Supporting Information

Mechanically driven stainless steel-initiated activation of S-H bonds to construct disulfides

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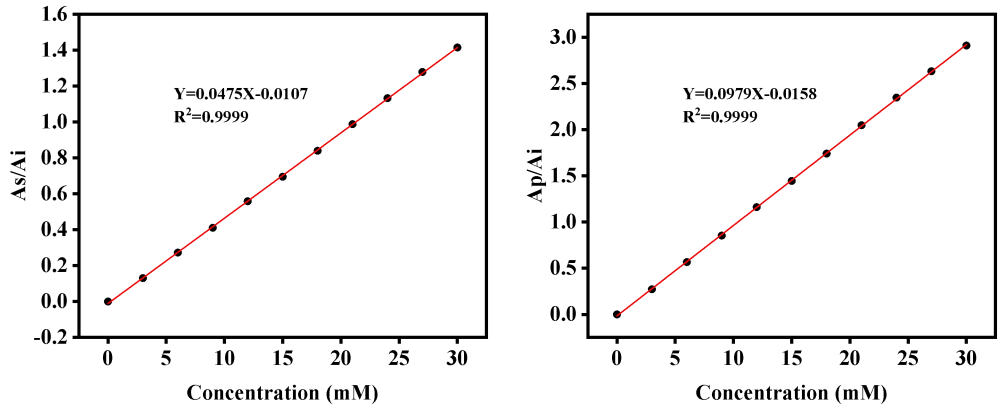
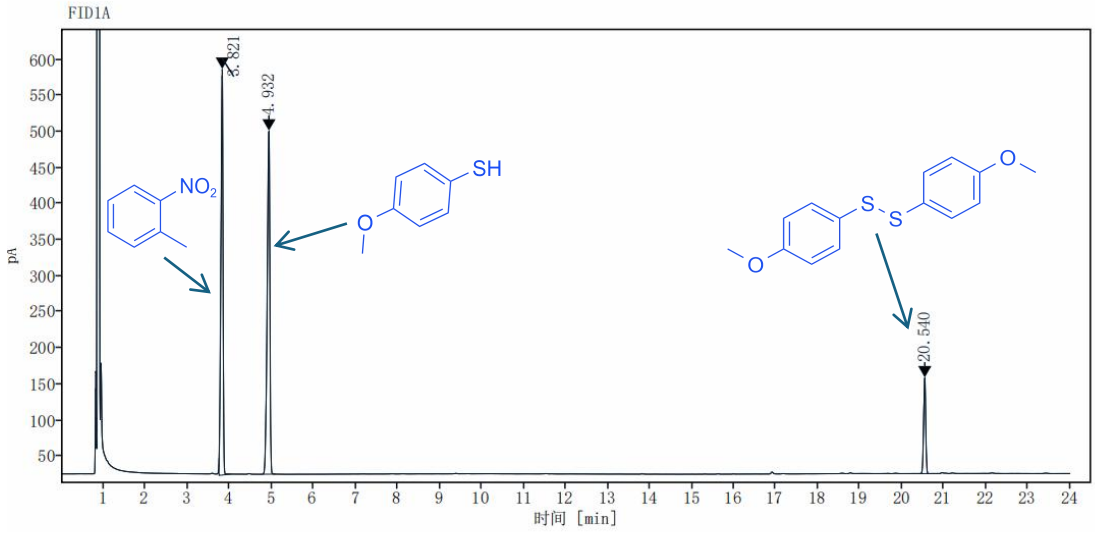


Figure S1 The standard curve of substrate (left) and product (right)

Use the internal standard method for quantitative analysis of reactants to minimize errors. A 20 mM solution of 2-nitrotoluene was employed as the internal standard to establish standard curves. A series of standard solutions with known concentrations (3 mM, 6 mM, 9 mM, 12 mM, 15 mM, 18 mM, 21 mM, 24 mM, 27 mM, and 30 mM) of the substrate 4-methoxyphenylthiophenol (**1a**) and the product bis(4-methoxyphenyl)disulfide (**2a**) were prepared. The standard curve is drawn by using the linear relationship between the peak area ratio of **1a (or 2a)** and the internal standard and the concentration. Finally, **1a (or 2a)** internal standard solutions of different concentrations were analysis by gas chromatography. Analysis method: HP-5 (30 m × 0.32 mm × 0.25 μm, the initial temperature was 90 °C for 0 min, then it was heated to 100 °C at a rate of 2 °C/min for 2 min, and then heated to 250 °C at a rate of 10 °C/min for 2 min).

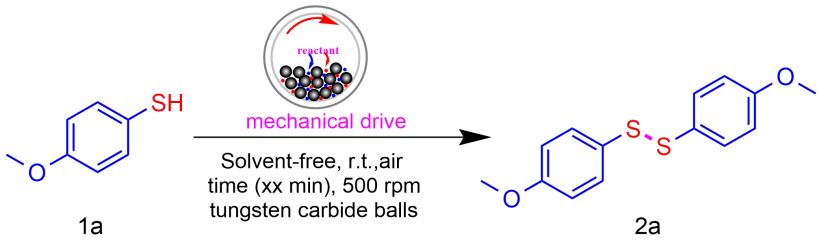


信号: FID1A

保留时间 [min]	类型	峰宽 [min]	峰面积	峰高	峰面积%	名称
3.821	MM m	0.2708	1990.7952	562.5773	43.8088	
4.932	MM m	0.3070	2094.0354	475.8050	46.0807	
20.540	BB	0.2344	459.4528	133.0826	10.1106	

Figure S2 GC chromatography of reaction solution with internal standard. Analysis method: HP-5 (30 m × 0.32 mm × 0.25 μm, the initial temperature was 90 °C for 0 min, then it was heated to 100 °C at a rate of 2 °C/min for 2 min, and then heated to 250 °C at a rate of 10 °C/min for 2 min).

Table S1 The reaction time screening.



Entry[a]	Time (min)	Conversion (%)-1a[b]]	Yield (%)-2a[b]
1	5	75.5	69.9
2	10	83.8	75.1
3	20	98.5	89.0
4	**30**	**98.5**	**92.2**
5	40	98.5	88.5
6	50	98.5	90.9
7	60	98.5	90.1
8	120	98.5	87.1
9	240	98.5	92.1

[a] Reaction conditions: **1a** (0.3 mmol), solvent-free, ten 8 mm and thirty 5 mm tungsten carbide balls were added into a 100 mL stainless steel jar, and the milling speed was 500 rpm, reaction at room temperature for xx min.

[b] Yields and conversions were determined by GC analysis with an internal standard.

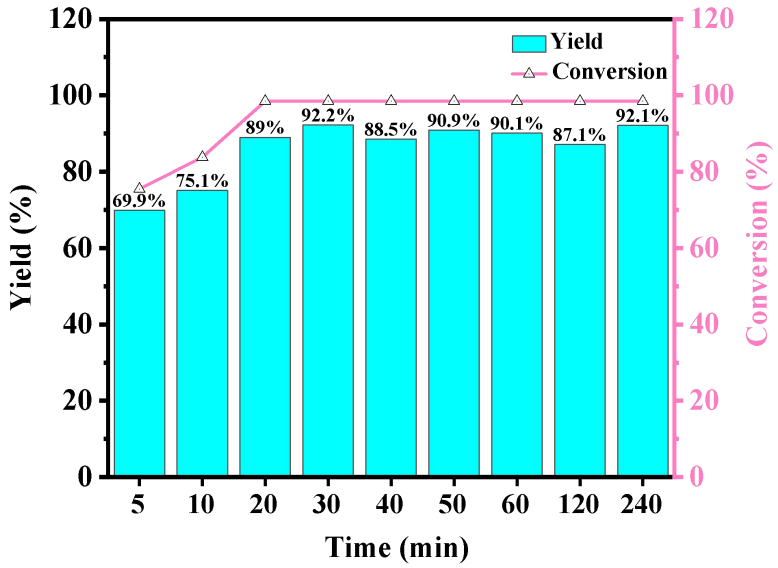
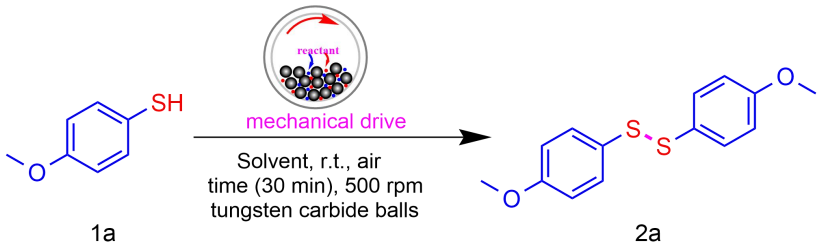


Figure S3 The reaction time screening.

Table S2 The reaction solvent screening.



1a 2a

Entry[a]	Solvent (mL)	Conversion (%)-1a[b]]	Yield (%)-2a[b]
1	**free**	**98.5**	**92.2**
2	H_2O (2)	98.5	88.7
3	EA (2)	48.8	40.3
4	MeCN (2)	98.5	88.9
5	MeOH (2)	98.5	93.2
6	DCM (2)	55.4	45.6
7	n-hexane (2)	83.5	79.8
8	acetone (2)	80.3	77.4
9	PE (2)	70.5	58.6
10	DMF (2)	98.5	83.4

[a] Reaction conditions: **1a** (0.3 mmol), solvent was xx (2 mL), ten 8 mm and thirty 5 mm tungsten carbide balls were added into a 100 mL stainless steel jar, and the milling speed was 500 rpm, reaction at room temperature for 30 min.

[b] Yields and conversions were determined by GC analysis with an internal standard.

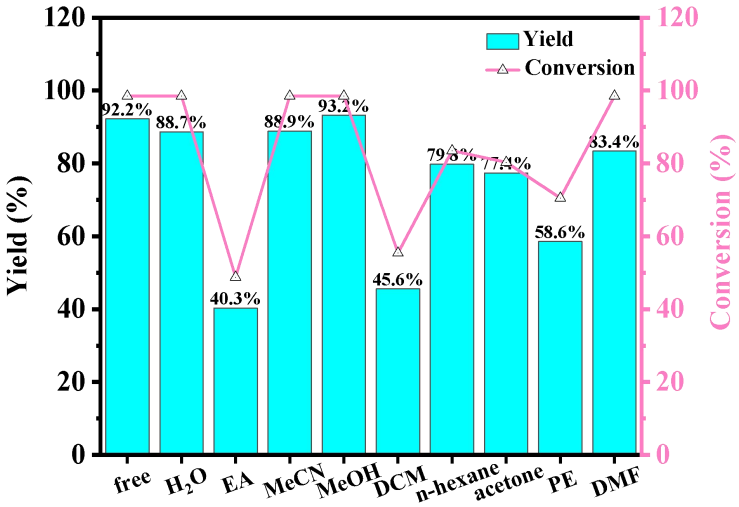
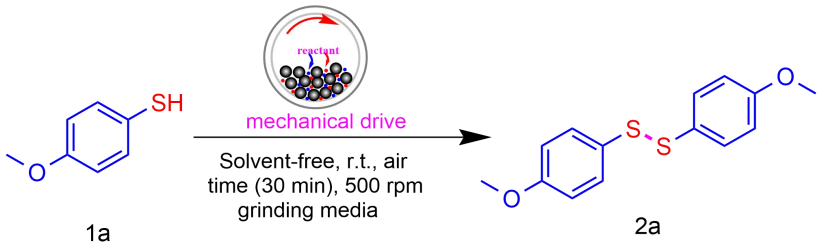


Figure S4 The reaction solvent screening.

Table S3 Screening of ball mill media types.



Entry[a]	Grinding Ball	Conversion (%)-1a[b]]	Yield (%)-2a[b]
1	Tungsten carbide balls	98.5	92.2
2[c]	Agate beads	21.3	19.1
3[c]	Zirconia balls	17.6	13.2

[a] Reaction conditions: **1a** (0.3 mmol), solvent-free, ten 8 mm and thirty 5 mm different grinding balls were added into a 100 mL stainless steel jar, and the milling speed was 500 rpm, reaction at room temperature for 30 min.

[b] Yields and conversions were determined by GC analysis with an internal standard.

[c] The ball mill jar used is an agate jar.

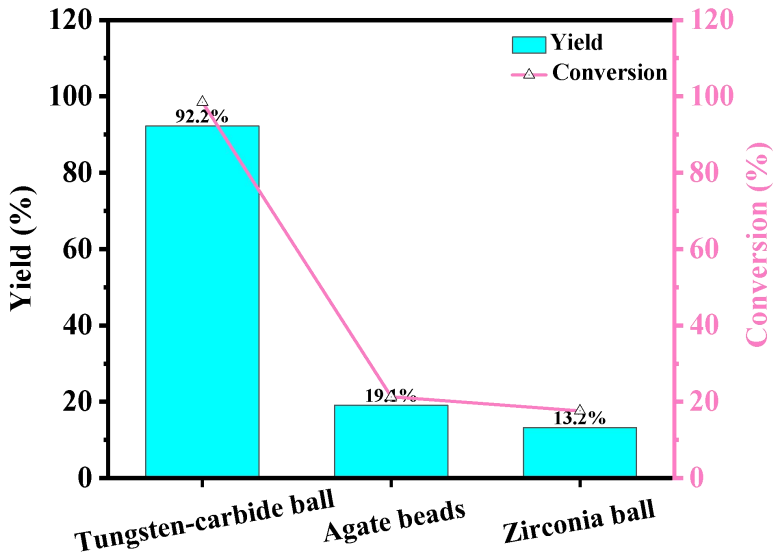
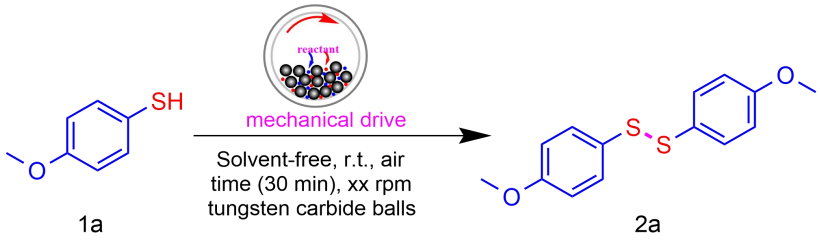


Figure S5 Screening of ball mill media types.

Table S4 Ball material ratio screening.



Entry[a]	Ball Material Ratio	Conversion (%)-1a[b]]	Yield (%)-2a[b]
1	300：1	31.9	31.3
2	600：1	67.1	65.5
3	900：1	96.8	95.3
4	1200：1	99.9	99.8
5	1500：1	97.8	97.4

[a] Reaction conditions: **1a** (0.3 mmol), solvent-free, x 8 mm and x 5 mm tungsten carbide balls were added into a 100 mL stainless steel jar, and the milling speed was 500 rpm, reaction at room temperature for 30 min.

[b] Yields and conversions were determined by GC analysis with an internal standard.

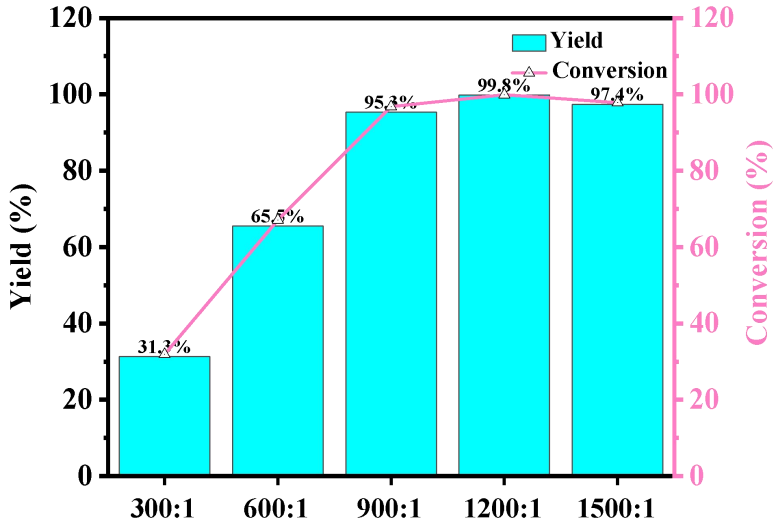
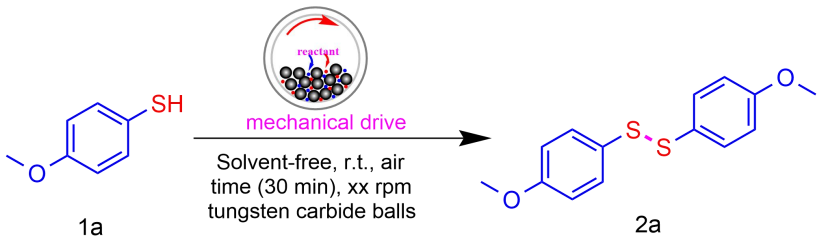


Figure S6 Ball material ratio screening.

Table S5 Ball mill speed screening.



Entry[a]	Speed (rpm)	Conversion (%)-1a[b]]	Yield (%)-2a[b]
1	100	64.8	59.5
2	200	72.1	61.6
3	300	85.4	86.3
4	400	99.0	97.0
5	500	99.9	99.8

[a] Reaction conditions: **1a** (0.3 mmol), solvent-free, eight 8 mm and twenty-four 5 mm tungsten carbide balls were added into a 100 mL stainless steel jar, and the milling speed was xx rpm, reaction at room temperature for 30 min.

[b] Yields and conversions were determined by GC analysis with an internal standard.

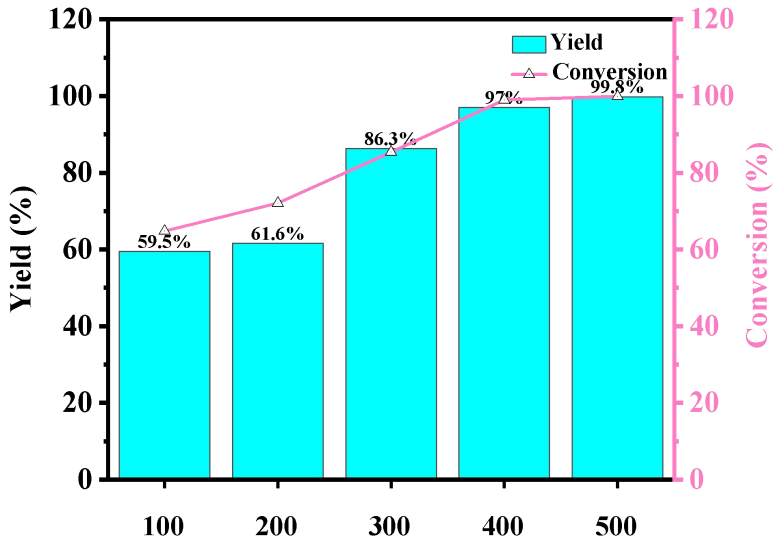
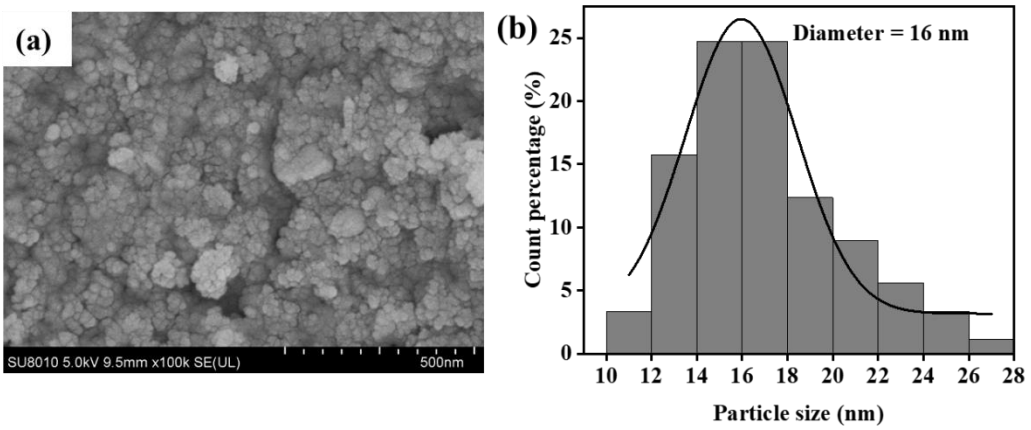


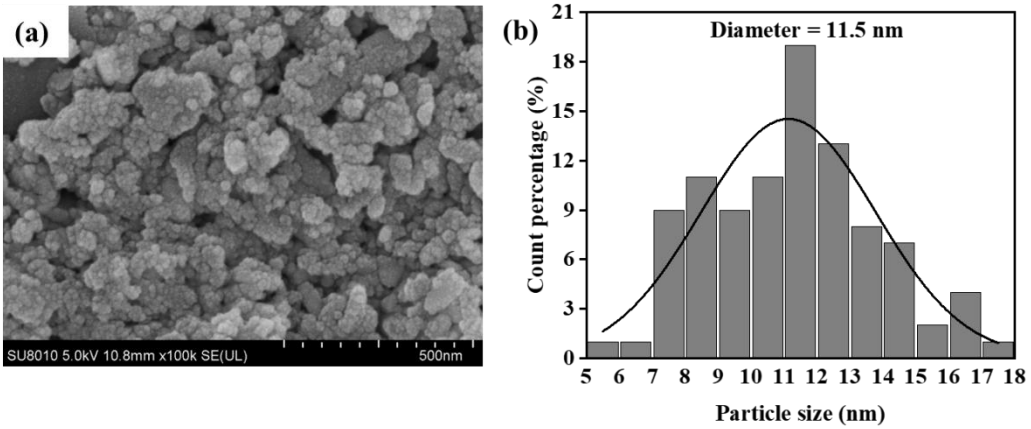
Figure S7 Ball mill speed screening.



112

113 **Figure S8** SEM image and particle size distribution of SS NPs before reaction.

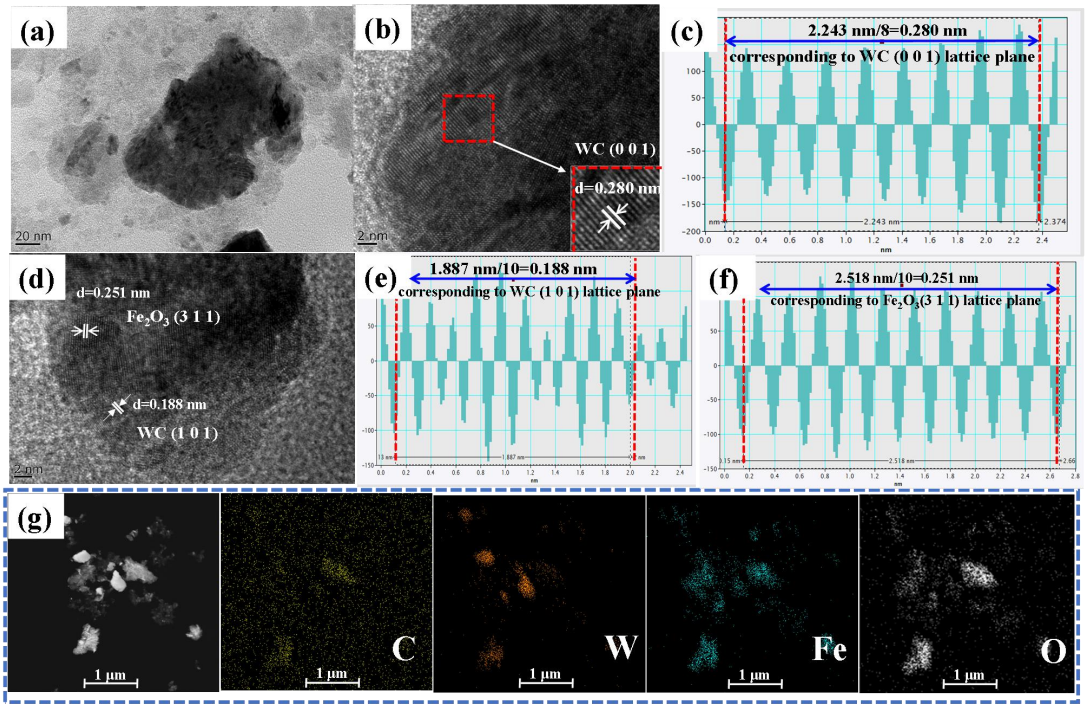
114



115

116 **Figure S9** SEM image and particle size distribution of SS NPs after reaction.

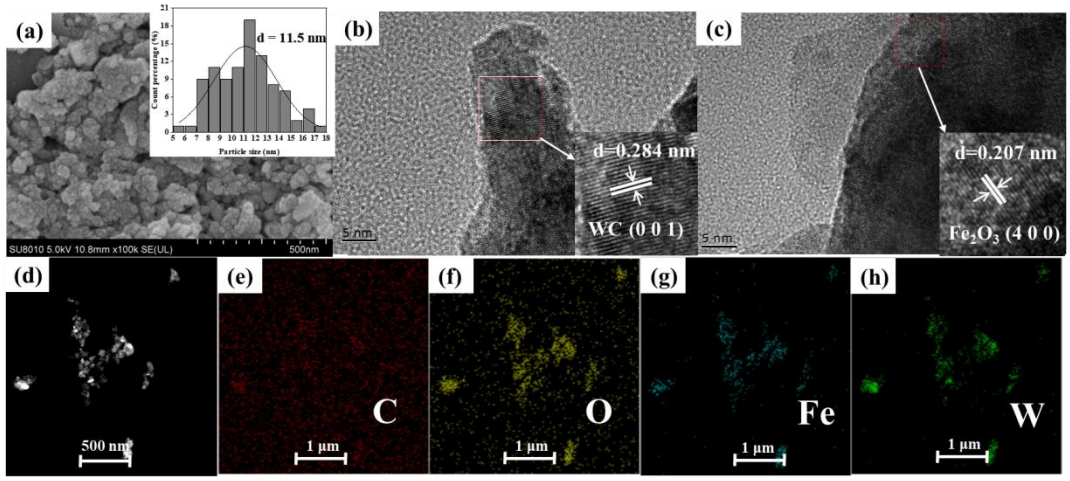
117



118

119 **Figure S10** SS NPs (a, b, d) HRTEM images and lattice fringe analysis at greater magnifications;
120 (c, e, f) Fast Fourier Transform plots (FFT); (g) Electronic image and elemental content
121 distribution of EDS spectra.

122



123

124 **Figure S11** SEM and TEM characterization of SS NPs after 5 cycles. (a) SEM images of SS NPs;
125 (b~c) HRTEM images and lattice fringe analysis of SS NPs; (d~h) Electronic image and elemental
126 distribution of EDS spectra.

127

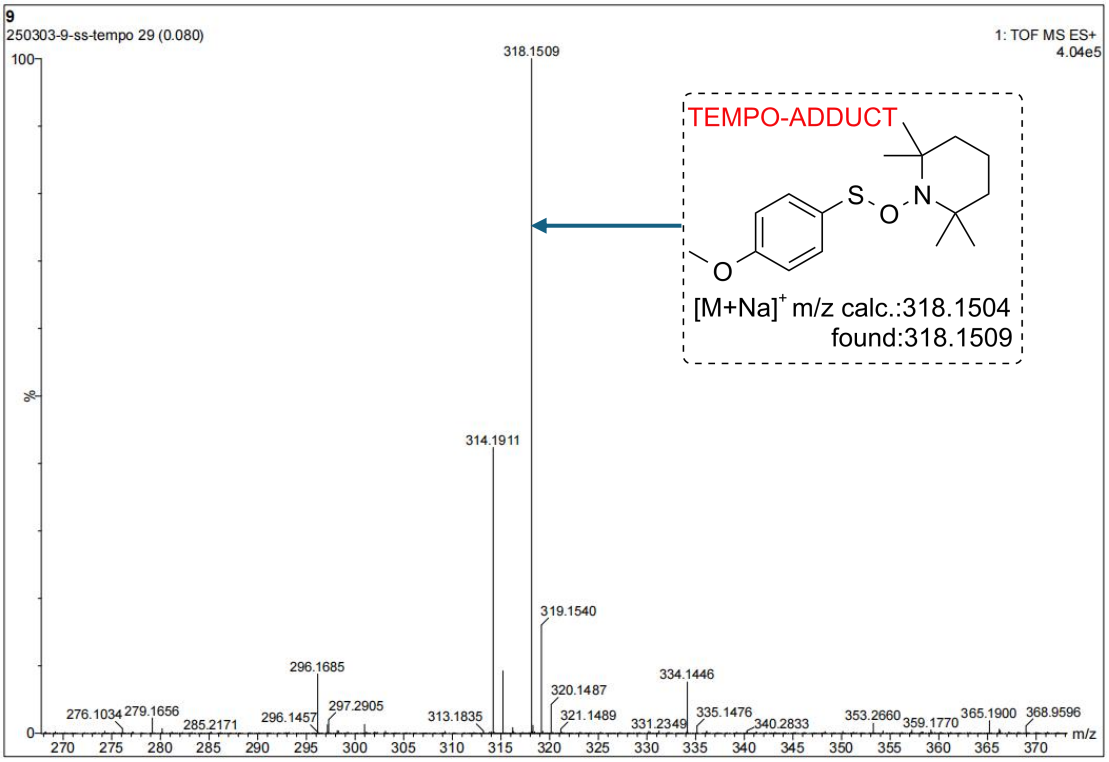
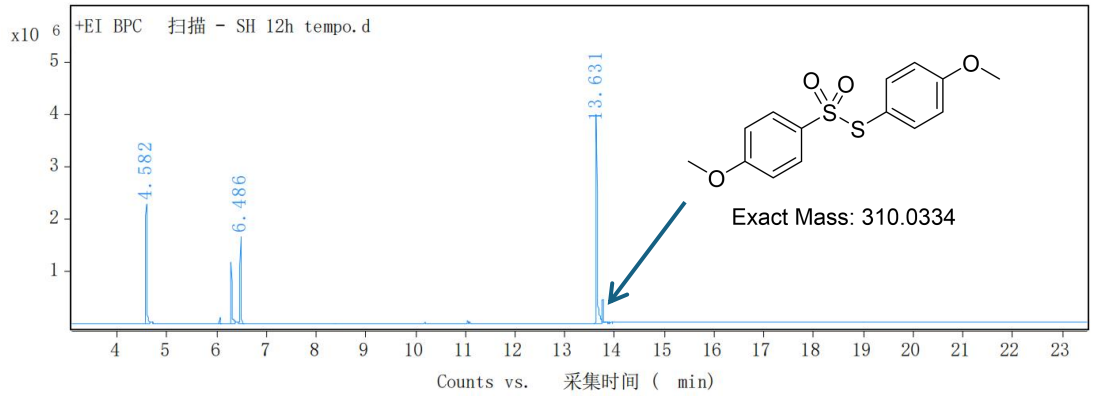


Figure S12 The HRMS of tempo-adduct: calcd. for $C_{16}H_{25}NO_2NaS$ $[M + Na]^+$ 318.1504, found 318.1509.



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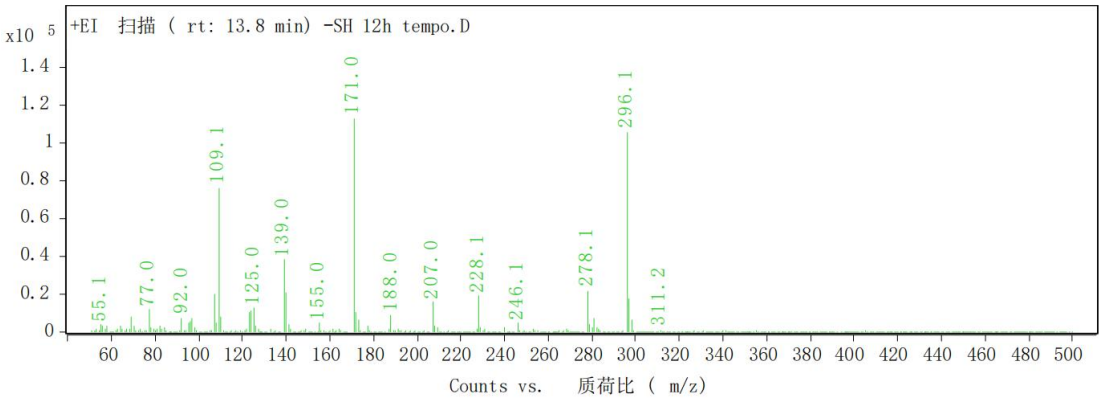


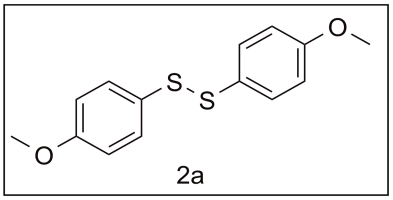
132

133 **Figure S13** GC-MS spectra of Tempo hyperoxidation of thiols to S-(4-methoxyphenyl)

134 4-methoxybenzenesulfonothioate byproducts.

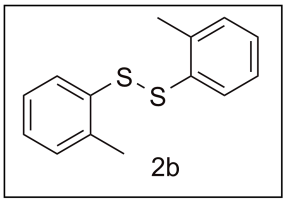
Spectroscopic Data

1,2-bis(4-methoxyphenyl)disulfane (2a): CAS Number 5335-87-5.



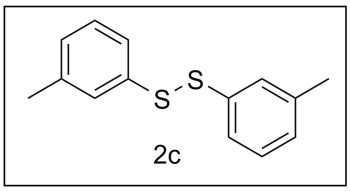
Following the general procedure, the title compound was isolated as a yellow solid (Yield 98%, 41.0 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.40 (d, *J* = 8.8 Hz, 4H), 6.84 (d, *J* = 8.8 Hz, 4H), 3.80 (s, 6H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 160.0, 132.8, 128.5, 114.7, 55.5.

1,2-di-o-tolyldisulfane (2b): CAS Number 4032-80-8.



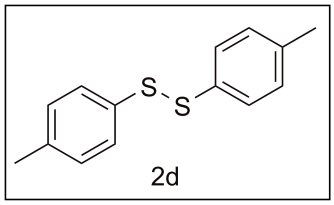
Following the general procedure, the title compound was isolated as a yellow brown solid (Yield 91%, 33.6 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.53 (dd, *J* = 5.6 Hz, 2H), 7.19-7.12 (m, 6H), 2.45 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 137.3, 135.5, 130.4, 128.5, 127.4, 126.8, 20.1.

1,2-di-m-tolyldisulfane (2c): CAS Number 20333-41-9.



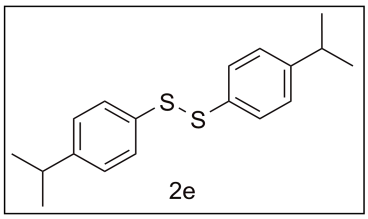
Following the general procedure, the title compound was isolated as a yellow solid (Yield 94%, 34.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.33–7.31 (m, 4H), 7.22-7.18 (m, 2H), 7.06–7.04 (m, 2H), 2.34 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 139.1, 137.0, 129.0, 128.1, 128.0, 124.6, 21.5.

1,2-di-p-tolyldisulfane (2d): CAS Number 103-19-5.



Following the general procedure, the title compound was isolated as a yellow solid (Yield 88%, 32.6 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.40 (d, *J* = 8.4 Hz, 4H), 7.12 (d, *J* = 8.0 Hz, 4H), 2.34 (s, 6H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 137.5, 134.0, 129.9, 128.6, 21.2.

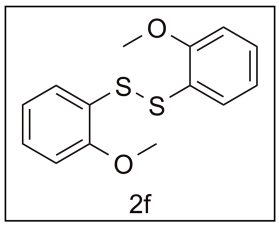
1,2-bis(4-isopropylphenyl)disulfane (2e): CAS Number 622407-64-1.



Following the general procedure, the title compound was isolated as a yellow oil liquid (Yield 91%, 41.1

mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.45 (d, *J* = 8.4 Hz, 4H), 7.19 (d, *J* = 8.0 Hz, 4H), 2.95-2.86 (m, 2H), 1.24 (d, *J* = 7.2 Hz, 12H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 148.4, 134.4, 128.3, 127.4, 33.9, 24.0.

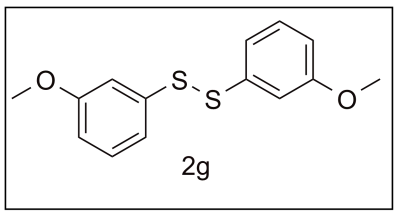
1,2-bis(2-methoxyphenyl)disulfane (2f): CAS Number 59014-89-0.



2f

Following the general procedure, the title compound was isolated as a white solid (Yield 95%, 39.8 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.54 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.22-7.17 (m, 2H), 6.92 (td, *J* = 7.6, 1.2 Hz, 2H), 6.86 (dd, *J* = 8.0, 1.2 Hz, 2H), 3.90 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 156.6, 127.8, 127.6, 124.6, 121.4, 110.5, 56.0.

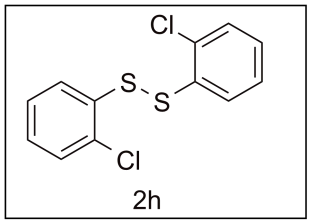
1,2-bis(3-methoxyphenyl)disulfane (2g): CAS Number 59014-89-0.



2g

Following the general procedure, the title compound was isolated as a yellow liquid (Yield 97%, 40.3 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.22 (t, *J* = 8.0 Hz, 2H), 7.10–7.07 (m, 4H), 6.78-6.75 (m, 2H), 3.77 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 160.1, 138.4, 130.0, 119.6, 113.2, 112.6, 55.4.

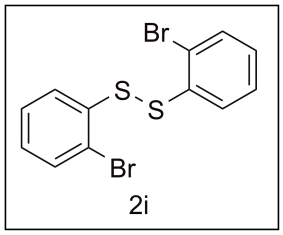
1,2-bis(2-chlorophenyl)disulfane (2h): CAS Number 31121-19-4.



2h

Following the general procedure, the title compound was isolated as a white solid (Yield 88%, 37.8 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.55 (dt, *J* = 8.0, 1.2 Hz, 2H), 7.37 (dt, *J* = 7.6, 1.2 Hz, 2H), 7.25-7.20 (m, 2H), 7.18-7.14 (m, 2H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 134.4, 131.9, 129.8, 127.9, 127.7, 127.2.

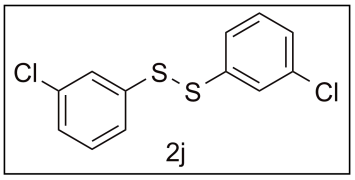
1,2-bis(2-bromophenyl)disulfane (2i): CAS Number 71112-91-9.



2i

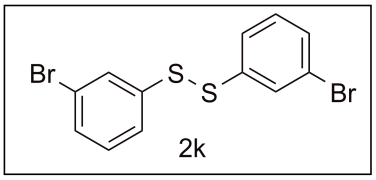
Following the general procedure, the title compound was isolated as a white solid (Yield 87%, 48.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.55-7.51 (m, 4H), 7.29–7.25 (m, 2H), 7.08 (td, *J* = 7.6, 1.6 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$)： δ 136.2, 133.0, 128.3, 128.0, 126.9, 121.1.

1,2-bis(3-chlorophenyl)disulfane (2j): CAS Number 19742-92-8.



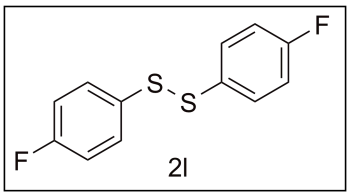
Following the general procedure, the title compound was isolated as a yellow liquid (Yield 89%, 38.3 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.47 (s, 2H), 7.35 (dt, *J* = 7.6, 1.6 Hz, 2H), 7.24–7.19 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 138.5, 135.2, 130.3, 127.7, 127.1, 125.4.

1,2-bis(3-bromophenyl)disulfane (2k)： CAS Number 19742-90-6.



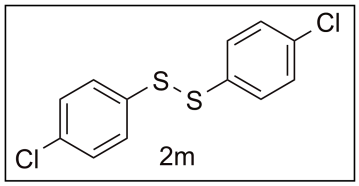
Following the general procedure, the title compound was isolated as a yellow oil liquid (Yield 84%, 47.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.62 (d, *J* = 1.2 Hz, 2H), 7.41–7.35 (m, 4H), 7.18 (t, *J* = 8.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 138.7, 130.6, 130.6, 129.9, 125.9, 123.3.

1,2-bis(4-fluorophenyl)disulfane (2l): CAS Number 405-31-2.



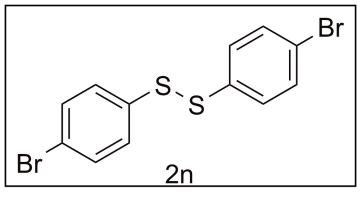
Following the general procedure, the title compound was isolated as a yellow liquid (Yield 99.6%, 38.0 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.47–7.43 (m, 4H), 7.04–6.99 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$)： δ 162.7 ($J_{C\text{-}F}$ = 249.5 Hz), 132.2 ($J_{C\text{-}F}$ = 3.0 Hz), 131.4 ($J_{C\text{-}F}$ = 8.1 Hz), 116.4 ($J_{C\text{-}F}$ = 22.2 Hz).

1,2-bis(4-chlorophenyl)disulfane (2m): CAS Number 1142-19-4.



Following the general procedure, the title compound was isolated as a yellowish-white solid (Yield 90%, 38.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.41–7.38 (m, 4H), 7.29–7.25 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 135.2, 133.7, 129.4, 129.4.

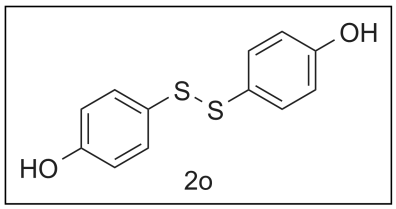
1,2-bis(3-bromophenyl)disulfane (2n): CAS Number 5335-84-2.



Following the general procedure, the title compound was isolated as a yellowish-white solid (Yield 84%, 47.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.44–7.41 (m, 4H), 7.34 –7.32 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$)：

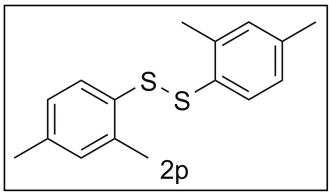
δ 135.8, 132.3, 129.4, 121.6.

4,4'-disulfanediyldiphenol (2o): CAS Number 15015-57-3.



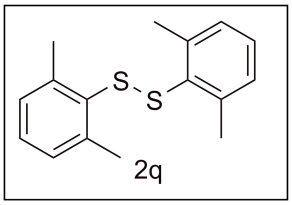
Following the general procedure, the title compound was isolated as a white to light yellow solid (Yield 87%, 32.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.27–7.24 (m, 4H), 6.74–6.72 (m, 4H), 4.90 (s, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 158.1, 133.3, 126.8, 115.6.

1,2-bis(2,4-dimethylphenyl)disulfane (2p)： CAS Number 13616-83-6.



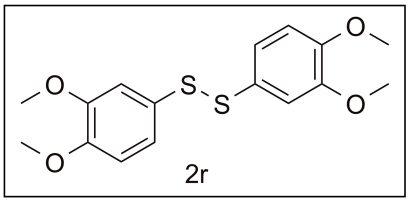
Following the general procedure, the title compound was isolated as a yellow liquid (Yield 89%, 37.8 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.40 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 2.0 Hz, 2H), 6.95 (dd, *J* = 8.0, 2.0 Hz, 2H), 2.39 (s, 6H), 2.31 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 138.3, 137.9, 132.4, 131.3, 130.4, 127.5, 21.1, 20.3.

1,2-bis(2,6-dimethylphenyl)disulfane (2q)： CAS Number 2905-17-1.



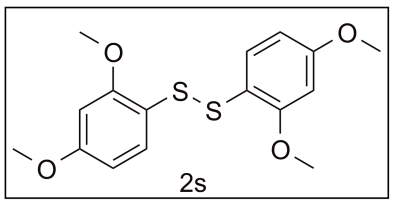
Following the general procedure, the title compound was isolated as a white solid (Yield 86%, 35.5 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.12 (dd, *J* = 8.0, 6.8 Hz, 2H), 7.02 (d, *J* = 7.6 Hz, 4H), 2.24 (s, 12H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 143.5, 134.8, 129.4, 128.2, 21.6.

1,2-bis(3,4-dimethoxyphenyl)disulfane (2r)： CAS Number 66086-38-2.



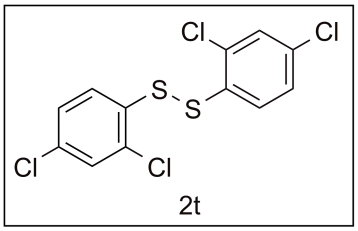
Following the general procedure, the title compound was isolated as a yellow solid (Yield 93%, 55.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)： δ 7.03 (dt, *J* = 8.0, 2.0 Hz, 2H), 7.00–6.99 (m, 2H), 6.77 (d, *J* = 8.4 Hz, 2H), 3.86 (s, 6H), 3.82 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 149.5, 149.1, 128.7, 124.0, 114.1, 111.3, 56.0, 55.9.

1,2-bis(2,5-dimethoxyphenyl)disulfane (2s)： CAS Number 29945-69-5.



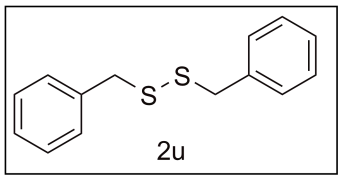
Following the general procedure, the title compound was isolated as a yellow solid (Yield 99.9%, 50.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.15 (d, *J* = 2.8 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.68 (dd, *J* = 8.8, 2.8 Hz, 2H), 3.85 (s, 6H), 3.70 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 154.3, 150.9, 125.7, 113.5, 112.4, 111.7, 56.6, 55.8.

1,2-bis(2,4-dichlorophenyl)disulfane (2t)： CAS Number 15433-50-8.



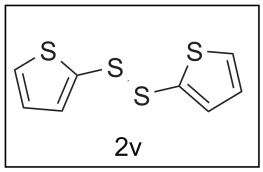
Following the general procedure, the title compound was isolated as a white solid (Yield 90%, 48.0 mg). **^{1}H NMR** (400 MHz, $CDCl_3$)：δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 2.0 Hz, 2H), 7.21 (dd, *J* = 8.4, 2.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$)：δ 133.6, 132.9, 132.8, 129.7, 128.6, 128.1.

1,2-dibenzyldisulfane (2u)： CAS Number 150-60-7.



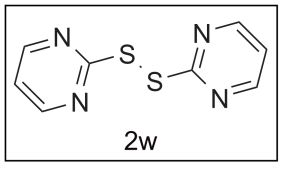
Following the general procedure, the title compound was isolated as a colorless oil liquid (Yield 88%, 32.5 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)： δ 7.37–7.24 (m, 10H), 3.61 (s, 4H). **^{13}C-NMR** (101 MHz, $CDCl_3$)： δ 137.4, 129.5, 128.6, 127.6, 43.3.

1,2-di(thiophen-2-yl)disulfane (2v): CAS Number 6911-51-9.



Following the general procedure, the title compound was isolated as a yellow solid (Yield 72%, 24.9 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 5.6, 1.6 Hz, 2H), 7.16 (dd, *J* = 3.6, 1.2 Hz, 2H), 7.02 (dd, *J* = 5.2, 3.6 Hz, 2H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 135.8, 135.8, 132.4, 127.9.

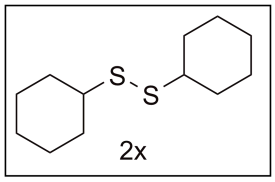
1,2-di(pyrimidin-2-yl)disulfane (2w): CAS Number 15718-46-4.



Following the general procedure, the title compound was isolated as a white solid (Yield 41%, 13.5 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 8.57 (d, *J* = 4.8 Hz, 4H), 7.08 (t, *J* =

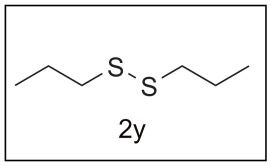
4.8 Hz, 2H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 169.8, 158.0, 118.3.

1,2-dicyclohexyldisulfane (2x): CAS Number 2550-40-5.



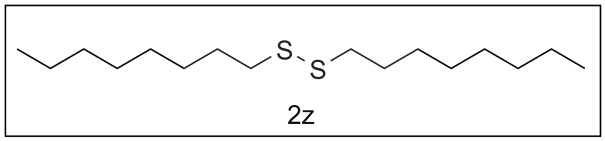
Following the general procedure the title compound was isolated as a yellow liquid (Yield 68%, 23.3 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 2.71-2.64 (m, 2H), 2.06-2.01 (m, 4H), 1.80-1.75 (m, 4H), 1.63 – 1.59 (m, 2H), 1.33-1.25 (m, 10H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 50.1, 33.0, 26.2, 25.8.

1,2-dipropyldisulfane (2y): CAS Number 629-19-6.



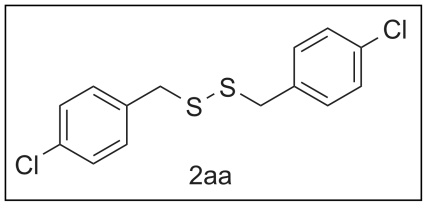
Following the general procedure the title compound was isolated as a colorless to pale yellow liquid (Yield 99%, 22.3 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 2.66 (t, J = 7.2 Hz, 4H), 1.75-1.66 (m, 4H), 0.99 (t, J = 7.6 Hz, 6H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 41.3, 22.6, 13.3.

1,2-dioctyldisulfane (2z): CAS Number 822-27-5.



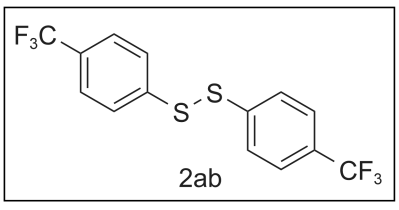
Following the general procedure the title compound was isolated as a yellow-brown liquid (Yield 82%, 35.6 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 2.68 (t, J = 7.2 Hz, 4H), 1.70-1.63 (m, 4H), 1.37 (t, J = 8.0 Hz, 4H), 1.30-1.27 (m, 16H), 0.88 (t, J = 6.4 Hz, 6H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 39.3, 32.0, 29.4, 29.3, 29.3, 28.7, 22.8, 14.2.

1,2-bis(4-chlorobenzyl)disulfane (2aa): CAS Number 23566-17-8.



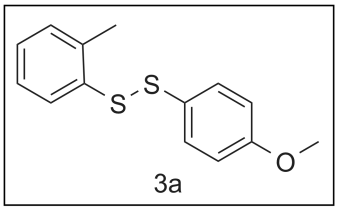
Following the general procedure the title compound was isolated as a colorless oily liquid (Yield 32%, 30.0 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.31–7.29 (m, 4H), 7.17–7.15 (m, 4H), 3.58 (s, 4H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 136.0, 133.5, 130.8, 128.8, 42.6.

1,2-bis(4-(trifluoromethyl)phenyl)disulfane (2ab): CAS Number 18715-45-2.



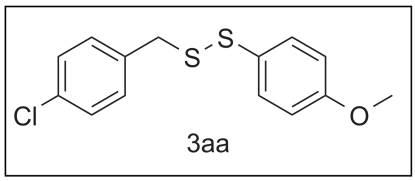
Following the general procedure the title compound was isolated as a white solid (Yield 21%, 21.9 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.60-7.56 (m, 8H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 140.9, 129.6 (q, $J_{C\text{-}F}$ = 32.3 Hz), 126.7, 126.3 (q, $J_{C\text{-}F}$ = 4.04 Hz), 124.0 (q, $J_{C\text{-}F}$ = 272.7 Hz).

1-(4-methoxyphenyl)-2-(o-tolyl)disulfane (3a): CAS Number 2445788-86-1.



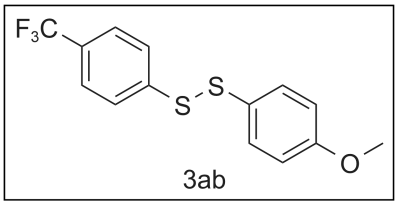
Following the general procedure the title compound was isolated as a yellow oily liquid (Yield 33%, 26.3 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.63–7.59 (m, 1H), 7.43–7.39 (m, 2H), 7.17 (d, J = 3.2 Hz, 3H), 6.84–6.81 (m, 2H), 3.79 (s, 3H), 2.39 (s, 3H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 160.0, 137.8, 136.1, 132.3, 130.5, 129.4, 128.0, 127.6, 126.7, 114.8, 55.5, 20.2.

1-(4-chlorobenzyl)-2-(4-methoxyphenyl)disulfane (3aa): CAS Number 2492436-80-1.

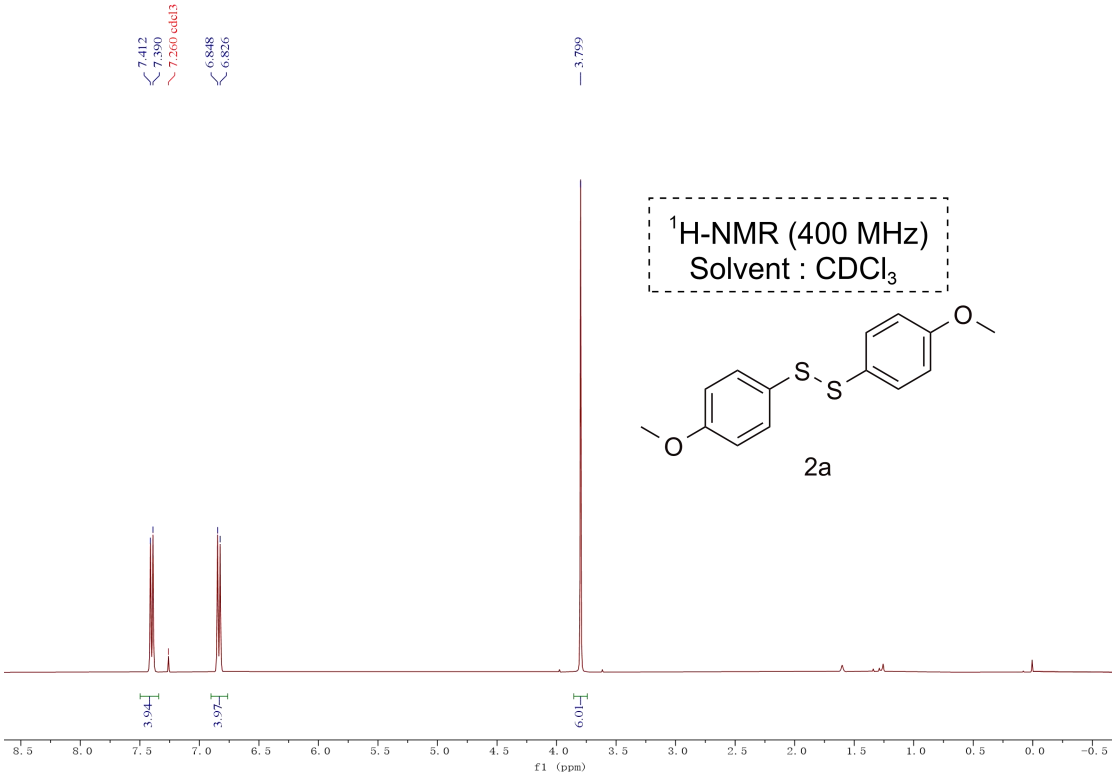


Following the general procedure the title compound was isolated as a yellow oily liquid (Yield 33%, 29.6 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$)：δ 7.35 (d, J = 8.4 Hz, 2H), 7.24–7.17 (m, 4H), 6.83–6.81 (m, 2H), 3.89 (s, 2H), 3.81 (s, 3H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 159.8, 135.5, 133.4, 132.2, 130.9, 128.7, 127.8, 114.7, 55.5, 42.5.

1-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)disulfane (3ab): CAS Number 1670249-61-2.



Following the general procedure the title compound was isolated as a yellow oily liquid (Yield 46%, 43.6 mg). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.63 (d, J = 7.6 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 7.45–7.41 (m, 2H), 6.86–6.83 (m, 2H), 3.79 (s, 3H). **^{13}C-NMR** (101 MHz, $CDCl_3$)：δ 160.2, 142.5 (d, $J_{C\text{-}F}$ = 2.02 Hz), 132.0, 129.0 (q, $J_{C\text{-}F}$ = 33.3 Hz), 127.2, 127.1, 126.0 (q, $J_{C\text{-}F}$ = 4.04 Hz), 124.2 (q, $J_{C\text{-}F}$ = 272.7 Hz), 115.0, 55.5.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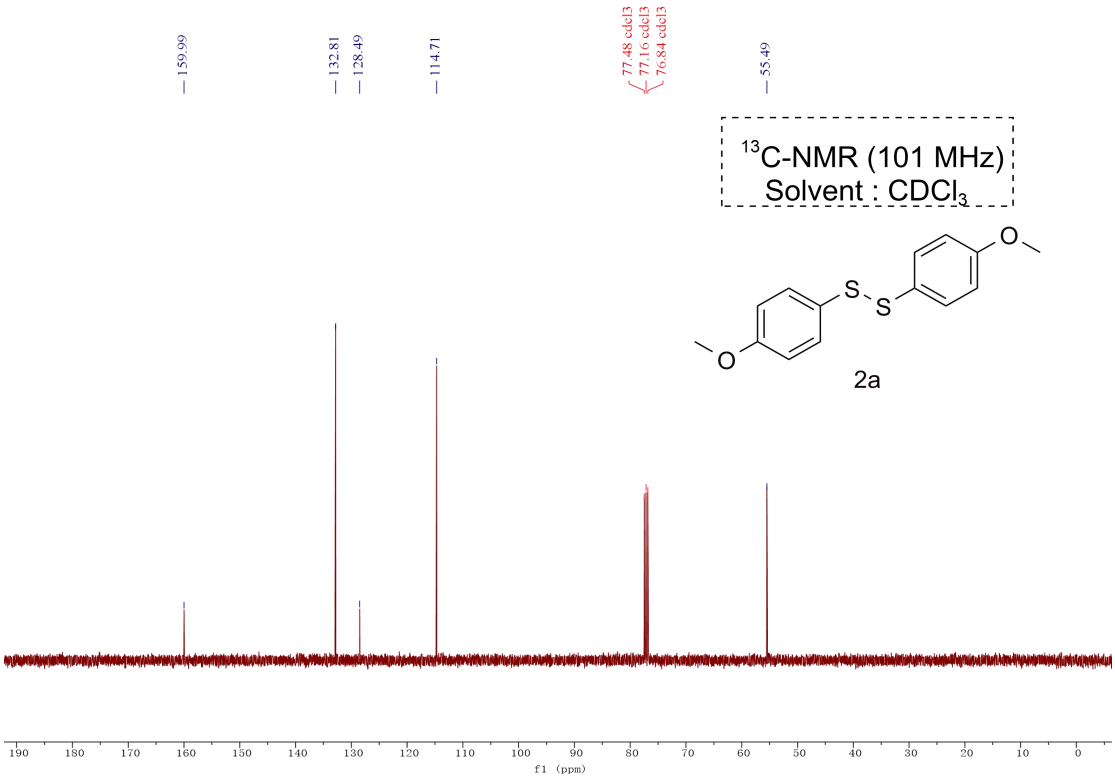
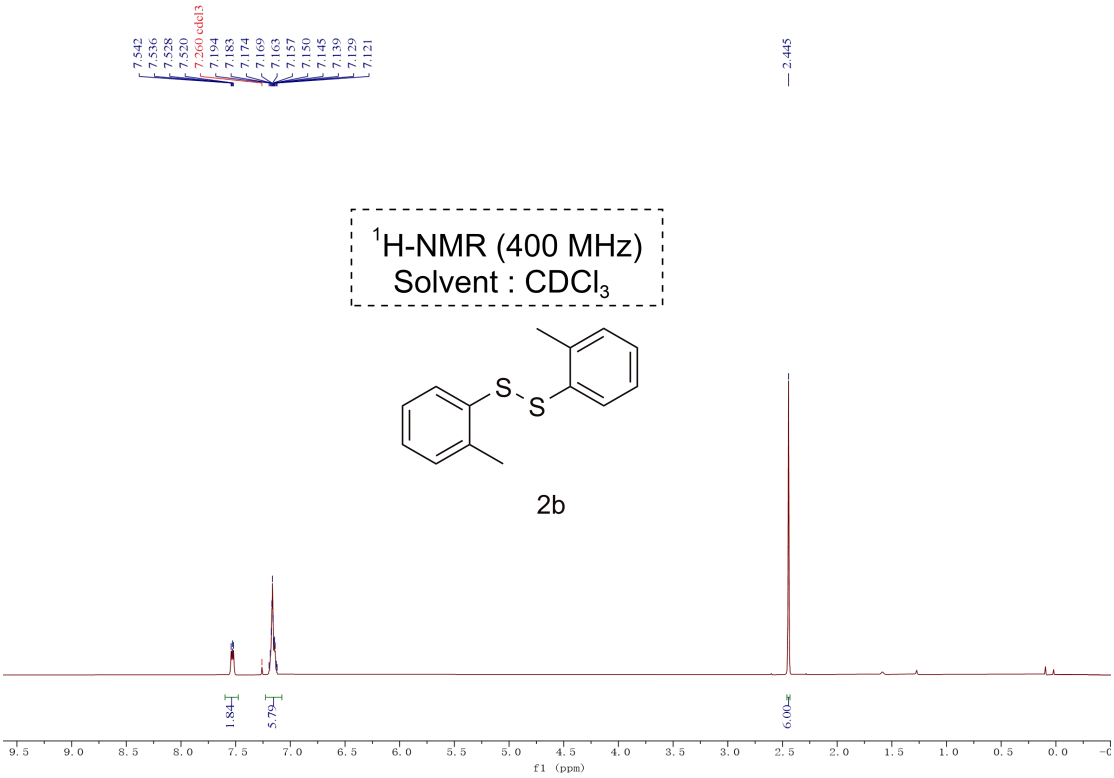
7.390
7.260 cdcl3
6.848
6.826
3.799
3.94
3.97
6.01
f1 (ppm)
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$
S
S
O
O
2a


159.99
132.81
128.49
114.71
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
55.49
f1 (ppm)
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$
S
S
O
O
2a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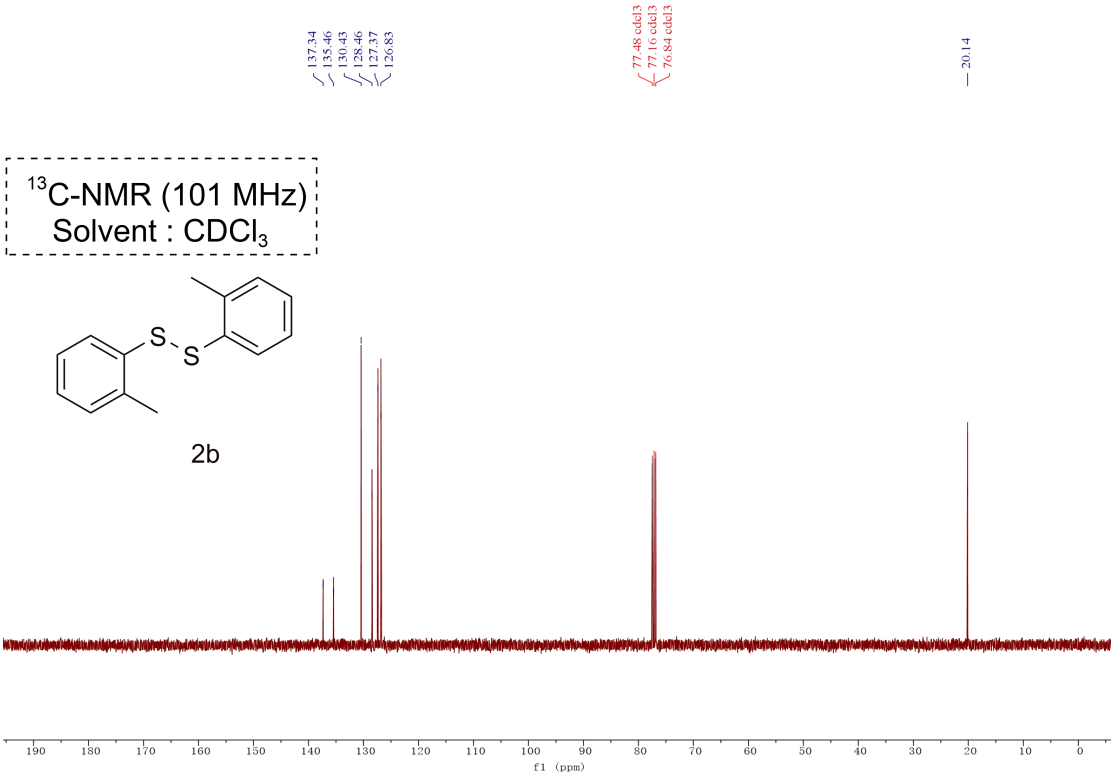
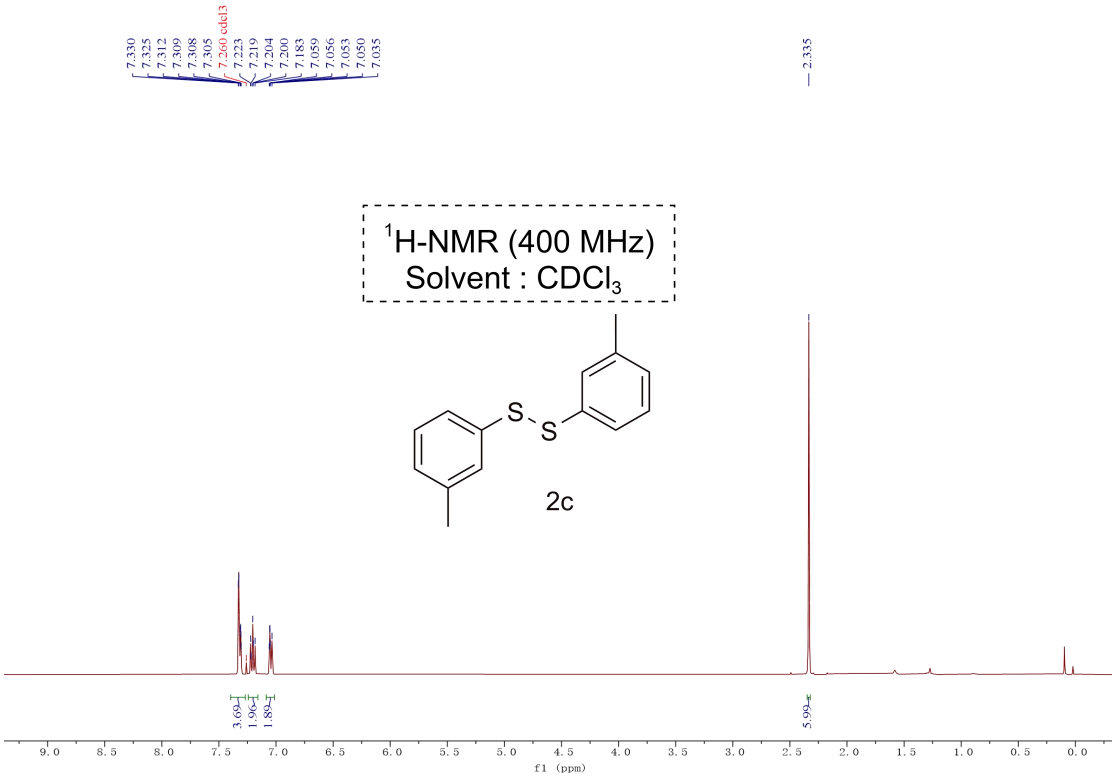
7.536
7.528
7.520
7.260 cdcl3
7.194
7.183
7.174
7.169
7.163
7.157
7.150
7.145
7.139
7.129
7.121
2.445
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2b
1.84
5.79
6.00
f1 (ppm)


137.34
135.46
130.43
128.46
127.37
126.83
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
20.14
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2b
f1 (ppm)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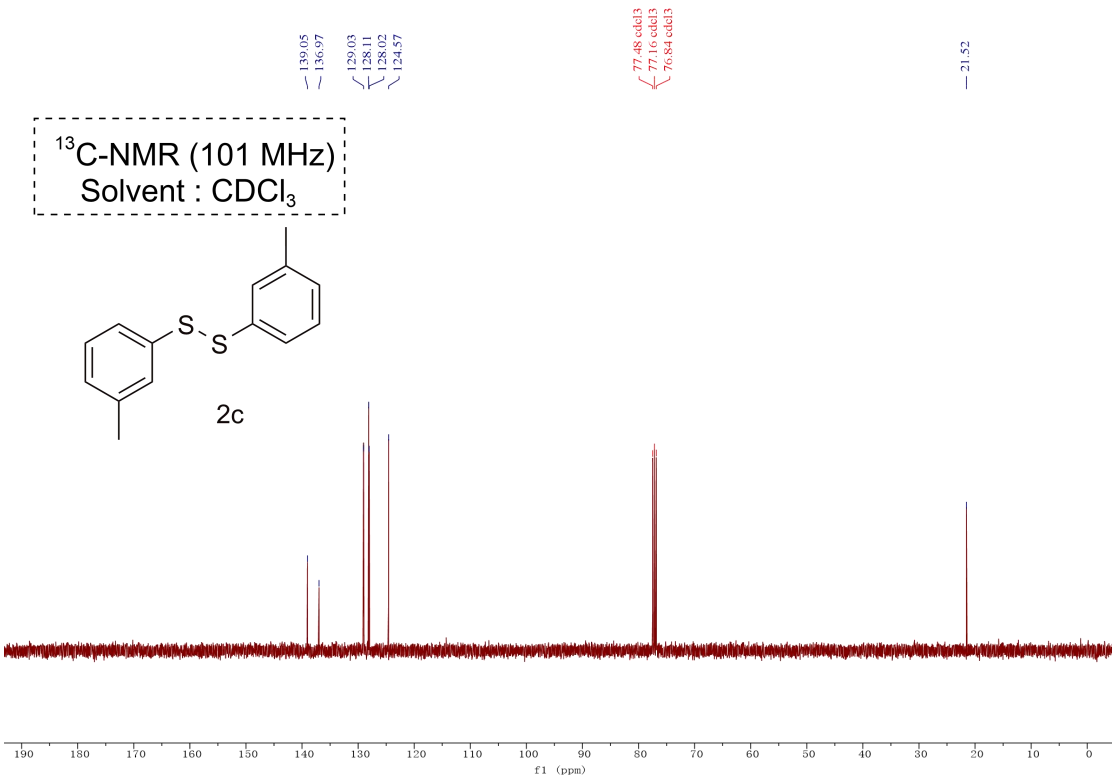
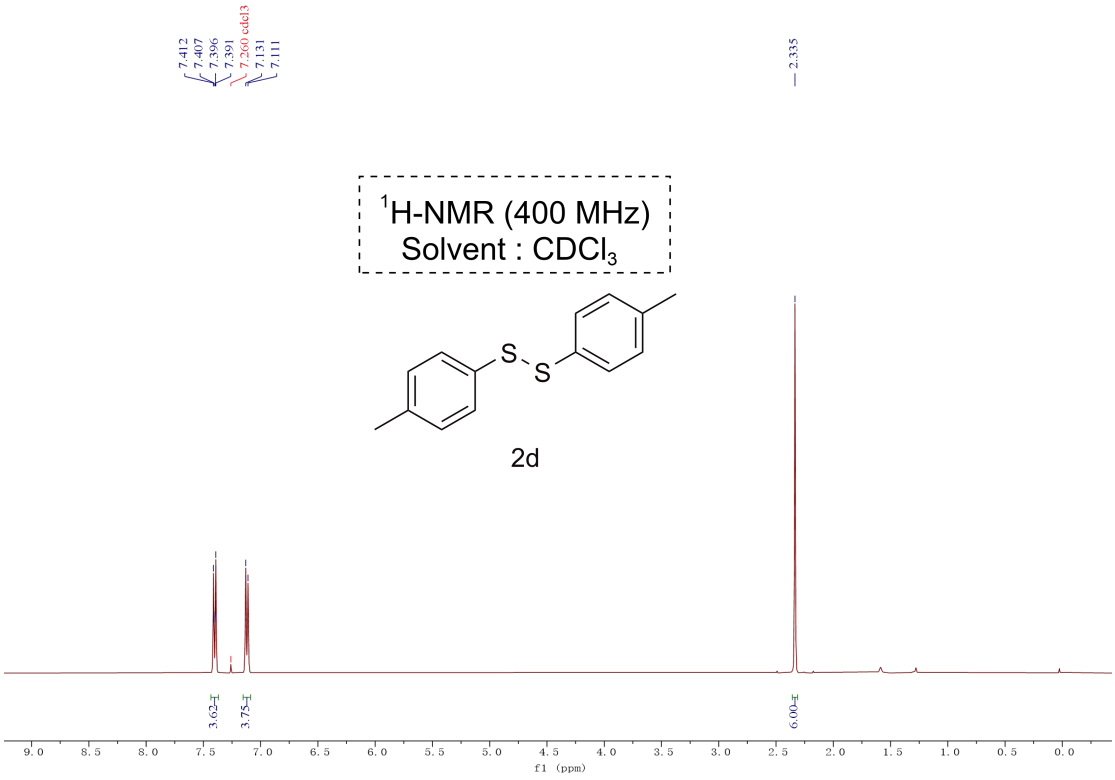
7.325
7.312
7.309
7.308
7.305
7.260 cdcl3
7.223
7.219
7.204
7.200
7.183
7.059
7.056
7.053
7.050
7.035
2.335
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2c
3.69
1.96
1.80
5.99
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


139.05
136.97
129.03
128.11
128.02
124.57
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
21.52
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.407
7.396
7.391
7.260 cdcl3
7.131
7.111
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1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2d
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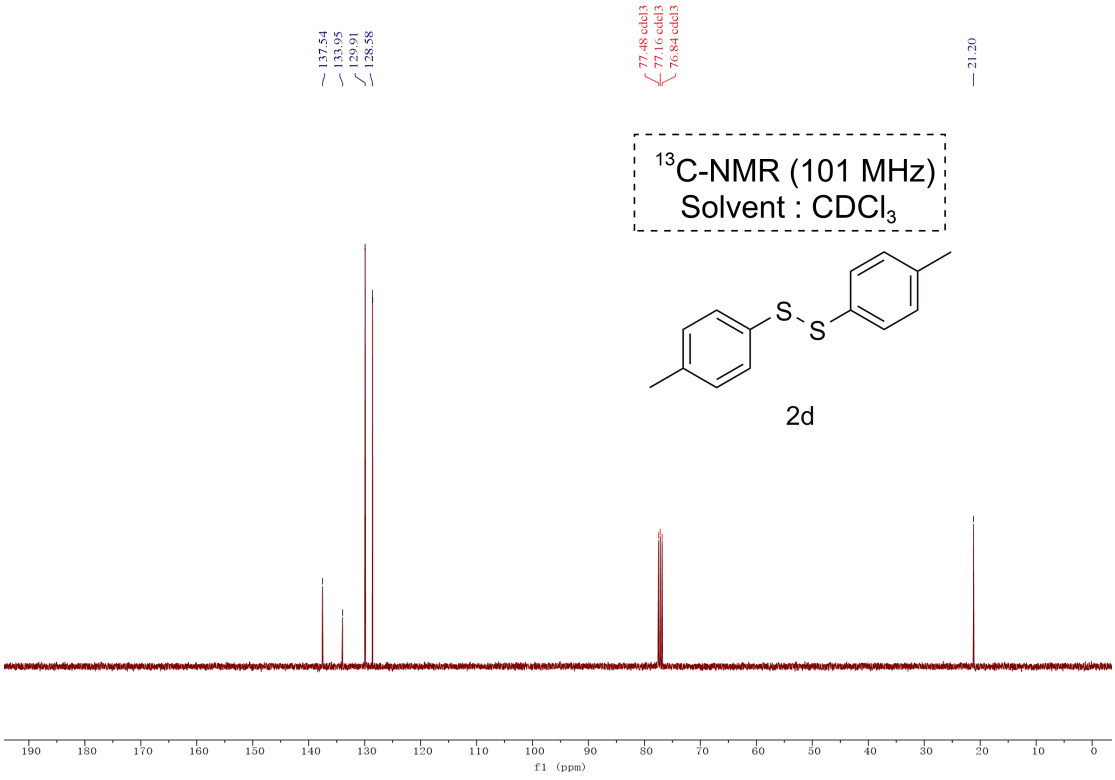
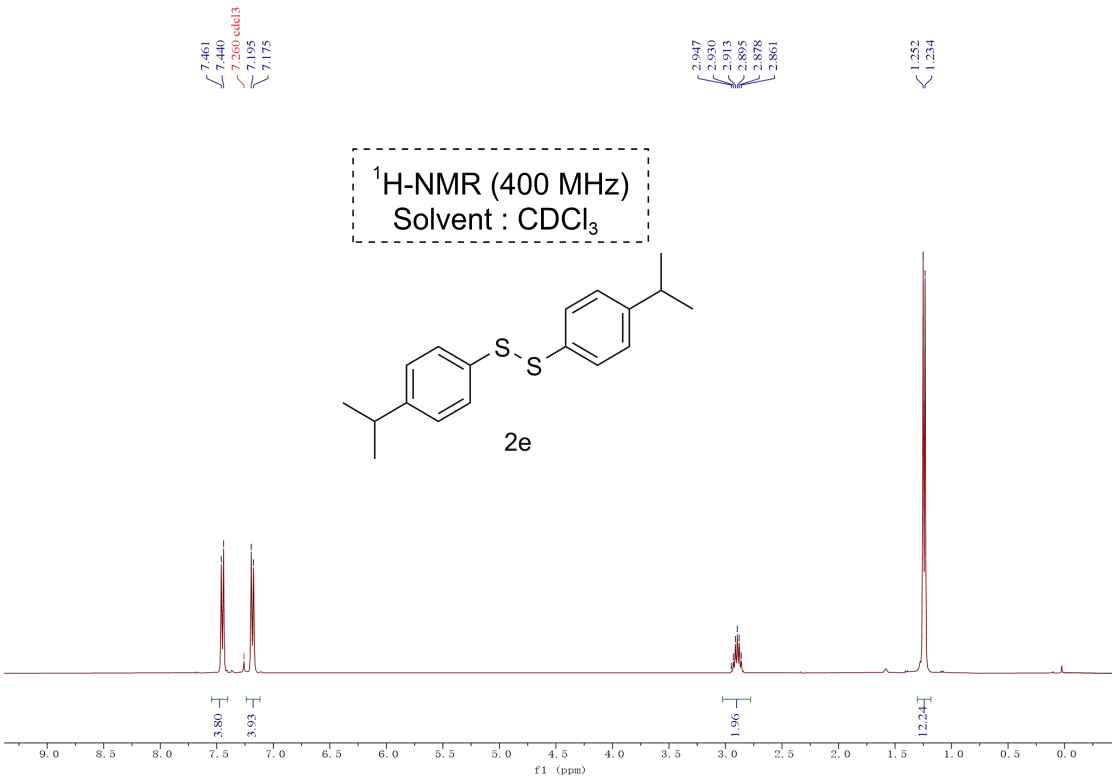
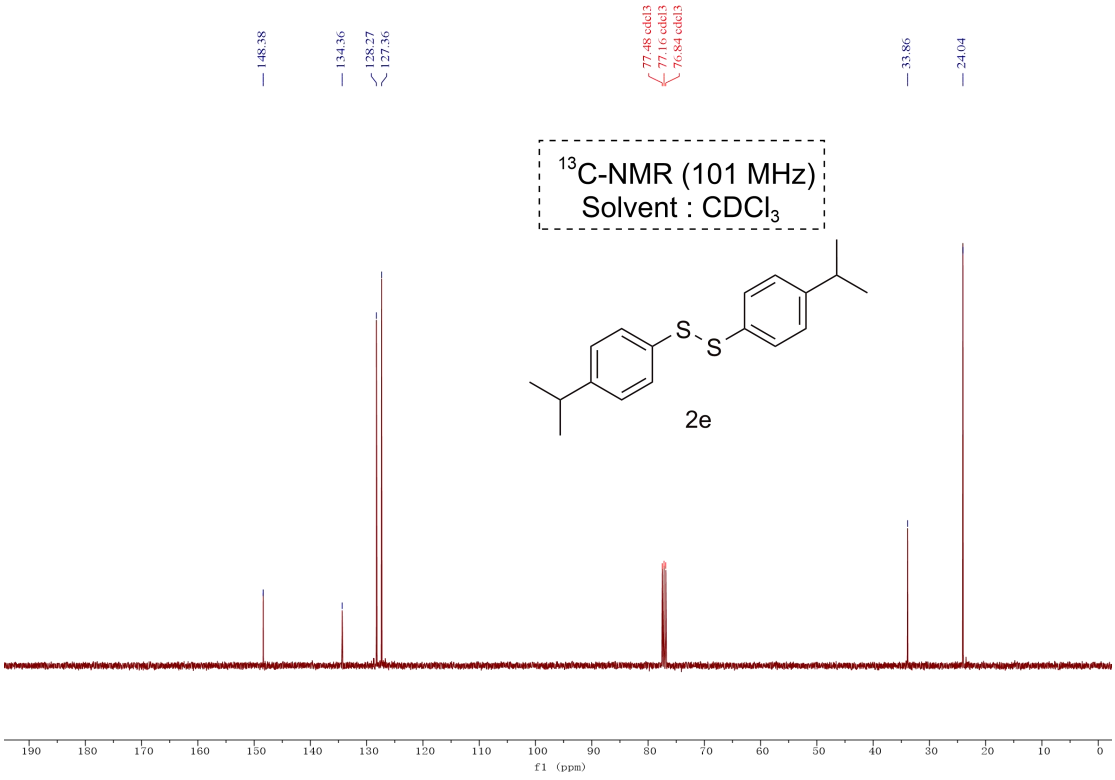
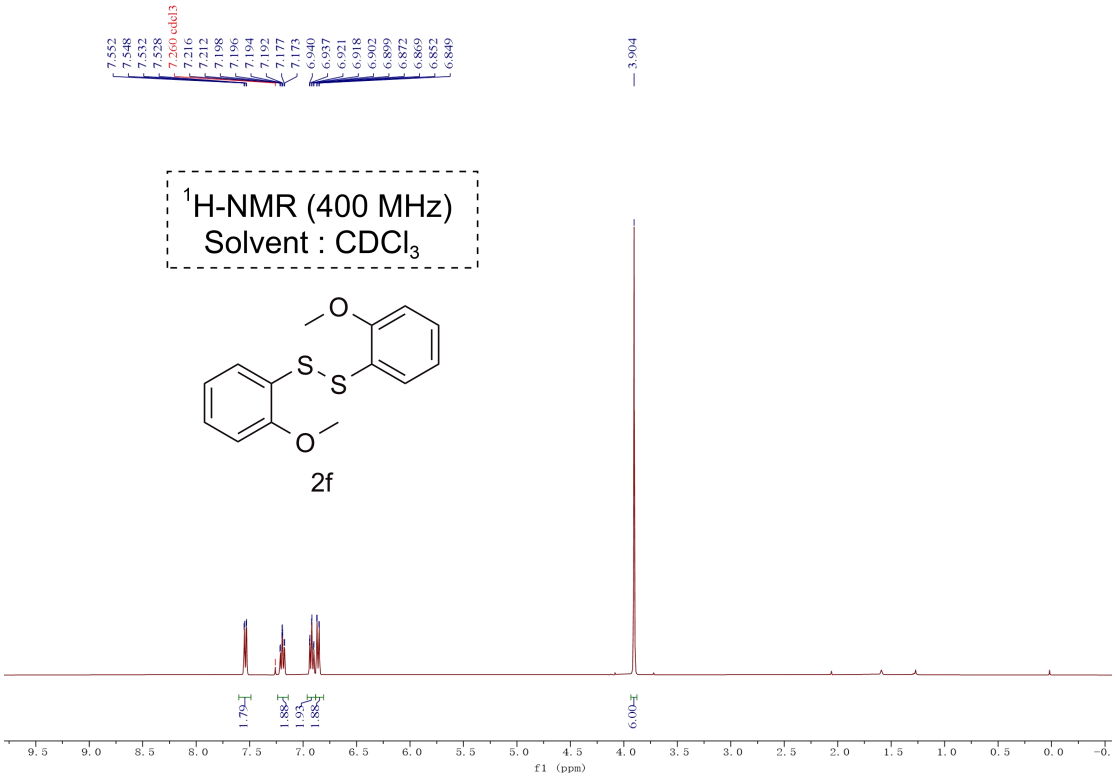
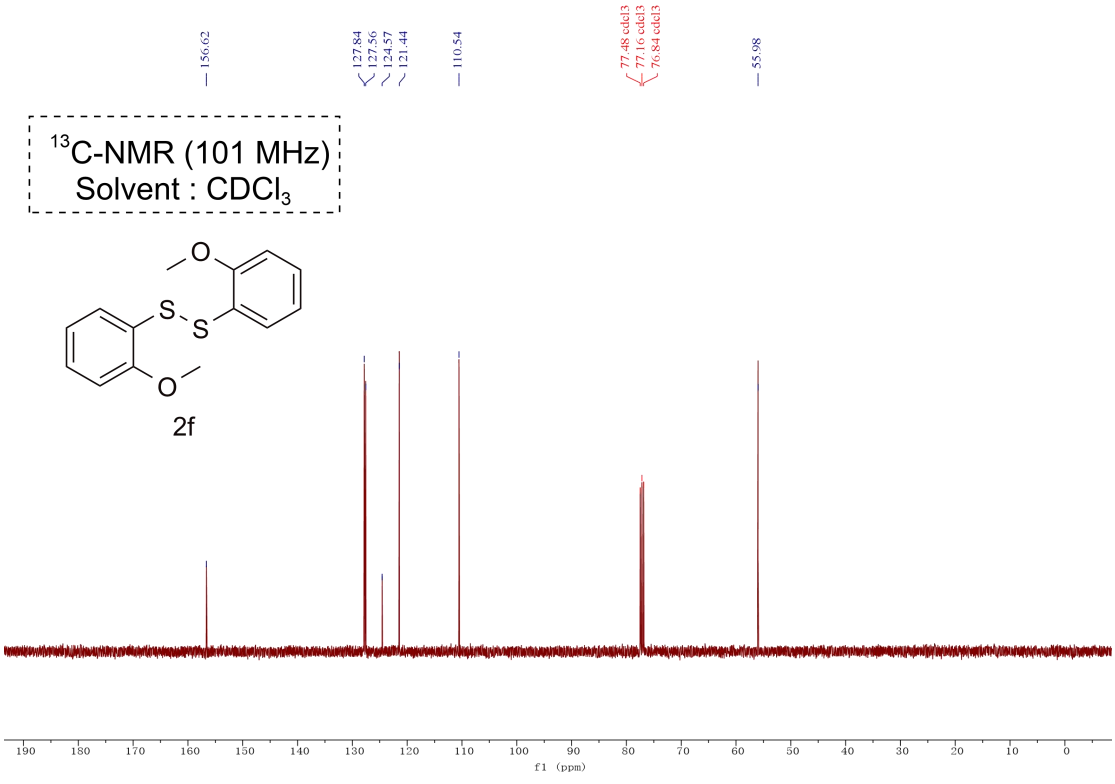
6.00
f1 (ppm)

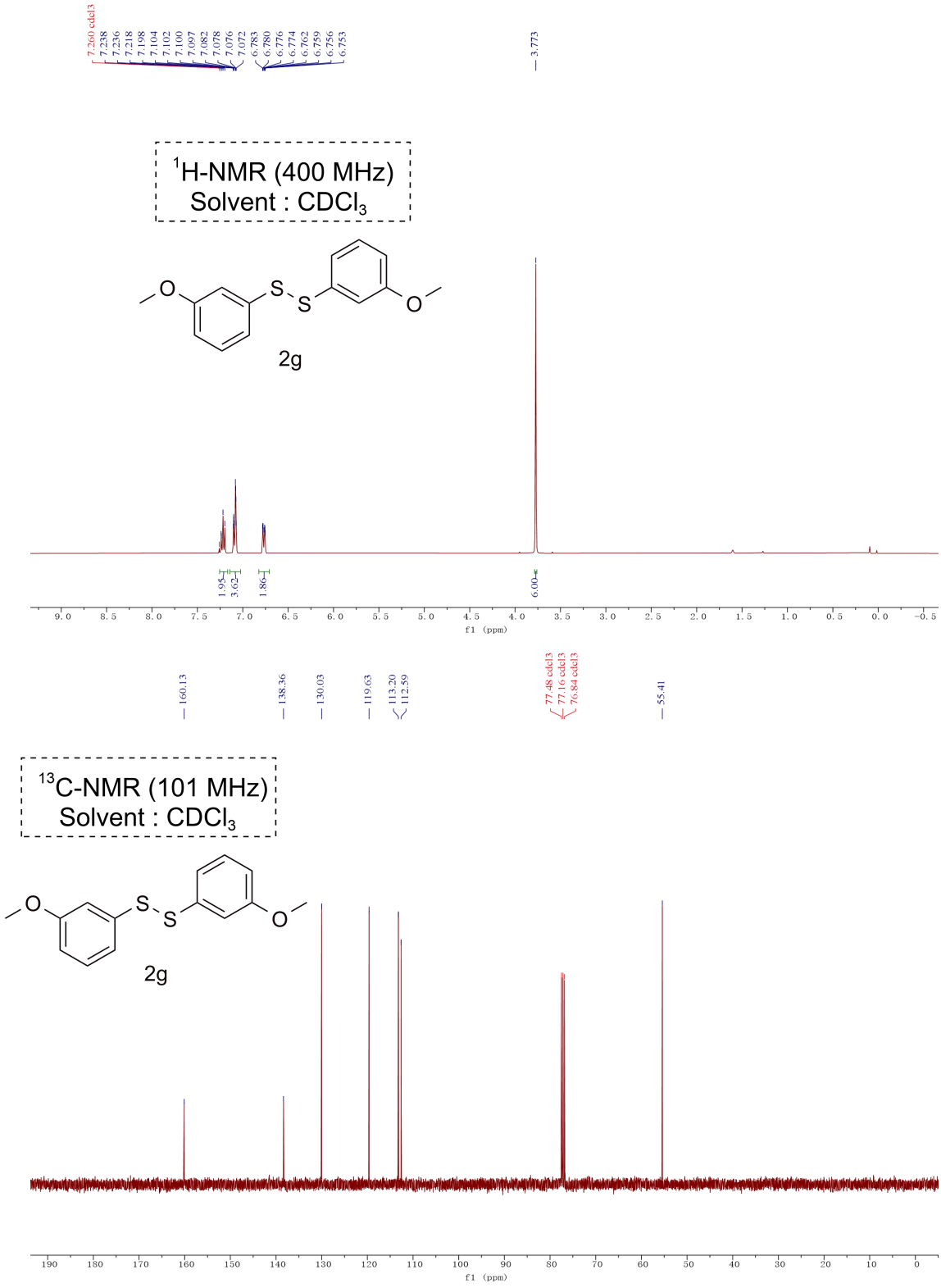

137.54
133.95
129.91
128.58
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
21.20
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2d
f1 (ppm)

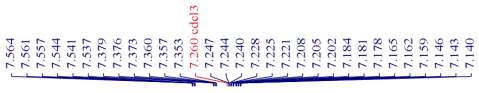

7.461
7.440
7.260 cdcl3
7.195
7.175
2.947
2.930
2.913
2.895
2.878
2.861
1.252
1.234
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$
S
S
2e
3.80
3.93
1.96
12.24
f1 (ppm)


148.38
134.36
128.27
127.36
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
33.86
24.04
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$
S
S
2e
f1 (ppm)

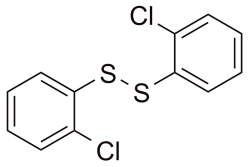

7.552
7.548
7.532
7.528
7.260 cdcl3
7.216
7.212
7.198
7.196
7.194
7.192
7.177
7.173
6.940
6.937
6.921
6.918
6.902
6.899
6.872
6.869
6.852
6.849
3.904
1H-NMR (400 MHz)
Solvent : CDCl3
O
S
S
O
2f
1.79
1.88
1.93
1.88
6.00
f1 (ppm)


156.62
127.84
127.56
124.57
121.44
110.54
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
55.98
13C-NMR (101 MHz)
Solvent : CDCl3
O
S
S
O
2f
f1 (ppm)

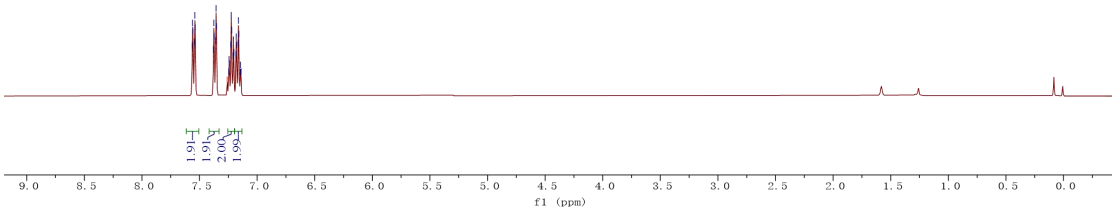

7.260 cdcl3
7.238
7.236
7.218
7.198
7.104
7.102
7.100
7.097
7.082
7.078
7.076
7.072
6.783
6.780
6.776
6.774
6.762
6.759
6.756
6.753
3.773
1H-NMR (400 MHz)
Solvent : CDCl3
O
S
S
O
2g
1.95
3.62
1.86
6.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)
160.13
138.36
130.03
119.63
113.20
112.59
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
55.41
13C-NMR (101 MHz)
Solvent : CDCl3
O
S
S
O
2g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



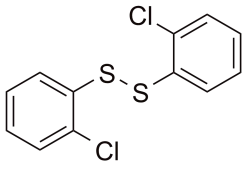
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



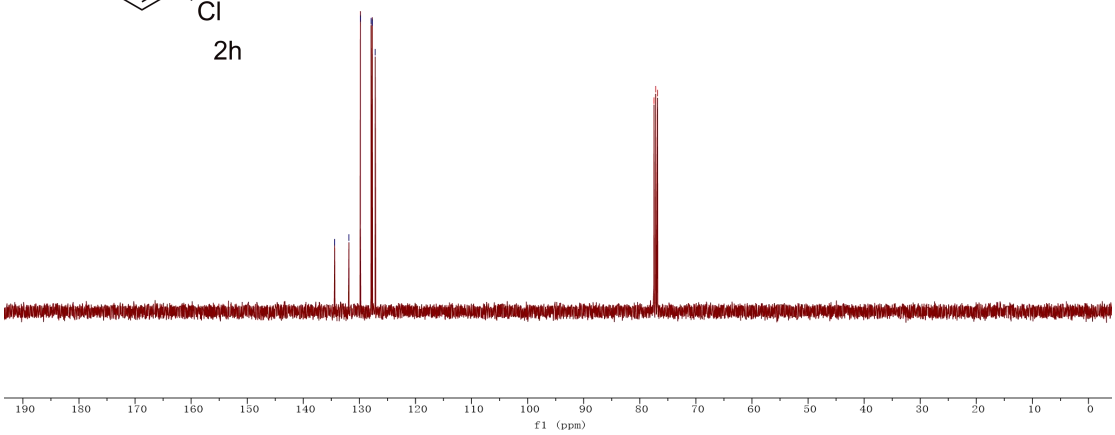
2h



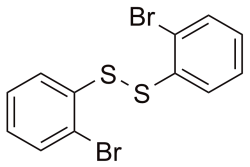
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$



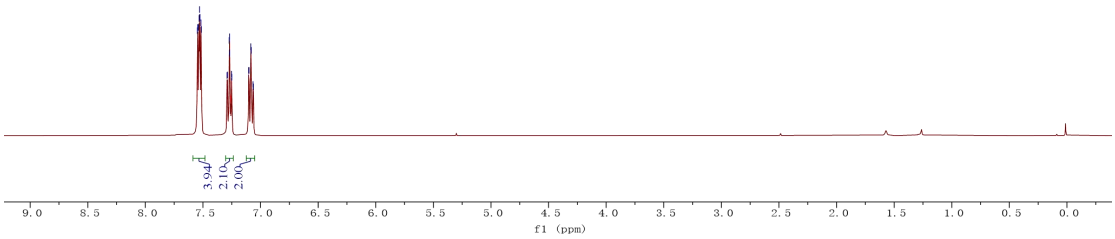
2h



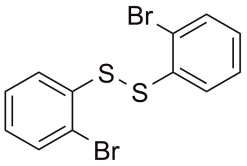
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



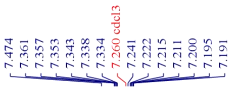
2i



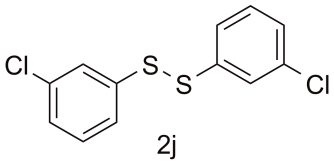
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$



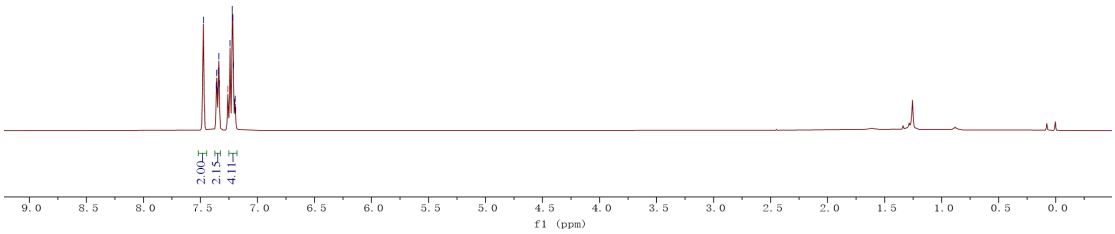
2i



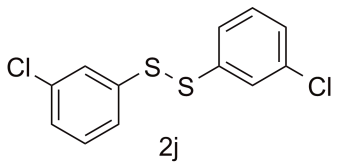
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



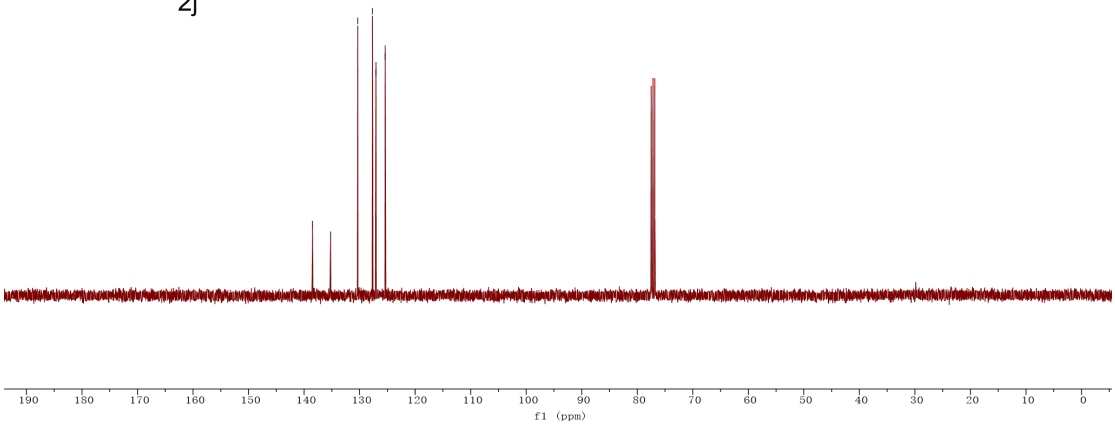
2j

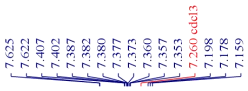


^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$

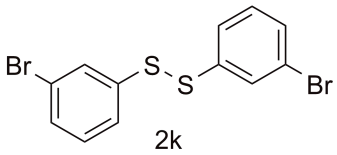


2j

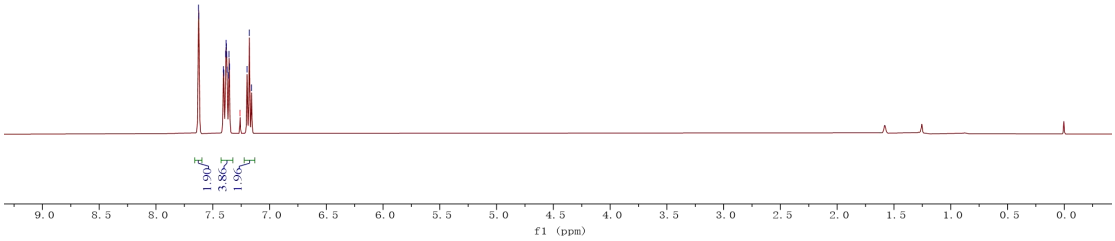




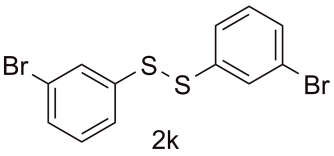
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



2k

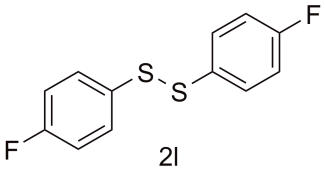


^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$

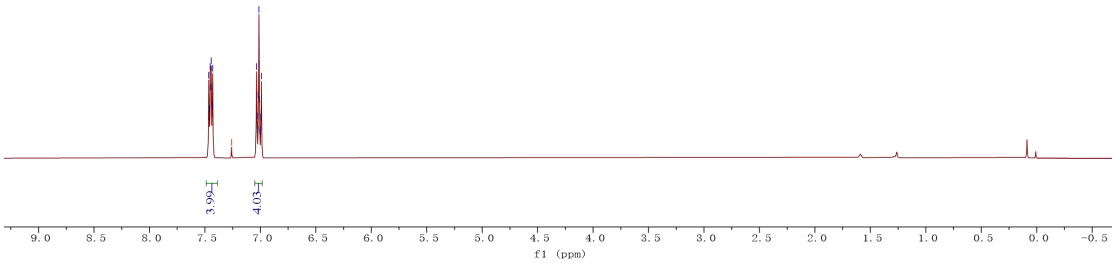


2k

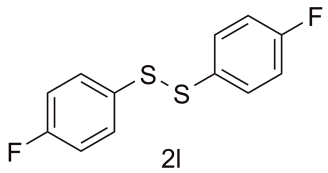
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



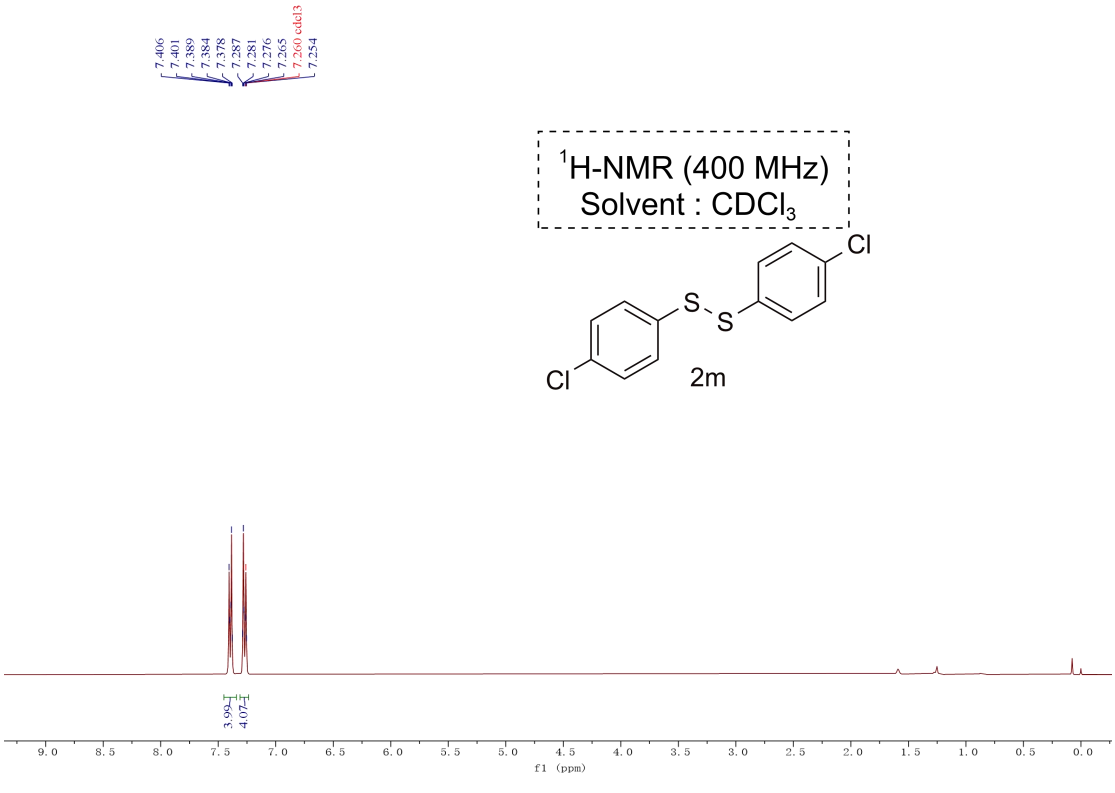
2l

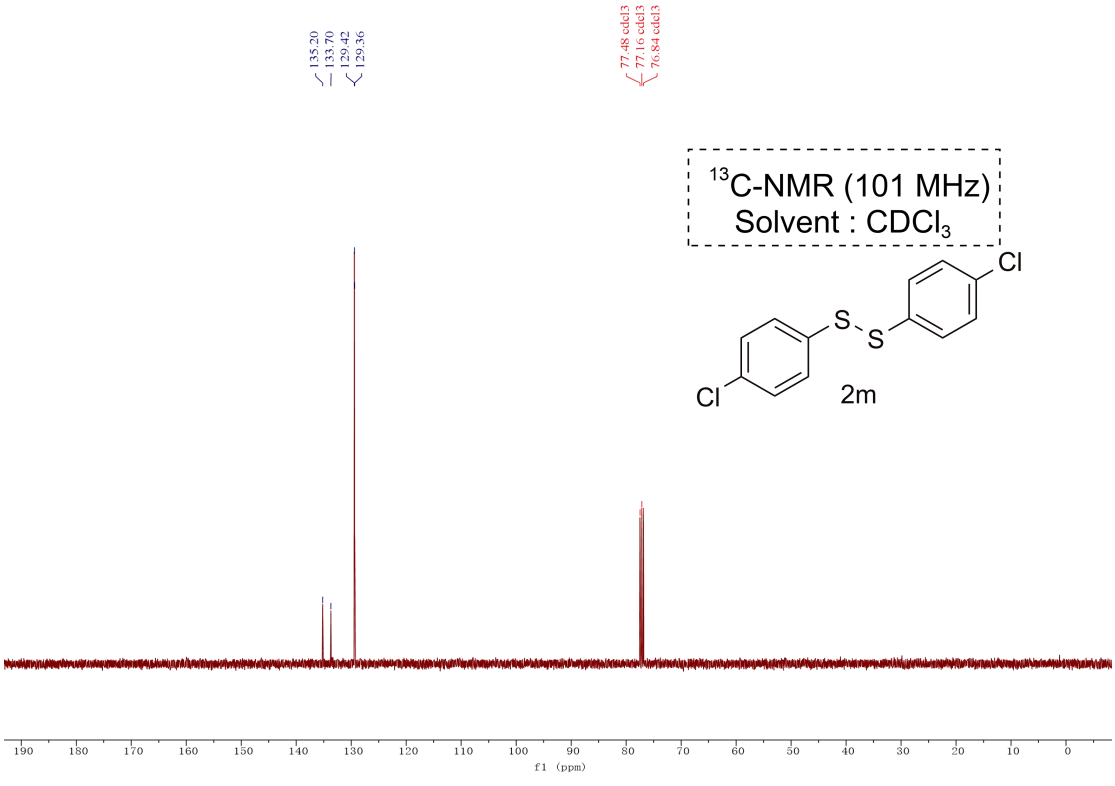


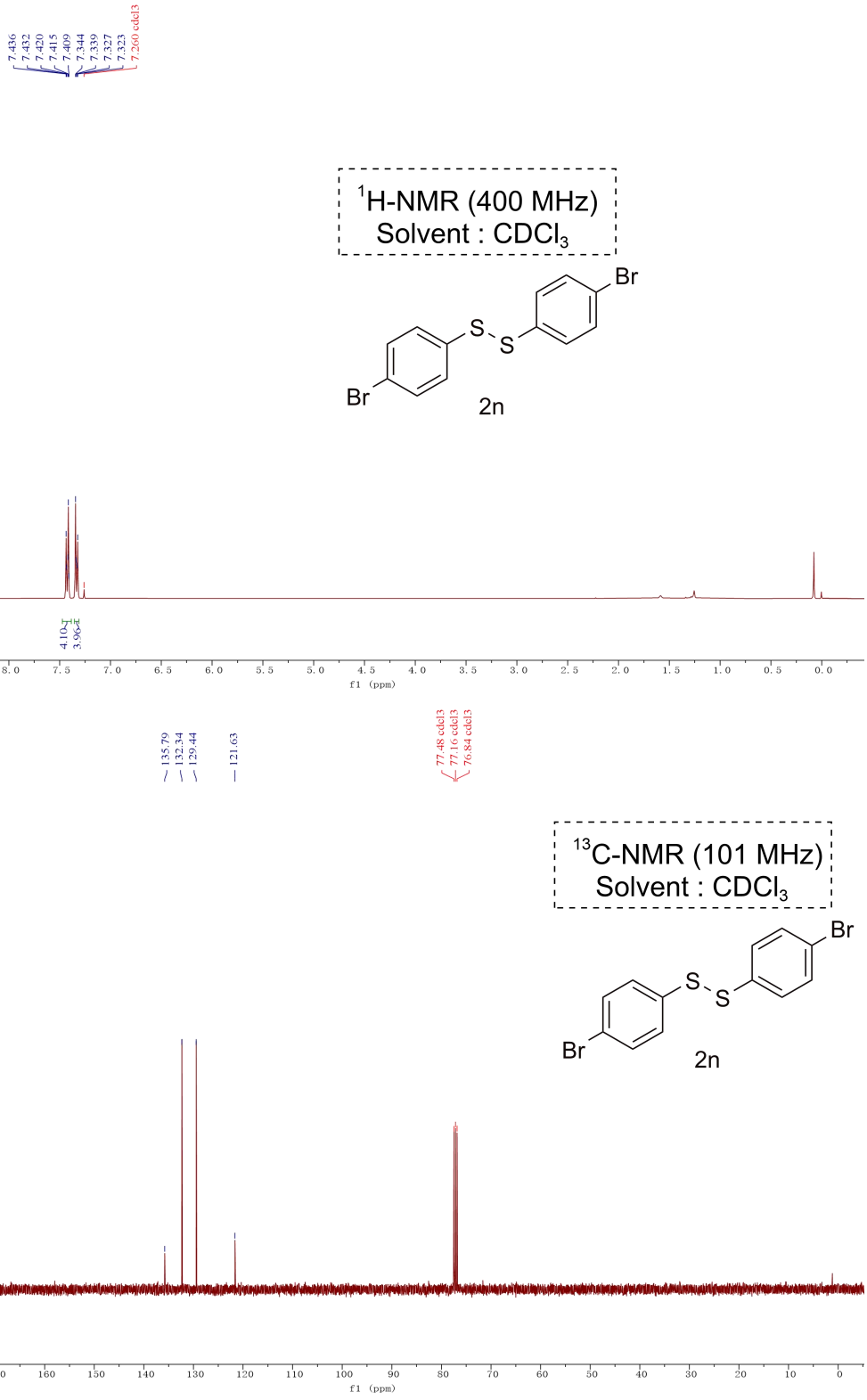
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$

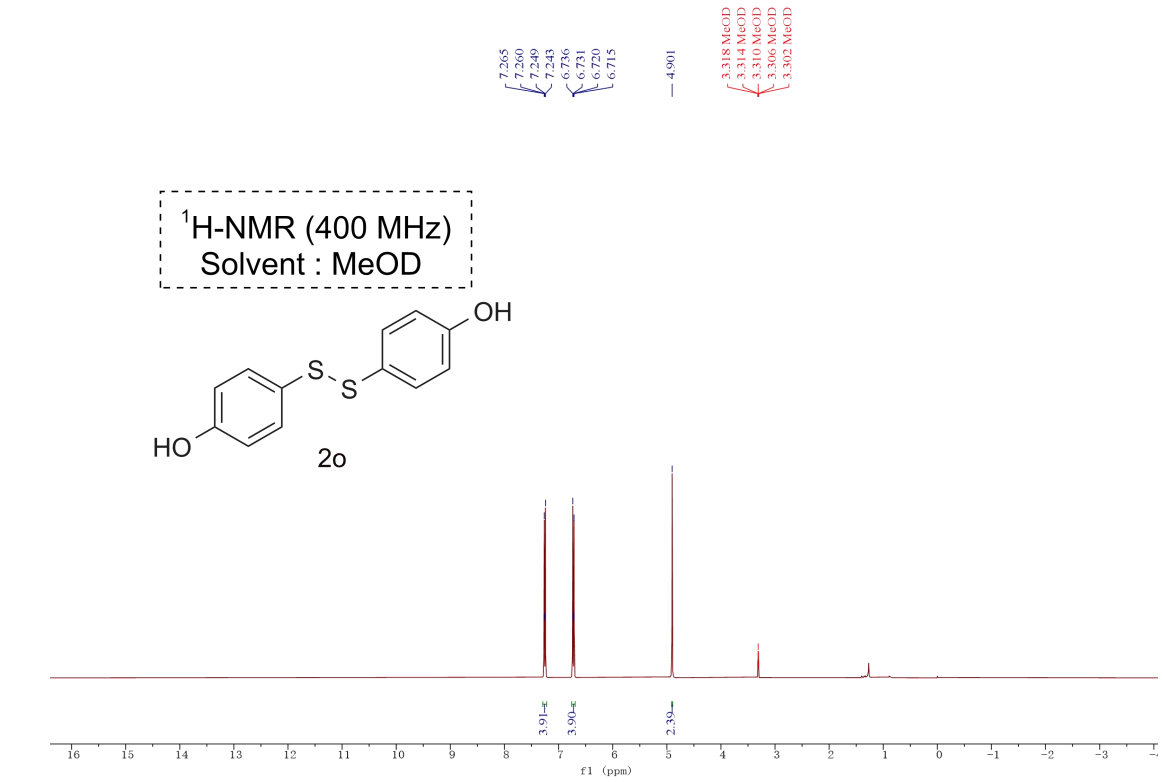


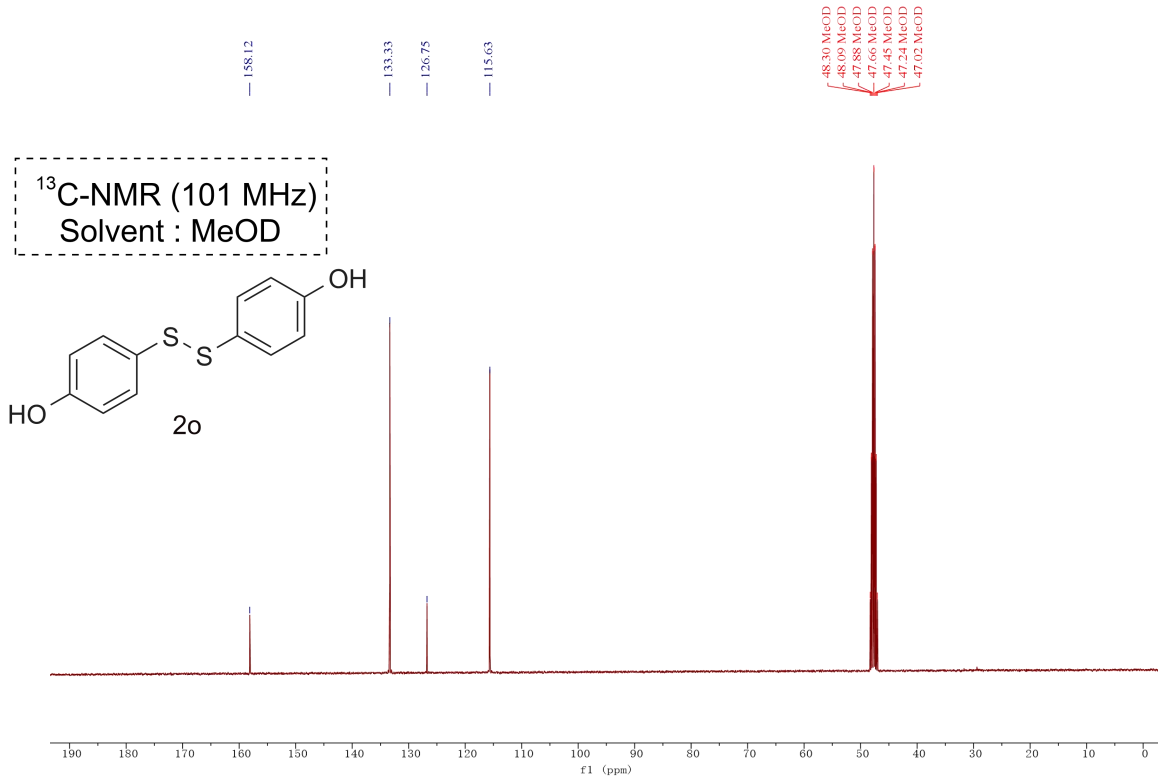
2l

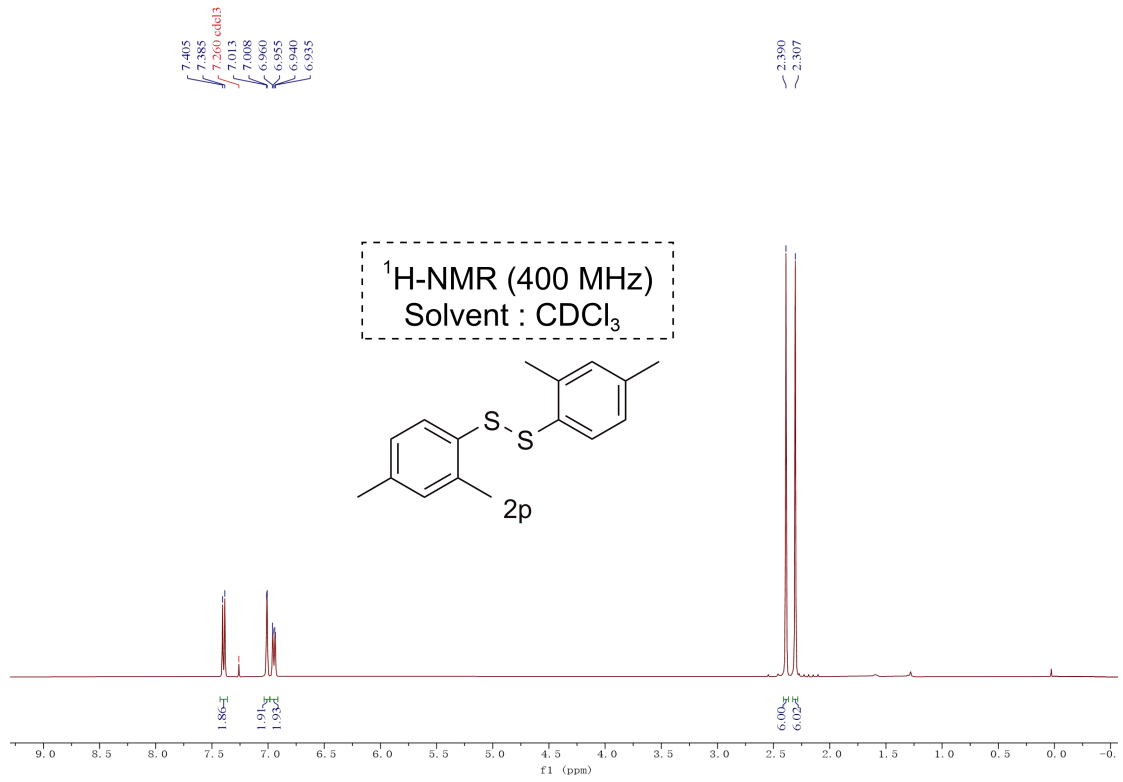

7.406
7.401
7.389
7.384
7.378
7.287
7.281
7.276
7.265
7.260 cdcl3
7.254
1H-NMR (400 MHz)
Solvent : CDCl3
Cl
S
S
Cl
2m
3.99
4.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

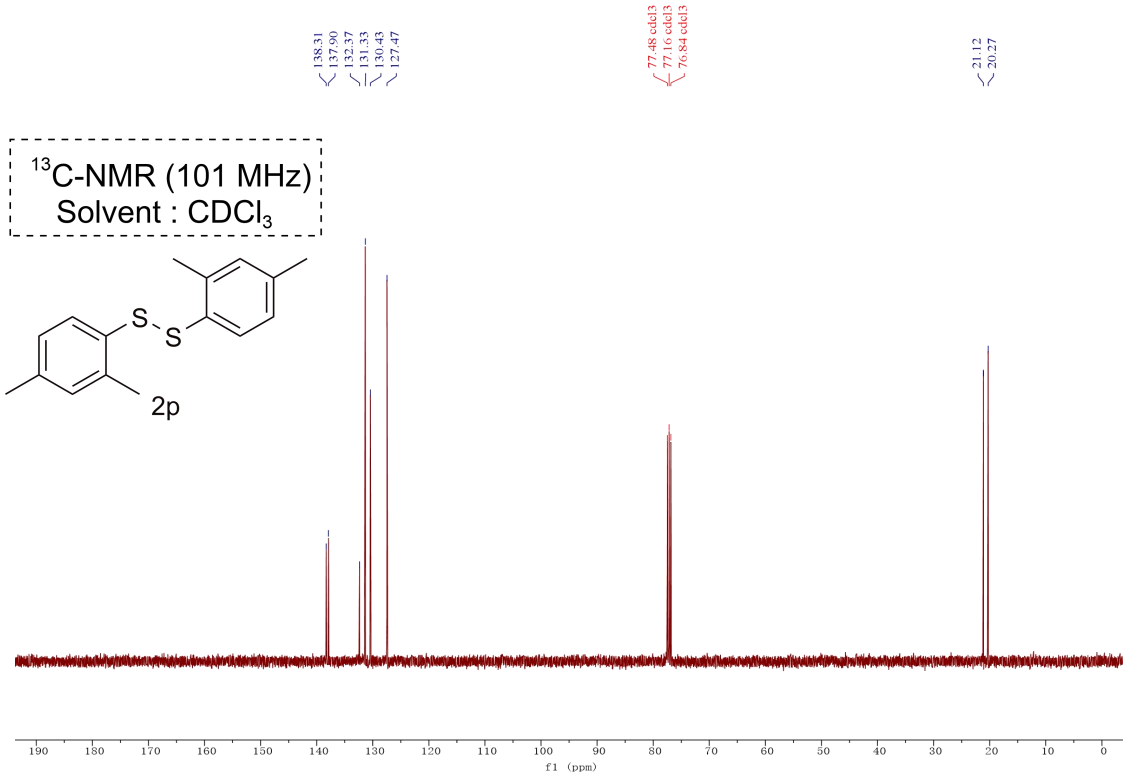

135.20
133.70
129.42
129.36
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
13C-NMR (101 MHz)
Solvent : CDCl3
Cl
S
S
Cl
2m
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

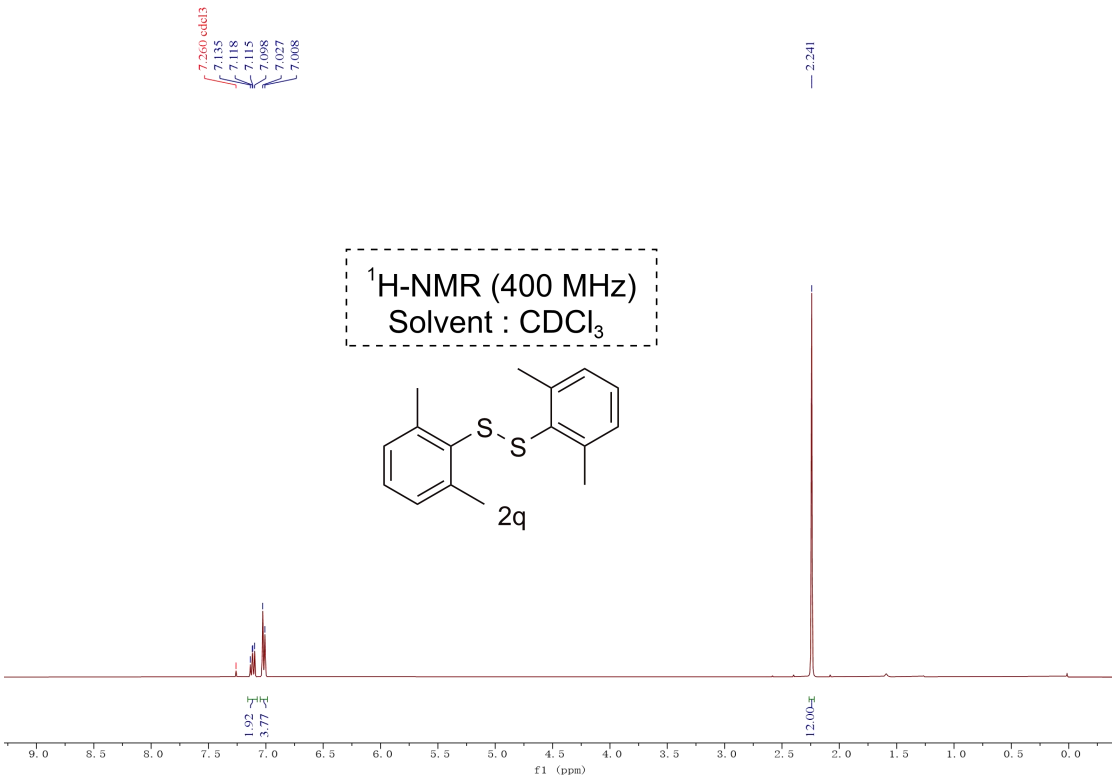

7.436
7.432
7.420
7.415
7.409
7.344
7.339
7.327
7.323
7.260 cdcl3
1H-NMR (400 MHz)
Solvent : CDCl3
Br
S
S
Br
2n
4.10
3.96
f1 (ppm)
135.79
132.34
129.44
121.63
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
13C-NMR (101 MHz)
Solvent : CDCl3
Br
S
S
Br
2n
f1 (ppm)

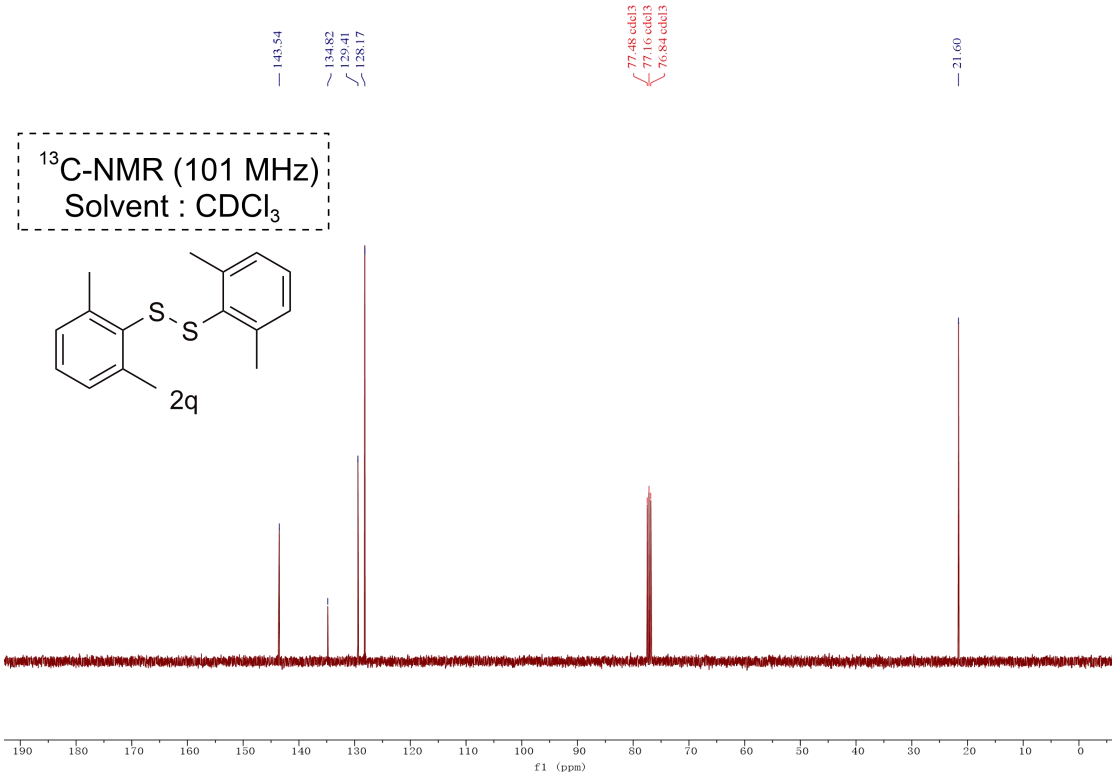

7.265
7.260
7.249
7.243
6.736
6.731
6.720
6.715
4.901
3.318 MeOD
3.314 MeOD
3.310 MeOD
3.306 MeOD
3.302 MeOD
1H-NMR (400 MHz)
Solvent : MeOD
OH
S
S
HO
2o
3.91
3.90
2.39
f1 (ppm)

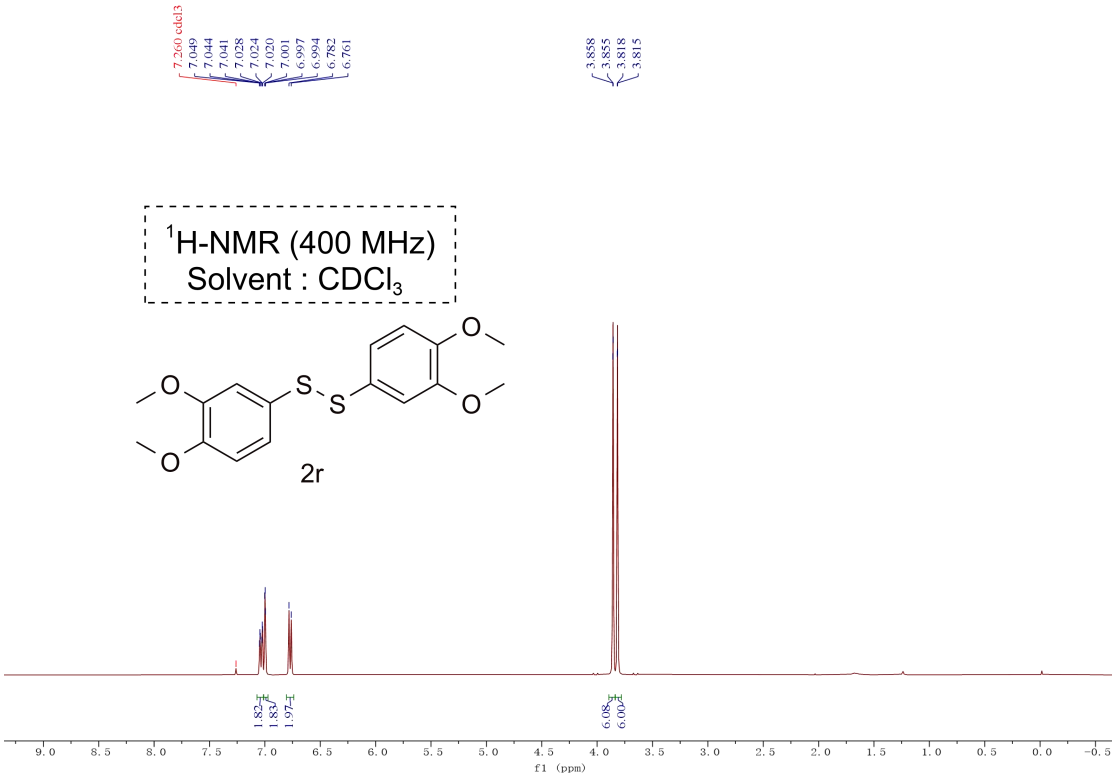

158.12
133.33
126.75
115.63
48.30 MeOD
48.09 MeOD
47.88 MeOD
47.66 MeOD
47.45 MeOD
47.24 MeOD
47.02 MeOD
13C-NMR (101 MHz)
Solvent : MeOD
OH
S
S
HO
2o
f1 (ppm)

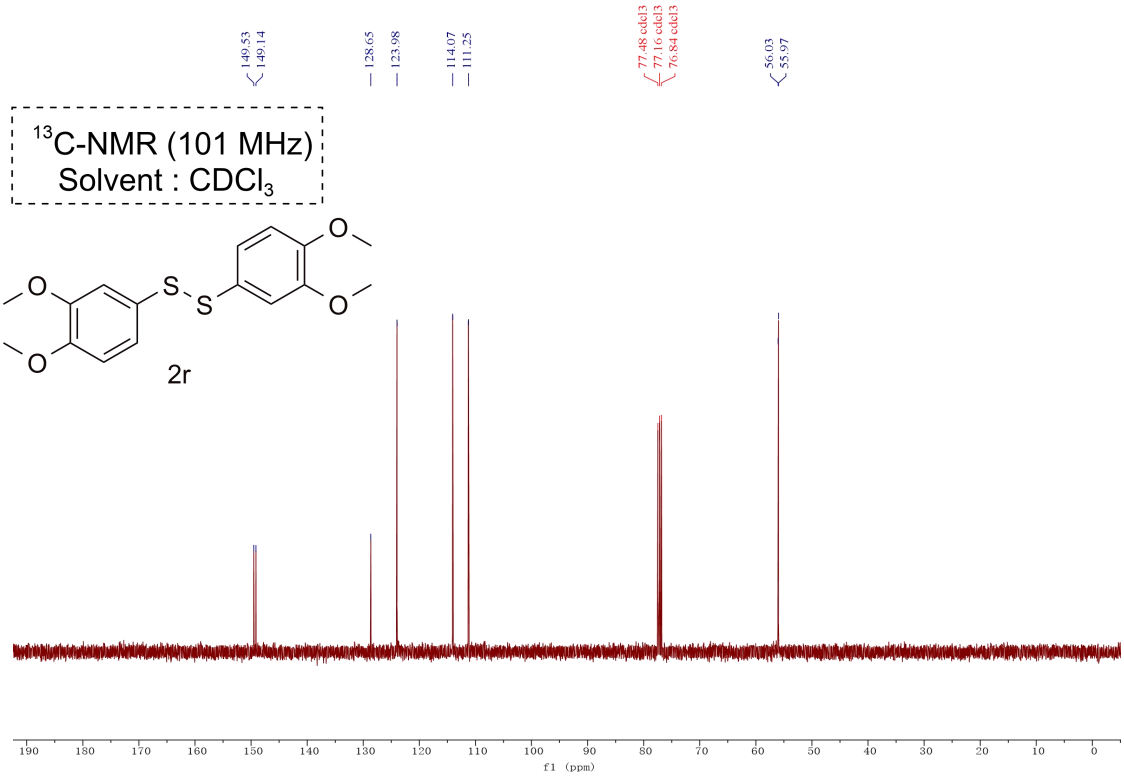

7.405
7.385
7.260 cdcl3
7.013
7.008
6.960
6.955
6.940
6.935
2.390
2.307
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$
S
S
2p
1.86
1.91
1.93
6.00
6.02
f1 (ppm)

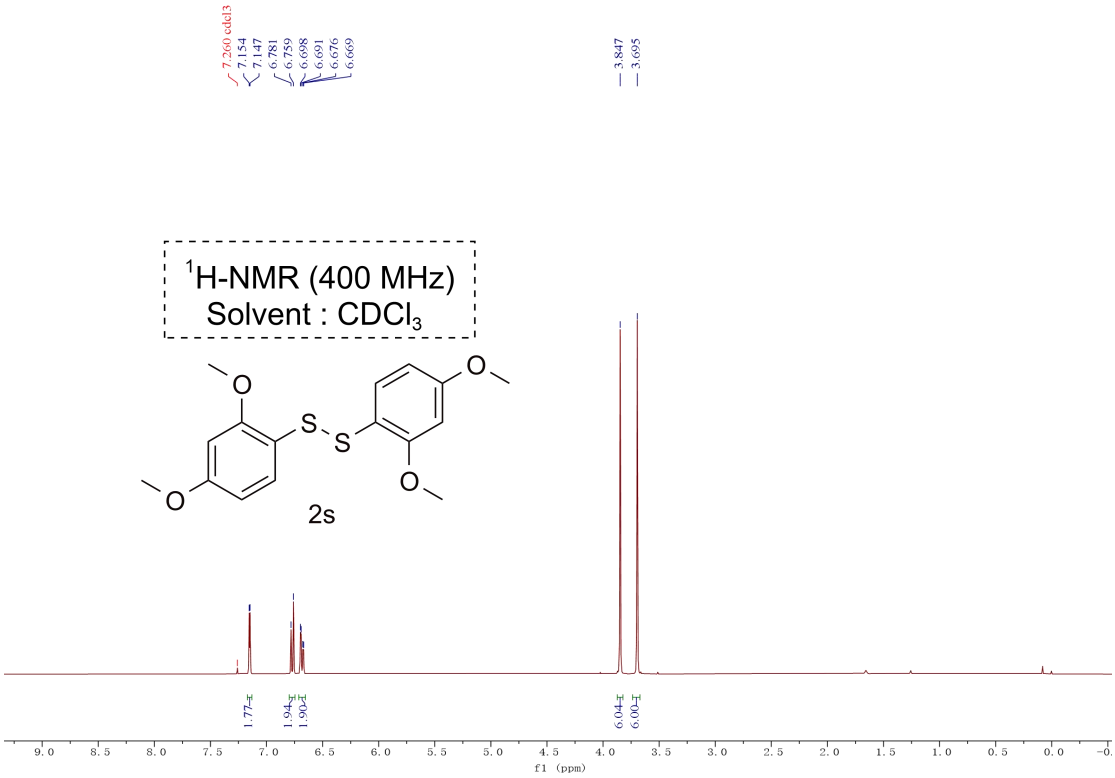

138.31
137.90
132.37
131.33
130.43
127.47
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
21.12
20.27
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$
S
S
2p
f1 (ppm)

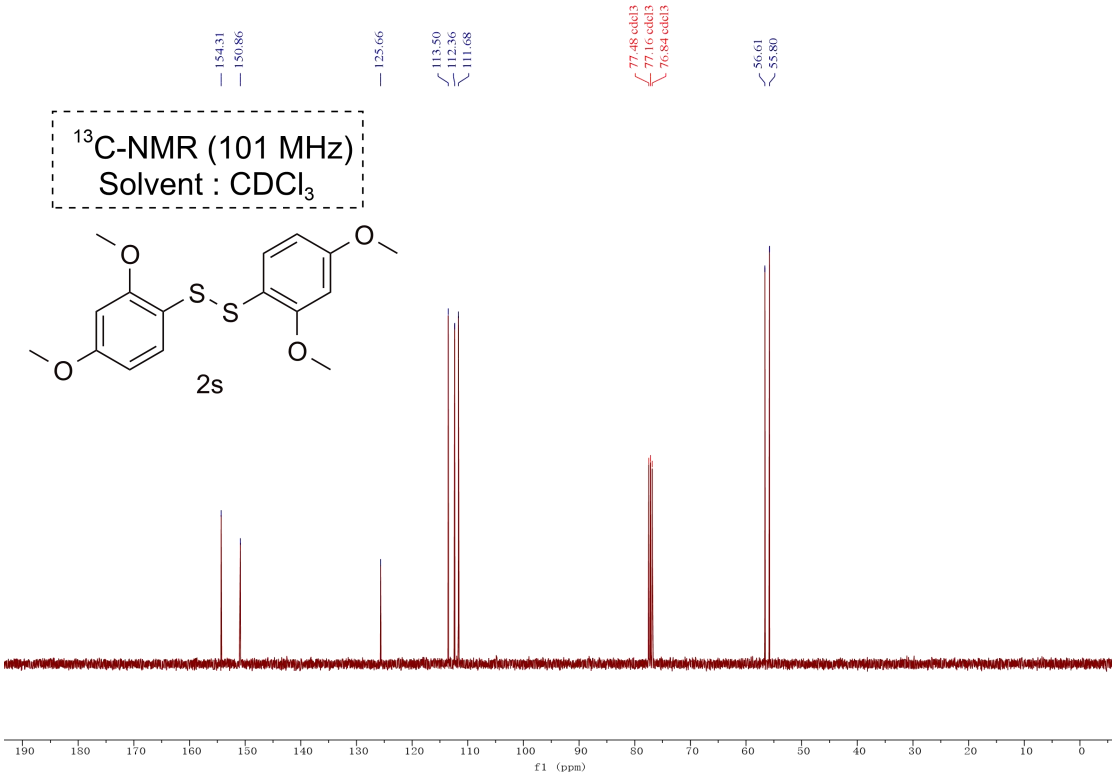

7.260 cdcl3
7.135
7.118
7.115
7.098
7.027
7.008
2.241
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2q
1.92
3.77
12.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

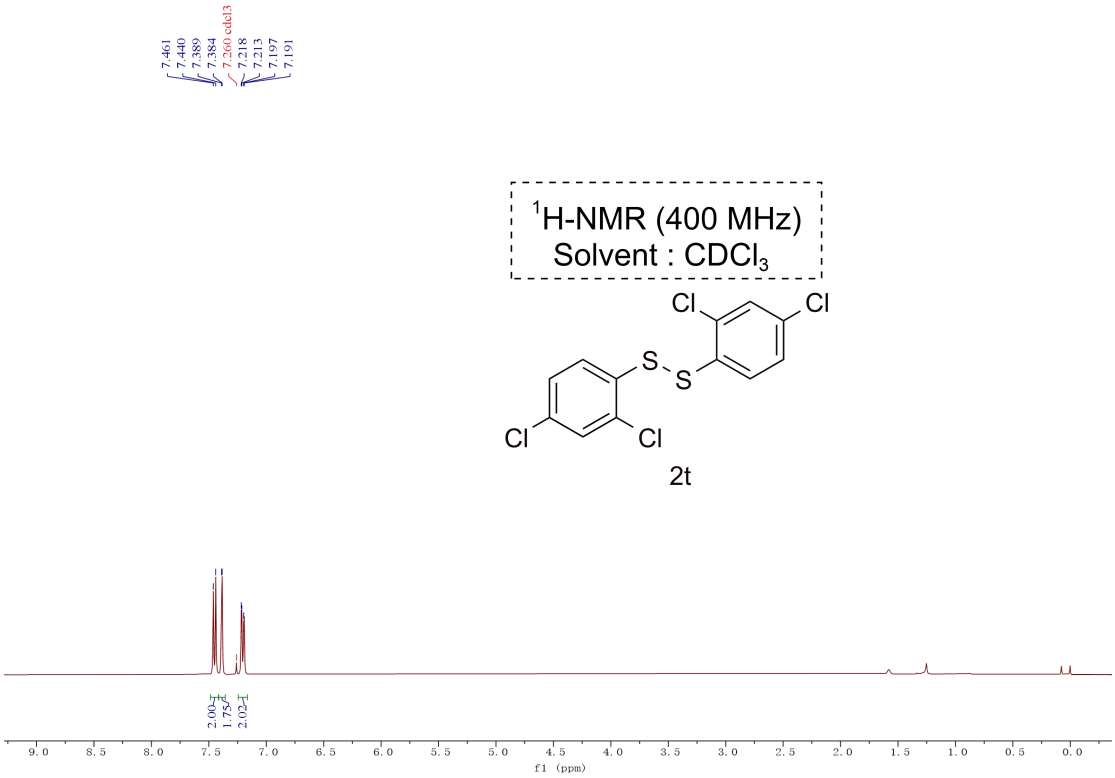

143.54
134.82
129.41
128.17
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
21.60
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

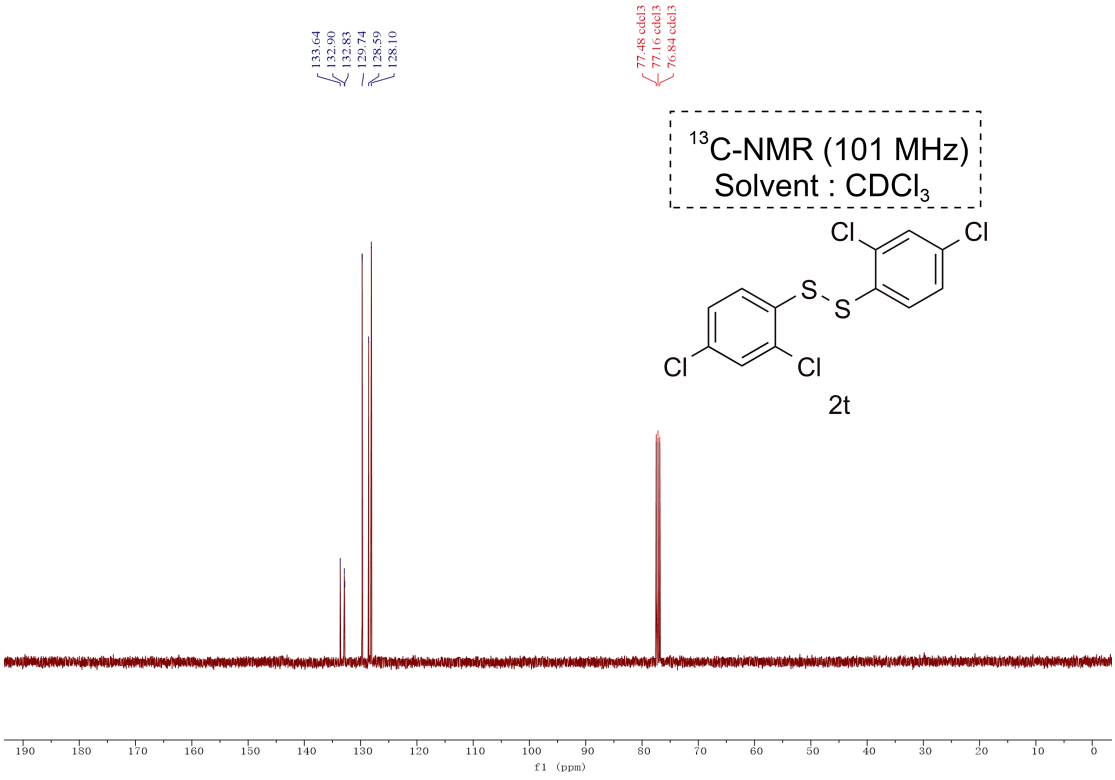

7.260 cdcl3
7.049
7.044
7.041
7.028
7.024
7.020
7.001
6.997
6.994
6.782
6.761
3.858
3.855
3.818
3.815
1H-NMR (400 MHz)
Solvent : CDCl3
2r
1.82
1.83
1.97
6.08
6.00
f1 (ppm)

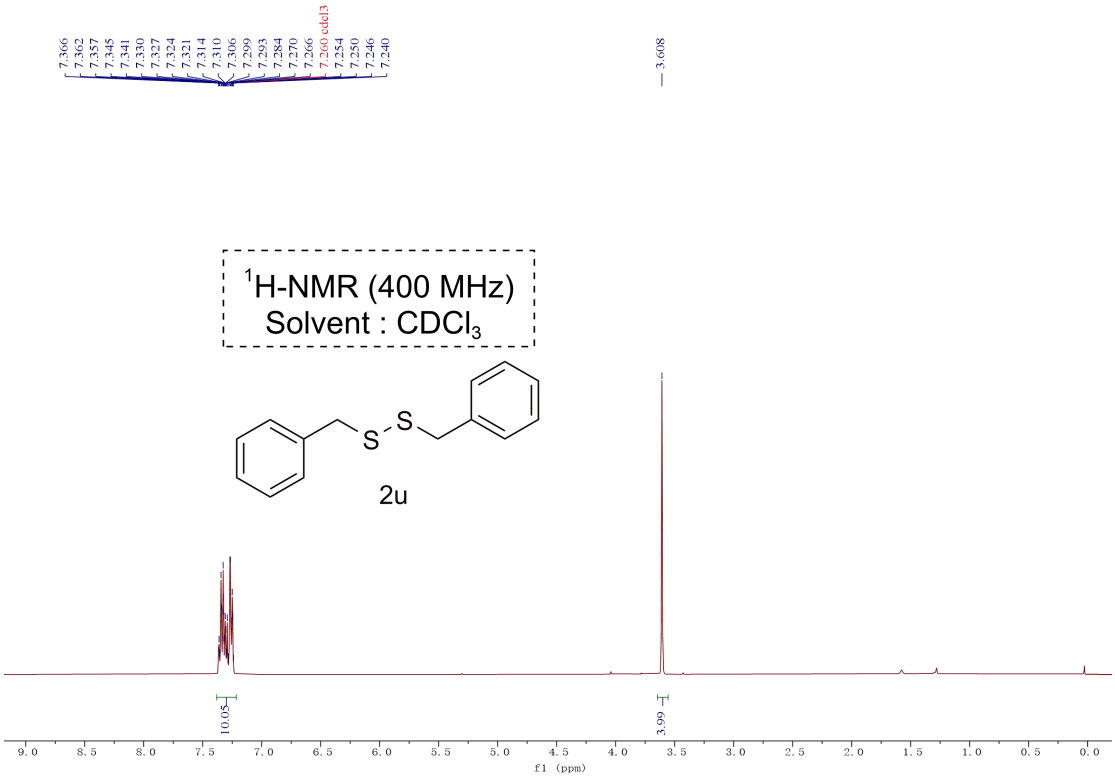

149.53
149.14
128.65
123.98
114.07
111.25
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
56.03
55.97
13C-NMR (101 MHz)
Solvent : CDCl3
2r
f1 (ppm)

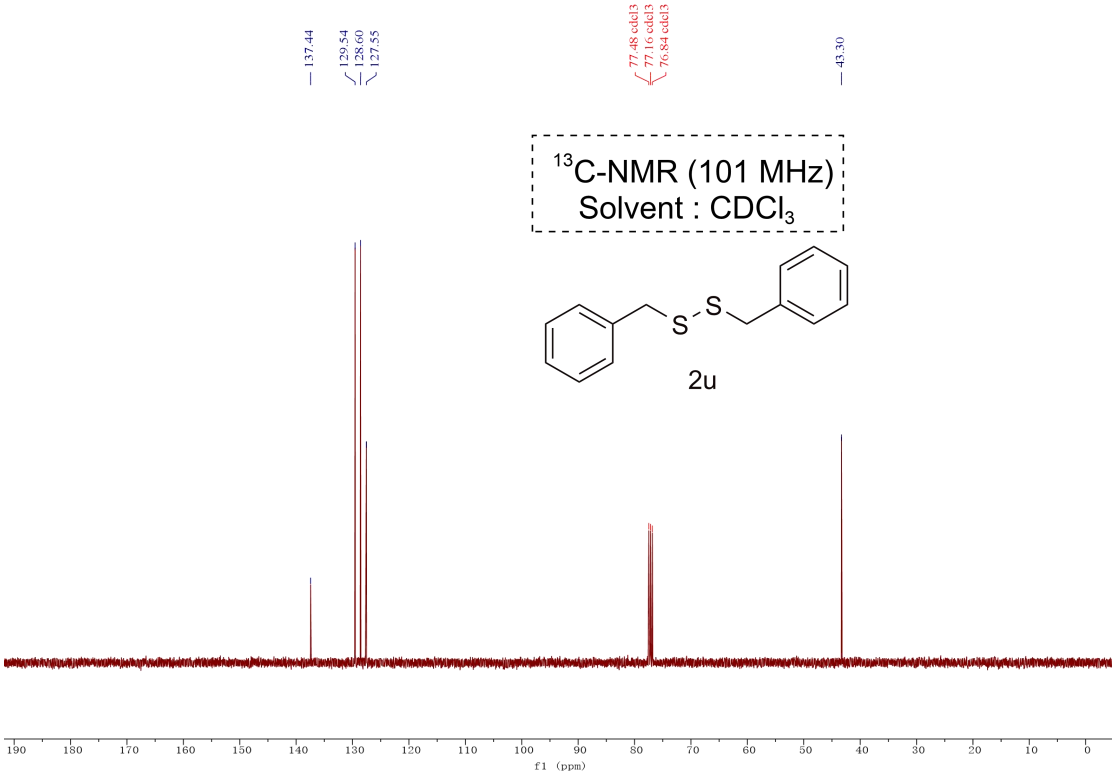

7.260 cdcl3
7.154
7.147
6.781
6.759
6.698
6.691
6.676
6.669
3.847
3.695
1H-NMR (400 MHz)
Solvent : CDCl3
2s
1.77
1.94
1.90
6.04
6.00
f1 (ppm)


154.31
150.86
125.66
113.50
112.36
111.68
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
56.61
55.80
13C-NMR (101 MHz)
Solvent : CDCl3
2s
f1 (ppm)

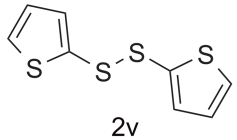

7.461
7.440
7.389
7.384
7.260 cdcl3
7.218
7.213
7.197
7.191
1H-NMR (400 MHz)
Solvent : CDCl3
Cl
Cl
S
S
Cl
Cl
2t
2.00
1.75
2.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


133.64
132.90
132.83
129.74
128.59
128.10
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
13C-NMR (101 MHz)
Solvent : CDCl3
Cl
Cl
S
S
Cl
Cl
2t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

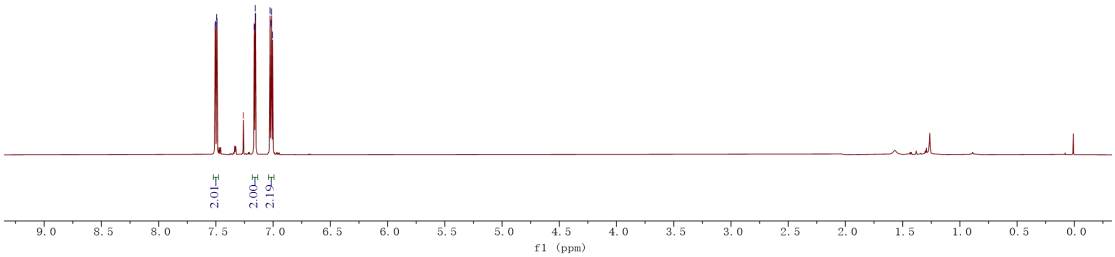

7.366
7.362
7.357
7.345
7.341
7.330
7.327
7.324
7.321
7.314
7.310
7.306
7.299
7.293
7.284
7.270
7.266
7.260 cdcl3
7.254
7.250
7.246
7.240
3.608
1H-NMR (400 MHz)
Solvent : CDCl3
2u
10.05
3.99
f1 (ppm)


137.44
129.54
128.60
127.55
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
43.30
13C-NMR (101 MHz)
Solvent : CDCl3
2u
f1 (ppm)

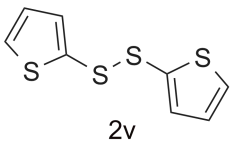
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$



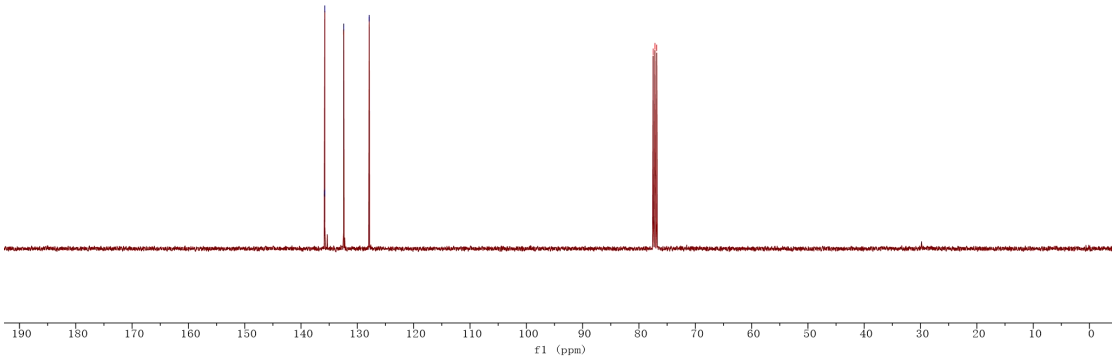
2v

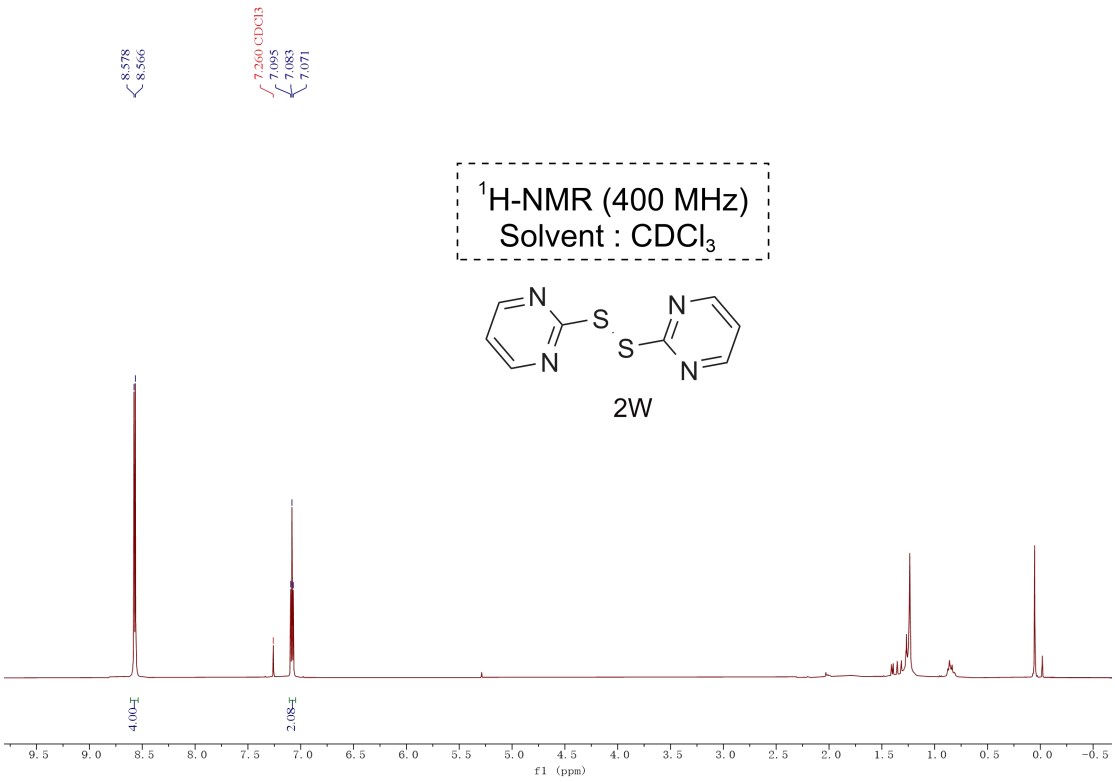


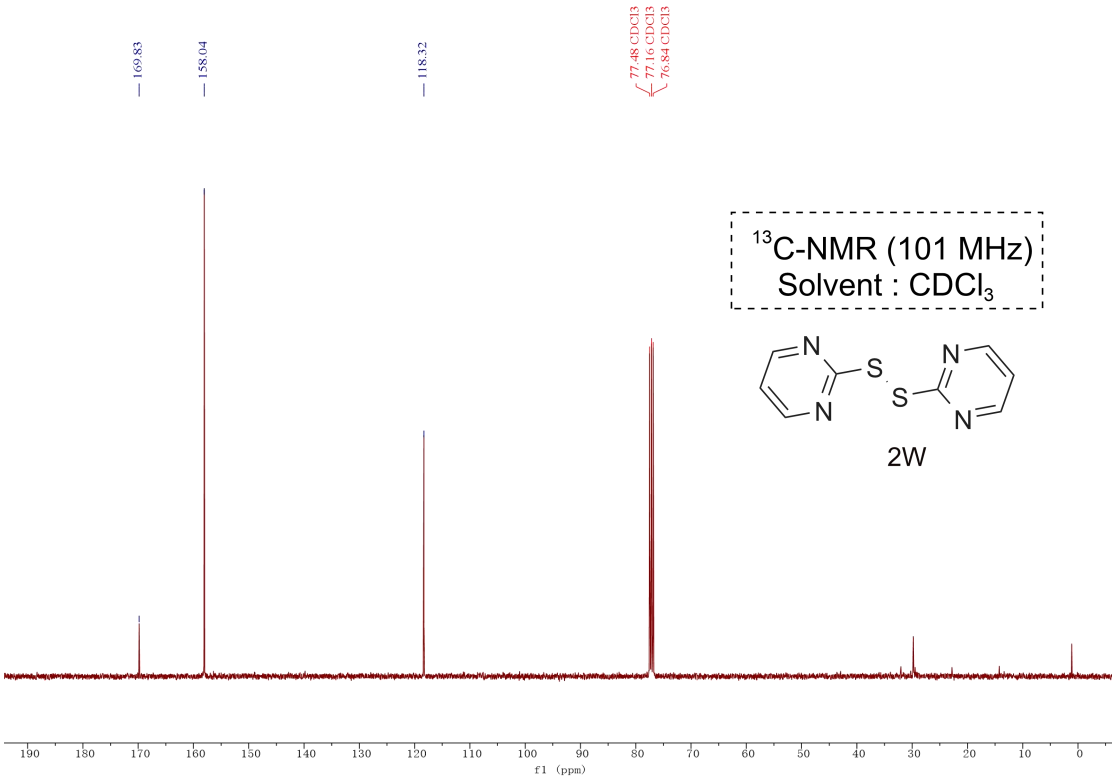
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$

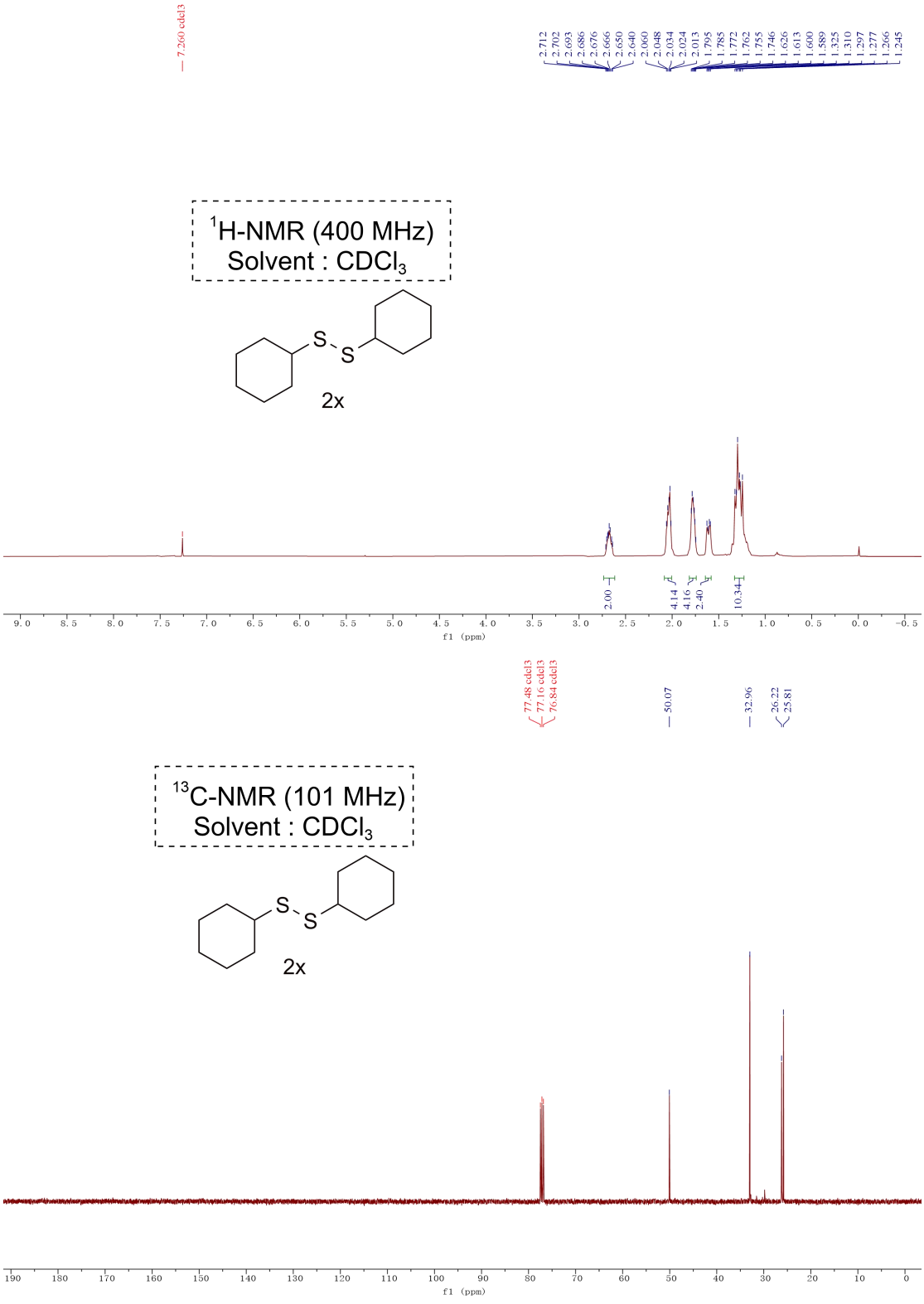


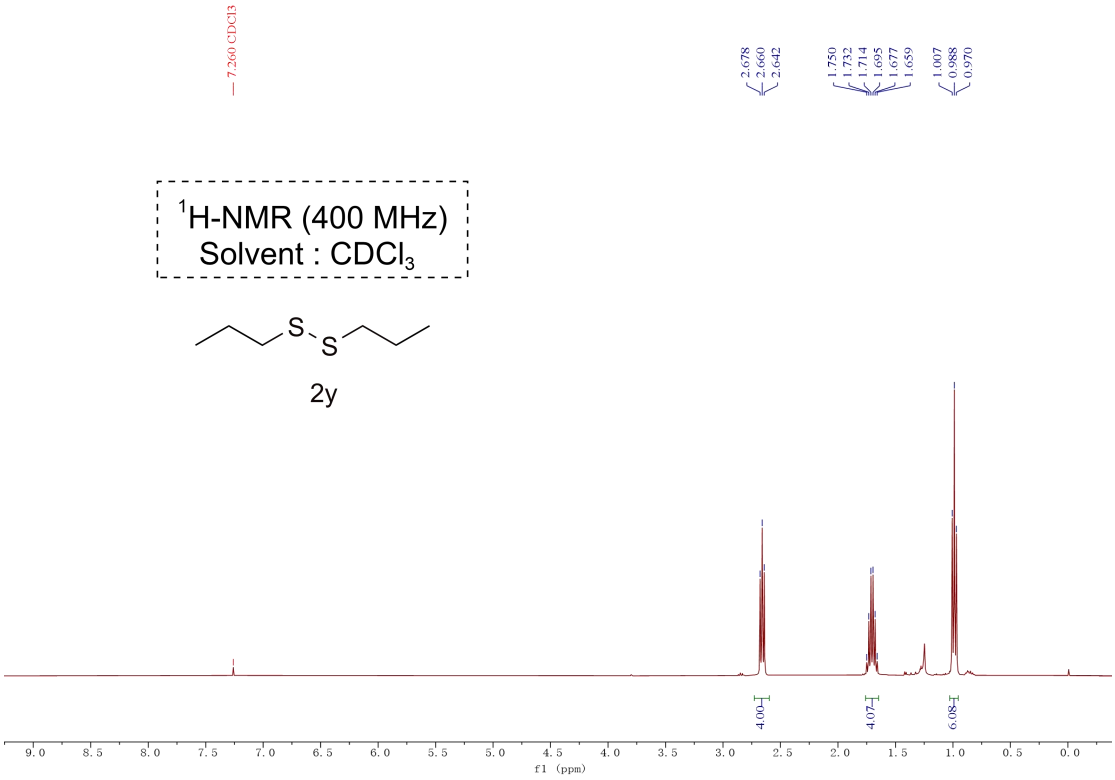
2v

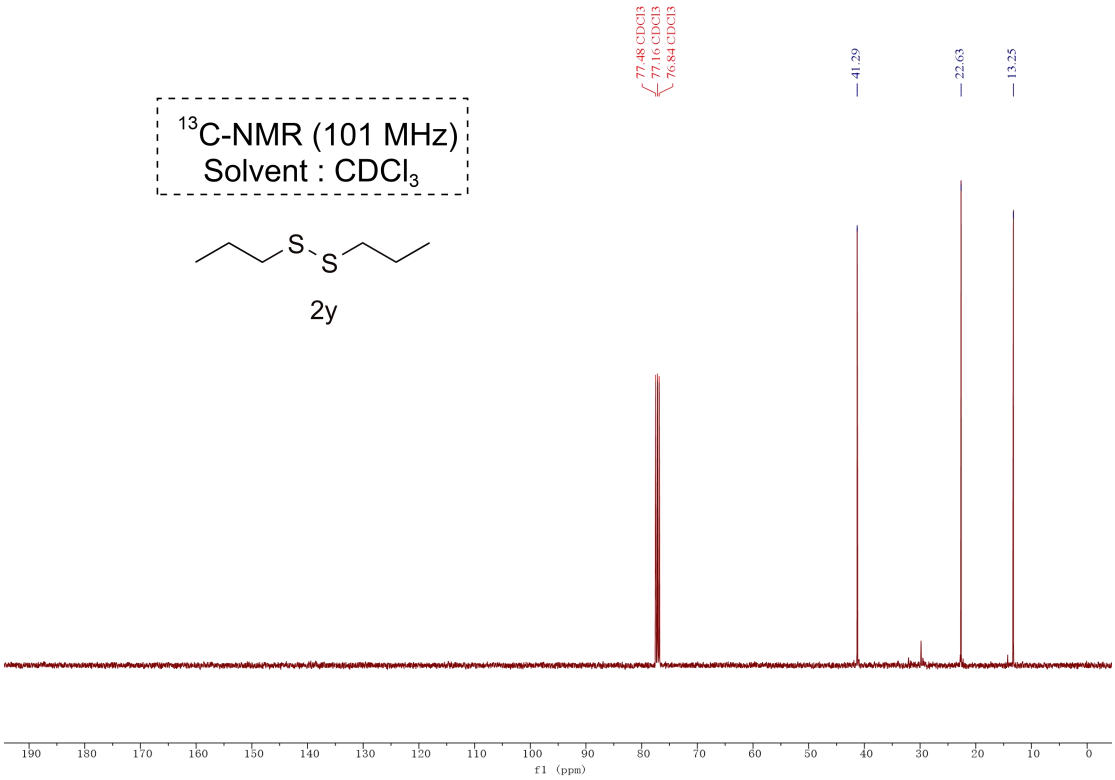


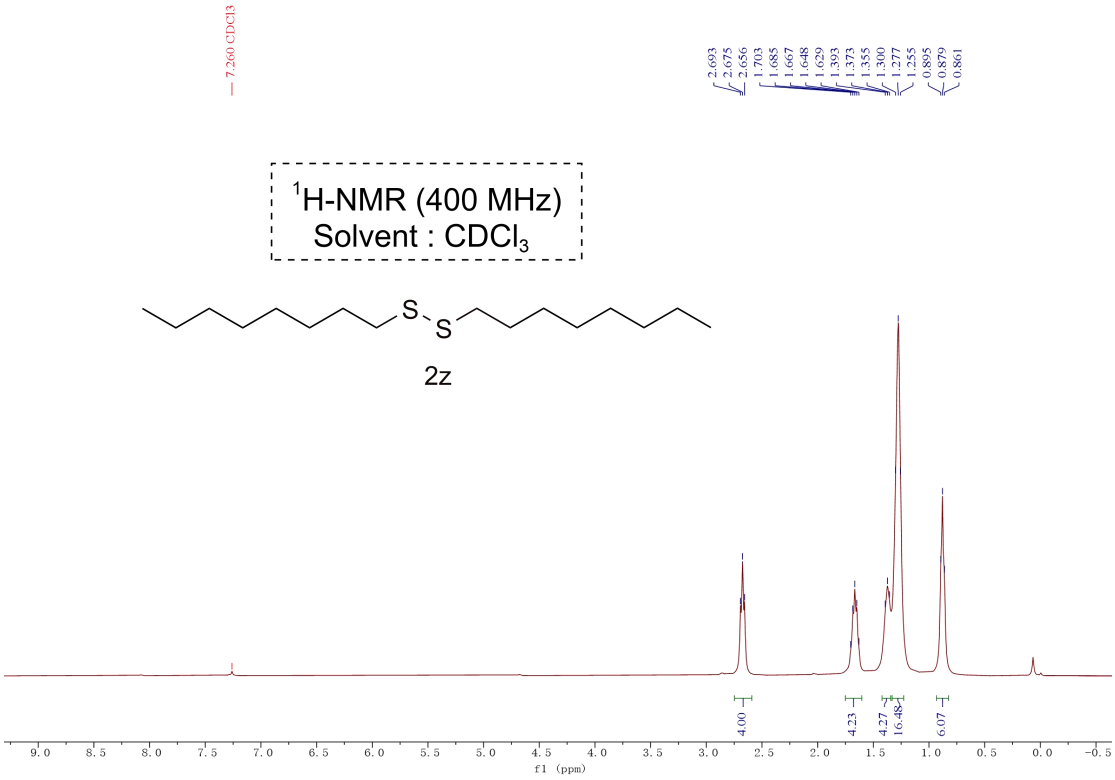

8.578
8.566
7.260 CDCl3
7.095
7.083
7.071
1H-NMR (400 MHz)
Solvent : CDCl3
2W
4.00
2.08
f1 (ppm)

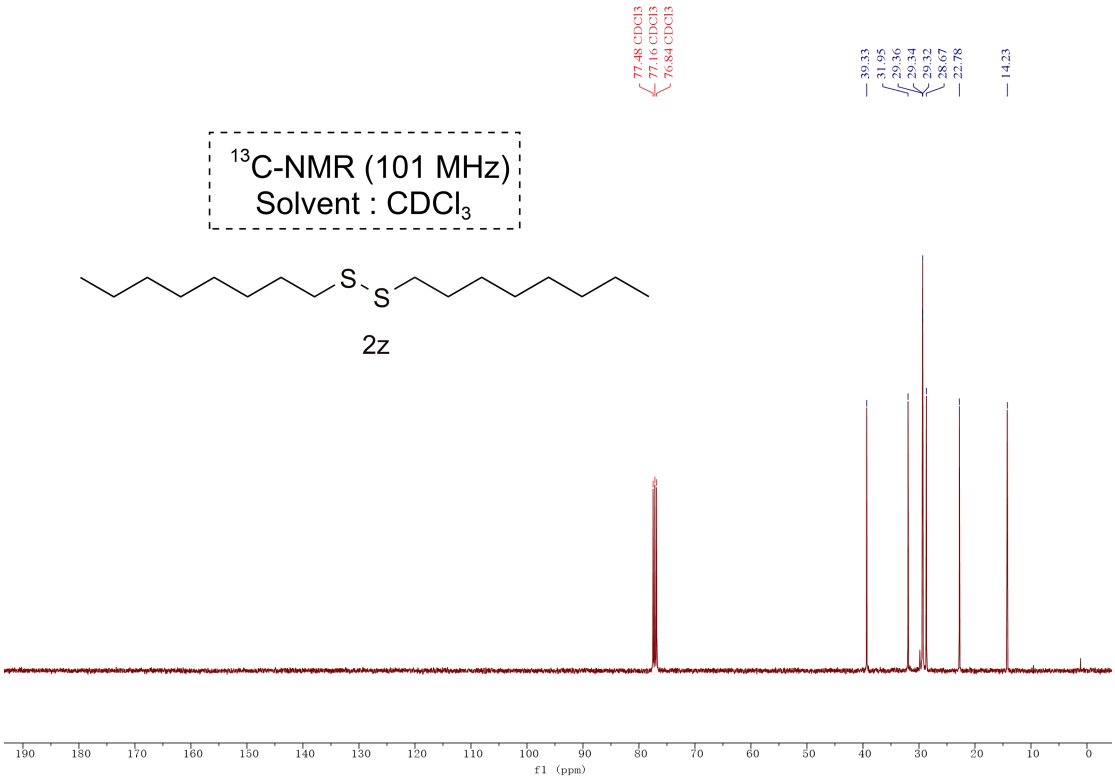

169.83
158.04
118.32
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
13C-NMR (101 MHz)
Solvent : CDCl3
2W
f1 (ppm)

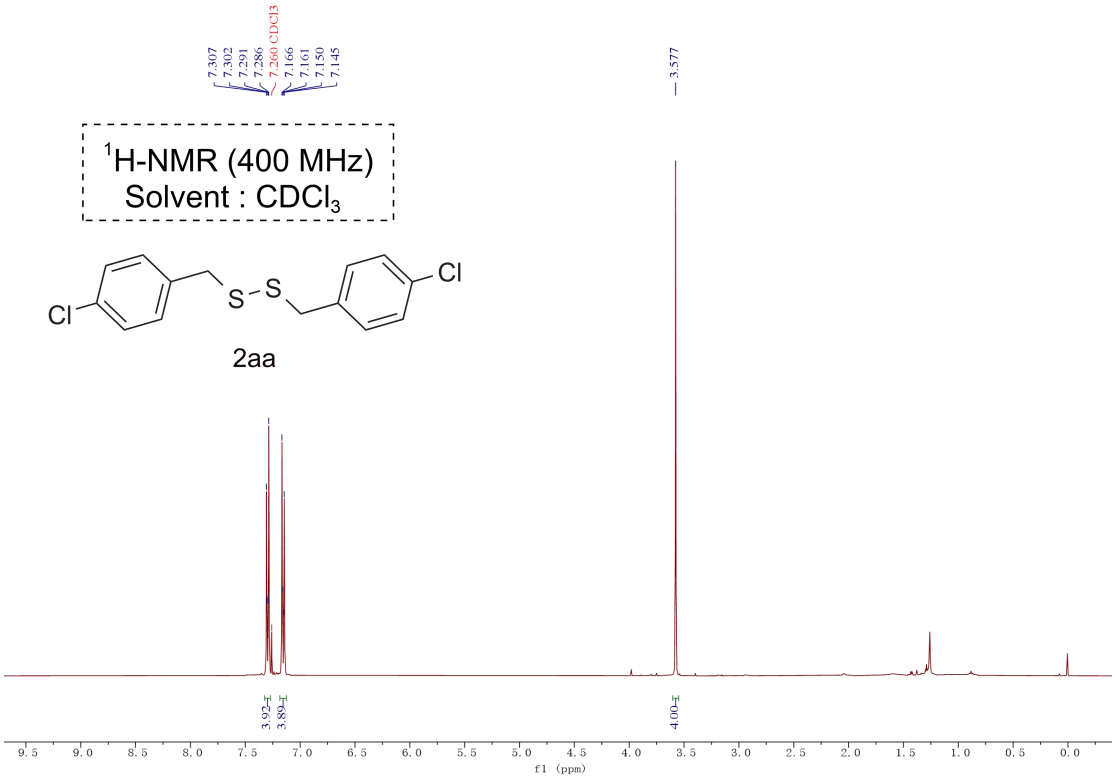

7.260 cdcl3
2.712
2.702
2.693
2.686
2.676
2.666
2.650
2.640
2.060
2.048
2.034
2.024
2.013
1.795
1.785
1.772
1.762
1.755
1.746
1.626
1.613
1.600
1.589
1.325
1.310
1.297
1.277
1.266
1.245
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2x
2.00
4.14
4.16
2.40
10.34
f1 (ppm)
77.48 cdcl3
77.16 cdcl3
76.84 cdcl3
50.07
32.96
26.22
25.81
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2x
f1 (ppm)

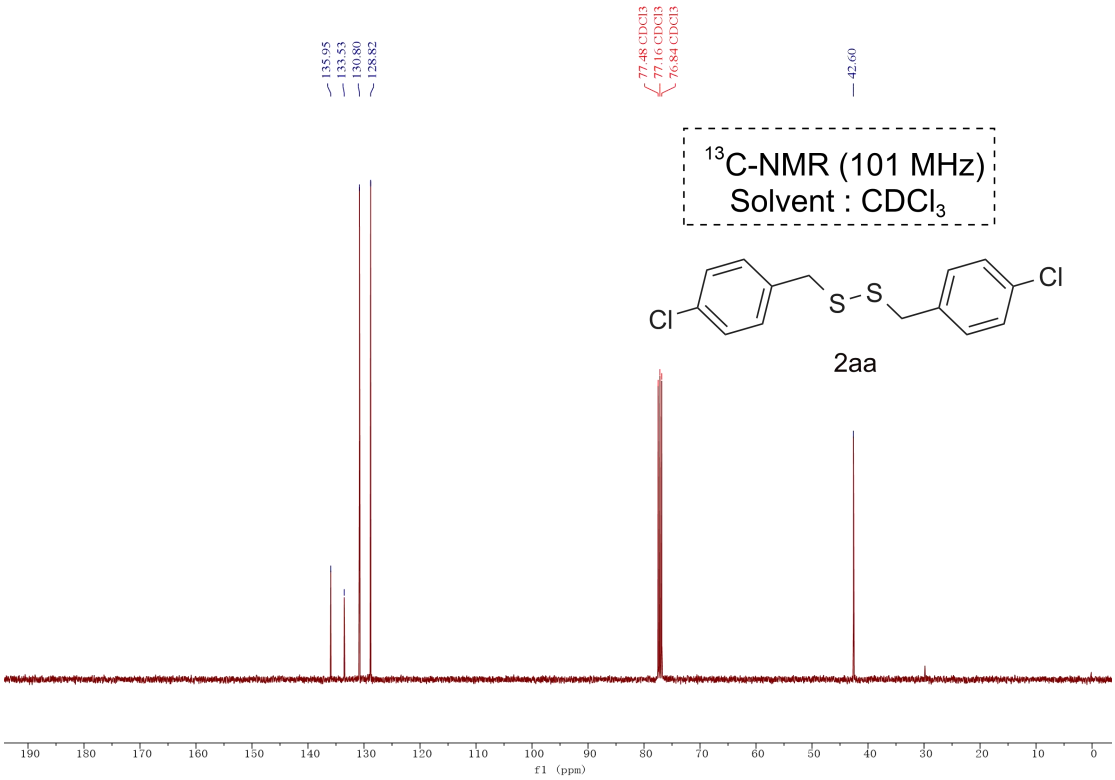

7.260 CDCl3
2.678
2.660
2.642
1.750
1.732
1.714
1.695
1.677
1.659
1.007
0.988
0.970
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2y
4.00
4.07
6.08
f1 (ppm)

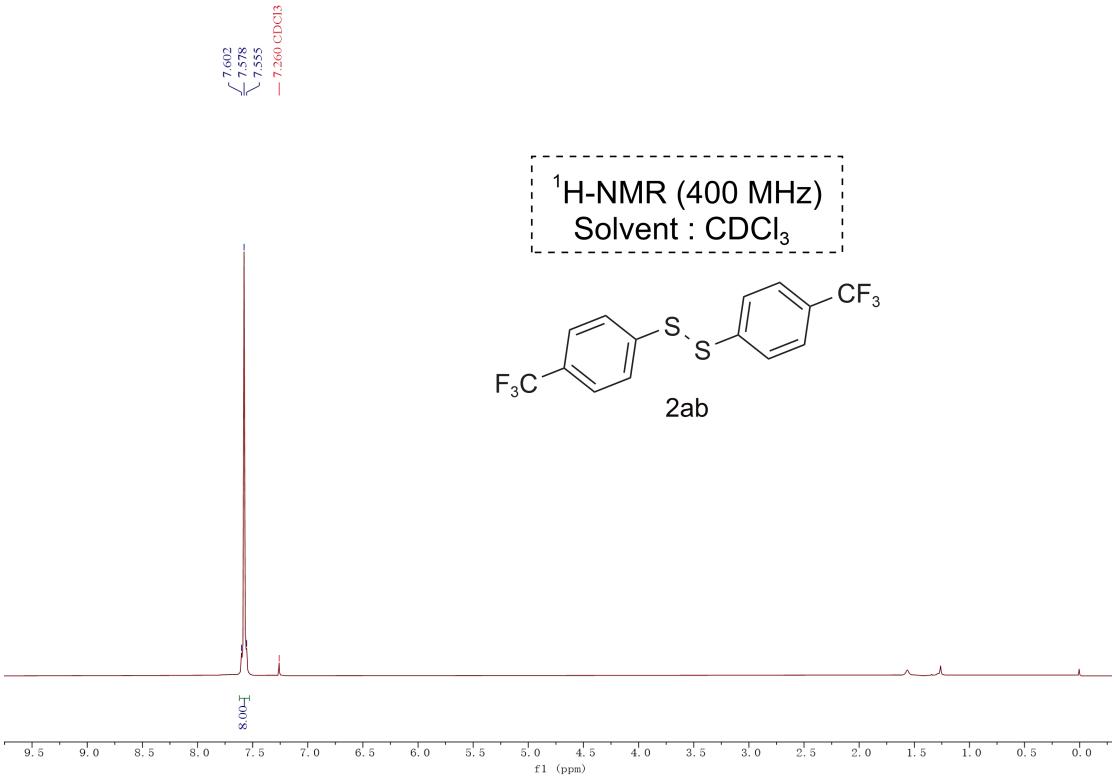

77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
41.29
22.63
13.25
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2y
f1 (ppm)

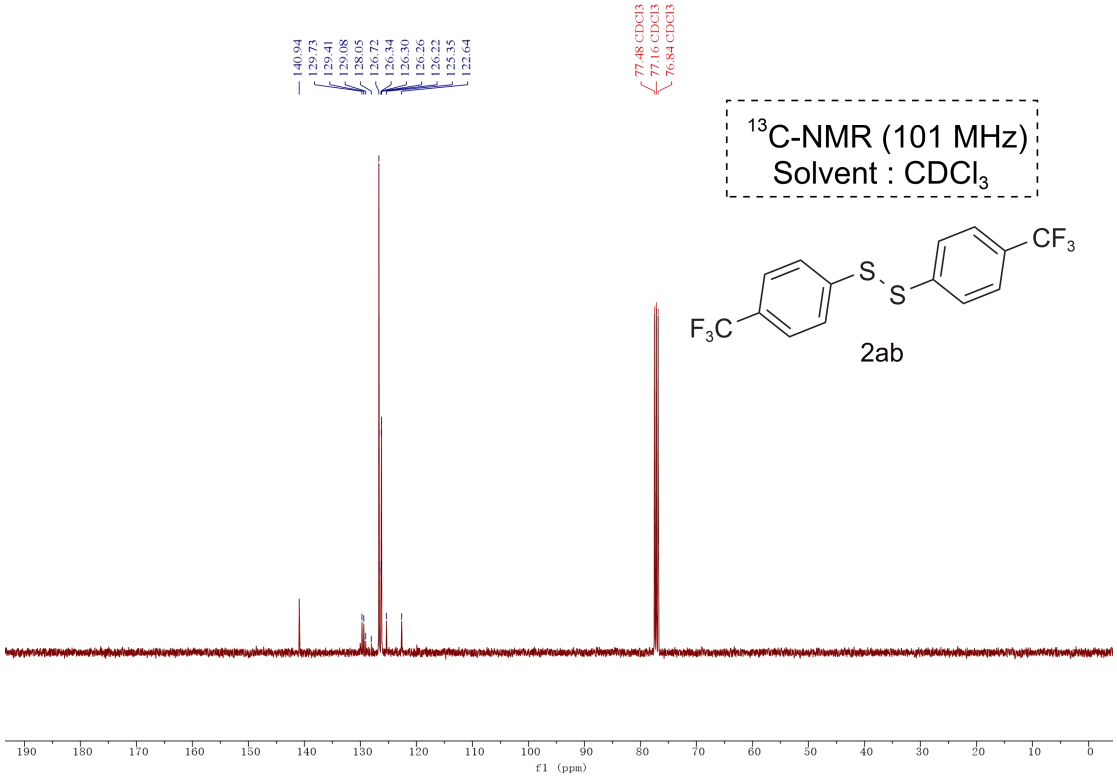

7.260 CDCl3
2.693
2.675
2.656
1.703
1.685
1.667
1.648
1.629
1.393
1.373
1.355
1.300
1.277
1.255
0.895
0.879
0.861
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
2z
4.00
4.23
4.27
16.48
6.07
f1 (ppm)

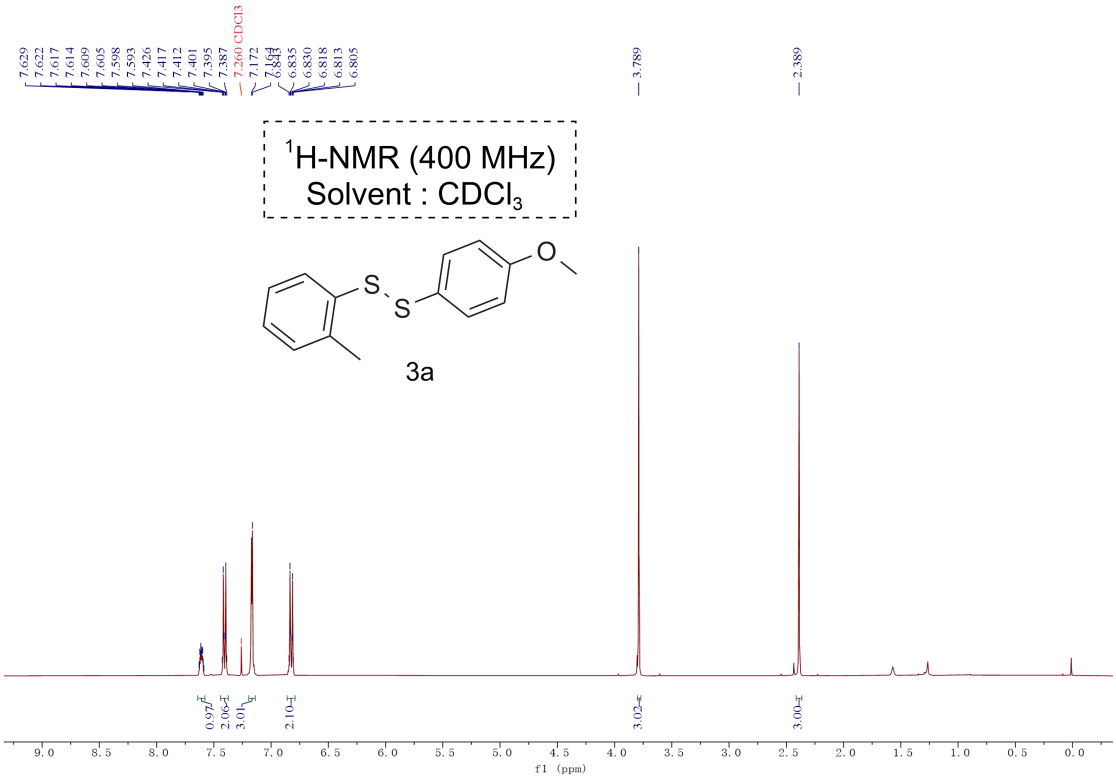

77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
39.33
31.95
29.36
29.34
29.32
28.67
22.78
14.23
13C-NMR (101 MHz)
Solvent : CDCl3
S
S
2z
f1 (ppm)

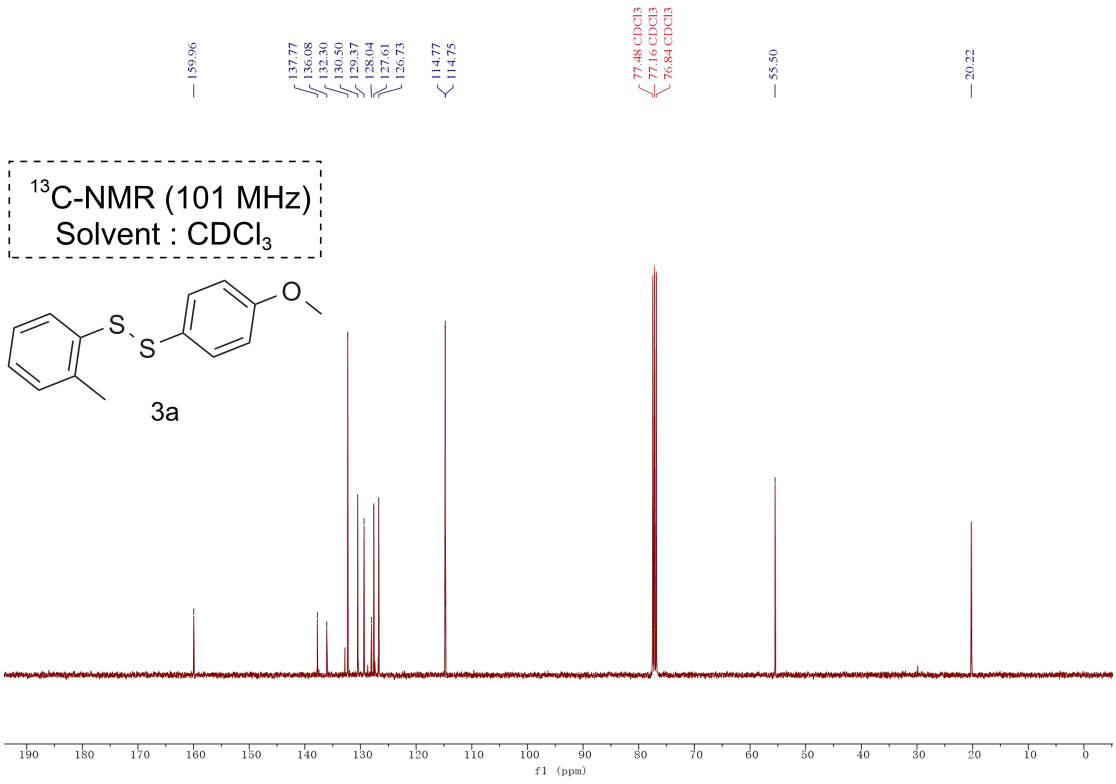

7.307
7.302
7.291
7.286
7.260 CDCl3
7.166
7.161
7.150
7.145
3.577
1H-NMR (400 MHz)
Solvent : CDCl3
Cl
S
S
Cl
2aa
3.92
3.89
4.00
f1 (ppm)

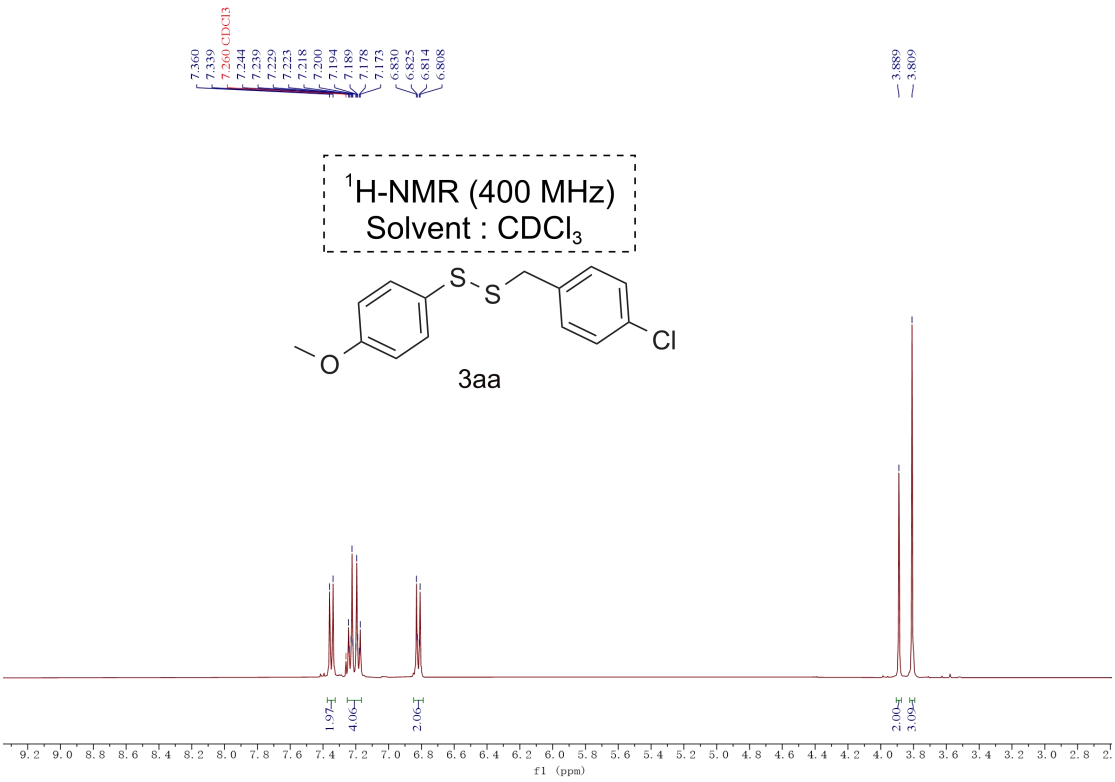

135.95
133.53
130.80
128.82
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
42.60
13C-NMR (101 MHz)
Solvent : CDCl3
Cl
S
S
Cl
2aa
f1 (ppm)

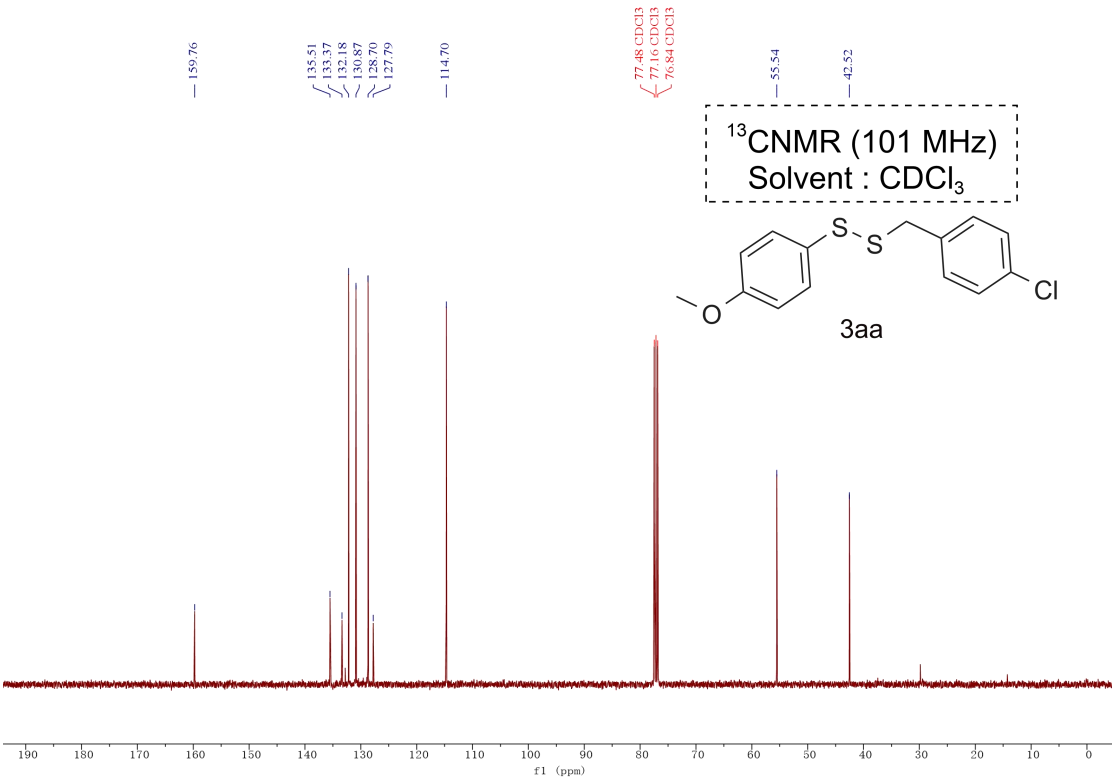

7.602
7.578
7.555
7.260 CDCl3
1H-NMR (400 MHz)
Solvent : CDCl3
CF3
S
S
F3C
2ab
8.00
f1 (ppm)

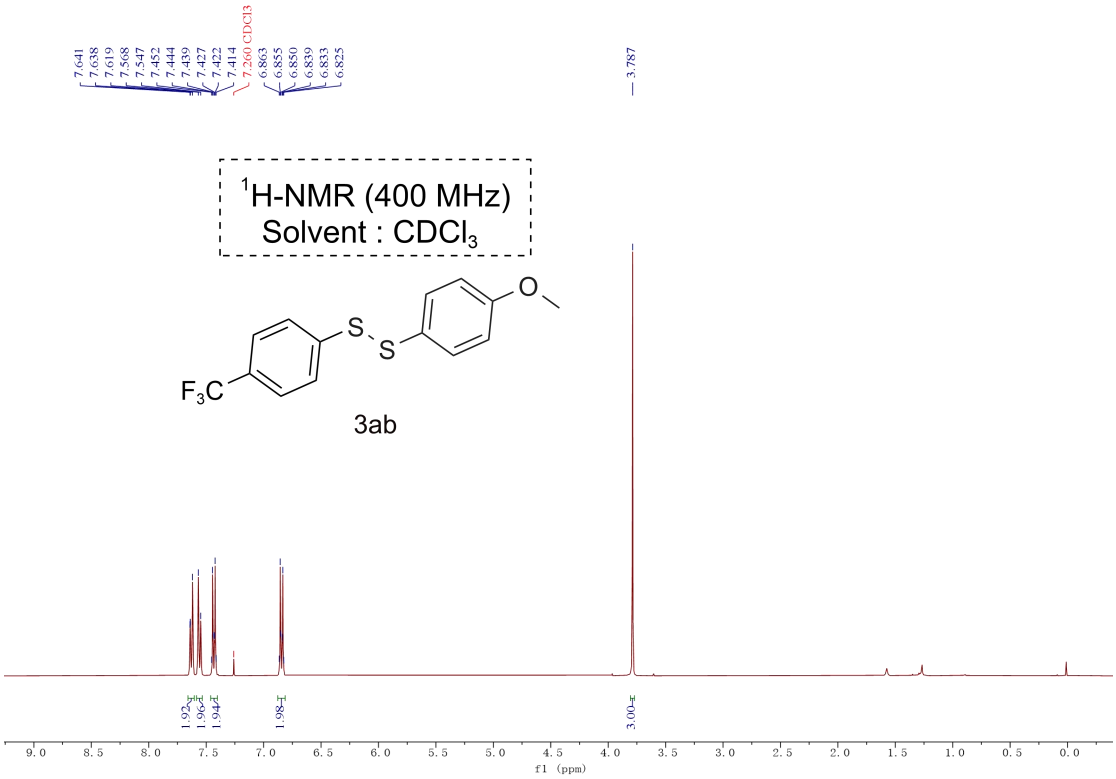

140.94
129.73
129.41
129.08
128.05
126.72
126.34
126.30
126.26
126.22
125.35
122.64
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
13C-NMR (101 MHz)
Solvent : CDCl3
CF3
S
S
F3C
2ab
f1 (ppm)

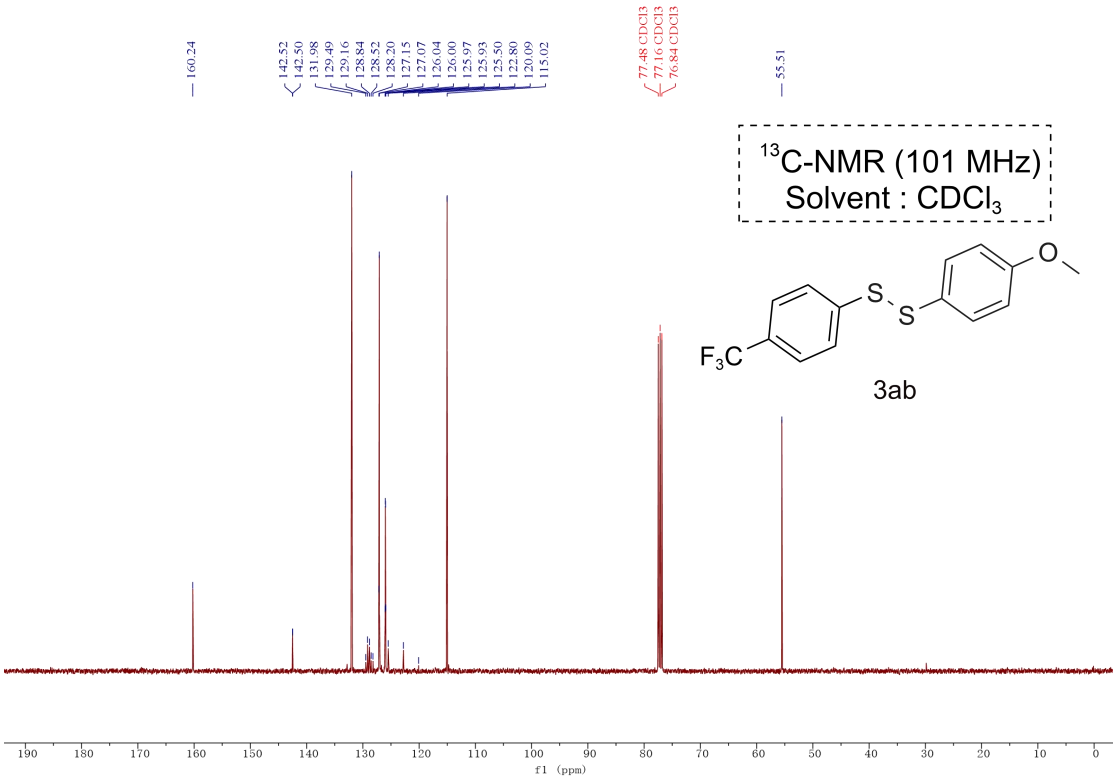

7.629
7.622
7.617
7.614
7.609
7.605
7.598
7.593
7.426
7.417
7.412
7.401
7.395
7.387
7.260 CDCl3
7.172
7.164
6.844
6.835
6.830
6.818
6.813
6.805
3.789
2.389
^{1}H-NMR (400 MHz)
Solvent : $CDCl_3$
S
S
O
3a
0.97
2.06
3.01
2.10
3.02
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.96
137.77
136.08
132.30
130.50
129.37
128.04
127.61
126.73
114.77
114.75
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
55.50
20.22
^{13}C-NMR (101 MHz)
Solvent : $CDCl_3$
S
S
O
3a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.360
7.339
7.260 CDCl3
7.244
7.239
7.229
7.223
7.218
7.200
7.194
7.189
7.178
7.173
6.830
6.825
6.814
6.808
3.889
3.809
1H-NMR (400 MHz)
Solvent : CDCl3
S
S
Cl
O
3aa
1.97
4.06
2.06
2.00
3.09
f1 (ppm)


159.76
135.51
133.37
132.18
130.87
128.70
127.79
114.70
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
55.54
42.52
13CNMR (101 MHz)
Solvent : CDCl3
S
S
Cl
O
3aa
f1 (ppm)


7.641
7.638
7.619
7.568
7.547
7.452
7.444
7.439
7.427
7.422
7.414
7.260 CDCl3
6.863
6.855
6.850
6.839
6.833
6.825
3.787
1H-NMR (400 MHz)
Solvent : CDCl3
O
S
S
F3C
3ab
1.92
1.96
1.94
1.98
3.00
f1 (ppm)


160.24
142.52
142.50
131.98
129.49
129.16
128.84
128.52
128.20
127.15
127.07
126.04
126.00
125.97
125.93
125.50
122.80
120.09
115.02
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
55.51
13C-NMR (101 MHz)
Solvent : CDCl3
O
S
S
F3C
3ab
f1 (ppm)