Supporting Information

Access to Functionalized Thienopyridines via a Reagent-Capsule-Assisted Coupling, Thiolation and Cyclization Cascade Sequence

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I. General methods and materials

Unless otherwise noted, all commercial materials and solvents were used without further purification. ^{1}H NMR spectra were recorded in $CDCl_3$ at 400 MHz and ^{13}C NMR spectra were recorded in $CDCl_3$ at 100 MHz respectively, ^{1}H and ^{13}C NMR were referenced to $CDCl_3$ at δ 7.260 and 77.0 respectively. The different types of carbon in the structures have been identified by HSQC, HMBC and DEPT techniques. GC–MS was obtained using electron ionization. HRMS was carried out on a MAT 95XP (Thermo). IR spectra were obtained as potassium bromide pellets or as liquid films between two potassium bromide pellets with a Brucker Vector 22 spectrometer. TLC was performed using commercially prepared 100-400 mesh silica gel plates (GF_{254}), and visualization was effected at 254 nm. All the other chemicals were purchased from Aldrich Chemicals. Commercial reagents were used without further purification.

II. General methods for the preparation of paraffin wax capsules containing potassium ethylxanthate

Paraffin wax (mp. 62-65 ^{o}C) was placed in a beaker (200 mL) and heated to 70-75 ^{o}C. Then molten, the volume of the molten wax was approximately 150 mL. A room temperature glass rod was repeatedly dipped into the molten wax (to a depth of 4-5 cm) 5-7 times to develop a wax coating on the glass rod. The coated rod of wax was allowed to cool to room temperature and the wax coating was removed from the glass rod to provide a hollow paraffin wax cone shell. The shell is charged with EtOC(S)SK (160 mg, 1.0 mmol) respectively. And then 3-4 drops of molten wax were dropped onto the EtOC(S)SK crystal to seal the open end of capsules. Once sealed, the extra wax was trimmed with a razor blade and dipped quickly into the molten wax to secure the seal. [1]

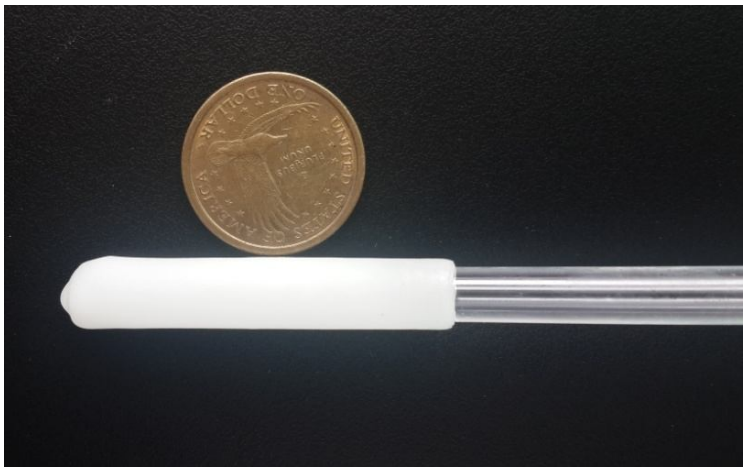


Figure S1. The glass rod for preparing paraffin wax capsules.

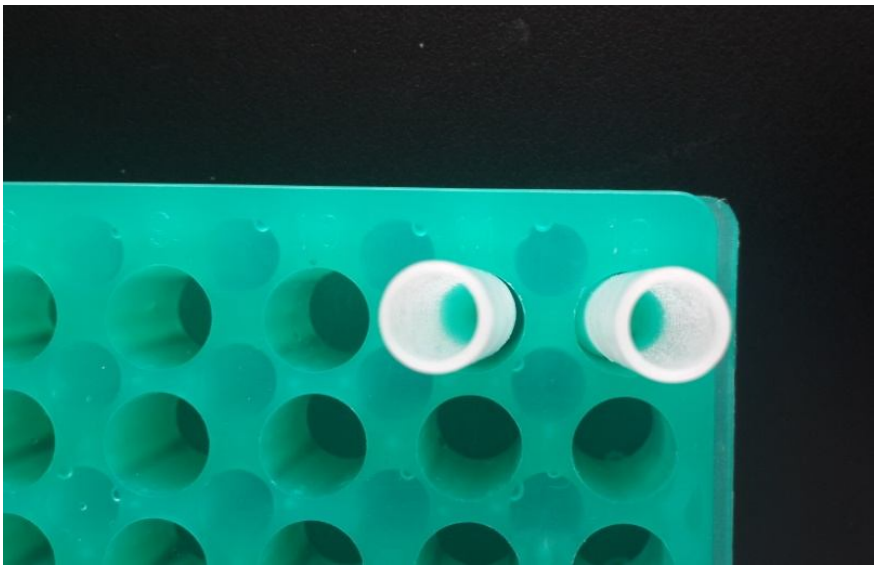
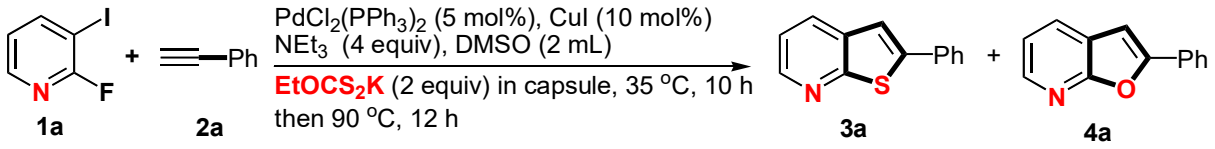


Figure S2. The finished hollow wax shell.

Figure S3. The finished paraffin wax capsule.

III. Optimization of reaction conditions

Table S1. Optimization of reaction conditions[a, b]



Entry	Variations from the standard conditions	Yield(%)	
		3a	**4a**
1	-	88	0
2	$EtOCS_2K$ without in paraffin wax capsule	0	0
3	$(NH_4)_2S$ (2.0 equiv) instead of $EtOCS_2K$ in capsule	0	0
4	$Na_2S·9H_2O$ (2.0 equiv) instead of $EtOCS_2K$ in capsule	23	69
5	thiourea (2.0 equiv) instead of $EtOCS_2K$ in capsule	0	0
6	DMF instead of DMSO	75	0
7	toluene instead of DMSO	< 5	0
8	CH_3CN instead of DMSO	< 5	0
9	NaOH (2.0 equiv) instead of $EtOCS_2K$ in capsule	0	87
10	Cs_2CO_3 (2.0 equiv) instead of Et_3N	25	0
11	Diisopropylamine (4.0 equiv) instead of Et_3N	85	0
12	DBU (4.0 equiv) instead of Et_3N	65	0
13	3-bromo-2-fluoropyridine instead of **1a**	73	0
14	2-bromo-3-iodopyridine instead of **1a**	31	0
15	$Pd(PPh_3)_4$ (10 mmol%)instead of $PdCl_2(PPh_3)_2$	56	0
16	$Pd(OAc)_2$ (5 mmol%)instead of $PdCl_2(PPh_3)_2$	43	0

[a] Reaction conditions: fluoroiodopyridine **1** (1.0 mmol), terminal alkyne **2** (1.3 mmol), $PdCl_2(PPh_3)_2$ (5 mol%), CuI (10 mol%), Et_3N (4.0 mmol), DMSO (2 mL) and paraffin wax capsule (NaOH (2.0 mmol) at 35 °C for 10 h, then 90 °C for 12 h. [b] Yields are given for isolated products. $EtOCS_2K$=Potassium Ethylxanthate.

IV. General methods for the synthesis of furopyridines

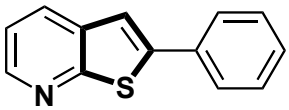
A 25 mL Schlenk tube was charged with $PdCl_2(PPh_3)_2$ (35 mg, 5 mol%), CuI (19 mg, 10 mol%), one paraffin wax capsule of NaOH (80 mg) and a magnetic stirring bar. Then under

nitrogen, DMSO (2 mL) solution of 2-fluoro-3-iodopyridine (1.0 mmol), terminal alkyne (1.3 mmol), Et_3N (400 mg, 4.0 mmol) were injected by syringe. The reaction was performed for 10 h at 35 °C. Then the temperature was elevated to 80 °C for 12 h. After the reaction finished, the reaction mixture was diluted with ethyl acetate and passed through Celite. After evaporation of the solvent the residue was adsorbed on silica gel and the crude product was purified by column chromatography using n-hexane/ethyl acetate (10:1) as eluent.

V General methods for the synthesis of thienopyridine and related heteocycles

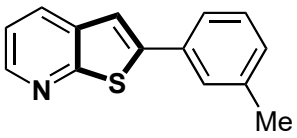
A 25 mL Schlenk tube was charged with $PdCl_2(PPh_3)_2$ (35 mg, 5 mol%), CuI (19 mg, 10 mol%), two paraffin wax capsule of $EtOCS_2K$ (160 mg/capsule) and a magnetic stirring bar. Then under nitrogen, DMSO (2 mL) solution of 2-fluoro-3-iodopyridine (1.0 mmol), terminal alkyne (1.3 mmol), Et_3N (400 mg, 4.0 mmol) were injected by syringe. The reaction was performed for 10 h at 35 °C. Then the temperature was elevated to 90 °C for 12 h. After the reaction finished, the reaction mixture was diluted with ethyl acetate and passed through Celite. After evaporation of the solvent the residue was adsorbed on silica gel and the crude product was purified by column chromatography using n-hexane/ethyl acetate (10:1) as eluent.

VI. Characterization data for all prepared compounds



2-phenylthieno[2,3-*b*]pyridine [2]

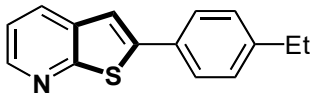
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.46 (d, J = 4.4 Hz, 1H), 7.92 – 7.84 (m, 1H), 7.64 (d, J = 7.2 Hz, 2H), 7.42 – 7.27 (m, 4H), 7.22 – 7.14 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.3, 146.1, 144.2, 134.0, 133.6, 130.4, 128.8 (2C), 128.6, 126.3 (2C), 119.6, 116.5; MS (EI, 70 eV) m/z (%): 211, 166, 139.



2-*m*-tolylthieno[2,3-*b*]pyridine

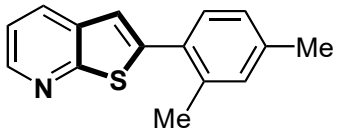
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.54 (d, J = 3.6 Hz, 1H), 8.02 (dd, J = 8.0, 1.6 Hz, 1H), 7.61 – 7.51 (m, 2H), 7.47 (s, 1H), 7.39 – 7.33 (m, 1H), 7.33 – 7.28 (m, 1H), 7.22 (d, J = 7.6 Hz, 1H), 2.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.5, 146.2, 144.7, 138.8, 134.2, 133.7, 130.6,

129.6, 128.9, 127.3, 123.7, 119.7, 116.5; MS (EI, 70 eV) m/z (%): 225, 191, 177, 111, 73; HRMS (EI) calcd for $C_{14}H_{11}NS$ (M^+) 225.0612; found, 225.0606.



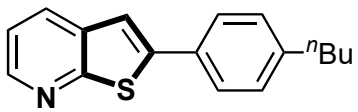
2-(4-ethylphenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.62 – 8.48 (m, 1H), 8.00 (dd, J = 8.0, 1.2 Hz, 1H), 7.67 (d, J = 8.1 Hz, 2H), 7.43 (s, 1H), 7.29 (t, J = 7.0 Hz, 3H), 2.72 (q, J = 7.6 Hz, 2H), 1.30 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 146.1, 145.3, 144.7, 134.3, 131.3, 130.4, 128.5 (2C), 126.5 (2C), 119.7, 116.0, 28.6, 15.4; MS (EI, 70 eV) m/z (%): 239, 225, 166; HRMS (EI) calcd for $C_{15}H_{13}NS$ (M^+) 239.0769; found, 239.0761.



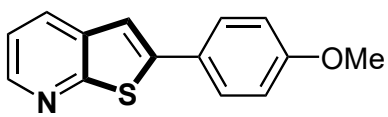
2-(2,4-dimethylphenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.57 (d, J = 4.4 Hz, 1H), 8.04 (dd, J = 8.0, 1.6 Hz, 1H), 7.40 (d, J = 7.6 Hz, 1H), 7.32 (dd, J = 8.0, 4.6 Hz, 1H), 7.17 (s, 1H), 7.16 (s, 1H), 7.11 (d, J = 7.8 Hz, 1H), 2.48 (s, 3H), 2.40 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.9, 145.97, 144.1, 138.6, 136.1, 133.6, 131.6, 130.8, 130.5 (2C), 126.7, 120.0, 119.5, 21.1, 20.9; MS (EI, 70 eV) m/z (%): 239, 224, 116; HRMS (EI) calcd for $C_{15}H_{13}NS$ (M^+) 239.0769; found, 239.0760.



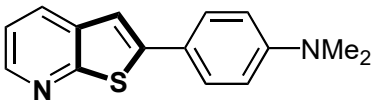
2-(4-butylphenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.52 (d, J = 4.4 Hz, 1H), 7.99 (dd, J = 8.0, 1.2 Hz, 1H), 7.65 (d, J = 8.0 Hz, 2H), 7.42 (s, 1H), 7.28 (t, J = 6.4 Hz, 3H), 2.67 (t, J = 7.6 Hz, 2H), 1.71 – 1.59 (m, 2H), 1.47 – 1.35 (m, 2H), 0.97 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 146.0, 144.7, 143.9, 134.3, 131.2, 130.4, 129.0 (2C), 126.4 (2C), 119.7, 115.9, 35.3, 33.4, 22.3, 13.9; MS (EI, 70 eV) m/z (%): 267, 224, 207; HRMS (EI) calcd for C17H17NS (M^+) 267.1082; found, 267.1077.



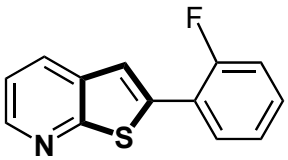
2-(4-methoxyphenyl)thieno[2,3-*b*]pyridine[2]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.50 (dd, J = 4.6, 1.5 Hz, 1H), 7.98 (dd, J = 8.0, 1.5 Hz, 1H), 7.75 – 7.59 (m, 2H), 7.34 (s, 1H), 7.31 – 7.23 (m, 1H), 7.06 – 6.92 (m, 2H), 3.88 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.3, 160.2, 145.9, 144.4, 134.4, 130.2, 127.8 (2C), 126.5, 119.7, 115.3, 114.4 (2C), 7 55.4; MS (EI, 70 eV) m/z (%): 241, 226, 198.



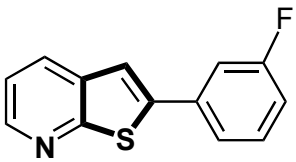
N,N-dimethyl-4-(thieno[2,3-*b*]pyridin-2-yl)benzenamine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.43 (d, J = 4.0 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.60 (d, J = 8.8 Hz, 2H), 7.26 – 7.17 (m, 2H), 6.75 (d, J = 8.8 Hz, 2H), 3.01 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.0, 150.7, 145.2, 134.8, 129.7, 127.5 (4C), 121.9, 119.6, 113.6, 112.3, 40.34 (2C); MS (EI, 70 eV) m/z (%): 254, 238, 210, 126; HRMS (EI) calcd for $C_{15}H_{14}N2S$ (M^+) 254.0878; found, 254.0872.



2-(2-fluorophenyl)thieno[2,3-*b*]pyridine

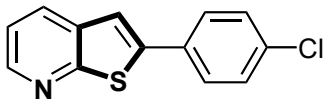
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.53 (d, J = 4.5 Hz, 1H), 8.00 (d, J = 8.0 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.61 (s, 1H), 7.36 – 7.24 (m, 2H), 7.24 – 7.11 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.2 (d, $J_{C–F}$ = 2.9 Hz, 1C), 159.6 (d, $J_{C–F}$ = 250.3 Hz, 1C), 146.6, 137.6 (d, $J_{C–F}$ = 3.5 Hz, 1C), 133.7, 130.9, 129.9 (d, $J_{C–F}$ = 8.6 Hz, 1C), 129.5(d, $J_{C–F}$ = 2.9 Hz, 1C), 124.6 (d, $J_{C–F}$ = 3.6 Hz, 1C), 121.6 (d, $J_{C–F}$ = 11.8 Hz, 1C), 120.7 (d, $J_{C–F}$ = 9.3 Hz, 1C), 119.7, 116.5, 116.3; MS (EI, 70 eV) m/z (%): 229, 210, 184, 114; HRMS (EI) calcd for $C_{13}H_8FNS$ (M^+) 229.0361; found, 229.0354.



2-(3-fluorophenyl)thieno[2,3-*b*]pyridine [2]

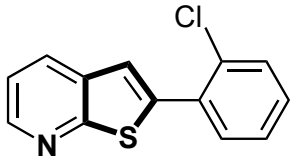
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.54 (d, J = 4.2 Hz, 1H), 8.02 (d, J = 8.0 Hz, 1H), 7.54 – 7.38 (m, 4H), 7.30 (dd, J = 7.2, 5.3 Hz, 1H), 7.08 (t, J = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz)

δ 163.0 (d, J_{C-F} = 245.2 Hz, 1C), 161.4, 146.6, 142.9, 135.9 (d, J_{C-F} = 8.1 Hz, 1C), 133.9, 130.9, 130.6 (d, J_{C-F} = 8.4 Hz, 1C), 122.2 (d, J_{C-F} = 2.8 Hz, 1C), 119.9, 117.5, 115.6 (d, J_{C-F} = 22.1 Hz, 1C), 113.2 (d, J_{C-F} = 22.8 Hz, 1C); MS (EI, 70 eV) m/z (%): 229, 184, 157, 114.



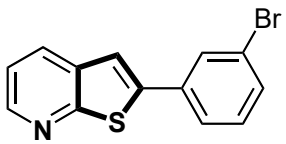
2-(4-chlorophenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.55 (d, J = 4.4 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 9.4 Hz, 3H), 7.36 – 7.28 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.5, 146.6, 143.1, 134.7, 134.1, 132.3, 130.8, 129.2 (2C), 127.7 (2C), 119.9, 117.1; MS (EI, 70 eV) m/z (%): 245, 210, 166; HRMS (EI) calcd for $C_{13}H_8ClNS$ (M^+) 245.0066; found, 245.0061.



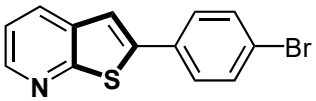
2-(2-chlorophenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.56 (s, 1H), 8.02 (dd, J = 8.0, 1.2 Hz, 1H), 7.61 – 7.53 (m, 1H), 7.52 – 7.45 (m, 2H), 7.26-7.33 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.9, 146.5, 140.6, 132.7, 132.7, 133.2, 131.8, 131.0, 130.6, 129.6, 127.0, 121.8, 119.7; MS (EI, 70 eV) m/z (%): 245, 210, 166; HRMS (EI) calcd for $C_{13}H_8ClNS$ (M^+) 245.0066; found, 245.0064.



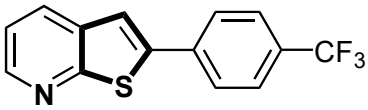
2-(3-bromophenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.44 (d, J = 4.6 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.76 (s, 1H), 7.53 (d, J = 7.6 Hz, 1H), 7.40 (d, J = 8.0 Hz, 1H), 7.35 (s, 1H), 7.26 – 7.15 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.5, 146.7, 142.6, 135.8, 133.9, 131.6, 130.9, 130.5, 129.3, 125.2, 123.1, 119.9, 117.6; MS (EI, 70 eV) m/z (%): 291, 289, 211, 209, 166, 139; HRMS (EI) calcd for $C_{13}H_8BrNS$ (M^+) 288.9561; found, 288.9556.



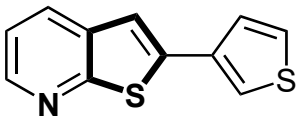
2-(4-bromophenyl)thieno[2,3-*b*]pyridine[2]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.55 (d, J = 4.6 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.59 (s, 4H), 7.46 (s, 1H), 7.37 – 7.28 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 146.6, 143.1, 134.1, 132.8, 132.2 (2C), 130.8, 128.0 (2C), 122.9, 119.9, 117.1; MS (EI, 70 eV) m/z (%): 291, 289, 245, 166.



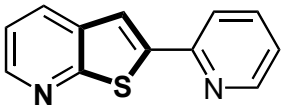
2-(4-(trifluoromethyl)phenyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.56 (d, J = 4.0 Hz, 1H), 8.05 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 8.0 Hz, 2H), 7.69 (d, J = 8.0 Hz, 2H), 7.54 (s, 1H), 7.32 (dd, J = 7.6, 4.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.7, 147.0, 142.6, 137.3, 134.0, 131.1, 126.7 (4C), 126.0 (q, J = 3.7 Hz, 1C), 120.1, 118.3; MS (EI, 70 eV) m/z (%): 279, 278, 260, 257, 239; HRMS (EI) calcd for $C_{14}H_8F_3NS$ (M^+) 279.0330; found, 279.0324.



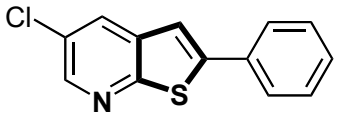
2-(thiophen-3-yl)thieno[2,3-*b*]pyridine[2]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.50 (d, J = 4.4 Hz, 1H), 7.97 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 0.9 Hz, 1H), 7.42 (s, 2H), 7.32 (s, 1H), 7.26 (t, J = 4.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.0, 146.1, 139.2, 135.3, 134.1, 130.5, 126.9, 125.8, 122.2, 119.8, 116.4; MS (EI, 70 eV) m/z (%): 217, 172, 145, 108.



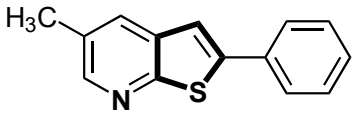
2-(pyridin-2-yl)thieno[2,3-*b*]pyridine[2]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.64 (4.0 Hz, 1H), 8.55 (4.0 Hz, 1H), 8.02 (d, J = 8.0 Hz, 1H), 7.86 – 7.67 (m, 3H), 7.28-7.21(m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.3, 151.9, 149.7, 147.1, 144.9, 136.6, 133.9, 131.2, 123.1, 119.7, 119.6, 118.3; MS (EI, 70 eV) m/z (%): 212, 184, 168.



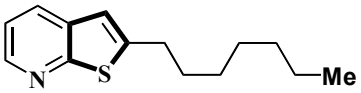
5-chloro-2-phenylthieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.46 (s, 1H), 7.98 (s, 1H), 7.70 (d, J = 7.6 Hz, 2H), 7.45 (t, J = 7.4 Hz, 2H), 7.40 (d, J = 11.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 147.0, 145.1, 135.0, 133.5, 129.7, 129.2, 129.1 (2C), 126.6 (2C), 125.6, 115.8; MS (EI, 70 eV) m/z (%): 247, 245 (100%), 177, 166, 139; HRMS (EI) calcd for $C_{13}H_8ClNS$ (M^+) 245.0066; found, 245.0061.



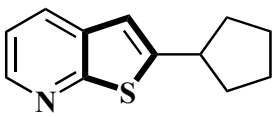
5-methyl-2-phenylthieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.34 (s, 1H), 7.76 (s, 1H), 7.69 (d, J = 7.6 Hz, 2H), 7.48 – 7.29 (m, 4H), 7.25 (s, 1H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.7, 147.5, 144.5, 134.1, 133.9, 130.7, 129.3, 128.9 (2C), 128.6, 126.4 (2C), 116.3, 18.4; MS (EI, 70 eV) m/z (%): 225, 191, 176, 133; HRMS (EI) calcd for $C_{14}H_{11}NS$ (M^+) 225.0612; found, 225.0605.



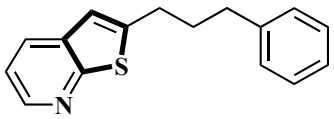
2-heptylthieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.45 (dd, J = 4.6, 1.5 Hz, 1H), 7.88 (dd, J = 8.0, 1.5 Hz, 1H), 7.22 (dd, J = 8.0, 4.7 Hz, 1H), 6.91 (s, 1H), 2.90 (t, J = 7.6 Hz, 2H), 1.75 (dt, J = 15.2, 7.6 Hz, 2H), 1.41-1.24 (m, 8H), 0.88 (t, J = 5.2 Hz, 3H).; ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.5, 147.5, 145.4, 133.6, 129.7, 119.3, 117.7, 31.7, 31.3, 30.9, 29.0, 28.97, 22.6, 14.0; MS (EI, 70 eV) m/z (%): 233, 200, 162, 149, 148; HRMS (EI) calcd for $C_{14}H_{19}NS$ (M^+) 233.1238; found, 233.1231.



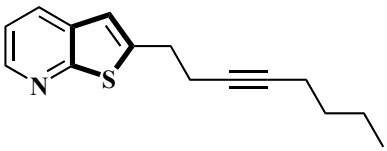
2-cyclopentylthieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.44 (d, J = 4.0 Hz, 1H), 7.88 (d, J = 7.9 Hz, 1H), 7.24 – 7.13 (m, 1H), 6.93 (s, 1H), 3.32 (dd, J = 15.3, 7.8 Hz, 1H), 2.31 – 2.09 (m, 2H), 1.88 – 1.65 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.3, 152.3, 145.4, 133.5, 129.7, 119.3, 116.2, 42.1, 34.8, 25.2; MS (EI, 70 eV) m/z (%): 203, 189, 174, 161, 148; HRMS (EI) calcd for $C_{12}H_{13}NS$ (M^+) 203.0769; found, 203.0762.



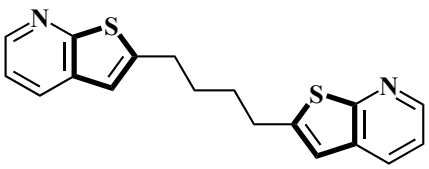
2-(3-phenylpropyl)thieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.45 (d, J = 4.0 Hz, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.29 (t, J = 7.4 Hz, 2H), 7.23-7.08 (m, 4H), 6.91 (s, 1H), 2.93 (t, J = 7.6 Hz, 2H), 2.71 (t, J = 7.6 Hz, 2H), 2.16 – 2.01 (m, 2H).; ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.38, 146.79, 145.39, 141.44, 133.55, 129.80, 128.41, 128.36, 125.92, 119.33, 118.04, 77.32, 77.00, 76.68, 35.02, 32.27, 30.61; MS (EI, 70 eV) m/z (%): 253, 239, 218, 204, 161, 129, 91; HRMS (EI) calcd for $C_{16}H_{15}NS$ (M^+) 253.0925; found, 253.0919.



2-(oct-3-ynyl)thieno[2,3-*b*]pyridine

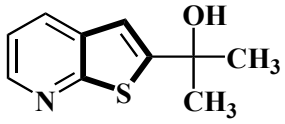
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.44 (dd, J = 4.6, 1.3 Hz, 1H), 7.88 (dd, J = 8.0, 1.4 Hz, 1H), 7.20 (dd, J = 8.0, 4.7 Hz, 1H), 6.97 (s, 1H), 3.07 (t, J = 7.2 Hz, 2H), 2.57 (tt, J = 7.2, 2.3 Hz, 2H), 2.13 (tt, J = 7.0, 2.3 Hz, 2H), 1.50 – 1.39 (m, 2H), 1.34 (dd, J = 15.0, 7.2 Hz, 2H), 0.85 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.5, 145.5, 145.1, 133.3, 129.9, 119.3, 118.5, 82.0, 78.3, 31.1, 30.9, 21.8, 20.5, 18.3, 13.5.; MS (EI, 70 eV) m/z (%): 243, 201, 186, 148; HRMS (EI) calcd for $C_{15}H_{17}NS$ (M^+) 243.1082; found, 243.1078.



2-(4-(thieno[2,3-*b*]pyridin-2-yl)butyl)thieno[2,3-*b*]pyridine

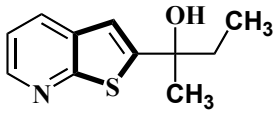
^{1}H NMR (400 MHz, DMSO-d6) δ 8.48 (dd, J = 4.4, 1.6 Hz, 2H), 8.13 (dd, J = 8.0, 1.6 Hz, 2H), 7.40 (dd, J = 8.0, 4.6 Hz, 2H), 7.19 (s, 2H), 3.01 (t, J = 6.6 Hz, 4H), 1.88 – 1.74 (m, 4H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4 (2C), 146.5 (2C), 145.5 (2C), 133.6 (2C), 129.8 (2C), 119.4 (2C), 118.1 (2C), 30.9 (2C), 30.1 (2C).



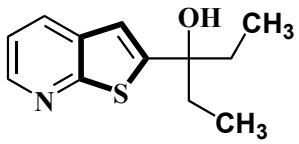
2-(thieno[2,3-*b*]pyridin-2-yl)propan-2-ol

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.50 (s, 1H), 8.02 – 7.86 (m, 1H), 7.27 (dd, J = 7.6, 4.4 Hz, 1H), 7.12 (d, J = 3.3 Hz, 1H), 2.76 (s, 1H), 1.75 (s, 3H), 1.75 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.1, 155.9, 145.7, 133.4, 130.6, 119.4, 115.5, 71.5, 31.9 (2C); MS (EI, 70 eV) m/z (%): 193, 178, 160, 136, 116; HRMS (EI) calcd for $C_{10}H_{11}NOS$ (M^+) 193.0561; found, 193.0557.



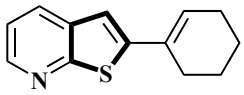
2-(thieno[2,3-*b*]pyridin-2-yl)butan-2-ol

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.34 (d, J = 3.8 Hz, 1H), 7.81 (d, J = 7.6 Hz, 1H), 7.12 (dd, J = 7.2, 5.4 Hz, 1H), 6.97 (s, 1H), 3.11 (s, 1H), 1.84 (q, J = 7.6 Hz, 2H), 1.57 (s, 3H), 0.83 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.1, 154.8, 145.5, 133.4, 130.5, 119.3, 116.1, 74.2, 37.0, 29.4, 8.3; MS (EI, 70 eV) m/z (%): 207, 189, 178, 174, 160, 136; HRMS (EI) calcd for $C_{11}H_{13}NOS$ (M^+) 207.0718; found, 207.0711.



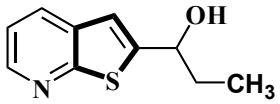
3-(thieno[2,3-*b*]pyridin-2-yl)pentan-3-ol

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.45 (d, J = 3.1 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.24-7.21 (m, 1H), 7.05 (s, 1H), 2.74 (s, 1H), 1.91 (q, J = 7.6 Hz, 4H), 0.89 (t, J = 7.6 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 153.1, 145.5, 133.5, 130.4, 119.3, 116.9, 77.1, 35.1 (2C), 7.8 (2C); MS (EI, 70 eV) m/z (%): 221, 207, 204, 192, 174, 136; HRMS (EI) calcd for $C_{12}H_{15}NOS$ (M^+) 221.0874; found, 221.0867.



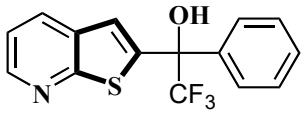
2-cyclohexenylthieno[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.42 (d, J = 4.6 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.18-7.15 (m, 1H), 6.97 (s, 1H), 6.33 (s, 1H), 2.45 (s, 2H), 2.23 (d, J = 2.0 Hz, 2H), 1.81-1.75 (m, 2H), 1.69-1.65 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.6, 146.9, 145.8, 134.0, 131.3, 123.0, 128.6, 119.4, 114.8, 26.5, 25.8, 22.4, 21.9; MS (EI, 70 eV) m/z (%): 215, 200, 186, 148; HRMS (EI) calcd for $C_{13}H_{13}NS$ (M^+) 215.0769; found, 215.0762.



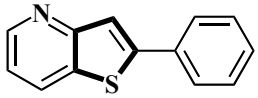
1-(thieno[2,3-*b*]pyridin-2-yl)propan-1-ol

^{1}H NMR (400 MHz, DMSO-d6) δ 8.51 (dd, J = 4.4, 1.2 Hz, 1H), 8.18 (d, J = 8.0 Hz, 1H), 7.41 (dd, J = 8.0, 4.6 Hz, 1H), 7.27 (s, 1H), 5.84 (s, 1H), 4.84 (t, J = 6.0 Hz, 1H), 1.81 (p, J = 7.2 Hz, 2H), 0.95 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.3, 150.1, 146.0, 133.1, 130.7, 119.5, 117.3, 72.3, 31.9, 9.9; MS (EI, 70 eV) m/z (%): 193, 189, 174, 164, 136; HRMS (EI) calcd for $C_{10}H_{11}NOS$ (M^+) 193.0561; found, 193.0557.



2,2,2-trifluoro-1-phenyl-1-(thieno[2,3-*b*]pyridin-2-yl)ethanol

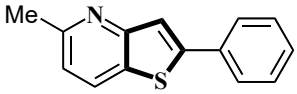
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.45 (d, J = 4.0 Hz, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.66 (s, 2H), 7.4—7.25 (m, 3H), 7.28-7.25 (m, 1H), 5.28 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.1, 146.8, 144.9, 137.4, 132.6, 131.8, 129.2, 128.9 (q, J = 3.0 Hz, 1C), 128.3 (2C), 127.0 (2C), 120.8, 119.8, 29.7; MS (EI, 70 eV) m/z (%): 309, 240, 162, 105, 77; HRMS (EI) calcd for $C_{15}H_{10}F_3NOS$ (M^+) 309.0435; found, 309.0428.



2-phenylthieno[3,2-*b*]pyridine[2]

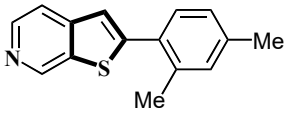
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.63 (d, J = 4.0 Hz, 1H), 8.05 (d, J = 8.0 Hz, 1H), 7.78 – 7.66 (m, 3H), 7.38 (dt, J = 24.5, 7.2 Hz, 3H), 7.14 (dd, J = 7.9, 4.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100

MHz) δ 156.7, 148.2, 147.3, 133.5, 133.3, 129.9, 129.0, 128.9 (2C), 126.3 (2C), 120.4, 118.5; MS (EI, 70 eV) m/z (%): 211, 167, 105.



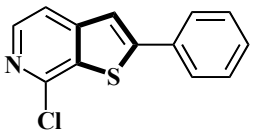
5-methyl-2-phenylthieno[3,2-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.90 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 6.8 Hz, 3H), 7.39 (t, J = 7.2 Hz, 2H), 7.33 (d, J = 7.2 Hz, 1H), 7.01 (d, J = 8.0 Hz, 1H), 2.64 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.3, 156.0, 147.9, 133.6, 130.4, 129.9, 128.9 (2C), 128.8, 126.2 (2C), 120.1, 118.8, 24.4; MS (EI, 70 eV) m/z (%): 225, 197, 139, 112; HRMS (EI) calcd for $C_{14}H_{11}NS$ (M^+) 225.0612; found, 225.0607.



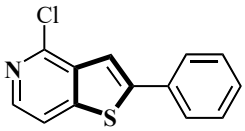
2-(2,4-dimethylphenyl)thieno[2,3-*c*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.09 (s, 1H), 8.49 (d, J = 5.4 Hz, 1H), 7.62 (d, J = 5.4 Hz, 1H), 7.35 (d, J = 7.6 Hz, 1H), 7.22 (s, 1H), 7.12 (s, 1H), 7.07 (d, J = 7.6 Hz, 1H), 2.42 (s, 3H), 2.37 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.7, 145.2, 143.9, 143.1, 138.93, 136.4, 136.0, 131.6, 130.3, 130.1, 126.7, 121.5, 117.3, 21.0, 20.8; MS (EI, 70 eV) m/z (%): 239; 224, 116; HRMS (EI) calcd for $C_{15}H_{13}NS$ (M^+) 239.0769; found, 239.0762.



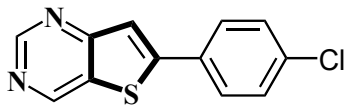
7-chloro-2-phenylthieno[2,3-*c*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.21 (d, J = 5.2 Hz, 1H), 7.70 – 7.59 (m, 2H), 7.46 (d, J = 7.8 Hz, 2H), 7.43 – 7.35 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.1, 147.7, 144.4, 143.2, 134.6, 132.6, 129.6, 129.0 (2C), 126.7 (2C), 118.7, 116.6; MS (EI, 70 eV) m/z (%): 245, 210, 177, 139; HRMS (EI) calcd for $C_{13}H_8ClNS$ (M^+) 245.0066; found, 245.0061.



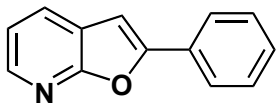
4-chloro-2-phenylthieno[3,2-*c*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.53 (d, J = 4.4 Hz, 1H), 8.01 (d, J = 8.0 Hz, 1H), 7.57 (s, 4H), 7.44 (s, 1H), 7.31-7.28 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 146.6, 143.1, 134.1, 132.8, 132.2 (2C), 130.8, 128.0 (2C), 122.9, 119.9, 117.1; MS (EI, 70 eV) m/z (%): 245, 210, 177, 139; HRMS (EI) calcd for $C_{13}H_8ClNS$ (M^+) 245.0066; found, 245.0063.



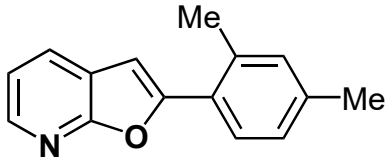
6-(4-chlorophenyl)thieno[3,2-*d*]pyrimidine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.20 (d, J = 7.6 Hz, 2H), 7.71 (d, J = 6.0 Hz, 3H), 7.47 (d, J = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.4, 154.8, 150.9, 136.5, 131.2, 129.9, 129.6 (2C), 128.7, 128.2 (2C), 119.9; MS (EI, 70 eV) m/z (%): 248, 246, 192, 157, 113; HRMS (EI) calcd for $C_{12}H_7ClN_2S$ (M^+) 246.0018; found, 246.0012.



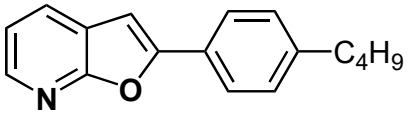
2-phenylfuro[2,3-*b*]pyridine[3]

^{1}H NMR ($CDCl_3$, 400 MHz) δ. 8.28 (dd, J = 4.9, 1.6 Hz, 1H), 7.95 – 7.83 (m, 3H), 7.45 (t, J = 7.6 Hz, 2H), 7.38 (t, J = 7.4 Hz, 1H), 7.20 (dd, J = 7.6, 4.9 Hz, 1H), 6.98 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.8, 155.5, 143.6, 129.5, 129.5, 129.2, 128.8 (2C), 125.1 (2C), 121.4, 119.5, 100.0; MS (EI, 70 eV) m/z (%): 195 (100%), 166, 139.



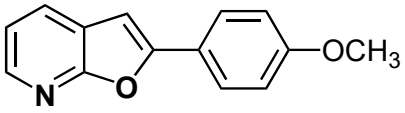
2-(2,4-dimethylphenyl)furo[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.28 (d, J = 2.8 Hz, 1H), 7.86 (d, J = 7.6 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.18 (dd, J = 6.8, 4.4 Hz, 1H), 7.10 (d, J = 11.6 Hz, 2H), 6.79 (d, J = 2.0 Hz, 1H), 2.54 (s, 3H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.3, 155.6, 143.5, 139.0, 135.8, 132.0, 129.2, 128.2, 126.8, 126.1, 121.3, 119.2, 103.1, 21.8, 21.1; MS (EI, 70 eV) m/z (%): 223 (100%), 208, 194, 180.



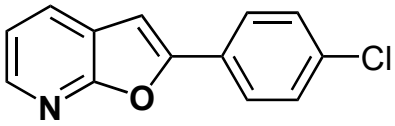
2-(4-butylphenyl)furo[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.29 (d, J = 4.8 Hz, 1H), 7.86 (dd, J = 18.4, 7.6 Hz, 3H), 7.29 (d, J = 7.6 Hz, 2H), 7.21 (dd, J = 7.2, 5.2 Hz, 1H), 6.96 (s, 1H), 2.68 (t, J = 7.7 Hz, 2H), 1.73 – 1.58 (m, 2H), 1.52 – 1.34 (m, 2H), 0.96 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.8, 155.9, 144.5, 143.4, 129.2, 128.9 (2C), 127.0, 125.1 (2C), 121.6, 119.4, 99.2, 35.49, 33.4, 22.3, 13.9; MS (EI, 70 eV) m/z (%): 251, 208 (100%.)



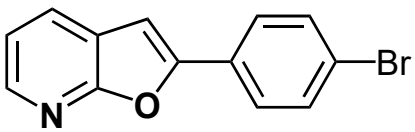
2-(4-methoxyphenyl)furo[2,3-*b*]pyridine[3]

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.24 (dd, J = 4.8, 1.6 Hz, 1H), 7.84-7.81 (m, 3H), 7.18 (dd, J = 7.6, 4.9 Hz, 1H), 7.02 – 6.95 (m, 2H), 6.84 (d, J = 3.7 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.8, 160.5, 155.7, 143.1, 129.0, 126.7 (2C), 122.3, 121.7, 119.4, 114.3 (2C), 98.3, 55.3; MS (EI, 70 eV) m/z (%): 225 (100%), 210, 182.



2-(4-chlorophenyl)furo[2,3-*b*]pyridine

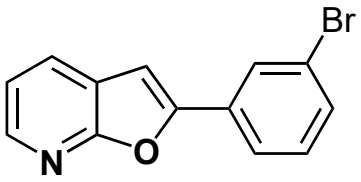
^{1}H NMR ($CDCl_3$, 400 MHz) δ. 8.27 (d, J = 4.0 Hz, 1H), 7.85 (d, J = 7.6 Hz, 1H), 7.77 (d, J = 7.6 Hz, 2H), 7.38 (d, J = 7.6 Hz, 2H), 7.23 – 7.13 (m, 1H), 6.93 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.7, 154.3, 144.0, 135.0, 129.6, 129.0 (2C), 127.9, 126.2 (2C), 121.2, 119.6, 100.4; MS (EI, 70 eV) m/z (%): 231, 229 (100%), 166, 139.



2-(4-bromophenyl)furo[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.30 (dd, J = 4.8, 1.4 Hz, 1H), 7.90 (dd, J = 7.6, 1.6 Hz, 1H), 7.80 – 7.72 (m, 2H), 7.59 (d, J = 8.6 Hz, 2H), 7.22 (dd, J = 7.6, 4.9 Hz, 1H), 7.00 (s, 1H); ^{13}C

NMR ($CDCl_3$, 100 MHz) δ 161.8, 154.5, 144.2, 132.1 (2C), 129.7, 128.5, 126.6 (2C), 123.4, 121.3, 119.7, 100.5; MS (EI, 70 eV) m/z (%): 275 (100%), 273, 166, 139.



2-(3-bromophenyl)furo[2,3-*b*]pyridine

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.31 (d, J = 3.6 Hz, 1H), 8.01 (s, 1H), 7.88 (d, J = 7.6 Hz, 1H), 7.79 (d, J = 7.8 Hz, 1H), 7.48 (d, J = 8.0 Hz, 1H), 7.30 (t, J = 8.0 Hz, 1H), 7.24 – 7.17 (m, 1H), 6.98 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.7, 153.7, 144.3, 132.0, 131.4, 130.3, 129.8, 127.9, 123.5, 122.9, 121.0, 119.6, 101.0; MS (EI, 70 eV) m/z (%): 275 (100%), 273, 166, 139.

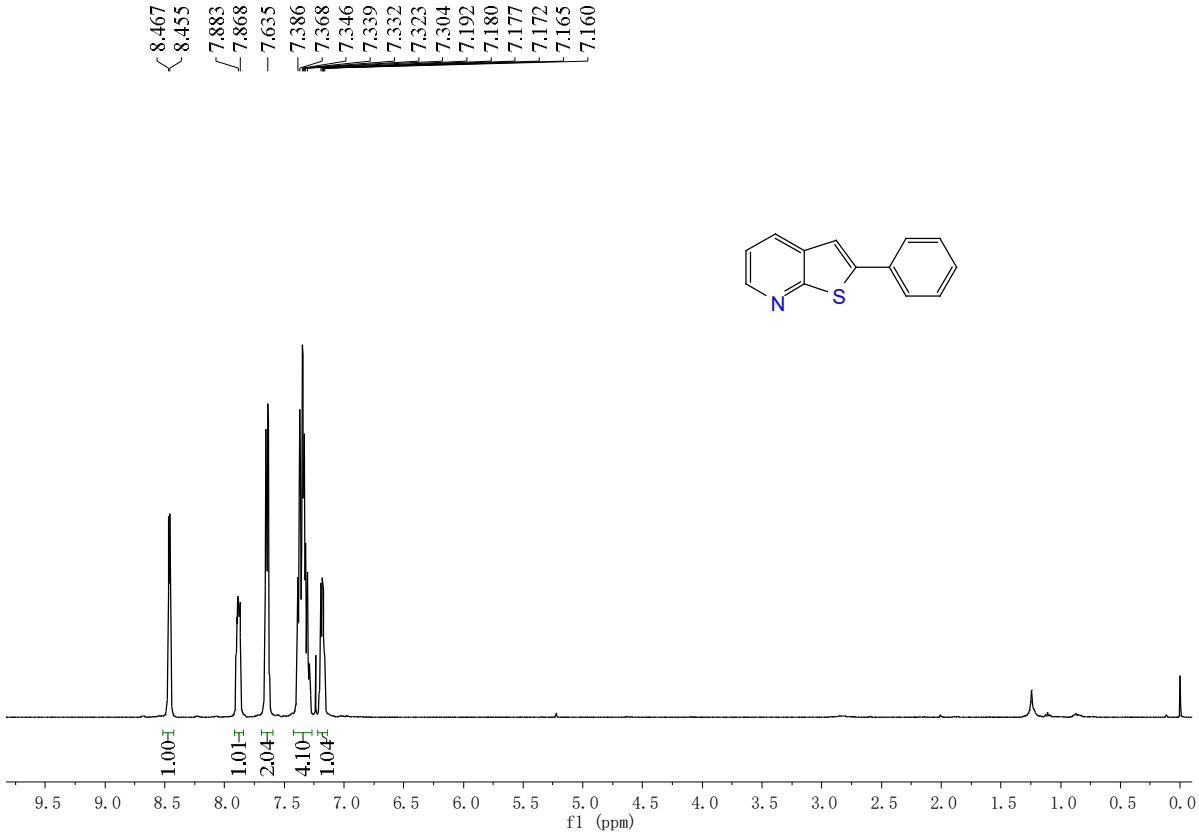
References:

(1) Shen, C.; Spannenberg, A.; Wu, X. F. *Angew. Chem. Int. Ed*., 2016, **128**, 5151-5154.

(2) Peixoto, D.; Begouin, A.; Queiroz, M. J. B. P. *Tetrahedron,* 2012, **68**, 7082-7094.

(3) Hudson, R.; Bizier, N. P.; Esdale, K. N.; Katz, J. L. *Org. Biomol. Chem*., 2015, **13**, 2273-2284.

V. NMR Data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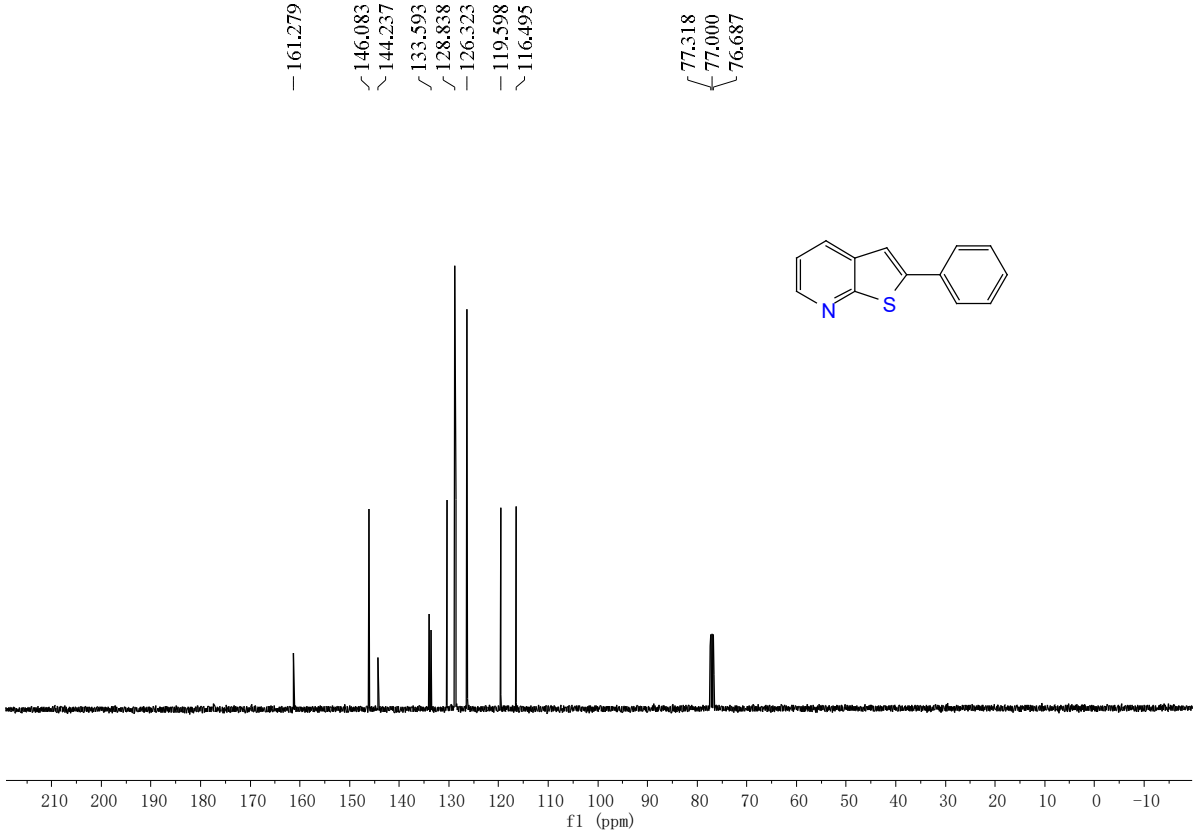
8.455
7.883
7.868
7.635
7.386
7.368
7.346
7.339
7.332
7.323
7.304
7.192
7.180
7.177
7.172
7.165
7.160
N
S
1.00
1.01
2.04
4.10
1.04
f1 (ppm)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146.083
144.237
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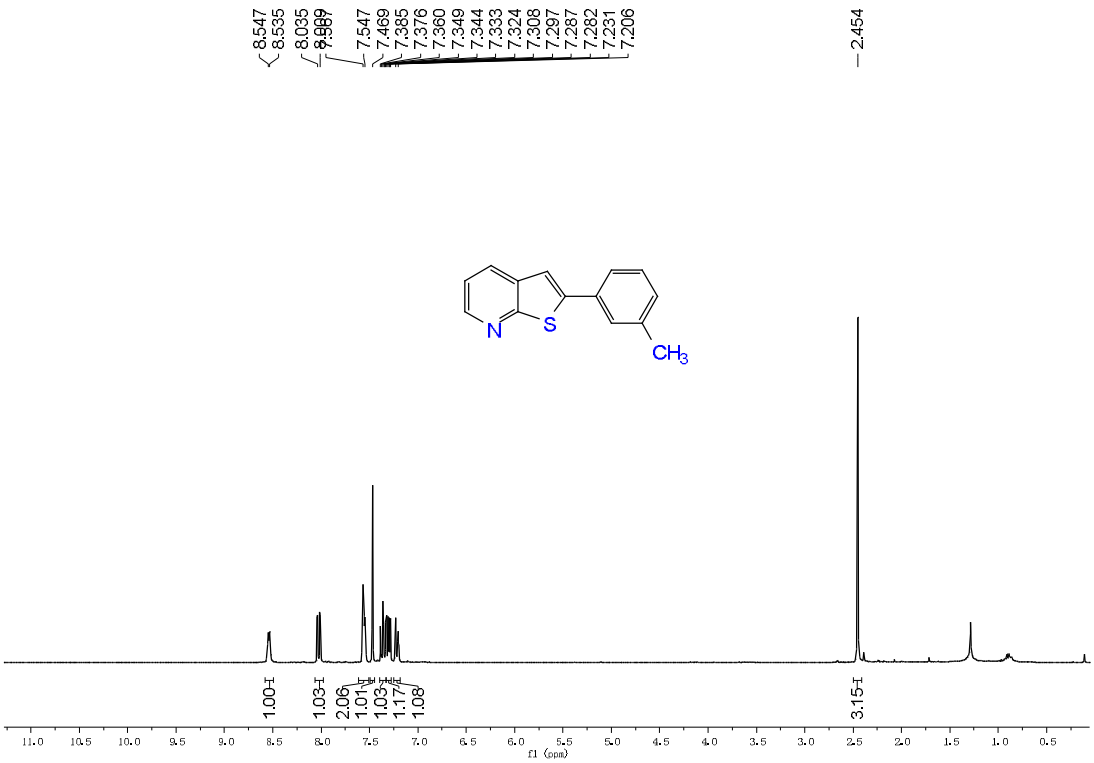
128.838
126.323
119.598
116.495
77.318
77.000
76.687
N
S
f1 (ppm)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8.535
8.035
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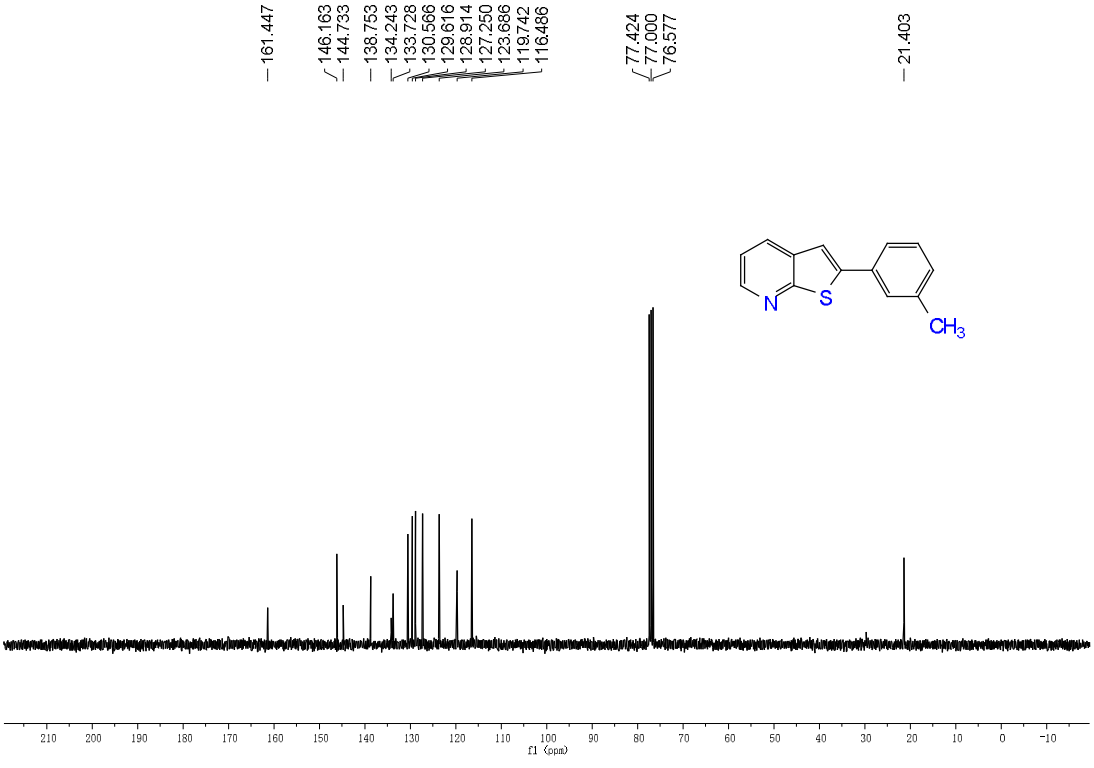
7.469
7.385
7.376
7.360
7.349
7.344
7.333
7.324
7.308
7.297
7.287
7.282
7.231
7.206
2.454
N
S
CH3
1.00
1.03
2.06
1.01
1.03
1.17
1.08
3.15
f1 (ppm)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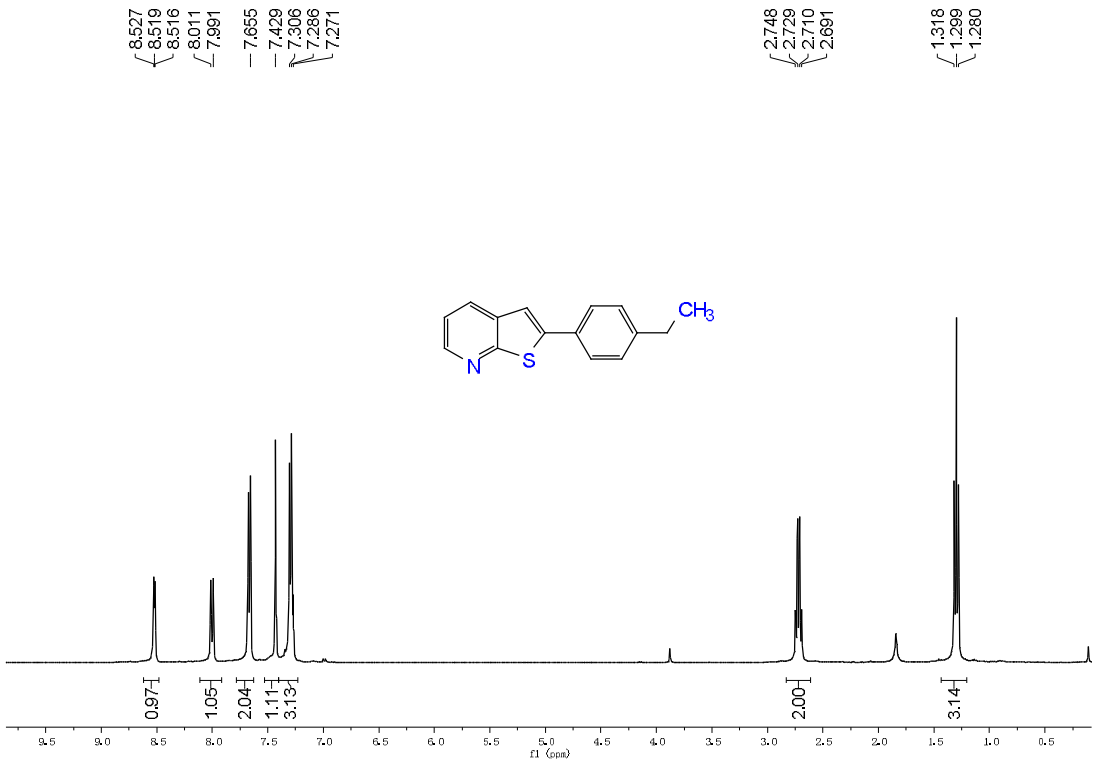
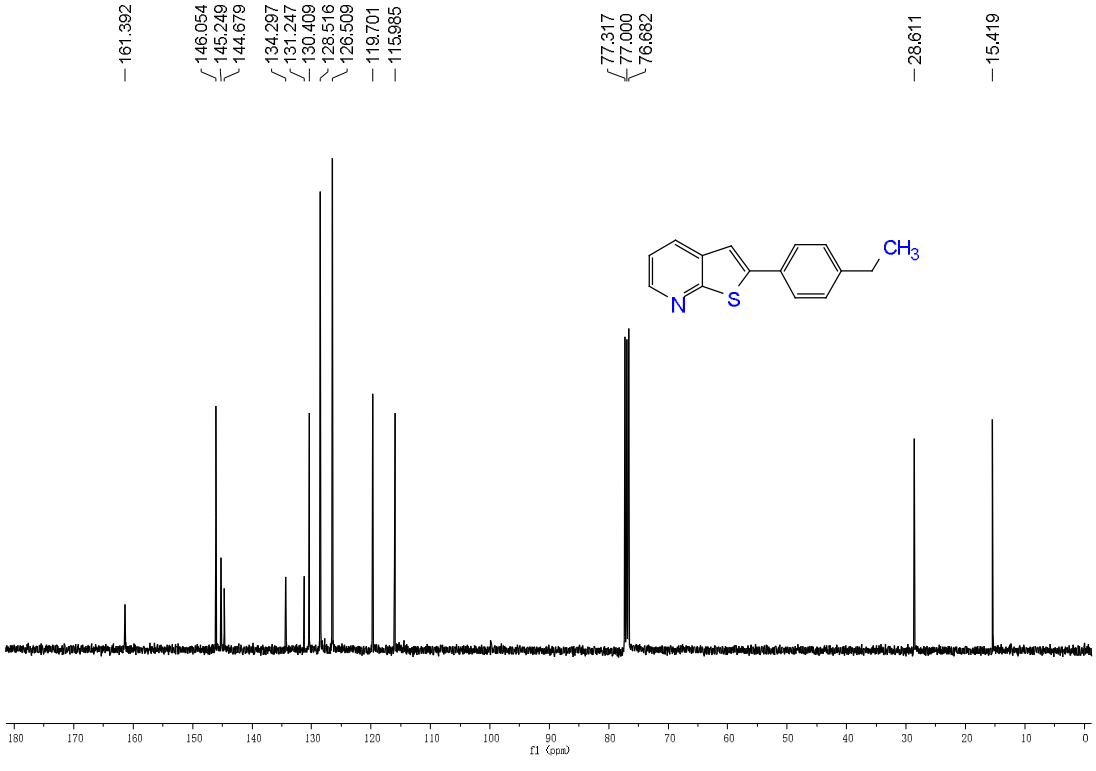
146.163
144.733
138.753
134.243
133.728
130.566
129.616
128.914
127.250
123.686
119.742
116.486
77.424
77.000
76.577
21.403
N
S
CH3
f1 (ppm)


8.527
8.519
8.516
8.011
7.991
7.655
7.429
7.306
7.286
7.271
2.748
2.729
2.710
2.691
1.318
1.299
1.280
CH3
N
S
0.97
1.05
2.04
1.11
3.13
2.00
3.14
f1 (ppm)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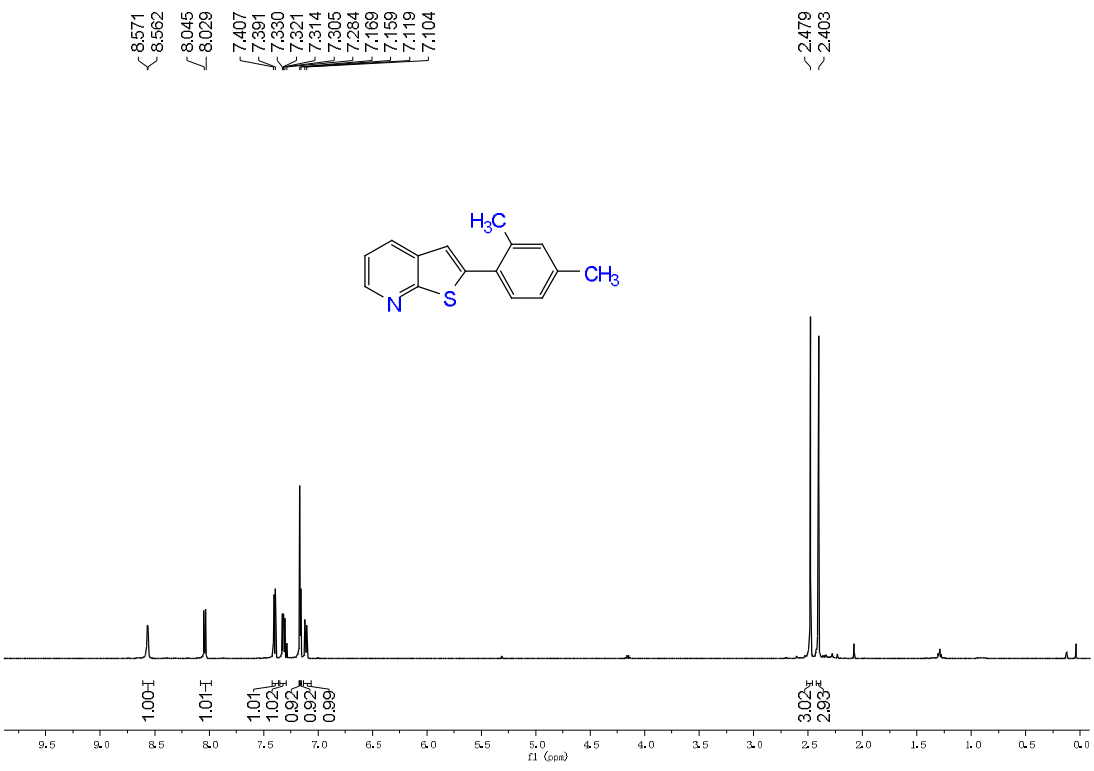
145.249
144.679
134.297
131.247
130.409
128.516
126.509
119.701
115.985
77.317
77.000
76.682
28.611
15.419
CH3
N
S
f1 (ppm)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8.045
8.029
7.407
7.391
7.330
7.321
7.314
7.305
7.284
7.169
7.159
7.119
7.104
2.479
2.403
H3C
CH3
N
S
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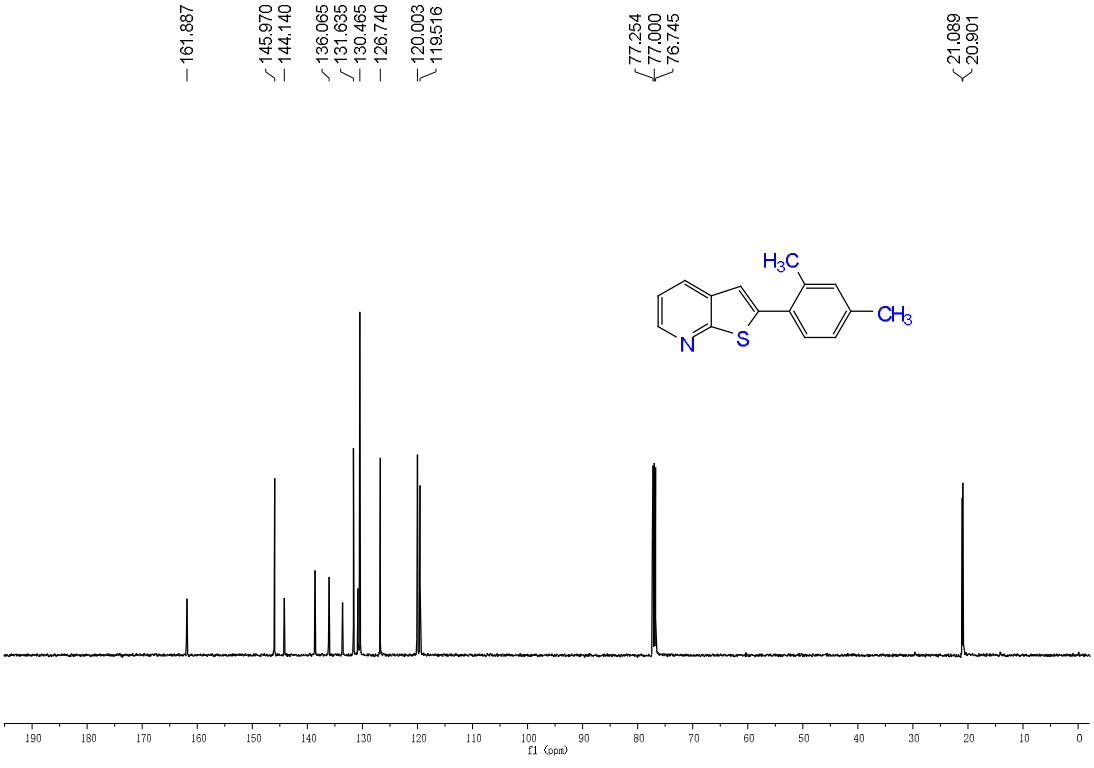
1.01
1.01
1.02
0.92
0.92
0.99
3.02
2.93
f1 (ppm)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144.140
136.065
131.635
130.465
126.740
120.003
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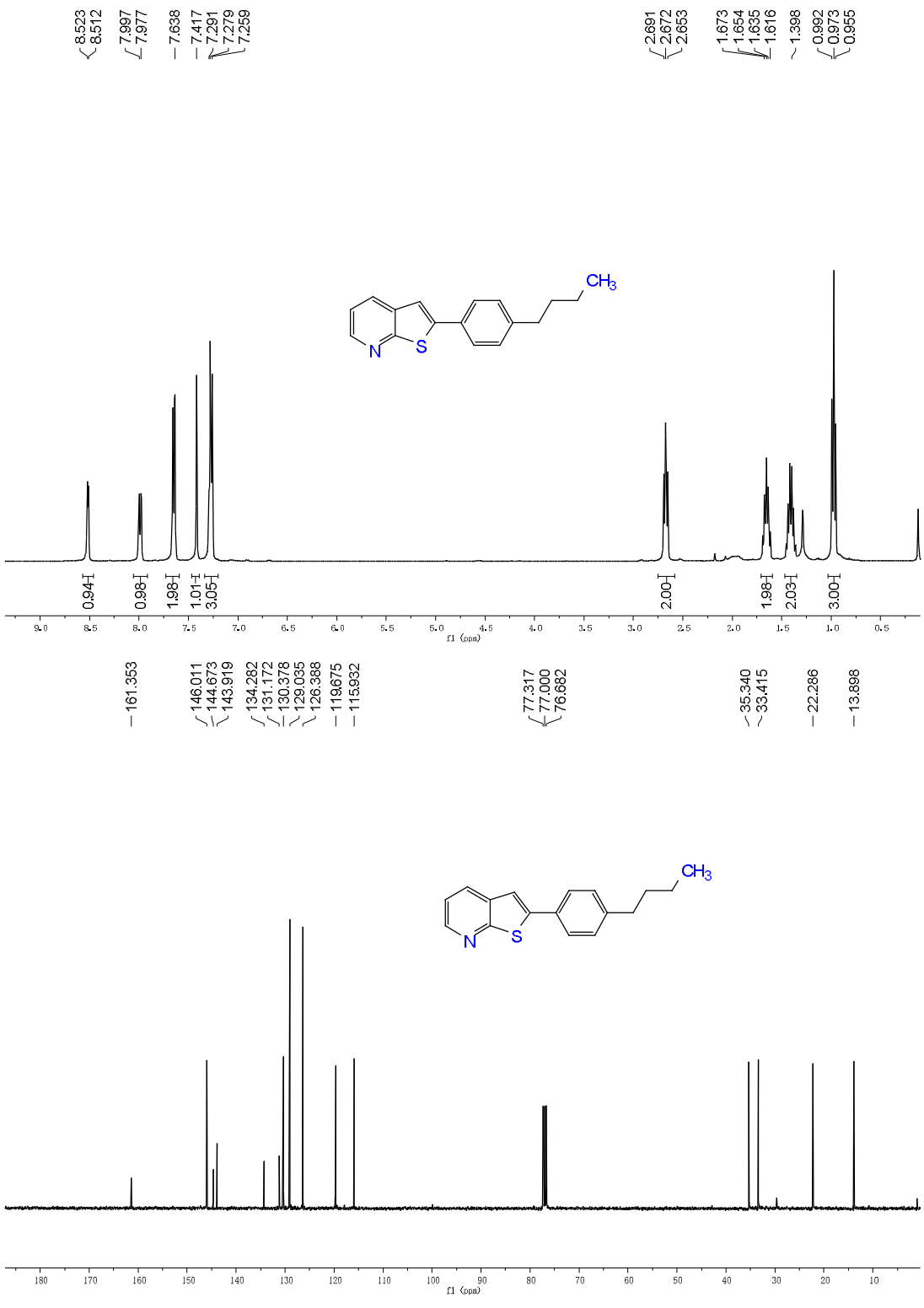
77.000
76.745
21.089
20.901
H3C
CH3
N
S
f1 (ppm)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8.512
7.997
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7.638
7.417
7.291
7.279
7.259
2.691
2.672
2.653
1.673
1.654
1.635
1.616
1.398
0.992
0.973
0.955
CH3
N
S
0.94
0.98
1.98
1.01
3.05
2.00
1.98
2.03
3.00
f1 (ppm)
161.353
146.011
144.673
143.919
134.282
131.172
130.378
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77.000
76.682
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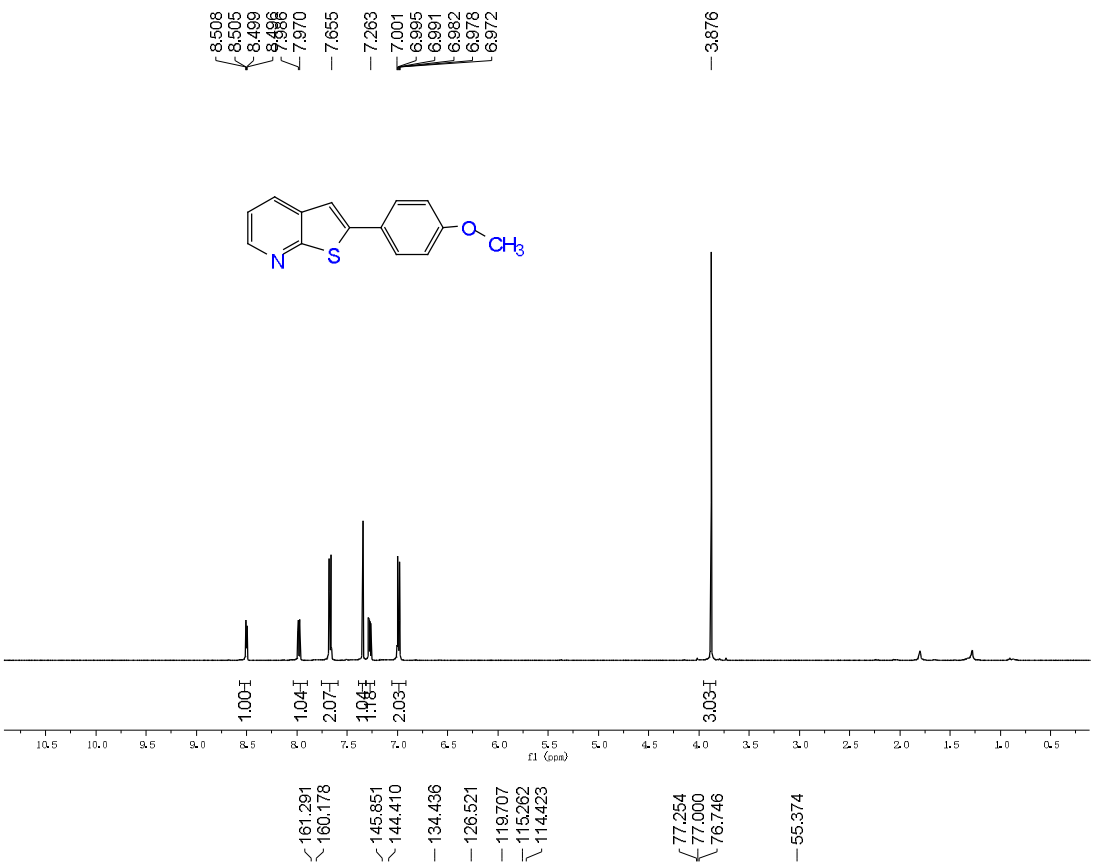
22.286
13.898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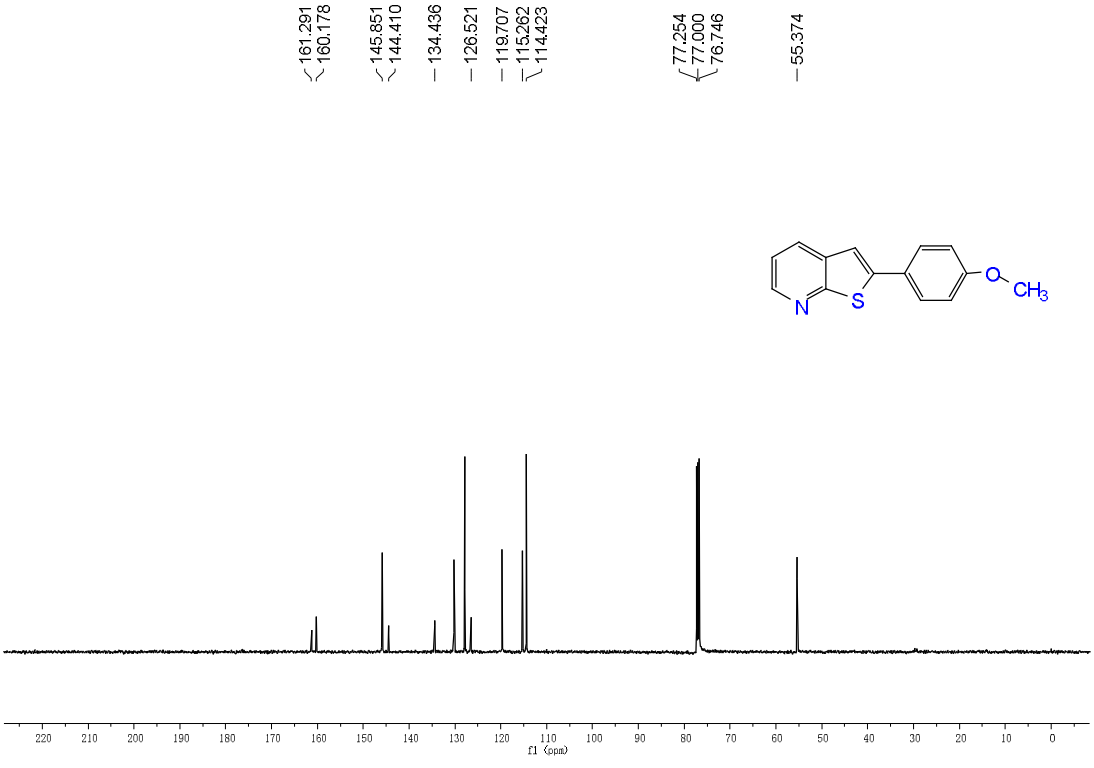
8.505
8.499
7.970
7.655
7.263
7.001
6.995
6.991
6.982
6.978
6.972
3.876
O
CH3
N
S
1.00
1.04
2.07
1.94
1.18
2.03
3.03
f1 (ppm)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126.521
119.707
115.262
114.423
77.254
77.000
76.746
55.374
O
CH3
N
S
f1 (ppm)

8.436
8.426
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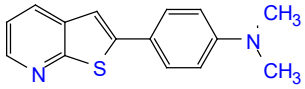
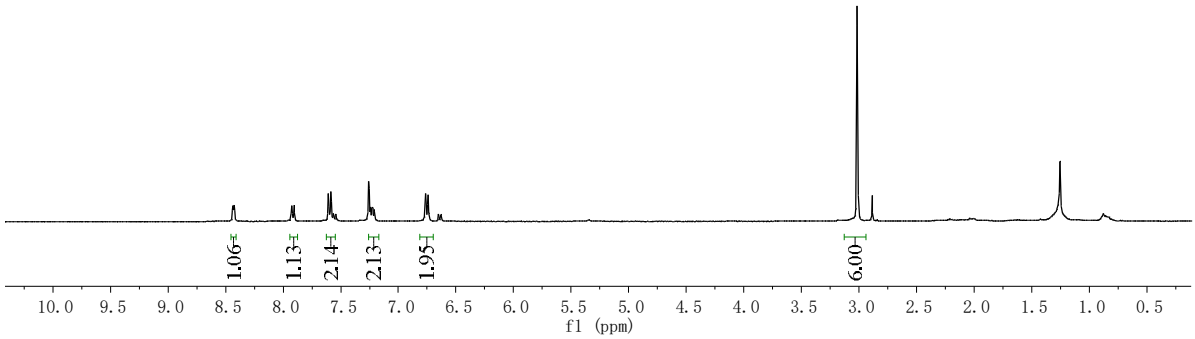
7.905
7.586
7.229
6.764
6.742
3.015





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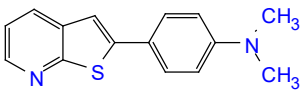
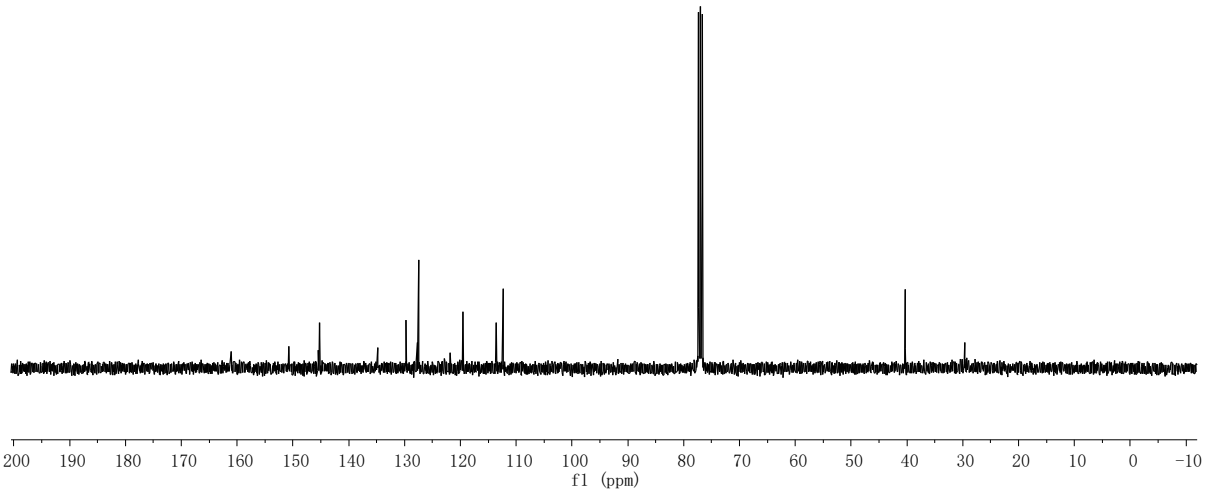
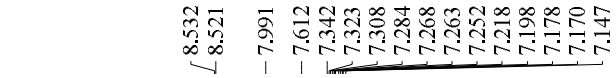
77.000
76.683
40.337



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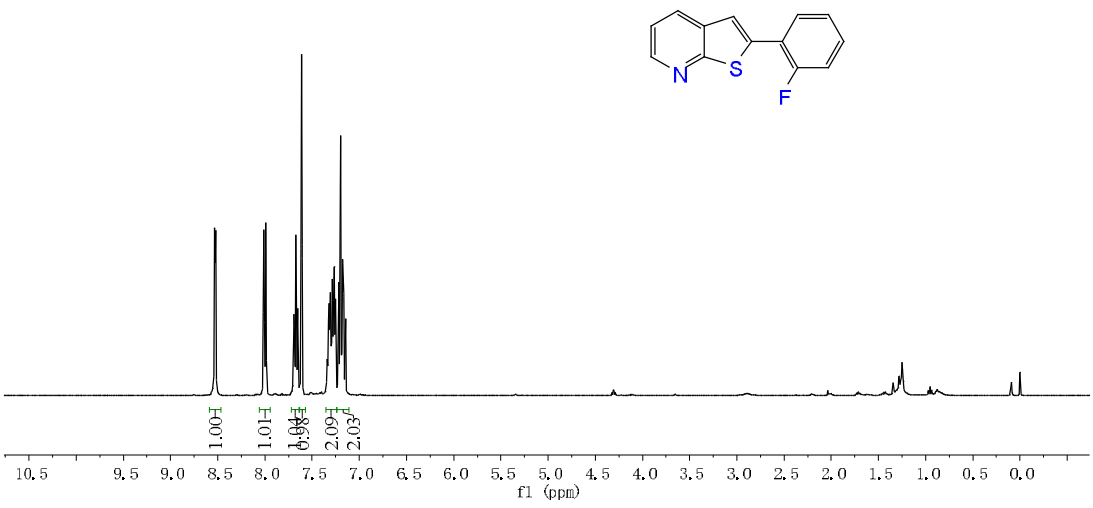
7.178
7.170
7.147


N
S
F
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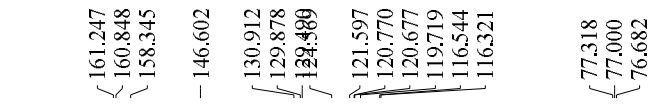
1.01
0.98
2.09
2.03
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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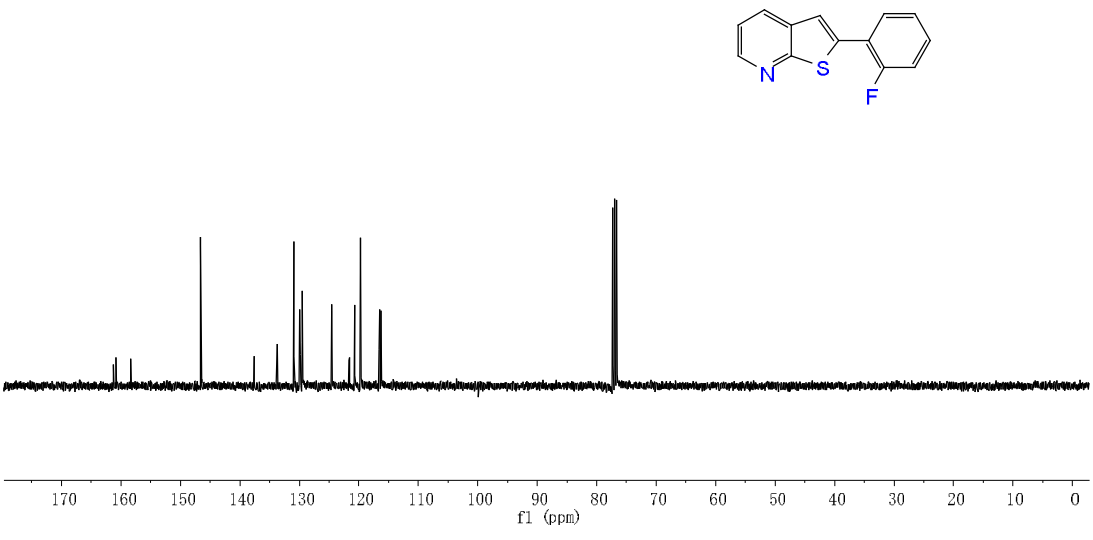
76.682


N
S
F
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140
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120
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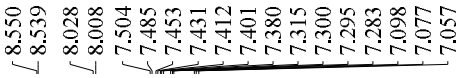
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.485
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7.295
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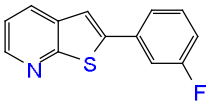
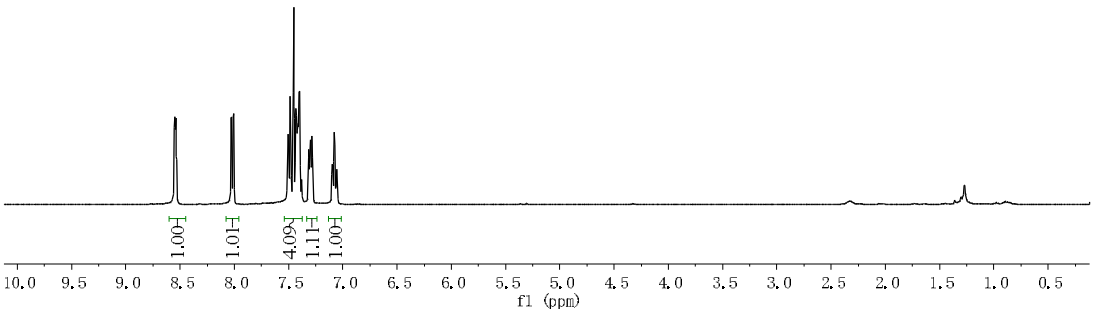
7.077
7.057


N
S
F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1.01
4.09
1.11
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


164.244
161.792
161.394
146.642
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130.522
119.901
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113.396
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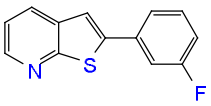
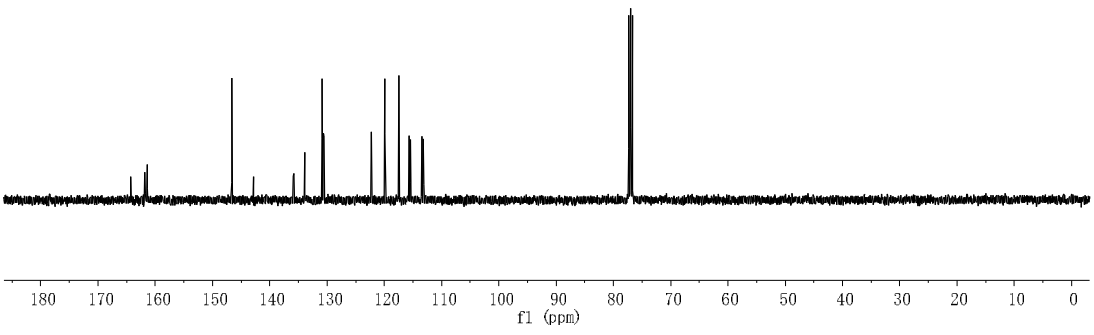
76.682


N
S
F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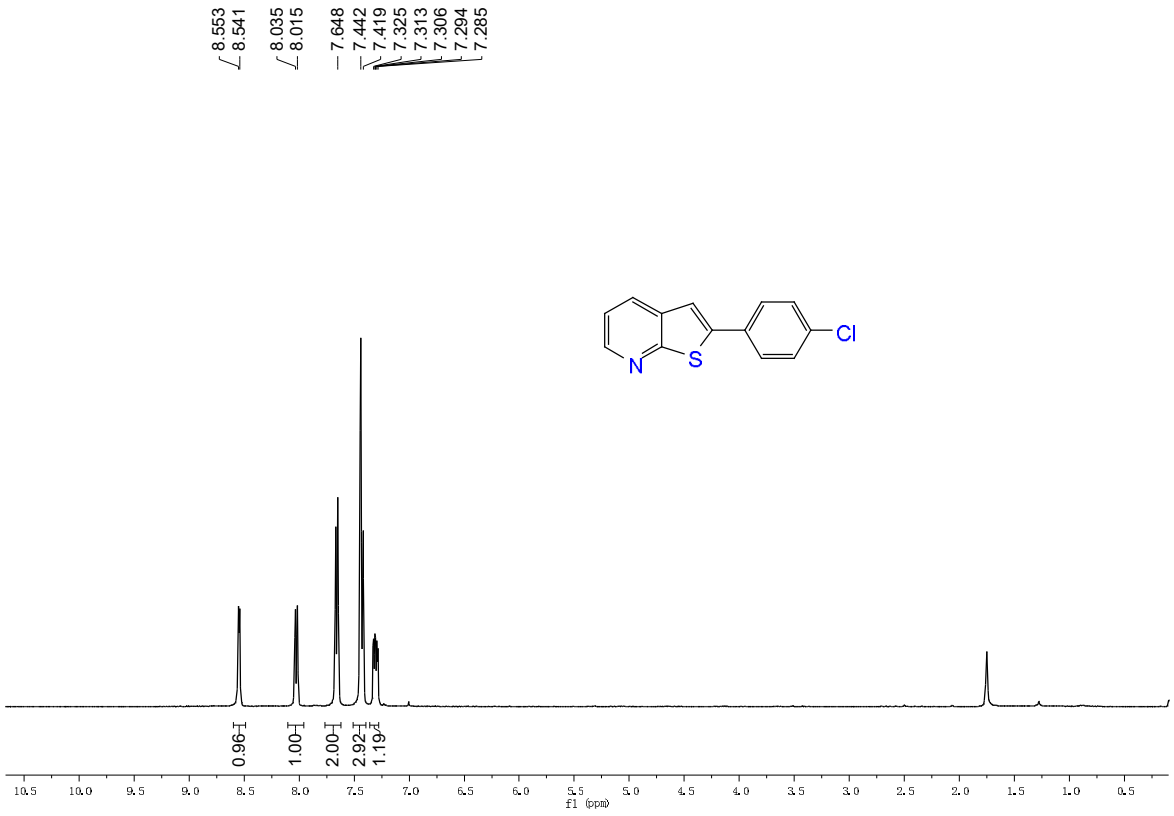
f1 (ppm)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7.442
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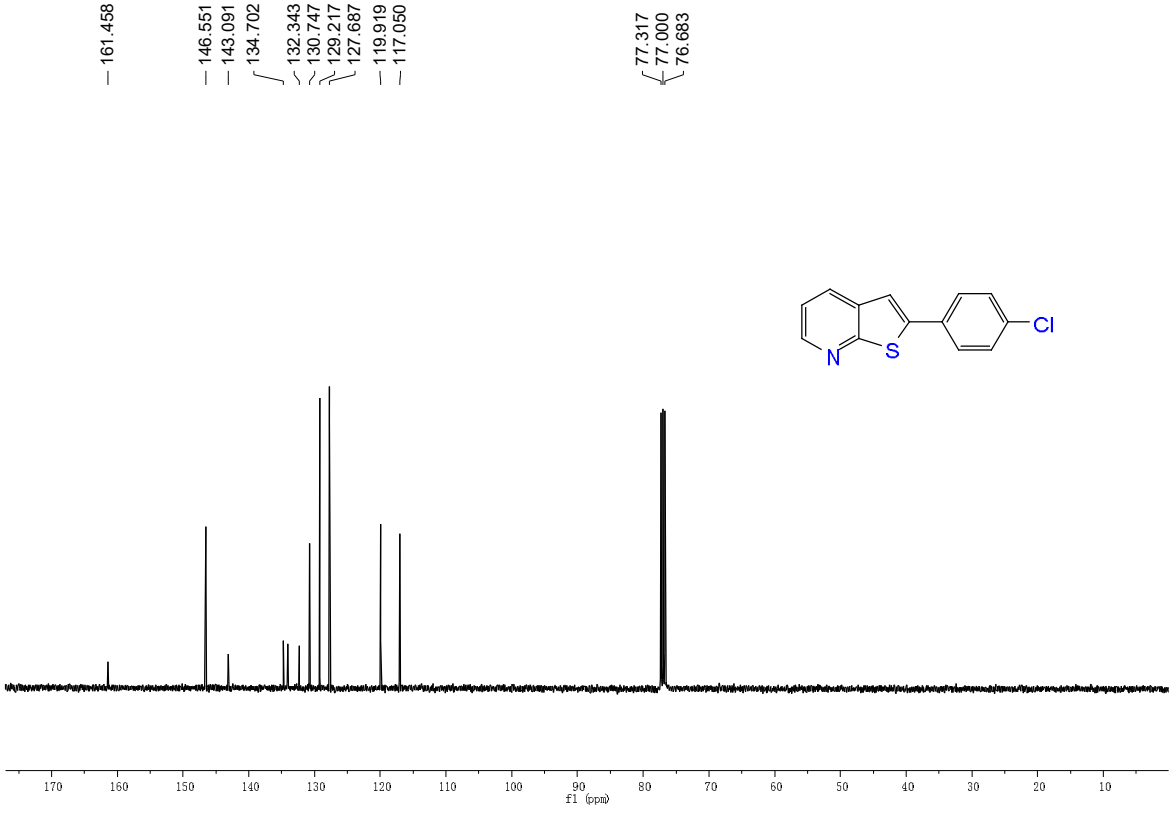
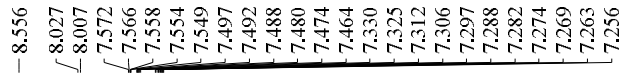
7.306
7.294
7.285
Cl
N
S
0.96
1.00
2.00
2.92
1.19
fl (ppm)


161.458
146.551
143.091
134.702
132.343
130.747
129.217
127.687
119.919
117.050
77.317
77.000
76.683
Cl
N
S
fl (ppm)


8.556
8.027
8.007
7.572
7.566
7.558
7.554
7.549
7.497
7.492
7.488
7.480
7.474
7.464
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7.306
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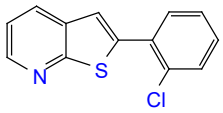
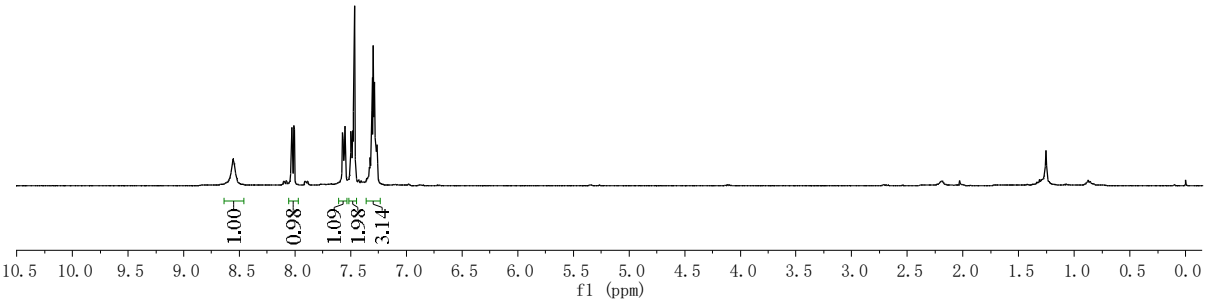
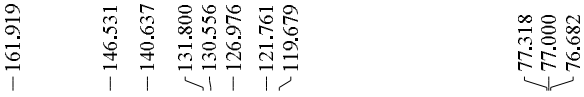
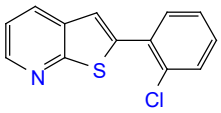
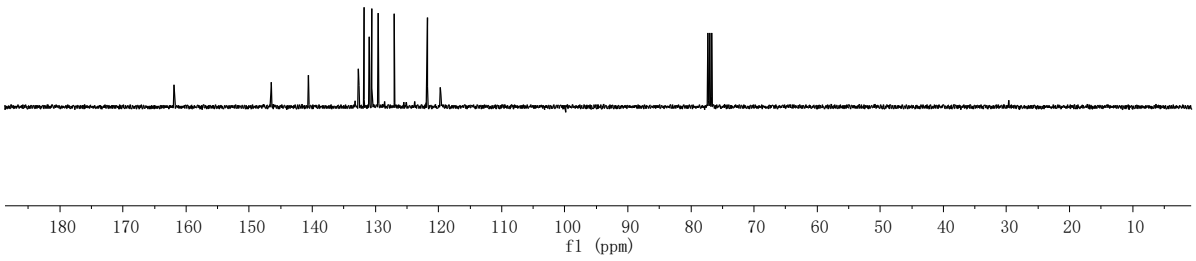
7.274
7.269
7.263
7.256

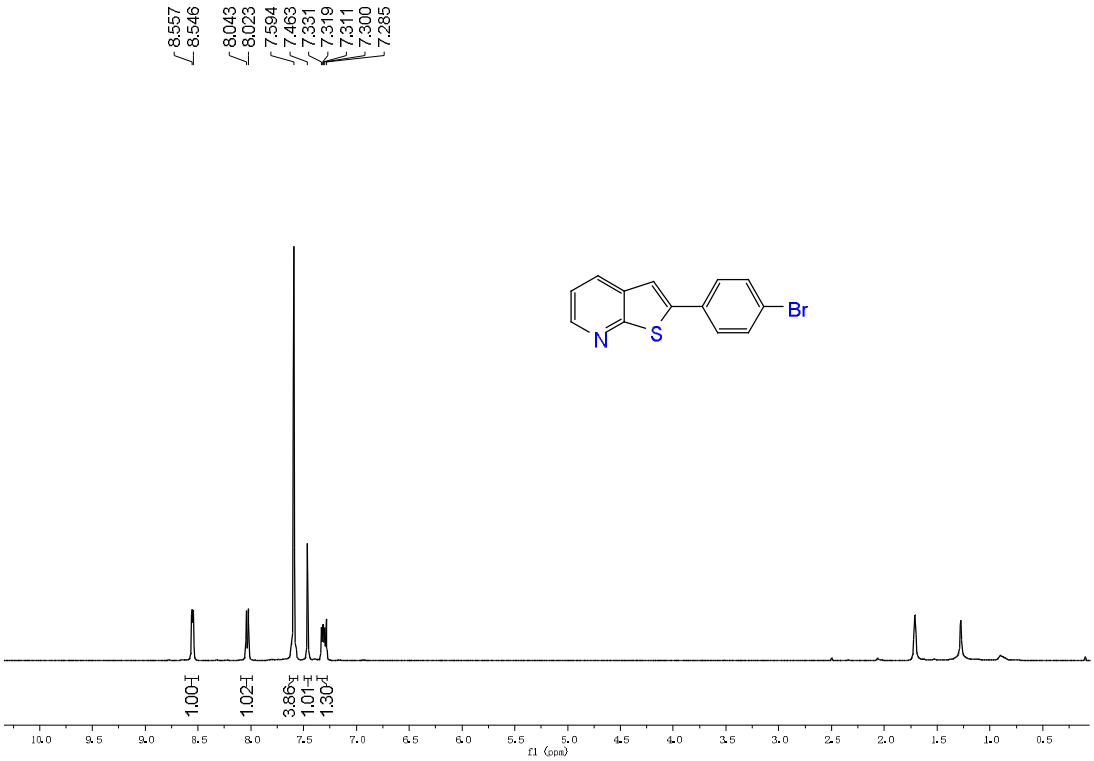

N
S
Cl

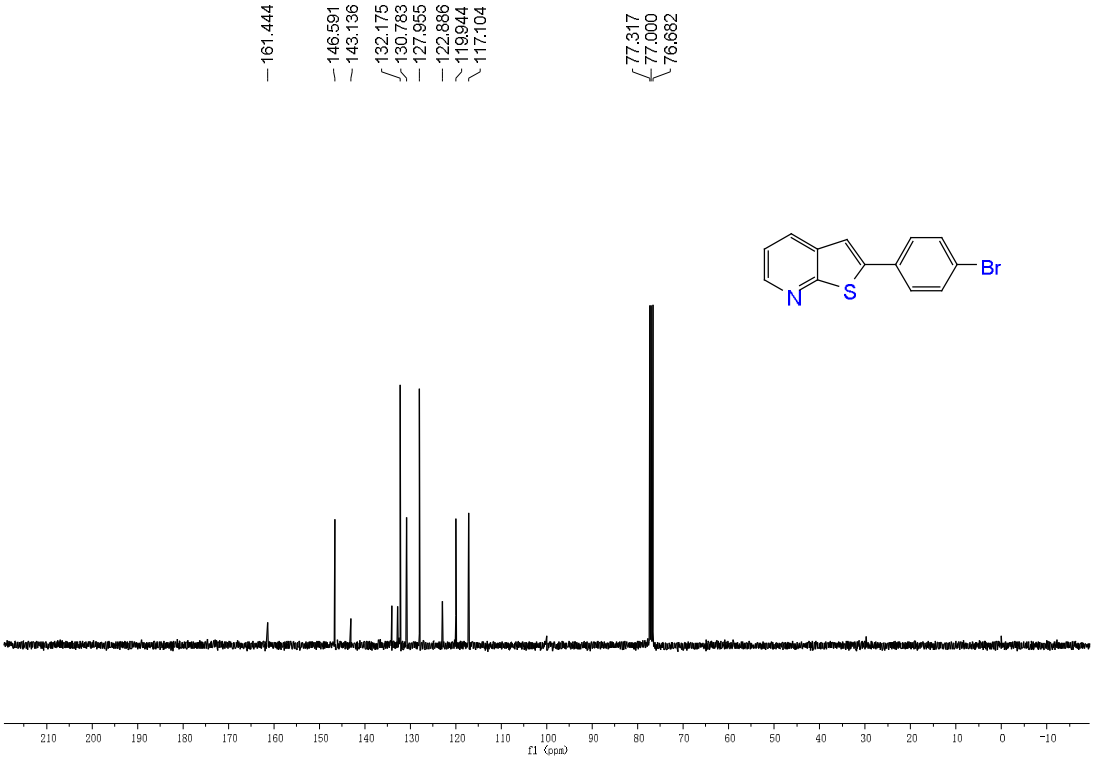

1.00
0.98
1.09
1.98
3.14
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

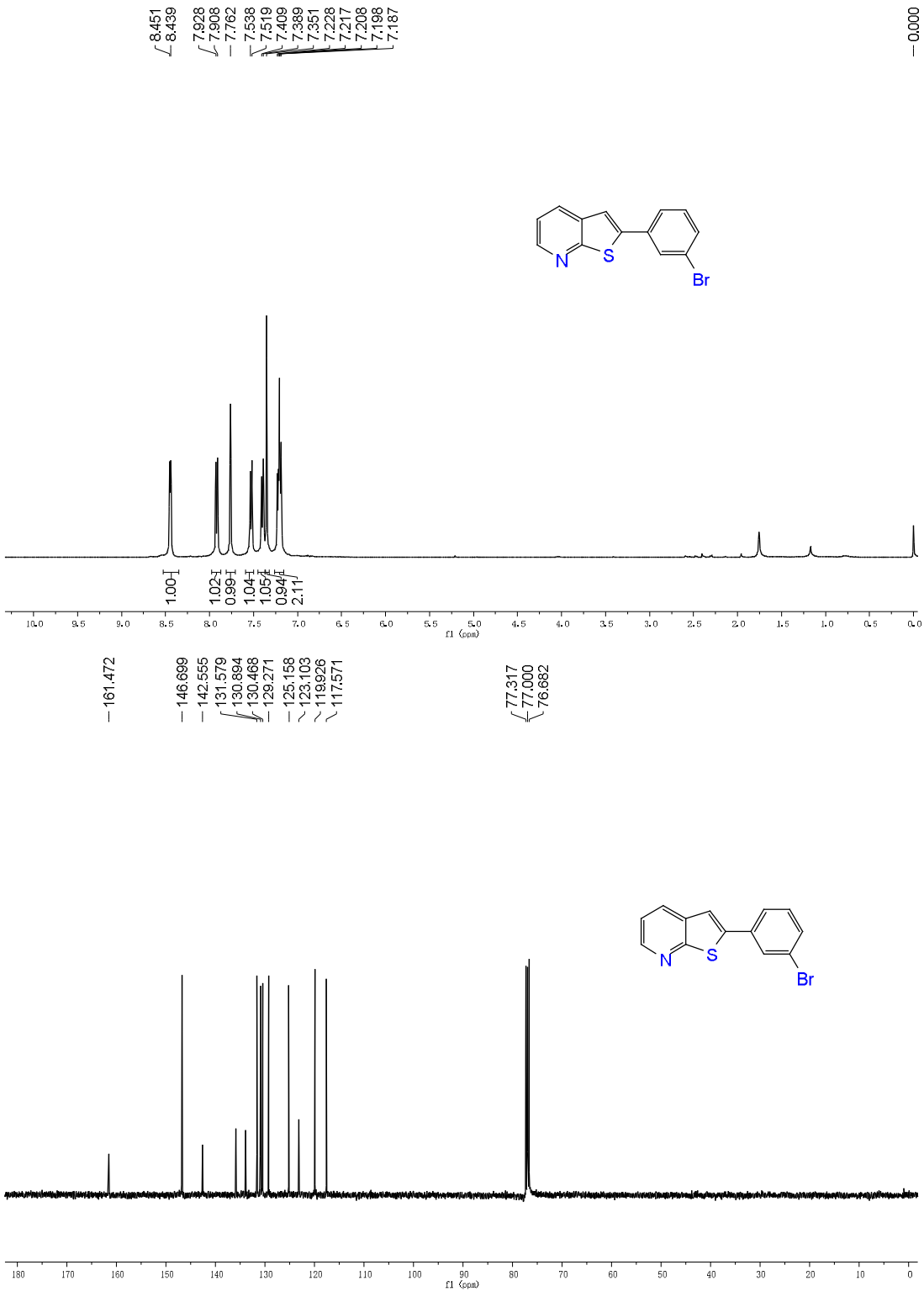

161.919
146.531
140.637
131.800
130.556
126.976
121.761
119.679
77.318
77.000
76.682

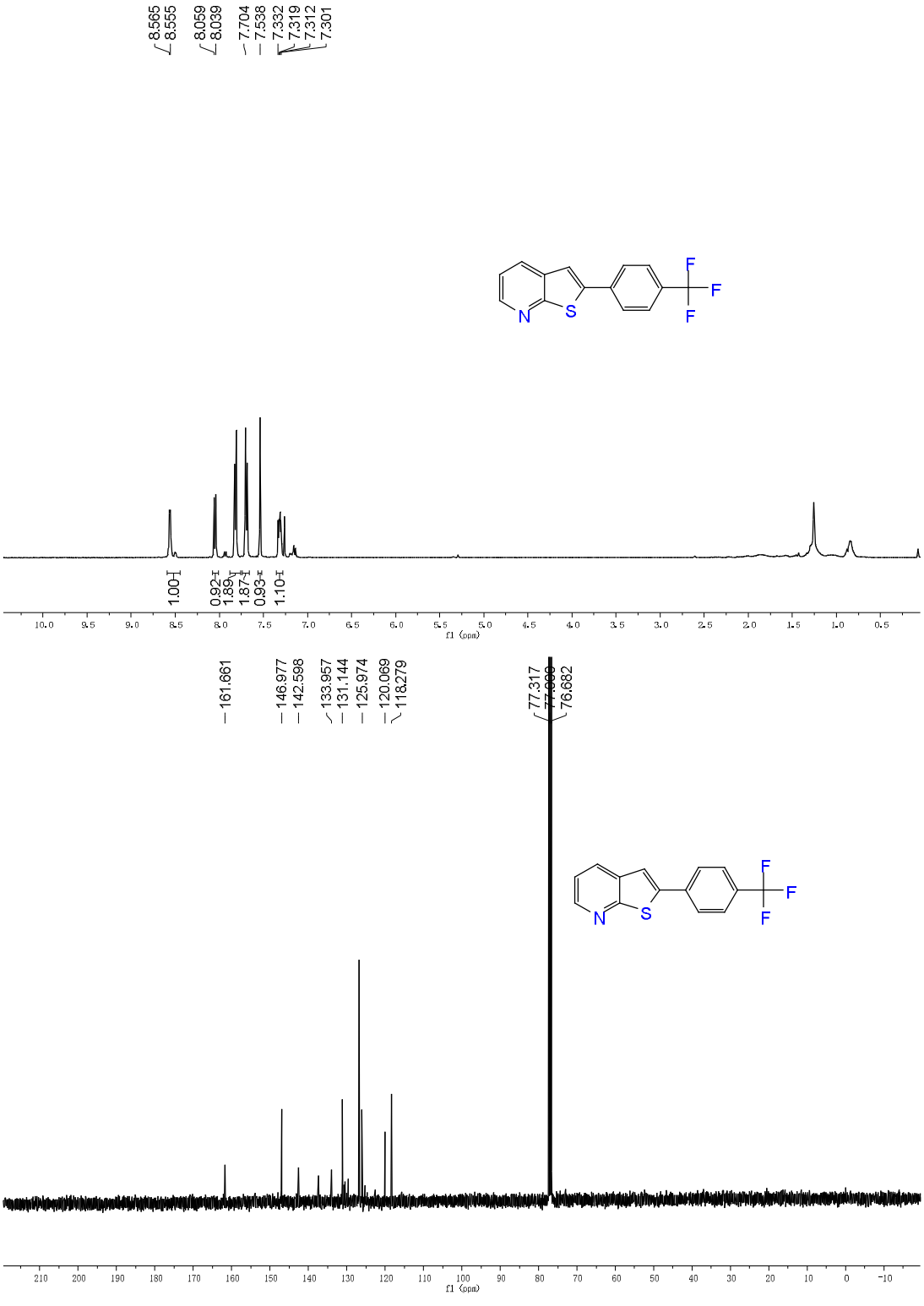

N
S
Cl

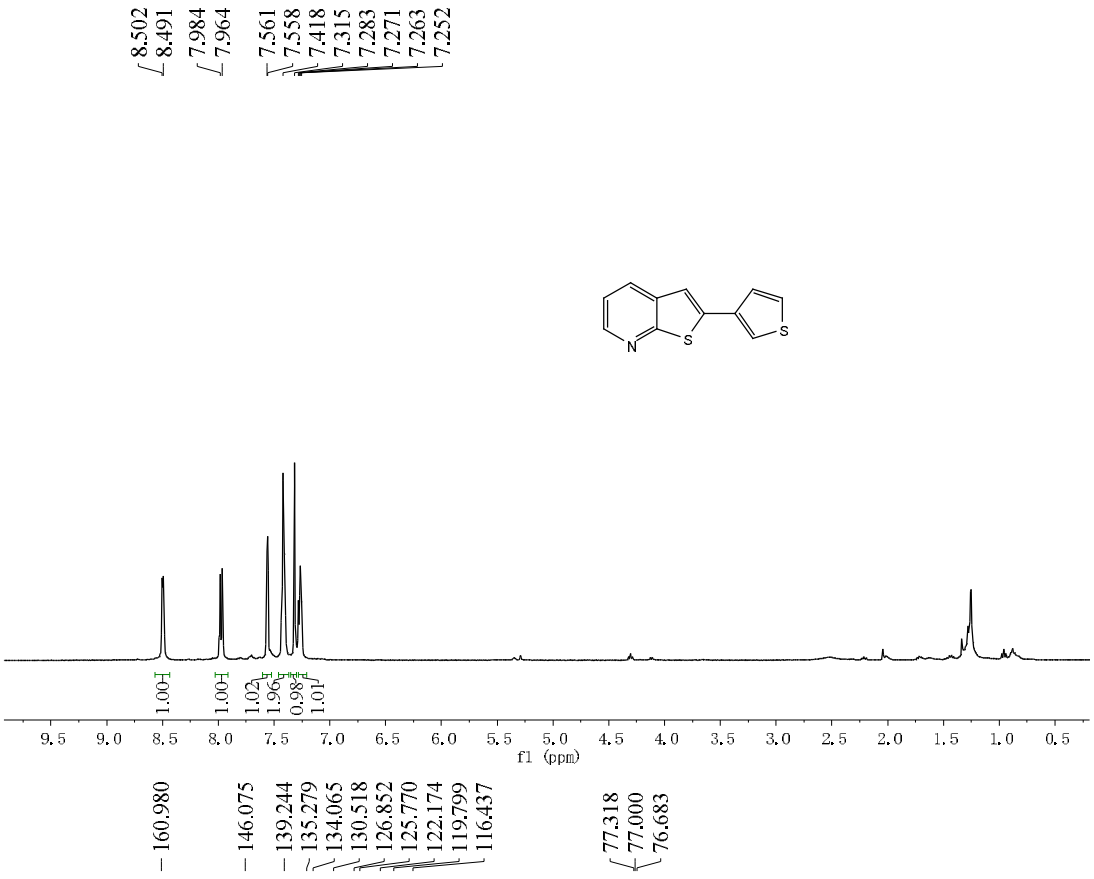

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

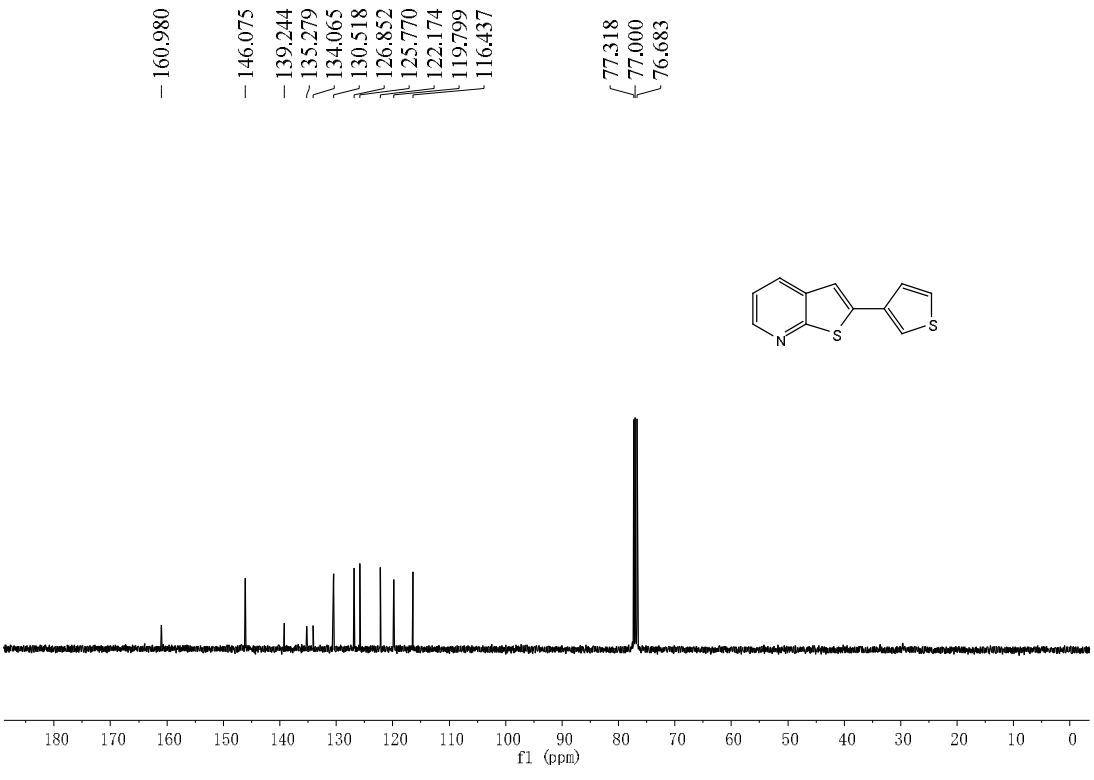

8.557
8.546
8.043
8.023
7.594
7.463
7.331
7.319
7.311
7.300
7.285
Br
N
S
1.00
1.02
3.86
1.01
1.30
f1 (ppm)


161.444
146.591
143.136
132.175
130.783
127.955
122.886
119.944
117.104
77.317
77.000
76.682
Br
N
S
f1 (ppm)

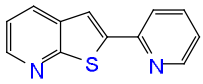

8.451
8.439
7.928
7.908
7.762
7.538
7.519
7.409
7.389
7.351
7.228
7.217
7.208
7.198
7.187
0.000
N
S
Br
1.00
1.02
0.99
1.04
1.05
0.94
2.11
f1 (ppm)
161.472
146.699
142.555
131.579
130.894
130.468
129.271
125.158
123.103
119.926
117.571
77.317
77.000
76.682
N
S
Br
f1 (ppm)

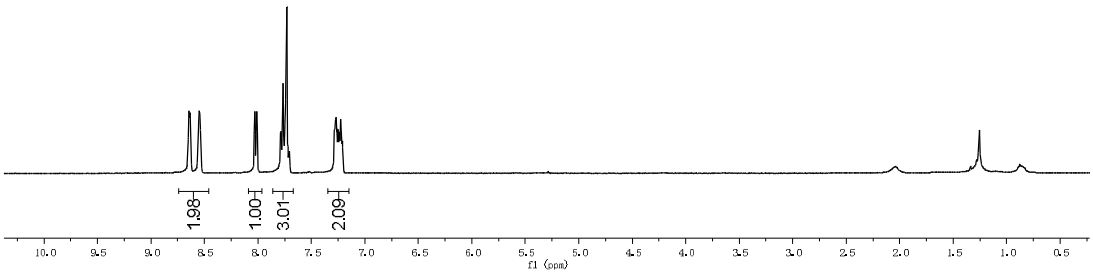

8.565
8.555
8.059
8.039
7.704
7.538
7.332
7.319
7.312
7.301
F
F
F
N
S
1.00
0.92
1.89
1.87
0.93
1.10
f1 (ppm)
161.661
146.977
142.598
133.957
131.144
125.974
120.069
118.279
77.317
76.682
f1 (ppm)


8.502
8.491
7.984
7.964
7.561
7.558
7.418
7.315
7.283
7.271
7.263
7.252
N
S
S
1.00
1.00
1.02
1.96
0.98
1.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

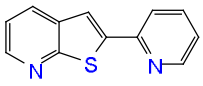

160.980
146.075
139.244
135.279
134.065
130.518
126.852
125.770
122.174
119.799
116.437
77.318
77.000
76.683
N
S
S
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

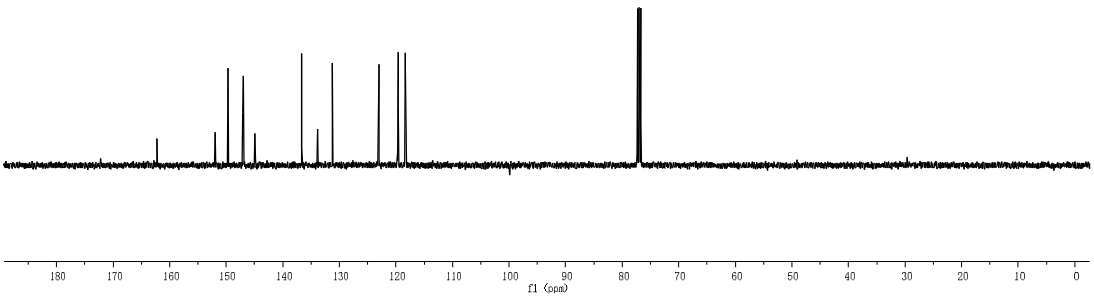
8.643
8.633
8.551
8.541
8.029
8.009
7.708
7.281
7.273
7.270
7.261
7.250
7.240
7.227
7.210

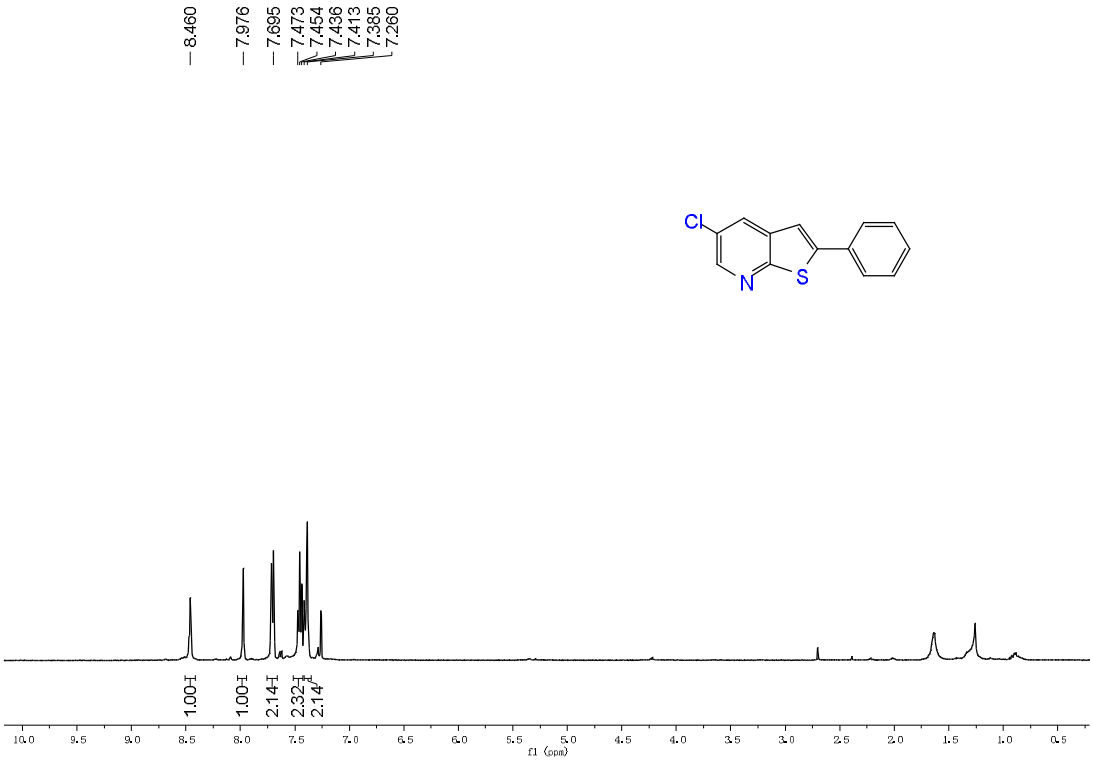


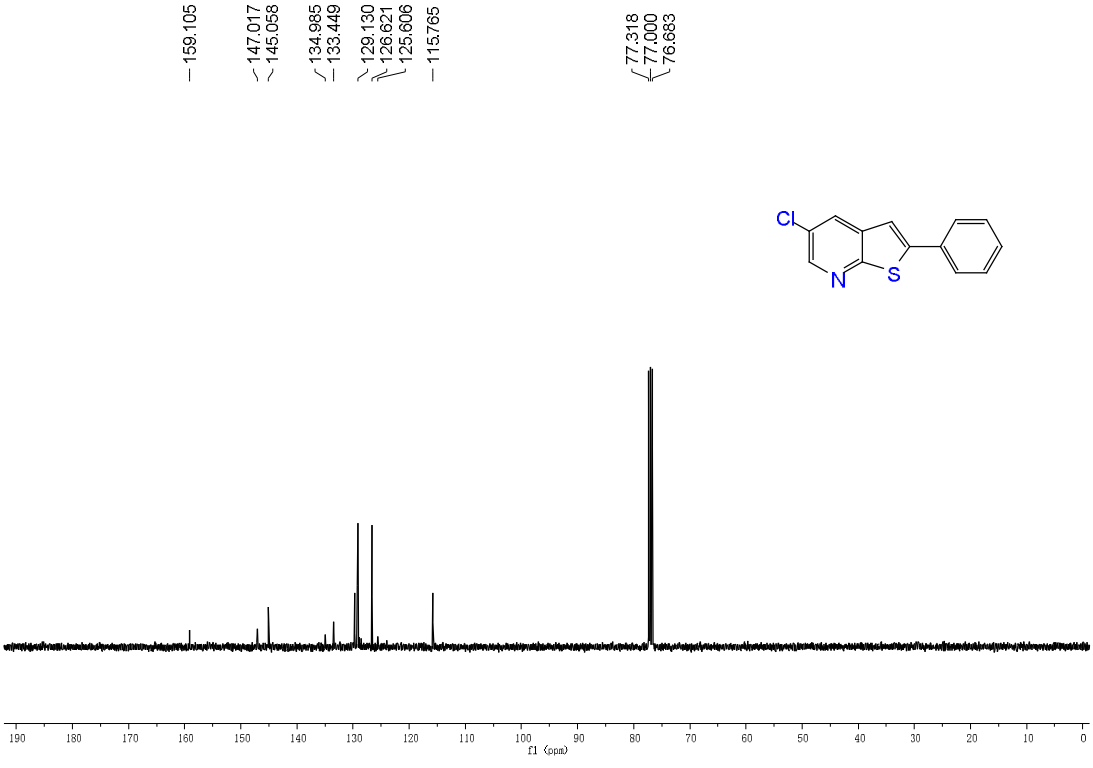


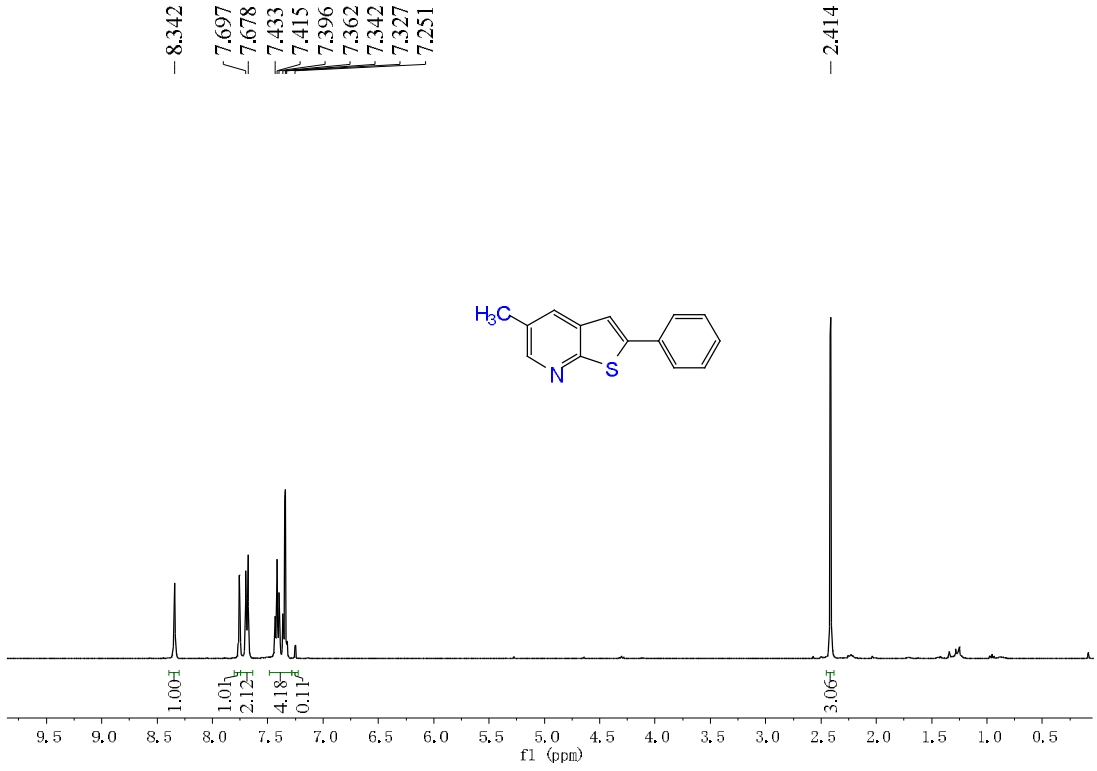
162.280
151.944
149.727
147.029
144.902
136.640
133.894
131.212
123.021
119.718
119.575
118.313
77.318
77.000
76.683

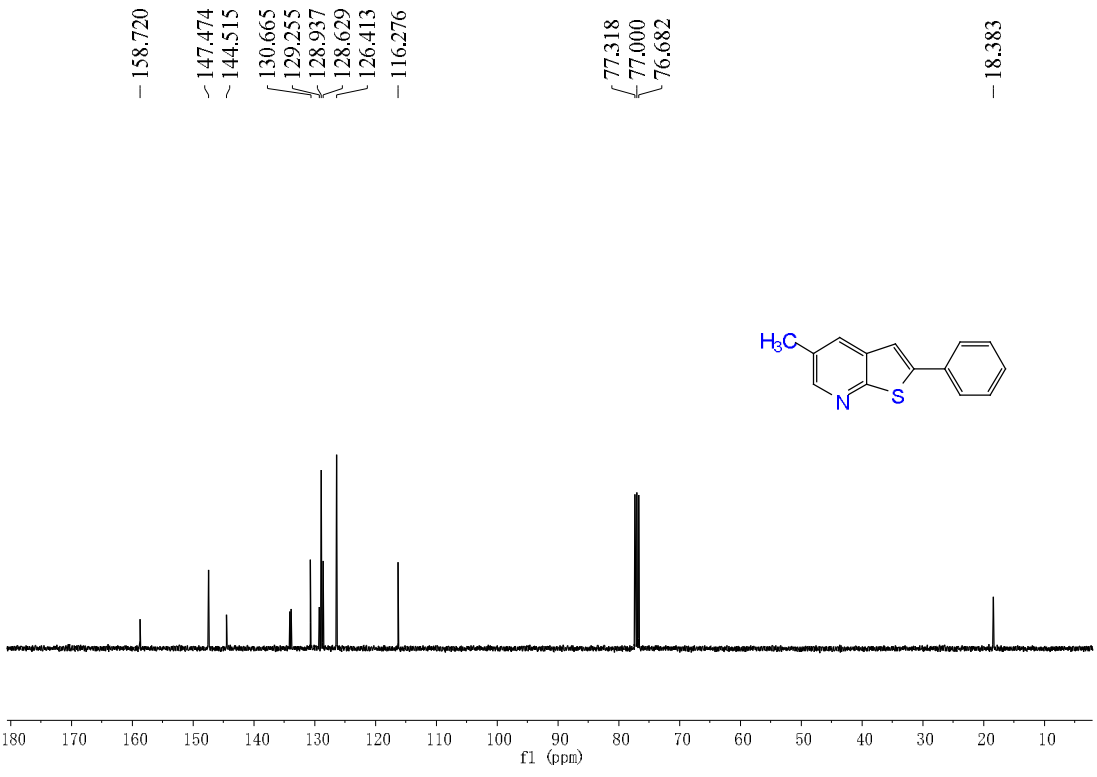


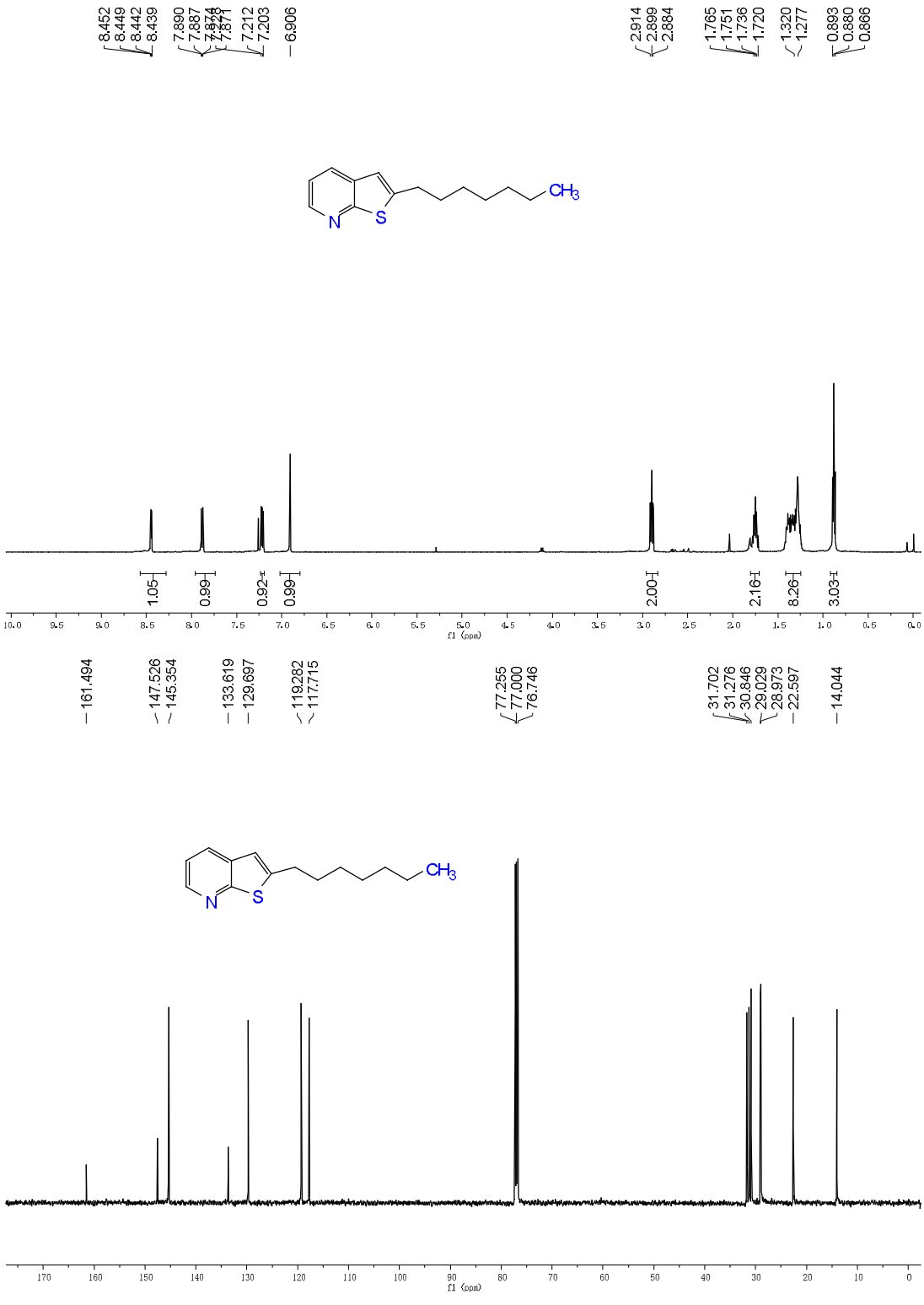


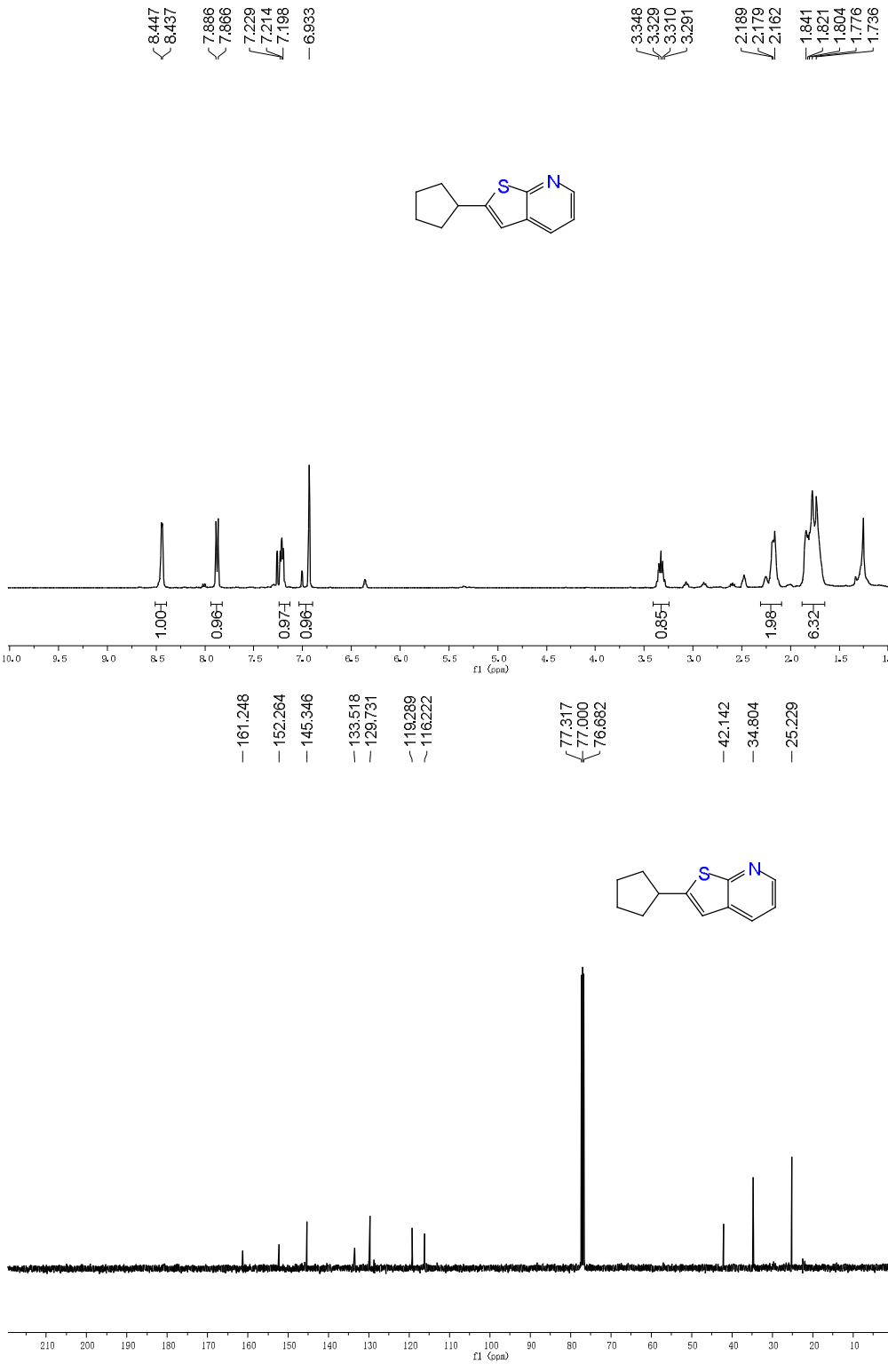

8.460
7.976
7.695
7.473
7.454
7.436
7.413
7.385
7.260
Cl
N
S
1.00
1.00
2.14
2.32
2.14
f1 (ppm)


159.105
147.017
145.058
134.985
133.449
129.130
126.621
125.606
115.765
77.318
77.000
76.683
Cl
N
S
f1 (ppm)


8.342
7.697
7.678
7.433
7.415
7.396
7.362
7.342
7.327
7.251
2.414
H3C
N
S
1.00
1.01
2.12
4.18
0.11
3.06
f1 (ppm)

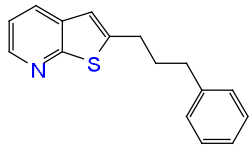

158.720
147.474
144.515
130.665
129.255
128.937
128.629
126.413
116.276
77.318
77.000
76.682
18.383
H3C
N
S
f1 (ppm)

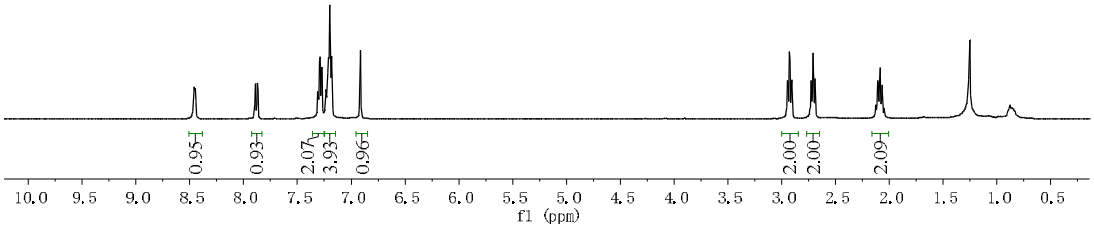

8.452
8.449
8.442
8.439
7.890
7.887
7.874
7.871
7.212
7.203
6.906
2.914
2.899
2.884
1.765
1.751
1.736
1.720
1.320
1.277
0.893
0.880
0.866
CH3
N
S
1.05
0.99
0.92
0.99
2.00
2.16
8.26
3.03
f1 (ppm)
161.494
147.526
145.354
133.619
129.697
119.282
117.715
77.255
77.000
76.746
31.702
31.276
30.846
29.029
28.973
22.597
14.044
CH3
N
S
f1 (ppm)


8.447
8.437
7.886
7.866
7.229
7.214
7.198
6.933
3.348
3.329
3.310
3.291
2.189
2.179
2.162
1.841
1.821
1.804
1.776
1.736
S
N
1.00
0.96
0.97
0.96
0.85
1.98
6.32
f1 (ppm)
161.248
152.264
145.346
133.518
129.731
119.289
116.222
77.317
77.000
76.682
42.142
34.804
25.229
S
N
f1 (ppm)

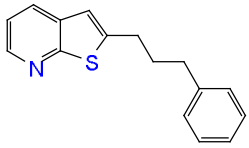
8.460
8.450
7.891
7.871
7.233
7.181
6.914

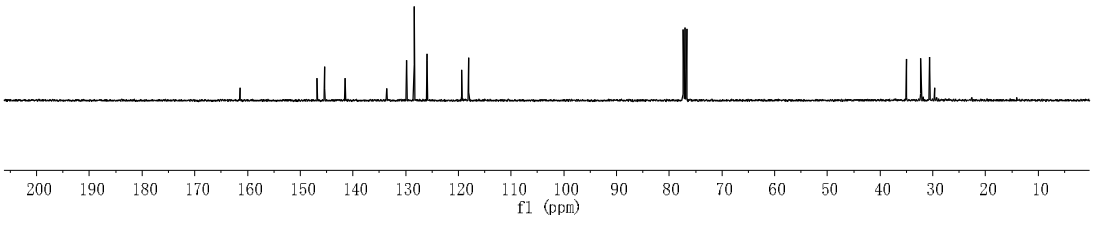
2.945
2.926
2.907
2.727
2.708
2.689
2.124
2.106
2.087
2.068
2.049

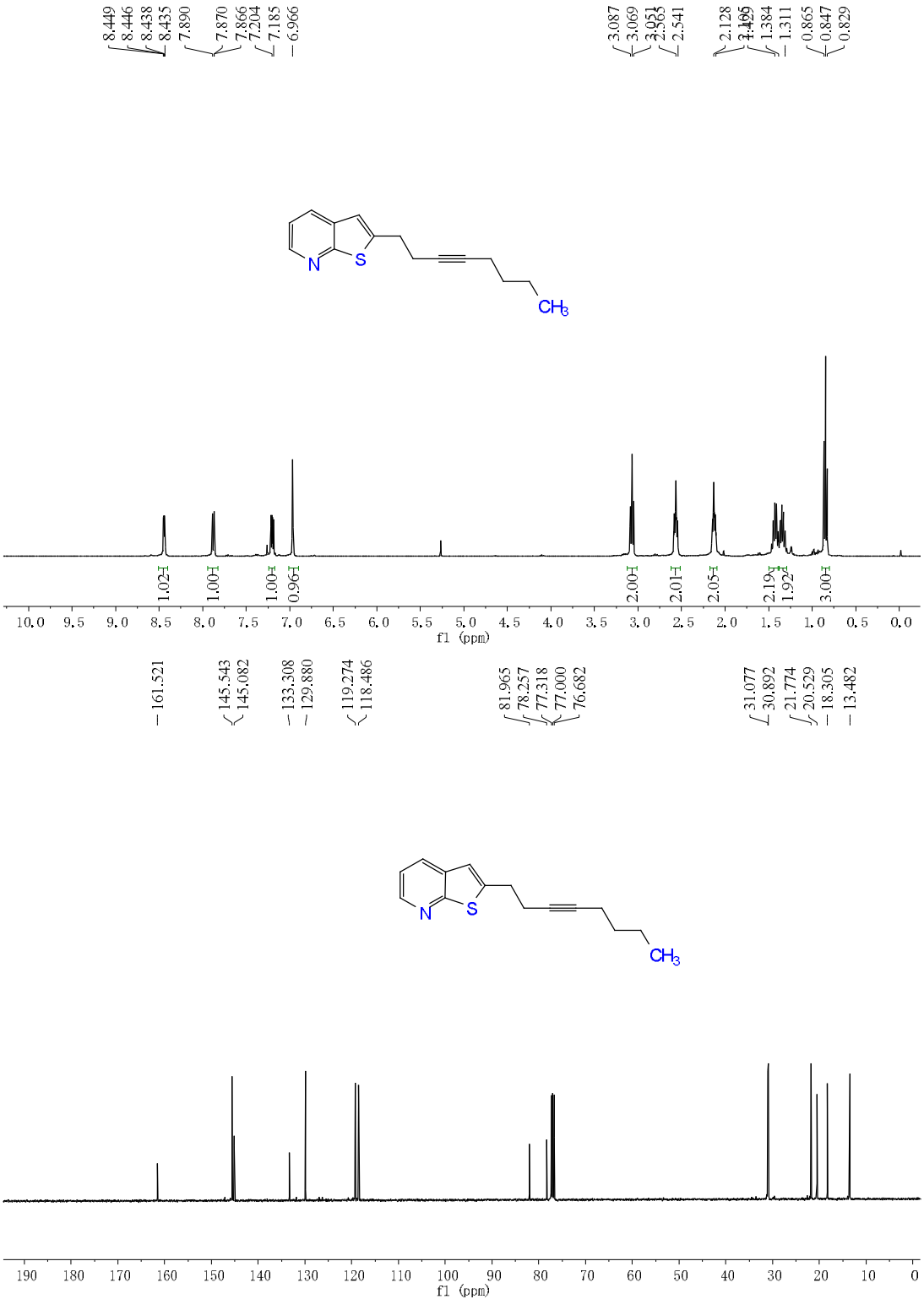




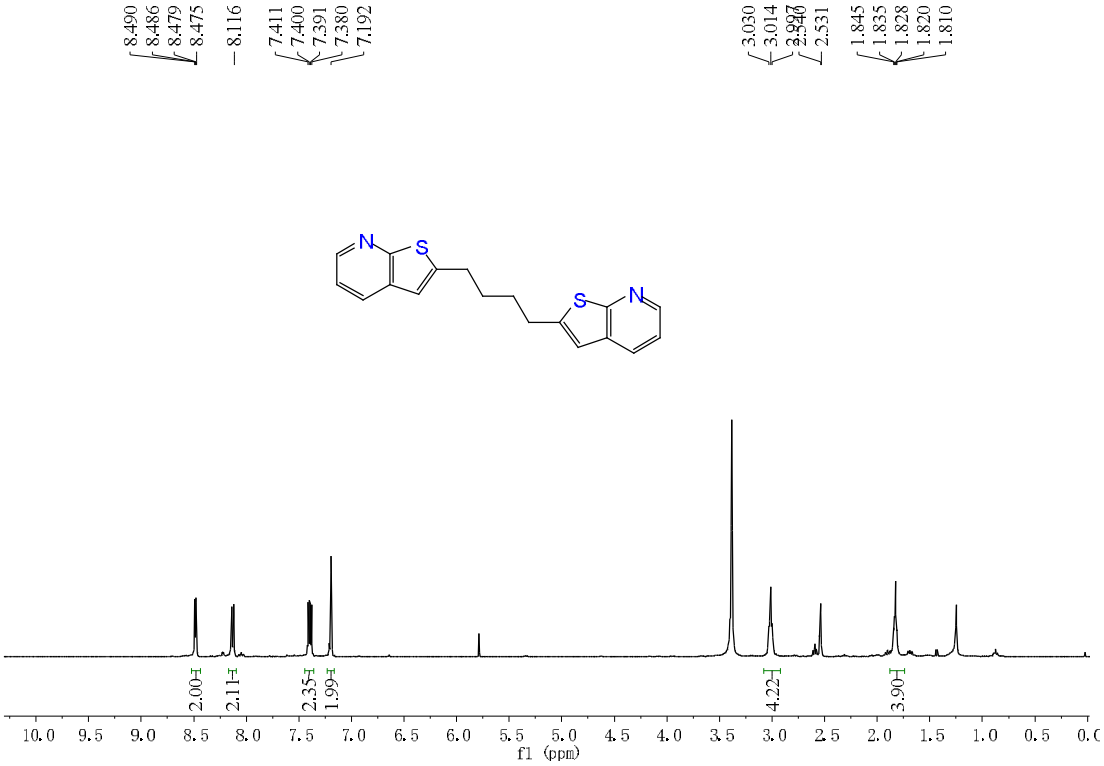
161.384
146.792
145.386
141.439
128.411
125.917
119.335
118.036
77.318
77.000
76.682
35.021
32.275
30.607



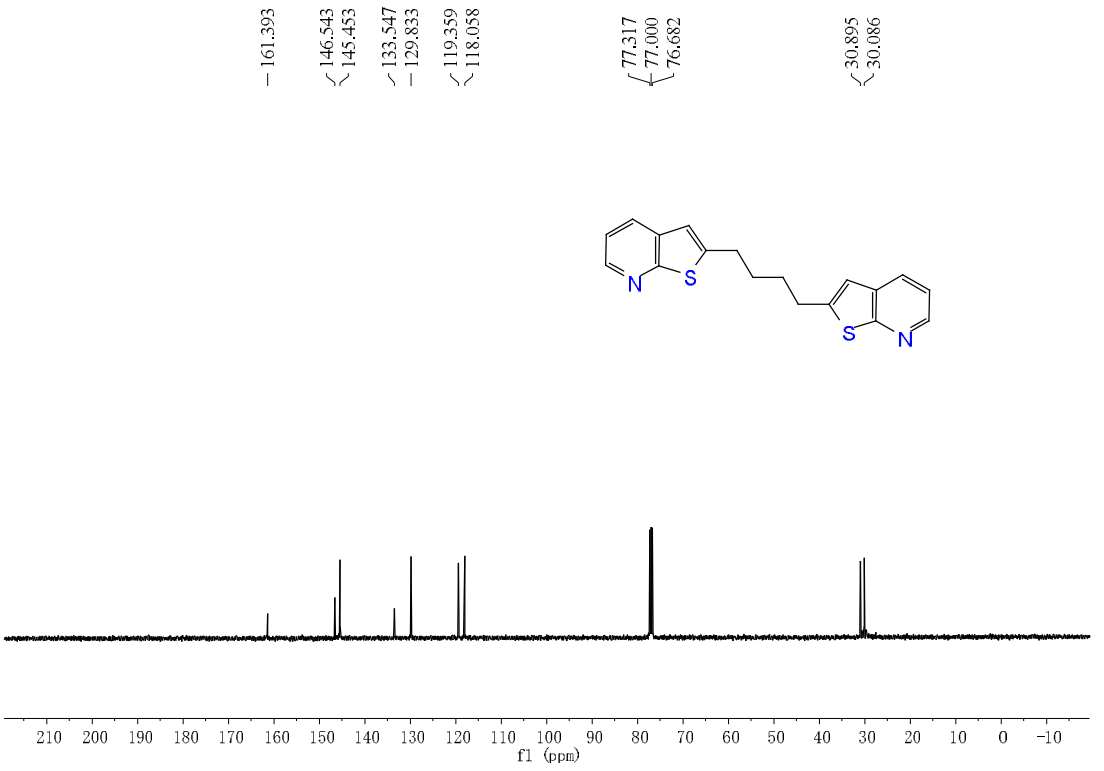


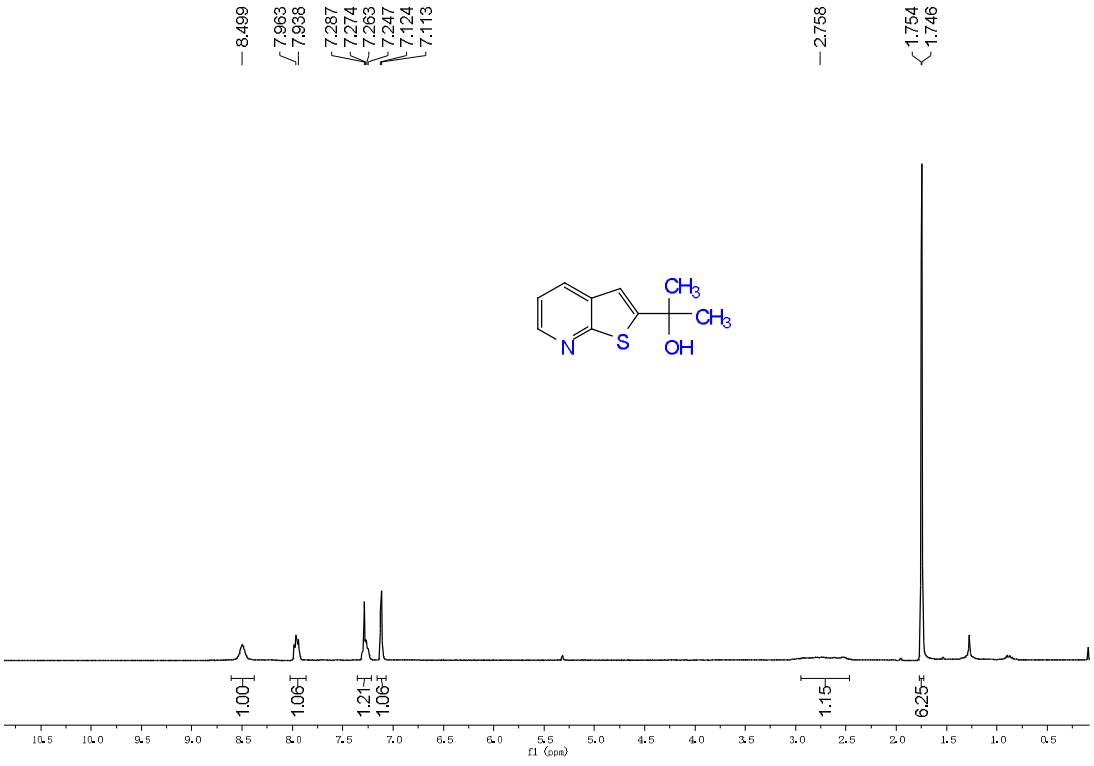

8.449
8.446
8.438
8.435
7.890
7.870
7.866
7.204
7.185
6.966
3.087
3.069
3.051
3.565
2.541
2.128
1.384
1.311
0.865
0.847
0.829
N
S
CH3
1.02
1.00
1.00
0.96
2.00
2.01
2.05
2.19
1.92
3.00
f1 (ppm)
161.521
145.543
145.082
133.308
129.880
119.274
118.486
81.965
78.257
77.318
77.000
76.682
31.077
30.892
21.774
20.529
18.305
13.482
f1 (ppm)

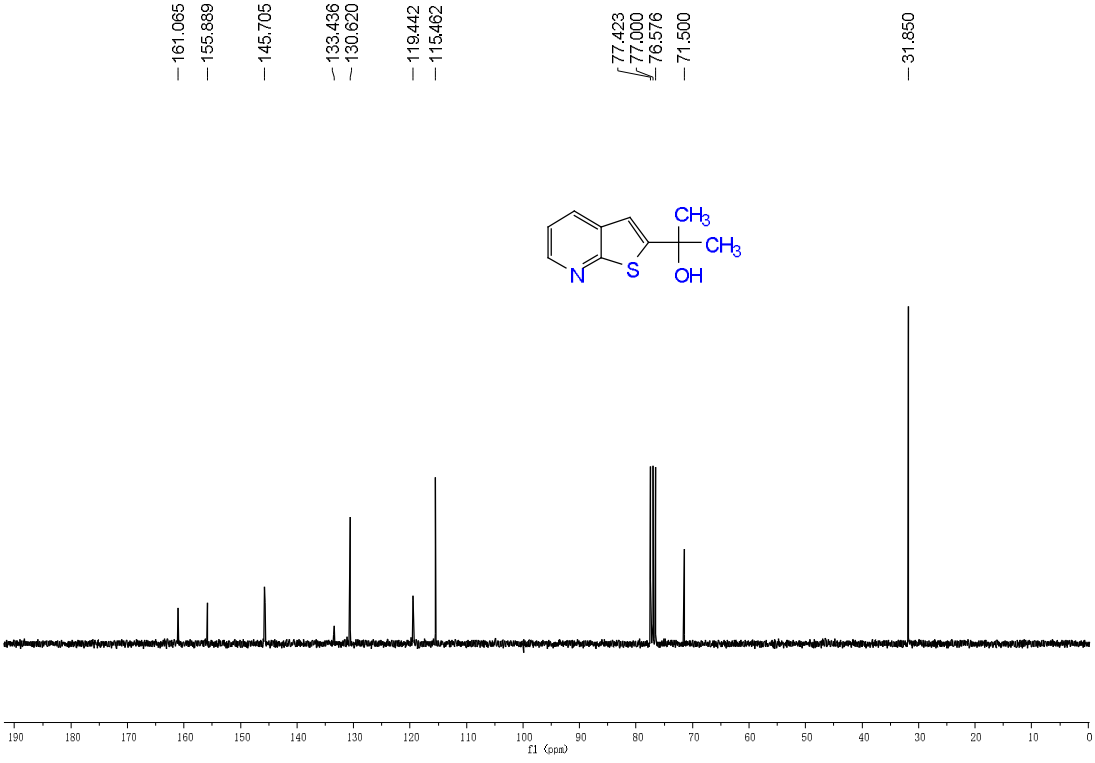
DMSO-d6 as solvent

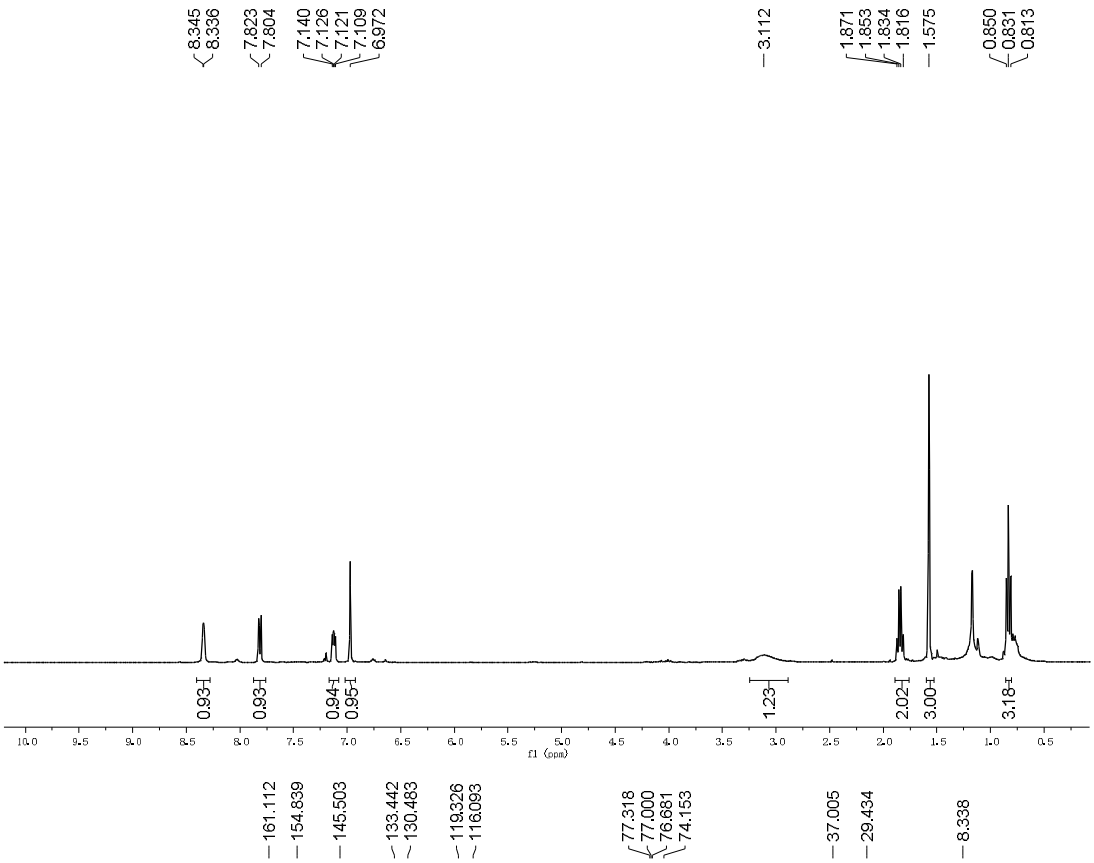


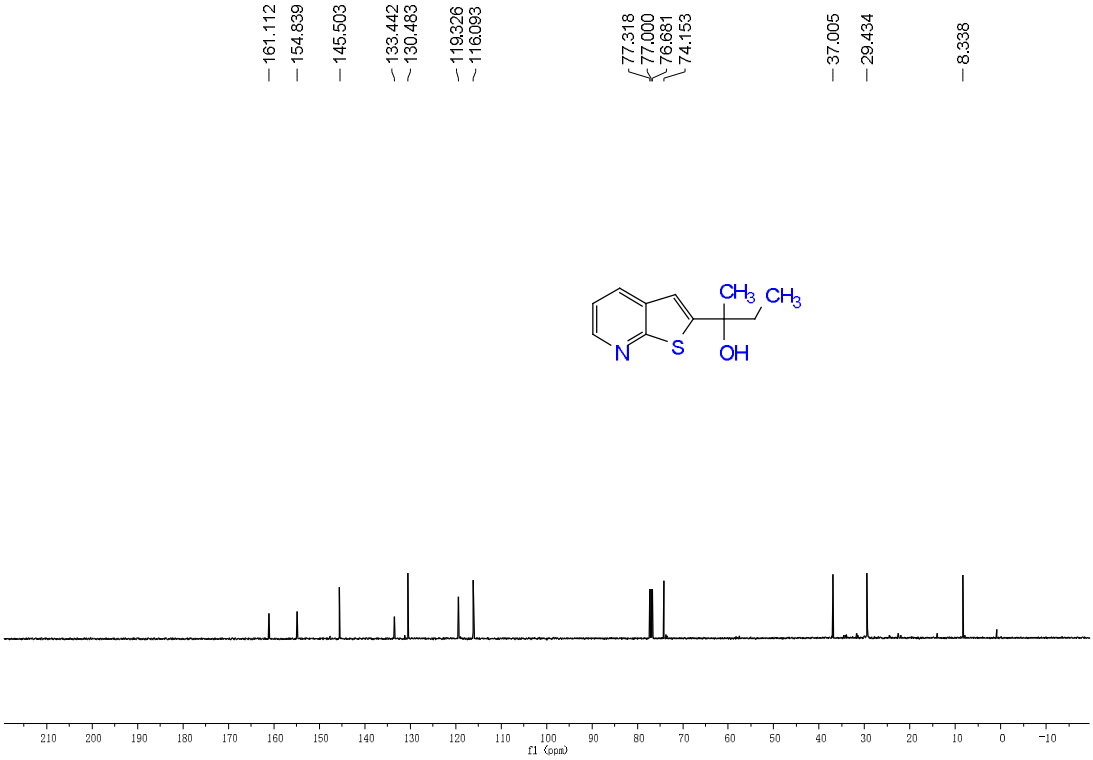
$CDCl_3$ as solvent



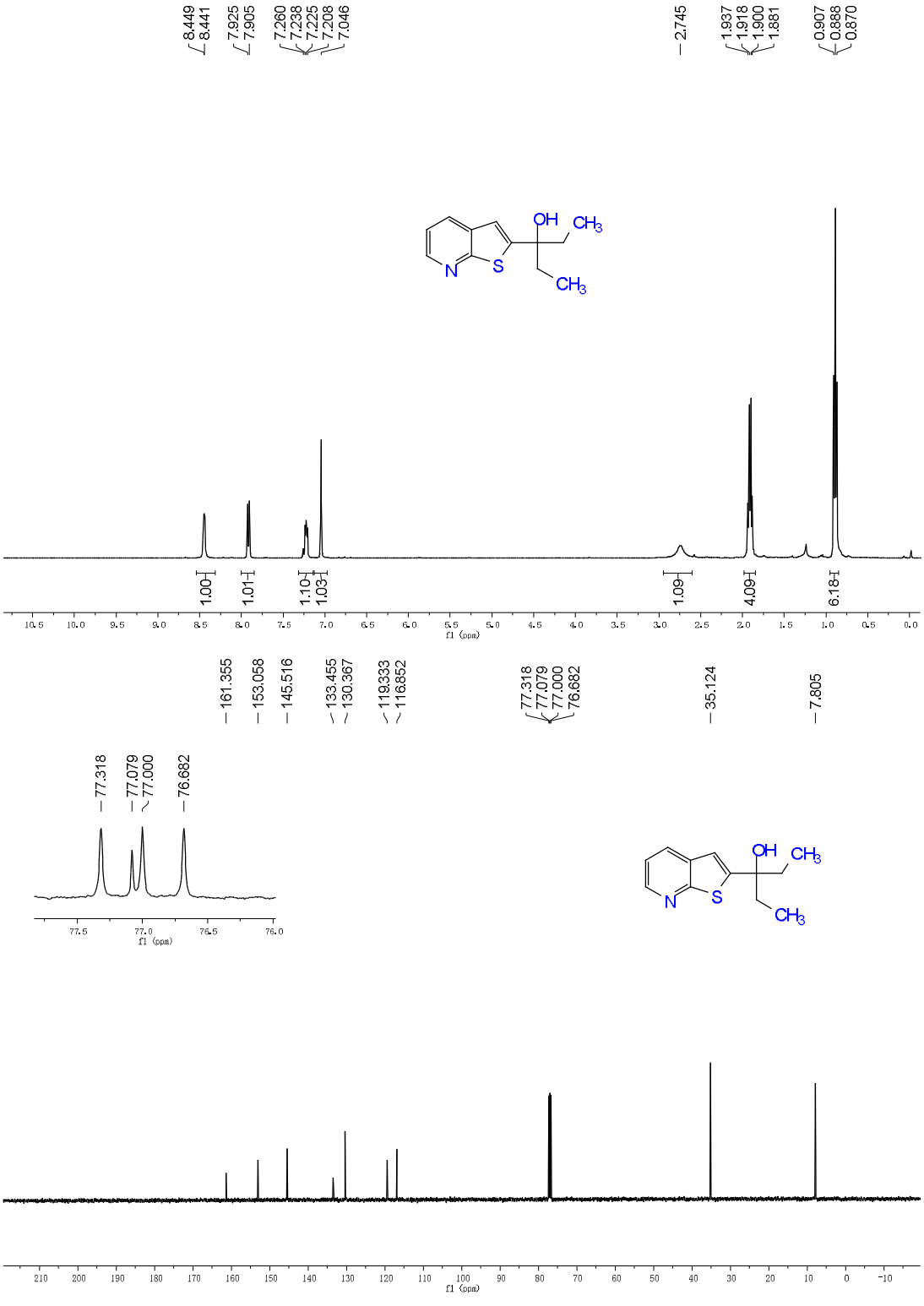

8.499
7.963
7.938
7.287
7.274
7.263
7.247
7.124
7.113
2.758
1.754
1.746
CH3
CH3
OH
N
S
1.00
1.06
1.21
1.06
1.15
6.25
f1 (ppm)

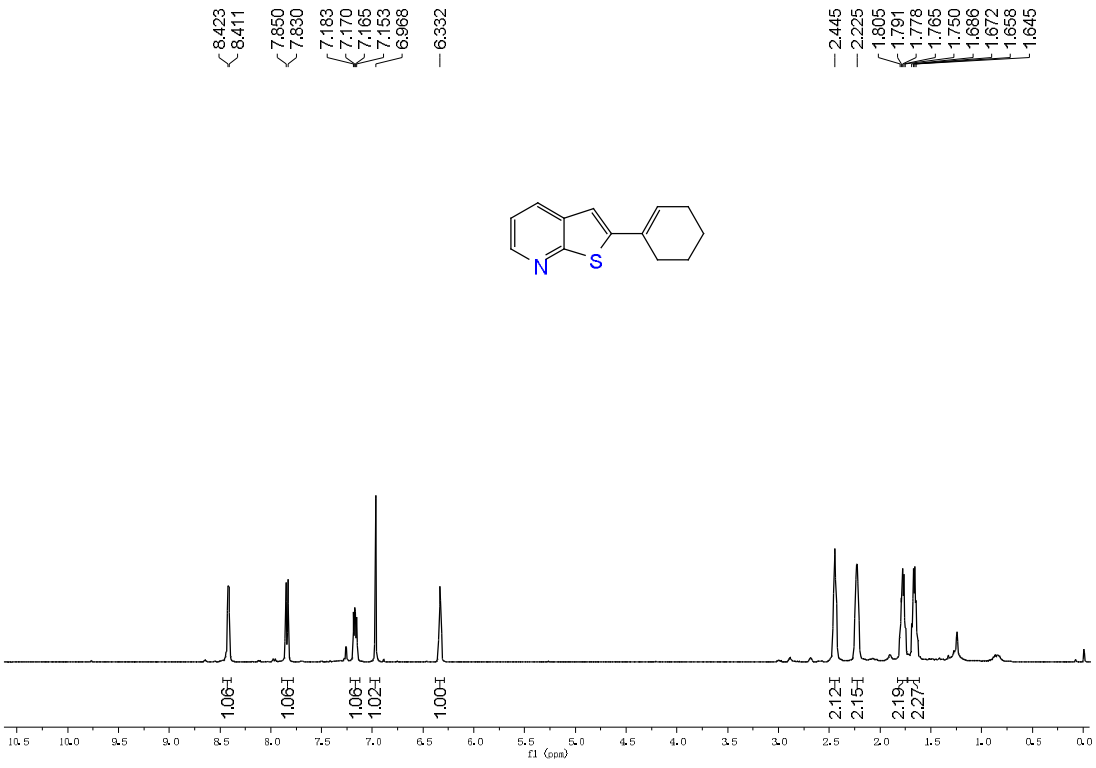

161.065
155.889
145.705
133.436
130.620
119.442
115.462
77.423
77.000
76.576
71.500
31.850
CH3
CH3
OH
N
S
f1 (ppm)

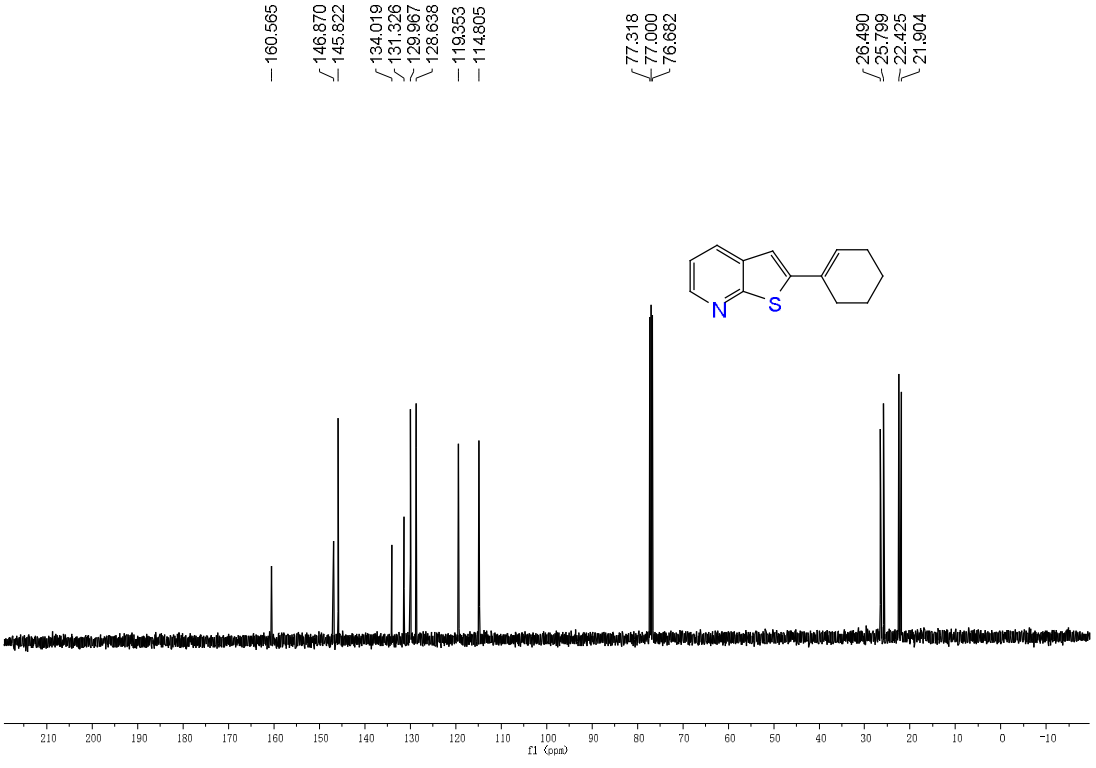

8.345
8.336
7.823
7.804
7.140
7.126
7.121
7.109
6.972
3.112
1.871
1.853
1.834
1.816
1.575
0.850
0.831
0.813
0.93
0.93
0.94
0.95
1.23
2.02
3.00
3.18
f1 (ppm)


161.112
154.839
145.503
133.442
130.483
119.326
116.093
77.318
77.000
76.681
74.153
37.005
29.434
8.338
f1 (ppm)

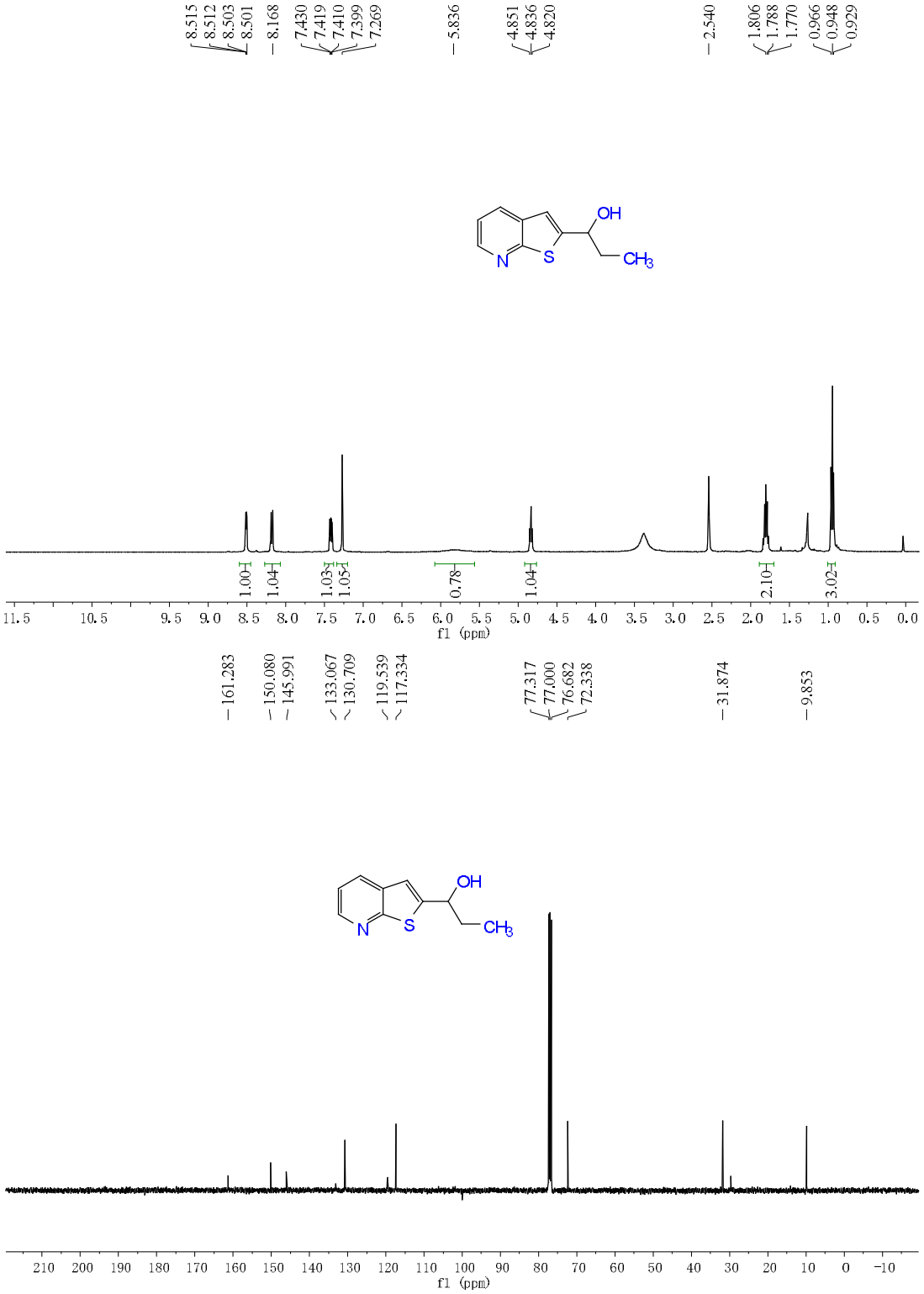
CH3
CH3
N
S
OH

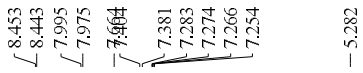

8.449
8.441
7.925
7.905
7.260
7.238
7.225
7.208
7.046
2.745
1.937
1.918
1.900
1.881
0.907
0.888
0.870
OH
CH3
CH3
N
S
1.00
1.01
1.10
1.03
1.09
4.09
6.18
f1 (ppm)
161.355
153.058
145.516
133.455
130.367
119.333
116.852
77.318
77.079
77.000
76.682
35.124
7.805
77.318
77.079
77.000
76.682
77.5
77.0
76.5
76.0
f1 (ppm)
OH
CH3
CH3
N
S
f1 (ppm)

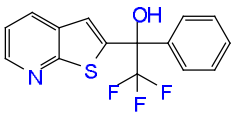

8.423
8.411
7.850
7.830
7.183
7.170
7.165
7.153
6.968
6.332
2.445
2.225
1.805
1.791
1.778
1.765
1.750
1.686
1.672
1.658
1.645
N
S
1.06
1.06
1.06
1.02
1.00
2.12
2.15
2.19
2.27
f1 (ppm)

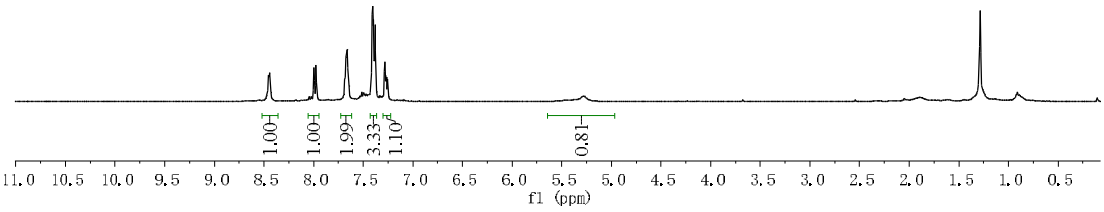

160.565
146.870
145.822
134.019
131.326
129.967
128.638
119.353
114.805
77.318
77.000
76.682
26.490
25.799
22.425
21.904
N
S
f1 (ppm)

DMSO-d6 as solvent

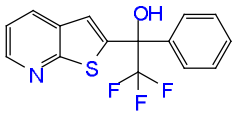


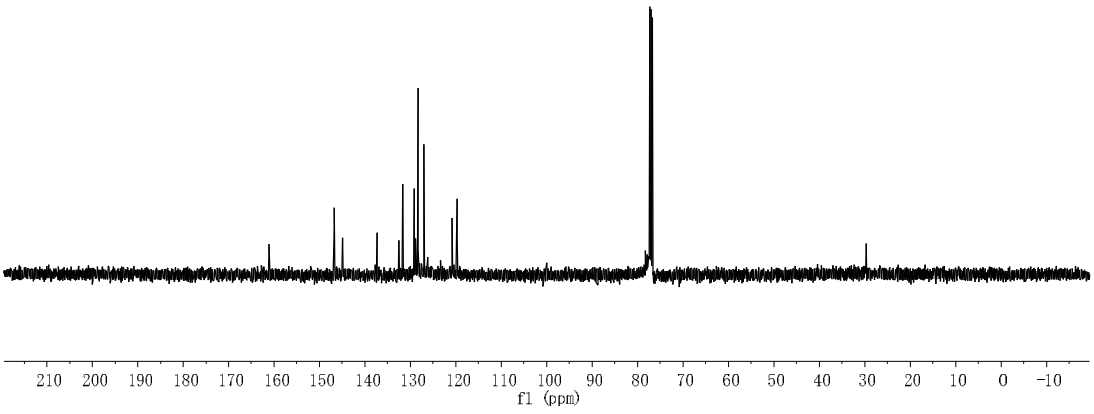

8.453
8.443
7.995
7.975
7.664
7.644
7.381
7.283
7.274
7.266
7.254
5.282

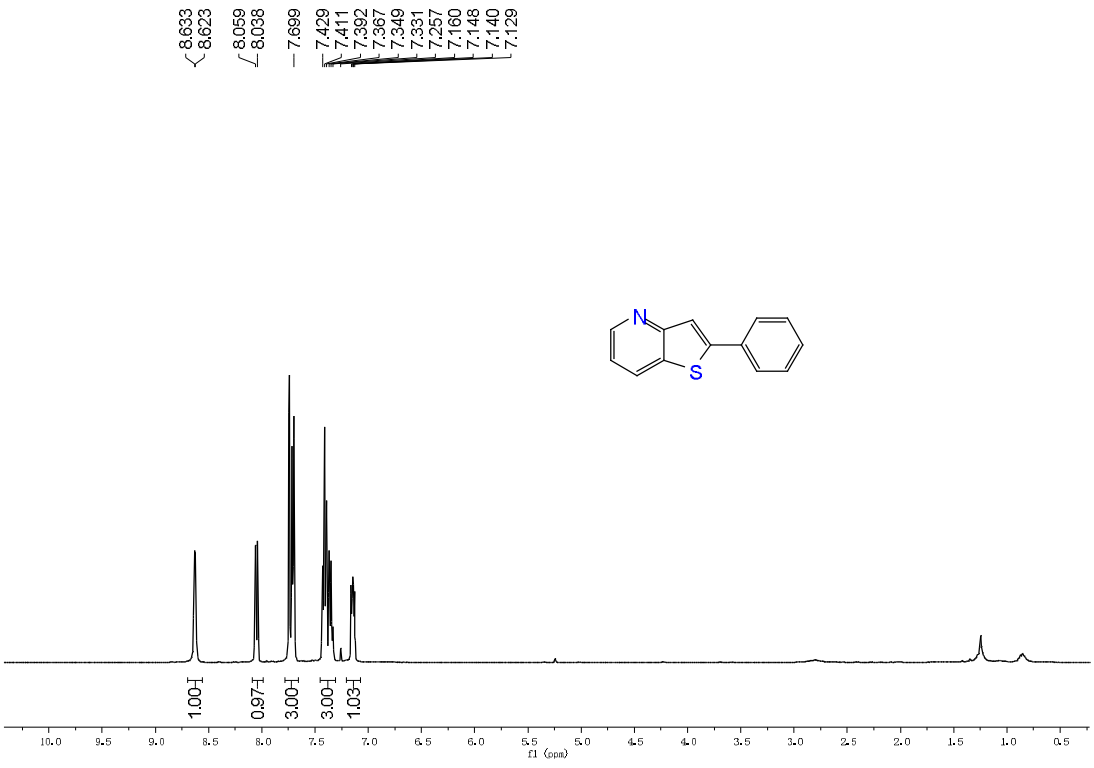

OH
N
S
F
F
F

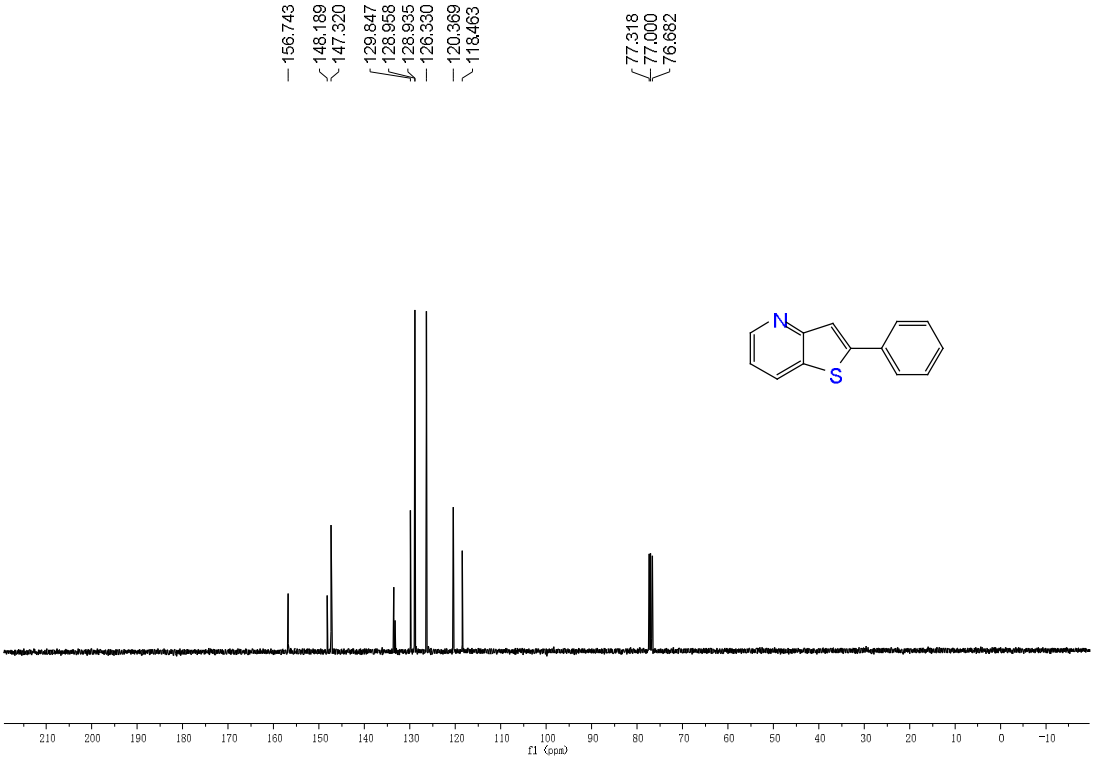

1.00
1.00
1.99
3.33
1.10
0.81
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

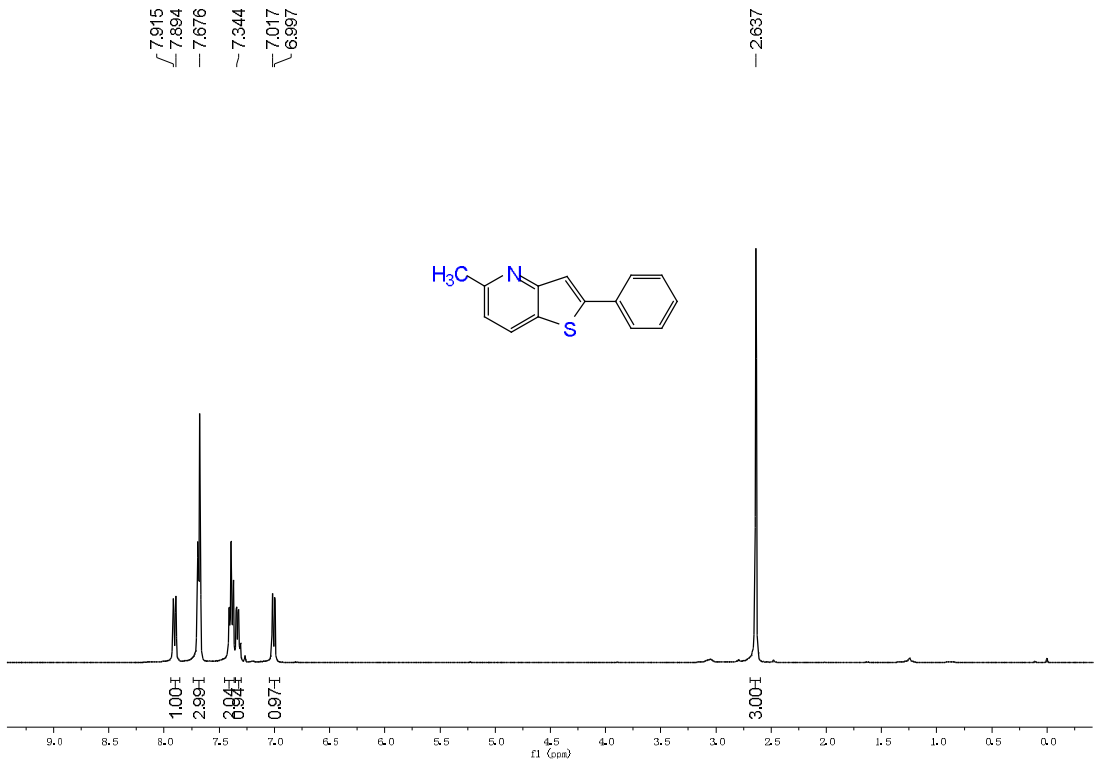

161.079
146.776
144.886
131.749
128.848
126.993
120.816
119.771
77.318
77.000
76.682
29.670

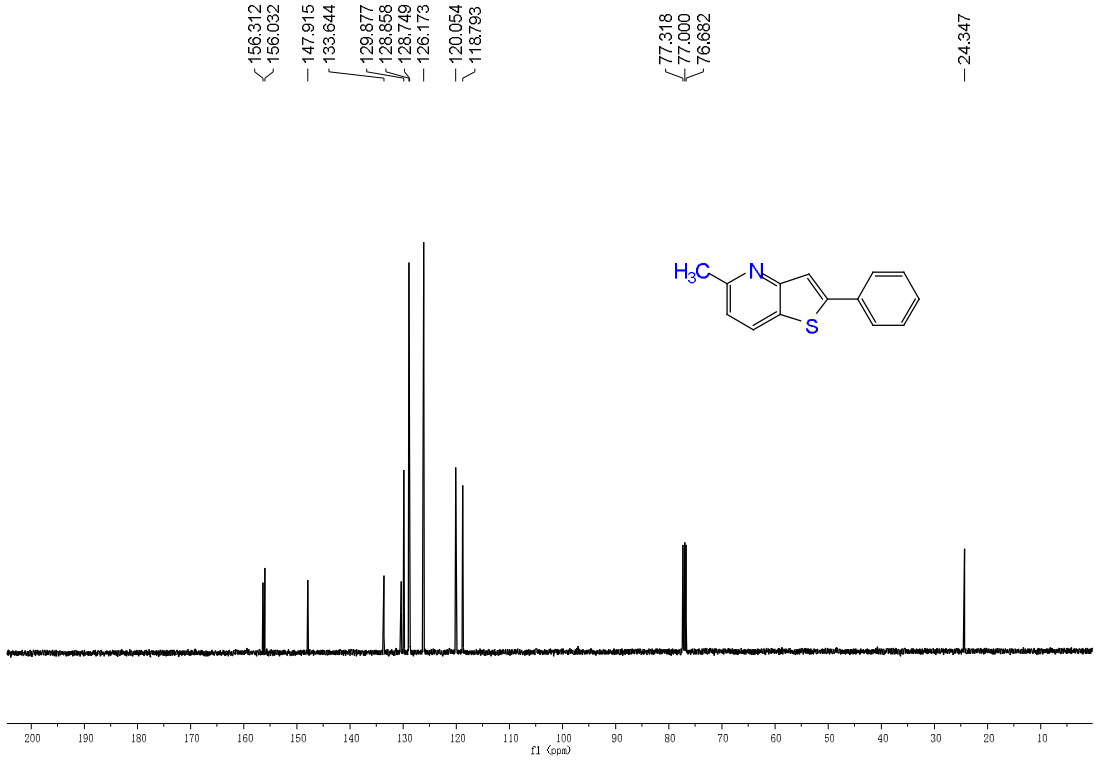

OH
N
S
F
F
F

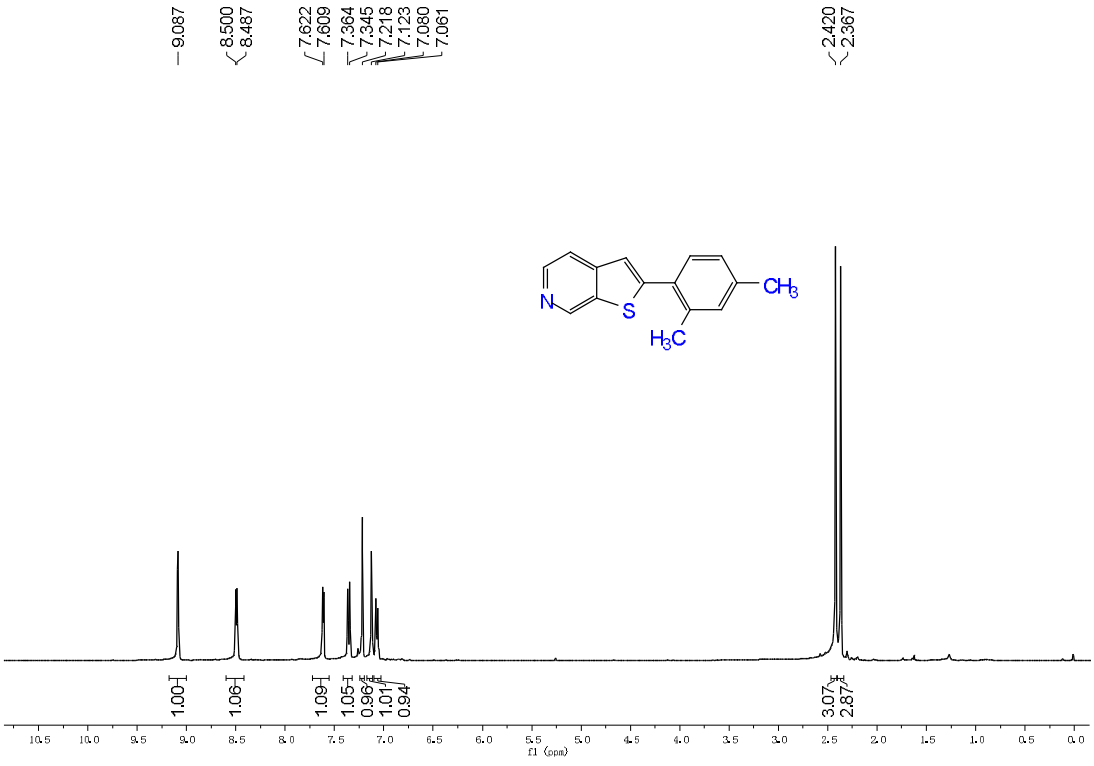

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

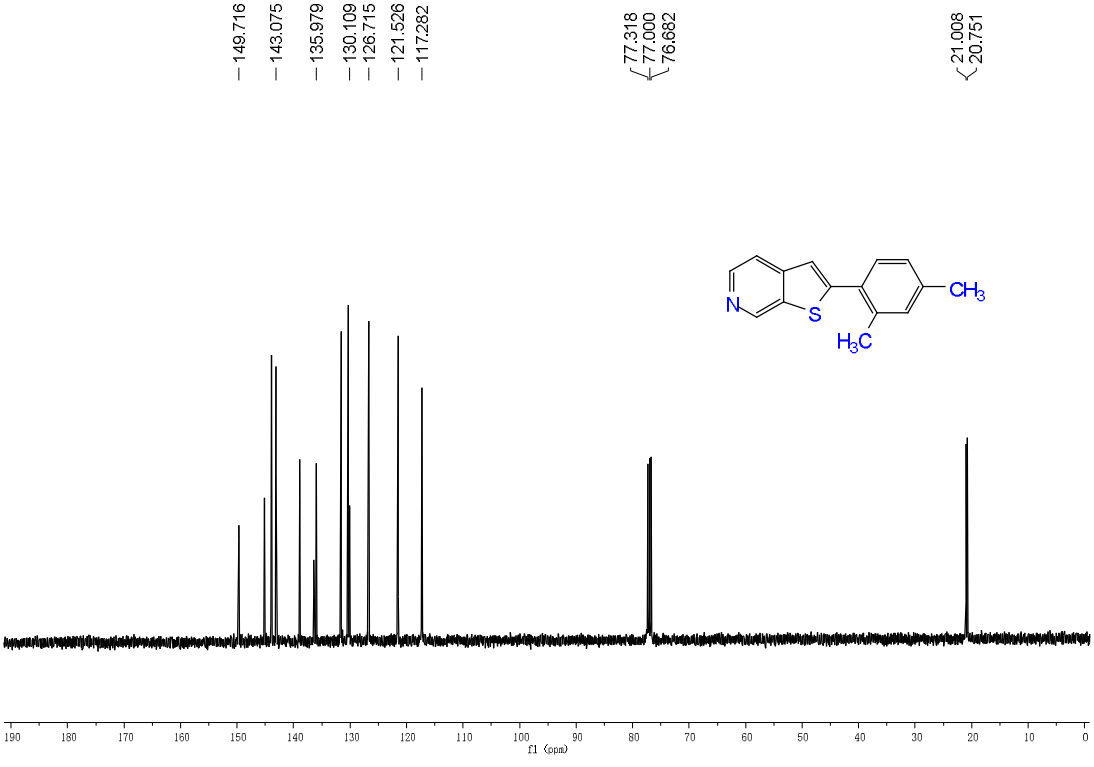

8.633
8.623
8.059
8.038
7.699
7.429
7.411
7.392
7.367
7.349
7.331
7.257
7.160
7.148
7.140
7.129
N
S
1.00
0.97
3.00
3.00
1.03
f1 (ppm)

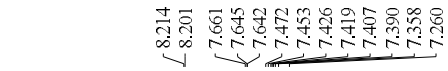

156.743
148.189
147.320
129.847
128.958
128.935
126.330
120.369
118.463
77.318
77.000
76.682
N
S
f1 (ppm)

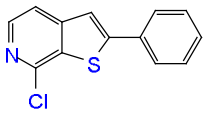

7.915
7.894
7.676
7.344
7.017
6.997
2.637
H3C
N
S
1.00
2.99
2.04
0.94
0.97
3.00
f1 (ppm)

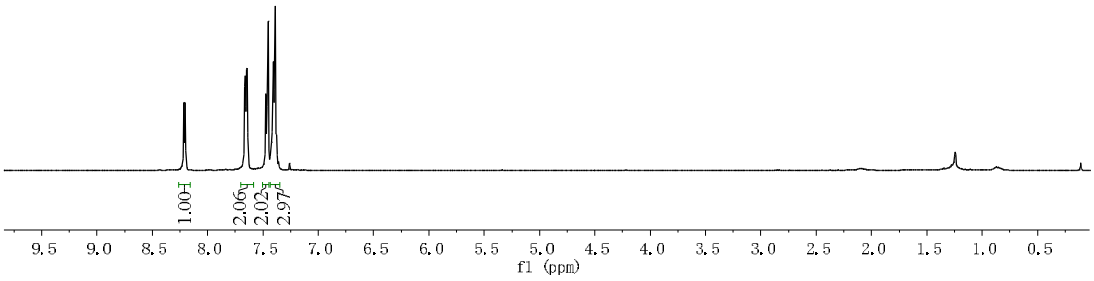

156.312
156.032
147.915
133.644
129.877
128.858
128.749
126.173
120.054
118.793
77.318
77.000
76.682
24.347
H3C
N
S
f1 (ppm)


9.087
8.500
8.487
7.622
7.609
7.364
7.345
7.218
7.123
7.080
7.061
2.420
2.367
N
S
CH3
H3C
1.00
1.06
1.09
1.05
0.96
1.01
0.94
3.07
2.87
f1 (ppm)


149.716
143.075
135.979
130.109
126.715
121.526
117.282
77.318
77.000
76.682
21.008
20.751
N
S
CH3
H3C
f1 (ppm)

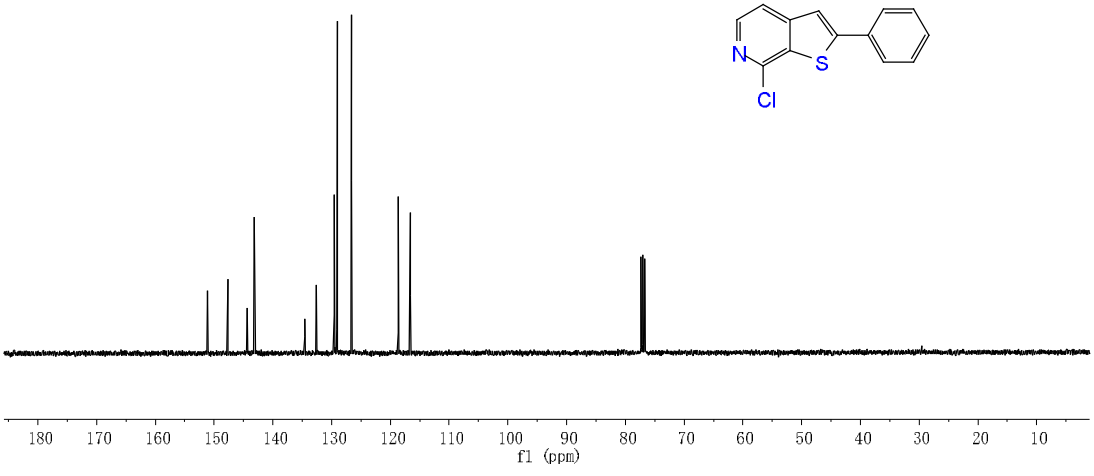

8.214
8.201
7.661
7.645
7.642
7.472
7.453
7.426
7.419
7.407
7.390
7.358
7.260

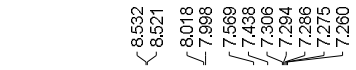

N
S
Cl

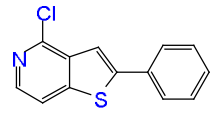

1.00
2.06
2.02
2.97
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

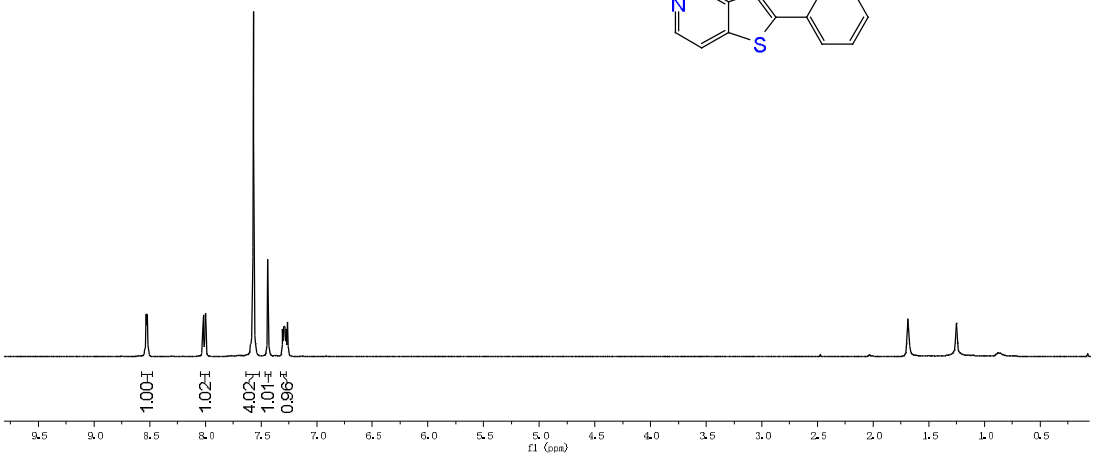

151.094
147.669
144.413
143.162
132.587
129.574
129.043
126.655
118.677
116.633
77.317
77.000
76.682

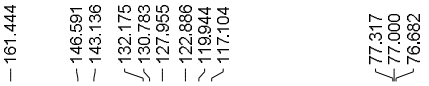
N
S
Cl

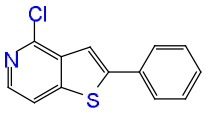

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

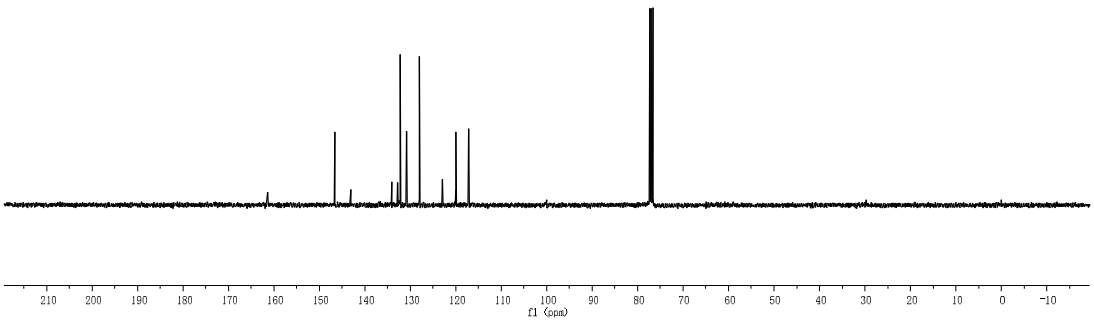

8.532
8.521
8.018
7.998
7.569
7.438
7.306
7.294
7.286
7.275
7.260

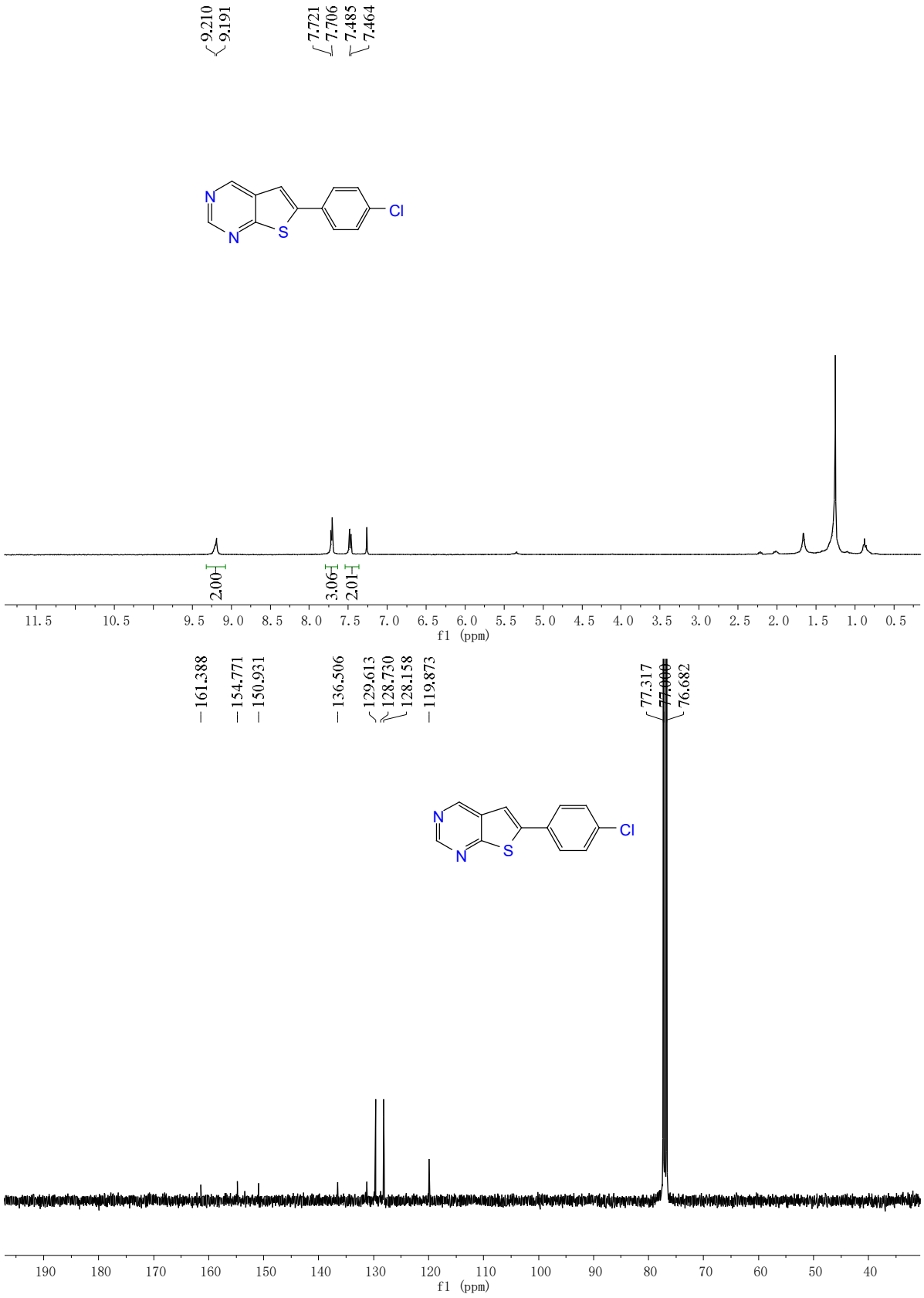

Cl
N
S

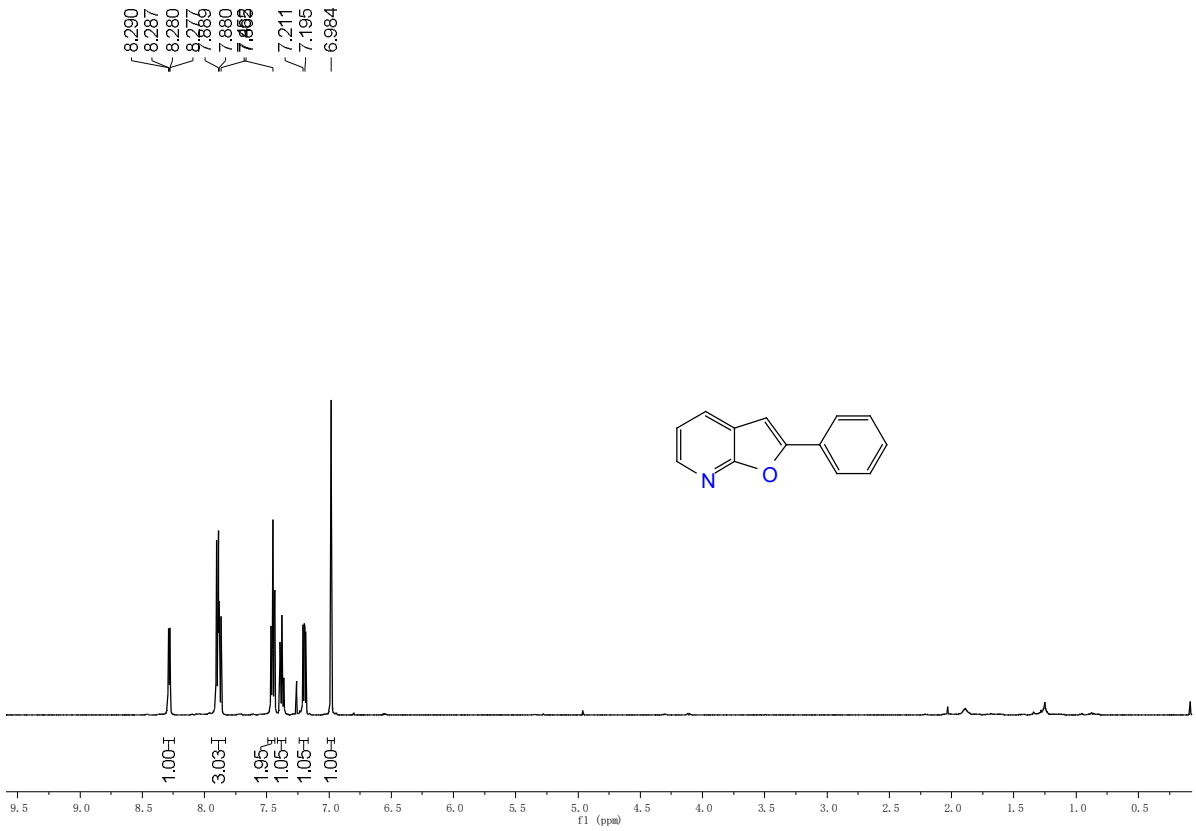

1.00
1.02
4.02
1.01
0.96
f1 (ppm)

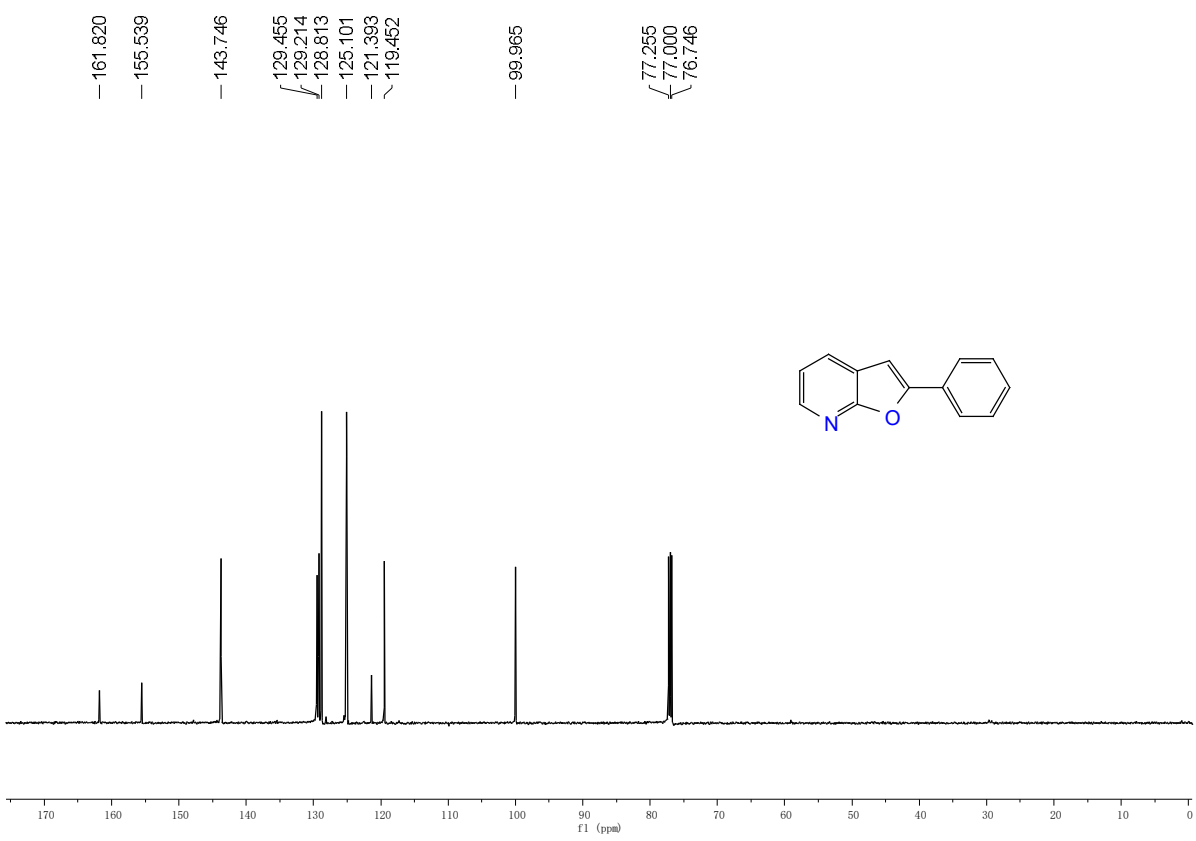

161.444
146.591
143.136
132.175
130.783
127.955
122.886
119.944
117.104
77.317
77.000
76.682

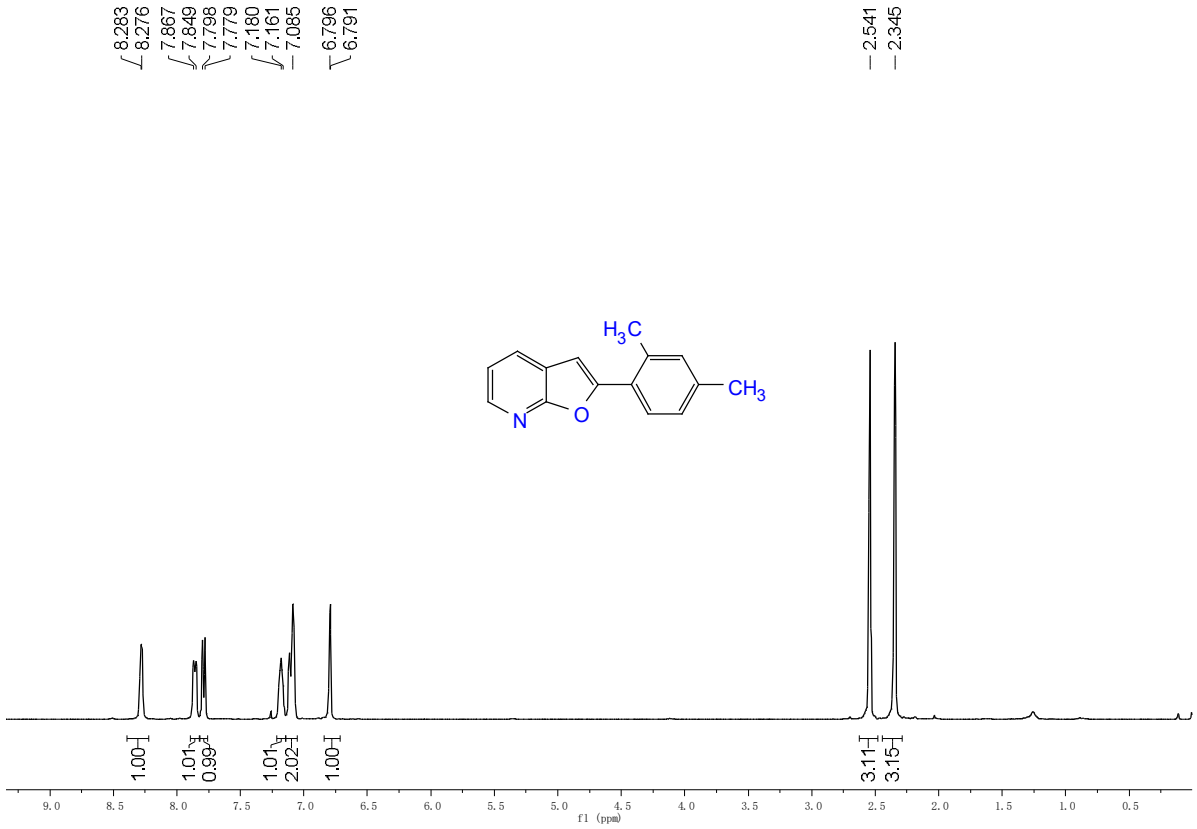

Cl
N
S

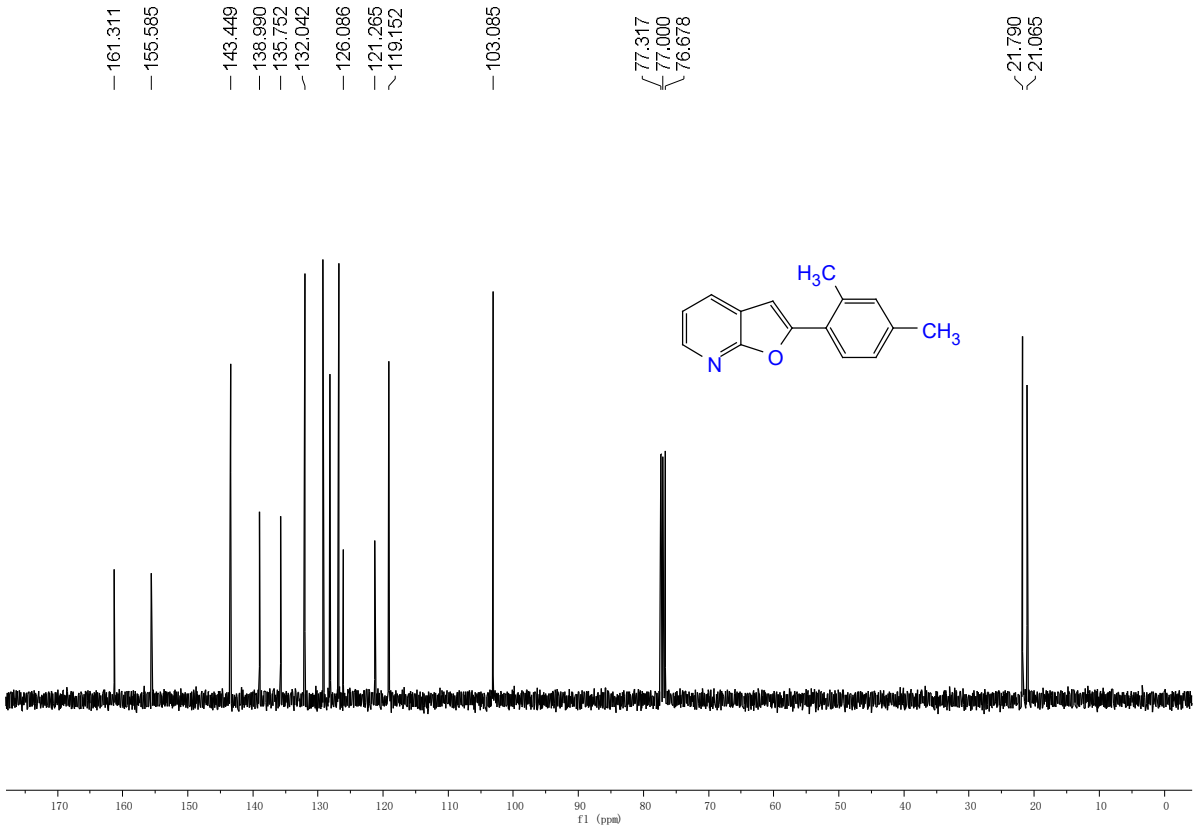

f1 (ppm)

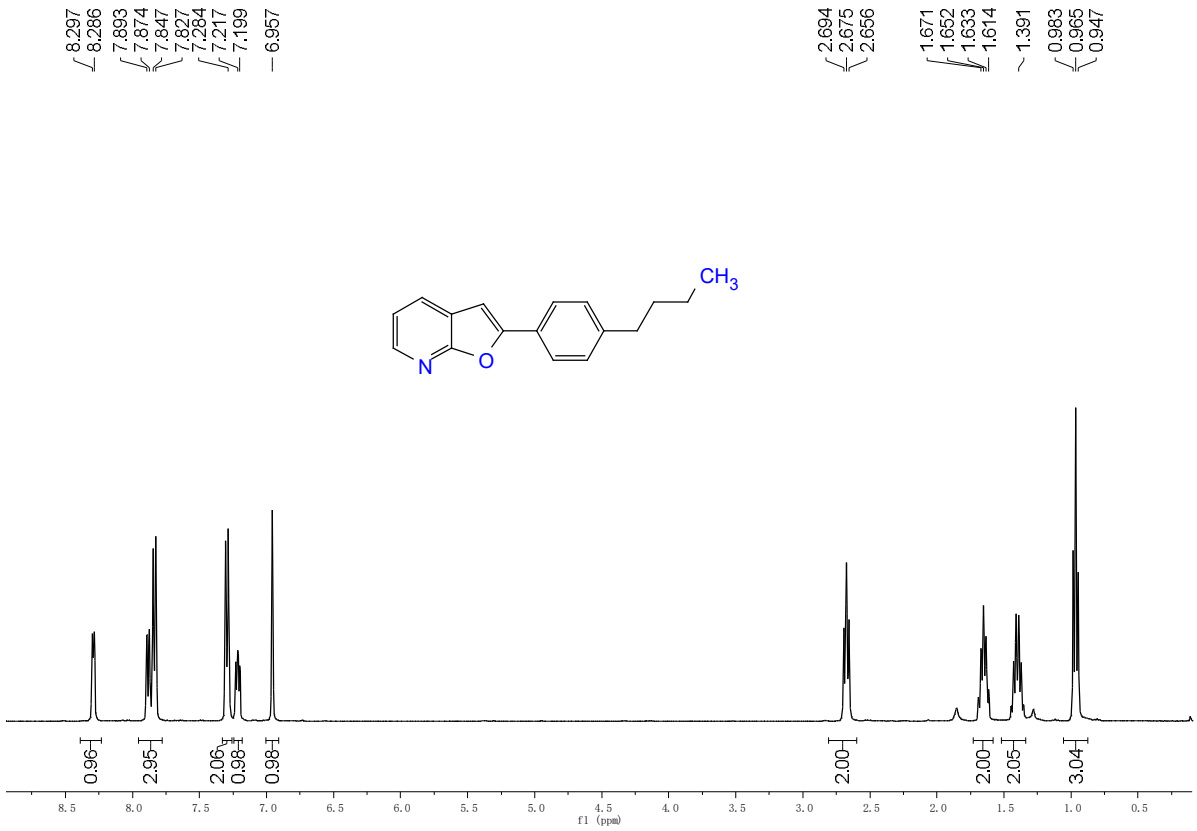

9.210
9.191
7.721
7.706
7.485
7.464
N
S
N
Cl
2.00
3.06
2.01
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
161.388
154.771
150.931
136.506
129.613
128.730
128.158
119.873
77.317
77.000
76.682
N
S
N
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
f1 (ppm)

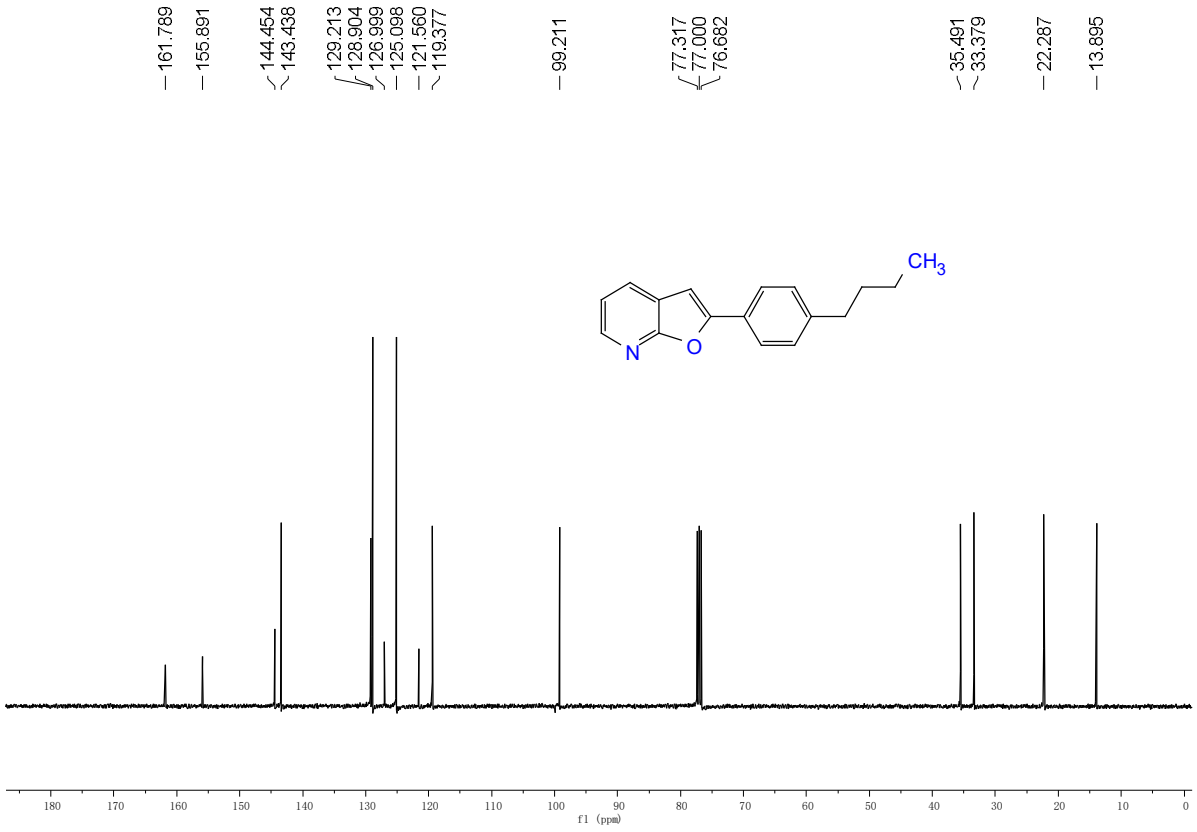

8.290
8.287
8.280
8.277
7.889
7.880
7.211
7.195
6.984
1.00
3.03
1.95
1.05
1.05
1.00
f1 (ppm)

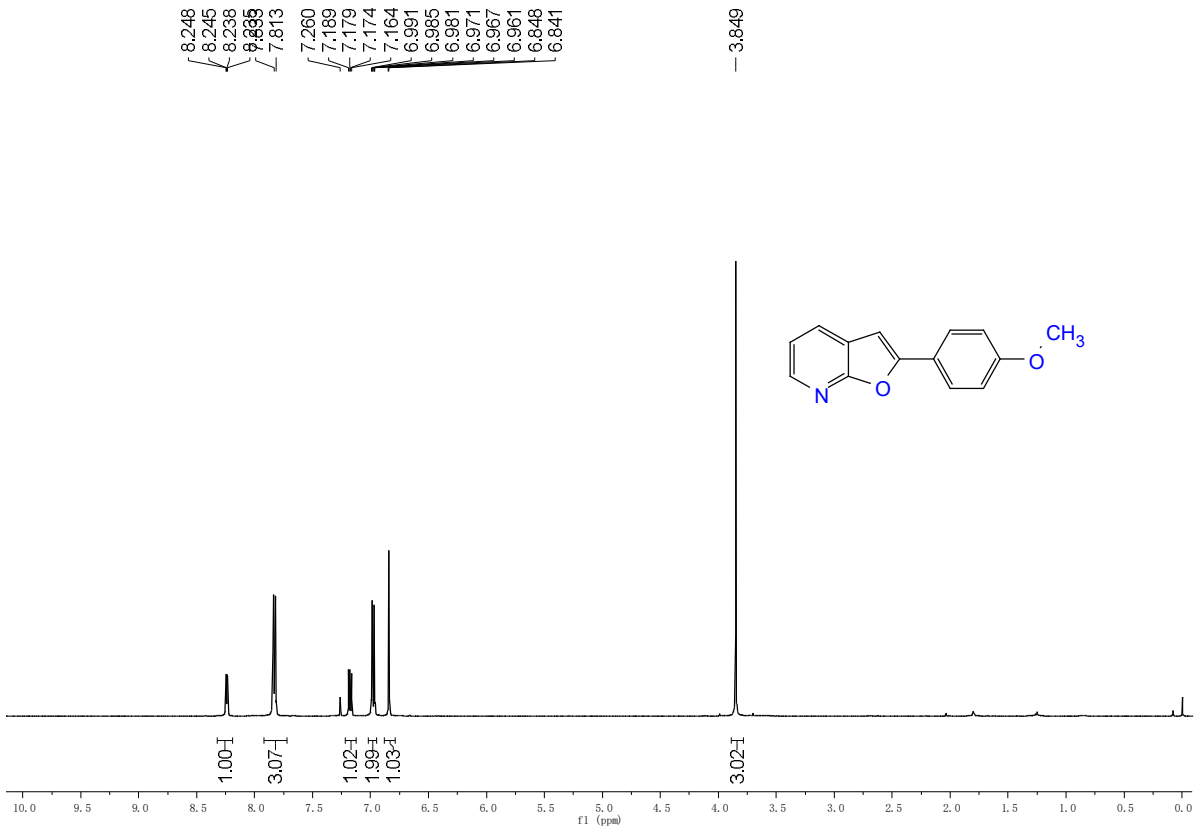

161.820
155.539
143.746
129.455
129.214
128.813
125.101
121.393
119.452
99.965
77.255
77.000
76.746
f1 (ppm)

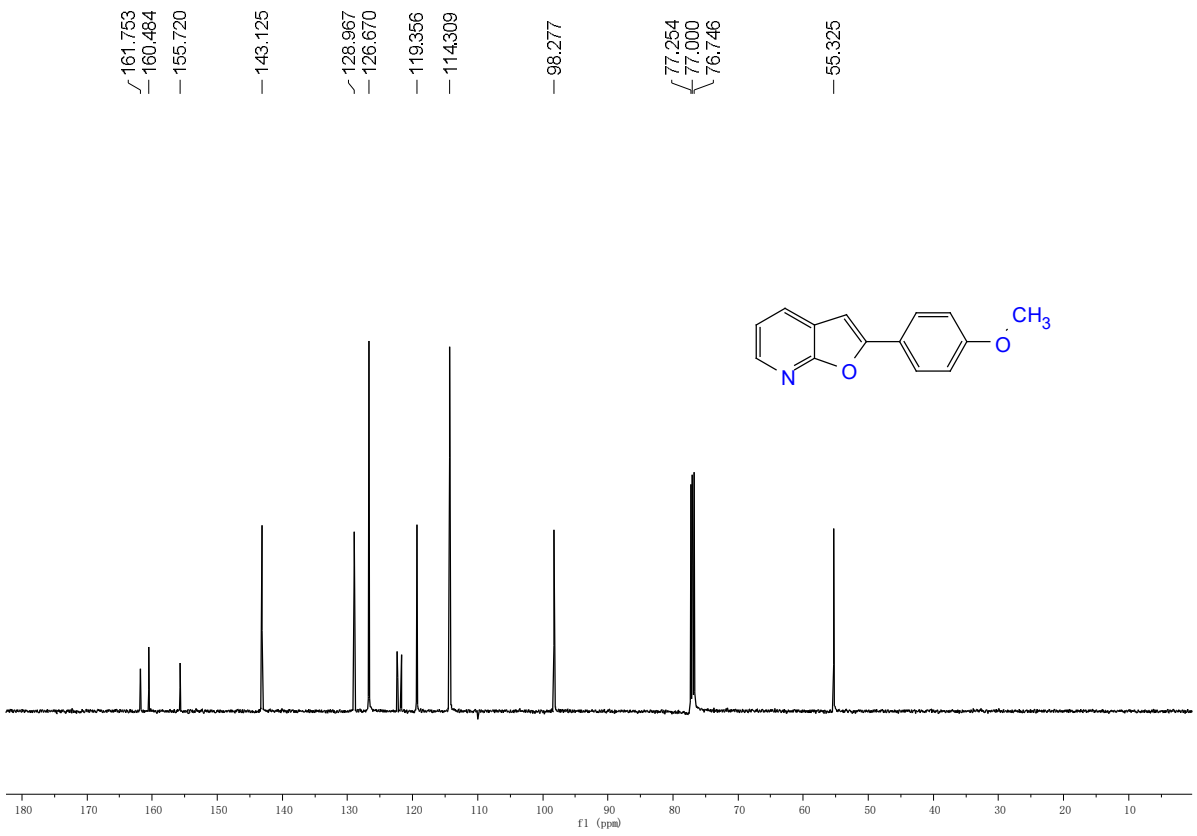

8.283
8.276
7.867
7.849
7.798
7.779
7.180
7.161
7.085
6.796
6.791
2.541
2.345
H3C
CH3
N
O
1.00
1.01
0.99
1.01
2.02
1.00
3.11
3.15
f1 (ppm)

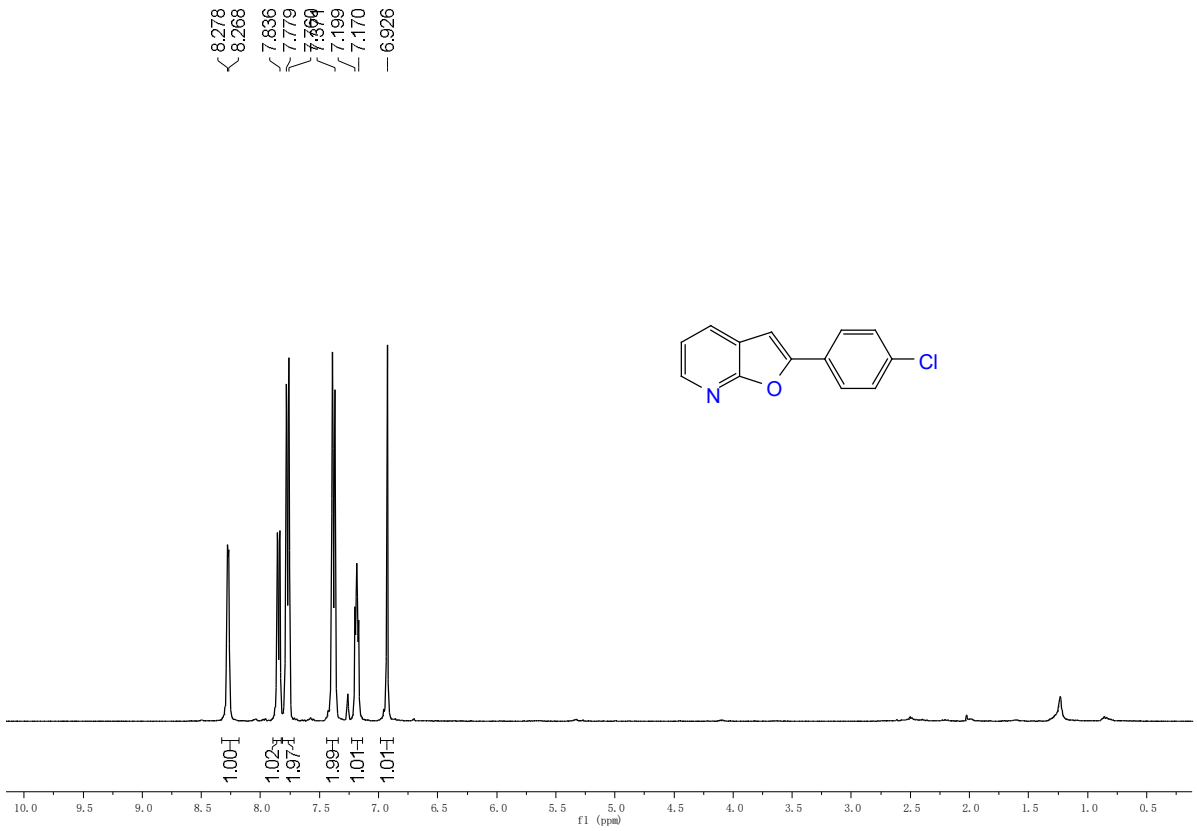

161.311
155.585
143.449
138.990
135.752
132.042
126.086
121.265
119.152
103.085
77.317
77.000
76.678
21.790
21.065
H3C
CH3
N
O
f1 (ppm)

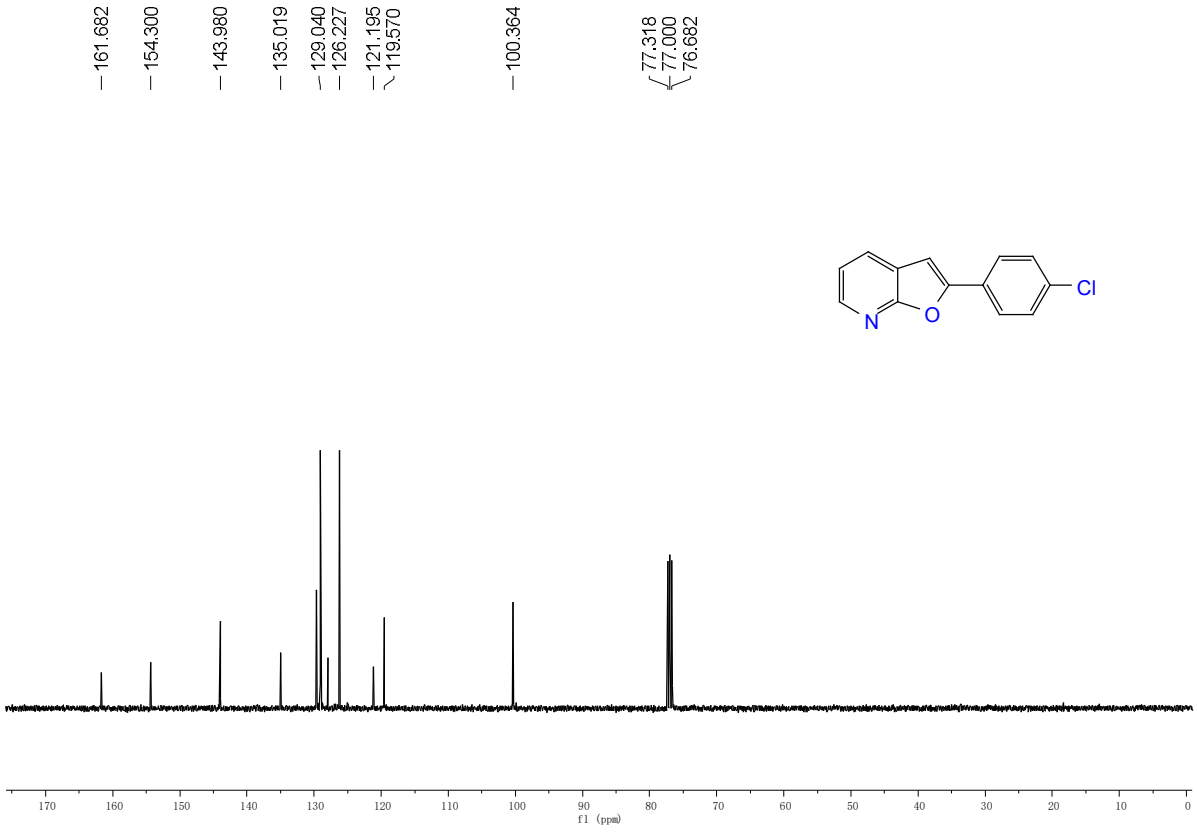

8.297
8.286
7.893
7.874
7.847
7.827
7.284
7.217
7.199
6.957
2.694
2.675
2.656
1.671
1.652
1.633
1.614
1.391
0.983
0.965
0.947
CH3
N
O
0.96
2.95
2.06
0.98
0.98
2.00
2.00
2.05
3.04
f1 (ppm)

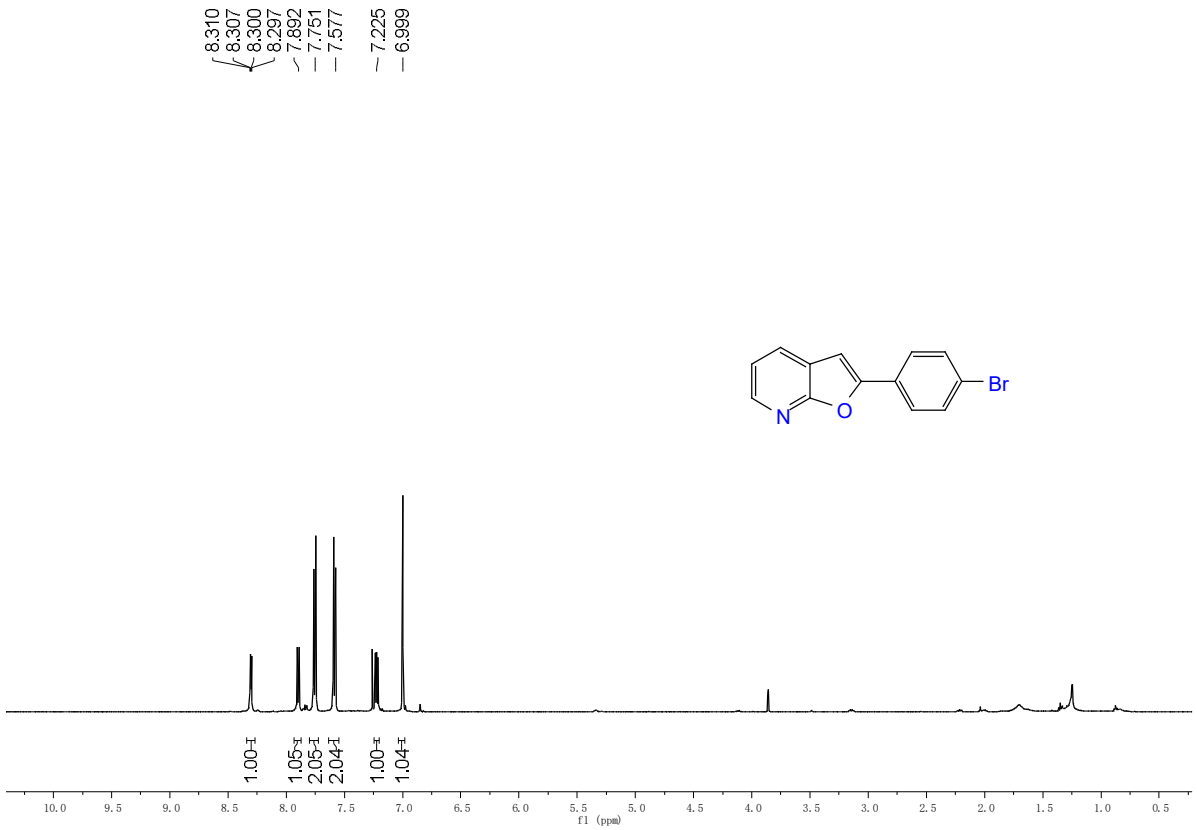

161.789
155.891
144.454
143.438
129.213
128.904
126.999
125.098
121.560
119.377
99.211
77.317
77.000
76.682
35.491
33.379
22.287
13.895
CH3
N
O
f1 (ppm)

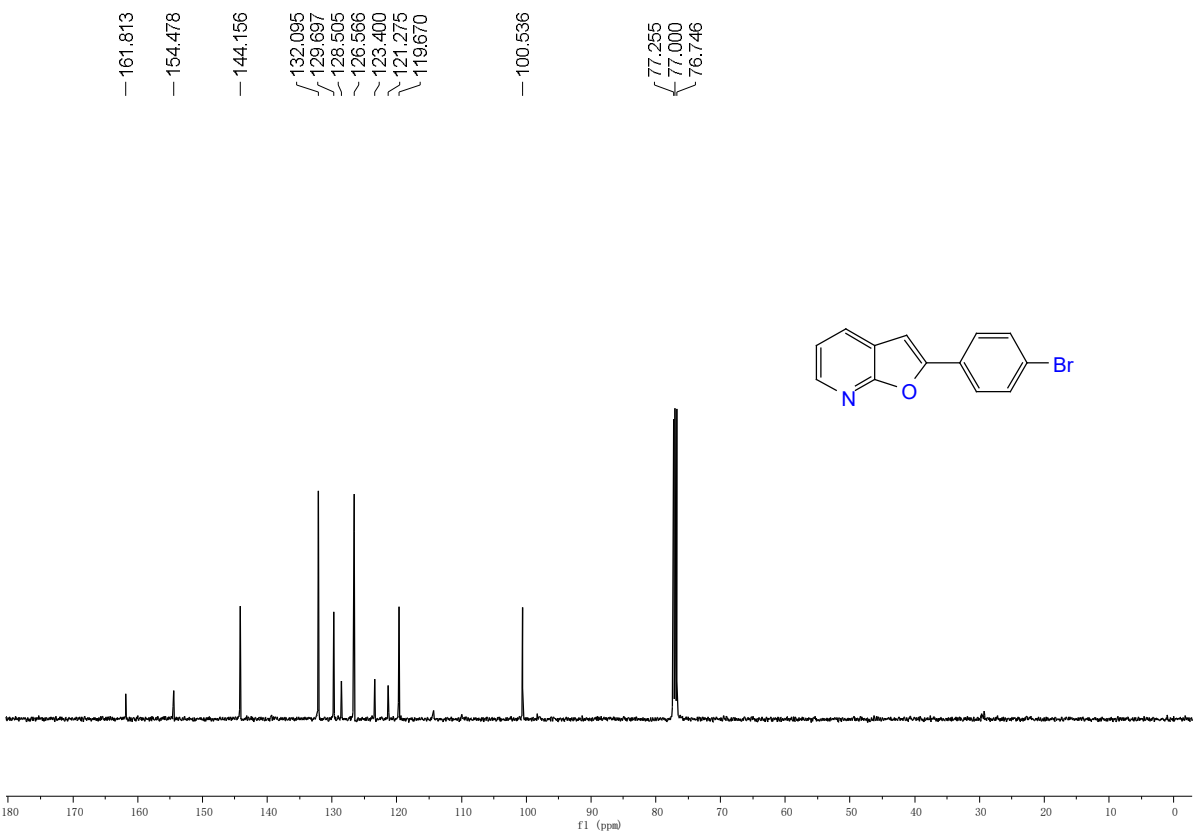

8.248
8.245
8.238
7.813
7.260
7.189
7.179
7.174
7.164
6.991
6.985
6.981
6.971
6.967
6.961
6.848
6.841
3.849
1.00
3.07
1.02
1.99
1.03
3.02
CH3
O
N
O
f1 (ppm)

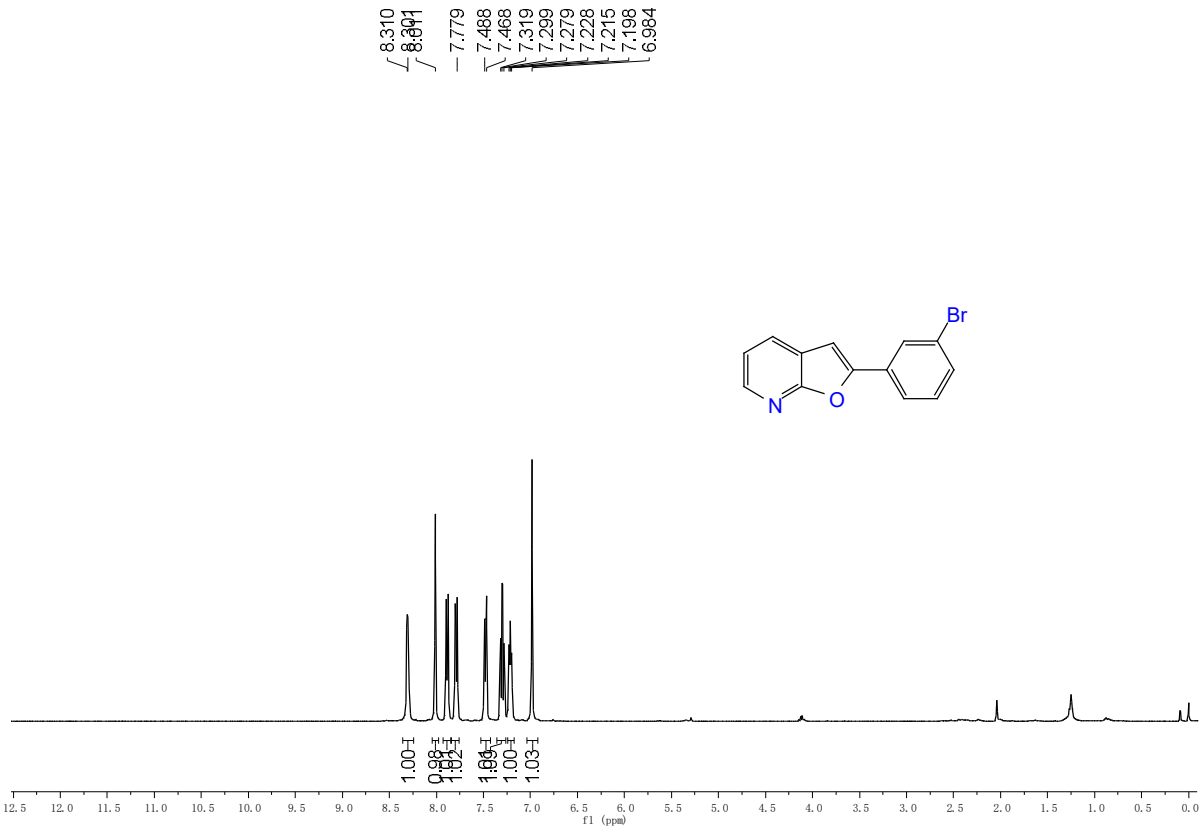

161.753
160.484
155.720
143.125
128.967
126.670
119.356
114.309
98.277
77.254
77.000
76.746
55.325
CH3
O
N
O
f1 (ppm)

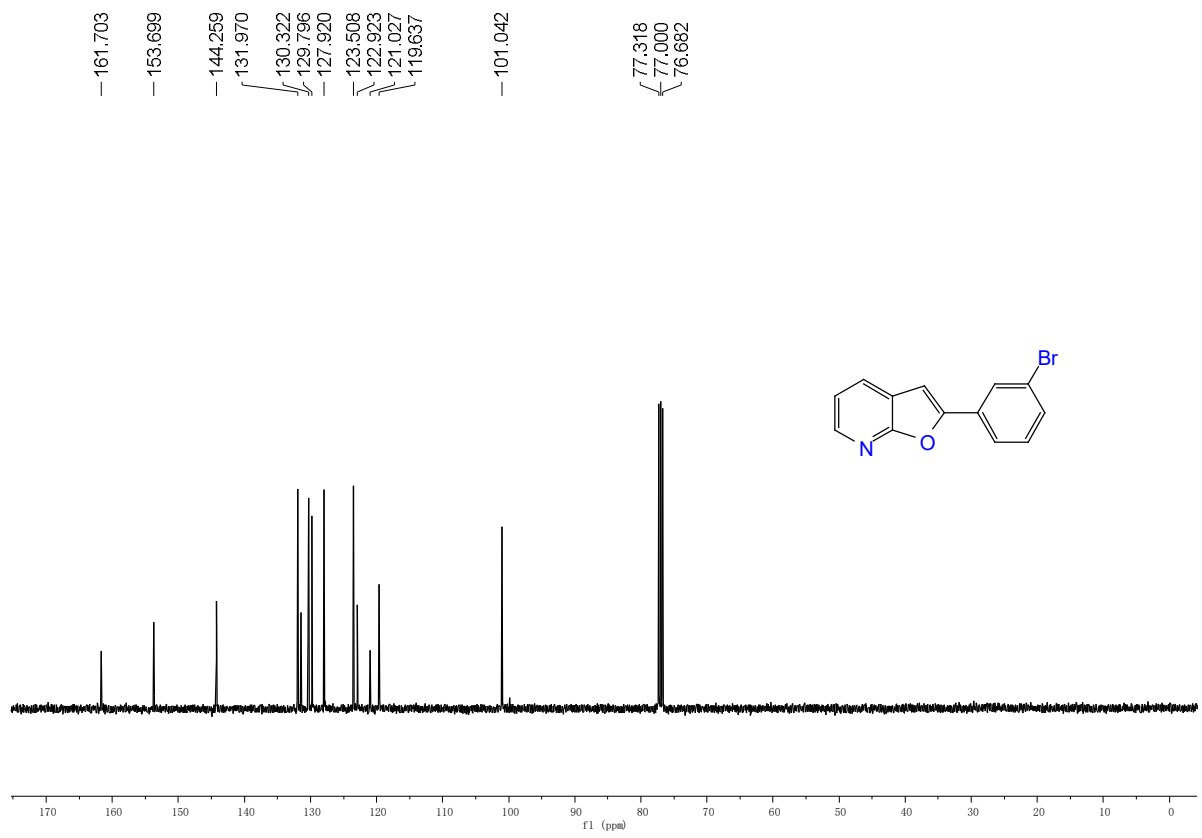

8.278
8.268
7.836
7.779
7.199
7.170
6.926
Cl
N
O
1.00
1.02
1.97
1.99
1.01
1.01
f1 (ppm)


161.682
154.300
143.980
135.019
129.040
126.227
121.195
119.570
100.364
77.318
77.000
76.682
Cl
N
O
f1 (ppm)


8.310
8.307
8.300
8.297
7.892
7.751
7.577
7.225
6.999
Br
N
O
1.00
1.05
2.05
2.04
1.00
1.04
f1 (ppm)


161.813
154.478
144.156
132.095
129.697
128.505
126.566
123.400
121.275
119.670
100.536
77.255
77.000
76.746
Br
N
O
f1 (ppm)


8.310
8.301
8.301
7.779
7.488
7.468
7.319
7.299
7.279
7.228
7.215
7.198
6.984
Br
N
O
1.00
0.98
1.02
1.01
1.00
1.03
f1 (ppm)


161.703
153.699
144.259
131.970
130.322
129.796
127.920
123.508
122.923
121.027
119.637
101.042
77.318
77.000
76.682
Br
N
O
f1 (ppm)