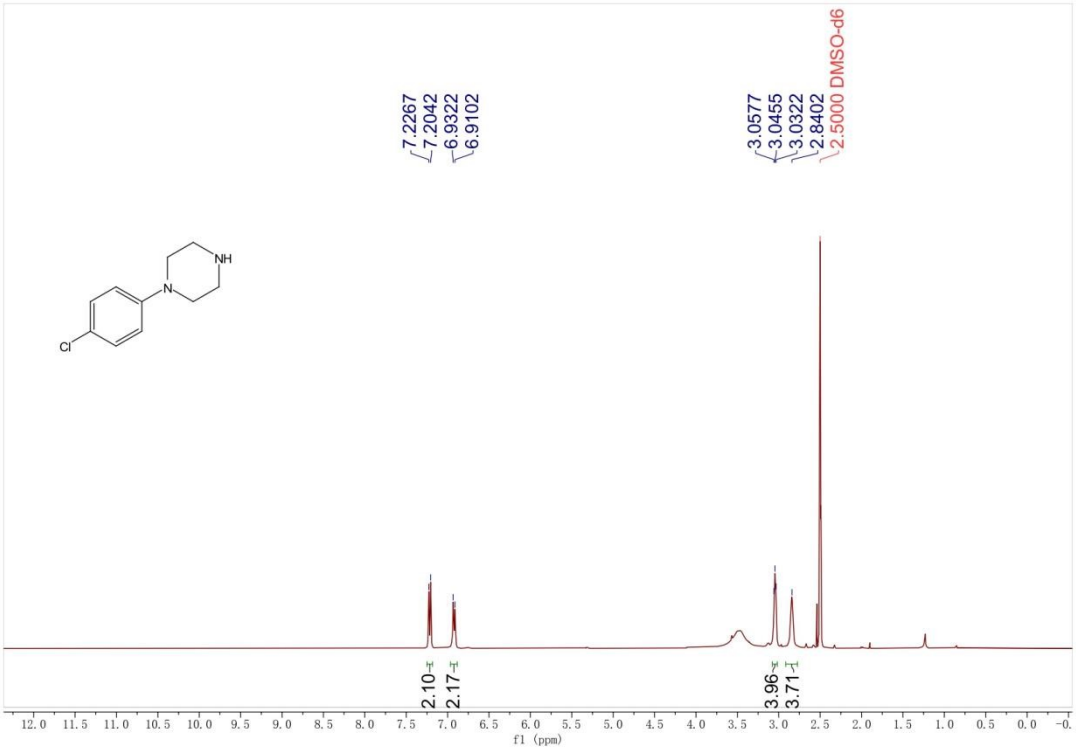
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-09-18 23-03-27\049-3901.D
样品名称 : PQ-3

操作者 : liutao 序列行 : 39
仪器 : 仪器 1 位置 : 样品瓶 49
进样日期 : 2023/9/19 3:17:58 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-09-18 23-03-27\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/9/18 21:01:07 : liutao
(调用后修改)
方法信息 : 2080F



仪器 1 2023/9/19 13:01:45 liutao 页 1/1

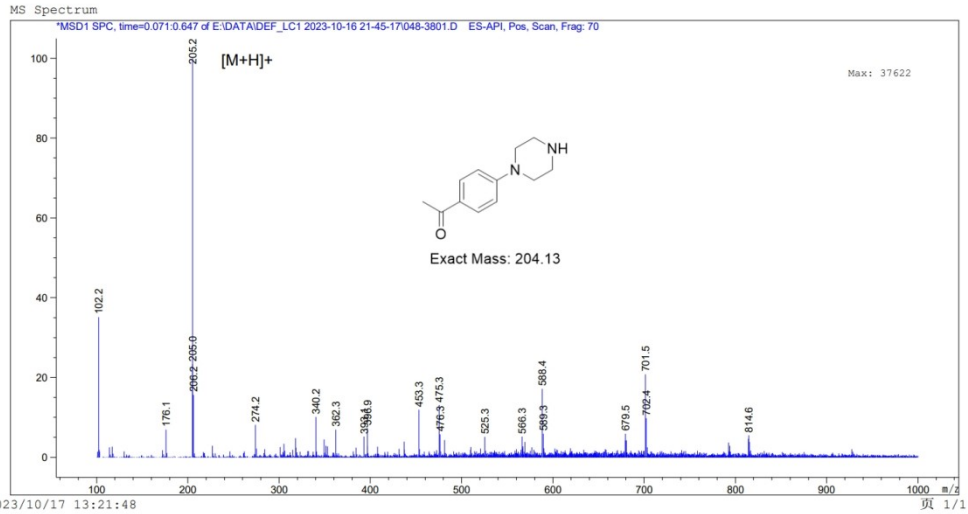
The MS spectrum of **5g**



^{1}H NMR spectrum of **5g**(400 MHz, DMSO-d6)

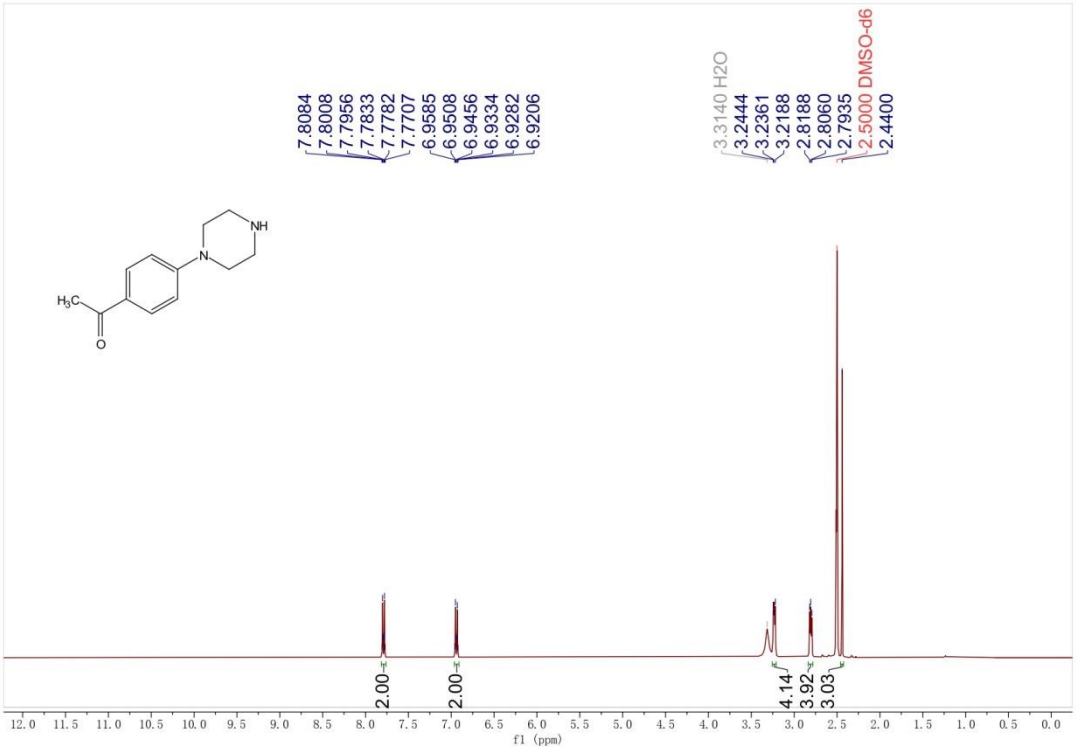
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\048-3801.D
样品名称 : PQ-4

操作者 : 序列行 : 38
仪器 : 仪器 1 位置 : 样品瓶 48
进样日期 : 2023/10/17 1:55:17 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F

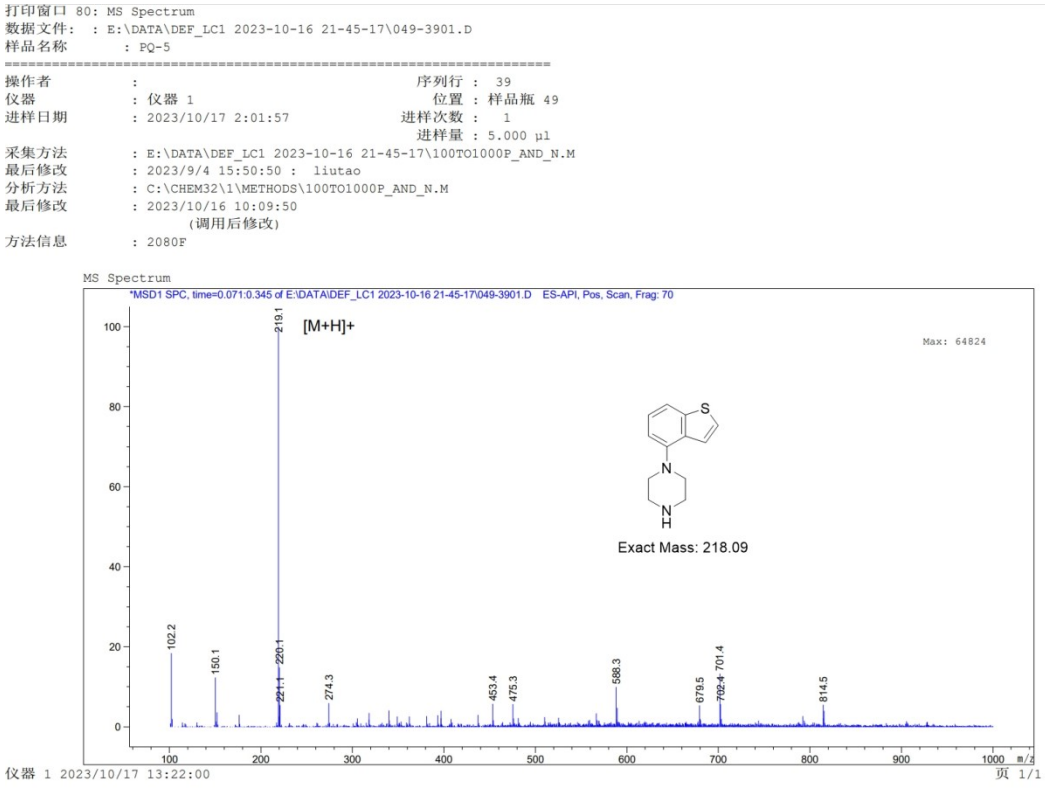


仪器 1 2023/10/17 13:21:48 页 1/1

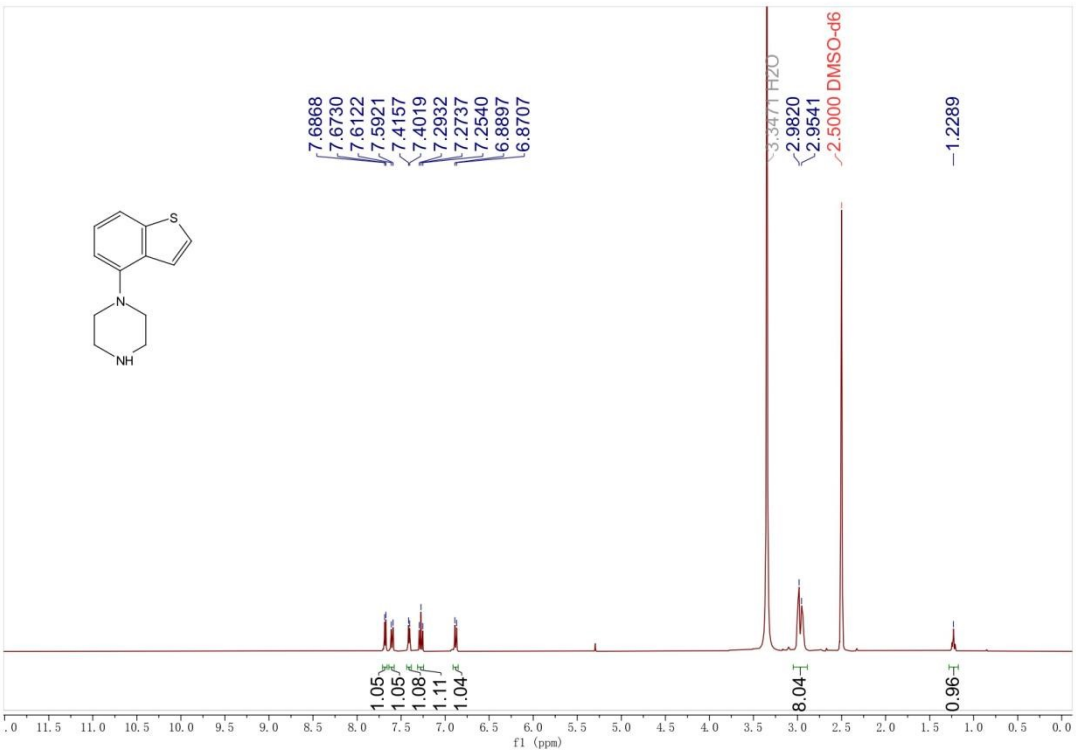
The MS spectrum of **5h**



^{1}H NMR spectrum of **5h**(400 MHz, DMSO-d6)



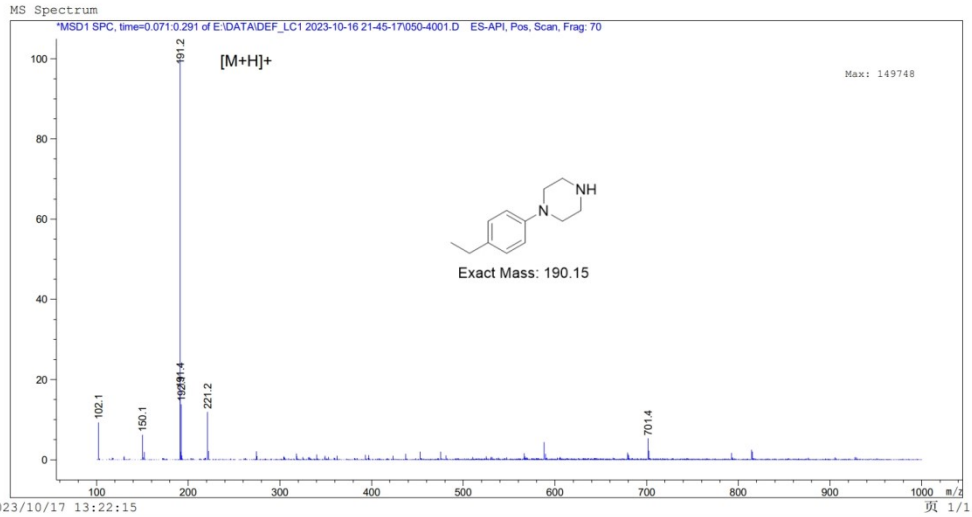
The MS spectrum of **5i**



^{1}H NMR spectrum of **5i**(400 MHz, DMSO-d6)

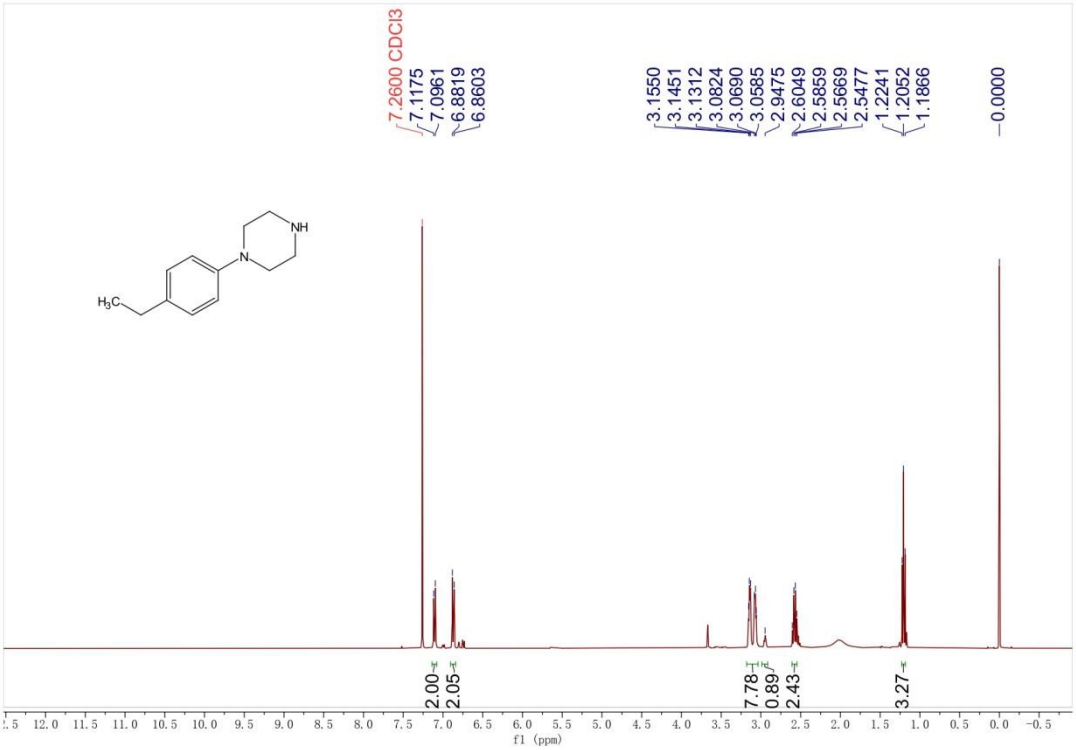
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\050-4001.D
样品名称 : PQ-7

操作者 : 序列行 : 40
仪器 : 仪器 1 位置 : 样品瓶 50
进样日期 : 2023/10/17 2:08:42 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F



仪器 1 2023/10/17 13:22:15 页 1/1

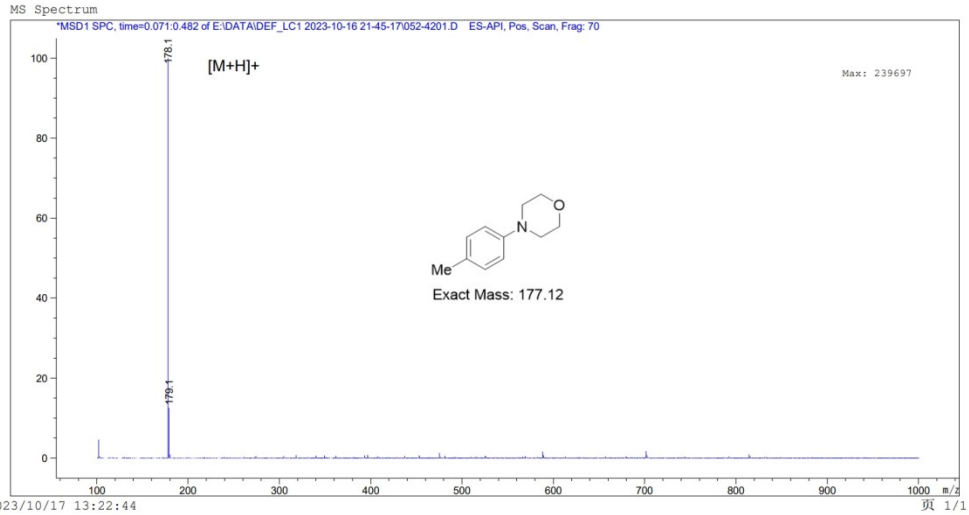
The MS spectrum of **5j**



^{1}H NMR spectrum of **5j**(400 MHz, $CDCl_3$)

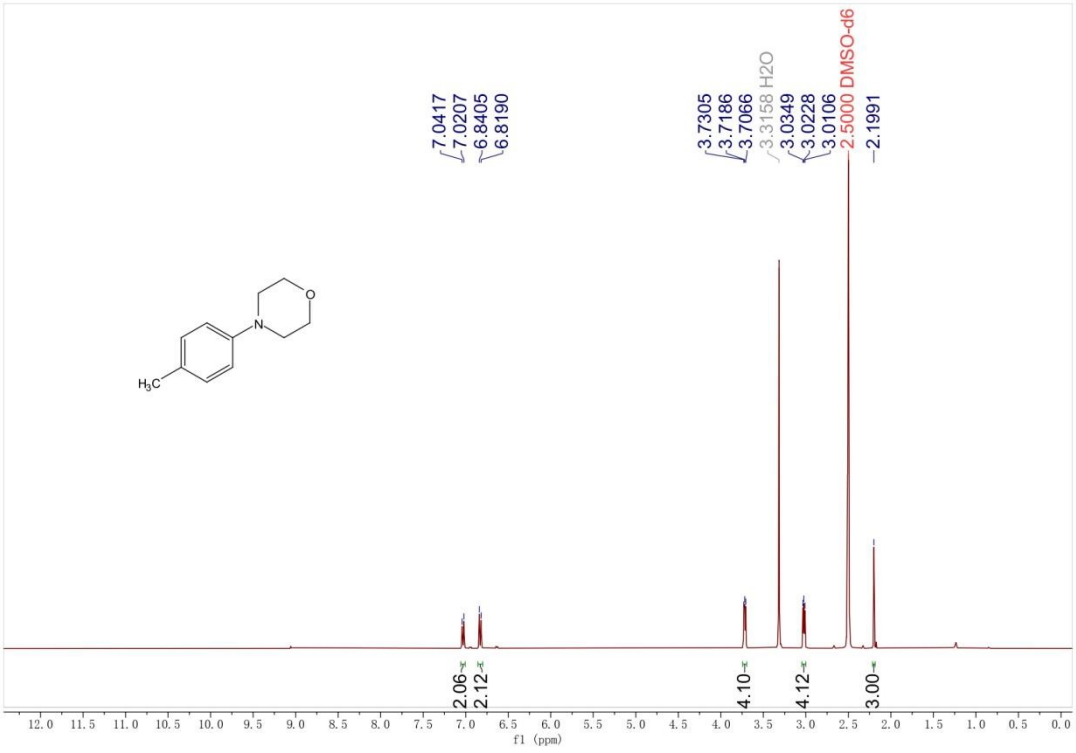
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\052-4201.D
样品名称 : ML-2

操作者 : 序列行 : 42
仪器 : 仪器 1 位置 : 样品瓶 52
进样日期 : 2023/10/17 2:22:05 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F



仪器 1 2023/10/17 13:22:44 页 1/1

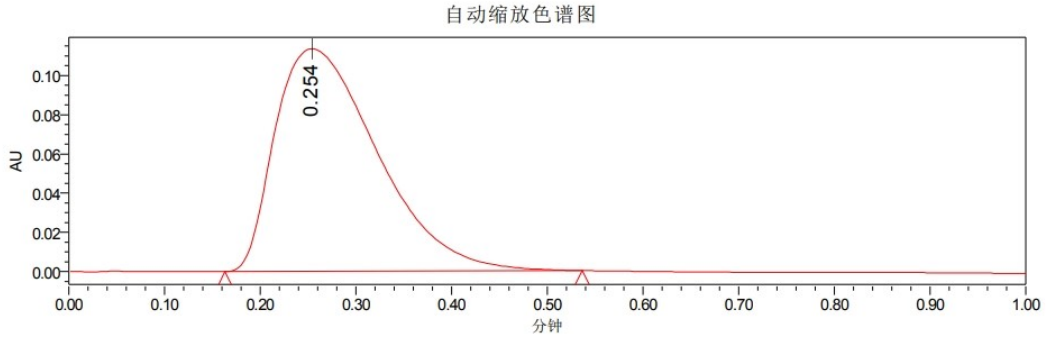
The MS spectrum of **5k**

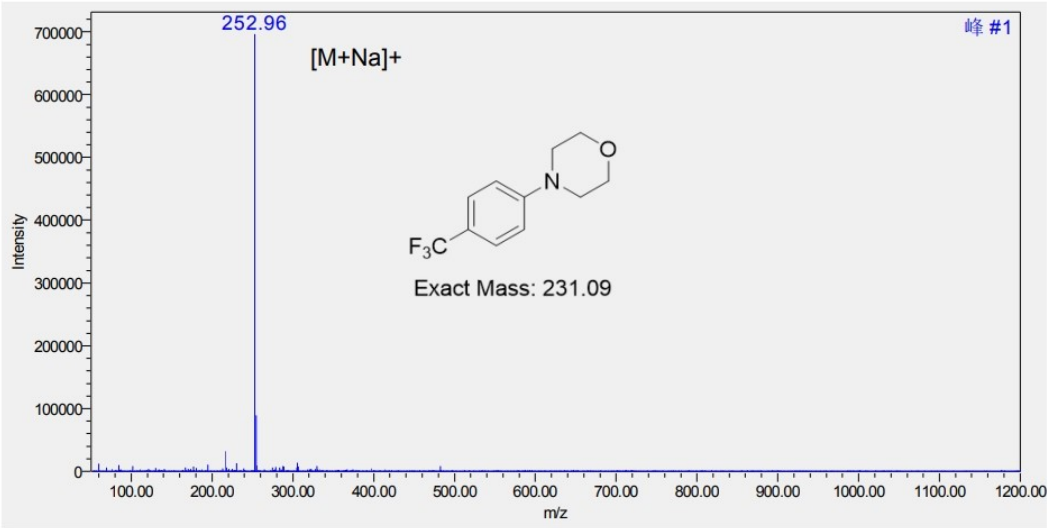


^{1}H NMR spectrum of **5k**(400 MHz, DMSO-d6)

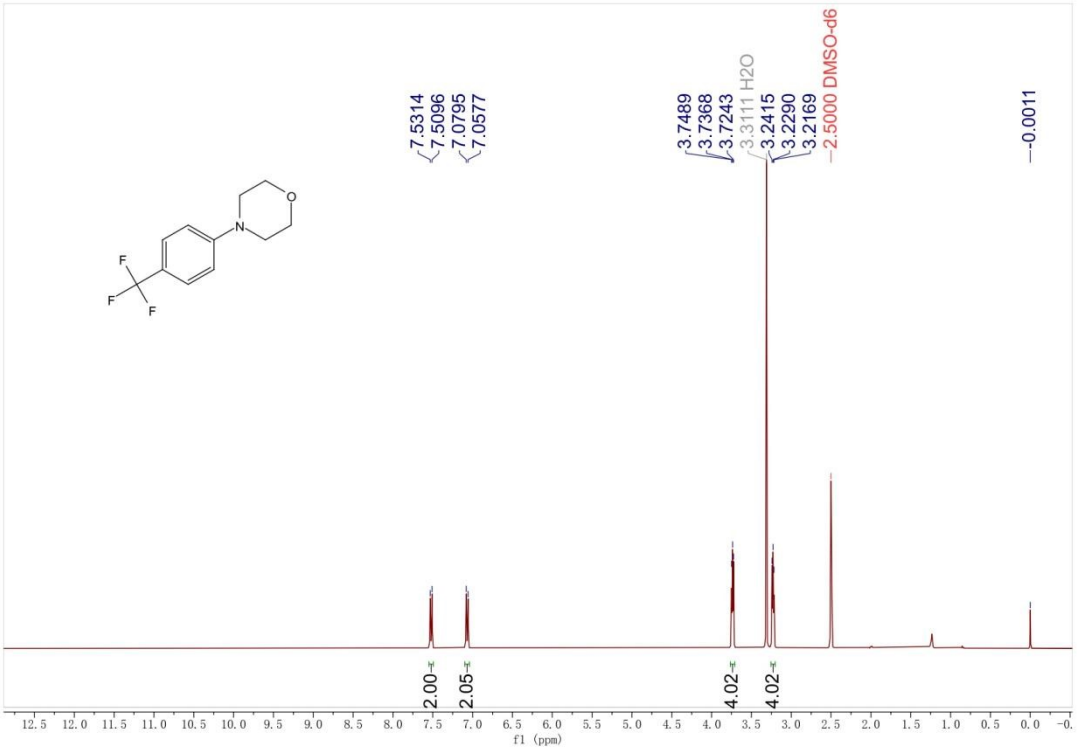
样品信息

样品名称:	ML-2	采集者:	test1
样品类型:	标准样	样品组名称:	20240110
瓶号:	1:A,5	采集方法组:	MS_ZHENG
进样次数:	1	处理方法:	MS_YY
进样体积:	15.00 ul	通道名称:	W2489 ChA
运行时间:	1.0 Minutes	处理通道注释:	W2489 ChA 210nm
采集时间:	2024/1/10 13:42:25 CST		
处理时间:	2024/1/10 13:43:35 CST		

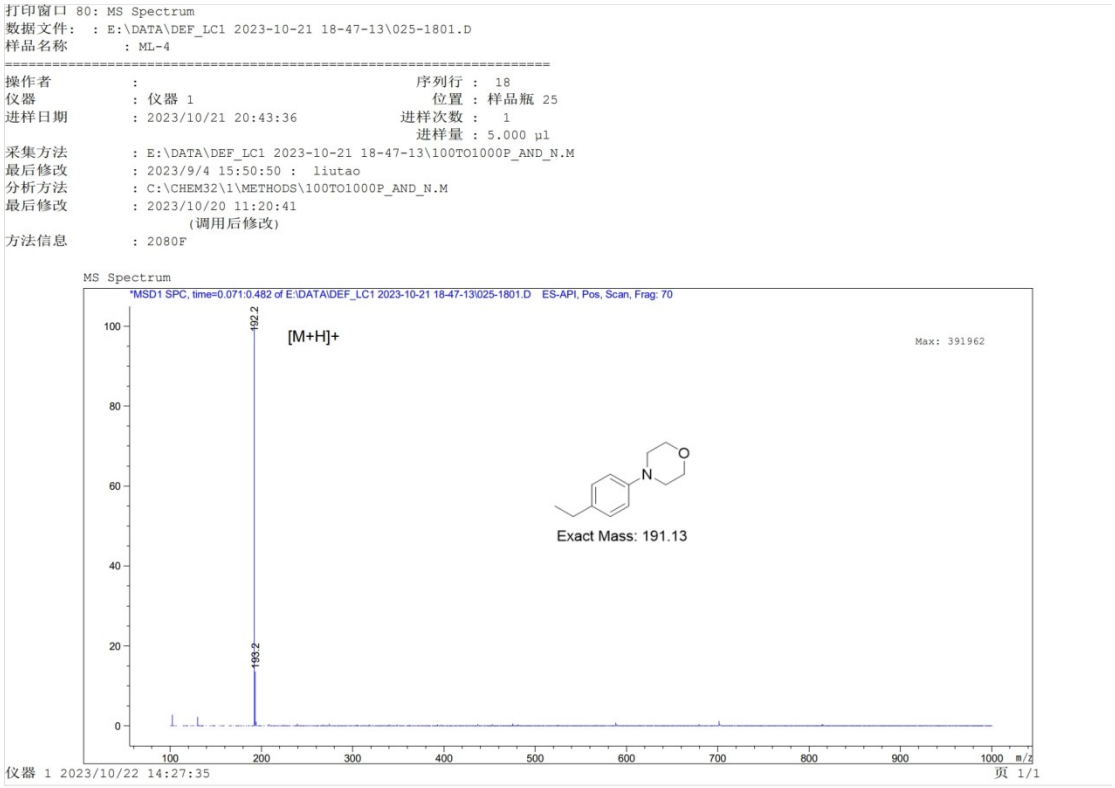




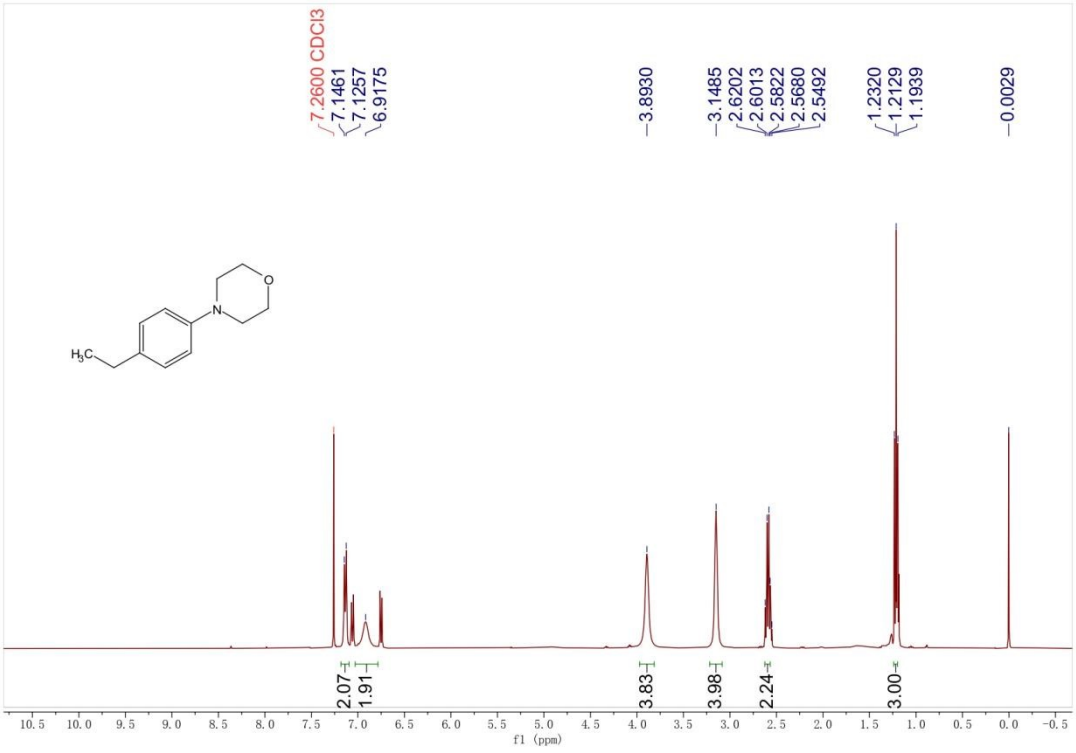
The MS spectrum of **5l**



^{1}H NMR spectrum of **5l**(400 MHz, DMSO-d6)



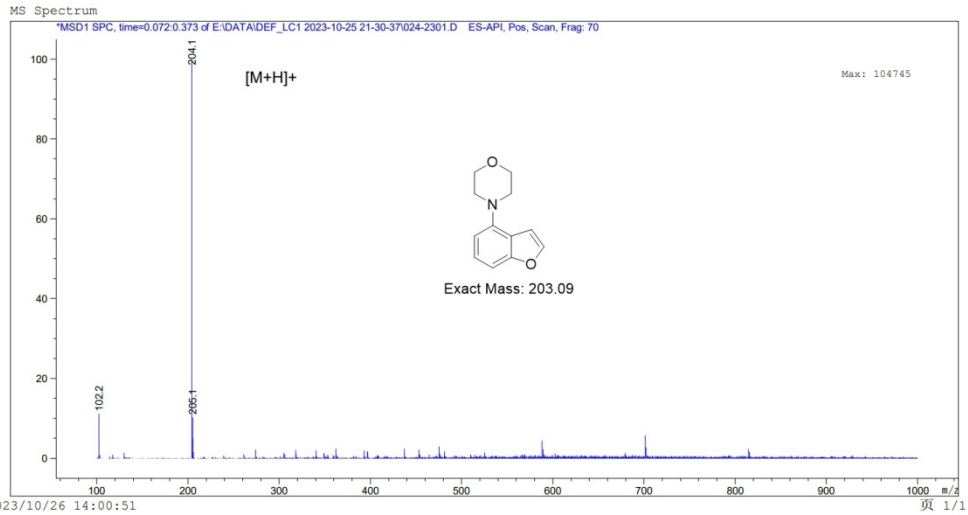
The MS spectrum of **5m**



^{1}H NMR spectrum of **5m**(400 MHz, $CDCl_3$)

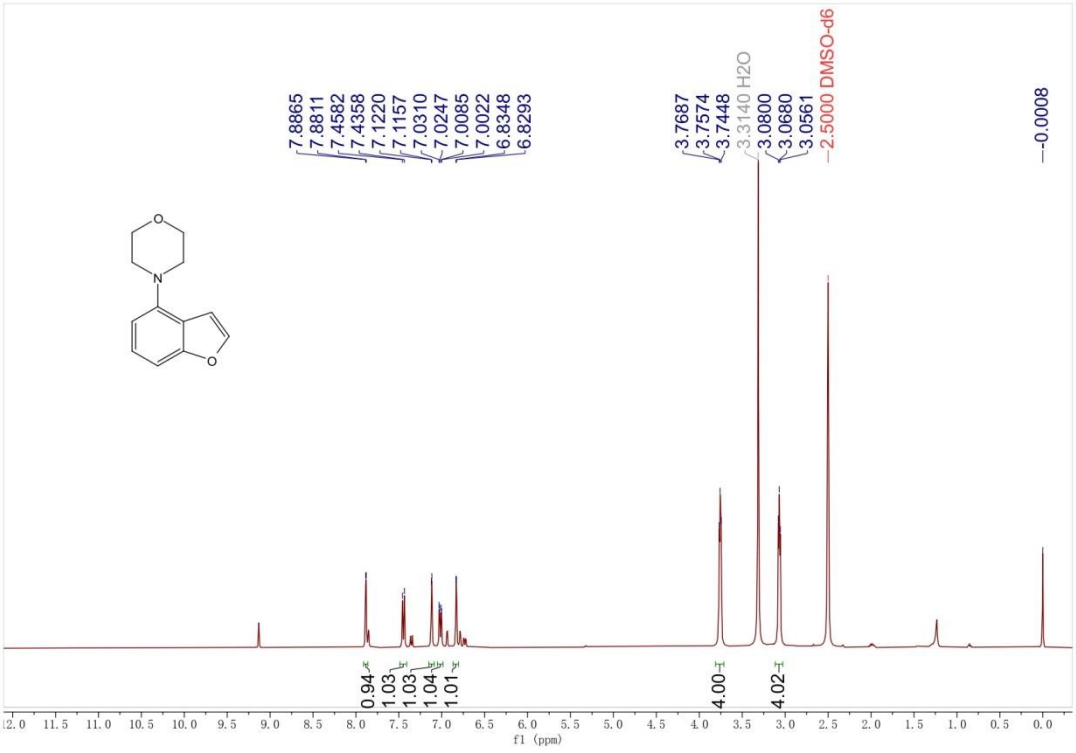
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-25 21-30-37\024-2301.D
样品名称 : ML-5

操作者 : 序列行 : 23
仪器 : 仪器 1 位置 : 样品瓶 24
进样日期 : 2023/10/26 0:00:24 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-25 21-30-37\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/25 9:14:42
(调用后修改)
方法信息 : 2080F



仪器 1 2023/10/26 14:00:51 页 1/1

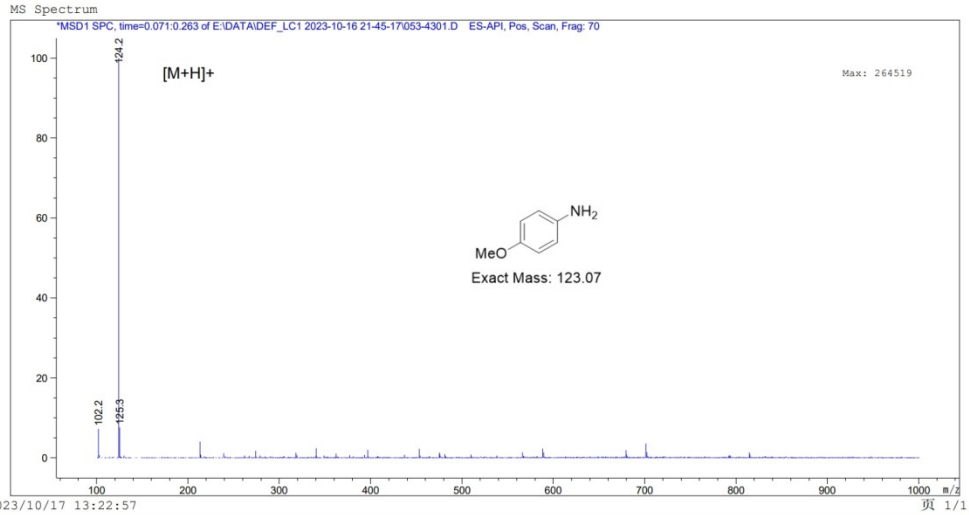
The MS spectrum of **5n**



^{1}H NMR spectrum of **5n**(400 MHz, DMSO-d6)

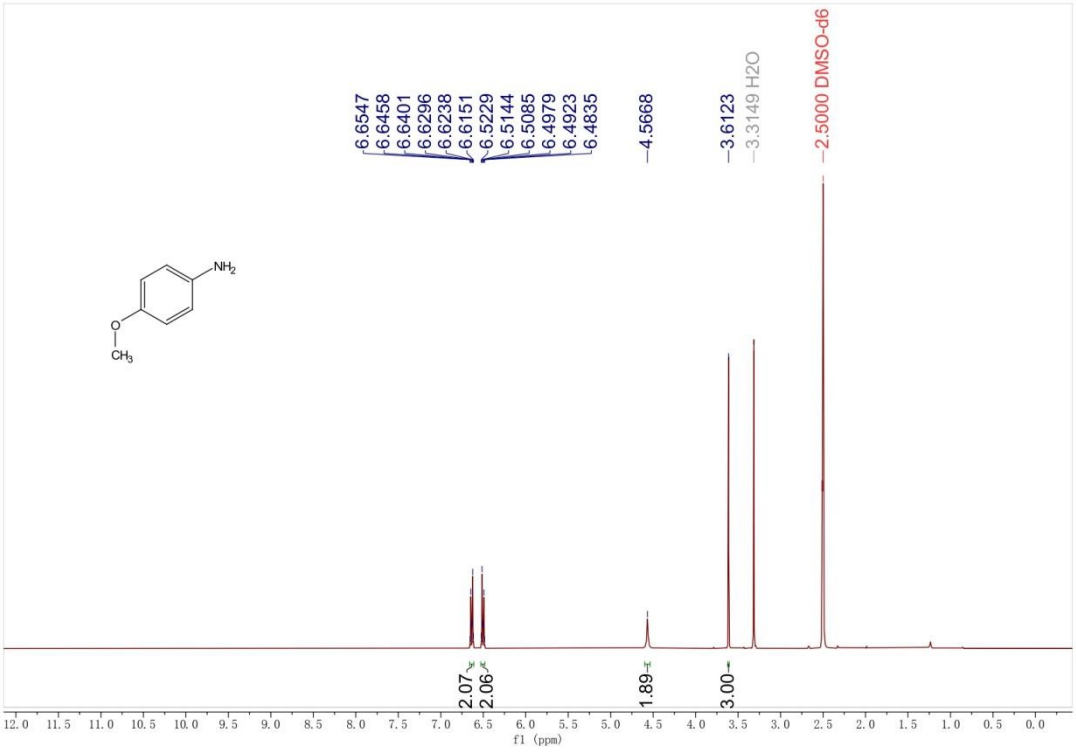
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\053-4301.D
样品名称 : XA-1

操作者 : 序列行 : 43
仪器 : 仪器 1 位置 : 样品瓶 53
进样日期 : 2023/10/17 2:28:49 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F

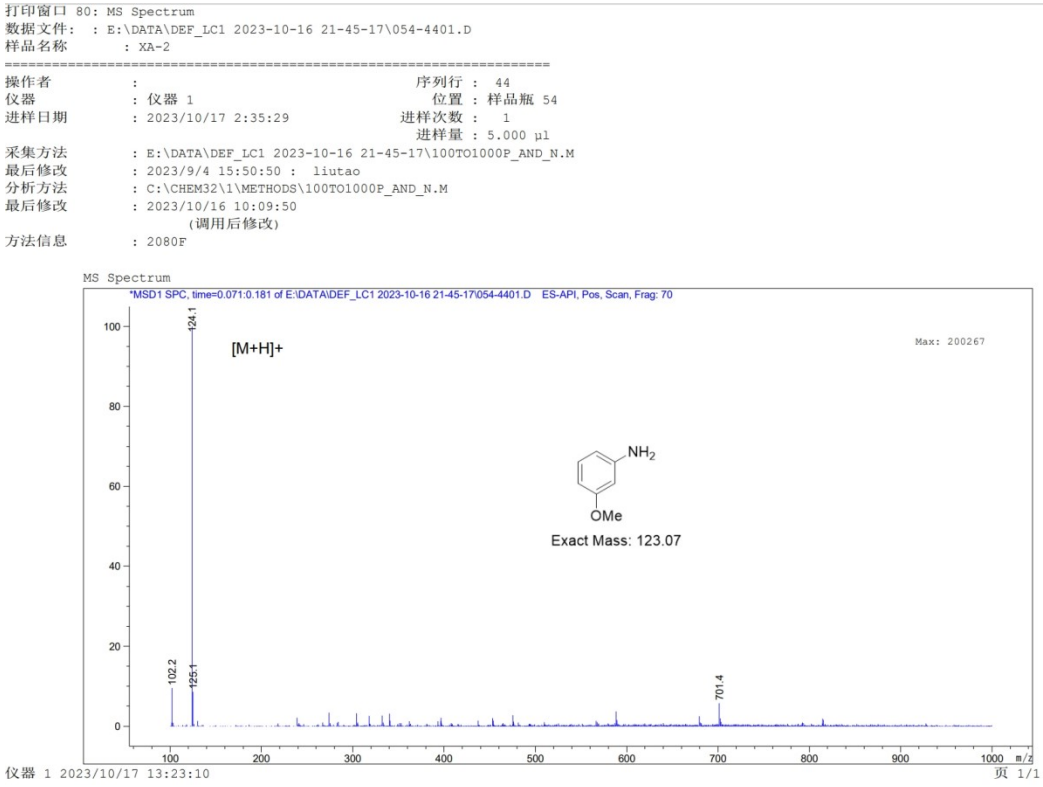


仪器 1 2023/10/17 13:22:57 页 1/1

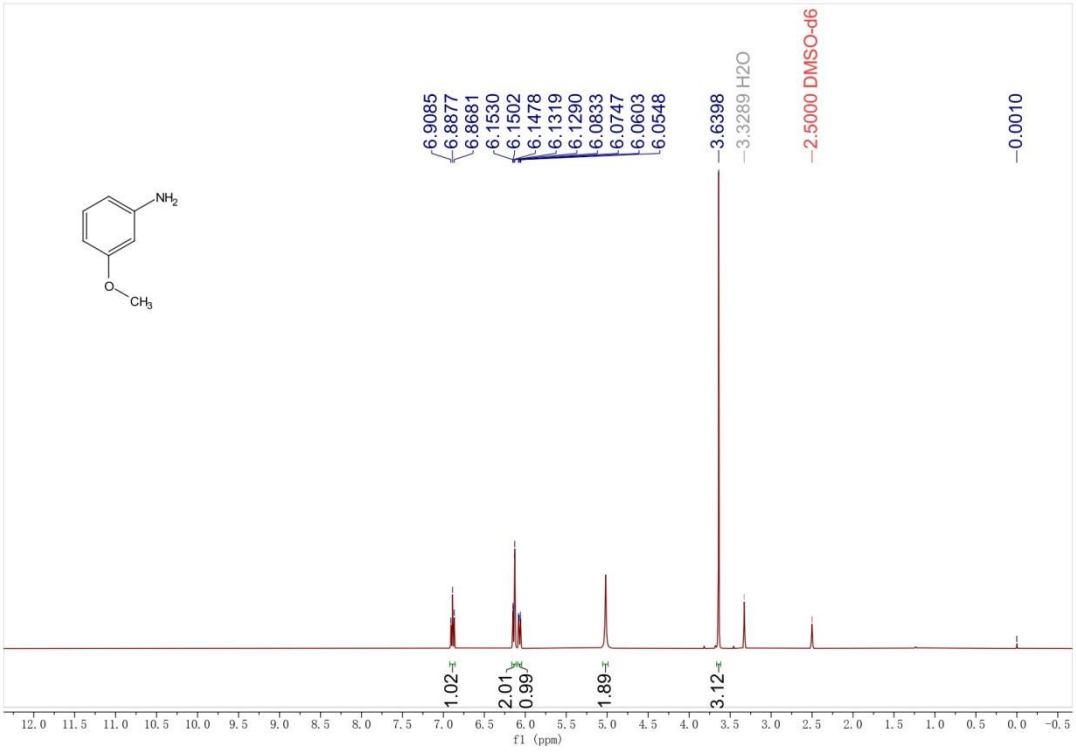
The MS spectrum of **6a**



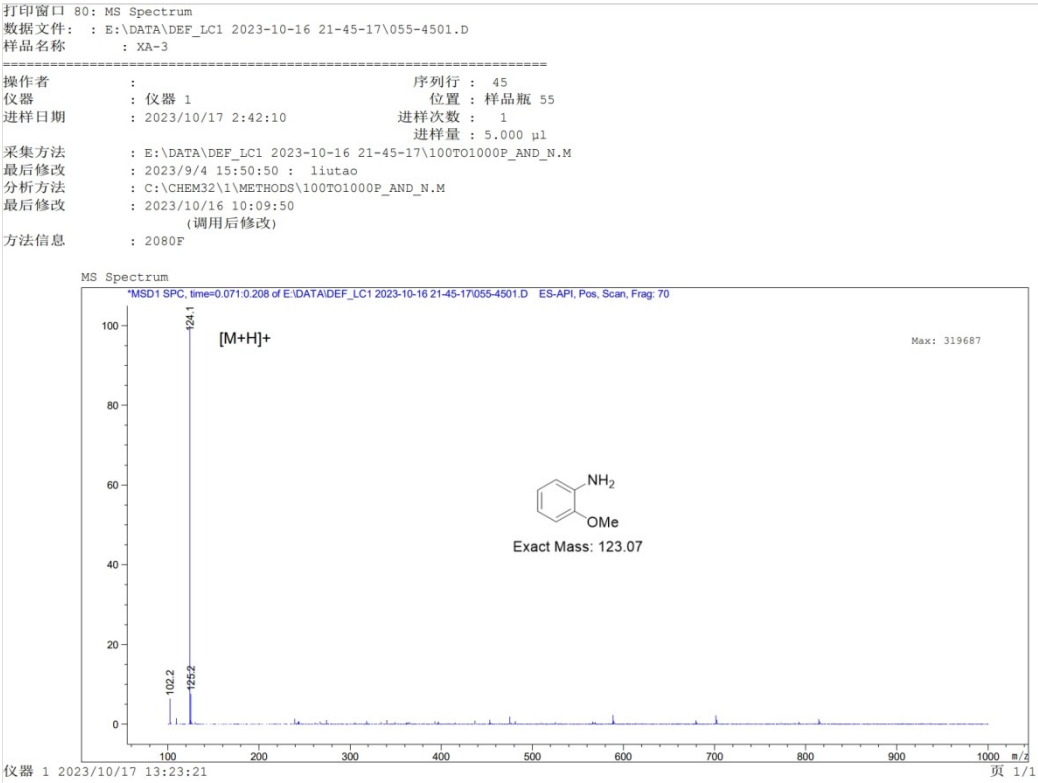
^{1}H NMR spectrum of **6a**(400 MHz, DMSO-d6)



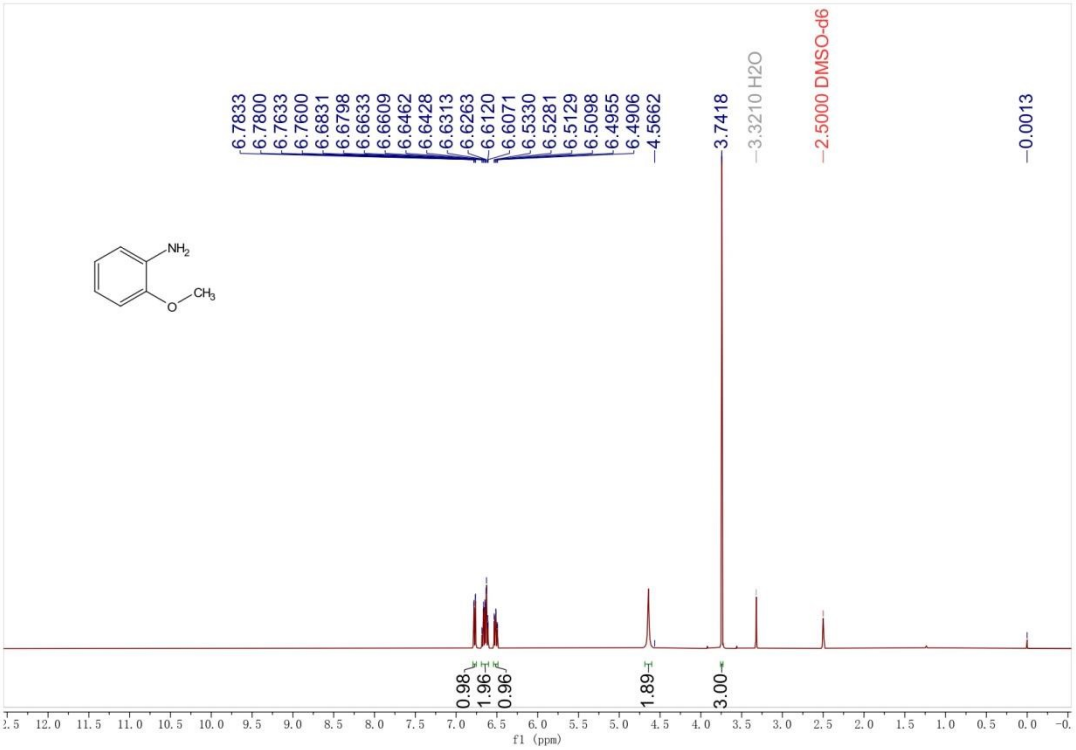
The MS spectrum of **6b**



^{1}H NMR spectrum of **6b**(400 MHz, DMSO-d6)



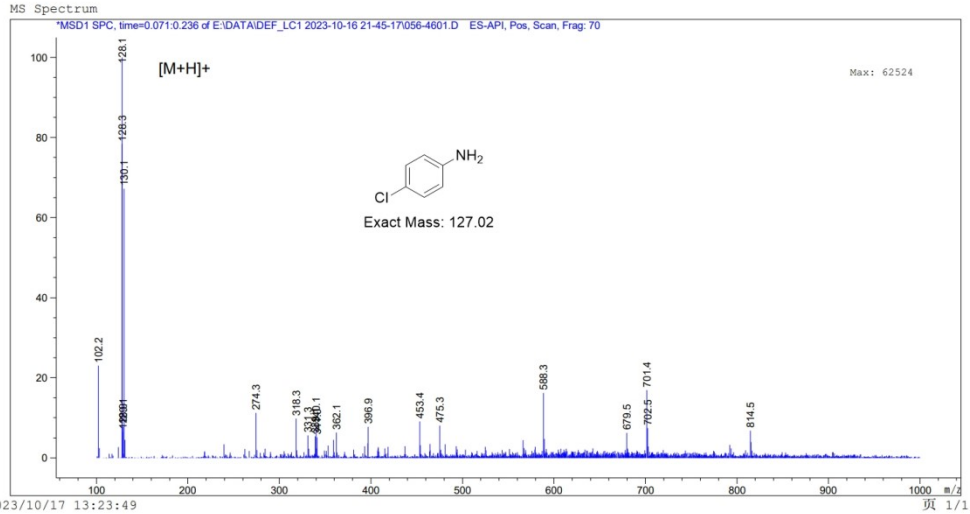
The MS spectrum of **6c**



^{1}H NMR spectrum of **6c**(400 MHz, DMSO-d6)

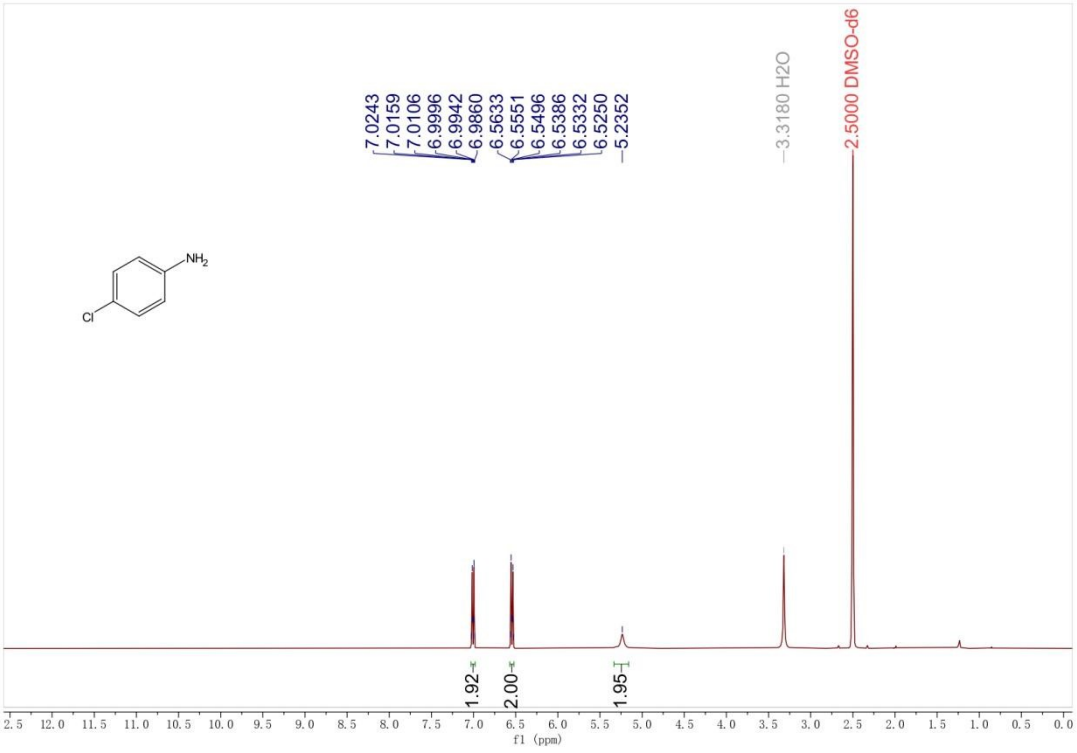
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2023-10-16 21-45-17\056-4601.D
样品名称 : XA-4

操作者 : 序列行 : 46
仪器 : 仪器 1 位置 : 样品瓶 56
进样日期 : 2023/10/17 2:48:52 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2023-10-16 21-45-17\100TO1000P_AND_N.M
最后修改 : 2023/9/4 15:50:50 : liutao
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2023/10/16 10:09:50
(调用后修改)
方法信息 : 2080F

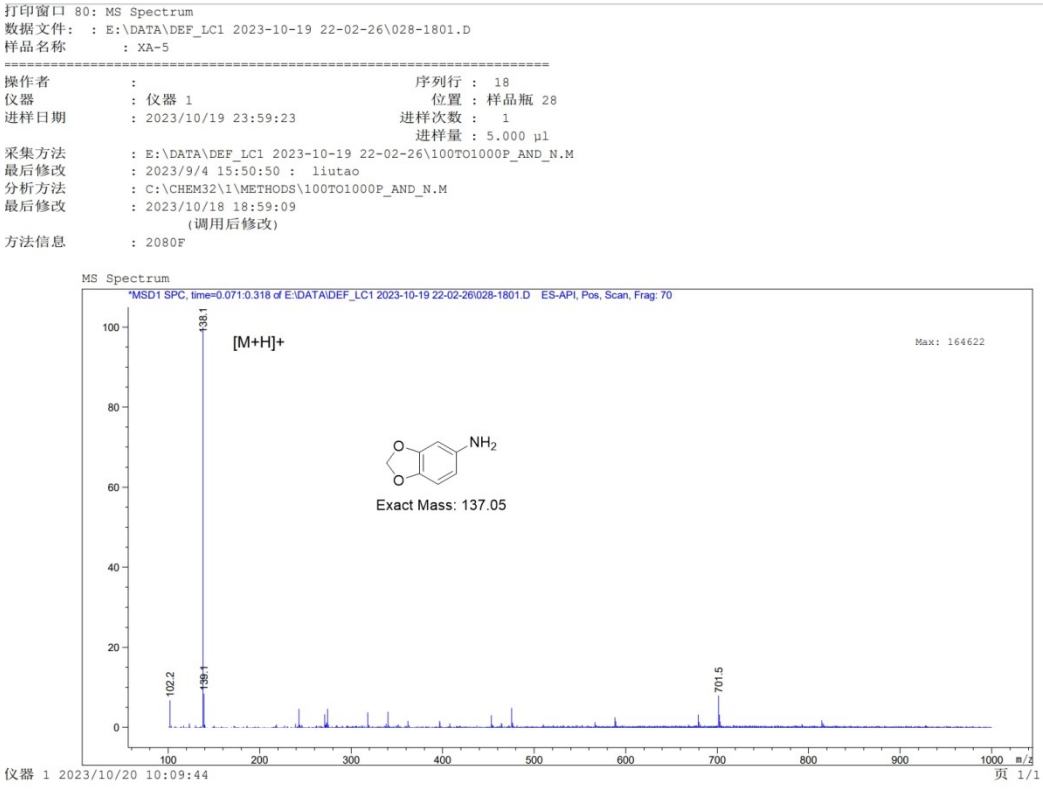


仪器 1 2023/10/17 13:23:49 页 1/1

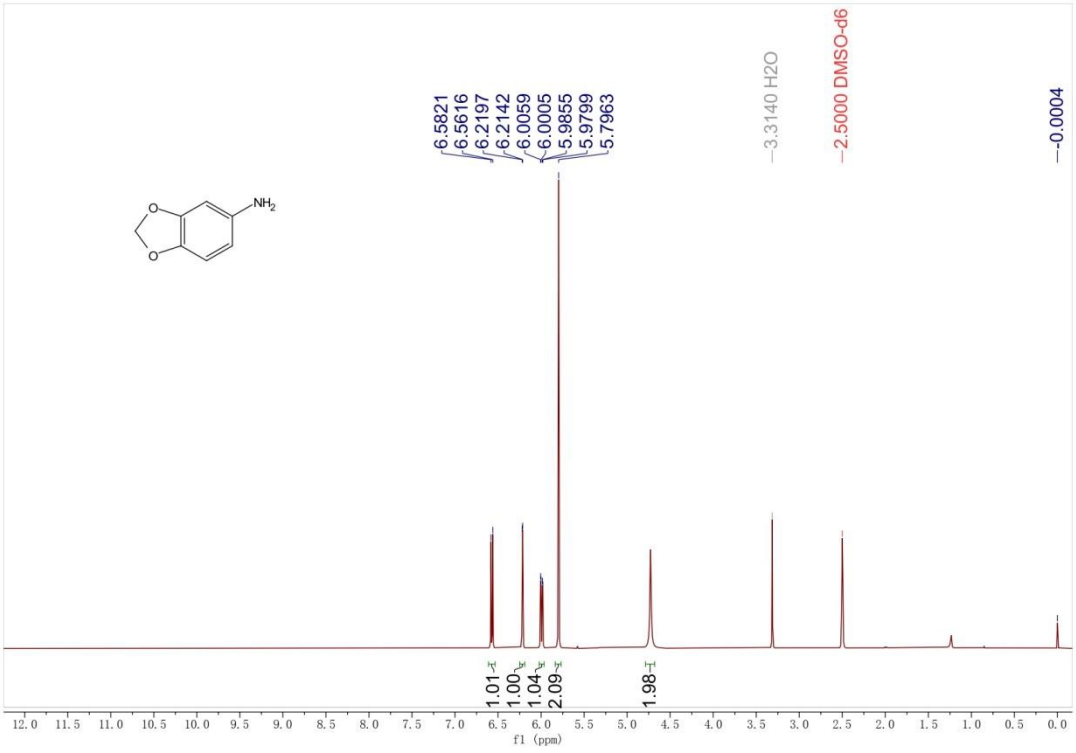
The MS spectrum of **6d**



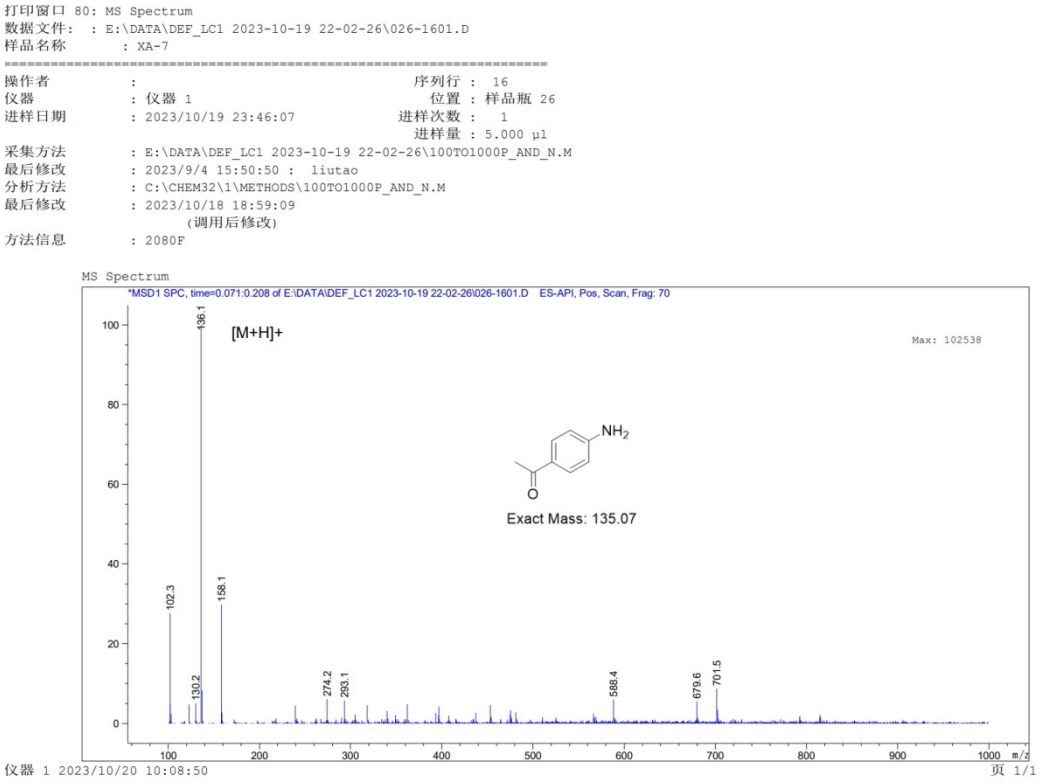
^{1}H NMR spectrum of **6d**(400 MHz, DMSO-d6)



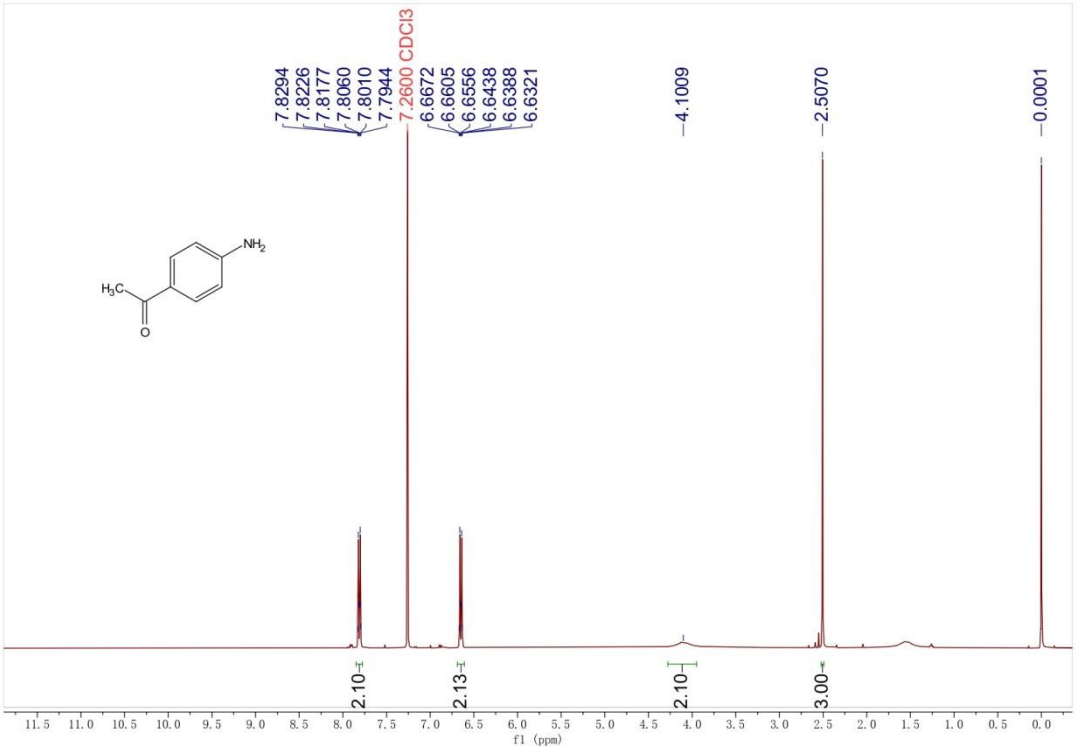
The MS spectrum of **6e**



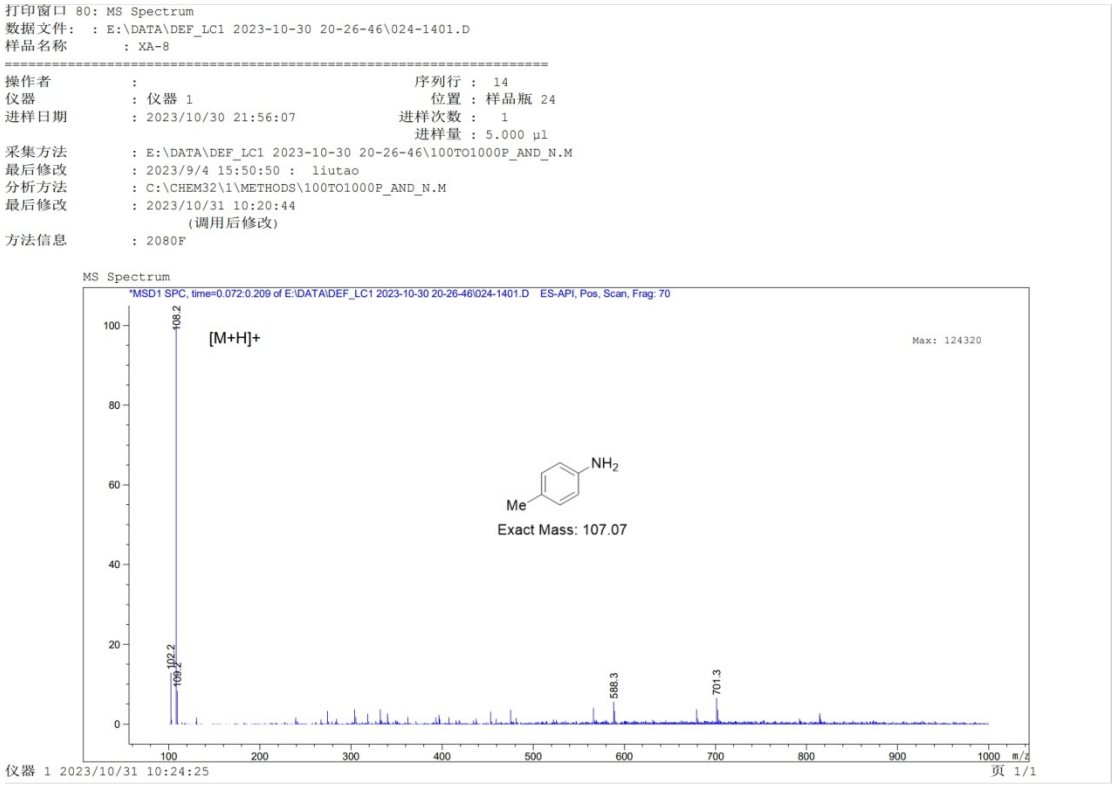
^{1}H NMR spectrum of **6e**(400 MHz, DMSO-d6)



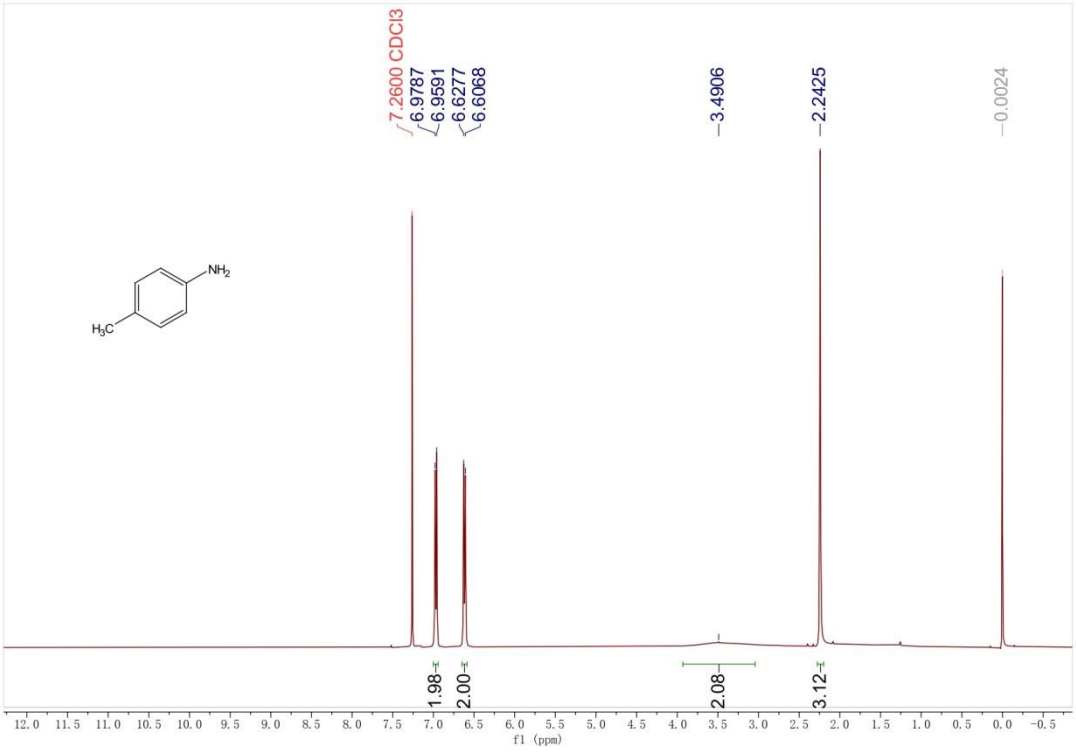
The MS spectrum of **6f**



^{1}H NMR spectrum of **6f**(400 MHz, $CDCl_3$)



The MS spectrum of **6g**



^{1}H NMR spectrum of **6g**(400 MHz, $CDCl_3$)