Supporting Information

Visible Light-induced Sulfonylation Reaction of Copper-catalyzed Thianthrenium Salts

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1 General Information

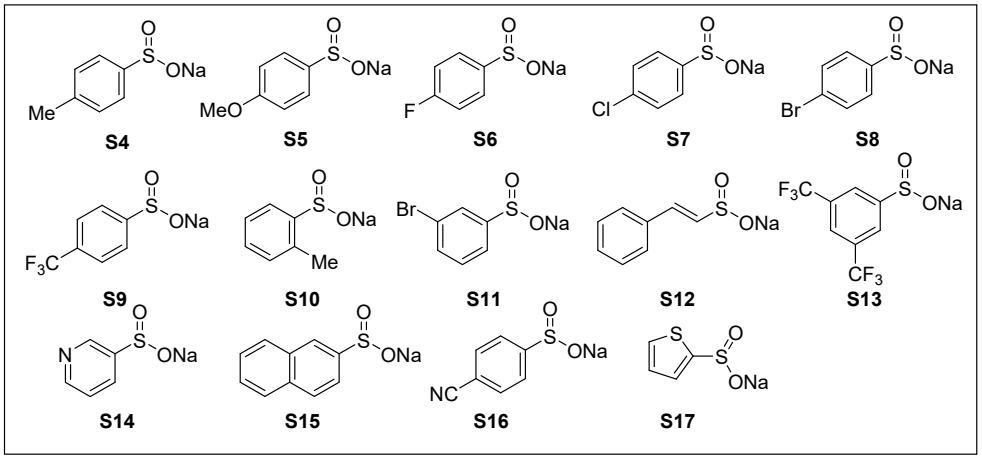
A. Materials:

All reactions were carried out in oven-dried Schlenk tubes under argon atmosphere (purity≥99.999%) unless otherwise mentioned. Other commercial reagents were purchased from Adamas-beta, TCI and Aldrich and used without further purification or degassed. Organic solutions were concentrated under reduced pressure on Buchi rotary evaporator. Flash column chromatographic purification of products was accomplished using forced-flow chromatography on Silica Gel (200-300 mesh). The blue LED lamps were purchased from Kessil company (PR160–395 nm, 427 nm, 440 nm, 456 nm). The Photo Reaction Setup was purchased from Anhui kemi machinery technology Co., Ltd. The material of the irradiation vessel if other than borosilicate glass.

B. Analytical Methods:

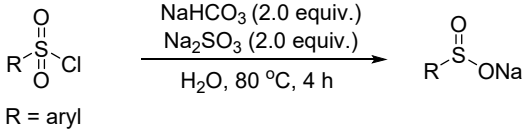
^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Data for ^{1}H-NMR are reported as follows: chemical shift (ppm, scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiplet resonances, br = broad), coupling constant (Hz), and integration. Data for ^{13}C-NMR are reported in terms of chemical shift (ppm, scale), multiplicity, and coupling constant (Hz). HRMS analysis was performed on Finnigan LCQ advantage Max Series MS System. ESI-mass data or EI-mass data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI or EI source and controlled by Xcalibur software.

2 Procedure of Substrates



2.1 General procedure for preparation of Sodium sulfinates

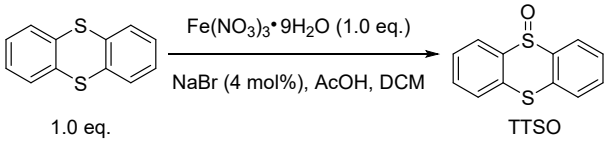
Sodium sulfinates S4-S12, S14-S17 are known compounds and were prepared according to the literature procedures 1[1], Sodium sulfinate S13 was prepared according to the literature procedures 2[2].



General Procedure 1: Sodium sulfite (4.0 mmol, 2.0 equiv.), sodium bicarbonate (4.0 mmol, 2.0 equiv.), and the corresponding aryl sulfonyl chloride (2.0 mmol, 1.0 equiv.) were dissolved in distilled water (10.0 mL). The reaction mixture was stirred for 4 h at 80 °C using an oil bath. After cooling down to room temperature, water was removed in vacuo. Ethanol (25.0 mL) was then added to this white residue and the resulting heterogeneous solution was filtered. The filtrate was concentrated under reduced pressure and the desired sodium sulfinates were obtained as white crystalline powders in 82-96% yields.

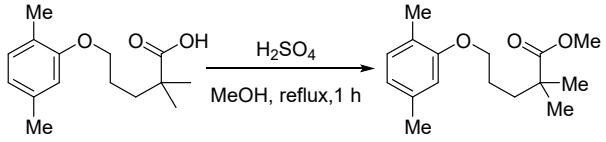
General Procedure 2: Sulfonyl chloride (5.00 mmol) was added to a solution of sodium sulfite (1.26 g, 10.0 mmol) and sodium bicarbonate (840 mg, 10.0 mmol) in water (5.0 mL). The suspension was then heated at 80 °C for 4 h. After cooling to room temperature, water was removed in vacuo. The resultant solid was stirred in EtOH (5 mL) for 30 min at 40 °C, then filtered and washed with warm EtOH (3 × 10 mL). The combined ethanol washes were concentrated in vacuo to yield the sulfinate salts.

2.2 General procedure for preparation of thianthrene *S*-oxide[3]

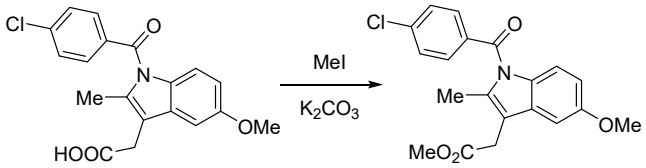


General Procedure 3: According to the modified procedure, 500 mL round-bottom flask was charged with thianthrene (21.6 g, 100 mmol, 1.0 equiv.), $Fe(NO_3)_3 \cdot 9H_2O$ (40.4 g, 100 mmol, 1.0 equiv.) and NaBr (0.4 g, 4 mmol, 4.0 mol%), DCM (200 mL) and AcOH (4.5 mL) were then injected. The reaction mixture was stirred at room temperature and monitored by TLC until thianthrene was consumed. After that, the reaction was dilute with DCM, and then washed with water. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under vacuum, giving thianthrene *S*-oxide in 98% yield.

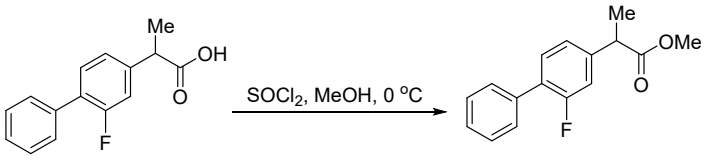
2.3 General procedure of esterification



General Procedure 4:[4] The carboxylic acid (1.0 equiv.) was dissolved in MeOH (0.05 M) and a drop of H_2SO_4 was added. The mixture was refluxed for 1 hour, cooled to room temperature and evaporated. Et_2O (10 mL) was added and the organic layer was washed with $NaHCO_3$ (10 mL). The organic layer was dried ($MgSO_4$), filtered and evapoarted to give the methyl ester. No further purification was required.

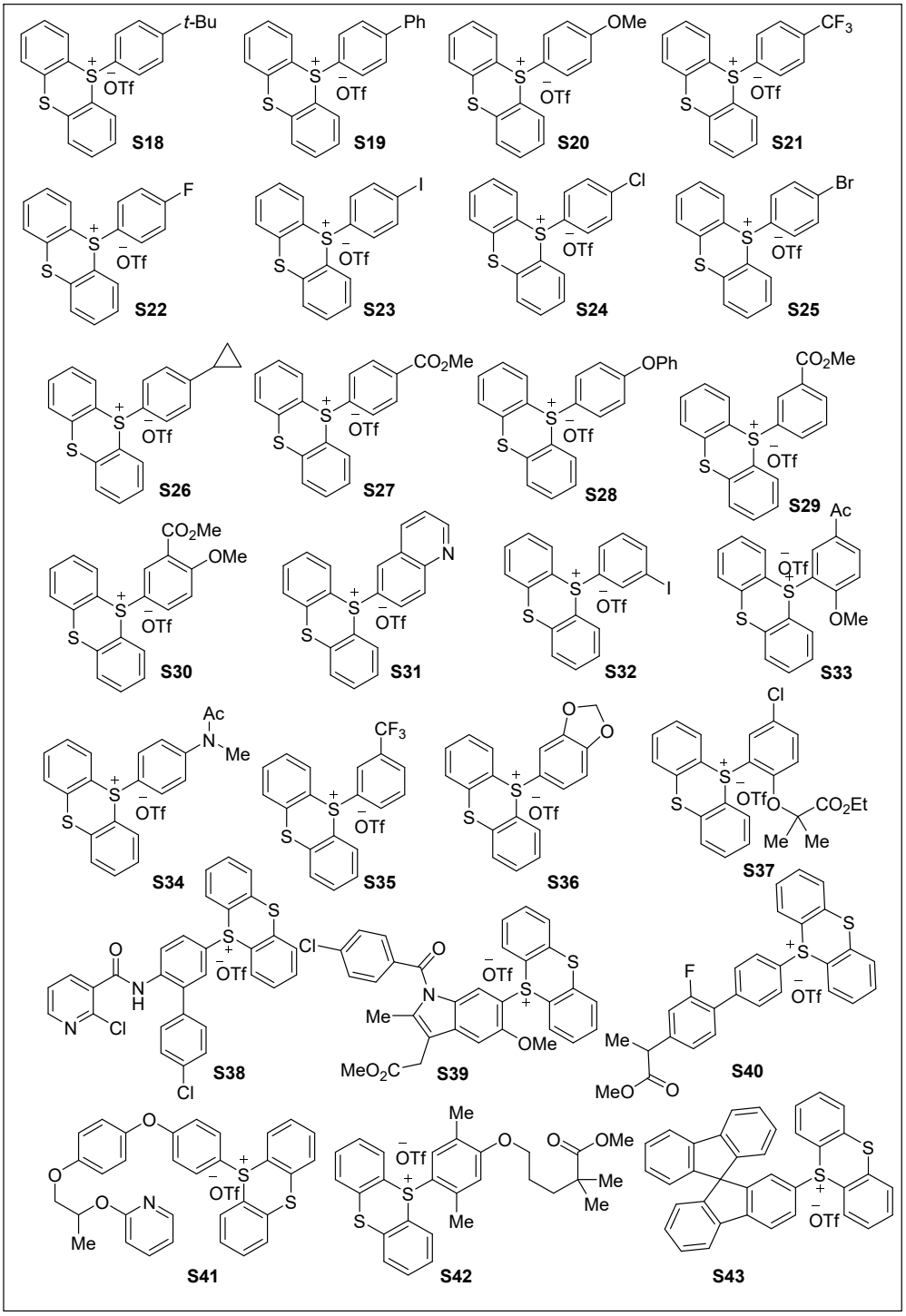


General Procedure 5:[5] To a 50 mL round bottom flask equipped with a stir bar was added indomethacin (Sigma-Aldrich I7378) (4.0 mmol) and K_2CO_3 (6.0 mmol, 1.5 equiv.), and acetone (8 mL). Iodomethane (TCI 10060) (8 mmol, 2 equiv.) was then added at 0 °C. The reaction mixture was warmed at 60 °C with an oil bath and stirred for 5 h. After being cooled to room temperature, the reaction mixture was filtered through a pad of celite and the filtrate was concentrated in vacuo. The resulting crude residue was purified by flash column chromatography (silica gel, hexane: ethyl acetate = 80: 20) to give methyl ester in 92% yield as a white solid.



General Procedure 6:[6] A 100 mL round-bottomed flask equipped with a Teflon-coated magnetic stirring bar was charged with flurbiprofen (1.22 g, 5.00 mmol, 1.00 equiv.). The solid was dissolved in MeOH (33 mL, c = 0.15 M) and the solution was cooled to 0 °C with an ice bath. Thionyl chloride (0.73 mL, 10 mmol, 2.0 equiv.) was added at 0 °C. The reaction was allowed to warm to 25 °C and stirred for 2 h at 25 °C. Saturated aqueous $NaHCO_3$ solution (10 mL) was added to the reaction mixture, and MeOH was evaporated under reduced pressure. The resulting mixture was extracted with EtOAc (3 × 20 mL), the combined organic layers were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure.

2.4 General procedure for preparation of Aryl sulfonium salts



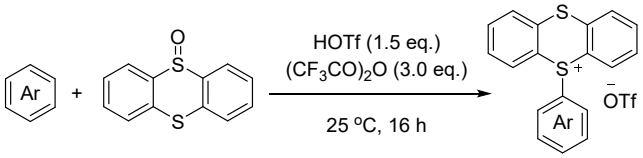
Aryl sulfonium salts S20, S22, S24, S28, S37, S38 are known compounds and were prepared according to the literature procedures 7.[7]

Aryl sulfonium salts S18, S25, S30, S33, S34, S36, S40 are known compounds and were prepared according to the literature procedures 8.[8]

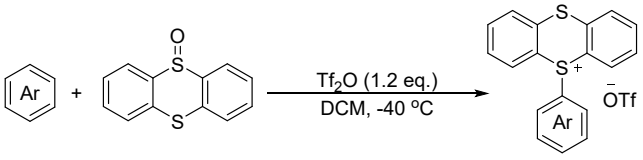
Aryl sulfonium salts S19, S23, S26, S41, S42 are known compounds and were prepared according to the literature procedures 9.[9]

Aryl sulfonium salts S21, S27, S29, S31, S32, S35, S43 are known compounds and were prepared according to the literature procedures 10.[10]

Aryl sulfonium salts S39 are known compounds and were prepared according to the literature procedures 11.[11]

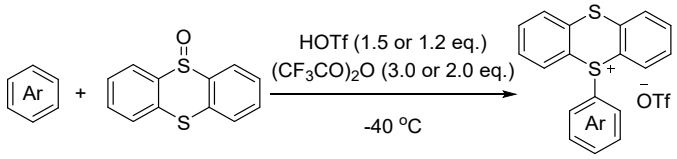


General Procedure 7: Under ambient atmosphere, a 50 mL glass-vial was charged with thianthrene *S*-oxide (3.0 mmol, 1.0 equiv.), arenes (3.3 mmol, 1.1 equiv.) and dry MeCN (c = 0.25 M). At 0 °C, trifluoromethanesulfonic acid (4.5 mmol, 1.5 equiv.) was added, followed by trifluoroacetic anhydride (9.0 mmol, 3.0 equiv.). The vial was sealed with a screw-cap, and the mixture was stirred at 25 °C for 16 h. The reaction mixture was concentrated under reduced pressure, and diluted with CH_2Cl_2 (30 mL). The CH_2Cl_2 solution was poured onto a saturated aqueous $NaHCO_3$ solution (30 mL). The mixture was poured into a separatory funnel, and the layers were separated. The CH_2Cl_2 layer was collected, and the aqueous layer was further extracted with CH_2Cl_2 (2 × 20 mL). The CH_2Cl_2 layer was dried over Na_2SO_4, filtered, and the solvent was removed under reduced pressure. The resulting mixture was dissolved in a small amount of anhydrous dichloromethane, which was slowly dropped into anhydrous ether to precipitate out the sulfonium salts solid. The solid was collected by filtration and washed with ether. Then, solid was dried in vacuo to afford the Aryl sulfonium salts (Thianthrene *S*-oxide in S41 is added last).

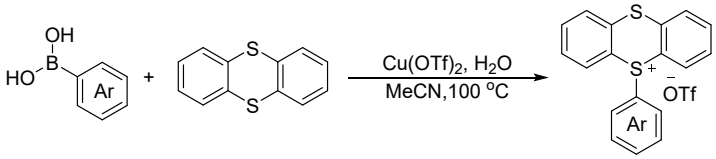


General Procedure 8: A 50 mL Schlenk tube was charged with thianthrene *S*-oxide (1.0-1.2 equiv.), DCM (15 mL) and arenes (3 mmol, 1.0 equiv.) under a nitrogen atmosphere. The reaction mixture was then cooled to -40 °C and stirred at this temperature. Tf_2O (3.6 mmol, 1.2 equiv.) was added dropwise. The reaction mixture was stirred at -40 °C for 30 min, and then allowed to stir at room temperature for 12 h, neutralized by a saturated aqueous $NaHCO_3$ solution, and extracted with DCM. The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated to

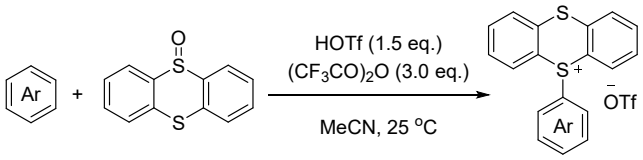
dryness under reduced pressure. The crude product was purified by crystallization from DCM / diethyl ether system to afford Aryl sulfonium salts.



General Procedure 9: To a solution of thianthrene *S*-oxide (3.0 mmol, 1.0 equiv.) and arenes (3.0 mmol, 1.0 equiv.) in MeCN (6 mL) in an oven-dried flask at -40 °C under argon atmosphere, was added dropwise HOTf (1.2-3.0 equiv.) and TFAA (3.0 or 2.0 equiv.). The reaction mixture was stirred at -40 °C for 30 minutes, and then allowed to stir at room temperature for 12 h. The reaction mixture was neutralized by a saturated aqueous $NaHCO_3$ solution, and extracted with DCM three times. The combined organic layers were washed three times with aqueous NaOTf or $NaBF_4$ solution (10% w/w), then dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The crude product was purified by crystallization from DCM/diethyl ether system to afford Aryl sulfonium salts.



General Procedure 10: A 10 mL Schlenk tube were charged with arylboronic acid (1.0 mmol, 1.0 equiv.) and thianthrene (324 mg, 1.5 mmol, 1.5 equiv.), and the reaction tube was moved to a N_2-filled glove box, followed by the addition of $Cu(OTf)_2$ (724 mg, 2.0 mmol, 2.0 equiv.). After removal of the Schlenk tube out of the glove box, H_2O (36 μL, 2.0 mmol, 2.0 equiv.) and MeCN (10.0 mL) were added under a nitrogen atmosphere. The reaction mixture was heated through an oil bath to 100 °C and stirred for 3.0 hours. After cooling to room temperature, the reaction mixture was added into ammonia solution (30 mL, 25-28% solution in water), and the water phase was extracted with DCM (2 × 30 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated under vacuum. The residue was dissolved in DCM (4.0 mL), and precipitated by adding the solution into the stirring Et_2O (200 mL). The solid was collected afford the Aryl sulfonium salts without further purification.

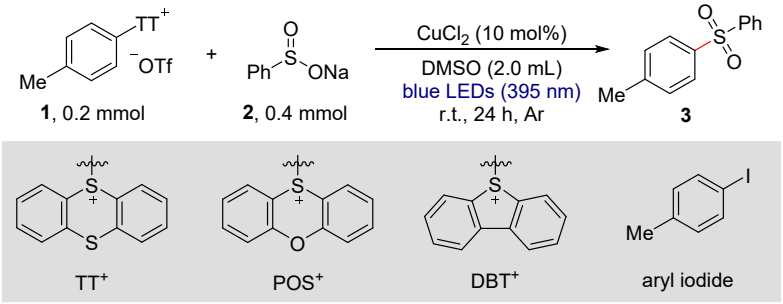


General Procedure 11: Under ambient atmosphere, a 50 mL glass-vial was charged with arene (3.0 mmol, 1.0 equiv.) and dry MeCN (c = 0.25 M). After cooling to 0 °C, $HBF_4 \cdot OEt_2$ (1.2 equiv. + 1.0 equiv. per basic functional group) was added to the vial while stirring the reaction mixture. Other acids may be used instead of $HBF_4 \cdot OEt_2$ like triflic acid (TfOH). For acid sensitive substrates, $BF_3 \cdot OEt_2$ or trimethylsilyl triflate (TMSOTf) can be used. After all solids were dissolved, thianthrene-*S*-oxide (3.0 mmol, 1.0 equiv.) was added in one portion to the solution at 0 °C, leading to a suspension. Subsequently, trifluoroacetic anhydride (9.0 mmol, 3.0 equiv.) was added in one portion at 0 °C, resulting in a color change to deep purple. The vial was sealed with a screw-cap. The mixture was stirred at 0 °C for 1 h, subsequently, the reaction mixture was warmed to 25 °C and stirred until all solid dissolved and the intensity of the purple color decreased. The solution was diluted with 30 mL DCM and poured onto a mixture of DCM, saturated aqueous Na_2CO_3 solution, and water. After stirring for 5 min at 25 °C, the mixture was poured into a separatory funnel, and the layers were separated. The DCM layer was washed with aqueous NaOTf solution and with water. Washing with NaOTf solution is only required if it is of interest that the product contains only one type of counterion, solutions containing other ions, like triflate or hexafluorophosphate may be used as well. The DCM layer was dried over Na_2SO_4, filtered, and the solvent was removed under reduced pressure. Subsequently, the product was dissolved in 2 mL DCM and precipitated with 20 mL Et_2O. The solid was dried in vacuo to afford the thianthrenium salts.

3 Investigation of the Key Reaction Parameters

3.1 Control Experiments

Table S1: Control experiments[a]

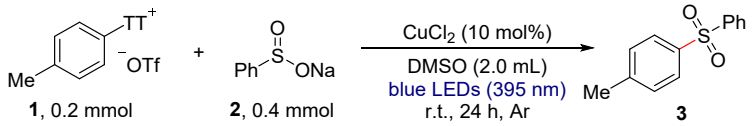


Entry	Variations from standard conditions	Yield (%)[b]
1	None	83[c]
2	Without $CuCl_2$	49
3	Without blue LEDs	trace
4	POS^+ instead of TT^+	62
5	DBT^+ instead of TT^+	58
6	Aryl iodide instead of sulfonium salt	trace
7	benzenesulfonyl hydrazide instead of 2	trace
8	100 °C instead of blue LEDs	trace
9	Red LEDs (620 nm) instead of blue LEDs	trace
10	Green LEDs (520 nm) instead of blue LEDs	trace
11	460 nm instead of 395 nm	36
12	456 nm instead of 395 nm	63
13	440 nm instead of 395 nm	70
14	427 nm instead of 395 nm	68
15	Open to air	61

[a]Reaction condition: sulfonium salts (0.2 mmol), sodium benzene sulfinates (0.4 mmol), $CuCl_2$ (10 %), DMSO (2.0 mL), irradiation with blue LEDs (395 nm) at room temperature for 24 hours under an argon atmosphere. [b]Yield determined by GC using diphenylmethane as an internal standard. [c]Yield of isolated product.

3.2 Optimize the Reaction Conditions

Table S2: Investigation of the Key Reaction Parameters[a]



Entry	Variations from standard conditions	Yield[b] (%)
1	None	83[c]
2	DMA instead of DMSO	65
3	DMF instead of DMSO	68
4	MeCN instead of DMSO	12
5	Toluene instead of DMSO	trace
6	EtOAc instead of DMSO	<5
7	1,4-Dioxane instead of DMSO	trace
8	THF instead of DMSO	13
9	NMP instead of DMSO	29
10	DCM instead of DMSO	<5
11	Acetone instead of DMSO	43
12	$CuCl_2$ (5%) instead of $CuCl_2$ (10%)	68
13	$CuCl_2$ (15%) instead of $CuCl_2$ (10%)	65
14	$CuCl_2$ (20%) instead of $CuCl_2$ (10%)	61
15	CuCl instead of $CuCl_2$	77
16	CuBr instead of $CuCl_2$	68
17	CuI instead of $CuCl_2$	45
18	CuSCN instead of $CuCl_2$	70
19	$Cu(OAc)_2$ instead of $CuCl_2$	65
20	$Cu(acac)_2$ instead of $CuCl_2$	32
21	$CuBr_2$ instead of $CuCl_2$	70
22	$CuSO_4$ instead of $CuCl_2$	32
23	$CuBr_2$ (10%), PPh_3 (20%) instead of $CuCl_2$	62
24	$CuBr_2$ (10%), Xantphos (12%) instead of $CuCl_2$	72
25	$CuBr_2$ (10%), BINAP (12%) instead of $CuCl_2$	19
26	$CuBr_2$ (10%), 1,10-phen (12%) instead of $CuCl_2$	51
27	$CuBr_2$ (10%), dtbbpy (12%) instead of $CuCl_2$	47
28	$KHCO_3$ (1.5 equiv.), $CuCl_2$	45

29	Na_2HPO_4 (1.5 equiv.), $CuCl_2$	49
30	DABCO (1.5 equiv.), $CuCl_2$	53
31	DBU (1.5 equiv.), $CuCl_2$	34

[a]Reaction condition: sulfonium salts (0.2 mmol), sodium benzene sulfinate (0.4 mmol), $CuCl_2$ (10%), DMSO (2.0 mL), irradiation with blue LEDs (395 nm) at room temperature for 24 hours under an argon atmosphere. [b]Yield determined by GC using diphenylmethane as an internal standard. [c]Yield of isolated

4 Mechanism Study

4.1 Radical trapping experiment

Sodium benzene sulfinates (2.0 equiv., 0.4 mmol), sulfonium salts (1.0 equiv., 0.2 mmol), 2,2,6,6-Tetramethyl-1-piperidinoxyl (TEMPO) (5.0 equiv., 1.0 mmol) or 1,1-Diphenylethylene (DPE) (5.0 equiv., 1.0 mmol) and $CuCl_2$ (10%, 0.02 mmol) were placed in a transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, DMSO (2.0 mL) was added via a gastight syringe under argon atmosphere. The reaction mixture was stirred at room temperature (25±3 °C) and irradiated by blue LEDs (395 nm) for 24 h. The mixture was quenched with water and extracted with ethyl acetate (3 × 20 mL). The organic layers were combined and concentrated under vacuo. The organic layers were combined and concentrated under vacuo and the radical trapping products were detected by HR-MS (Fig. S1-S3).

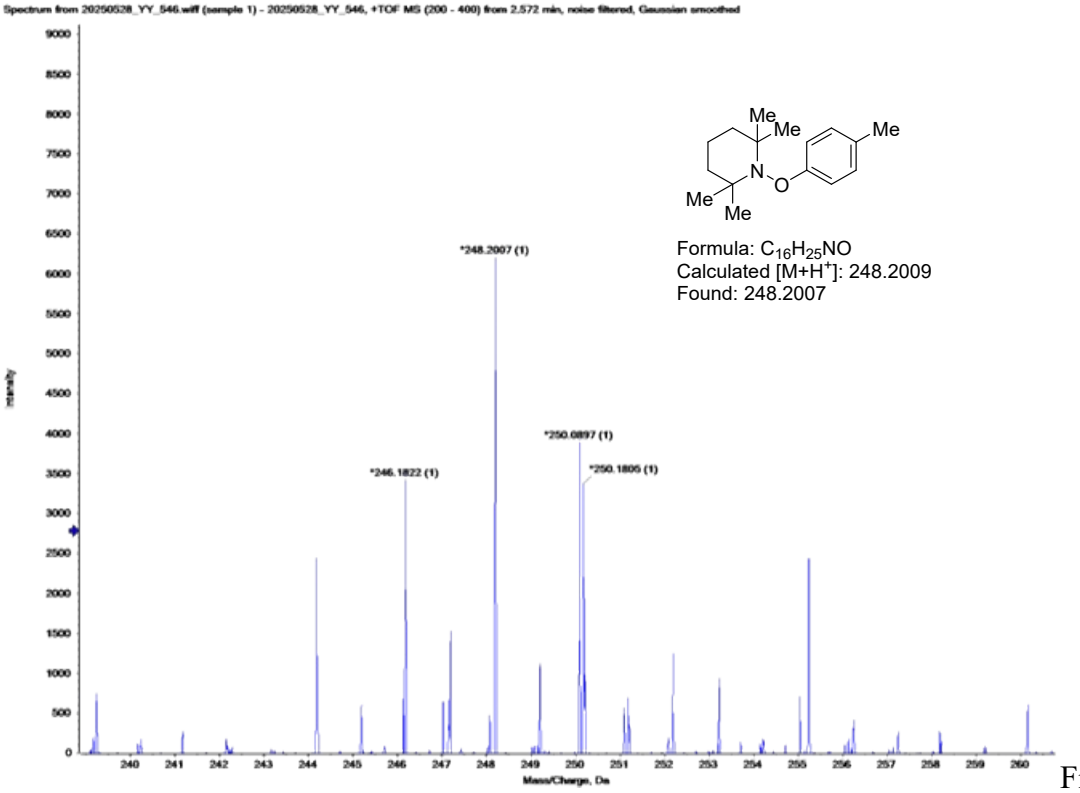


Fig. **S1** Results of the free radical scavenging experiment

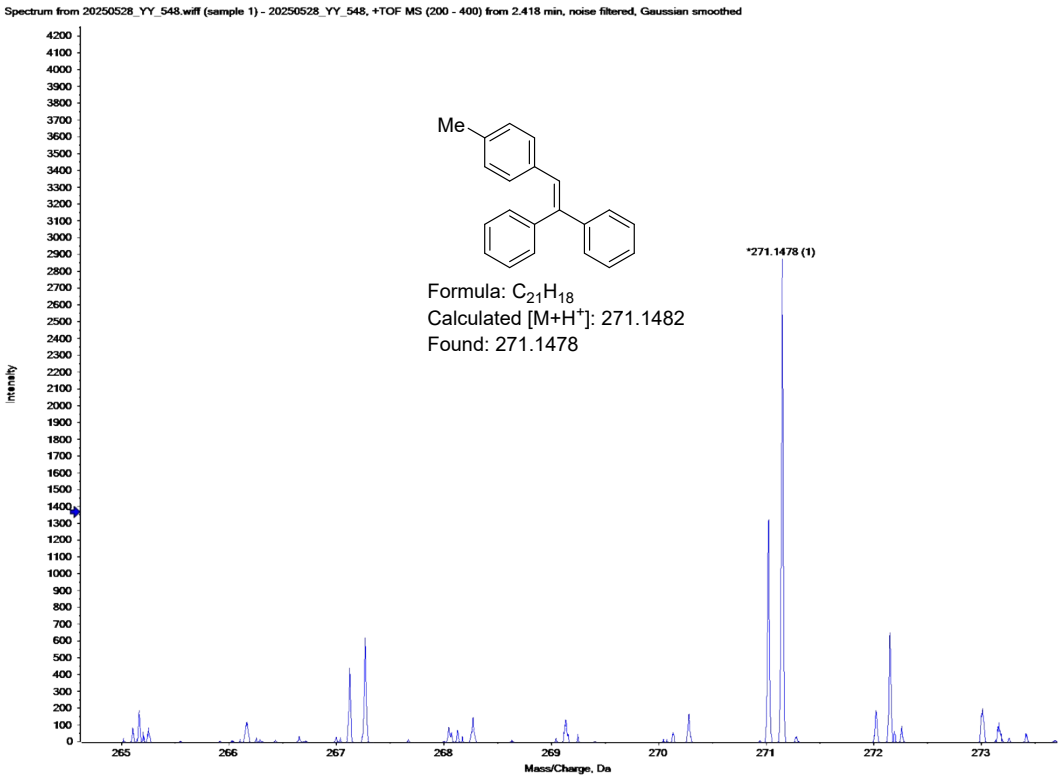


Fig. **S2** Results of the free radical scavenging experiment

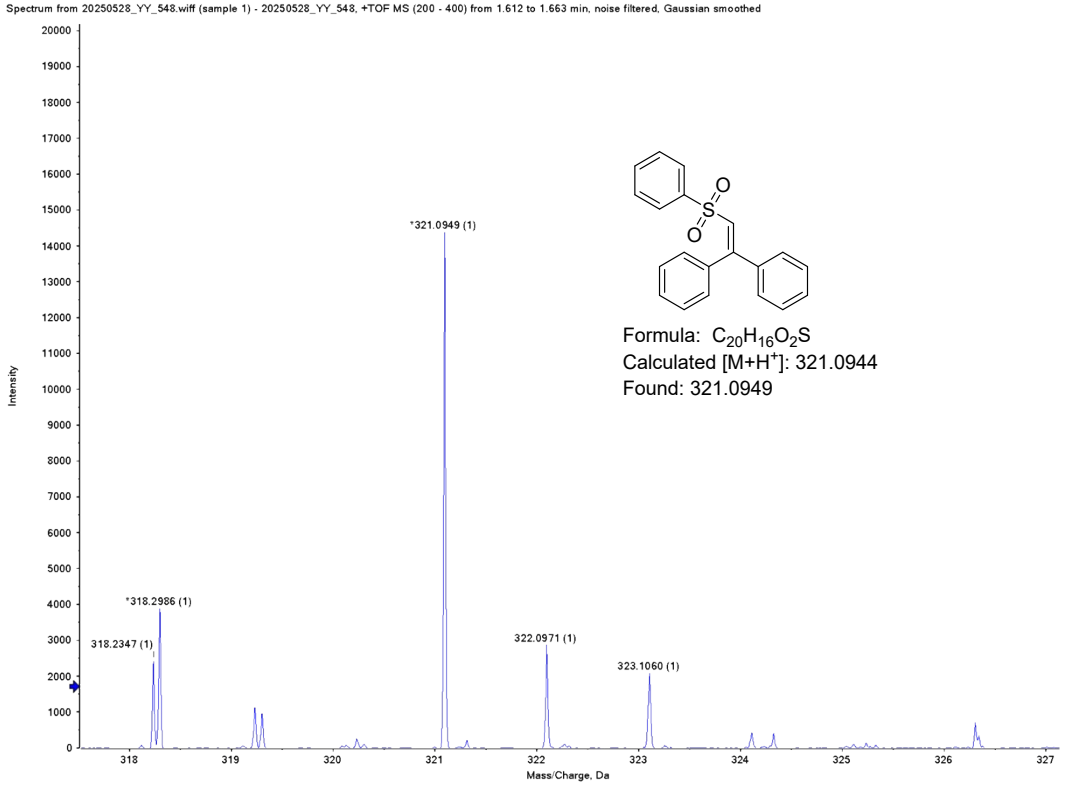
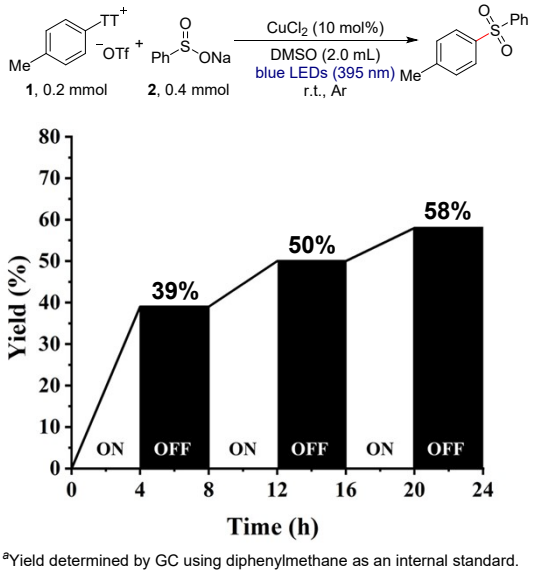


Fig. **S3** Results of the free radical scavenging experiment

4.2 Light on/off experiment



[a]Yield determined by GC using diphenylmethane as an internal standard.

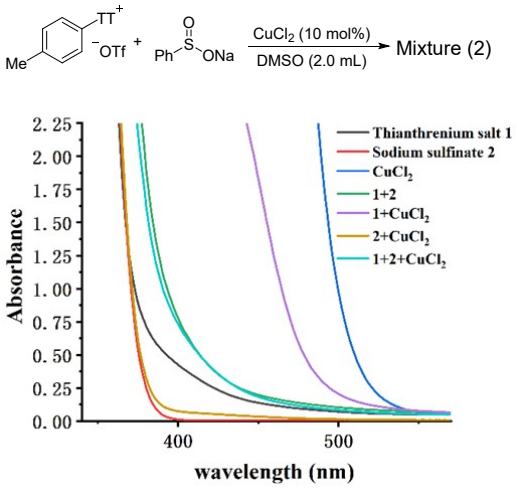
Fig. **S4** Light on-off experiment

The reaction between Sodium benzene sulfinates and sulfonium salts was conducted under the standard conditions on a 0.4 mmol scale. The mixture was subjected to sequential periods of stirring under blue LEDs (395 nm) followed by stirring in the absence of light. At each time point, one reaction system was suspended. Product yields are determined using authentic materials and response-factor calibrated GC data. To examine the effects of light, we conducted Light-On-Off experiments involving alternating periods of radiation and darkness. The results showed that the reaction process was completely interrupted without light, but restored with further irradiation. This demonstrates that current conversion requires continuous illumination and further corroborates the idea that the reaction may have proceeded via a radical mechanism.

4.3 *UV-vis* experiment

UV-Vis experiments were conducted on Agilent Cary 5000 Ultraviolet Visible Near Infrared Spectrophotometer. Optical absorption spectra of reaction components (sulfonium salts 1 and sodium benzene sulfinates 2) and $CuCl_2$ recorded in DMSO using the UV-Vis spectrophotometer. [1] = 0.1 M, [2] = 0.2 M, [$CuCl_2$] = 0.01 M. Furthermore, UV-vis spectroscopy showed no significant redshift of the reaction mixtures (1+2 or 1+2+$CuCl_2$) compared to 1 (Thianthrenium salt) and 2 (Sodium sulfinate) when considered individually. This suggests that no electron donor–acceptor complex was formed between 1 and 2 during this process. Further evidence

demonstrates that the copper catalyst participates in this reaction.



Optical absorption spectra of reaction components (sulfonium salts 1 and sodium benzene sulfinates 2) and CuCl2 recorded in DMSO using the UV-Vis spectrophotometer. [1] = 0.1 M, [2] = 0.2 M, [CuCl2] = 0.01 M.

Fig. **S5** *UV-vis* absorption spectra

4.4 EPR experiment

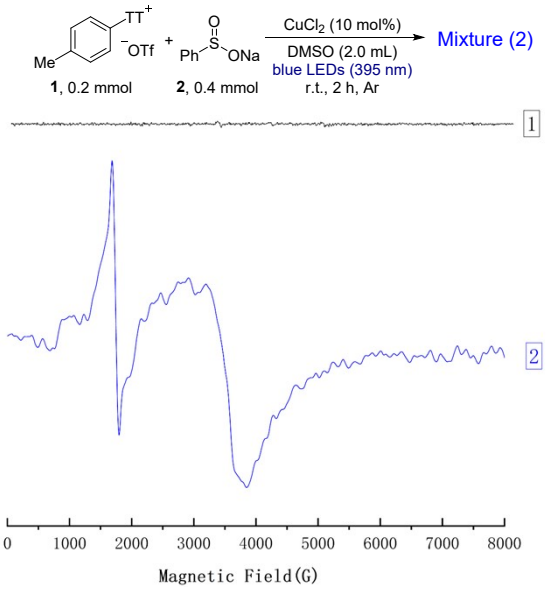


Figure 1: EPR spectrum of reaction mixture using stoichiometric amount of $CuCl_2$. **1**: DMSO. **2**: Add DMPO (5,5-dimethyl-1-pyrrolidone N-oxide), irradiation by blue LEDs (395 nm) for 2 h.

Fig. **S6** EPR spectrum of reaction mixture

Electron paramagnetic resonance (EPR) studies using 5,5-dimethyl-1-pyrrolidone N-oxide (DMPO) as a spin trap provided further confirmation of the generation of radicals during the reaction process. EPR experiment were recorded using a Bruker EMX-9.5/12 Spectrophotometer. Sodium benzene sulfinates (2.0 equiv., 0.4 mmol), sulfonium salts (1.0 equiv., 0.2 mmol) and $CuCl_2$ (10%, 0.02 mmol) were placed in a transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, DMSO (2.0 mL) were added via a gastight syringe under

argon atmosphere. The reaction mixture was stirred at room temperature (25±3 °C) and irradiated by blue LEDs (395 nm) for 2 h. To another 2mL tube was added DMPO (45.2 mg, 0.40 mmol) and DMSO (1.0 mL). Took 2 mL solvent for each of the two tubes and mixed well, then directly detected by EPR.

5. References

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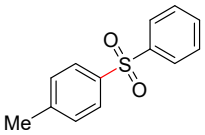
6. Spectral Data and NMR Spectra

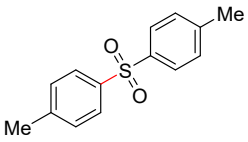
6.1 Spectral Data



1-methyl-4-(phenylsulfonyl)benzene (3): Following the general procedure, obtained in 83% yield as a white solid after silica gel chromatography. (38.6 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Angew. Chem., Int. Ed.* **2013**, *52*, 12679-12683).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.1 Hz, 2H), 7.82 (d, *J* = 8.4 Hz, 2H), 7.55 – 7.46 (m, 3H), 7.29 (d, *J* = 8.6 Hz, 2H), 2.39 (s, 3H).

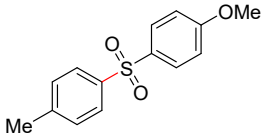
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 142.1, 138.7, 133.1, 130.0, 129.3, 127.8, 127.6, 21.7.



4,4'-sulfonylbis(methylbenzene) (4): Following the general procedure, obtained in 86% yield as a white solid after silica gel chromatography. (42.4 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Org. Lett.* **2022**, *24*, 7961-7966).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.78 (m, 4H), 7.30 – 7.26 (m, 4H), 2.38 (s, 6H).

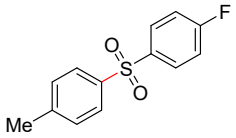
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.1, 139.1, 130.0, 127.7, 21.7.



1-methoxy-4-tosylbenzene (5): Following the general procedure, obtained in 78% yield as a white solid after silica gel chromatography. (40.9 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2023**, *88*, 2773-2783).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.83 (m, 2H), 7.81 – 7.76 (m, 2H), 7.27 (d, J = 8.3 Hz, 2H), 6.97 – 6.93 (m, 2H), 3.83 (s, 3H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4, 143.9, 139.6, 133.7, 130.0, 129.8, 127.5, 114.6, 55.8, 21.7.

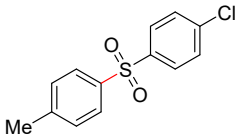


1-fluoro-4-tosylbenzene (6): Following the general procedure, obtained in 71% yield as a white solid after silica gel chromatography. (35.5 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2004**, *69*, 5608-5614).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.59 (m, 2H), 7.50 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.13 (ddt, *J* = 8.6, 6.6, 1.5 Hz, 2H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3 (d, *J* = 251.6 Hz), 142.4, 142.0, 141.5 (d, *J* = 2.8 Hz), 130.3, 127.2 (d, *J* = 8.8 Hz), 125.0, 116.7 (d, *J* = 22.5 Hz), 21.6.

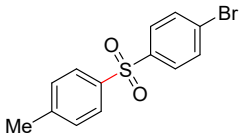
^{19}F NMR (376 MHz, $CDCl_3$) δ -108.52.



1-chloro-4-tosylbenzene (7): Following the general procedure, obtained in 74% yield as a white solid after silica gel chromatography. (39.5 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Eur. J. Org. Chem.* **2022**, *2022*, e202200477).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 9.0 Hz, 2H), 7.81 (d, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 7.30 (d, *J* = 7.9 Hz, 2H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.7, 140.6, 139.8, 138.3, 130.2, 129.7, 129.1, 127.8, 21.7.

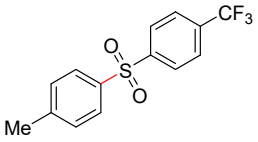


1-bromo-4-tosylbenzene (8): Following the general procedure, obtained in 56% yield as a white solid after silica gel chromatography. (34.9 mg, eluent: petroleum ether/ethyl

acetate = 50/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2004**, *69*, 5608-5614).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.75 (m, 4H), 7.66 – 7.58 (m, 2H), 7.30 (d, J = 8.1 Hz, 2H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.7, 141.2, 138.3, 132.7, 130.2, 129.2, 128.4, 127.8, 21.7.

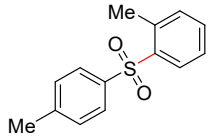


1-methyl-4-((4-(trifluoromethyl)phenyl)sulfonyl)benzene (9): Following the general procedure, obtained in 75% yield as a white solid after silica gel chromatography. (45 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Angew. Chem., Int. Ed.* **2018**, *57*, 1371-1375).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.7, 145.1, 137.7, 134.8 (q, J = 33.1 Hz), 130.3, 128.2, 128.1, 126.5 (q, J = 3.7 Hz), 127.4 – 119.0 (m), 21.8.

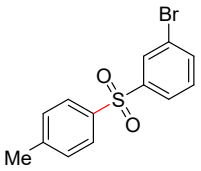
^{19}F NMR (376 MHz, $CDCl_3$) δ -63.05.



1-methyl-2-tosylbenzene (10): Following the general procedure, obtained in 77% yield as a white solid after silica gel chromatography. (38 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2019**, *84*, 3919-3926).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.76 – 7.72 (m, 2H), 7.46 (td, *J* = 7.5, 1.5 Hz, 1H), 7.40 – 7.35 (m, 1H), 7.30 – 7.26 (m, 2H), 7.21 (dt, *J* = 7.5, 0.9 Hz, 1H), 2.43 (s, 3H), 2.40 (s, 3H).

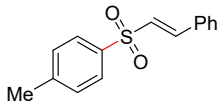
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.1, 139.2, 138.3 137.9, 133.6, 132.7, 129.7, 129.3, 127.8, 126.5, 21.7, 20.3.



1-bromo-3-tosylbenzene (11): Following the general procedure, obtained in 58% yield as a white solid after silica gel chromatography. (36.1 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Chem. Commun. (Cambridge, U. K.)* **2024**, *60*, 6805-6808).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.98 (m, 2H), 7.59 (td, *J* = 7.2, 1.4 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.44 – 7.36 (m, 2H), 7.28 (s, 1H), 2.41 (s, 3H).

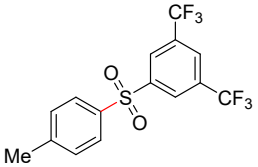
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.9, 144.0, 138.0, 136.2, 130.9, 130.5, 130.2, 128.0, 126.2, 123.3, 21.8.



(*E*)-1-methyl-4-(styrylsulfonyl)benzene (12): Following the general procedure, obtained in 82% yield as a white solid after silica gel chromatography. (42.4 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*ACS Catal.* **2019**, *9*, 1103-1109).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 15.4 Hz, 1H), 7.49 – 7.46 (m, 2H), 7.42 – 7.36 (m, 3H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 15.5 Hz, 1H), 2.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.5, 142.1, 137.8, 132.5, 131.2, 130.1, 129.2, 128.7, 127.8, 127.7, 21.7.



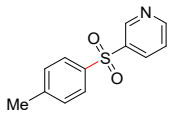
1-tosyl-3,5-bis(trifluoromethyl)benzene (13): Following the general procedure, obtained in 63% yield as a white solid after silica gel chromatography. (46.4 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Adv. Synth. Catal.* **2019**, *361*, 956-960).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (s, 2H), 8.03 (s, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.38

(d, J = 8.1 Hz, 2H), 2.44 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.8, 145.2, 136.8, 133.3 (q, J = 34.7 Hz), 130.7, 128.3, 128.1 – 127.7 (m), 126.9 – 126.7 (m), 123.9 (t, J = 274.1 Hz), 21.8.

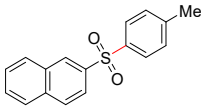
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.81.



3-tosylpyridine (14): Following the general procedure, obtained in 68% yield as a white solid after silica gel chromatography. (31.7 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Adv. Synth. Catal.* **2019**, *361*, 956-960).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 – 9.09 (m, 1H), 8.76 (dd, J = 4.9, 1.6 Hz, 1H), 8.21 – 8.18 (m, 1H), 7.86 – 7.82 (m, 2H), 7.46 – 7.41 (m, 1H), 7.35 – 7.31 (m, 2H), 2.41 (s, 3H).

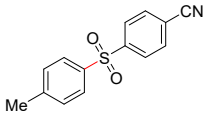
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.6, 148.7, 145.1, 138.8, 137.9, 135.3, 130.4, 128.0, 124.0, 21.7.



2-tosylnaphthalene (15): Following the general procedure, obtained in 80% yield as a white solid after silica gel chromatography. (45.2 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2023**, *88*, 2296-2305).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 7.97 (d, J = 6.9 Hz, 1H), 7.92 – 7.80 (m, 5H), 7.63 – 7.57 (m, 2H), 7.29 (d, J = 7.7 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 138.9, 138.8, 135.1, 132.4, 130.1, 129.7, 129.5, 129.2, 129.0, 128.1, 127.9, 127.7, 122.8, 21.7.



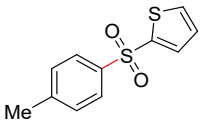
4-tosylbenzonitrile (16): Following the general procedure, obtained in 68% yield as a white solid after silica gel chromatography. (35 mg, eluent: petroleum ether/ethyl

Comment [□□]:

acetate = 20/1). The compound data was in agreement with the literature (*Adv. Synth. Catal.* **2019**, *361*, 956-960).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, J = 8.5 Hz, 2H), 7.83 (d, J = 8.3 Hz, 2H), 7.78 (d, J = 8.5 Hz, 2H), 7.34 (d, J = 8.1 Hz, 2H), 2.42 (s, 3H).

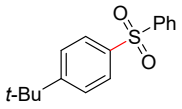
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.4, 145.4, 137.3, 133.2, 130.4, 128.3, 128.2, 117.3, 116.9, 21.8.



2-tosylthiophene (17): Following the general procedure, obtained in 56% yield as a white solid after silica gel chromatography. (26.7 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Adv. Synth. Catal.* **2024**, *366*, 70-76).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.57 (m, 3H), 7.56 – 7.53 (m, 1H), 7.31 (d, *J* = 7.9 Hz, 2H), 7.06 (dd, *J* = 5.0, 3.7 Hz, 1H), 2.41 (s, 3H).

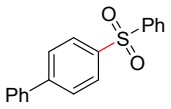
^{13}C NMR (101 MHz, $CDCl_3$) δ 148.6, 142.3, 141.9, 132.3, 131.3, 130.1, 127.4, 124.6, 21.6.



1-(tert-butyl)-4-(phenylsulfonyl)benzene (18): Following the general procedure, obtained in 78% yield as a white solid after silica gel chromatography. (42.8 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2025**, *90*, 44-51).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.93 (m, 2H), 7.88 – 7.84 (m, 2H), 7.56 – 7.47 (m, 5H), 1.30 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 157.2, 142.1, 138.7, 133.1, 129.4, 127.8, 127.7, 126.5, 35.3, 31.2.

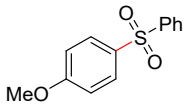


4-(phenylsulfonyl)-1,1'-biphenyl (19): Following the general procedure, obtained in 82% yield as a white solid after silica gel chromatography. (48.3 mg, eluent: petroleum

ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem*. **2025**, *90*, 44-51).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.97 (m, 4H), 7.72 – 7.68 (m, 2H), 7.58 – 7.50 (m, 5H), 7.49 – 7.40 (m, 3H).

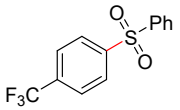
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.3, 141.9, 140.2, 139.3, 133.3, 129.5, 129.2, 128.7, 128.3, 128.1, 127.8, 127.5.



1-methoxy-4-(phenylsulfonyl)benzene (20): Following the general procedure, obtained in 86% yield as a white solid after silica gel chromatography. (42.7 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem*. **2025**, *90*, 44-51).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.86 (m, 4H), 7.56 – 7.46 (m, 3H), 6.96 (d, J = 8.9 Hz, 2H), 3.83 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5, 142.4, 133.2, 133.0, 130.0, 129.3, 127.4, 114.6, 55.8.

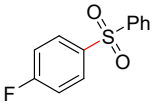


1-(phenylsulfonyl)-4-(trifluoromethyl)benzene (21): Following the general procedure, obtained in 62% yield as a white solid after silica gel chromatography. (35.5 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2019**, *84*, 3919-3926).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.2 Hz, 2H), 8.00 – 7.94 (m, 2H), 7.77 (d, J = 8.6 Hz, 2H), 7.63 – 7.59 (m, 1H), 7.57 – 7.51 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.4 – 145.2 (m), 140.7, 134.9 (q, J = 33.2 Hz), 133.9, 129.7, 128.3, 128.0, 126.6 (q, J = 3.8 Hz), 125.2 – 121.1 (m).

^{19}F NMR (376 MHz, $CDCl_3$) δ -63.05.



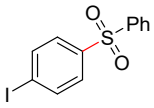
1-fluoro-4-(phenylsulfonyl)benzene (22): Following the general procedure, obtained in 65% yield as a white solid after silica gel chromatography. (30.7 mg, eluent:

petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Eur. J. Org. Chem.* **2018**, *2018*, 1208-1210).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.91 (m, 4H), 7.60 – 7.55 (m, 1H), 7.54 – 7.49 (m, 2H), 7.21 – 7.14 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6 (d, J = 256.0 Hz), 141.6, 137.8 (d, J = 3.3 Hz), 133.5, 130.6 (d, J = 9.6 Hz), 129.5, 127.7, 116.7 (d, J = 22.6 Hz).

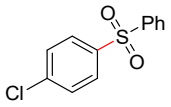
^{19}F NMR (376 MHz, $CDCl_3$) δ -104.09.



1-iodo-4-(phenylsulfonyl)benzene (23): Following the general procedure, obtained in 40% yield as a white solid after silica gel chromatography. (27.5 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Eur. J. Org. Chem.* **2023**, *26*, e202300812).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.91 (m, 2H), 7.85 (dd, J = 8.7, 0.7 Hz, 2H), 7.67 – 7.63 (m, 2H), 7.59 – 7.56 (m, 1H), 7.54 – 7.49 (m, 2H).

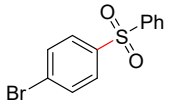
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.4, 141.2, 138.7, 133.6, 129.6, 129.2, 127.8, 101.2.



1-chloro-4-(phenylsulfonyl)benzene (24): Following the general procedure, obtained in 70% yield as a white solid after silica gel chromatography. (35.4 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Org. Lett.* **2013**, *15*, 188-191).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.90 – 7.86 (m, 2H), 7.61 – 7.56 (m, 1H), 7.54 – 7.50 (m, 2H), 7.49 – 7.46 (m, 2H).

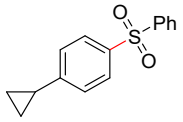
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 140.2, 140.0, 133.6, 129.8, 129.6, 129.3, 127.8.



1-bromo-4-(phenylsulfonyl)benzene (25): Following the general procedure, obtained in 58% yield as a white solid after silica gel chromatography. (34.5 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Org. Lett.* **2013**, *15*, 188-191).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.87 – 7.74 (m, 2H), 7.67 – 7.62 (m, 2H), 7.60 – 7.55 (m, 1H), 7.54 – 7.49 (m, 2H).

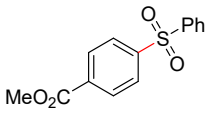
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 140.8, 133.6, 132.8, 129.6, 129.3, 128.6, 127.8.



1-cyclopropyl-4-(phenylsulfonyl)benzene (26): Following the general procedure, obtained in 85% yield as a white solid after silica gel chromatography. (43.9 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Chem. - Asian J.* **2023**, *18*, e202201132).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.0 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 7.55 – 7.52 (m, 1H), 7.51 – 7.46 (m, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 1.92 (tt, *J* = 8.3, 5.0 Hz, 1H), 1.09 – 1.03 (m, 2H), 0.74 (dt, *J* = 7.0, 4.7 Hz, 2H).

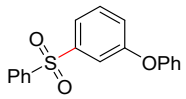
^{13}C NMR (101 MHz, $CDCl_3$) δ 150.5, 141.9, 138.0, 132.8, 129.0, 127.6, 127.3, 126.0, 15.4, 10.4.



methyl 4-(phenylsulfonyl)benzoate (27): Following the general procedure, obtained in 67% yield as a white solid after silica gel chromatography. (37 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2021**, *86*, 13790-13799).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.13 (m, 2H), 8.02 – 7.99 (m, 2H), 7.97 – 7.93 (m, 2H), 7.62 – 7.57 (m, 1H), 7.54 – 7.50 (m, 2H), 3.93 (s, 3H).

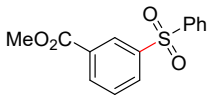
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 145.6, 140.9, 134.4, 133.8, 130.6, 129.6, 128.0, 127.8, 52.8.



1-phenoxy-3-(phenylsulfonyl)benzene (28): Following the general procedure, obtained in 68% yield as a white solid after silica gel chromatography. (42.2 mg, eluent: petroleum ether/ethyl acetate = 20/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.92 (m, 2H), 7.91 – 7.86 (m, 2H), 7.59 – 7.54 (m, 1H), 7.53 – 7.48 (m, 2H), 7.43 – 7.36 (m, 2H), 7.25 – 7.19 (m, 1H), 7.07 – 6.98 (m, 4H).

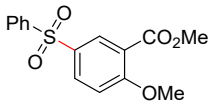
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3, 155.0, 142.2, 135.1, 133.2, 130.3, 130.1, 129.4, 127.6, 125.2, 120.5, 117.8.



methyl 3-(phenylsulfonyl)benzoate (29): Following the general procedure, obtained in 65% yield as a white solid after silica gel chromatography. (35.9 mg, eluent: petroleum ether/ethyl acetate = 40/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2021,** *86*, 13790-13799).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (t, *J* = 1.8 Hz, 1H), 8.22 (dt, *J* = 7.8, 1.5 Hz, 1H), 8.13 (dt, *J* = 7.9, 1.6 Hz, 1H), 7.99 – 7.94 (m, 2H), 7.62 – 7.56 (m, 2H), 7.54 – 7.49 (m, 2H), 3.94 (s, 3H).

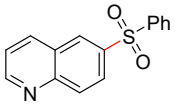
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.4, 142.5, 141.2, 134.2, 133.7, 131.8, 131.7, 129.7, 129.6, 128.9, 127.9, 52.8.



methyl 2-methoxy-5-(phenylsulfonyl)benzoate (30): Following the general procedure, obtained in 78% yield as a white solid after silica gel chromatography. (47.8 mg, eluent: petroleum ether/ethyl acetate = 40/1). The compound data was in agreement with the literature (*ACS Catal.* **2022**, *12*, 1986-1991).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, *J* = 2.4 Hz, 1H), 8.04 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.94 – 7.91 (m, 2H), 7.59 – 7.53 (m, 1H), 7.53 – 7.48 (m, 2H), 7.06 (d, *J* = 8.9 Hz, 1H), 3.94 (s, 3H), 3.89 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.0, 162.7, 141.9, 133.3, 133.2, 133.1, 131.9, 129.5, 127.6, 120.9, 112.6, 56.6, 52.6.

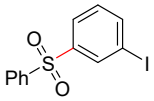


6-(phenylsulfonyl)quinoline (31): Following the general procedure, obtained in 62% yield as light yellow solid after silica gel chromatography. (33.4 mg, eluent: petroleum

ether/ethyl acetate = 40/1). The compound data was in agreement with the literature (*J. Org. Chem.* **2018**, *83*, 6589-6598).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (s, 1H), 8.59 (s, 1H), 8.32 (d, J = 6.9 Hz, 1H), 8.22 (d, J = 8.9 Hz, 1H), 8.09 (dd, J = 8.8, 2.1 Hz, 1H), 8.01 (dd, J = 7.2, 1.9 Hz, 2H), 7.62 – 7.48 (m, 4H).

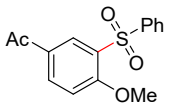
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.3, 149.3, 141.1, 139.5, 137.8, 133.7, 131.3, 129.6, 129.2, 128.0, 127.5, 126.8, 122.9.



1-iodo-3-(phenylsulfonyl)benzene (32): Following the general procedure, obtained in 51% yield as a white solid after silica gel chromatography. (35.1 mg, eluent: petroleum ether/ethyl acetate = 40/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (t, J = 1.8 Hz, 1H), 7.95 – 7.92 (m, 2H), 7.91 – 7.86 (m, 2H), 7.62 – 7.57 (m, 1H), 7.55 – 7.50 (m, 2H), 7.23 (t, J = 7.9 Hz, 1H).

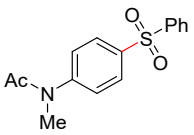
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.5, 142.3, 141.0, 136.2, 133.7, 131.0, 129.6, 127.9, 126.9, 94.5.



1-(4-methoxy-3-(phenylsulfonyl)phenyl)ethan-1-one (33): Following the general procedure, obtained in 45% yield as a white solid after silica gel chromatography. (26.1 mg, eluent: petroleum ether/ethyl acetate = 40/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, J = 2.2 Hz, 1H), 8.19 (dd, J = 8.7, 2.3 Hz, 1H), 7.98 (dd, J = 8.4, 1.4 Hz, 2H), 7.62 – 7.57 (m, 1H), 7.50 (dd, J = 8.4, 7.0 Hz, 2H), 6.97 (d, J = 8.7 Hz, 1H), 3.84 (s, 3H), 2.64 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.6, 160.5, 141.0, 135.7, 133.4, 130.9, 130.1, 129.2, 128.8, 128.7, 112.6, 56.4, 26.6.

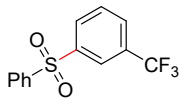


N-methyl-N-(4-(phenylsulfonyl)phenyl)acetamide (34): Following the general procedure, obtained in 55% yield as a white solid after silica gel chromatography. (31.8

mg, eluent: petroleum ether/ethyl acetate = 40/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.93 (m, 4H), 7.63 – 7.57 (m, 1H), 7.55 – 7.51 (m, 2H), 7.37 – 7.31 (m, 2H), 3.27 (s, 3H), 1.94 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 148.7, 141.2, 137.3, 133.6, 129.6, 129.3, 127.9, 127.7, 37.4, 22.8.

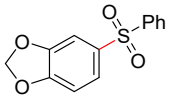


1-(phenylsulfonyl)-3-(trifluoromethyl)benzene (35): Following the general procedure, obtained in 69% yield as a white solid after silica gel chromatography. (39.5 mg, eluent: petroleum ether/ethyl acetate = 40/1). The compound data was in agreement with the literature (*Org. Lett.* **2018**, *20*, 760-763).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (s, 1H), 8.13 (d, J = 8.0 Hz, 1H), 7.99 – 7.95 (m, 2H), 7.82 (d, J = 7.9 Hz, 1H), 7.66 (t, J = 7.9 Hz, 1H), 7.63 – 7.59 (m, 1H), 7.57 – 7.52 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.0, 140.7, 133.9, 132.4 – 131.8 (m), 131.4 – 130.8 (m), 130.3, 130.0 (q, J = 3.6 Hz), 129.7, 124.7 (q, J = 3.9 Hz), 128.0 – 119.1 (m).

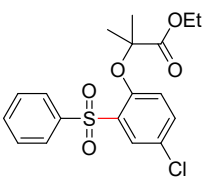
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.69.



5-(phenylsulfonyl)benzo[d][1,3]dioxole (36): Following the general procedure, obtained in 60% yield as a white solid after silica gel chromatography. (31.5 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Org. Lett.* **2014**, *16*, 3836-3839).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H), 7.58 – 7.53 (m, 2H), 7.52 – 7.47 (m, 2H), 7.32 (d, J = 1.8 Hz, 1H), 6.87 (d, J = 8.2 Hz, 1H), 6.04 (s, 2H).

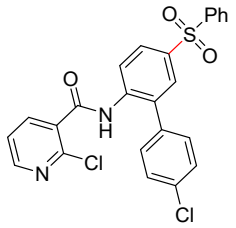
^{13}C NMR (101 MHz, $CDCl_3$) δ 152.0, 148.5, 142.1, 135.0, 133.1, 129.4, 127.5, 123.7, 108.7, 107.9, 102.5.



ethyl 2-(4-chloro-2-(phenylsulfonyl)phenoxy)-2-methylpropanoate (37): Following the general procedure, obtained in 62% yield as a white solid after silica gel chromatography. (47.5 mg, eluent: petroleum ether/ethyl acetate = 10/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 2.7 Hz, 1H), 7.93 – 7.90 (m, 2H), 7.61 – 7.56 (m, 1H), 7.51 – 7.47 (m, 2H), 7.38 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.64 (d, *J* = 8.9 Hz, 1H), 4.14 – 4.08 (m, 2H), 1.50 (s, 6H), 1.11 (t, *J* = 7.1 Hz, 3H).

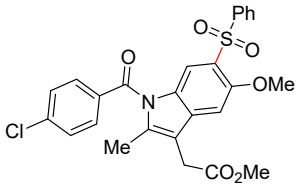
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 152.5, 141.2, 134.5, 133.3, 132.2, 130.3, 128.7, 128.3, 126.7, 118.6, 81.3, 61.8, 24.9, 14.1.



2-chloro-N-(4'-chloro-5-(phenylsulfonyl)-[1,1'-biphenyl]-2-yl)nicotinamide (38): Following the general procedure, obtained in 48% yield as a white solid after silica gel chromatography. (46.4 mg, eluent: petroleum ether/ethyl acetate = 5/1). The compound data was in agreement with the literature (*J. Am. Chem. Soc.* **2025**, *147*, 4268-4283).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, J = 8.8 Hz, 1H), 8.49 – 8.41 (m, 2H), 8.18 (dd, J = 7.7, 2.0 Hz, 1H), 8.03 – 7.93 (m, 3H), 7.83 (d, J = 2.3 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.55 – 7.45 (m, 4H), 7.37 (dd, J = 7.7, 4.7 Hz, 1H), 7.33 – 7.29 (m, 2H).

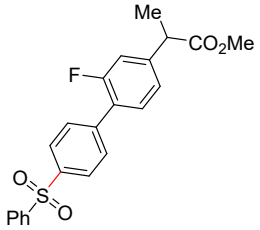
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7, 152.0, 146.5, 141.8, 140.8, 139.10, 137.4, 135.7, 134.2, 133.5, 132.3, 130.9, 130.3, 129.9, 129.6, 128.9, 127.8, 123.2, 121.5.



methyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-6-(phenylsulfonyl)-1H-indol-3-yl)acetate (39): Following the general procedure, obtained in 43% yield as a white solid after silica gel chromatography. (44.0 mg, eluent: petroleum ether/ethyl acetate = 50/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.84 (m, 2H), 7.78 (s, 1H), 7.70 – 7.66 (m, 2H), 7.55 – 7.50 (m, 3H), 7.47 – 7.42 (m, 2H), 7.26 (s, 1H), 6.92 (s, 1H), 3.80 (s, 3H), 3.68 (s, 3H), 3.66 (s, 2H), 2.42 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.1, 168.0, 153.5, 141.8, 140.4, 140.1, 135.0, 132.9, 132.9, 131.5, 129.6, 129.5, 128.5, 128.4, 125.0, 116.1, 112.1, 101.1, 56.4, 52.4, 30.1, 13.6.

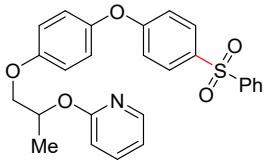


methyl 2-(2-fluoro-4'-(phenylsulfonyl)-[1,1'-biphenyl]-4-yl)propanoate (40): Following the general procedure, obtained in 66% yield as a white solid after silica gel chromatography. (52.6 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Am. Chem. Soc.* **2025**, *147*, 4268-4283).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 4H), 7.67 – 7.63 (m, 2H), 7.59 – 7.49 (m, 3H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.19 – 7.12 (m, 2H), 3.76 (t, *J* = 7.1 Hz, 1H), 3.69 (s, 3H), 1.53 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 174.4, 161.0, 158.5, 143.5 (d, *J* = 7.8 Hz), 141.7, 140.7, 133.4, 130.8 (d, *J* = 3.4 Hz), 129.9 (d, *J* = 3.3 Hz), 129.5, 127.9 (d, *J* = 10.0 Hz), 126.0 (d, *J* = 13.3 Hz), 124.1 (d, *J* = 3.4 Hz), 115.8, 115.6, 52.4, 45.1, 18.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ -116.99.

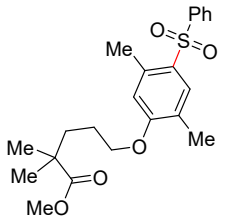


2-((1-(4-(4-(phenylsulfonyl)phenoxy)phenoxy)propan-2-yl)oxy)pyridine (41): Following the general procedure, obtained in 70% yield as a colorless oil after silica gel chromatography. (1.62 g, 5.0 mmol scale, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*J. Am. Chem. Soc.* **2025**, *147*, 4268-4283).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.94 – 7.90 (m, 2H), 7.87

– 7.82 (m, 2H), 7.60 – 7.52 (m, 2H), 7.52 – 7.46 (m, 2H), 6.97 – 6.93 (m, 6H), 6.91 – 6.83 (m, 1H), 6.75 (dd, *J* = 8.3, 1.0 Hz, 1H), 5.59 (dt, *J* = 6.4, 5.1 Hz, 1H), 4.20 (dd, *J* = 9.9, 5.3 Hz, 1H), 4.08 (dd, *J* = 9.9, 4.8 Hz, 1H), 1.48 (d, *J* = 6.4 Hz, 3H).

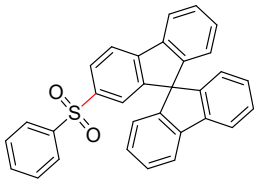
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.2, 163.1, 156.4, 148.2, 146.9, 142.2, 138.9, 134.5, 133.1, 130.0, 129.4, 127.5, 121.9, 117.0, 117.0, 116.2, 111.8, 71.1, 69.3, 17.1.



methyl 5-(2,5-dimethyl-4-(phenylsulfonyl)phenoxy)-2,2-dimethylpentanoate (42): Following the general procedure, obtained in 58% yield as a colorless oil after silica gel chromatography. (46.9 mg, eluent: petroleum ether/ethyl acetate = 20/1). The compound data was in agreement with the literature (*Chem Catal.* **2022**, *2*, 898-907).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.85 – 7.82 (m, 2H), 7.57 – 7.42 (m, 4H), 3.94 (t, *J* = 5.8 Hz, 2H), 3.65 (s, 3H), 2.37 (s, 3H), 2.24 (s, 3H), 1. 74 – 1.66 (m, 4H), 1.21 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 178.3, 161.0, 142.4, 137.9, 132.7, 132.0, 129.5, 129.0, 127.4, 125.0, 114.3, 68.4, 51.9, 42.2, 37.0, 25.3, 25.1, 20.5, 15.8.



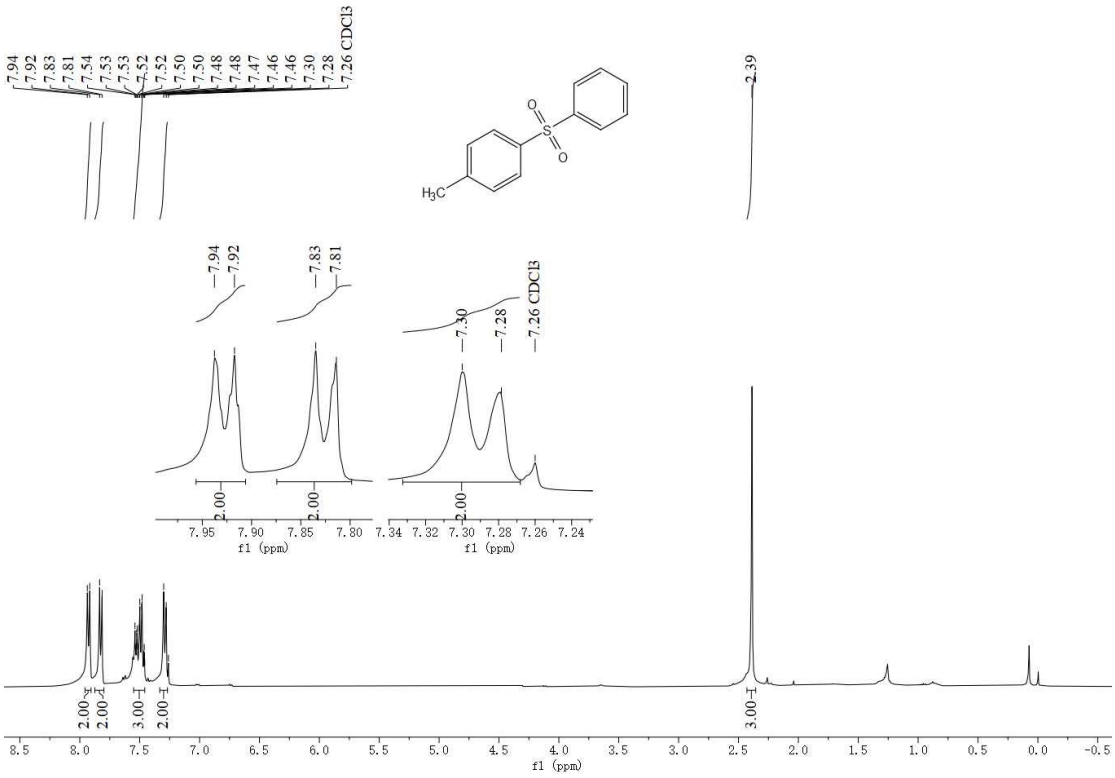
2-(phenylsulfonyl)-9,9'-spirobi[fluorene] (43): Following the general procedure, obtained in 53% yield as a colorless oil after silica gel chromatography. (48.3 mg, eluent: petroleum ether/ethyl acetate = 20/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.84 (m, 5H), 7.81 – 7.75 (m, 2H), 7.55 – 7.47 (m, 1H), 7.45 – 7.36 (m, 6H), 7.18 (td, J = 7.5, 1.1 Hz, 1H), 7.09 (td, J = 7.5, 1.1 Hz, 2H), 6.74 (dt, J = 7.7, 0.9 Hz, 1H), 6.64 (dt, J = 7.6, 0.9 Hz, 2H).

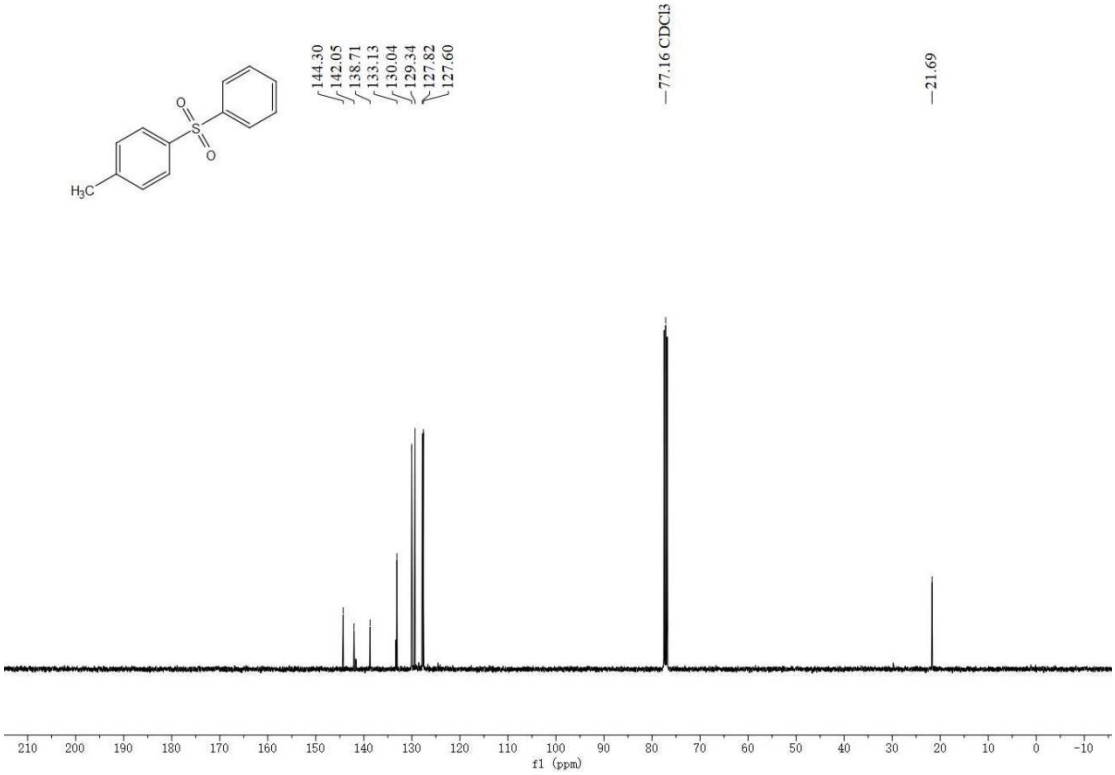
^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2, 150.1, 147.1, 147.0, 142.1, 141.9, 140.4, 139.6, 133.1, 129.8, 129.3, 128.4, 128.2, 128.1, 127.6, 124.4, 124.0, 123.6, 121.2, 120.8, 120.5, 66.1.

6.2 NMR Spectral

^{1}H NMR spectrum of **1-methyl-4-(phenylsulfonyl)benzene (3)**

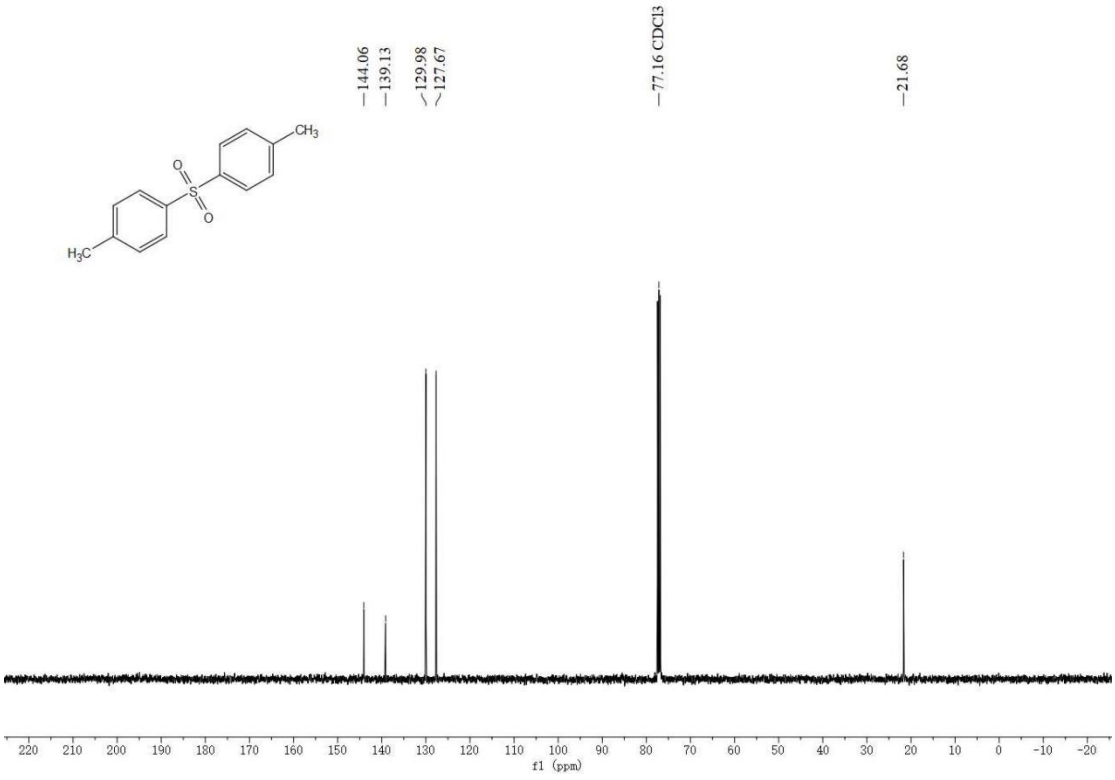


^{13}C NMR spectrum of **1-methyl-4-(phenylsulfonyl)benzene (3)**

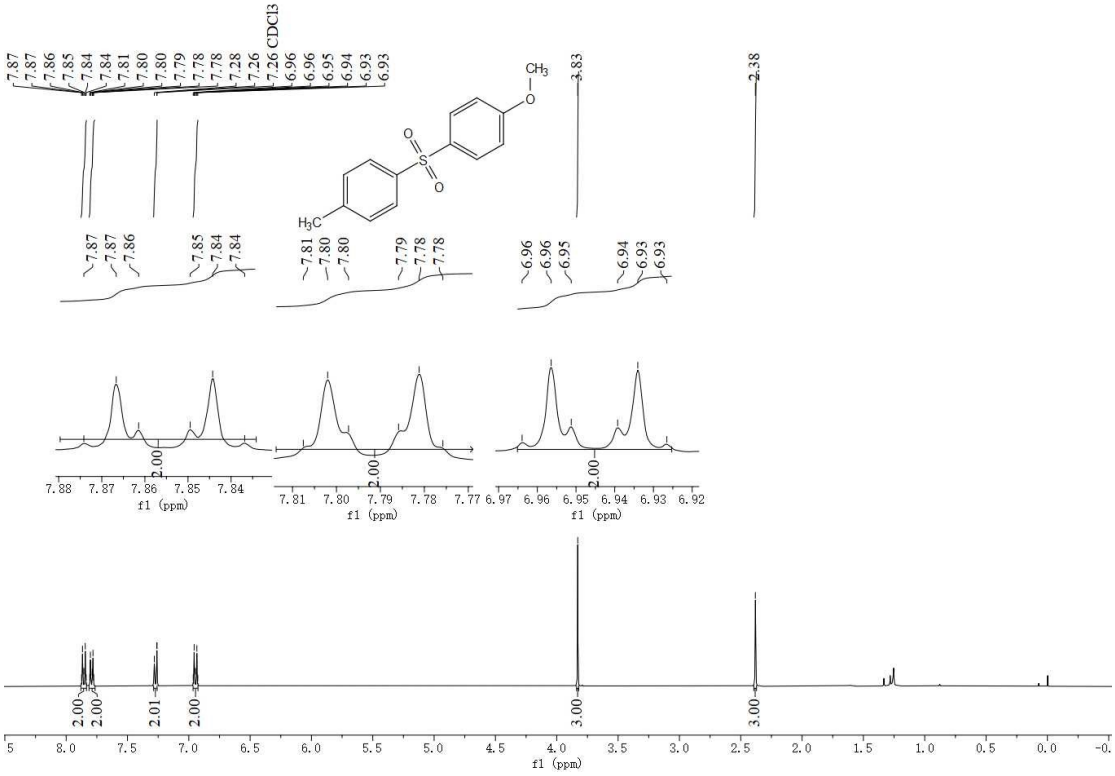


^{1}H NMR spectrum of **4,4'-sulfonylbis(methylbenzene) (4)**

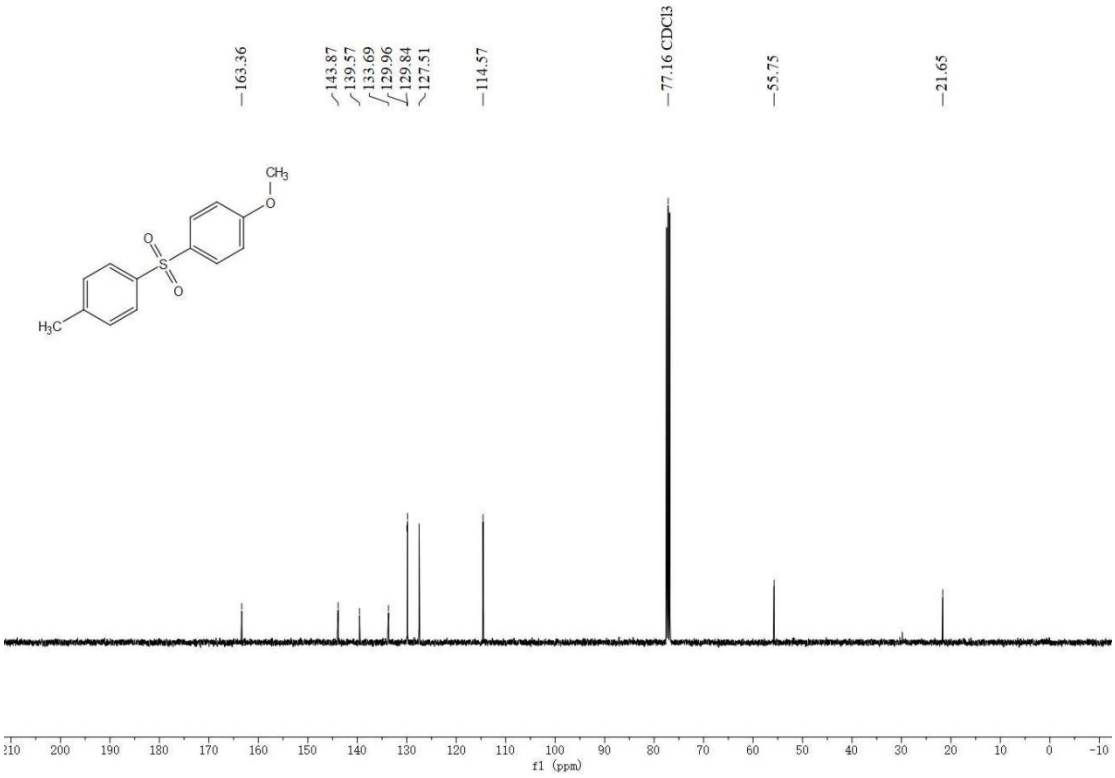
^{13}C NMR spectrum of **4,4'-sulfonylbis(methylbenzene) (4)**



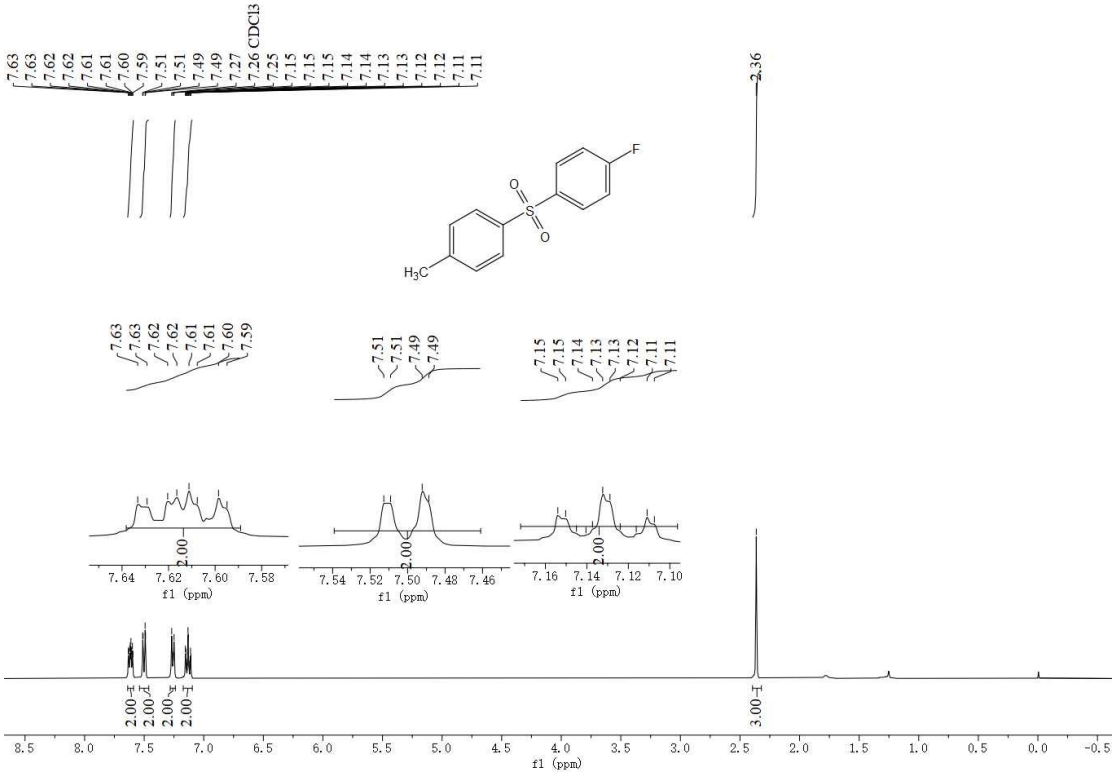
^{1}H NMR spectrum of **1-methoxy-4-tosylbenzene (5)**



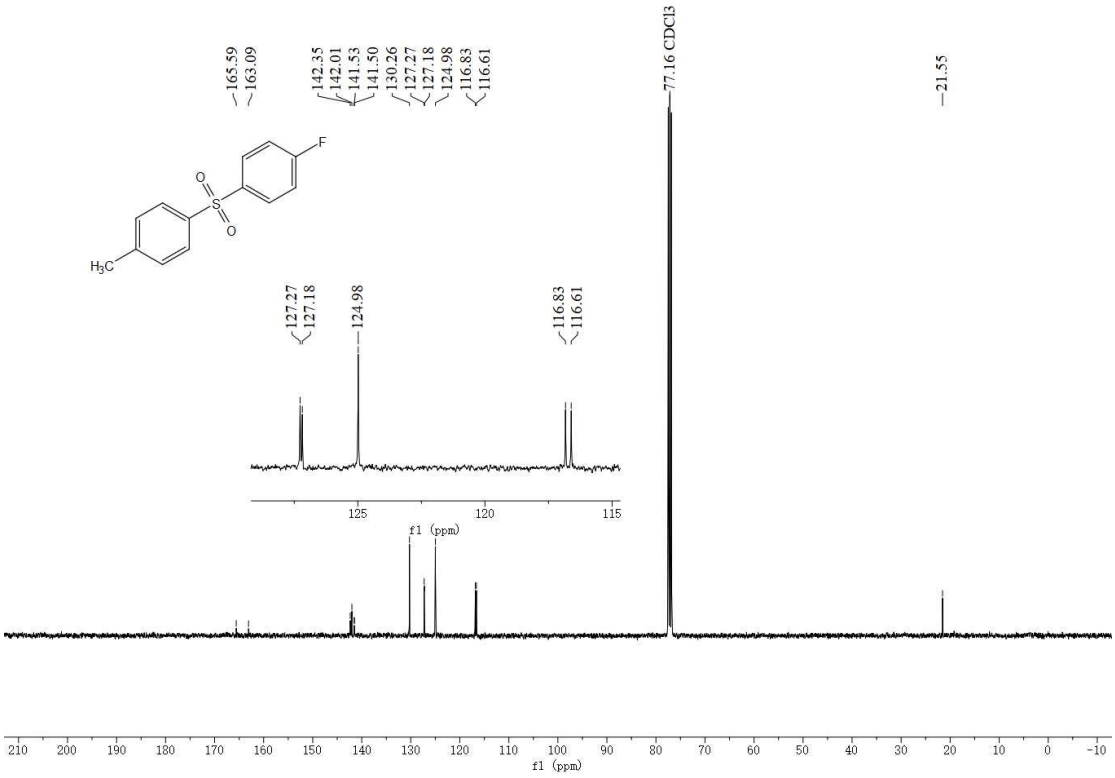
^{13}C NMR spectrum of **1-methoxy-4-tosylbenzene (5)**



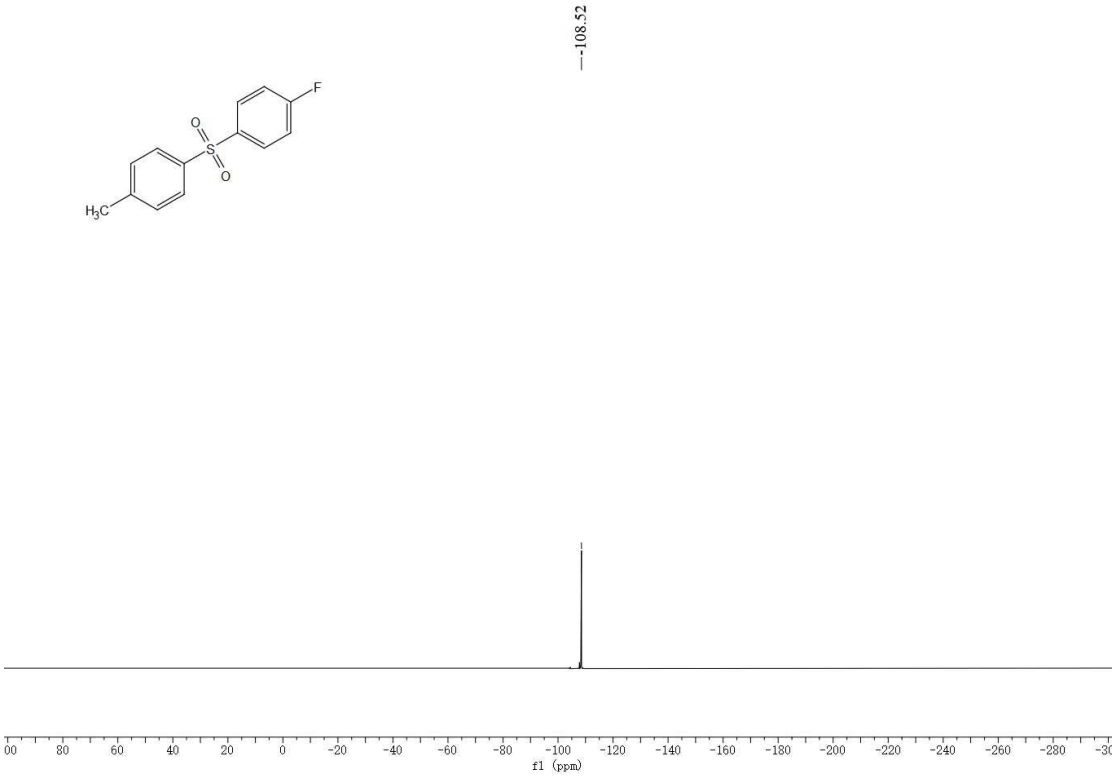
^{1}H NMR spectrum of **1-fluoro-4-tosylbenzene (6)**



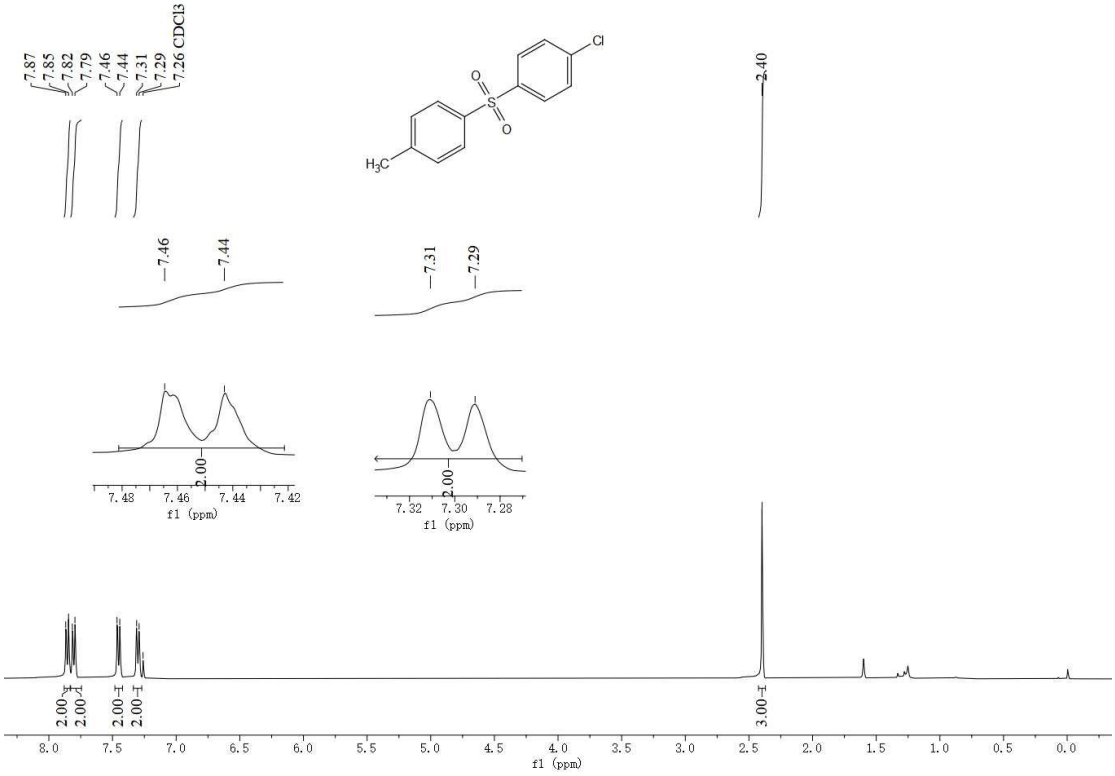
^{13}C NMR spectrum of **1-fluoro-4-tosylbenzene (6)**



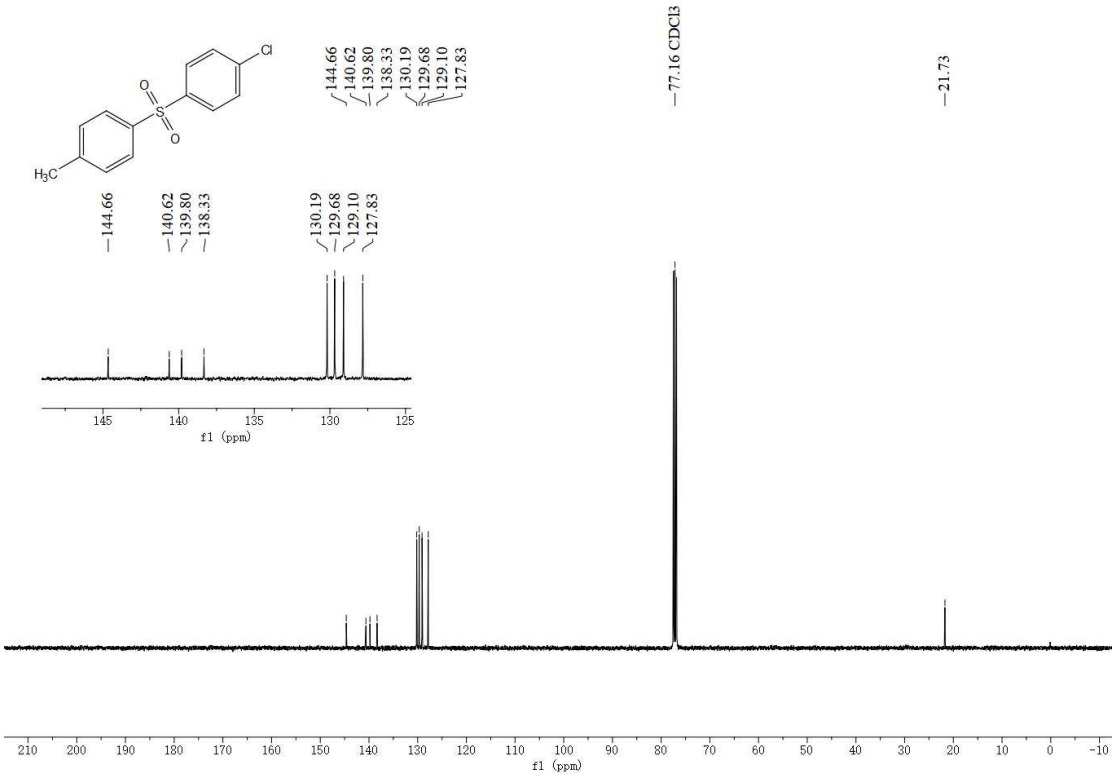
^{19}F NMR spectrum of **1-fluoro-4-tosylbenzene (6)**



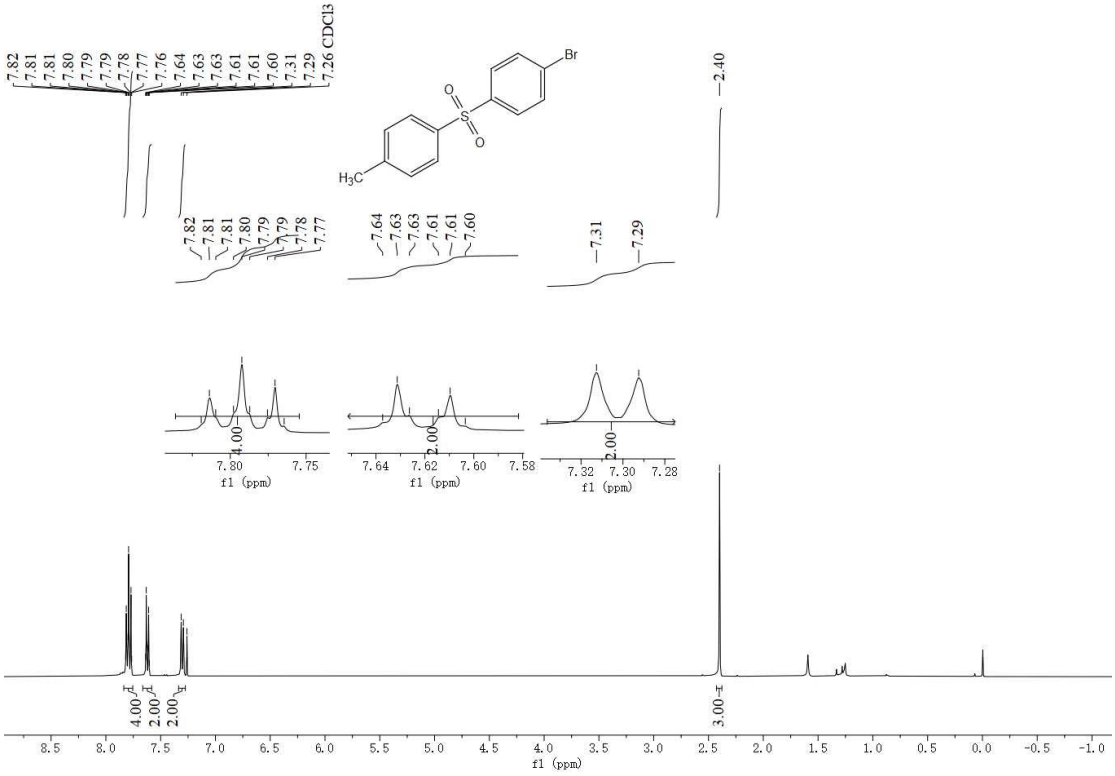
^{1}H NMR spectrum of **1-chloro-4-tosylbenzene (7)**



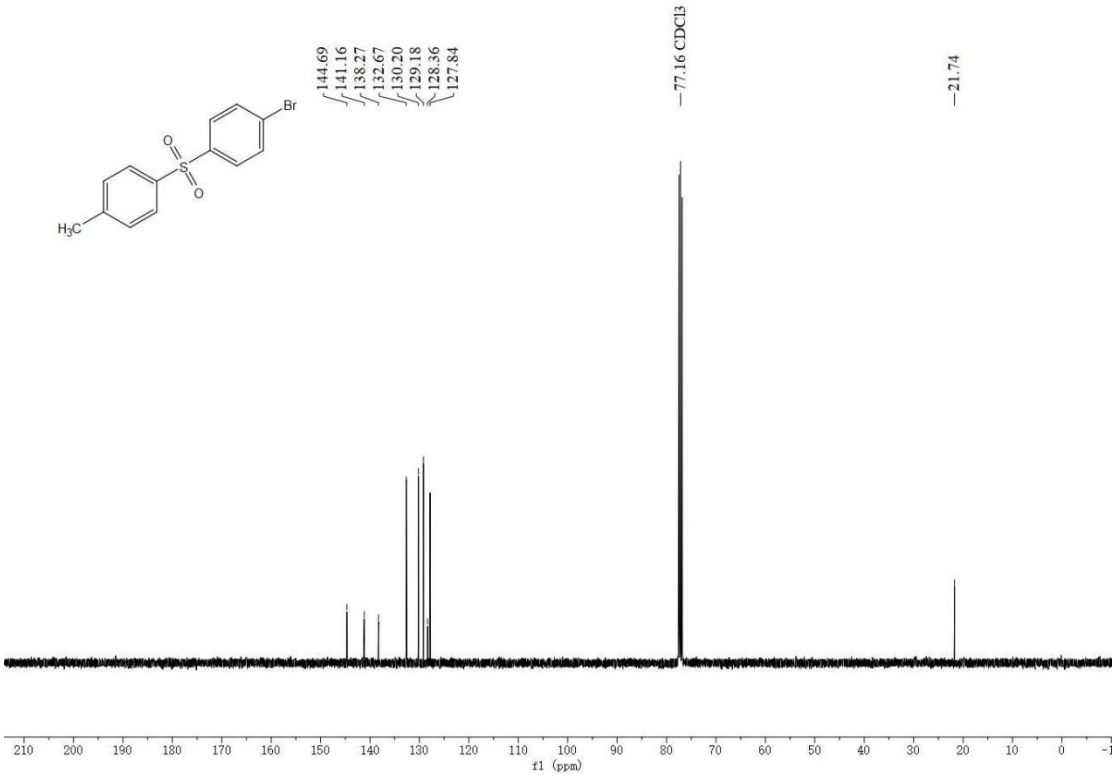
^{13}C NMR spectrum of **1-chloro-4-tosylbenzene (7)**



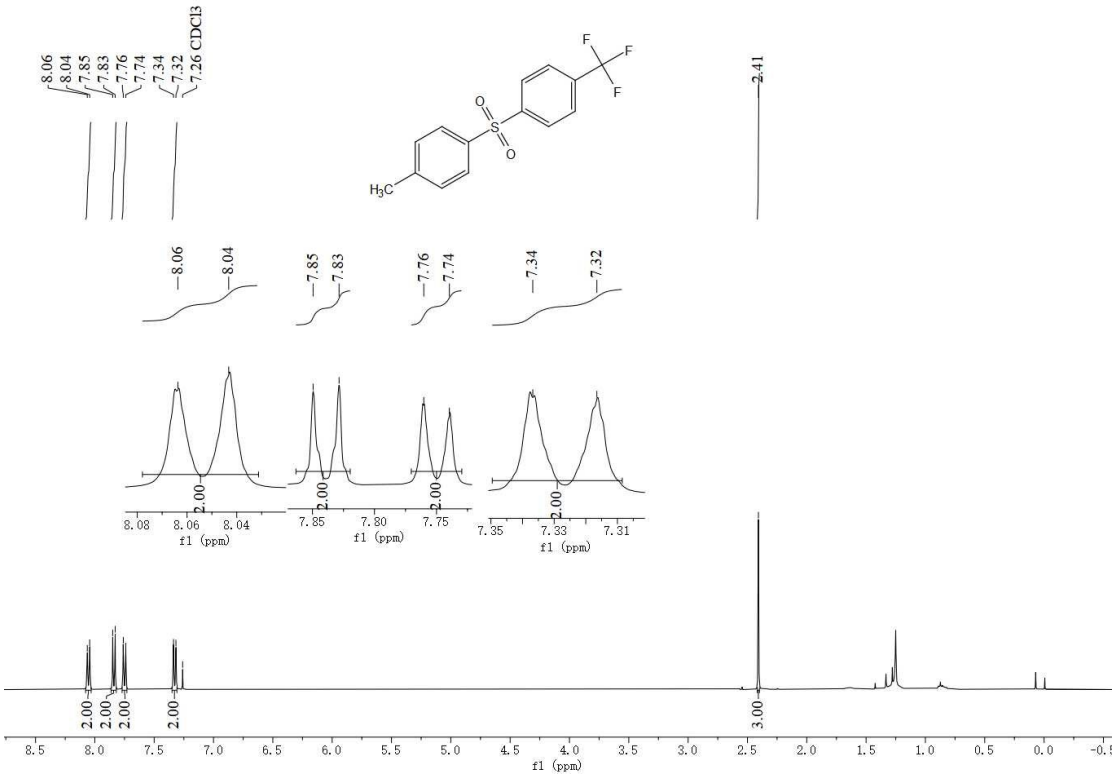
^{1}H NMR spectrum of **1-bromo-4-tosylbenzene (8)**



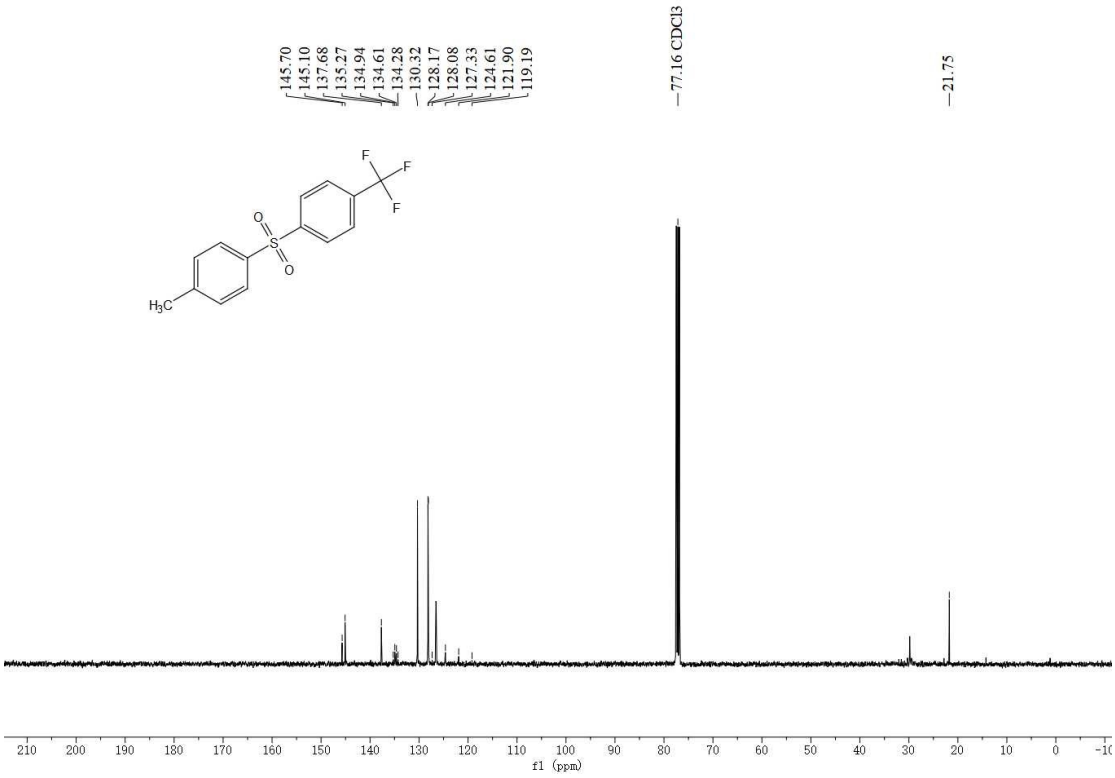
^{13}C NMR spectrum of **1-bromo-4-tosylbenzene (8)**



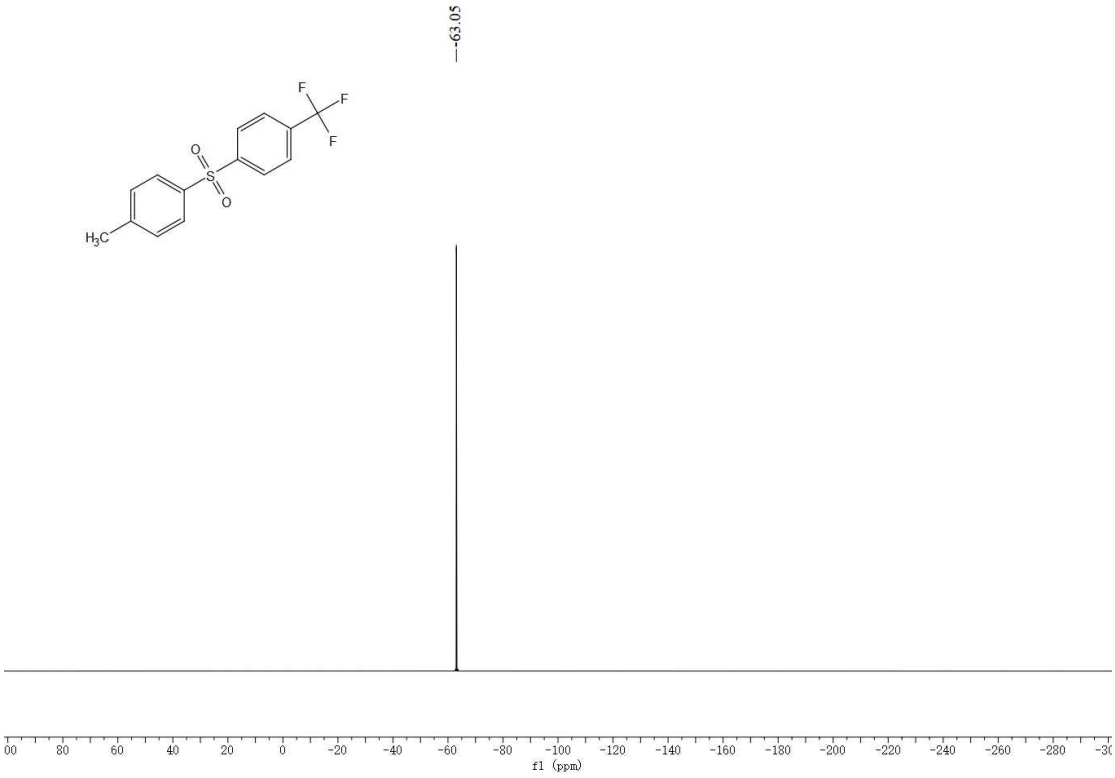
^{1}H NMR spectrum of **1-methyl-4-((4-(trifluoromethyl)phenyl)sulfonyl)benzene (9)**



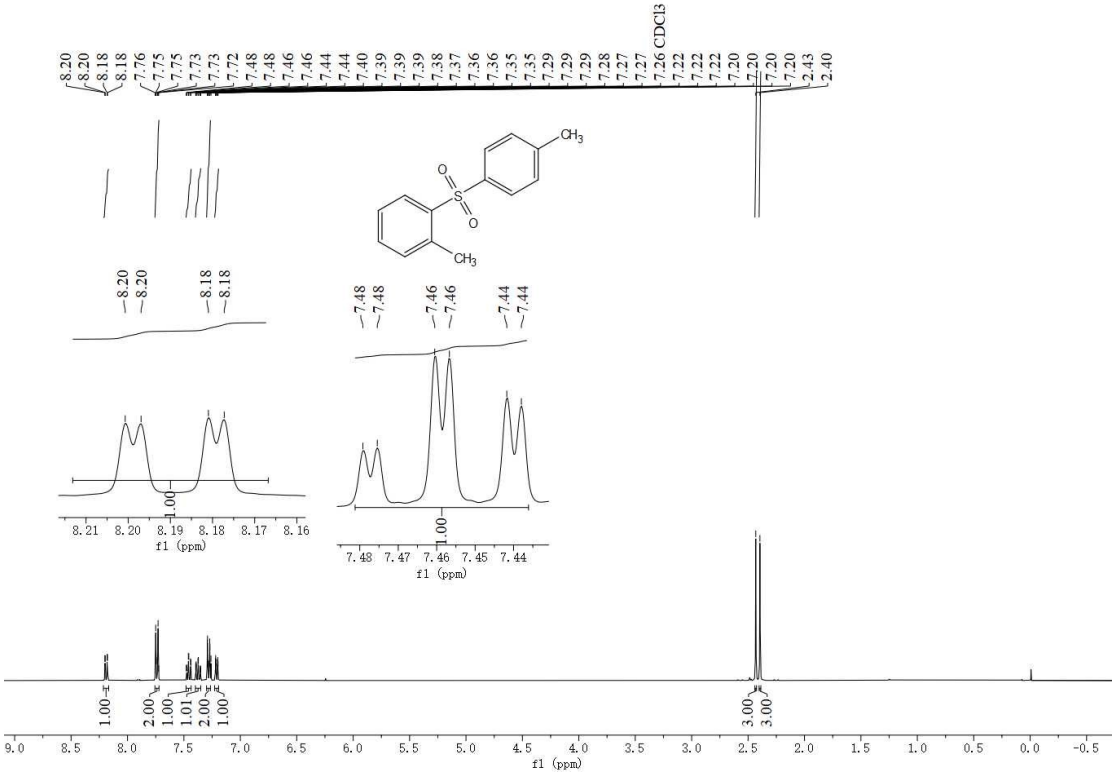
^{13}C NMR spectrum of **1-methyl-4-((4-(trifluoromethyl)phenyl)sulfonyl)benzene (9)**



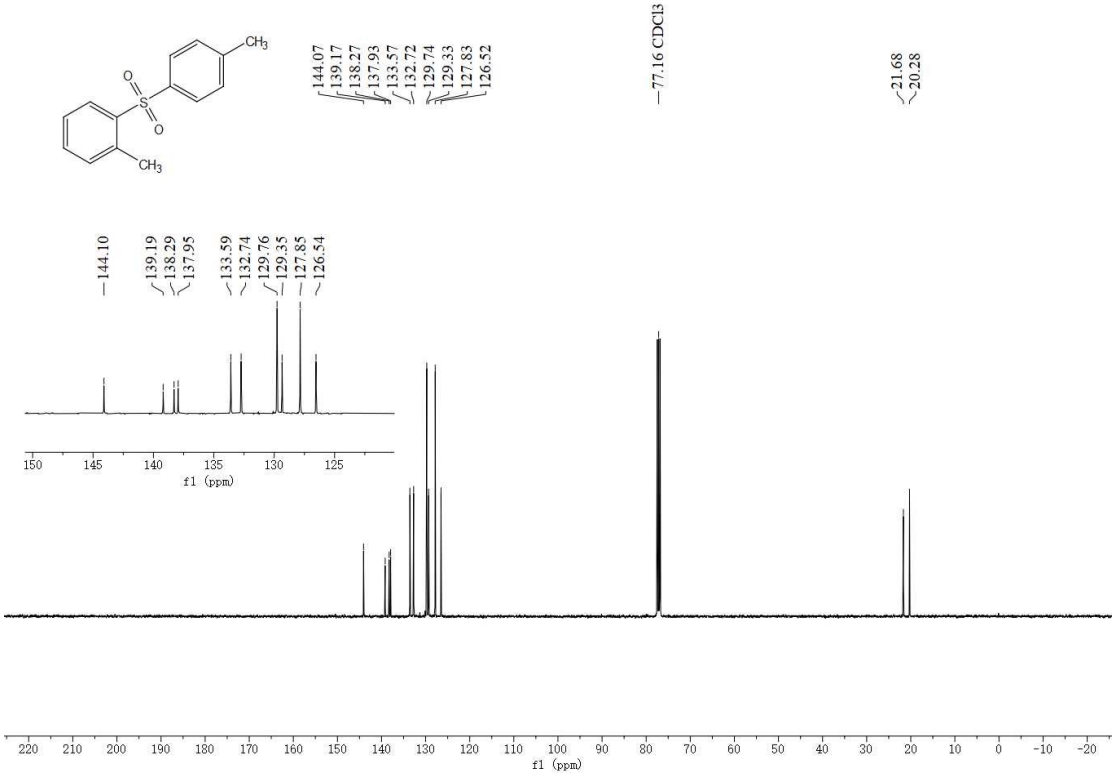
^{19}F NMR spectrum of **1-methyl-4-((4-(trifluoromethyl)phenyl)sulfonyl)benzene (9)**



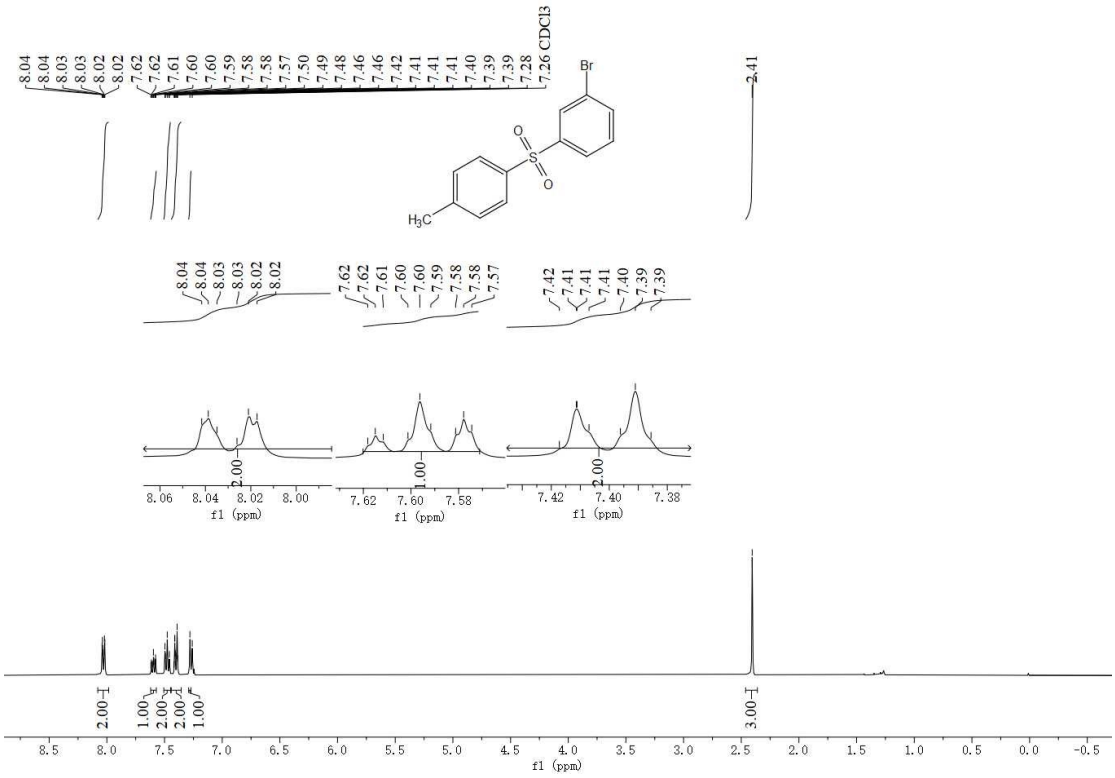
^{1}H NMR spectrum of **1-methyl-2-tosylbenzene (10)**



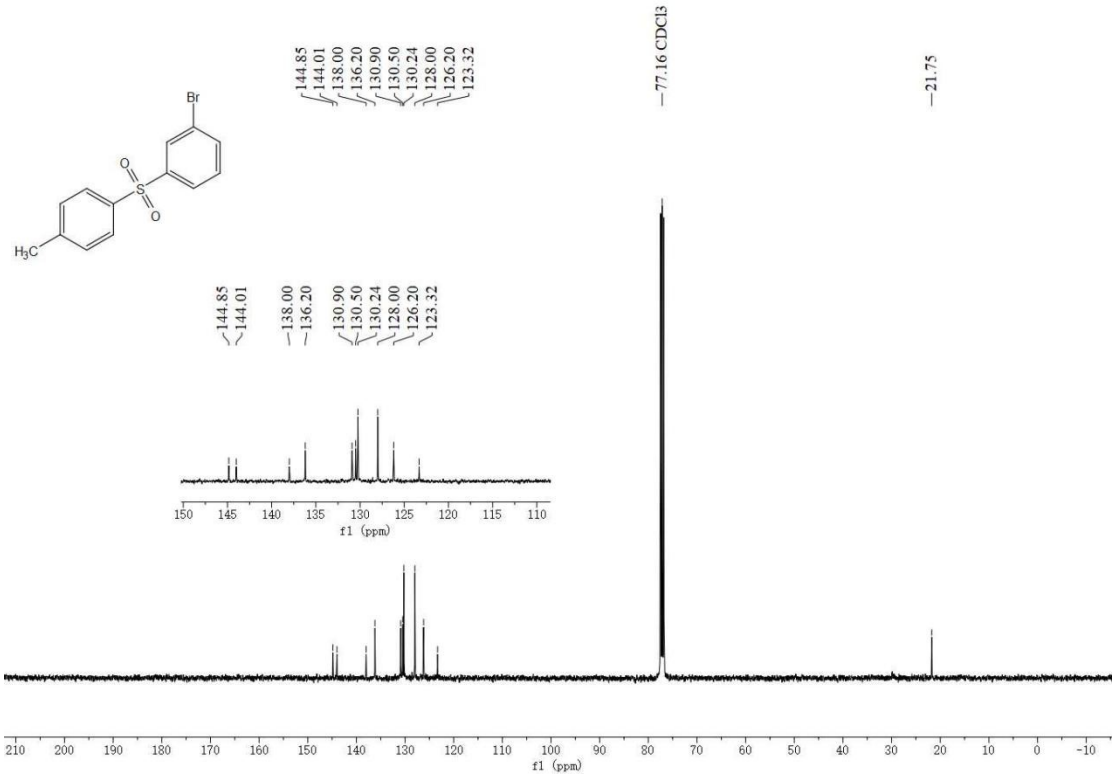
^{13}C NMR spectrum of **1-methyl-2-tosylbenzene (10)**



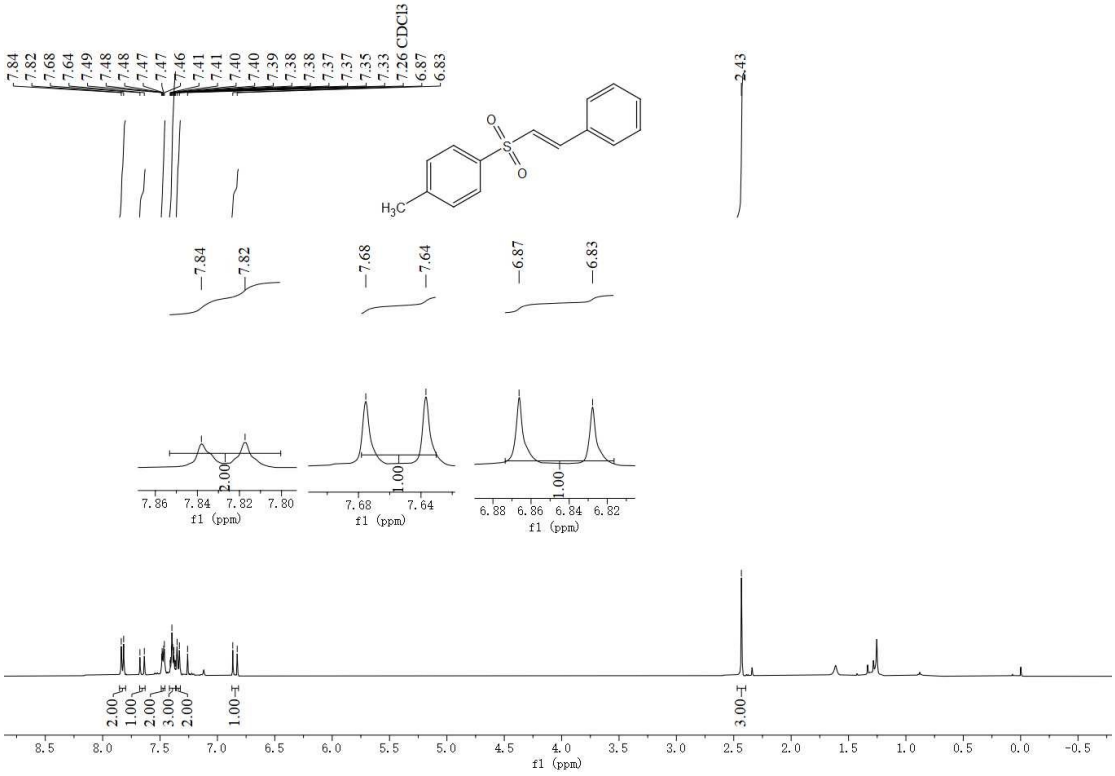
^{1}H NMR spectrum of **1-bromo-3-tosylbenzene (11)**



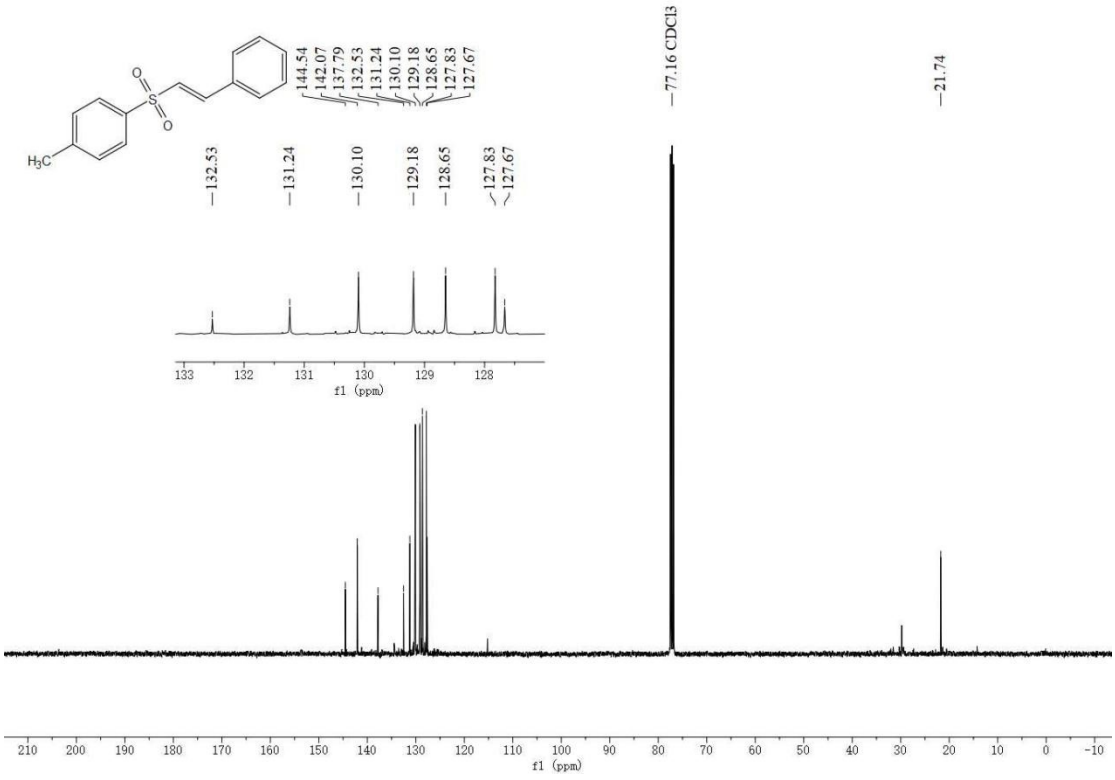
^{13}C NMR spectrum of **1-bromo-3-tosylbenzene (11)**



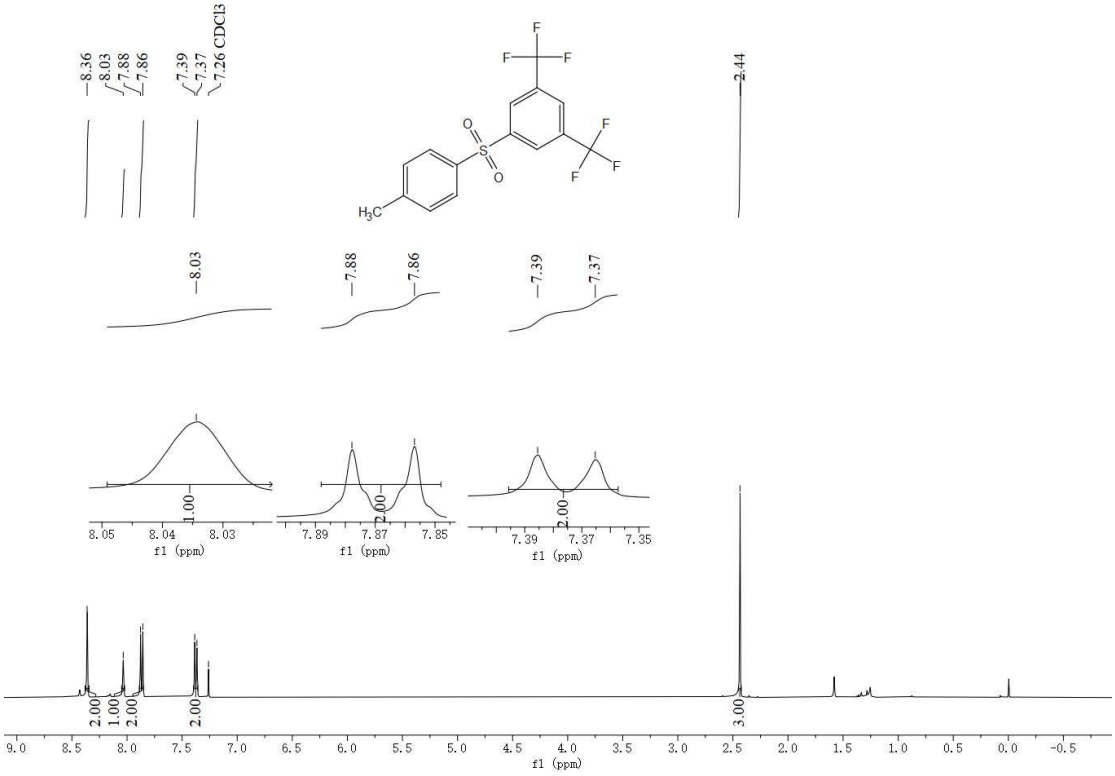
^{1}H NMR spectrum of **(E)-1-methyl-4-(styrylsulfonyl)benzene (12)**



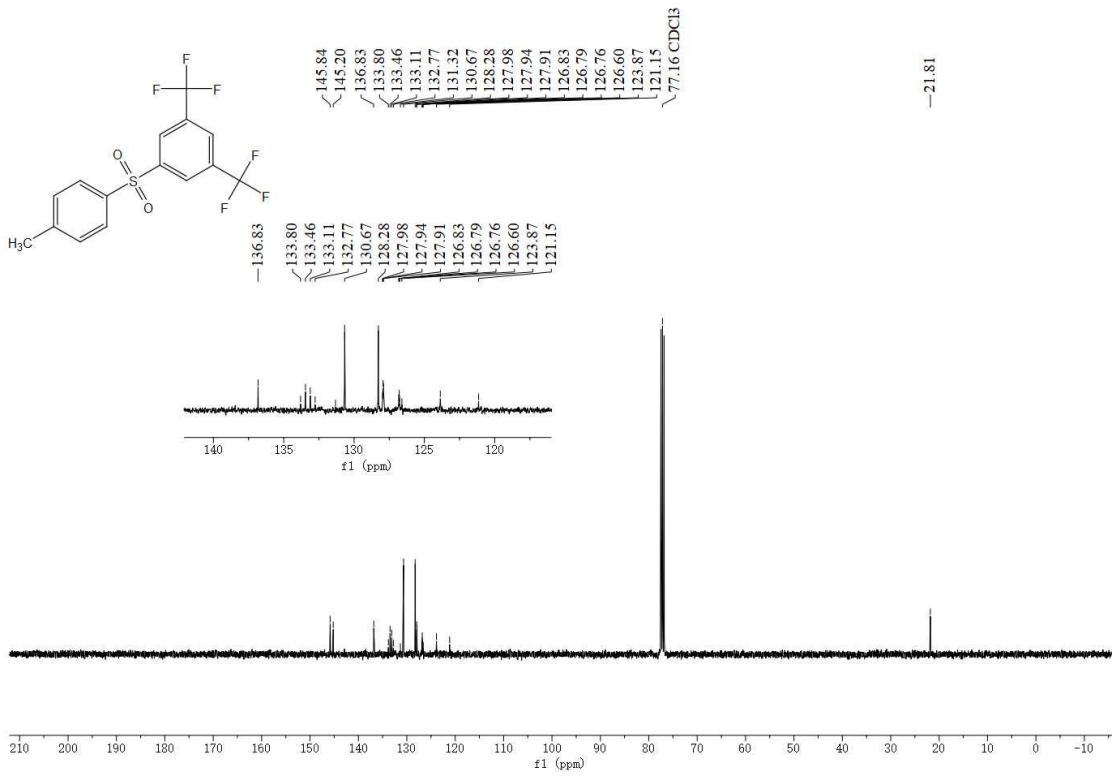
^{13}C NMR spectrum of **(E)-1-methyl-4-(styrylsulfonyl)benzene (12)**



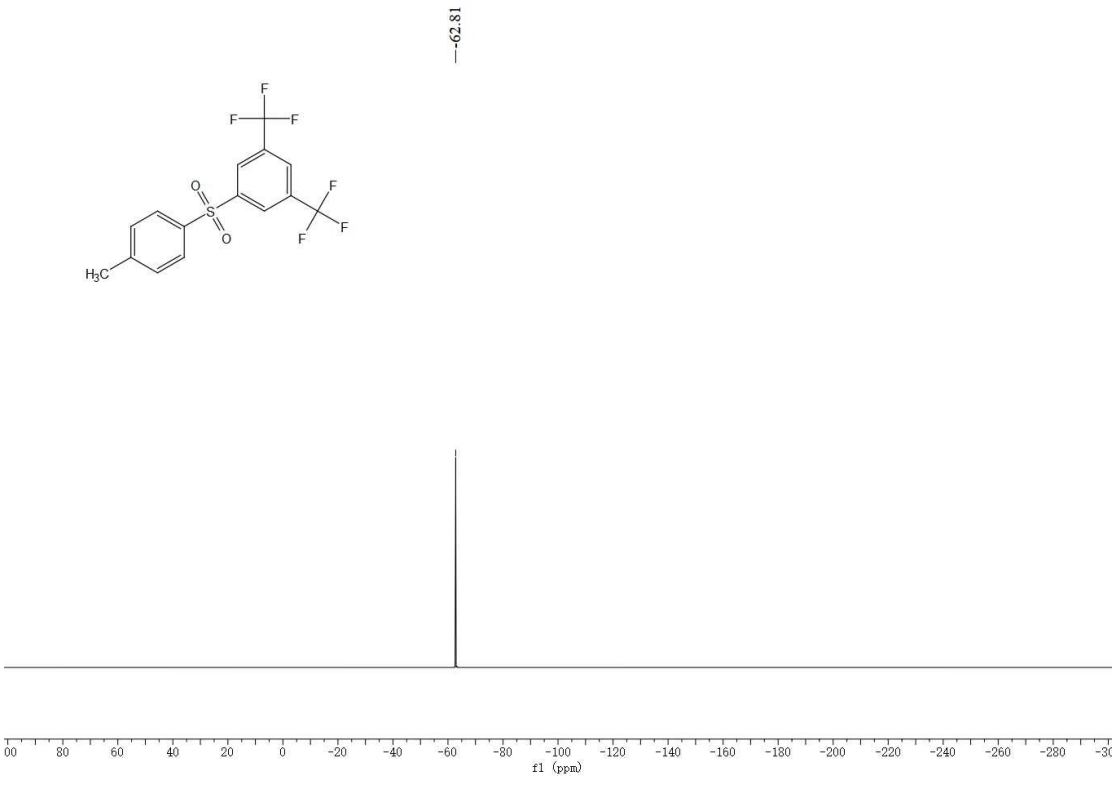
^{1}H NMR spectrum of **1-tosyl-3,5-bis(trifluoromethyl)benzene (13)**



^{13}C NMR spectrum of **1-tosyl-3,5-bis(trifluoromethyl)benzene (13)**



^{19}F NMR spectrum of **1-tosyl-3,5-bis(trifluoromethyl)benzene (13)**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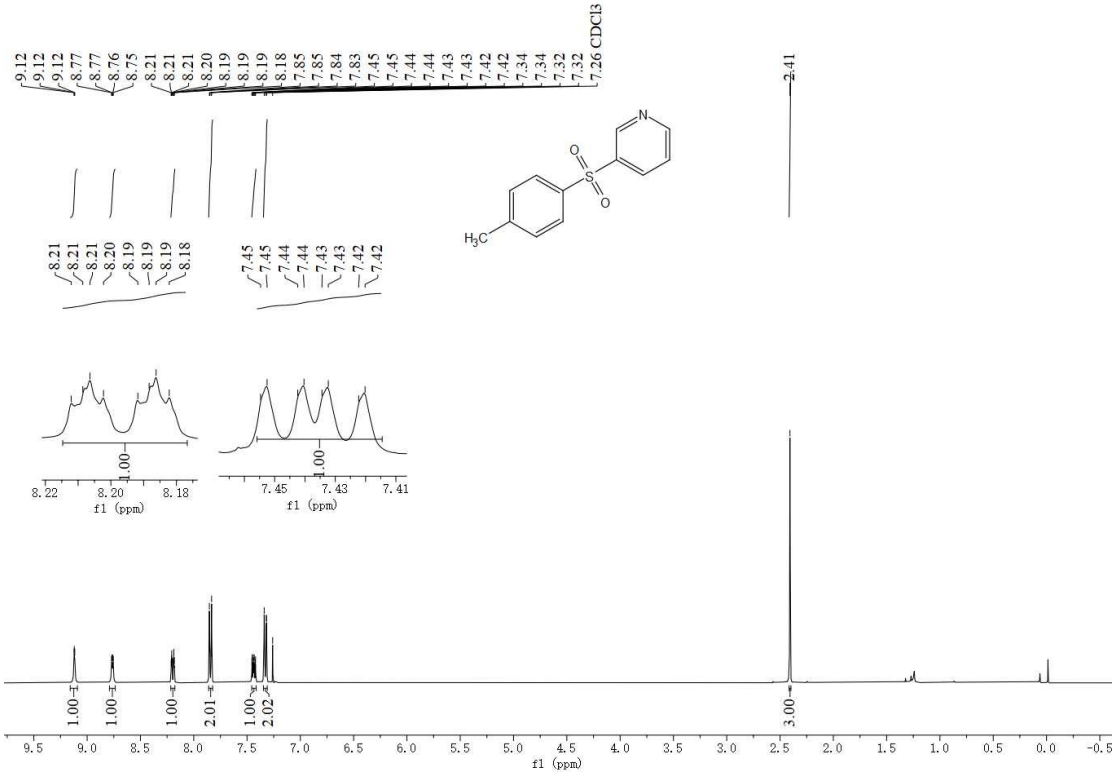
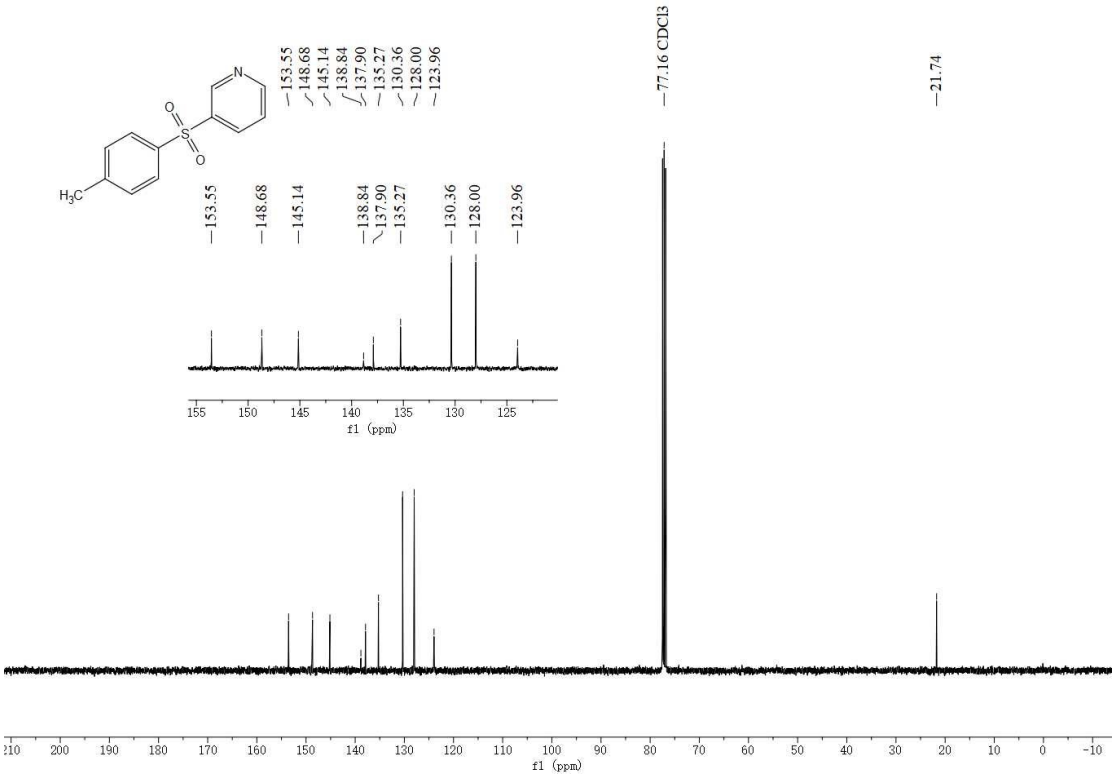
F
F
F
O
S
O
F
F
F
H3C
00
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
-30
f1 (ppm)

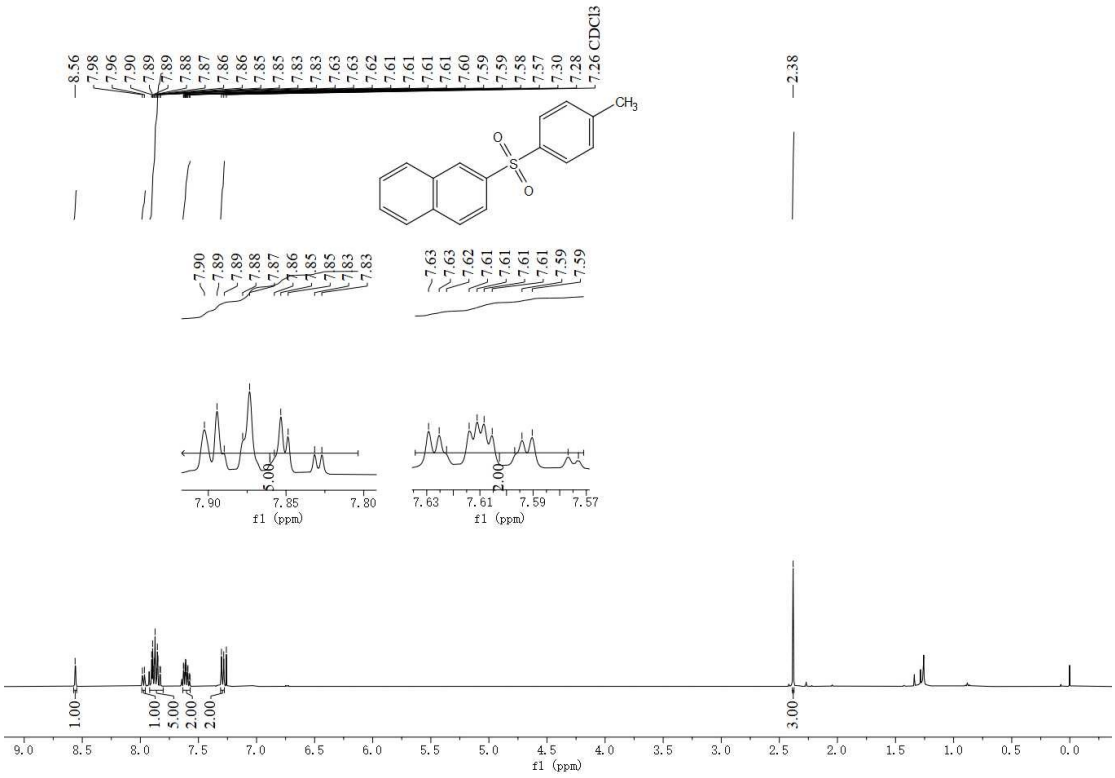
^{1}H NMR spectrum of **3-tosylpyridine (14)**



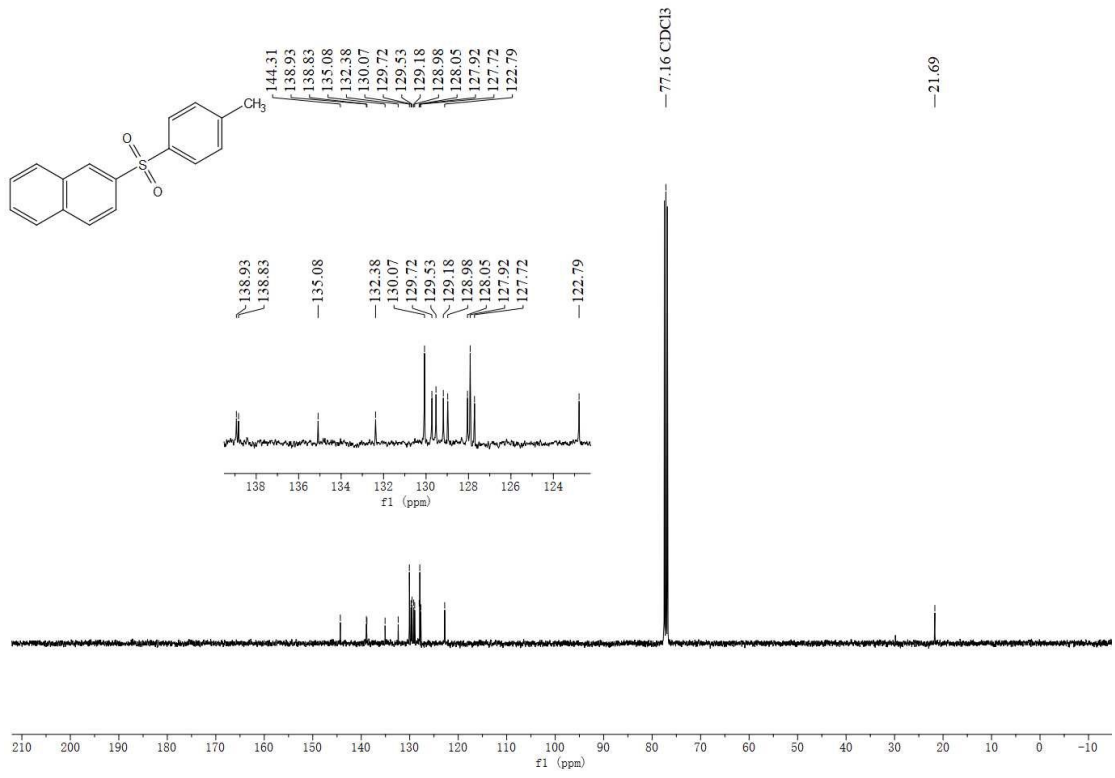
^{13}C NMR spectrum of **3-tosylpyridine (14)**



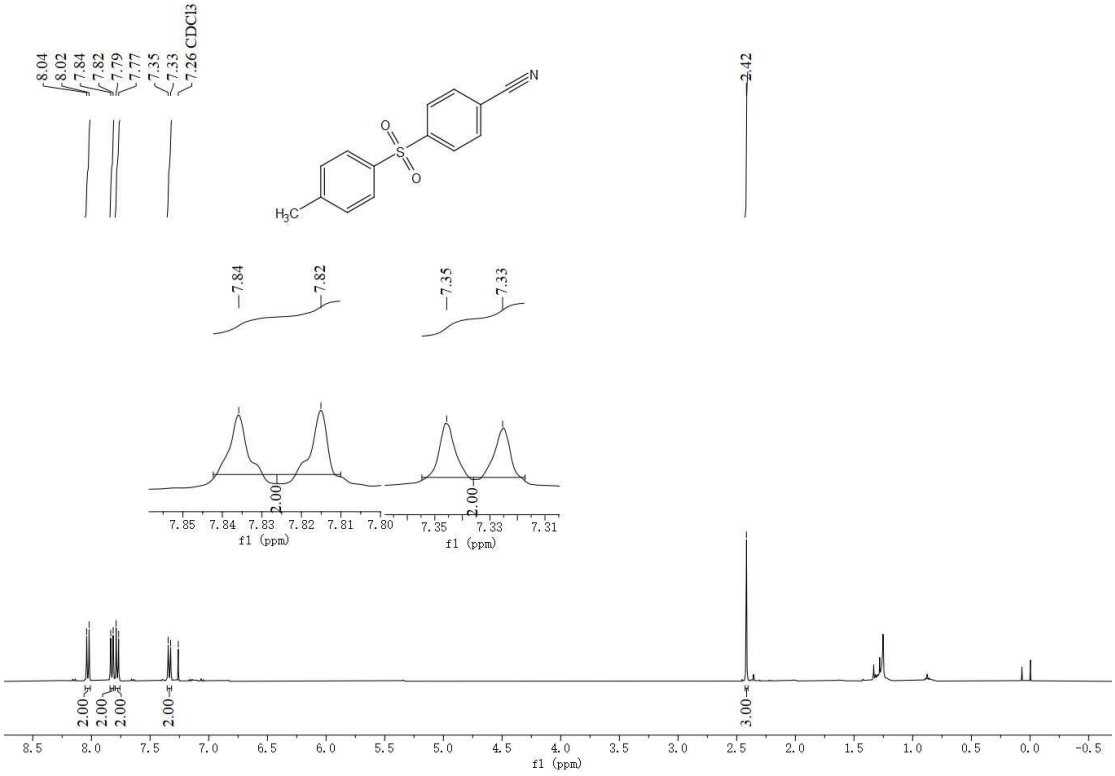
^{1}H NMR spectrum of **2-tosylnaphthalene (15)**



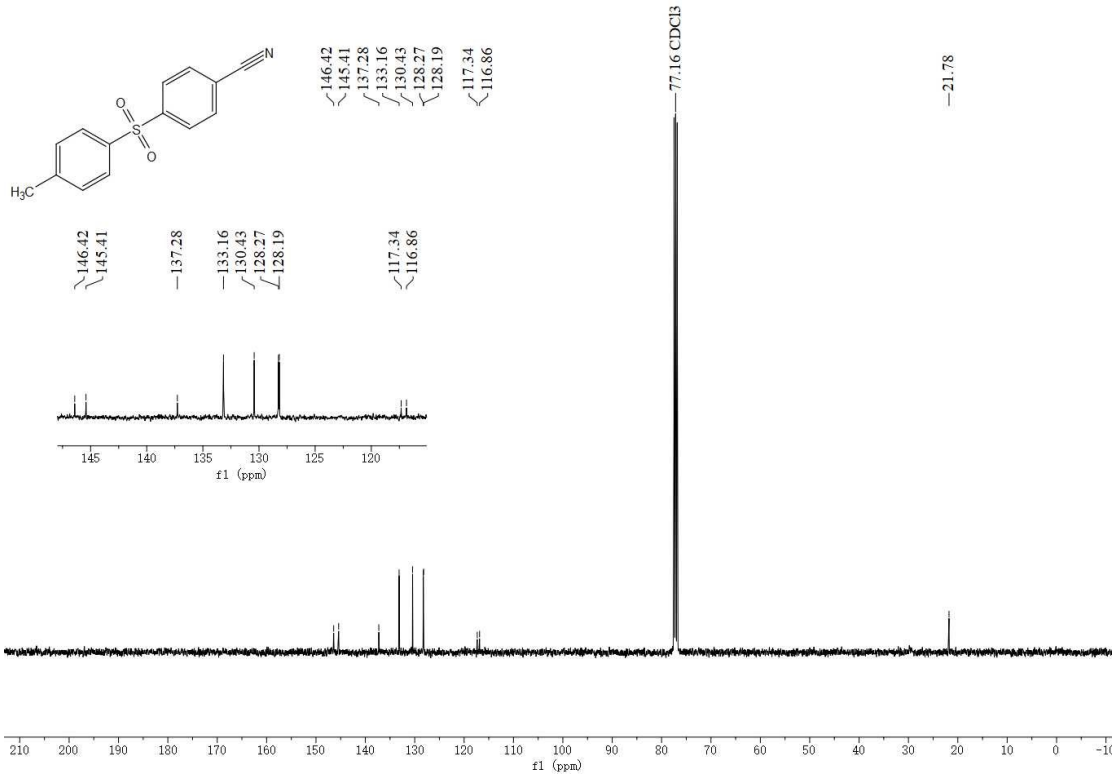
^{13}C NMR spectrum of **2-tosylnaphthalene (15)**



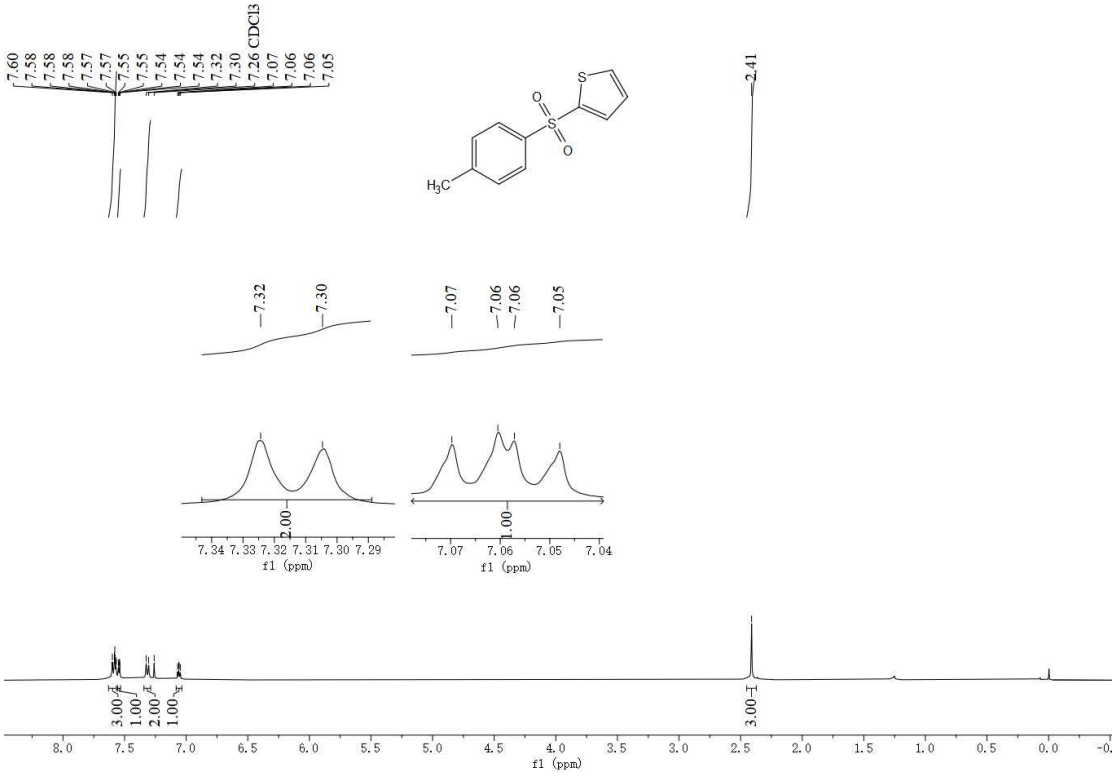
^{1}H NMR spectrum of **4-tosylbenzonitrile (16)**



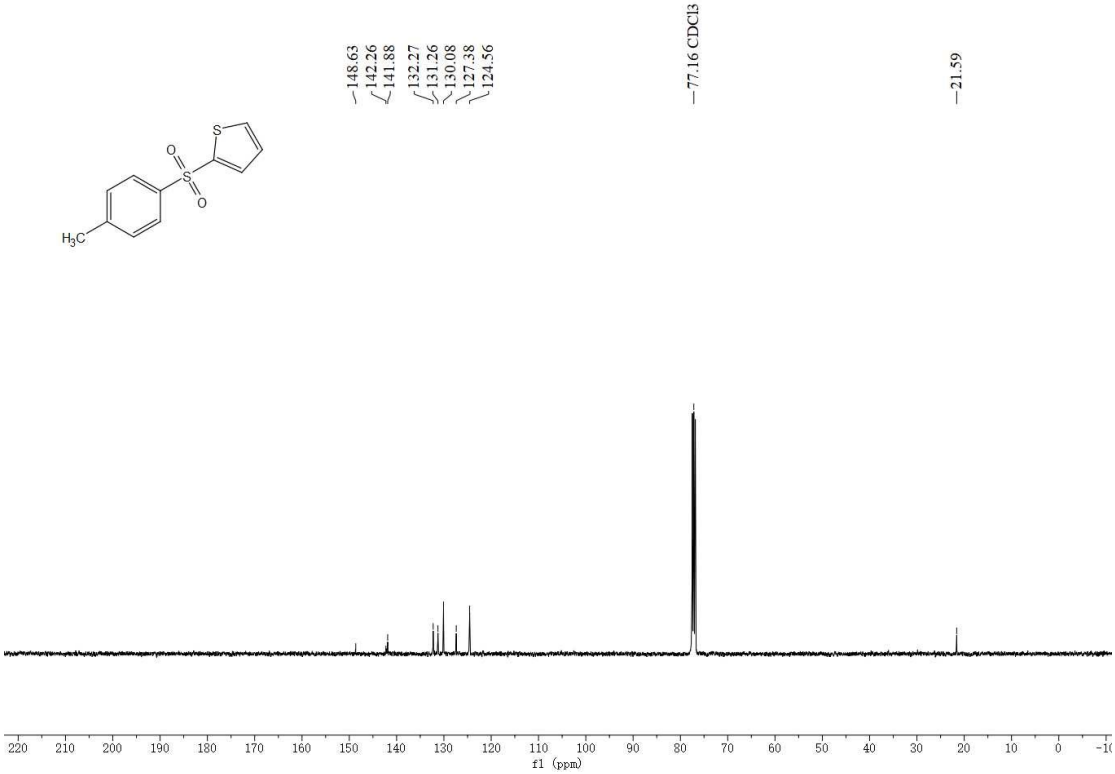
^{13}C NMR spectrum of **4-tosylbenzonitrile (16)**



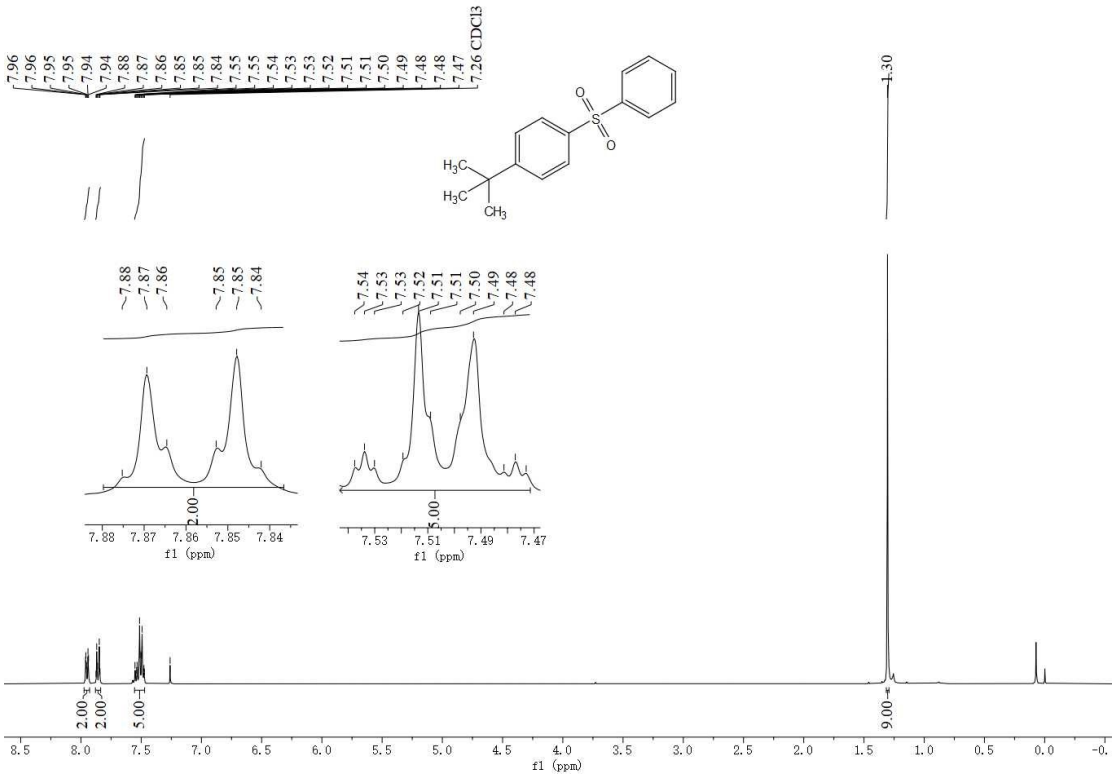
^{1}H NMR spectrum of **2-tosylthiophene (17)**



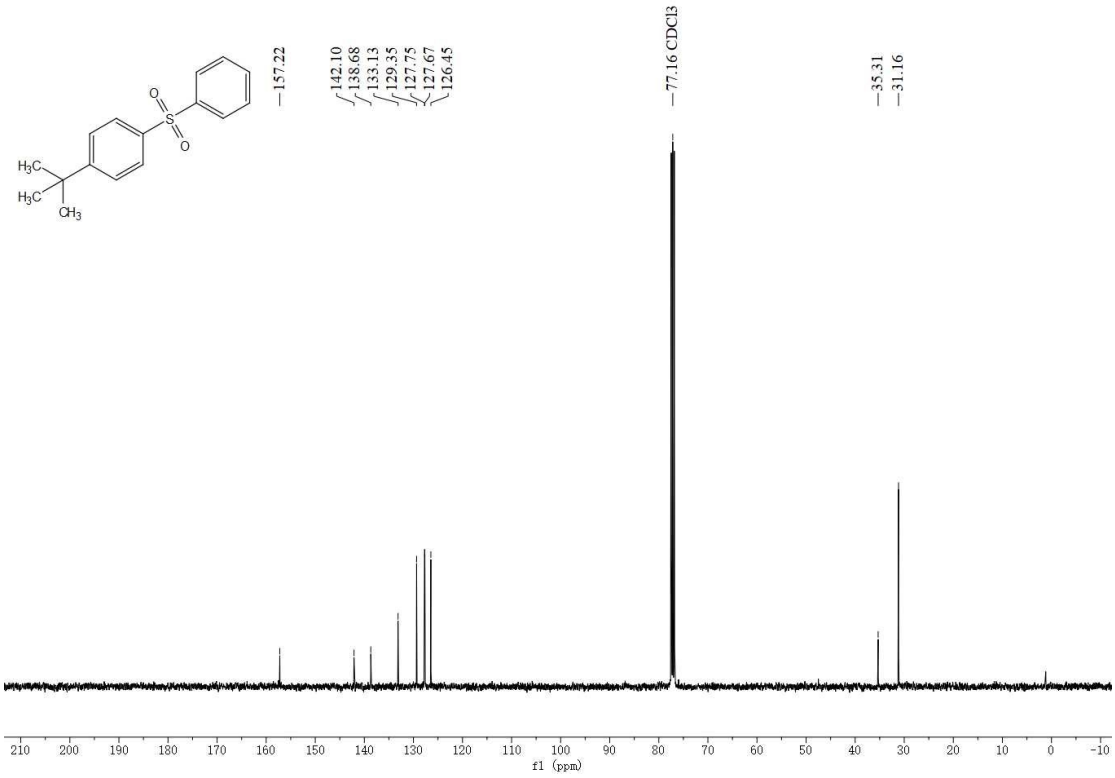
^{13}C NMR spectrum of **2-tosylthiophene (17)**



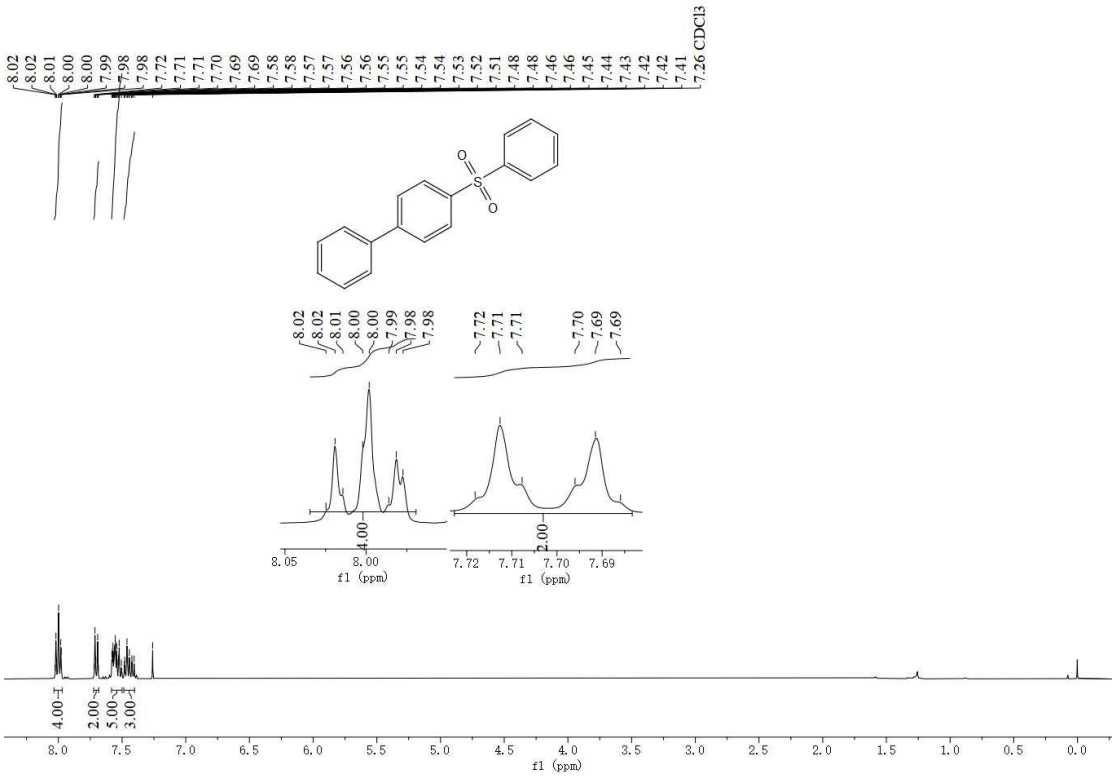
^{1}H NMR spectrum of **1-(tert-butyl)-4-(phenylsulfonyl)benzene (18)**



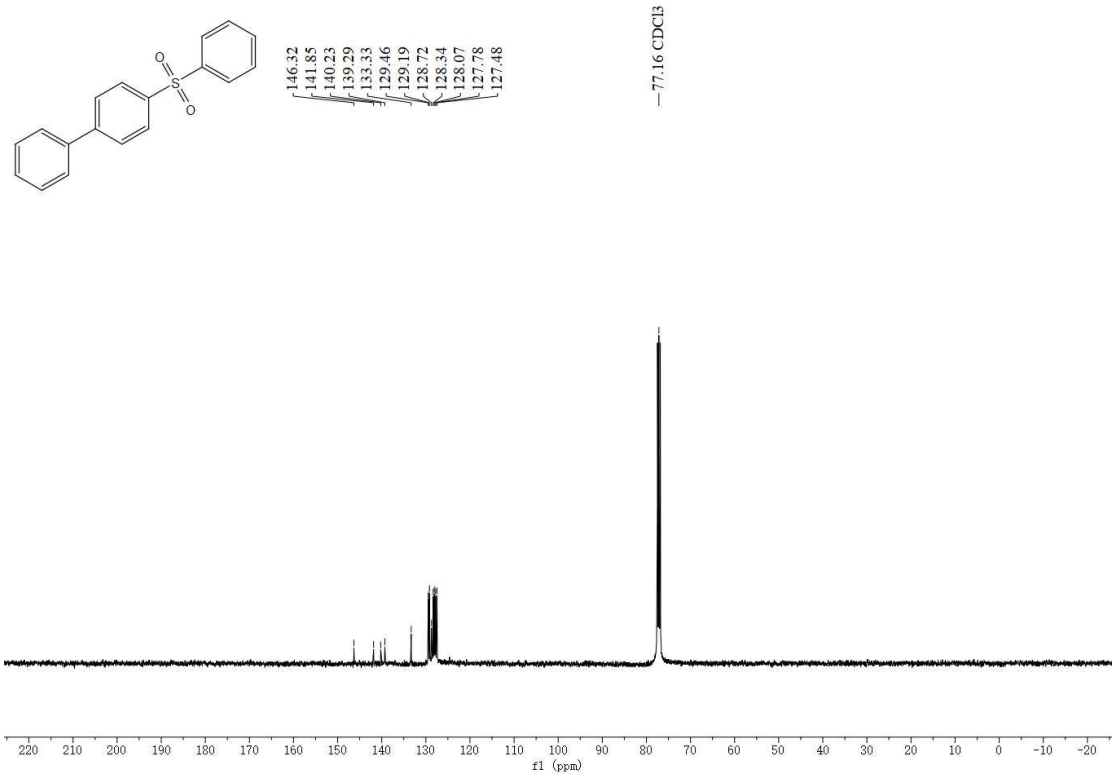
^{13}C NMR spectrum of **1-(tert-butyl)-4-(phenylsulfonyl)benzene (18)**



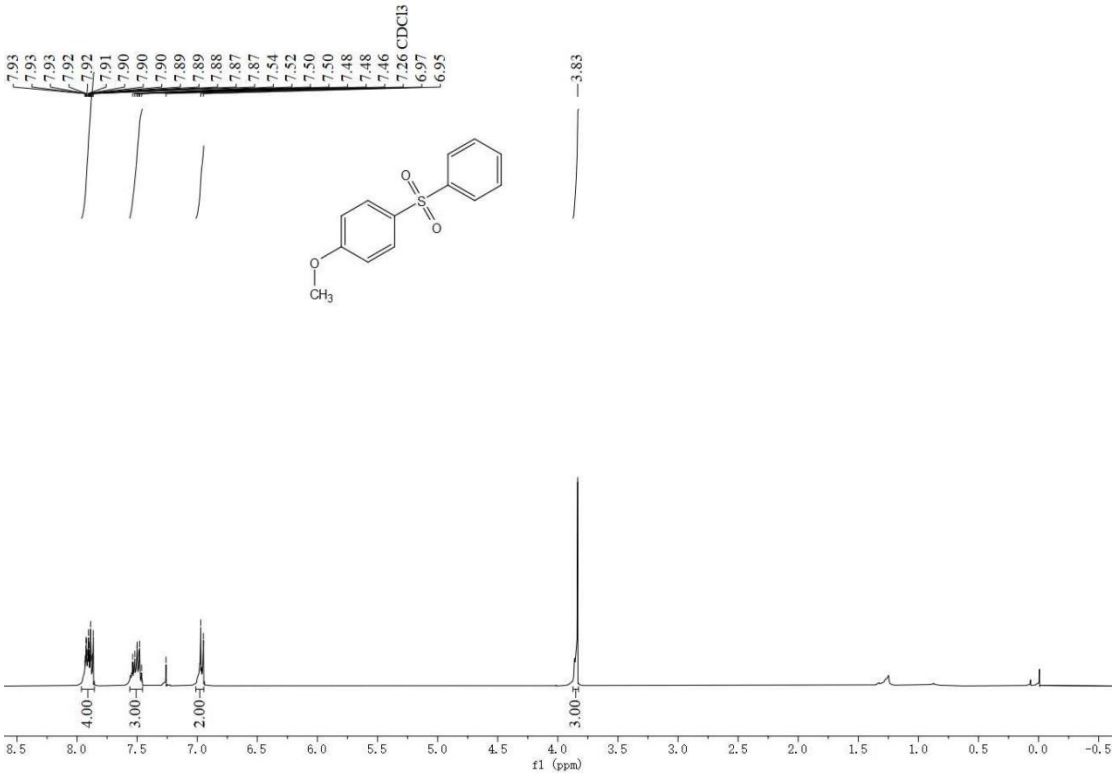
^{1}H NMR spectrum of **4-(phenylsulfonyl)-1,1'-biphenyl (19)**



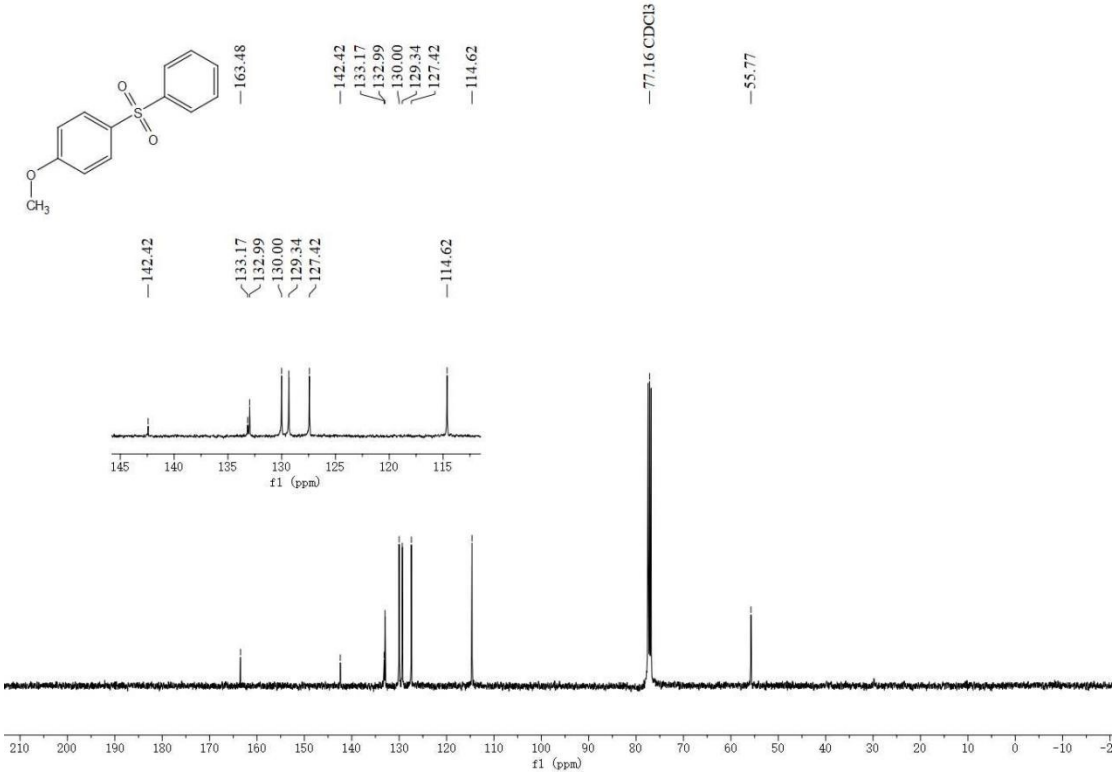
^{13}C NMR spectrum of **4-(phenylsulfonyl)-1,1'-biphenyl (19)**



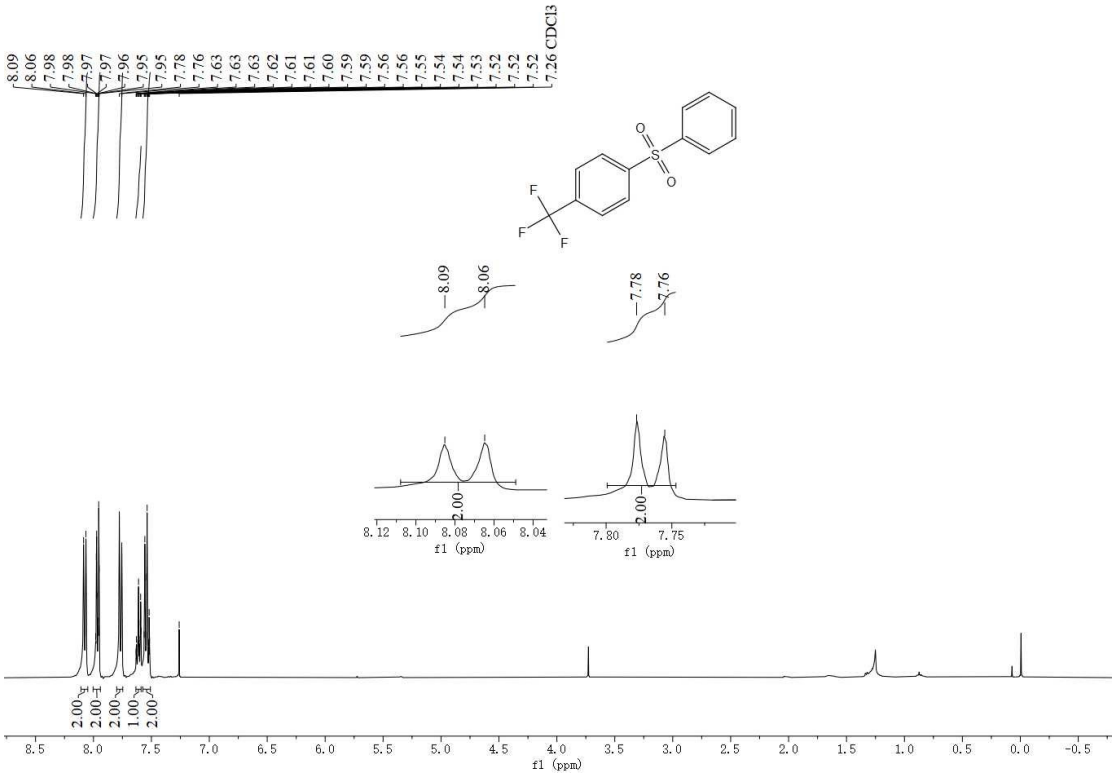
^{1}H NMR spectrum of **1-methoxy-4-(phenylsulfonyl)benzene (20)**



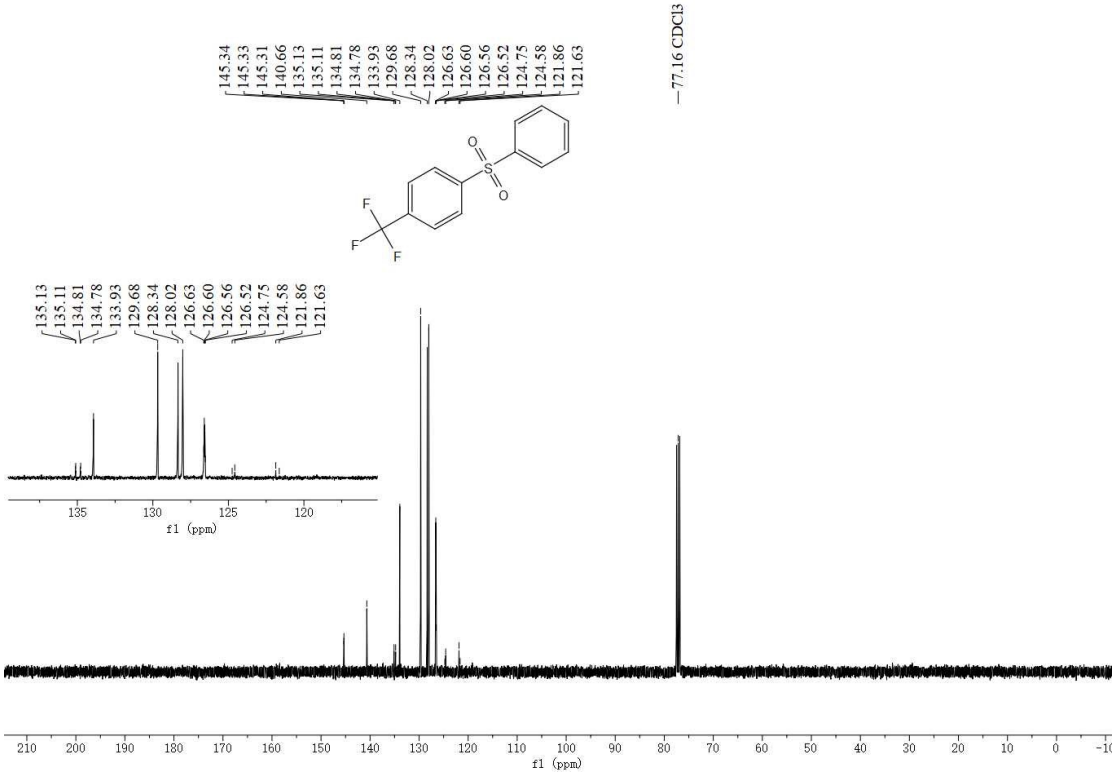
^{13}C NMR spectrum of **1-methoxy-4-(phenylsulfonyl)benzene (20)**



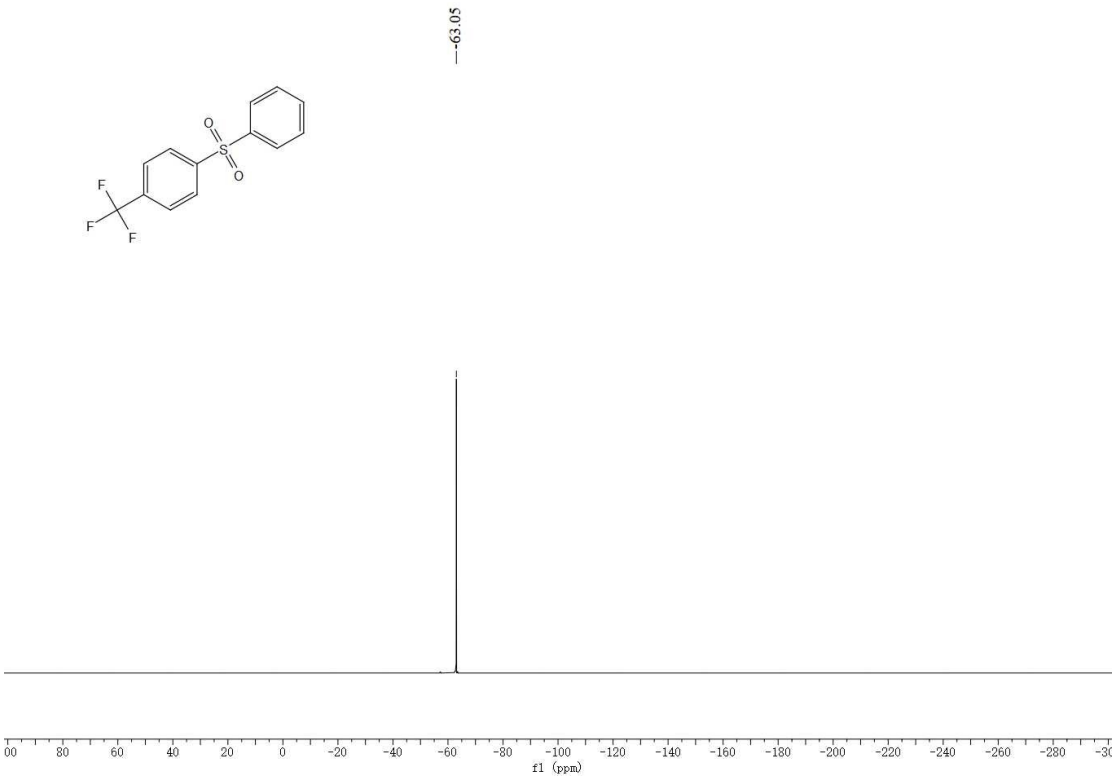
^{1}H NMR spectrum of **1-(phenylsulfonyl)-4-(trifluoromethyl)benzene (21)**



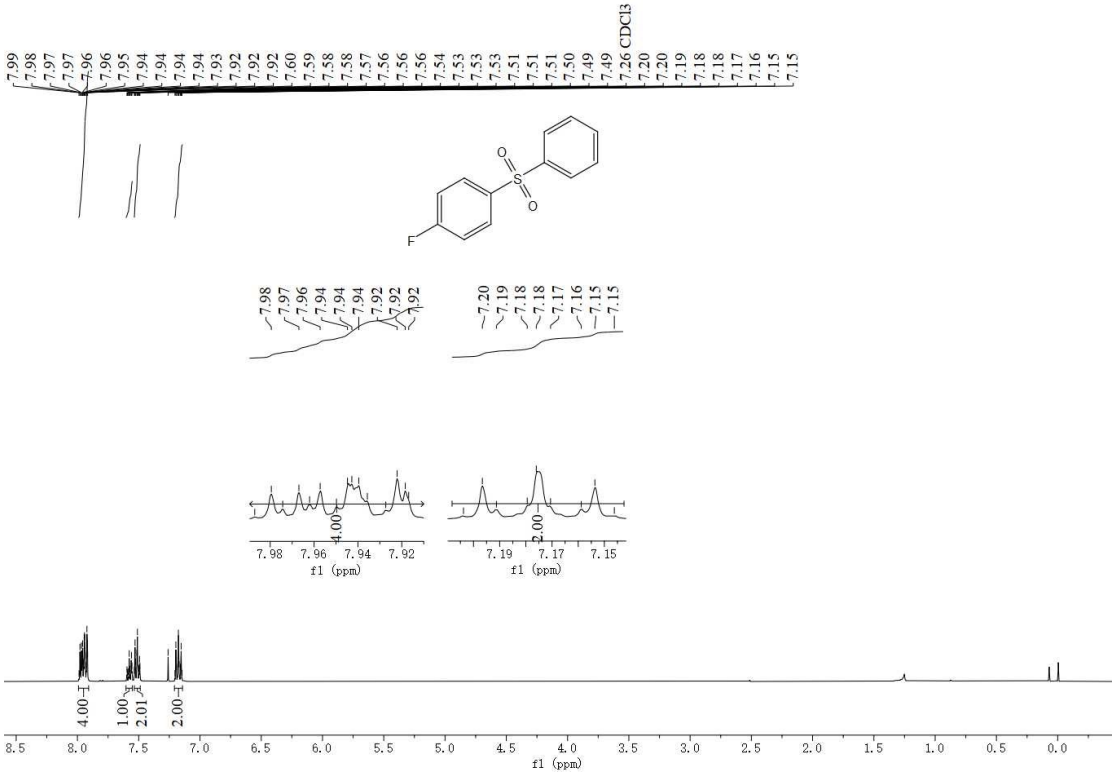
^{13}C NMR spectrum of **1-(phenylsulfonyl)-4-(trifluoromethyl)benzene (21)**



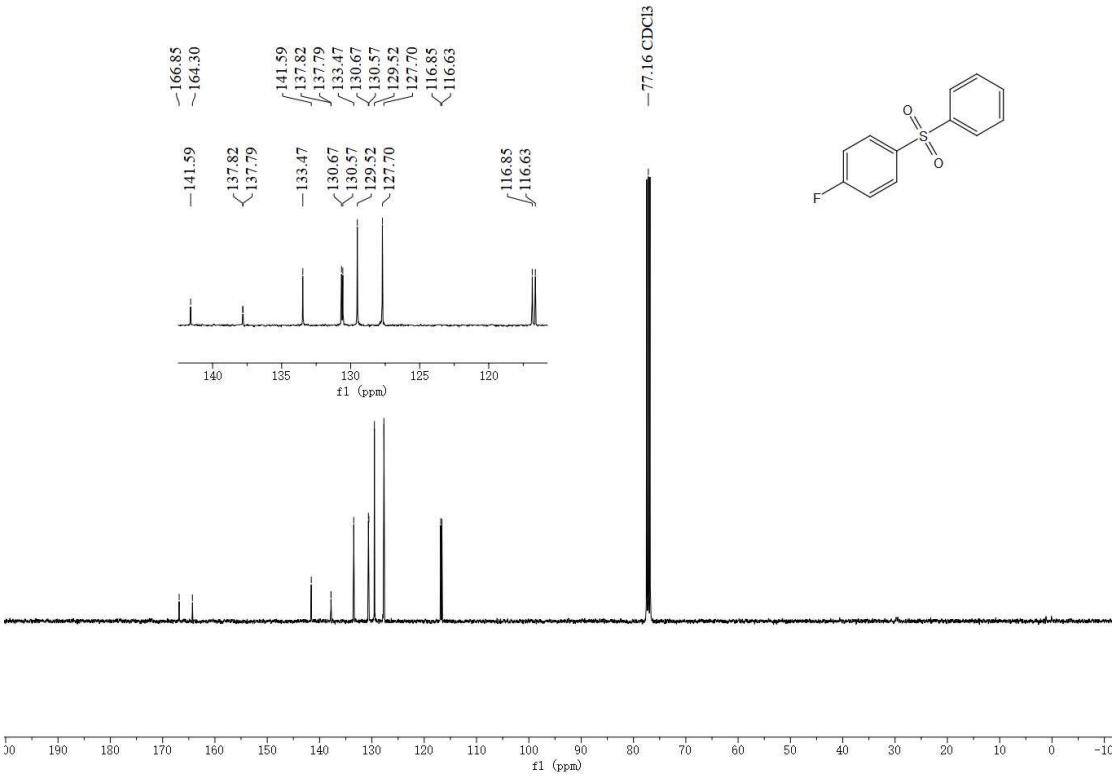
^{19}F NMR spectrum of **1-(phenylsulfonyl)-4-(trifluoromethyl)benzene (21)**



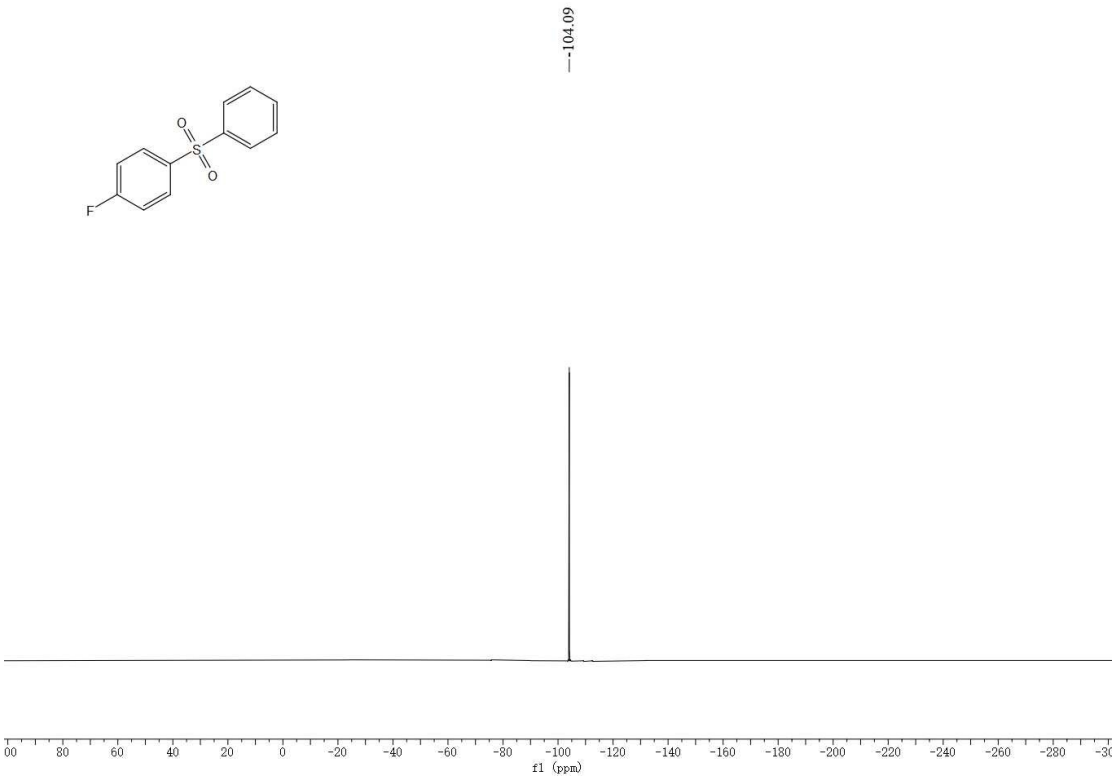
^{1}H NMR spectrum of **1-fluoro-4-(phenylsulfonyl)benzene (22)**



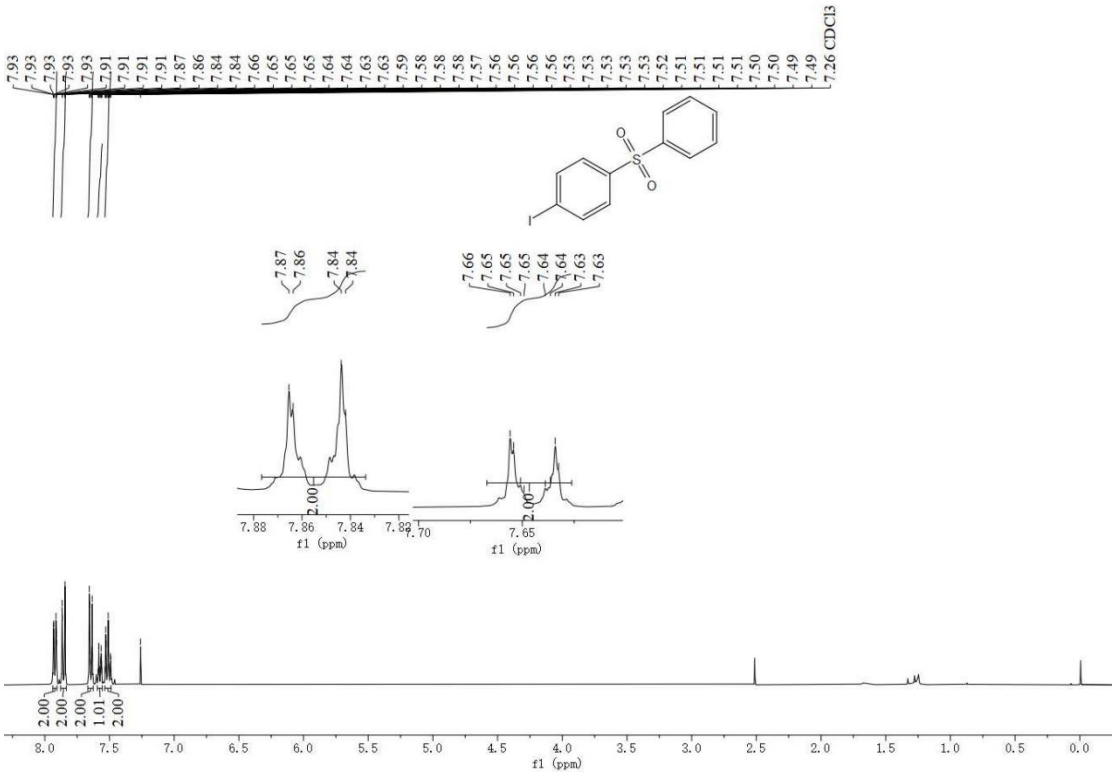
^{13}C NMR spectrum of **1-fluoro-4-(phenylsulfonyl)benzene (22)**



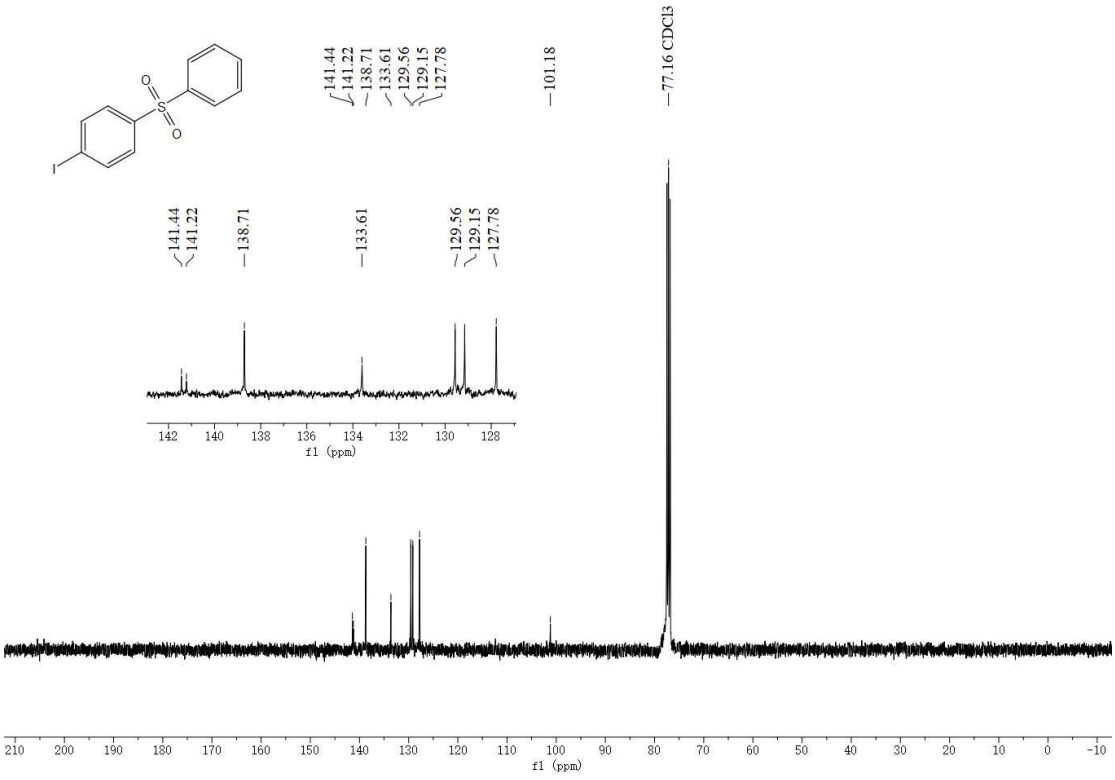
^{19}F NMR spectrum of **1-fluoro-4-(phenylsulfonyl)benzene (22)**



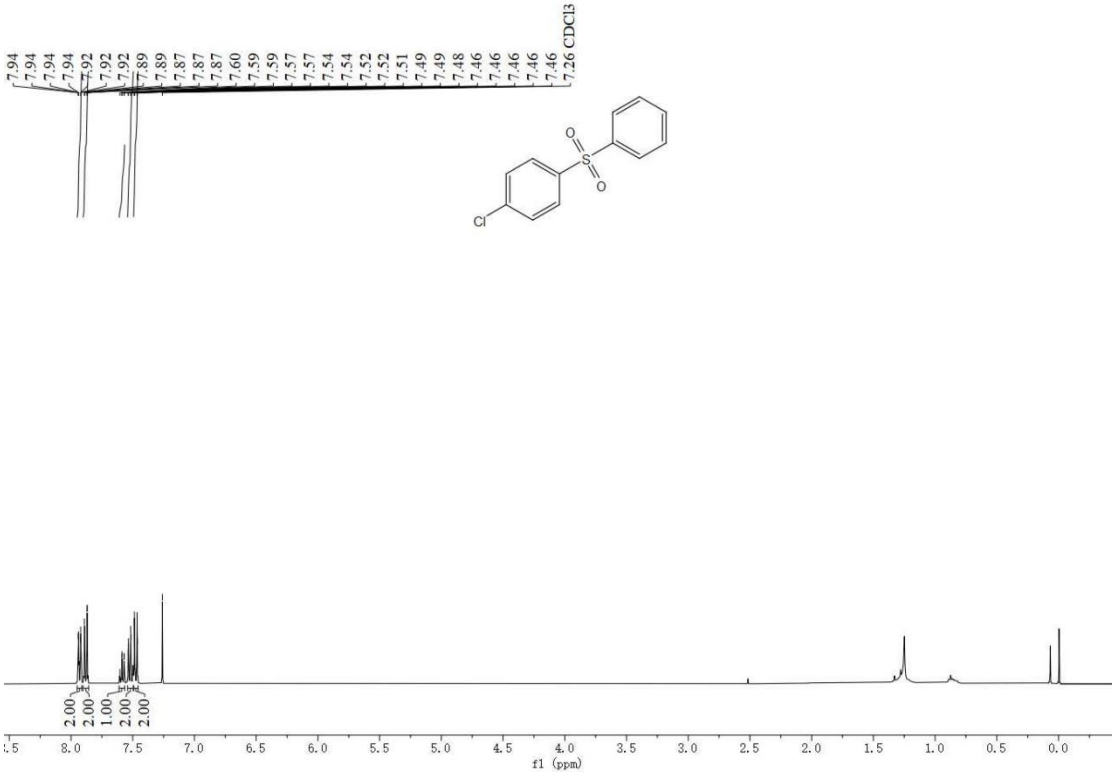
^{1}H NMR spectrum of **1-iodo-4-(phenylsulfonyl)benzene (23)**



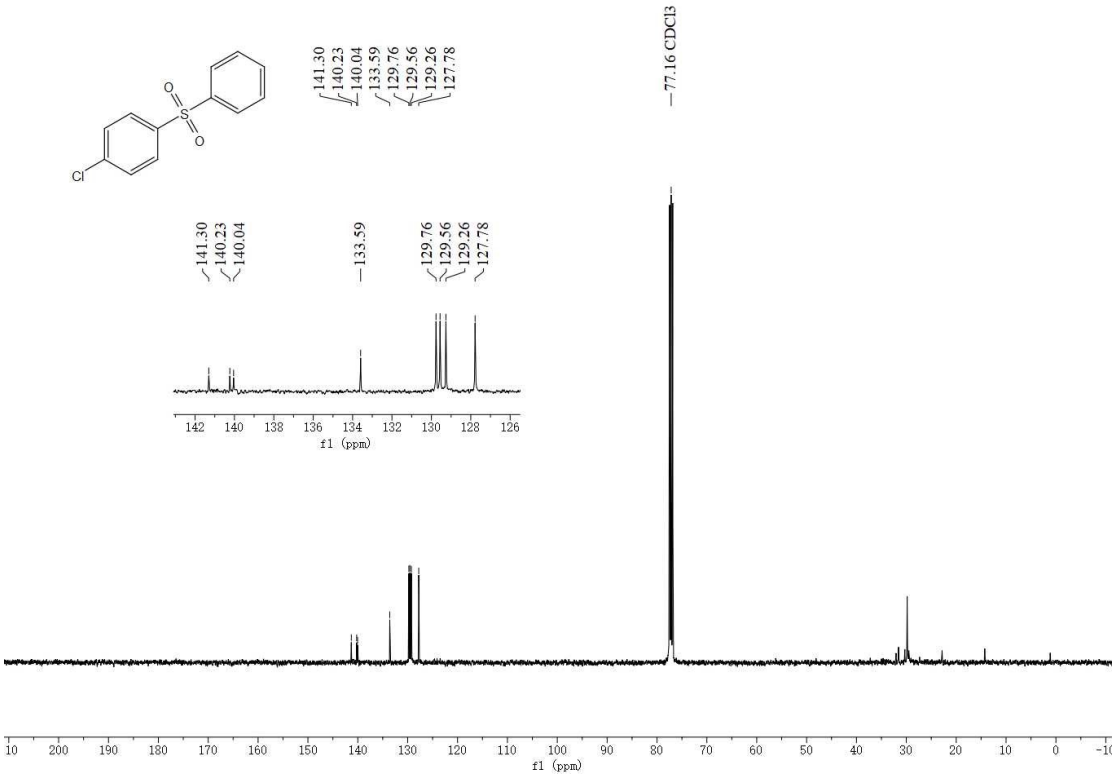
^{13}C NMR spectrum of **1-iodo-4-(phenylsulfonyl)benzene (23)**



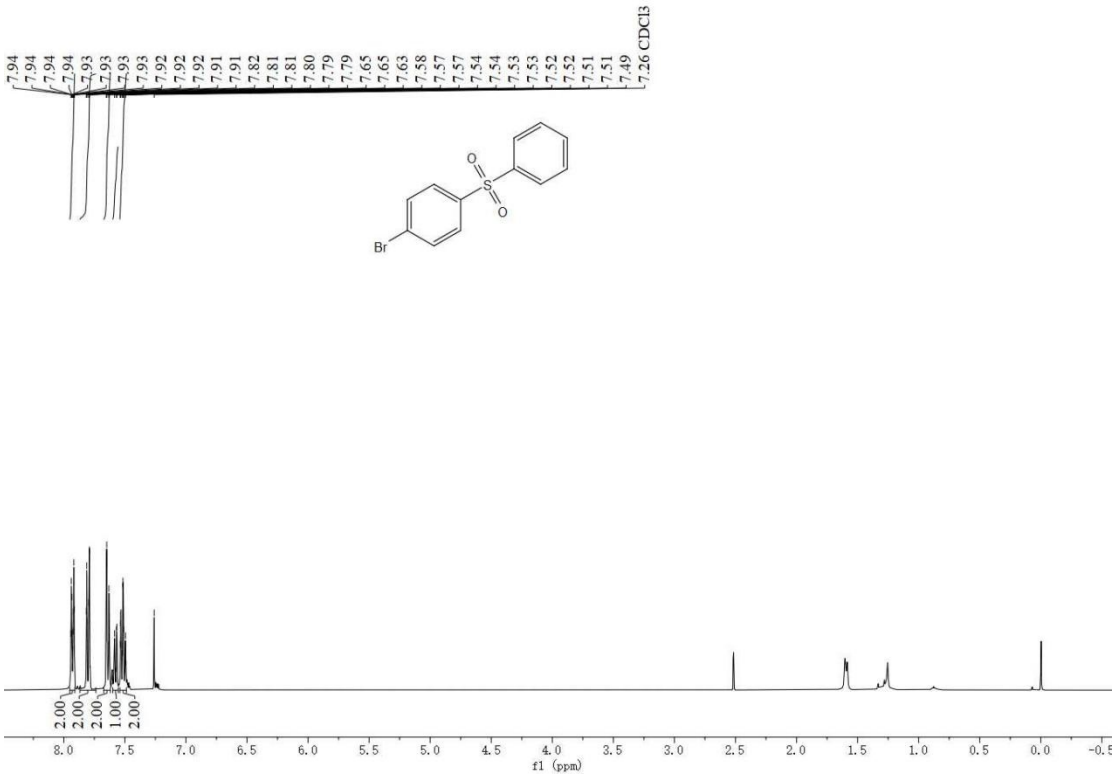
^{1}H NMR spectrum of **1-chloro-4-(phenylsulfonyl)benzene (24)**



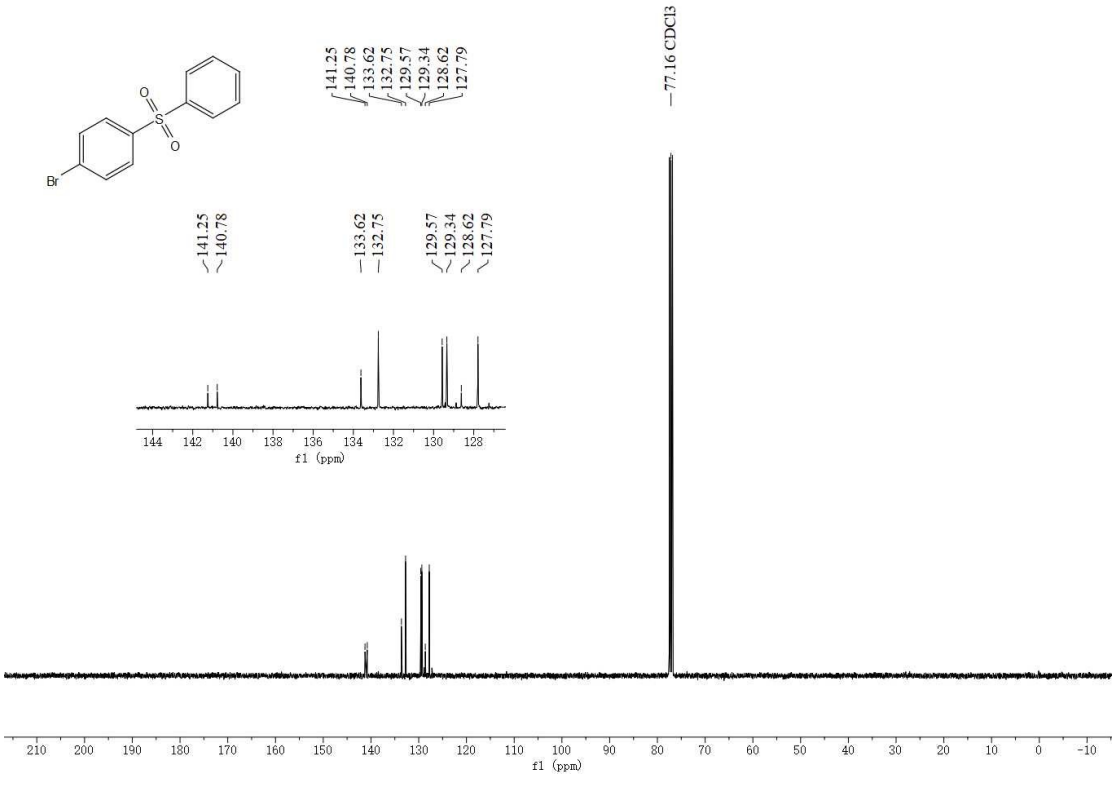
^{13}C NMR spectrum of **1-chloro-4-(phenylsulfonyl)benzene (24)**



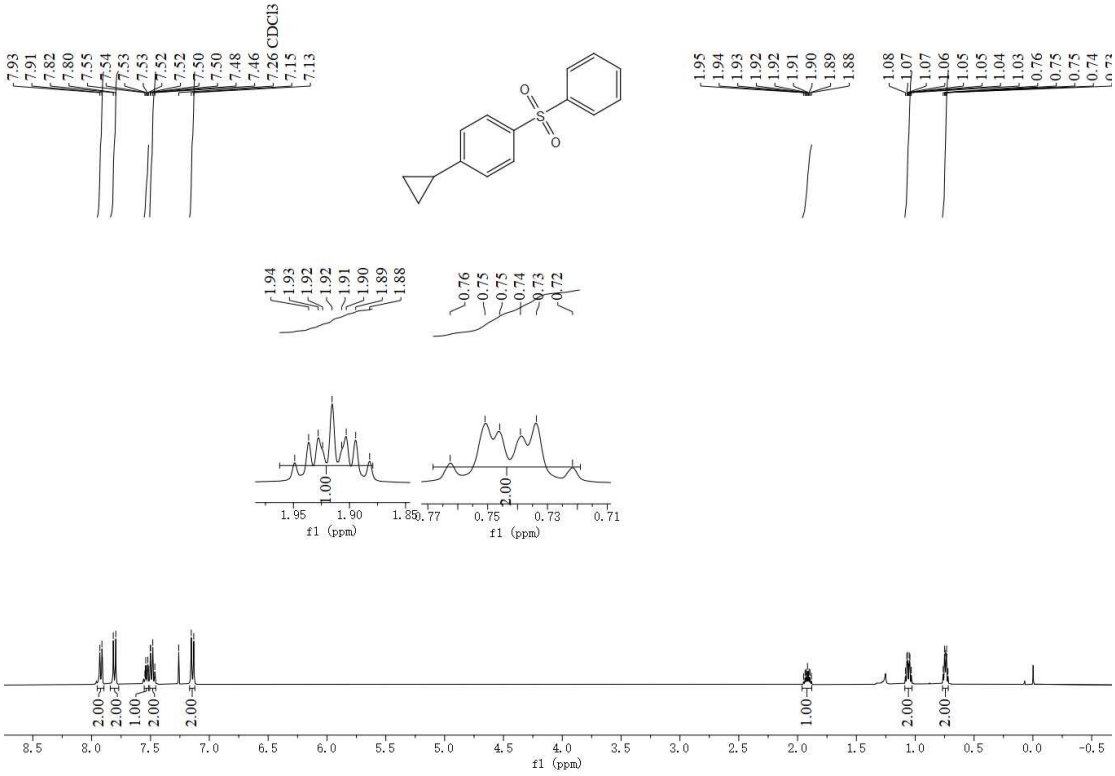
^{1}H NMR spectrum of **1-bromo-4-(phenylsulfonyl)benzene (25)**



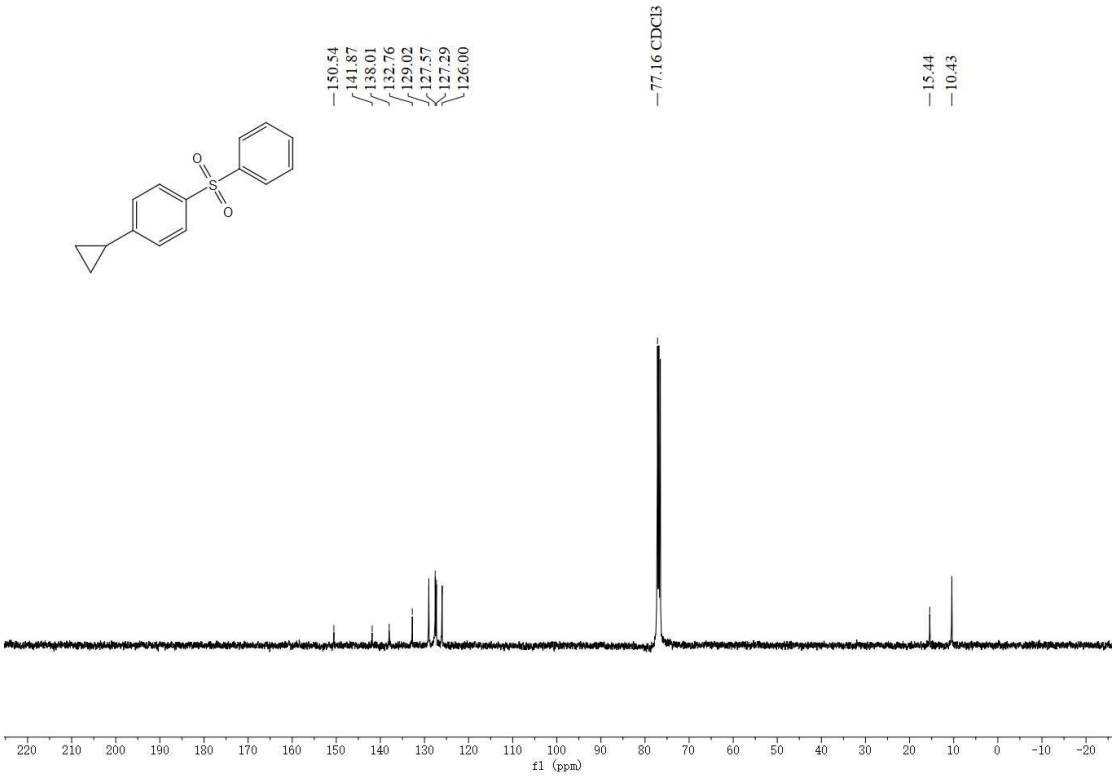
^{13}C NMR spectrum of **1-bromo-4-(phenylsulfonyl)benzene (25)**



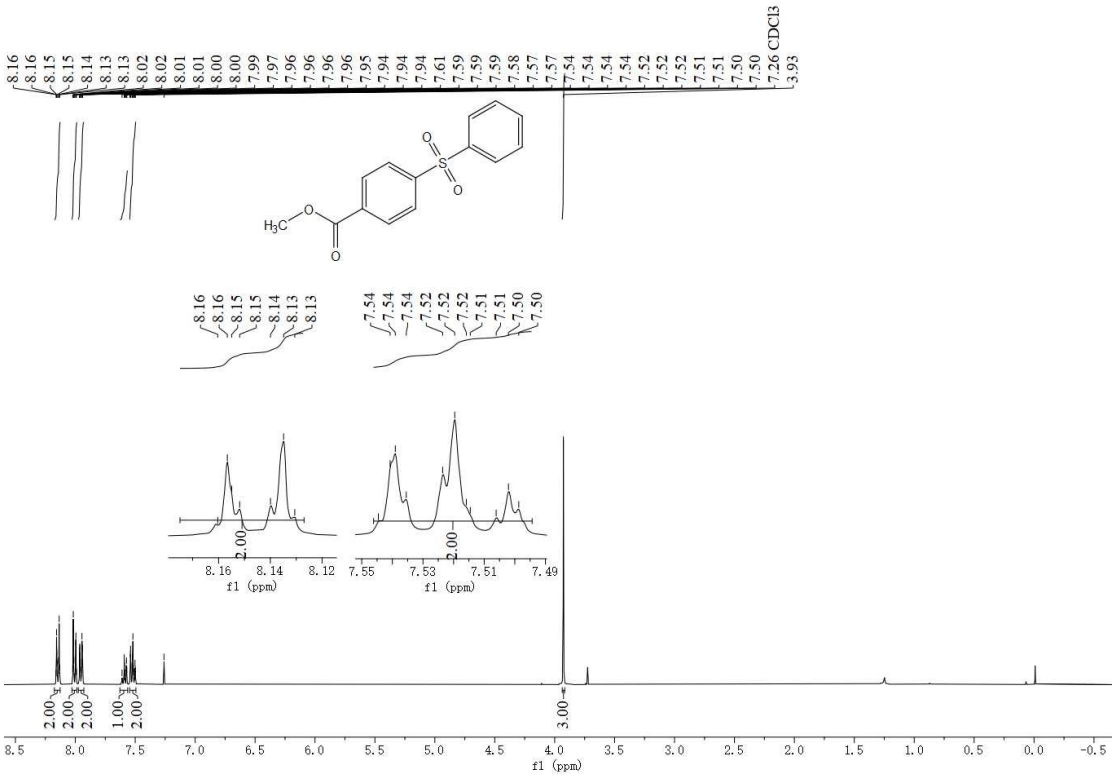
^{1}H NMR spectrum of **1-cyclopropyl-4-(phenylsulfonyl)benzene (26)**



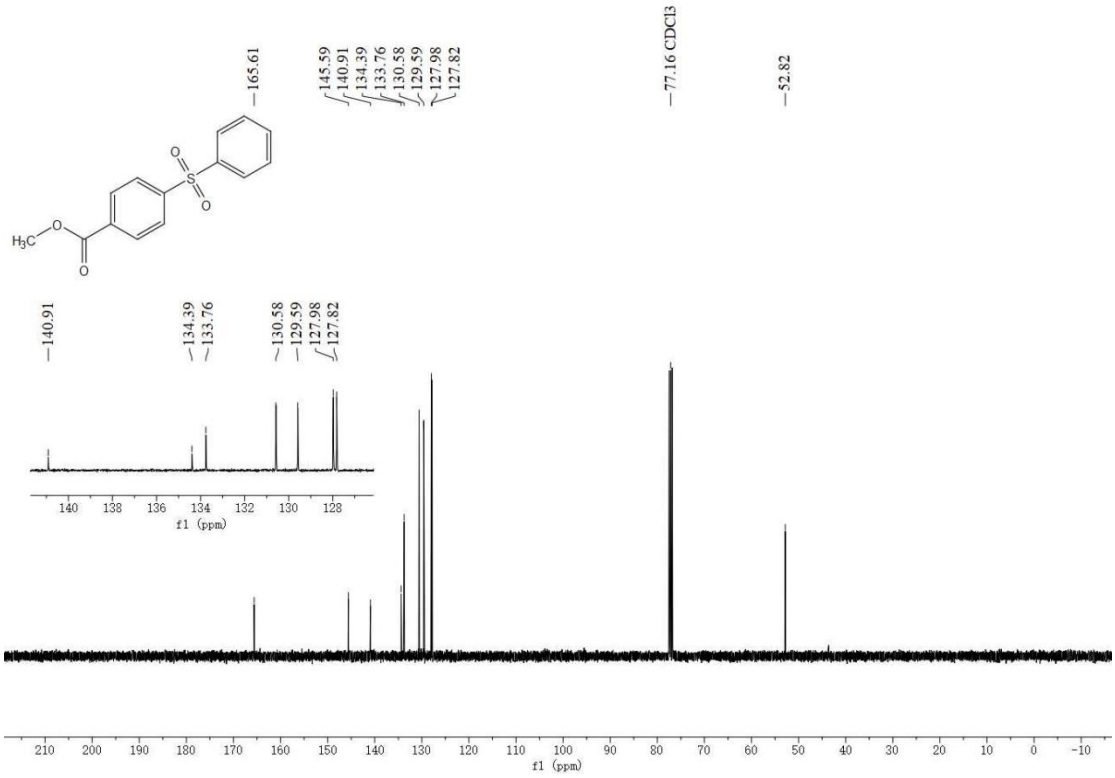
^{13}C NMR spectrum of **1-cyclopropyl-4-(phenylsulfonyl)benzene (26)**



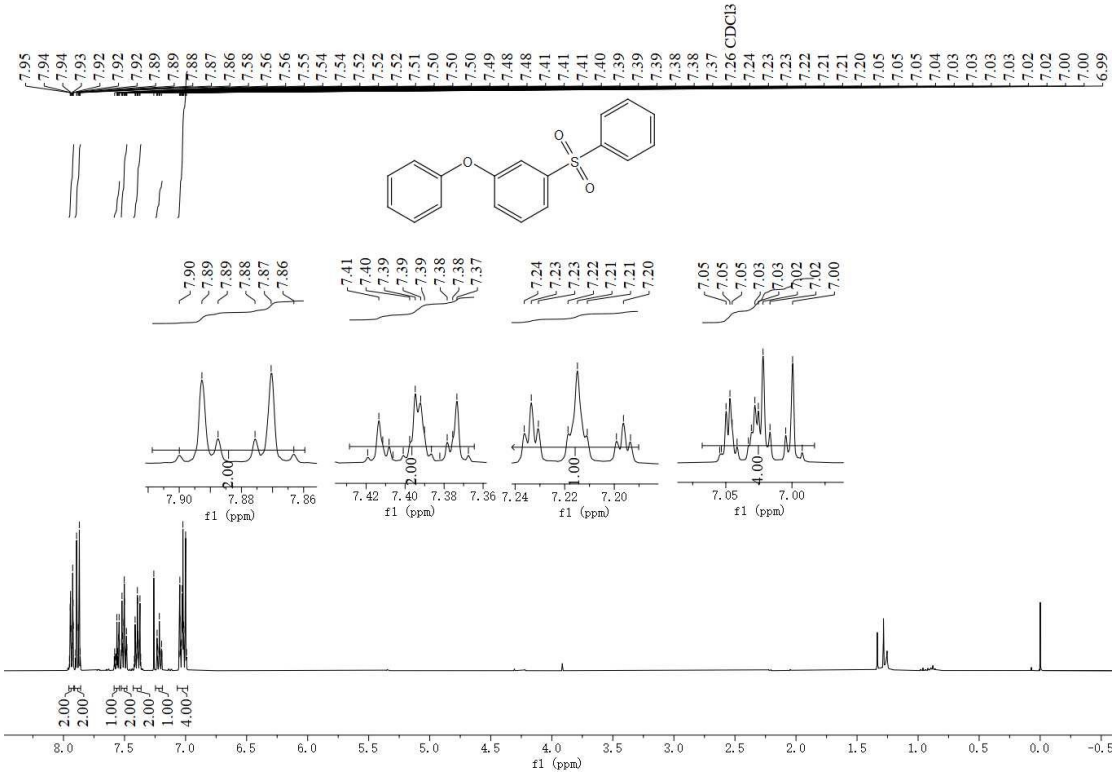
^{1}H NMR spectrum of **methyl 4-(phenylsulfonyl)benzoate (27)**



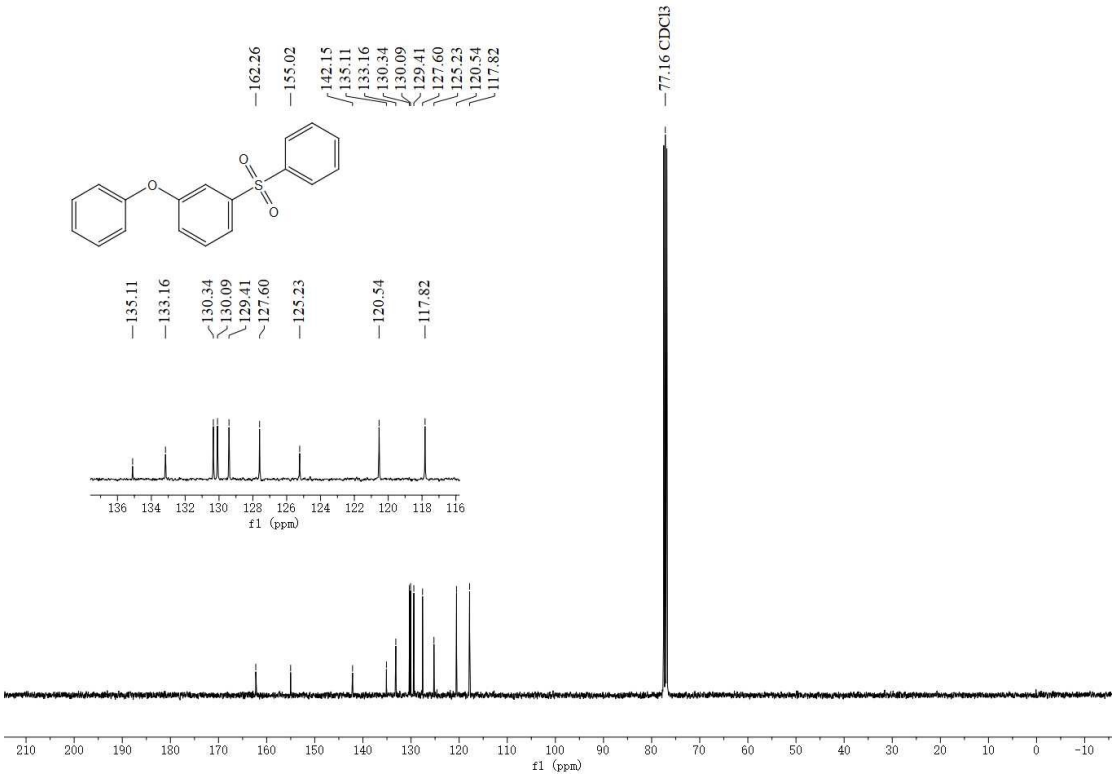
^{13}C NMR spectrum of **methyl 4-(phenylsulfonyl)benzoate (27)**



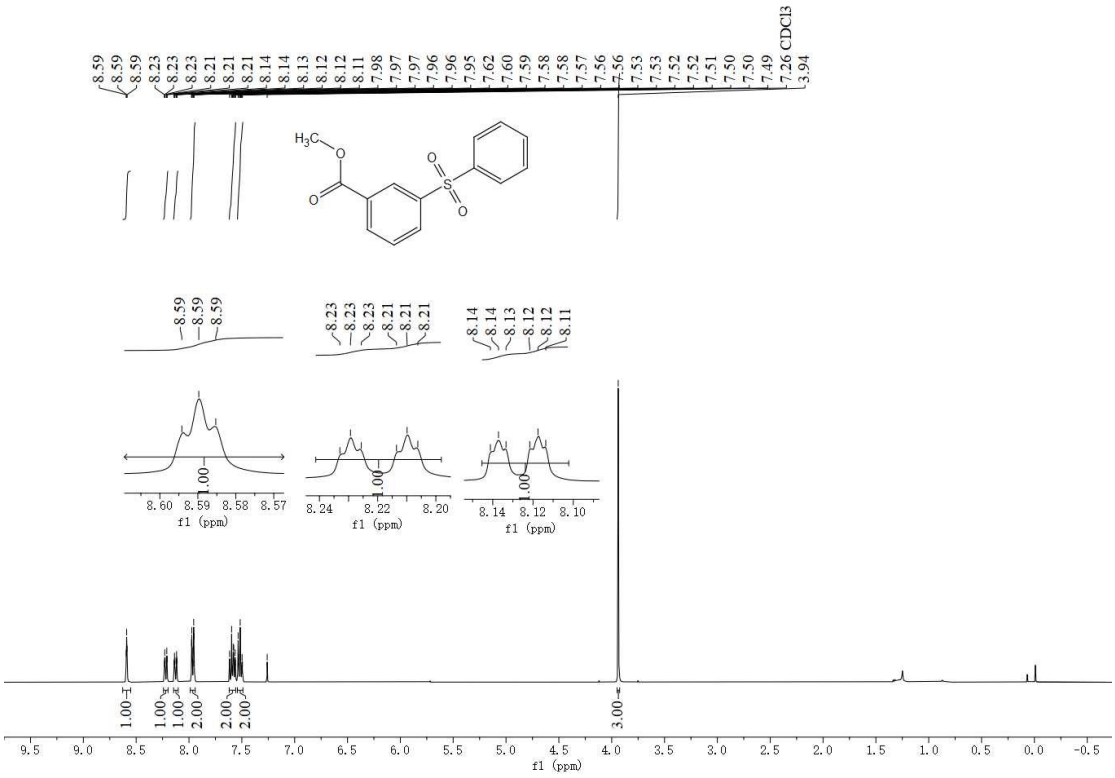
^{1}H NMR spectrum of **1-phenoxy-3-(phenylsulfonyl)benzene (28)**



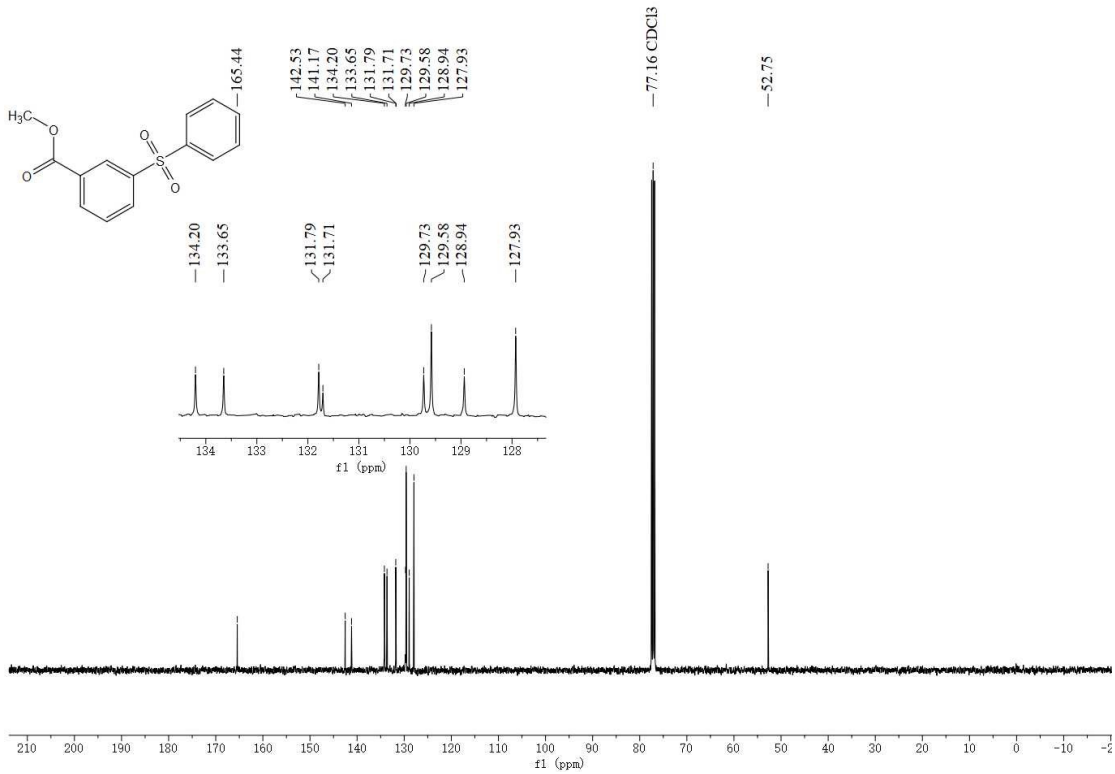
^{13}C NMR spectrum of **1-phenoxy-3-(phenylsulfonyl)benzene (28)**



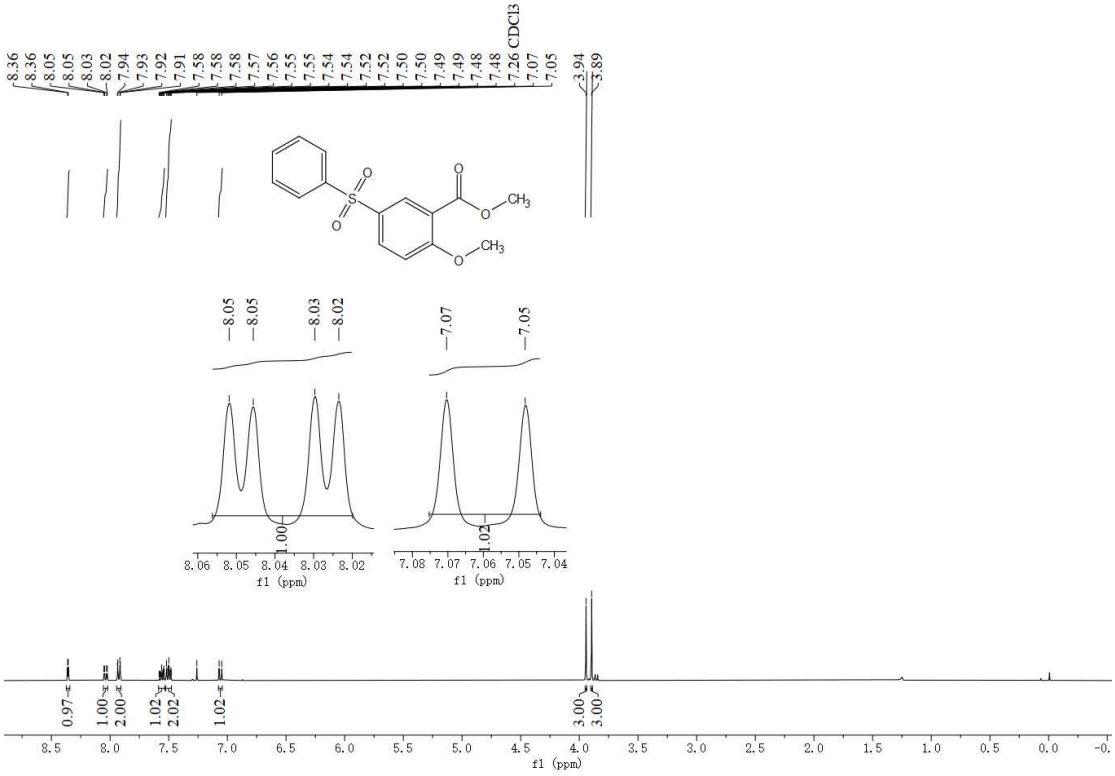
^{1}H NMR spectrum of **methyl 3-(phenylsulfonyl)benzoate (29)**



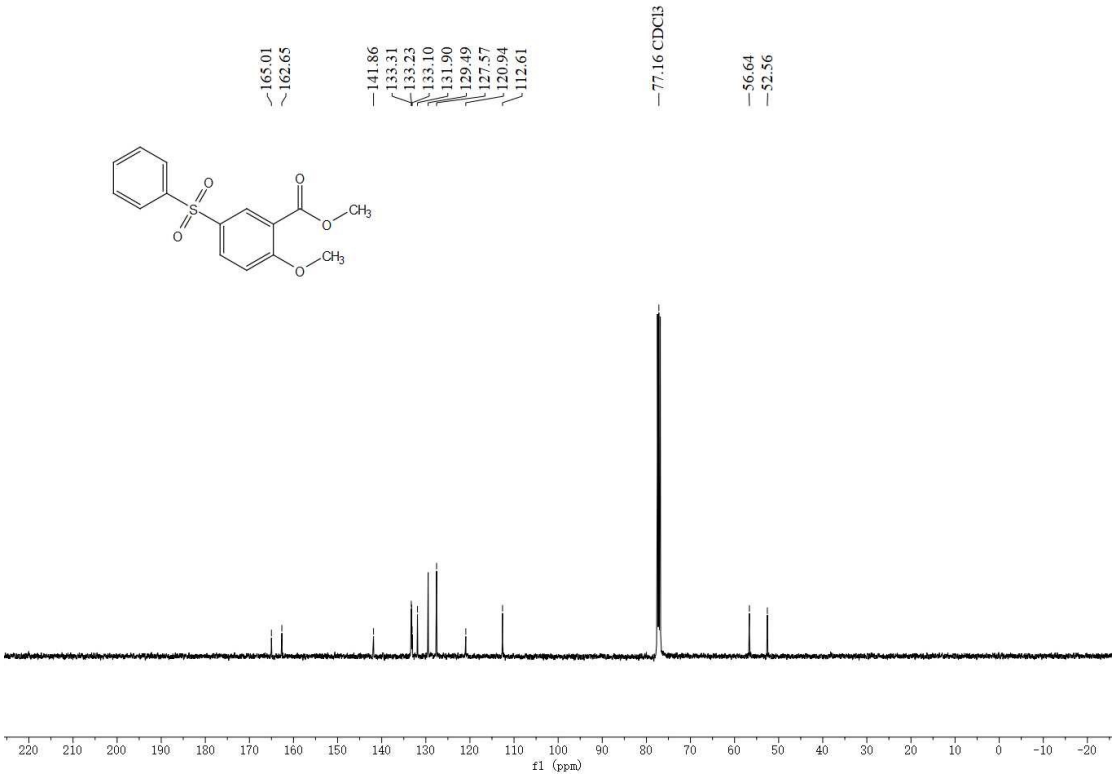
^{13}C NMR spectrum of **methyl 3-(phenylsulfonyl)benzoate (29)**



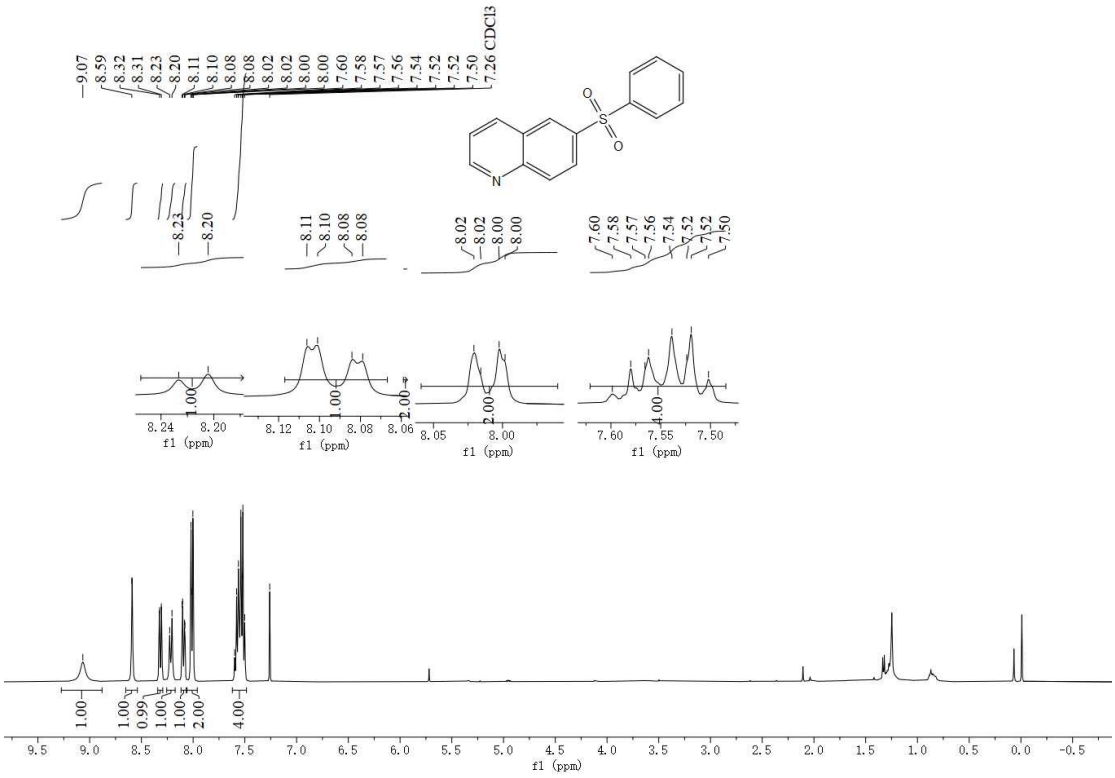
^{1}H NMR spectrum of **methyl 2-methoxy-5-(phenylsulfonyl)benzoate (30)**



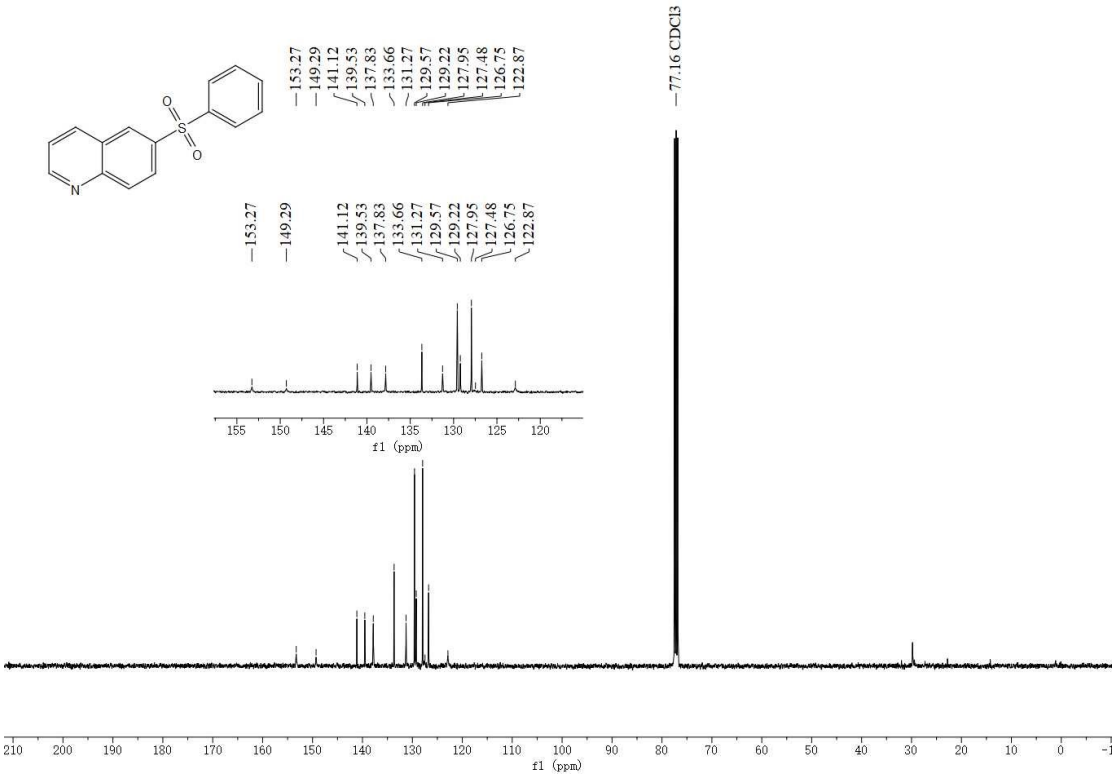
^{13}C NMR spectrum of **methyl 2-methoxy-5-(phenylsulfonyl)benzoate (30)**



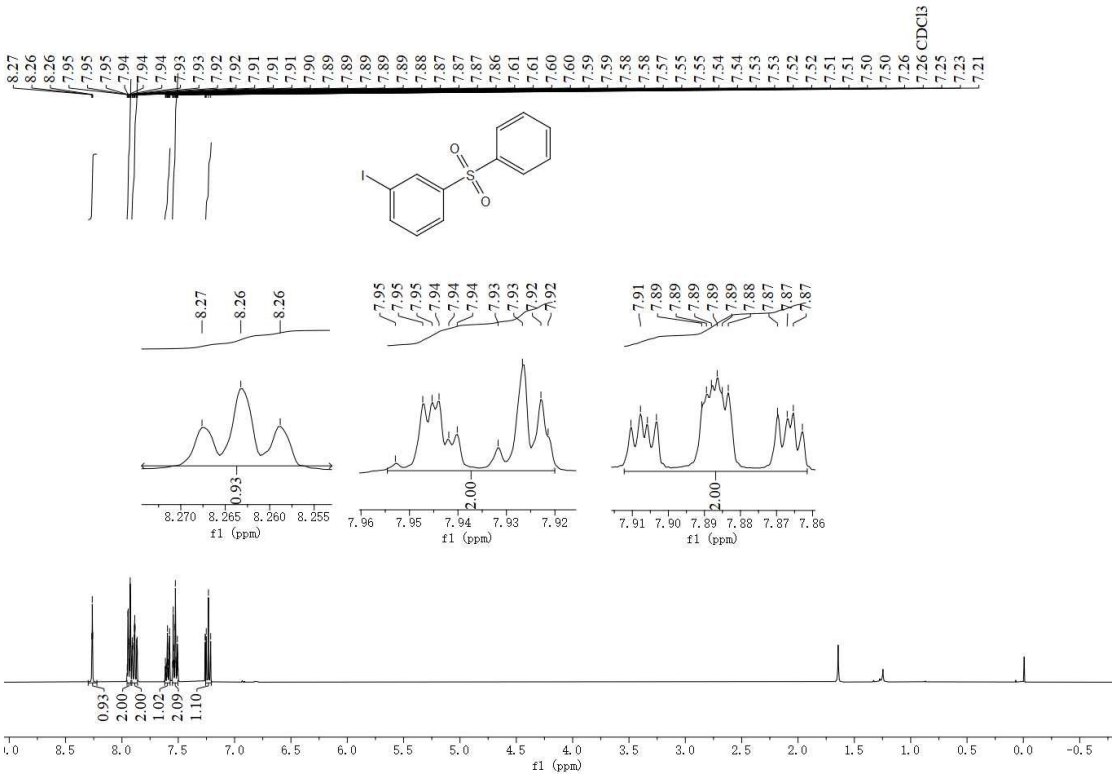
^{1}H NMR spectrum of **6-(phenylsulfonyl)quinoline (31)**



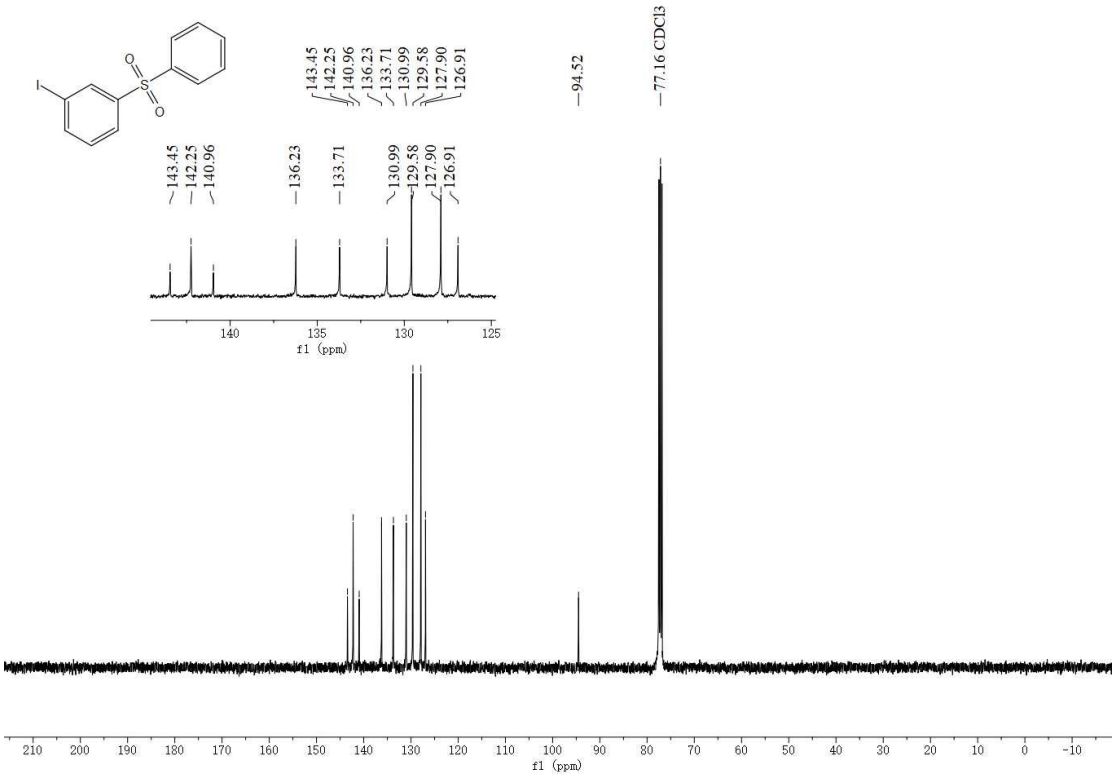
^{13}C NMR spectrum of **6-(phenylsulfonyl)quinoline (31)**



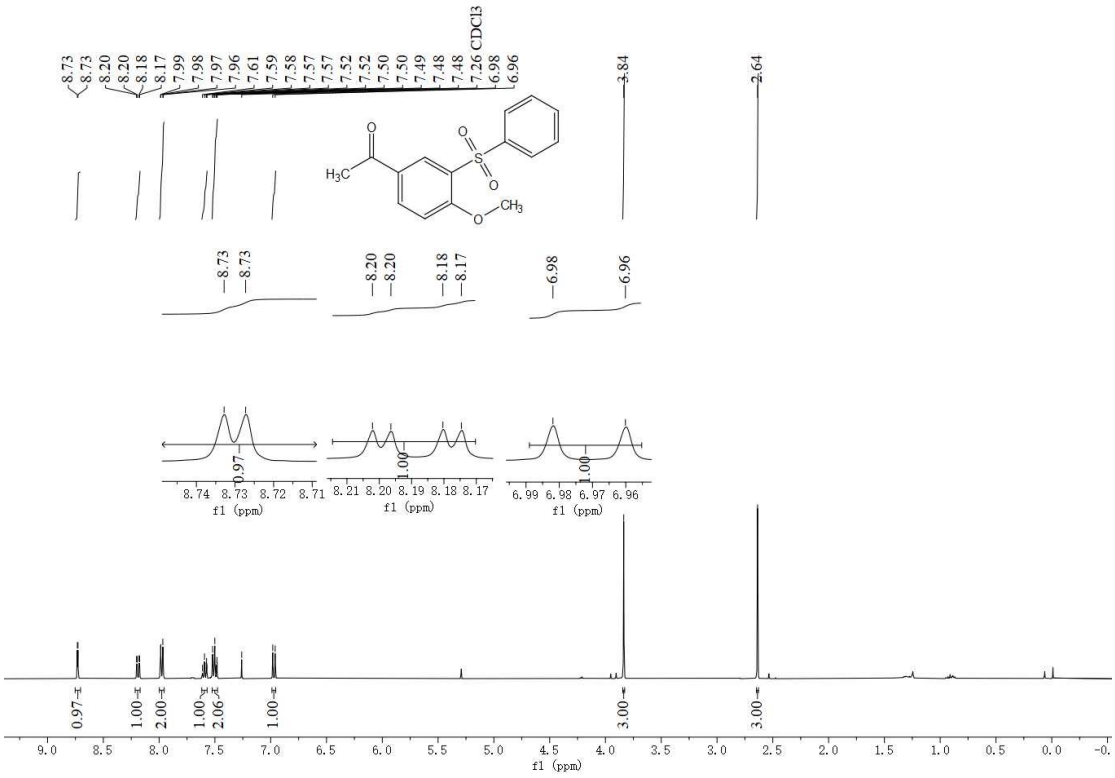
^{1}H NMR spectrum of **1-iodo-3-(phenylsulfonyl)benzene (32)**



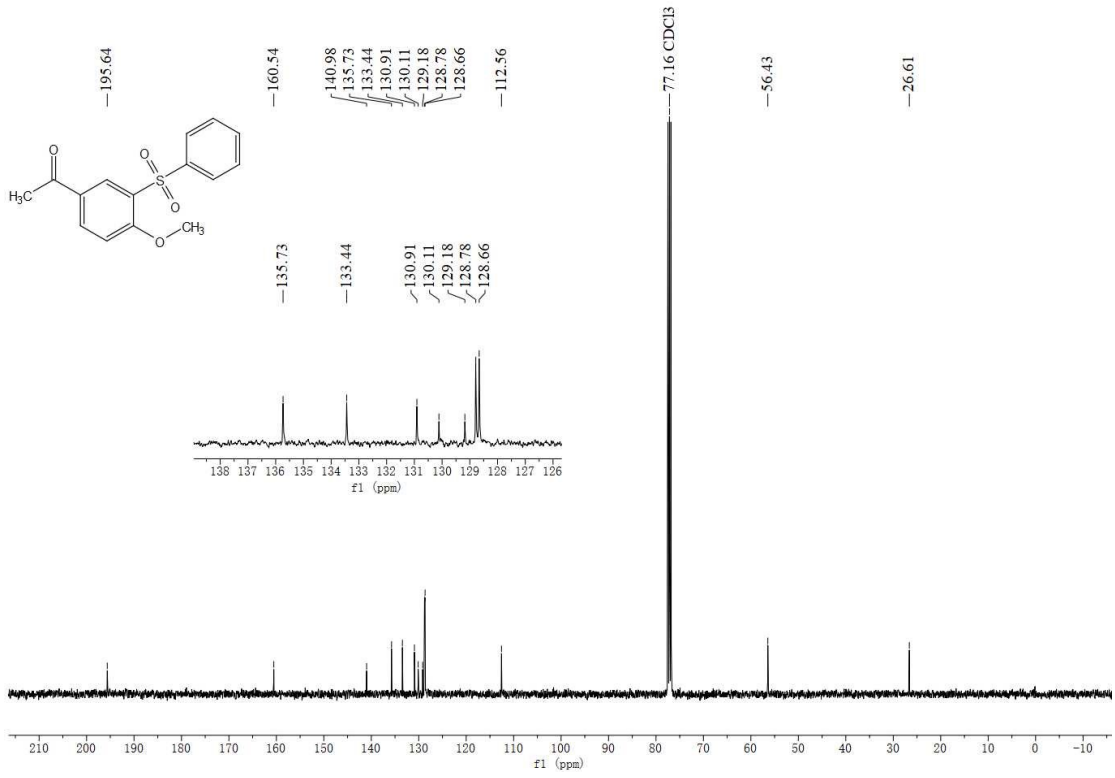
^{13}C NMR spectrum of **1-iodo-3-(phenylsulfonyl)benzene (32)**



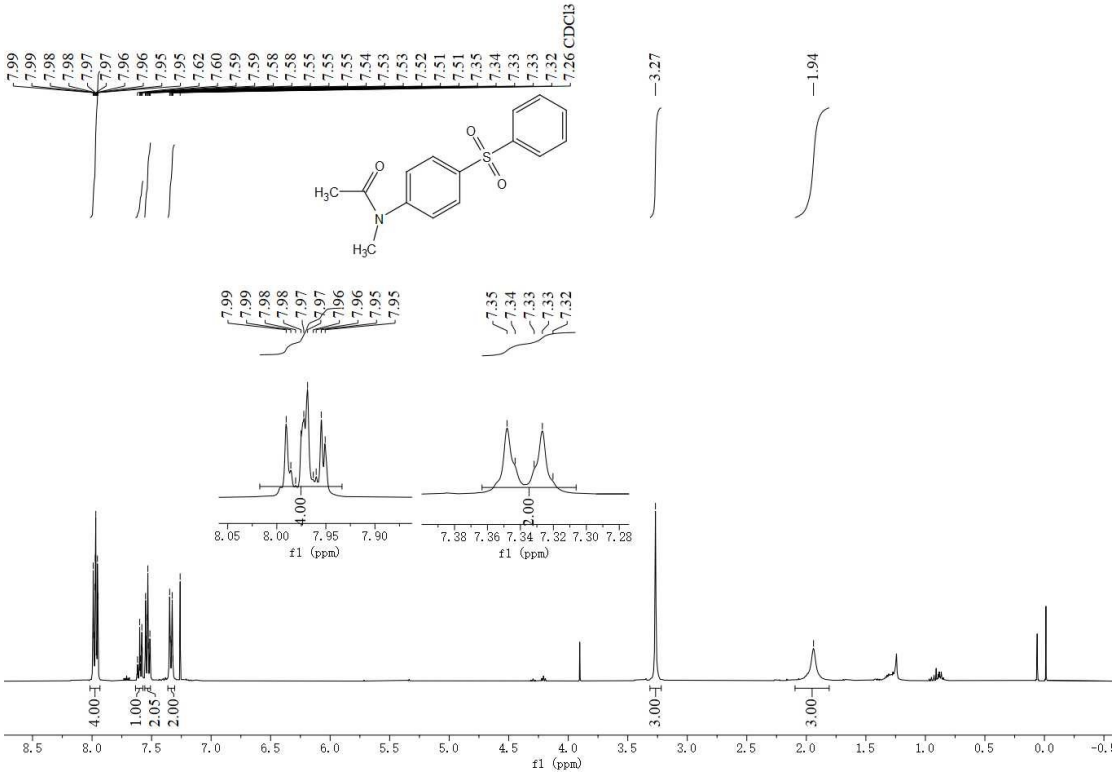
^{1}H NMR spectrum of **1-(4-methoxy-3-(phenylsulfonyl)phenyl)ethan-1-one (33)**



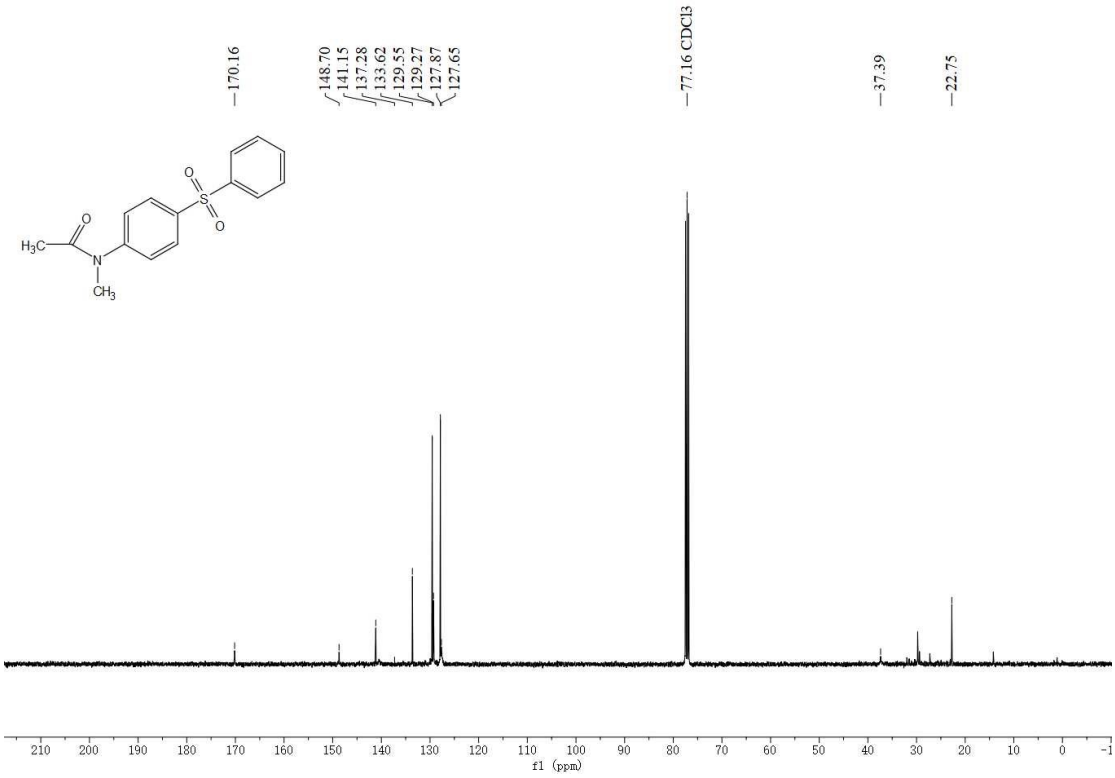
^{13}C NMR spectrum of **1-(4-methoxy-3-(phenylsulfonyl)phenyl)ethan-1-one (33)**



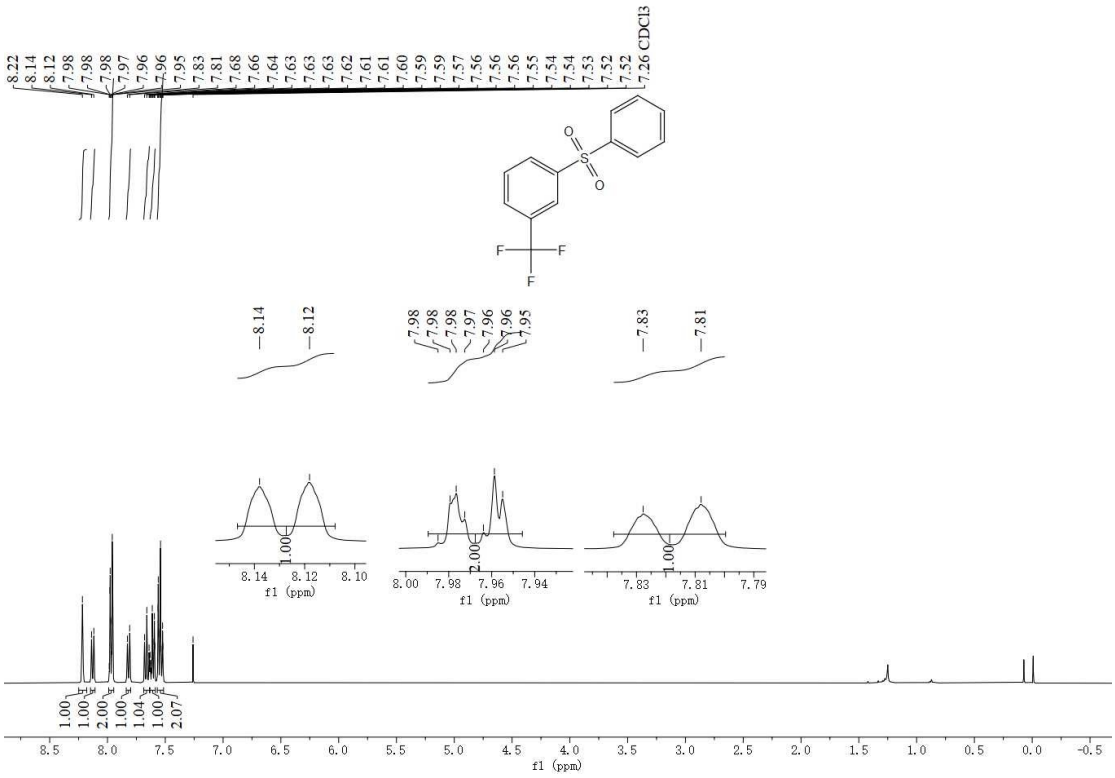
^{1}H NMR spectrum of **N-methyl-N-(4-(phenylsulfonyl)phenyl)acetamide (34)**



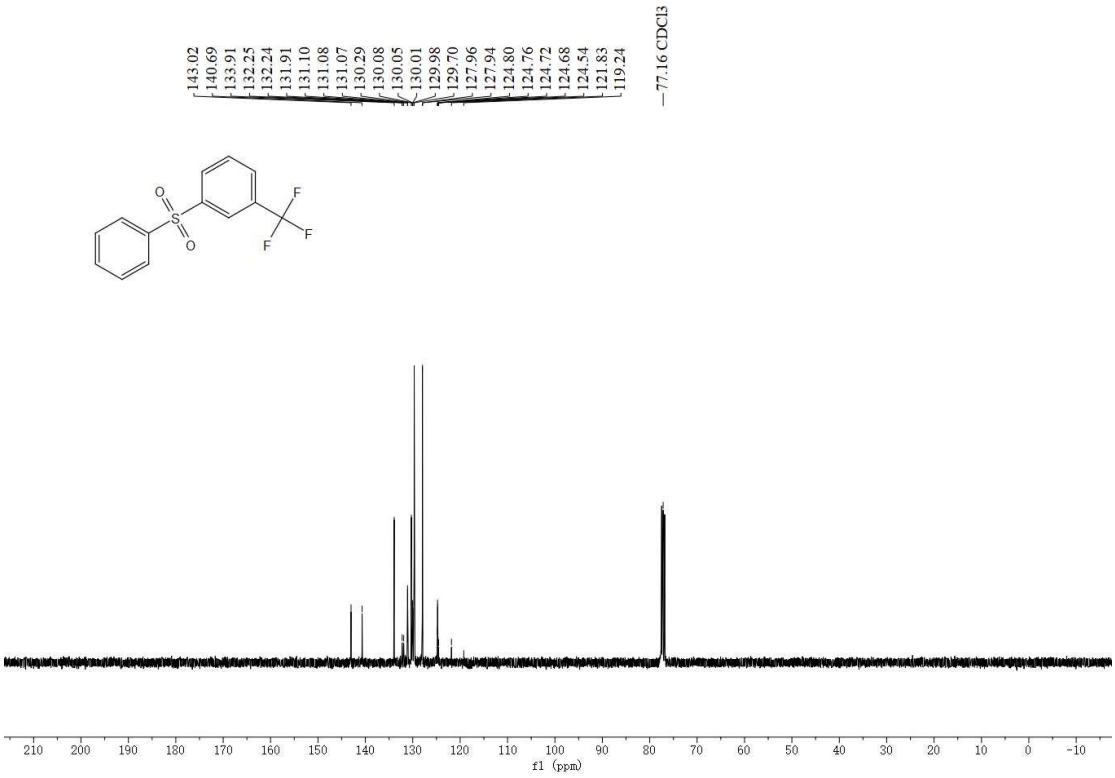
^{13}C NMR spectrum of **N-methyl-N-(4-(phenylsulfonyl)phenyl)acetamide (34)**



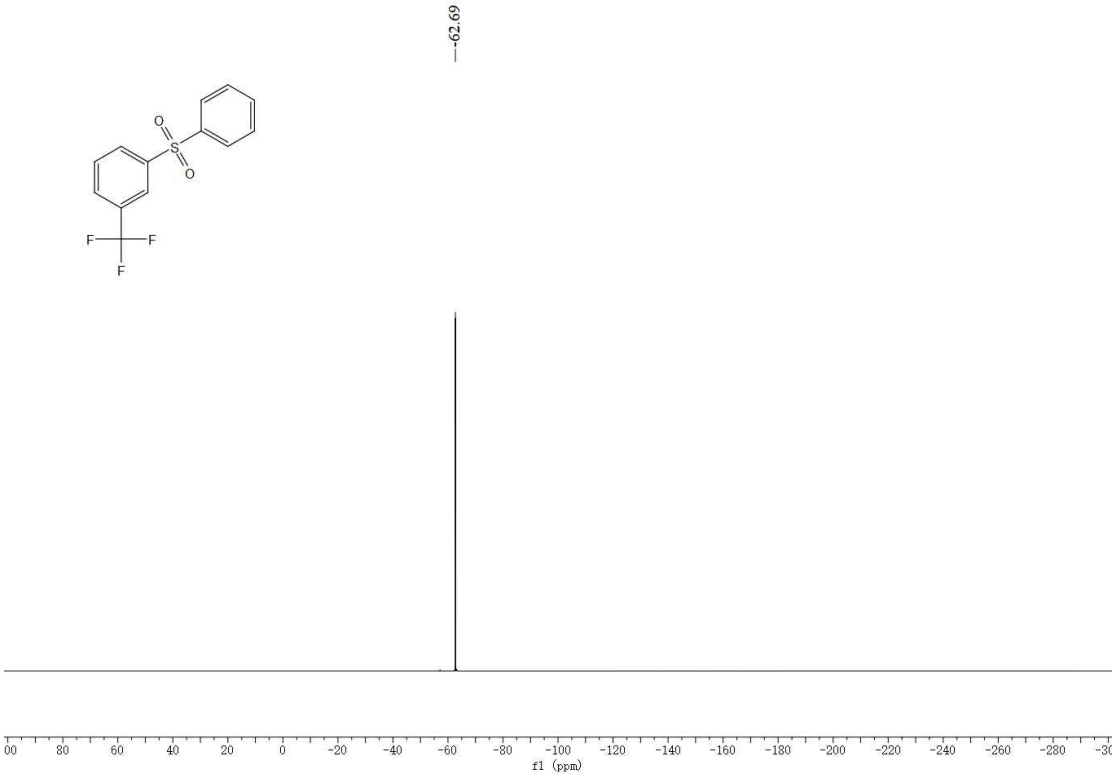
^{1}H NMR spectrum of **1-(phenylsulfonyl)-3-(trifluoromethyl)benzene (35)**



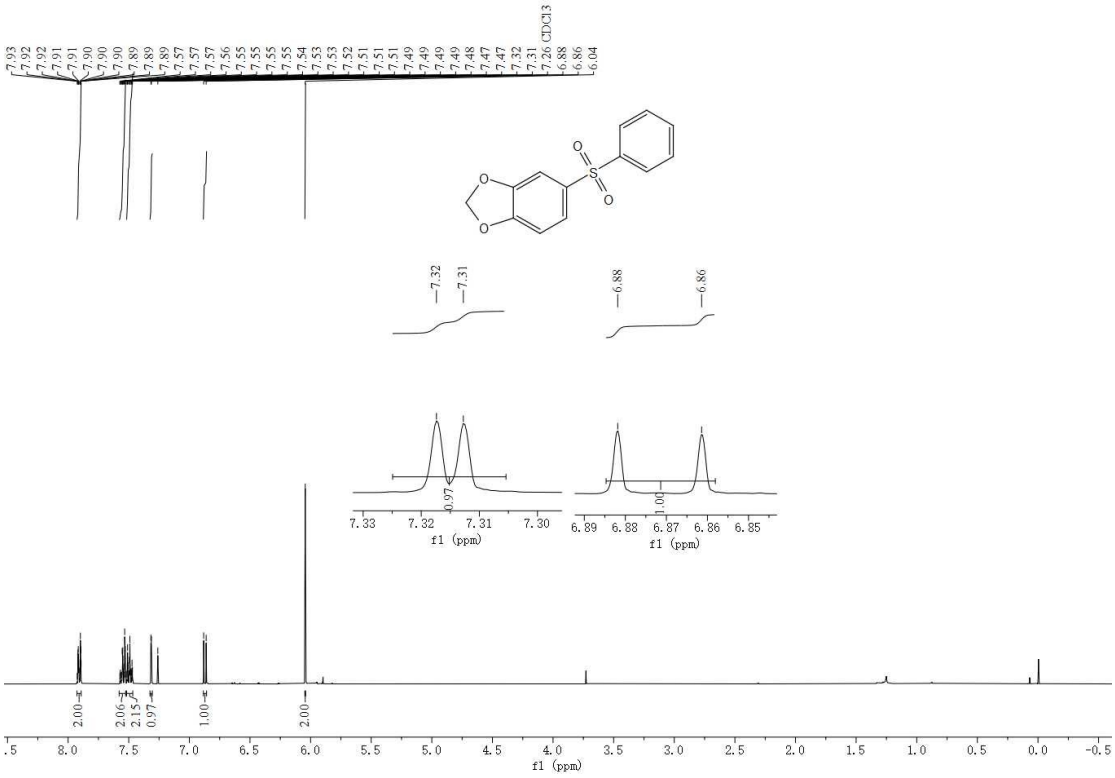
^{13}C NMR spectrum of **1-(phenylsulfonyl)-3-(trifluoromethyl)benzene (35)**



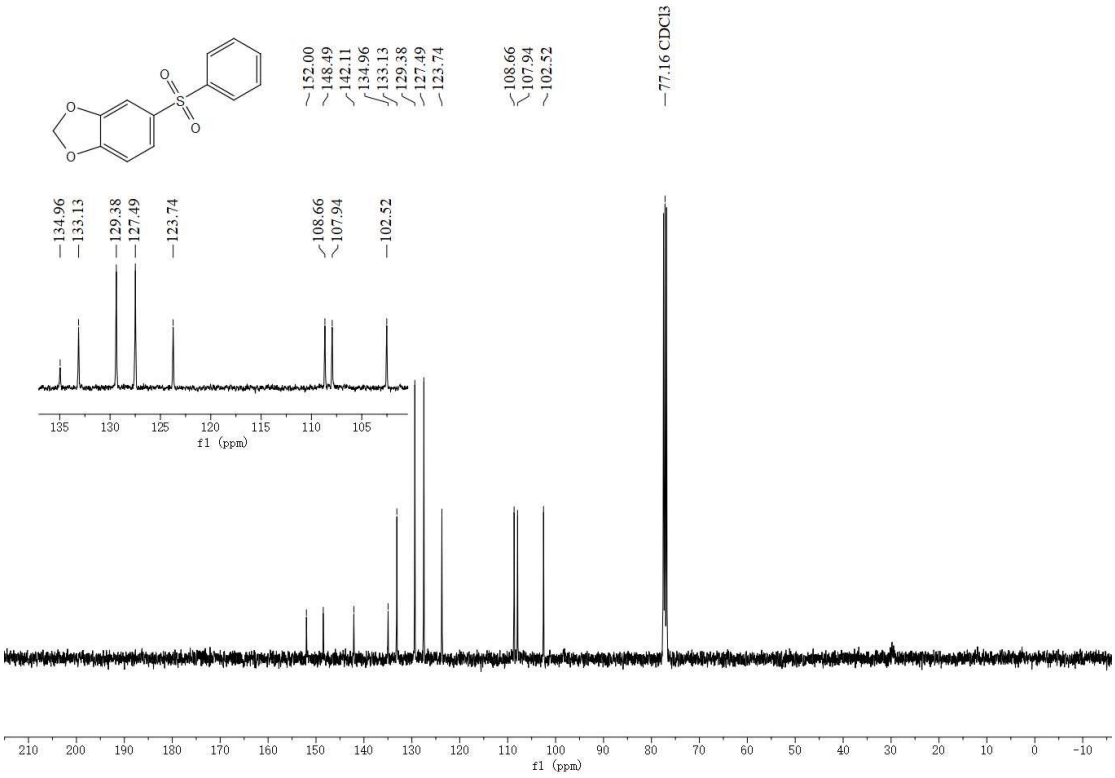
^{19}F NMR spectrum of **1-(phenylsulfonyl)-3-(trifluoromethyl)benzene (35)**



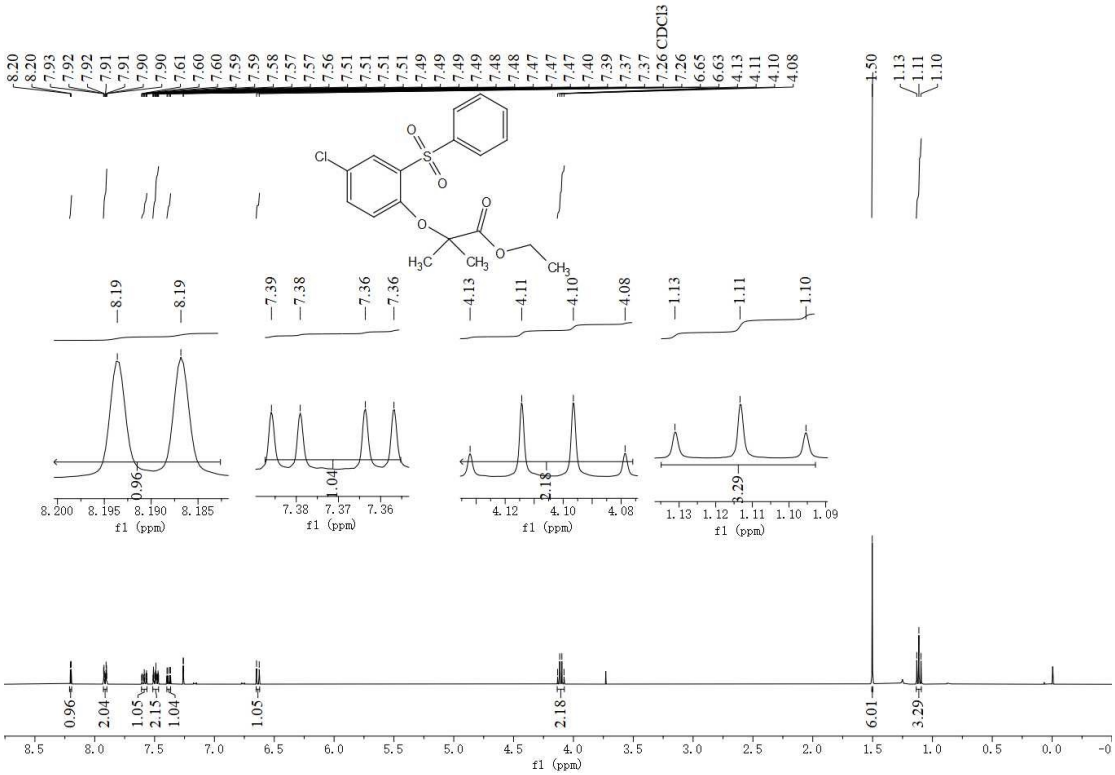
^{1}H NMR spectrum of **5-(phenylsulfonyl)benzo[d][1,3]dioxole (36)**



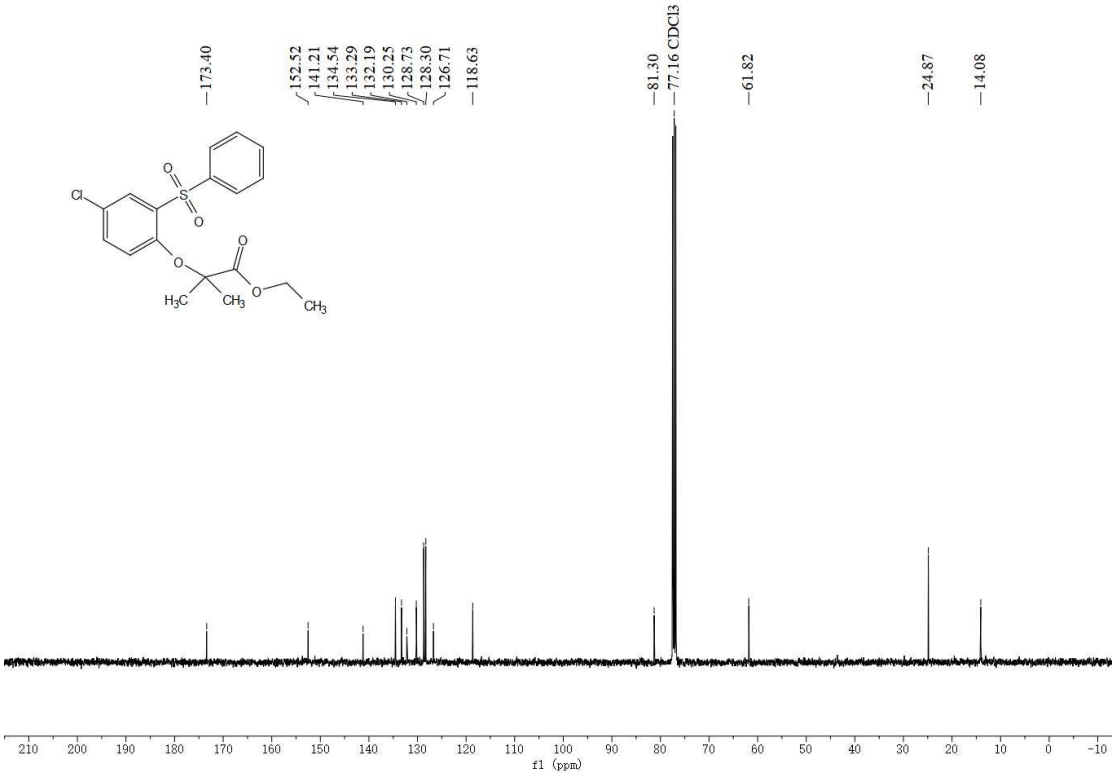
^{13}C NMR spectrum of **5-(phenylsulfonyl)benzo[d][1,3]dioxole (36)**



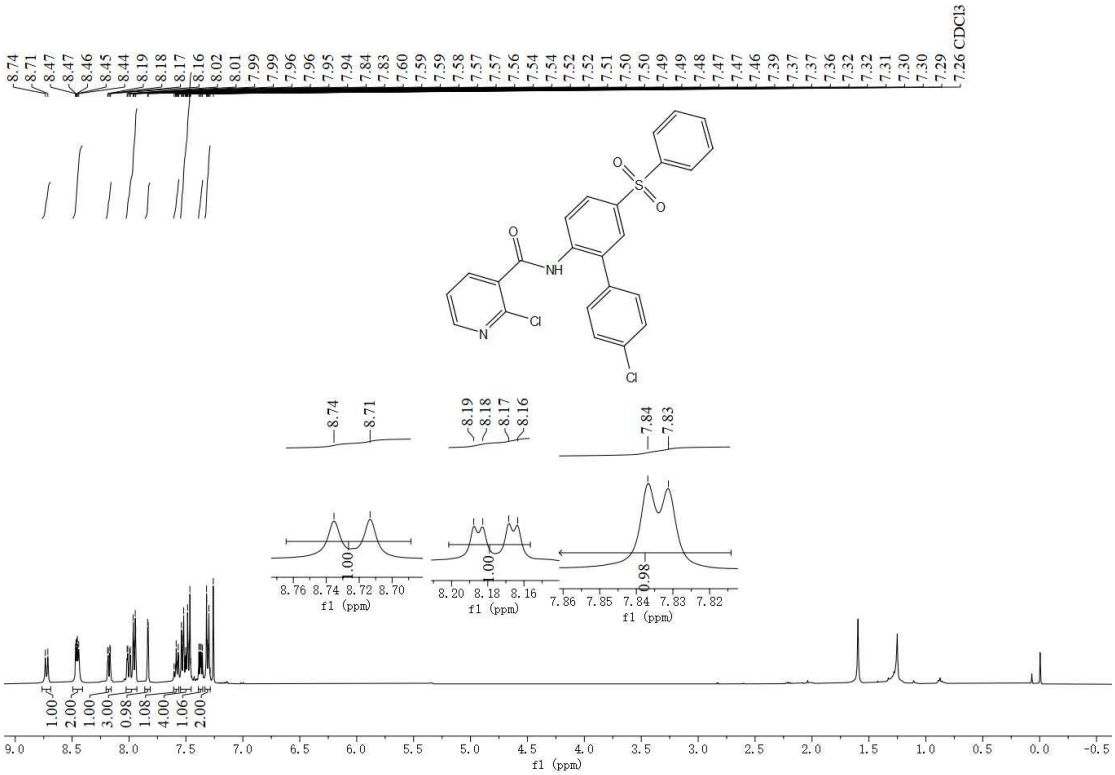
^{1}H NMR spectrum of **ethyl 2-(4-chloro-2-(phenylsulfonyl)phenoxy)-2-methylpropanoate (37)**



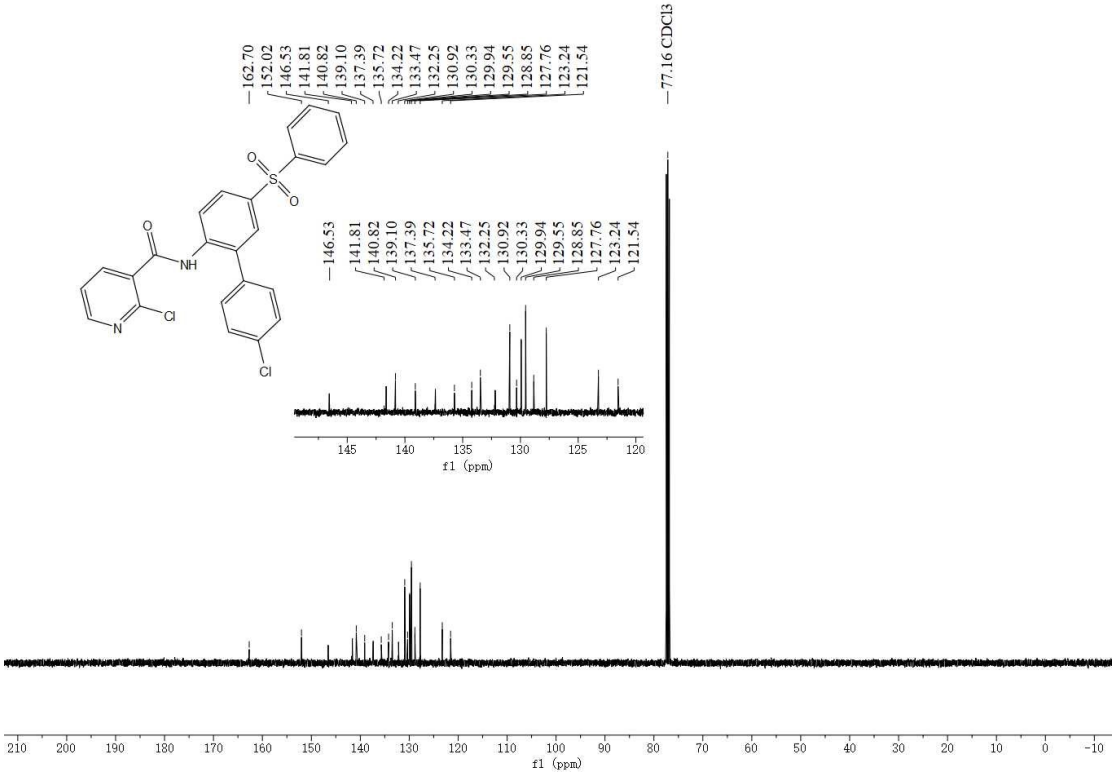
^{13}C NMR spectrum of **ethyl 2-(4-chloro-2-(phenylsulfonyl)phenoxy)-2-methylpropanoate (37)**



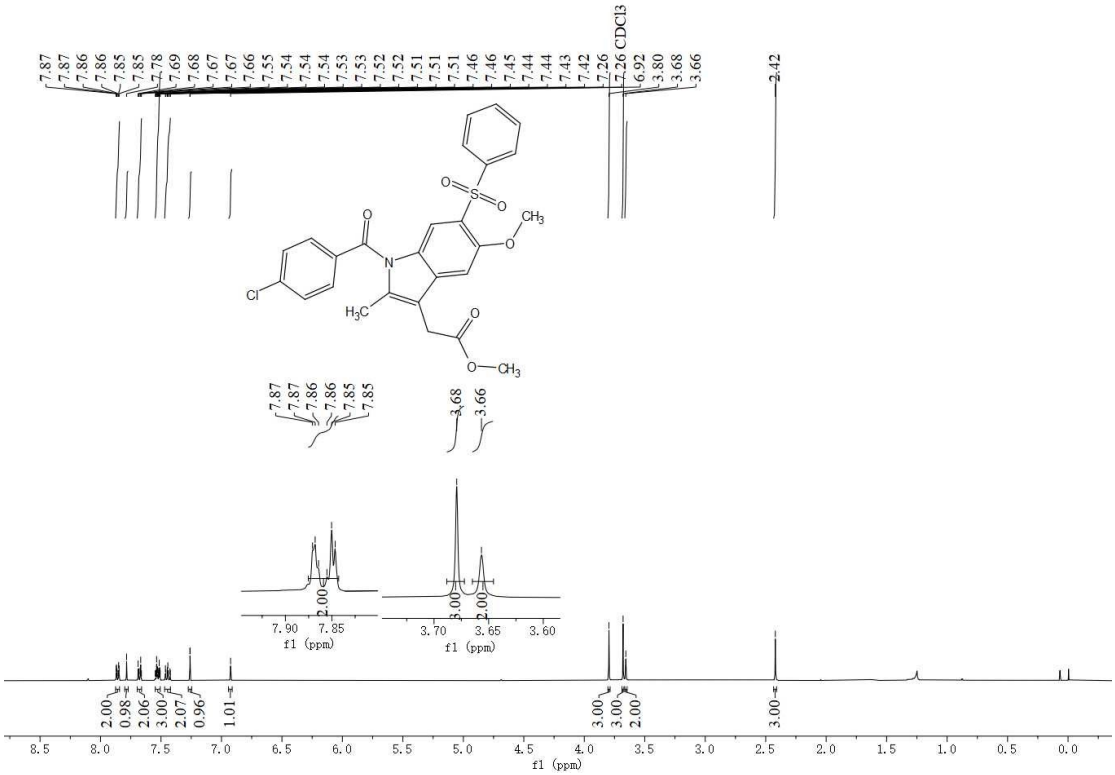
^{1}H NMR spectrum of **2-chloro-N-(4'-chloro-5-(phenylsulfonyl)-[1,1'-biphenyl]-2-yl)nicotinamide (38)**



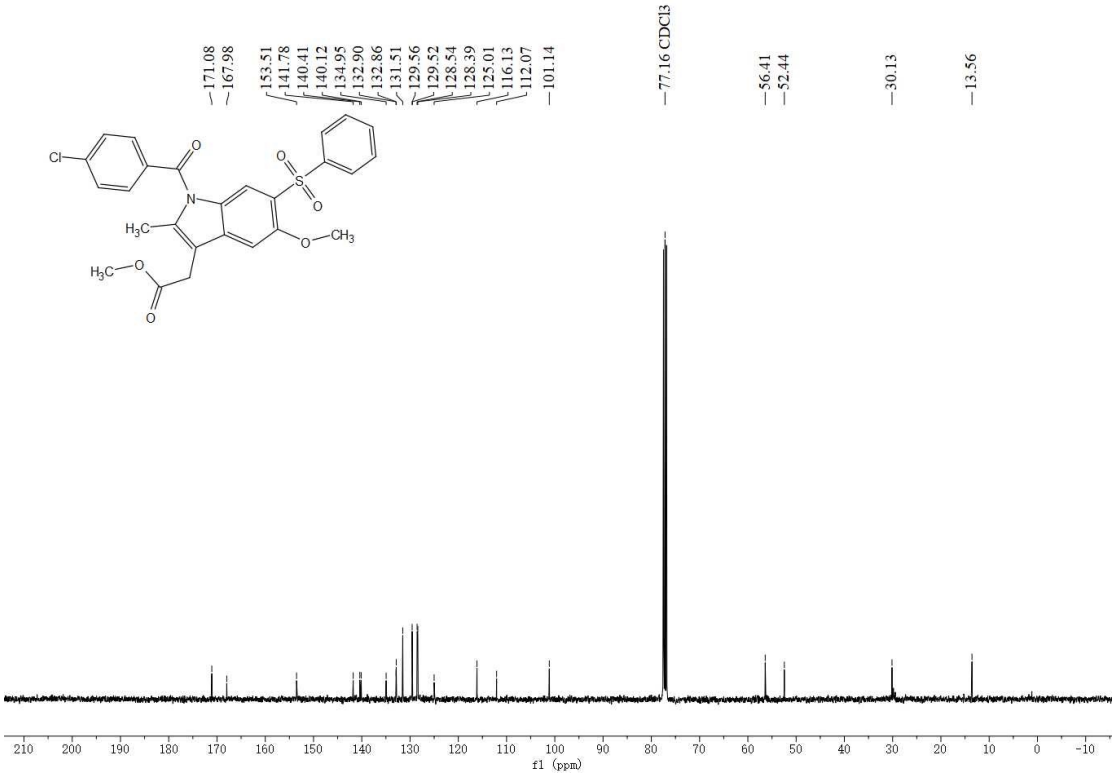
^{13}C NMR spectrum of **2-chloro-N-(4'-chloro-5-(phenylsulfonyl)-[1,1'-biphenyl]-2-yl)nicotinamide (38)**



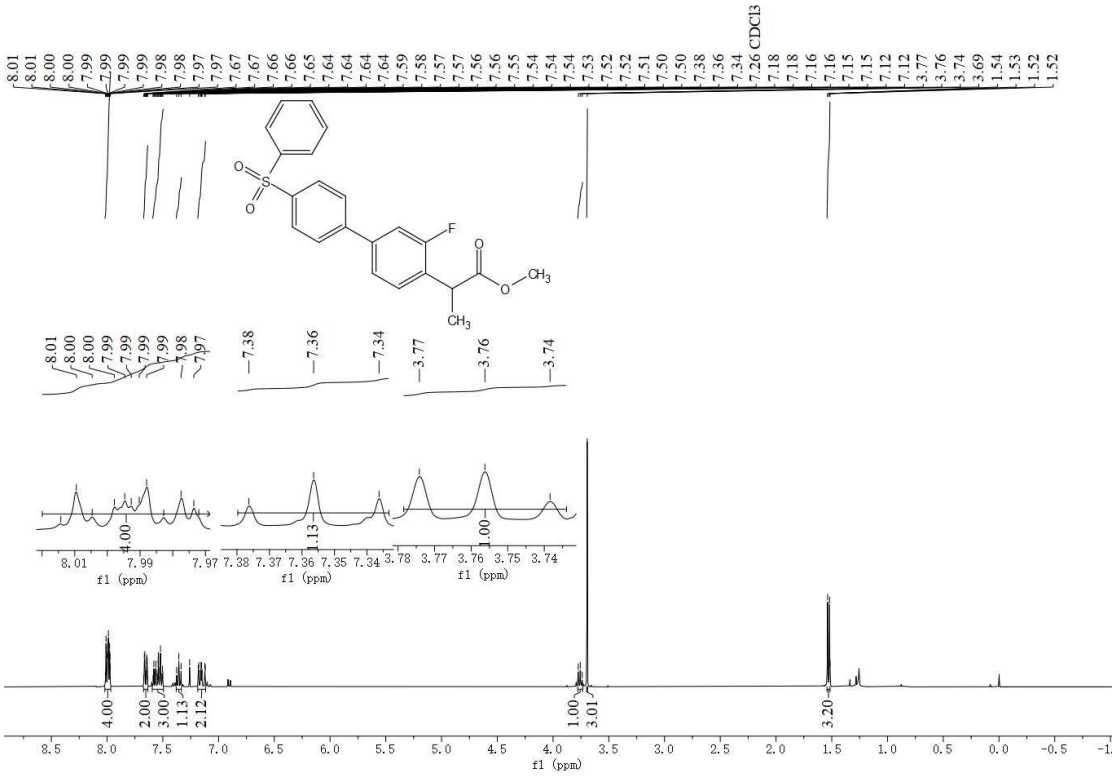
^{1}H NMR spectrum of **methyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-6-(phenylsulfonyl)-1H-indol-3-yl)acetate (39)**



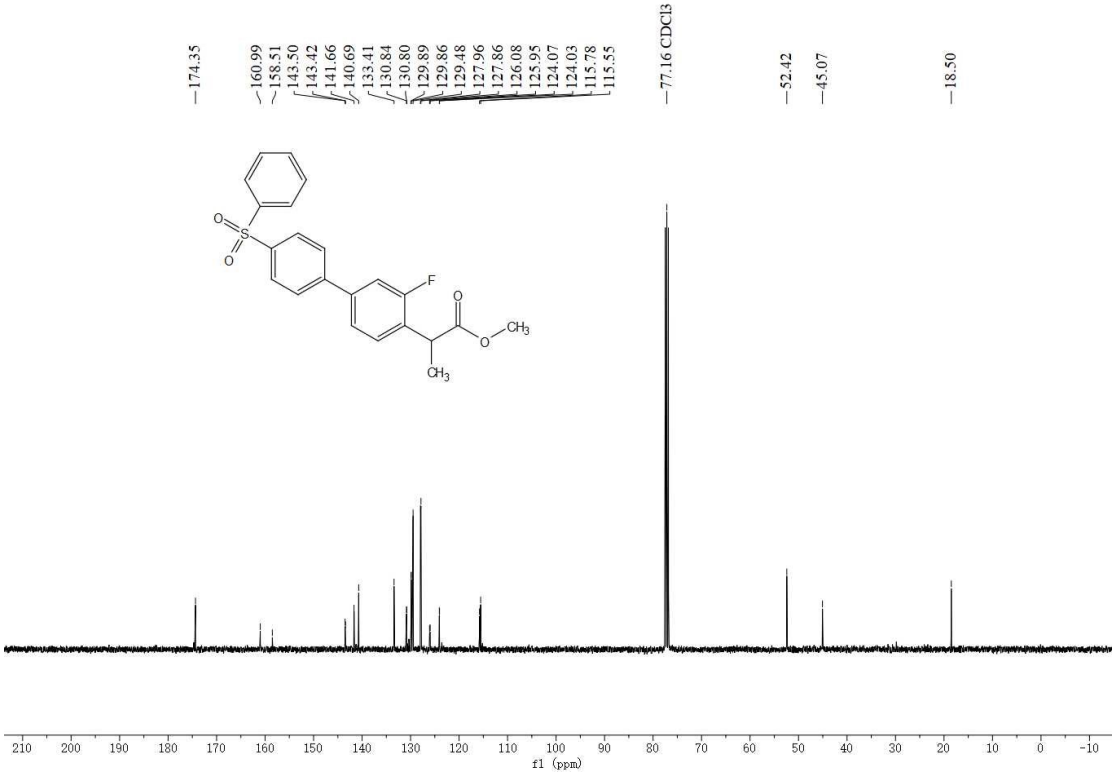
^{13}C NMR spectrum of **methyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-6-(phenylsulfonyl)-1H-indol-3-yl)acetate (39)**



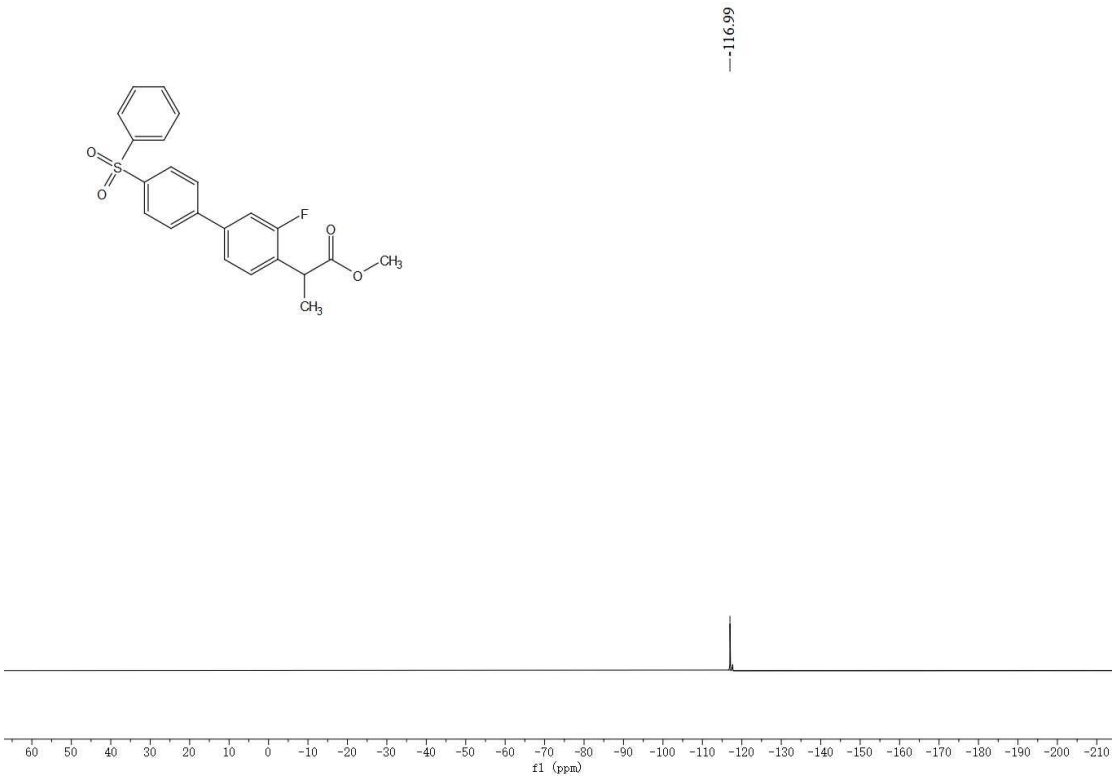
^{1}H NMR spectrum of **methyl 2-(2-fluoro-4'-(phenylsulfonyl)-[1,1'-biphenyl]-4-yl) propanoate (40)**



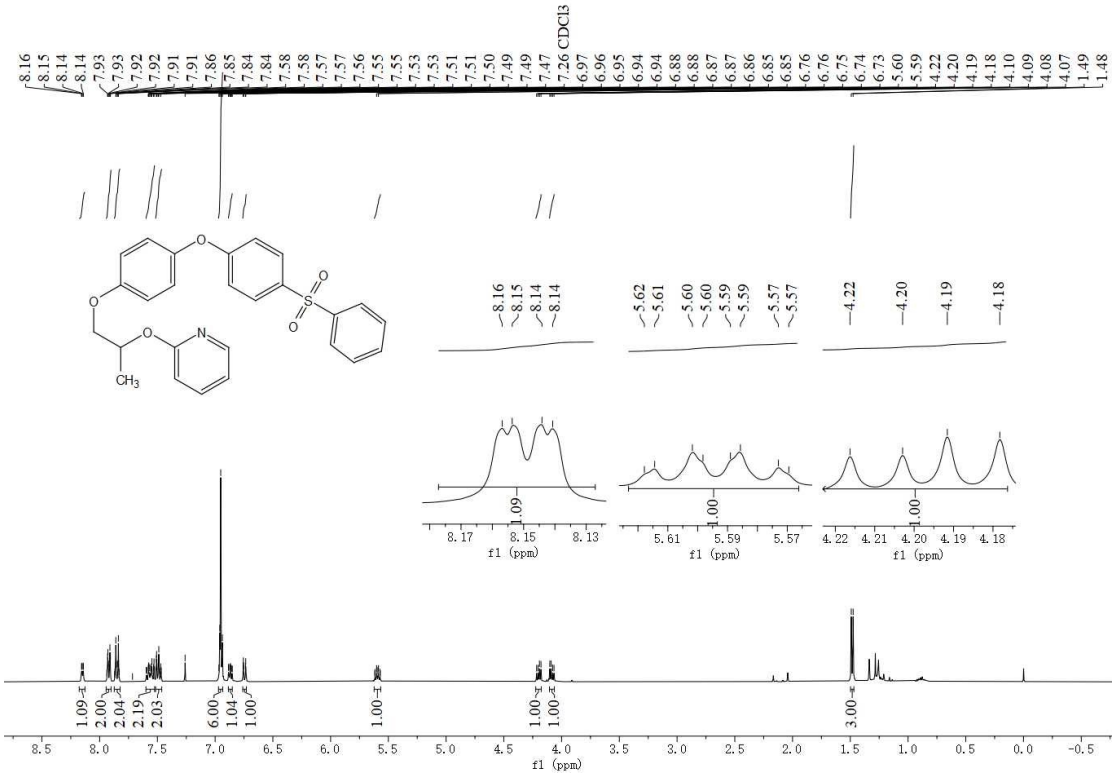
^{13}C NMR spectrum of **methyl 2-(2-fluoro-4'-(phenylsulfonyl)-[1,1'-biphenyl]-4-yl)propanoate (40)**



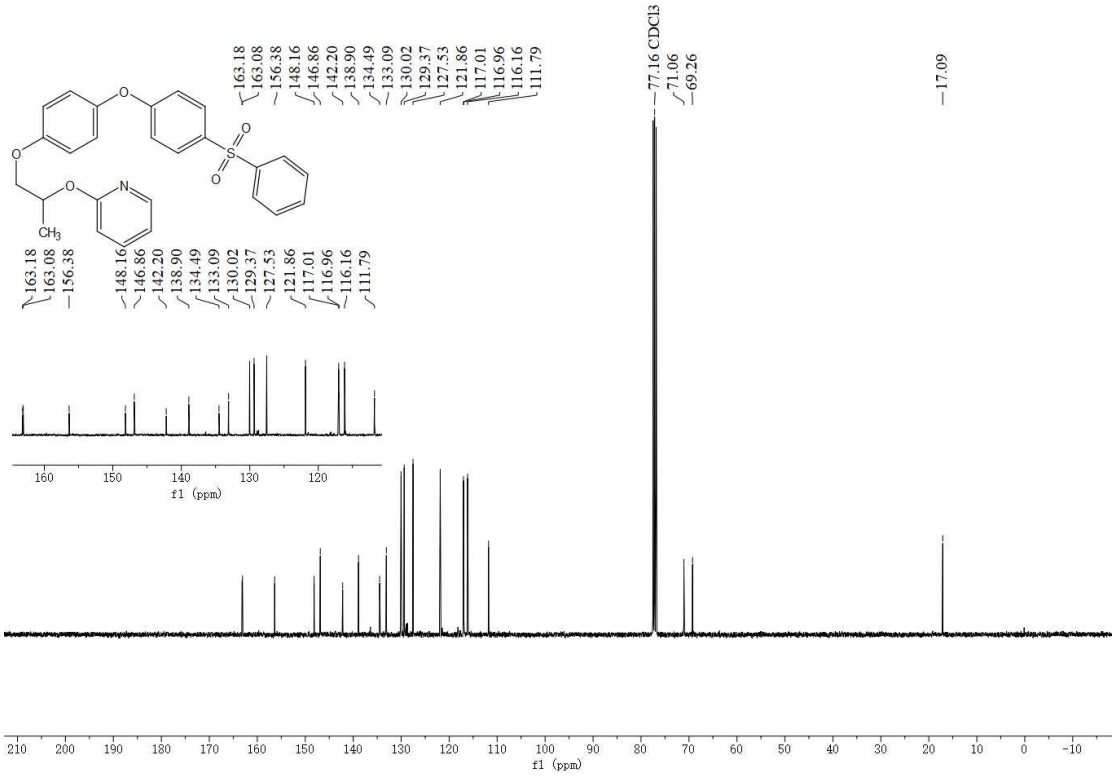
^{19}F NMR spectrum of **methyl 2-(2-fluoro-4'-(phenylsulfonyl)-[1,1'-biphenyl]-4-yl)propanoate (40)**



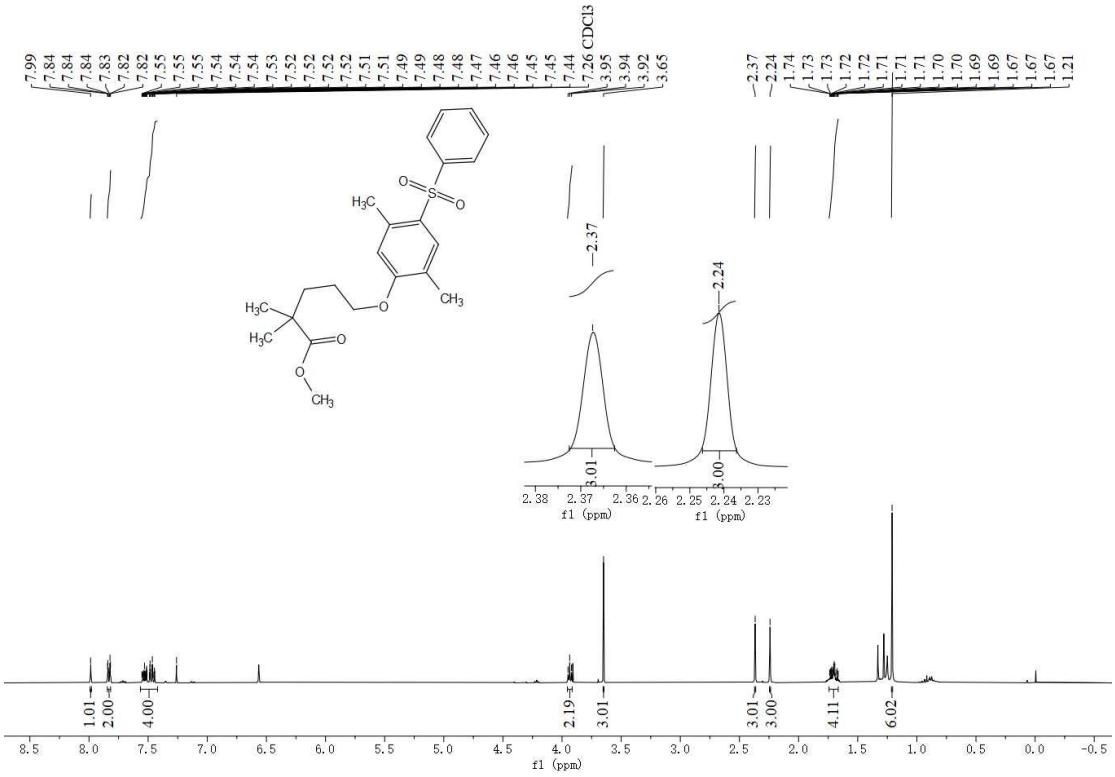
^{1}H NMR spectrum of **2-((1-(4-(4-(phenylsulfonyl)phenoxy)phenoxy)propan-2-yl)oxy)pyridine (41)**



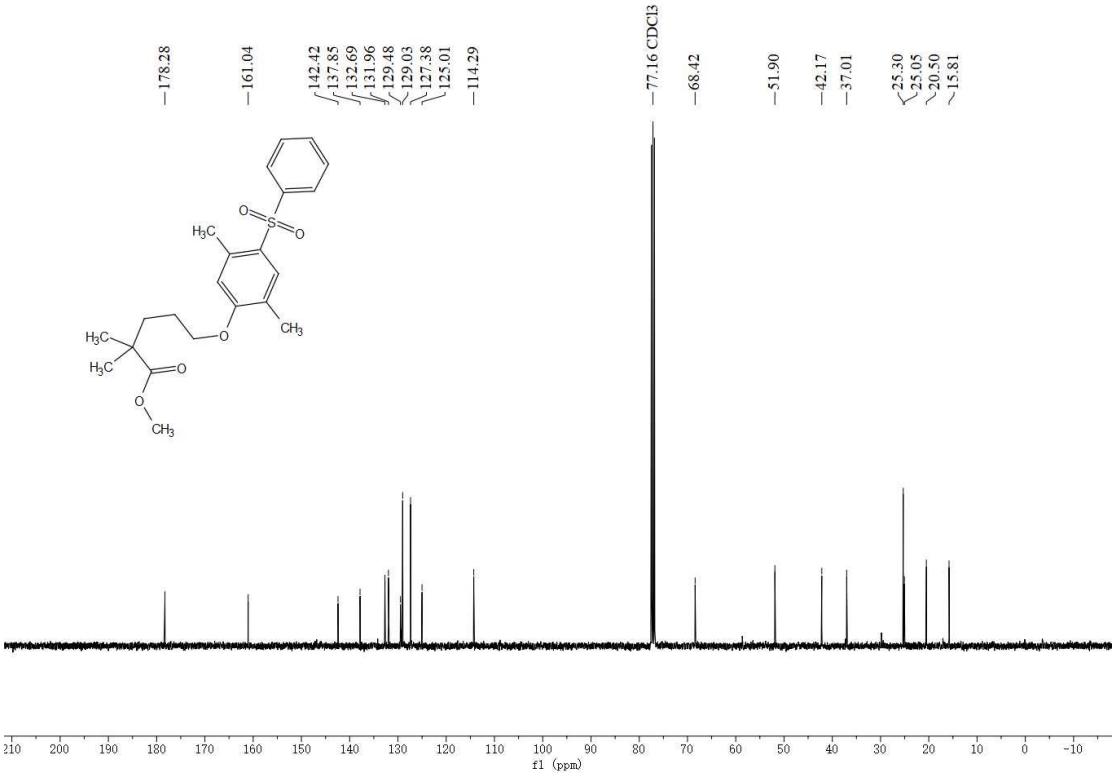
^{13}C NMR spectrum of **2-((1-(4-(4-(phenylsulfonyl)phenoxy)phenoxy)propan-2-yl)oxy)pyridine (41)**



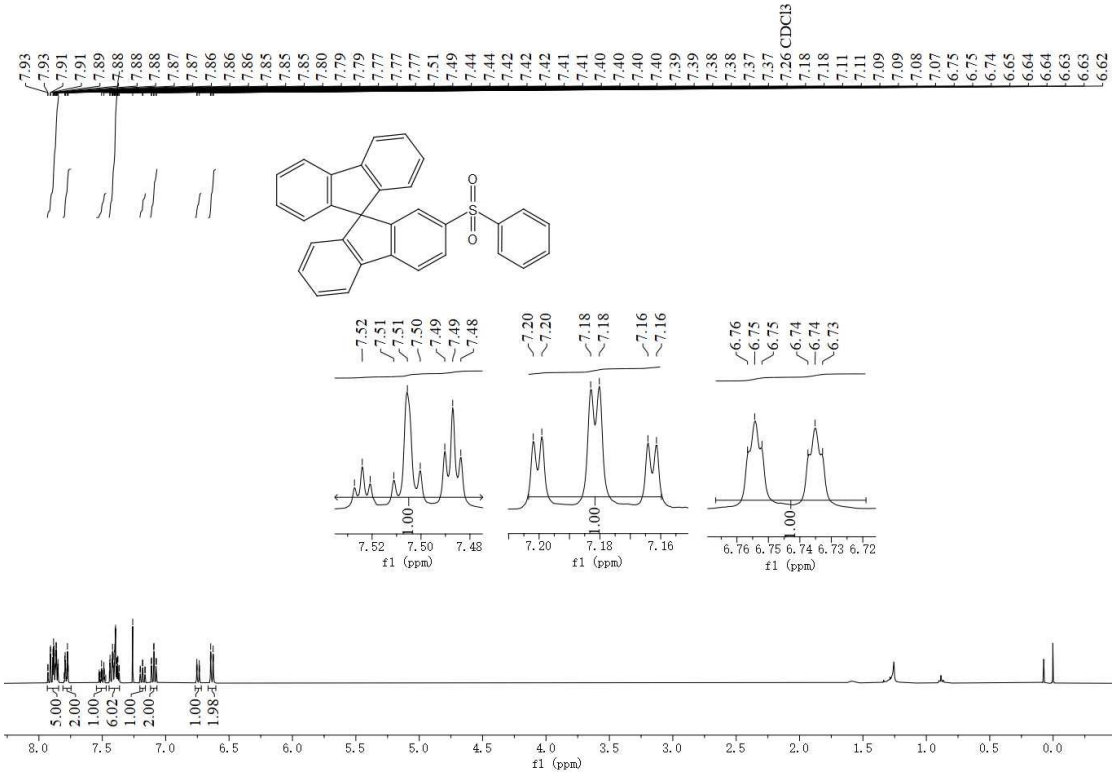
^{1}H NMR spectrum of **methyl 5-(2,5-dimethyl-4-(phenylsulfonyl)phenoxy)-2,2-di methylpentanoate (42)**



^{13}C NMR spectrum of **methyl 5-(2,5-dimethyl-4-(phenylsulfonyl)phenoxy)-2,2-dimethylpentanoate (42)**



^{1}H NMR spectrum of **2-(phenylsulfonyl)-9,9'-spirobi[fluorene] (43)**



^{13}C NMR spectrum of **2-(phenylsulfonyl)-9,9'-spirobi[fluorene] (43)**