Supporting Information

Electrochemically enabled synthesis of oxathiazinane via a radical cyclization cascade

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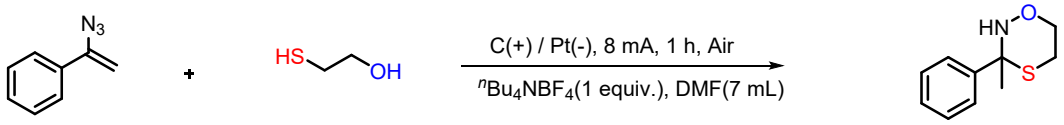
1. General methods

Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification. Column chromatography on silica gel (300-400 mesh) was carried out using technical grade 60-90 °C petroleum ether and analytical grade EtOAc (without further purification). ^{1}H and ^{13}C and ^{19}F spectra were recorded on a 400 MHz or 600MHz spectrometer. Chemical shifts were reported in ppm. ^{1}H and ^{19}F NMR spectra were referenced to $CDCl_3$ (7.26 ppm) or DMSO (2.5 ppm) or MeOD (4.87 ppm), and ^{13}C-NMR spectra were referenced to $CDCl_3$ (77.0 ppm) or DMSO (39.5 ppm) or MeOD (49.0 ppm). Peak multiplicities were designated by the following abbreviations: s, singlet; d, doublet; t, triplet; m, multiplet; brs, broad singlet and J, coupling constant in Hz. The HRMS spectrum was measured by micromass $QTOF_2$ Quadrupole/Time of Flight Tandem mass spectrometer with electron spray ionization. Potentiostat was purchased from Shanghai Xinrui Companyand the model is DJS-292B.

2. Supplementary experiments

Table S1 Screening of Reaction Conditions[a]



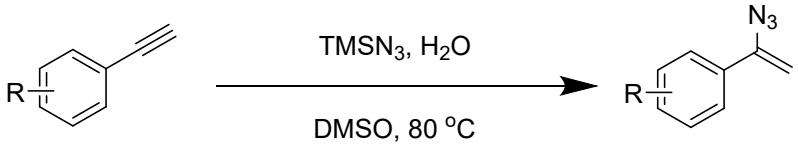
1a **2a** **3a**

Entry	Electrode	Current (mA)	additive	Solvent	time	Yield (%)[b]
1	C(+)|Pt(-)	5 mA	$TBABF_4$	MeCN	3 h	51%
2	C(+)|C(-)	5 mA	$TBABF_4$	MeCN	3 h	Trace
3	Pt(+)|Pt(-)	5 mA	$TBABF_4$	MeCN	3 h	Trace
4	C(+)|Pt(-)	7 mA	$TBABF_4$	MeCN	3 h	45%
5	C(+)|Pt(-)	3 mA	$TBABF_4$	MeCN	3 h	17%
6	C(+)|Pt(-)	5 mA	$TBABF_4$	MeCN	2 h	36%
7	C(+)|Pt(-)	5 mA	$TBABF_4$	MeCN	4 h	44%
8	C(+)|Pt(-)	5 mA	$TBABF_4$	DCM	3 h	nd
9	C(+)|Pt(-)	5 mA	$TBABF_4$	THF	3 h	Trace
10	C(+)|C(-)	5 mA	$TBABF_4$	DMSO	3 h	65%
11	C(+)|C(-)	5 mA	$TBABF_4$	MeOH	2 h	31%
12	C(+)|C(-)	5 mA	$TBABF_4$	DMF	3 h	77%

13	**C(+)|Pt(-)**	**8 mA**	**$TBABF_4$**	**DMF**	**1 h**	**87%**
14	C(+)|Pt(-)	8 mA	TBAI	DMF	1 h	70%
15	C(+)|Pt(-)	8 mA	TBAP	DMF	1 h	56%
16	C(+)|Pt(-)	8 mA	TBAHB	MeOH	1 h	nd
17	C(+)|Pt(-)	8 mA	n-Bu_4PF_6	DMF	1 h	51%
18	C(+)|Pt(-)	8 mA	n-Bu_4ClO_4N	DMF	1 h	41%
19	RVC(+)|RVC(-)	8 mA	TBABF4	DMF	1 h	Trace
20	RVC(+)|Pt(-)	8 mA	TBABF4	DMF	1 h	64%

[a]Reaction conditions: **1a** (0.4 mmol), **2a** (1.4 mmol), DMF as solvent (7 mL), electrolysis at a constant current of 8 mA for 1 h in an undivided cell. [b] Isolated yield.

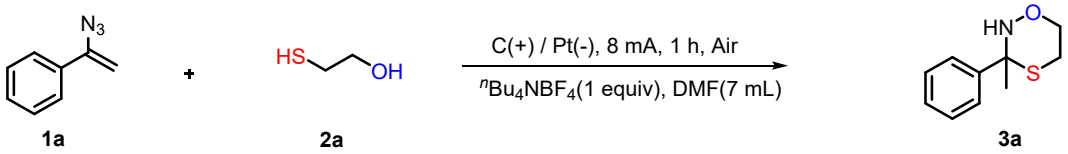
3. Synthesis of substrates



To a solution of $TMSN_3$ and $AgCO_3$ in DMSO (20 mL), alkyne and H_2O was added in a 50 mL round-bottomed flask. Then the mixture was stirred at 80 °C for 90-120 minutes.[1] After completion (evidenced by TLC), the reaction is cooled to room temperature and the solution is transferred to a beaker containing dichloromethane and extracted with water. The resulting mixture was extraction by dichloromethane. The organic layer was combined, washed with brine, and dried over $MgSO_4$. The organic phase after drying is concentrated under reduced pressure and the residue is purified by column chromatography on silica gel, with petroleum ether as eluent to provide **1a-1t.**

4. General procedure for the preparation of products

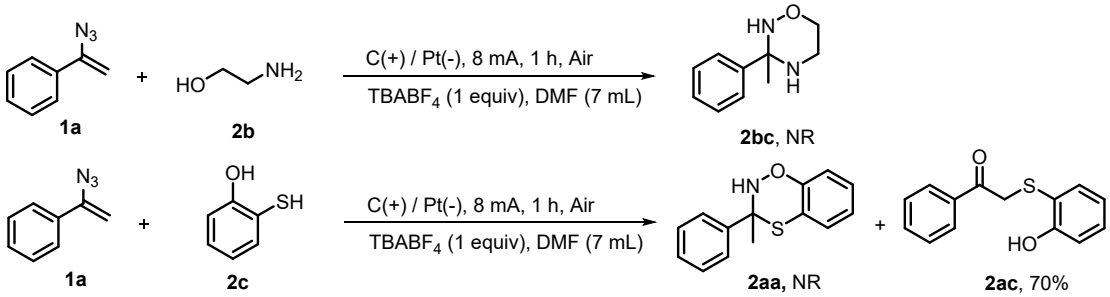
Electrochemical synthesis of compounds **3a-3t**



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

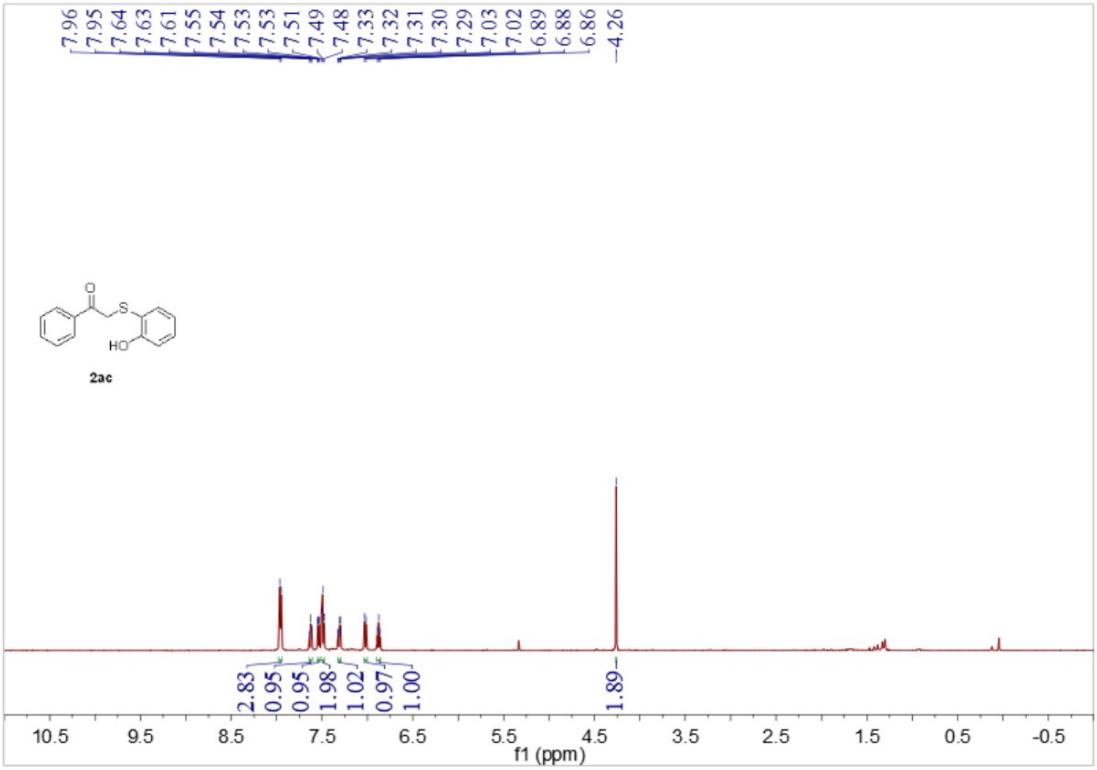
A 10 ml three-necked round-bottomed flask was charged sequentially with **1a** (0.4 mmol, 1.0 eq), **2a** (1.4 mmol, 3.5 eq) and $TBABF_4$ (0.4 mmol). The electrolytic cell was equipped with a graphite rod anode (Φ6 mm) and a platinum plate cathode (1 cm × 1 cm). The three-neck flask was then filled with DMF (7 mL). Electrolysis was carried out at room temperature at a constant current of 8 mA for the appropriate time. At the end of the reaction, the reaction mixture was washed with water and extracted with dichloromethane (3 × 10 mL). The organic layers were combined, dried with Na_2SO_4 and concentrated. Flash column chromatography on silica gel gave the pure product .

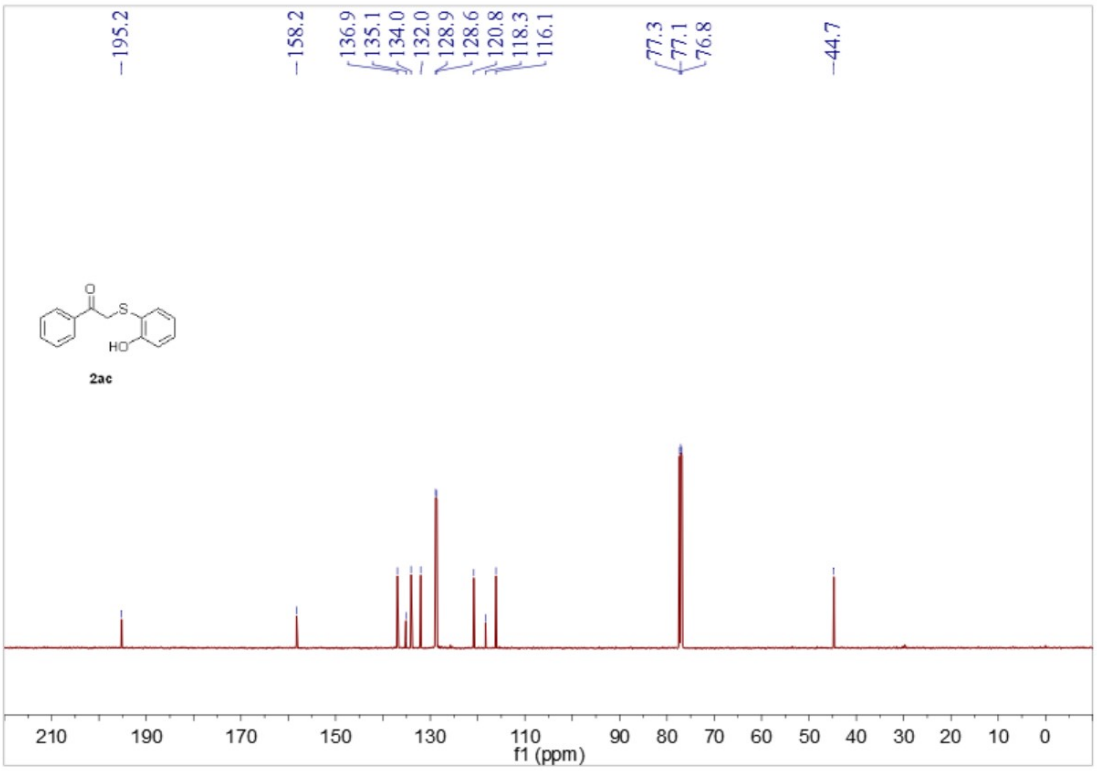
Other attempts with other types of double nucleophilic reagents



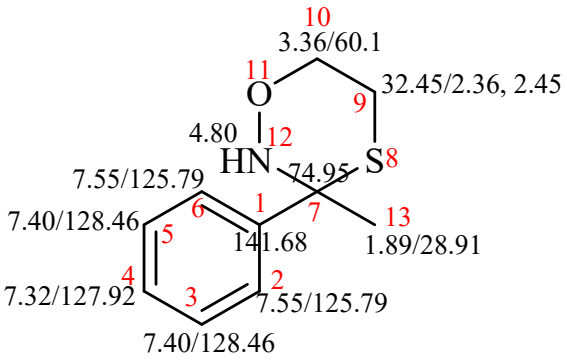
2-((2-hydroxyphenyl)thio)-1-phenylethan-1-one (2ac). The title compound (68.3 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 70% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96-7.95 (m, 3H), 7.64-7.61 (m, 1H), δ 7.55-7.53 (m, 1H), δ 7.51-7.48 (m, 2H), δ 7.33-7.29 (m, 1H), δ 7.03-7.02 (m, 1H), δ 6.89-6.86 (m, 1H), 4.26 (s, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 195.2, 158.2, 136.9, 135.1, 134.0, 132.0, 128.9, 128.6, 120.8, 118.3, 116.1, 44.7. **HRMS (m/z) (ESI)**: calcd for $C_{14}H_{13}O_2S^+$ $[M+H]^+$ 245.0631, found 245.0633.





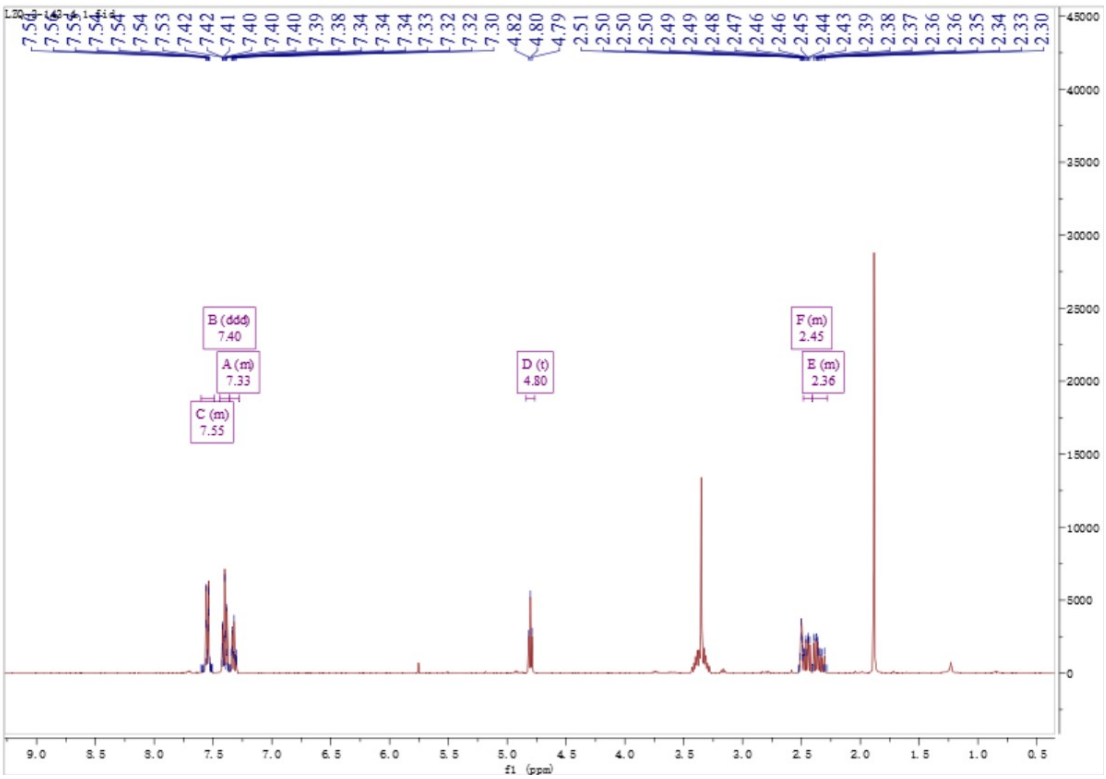
5. Structural analysis of substrate 3a

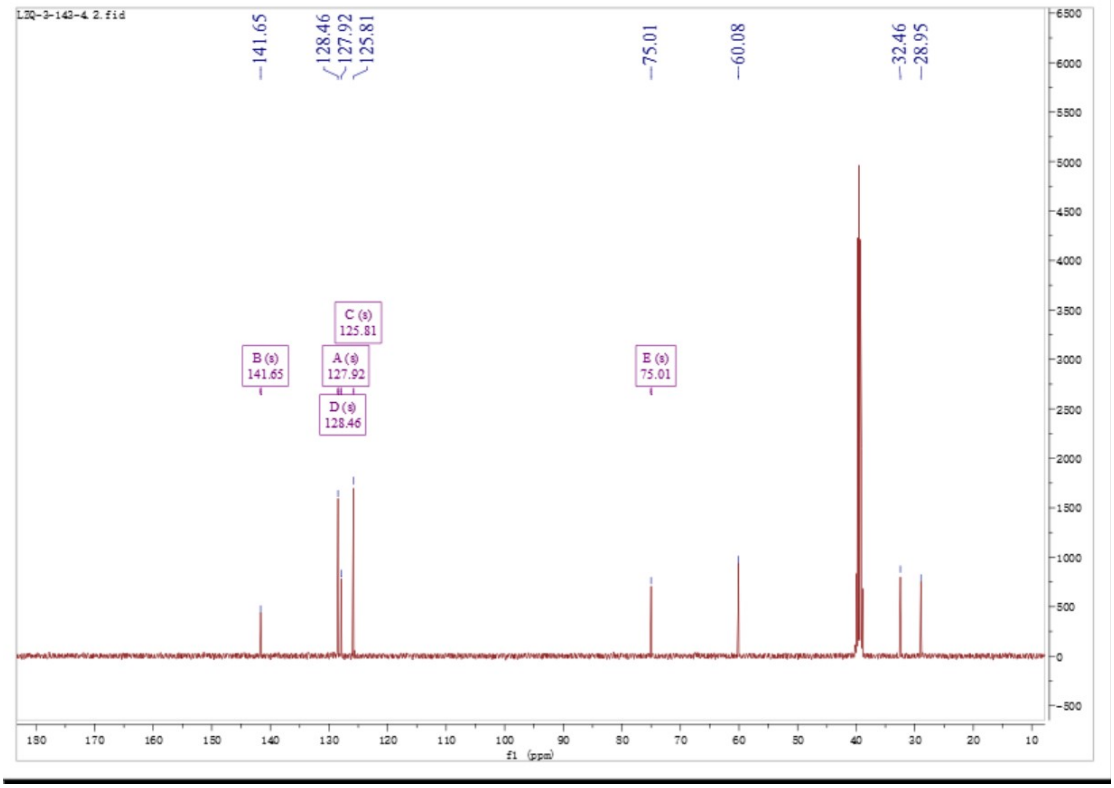


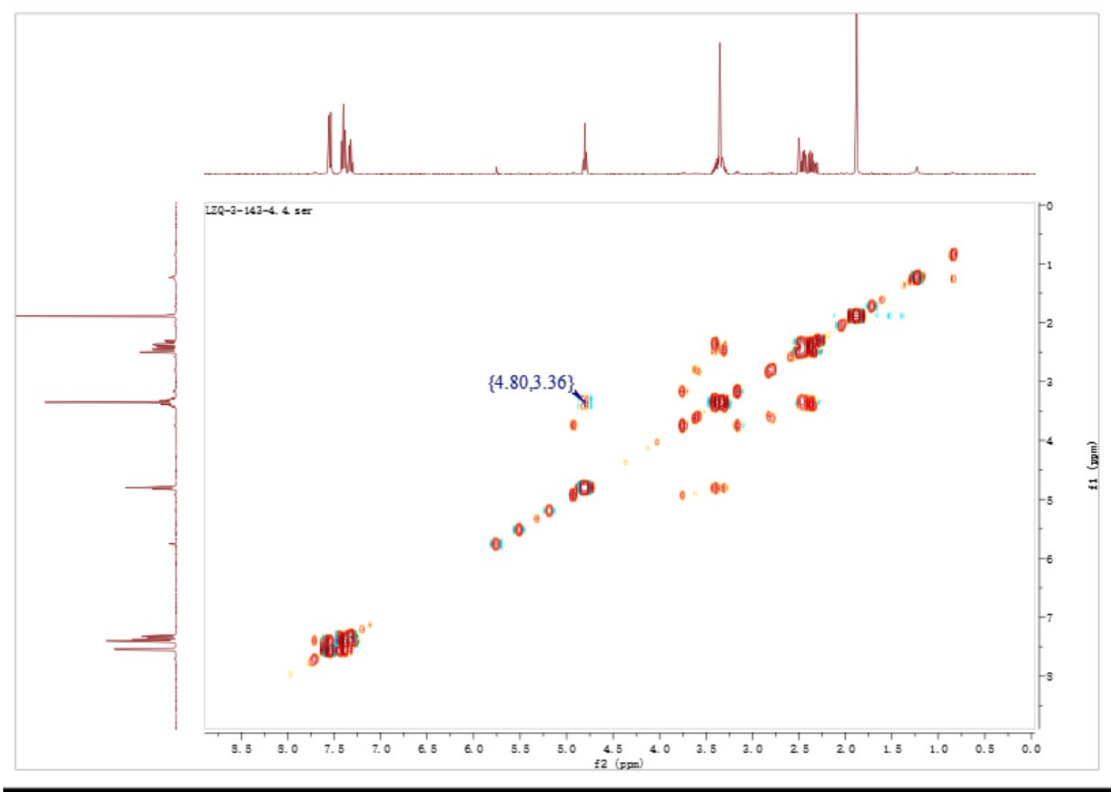
From the ^{1}H-^{1}H COSY spectrum, correlations were observed among the protons at δ 7.55, 7.40, and 7.32, indicating that H-2/H-3/H-4/H-5/H-6 are connected. Furthermore, the proton at δ 4.80 showed correlations with the proton at δ 3.36, and the proton at δ 3.36 showed correlations with the protons at δ 2.36 and 2.45, establishing the HN-O-CH_2-CH_2-S fragment.

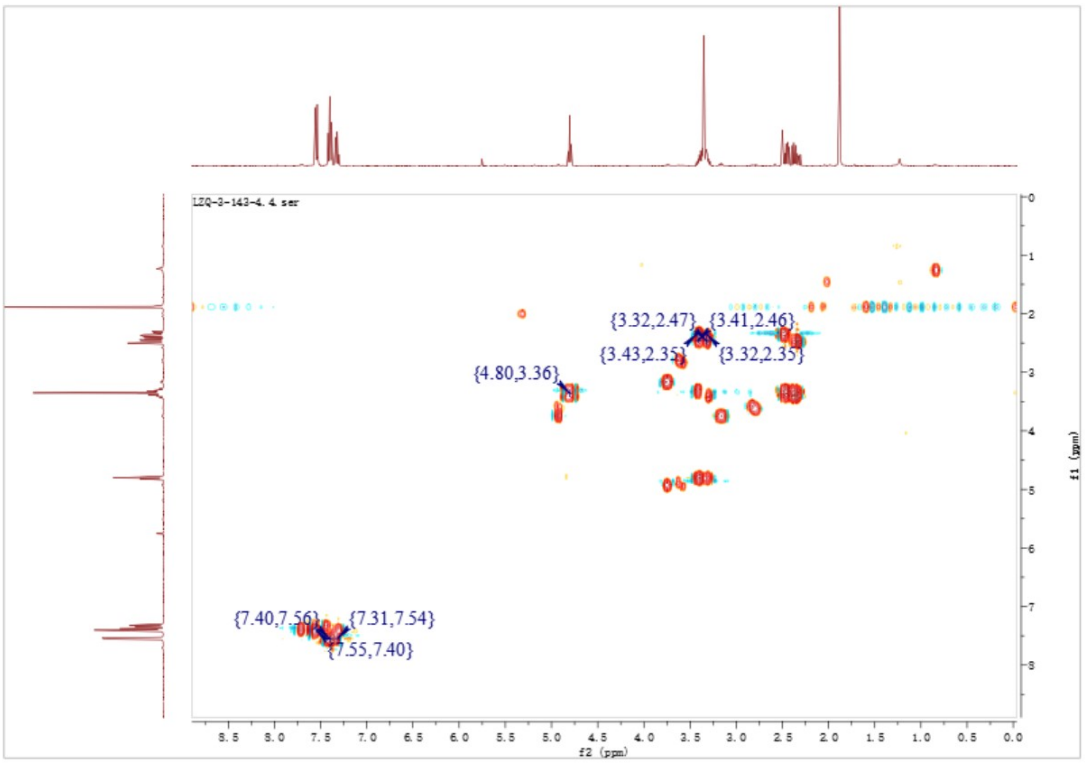
The HMBC spectrum revealed that the methyl protons at δ 1.89 exhibited correlations with carbons at δ 32.33, 74.95, and 141.69, demonstrating that the methyl group at position 13 is attached to the quaternary carbon at position 7. The proton at δ 7.55 showed a correlation with the quaternary carbon at δ 74.95, confirming that position 1

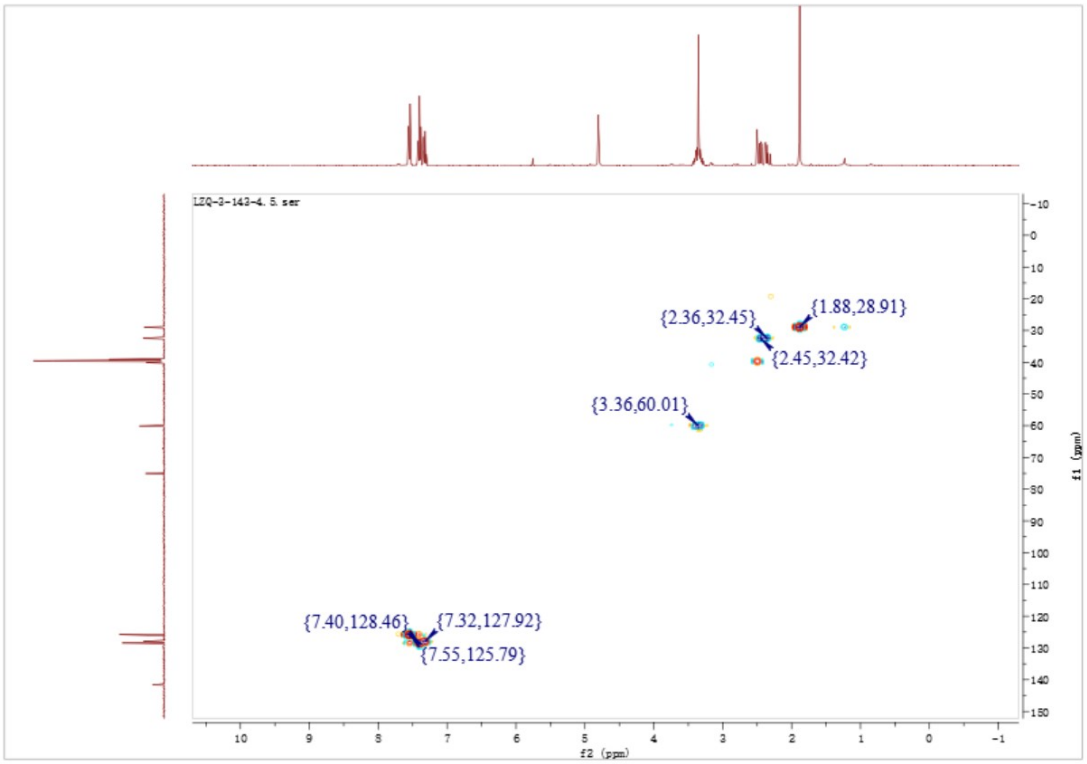
of the benzene ring is connected to this quaternary carbon. Additionally, the proton at δ 7.55, along with the protons at δ 2.36 and 2.45, exhibited correlations with the quaternary carbon at δ 74.95, indicating that the sulfur at position 8 is bonded to the quaternary carbon at position 7. The correlations of the proton at δ 4.80 with carbons at δ 32.47 and 60.08, the proton at δ 3.35 with the carbon at δ 32.47, and the protons at δ 2.36 and 2.45 with the carbon at δ 60.15 further supported the existence of the HN-O-CH_2-CH_2-S fragment.



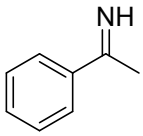

LZQ-3-143-4.2.fid
141.65
128.46
127.92
125.81
75.01
60.08
32.46
28.95
B (s) 141.65
A (s) 127.92
D (s) 128.46
C (s) 125.81
E (s) 75.01
f1 (ppm)


LZQ-3-143-4.4.ser
{4.80,3.36}
f2 (ppm)
f1 (ppm)


LZQ-3-143-4. 4. ser
{3.32,2.47}
{3.41,2.46}
{3.43,2.35}
{3.32,2.35}
{4.80,3.36}
{7.40,7.56}
{7.31,7.54}
{7.55,7.40}
f2 (ppm)
f1 (ppm)

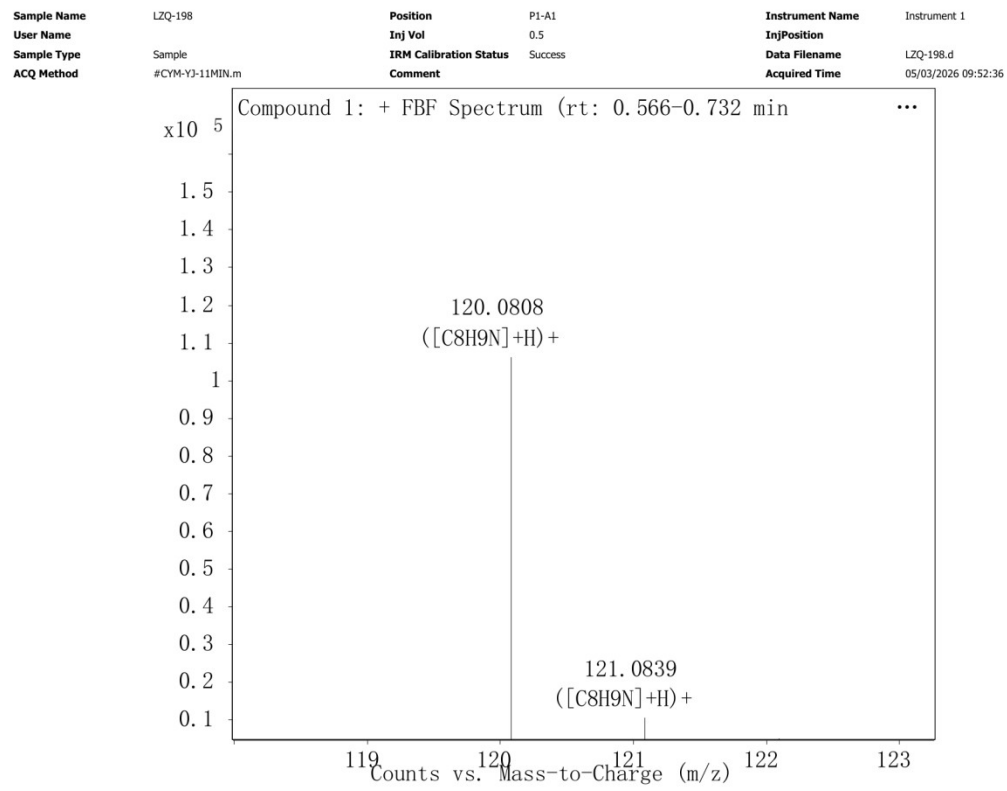

LZQ-3-143-4. 5. ser
{1.88,28.91}
{2.36,32.45}
{2.45,32.42}
{3.36,60.01}
{7.40,128.46}
{7.32,127.92}
{7.55,125.79}
f2 (ppm)
f1 (ppm)

6. Control experiments



$(M+H^+)$ 120.0808
detected in HRMS

Figure S1: Compound **4a**: **HRMS** (m/z) [ESI]: calculated for $C_8H_{10}N^+$ $[M+H]^+$: 120.0808, found 120.0808 .



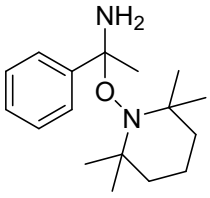
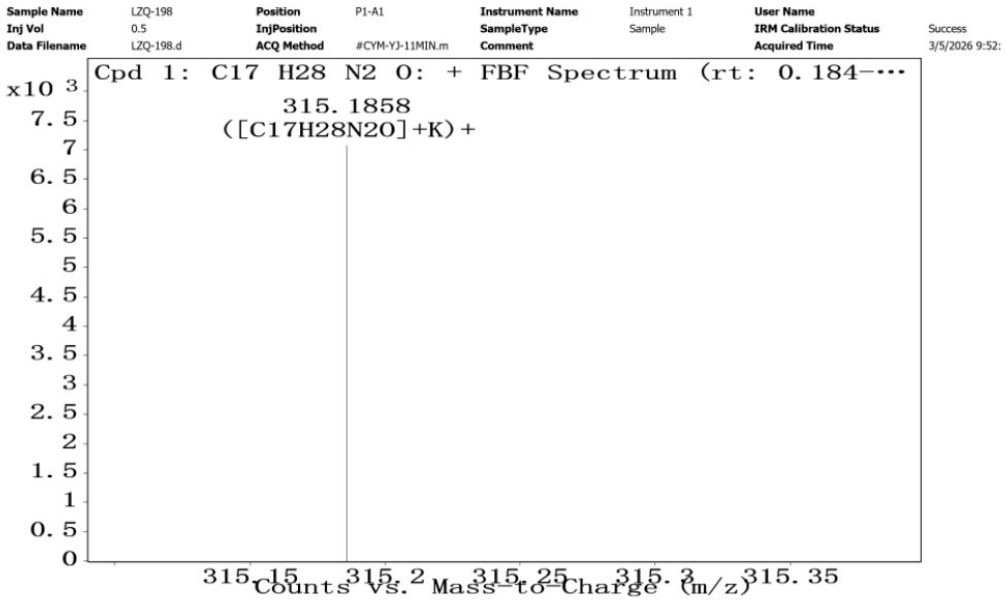
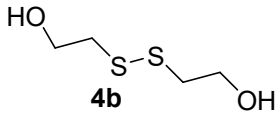


Figure S2: Compound **4c** :**HRMS** (m/z) [ESI]: calculated for $C_{17}H_{28}N_2O^+$ $[M+K]^+$: 315.1834, found 315.1858 .

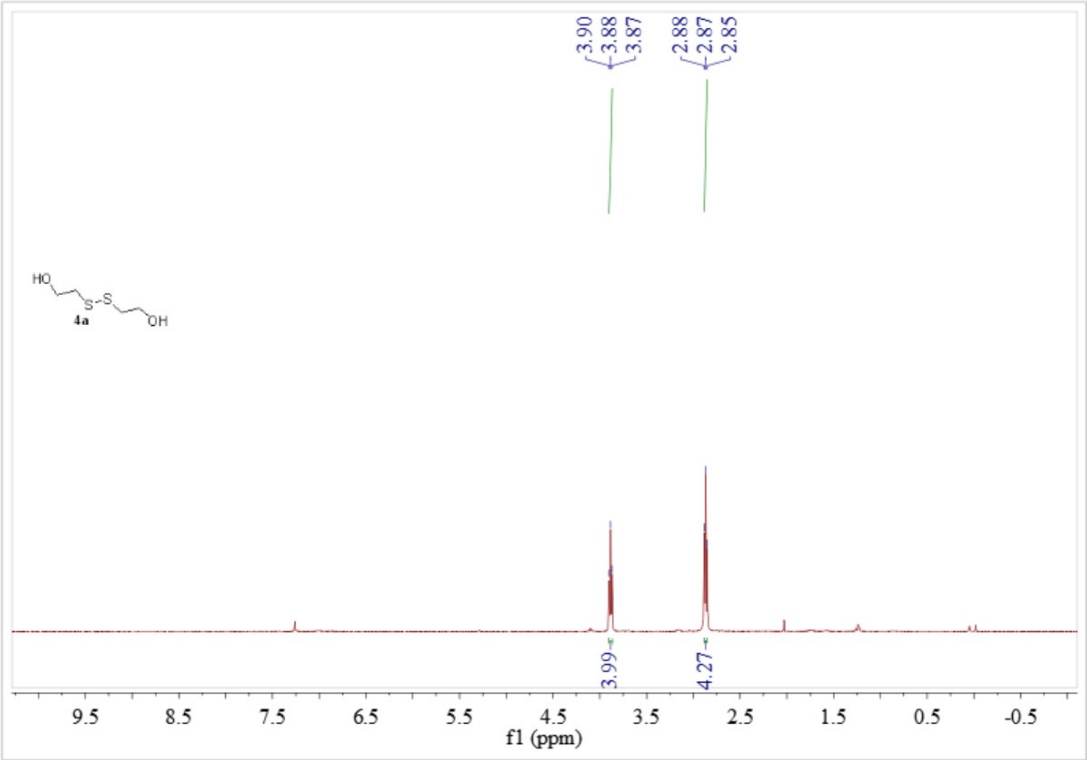


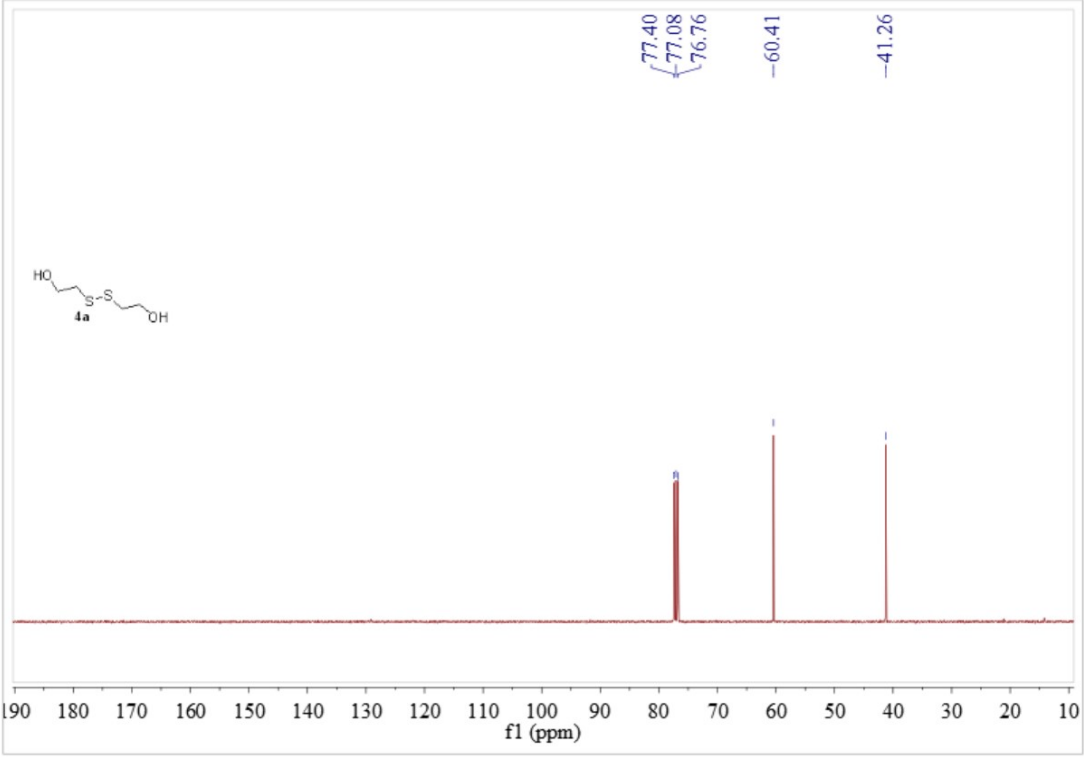


4b

2,2'-disulfanediylbis(ethan-1-ol) (4b). The title compound (170 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 3 : 1 to 1 : 1) in 80% yield.

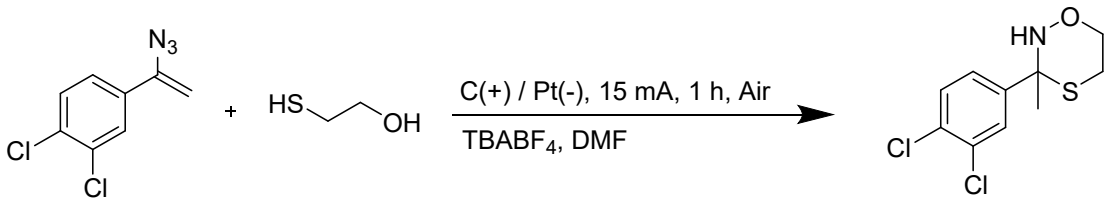
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.88 (t, *J* = 5.9 Hz, 4H), 2.87 (3, *J* = 5.9 Hz, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** 60.4, 41.3 **HRMS (m/z) (ESI)**: calcd for $C_4H_{11}O_2S_2^+$ $[M+H]^+$ 155.0195, found 155.0198.


3.90
3.88
3.87
2.88
2.87
2.85
HO
S
S
OH
4a
3.99
4.27
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


77.40
77.08
76.76
60.41
41.26
HO
S
S
OH
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

7. The application of the product

Preparation of scale-up reactions



A 25 ml round-bottomed flask was filled sequentially with **1o** (6 mmol, 1.0 eq), **2a** (10.5 mmol, 1.75 eq). The cell was equipped with a graphite rod anode (Φ6 mm) and a Platinum plate cathode (15 mm × 15 mm × 0.3 mm). DMF (20 ml) were added to a round-bottomed flask. Electrolysis was carried out at room temperature at a constant current of 15 mA for 1 h. At the end of the reaction, the reaction mixture was washed with water and extracted with dichloromethane (3 × 30 mL). The organic layers were combined, dried with Na_2SO_4 and concentrated. Flash column chromatography on silica gel gave the pure product **4o** in 70% (1.1 g) yield (petroleum ether: ethyl acetate = 7 : 1 to 3 : 1).

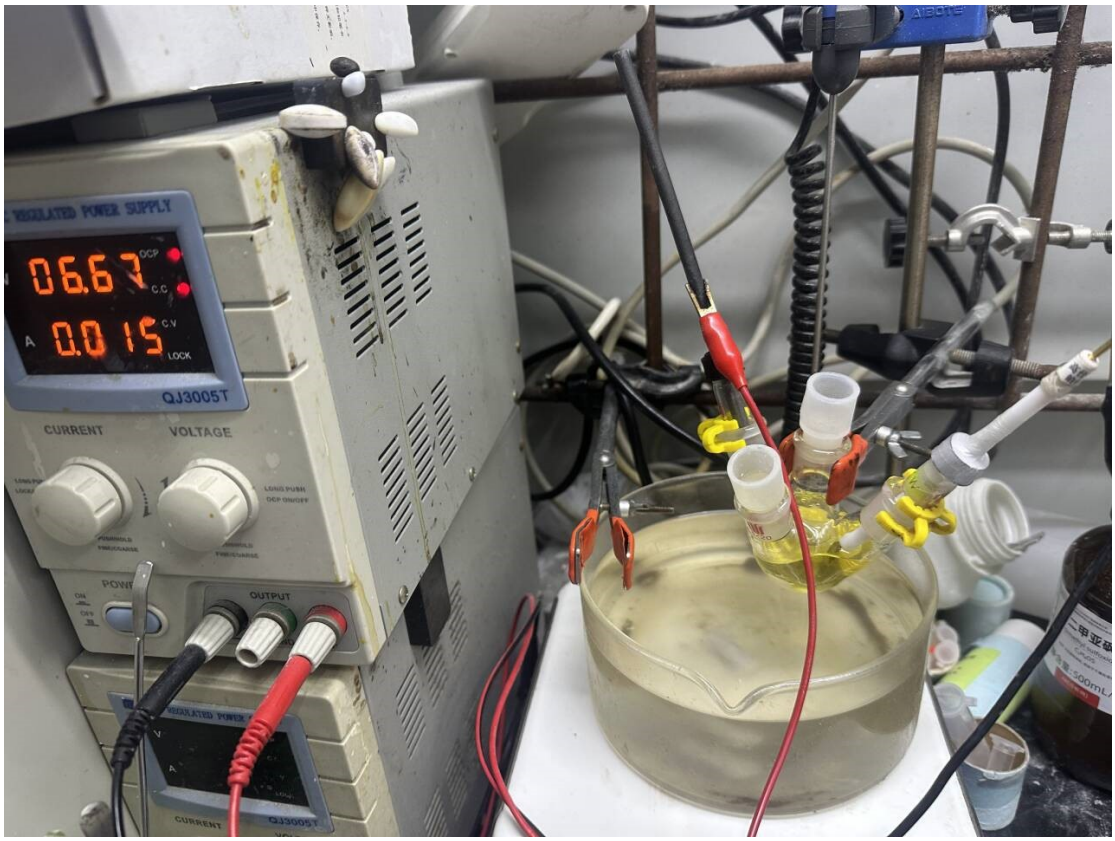
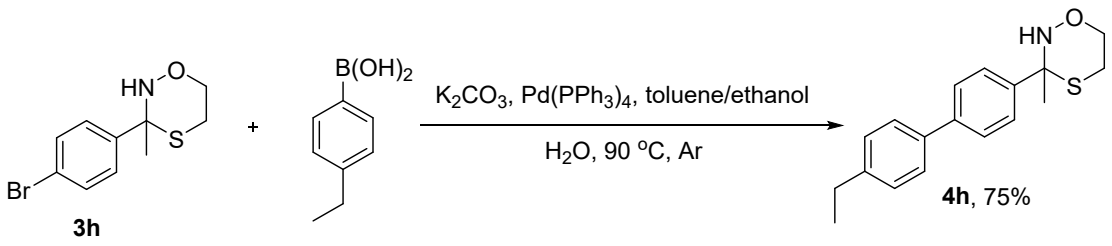


Figure S3 Electrolysis setup

Derivatisation of products



3h (0.2 mmol, 1.0 eq), 4-ethylphenylboronic acid (0.24 mmol, 1.2 eq), K_2CO_3 (0.4 mmol, 2.0 eq), tetrakis (triphenylphosphine) palladium (0.00002 mmol, 0.0001 eq). Add the above drugs into the branch mouth bottle, repeatedly pumpi ng up three times, after adding toluene (7 mL), ethanol (2 mL), H_2O (1 mL) in turn, the reaction at 90 °C 5 h and extracted with ethyl acetate (3 × 10 mL). The organic layers were combined, dried with Na_2SO_4 and concentrated. Flash column chromatography on silica gel gave the pure product **4h** (petroleum ether: ethyl acetate = 3 : 1 to 1 : 1).

8. Cyclic voltammetry studies

The cyclic voltammograms were recorded in an electrolyte solution of $LiClO_4$ (0.1 M) in CH_3CN using a glassy carbon disk working electrode (diameter, 3 mm), a Pt wire auxiliary electrode and a Ag/AgCl reference electrode. The scan rate was 100 mV/s.

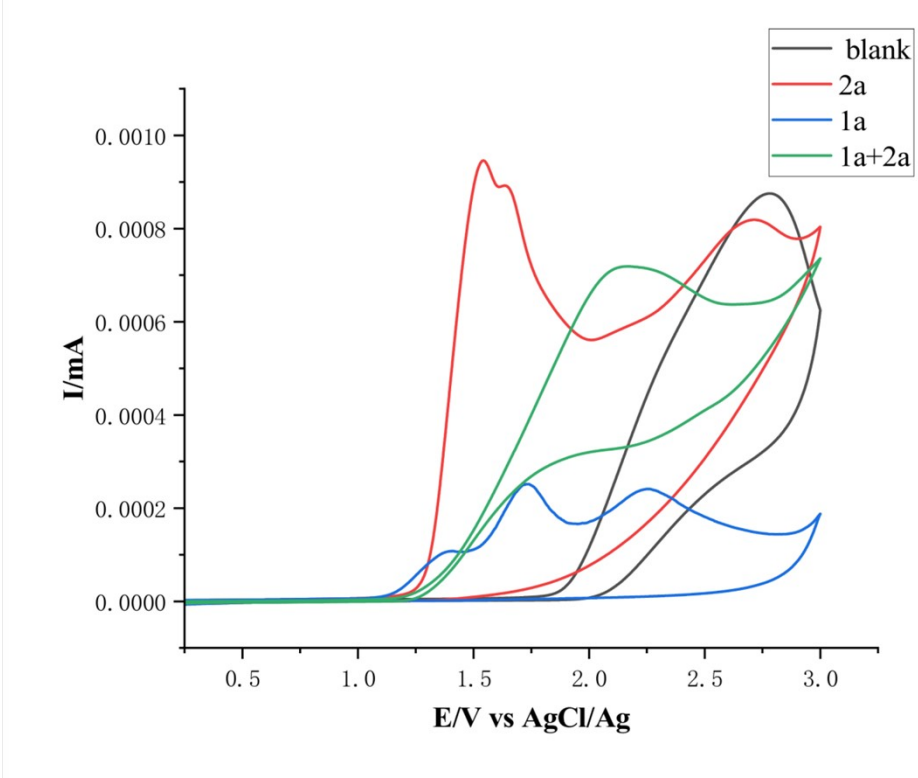
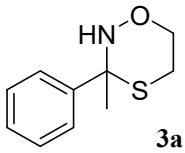


Figure S4 Cyclic voltammograms in CH_3CN + 0.1 M $LiClO_4$. Black line: blank; red line: **2a** (0.3 mmol);

blue line: **1a** (0.1 mmol); green line: **2a** (0.3 mmol) +**1a** (0.1 mmol).

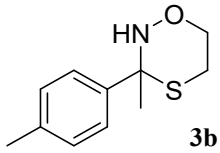
9. Characterization data for the products



3a

3-methyl-3-phenyl-1,4,2-oxathiazinane (3a). The title compound (63 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 80% yield.

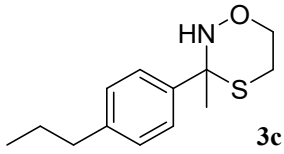
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (m, 2H), 7.36 (m, 2H), 7.28 (m, 1H), 3.58-3.49 (m, 2H), 2.56 (m, 2H), 1.94 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 141.8, 128.5, 128.0, 126.1, 75.2, 61.2, 33.4, 29.5. **HRMS (m/z) (ESI)**: calcd for $C_{10}H_{14}NOS^+$ $[M+H]^+$ 196.0791, found 196.0792.



3b

3-methyl-3-(p-tolyl)-1,4,2-oxathiazinane (3b). The title compound (70.5 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 85% yield.

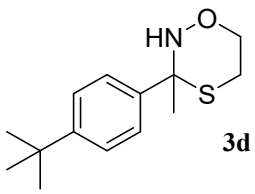
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (m, 2H), 7.17-7.15 (m, 2H), 3.60-3.49 (m, 2H), 2.61-2.52 (m, 2H), 2.34 (s, 3H), 1.92 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 139.0, 137.9, 129.3, 126.1, 75.3, 61.3, 33.6, 29.6, 21.1. **HRMS (m/z) (ESI)**: calcd for $C_{11}H_{16}NOS^+$ $[M+H]^+$ 210.0948, found 210.0948



3c

3-methyl-3-(4-propylphenyl)-1,4,2-oxathiazinane (3c). The title compound (74 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 78% yield.

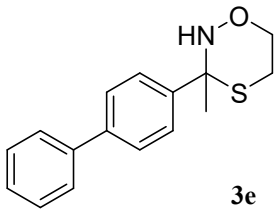
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.45(m, 2H), 7.17-7.15 (m, 2H), 3.57-3.50 (m, 2H), 2.59-2.55 (m, 4H), 1.93 (s, 3H), 1.66-1.61 (m, 2H), 0.95-0.92 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 142.7, 139.2, 128.7, 126.0, 75.3, 61.3, 37.7, 33.6, 29.6, 24.5, 14.0. **HRMS (m/z) (ESI)**: calcd for $C_{13}H_{20}NOS^+$ $[M+H]^+$ 238.1261, found 238.1266.



3d

3-(4-(tert-butyl)phenyl)-3-methyl-1,4,2-oxathiazinane (3d). The title compound (70.3 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 70% yield.

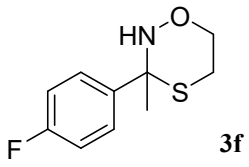
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.46 (m, 2H), 7.37-735(m, 2H), 3.62-3.50 (m, 2H), 2.59-2.56 (m, 2H), 1.93 (s, 3H), 1.31 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 151.1, 138.8, 125.8, 125.5, 75.2, 61.4, 34.6, 33.6, 31.4, 29.6. **HRMS (m/z) (ESI)**: calcd for $C_{14}H_{22}NOS^+$ $[M+H]^+$ 252.1417, found 252.1416.



3e

3-([1,1'-biphenyl]-4-yl)-3-methyl-1,4,2-oxathiazinane (3e). The title compound (83.4 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 77% yield.

1H NMR (500 MHz, CDCl3) δ 7.65-7.57 (m, 6H), 7.44 (m, 2H), 7.36 (m, 1H), 3.62-3.54 (m, 2H), 2.67-2.56 (m, 2H), 1.98 (s, 3H).**^{13}C NMR (126 MHz, $CDCl_3$)** δ 140.9, 140.3, 129.0, 127.7, 127.2, 126.7, 33.6, 29.6. **HRMS (m/z) (ESI)**: calcd for $C_{16}H_{17}NOSNa^+$ $[M+Na]^+$ 294.0941, found 294.0945

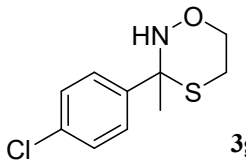


3f

3-(4-fluorophenyl)-3-methyl-1,4,2-oxathiazinane (3f). The title compound (68 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 80% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57-7.53 (m, 2H), 7.05-7.01(m, 2H), 3.59-3.54 (m, 2H), 2.57 – 2.52 (m, 2H), 1.92 (s, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.5, 161.0,

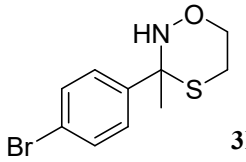
137.8, 128.1, 115.5, 115.3, 74.8, 61.3, 33.5, 29.8.**^{19}F NMR (376 MHz, $CDCl_3$)** δ -114.24 (s, 1F). **HRMS (m/z) (ESI)**: calcd for $C_{10}H_{13}FNOS^+$ $[M+H]^+$ 214.0701, found 214.0702.



3g

3-(4-chlorophenyl)-3-methyl-1,4,2-oxathiazinane (3g). The title compound (75.1 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 82% yield.

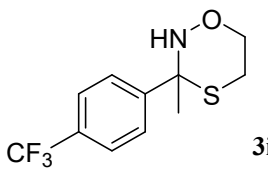
1H NMR (500 MHz, CDCl3) δ 7.52-7.50 (m, 2H), 7.33-7.26 (m, 2H), 3.59-3.53 (m, 2H), 2.57-2.50 (m, 2H), 1.90 (s, 3H). **13C NMR (126 MHz, CDCl3)** δ 140.6, 133.9, 128.7, 127.7, 74.7, 61.2, 33.4, 29.6. **HRMS (m/z) (ESI)**: calcd for $C_{10}H_{13}ClNOS^+$ $[M+H]^+$ 230.0401, found 230.0401.



3h

3-(4-bromophenyl)-3-methyl-1,4,2-oxathiazinane (3h). The title compound (92.8 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 85% yield.

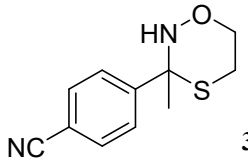
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (mz, 4H), 3.61-3.49 (m, 2H), 2.52 (m, 7.2 Hz, 2H), 1.89 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.1, 131.6, 128.0, 122.0, 74.7, 61.1, 33.3, 29.5. **HRMS** (m/z) (ESI): calcd for $C_{10}H_{13}BrNOS^+$ $[M+H]^+$ 273.9896, found 273.9895.



3i

3-methyl-3-(4-(trifluoromethyl)phenyl)-1,4,2-oxathiazinane (3i). The title compound (85.2 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 81% yield.

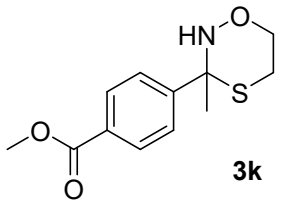
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.70 (m, 2H), 7.62-7.60 (m, 2H), 3.64-3.53 (m, 2H), 2.59-2.47 (m, 2H), 1.94 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.0, 130.4, 130.1, 126.8, 125.6,, 74.7, 61.2, 33.4, 29.6. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -62.63 (s, 3F). **HRMS** (m/z) (ESI): calcd for $C_{11}H_{13}F_3NOS^+$ $[M+H]^+$ 264.0665, found 264.0663.



3j

4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzonitrile (3j). The title compound (46.7 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 53% yield.

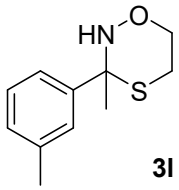
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.69 (m, 2H), 7.64 (m, 2H), 3.59-3.54 (m, 2H), 2.57-2.43 (m, 2H), 1.92 (s, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 147.3, 132.4, 127.2, 111.9, 61.1, 33.2, 29.4. **HRMS** (m/z) (ESI): calcd for $C_{11}H_{13}N_2OS^+$ $[M+H]^+$ 221.0744, found 221.0743.



3k

methyl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3k). The title compound (80 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 79% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, 2H), 7.65 (d, 2H), 3.92 (s, 3H), 3.60-3.48 (m, 2H), 2.56-2.50 (m, 2H), 1.94 (s, 3H)). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 166.7, 147.0, 129.9, 126.37 (s), 74.8, 61.2, 52.4, 33.5, 29.4. **HRMS (m/z) (ESI)**: calcd for $C_{12}H_{16}NO_3S^+$ $[M+H]^+$ 254.0846, found 254.0844.

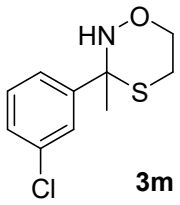


3l

3-methyl-3-(m-tolyl)-1,4,2-oxathiazinane (3l). The title compound (66.1 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 79% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.36 (m, 2H), 7.23 (m, 1H), 7.10-7.08(m, 1H), 3.61-3.49

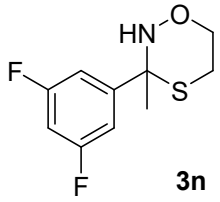
(m, 2H), 2.59-2.55 (m, 4.0 Hz, 2H), 2.37 (s, 3H), 1.93 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.9, 138.3, 128.8, 128.5, 126.8, 123.3, 75.3, 61.3, 33.6, 29.6, 21.7. **HRMS (m/z) (ESI)**: calcd for $C_{11}H_{15}NOS^+$ $[M+H]^+$ 210.0947, found 210.0948.



3m

3-(3-chlorophenyl)-3-methyl-1,4,2-oxathiazinane (3m). The title compound (77.9 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 85% yield.

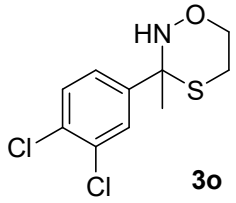
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (m, 1H), 7.43-7.40 (m, 1H), 7.26-7.19 (m, 2H), 3.58-3.47 (m, 2H), 2.56-2.44 (m, 2H), 1.86 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.2, 134.6, 129.9, 128.2, 126.6, 124.5, 74.6, 61.3, 33.5, 29.6. **HRMS (m/z) (ESI)**: calcd for $C_{10}H_{13}ClNOS^+$ $[M+H]^+$ 230.0401, found 230.0400.



3n

3-(3,5-difluorophenyl)-3-methyl-1,4,2-oxathiazinane (3n). The title compound (80 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 86% yield.

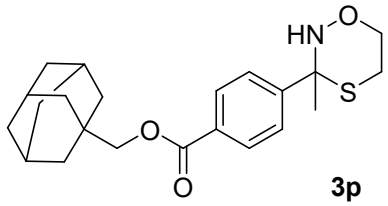
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (m, 2H), 6.73 (m, 1H), 3.66-3.55 (m, 2H), 2.63-2.49 (m, 2H), 1.89 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.3, 161.8, 146.4, 109.89-109.42 (m), 103.5 , 74.2, 61.2, 33.4, 29.5. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -109.44 (s, 2F)**.HRMS (m/z) (ESI)**: calcd for $C_{10}H_{12}F_2NOS^+$ $[M+H]^+$ 232.0603, found 232.0601.



3o

3-(3,4-dichlorophenyl)-3-methyl-1,4,2-oxathiazinane (3o). The title compound (78.9 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 75% yield.

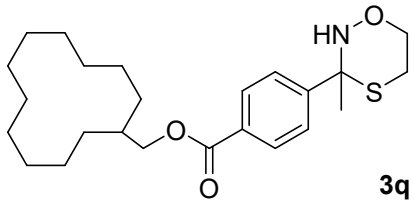
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.64 (m, 1H), 7.41 (m, 2H), 3.60-3.56 (m, 2H), 2.57-2.48 (m, 2H), 1.88 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 142.4, 132.7), 132.1, 130.5, 128.4, 125.8, 74.1, 61.2, 33.3, 29.6. **HRMS (m/z) (ESI)**: calcd for $C_{10}H_{12}Cl_2NOS^+$ $[M+H]^+$ 264.0012, found 264.0011.



3p

adamantan-1-ylmethyl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3p). The title compound (116.1 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 75% yield.

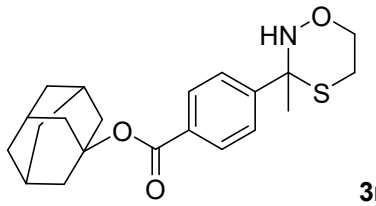
1H NMR (400 MHz, CDCl3) δ 8.04-8.02 (m, 2H), 7.67-7.65 (m, 2H), 3.92 (s, 2H), 3.59-3.53 (m, 2H), 2.60-2.48 (m, 2H), 2.04-2.02 (m, 3H), 1.94 (s, 3H), 1.77-1.67 (m, 7H), 1.66-1.62 (m, 5H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 166.2, 146.8, 130.3, 129.9, 126.4, 74.9, 74.8, 61.3, 39.6, 37.1, 33.7, 33.5, 29.5, 28.2. **HRMS (m/z) (ESI)**: calcd for $C_{22}H_{30}NO_3S^+$ $[M+H]^+$ 388.1993, found 388.1997.



3q

Cyclododecylmethyl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3q). The title compound (117.3 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 70% yield.

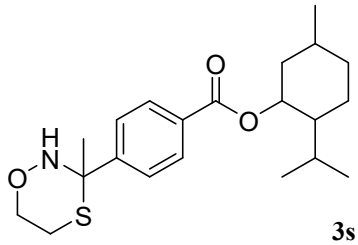
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02-8.00 (m, 2H), 7.65-7.63(m, 2H), 3.62-3.49 (m, 2H), 2.60-2.47 (m, 2H), 1.94 (s, 3H), 1.81 (m, 1H), 1.66 (m, 1H), 1.51-1.31 (m, 20H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.8, 146.7, 130.7, 129.8, 126.3, 74.9, 73.3, 61.2, 33.5, 29.5, 29.3, 24.3, 24.1, 23.5, 24.3, 21.0. **HRMS (m/z) (ESI)**: calcd for $C_{24}H_{37}NO_3SNa^+$ $[M+H]^+$ 420.2387, found 442.2380.



3r

adamantan-1-yl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3r). The title compound (116.2 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 78% yield.

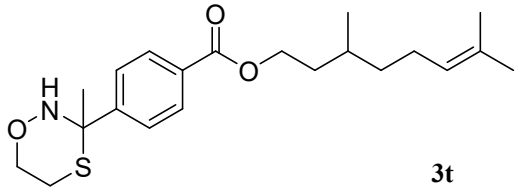
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.94 (m, 2H), 7.63-7.60 (m, 2H), 3.59-3.48 (m, 2H), 2.59-2.46 (m, 2H), 2.24-2.22 (m, 9H), 1.93 (s, 3H), 1.74-1.67 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.0, 146.3, 131.8, 129.7, 126.1, 81.5, 74.9, 61.2, 41.5, 36.3, 33.5, 31.0, 29.5. **HRMS (m/z) (ESI)**: calcd for $C_{21}H_{28}NO_3S^+$ $[M+H]^+$ 374.1785, found 374.1778.



3s

2-isopropyl-5-methylcyclohexyl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3s). The title compound (108 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 72% yield.

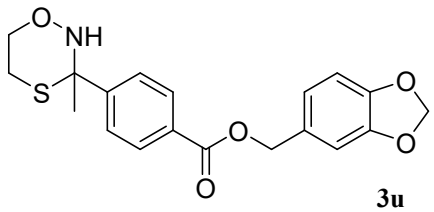
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (m, 2H), 7.64 (m, 2H), 4.93 (m, 1H), 3.65-3.50 (m, 2H), 2.61-2.45 (m, 2H), 1.93 (s, 3H), 1.73 (m, 2H), 1.60-1.51 (m, 2H), 1.25 m, 1H), 0.92 (m, 8H), 0.79 (m , 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.7, 146.7, 130.5, 129.9, 126.3, 75.2, 61.3, 47.4, 41.1, 34.4, 33.5, 31.5, 29.5, 26.6, 23.7, 22.16, 20.9, 16.6. **HRMS (m/z) (ESI)**: calcd for $C_{21}H_{32}NO_3S^+$ $[M+H]^+$ 378.2098, found 378.2094.



3t

3,7-dimethyloct-6-en-1-yl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3t). The title compound (107.1 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in71% yield.

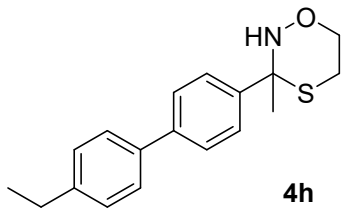
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (m, 2H), 7.65 (m, 2H), 5.38-5.34 (m, 1H), 4.35-4.28 (m, 2H), 3.54 (m, 2H), 2.61-2.48 (m, 2H), 2.42 (m, 2H), 2.39-2.36 (m, 1H), 2.24 (m, 2H), 2.13-2.07 (m, 2H), 1.94 (s, 3H), 1.84-1.68 (m, 1H), 1.27 (s, 3H), 1.25 (m, 1H), 1.17 (m, 1H), 0.83 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 166.1, 146.8, 144.3, 130.1, 129.9, 126.3, 119.1, 74.9, 63.2, 61.2, 45.9, 40.8, 38.2, 36.2, 33.5, 31.8, 31.5), 29.4, 26.4, 21.3 . **HRMS (m/z) (ESI)**: calcd for $C_{21}H_{32}NO_3S^+$ $[M+H]^+$ 378.2098, found 378.2094.



3u

benzo[d][1,3]dioxol-5-ylmethyl 4-(3-methyl-1,4,2-oxathiazinan-3-yl)benzoate (3u). The title compound (118 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 79% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05-8.00 (m, 2H), 7.66-7.61 (m, 2H), 6.95-6.90 (m, 2H), 6.81 (d, J = 7.8 Hz, 1H), 5.97 (s, 2H), 5.25 (s, 2H), 3.59 – 3.46 (m, 2H), 2.58 – 2.44 (m, 2H), 1.93 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.9, 147.8, 147.0, 129.9, 129.7, 126.29 (s), 122.4, 109.1, 108.3, 101.3, 74.7, 66.9, 61.1, 33.2, 29.3. **HRMS (m/z) (ESI)**: calcd for $C_{19}H_{20}NO_5S^+$ $[M+H]^+$ 374.1057, found 374.1053.



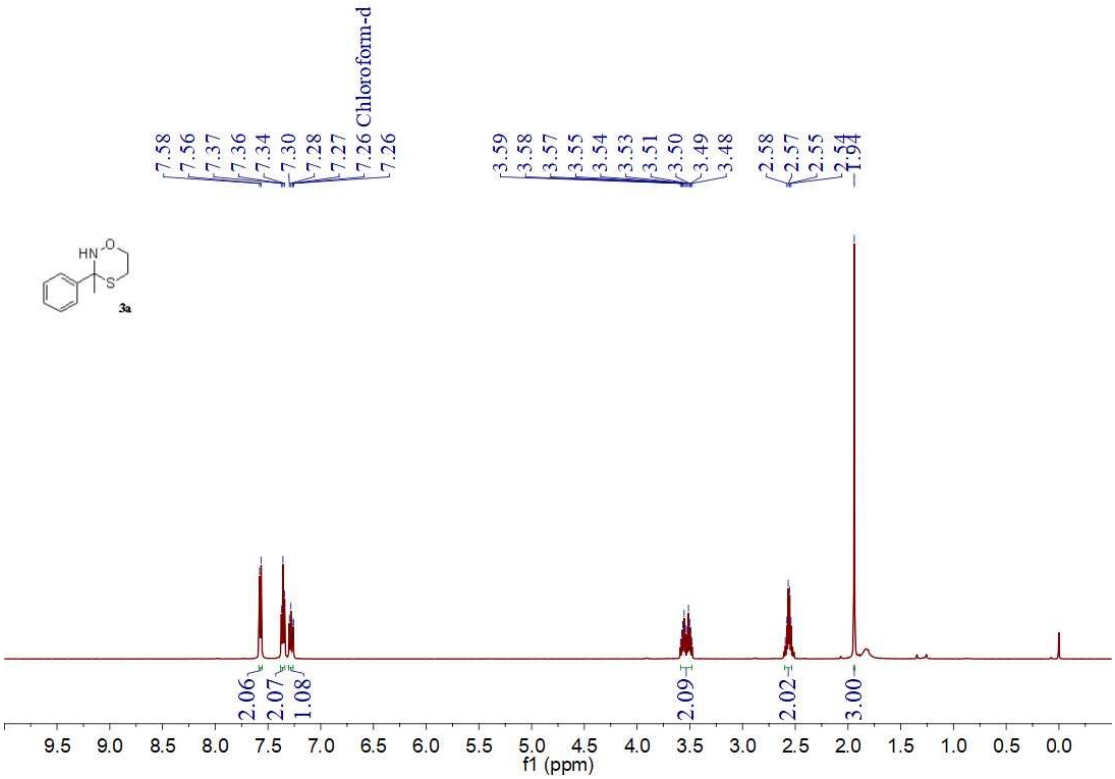
4h

3-(4'-ethyl-[1,1'-biphenyl]-4-yl)-3-methyl-1,4,2-oxathiazinane (4h). The title compound (102 mg) was isolated by flash chromatography (petroleum ether : ethyl acetate = 10 : 1 to 5 : 1) in 85% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.52 (m, 6H), 7.30 (m, 2H), 3.66-3.53 (m, 2H), 2.72 (qm, 2H), 2.66-2.54 (m, 2H), 1.98 (s, 3H), 1.30 (m, 3H). **^{13}C NMR (101 MHz, CDCl3)** δ 143.9, 140.8, 140.6, 137.6, 128.5, 127.0, 126.6, 75.2, 61.3, 33.5, 29.6, 28.6, 15.7. **HRMS (m/z) (ESI)**: calcd for $C_{18}H_{22}NOS^+$ $[M+H]^+$ 300.1412, found 300.1413.

10. NMR Data

3-methyl-3-phenyl-1,4,2-oxathiazinane (**3a**).


7.58
7.56
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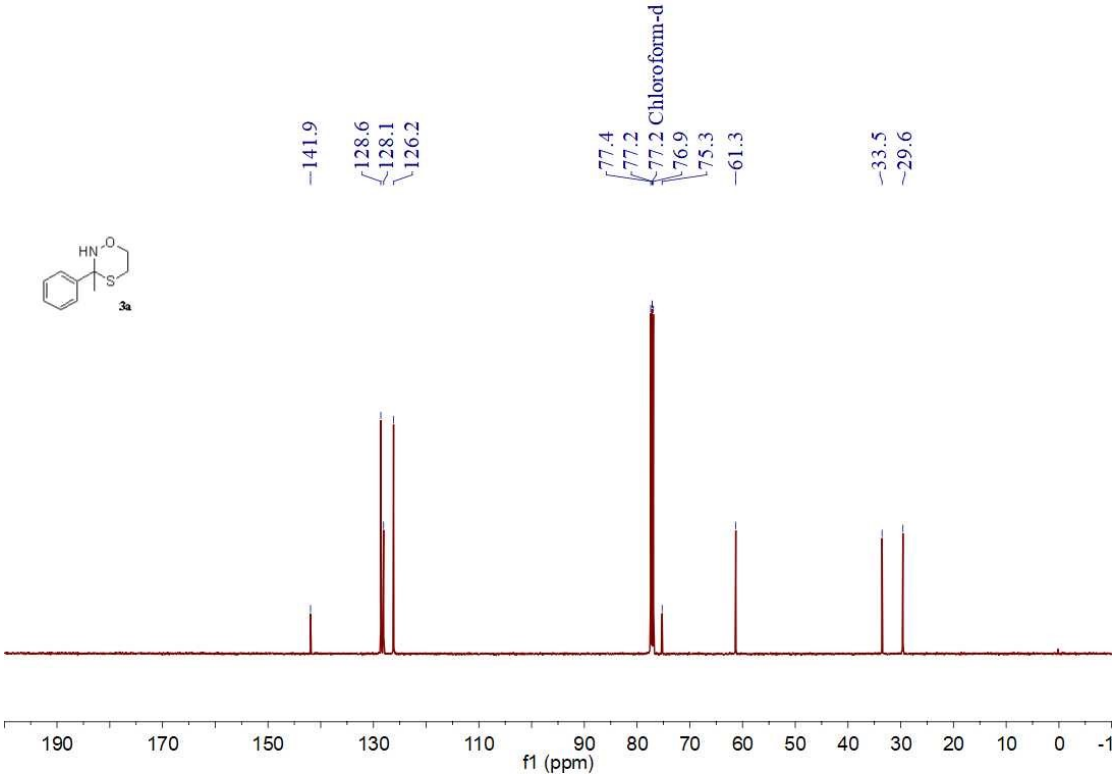
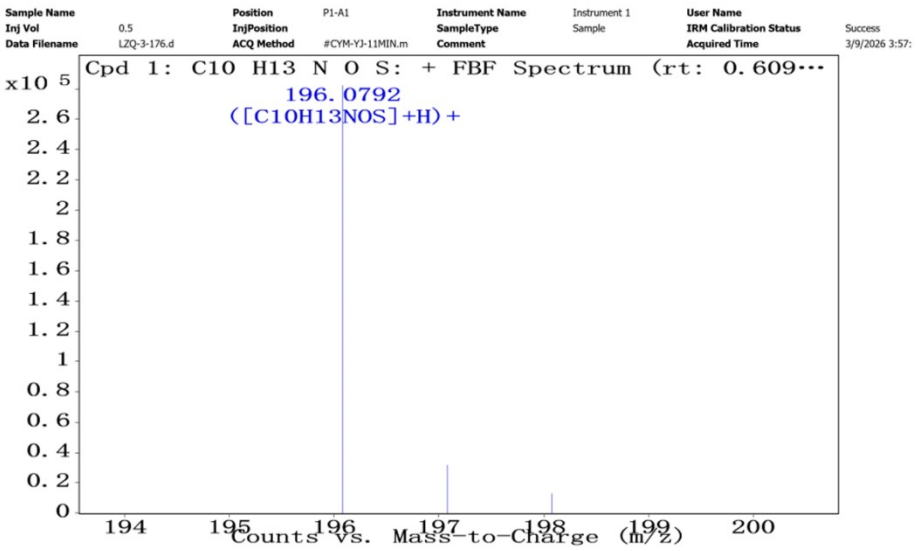
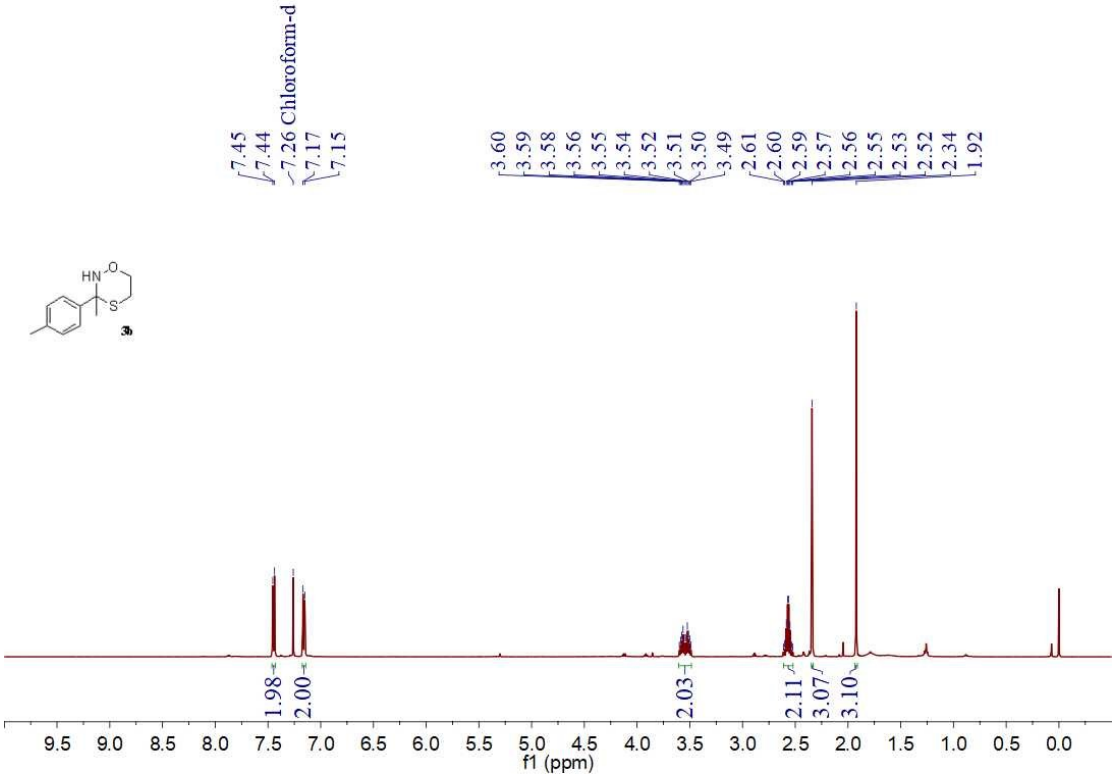
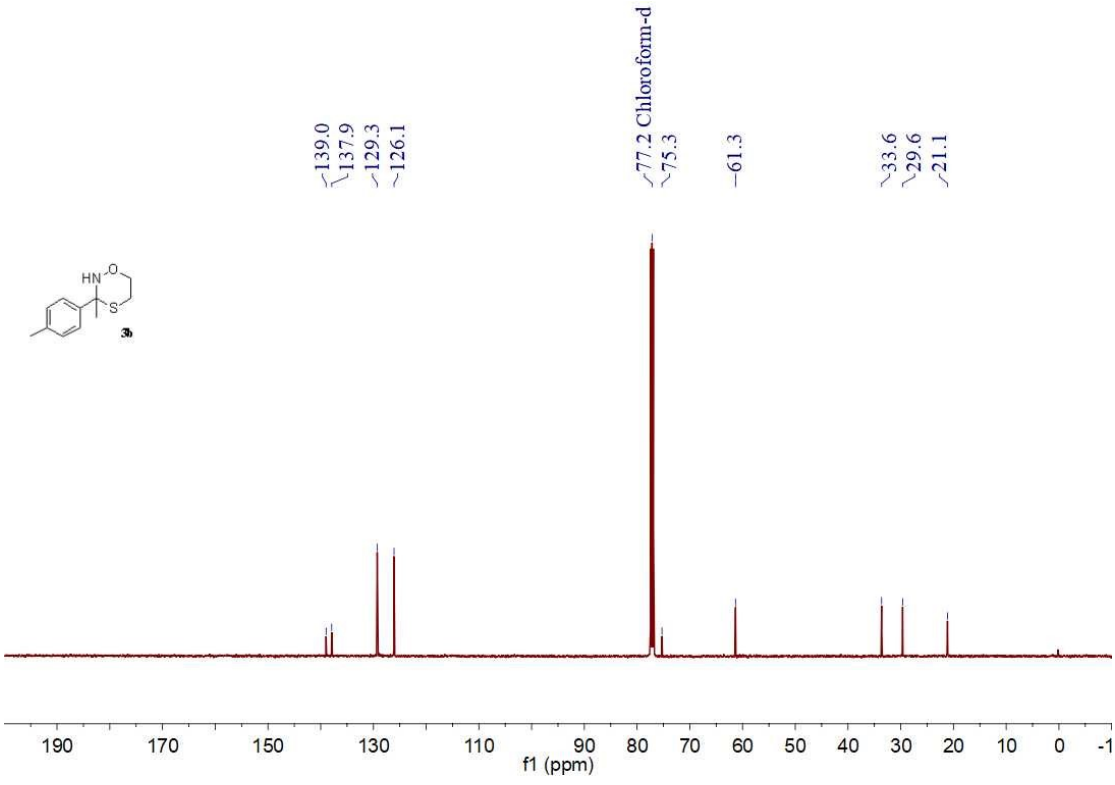
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

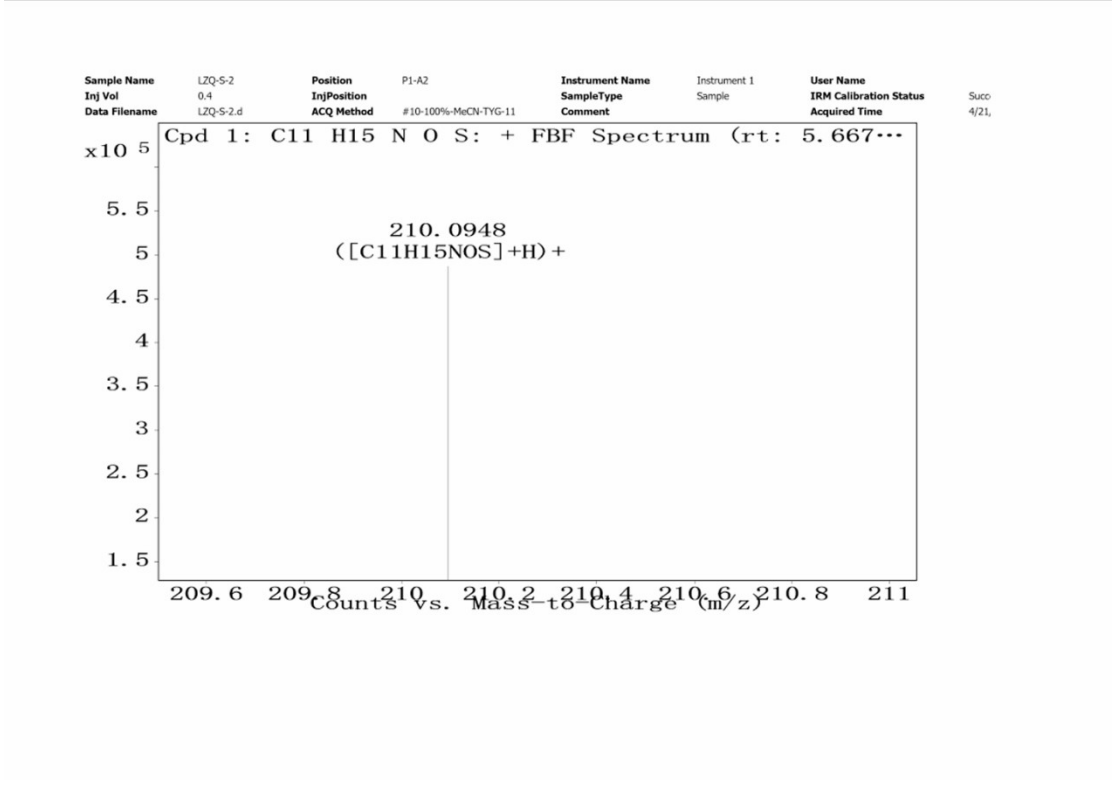

141.9
128.6
128.1
126.2
77.4
77.2
77.2 Chloroform-d
76.9
75.3
61.3
33.5
29.6
HN
O
S
3a
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



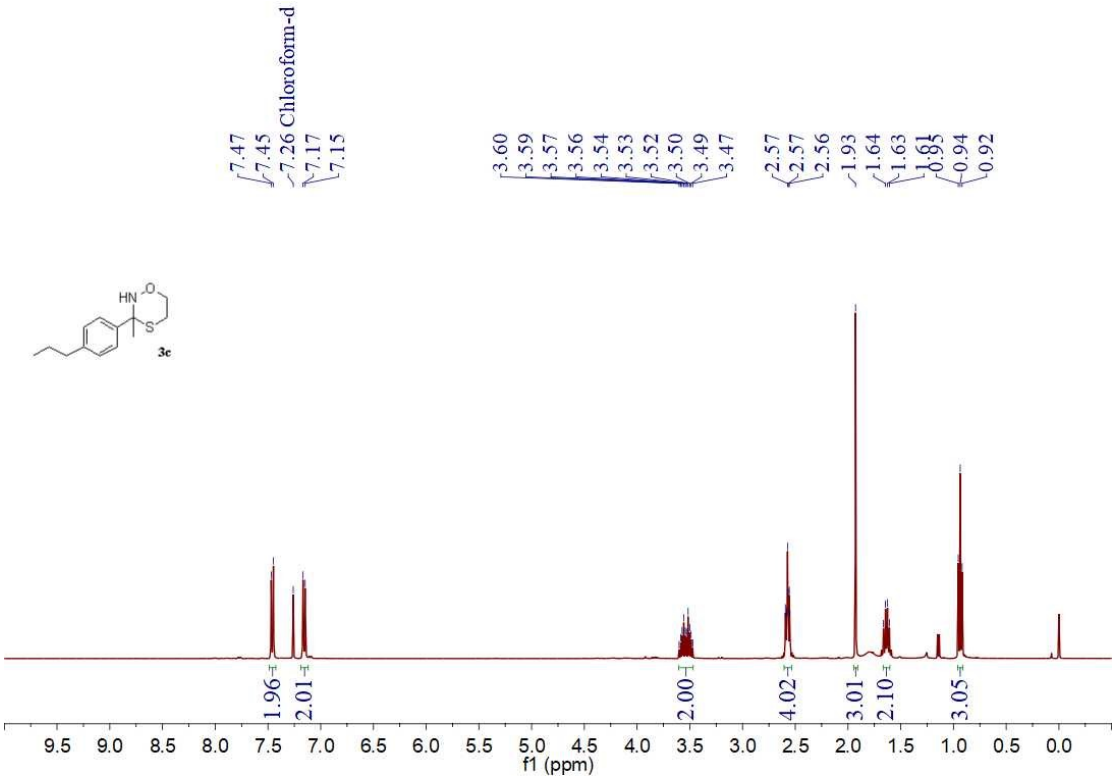
3-methyl-3-(p-tolyl)-1,4,2-oxathiazinane (**3b**).

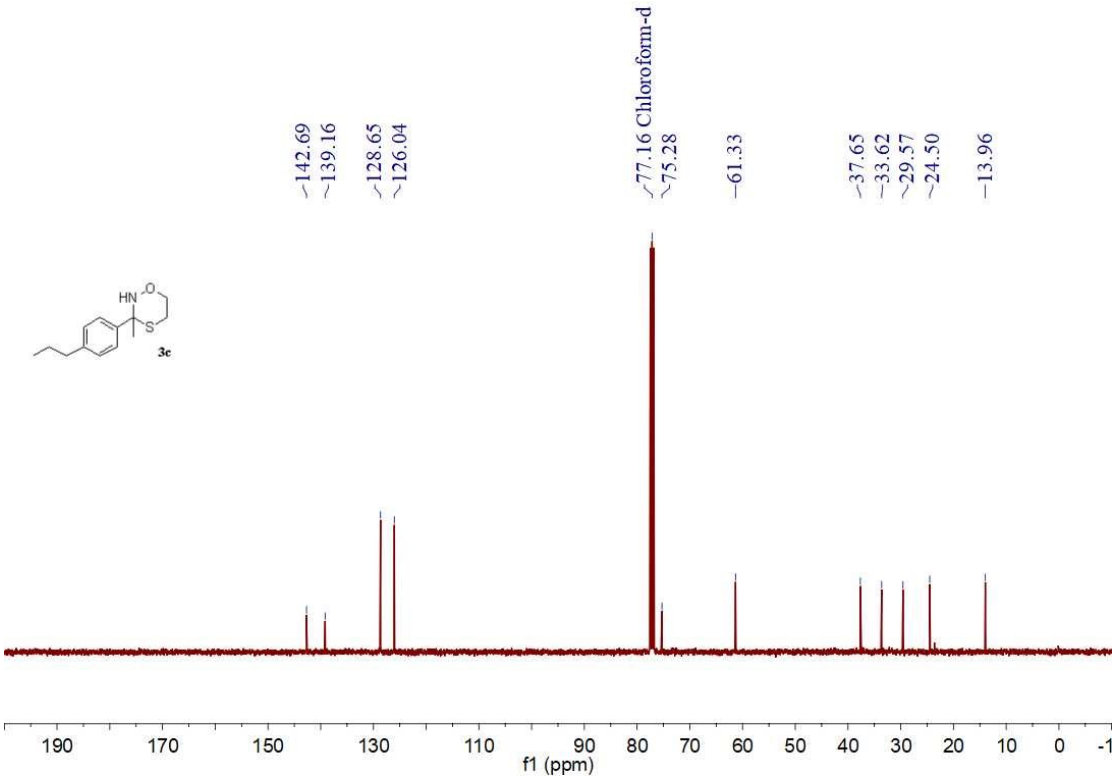


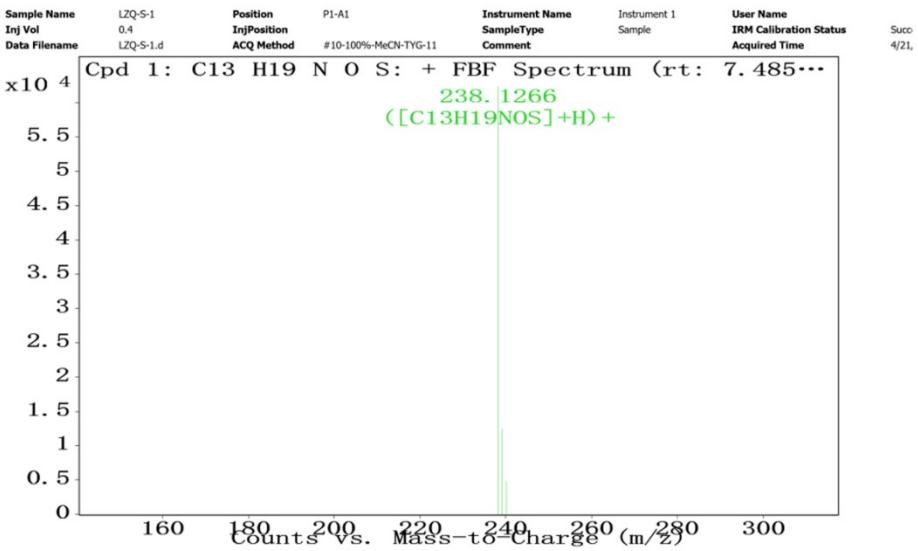




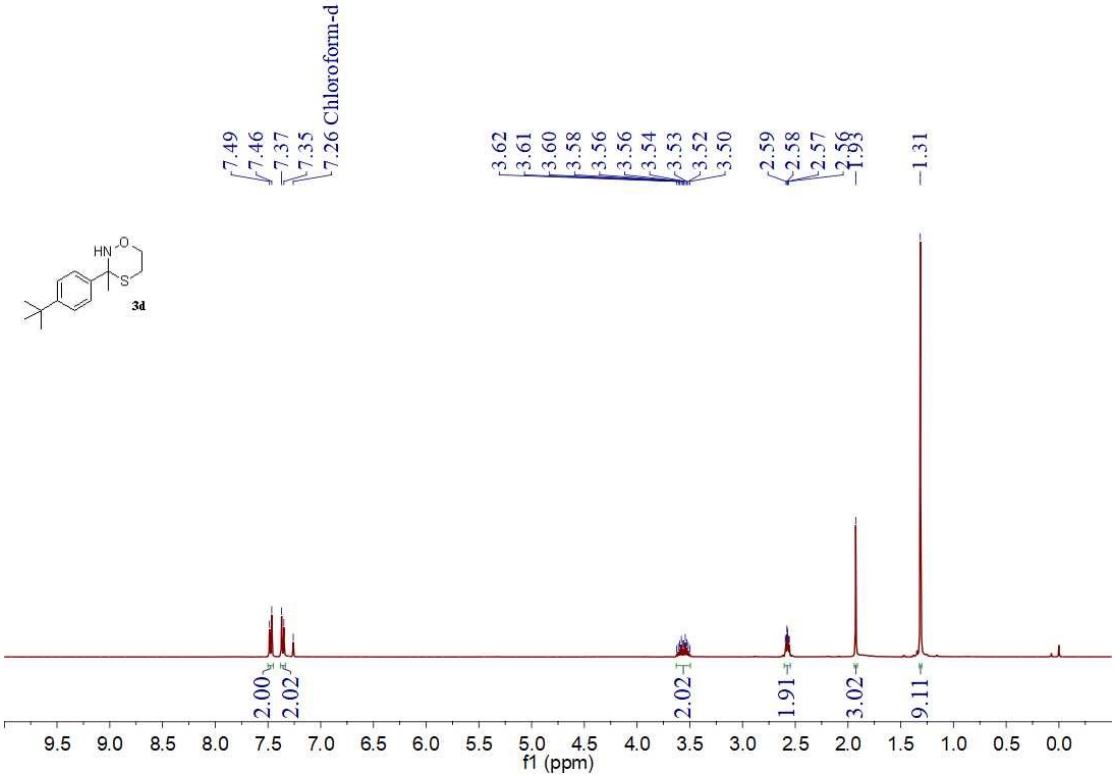
3-methyl-3-(4-propylphenyl)-1,4,2-oxathiazinane (**3c**).

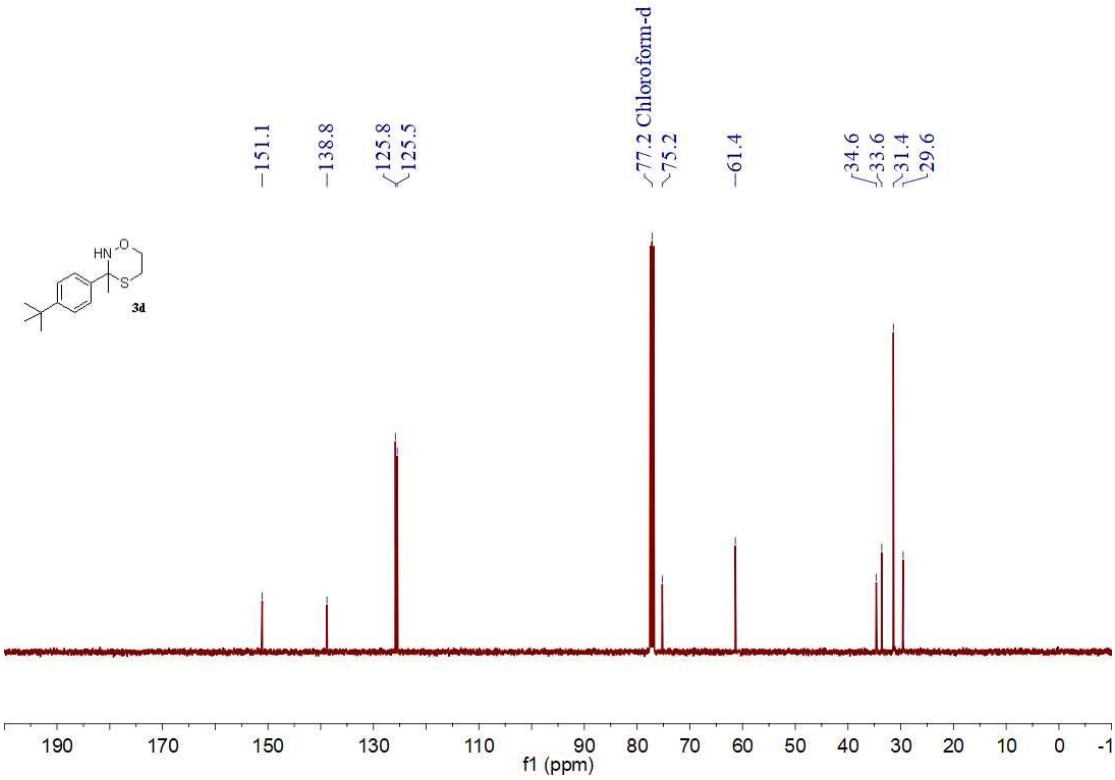

7.47
7.45
7.26 Chloroform-d
7.17
7.15
3.60
3.59
3.57
3.56
3.54
3.53
3.52
3.50
3.49
3.47
2.57
2.57
2.56
1.93
1.64
1.63
1.61
0.95
0.94
0.92
HN
O
S
3c
1.96
2.01
2.00
4.02
3.01
2.10
3.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

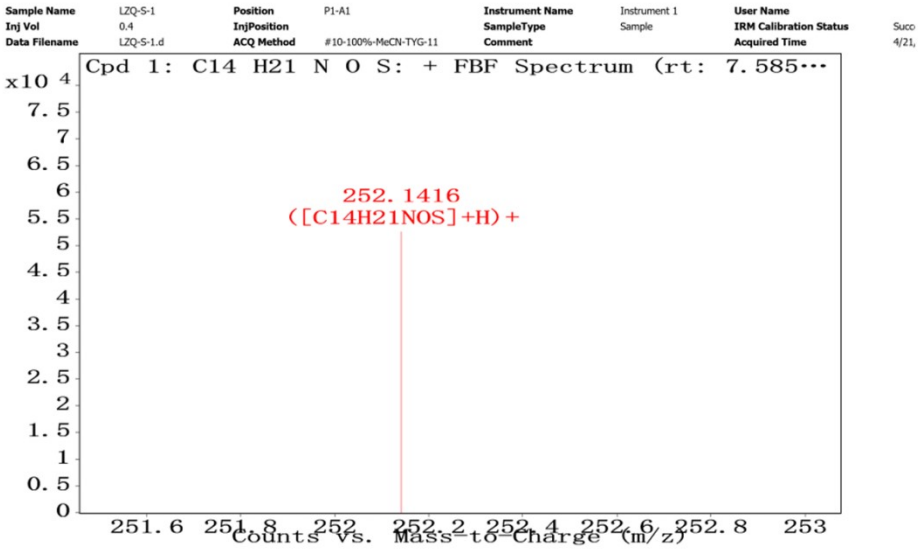

142.69
139.16
128.65
126.04
77.16 Chloroform-d
75.28
61.33
37.65
33.62
29.57
24.50
13.96
HN
O
S
3c
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



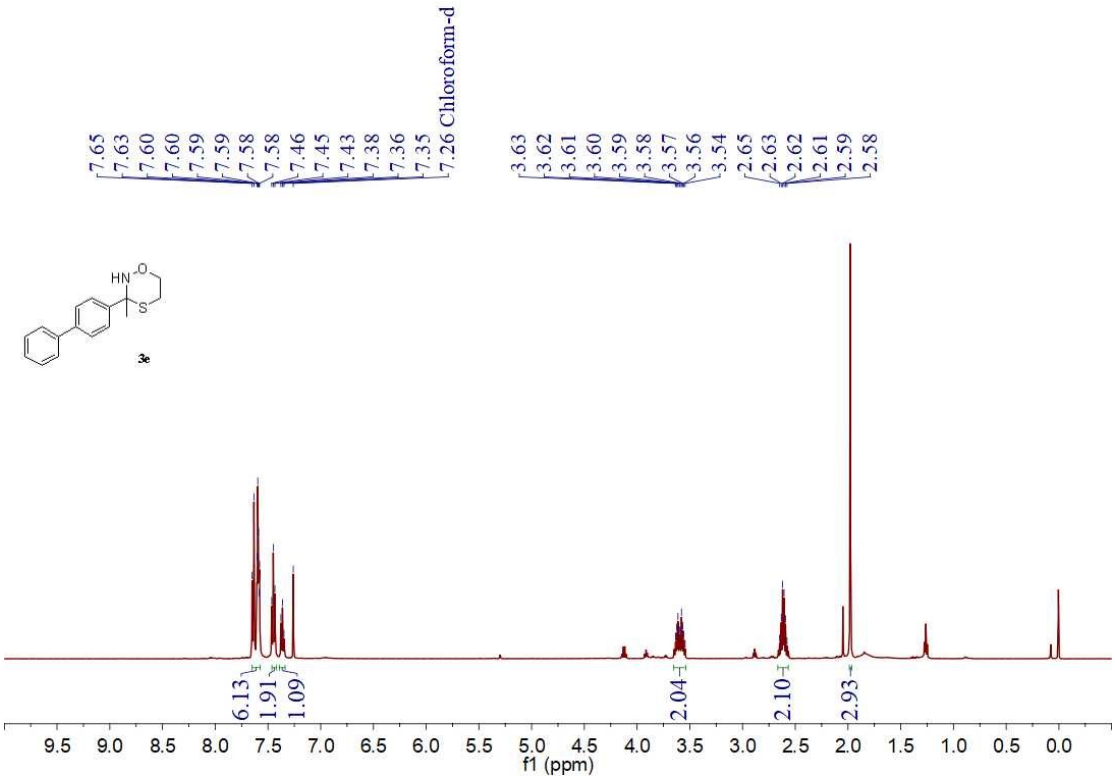
3-(4-(tert-butyl)phenyl)-3-methyl-1,4,2-oxathiazinane (**3d**).

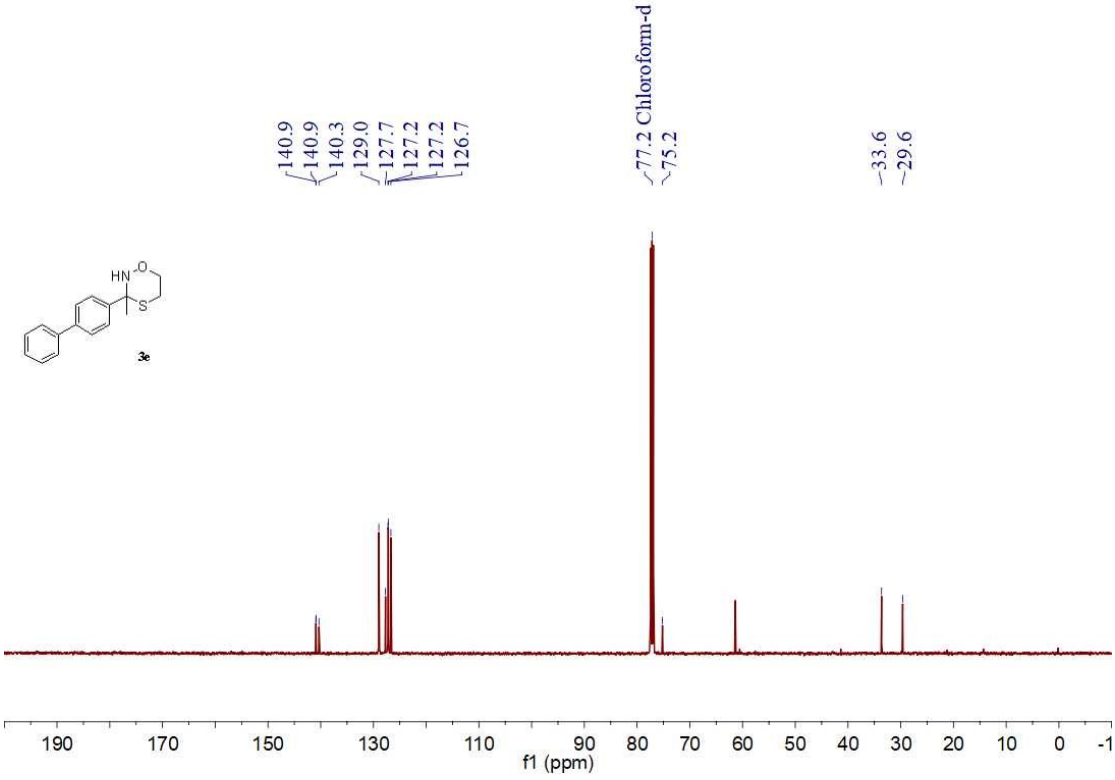


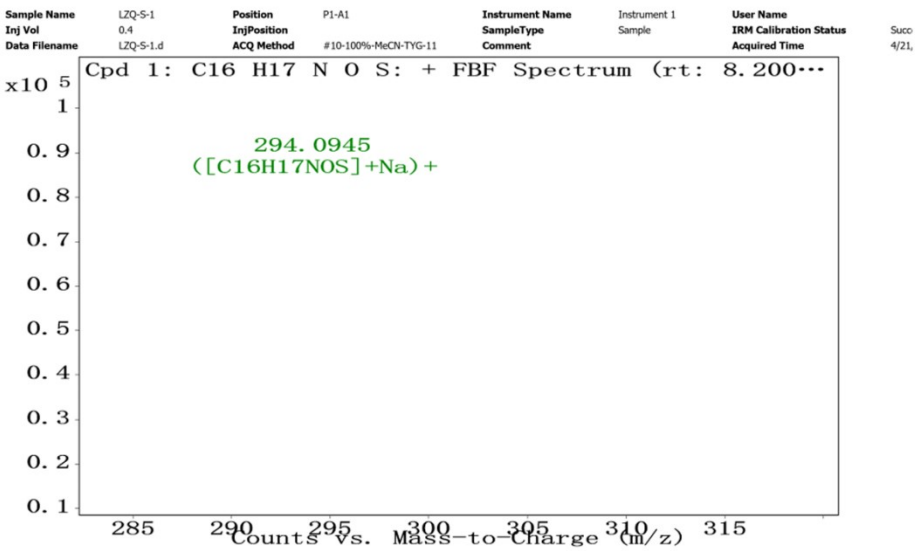




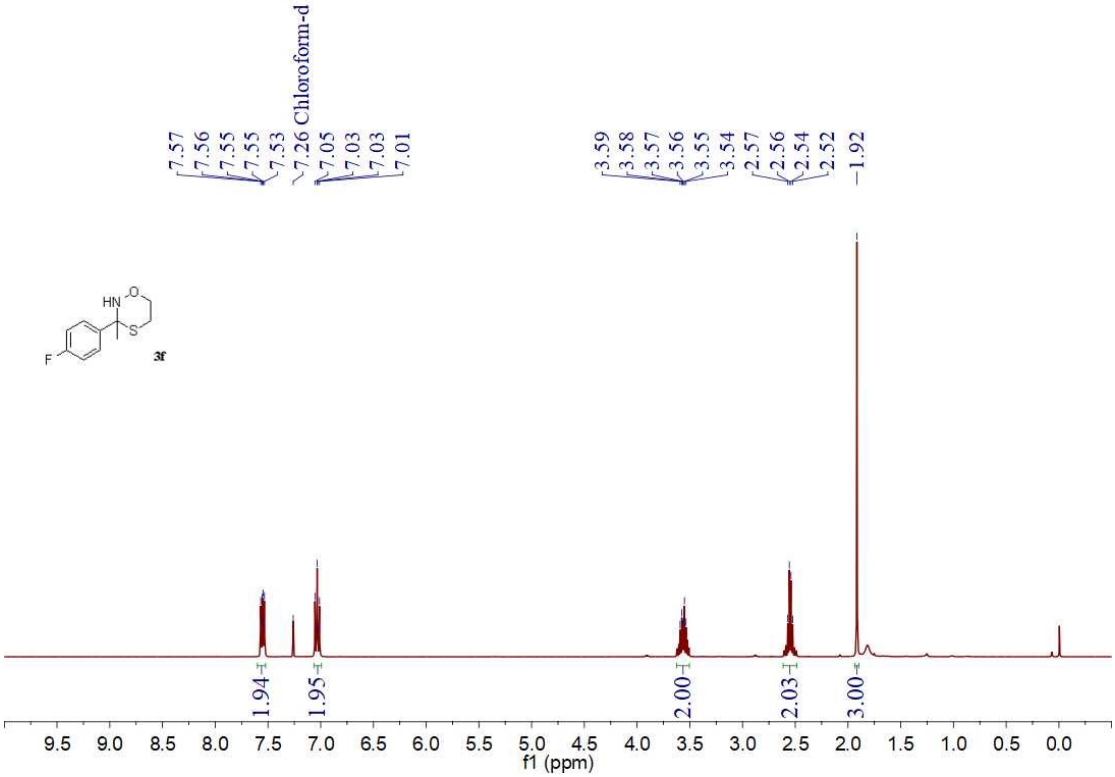
2-([1,1'-biphenyl]-4-yl)-2-methyl-1,4,3-oxathiazinane (**3e**).

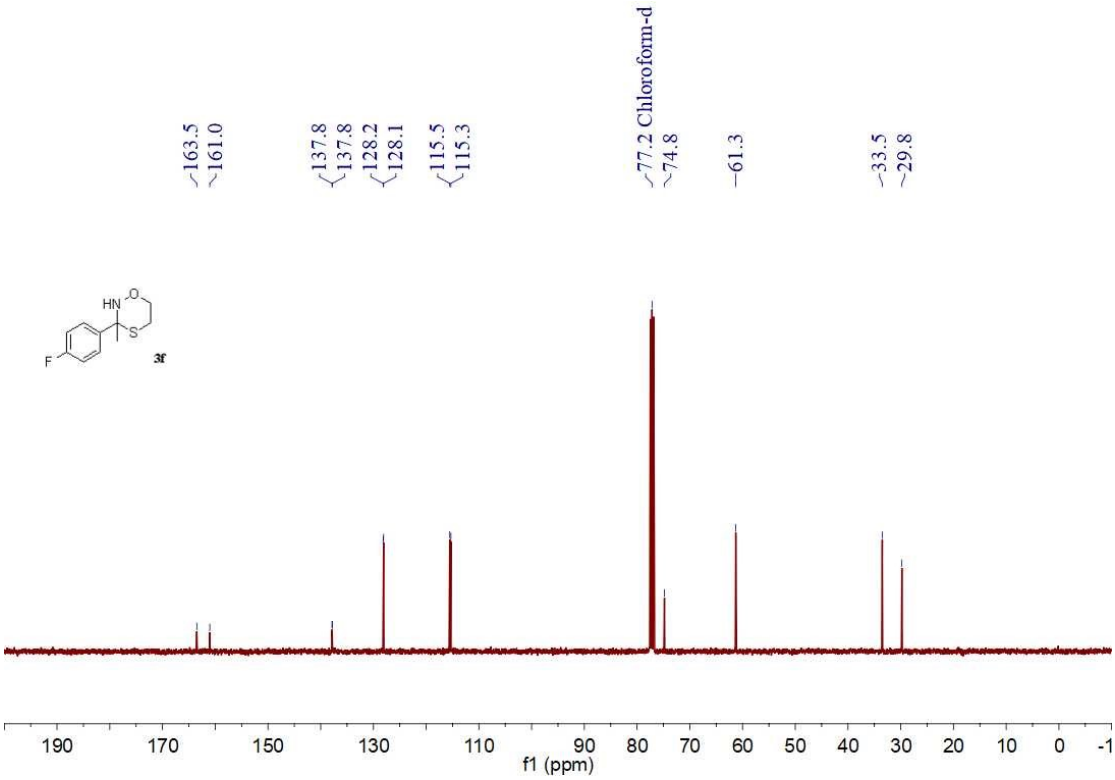

7.65
7.63
7.60
7.60
7.59
7.59
7.58
7.58
7.46
7.45
7.43
7.38
7.36
7.35
7.26 Chloroform-d
3.63
3.62
3.61
3.60
3.59
3.58
3.57
3.56
3.54
2.65
2.63
2.62
2.61
2.59
2.58
HN
O
S
3e
6.13
1.91
1.09
2.04
2.10
2.93
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

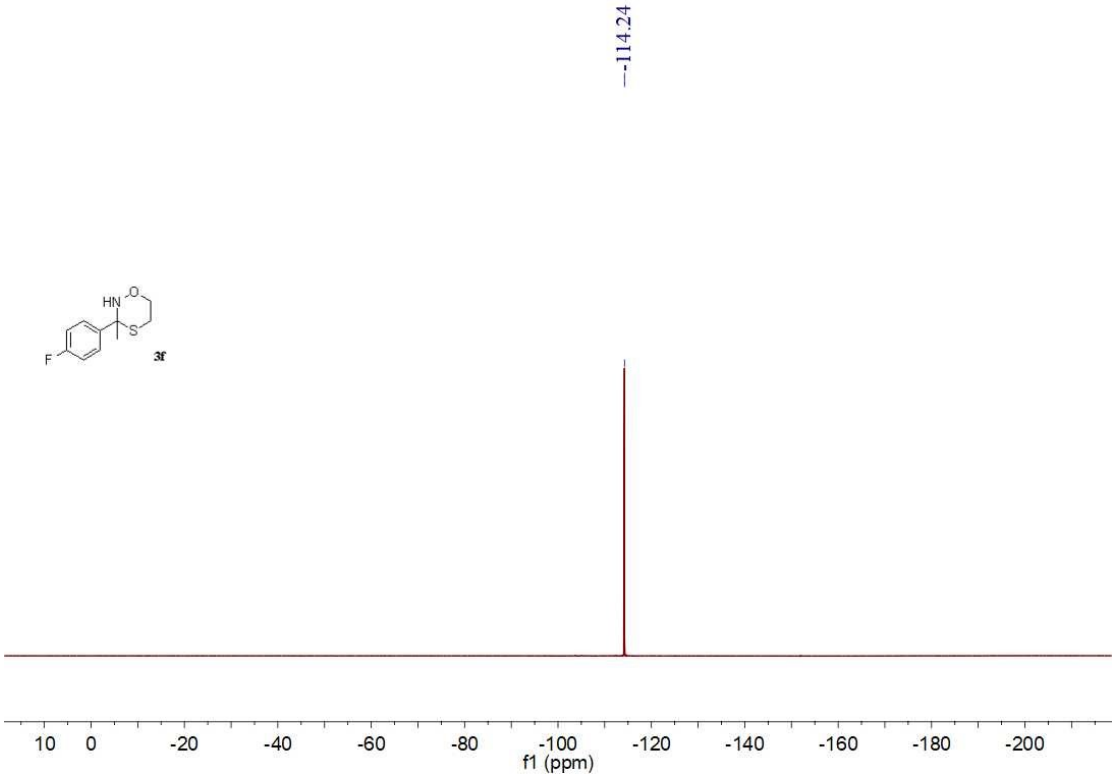

140.9
140.9
140.3
129.0
127.7
127.2
127.2
126.7
77.2 Chloroform-d
75.2
33.6
29.6
HN
O
S
3e
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

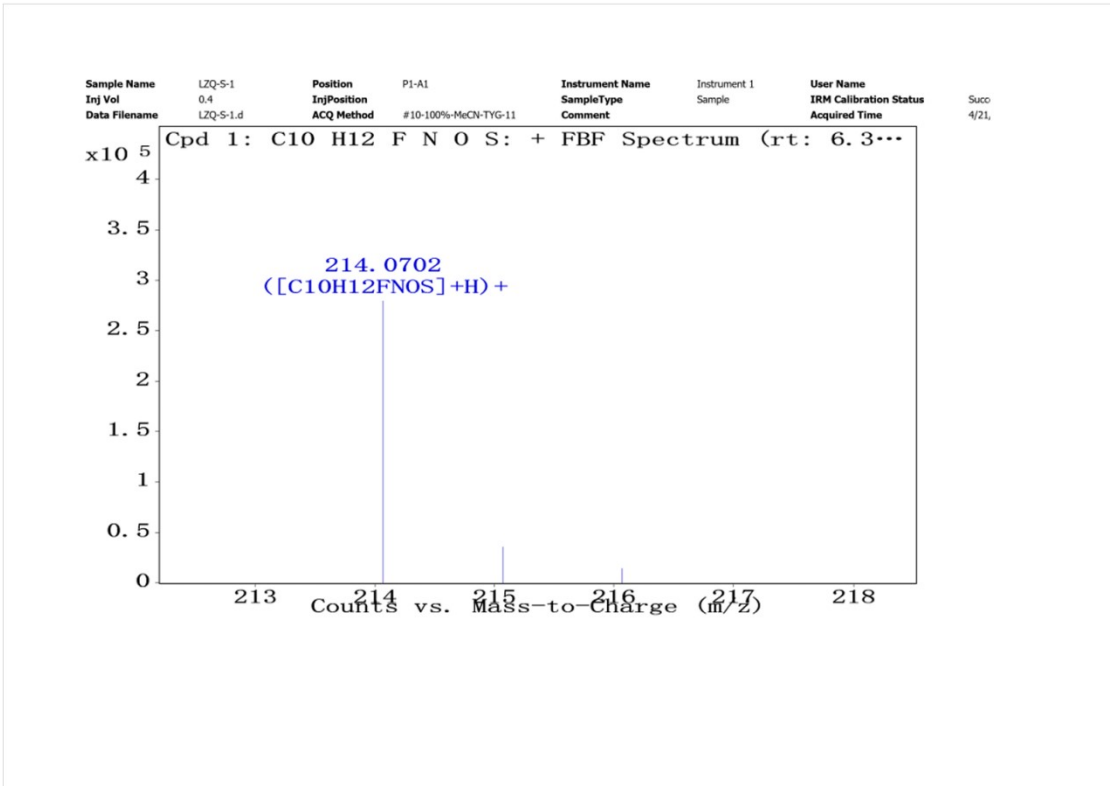


2-(4-fluorophenyl)-2-methyl-1,4,3-oxathiazinane(3f).

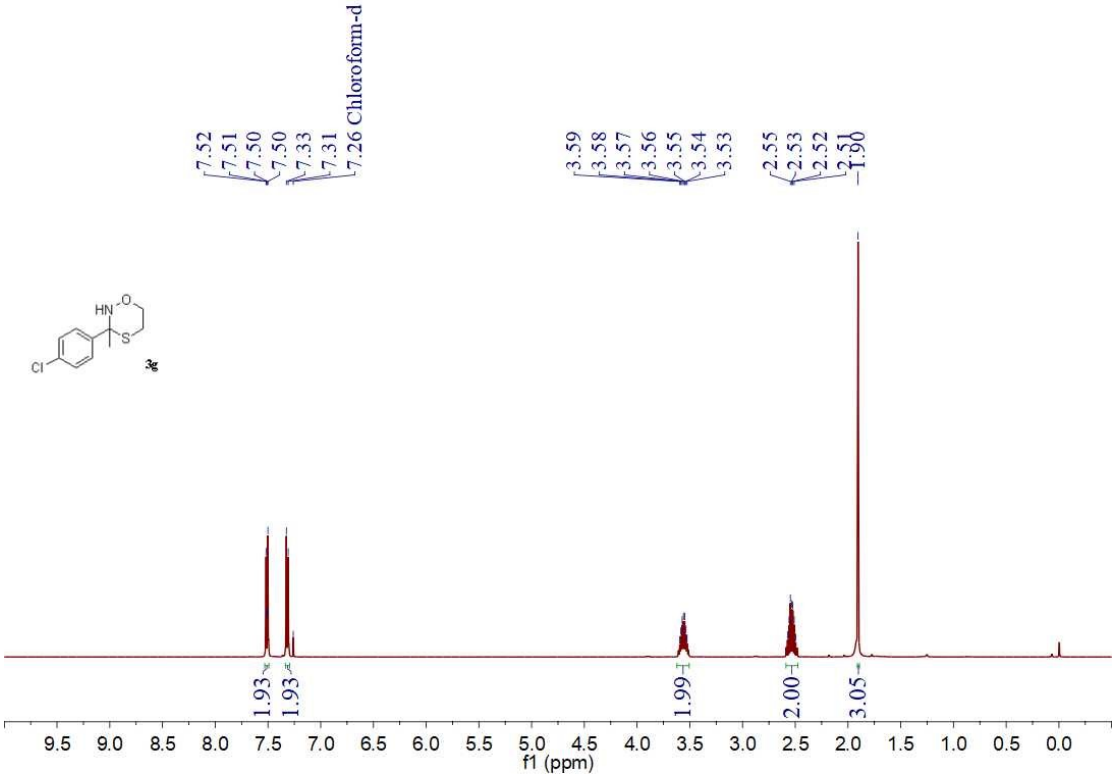


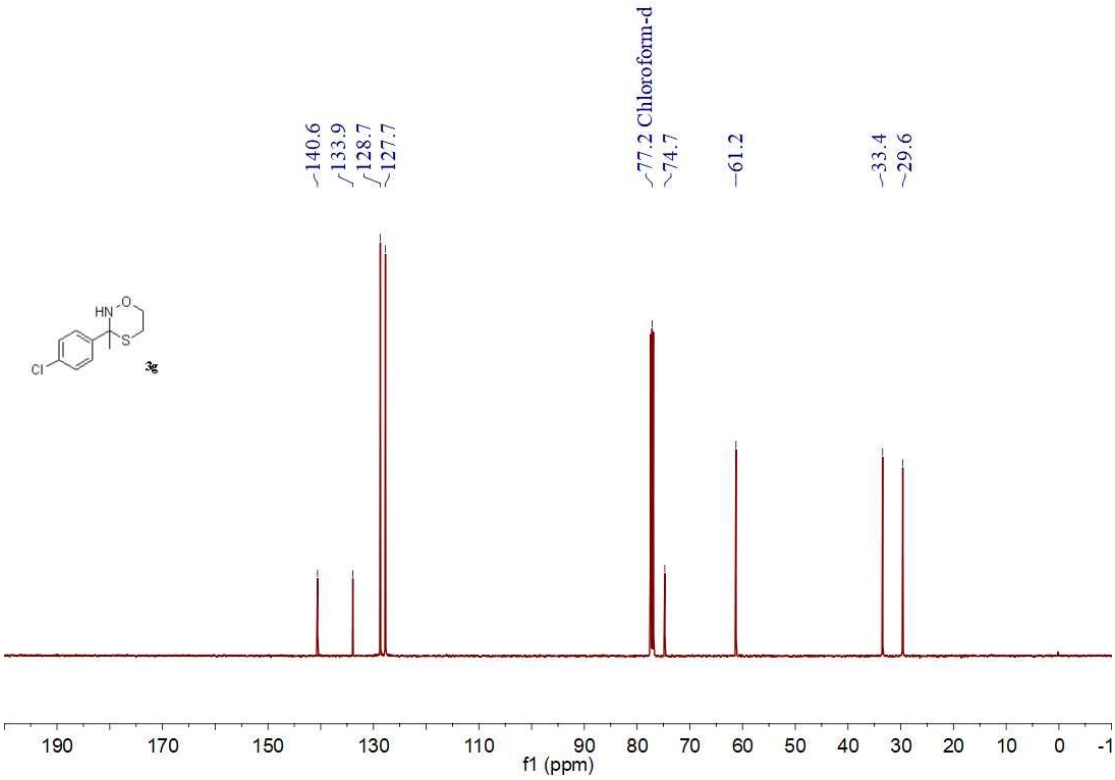

163.5
161.0
137.8
137.8
128.2
128.1
115.5
115.3
77.2 Chloroform-d
74.8
61.3
33.5
29.8
HN
O
S
F
3f
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

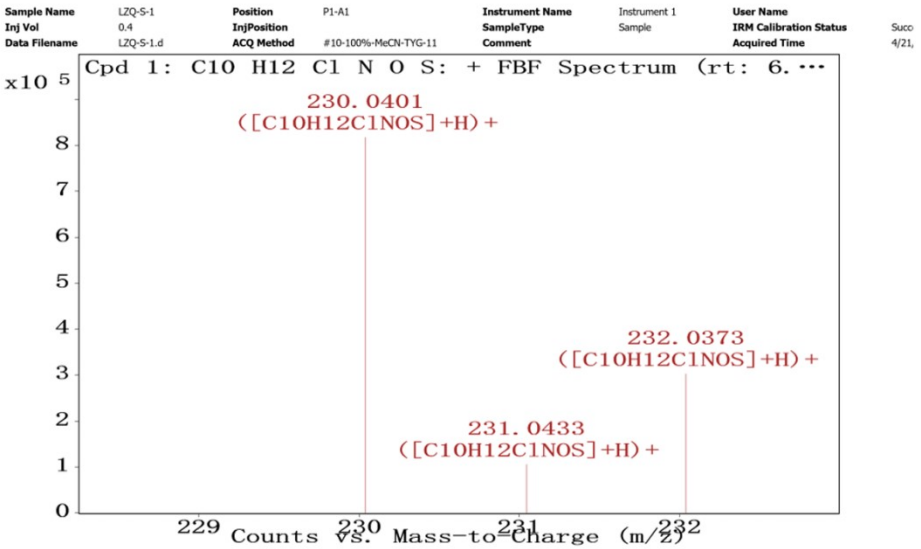

-114.24
HN
O
S
F
3f
10
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)



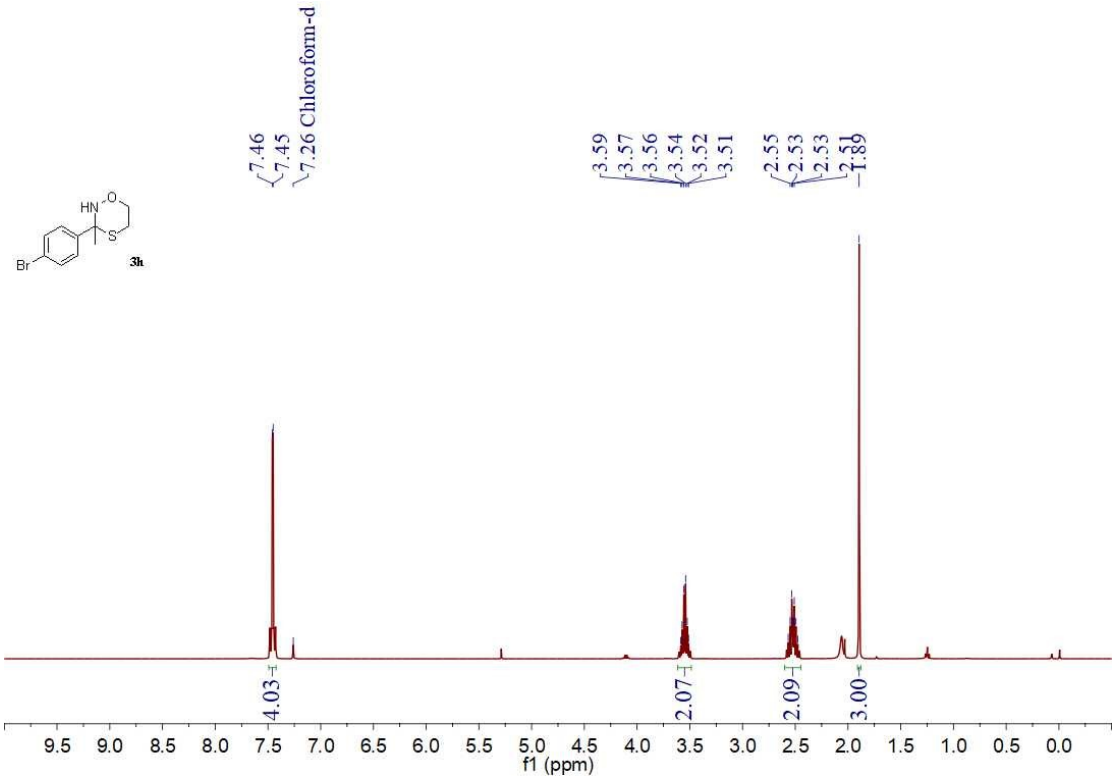
2-(4-chlorophenyl)-2-methyl-1,4,3-oxathiazinane (3g).

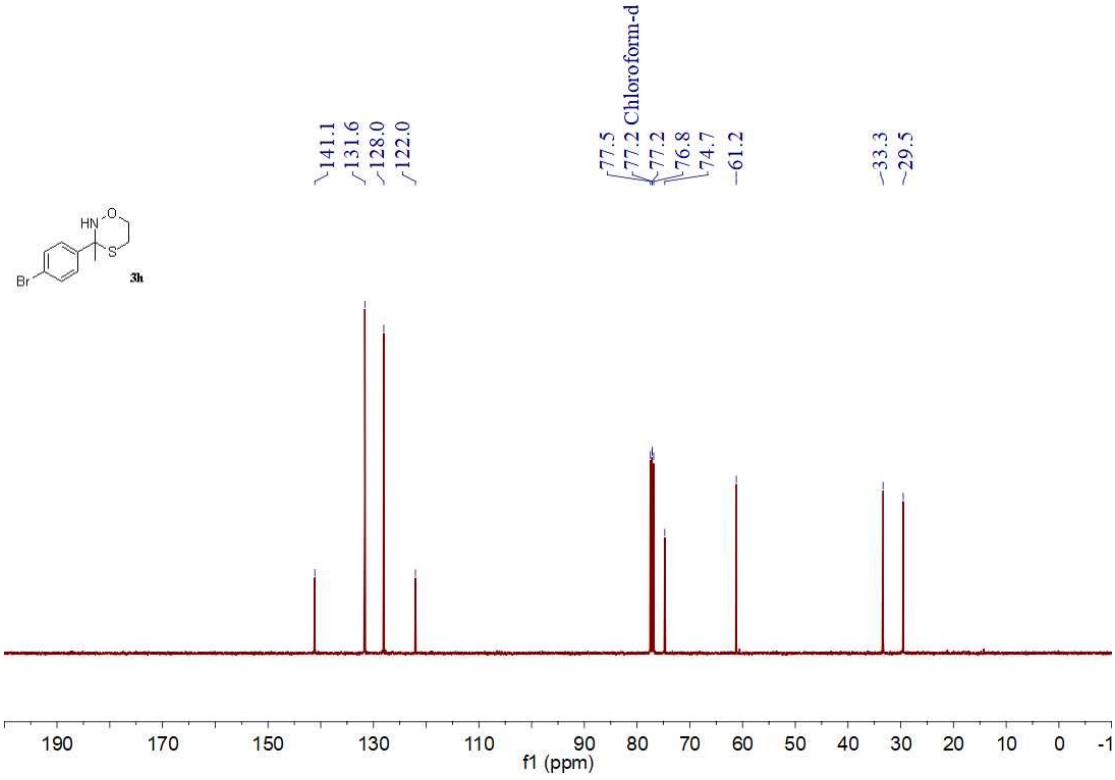


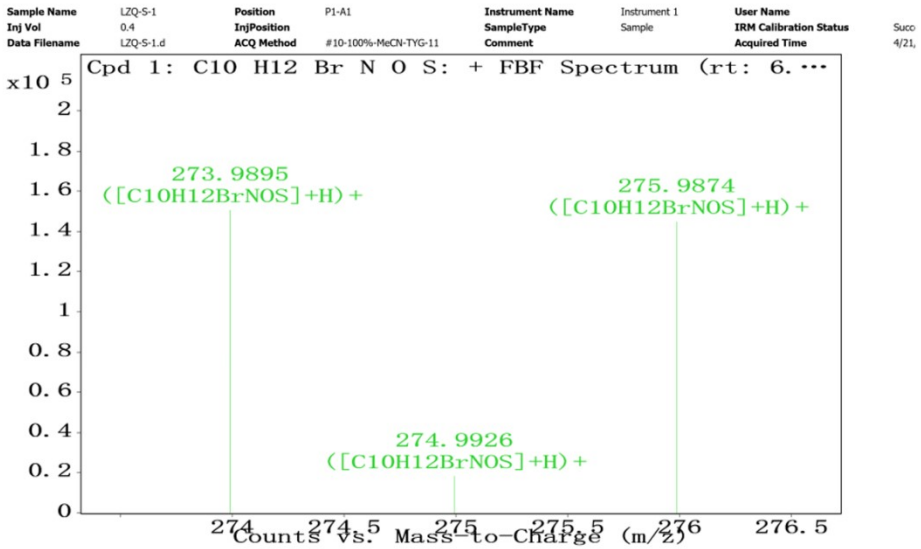




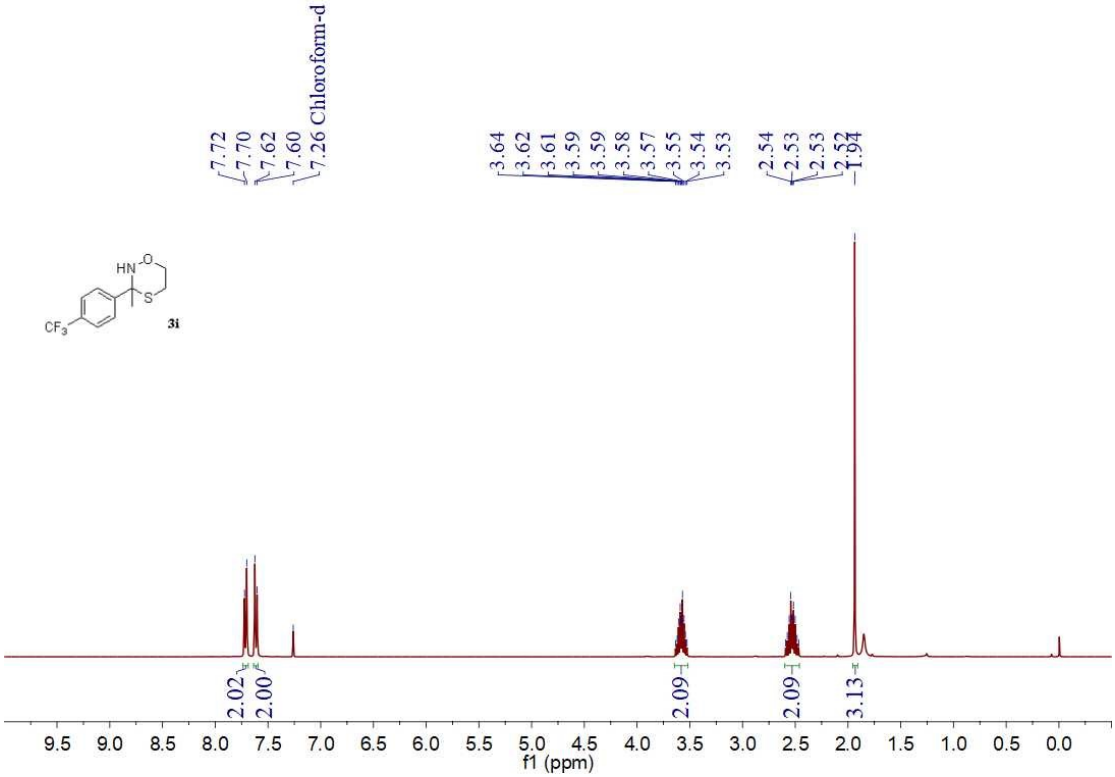
2-(4-bromophenyl)-2-methyl-1,4,3-oxathiazinane (3h).

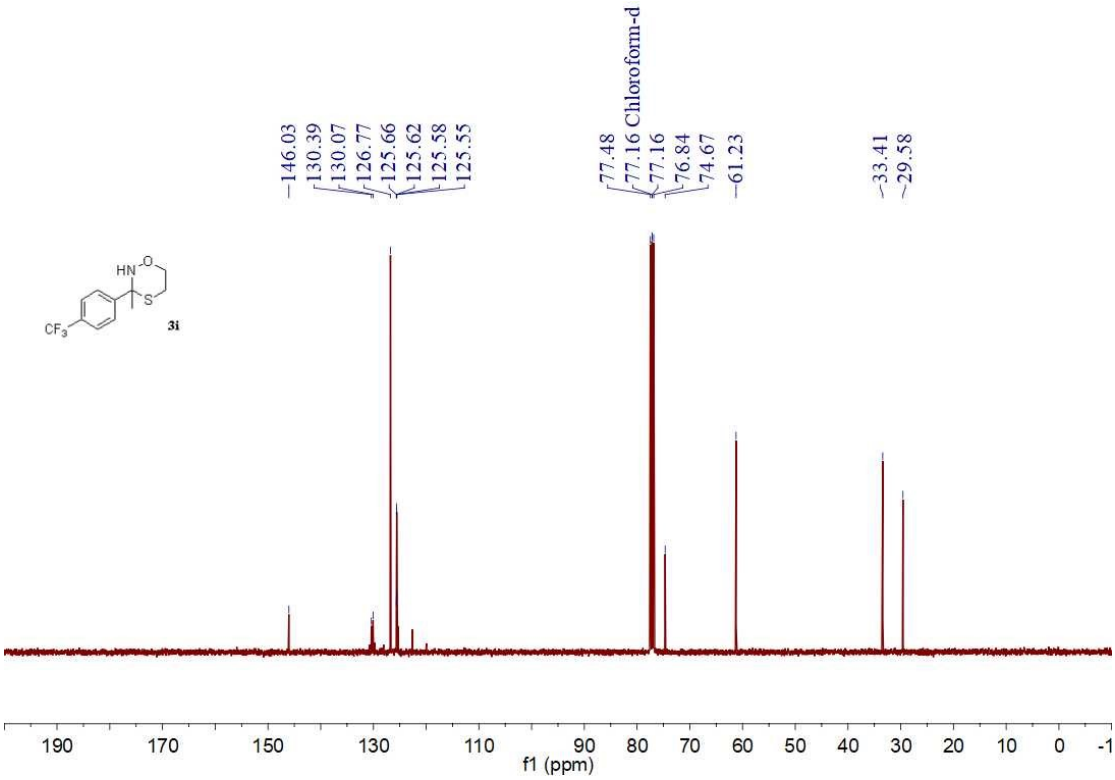

7.46
7.45
7.26 Chloroform-d
3.59
3.57
3.56
3.54
3.52
3.51
2.55
2.53
2.53
1.89
HN
O
S
Br
3h
4.03
2.07
2.09
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

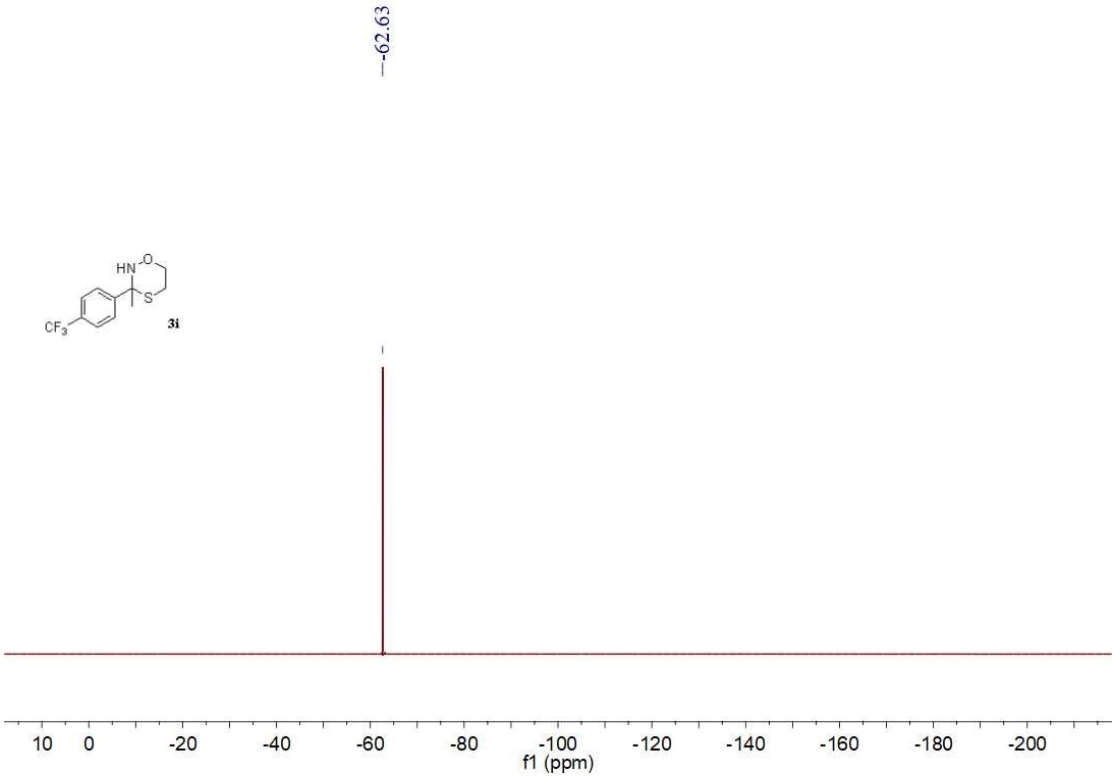

141.1
131.6
128.0
122.0
77.5
77.2 Chloroform-d
77.2
76.8
74.7
61.2
33.3
29.5
HN
O
S
Br
3h
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

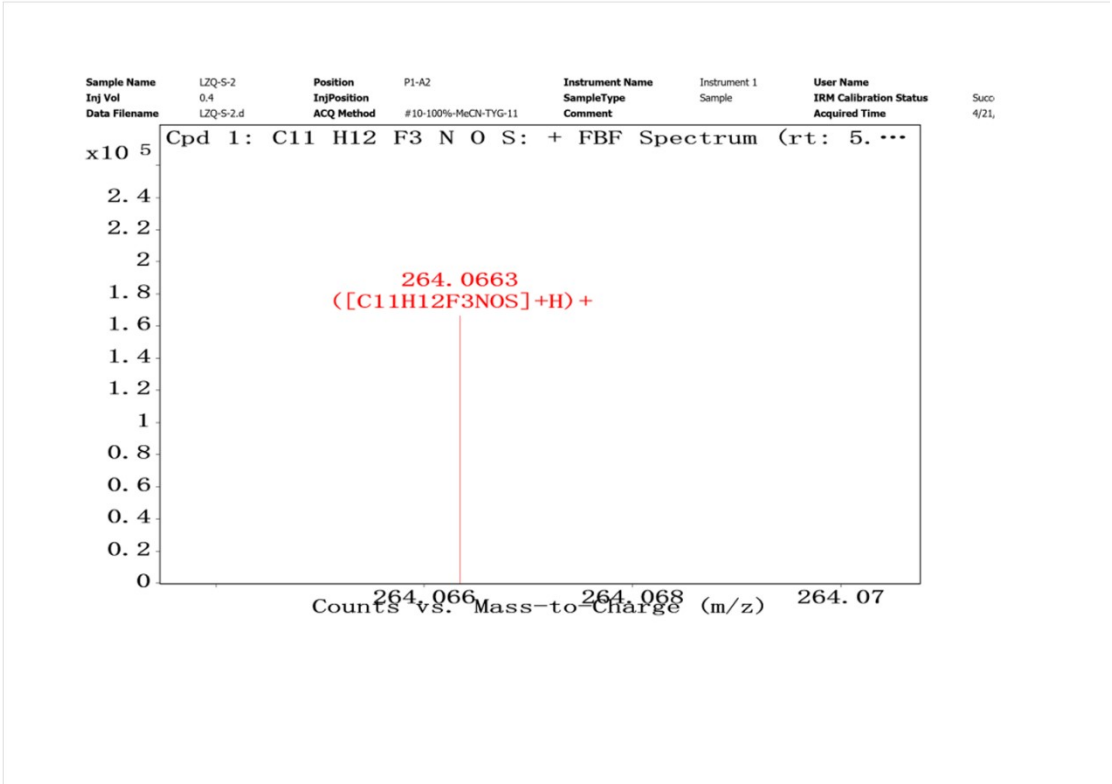


2-methyl-2-(4-(trifluoromethyl)phenyl)-1,4,3-oxathiazinane (3i).

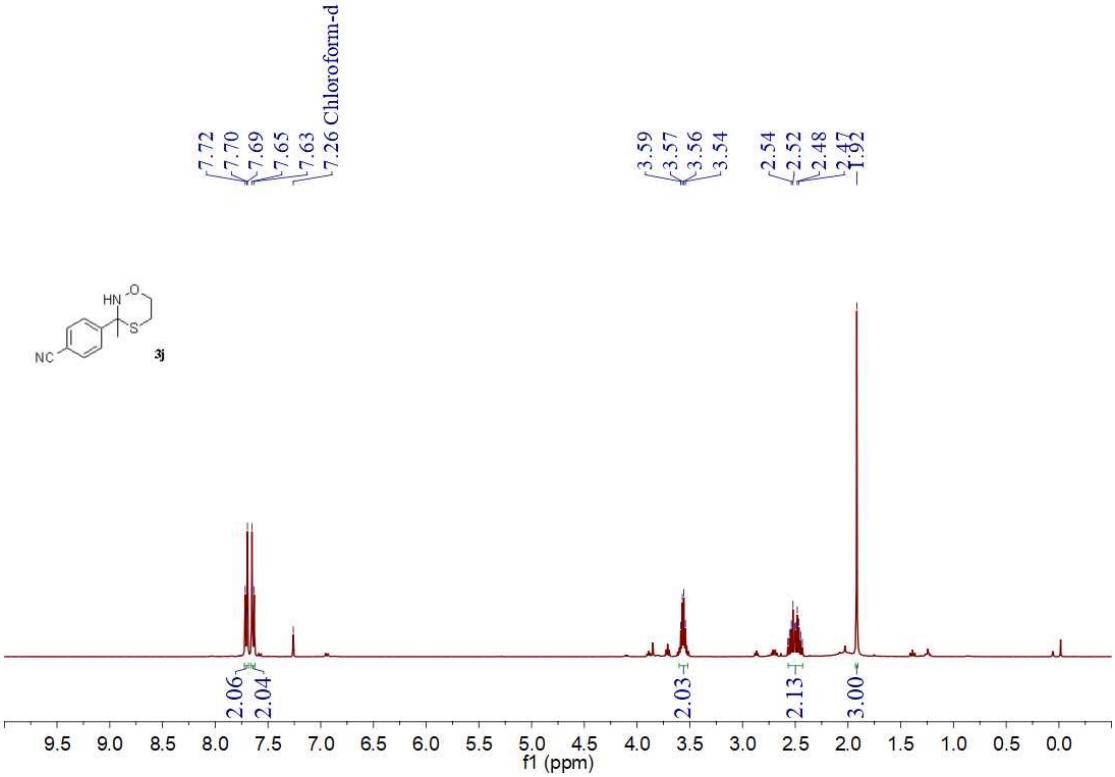


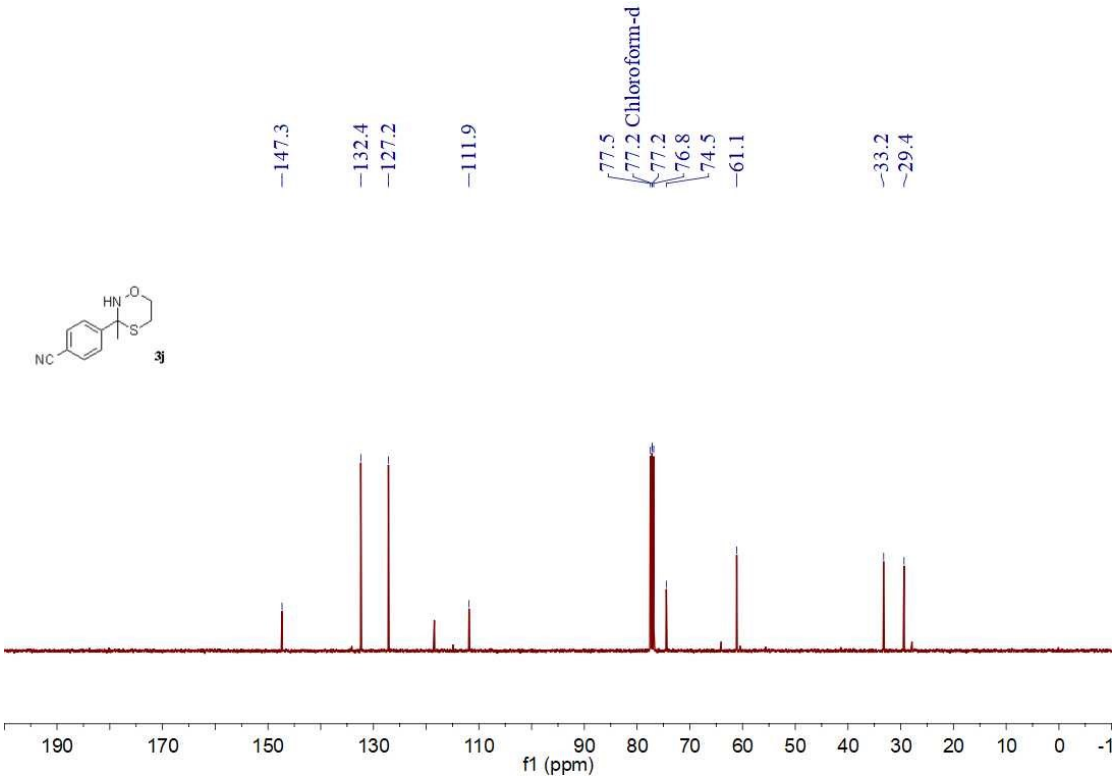

146.03
130.39
130.07
126.77
125.66
125.62
125.58
125.55
77.48
77.16 Chloroform-d
77.16
76.84
74.67
61.23
33.41
29.58
HN
O
S
CF3
3i
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

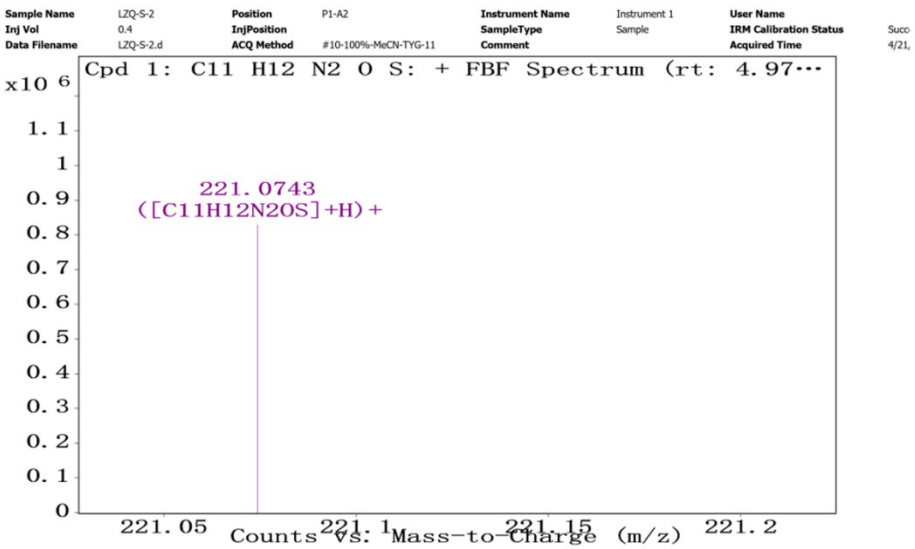

-62.63
HN
O
S
CF3
3i
10
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)



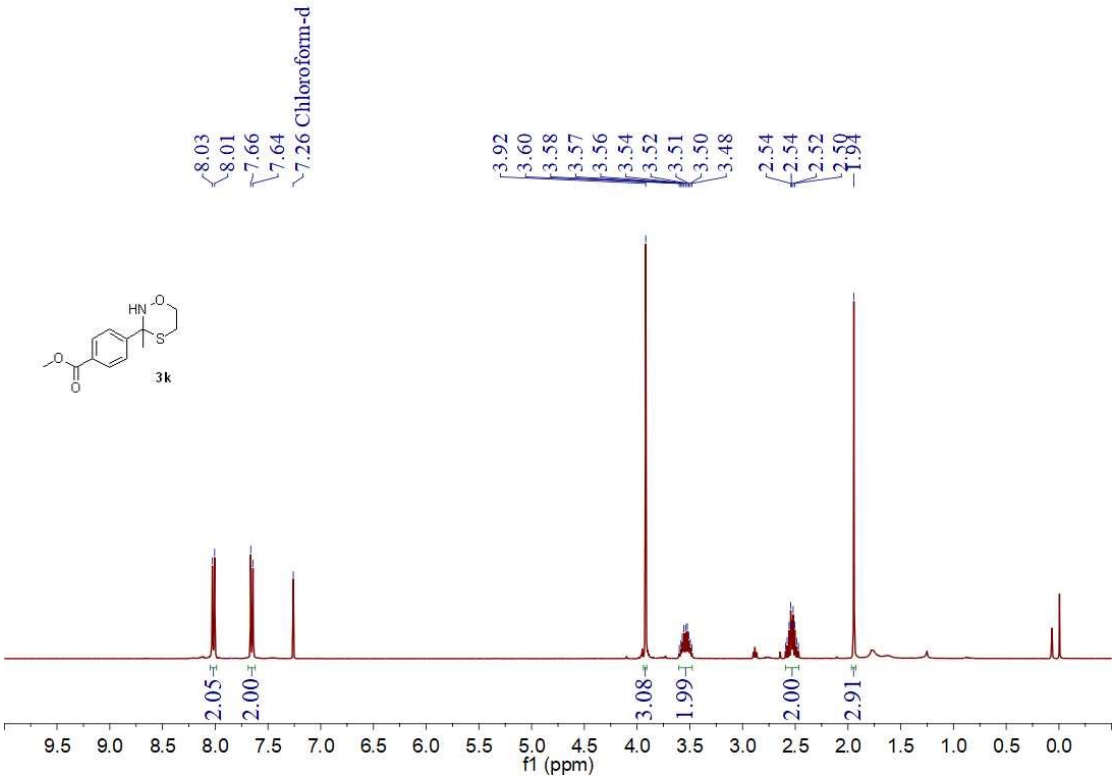
(2-methyl-1,4,3-oxathiazinan-2-yl)benzonitrile (3j).

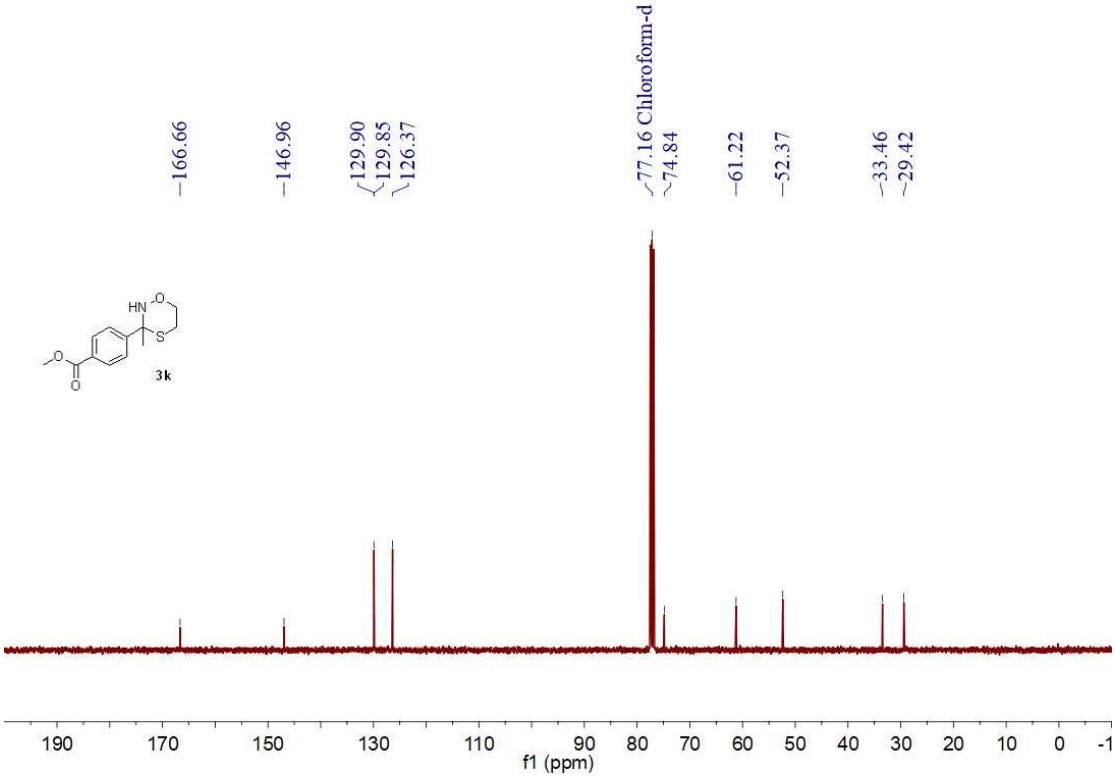


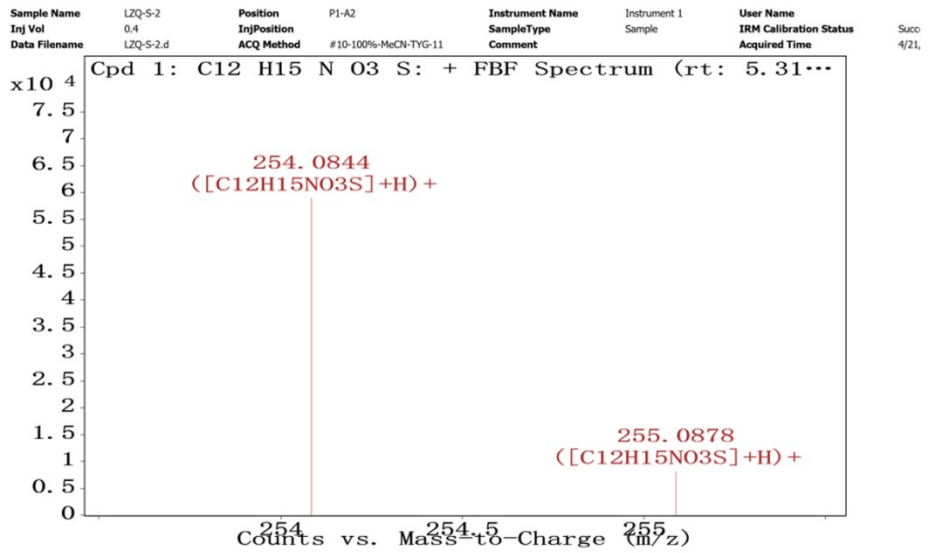




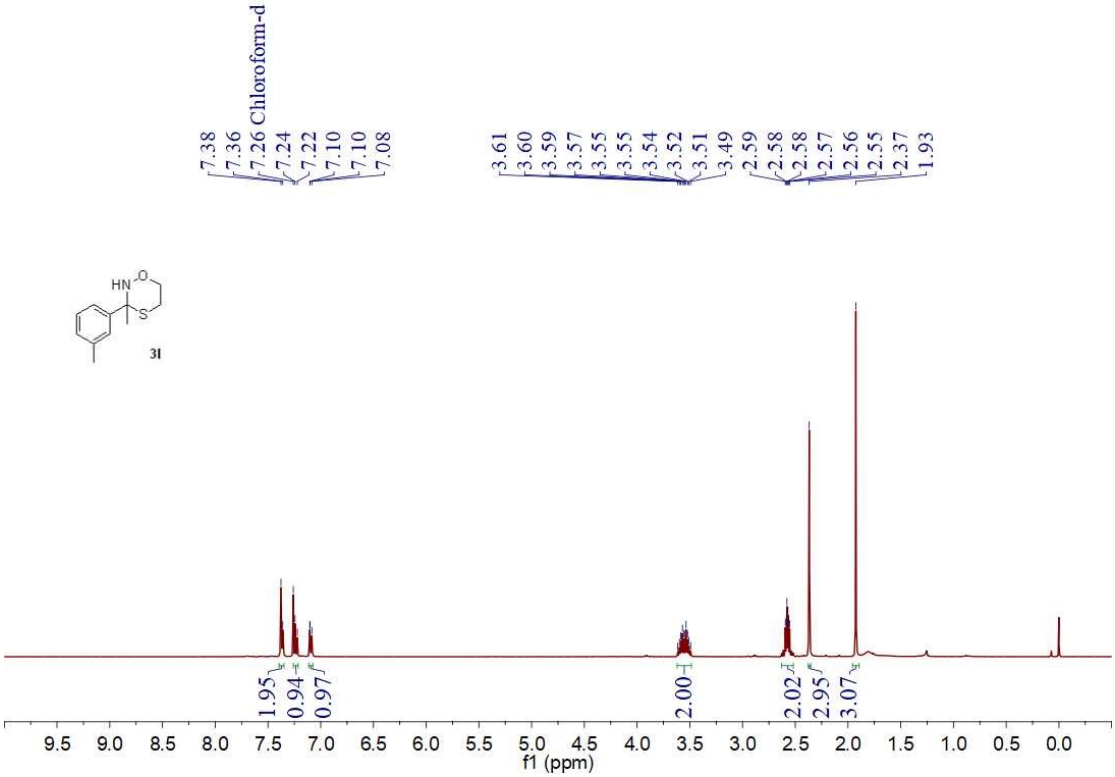
methyl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3k).

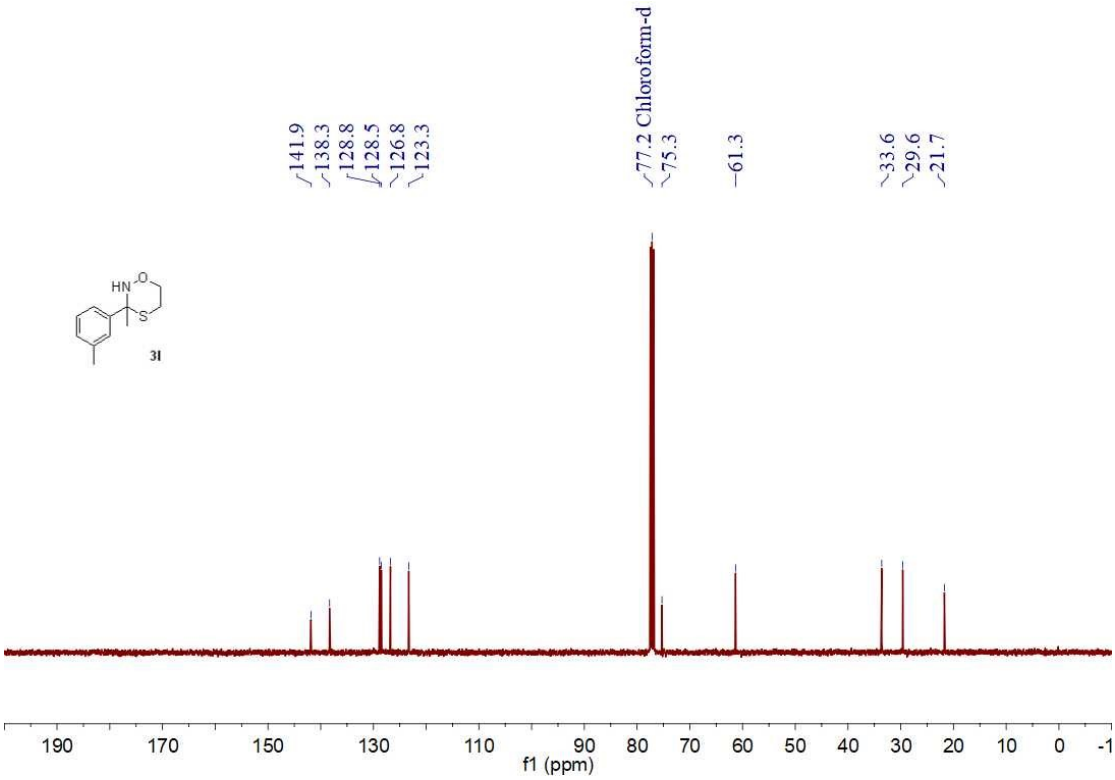

8.03
8.01
7.66
7.64
7.26 Chloroform-d
3.92
3.60
3.58
3.57
3.56
3.54
3.52
3.51
3.50
3.48
2.54
2.54
2.52
2.50
1.94
HN
O
S
O
O
3k
2.05
2.00
3.08
1.99
2.00
2.91
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

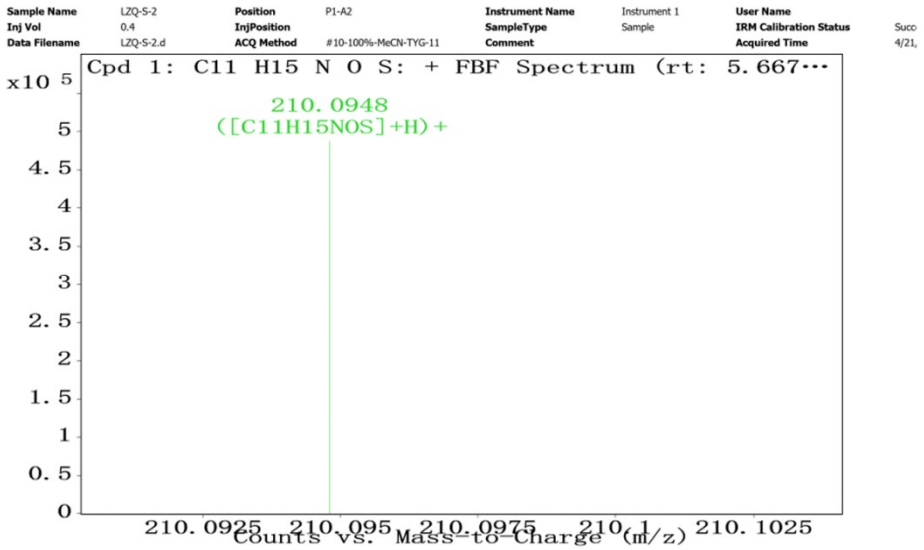

166.66
146.96
129.90
129.85
126.37
77.16 Chloroform-d
74.84
61.22
52.37
33.46
29.42
HN
O
S
O
O
3k
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



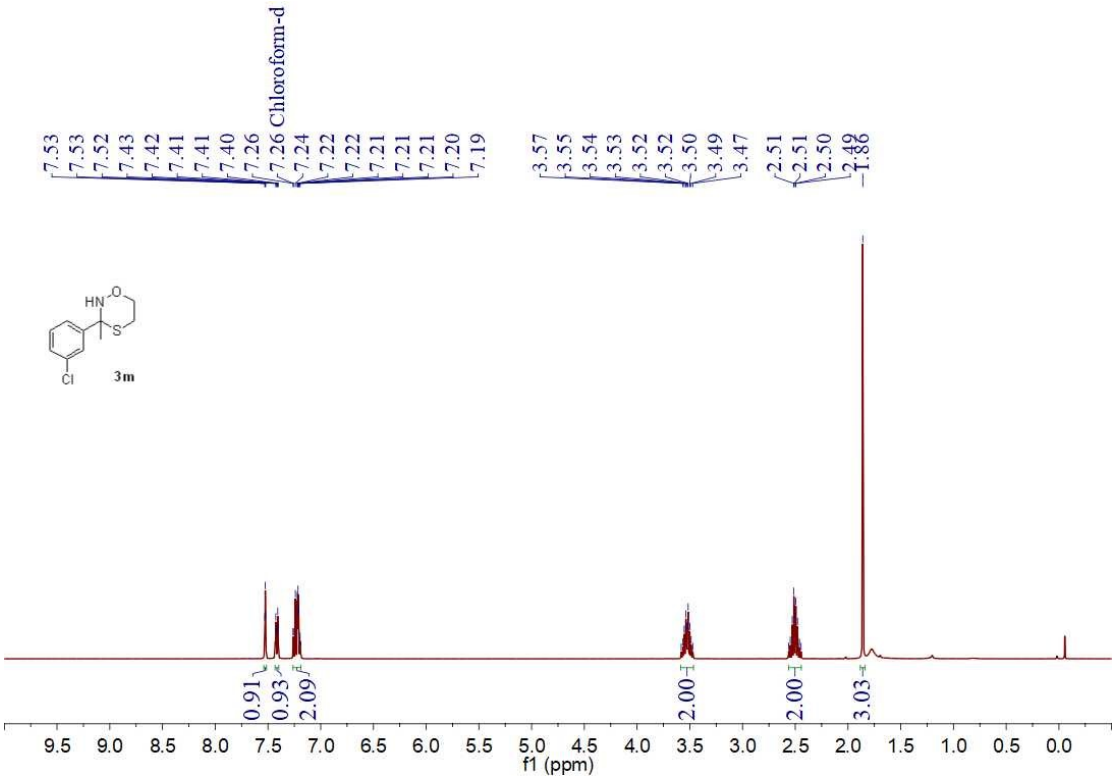
2-methyl-2-(m-tolyl)-1,4,3-oxathiazinane (3l).

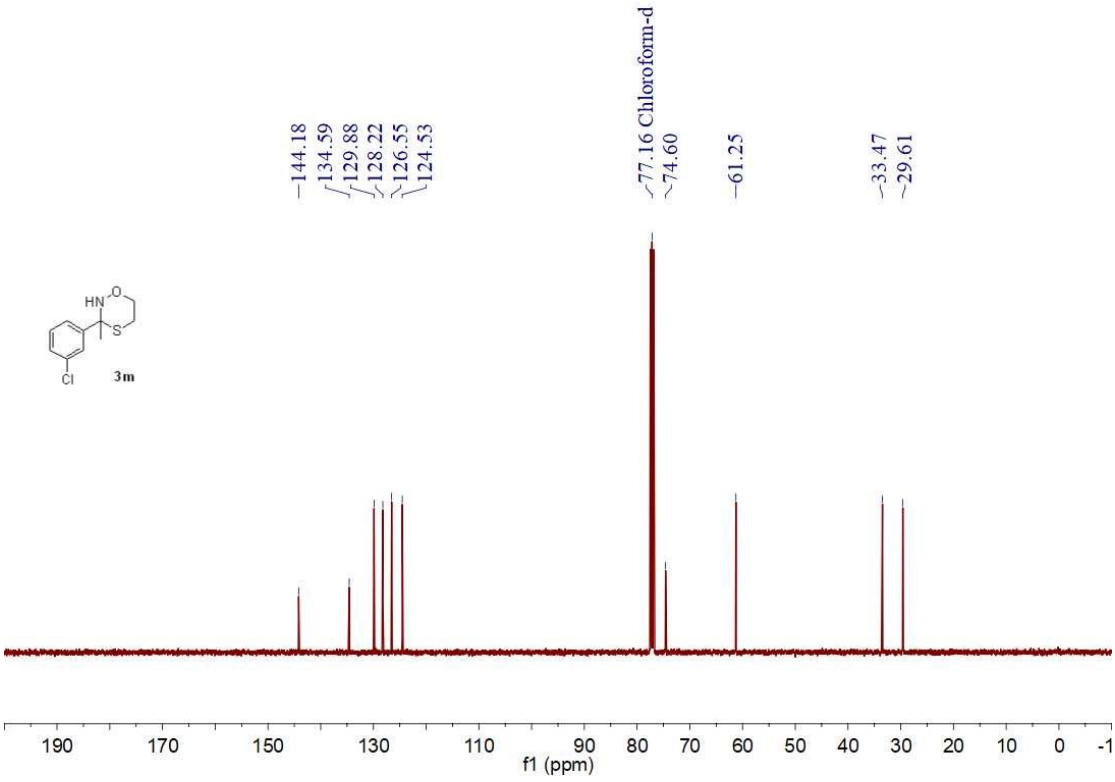


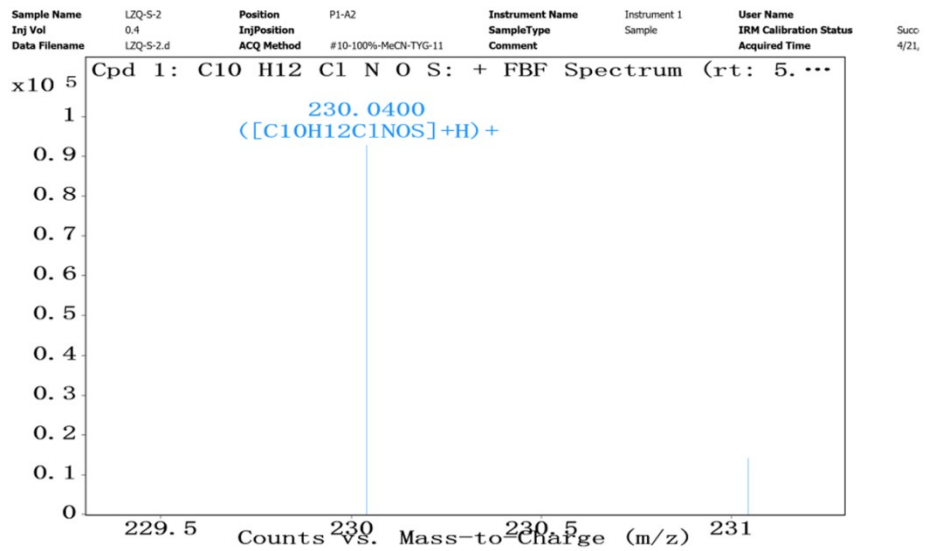




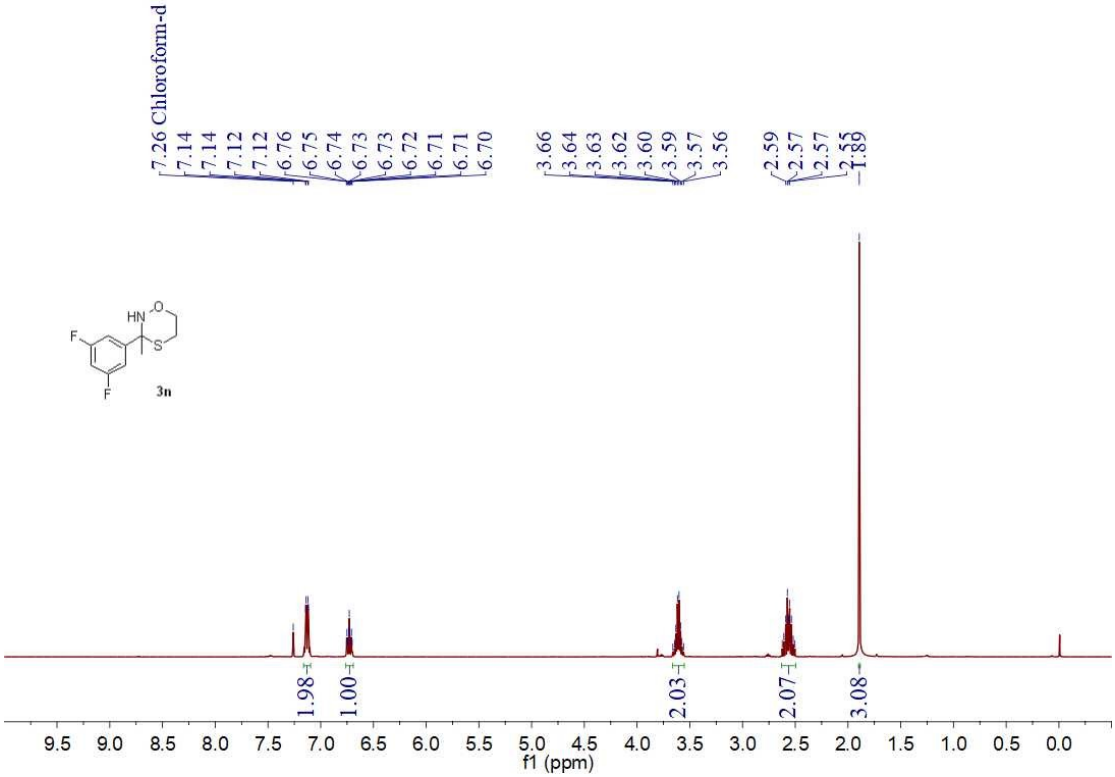
2-(3-chlorophenyl)-2-methyl-1,4,3-oxathiazinane (3m).

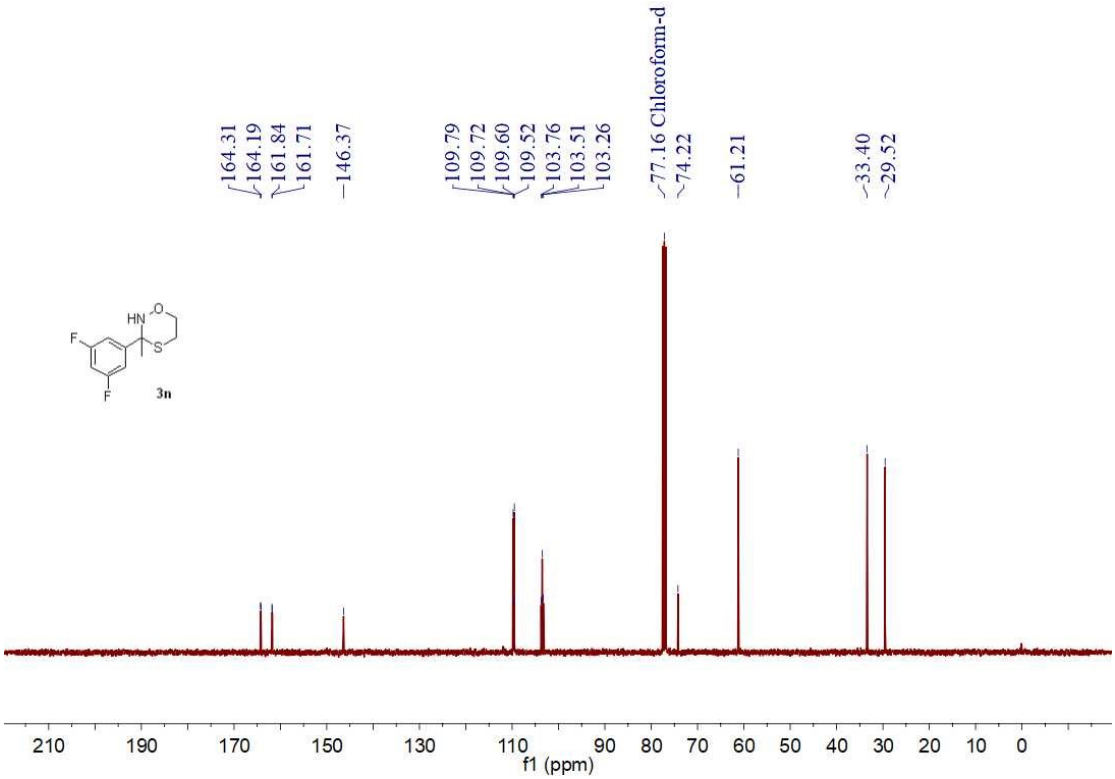

7.53
7.53
7.52
7.43
7.42
7.41
7.41
7.40
7.26 Chloroform-d
7.24
7.22
7.22
7.21
7.21
7.21
7.20
7.19
3.57
3.55
3.54
3.53
3.52
3.52
3.50
3.49
3.47
2.51
2.51
2.50
2.49
1.88
HN
O
S
Cl
3m
0.91
0.93
2.09
2.00
2.00
3.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

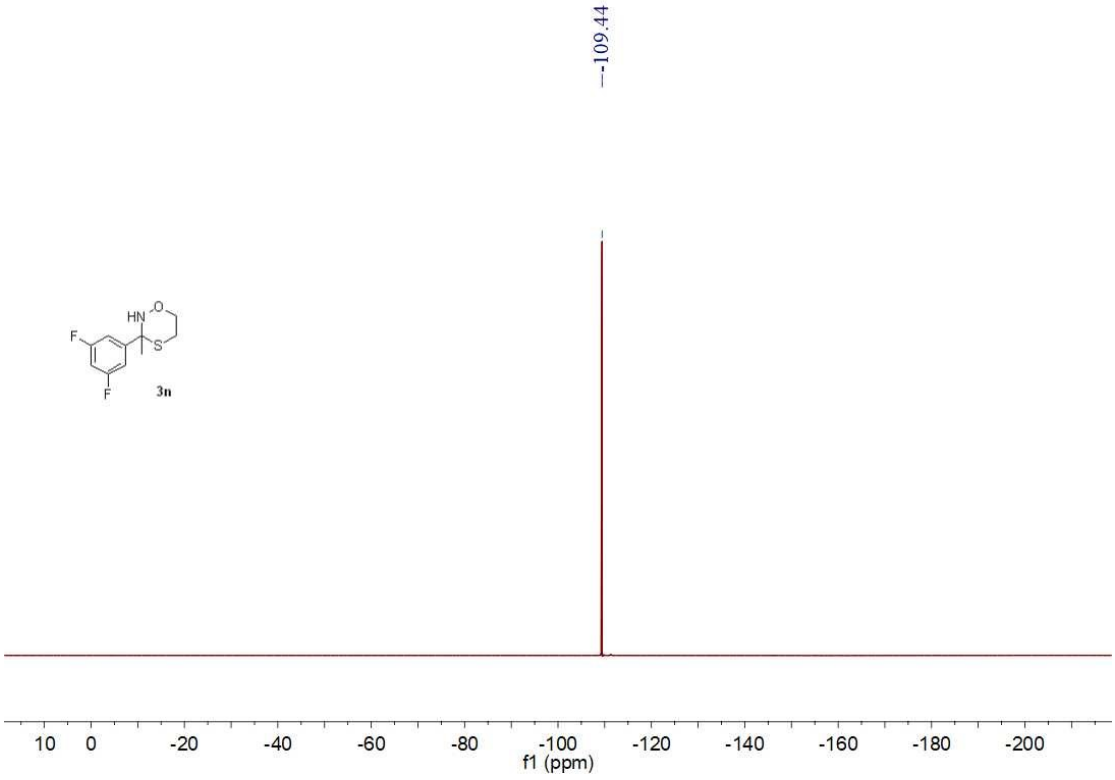

144.18
134.59
129.88
128.22
126.55
124.53
77.16 Chloroform-d
74.60
61.25
33.47
29.61
HN
O
S
Cl
3m
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

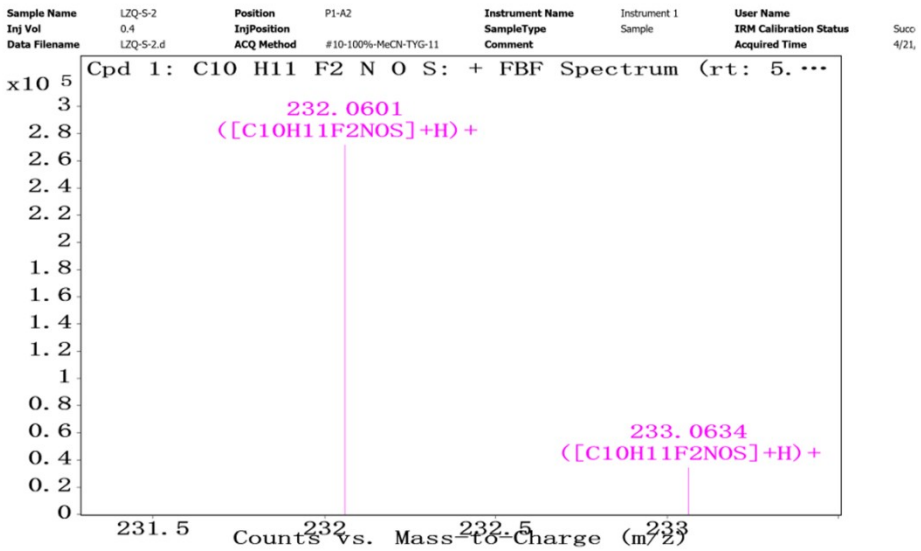


2-(3,5-difluorophenyl)-2-methyl-1,4,3-oxathiazinane (3n).

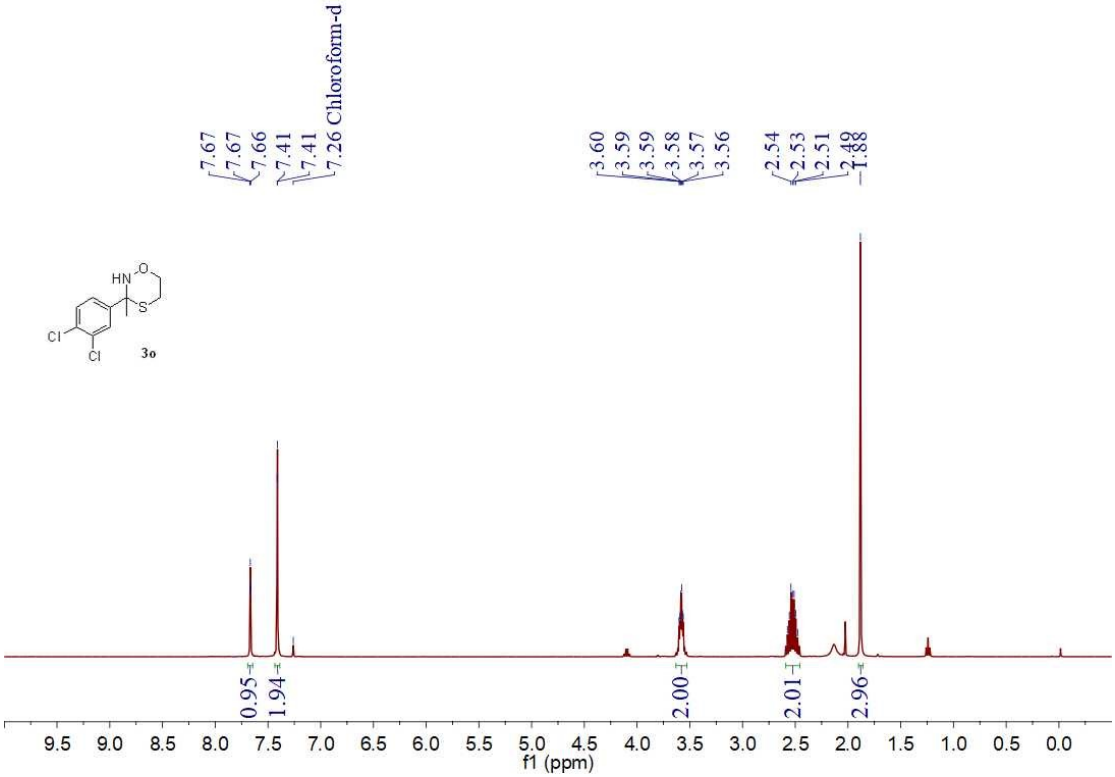


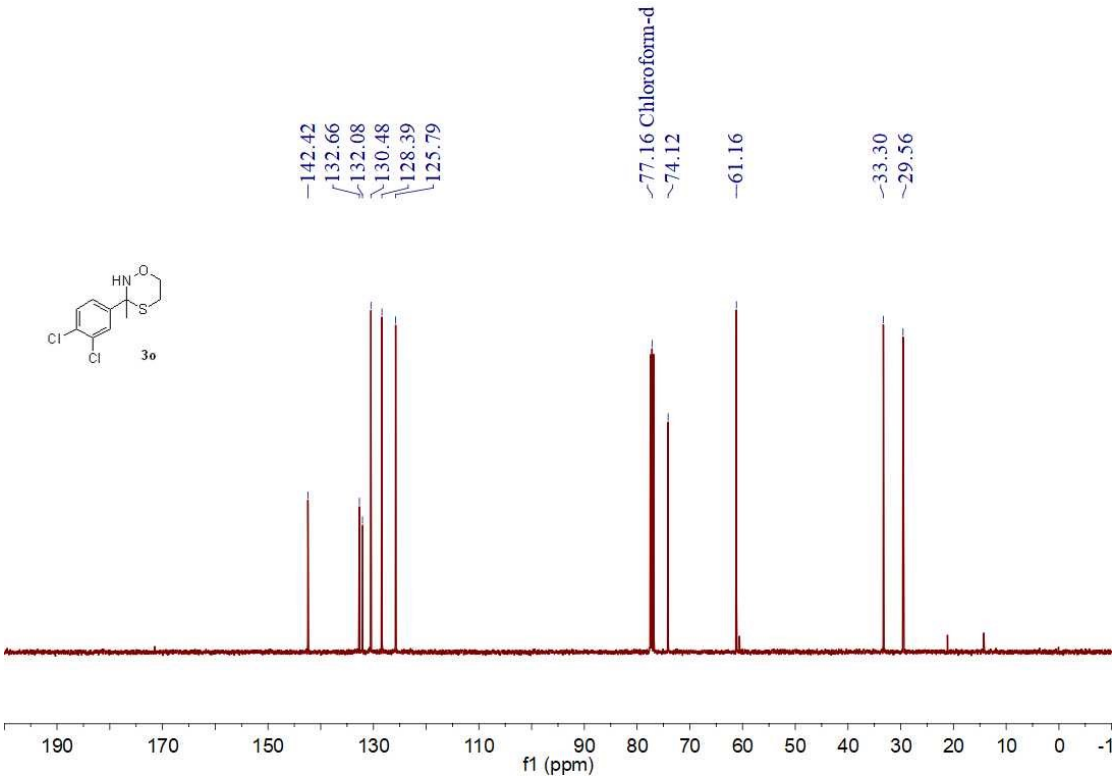

164.31
164.19
161.84
161.71
146.37
109.79
109.72
109.60
109.52
103.76
103.51
103.26
77.16 Chloroform-d
74.22
61.21
33.40
29.52
HN
O
S
F
F
3n
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

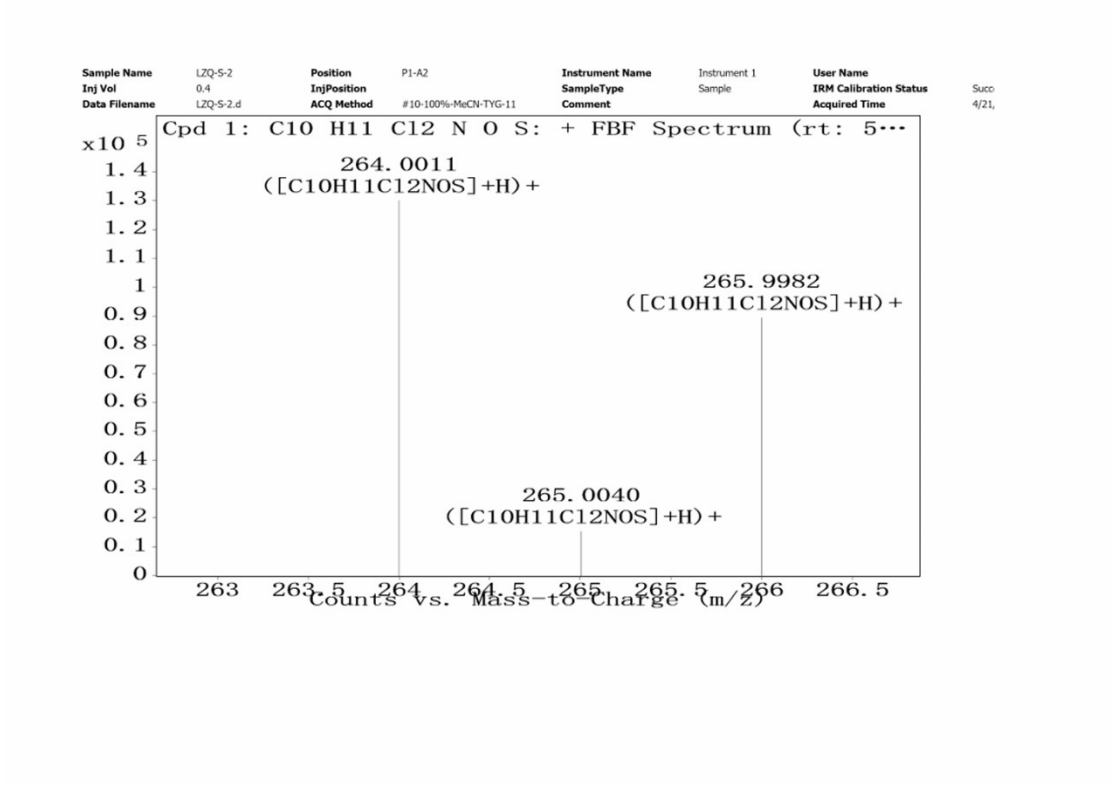

-109.44
HN
O
S
F
F
3n
10
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)



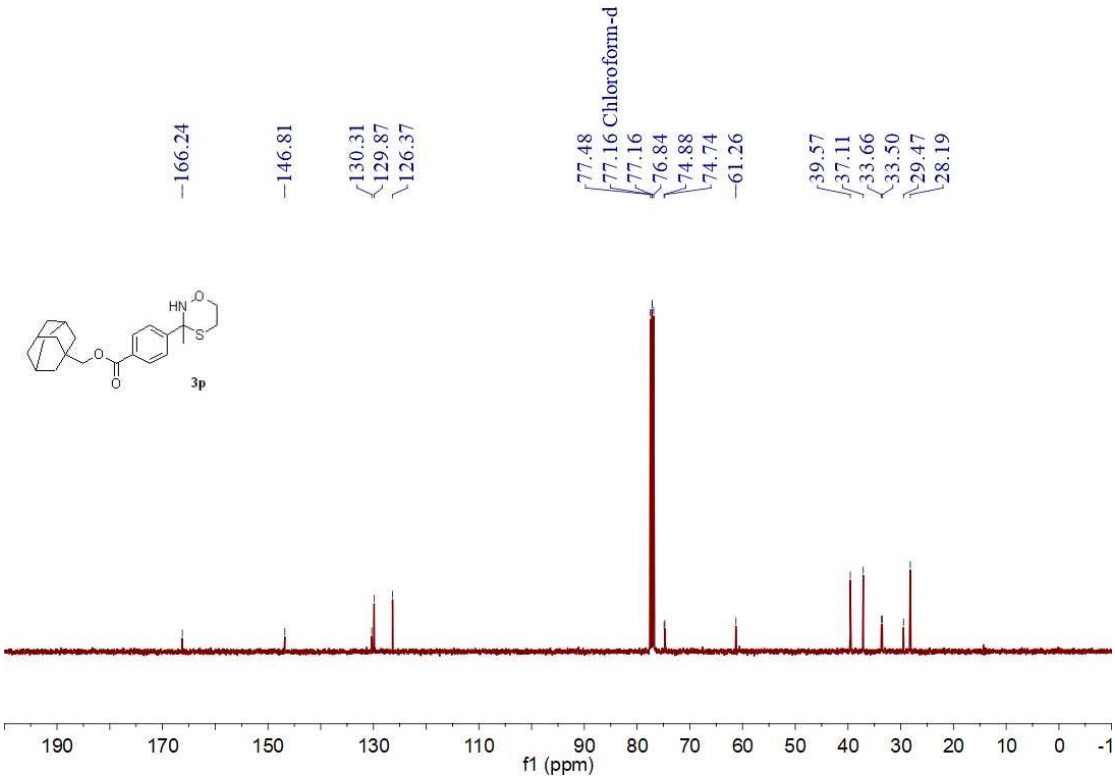
2-(3,4-dichlorophenyl)-2-methyl-1,4,3-oxathiazinane (3o).

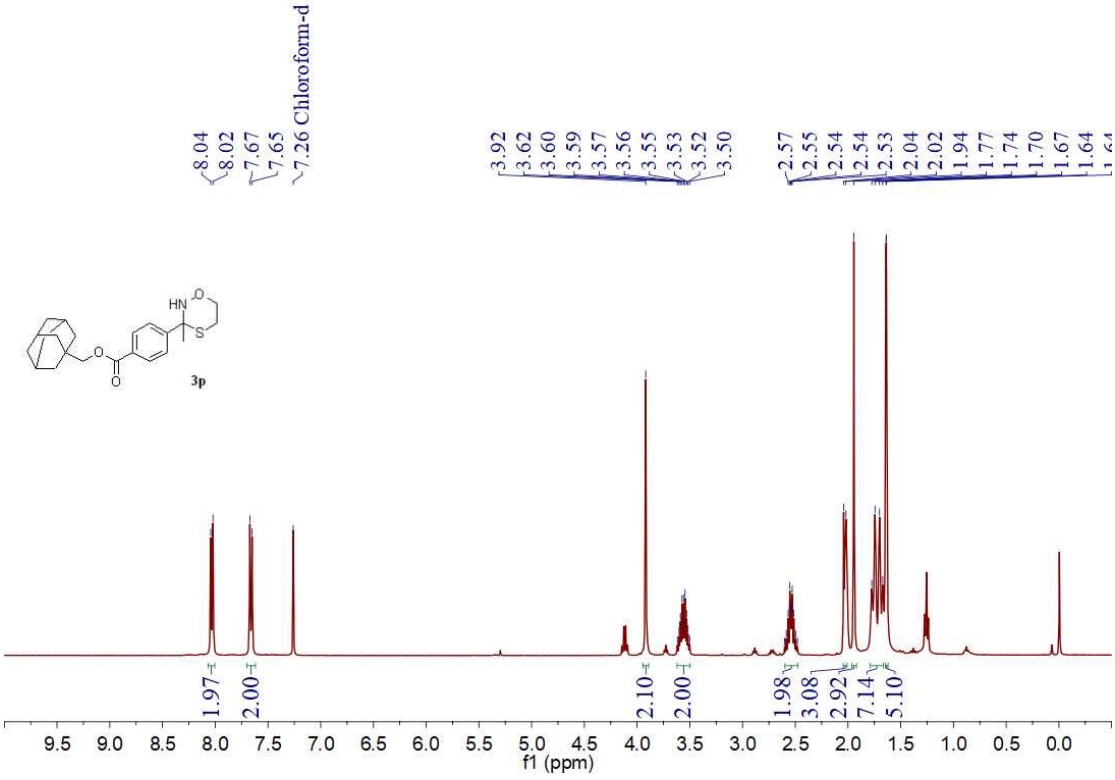


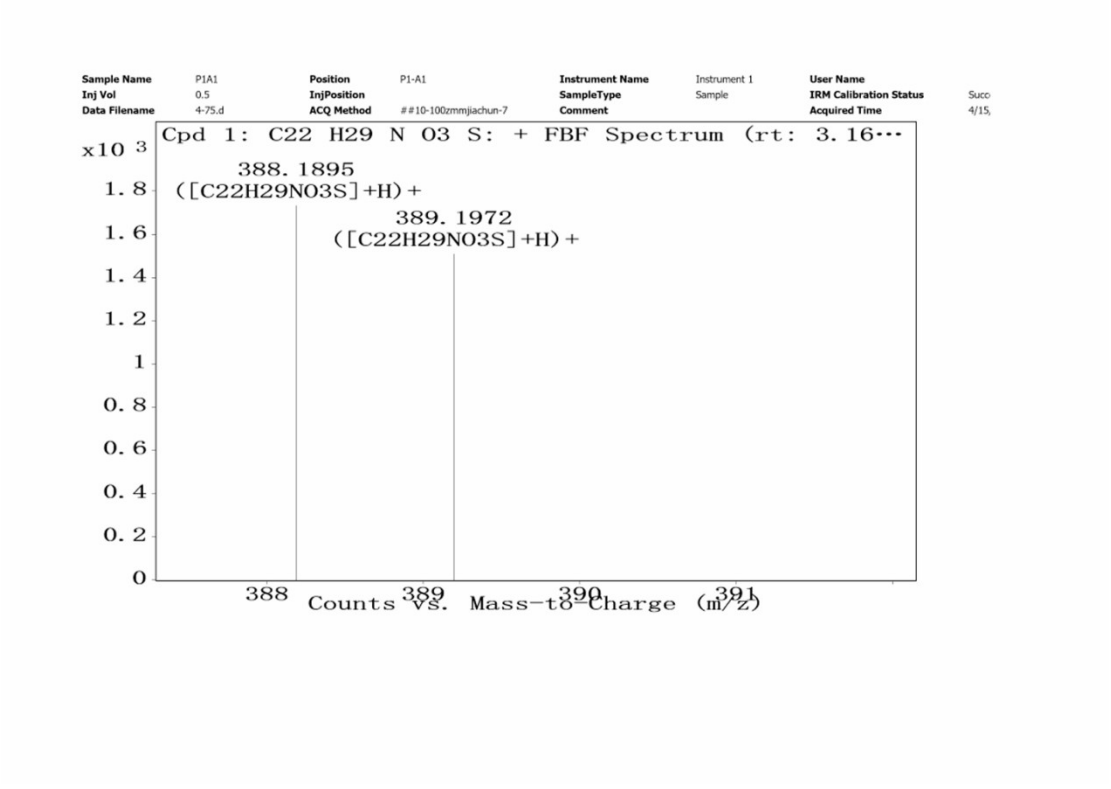




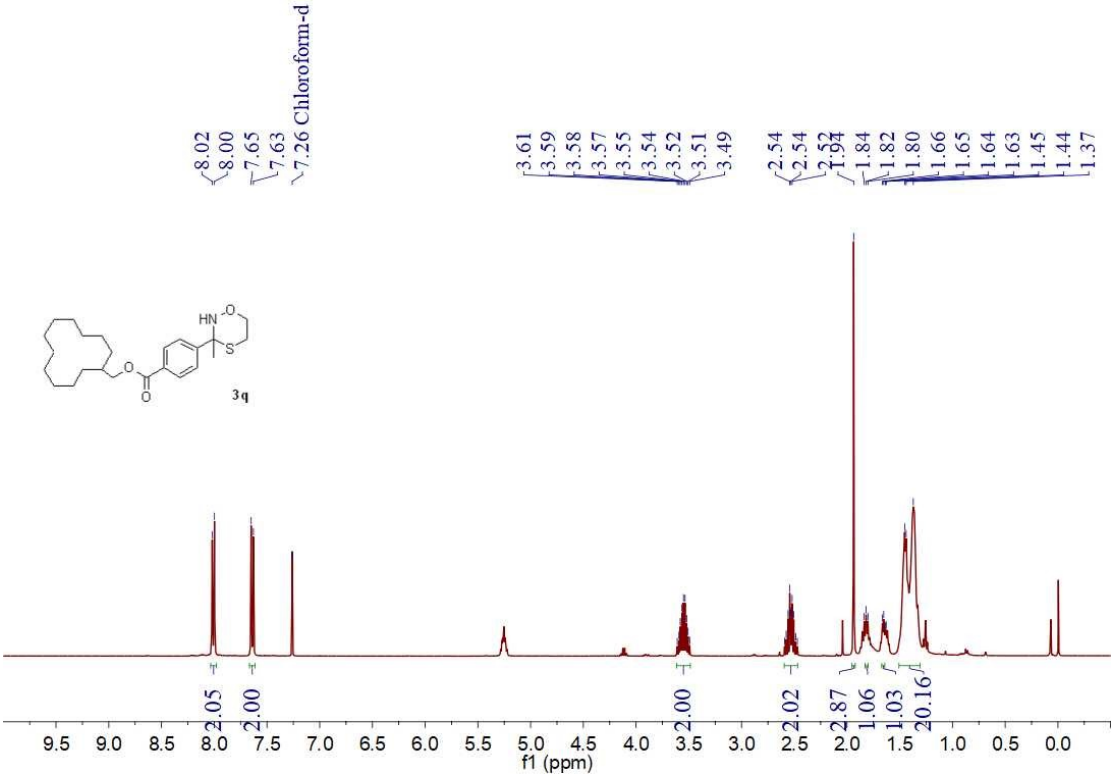
adamantan-1-ylmethyl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3p).

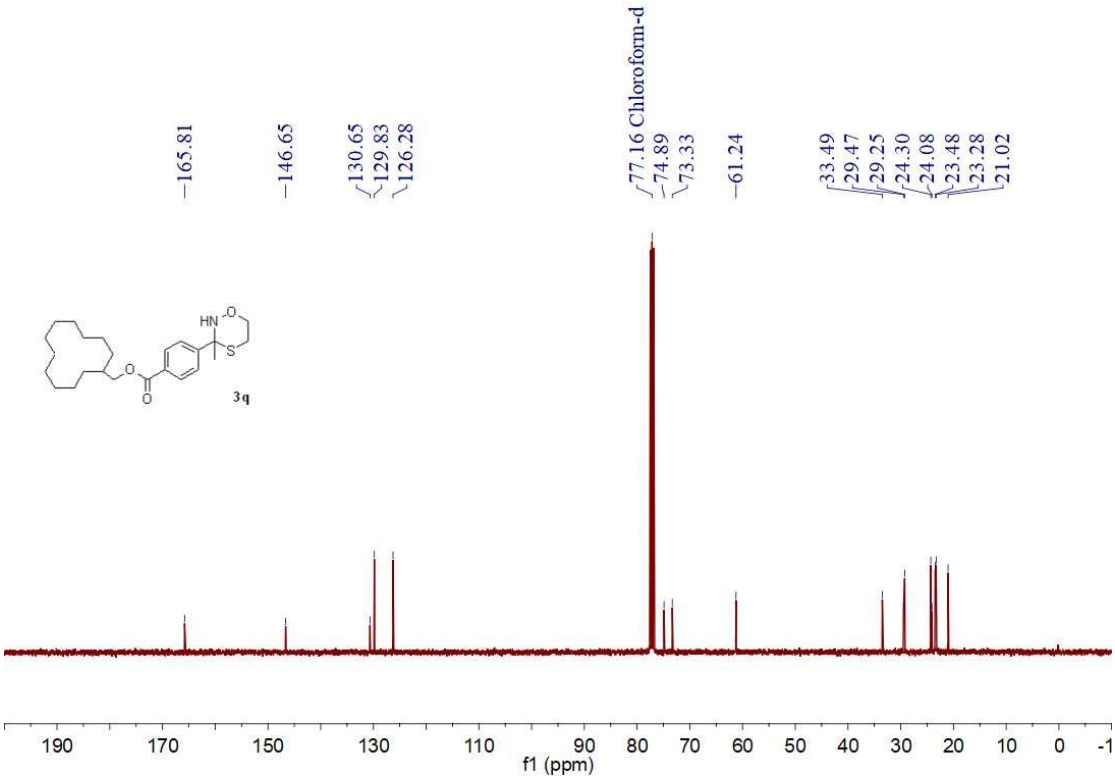

166.24
146.81
130.31
129.87
126.37
77.48
77.16 Chloroform-d
77.16
76.84
74.88
74.74
61.26
39.57
37.11
33.66
33.50
29.47
28.19
HN
O
S
O
O
3p
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

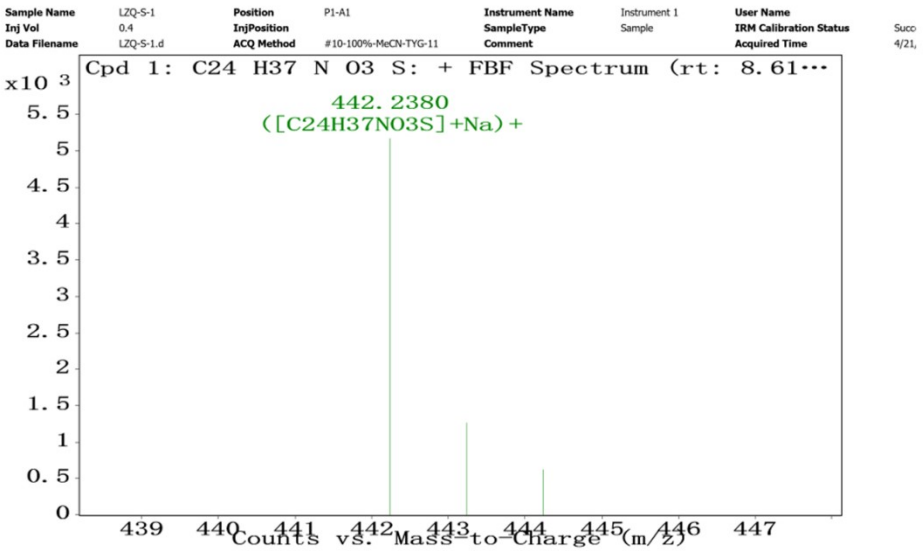

8.04
8.02
7.67
7.65
7.26 Chloroform-d
3.92
3.62
3.60
3.59
3.57
3.56
3.55
3.53
3.52
3.50
2.57
2.55
2.54
2.54
2.53
2.04
2.02
1.94
1.77
1.74
1.70
1.67
1.64
HN
O
S
O
O
3p
1.97
2.00
2.10
2.00
1.98
3.08
2.92
7.14
5.10
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



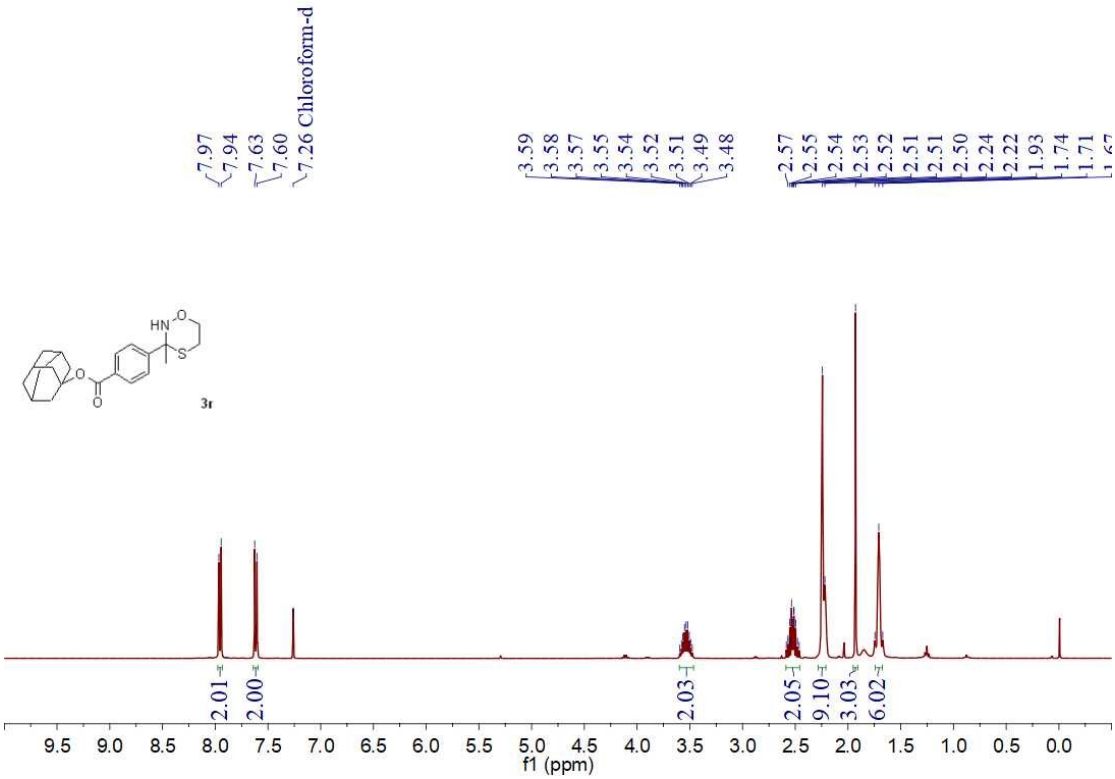
cyclododecylmethyl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3q).

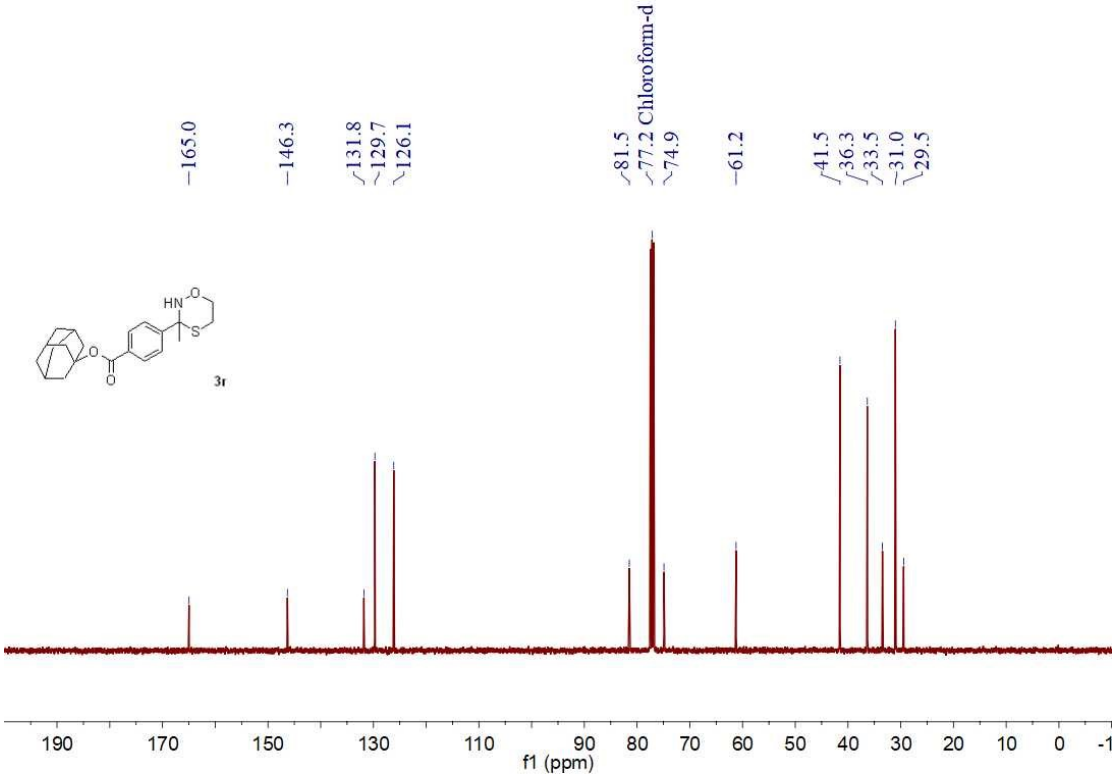


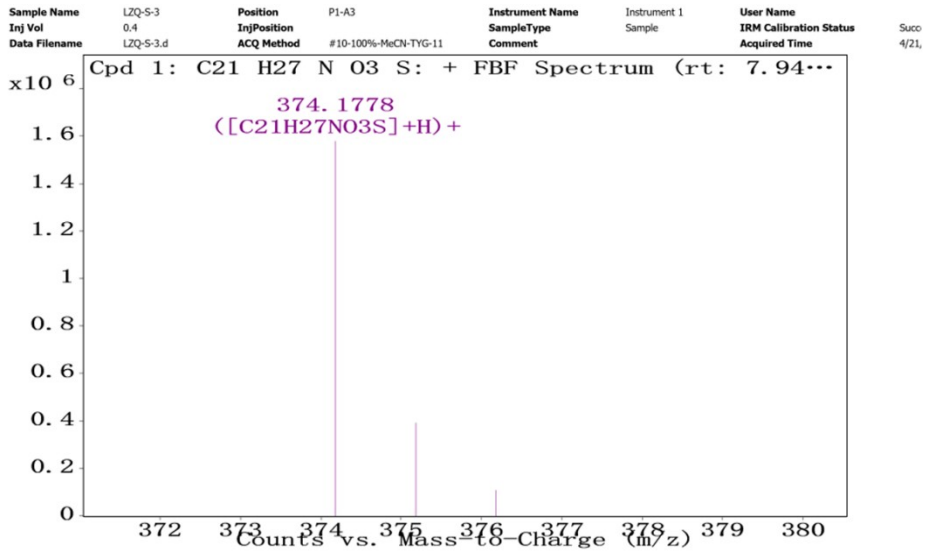




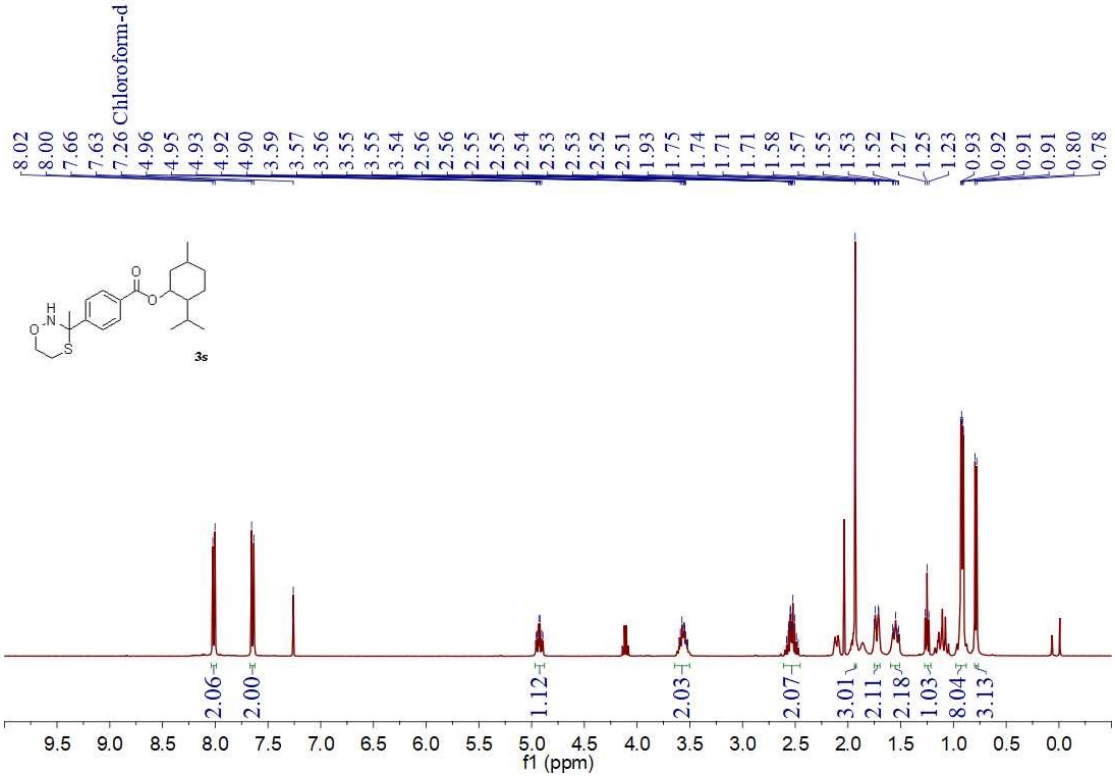
adamantan-1-yl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3r).

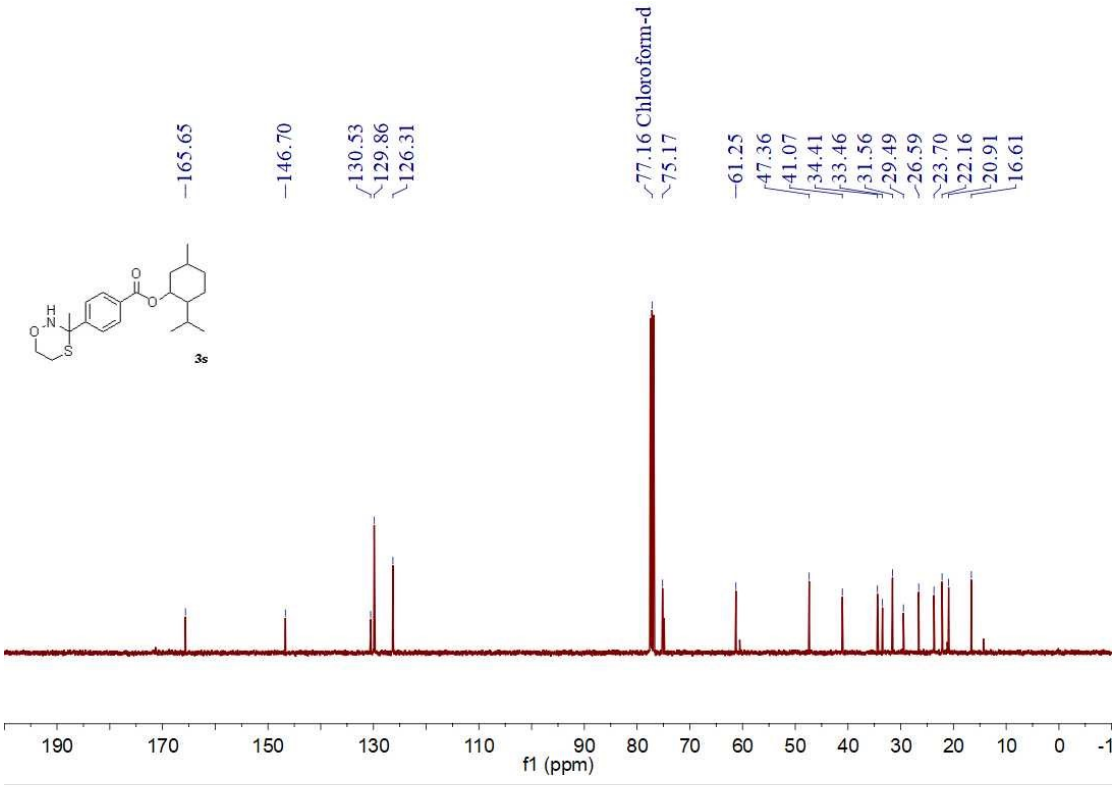

7.97
7.94
7.63
7.60
7.26 Chloroform-d
3.59
3.58
3.57
3.55
3.54
3.52
3.51
3.49
3.48
2.57
2.55
2.54
2.53
2.52
2.51
2.51
2.50
2.24
2.22
1.93
1.74
1.71
HN
O
S
O
O
3r
2.01
2.00
2.03
2.05
9.10
3.03
6.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

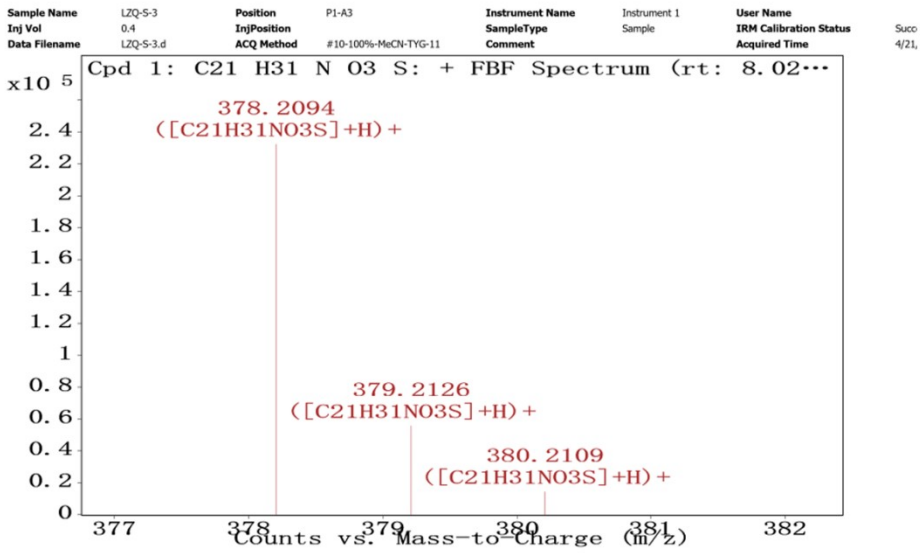

165.0
146.3
131.8
129.7
126.1
81.5
77.2 Chloroform-d
74.9
61.2
41.5
36.3
33.5
31.0
29.5
HN
O
S
O
O
3r
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



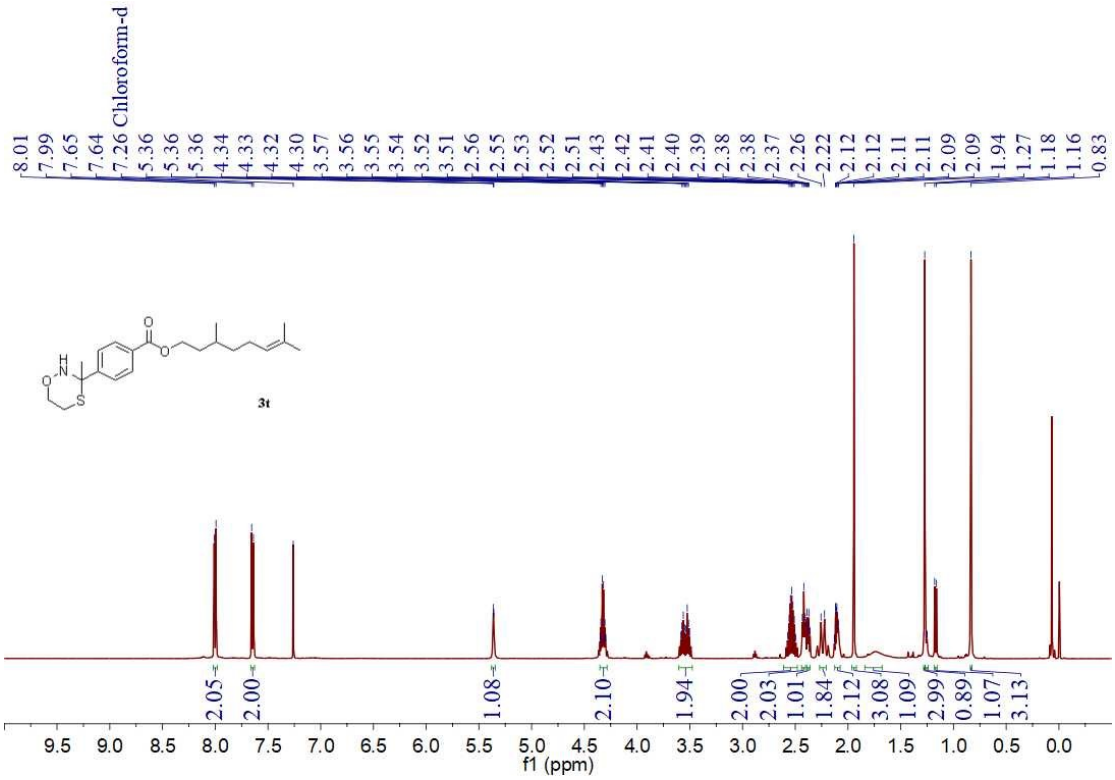
2-isopropyl-5-methylcyclohexyl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3s).

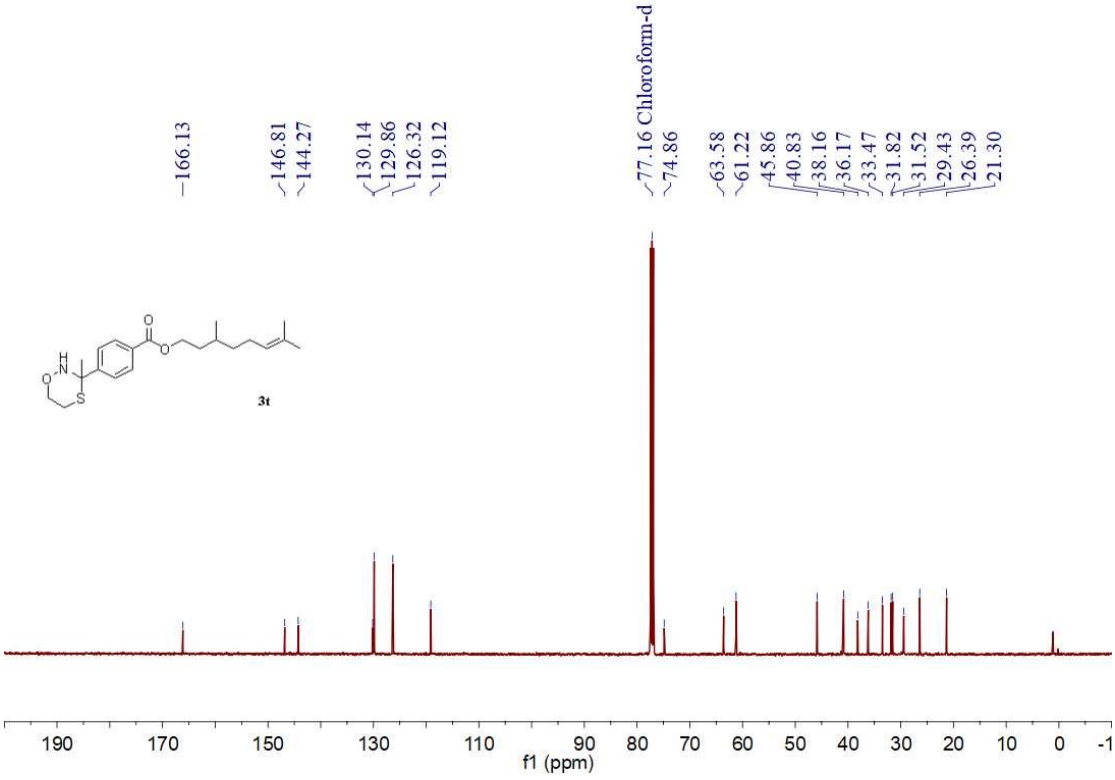


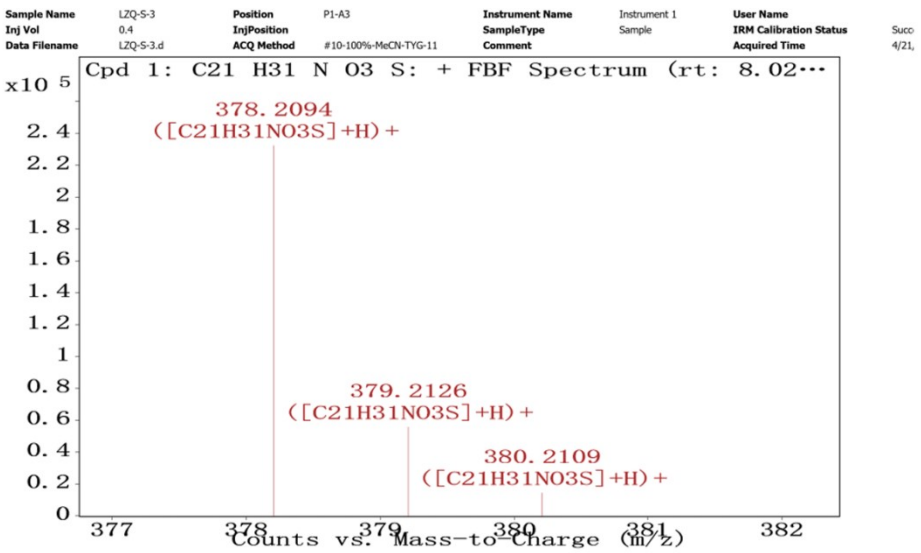




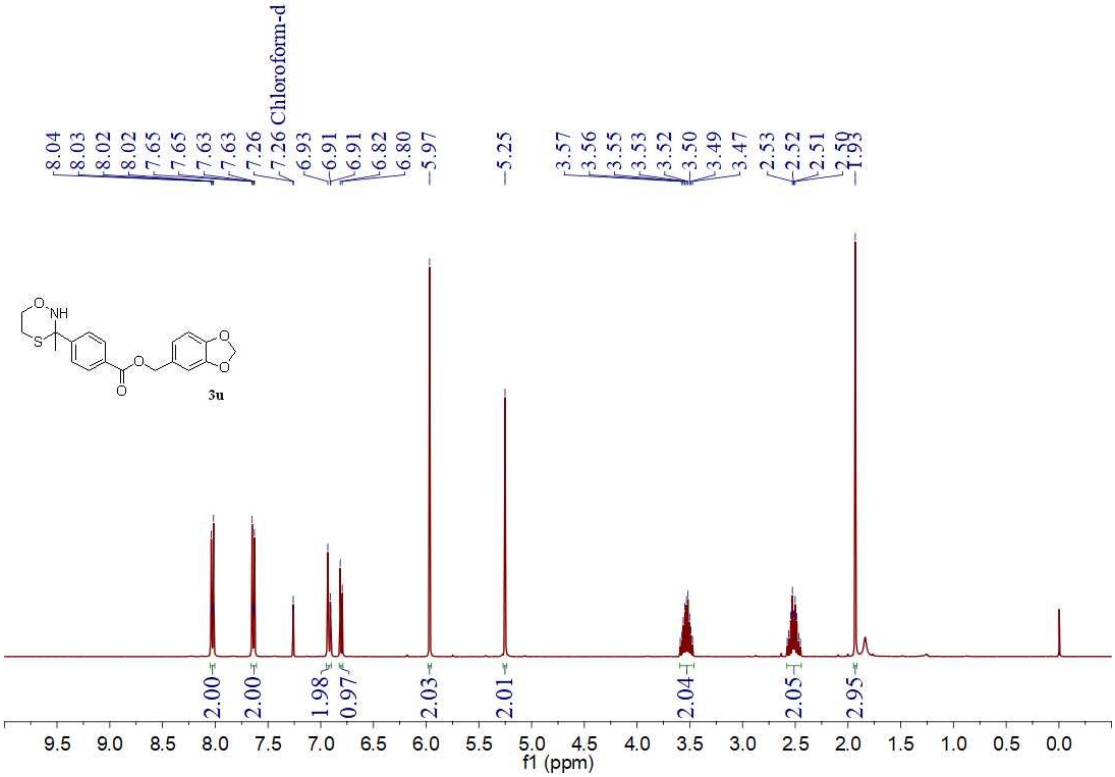
3,7-dimethyloct-6-en-1-yl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3t).

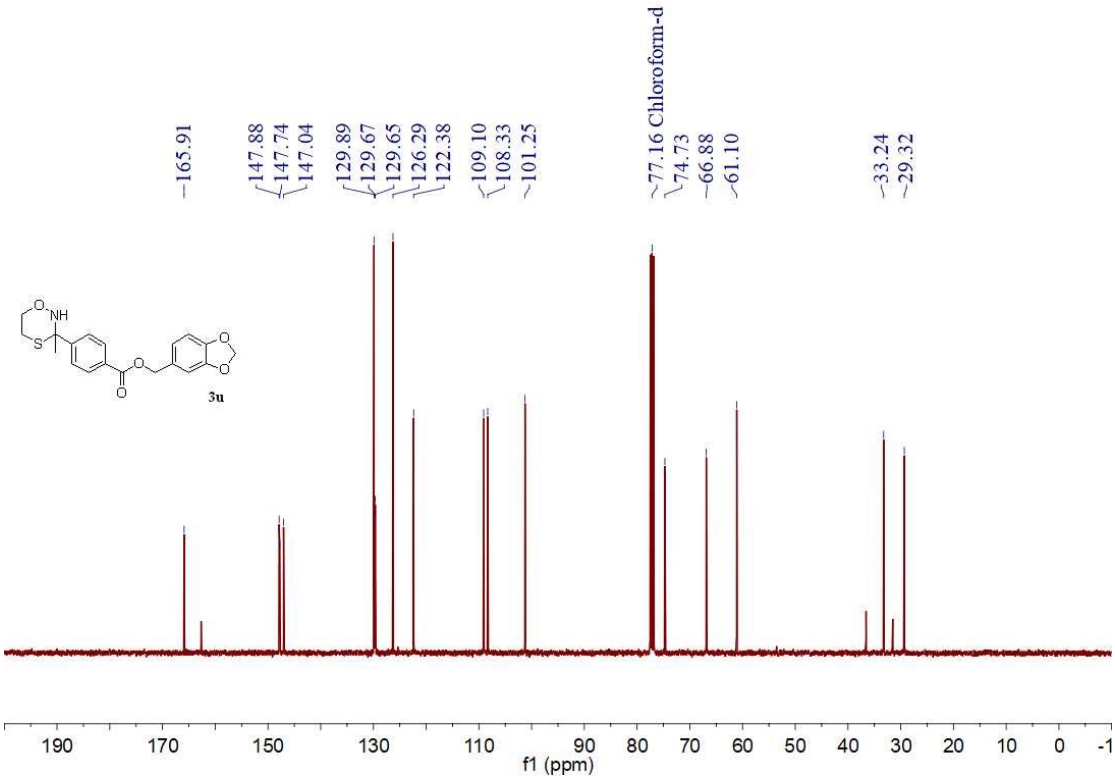

8.01
7.99
7.65
7.64
7.26 Chloroform-d
5.36
5.36
5.36
4.34
4.33
4.32
4.30
3.57
3.56
3.55
3.54
3.52
3.51
2.56
2.55
2.53
2.52
2.51
2.43
2.42
2.41
2.40
2.39
2.38
2.38
2.37
2.26
2.22
2.12
2.12
2.11
2.11
2.09
2.09
1.94
1.27
1.18
1.16
0.83
3t
2.05
2.00
1.08
2.10
1.94
2.00
2.03
1.01
1.84
2.12
3.08
1.09
2.99
0.89
1.07
3.13
f1 (ppm)

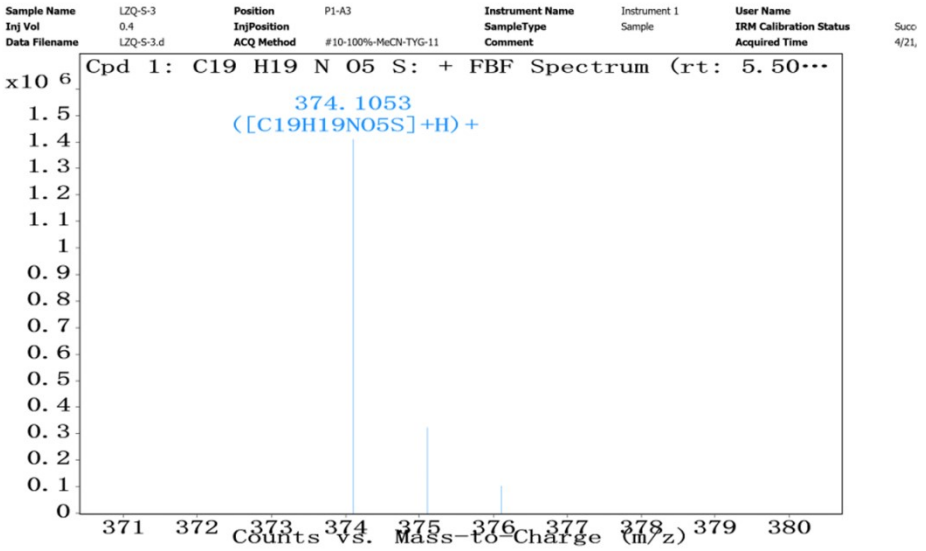

166.13
146.81
144.27
130.14
129.86
126.32
119.12
77.16 Chloroform-d
74.86
63.58
61.22
45.86
40.83
38.16
36.17
33.47
31.82
31.52
29.43
26.39
21.30
3t
f1 (ppm)



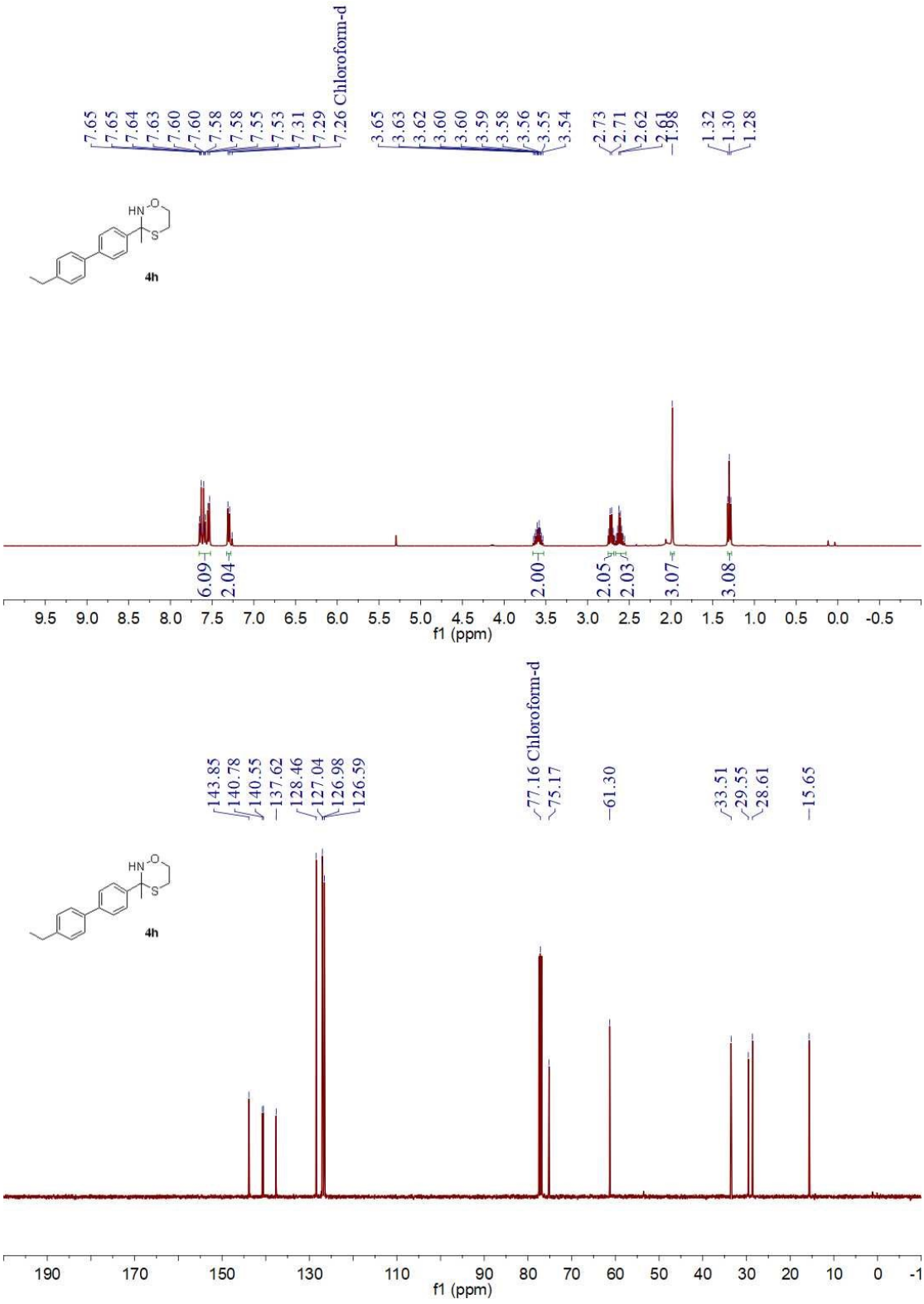
benzo[d][1,3]dioxol-5-ylmethyl 4-(2-methyl-1,4,3-oxathiazinan-2-yl)benzoate (3u)

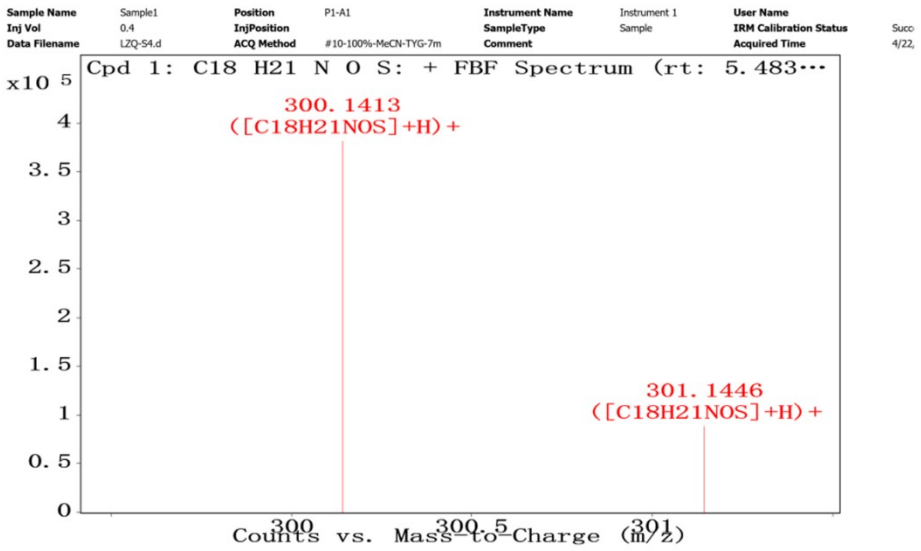






3-(4'-ethyl-[1,1'-biphenyl]-4-yl)-3-methyl-1,4,2-oxathiazinane (4h).


7.65
7.65
7.64
7.63
7.60
7.60
7.58
7.58
7.55
7.53
7.31
7.29
7.26 Chloroform-d
3.65
3.63
3.62
3.60
3.60
3.59
3.58
3.56
3.55
3.54
2.73
2.71
2.62
2.61
1.98
1.32
1.30
1.28
HN
O
S
4h
6.09
2.04
2.00
2.05
2.03
3.07
3.08
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
f1 (ppm)
143.85
140.78
140.55
137.62
128.46
127.04
126.98
126.59
77.16 Chloroform-d
75.17
61.30
33.51
29.55
28.61
15.65
HN
O
S
4h
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



11. References

G. Li, K. Yu, J. Yang, B. Xu, Q. Chen, *J. Org. Chem.* **2021**, 86, 15946-15952.