Cu(II)-Catalyzed C6-Selective C-H Thiolation of 2-Pyridones Using Air as the Oxidant

Supporting Information

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Contents

1. **General Information** S2
2. **Experimental Procedures** S2
3. **Mechanistic Investigations** S3
4. **References** S4
5. **X-ray Single Crystal Structure Analysis of 3a** S4
6. **Analytical Characterization Data of the Products** S5
7. **Copies of ^{1}H NMR and ^{13}C NMR Spectra for the Products** S16

1. General Information.

The chemicals were purchased from commercial sources and used without further purification. Analytical thin layer chromatography (TLC) was performed on 0.15−0.2 mm thickness silica gel plates. All products were characterized by NMR and MS spectra. ^{1}H and ^{13}C NMR spectra were recorded in deuterochloroform ($CDCl_3$) on 400 or 500MHz instruments. Chemical shifts were reported in parts per million (ppm, δ) downfield from tetramethylsilane. Proton coupling patterns were described as singlet (s), doublet (d), triplet (t), quartet (q), quintet (p), doublet of triplets (dt) and multiplet(m). High-resolution mass spectra (HRMS) was measured on Q-TOF spectrometer. The determination of de was performed via LC/MS analysis. Melting points were measured on melting point apparatus.

2. Experimental Procedures.

General Procedure for the Preparation of 1-(2-Pyridyl)-2-pyiridones 1[1]

Synthesis of **1a** is representative. To a mixture of 2-hydroxypyridine (950 mg, 10 mmol), copper(I) iodide (95 mg, 0.5 mmol), and potassium phosphate (4.2 g, 20 mmol) in toluene (20 mL) were added *N,N'*-dimethylethylenediamine (DMEDA, 0.1 mL, 1 mmol) and 2-bromopyridine (1.9 mL, 20 mmol), and the resulting mixture was stirred under N_2 at 120 ^{o}C for 20 h. The resulting mixture was allowed to cool to room temperature and then diluted with ethyl acetate and washed by water. A small amount of ethylenediamine was added to dissolve the residual copper salts. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (PE/EA = 2/1- 1/1) to give 2*H*-[1,2'-bipyridin]-2-one (**1a**, 1.2 g, 7.0 mmol) in 70% yield.

Copper-Catalyzed C6-Selective C-H Thiolation of 2-Pyridones with disulfides 2

Synthesis of **3a** is representative. The dry sealed tube was charged with 2*H*-[1,2'-bipyridin]-2-one (**1a**, 34.4 mg, 0.2 mmol), diphenyl disulfide (**2a**, 43.6 mg, 0.2 mmol), $Cu(OAc)_2$ (7.3 mg, 0.04 mmol), 2-biphenylcarboxylic acid (7.9 mg, 0.04 mmol), and toluene (2mL). The resulting mixture was stirred at 130°C for 20 h. The mixture was diluted with ethyl acetate and washed by water. A small amount of ethylenediamine was added to dissolve the residual copper salts. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (PE/EA = 2/1- 1/1) to give 6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one(**3a**, 49.3 mg, 0.18 mmol) in 88% yield.

Preparation of Sulfone 5a

3-Chloroperoxybenzoic acid (82.8 mg, 0.48 mmol) was added to a solution of product **3a** (56 mg, 0.2 mmol) in chloroform (3 mL) at room temperature. After the

addition was complete, the reaction mixture was allowed to stir at room temperature for 20 h. The reaction mixture was washed with water/NaOH. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (PE/EA = 1/1-1/2) to give 6-(phenylsulfonyl)-2*H*-[1,2'-bipyridin]-2-one (**5a**, 47.4 mg, 0.15 mmol) in 75% yield.

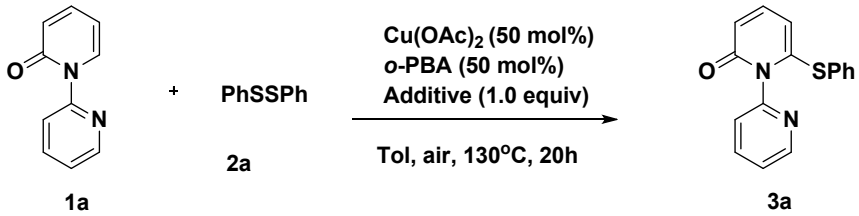
Removal of Directing Group[1]

6-(phenylthio)-2H-[1,2'-bipyridin]-2-one (**3a**, 56 mg, 0.2 mmol) was placed in a dry sealed tube, which was filled with nitrogen. The tube was cooled to 0°C with an ice water bath, and CH_2Cl_2 (3 mL) and methy trifluoromethanesulfonate (24.75 μL, 0.22 mmol) were then added to the tube. The mixture was allowed to warm to room temperature and stirred for 20 h. Volatile materials were evaporated in vacuo, and potassium *tert*-butoxide (68 mg, 0.6 mmol) was added to the tube, which was again filled with nitrogen. EtOH (0.4 mL) and Et_2O (1.6 mL) were added, and the resulting suspension was stirred for 4 h at room temperature. The mixture was diluted with CH_2Cl_2 and washed by water. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. Anion-exchange resin purification afforded 6-(phenylthio)pyridin-2(1*H*)-one (**6a**, 26.4 mg, 0.13 mmol) in 65% yield.

3. Mechanistic Investigations

Radical scavenger experiments:

The dry sealed tube was charged with **1a** (34.4 mg, 0.2 mmol), diphenyl disulfide (**2a**, 43.6 mg, 0.2 mmol), additive (0.2 mmol), $Cu(OAc)_2$ (18.2 mg, 0.1 mmol), 2-biphenylcarboxylic acid (19.8 mg, 0.1 mmol), and toluene (2mL). The resulting mixture was stirred at 130°C for 20h. The mixture was diluted with ethyl acetate and washed by water. A small amount of ethylenediamine was added to dissolve the residual copper salts. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (PE/EA = 2/1- 1/1) to give 6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one **3a** (Scheme S1). We observed only slight effects on the reaction efficiency.



additive (1.0 equiv)	Yeild
-	81%
TEMPO	79%
BHT	77%

Scheme S1 Radical scavenger experiments

KIE studies experimental

The dry sealed tube was charged with **1a** (34.4 mg, 0.2 mmol) and [D_1]-**1a** (34.6 mg, 0.2 mmol), diphenyl disulfide (**2a**, 87.2 mg, 0.4 mmol), $Cu(OAc)_2$ (14.6 mg, 0.08 mmol), 2-biphenylcarboxylic acid (15.8 mg, 0.08 mmol), and toluene (4mL). The resulting mixture was stirred at 130°C for 10min. The mixture was diluted with ethyl acetate and washed by water. A small amount of ethylenediamine was added to dissolve the residual copper salts. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (PE/EA = 2/1- 1/1). The KIE experiment was duplicated, the results were listed below:

Runs	Recovery of **1a** and [D_1]-**1a**	Intergration of multiplet peak at ppm 7.82	KIE
First run	49.8	1.48	1.2
Second run	51.2	1.51	0.9

Table S1 KIE studies experiments

4. References

1. R. Odani, K. Hirano, T. Satoh and M. Miura, *Angew. Chem. Int. Ed.*, **2014**, 53, 10784.
2. S. Terence, PCT Int. Appl., 2003047577 A2.

5. X-ray Single Crystal Structure Analysis of 3a

X-ray crystallographic data of **3a** was block at T = 170 K: $C_{16}H_{12}N_2OS$, Mr = 280.34, triclinic. Space group P 1, a = 7.2154 (2) Å, b = 9.4198 (2) Å, c = 10.4296 (3) Å, α = 76.342 (2)°, β = 79.372 (2)°, γ = 84.642 (1)°, V = 676.09 (3) $Å^3$, Z = 2.

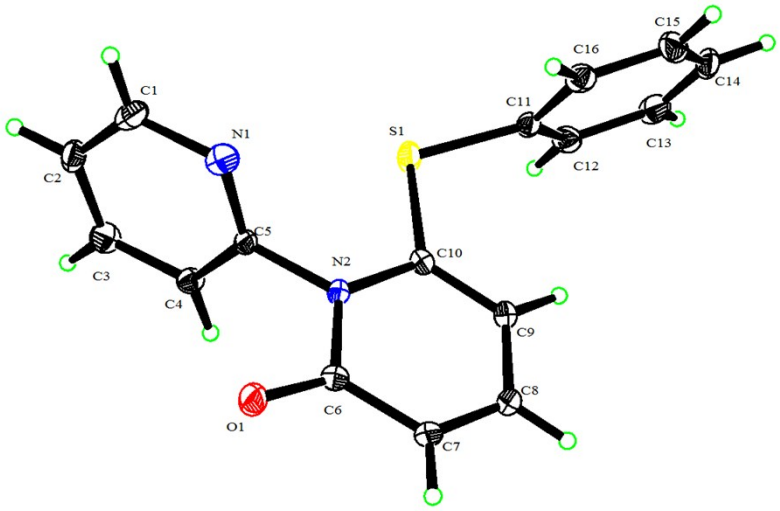
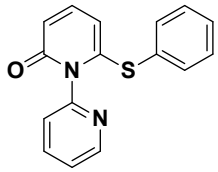


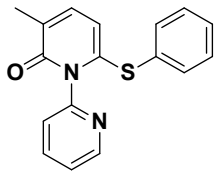
Figure S1. The crystal structure of **3a** by X-ray analysis. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif, the CCDC number is 1439142.

6. Analytical Characterization Data of Products



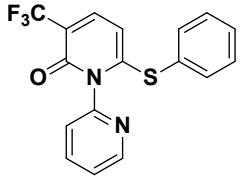
6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3a)

White solid, m.p. 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.84 (td, *J* = 7.8, 1.9 Hz, 1H), 7.43 – 7.28 (m, 7H), 7.16 (dd, *J* = 9.2, 7.3 Hz, 1H), 6.40 (dd, *J* = 9.2, 0.8 Hz, 1H), 5.72 (d, *J* = 7.3 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.33, 149.61, 139.50, 138.39, 134.44, 129.74, 129.68, 124.36, 124.06, 117.05, 106.19; HRMS (ESI) *m/z*: calcd for $C_{16}H_{13}N_2OS^+$ $[M + H]^+$: 281.0749, found: 281.0753.



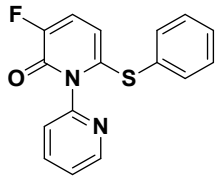
3-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3b)

White solid, m.p. 94-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.78 (td, *J* = 7.8, 1.8 Hz, 1H), 7.39 – 7.20 (m, 7H), 7.10 (d, *J* = 7.3 Hz, 1H), 5.91 (d, *J* = 7.2 Hz, 1H), 2.10 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.80, 149.41, 138.00, 136.74, 133.01, 129.45, 128.84, 123.99, 108.49, 16.71; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{14}N_2NaOS^+$ [M + Na]$^+$: 317.0725, found: 317.0718.



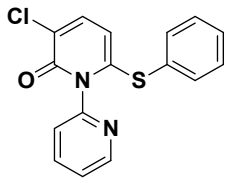
6-(phenylthio)-3-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3c)

White solid, m.p. 129-130 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (dd, *J* = 4.8, 0.9 Hz, 1H), 7.94 (td, *J* = 7.8, 1.8 Hz, 1H), 7.55 – 7.38 (m, 8H), 5.57 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.99, 156.22, 150.05, 149.90, 138.77, 138.35, 135.58, 130.78, 130.20, 127.50, 124.97, 124.10, 102.12; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{11}F_3N_2NaOS^+$ [M + Na]$^+$: 371.0442, found: 371.0438.



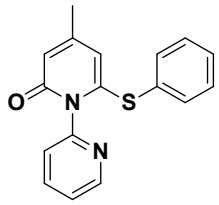
3-fluoro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3d)

Yellow solid, m.p. 109-110 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.82 (td, *J* = 7.7, 1.9 Hz, 1H), 7.43 – 7.21 (m, 7H), 7.02 (dd, *J* = 9.3, 8.0 Hz, 1H), 5.86 (dd, *J* = 8.0, 4.2 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.79, 151.79, 150.38, 149.80, 149.59, 141.38, 138.38, 133.24, 130.53, 129.65, 129.26, 124.57, 123.86, 120.48, 120.34, 106.14; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{11}FN_2NaOS^+$ [M + Na]$^+$: 321.0474, found: 321.0468.



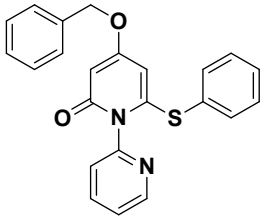
3-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3e)

White solid, m.p. 91-92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (dd, *J* = 4.9, 0.8 Hz, 1H), 7.87 (td, *J* = 7.8, 1.9 Hz, 1H), 7.45 – 7.31 (m, 8H), 5.69 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.42, 150.83, 149.62, 147.27, 138.55, 137.49, 134.42, 129.90, 129.87, 129.33, 124.67, 123.85, 122.63, 105.62; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{11}ClN_2NaOS^+$ [M + Na]$^+$: 337.0178, found: 337.0172.



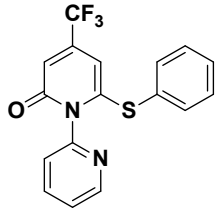
4-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3f)

White solid, m.p. 107-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (m, 1H), 7.81 (td, *J* = 7.8, 1.9 Hz, 1H), 7.4 – 7.27 (m, 7H), 6.27 – 6.22 (m, 1H), 5.65 (d, *J* = 1.5 Hz, 1H), 2.04 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.31, 151.22, 151.15, 149.48, 146.39, 138.18, 134.04, 130.05, 129.65, 129.44, 124.21, 124.16, 116.12, 109.32, 21.46; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ $[M + H]^+$: 295.0905, found: 295.0908.



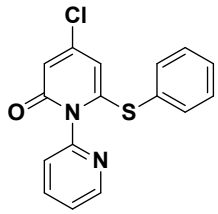
4-(benzyloxy)-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3g)

White solid, m.p. 135-136 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72-8.63 (m, 1H), 7.87 (td, *J* = 7.7, 1.9 Hz, 1H), 7.49 – 7.28 (m, 12H), 5.89 (d, *J* = 2.4 Hz, 1H), 5.50 (d, *J* = 2.4 Hz, 1H), 4.94 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.98, 164.67, 151.00, 149.54, 148.90, 138.27, 134.91, 129.94, 129.84, 128.98, 128.58, 128.41, 127.74, 124.54, 124.26, 100.44, 94.93, 70.16; HRMS (ESI) *m*/*z*: calcd for $C_{23}H_{19}N_2O_2S^+$ $[M + H]^+$: 387.1167, found: 387.1156.



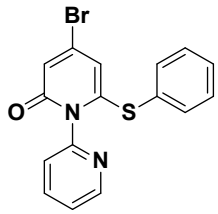
6-(phenylthio)-4-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3h)

White solid, m.p. 118-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 – 8.67 (m, 1H), 7.92 (td, *J* = 7.8, 1.9 Hz, 1H), 7.51 – 7.37 (m, 7H), 6.66 (d, *J* = 0.6 Hz, 1H), 5.70 (d, *J* = 1.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.22, 152.48, 150.33, 149.94, 138.75, 135.06, 130.58, 130.13, 127.91, 124.86, 123.85, 113.70, 113.67, 99.66; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{12}F_3N_2OS^+$ $[M + H]^+$: 349.0622, found: 349.0614.



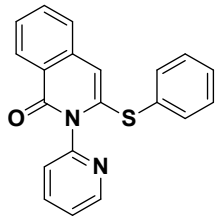
4-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3i)

White solid, m.p. 119-120 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (dd, *J* = 4.8, 1.1 Hz, 1H), 7.90 (td, *J* = 7.8, 1.9 Hz, 1H), 7.51 – 7.36 (m, 7H), 6.44 (d, *J* = 2.0 Hz, 1H), 5.59 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.11, 150.64, 150.39, 149.79, 147.25, 138.61, 135.19, 130.49, 130.10, 128.04, 124.69, 124.12, 114.92, 106.10; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}ClN_2OS^+$ $[M + H]^+$: 315.0359, found: 315.0350.



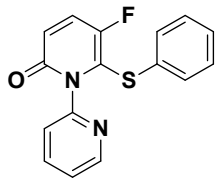
4-bromo-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3j)

Yellow solid, m.p. 118-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (dd, *J* = 4.8, 0.9 Hz, 1H), 7.89 (td, *J* = 7.8, 1.8 Hz, 1H), 7.50 – 7.36 (m, 7H), 6.65 (d, *J* = 1.8 Hz, 1H), 5.73 (d, *J* = 1.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.81, 150.40, 150.27, 149.79, 138.61, 136.25, 135.11, 130.46, 130.09, 128.08, 124.69, 124.06, 118.50, 108.64; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{11}BrN_2NaOS^+$ $[M + Na]^+$: 380.9673, found: 380.9662.



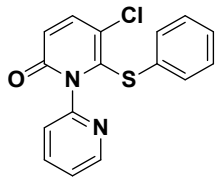
3-(phenylthio)-2-(pyridin-2-yl)isoquinolin-1(2*H*)-one (3k)

White solid, m.p. 97-98 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 – 8.57 (m, 1H), 8.37 (d, *J* = 8.0 Hz, 1H), 7.74 (td, *J* = 7.8, 1.9 Hz, 1H), 7.67 – 7.58 (m, 1H), 7.53 – 7.42 (m, 2H), 7.39 (d, *J* = 7.9 Hz, 1H), 7.34 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.32 – 7.24 (m, 6H), 7.22 – 7.18 (m, 1H), 6.56 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.16, 151.62, 149.28, 137.75, 137.66, 136.48, 132.92, 132.16, 131.94, 129.32, 128.94, 128.34, 128.07, 127.33, 127.03, 125.59, 124.96, 124.50, 123.82, 111.08; HRMS (ESI) *m*/*z*: calcd for $C_{20}H_{15}N_2OS^+$ $[M + H]^+$: 331.0905, found: 331.0898.



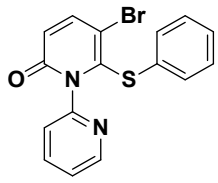
5-fluoro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3l)

White solid, m.p. 110-111 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (ddd, *J* = 4.8, 1.8, 0.8 Hz, 1H), 7.73 (td, *J* = 7.7, 1.9 Hz, 1H), 7.39 (dd, *J* = 10.1, 7.1 Hz, 1H), 7.33 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.24 – 7.16 (m, 3H), 7.10 (ddd, *J* = 6.0, 4.3, 2.4 Hz, 3H), 6.68 (dd, *J* = 10.1, 5.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.45, 151.02, 150.52, 149.41, 148.71, 138.01, 132.56, 131.83, 131.61, 129.80, 129.12, 127.69, 124.03, 123.88, 123.61, 123.56; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}FN_2OS^+$ [M + H]$^+$: 299.0654, found: 299.0660.



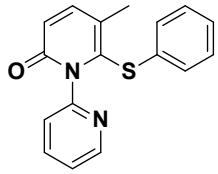
5-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3m)

White solid, m.p. 127-128 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.56 (ddd, *J* = 4.9, 1.8, 0.7 Hz, 1H), 7.70 (td, *J* = 7.7, 1.9 Hz, 1H), 7.47 (d, *J* = 9.8 Hz, 1H), 7.32 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.25 – 7.16 (m, 3H), 7.10 – 7.01 (m, 3H), 6.69 (d, *J* = 9.8 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.29, 152.09, 149.57, 141.46, 139.14, 138.11, 132.99, 129.31, 129.12, 127.51, 124.14, 123.74, 123.61, 121.18; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}ClN_2OS^+$ [M + H]$^+$: 315.0359, found: 315.0350.



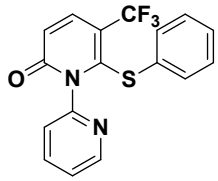
5-bromo-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3n)

Brown solid, m.p. 128-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (ddd, *J* = 4.9, 1.8, 0.8 Hz, 1H), 7.67 (td, *J* = 7.7, 1.9 Hz, 1H), 7.59 (d, *J* = 9.8 Hz, 1H), 7.29 (ddd, *J* = 4.9, 2.5, 1.0 Hz, 1H), 7.23 – 7.13 (m, 3H), 7.06 – 6.95 (m, 3H), 6.63 (d, *J* = 9.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.40, 152.16, 149.35, 143.72, 140.45, 137.86, 132.97, 130.43, 129.15, 128.69, 127.25, 123.95, 123.60, 123.47, 109.99; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}BrN_2OS^+$ [M + H]$^+$: 358.9854, found: 358.9860.



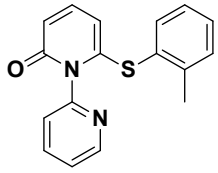
5-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3o)

White solid, m.p. 131-132 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 – 8.49 (m, 1H), 7.65 (td, *J* = 7.7, 1.9 Hz, 1H), 7.36 (d, *J* = 9.4 Hz, 1H), 7.30 – 7.25 (m, 1H), 7.22 – 7.11 (m, 3H), 7.02 (d, *J* = 7.9 Hz, 1H), 6.97 – 6.91 (m, 2H), 6.70 (d, *J* = 9.4 Hz, 1H), 2.21 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.11, 152.50, 149.27, 142.91, 137.73, 136.95, 134.32, 129.07, 127.46, 126.49, 123.62, 123.53, 123.12, 122.86, 18.97; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ [M + H]$^+$: 295.0905, found: 295.0909.



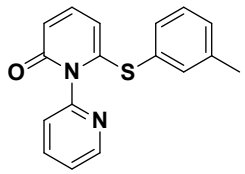
6-(phenylthio)-5-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3p)

White solid, m.p. 94-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 – 8.41 (m, 1H), 7.71 (d, *J* = 9.9 Hz, 1H), 7.50 (td, *J* = 7.8, 1.9 Hz, 1H), 7.27 – 7.10 (m, 4H), 6.86 – 6.77 (m, 2H), 6.72 (dd, *J* = 18.4, 8.9 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.77, 150.59, 149.11, 143.62, 137.30, 136.05, 136.01, 132.20, 129.25, 129.13, 127.69, 124.06, 123.79, 122.22; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{12}F_3N_2OS^+$ [M + H]$^+$: 349.0622, found: 349.0613.



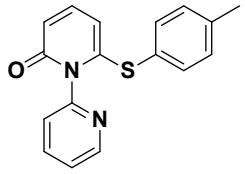
6-(*o*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4a)

White solid, m.p. 152-153 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (dd, *J* = 4.7, 0.9 Hz, 1H), 7.89 (td, *J* = 7.8, 1.8 Hz, 1H), 7.50 – 7.37 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.27 (d, *J* = 7.0 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.14 (dd, *J* = 9.2, 7.3 Hz, 1H), 6.39 (dd, *J* = 9.2, 0.6 Hz, 1H), 5.52 (dd, *J* = 7.3, 0.6 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.89, 139.74, 138.64, 136.23, 131.31, 130.57, 127.40, 124.57, 124.01, 116.69, 104.89, 20.52; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ [M + H]$^+$: 295.0905, found: 295.0903.



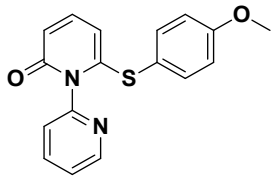
6-(*m*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4b)

White solid, m.p. 81-82 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (ddd, *J* = 4.9, 1.9, 0.8 Hz, 1H), 7.86 (qd, *J* = 7.6, 1.9 Hz, 1H), 7.40 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.38 – 7.34 (m, 1H), 7.28 – 7.16 (m, 5H), 6.41 (dd, *J* = 9.2, 1.1 Hz, 1H), 5.72 (dd, *J* = 7.3, 1.1 Hz, 1H), 2.31 (d, *J* = 0.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.45, 153.29, 151.70, 150.84, 141.82, 141.60, 140.44, 137.15, 133.69, 132.64, 131.61, 131.28, 126.40, 126.15, 118.88, 108.01, 23.14; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ [M + H]$^+$: 295.0905, found: 295.0908.



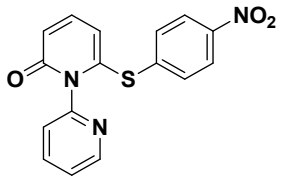
6-(*p*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4c)

White solid, m.p. 115-116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H), 7.89 (td, *J* = 7.7, 1.9 Hz, 1H), 7.45 – 7.37 (m, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.21 – 7.13 (m, 3H), 6.38 (dd, *J* = 9.2, 1.0 Hz, 1H), 5.63 (dd, *J* = 7.3, 1.0 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.53, 151.38, 149.83, 149.73, 140.53, 139.68, 138.60, 135.11, 130.76, 125.83, 124.55, 124.27, 116.51, 105.29, 21.34; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ [M + H]$^+$: 295.0905, found: 295.0909.



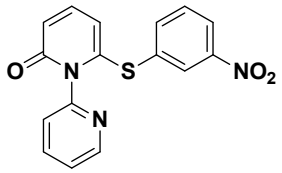
6-((4-methoxyphenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4d)

White solid, m.p. 113-114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 – 8.64 (m, 1H), 7.90 (td, *J* = 7.7, 1.9 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 2H), 7.36 (d, *J* = 8.9 Hz, 2H), 7.14 (dd, *J* = 9.2, 7.4 Hz, 1H), 6.90 (d, *J* = 8.9 Hz, 2H), 6.35 (dd, *J* = 9.1, 1.0 Hz, 1H), 5.54 (dd, *J* = 7.3, 1.0 Hz, 1H), 3.80 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.50, 161.29, 151.33, 150.66, 149.85, 139.69, 138.65, 137.22, 124.58, 124.30, 119.34, 116.03, 115.59, 104.42, 55.47; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2O_2S^+$ [M + H]$^+$: 311.0854, found: 311.0861.



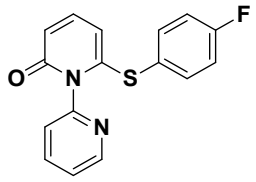
6-((4-nitrophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4e)

White solid, m.p. 151-152 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (ddd, *J* = 4.8, 1.8, 0.8 Hz, 1H), 8.14 – 8.05 (m, 2H), 7.78 (td, *J* = 7.7, 1.9 Hz, 1H), 7.33 (m, 4H), 7.24 (t, *J* = 6.9 Hz, 1H), 6.64 (dd, *J* = 9.3, 1.1 Hz, 1H), 6.34 (dd, *J* = 7.0, 1.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.35, 151.34, 149.57, 147.10, 141.97, 141.35, 139.46, 138.39, 130.58, 124.48, 124.34, 123.82, 121.80, 113.00; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}N_3O_3S^+$ [M + H]$^+$: 326.0599, found: 326.0591.



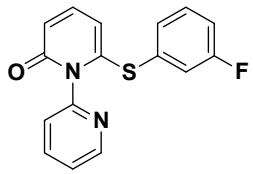
6-((3-nitrophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4f)

Yellow solid, m.p. 102-103 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (ddd, *J* = 4.9, 1.9, 0.8 Hz, 1H), 8.17 (ddd, *J* = 8.2, 2.2, 1.1 Hz, 1H), 8.12 – 8.07 (m, 1H), 7.87 – 7.79 (m, 1H), 7.63 (ddd, *J* = 7.8, 1.7, 1.1 Hz, 1H), 7.55 – 7.48 (m, 1H), 7.38 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.33 – 7.23 (m, 2H), 6.58 (dd, *J* = 9.3, 1.1 Hz, 1H), 6.09 (dd, *J* = 7.1, 1.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.30, 151.24, 149.60, 148.66, 143.84, 139.46, 138.45, 138.36, 134.17, 130.48, 127.18, 124.54, 124.07, 123.75, 120.33, 110.31; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}N_3O_3S^+$ [M + H]$^+$: 326.0599, found: 326.0597.



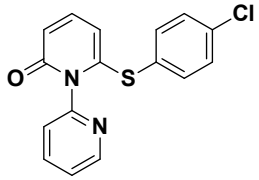
6-((4-fluorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4g)

White solid, m.p. 137-138 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 4.8, 1.8, 0.7 Hz, 1H), 7.88 (td, *J* = 7.8, 1.9 Hz, 1H), 7.48 – 7.31 (m, 4H), 7.16 (dd, *J* = 9.2, 7.3 Hz, 1H), 7.12 – 6.99 (m, 2H), 6.40 (dd, *J* = 9.2, 1.0 Hz, 1H), 5.64 (dd, *J* = 7.3, 1.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.75, 163.39, 162.74, 151.26, 149.79, 148.66, 139.60, 138.60, 137.12, 137.05, 124.94, 124.92, 124.59, 124.25, 117.32, 117.21, 117.15, 105.79; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}FN_2OS^+$ [M + H]$^+$: 299.0654, found: 299.0660.



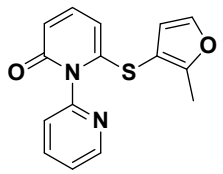
6-((3-fluorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4h)

Brown solid, m.p. 68-69 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (ddd, *J* = 4.9, 1.8, 0.8 Hz, 1H), 7.82 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.38 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.34 – 7.26 (m, 2H), 7.22 (dd, *J* = 9.3, 7.2 Hz, 1H), 7.15 – 7.08 (m, 1H), 7.09 – 7.00 (m, 2H), 6.48 (dd, *J* = 9.3, 1.1 Hz, 1H), 5.91 (dd, *J* = 7.2, 1.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.81, 163.42, 161.81, 151.29, 149.69, 146.23, 139.59, 138.45, 132.65, 132.59, 131.12, 131.05, 129.32, 129.29, 124.49, 124.13, 120.48, 120.30, 118.65, 116.72, 116.55, 108.22; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}FN_2OS^+$ [M + H]$^+$: 299.0654, found: 299.0650.



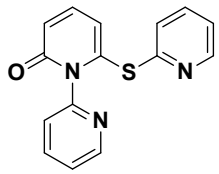
6-((4-chlorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4i)

Yellow solid, m.p. 120-121 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (ddd, *J* = 4.9, 1.8, 0.7 Hz, 1H), 7.86 (td, *J* = 7.8, 1.9 Hz, 1H), 7.40 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.37 – 7.33 (m, 1H), 7.33 – 7.27 (m, 4H), 7.19 (dd, *J* = 9.2, 7.3 Hz, 1H), 6.44 (dd, *J* = 9.2, 1.0 Hz, 1H), 5.76 (dd, *J* = 7.2, 1.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.38, 151.27, 149.75, 147.51, 139.58, 138.56, 136.20, 135.60, 130.12, 128.61, 124.56, 124.19, 117.87, 106.87; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{12}ClN_2OS^+$ [M + H]$^+$: 315.0359, found: 315.0355.



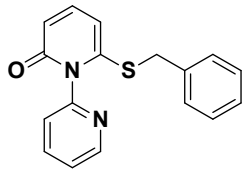
6-((2-methylfuran-3-yl)thio)-2*H*-[1,2'-bipyridin]-2-one (4j)

Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (ddd, *J* = 4.8, 1.8, 0.8 Hz, 1H), 7.95 (td, *J* = 7.7, 1.9 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.38 (d, *J* = 2.0 Hz, 1H), 7.22 (dd, *J* = 9.2, 7.4 Hz, 1H), 6.40 (dd, *J* = 9.2, 1.0 Hz, 1H), 6.33 (d, *J* = 2.0 Hz, 1H), 5.71 (dd, *J* = 7.3, 1.0 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.00, 159.80, 152.63, 151.35, 150.17, 143.34, 141.15, 140.15, 126.09, 125.69, 117.60, 116.26, 106.53, 104.70, 13.25; HRMS (ESI) *m*/*z*: calcd for $C_{15}H_{13}N_3OS^+$ [M + H]$^+$: 285.0695, found: 285.0691.



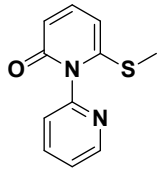
6-(pyridin-2-ylthio)-2*H*-[1,2'-bipyridin]-2-one (4k)

Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (dd, *J* = 4.8, 0.9 Hz, 1H), 8.39 (dd, *J* = 4.8, 0.9 Hz, 1H), 7.67 (td, *J* = 7.8, 1.8 Hz, 1H), 7.51 (td, *J* = 7.8, 1.8 Hz, 1H), 7.37 (dd, *J* = 9.3, 7.0 Hz, 1H), 7.32 – 7.24 (m, 1H), 7.17 (d, *J* = 7.9 Hz, 1H), 7.13 – 7.04 (m, 2H), 6.67 (dd, *J* = 9.3, 0.9 Hz, 1H), 6.55 (dd, *J* = 6.9, 0.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.74, 156.43, 151.81, 150.08, 149.37, 140.30, 139.58, 137.98, 137.21, 124.14, 124.04, 123.83, 122.05, 121.77, 114.55; HRMS (ESI) *m*/*z*: calcd for $C_{12}H_{12}N_2OS^+$ [M + H]$^+$: 282.0710, found: 282.0706.



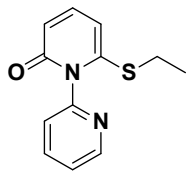
6-(benzylthio)-2*H*-[1,2'-bipyridin]-2-one (4l)

Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 4.9, 1.8, 0.8 Hz, 1H), 7.86 (td, *J* = 7.7, 1.9 Hz, 1H), 7.39 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.35 – 7.18 (m, 7H), 6.48 (dd, *J* = 9.3, 1.0 Hz, 1H), 6.16 (dd, *J* = 7.2, 1.0 Hz, 1H), 4.02 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.53, 151.48, 149.80, 146.95, 139.58, 138.62, 134.57, 129.05, 128.76, 127.93, 124.49, 124.18, 117.90, 105.99, 39.22; HRMS (ESI) *m*/*z*: calcd for $C_{17}H_{15}N_2OS^+$ [M + H]$^+$: 295.0905, found: 295.0913.



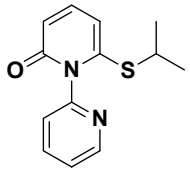
6-(methylthio)-2*H*-[1,2'-bipyridin]-2-one (4m)

White solid, m.p. 109-110 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 4.9, 1.8, 0.8 Hz, 1H), 7.89 (td, *J* = 7.8, 1.9 Hz, 1H), 7.45 – 7.37 (m, 1H), 7.34 (dt, *J* = 9.2, 6.8 Hz, 2H), 6.41 (dd, *J* = 9.2, 1.0 Hz, 1H), 6.00 (dd, *J* = 7.3, 0.7 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.47, 151.31, 149.94, 149.73, 139.70, 138.70, 124.60, 124.26, 116.23, 102.13, 16.40; HRMS (ESI) *m*/*z*: calcd for $C_{11}H_{11}N_2OS^+$ [M + H]$^+$: 219.0592, found: 219.0597.



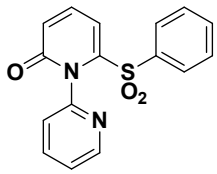
6-(ethylthio)-2*H*-[1,2'-bipyridin]-2-one (4n)

White solid, m.p. 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 4.9, 1.8, 0.8 Hz, 1H), 7.88 (td, *J* = 7.8, 1.9 Hz, 1H), 7.41 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 7.37 – 7.29 (m, 2H), 6.43 (dd, *J* = 9.2, 1.0 Hz, 1H), 6.09 (dd, *J* = 7.3, 0.9 Hz, 1H), 2.83 (q, *J* = 7.4 Hz, 2H), 1.25 (t, *J* = 7.4 Hz, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.65, 151.48, 149.87, 148.17, 139.66, 138.63, 124.50, 124.23, 116.71, 103.79, 27.95, 12.99; HRMS (ESI) *m*/*z*: calcd for $C_{12}H_{13}N_2OS^+$ $[M + H]^+$: 233.0749, found: 233.0747.



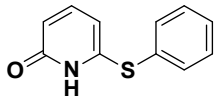
6-(isopropylthio)-2*H*-[1,2'-bipyridin]-2-one (4o)

Yellow solid, m.p. 94-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.92 – 7.81 (m, 1H), 7.45 – 7.37 (m, 1H), 7.37 – 7.27 (m, 2H), 6.47 (dd, *J* = 9.2, 0.9 Hz, 1H), 6.23 (d, *J* = 7.2 Hz, 1H), 3.30 (dq, *J* = 13.1, 6.6 Hz, 1H), 1.24 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.78, 151.80, 149.72, 146.69, 139.55, 138.50, 124.31, 124.18, 117.81, 106.94, 38.97, 22.57; HRMS (ESI) *m*/*z*: calcd for $C_{13}H_{15}N_2OS^+$ $[M + H]^+$: 247.0905, found: 247.0909.



6-(phenylsulfonyl)-2*H*-[1,2'-bipyridin]-2-one (5a)

White solid, m.p. 141-142 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11 (d, *J* = 4.4 Hz, 1H), 7.81 (td, *J* = 7.7, 1.6 Hz, 1H), 7.60-7.50 (m, 2H), 7.44-7.32 (m, 6H), 7.30 (dd, *J* = 7.4, 4.9 Hz, 1H), 6.83 (d, *J* = 9.3 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 162.23, 149.19, 148.88, 145.24, 138.91, 138.14, 133.98, 129.15, 127.82, 127.27, 125.90, 124.60, 111.14; HRMS (ESI) *m*/*z*: calcd for $C_{16}H_{13}N_2O_3S^+$ $[M + H]^+$: 313.0647, found: 313.0649.

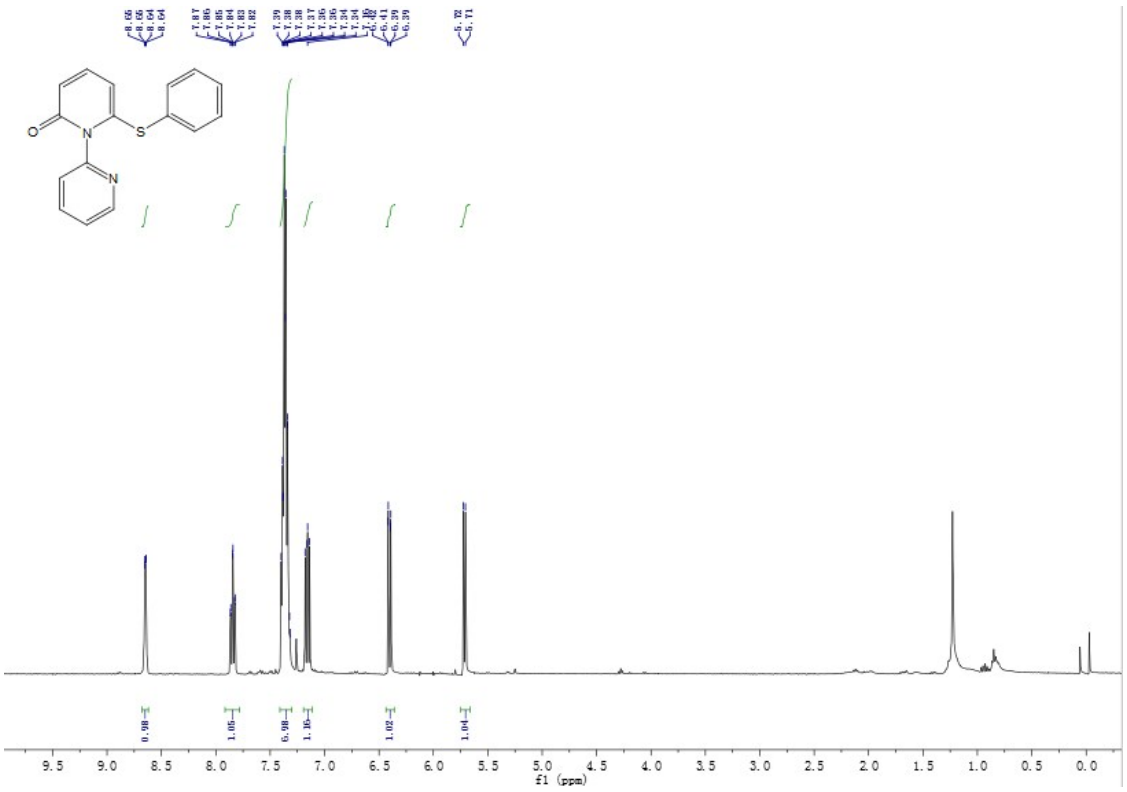


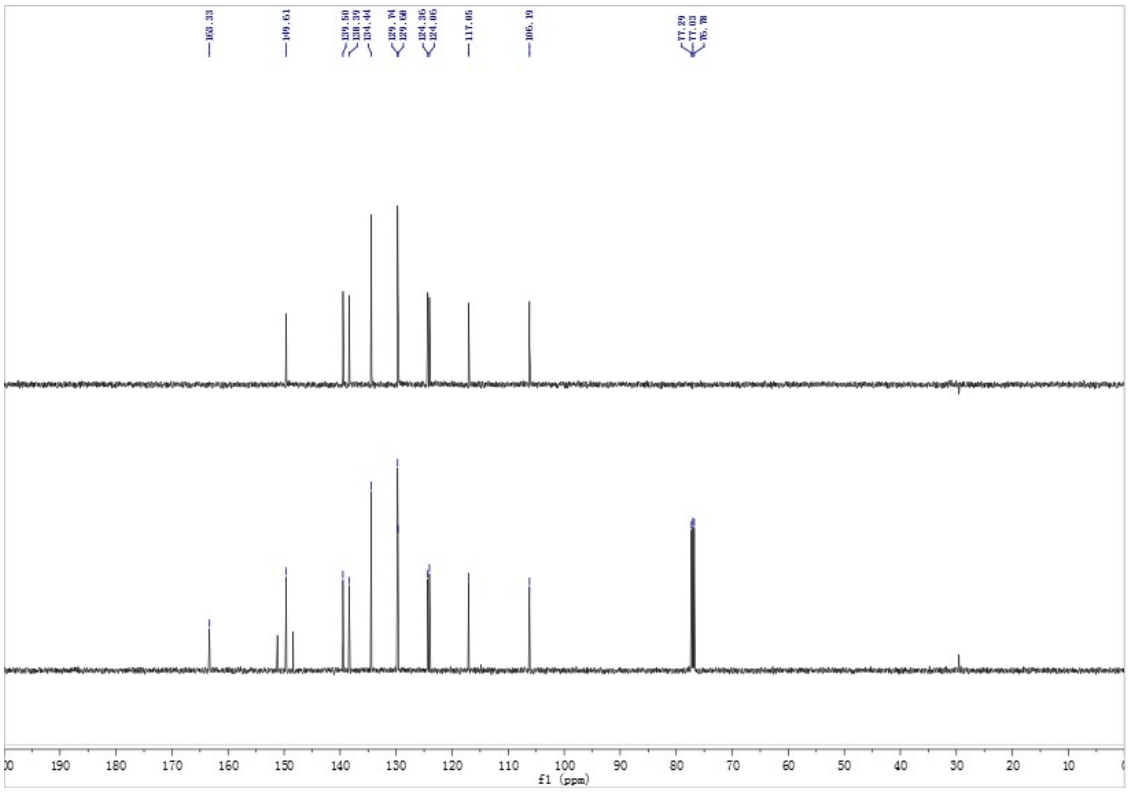
6-(phenylthio)pyridin-2(1*H*)-one (6a)

White solid, m.p. 106-107 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.52 (m, 2H), 7.49 – 7.36 (m, 3H), 7.34 – 7.21 (m, 1H), 6.41 (d, *J* = 9.0 Hz, 1H), 5.97 (d, *J* = 7.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.78, 141.03, 134.32, 129.87, 129.65, 129.21, 116.77, 107.20; HRMS (ESI) *m*/*z*: calcd for $C_{11}H_{10}NOS^+$ $[M + H]^+$: 204.0483, found: 204.0473.

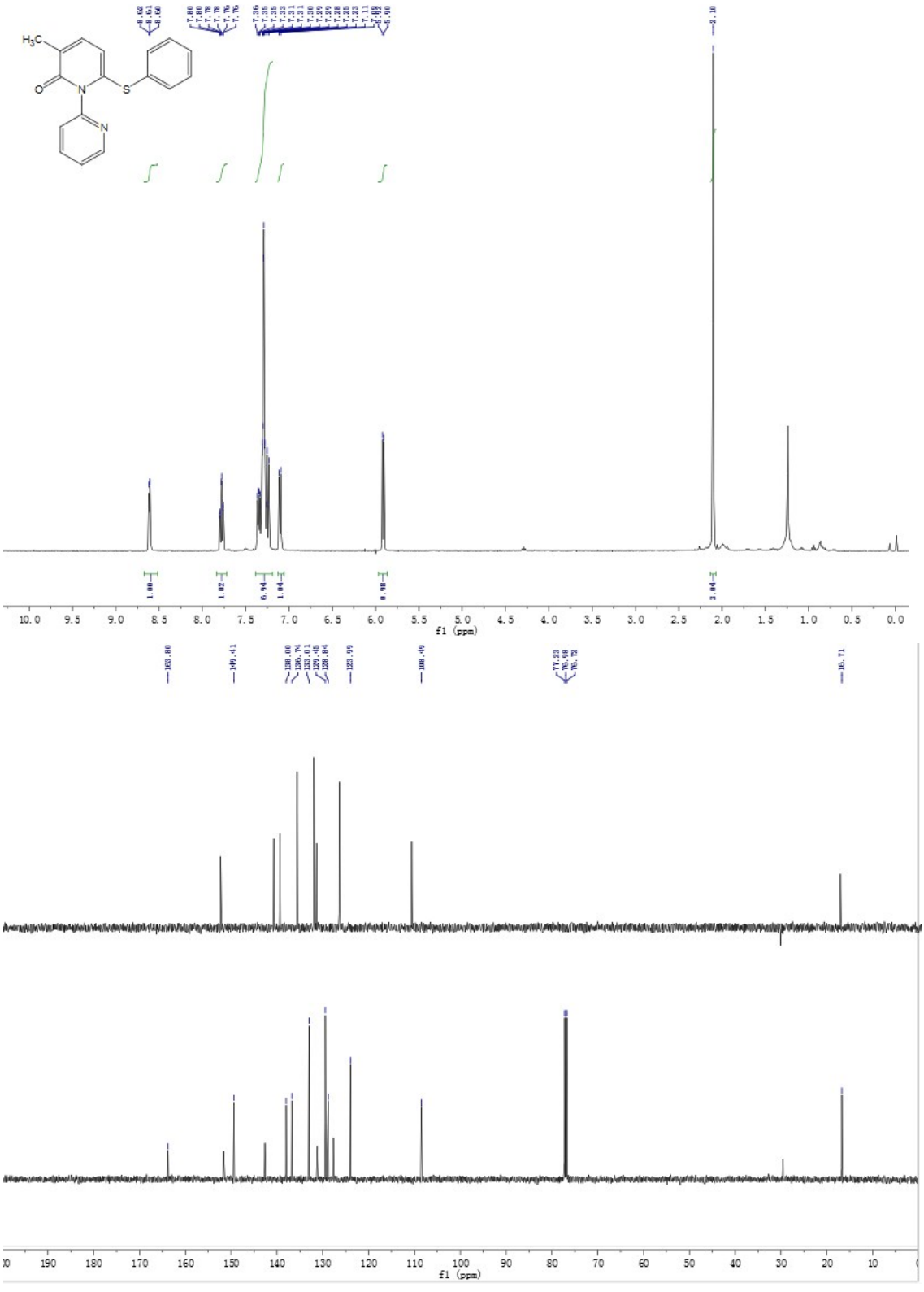
7. Copies of ^{1}H NMR and ^{13}C NMR Spectra for the Products

6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3a)

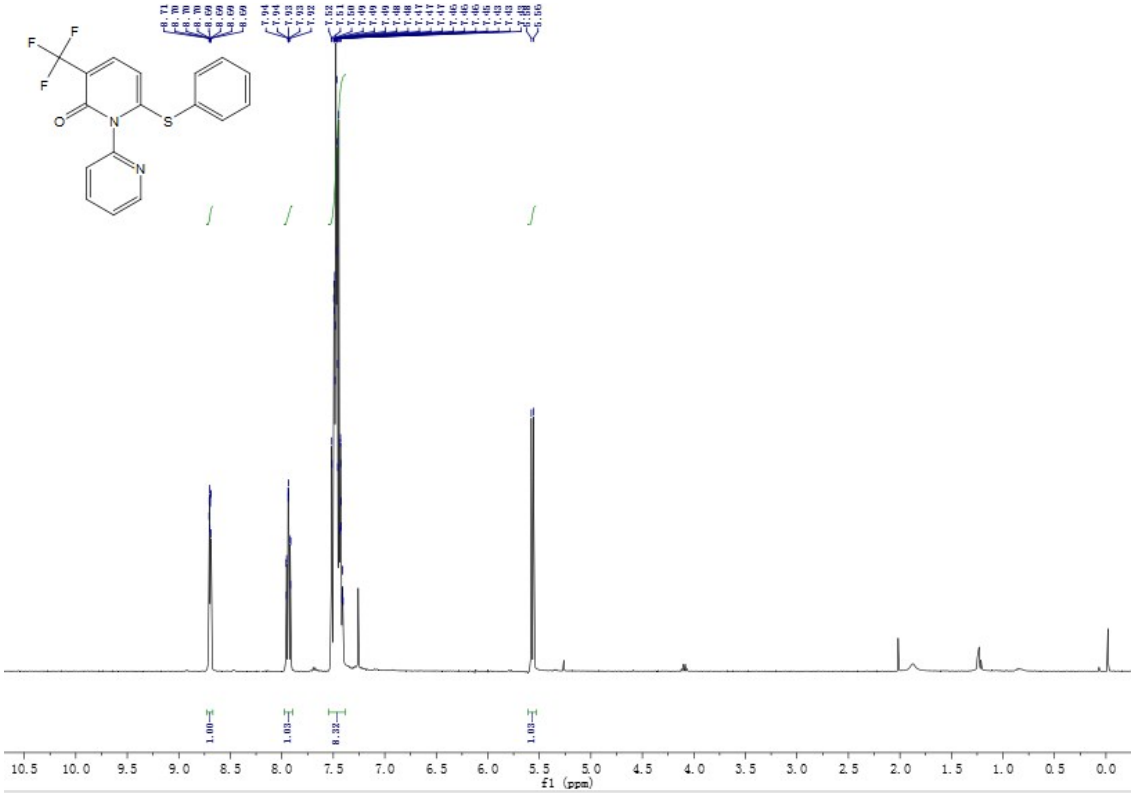


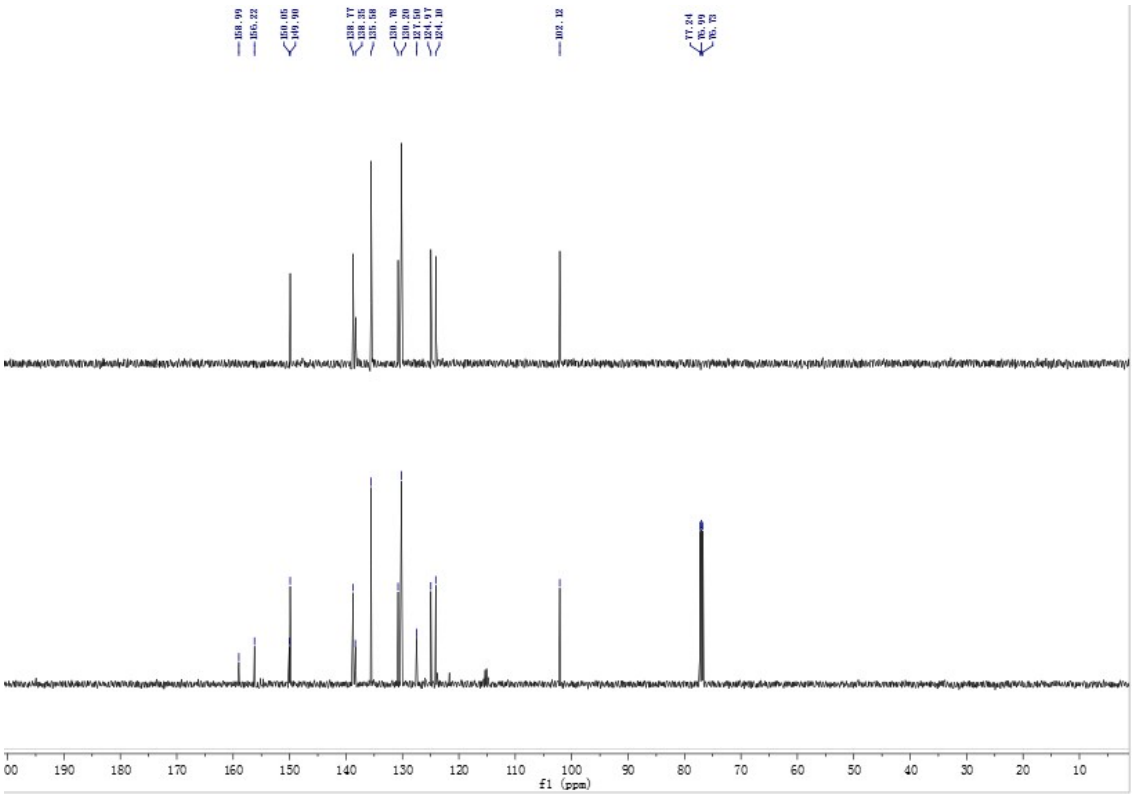


3-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3b)

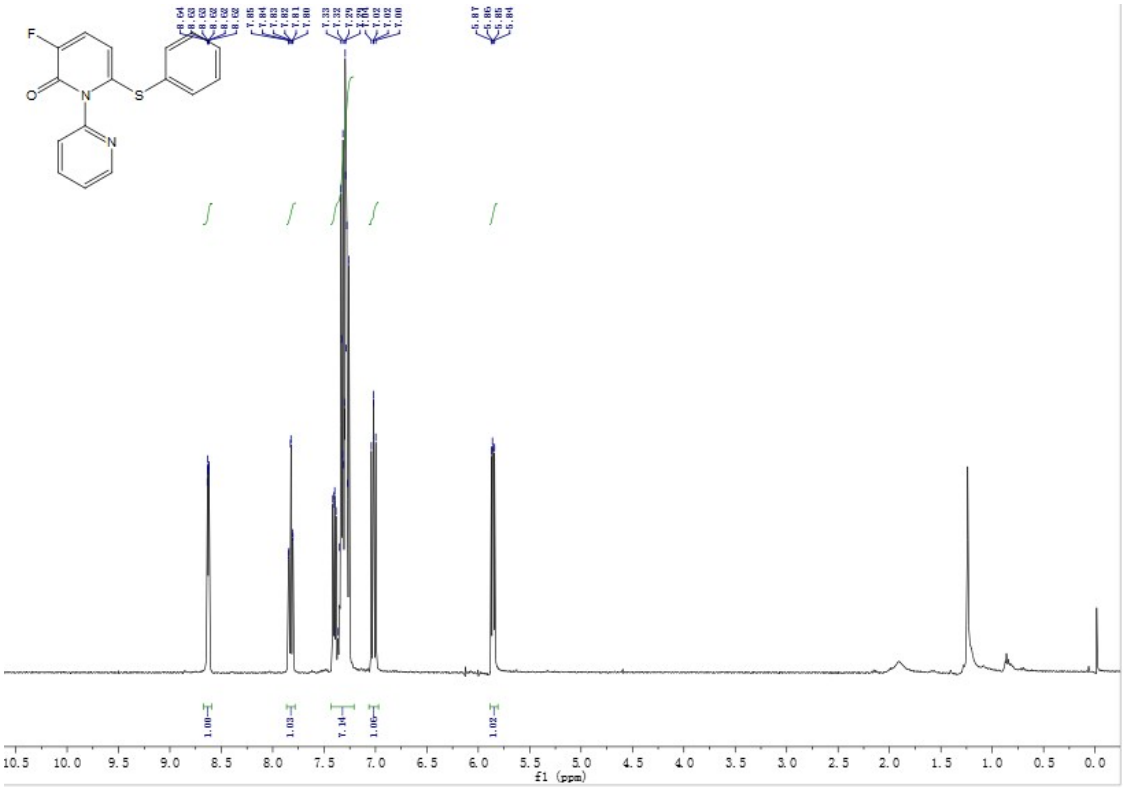


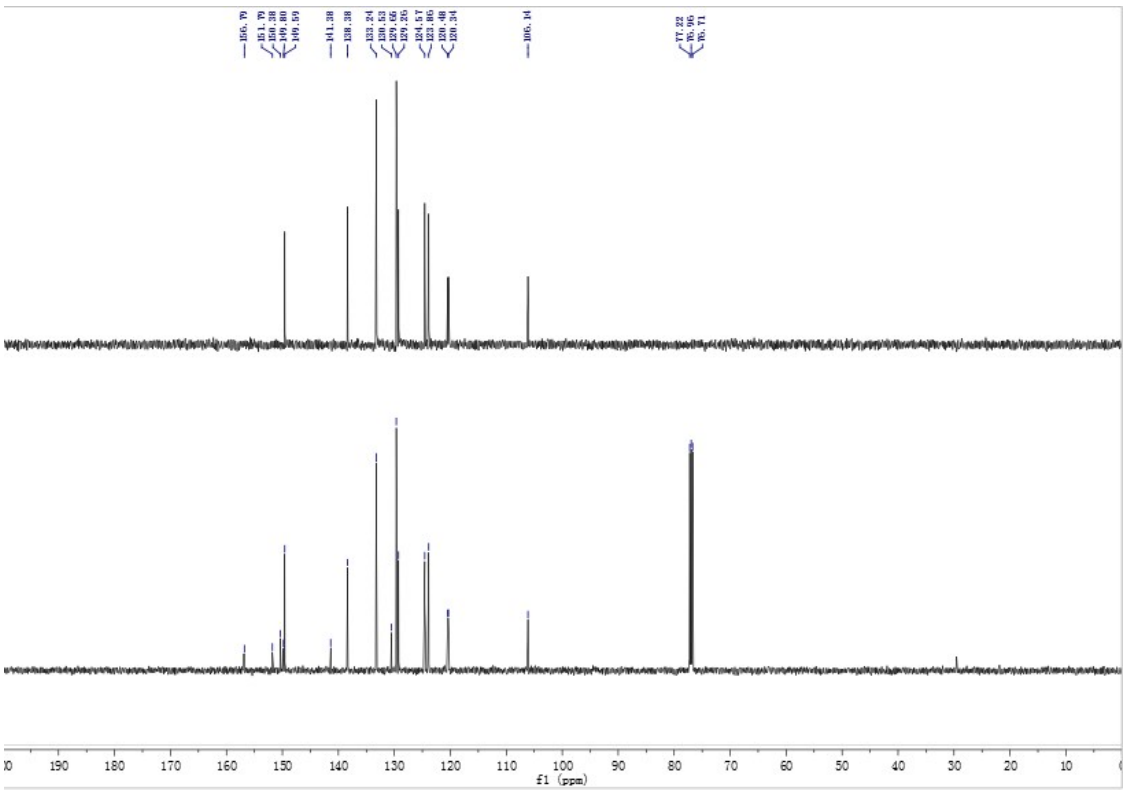
6-(phenylthio)-3-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3c)



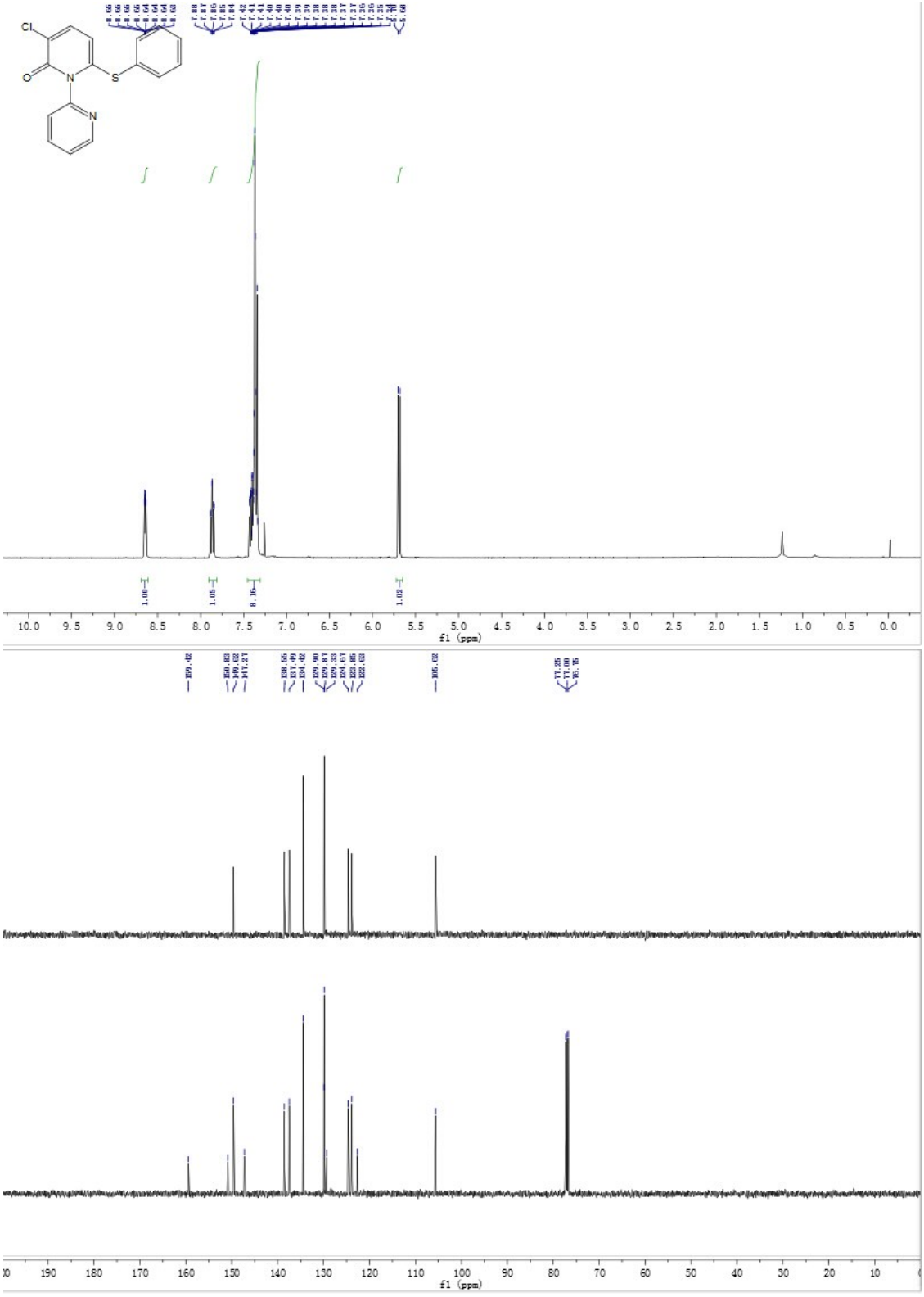


3-fluoro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3d)

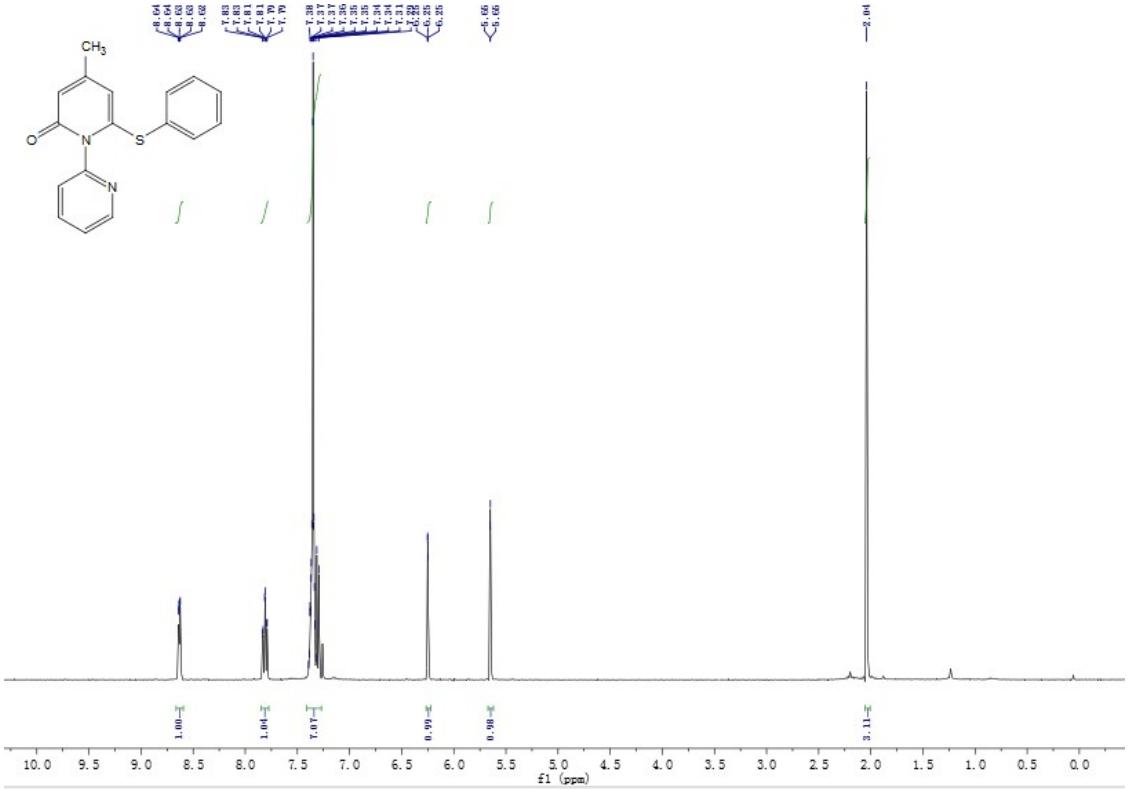


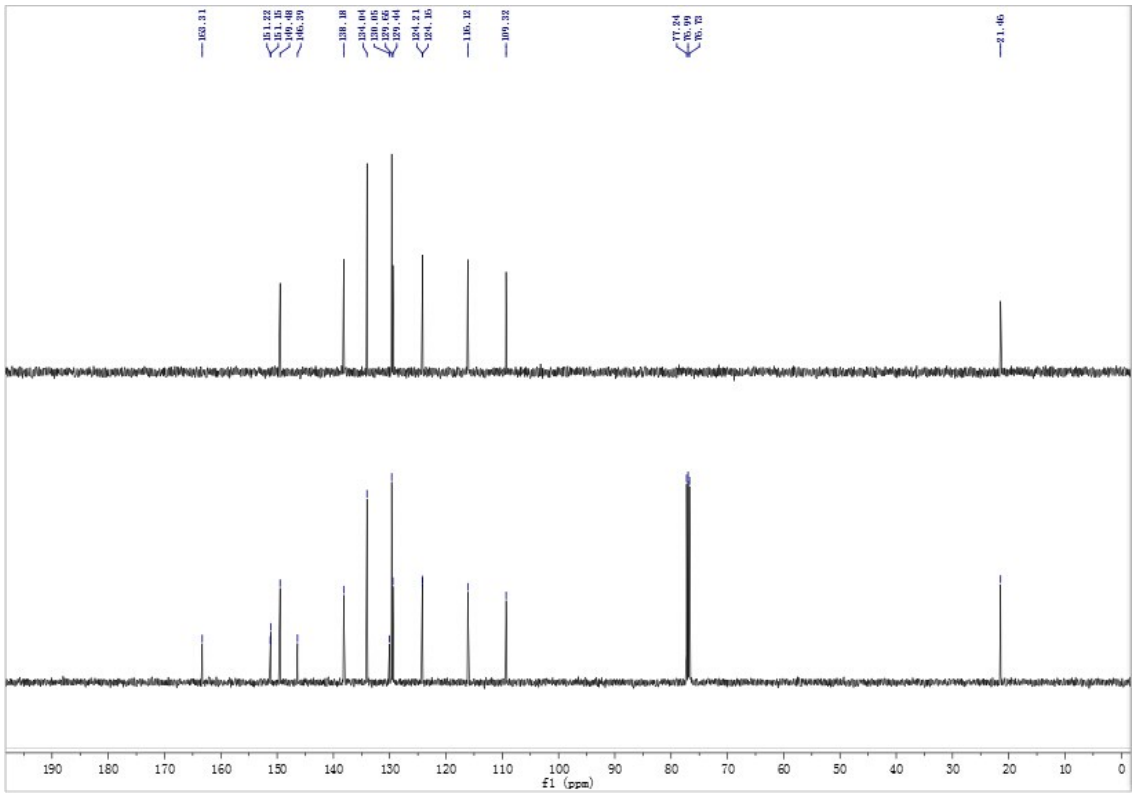


3-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3e)

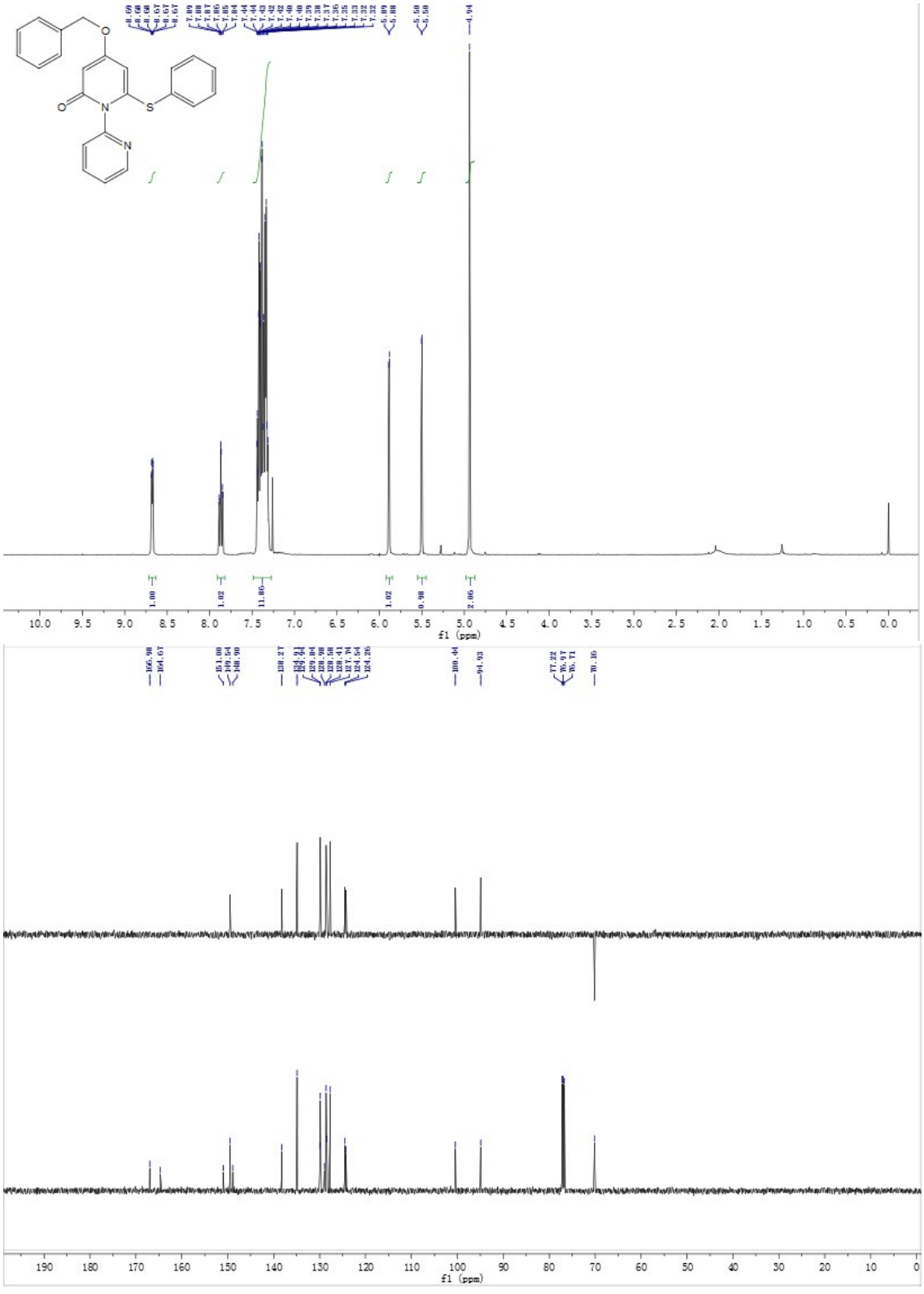


4-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3f)

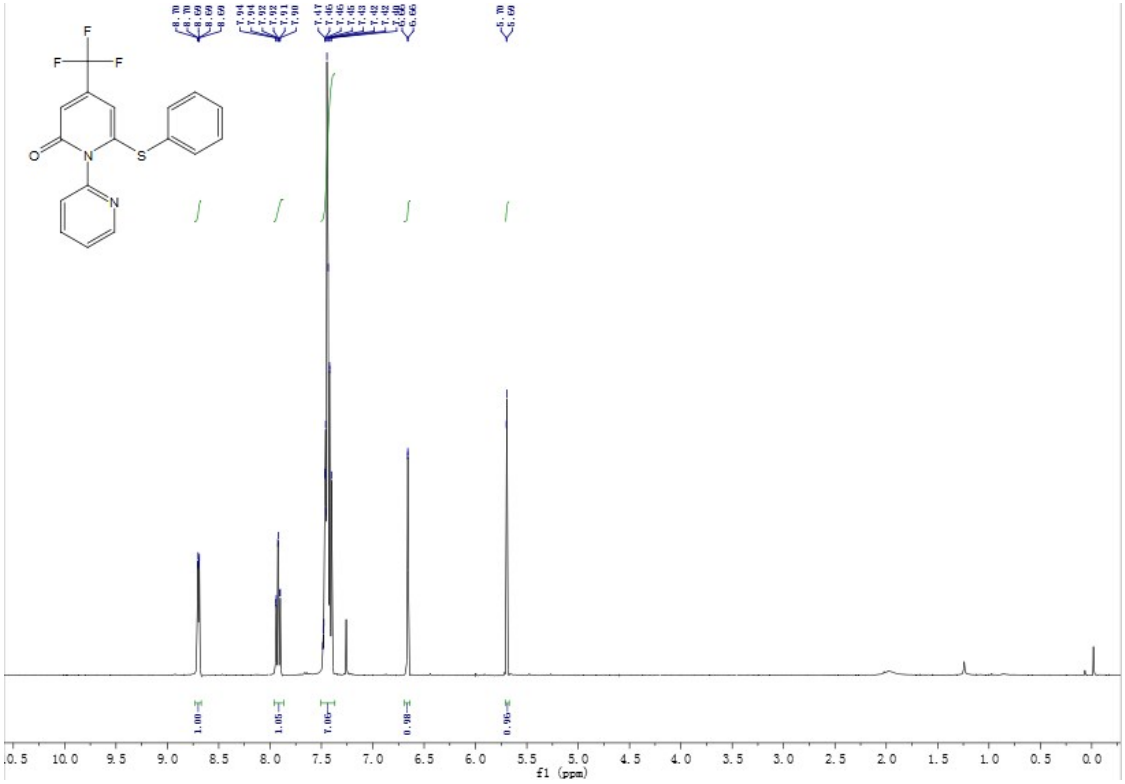


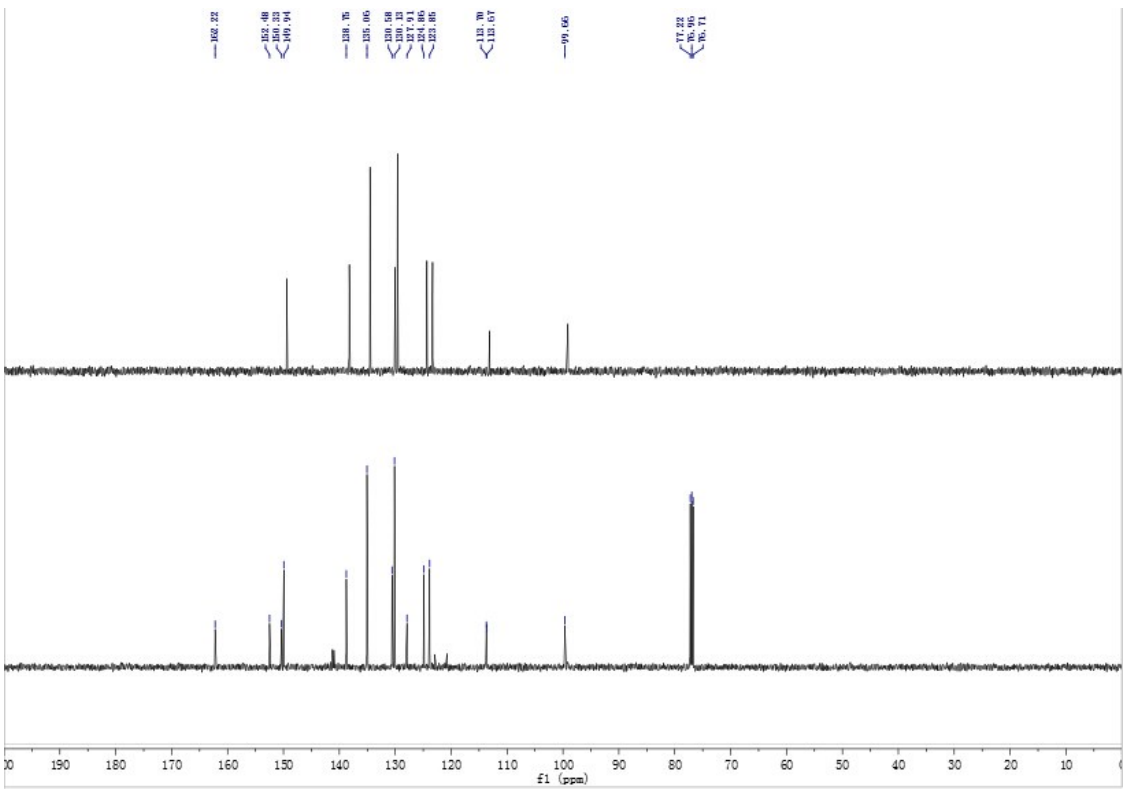


4-(benzyloxy)-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3g)

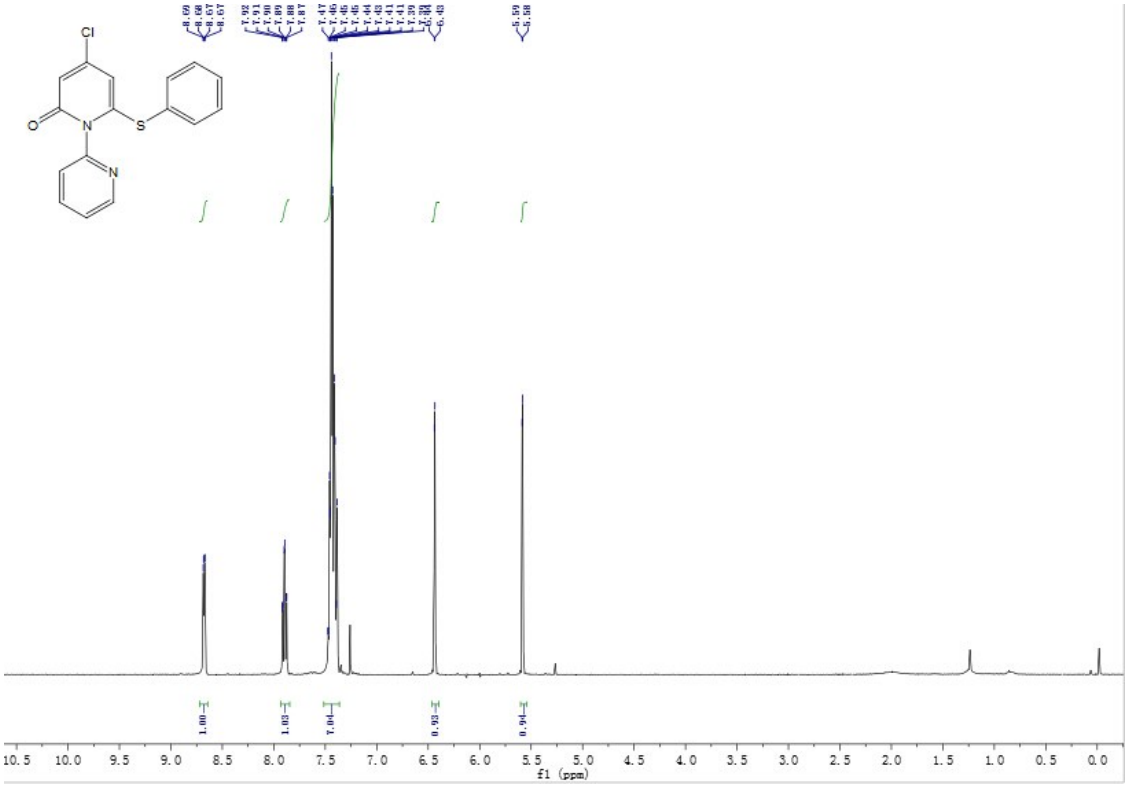


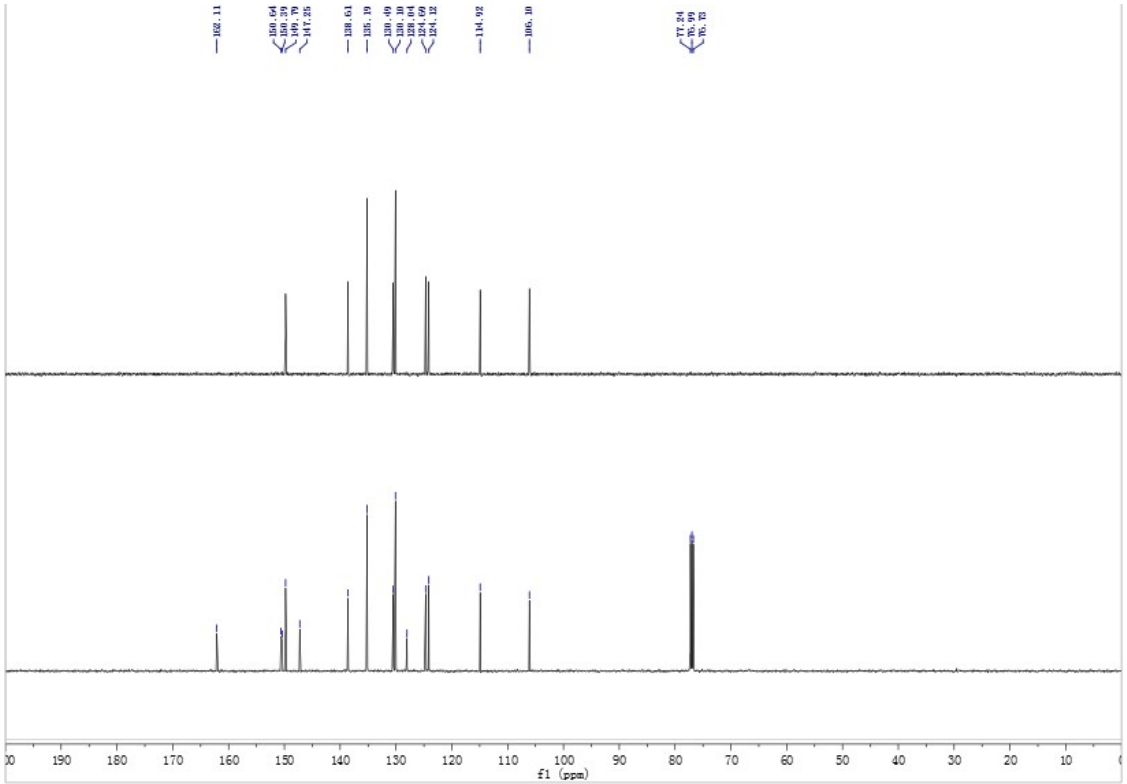
6-(phenylthio)-4-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3h)



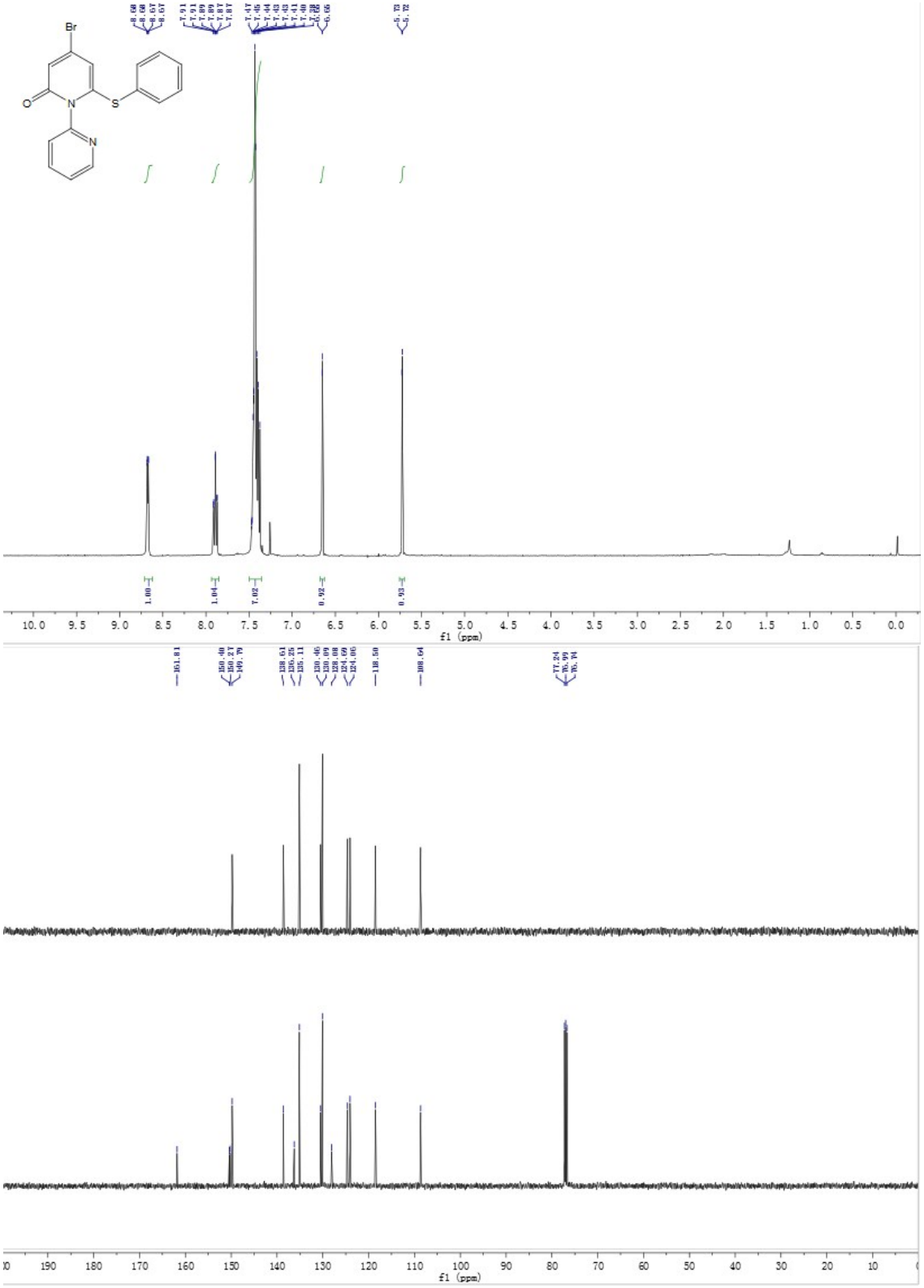


4-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3i)

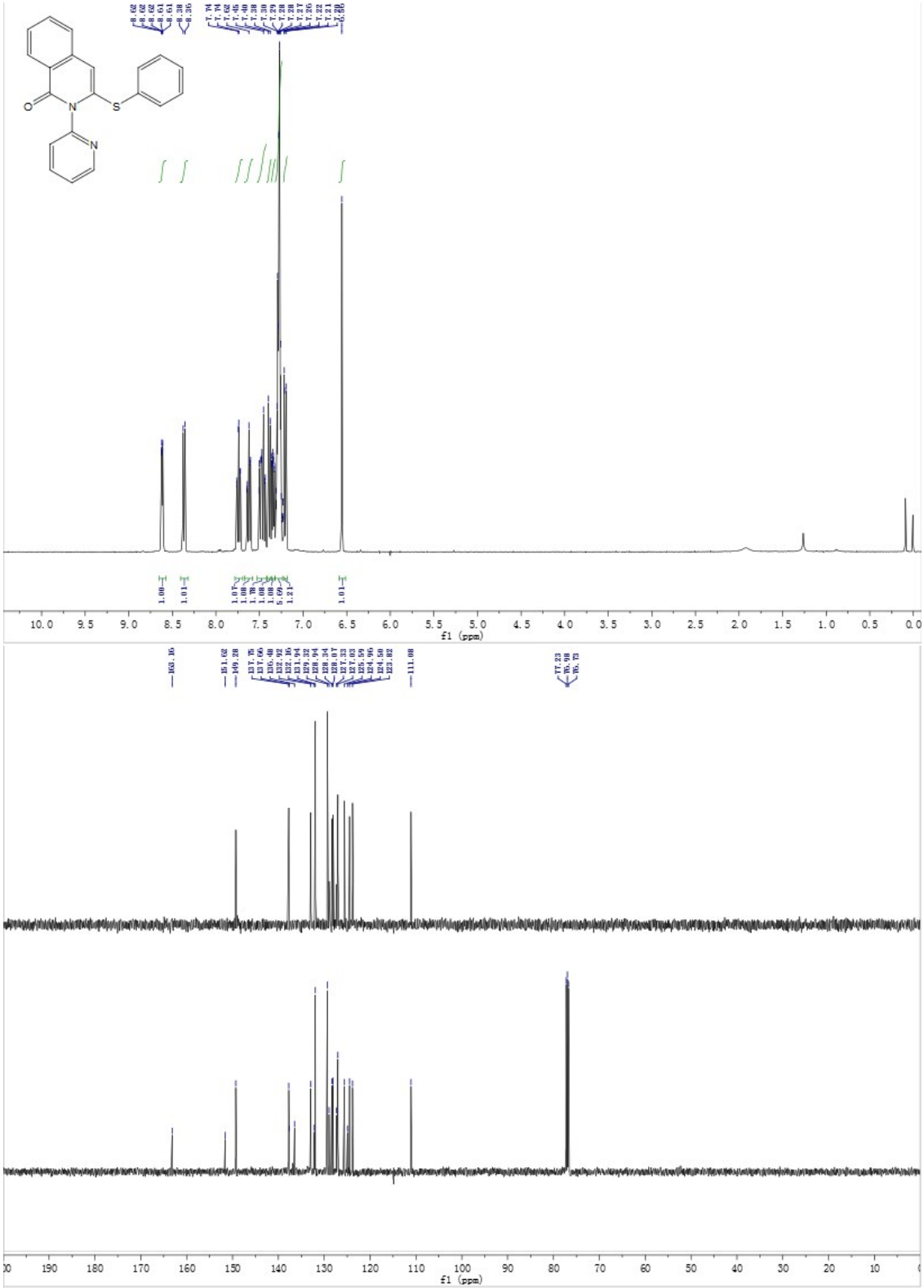




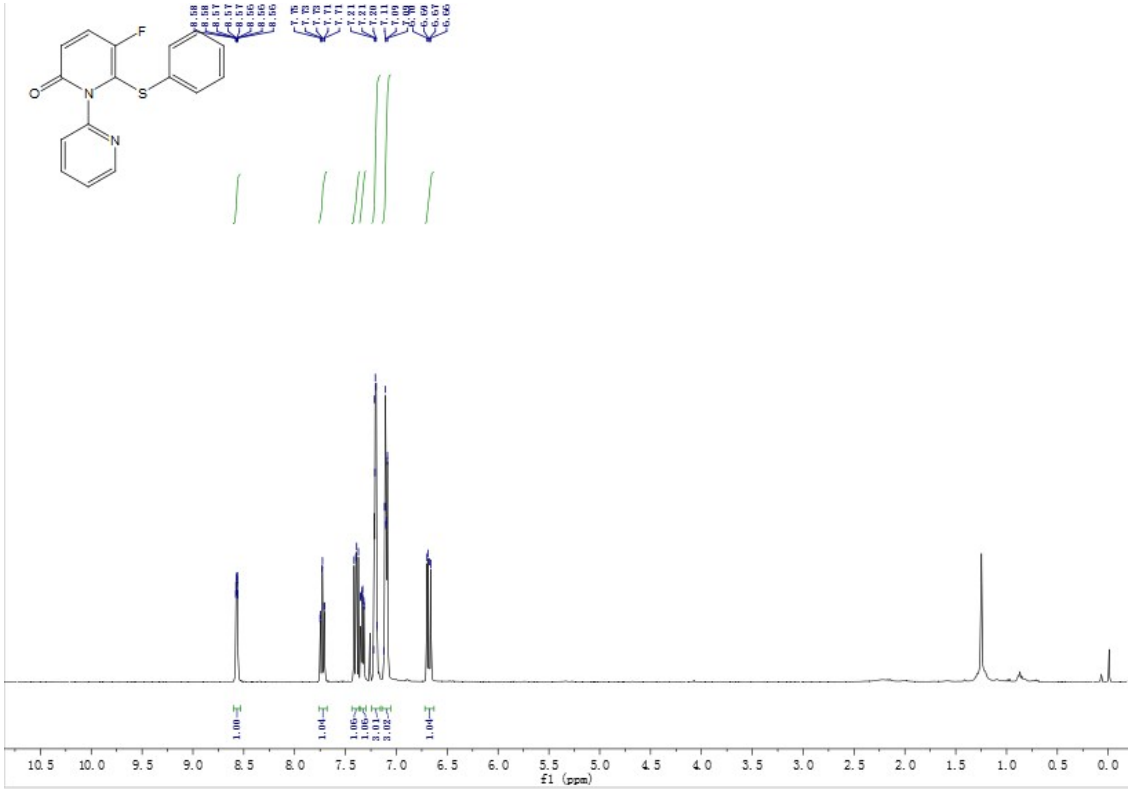
4-bromo-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3j)

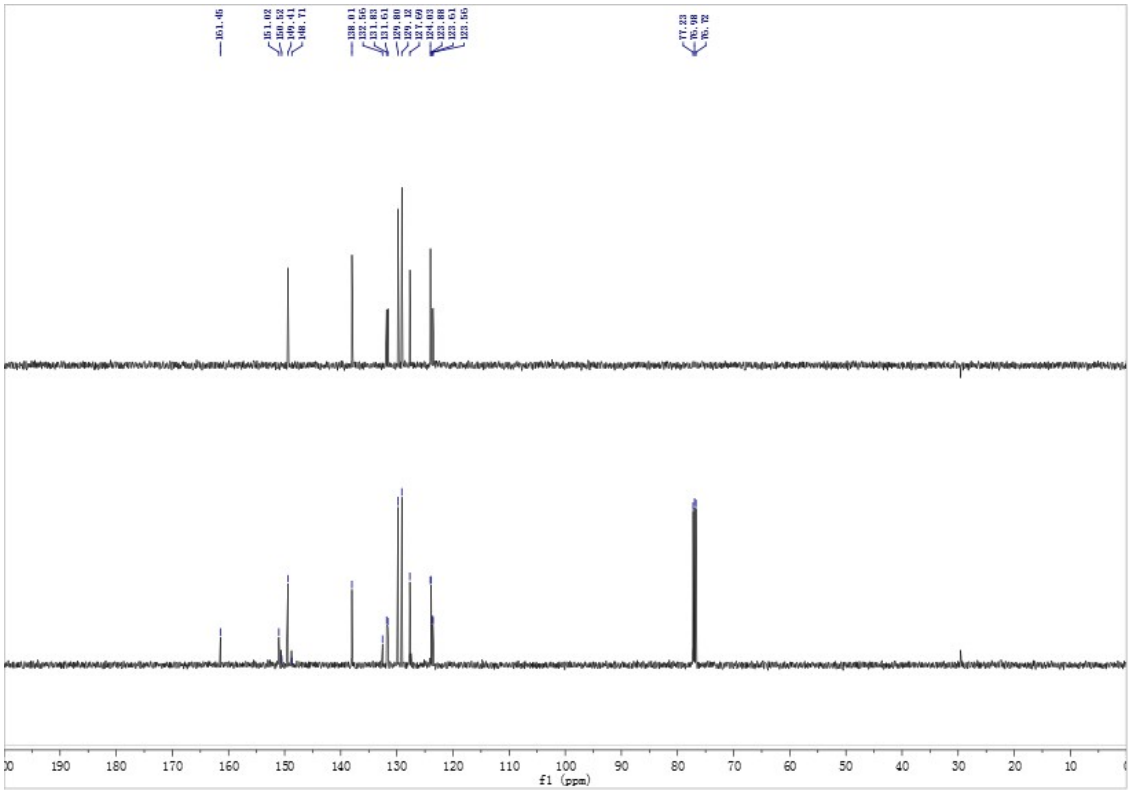


3-(phenylthio)-2-(pyridin-2-yl)isoquinolin-1(2*H*)-one (3k)

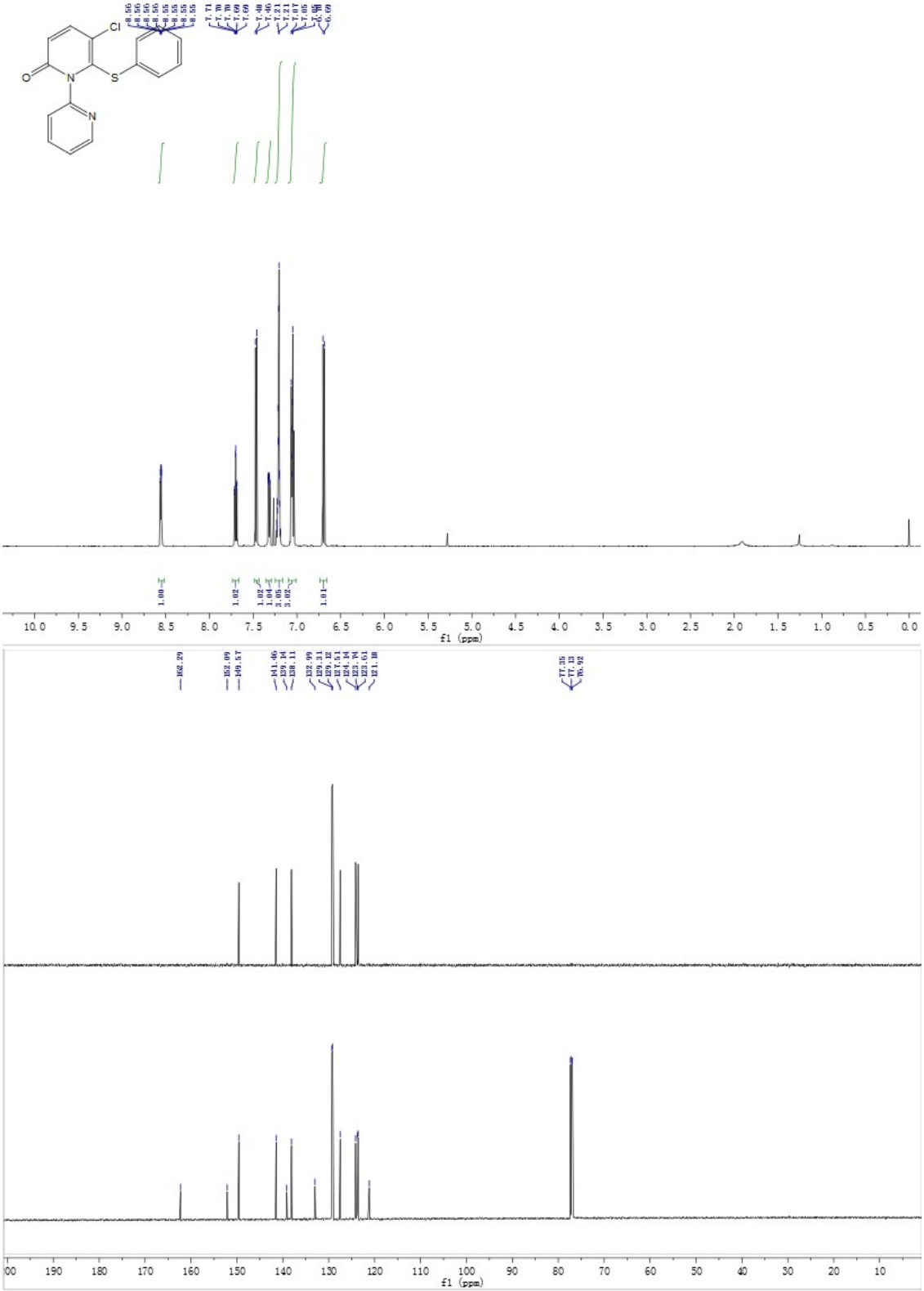


5-fluoro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3l)

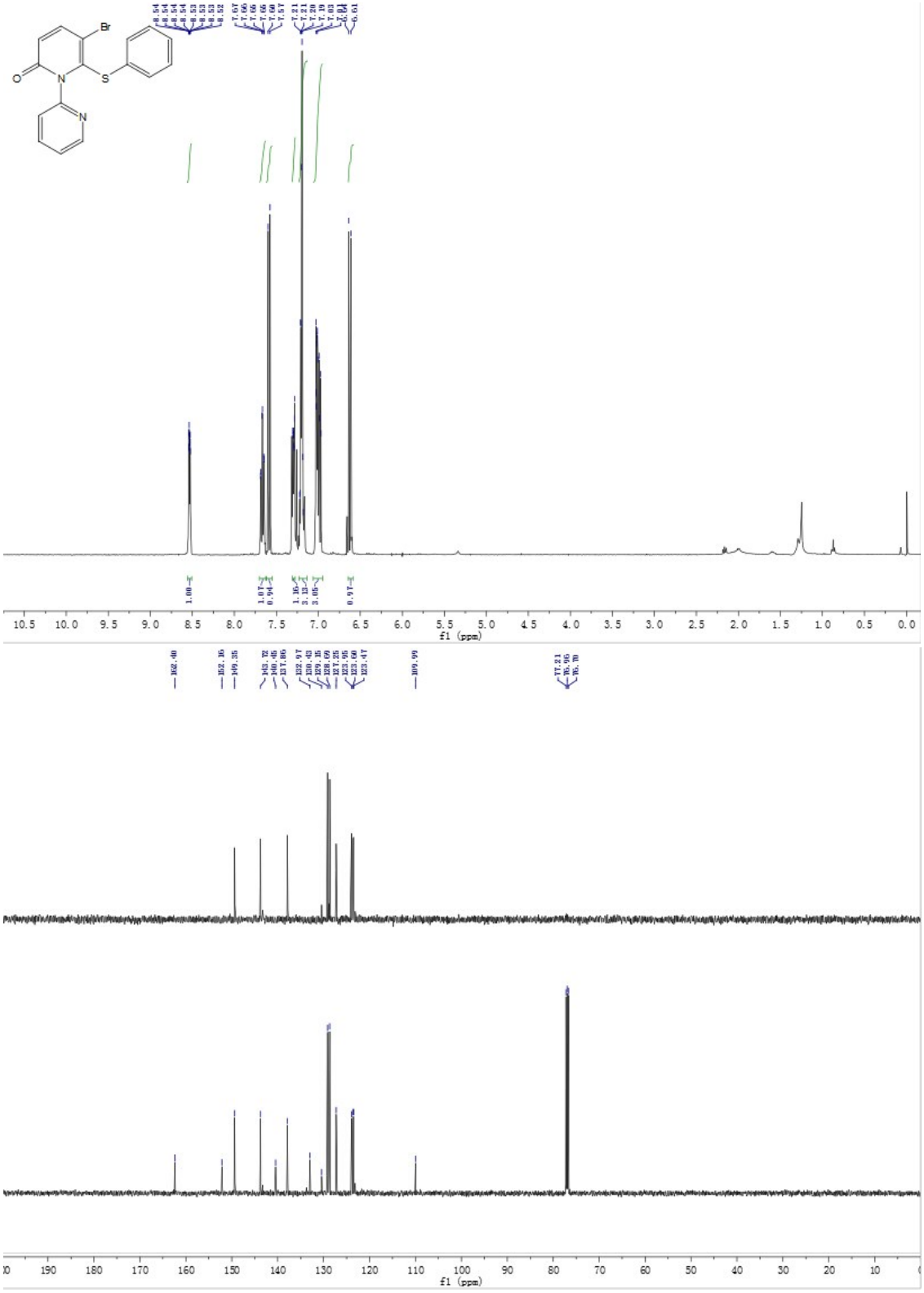




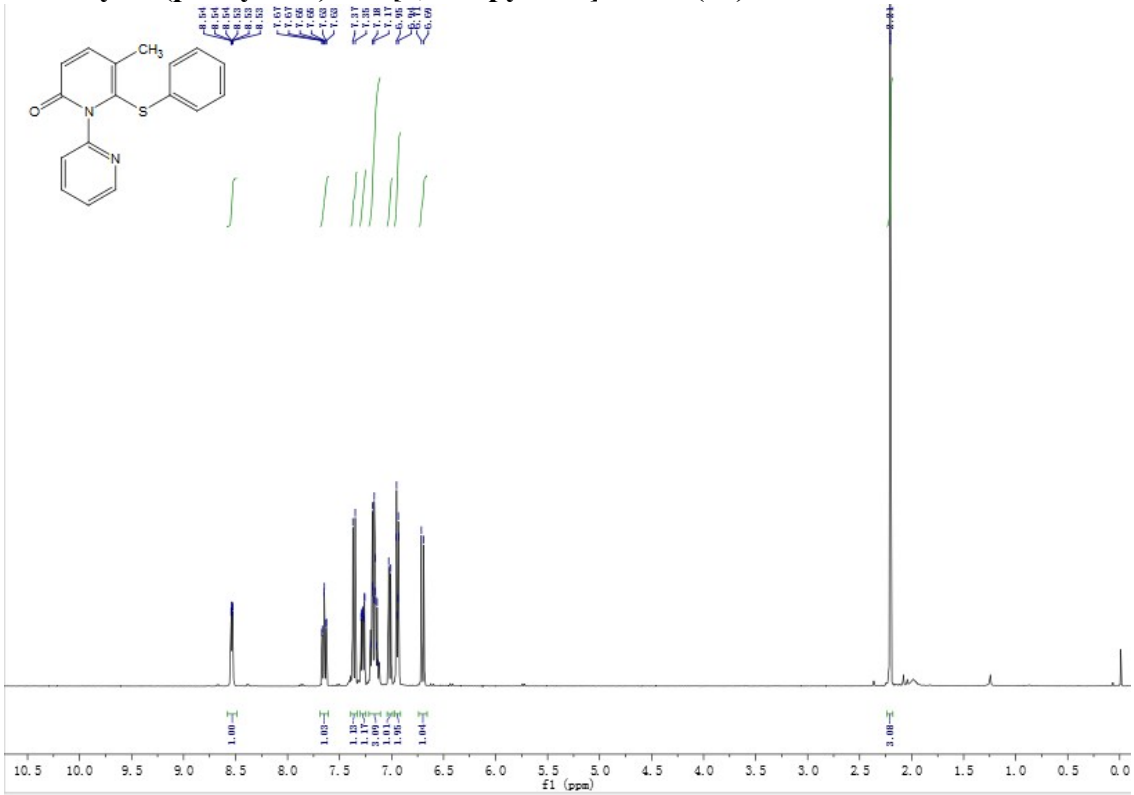
5-chloro-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3m)

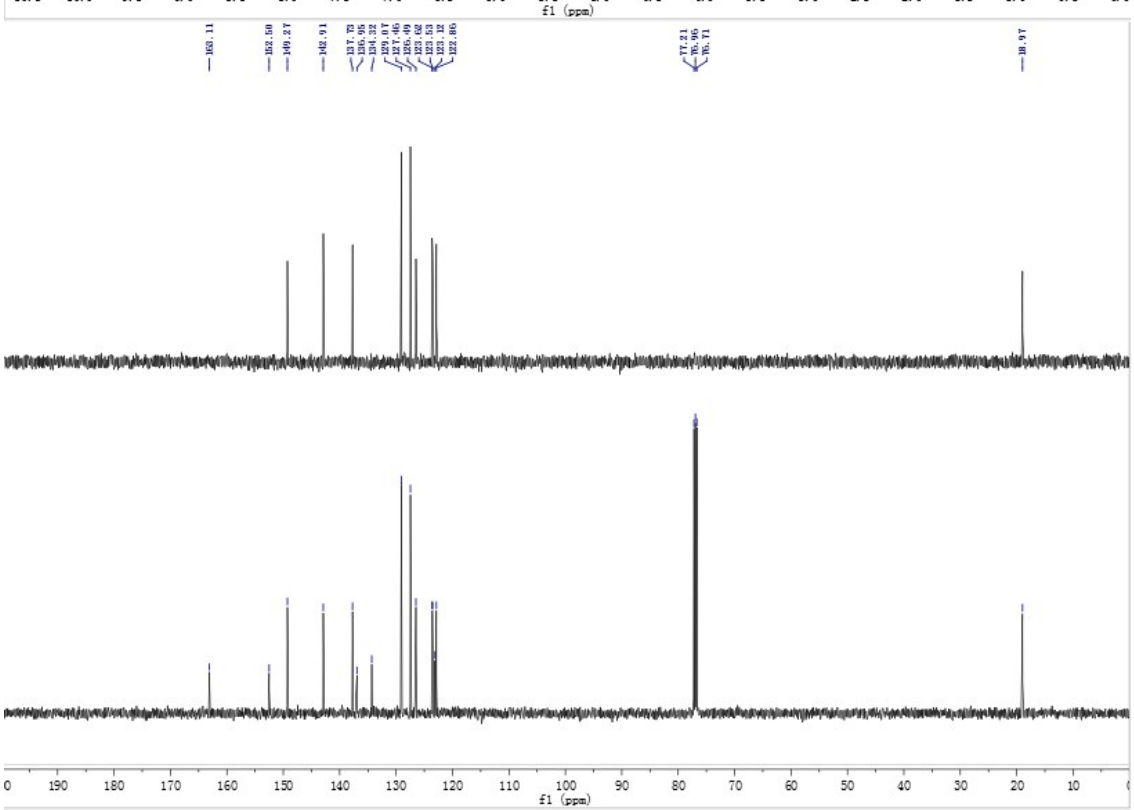


5-bromo-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3n)

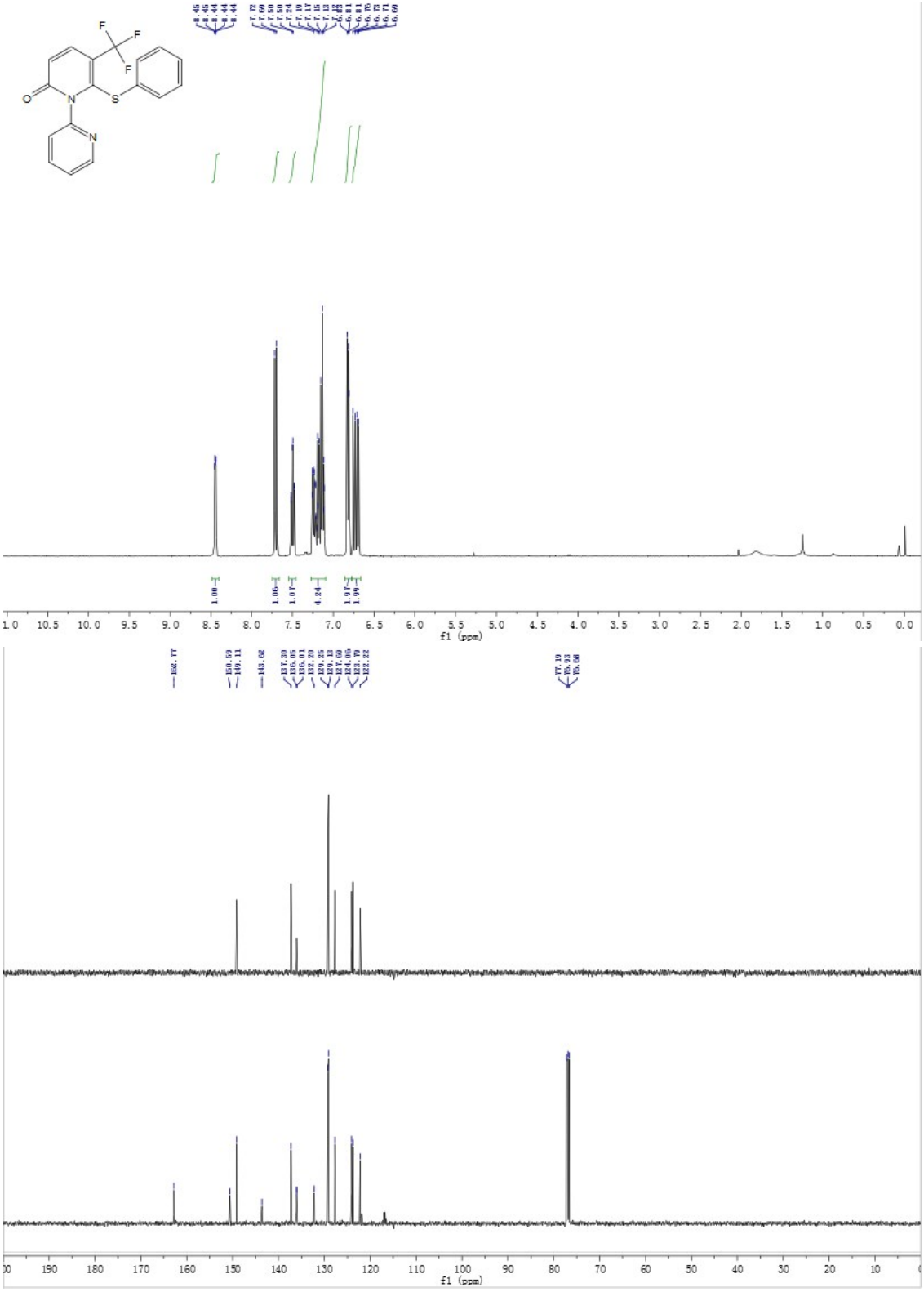


5-methyl-6-(phenylthio)-2*H*-[1,2'-bipyridin]-2-one (3o)

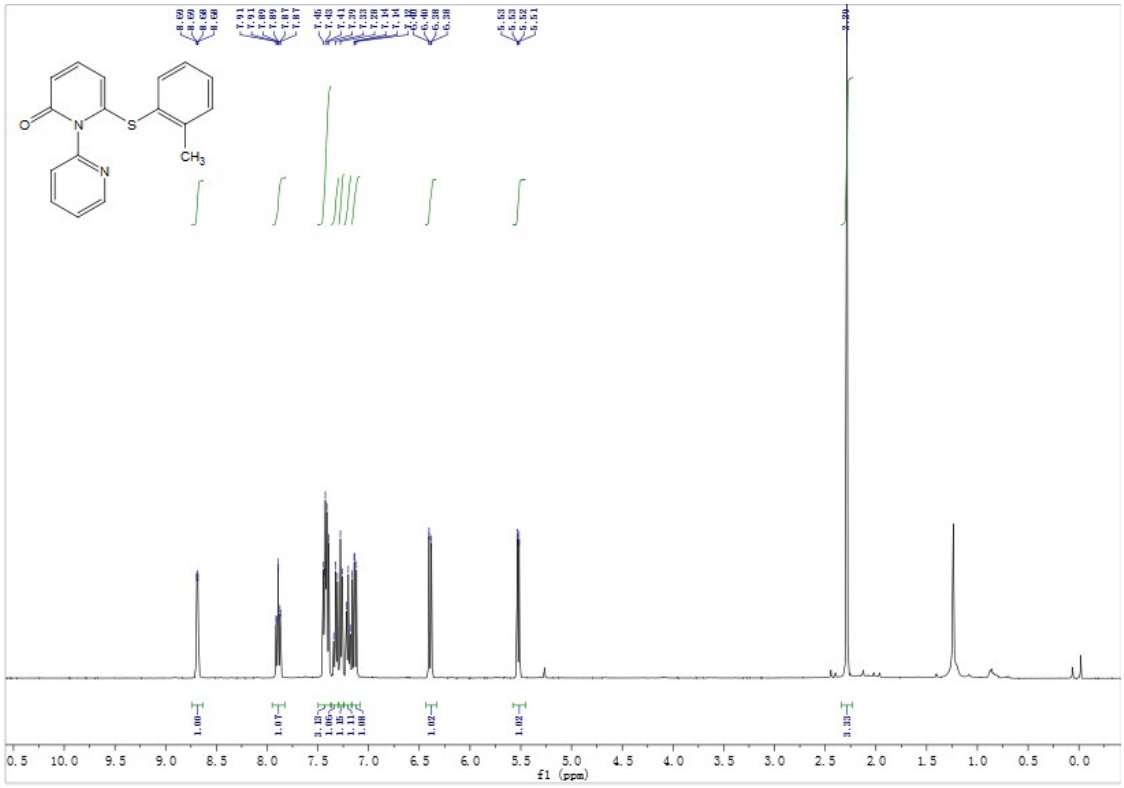


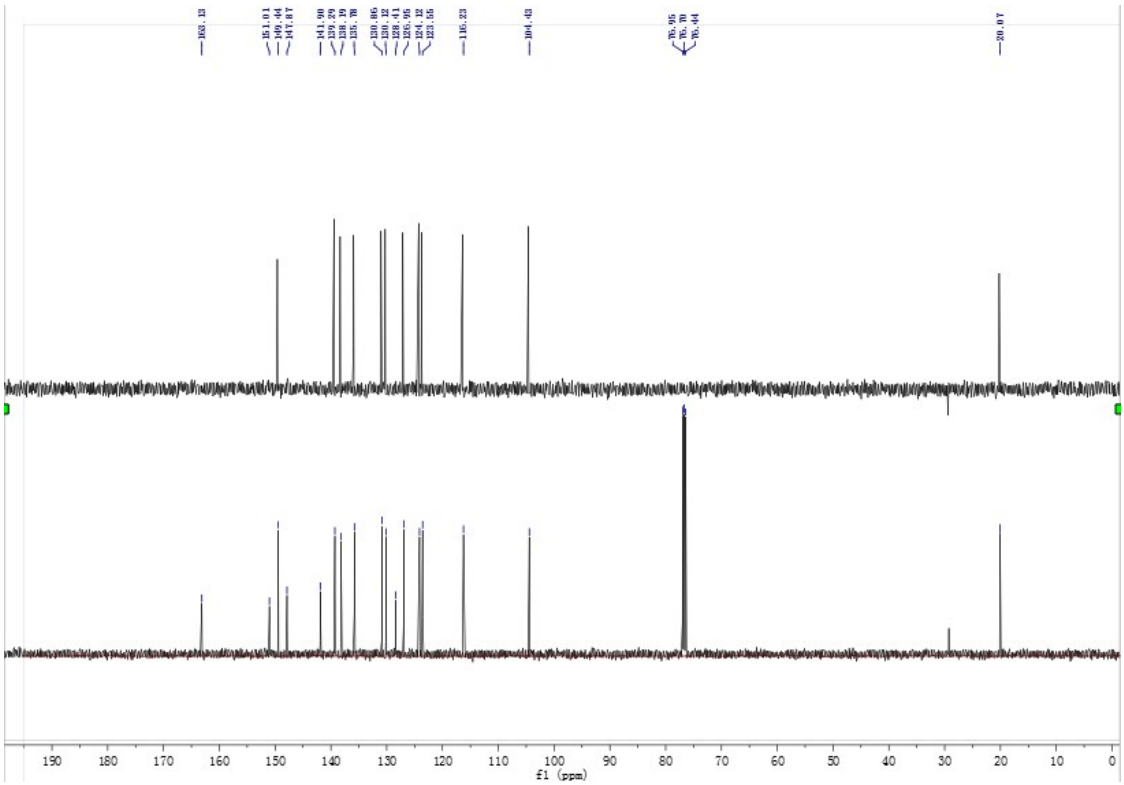


6-(phenylthio)-5-(trifluoromethyl)-2*H*-[1,2'-bipyridin]-2-one (3p)

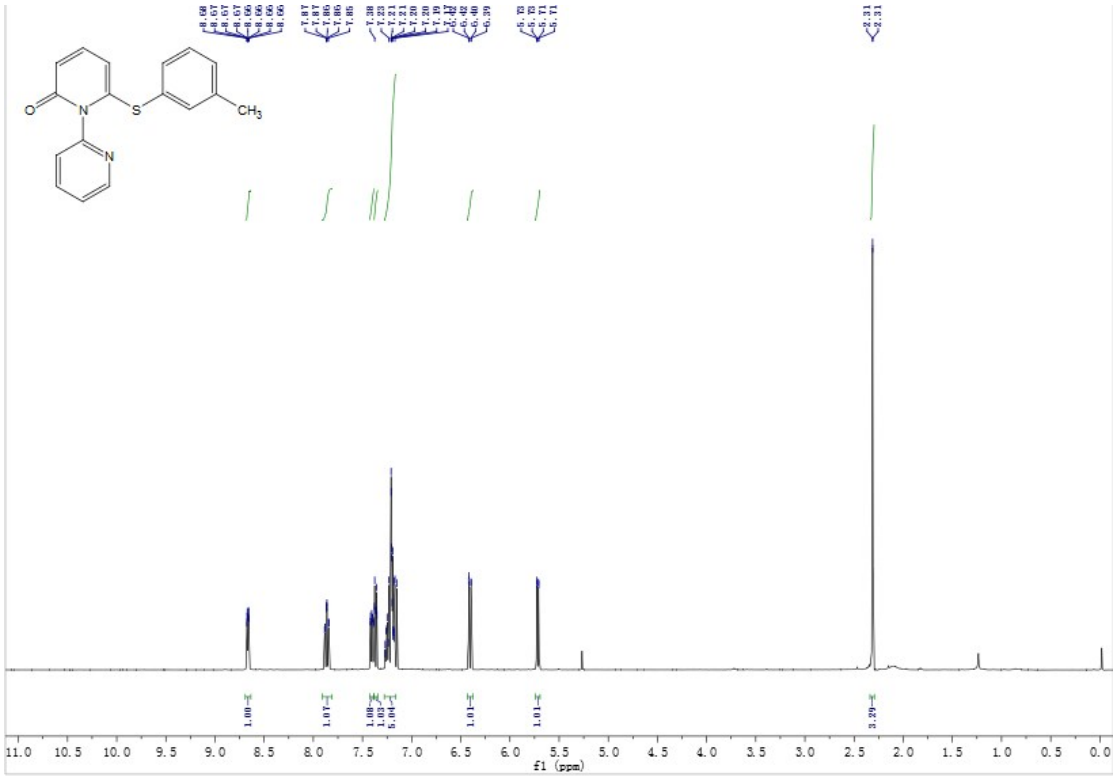


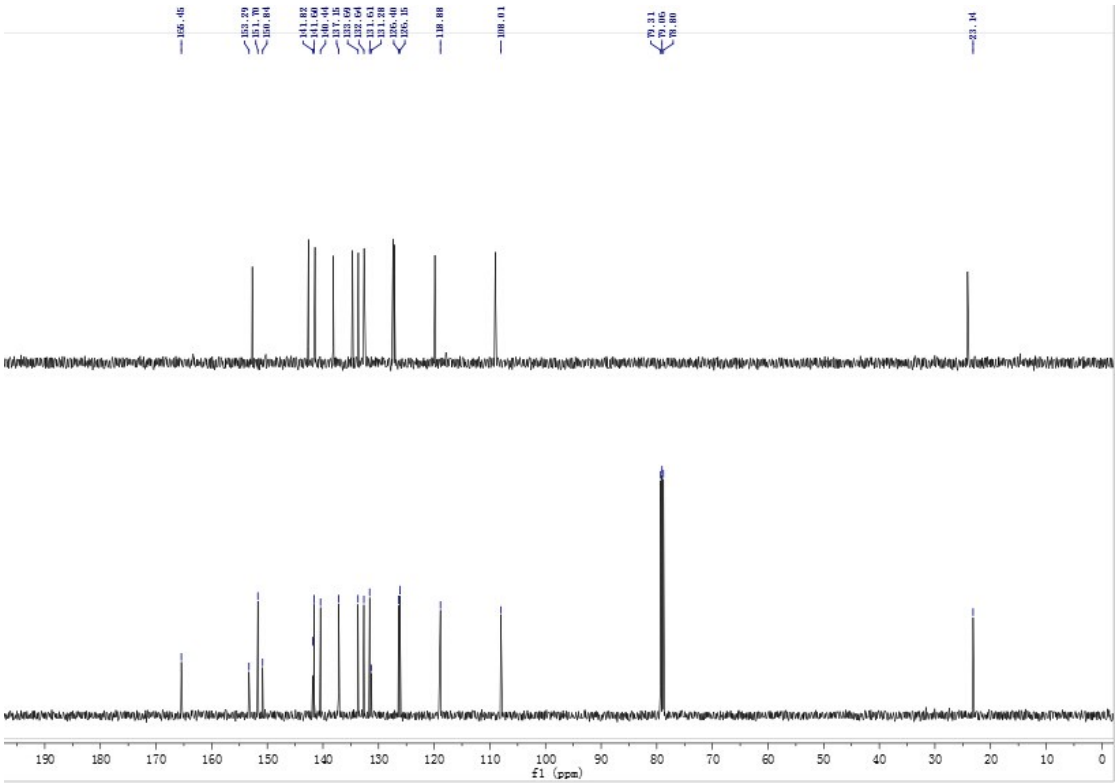
6-(*o*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4a)



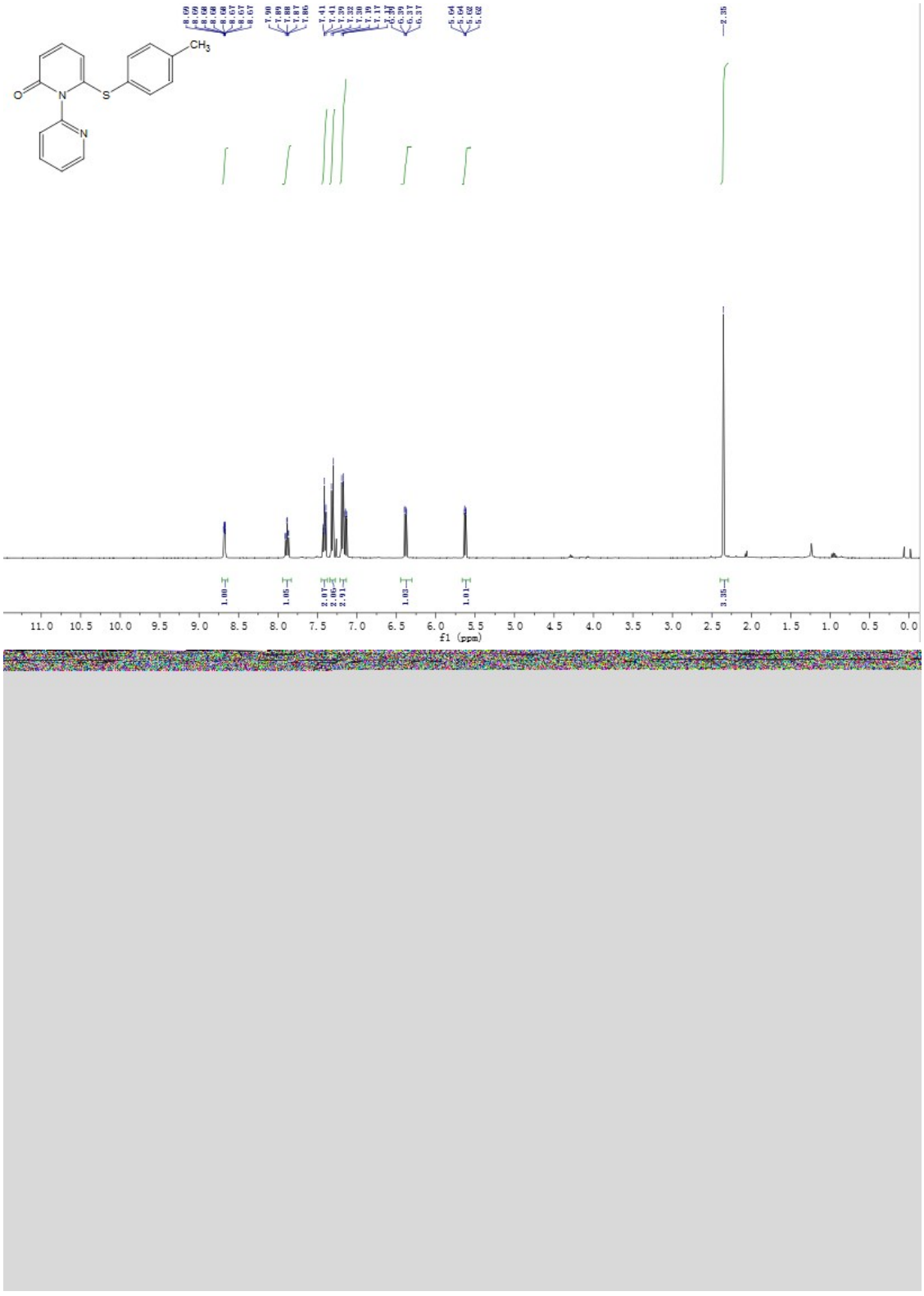


6-(*m*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4b)

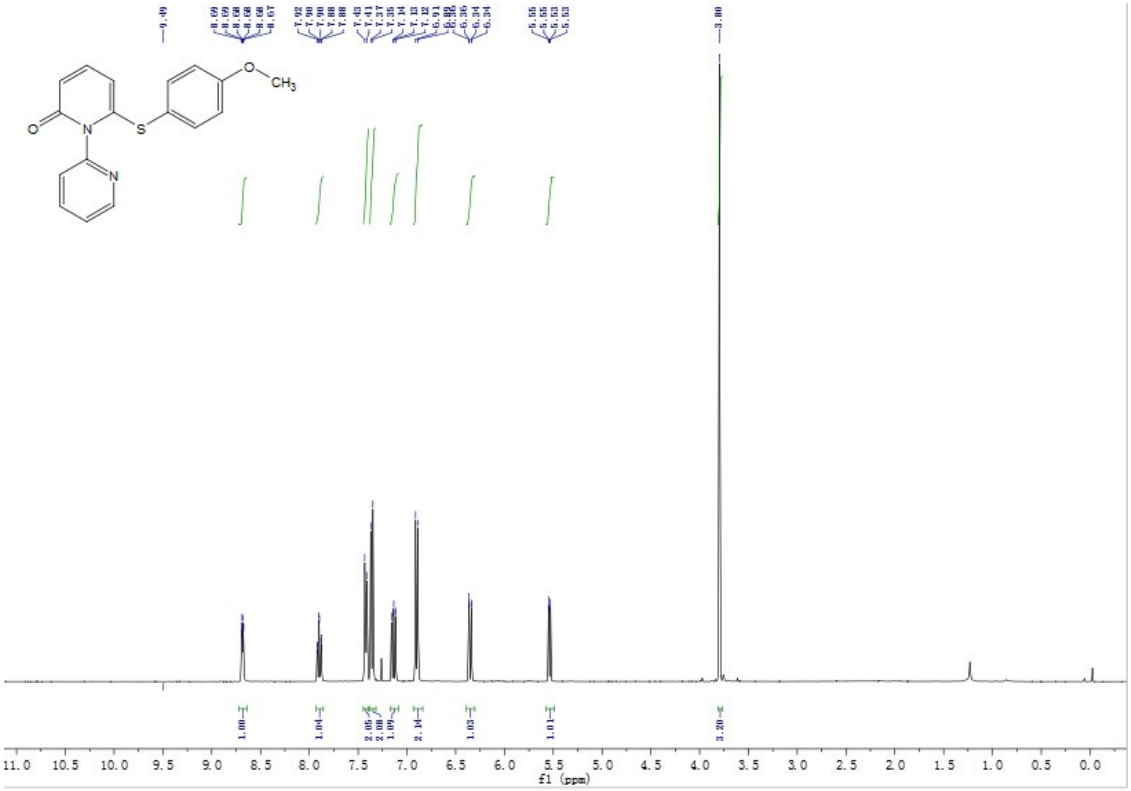


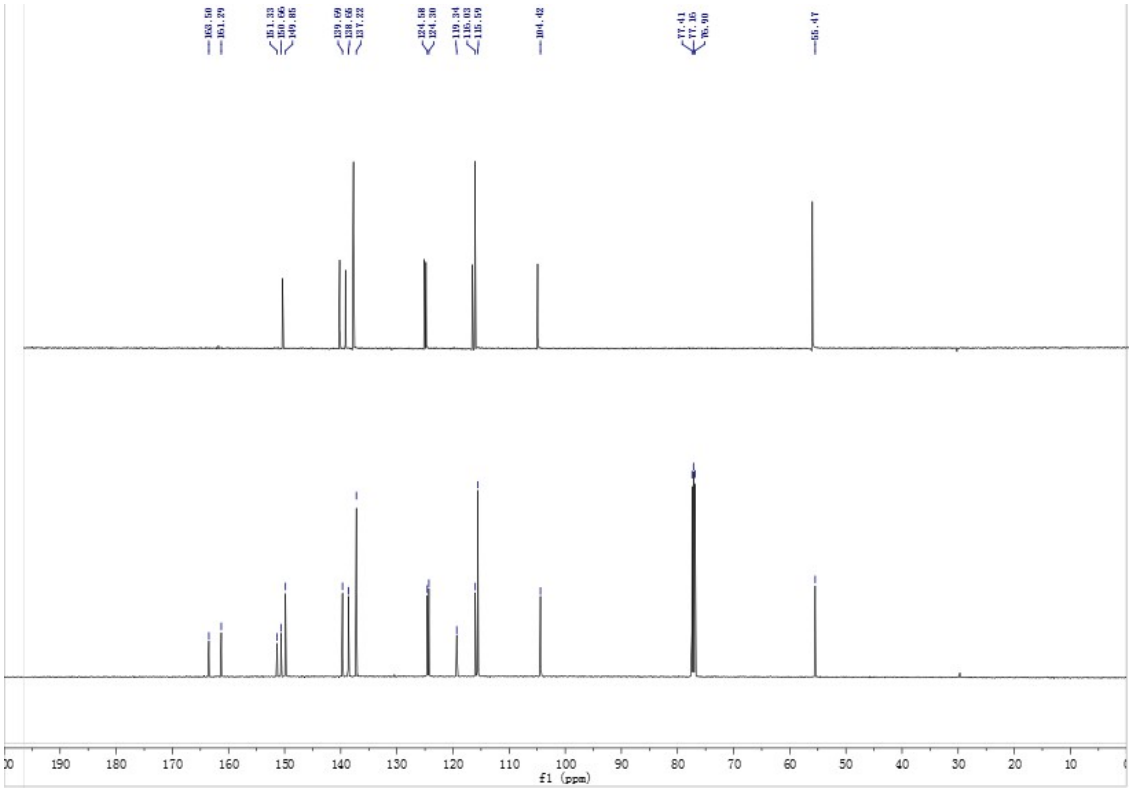


6-(*p*-tolylthio)-2*H*-[1,2'-bipyridin]-2-one (4c)

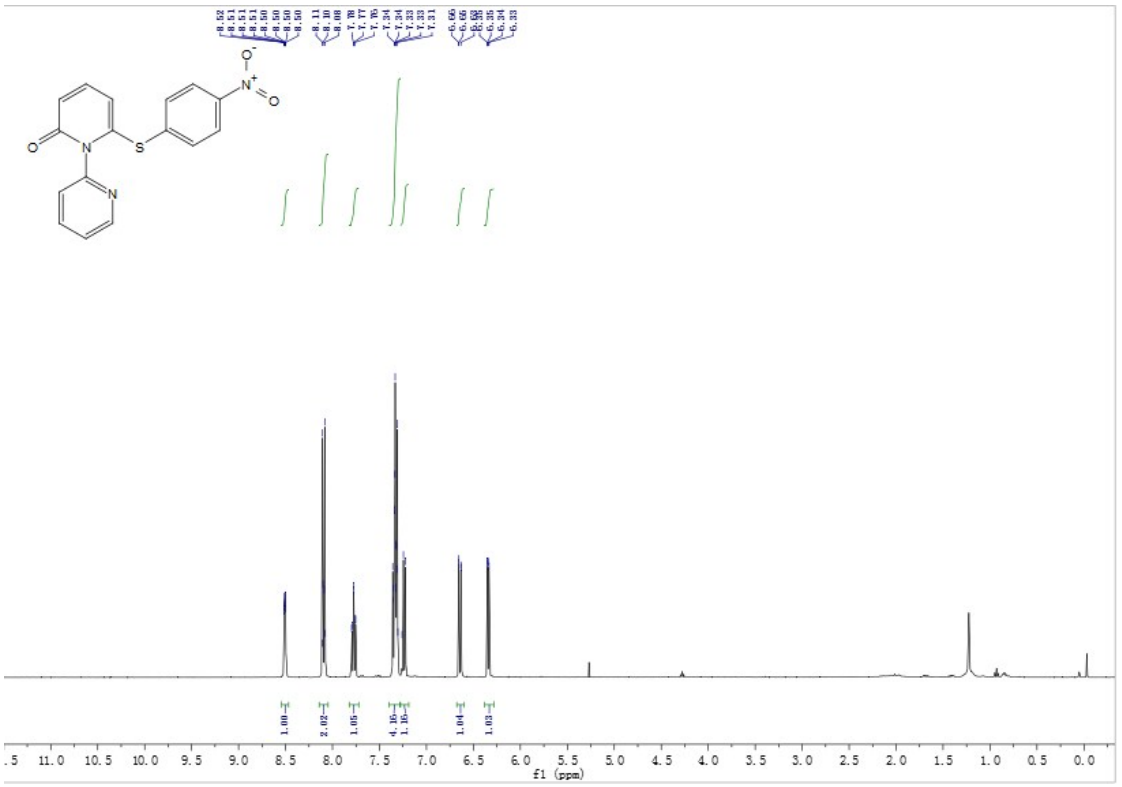


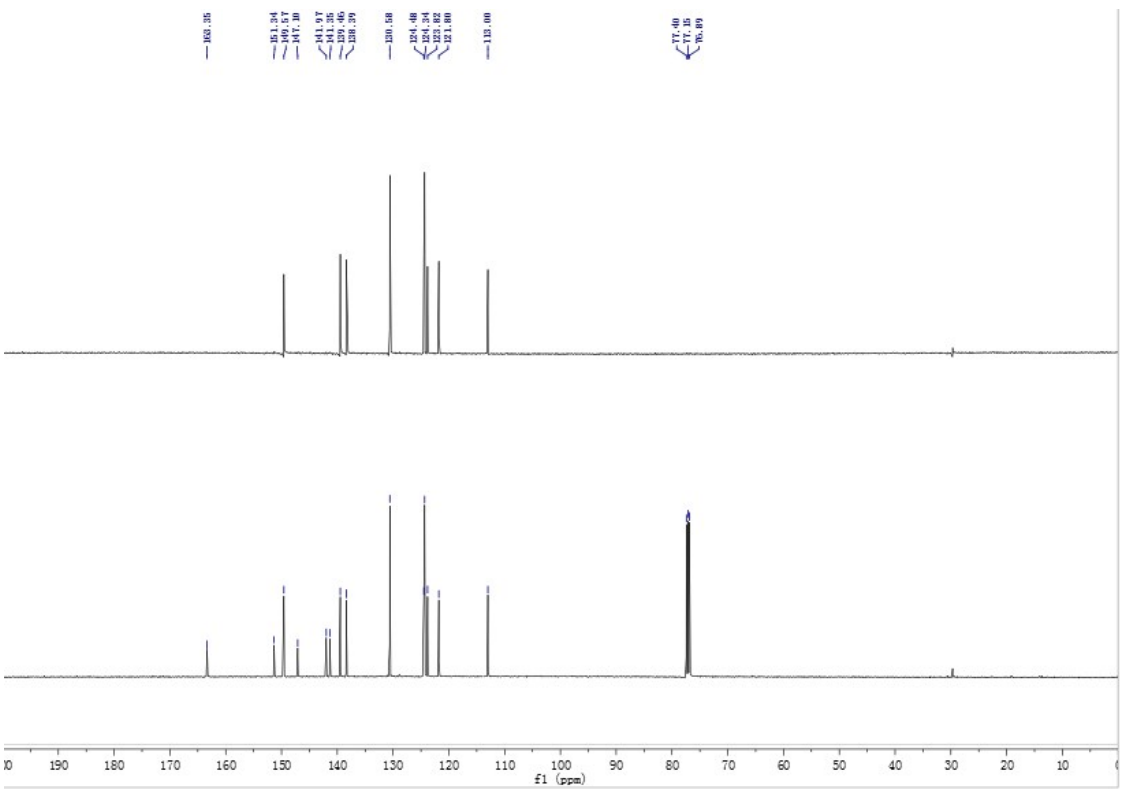
6-((4-methoxyphenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4d)



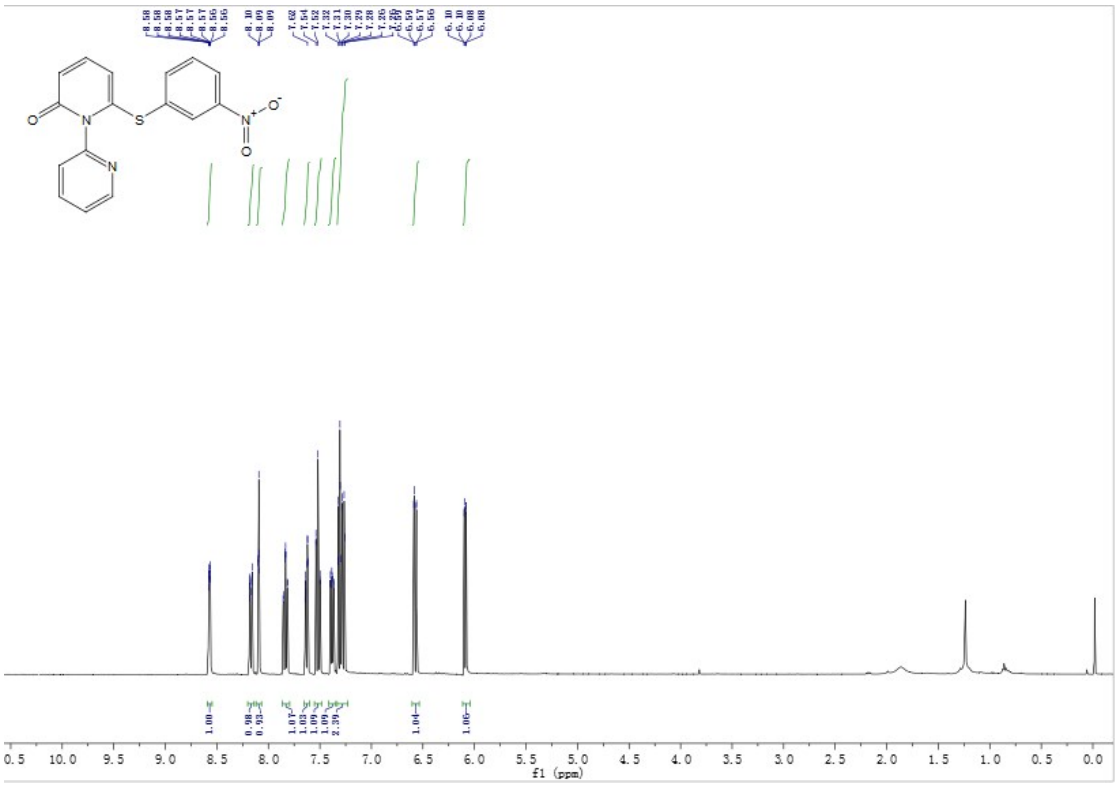


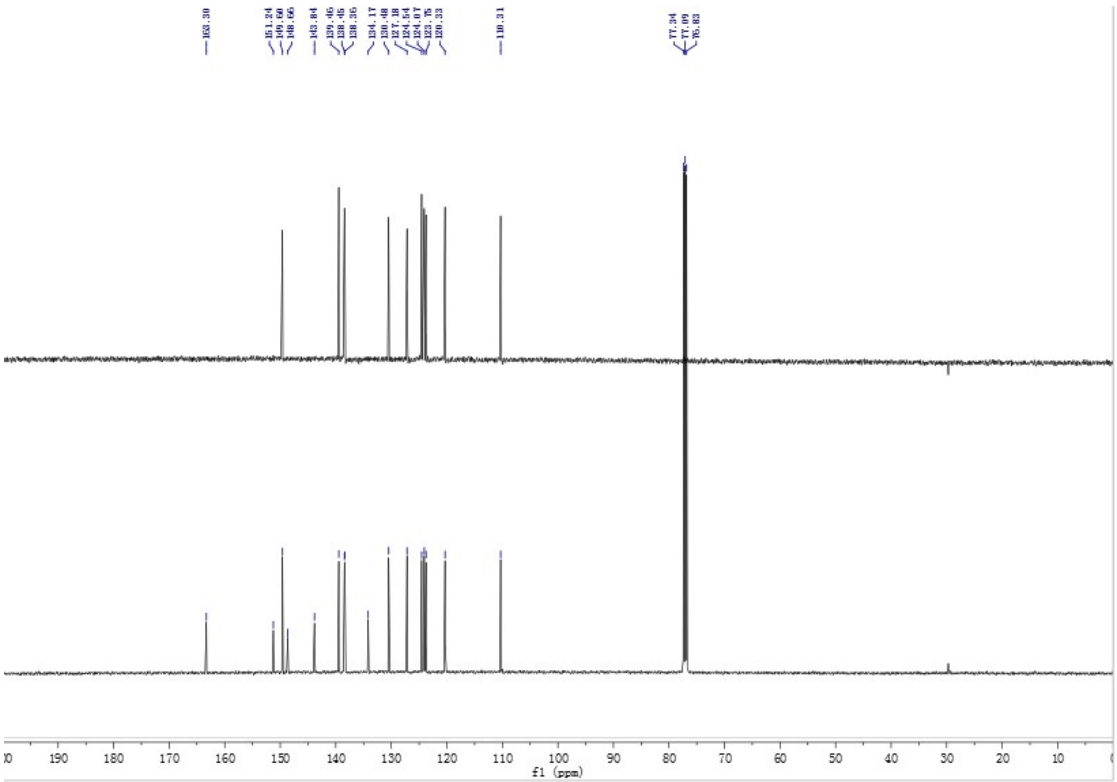
6-((4-nitrophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4e)



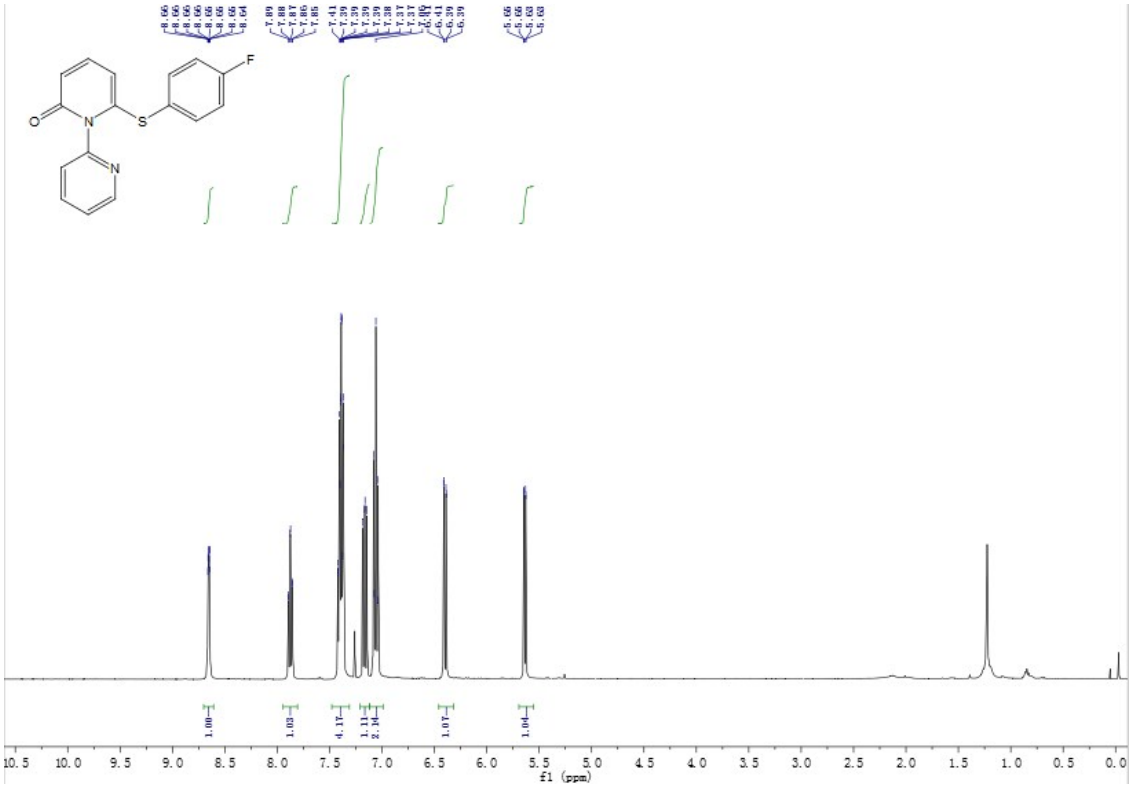


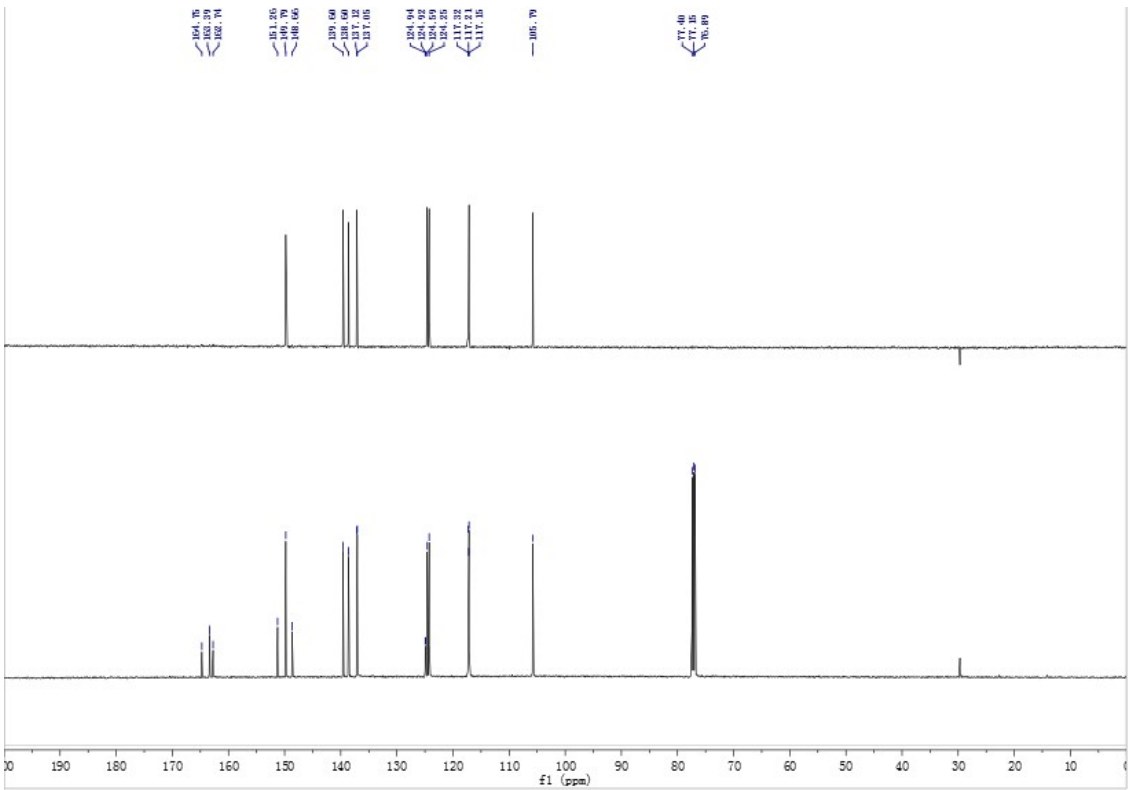
6-((3-nitrophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4f)



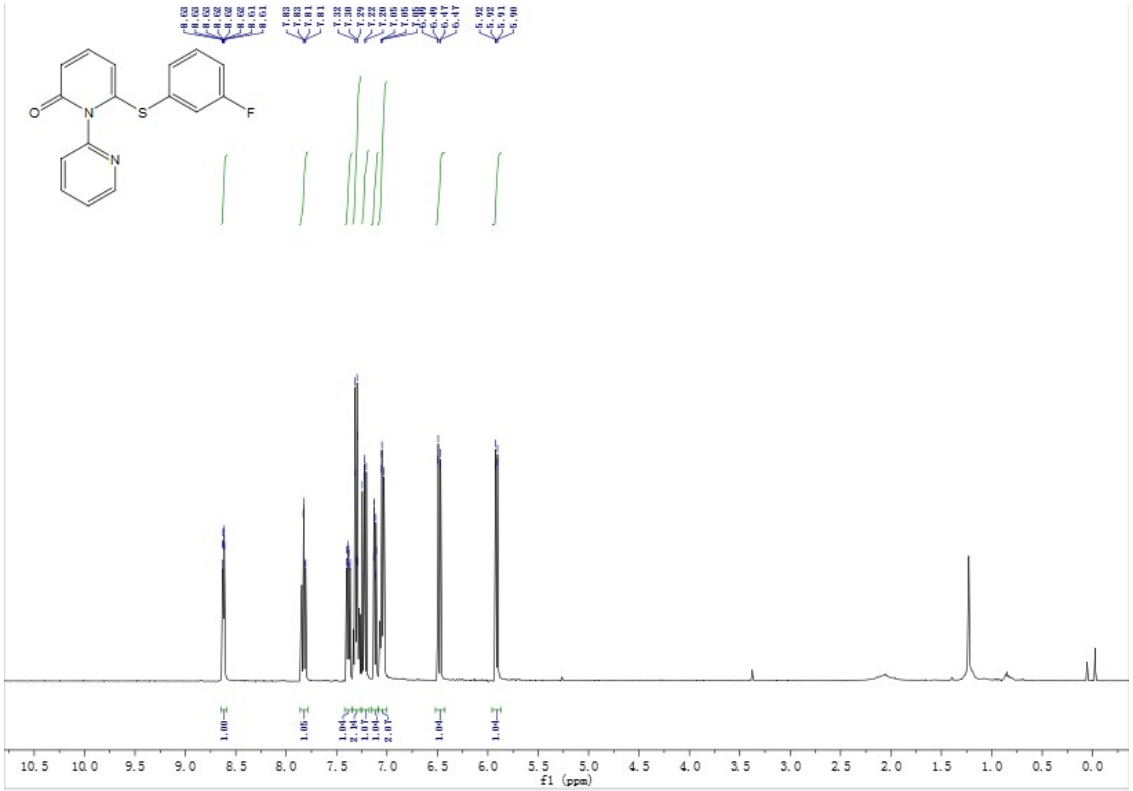


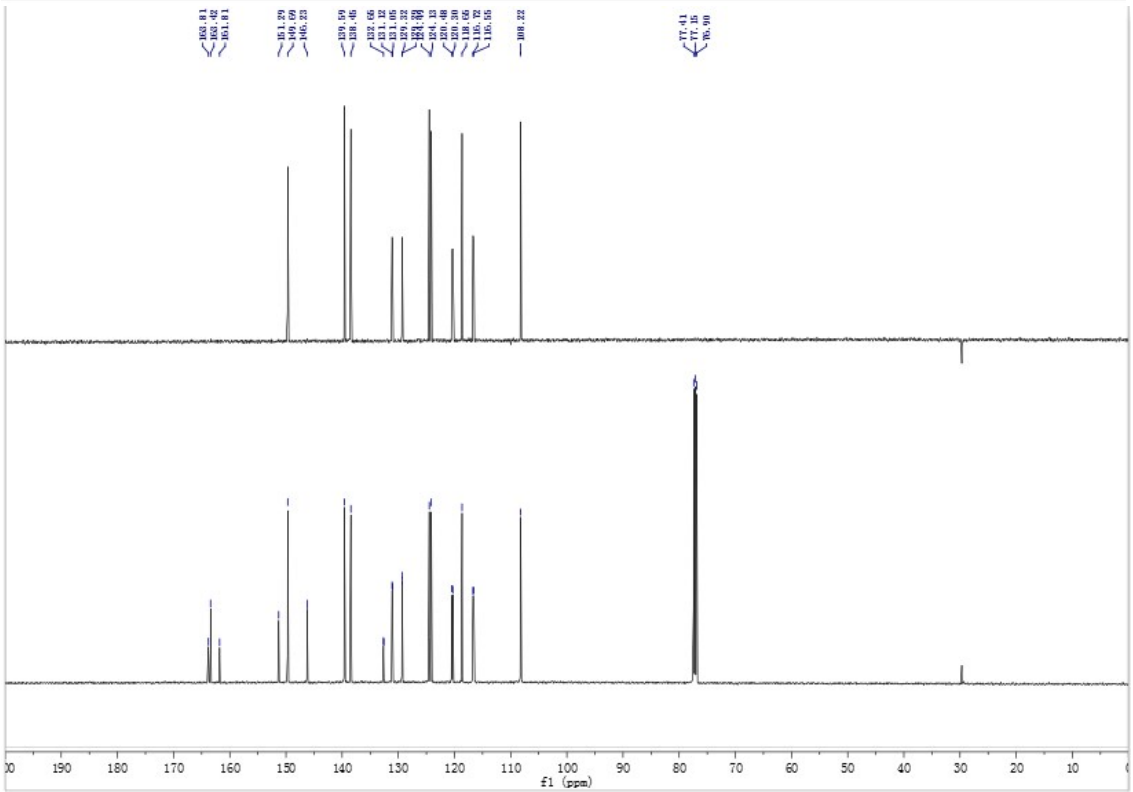
6-((4-fluorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4g)



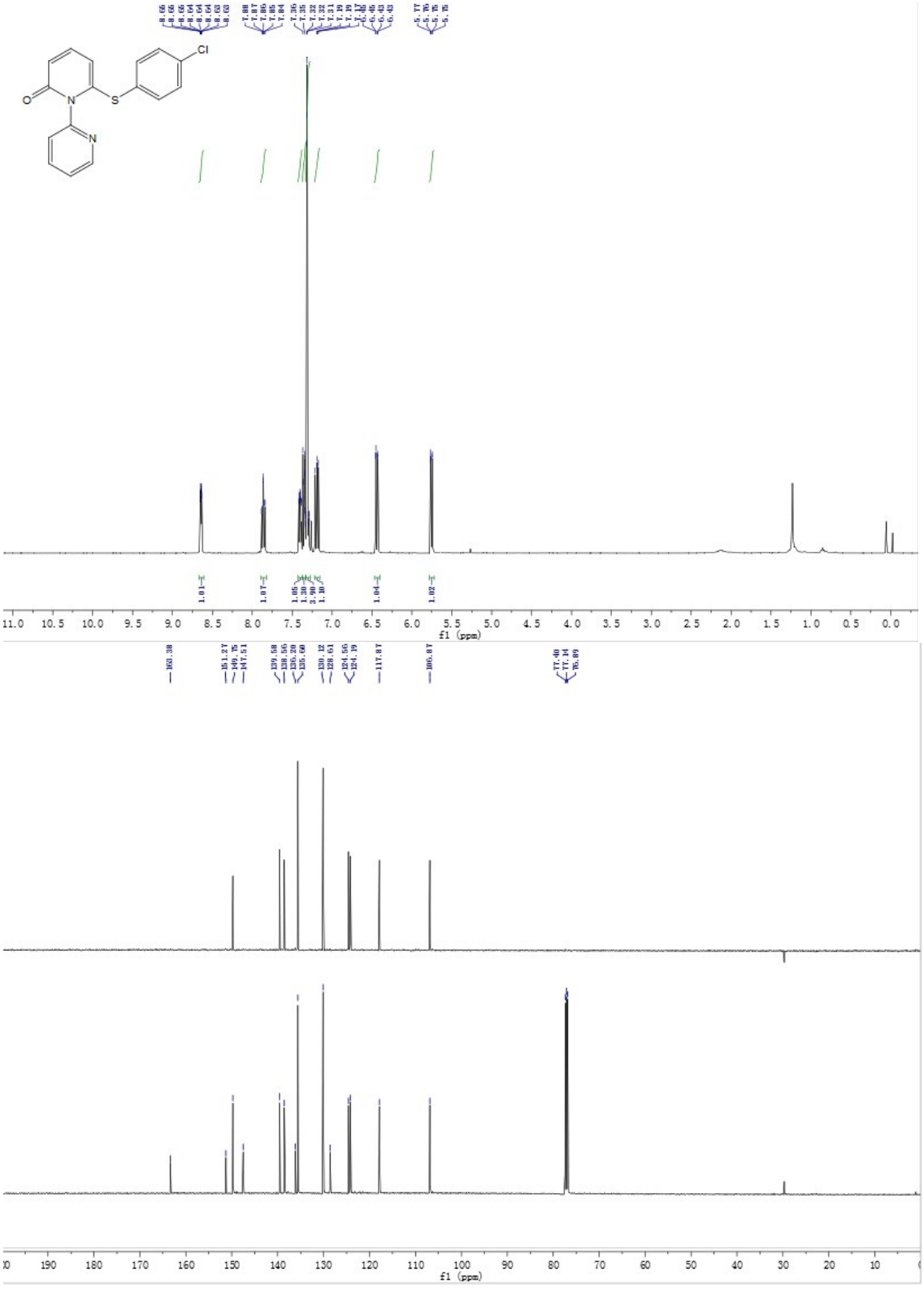


6-((3-fluorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4h)

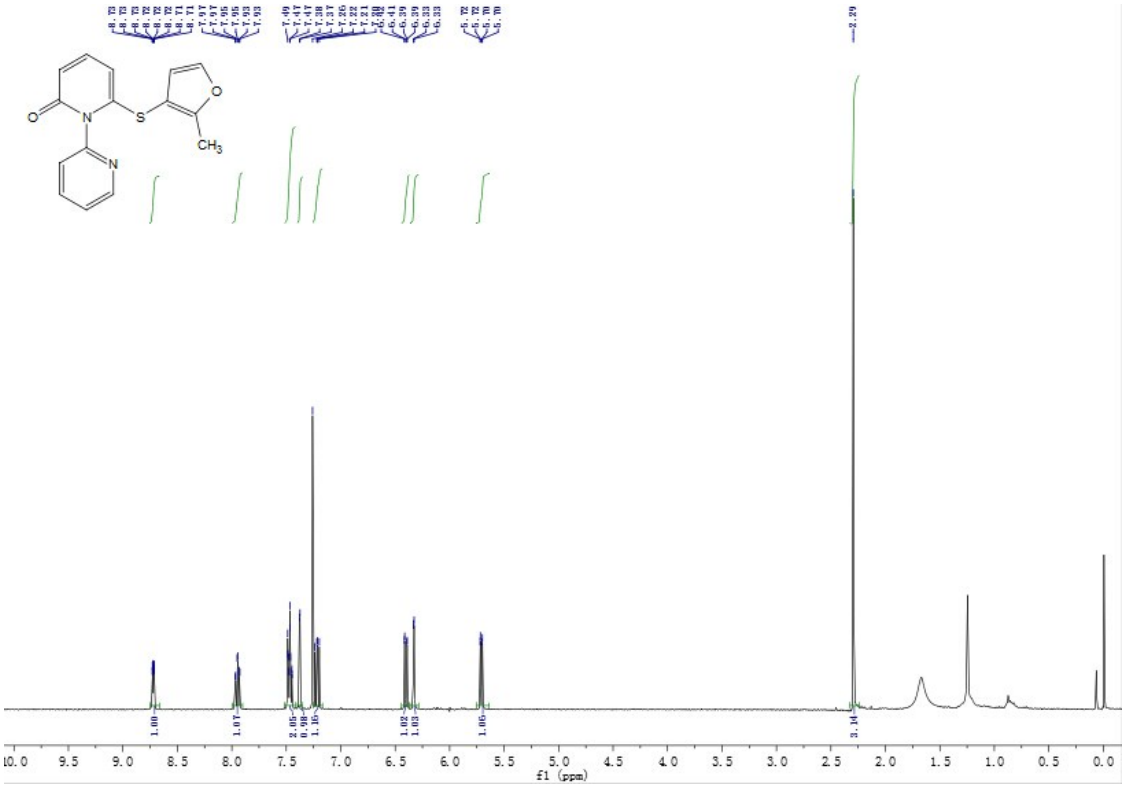


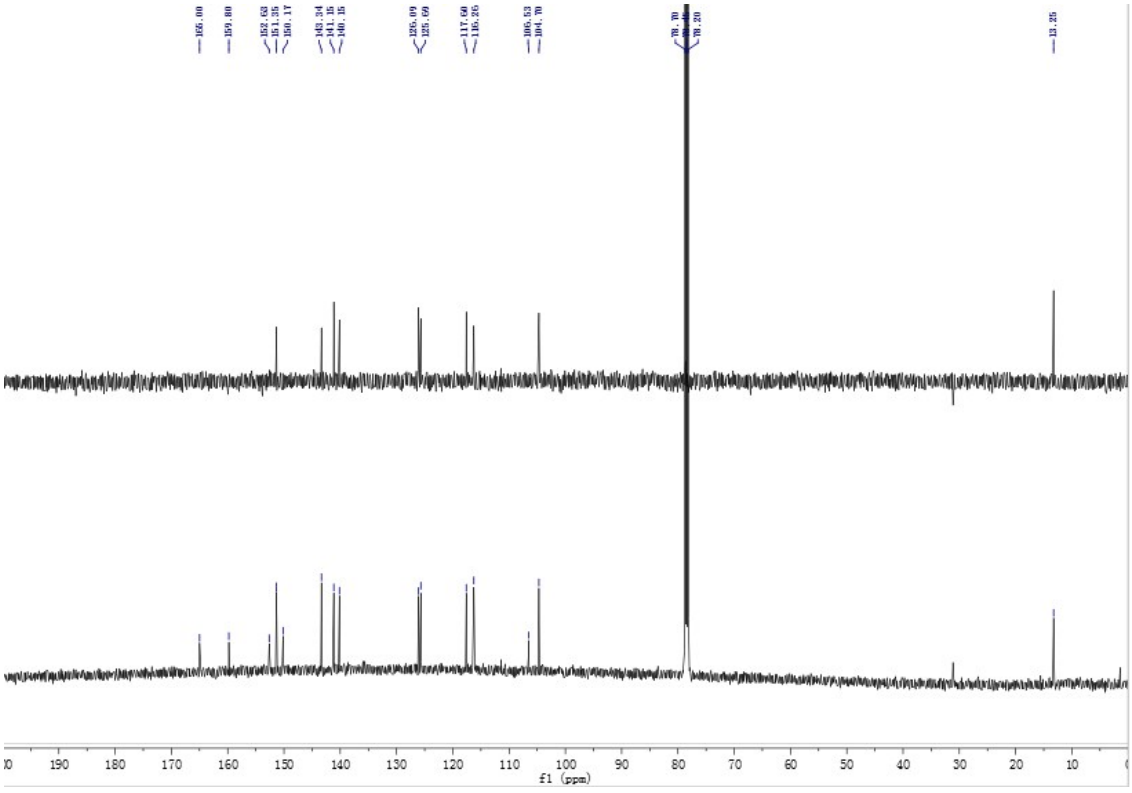


6-((4-chlorophenyl)thio)-2*H*-[1,2'-bipyridin]-2-one (4i)

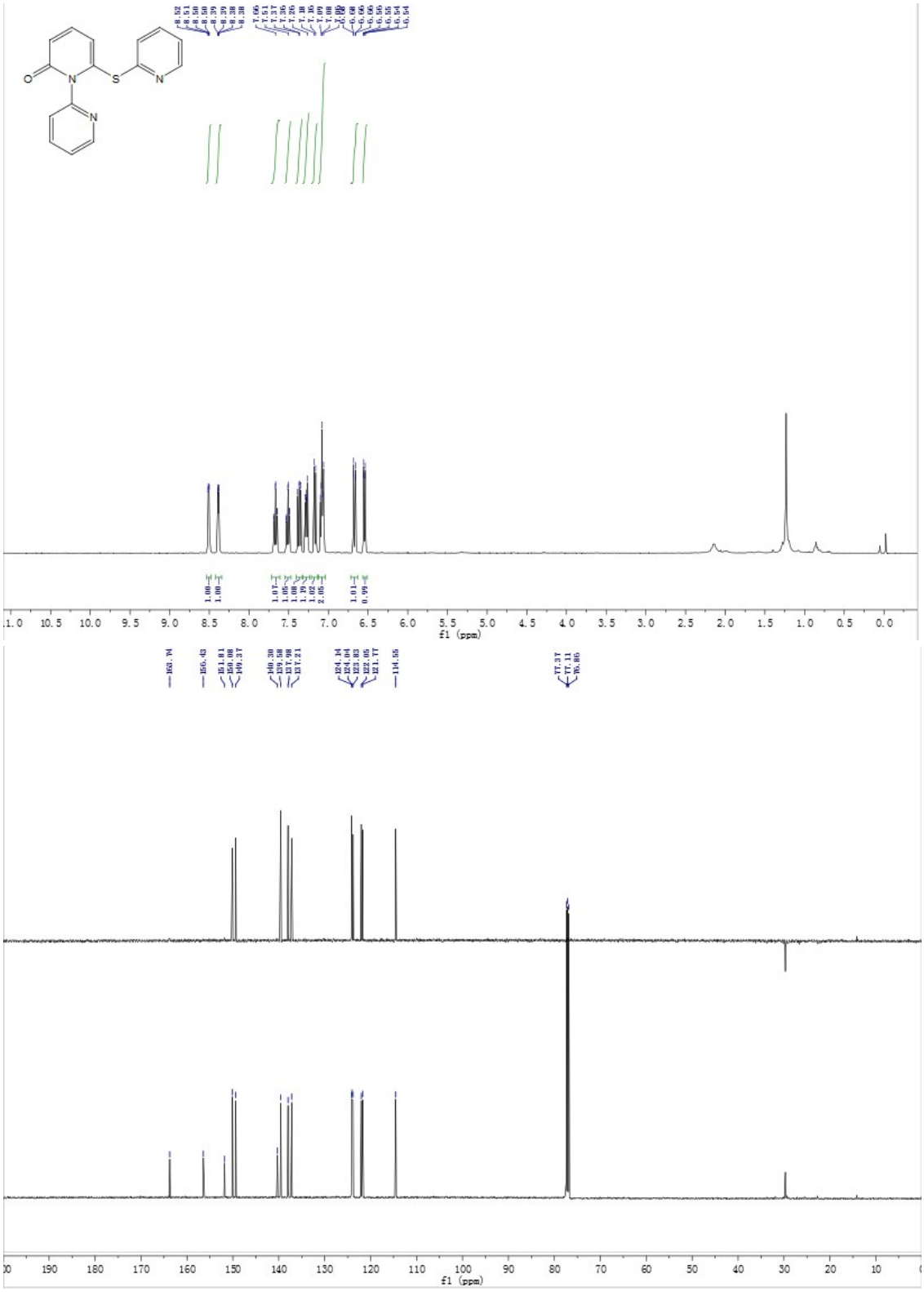


6-((2-methylfuran-3-yl)thio)-2*H*-[1,2'-bipyridin]-2-one (4j)

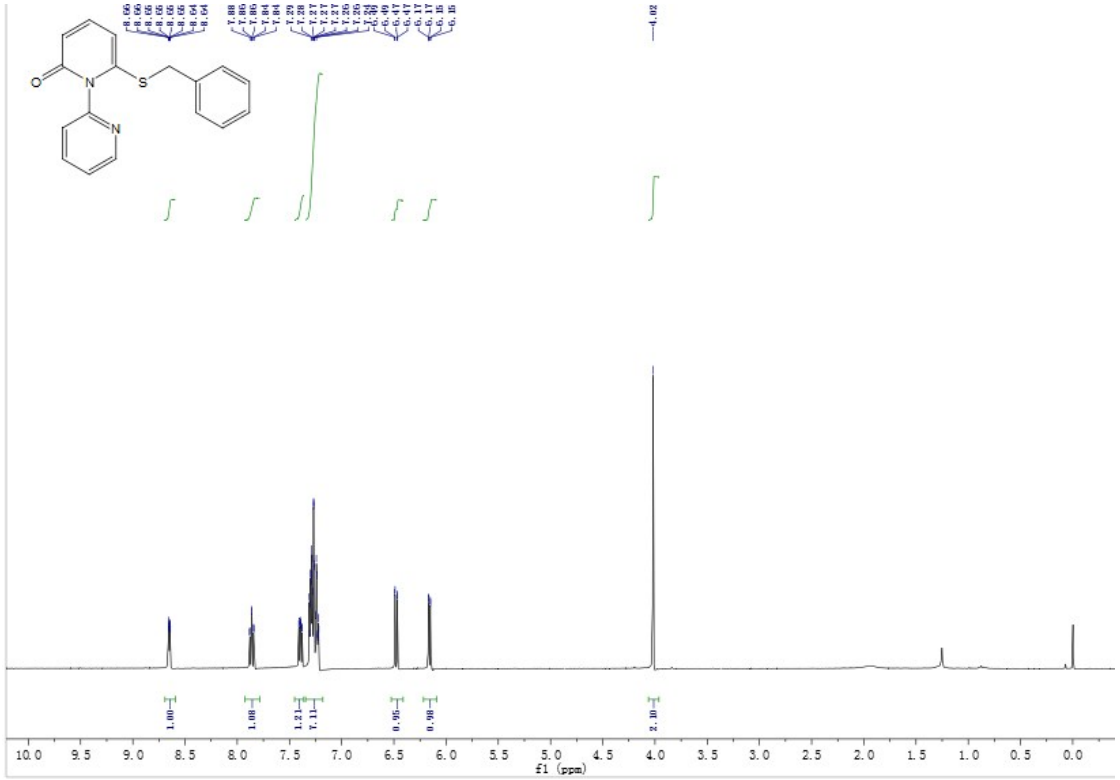


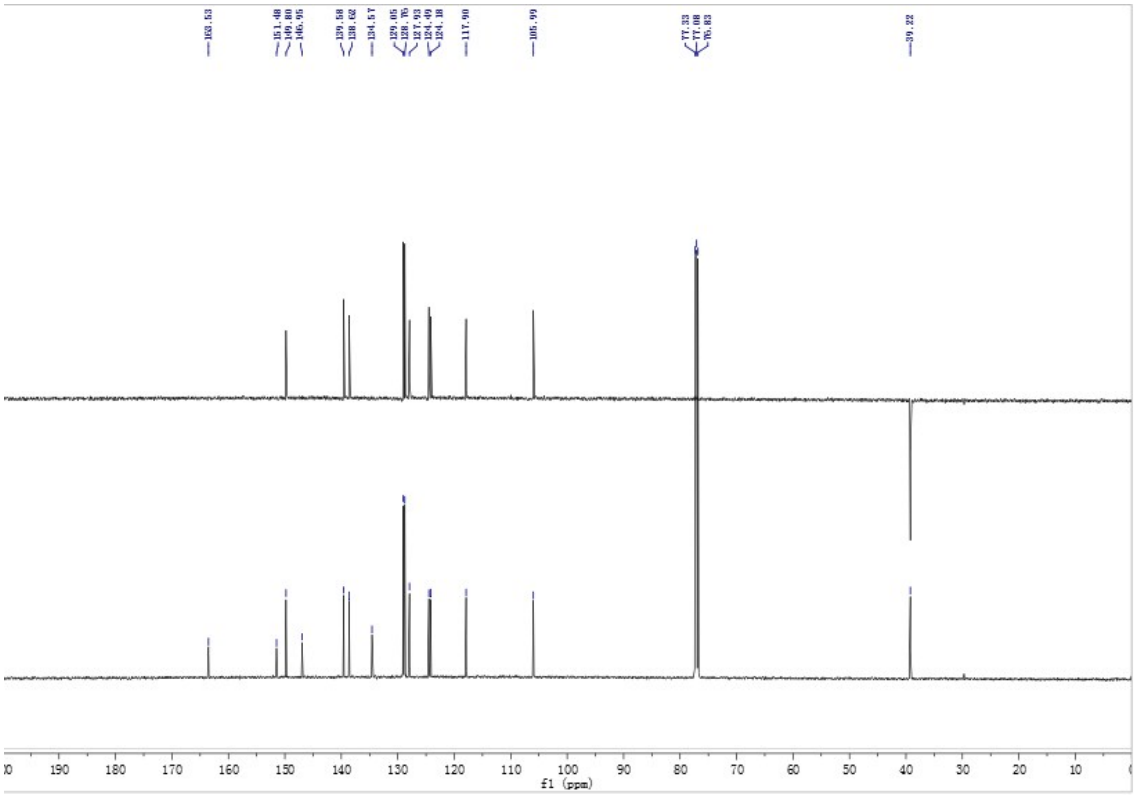


6-(pyridin-2-ylthio)-2*H*-[1,2'-bipyridin]-2-one (4k)

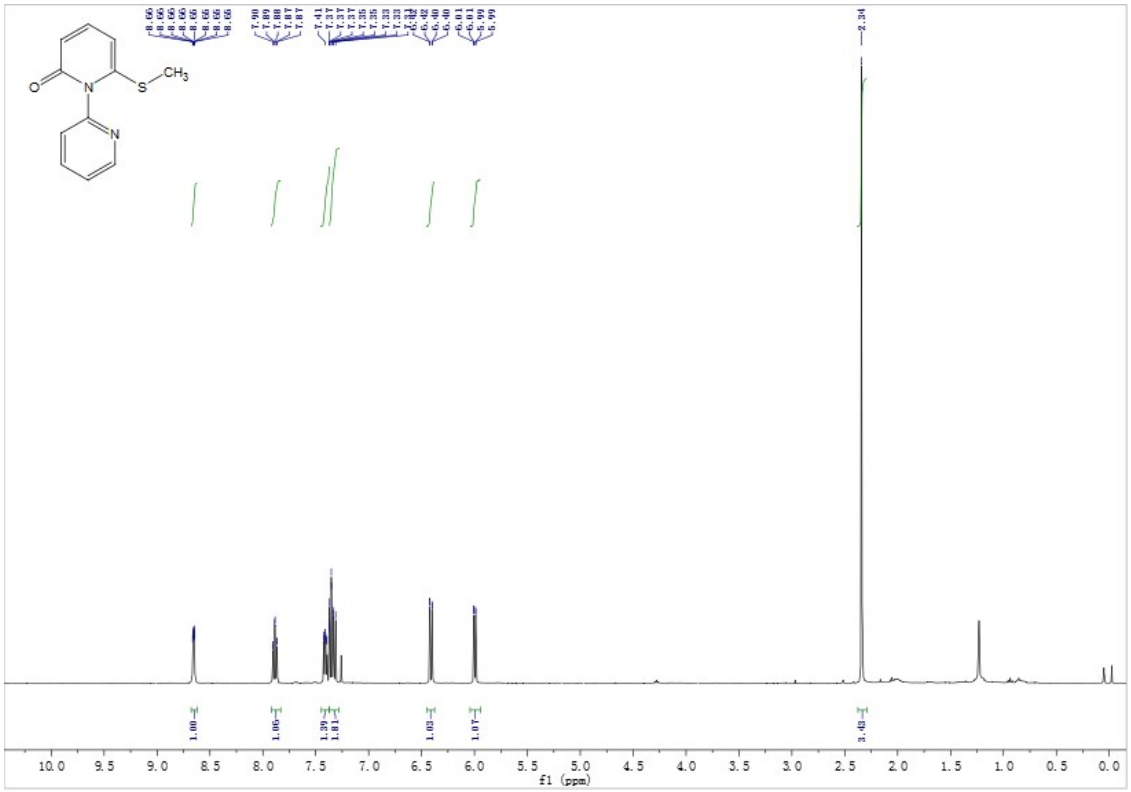


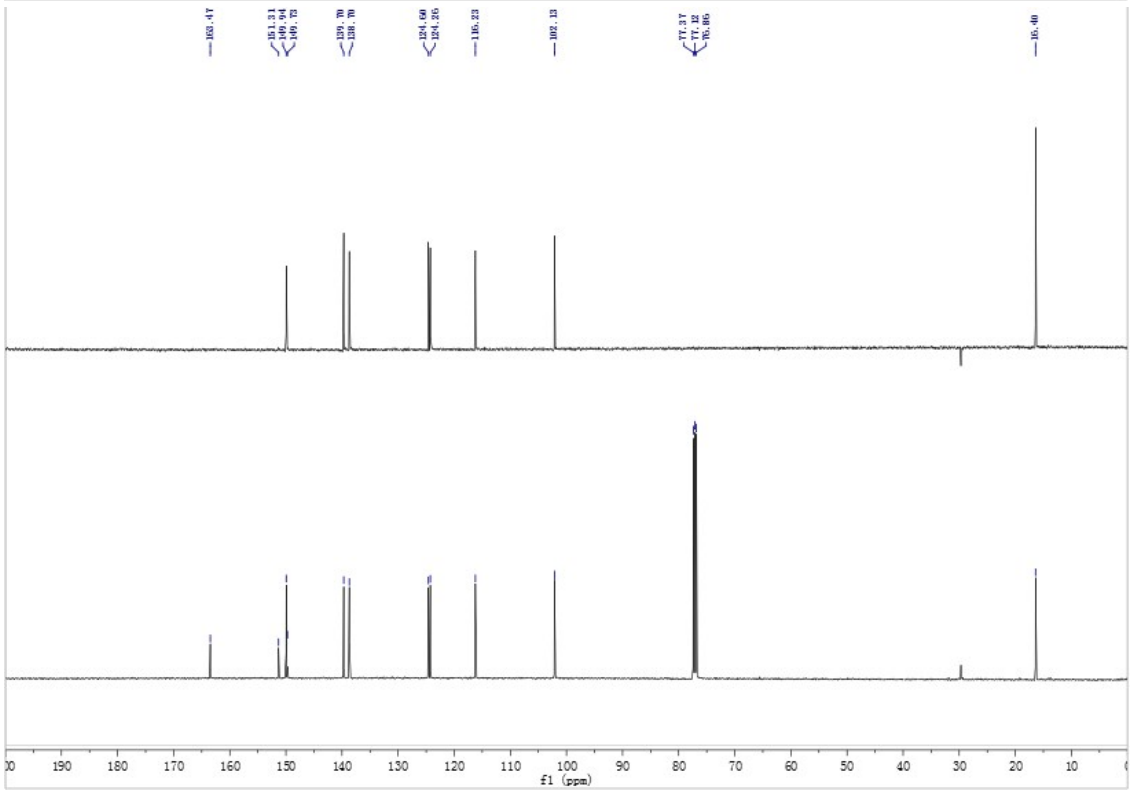
6-(benzylthio)-2*H*-[1,2'-bipyridin]-2-one (4l)



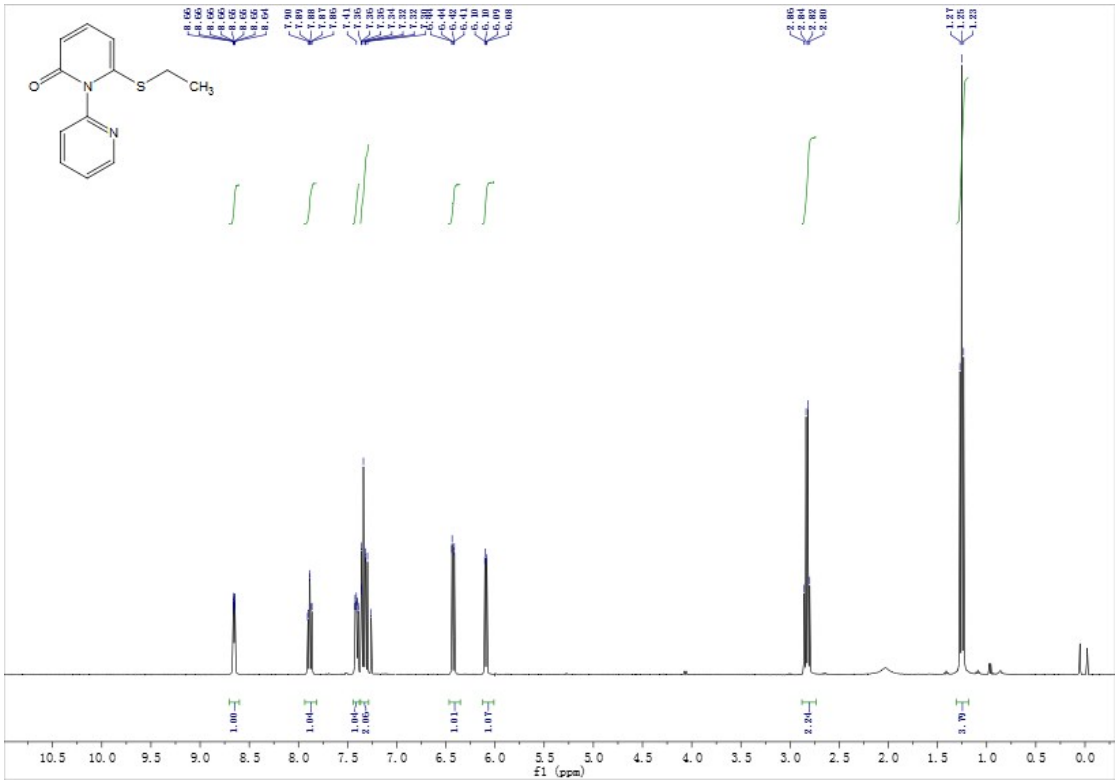


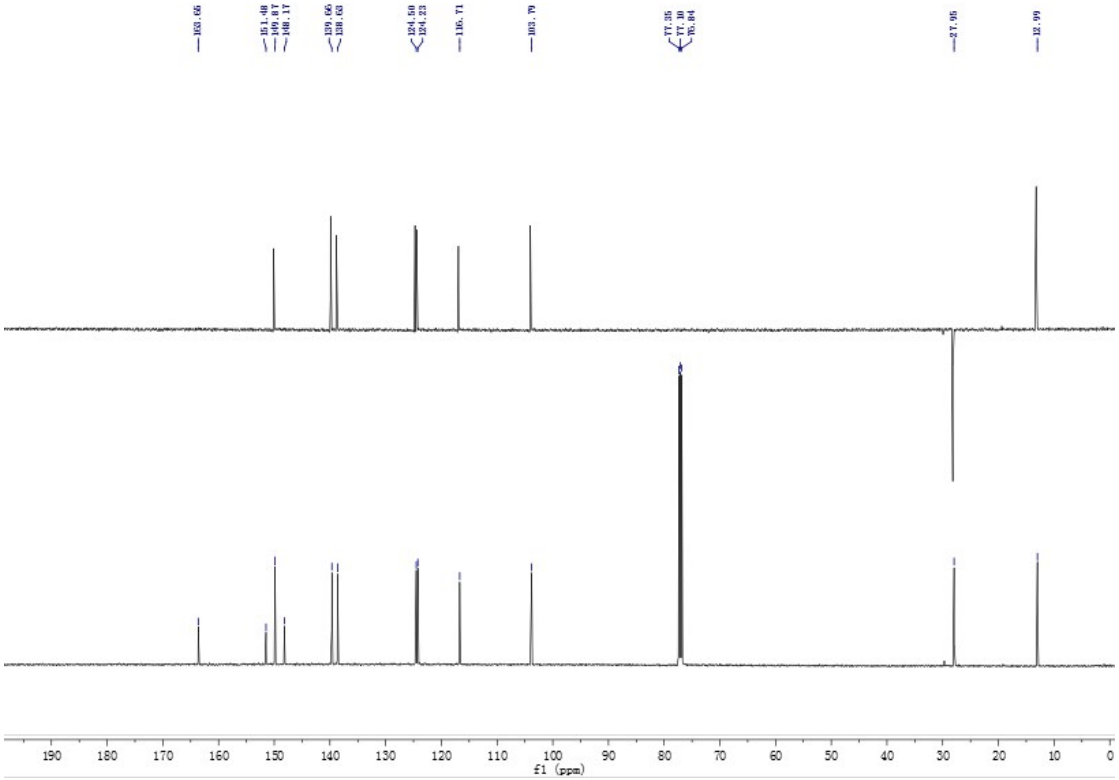
6-(methylthio)-2*H*-[1,2'-bipyridin]-2-one (4m)



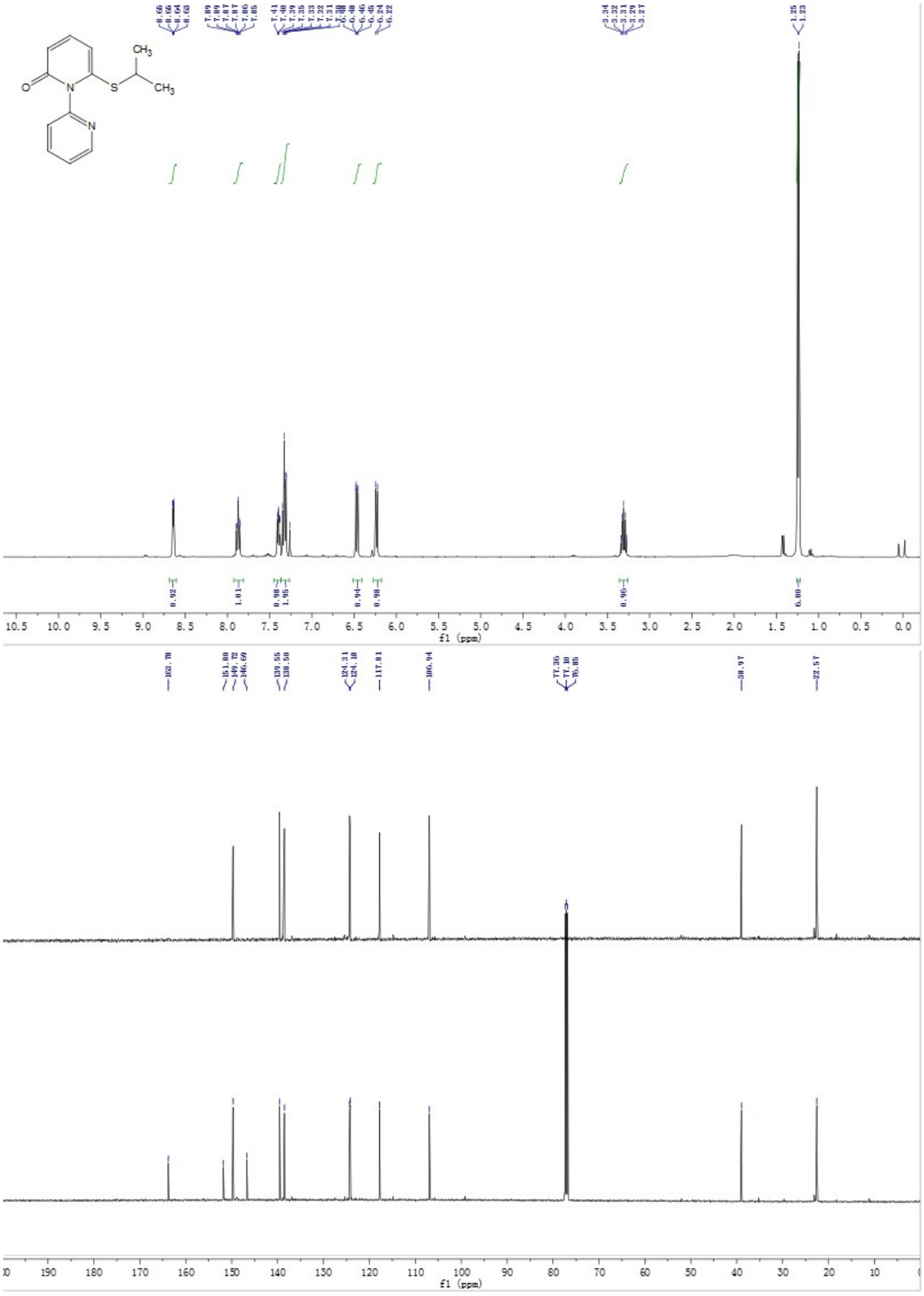


6-(ethylthio)-2*H*-[1,2'-bipyridin]-2-one (4n)

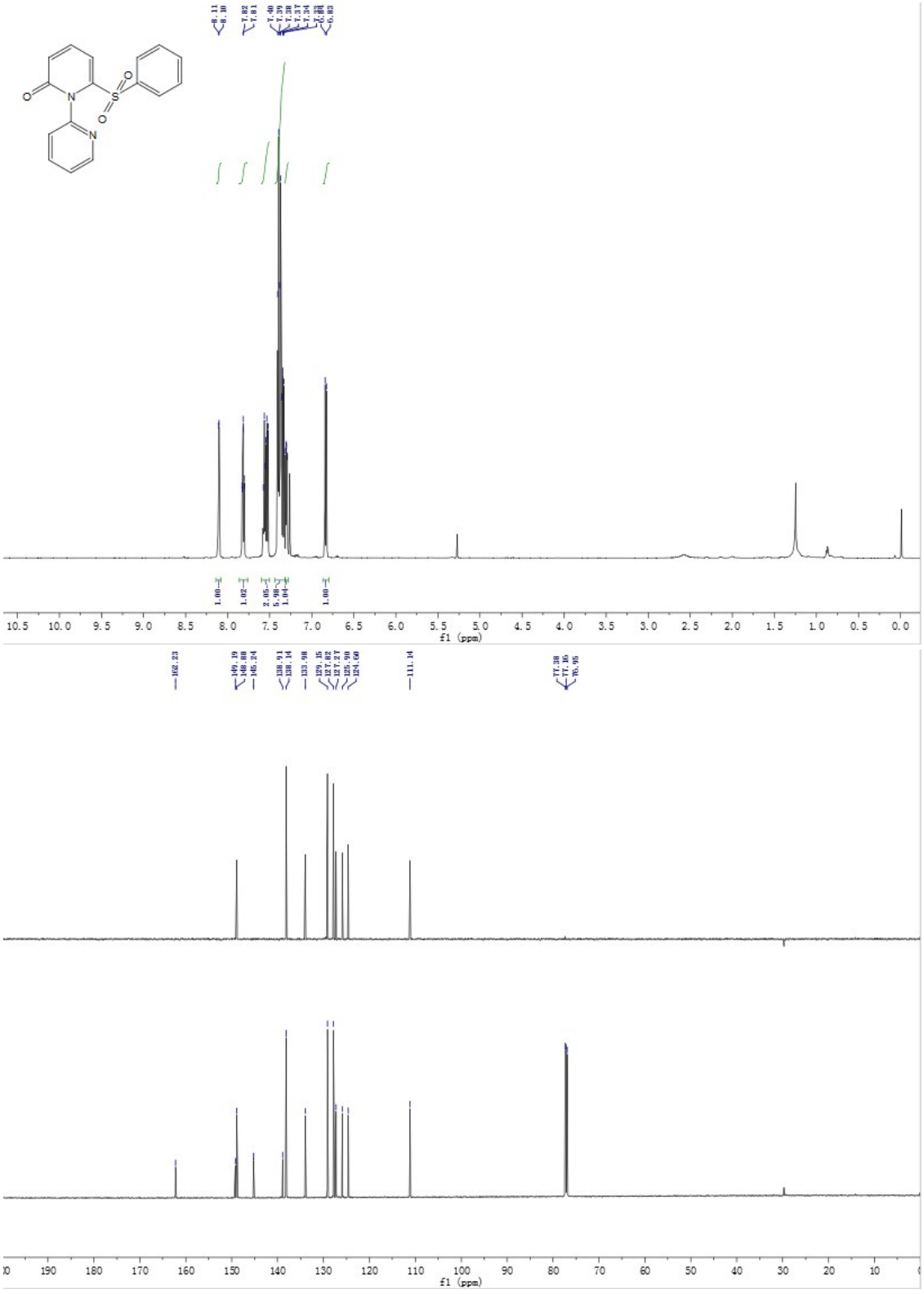




6-(isopropylthio)-2*H*-[1,2'-bipyridin]-2-one (4o)



6-(phenylsulfonyl)-2*H*-[1,2'-bipyridin]-2-one (5a)



6-(phenylthio)pyridin-2(1*H*)-one (6a)