Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

SO_4^{2-} ions as nucleophilic reagent: straightforward electrochemical access to organosulfates

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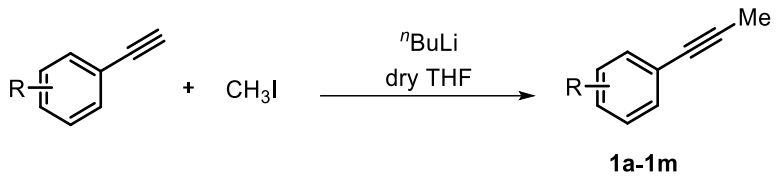
1. General information

All reactions were carried out in sealed N_2 using oven dried glassware. 1,1,1,3,3,3-Hexafluoro-2-propanol, Chloroform, Tetrabutylammonium hydrogen sulfate, Diethylamine, Carbon rod electrodes, platinum electrodes were all available from commercial sources. The electrochemical instrument was HONGSHENGFENG DPS-305CF. Column chromatography was performed on silica gel (200-300 mesh). NMR spectra were recorded in $CDCl_3$ on 500 MHz spectrometers. ^1H NMR chemical shifts (δ) are reported in parts per million relatives to tetramethylsilane (0 ppm). The following abbreviations are used for multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, and dt = doublet of triplets, m = multiplet. HRMS(ESI-TOF) were obtained on an Ultima Global spetrometer with an ESI source and were produced by America waters (acquit uplc h-class/xevo g2).

Scheme S1. Components required for the reaction

2. General Procedure for the Synthesis of the starting materials

Preparation of substrates 1[1]

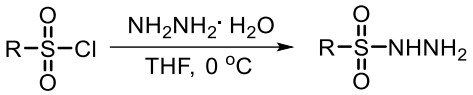


1a-1m

The compounds **1a-1m** were prepared according to previously described methods. To a flame-dried round-bottom flask under N_2 was added alkyne (5.0 mmol) followed by dry THF (33.0 mL, 0.2 M). Cool the flask to 0 °C. ^{n}BuLi (4.0 mL, 2.5 M in hexanes, 10.0 mmol, 2.0 equiv.) was added slowly and the reaction was allowed to stir for 1 hour. Iodomethane (0.65 mL, 10.2 mmol, 2.1 equiv.) was added at -20 °C and the reaction was allowed to stir at room temperature for 3 ~ 5 hours (when most of alkyne was consumed as detected by TLC). The reaction was quenched with a saturated solution of ammonium chloride and extracted with ethyl acetate. The organics were dried over $MgSO_4$ and the solvents were removed under reduced pressure. The residue was purified by silica chromatography to afford the corresponding compounds **1a-1m**.

Preparation of substrates 2[2]

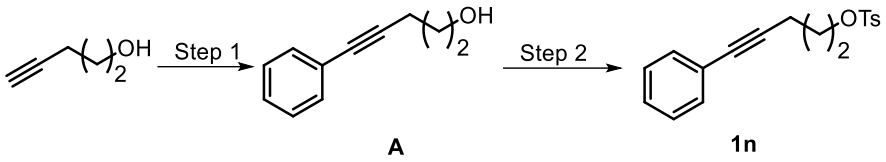
The compound **2a-2m** according to previously described methods.



2a-2m

The hydrazine hydrate (80%, 3.0 mmol) was added dropwise into the solution of sulfonyl chloride (1.0 mmol) in THF (5.0 mL) under air at 0 °C. Subsequently, the mixture was further stirred at 0 °C for 5 minutes. After the completion of the reaction, the residue was extracted with dichloromethane, and the combined organic layer was ashed with water, and brine, and dried over $MgSO_4$. Concentration in vacuum followed by silica column purification with petroleum ether/ethyl acetate eluent gave the desired products **2a-2m**.

Preparation of substrates 3[3]



Step1: To a 100.0 mL flame-dried round-bottom flask, under N_2, was added $PdCl_2(PPh_3)_2$ (0.1 mmol, 0.01 equiv.), CuI (0.2 mmol, 0.02 equiv.), iodobenzene (12.0 mmol, 1.2 equiv.), alkynol (10.0 mmol, 1.0 equiv.) and dry Et_3N (20.0 mL), the reaction was allowed to stir at room temperature. The reaction was stirred overnight checked by TLC. The reaction is filtered over celite, washing with dichloromethane. The solvent was removed and the residue purified by flash column chromatography on silica gel to give compounds **A**.

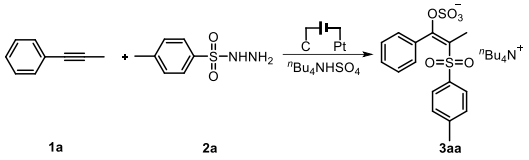
Step2: To a stirred solution of Compound **A** (5.0 mmol, 1.0 equiv.), dimethylaminopyridine (DMAP) (0.5 mmol, 0.1 equiv.) and TEA (10.0 mmol, 2.0 equiv.) in dry DCM (10.0 mL) at 0 ^oC was added ptoluenesulfonyl chloride (TsCl) (6.0 mmol, 1.2 equiv.) portion wise. The reaction mixture was stirred for 1 hour and then quenched with water, extracted with ethyl acetate, dried over anhydrous $MgSO_4$, concentrated and chromatography on silica gel to give compounds **1n**.

3. General procedure

In an undivided Schlenk flask (10.0 mL) equipped with a stir bar, substrate **1** (0.2 mmol, 1.0 equiv.), substrate **2** (0.6 mmol, 3.0 equiv.), $^nBu_4NHSO_4$ (0.8 mmol, 4.0 equiv.) and Et_2NH (0.2 mmol, 1.0 equiv.) were combined and added. The flask was equipped with a rubber stopper, a carbon rod (C) anode and a platinum cathode (10 mm x 10 mm x 0.1 mm) and then flushed with nitrogen. Then HFIP (3.0 mL) and $CHCl_3$ (2.0 mL) were injected respectively into the flask *via* syringes. The reaction mixture was stirred and electrolyzed at a controlled current of 3.0 mA at 25 ^oC for 6.5 hours. After the reaction (monitored by TLC), the reaction system was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with ethyl acetate to give the product.

4. Optimization of reaction conditions

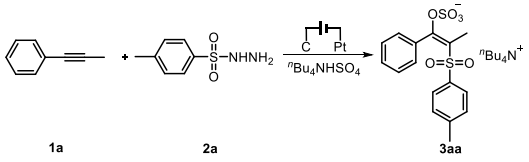
Table S1. Optimization of reaction conditions[a]



Entry	Deviation from standard conditions[a]	Yield[b](%)
1	none	72
2	5 mA, 3.5 h	43
3	1 mA, 10 h	51
4	Mg cathode	36
5	Ni cathode	41
6	DCE instead of $CHCl_3$	51
7	DCM instead of $CHCl_3$	61
8	CH_3CN instead of $CHCl_3$	trace
9	Et_3N instead of Et_2NH	64
10	^{i}PrNH instead of Et_2NH	48
11	K_2CO_3 instead of Et_2NH	21
12	Without Et_2NH	45
13	40 °C	43
14	under air	34
15	no electric current	n.r.

[a]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell (3.63 F/mol). [b]Isolated yield. n.r. = no reaction. RT = room temperature.

Table S2. The effect of HFIP

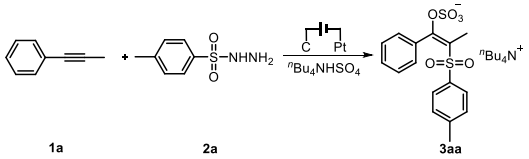


Entry	Deviation from standard conditions	Yield[d](%)
[a]1	HFIP: $CHCl_3$ = 5.0 mL: 0.0 mL	32

[a]2	HFIP: $CHCl_3$ = 4.5 mL: 0.5 mL	41
[a]3	HFIP: $CHCl_3$ = 4.0 mL: 1.0 mL	52
[a]4	HFIP: $CHCl_3$ = 3.5 mL: 1.5 mL	64
[a]5	HFIP: $CHCl_3$ = 3.0 mL: 2.0 mL	72
[a]6	HFIP: $CHCl_3$ = 2.0 mL: 3.0 mL	56
[a]7	HFIP: $CHCl_3$ = 1.0 mL: 4.0 mL	42
[a]8	HFIP: $CHCl_3$ = 0.5 mL: 4.5 mL	0
[b]9	HFIP: $CHCl_3$ = 3.0 mL: 2.0 mL	33
[b]10	$CHCl_3$ = 5.0 mL	trace
[b]11	CH_3CN = 5.0 mL	trace
[b]12	CH_3NO_2 = 5.0 mL	trace
[b]13	DCE = 5.0 mL	trace
[b]14	CH_3OH = 5.0 mL	trace
[c]15	$^{i}Pr_2NH$ as base	trace
[c]16	Et_3N as base	trace
[c]17	Pyridine as base	trace
[c]18	Na_2CO_3 as base	trace

[a]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell (3.63 F/mol). [b]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, divided cell. [c]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), base (0.2 mmol), CH_3CN (5.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, divided cell. [d]Isolated yield. RT = room temperature.

Table S3. The effect of various additives



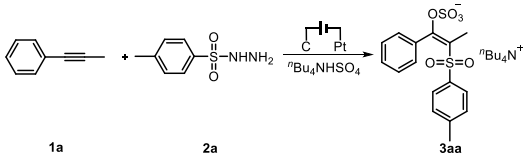
1a 2a 3aa

Entry	Deviation from standard conditions[a]	Yield[b](%)
1	DMAP	46
2	$^{i}PrNEt$	51
3	N,N-dimethylaniline	63

4	Morpholine	69
5	Dibenzylamine	70
6	Dibutylamine	71
7	NaOH	34
8	Cs_2CO_3	31
9	AcOH	46
10	DBU	58
11	Cp_2Fe	52
12	DABCO	47

[a]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^nBu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell (3.63 F/mol). [b]Isolated yield. RT = room temperature.

Table S4. The effect of different ratios of **1a** and **2a**

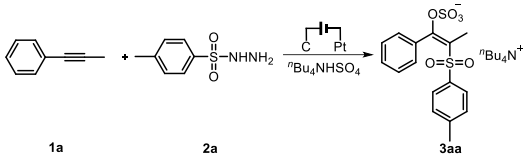


1a **2a** **3aa**

Entry	Deviation from standard conditions	Yield[c](%)
[a]1	**1a: 2a** = 1:1	42
[a]2	**1a: 2a** = 1:2	61
[a]3	**1a: 2a** = 1:3	72
[a]4	**1a: 2a** = 1:4	54
[a]5	**1a: 2a** = 1:5	39
[b]6	**1a: 2a** = 2:1	43
[b]7	**1a: 2a** = 3:1	51
[b]8	**1a: 2a** = 4:1	39
[b]9	**1a: 2a** = 5:1	34

[a]Standard conditions: **1a**, **2a**, $^nBu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell (3.63 F/mol). [b]Standard conditions: **1a** (0.6 mmol), **2a** (0.2 mmol), $^nBu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell. [c]Isolated yield. RT = room temperature.

Table S5. The reaction times of **1a**

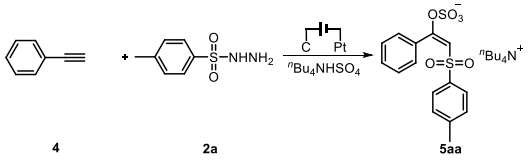


1a 2a 3aa

Entry	Deviation from standard conditions[a]	Yield[b](%)
1	2.0 h	24
2	4.0 h	41
3	6.0 h	67
4	8.0 h	61
5	10.0 h	54

[a]Standard conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), Et_2NH (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 6.5 h, undivided cell (3.63 F/mol). [b]Isolated yield. RT = room temperature.

Table S6. The reaction times of **4**

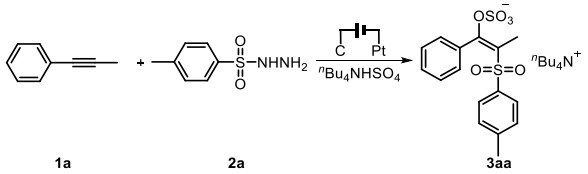


4 2a 5aa

Entry	Deviation from standard conditions[a]	Yield[b](%)
1	2.0 h	29
2	4.0 h	51
3	6.0 h	43
4	8.0 h	37
5	10.0 h	35

[a]Standard conditions: **4** (0.2 mmol), **2a** (0.6 mmol), $^{n}Bu_4NSO_4$ (0.8 mmol), Et_3N (0.2 mmol), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), carbon rod anode, platinum cathode (10 mm x 10 mm x 0.1 mm), constant current = 3.0 mA, under N_2, RT, 4.0 h, undivided cell (2.24 F/mol). [b]Isolated yield. RT = room temperature.

5. Gram-scale reaction



1a 2a 3aa

In an undivided Schlenk flask (100.0 mL) equipped with a stir bar, substrate **1a** (5.0 mmol, 1.0 equiv.), sulfonyl hydrazide **2a** (15.0 mmol, 3.0 equiv.), ^{n}Bu$_4$NHSO$_4$ (20.0 mmol, 4.0 equiv.), Et$_2$NH (5.0 mmol 1.0 equiv.) were combined and added. The flask was equipped with a rubber stopper, a carbon rod (C) anode and a platinum cathode (30 mm x 30 mm x 0.1 mm) and then flushed with nitrogen. Then HFIP (60.0 mL) and CHCl$_3$ (30.0 mL) were injected respectively into the undivided Schlenk flask *via* syringes. The reaction mixture was stirred and electrolyzed at a controlled current of 10.0 mA at room temperature for 48 hours. After the reaction (monitored by TLC), the reaction system was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with ethyl acetate to give the product **3aa** (2.1 g, 74%).

6. Fluorescent spectra in solution

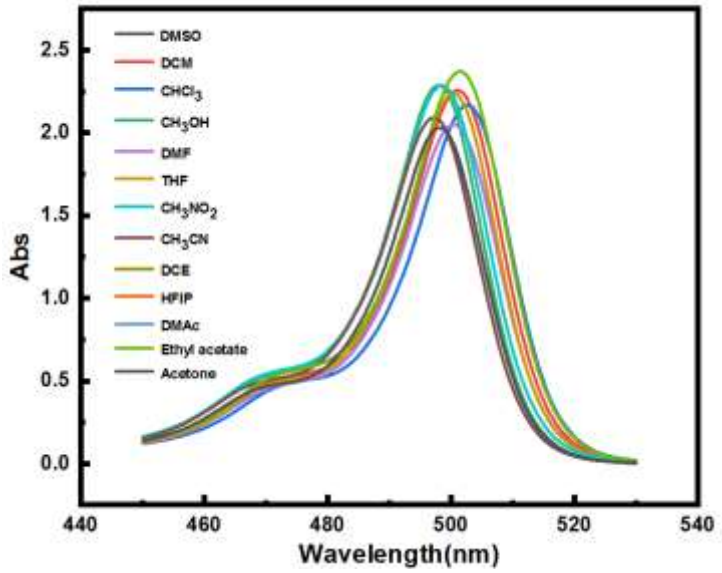


Figure S1. Ultraviolet absorption spectra of **7** in different solution

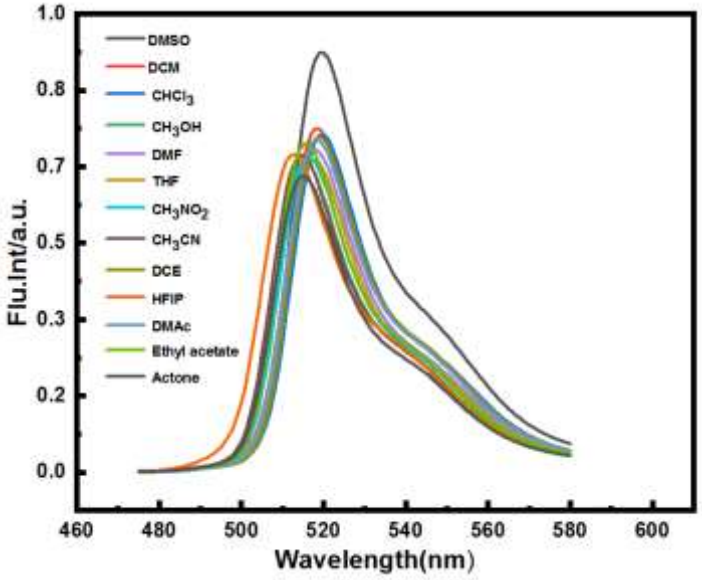


Figure S2. Fluorescent spectra of **7** in different solution

Table S7. Fluorescent spectra data of **7** in different solvent

Solvent	$^{Max}\lambda_{em}$ [nm]	a.u.	Solvent	$^{Max}\lambda_{em}$ [nm]	a.u.
DMSO	519	0.88	CH_3CN	514	0.65
DCM	518	0.70	DCE	519	0.66
$CHCl_3$	519	0.81	HFIP	513	0.94
CH_3OH	515	0.61	DMAc	518	0.65
DMF	518	0.75	Ethyl acetate	516	0.60
THF	517	0.66	Actone	515	0.64
CH_3NO_2	516	0.64			

7. Fluorescence Microscopic Studies

Cell culture. All cells were cultured in confocal culture dishes and incubates overnight at 37 °C in a humidified atmosphere of 5% CO_2 for 24 hours to allow for firm adherence. After being rinsed with 1xPBS buffer, the cells were incubated with the 7 for 2 h at 37 C.

Fluorescence microscopic imaging. All the experiments were conducted in live cells. The living cell imaging was measured by Nikon C2 plus with an excitation filter of 488 nm and the collection wavelength range is at 490-550 nm.

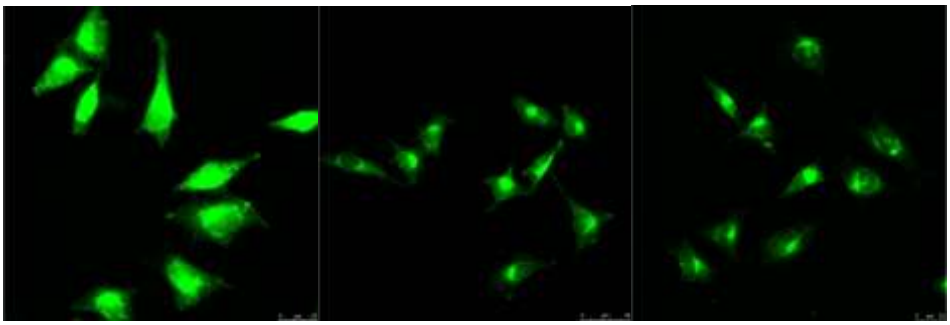
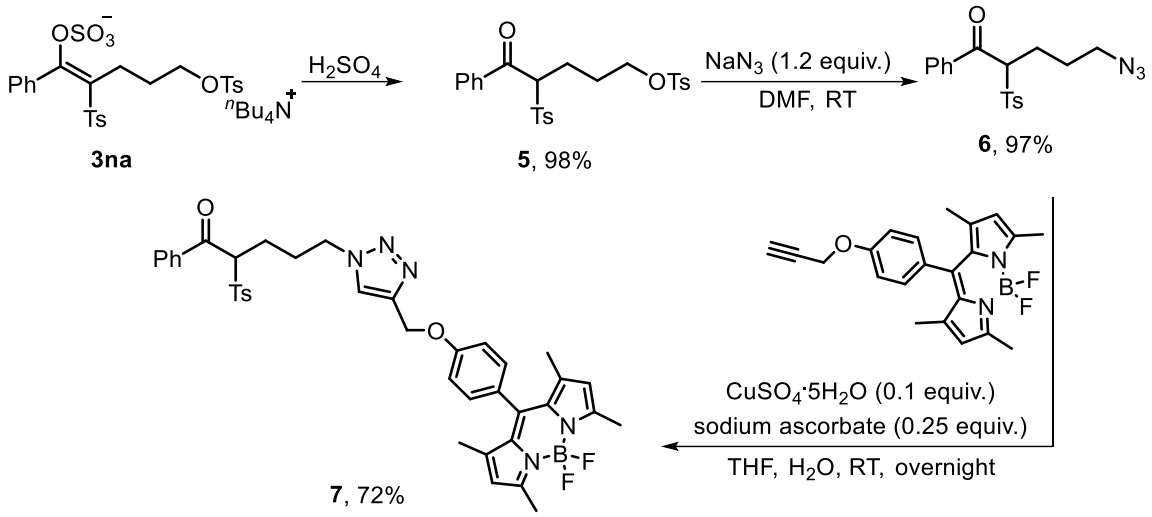


Figure S3. Fluorescence intensity of **7** in MCF-7 cells

8. Product transformation



Step 1: Take 0.1 mmol **3na** into 25.0 mL round flask, add sulfuric acid solution and stir for 30 min, extract the system with dichloromethane, dried over anhydrous Na_2SO_4. The solvent was removed and the residue purified by flash column chromatography on silica gel to give compound **5** (yellow oil, 98 %).

Step 2: Sodium azide (0.24 mmol, 1.2 equiv.) was added to a solution of **5** (0.2 mmol, 1.0 equiv.) in DMF (1.1 mL) and stirred at room temperature overnight. H_2O was added to the mixture and the aqueous phase was extracted with ethyl acetate. The combined organic extract was washed with saturated aqueous NaCl solution, dried over anhydrous Na_2SO_4. The solvent was removed and the residue purified by flash column chromatography on silica gel to give compound **6** (yellow oil, 97 %).

Step 3: To a solution of compound **6** (0.2 mmol, 1.0 equiv.) in a 1:1 mixture of THF: H_2O (5.0 mL) were added copper (II) sulfate (0.02 mmol, 0.1 equiv.), sodium ascorbate (0.05 mmol, 0.25 equiv.) and the BODIPY (0.26 mmol, 1.3 equiv.). The mixture was brine, dried over anhydrous Na_2SO_4. The solvent was evaporated in vacuum and purified by silica gel column chromatography to avail products **7** (orange solid, 72%).

9. Cyclic voltammetry study

The cyclic voltammetry experiments were carried out with a computer-controlled electrochemical analyzer for electrochemical measurements. The cyclic voltammetry experiments were measured at room temperature. The data was collected with the

CS300H potentiostat (Wuhan COster Instrument Co., LTD). The experiment was performed in a three-electrode cell with HFIP (3.0 mL) and $CHCl_3$ (2.0 mL) as the solvent, $^{n}Bu_4NHSO_4$ (0.4 mmol) as the supporting electrolyte, and the concentration of the **1a** was 0.1 mmol; the concentration of the **2a** was 0.3 mmol. The scan speed was 100 mV/s. The potential ranges investigated were 0.0 V to 3.0 V vs. SCE (saturated aqueous KCl). CV plotting convention is Origin.

Working electrode: The working electrode is a 3 mm diameter glassy carbon working electrode. Polished with 0.05 μm aluminum oxide and then sonicated in distilled water and ethanol before measurements.

Reference electrode: The reference electrode is SCE (saturated aqueous KCl) that was washed with water and ethanol before measurements.

Counter electrode: The counter electrode is a platinum wire that was polished with 0.05 μm aluminum oxide and then sonicated in distilled water and ethanol before measurements.

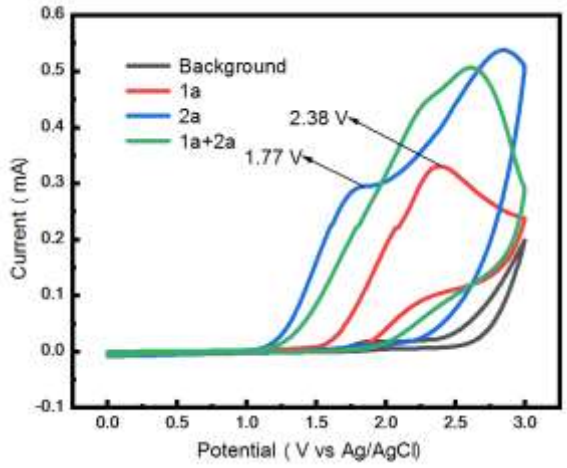


Figure S4. Cyclic voltammogram

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of **1a** (0.1 mmol), **2a** (0.3 mmol), were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent [HFIP (3.0 mL) and $CHCl_3$ (2.0 mL)] containing $^{n}Bu_4NHSO_4$ (0.4 mmol) was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.

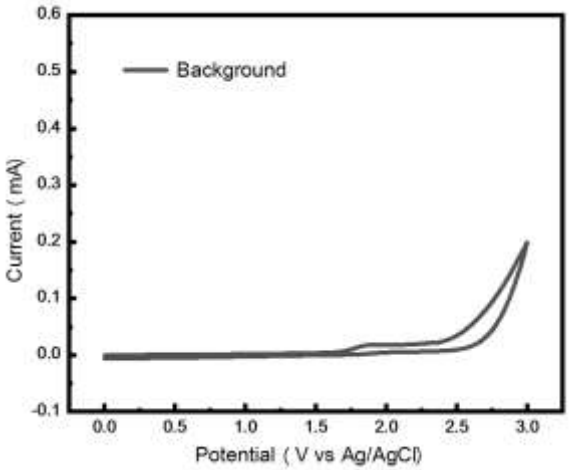


Figure S5. Cyclic voltammogram of Background

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of Background was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent [HFIP (3.0 mL) and $CHCl_3$ (2.0 mL)] containing $^{n}Bu_4NHSO_4$ (0.4 mmol) was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.

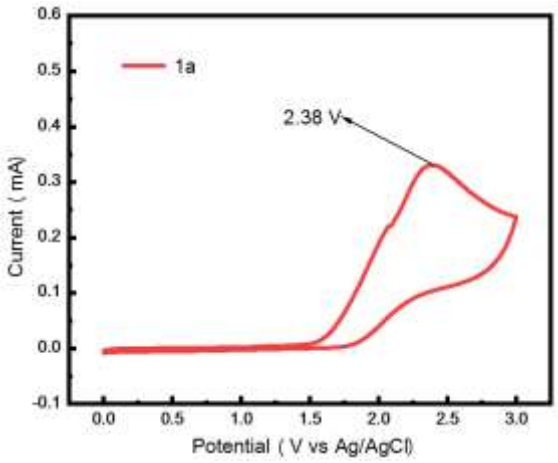


Figure S6. Cyclic voltammogram of **1a**

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of **1a** (0.1 mmol) was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent [HFIP (3.0 mL) and $CHCl_3$ (2.0 mL)] containing $^{n}Bu_4NHSO_4$ (0.4 mmol)

was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.

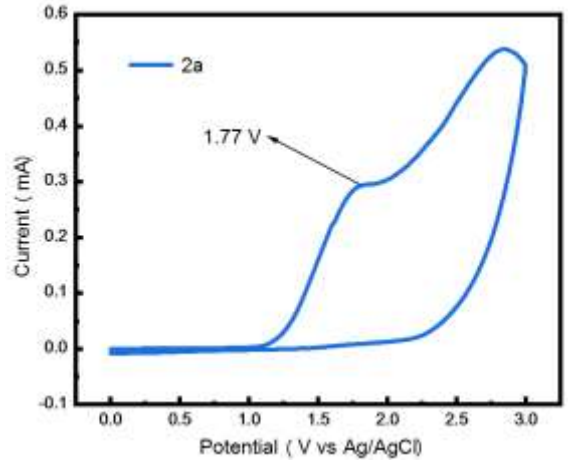


Figure S7. Cyclic voltammogram of **2a**

General procedure for cyclic voltammetry (CV): Cyclic voltammogram of **2a** (0.3 mmol) was performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent [HFIP (3.0 mL) and $CHCl_3$ (2.0 mL)] containing $^{n}Bu_4NHSO_4$ (0.4 mmol) was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.

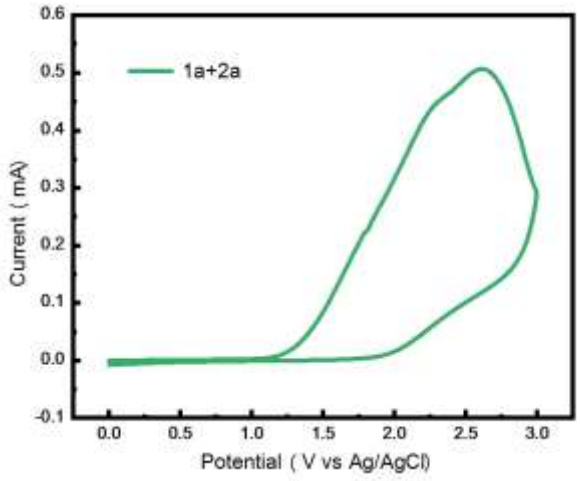
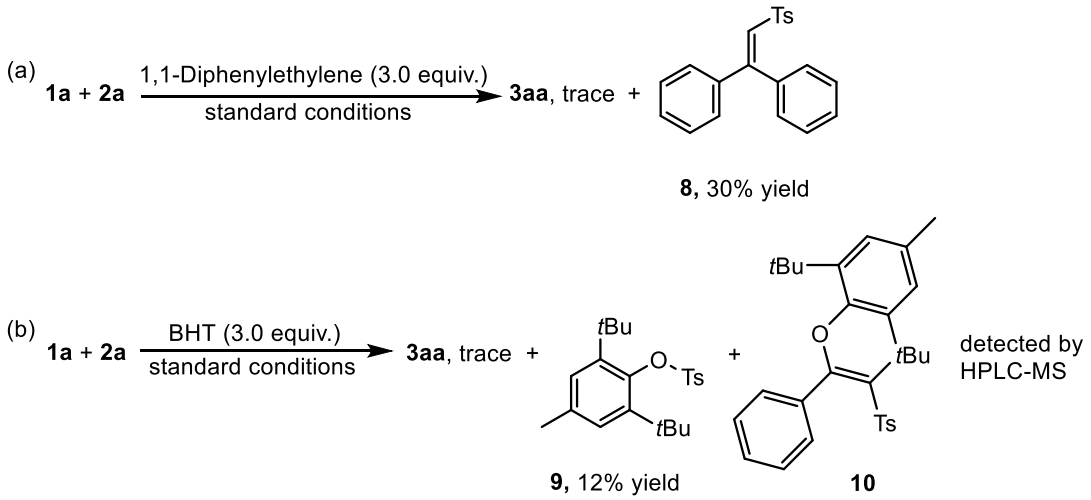


Figure S8. Cyclic voltammogram of **1a**+**2a**

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of **1a** (0.1 mmol), **2a** (0.3 mmol), were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum

wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent [HFIP (3.0 mL) and $CHCl_3$ (2.0 mL)] containing $^{n}Bu_4NHSO_4$ (0.4 mmol) was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.

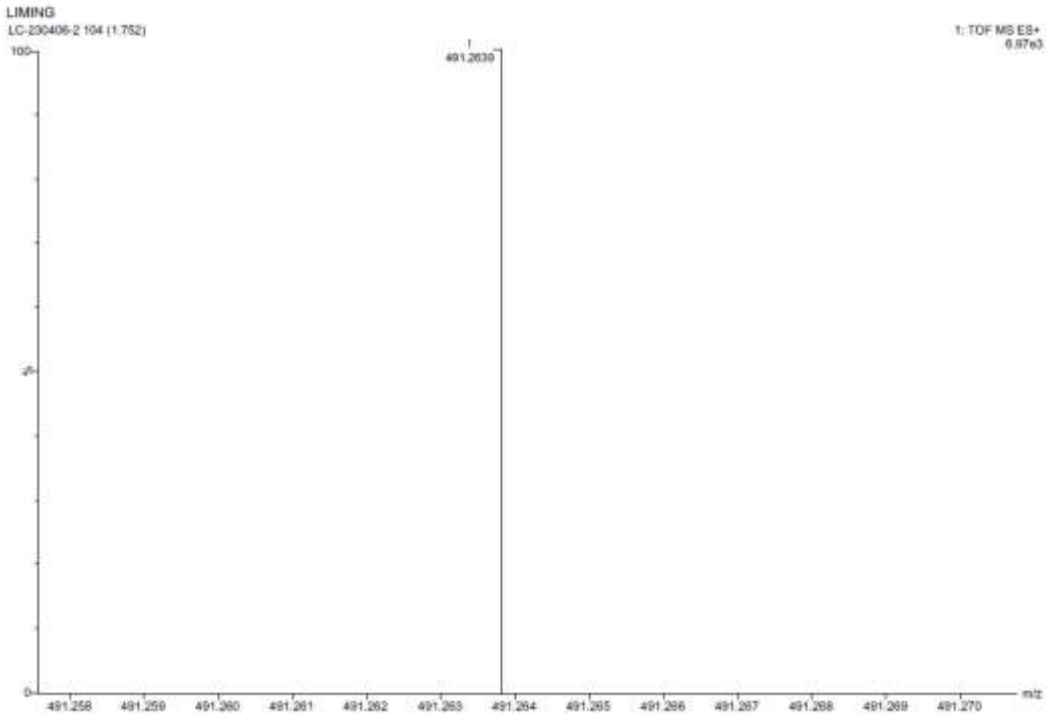
10. Control experiments



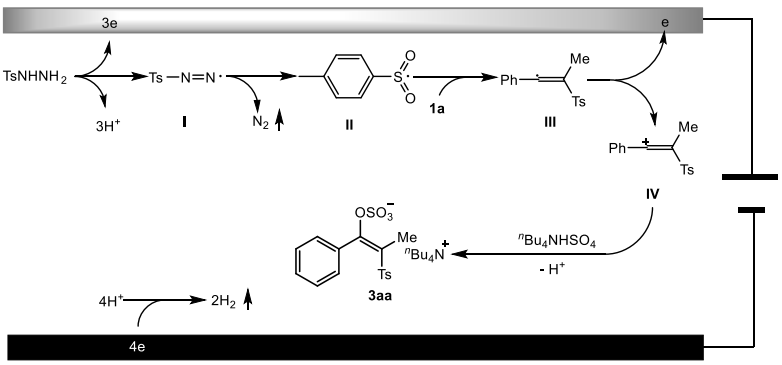
(a) In an undivided Schlenk flask (10.0 mL) equipped with a stir bar, substrate **1a** (0.2 mmol, 23.2 mg, 1.0 equiv.), sulfonyl hydrazides **2a** (0.6 mmol, 111.7 mg, 3.0 equiv.), $^{n}Bu_4NHSO_4$ (0. 8 mmol, 271.6 mg, 4.0 equiv.), and 1,1-diphenylethylene (0.6 mmol, 54.1 mg, 3.0 equiv.) were combined and added. The flask was equipped with a rubber stopper, a carbon rod (C) anode and a platinum cathode (10 mm x 10 mm x 0.1 mm) and then flushed with nitrogen. Then HFIP (3.0 mL) and $CHCl_3$ (2.0 mL) were injected respectively into the flask *via* syringes. The reaction mixture was stirred and electrolyzed at a controlled current of 3.0 mA at room temperature for 6.5 hours. After the reaction (monitored by TLC), the reaction system was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate to give the product **8** (60.1 mg, 30%).

(b) In an undivided Schlenk flask (10.0 mL) equipped with a stir bar, substrate **1a** (0.2 mmol, 23.2 mg, 1.0 equiv.), sulfonyl hydrazides **2a** (0.3 mmol, 111.7 mg, 3.0 equiv.), $^{n}Bu_4NHSO_4$ (0.8 mmol, 271.6 mg, 4.0 equiv.), and BHT (0.6 mmol, 66.1 mg, 3.0 equiv.) were combined and added. The flask was equipped with a rubber stopper, a carbon rod

(C) anode and a platinum cathode (10 mm x 10 mm x 0.1 mm) and then flushed with nitrogen. Then HFIP (3.0 mL) and $CHCl_3$ (2.0 mL) were injected respectively into the flask *via* syringes. The reaction mixture was stirred and electrolyzed at a controlled current of 3.0 mA at room temperature for 6.5 hours. After the reaction (monitored by TLC), the reaction system was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate to give the product **9** (25.8 mg, 12%) and product **10** (detected by HPLC-MS).

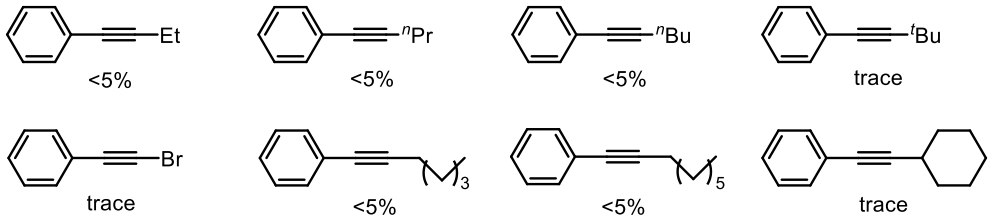


11. Proposed mechanism



Scheme S2. Proposed mechanism

12. Unsuccessful substrates



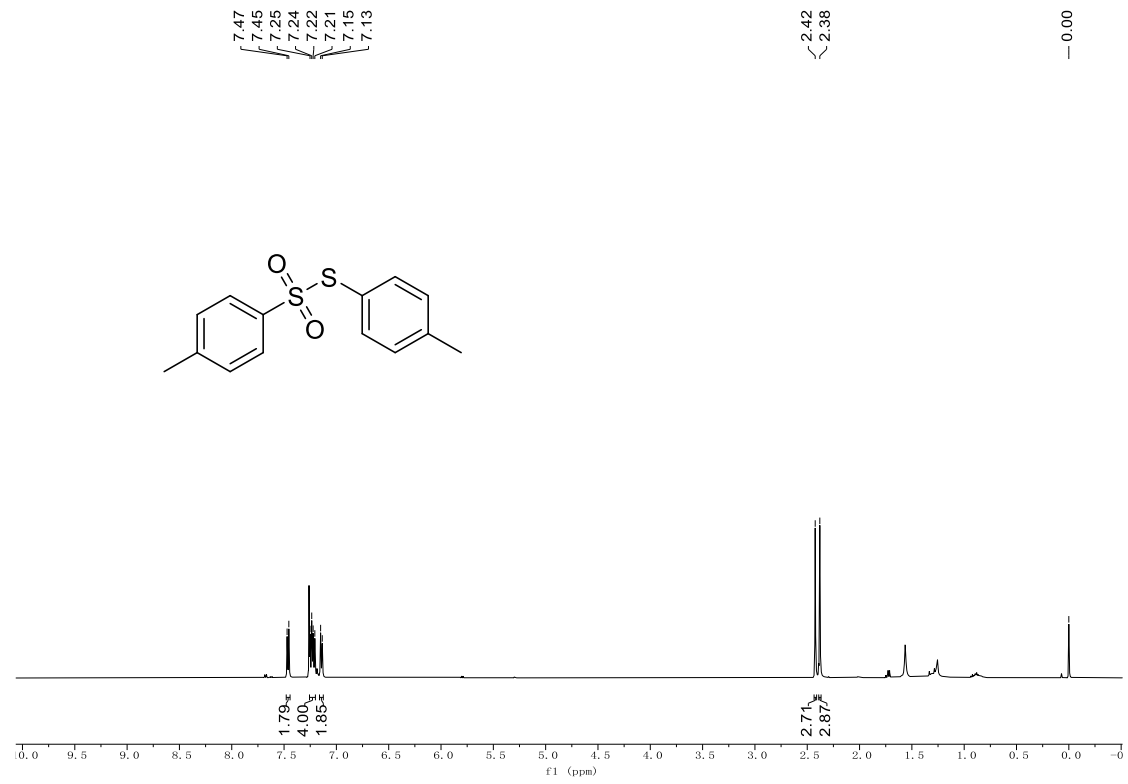
13. The reaction promoted by external oxidants

Table S8. The transformation promoted by external oxidants

Entry	Conditions	Yield
1	TBAI (20 mol%), TBHP (2.0 equiv.), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), 80 °C	trace
2	I_2 (50 mol%), TBHP (2.0 equiv.), HFIP (3.0 mL), $CHCl_3$ (2.0 mL), 80 °C	0
3	$FeCl_3$ (10 mol%), air, HFIP (3.0 mL), $CHCl_3$ (2.0 mL), 80 °C	0
4	CAN (2.0 equiv.), air, HFIP (3.0 mL), $CHCl_3$ (2.0 mL), RT	0
5	PCC (2.0 equiv.), air, HFIP (3.0 mL), $CHCl_3$ (2.0 mL), RT	0
6	$KMnO_4$ (10 mol%), air, HFIP (3.0 mL), $CHCl_3$ (2.0 mL), RT	0

RT = room temperature.

Substrate **1a** hardly took part in the reaction and the recovery was 92%. **2a** was consumed. Moreover, the self-coupling product of sulfonyl hydrazide (S-(p-tolyl) 4-methylbenzenesulfonothioate) was appeared in the reaction.



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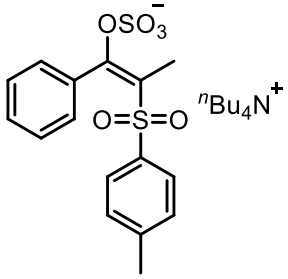
14. Characterization of products

(E)-1-phenyl-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3aa)



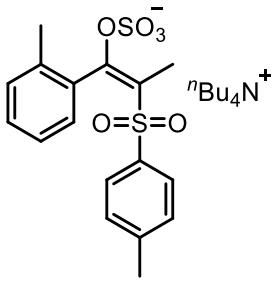
3aa was obtained in 72% (87.7 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 – 7.38 (m, 4H), 7.27 (m, 1H), 7.19 (t, *J* = 7.5 Hz, 2H), 7.12 (d, *J* =8.0 Hz, 2H), 3.06 – 3.03 (m, 8H), 2.35 (s, 3H), 2.26 (s, 3H), 1.51 – 1.45 (m, 8H), 1.34 – 1.27 (m, 8H), 0.93 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.9, 143.2, 138.7, 135.0, 130.6, 129.1, 128.7, 128.60, 127.4, 126.6, 58.5, 23.8, 21.4, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{15}O_6S_2^-$: 367.0316, found 367.0313.

(E)-1-(o-tolyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ba)



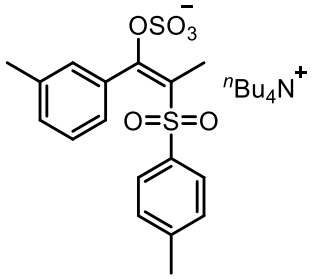
3ba was obtained in 51% (63.6 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 – 7.32 (m, 3H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.11 (d, *J* = 7.9 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.96 (d, *J* = 7.5 Hz, 1H), 3.07 – 3.04 (m, 8H), 2.36 (s, 3H), 2.29 (s, 3H), 2.07 (s, 3H), 1.52 – 1.46 (m, 8H), 1.36 –1.28 (m, 8H), 0.93 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.8, 143.2, 138.6, 137.9, 133.7, 132.1, 129.0, 128.8, 128.4, 127.4, 123.8, 58.5, 23.8, 21.5, 19.8, 19.6, 14.0, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{17}O_6S_2^-$: 381.0472, found 381.0464.

(E)-1-(m-tolyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ca)



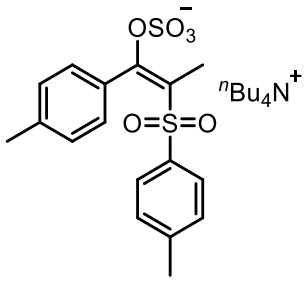
3ca was obtained in 55% (68.6 mg) as a colorless oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (d, *J* = 8.2 Hz, 3H), 7.21 (d, *J* = 7.3 Hz, 1H), 7.10 – 7.05 (m, 5H), 3.11 – 3.03 (m, 8H), 2.35 (s, 3H), 2.28 (s, 3H), 2.22 (s, 3H), 1.58 – 1.46 (m, 8H), 1.35 – 1.28 (m, 8H), 0.93 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.9, 143.0, 138.7, 135.9, 134.6, 130.9, 129.5, 129.1, 129.0, 128.0, 127.4, 126.5, 58.5, 23.8, 21.4, 21.3, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{17}O_6S_2^-$: 381.0472, found 381.0474.

(E)-1-(p-tolyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3da)



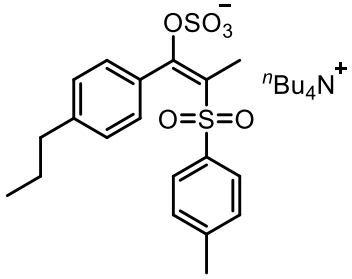
3da was obtained in 60% (74.8 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 3.9 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 7.7 Hz, 2H), 3.10 – 3.07 (m, 8H), 2.36 (s, 3H), 2.30 (s, 3H), 2.21 (s, 3H), 1.54 – 1.48 (m, 8H), 1.37 – 1.29 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.5, 143.3, 138.9, 138.3, 131.5, 130.5, 129.1, 128.7, 127.54, 127.50, 58.5, 23.8, 21.5, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{17}O_6S_2^-$: 381.0472, found 381.0473.

(E)-1-(4-propylphenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ea)



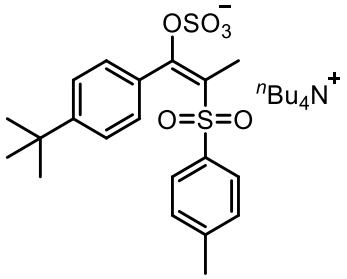
3ea was obtained in 50% (65.1 mg) as a colorless oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (d, *J* = 8.4 Hz, 2H), 7.28 – 7.26 (m, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.97 (d, *J* = 8.5 Hz, 2H), 3.10 – 3.07 (m, 8H), 2.54 – 2.51 (m, 2H), 2.33 (s, 3H), 2.27 (s, 3H), 1.62 (q, *J* = 7.3 Hz, 2H), 1.54 – 1.48 (m, 8H), 1.36 – 1.29 (m, 8H), 0.94 (m, 15H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.8, 143.5, 143.0, 138.5, 131.8, 130.5, 129.1, 129.0, 127.4, 126.8, 58.5, 38.0, 24.4, 23.8, 21.5, 19.6, 14.6, 14.0, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{19}H_{21}O_6S_2^-$: 409.0785, found 409.0785.

(E)-1-(4-(tert-butyl)phenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3fa)



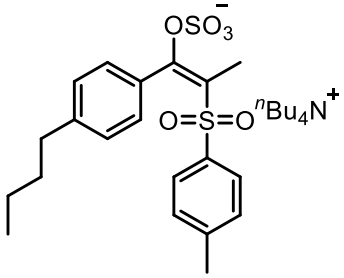
3fa was obtained in 60% (79.8 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30 – 7.25 (m, 4H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 3.13 – 3.10 (m, 8H), 2.32 (s, 3H), 2.31 (s, 3H), 1.56 – 1.50 (m, 8H), 1.38 – 1.32 (m, 8H), 1.28 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.3, 151.8, 142.9, 138.3, 131.2, 130.3, 130.1, 128.9, 127.3, 123.8, 58.5, 34.6, 31.3, 23.8, 21.4, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{20}H_{23}O_6S_2^-$: 423.0938, found 423.0942.

(E)-1-(4-butylphenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ga)



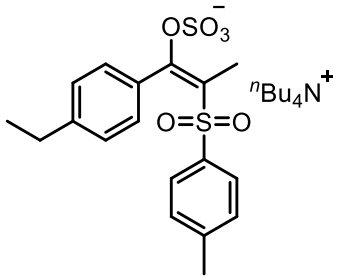
3ga was obtained in 61% (81.2 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (d, *J* = 8.2 Hz, 2H), 7.29 – 7.26 (m, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.96 (d, *J* = 8.1 Hz, 2H), 3.10 – 3.07 (m, 8H), 2.55 (t, *J* = 7.8 Hz, 2H), 2.33 (s, 3H), 2.29 (s, 3H), 1.58 (t, *J* = 7.7 Hz, 2H), 1.54 – 1.47 (m, 8H), 1.40 – 1.28 (m, 10H), 0.96 – 0.92 (m,15H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.9, 143.6, 143.0, 138.6, 131.9, 130.5, 128.9, 127.3, 126.7, 58.4, 35.6, 33.5, 23.8, 22.5, 21.5, 19.6, 14.6, 14.0, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{20}H_{23}O_6S_2^-$: 423.0942, found 423.0937.

(E)-1-(4-ethylphenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ha)



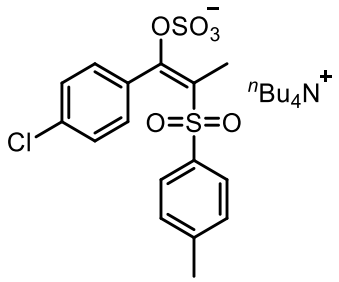
3ha was obtained in 68% (86.7 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 7.8 Hz, 2H), 3.13 – 3.06 (m, 8H), 2.60 (q, *J* = 7.6 Hz, 2H), 2.34 (s, 3H), 2.27 (s, 3H), 1.54 – 1.48 (m, 8H), 1.37 – 1.29 (m, 8H), 1.22 (t, *J* = 7.6 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.0, 144.8, 143.0, 138.6, 132.1, 130.6, 129.0, 128.7, 127.4, 126.2, 58.5, 28.8, 23.8, 21.5, 19.6, 15.4, 14.6, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{18}H_{19}O_6S_2^-$: 395.0629, found 395.0635.

(E)-1-(4-chlorophenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ia)



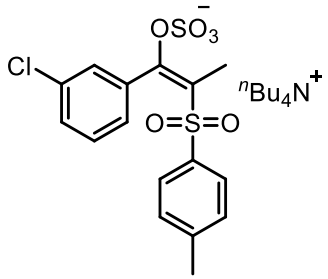
3ia was obtained in 59% (75.9 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.18 – 7.15 (m, 4H), 3.08 – 3.05 (m, 8H), 2.38 (s, 3H), 2.23 (s, 3H), 1.54 – 1.48 (m, 8H), 1.37 – 1.29 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.4, 143.6, 138.3, 134.7, 133.4, 132.1, 129.2, 128.9, 127.4, 126.9, 58.5, 23.8, 21.5, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{14}H_{16}ClO_6S_2^-$: 400.9926, found 400.9918.

(E)-1-(3-chlorophenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ja)



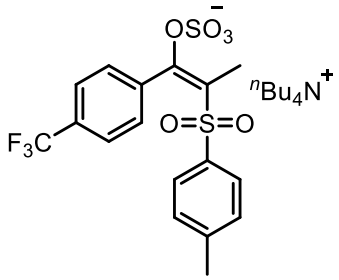
3ja was obtained in 58% (74.6 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.24 – 7.21 (m, 2H), 7.16 (t, *J* = 8.1 Hz, 3H), 3.09 – 3.06 (m, 8H), 2.38 (s, 3H), 2.28 (s, 3H), 1.54 – 1.48 (m,8H), 1.36 – 1.29 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 155.9, 143.6, 138.3, 136.7, 132.3, 130.3, 129.9, 129.22, 129.19, 128.7, 128.0, 127.4, 58.5, 23.8, 21.5, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{14}ClO_6S_2^-$: 400.9926, found 400.9919.

(E)-2-tosyl-1-(4-(trifluoromethyl)phenyl)prop-1-en-1-yl sulfate tetrabutylammonium (3ka)



3ka was obtained in 38% (51.5 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

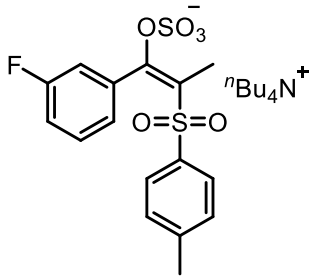
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.41 (d, *J* = 8.2 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 3.11 – 3.08 (m, 8H), 2.36 (s, 3H), 2.28 (s, 3H), 1.56 – 1.50 (m, 8H), 1.38 – 1.31 (m, 8H), 0.95 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 143.6, 138.7, 138.2, 133.6, 131.0, 130.1, 129.7, 129.2, 129.1, 128.3, 127.4, 127.3, 123.51, 123.47, 58.6, 23.8, 21.4, 19.5, 14.3, 13.5.

^{19}F NMR (376 MHz, $CDCl_3$): δ -63.26.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{14}F_3O_6S_2^-$: 435.0189, found 435.0178.

(E)-1-(3-fluorophenyl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3la)



3la was obtained in 48% (60.2 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

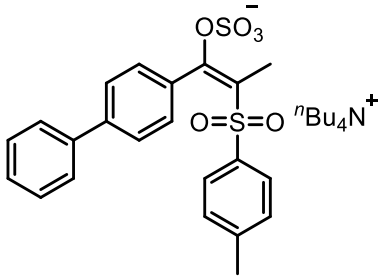
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 6.3 Hz, 2H), 7.22 – 7.15 (m, 4H), 7.07 (d, *J* = 9.3 Hz, 1H), 6.98 – 6.94 (m, 1H), 3.12 – 3.08 (m, 8H), 2.37 (s, 3H), 2.25 (s, 3H), 1.56 – 1.50 (m, 8H), 1.38 – 1.31 (m, 8H), 0.96 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 161.4, 159.4, 154.7, 142.5, 137.2, 135.8, 128.8, 128.2, 127.2, 126.5, 125.6, 116.6, 116.4, 114.7, 114.6, 57.6, 22.8, 20.5, 18.6, 13.4, 12.5.

^{19}F NMR (376 MHz, $CDCl_3$): δ -114.9.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{14}FO_6S_2^-$: 385.0221, found 385.0224.

(E)-1-([1,1'-biphenyl]-4-yl)-2-tosylprop-1-en-1-yl sulfate tetrabutylammonium (3ma)



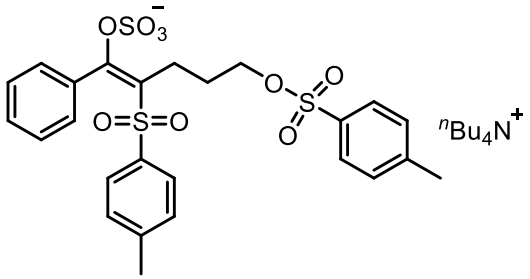
3ma was obtained in 52% (71.3 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.6 Hz, 2H), 7.45 – 7.39 (m, 8H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 2H), 3.07 – 3.03 (m, 8H), 2.33 (s, 3H), 2.27 (s, 3H), 1.51 – 1.45 (m, 8H), 1.34 – 1.26 (m, 8H), 0.90 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.2, 143.2, 141.2, 140.74, 140.71, 138.5, 133.7, 131.2, 129.4, 129.0, 128.7, 127.4, 127.4, 127.0, 125.3, 58.5, 23.8, 21.5, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{22}H_{19}O_6S_2^-$: 443.0629, found 443.0627.

(E)-1-phenyl-2-tosyl-5-(tosyloxy)pent-1-en-1-yl sulfate tetrabutylammonium (3na)



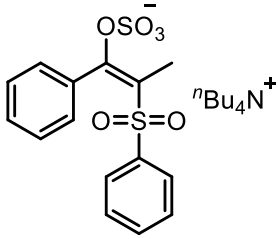
3na was obtained in 40% (64.6 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.24 – 7.20 (m, 5H), 7.10 (t, *J* = 7.5 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 4.11 (t, *J* = 6.5 Hz, 2H), 3.08 – 3.04 (m, 8H), 2.78 – 2.74 (m, 2H), 2.44 (s, 3H), 2.32 (s, 3H), 2.10 (t, *J* = 7.7 Hz, 2H), 1.53 – 1.47 (m,8H), 1.35 – 1.28 (m, 8H), 0.92 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 159.4, 144.7, 143.1, 138.7, 133.8, 132.9, 131.5, 130.8, 129.9, 129.0, 128.0, 127.3, 126.6, 71.2, 58.5, 28.4, 24.4, 23.8, 21.6, 21.4, 19.6, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{25}H_{25}O_9S_2^-$: 565.0666, found 565.0660.

(E)-1-phenyl-2-(phenylsulfonyl)prop-1-en-1-yl sulfate tetrabutylammonium (3ab)



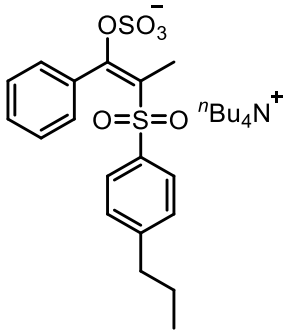
3ab was obtained in 60% (71.4 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53 – 7.51 (m, 2H), 7.46 – 7.43 (m, 1H), 7.42 – 7.39 (m, 2H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.30 – 7.27 (m, 1H), 7.26 –7.24 (m, 1H), 7.18 (t, *J* =7.4 Hz, 2H), 3.12 – 3.08 (m, 8H), 2.31 (s, 1H), 1.57 – 1.49 (m, 8H), 1.40 – 1.30 (m, 8H), 0.96 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.3, 141.5, 134.8, 132.4, 130.6, 128.8, 128.47, 128.45, 127.3, 126.7, 58.5, 23.8, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{13}O_6S_2^-$: 353.0159, found 353.0155.

(E)-1-phenyl-2-((4-propylphenyl)sulfonyl)prop-1-en-1-yl sulfate tetrabutylammonium (3ac)



3ac was obtained in 70% (89.2mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

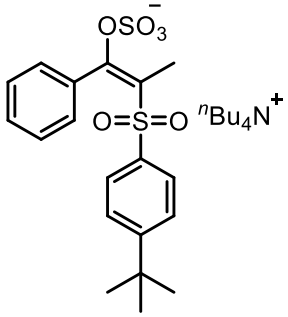
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 (m, 4H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 3.07 – 3.04 (m, 8H), 2.58 (t, *J* = 7.6 Hz, 2H), 2.28 (s, 1H), 1.61 (q, *J* = 7.4 Hz, 2H), 1.52 – 1.46 (m, 8H), 1.35 – 1.28 (m, 8H), 0.94 – 0.91 (m, 15H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.8, 147.8, 138.8, 134.9, 130.6, 128.8, 128.7, 128.5,

127.3, 126.6, 58.5, 37.8, 24.2, 23.8, 19.6, 14.5, 13.7, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{18}H_{19}O_6S_2^-$: 395.0629, found 395.0615.

(E)-2-((4-(tert-butyl)phenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3ad)



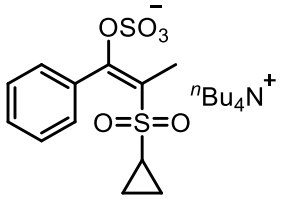
3ad was obtained in 71% (92.5 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.6 Hz, 2H), 7.37 (d, *J* = 6.9 Hz, 2H), 7.31(d, *J* = 8.7 Hz, 2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 2H), 3.09 – 3.06 (m, 8H), 2.30 (s, 3H), 1.64 – 1.48 (m, 8H), 1.37 – 1.31 (m, 8H), 1.29 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.4, 156.1, 138.2, 134.6, 130.6, 129.2, 128.8, 127.2, 126.7, 125.5, 58.5, 35.0, 31.0, 23.8, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{19}H_{21}O_6S_2^-$: 409.0785, found 409.0775.

(E)-2-(cyclopropylsulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3ae)



3ae was obtained in 40% (44.8 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

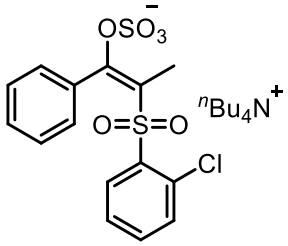
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.3 Hz, 2H), 7.32 – 7.27 (m, 3H), δ 3.11 – 3.08 (m, 8H), 2.34 (s, 3H), 2.15 – 2.10 (m, 1H), 1.56 – 1.49 (m, 8H), 1.39 – 1.32 (m, 8H), 1.04 – 1.00 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 12H), 0.82 – 0.80 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.9, 135.1, 130.6, 129.0, 128.7, 126.9, 58.5, 31.5,

23.8, 19.6, 14.5, 13.6, 5.2.

HRMS (ESI-TOF) m/z: Calcd for $C_{12}H_{13}O_6S_2^-$: 317.0159, found 317.0150.

(E)-2-((2-chlorophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3af)



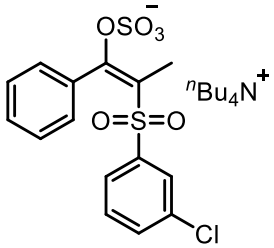
3af was obtained in 53% (66.7 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 – 7.20 (m, 5H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 2H), 6.93 – 6.89 (m, 1H), 3.08 – 3.12 (m, 8H), 2.43 (s, 3H), 1.57 – 1.49 (m, 8H), 1.40 – 1.30 (m, 8H), 0.96 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.6, 138.9, 133.8, 132.9, 131.9, 130.8, 130.6, 130.5, 128.8, 128.7, 126.6, 126.2, 58.6, 23.8, 19.6, 13.6, 13.5.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}ClO_6S_2^-$: 386.9769, found 386.9760.

(E)-2-((3-chlorophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3ag)



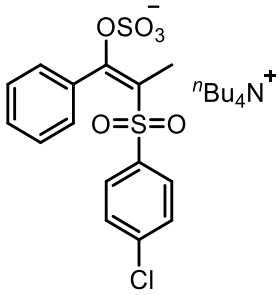
3ag was obtained in 51% (64.2 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (d, *J* = 7.8 Hz, 1H), 7.38 – 7.36 (m, 4H), 7.30 – 7.23 (m, 2H), 7.19 (t, *J* = 7.5 Hz, 2H), 3.14 – 3.10 (m, 8H), 2.34 (s, 3H), 1.58 – 1.51 (m, 8H), 1.37 (m, 8H), 0.97 (t, J = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 143.3, 134.4, 134.3, 132.5, 130.7, 129.8, 129.1, 128.4, 127.5, 126.7, 125.4, 58.5, 23.8, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}ClO_6S_2^-$: 386.9769, found 386.9774.

(E)-2-((4-chlorophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3ah)



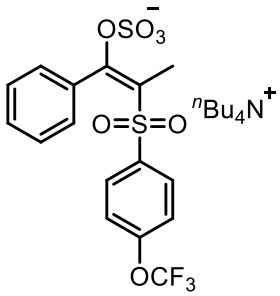
3ah was obtained in 52% (65.4 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 6.9 Hz, 1H), 7.29 – 7.25 (m, 3H), 7.18 (t, *J* = 7.5 Hz, 2H), 3.08 – 3.05 (m, 8H), 2.30 (s, 3H), 1.53 – 1.47 (m, 8H), 1.36 – 1.29 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 158.5, 140.0, 138.8, 134.6, 130.6, 129.0, 128.7, 128.64, 128.60, 126.8, 58.5, 23.8, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}ClO_6S_2^-$: 386.9769, found 386.9764.

(E)-1-phenyl-2-((4-(trifluoromethoxy)phenyl)sulfonyl)prop-1-en-1-yl sulfate tetrabutylammonium (3ai)



3ai was obtained in 50% (67.9 mg) as a colorless oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (d, *J* = 8.8 Hz, 2H), 7.34 (d, *J* = 6.8 Hz, 2H), 7.25 (m, 1H), 7.15 (t, *J* = 7.6 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 3.10 – 3.07 (m, 8H), 2.35 (s, 3H), 1.55 – 1.48 (m, 8H), 1.37 – 1.33 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

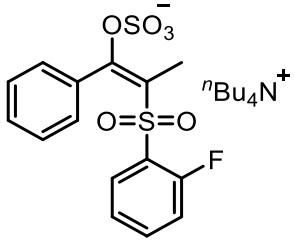
^{13}C NMR (125 MHz, $CDCl_3$): δ 158.6, 151.7, 140.0, 134.6, 130.6, 129.3, 128.9, 128.8,

126.8, 121.2, 120.3, 119.1, 58.5, 23.7, 19.5, 14.3, 13.5.

^{19}F NMR (376 MHz, $CDCl_3$): δ -57.68.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{12}F_3O_7S_2^-$: 436.9982, found 436.9975.

(E)-2-((2-fluorophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3aj)



3aj was obtained in 50% (61.3 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

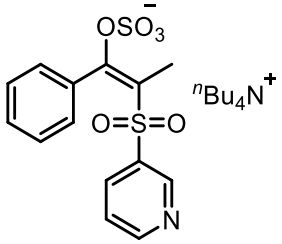
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 – 7.34 (m, 1H), 7.29 (d, *J* = 8.3 Hz, 2H), 7.20 – 7.15 (m, 2H), 7.07 (m, 3H), 7.03 – 6.99 (m, 1H), 6.87 (t, *J* = 7.3 Hz, 1H), 3.14 – 3.11 (m, 8H), 2.39 (s, 3H), 1.58 – 1.51 (m, 8H), 1.40 – 1.33 (m, 8H), 0.97 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.2, 158.6, 157.7, 134.7, 134.6, 134.0, 130.6, 129.7, 129.6, 129.4, 129.3, 128.8, 126.6, 123.63, 123.59, 116.5, 116.3, 58.5, 23.8, 19.6, 13.6.

^{19}F NMR (376 MHz, $CDCl_3$): δ -108.50.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}FO_6S_2^-$: 371.0065, found 371.0059.

(E)-1-phenyl-2-(pyridin-3-ylsulfonyl)prop-1-en-1-yl sulfate tetrabutylammonium (3ak)



3ak was obtained in 52% (62.0 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

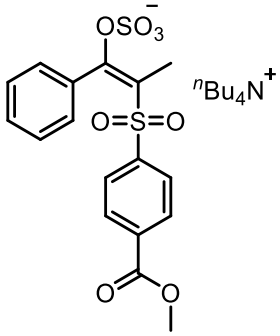
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.62 – 8.60 (m, 2H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.28 (m, 1H), 7.23 (m, 1H), 7.17 (t, *J* = 7.6 Hz, 2H), 3.12 – 3.08 (m,

8H), 2.36 (s, 3H), 1.56 – 1.49 (m, 8H), 1.37 – 1.30 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 159.3, 152.7, 148.1, 138.2, 134.8, 134.4, 130.7, 129.1, 128.4, 126.8, 123.1, 58.5, 23.8, 19.6, 14.3, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{14}H_{12}NO_6S_2^-$: 354.0112, found 354.0103.

(E)-2-((4-(methoxycarbonyl)phenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3al)



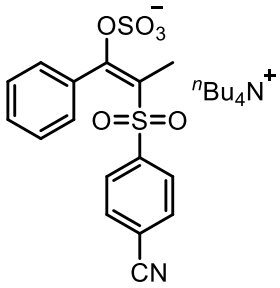
3al was obtained in 42% (54.9 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.5 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 2H), 3.93 (s, 3H), 3.10 – 3.06 (m, 8H), 2.33 (s, 3H), 1.54 – 1.48 (m, 8H), 1.35 – 1.29 (m, 8H), 0.94 (t, *J* = 7.5 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 165.7, 159.1, 145.7, 134.6, 133.3, 130.7, 129.5, 128.9, 128.1, 127.2, 126.7, 58.6, 52.5, 23.8, 19.6, 14.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{15}O_8S_2^-$: 411.0214, found 411.0206.

(E)-2-((4-cyanophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3am)



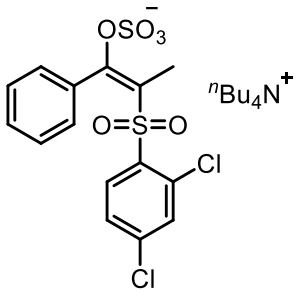
3am was obtained in 53% (65.8 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 (s, 3H), 7.33 (d, *J* = 7.5 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.16 (t, *J* = 7.5 Hz, 2H), 3.11 – 3.09 (m, 8H), 2.35 (s, 3H), 1.56 – 1.50 (m, 8H), 1.36 – 1.32 (m, 8H), 0.95 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 159.6, 146.0, 134.4, 132.0, 130.7, 129.1, 128.1, 127.8, 126.8, 117.5, 115.7, 58.6, 23.8, 19.6, 14.4, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{12}NO_6S_2^-$: 378.0112, found 378.0104.

(E)-2-((2,4-dichlorophenyl)sulfonyl)-1-phenylprop-1-en-1-yl sulfate tetrabutylammonium (3an)



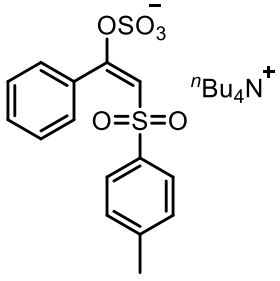
3an was obtained in 40% (53.1 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 (s, 1H), 7.23 (d, *J* = 7.6 Hz, 2H), 7.17 – 7.12 (m, 2H), 7.02 (t, *J* = 7.6 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 1H), 3.12 – 3.08 (m, 8H), 2.41 (s, 3H), 1.56 – 1.50 (m, 8H), 1.39 – 1.32 (m, 8H), 0.96 (t, *J* = 7.2 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 157.6, 138.7, 137.6, 133.6, 132.9, 131.5, 130.53, 130.51, 129.1, 128.9, 126.7, 126.4, 58.6, 23.8, 19.6, 13.6, 13.4.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{11}Cl_2O_6S_2^-$: 420.9380, found 420.9371.

(E)-1-phenyl-2-tosylvinyl sulfate tetrabutylammonium (4aa)



4aa was obtained in 51% (60.7 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

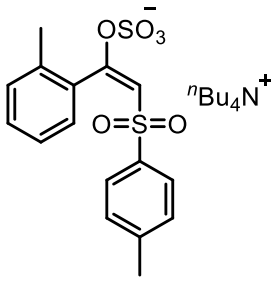
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.8 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 3H), 7.20

(t, *J* = 7.5 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 3H), 3.13 – 3.10 (m, 8H), 2.31 (s, 3H), 1.55 – 1.49 (m, 8H), 1.37 – 1.29 (m, 8H), 0.93 (t, *J* = 7.4 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 162.3, 143.0, 140.0, 133.2, 129.7, 129.5, 129.1, 127.3, 127.1, 113.7, 58.4, 23.7, 21.4, 19.6, 13.5.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{13}O_6S_2^-$: 353.0159, found 353.0159.

(E)-1-(o-tolyl)-2-tosylvinyl sulfate tetrabutylammonium (4ba)



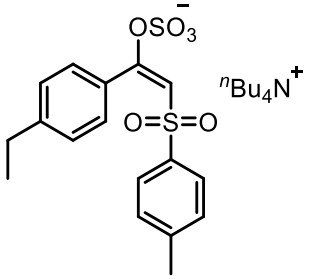
4ba was obtained in 37% (45.1 mg) as a canary yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28 (d, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.10 – 7.05 (m, 5H), 6.99 (d, *J* = 7.7 Hz, 1H), 3.13 – 3.10 (m, 8H), 2.35 (s, 3H), 1.98 (s, 3H), 1.56 – 1.49 (m, 8H), 1.38 – 1.30 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 163.1, 143.0, 139.9, 137.4, 132.5, 130.0, 129.5, 129.4, 128.9, 127.3, 124.5, 114.0, 58.4, 23.7, 21.4, 19.6, 18.9, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{16}H_{15}O_6S_2^-$: 367.0316, found 367.0308.

(E)-1-(4-ethylphenyl)-2-tosylvinyl sulfate tetrabutylammonium (4ca)



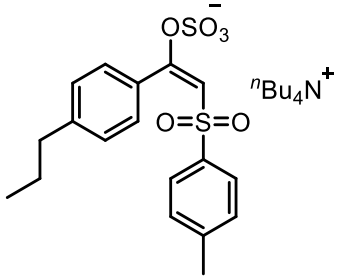
4ca was obtained in 32% (39.9mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.8 Hz, 2H), 7.28 (m, 2H), 7.09 – 7.01 (m, 5H), 3.16 – 3.13 (m, 8H), 2.61 (q, *J* = 7.7 Hz, 2H), 2.33 (s, 3H), 1.57 – 1.51 (m, 8H), 1.38 – 1.31 (m, 8H), 1.21 (t, *J* = 7.6 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 162.5, 146.2, 142.9, 140.1, 130.6, 129.6, 129.0, 127.1, 126.8, 113.1, 58.4, 28.8, 23.8, 21.4, 19.6, 15.5, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{17}H_{17}O_6S_2^-$: 381.0472, found 381.0489.

(E)-1-(4-propylphenyl)-2-tosylvinyl sulfate tetrabutylammonium (4da)



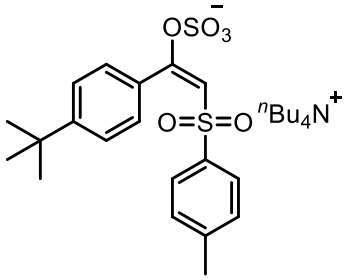
4da was obtained in 31% (39.5 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44 (d, *J* = 7.9 Hz, 2H), 7.28 – 7.26 (m, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 7.02 (d, *J* = 8.2 Hz, 2H), 3.17 – 3.14 (m, 8H), 2.55 (t, *J* = 7.6 Hz, 2H), 2.33 (s, 3H), 1.63 – 1.52 (m, 10H), 1.39 – 1.32 (m, 8H), 0.96 – 0.91 (m, 15H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 162.6, 144.6, 142.8, 140.1, 130.6, 129.5, 128.9, 127.4, 127.1, 113.1, 58.4, 37.9, 24.4, 23.8, 21.4, 19.6, 13.7, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{18}H_{19}O_6S_2^-$: 395.0629, found 395.0619.

(E)-1-(4-(tert-butyl)phenyl)-2-tosylvinyl sulfate tetrabutylammonium (4ea)



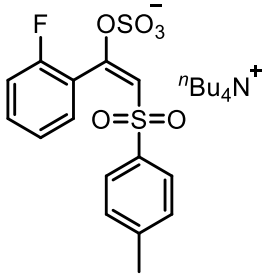
4ea was obtained in 35% (45.6 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (d, *J* = 7.9 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 7.03 (d, *J* = 7.6 Hz, 3H), 3.18 – 3.14 (m, 8H), 2.31 (s, 3H), 1.59 – 1.52 (m, 8H), 1.39 – 1.32 (m, 8H), 1.28 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 162.6, 153.0, 142.7, 139.9, 130.3, 129.3, 128.8, 127.2, 124.2, 113.3, 58.4, 34.7, 31.2, 23.7, 21.4, 19.6, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{19}H_{21}O_6S_2^-$: 409.0785, found 409.0779.

(E)-1-(2-fluorophenyl)-2-tosylvinyl sulfate tetrabutylammonium (4fa)



4fa was obtained in 40% (49.1 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

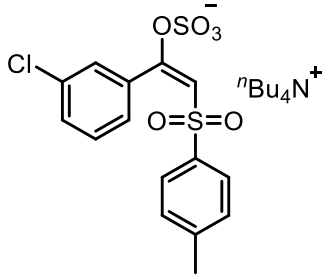
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 (d, *J* = 7.8 Hz, 2H), 7.29 – 7.25 (m, 1H), 7.20 – 7.17 (m, 2H), 7.09 (d, *J* = 7.8 Hz, 2H), 7.02 – 6.97 (m, 2H), 6.87 (t, *J* = 8.9 Hz, 1H), 3.10 – 3.07 (m, 8H), 2.30 (s, 3H), 1.52 – 1.46 (m, 8H), 1.32 – 1.25 (m, 8H), 0.87 (t, *J* = 7.4 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.7, 158.7, 157.0, 143.2, 140.0, 131.4, 131.3, 129.2, 127.1, 123.3, 123.2, 121.9, 121.7, 115.1, 114.9, 114.4, 58.5, 23.8, 21.5, 19.6, 13.6.

^{19}F NMR (376 MHz, $CDCl_3$): δ -112.7.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}FO_6S_2^-$: 371.0065, found 371.0062.

(E)-1-(3-chlorophenyl)-2-tosylvinyl sulfate tetrabutylammonium (4ga)



4ga was obtained in 42% (52.9 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

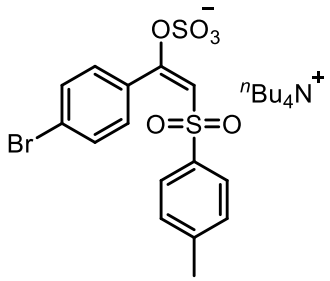
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.29 (m, 2H), 7.21 – 7.18 (m, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.06 (s, 1H), 3.16 – 3.12 (m, 8H), 2.37 (s, 3H), 1.59 – 1.52 (m, 8H), 1.39 – 1.32 (m, 8H), 0.95 (t, *J* = 7.4 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.2, 142.9, 139.3, 134.6, 132.8, 129.2, 128.74,

128.71, 128.2, 127.5, 126.6, 113.7, 58.0, 23.2, 21.0, 19.1, 13.1.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}ClO_6S_2^-$: 386.9769, found 386.9763.

(E)-1-(4-bromophenyl)-2-tosylvinyl sulfate tetrabutylammonium (4ha)



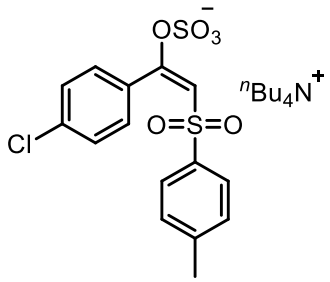
4ha was obtained in 47% (63.3 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (d, *J* = 8.2 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.28 – 7.25 (m, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.02 (s, 1H), 3.16 – 3.12 (m, 8H), 2.36 (s, 3H), 1.58 – 1.52 (m, 8H), 1.39 – 1.31 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 160.2, 142.4, 139.1, 131.5, 130.4, 129.6, 128.3, 126.1, 123.3, 112.9, 57.6, 22.9, 20.6, 18.7, 12.7.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}BrO_6S_2^-$: 430.9264, found 430.9254.

(E)-1-(4-chlorophenyl)-2-tosylvinyl sulfate tetrabutylammonium (4ia)



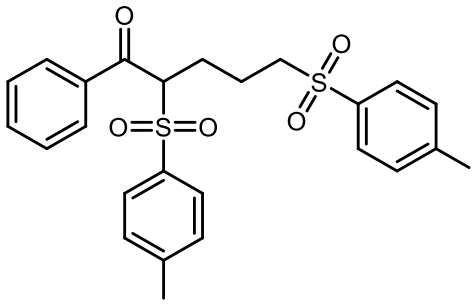
4ia was obtained in 49% (61.7 mg) as a yellow oil after column chromatography (eluent: ethyl acetate, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (d, *J* = 7.9 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.02 (s, 1H), 3.15 – 3.12 (m, 8H), 2.36 (s, 3H), 1.58 – 1.52 (m, 8H), 1.38 – 1.31 (m, 8H), 0.94 (t, *J* = 7.3 Hz, 12H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 161.1, 143.3, 140.0, 135.8, 131.9, 131.0, 129.2, 127.6, 127.0, 113.8, 58.5, 23.8, 21.5, 19.6, 13.6.

HRMS (ESI-TOF) m/z: Calcd for $C_{15}H_{12}ClO_6S_2^-$: 386.9769, found 386.9763.

1-phenyl-2,5-ditosylpentan-1-one (5)



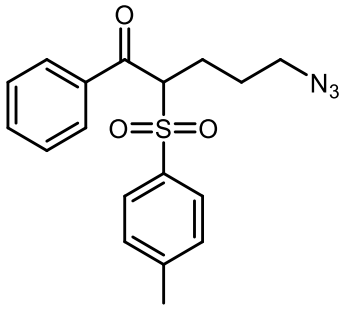
5 was obtained in 98%(46.1 mg) as yellow oil after column chromatography (eluent: petroleum ether/ethyl acetate = 2/1 v/v, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.0 Hz, 2H), 7.72 (d, *J* = 8.5 Hz, 2H), 7.62 – 7.59 (m, 3H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 5.06 (dd, *J* = 10.4, 4.1 Hz, 1H), 3.94 (t, *J* = 6.2 Hz, 2H), 2.45 (s, 3H), 2.42 (s, 3H), 2.19 – 2.03 (m, 1H), 1.64 – 1.57 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 192.1, 145.5, 145.0, 136.8, 134.1, 133.2, 132.7, 129.9, 129.7, 129.6, 129.1, 128.8, 127.9, 69.2, 69.0, 26.1, 24.5, 21.7, 21.6.

HRMS (ESI-TOF, [M + Na]$^+$): Calcd for $C_{25}H_{26}O_6S_2$: 509.1068, found 509.1067.

5-azido-1-phenyl-2-tosylpentan-1-one (6)



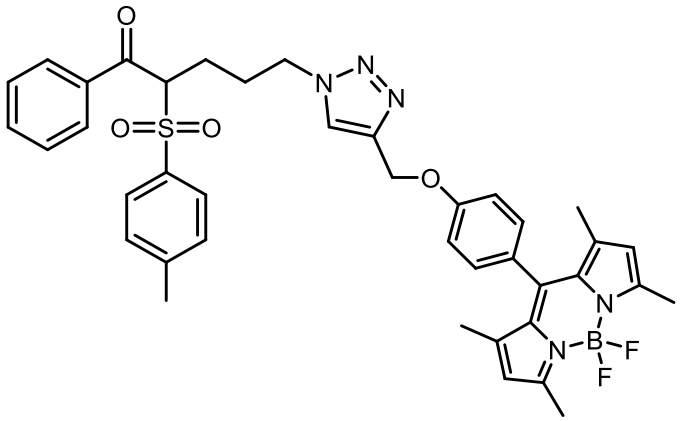
6 was obtained in 97% (69.3 mg) as a yellow oil after column chromatography (eluent: petroleum ether/ethyl acetate = 4/1 v/v, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.2 Hz, 2H), 7.62 (t, *J* = 8.9 Hz, 3H), 7.48 (t, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.11 (dd, *J* = 10.6, 3.8 Hz, 1H), 3.29 – 3.20 (m, 2H), 2.43 (s, 3H), 2.23 – 2.06 (m, 2H), 1.58 – 1.45 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 192.2, 145.5, 137.0, 134.1, 133.2, 129.8, 129.6, 129.1, 128.8, 69.5, 50.9, 29.7, 26.3, 25.7, 21.7.

HRMS (ESI-TOF, [M + Na]$^+$): Calcd for $C_{18}H_{19}N_3O_3S_2$: 380.1045, found 380.1045.

5-(4-((4-(5,5-difluoro-1,3,7,9-tetramethyl-5H-4l4,5l4-dipyrrolo[1,2-c:2',1'-f][1,3,2]diazaborinin-10-yl)phenoxy)methyl)-1H-1,2,3-triazol-1-yl)-1-phenyl-2-tosylpentan-1-one (7)



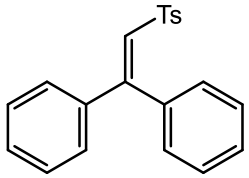
7 was obtained in 72% (105.8 mg) as an orange solid after column chromatography (eluent: petroleum ether/ethyl acetate = 1/1 v/v, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 (d, *J* = 7.5 Hz, 2H), 7.61 – 7.58 (m, 4H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.28 – 7.26 (m, 3H), 7.16 (d, *J* = 8.6 Hz, 2H), 7.08 (d, *J* = 8.7 Hz, 2H), 5.97 (s, 2H), 5.20 (s, 2H), 5.10 (dd, *J* = 8.5, 5.7 Hz, 1H), 4.34 (t, *J* = 7.0 Hz, 2H), 2.54 (s, 6H), 2.40 (s, 3H), 2.18 – 2.15 (m, 2H), 2.02 – 1.85 (m, 2H), 1.40 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 192.0, 158.8, 155.3, 145.7, 143.8, 143.1, 141.6, 136.7, 134.3, 133.2, 131.8, 129.7, 129.6, 129.3, 129.1, 128.8, 127.7, 122.8, 121.2, 115.4, 69.1, 62.0, 49.7, 27.5, 25.1, 21.7, 14.6.

HRMS (ESI-TOF, $[M + H]^+$): Calcd for $C_{40}H_{40}BF_2N_5O_4S$: 736.2940, found 736.2947.

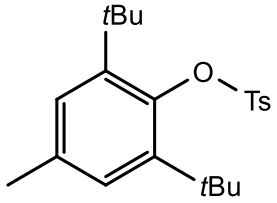
(2-tosylethene-1,1-diyl)dibenzene (8)[4]



8 was obtained in 30% (60.1 mg) as a white solid after column chromatography (eluent: petroleum ether/ethyl acetate = 20/1 v/v, Rf = 0.2).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.38 – 7.34 (m, 2H), 7.29 (t, *J* = 7.5 Hz, 4H), 7.20 (d, *J* = 7.1 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 6.7 Hz, 1H), 6.99 (s, 1H), 2.37 (s, 3H).

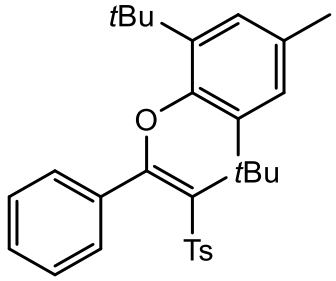
2,6-di-*tert*-butyl-4-methylphenyl 4-methylbenzenesulfonate (9)[4]



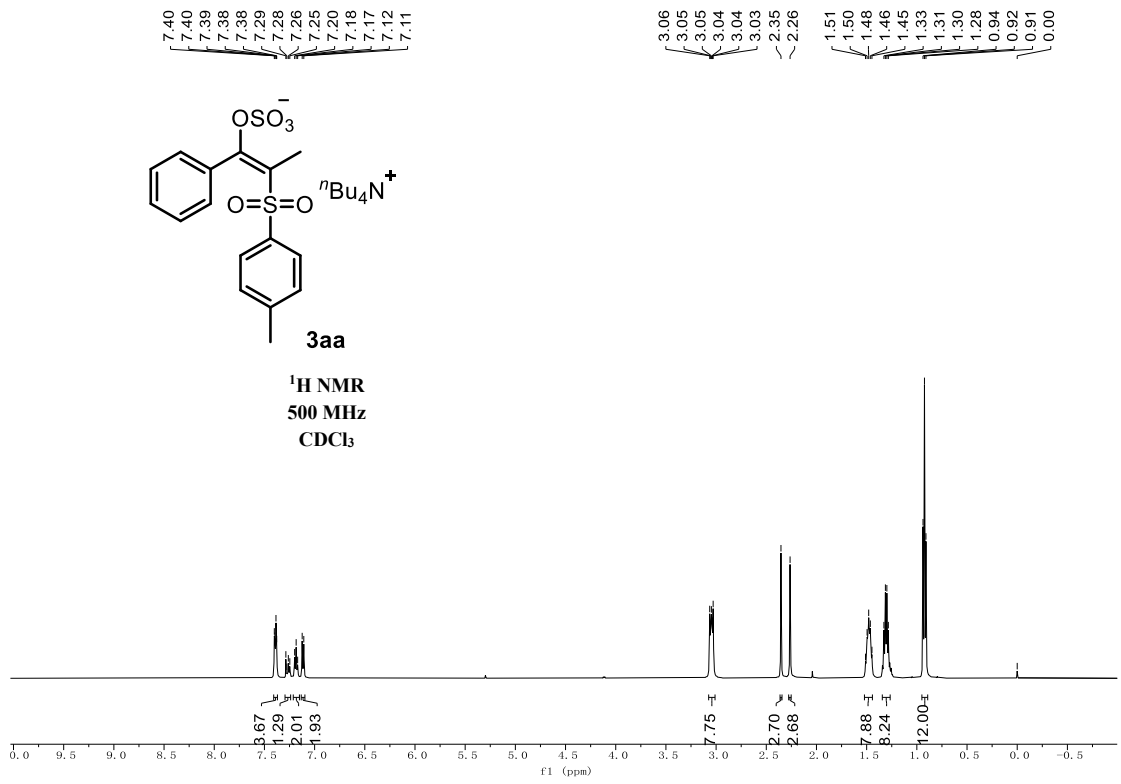
9 was obtained in 12% (25.8 mg) as a white solid after column chromatography (eluent: petroleum ether/ethyl acetate = 20/1 v/v, Rf = 0.3).

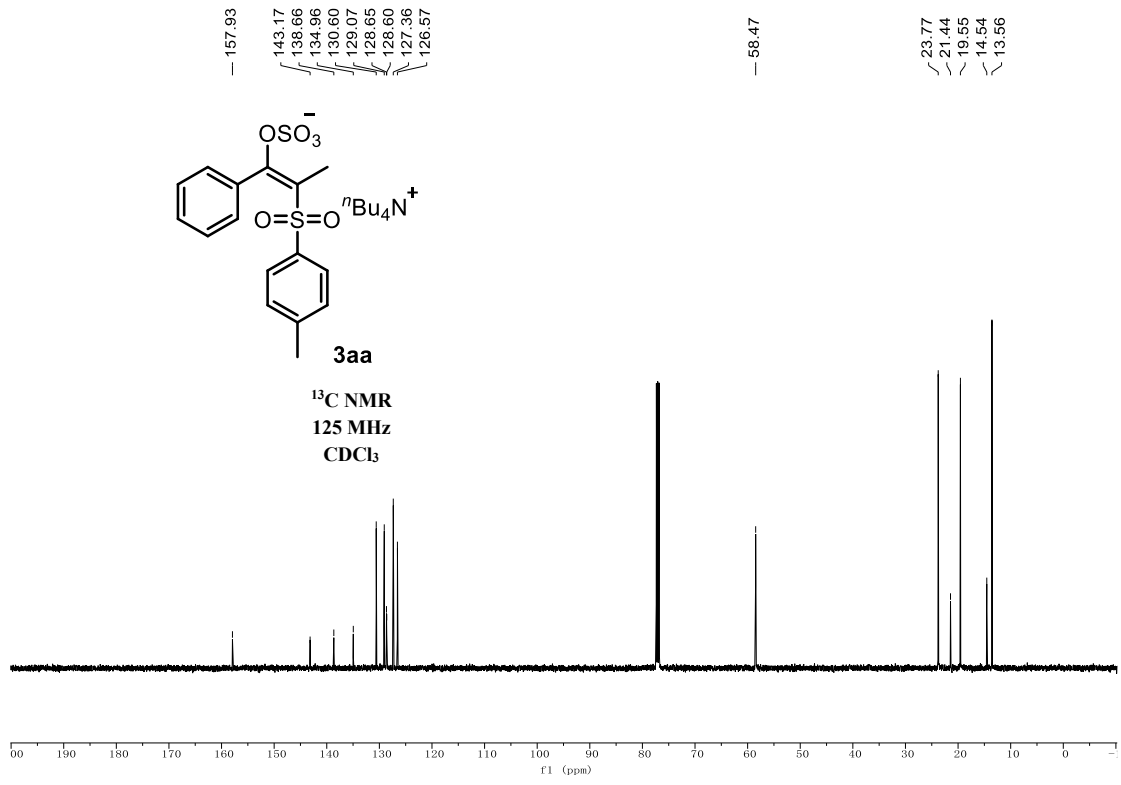
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 6.64 (s, 2H), 2.37 (s, 3H), 1.81 (s, 3H), 1.10 (s, 18H).

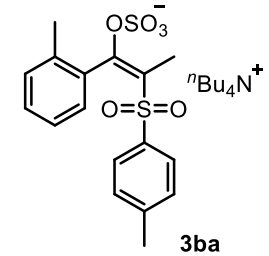
(E)-1,3-di-tert-butyl-5-methyl-2-((1-phenyl-2-tosylprop-1-en-1-yl)oxy)benzene (10)



HRMS (ESI-TOF, $[M + H]^+$): Calcd for $C_{31}H_{38}O_3S$: 491.2620, found 491.2639.

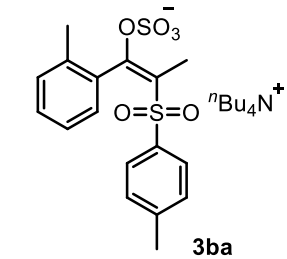

7.40
7.40
7.39
7.38
7.38
7.29
7.28
7.26
7.25
7.20
7.18
7.17
7.12
7.11
3.06
3.05
3.05
3.04
3.04
3.03
2.35
2.26
1.51
1.50
1.48
1.46
1.45
1.33
1.31
1.30
1.28
0.94
0.92
0.91
0.00
OSO3−
O=S=O
nBu4N+
3aa
1H NMR
500 MHz
CDCl3
3.67
1.29
2.01
1.93
7.75
2.70
2.68
7.88
8.24
12.00
f1 (ppm)


157.93
143.17
138.66
134.96
130.60
129.07
128.65
128.60
127.36
126.57
58.47
23.77
21.44
19.55
14.54
13.56
OSO3−
O=S=O
nBu4N+
3aa
13C NMR
125 MHz
CDCl3
f1 (ppm)



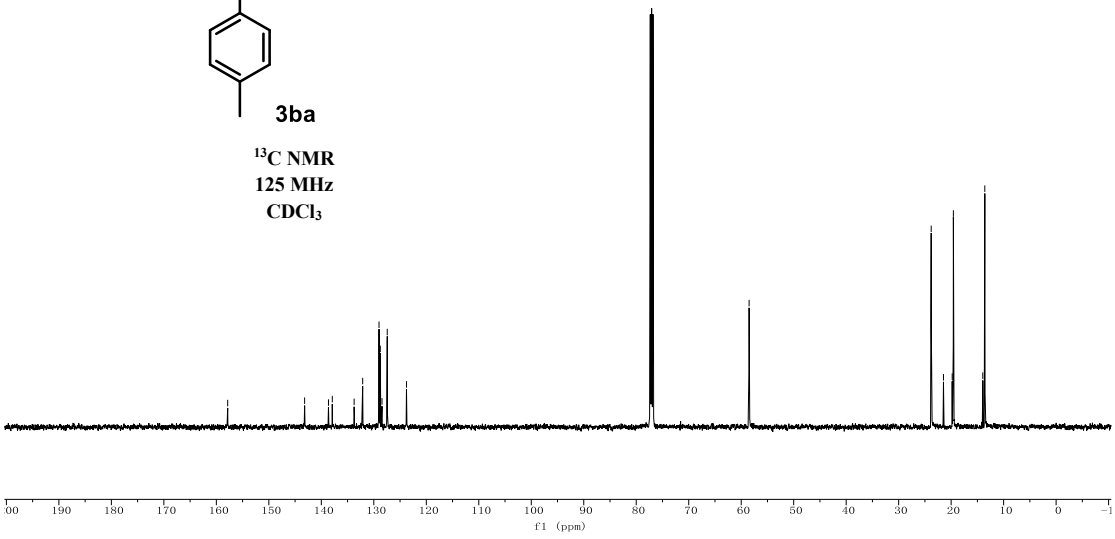
3ba

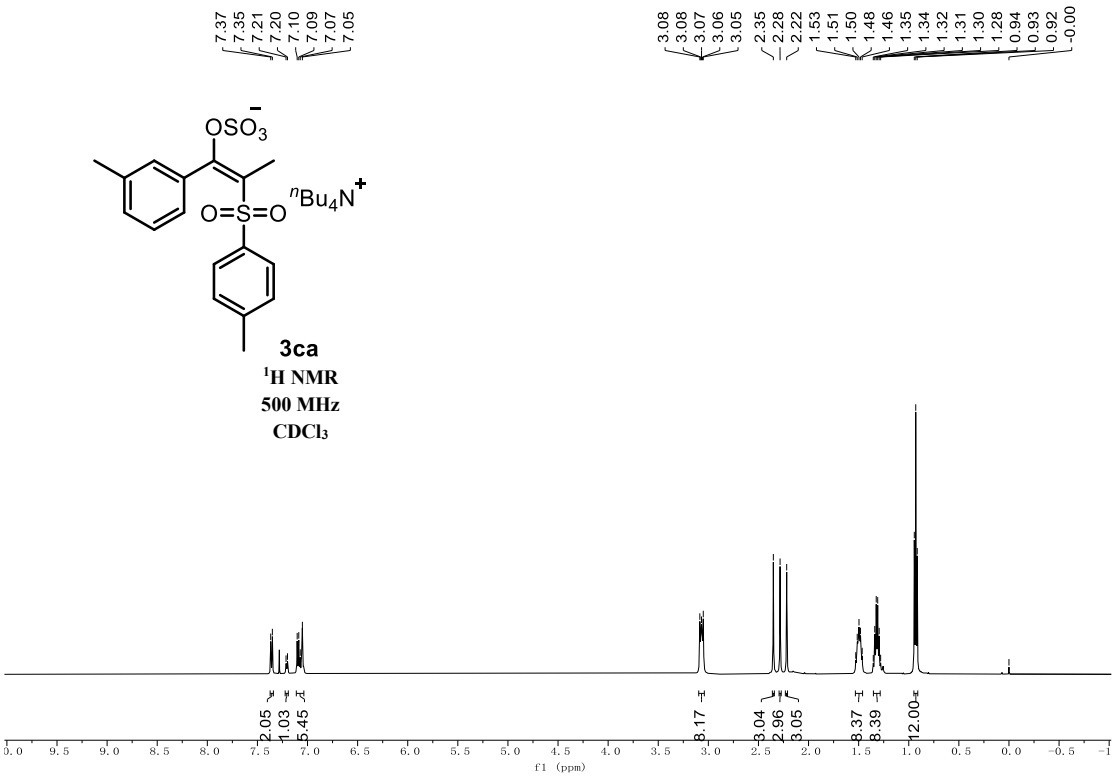
^{1}H NMR
500 MHz
$CDCl_3$



3ba

^{13}C NMR
125 MHz
$CDCl_3$



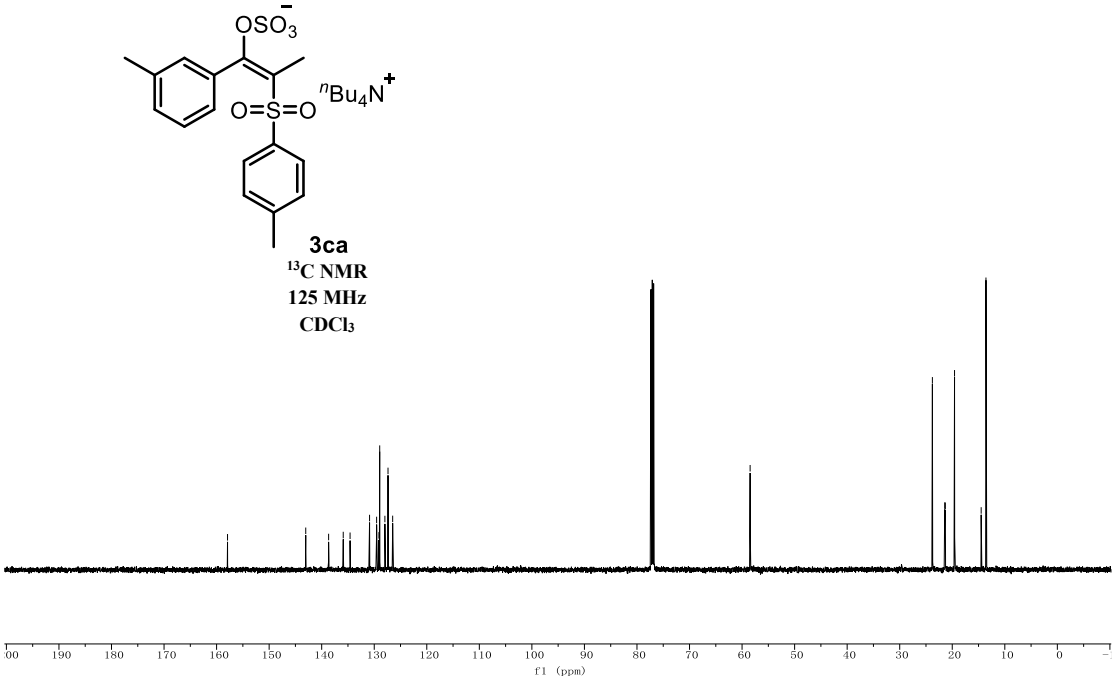


3ca

^{1}H NMR

500 MHz

$CDCl_3$

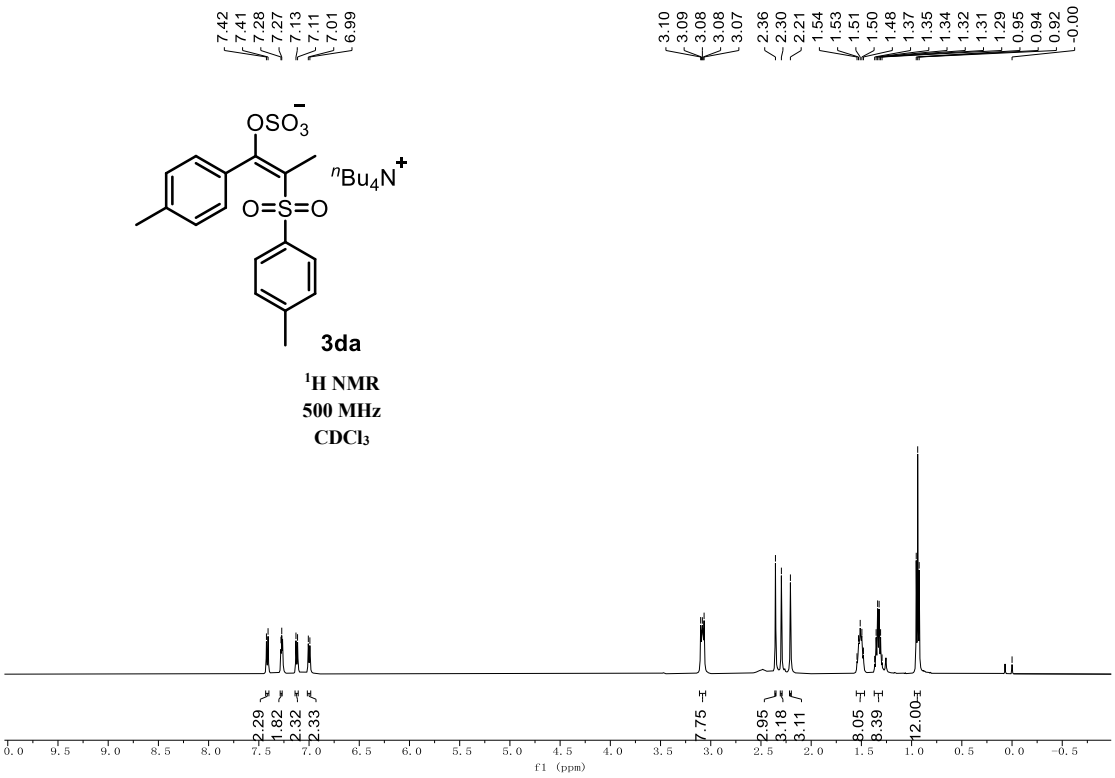


3ca

^{13}C NMR

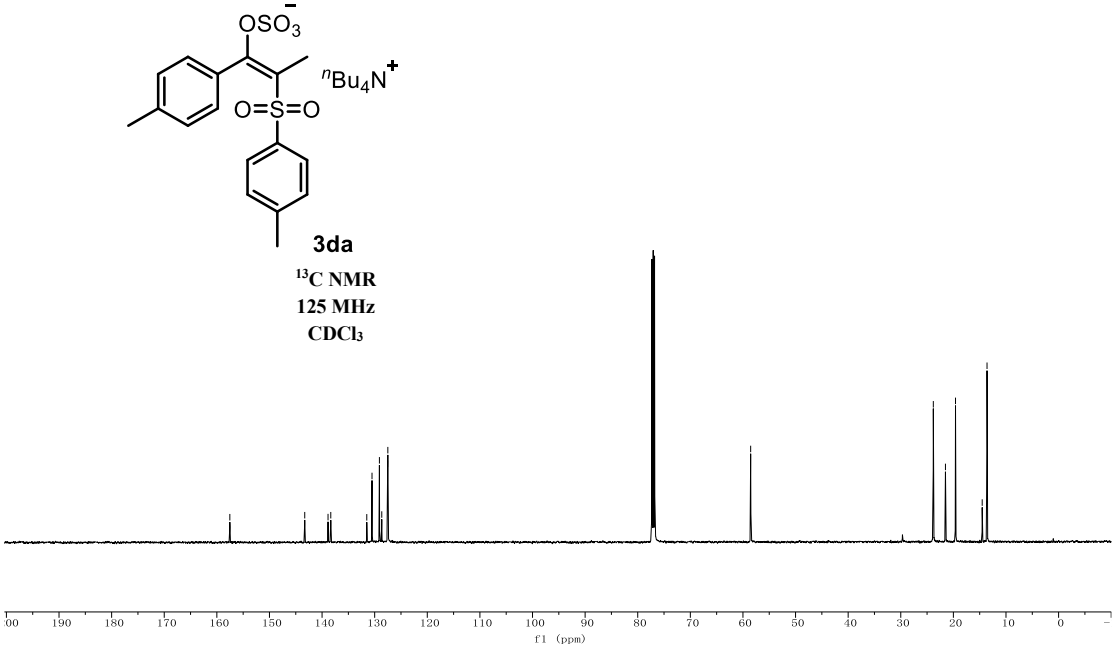
125 MHz

$CDCl_3$



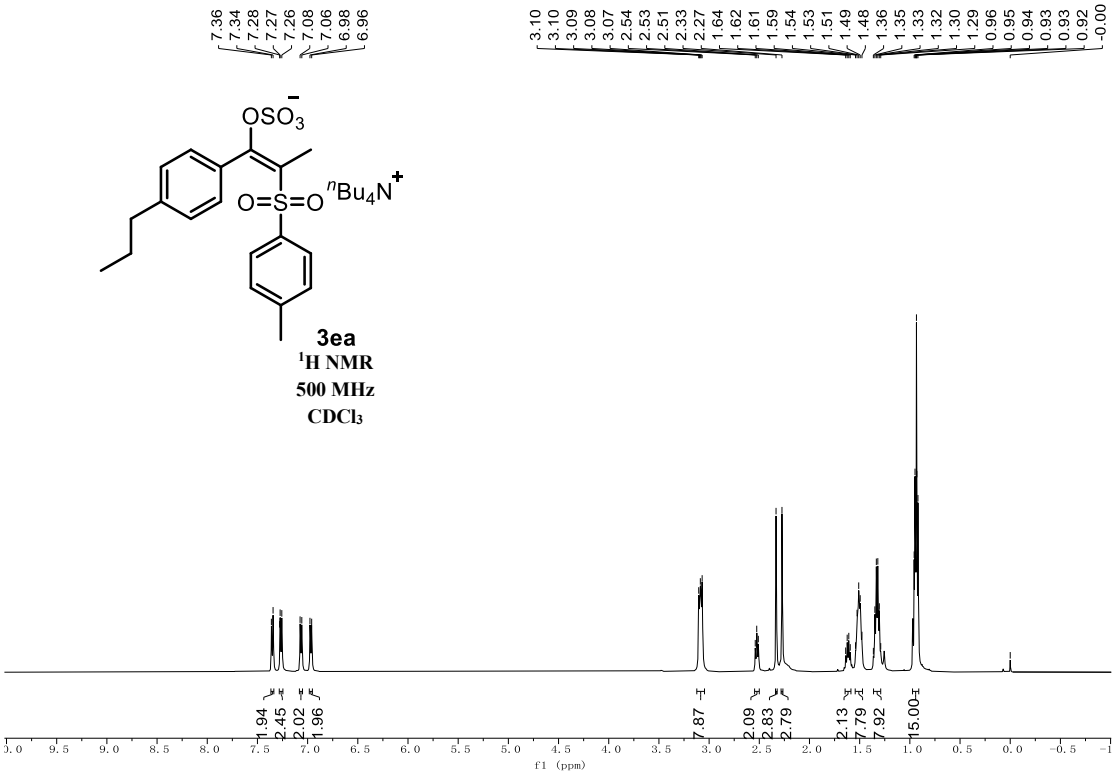
3da

^{1}H NMR
500 MHz
$CDCl_3$

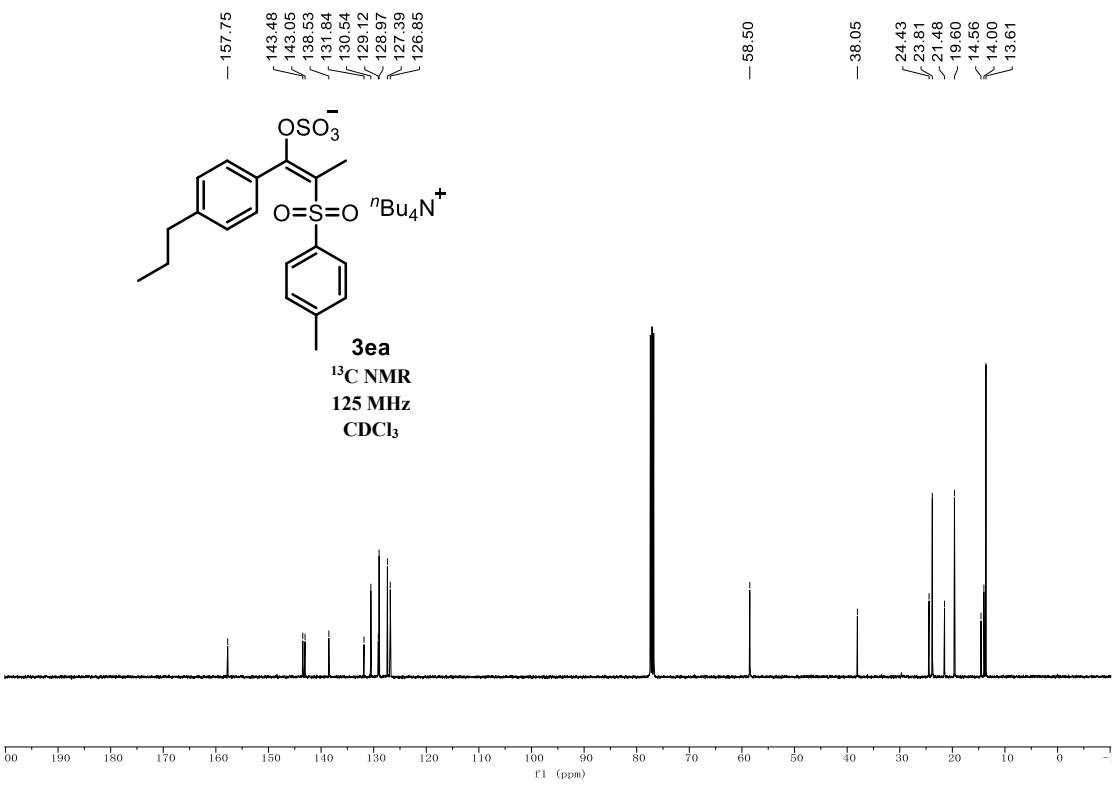


3da

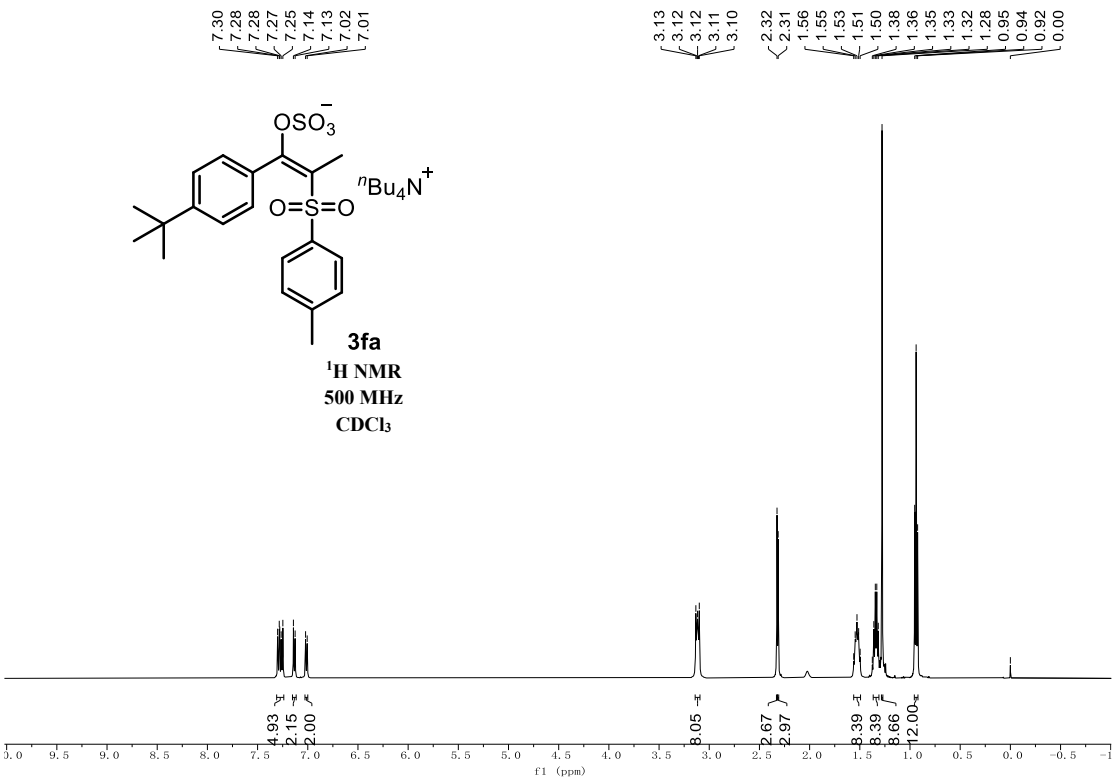
^{13}C NMR
125 MHz
$CDCl_3$



3ea
^{1}H NMR
500 MHz
$CDCl_3$



3ea
^{13}C NMR
125 MHz
$CDCl_3$

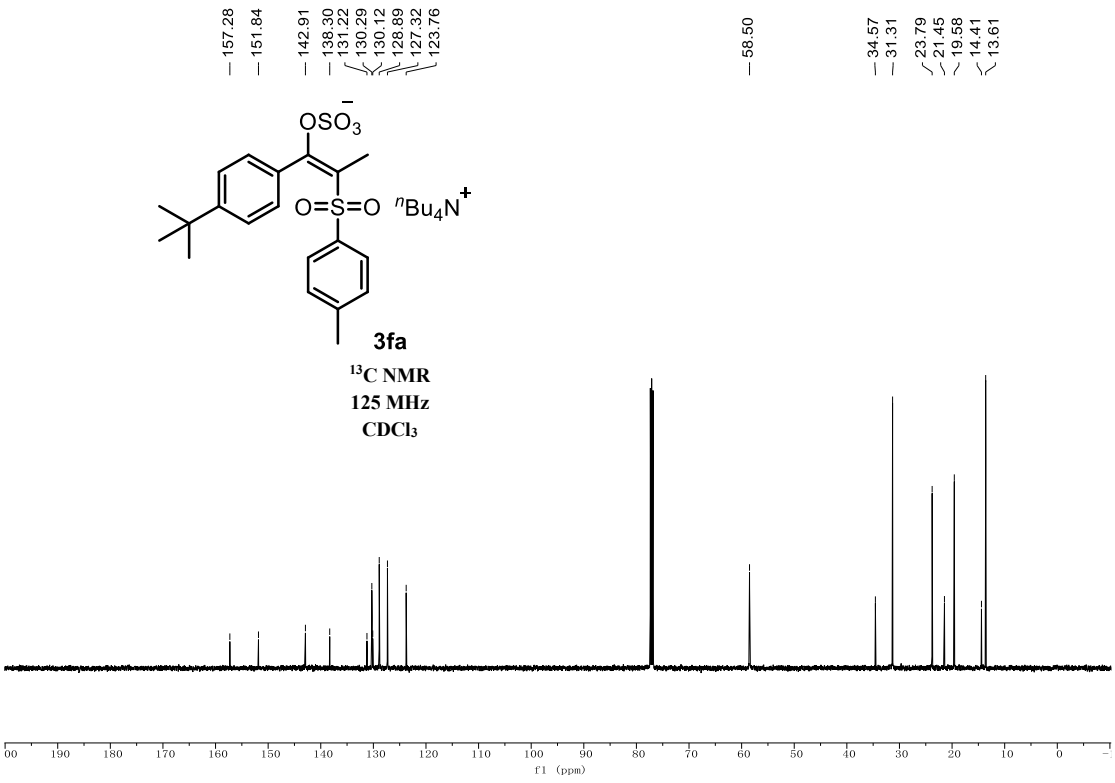


3fa

^{1}H NMR

500 MHz

$CDCl_3$

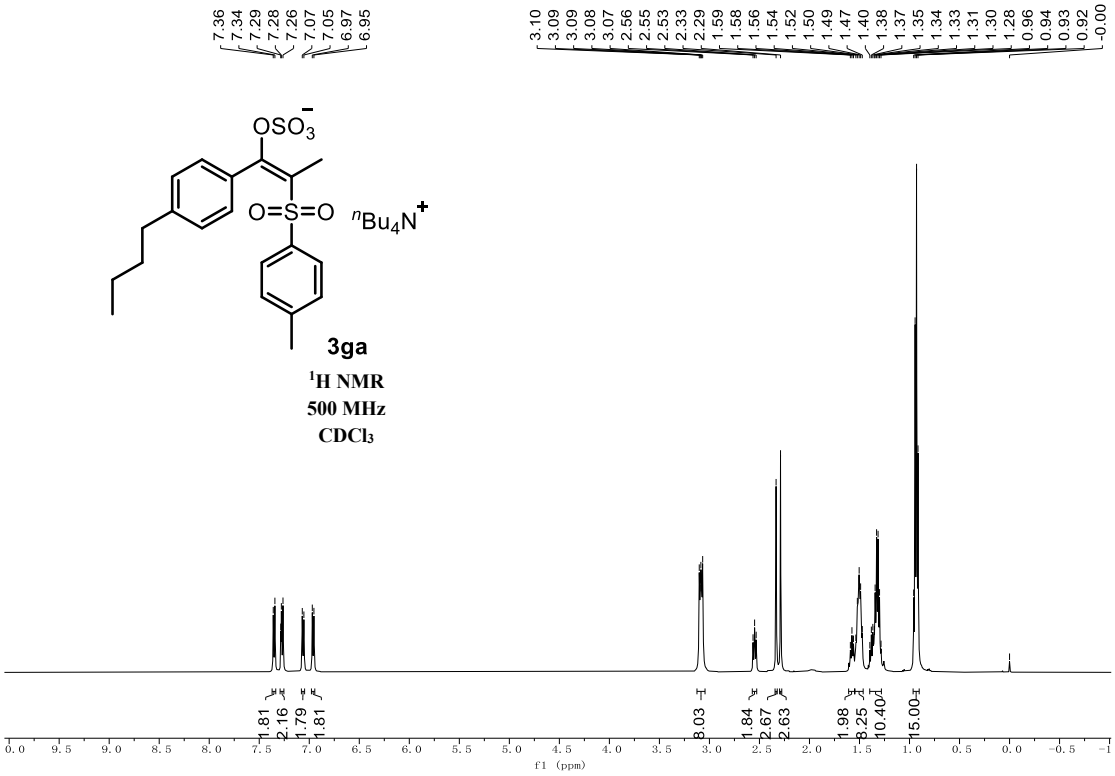


3fa

^{13}C NMR

125 MHz

$CDCl_3$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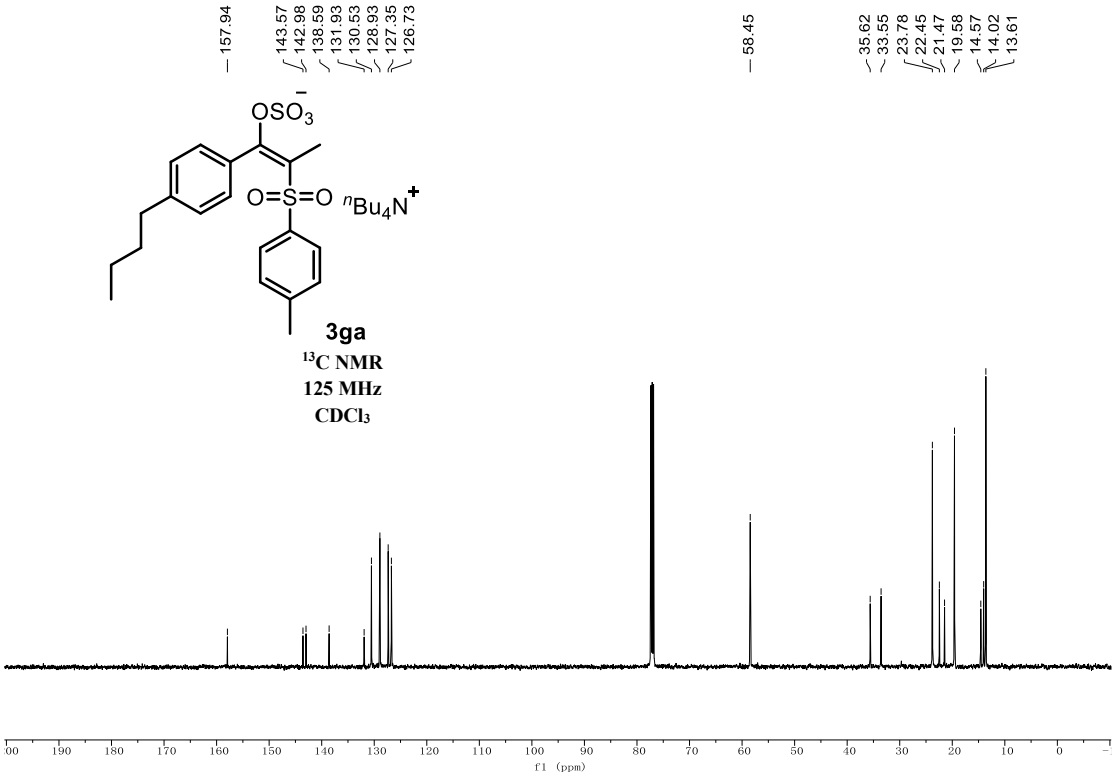
7.05
6.97
6.95
3.10
3.09
3.09
3.08
3.07
2.56
2.55
2.53
2.33
2.29
1.59
1.58
1.56
1.54
1.52
1.50
1.49
1.47
1.40
1.38
1.37
1.35
1.34
1.33
1.31
1.30
1.28
0.96
0.94
0.93
0.92
-0.00
OSO3−
O=S=O
nBu4N+
3ga
1H NMR
500 MHz
CDCl3
1.81
2.16
1.79
1.81
8.03
1.84
2.67
2.63
1.98
8.25
10.40
15.00
f1 (ppm)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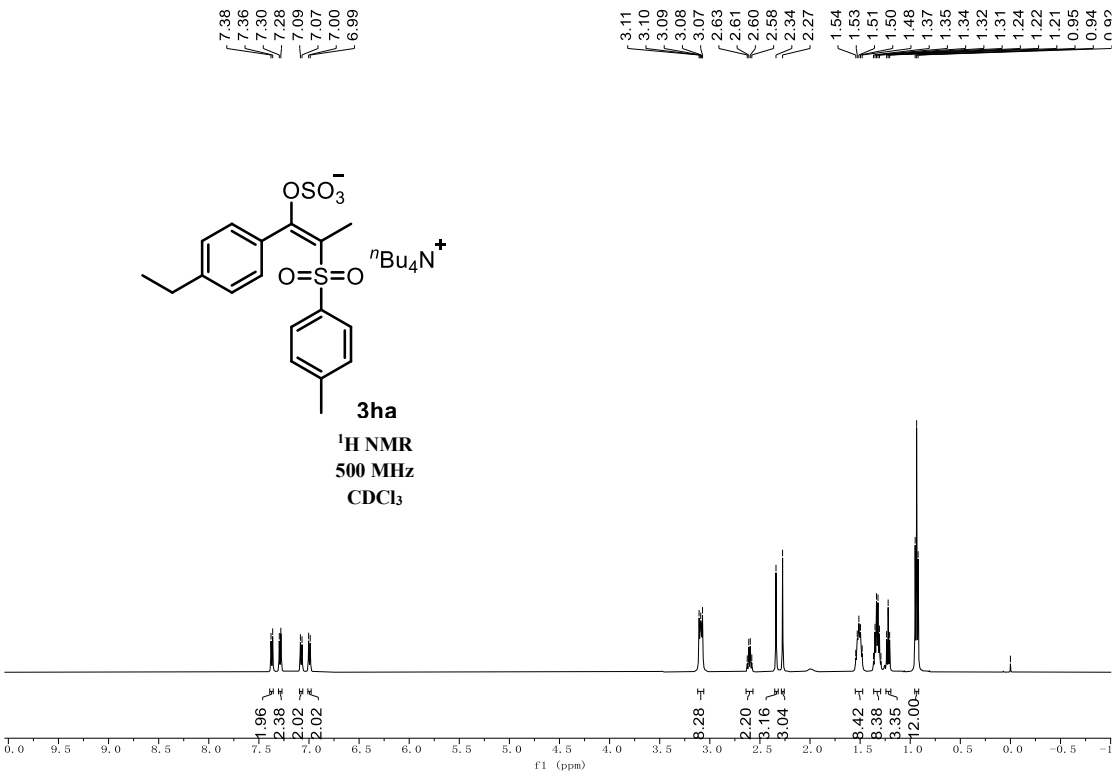
131.93
130.53
128.93
127.35
126.73
58.45
35.62
33.55
23.78
22.45
21.47
19.58
14.57
14.02
13.61
OSO3−
O=S=O
nBu4N+
3ga
13C NMR
125 MHz
CDCl3
f1 (ppm)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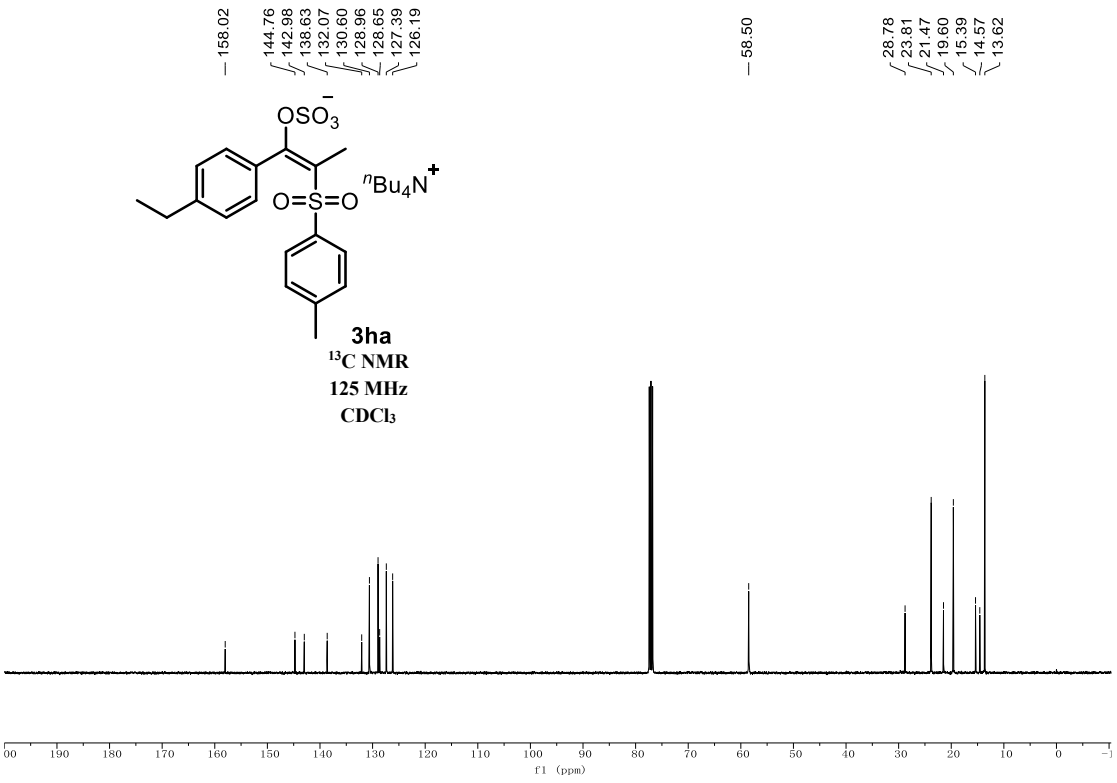
3.11
3.10
3.09
3.08
3.07
2.63
2.61
2.60
2.58
2.34
2.27
1.54
1.53
1.51
1.50
1.48
1.37
1.35
1.34
1.32
1.31
1.24
1.22
1.21
0.95
0.94
OSO3−
nBu4N+
O=S=O
3ha
1H NMR
500 MHz
CDCl3
1.96
2.38
2.02
2.02
8.28
2.20
3.16
3.04
8.42
8.38
3.35
12.00
f1 (ppm)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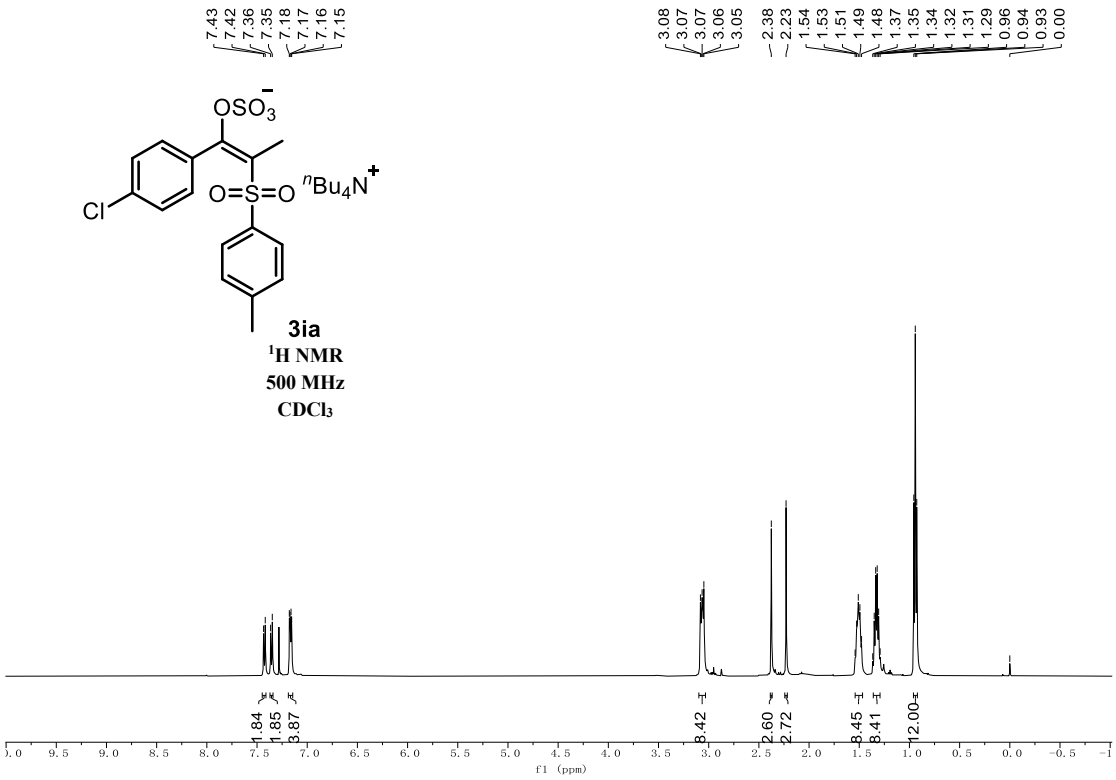
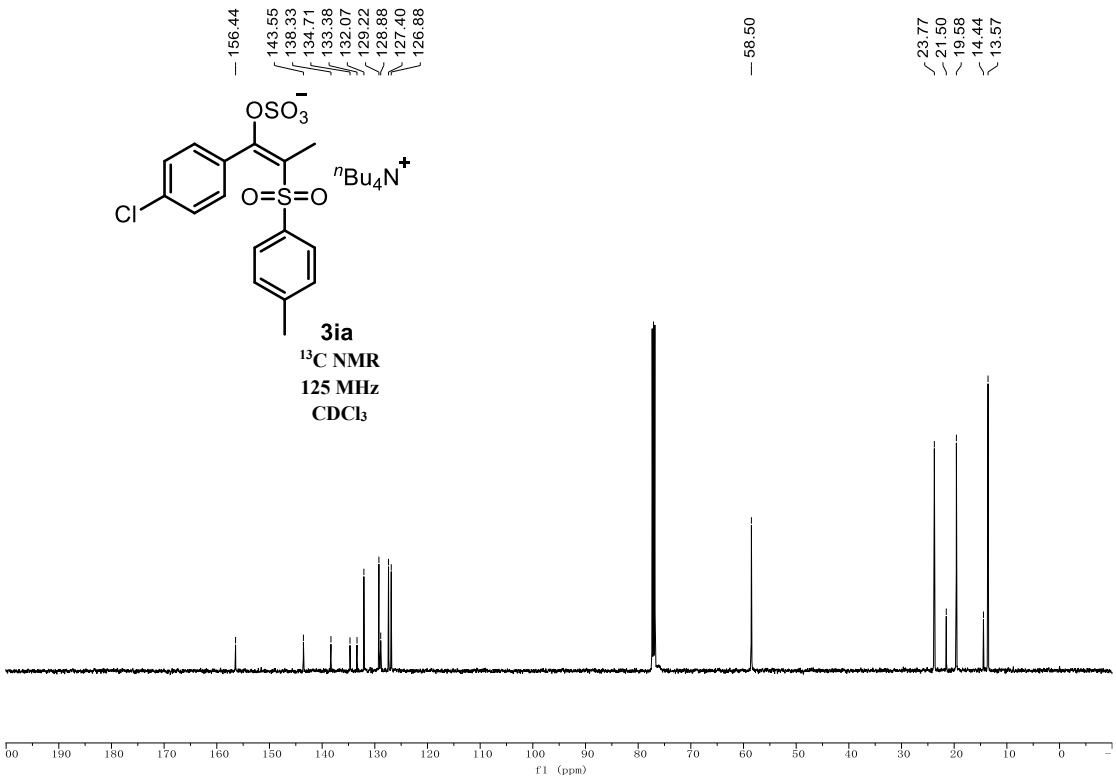
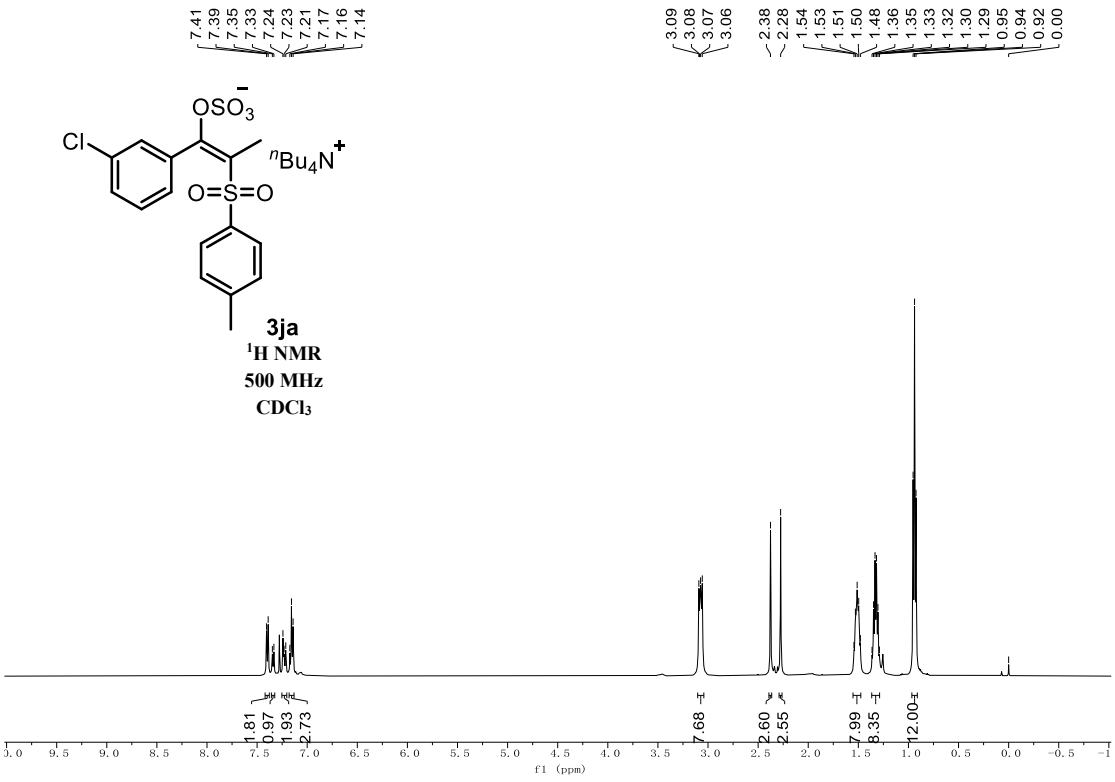
132.07
130.60
128.96
128.65
127.39
126.19
58.50
28.78
23.81
21.47
19.60
15.39
14.57
13.62
OSO3−
nBu4N+
O=S=O
3ha
13C NMR
125 MHz
CDCl3
f1 (ppm)



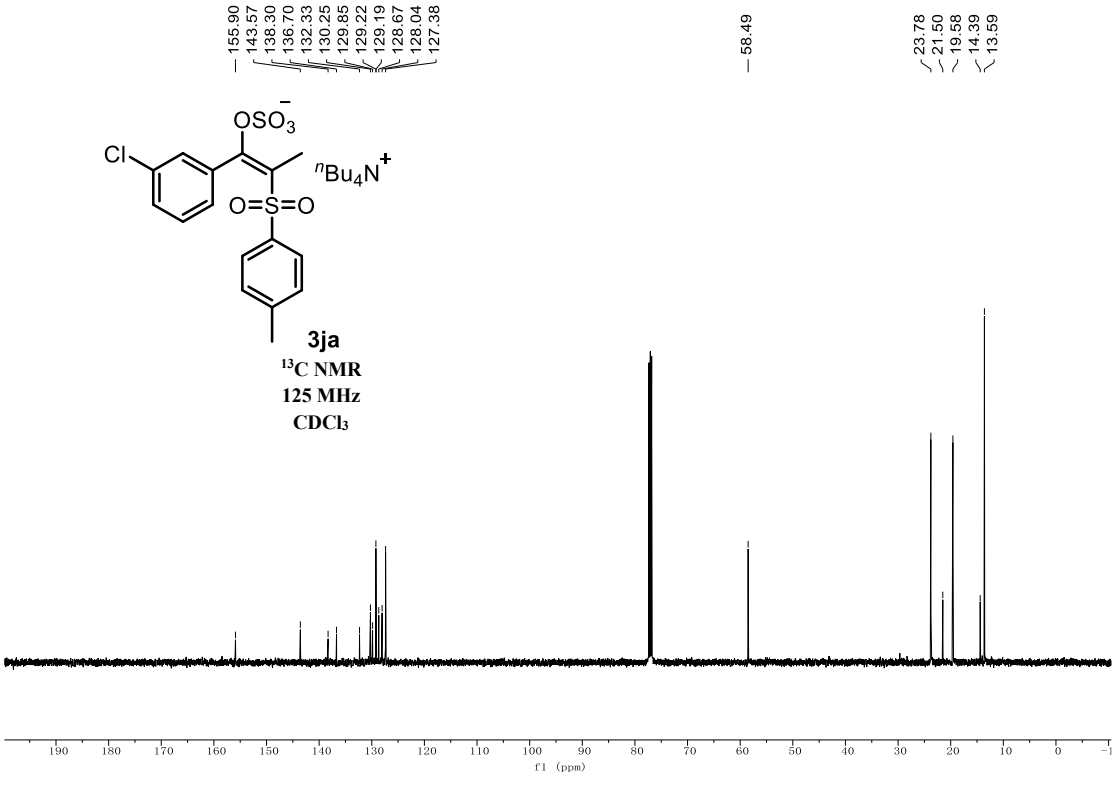
3ia
^{1}H NMR
500 MHz
$CDCl_3$



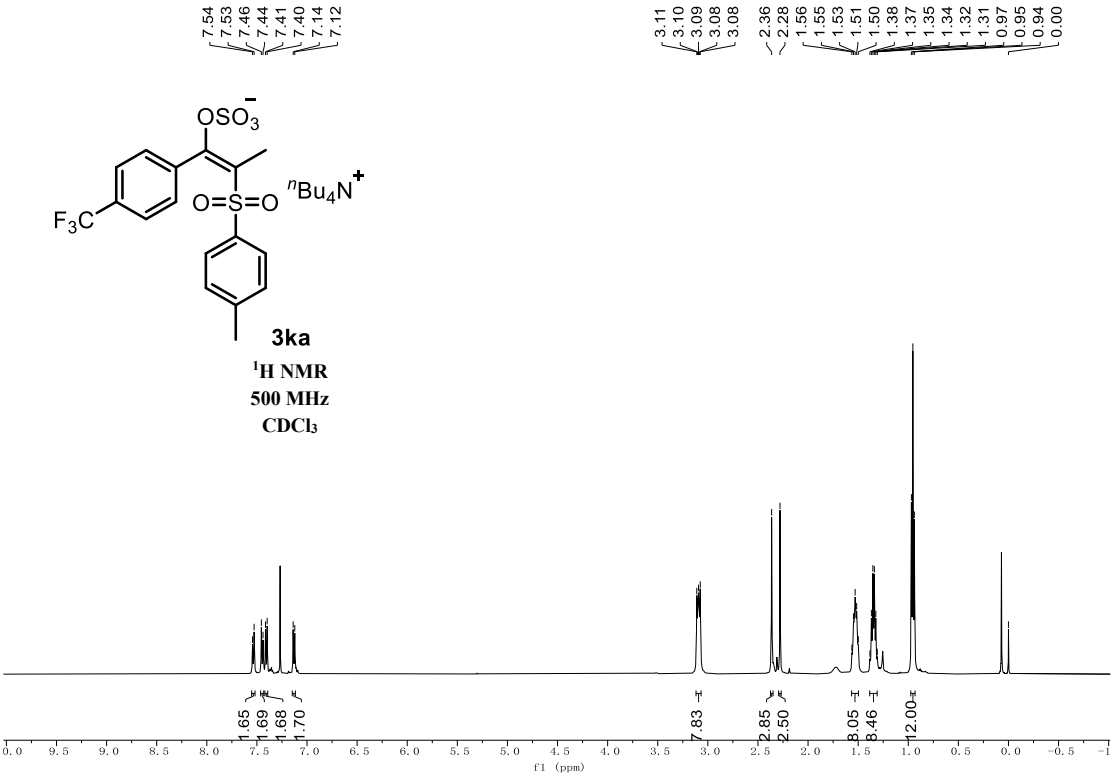
3ia
^{13}C NMR
125 MHz
$CDCl_3$



3ja
^{1}H NMR
500 MHz
$CDCl_3$



3ja
^{13}C NMR
125 MHz
$CDCl_3$


7.54
7.53
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7.41
7.40
7.14
7.12
3.11
3.10
3.09
3.08
3.08
2.36
2.28
1.56
1.55
1.53
1.51
1.50
1.38
1.37
1.35
1.34
1.32
1.31
0.97
0.95
0.94
0.00
OSO3−
nBu4N+
O=S=O
F3C
3ka
1H NMR
500 MHz
CDCl3
1.65
1.69
1.68
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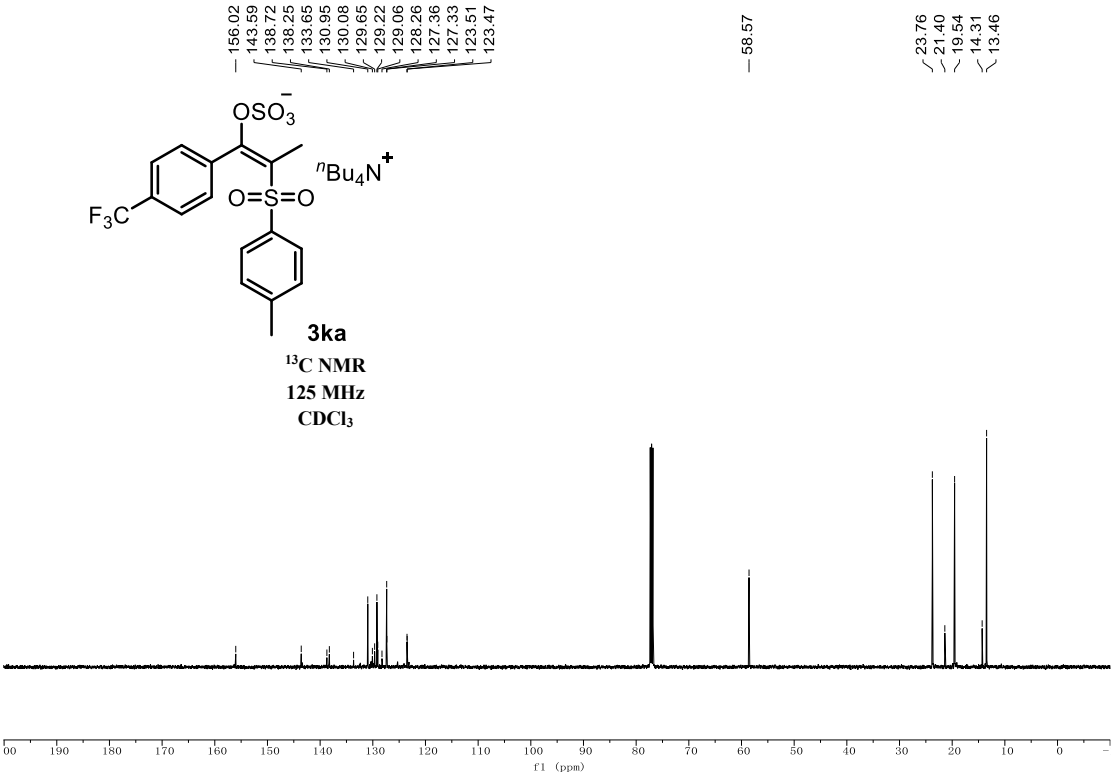
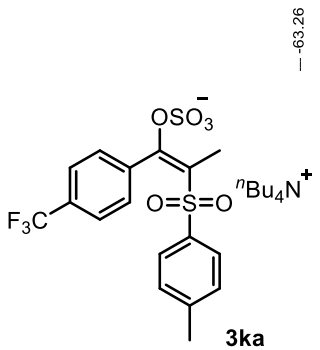
7.83
2.85
2.50
8.05
8.46
12.00
f1 (ppm)


156.02
143.59
138.72
138.25
133.65
130.95
130.08
129.65
129.22
129.06
128.26
127.36
127.33
123.51
123.47
58.57
23.76
21.40
19.54
14.31
13.46
OSO3−
nBu4N+
O=S=O
F3C
3ka
13C NMR
125 MHz
CDCl3
f1 (ppm)

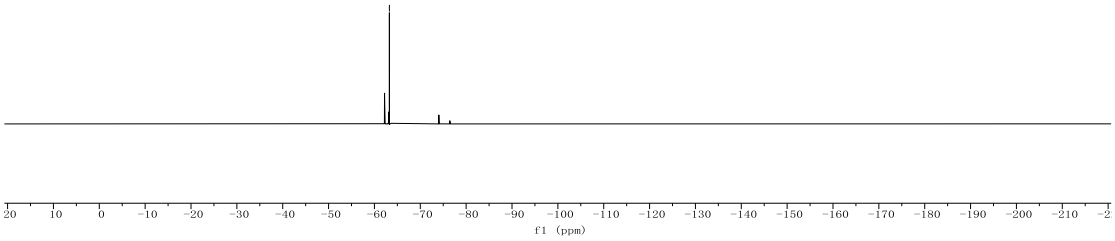


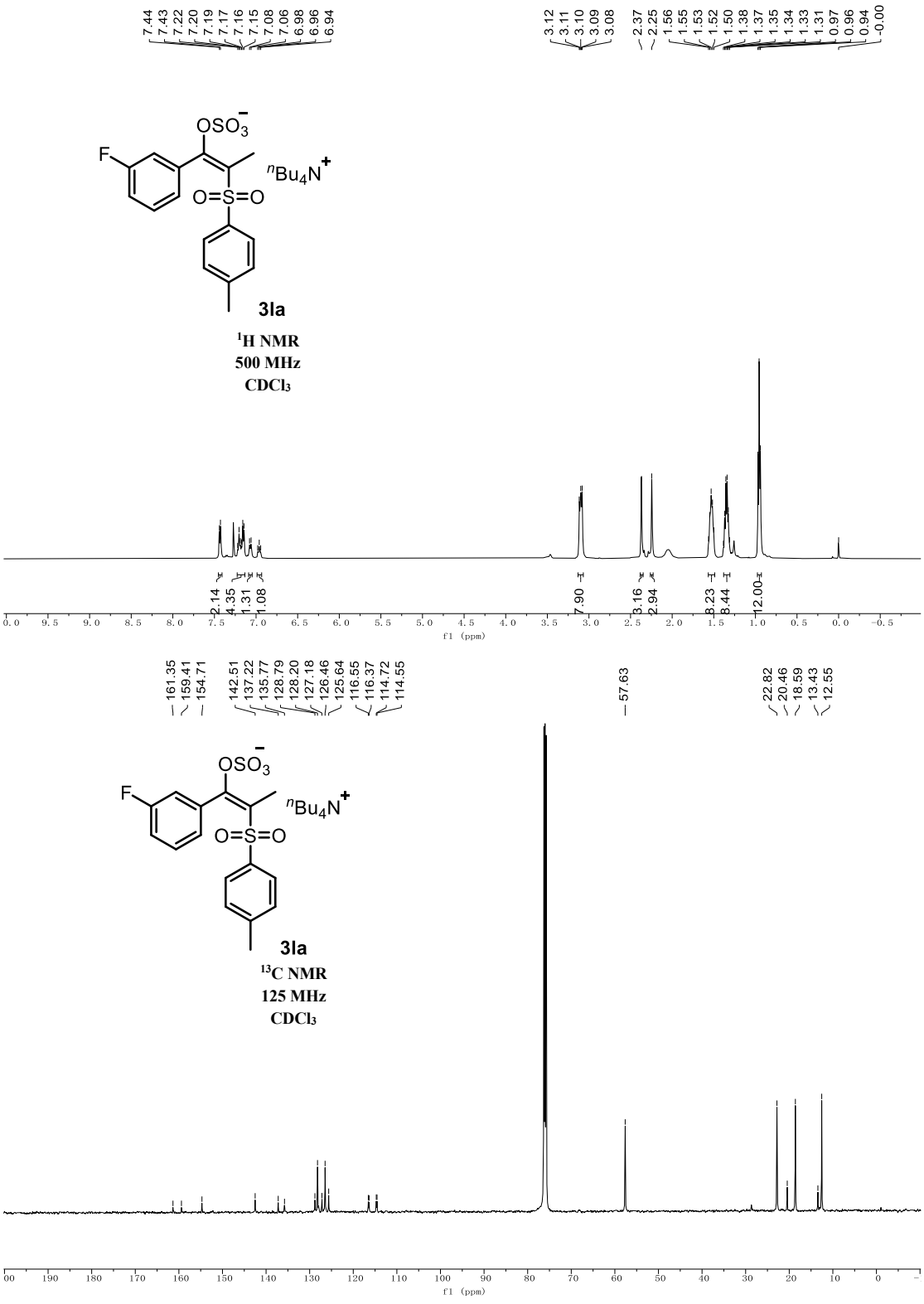
3ka

^{19}F NMR

376 MHz

$CDCl_3$




7.44
7.43
7.22
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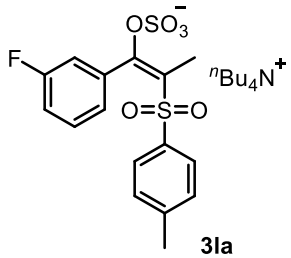
7.19
7.17
7.16
7.15
7.08
7.06
6.98
6.96
6.94
3.12
3.11
3.10
3.09
3.08
2.37
2.25
1.56
1.55
1.53
1.52
1.50
1.38
1.37
1.35
1.34
1.33
1.31
0.97
0.96
0.94
-0.00
OSO3−
F
nBu4N+
O=S=O
3Ia
1H NMR
500 MHz
CDCl3
2.14
4.35
1.31
1.08
7.90
3.16
2.94
8.23
8.44
12.00
f1 (ppm)
161.35
159.41
154.71
142.51
137.22
135.77
128.79
128.20
127.18
126.46
125.64
116.55
116.37
114.72
114.55
57.63
22.82
20.46
18.59
13.43
12.55
OSO3−
F
nBu4N+
O=S=O
3Ia
13C NMR
125 MHz
CDCl3
f1 (ppm)

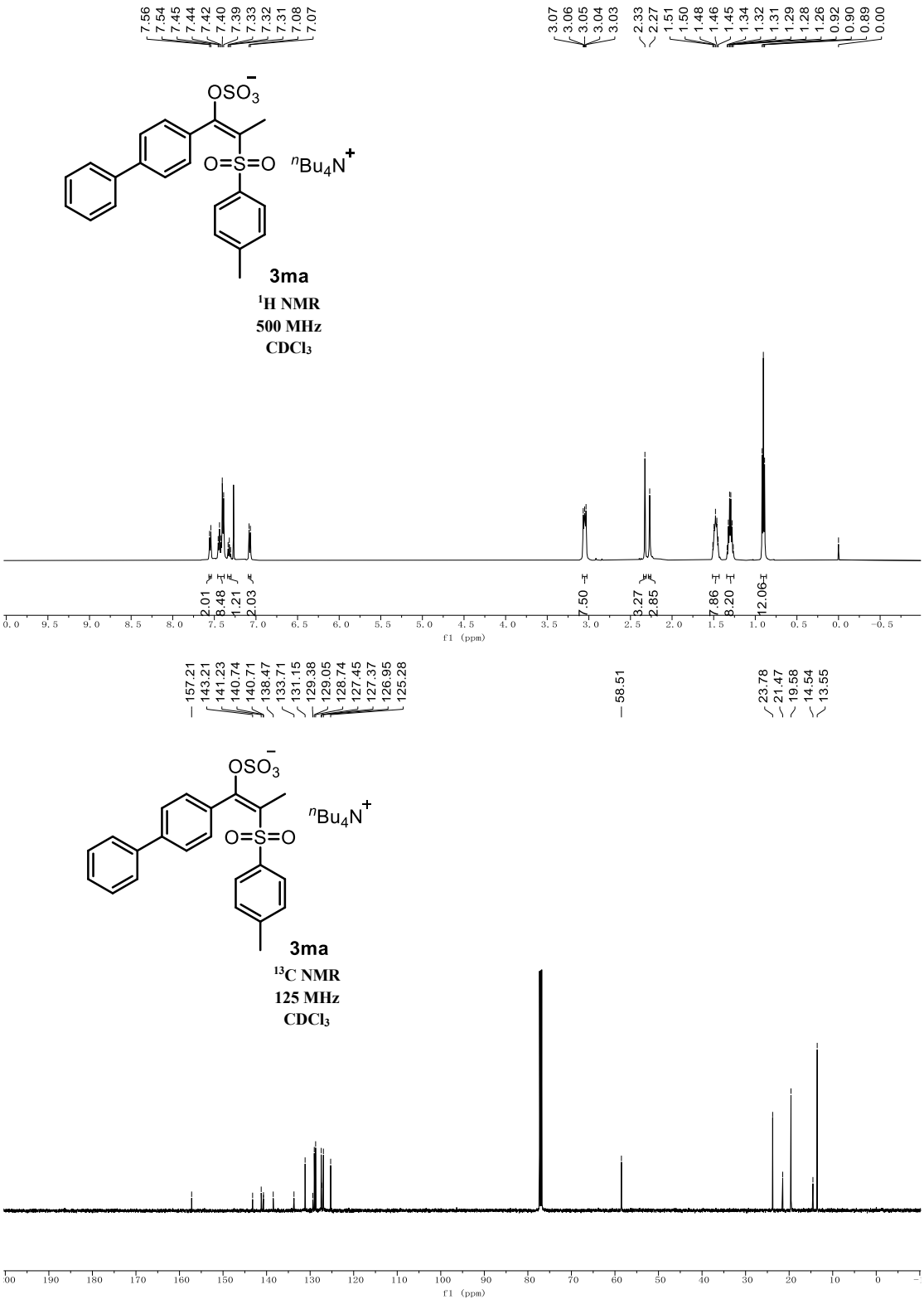


3la

^{19}F NMR

376 MHz

$CDCl_3$


7.56
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3.07
3.06
3.05
3.04
3.03
2.33
2.27
1.51
1.50
1.48
1.46
1.45
1.34
1.32
1.31
1.29
1.28
1.26
0.92
0.90
0.89
0.00
OSO3−
O=S=O
nBu4N+
3ma
1H NMR
500 MHz
CDCl3
2.01
3.48
1.21
2.03
7.50
3.27
2.85
7.86
8.20
12.06
f1 (ppm)
157.21
143.21
141.23
140.74
140.71
138.47
133.71
131.15
129.38
129.05
128.74
127.45
127.37
126.95
125.28
58.51
23.78
21.47
19.58
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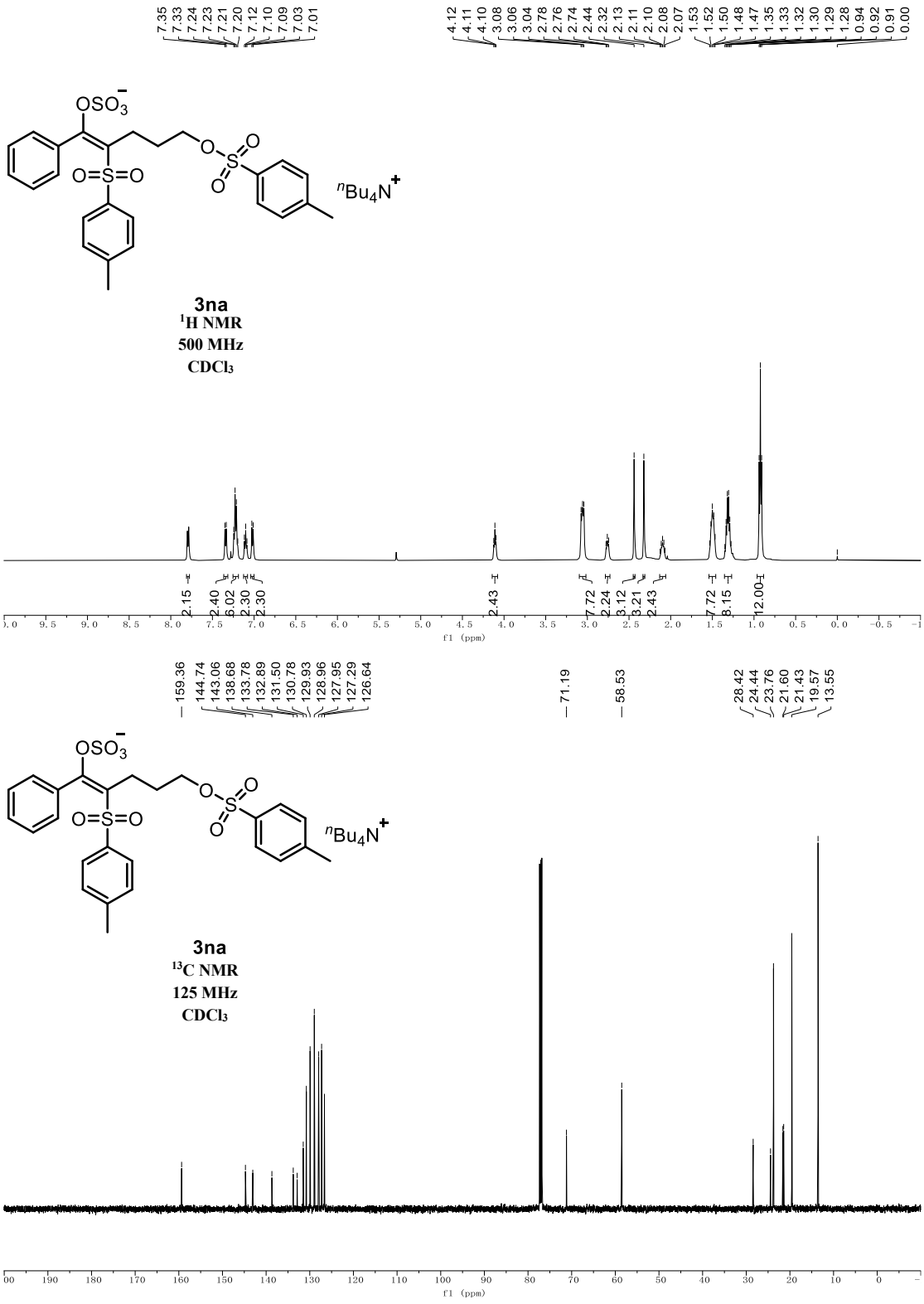
13.55
OSO3−
nBu4N+
O=S=O
3ma
13C NMR
125 MHz
CDCl3
f1 (ppm)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0.91
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O=S=O
O
S
O
O
nBu4N+
3na
1H NMR
500 MHz
CDCl3
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2.30
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3.12
3.21
2.43
7.72
8.15
12.00
f1 (ppm)
159.36
144.74
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131.50
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OSO3−
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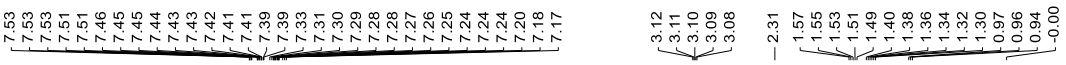
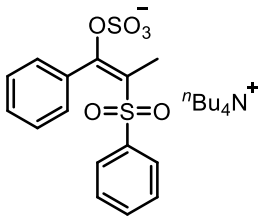
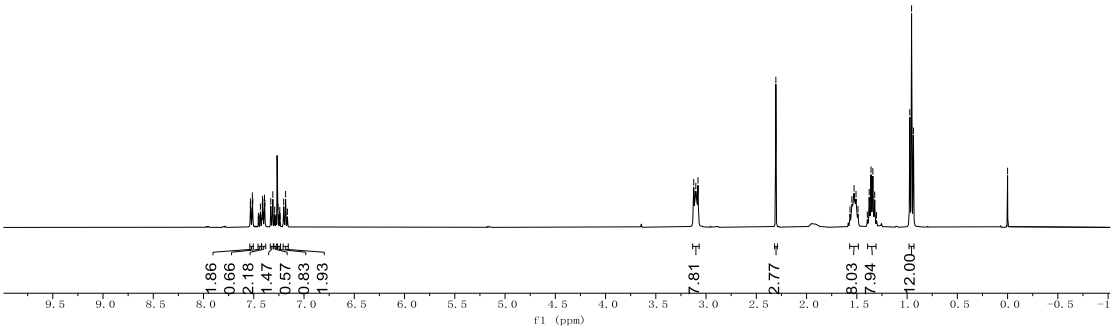
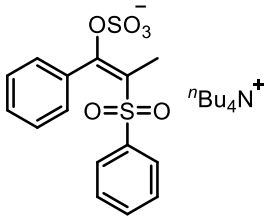
nBu4N+
3na
13C NMR
125 MHz
CDCl3
f1 (ppm)



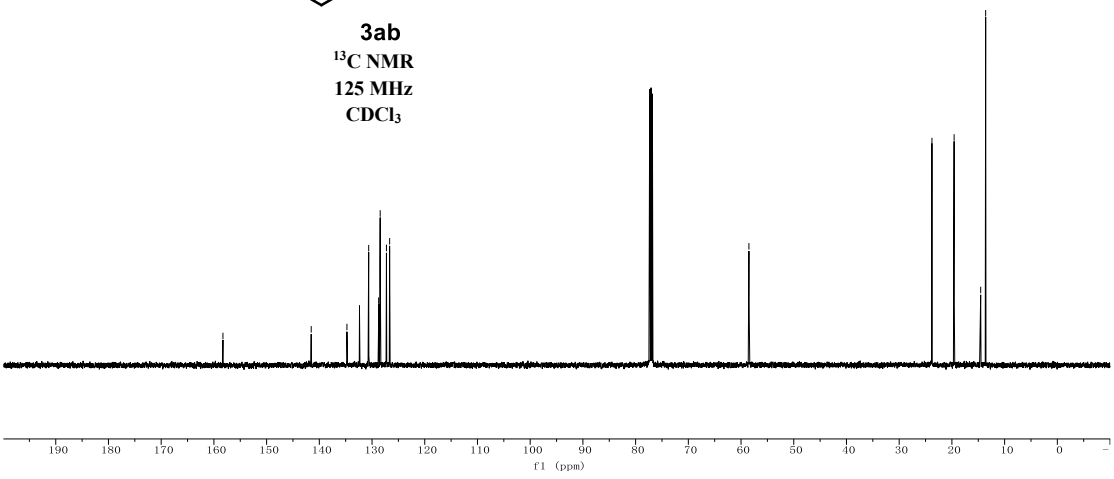


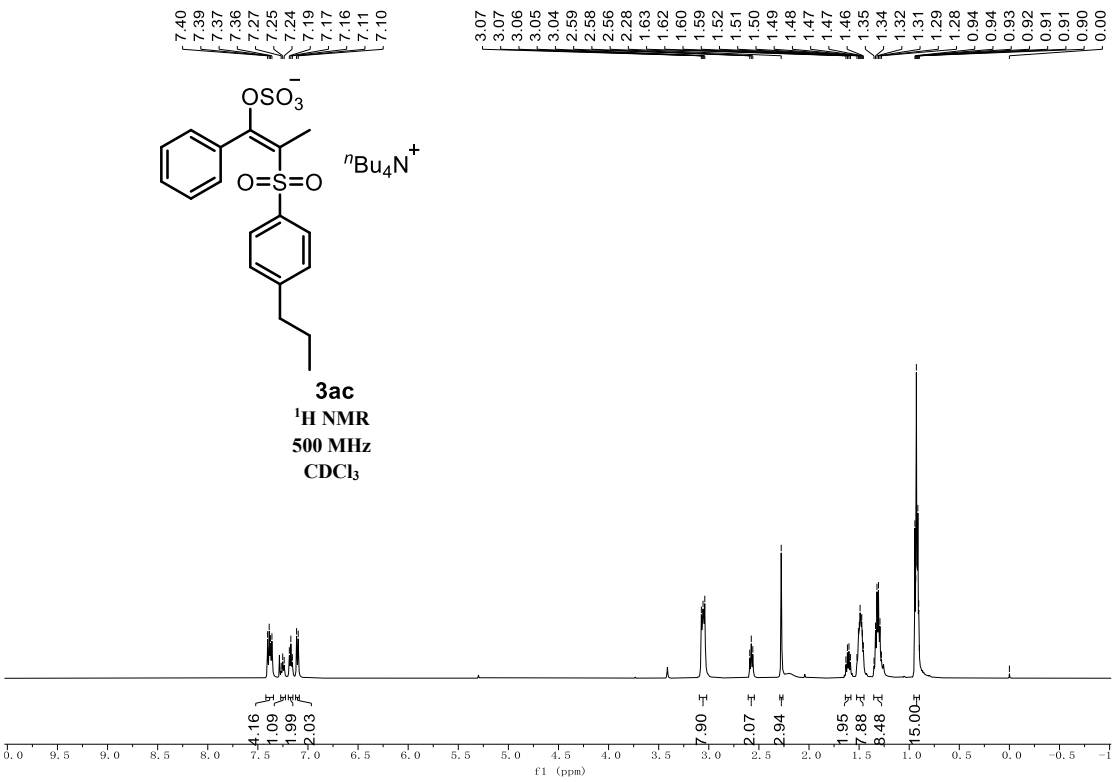
3ab
^{1}H NMR
500 MHz
$CDCl_3$



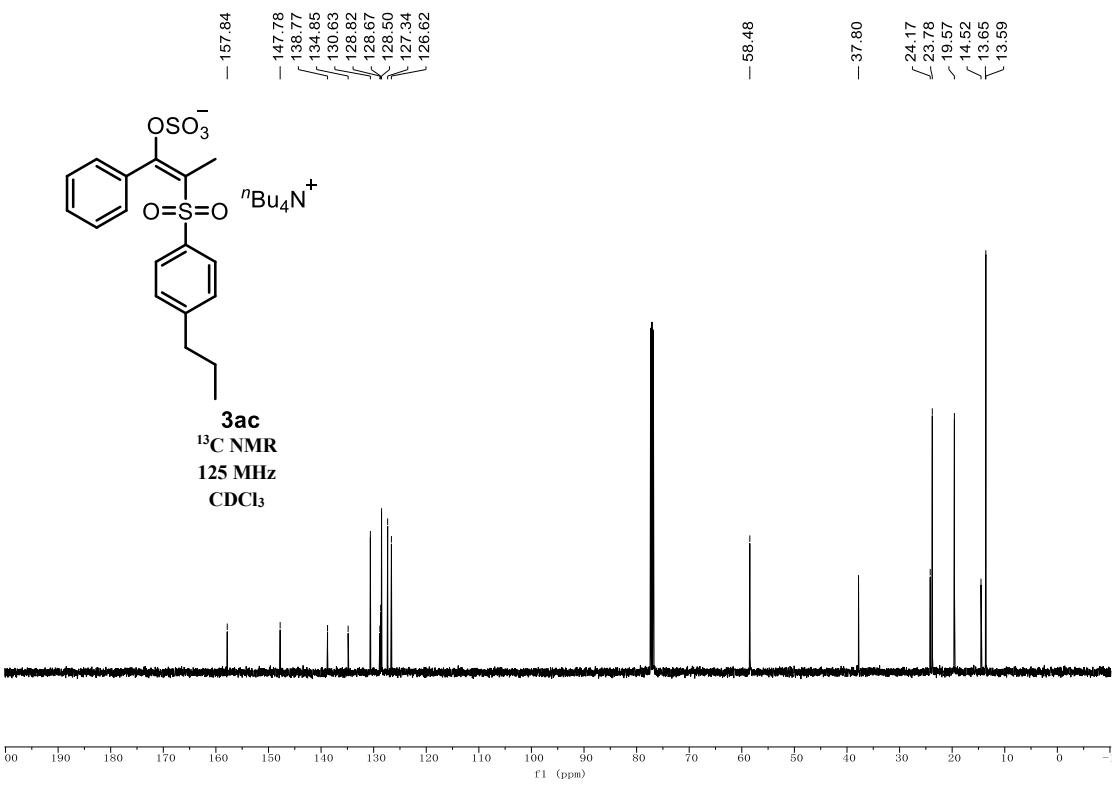


3ab
^{13}C NMR
125 MHz
$CDCl_3$

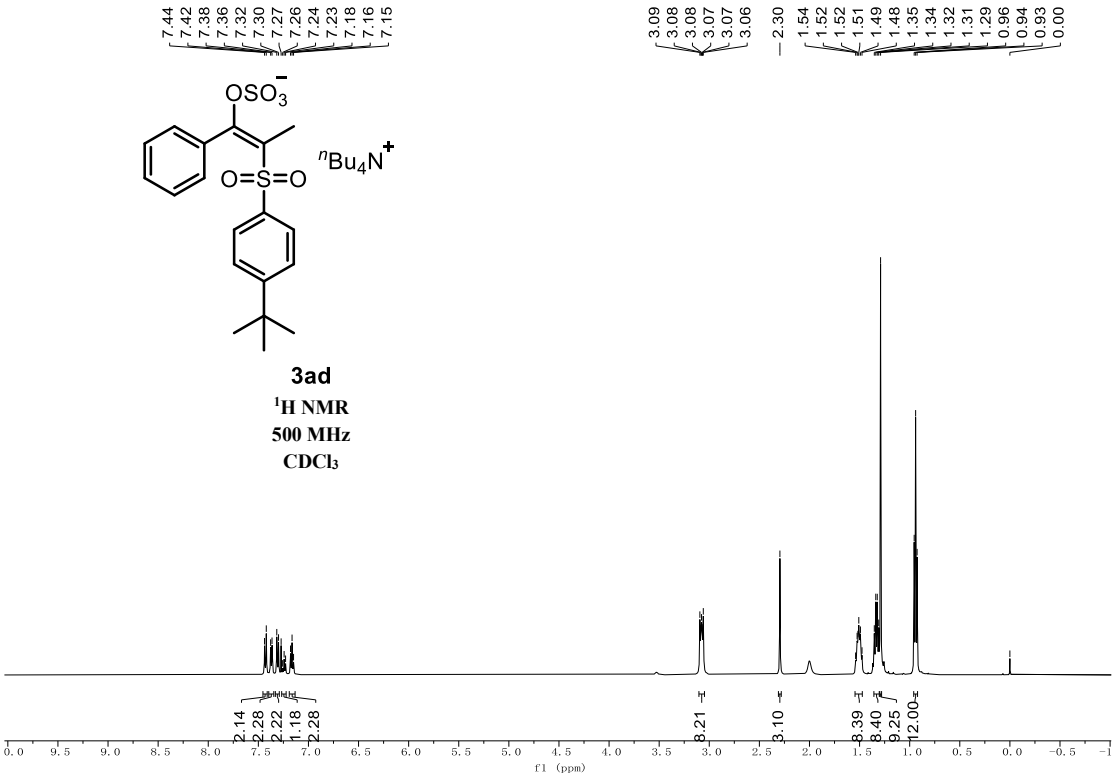




3ac
^{1}H NMR
500 MHz
$CDCl_3$



3ac
^{13}C NMR
125 MHz
$CDCl_3$

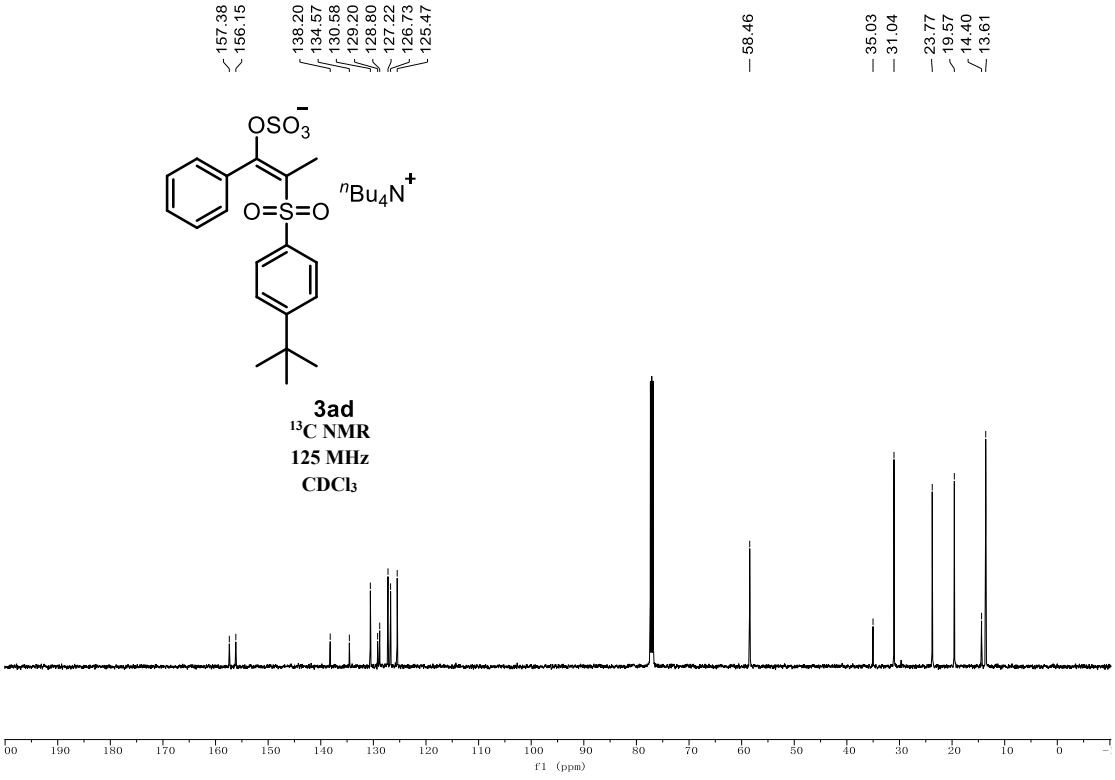


3ad

^{1}H NMR

500 MHz

$CDCl_3$

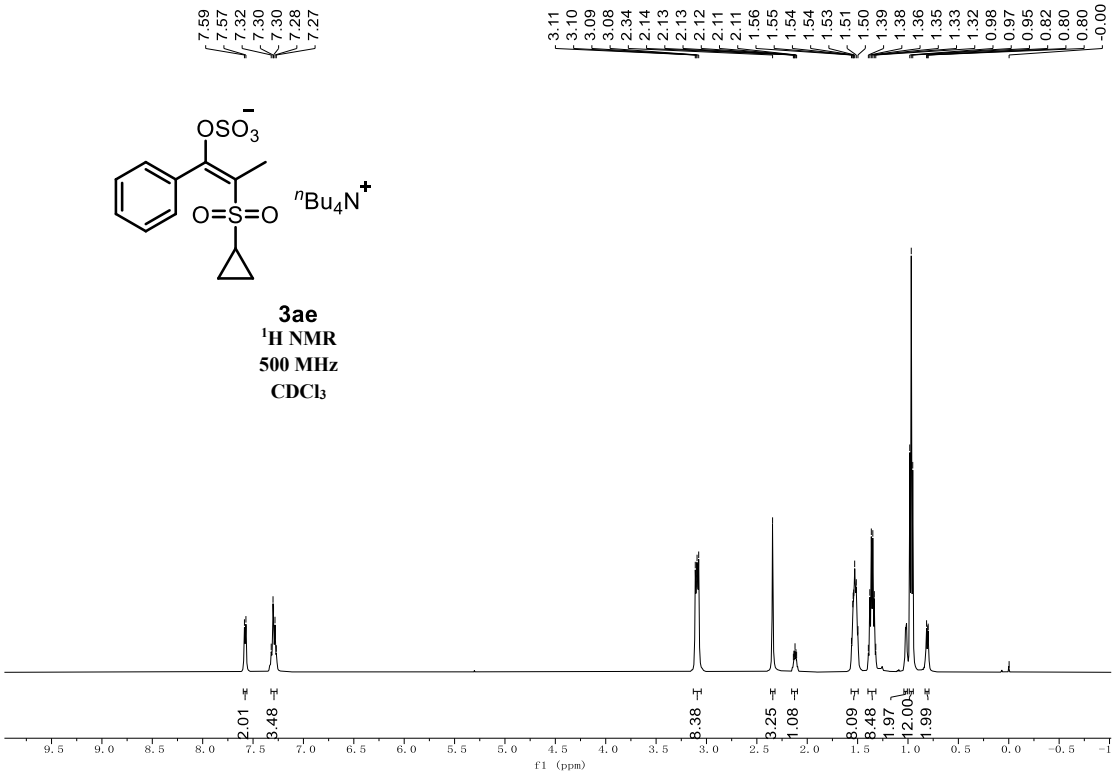


3ad

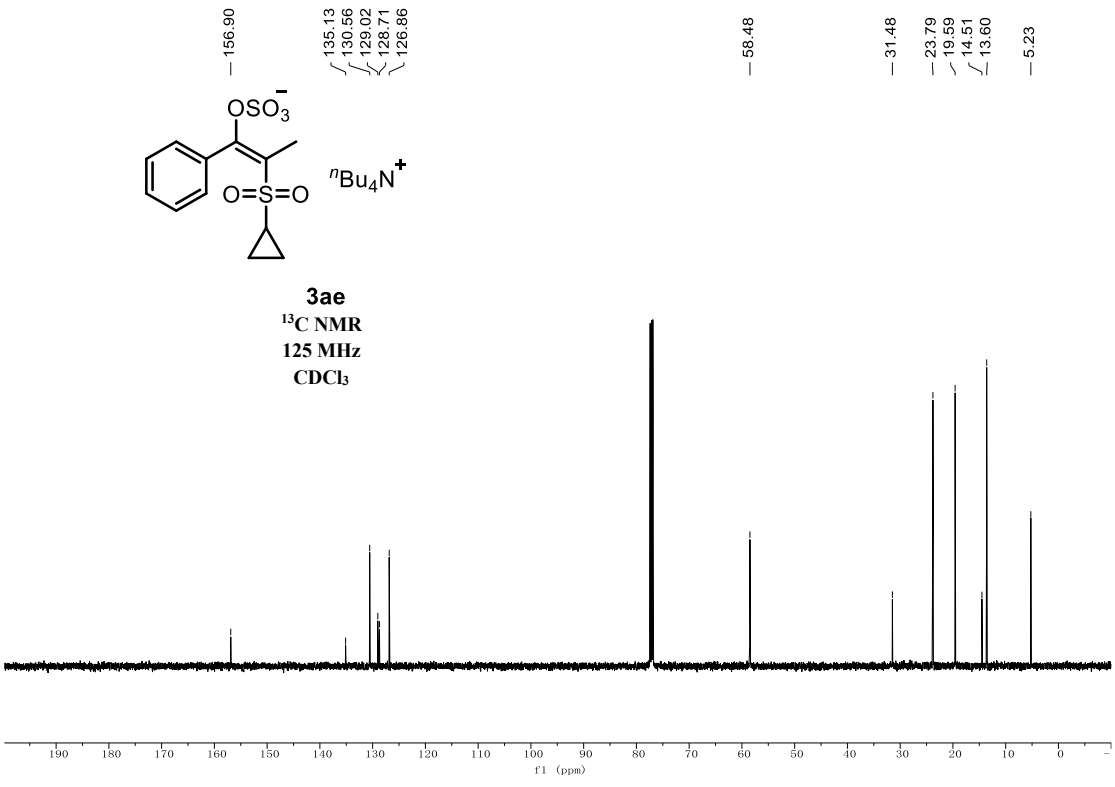
^{13}C NMR

125 MHz

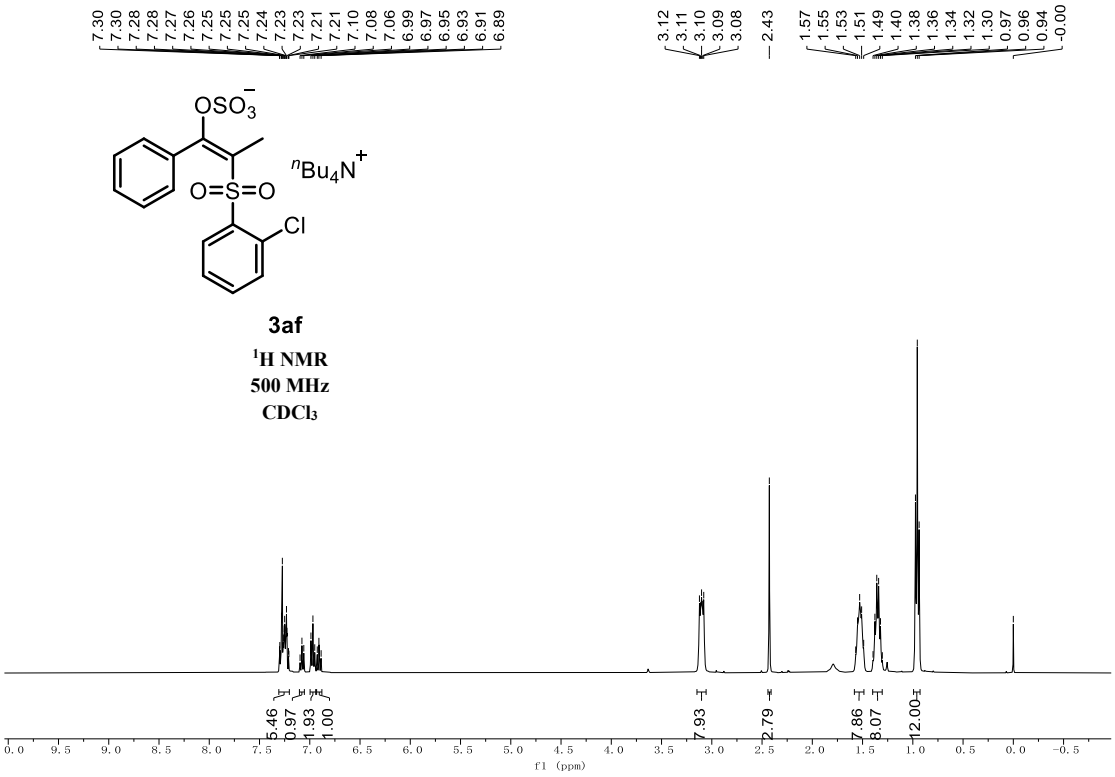
$CDCl_3$



3ae
^{1}H NMR
500 MHz
$CDCl_3$

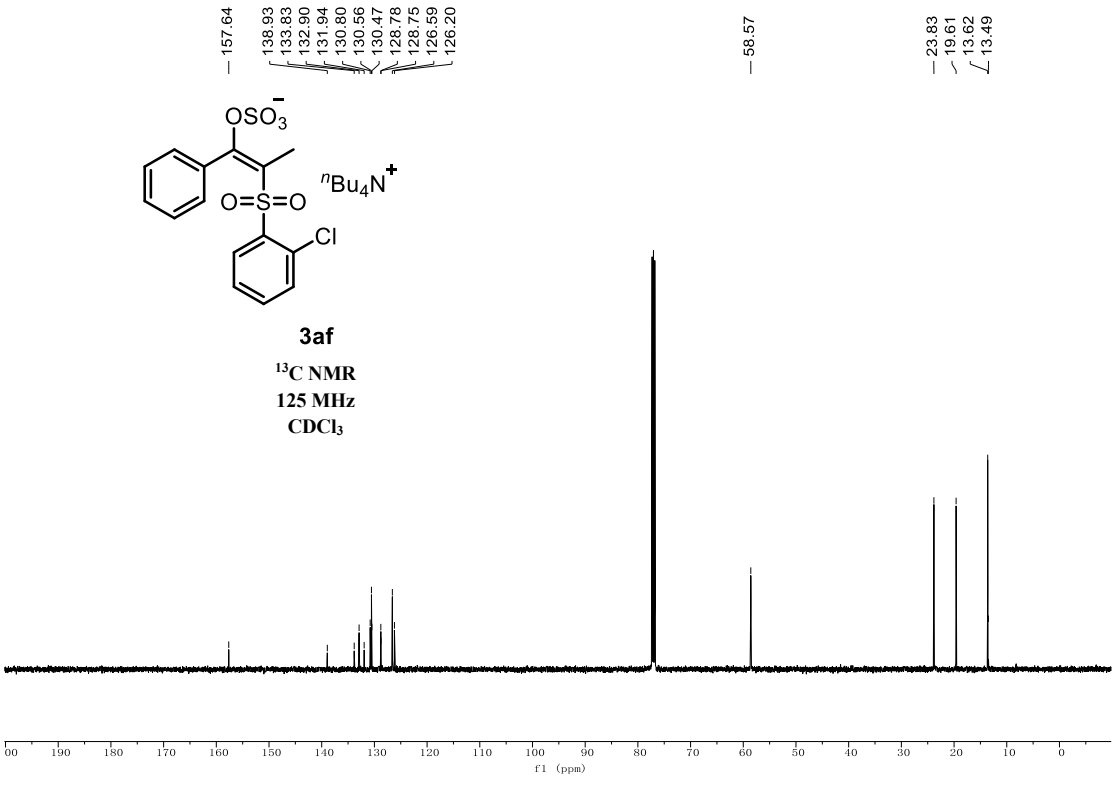


3ae
^{13}C NMR
125 MHz
$CDCl_3$



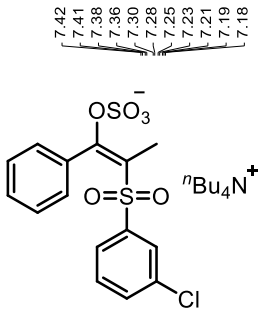
3af

^{1}H NMR
500 MHz
$CDCl_3$



3af

^{13}C NMR
125 MHz
$CDCl_3$

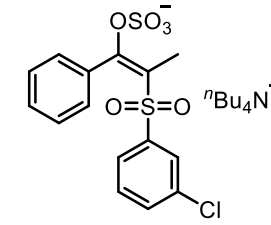


3ag

^{1}H NMR

500 MHz

$CDCl_3$

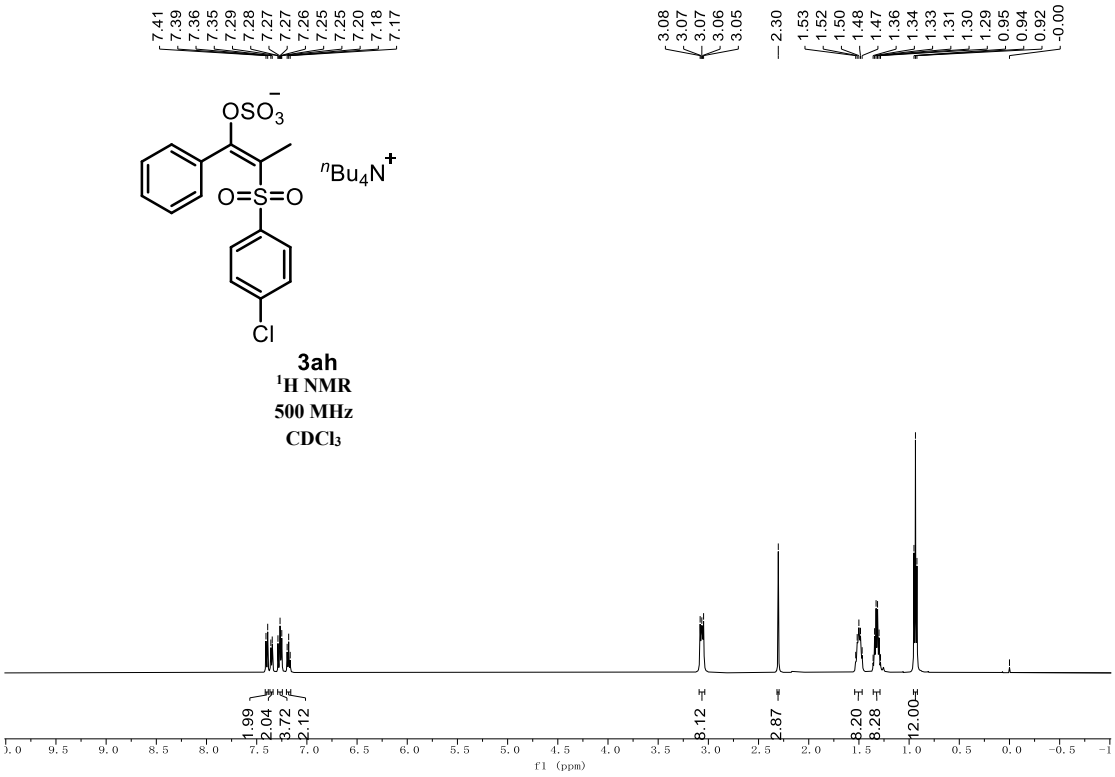


3ag

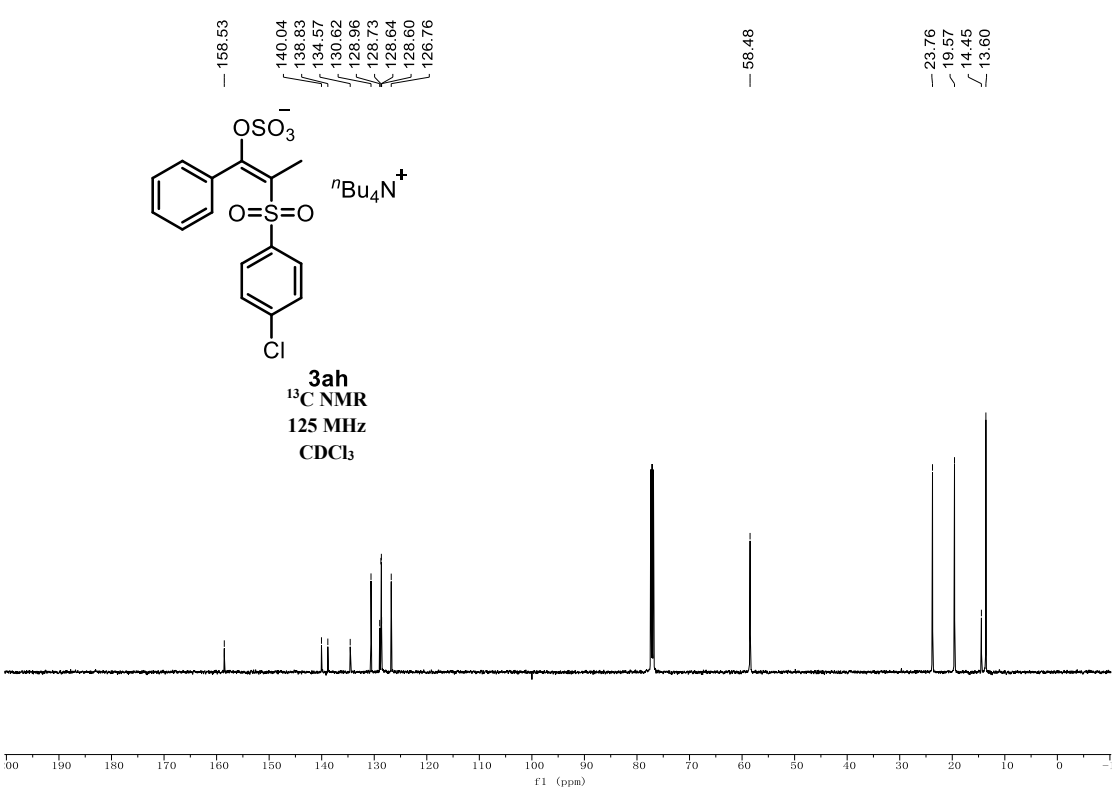
^{13}C NMR

125 MHz

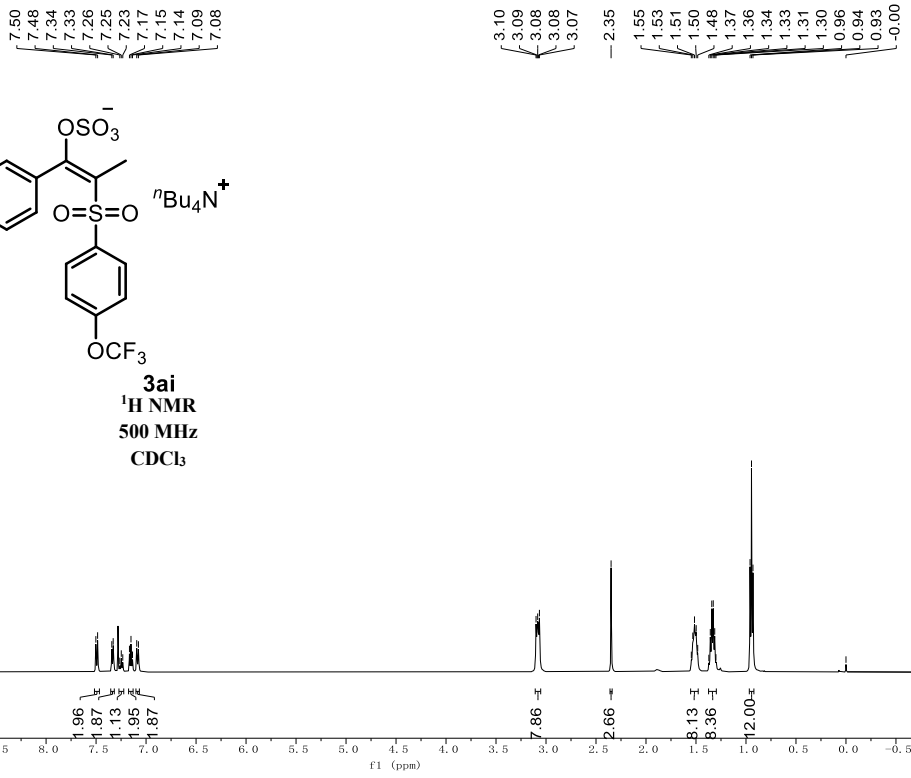
$CDCl_3$



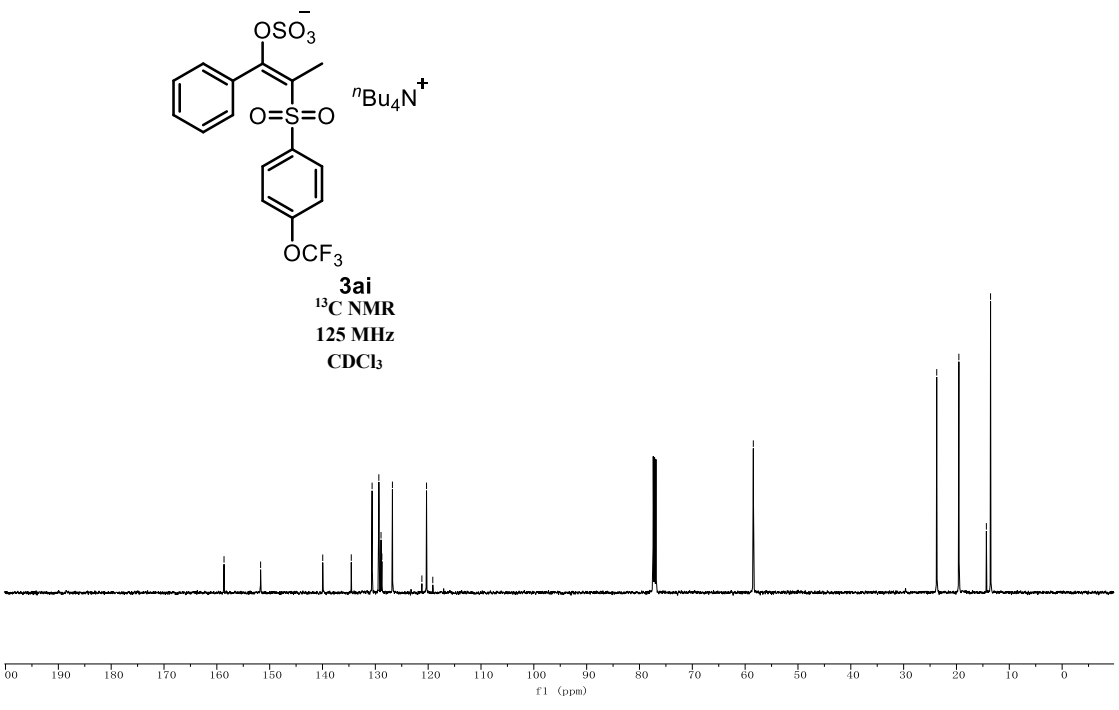
3ah
^{1}H NMR
500 MHz
$CDCl_3$



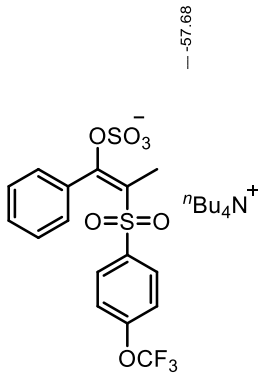
3ah
^{13}C NMR
125 MHz
$CDCl_3$



3ai
^{1}H NMR
500 MHz
$CDCl_3$



3ai
^{13}C NMR
125 MHz
$CDCl_3$

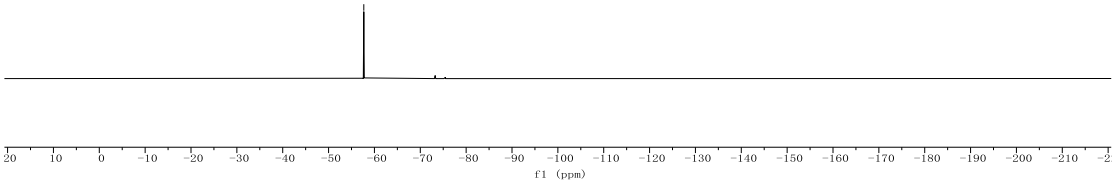


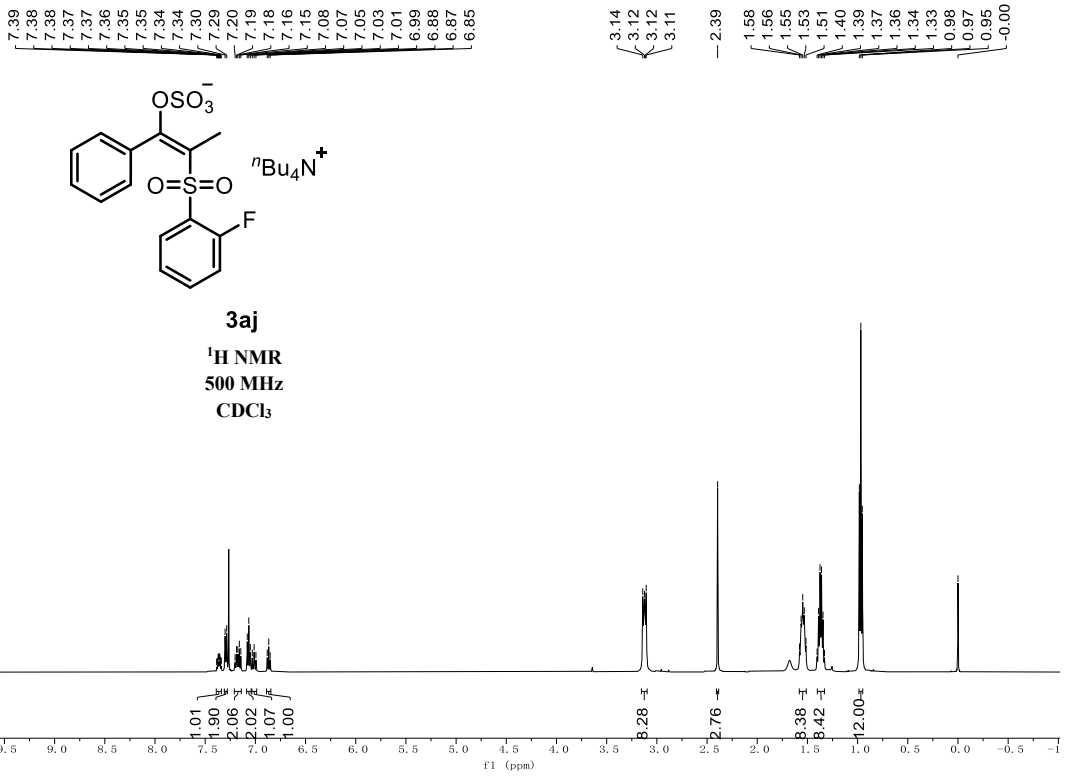
3ai

^{19}F NMR

376 MHz

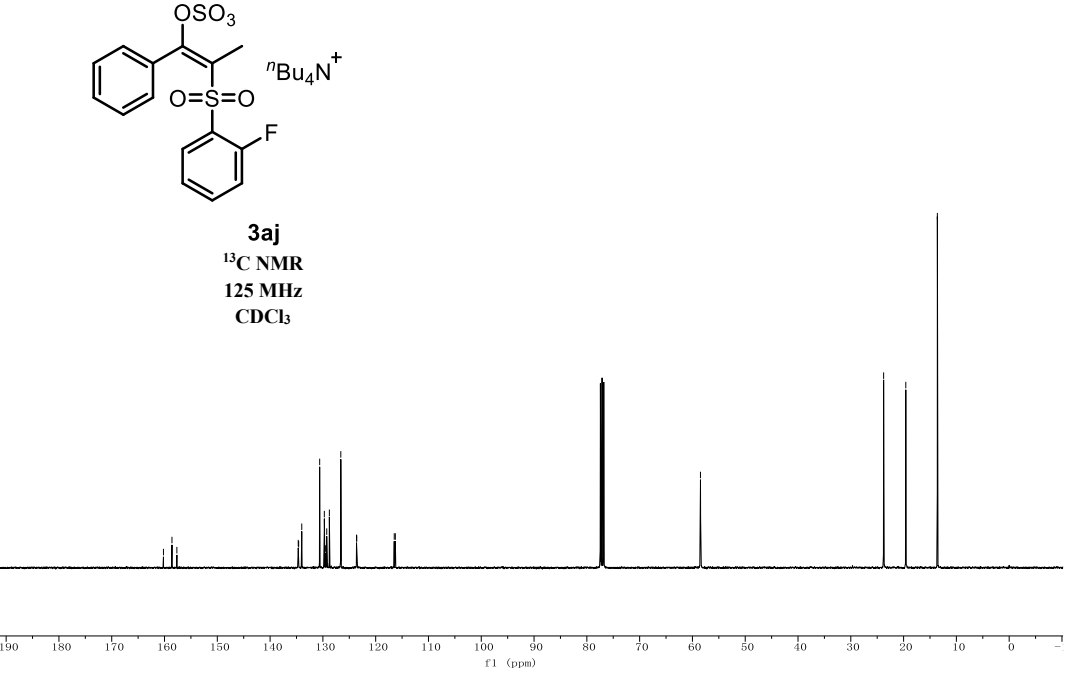
$CDCl_3$





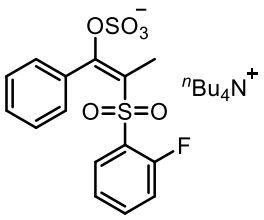
3aj

^{1}H NMR
500 MHz
$CDCl_3$



3aj

^{13}C NMR
125 MHz
$CDCl_3$

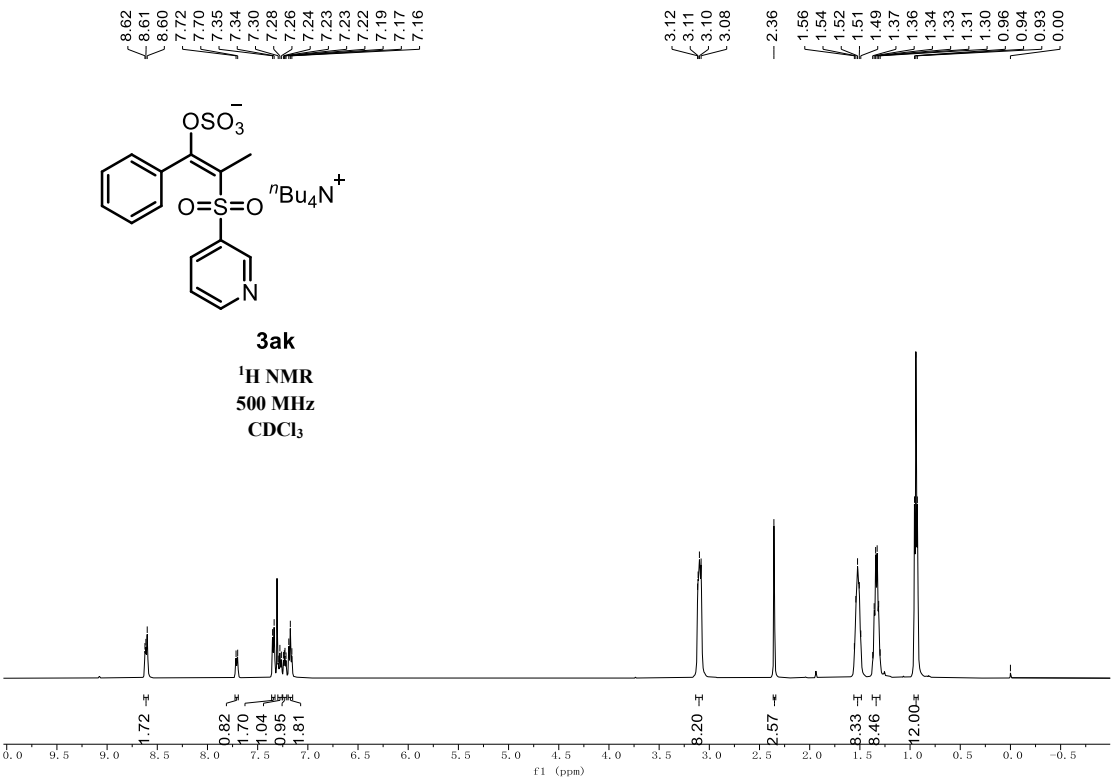


3aj

^{19}F NMR

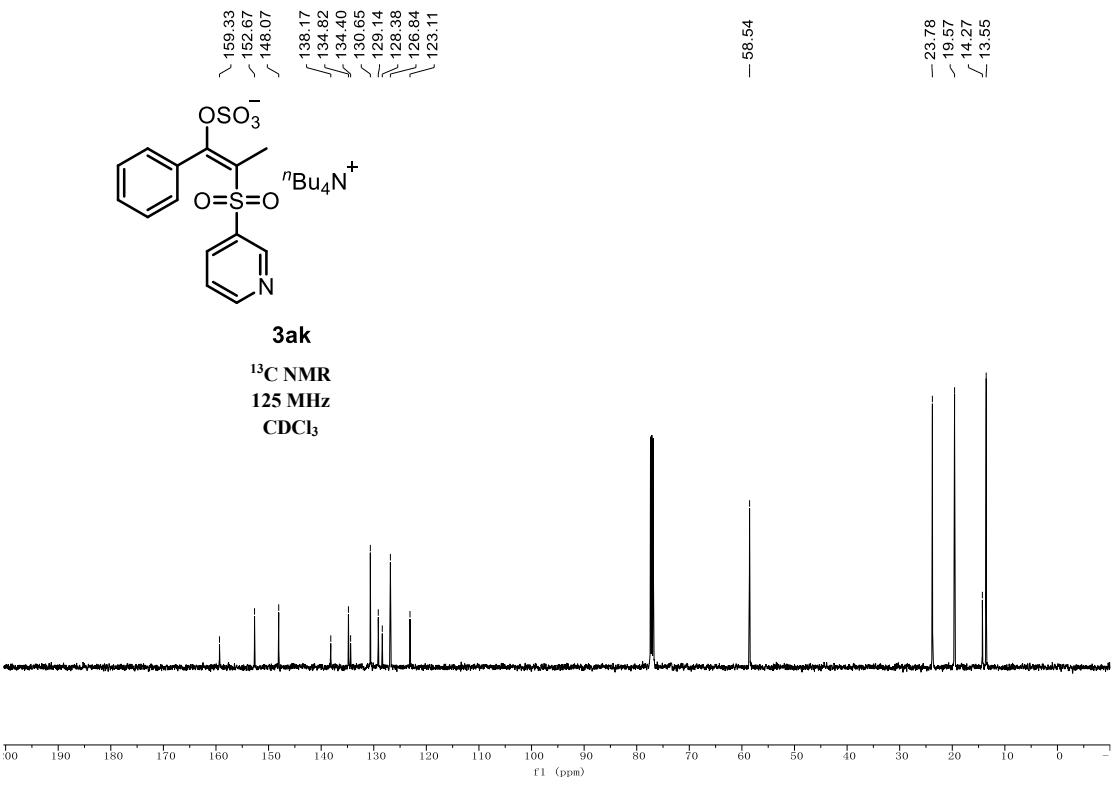
376 MHz

$CDCl_3$



3ak

^{1}H NMR
500 MHz
$CDCl_3$



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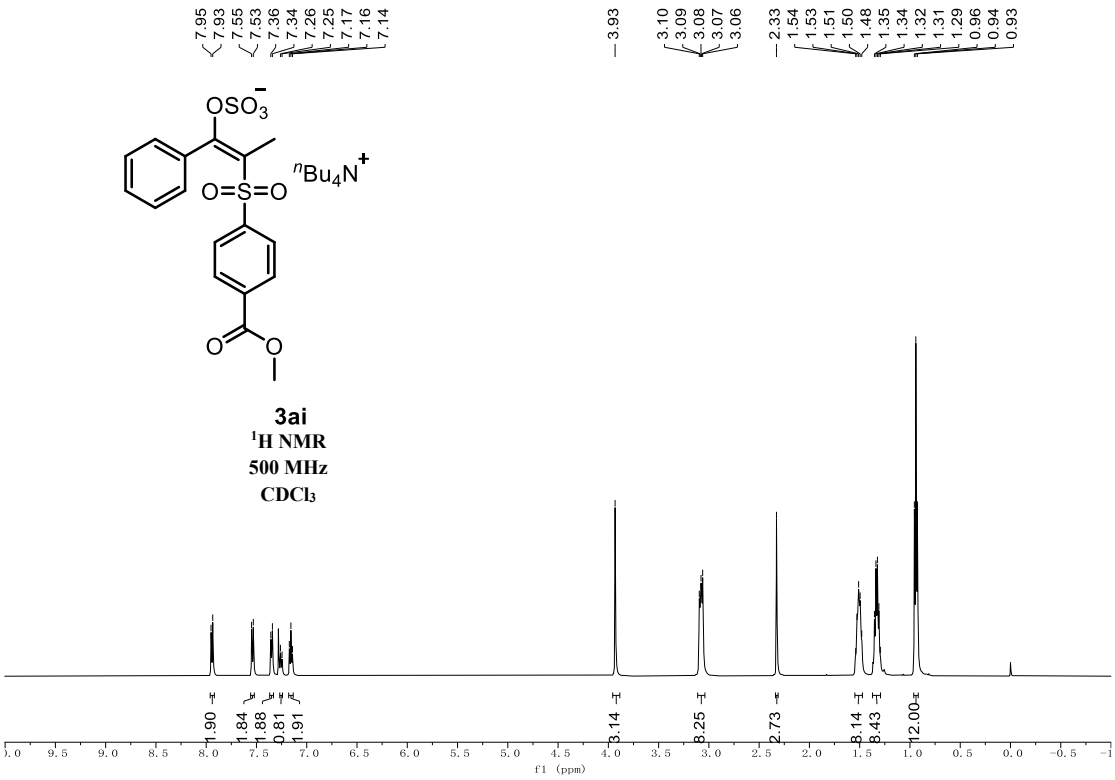
125 MHz
$CDCl_3$



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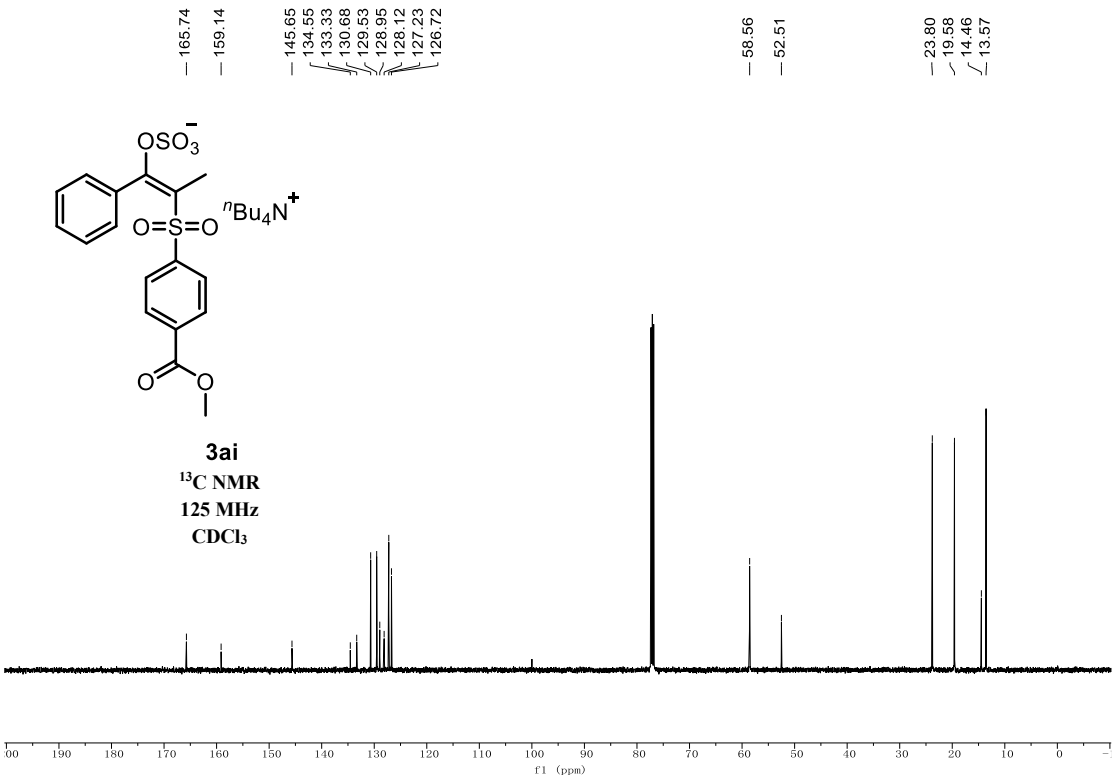
^{1}H NMR
500 MHz
$CDCl_3$



3ai
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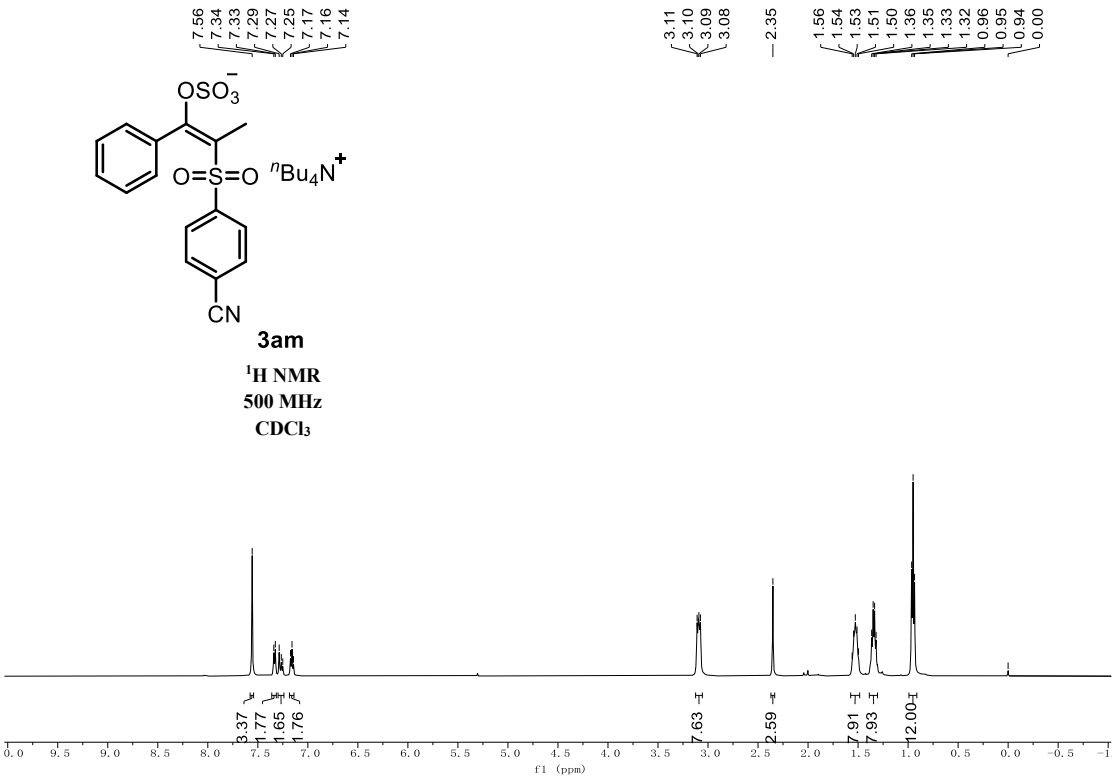
125 MHz
$CDCl_3$

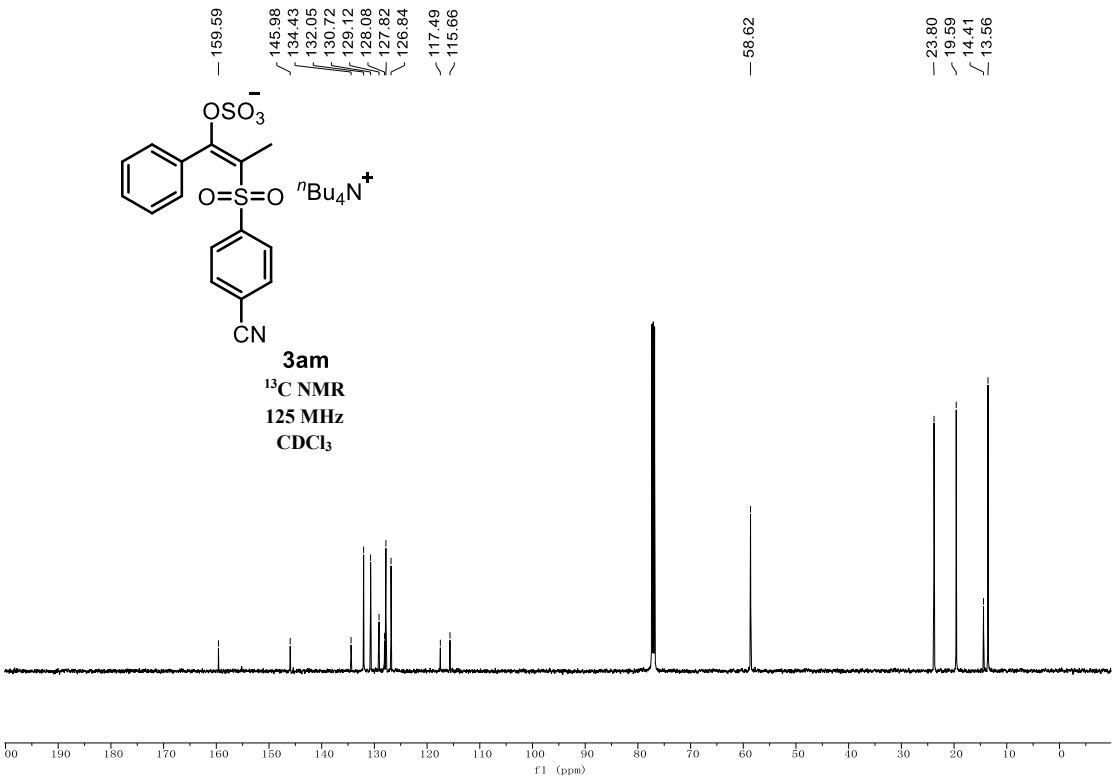


3am

^{1}H NMR

500 MHz

$CDCl_3$

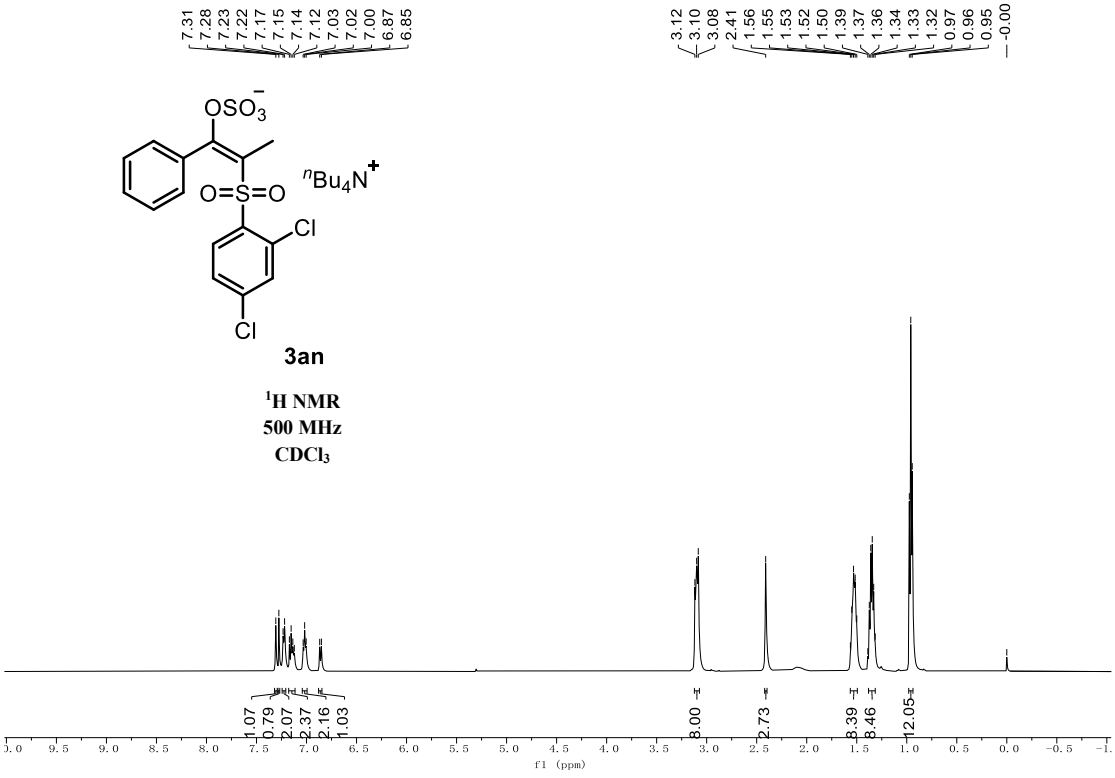


3am

^{13}C NMR

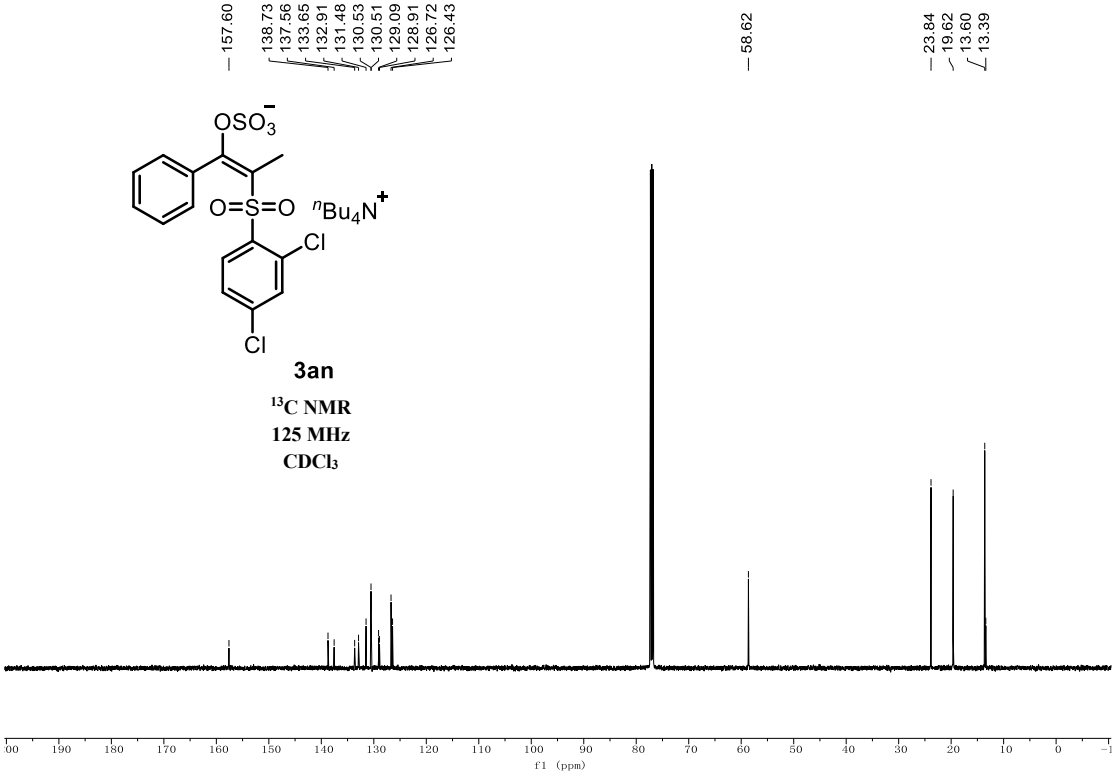
125 MHz

$CDCl_3$



3an

^{1}H NMR
500 MHz
$CDCl_3$



3an

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125 MHz
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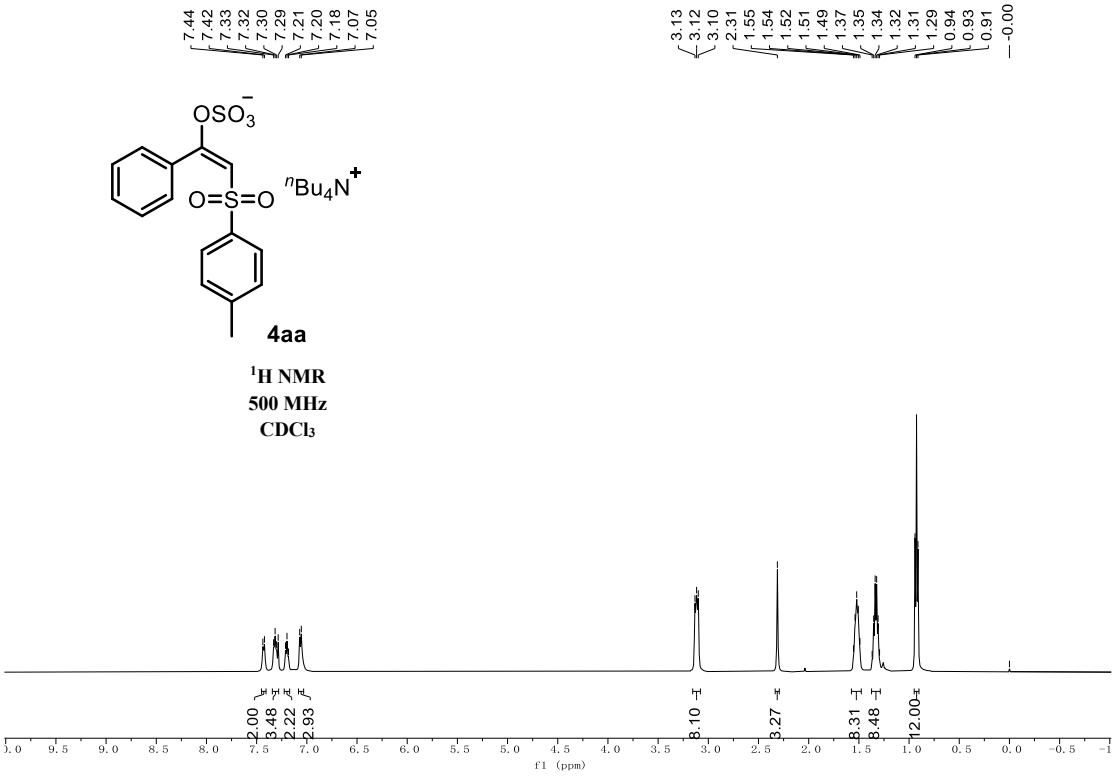

4aa

^{1}H NMR
500 MHz
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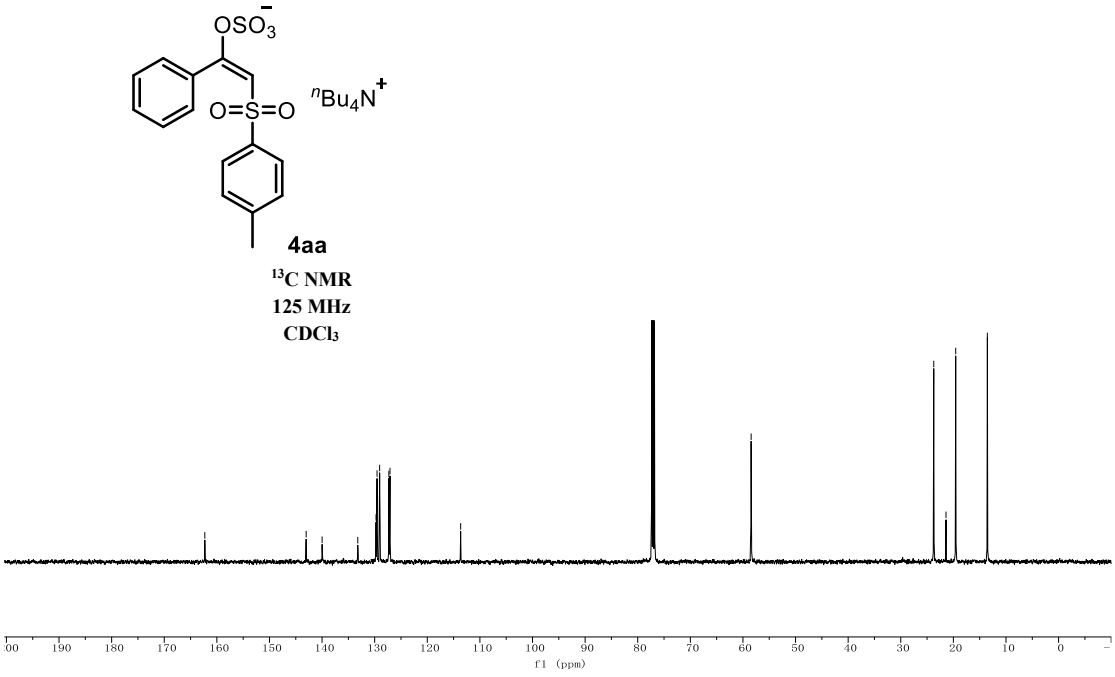
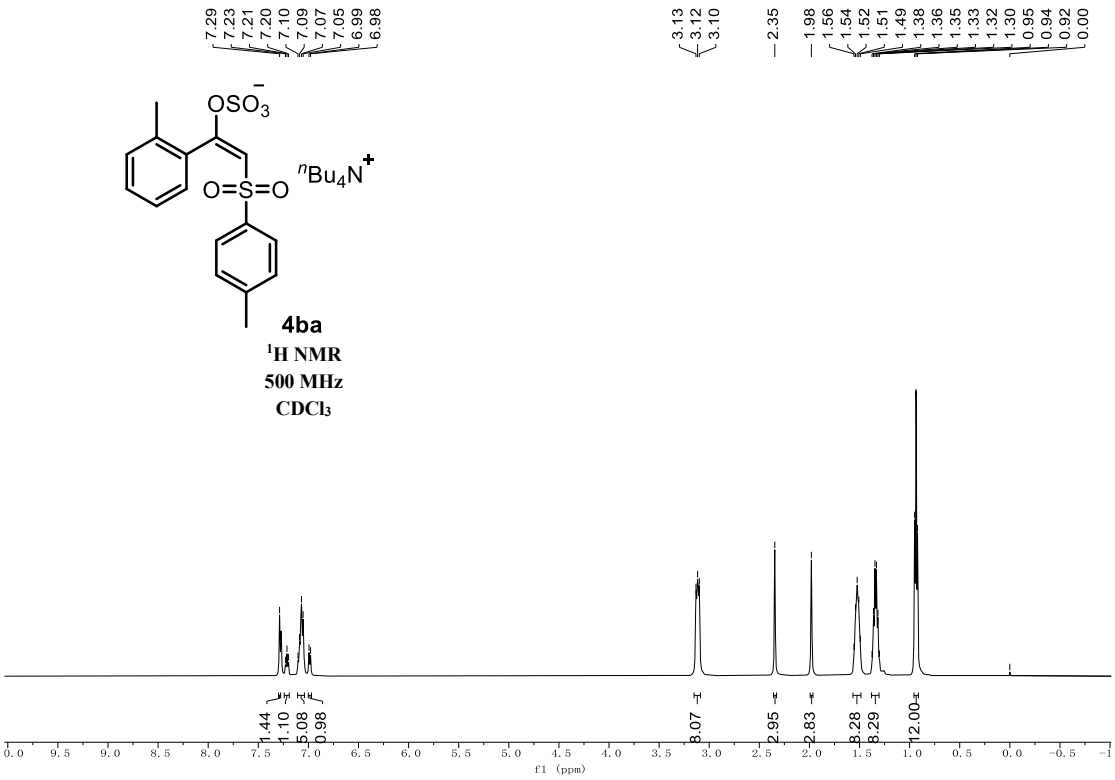


4aa

^{13}C NMR
125 MHz
$CDCl_3$

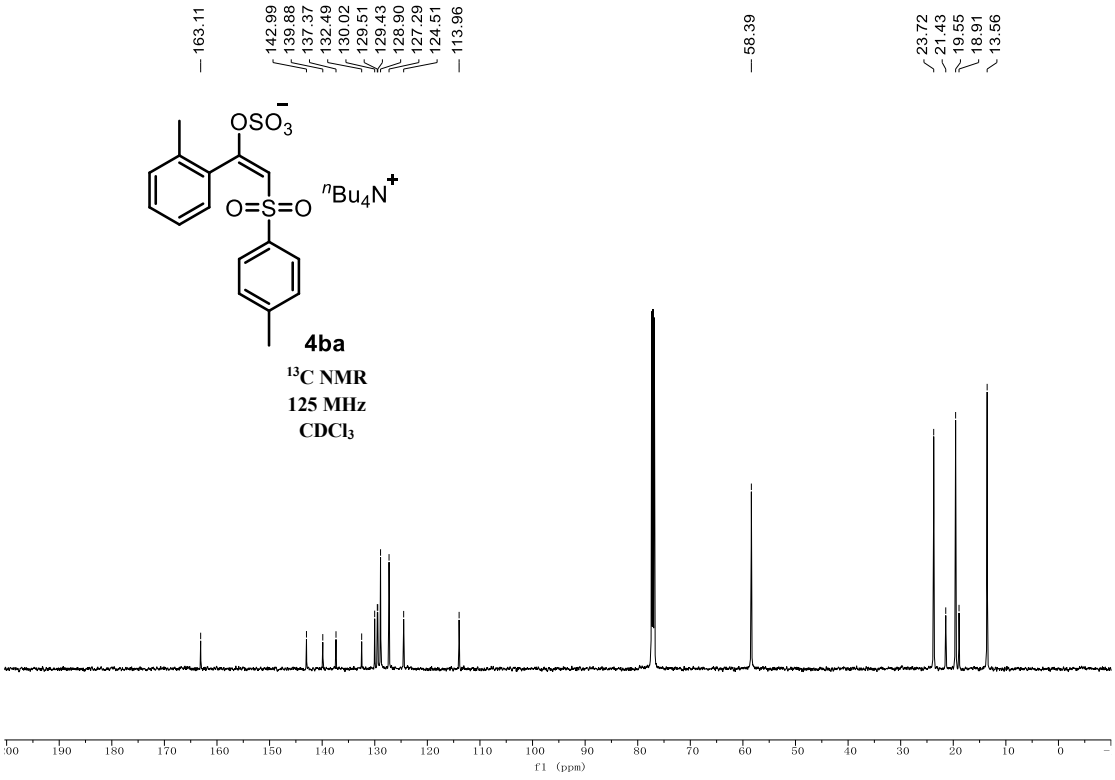


4ba

^{1}H NMR

500 MHz

$CDCl_3$

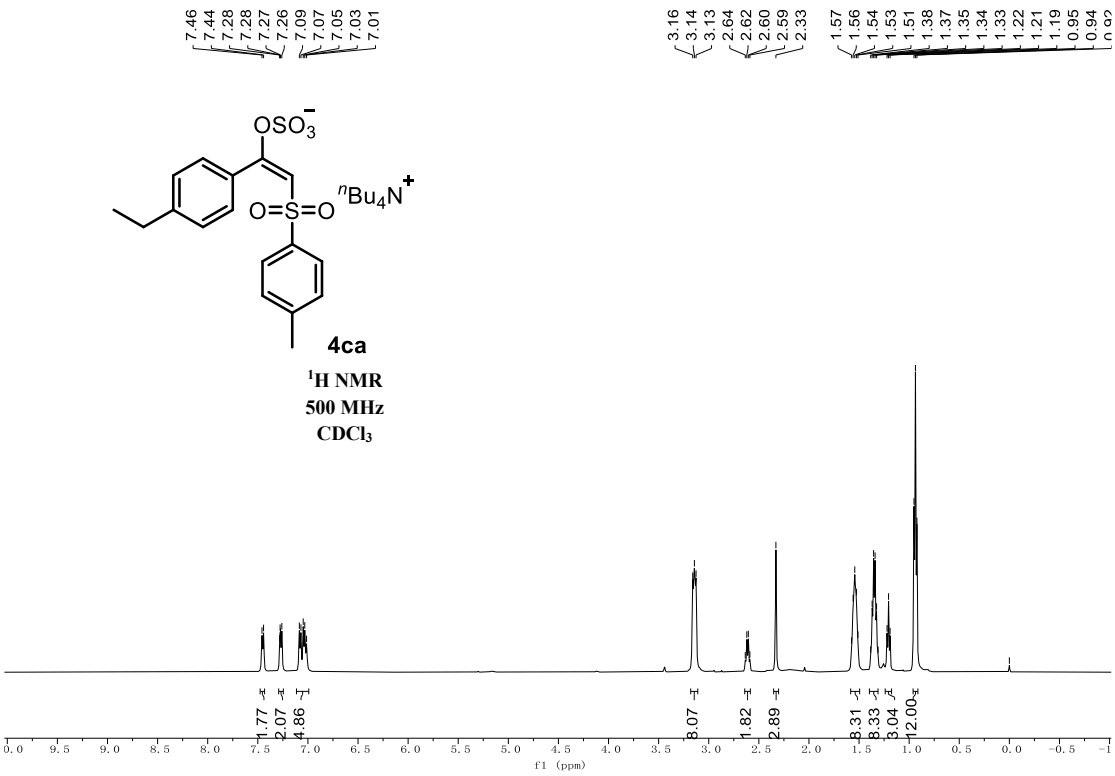


4ba

^{13}C NMR

125 MHz

$CDCl_3$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2.62
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0.94
OSO3−
nBu4N+
O=S=O
4ca
1H NMR
500 MHz
CDCl3
1.77
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4.86
8.07
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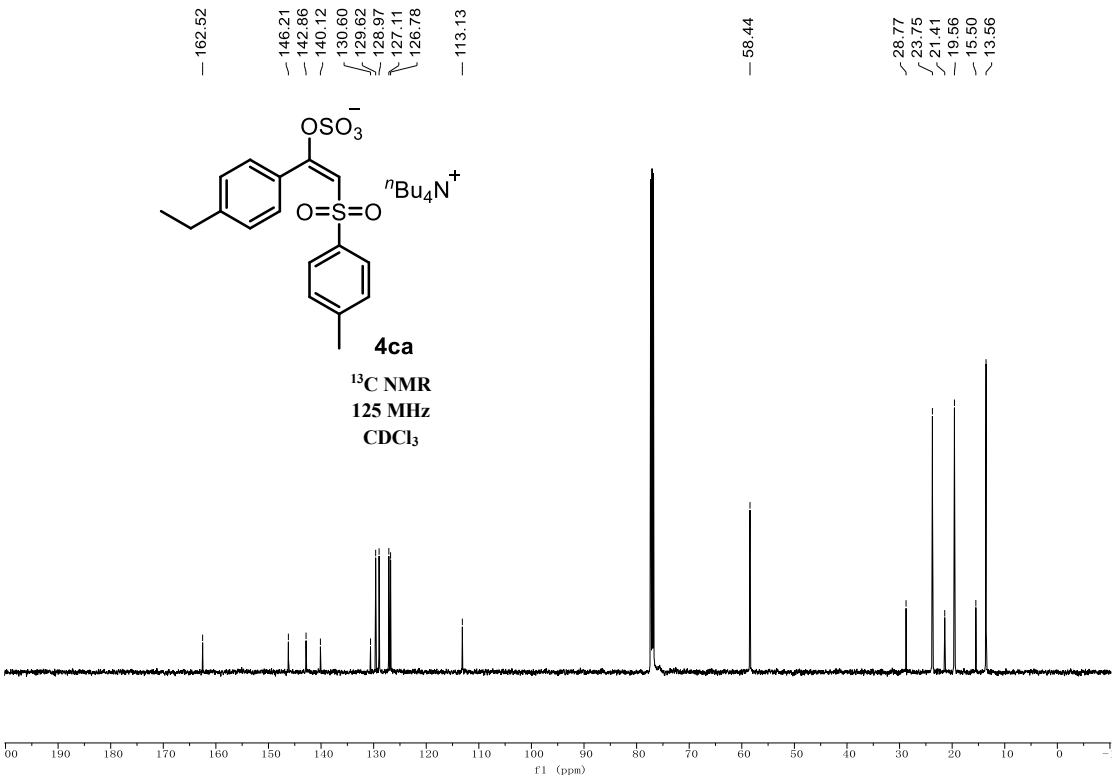
8.31
8.33
8.04
12.00
f1 (ppm)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127.11
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113.13
58.44
28.77
23.75
21.41
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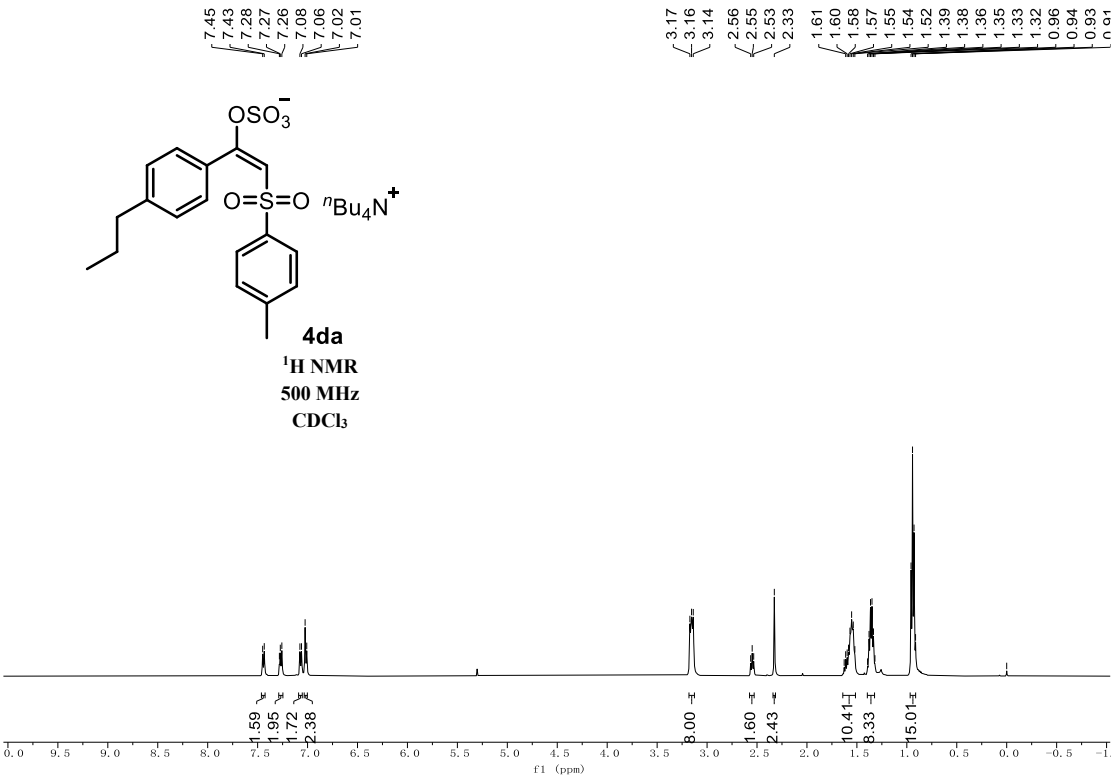
13.56
OSO3−
nBu4N+
O=S=O
4ca
13C NMR
125 MHz
CDCl3
f1 (ppm)

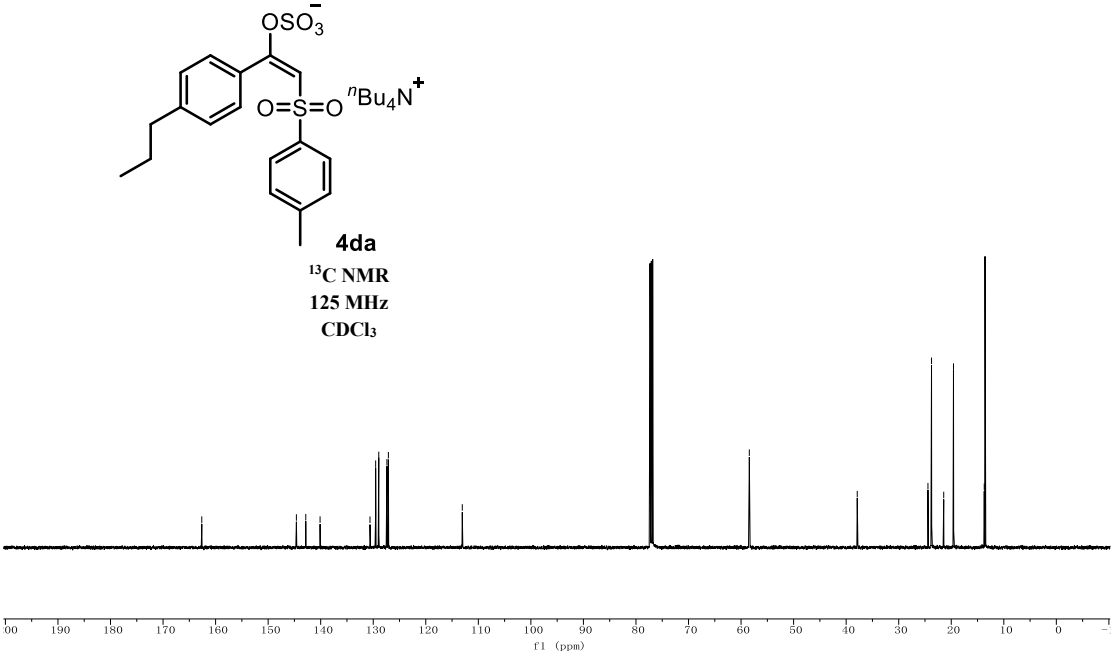


4da

^{1}H NMR

500 MHz

$CDCl_3$

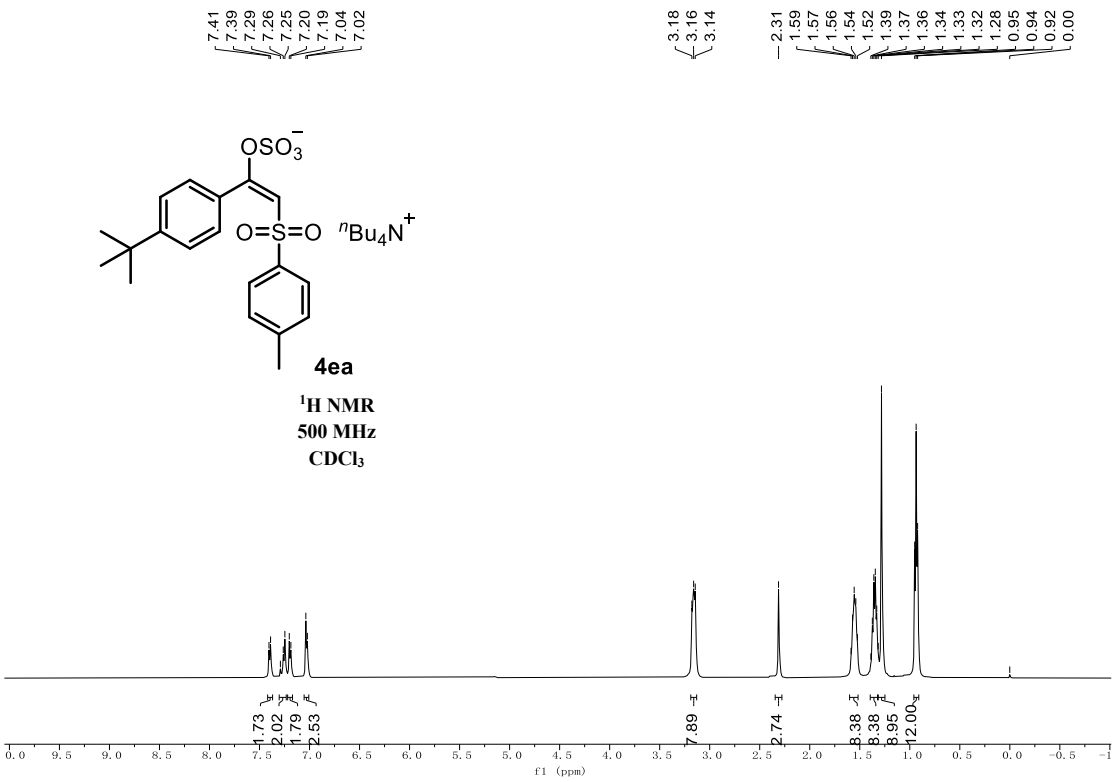


4da

^{13}C NMR

125 MHz

$CDCl_3$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1.57
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1.52
1.39
1.37
1.36
1.34
1.33
1.32
1.28
0.95
0.94
0.92
0.00
OSO3−
O=S=O
nBu4N+
4ea
1H NMR
500 MHz
CDCl3
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8.38
8.38
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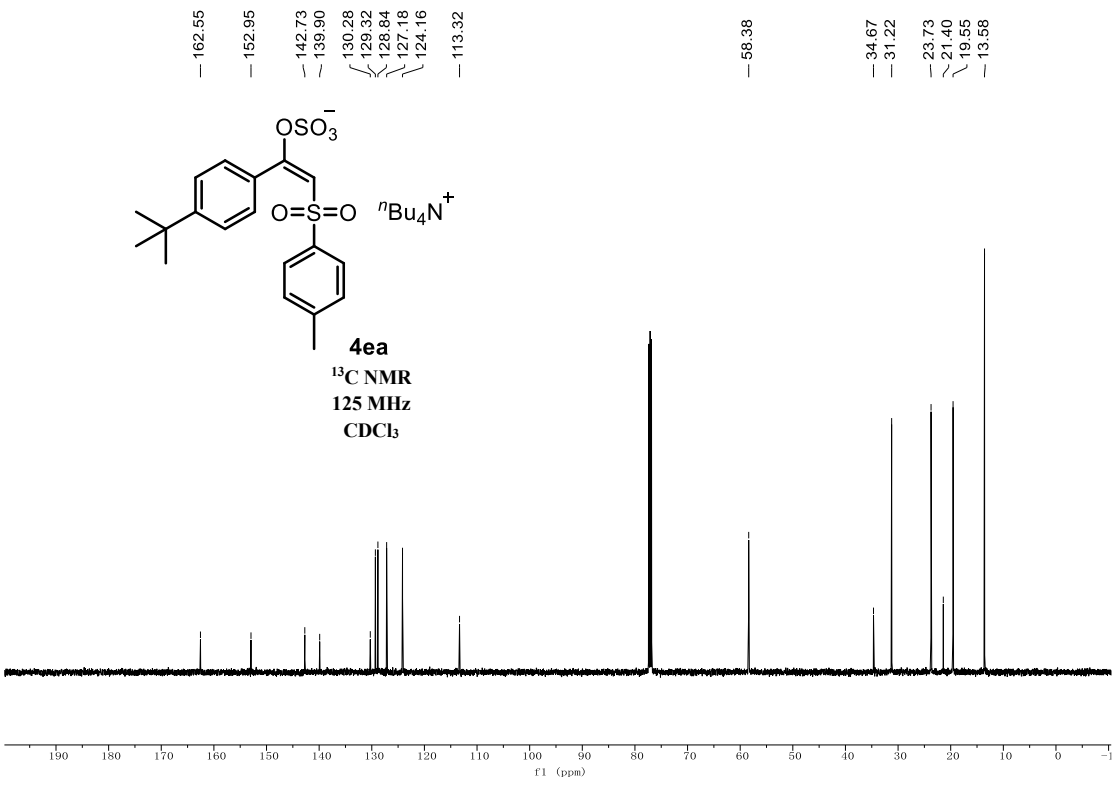
12.00
f1 (ppm)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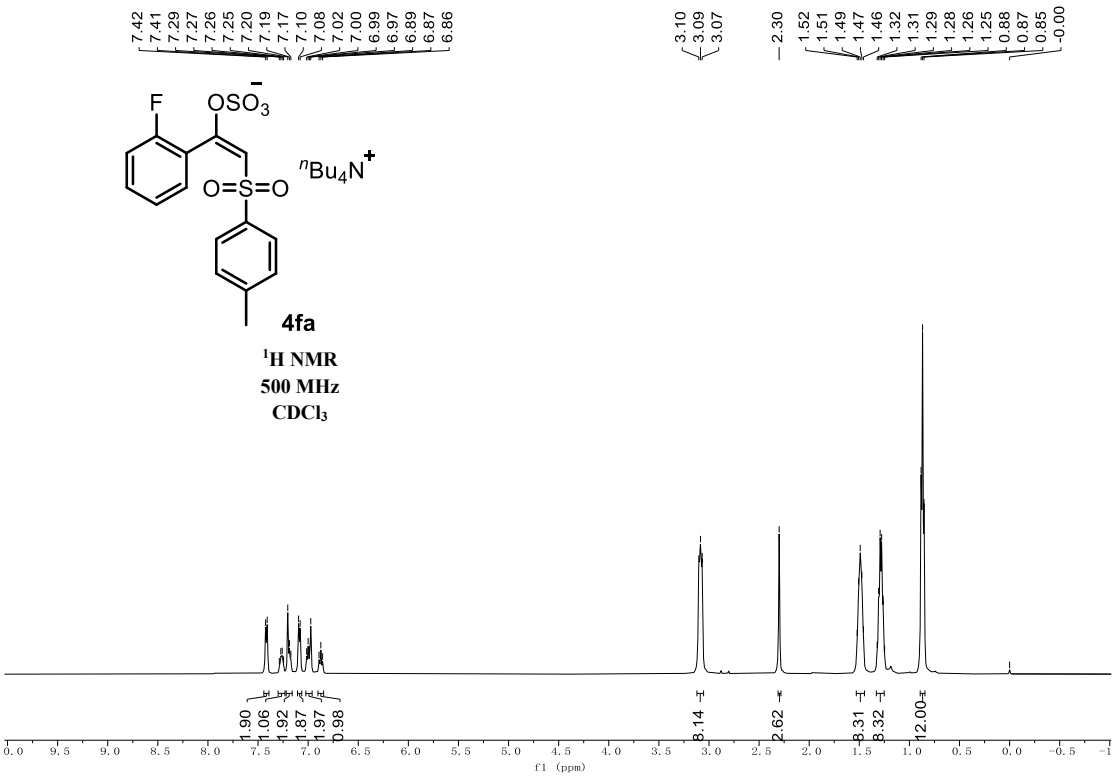
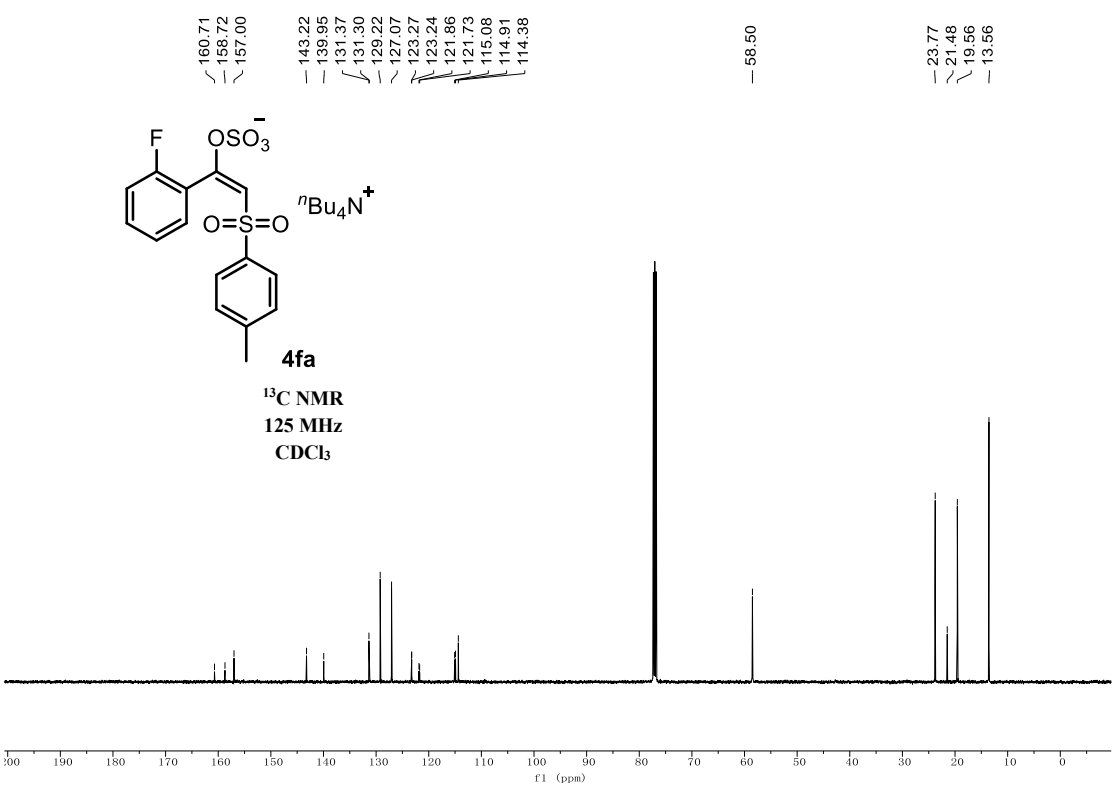
113.32
58.38
34.67
31.22
23.73
21.40
19.55
13.58
OSO3−
O=S=O
nBu4N+
4ea
13C NMR
125 MHz
CDCl3
f1 (ppm)



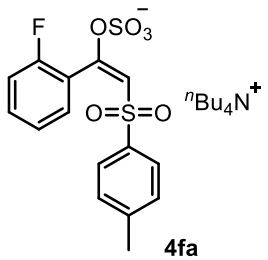
4fa

^{1}H NMR
500 MHz
$CDCl_3$



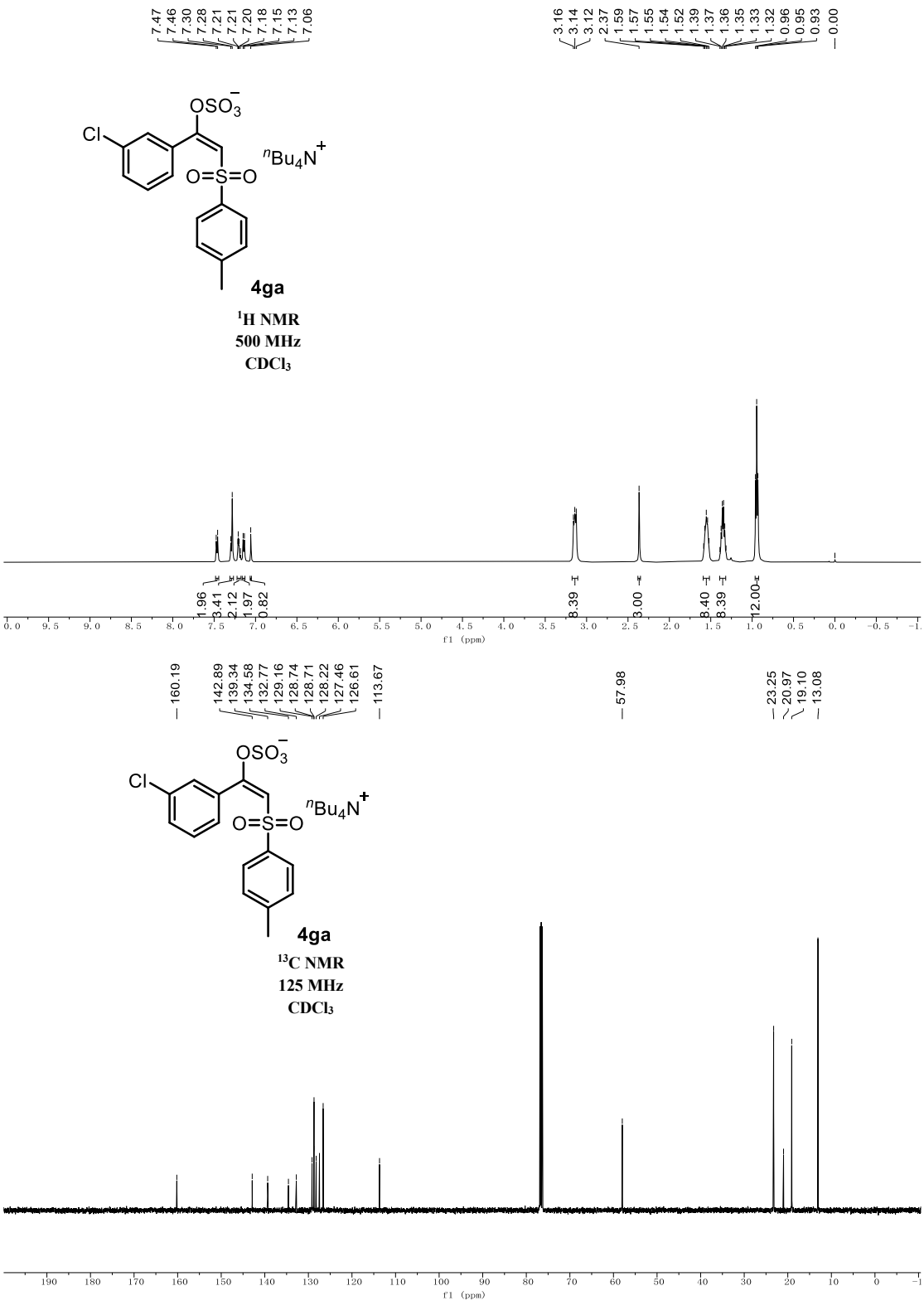
4fa

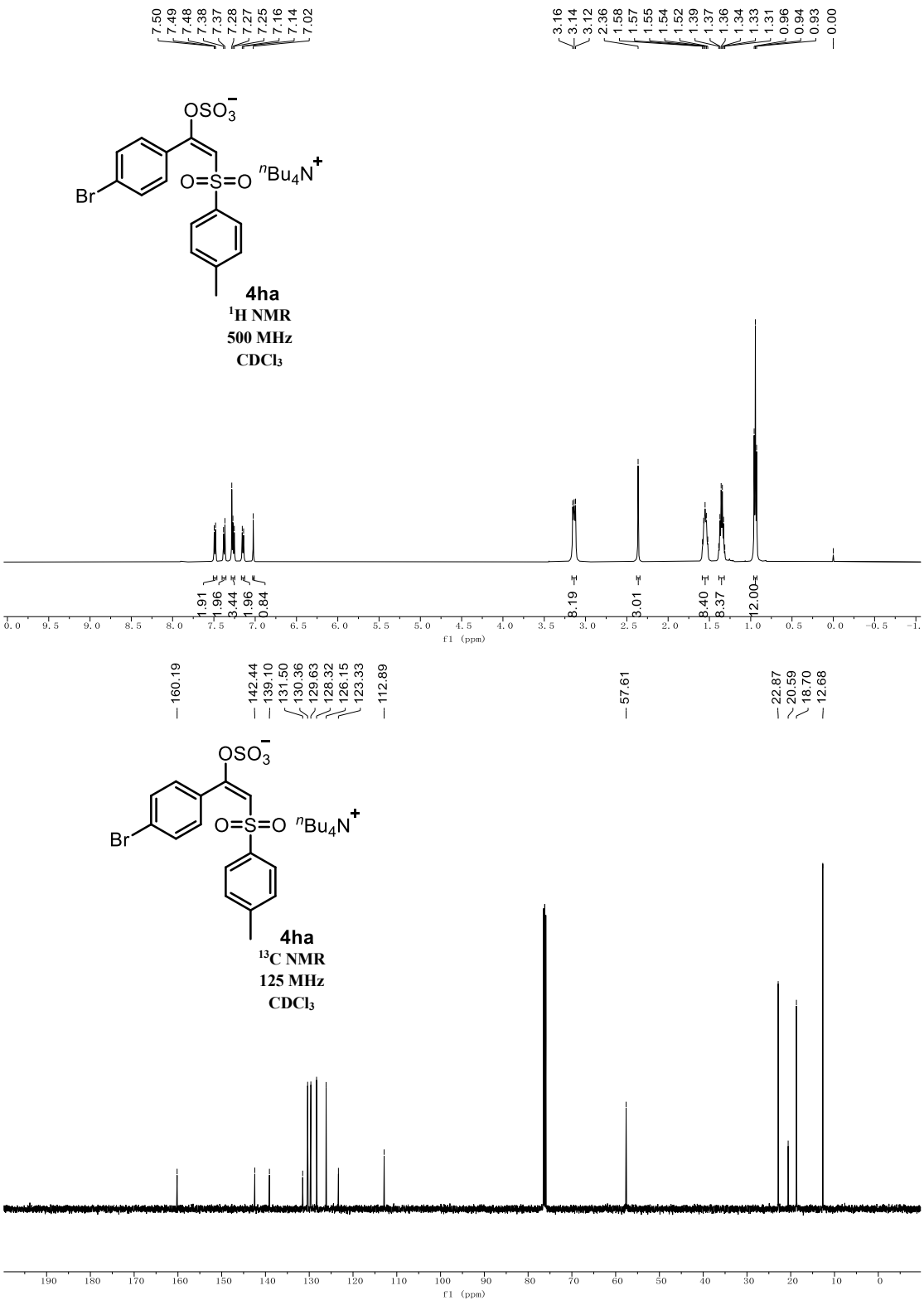
^{13}C NMR
125 MHz
$CDCl_3$

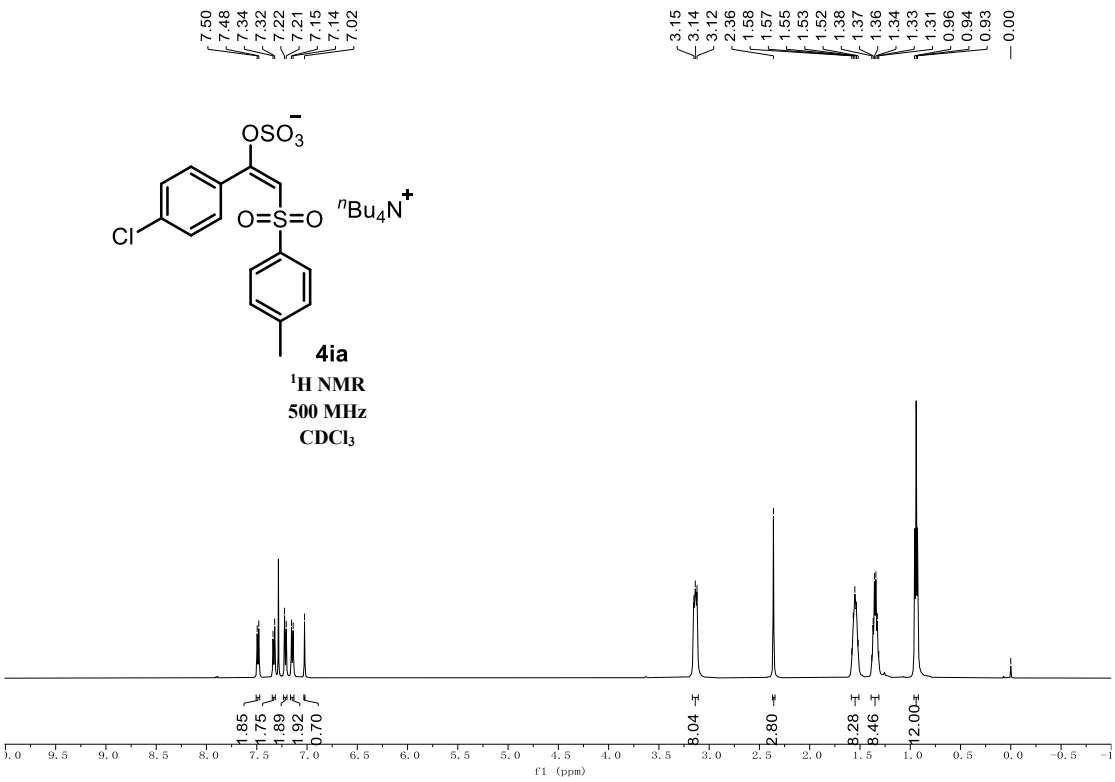


4fa

^{19}F NMR
376 MHz
$CDCl_3$


7.47
7.46
7.30
7.28
7.21
7.21
7.20
7.18
7.15
7.13
7.06
3.16
3.14
3.12
2.37
1.59
1.57
1.55
1.54
1.52
1.39
1.37
1.36
1.35
1.33
1.32
0.96
0.95
0.93
0.00
OSO3−
Cl
nBu4N+
O=S=O
4ga
1H NMR
500 MHz
CDCl3
1.96
3.41
2.12
1.97
0.82
8.39
3.00
8.40
8.39
12.00
f1 (ppm)
160.19
142.89
139.34
134.58
132.77
129.16
128.74
128.71
128.22
127.46
126.61
113.67
57.98
23.25
20.97
19.10
13.08
OSO3−
Cl
nBu4N+
O=S=O
4ga
13C NMR
125 MHz
CDCl3
f1 (ppm)


7.50
7.49
7.48
7.38
7.37
7.28
7.27
7.25
7.16
7.14
7.02
3.16
3.14
3.12
2.36
1.58
1.57
1.55
1.54
1.52
1.39
1.37
1.36
1.34
1.33
1.31
0.96
0.94
0.93
0.00
OSO3−
O=S=O
nBu4N+
Br
4ha
1H NMR
500 MHz
CDCl3
1.91
1.96
3.44
1.96
0.84
8.19
3.01
8.40
8.37
12.00
f1 (ppm)
160.19
142.44
139.10
131.50
130.36
129.63
128.32
126.15
123.33
112.89
57.61
22.87
20.59
18.70
12.68
OSO3−
O=S=O
nBu4N+
Br
4ha
13C NMR
125 MHz
CDCl3
f1 (ppm)

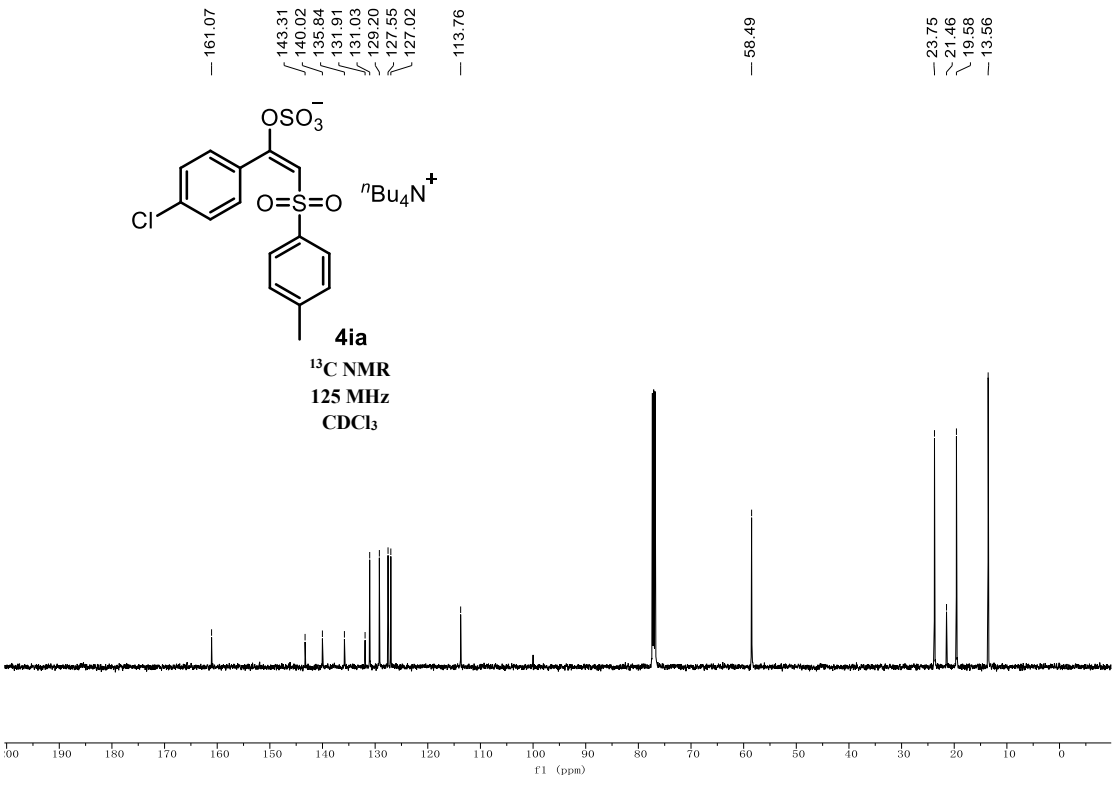


4ia

^{1}H NMR

500 MHz

$CDCl_3$

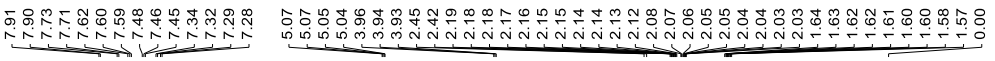


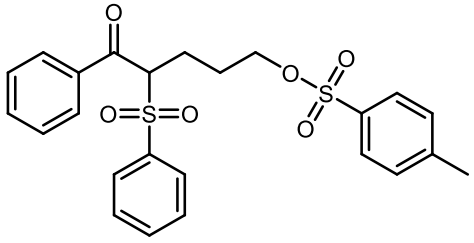
4ia

^{13}C NMR

125 MHz

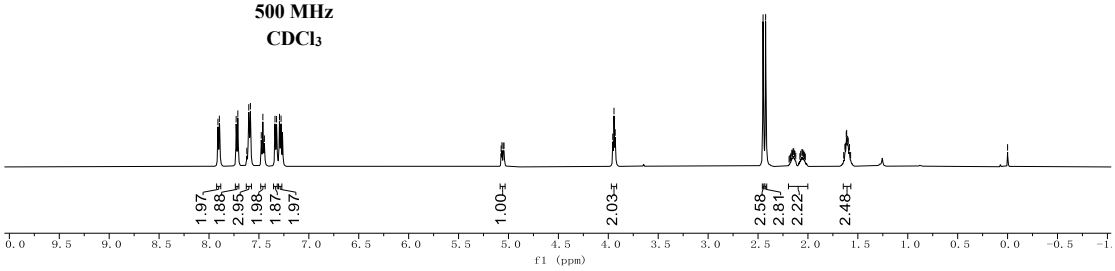
$CDCl_3$

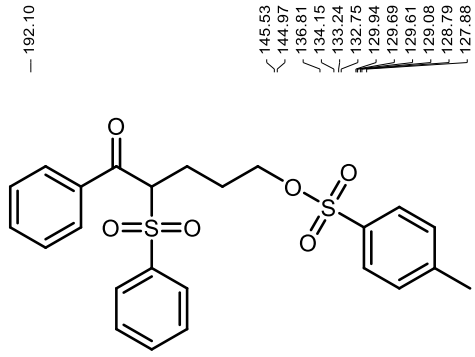




5

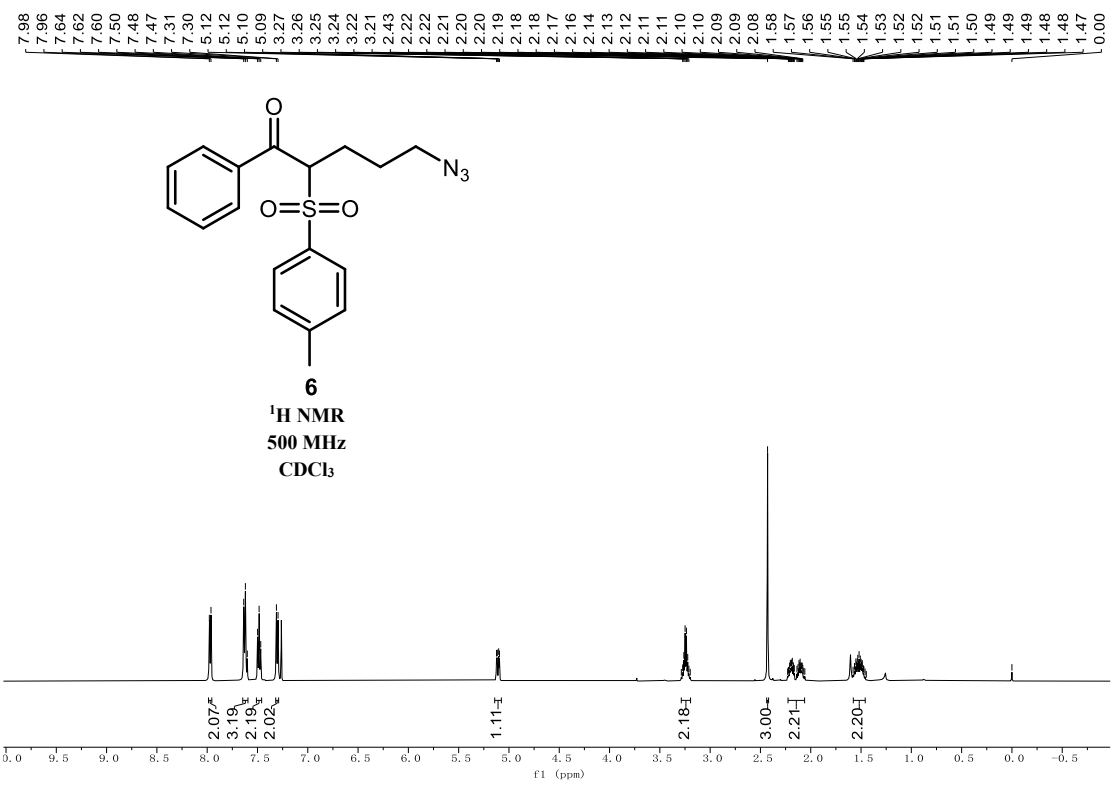
^{1}H NMR
500 MHz
$CDCl_3$

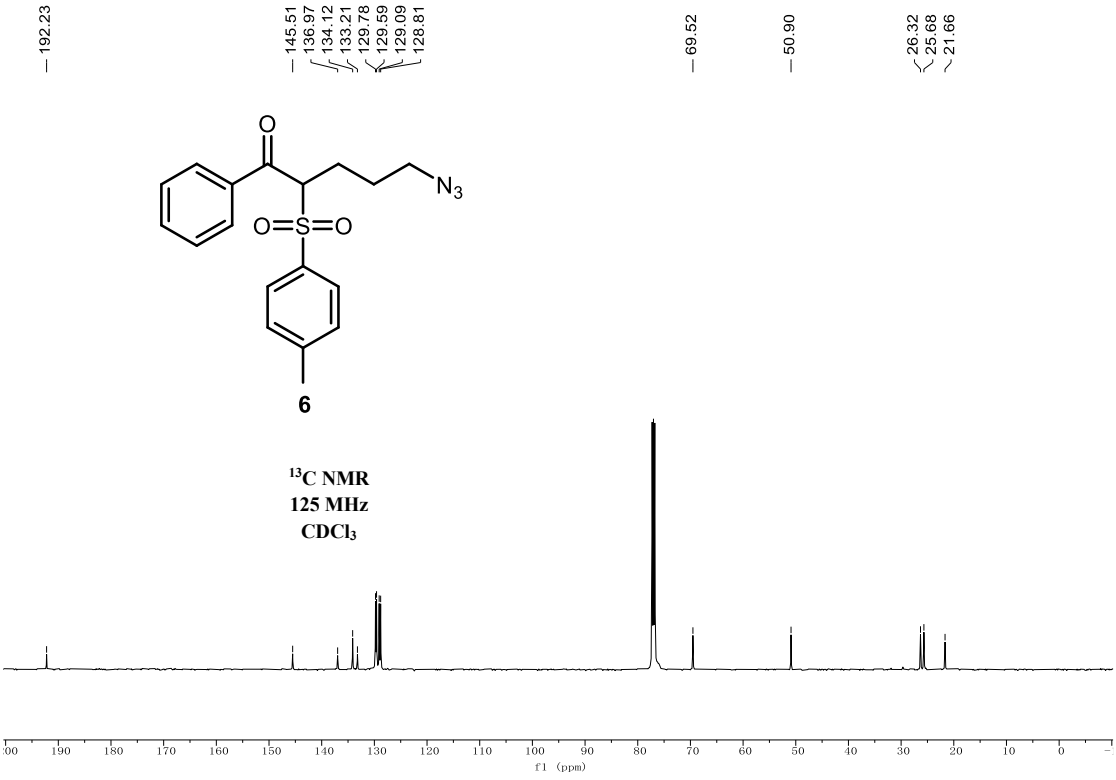


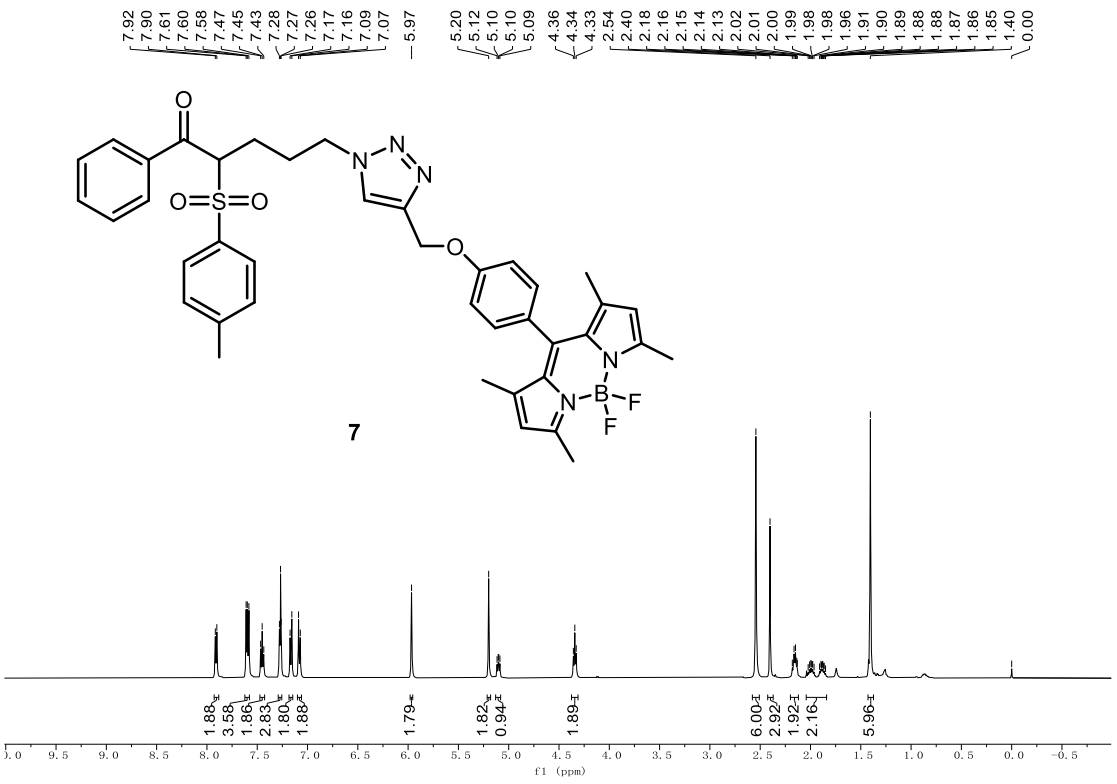


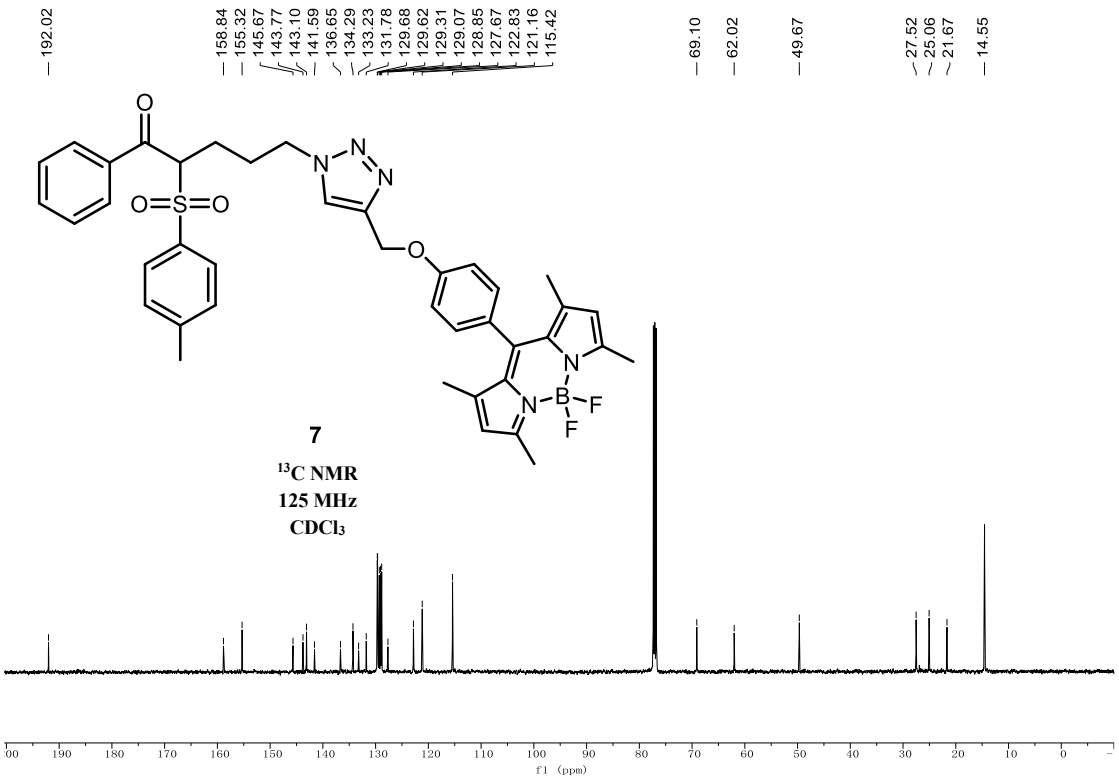
5

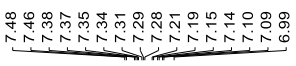
^{13}C NMR
125 MHz
$CDCl_3$

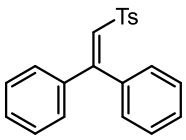

7.98
7.96
7.64
7.62
7.60
7.50
7.48
7.47
7.31
7.30
5.12
5.12
5.10
5.09
3.27
3.26
3.25
3.24
3.22
3.21
2.43
2.22
2.22
2.21
2.20
2.20
2.19
2.18
2.18
2.17
2.16
2.14
2.13
2.12
2.11
2.11
2.10
2.10
2.09
2.09
2.08
1.58
1.57
1.56
1.55
1.55
1.54
1.53
1.52
1.52
1.51
1.51
1.50
1.49
1.49
1.49
1.48
1.48
1.47
0.00
O
N3
O=S=O
6
1H NMR
500 MHz
CDCl3
2.07
3.19
2.19
2.02
1.11
2.18
3.00
2.21
2.20
f1 (ppm)


192.23
145.51
136.97
134.12
133.21
129.78
129.59
129.09
128.81
69.52
50.90
26.32
25.68
21.66
O
N3
O=S=O
6
13C NMR
125 MHz
CDCl3
f1 (ppm)


7.92
7.90
7.61
7.60
7.58
7.47
7.45
7.43
7.28
7.27
7.26
7.17
7.16
7.09
7.07
5.97
5.20
5.12
5.10
5.10
5.09
4.36
4.34
4.33
2.54
2.40
2.18
2.16
2.15
2.14
2.13
2.02
2.01
2.00
1.99
1.98
1.98
1.96
1.91
1.90
1.89
1.88
1.88
1.87
1.86
1.85
1.40
0.00
7
1.88
3.58
1.86
2.83
1.80
1.88
1.79
1.82
0.94
1.89
6.00
2.92
1.92
2.16
5.96
f1 (ppm)

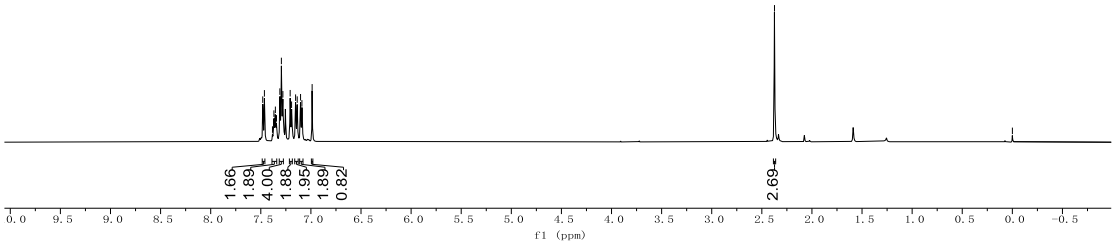

192.02
158.84
155.32
145.67
143.77
143.10
141.59
136.65
134.29
133.23
131.78
129.68
129.62
129.31
129.07
128.85
127.67
122.83
121.16
115.42
69.10
62.02
49.67
27.52
25.06
21.67
14.55
7
13C NMR
125 MHz
CDCl3
f1 (ppm)

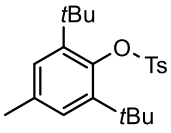




8

^{1}H NMR
500 MHz
$CDCl_3$





9

^{1}H NMR
500 MHz
$CDCl_3$