

# *Supporting Information for*

## **C(sp<sup>3</sup>)-H 1,3-Diamination of Cumene Derivatives Catalyzed by a Dirhodium(II) Catalyst**

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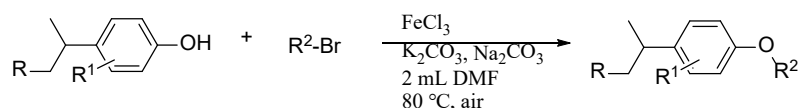
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## 1. General experimental details

$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR spectra were obtained on a Bruker spectrometer at 400 MHz, 101 MHz and 376 MHz. Unless otherwise stated, the  $^1\text{H}$  NMR (400 MHz) chemical shifts were recorded relative to  $\text{CDCl}_3$  as the internal reference ( $\text{CDCl}_3$ :  $\delta\text{H} = 7.260$  ppm). The  $^{13}\text{C}$  NMR (100 MHz) chemical shifts were given using  $\text{CDCl}_3$  as the internal standard ( $\text{CDCl}_3$ :  $\delta\text{C} = 77.160$  ppm).  $^1\text{H}$  NMR data are reported as: chemical shift (ppm), multiplicity (s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, p = quintet, hept = heptet, m = multiplet), and coupling constant (Hz). All solvents before used were dried and degassed by standard methods. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Yields reported are NMR yields or isolated yields.  $\text{Rh}_2(\text{esp})_2$ ,<sup>1</sup>  $\text{Rh}_2(5 - \text{Br} - \text{esp})_2$ ,<sup>2</sup>  $\text{Rh}_2(5 - \text{Cl} - \text{esp})_2$ ,  $\text{Rh}_2(5 - \text{tBu} - \text{esp})_2$ ,<sup>3</sup>  $\text{Rh}_2(5 - \text{Me} - \text{esp})_2$ ,  $\text{Rh}_2(\text{h} - \text{esp})_4$ ,  $\text{Rh}_2(\text{OPiv})_4$ ,  $\text{Rh}_2(\text{cap})_4$  and  $\text{Rh}_2(6 - \text{NO}_2 - \text{esp})_2$  were prepared as previously described. **1c** – **1j**, **1n** – **1t** and **1x** – **1z** were synthesized following the general procedure. High resolution mass spectrometric measurements were carried out using a Bruker autoflex MALDI-TOF mass spectrometer and Waters-Q-TOF Premier (ESI). X-Ray single-crystal diffraction data were collected on an Agilent Technologies Gemini plus single crystal diffraction and solved using SHELX program. Flash column chromatography was carried out using 300-400 or 200-300 mesh silica gel at increased pressure.

## 2. General procedure.

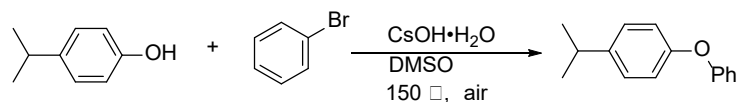
### General procedure for **1c** and **1x**<sup>4</sup>



2 mL DMF was added to a mixture of phenol (5 mmol, 1 eq.), Alkyl bromide (5.5 mmol, 1.1 eq.), K<sub>2</sub>CO<sub>3</sub>(0.6 mmol, 0.12 eq.), Na<sub>2</sub>CO<sub>3</sub>(5.4 mmol, 1.08 eq.) and Ferric chloride (0.25 mmol, 0.05 eq.) in a 10 mL sealed tube, The resulting mixture was stirred at 80 °C under air for 6 h. After cooling, the mixture was filtered through a short pad of silica gel, the filter cake was subsequently washed with CH<sub>2</sub>Cl<sub>2</sub>. After evaporation of the

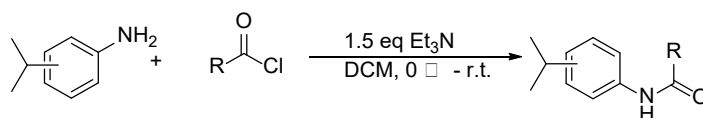
organic solvent, the residue was purified by silica gel column chromatography (hexanes) to give the title product as a colorless oil.

#### General procedure for 1d<sup>5</sup>



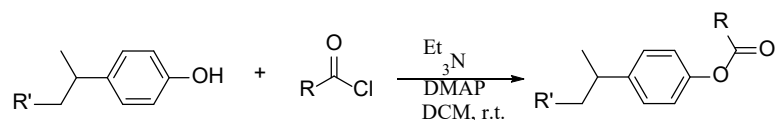
1 mL DMSO was added to a mixture of 4-isopropylphenol (163 mg, 1.2 mmol), bromobenzene (156 mg, 1 mmol), CsOH · H<sub>2</sub>O (330 mg, 2.2 mmol) in a 10 mL tube. The resulting mixture was stirred at 150 °C under air for 15 h. Then the mixture was filtered through a short pad of silica gel, which was subsequently washed with CH<sub>2</sub>Cl<sub>2</sub>. After evaporation of the organic solvent, the residue was purified by silica gel column chromatography (hexanes) to give the title product as a colorless oil.

#### General procedure for 1e - 1g<sup>6</sup>



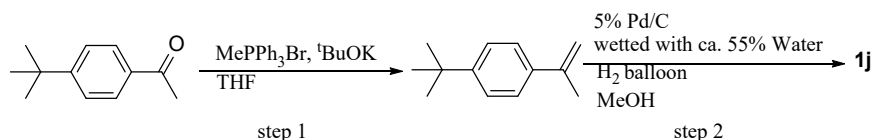
To a solution of aniline (500 mg, 1.0 eq.), triethylamine (1.5 eq.) in dry DCM (10 mL) at 0 °C under N<sub>2</sub> atmosphere, acyl chloride (1.03 eq.) was added slowly by syringe. The resulting solution was slowly allowed to warm to room temperature and monitored by TLC. After the reaction was finished, water was added to quench this reaction. Then the mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> and the combined organic phase was washed with brine. The organic phase was then dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the corresponding products.

#### General procedure for 1h, 1i and 1y<sup>7</sup>



To a solution of phenol (500 mg, 1.0 eq.), triethylamine (1.2 eq.), DMAP (4-Dimethylaminopyridine, 0.1 eq.) in dry DCM (15 mL) at 0 °C under N<sub>2</sub> atmosphere, acyl chloride (1.2 eq.) was added slowly by syringe. The resulting solution was slowly allowed to warm to room temperature and monitored by TLC. After the reaction was finished, water was added to quench this reaction. Then the mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> and the combined organic phase was washed with brine. The organics was then dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The obtained residue was purified by flash chromatography to afford the corresponding products.

### General procedure for 1j



#### Step 1:<sup>8</sup>

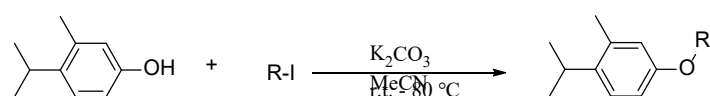
MePPh<sub>3</sub>Br (2.5 g, 6.8 mmol, 1.2 eq.) was added to a suspension of <sup>t</sup>BuOK (0.77 g, 6.8 mmol, 1.2 eq.) in anhydrous THF (10 mL) under argon atmosphere. 1-(4-(tert-butyl)phenyl)ethan-1-one (1 g, 5.7 mmol, 1 eq.) was then added after the suspension was stirred at room temperature for 1 h. The resulting mixture was stirred at same temperature for 1 h. Then the mixture was filtered through a short pad of silica gel and the filter cake was subsequently washed with hexanes. After evaporation of the organic solvent, the residue was purified by silica gel column chromatography (hexanes) to give the title product as a colorless oil (660 mg, 67% yield).

#### Step 2:<sup>9</sup>

1-(tert-butyl)-4-(prop-1-en-2-yl)benzene (1 g) was added to a suspension of Pd/C (100 mg, 5% Pd/C wetted with ca. 55% Water) in anhydrous MeOH (10 m

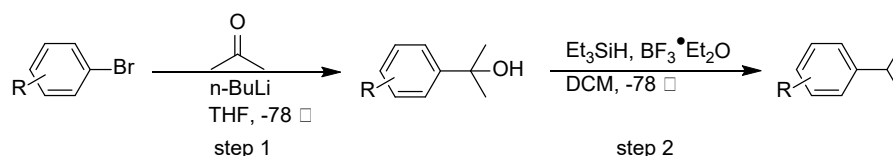
L) in a 25 mL round bottom flask equipped with a hydrogen balloon. Then the resulting mixture was stirred at room temperature and monitored by TLC. After the reaction finished, the mixture was filtered through a short pad of silica gel, the filter cake was subsequently washed with hexanes. After evaporation of the organic solvent, the residue was purified by silica gel column chromatography (hexanes) to give the title product as a colorless oil (750 mg, 75% yield).

### General procedure for 1n and 1o<sup>10</sup>



To a 50 mL sealing tube equipped with a stir bar was charged with phenol (1 g, 1 eq.) and K<sub>2</sub>CO<sub>3</sub> (1.5 eq.), 10 mL CH<sub>3</sub>CN was added. Iodoalkane (1.5 eq.) was added after the resulting mixture was stirred for 1 hour at room temperature. Then the reaction temperature was raised to 80 °C. Once the reaction completed monitored by TLC, the mixture was filtered through a short pad of silica gel, the filter cake was subsequently washed with hexanes. After evaporation of the organic solvent, the residue was purified by silica gel column chromatography (hexanes) to give the title product as a colorless oil.

### General procedure for 1p – 1s



#### Step 1:<sup>11</sup>

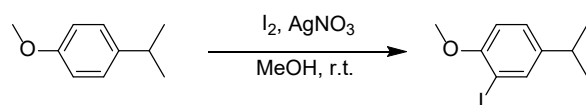
To a 100 mL tube equipped with a stir bar, bromobenzene (1 g, 1eq.) was added. After sealed by a rubber plug, the tube was evacuated and backfilled with argon for three times, THF (20 mL) was added then. After the mixture was cooled to -78 °C, n-BuLi (1.2 eq. 2.5 M in hexane) was added slowly with syringe.

ringe. Acetone (1.2 eq.) was added after the mixture was stirred for 30 minutes. Once the reaction completed monitored by TLC, the mixture was quenched with saturated  $\text{NH}_4\text{Cl}$  solution. Then this mixture was extracted with dichloromethane. The combined organic phase was dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether/ethyl acetate) to afford the tertiary alcohol.

### Step 2:<sup>12</sup>

To a 100 mL tube equipped with a stir bar, tertiary alcohol (1eq.) was added. After sealed by a rubber plug, the tube was evacuated and backfilled with argon for three times, DCM (20 mL) was added then. After the mixture was cooled to  $-78\text{ }^\circ\text{C}$ ,  $\text{Et}_3\text{SiH}$  (2 eq.) was added. After 30 minutes,  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  (3.2 eq.) was added slowly by syringe at same temperature. Once the reaction completed monitored by TLC, the mixture was quenched with saturated  $\text{NaHCO}_3$  solution. Then this mixture was extracted with dichloromethane. The combined organic phase was dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether) to give the title product.

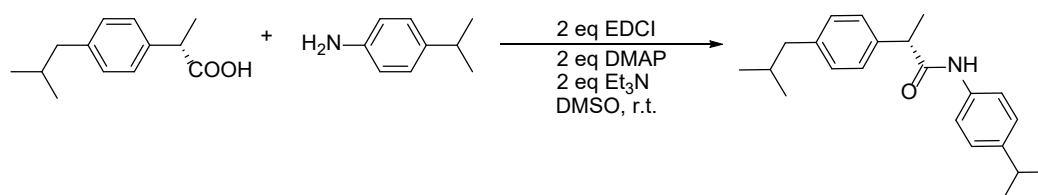
### Procedure for synthesis of 1t<sup>13</sup>



To a 10 mL tube equipped with a stir bar, 1.8 M solution of iodine (1 eq.) in methanol and  $\text{AgNO}_3$  (1 eq.) was added, then the 1.8M solution of 1-isopropyl-4-methoxybenzene (1 eq.) in methanol was added. The resulting mixture was stirred at room temperature for 18h. Then the mixture was filtered through a short pad of silica gel, the filter cake was subsequently washed with dichloromethane. Saturated  $\text{Na}_2\text{S}_2\text{O}_3$  solution was added then to the mother liquid until t

he solution became colorless. Then this mixture was extracted with dichloromethane. The combined organic phase was dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether) to give the title product.

### Procedure for synthesis of **1y**



To a 25 mL round-bottomed flask equipped with a stir bar was charged with (S)-Ibuprofen (14.8 mmol, 2 eq.), 4-isopropylaniline (7.4 mmol, 1 eq.), EDCI (3-(((ethylimino)methylene)amino)-N,N-dimethylpropan-1-amine hydrochloride (14.8 mmol, 2eq.), DMAP (4-Dimethylaminopyridine, 14.8 mmol, 2 eq.) and  $\text{Et}_3\text{N}$  (14.8 mmol, 2 eq.), 10 mL DMSO was added. The mixture was stirred at room temperature for 12h. After the reaction completed, water was added to quench this reaction. Then the mixture was extracted with  $\text{CH}_2\text{Cl}_2$  and the organic phase was washed with brine. The organics was then dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the corresponding products as a white solid (2 g, 84% yield).

### General procedure for synthesis of **2a – 2z, 3a – 3z, 2aa and 3aa**

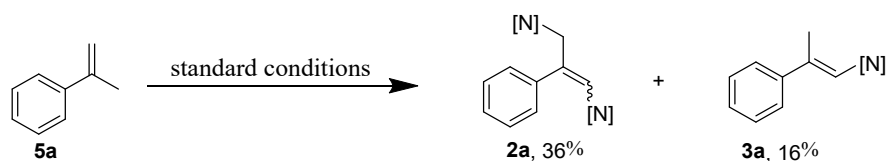
To a 25 mL tube equipped with a stir bar, substrates **1a – 1z** and **1aa** (0.4 mmol), Dirhodium(II) - catalyst, NFSI (378 mg, 1.2 mmol) and  $\text{NaHCO}_3$  (67 mg, 0.8 mmol) were added. After sealed by a rubber plug, the tube was evacuated and backfilled with nitrogen for three times,  $\text{CHCl}_3$  (1 mL or 2 mL) was added then. The mixture was subsequently stirred at 65 °C. Monitored by TLC,

after the reaction finished, Et<sub>3</sub>N was added to quench excess NFSI. Then the resulting mixture was added 20 mL H<sub>2</sub>O and extracted with dichloromethane. The combined organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether/ethyl acetate) to afford the desired products.

### 3. Mechanism studies ([N] = N(SO<sub>2</sub>Ph)<sub>2</sub>)

#### 2-Phenyl-1-propene (**5a**) is the probable intermediate

To a 25 mL tube equipped with a stir bar, **5a** (0.4 mmol), Rh<sub>2</sub>(5-<sup>t</sup>Bu-esp)<sub>2</sub> (3.5 mg, 0.004 mmol), NFSI (378 mg, 1.2 mmol) and NaHCO<sub>3</sub> (67 mg, 0.8 mmol) were added. After sealed by a rubber plug, the tube was evacuated and backfilled with nitrogen for three times, CHCl<sub>3</sub> (2 mL) was added then. The mixture was subsequently stirred at 65 °C. Monitored by TLC, After the reaction finished, Et<sub>3</sub>N was added to quench excess NFSI. Then the resulting mixture was added 20 mL H<sub>2</sub>O and extracted subsequently with dichloromethane. The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether/ethyl acetate) to afford the desired products.

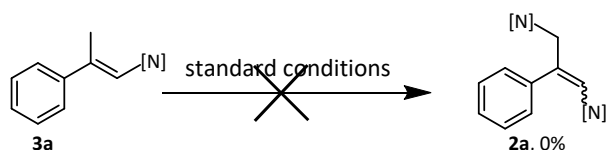


#### **2a and 3a are obtained by two different paths**

To a 25 mL tube equipped with a stir bar, **3a** (50 mg, 0.121 mmol), Rh<sub>2</sub>(5-<sup>t</sup>Bu-esp)<sub>2</sub> (1 mg, 0.0012 mmol), NFSI (114 mg, 0.363 mmol), NaHCO<sub>3</sub> (20 mg, 0.242 mmol) were added. After sealed by a rubber plug, the tube was evacuated and backfilled with nitrogen for three times, CHCl<sub>3</sub> (1 mL) was added then. The mixture was subsequently

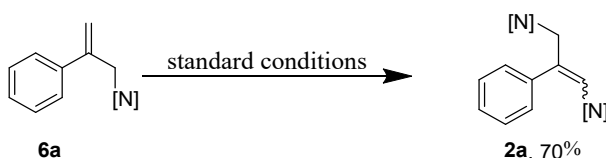


stirred at 65 °C. Not any reaction was detected with TLC.



### **6a<sup>14</sup> is the possible important intermediate for the 2a's generation**

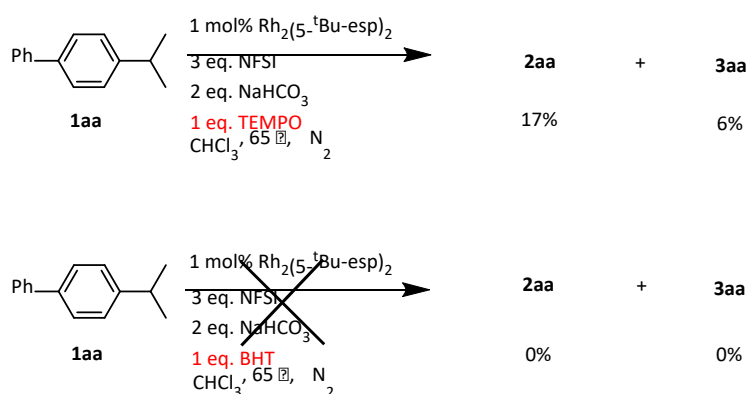
To a 25 mL tube equipped with a stir bar, **6a** (50 mg, 0.121 mmol), Rh<sub>2</sub>(5 - <sup>t</sup>Bu - esp)<sub>2</sub> (1 mg, 0.0012 mmol), NFSI (114 mg, 0.363 mmol) and NaHCO<sub>3</sub> (20 mg, 0.242 mmol) were added. After sealed by a rubber plug, the tube was evacuated and backfilled with nitrogen for three times, CHCl<sub>3</sub> (1 mL) was added then. The mixture was subsequently stirred at 65 °C. Monitored by TLC, After the reaction finished, Et<sub>3</sub>N was added to quench excess NFSI. Then the resulting mixture was added 20 mL H<sub>2</sub>O and extracted subsequently with dichloromethane. The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (petrol ether/ethyl acetate) to afford the desired products.



### **Experiments of radical inhibition.**

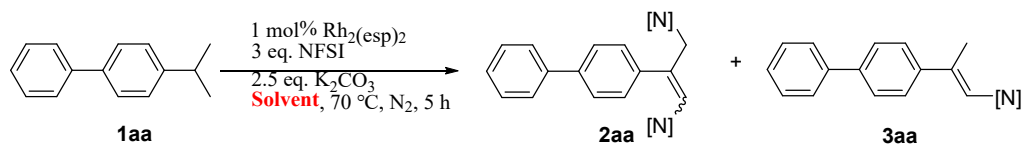
To a 25 mL tube equipped with a stir bar was charged with **1aa** (0.4 mmol), Rh<sub>2</sub>(5 - <sup>t</sup>Bu - esp)<sub>2</sub> (3.5 mg, 0.004 mmol), NFSI (378 mg, 1.2 mmol), NaHCO<sub>3</sub> (67 mg, 0.8 mmol), and CHCl<sub>3</sub> (2 mL), TEMPO (2, 2, 6, 6 - Tetramethylpiperidinoxy, 63 mg, 0.4 mmol) or BHT (2, 6 - Di - tert - butyl - 4 - methylphenol, 88 mg, 0.4 mmol) was added at last. After sealed by a rubber plug, the tube was evacuated and backfilled with nitrogen for three times at low temp

erature. The mixture was subsequently stirred at 65 °C. After five hours, Et<sub>3</sub>N was added to quench NFSI. Then the resulting mixture was added 20 mL H<sub>2</sub>O and extracted subsequently with dichloromethane. The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The yields of **2aa**, **3aa** and the ratio of *E/Z* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard.



#### 4. Optimization details ([N] = N(SO<sub>2</sub>Ph)<sub>2</sub>)

Table S1 Screening of **solvents**<sup>a</sup>.

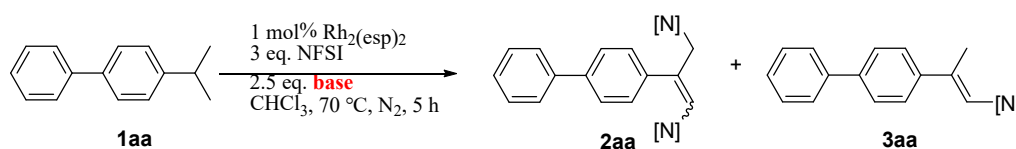


Entry	Solvent	Yield <sup>b</sup> (%)	
		<b>2aa</b> ( <i>Z/E</i> )	<b>3aa</b>
1	CH <sub>2</sub> Cl <sub>2</sub>	28 (75/25)	9
2	DME <sup>c</sup>	9 (68/32)	3
3	EtOAc	N.R.	N.R.
4	Acetonitrile	N.R.	N.R.
5	1,4 - Dioxane	19 (74/26)	6
6	THF	N.R.	N.R.
7	MeOH	N.R.	N.R.

8	CHCl <sub>3</sub>	70 (78/22)	20
9	Acetone	trace	trace
10	Hexane	trace	trace
11	DCE	34 (77/23)	10
12	Benzene	46 (78/22)	14

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), Rh<sub>2</sub>(esp)<sub>2</sub> (0.004 mmol), NFSI (3 eq., 1.2 mmol), K<sub>2</sub>CO<sub>3</sub> (2.5 eq., 1 mmol) and solvent (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**, **3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. DME = Dimethoxyethane. DCE = 1, 2 - Dichloroethane. NFSI = N - Fluorobenzenesulfonimide.

**Table S2** Screening of **bases**<sup>a</sup>.



Entry	Base	Yield <sup>b</sup> (%)	
		<b>2aa</b> ( <i>Z/E</i> )	<b>3aa</b>
1	Na <sub>2</sub> CO <sub>3</sub>	75(81/19)	21
2	NaHCO <sub>3</sub>	75(79/21)	21
3	K <sub>2</sub> CO <sub>3</sub>	64(78/22)	20
4	K <sub>2</sub> HPO <sub>4</sub>	74(79/21)	20
5	K <sub>2</sub> HPO <sub>4</sub> ·3H <sub>2</sub> O	64(78/22)	18
6	KF	65(78/22)	18
7	NaOH	60(74/26)	25
8	NaOAc	8(65/35)	4
9	MgO	12(67/33)	6
10	None	8(71/29)	6

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), Rh<sub>2</sub>(esp)<sub>2</sub> (0.004 mmol), NFSI (3 eq., 1.2 mmol), base (2.5 eq., 1 mmol) and CHCl<sub>3</sub> (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**, **3aa** and the ratio

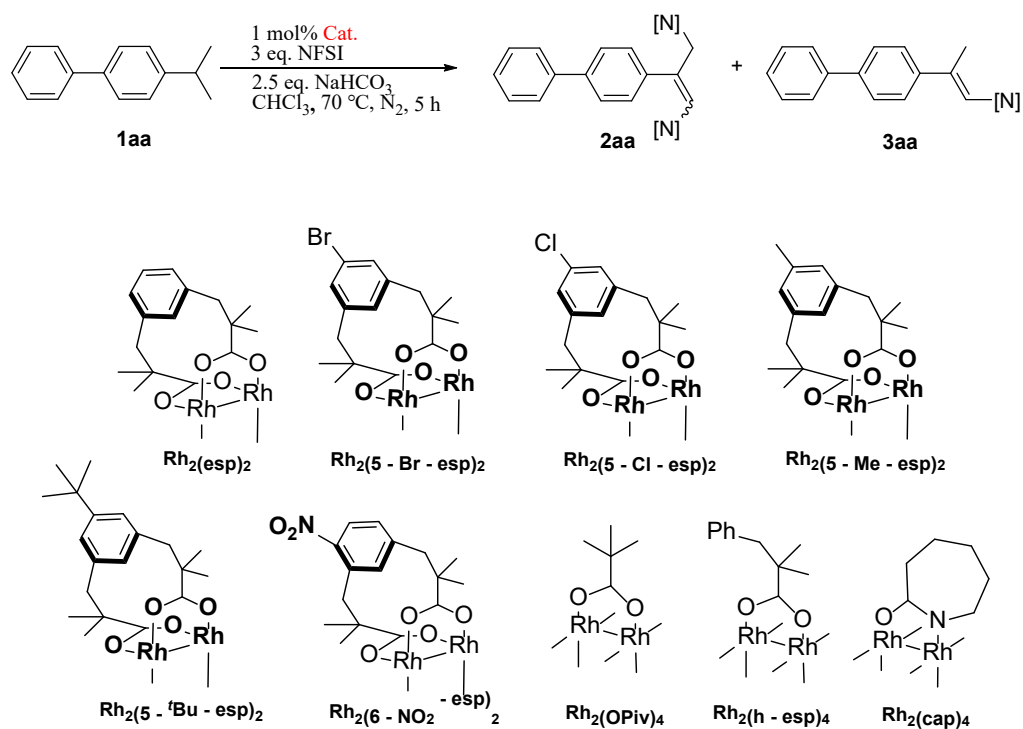
of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. NFSI = N - Fluorobenzenesulfonimide.

**Table S3** Screening of other metal catalysts<sup>a</sup>.

Entry	Catalyst	Yield <sup>b</sup> (%)	
		2aa ( <i>Z/E</i> )	3aa
1	Cu(OAc) <sub>2</sub>	N.D.	N.D.
2	Cu(acac) <sub>2</sub>	trace	trace
3	Cu(TFA) <sub>2</sub>	N.D.	N.D.
4	CuI	N.D.	N.D.
5	CuCl	N.D.	N.D.
6	Fe(acac) <sub>2</sub>	N.R.	N.R.
7	FeCl <sub>3</sub>	N.R.	N.R.
8	Mn(OAc) <sub>2</sub>	N.R.	N.R.
9	Mn(acac) <sub>2</sub>	N.D.	N.D.
10	Co(OAc) <sub>2</sub>	N.D.	N.D.
11	Co(acac) <sub>2</sub>	N.R.	N.R.
12	(Cp* <sup>+</sup> RhCl <sub>2</sub> ) <sub>2</sub>	N.R.	N.R.
13	RhCl <sub>3</sub> · 3H <sub>2</sub> O	N.R.	N.R.

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), catalyst (0.02 mmol), NFSI (3 eq., 1.2 mmol), NaHCO<sub>3</sub> (2.5 eq., 1 mmol) and CHCl<sub>3</sub> (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2a**, **3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. (Cp\*<sup>+</sup>RhCl<sub>2</sub>)<sub>2</sub> = Bis[(pentamethylcyclopentadienyl)dichloro-rhodium]. NFSI = N - Fluorobenzenesulfonimide.

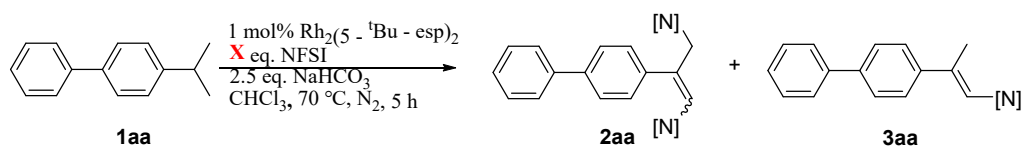
**Table S4** Screening of Dirhodium catalysts <sup>a</sup>.



Entry	Cat.	Yield <sup>b</sup> (%)	
		2aa ( <i>Z/E</i> )	3aa
1	Rh <sub>2</sub> (OAc) <sub>4</sub>	65(78/22)	18
2	Rh <sub>2</sub> (tfa) <sub>4</sub>	N.D.	N.D.
3	Rh <sub>2</sub> (cap) <sub>4</sub>	7(74/26)	3
4	Rh <sub>2</sub> (opiv) <sub>4</sub>	51(76/24)	15
5	Rh <sub>2</sub> (esp) <sub>2</sub>	75(78/22)	21
6	Rh <sub>2</sub> (5 - <sup>t</sup> Bu - esp) <sub>2</sub>	77(80/20)	21
7	Rh <sub>2</sub> (5 - Br - esp) <sub>2</sub>	71(80/20)	20
8	Rh <sub>2</sub> (5 - Cl - esp) <sub>2</sub>	71(79/21)	19
9	Rh <sub>2</sub> (5 - Me - esp) <sub>2</sub>	77(79/21)	20
10	Rh <sub>2</sub> (h - esp) <sub>4</sub>	65(77/23)	17
11	Rh <sub>2</sub> (6 - NO <sub>2</sub> - esp) <sub>2</sub>	74(79/21)	22
12	None	N.R.	N.R.

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), catalyst (0.004 mmol), NFSI (3 eq., 1.2 mmol), NaHCO<sub>3</sub> (2.5 eq., 1 mmol) and CHCl<sub>3</sub> (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**, **3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. NFSI = N - Fluorobenzenesulfonimide.

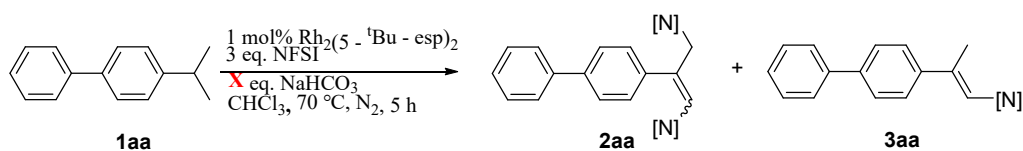
**Table S5** Screening of equivalent of NFSI <sup>a</sup>.



Entry	X	Yield <sup>b</sup> (%)	
		<b>2aa</b> ( <i>Z/E</i> )	<b>3aa</b>
1	1	25(80/20)	9
2	2	54(79/21)	18
3	3	71(78/22)	22
4	4	59(79/21)	18
5	5	60(77/23)	18

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), Rh<sub>2</sub>(5-<sup>t</sup>Bu-esp)<sub>2</sub> (0.004 mmol), NFSI (X eq., X \* 0.4 mmol), NaHCO<sub>3</sub> (2.5 eq., 1 mmol) and CHCl<sub>3</sub> (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**, **3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. NFSI = N - Fluorobenzenesulfonimide.

**Table S6** Screening of equivalent of NaHCO<sub>3</sub> <sup>a</sup>.

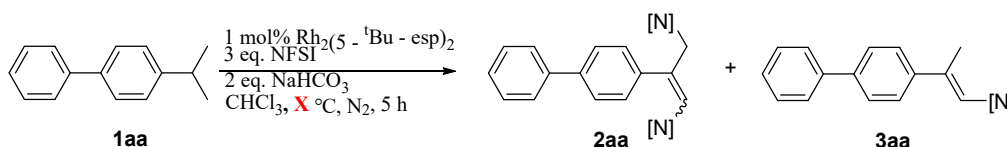


Entry	X	Yield <sup>b</sup> (%)	
		<b>2aa</b> ( <i>Z/E</i> )	<b>3aa</b>
1	2	76(79/21)	22

2	2.5	74(79/21)	22
3	3	77(79/21)	22

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), Rh<sub>2</sub>(5 - <sup>t</sup>Bu - esp)<sub>2</sub> (0.004 mmol), NFSI (3 eq., 1.2 mmol), NaHCO<sub>3</sub> (X eq., X \* 0.4 mmol) and CHCl<sub>3</sub> (2 mL) at 70 °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**、**3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard. NFSI = N - Fluorobenzenesulfonimide.

**Table S7** Screening of reaction temperature <sup>a</sup>.



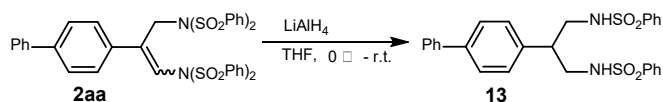
Entry	X	Yield <sup>b</sup> (%)	
		<b>2aa</b> ( <i>Z/E</i> )	<b>3aa</b>
1	r.t.	15(78/22)	5
2	40	28(75/25)	9
3	50	50(78/22)	17
4	60	72(80/20)	22
5	65	78(79/21) [70] <sup>c</sup>	21[25] <sup>c</sup>
6	70	77(79/21)	22
7	90	67(76/24)	19

<sup>a</sup>Reaction conditions: **1aa** (0.4 mmol), Rh<sub>2</sub>(5 - <sup>t</sup>Bu - esp)<sub>2</sub> (0.004 mmol), NFSI (3 eq., 1.2 mmol), NaHCO<sub>3</sub> (2 eq., 0.8 mmol) and CHCl<sub>3</sub> (2 mL) at X °C for 5 h under nitrogen. <sup>b</sup>Yields of **2aa**、**3aa** and the ratio of *Z/E* of **2aa** are determined via <sup>1</sup>H NMR with nitromethane as the internal standard.

<sup>c</sup>Isolated yields. NFSI = N - Fluorobenzenesulfonimide.

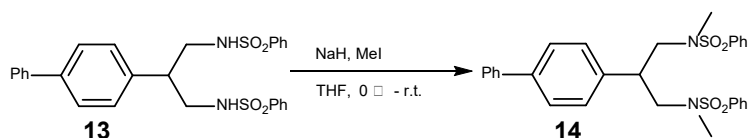
## 5. Reduction and further transformation of product **2aa**

Reduction and deprotection of **2aa**<sup>15</sup>



To a 250 mL round bottom flask equipped with a stir bar, **2aa** was added (500 mg). After sealed by a rubber plug, the flask was then evacuated and backfilled with argon for three times. 20 mL dry THF was then added to the flask. Once completed, 20 mL LiAlH<sub>4</sub> solution (1 M in THF) was added slowly with a syringe in the ice bath condition. The reaction mixture was then moved to room temperature. Monitored by TLC, after the reaction completed, THF/H<sub>2</sub>O = 10/1 and 1 M NaOH solution were added to quench this reaction. Then the mixture was filtered through a short pad of silica gel and the filter cake was washed with ethyl acetate. The filtrate was extracted with ethyl acetate after was added 100 mL water. Then the combined organic phase was concentrated in vacuum after the extracts was washed with brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The resulting residue was purified by silica gel column chromatography with a gradient eluant of petrol ether/ethyl acetate to afford the desired products **13** as a white solid (152 mg, 47% yield).

#### Synthesis of **14**

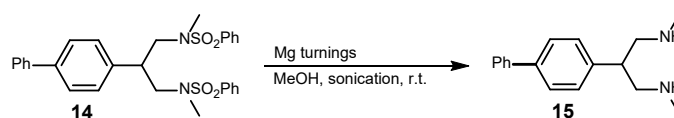


To a 25 mL round bottom flask equipped with a stir bar, **13** was added (20 mg), The round bottom flask was then evacuated and backfilled with argon for three times after sealed by a rubber plug. Once 5 mL THF was added, At Ar atmosphere, NaH (6 eq.) was added in the ice bath condition. After 30 minutes, MeI (3 eq.) was added. The mixture was then moved to room temperature. Monitored by TLC, after the reaction completed, it was quenched by 10 mL water and extracted with ethyl acetate. After washed with brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the combined organic layer was concentrated in vacuum and was purified by silica gel column chromatography with a



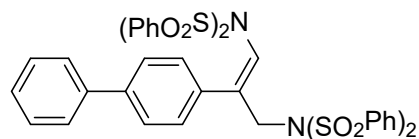
gradient eluant of petrol ether/ethyl acetate to afford the desired products **14** as a colorless viscous liquid (20 mg, 95% yield).

Deprotection of **14**<sup>16-18</sup>



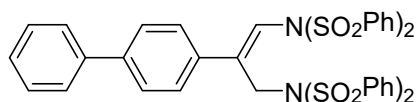
To a 25 mL tube charged with **14** (40 mg), Mg turnings (180 mg 100 eq.) was added. The tube was then evacuated and backfilled with argon for three times after sealed by a rubber plug, 2 mL MeOH was then added. After sonicated for 4 hours at room temperature, 20 mL 1 M HCl was added to the mixture. The aqueous phase was washed with ethyl acetate. Then saturated NaHCO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub> solution was added to the reserved aqueous phase successively, the aqueous phase was washed with ethyl acetate sequentially. At last, NaOH solid was added to tune the aqueous phase PH to 14, and then extracted by ethyl acetate. This last organic layer was reserved and was then concentrated in vacuum after washed by brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> to afford the desired product **15** as a light yellow oil (10 mg, 53% yield).

## 6. Characterization data of isolated products

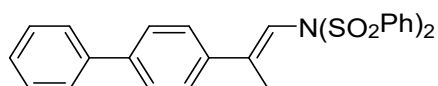


**(E)-N,N'-(2-([1,1'-biphenyl]-4-yl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2aa):** Data for **(E)-2aa**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC: R<sub>f</sub> = 0.17 (n-hexane/EtOAc = 8/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.060 - 8.000 (m, 4H), 7.645 - 7.593 (m, 6H), 7.590 - 7.495 (m, 8H), 7.480 - 7.395 (m, 7H), 7.375 - 7.300 (m, 4H), 5.672 (t, J = 1.6 Hz, 1H), 4.747

(d,  $J = 1.6$  Hz, 2H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  145.827, 141.614, 140.408, 139.000, 138.173, 134.190, 133.901, 133.034, 129.526, 129.198, 129.178, 129.137, 128.771, 127.925, 127.397, 127.113, 119.405, 52.329. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{39}\text{H}_{32}\text{N}_2\text{O}_8\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 807.0934, found 807.0939.

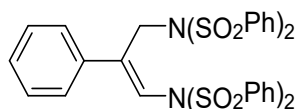


**(Z)-N,N'-(2-([1,1'-biphenyl]-4-yl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2aa):** Data for **(Z)-2aa**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC:  $R_f = 0.15$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.980 (m, 4H), 7.675 - 7.585 (m, 8H), 7.580 - 7.450 (m, 12H), 7.440 - 7.330 (m, 5H), 5.930 (t,  $J = 1.6$  Hz, 1H), 4.975 (d,  $J = 1.6$  Hz, 2H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  147.419, 141.651, 140.394, 138.934, 138.753, 134.498, 134.377, 133.889, 129.200, 129.149, 128.966, 128.867, 128.729, 127.902, 127.231, 127.134, 119.588, 48.062. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{39}\text{H}_{32}\text{N}_2\text{O}_8\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 807.0934, found 807.0939.

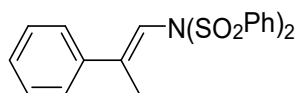


**(E)-N-(2-([1,1'-biphenyl]-4-yl)prop-1-en-1-yl)-N-(phenylsulfonyl) benzenesulfonamide (3aa):** Data for **3aa**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 49 mg (25% yield). White solid. TLC:  $R_f = 0.33$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.060 - 8.005 (m, 4H), 7.708 - 7.652 (m, 2H), 7.625 - 7.545 (m, 8H), 7.500 - 7.435 (m, 4H), 7.405 - 7.350 (m, 1H), 6.185 - 6.144 (m, 1H), 1.760 (d,  $J = 1.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.514, 142.015, 140.416, 139.632, 137.989, 134.110, 129.255, 129.026, 128.408, 127.811, 127.382, 127.1

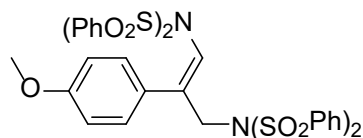
77, 126.977, 117.037, 16.496. HRMS (ESI, M/Z): calcd for C<sub>27</sub>H<sub>23</sub>NO<sub>4</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 512.0961, found 512.0962.



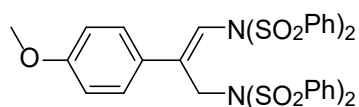
**(Z)-N,N'-(2-phenylprop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2a)**: Data for **(Z)-2a**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White powder. TLC: R<sub>f</sub> = 0.27 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.020 - 7.965 (m, 4H), 7.640 - 7.550 (m, 8H), 7.505 - 7.445 (m, 4H), 7.420 - 7.340 (m, 7H), 7.320 - 7.270 (m, 2H), 5.849 (t, J = 1.6 Hz, 1H), 4.956 (d, J = 1.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 147.780, 138.941, 138.673, 135.609, 134.376, 133.885, 129.184, 128.982, 128.916, 128.751, 128.710, 128.650, 128.599, 119.822, 48.033. HRMS (ESI, M/Z): calcd for C<sub>33</sub>H<sub>28</sub>N<sub>2</sub>O<sub>8</sub>S<sub>4</sub>Na [(M+Na)<sup>+</sup>]: 731.0621, found 731.0627.



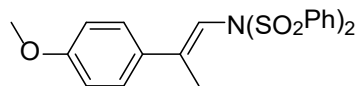
**(E)-N-(2-phenylprop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3a)**<sup>19</sup>: Data for **3a**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 20 mg (12% yield). White solid. TLC: R<sub>f</sub> = 0.34 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.020 (d, J = 8 Hz, 4H), 7.670 (t, J = 7.2 Hz, 2H), 7.600 - 7.530 (m, 4H), 7.419 - 7.357 (m, 5H), 6.092 (s, 1H), 1.723 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.084, 139.643, 139.220, 134.098, 129.246, 129.104, 128.725, 128.410, 126.568, 117.150, 16.614. HRMS (ESI, M/Z): calcd for C<sub>21</sub>H<sub>19</sub>NO<sub>4</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 436.0648, found 436.0653.



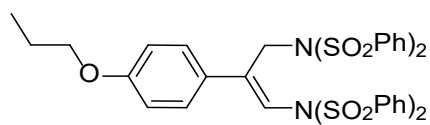
**(E)-N,N'-(2-(4-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2b):** Data for **(E)-2b**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). Yellow solid. TLC:  $R_f = 0.12$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.990 (m, 4H), 7.622 (dd,  $J = 8.4, 1.2$  Hz, 4H), 7.600 - 7.525 (m, 4H), 7.485 - 7.425 (m, 4H), 7.410 - 7.350 (m, 4H), 7.335 - 7.275 (m, 2H), 6.810 - 6.745 (m, 2H), 5.650 - 5.595 (m, 1H), 4.677 (d,  $J = 1.6$  Hz, 2H), 3.830 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  160.130, 145.624, 139.049, 138.271, 134.148, 133.846, 130.345, 129.148, 129.114, 128.763, 128.728, 126.105, 118.976, 114.187, 55.439, 52.420. HRMS (ESI, M/Z): calcd for  $\text{C}_{34}\text{H}_{30}\text{N}_2\text{O}_9\text{S}_4\text{N}$  a [(M+Na) $^+$ ]: 761.0727, found 761.0732.



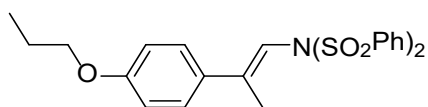
**(Z)-N,N'-(2-(4-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2b):** Data for **(Z)-2b**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). Yellow solid. TLC:  $R_f = 0.08$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.025 - 7.960 (m, 4H), 7.695 - 7.645 (m, 4H), 7.635 - 7.550 (m, 4H), 7.500 - 7.440 (m, 4H), 7.430 - 7.370 (m, 4H), 7.328 - 7.284 (m, 2H), 6.825 - 6.760 (m, 2H), 5.787 (t,  $J = 1.6$  Hz, 1H), 4.948 (d,  $J = 1.6$  Hz, 2H), 3.834 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  160.204, 147.262, 139.067, 138.664, 134.329, 133.842, 129.885, 129.140, 128.989, 128.862, 128.673, 127.782, 118.752, 113.968, 55.473, 48.194. HRMS (ESI, M/Z): calcd for  $\text{C}_{34}\text{H}_{30}\text{N}_2\text{O}_9\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 761.0727, found 761.0732.



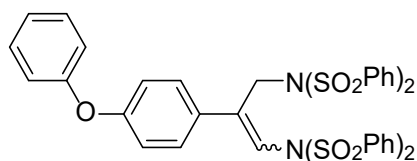
**(E)-N-(2-(4-methoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3b)**<sup>20</sup>: Data for **3b**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 36 mg (20% yield). Yellow solid. TLC:  $R_f$  = 0.32 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.045 - 7.965 (m, 4H), 7.690 - 7.630 (m, 2H), 7.595 - 7.525 (m, 4H), 7.390 - 7.310 (m, 2H), 6.920 - 6.865 (m, 2H), 6.037 (q,  $J$  = 1.2 Hz, 1H), 3.830 (s, 3H), 1.692 (d,  $J$  = 1.2 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  160.442, 149.315, 139.655, 134.040, 131.467, 129.208, 128.400, 127.780, 115.552, 114.058, 55.502, 16.453. HRMS (ESI, M/Z): calcd for C<sub>22</sub>H<sub>21</sub>NO<sub>5</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 466.0754, found 466.0765.



**(Z)-N,N'-(2-(4-propoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2c)**: Data for **(Z)-2c**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White powder. TLC:  $R_f$  = 0.16 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.000 (d,  $J$  = 7.6 Hz, 4H), 7.666 (d,  $J$  = 7.6 Hz, 4H), 7.588 (dt,  $J$  = 14, 7.2 Hz, 4H), 7.505 - 7.443 (m, 4H), 7.426 - 7.366 (m, 4H), 7.310 - 7.270 (m, 2H), 6.820 - 6.730 (m, 2H), 5.800 - 5.745 (m, 1H), 4.940 (d,  $J$  = 1.2 Hz, 2H), 3.935 (t,  $J$  = 6.4 Hz, 2H), 1.905 - 1.790 (m, 2H), 1.145 - 1.030 (m, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  159.827, 147.314, 139.090, 138.702, 134.313, 133.823, 129.859, 129.136, 129.001, 128.855, 128.696, 127.533, 118.625, 114.473, 69.692, 48.194, 22.710, 10.726. HRMS (ESI, M/Z): calcd for C<sub>36</sub>H<sub>34</sub>N<sub>2</sub>O<sub>9</sub>S<sub>4</sub>Na [(M+Na)<sup>+</sup>]: 789.1040, found 789.1050.

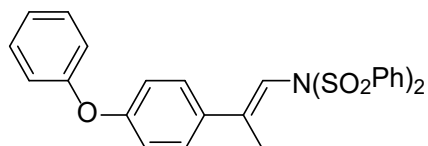


**(E)-N-(phenylsulfonyl)-N-(2-(4-propoxyphenyl)prop-1-en-1-yl)benzenesulfonamide (3c):** Data for **3c**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 42 mg (22% yield). Yellow solid. TLC:  $R_f$  = 0.41 (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.030 - 7.975 (m, 4H), 7.695 - 7.630 (m, 2H), 7.580 - 7.525 (m, 4H), 7.350 - 7.305 (m, 2H), 6.910 - 6.840 (m, 2H), 6.049 - 6.019 (m, 1H), 3.962 - 3.910 (m, 2H), 1.870 - 1.765 (m, 2H), 1.688 (d,  $J$  = 1.2 Hz, 3H), 1.073 - 1.015 (m, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  160.050, 149.368, 139.697, 134.020, 131.249, 129.201, 128.412, 127.746, 115.441, 114.617, 69.766, 22.668, 16.438, 10.631. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{24}\text{H}_{25}\text{NO}_5\text{S}_2\text{Na}$  [( $M+\text{Na}$ ) $^+$ ]: 494.1067, found 494.1073.

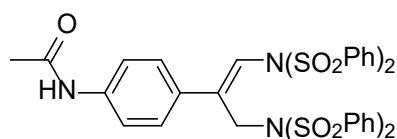


**(E/Z)-N, N'-(2-(4-phenoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E/Z)-2d):** Data for **(E/Z)-2d**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 102 mg (32% yield). Yellow solid. TLC:  $R_f$  = 0.17 (n-hexane/EtOAc = 5/1).  $Z:E$  = 3:1.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.060 - 7.970 (m, 5.52H), 7.740 - 7.690 (m, 4.10H), 7.685 - 7.640 (m, 1.87H), 7.630 - 7.555 (m, 5.30H), 7.515 - 7.435 (m, 9.40H), 7.434 - 7.374 (m, 4.56H), 7.330 - 7.275 (m, 2.84H), 7.220 - 7.145 (m, 1.50H), 7.090 - 7.020 (m, 2.79H), 6.900 - 6.840 (m, 2.70H), 5.824 (t,  $J$  = 1.6 Hz, 1H), 5.680 - 5.650 (m, 0.33H), 4.974 (d,  $J$  = 1.2 Hz, 2H), 4.680 (d,  $J$  = 1.2 Hz, 0.68H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.201, 158.172, 156.352, 156.171, 146.976, 145.633, 139.107, 138.601, 138.200, 134.395, 134.204, 133.967, 133.928, 130.535, 130.083, 130.059, 129.996, 129.946, 129.166, 1

29.096, 129.030, 128.937, 128.816, 128.755, 128.677, 128.273, 124.200, 124.109, 119.694, 119.611, 119.425, 119.224, 118.116, 118.083, 52.427, 48.140. HRMS (ESI, M/Z): calcd for C<sub>39</sub>H<sub>32</sub>N<sub>2</sub>O<sub>9</sub>S<sub>4</sub>Na [(M+Na)<sup>+</sup>]: 823.0883, found 823.0892.

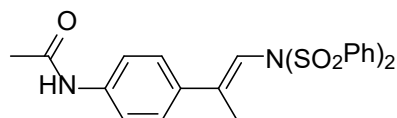


**(E)-N-(2-(4-phenoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3d):** Data for **3d**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 34 mg (17% yield). Light yellow solid. TLC: R<sub>f</sub> = 0.43 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.045 - 7.980 (m, 4H), 7.700 - 7.640 (m, 2H), 7.590 - 7.530 (m, 4H), 7.400 - 7.330 (m, 4H), 7.175 - 7.115 (m, 1H), 7.060 - 7.010 (m, 2H), 7.005 - 6.960 (m, 2H), 6.080 - 6.040 (m, 1H), 1.711 (d, J = 1.2 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 158.396, 156.746, 149.193, 139.665, 134.092, 133.854, 130.027, 129.244, 128.418, 127.998, 123.939, 119.446, 118.588, 116.424, 16.557. HRMS (ESI, M/Z): calcd for C<sub>27</sub>H<sub>23</sub>NO<sub>5</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 528.0910, found 528.0916.

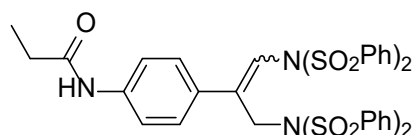


**(Z)-N-(4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)acetamide ((Z)-2e):** Data for **(Z)-2e**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 1.5/1). White powder. TLC: R<sub>f</sub> = 0.36 (n-hexane/EtOAc = 1/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.990 - 7.940 (m, 4H), 7.720 - 7.665 (m, 4H), 7.660 - 7.530 (m, 5H), 7.500 - 7.445 (m, 4H), 7.440 - 7.330 (m, 7H), 7.279 (s, 1H), 5.777 (s, 1H), 5.000 (d,

$J = 1.2$  Hz, 2H), 2.191 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.609, 147.123, 138.963, 138.599, 138.407, 134.451, 134.016, 131.097, 129.193, 129.127, 128.990, 128.977, 128.598, 119.772, 119.601, 48.149, 24.714. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{35}\text{H}_{31}\text{N}_3\text{O}_9\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 788.0836, found 788.0826.



**(*E*)-N-(4-(1-(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)acetamide (**3e**)**<sup>20</sup>: Data for **3e**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 2/1) afforded 52 mg (28% yield). White powder. TLC:  $R_f = 0.49$  (n-hexane/EtOAc = 1/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.022 - 7.950 (m, 4H), 7.700 - 7.625 (m, 2H), 7.588 - 7.528 (m, 4H), 7.504 (d,  $J = 8.8$  Hz, 2H), 7.335 (d,  $J = 8.4$  Hz, 2H), 7.306 (s, 1H), 6.055 (d,  $J = 1.2$  Hz, 1H), 2.173 (s, 3H), 1.698 (d,  $J = 1.6$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.500, 149.238, 139.527, 138.793, 134.761, 134.150, 129.265, 128.389, 127.209, 119.784, 116.429, 24.767, 16.380. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_5\text{S}_2\text{Na}$   $[(M+\text{Na})^+]$ : 493.0863, found 493.0860.

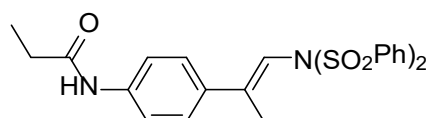


**(*E/Z*)-N-(4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)propionamide ((*E/Z*)-**2f**)**: Data for (*E/Z*)-**2f**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 2/1) afforded 175 mg (56% yield). White solid. TLC:  $R_f = 0.06$  (n-hexane/EtOAc = 2/1).  $Z:E = 2:1$   $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.040 - 7.940 (m, 5.81H), 7.740 - 7.670 (m, 4.15H), 7.660 - 7.525 (m, 7.87H), 7.505 - 7.435 (m, 5.98H), 7.435 - 7.340 (m, 8.82H), 7.265 (s, 1.62H), 7.244 (s, 1.35H), 7.191 (s, 1.45H), 5.772 (s, 1H), 5.696 (s, 0.44H), 5.011 (d,  $J = 1.2$  Hz, 2H), 4.660 (d,  $J = 1.2$  Hz, 0.85H), 2.419



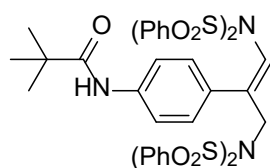
(qd,  $J = 7.6, 2.4$  Hz, 2.98H), 1.275 (td,  $J = 7.6, 2.8$  Hz, 4.89H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.348, 172.337, 147.115, 147.097, 145.698, 145.688, 138.838, 138.759, 138.727, 138.703, 138.254, 137.950, 134.378, 134.183, 133.949, 130.772, 129.442, 129.106, 128.991, 128.902, 128.851, 128.775, 128.592, 128.486, 119.701, 119.567, 119.452, 119.115, 52.255, 48.062, 30.675, 9.716, 9.674.

HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{36}\text{H}_{33}\text{N}_3\text{O}_9\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 802.0992, found 802.0993.



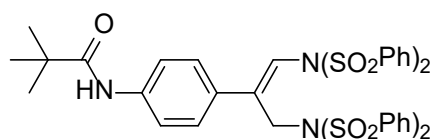
**(E)-N-(4-(1-(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)propionamide (3f):** Data for **3f**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 2/1) afforded 40 mg (21% yield). White solid.

TLC:  $R_f = 0.11$  (n-hexane/EtOAc = 2/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.025 - 7.960 (m, 4H), 7.700 - 7.625 (m, 2H), 7.585 - 7.500 (m, 6H), 7.365 - 7.310 (m, 2H), 7.213 (s, 1H), 6.057 (d,  $J = 1.2$  Hz, 1H), 2.399 (q,  $J = 7.6$  Hz, 2H), 1.699 (d,  $J = 1.6$  Hz, 3H), 1.276 - 1.228 (m, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.213, 149.252, 139.522, 138.901, 134.585, 134.138, 129.253, 128.382, 127.193, 119.673, 116.354, 30.852, 16.372, 9.690. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_5\text{S}_2\text{Na}$   $[(M+\text{Na})^+]$ : 507.1019, found 507.1021.

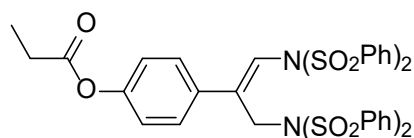


**(E)-N-(4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)propionamide ((E)-2g):** Data for **(E)-2g**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White solid. TLC:  $R_f = 0.30$  (n-hexane/EtOAc = 2/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.980 (m, 4H), 7.665 - 7.615 (m, 4H), 7.605 - 7.515 (m, 4H), 7.495 - 7.4

35 (m, 4H), 7.410 - 7.345 (m, 6H), 7.302 (s, 1H), 7.249 (d,  $J = 9.2$  Hz, 2H), 5.712 (s, 1H), 4.658 (d,  $J = 1.6$  Hz, 2H), 1.362 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  176.671, 145.517, 138.973, 138.661, 138.232, 134.210, 133.901, 129.649, 129.489, 129.194, 129.022, 128.832, 128.763, 119.989, 119.546, 52.295, 39.853, 27.791. HRMS (ESI, M/Z): calcd for  $\text{C}_{38}\text{H}_{37}\text{N}_3\text{O}_9\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 830.1305, found 830.1305.

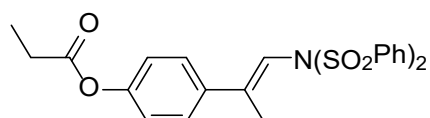


**(Z)-N-(4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)pi valamide ((Z)-2g):** Data for **(Z)-2g**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White solid. TLC:  $R_f = 0.24$  (n-hexane/EtOAc = 2/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.004 - 7.966 (m, 4H), 7.745 - 7.695 (m, 4H), 7.640 - 7.560 (m, 4H), 7.474 (t,  $J = 8$  Hz, 4H), 7.409 (t,  $J = 8.4$  Hz, 6H), 7.322 (s, 1H), 7.248 (d,  $J = 10$  Hz, 2H), 5.780 - 5.740 (m, 1H), 5.024 (d,  $J = 1.6$  Hz, 2H), 1.352 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  176.758, 146.997, 139.226, 138.616, 138.510, 134.413, 133.916, 131.141, 129.172, 129.115, 129.080, 128.977, 128.631, 119.857, 119.728, 48.293, 39.854, 27.787. HRMS (ESI, M/Z): calcd for  $\text{C}_{38}\text{H}_{37}\text{N}_3\text{O}_9\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 830.1305, found 830.1305.

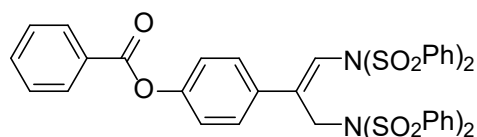


**(Z)-4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl prop ionate ((Z)-2h):** Data for **(Z)-2h**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White powder. TLC:  $R_f = 0.11$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98

5 (d,  $J = 7.6$  Hz, 4H), 7.656 (d,  $J = 7.6$  Hz, 4H), 7.625 - 7.560 (m, 4H), 7.510 - 7.420 (m, 8H), 7.387(d,  $J = 8.4$  Hz, 2H), 7.013 (d,  $J = 8.4$  Hz, 2H), 5.833 (s, 1H), 4.949 (d,  $J = 1.2$  Hz, 2H), 2.637 (q,  $J = 7.6$  Hz, 2H), 1.311 (t,  $J = 7.6$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.886, 151.343, 146.941, 138.821, 138.583, 134.413, 134.001, 132.949, 129.697, 129.213, 129.029, 128.956, 128.673, 121.858, 119.972, 47.873, 27.906, 9.225. HRMS (ESI, M/Z): calcd for  $\text{C}_{36}\text{H}_{32}\text{N}_2\text{O}_{10}\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 803.0832, found 803.0817.

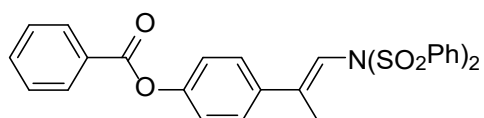


**(E)-4-(1-(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl propionate (3h):** Data for **3h**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 22 mg (12% yield). White solid. TLC:  $R_f = 0.27$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.025 - 7.970 (m, 4H), 7.670 (tt,  $J = 6.8, 1.2$  Hz, 2H), 7.590 - 7.530 (m, 4H), 7.397 (dt,  $J = 8.8, 2.8$  Hz, 2H), 7.089 (dt,  $J = 8.8, 2.8$  Hz, 2H), 6.072 (d,  $J = 1.2$  Hz, 1H), 2.608 (q,  $J = 7.6$  Hz, 2H), 1.707 (d,  $J = 1.2$  Hz, 3H), 1.278 (t,  $J = 7.6$  Hz, 4H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  173.009, 151.417, 149.136, 139.581, 136.702, 134.130, 129.270, 128.401, 127.671, 121.903, 117.28, 27.890, 16.610, 9.182. HRMS (ESI, M/Z): calcd for  $\text{C}_{24}\text{H}_{23}\text{NO}_6\text{S}_2\text{Na}$  [(M+Na) $^+$ ]: 508.0859, found 508.0862.



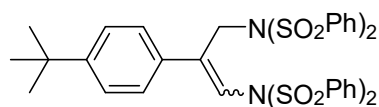
**(Z)-4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl benzoate ((Z)-2i):** Data for **(Z)-2i**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White powder. TLC:  $R_f = 0.12$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.270

- 8.210 (m, 2H), 8.025 - 7.970 (m, 4H), 7.705 - 7.670 (m, 4H), 7.655 - 7.535 (m, 7H), 7.520 - 7.465 (m, 7H), 7.460 - 7.415 (m, 3H), 7.180 - 7.120 (m, 2 H), 5.862 (t,  $J = 1.6$  Hz, 1H), 4.984 (d,  $J = 1.6$  Hz, 2H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.080, 151.523, 146.979, 138.880, 138.588, 134.435, 134.028, 133.961, 133.143, 130.362, 129.798, 129.468, 129.235, 129.075, 128.985, 128.831, 128.702, 121.987, 120.079, 47.957. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{40}\text{H}_{32}\text{N}_2\text{O}_{10}\text{S}_4\text{Na}$  [ $(M+\text{Na})^+$ ]: 851.0832, found 851.0829.



**(*E*)-4-(1-(*N*-(phenylsulfonyl)phenylsulfonyl)prop-1-en-2-yl)phenyl benzoate**

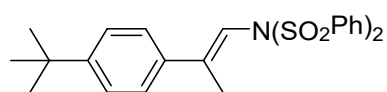
**(3i):** Data for **3i**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 25 mg (12% yield). Light yellow solid. TLC:  $R_f = 0.28$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.232 - 8.194 (m, 2H), 8.045 - 7.990 (m, 4H), 7.705 - 7.635 (m, 3H), 7.595 - 7.510 (m, 6H), 7.480 - 7.435 (m, 2H), 7.250 - 7.208 (m, 2H), 6.106 (d,  $J = 1.2$  Hz, 1H), 1.739 (d,  $J = 1.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.229, 151.626, 149.159, 139.607, 136.908, 134.138, 133.926, 130.374, 129.441, 129.281, 128.779, 128.419, 127.767, 122.074, 117.390, 16.651. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{28}\text{H}_{23}\text{NO}_6\text{S}_2\text{Na}$  [ $(M+\text{Na})^+$ ]: 556.0859, found 556.0865.



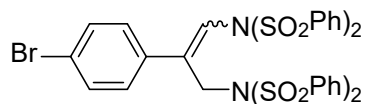
**(*E/Z*)-*N, N'*-(2-(4-(tert-butyl)phenyl)prop-1-ene-1,3-diyl)bis(*N*-(phenylsulfonyl)benzenesulfonamide) ((*E/Z*)-2j):**

Data for **(*E/Z*)-2j**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 165 mg (54% yield). White powder. TLC:  $R_f = 0.27$  (n-hexane/EtOAc = 5/1).  $Z:E = 2:1$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.960 (m, 6.20H), 7.650 - 7.515 (m, 12.66H), 7.505 - 7.420 (m, 6.44H), 7.415 - 7.295 (m, 12.75H), 5.874 (t,  $J$

= 1.6 Hz, 1H), 5.563 (t,  $J$  = 1.6 Hz, 0.54H), 4.922 (d,  $J$  = 1.6 Hz, 2H), 4.672 (d,  $J$  = 1.6 Hz, 1H), 1.373 (d,  $J$  = 2.0 Hz, 13.98H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.035, 151.839, 147.521, 146.515, 138.941, 138.909, 138.776, 138.118, 134.298, 134.175, 133.861, 132.487, 131.193, 129.160, 129.150, 128.946, 128.836, 128.767, 128.727, 128.625, 128.215, 125.629, 125.480, 119.101, 118.273, 52.523, 47.845, 34.858, 34.845, 31.593, 31.559. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{37}\text{H}_{36}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [ $(M+\text{Na})^+$ ]: 787.1247, found 787.1252.

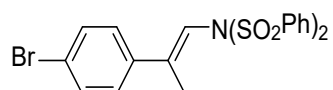


**(*E*)-*N*-(2-(4-(*tert*-butyl)phenyl)prop-1-en-1-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (3j):** Data for **3j**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 22 mg (12% yield). Light yellow solid. TLC:  $R_f$  = 0.44 (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.040 - 7.980 (m, 4H), 7.690 - 7.630 (m, 2H), 7.590 - 7.515 (m, 4H), 7.420 - 7.370 (m, 2H), 7.365 - 7.315 (m, 2H), 6.095 (d,  $J$  = 1.6 Hz, 1H), 1.702 (d,  $J$  = 1.2 Hz, 3H), 1.333 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.425, 149.708, 139.684, 136.212, 134.044, 129.224, 128.405, 126.274, 125.640, 116.485, 34.812, 31.383, 16.435. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{25}\text{H}_{27}\text{NO}_4\text{S}_2\text{Na}$  [ $(M+\text{Na})^+$ ]: 492.1274, found 492.1273.

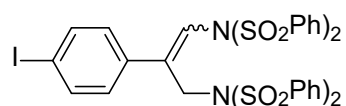


**(*E/Z*)-*N, N'*-(2-(4-bromophenyl)prop-1-ene-1,3-diyl)bis(*N*-(phenylsulfonyl)benzenesulfonamide) ((*E/Z*)-2k):** Data for **(*E/Z*)-2k**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 118 mg (37% yield). White powder. TLC:  $R_f$  = 0.26 (n-hexane/EtOAc = 5/1).  $Z:E$  = 3:2.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.025 - 7.945 (m, 6.95H), 7.695 - 7.560 (m, 14.27H), 7.510 - 7.330 (m, 17.87H), 7.235 - 7.165 (m, 4H), 5.812 (t,  $J$  = 1.6

Hz, 1H), 5.730 (s, 0.71H), 4.951 (d,  $J = 1.6$  Hz, 2H), 4.670 (d,  $J = 1.6$  Hz, 1.45H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  146.752, 145.995, 138.946, 138.926, 138.526, 138.109, 134.490, 134.416, 134.259, 134.065, 134.007, 132.756, 131.923, 131.745, 130.749, 130.243, 129.222, 128.997, 128.901, 128.722, 128.610, 123.326, 123.234, 120.256, 120.199, 52.069, 48.087. HRMS (ESI, M/Z): calcd for  $\text{C}_{33}\text{H}_{27}\text{BrN}_2\text{O}_8\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 808.9726, found 808.9718.

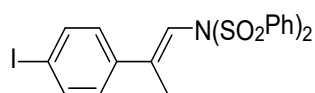


**(E)-N-(2-(4-bromophenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3k):** Data for **3k**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 29 mg (15% yield). White powder. TLC:  $R_f = 0.53$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.024 - 7.970 (m, 4H), 7.700 - 7.640 (m, 2H), 7.600 - 7.530 (m, 4H), 7.520 - 7.470 (m, 2H), 7.265 - 7.230 (m, 2H), 6.076 (d,  $J = 1.2$  Hz, 1H), 1.703 (d,  $J = 1.6$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  148.937, 139.531, 138.046, 134.188, 131.893, 129.283, 128.389, 128.116, 123.305, 117.580, 16.504. HRMS (ESI, M/Z): calcd for  $\text{C}_{21}\text{H}_{18}\text{BrNO}_4\text{S}_2\text{Na}$  [(M+Na) $^+$ ]: 513.9753, found 513.9763.



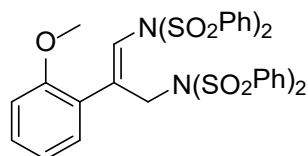
**(E/Z)-N,N'-(2-(4-iodophenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E/Z)-2l):** Data for **(E/Z)-2l**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 140 mg (42% yield). White powder. TLC:  $R_f = 0.27$  (n-hexane/EtOAc = 5/1). Z:E = 2:1.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.020 - 7.930 (m, 6.17H), 7.680 - 7.535 (m, 15.64H), 7.508 - 7.404 (m, 12.50H), 7.120 - 7.040 (m, 3.10H), 5.816 (t,  $J = 1.6$  Hz, 1H), 5.716 (s, 0.50H), 4.940 (d,  $J = 1.6$  Hz, 2H), 4.665 (d,  $J = 1.6$  Hz, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  146.885, 145.086, 138.913, 138.541, 138.

103, 137.878, 137.715, 135.031, 134.480, 134.257, 134.056, 134.008, 133.377, 130.861, 130.398, 129.223, 129.010, 128.975, 128.929, 128.721, 128.615, 120.206, 120.111, 95.215, 95.015, 52.007, 48.017. HRMS (ESI, M/Z): calcd for  $C_3H_3IN_2O_8S_4Na$  [(M+Na)<sup>+</sup>]: 856.9588, found 856.9586.



**(E)-N-(2-(4-iodophenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide**

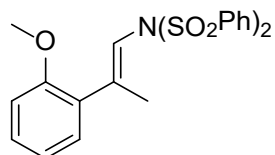
**(3I):** Data for **3I**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 20 mg (10% yield). Light yellow solid. TLC:  $R_f = 0.50$  (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.020 - 7.970 (m, 4H), 7.715 - 7.645 (m, 4H), 7.590 - 7.535 (m, 4H), 7.140 - 7.095 (m, 2H), 6.076 (q,  $J = 1.2$  Hz, 1H), 1.693 (d,  $J = 1.6$  Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  149.036, 139.549, 138.668, 137.873, 134.177, 129.275, 128.393, 128.273, 117.602, 94.970, 16.425. HRMS (ESI, M/Z): calcd for  $C_{21}H_{18}INO_4S_2Na$  [(M+Na)<sup>+</sup>]: 561.9615, found 561.9619.



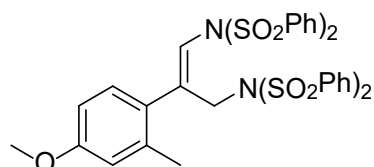
**(Z)-N,N'-(2-(2-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2m):**

Data for **(Z)-2m**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White solid. TLC:  $R_f = 0.44$  (n-hexane/EtOAc = 3/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.974 (d,  $J = 7.6$  Hz, 4H), 7.708 - 7.637 (m, 4H), 7.625 - 7.550 (m, 4H), 7.476 - 7.370 (m, 8H), 7.331 (td,  $J = 8, 1.6$  Hz, 1H), 6.964 (dd,  $J = 7.2, 1.2$  Hz, 1H), 6.860 - 6.775 (m, 2H), 5.678 (s, 1H), 5.000 (d,  $J = 1.2$  Hz, 2H), 3.898 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  157.849, 146.057, 139.162, 138.256, 134.264, 133.698, 131.601, 130.118, 129.195, 128.975, 128.898, 128.706,

125.056, 120.673, 120.493, 110.245, 55.364, 47.962. HRMS (ESI, M/Z): calcd for  $C_{34}H_{30}N_2O_9S_4H [(M+H)^+]$ : 739.0907, found 739.0910.



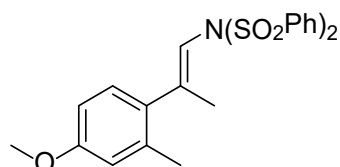
**(E)-N-(2-(2-methoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3m):** Data for **3m**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 30 mg (17% yield). Light yellow viscous liquid. TLC:  $R_f = 0.25$  (n-hexane/EtOAc = 5/1).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.085 – 7.970 (m, 4H), 7.659 (t,  $J = 7.6$  Hz, 2H), 7.585 – 7.525 (m, 4H), 7.305 (td,  $J = 8.0, 1.6$  Hz, 1H), 7.168 (dd,  $J = 8.0, 1.6$  Hz, 1H), 6.990 – 6.855 (m, 2H), 5.918 (d,  $J = 1.6$  Hz, 1H), 3.837 (s, 3H), 1.640 (d,  $J = 1.2$  Hz, 3H).  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  156.963, 150.058, 139.770, 133.947, 129.769, 129.546, 129.380, 129.117, 128.466, 120.679, 118.799, 111.310, 55.700, 17.539. HRMS (ESI, M/Z): calcd for  $C_{22}H_{21}NO_5S_2H [(M+H)^+]$ : 444.0934, found 444.0940.



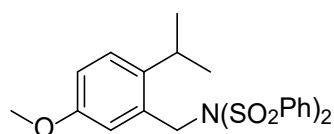
**(Z)-N, N'-(2-(4-methoxy-2-methylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2n):** Data for **(Z)-2n**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 3/1) afforded 151 mg (50% yield). White solid. TLC:  $R_f = 0.5$  (n-hexane/EtOAc = 3/1).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.975 – 7.881 (m, 4H), 7.657 – 7.512 (m, 8H), 7.475 – 7.420 (m, 4H), 7.414 – 7.354 (m, 4H), 7.281 – 7.244 (m, 1H), 6.735 – 6.614 (m, 2H), 5.685 (t,  $J = 1.6$  Hz, 1H), 4.781 (d,  $J = 1.6$  Hz, 2H), 3.830 (s, 3H), 2.405 (s, 3H).  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  159.836, 148.132, 139.178, 1



38.550, 138.509, 134.360, 133.750, 132.024, 129.190, 128.834, 128.782, 127.617, 120.668, 115.861, 111.126, 55.301, 47.974, 20.016. HRMS (ESI, M/Z): calcd for C<sub>35</sub>H<sub>32</sub>N<sub>2</sub>O<sub>9</sub>S<sub>4</sub>H [(M+H)<sup>+</sup>]: 753.1064, found 753.1073.

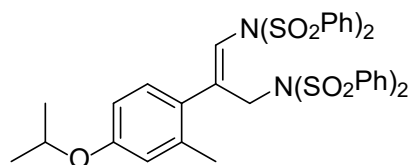


**(E)-N-(2-(4-methoxy-2-methylphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3n):** Data for **3n**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 29 mg (16% yield). Light yellow viscous liquid. TLC: R<sub>f</sub> = 0.27 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.085 – 7.972 (m, 4H), 7.705 – 7.627 (m, 2H), 7.600 – 7.508 (m, 4H), 6.990 (d, J = 8.4 Hz, 1H), 6.770 – 6.664 (m, 2H), 5.697 (d, J = 1.6 Hz, 1H), 3.797 (s, 3H), 2.303 (s, 3H), 1.613 (d, J = 1.6 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 159.387, 151.908, 139.697, 136.863, 134.073, 132.594, 129.223, 129.033, 128.390, 118.836, 115.939, 111.151, 55.391, 20.089, 19.149. HRMS (ESI, M/Z): calcd for C<sub>23</sub>H<sub>23</sub>NO<sub>5</sub>S<sub>2</sub>H [(M+H)<sup>+</sup>]: 458.1091, found 458.1098.

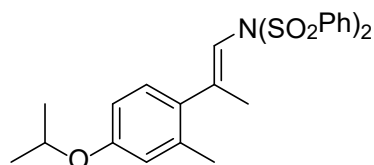


**N-(2-isopropyl-5-methoxybenzyl)-N-(phenylsulfonyl)benzenesulfonamide (4n):** Data for **4n**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 44 mg (24% yield). White solid. TLC: R<sub>f</sub> = 0.38 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.875 – 7.785 (m, 4H), 7.639 – 7.545 (m, 2H), 7.500 – 7.398 (m, 4H), 7.154 (d, J = 8.8 Hz, 1H), 6.774 – 6.662 (m, 2H), 5.081 (s, 2H), 3.450 (s, 3H), 3.192 (hept, J = 6.8 Hz, 1H), 1.199 (d, J = 6.8 Hz, 6H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 157.460,

140.177, 138.811, 133.766, 131.878, 128.923, 128.334, 126.378, 115.054, 112.906, 55.003, 49.390, 27.943, 23.949. HRMS (ESI, M/Z): calcd for C<sub>23</sub>H<sub>25</sub>NO<sub>5</sub>S<sub>2</sub>H [(M+H)<sup>+</sup>]: 460.1247, found 460.1254.

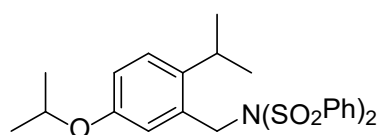


**(Z)-N, N'-(2-(4-isopropoxy-2-methylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2o):** Data for **(Z)-2o**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 3/1) afforded 165 mg (53% yield). White solid. TLC: R<sub>f</sub> = 0.57 (n-hexane/EtOAc = 3/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.983 - 7.880 (m, 4H), 7.665 - 7.513 (m, 8H), 7.498 - 7.340 (m, 8H), 7.241 (d, J = 8.4 Hz, 1H), 6.696 (d, J = 2.4 Hz, 1H), 6.631 (dd, J = 8.4, 2.8 Hz, 1H), 5.689 (t, J = 1.6 Hz, 1H), 4.768 (d, J = 1.2 Hz, 2H), 4.553 (hept, J = 6 Hz, 1H), 2.392 (s, 3H), 1.394 (d, J = 6.4 Hz, 6 H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 158.287, 148.164, 139.221, 138.543, 134.342, 133.746, 132.029, 129.191, 128.888, 128.831, 128.785, 127.186, 120.624, 117.370, 112.393, 69.624, 47.853, 22.343, 20.033. HRMS (ESI, M/Z): calcd for C<sub>37</sub>H<sub>36</sub>N<sub>2</sub>O<sub>9</sub>S<sub>4</sub>H [(M+H)<sup>+</sup>]: 781.1377, found 781.1382.



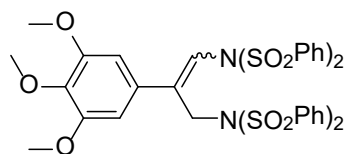
**(E)-N-(2-(4-isopropoxy-2-methylphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3o):** Data for **3o**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 31 mg (16% yield). Colorless viscous liquid. TLC: R<sub>f</sub> = 0.36 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.062 - 7.989 (m, 4H), 7.714 - 7.617 (m, 2H), 7.617 - 7.50

4 (m, 4H), 6.966 (d,  $J = 8.4$  Hz, 1H), 6.750 – 6.646 (m, 2H), 5.700 (q,  $J = 1.2$  Hz, 1H), 4.532 (hept,  $J = 6$  Hz, 1H), 2.283 (s, 3H), 1.608 (d,  $J = 1.6$  Hz, 3H), 1.333 (d,  $J = 6$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  157.716, 151.984, 139.713, 136.828, 134.056, 132.331, 129.215, 129.023, 128.390, 118.756, 117.838, 112.782, 69.914, 22.225, 20.094, 19.133. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{25}\text{H}_{27}\text{NO}_5\text{S}_2\text{H}$  [( $M+H$ ) $^+$ ]: 486.1404, found 486.1331.



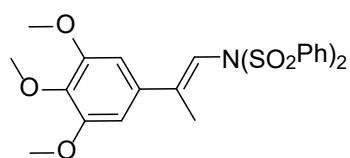
**N-(5-isopropoxy-2-isopropylbenzyl)-N-(phenylsulfonyl)benzenesulfonamide (4o):**

Data for **4o**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 43 mg (22% yield). White solid. TLC:  $R_f = 0.42$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.856 – 7.780 (m, 4H), 7.632 – 7.538 (m, 2H), 7.500 – 7.392 (m, 4H), 7.135 (d,  $J = 8.4$  Hz, 1H), 6.755 – 6.664 (m, 2H), 5.063 (s, 2H), 4.068 (hept,  $J = 6$  Hz, 1H), 3.199 (hept,  $J = 6.8$  Hz, 1H), 1.191 (d,  $J = 6.8$  Hz, 6H), 1.156 (d,  $J = 6$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  155.871, 140.177, 138.484, 133.728, 131.869, 128.917, 128.319, 126.357, 116.354, 114.816, 69.398, 49.388, 27.894, 23.988, 22.056. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{25}\text{H}_{29}\text{NO}_5\text{S}_2\text{H}$  [( $M+H$ ) $^+$ ]: 488.1560, found 488.1567.

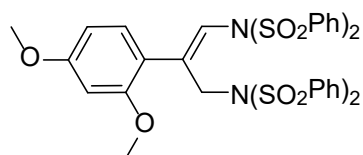


**(*E/Z*)-N,N'-(2-(3,4,5-trimethoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide)((*E/Z*)-2p):** Data for (*E/Z*)-**2p**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1) to afford 156 mg (49% yield) of (*E/Z*)-**2p**. Yellow powder. TLC:  $R_f = 0.16$  (n-hexane/EtOAc = 5/1).  $Z:E = 1:3$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.055 –

7.970 (m, 5.11H), 7.753 – 7.665 (m, 5.22H), 7.662 – 7.530 (m, 5.63H), 7.530 - 7.334 (m, 11.07H), 6.691 (s, 1.89H), 6.620 (s, 0.70H), 5.875 (s, 0.36H), 5.718 (s, 1H), 5.000 (s, 0.70H), 4.754 (s, 1.95H), 3.925 - 3.850 (m, 4H), 3.809 (s, 2.11H), 3.700 (s, 5.89H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.201, 147.714, 145.068, 139.125, 139.074, 138.625, 138.560, 138.318, 138.271, 134.425, 1344.122, 134.036, 133.948, 130.624, 129.154, 129.082, 129.049, 128.849, 128.768, 128.608, 128.471, 128.372, 119.734, 119.355, 106.117, 105.577, 61.022, 60.923, 56.420, 56.296, 52.130, 48.061. HRMS (ESI, M/Z): calcd for  $\text{C}_{36}\text{H}_{34}\text{N}_2\text{O}_{11}\text{S}_4$  H [(M+H) $^+$ ]: 799.1119, found 799.1120.

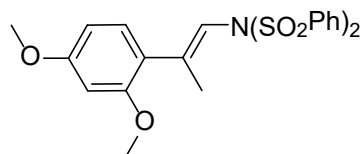


**(E)-N-(phenylsulfonyl)-N-(2-(3,4,5-trimethoxyphenyl)prop-1-en-1-yl)benzenesulfonamide(3p):** Data for **3p**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 5/1) to afford 16 mg (8% yield) of **3p**. Yellow solid. TLC:  $R_f$  = 0.3 (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.045 – 7.980 (m, 4H), 7.710 – 7.638 (m, 2H), 7.589 - 7.540 (m, 4H), 6.574 (s, 2H), 6.04 (d,  $J$  = 1.4 Hz, 1H), 3.880 (s, 6H), 3.864 (s, 3H), 1.736 (d,  $J$  = 1.2 Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.349, 149.946, 139.602, 139.123, 134.887, 134.144, 129.244, 128.446, 116.838, 104.007, 61.074, 56.424, 16.869. HRMS (ESI, M/Z): calcd for  $\text{C}_{24}\text{H}_{25}\text{NO}_7\text{S}_2\text{H}$  [(M+H) $^+$ ]: 504.1146, found 504.1150.

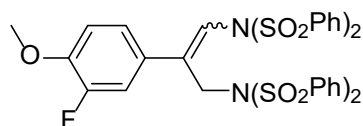


**(Z)-N,N'-(2-(2,4-dimethoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide)((Z)-2q):** Data for **(Z)-2q**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). Yellow solid. TLC:  $R_f$  = 0.39 (n-hexane/EtOAc = 3/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.000 - 7.930 (m, 4H), 7.745 - 7.675 (m, 4H), 7.630 – 7.545 (m, 4H), 7.480 – 7.365 (m, 8H), 6.908 (d,  $J$  = 8 Hz, 1H), 6.370 – 6.275 (m, 2H), 5.638

(t,  $J = 1.2$  Hz, 1H), 4.958 (d,  $J = 1.2$  Hz, 2H), 3.848 (s, 3H), 3.824 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  161.563, 158.765, 145.870, 139.361, 138.384, 134.228, 133.633, 132.124, 129.221, 128.970, 128.861, 128.687, 120.564, 117.668, 104.054, 98.298, 55.518, 55.390, 48.372. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{35}\text{H}_{32}\text{N}_2\text{O}_{10}\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 791.0832, found 791.0825.

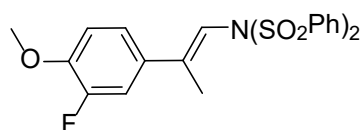


**(E)-N-(2-(2,4-dimethoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide(3q):** Data for **3q**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 10/1) to afford 15 mg (8% yield) of **3q**. Yellow solid. TLC:  $R_f = 0.14$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.070 – 8.000 (m, 4H), 7.695 – 7.610 (m, 2H), 7.600 – 7.495 (m, 4H), 7.135 – 7.060 (m, 1H), 6.505 – 6.420 (m, 2H), 5.920 (d,  $J = 1.2$  Hz, 1H), 3.817 (s, 3H), 3.805 (s, 3H), 1.625 (d,  $J = 1.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  161.254, 158.197, 149.506, 139.794, 133.908, 130.162, 129.095, 128.468, 122.069, 118.222, 104.301, 99.202, 55.678, 55.571, 17.566. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{23}\text{H}_{23}\text{NO}_6\text{S}_2\text{H}$   $[(M+\text{H})^+]$ : 474.1040, found 474.1043.

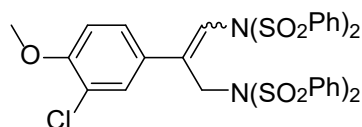


**(E/Z)-N,N'-(2-(3-fluoro-4-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide)((E/Z)-2r):** Data for **(E/Z)-2r**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1) to afford 194 mg (64% yield) of **(E/Z)-2r**. White powder. TLC:  $R_f = 0.32$  (n-hexane/EtOAc = 3/1).  $Z:E = 5:1$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.076 – 7.902 (m, 4.85H), 7.807 – 7.728 (m, 3.89H), 7.725 – 7.665 (m, 1.06H), 7.665 – 7.545 (m, 4.94H), 7.540 – 7.360 (m, 9.80H), 7.235 – 7.098 (m, 1.29H), 6.922 – 6.749 (m, 2.44H), 5.810 – 5.705 (m, 1.21H), 5.028 (d,  $J = 0.8$  Hz, 2H), 4.648 (s, 0.43H), 3.930 – 3.855 (m, 3.57H).  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -

134.225, -134.368.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.045, 153.010, 150.597, 150.557, 148.188 (d,  $J = 10.0$  Hz), 148.040, 145.862 (d,  $J = 1.0$  Hz), 144.349, 139.238, 138.955, 138.367, 138.277, 134.465, 134.209, 134.004, 133.948, 129.151, 129.055, 128.922, 128.868, 128.805, 128.652, 128.477, 127.994 (d,  $J = 6.7$  Hz), 126.141 (d,  $J = 6.9$  Hz), 125.289 (d,  $J = 3.4$  Hz), 124.553 (d,  $J = 3.3$  Hz), 120.117, 119.983, 116.719 (d,  $J = 19.4$  Hz), 116.002 (d,  $J = 19.4$  Hz), 113.017 (d,  $J = 1.2$  Hz), 112.924 (d,  $J = 1.3$  Hz), 56.296, 56.238, 52.160, 48.187. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{34}\text{H}_{29}\text{FN}_2\text{O}_9\text{S}_4\text{Na}$   $[(M+\text{Na})^+]$ : 779.0633, found 779.0635.

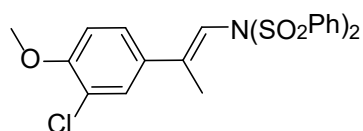


**(E)-N-(2-(3-fluoro-4-methoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide(3r):** Data for **3r**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 10/1) to afford 48 mg (26% yield) of **3r**. White solid. TLC:  $R_f = 0.16$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.040 - 7.950 (m, 4H), 7.715 - 7.638 (m, 2H), 7.595 - 7.520 (m, 4H), 7.185 - 7.090 (m, 2H), 6.937 (t,  $J = 8.8$  Hz, 1H), 6.041 (q,  $J = 1.6$  Hz, 1H), 3.909 (s, 3H), 1.683 (d,  $J = 1.6$  Hz, 3H).  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -134.618.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.464, 151.016, 148.370 (dd,  $J = 10.9, 6.4$  Hz), 139.523, 134.137, 131.962 (d,  $J = 6.5$  Hz), 129.248, 128.365, 122.508 (d,  $J = 3.4$  Hz), 116.503, 114.269 (d,  $J = 19.4$  Hz), 113.276 (d,  $J = 2.3$  Hz), 56.428, 16.359. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{22}\text{H}_{20}\text{FNO}_5\text{S}_2\text{H}$   $[(M+\text{H})^+]$ : 462.0840, found 462.0842.

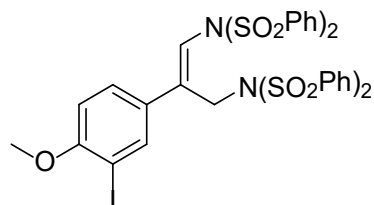


**(E/Z)-N,N'-(2-(3-chloro-4-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide)((E/Z)-2s):** Data for **(E/Z)-2s**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1) to afford 185 mg (60% yield) of **(E/Z)-2s**. White powder. TLC:  $R_f = 0.2$

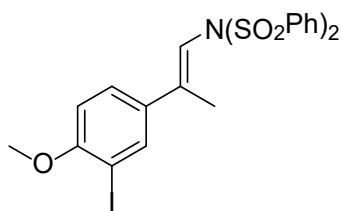
1 (n-hexane/EtOAc = 3/1). *Z:E* = 3:1. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.060 - 7.955 (m, 5.40H), 7.830 - 7.745 (m, 4.19H), 7.735 - 7.675 (m, 1.31H), 7.665 - 7.550 (m, 5.57H), 7.542 - 7.363 (m, 12.13H), 7.318 (dd, *J* = 8.8, 2 Hz, 0.36H), 7.114 (d, *J* = 2 Hz, 0.32H), 7.001 (d, *J* = 2 Hz, 1.01H), 6.809 (d, *J* = 8.8 Hz, 1.05H), 6.735 (d, *J* = 8.8 Hz, 0.32H), 5.791 (s, 0.36H), 5.755 (t, *J* = 1.2 Hz, 1H), 5.063 (d, *J* = 1.2 Hz, 2.04H), 4.652 (d, *J* = 1.2 Hz, 0.63H), 3.904 (s, 2.93H), 3.889 (s, 1.00H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.429, 155.327, 145.569, 144.091, 139.306, 138.984, 138.375, 138.307, 134.491, 134.235, 134.021, 130.421, 129.779, 129.170, 129.146, 128.922, 128.855, 128.825, 128.686, 128.478, 128.341, 127.934, 126.547, 122.489, 122.430, 120.292, 120.090, 111.792, 111.617, 56.303, 56.237, 52.157, 48.221. HRMS (ESI, *M/Z*): calcd for C<sub>34</sub>H<sub>29</sub>ClN<sub>2</sub>O<sub>9</sub>S<sub>4</sub>Na [(*M*+Na)<sup>+</sup>]: 795.0337, found 795.0338.



**(*E*)-N-(2-(3-chloro-4-methoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide(3s):** Data for **3s**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 10/1) to afford 23 mg (12% yield) of **3s**. White solid. TLC: *R<sub>f</sub>* = 0.14 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.045 - 7.970 (m, 4H), 7.715 - 7.630 (m, 2H), 7.610 - 7.520 (m, 4H), 7.409 (d, *J* = 2.4 Hz, 1H), 7.294 - 7.242 (m, 1H), 6.910 (d, *J* = 8.4 Hz, 1H), 6.039 (d, *J* = 1.6 Hz, 1H), 3.922 (s, 3H), 1.685 (d, *J* = 1.6 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.682, 148.205, 139.524, 134.137, 132.290, 129.249, 128.367, 128.301, 125.985, 122.785, 116.536, 111.956, 56.380, 16.397. HRMS (ESI, *M/Z*): calcd for C<sub>22</sub>H<sub>20</sub>ClNO<sub>5</sub>S<sub>2</sub>H [(*M*+H)<sup>+</sup>]: 478.0545, found 478.0550.

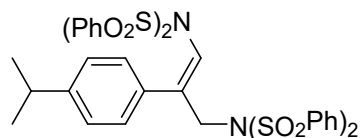


**(Z)-N,N'-(2-(3-iodo-4-methoxyphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2t):** Data for **(Z)-2t**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White solid. TLC:  $R_f = 0.52$  (n-hexane/EtOAc = 3/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.040 - 7.955 (m, 4H), 7.820 - 7.730 (m, 4H), 7.680 - 7.570 (m, 4H), 7.555 - 7.374 (m, 10H), 6.692 (d,  $J = 8.4$  Hz, 1H), 5.752 (t,  $J = 1.6$  Hz, 1H), 5.045 (d,  $J = 1.6$  Hz, 2H), 3.881 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.517, 145.363, 139.248, 138.630, 138.390, 134.482, 134.093, 129.767, 129.404, 129.177, 129.145, 128.924, 128.496, 119.927, 110.322, 86.087, 56.553, 48.228. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{34}\text{H}_{29}\text{I}\text{N}_2\text{O}_9\text{S}_4\text{H} [(M+H)^+]$ : 864.9874, found 864.9880.

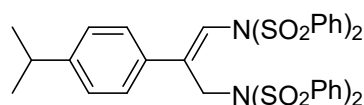


**(E)-N-(2-(3-iodo-4-methoxyphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3t):** Data for **3t**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 5/1) afforded 52 mg (23% yield). White solid. TLC:  $R_f = 0.17$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.045 - 7.957 (m, 4H), 7.796 (d,  $J = 2.4$  Hz, 1H), 7.704 - 7.644 (m, 2H), 7.595 - 7.540 (m, 4H), 7.352 (dd,  $J = 8.8, 2.4$  Hz, 1H), 6.796 (d,  $J = 8.4$  Hz, 1H), 6.018 (d,  $J = 1.6$  Hz, 1H), 3.901 (s, 3H), 1.680 (d,  $J = 1.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.838, 148.019, 139.518, 137.409, 134.129, 133.416, 129.248, 128.374, 127.813, 116.444, 110.640, 86.304, 56.624, 16.475. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{22}\text{H}_{20}\text{INO}_5\text{S}_2\text{H} [(M+H)^+]$ : 569.9901, found 569.9910.

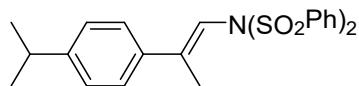




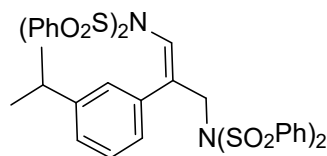
**(E)-N, N'-(2-(4-isopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2u):** Data for **(E)-2u**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC:  $R_f = 0.19$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.985 (m, 4H), 7.625 - 7.515 (m, 8H), 7.484 - 7.420 (m, 4H), 7.395 - 7.300 (m, 6H), 7.176 (d,  $J = 8$  Hz, 2H), 5.559 (t,  $J = 1.6$  Hz, 1H), 4.670 (d,  $J = 1.6$  Hz, 2H), 2.955 (hept,  $J = 6.8$  Hz, 1H), 1.317 (d,  $J = 7.2$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.533, 146.509, 138.969, 138.149, 134.179, 133.864, 131.563, 129.172, 128.924, 128.792, 128.716, 126.831, 118.317, 52.550, 34.016, 24.190. HRMS (ESI, M/Z): calcd for  $\text{C}_{36}\text{H}_{34}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [(M+N a) $^+$ ]: 773.1091, found 773.1067.



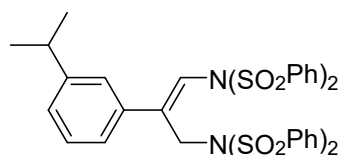
**(Z)-N, N'-(2-(4-isopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2u):** Data for **(Z)-2u**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC:  $R_f = 0.15$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.025 - 7.960 (m, 4H), 7.650 - 7.610 (m, 4H), 7.610 - 7.530 (m, 4H), 7.470 (t,  $J = 8$  Hz, 4H), 7.415 - 7.325 (m, 6H), 7.169 (d,  $J = 8.4$  Hz, 2H), 5.862 (t,  $J = 1.6$  Hz, 1H), 4.924 (d,  $J = 1.6$  Hz, 2H), 2.950 (hept,  $J = 6.8$  Hz, 1H), 1.308 (d,  $J = 6.8$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.730, 147.649, 138.931, 138.785, 134.305, 133.858, 132.939, 129.156, 128.966, 128.845, 128.739, 128.540, 126.651, 119.114, 47.910, 34.010, 24.174. HRMS (ESI, M/Z): calcd for  $\text{C}_{36}\text{H}_{34}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 773.1091, found 773.1067.



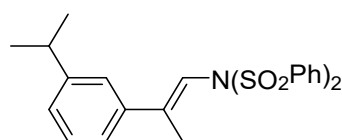
**(E)-N-(2-(4-isopropylphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3u):** Data for **3u**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 19 mg (10% yield). White solid. TLC:  $R_f$  = 0.42 (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.030 - 7.990 (m, 4H), 7.690 - 7.635 (m, 2H), 7.585 - 7.520 (m, 4H), 7.350 - 7.305 (m, 2H), 7.245 - 7.210 (m, 2H), 6.086 (q,  $J$  = 1.2 Hz, 1H), 2.927 (hept,  $J$  = 6.8 Hz, 1H), 1.699 (d,  $J$  = 1.6 Hz, 3H), 1.262 (d,  $J$  = 7.2 Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.147, 149.830, 139.677, 136.628, 134.043, 129.218, 128.400, 126.771, 126.539, 116.442, 34.021, 24.026, 16.485. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{24}\text{H}_{25}\text{NO}_4\text{S}_2\text{Na}$  [( $M+\text{Na}$ ) $^+$ ]: 478.1118, found 478.1120.



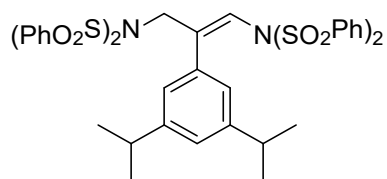
**(E)-N,N'-(2-(3-isopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2v):** Data for **(E)-2v**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC:  $R_f$  = 0.30 (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.065 - 8.000 (m, 4H), 7.595 - 7.505 (m, 8H), 7.495 - 7.425 (m, 4H), 7.400 - 7.325 (m, 5H), 7.260 - 7.235 (m, 2H), 7.208 - 7.160 (m, 1H), 5.558 (t,  $J$  = 1.6 Hz, 1H), 4.707 (d,  $J$  = 1.6 Hz, 2H), 2.845 (hept,  $J$  = 6.8 Hz, 1H), 1.174 (d,  $J$  = 6.8 Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.661, 146.669, 138.994, 138.187, 134.169, 133.804, 129.159, 128.875, 128.814, 128.722, 127.473, 126.866, 126.342, 118.775, 52.482, 34.194, 23.996. HRMS (ESI,  $M/Z$ ): calcd for  $\text{C}_{36}\text{H}_{34}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [( $M+\text{Na}$ ) $^+$ ]: 773.1091, found 773.1099.



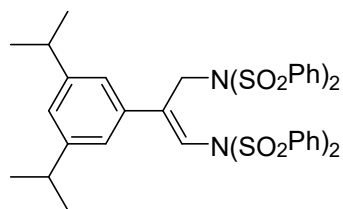
**(Z)-N,N'-(2-(3-isopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2v):** Data for **(Z)-2v**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). White solid. TLC:  $R_f = 0.29$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.045 - 7.975 (m, 4H), 7.640 - 7.530 (m, 8H), 7.505 - 7.445 (m, 4H), 7.430 - 7.345 (m, 5H), 7.293 - 7.220 (m, 3H), 7.208 - 7.168 (m, 1H), 5.894 (t,  $J = 1.6$  Hz, 1H), 4.901 (d,  $J = 1.6$  Hz, 2H), 2.894 (hept,  $J = 6.8$  Hz, 1H), 1.221 (d,  $J = 6.8$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.471, 148.164, 138.852, 138.830, 135.536, 134.312, 133.833, 129.172, 128.927, 128.836, 128.768, 128.700, 127.166, 126.920, 126.280, 119.234, 47.972, 34.292, 24.033. HRMS (ESI, M/Z): calcd for  $\text{C}_{36}\text{H}_{34}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 773.1091, found 773.1099.



**(E)-N-(2-(3-isopropylphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3v):** Data for **3v**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 21 mg (12% yield). White solid. TLC:  $R_f = 0.57$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.050 - 7.995 (m, 4H), 7.698 - 7.642 (m, 2H), 7.595 - 7.530 (m, 4H), 7.325 - 7.274 (m, 1H), 7.255 - 7.180 (m, 3H), 6.079 (d,  $J = 1.2$  Hz, 1H), 2.924 (hept,  $J = 6.8$  Hz, 1H), 1.724 (d,  $J = 1.6$  Hz, 3H), 1.270 (d,  $J = 6.8$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.391, 149.411, 139.676, 139.237, 134.076, 129.233, 128.704, 128.439, 127.150, 124.811, 124.122, 116.918, 34.342, 24.132, 16.720. HRMS (ESI, M/Z): calcd for  $\text{C}_{24}\text{H}_{25}\text{NO}_4\text{S}_2\text{Na}$  [(M+Na) $^+$ ]: 478.1118, found 478.1118.

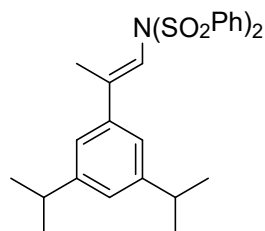


**(E)-N,N'-(2-(3,5-diisopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2w):** Data for **(E)-2w**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). Light yellow solid. TLC:  $R_f = 0.41$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.070 - 8.005 (m, 4H), 7.575 - 7.500 (m, 8H), 7.480 - 7.415 (m, 4H), 7.380 - 7.315 (m, 4H), 7.197 (d,  $J = 1.6$  Hz, 2H), 7.145 - 7.120 (m, 1H), 5.491 (t,  $J = 1.6$  Hz, 1H), 4.725 (d,  $J = 1.6$  Hz, 2H), 2.840 (hept,  $J = 6.8$  Hz, 2H), 1.174 (d,  $J = 7.2$  Hz, 12H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.688, 147.012, 139.058, 138.308, 134.134, 134.114, 133.707, 129.221, 129.128, 128.859, 128.674, 125.164, 124.798, 118.540, 52.497, 34.327, 24.094. HRMS (ESI, M/Z): calcd for  $\text{C}_{39}\text{H}_{40}\text{N}_2\text{O}_8\text{S}_4\text{Na}$  [(M+Na) $^+$ ]: 815.1560, found 815.1559.

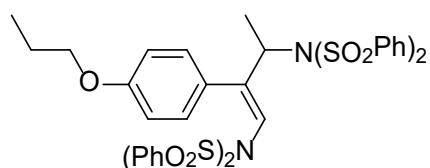


**(Z)-N,N'-(2-(3,5-diisopropylphenyl)prop-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((Z)-2w):** Data for **(Z)-2w**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 8/1). Light yellow solid. TLC:  $R_f = 0.33$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.040 - 8.000 (m, 4H), 7.626 - 7.576 (m, 2H), 7.574 - 7.529 (m, 2H), 7.525 - 7.440 (m, 8H), 7.395 - 7.340 (m, 4H), 7.238 (d,  $J = 1.6$  Hz, 2H), 7.190 - 7.165 (m, 1H), 5.936 (t,  $J = 1.6$  Hz, 1H), 4.824 (d,  $J = 1.6$  Hz, 2H), 2.893 (hept,  $J = 6.8$  Hz, 2H), 1.222 (d,  $J = 6.8$  Hz, 12H).  $^{13}\text{C}$  NMR

(101 MHz, CDCl<sub>3</sub>)  $\delta$  149.595, 148.672, 139.037, 138.771, 135.479, 134.240, 133.793, 129.171, 128.887, 128.761, 125.230, 124.869, 118.644, 47.921, 34.403, 24.139. HRMS (ESI, M/Z): calcd for C<sub>39</sub>H<sub>40</sub>N<sub>2</sub>O<sub>8</sub>S<sub>4</sub>Na [(M+Na)<sup>+</sup>]: 815.1560, found 815.1559.

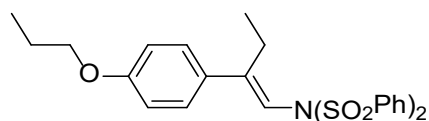


**(E)-N-(2-(3,5-diisopropylphenyl)prop-1-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (3w):** Data for **3w**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 22 mg (11% yield). Light yellow solid. TLC: R<sub>f</sub> = 0.66 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.050 - 8.005 (m, 4H), 7.700 - 7.640 (m, 2H), 7.590 - 7.530 (m, 4H), 7.099 (s, 1H), 7.040 (d, *J* = 1.6 Hz, 2H), 6.068 (d, *J* = 1.2 Hz, 1H), 2.965 - 2.845 (m, 2H), 1.729 (d, *J* = 1.2 Hz, 3H), 1.265 (d, *J* = 6.8 Hz, 12H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  150.675, 149.365, 139.690, 139.206, 134.050, 129.212, 128.453, 125.455, 122.315, 116.694, 34.389, 24.182, 16.819. HRMS (ESI, M/Z): calcd for C<sub>27</sub>H<sub>31</sub>NO<sub>4</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 520.1587, found 520.1587.

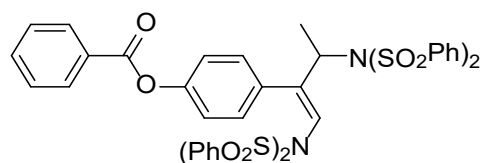


**(E)-N, N'-(2-(4-propoxyphenyl)but-1-ene-1,3-diyl)bis(N-(phenylsulfonyl)benzenesulfonamide) ((E)-2x):** Data for **(E)-2x**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White powder. TLC: R<sub>f</sub> = 0.17 (n-hexane/EtOAc = 5/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.989 (s, 4H), 7.682 - 7.515 (m, 8H), 7.498 - 7.328 (m, 8H), 7.168 - 7.105

(m, 2H), 6.710 - 6.645 (m, 2H), 5.813 (d,  $J = 2$  Hz, 1H), 5.406 (qd,  $J = 7.2$ , 1.6 Hz, 1H), 3.910 (t,  $J = 6.8$  Hz, 2H), 1.920 - 1.815 (m, 2H), 1.527 (d,  $J = 7.2$  Hz, 3H), 1.112 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  159.286, 149.709, 138.445, 133.916, 133.840, 133.800, 130.328, 129.187, 129.008, 128.863, 128.701, 126.810, 119.733, 114.355, 69.523, 61.278, 22.778, 18.885, 10.811. HRMS (ESI, M/Z): calcd for  $\text{C}_{37}\text{H}_{36}\text{N}_2\text{O}_9\text{S}_4\text{K}$  [(M+K) $^+$ ]: 819.0936, found 819.0899.

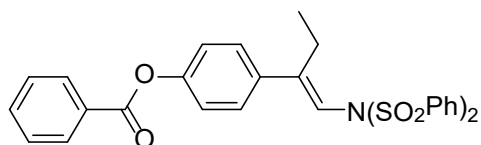


**(E)-N-(phenylsulfonyl)-N-(2-(4-propoxyphenyl)but-1-en-1-yl)benzenesulfonamide (3x):** Data for **3x**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 27 mg (14% yield). Light yellow solid. TLC:  $R_f = 0.51$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.030 - 7.970 (m, 4H), 7.695 - 7.630 (m, 2H), 7.580 - 7.523 (m, 4H), 7.255 - 7.205 (m, 2H), 6.910 - 6.855 (m, 2H), 5.836 (s, 1H), 3.935 (t,  $J = 6.8$  Hz, 2H), 2.245 (q,  $J = 7.6$  Hz, 2H), 1.875 - 1.770 (m, 2H), 1.046 (t,  $J = 7.6$  Hz, 3H), 0.596 (t,  $J = 7.6$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  159.674, 155.579, 139.468, 133.886, 129.827, 129.045, 128.361, 128.264, 114.782, 114.509, 69.588, 22.568, 11.696, 10.527. HRMS (ESI, M/Z): calcd for  $\text{C}_{25}\text{H}_{27}\text{NO}_5\text{S}_2\text{Na}$  [(M+Na) $^+$ ]: 508.1223, found 508.1227.

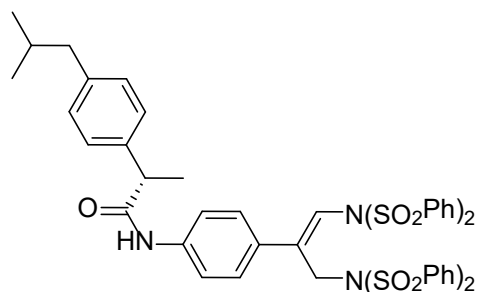


**(E)-4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)but-1-en-2-yl)phenyl benzoate ((E)-2y):** Data for **(E)-2y**: The general procedure was followed, and was purified with flash chromatography (n-hexane/EtOAc = 3/1). White powder. TLC:  $R_f = 0.14$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.285

- 8.225 (m, 2H), 7.988 (s, 4H), 7.717 - 7.660 (m, 2H), 7.645 - 7.545 (m, 10 H), 7.520 - 7.450 (m, 7H), 7.248 (d,  $J = 9.6$  Hz, 2H), 7.051 (d,  $J = 8.8$  Hz, 2H), 5.898 (d,  $J = 2$  Hz, 1H), 5.396 (qd,  $J = 6.8, 2$  Hz, 1H), 1.557 (d,  $J = 6.4$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  164.829, 151.013, 149.676, 138.131, 134.070, 133.925, 132.474, 130.315, 129.577, 129.304, 129.209, 129.147, 128.977, 128.849, 121.757, 120.077, 61.146, 18.900. HRMS (ESI, M/Z): calcd for  $\text{C}_{41}\text{H}_{34}\text{N}_2\text{O}_{10}\text{S}_4\text{Na}$   $[(\text{M}+\text{Na})^+]$ : 865.0989, found 865.0980.

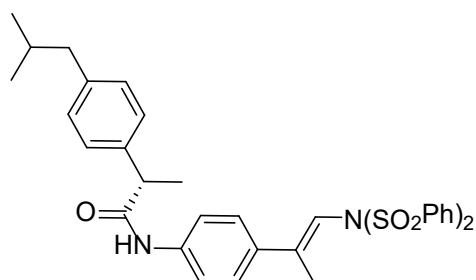


**(E)-4-(1-(N-(phenylsulfonyl)phenylsulfonamido)but-1-en-2-yl)phenyl benzoate (3y):** Data for **3y**: The general procedure was followed, and flash chromatography (n-hexane/EtOAc = 10/1) afforded 18 mg (8% yield). White solid. TLC:  $R_f = 0.21$  (n-hexane/EtOAc = 5/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.235 - 8.190(m, 2H), 8.040 - 7.982 (m, 4H), 7.705 - 7.640 (m, 3H), 7.595 - 7.505 (m, 6H), 7.400 - 7.340 (m, 2H), 7.254 - 7.222 (m, 2H), 5.905 (s, 1H), 2.300 (q,  $J = 7.6$  Hz, 2H), 0.623 (t,  $J = 7.6$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.217, 155.394, 151.404, 139.489, 135.643, 134.142, 133.917, 130.364, 129.258, 128.788, 128.495, 128.394, 122.065, 116.619, 22.967, 11.721. HRMS (ESI, M/Z): calcd for  $\text{C}_{29}\text{H}_{25}\text{NO}_6\text{S}_2\text{Na}$   $[(\text{M}+\text{Na})^+]$ : 570.1016, found 570.0999.



**(S, Z)-N-(4-(1,3-bis(N-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)-2-(4-isobutylphenyl)propenamide ((S, Z)-2z):** Data for **(S, Z)-2z**: The gener

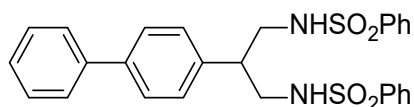
al procedure was followed and was purified by silica gel column chromatography with a gradient eluant of petrol ether/ethyl acetate to afford the desired products. Yellow solid. TLC:  $R_f = 0.20$  (n-hexane/EtOAc = 3/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.996 – 7.928 (m, 4H), 7.699 – 7.648 (m, 4H), 7.630 – 7.575 (m, 2H), 7.535 – 7.490 (m, 2H), 7.484 - 7.438 (m, 4H), 7.379 - 7.333 (m, 4H), 7.315 - 7.264 (m, 4H), 7.223 – 7.187 (m, 4H), 7.082 (s, 1H), 5.754 - 5.730 (m, 1H), 4.980 (d,  $J = 0.8$  Hz, 2H), 3.712 (q,  $J = 7.2$  Hz, 1H), 2.503 (d,  $J = 7.2$  Hz, 2H), 1.889 (hept,  $J = 6.8$  Hz, 1H), 1.613 (d,  $J = 7.2$  Hz, 3H), 0.927 (d,  $J = 6.8$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.729, 147.070, 141.452, 139.089, 138.477, 138.111, 134.401, 133.876, 131.066, 130.123, 129.164, 129.120, 129.015, 128.928, 128.574, 127.491, 119.547, 119.447, 48.279, 47.945, 45.164, 30.331, 22.537, 18.537. HRMS (ESI, M/Z): calcd for  $\text{C}_{46}\text{H}_{45}\text{N}_3\text{O}_9\text{S}_4\text{H}$  [(M+H) $^+$ ]: 912.2112, found 912.2118.



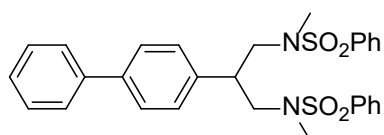
**(*S*, *E*)-2-(4-isobutylphenyl)-*N*-(4-(1-(*N*-(phenylsulfonyl)phenylsulfonamido)prop-1-en-2-yl)phenyl)propanamide (3z):** Data for **3z**: The general procedure was followed and was purified by silica gel column chromatography with a gradient eluant of petrol ether/ethyl acetate to afford the desired products 32 mg (13% yield). White solid. TLC:  $R_f = 0.35$  (n-hexane/EtOAc = 3/1).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.044 - 7.940 (m, 4H), 7.702 – 7.605 (m, 2H), 7.595 – 7.498 (m, 4H), 7.421 (d,  $J = 8.8$  Hz, 2H), 7.352 - 7.206 (m, 5H), 7.185 – 7.114 (m, 3H), 6.020 (d,  $J = 1.2$  Hz, 1H), 3.694 (q,  $J = 7.2$  Hz, 1H), 2.477 (d,  $J = 7.2$  Hz, 2H), 1.867 (hept,  $J = 6.8$  Hz, 1H), 1.676 (d,  $J = 1.2$  Hz, 3H), 1.586 (d,  $J = 7.2$  Hz, 3H), 0.912 (d,  $J = 6.8$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz, C



DCl<sub>3</sub>)  $\delta$  172.792, 149.197, 141.349, 139.522, 138.804, 138.024, 134.683, 134.099, 130.059, 129.225, 128.383, 127.532, 127.100, 119.559, 116.376, 47.923, 45.144, 30.313, 22.510, 18.633, 16.373. HRMS (ESI, M/Z): calcd for C<sub>34</sub>H<sub>36</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H [(M+H)<sup>+</sup>]: 617.2139, found 617.2142.

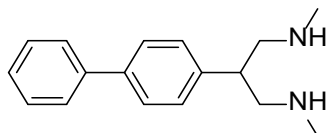


**N, N'-(2-([1,1'-biphenyl]-4-yl)propane-1,3-diyl)dibenzenesulfonamide (13):** Data for **13**: The general procedure was followed and was purified by silica gel column chromatography with a gradient eluant of petrol ether/ethyl acetate to afford the desired products 152 mg(47% yield). White solid. TLC: R<sub>f</sub> = 0.38 (n-hexane/EtOAc = 2/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.865 - 7.760 (m, 4H), 7.640 - 7.400 (m, 12H), 7.390 - 7.325 (m, 1H), 7.106 (d, *J* = 8 Hz, 2H), 4.751 (t, *J* = 6.4 Hz, 2H), 3.376 - 3.175 (m, 4H), 3.069 (p, *J* = 6.4 Hz, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  141.001, 140.426, 139.842, 137.436, 132.975, 129.403, 129.021, 128.325, 127.982, 127.706, 127.146, 127.104, 45.399, 45.071. HRMS (ESI, M/Z): calcd for C<sub>27</sub>H<sub>26</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 529.1227, found 529.1226.



**N, N'-(2-([1,1'-biphenyl]-4-yl)propane-1,3-diyl)bis(N-methylbenzenesulfonamide) (14):** Data for **14**: The general procedure was followed and was purified by silica gel column chromatography with a gradient eluant of petrol ether/ethyl acetate to afford the desired products 20 mg(95% yield). Colorless oil. TLC: R<sub>f</sub> = 0.44 (n-hexane/EtOAc = 3/1). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.810 - 7.730 (m, 4H), 7.616 - 7.552 (m, 6H), 7.550 - 7.490 (m, 4H), 7.475 - 7.410 (m, 2H), 7.385 - 7.300 (m, 3H), 3.745 - 3.620 (m, 2H), 3.393 (p, *J* = 7.6 Hz, 1H), 3.040 - 2.930 (m, 2H), 2.662 (s, 6H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  140.606, 140.521, 138.722, 137.335, 132.839, 129.304, 128.951,

128.692, 127.657, 127.535, 127.504, 127.113, 53.838, 43.995, 36.193. HRMS (ESI, M/Z): calcd for C<sub>29</sub>H<sub>30</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub>Na [(M+Na)<sup>+</sup>]: 557.1540, found 557.1496.



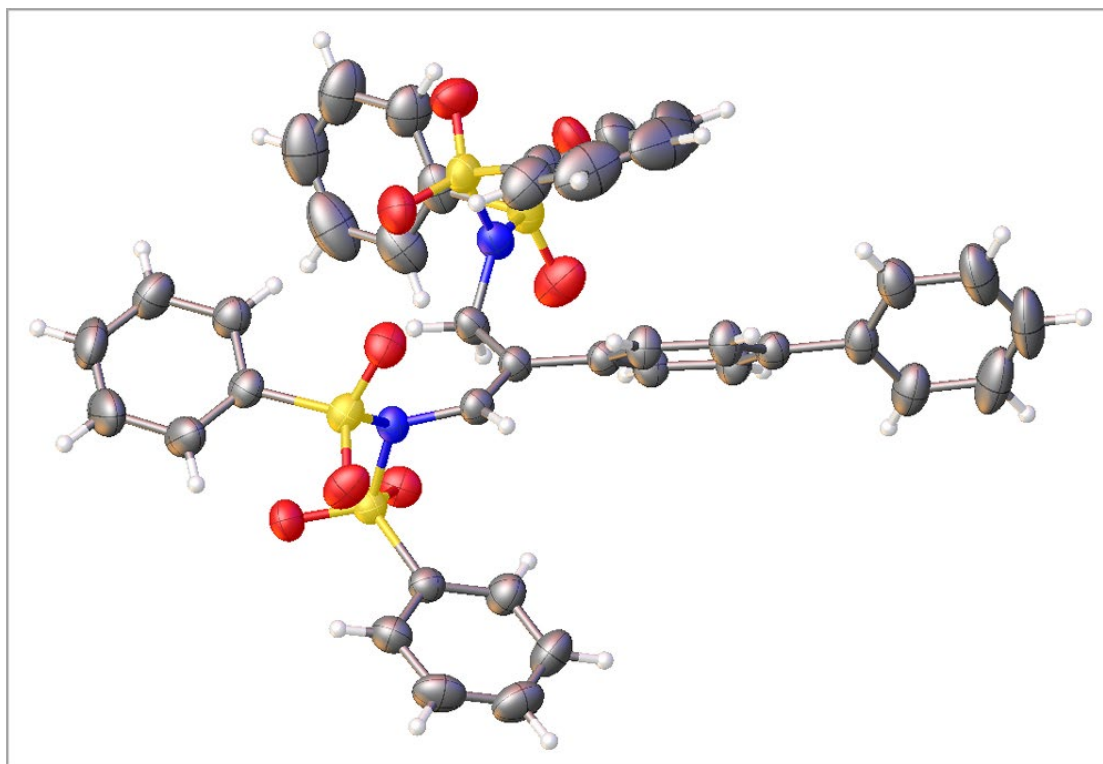
**2-([1,1'-biphenyl]-4-yl)-N<sub>1</sub>, N<sub>3</sub>-dimethylpropane-1, 3-diamine (15):** Data for **15**: The general procedure was followed. Light yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.600 - 7.530 (m, 4H), 7.460 - 7.400 (m, 2H), 7.360 - 7.275 (m, 3H), 3.220 - 3.130 (m, 1H), 2.960 - 2.840 (m, 4H), 2.426 (s, 6H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 140.890, 140.688, 140.095, 128.909, 128.246, 127.705, 127.381, 127.139, 56.670, 44.839, 36.392. HRMS (ESI, M/Z): calcd for C<sub>17</sub>H<sub>22</sub>N<sub>2</sub>H [(M+H)<sup>+</sup>]: 255.1856, found 255.1852.

### 7. X-ray Crystal Data

Crystal of (*Z*)-**2aa**、(*E*)-**2b** and (*E*)-**2y** suitable for X-ray diffraction was obtained according the following procedure: Under ambient atmosphere, (*Z*)-**2aa**、(*E*)-**2b** and (*E*)-**2y** (30 mg) was dissolved separately in ethyl acetate (1 mL) completely, then hexane (2 mL) was dropped to the upper layer. The mixture was then allowed to stand and single crystals of (*Z*)-**2aa**、(*E*)-**2b** and (*E*)-**2y** were grown by slow evaporation of the organic phase.

### *Crystal structure of compound (Z)-2aa (CCDC 2088081)*

#### **Crystal data and structure refinement for (Z)-2aa**

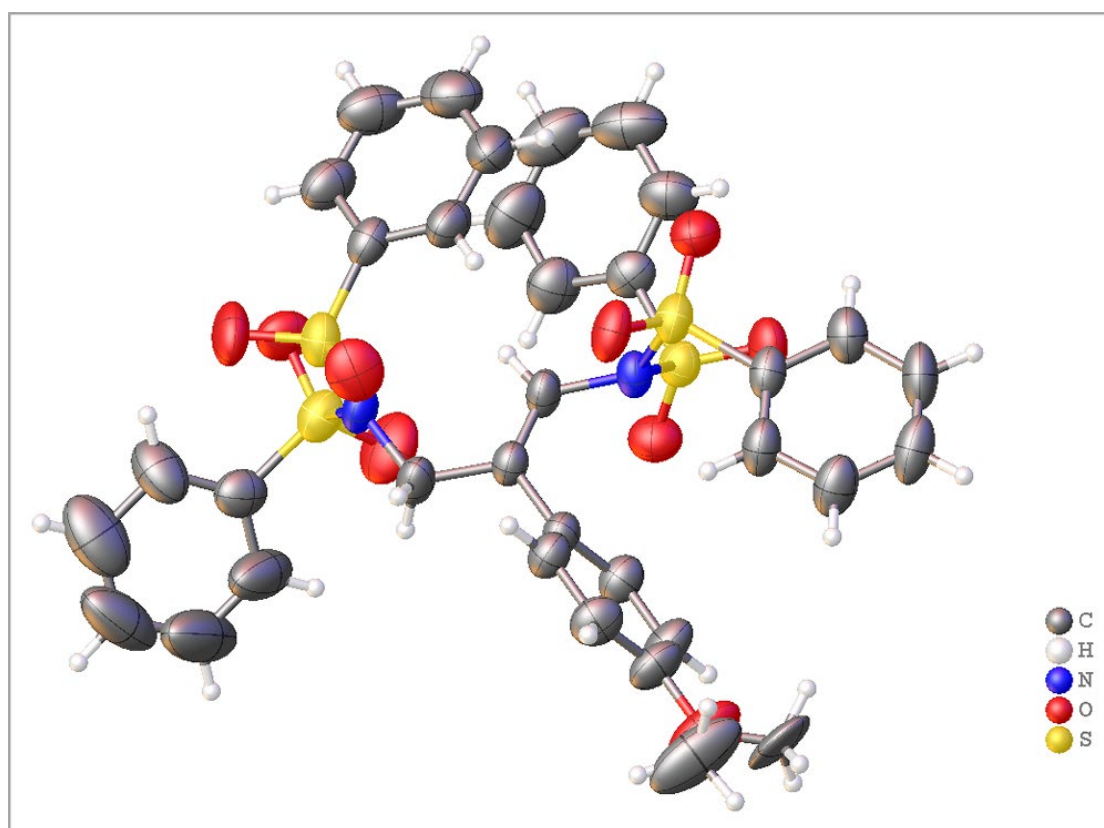


Identification code	<b>(Z)-2aa</b>
Empirical formula	C <sub>39</sub> H <sub>32</sub> N <sub>2</sub> O <sub>8</sub> S <sub>4</sub>
Formula weight	784.90
Temperature/K	274(30)
Crystal system	triclinic
Space group	P-1
a/Å	11.5994(4)
b/Å	13.0157(5)
c/Å	13.7849(6)
α/°	92.349(3)
β/°	109.747(3)
γ/°	104.027(3)
Volume/Å <sup>3</sup>	1883.09(13)
Z	2
ρ <sub>calc</sub> /cm <sup>3</sup>	1.384
μ/mm <sup>-1</sup>	2.781
F(000)	816.0
Crystal size/mm <sup>3</sup>	0.5 × 0.4 × 0.1
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	7.064 to 142.962

Index ranges	-12 ≤ h ≤ 14, -15 ≤ k ≤ 16, -16 ≤ l ≤ 16
Reflections collected	20652
Independent reflections	7205 [R <sub>int</sub> = 0.0500, R <sub>sigma</sub> = 0.0436]
Data/restraints/parameters	7205/0/478
Goodness-of-fit on F <sup>2</sup>	1.025
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0611, wR <sub>2</sub> = 0.1647
Final R indexes [all data]	R <sub>1</sub> = 0.0677, wR <sub>2</sub> = 0.1769
Largest diff. peak/hole / e Å <sup>-3</sup>	0.49/-0.46

***Crystal structure of compound (E)-2b (CCDC 2087231)***

**Crystal data and structure refinement for (E)-2b**

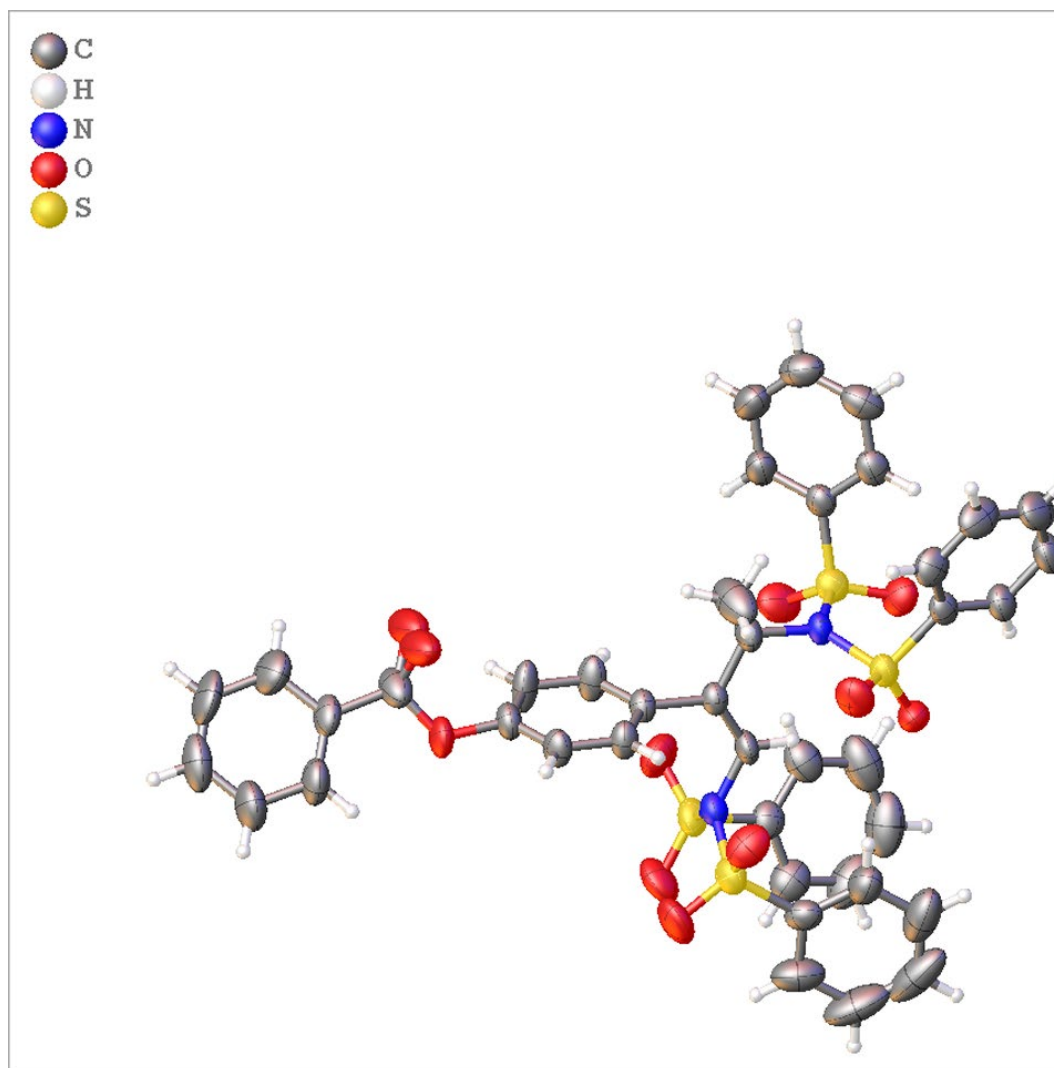


Identification code	<b>(E)-2b</b>
Empirical formula	C <sub>34</sub> H <sub>30</sub> N <sub>2</sub> O <sub>9</sub> S <sub>4</sub>
Formula weight	738.84
Temperature/K	273.15
Crystal system	monoclinic
Space group	P2 <sub>1</sub> /n
a/Å	13.4039(5)
b/Å	19.3046(6)

c/Å	13.9983(5)
$\alpha$ /°	90
$\beta$ /°	107.3350(10)
$\gamma$ /°	90
Volume/Å <sup>3</sup>	3457.6(2)
Z	4
$\rho_{\text{calc}}$ /cm <sup>3</sup>	1.419
$\mu$ /mm <sup>-1</sup>	0.332
F(000)	1536.0
Crystal size/mm <sup>3</sup>	0.15 × 0.15 × 0.12
Radiation	MoK $\alpha$ ( $\lambda$ = 0.71073)
2 $\Theta$ range for data collection/°	3.706 to 55.162
Index ranges	-17 ≤ h ≤ 17, -25 ≤ k ≤ 25, -18 ≤ l ≤ 18
Reflections collected	129729
Independent reflections	7984 [ $R_{\text{int}}$ = 0.1352, $R_{\text{sigma}}$ = 0.0536]
Data/restraints/parameters	7984/0/454
Goodness-of-fit on F <sup>2</sup>	1.018
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1$ = 0.0519, $wR_2$ = 0.1174
Final R indexes [all data]	$R_1$ = 0.1107, $wR_2$ = 0.1443
Largest diff. peak/hole / e Å <sup>-3</sup>	0.30/-0.47

***Crystal structure of compound (E)-2y (CCDC 2088082)***

**Crystal data and structure refinement for (E)-2y**



Identification code	<b>(E)-2y</b>
Empirical formula	C <sub>41</sub> H <sub>34</sub> N <sub>2</sub> O <sub>10</sub> S <sub>4</sub>
Formula weight	842.94
Temperature/K	293.15
Crystal system	triclinic
Space group	P-1
a/Å	11.7105(6)
b/Å	13.1872(8)
c/Å	13.7902(7)
α/°	110.882(5)

$\beta/^\circ$	93.715(4)
$\gamma/^\circ$	93.825(4)
Volume/ $\text{\AA}^3$	1976.32(19)
Z	2
$\rho_{\text{calc}}/\text{cm}^3$	1.417
$\mu/\text{mm}^{-1}$	0.302
F(000)	876.0
Crystal size/ $\text{mm}^3$	$0.35 \times 0.3 \times 0.25$
Radiation	MoK $\alpha$ ( $\lambda = 0.71073$ )
$2\Theta$ range for data collection/ $^\circ$	6.008 to 52.742
Index ranges	$-14 \leq h \leq 14, -15 \leq k \leq 16, -17 \leq l \leq 17$
Reflections collected	16224
Independent reflections	8075 [ $R_{\text{int}} = 0.0298, R_{\text{sigma}} = 0.0587$ ]
Data/restraints/parameters	8075/0/519
Goodness-of-fit on $F^2$	1.022
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1 = 0.0556, wR_2 = 0.1189$
Final R indexes [all data]	$R_1 = 0.0938, wR_2 = 0.1354$
Largest diff. peak/hole / $e \text{\AA}^{-3}$	0.64/-0.38

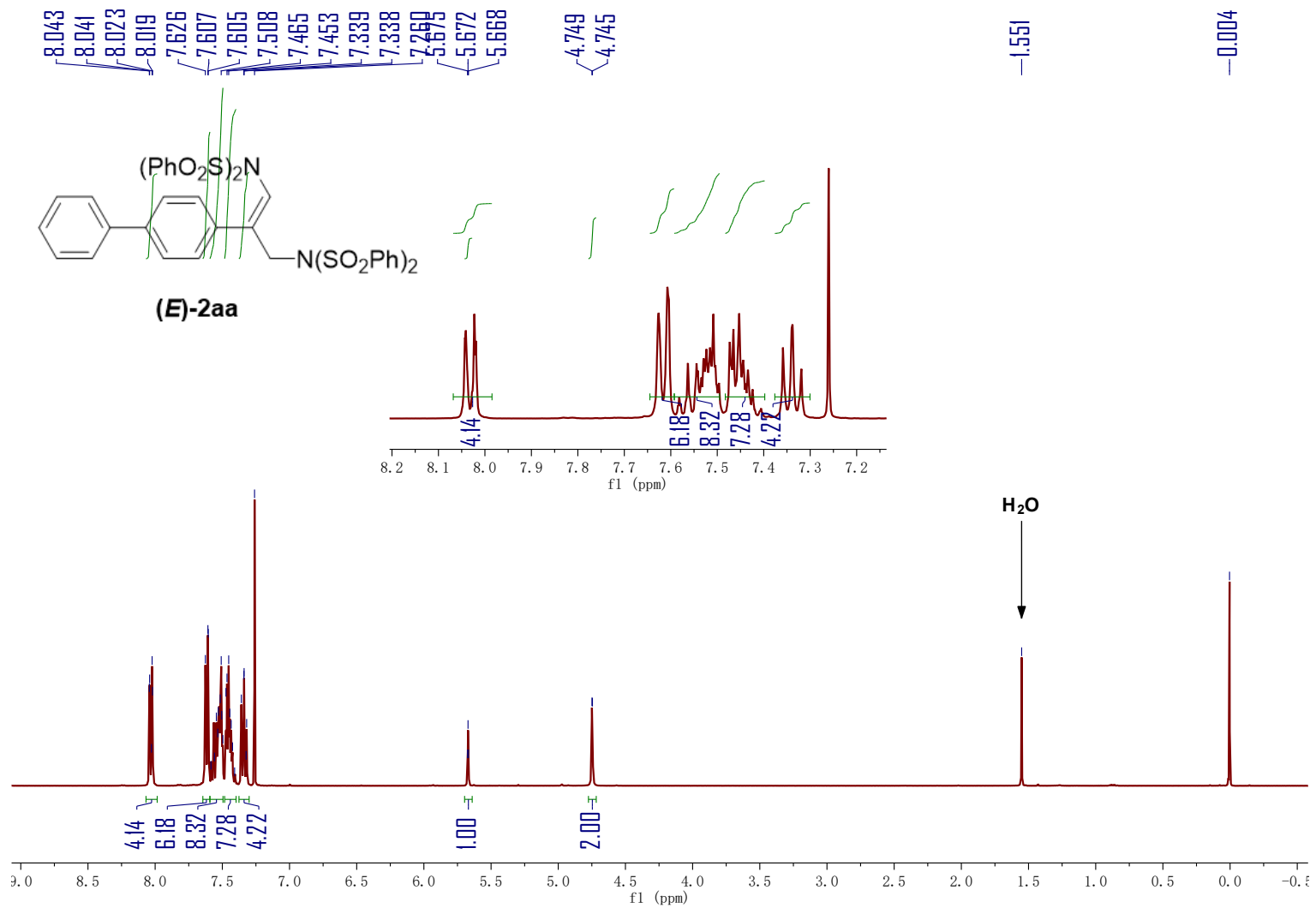
## 8. References

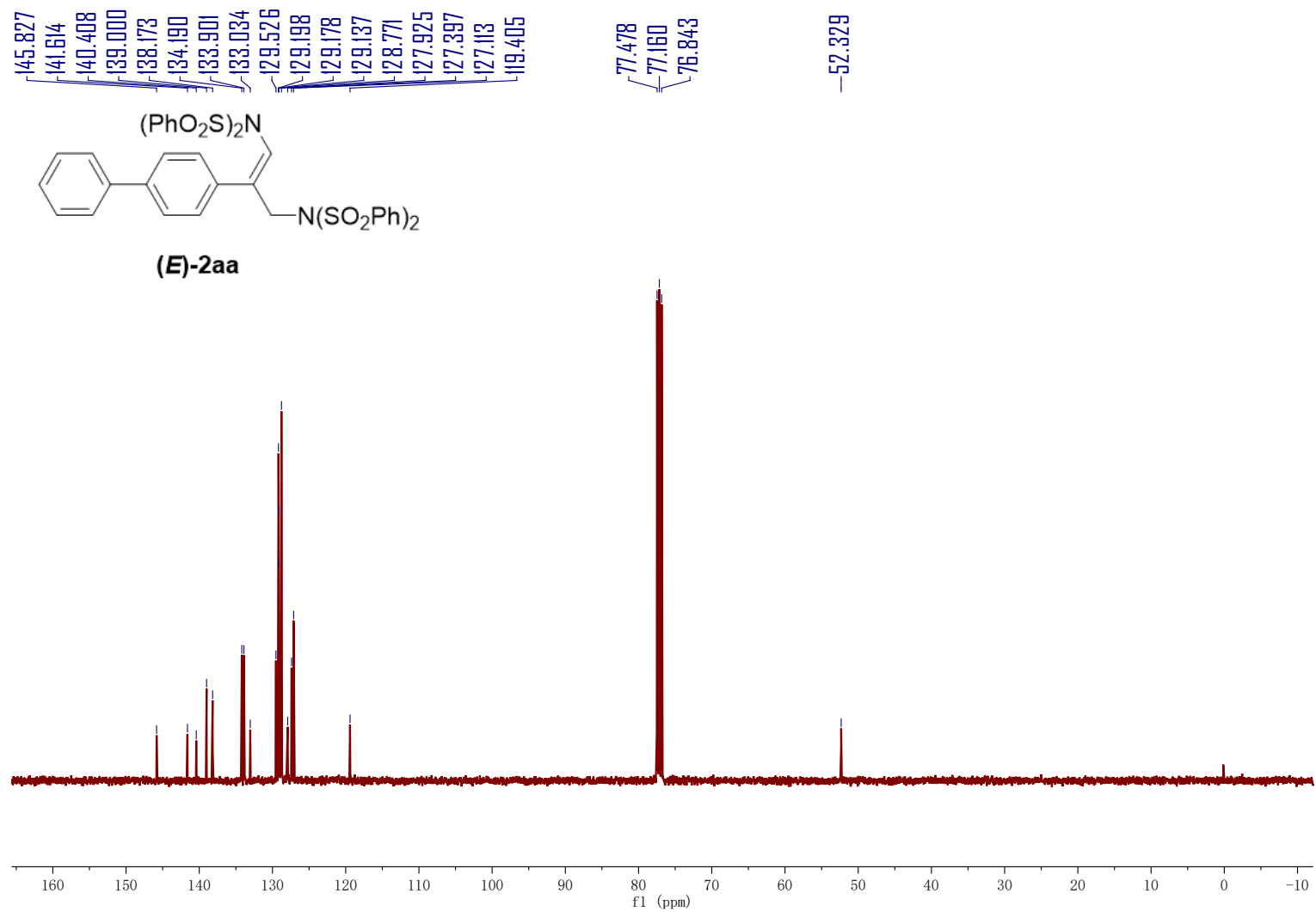
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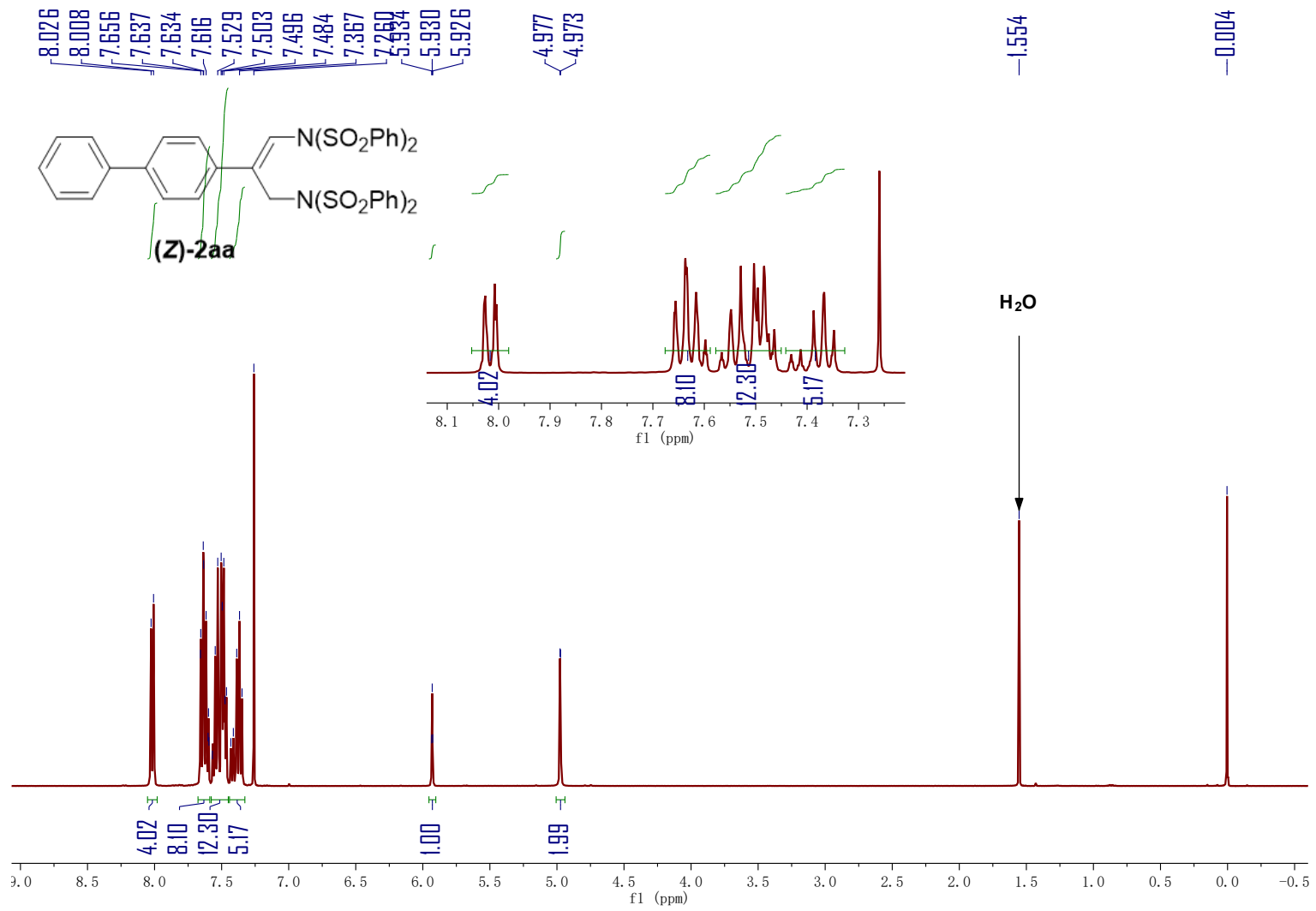
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## 9. <sup>1</sup>H and <sup>13</sup>C NMR spectrum of compounds





S59

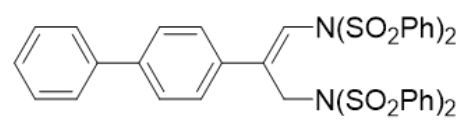


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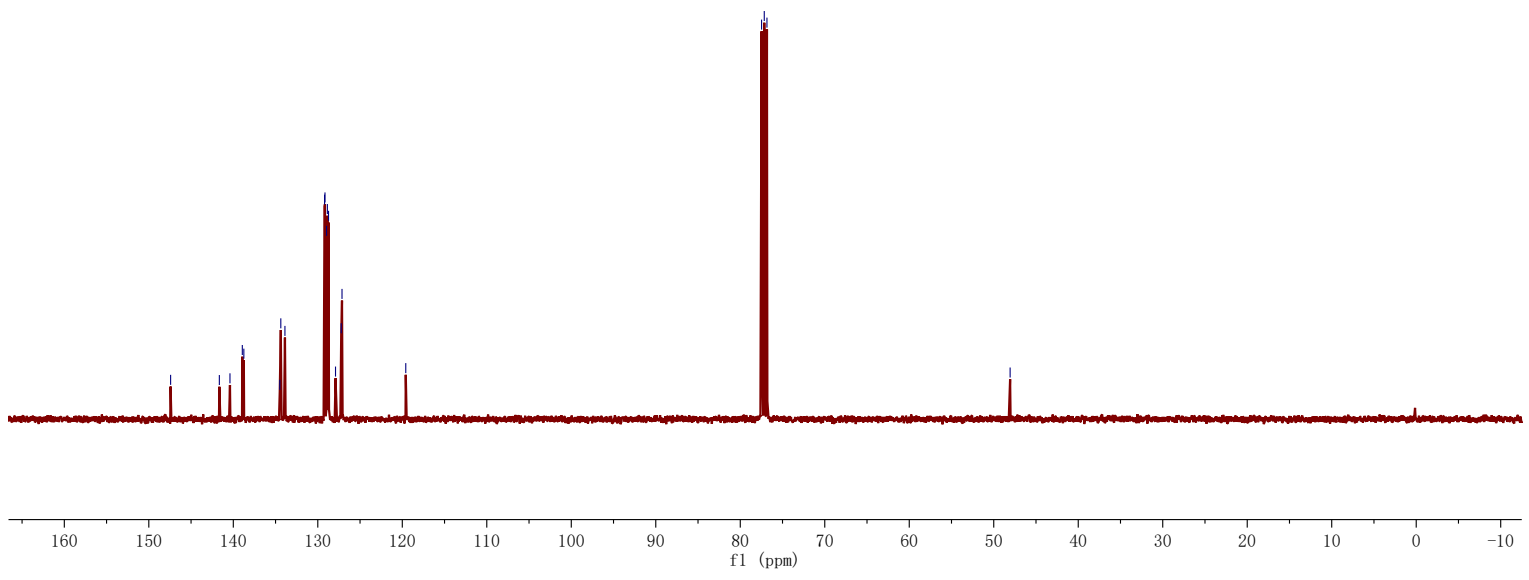
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138.753  
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128.867  
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127.902  
127.231  
127.134  
119.588

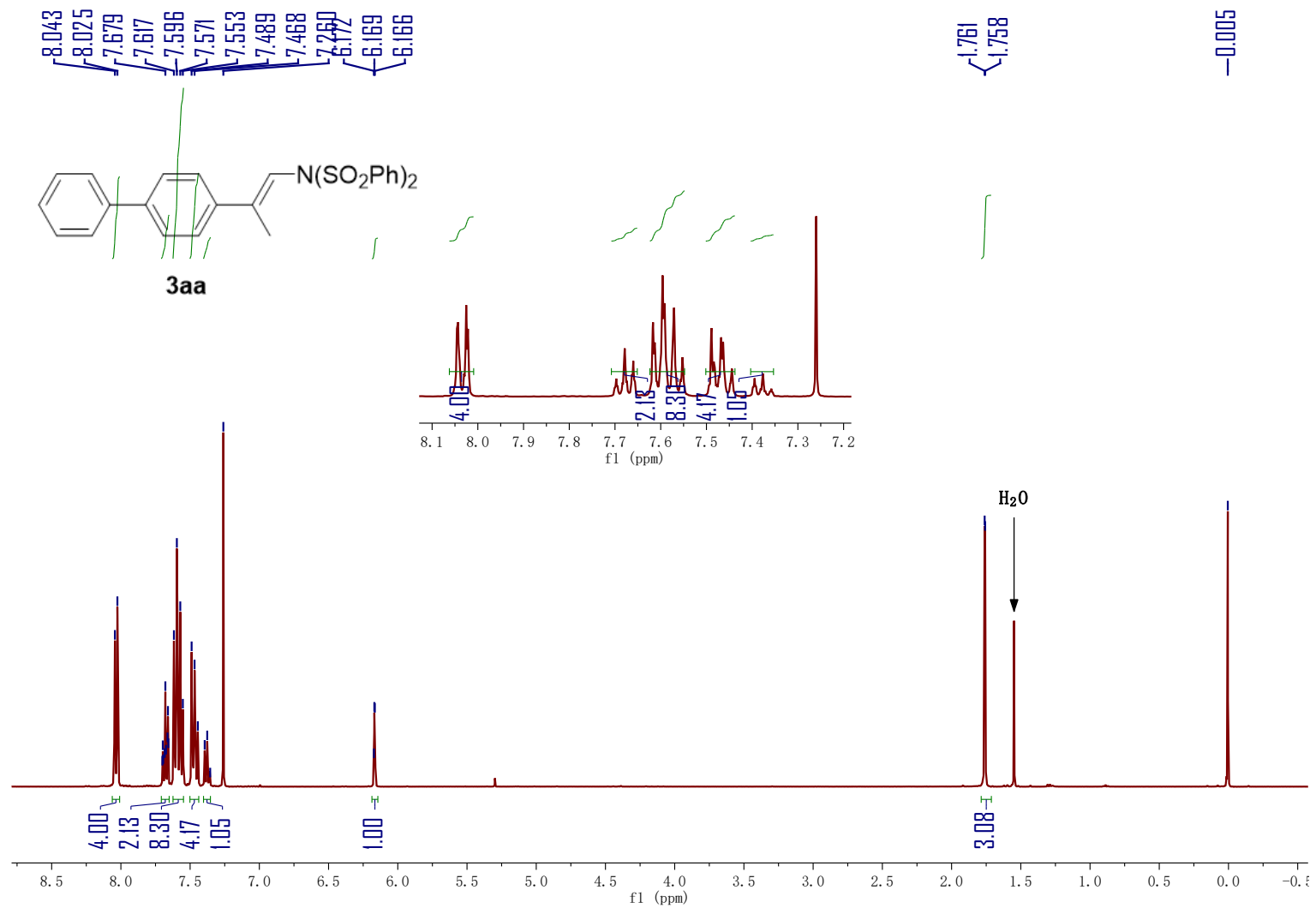
77.477  
77.160  
76.842

48.062



**(Z)-2aa**



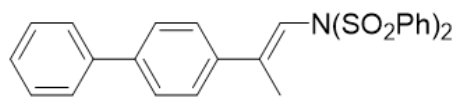


139.632  
137.989  
134.110  
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128.408  
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127.382  
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126.977  
117.037

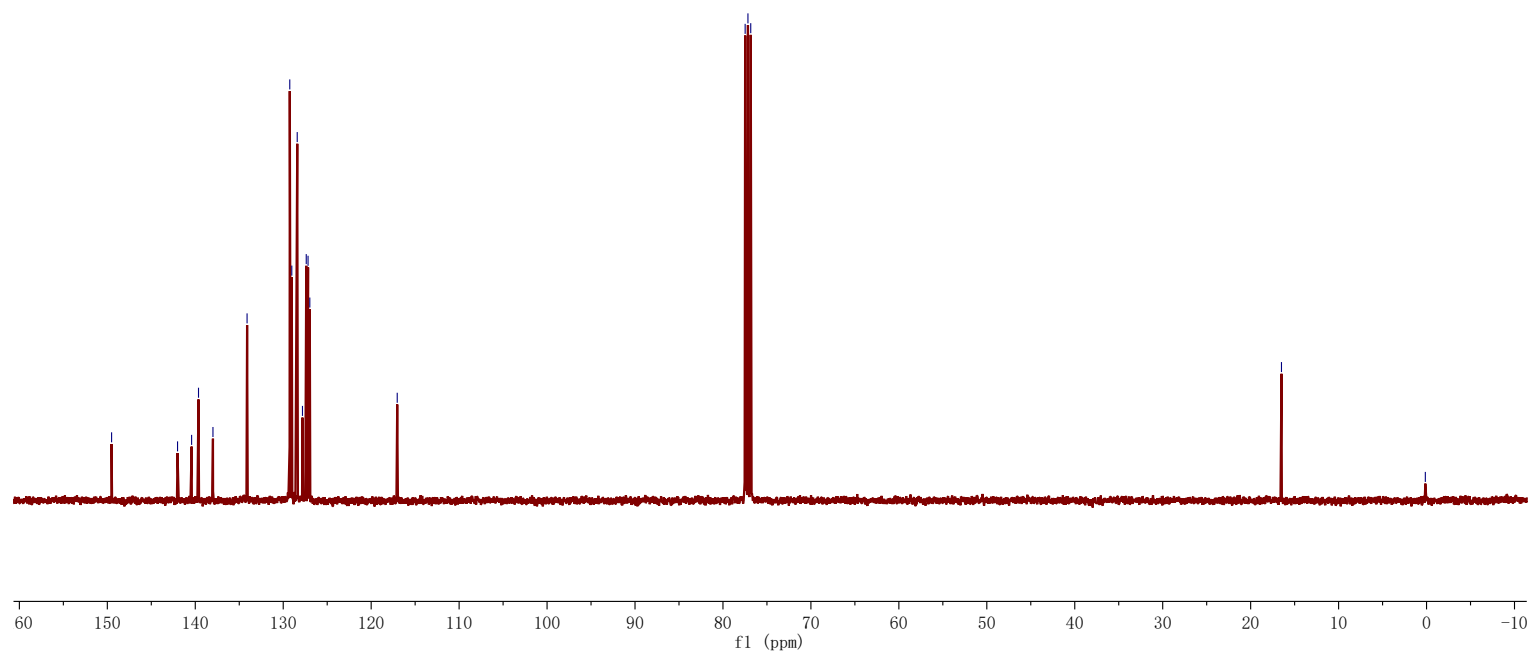
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77.160  
76.842

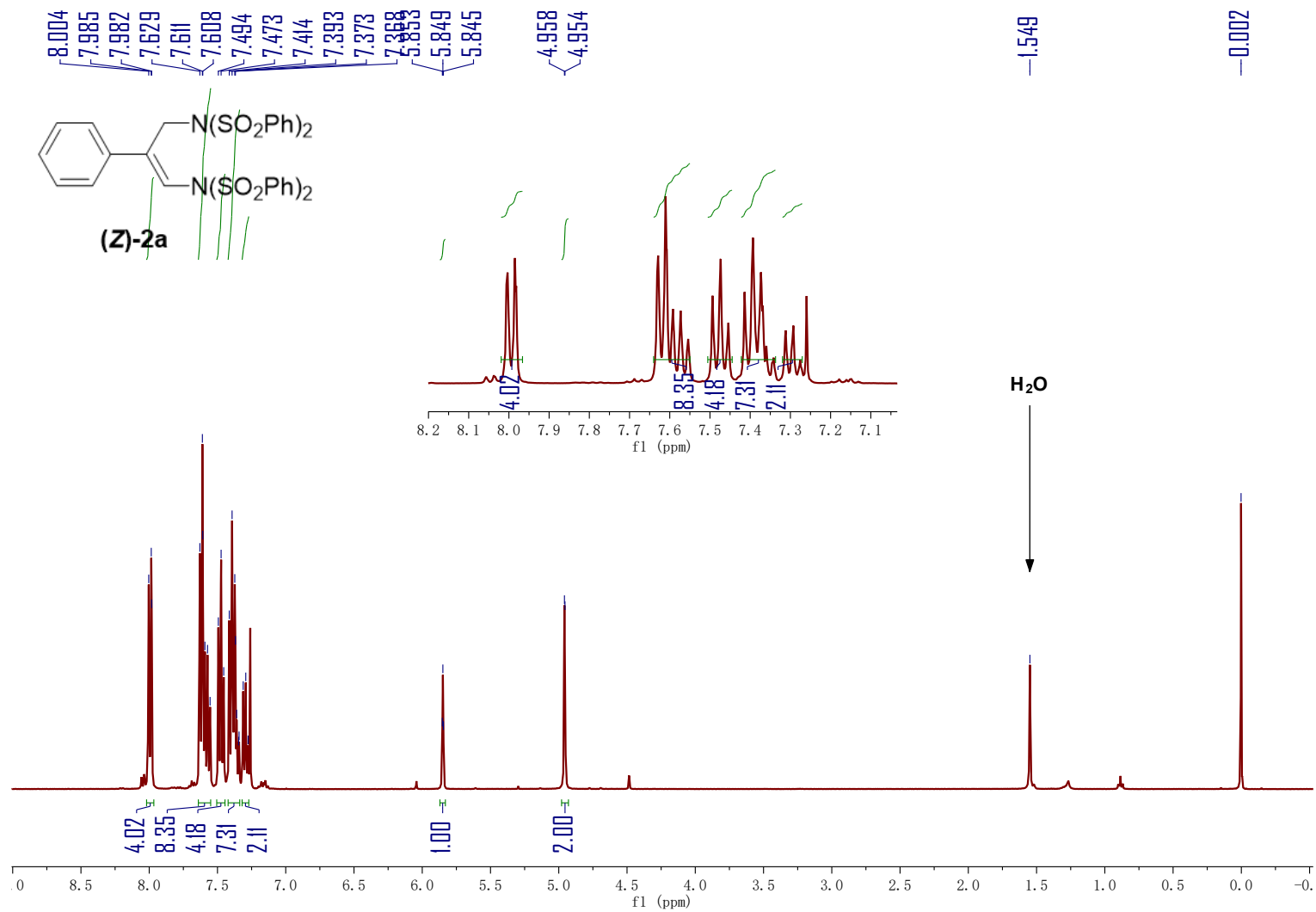
16.496

0.139

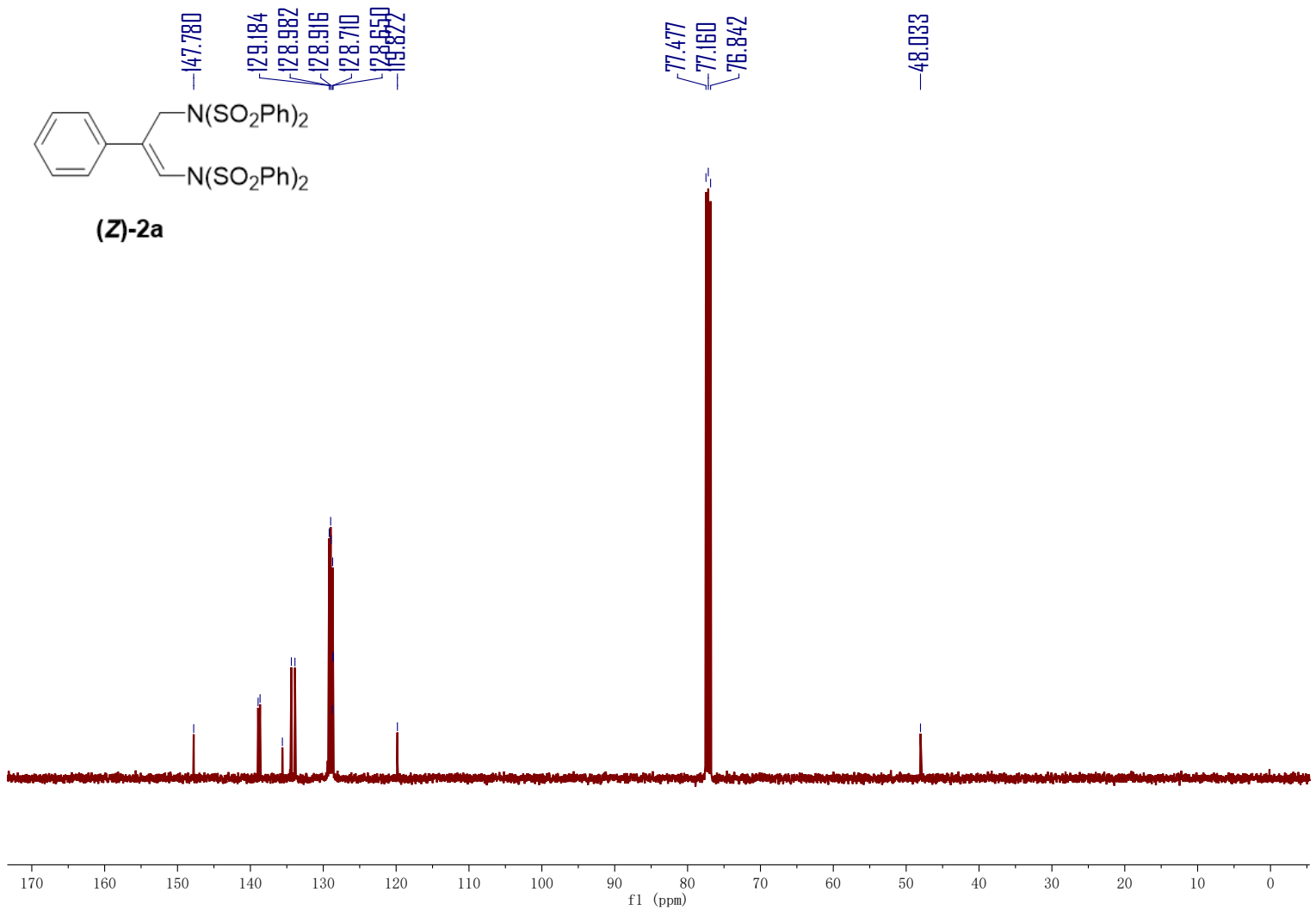


3aa

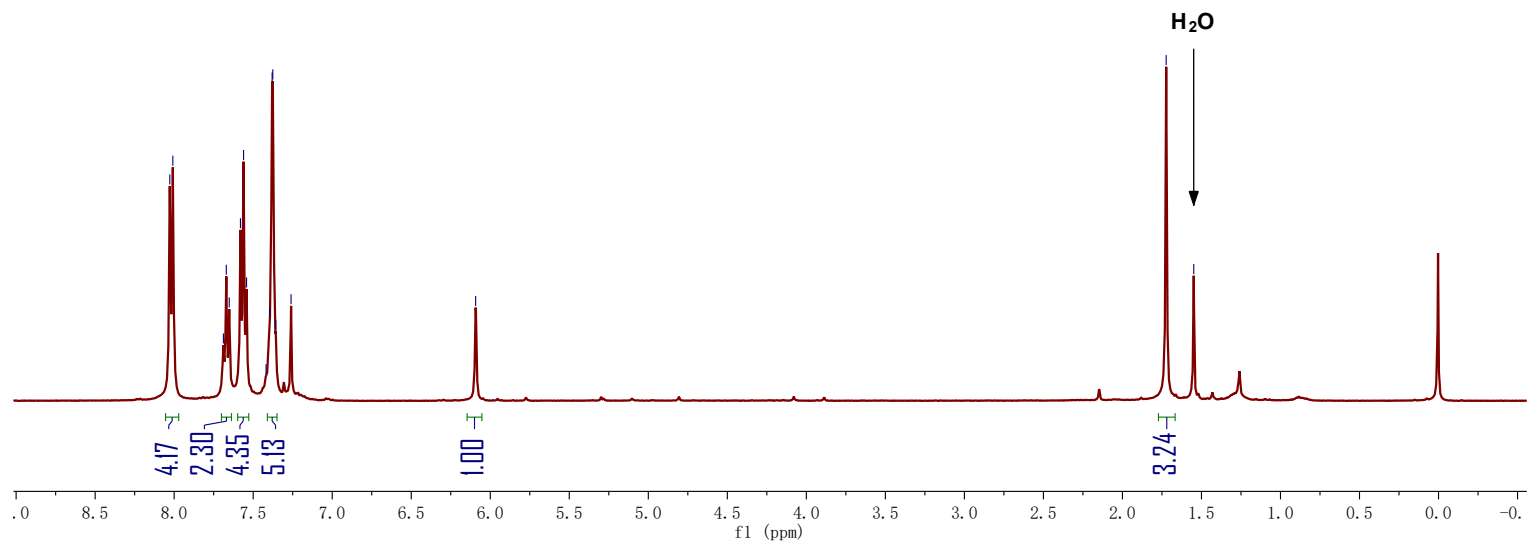
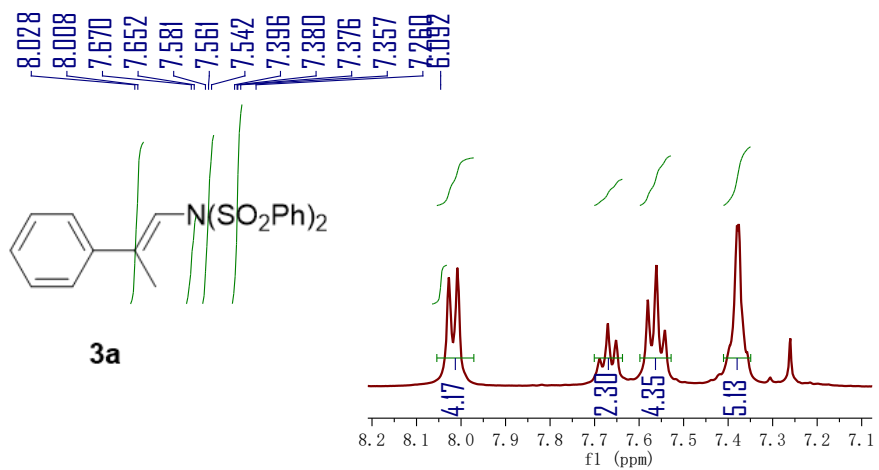




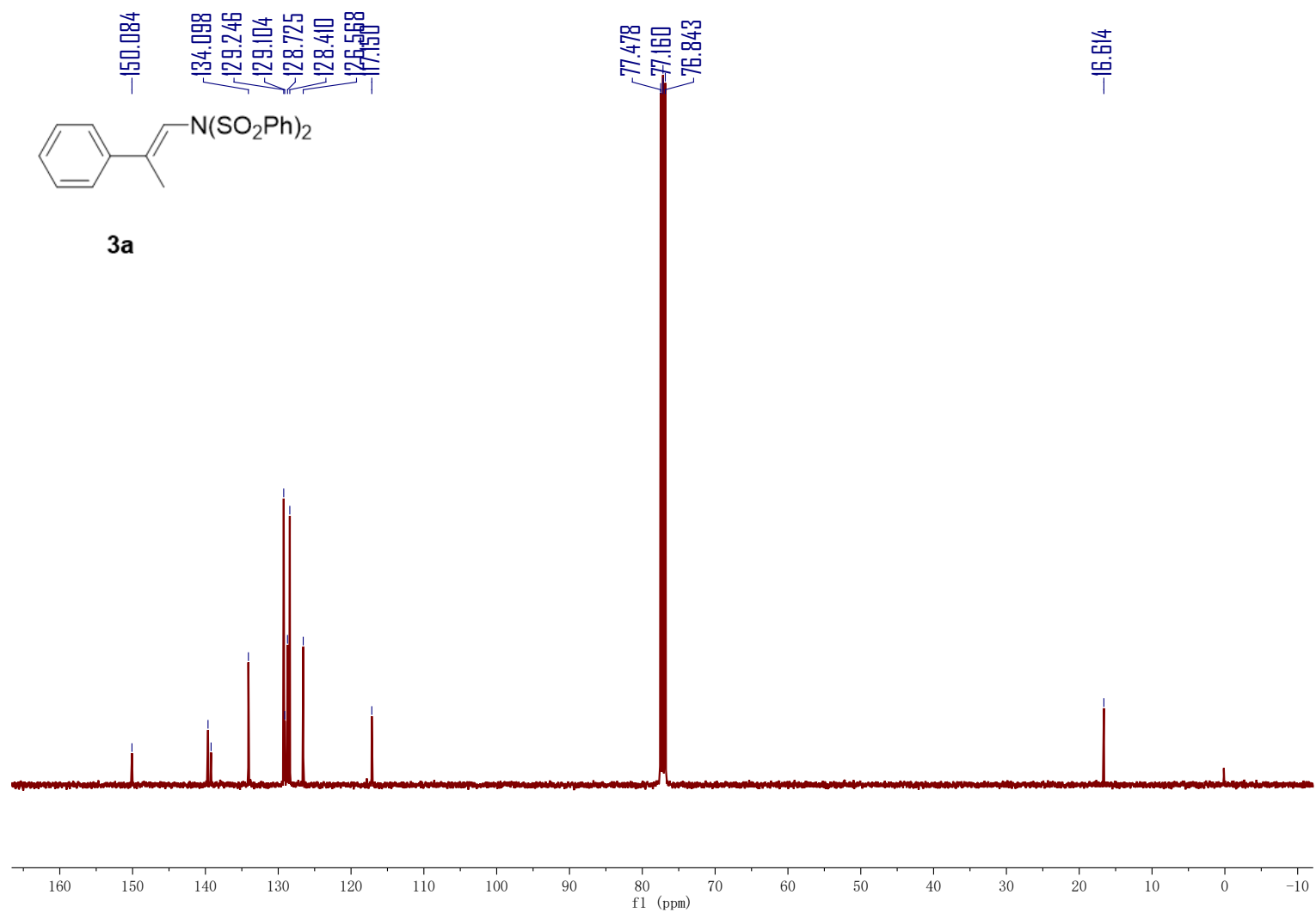




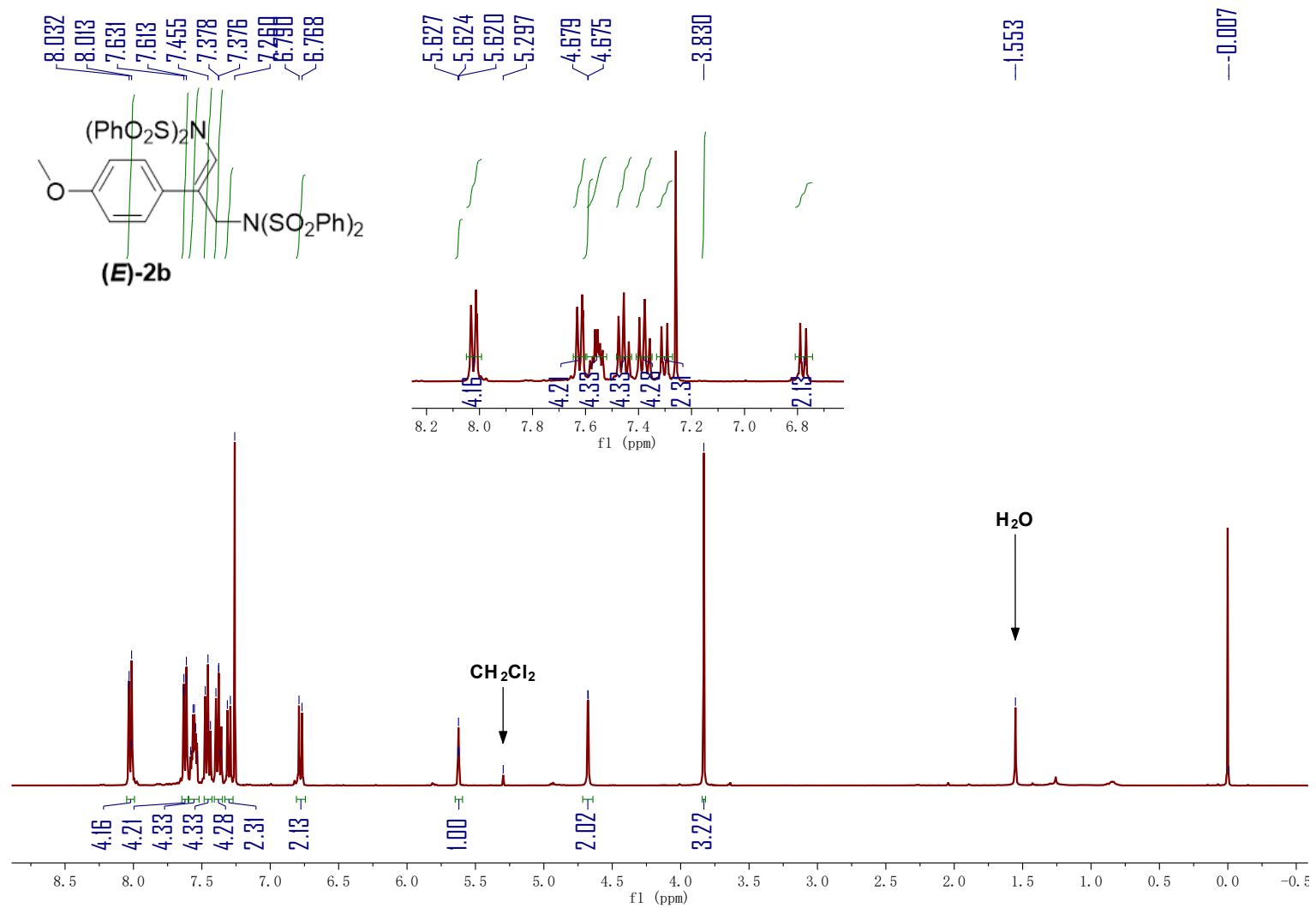
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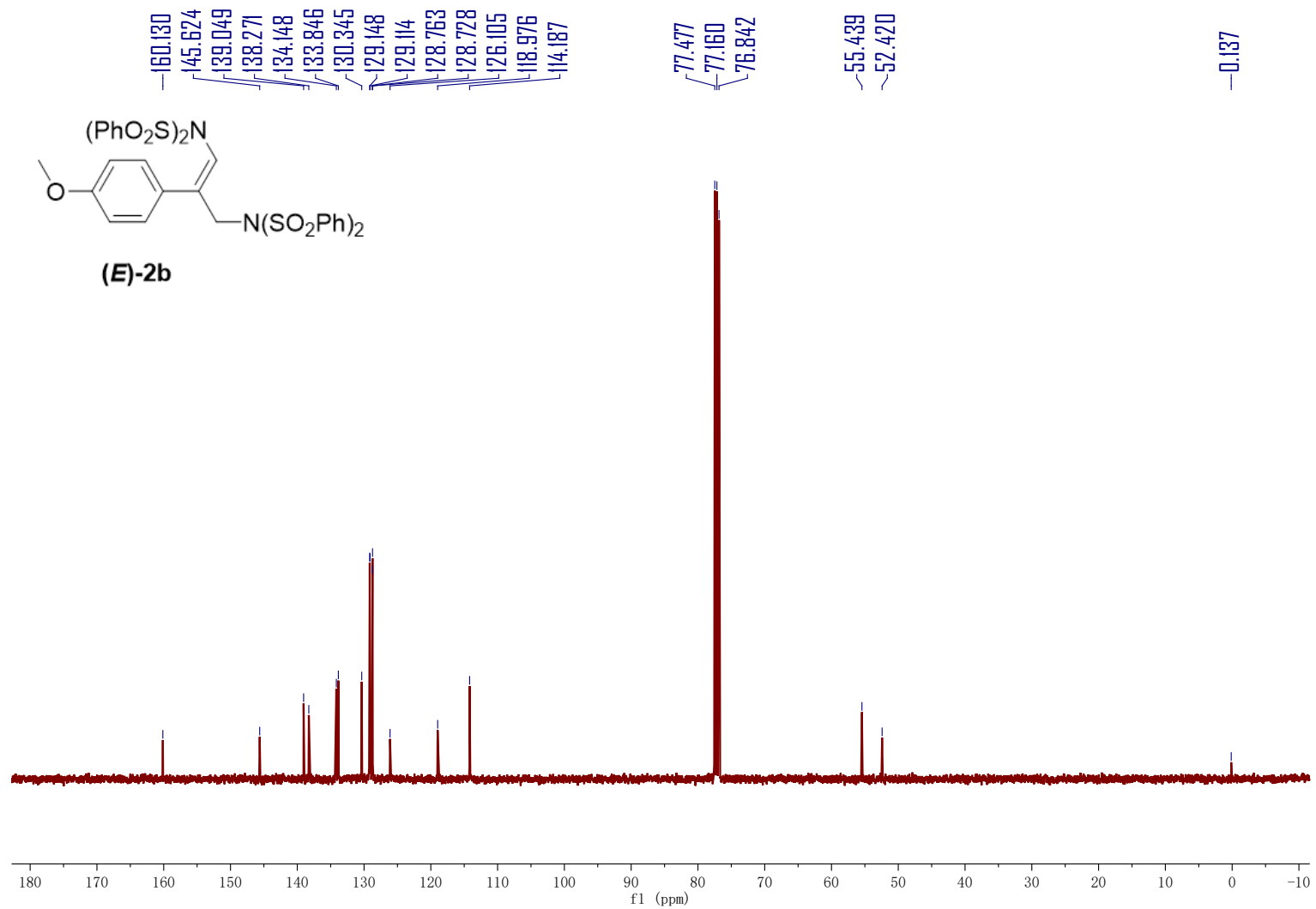


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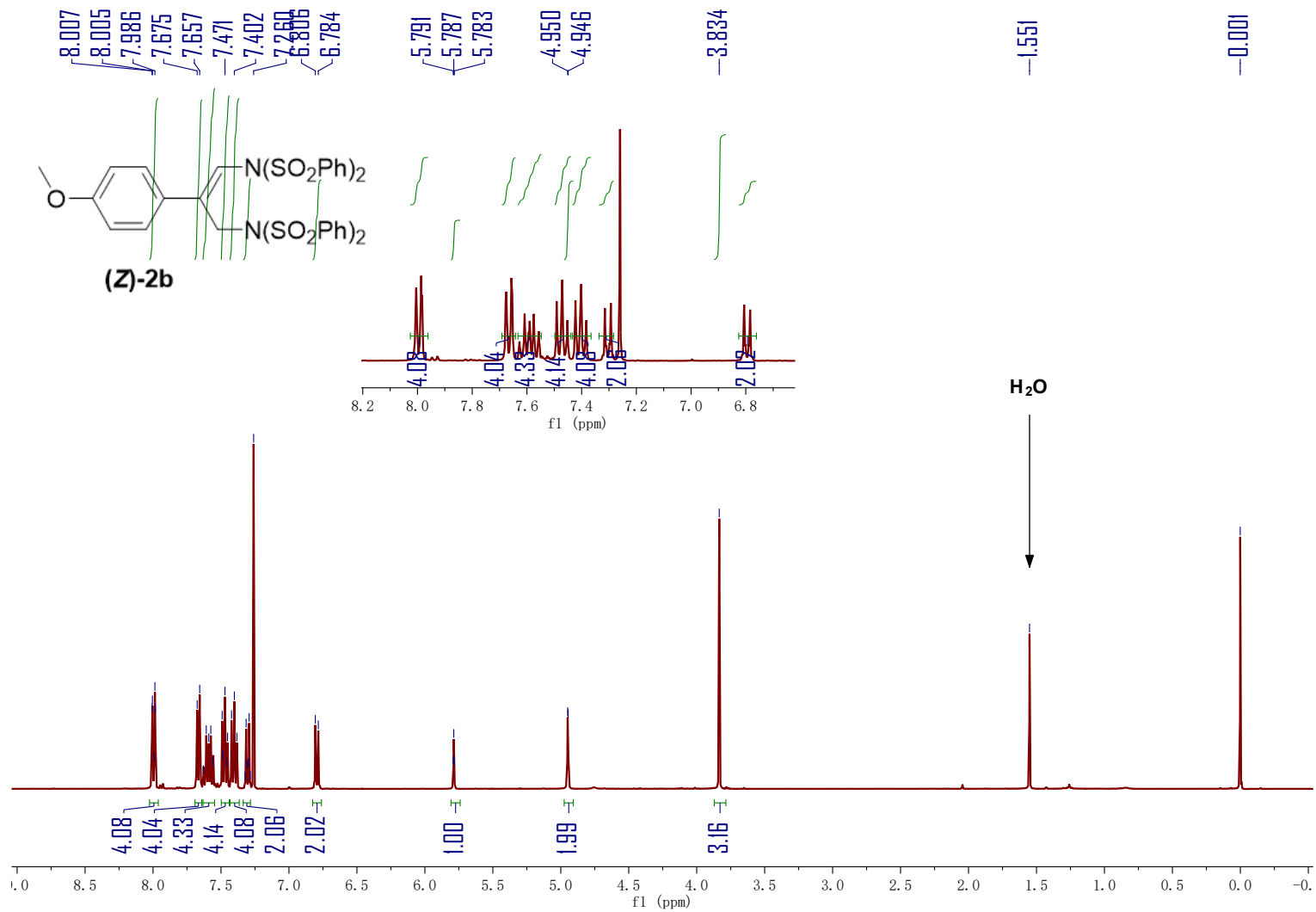


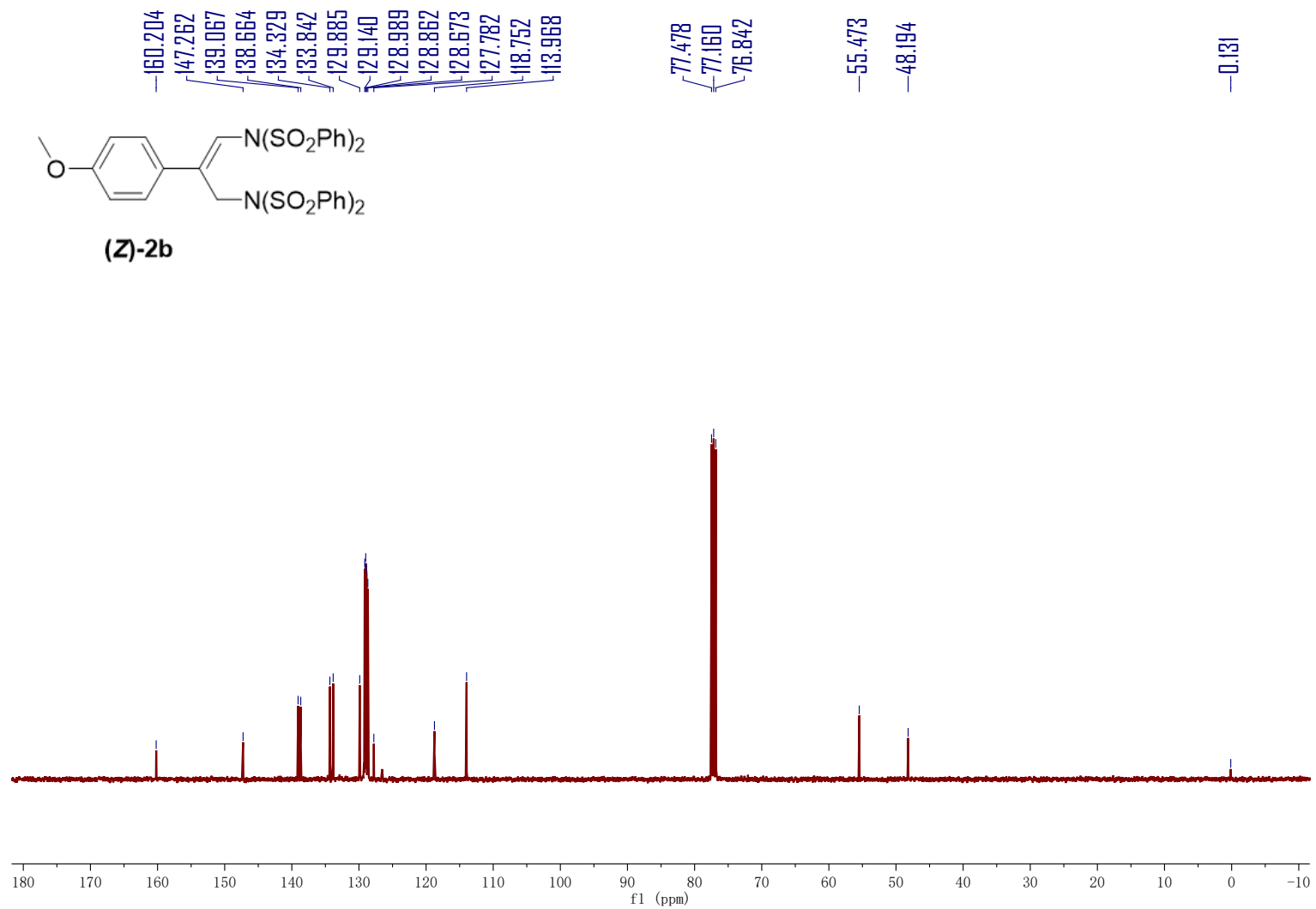
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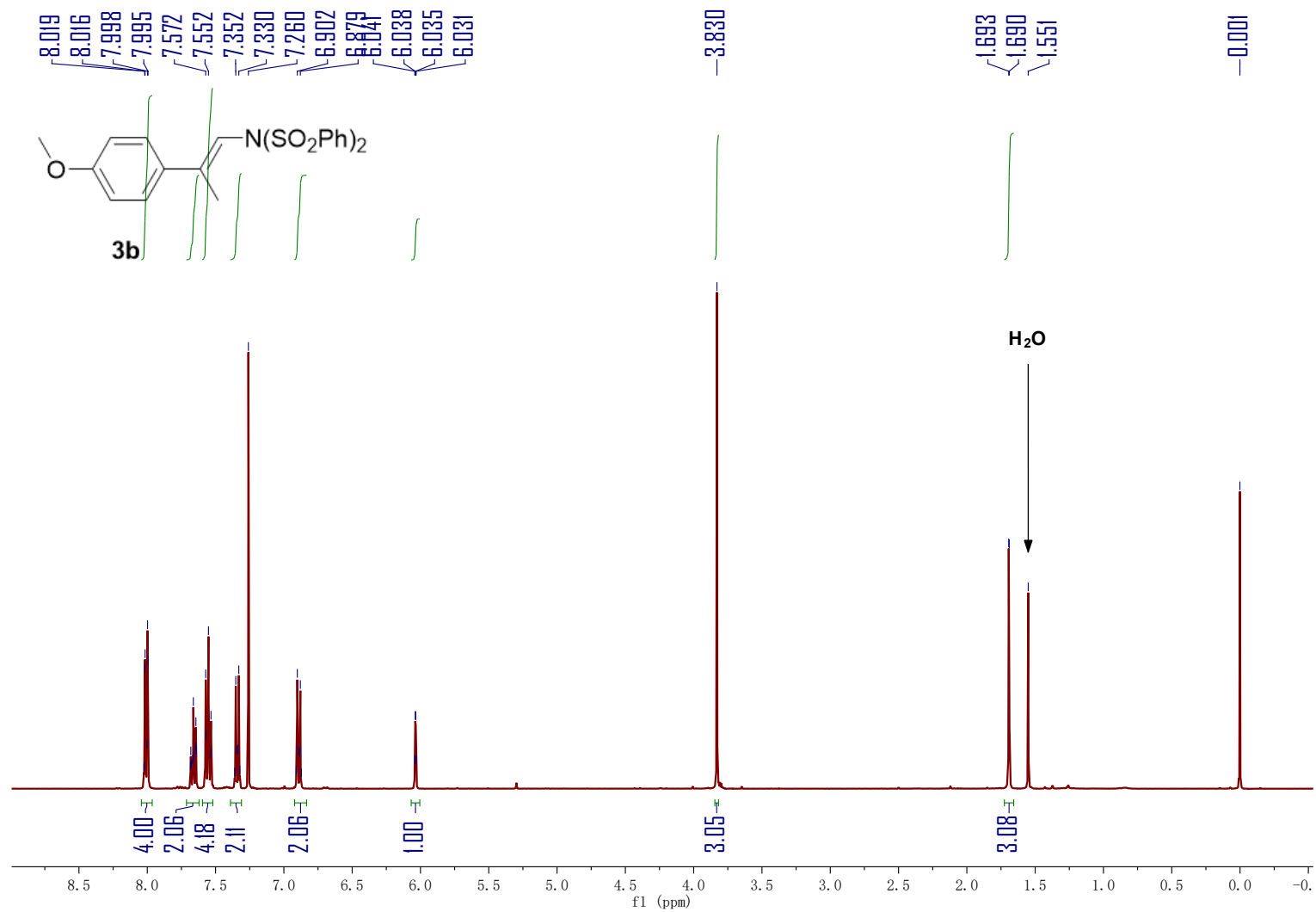


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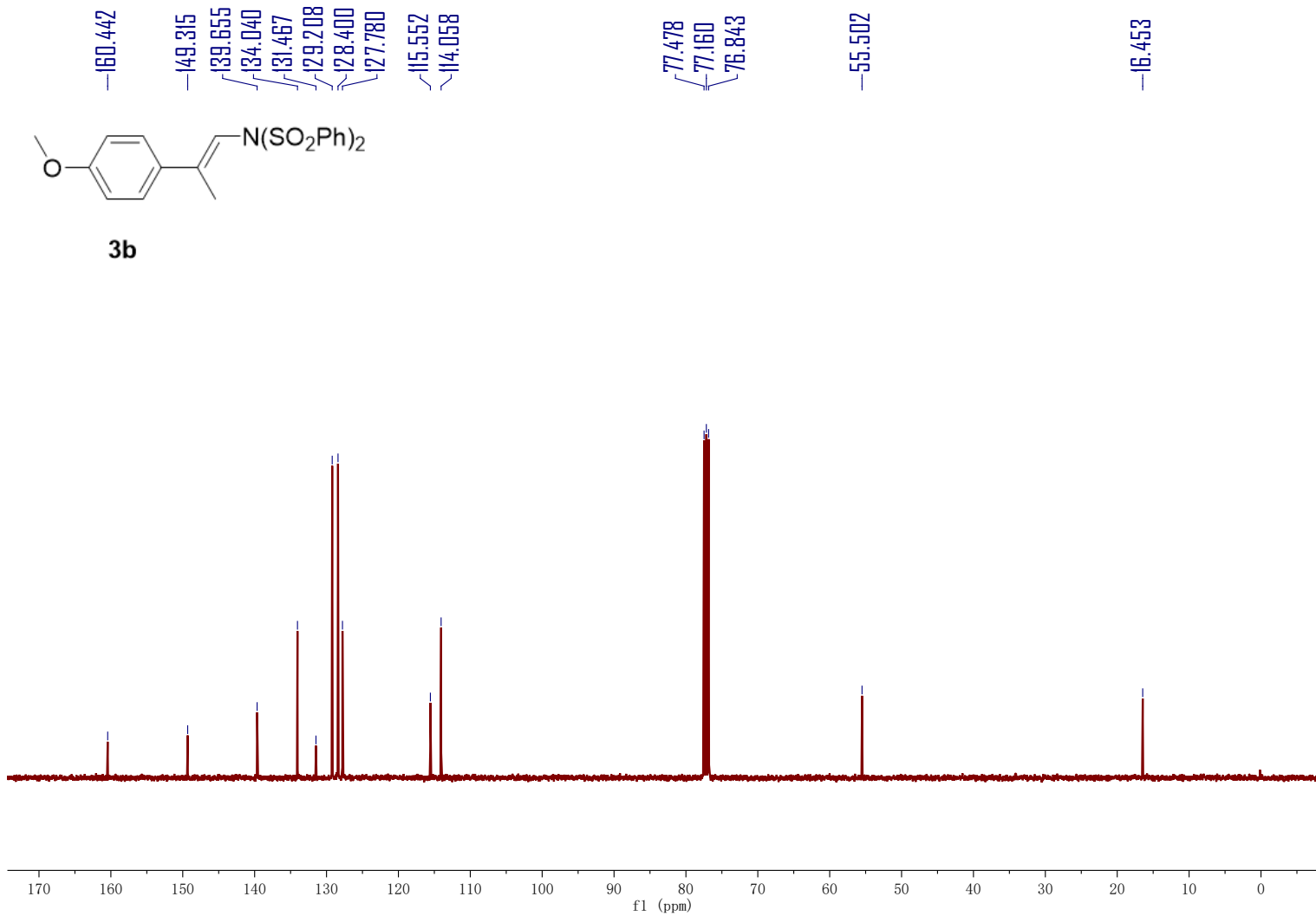




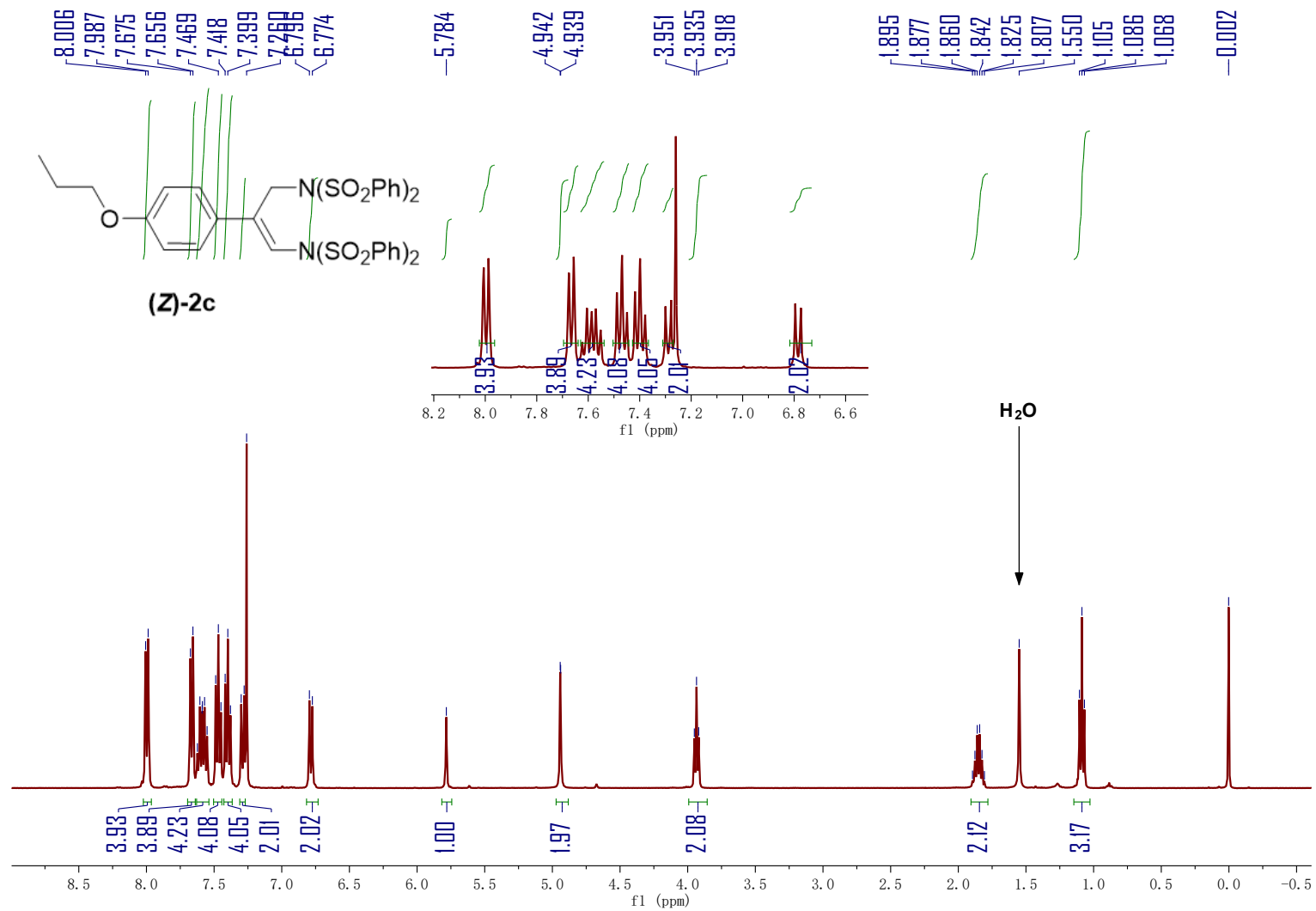
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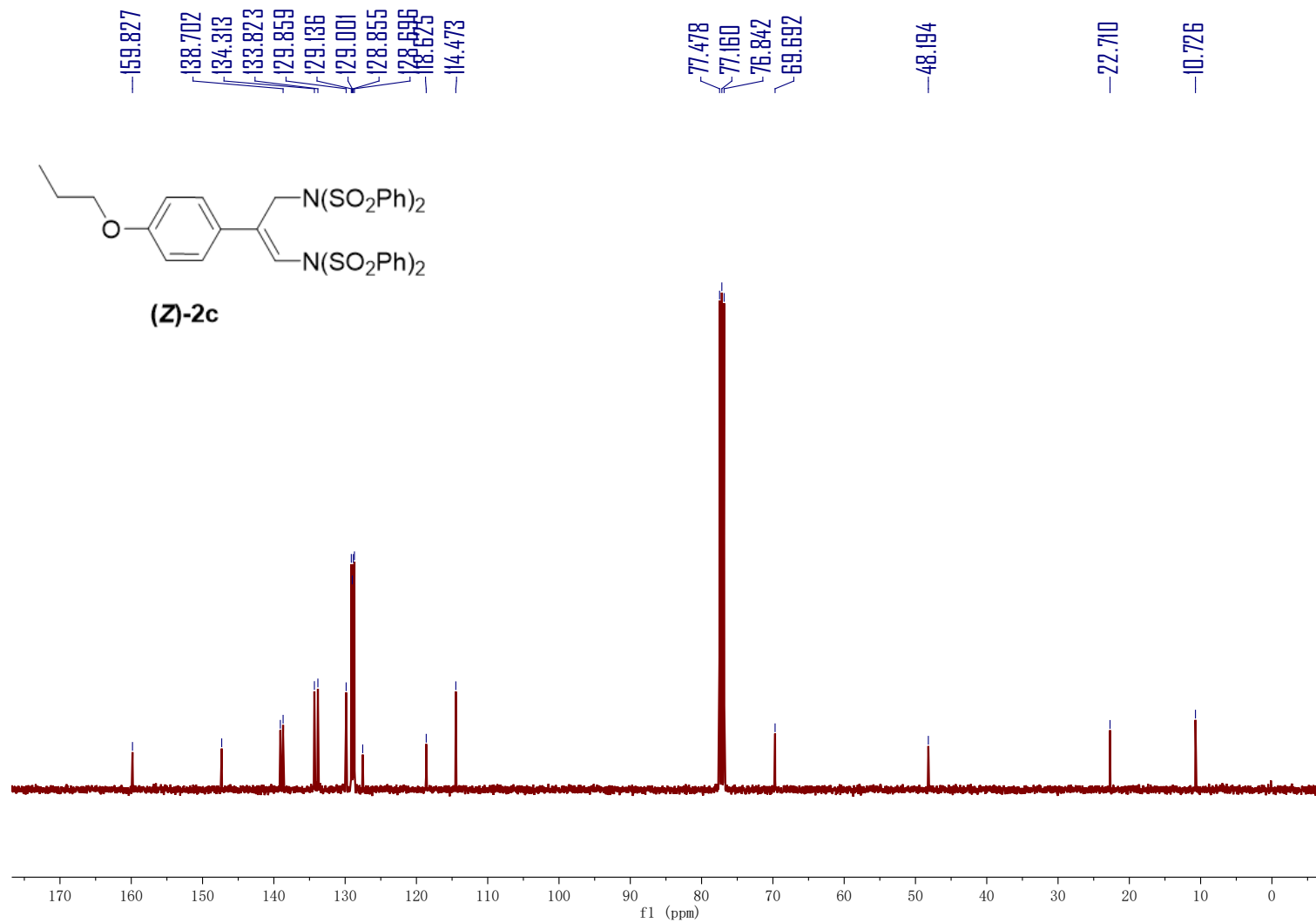




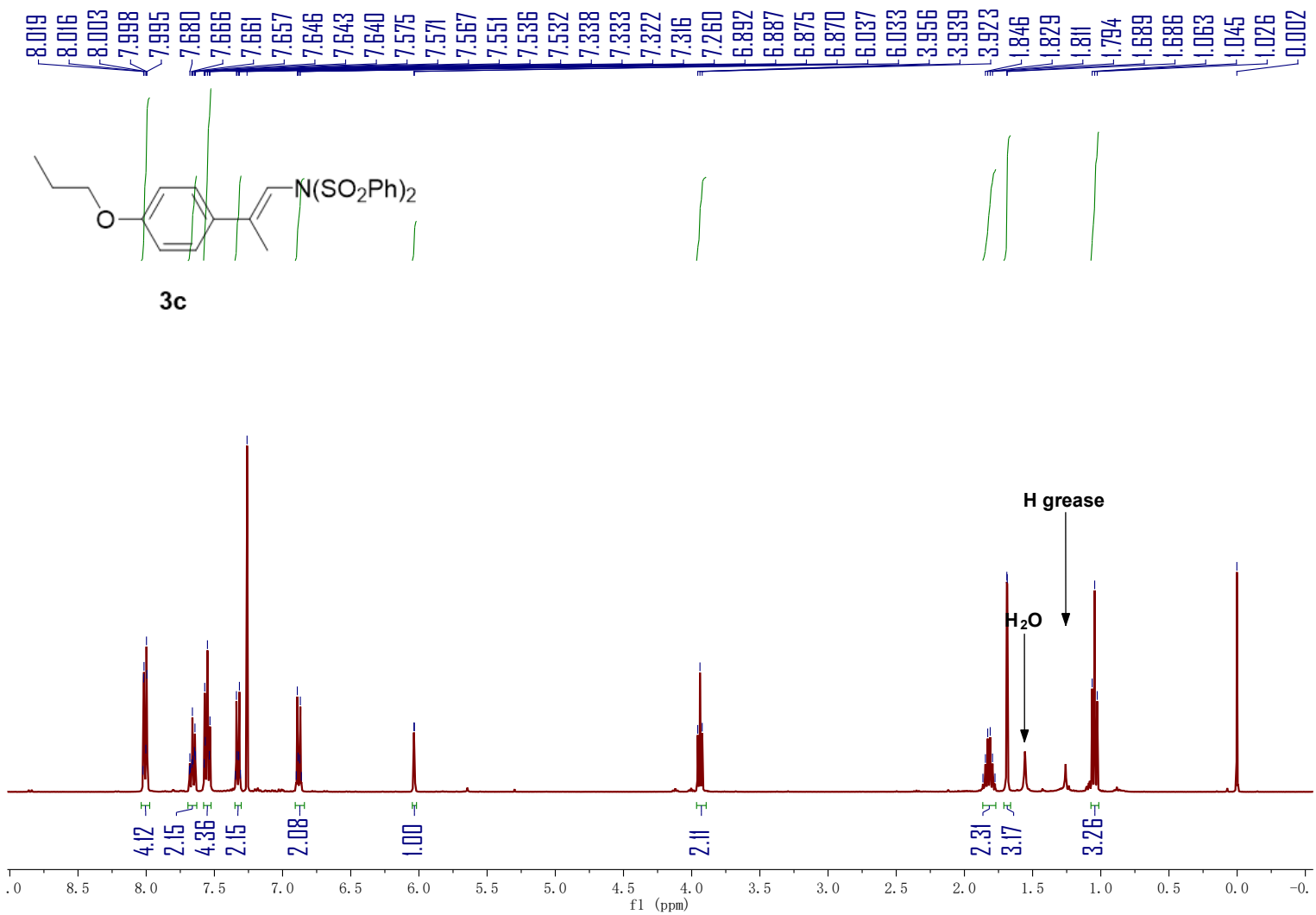


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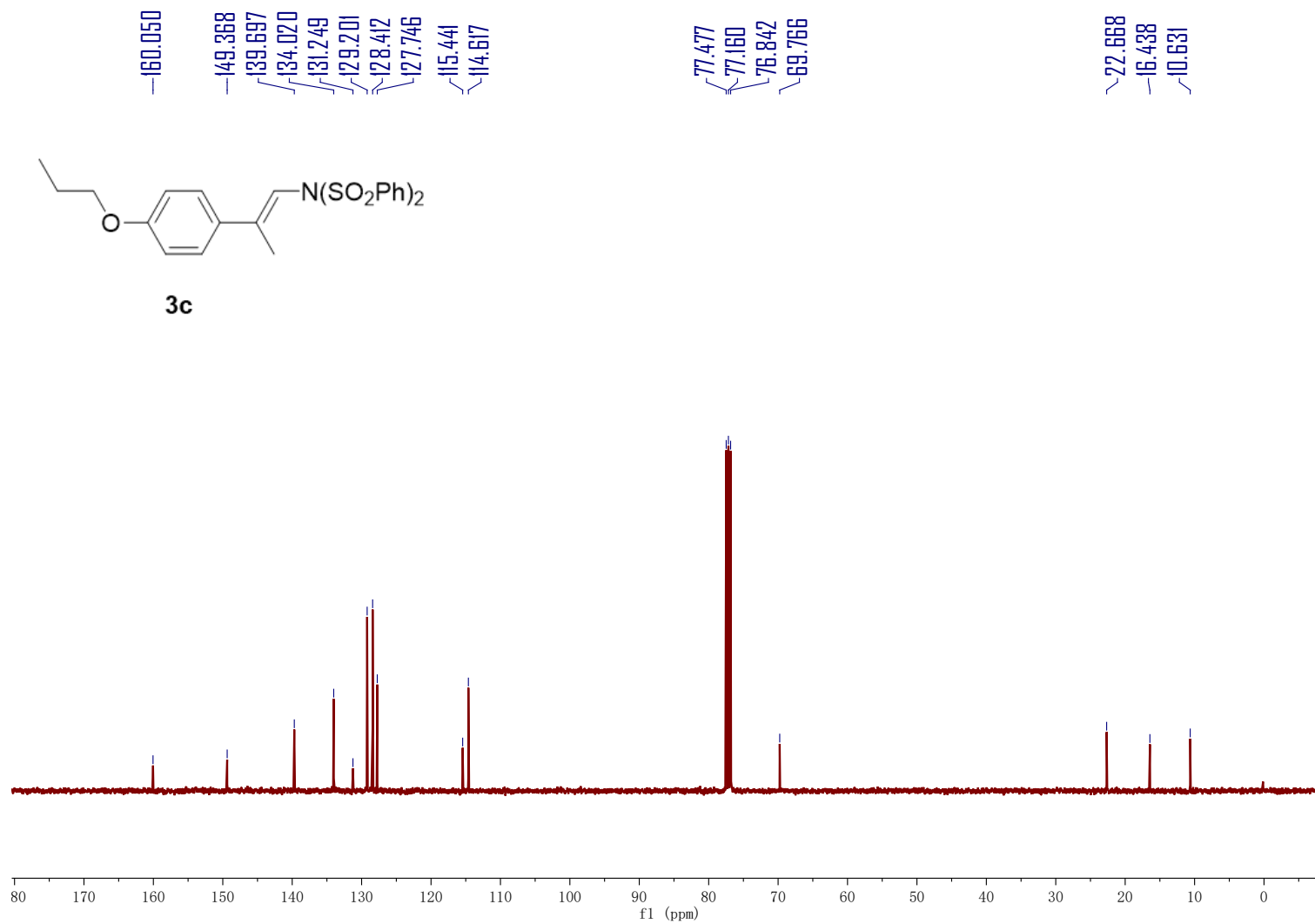




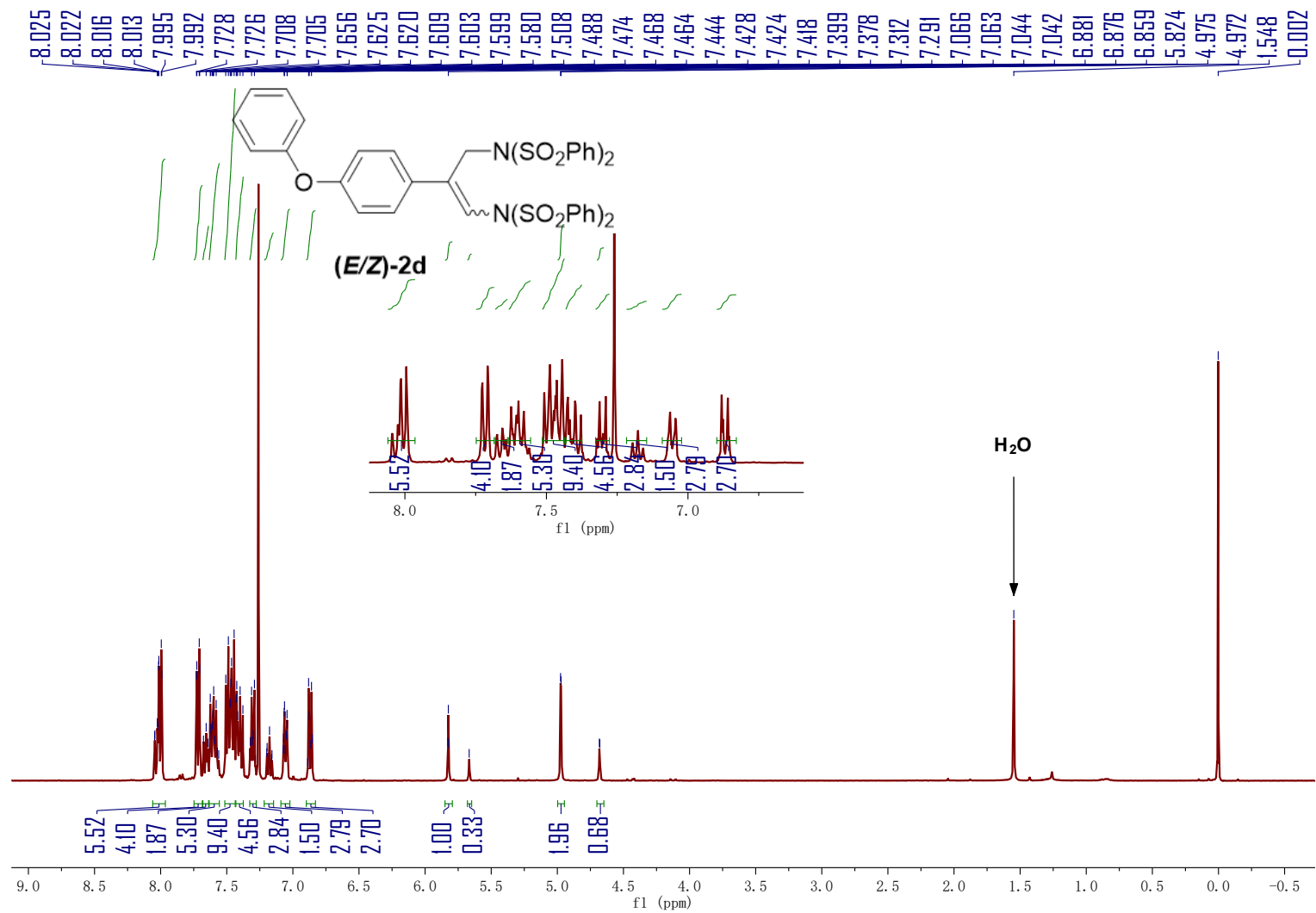
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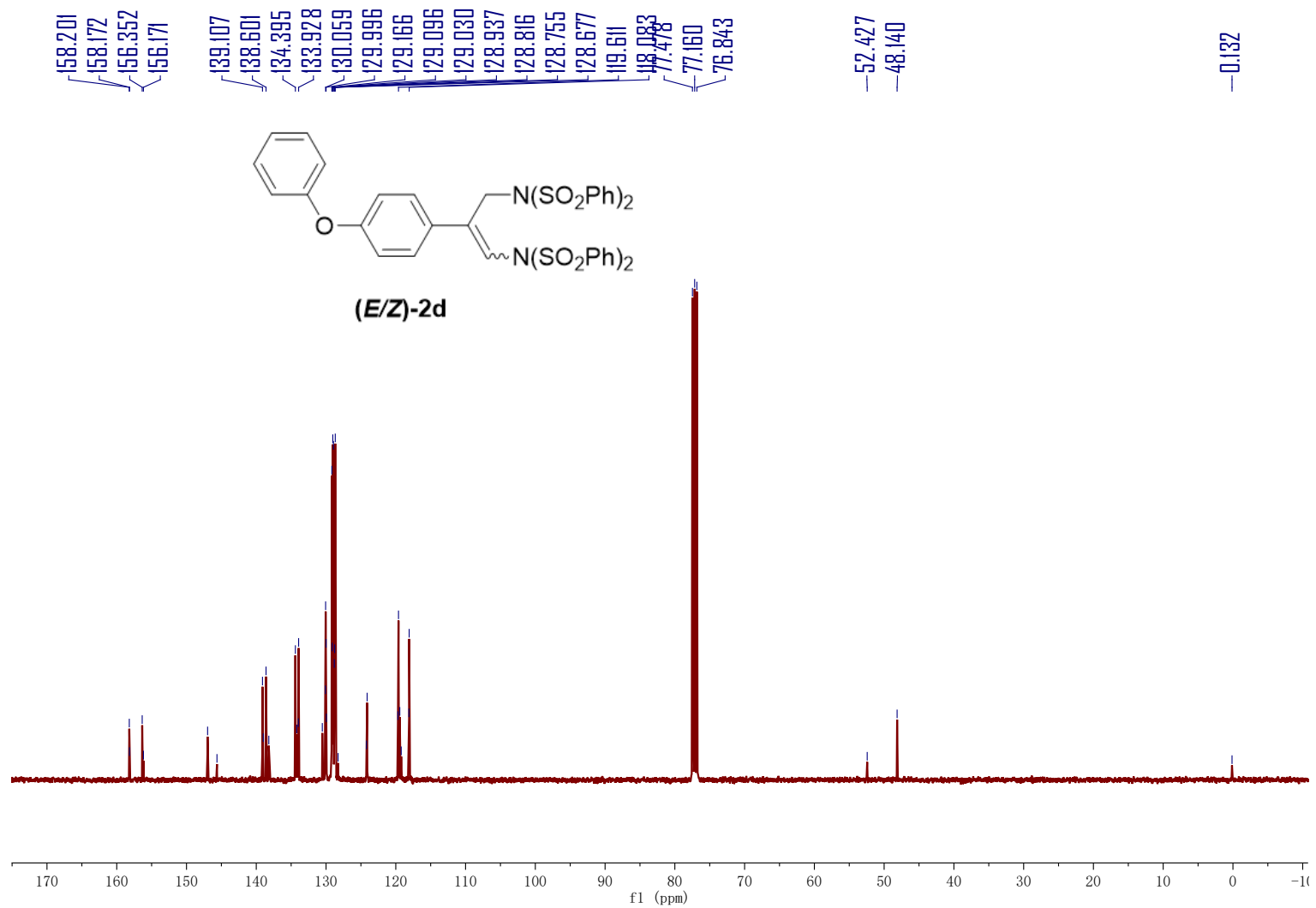


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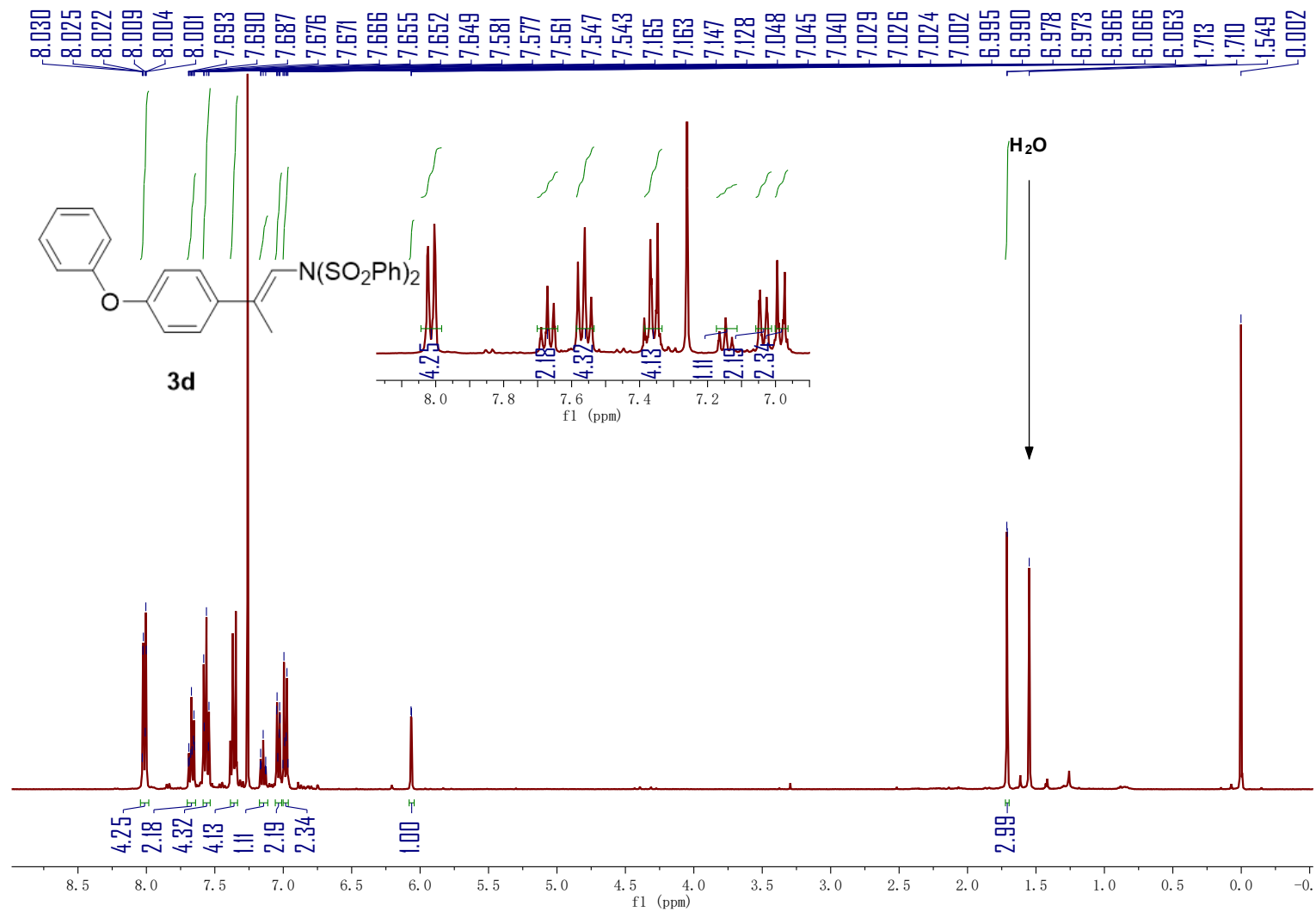


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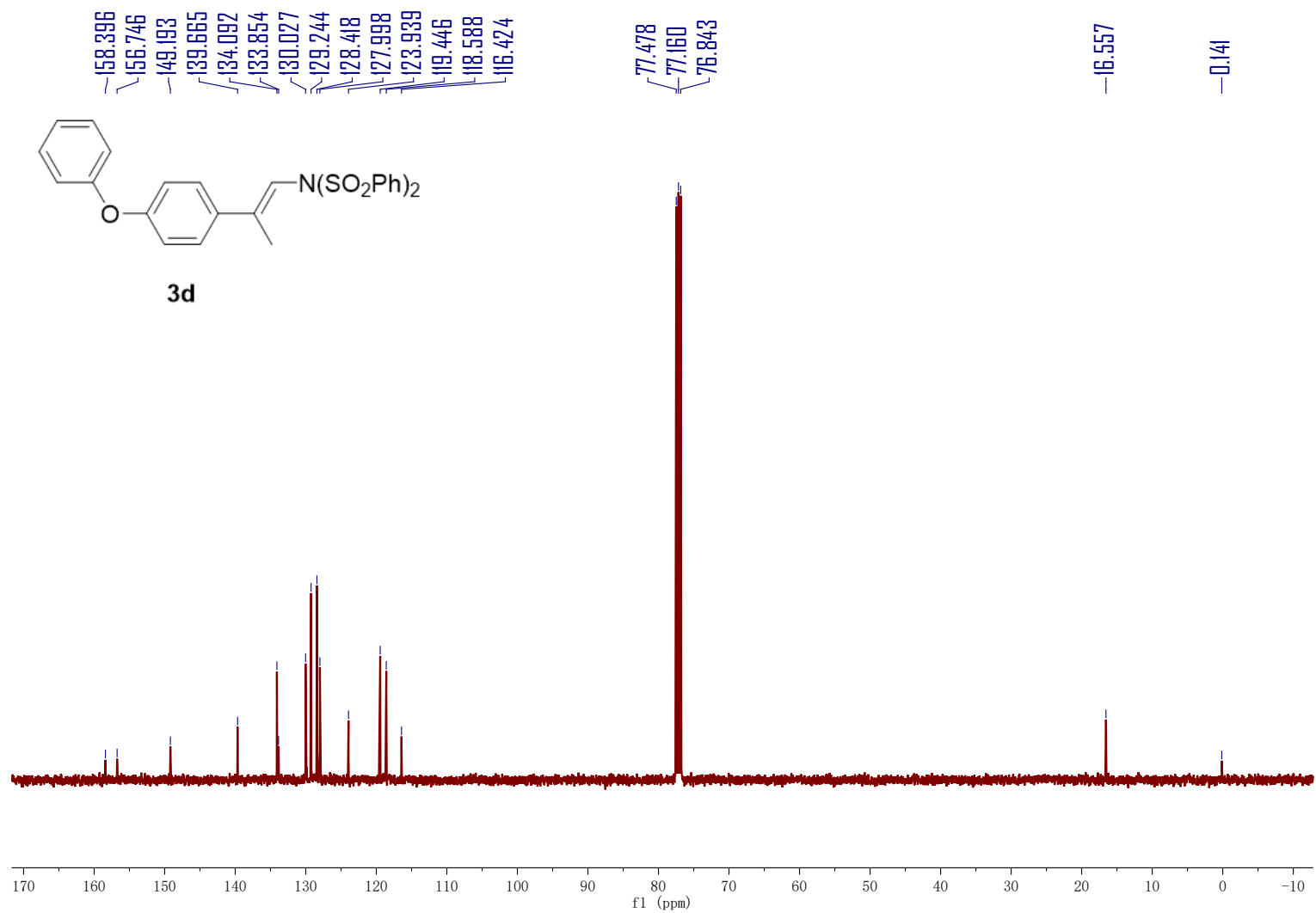


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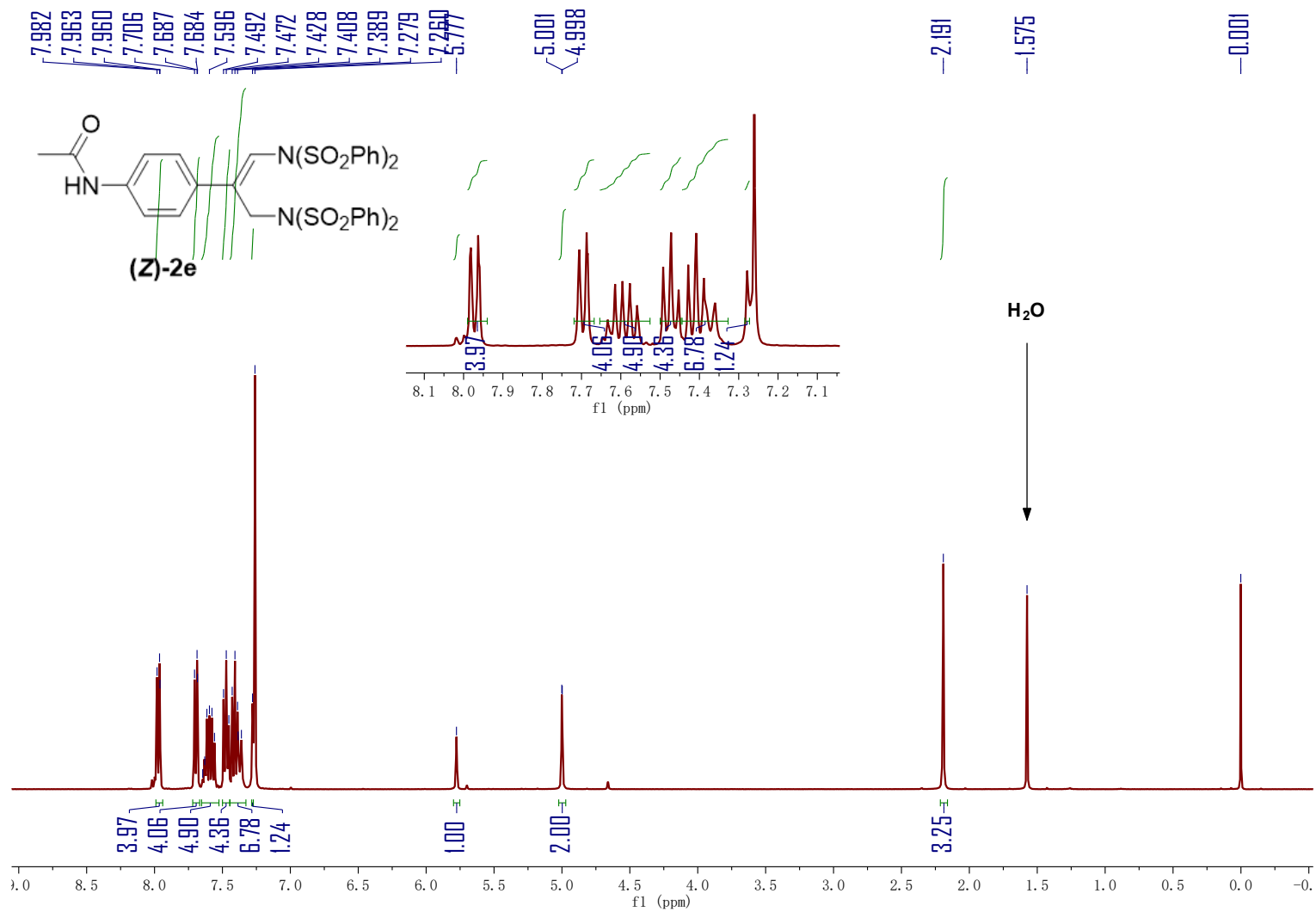


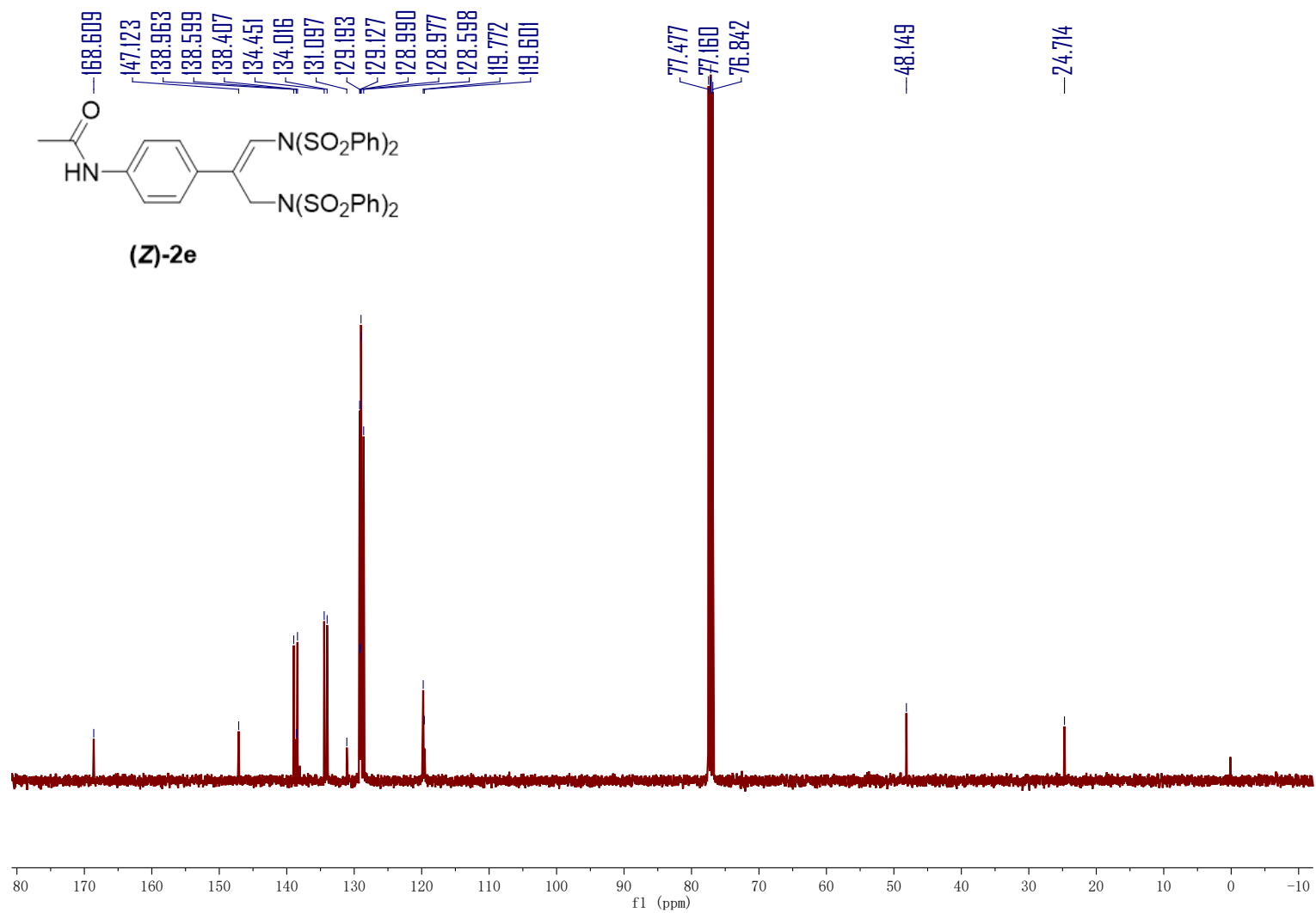
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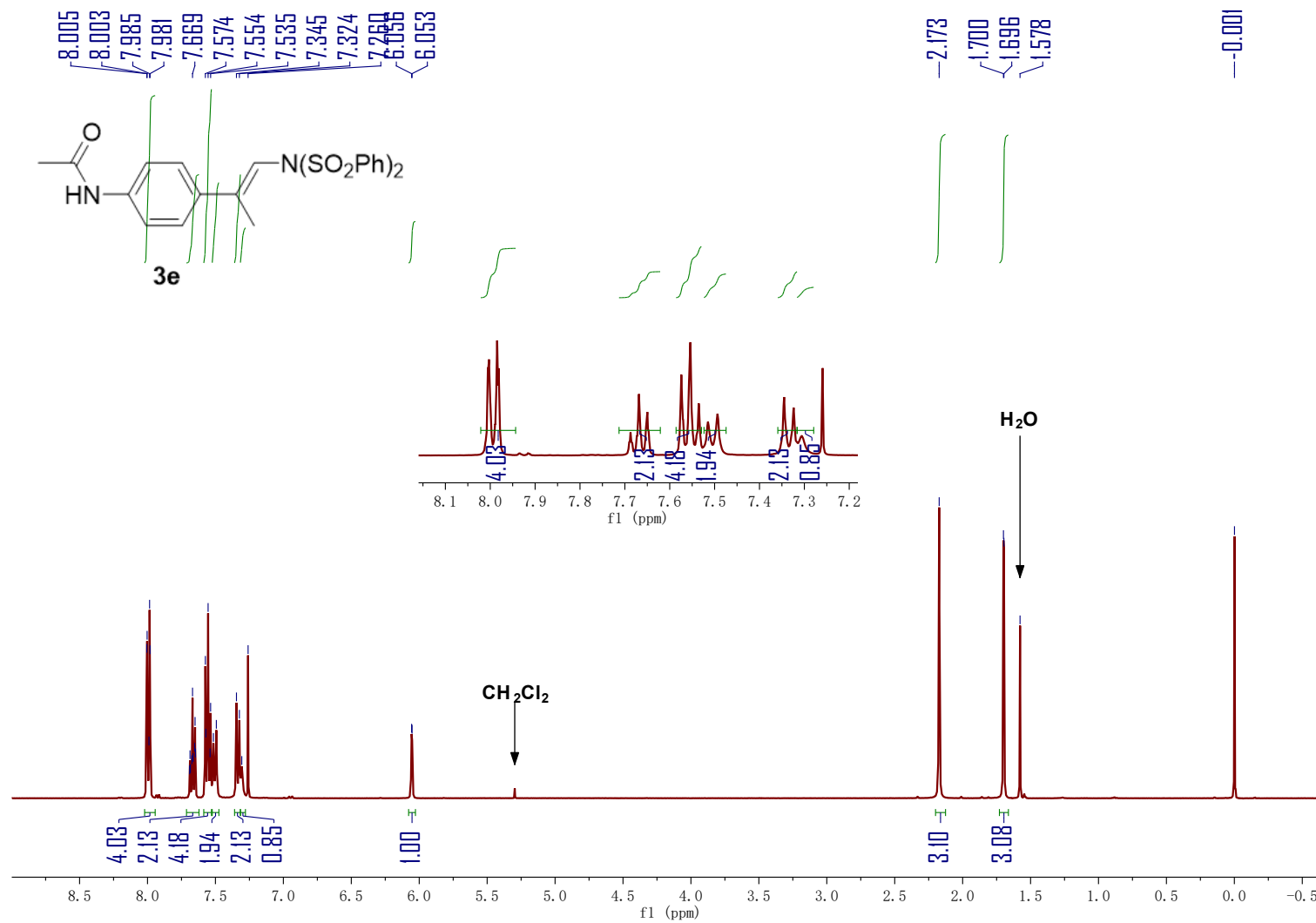


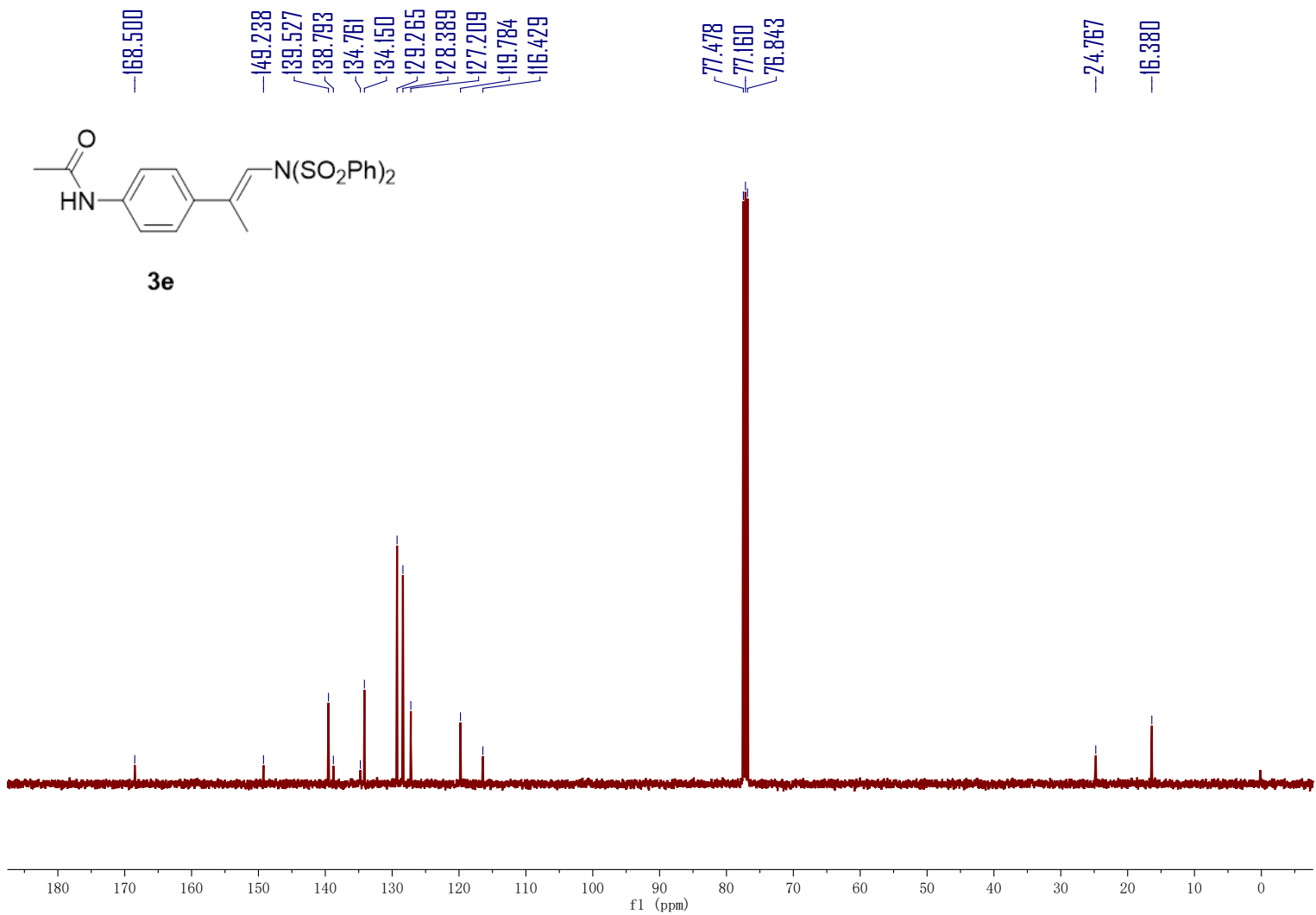
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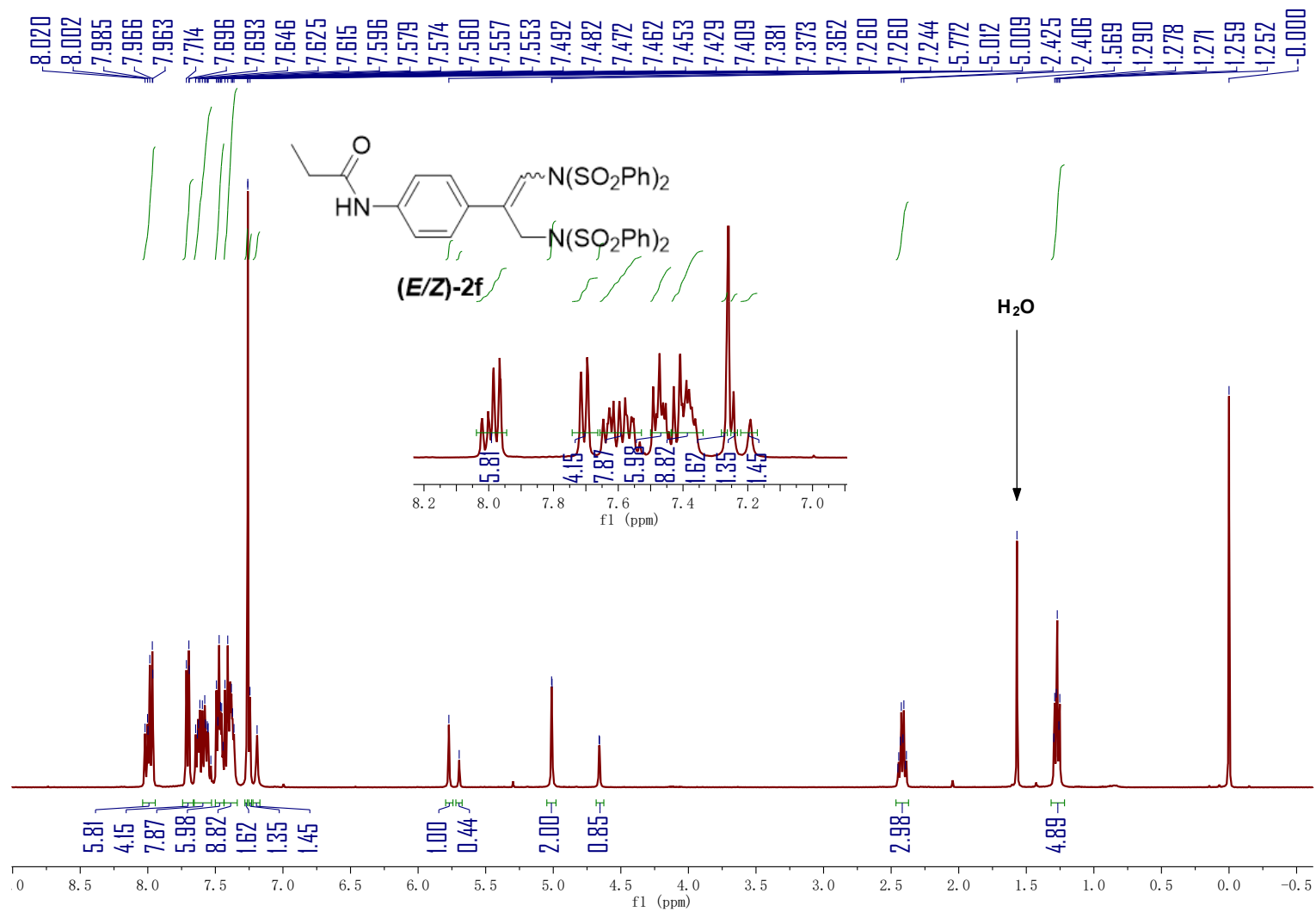


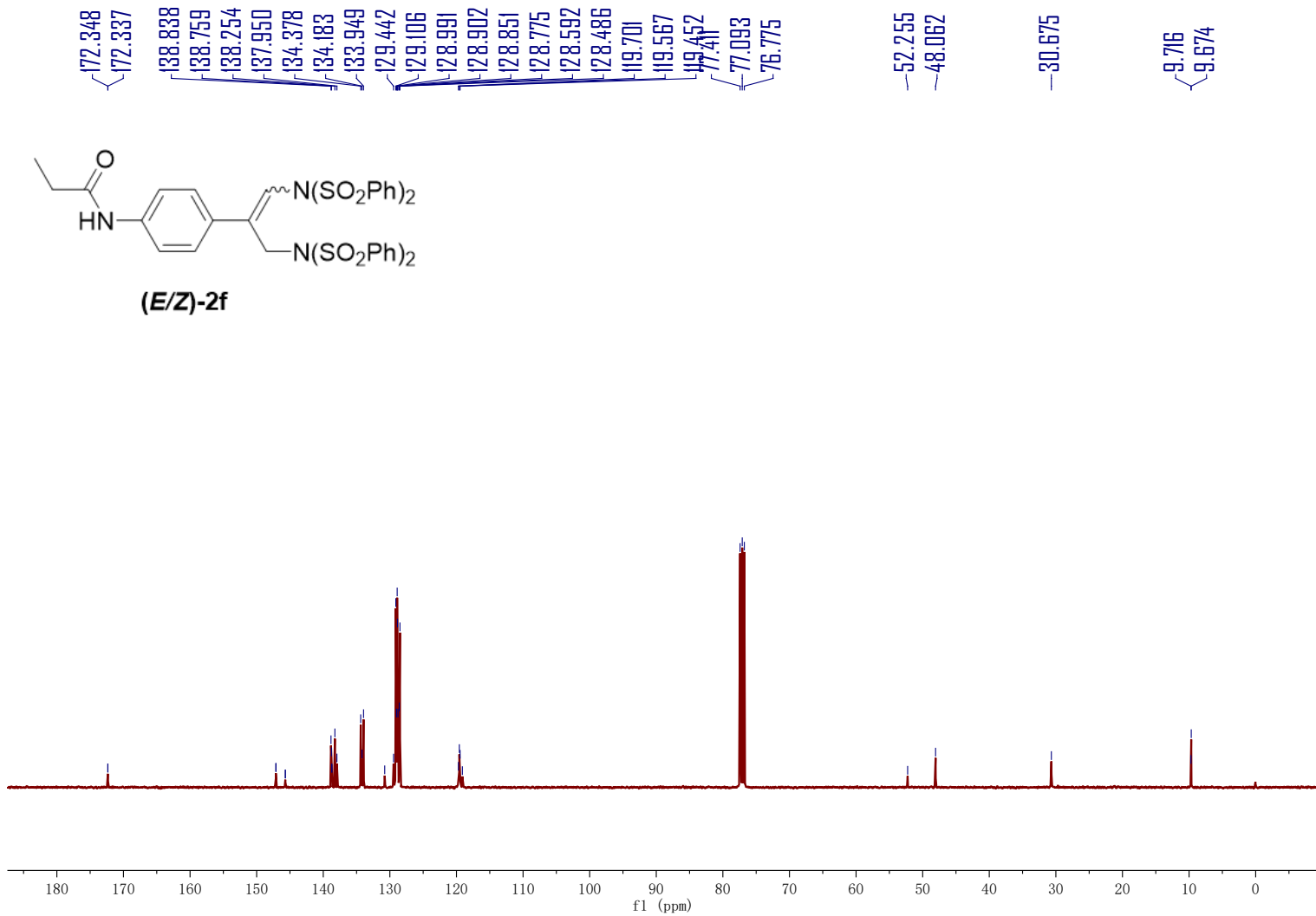
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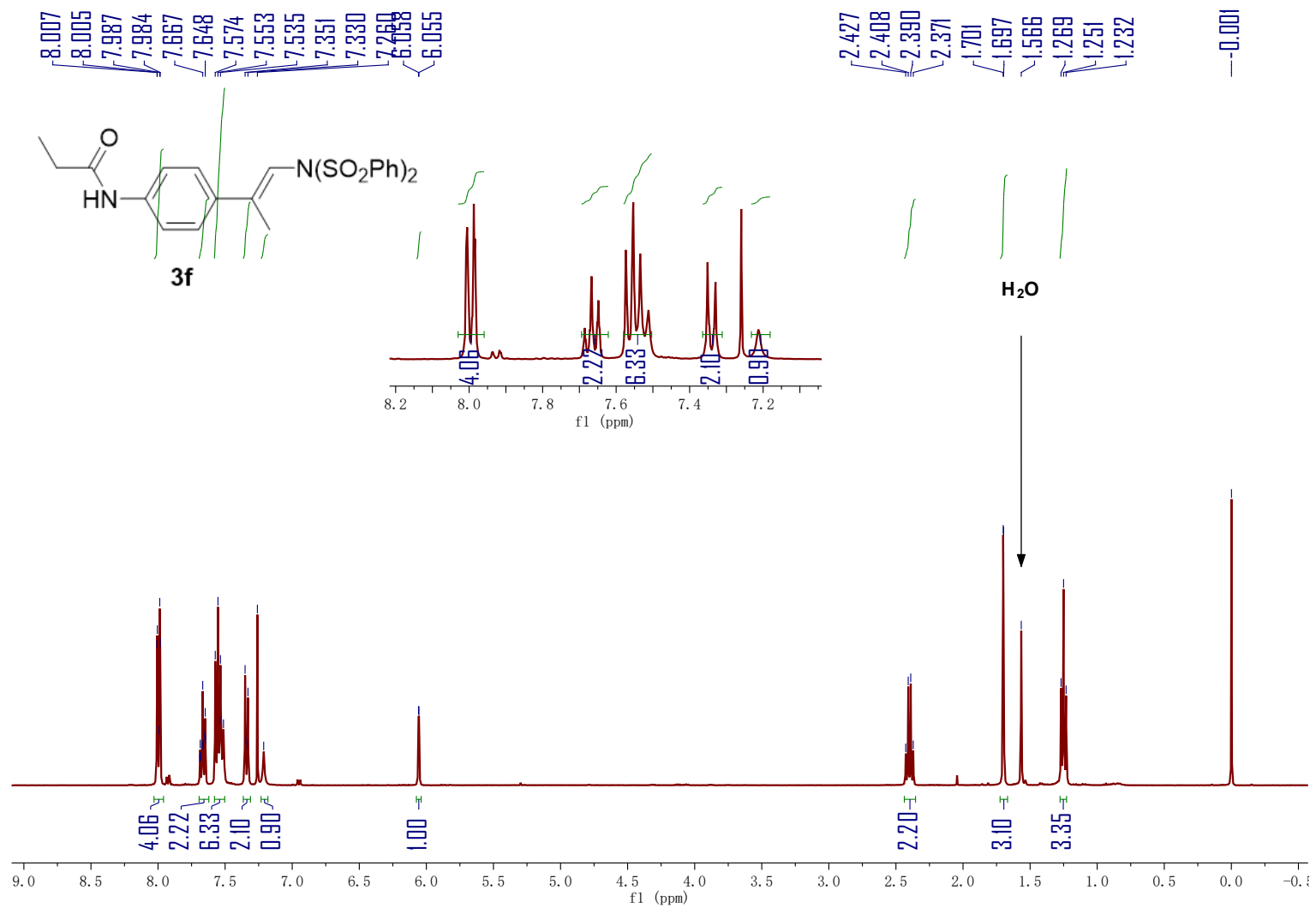


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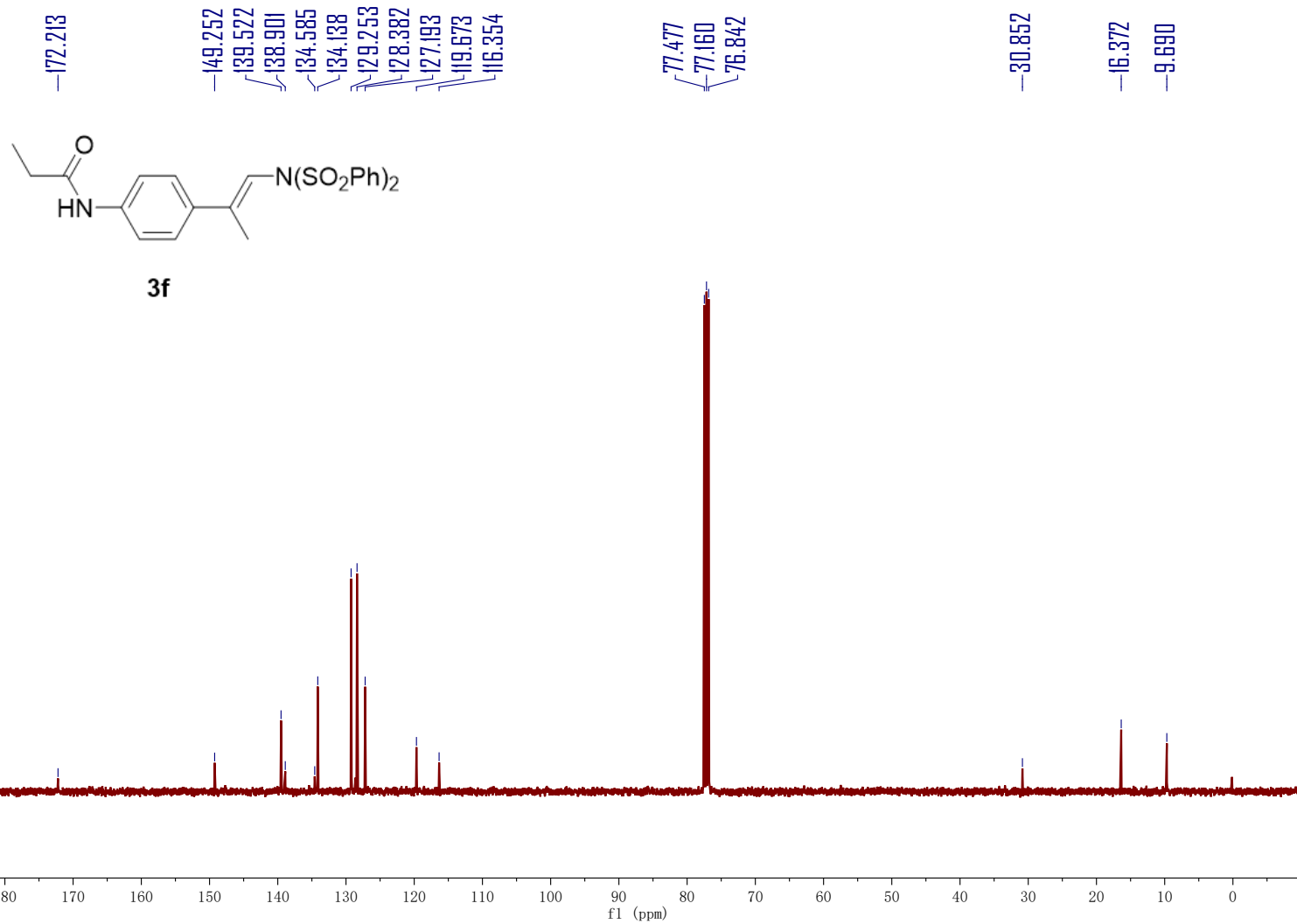


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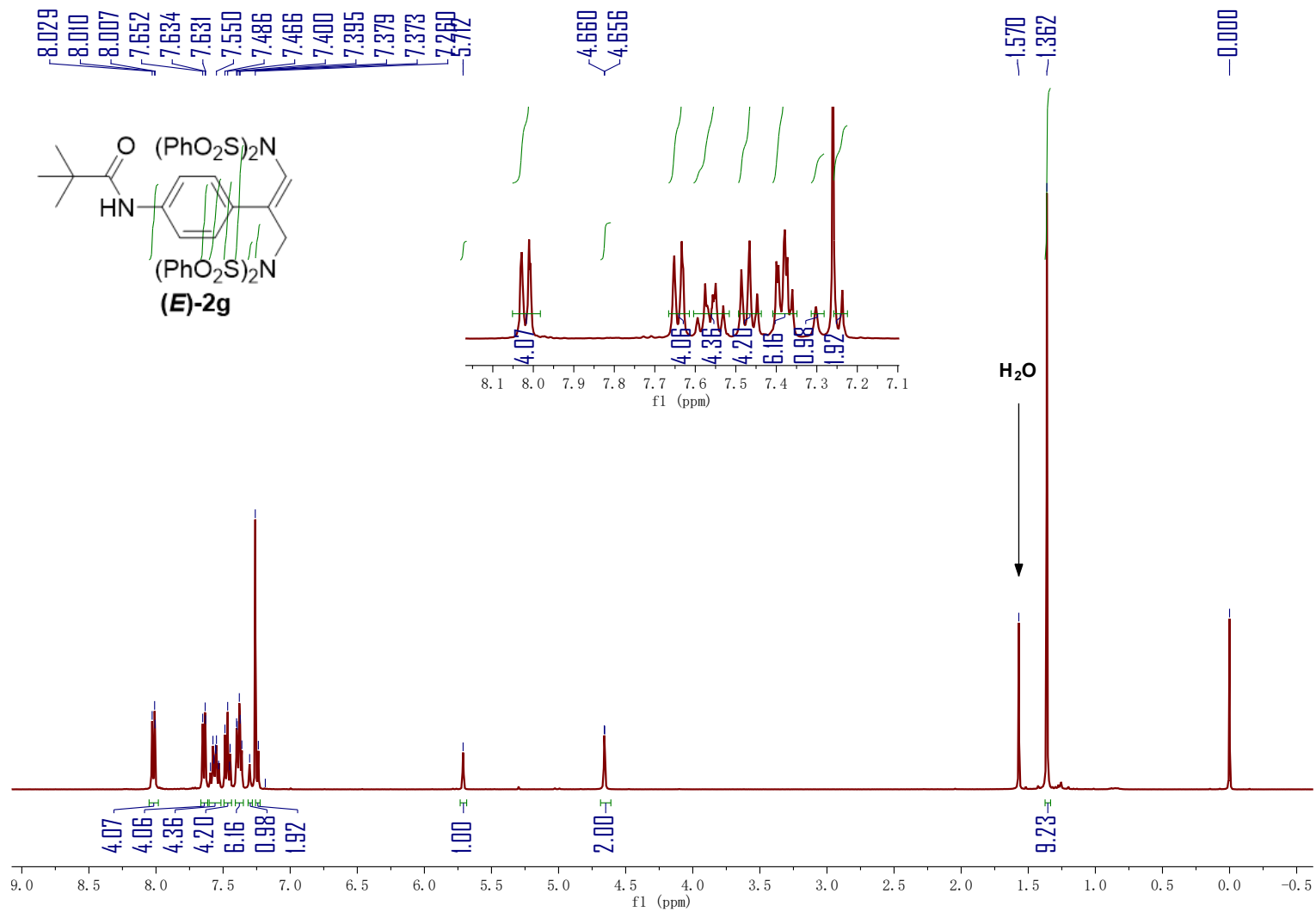


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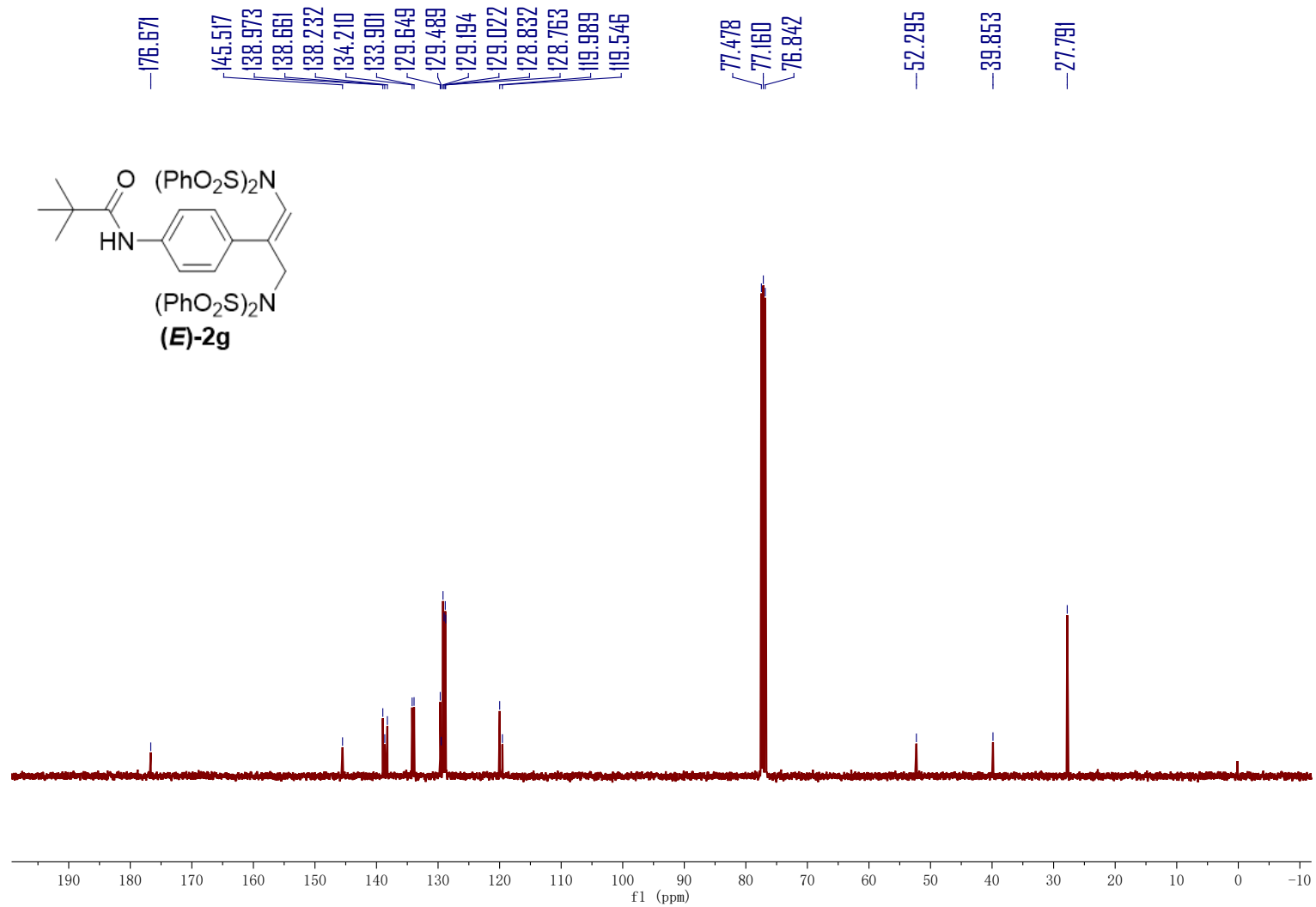




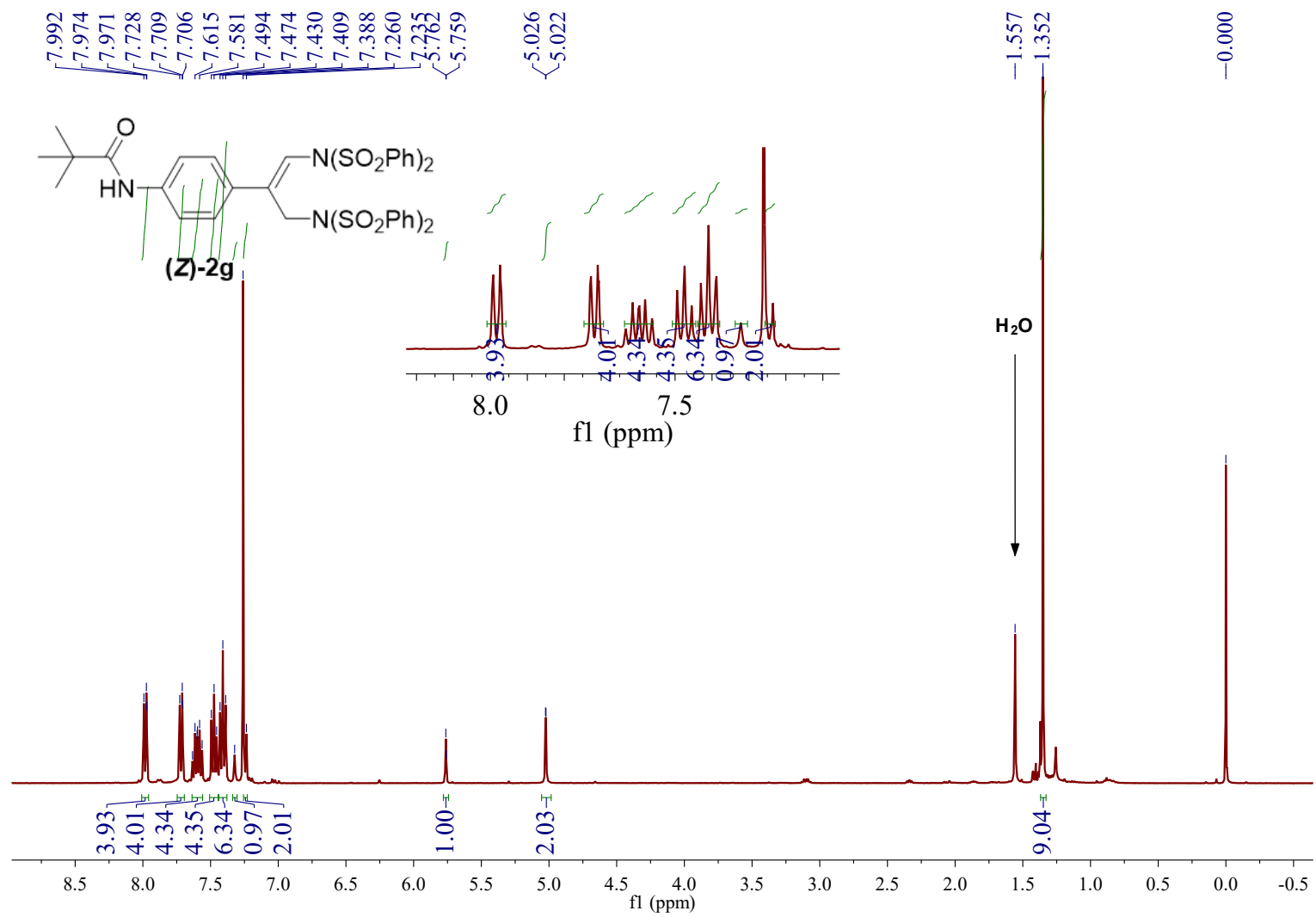
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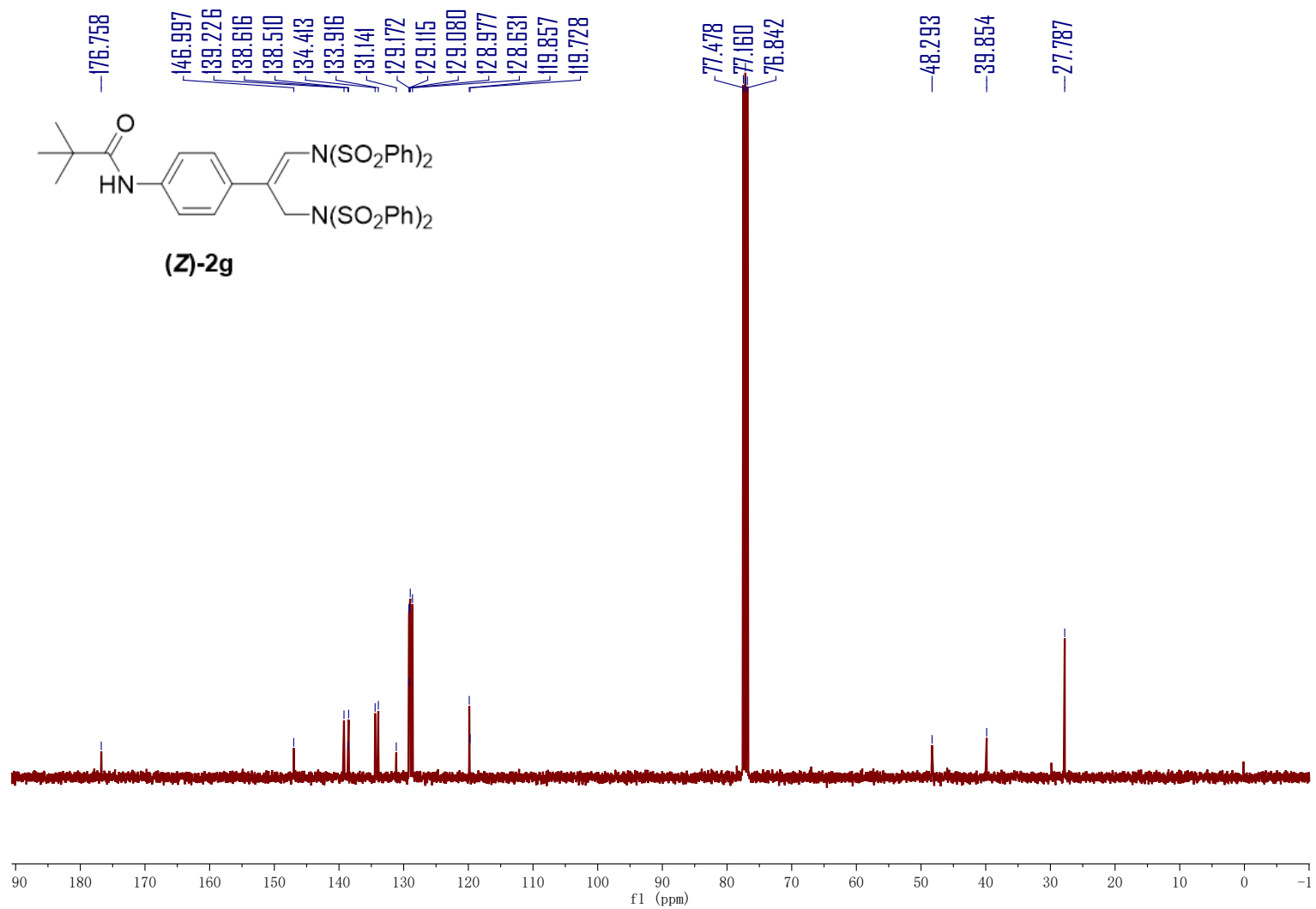
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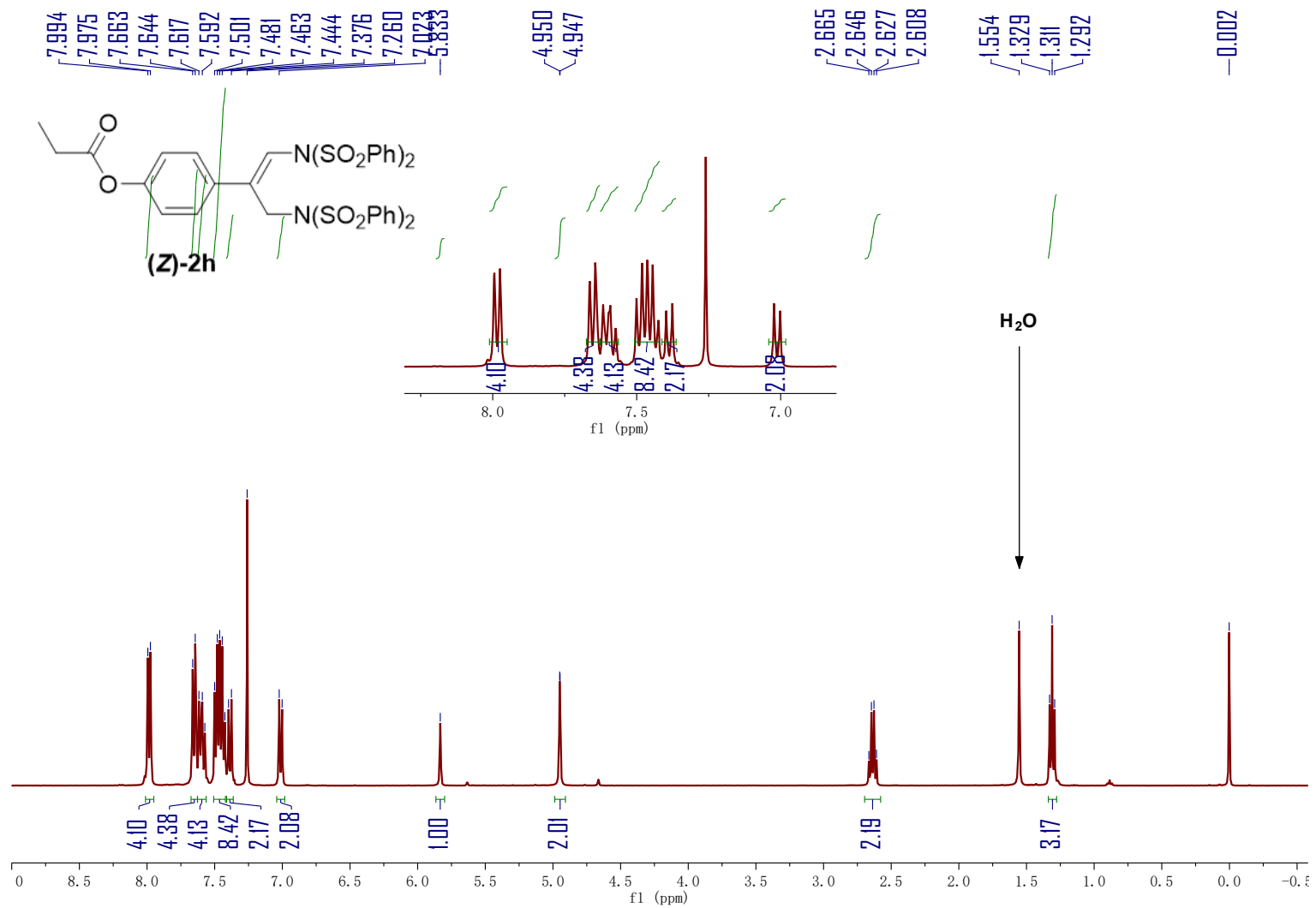
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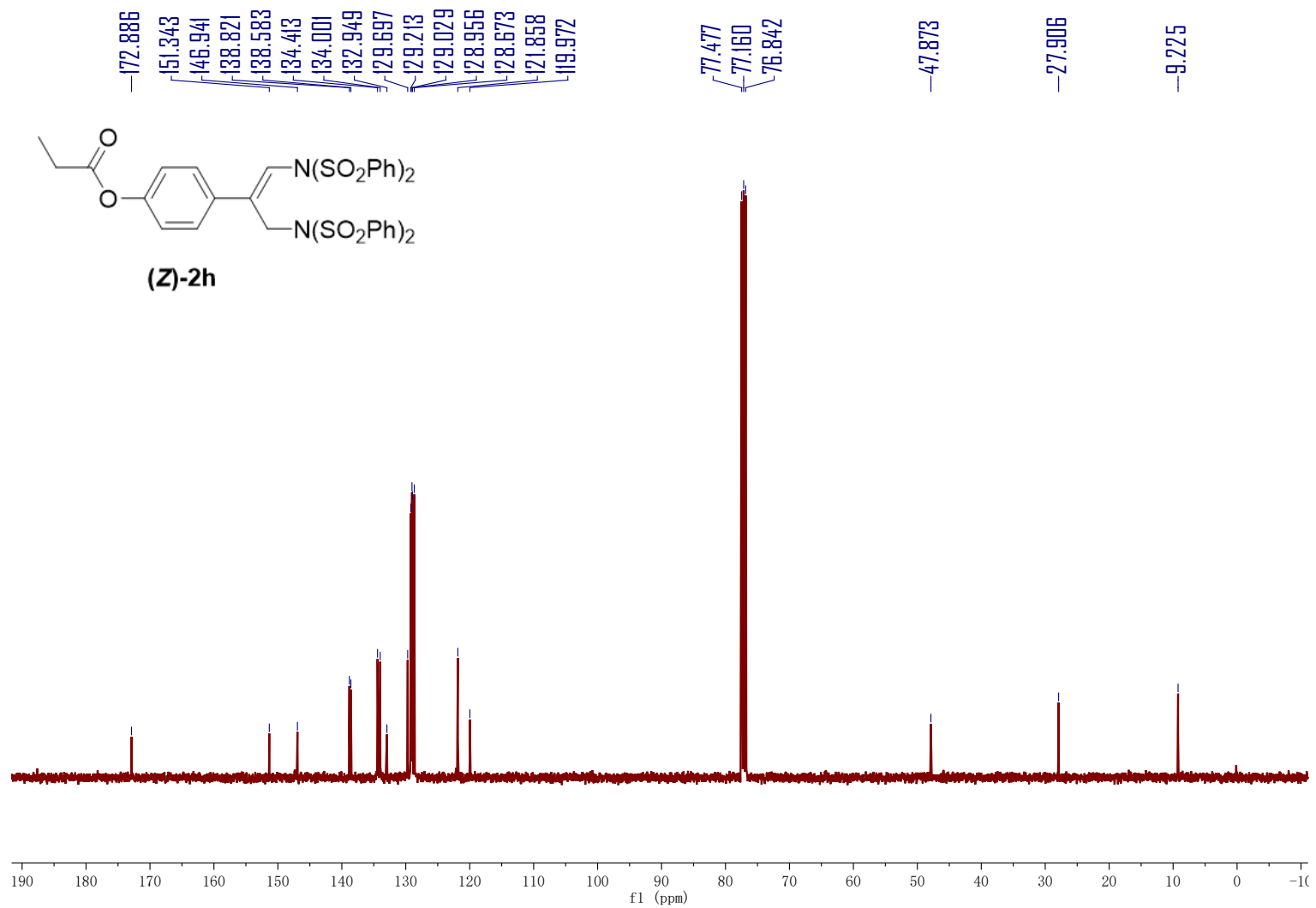


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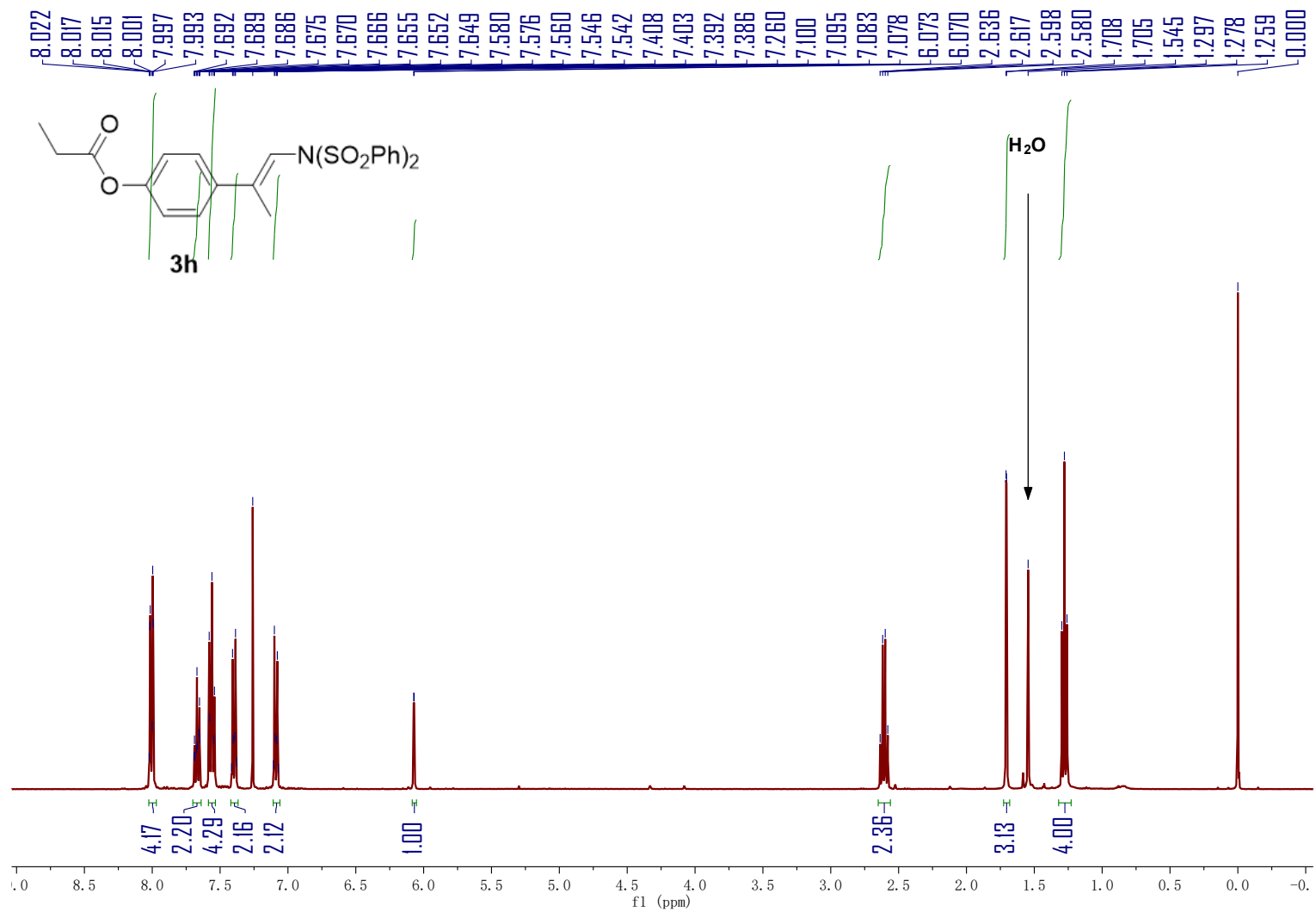


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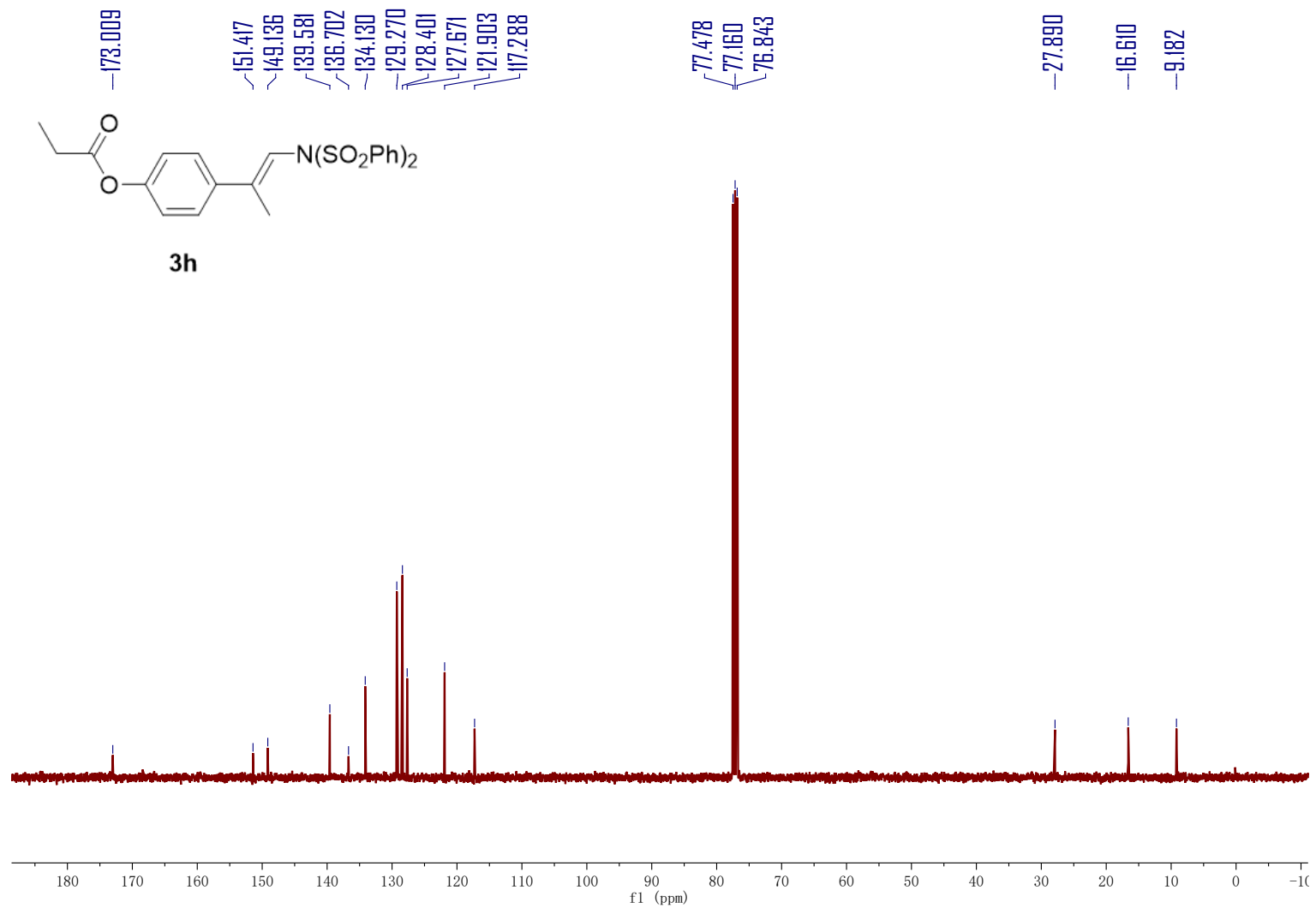




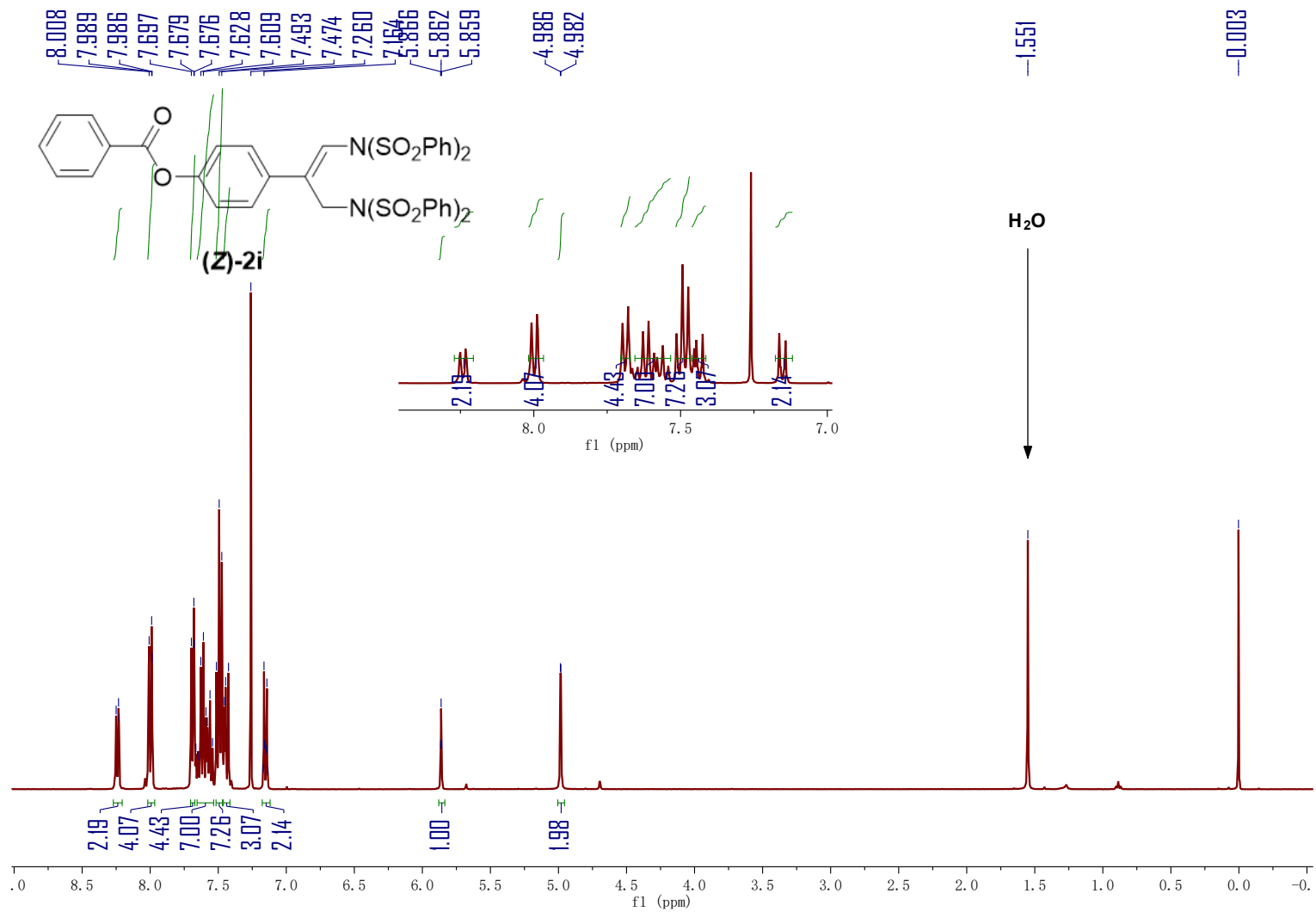
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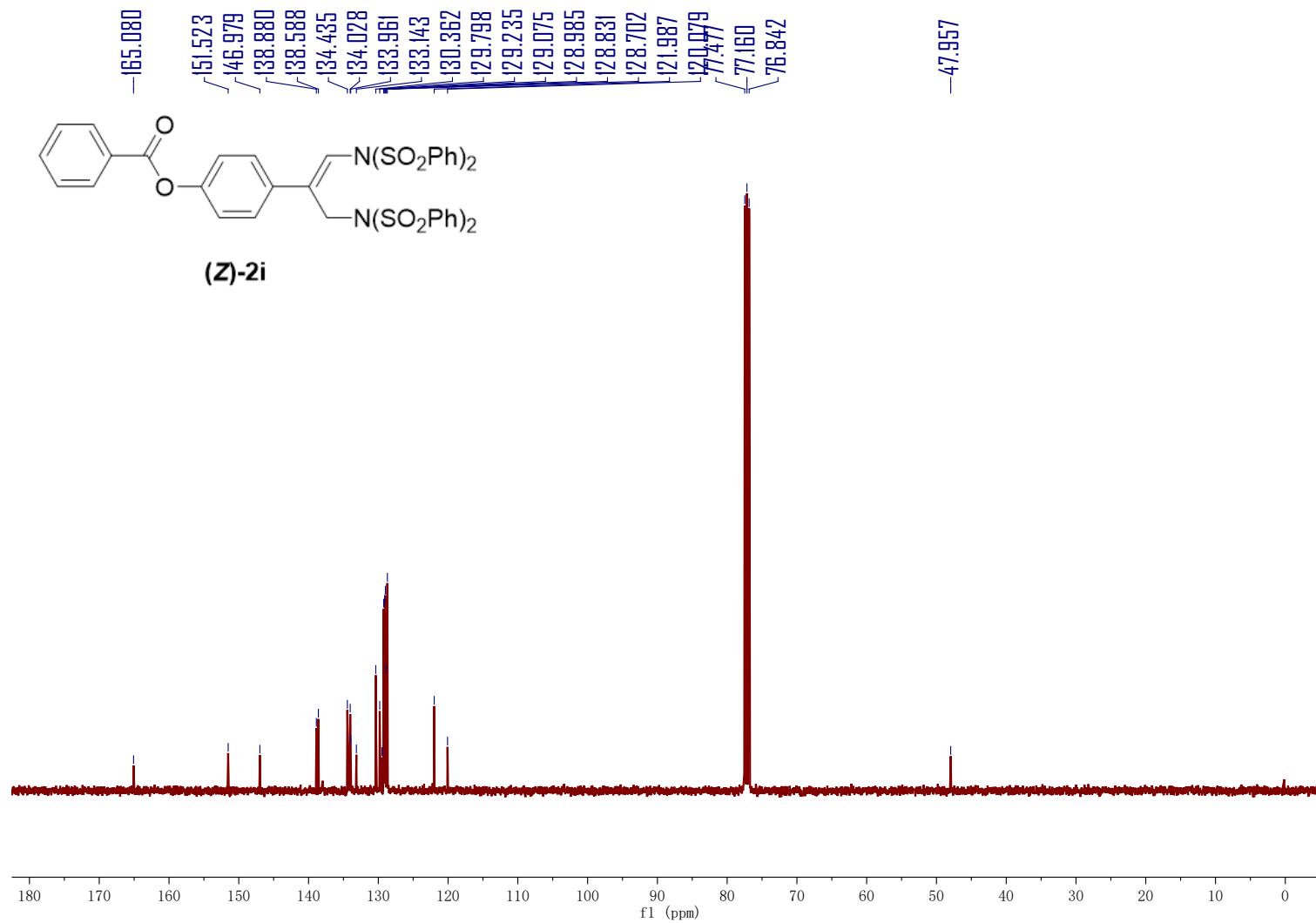




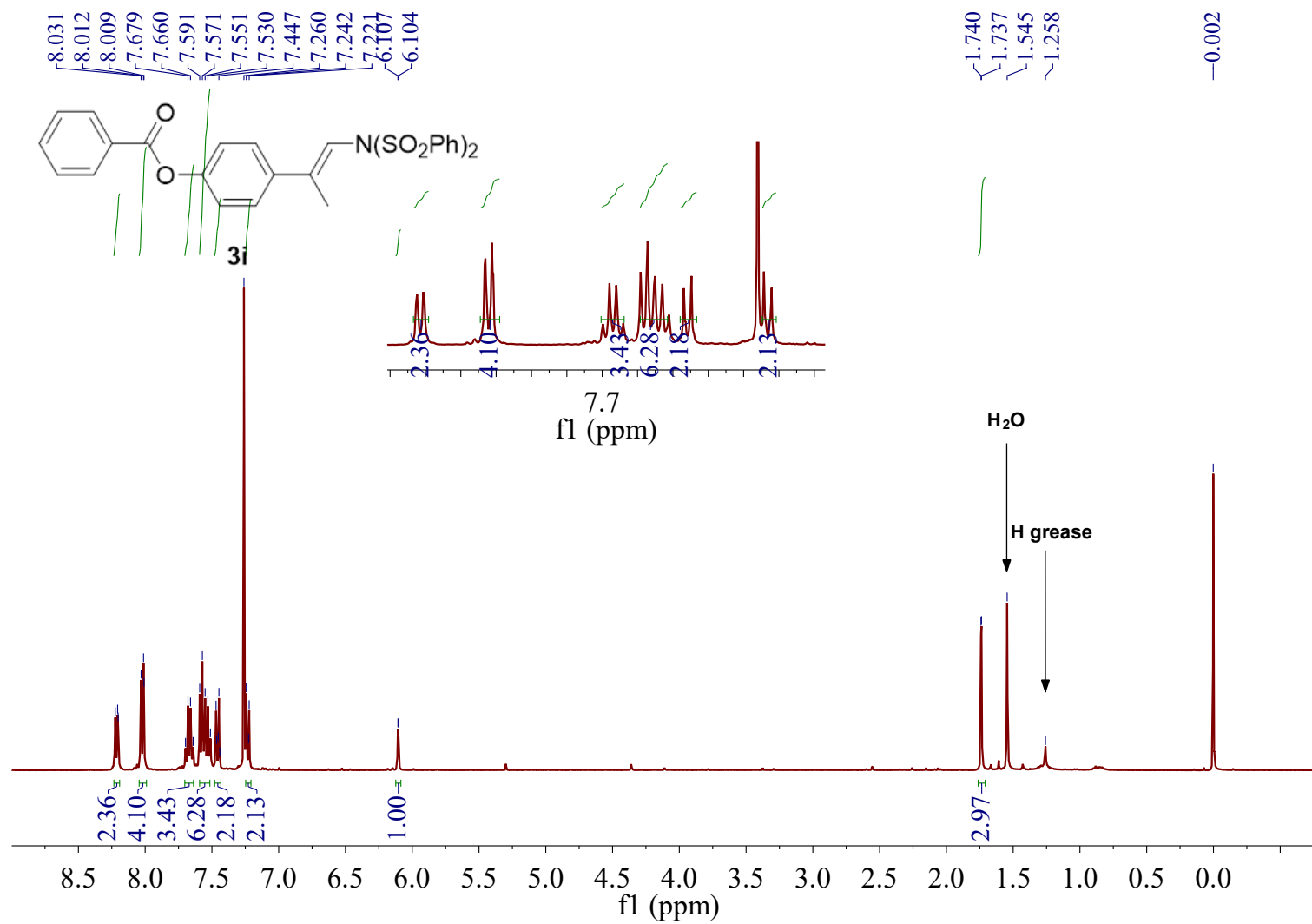
S97



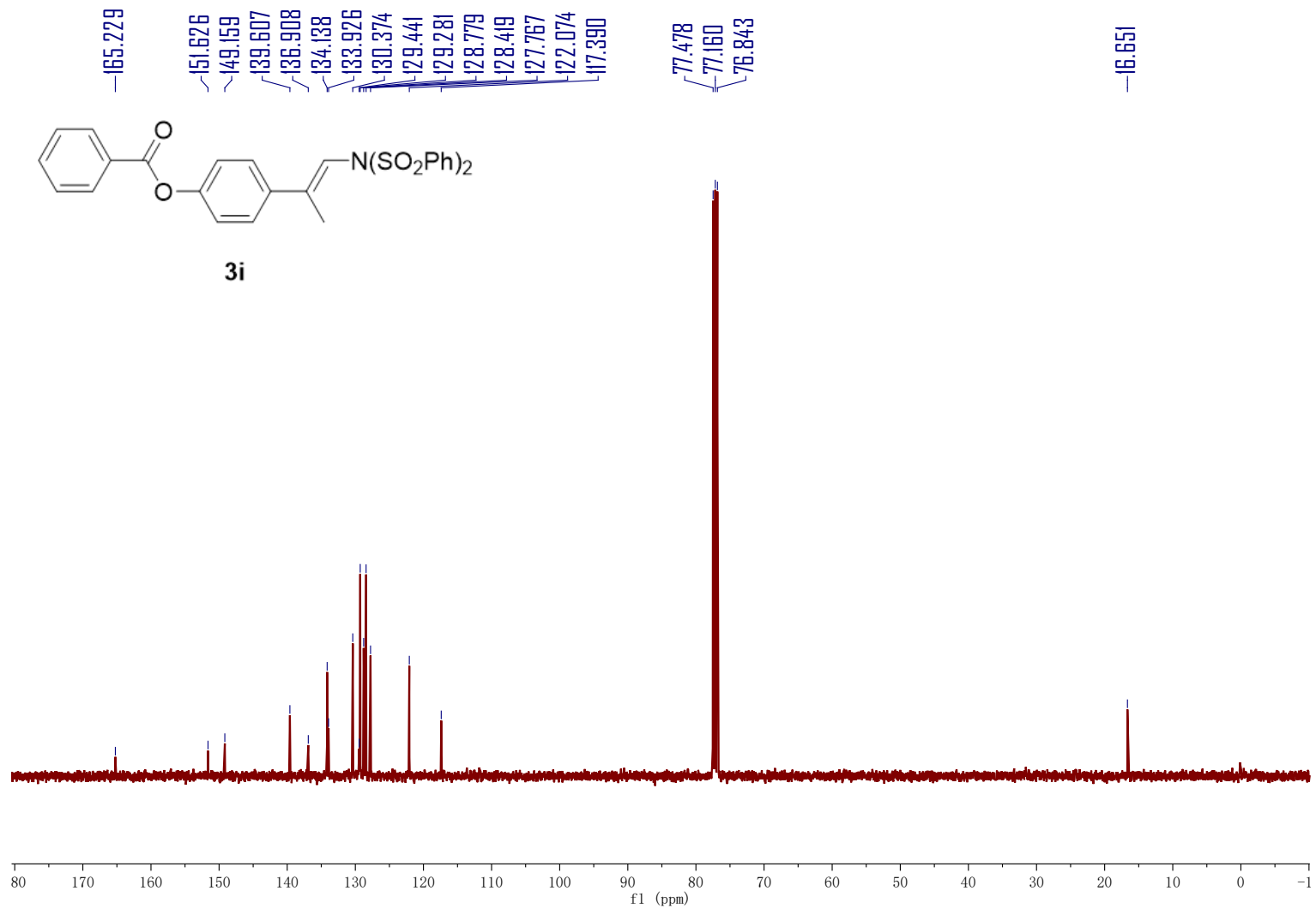
S98



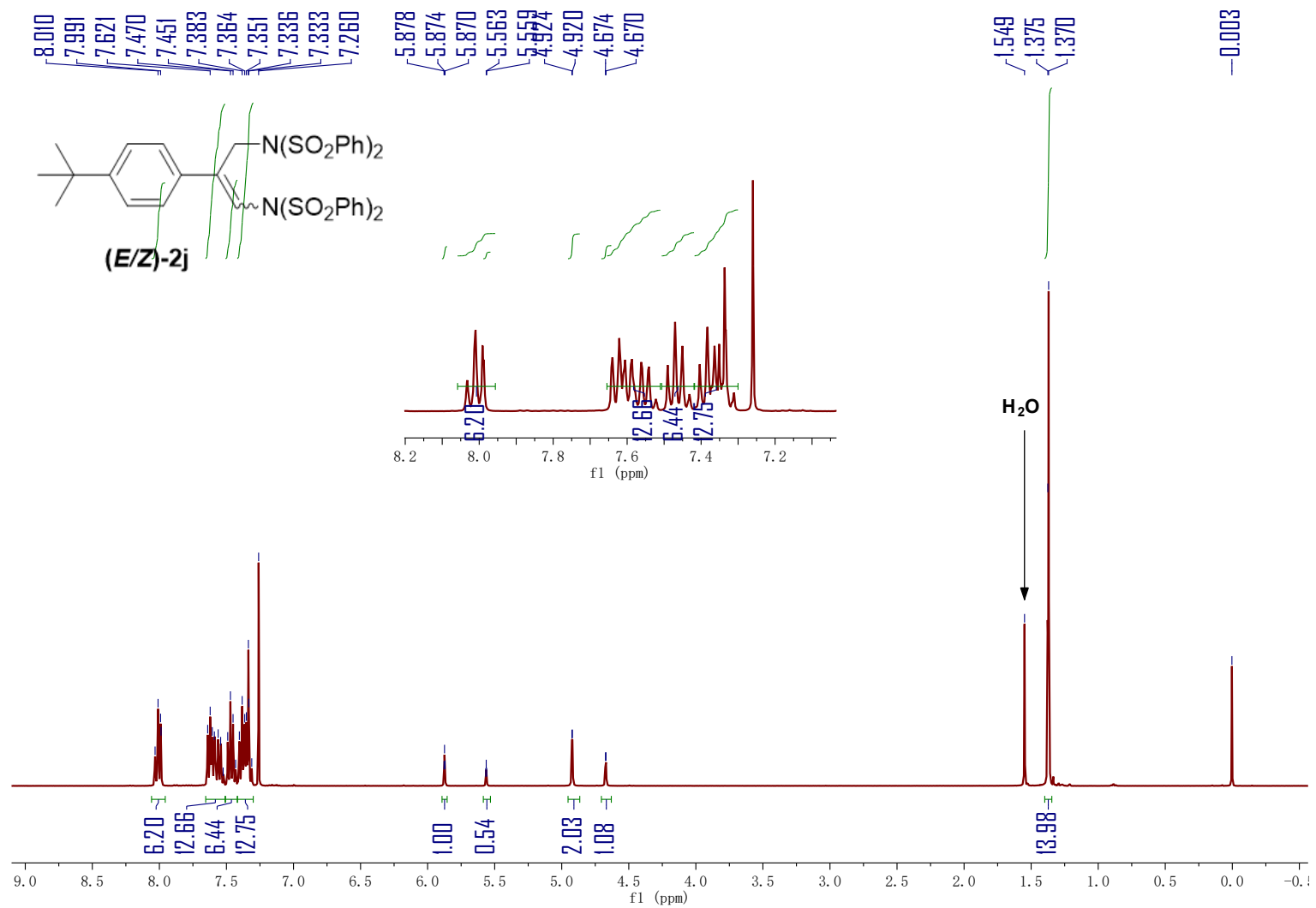
S99



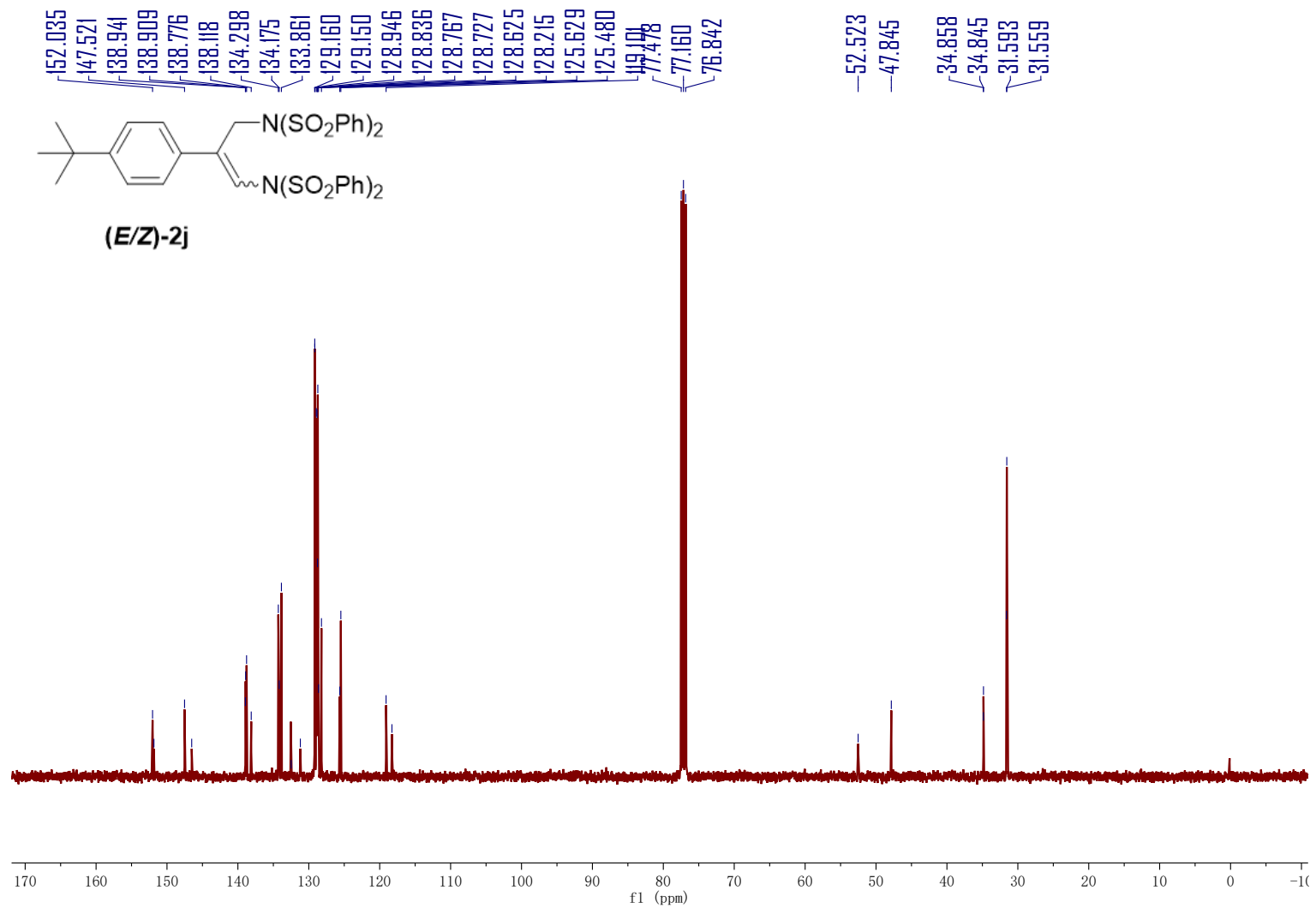
S100



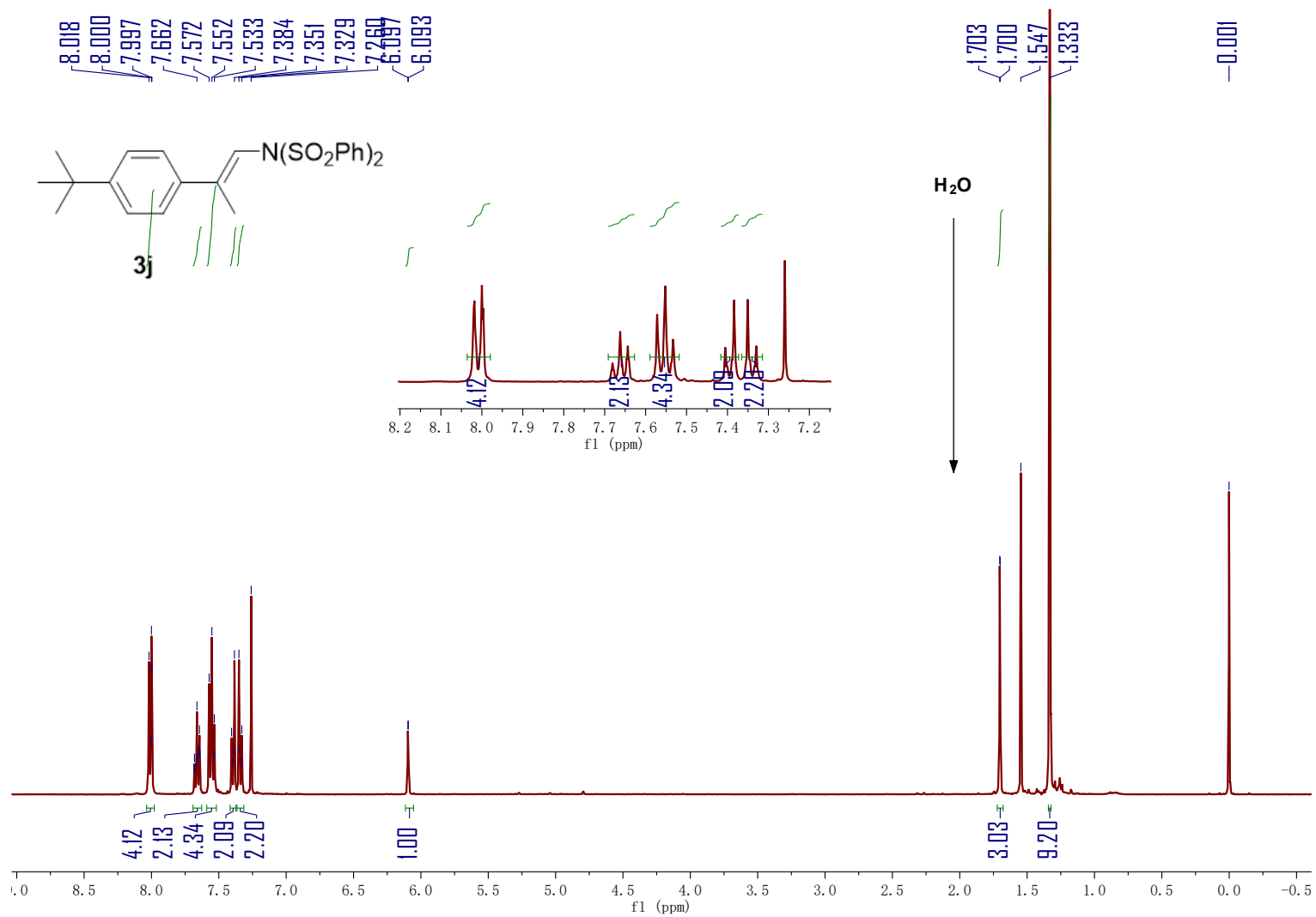
S101



S102

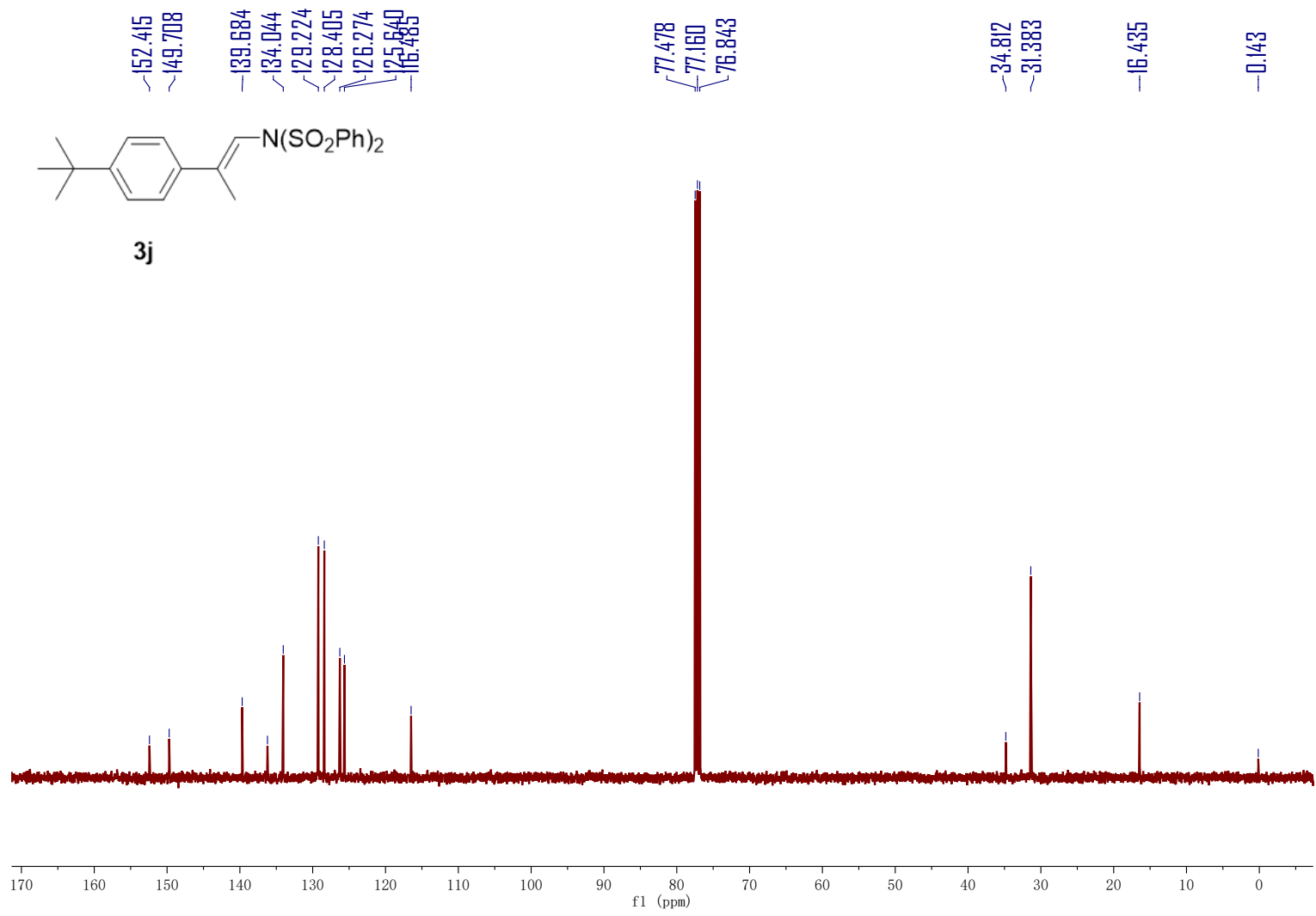


S103

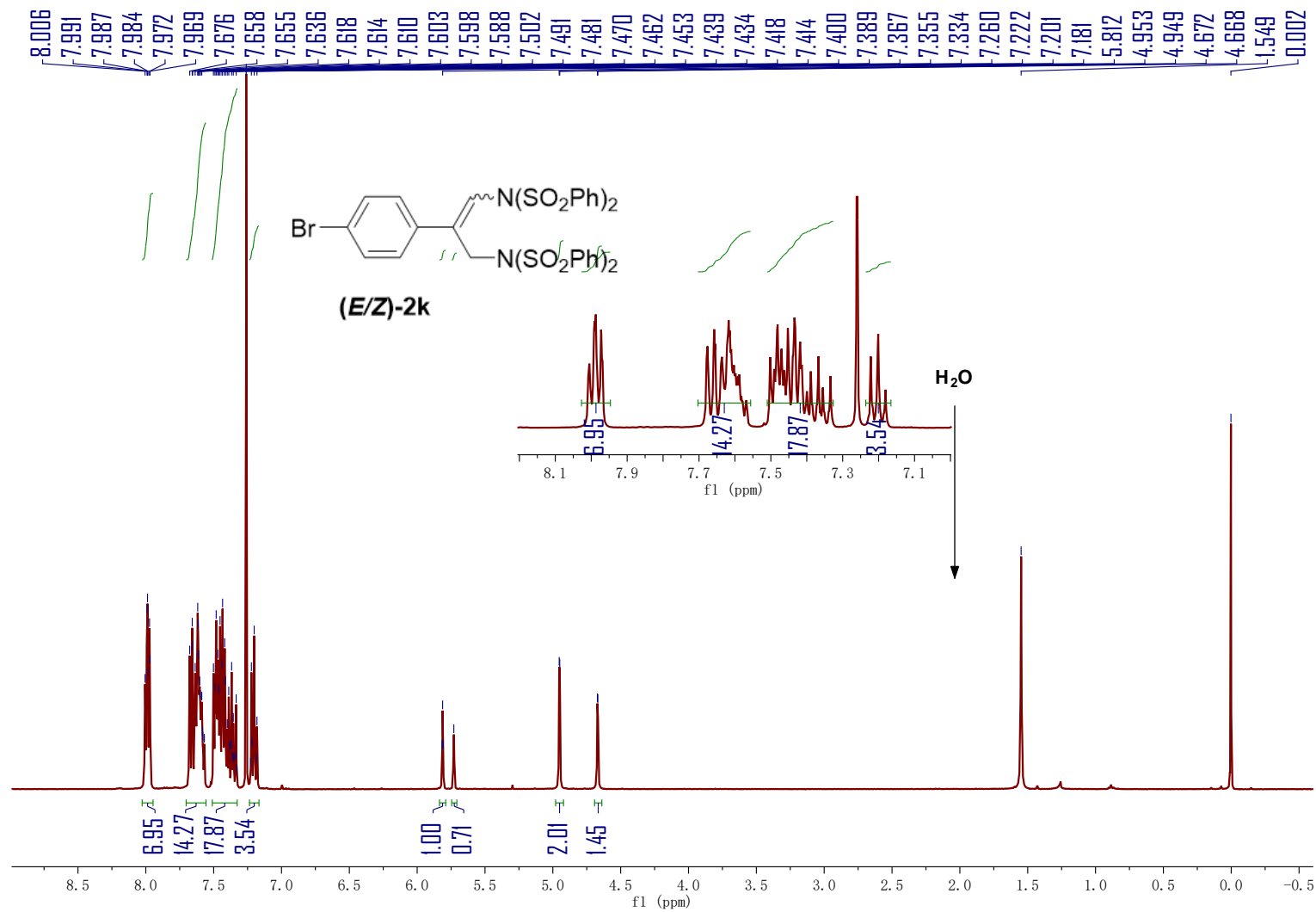


S104

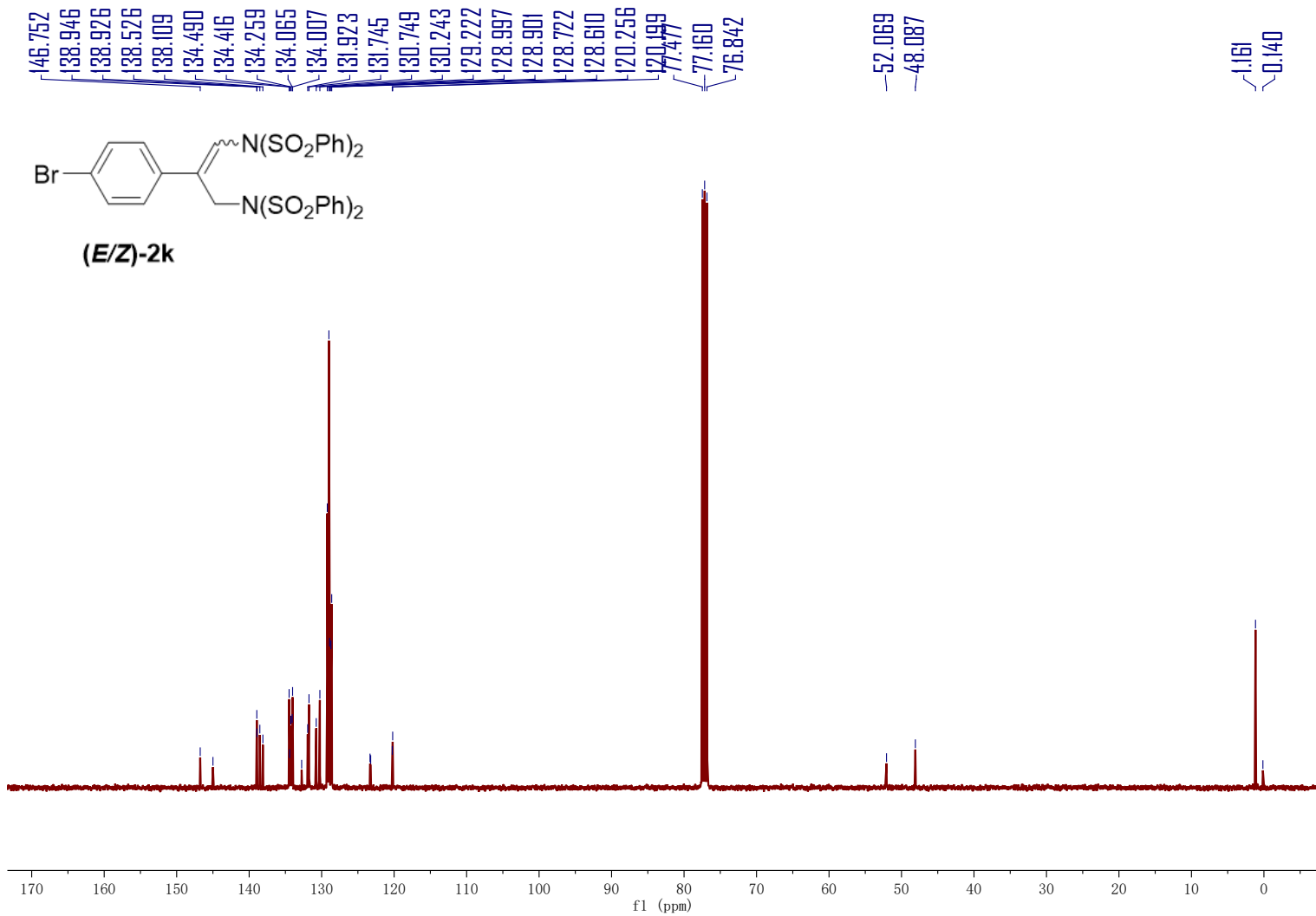




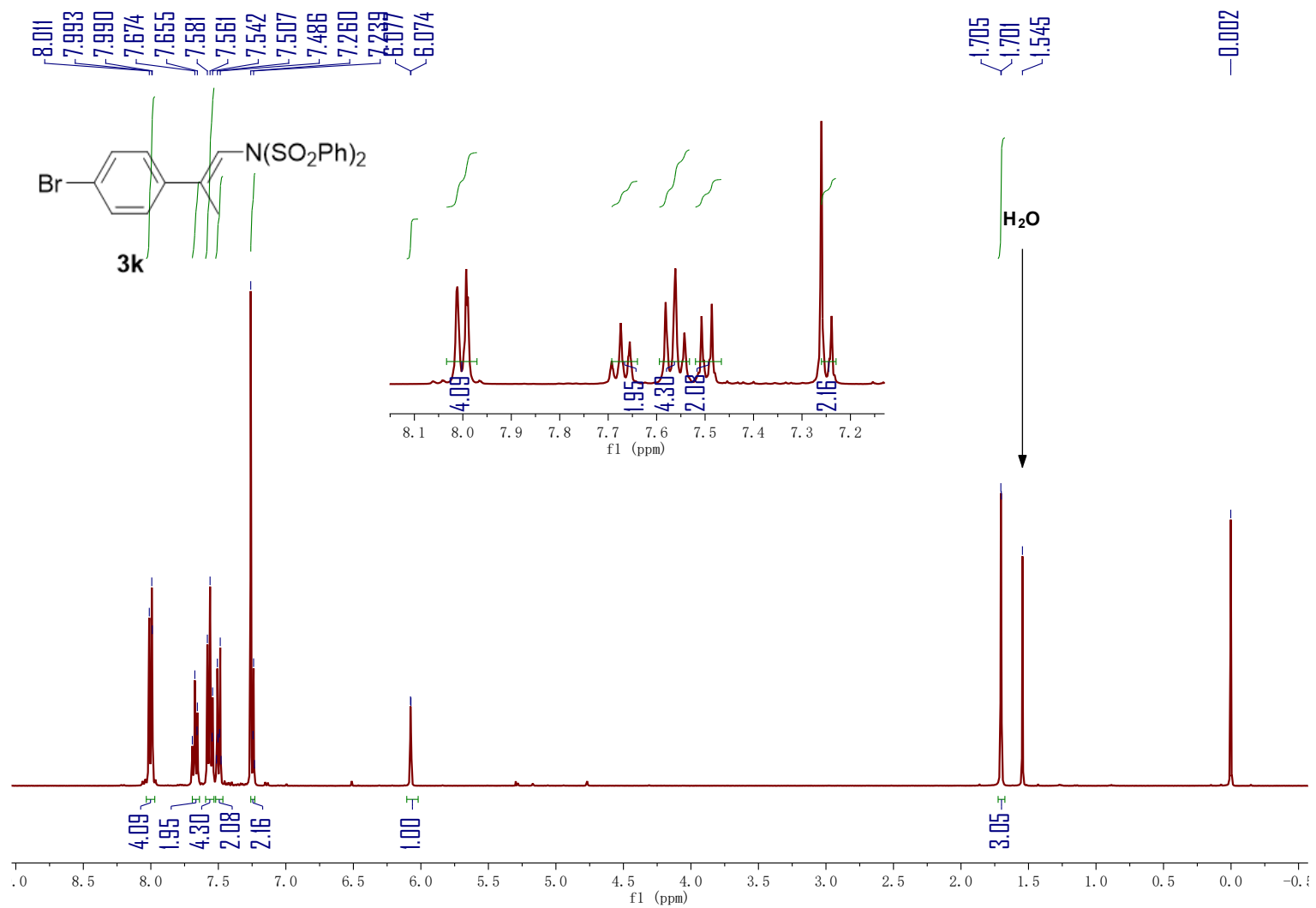
S105



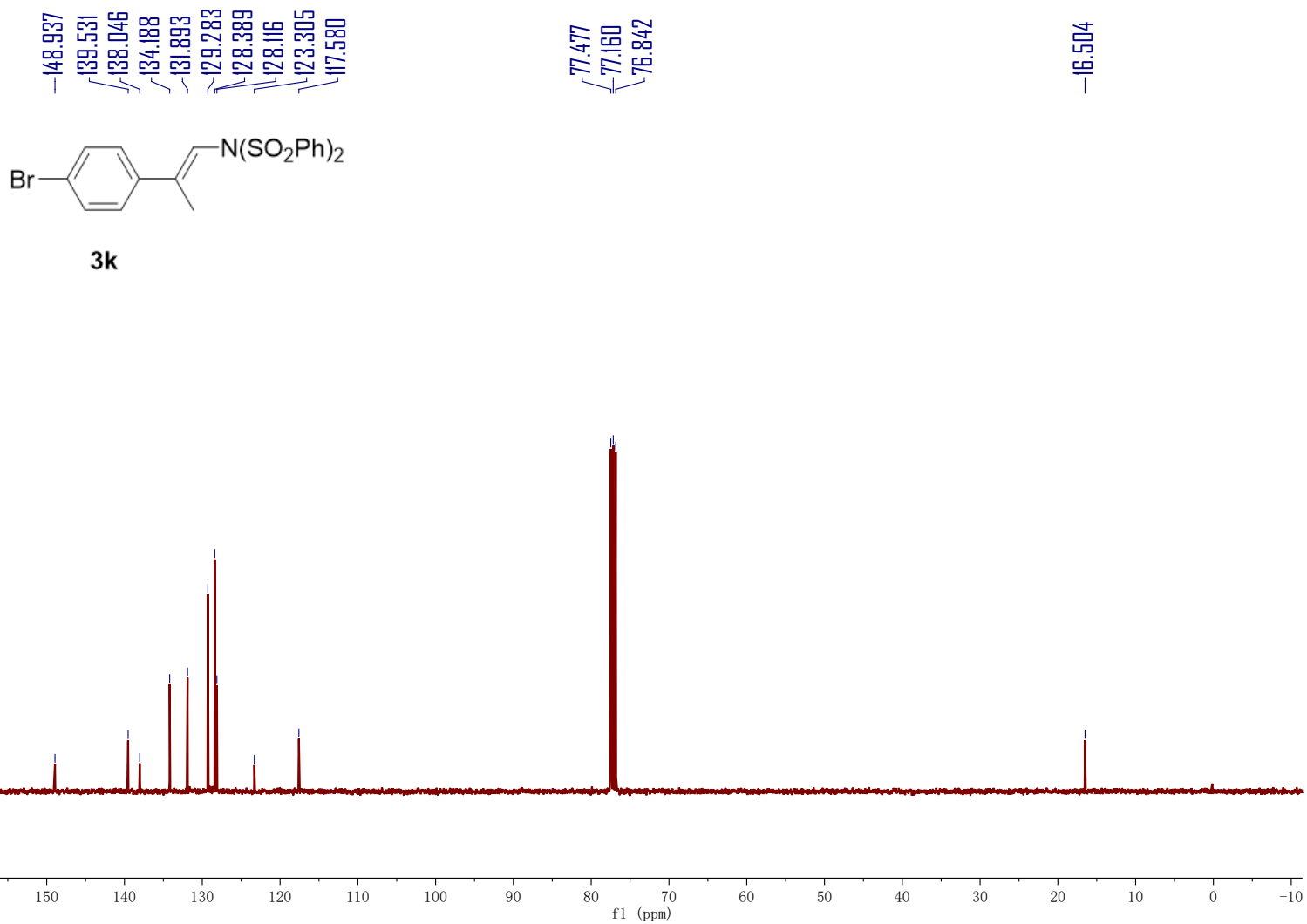
S106



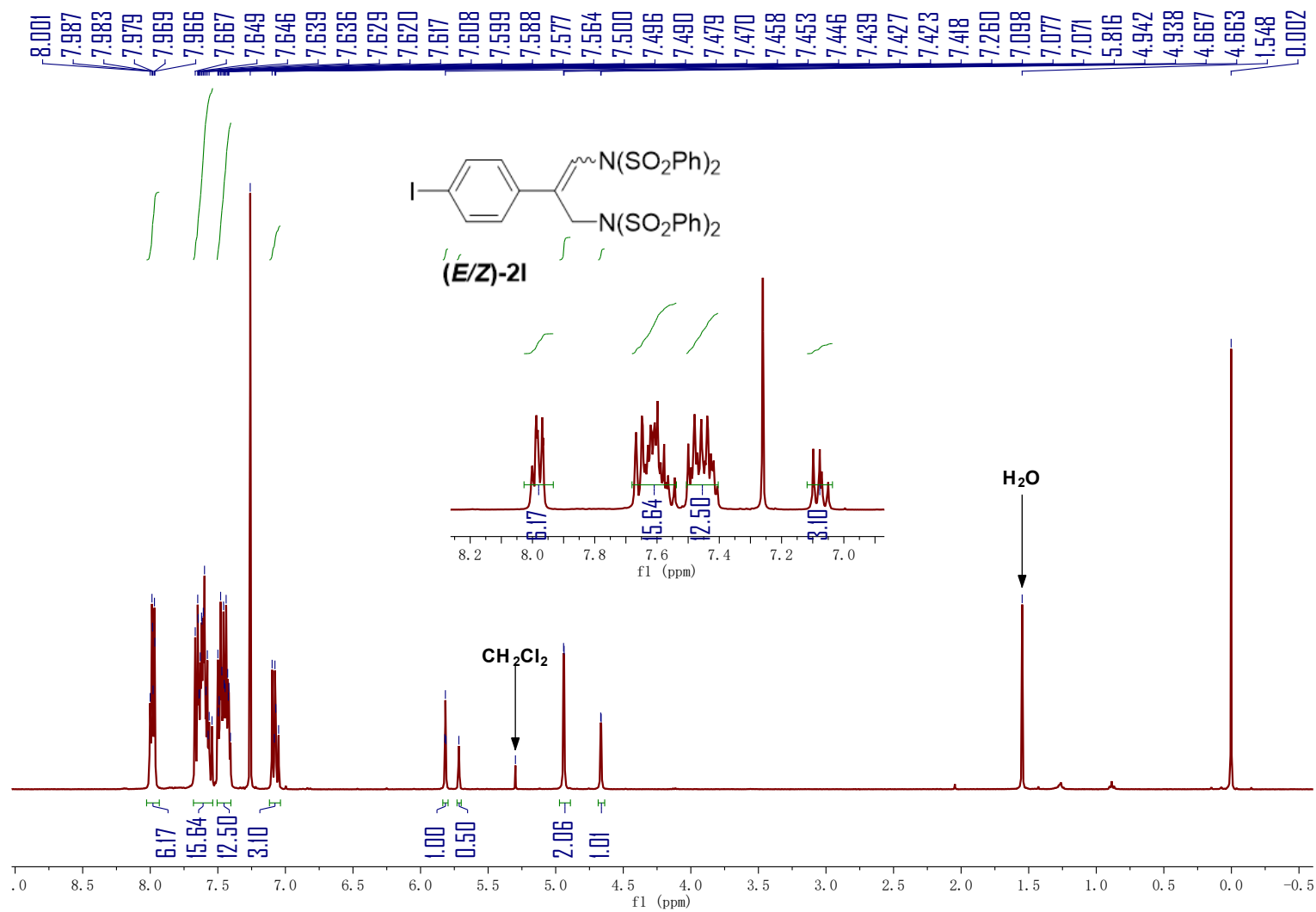
S107



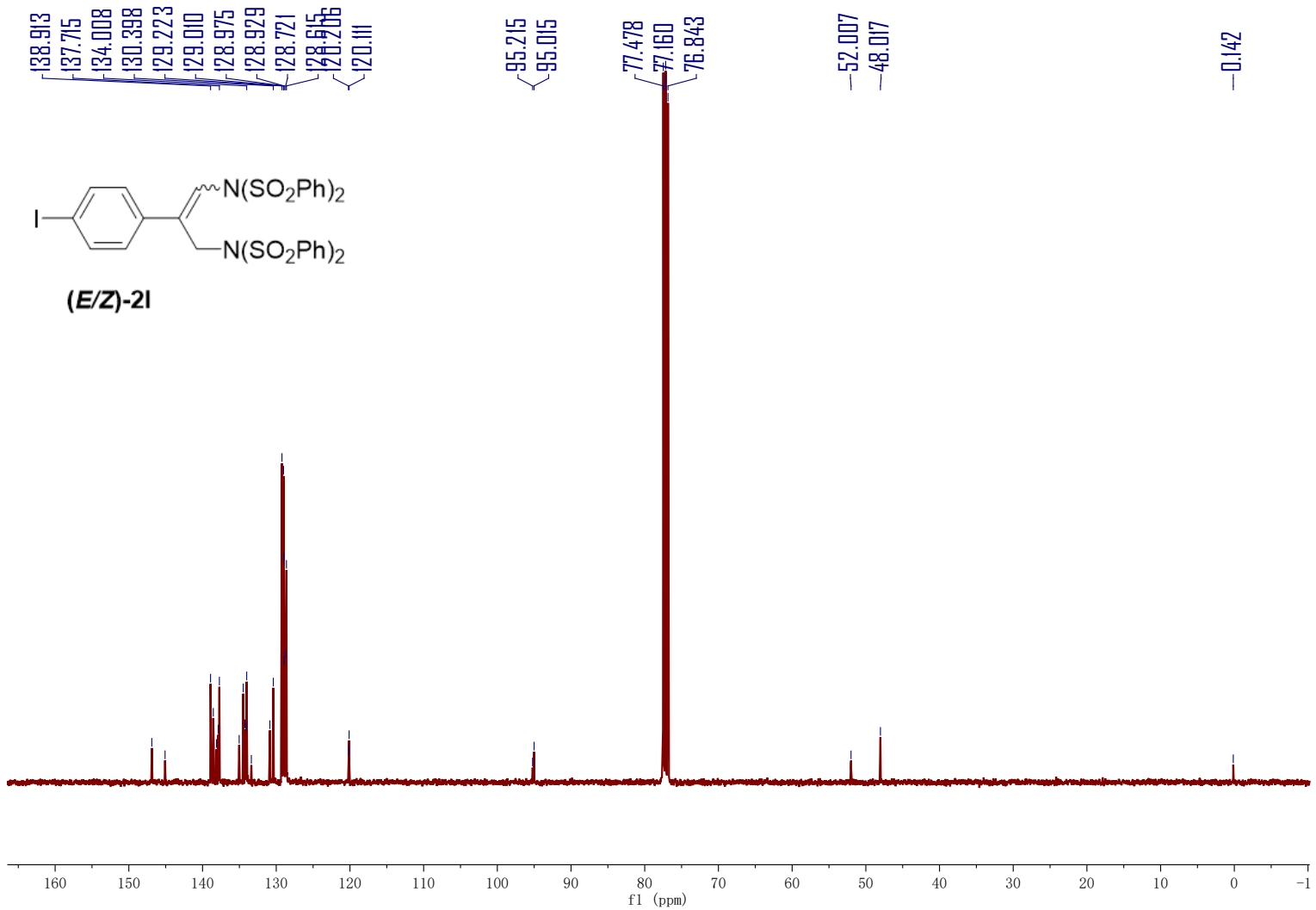
S108



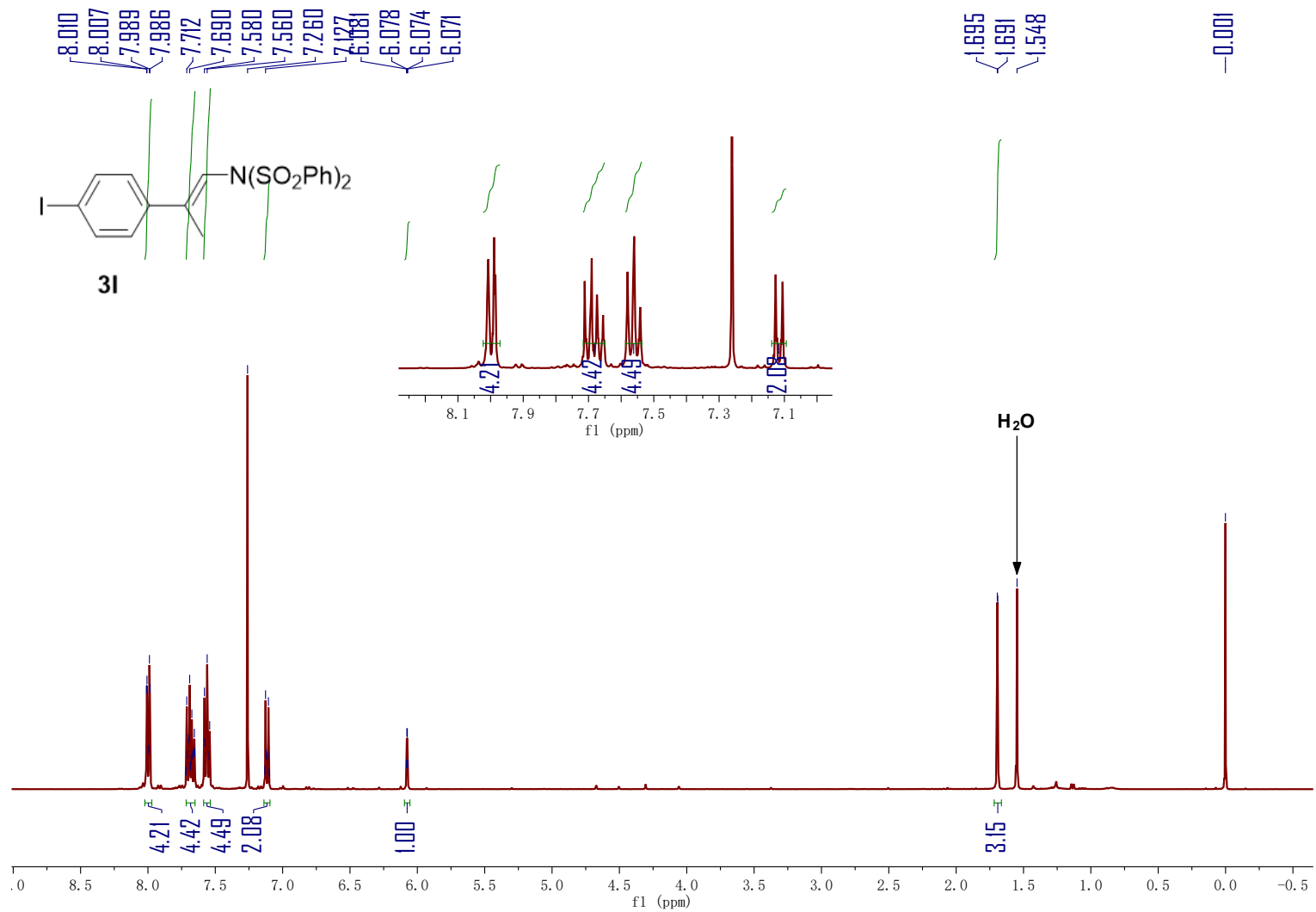
S109



S110

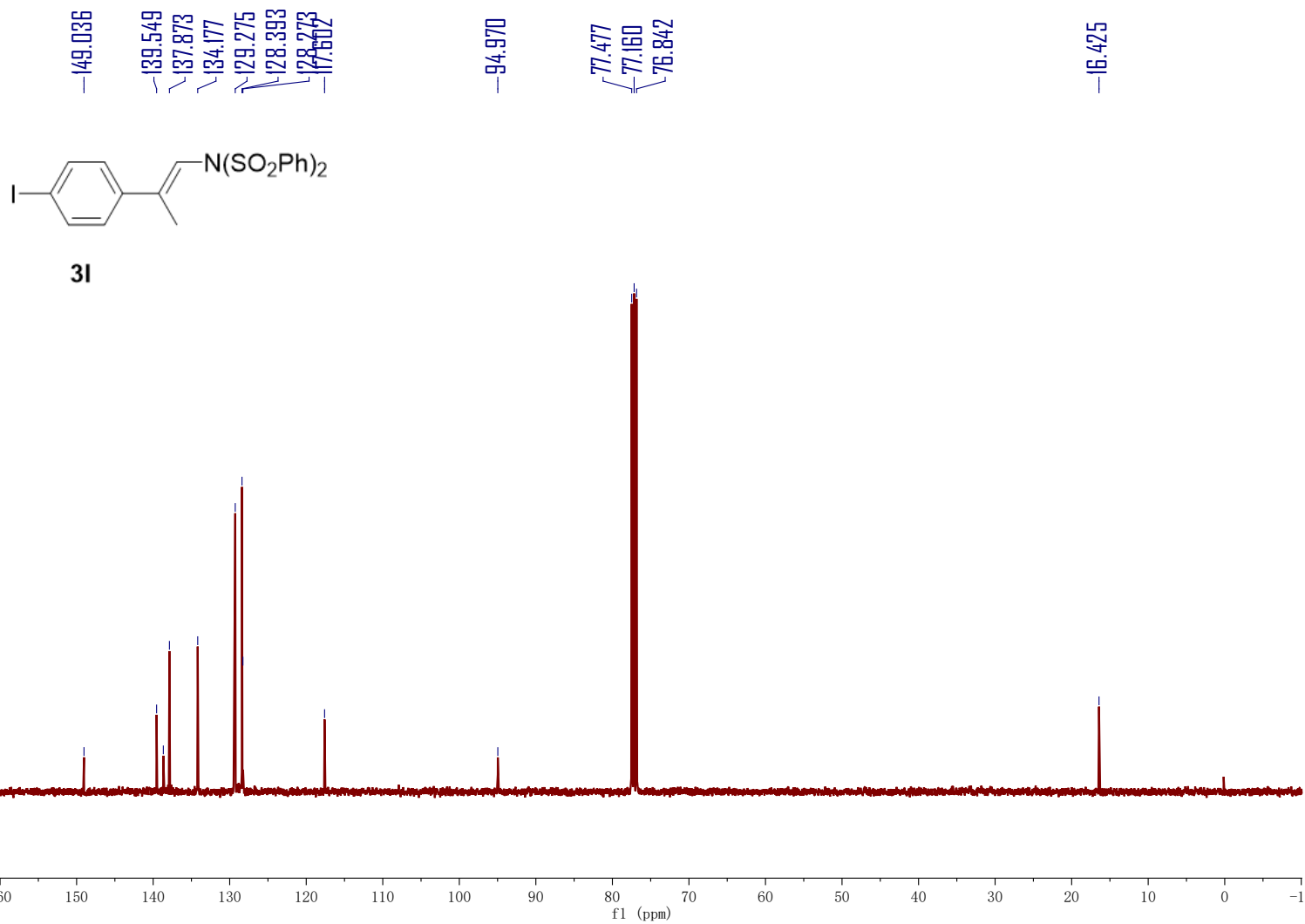


S111

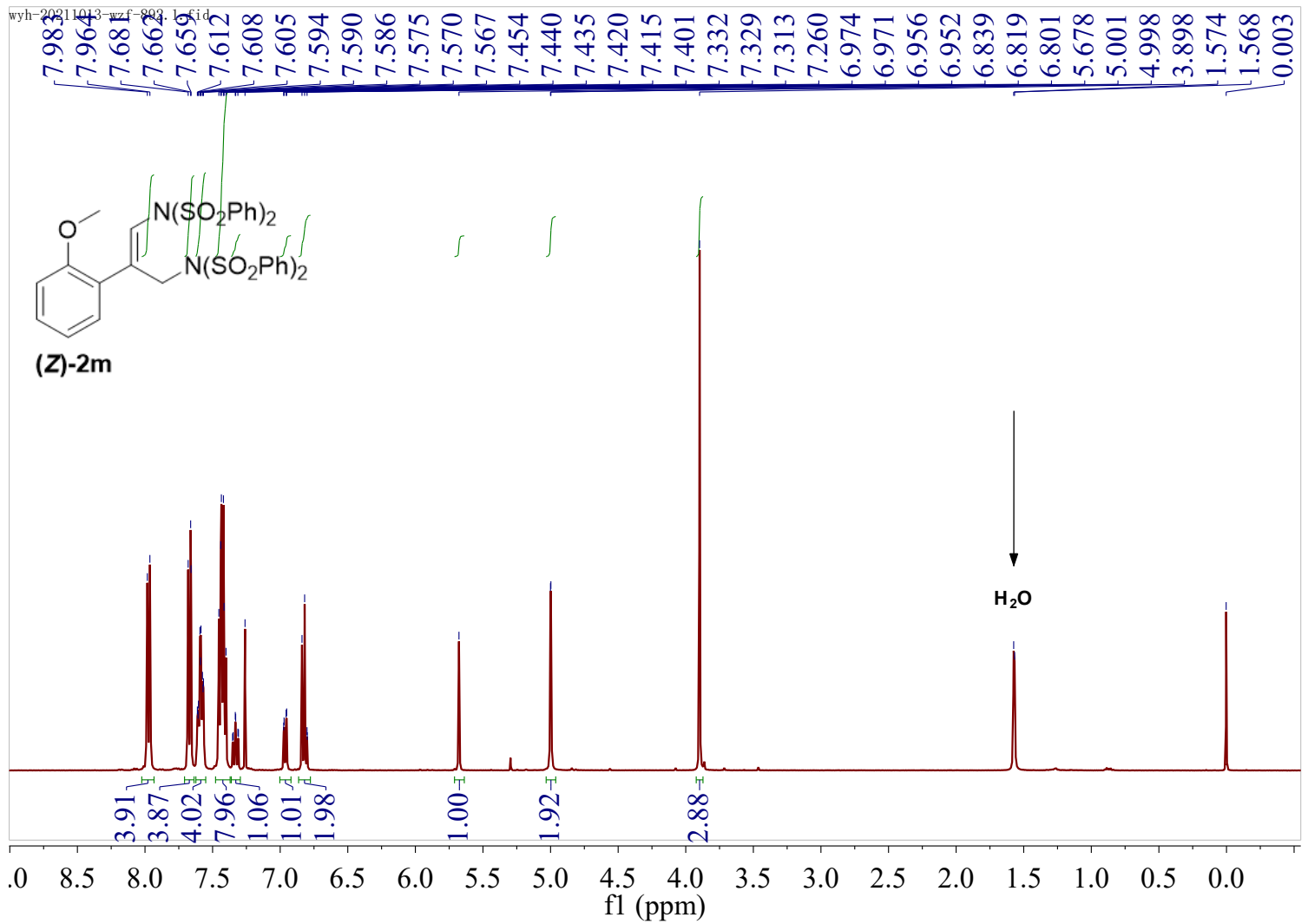


S112



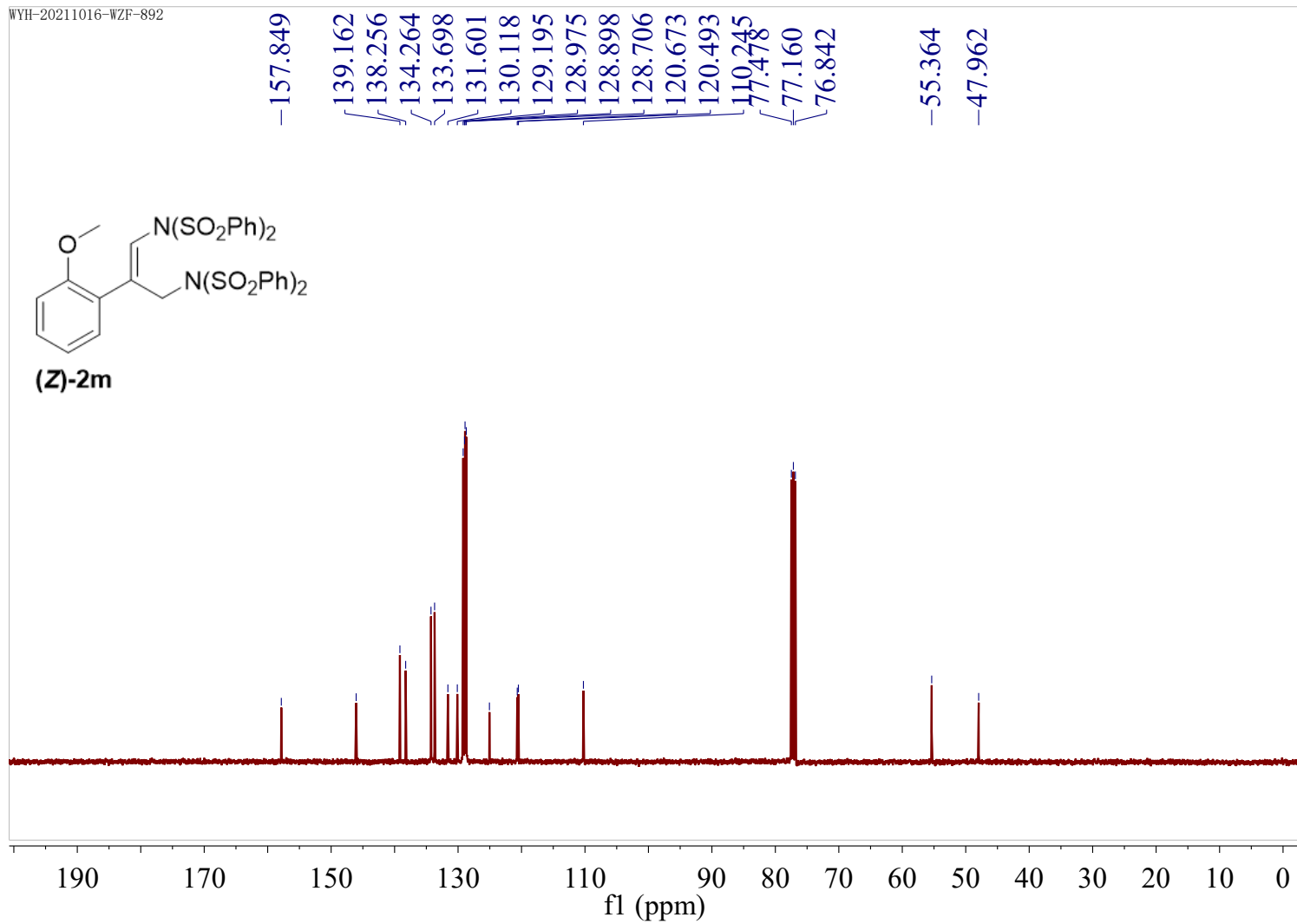
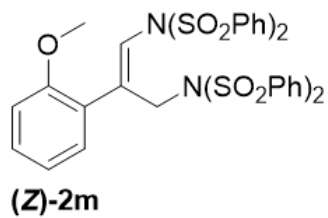


S113

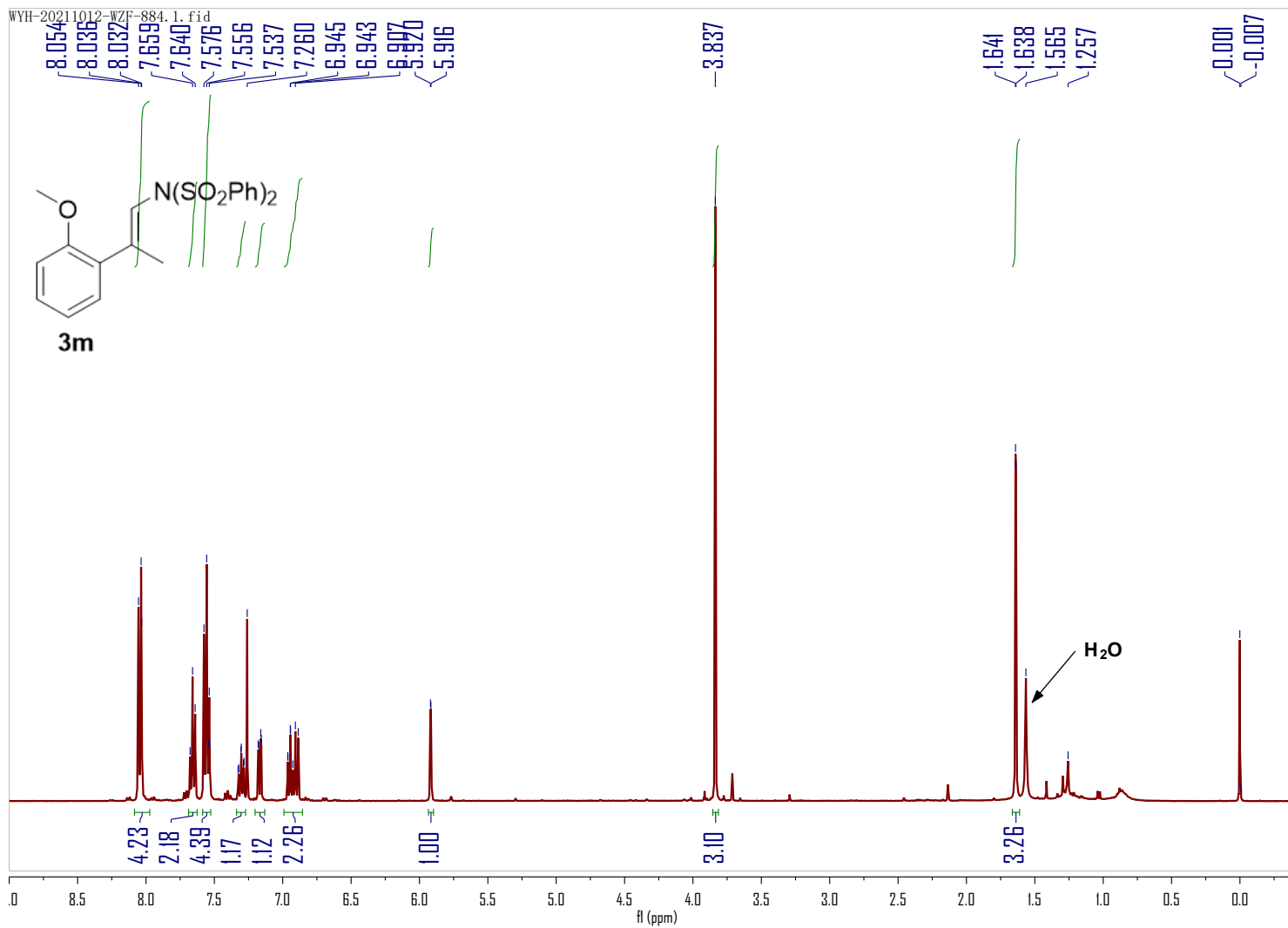


S114

WYH-20211016-WZF-892

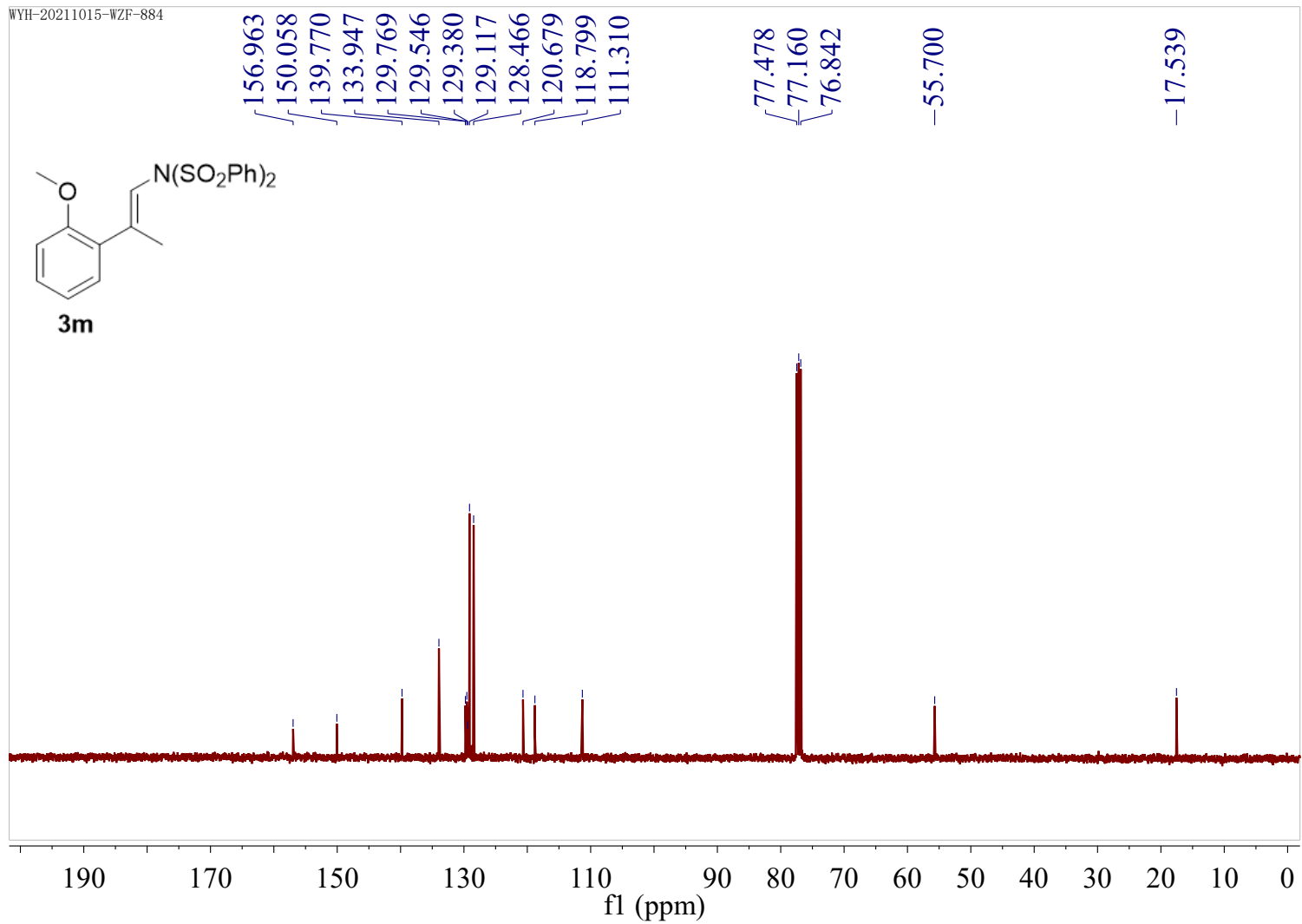


S115

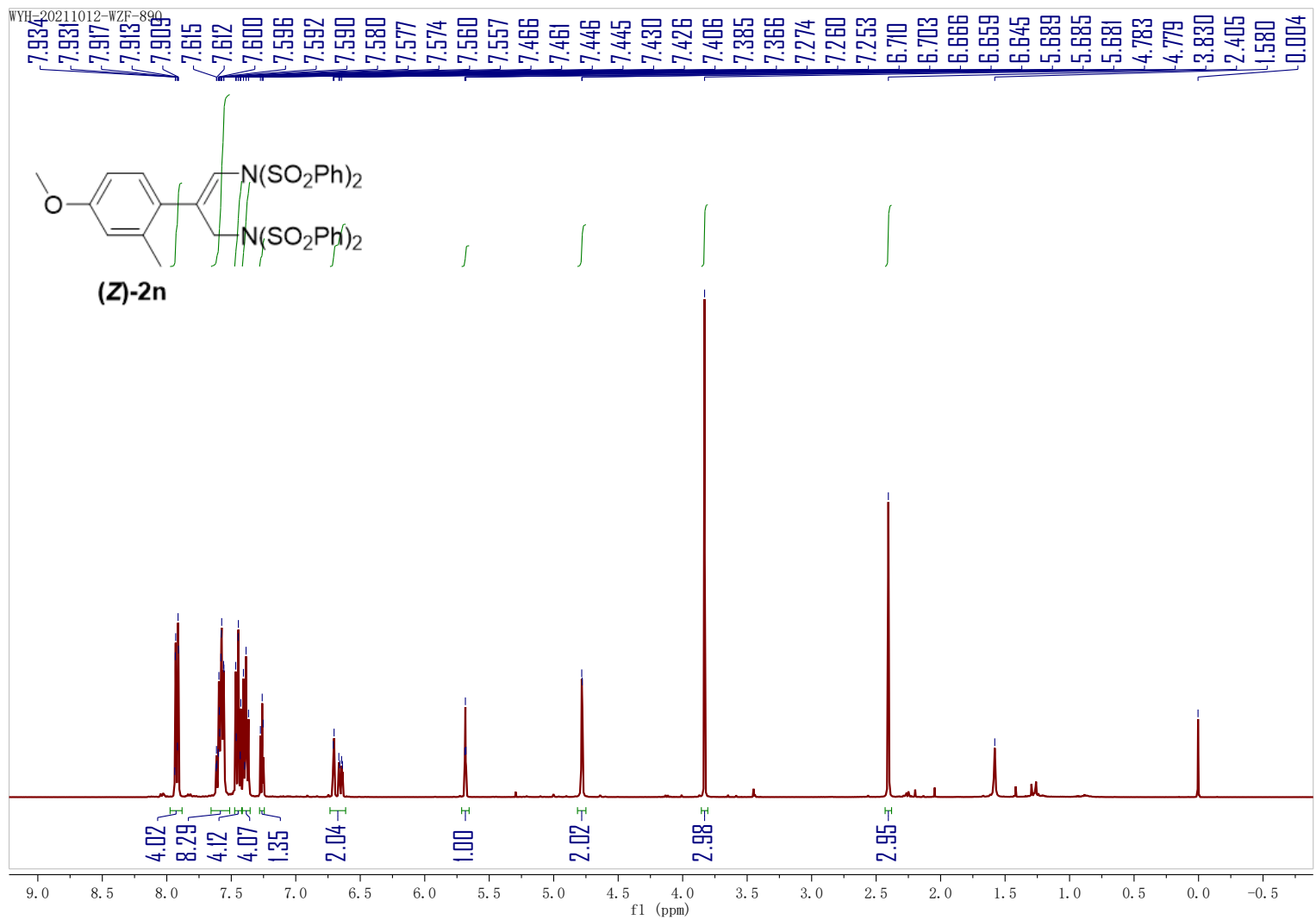


S116

WYH-20211015-WZF-884

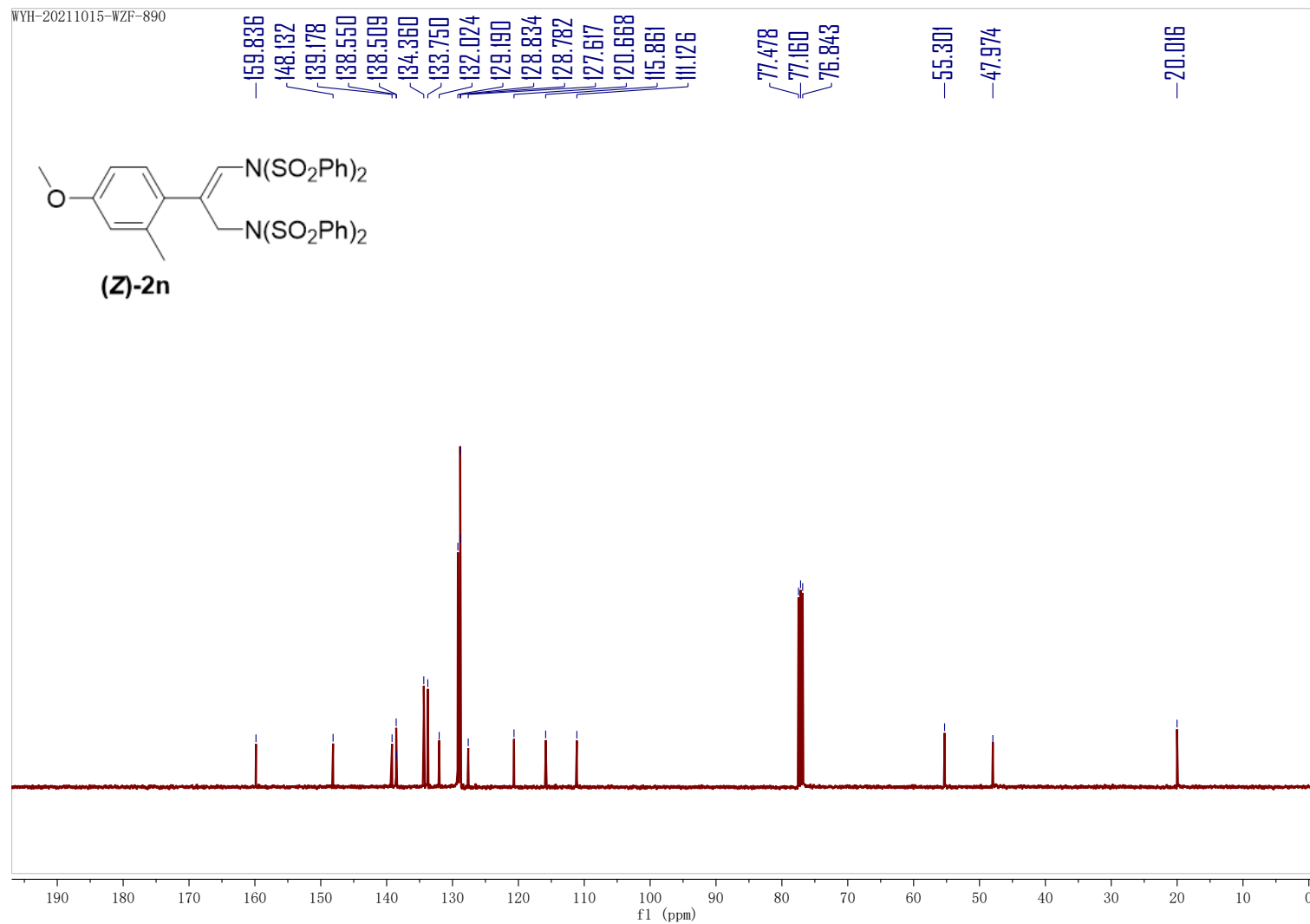
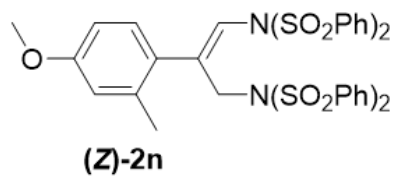


S117

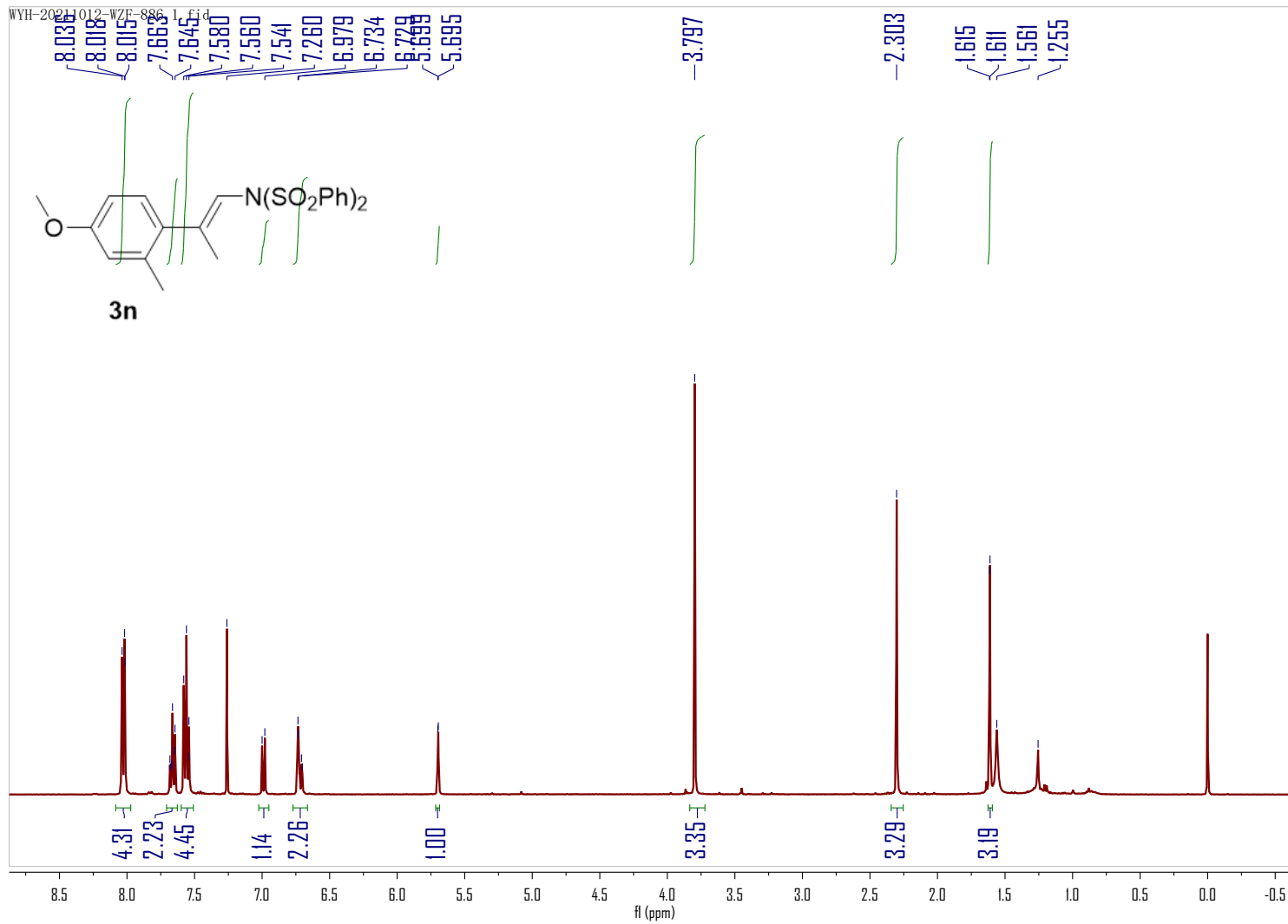


S118

WYH-20211015-WZF-890



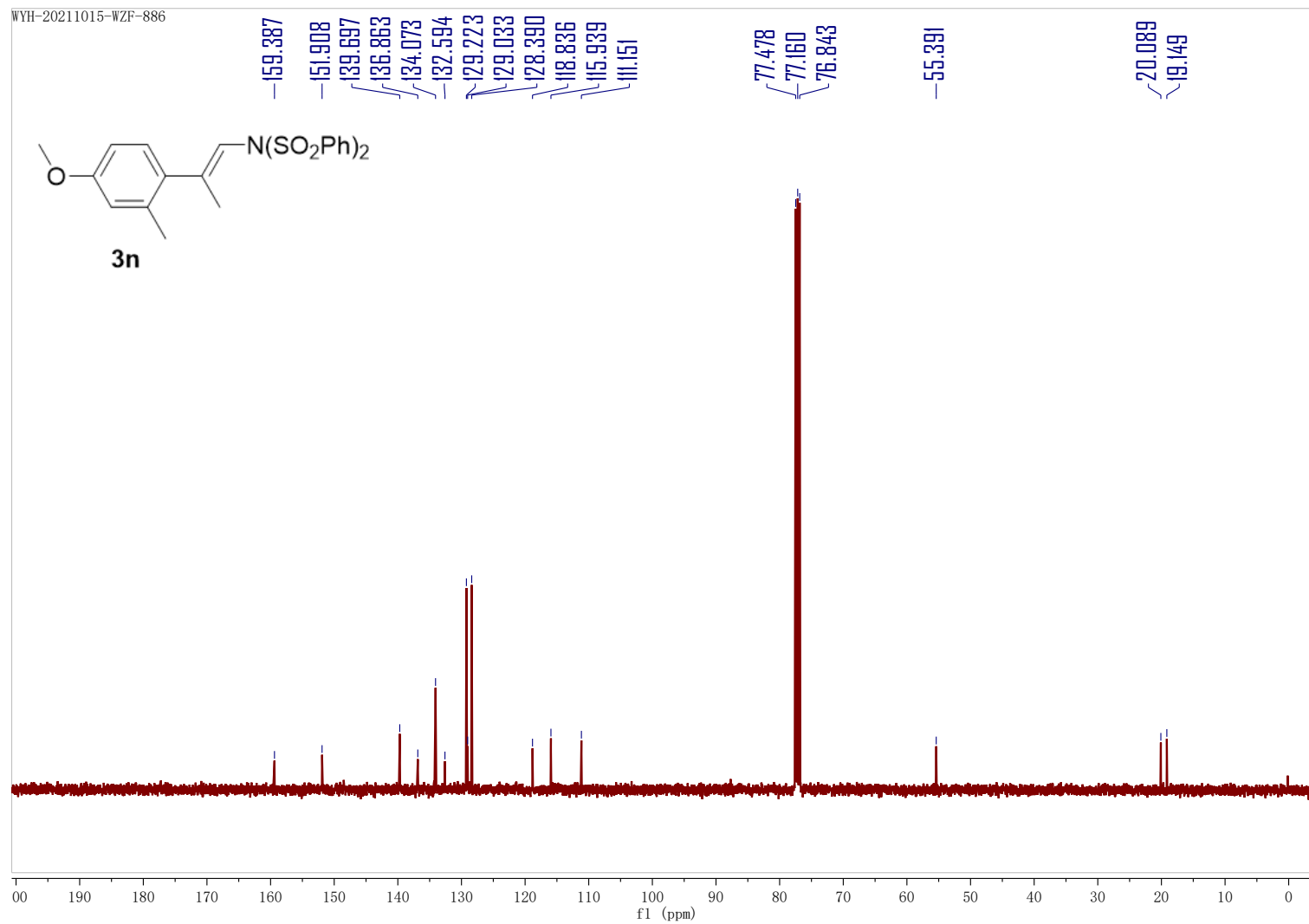
S119



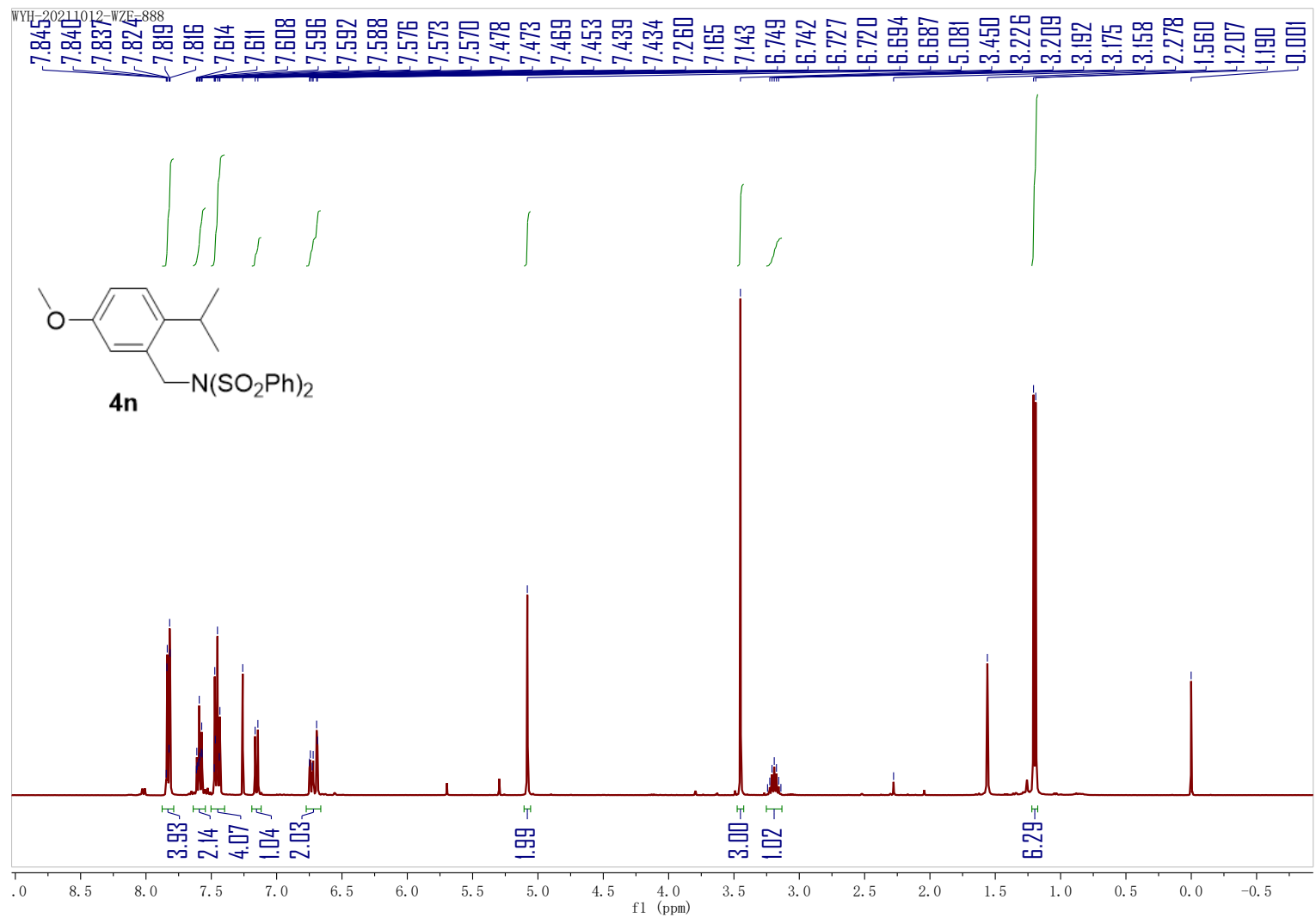
S120



WYH-20211015-WZF-886

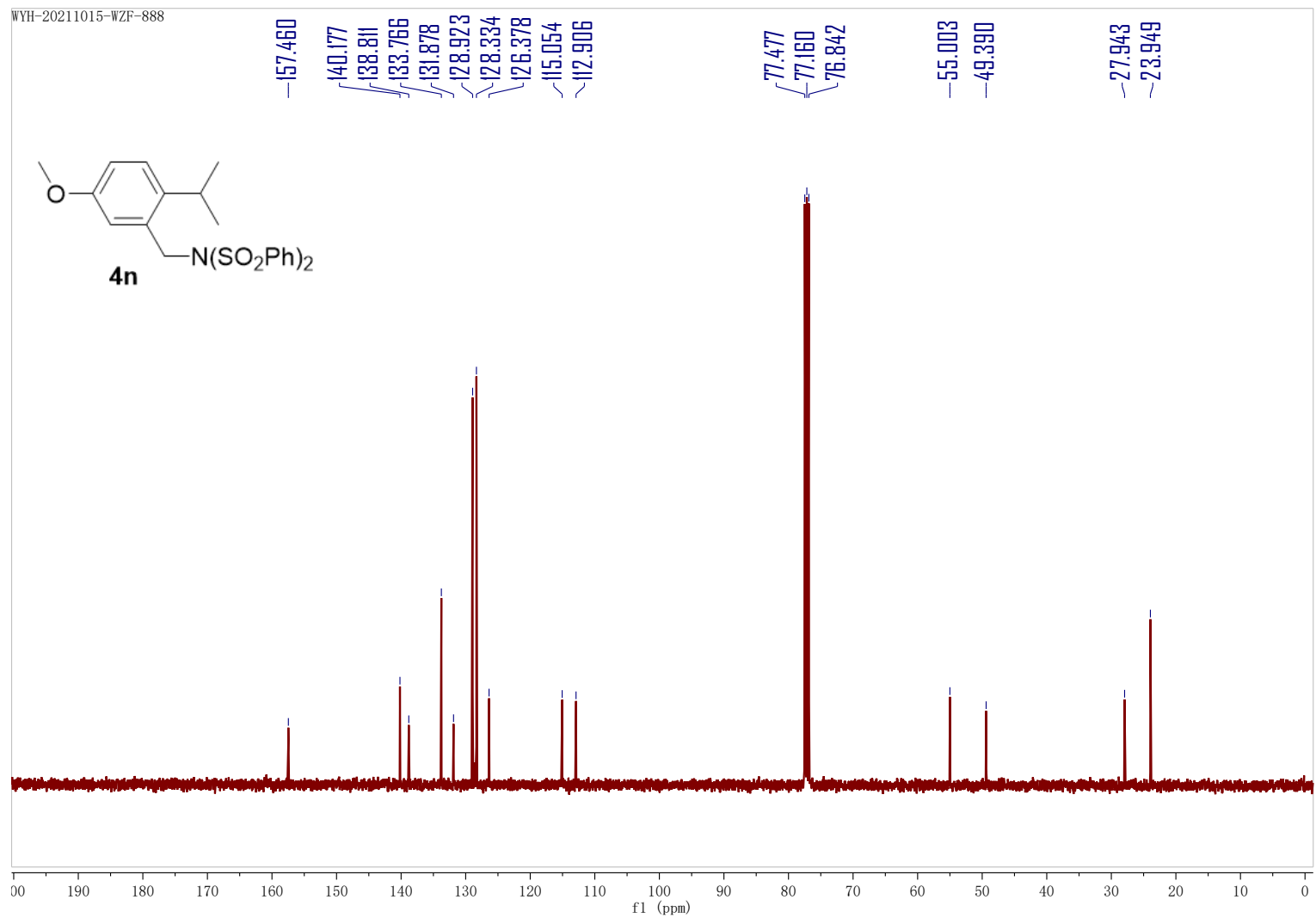


S121

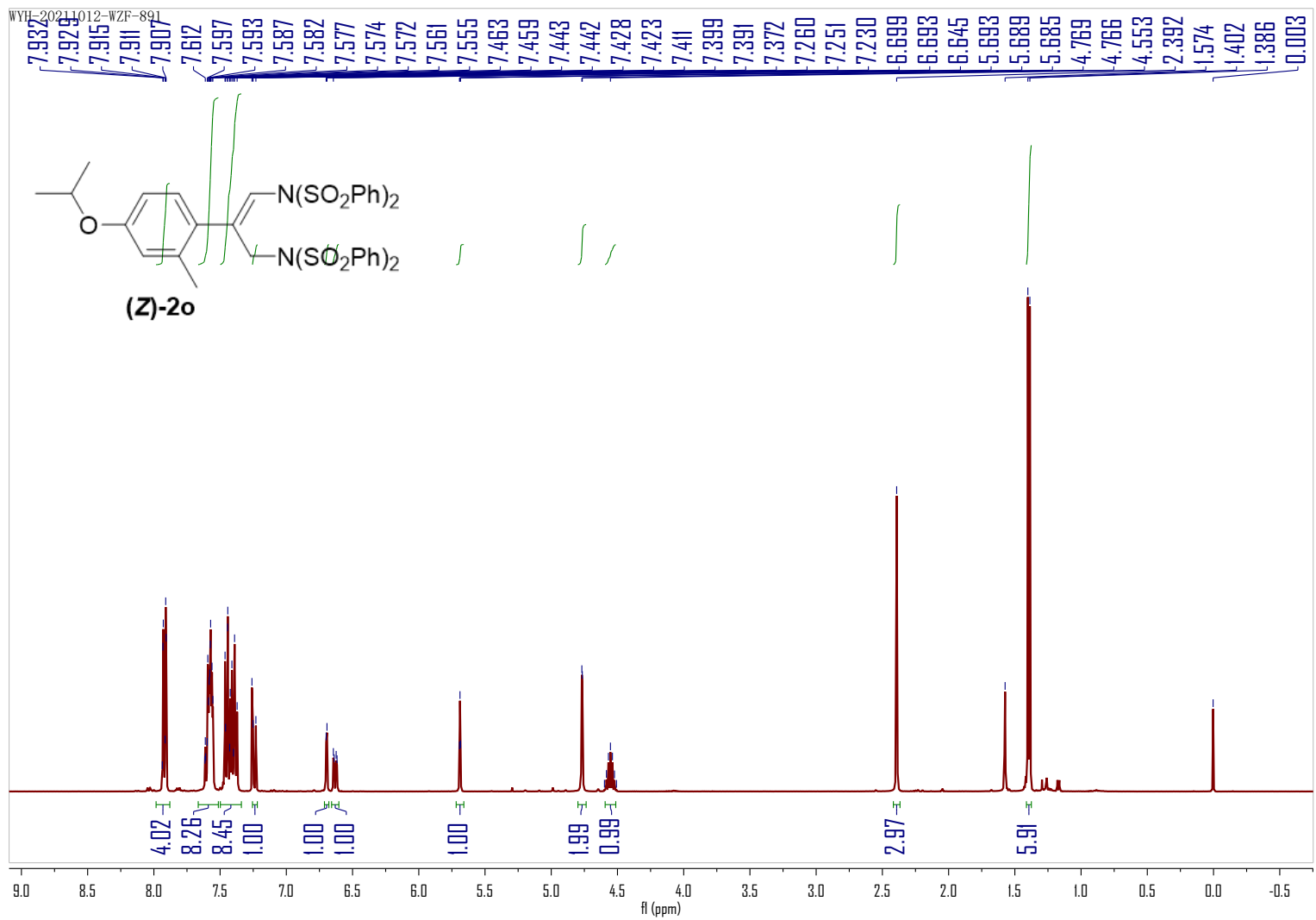


S122

WYH-20211015-WZF-888

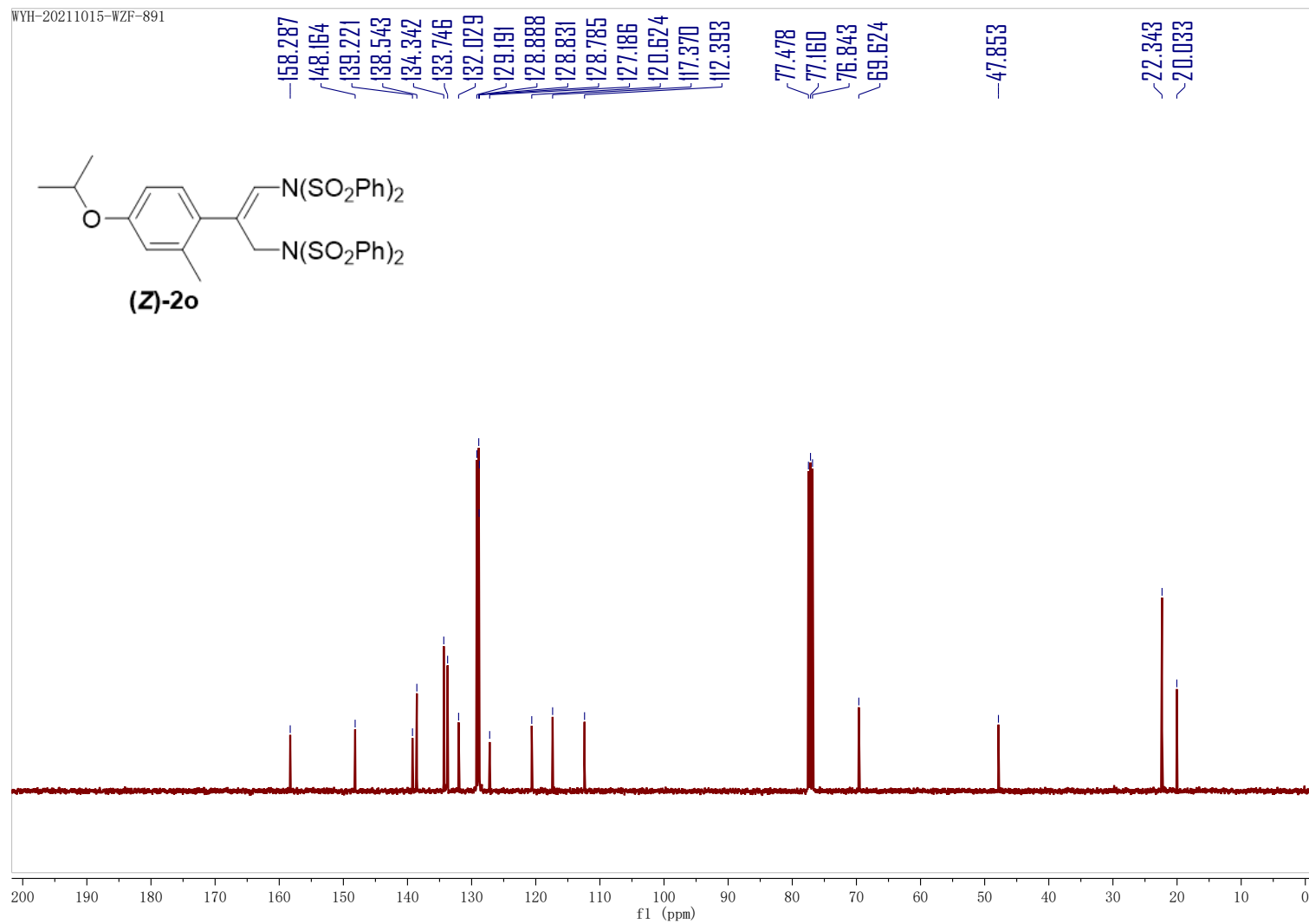


S123

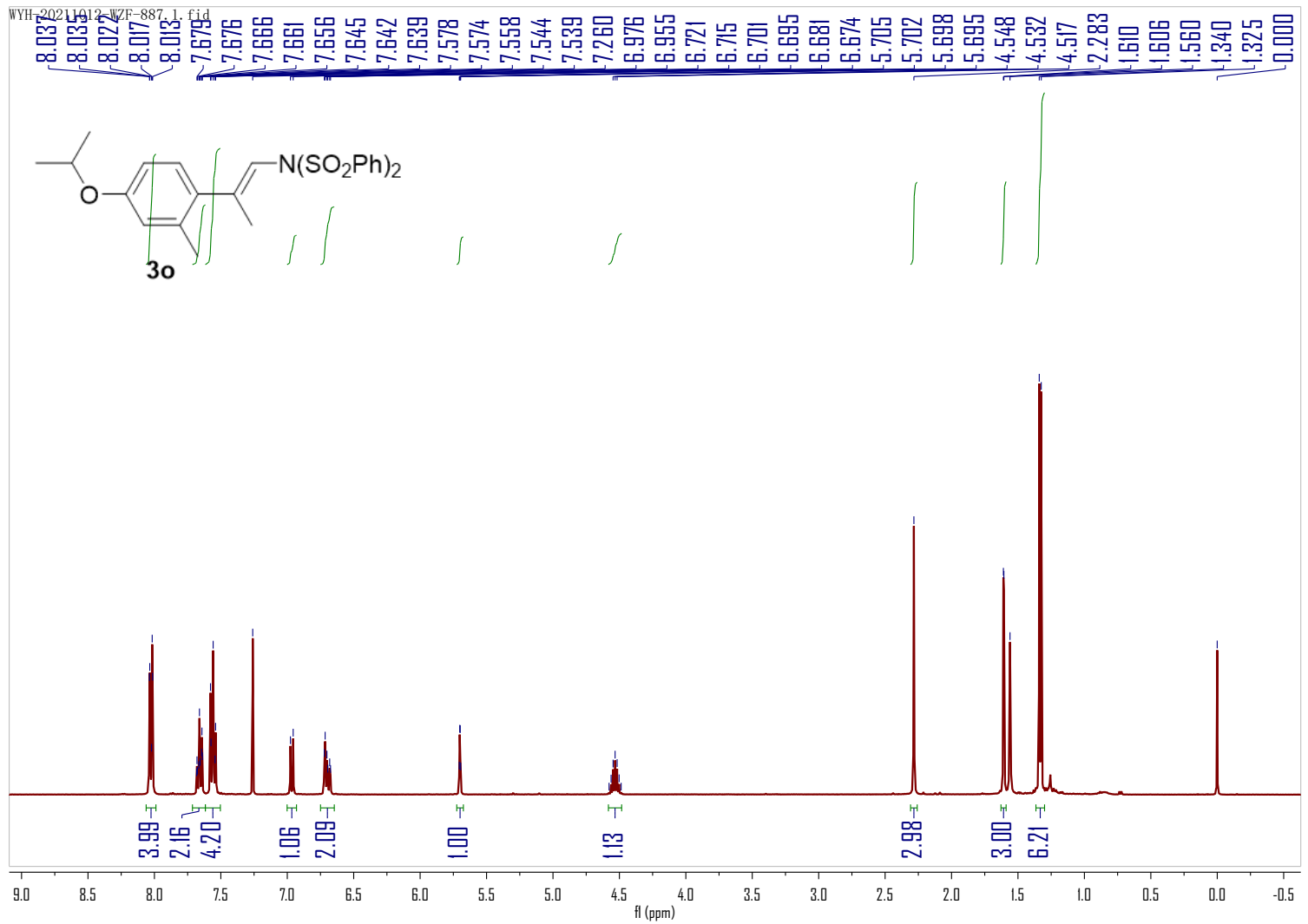


S124

WYH-20211015-WZF-891

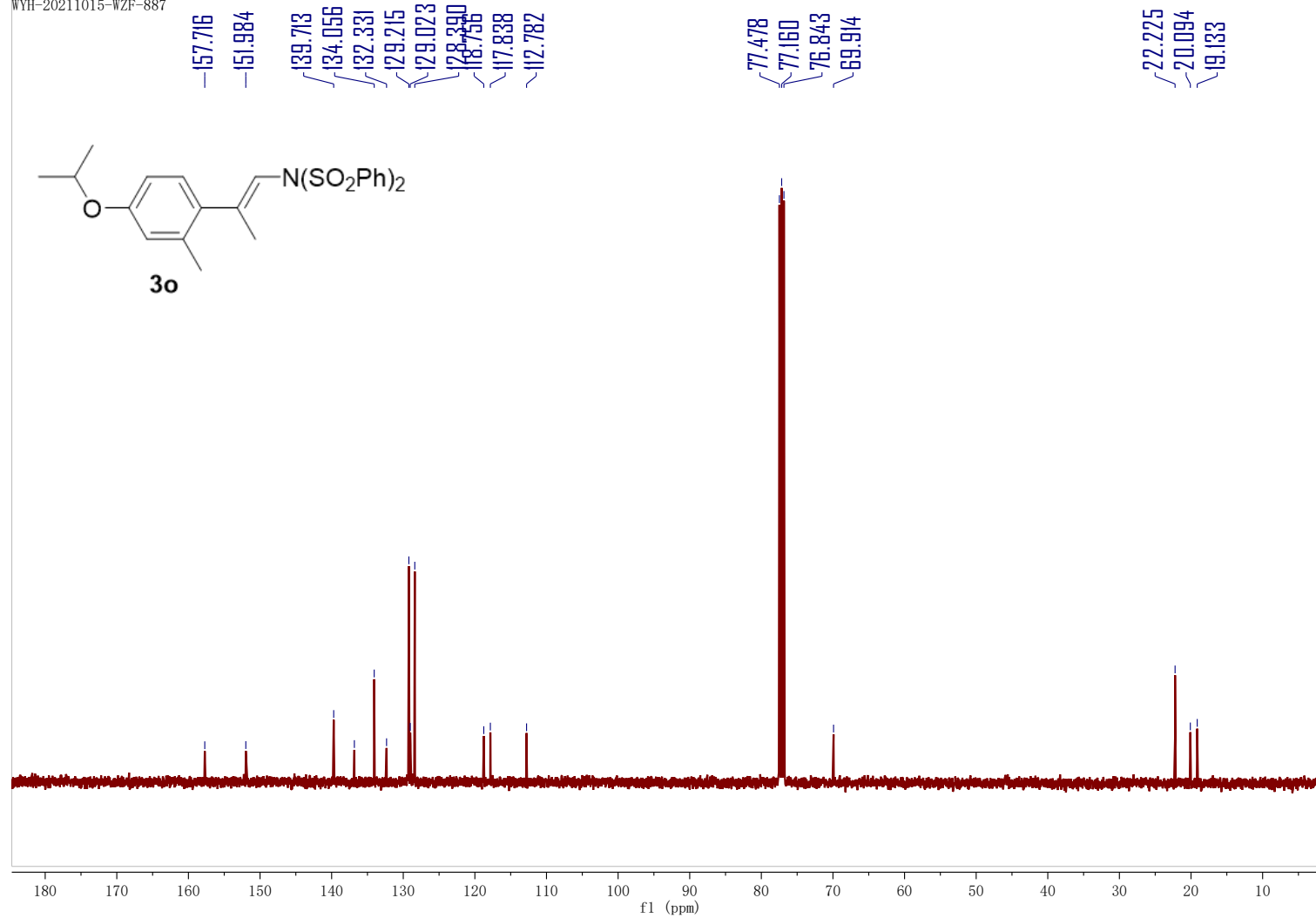


S125

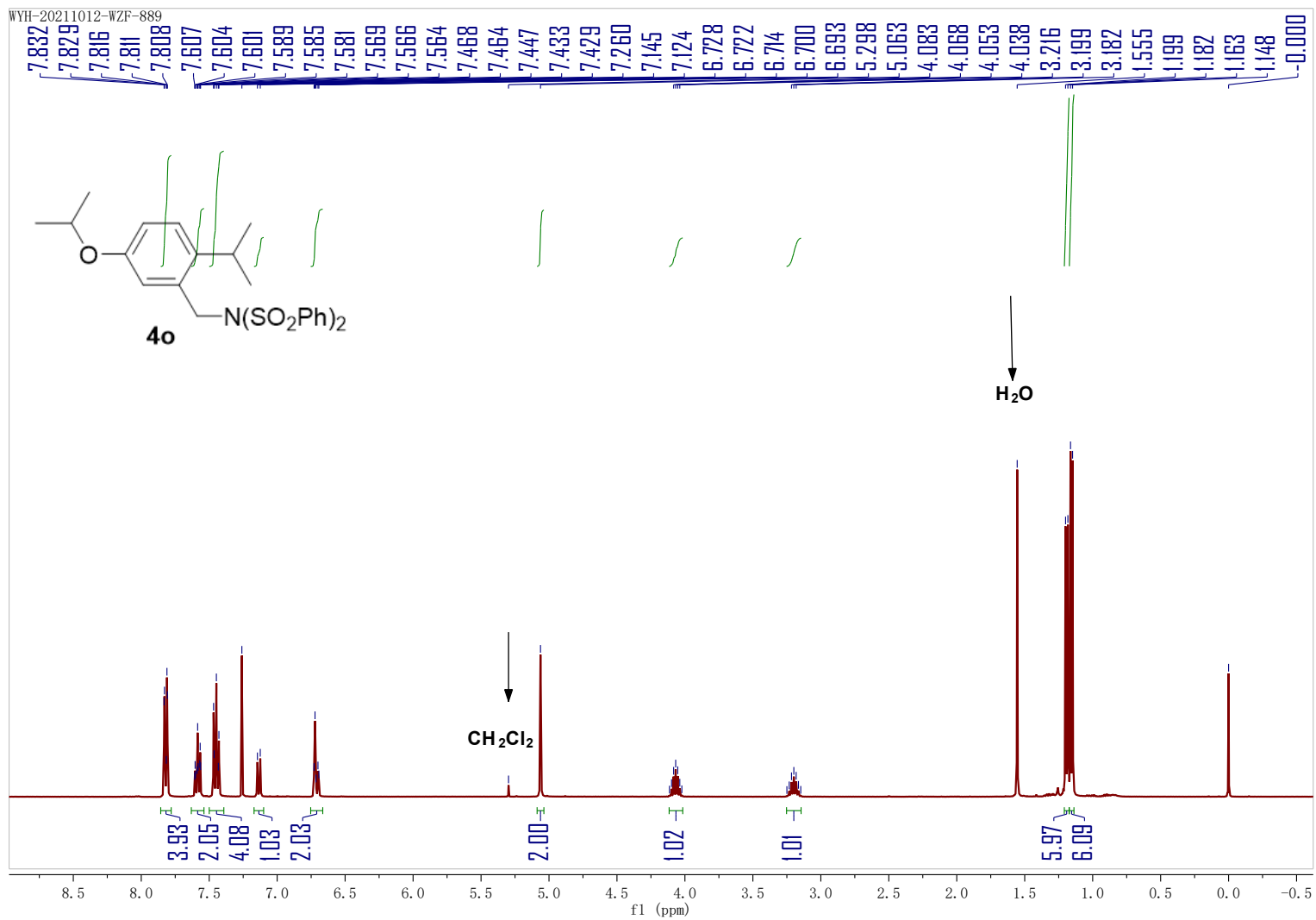


S126

WYH-20211015-WZF-887



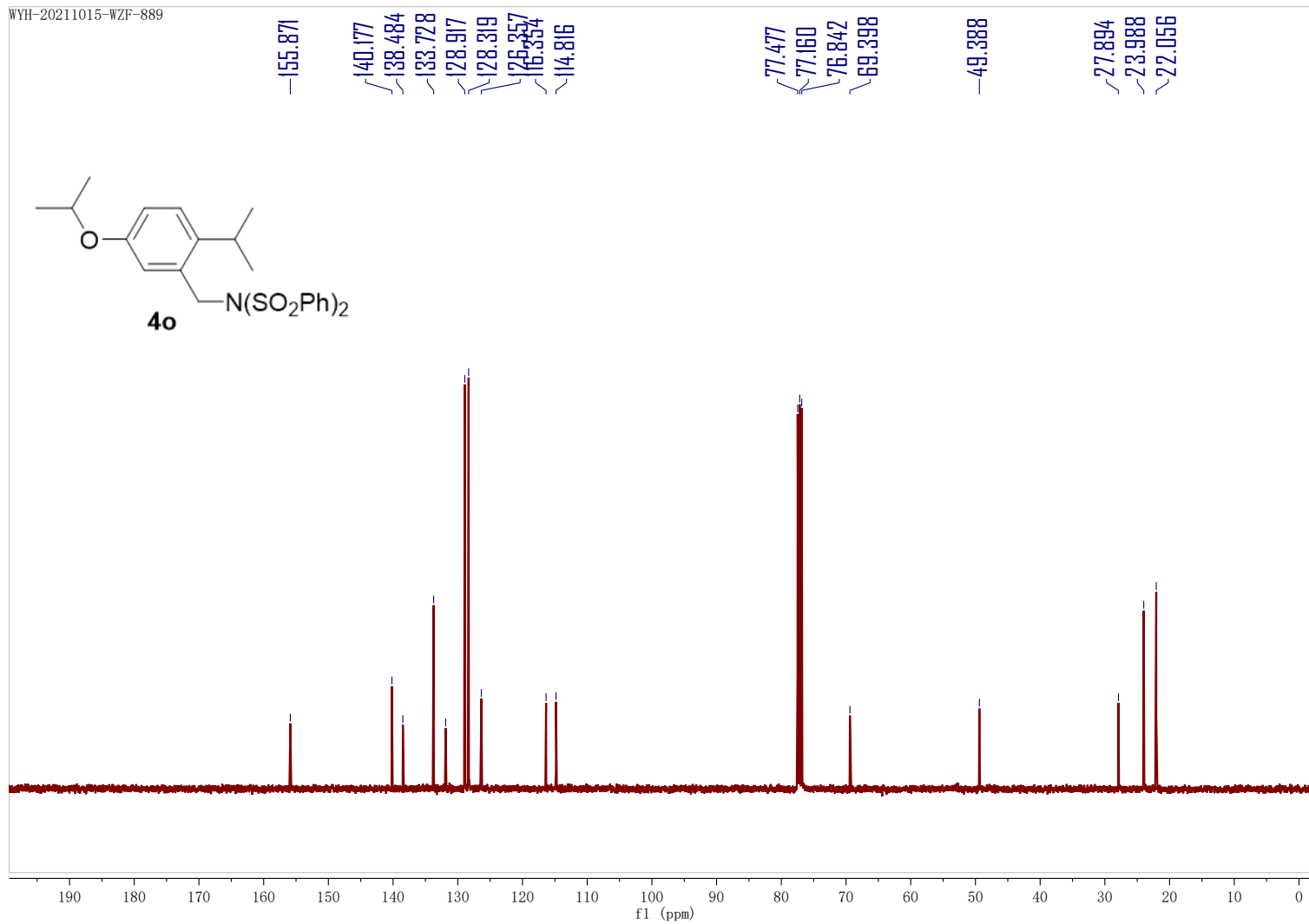
S127



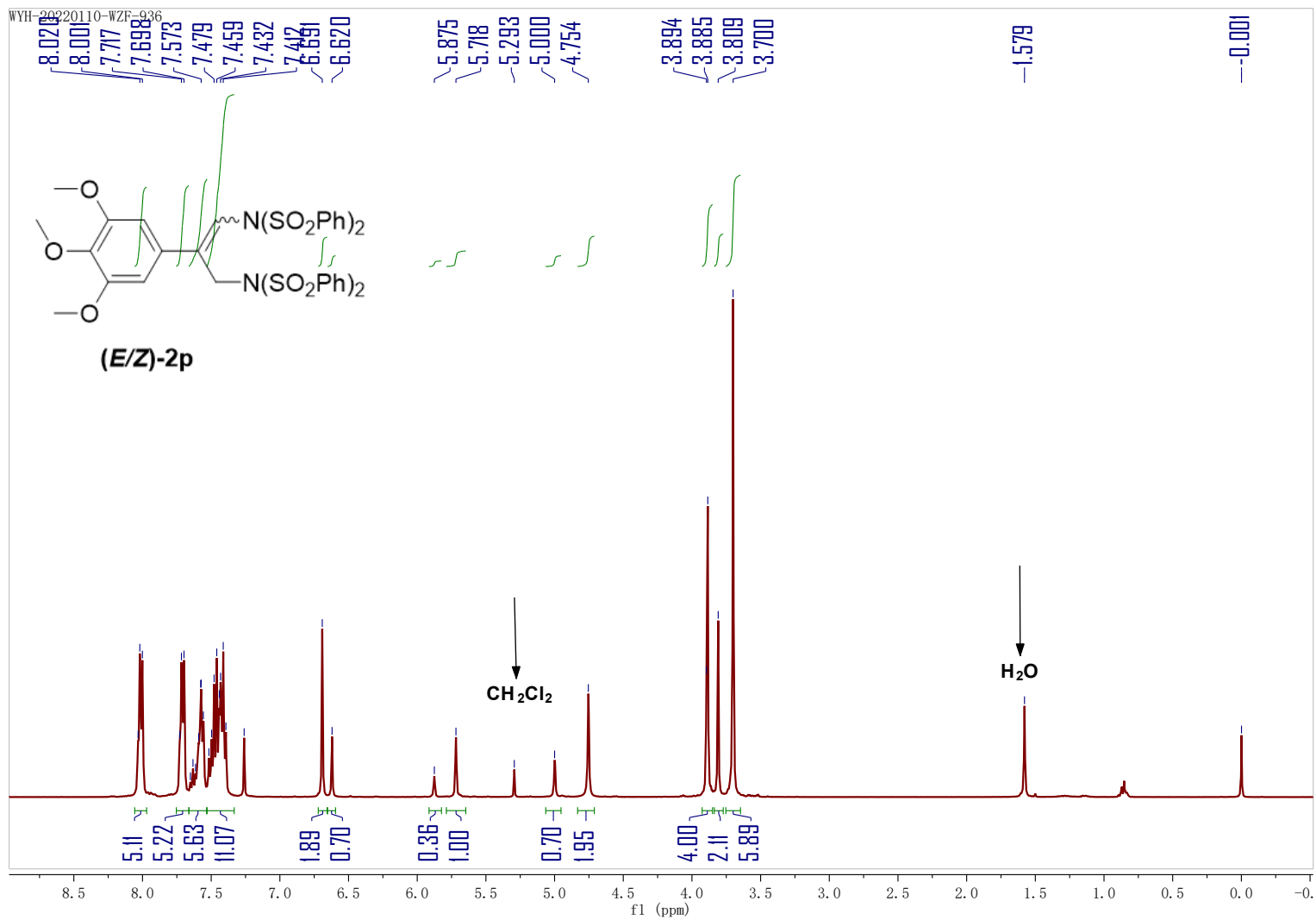
S128



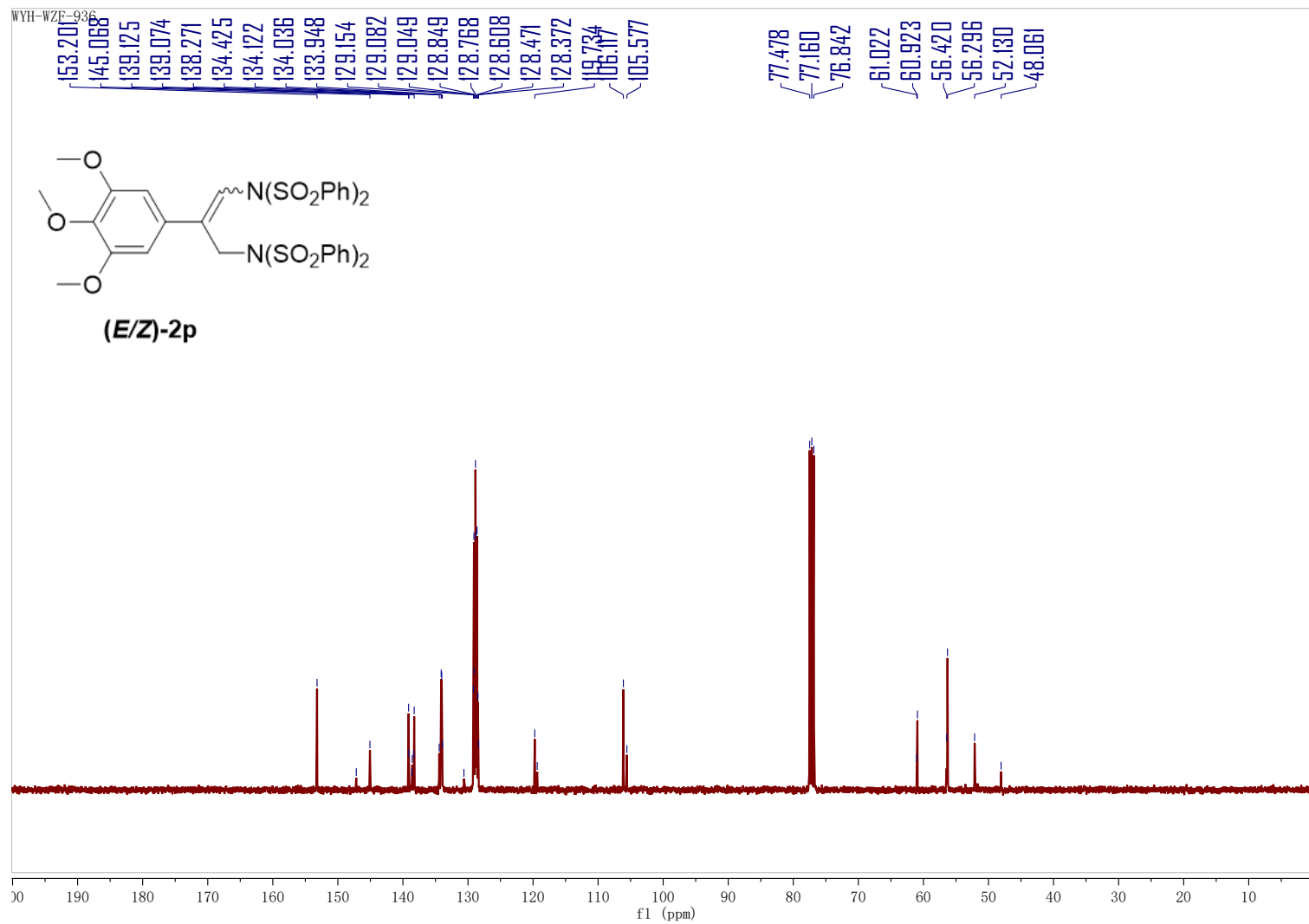
WYH-20211015-WZF-889



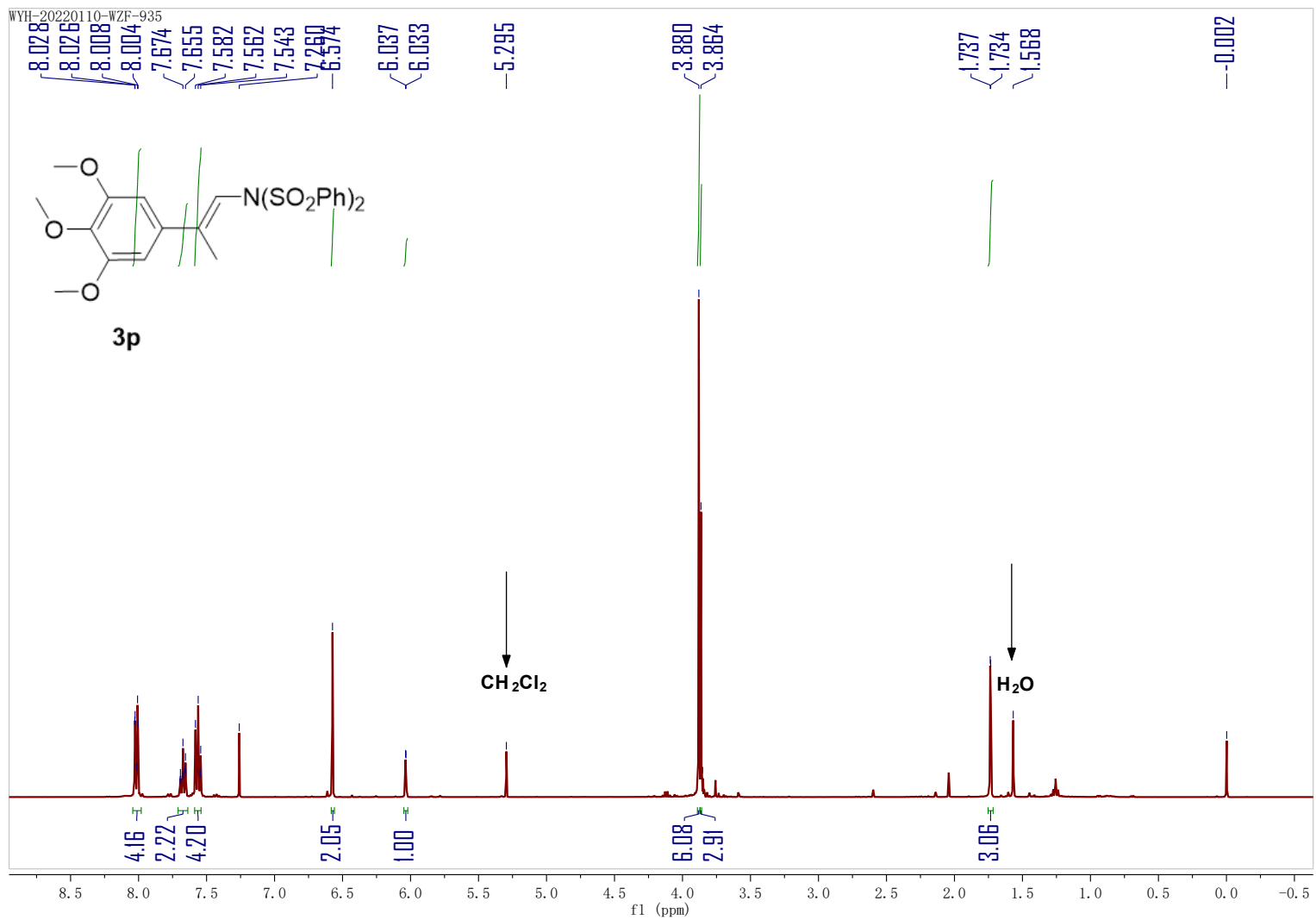
S129



S130

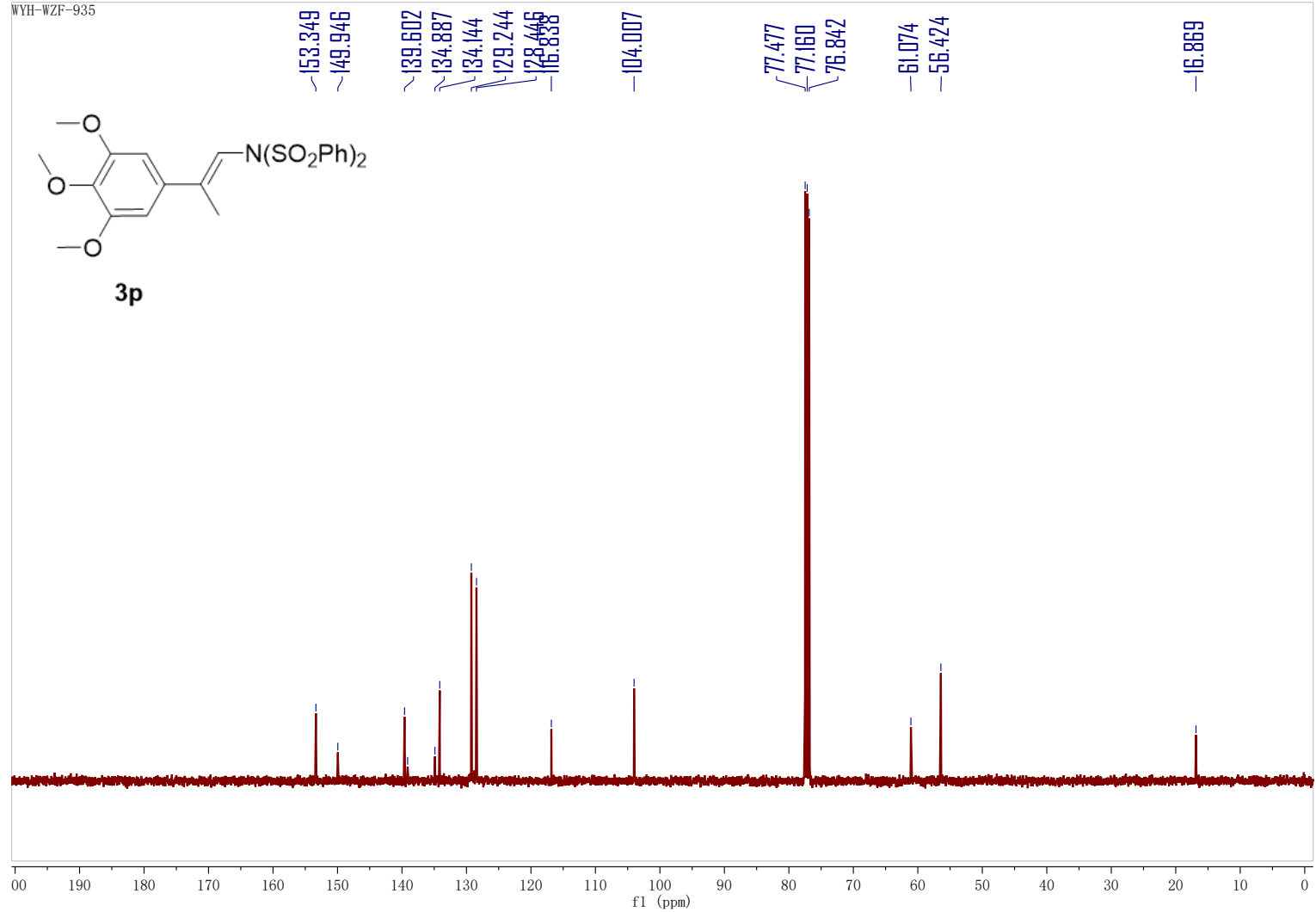
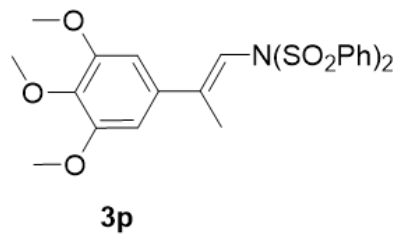


S131

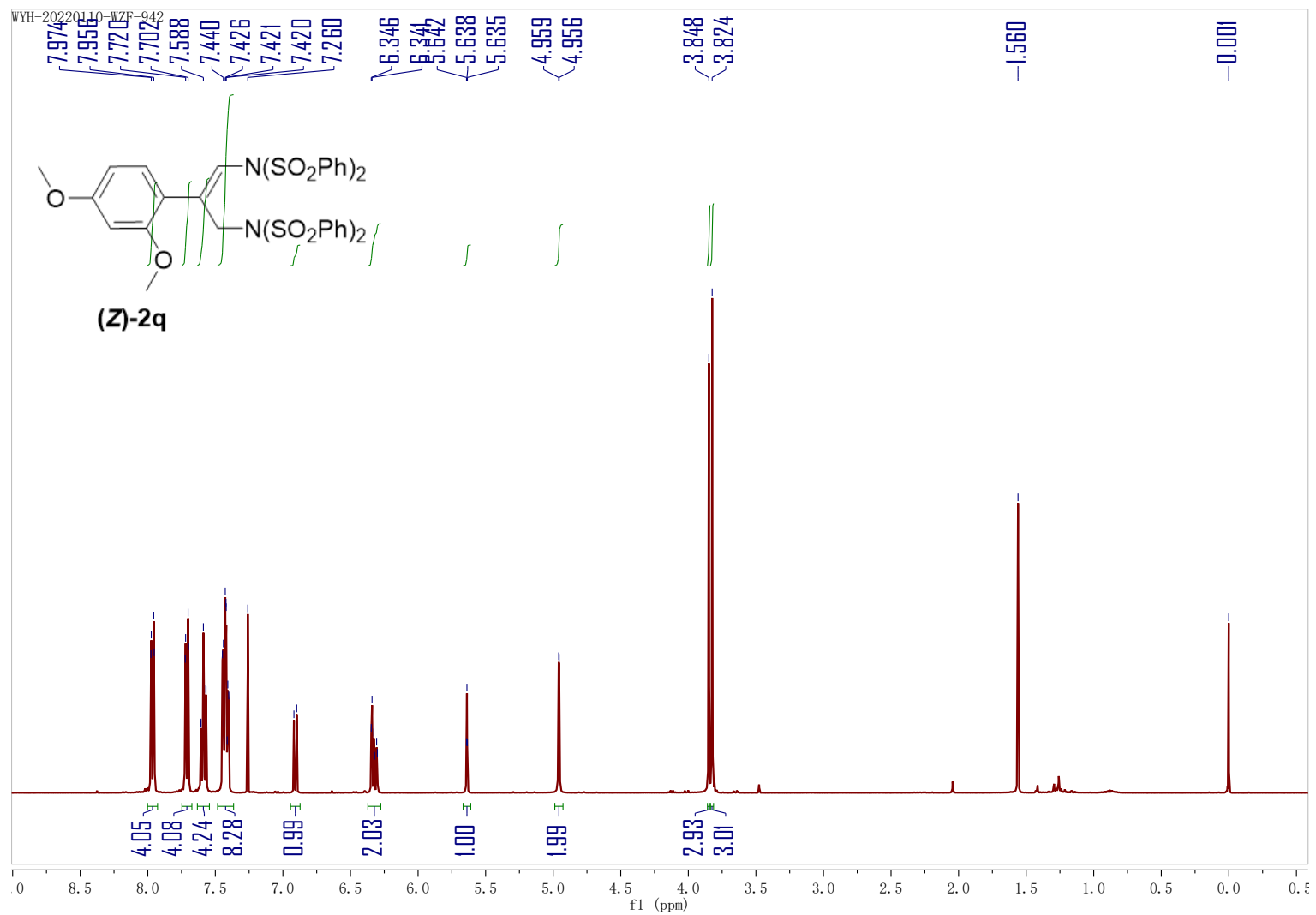


S132

WYH-WZF-935

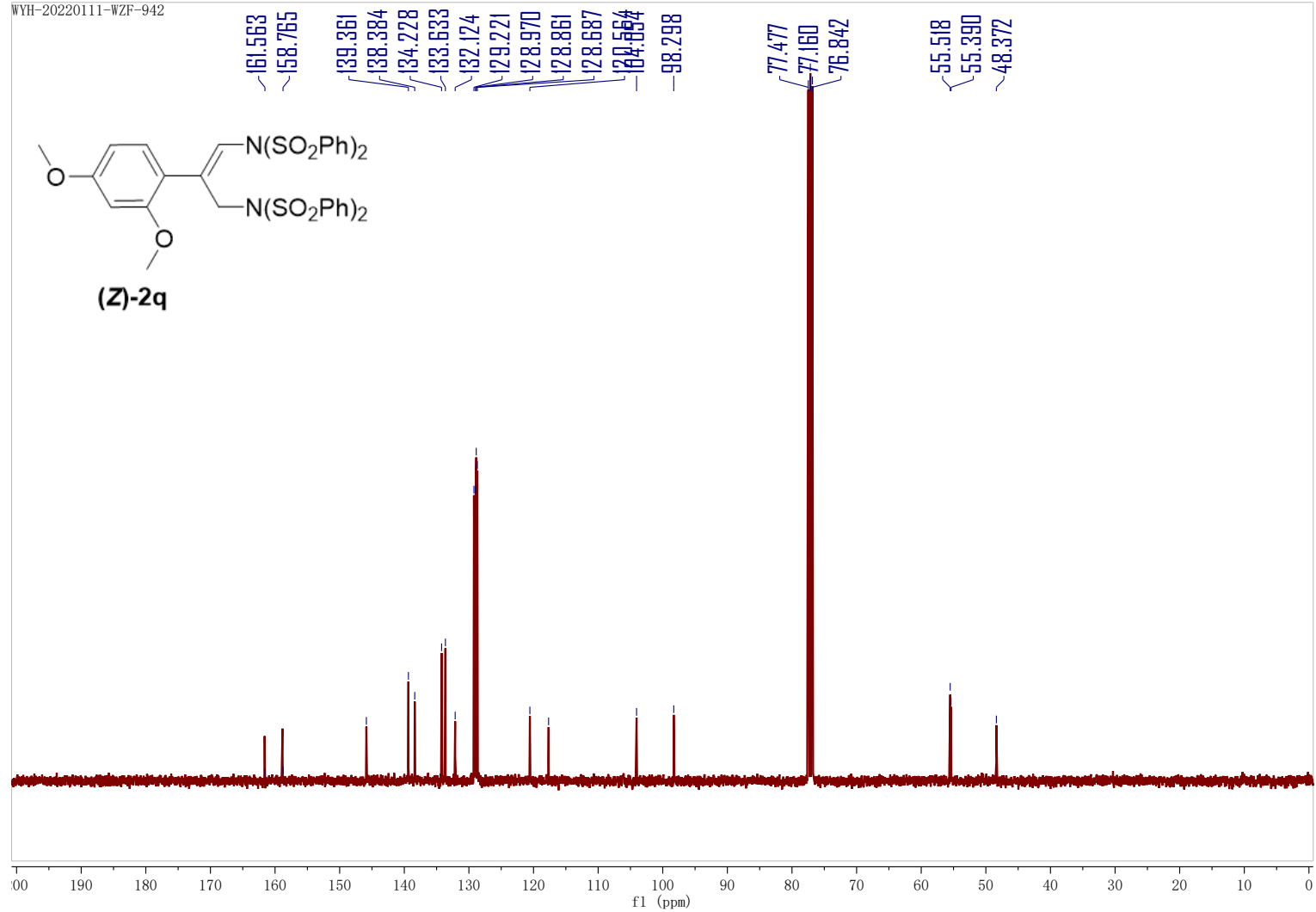
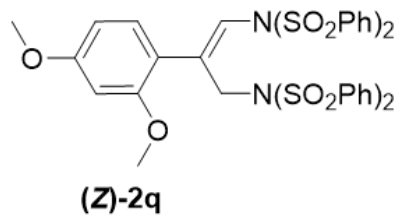


S133

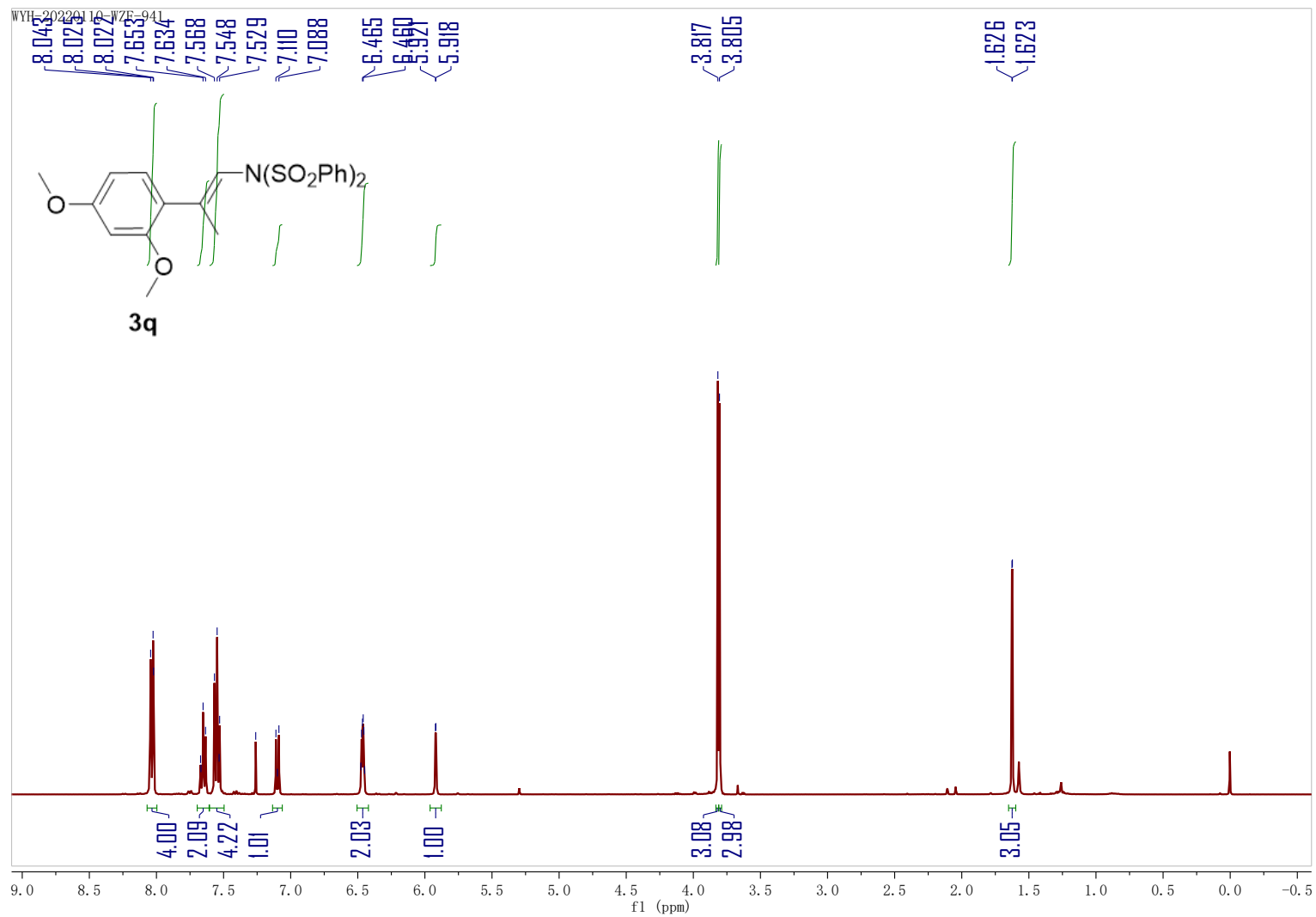


S134

WYH-20220111-WZF-942



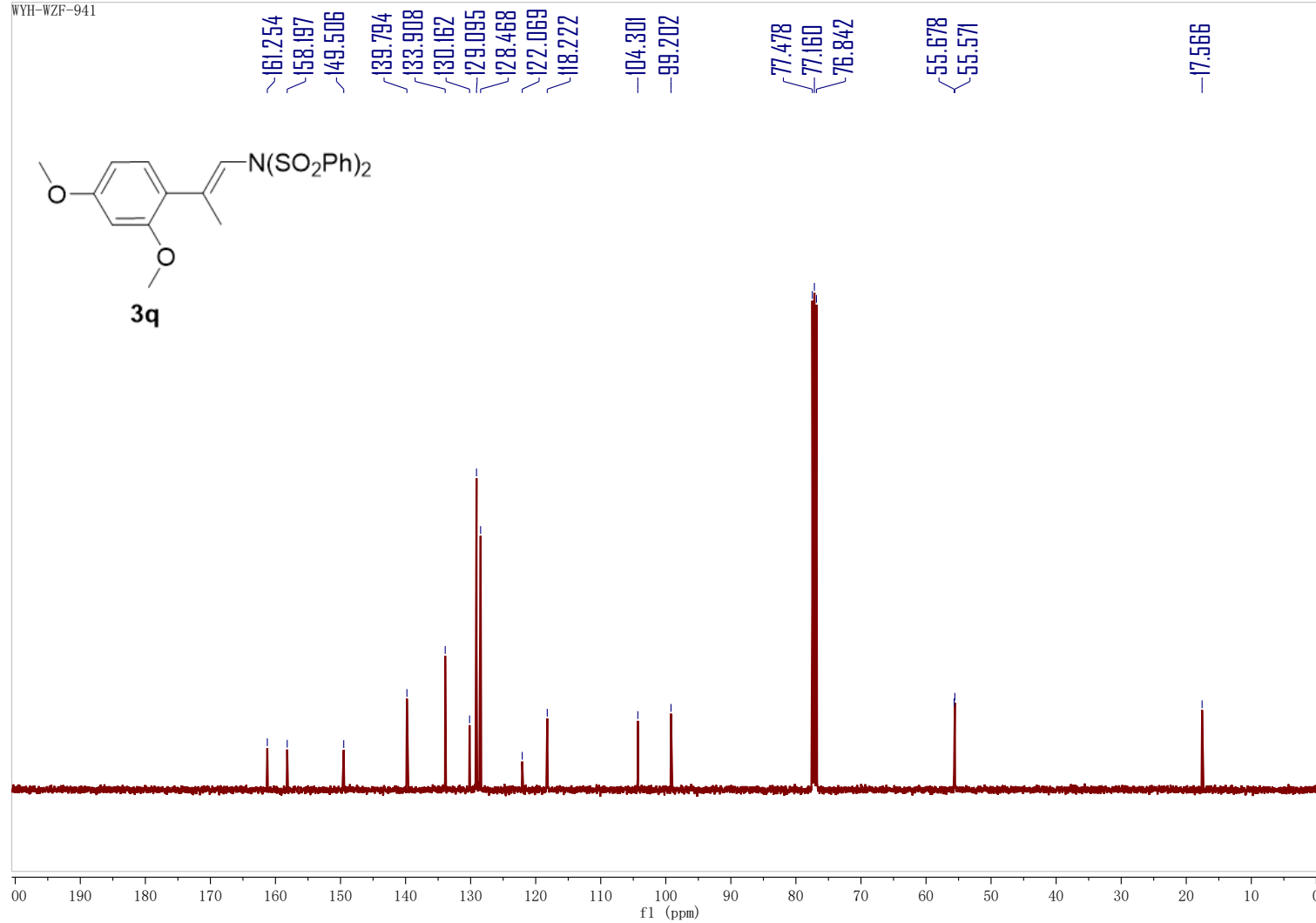
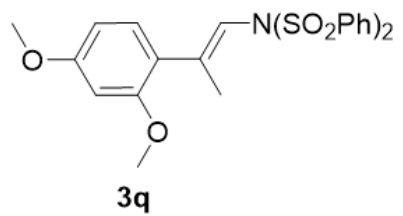
S135



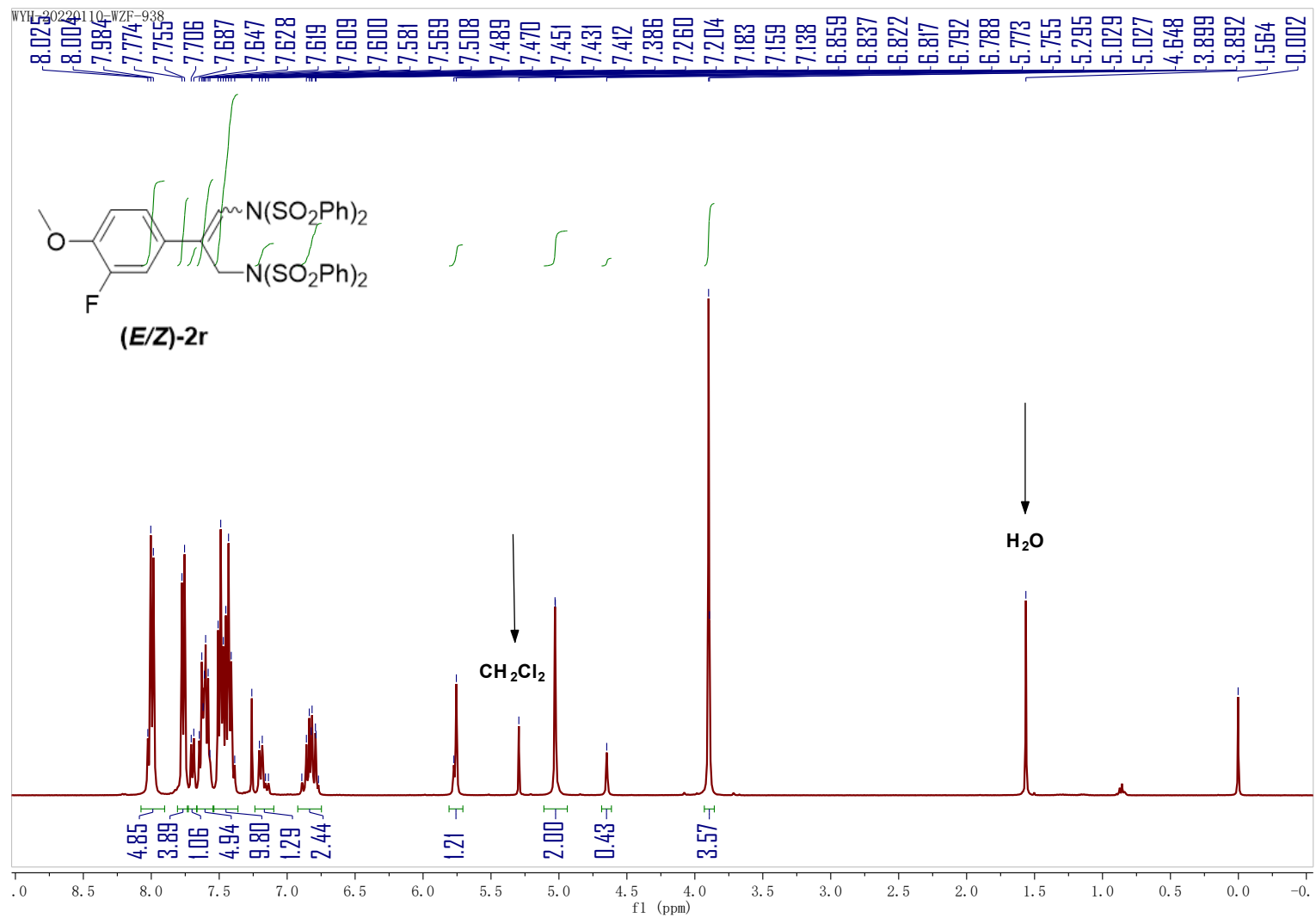
S136



WYH-WZF-941

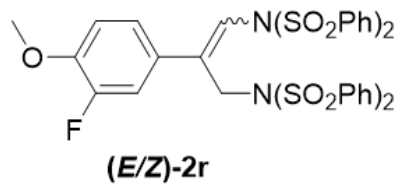


S137

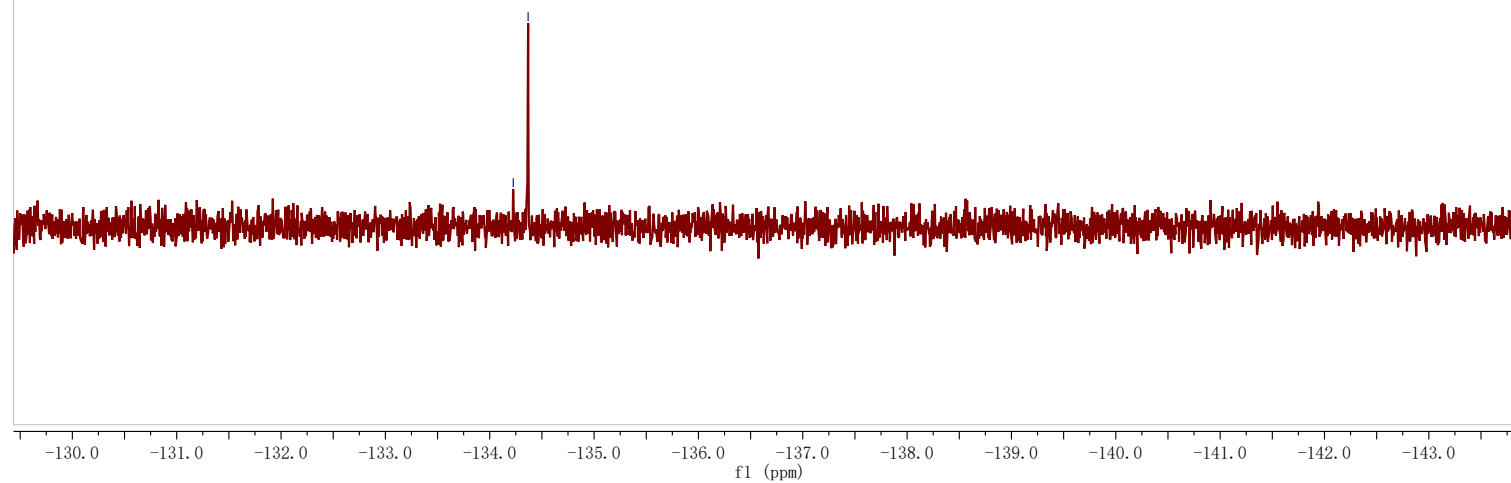


S138

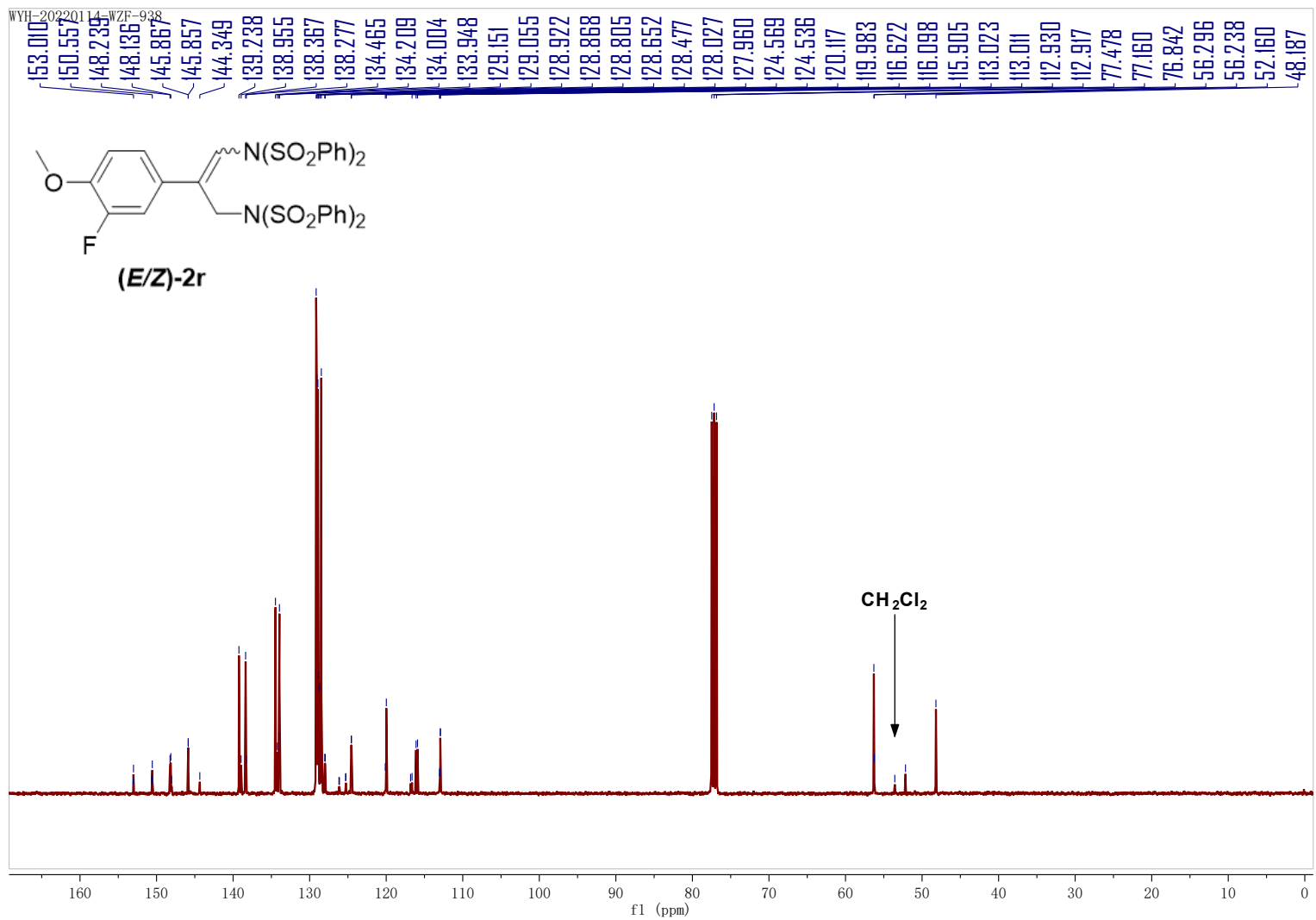
WYH-20220110-WZF-938



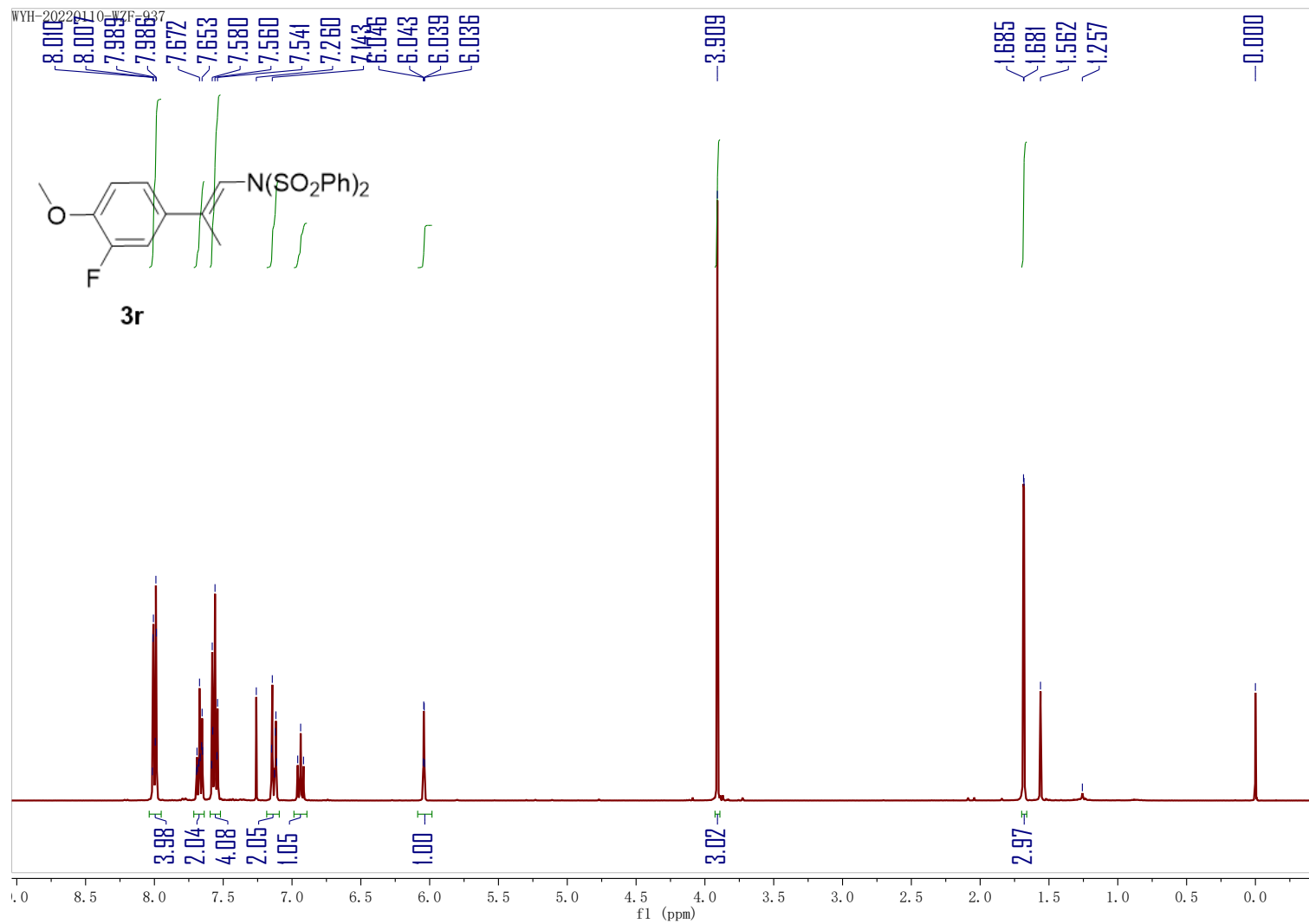
134.225  
134.358



S139

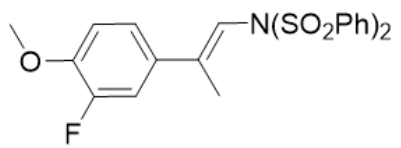


S140



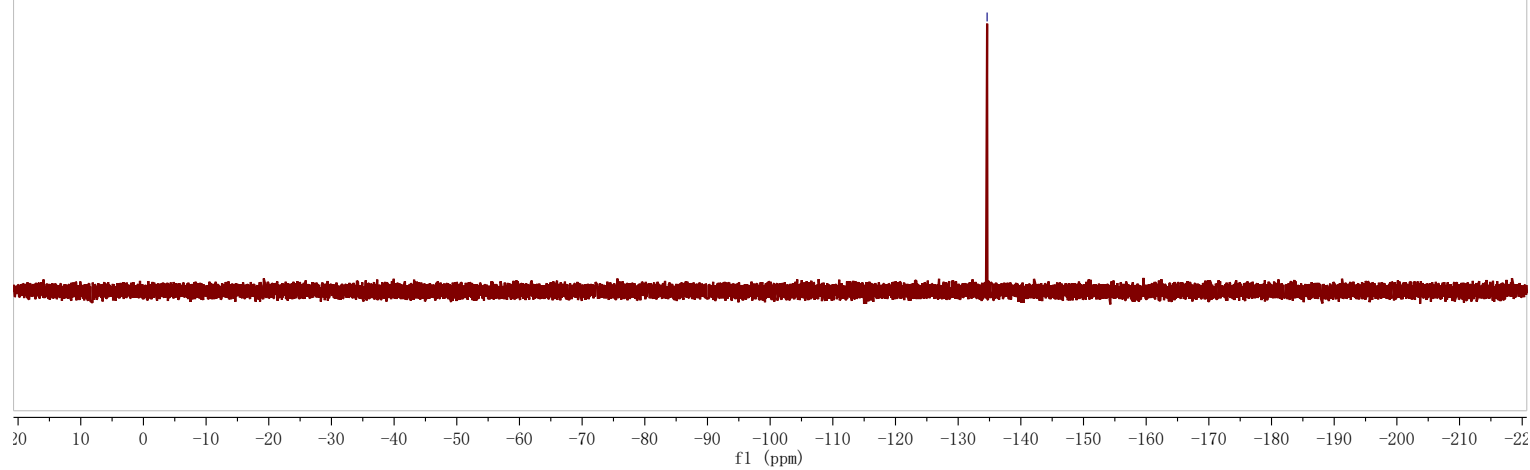
S141

WYH-20220111-WZF-937

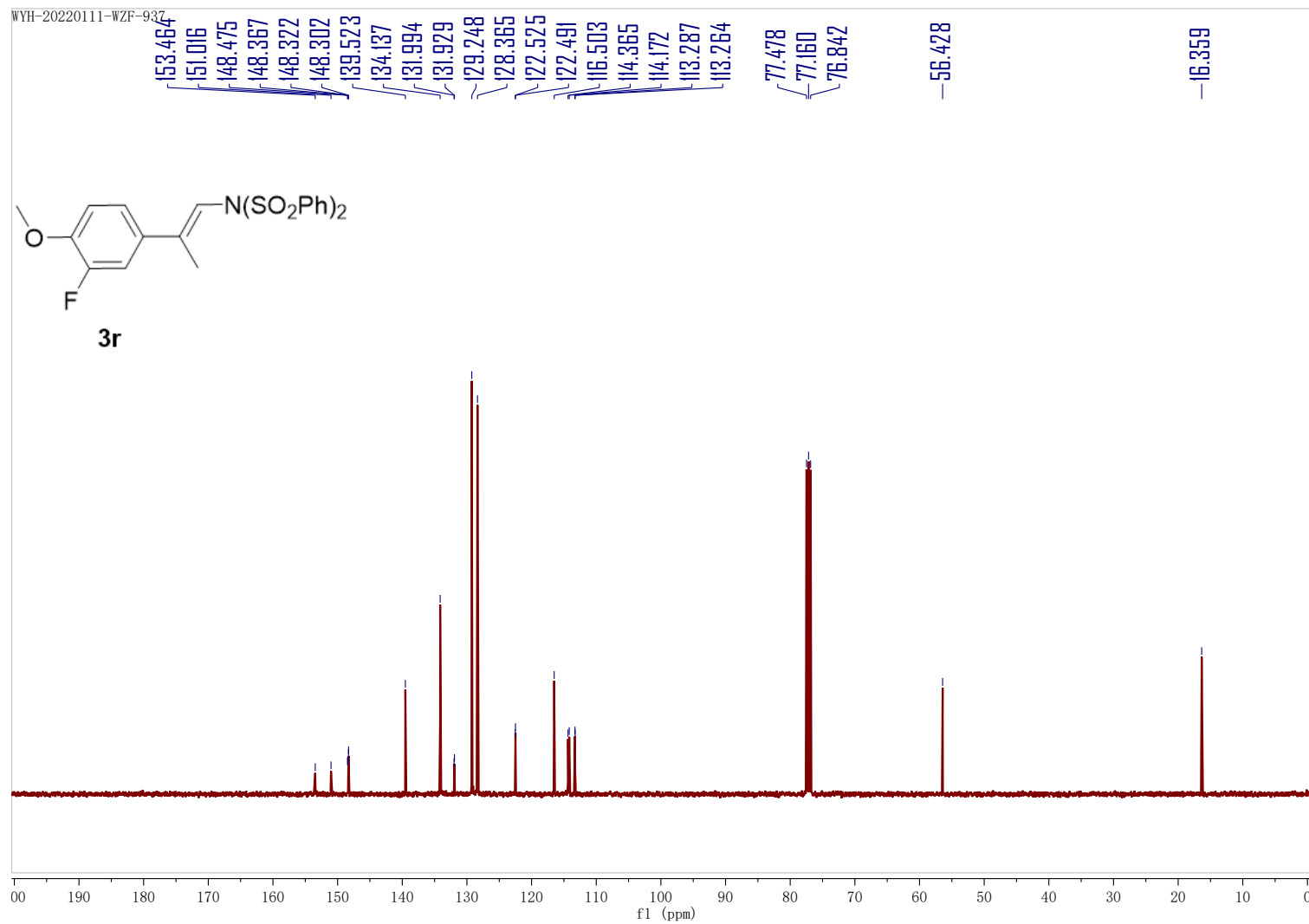


**3r**

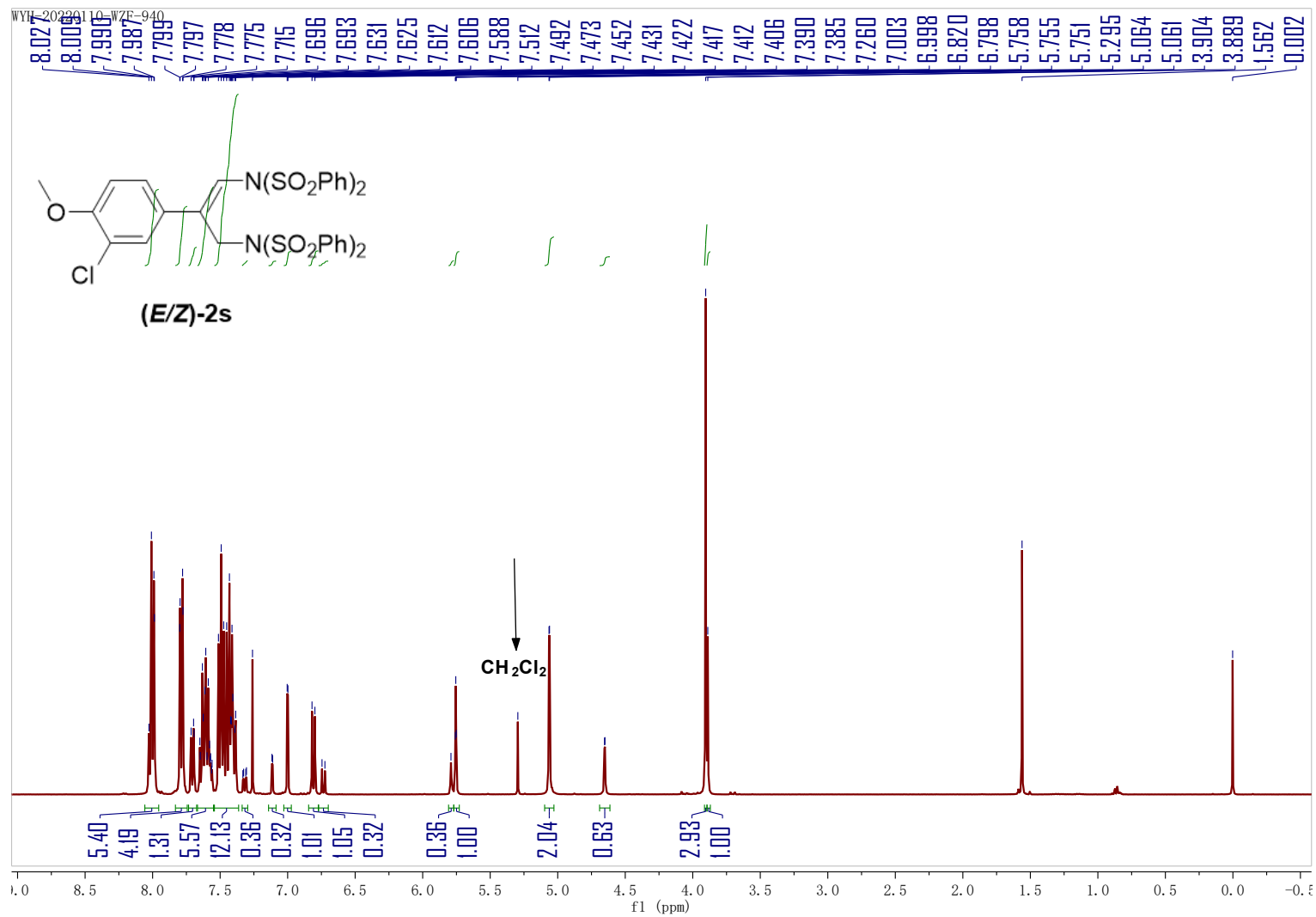
134.618



S142



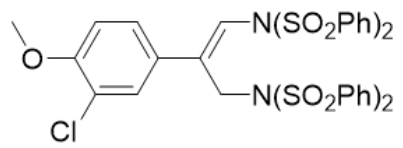
S143



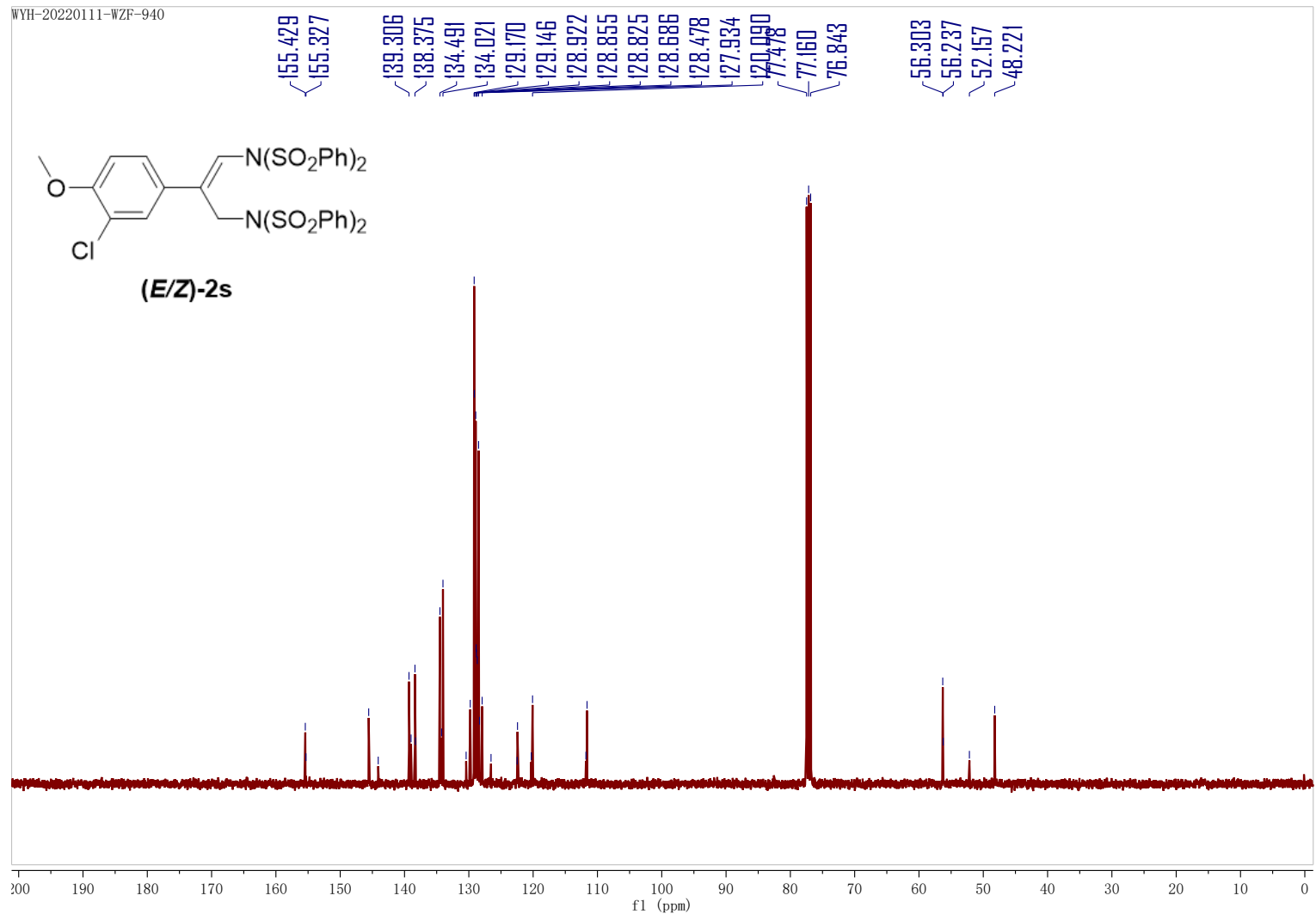
S144



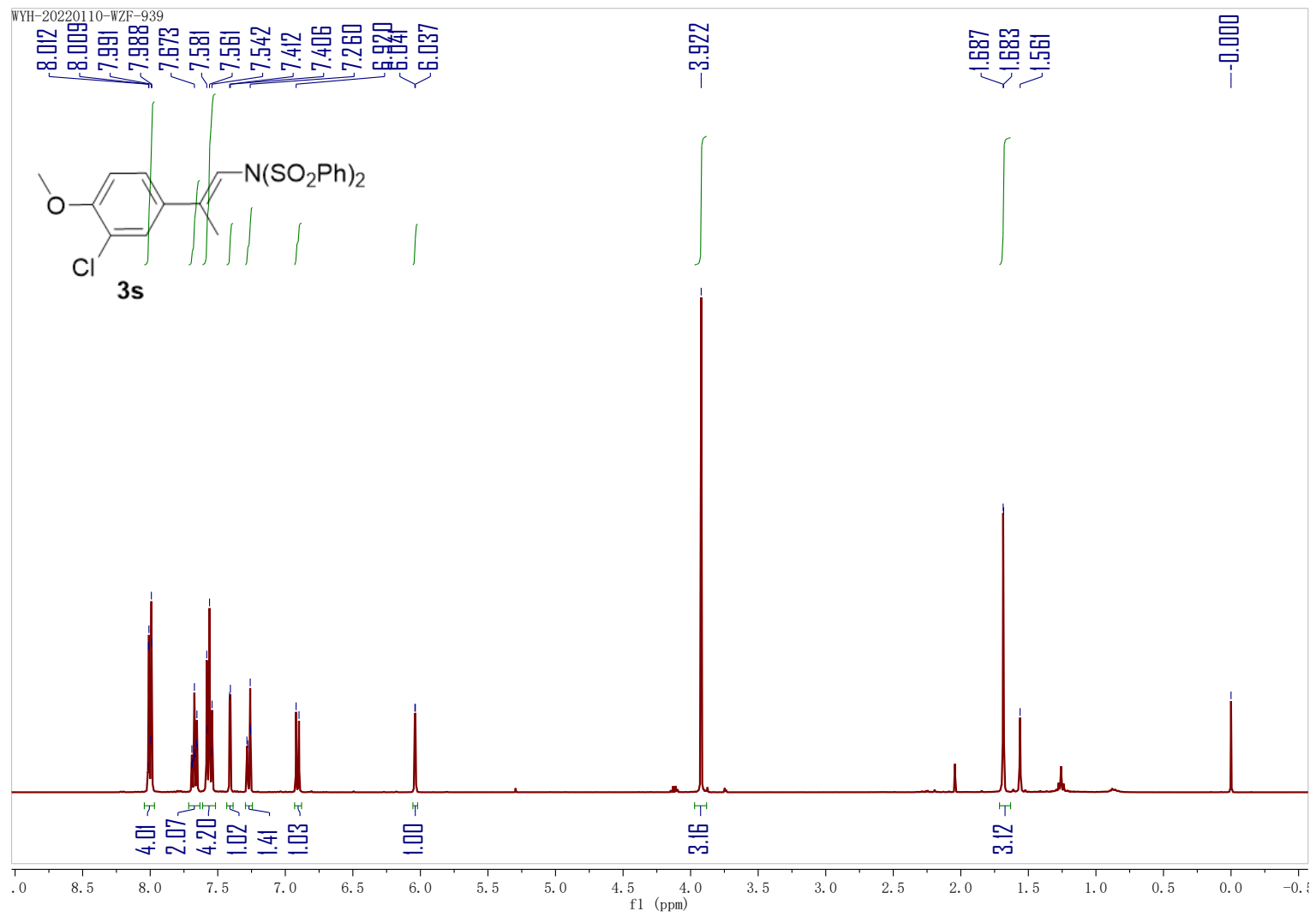
WYH-20220111-WZF-940



(E/Z)-2s

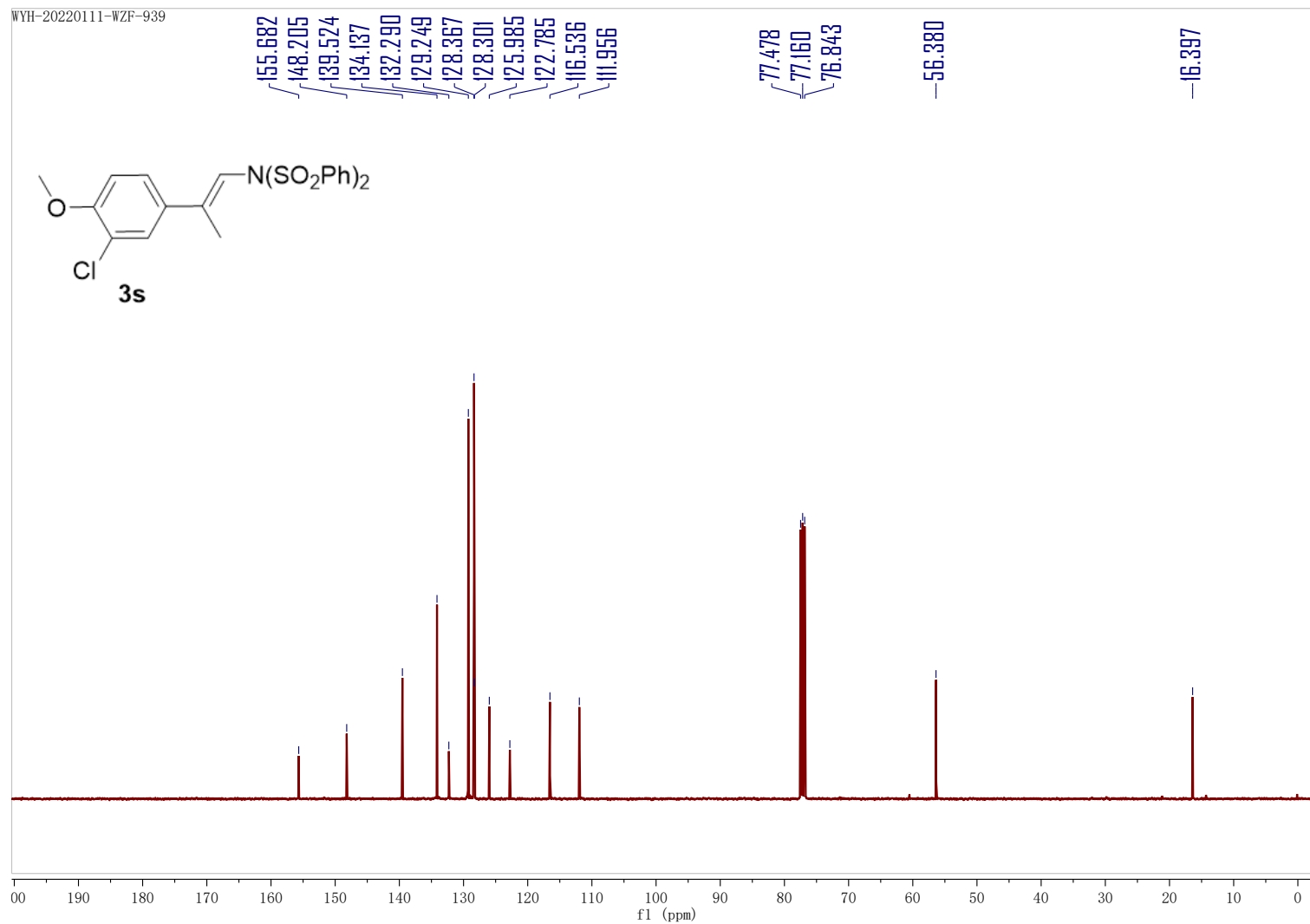


S145

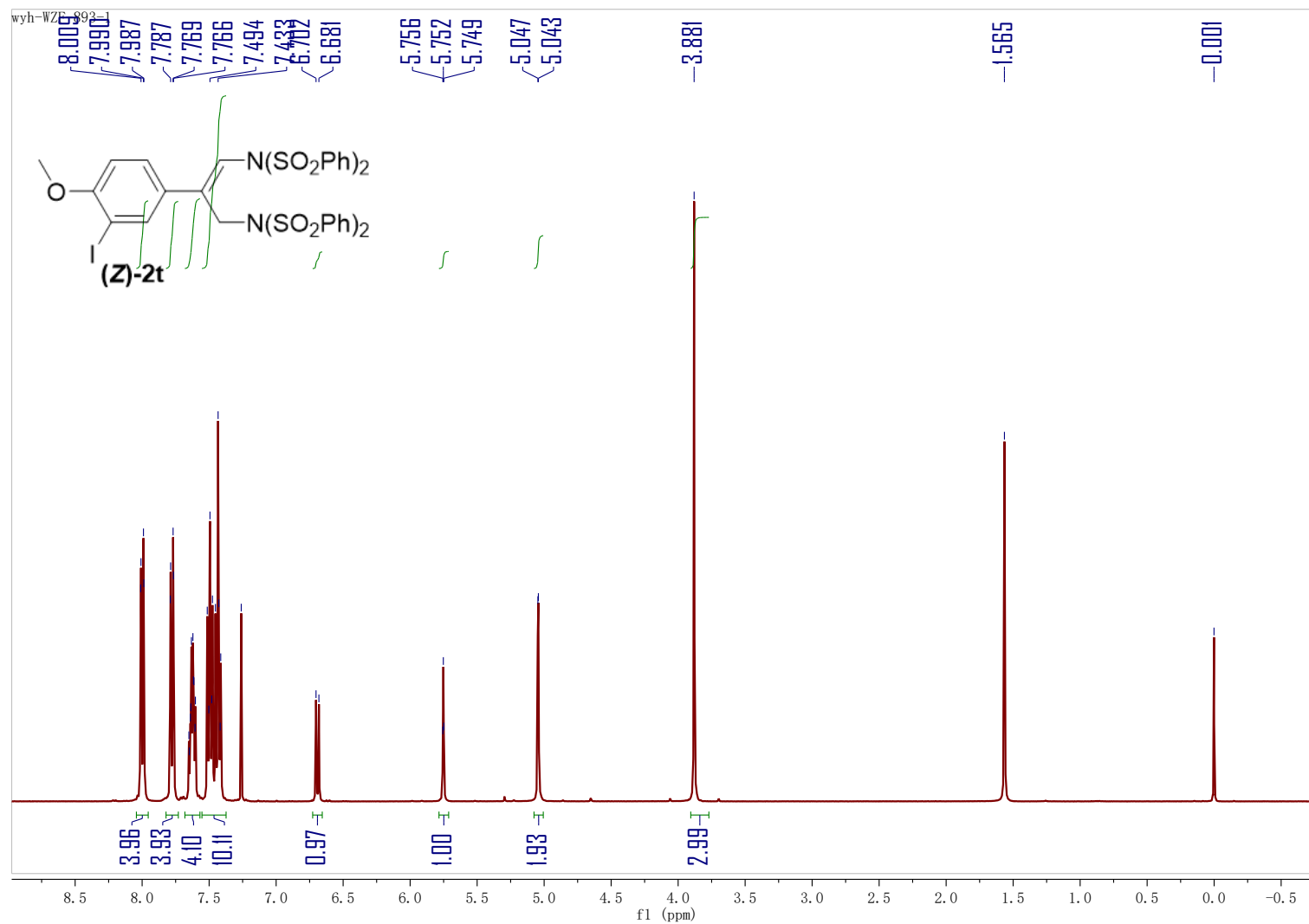


S146

WYH-20220111-WZF-939

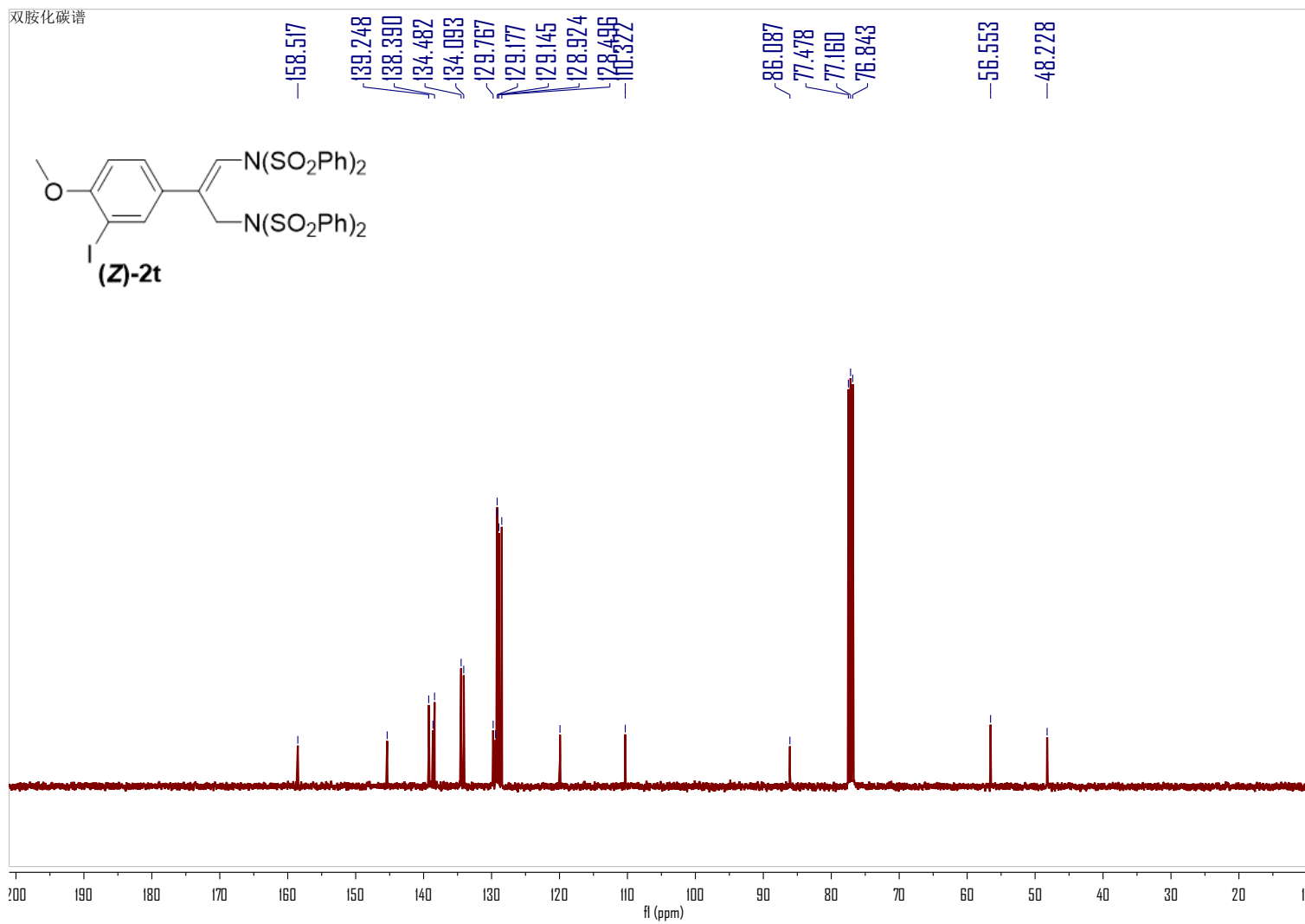


S147

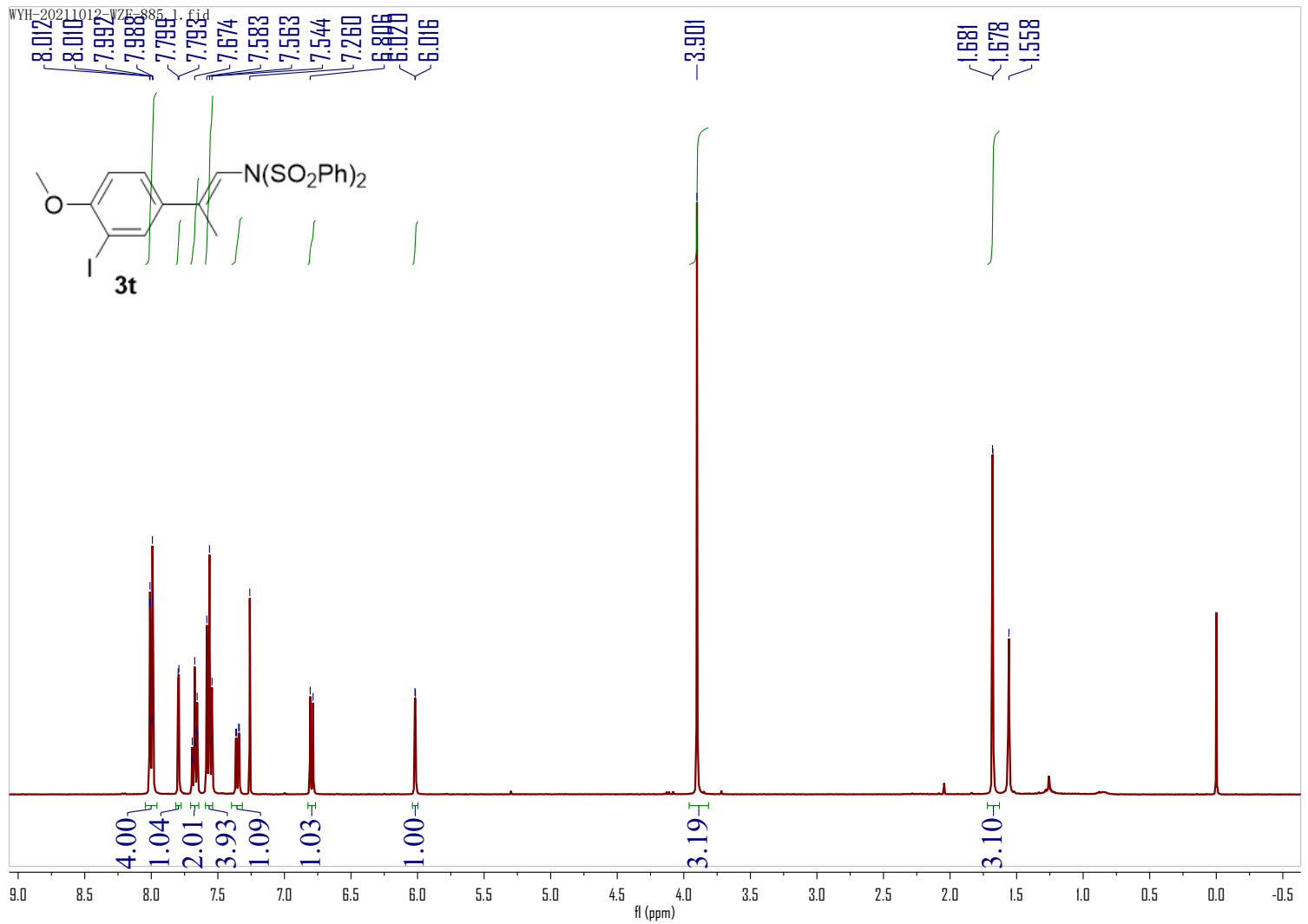


S148

双胺化碳谱

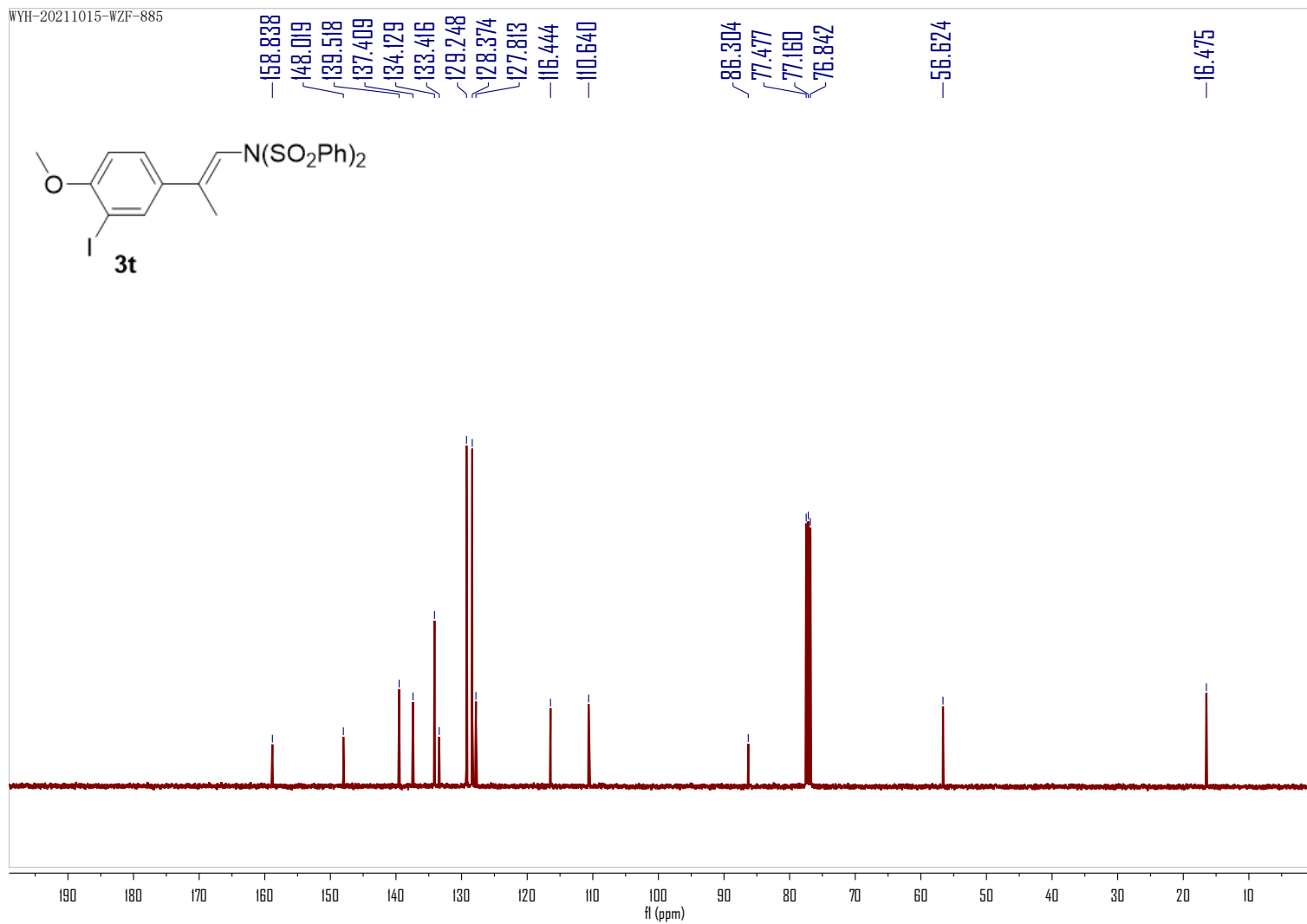
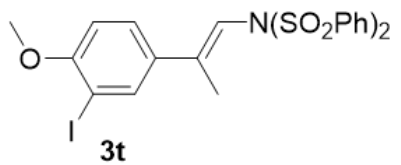


S149

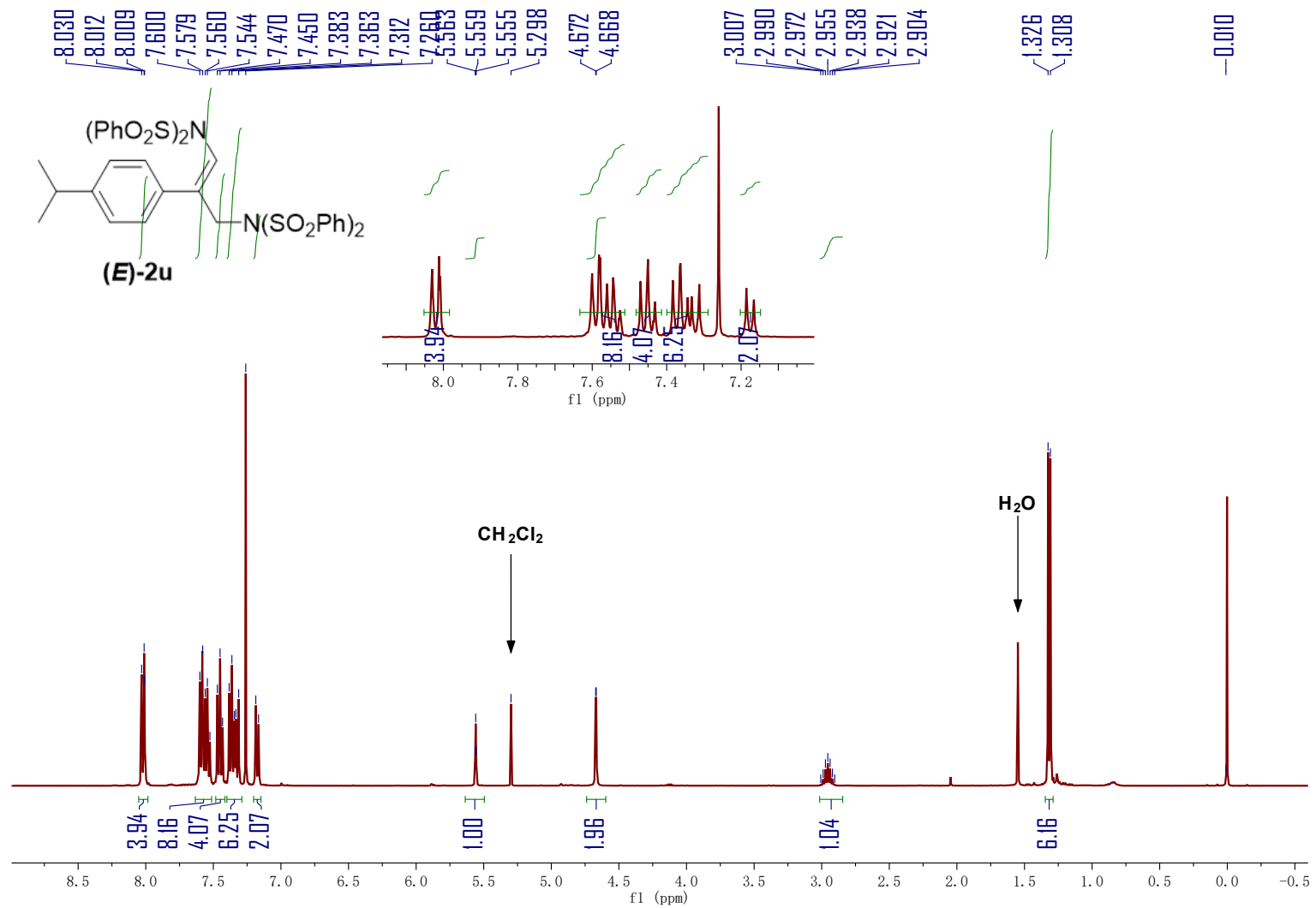


S150

WYH-20211015-WZF-885

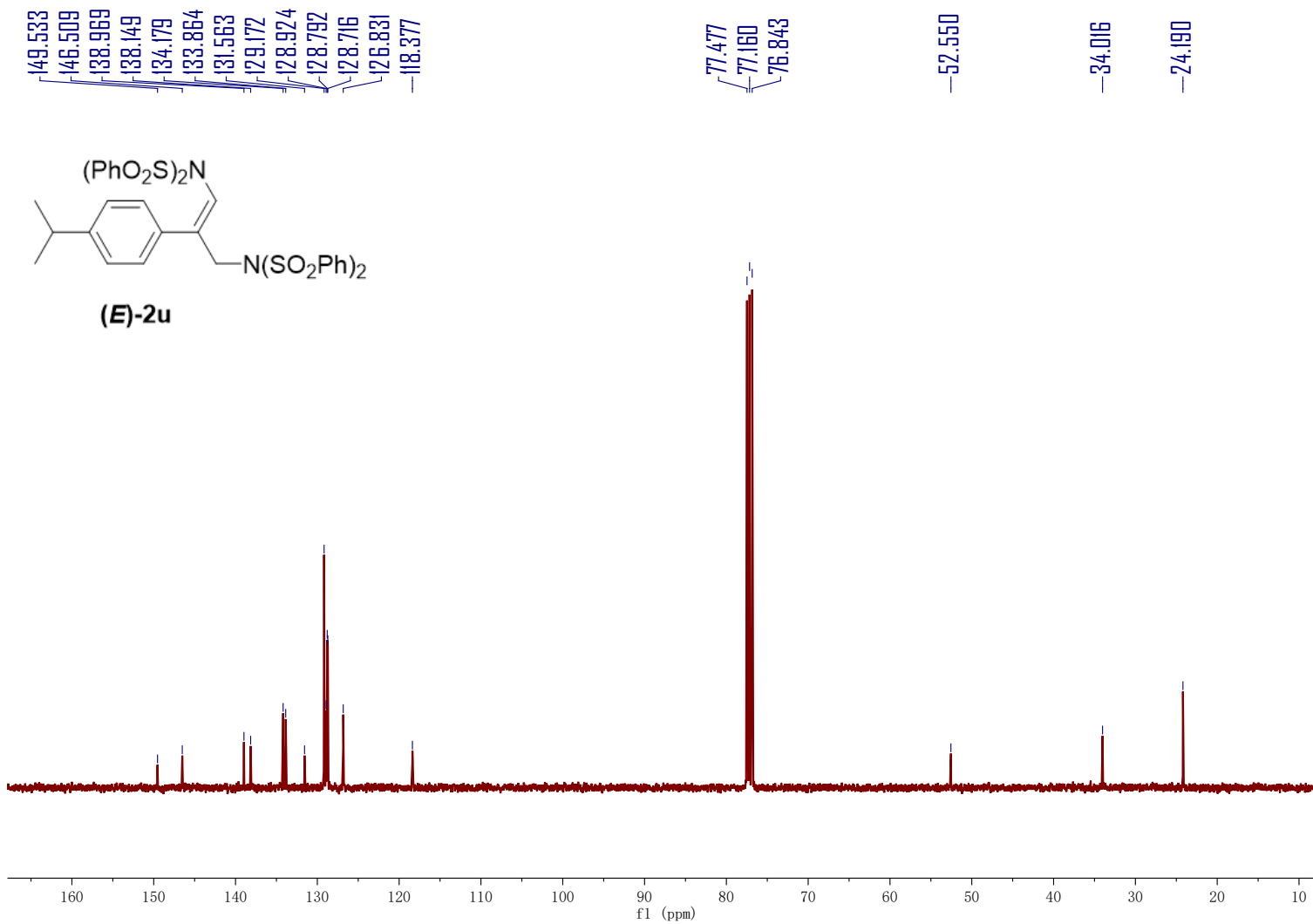


S151

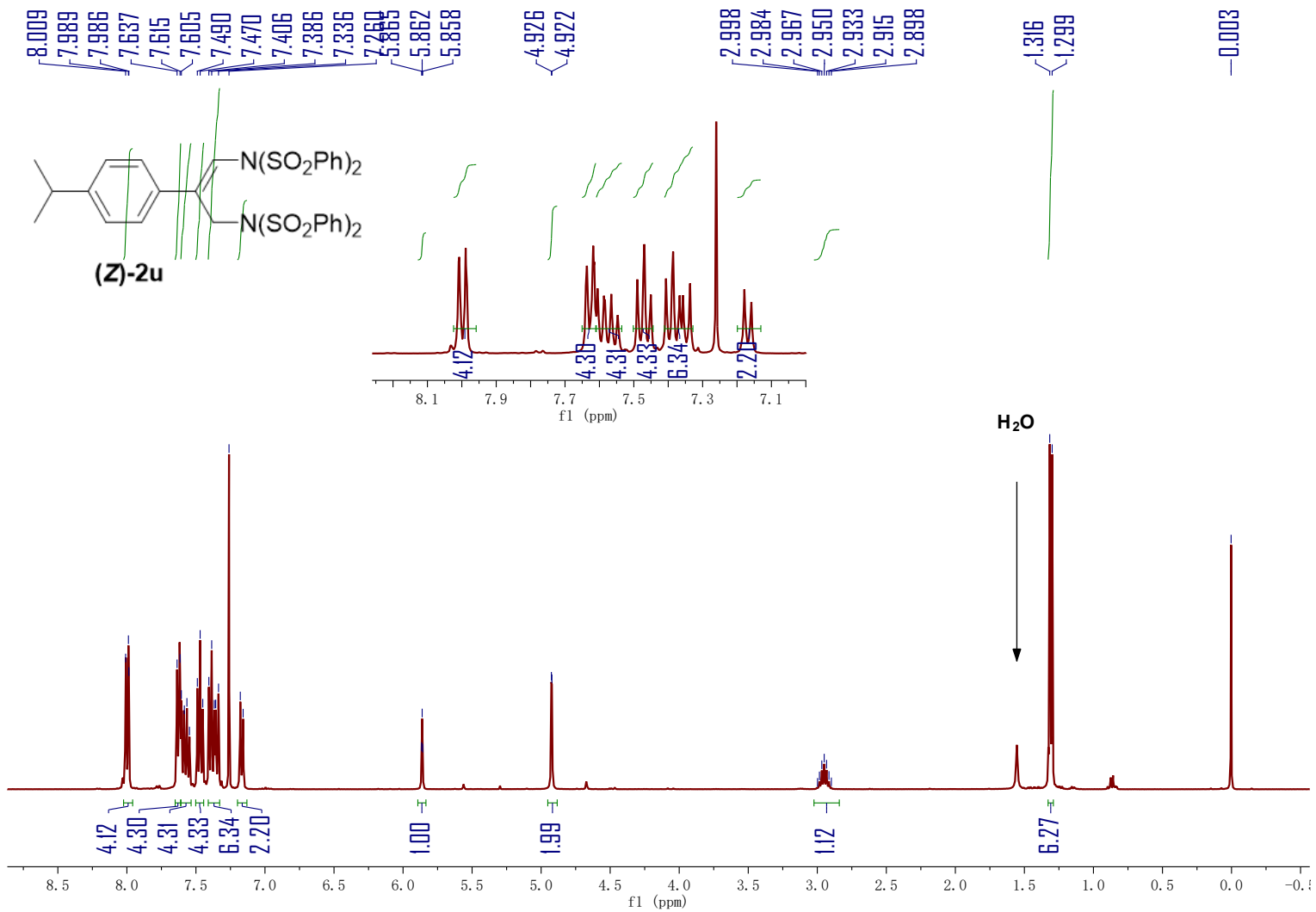


S152

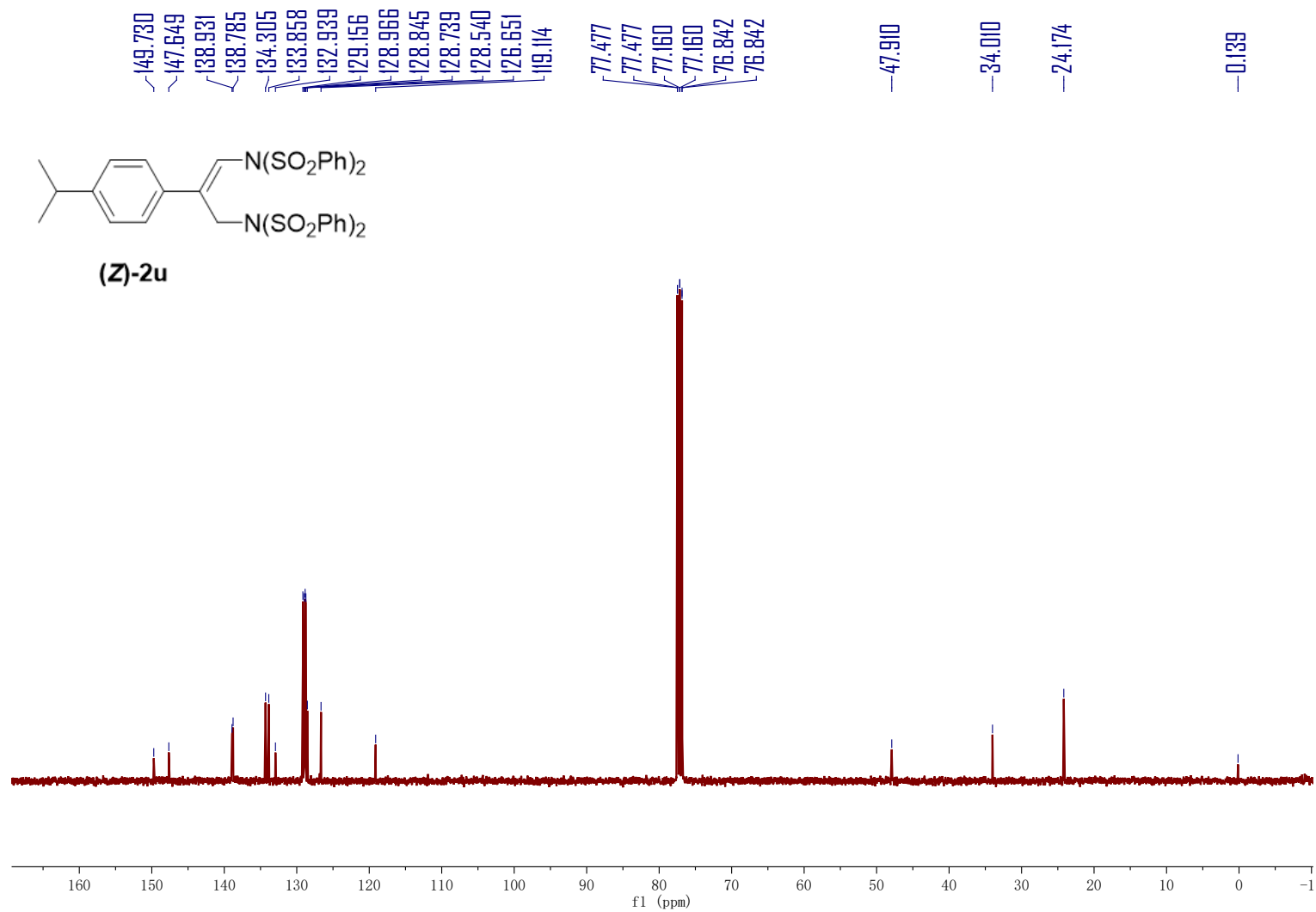




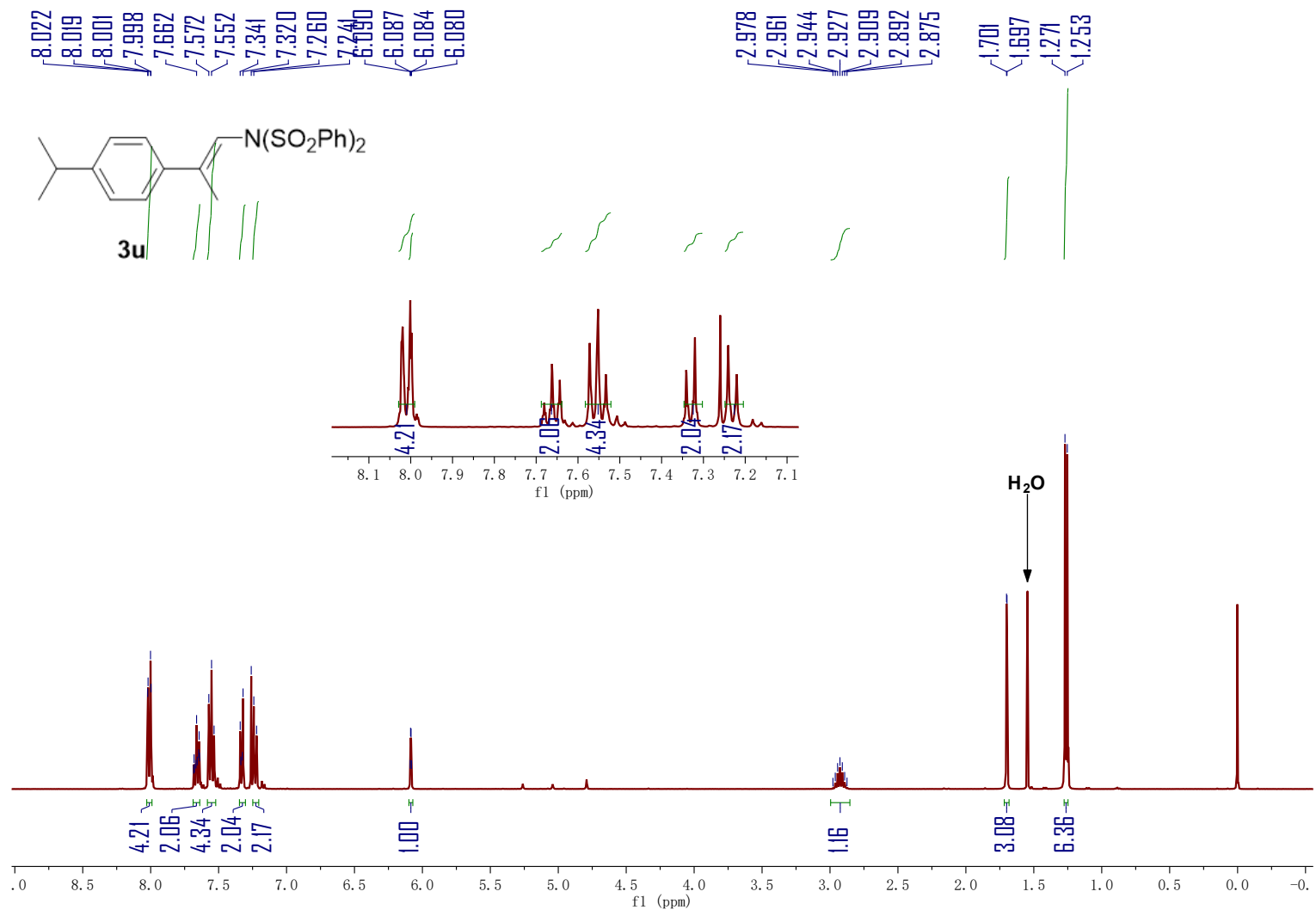
S153



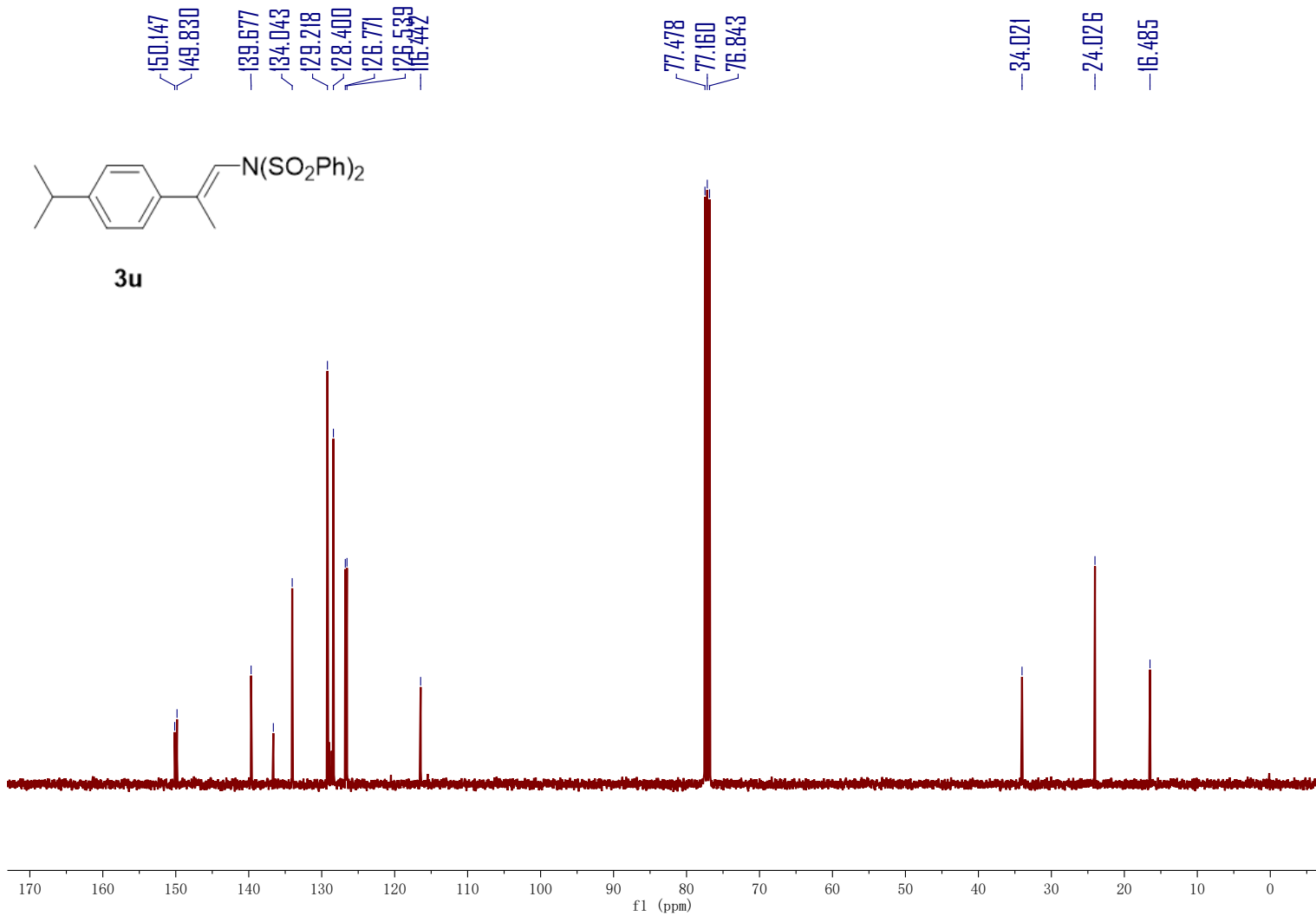
S154



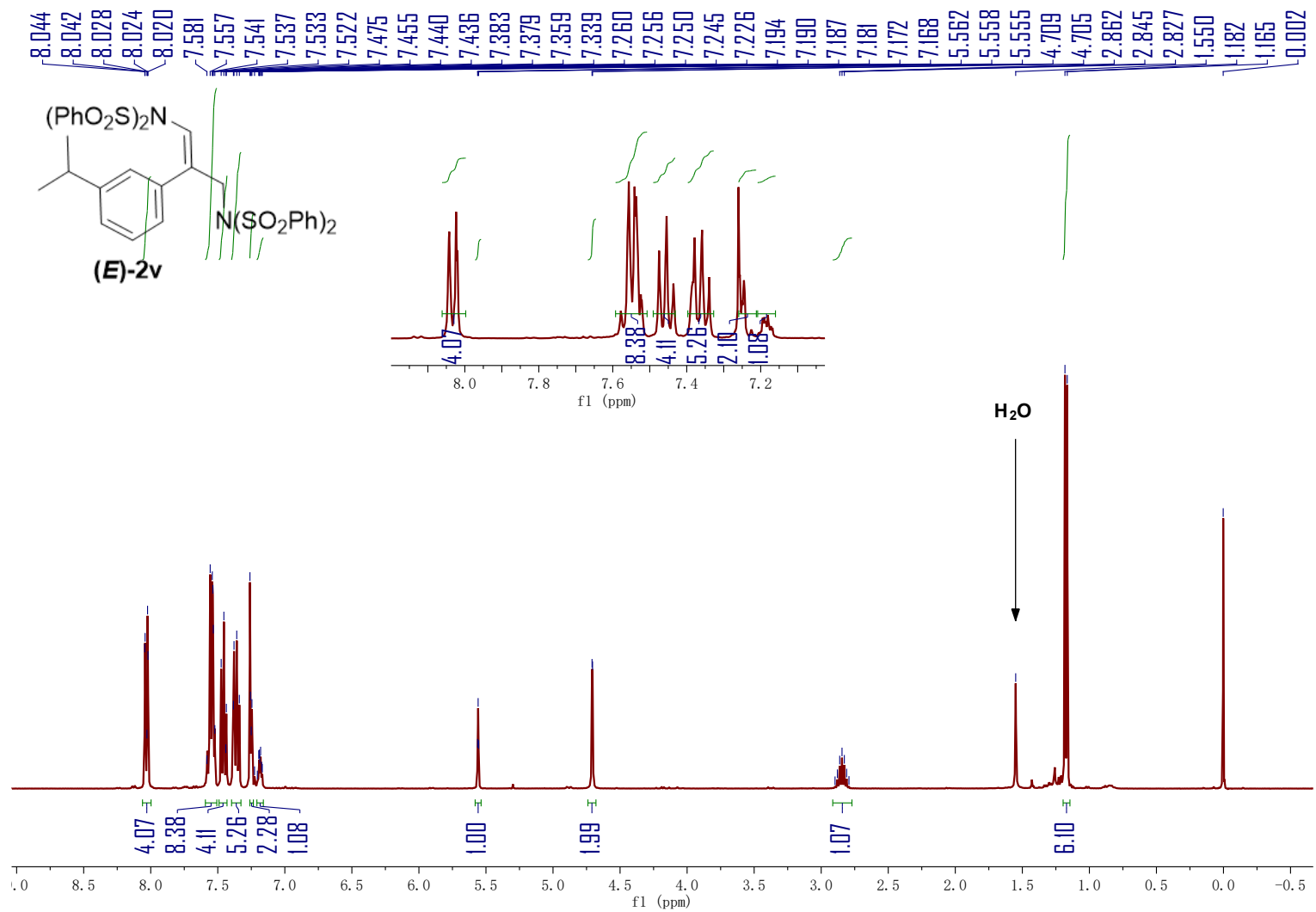
S155



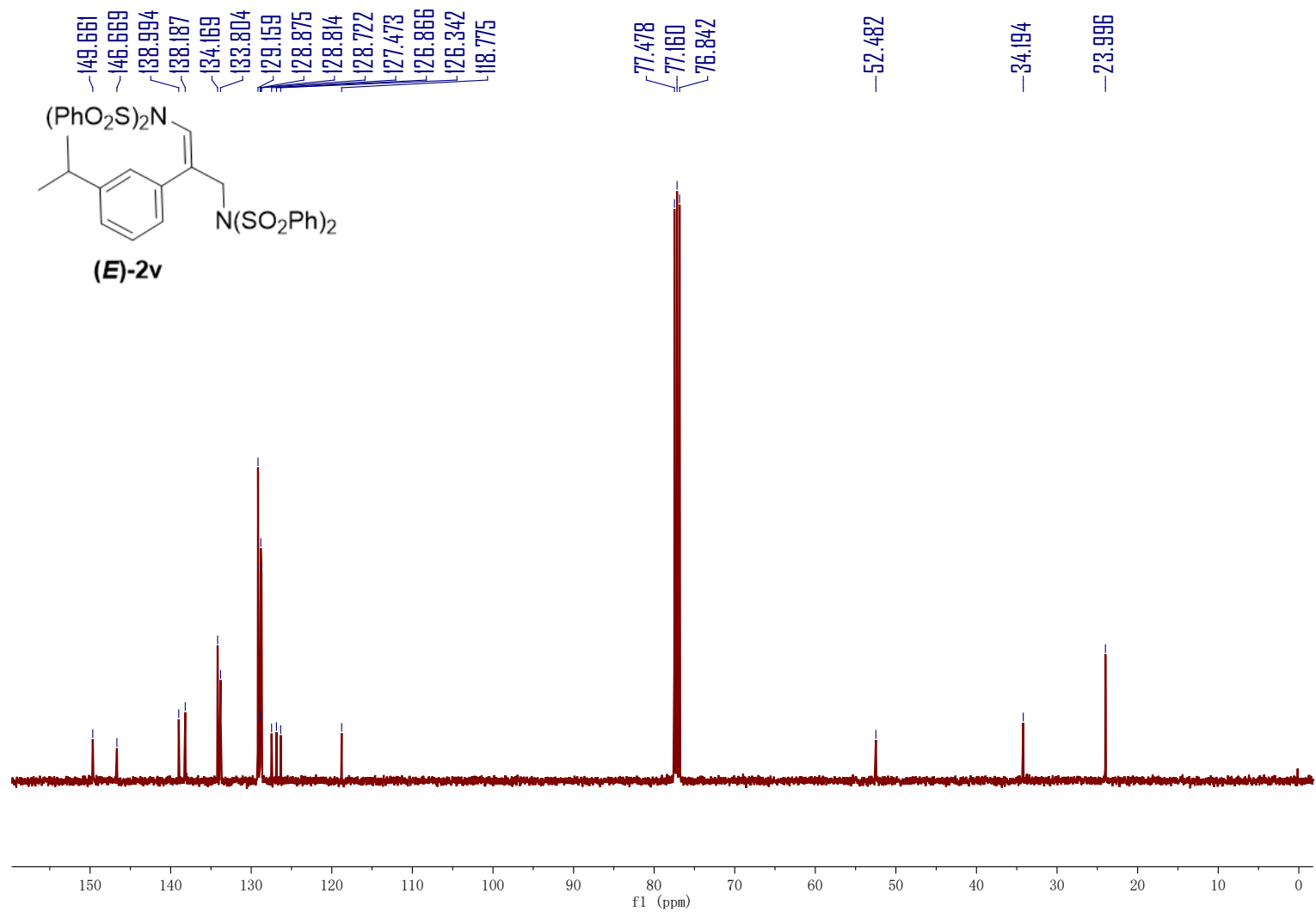
S156



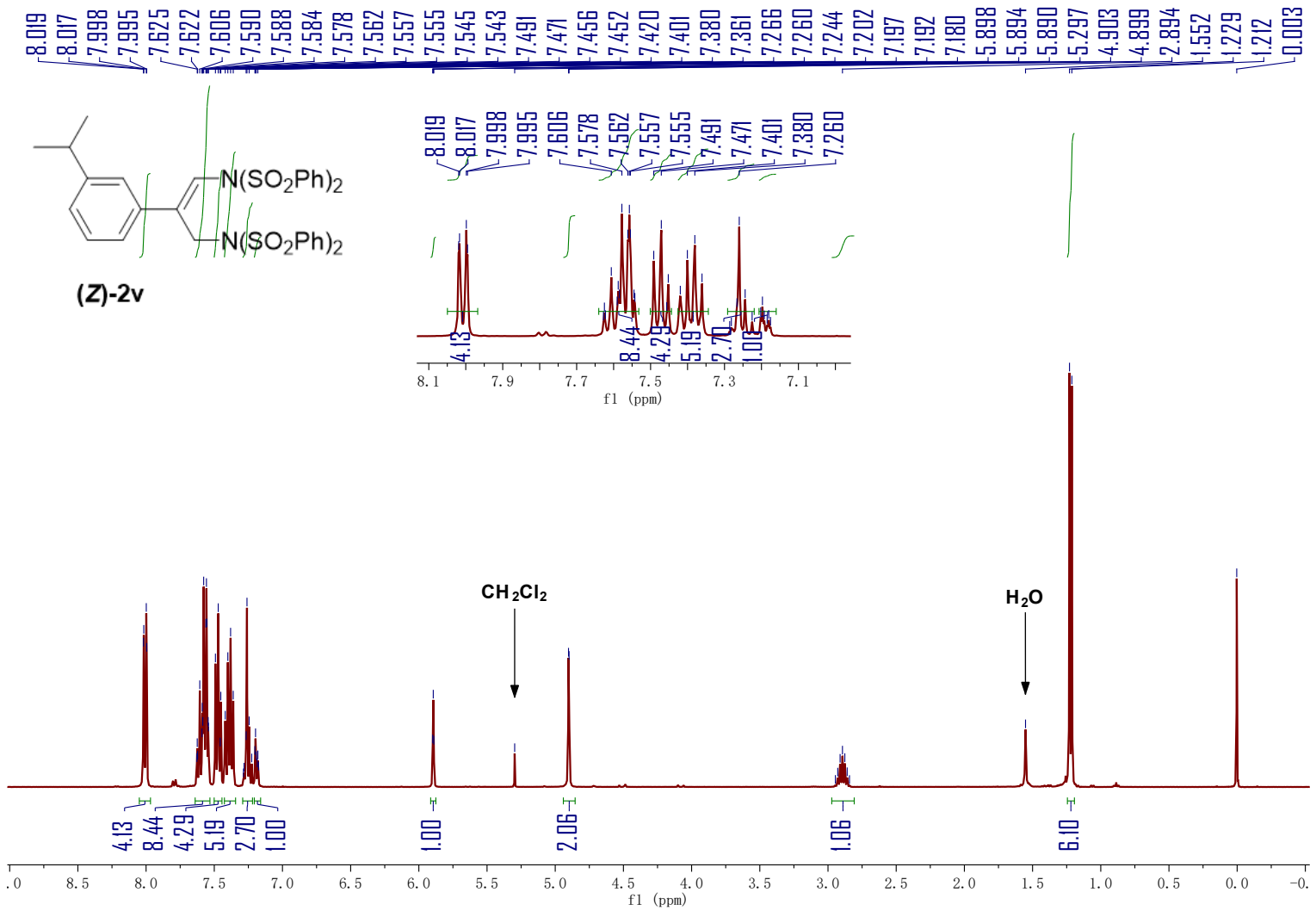
S157



S158

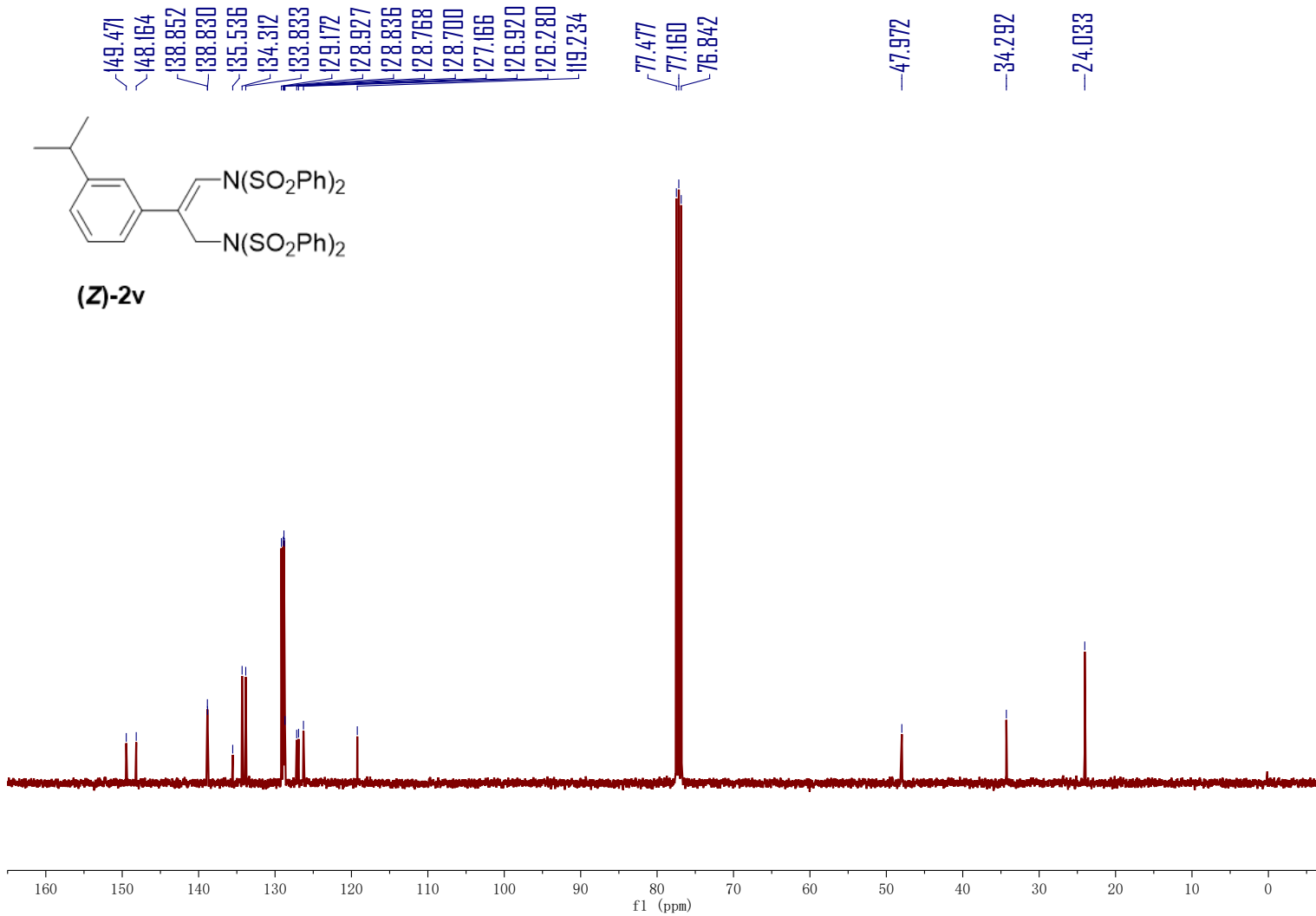


S159

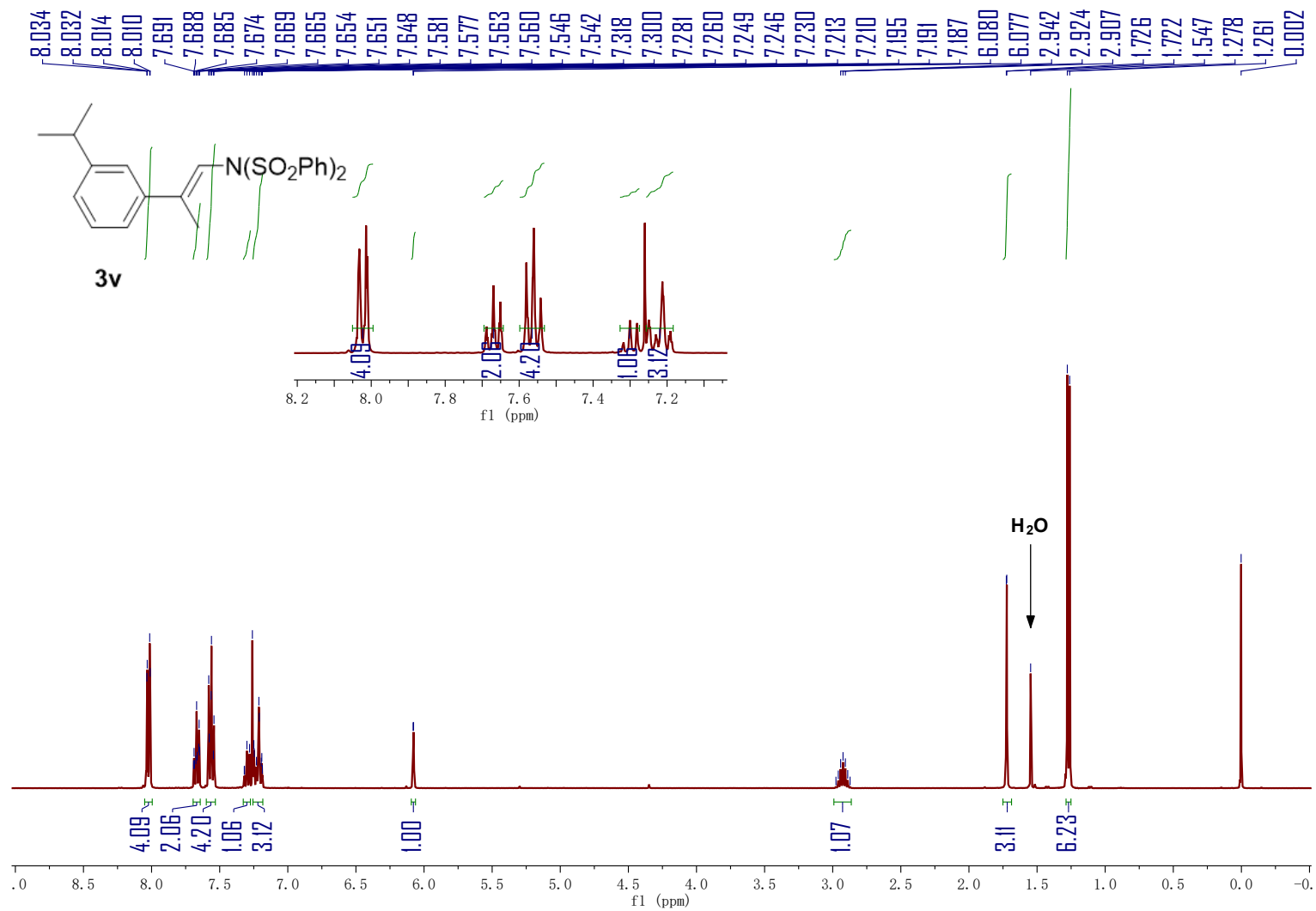


S160

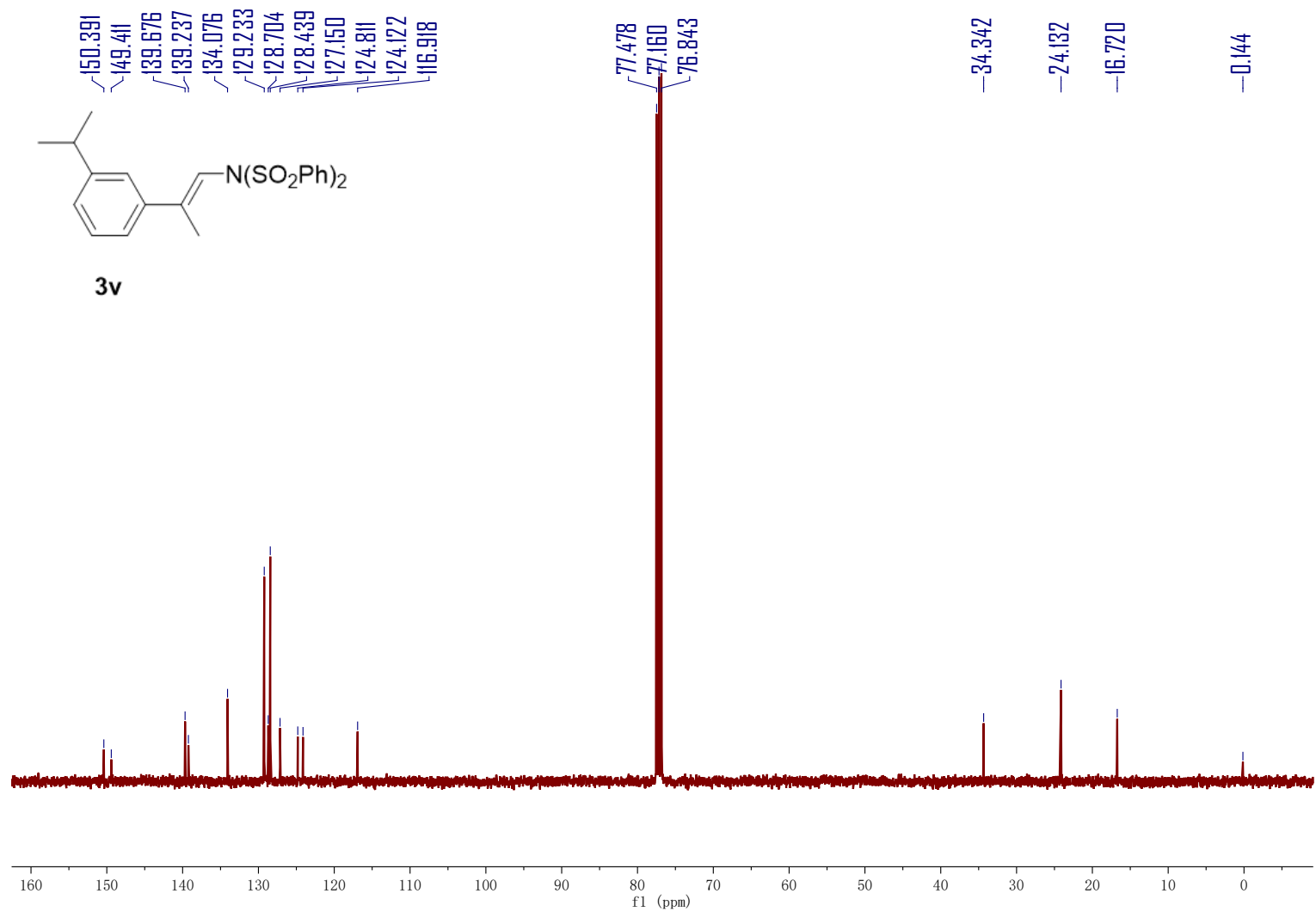




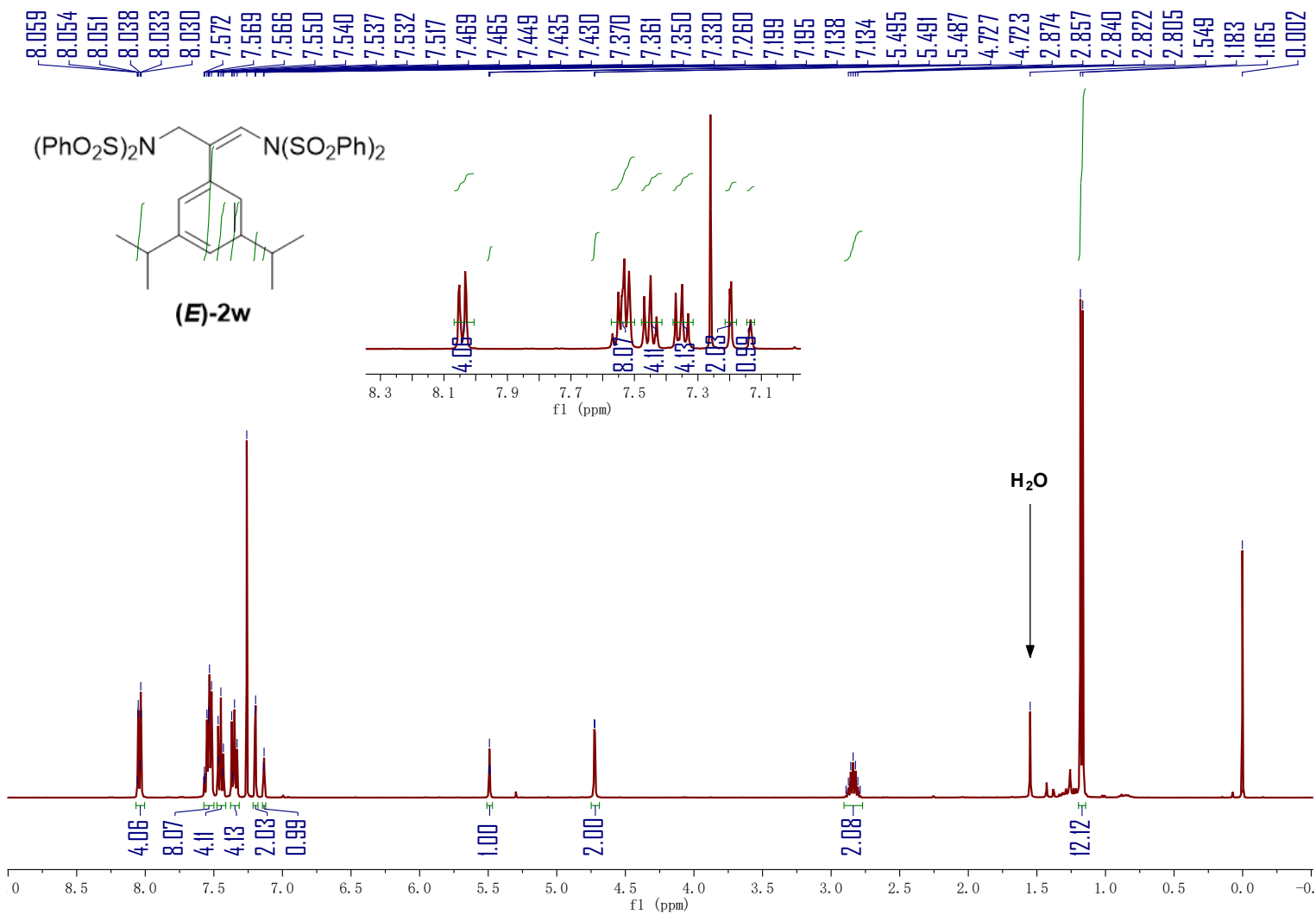
S161



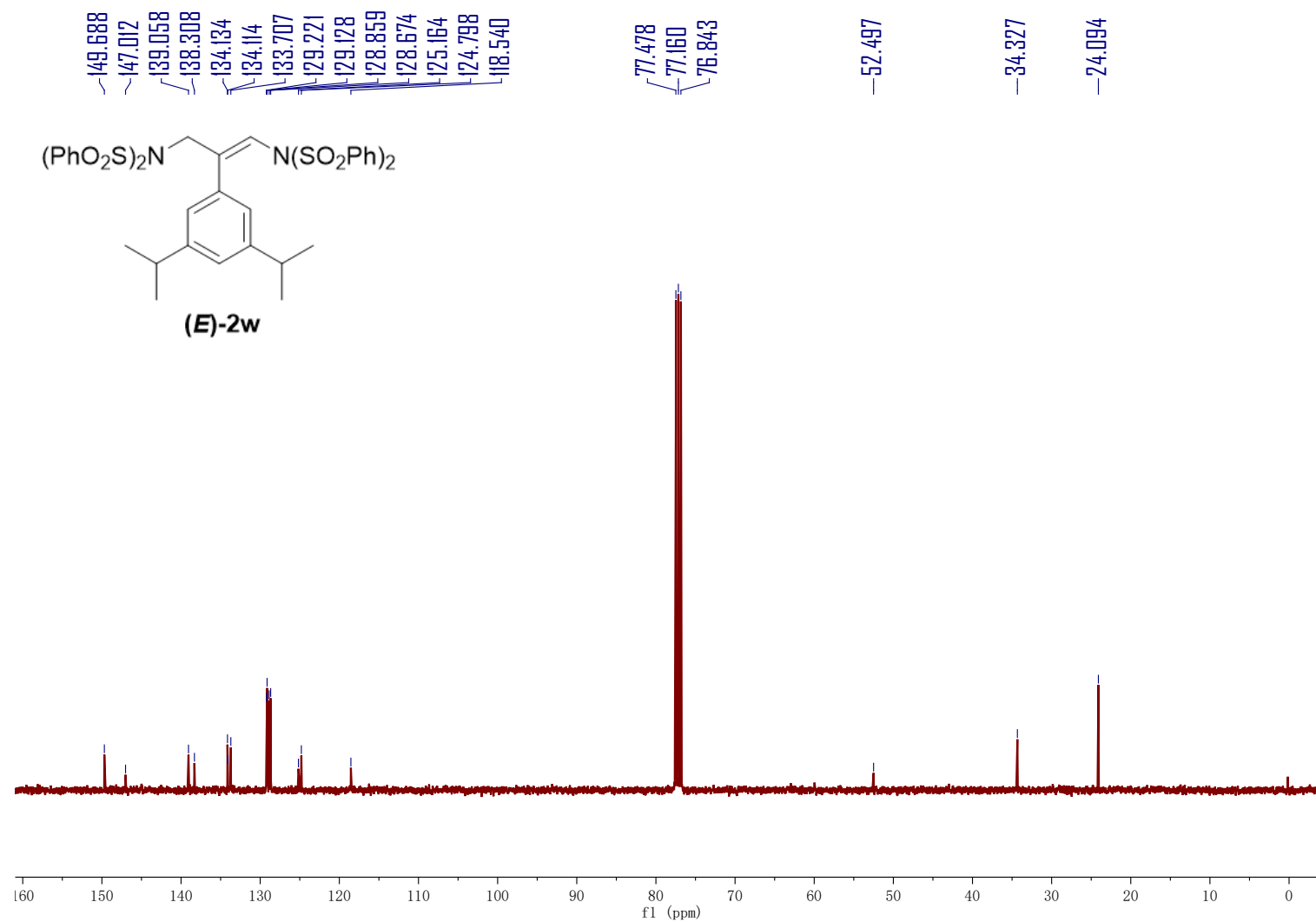
S162



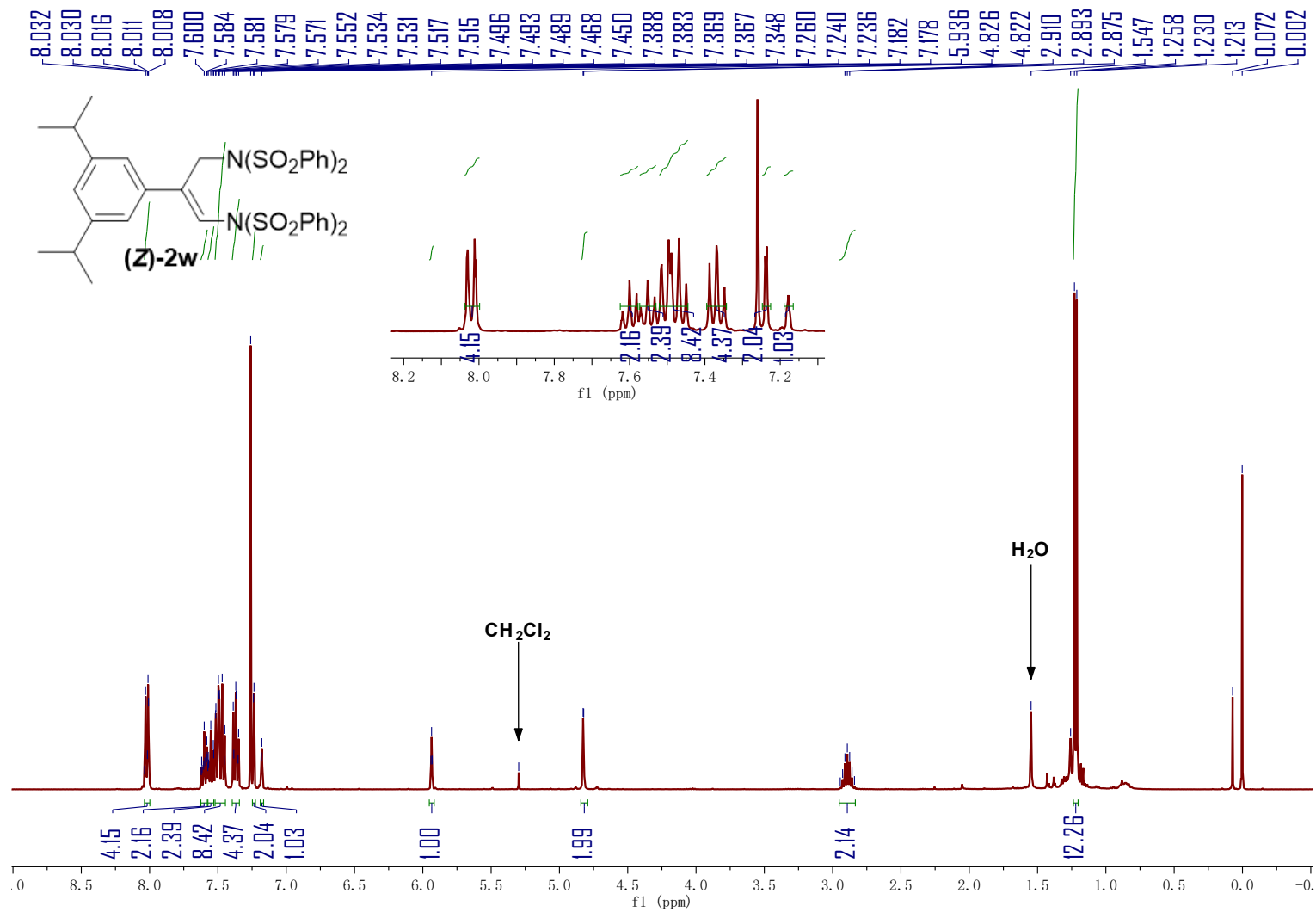
S163



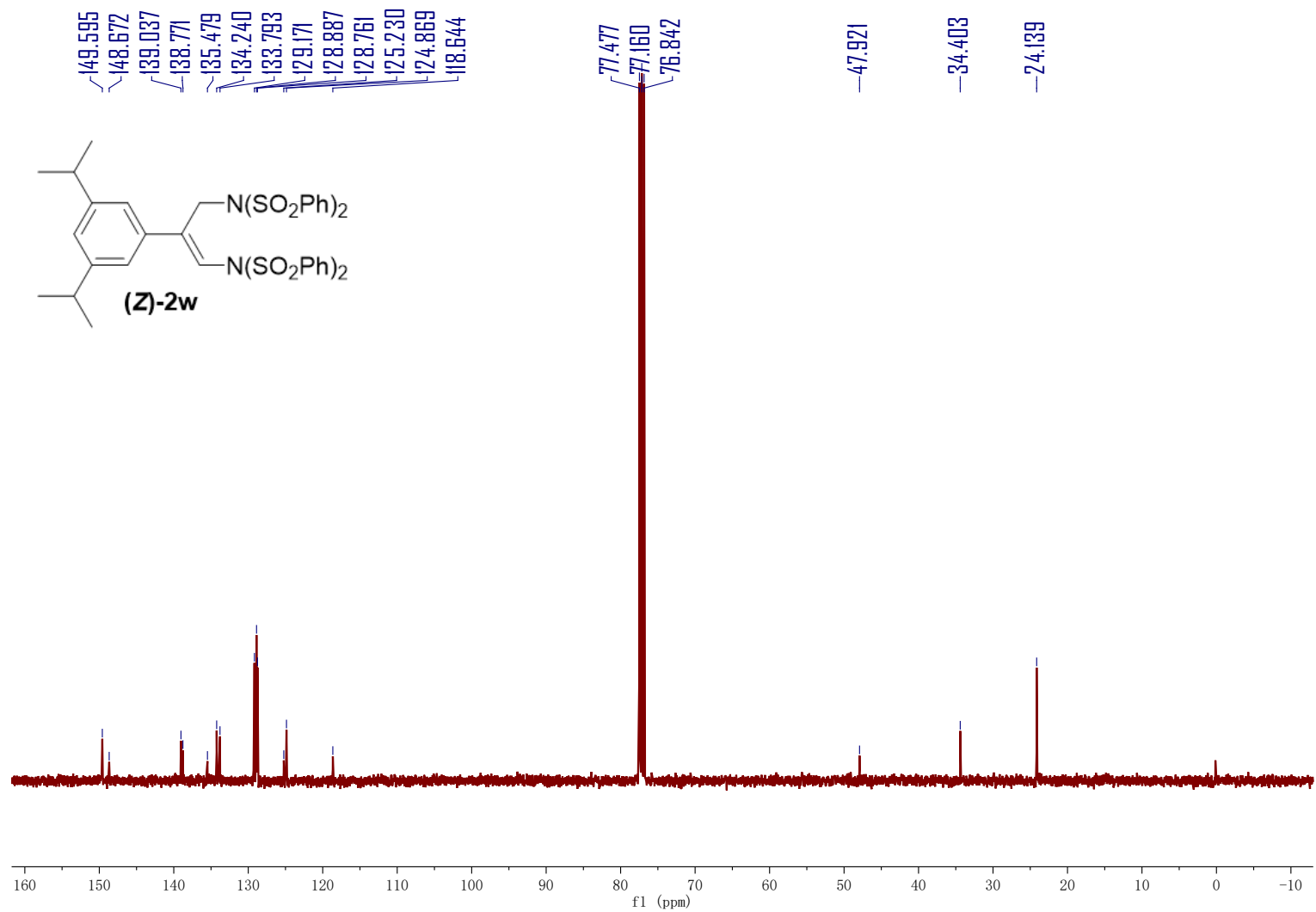
S164



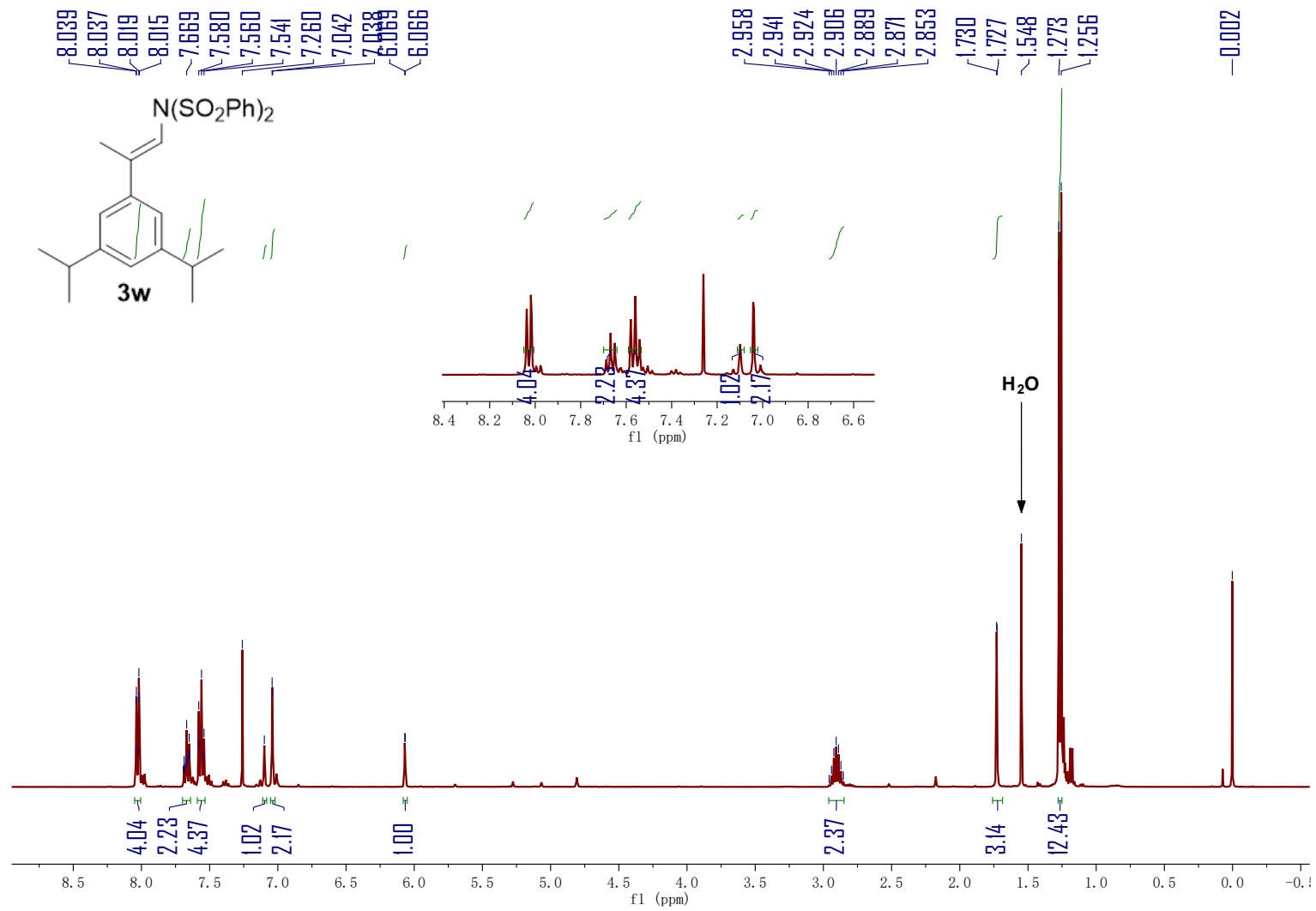
S165



S166

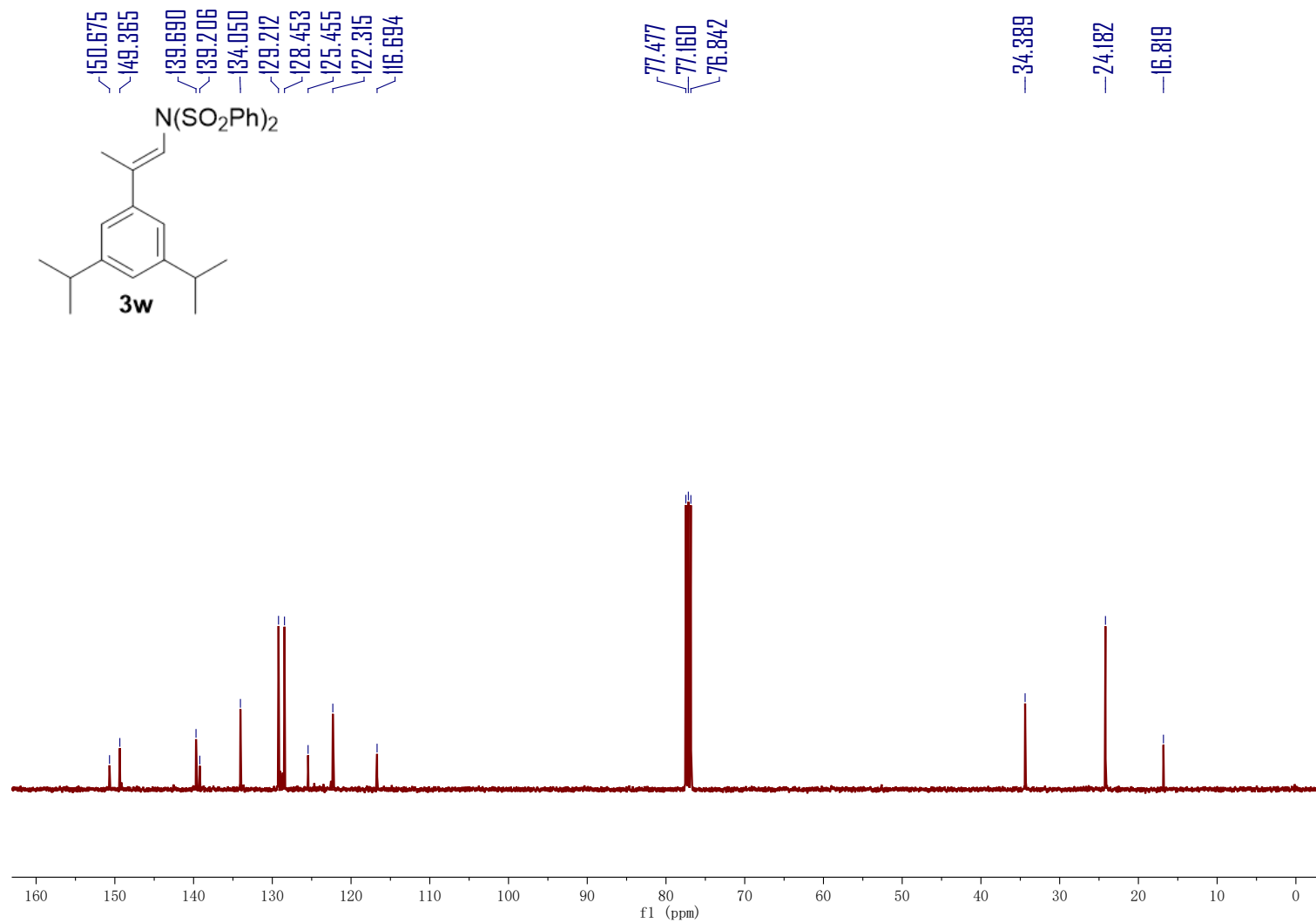


S167

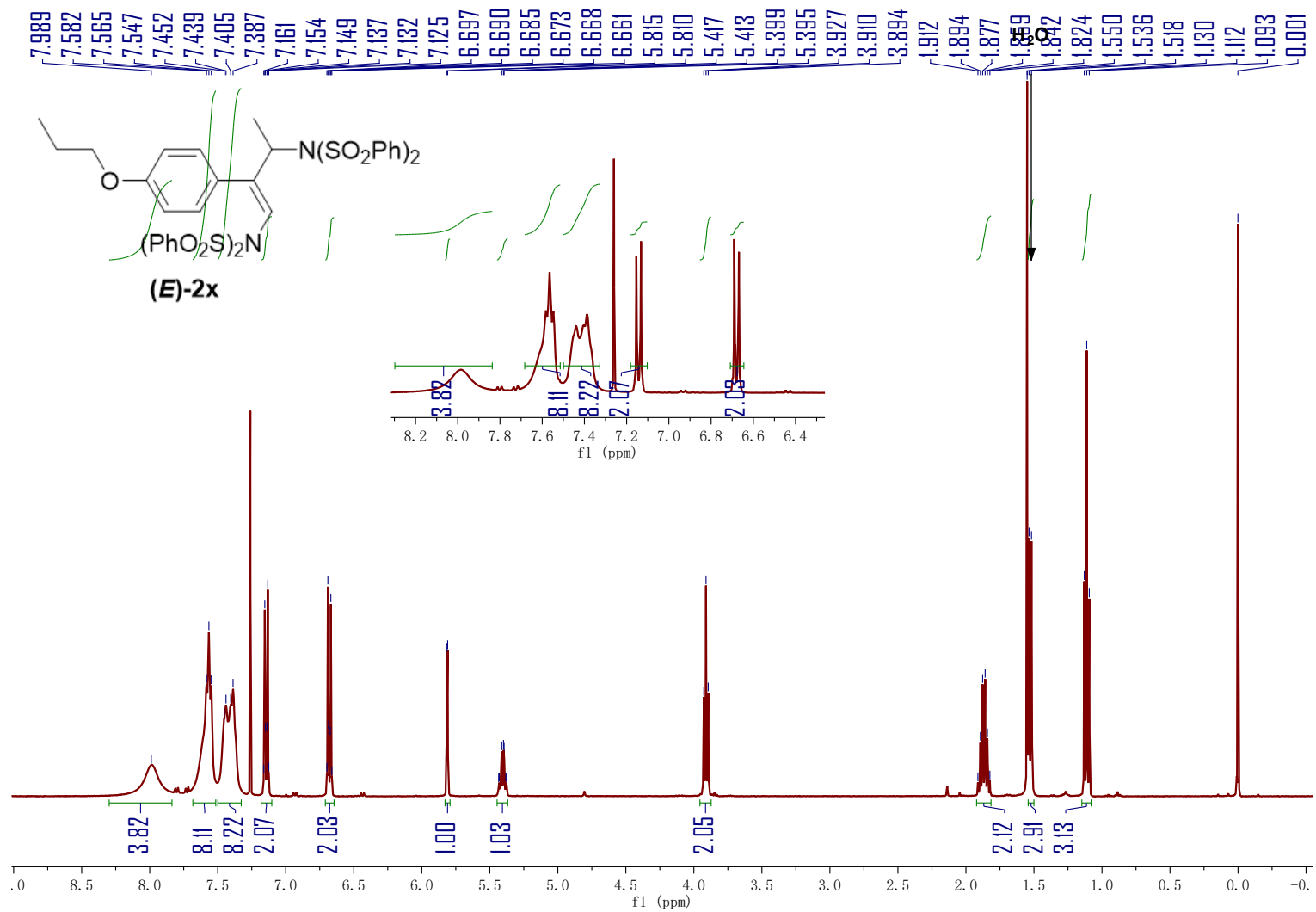


S168

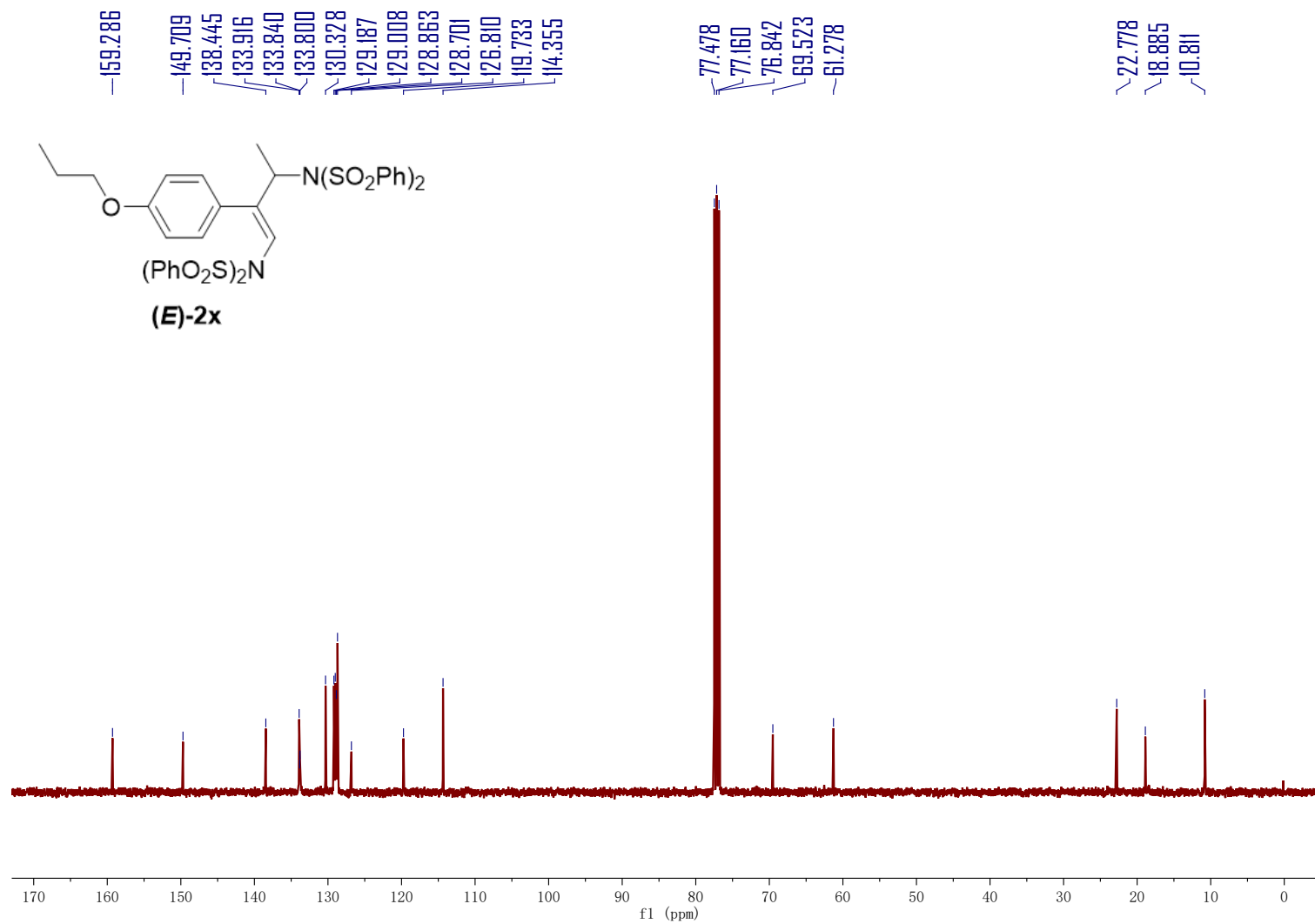




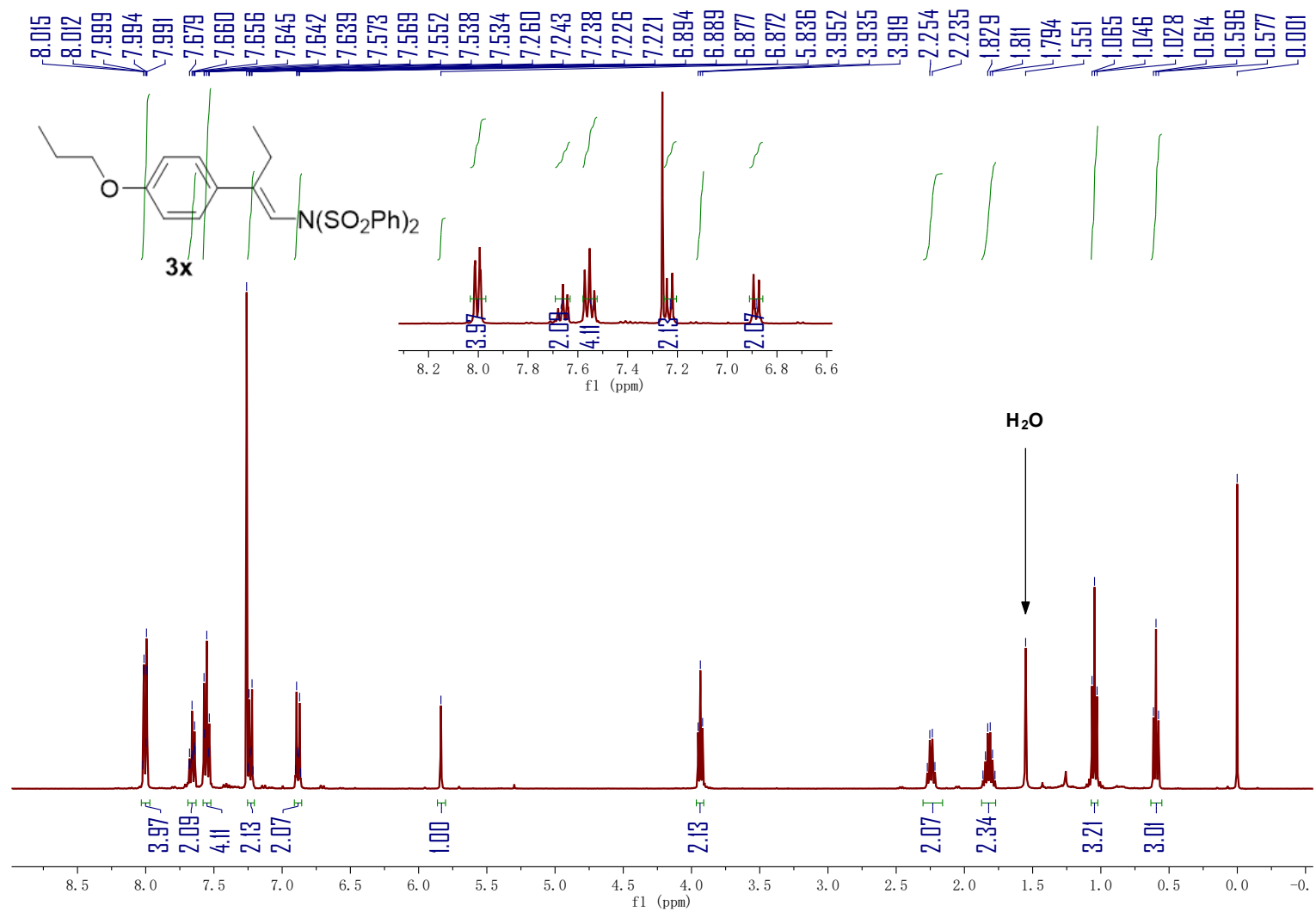
S169



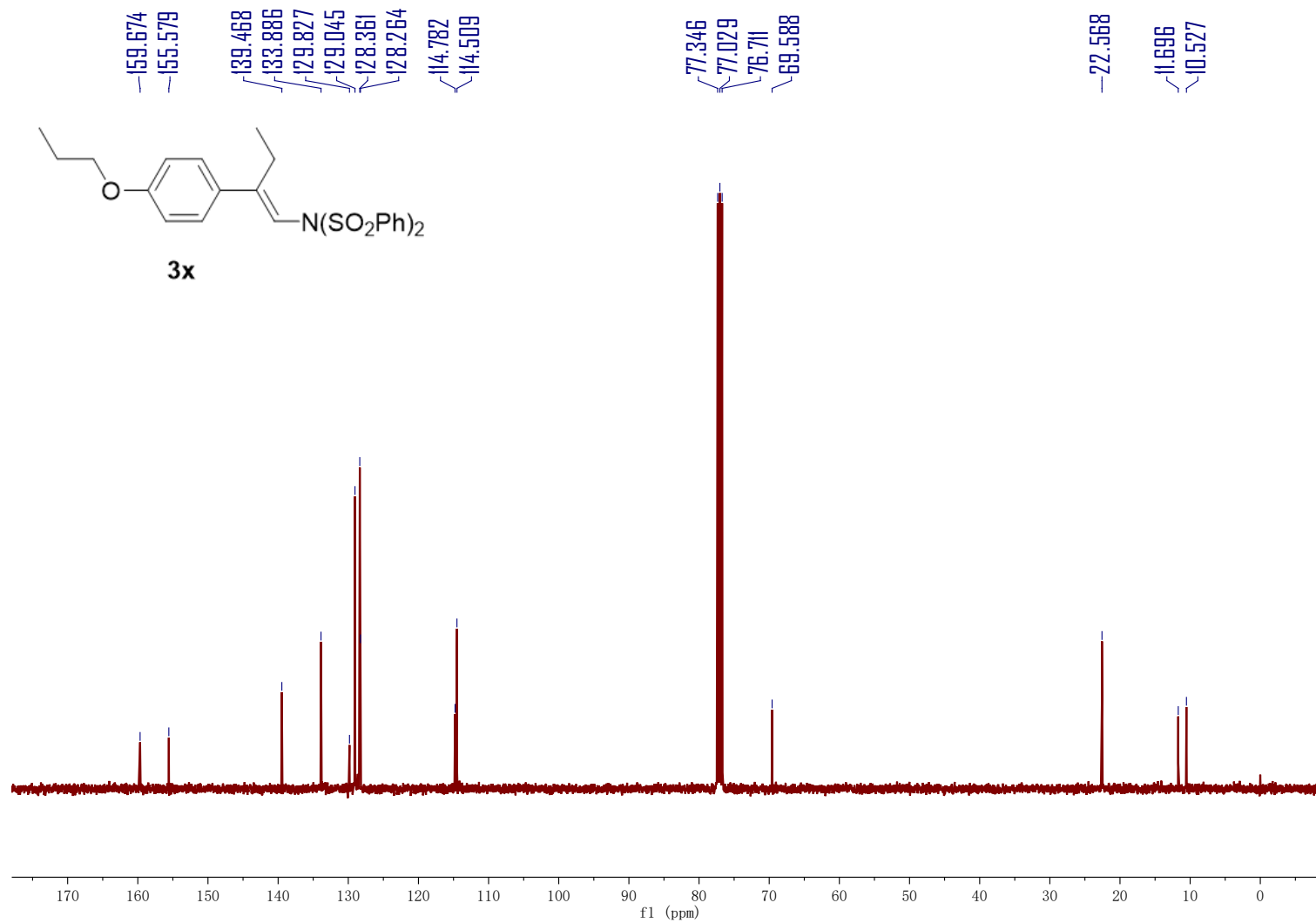
S170



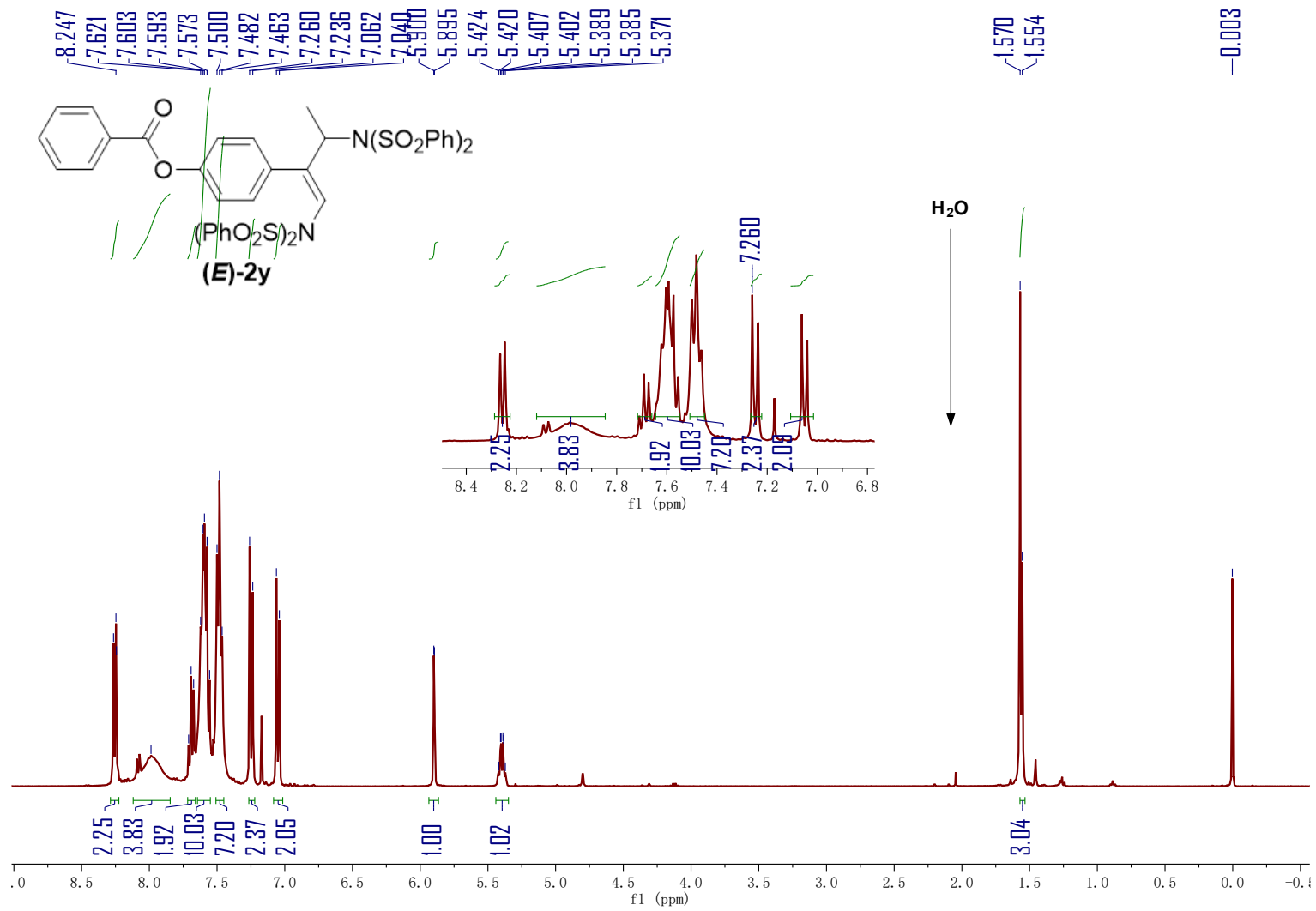
S171



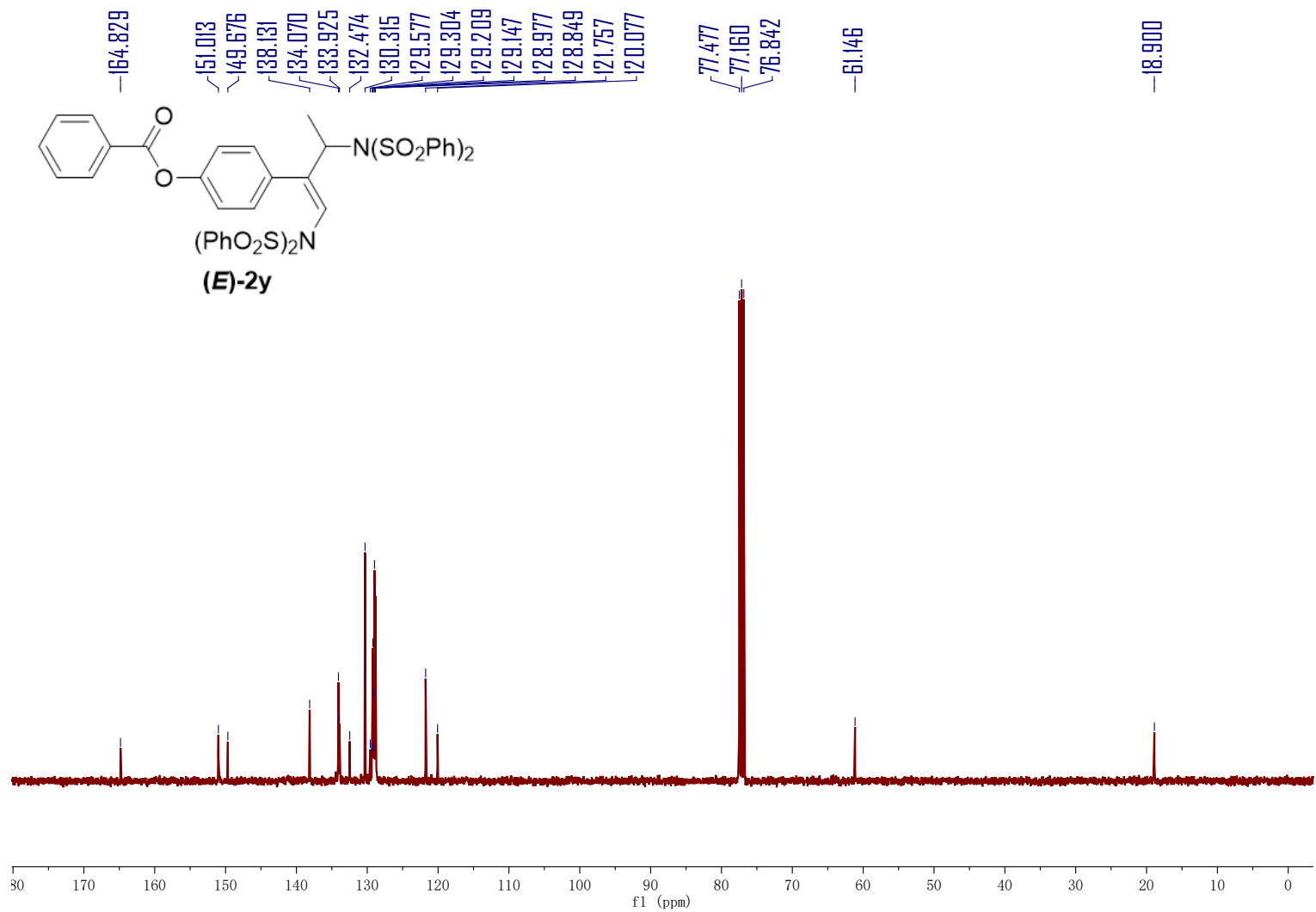
S172



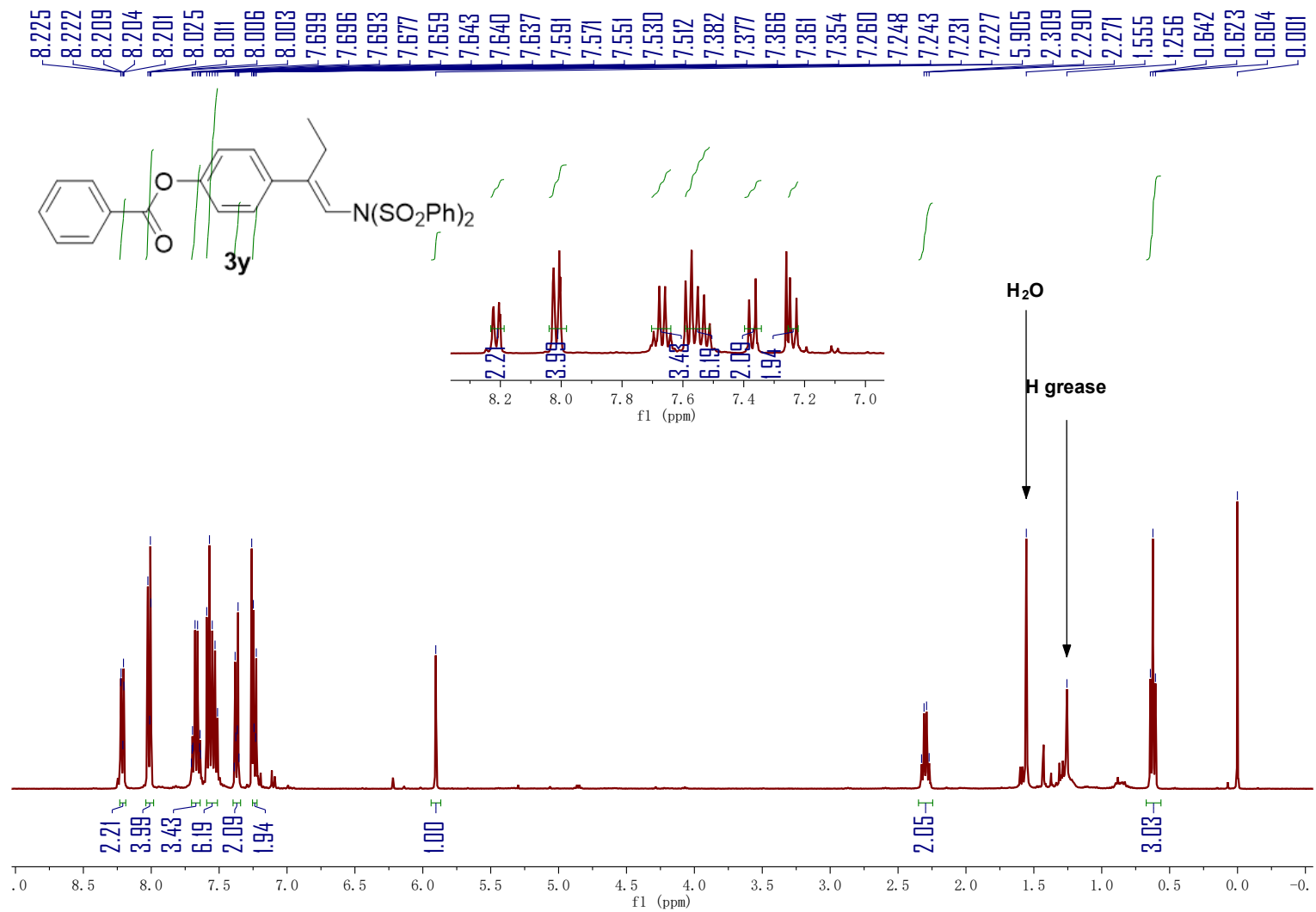
S173



S174

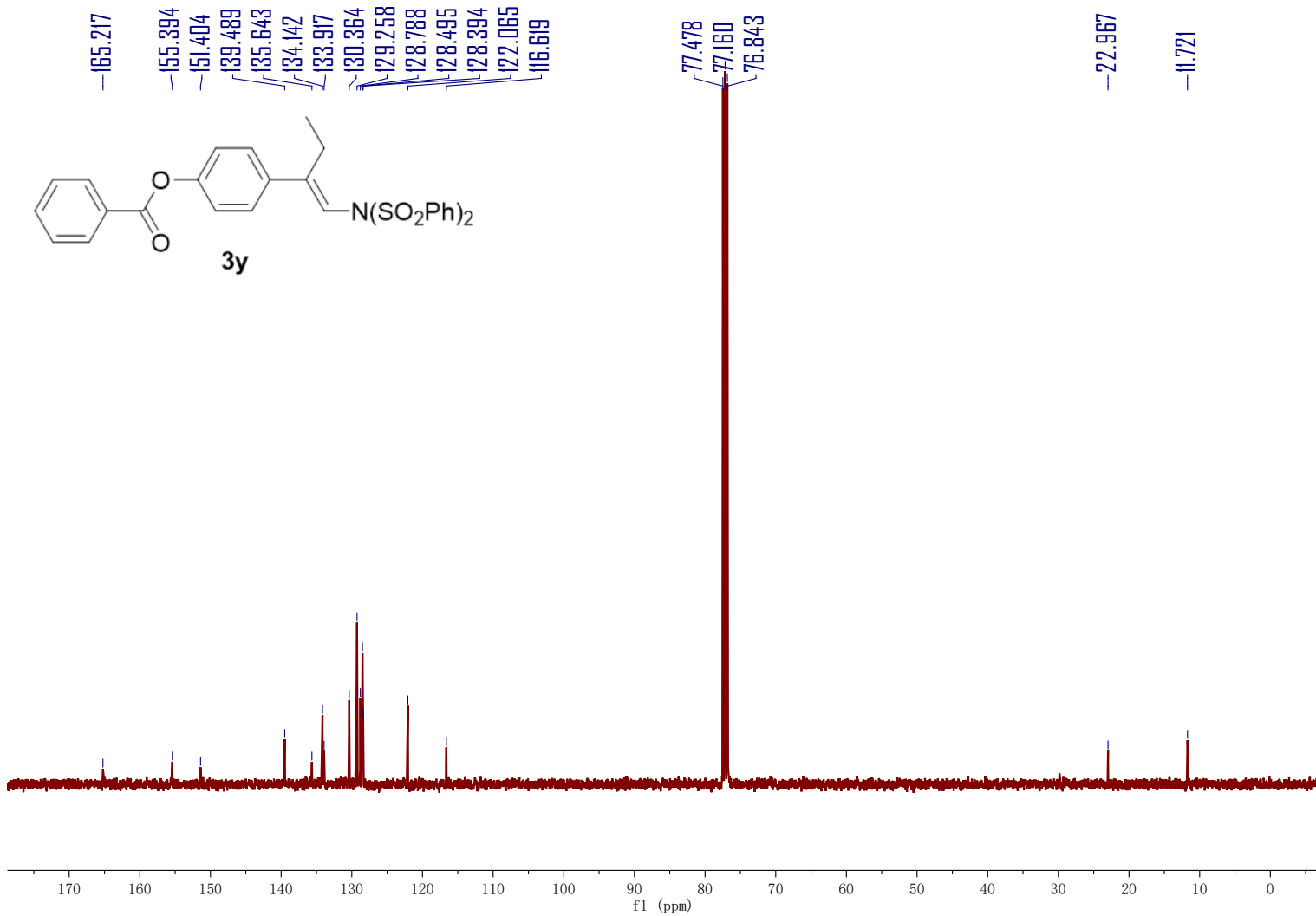


S175

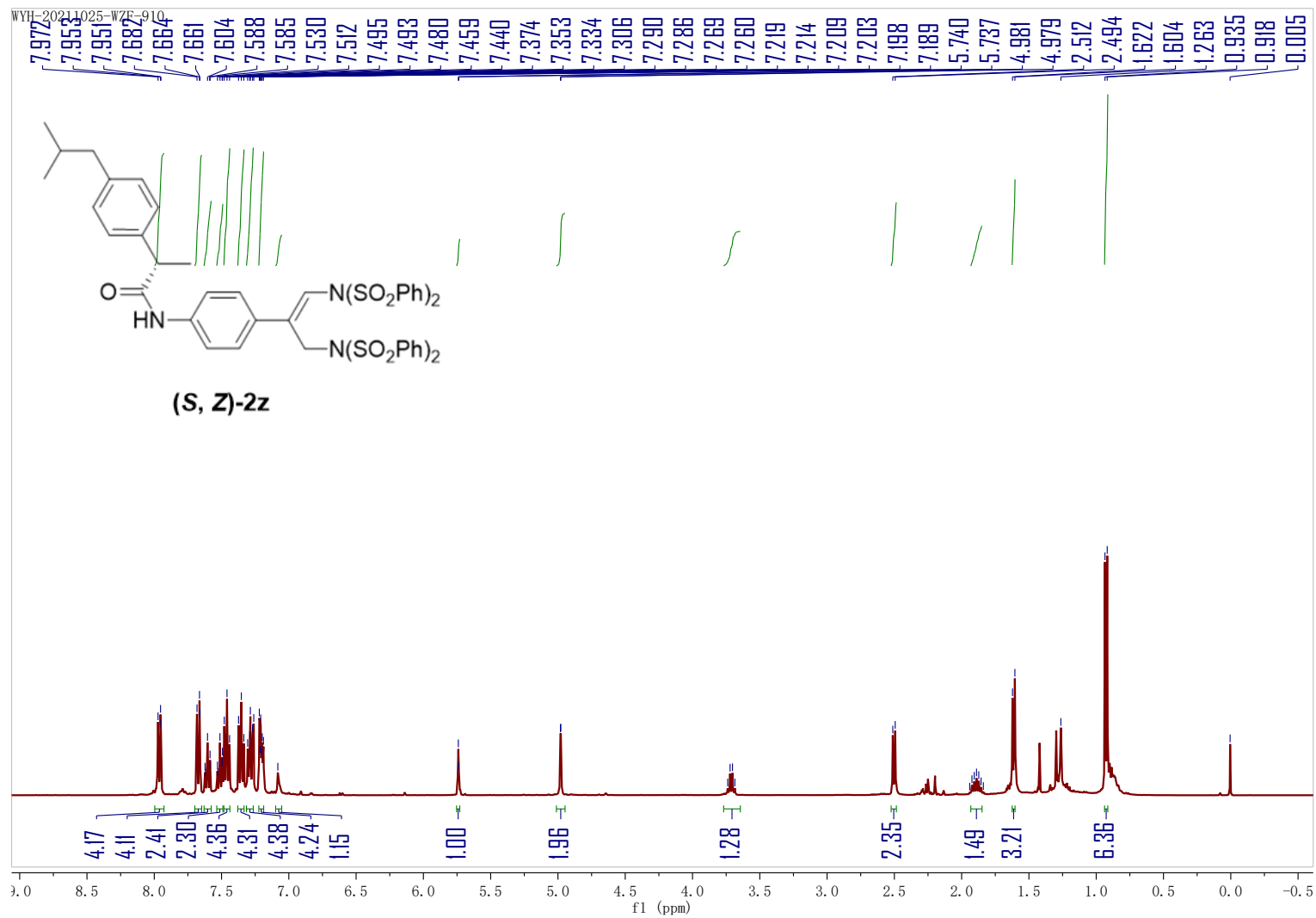


S176

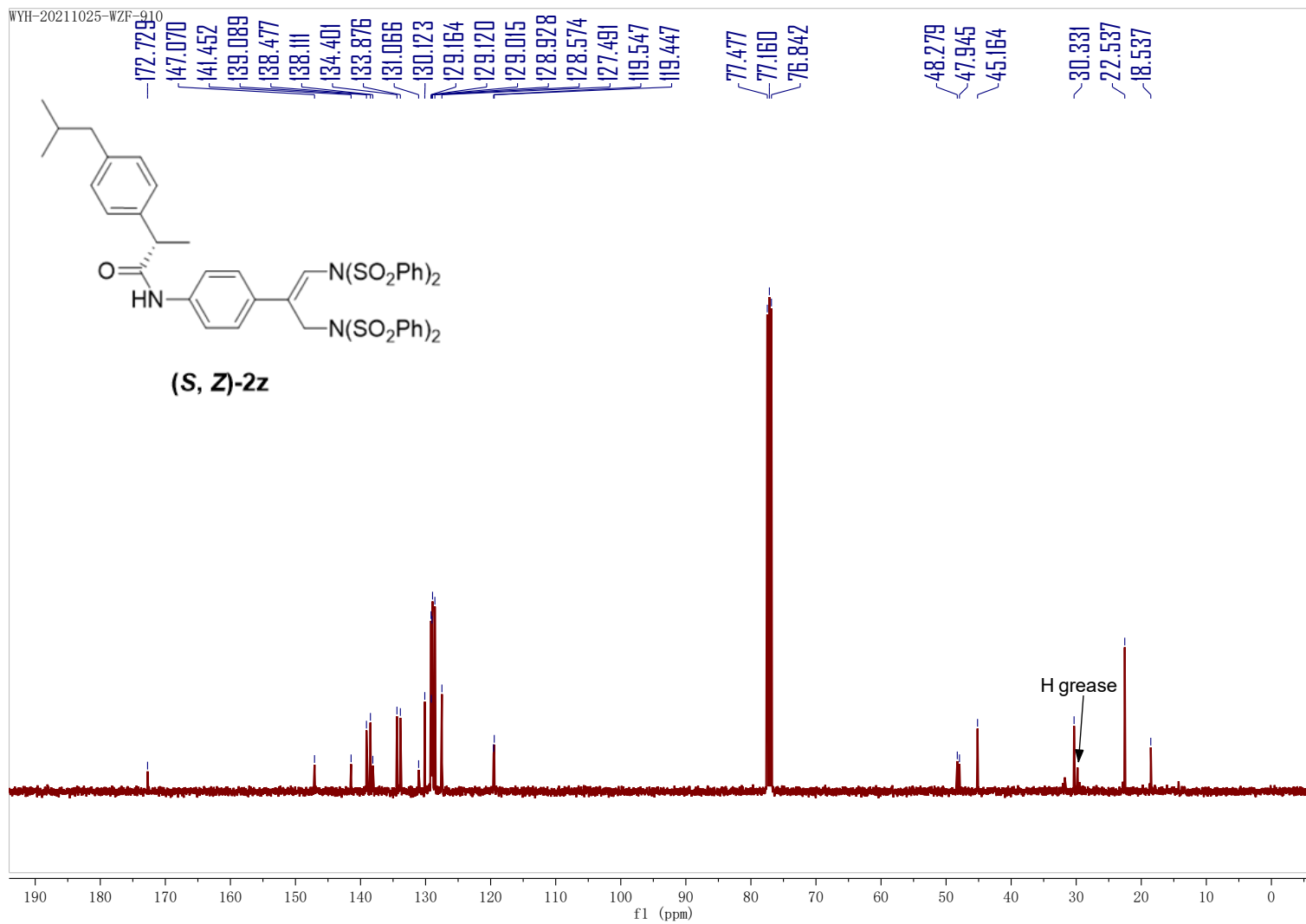




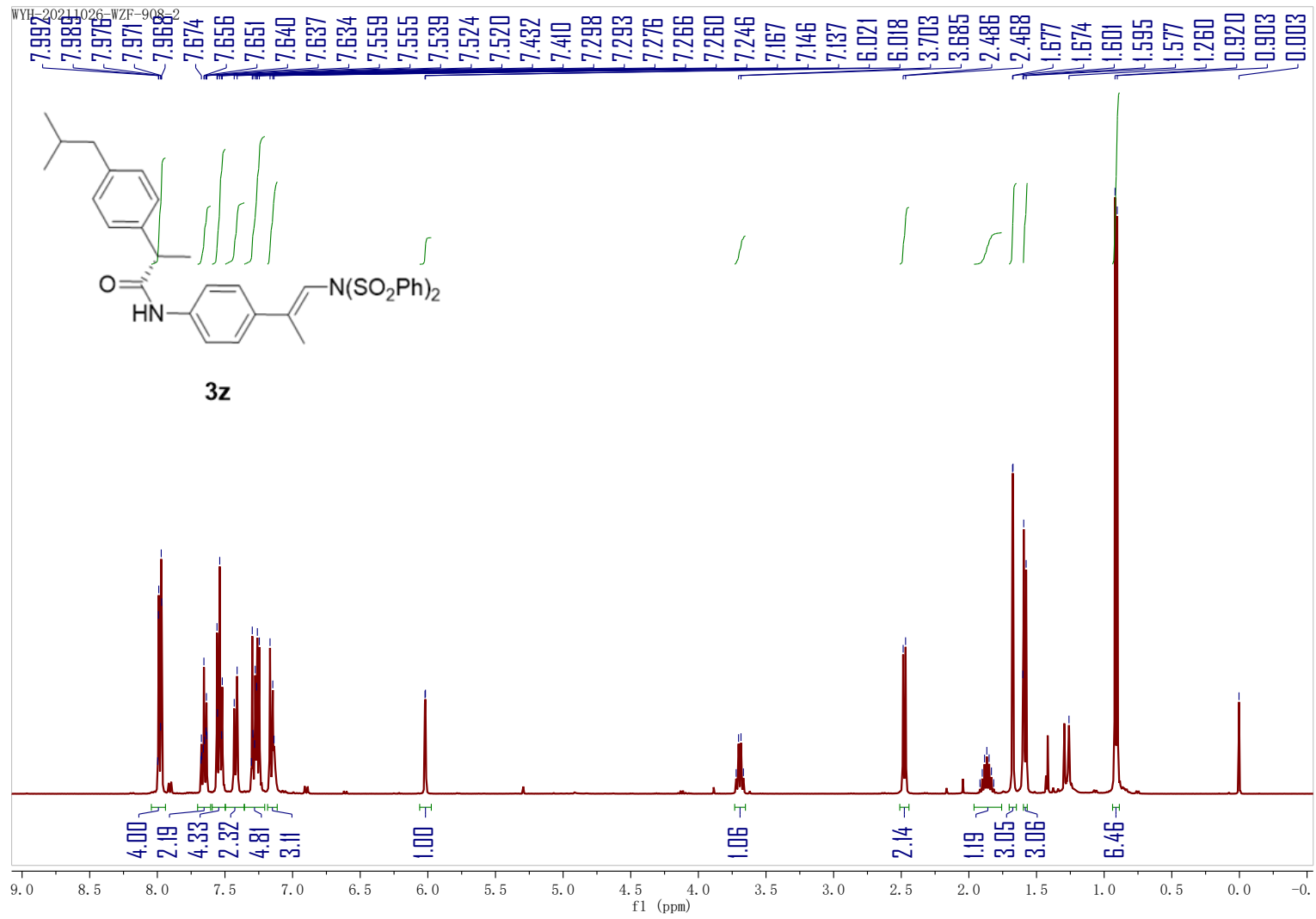
S177



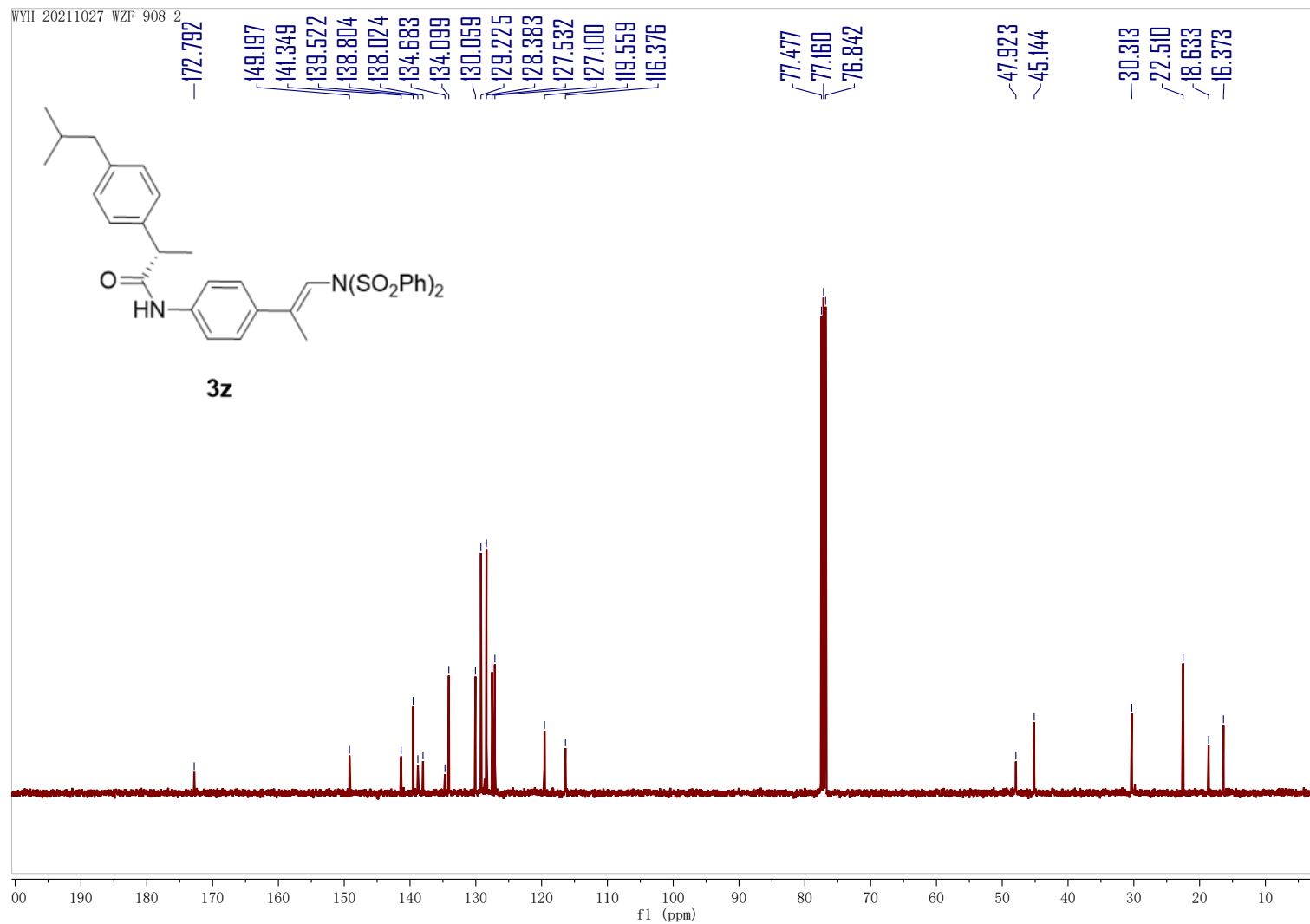
S178



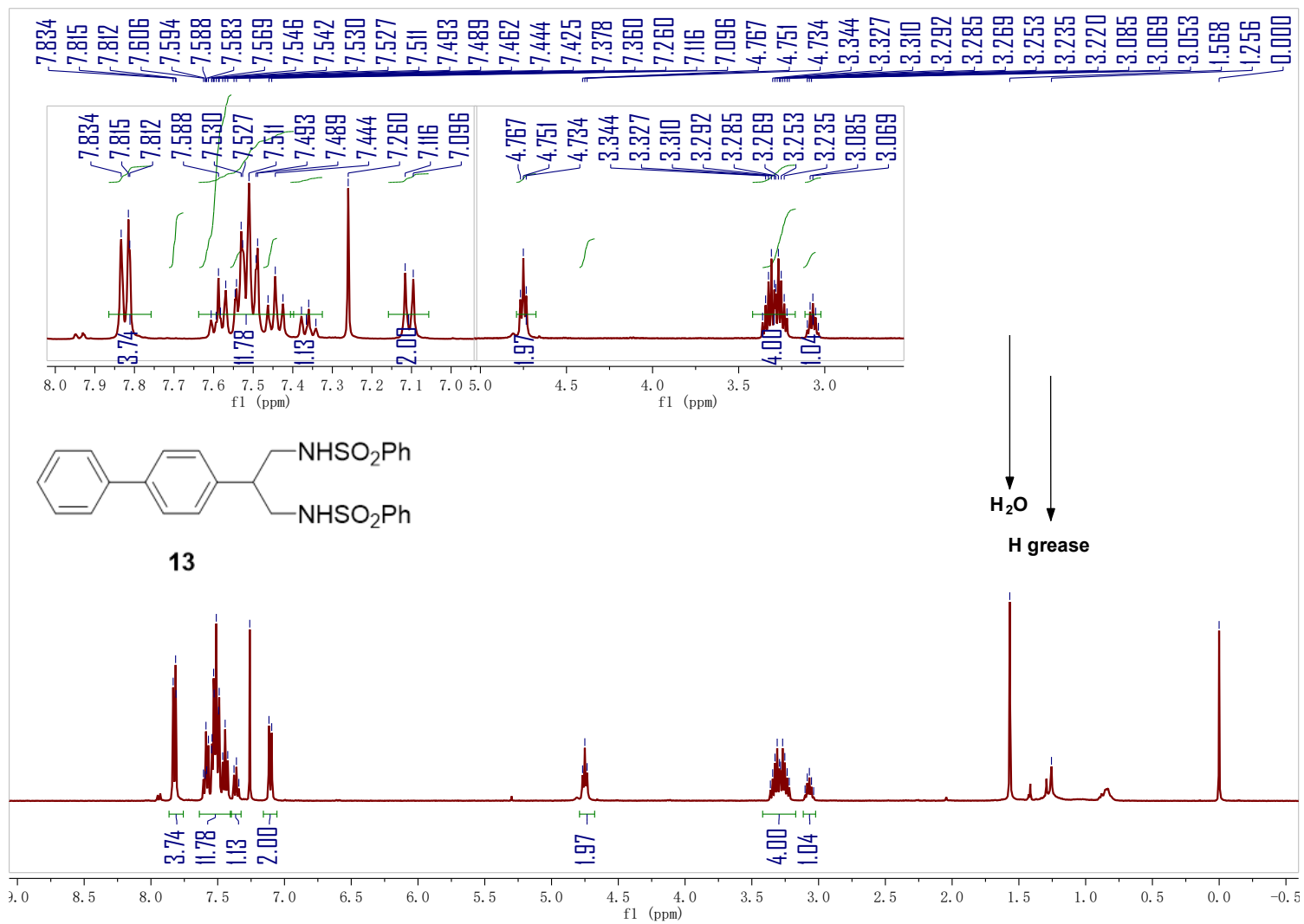
S179



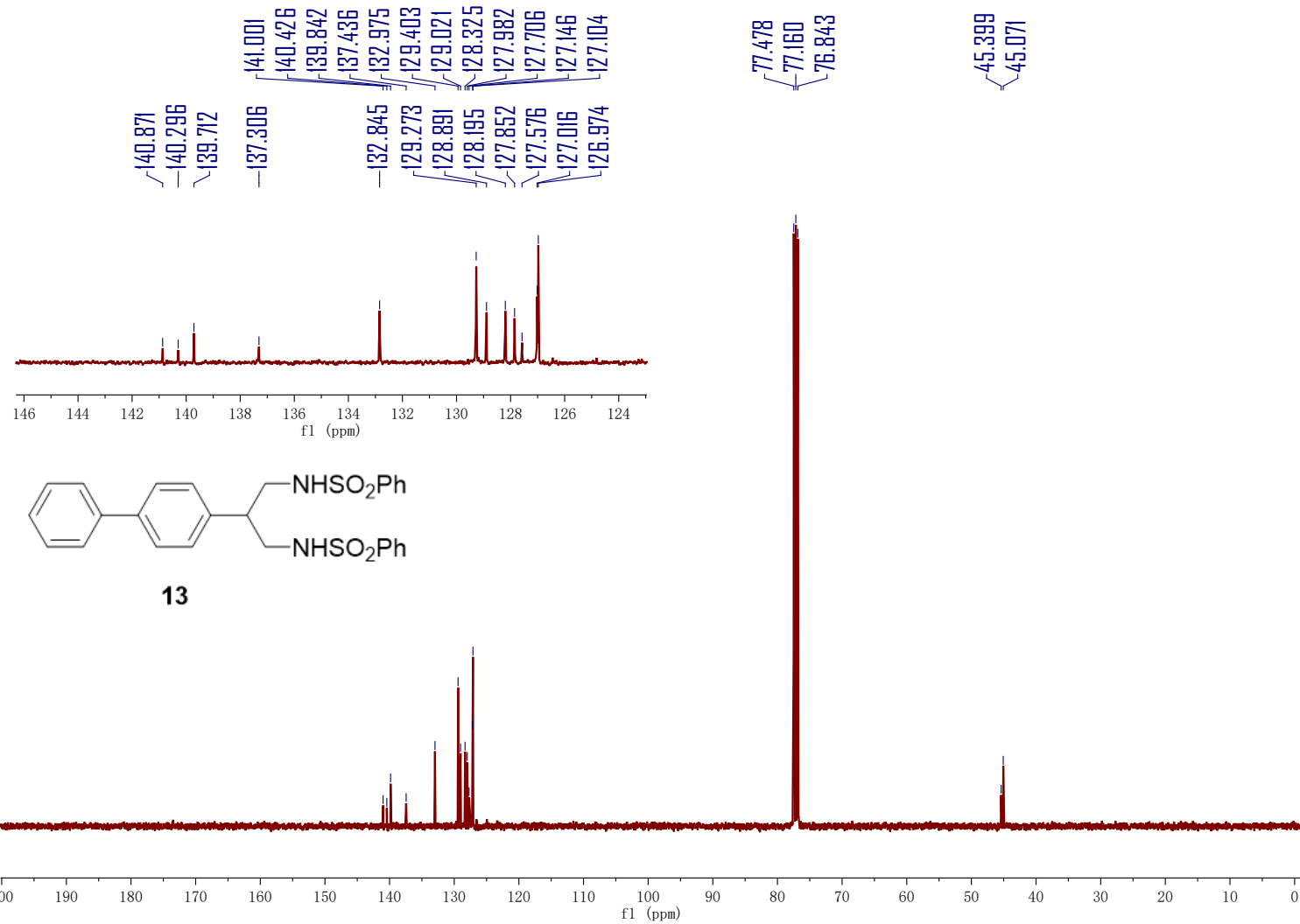
S180



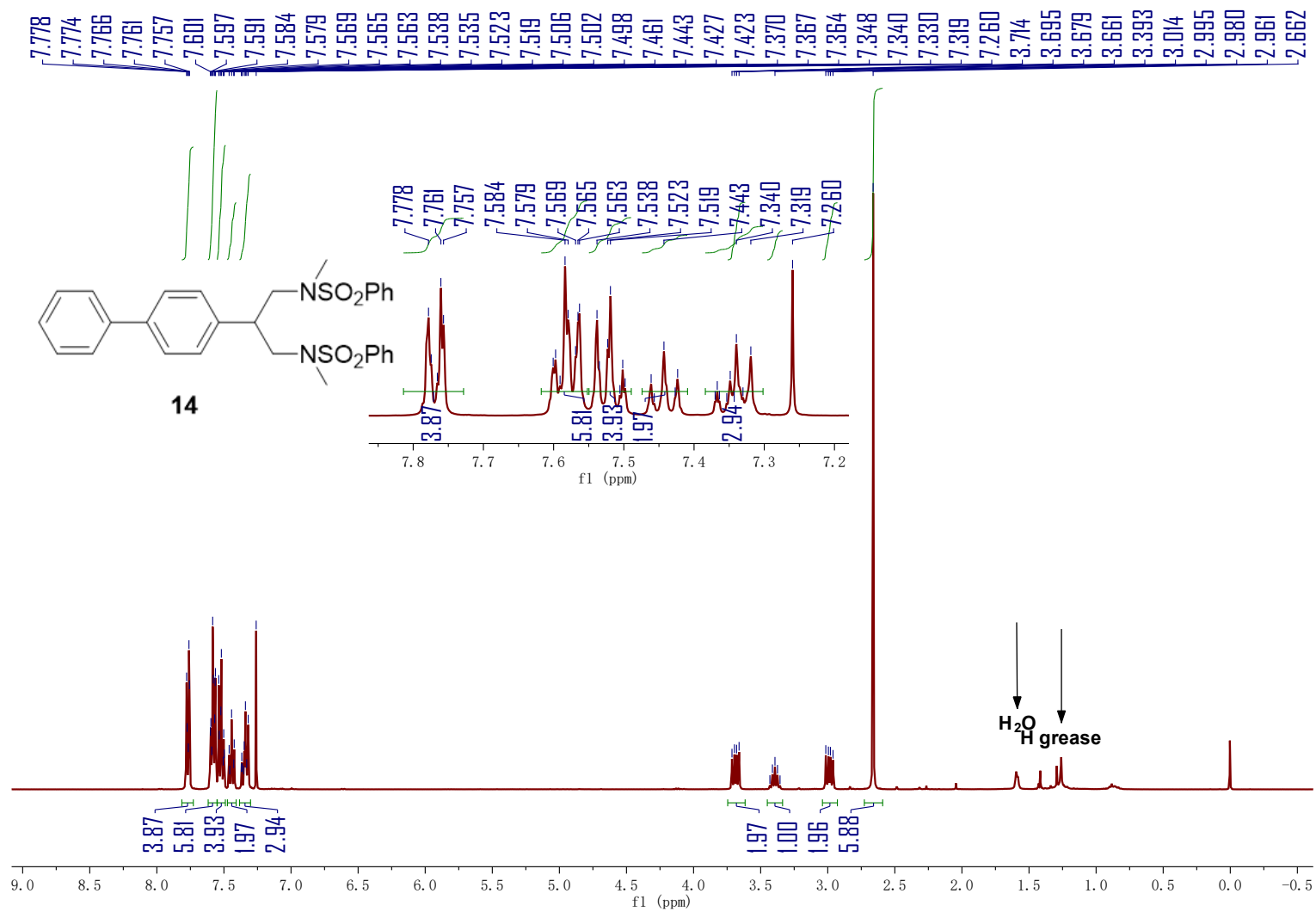
S181



S182

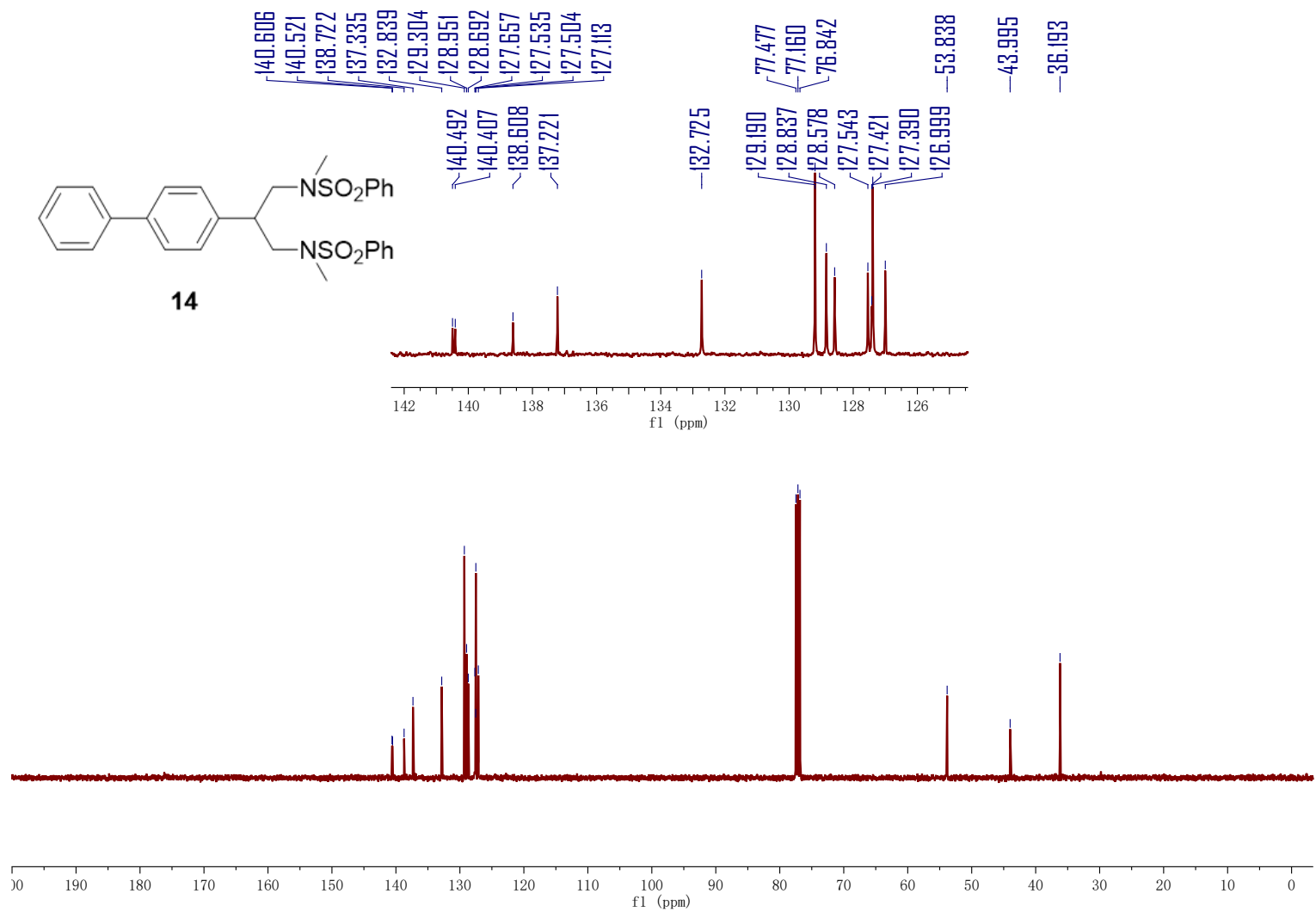


S183

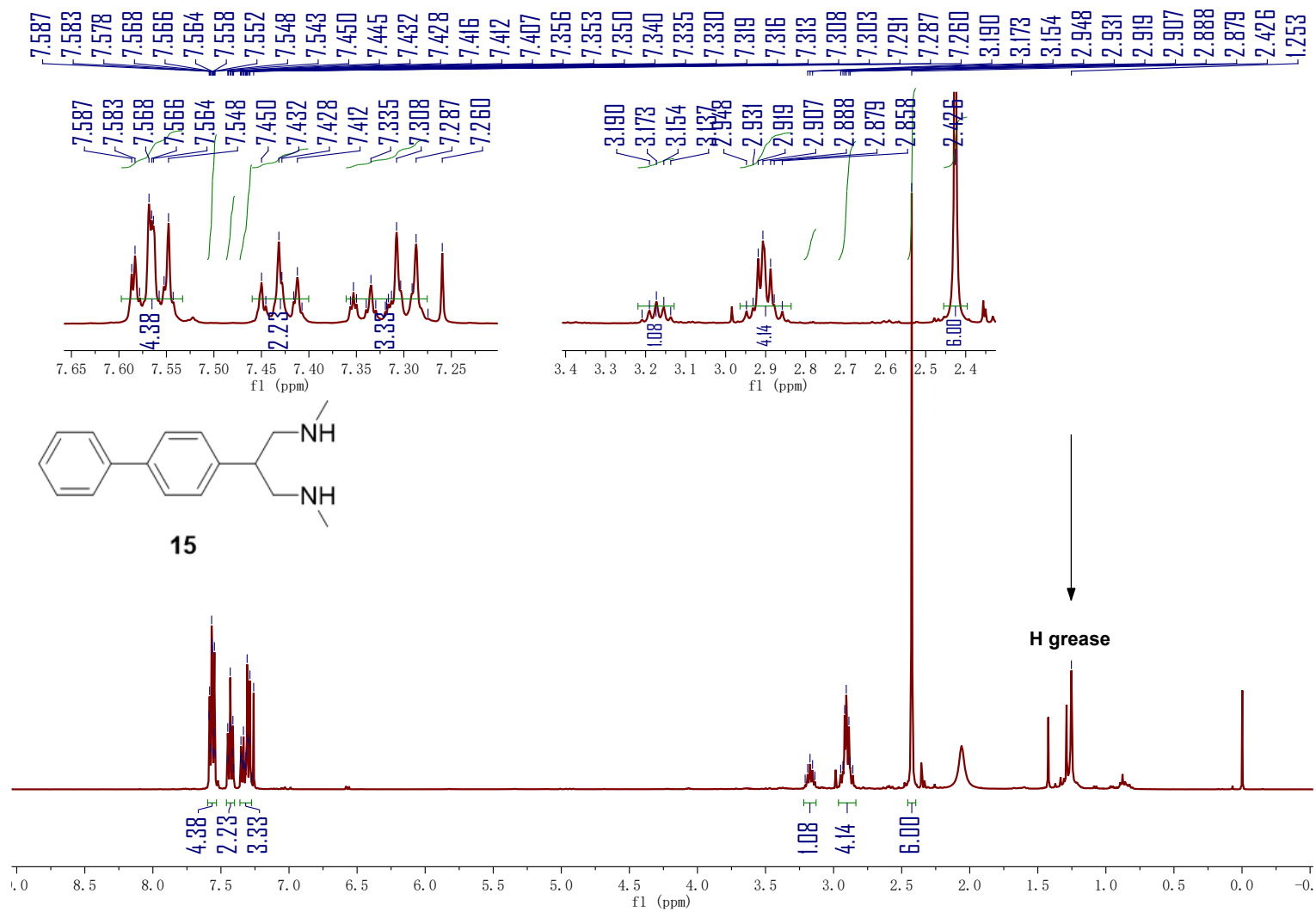


S184

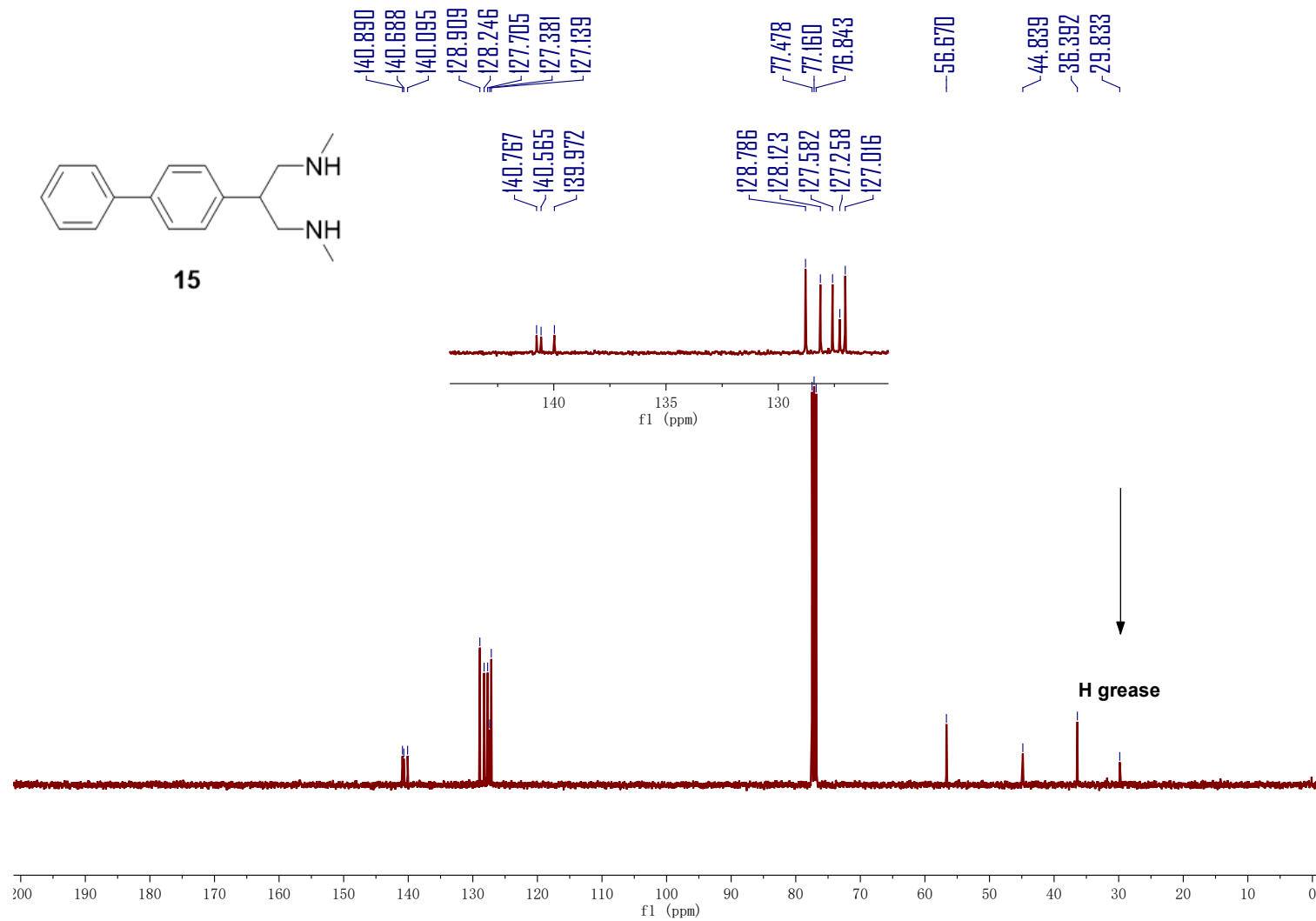
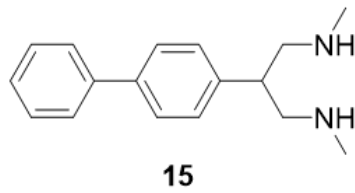




S185



S186



S187