# Supporting information

## Highly efficient photocatalytic synthesis of disulfides by a self-assembled $CsPbBr_3/Ti_3C_2T_x$ MXene heterojunction

Yangbo Zhong,[a] Haibo Zhu, [a*] Hongben Zhang,[a] Xinmei Xie,[a] Liu Yang,[a] Bo Wang,[a] Qiangwen Fan,[a] Zongbo Xie[a*] and Zhanggao Le[a*]

[a] Jiangxi Province Key Laboratory of Functional Organic Polymers, East China University of Technology, Nanchang 330013, P. R. China

*Email: hbzhu@ecut.edu.cn, zbxie@ecut.edu.cn, zhgle@ecut.edu.cn

## Table of Contents

1. General information ..... 1

2. Experimental sections ..... 1

2.1 Preparation of $Ti_3C_2T_x$ MXene composite ..... 1

2.2 Preparation of $CsPbBr_3/Ti_3C_2T_x$ MXene composites ..... 1

2.3 Photocatalytic reactions ..... 2

2.4 General Procedure for the recycle experiments ..... 2

2.5 General Procedure for gram-scale synthesis ..... 2

2.6 Optimizing conditions ..... 3

2.7 Characterization of $Ti_3C_2T_x$, $CsPbBr_3$ and $CsPbBr_3$/10-$Ti_3C_2T_x$ ..... 3

2.8 The cyclic voltammetry experiments ..... 6

2.9 Detection of $H_2O_2$ by chemical colorimetric method ..... 8

2.10 Typical reported catalysts ..... 8

3. Spectral Data of compounds ..... 9

4. $^1H$ NMR and Mass spectra for compounds ..... 15

5. References ..... 44

# 1. General information

All commercial reagents were used directly without further purification, unless otherwise stated. $CDCl_3$ were purchased from Shanghai aladdin Biochemical Technology Co. The following abbreviations were used to describe NMR signals: s = singlet, d = doublet, t = triplet, m = mulitplet, dd = doublet of doublets, q = quartet.

Characterization. Transmission electron microscopy (TEM) images were obtained on a JEM F200 microscopy with an accelerating voltage of 200 kV. The energy dispersive X-ray (EDX) composition analysis was carried out using an accessory attached on TEM. Powder X-ray diffraction (XRD) patterns were collected on a Rigaku SmartLab diffractometer using a Cu Kα radiation at a scan rate of $10^{o}$ $min^{-1}$. X-ray photoelectron spectroscopy (XPS) measurements were carried out using an electron spectrometer (ESCALAB250XI, VG, USA). Fourier transform infrared spectra (FTIR) were acquired on a Thermo Nicolet iS5 spectrophotometer with the samples dispersed in KBr. The steady-state photoluminescence (PL) spectra were recorded on a FLS1000 fluorescence spectrometer with an excitation wavelength of 365 nm. UV-vis diffuse reflectance spectra (DRS) of the samples were obtained on a Lambda 1050 spectrometer (TU-1901, CN).

# 2. Experimental sections

### 2.1 Preparation of $Ti_3C_2T_x$ MXene composite

MXene nanosheets were prepared by selective etching of the Al layer from $Ti_3AlC_2$ powders.[1] Firstly, 1 g $Ti_3AlC_2$ powders were gradually added into the mixture solution of 1.6 g LiF powders and 20.0 mL of HCl (9 $mol \cdot L^{-1}$). Secondly, the mixture solution was reacted at 40 ℃ for 48 h with continuous stirring. Thirdly, the reacted mixture solution was centrifuged and washed with deionized water. The centrifuged and washed process was repeated until the pH of the supernatant of the mixture solution was nearly 7.0. Next, the above solution was sonicated in an ice bath for 30 min and then centrifuged. Finally, the MXene nanosheets dispersion solution were obtained by collecting the upper solution.

### 2.2 Preparation of $CsPbBr_3/Ti_3C_2T_x$ MXene composites

Preparation of the ammonium bromide precursor involves combining 10 mL of oleylamine (OAm) and 1.3 mL of hydrobromic acid (HBr) in a 50 mL three-neck round-

bottom flask. The resulting solidified reaction mixture is heated at 120 ℃. It is heated for 2 h under a nitrogen purge. Then, the reaction temperature is increased to 150 ℃ and heated for 30 min. Finally, the solution is maintained at 80 ℃ for further use.

In the second step, 0.2 mmol of cesium carbonate ($Cs_2CO_3$), 50 mg of $Ti_3C_2T_x$ MXene, 0.4 mmol of lead oxide (PbO), along with 20 mL of octadecene (ODE) and 1.5 mL of oleic acid (OA) are placed in a 50 mL three-neck reaction flask. The mixture is purged with nitrogen for 60 min at 120 ℃. After increasing the reaction temperature to 220 ℃, 1.6 mL of the ammonium bromide precursor is immediately injected. After annealing for 6 min, the solution is cooled in an ice-water bath for 30 min. The composite photocatalyst ($CsPbBr_3$/x mg-$Ti_3C_2T_x$, x=10, 20, 30) is collected by centrifugation and washed three times with n-hexane. It is then dried in a vacuum oven for 2 h, ground into a brown powdery solid, and stored in a desiccator for future use.

### 2.3 Photocatalytic reactions

In a 10 mL glass tube, a mixture of 4-methoxyphenyl methanethiol (1a, 0.2 mmol), $CsPbBr_3$/10-$Ti_3C_2T_x$ (10 mg), and in 3.0 mL Ethyl acetate was allowed to stir with irradiation of the 457 nm LED (50 W) in air atmosphere at room temperature for 20 min. Once the reaction is completed, the product is separated using a silica gel plate and the yield is calculated.

### 2.4 General Procedure for the recycle experiments

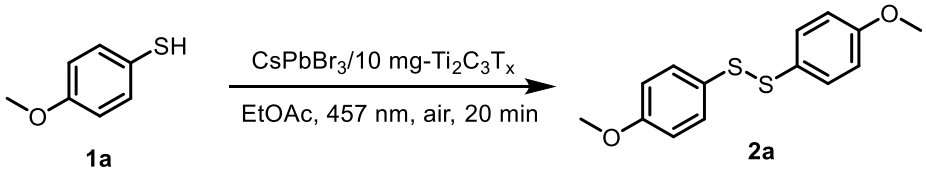
In a 10 mL glass tube, a mixture of 4-methoxyphenyl methanethiol (1a, 0.2 mmol), $CsPbBr_3$/10-$Ti_3C_2T_x$ (10 mg), and in 3.0 mL Ethyl acetate was allowed to stir with irradiation of the 457 nm LED (50 W) in air atmosphere at room temperature for 20 min. After the reaction, the $CsPbBr_3$/10-$Ti_3C_2T_x$ in the reaction was filtrated and washed with n-hexane. Subsequently, the composite was dried under 60 ℃ in the vacuum oven, and the dried catalyst was directly used in the next experiment without any further purification. Individual yields of product 3a have been given.

### 2.5 General Procedure for gram-scale synthesis

The mixture of 4-methoxyphenyl methanethiol (8 mmol), $CsPbBr_3$/10-$Ti_3C_2T_x$ (400 mg), and ethyl acetate (120 mL) were sequentially added in a 250 mL round bottom flask, then the reaction was carried out in air under 457 nm LED (50 W) for 1 h.

## 2.6 Optimizing conditions

**Table S1.** Optimization of reaction conditions




| Entry | Variation from standard conditions | Yield (%) |
|---|---|---|
| 1 | None | 99 |
| 2 | $CsPbBr_3$ | 36 |
| 3 | $Ti_3C_2T_x$ | 21 |
| 4 | $CsPbBr_3$/20mg-$Ti_3C_2T_x$ | 95 |
| 5 | $CsPbBr_3$/30mg-$Ti_3C_2T_x$ | 83 |
| 6 | without catalyst | 16 |
| 7 | without light | Trace |
| 8 | $N_2$ instead of air | 19 |
| 9 | DCM | 71 |
| 10 | DCE | 62 |
| 11 | THF | 83 |
| 12 | MeCN | 80 |

[a]Reaction condition: **1a** (0.2 mmol), Cat. (10 mg) in EtOAc (3.0 mL) under 457 nm LEDs for 20 min. [b]Isolated yields.

## 2.7 Characterization of $Ti_3C_2T_x$, $CsPbBr_3$ and $CsPbBr_3$/10-$Ti_3C_2T_x$

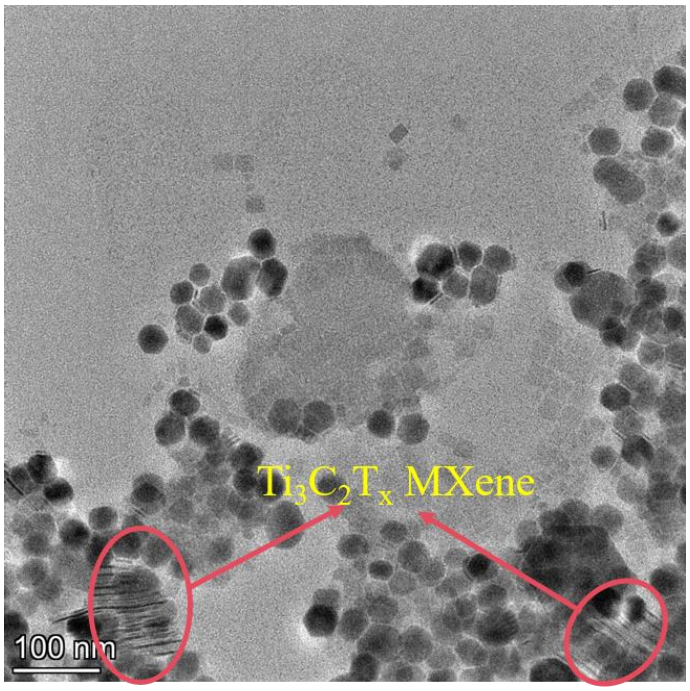



**Figure S1.**TEM of $CsPbBr_3$/10-$Ti_3C_2T_x$ nanocomposites

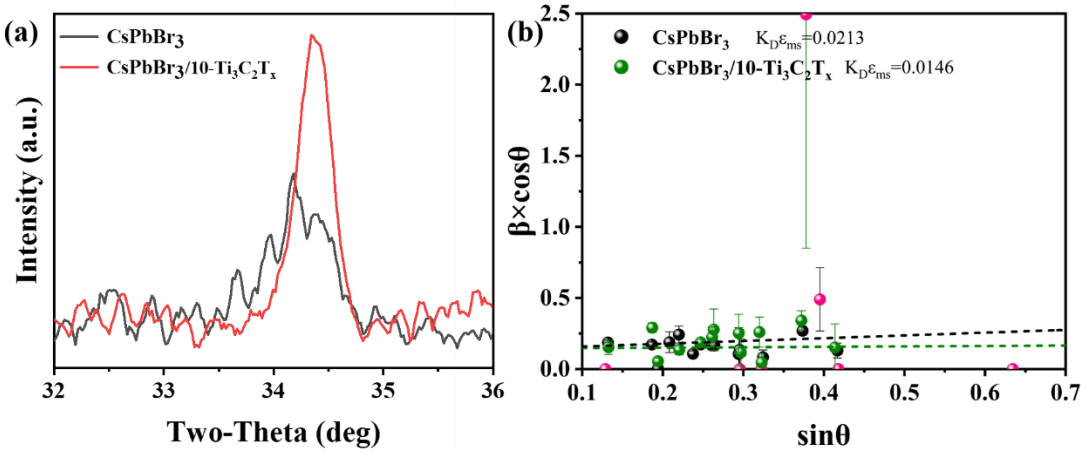



**Figure S2.** (a) amplified XRD patterns of the (120) lattice plane. (b) Williamson−Hall plots of $CsPbBr_3$ NCs and $CsPbBr_3$/10-$Ti_3C_2T_x$ nanocomposites.

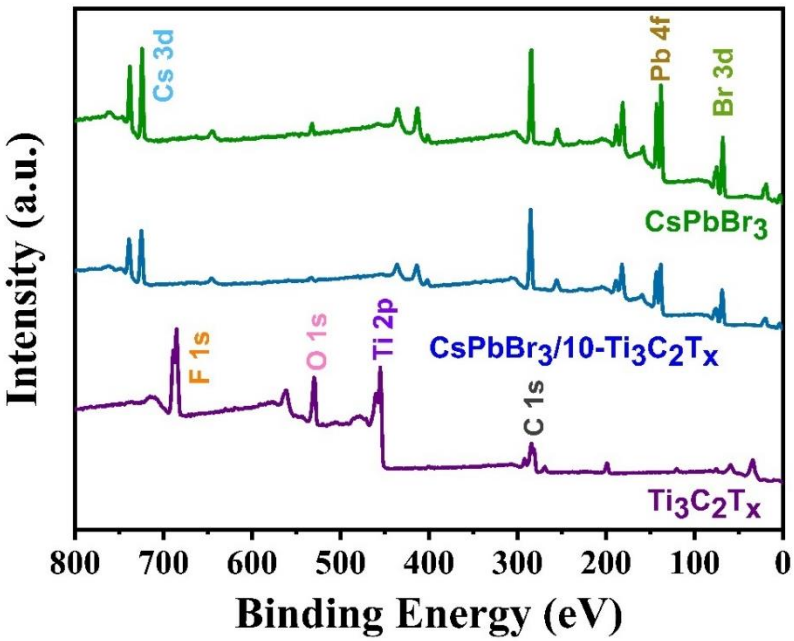



**Figure S3.** XPS spectra of $Ti_3C_2T_x$ nanosheets, $CsPbBr_3$ NCs, $CsPbBr_3$/10-$Ti_3C_2T_x$ nanocomposites

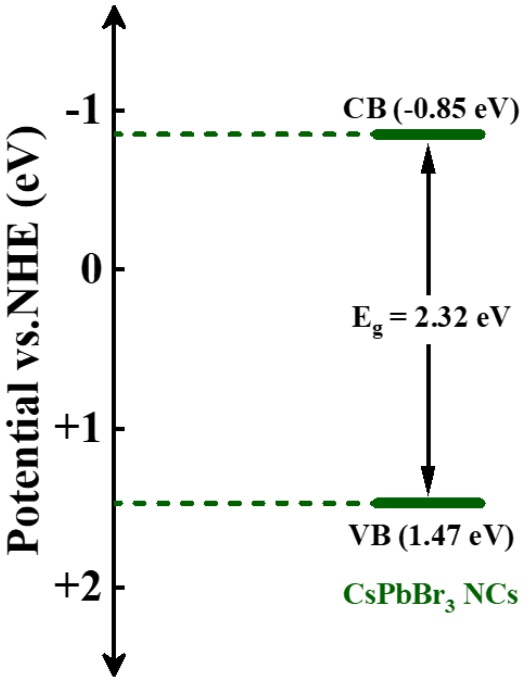



**Figure S4.** Band-edge alignment of $CsPbBr_3$ NCs

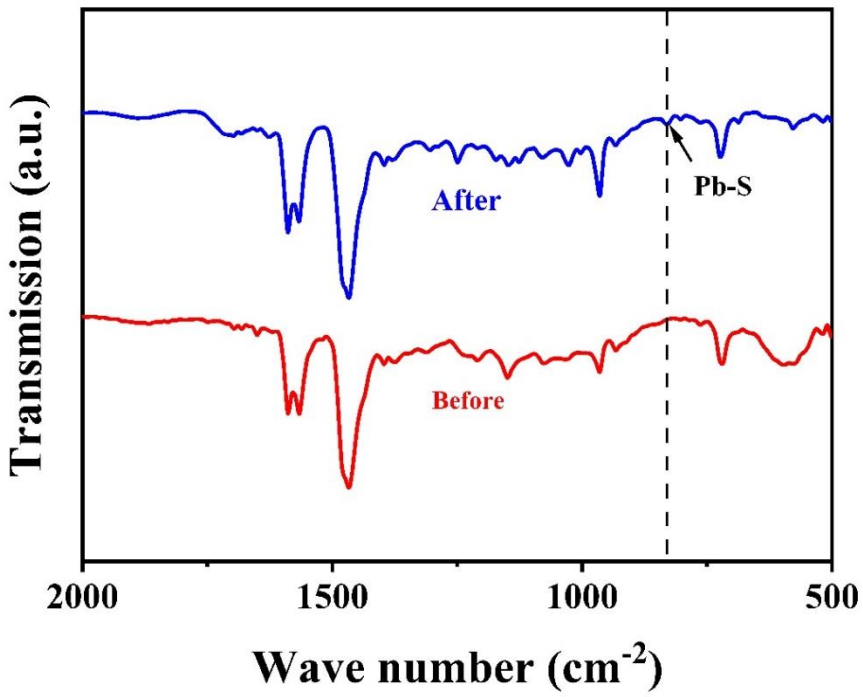



**Figure S5.** FTIR Spectra of $CsPbBr_3$/10-$Ti_3C_2T_x$ nanocomposites before and after cycling.

**Table S2.** Ti, Cs and Pb contents of the as-prepared $CsPbBr_3$/10-$Ti_3C_2T_x$ obtained by ICP-MS and calculations.

$$Cx(ug/kg) = \frac{C_0(ug/L) * f * V_0(mL) * 10^{-3}}{m(g) * 10^{-3}} = \frac{C_1(ug/L) * V_0(mL) * 10^{-3}}{m(g) * 10^{-3}}$$

$$W(\%) = \frac{x(ug/kg)}{10^9} * 100\%$$

| Element | Sample $m_o$ (g) | Vo (mL) | $C_o$ (ug/L) | Dilution ratio f | $C_1$ (ug/L) | $C_x$ (ug/kg) | W (%) |
|---|---|---|---|---|---|---|---|
| Ti | 0.0447 | 25 | 52.938 | 1000 | 52938.480 | 29607651.007 | 2.96% |
| Cs | 0.0447 | 25 | 329.519 | 1000 | 329518.770 | 184294614.094 | 18.43% |
| Pb | 0.0447 | 25 | 298.181 | 1000 | 298181.150 | 166767980.984 | 16.68% |

**Table S3.** Apparent quantum yield of the $Ti_3C_2T_x$**,** $CsPbBr_3$ and $CsPbBr_3$/10-$Ti_3C_2T_x$.

$$AQY = \frac{N_e}{N_p} \times 100\%$$

$$= \frac{10^9(v \times N_A \times K) \times (h \times c)}{(I \times A \times \lambda)} \times 100\%$$

$$= \frac{1.2 \times 10^8(v \times K)}{(I \times A \times \lambda)} \times 100\%$$

$$= \frac{1.2 \times 10^8\left(\frac{n \times Yield}{Time} \times K\right)}{(I \times A \times \lambda)} \times 100\%$$

| Photocatalyst | Yield (%) | I ($W \cdot m^{-2}$) | A ($m^2$) | λ (nm) | AQY (%) |
|---|---|---|---|---|---|
| $Ti_3C_2T_x$ | 21 | 445 | $2.6\times10^{-4}$ | 457 | 7.94 |
| $CsPbBr_3$ | 36 | 445 | $2.6\times10^{-4}$ | 457 | 13.62 |
| $CsPbBr_3$/10-$Ti_3C_2T_x$ | 99 | 445 | $2.6\times10^{-4}$ | 457 | 37.45 |

## 2.8 The cyclic voltammetry experiments

Cyclic voltammetric investigations were performed on the CHI-660E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., China) with the conventional three electrode system. The solvent exhaust employs a nitrogen blast for 10 min. A Pt electrode (Φ3 mm) was used as the working electrode; a saturated calomel electrode (SCE) and Pt column (Φ1 mm x 5 mm) were used as the reference electrode and counter

electrode, respectively. **1a** (0.1 M) were tested with tetrabutylammonium tetrafluoroborate (0.1 M) as the supporting electrolyte, respectively. The scan direction was reversed, the starting point was -2.0 V (initial potential: -2.0 V), and the scan rate was 100 mV/s. The test temperature is room temperature. The measured oxidation potential was **1a** ($E_{1/2}^{Ox}$= 1.47 V *vs*. SCE; $E_{1/2}^{Red}$= -1.75 V *vs*. SCE).

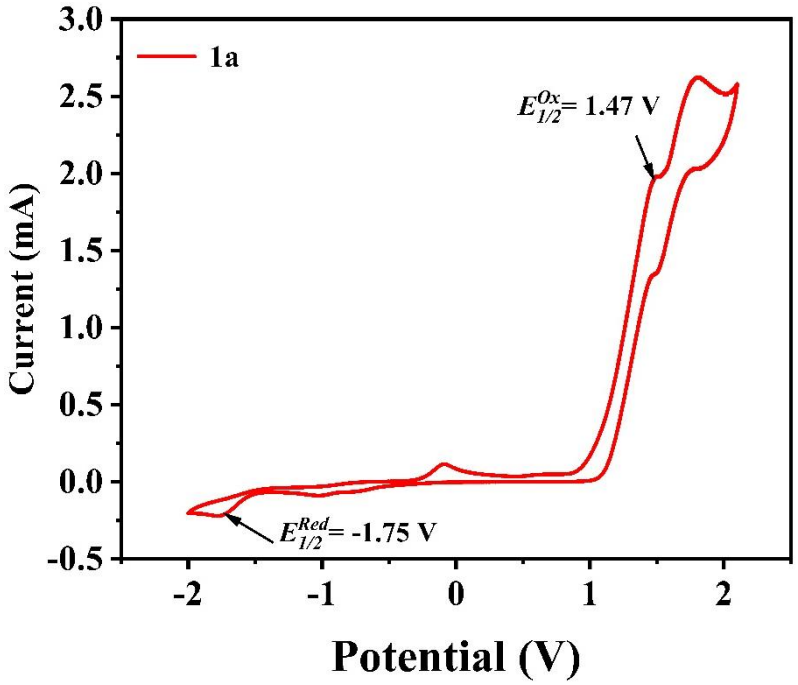



**Figure S6.** The cyclic voltammetry.

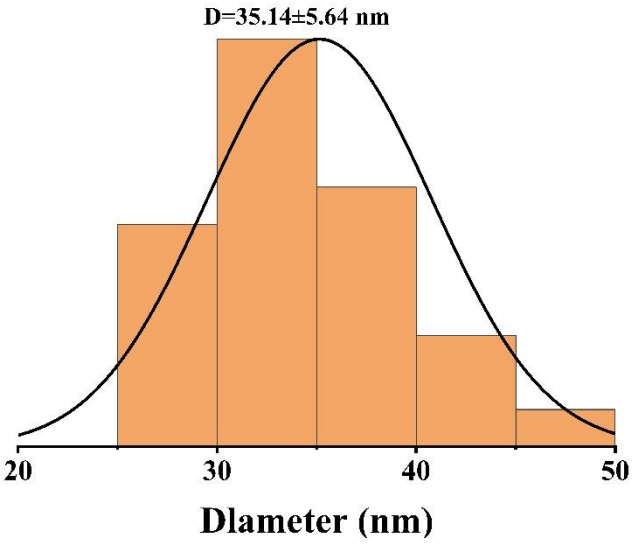



**Figure S7.** Particle size distribution diagram of $CsPbBr_3$/10-$Ti_3C_2T_x$ nanocomposites.

## 2.9 Detection of $H_2O_2$ by chemical colorimetric method

Potassium titanium oxalate was used as a chromogenic agent to detect $H_2O_2$ in the reaction system. Detailed procedures: upon the complete conversion of 1a, transfer 1.0 mL of the reaction solution by a pipette to a centrifugal tube, and then filtered by a syringe equipment with a 0.45 μM filter. Subsequently, it was mixed with 1.0 mL of potassium titanium oxalate (TPO) solution (0.2 M). The detection of $H_2O_2$ was conducted on a UV-visible spectrophotometer. As shown in Figure S3, compared with the pure reaction solution and TPO, an apparent absorption peak with a value of 0.215 was observed at around 390 nm, which clearly confirmed the formation of $H_2O_2$ in the reaction. By measuring the absorbance of $H_2O_2$ at standard concentrations, a standard curve is obtained. Substituting the absorbance of the reaction solution into this curve yields a concentration of 0.8 mM.

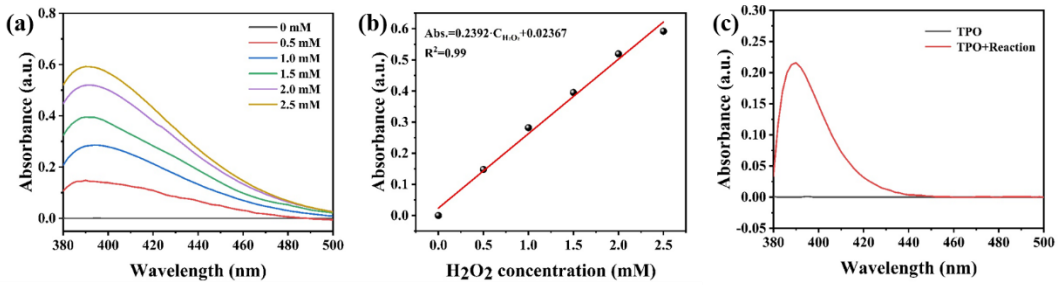



**Figure S8.** (a) The absorbance spectrum for $H_2O_2$ quantification, (b) The Linear fitting of $H_2O_2$ concentration and absorbance, (c) UV-Vis spectroscopy for detection of hydrogen peroxide in a reaction system.

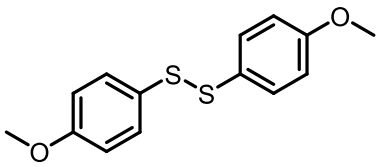
## 2.10 Typical reported catalysts

**Table S4.** Comparison results with some typical reported catalysts

| Materials | Light source | Gas atmosphere | Production rate (*μmol g-1 h-1*) | Ref. |
|---|---|---|---|---|
| $CsPbBr_3$ | 27 W white LED | Air | 6693 | 2 |
| g-$C_3N_4$/$Bi_2WO_6$ | 18 W blue LED | Air | 1078 | 3 |
| anatase $TiO_2$ | 520 nm green LEDs (3 W × 4) | $O_2$ | 21840 | 4 |
| GR-CdS-15% (Co-Pi) | 300 W Xe lamp | Ar | 912 | 5 |

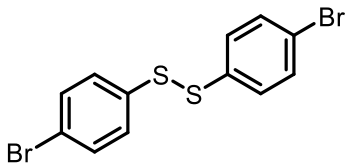
| | | | | |
|---|---|---|---|---|
| MFC-CMP | 460 ± 10 nm blue LEDs (3 W × 4) | $O_2$ | 165600 | 6 |
| $CsPbBr_3$/10-$Ti_3C_2T_x$ | 50 W blue LED | Air | 29700 | This work |

# 3. Spectral Data of compounds



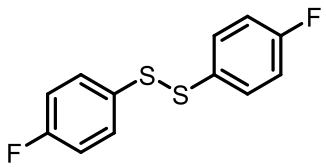
**1,2-bis(4-methoxyphenyl)disulfane**

3**a** (99%, 27.5 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 8.7 Hz, 4H), 6.82 (d, *J* = 8.7 Hz, 4H), 3.77 (s, 6H); MS (m/z) = 278 (M).



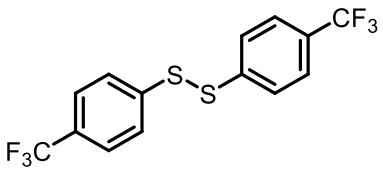
**1,2-bis(4-bromophenyl)disulfane**

3**b** (91%, 34.2 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^1$H NMR (500 MHz, Chloroform-d) δ 7.42 (d, J = 8.4 Hz, 4H), 7.33 (d, J = 8.5 Hz, 4H); MS (m/z) = 376 (M).



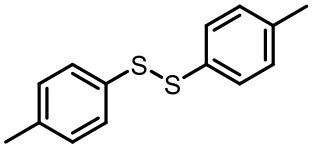
**1,2-bis(4-fluorophenyl)disulfane**

3**c** (91%, 23.1 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.52 – 7.39 (m, 4H), 7.00 (t, *J* = 8.6 Hz, 4H); MS (m/z) = 254 (M).



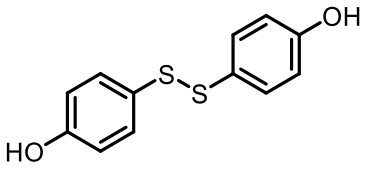

**1,2-bis(4-(trifluoromethyl)phenyl)disulfane**

3**d** (84%, 29.9 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.57 (s, 8H); MS (m/z) = 354 (M).



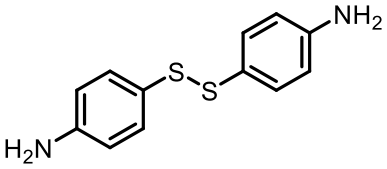

**1,2-di-*p*-tolyldisulfane**

3**e** (80%, 19.6 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.2 Hz, 4H), 7.10 (d, *J* = 8.0 Hz, 4H), 2.31 (s, 6H); MS (m/z) = 246 (M).



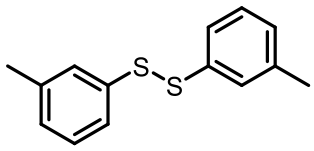

**4,4'-disulfanediyldiphenol**

3**f** (94%, 23.4 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 2:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.4 Hz, 4H), 6.77 (d, *J* = 8.4 Hz, 4H), 5.09 (s, 2H); MS (m/z) = 250 (M).



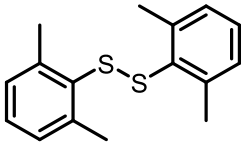

**4,4'-disulfanediyldianiline**

3**g** (97%, 24.1 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 2:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 8.3 Hz, 4H), 6.57 (d, *J* = 8.3 Hz, 4H), 3.75 (s, 4H); MS (m/z) = 248 (M).



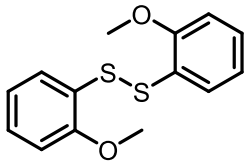
**1,2-di-*m*-tolyldisulfane**

3**h** (82%, 20.2mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 7.2 Hz, 4H), 7.18 (t, *J* = 7.9 Hz, 2H), 7.02 (d, *J* = 7.3 Hz, 2H), 2.31 (s, 6H); MS (m/z) = 246 (M).



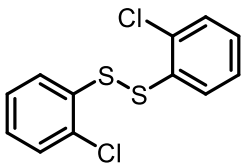
**1,2-bis(2,6-dimethylphenyl)disulfane**

3**i** (93%, 25.4 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.10 (t, *J* = 7.5 Hz, 2H), 7.00 (d, *J* = 7.5 Hz, 4H), 2.23 (s, 12H); MS (m/z) = 274 (M).



**1,2-bis(2-methoxyphenyl)disulfane**

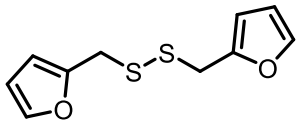
3**j** (99%, 27.4 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 8.7 Hz, 2H), 7.18 (t, *J* = 7.8 Hz, 2H), 6.91 (t, *J* = 7.6 Hz, 2H), 6.85 (d, *J* = 8.1 Hz, 2H), 3.89 (s, 6H); MS (m/z) = 278 (M).



**1,2-bis(2-chlorophenyl)disulfane**

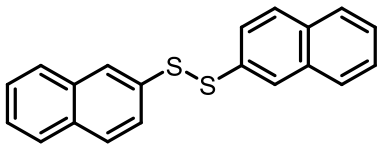
3**k** (95%, 27.2 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 7.9 Hz, 2H),

7.36 (d, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.6 Hz, 2H), 7.15 (t, *J* = 7.6 Hz, 2H); MS (m/z) = 287 (M).



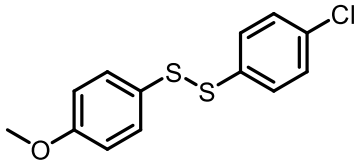
**1,2-bis(furan-2-ylmethyl)disulfane**

3**l** (79%, 17.8 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 2.0 Hz, 1H), 6.34 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.23 (d, *J* = 3.1 Hz, 1H), 3.68 (s, 2H); MS (m/z) = 226 (M).



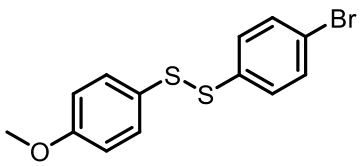
**1,2-di(naphthalen-2-yl)disulfane**

3**m** (64%, 20.4 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 8.00 – 7.95 (m, 2H), 7.77 (dd, *J* = 9.1, 3.5 Hz, 4H), 7.74 – 7.69 (m, 2H), 7.61 (dd, *J* = 8.7, 2.0 Hz, 2H), 7.47 – 7.41 (m, 4H); MS (m/z) = 318 (M).



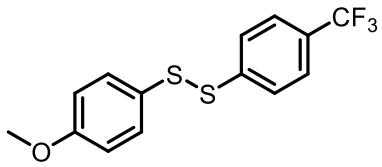
**1-(4-chlorophenyl)-2-(4-methoxyphenyl)disulfane**

3**n** (63%, 17.8 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.41 (dd, *J* = 12.7, 8.6 Hz, 4H), 7.27 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H); MS (m/z) = 282 (M).



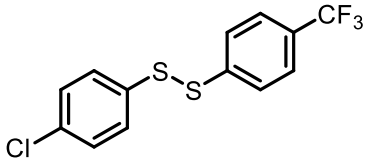
**1-(4-bromophenyl)-2-(4-methoxyphenyl)disulfane**

3**o** (59%, 19.1 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.38 (ddt, *J* = 23.2, 15.4, 8.0 Hz, 6H), 6.83 (d, *J* = 7.4 Hz, 2H), 3.79 (s, 3H); MS (m/z) = 328 (M).



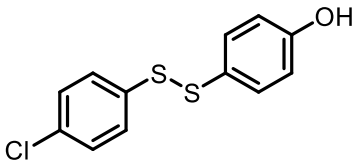

**1-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)disulfane**

3**p** (67%, 21.2 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.62 (d, *J* = 8.2 Hz, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 3.79 (s, 3H); MS (m/z) = 316 (M).



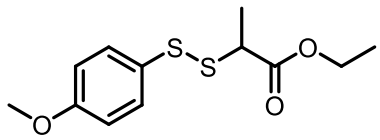

**1-(4-chlorophenyl)-2-(4-(trifluoromethyl)phenyl)disulfane**

3**q** (89%, 28.3 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.61 – 7.52 (m, 2H), 7.40 (dd, *J* = 8.7, 2.8 Hz, 3H), 7.31 – 7.25 (m, 3H); MS (m/z) = 320 (M).



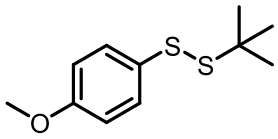

**4-((4-chlorophenyl)disulfaneyl)phenol**

3**r** (51%, 13.6 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.34 (t, *J* = 9.4 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.6 Hz, 2H), 6.70 (d, *J* = 8.2 Hz, 2H), 3.96 (s, 1H); MS (m/z) = 268 (M).



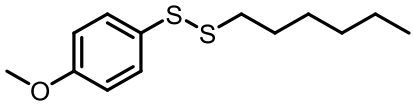
**ethyl 2-((4-methoxyphenyl)disulfaneyl)propanoate**

3**s** (89%, 24.2 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.1 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 4.21 (q, *J* = 7.0 Hz, 1H), 4.06 – 3.91 (m, 2H), 3.80 (s, 3H), 1.49 (d, *J* = 8.8 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H); MS (m/z) = 272 (M).



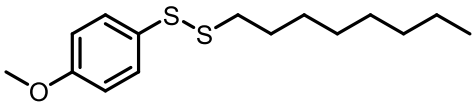
**1-(*tert*-butyl)-2-(4-methoxyphenyl)disulfane**

3**t** (72%, 16.4 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 3.79 (s, 3H), 1.29 (s, 9H); MS (m/z) = 228 (M).



**1-hexyl-2-(4-methoxyphenyl)disulfane**

3**u** (50%, 12.7 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 3.80 (s, 3H), 2.72 (t, *J* = 7.3 Hz, 2H), 1.67 (d, *J* = 7.4 Hz, 2H), 1.34 (p, *J* = 7.5 Hz, 2H), 1.29 – 1.22 (m, 4H), 0.87 (t, *J* = 6.8 Hz, 3H); MS (m/z) = 256 (M).



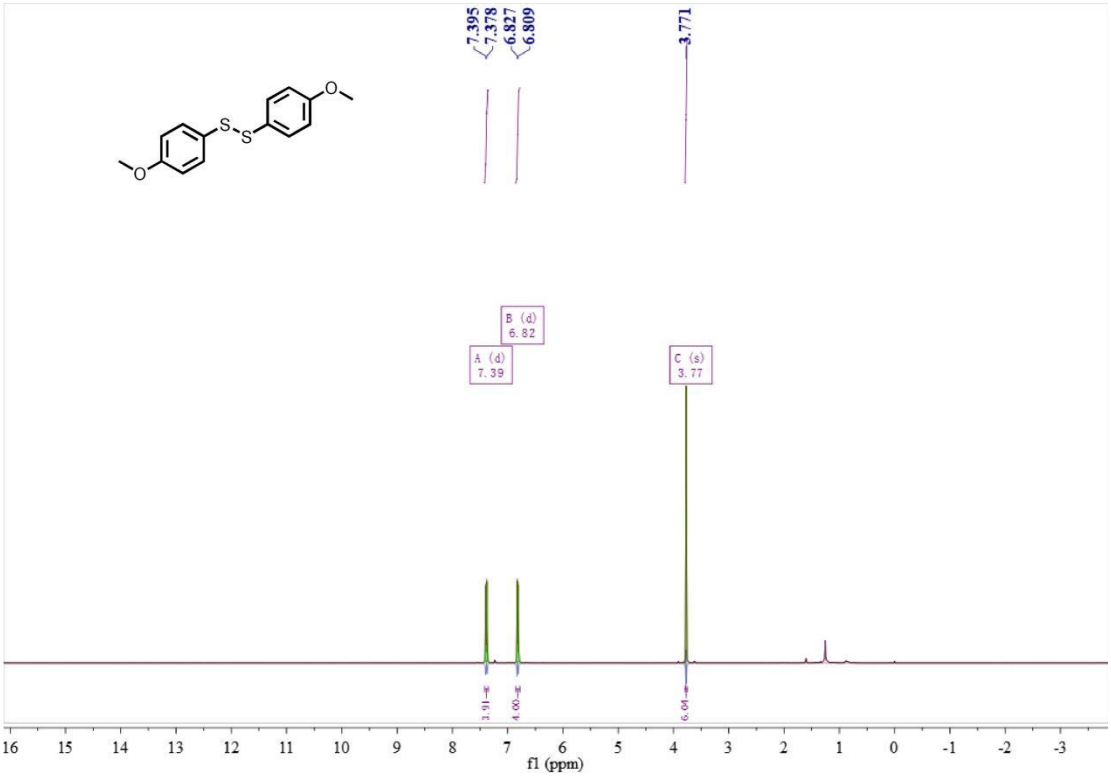
**1-(4-methoxyphenyl)-2-octyldisulfane**

3**v** (62%, 17.5 mg): Obtained through thin-layer chromatography (petroleum ether/ethyl acetate 15:1) $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 3.80 (s, 3H), 2.72 (t, *J* = 7.3 Hz, 2H), 1.66 (p, *J* = 7.4 Hz, 2H),

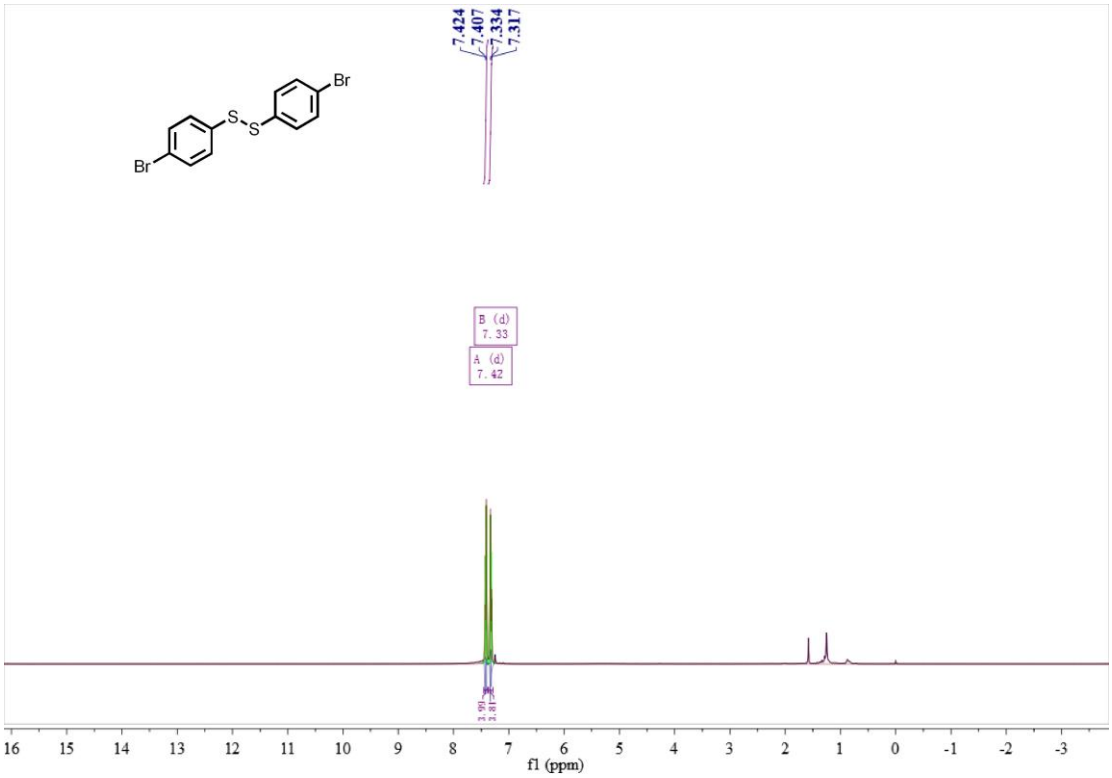
1.38 – 1.31 (m, 2H), 1.26 (d, *J* = 15.2 Hz, 8H), 0.88 (t, *J* = 6.9 Hz, 3H); MS (m/z) = 284 (M).

# 4. $^{1}$H NMR and Mass spectra for compounds

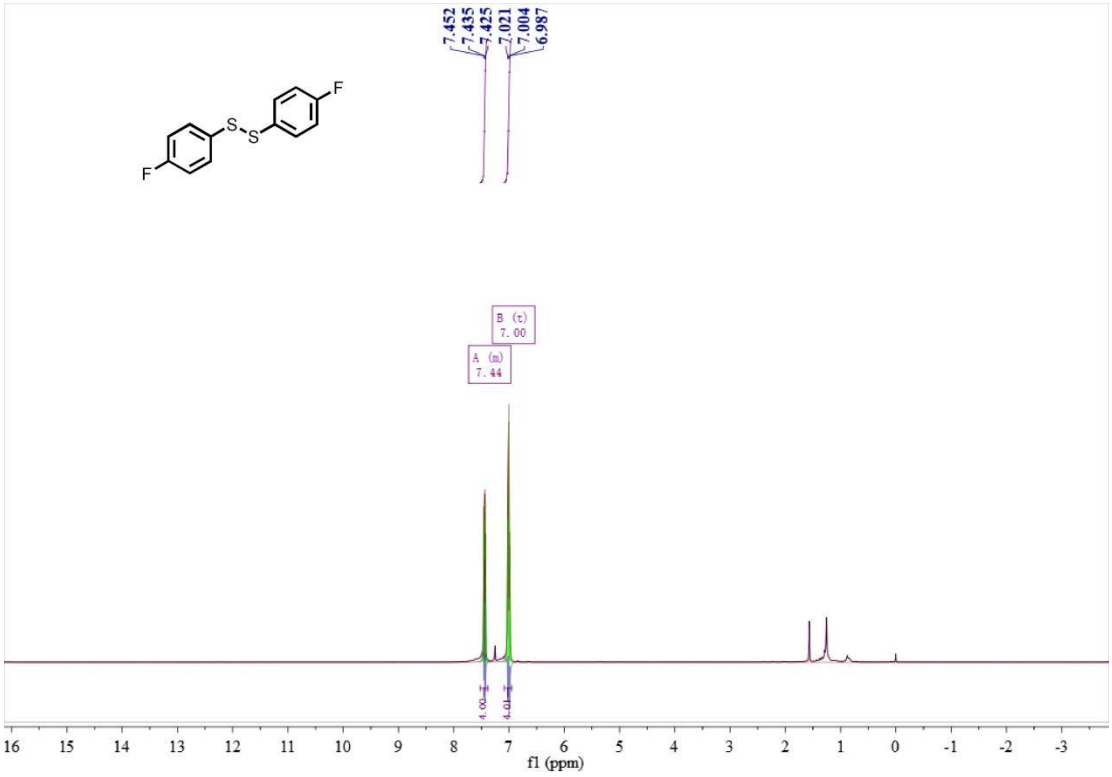
$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(4-methoxyphenyl)disulfane (3a)** in $CDCl_3$



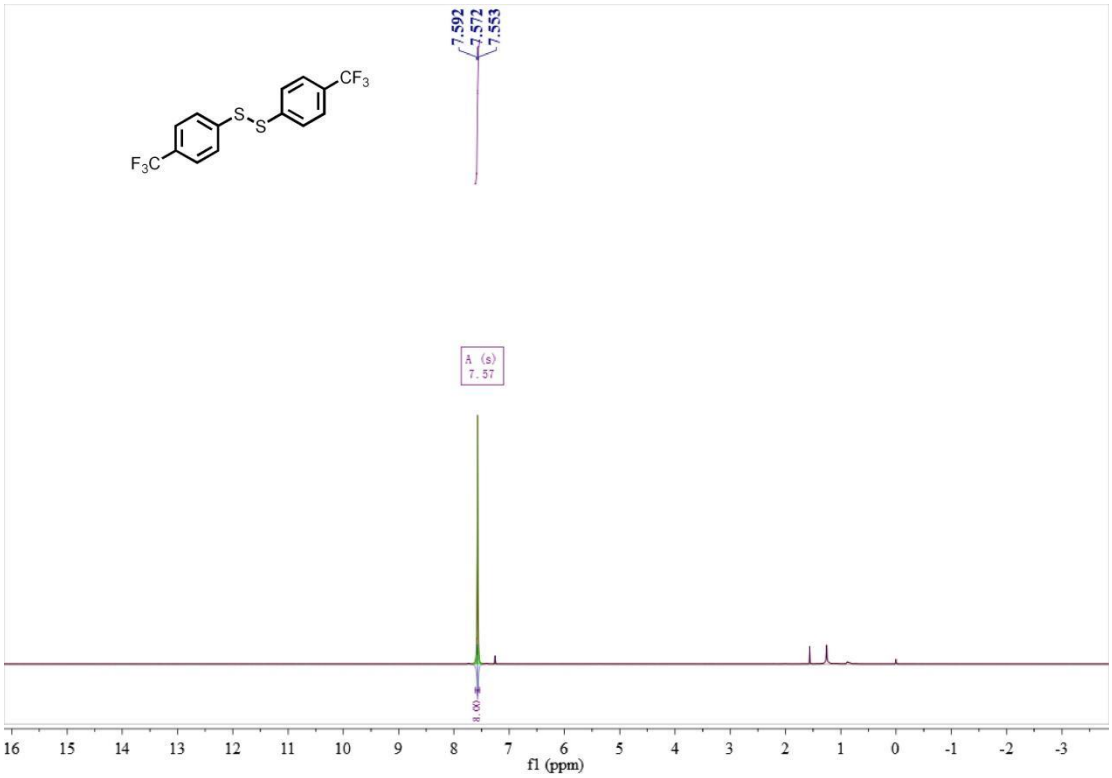

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(4-bromophenyl)disulfane (3b)** in $CDCl_3$



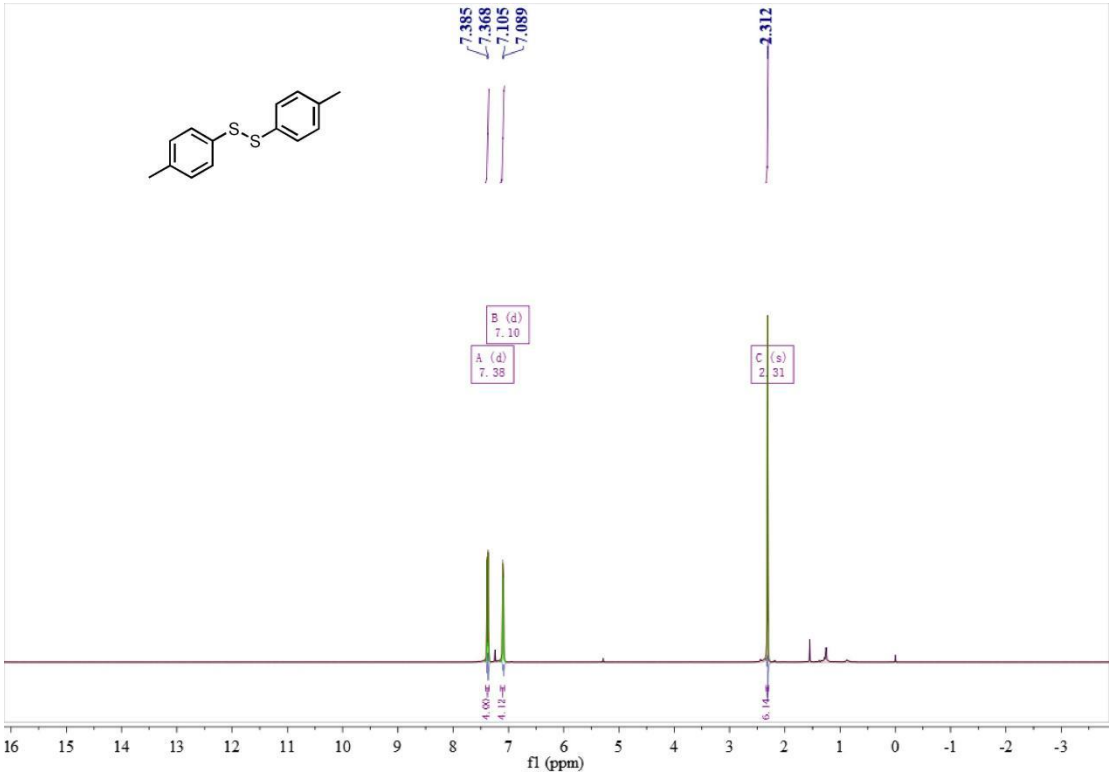

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(4-(trifluoromethyl)phenyl)disulfane (3c)** in $CDCl_3$



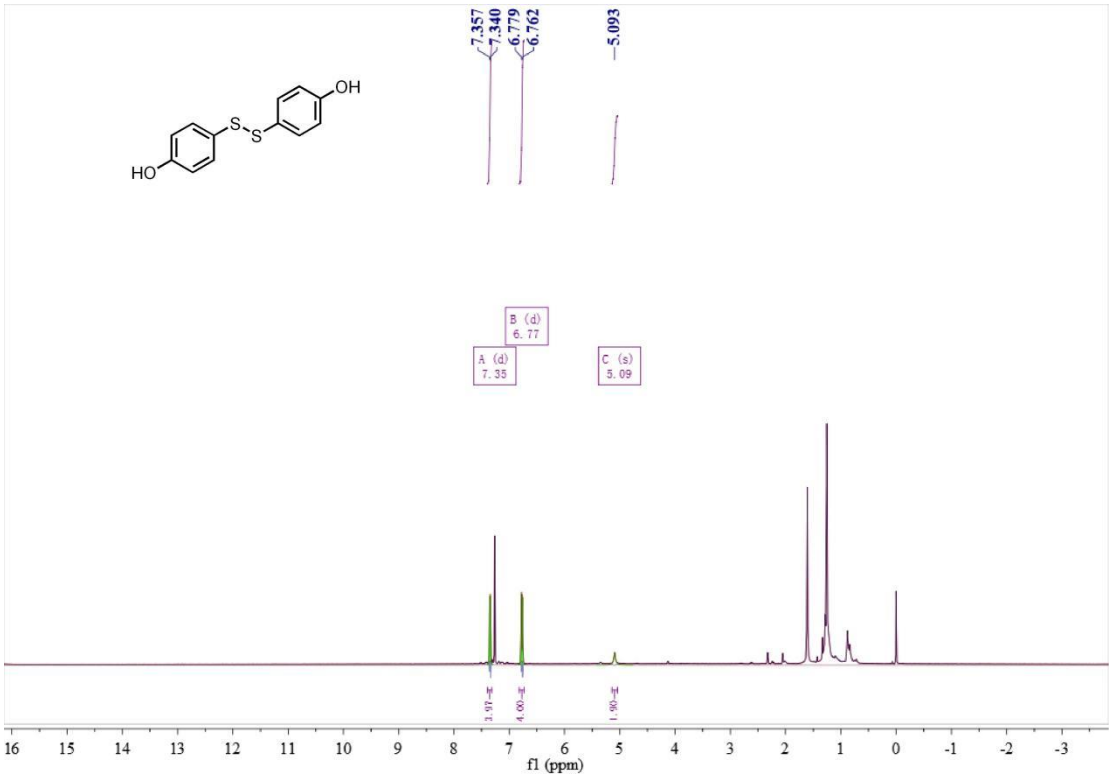

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(4-(trifluoromethyl)phenyl)disulfane (3d)** in $CDCl_3$



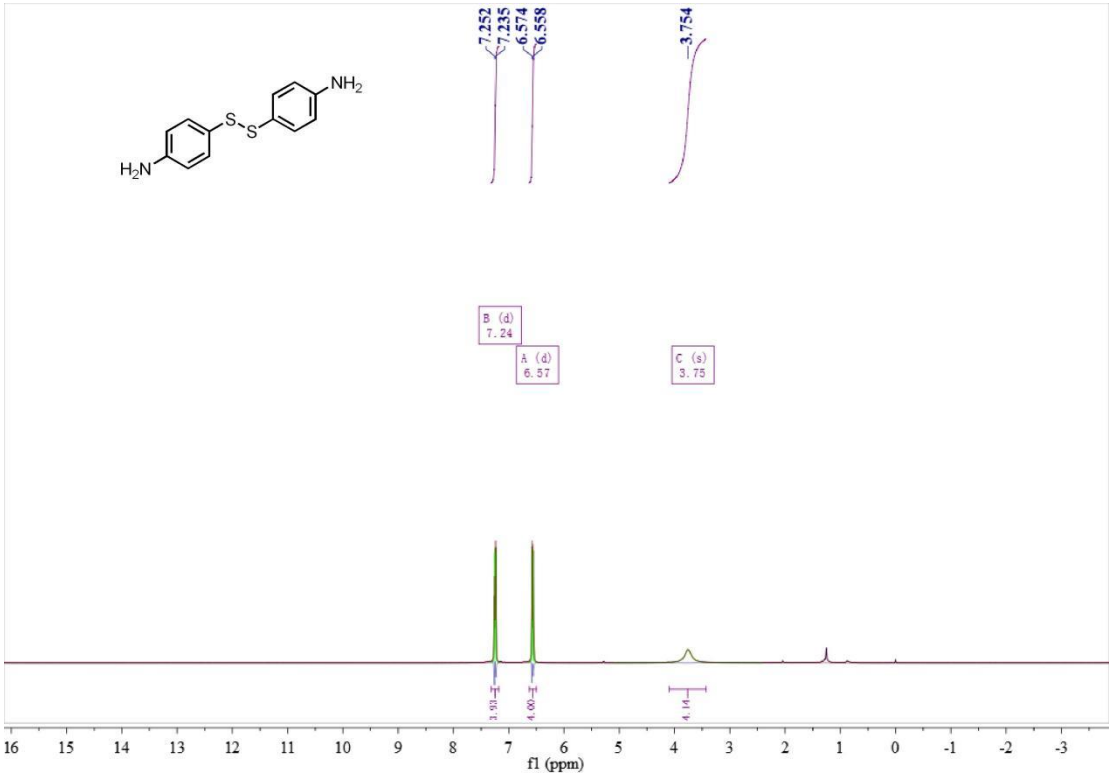

$^1$H NMR (500MHz) Spectrum of **1,2-di-p-tolyldisulfane (3e)** in $CDCl_3$



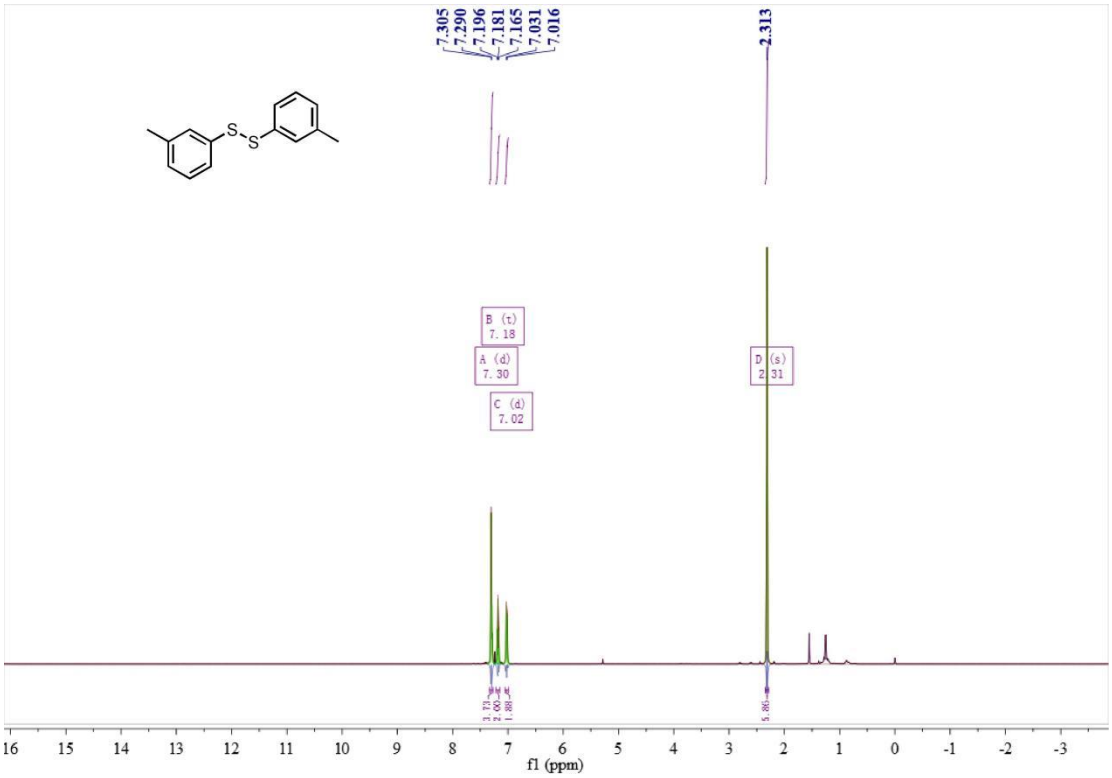

$^1$H NMR (500MHz) Spectrum of **4,4'-disulfanediyldiphenol (3f)** in $CDCl_3$



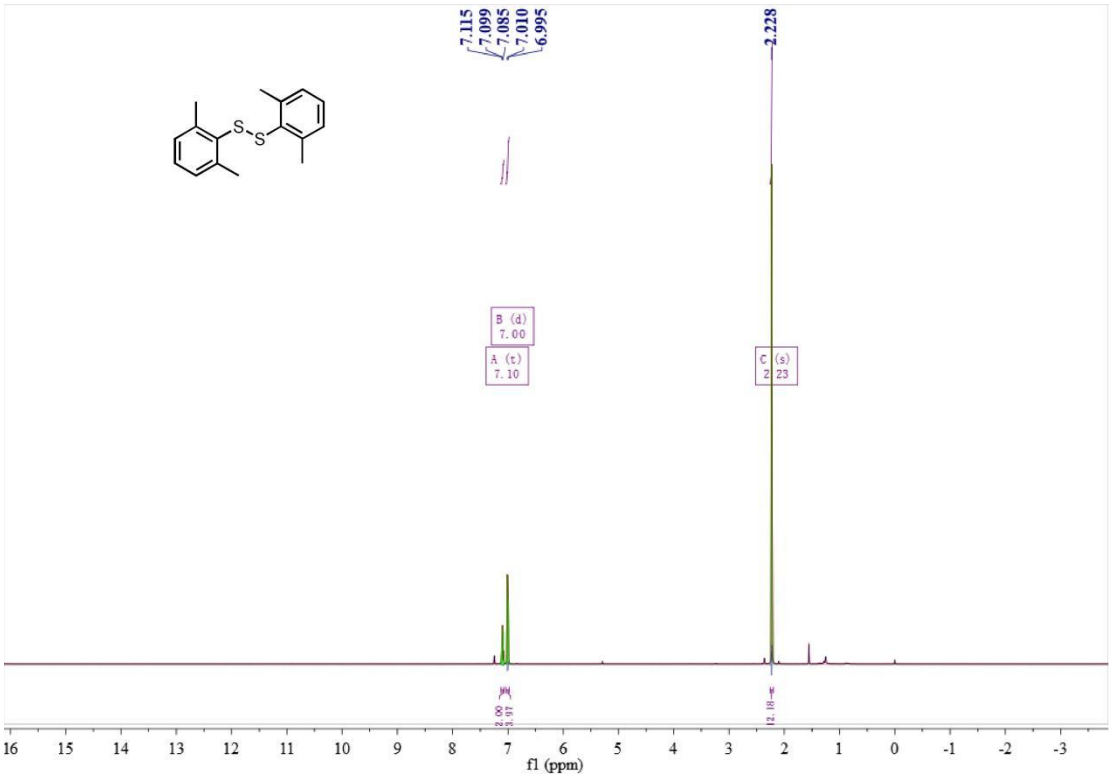

$^{1}$H NMR (500MHz) Spectrum of **4,4'-disulfanediyldianiline (3g)** in $CDCl_3$



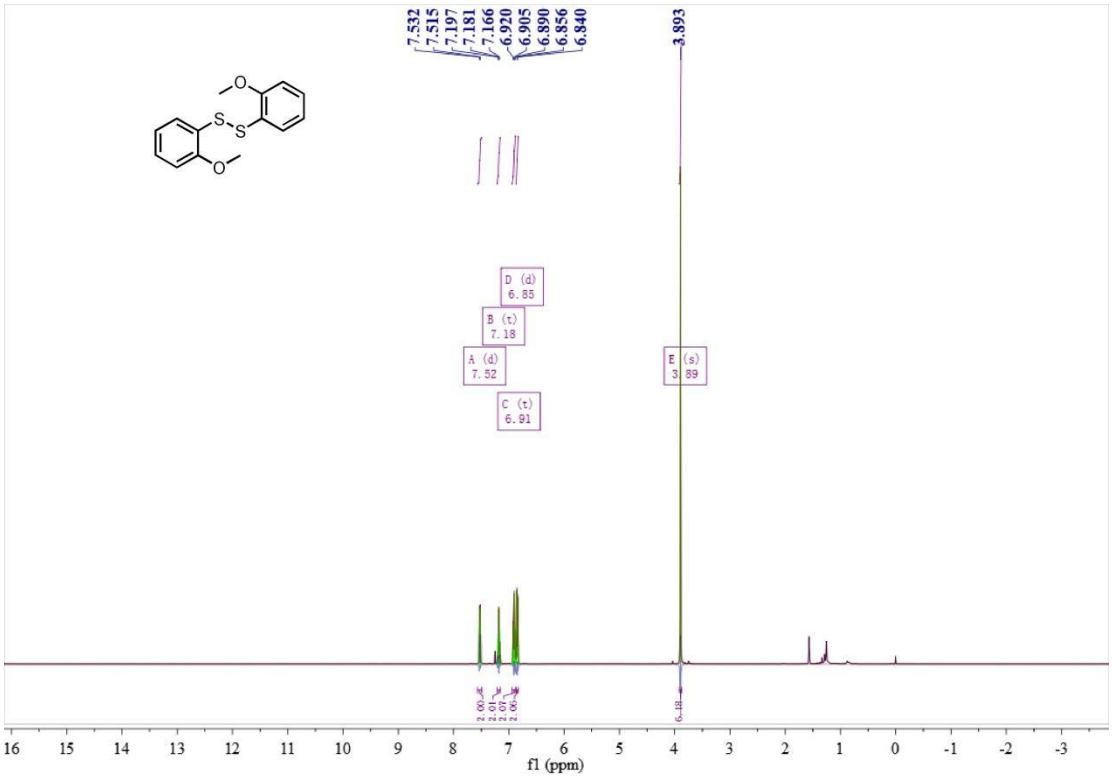

$^{1}$H NMR (500MHz) Spectrum of **1,2-di-m-tolyldisulfane (3h)** in $CDCl_3$



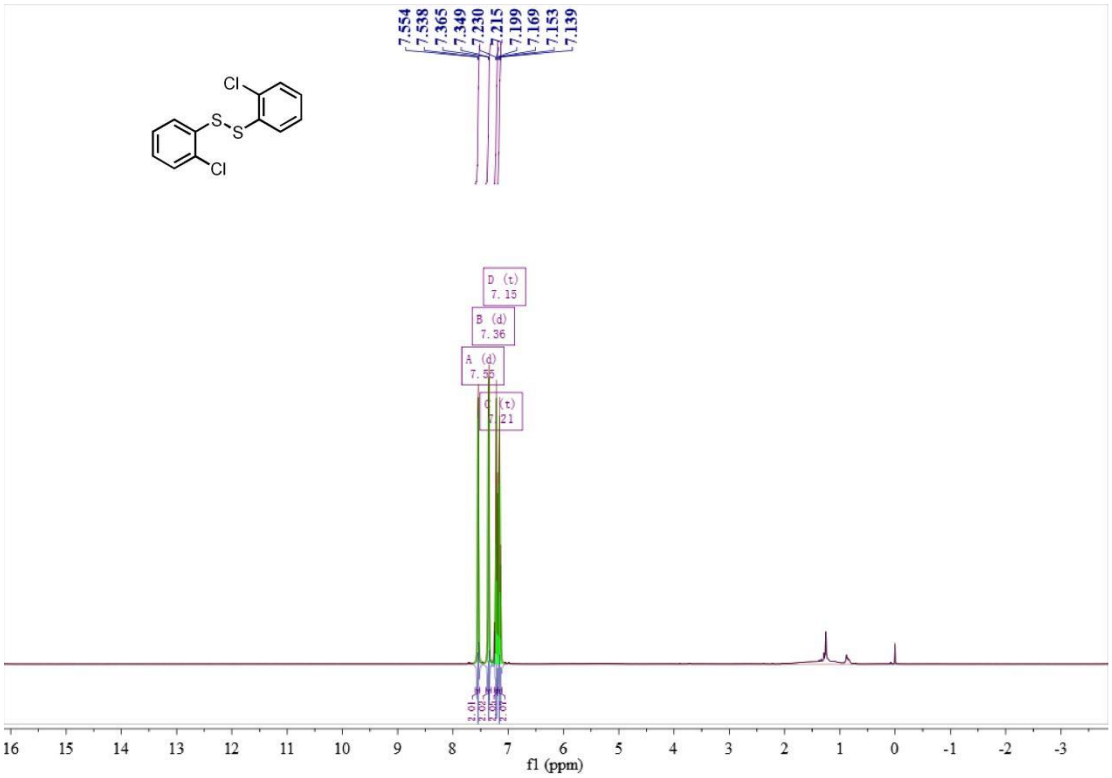

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(2,6-dimethylphenyl)disulfane (3i)** in $CDCl_3$



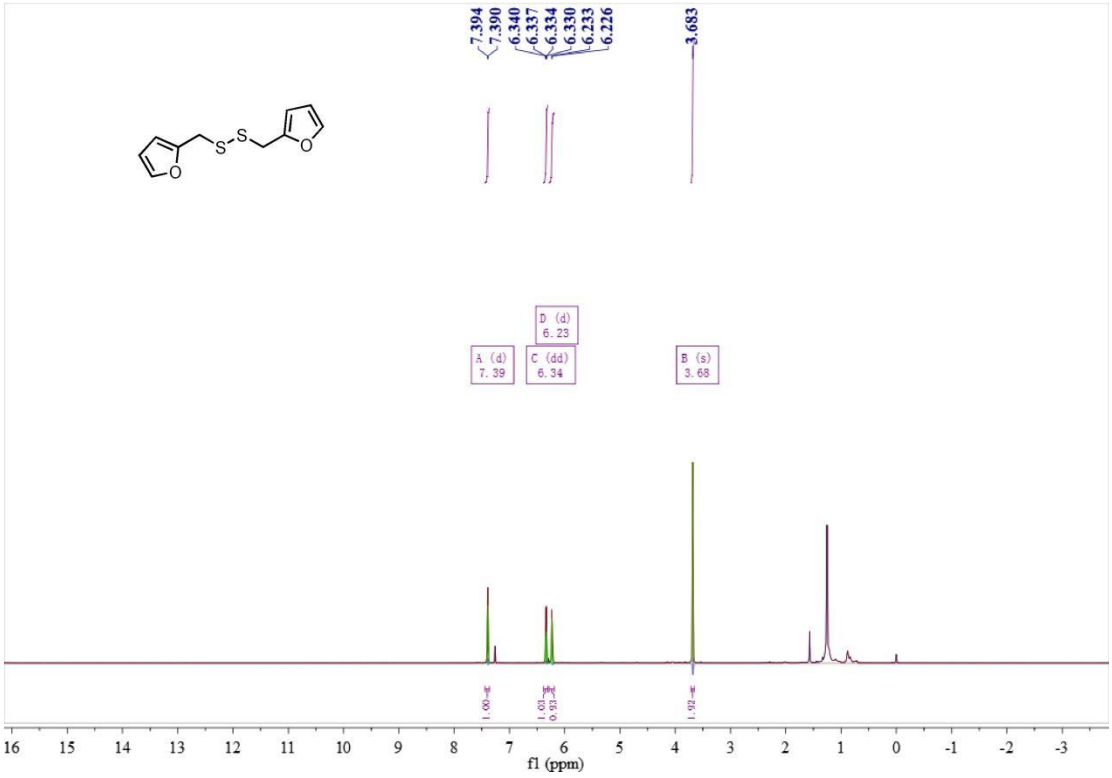

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(2-methoxyphenyl)disulfane (3j)** in $CDCl_3$



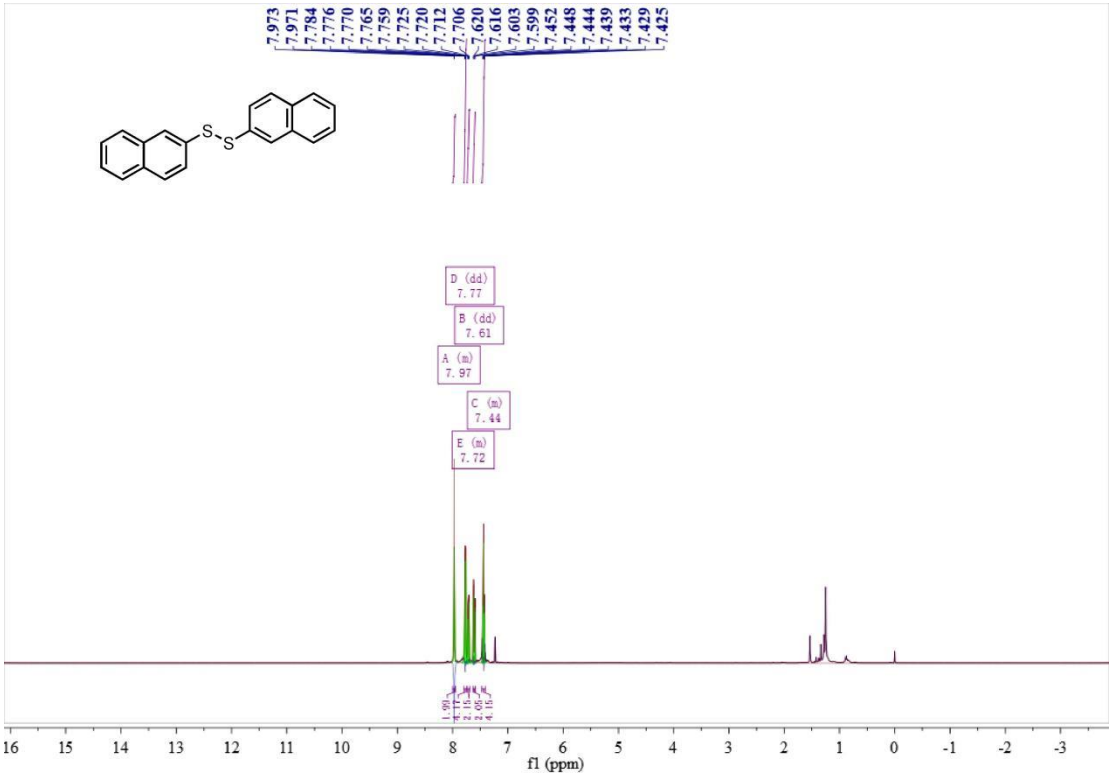

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(2-chlorophenyl)disulfane (3k)** in $CDCl_3$



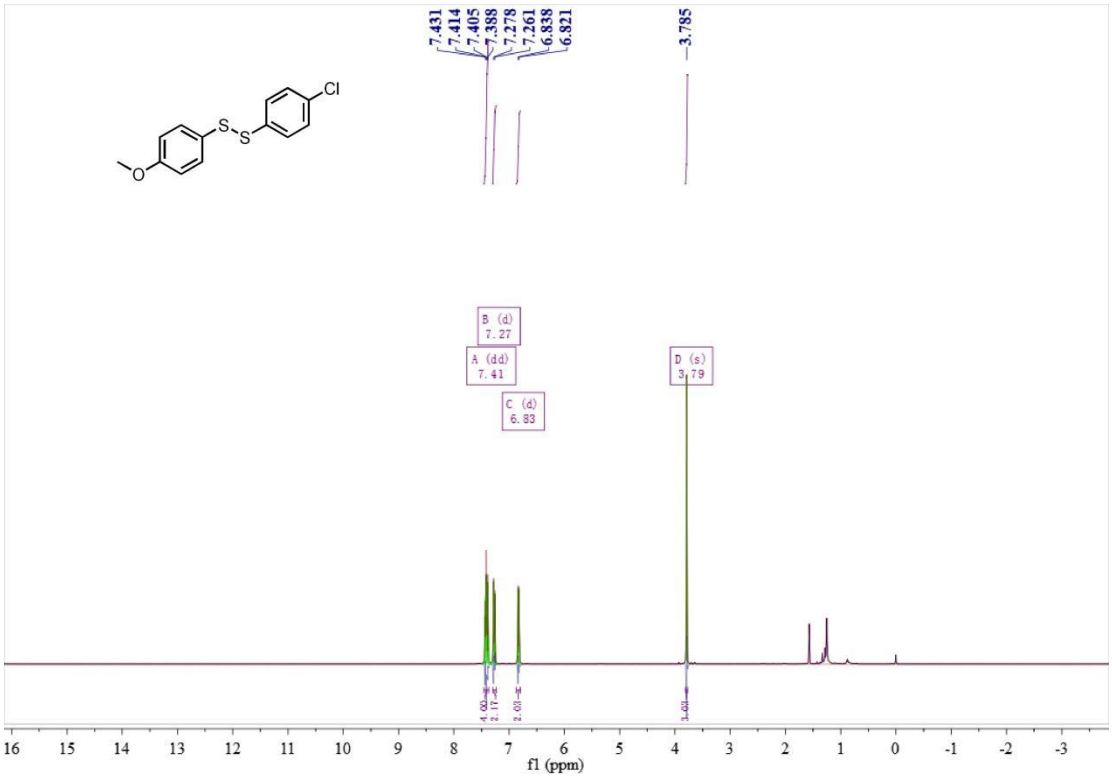

$^{1}$H NMR (500MHz) Spectrum of **1,2-bis(furan-2-ylmethyl)disulfane (3l)** in $CDCl_3$



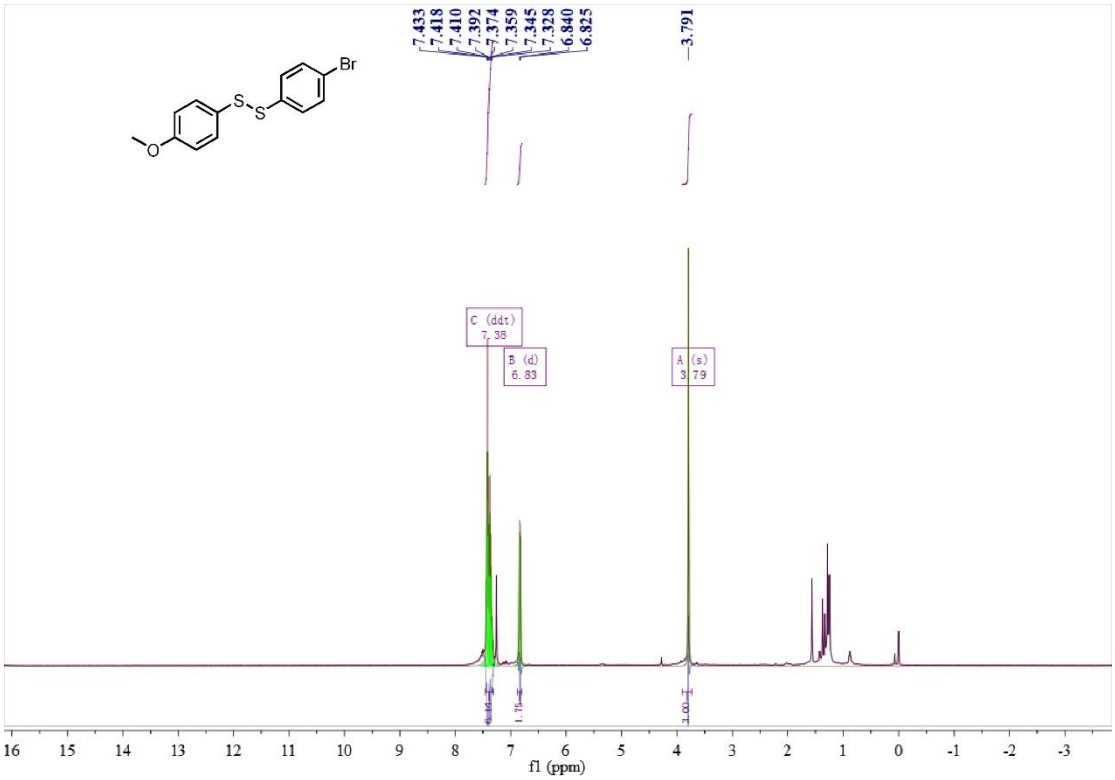

$^{1}$H NMR (500MHz) Spectrum of **1,2-di(naphthalen-2-yl)disulfane (3m)** in $CDCl_3$



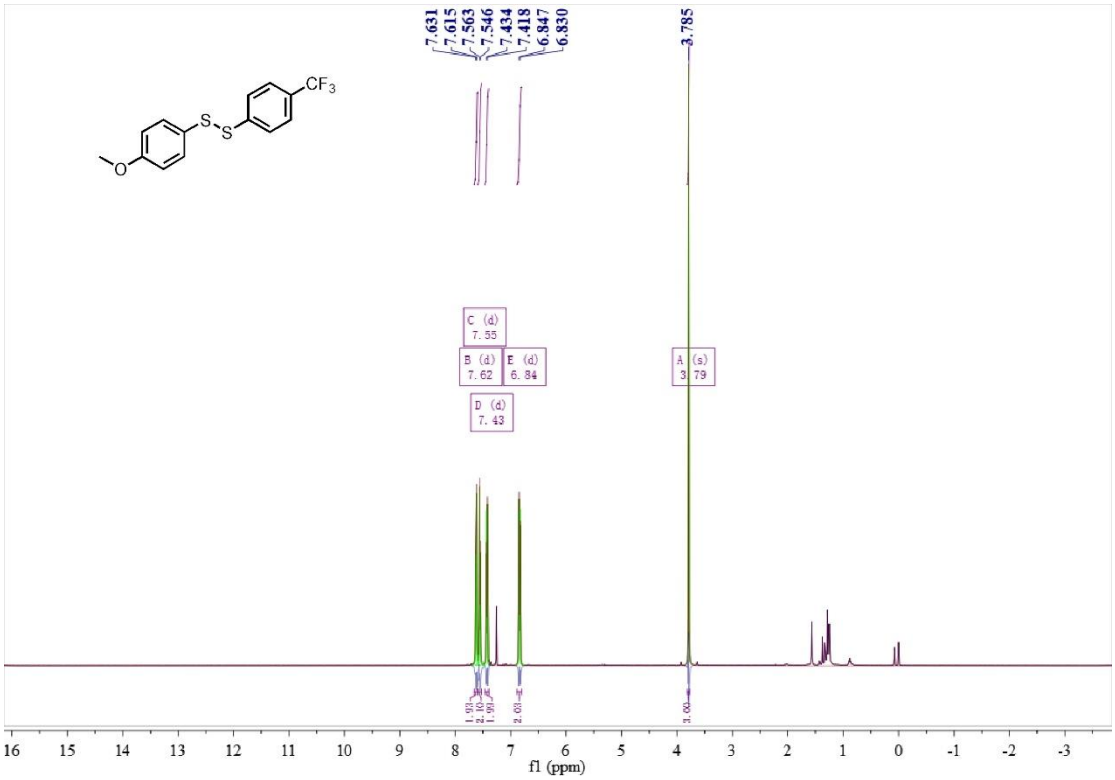
$^{1}$H NMR (500MHz) Spectrum of **1-(4-chlorophenyl)-2-(4-methoxyphenyl)disulfane (3n)** in $CDCl_3$



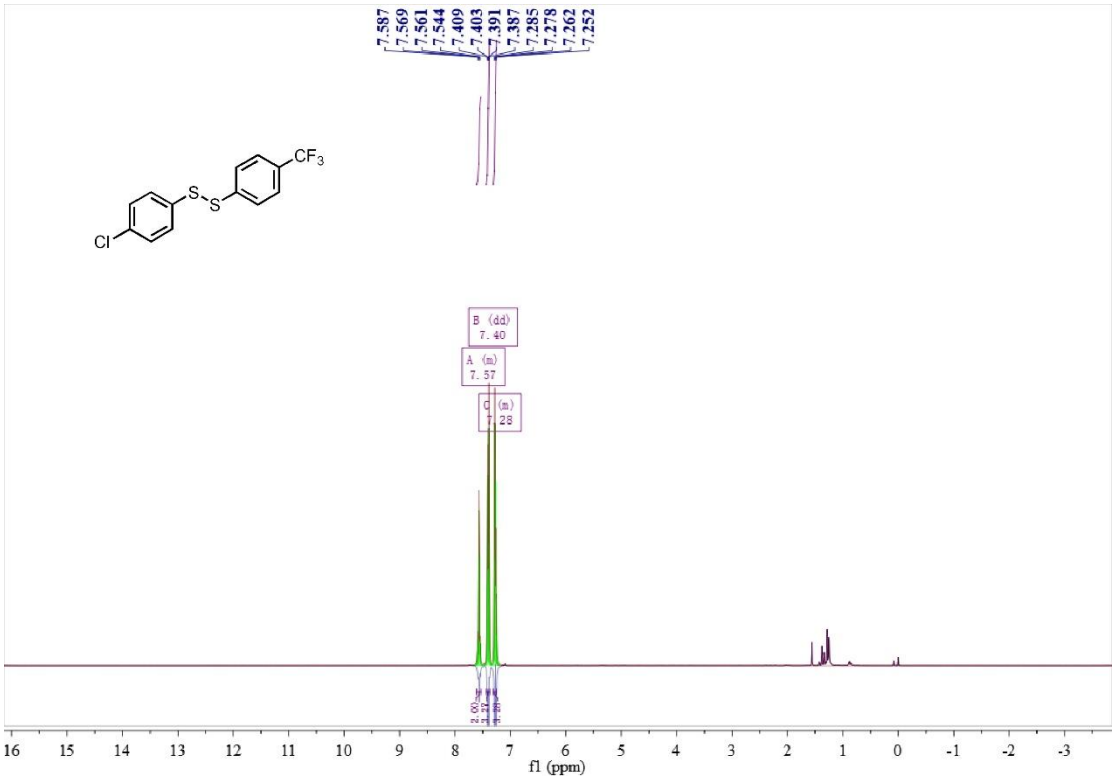

$^{1}$H NMR (500MHz) Spectrum of **1-(4-bromophenyl)-2-(4-methoxyphenyl) disulfane (3o)** in $CDCl_3$



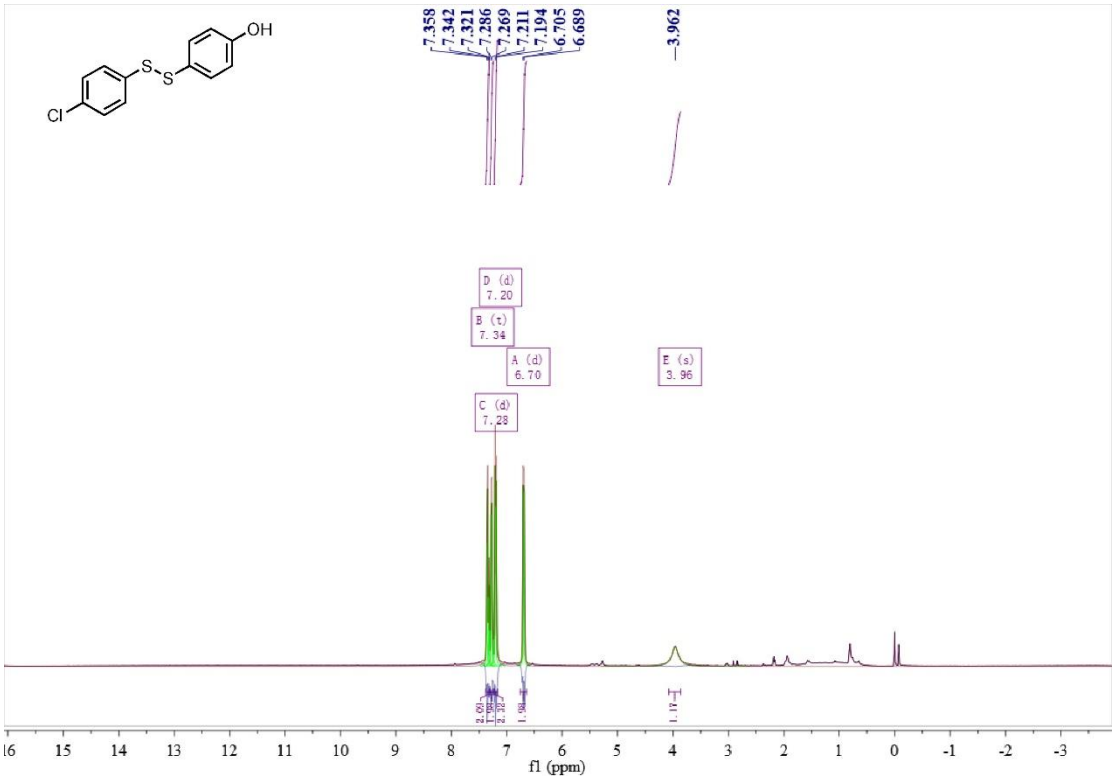

$^1$H NMR (500MHz) Spectrum of **1-(4-methoxyphenyl)-2-(4-(trifluoromethyl) phenyl)disulfane (3p)** in $CDCl_3$



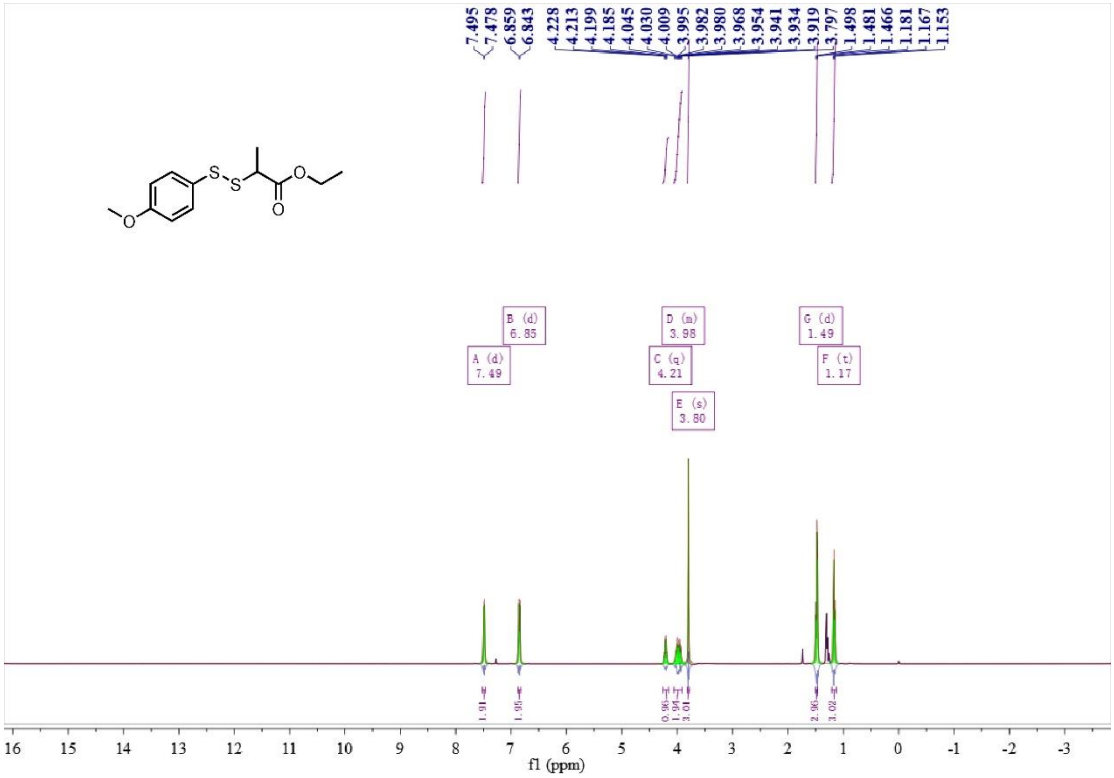

$^1$H NMR (500MHz) Spectrum of **1-(4-chlorophenyl)-2-(4-(trifluoromethyl) phenyl)disulfane (3q)** in $CDCl_3$



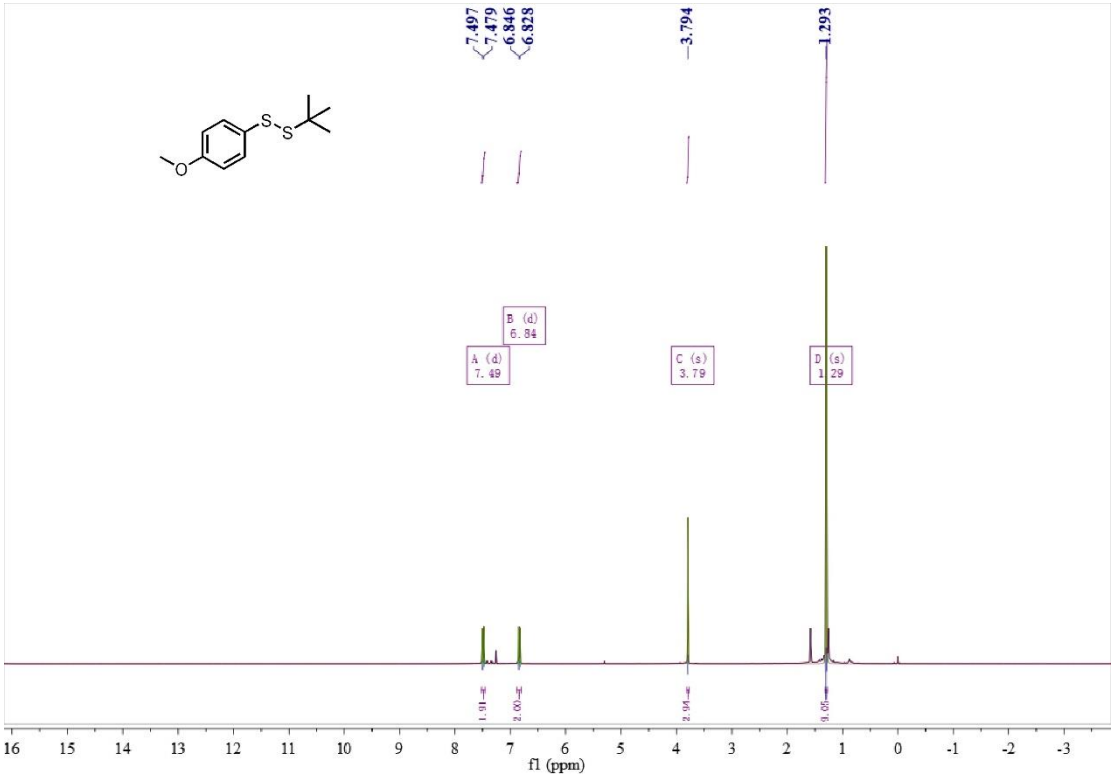

$^1$H NMR (500MHz) Spectrum of **4-((4-chlorophenyl)disulfaneyl)phenol (3r)** in $CDCl_3$



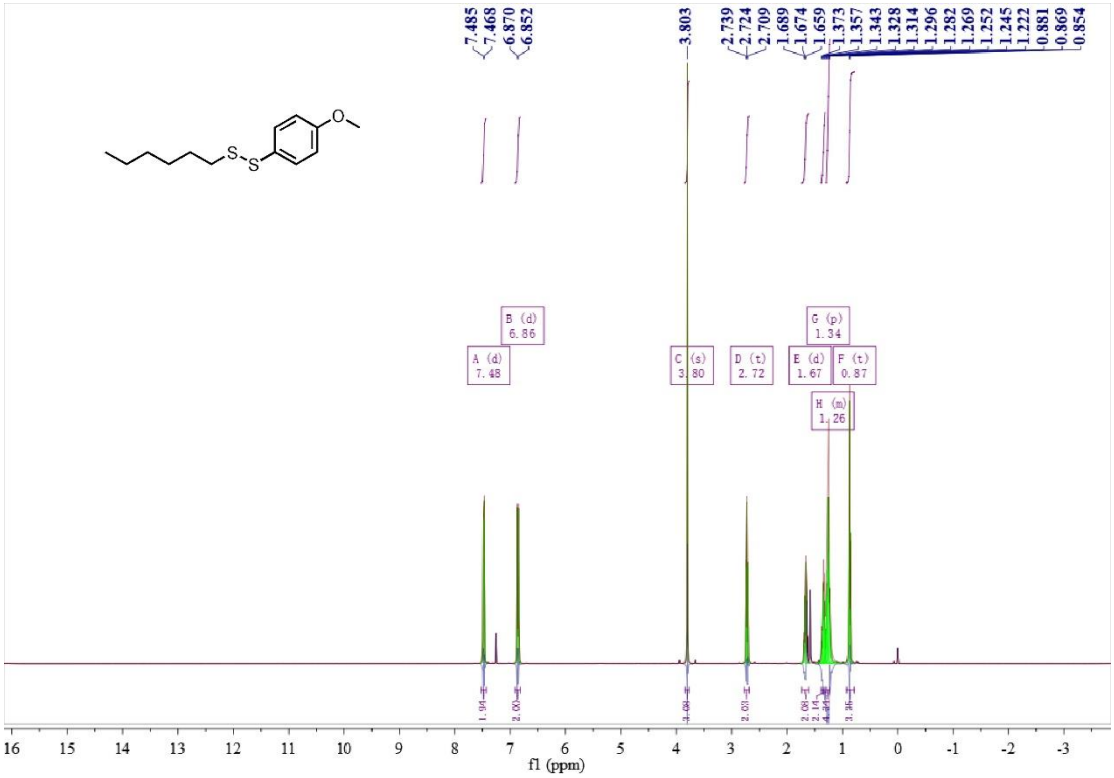

$^1$H NMR (500MHz) Spectrum of **ethyl 2-((4-methoxyphenyl)disulfaneyl) propanoate (3s)** in $CDCl_3$



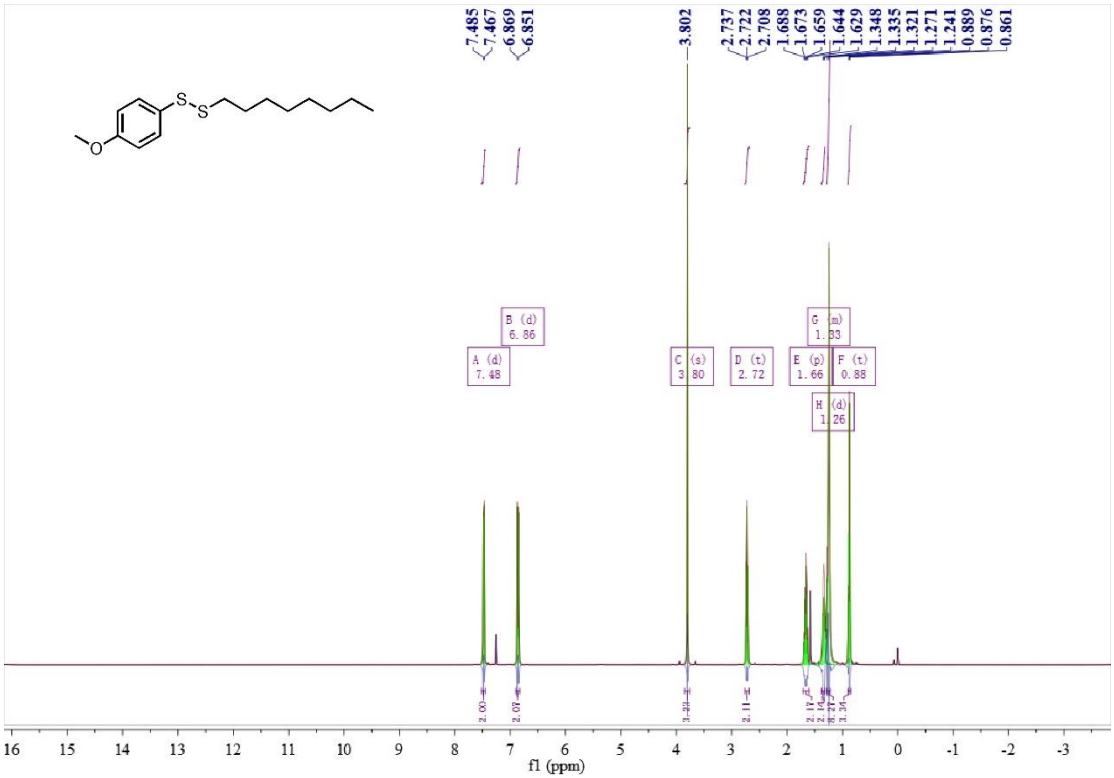

$^1$H NMR (500MHz) Spectrum of **1-(tert-butyl)-2-(4-methoxyphenyl)disulfane (3t)** in $CDCl_3$



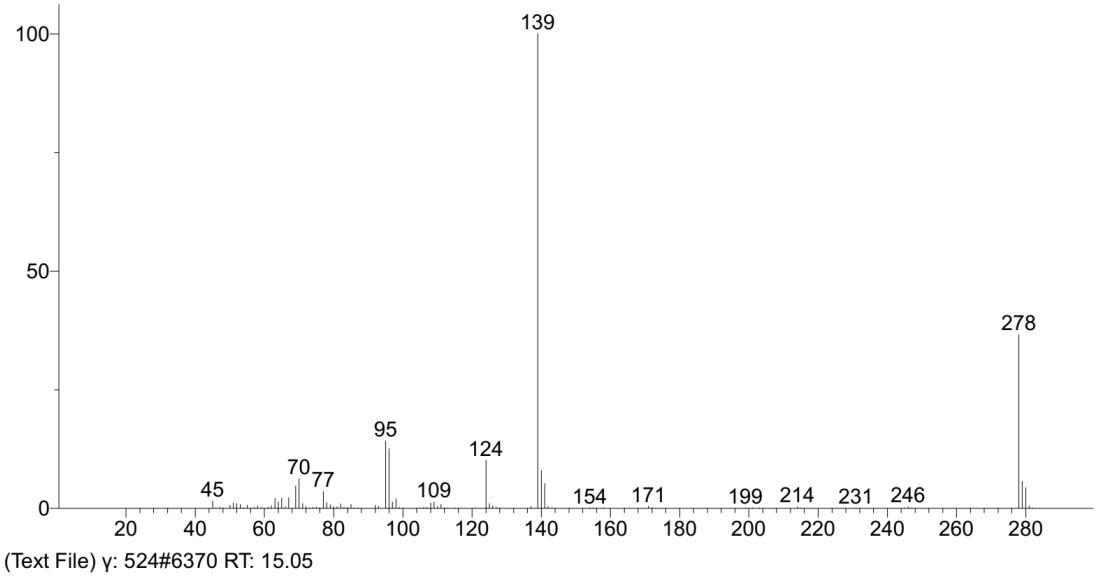

$^1$H NMR (500MHz) Spectrum of **1-hexyl-2-(4-methoxyphenyl)disulfane (3u)** in $CDCl_3$

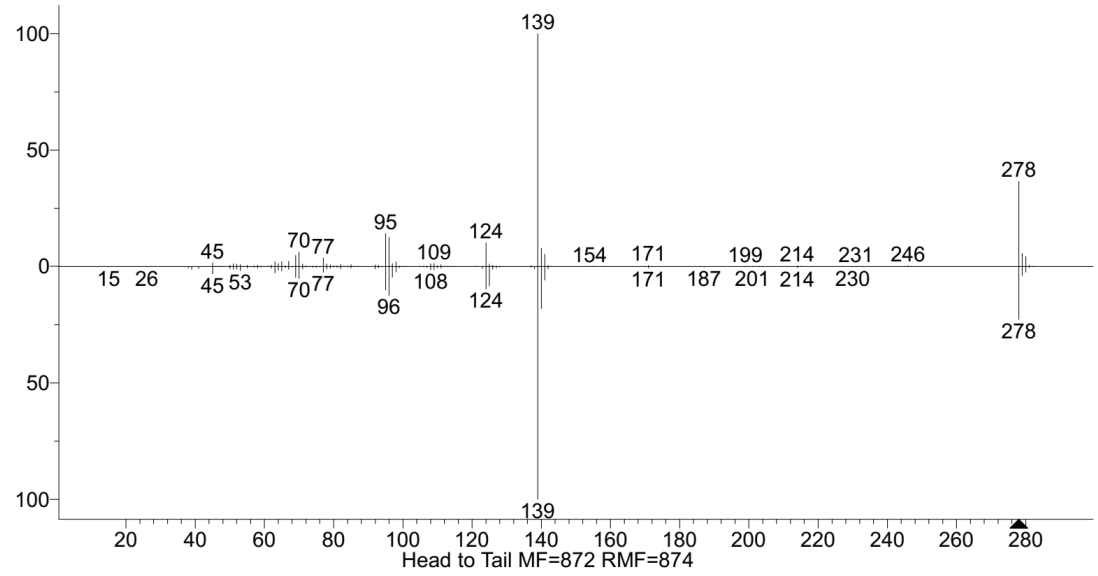


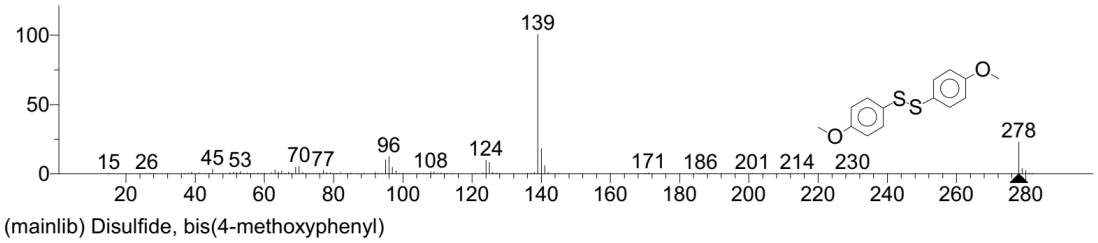

$^{1}$H NMR (500MHz) Spectrum of **1-(4-methoxyphenyl)-2-octyldisulfane (3v)** in $CDCl_3$



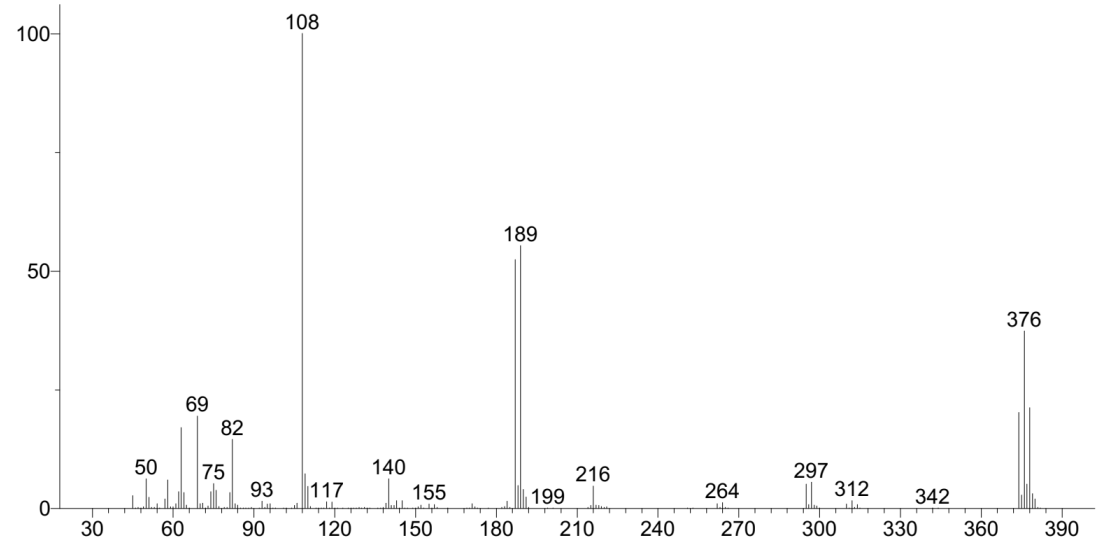
GC-MS of Compound **1,2-bis(4-methoxyphenyl)disulfane (3a)**



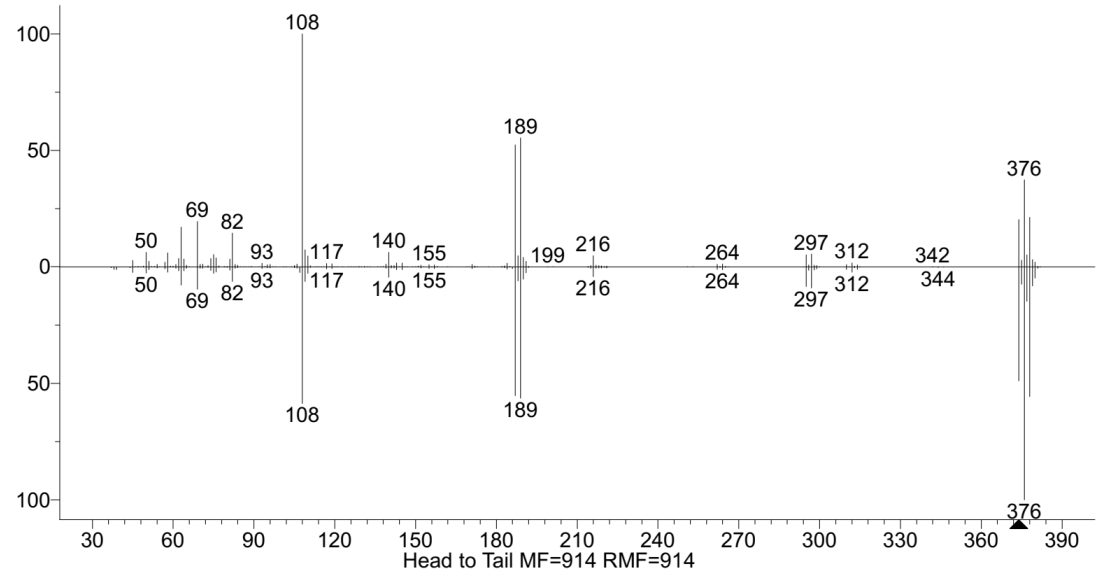



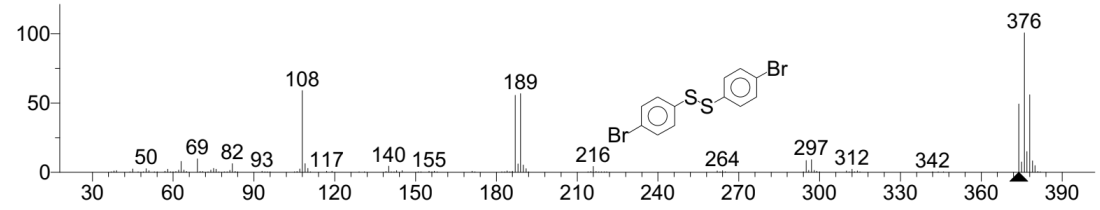
GC-MS of Compound **1,2-bis(4-bromophenyl)disulfane (3b)**




(Text File) γ: 542#6907 RT: 15.98

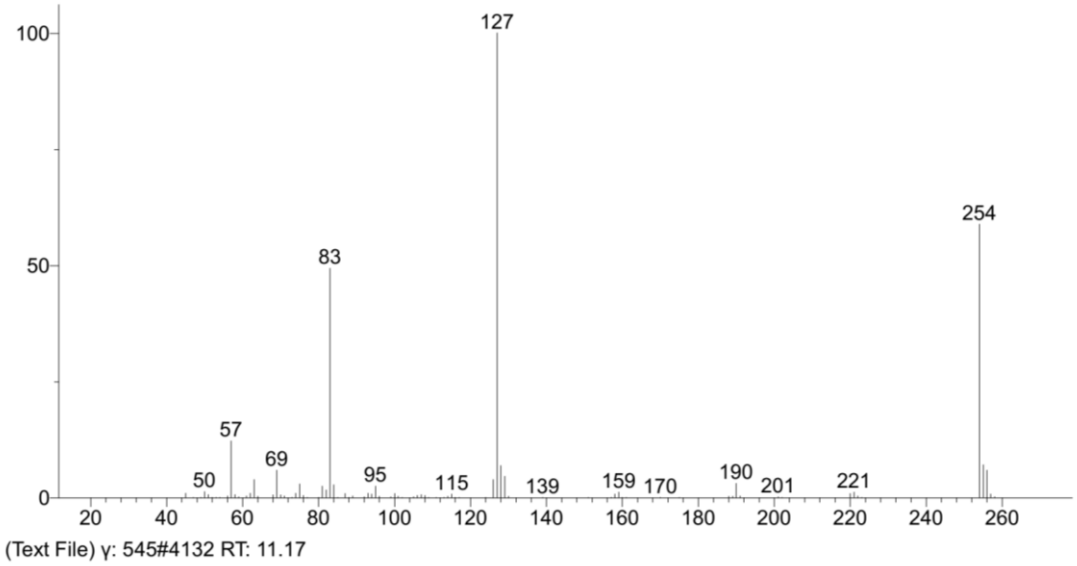


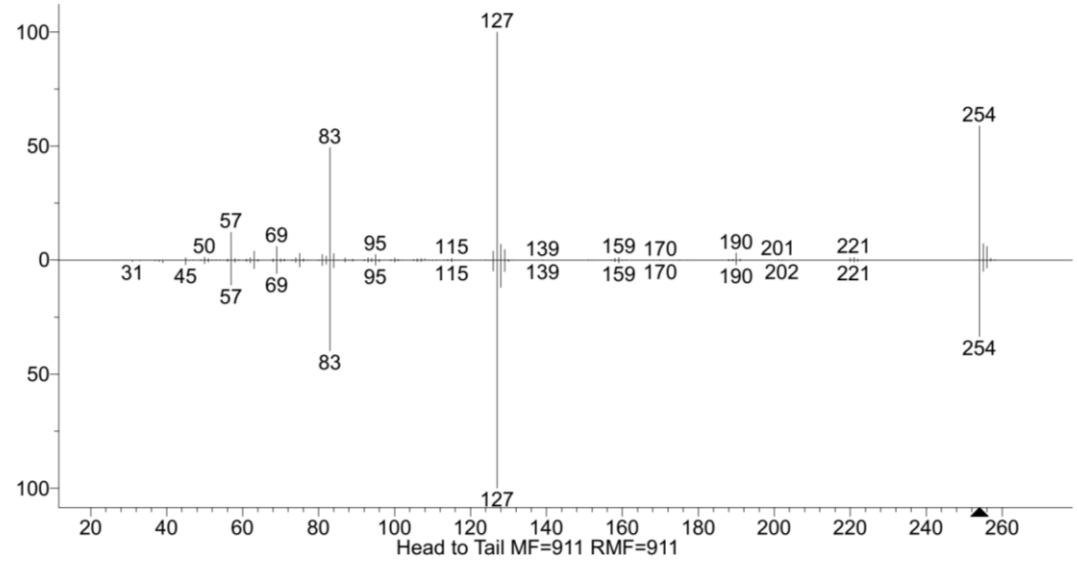

Head to Tail MF=914 RMF=914

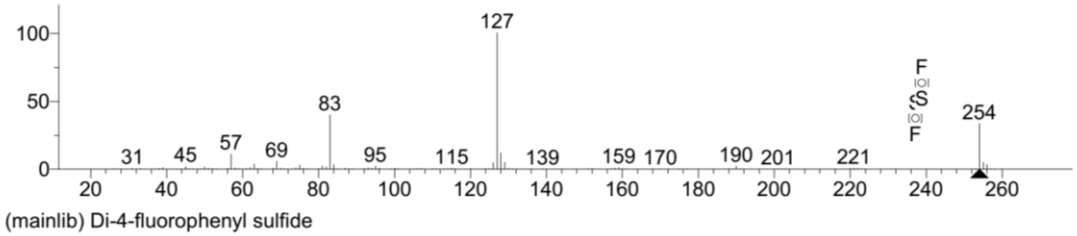



(mainlib) Bis(p-Bromophenyl) disulfide

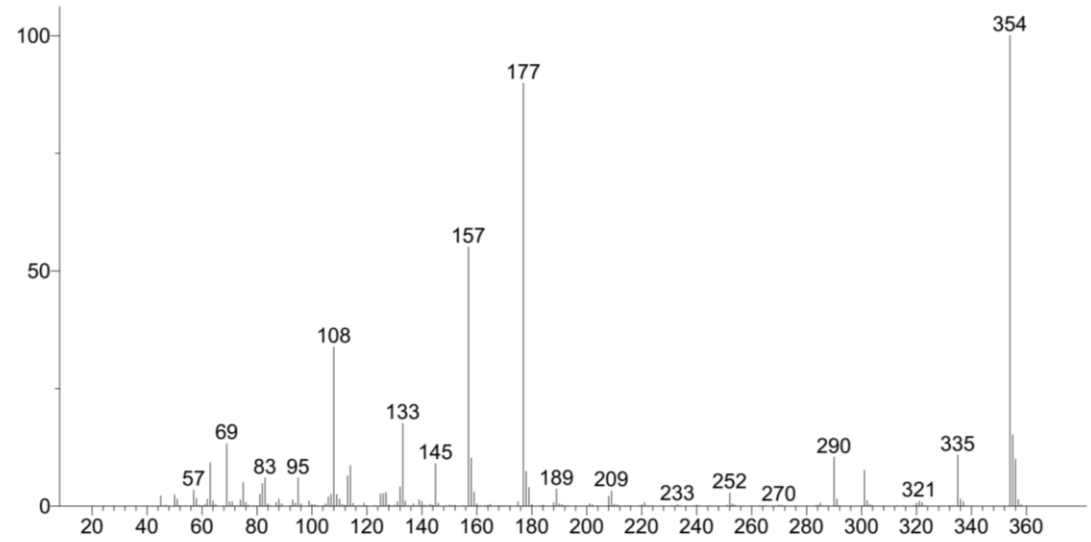
GC-MS of Compound **1,2-bis(4-(trifluoromethyl)phenyl)disulfane (3c)**

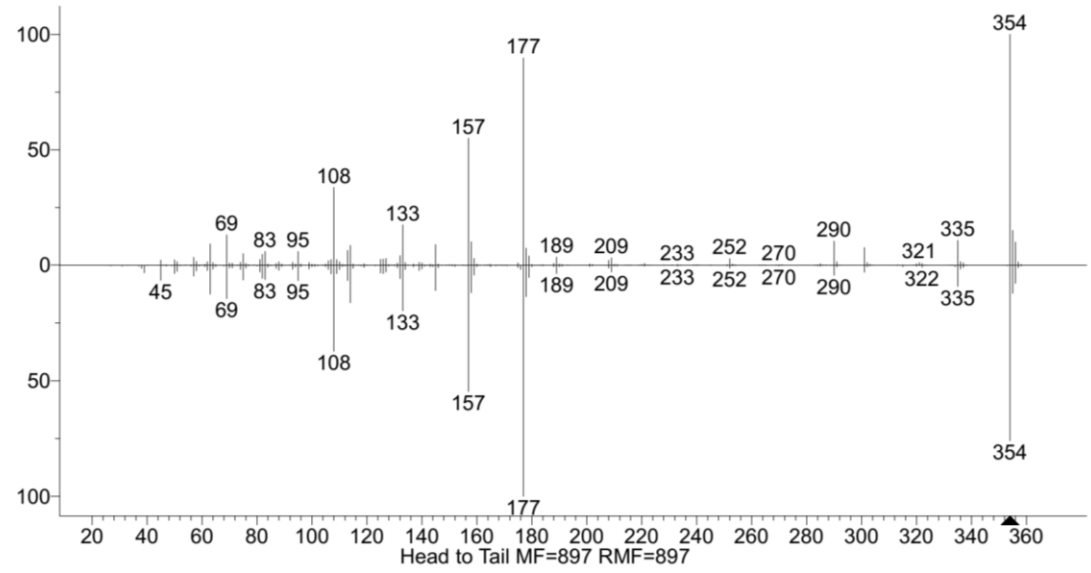


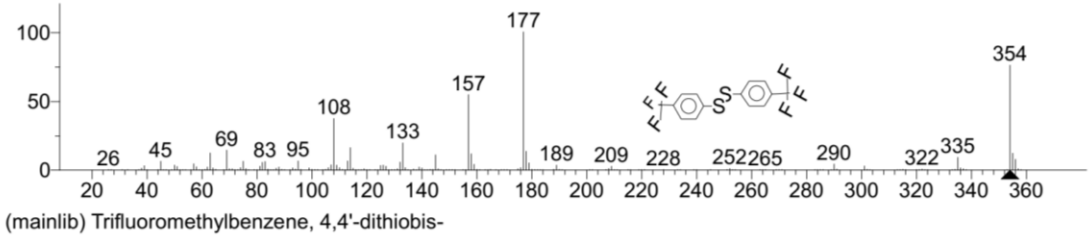



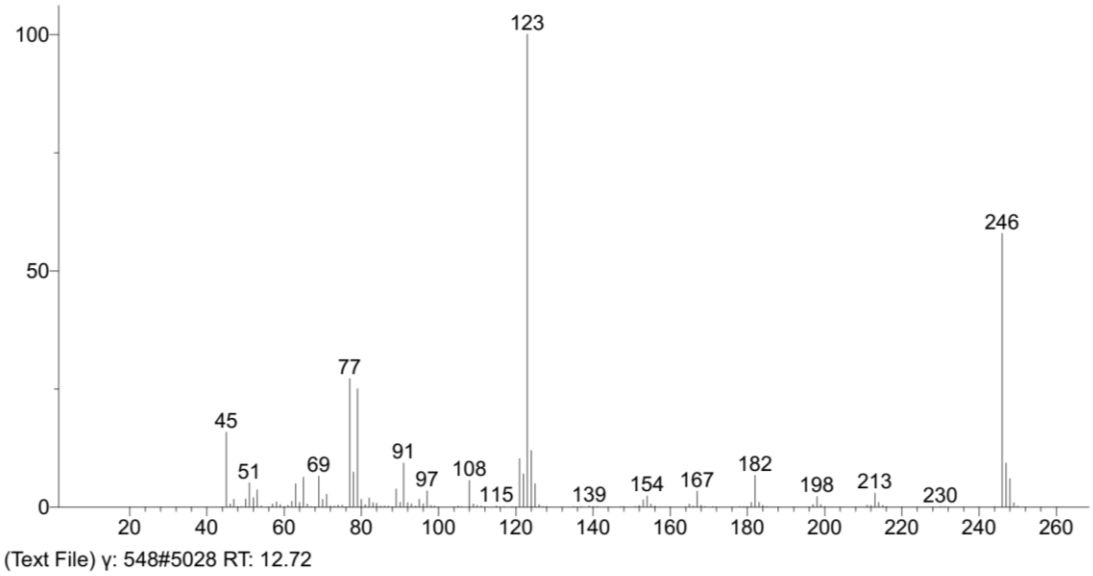
GC-MS of Compound **1,2-bis(4-(trifluoromethyl)phenyl)disulfane (3d)**

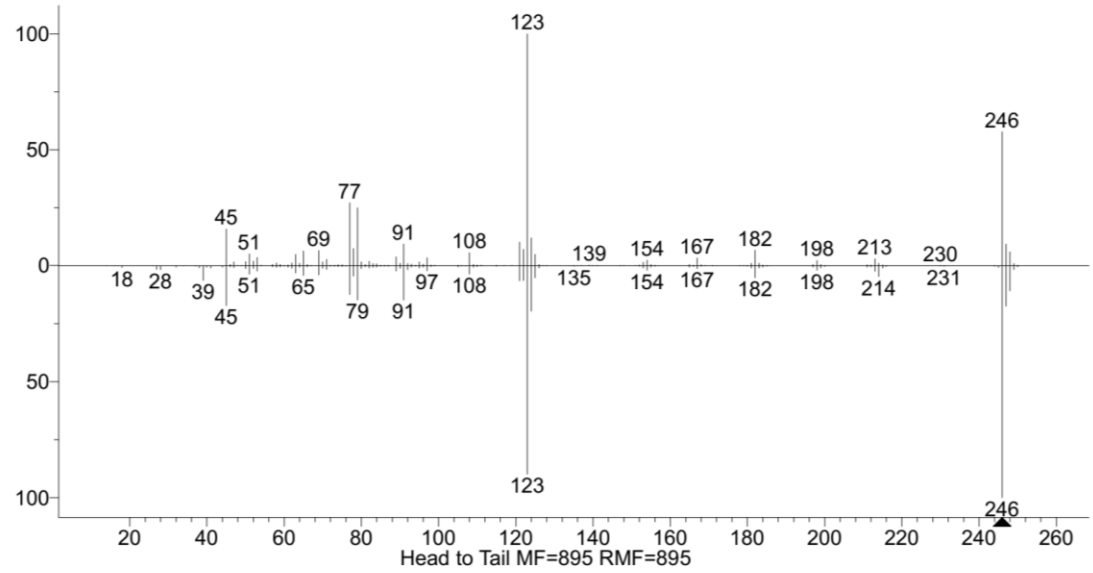


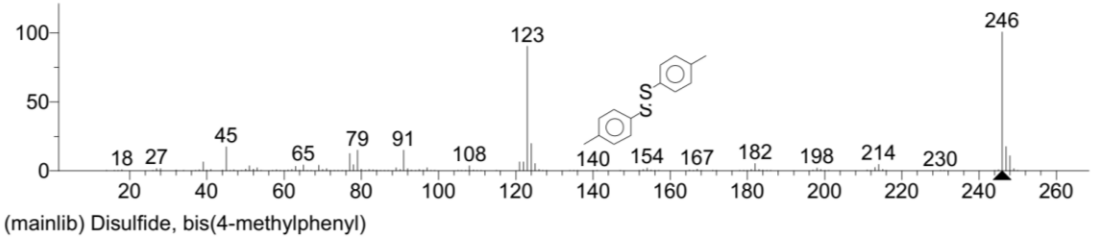



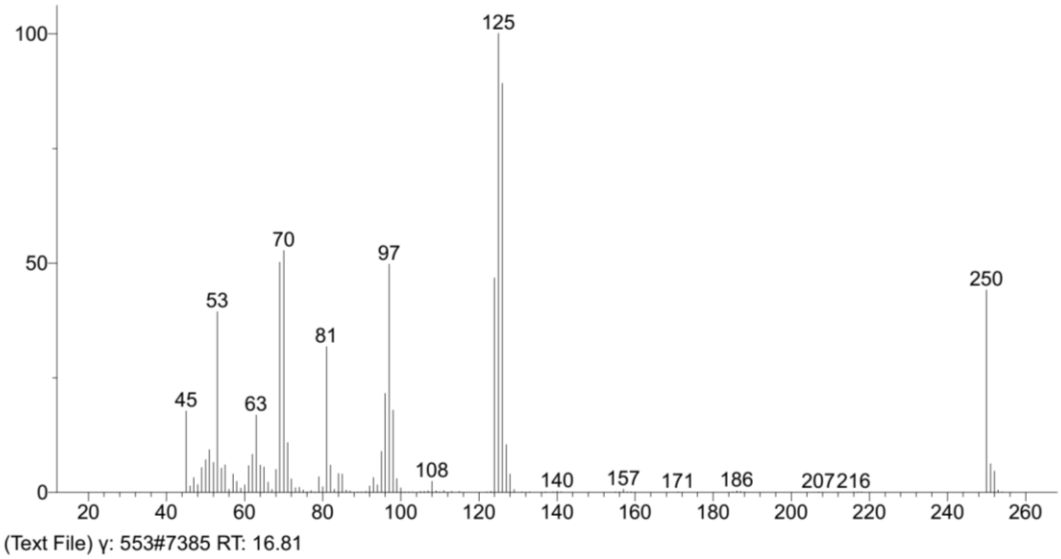
GC-MS of Compound **1,2-di-p-tolyldisulfane (3e)**

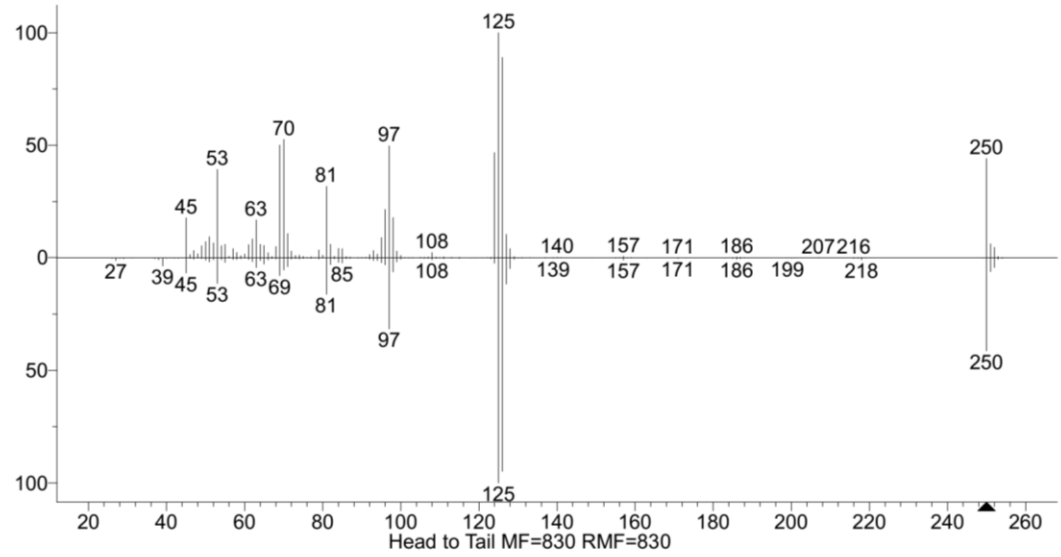


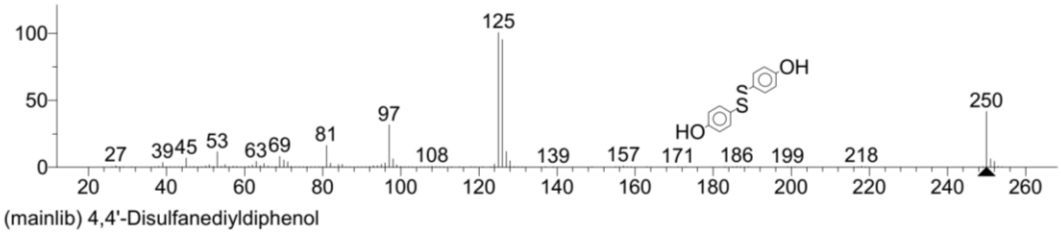



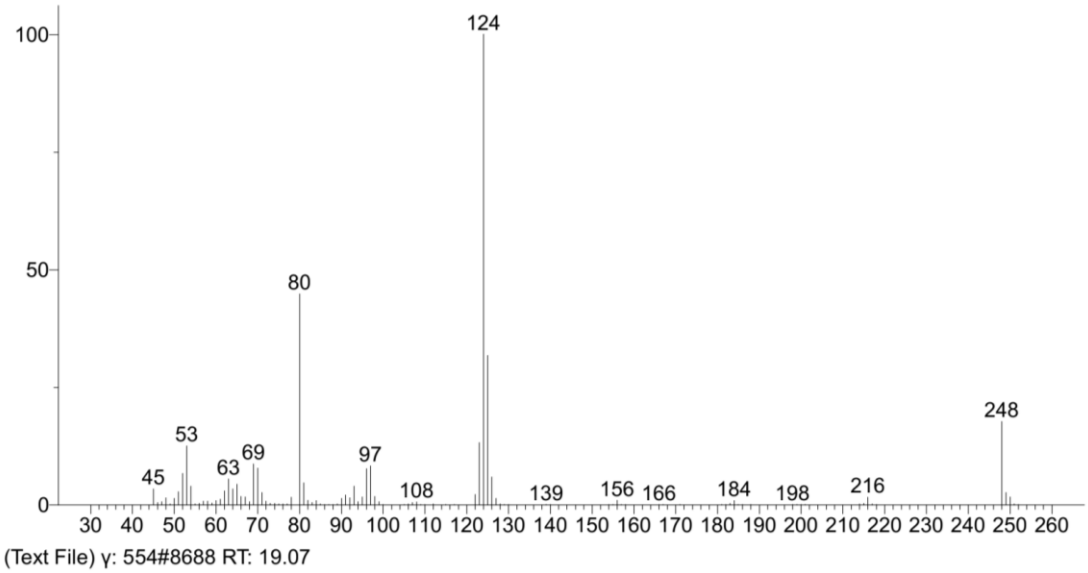
GC-MS of Compound **4,4'-disulfanediyldiphenol (3f)**



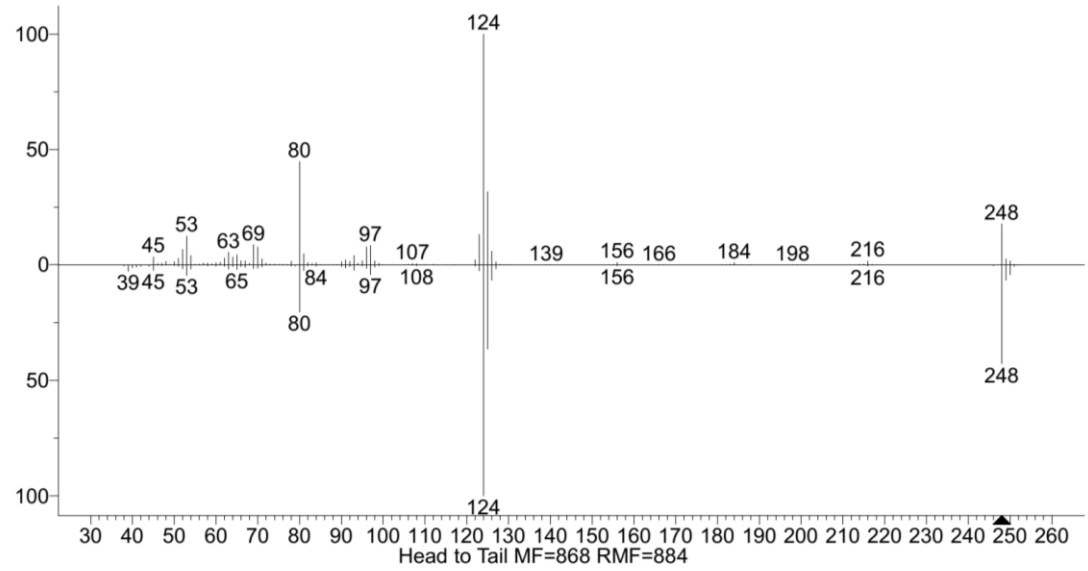



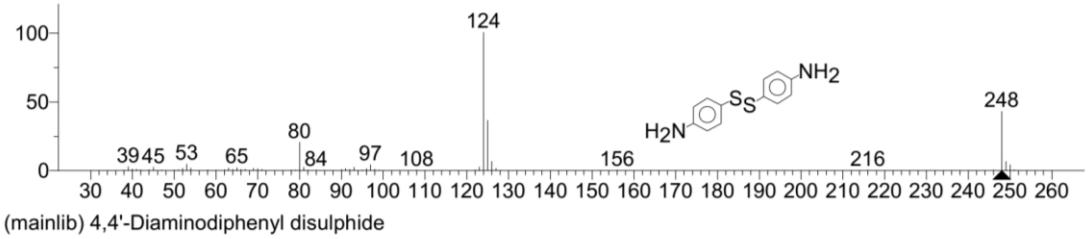
GC-MS of Compound **4,4'-disulfanediyldianiline (3g)**




(Text File) γ: 554#8688 RT: 19.07

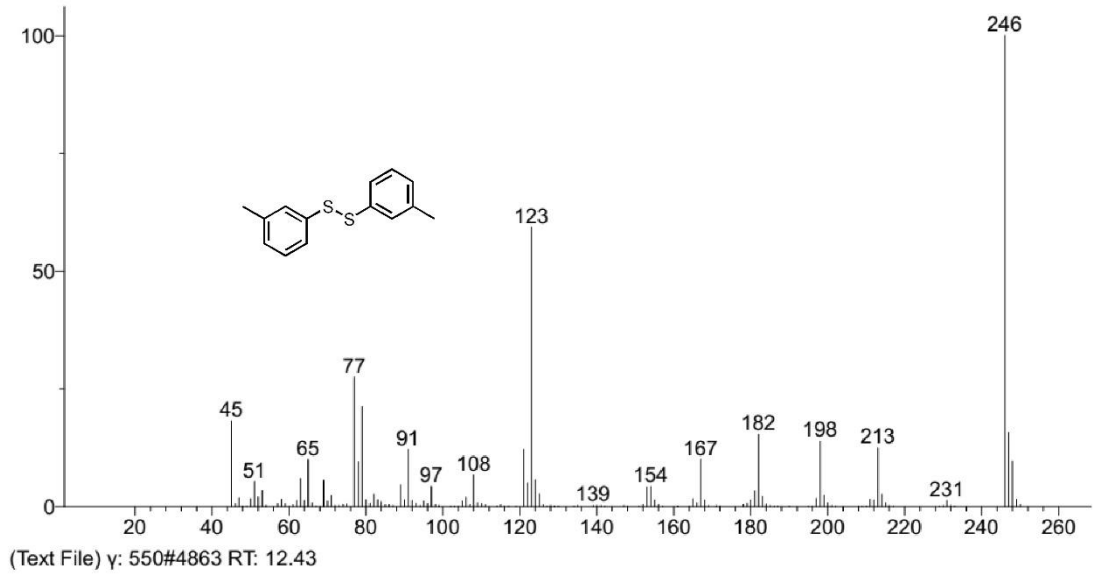



Head to Tail MF=868 RMF=884

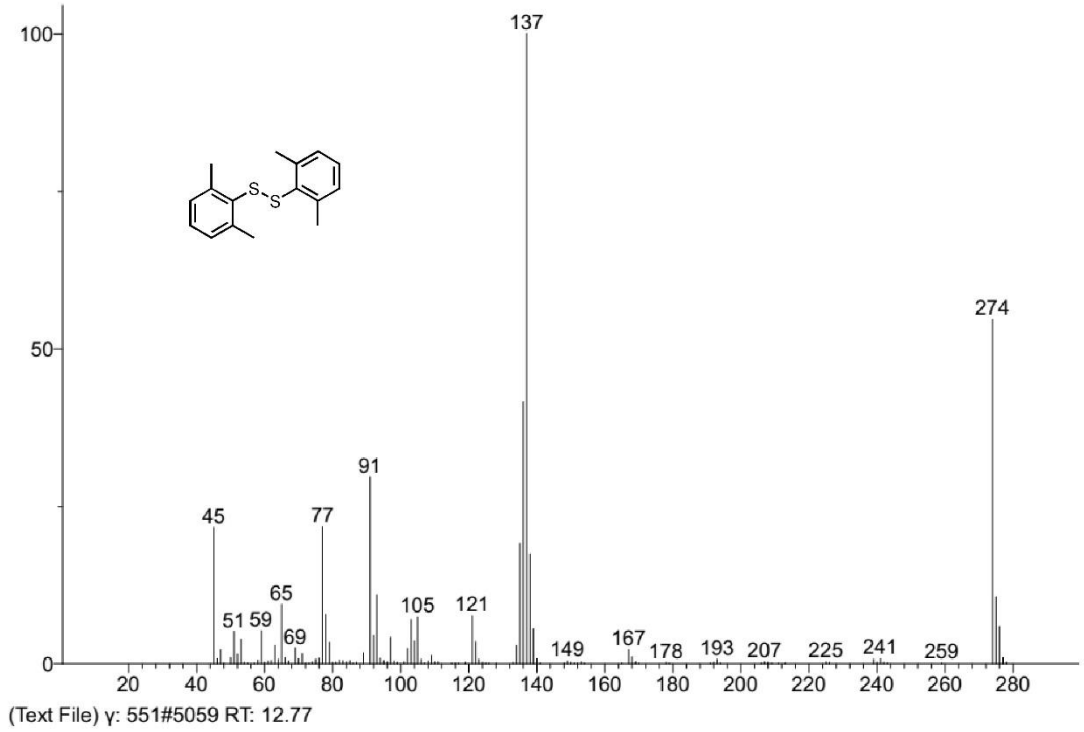



(mainlib) 4,4'-Diaminodiphenyl disulphide

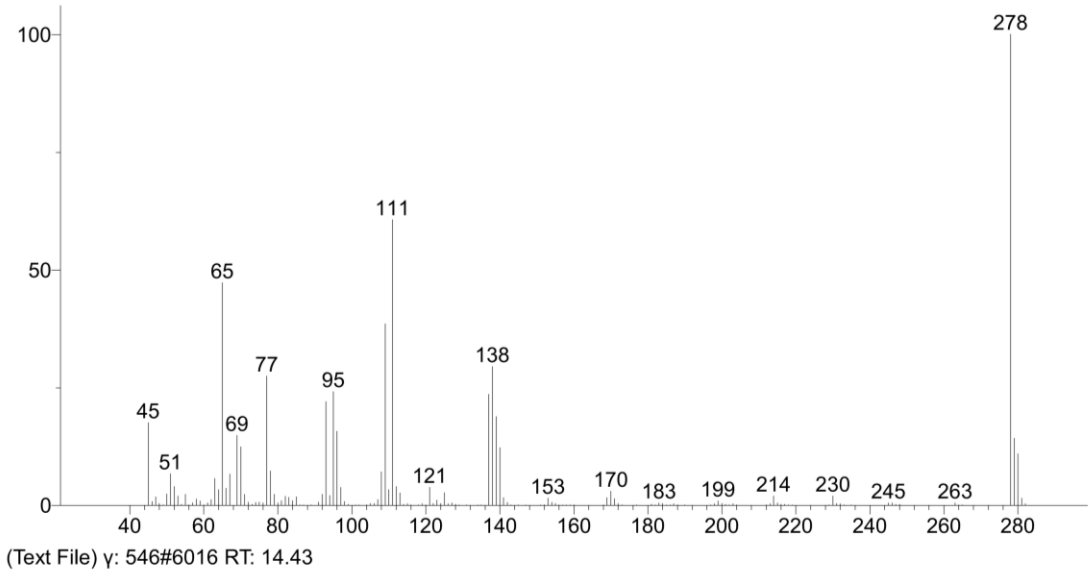
GC-MS of Compound **1,2-di-m-tolyldisulfane (3h)**



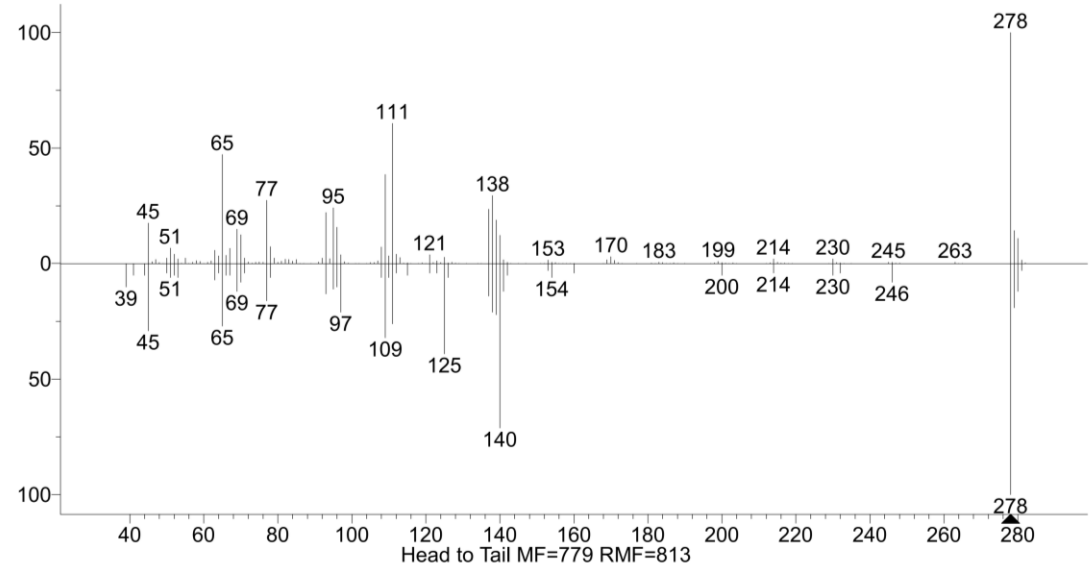

GC-MS of Compound **1,2-bis(2,6-dimethylphenyl)disulfane (3i)**



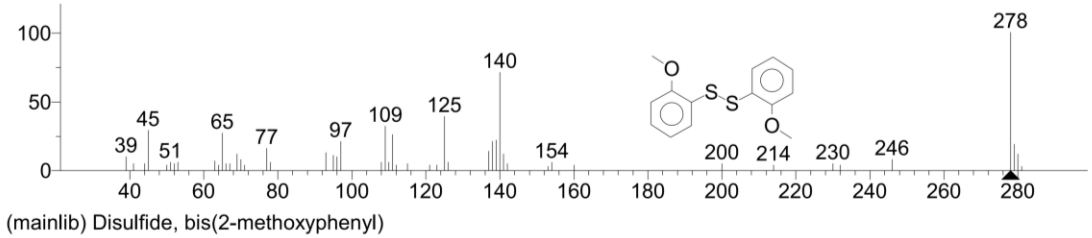
GC-MS of Compound **1,2-bis(2-methoxyphenyl)disulfane (3j)**




(Text File) γ: 546#6016 RT: 14.43

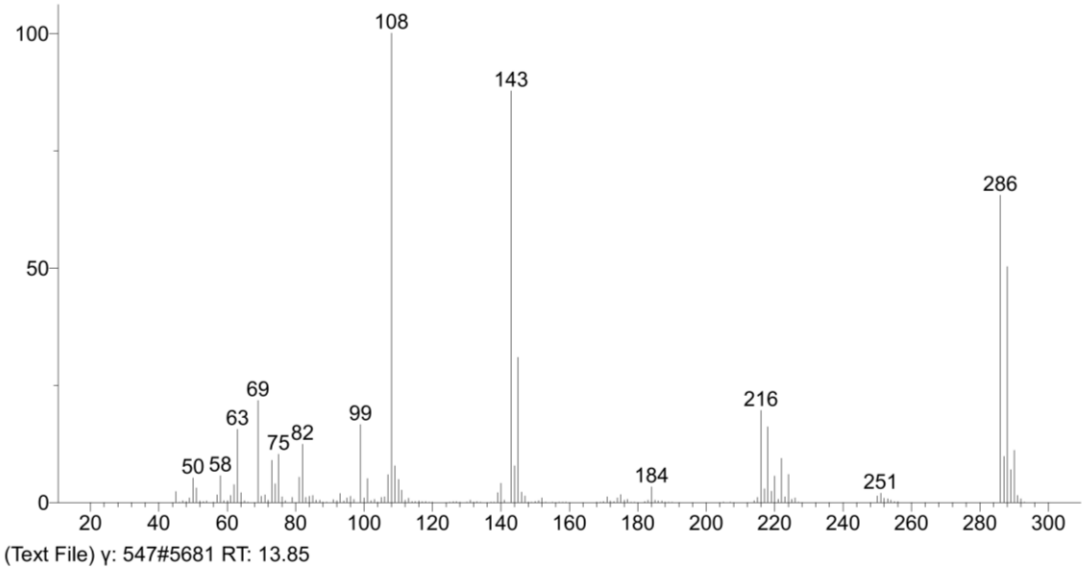


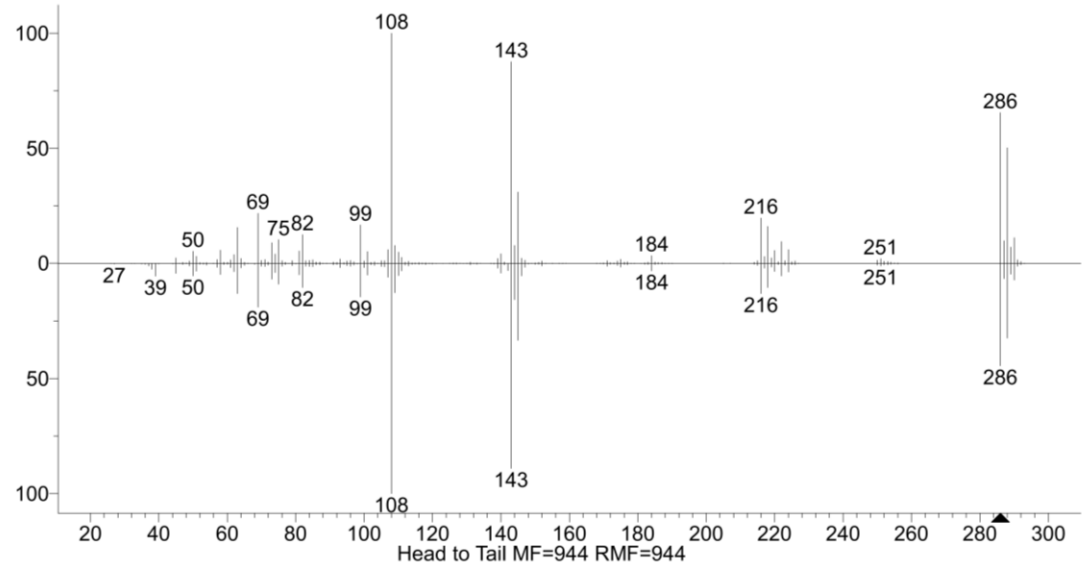

Head to Tail MF=779 RMF=813

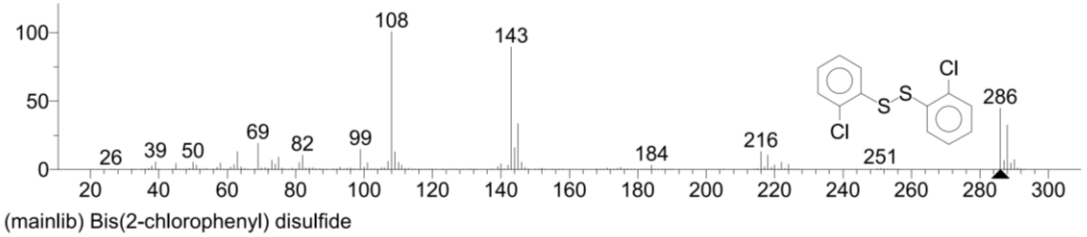



(mainlib) Disulfide, bis(2-methoxyphenyl)

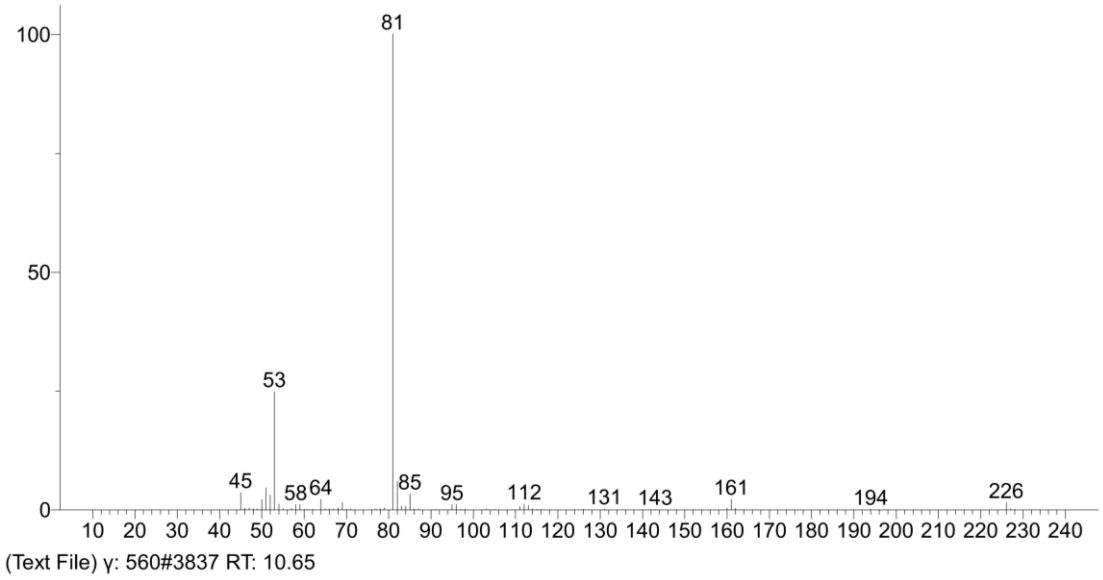
GC-MS of Compound **1,2-bis(2-chlorophenyl)disulfane (3k)**

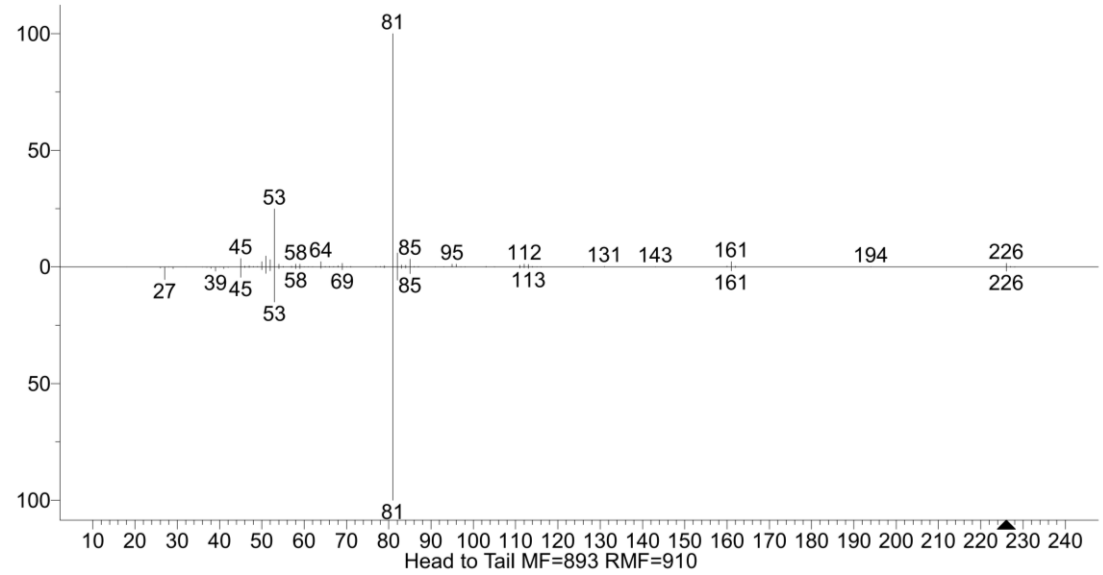


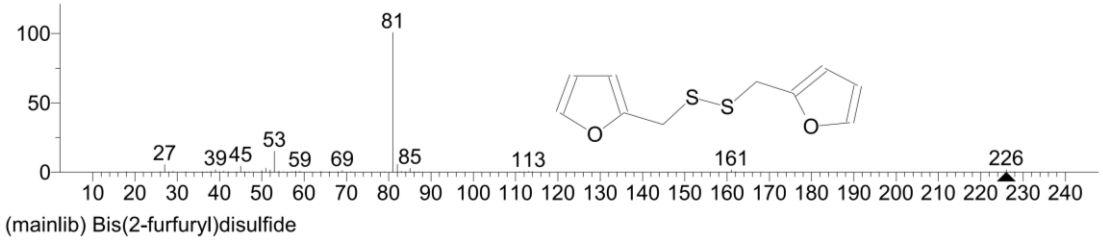



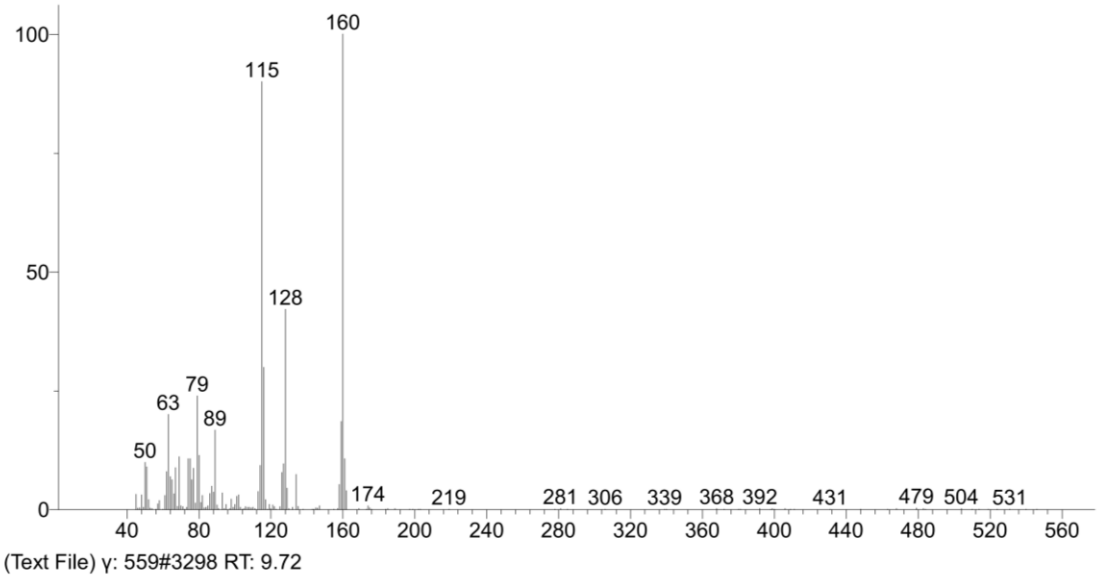
GC-MS of Compound **1,2-bis(furan-2-ylmethyl)disulfane (3l)**



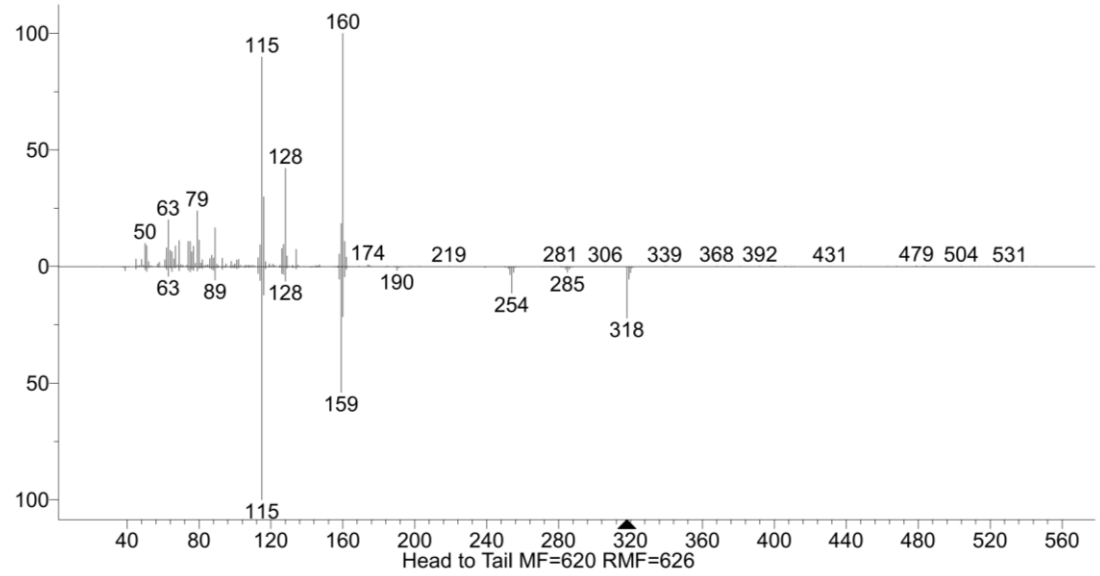



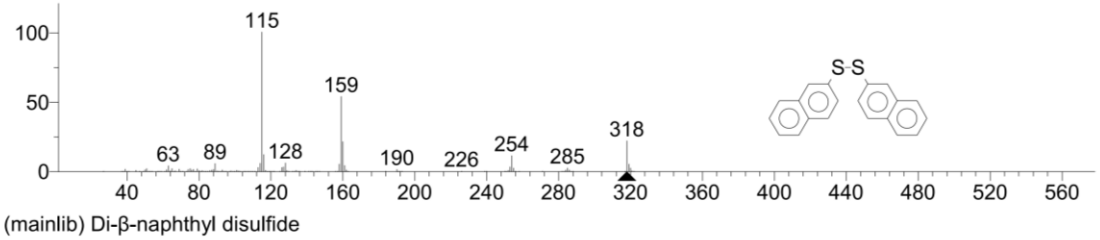
GC-MS of Compound **1,2-di(naphthalen-2-yl)disulfane (3m)**




(Text File) γ: 559#3298 RT: 9.72

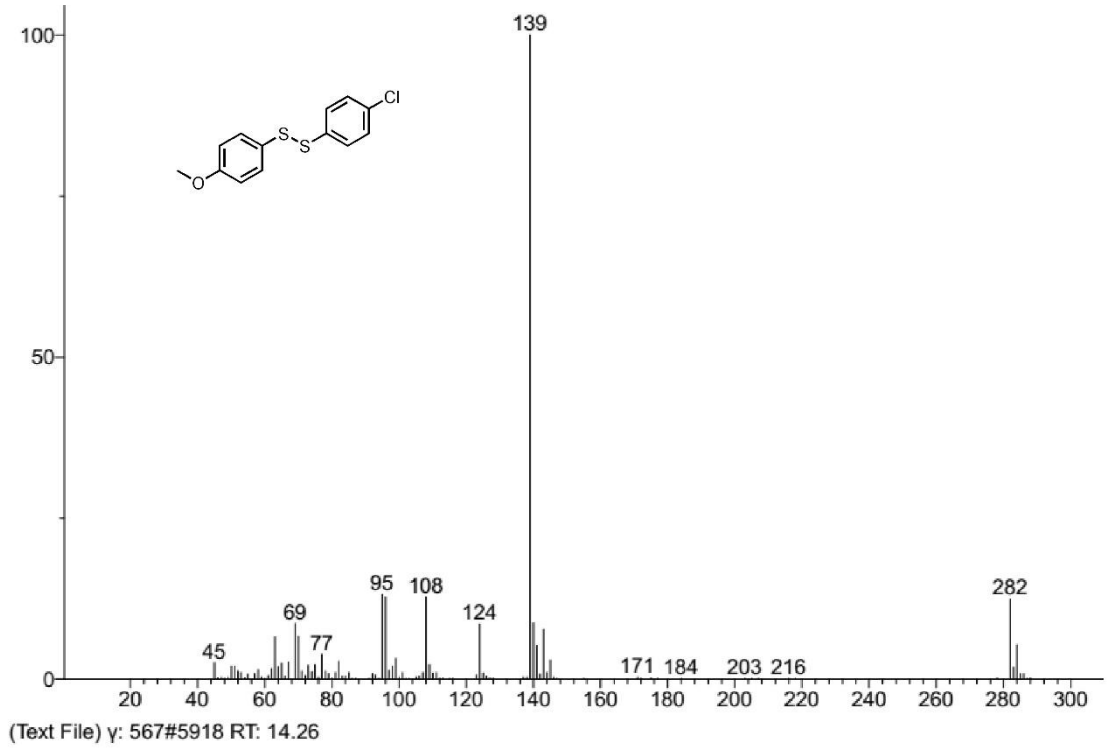



Head to Tail MF=620 RMF=626

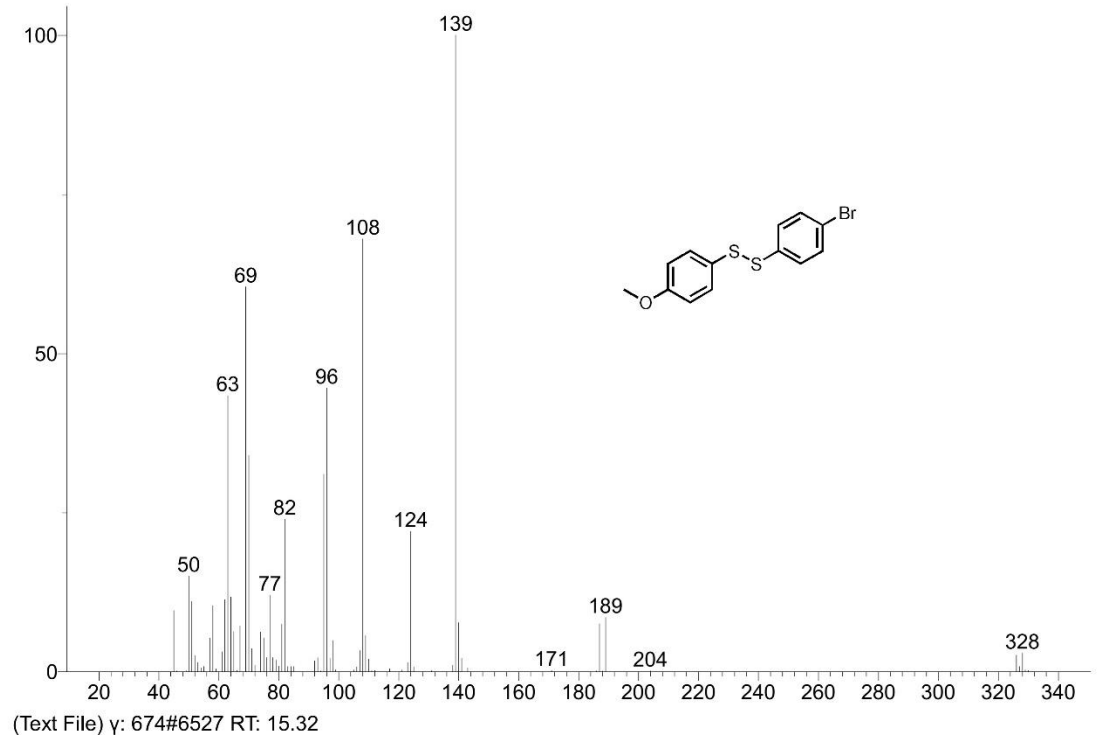



(mainlib) Di-β-naphthyl disulfide

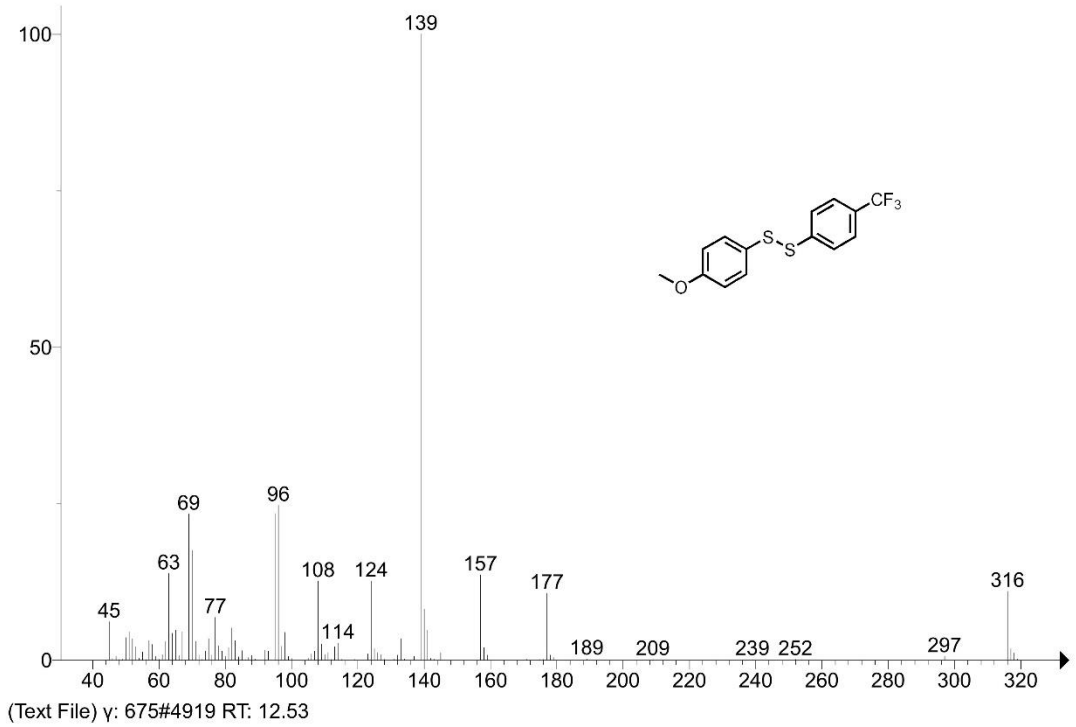
GC-MS of Compound **1-(4-chlorophenyl)-2-(4-methoxyphenyl)disulfane (3n)**



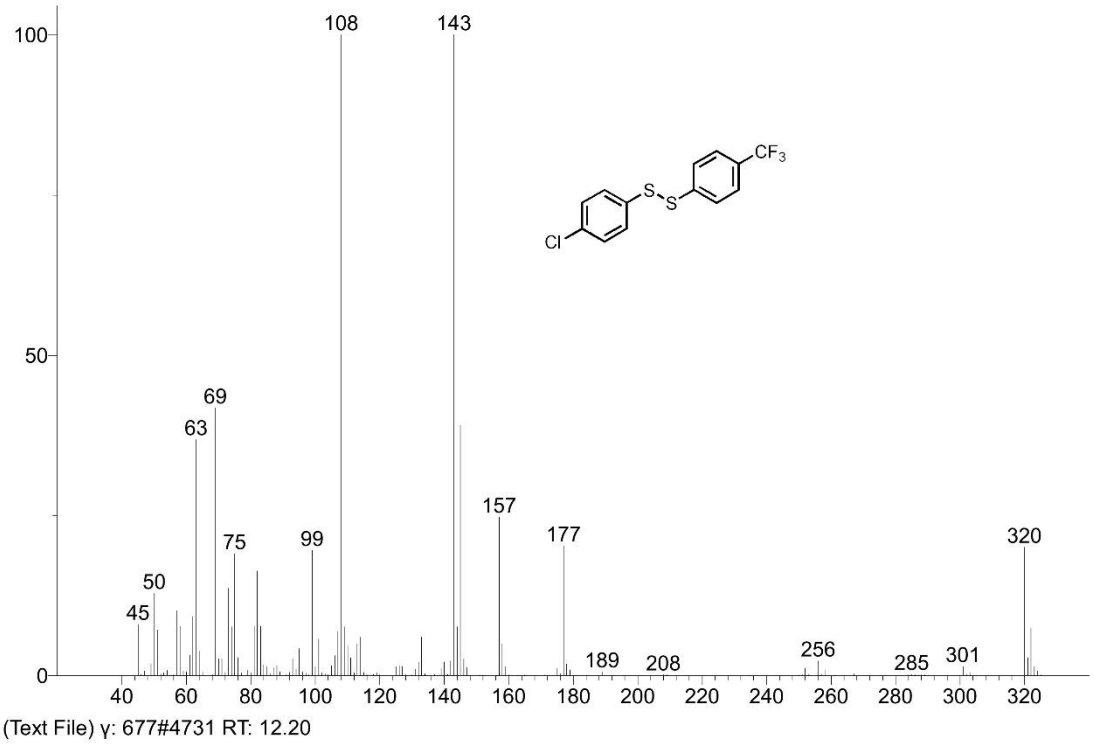

GC-MS of Compound **1-(4-bromophenyl)-2-(4-methoxyphenyl) disulfane (3o)**



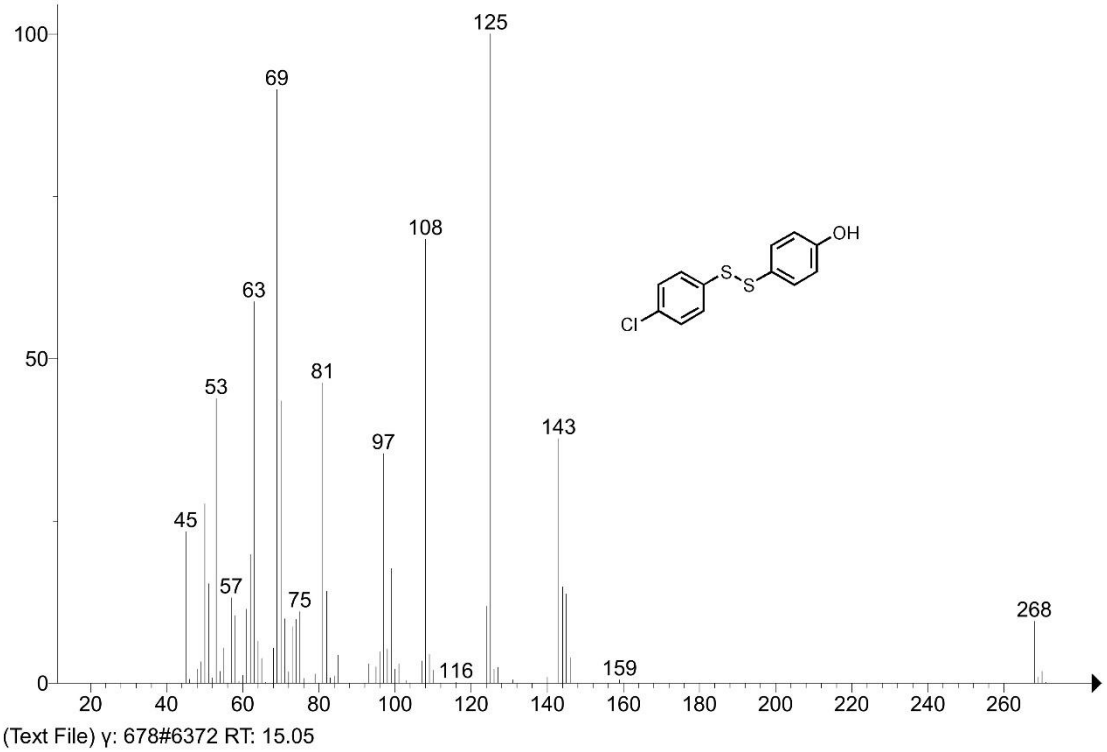
GC-MS of Compound **1-(4-methoxyphenyl)-2-(4-(trifluoromethyl) phenyl) disulfane (3p)**



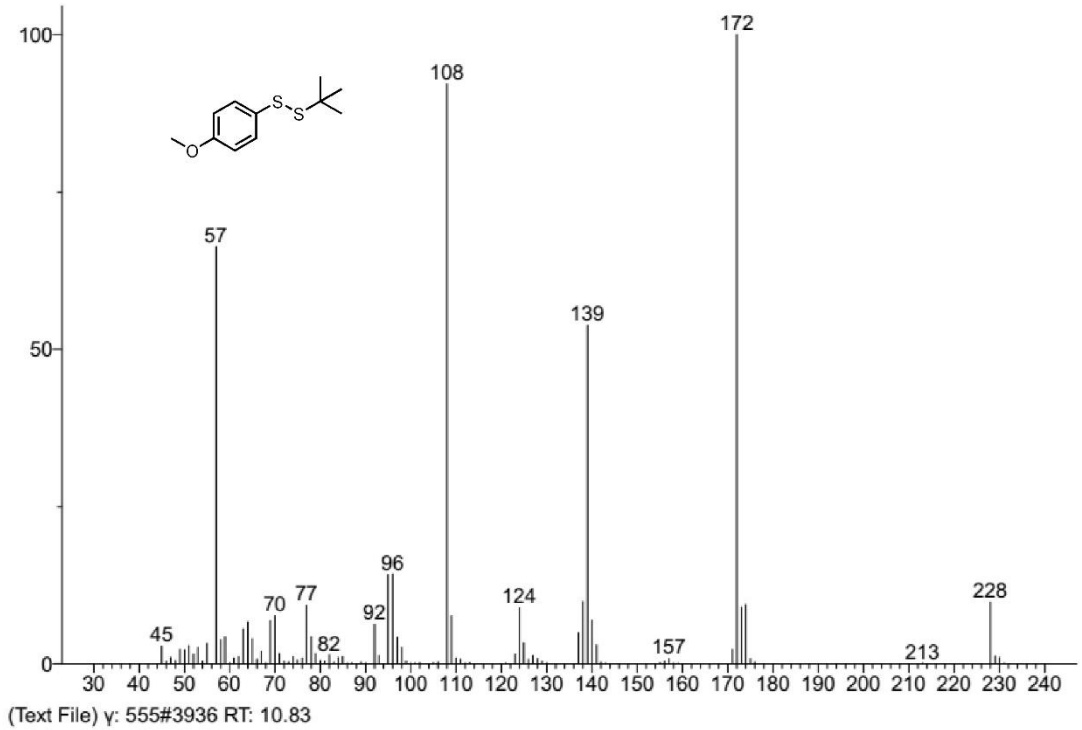

GC-MS of Compound **1-(4-chlorophenyl)-2-(4-(trifluoromethyl) phenyl)disulfane (3q)**



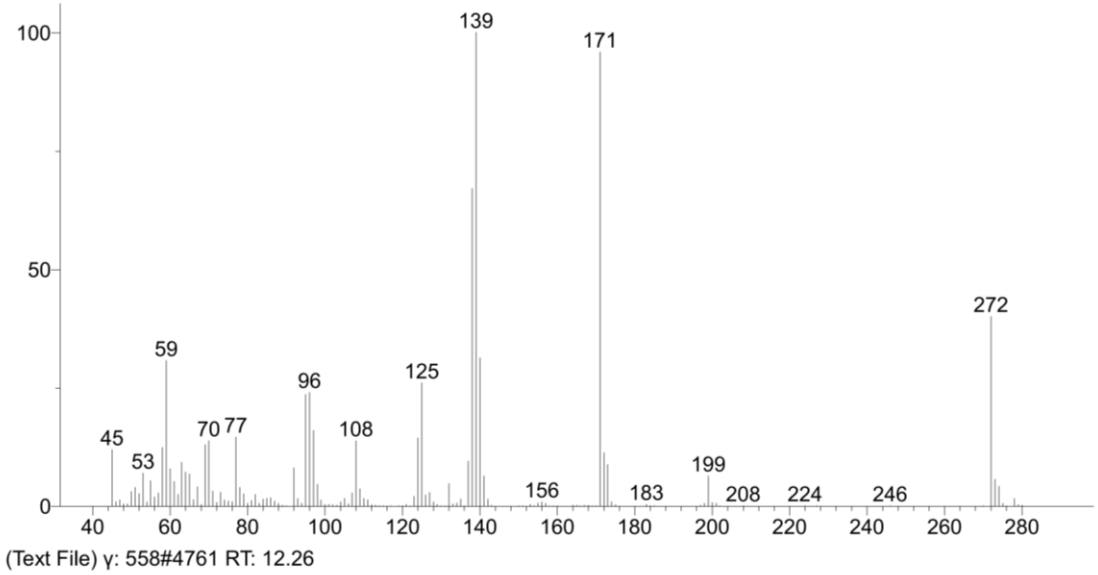
GC-MS of Compound **4-((4-chlorophenyl)disulfaneyl)phenol (3r)**

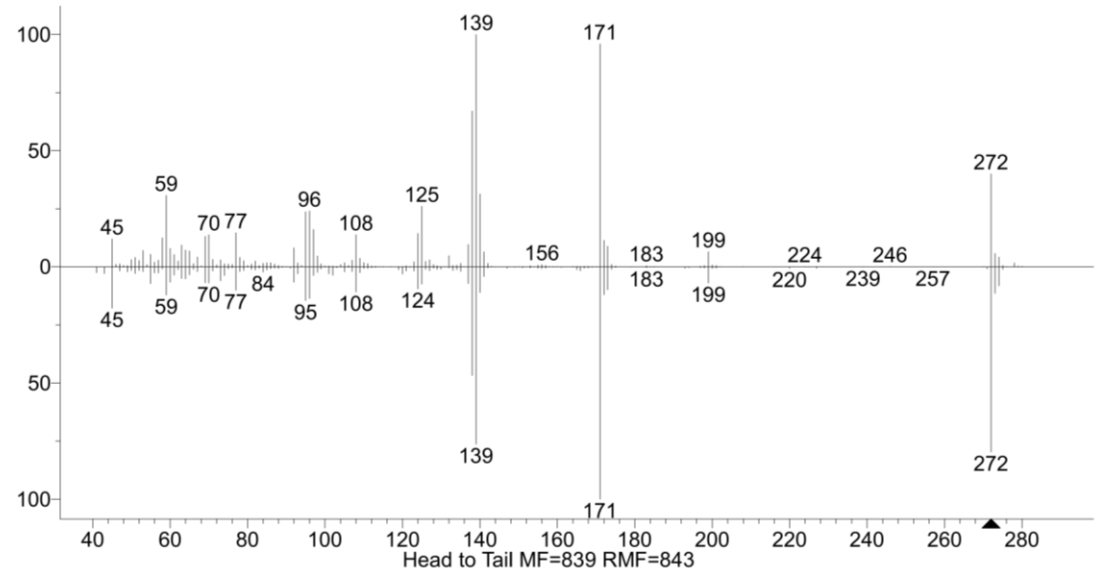


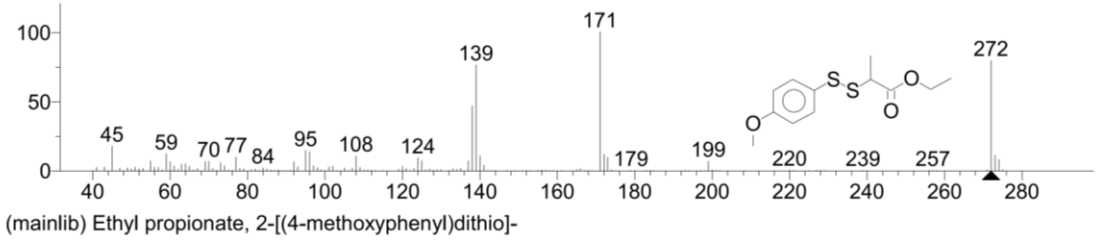

GC-MS of Compound **1-(tert-butyl)-2-(4-methoxyphenyl)disulfane (3t)**



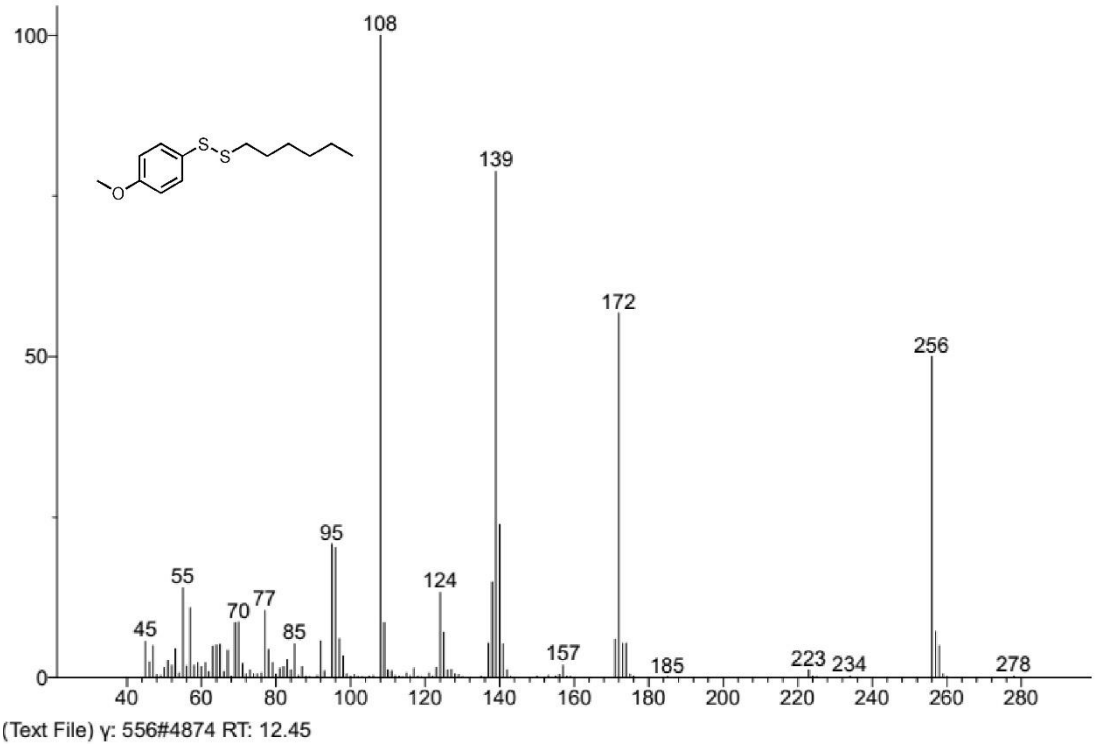
GC-MS of Compound **ethyl 2-((4-methoxyphenyl)disulfaneyl) propanoate (3s)**



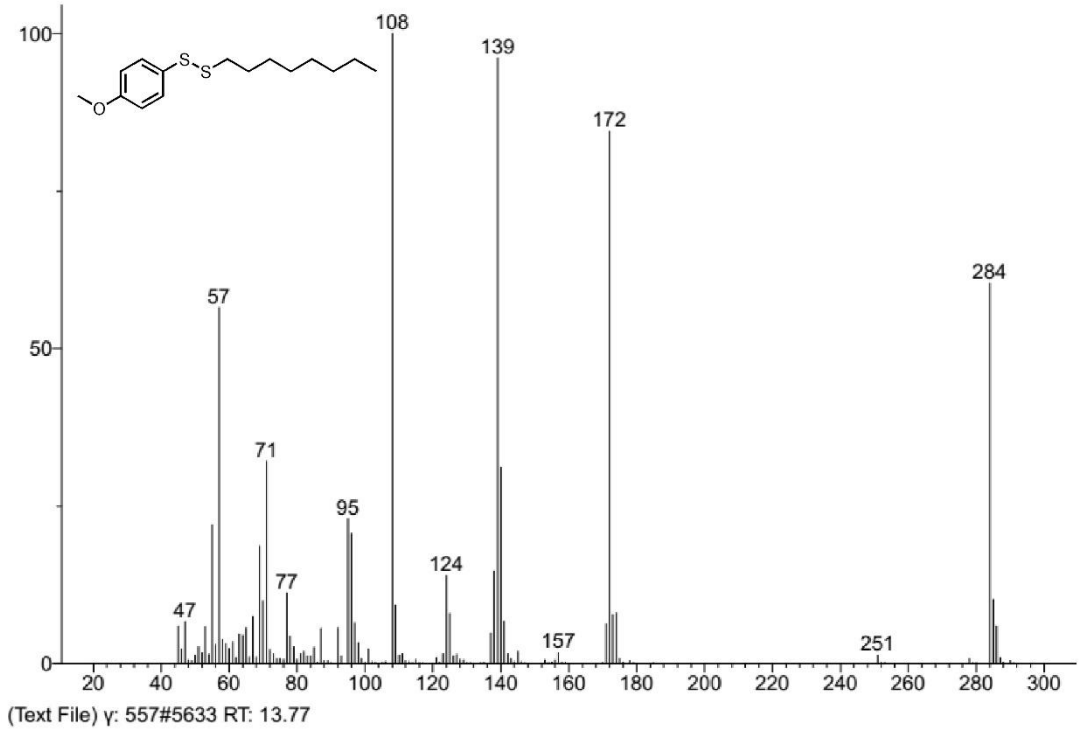



GC-MS of Compound **1-hexyl-2-(4-methoxyphenyl)disulfane (3u)**




GC-MS of Compound **1-(4-methoxyphenyl)-2-octyldisulfane (3v)**



# 5. References


1. C. Liu, Q. Chen, C. Cai and J. Xu, *Macromol. Chem. Phys.*, 2024, **225**, 2300394.
2. W.-B. Wu, Y.-C. Wong, Z.-K. Tan and J. Wu, *Catal. Sci. Technol.*, 2018, **8**, 4257-4263.
3. Y. Shen, H. Zhu, L. Deng, L. Yang, Y. Shen, Q. Fan, Z. Le and Z. Xie, *ACS Appl. Mater. Interfaces*, 2024, **16**, 56073-56081.
4. H. Xu, J.-L. Shi, S. Lyu, X. Lang, *Chin. J. Catal.*, 2020, **41**, 1468-1473.
5. M.-H. Sun, M.-Y. Qi, Z.-R. Tang and Y.-J. Xu, *Appl. Catal., B*, 2023, **321**, 122019.
6. X. Dong, H. Hao, F. Zhang and X. Lang, *J. Colloid Interface Sci.*, 2022, **622**, 1045-1053.