Supporting Information

Alcohol-mediated direct dithioacetalization of alkynes with 2-chloro-1,3-dithiane for the synthesis of Markovnikov dithianes

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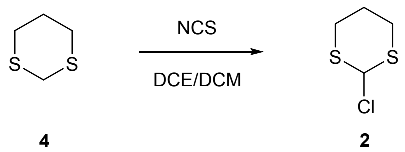
1. General information

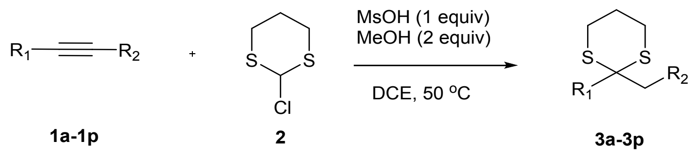
Analytical grade solvents and commercially available reagents were used as received. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were generally performed on silica gel (200-300 mesh) in petroleum (bp. 60-90 °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 data were recorded with Bruker 300 MHz or 400 MHz with tetramethylsilane as internal standard. ^{19}F NMR data was recorded with Bruker 400 MHz with tetramethylsilane as internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane (0 ppm for ^{1}H) and $CDCl_3$ (77.00 ppm for ^{13}C), respectively. MS were measured on a HP-5988 spectrometer by direct inlet at 70 eV.

2. Experimental section

2.1 General procedure for the synthesis of Markovnikov dithianes

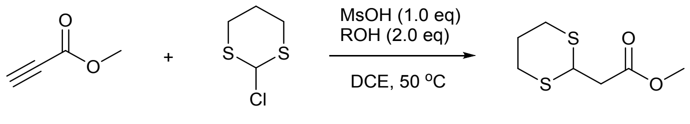


To a flame-dried 10 mL flask were sequentially added 1,3-dithiane **4** (300 mg, 2.5 mmol) and N-chlorosuccinimide (NCS) (400 mg, 3 mmol), 1,2-dichloroethane (DCE) or dichloromethane (DCM) (3 mL), after dissolved the mixture was stirred at 0 °C for 40 mins. The mixture was allowed to stir at room temperature for 2 h. Then the reaction mixture can be used directly for dithioacetalization with a variety of alkynes in DCE/DCM solutions. Alternatively, the reaction mixture in DCE can be used directly for the dithioacetalization in a one-pot procedure without purification.[1]



To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (31 mg, 0.2 mmol), **1a-1p** (0.18 mmol) and MsOH (19 mg, 0.2 mmol), MeOH (13 mg, 0.40 mmol) were added successively via syringe at room temperature. Reaction mixture was stirred at 50 °C for 8-24 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: 50～2: 50) to yield the product **3a-3p**.

2.2 The synthesis of dithiane 3q

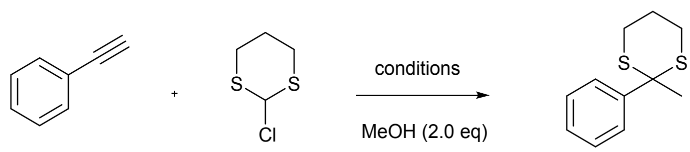


1q **2** **3q**

R = Me, Et, *t*-Bu,

To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (31 mg, 0.2 mmol), **1q** (15 mg, 0.18 mmol), MsOH (19 mg, 0.2 mmol), ROH (R = Me, Et, *t*-Bu; 0.40 mmol) were added successively via syringe at reaction temperature. Reaction mixture was stirred at 50 °C for 12 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 = 2: 50) to yield the **3q** product.

2.3 Optimization of acid, and temperature



1a **2** **3a**

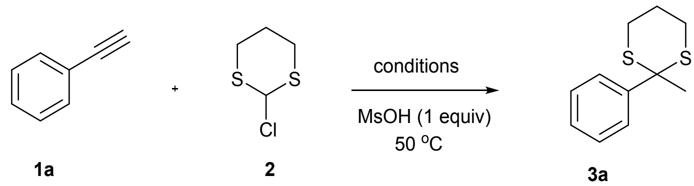
To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (31 mg, 0.2 mmol), **1a** (0.18 mmol), acid (*x* equiv) and MeOH (13mg, 0.40 mmol) were added successively via syringe at reaction temperature. Reaction mixture was stirred at 50～100 °C for 8-24 h until TLC analysis showed the reaction was completed. 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. Optimization of acid, and temperature

entry	acid (*x* equiv)	temp (°C)	time (h)	yield (%)

1	HCl (1.0)	50	24	trace
2	HBF_4 (1.0)	50	24	trace
3	BF_3 Et_2O (1.0)	50	10	82
4	$FeCl_3$ (0.2)	50	10	68
5	TsOH (1.0)	50	10	50
6	MsOH (1.0)	50	8	84
7	MsOH (0.2)	50	8	32
8	MsOH (0.6)	50	8	46
9	MsOH (0.8)	50	8	76
10	MsOH (2.0)	50	8	40
11	MsOH (1.0)	25	12	45
12	MsOH (1.0)	80	12	55
13	MsOH (1.0)	100	12	50
14	MsOH (0.8)	80	12	64
15	MsOH (0)	50	24	trace

2.4 Optimization of solvent and alcohol



To a flame-dried 10 mL flask 2-chloro-1,3-dithiane **2** (31 mg,0.2 mmol), **1a** (0.18 mmol), MsOH (19 mg, 0.2 mmol) and MeOH (*x* equiv) were added successively via syringe at reaction temperature in solvent (2 mL). Reaction mixture was stirred at 50 °C for 8-24 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.

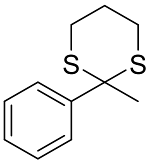
Table S2. Optimization of solvent and alcohol

entry	ROH(*x* equiv)	Solvent	yield (%)
1	H_2O	DCE	trace
2	MeOH (1.0)	DCE	76
3	MeOH (2.0)	DCE	84
4	MeOH (3.0)	DCE	69
5	C_2H_5OH (2.0)	DCE	82
6	*n*-PrOH (2.0)	DCE	80
7	*i*-PrOH(2.0)	DCE	32

8	*t*-BuOH (2.0)	DCE	56
9	MeOH (2.0)	DCM	79
10	MeOH (2.0)	THF	46
11	MeOH (2.0)	CH_3NO_2	48

3. Characterization of synthesized compounds 3a-3q

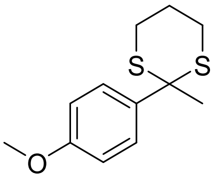
2-methyl-2-phenyl-1,3-dithiane(3a)[2]



3a

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.93 (m, 2H), 7.40 – 7.29 (m, 2H), 7.25 (d, *J* = 7.5 Hz, 1H), 2.75 – 2.65 (m, 4H), 1.97 – 1.87 (m, 2H), 1.78 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 143.6, 128.4, 127.6, 126.9, 53.8, 32.6, 27.9, 24.5.

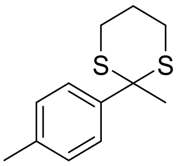
2-(4-methoxyphenyl)-2-methyl-1,3-dithiane(3b)[3]



3b

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.77 (d, *J* = 9.0 Hz, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 3.75 (s, 3H), 2.71 – 2.65 (m, 4H), 1.91 – 1.85 (m, 2H), 1.74 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 158.3, 135.5, 128.8, 113.5, 55.0, 53.2, 32.4, 27.9, 24.5.

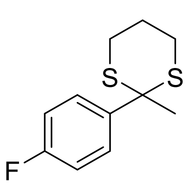
2-methyl-2-(p-tolyl)-1,3-dithiane(3c)[2]



3c

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 2.72 (m, 4H), 2.35 (s, 3H), 1.94 (m, 2H), 1.79 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 140.7, 136.7, 129.2, 127.6, 53.7, 32.7, 28.0, 24.7, 20.9.

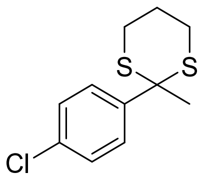
2-(4-fluorophenyl)-2-methyl-1,3-dithiane(3d)[3]



3d

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.91 (m, 2H), 7.04 (t, *J* = 8.8 Hz, 2H), 2.96 – 2.57 (m, 4H), 2.08 – 1.87 (m, 2H), 1.79 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 161.7 (d, *J* = 246.8 Hz), 139.5, 129.6 (d, *J* = 8.0 Hz), 115.1 (d, *J* = 21.2 Hz), 53.3, 32.8, 28.0, 24.5. **^{19}F NMR (376 MHz, CDCl$_3$)** δ -116.0 (s, 1F).

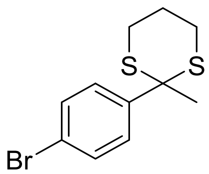
2-(4-chlorophenyl)-2-methyl-1,3-dithiane(3e)[2]



3e

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.88 (d, *J* = 8.7 Hz, 2H), 7.32 (d, *J* = 8.7 Hz, 2H), 2.74 – 2.62 (m, 4H), 1.99 – 1.87 (m, 2H), 1.75 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 142.4, 132.7, 129.2, 128.4, 53.2, 32.6, 27.9, 24.3.

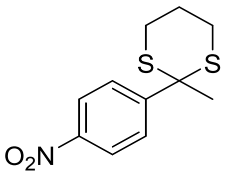
2-(4-bromophenyl)-2-methyl-1,3-dithiane(3f)[4]



3f

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.83 (d, *J* = 9.0 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 2.78 – 2.61 (m, 4H), 2.00 – 1.88 (m, 2H), 1.75 (d, *J* = 0.9 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 143.0, 131.5, 129.8, 121.1, 53.5, 32.8, 28.0, 24.4.

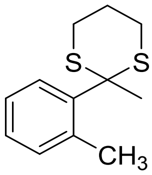
2-methyl-2-(4-nitrophenyl)-1,3-dithiane(3g)[5]



3g

^{1}H NMR (300 MHz, CDCl$_3$) δ 8.23 (d, *J* = 9.0 Hz, 2H), 8.16 (d, *J* = 9.0 Hz, 2H), 2.79 – 2.61 (m, 4H), 1.99 (m, 2H), 1.78 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 151.6, 146.7, 129.0, 123.7, 53.3, 32.7, 27.9, 24.1.

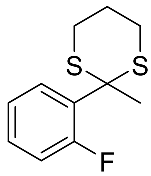
2-methyl-2-(o-tolyl)-1,3-dithiane(3h)[4]



3h

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.98 – 7.90 (m, 1H), 7.24 – 7.12 (m, 3H), 2.84 (m, 3H), 2.75 (s, 3H), 2.74 – 2.69 (m, 1H), 2.02 (s, 3H), 1.94 (m, 2H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 139.8, 137.6, 133.5, 129.2, 127.4, 125.5, 53.1, 29.4, 28.2, 24.4, 23.4.

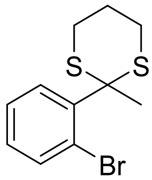
2-(2-fluorophenyl)-2-methyl-1,3-dithiane(3i)[4]



3i

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.96 – 7.81 (m, 1H), 7.34 – 7.21 (m, 1H), 7.17 – 6.99 (m, 2H), 2.89 – 2.72 (m, 4H), 2.06 – 1.95 (m, 2H), 1.95 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 160.7 (d, *J* = 250.5 Hz), 131.1 (d, *J* = 2.2 Hz), 130.7 (d, *J* = 8.1 Hz), 129.3 (d, *J* = 9.0 Hz), 123.4 (d, *J* = 3.7 Hz), 117.2 (d, *J* = 24.4 Hz), 50.1, 29.5, 28.4, 24.4. **^{19}F NMR (376 MHz, CDCl$_3$)** δ -103.8 (s, 1F).

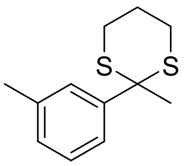
2-(2-bromophenyl)-2-methyl-1,3-dithiane(3j)[4]



3j

^{1}H NMR (300 MHz, CDCl$_3$) δ 8.23 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.66 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.38 – 7.24 (m, 1H), 7.17 – 6.99 (m, 1H), 2.93 – 2.59 (m, 4H), 2.07 (s, 3H), 2.01 – 1.79 (m, 2H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 140.3, 136.8, 132.1, 128.6, 126.9, 122.5, 53.7, 28.8, 28.6, 24.2.

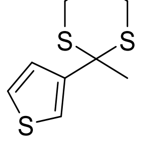
2-methyl-2-(m-tolyl)-1,3-dithiane(3k)[4]



3k

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.82 – 7.68 (m, 2H), 7.30 – 7.22 (m, 1H), 7.13 – 7.01 (m, 1H), 2.85 – 2.60 (m, 4H), 2.38 (s, 3H), 2.00 – 1.89 (m, 2H), 1.80 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 143.7, 138.1, 128.4, 128.3, 127.8, 124.7, 53.8, 32.6, 28.1, 24.7, 21.6.

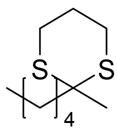
2-methyl-2-(thiophen-3-yl)-1,3-dithiane(3l)[6]



3l

^{1}H **NMR (300 MHz, $CDCl_3$)** δ 7.42 (d, *J* = 1.2 Hz, 1H), 7.27 – 7.20 (m, 2H), 2.76 – 2.65 (m, 4H), 1.94 – 1.81 (m, 2H), 1.74 (s, 3H). ^{13}C **NMR (75 MHz, $CDCl_3$)** δ 146.2, 127.6, 125.8, 123.3, 50.0, 32.1, 27.8, 24.6.

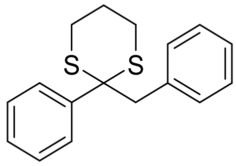
2-methyl-2-pentyl-1,3-dithiane(3m)[7]



3m

^{1}H **NMR (300 MHz, $CDCl_3$)** δ 2.80 – 2.75 (m, 4H), 1.89 (m, 2H), 1.85 – 1.79 (m, 2H), 1.55 (s, 3H), 1.47 – 1.36 (m, 2H), 1.29 – 1.20 (m, 4H), 0.83 (m, 3H). ^{13}C **NMR (75 MHz, $CDCl_3$)** δ 49.3, 41.7, 32.0, 27.7, 26.5, 25.4, 24.1, 22.5, 14.0.

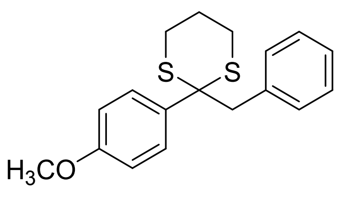
2-benzyl-2-phenyl-1,3-dithiane(3n)[8]



3n

^{1}H **NMR (300 MHz, $CDCl_3$)** δ 7.66 – 7.60 (m, 2H), 7.21 – 7.12 (m, 3H), 7.05 – 6.94 (m, 3H), 6.66 – 6.58 (m, 2H), 3.17 (s, 2H), 2.58 – 2.50 (m, 4H), 1.83 – 1.75 (m, 2H). ^{13}C **NMR (75 MHz, $CDCl_3$)** δ 140.3, 134.1, 130.7, 129.3, 128.1, 127.1, 126.8, 126.7, 59.6, 51.4, 27.3, 24.9.

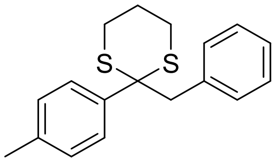
2-benzyl-2-(4-methoxyphenyl)-1,3-dithiane(3o)[9]



3o

^{1}H **NMR (300 MHz, $CDCl_3$)** δ 7.63 – 7.45 (m, 2H), 7.02 (m, 3H), 6.78 – 6.62 (m, 4H), 3.72 (s, 3H), 3.16 (s, 2H), 2.61 – 2.46 (m, 4H), 1.89 – 1.76 (m, 2H). ^{13}C **NMR (75 MHz, $CDCl_3$)** δ 158.3, 134.3, 132.2, 130.8, 130.7, 127.2, 126.7, 113.3, 59.2, 55.1, 51.5, 27.3, 25.0.

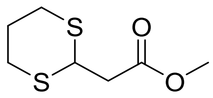
2-benzyl-2-(p-tolyl)-1,3-dithiane(3p)[10]



3p

^{1}H **NMR (300 MHz, $CDCl_3$)** δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.12 (m, 5H), 6.74 (d, *J* = 8.1 Hz, 2H), 3.24 (s, 2H), 2.77 – 2.51 (m, 4H), 2.34 (s, 3H), 1.96 – 1.83 (m, 2H). ^{13}C **NMR (75 MHz, $CDCl_3$)** δ 137.3, 136.4, 134.3, 130.8, 129.3, 128.9, 127.2, 126.7, 59.5, 51.4, 27.4, 25.0, 20.9.

methyl 2-(1,3-dithian-2-yl)acetate(3q)[11]



3q

^{1}H NMR (300 MHz, $CDCl_3$) δ 4.42 (t, *J* = 7.5 Hz, 1H), 3.74 (s, 3H), 3.00 – 2.86 (m, 4H), 2.81 (d, *J* = 7.5 Hz, 2H), 2.18 – 2.05 (m, 1H), 2.01 – 1.88 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.9, 51.9, 41.6, 40.2, 29.3, 25.0.

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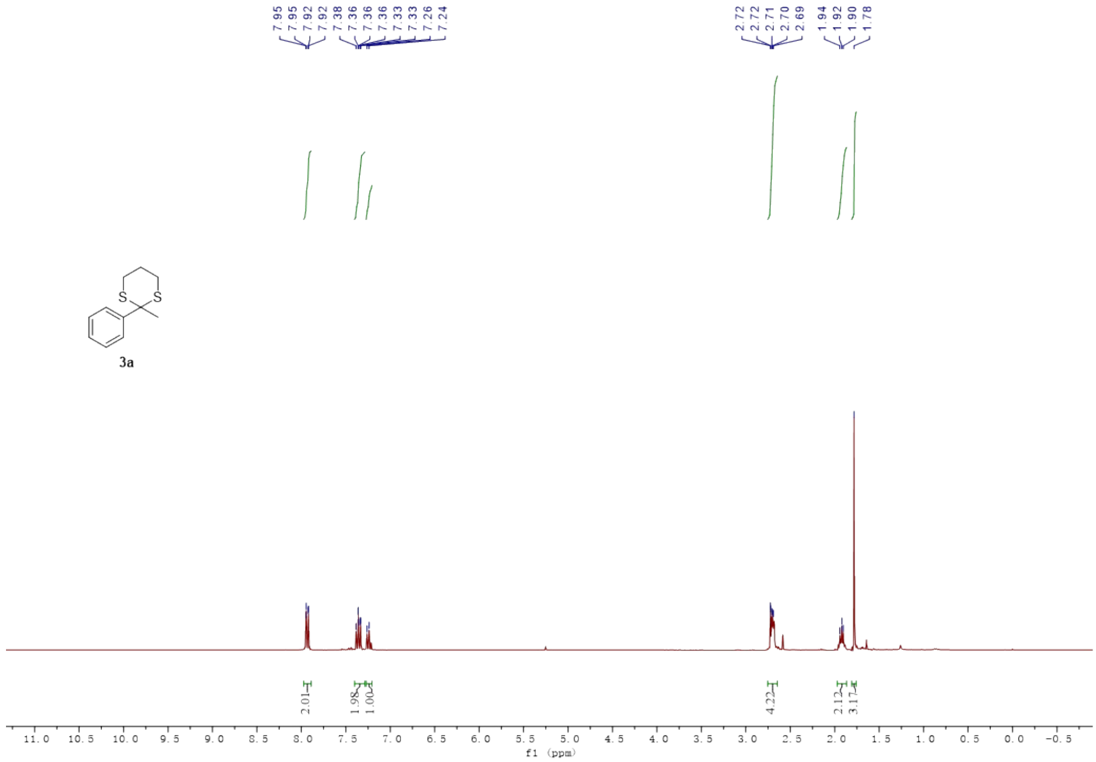
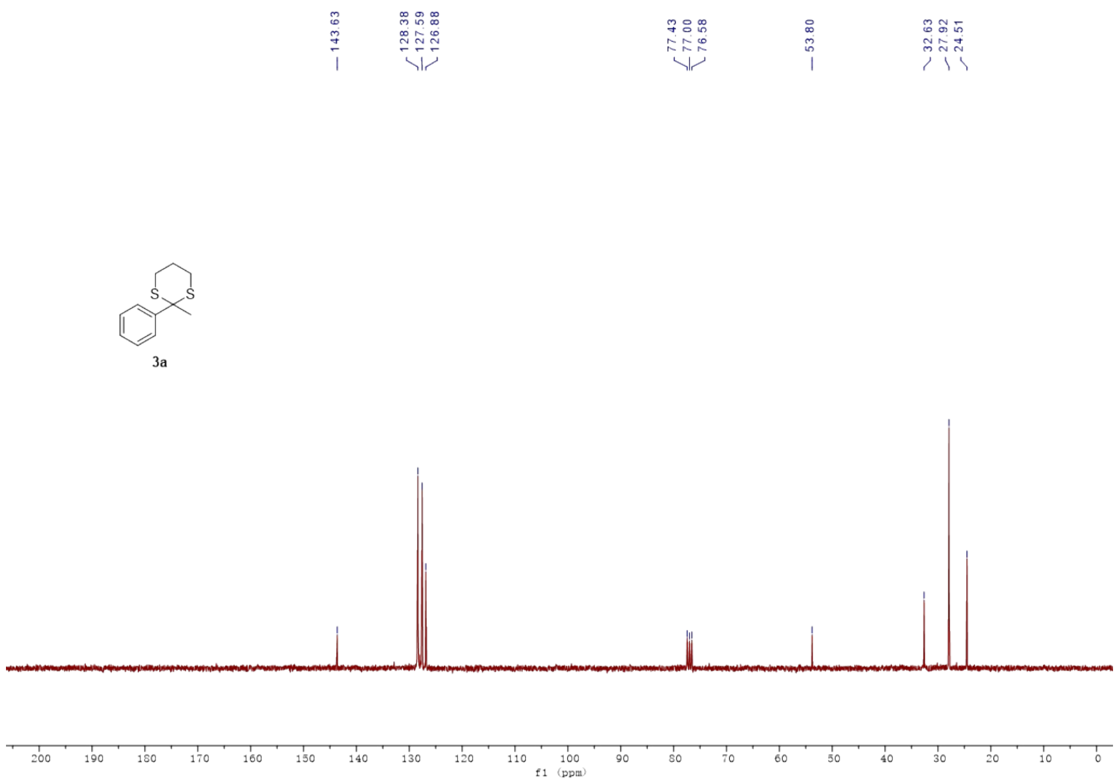
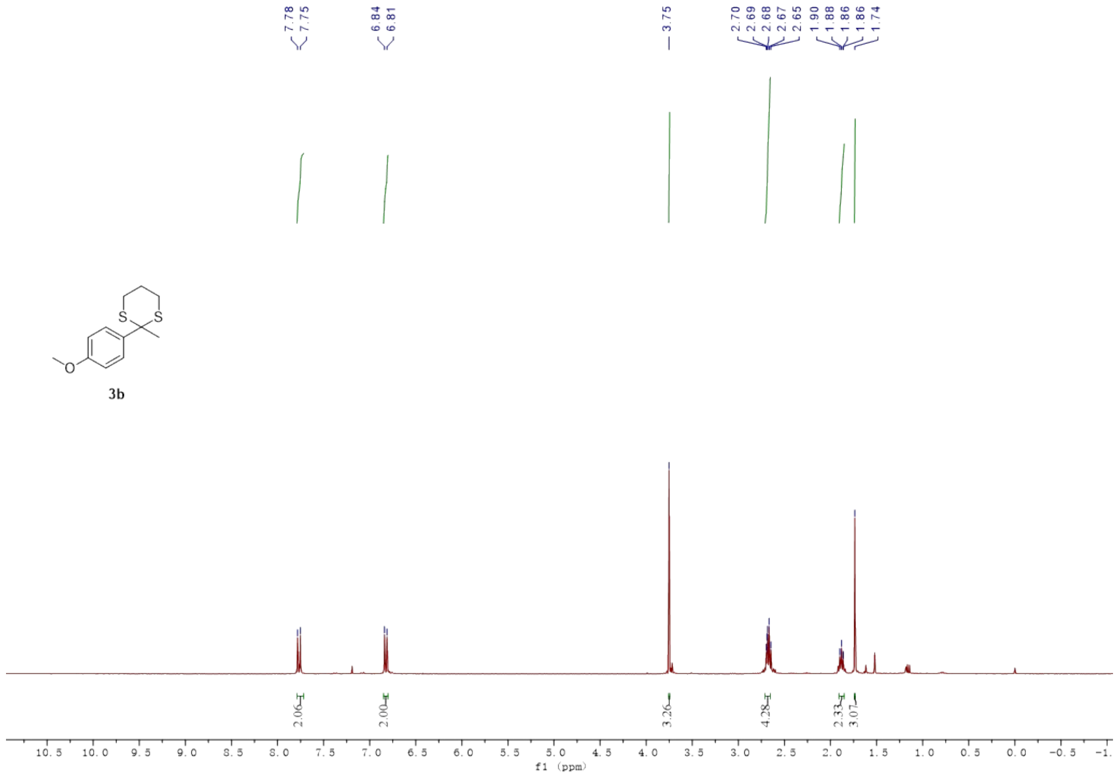
5. ^{1}H NMR and ^{13}C NMR spectra data of products



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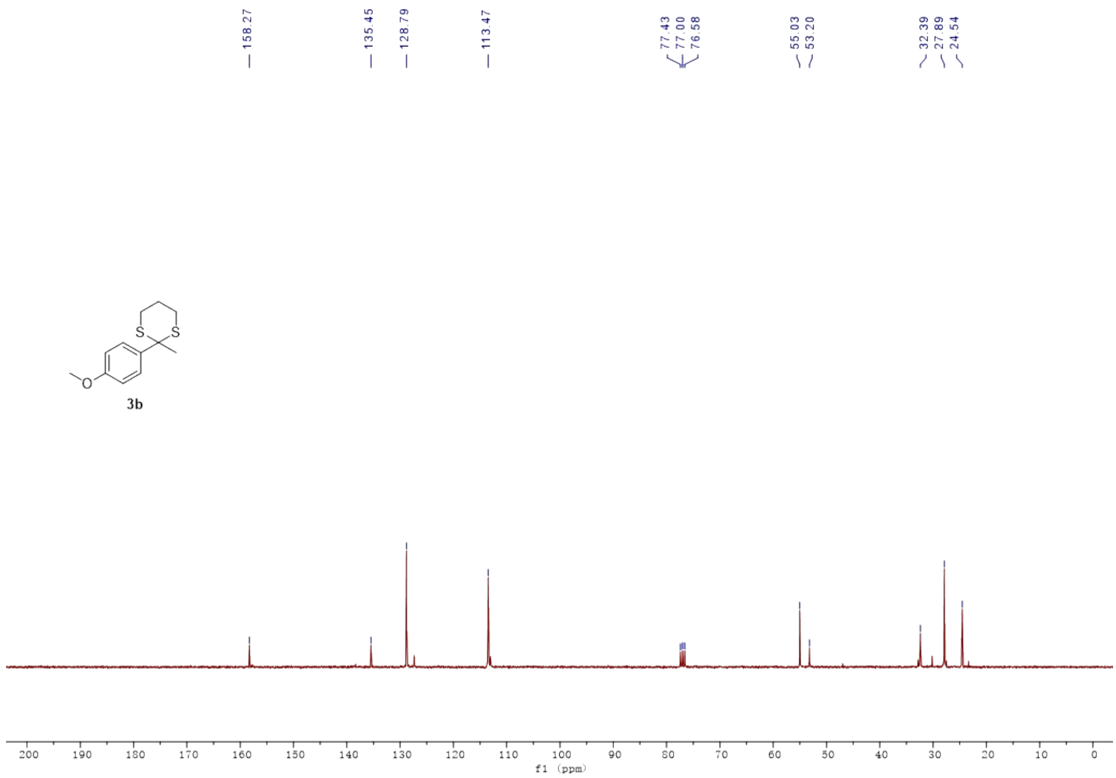
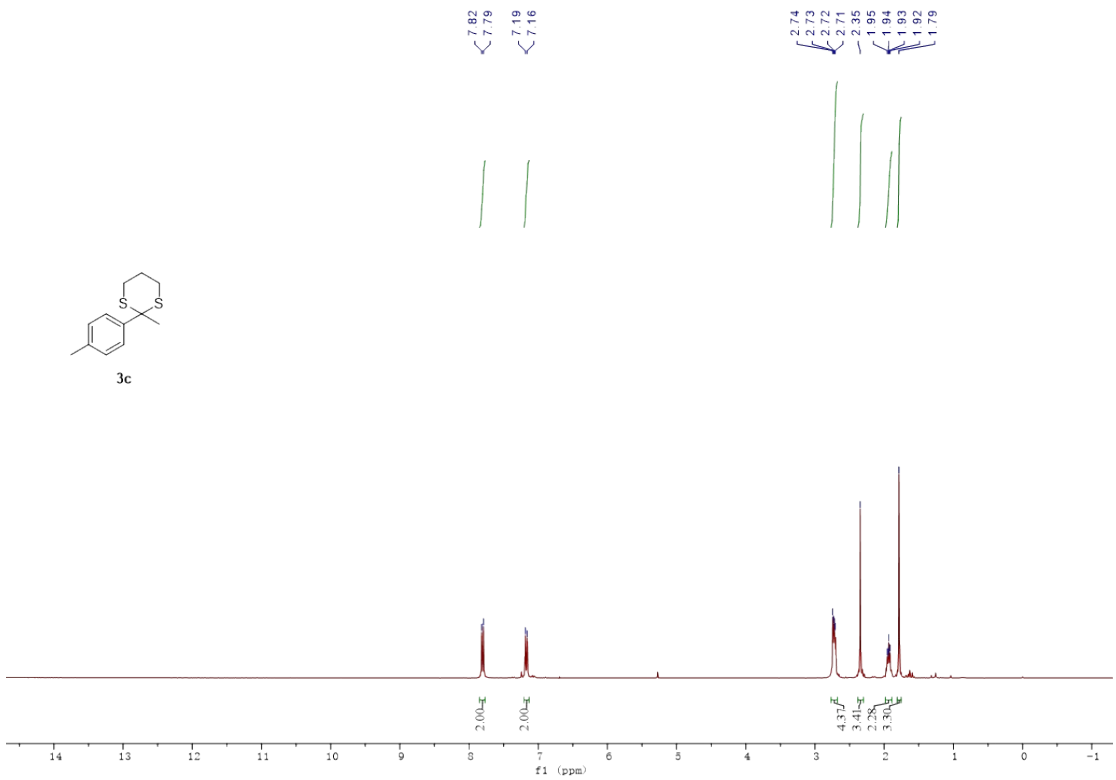
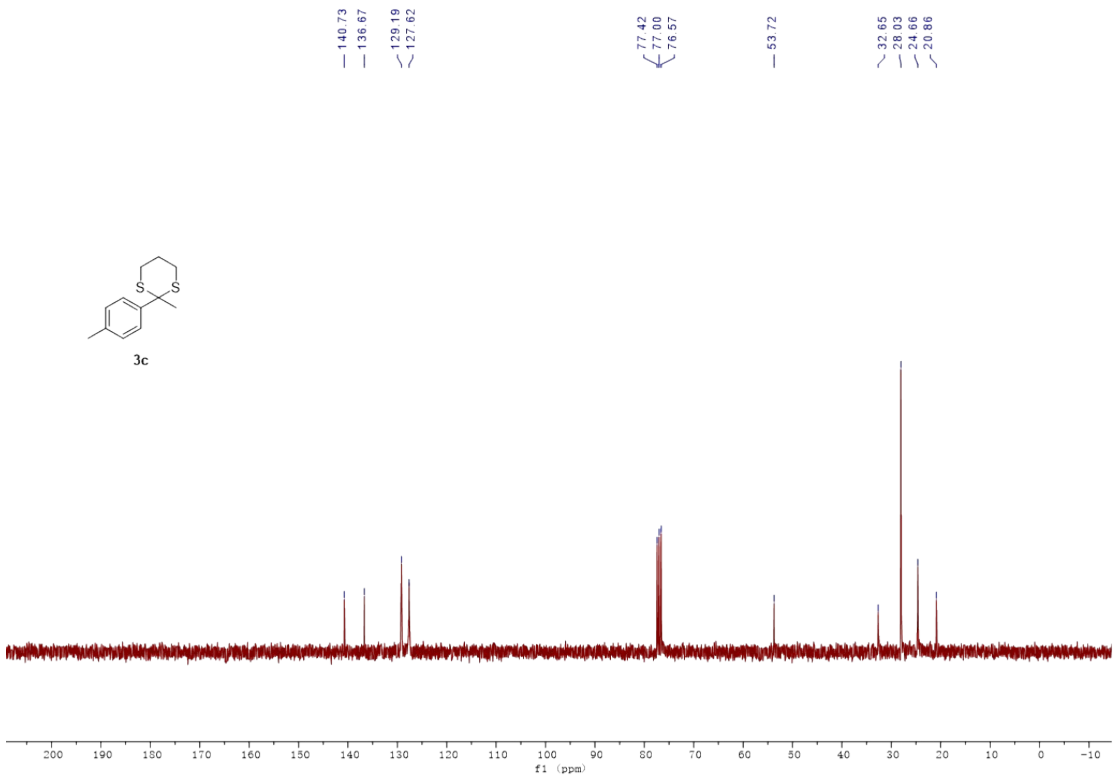
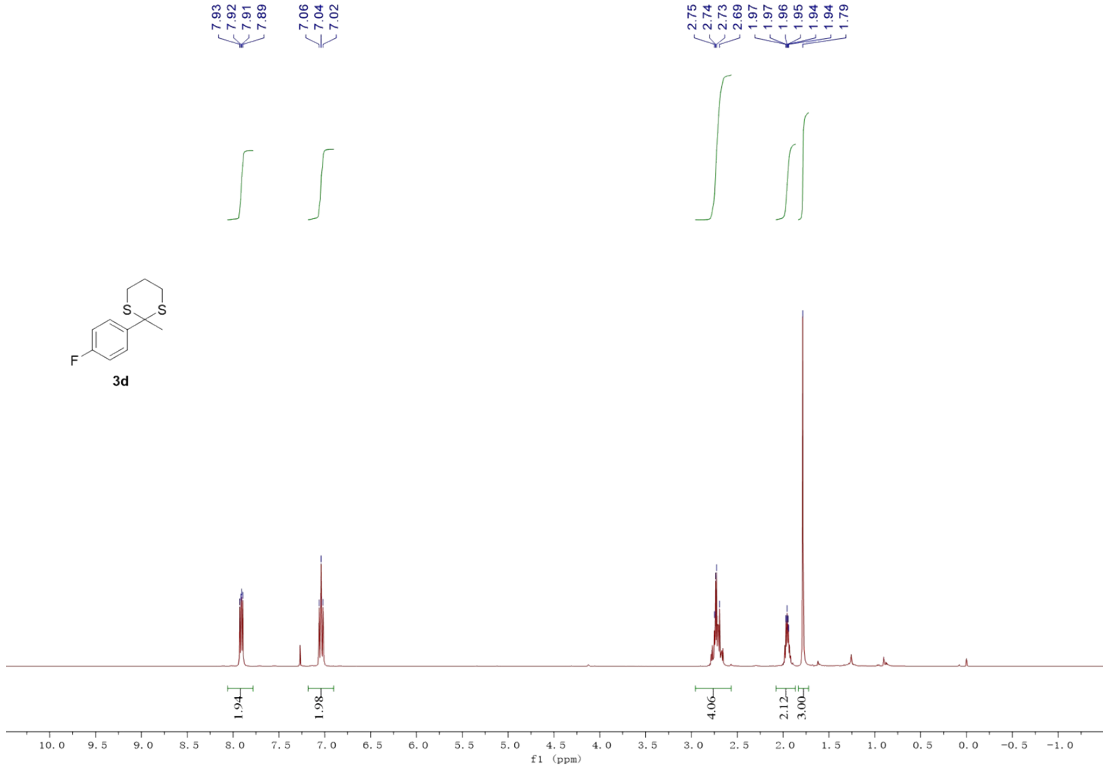
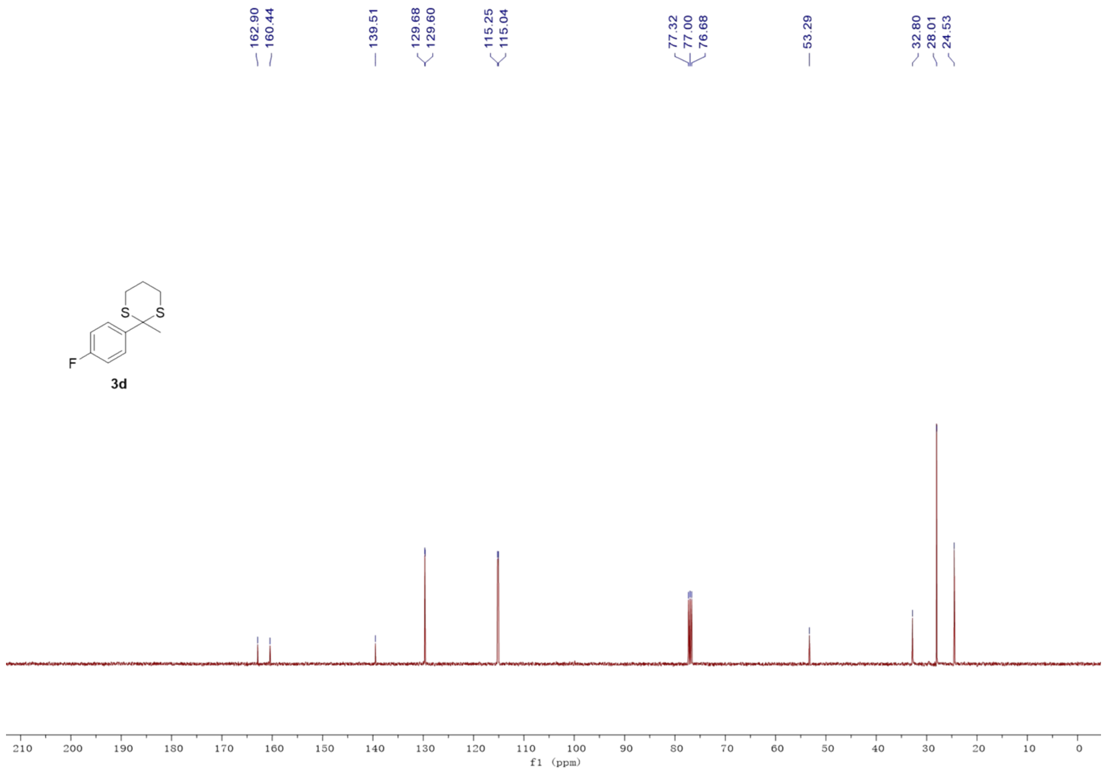
7.75
6.84
6.81
3.75
2.70
2.69
2.68
2.67
2.65
1.90
1.88
1.86
1.86
1.74
3b
2.06
2.00
3.26
4.28
2.33
3.07
f1 (ppm)


158.27
135.45
128.79
113.47
77.43
77.00
76.58
55.03
53.20
32.39
27.89
24.54
3b
f1 (ppm)


7.82
7.79
7.19
7.16
2.74
2.73
2.72
2.71
2.35
1.95
1.94
1.93
1.92
1.79
S
S
3c
2.00
2.00
4.37
3.41
2.28
3.30
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


140.73
136.67
129.19
127.62
77.42
77.00
76.57
53.72
32.65
28.03
24.66
20.86
S
S
3c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.93
7.92
7.91
7.89
7.06
7.04
7.02
2.75
2.74
2.73
2.69
1.97
1.97
1.96
1.95
1.94
1.94
1.79
S
S
F
3d
1.94
1.98
4.06
2.12
3.00
f1 (ppm)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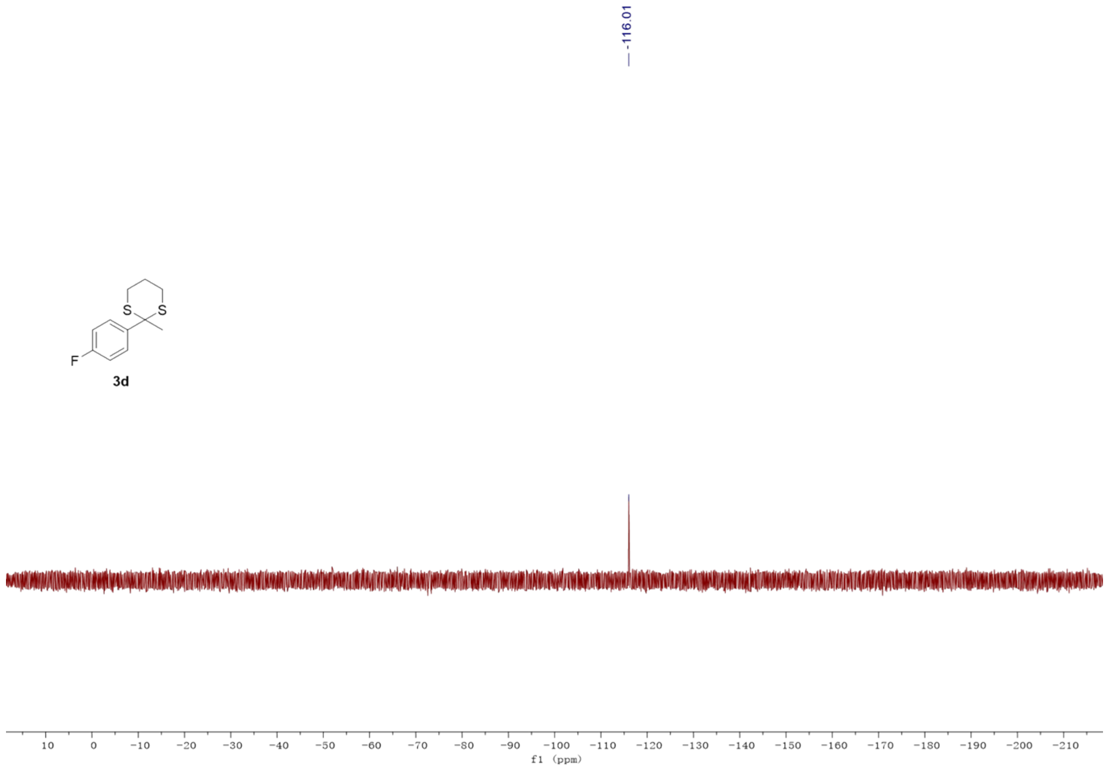
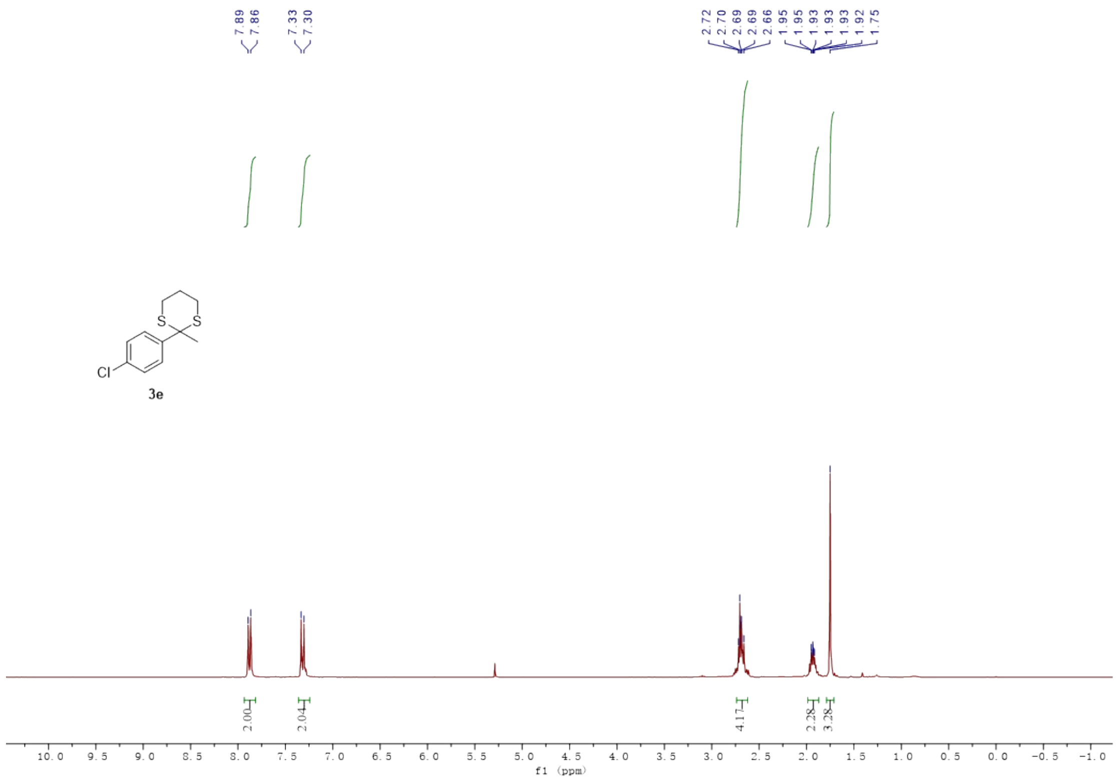
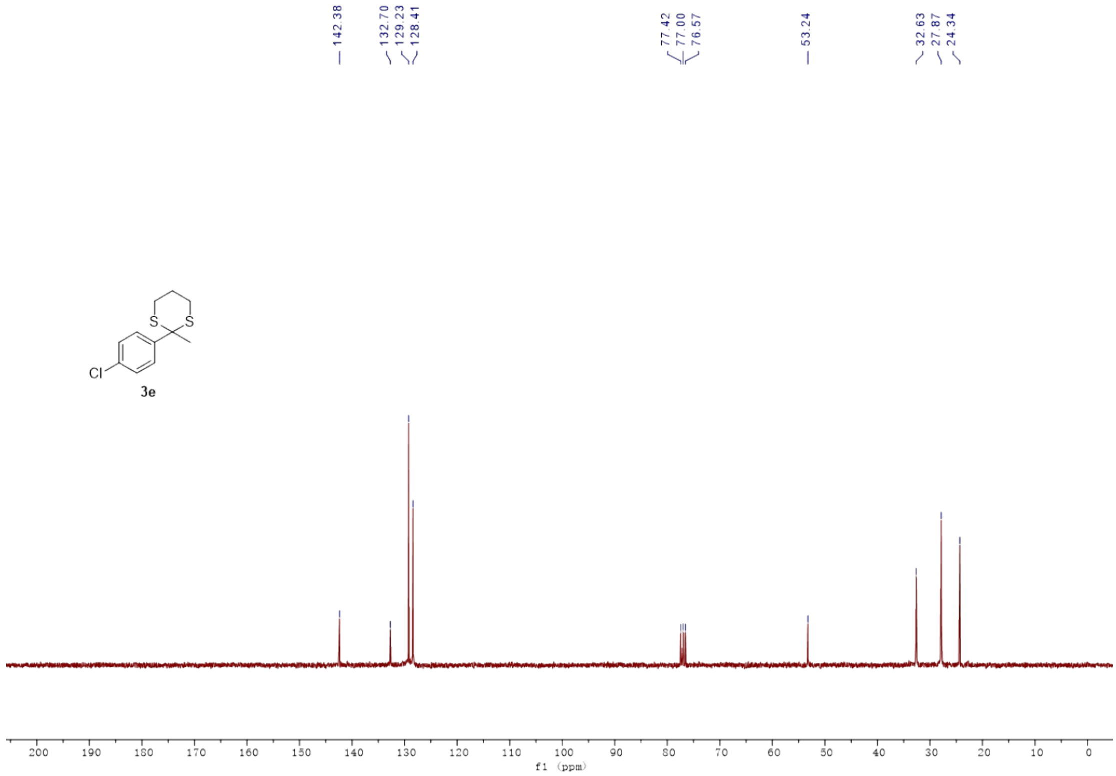
160.44
139.51
129.68
129.60
115.25
115.04
77.32
77.00
76.68
53.29
32.80
28.01
24.53
S
S
F
3d
f1 (ppm)


-116.01
F
S
S
f1 (ppm)

3d

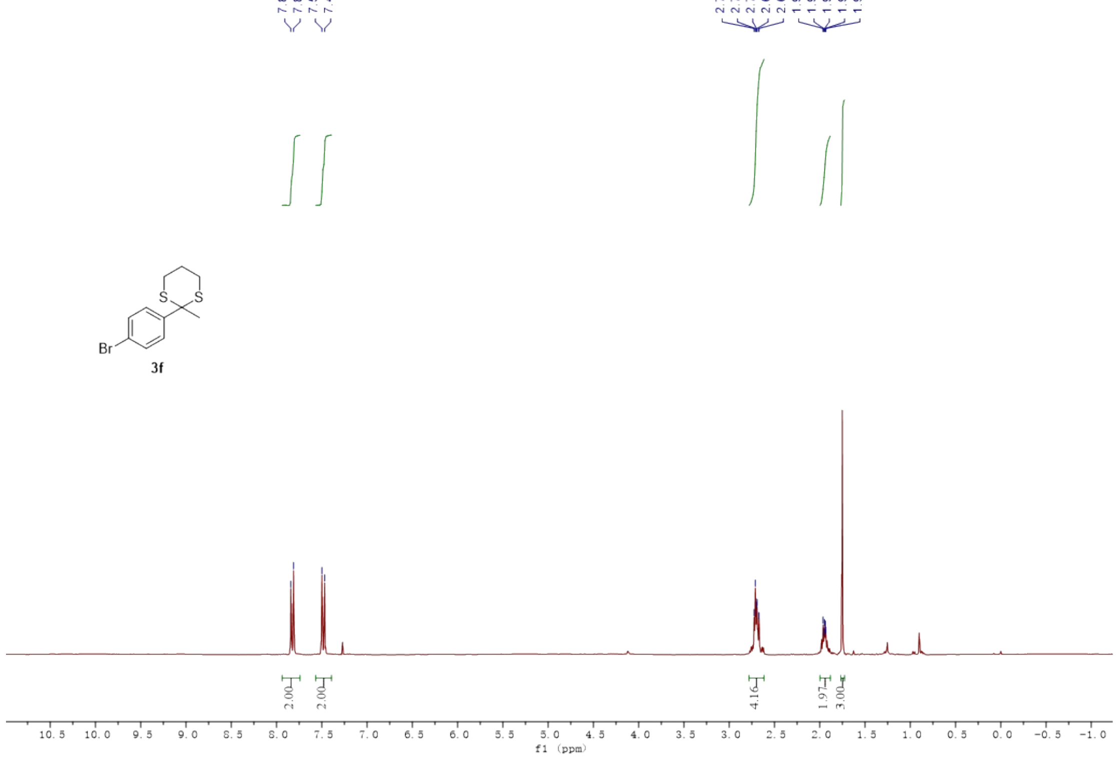

7.89
7.86
7.33
7.30
2.72
2.70
2.69
2.69
2.66
1.95
1.95
1.93
1.93
1.93
1.92
1.75
Cl
S
S
2.00
2.04
4.17
2.28
3.28
f1 (ppm)

3e



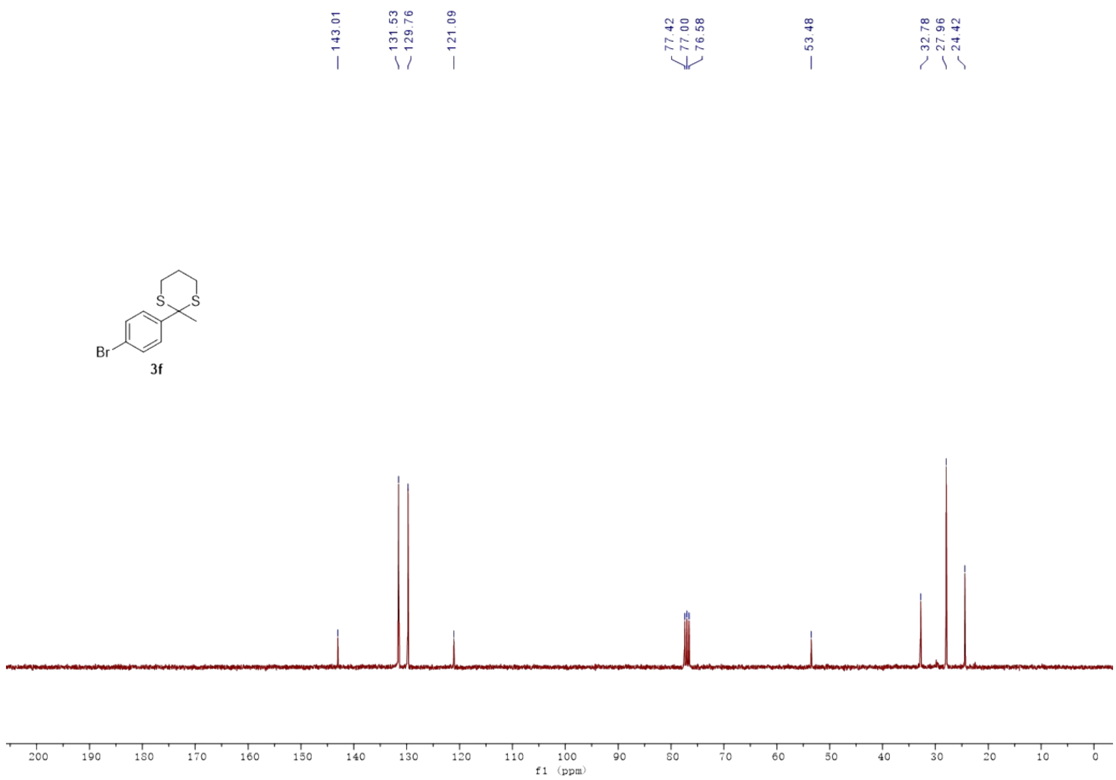
142.38
132.70
129.23
128.41
77.42
77.00
76.57
53.24
32.63
27.87
24.34
Cl
S
S
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

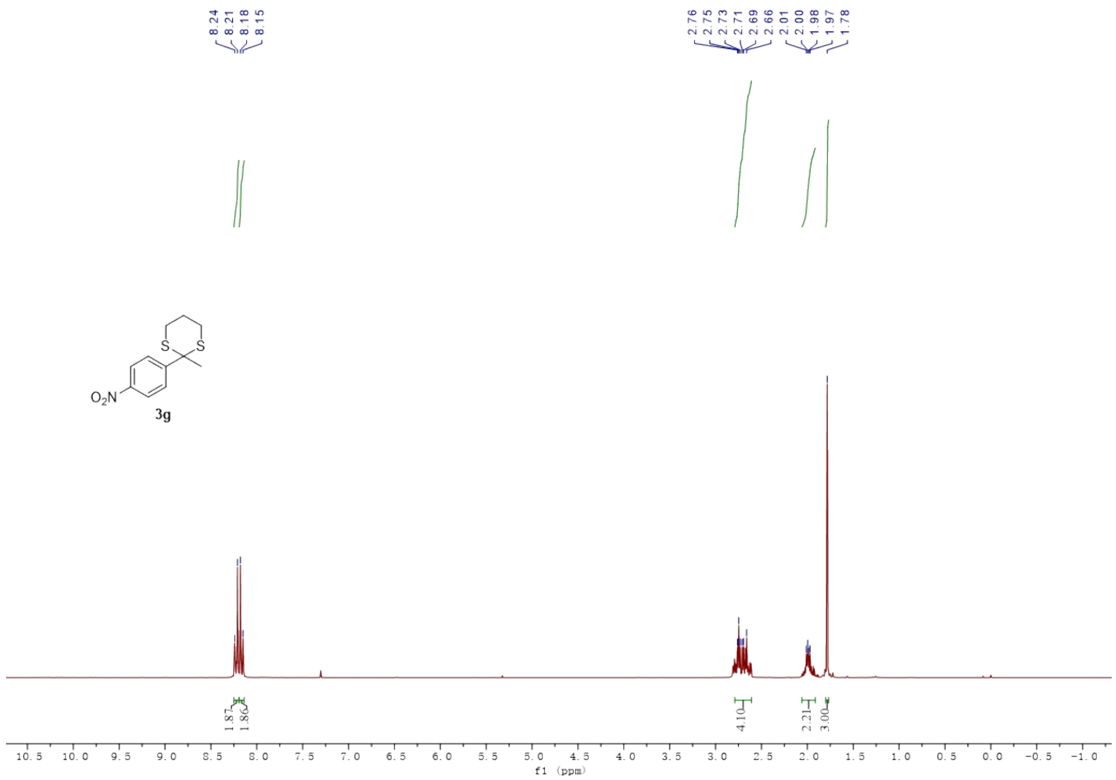
3e

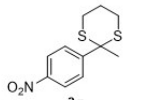


7.84
7.81
7.50
7.47
2.72
2.71
2.70
2.69
2.67
1.96
1.95
1.95
1.94
1.93
Br
S
S
2.00
2.00
4.16
1.97
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

3f

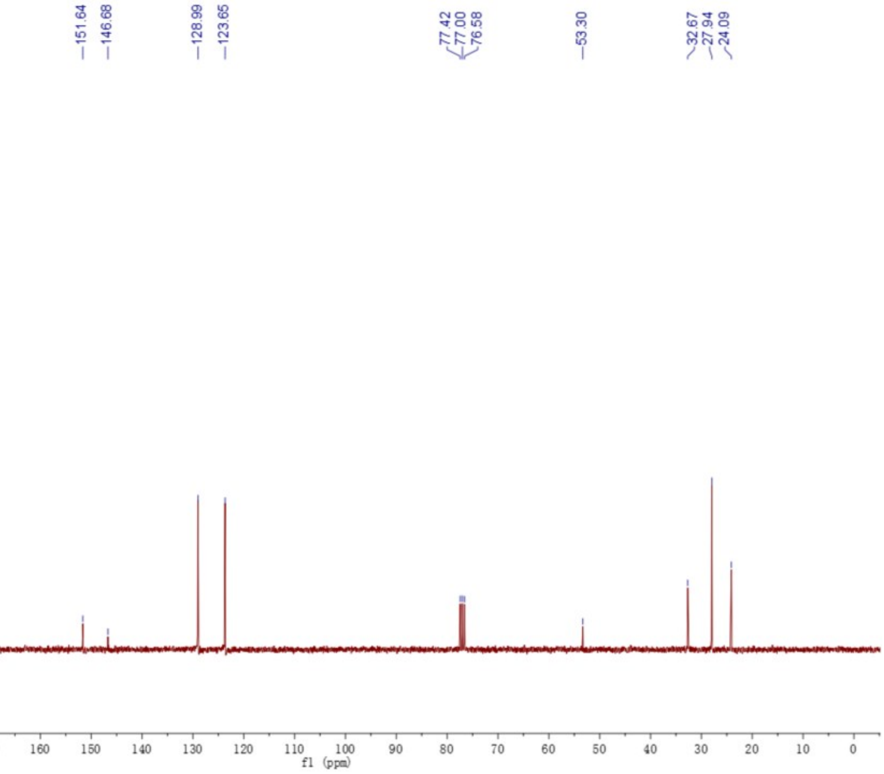

143.01
131.53
129.76
121.09
77.42
77.00
76.58
53.48
32.78
27.96
24.42
Br
S
S
3f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.24
8.21
8.18
8.15
2.76
2.75
2.73
2.71
2.69
2.66
2.01
2.00
1.98
1.97
1.78
O_2N
S
S
3g
1.87
1.86
4.10
2.21
3.00
10.5 10.0 9.5 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 -1.0
f1 (ppm)



O_2N
S
S

3g



151.64
146.68
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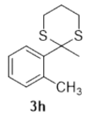
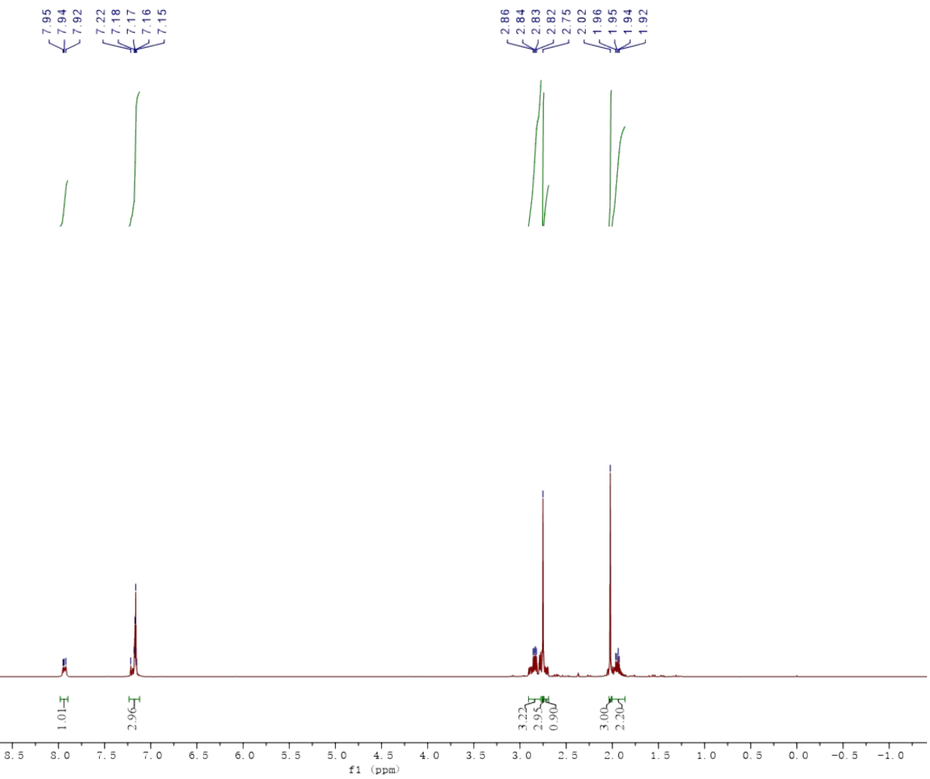
123.65
77.42
77.00
76.58
53.30
32.67
27.94
24.09
f1 (ppm)

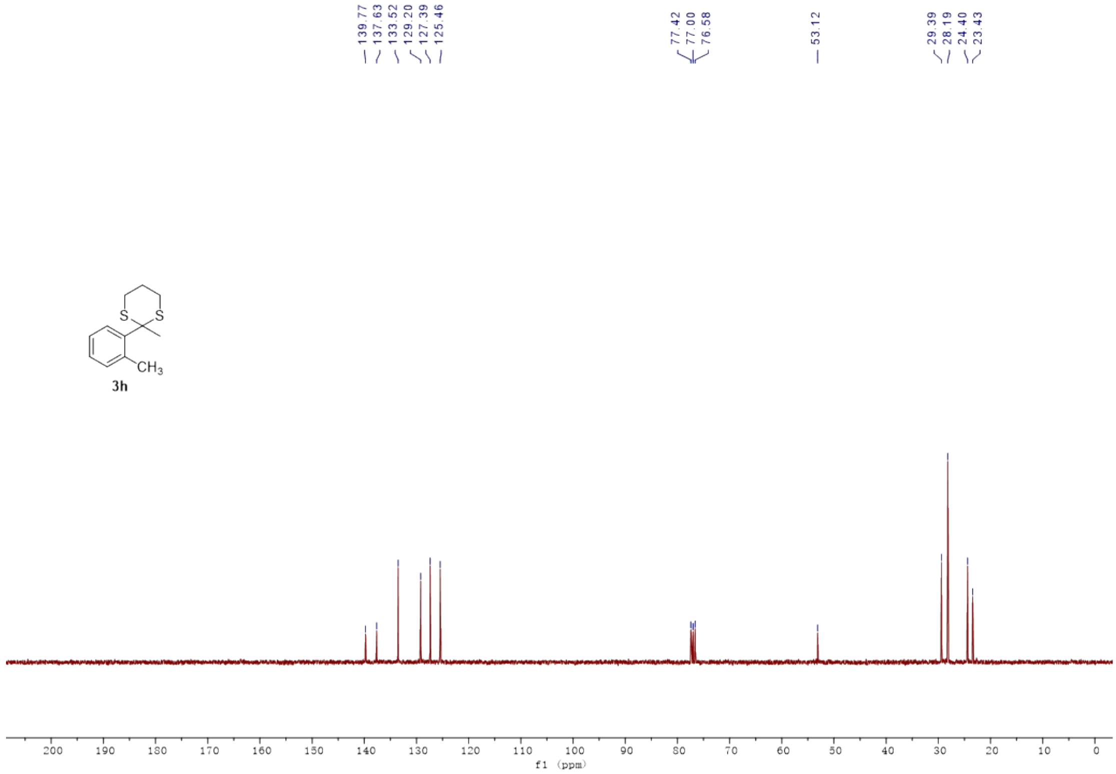


S
S
CH_3

3h



7.95
7.94
7.92
7.22
7.18
7.17
7.16
7.15
2.86
2.84
2.83
2.82
2.75
2.02
1.96
1.95
1.94
1.92
1.01
2.96
3.22
2.95
0.90
3.00
2.20
f1 (ppm)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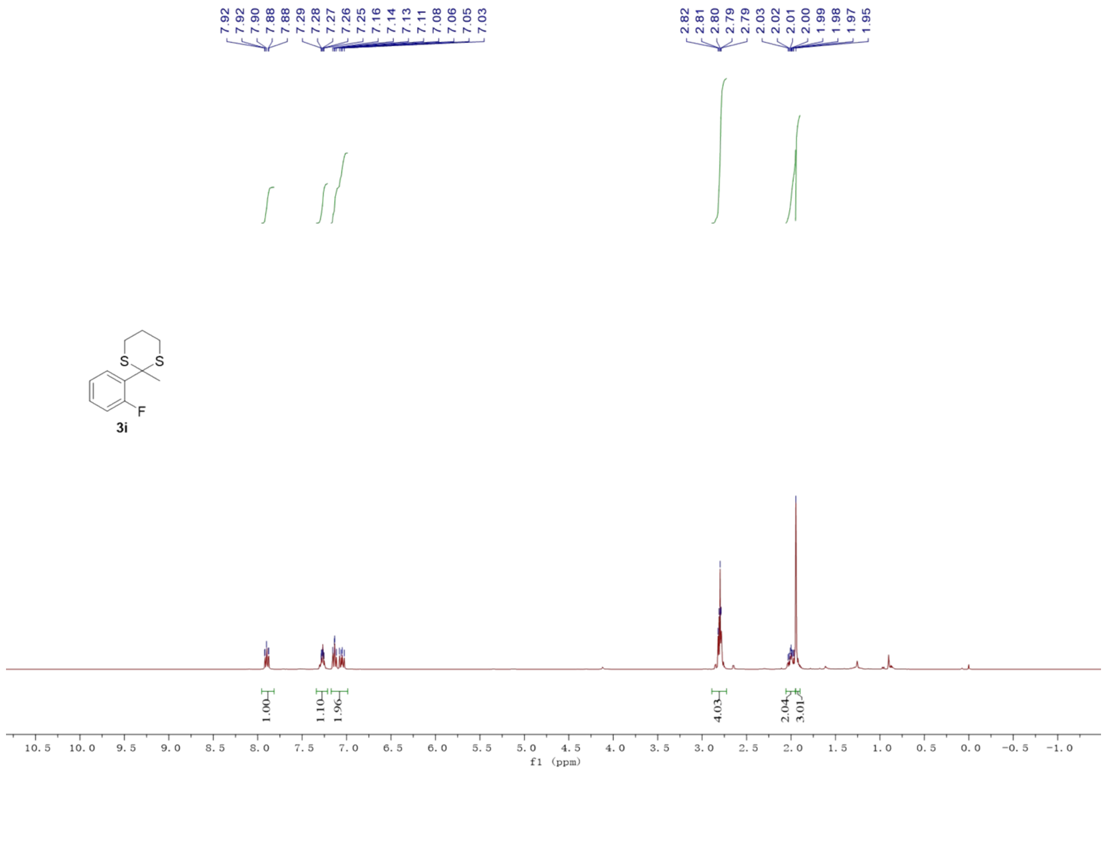
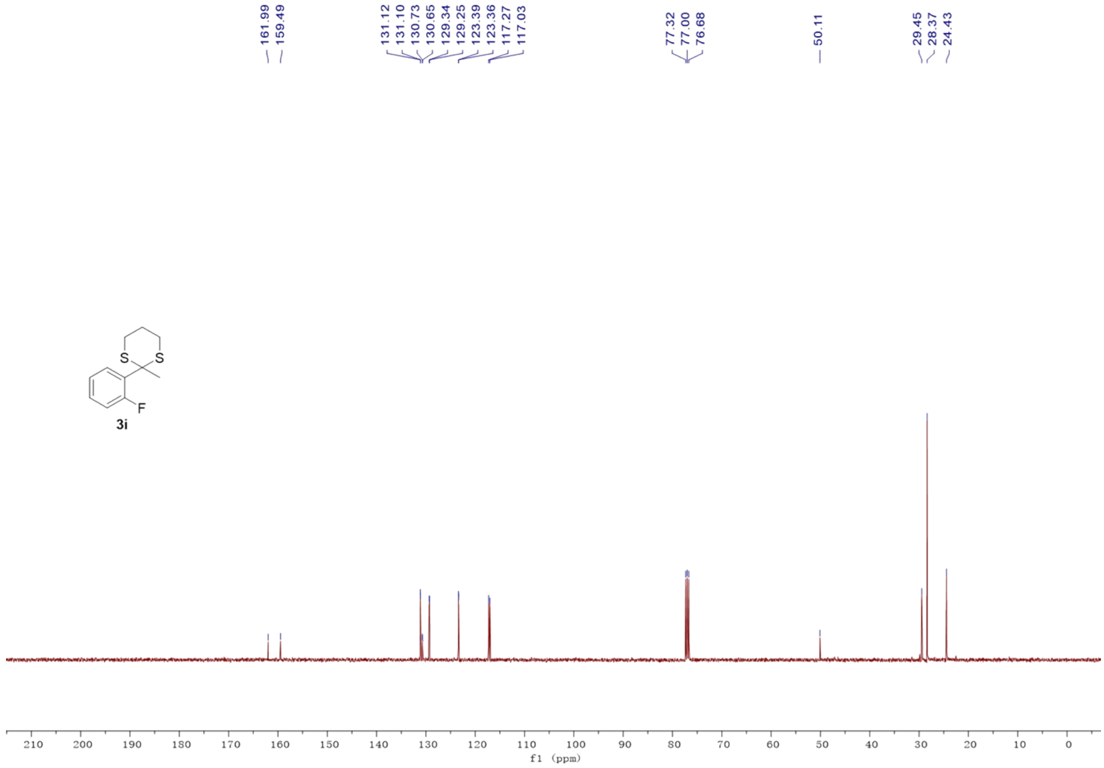
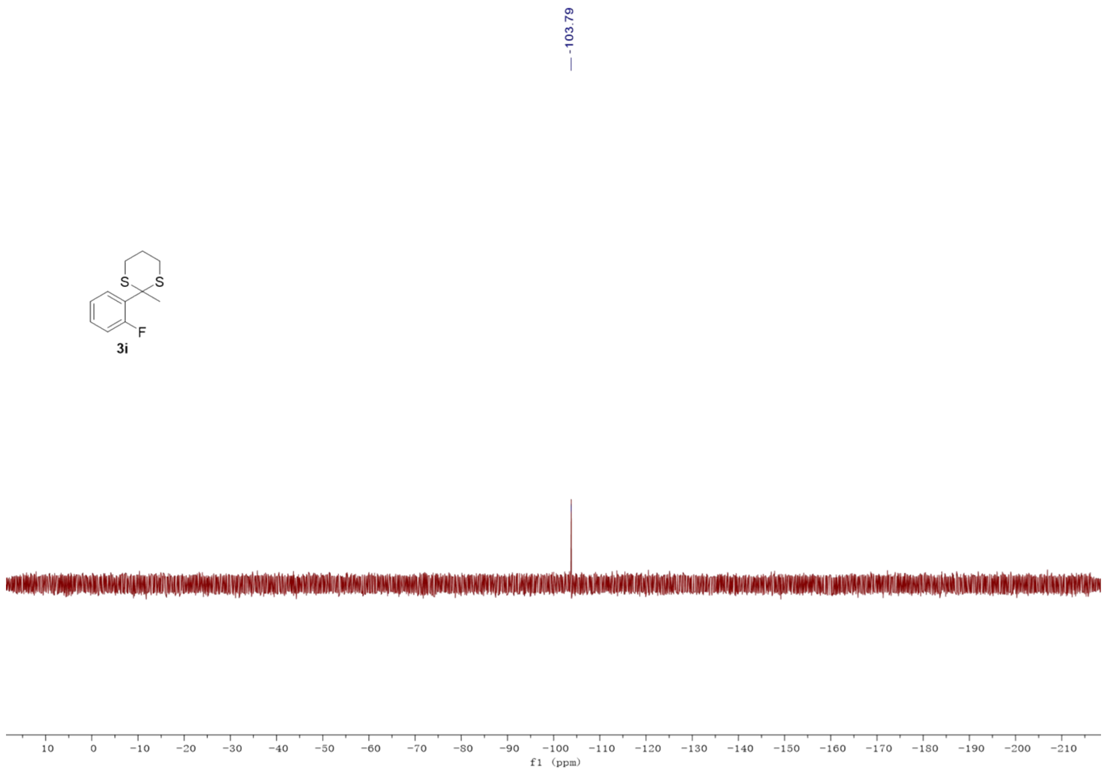
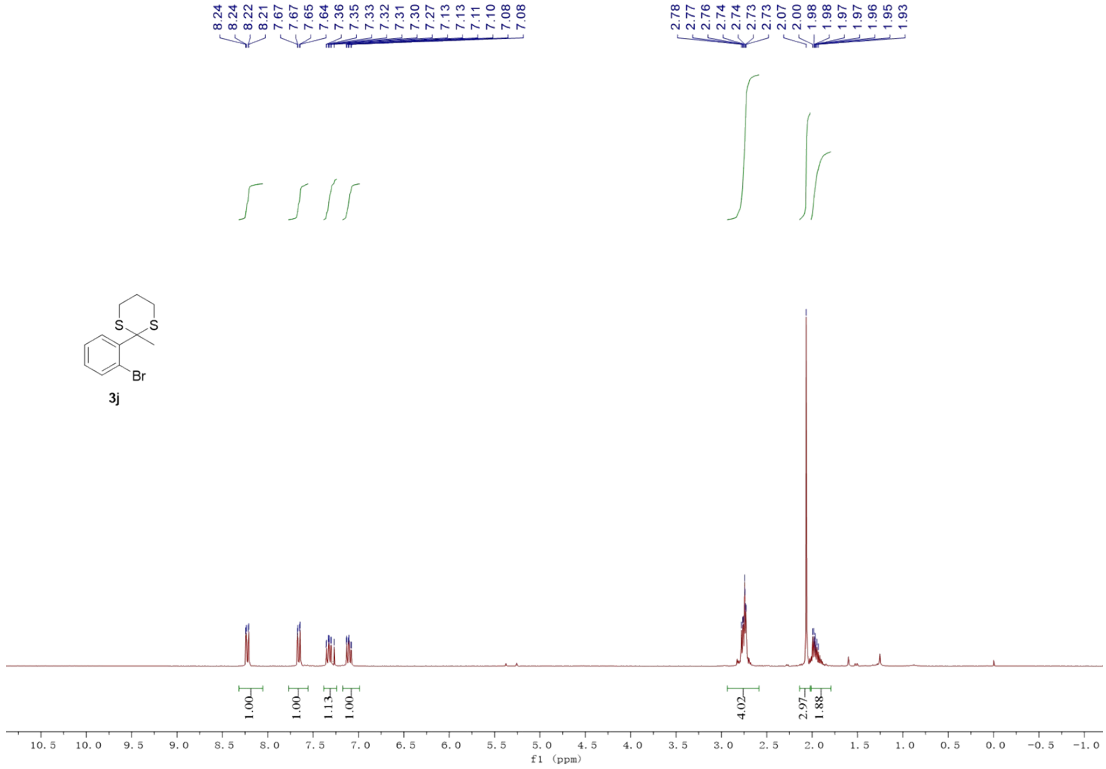
137.63
133.52
129.20
127.39
125.46
77.42
77.00
76.58
53.12
29.39
28.19
24.40
23.43
S S
CH3
3h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

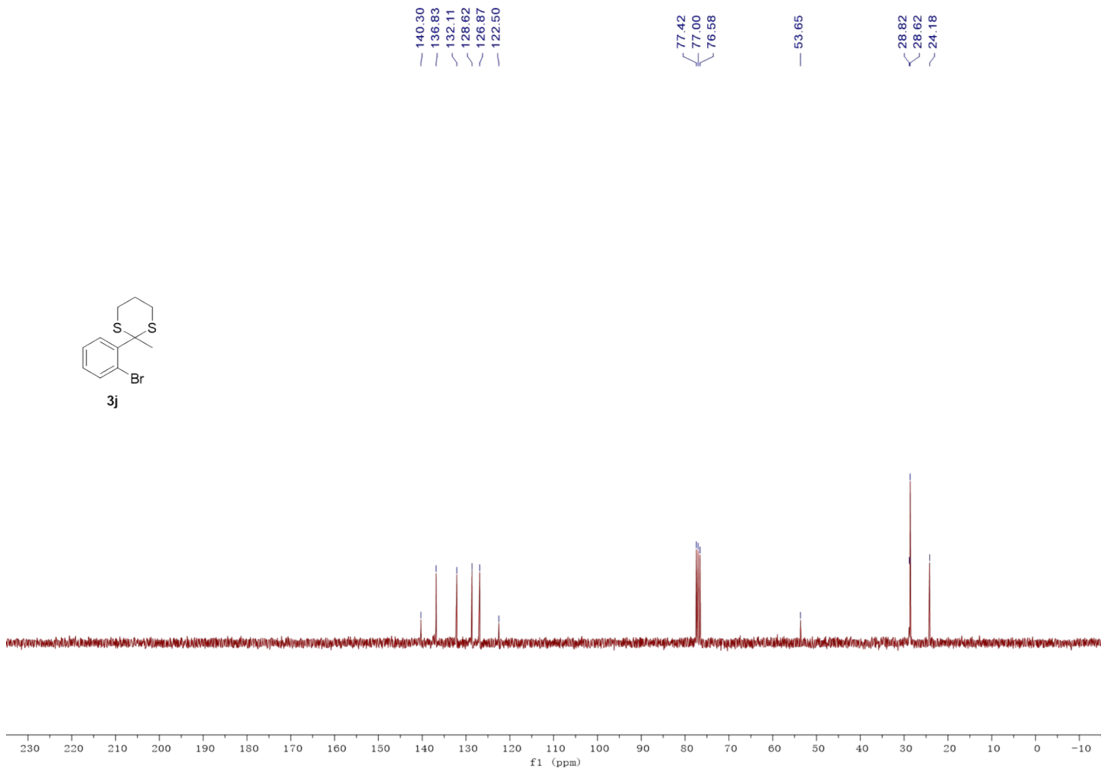

7.92
7.92
7.90
7.88
7.88
7.29
7.28
7.27
7.26
7.25
7.16
7.14
7.13
7.11
7.08
7.06
7.05
7.03
2.82
2.81
2.80
2.79
2.79
2.03
2.02
2.01
2.00
1.99
1.98
1.97
1.95
S S
F
3i
1.00
1.10
1.96
4.03
2.04
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


161.99
159.49
131.12
131.10
130.73
130.65
129.34
129.25
123.39
123.36
117.27
117.03
77.32
77.00
76.68
50.11
29.45
28.37
24.43
S
S
F
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

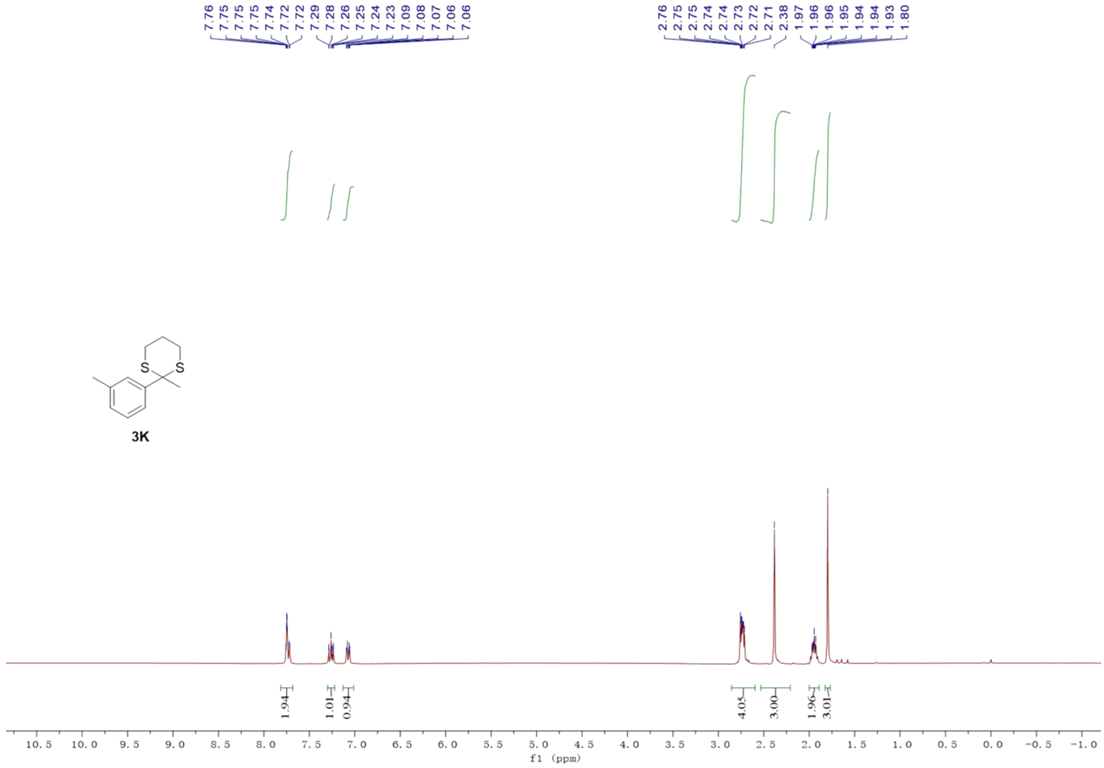

-103.79
S
S
F
3i
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)



3j



3j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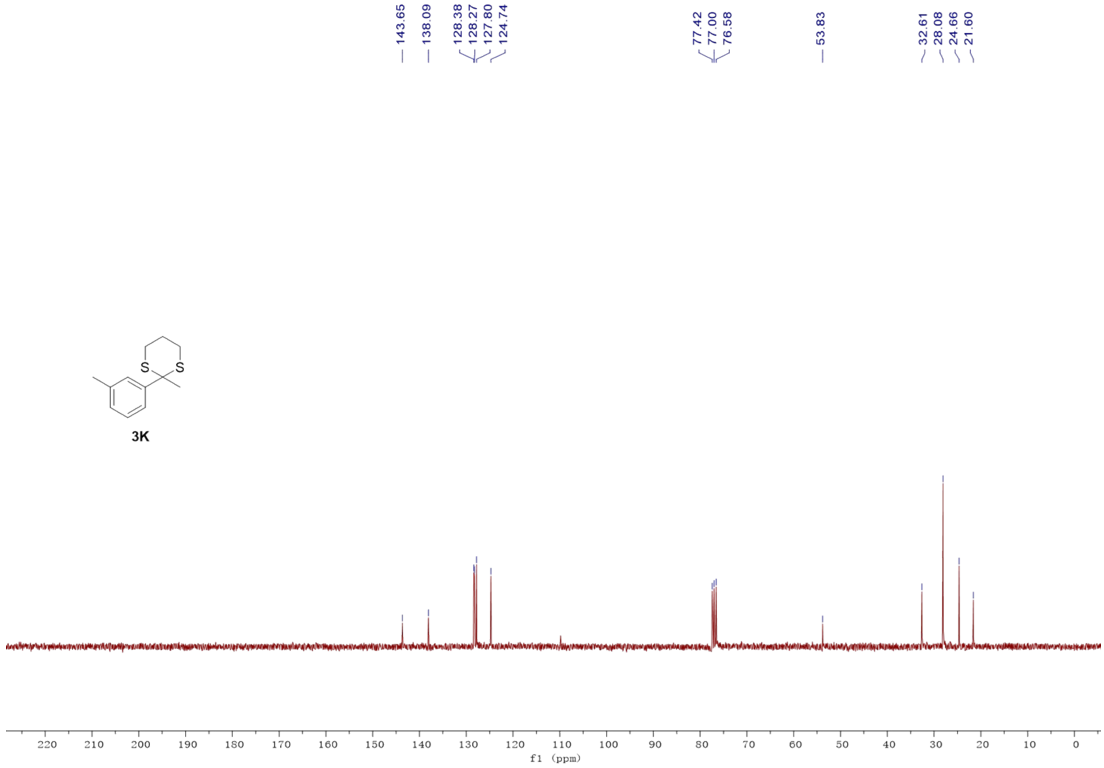
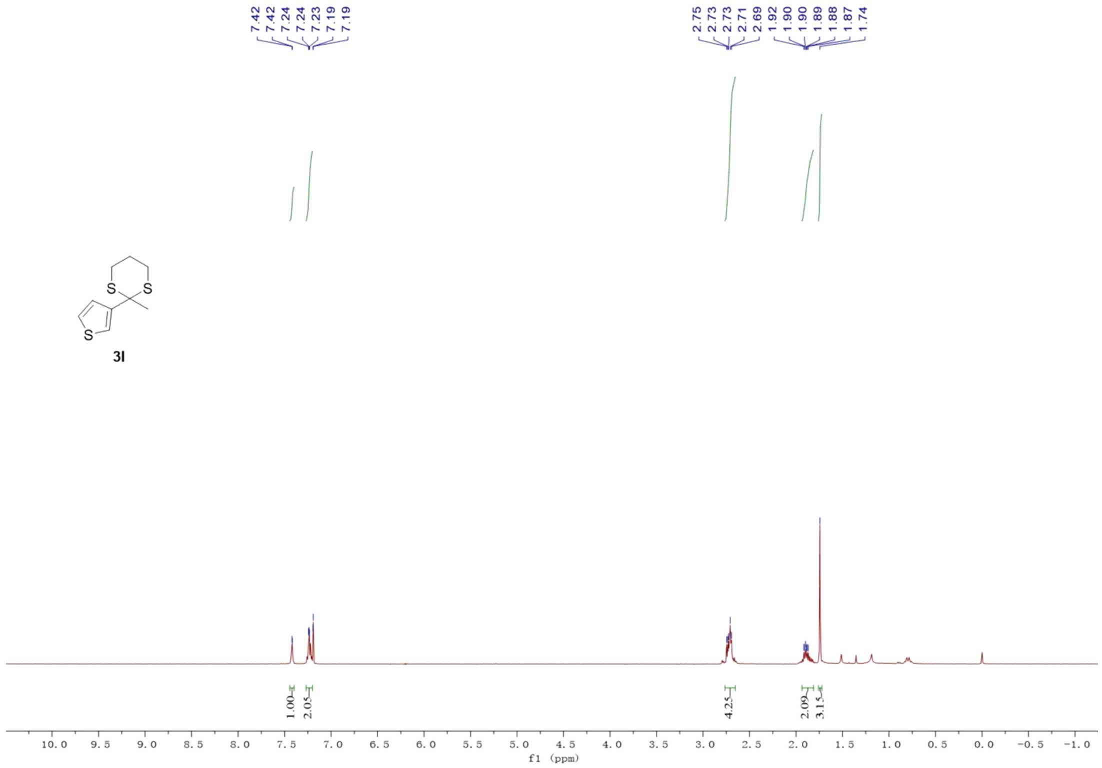
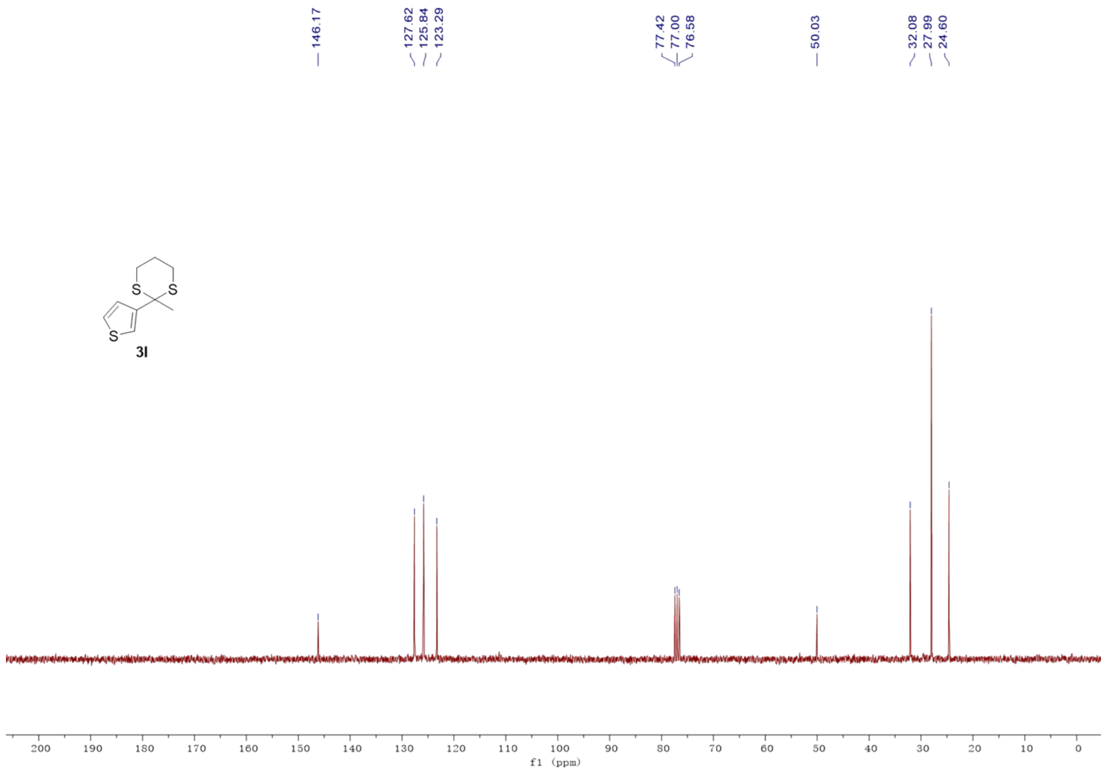
2.76 2.75 2.75 2.74 2.74 2.73 2.72 2.71 2.38 1.97 1.96 1.96 1.95 1.94 1.94 1.93 1.80
S
S
3K
1.94
1.01
0.94
4.03
3.00
1.96
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


143.65
138.09
128.38 128.27 127.80 124.74
77.42 77.00 76.58
53.83
32.61 28.08 24.66 21.60
S
S
3K
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.42
7.42
7.24
7.24
7.23
7.19
7.19
2.75
2.73
2.73
2.71
2.69
1.92
1.90
1.90
1.89
1.88
1.87
1.74
3l
1.00
2.05
4.25
2.09
3.15
f1 (ppm)


146.17
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123.29
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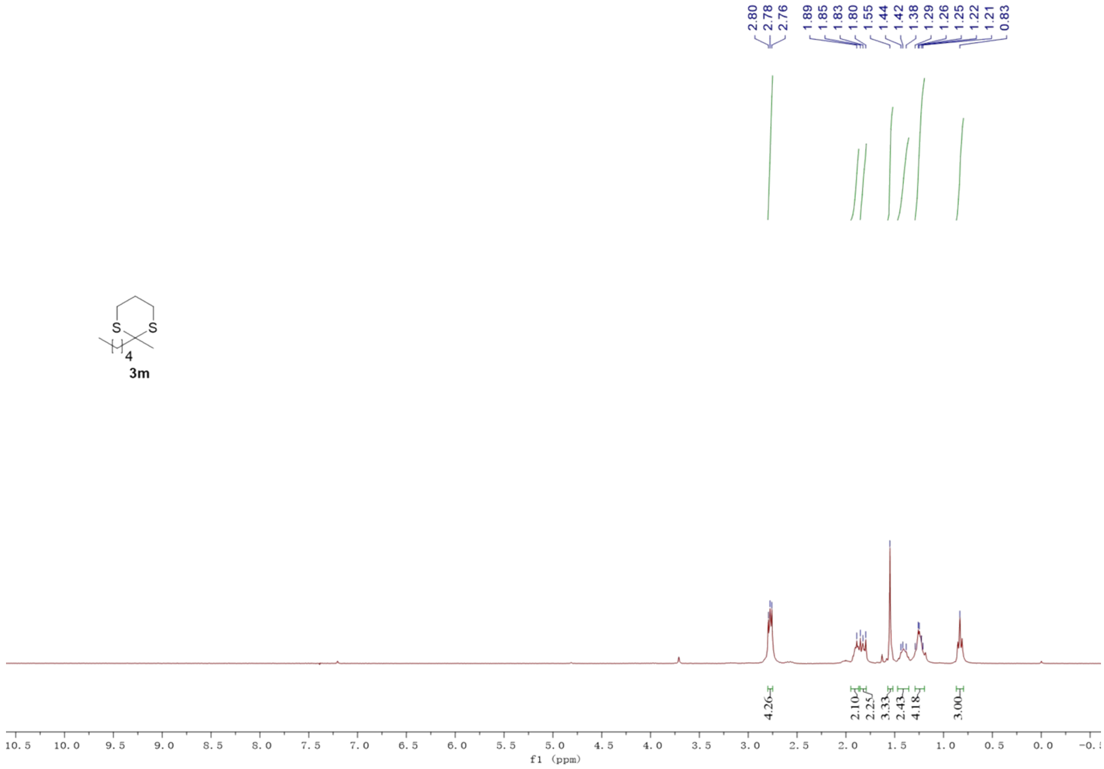
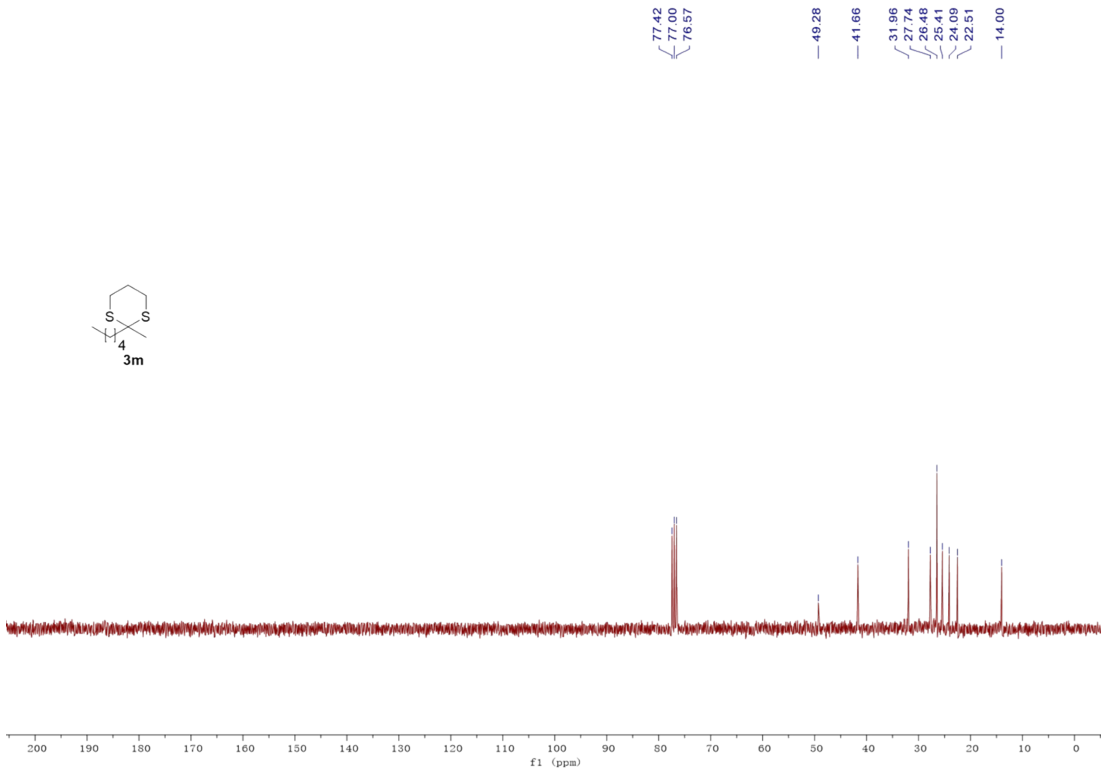
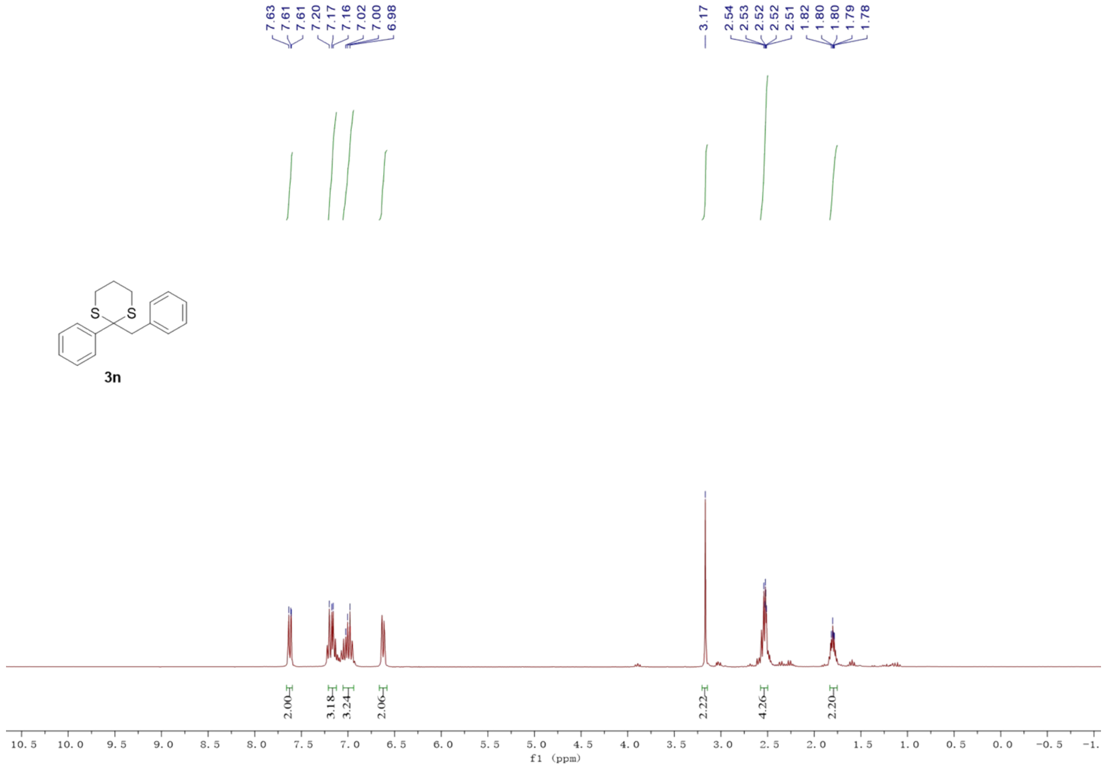
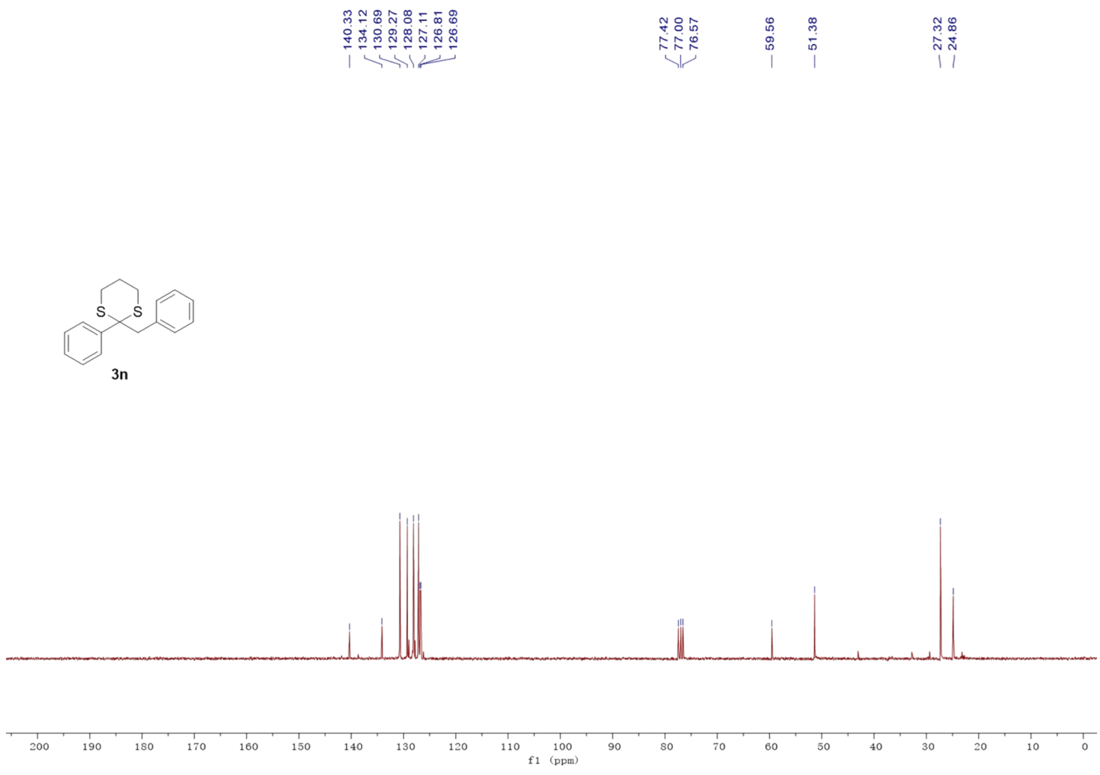
77.00
76.58
50.03
32.08
27.99
24.60
3l
f1 (ppm)


2.80
2.78
2.76
1.89
1.85
1.83
1.80
1.55
1.44
1.42
1.38
1.29
1.26
1.25
1.22
1.21
0.83
S
S
4
3m
4.26
2.10
2.25
3.33
2.43
4.18
3.00
10.5 10.0 9.5 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)

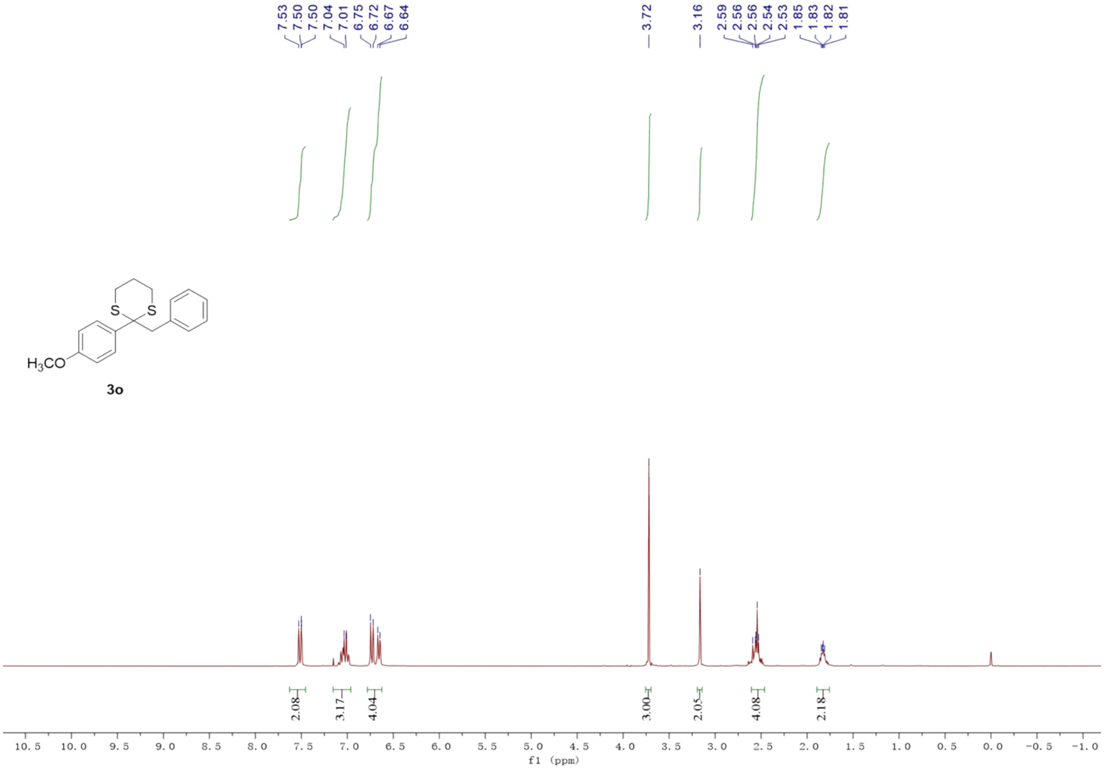

77.42
77.00
76.57
49.28
41.66
31.96
27.74
26.48
25.41
24.09
22.51
14.00
S
S
4
3m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

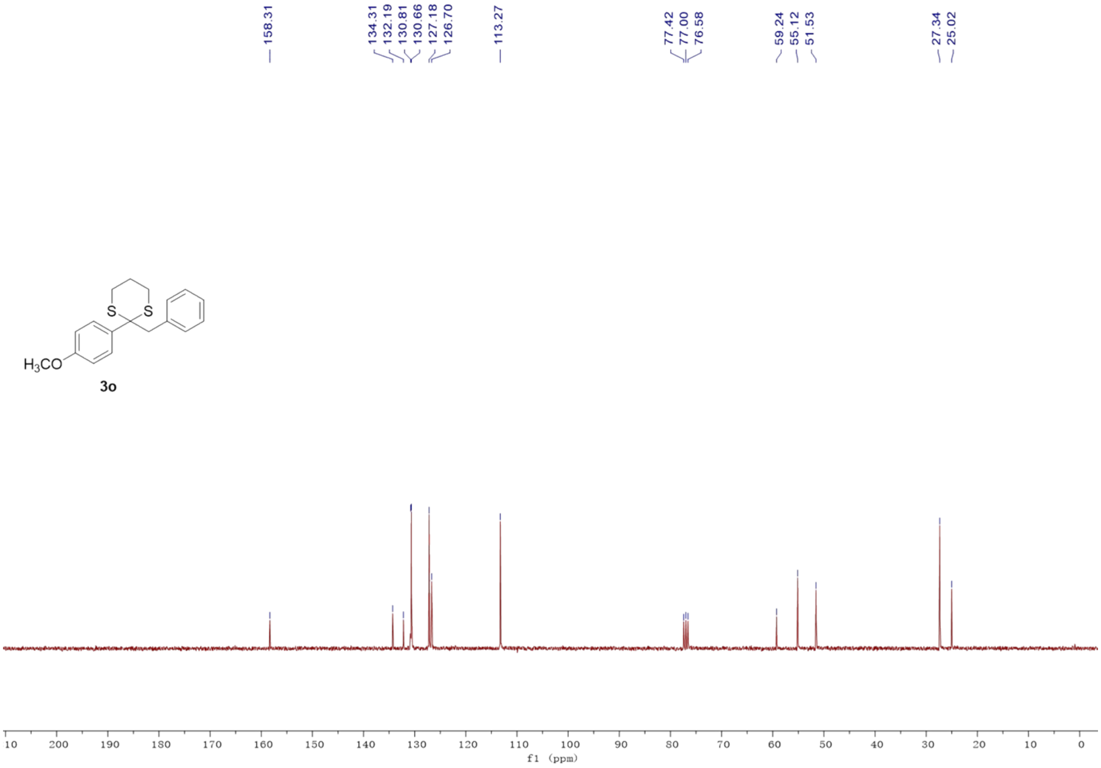


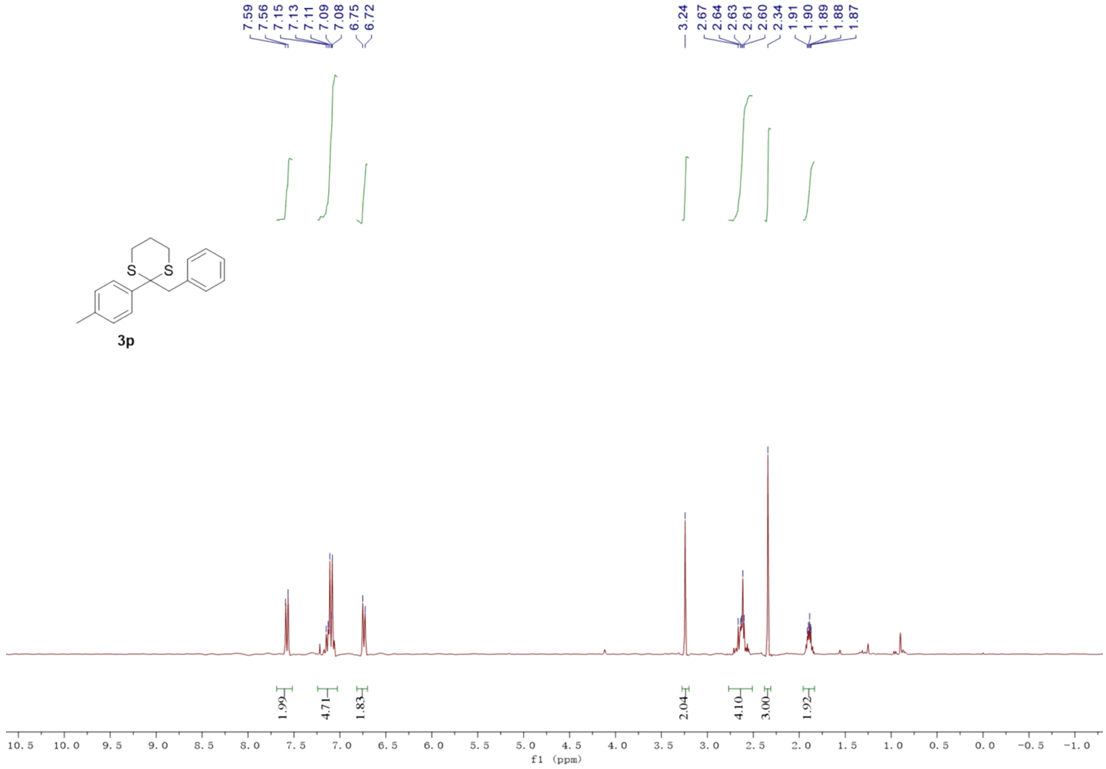
7.63
7.61
7.61
7.20
7.17
7.16
7.02
7.00
6.98
3.17
2.54
2.53
2.52
2.52
2.51
1.82
1.80
1.80
1.79
1.78
S
S
3n
2.00
3.18
3.24
2.06
2.22
4.26
2.20
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)



140.33
134.12
130.69
129.27
128.08
127.11
126.81
126.69
77.42
77.00
76.57
59.56
51.38
27.32
24.86
S
S
3n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


H3CO
3o
f1 (ppm)


H3CO
3o
f1 (ppm)


7.59
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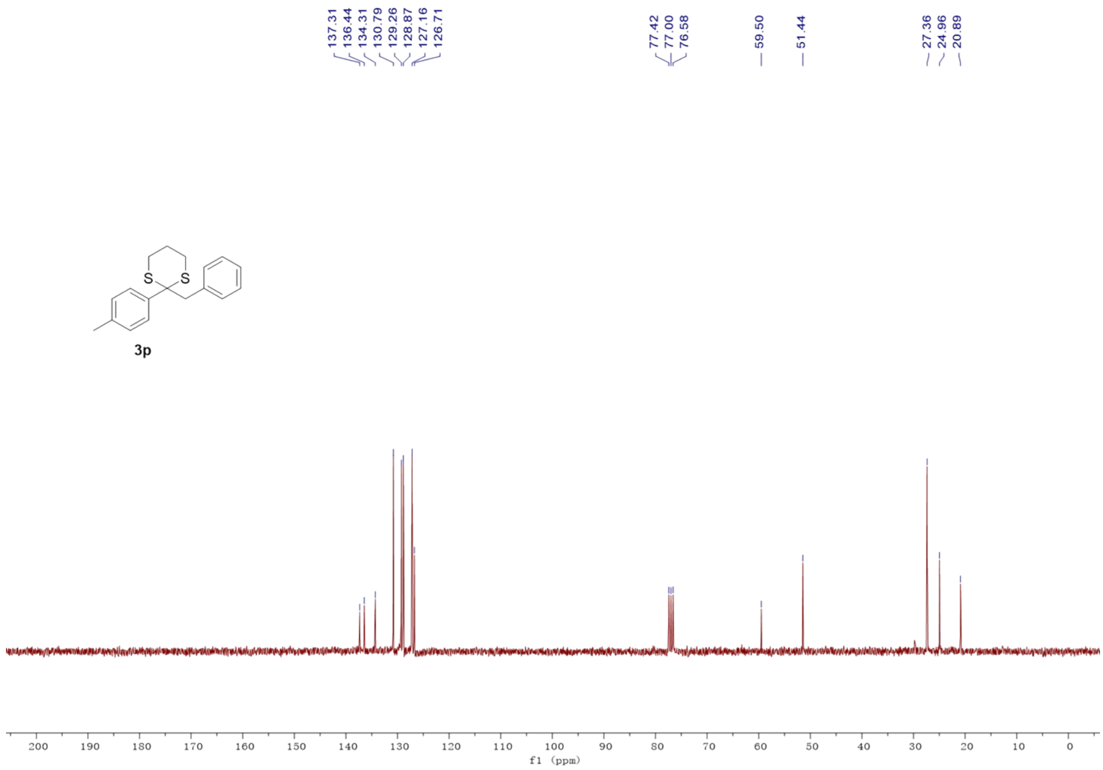
7.11
7.09
7.08
6.75
6.72
3.24
2.67
2.64
2.63
2.61
2.60
2.34
1.91
1.90
1.89
1.88
1.87
3p
1.99
4.71
1.83
2.04
4.10
3.00
1.92
f1 (ppm)


137.31
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130.79
129.26
128.87
127.16
126.71
77.42
77.00
76.58
59.50
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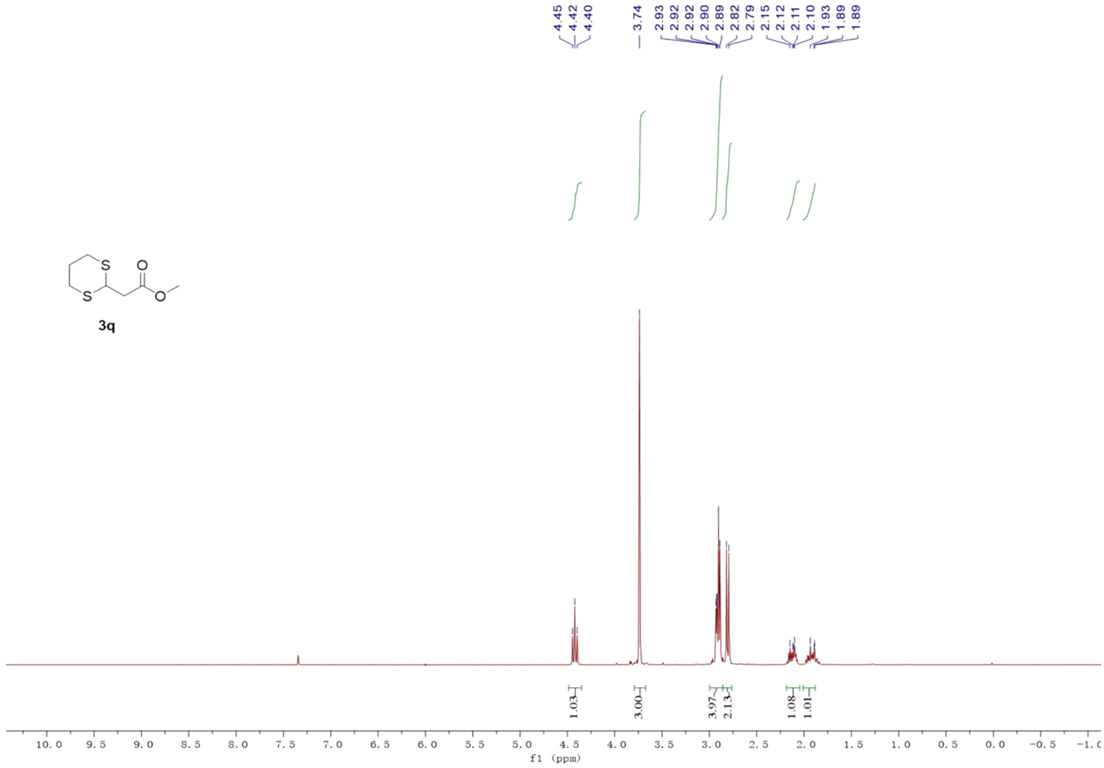
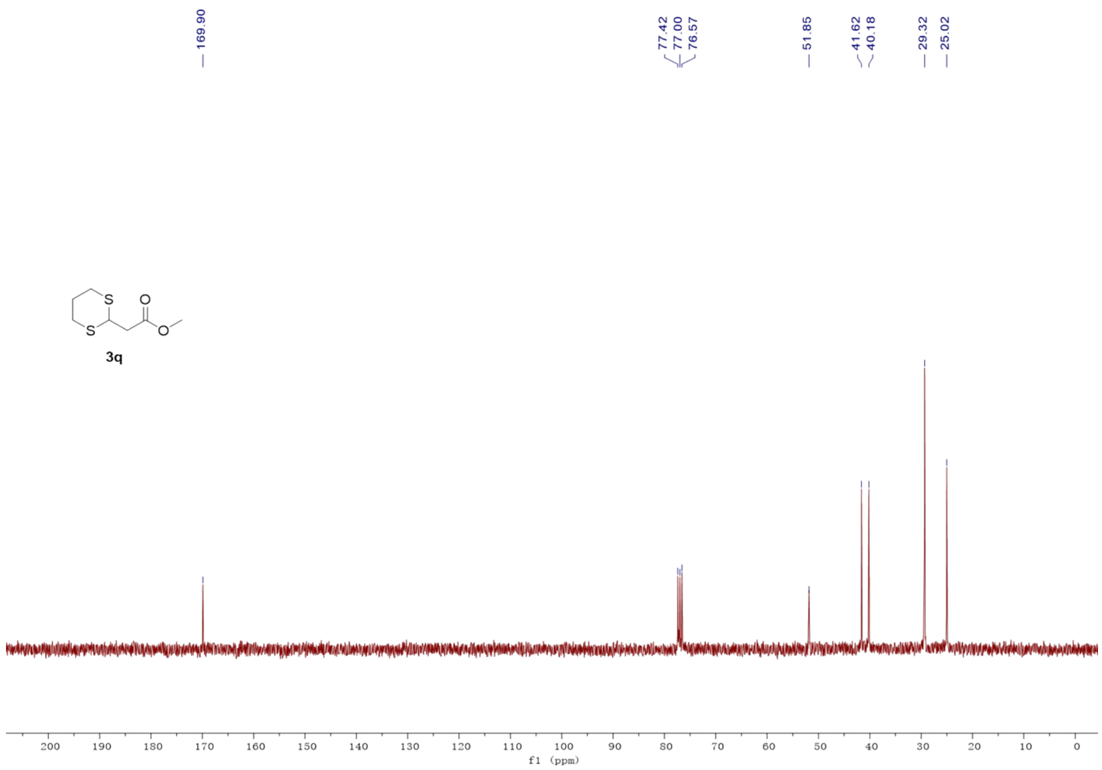
20.89
3p
f1 (ppm)



3q

4.45
4.42
4.40
3.74
2.93
2.92
2.92
2.90
2.89
2.82
2.79
2.15
2.12
2.11
2.10
1.93
1.89
1.89
1.03
3.00
3.97
2.13
1.08
1.01
f1 (ppm)



3q

169.90
77.42
77.00
76.57
51.85
41.62
40.18
29.32
25.02
f1 (ppm)