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Supporting Information

Recyclable porphyrin photocatalyst for highly efficient visible-light-driven aerobic oxidation of sulfides

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1. General information

UV-Vis absorption spectra were measured using a Shimadzu UV-2700 spectrophotometer across 200−800 nm. Target compounds were performed on a Shimadzu GC-2010 plus equipped with a capillary column (SH-Rtx-5 30 m × 0.25 μm) and a flame ionization detector, or a Shimadzu HPLC-16 equipped with a C18 column. Electron spin resonance (EPR) spectra were acquired on a JES-FA200 EPR spectrometer. Proton nuclear magnetic resonance (^{1}H-NMR) spectra were obtained on a Bruker Avance 400 or 500 spectrometer at 400 or 500 MHz. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) was obtained on Bruker Avance 400 or 500 spectrometer at 100 or 125 MHz. Chemical shifts are reported in parts per million (δ) referenced to tetramethylsilane (0.0 ppm), chloroform (7.26 ppm or 77.0 ppm) and methanol (3.31 ppm or 49.0 ppm), respectively. Data for ^{1}H-NMR and ^{13}C-NMR spectroscopy are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), integration. High-resolution mass spectra were recorded on an AB QSTAR Pulsar mass spectrometer. Photocatalytic reactions were carried out in a multi-channel photocatalytic reactor (PCX-50C Discover, Beijing Perfectlight Technology Co., Ltd.) equipped with 10 W white LED lamps (total light intensity ≈ 310 mW·cm^{-2}). The known compounds were characterized by ^{1}H NMR and ^{13}C NMR, and the NMR data were identical to those reported in the corresponding literatures. Silica gel (200–300 mesh) for column chromatography and silica GF_{254} for TLC were produced by Merch Chemicals Co. Ltd. (Shanghai). Petroleum ether with the boiling range of 60-90 °C is used for column chromatography. Starting materials and reagents used in reactions were obtained commercially from Acros, Aldrich, Fluka, Adamas-beta®, and were used without purification, unless otherwise indicated.

2. Experimental procedures

2.1 General procedures for the synthesis of products

General procedure A: A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide (2.0 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). The solution was purged with

oxygen via a steel needle for 5 min. The vial was then sealed, and an oxygen balloon was attached through the inlet port. The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.). The reaction was monitored by GC or HPLC until complete conversion of the starting material was achieved. After the reaction, the solvent was removed under reduced pressure and recovered for reuse. The resulting residue was dissolved in ethyl acetate (EtOAc, 10 mL) with sonication for 5 min. The solution was then transferred to a 50 mL centrifuge tube, diluted with petroleum ether (PE, 20 mL), and subjected to ultrasonication for an additional 10 min. The resulting mixture was centrifuged at 9800 rpm and 25 °C for 10 min. This centrifugation process was repeated twice, and the solid catalyst was collected by decantation. The organic layer was then concentrated under reduced pressure and dried under vacuum at 25 °C for 0.5 h to afford the product without further purification, while the solvent was recovered for reuse.

General procedure B: A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide (0.2 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and trifluoroacetic acid (5.0 mL). Otherwise, the procedure followed General procedure A.

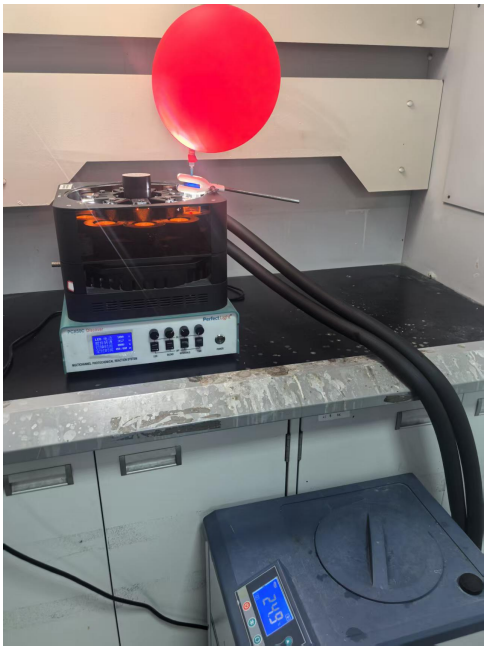
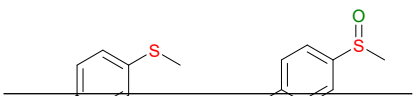


Fig. S1 Photograph of the photoreactor setup.

Table S1 Optimization of solvent system for oxidation-resistant sulfides.

Entry	Substrate	Product	Solvent	Additive	Time	Yield
1[a]			$EtOH/H_2O$	—	135 min	97%
2[b]			$EtOH/H_2O$	1.5 eq TFA	90 min	98%

Conversion and selectivity for sulfides and sulfoxides determined by GC were calculated as the following equations:

$$Conversion\ (\%) = \frac{reacted\ sulfide\ (mmol)}{total\ sulfide\ (mmol)} \times 100\%$$

$$Selectivity\ for\ sulfoxide\ (\%) = \frac{sulfoxide\ (mmol)}{sulfide\ (mmol) + sulfoxide\ (mmol) + sulfone\ (mmol)} \times 100\%$$

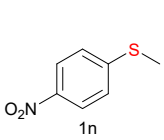
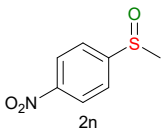
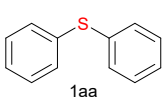
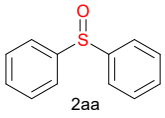
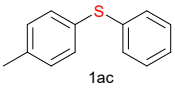
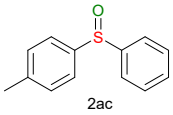
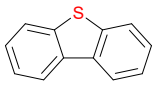
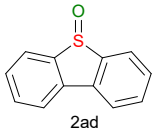
$$GC\ Yield\ (\%) = Conversion\ (\%) \times Selectivity\ (\%) \times 100\%$$

2.2 Optimization of reaction conditions

2.2.1 General Optimization Procedure

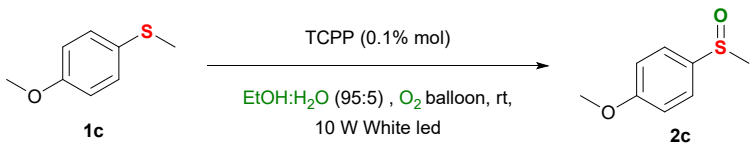
A 50 mL quartz vial equipped with a magnetic stir bar was charged with sulfide 1a (2.0 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and the solvent (5.0 mL). The solution was purged with oxygen via a steel needle for 5 min. The vial was then sealed, and an oxygen balloon was attached through the inlet port. The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.) for a predetermined time. After the reaction, an aliquot of the mixture was taken to determine the conversion and selectivity by GC-FID using benzophenone as an internal standard. The remaining solvent was then removed under reduced pressure and recovered for reuse. The residue was purified by flash chromatography using a gradient elution of petroleum ether/ethyl acetate (2:1, then 1:1) to afford pure sulfoxide 2a. The product was further dried under vacuum, and the isolated yield was calculated based on the weight.

2.2.2 Solvent Optimization for Oxidation-Resistant Sulfides

3[c]			TFA	—	20 min	96%
4[a]	 1n	 2n	EtOH/H_2O	—	210 min	79%
5[b]			EtOH/H_2O	1.5 eq TFA	210min	98%
6[c]			TFA	—	30 min	96%
7[a]	 1aa	 2aa	EtOH/H_2O	—	120 min	6%
8[b]			EtOH/H_2O	1.5 eq TFA	120 min	80%
9[c]			TFA	—	30 min	99%
10[a]	 1ac	 2ac	EtOH/H_2O	—	135 min	42%
11[b]			EtOH/H_2O	1.5 eq TFA	135 min	94%
12[c]			TFA	—	30 min	99%
13[a]	 1ad	 2ad	EtOH/H_2O	—	150 min	Trace
14[b]			EtOH/H_2O	1.5 eq TFA	150 min	Trace
15[c]			TFA	—	40 min	99%

Reaction conditions: [a]sulfide (0.2 mmol), TCPP (0.01% mol, 1.6mg), EtOH/H_2O (5 mL), O_2 atmosphere, white LED irradiation, room temperature. [b]sulfide (0.2 mmol), TCPP (0.01% mol, 1.6mg), EtOH/H_2O (5 mL), TFA (1.5 equiv, 22 ul), O_2 atmosphere, white LED irradiation, room temperature. [c]sulfide (0.2 mmol), TCPP (0.01% mol, 1.6 mg), TFA (5 mL), O_2 atmosphere, white LED irradiation, room temperature.

2.3 Gram scale synthesis of 2c



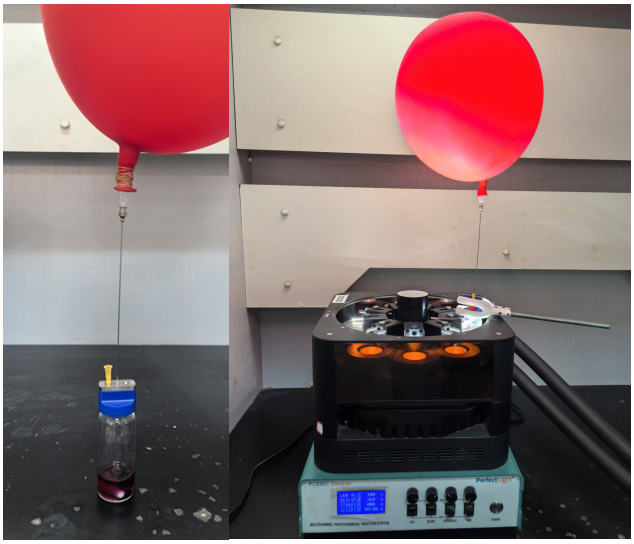
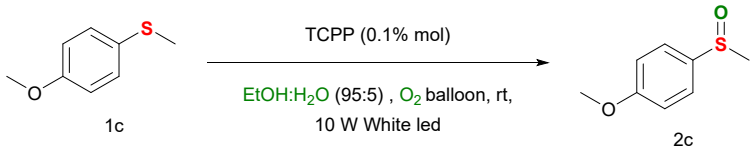


Fig. S2 Gram scale synthesis of **2c**.

A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide 1c (7.0 mmol, 1.08 g), tetrakis(4-carboxyphenyl)porphyrin (12.2 mg, 0.0140 mmol), and EtOH/H_2O (15.0 mL, 95:5, v/v). The solution was purged with oxygen via a steel needle for 5 min. The vial was then sealed, and an oxygen balloon was attached through the inlet port, with a hollow needle inserted through the lid to maintain connection with the atmosphere, ensuring continuous oxygen flow throughout the reaction. The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.) for 40 min. After the reaction, the solvent was removed under reduced pressure and recovered for reuse. The resulting residue was dissolved in ethyl acetate (EtOAc, 10 mL) with sonication for 5 min. The solution was then transferred to a 50 mL centrifuge tube, diluted with petroleum ether (PE, 20 mL), and subjected to ultrasonication for an additional 10 min. The resulting mixture was centrifuged at 9800 rpm and 25 °C for 10 min. This centrifugation process was repeated twice, and the solid catalyst was collected by decantation. The organic layer was then concentrated under reduced pressure to afford the product without further purification, and the solvent was recovered for reuse.

2.4 TCPP recycling experiment

2.4.1 Recycling procedure



Scheme S1 Recyclability test of TCPP for the synthesis of sulfoxide **2c**.

A 50 mL quartz vial equipped with a magnetic stir bar was charged with sulfide 1c (2.0 mmol, 308.4 mg), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). The solution was purged with oxygen via a steel needle for 5 min. The vial was then sealed, and an oxygen balloon was attached through the inlet port. The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.) for 15 min. After the reaction, the solvent was removed under reduced pressure and recovered for reuse. The resulting residue was dissolved in ethyl acetate (EtOAc, 10 mL) with sonication for 5 min. The solution was then transferred to a 50 mL centrifuge tube, diluted with petroleum ether (PE, 20 mL), and subjected to ultrasonication for an additional 10 min. The resulting mixture was centrifuged at 9800 rpm and 25 °C for 10 min. This centrifugation process was repeated twice, and the solid catalyst was collected by decantation, dried under vacuum at 25 °C for 1 h, and then directly used for the next run. The organic layer was collected, and the yield and selectivity of the product were determined by GC-FID using benzophenone as an internal standard.

2.4.2 NMR and HRMS characterization of the recovered TCPP

TCPP after reaction (recovered after eight cycles) ^{1}H NMR (600 MHz, DMSO) δ 13.30 (s, 4H), 8.82 (s, 8H), 8.35 (d, J = 8.0 Hz, 8H), 8.29 (d, J = 8.0 Hz, 8H), -2.97 (s, 2H). ^{13}C NMR (150 MHz, DMSO) δ 167.5, 145.5, 134.5, 130.5, 128.0, 119.4. HRMS (ESI) m/z calcd. for $C_{48}H_{31}N_4O_8$ [M+H]+: 791.2136, found: 791.2143.

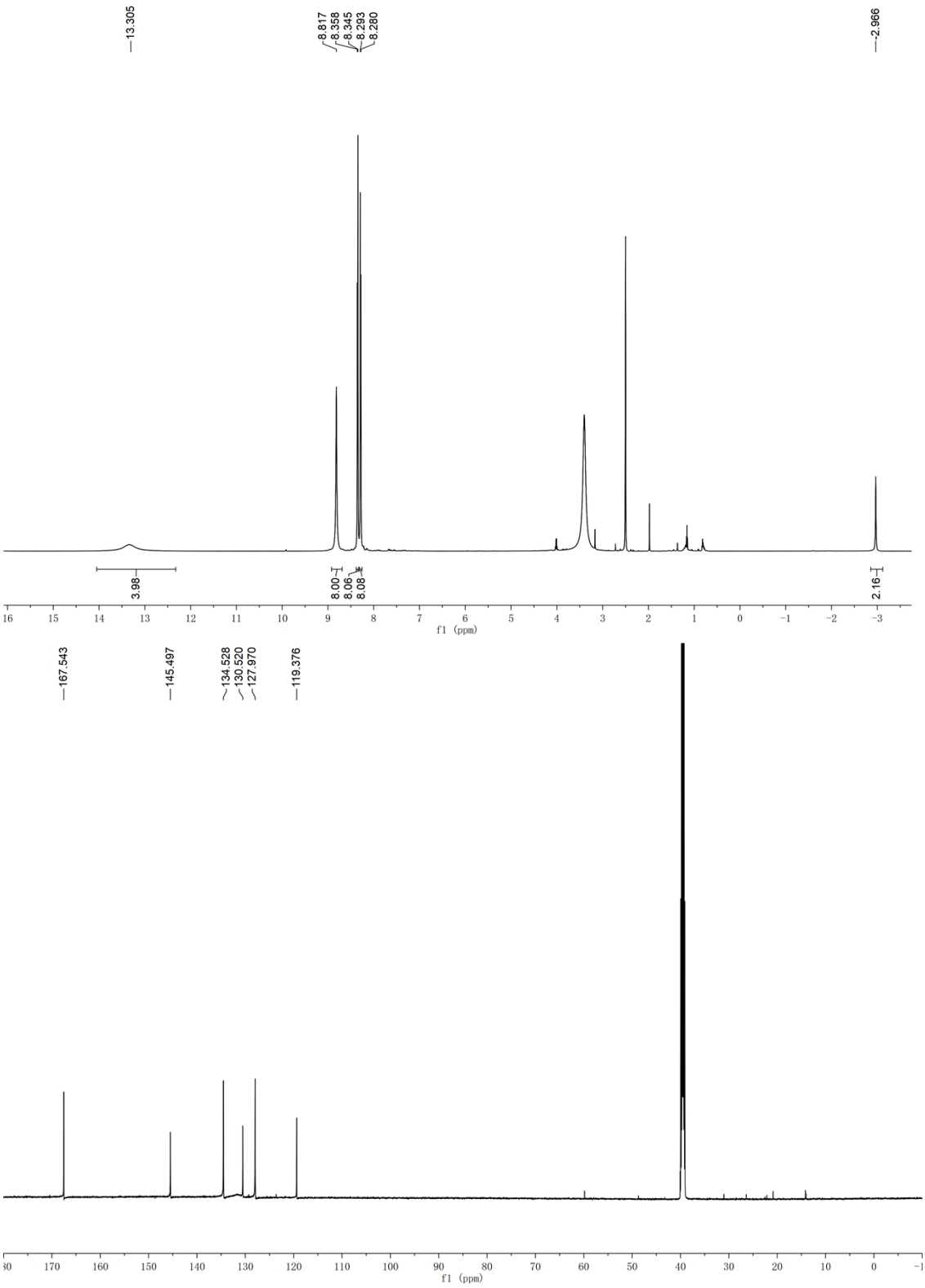


Fig. S3 ^{1}H NMR and ^{13}C NMR spectrum (DMSO-d_6) of TCPP recovered after eight cycles.

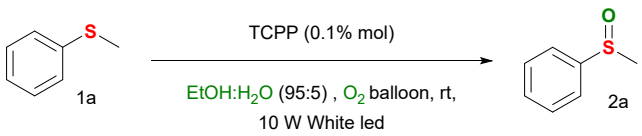
Fig. S4 HRMS spectrum of TCPP recovered after eight cycles (showing $[M+H]^+$ = 799.20, calcd 799.22).

2.5 UV-vis absorption measurements

Fresh TCPP or the recovered TCPP was dissolved to prepare a solution with a concentration of 0.096 μg mL^{-1}. The resulting solution was then transferred into a cuvette for UV–vis absorption measurement over the wavelength range of 200–800 nm.

3. Mechanistic studies

3.1 Light on-off experiment



Scheme S2 Light on-off experiment for synthesis 2a catalyzed by TCPP.

A 50 mL quartz vial equipped with a magnetic stir bar was charged with thioanisole (2.0 mmol, 248.4 mg), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). The solution was purged with oxygen via a steel needle for 5 min. The vial was then sealed, and an oxygen balloon was attached through the inlet port. The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.) using alternating intervals of light and darkness. To investigate the necessity of continuous irradiation, the yield of product 2a as a function of time.After the reaction, the solvent was removed under reduced pressure and recovered for reuse. After the reaction, the solvent was removed under reduced pressure and recovered for reuse. The residue was purified by flash chromatography using a gradient elution of petroleum ether/ethyl acetate (2:1, then 1:1) to afford pure sulfoxide 2a. The product was further dried under vacuum, and the yield was calculated based on the isolated weight.

3.2 Scavenging experiments

A 50 mL quartz vial equipped with a magnetic stir bar was charged with

thioanisole (2.0 mmol, 248.4 mg), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). The solution was purged with oxygen via a steel needle for 5 min. To investigate the role of reactive species, various additives were introduced separately: chloroform (1.0 equiv), 1,4-diazabicyclo[2.2.2]-octane (DABCO, 0.2 equiv), sodium azide (NaN_3, 0.3 equiv), silver nitrate ($AgNO_3$, 0.1 equiv), ethylenediaminetetraacetic acid (EDTA, 0.1 equiv), and isopropanol (0.1 equiv). Following the addition, The mixture was irradiated with a white LED light setup (PCX-50C, Beijing Perfectlight Technology Co., Ltd.). After the reaction, the solvent was removed under reduced pressure and recovered for reuse. The residue was purified by flash chromatography using a gradient elution of petroleum ether/ethyl acetate (2:1, then 1:1) to afford pure sulfoxide 2a. The product was further dried under vacuum, and the yield was calculated based on the isolated weight.

Table S2 Scavenging experiments for the photocatalytic oxidation of thioanisole by TCPP.

Entry	Scavengert	Target Species	Amount (equiv)	Yield of 2a (%)[a]
1	$CHCl_3$	$O_2^{\bullet-}$	1.0	97
2	DABCO	$^1O_2$	0.2	68
3	NaN_3	$^1O_2$	0.3	trace
4	$AgNO_3$	e^-	0.1	90
5	EDTA	h^+	0.1	94
6	*iso*-pronaol	•OH	0.1	93
7	None	-	-	97

[a]Yields are isolated yields. All reactions was performed with 1a (2 mmol) and the catalyst (0.1 mol%) in solvent (5 mL) under an O_2 atmosphere (balloon) and irradiation with a 10 W white LED

3.3 EPR spectroscopy experiments

Detection of reactive oxygen species with TCPP catalyst: A 50 mL quartz vial equipped with a magnetic stir bar was charged with TCPP (1.6 mg, 0.0020 mmol) and

EtOH/H_2O (5.0 mL, 95:5, v/v). The solution was purged with oxygen via a steel needle for 5 min in the dark. Subsequently, thioanisole (1a) (2.0 mmol, 248.4 mg) was added. For the detection of $^1O_2$, 2,2,6,6-tetramethylpiperidine (TEMP, 34 mg, 0.2 mmol) was added to the mixed solution. For the detection of superoxide radical anion ($O_2^{\bullet -}$), DMPO (4.5 mg, 0.04 mmol) was used in place of TEMP. After irradiation of the mixed solution with a 60 W incandescent lamp under O_2 for 5 min, the EPR spectrum was recorded (microwave frequency: 9.450059 GHz; power: 10.0 mW; center field: 336.00 mT; sweep width: 3.93 mT; modulation frequency: 125000 Hz; modulation amplitude: 100 μT.).

4. Comparison with reported porphyrin-based photocatalytic systems

Table S3 Comparison of reported porphyrin-based photocatalytic sulfide oxidation systems with this work.

Entry	Catalysts	System type	Substrate (mmol)	Solvent (mL)	Time (h/min)	Yield (%)	Substrate scope[a]	Light source/Oxidant	Ref.
1	**TCPP (commercial available, 0.1-1.0 mol%, 1.6 mg)**	**Homogeneous**	**2 mmol (21examples); 0.2mmol (8 examples)**	**EtOH/H_2O (5 mL, 95/5 v/v)**	**15-40 min**	**86-99%**	**29 examples: (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, strong/weak EWG); (3) diaryl sulfides**	**10W LED white light; O_2**	**This work**
2	TPP (**commercial availability,** 0.0001 mol%)	Homogeneous	0.68 or 1 mmol	CH_3COOH or CCl_4/CH_3COOH (2.4 mL)	16 or 32 h	58-98%,	**7 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (weak EDG, weak EWG)	Blue LED (λ_{max}=445 nm, 3W); Air	18
3	Porphyrin diacids H_4TPPY_2 (Y=CF_3COO^-, Cl_2CHCOO^-, ClO_4^-, HSO_4^-) (0.025 mol%)	Homogeneous	2.64 mmol	CH_3CN (10 mL)	1-18 h	45-96%,	**7 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (weak EDG, weak EWG) No reaction (diaryl sulfide, 10 h)	Sunlight irradiation O_2	17

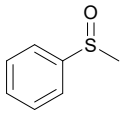
4	Phosphonate-substituted pyrazinoporphyrin (0.001 mol%)	Homogeneous	1 mmol	CH_2Cl_2/MeOH (2.4 mL, 2:1 v/v)	16 h	44%-100%	**17 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, weak EWG)	Blue LED (430-505 nm, 3W); O_2	31
5	In(III) pyrazinoporphyrinate (10^{-3}-5×10^{-4} mol%)	Homogeneous	0.5 mmol	CH_3COOH (2.4 mL)	8 or 16 h	53-100% (8 h); 97-100% (16 h)	**7 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (weak EDG, weak EWG)	Blue LED (430-505 nm, 3 W), Air	32
6	$PdF_{20}TPP$ (0.05 mol%),	Homogeneous	0.256 mmol	CH_3CN (1.6 mL)	2-6 h	87-94%	**8 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, weak EWG)	Light (λ>400nm); O_2 bubbling	34
7	Fe-EMindTPP (2 mg)	Heterogeneous	0.3 mmol	MeOH (1 mL)	1.5 -16 h	21.7-100%	**17 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, strong/weak EWG); (3) diaryl sulfides	LED (420 nm, 25 W); O_2	55

8	Porphyrin−polyoxotungstate (POM) hybrids (0.003 mol %))	Heterogeneous	0.1 mmol	CH_3CN/H_2O (2 mL, 95/5, v/v).	1 h	96%	**1 examples:** alkyl aryl sulfides	Visible light ($\lambda > 400$ nm), 25 °C, O_2 (1 atm)	35
9	PyPor-COF (1.0 mg)	Heterogeneous	0.2 mmol	H_2O (2.0 mL),	24 h	73-99%	**14 examples:** alkyl aryl sulfides (strong/weak EDG, strong/weak EWG)	Red LEDs (λ_{max}=660 nm); Air	58
10	ETBA-por COF. (10 mg)	Heterogeneous	0.5 mmol	CH_3CN (6.0 mL) and MeOH (2.5 mL),	3 h	90-96%	**5 examples:** alkyl aryl sulfides (weak EDG, weak EWG)	Xe lamp (150 W, λ = 400–780 nm); O_2	59
11	PdPor-sp^2c-COF (5 mg)	Heterogeneous	0.3 mmol	MeOH (1 mL)	48 min	47-98%	**13 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, weak EWG)	Green LED irradiation (520±10nm, 3 W×4); Air	60
12	Porphyrin−based COF (Sn-CPF-3, 0.3 mol% based on Sn)	Heterogeneous	0.1 mmol	MeOH (1 mL)	12 h	70-99%	**6 examples:** alkyl aryl sulfides (strong/weak EDG, weak EWG)	Blue LED, 3 W ; O_2	61

13	2D PMOF (Ti) (5 mg)	Heterogeneous	0.3 mmol	MeOH (1 mL)	24-180 min	77-99%	**16 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, strong/weak EWG)	Red LEDs (623 nm, 3 W × 4); air	56
14	PCBA-functionalized MOF (0.1 mol%)	Heterogeneous	1 mmol	MeOH (5 mL)	6 h	3-99%	**8 examples:** (1) dialkyl sulfides; (2) alkyl aryl sulfides (strong/weak EDG, strong/weak EWG) (3) diaryl sulfides	Xenon lamp, 150 W; Air	57
15	TPPS -BV (10 mg)	Heterogeneous	0.1 mmol	MeOH (2 mL)	1 h	80-99%	**4 examples:** (1) alkyl aryl sulfides (strong/weak EDG, weak EWG); No reaction (strong EDG (-NO_2) alkyl aryl sulfides)	Two LED lamp (420–750 nm), 17 W; O_2	40

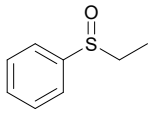
[a] EDG = electron-donating group; EWG = electron-withdrawing group.

5. Analytical data of compounds



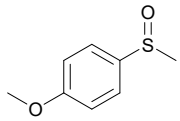
2a

2a: Following General procedure A, the desired product **2a** was obtained in 97% yield (272.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.59 (m, 2H), 7.50 – 7.43 (m, 3H), 2.68 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.6 , 131.1, 129.4, 123.5, 43.9.The spectra matched with the previous report.[1]



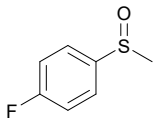
2b

2b: Following General procedure A, the desired product **2b** was obtained in 99% yield (306.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 – 7.55 (m, 2H), 7.50 – 7.43 (m, 3H), 2.93 – 2.82 (m, 1H), 2.80 – 2.70 (m, 1H), 1.17 (t, J = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.2, 131.0, 129.2, 124.25, 50.3, 6.1. The spectra matched with the previous report.[1]



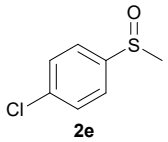
2c

2c: Following General procedure A, the desired product **2c** was obtained in 97% yield (33.9 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 – 7.54 (m, 2H), 7.00 – 6.97 (m, 2H), 3.81 (s, 3H), 2.66 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.0, 136.4, 125.5, 114.9, 55.6, 44.0. The spectra matched with the previous report.[1]



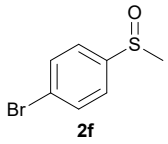
2d

2d: Following General procedure A, the desired product **2d** was obtained in 86% yield (272.4 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 – 7.61 (m, 2H), 7.23 – 7.18 (m, 2H), 2.70 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.6, 163.1, 141.1, 141.1, 126.0, 125.9, 116.9, 116.7, 44.2. The spectra matched with the previous report.[2]



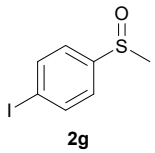
2e

2e: Following General procedure A, the desired product **2e** was obtained in 98% yield (337.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59 – 7.56 (m, 2H), 7.50 – 7.47 (m, 2H), 2.70 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.1, 137.3, 129.7, 125.1, 44.1. The spectra matched with the previous report.[3]



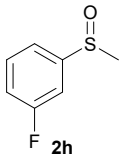
2f

2f: Following General procedure A, the desired product **2f** was obtained in 91% yield (272.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 – 7.63 (m, 2H), 7.51 – 7.48 (m, 2H), 2.69 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.9, 132.6, 125.5, 125.2, 44.0. The spectra matched with the previous report.[1]



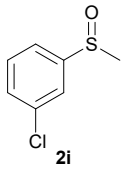
2g

2g: Following General procedure A, the desired product **2g** was obtained in 92% yield (491.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.84 (m, 2H), 7.38 – 7.35 (m, 2H), 2.70 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.7, 138.5, 125.2, 97.5, 44.1. The spectra matched with the previous report.[1]



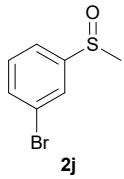
2h

2h: Following General procedure A, the desired product **2h** was obtained in 93% yield (293.0 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 – 7.44 (m, 1H), 7.38 – 7.33 (m, 2H), 7.16 – 7.12 (m, 1H), 2.69 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 164.1, 162.1, 148.4, 148.4, 131.1, 131.1, 119.2, 119.2, 118.2, 118.1, 110.9, 110.7, 44.0. The spectra matched with the previous report.[3]



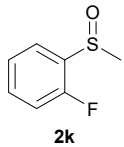
2i

2i: Following General procedure A, the desired product **2i** was obtained in 99% yield (344.7 mg).**^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.63 – 7.62 (m, 1H), 7.47 – 7.42 (m, 3H), 2.70 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.9, 135.7, 131.2, 130.6, 123.7, 121.7, 44.0. The spectra matched with the previous report.[2]



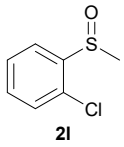
2j

2j: Following General procedure A, the desired product **2j** was obtained in 90% yield (396.2 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (t, J = 1.6 Hz, 1H), 7.59 – 7.57 (m, 1H), 7.51 – 7.49 (m, 1H), 7.36 (t, J = 7.8 Hz, 1H), 2.70 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.8, 134.1, 130.9, 126.5, 123.6, 122.1, 44.0. The spectra matched with the previous report.[3]



2k

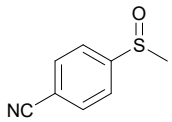
2k: Following General procedure A, the desired product **2k** was obtained in 95% yield (301.9 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 – 7.79 (m, 1H), 7.46 – 7.42 (m, 1H), 7.34 (t, J = 7.5 Hz, 1H), 7.06 (t, J = 9.0 Hz, 1H), 2.77 (s, 3H).**^{13}C NMR** (125 MHz, $CDCl_3$) δ 158.4, 156.5, 132.8, 132.7, 132.7, 132.7, 125.4, 125.4, 125.3, 125.3, 115.8, 115.6, 42.1, 42.1. The spectra matched with the previous report.[4]



2l

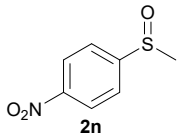
2l: Following General procedure A, the desired product **2l** was obtained in 99% yield (346.5 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.91 (dd, J = 7.8, 1.3 Hz, 1H), 7.51 – 7.47 (m, 1H), 7.42 – 7.38 (m, 1H), 7.35 – 7.34 (m, 1H), 2.77 (s, 3H). **^{13}C NMR** (125 MHz,

$CDCl_3$) δ 143.6, 132.0, 129.8, 128.2, 125.3, 41.7. The spectra matched with the previous report.[4]



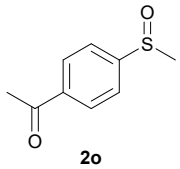
2m

2m: Following General procedure B, the desired product **2m** was obtained in 96% yield (31.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 2.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 151.5, 133.1, 124.4, 117.8, 114.9, 43.9. The spectra matched with the previous report.[1]



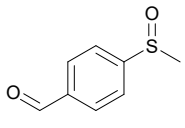
2n

2n: Following General procedure B, the desired product **2n** was obtained in 96% yield (35.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.8 Hz, 2H), 7.84 (d, *J* = 8.8 Hz, 2H), 2.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.3, 149.6, 124.8, 124.6, 44.0. The spectra matched with the previous report.[1]



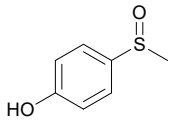
2o

2o: A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide **1z** (0.2 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). Otherwise, the procedure followed General procedure A, the desired product **2z** was obtained in 92% yield (33.6 mg). **^{1}H NMR** (400 MHz, CDCl3) δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 2.75 (s, 3H), 2.64 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.2, 151.0, 139.2, 129.3, 123.9, 43.9, 26.9. The spectra matched with the previous report.[1]



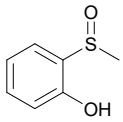
2p

2p: A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide **1z** (0.2 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). Otherwise, the procedure followed General procedure A, the desired product **2z** was obtained in 96% yield (32.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.07 (s, 1H), 8.03 (d, *J* = 8.3 Hz, 2H), 7.81 (d, *J* = 8.3 Hz, 2H), 2.77 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.3, 152.5, 138.2, 130.5, 124.3, 43.9. The spectra matched with the previous report.[1]



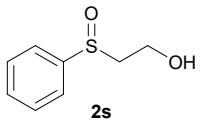
2q

2q: Following General procedure A, the desired product **2q** was obtained in 88% yield (274.9 mg). **^{1}H NMR** (400 MHz, CDCl3) δ 7.49 (d, J = 8.6 Hz, 2H), 6.96 (d, J = 8.6 Hz, 2H), 2.76 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.8, 132.9, 126.3, 117.0, 43.1. The spectra matched with the previous report.[5]



2r

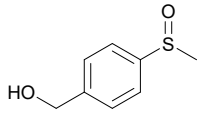
2r: Following General procedure A, the desired product **2r** was obtained in 90% yield (274.9 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) **^{1}H NMR** (500 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.39 – 7.35 (m, 1H), 7.30 – 7.28 (m, 1H), 6.99 – 6.96 (m, 2H), 2.95 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 157.3, 132.9, 125.3, 124.7, 120.2, 118.4, 41.4.The spectra matched with the previous report.[6]



2s

2s: Following General procedure A, the desired product **2s** was obtained in 93% yield (315.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 – 7.61 (m, 2H), 7.54 – 7.46 (m, 3H), 4.17 – 4.10 (m, 1H), 3.96 – 3.90 (m, 1H), 3.86 (s, 2H), 3.12 – 3.05 (m, 1H), 2.95 – 2.89 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.9, 131.3, 129.5, 124.0, 59.4, 56.3. The

spectra matched with the previous report.[7]

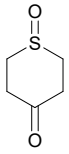


2t

2t: Following General procedure A, the desired product **2t** was obtained in 95% yield (322.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 4.69 (s, 2H), 2.67 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.2, 143.6, 127.7, 123.7, 64.1, 43.7. The spectra matched with the previous report.[8]

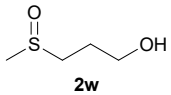
2u

2u: Following General procedure A, the desired product **2u** was obtained in 97% yield (206.1 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 2.83 – 2.73 (m, 4H), 2.40 – 2.31 (m, 2H), 2.98 – 1.90 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 54.3, 25.4. The spectra matched with the previous report.[9]



2v

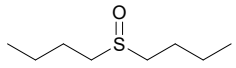
2v: Following General procedure A, the desired product **2v** was obtained in 98% yield (259.6 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 3.30 – 3.24 (m, 2H), 2.90 – 2.83 (m, 1H), 2.52 – 2.48 (m, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 204.9, 47.2, 32.3. The spectra matched with the previous report.[10]



2w

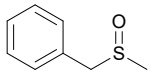
2w: Following General procedure A, the desired product **2w** was obtained in 90% yield (219.6 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 3.67 – 3.60 (m, 2H), 2.80 – 2.77 (m, 2H), 2.53 (s, 3H), 1.98 – 1.91 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 60.5, 51.3, 38.0, 25.9.

The spectra matched with the previous report.[11]



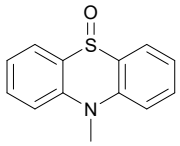
2x

2x: Following General procedure A, the desired product **2x** was obtained in 98% yield (318.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.67 – 2.54 (m, 4H), 1.73 – 1.63 (m, 4H), 1.51 – 1.34 (m, 4H), 0.90 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 52.1, 24.6, 22.1, 13.7. The spectra matched with the previous report.[1]



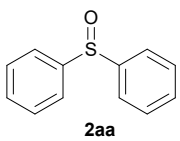
2y

2y: Following General procedure A, the desired product **2y** was obtained in 97% yield (300.1 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 3H), 7.23 – 7.21 (m, 2H) 3.99 (d, *J* = 12.8 Hz, 1H), 3.86 (d, *J* = 12.8 Hz, 1H), 2.39 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 130.1, 129.7, 129.1, 128.6, 60.3, 37.3.The spectra matched with the previous report.[3]



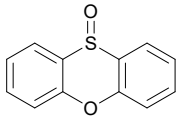
2z

2z: A 50 mL quartz vial equipped with a magnetic stir bar was charged with the sulfide **1z** (0.2 mmol), tetrakis(4-carboxyphenyl)porphyrin (1.6 mg, 0.0020 mmol), and EtOH/H_2O (5.0 mL, 95:5, v/v). Otherwise, the procedure followed General procedure A, the desired product **2z** was obtained in 99% yield (42.9 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.2 Hz, 2H), 7.62 (t, *J* = 6.5 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 7.0 Hz, 2H), 3.75 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 139.9, 132.9, 131.1, 124.6, 121.9, 115.6, 35.4. The spectra matched with the previous report.[1]



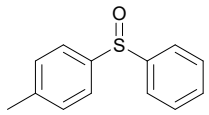
2aa

2aa: Following General procedure B, the desired product **2aa** was obtained in 96% yield (40.1 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.60 (m, 4H), 7.48 – 7.43 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.7, 131.2, 129.5, 124.9. The spectra matched with the previous report.[1]



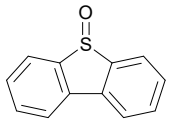
2ab

2ab: Following General procedure B, the desired product **2ab** was obtained in 99% yield (42.9 mg).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 7.8, 1.4 Hz, 2H), 7.68 – 7.59 (m, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.6, 133.9, 131.2, 125.0, 123.8, 119.0. The spectra matched with the previous report.[1]



2ac

2ac: Following General procedure B, the desired product **2ac** was obtained in 99% yield (42.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.47 – 7.40 (m, , 3H), 7.26 (d, *J* = 8.0 Hz, 2H), 2.36 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.8, 142.5, 141.8, 131.0, 130.1, 129.4, 125.1, 124.8, 21.5. The spectra matched with the previous report.[1]

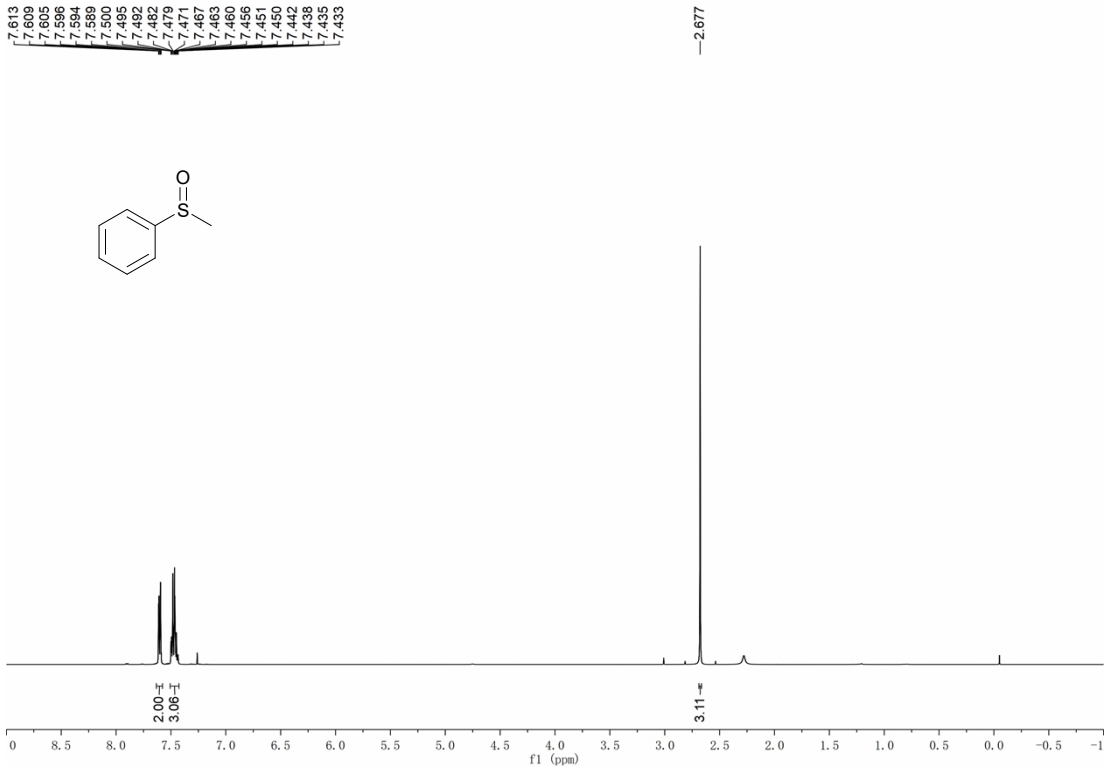


2ad

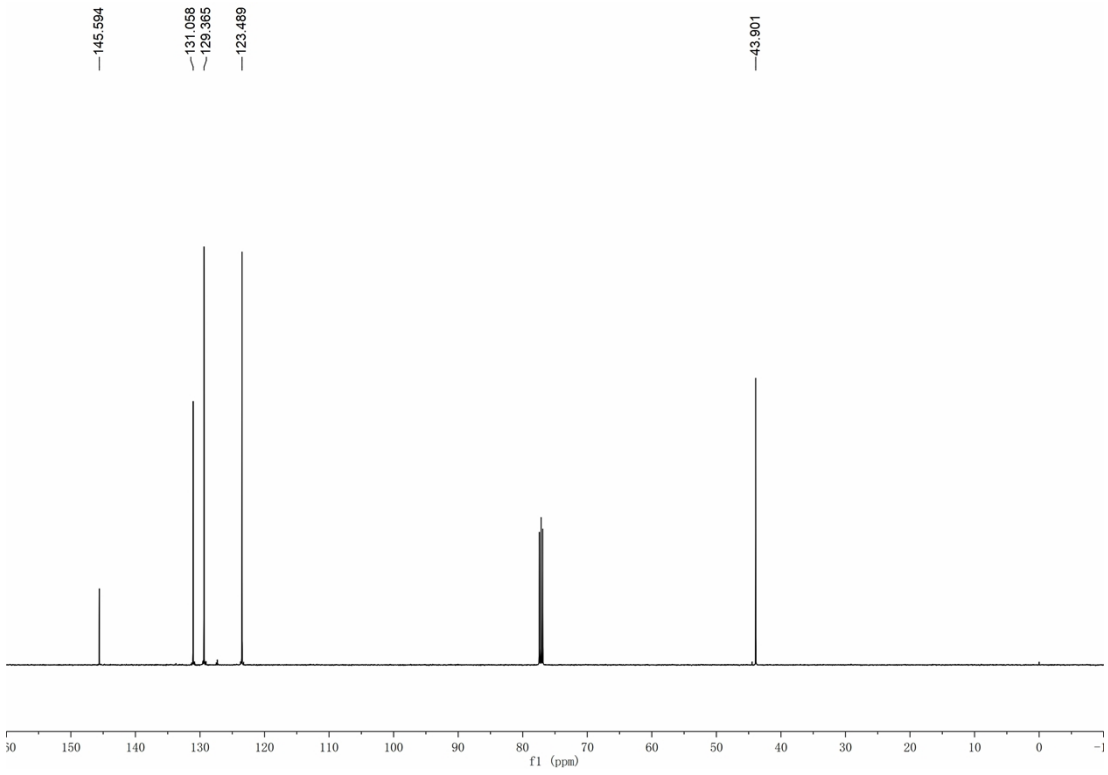
2ad: Following General procedure B, the desired product **2ad** was obtained in 99% yield (39.6 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 2H), 7.58 – 7.55 (m, 2H), 7.48 – 7.45 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.7, 137.2, 132.7, 129.6, 127.6, 122.0.The spectra matched with the previous report.[1]

6. NMR spectra of substrates and products

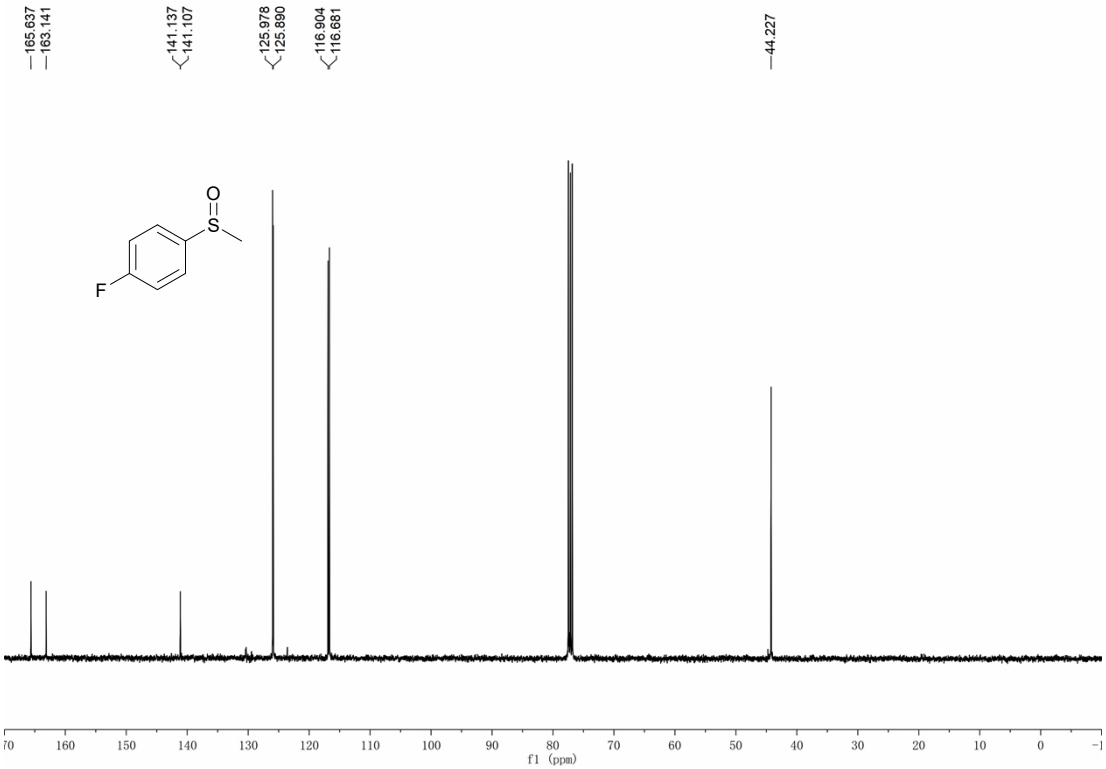
^{1}H NMR (500 MHz, $CDCl_3$) of 2a



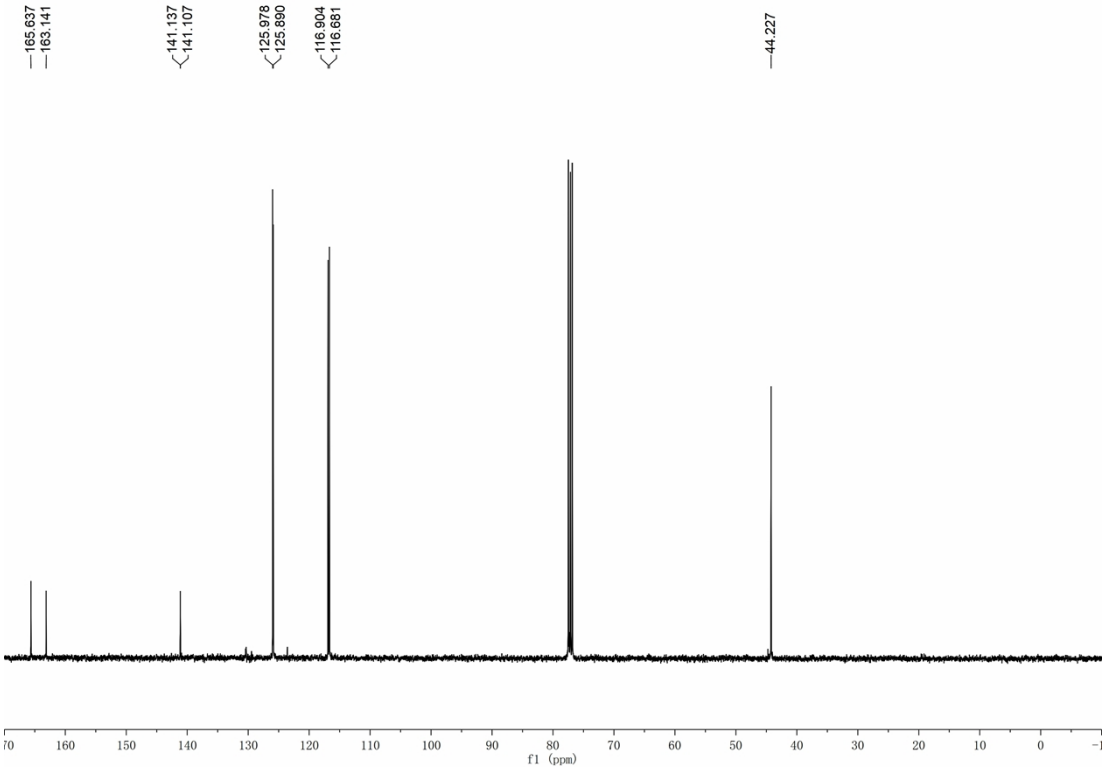
^{13}C NMR (500 MHz, $CDCl_3$) of 2a



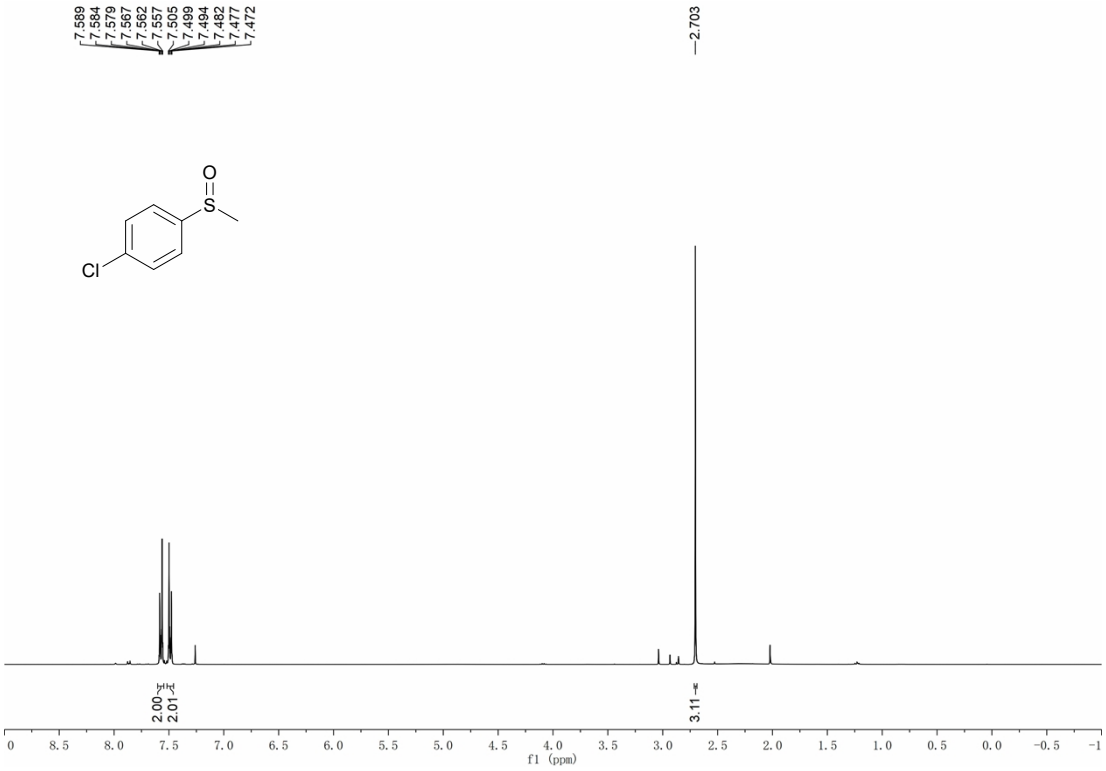
^{1}H NMR (400 MHz, $CDCl_3$) of 2d



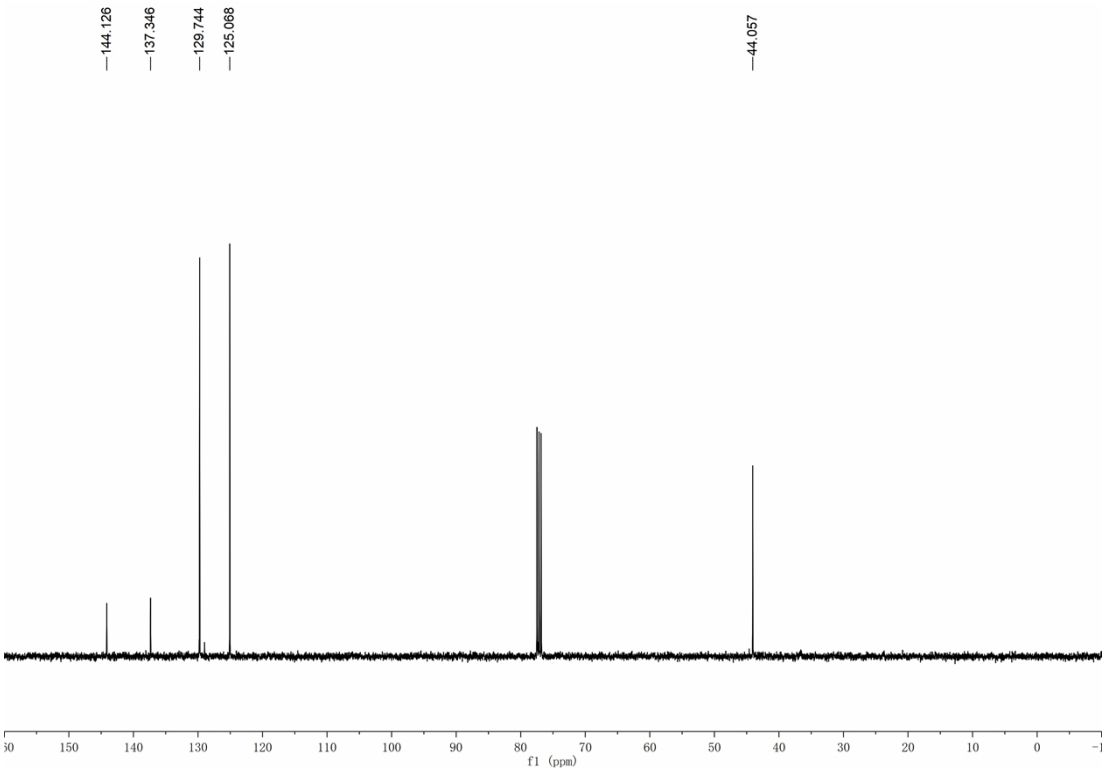
^{13}C NMR (400 MHz, $CDCl_3$) of 2d



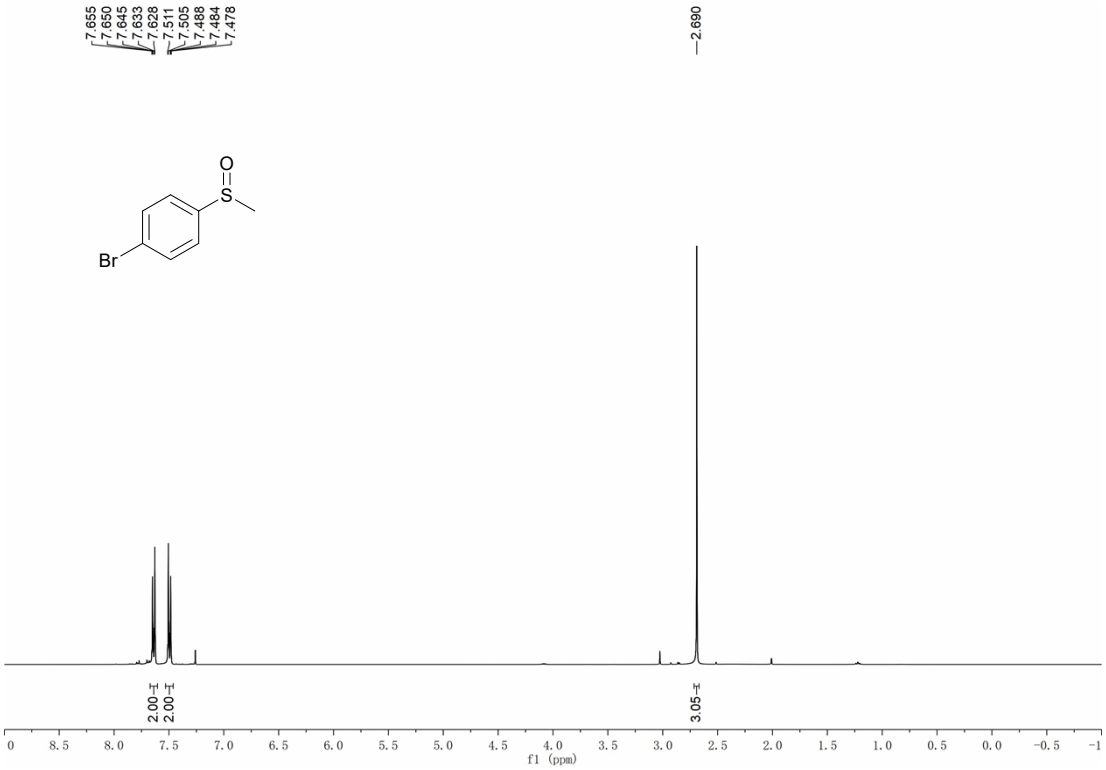
^{1}H NMR (400 MHz, $CDCl_3$) of 2e



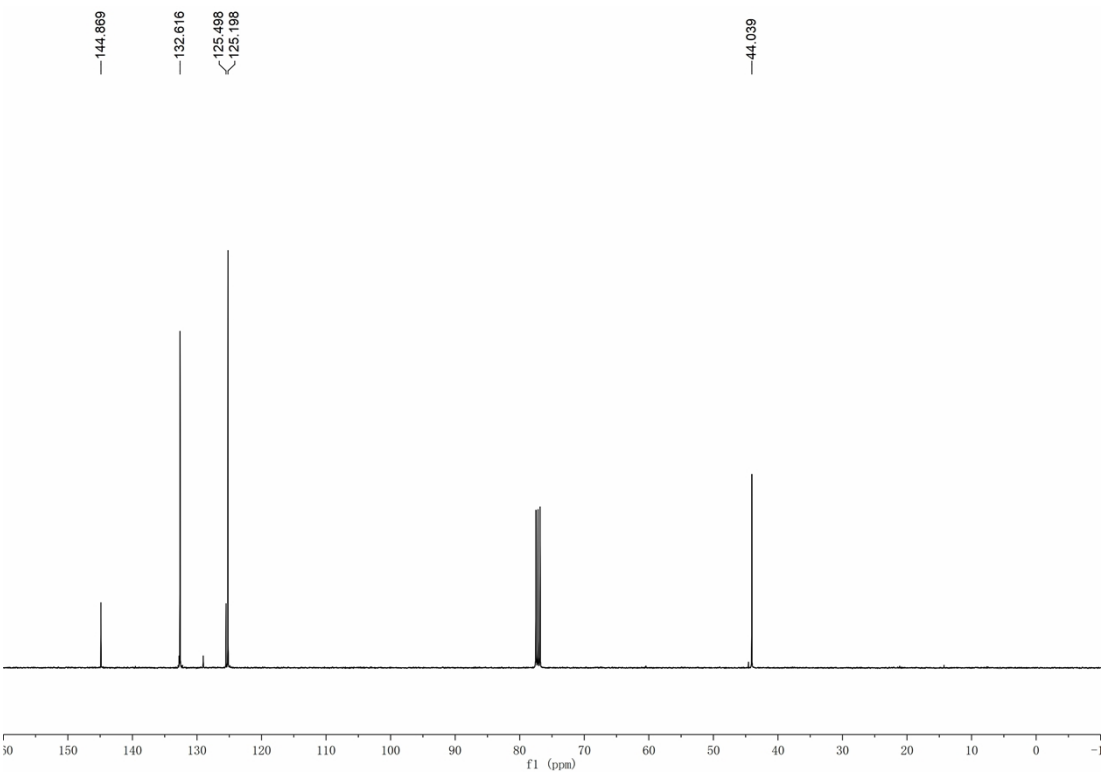
^{13}C NMR (400 MHz, $CDCl_3$) of 2e



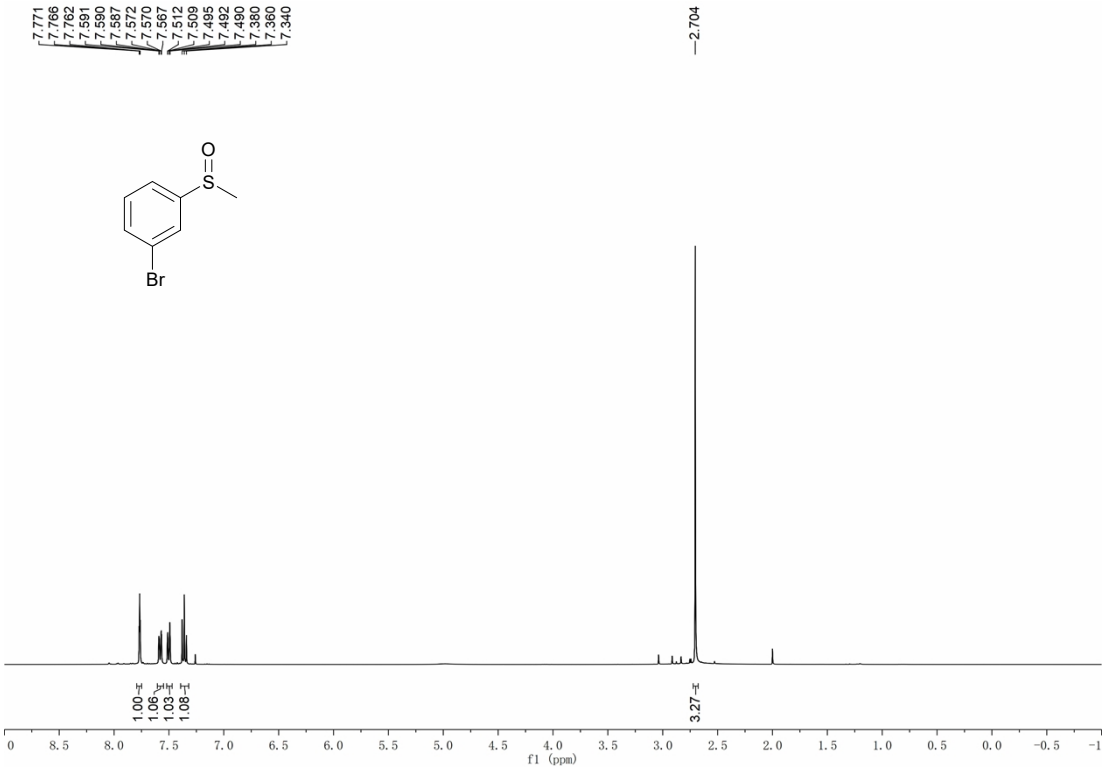
^{1}H NMR (400 MHz, $CDCl_3$) of 2f



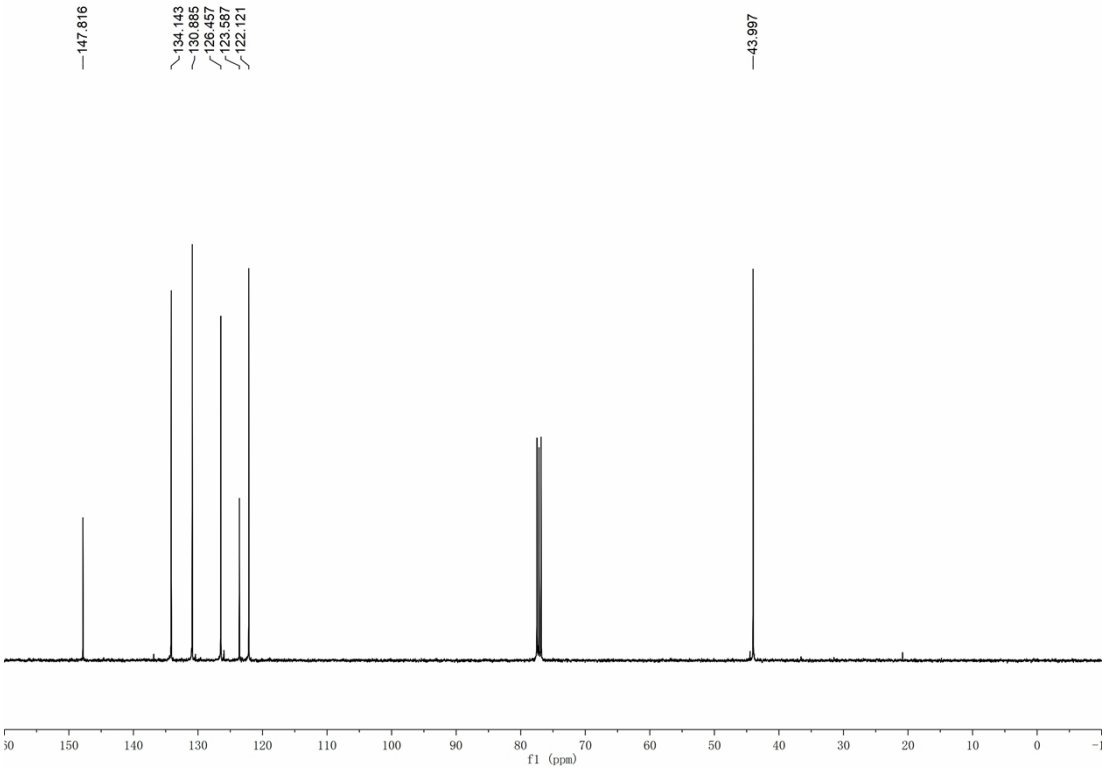
^{13}C NMR (400 MHz, $CDCl_3$) of 2f



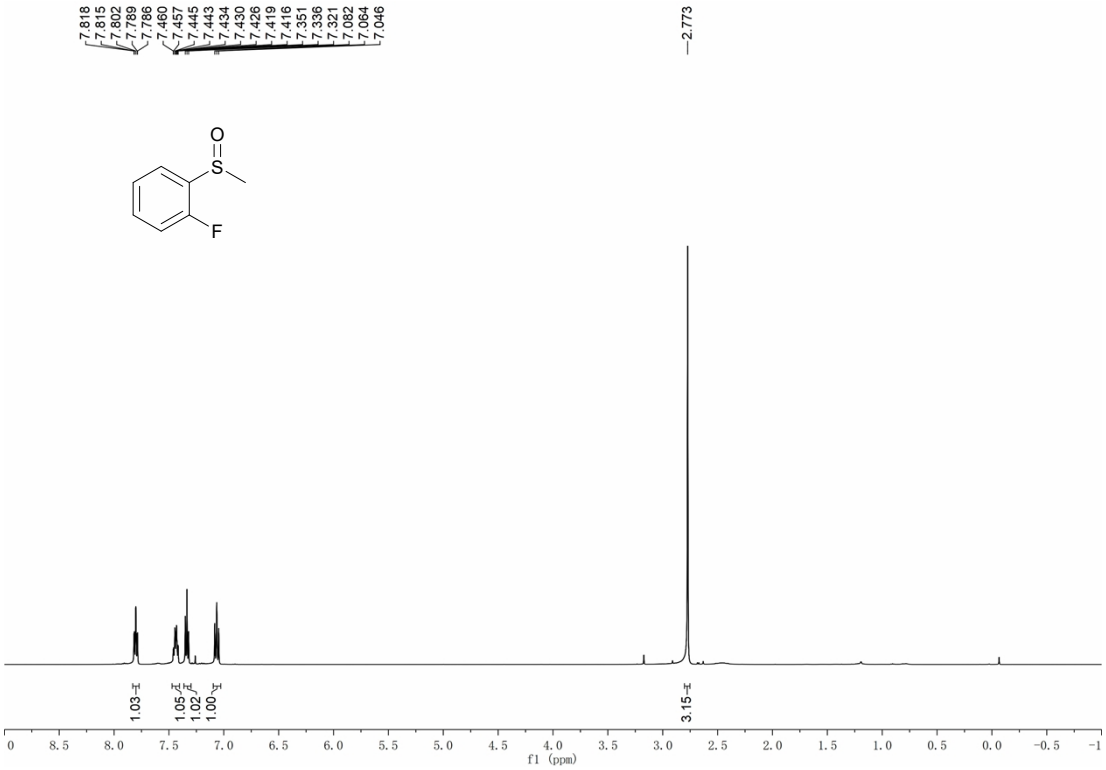
^{1}H NMR (400 MHz, $CDCl_3$) of 2j



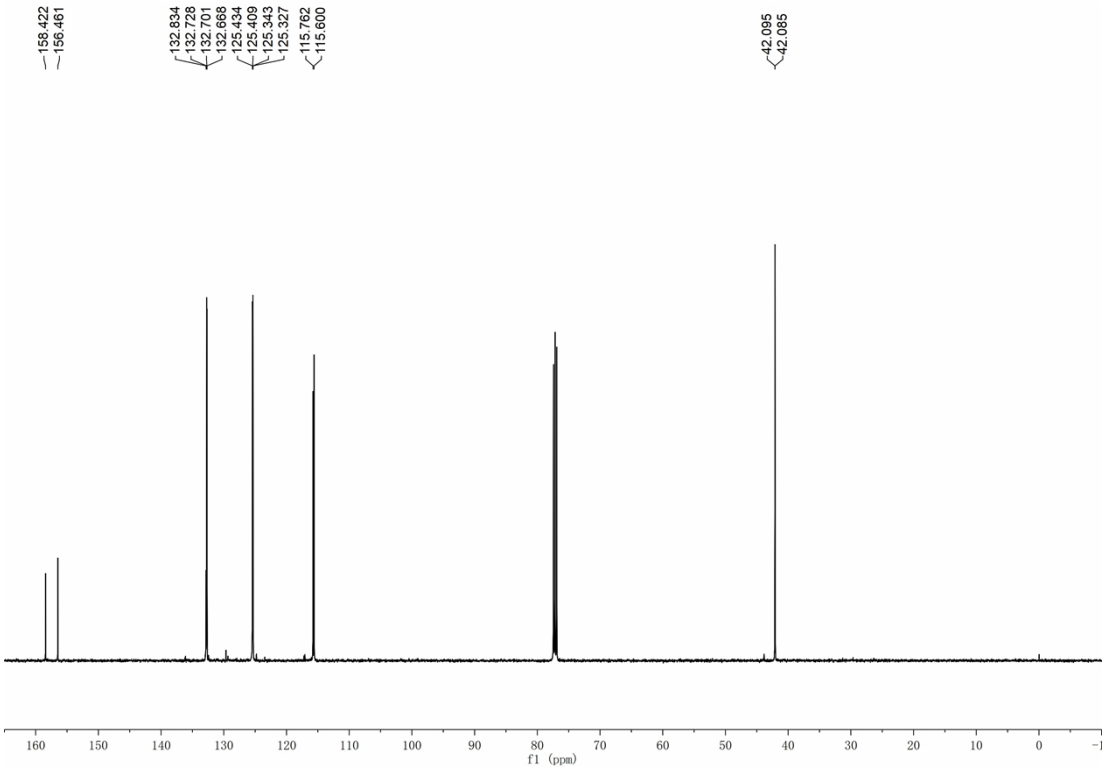
^{13}C NMR (400 MHz, $CDCl_3$) of 2j



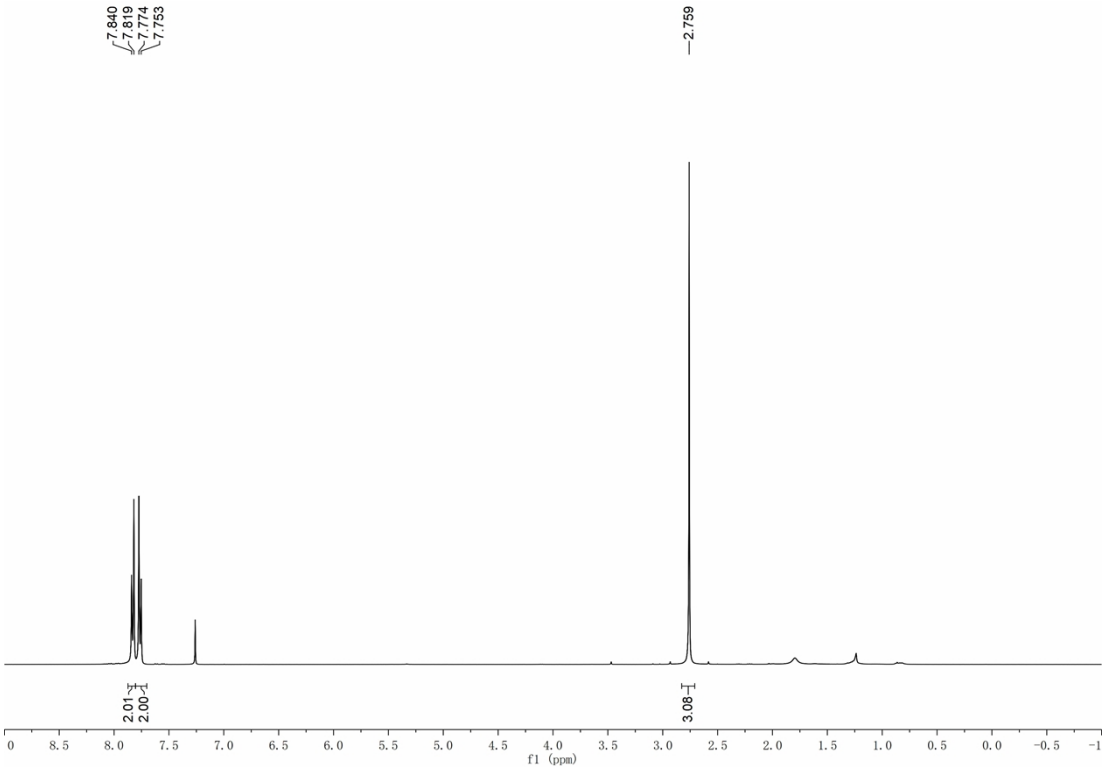
^{1}H NMR (500 MHz, $CDCl_3$) of 2k



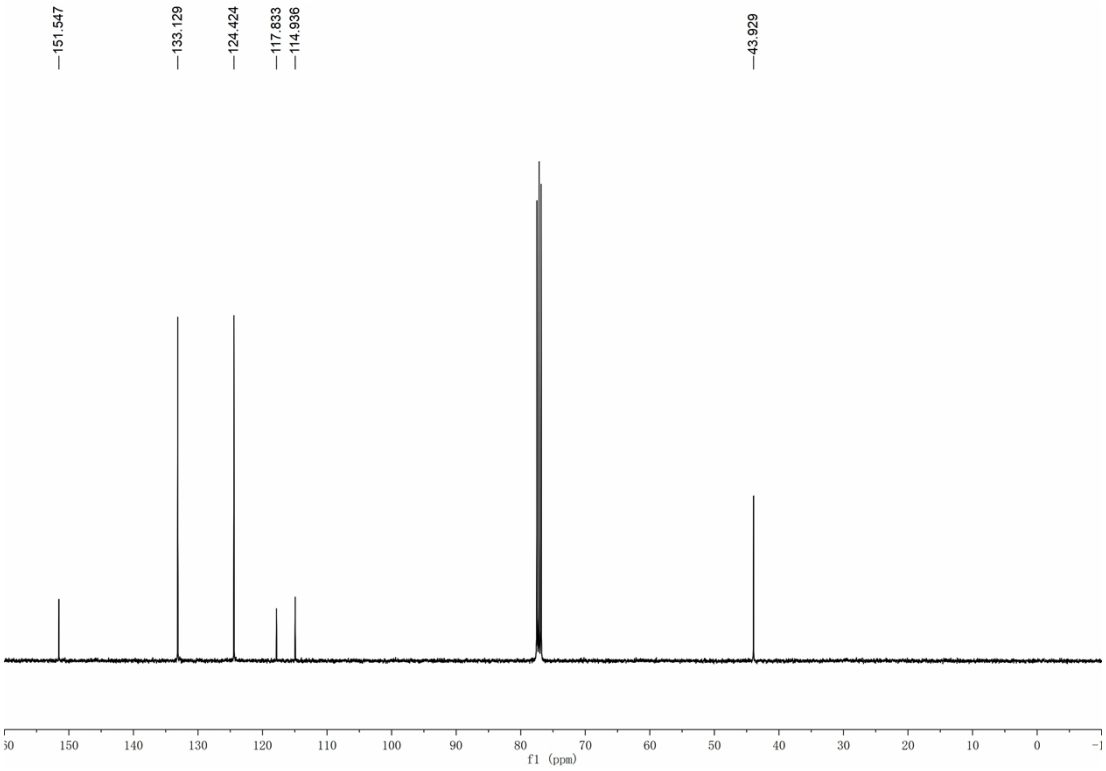
^{13}C NMR (500 MHz, $CDCl_3$) of 2k



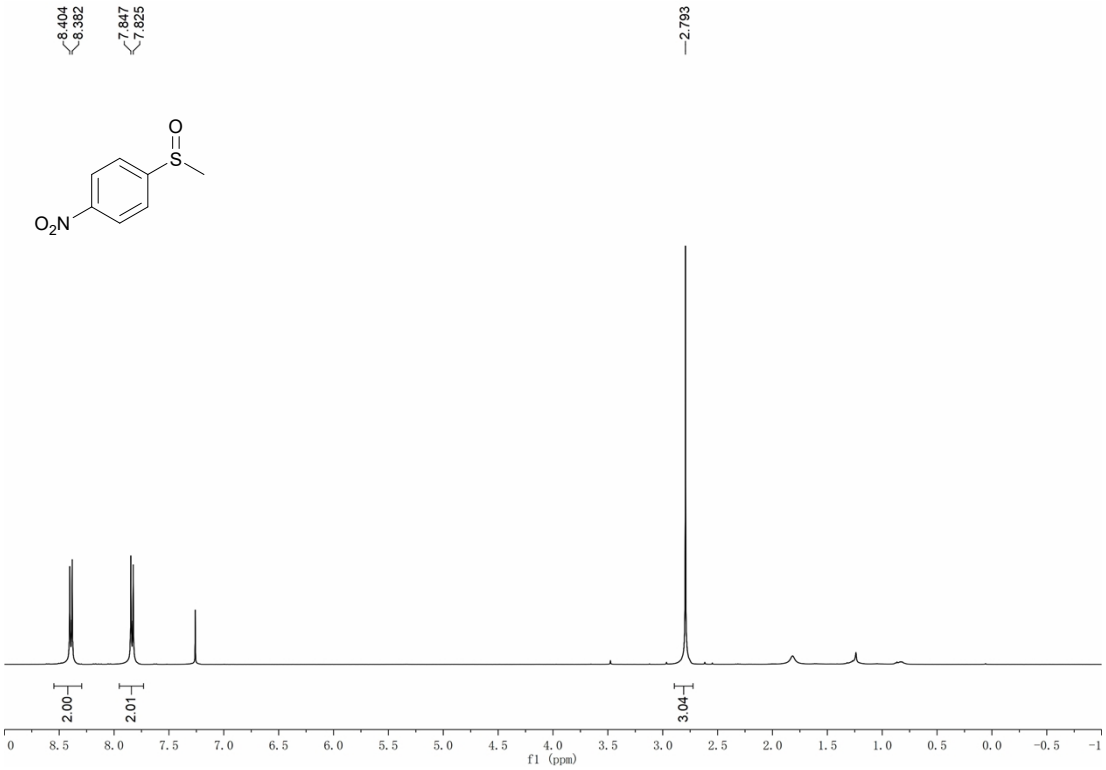
^{1}H NMR (400 MHz, $CDCl_3$) of 2m



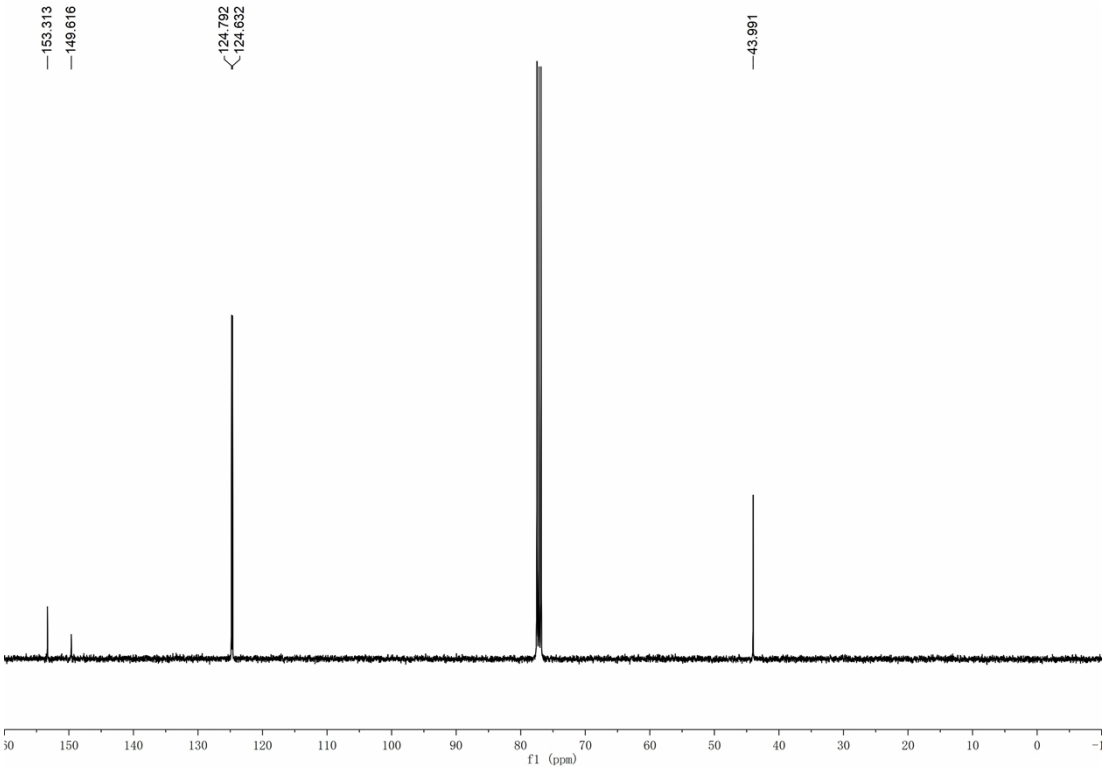
^{1}H NMR (400 MHz, $CDCl_3$) of 2m



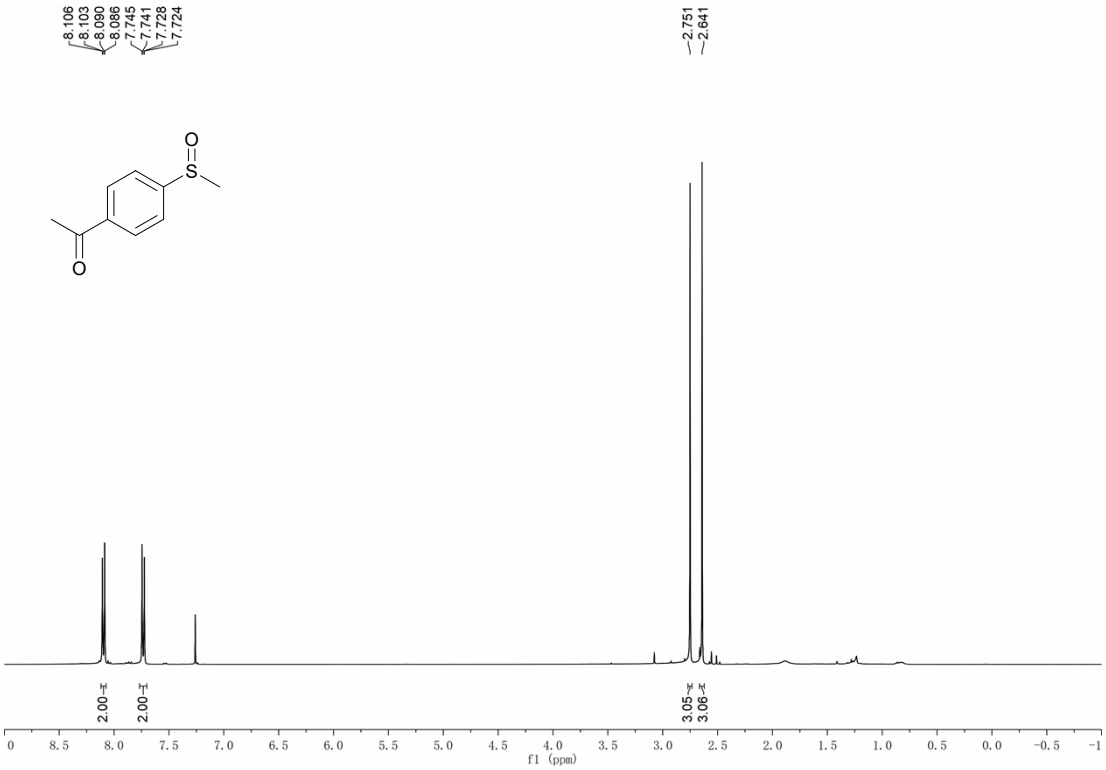
^{1}H NMR (400 MHz, $CDCl_3$) of 2n



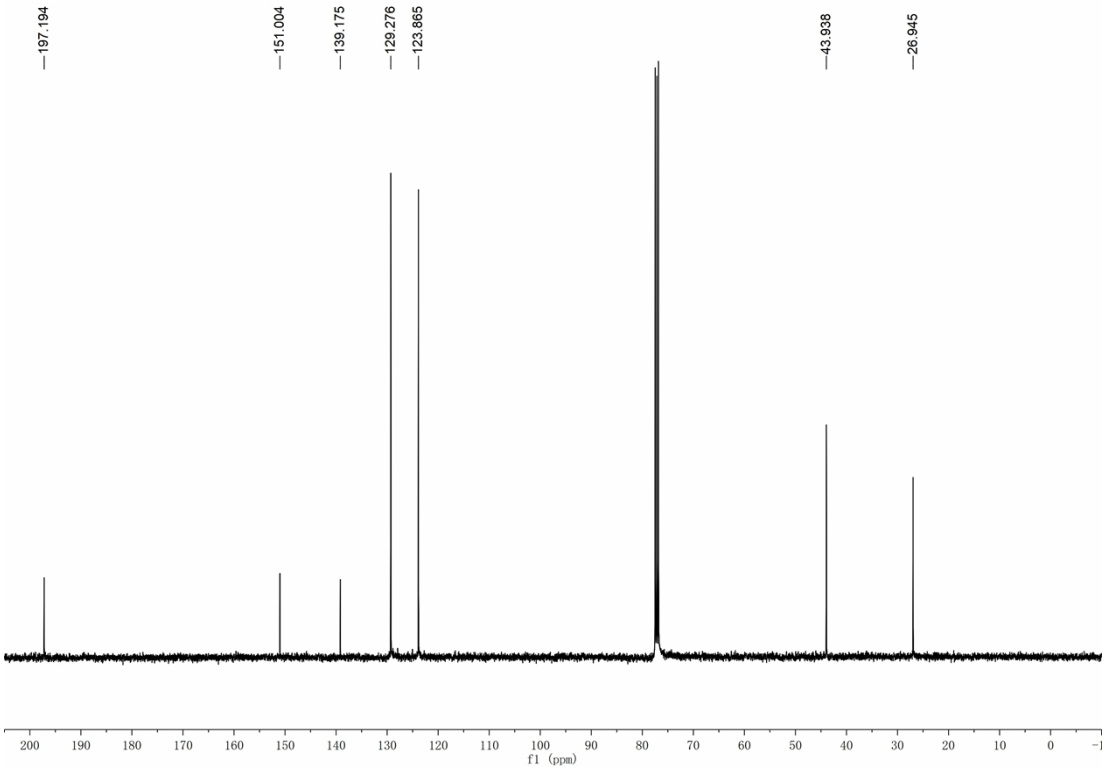
^{1}H NMR (400 MHz, $CDCl_3$) of 2n



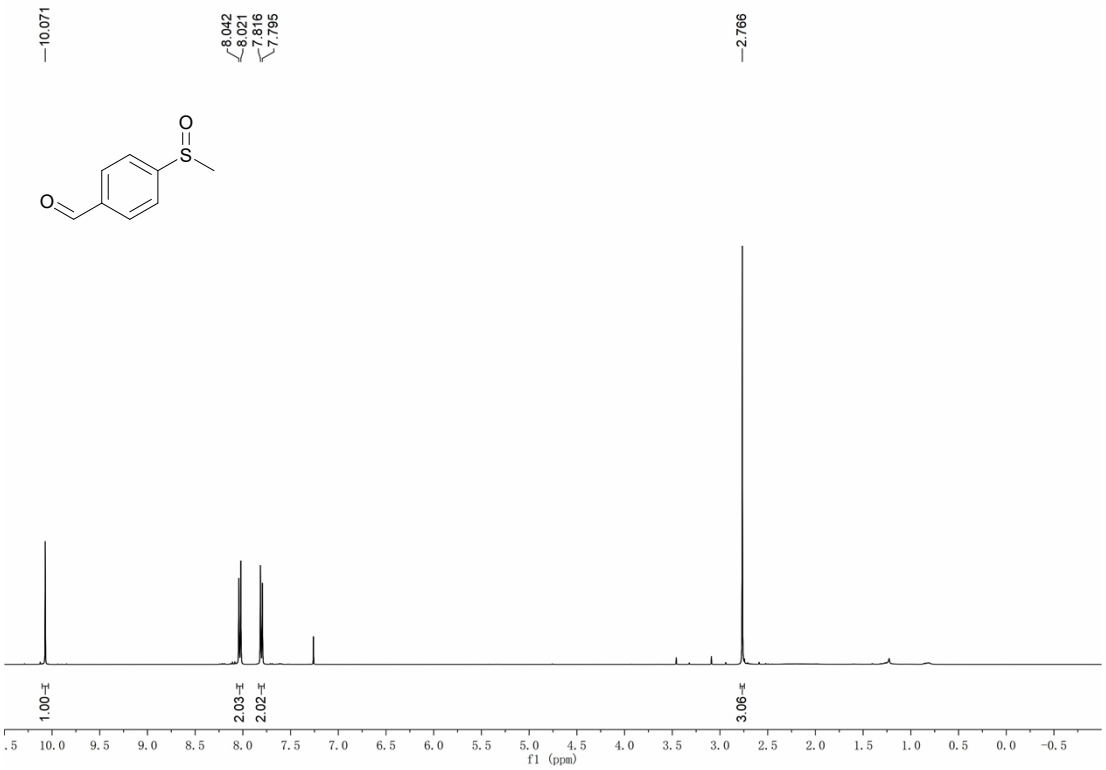
^{1}H NMR (400 MHz, $CDCl_3$) of 2o



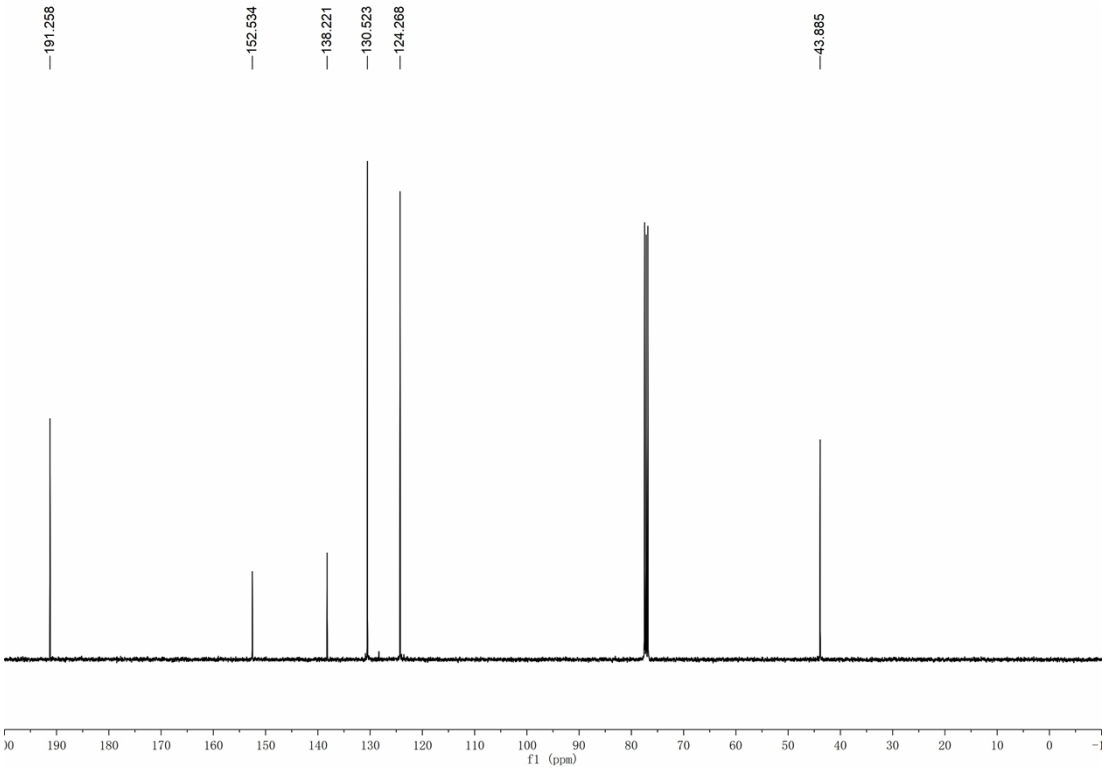
^{13}C NMR (400 MHz, $CDCl_3$) of 2o



^{1}H NMR (400 MHz, $CDCl_3$) of 2p

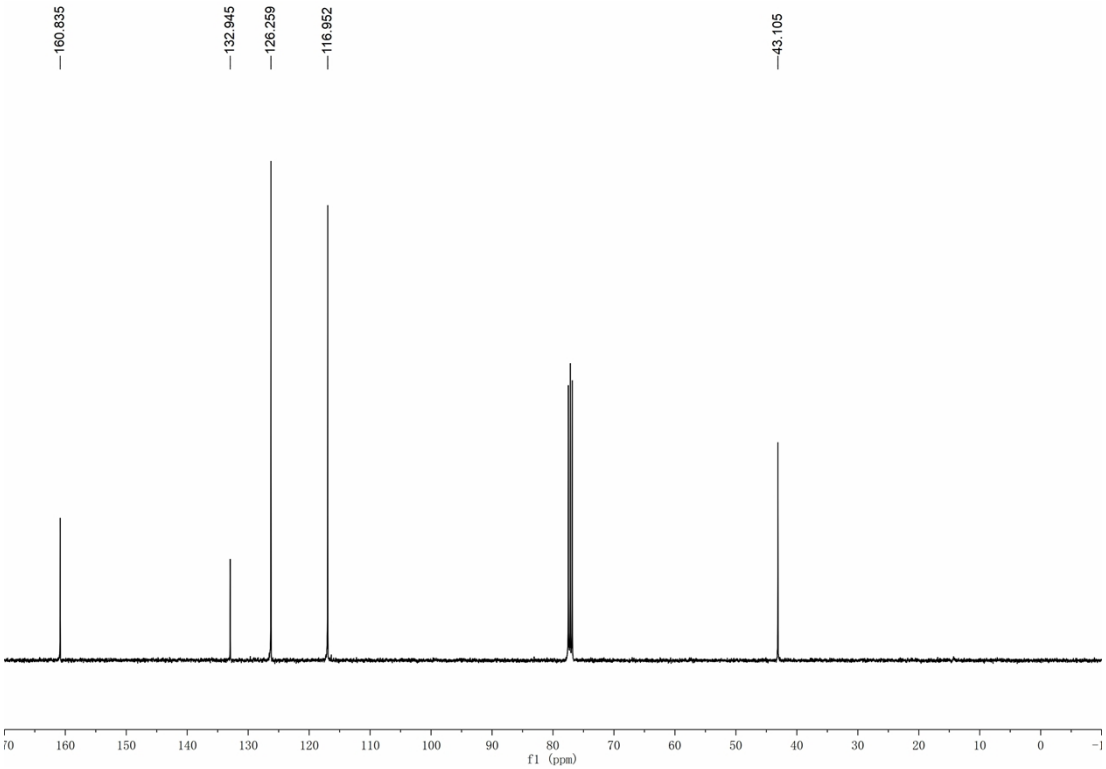


^{13}C NMR (400 MHz, $CDCl_3$) of 2p

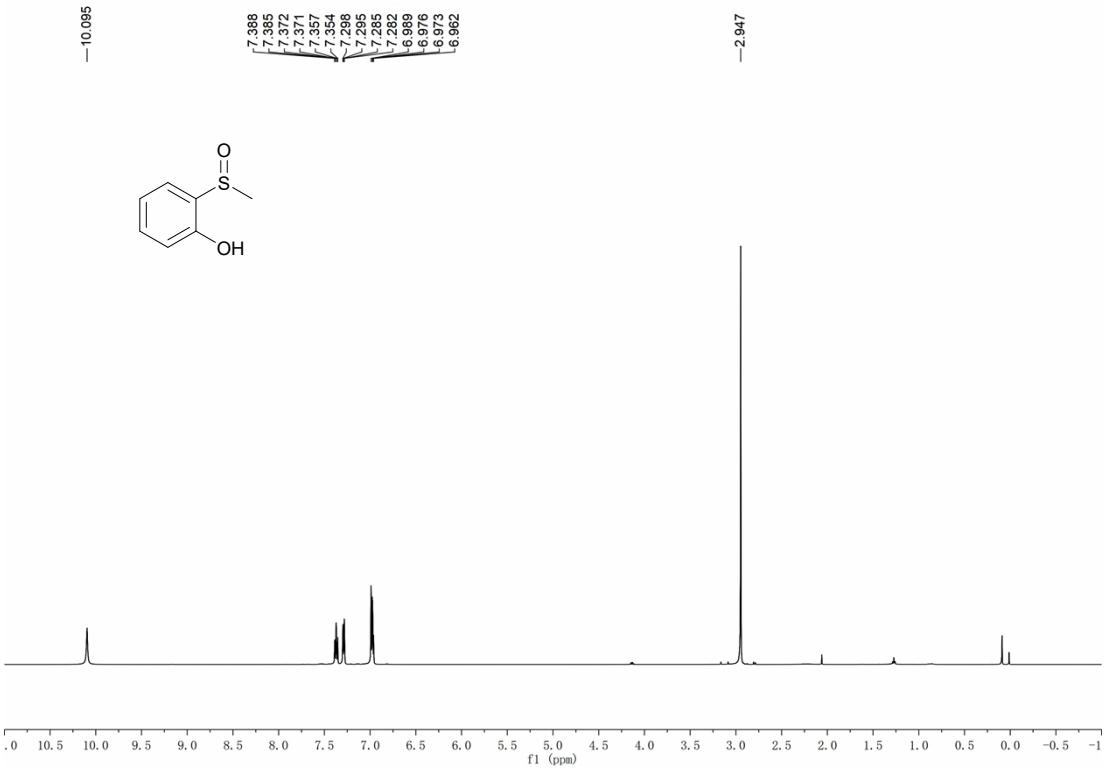


^{1}H NMR (400 MHz, $CDCl_3$) of 2q

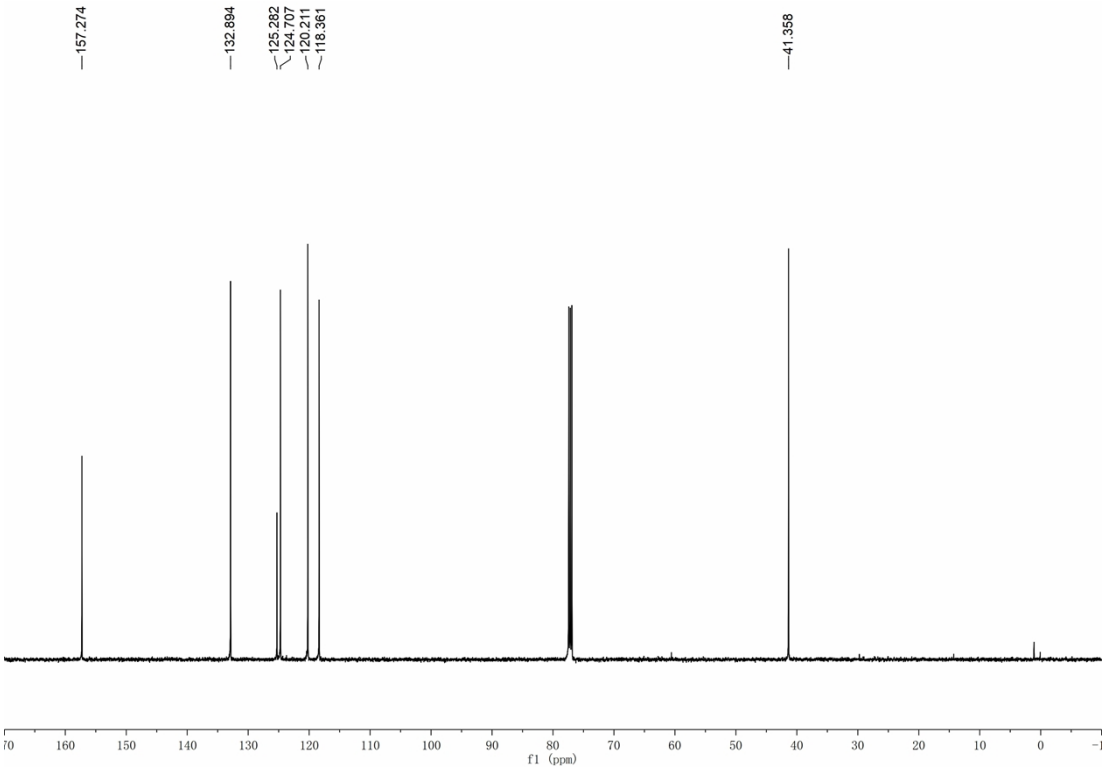
^{13}C NMR (400 MHz, $CDCl_3$) of 2q



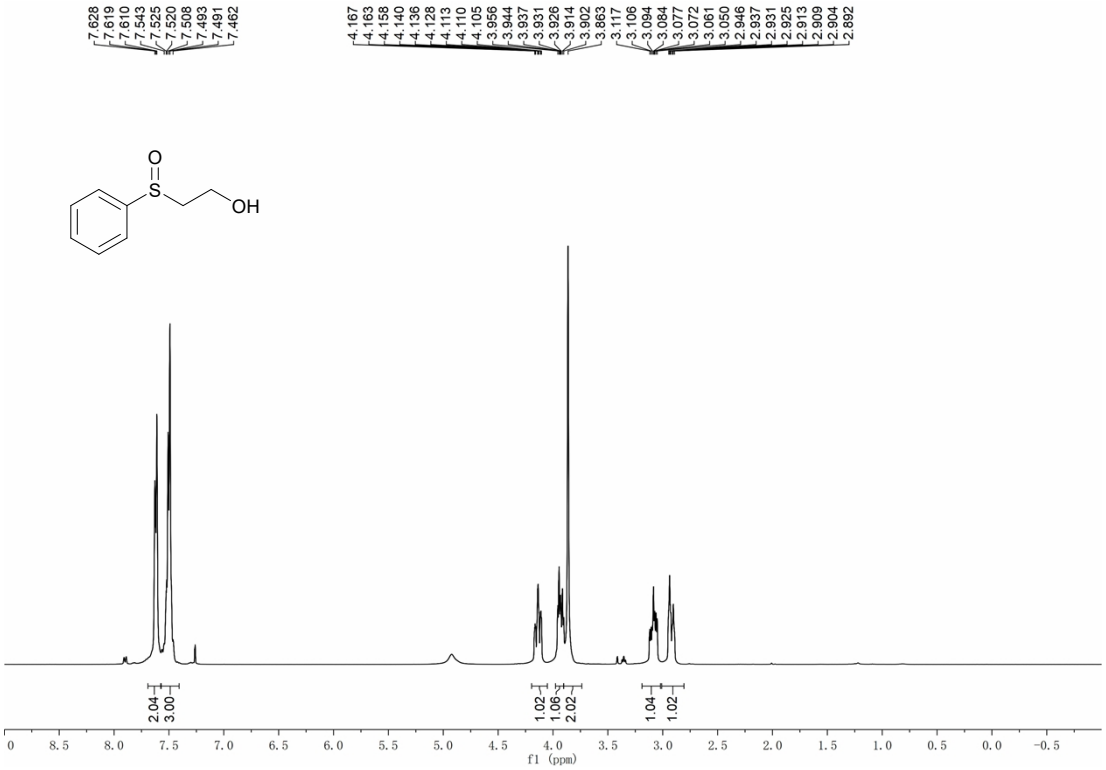
^{1}H NMR (500 MHz, $CDCl_3$) of 2r



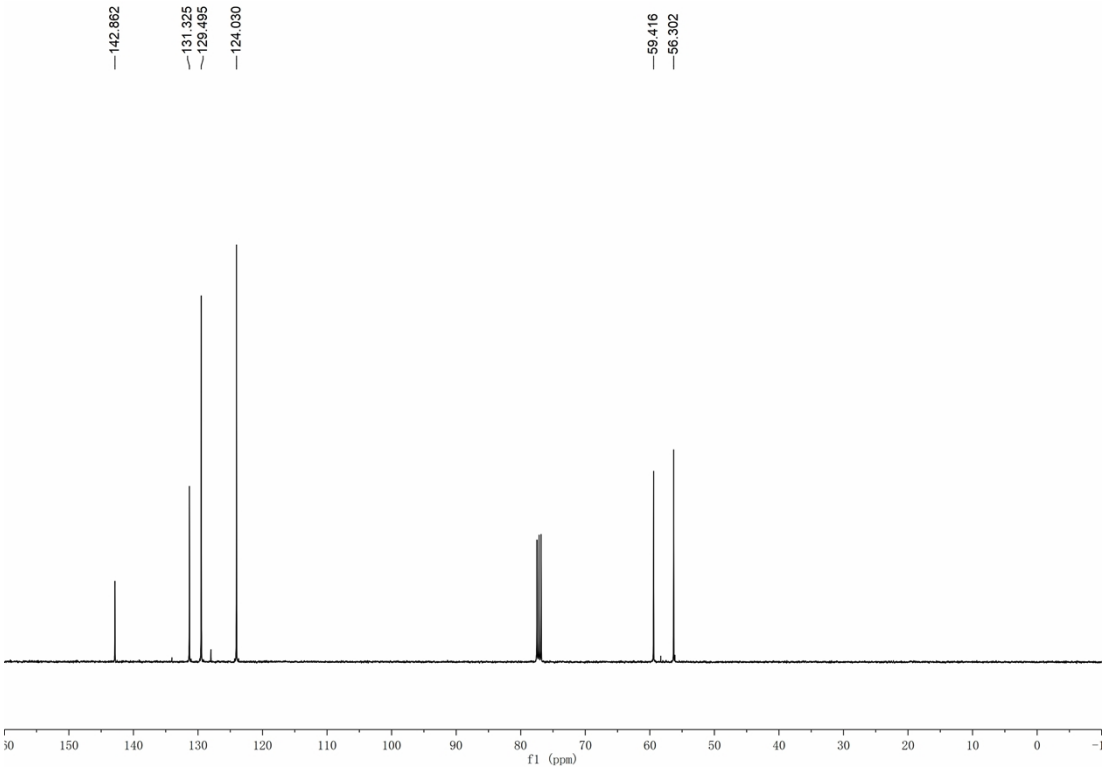
^{13}C NMR (500 MHz, $CDCl_3$) of 2r



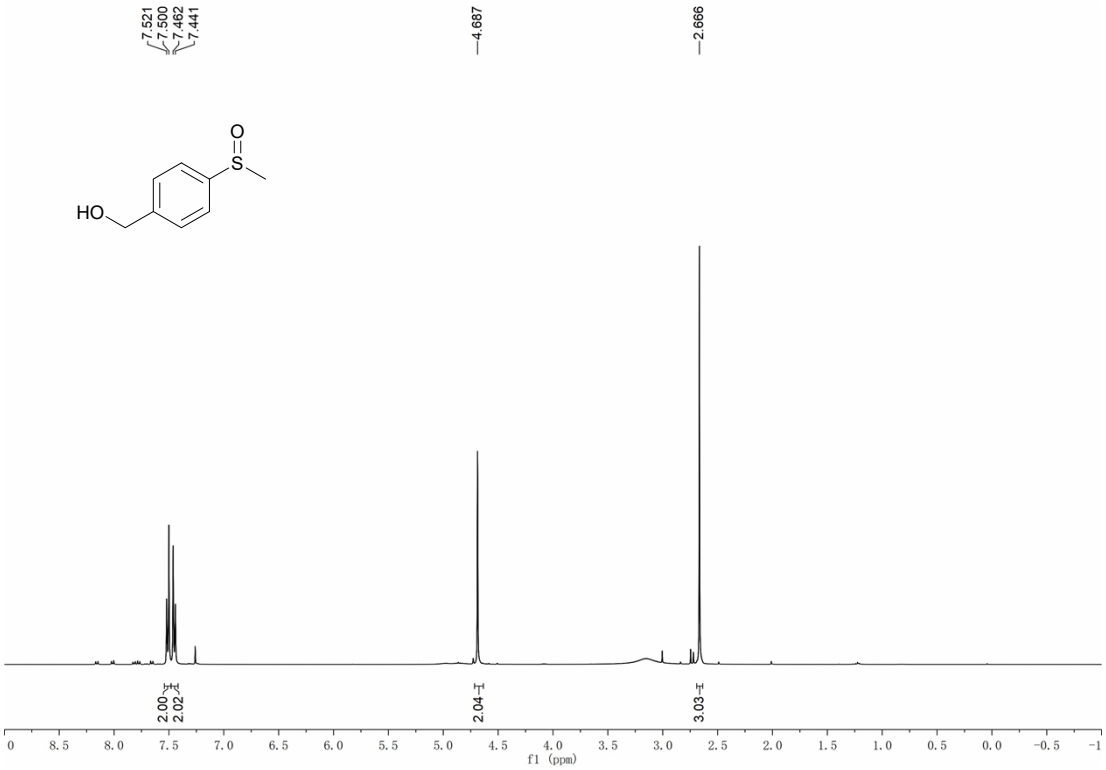
^{1}H NMR (400 MHz, $CDCl_3$) of 2s



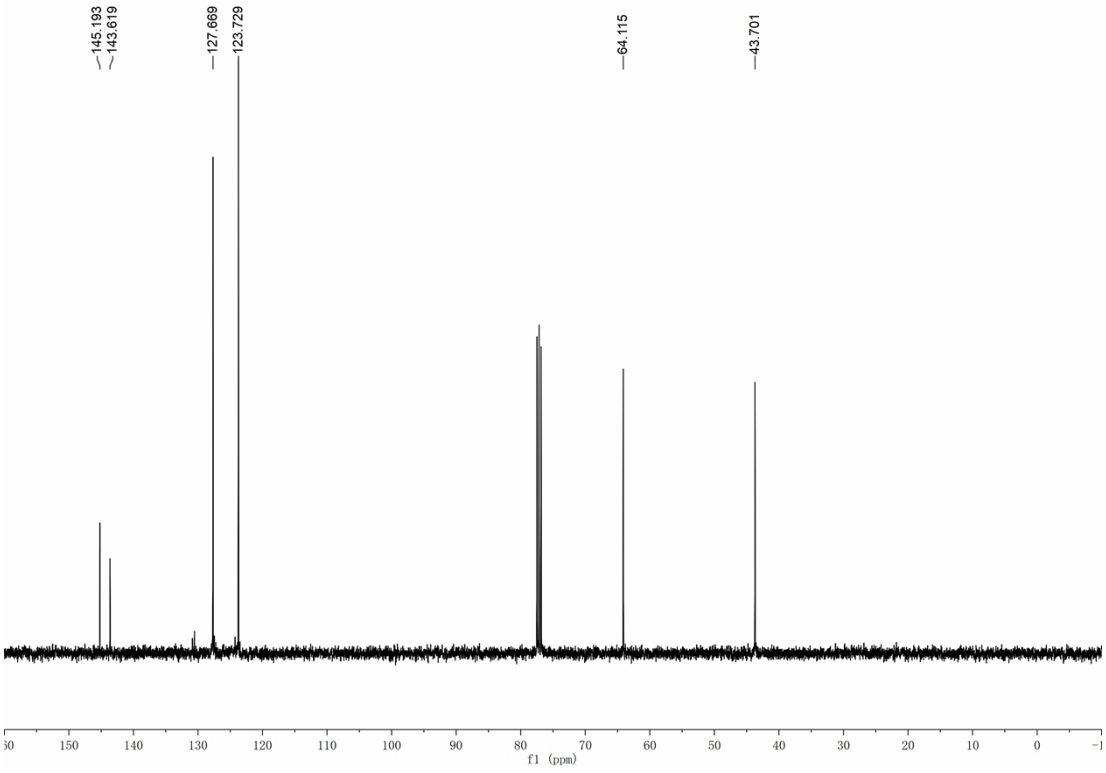
^{13}C NMR (400 MHz, $CDCl_3$) of 2s



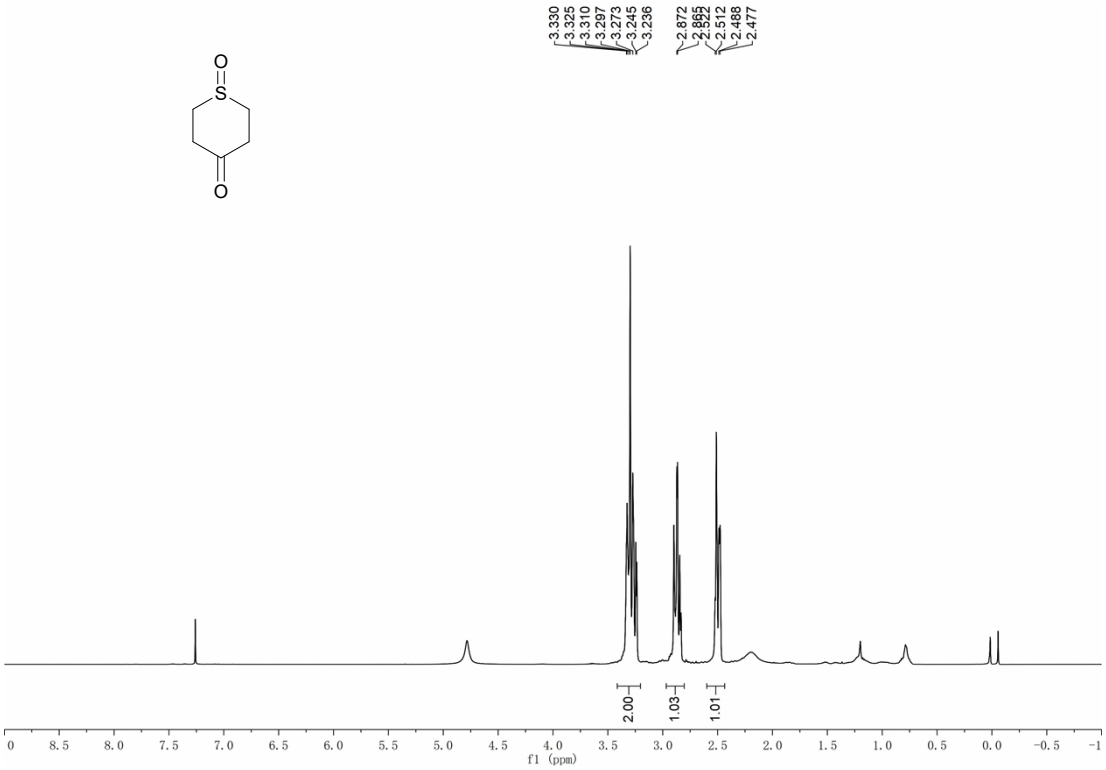
^{1}H NMR (400 MHz, $CDCl_3$) of 2t



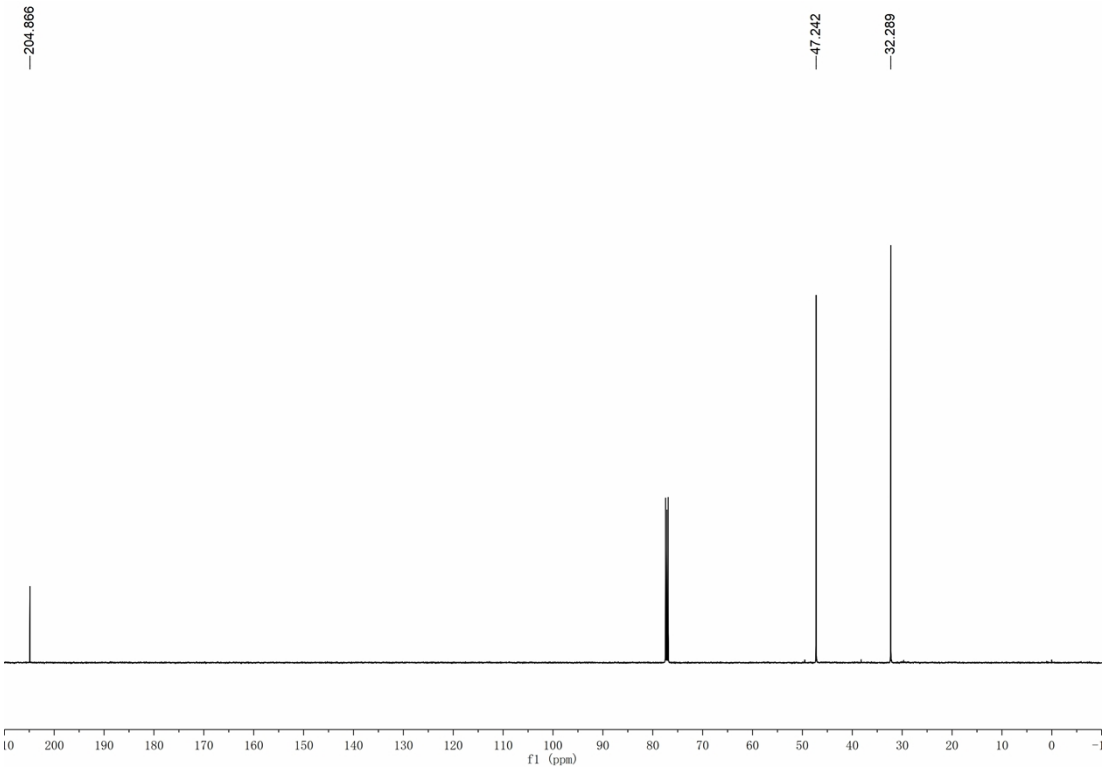
^{1}H NMR (400 MHz, $CDCl_3$) of 2t



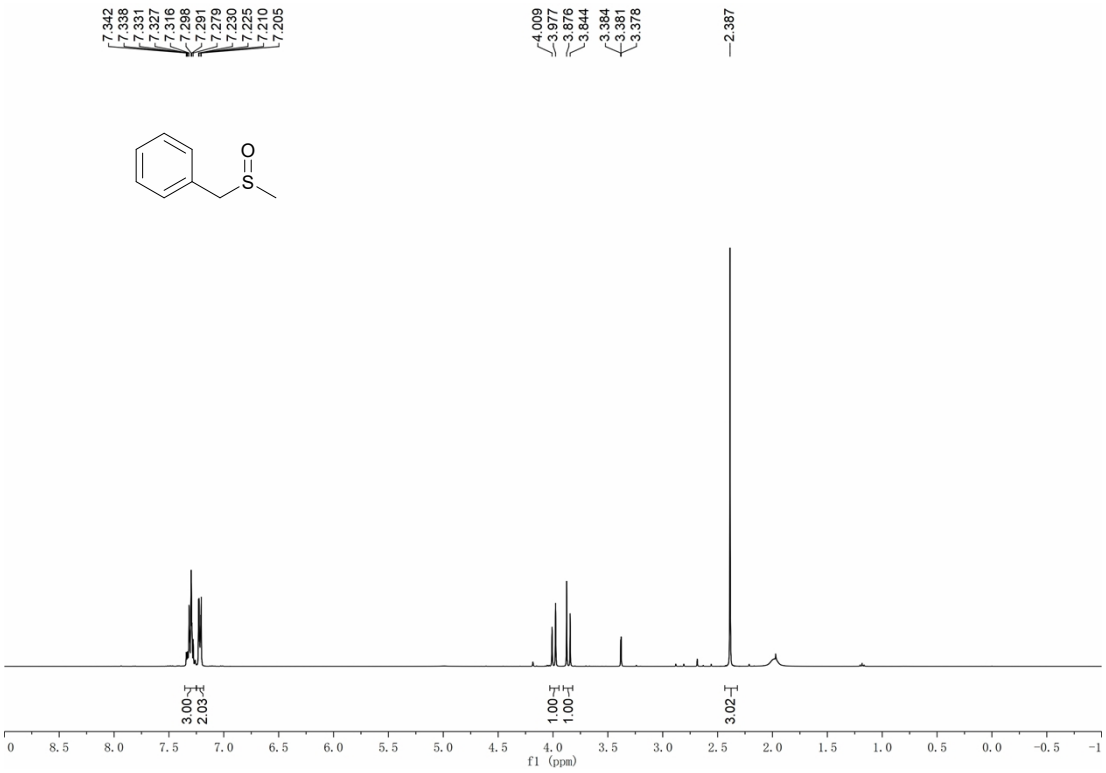
^{1}H NMR (500 MHz, $CDCl_3$) of 2v



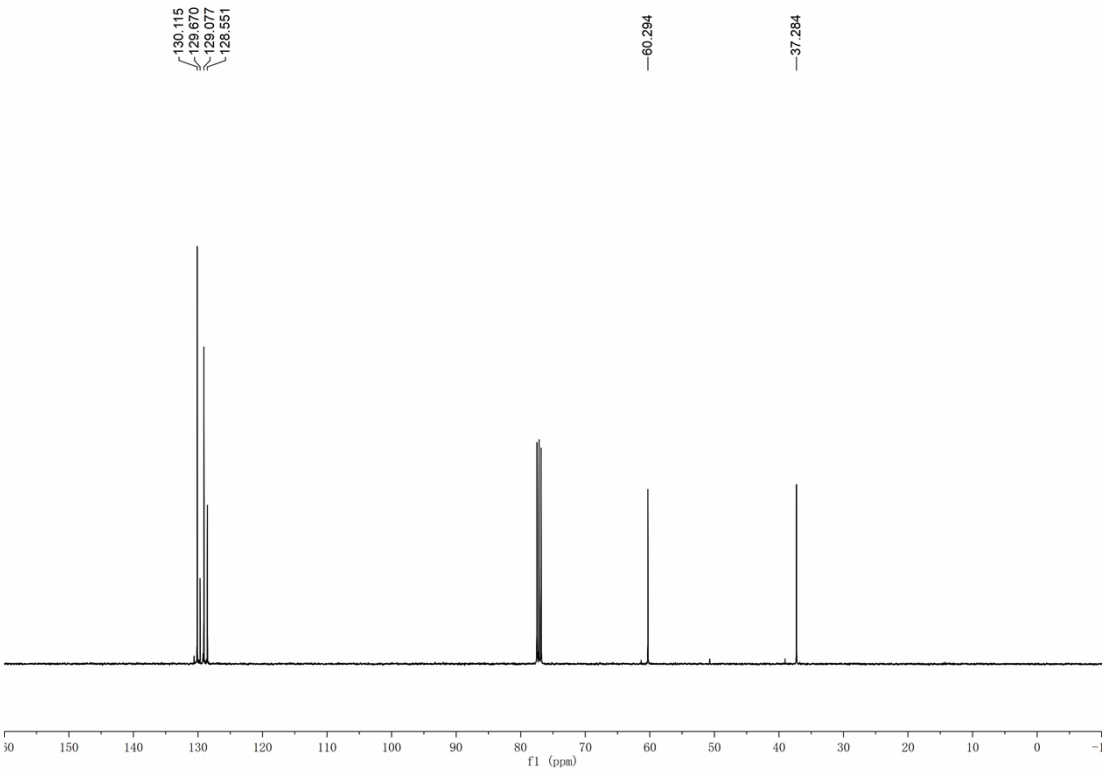
^{13}C NMR (500 MHz, $CDCl_3$) of 2v



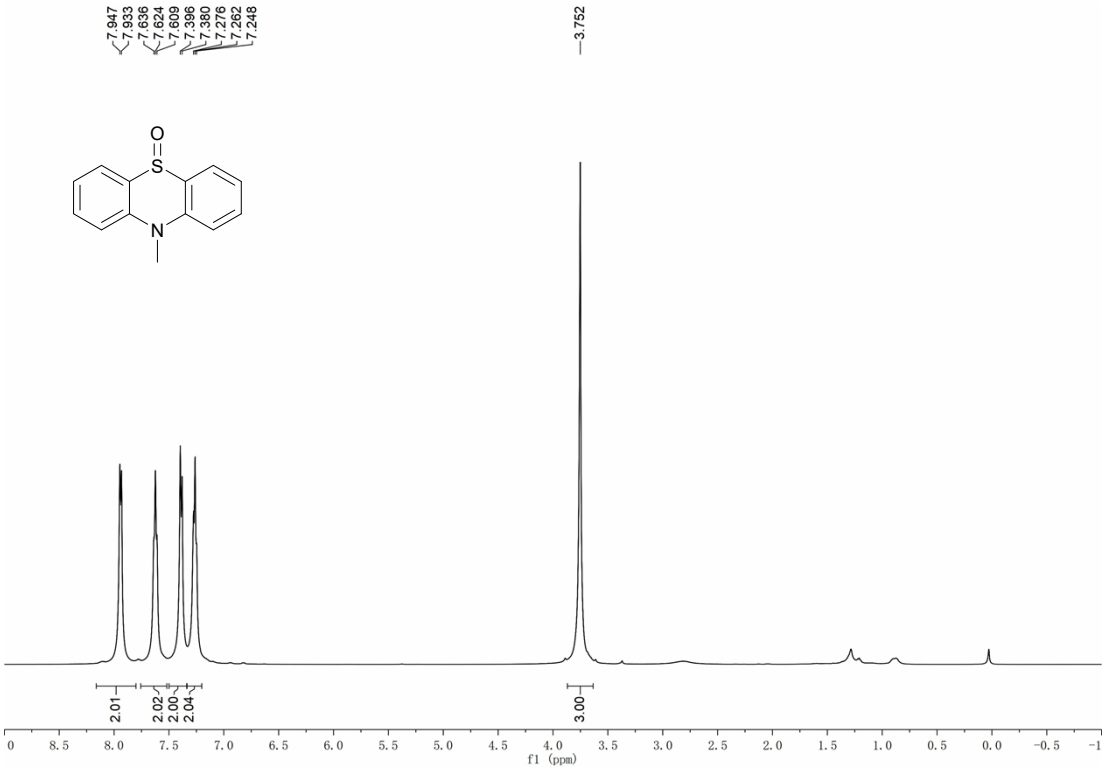
^{1}H NMR (400 MHz, $CDCl_3$) of 2y



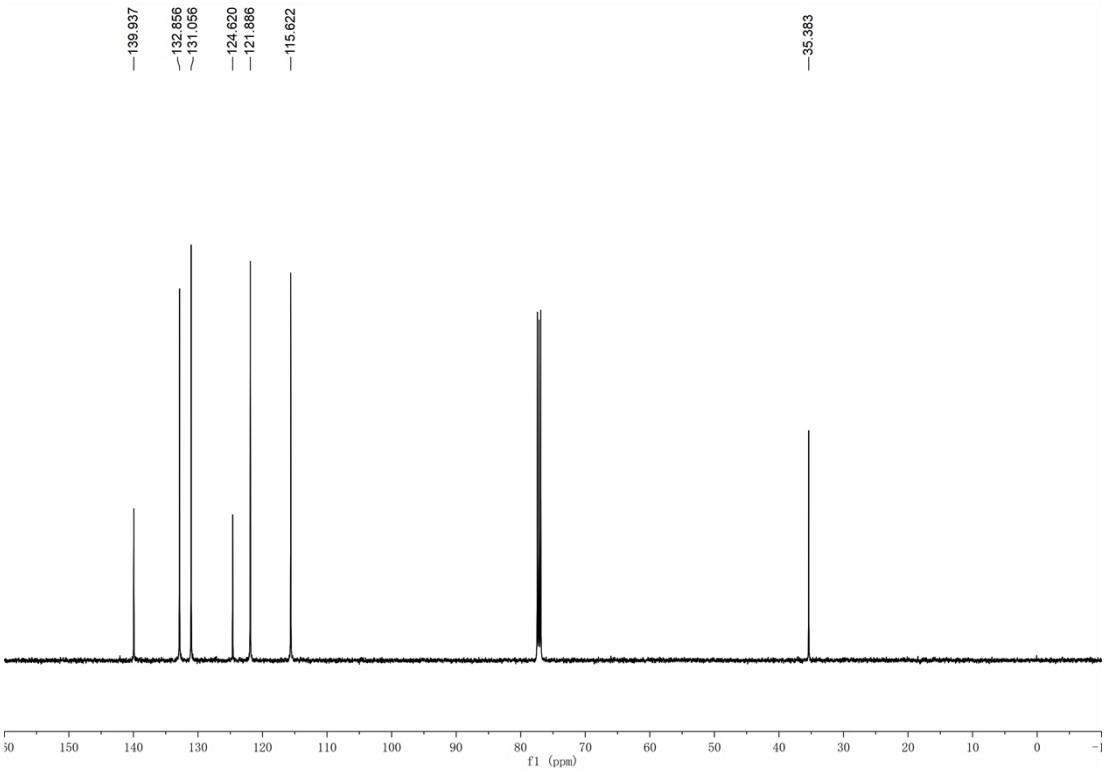
^{13}C NMR (400 MHz, $CDCl_3$) of 2y



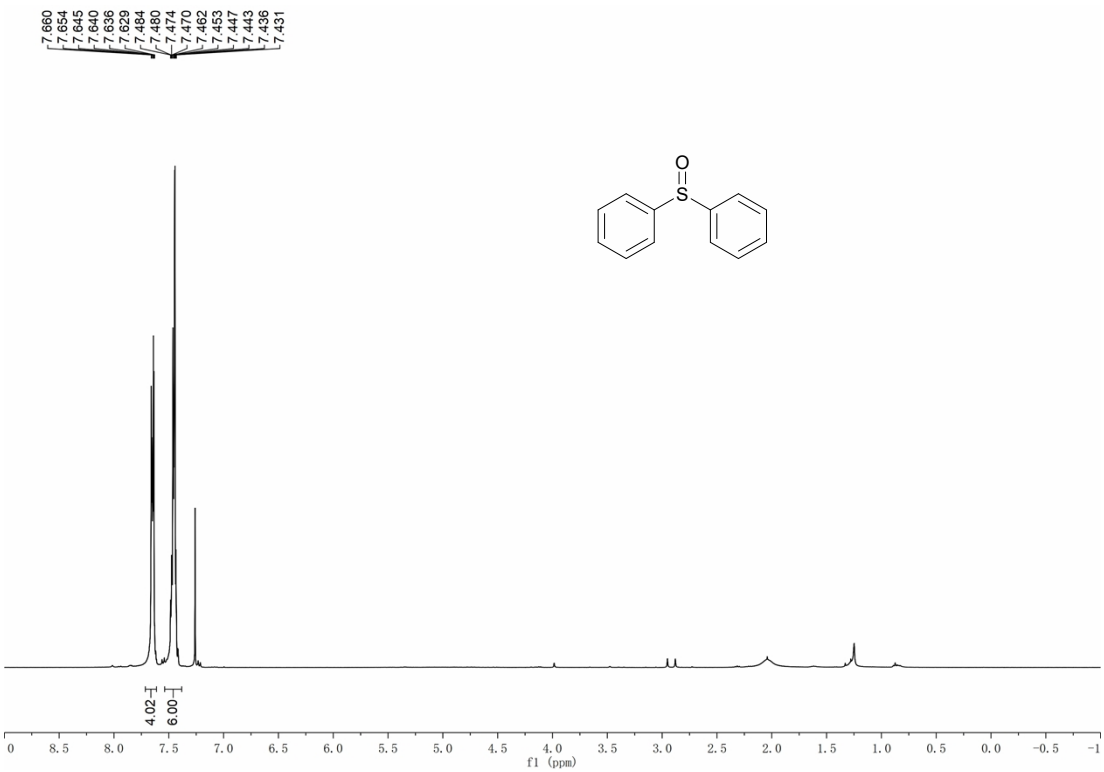
^{1}H NMR (500 MHz, $CDCl_3$) of 2z



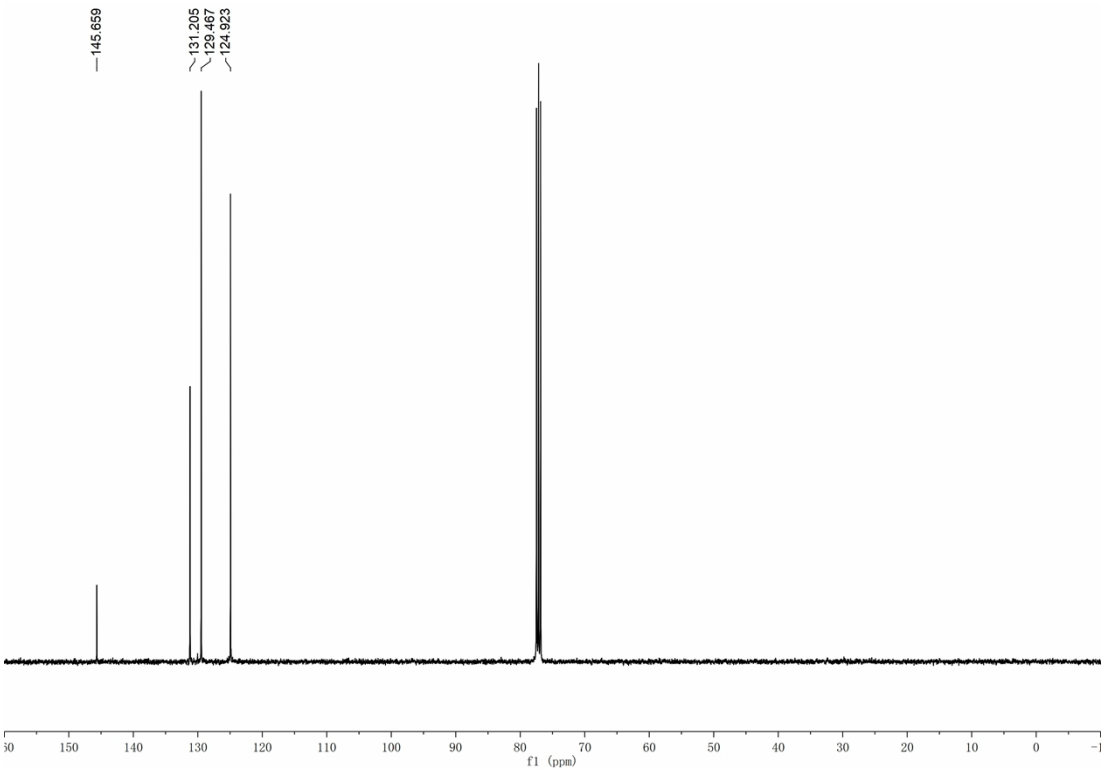
^{1}H NMR (500 MHz, $CDCl_3$) of 2z



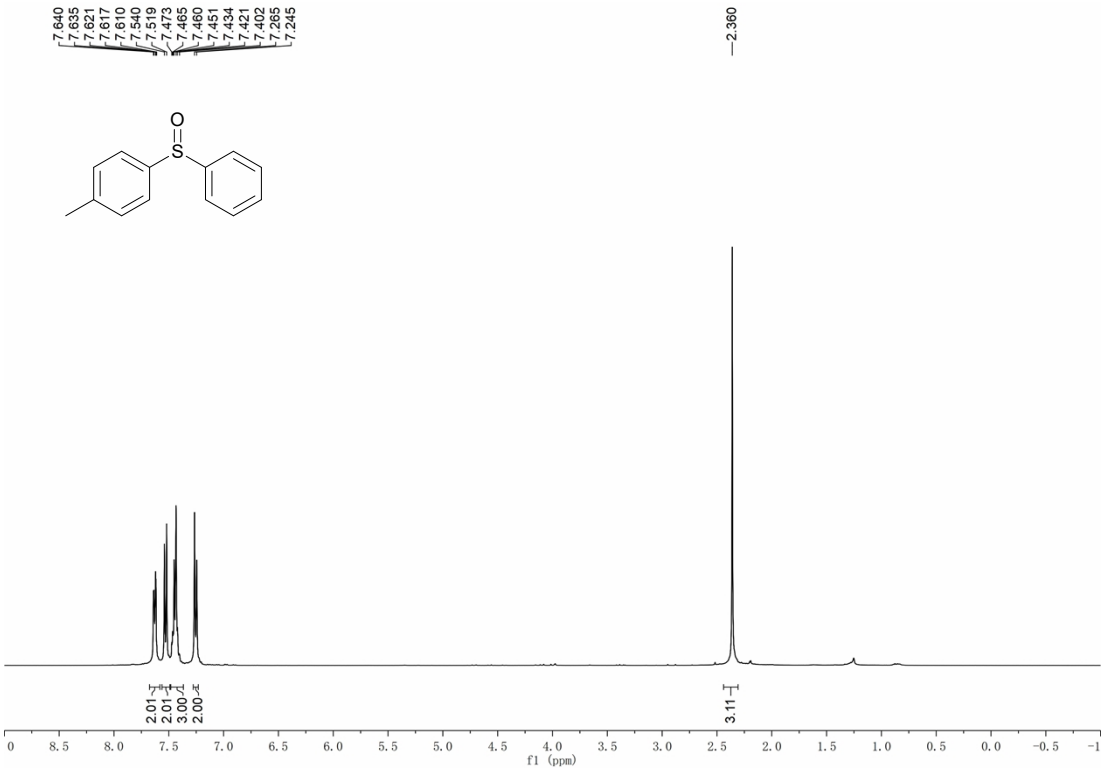
^{1}H NMR (400 MHz, $CDCl_3$) of 2aa



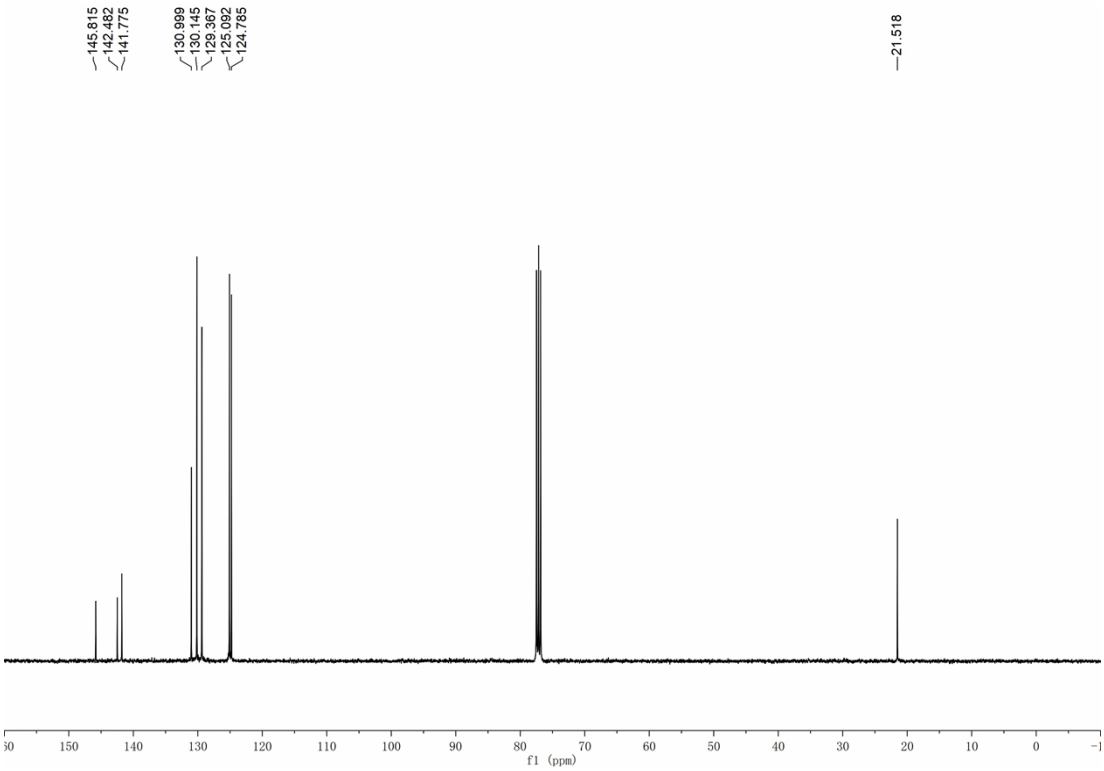
^{13}C NMR (400 MHz, $CDCl_3$) of 2aa



^{1}H NMR (400 MHz, $CDCl_3$) of 2ac



^{13}C NMR (400 MHz, $CDCl_3$) of 2ac



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