Supporting Information

Fe-Catalyzed Direct Dithioacetalization of Aldehydes with 2-Chloro-1,3-dithiane

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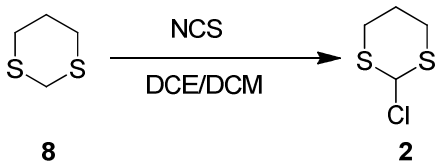
1. General information

Analytical grade solvents and commercially available reagents were used as received. Substituted aldehydes were all purchased from commercial sources and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were generally performed on silica gel (200-300 mesh) in petroleum (bp. 60-90 ^{o}C) and reactions were monitored by thin layer chromatography (TLC) using silica gel GF254 plates with UV light to visualize the course of reaction. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution on the Bruker 300 instruments, and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard. MS were measured on a HP-5988 spectrometer by direct inlet at 70 eV.

2. Optimization of conditions

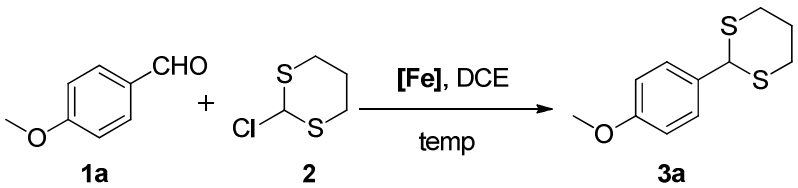
2.1. General procedure for preparation of 2-chloro-1,3-dithiane[1]



To a flame-dried 10 mL flask were sequentially added 1,3-dithiane **8** (300 mg, 2.5 mmol) and N-chlorosuccinimide (NCS) (400 mg, 3 mmol), 1,2-dichloroethane (DCE) or dichloromethane (DCM) (3 mL), after dissolved the mixture was stirred at 0 ^{o}C for 40 mins. The mixture was allowed to stir at room temperature for 2h. Then the reaction mixture was filtered to remove insoluble materials, and the filtrate immediately concentrated on a rotary evaporator at room temperature to yield 2-chloro-1,3-dithiane **2** as a pale-yellow solid **(**estimated to 90-95 % purity**).**[1]

Alternately, the reaction mixture in DCE can be used directly for the dithioacetalization in a one-pot procedure without purification.

2.2 Optimization of catalysts, additives, and temperature

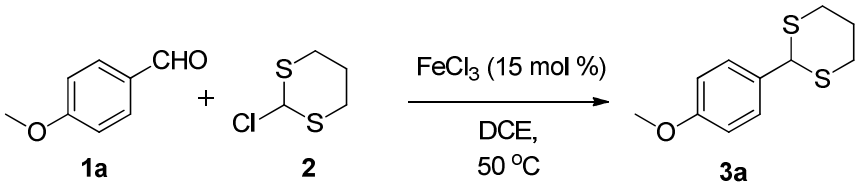


To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (38 mg, 0.25 mmol), **1a** (31 mg, 0.225 mmol) and catalyst (*x* mol %) were added at reaction temperature. Reaction mixture was stirred at 25 ^{o}C - 80 ^{o}C for 4 - 24 h until TLC analysis showed the reaction was completed. The mixture was concentrated under reduced pressure, and then purified by column chromatography on silica gel with petroleum/ethyl acetate (EA : PE = 1 : 50) to yield the **3a** product.

Table S1. Radical oxidative cross-coupling between **1a** and **2** under reaction conditions

entry	cat. (*x* mol %)	additives	temp (^{o}C)	time (h)	yield (%)
1	$FeCl_3$ (5)	-	50	18	43
2	$FeCl_3$ (15)	-	50	4	89
3	$FeCl_3$ (30)	-	50	3	88
4	$FeCl_2$ (5)	-	50	18	46
5	$FeCl_2$ (15)	-	50	6	86
6	$FeCl_2 \cdot 4H_2O$ (15)	-	50	48	80
7	$FeCl_3 \cdot 6H_2O$ (15)	-	50	4	87
8	$FeCl_3$ (15)	-	25	48	60
9	$FeCl_3$ (15)	-	80	4	85
10	$FeCl_3$ (15)	DTBP[2]	50	4	85
11	$FeCl_3$ (15)	H_2O_2[3]	50	48	trace
12	$FeCl_3$ (15)	TBHP[4]	50	48	trace
13	$FeCl_3$ (15)	DDQ[5]	50	48	trace
14	none	DTBP	50	48	0

2.3. Optimization of solvent

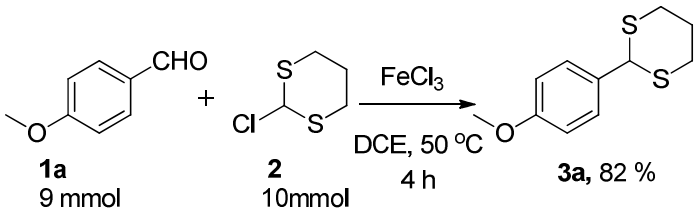


To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (38 mg, 0.25 mmol) and **1a** (31 mg, 0.225 mmol) were added simultaneously under air in solvent (2 mL), and $FeCl_3$ (6 mg, 15 mol %) were added finally at reaction temperature. Then reaction mixture was stirred at 50 ^{o}C for 4h. Then the reaction mixture was concentrated under reduced pressure, and then purified by column chromatography on silica gel with petroleum/ethyl acetate (EA : PE = 1 : 50) to yield **3a** product.

Table S2 Solvent effects on the coupling reaction between 1a and 2

entry	solvent	yield(%)	entry	solvent	yield(%)
1	DCM	80	6	EtOH	trace
2	DCE	89	7	DMSO	0
3	MeCN	trace	8	$MeNO_2$	trace
4	EtOAc	trace	9	DMF	0
5	MeOH	trace	10	toluene	44

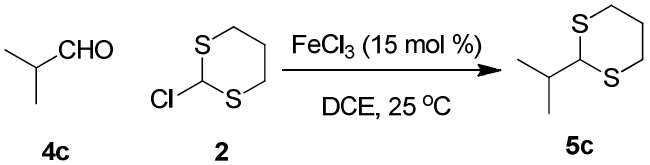
3. General procedure for the gram scale experiment



To a flame-dried 100 mL flask 2-chloro-1,3-dithiane (1.50 g, 10 mmol), 4-methoxybenzaldehyde **1a** (1.26 g, 9 mmol), DCE (50 mL) and $FeCl_3$ (162.5 mg, 1 mmol) were added under air. Reaction mixture was allowed to stir at 50 ^{o}C for 4 h until TLC analysis showed the reaction was completed. After the mixture was concentrated under reduced pressure, and then purified by column chromatography on silica gel with petroleum/ethyl acetate (EA : PE = 1 : 50) to the desired product in 82% (1.69 mg) yield.

4. Mechanistic investigations

4.1. EPR experiments. [6-10]



a) Procedure for EPR investigation of isobutyraldehyde 4g, $FeCl_3$, and 2-chloro-1,3-dithiane 2.

Isobutyraldehyde **4g** (7.2 mg, 0.1 mmol), $FeCl_3$ (16.3 mg, 0.1 mmol) and 2-chloro-1,3-dithiane **2** (15 mg, 0.1 mmol) which was given to the reaction of 1,3-dithiane (12 mg, 0.1 mmol) and NCS (15 mg, 0.1 mmol) , dissolved in 1 mL DCE, the mixture was stirred at room temperature for few minutes, then few of this solution was taken out by a capillary tube and sealed

up, then analyzed by EPR at room temperature, respectly (Figure S1).

b) Procedure for EPR investigation of the reaction between $FeCl_3$, isobutyraldehyde 4g or 2-chloro-1,3-dithiane 2.

To a flame-dried 10 mL flask were sequentially added $FeCl_3$ (2.4 mg, 0.015 mmol) and 1 mL DCE. Then 2-chloro-1,3-dithiane **2** (15 mg, 0.1 mmol) or isobutyraldehyde **4g** (7.2 mg, 0.1 mmol) was added. After the mixture was stirred for few minutes, few of this solution was taken out by a capillary tube and sealed up, then analyzed by EPR at room temperature, respectly (Figure S1, yellow & black line).

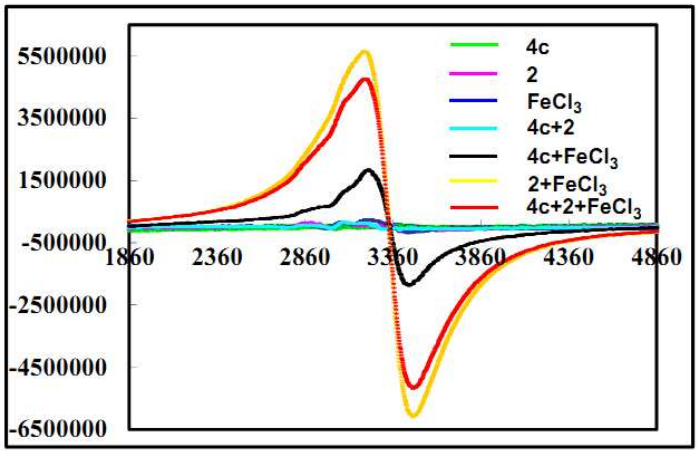
c) Procedure for EPR investigation of the mixture of isobutyraldehyde 4g and 2-chloro-1,3-dithiane 2.

To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (15 mg, 0.1 mmol), isobutyraldehyde **4g** (7.2 mg, 0.1 mmol) and 1 mL DCE were combined. After the mixture was stirred for few minutes, few of this solution was taken out by a capillary tube and sealed up, then analyzed by EPR at room temperature, respectly (Figure S1).

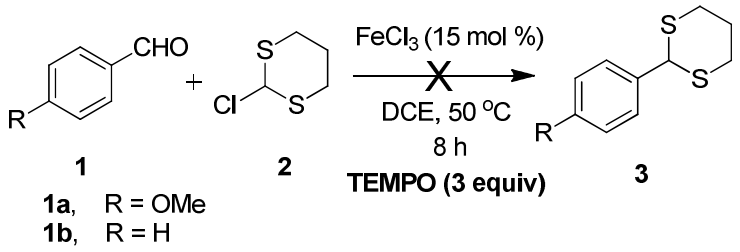
d) Procedure for EPR investigation of the reaction between isobutyraldehyde 4g and 2-chloro-1,3-dithiane 2.

To a flame-dried 10 mL flask were sequentially added isobutyraldehyde **4g** (7.2 mg, 0.1 mmol), $FeCl_3$ (2.4 mg, 0.015 mmol) and 2-chloro-1,3-dithiane **2** (15 mg, 0.1 mmol), at room temperature. After the mixture was stirred for few minutes, few of this solution was taken out by a capillary tube and sealed up, then analyzed by EPR at room temperature (Figure S1, red line).

Figure S1. EPR investigation of isobutyraldehyde **4c** and 2-chloro-1,3-dithiane **2**

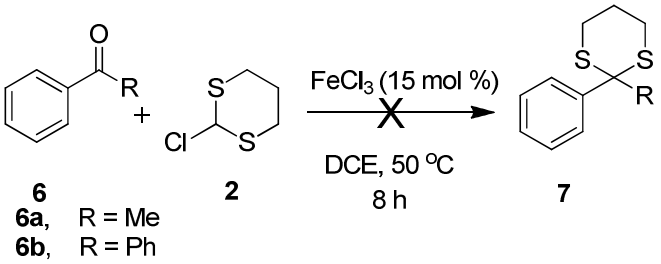


4.2. The procedure for radical trapping experiments[11-13]



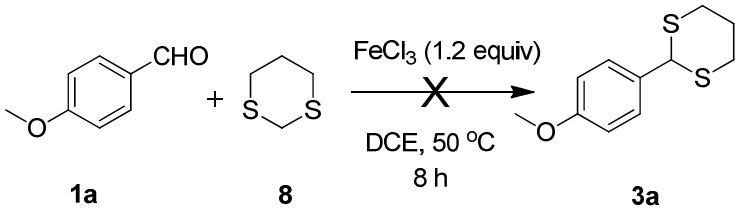
To a flame-dried 10 mL flask were sequentially added 2-chloro-1,3-dithiane (38 mg, 0.250 mmol), TEMPO (47 mg, 0.3 mmol), **1a** or **1b** (0.225 mmol) and $FeCl_3$ (5 mg, 0.03 mmol). Reaction mixture was stirred at 50 ^{o}C for 8 h. Then reaction mixture was diluted with ethyl acetate (3 mL) and H_2O (1 mL). The organic layer was separated, and the aqueous phase was re-extracted with ethyl acetate (3×3 mL). The combined organic extracts were washed with H_2O (10 mL), and dried over anhydrous Na_2SO_4.

4.3. The reaction of ketones with 2-chloro-1,3-dithiane



To a flame-dried 10 mL flask were sequentially added 2-chloro-1,3-dithiane (38.0 mg, 0.25 mmol), **6a** or **6b** (0.225 mmol) and $FeCl_3$ (5 mg, 0.03 mmol). Reaction mixture was stirred at 50 ^{o}C for 8 h. Then reaction mixture was diluted with ethyl acetate (3 mL) and H_2O (1 mL). The organic layer was separated, and the aqueous phase was re-extracted with ethyl acetate (3×3 mL). The combined organic extracts were washed with H_2O (10 mL), and dried over anhydrous Na_2SO_4.

4.4. The reaction of 1a with 1,3-dithiane 8

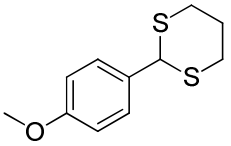


To a flame-dried 10 mL flask 1,3-dithiane (30 mg, 0.25 mmol), **1a** (31 mg, 0.225 mmol) and

$FeCl_3$ (49 mg, 0. 3 mmol) were added. Reaction mixture was stirred at 50 ^oC for 8 h. Then reaction mixture was diluted with ethyl acetate (3 mL) and H_2O (1 mL). The organic layer was separated, and the aqueous phase was re-extracted with ethyl acetate (3×3 mL). The combined organic extracts were washed with H_2O (10 mL), and dried over anhydrous Na_2SO_4.

5. ^{1}H NMR and ^{13}C NMR spectra data of products

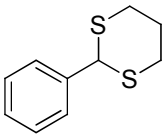
2-(4-methoxyphenyl)-1,3-dithiane (3a) [14]



3a

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.37 (d, *J* = 8.0 Hz, 2H), 6.83 (d, *J* = 7.9 Hz, 2H), 5.10 (s, 1H), 3.75 (t, *J* = 1.1 Hz, 3H), 3.02 (t, *J* = 13.3 Hz, 2H), 2.85 (m, 2H), 2.13-2.08 (m, 1H), 1.88-1.84 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 159.79, 131.56, 129.19, 114.33, 55.56, 50.98, 32.45, 25.33. **MS (EI, 70 ev) m/z:** 226, 152, 135, 121, 97.

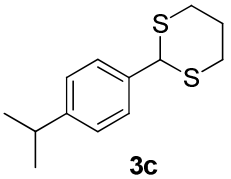
2-phenyl-1,3-dithiane (3b) [15]



3b

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.66-7.41 (m, 2H), 7.41-7.13 (m, 3H), 5.16 (s, 1H), 3.12-2.98 (m, 2H), 2.98-2.79 (m, 2H), 2.23-2.02 (m, 1H), 2.00-1.81 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 139.02, 128.63, 128.33, 127.65, 51.41, 32.00, 25.17, 25.01. **MS (EI, 70 ev) m/z:** 197, 135, 121, 105, 91.

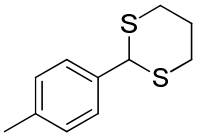
2-(4-isopropylphenyl)-1,3-dithiane (3c) [17]



3c

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46-7.22 (m, 2H), 7.10 (dd, *J* = 5.2, 2.9 Hz, 2H), 5.08 (s, 1H), 2.96-2.67 (m, 5H), 1.97-1.95 (m, 1H), 1.80-170 (m, 1H), 1.23-1.09 (d, *J* = 9 Hz, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 148.50, 136.14, 127.21, 126.29, 50.72, 33.37, 31.66, 29.43, 24.68, 23.52. **MS (EI, 70 ev) m/z:** 238, 164, 149, 131, 117, 105, 91.

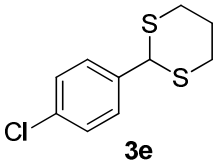
2-(p-tolyl)-1,3-dithiane (3d) [14]



3d

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.12 (t, *J* = 6.7 Hz, 2H), 5.12 (s, 1H), 3.03 - 2.91 (m, 2H), 2.82-2.78 (m, 2H), 2.30 (s, 3H), 2.12-1.98 (m, 1H), 1.86-1.83 (m, 6.1 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 137.82, 135.91, 129.02, 127.25, 50.81, 31.76, 24.77, 20.91. **MS (EI, 70 ev) m/z:** 210, 147, 136, 119, 105, 97.

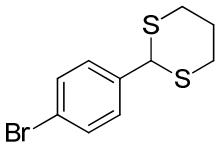
2-(4-chlorophenyl)-1,3-dithiane (3e) [14]



3e

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46-7.29 (m, 2H), 7.29-7.13 (m, 2H), 5.06 (s, 1H), 3.07-2.89 (m, 2H), 2.81-2.78 (m, 2H), 2.14-1.98 (m, 1H), 1.93-1.74 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 137.48, 133.87, 129.01, 128.70, 50.33, 31.77, 24.75. **MS (EI, 70 ev) m/z:** 230, 155, 129, 112, 55, 45.

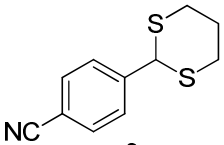
2-(4-bromophenyl)-1,3-dithiane (3f) [16]



3f

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.5 Hz, 2H), 5.03 (s, 1H), 2.96-2.84 (m, 2H), 2.79-2.74 (m, 2H), 2.06-1.88 (m, 1H), 1.88-1.67 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 137.89, 131.52, 129.22, 121.92, 50.24, 31.62, 24.63. **MS (EI, 70 ev) m/z:** 276, 274, 202, 153, 130, 120, 105.

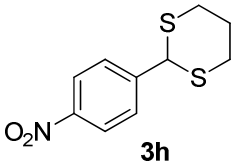
4-(1,3-dithian-2-yl)benzonitrile (3g) [15]



3g

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.63-7.54 (dt, *J* = 15.9, 4.3 Hz, 4H), 5.18 (s, 1H), 3.08-2.99 (m, 2H), 2.91-2.86 (m, 2H), 2.26-2.04 (m, 1H), 2.00-1.72 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 143.84, 132.09, 128.26, 118.05, 111.55, 50.14, 31.26, 24.37. **MS (EI, 70 ev) m/z:** 221, 156, 146, 130, 121, 105.

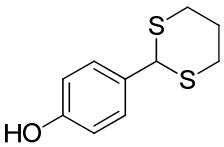
2-(4-nitrophenyl)-1,3-dithiane (3h) [14]



3h

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.79 (d, *J* = 8.7 Hz, 2H), 7.24 (d, *J* = 8.7 Hz, 2H), 4.84 (s, 1H), 2.77-2.58 (m, 2H), 2.58-2.40 (m, 2H), 1.88-1.70 (m, 1H), 1.59-1.50 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 147.44, 146.04, 128.74, 123.76, 50.16, 31.54, 24.62. **MS (EI, 70 ev) m/z:** 241, 176, 166, 130, 121.

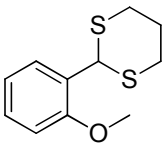
4-(1,3-dithian-2-yl)phenol (3i) [14]



3i

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.40 (d, *J* = 12.4 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 5.21 (s, 2H), 3.18-3.10 (m, 2H), 3.05-2.89 (m, 2H), 2.26-2.20 (m, 1H), 2.02-1.97 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.63, 155.53, 131.36, 129.12, 115.53, 50.69, 32.14, 25.00. **MS (EI, 70 ev) m/z:** 212, 167, 147, 137, 123, 97.

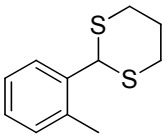
2-(2-methoxyphenyl)-1,3-dithiane (3j) [18]



3j

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.64-7.41 (m, 1H), 7.29-7.11 (m, 1H), 6.93-6.88 (m, 1H), 6.79 (d, *J* = 8.3 Hz, 1H), 5.65 (s, 1H), 3.77 (s, 3H), 3.02 (t, *J* = 13.4 Hz, 2H), 2.80 (t, *J* = 14.0 Hz, 2H), 2.17-1.98 (m, 1H), 1.88-1.80 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 155.15, 129.15, 128.89, 127.07, 120.75, 110.52, 55.50, 43.39, 32.15, 25.09. **MS (EI, 70 ev) m/z:** 226, 161, 152, 137, 119, 107, 91.

2-(o-tolyl)-1,3-dithiane (3k) [18]

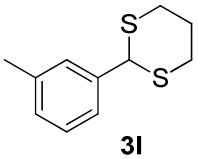


3k

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.61 (dd, *J* = 4.1, 2.5 Hz, 1H), 7.27-7.07 (m, 3H), 5.32 (s, 1H), 3.05 (t, *J* = 13.0 Hz, 2H), 2.87 (t, *J* = 14.4 Hz, 2H), 2.44 (d, *J* = 1.1 Hz, 3H), 2.19-2.06 (m, 1H), 1.92-1.88 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 136.98, 134.80, 130.31,

127.97, 127.72, 126.38, 48.17, 47.90, 32.17, 25.02, 18.90. **MS (EI, 70 ev) m/z:** 210, 145, 135, 121, 105, 91.

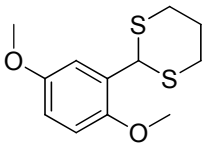
2-(m-tolyl)-1,3-dithiane (3l) [18]



3l

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.55-7.31 (m, 3H), 7.24 (d, *J* = 7.2 Hz, 1H), 5.28 (s, 1H), 3.21-3.09 (m, 2H), 3.07-2.93 (m, 2H), 2.48 (s, 3H), 2.35-2.18 (m, 1H), 2.06-2.00 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 138.73, 138.02, 128.85, 128.23, 128.00, 124.45, 51.14, 31.76, 24.80, 21.04. **MS (EI, 70 ev) m/z:** 211, 210**,** 152, 136, 120, 105.

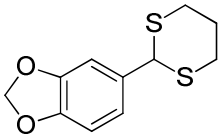
2-(2,5-dimethoxyphenyl)-1,3-dithiane (3m) [19]



3m

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 6.76 (d, *J* = 1.8 Hz, 1H), 6.39 (d, *J* = 4.8 Hz, 2H), 5.27 (s, 1H), 3.49-3.28 (m, 6H), 2.76-2.58 (m, 2H), 2.49-2.43 (m, 2H), 1.79-1.65 (m, 1H), 1.60-1.41 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 153.48, 149.17, 127.83, 114.32, 111.75, 55.99, 55.39, 43.48, 43.37, 31.97, 24.90. **MS (EI, 70 ev) m/z:** 256**,** 182, 149, 137, 121, 108, 91.

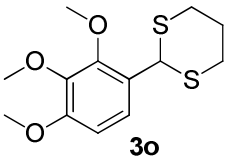
5-(1,3-dithian-2-yl)benzo[d][1,3]dioxole (3n) [14]



3n

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.01-6.85 (m, 2H), 6.72 (d, *J* = 8.0 Hz, 1H), 5.89 (s, 2H), 5.05 (s, 1H), 3.03-2.92 (m, 2H), 2.84-2.79 (m, 2H), 2.14-2.04 (m, 1H), 1.85-1.81 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 147.48, 147.34, 132.72, 121.01, 108.08, 101.00, 50.90, 31.89, 24.78. **MS (EI, 70 ev) m/z:** 240**,** 166, 147, 135, 122, 101.

2-(2,3,4-trimethoxyphenyl)-1,3-dithiane (3o) [20]

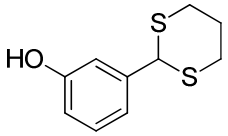


3o

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.7 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 1H), 5.75 (s, 1H), 4.25-3.89 (m, 9H), 3.37-3.19 (m, 2H), 3.08-3.03 (m, 2H), 2.38-2.31 (m, 1H), 2.09-2.05 (m, 1H).

Colorless oil. **^{13}C NMR (75 MHz, $CDCl_3$)** δ 153.34, 150.16, 141.63, 125.03, 123.15, 107.50, 61.57, 60.48, 55.68, 43.60, 32.16, 24.95. **MS (EI, 70 ev) m/z:** 286**,** 212, 179, 151, 136, 120, 106.

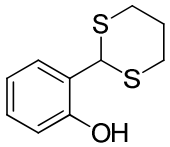
3-(1,3-dithian-2-yl)phenol (3p) [18]



3p

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.16-7.11 (m, 1H), 7.03-6.86 (m, 2H), 6.73-6.69 (m, 1H), 5.51 (s, 1H), 5.06 (s, 1H), 2.97 (dd, *J* = 19.3, 7.2 Hz, 2H), 2.91-2.75 (m, 2H), 2.09 (t, *J* = 14.0 Hz, 1H), 1.88 (t, *J* = 13.2 Hz, 1H).**^{13}C NMR (75 MHz, $CDCl_3$)** δ 155.65, 140.52, 129.97, 120.11, 115.60, 114.74, 51.10, 31.92, 24.98. **MS (EI, 70 ev) m/z:** 212, 166, 147, 138, 121, 105.

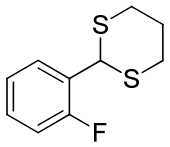
2-(1,3-dithian-2-yl)phenol (3q) [15]



3q

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 6.90 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.75 (td, *J* = 7.7, 1.6 Hz, 1H), 6.51 - 6.37 (m, 2H), 5.82 (s, 1H), 5.02 (s, 1H), 2.67-2.51 (m, 2H), 2.41-2.46 (m, 2H), 1.76-1.65 (m, 1H), 1.55-1.41 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 153.93, 129.86, 128.98, 123.70, 120.67, 116.94, 77.41, 76.99, 76.57, 46.73, 31.49, 24.68. **MS (EI, 70 ev) m/z:** 212, 147, 138, 119, 105.

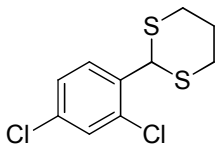
2-(2-fluorophenyl)-1,3-dithiane (3r) [21]



3r

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.62-7.56 (m, 1H), 7.24-7.19 (m, 1H), 7.13-7.08 (m, 1H), 7.03-6.97 (m, 1H), 5.52 (s, 1H), 3.14-2.94 (m, 2H), 2.87-2.80 (m, 2H), 2.15-2.03 (m, 1H), 1.97-1.79 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.22, 156.94, 129.69, 126.11, 124.41, 115.35, 42.79, 31.88, 24.78. **MS (EI, 70 ev) m/z:** 214, 167, 140, 139, 119, 105.

2-(2,4-dichlorophenyl)-1,3-dithiane (3s) [17]

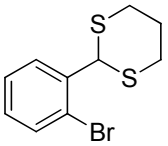


3s

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.55-7.50 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.36-7.24 (m,

1H), 7.16 (d, J = 8.4 Hz, 1H), 5.47 (s, 1H), 2.99 (t, J = 13.4 Hz, 2H), 2.79 (t, J = 14.4 Hz, 2H), 2.05 (t, J = 12.3 Hz, 1H), 1.82 (t, J = 12.5 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 135.11, 134.30, 132.99, 130.41, 129.17, 127.61, 46.70, 31.98, 24.81. **MS (EI, 70 ev) m/z:** 265, 129, 112, 105.

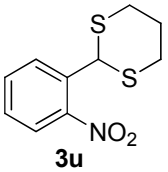
2-(2-bromophenyl)-1,3-dithiane (3t) [22]



3t

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.82-7.68 (m, 1H), 7.66-7.54 (m, 1H), 7.43-7.28 (m, 1H), 7.14-7.17 (m, 1H), 5.66 (s, 1H), 3.23-3.08 (m, 2H), 2.95 (t, J = 13.6 Hz, 2H), 2.28-2.13 (m, 1H), 2.05-1.87 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 138.03, 132.67, 129.54, 127.88, 122.76, 50.47, 32.04, 24.88. **MS (EI, 70 ev) m/z:** 276, 274, 202, 200, 149, 129, 121, 105.

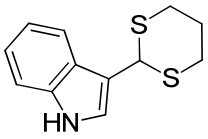
2-(2-nitrophenyl)-1,3-dithiane (3u) [23]



3u

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.98-7.93 (m, 2H), 7.69 (d, J = 7.5 Hz, 1H), 7.52 (d, J = 7.7 Hz, 1H), 5.96 (s, 1H), 3.19 (t, J = 13.0 Hz, 2H), 2.99 (t, J = 14.6 Hz, 2H), 2.26 (t, J = 14.2 Hz, 1H), 2.02 (t, J = 12.4 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 147.43, 133.22, 133.13, 130.44, 128.87, 124.44, 45.65, 31.97, 24.73. **MS (EI, 70 ev) m/z:** 241, 224, 166, 147, 135, 106.

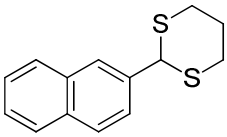
3-(1,3-dithian-2-yl)-1H-indole (3v) [24]



3v

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.57 (s, 1H), 7.45 (d, J = 4.9 Hz, 1H), 6.72 (d, J = 9.6 Hz, 4H), 5.11 (s, 1H), 2.59 (t, J = 12.5 Hz, 2H), 2.41 (t, J = 14.1 Hz, 2H), 1.64 (t, J = 14.0 Hz, 1H), 1.46 (t, J = 12.3 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 135.69, 125.24, 122.89, 122.24, 119.56, 119.39, 113.82, 111.36, 42.70, 31.95, 25.19. **MS (EI, 70 ev) m/z:** 235, 161, 120, 105.

2-(naphthalen-2-yl)-1,3-dithiane (3w) [25]

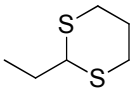


3w

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.78 (s, 1H), 7.70-7.51 (m, 3H), 7.48-7.35 (m, 1H),

7.33-7.17 (m, 2H), 5.12 (s, 1H), 2.82 (t, J = 13.4 Hz, 2H), 2.68 (t, J = 14.3 Hz, 2H), 1.91-1.86 (m, 1H), 1.82-1.63 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 136.31, 133.06, 132.99, 128.29, 127.82, 127.45, 126.60, 126.11, 125.49, 51.31, 31.87, 24.89. **MS (EI, 70 ev) m/z:** 246, 203, 185, 172, 167, 128, 115, 105.

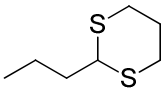
2-ethyl-1,3-dithiane (5a) [26]



5a

Colorless oil. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 3.98 (t, J = 6.8 Hz, 1H), 2.87-2.82 (m, 4H), 2.15-2.05 (m, 1H), 1.93-1.76 (*m*, 4.3 Hz, 3H), 1.07 (t, J = 10Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 49.30, 30.37, 29.66, 28.77, 26.02, 11.47. **MS (EI, 70 ev) m/z:** 148, 133, 97.

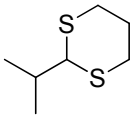
2-propyl-1,3-dithiane (5b) [26]



5b

Colorless oil. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 4.02 (t, J = 7.1 Hz, 1H), 2.91-2.72 (m, 4H), 2.14-2.00 (m, 1H), 1.86-1.78 (m, 1H), 1.77-1.64 (m, 2H), 1.49 (dt, J = 14.6, 7.4 Hz, 2H), 0.89 (t, J = 9Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 47.19, 37.36, 30.33, 25.94, 19.74, 13.56. **MS (EI, 70 ev) m/z:**162, 111, 97.

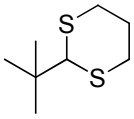
2-isopropyl-1,3-dithiane (5c) [27]



5c

Colorless oil. **^{1}H NMR (300 MHz, $CDCl_3$) δ** 4.01 (d, J = 5.1 Hz, 1H), 2.84 (dd, J = 7.9, 2.4 Hz, 4H), 2.13 – 1.91 (m, 2H), 1.82 (d, J = 4.5 Hz, 1H), 1.07 (d, J = 6.8 Hz, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 56.22, 33.46, 30.71, 29.59, 26.19, 19.87. **MS (EI, 70 ev) m/z:**162, 147, 111, 97.

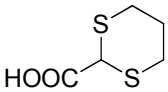
2-(tert-butyl)-1,3-dithiane (5d) [16]



5d

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 3.92 (s, 1H), 2.95-2.64 (m, 4H), 2.11-1.92 (m, 1H), 1.82-1.63 (m, 1H), 1.22 – 0.90 (s, 9H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 35.58, 31.12, 27.75, 25.85. **MS (EI, 70 ev) m/z:** 177, 149, 123, 111, 97.

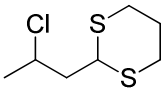
1,3-dithiane-2-carboxylic acid (5e) [28]



5e

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 11.99 (s, 1H), 4.31 (s, 1H), 3.61-3.50 (m, 2H), 2.81-2.68 (m, 2H), 2.39-2.12 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 176.29, 38.99, 25.43, 24.55. **MS (EI, 70 ev) m/z:** 164, 119, 99, 85.

2-(2-chloropropyl)-1,3-dithiane (5f) [18]



5f

White solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 4.35-4.26 (m, 1H), 4.25-4.15 (m, 1H), 2.97-2.75 (m, 4H), 2.17-1.99 (m, 3H), 1.98-1.79 (m, 1H), 1.55 (d, *J* = 15Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 54.58, 45.33, 44.10, 29.97, 29.51, 25.77, 25.20. **MS (EI, 70 ev) m/z:** 196, 123, 111, 97.

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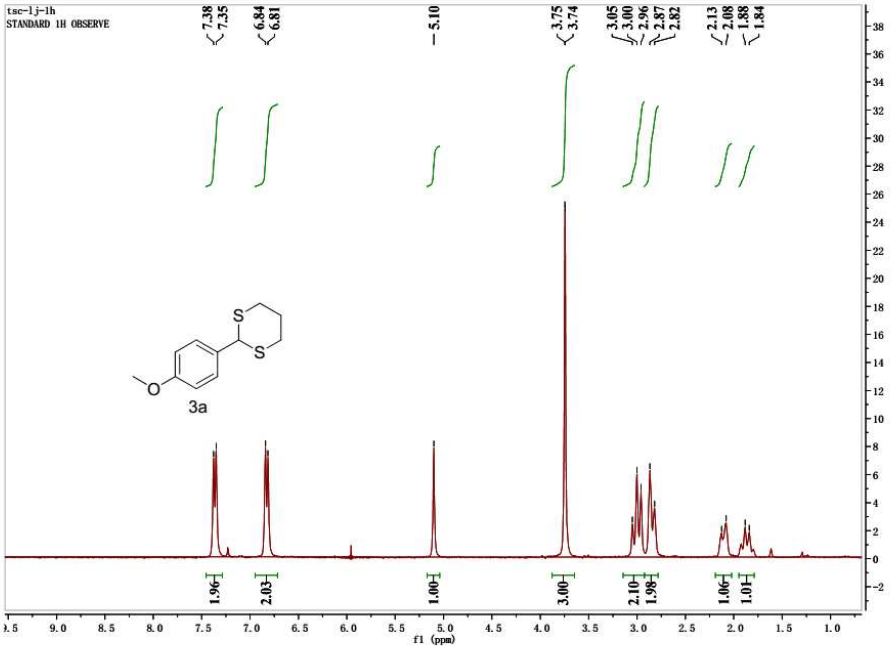
6. Spectra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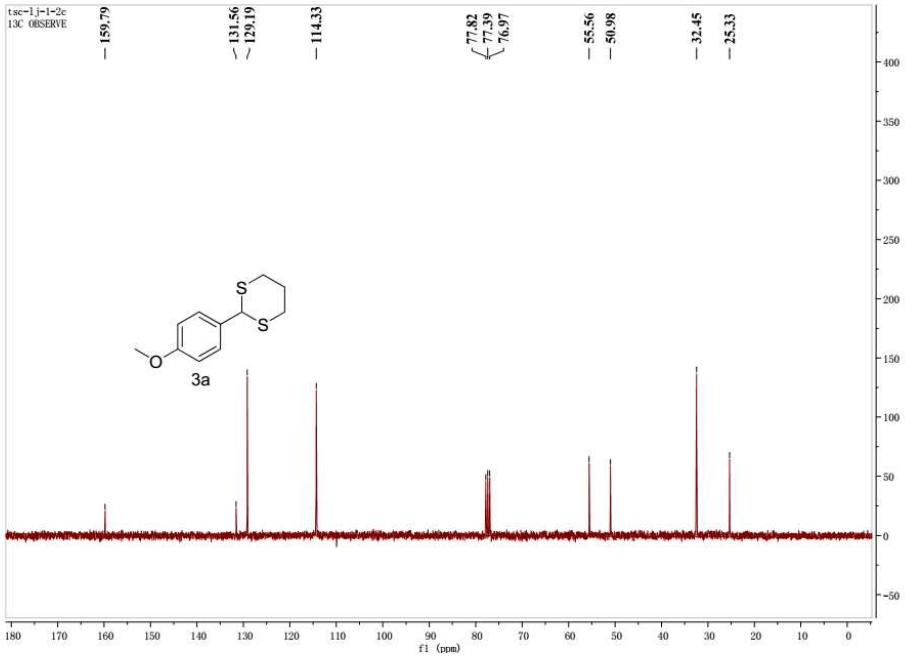
7.35
6.84
6.81
5.10
3.75
3.74
3.05
3.00
2.96
2.87
2.82
2.13
2.08
1.88
1.84
3a
1.96
2.03
1.00
3.00
2.10
1.98
1.06
1.01
f1 (ppm)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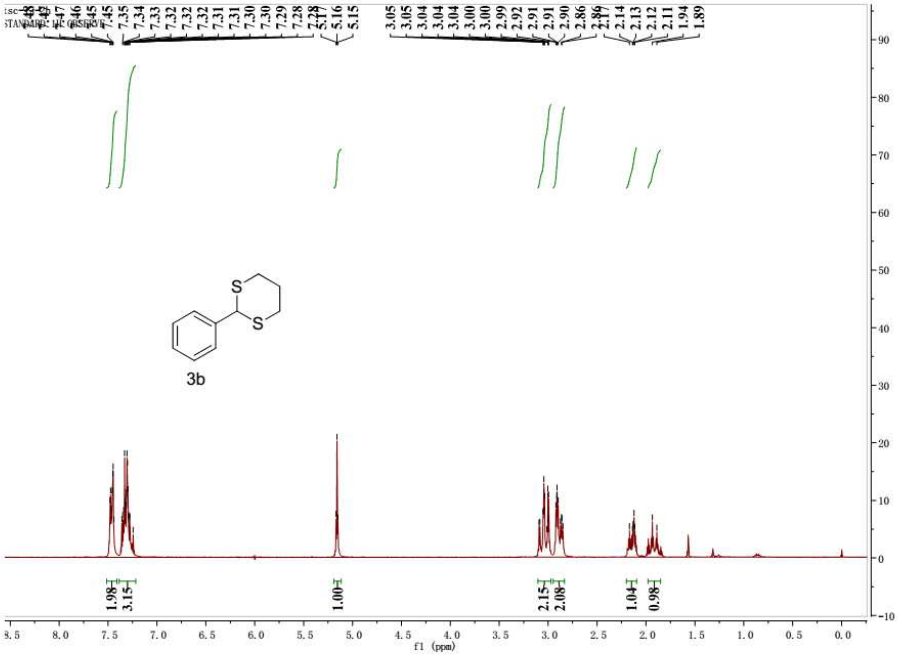
13C OBSERVE
159.79
131.56
129.19
114.33
77.82
77.39
76.97
55.56
50.98
32.45
25.33
3a
f1 (ppm)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3.15
1.00
2.15
2.09
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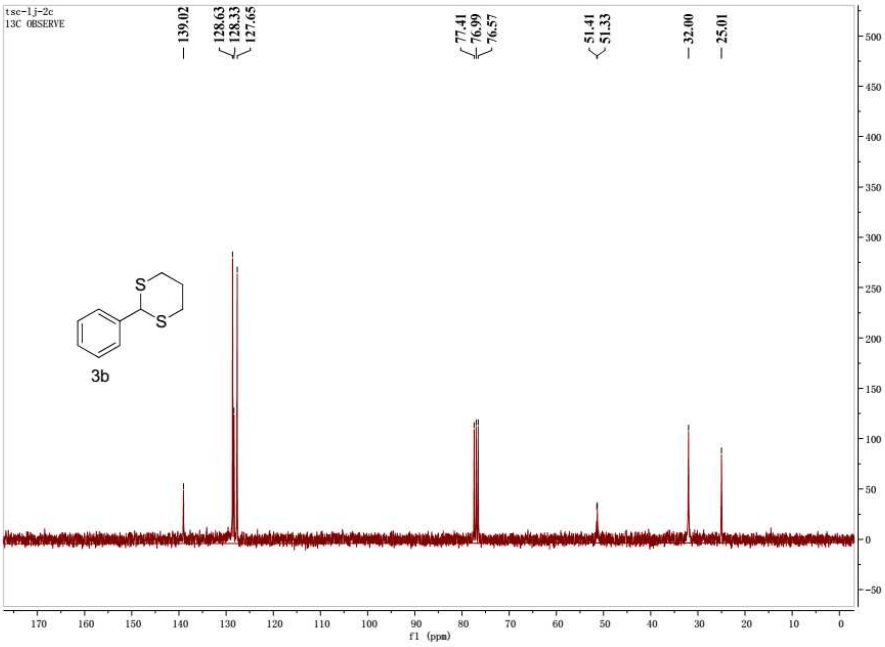
0.98
f1 (ppm)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128.63
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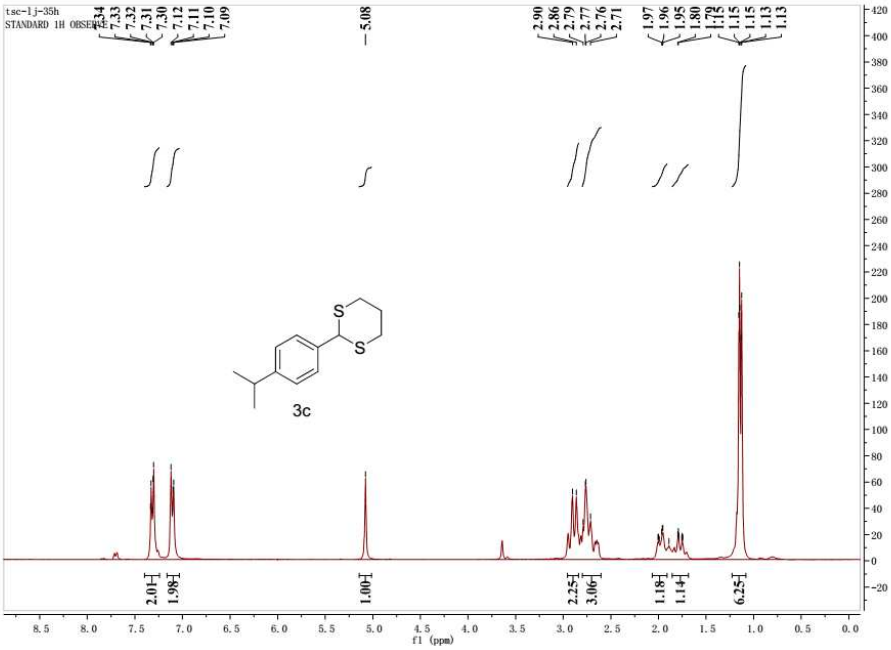
127.65
77.41
76.99
76.57
51.41
51.33
32.00
25.01
3b
f1 (ppm)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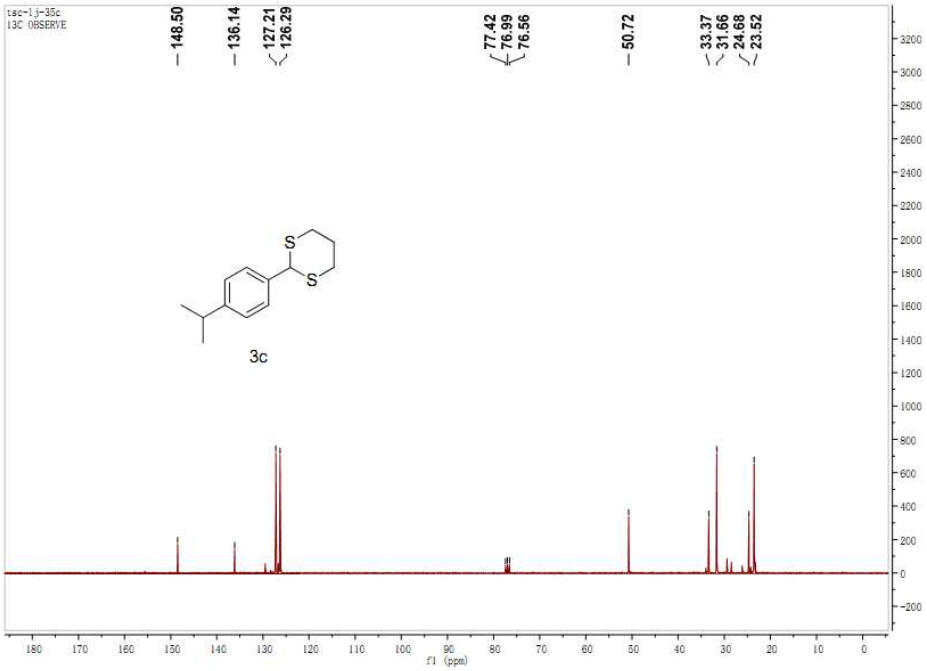
7.33
7.32
7.31
7.30
7.12
7.11
7.10
7.09
5.08
2.90
2.86
2.79
2.77
2.76
2.71
1.97
1.96
1.95
1.80
1.79
1.15
1.15
1.13
1.13
S
S
3c
2.01
1.98
1.00
2.25
3.06
1.18
1.14
6.25
f1 (ppm)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136.14
127.21
126.29
77.42
76.99
76.56
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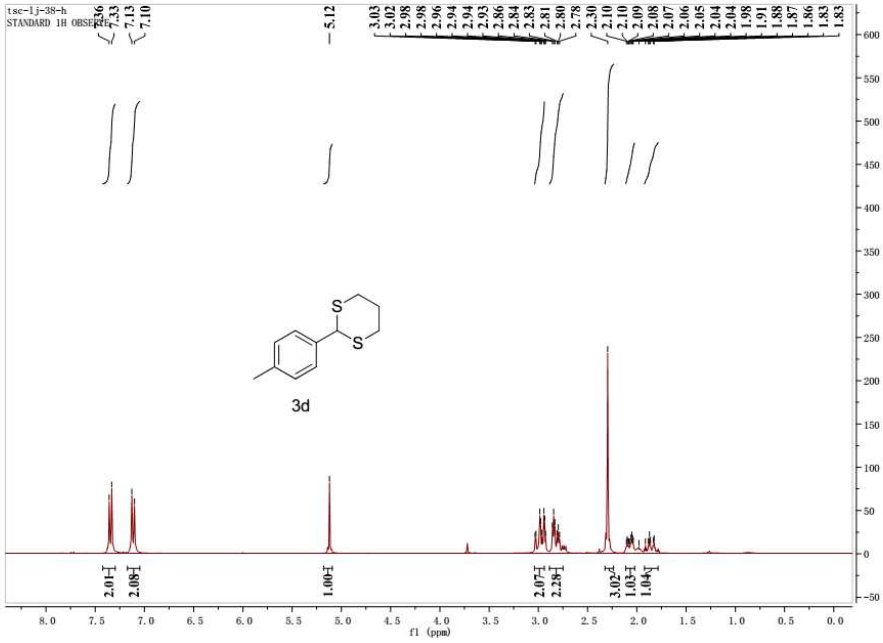
33.37
31.66
24.68
23.52
S
S
3c
f1 (ppm)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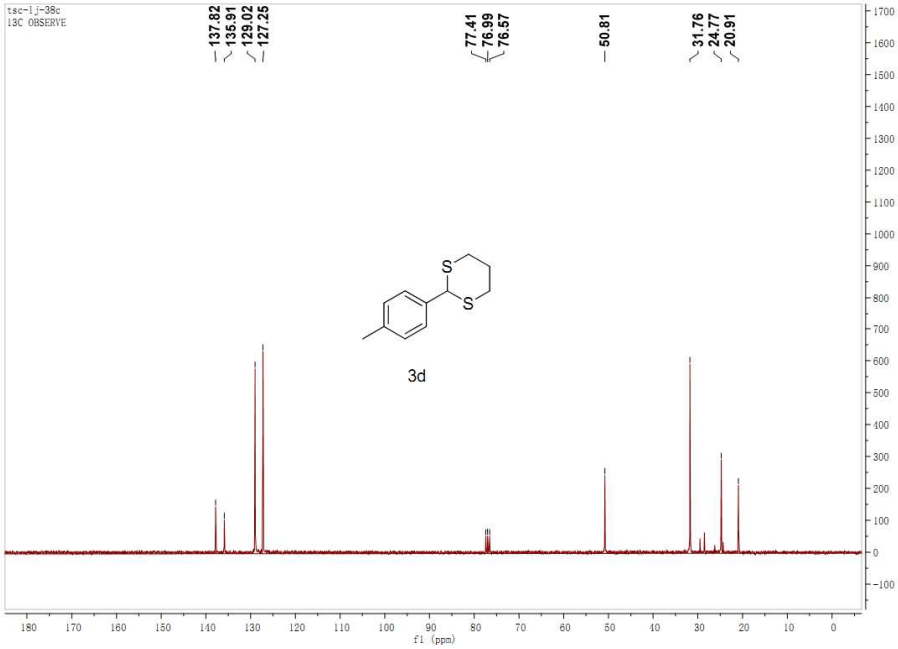
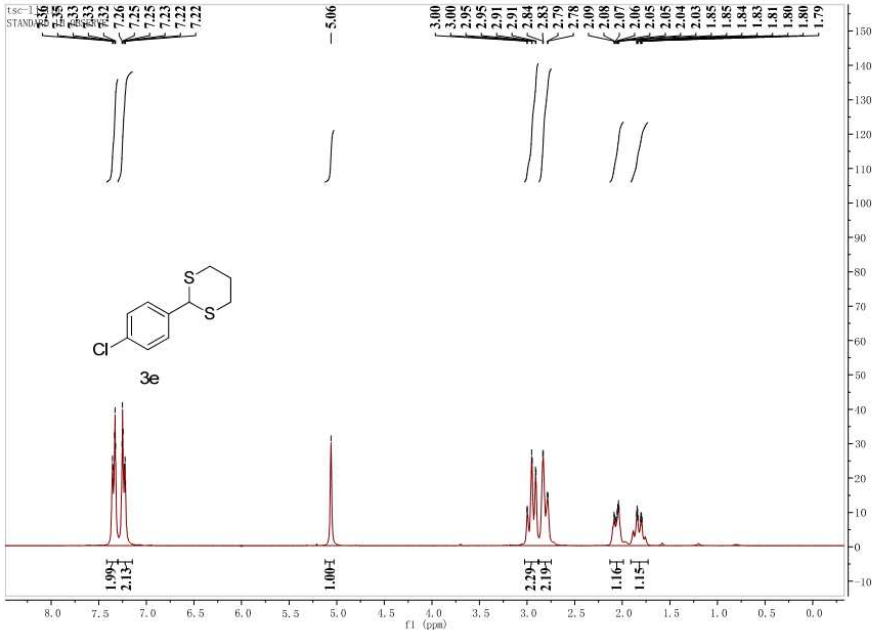
7.33
7.13
7.10
5.12
3.03
3.02
2.98
2.98
2.96
2.94
2.94
2.93
2.86
2.84
2.83
2.81
2.80
2.78
2.30
2.10
2.10
2.09
2.08
2.07
2.06
2.05
2.04
2.04
1.98
1.91
1.88
1.87
1.86
1.83
1.83
S
S
3d
2.01
2.08
1.00
2.07
2.29
3.02
1.03
1.04
f1 (ppm)


tsc-lj-38c
13C OBSERVE
137.82
135.91
129.02
127.25
77.41
76.99
76.57
50.81
31.76
24.77
20.91
S
S
3d
f1 (ppm)


tsc-1j
STANDARD
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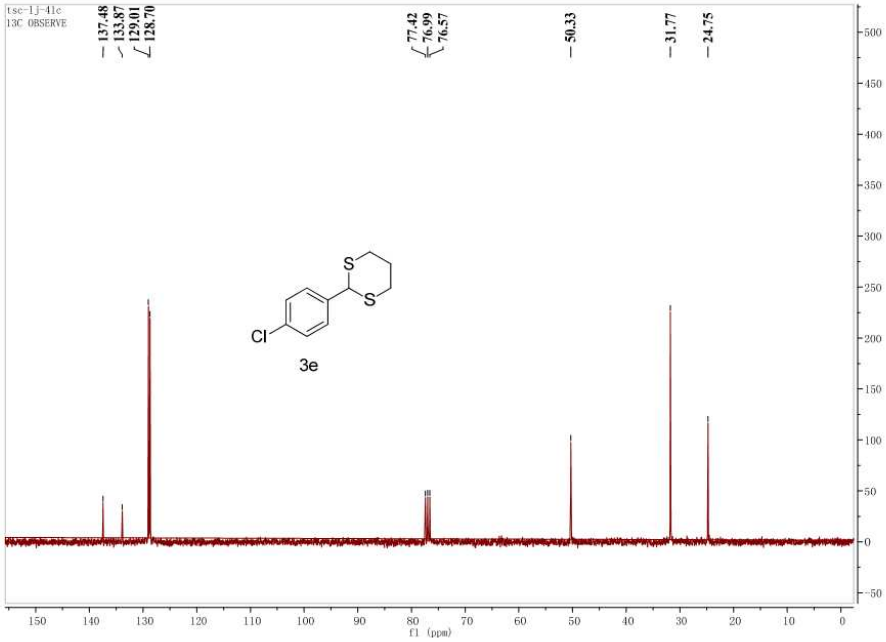
5.06
3.00 3.00 2.95 2.95 2.91 2.91 2.84 2.83 2.79 2.78
2.09 2.08 2.07 2.06 2.05 2.04 2.03 1.85 1.85 1.84 1.83 1.81 1.80 1.80 1.79
S
S
Cl
3e
1.99 2.13
1.00
2.29 2.19
1.16 1.15
f1 (ppm)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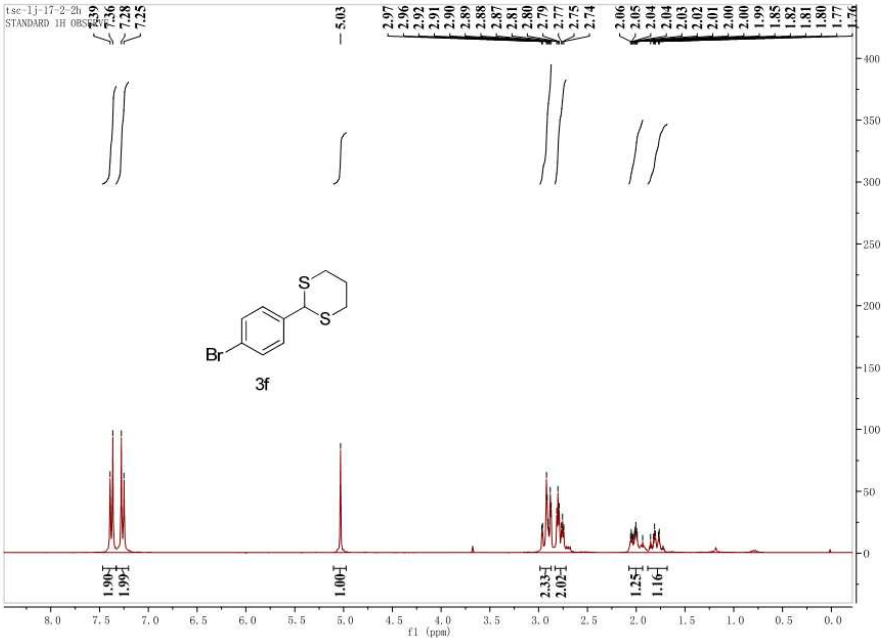
13C OBSERVE
137.48
133.87
129.01
128.70
77.42
76.99
76.57
50.33
31.77
24.75
S
S
Cl
3e
f1 (ppm)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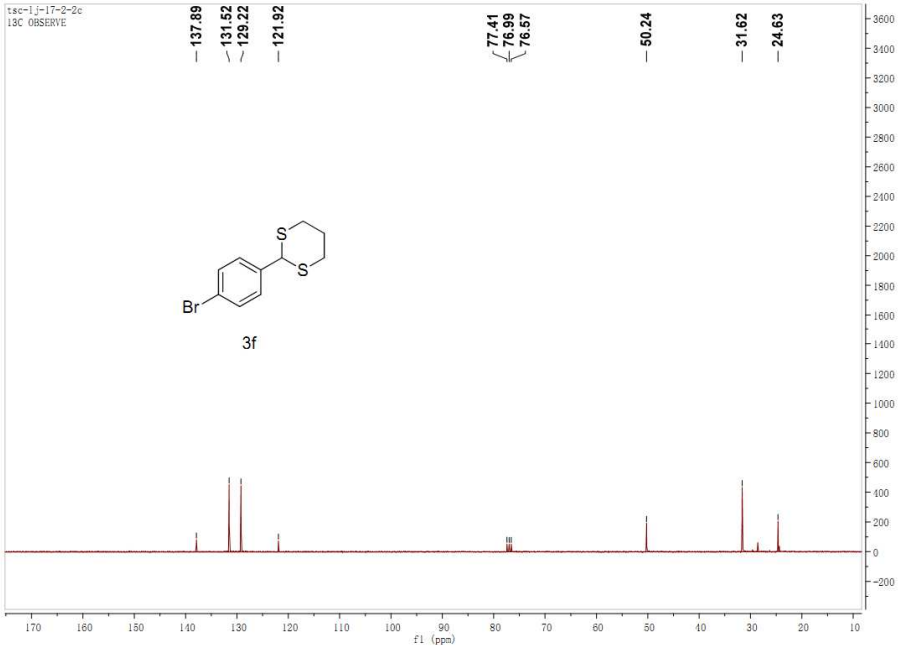
7.36
7.28
7.25
5.03
2.97
2.96
2.92
2.91
2.90
2.89
2.88
2.87
2.81
2.80
2.79
2.77
2.75
2.74
2.06
2.05
2.04
2.04
2.03
2.02
2.01
2.00
2.00
1.99
1.85
1.82
1.81
1.80
1.77
1.76
S
S
Br
3f
1.90
1.99
1.00
2.33
2.02
1.25
1.16
f1 (ppm)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13C OBSERVE
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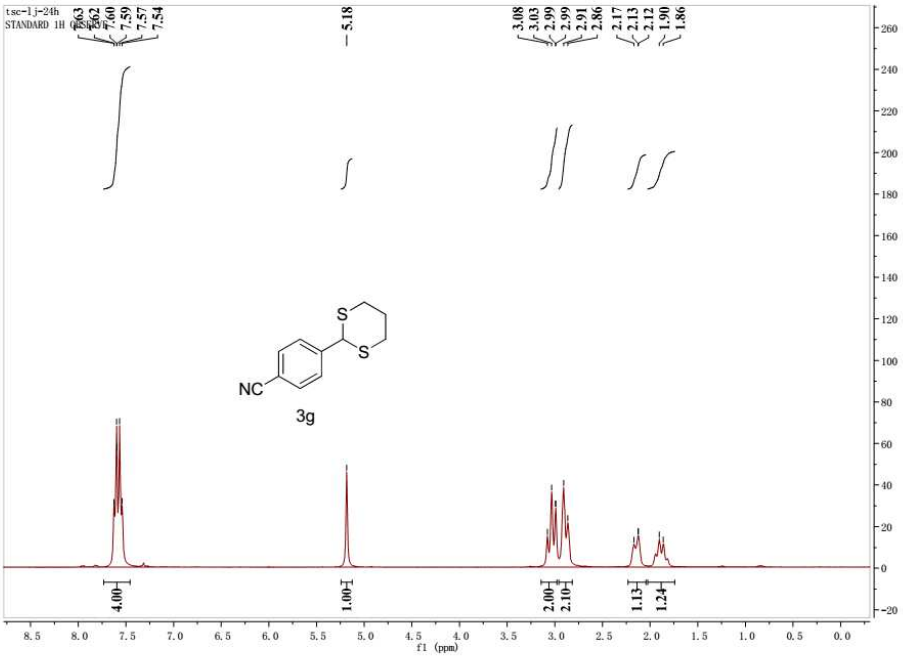
131.52
129.22
121.92
77.41
76.99
76.57
50.24
31.62
24.63
S
S
Br
3f
f1 (ppm)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STANDARD 1H
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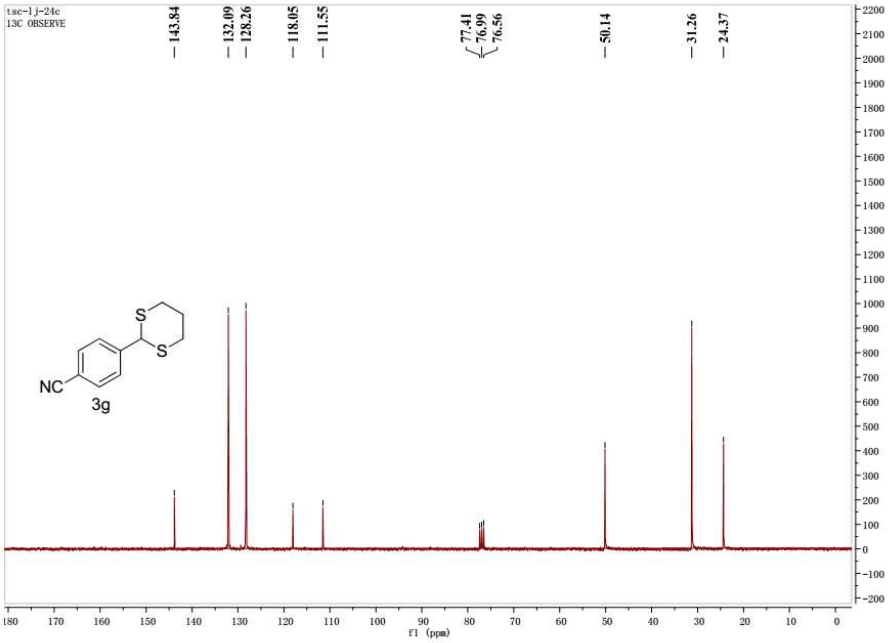
7.62
7.60
7.59
7.57
7.54
5.18
3.08
3.03
2.99
2.99
2.91
2.86
2.17
2.13
2.12
1.90
1.86
S
S
NC
3g
4.00
1.00
2.00
2.10
1.13
1.24
f1 (ppm)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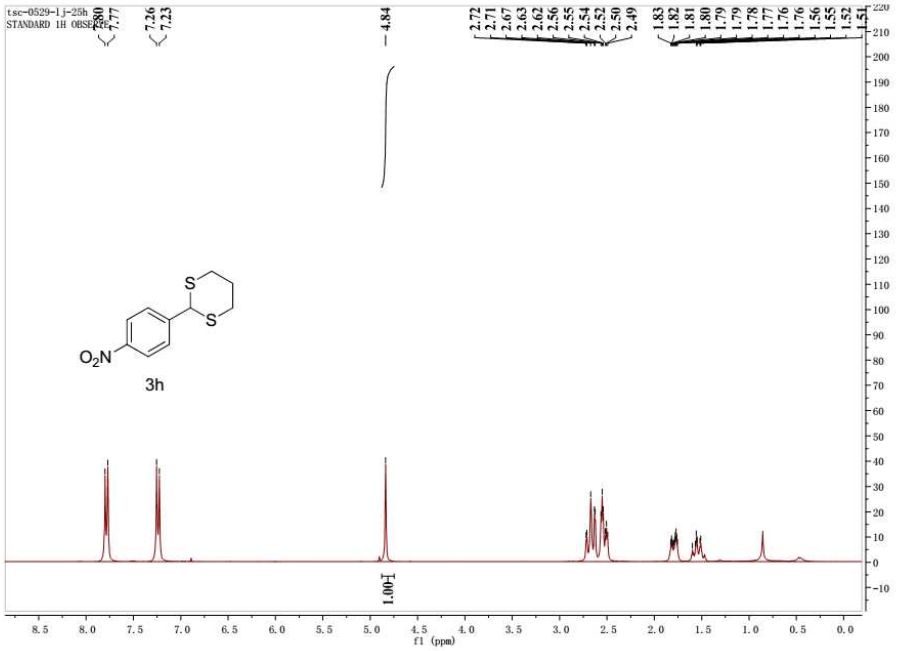
132.09
128.26
118.05
111.55
77.41
76.99
76.56
50.14
31.26
24.37
S
S
NC
3g
f1 (ppm)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STANDARD 1H OBSERVE
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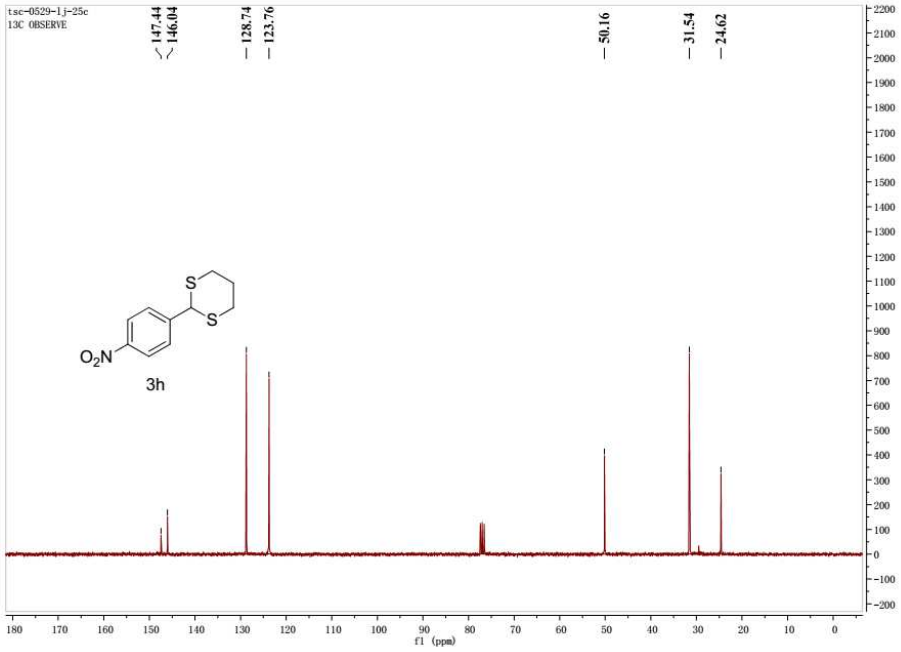
7.26
7.23
4.84
2.72
2.71
2.67
2.63
2.62
2.56
2.55
2.54
2.52
2.50
2.49
1.83
1.82
1.81
1.80
1.79
1.79
1.78
1.77
1.76
1.76
1.56
1.55
1.52
1.51
1.00
S
S
O_2N
3h
f1 (ppm)


tsc-0529-lj-25c
13C OBSERVE
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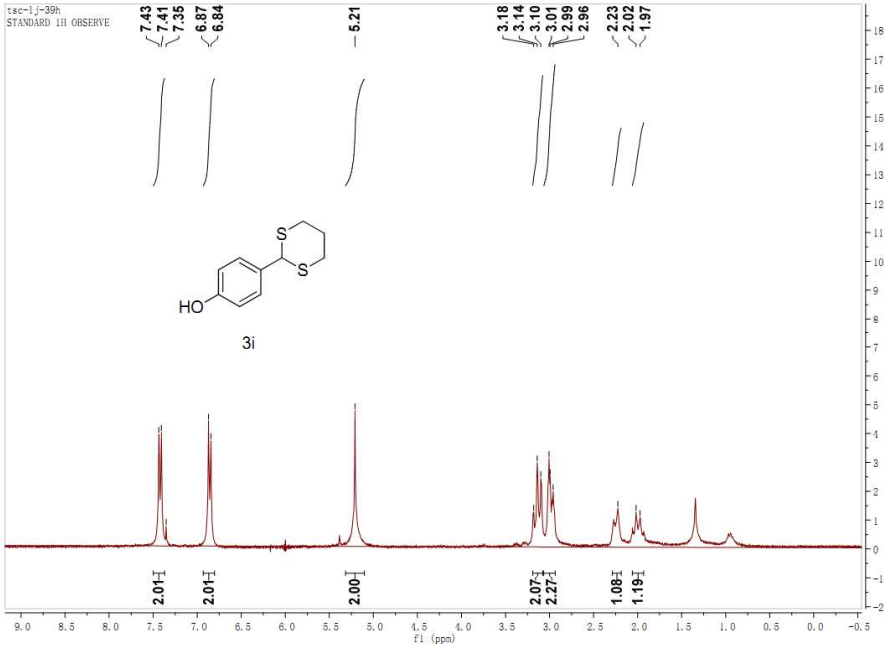
128.74
123.76
50.16
31.54
24.62
S
S
O_2N
3h
f1 (ppm)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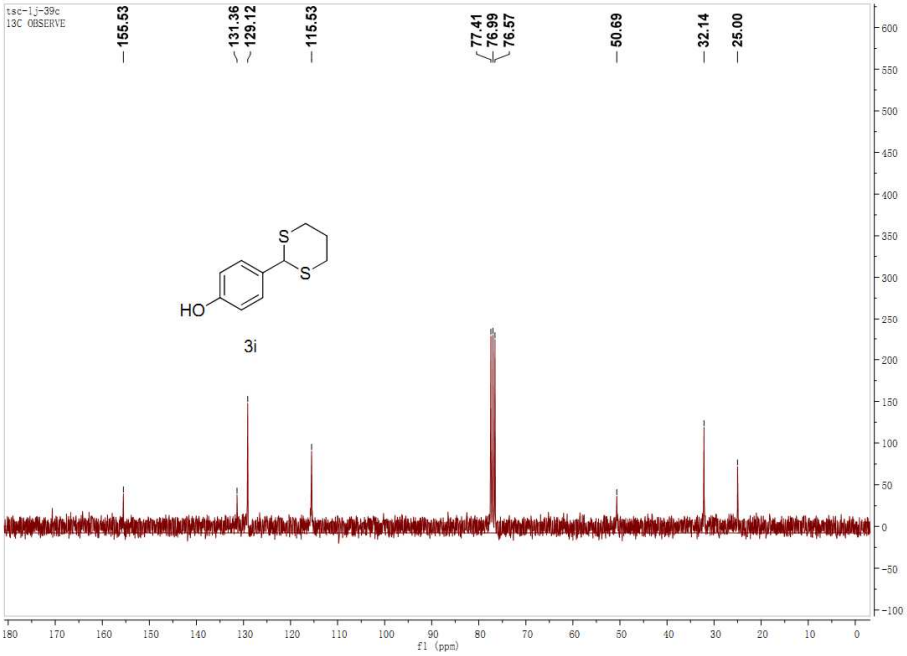
7.43
7.41
7.35
6.87
6.84
5.21
3.18
3.14
3.10
3.01
2.99
2.96
2.23
2.02
1.97
S
S
HO
3i
2.01
2.01
2.00
2.07
2.27
1.08
1.19
f1 (ppm)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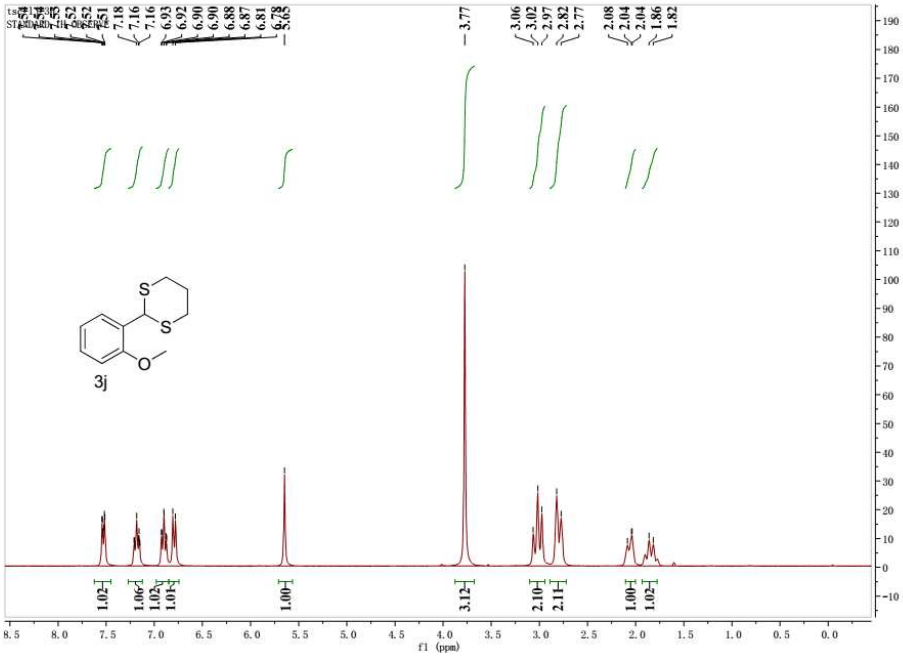
155.53
131.36
129.12
115.53
77.41
76.99
76.57
50.69
32.14
25.00
S
S
HO
3i
f1 (ppm)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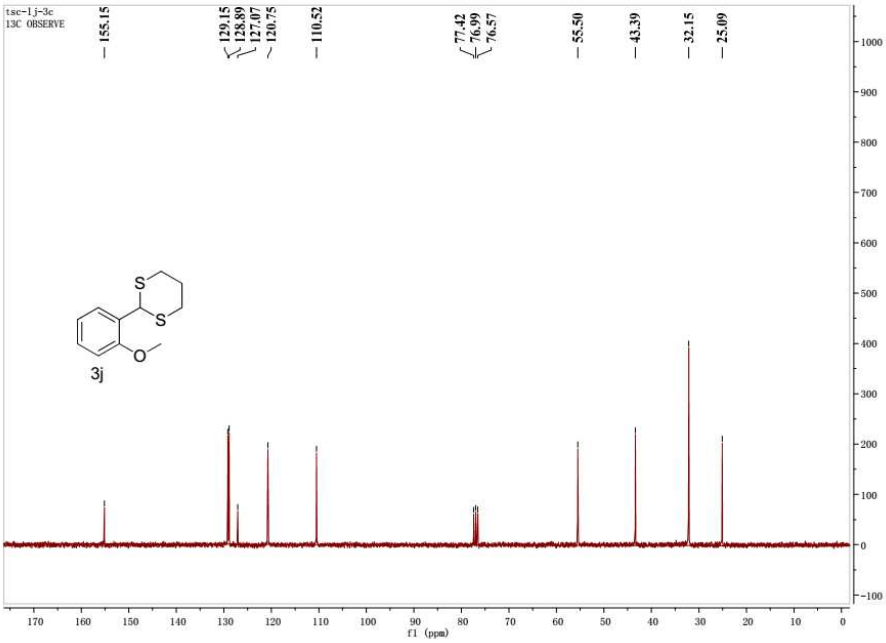
f1 (ppm)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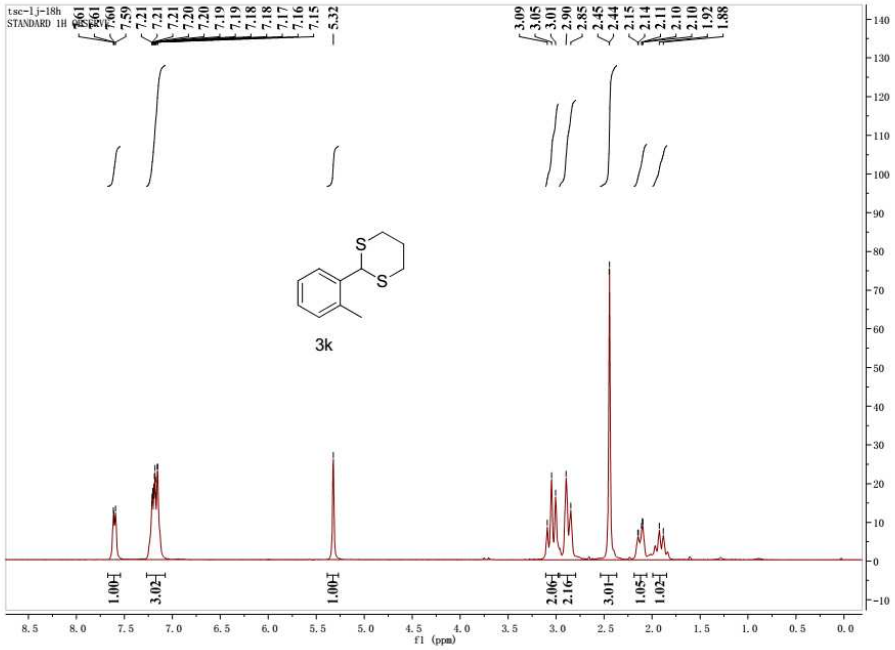
3j
f1 (ppm)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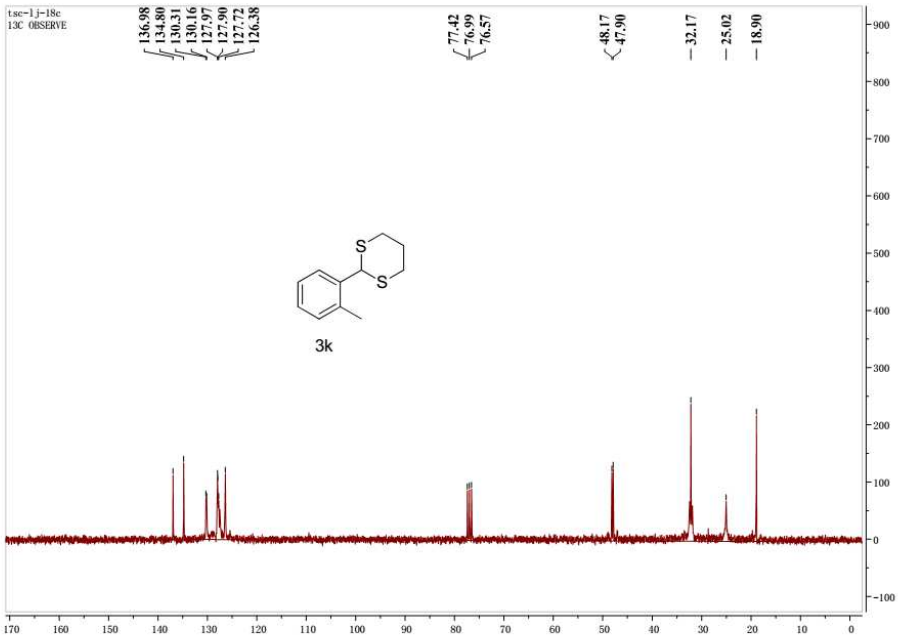
7.61
7.60
7.59
7.21
7.21
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
7.18
7.17
7.16
7.15
5.32
3.09
3.05
3.01
2.90
2.85
2.45
2.44
2.15
2.14
2.11
2.10
2.10
1.92
1.88
S
S
3k
1.00
3.02
1.00
2.06
2.16
3.01
1.05
1.02
f1 (ppm)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13C OBSERVE
136.98
134.80
130.31
130.16
127.97
127.90
127.72
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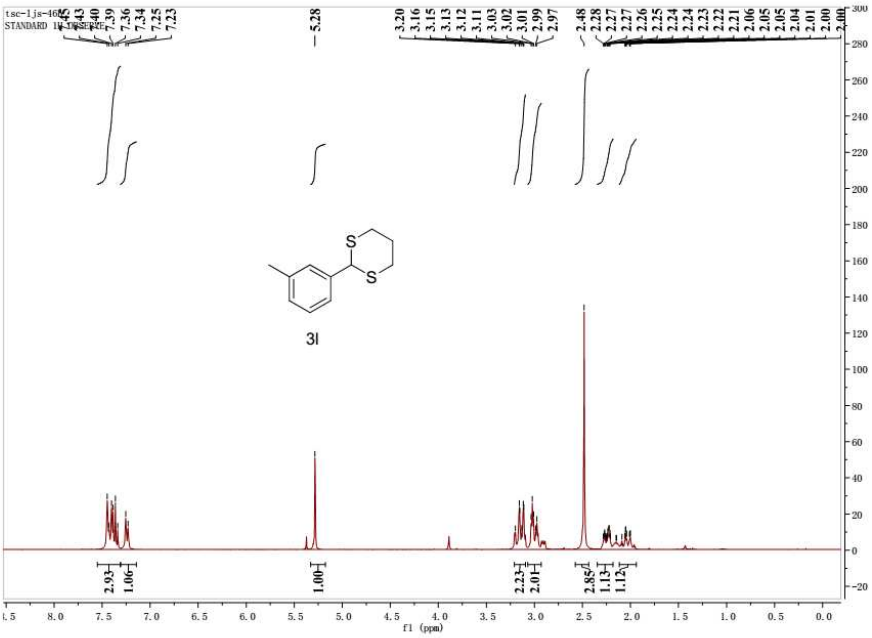
77.42
76.99
76.57
48.17
47.90
32.17
25.02
18.90
S
S
3k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3.20
3.16
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3.13
3.12
3.11
3.03
3.02
3.01
3.01
2.99
2.97
2.48
2.28
2.27
2.27
2.26
2.25
2.24
2.24
2.23
2.22
2.21
2.06
2.05
2.05
2.04
2.01
2.00
2.00
7.45
7.43
7.40
7.39
7.36
7.34
7.25
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S
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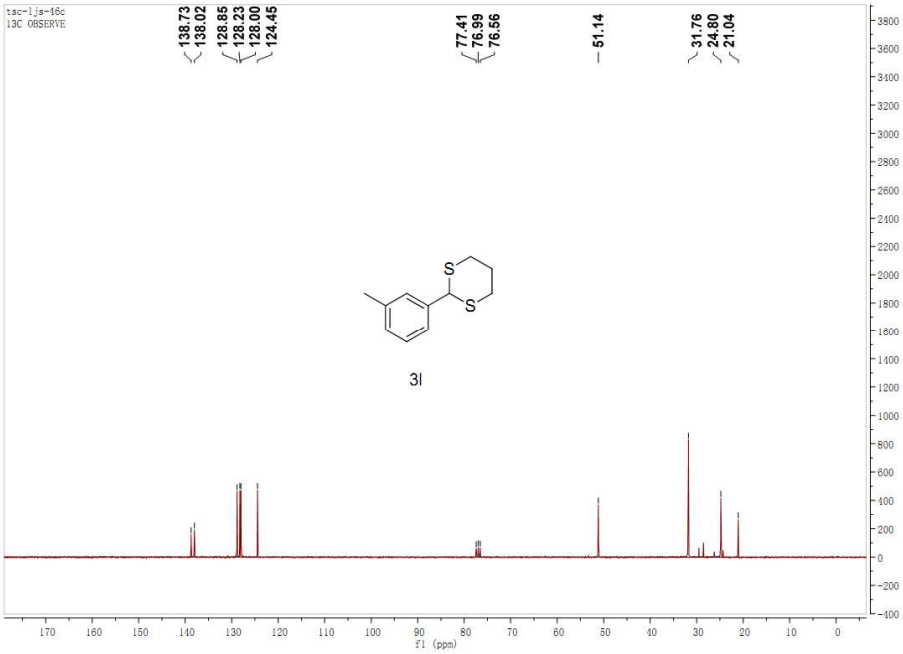
1.13
1.12
f1 (ppm)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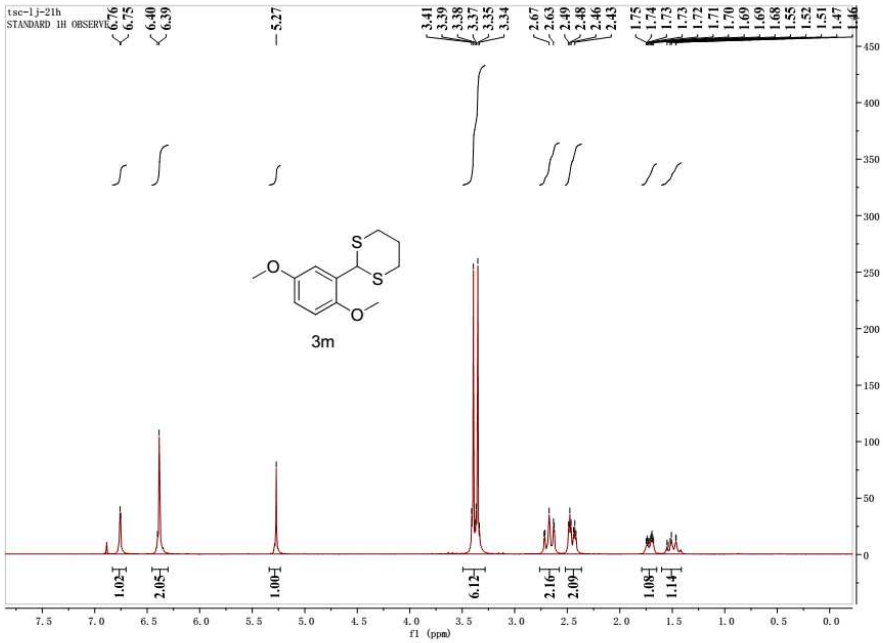
138.02
128.85
128.23
128.00
124.45
77.41
76.99
76.56
51.14
31.76
24.80
21.04
S
S
3l
f1 (ppm)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6.75
6.40
6.39
5.27
3.41
3.39
3.38
3.37
3.35
3.34
2.67
2.63
2.49
2.48
2.46
2.43
1.75
1.74
1.73
1.73
1.72
1.71
1.70
1.69
1.69
1.68
1.55
1.52
1.51
1.47
1.46
S
S
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O
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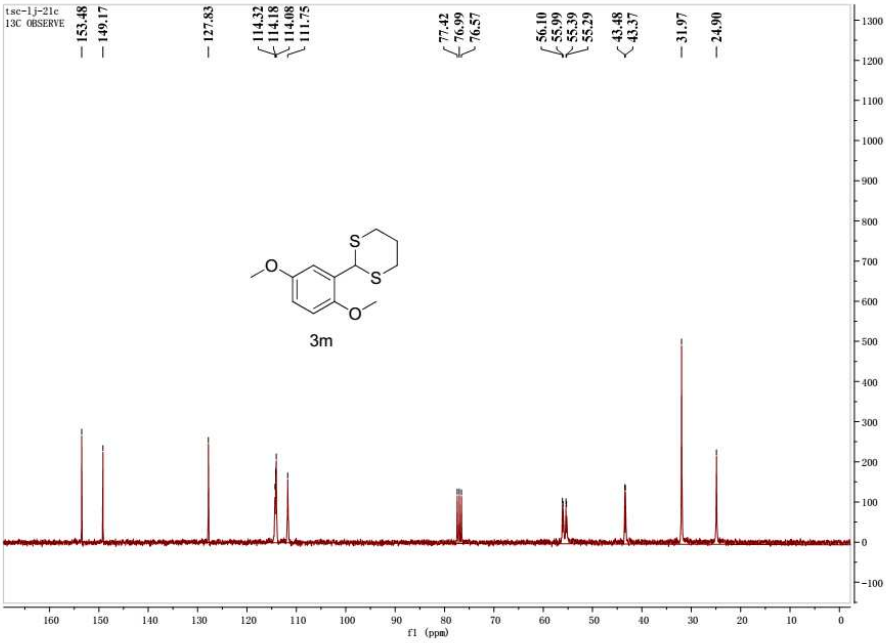
1.02
2.05
1.00
6.12
2.16
2.09
1.08
1.14
f1 (ppm)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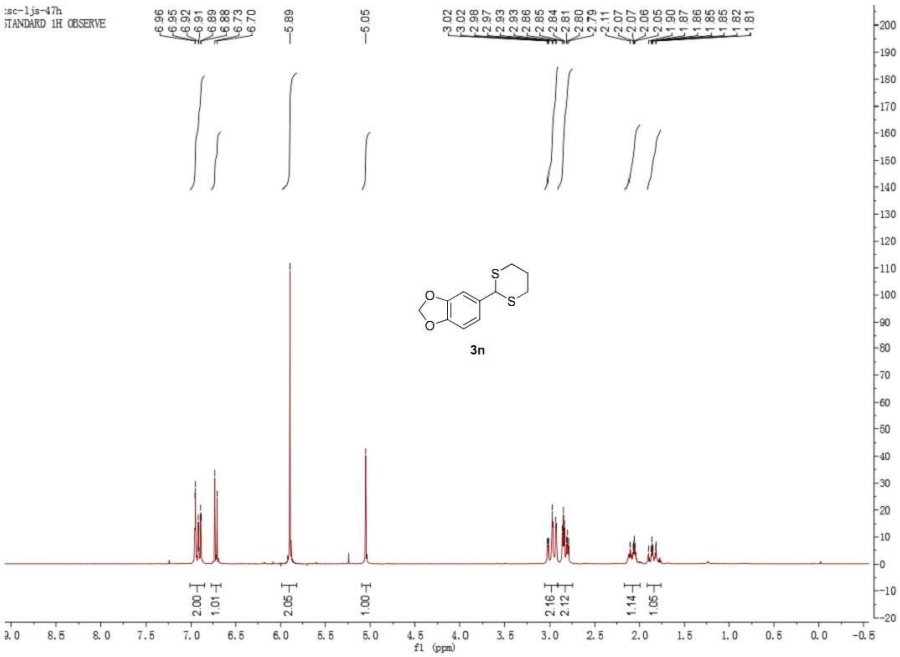
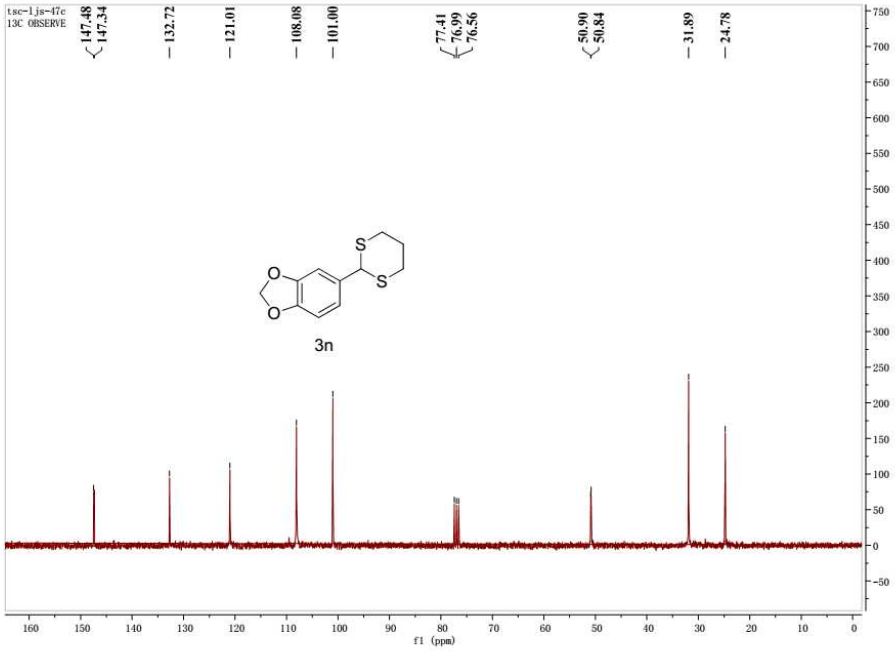
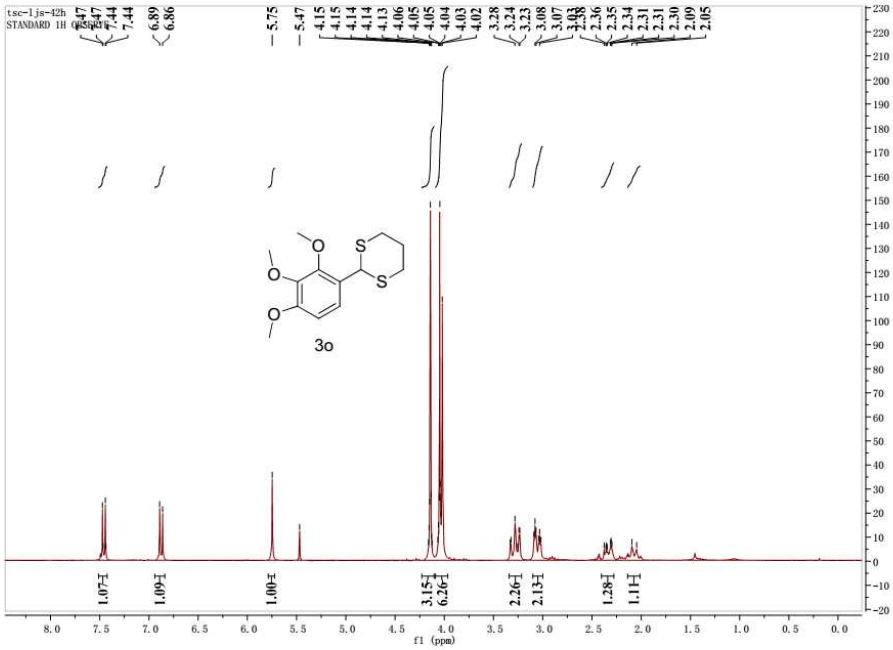
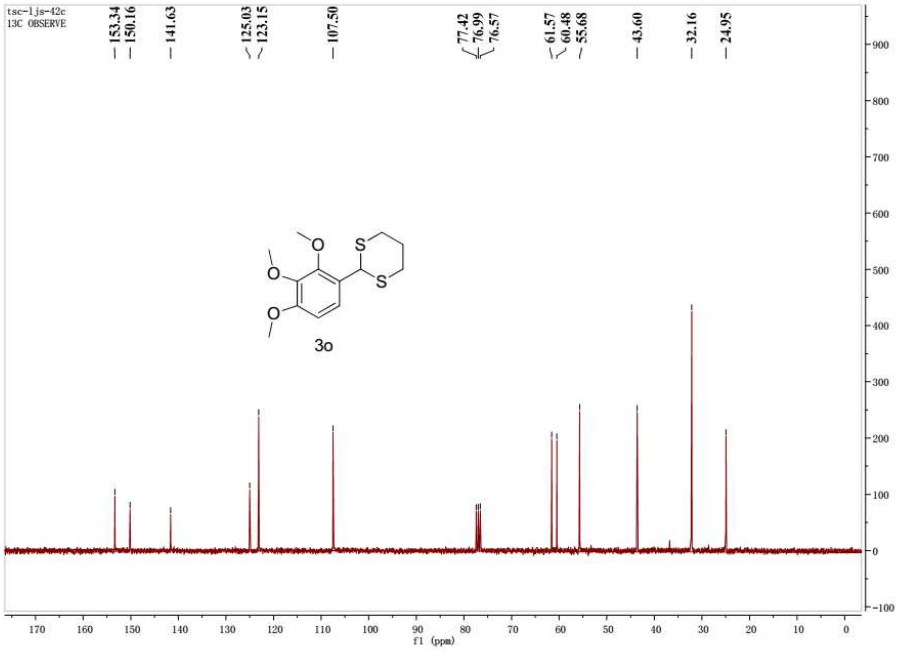
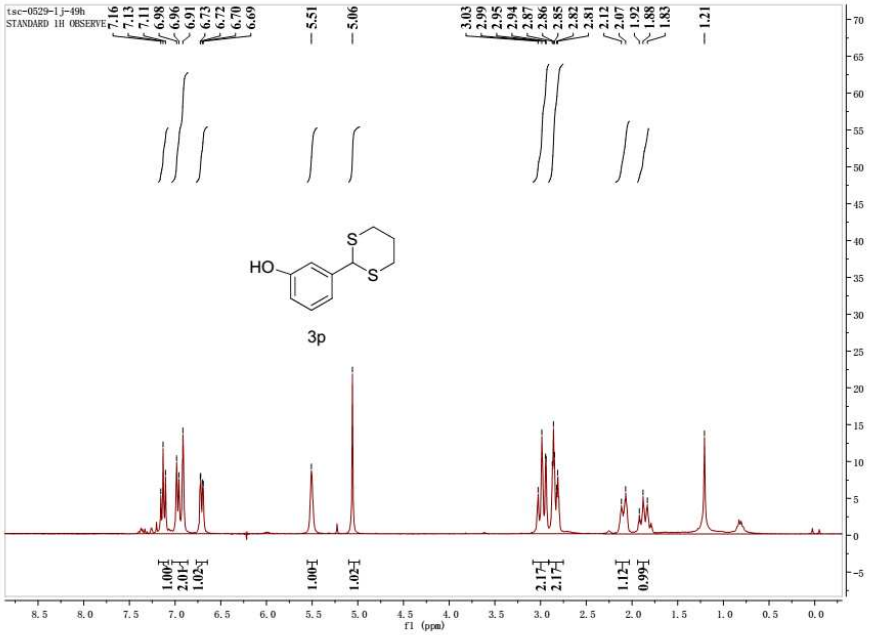
13C OBSERVE
153.48
149.17
127.83
114.32
114.18
114.08
111.75
77.42
76.99
76.57
56.10
55.99
55.39
55.29
43.48
43.37
31.97
24.90
S
S
O
O
3m
f1 (ppm)

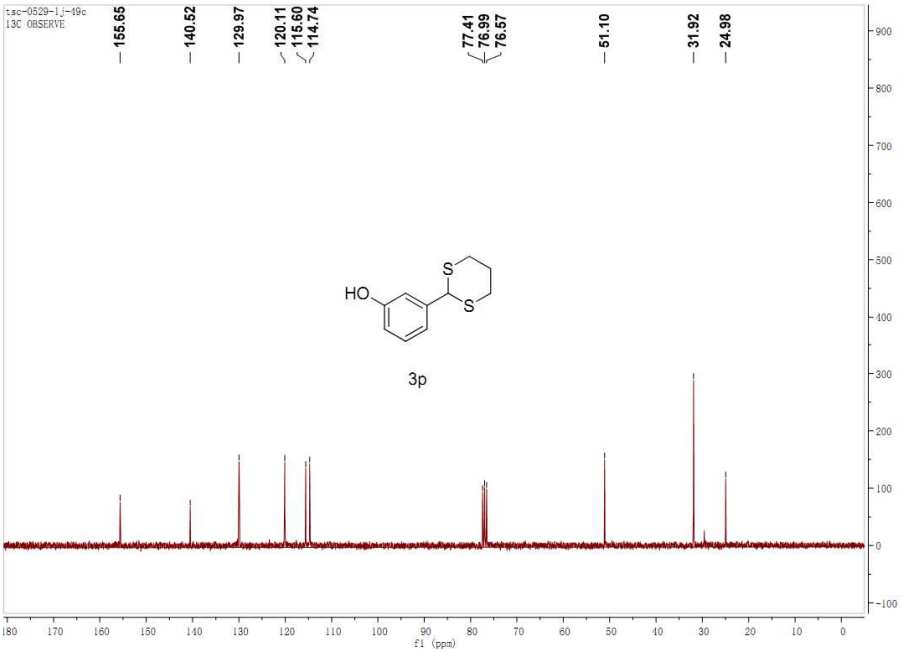

tsc-ljs-47h
STANDARD 1H OBSERVE
6.96
6.95
6.92
6.91
6.89
6.88
6.73
6.70
5.89
5.05
3.02
3.02
2.99
2.97
2.93
2.93
2.86
2.85
2.84
2.81
2.80
2.79
2.11
2.07
2.07
2.06
2.05
1.90
1.87
1.86
1.85
1.85
1.82
1.81
2.00
1.01
2.05
1.00
2.16
2.12
1.14
1.05
f1 (ppm)
3n

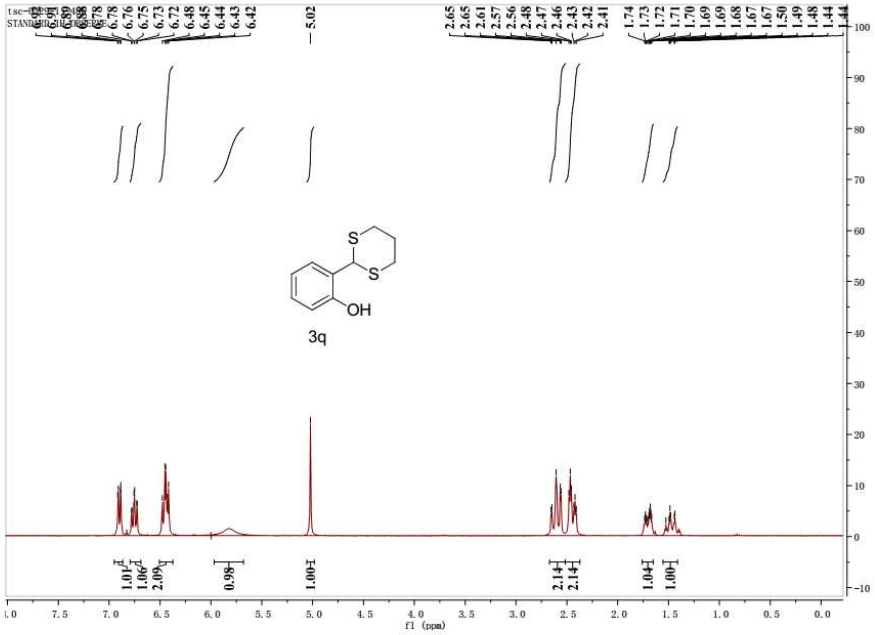

tsc-ljs-47c
13C OBSERVE
147.48
147.34
132.72
121.01
108.08
101.00
77.41
76.99
76.56
50.90
50.84
31.89
24.78
f1 (ppm)
3n

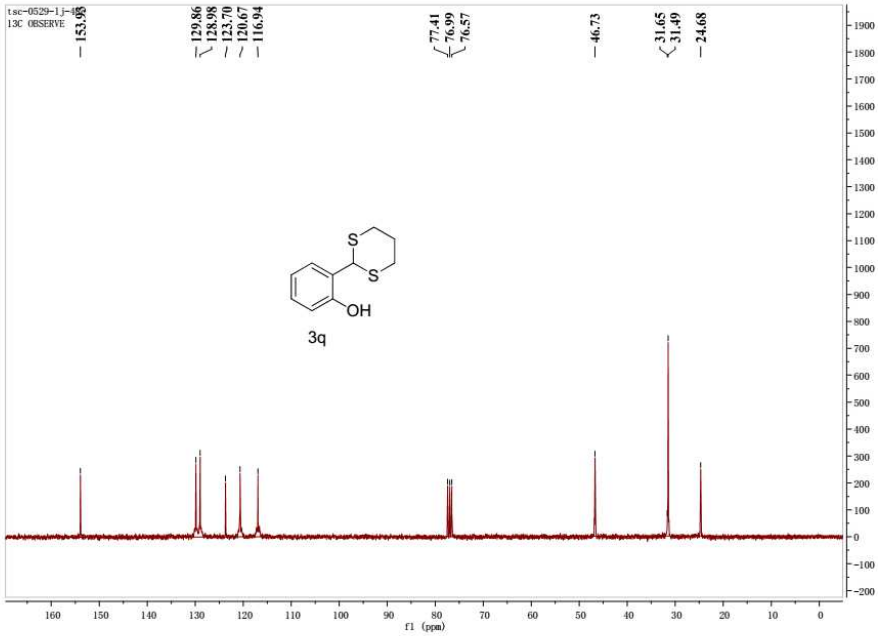

tsc-ljs-42h
STANDARD 1H OBSERVE
7.47
7.47
7.44
7.44
6.89
6.86
5.75
5.47
4.15
4.15
4.14
4.14
4.13
4.06
4.05
4.05
4.04
4.03
4.02
3.28
3.24
3.23
3.08
3.07
2.36
2.35
2.34
2.31
2.31
2.30
2.09
2.05
1.07
1.09
1.00
3.15
6.26
2.26
2.13
1.28
1.11
f1 (ppm)
3o

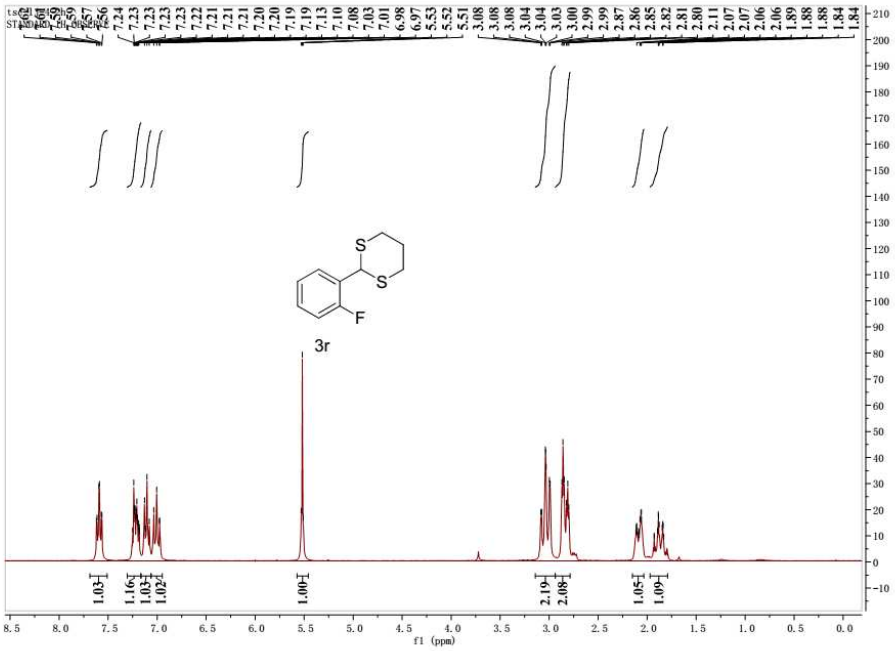

tsc-ljs-42c
13C OBSERVE
153.34
150.16
141.63
125.03
123.15
107.50
77.42
76.99
76.57
61.57
60.48
55.68
43.60
32.16
24.95
f1 (ppm)
3o

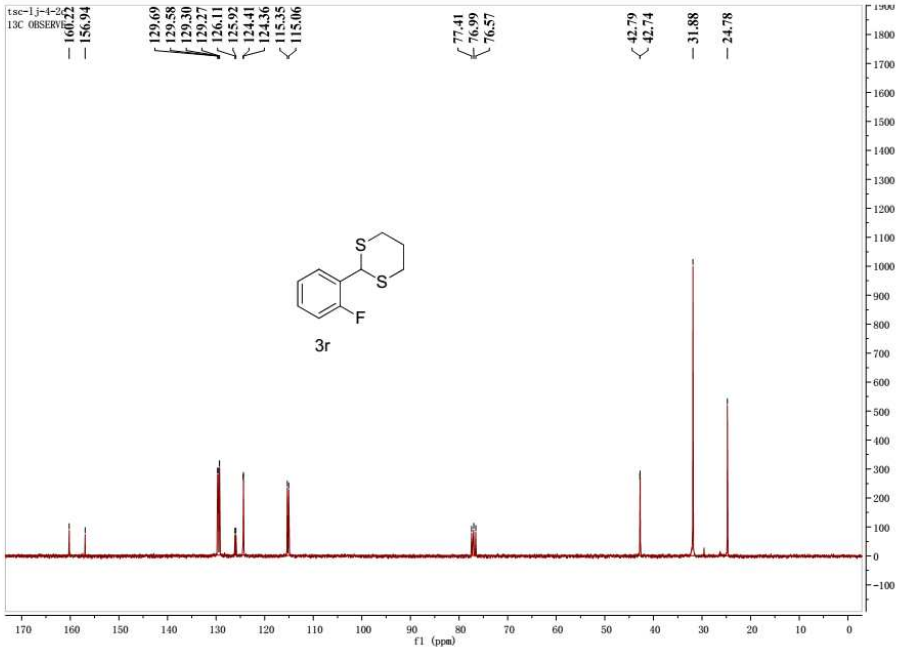

tsc-0529-lj-49h
STANDARD 1H OBSERVE
7.16
7.13
7.11
6.98
6.96
6.91
6.73
6.72
6.70
6.69
5.51
5.06
3.03
2.99
2.95
2.94
2.87
2.86
2.85
2.82
2.81
2.12
2.07
1.92
1.88
1.83
1.21
HO
S
S
3p
1.00
2.01
1.02
1.00
1.02
2.17
2.17
1.12
0.99
f1 (ppm)

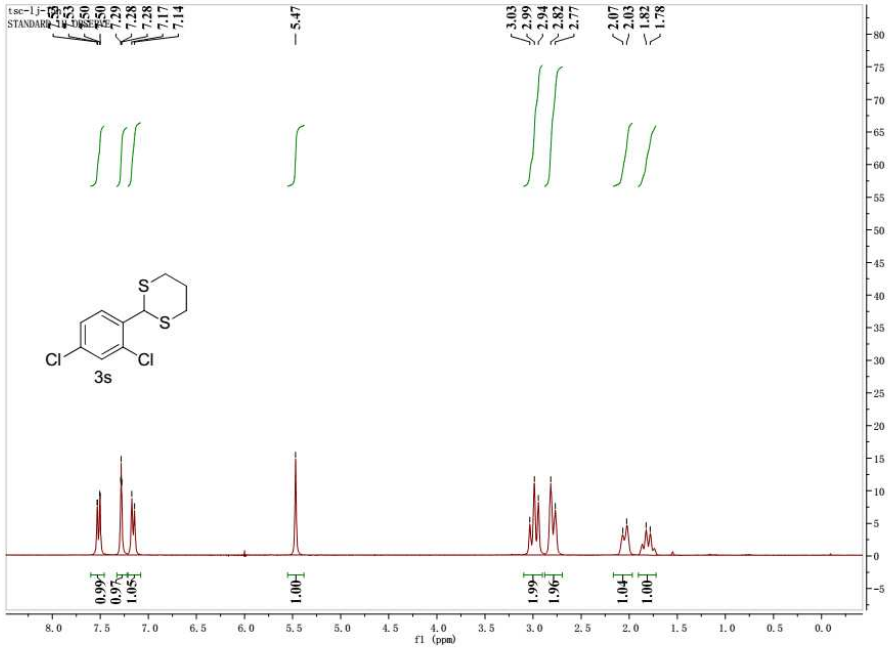

tsc-0529-lj-49c
13C OBSERVE
155.65
140.52
129.97
120.11
115.60
114.74
77.41
76.99
76.57
51.10
31.92
24.98
HO
S
S
3p
f1 (ppm)

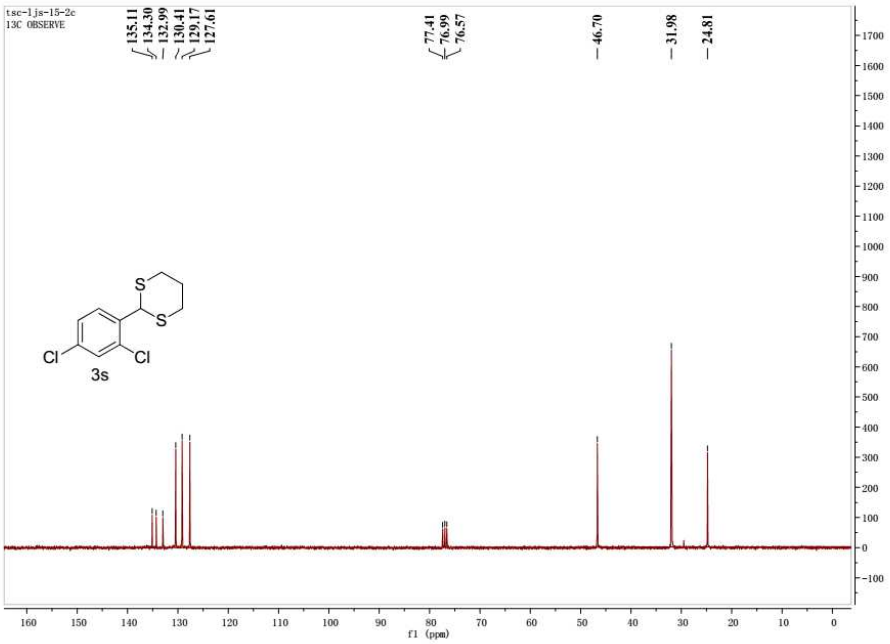

3q
f1 (ppm)

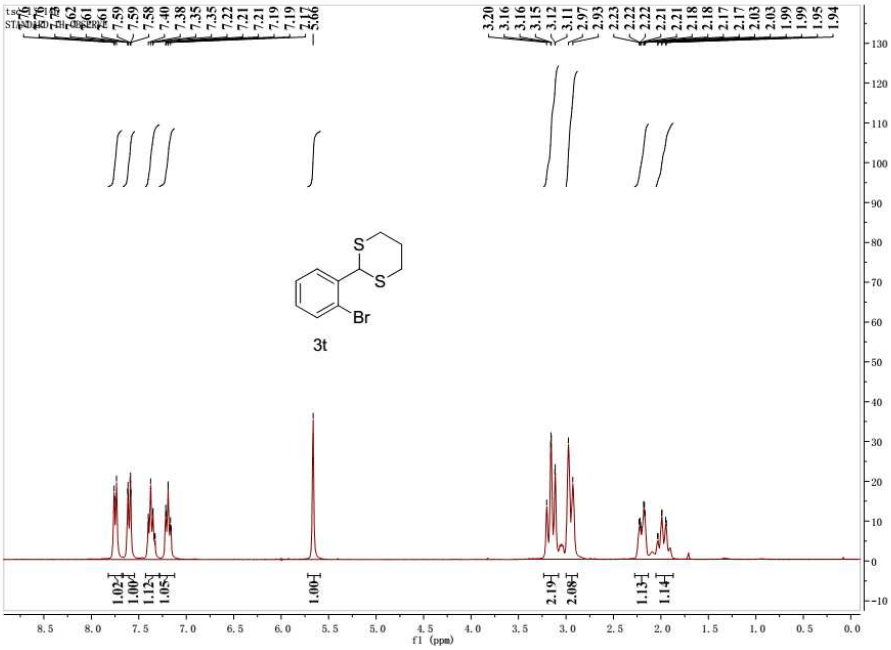

3q
f1 (ppm)

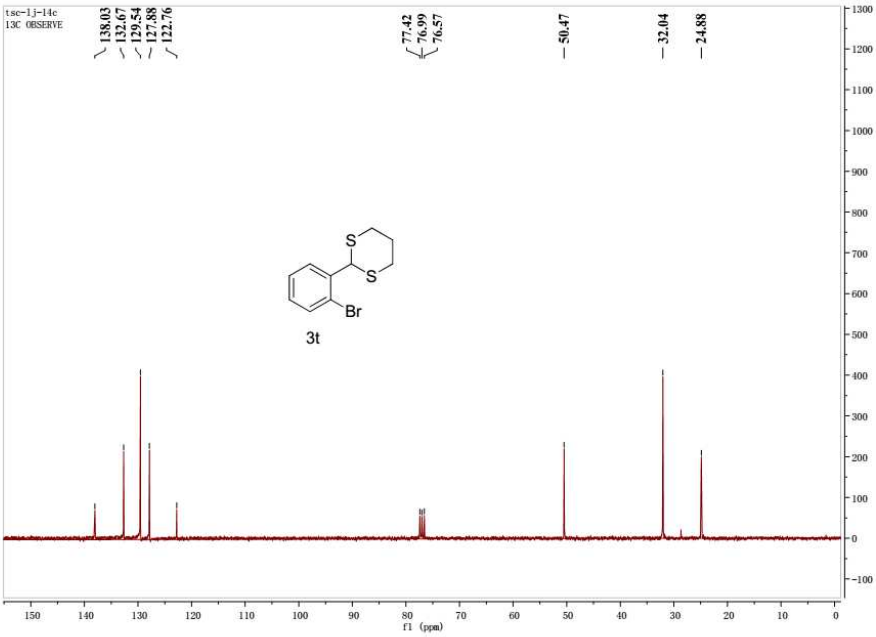

3r
f1 (ppm)

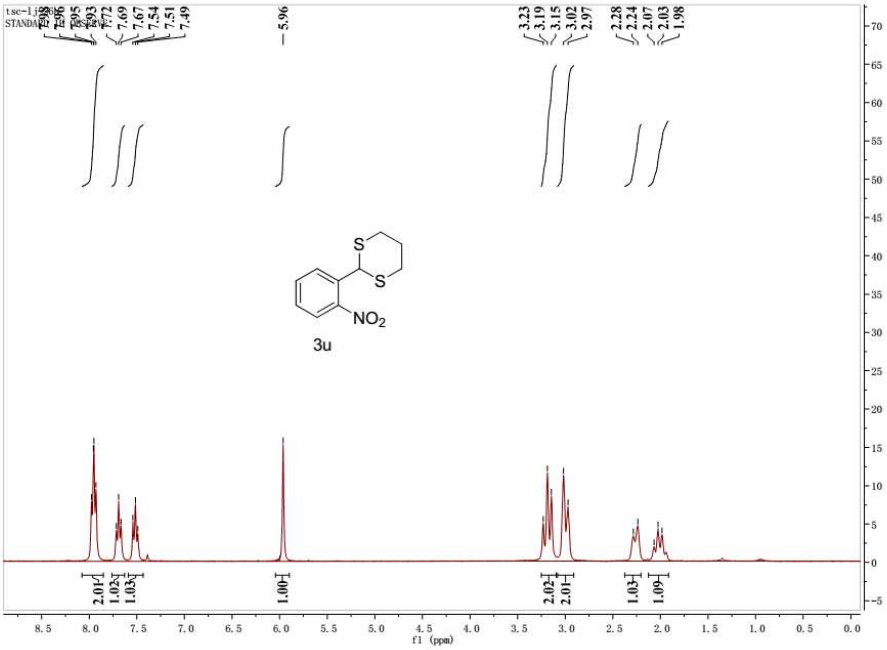

3r
f1 (ppm)

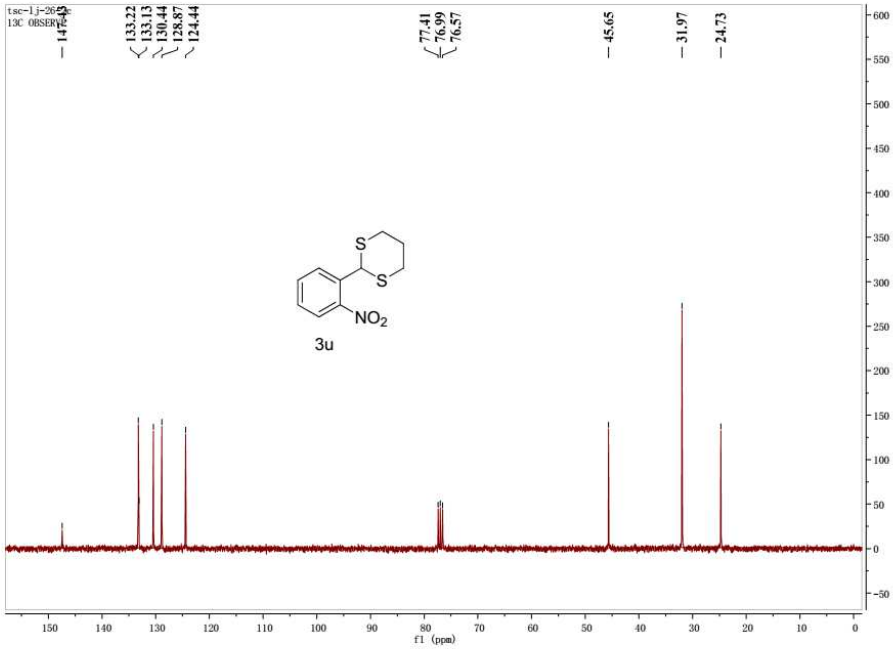

tsc-lj-
STANDARD
7.53
7.50
7.29
7.28
7.28
7.17
7.14
5.47
3.03
2.99
2.94
2.82
2.77
2.07
2.03
1.82
1.78
S
S
Cl
Cl
3s
0.99
0.97
1.05
1.00
1.99
1.96
1.04
1.00
f1 (ppm)

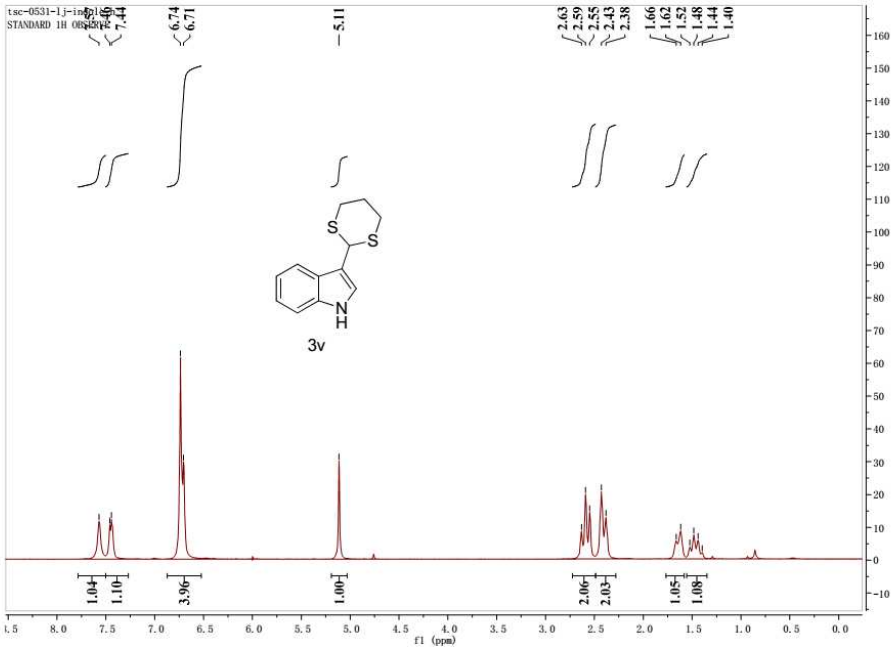

tsc-lj-15-2c
13C OBSERVE
135.11
134.30
132.99
130.41
129.17
127.61
77.41
76.99
76.57
46.70
31.98
24.81
S
S
Cl
Cl
3s
f1 (ppm)

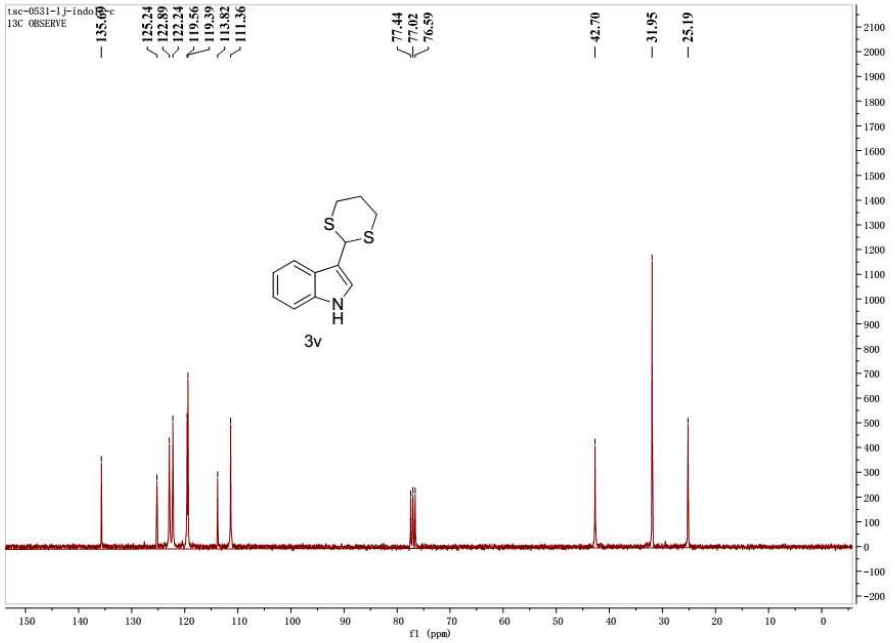

3t
f1 (ppm)

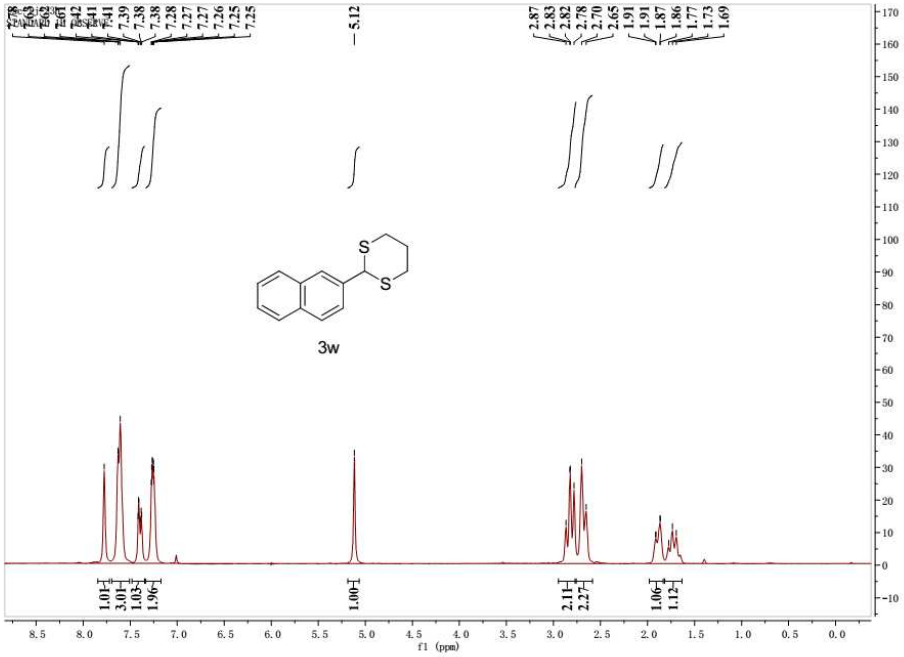

tsc-1j-14c
13C OBSERVE
138.03
132.67
129.54
127.88
122.76
77.42
76.99
76.57
50.47
32.04
24.88
3t
f1 (ppm)

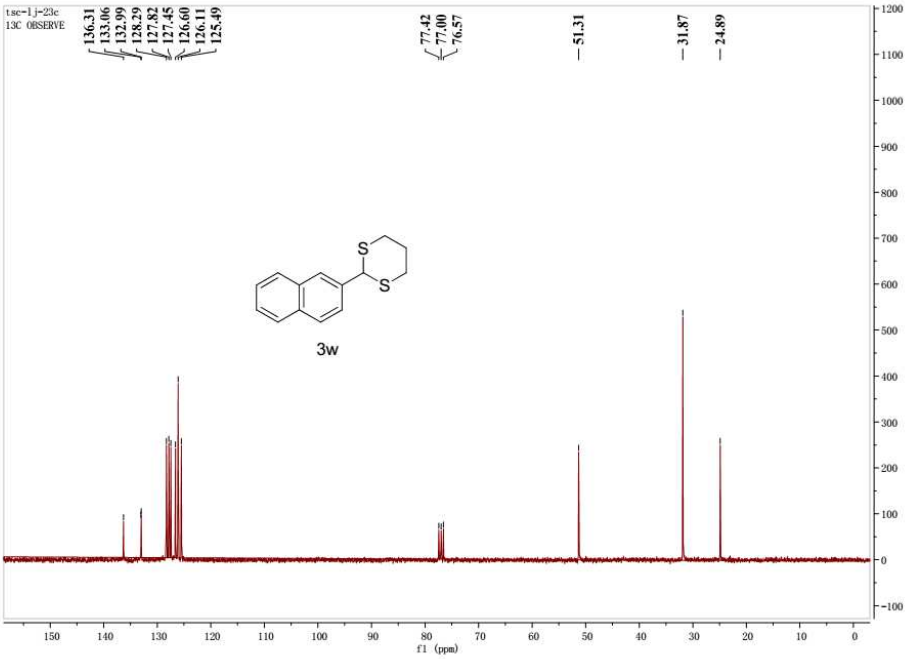

7.49
7.51
7.54
7.67
7.69
7.72
5.96
3.23
3.19
3.15
3.02
2.97
2.28
2.24
2.07
2.03
1.98
S
S
NO2
3u
2.01
1.03
1.03
1.00
2.02
2.01
1.03
1.09
f1 (ppm)

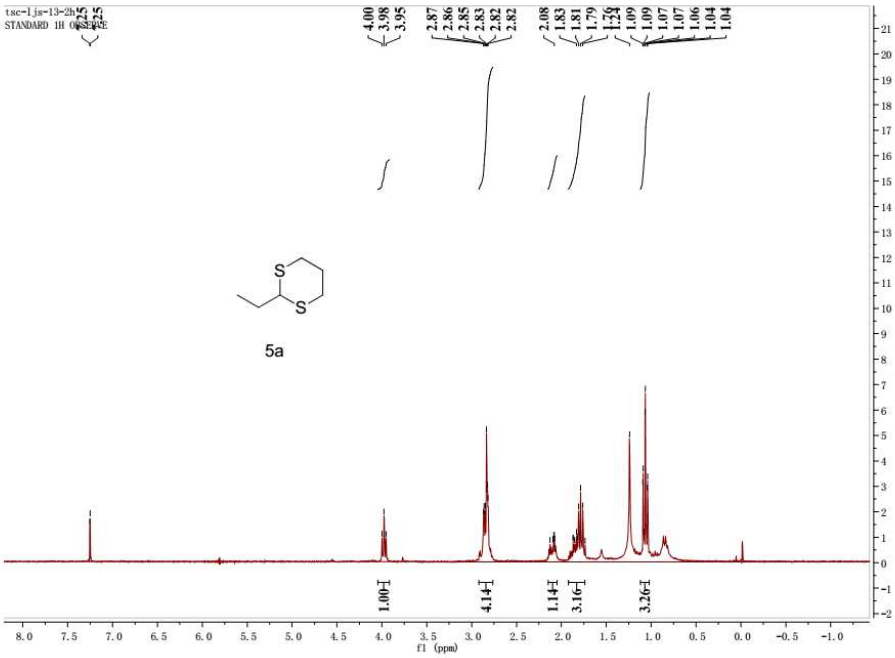

133.22
133.13
130.44
128.87
124.44
77.41
76.99
76.57
45.65
31.97
24.73
S
S
NO2
3u
f1 (ppm)

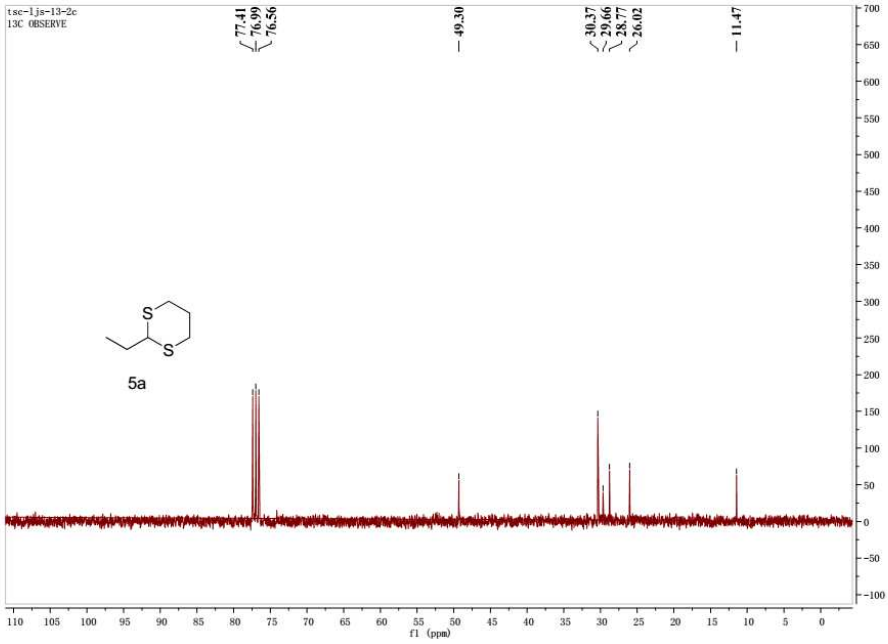

STANDARD 1H OBSERVE
7.46
7.44
6.74
6.71
5.11
2.63
2.59
2.55
2.43
2.38
1.66
1.62
1.52
1.48
1.44
1.40
1.04
1.10
3.96
1.00
2.06
2.03
1.05
1.08
f1 (ppm)
3v

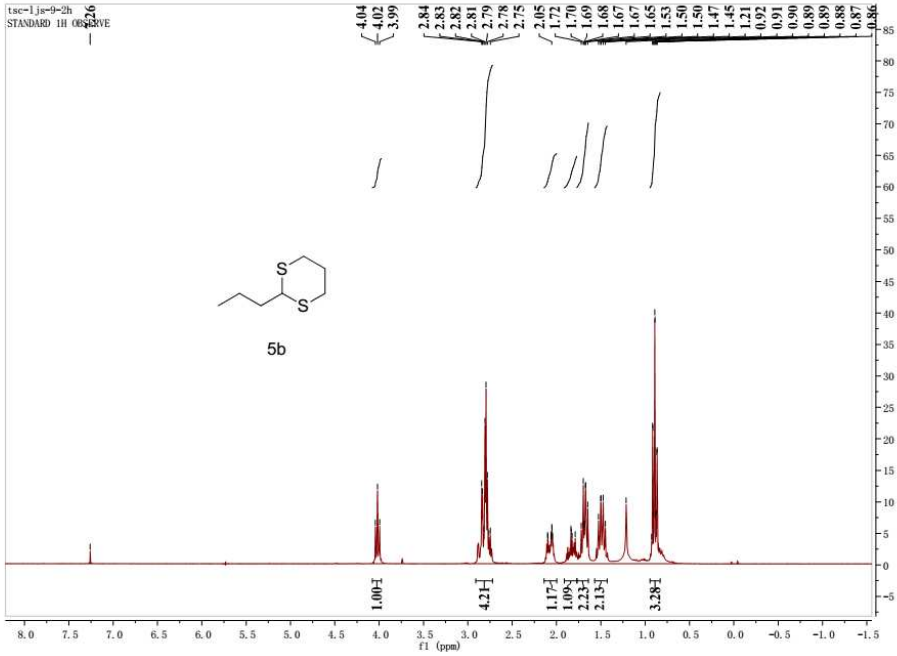

13C OBSERVE
135.60
125.24
122.89
122.24
119.56
119.39
113.82
111.36
77.44
77.02
76.59
42.70
31.95
25.19
f1 (ppm)
3v

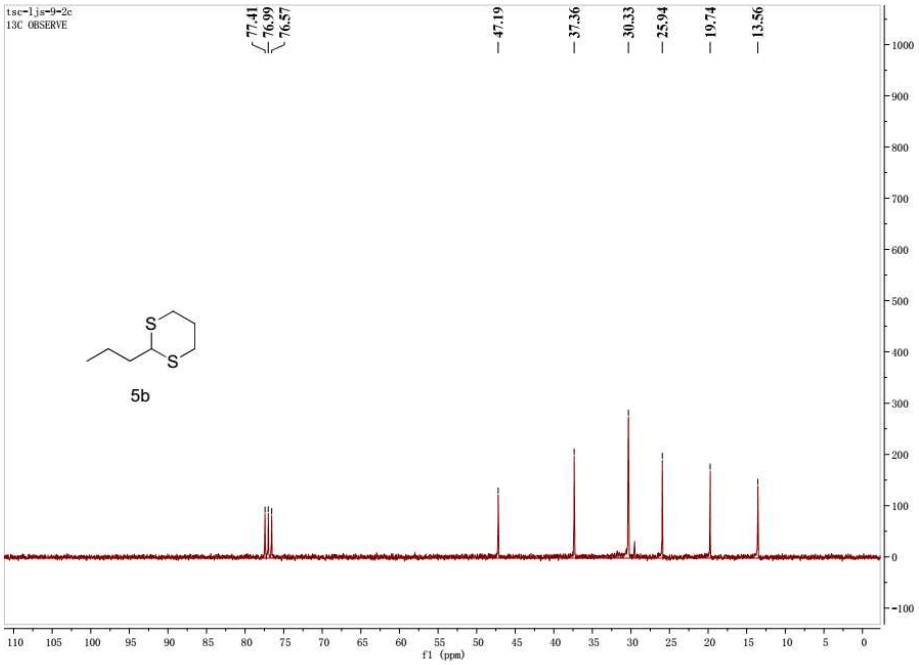

7.39
7.38
7.38
7.28
7.27
7.27
7.26
7.25
7.25
5.12
2.87
2.83
2.82
2.78
2.70
2.65
1.91
1.91
1.87
1.86
1.77
1.73
1.69
S
S
3w
1.01
3.01
1.03
1.96
1.00
2.11
2.27
1.06
1.12
f1 (ppm)

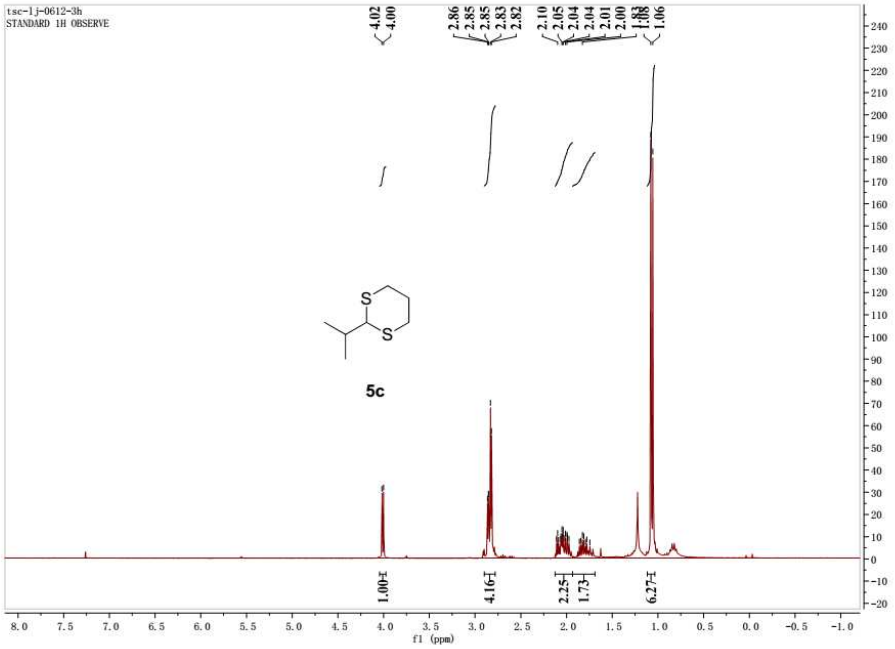

tsc-lj-23c
13C OBSERVE
136.31
133.06
132.99
128.29
127.82
127.45
126.60
126.11
125.49
77.42
77.00
76.57
51.31
31.87
24.89
S
S
3w
f1 (ppm)

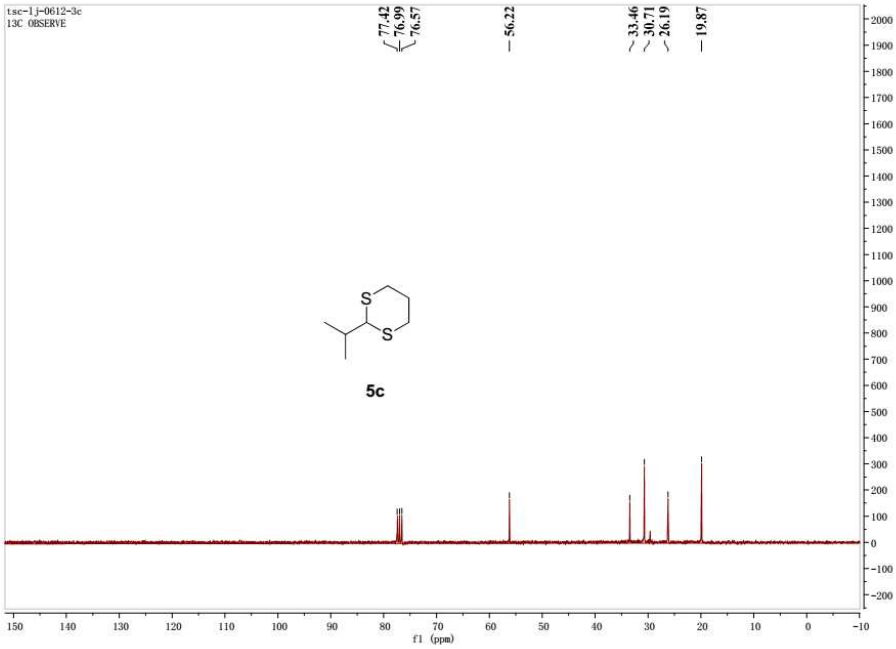

tsc-ljs-13-2h
STANDARD 1H OBSERVE
4.00
3.98
3.95
2.87
2.86
2.85
2.83
2.82
2.82
2.08
1.83
1.81
1.79
1.09
1.07
1.07
1.06
1.04
1.04
1.00
4.14
1.14
3.16
3.26
f1 (ppm)
5a

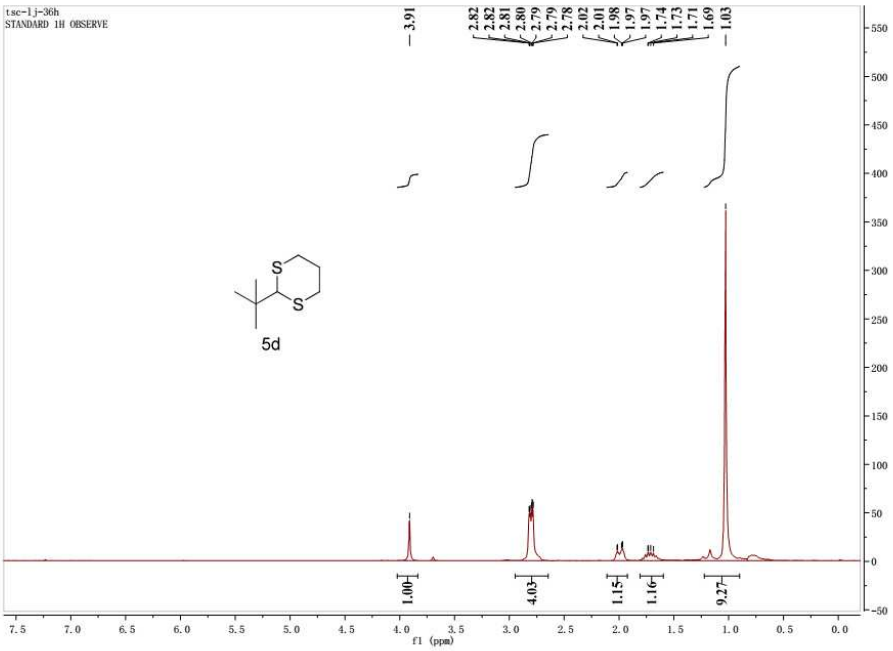

tsc-ljs-13-2c
13C OBSERVE
77.41
76.99
76.56
49.30
30.37
29.66
28.77
26.02
11.47
f1 (ppm)
5a

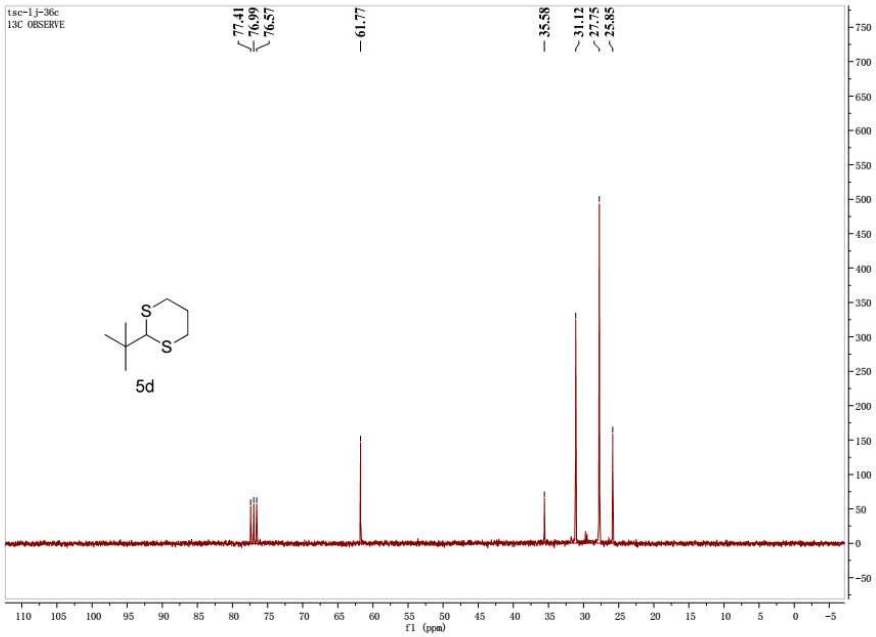

tsc-ljs-9-2h
STANDARD 1H OBSERVE
5b
f1 (ppm)

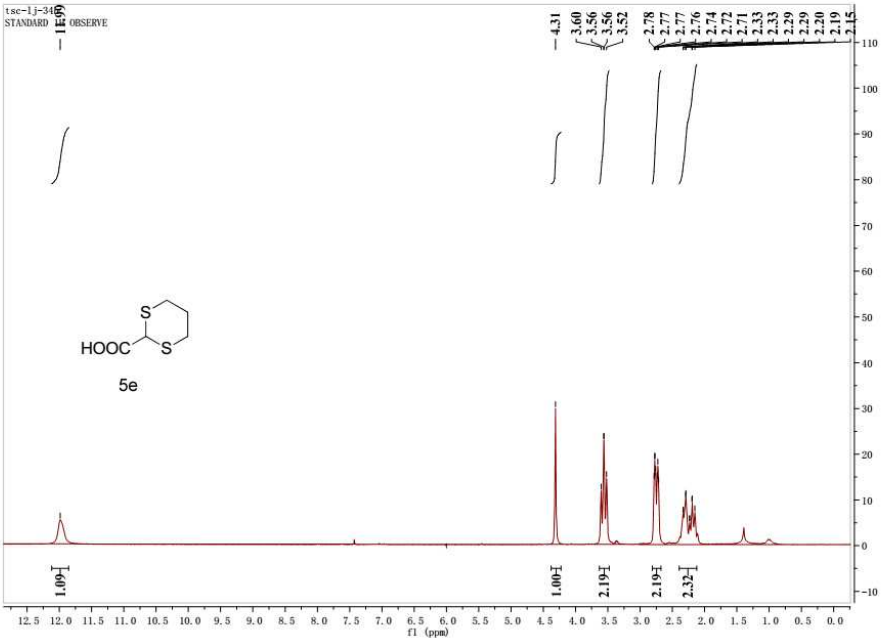

tsc-ljs-9-2c
13C OBSERVE
5b
f1 (ppm)

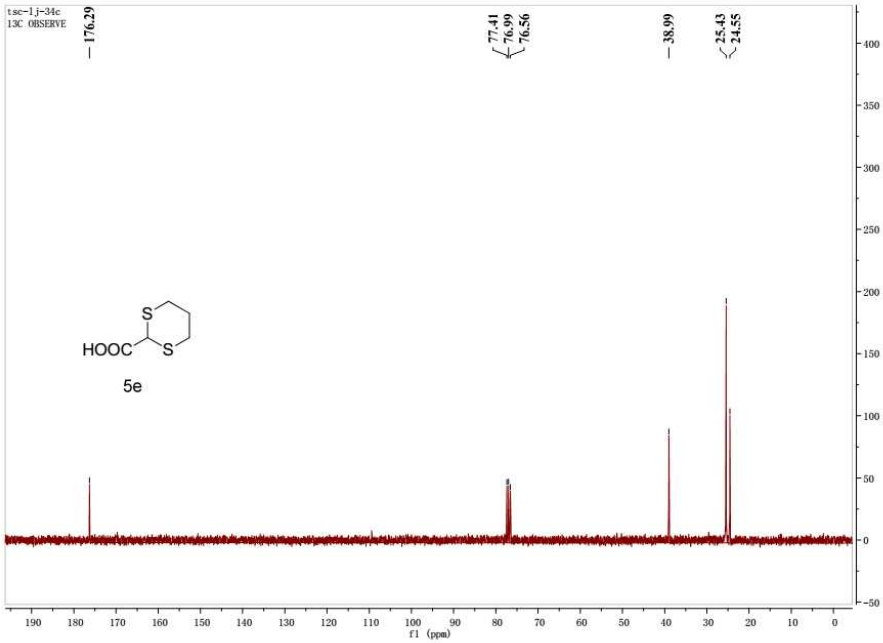

tsc-1j-0612-3h
STANDARD 1H OBSERVE
4.02
4.00
2.86
2.85
2.85
2.83
2.82
2.10
2.05
2.04
2.04
2.01
2.00
1.06
S
S
5c
1.00
4.16
2.25
1.73
6.27
f1 (ppm)

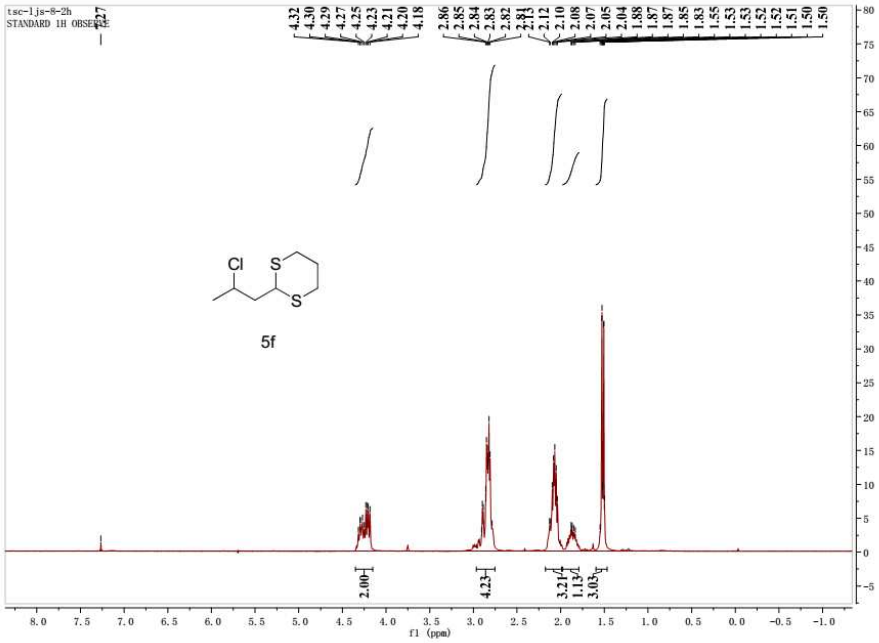

tsc-1j-0612-3c
13C OBSERVE
77.42
76.99
76.57
56.22
33.46
30.71
26.19
19.87
S
S
5c
f1 (ppm)

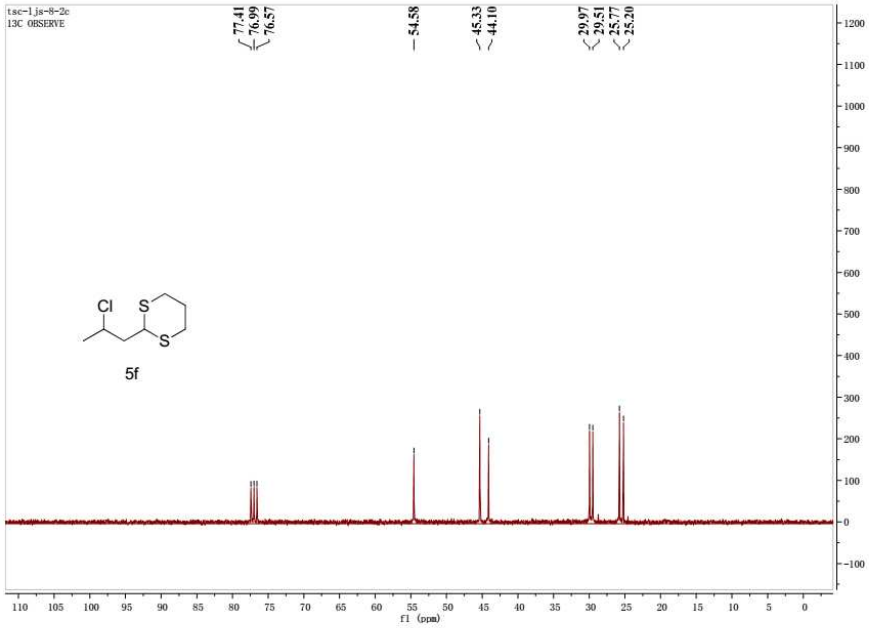

tsc-lj-36h
STANDARD 1H OBSERVE
3.91
2.82
2.82
2.81
2.80
2.79
2.79
2.78
2.02
2.01
1.98
1.97
1.97
1.74
1.73
1.71
1.69
1.03
1.00
4.03
1.15
1.16
9.27
f1 (ppm)
5d


tsc-lj-36c
13C OBSERVE
77.41
76.99
76.57
61.77
35.58
31.12
27.75
25.85
f1 (ppm)
5d


tsc-lj-34b
STANDARD 1H OBSERVE
12.09
4.31
3.60
3.56
3.56
3.52
2.78
2.77
2.77
2.76
2.74
2.72
2.71
2.33
2.33
2.29
2.29
2.20
2.19
2.15
1.09
1.00
2.19
2.19
2.32
HOOC
S
S
5e
f1 (ppm)


tsc-lj-34c
13C OBSERVE
176.29
77.41
76.99
76.56
38.99
25.43
24.55
HOOC
S
S
5e
f1 (ppm)


tsc-1js-8-2h
STANDARD 1H OBSERVE
Cl
S
S
5f
2.00
4.23
3.21
1.13
3.03
f1 (ppm)


tsc-1js-8-2c
13C OBSERVE
77.41
76.99
76.57
54.58
45.33
44.10
29.97
29.51
25.77
25.20
Cl
S
S
5f
f1 (ppm)