

## Supporting Information

# **Cobalt-catalyzed, directed C-H functionalization/annulation of phenylglycinol derivatives with alkynes**

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## Contents

General considerations .....	S3
Cobalt-catalyzed $sp^2$ C-H functionalization.....	S4
1. Optimization of cobalt-catalyzed $sp^2$ C-H functionalization .....	S4
3.1. Oxidant .....	S4
3.2. Base.....	S5
3.3. Solvent .....	S5
3.4. Catalyst .....	S6
3.5. Temperature .....	S7
2. Cobalt-catalyzed $sp^2$ C-H alkenylation using literature procedure for benzamides .....	S9
Mehanistic considerations.....	S10
NMR spectra .....	S11
HPLC data.....	S221

## General considerations

Reactions were performed using standard glassware or were run in 4 mL vials with PTFE/Liner screw caps and 30 mL vials using w/polyseal screw caps. Reactions were heated using Chemglass aluminium reaction blocks. Column chromatography was performed using Kieselgel silicagel (35 -70 and 60 - 200  $\mu\text{m}$ ). Thin layer chromatography (TLC) was performed on silica gel using Merck TLC Silica gel 60 F254 Aluminum sheets and was visualized by UV lamp, staining with  $\text{KMnO}_4$ .  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$  and 2D-NMR spectra were recorded on 400 MHz Bruker spectrometer using residual solvent peak as a reference. Compounds for HRMS were analyzed by positive mode electrospray ionization (ESI) using Waters Synapt G2-Si mass spectrometer. HPLC data were obtained using Waters Alliance 2695 HPLC system with a Phenomenex Lux Amylose-1 (4.6 x 150 mm) or Chiralpac IC-1 (4.6 x 250 mm) column (conditions specified on attached HPLC chromatograms). IR spectra were obtained using a Shimadzu IR Prestige-21 FT-IR spectrometer. Optical rotations were measured at 20  $^\circ\text{C}$  on a Rudolph Research Analytical Autopol VI Polarimeter, cell length 50 mm, using solvent and concentration stated, at 589 nm. All procedures were performed under ambient air unless otherwise noted. Reagents and starting materials were obtained from commercial sources and used without further purification unless otherwise noted.

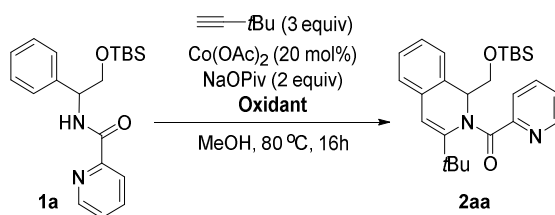
## Cobalt-catalyzed sp<sup>2</sup> C-H functionalization

### 1. Optimization of cobalt-catalyzed sp<sup>2</sup> C-H functionalization

#### 3.1. Oxidant

General procedure for oxidant optimization experiments.

A 4 mL vial with a screw cap (PTFE/Liner) was charged with *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)picolinamide (35.6 mg, 0.1 mmol), oxidant (0.2 – 0.3 mmol, 2 – 3 equiv), NaOPiv (24.8 mg, 0.2 mmol, 2 equiv), Co(OAc)<sub>2</sub> (3.5 mg, 0.02 mmol, 20 mol%), 3,3-dimethyl-1-butyne (37 μL, 0.3 mmol, 3 equiv), and MeOH (1.0 mL). Resulting mixture was heated at 80 °C for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1). To reaction mixture Ph<sub>3</sub>CH (24.4 mg, 0.1 mmol, 1 equiv) was added, mixture was diluted with dist. H<sub>2</sub>O (1.5 mL) and extracted with EtOAc (3 x 1.5 mL). Combined organic phase was separated, dried over anh. Na<sub>2</sub>SO<sub>4</sub>, filtered, evaporated. The residue was dissolved in CDCl<sub>3</sub> and analyzed by <sup>1</sup>H-NMR spectroscopy.



**Table S1.** Oxidant optimization experiments

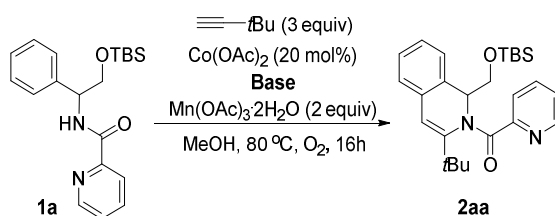
Entry	Oxidant (equiv)	Substrate : Product ratio	NMR yield, % <sup>a</sup>
1	AgOAc (2)	17:1	5
2	AgOPiv (2)	1:0	ND
3	MnO <sub>2</sub> (2)	11:1	4
4	Mn(acac) <sub>3</sub> (2)	1:0	ND
5	Mn(OAc) <sub>2</sub> ·4H <sub>2</sub> O (2)	19:1	5
6	Mn(OAc) <sub>3</sub> ·2H <sub>2</sub> O (2)	7:1	12
7	Mn(OAc) <sub>3</sub> ·2H <sub>2</sub> O (3)	7:1	12
8	O <sub>2</sub>	1:0	ND
9	Mn(OAc) <sub>3</sub> ·2H <sub>2</sub> O (2)/O <sub>2</sub>	5.3:1	16
10	w/o oxidant	1:0	ND

<sup>a</sup>NMR yield using triphenylmethane as an internal standard. ND – not determined.

### 3.2. Base

#### General procedure for base optimization experiments.

A 4 mL vial with a screw cap (PTFE/Liner) was charged with *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)picolinamide (35.6 mg, 0.1 mmol), Mn(OAc)<sub>3</sub> · 2 H<sub>2</sub>O (53.6 mg, 2 equiv), base (0.12 - 0.3 mmol, 1.2 - 3 equiv), Co(OAc)<sub>2</sub> ( 3.5 mg, 0.02 mmol, 20 mol%), 3,3-dimethyl-1-butyne (37 μL, 0.3 mmol, 3 equiv), and MeOH (1.0 mL). Vial was purged with O<sub>2</sub> for 1 minute and the resulting mixture was heated at 80 °C for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1). To reaction mixture Ph<sub>3</sub>CH (24.4 mg, 0.1 mmol, 1 equiv) was added, mixture was diluted with dist. H<sub>2</sub>O (1.5 mL) and extracted with EtOAc (3 x 1.5 mL). Combined organic phase was separated, dried over anh. Na<sub>2</sub>SO<sub>4</sub>, filtered, evaporated. The residue was dissolved in CDCl<sub>3</sub> and analyzed by <sup>1</sup>H-NMR spectroscopy.



**Table S2.** Base optimization experiments

Entry	Base (equiv)	Substrate : Product ratio	NMR yield, % <sup>a</sup>
1	NaOAc (2)	7.3:1	12
2	PhCOONa (2)	2.8:1	24
3	AdCOONa (2)	3.4:1	20
4	NaOPiv (2)	5.3:1	16
5	NaOPiv (1.2)	2.5:1	28
6	NaOPiv (3)	2.6:1	25
7	w/o base	4.5:1	16

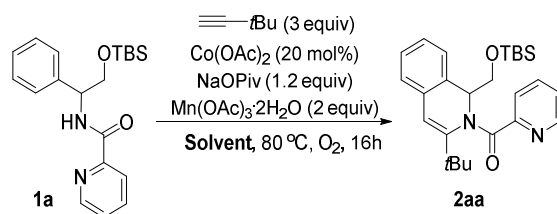
<sup>a</sup> NMR yield using triphenylmethane as an internal standard.

### 3.3. Solvent

#### General procedure for solvent optimization experiments.

A 4 mL vial with a screw cap (PTFE/Liner) was charged with *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)picolinamide (35.6 mg, 0.1 mmol), Mn(OAc)<sub>3</sub> · 2 H<sub>2</sub>O (53.6 mg, 2 equiv), NaOPiv (14.9 mg, 0.12 mmol, 1.2 equiv), Co(OAc)<sub>2</sub> ( 3.5 mg, 0.02 mmol, 20 mol%), 3,3-dimethyl-1-butyne (37 μL, 0.3 mmol, 3 equiv), and solvent (1.0 mL). Vial was purged with O<sub>2</sub> for 1 minute and

the resulting mixture was heated at 80 °C for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1). To reaction mixture Ph<sub>3</sub>CH (24.4 mg, 0.1 mmol, 1 equiv) was added, mixture was diluted with dist. H<sub>2</sub>O (1.5 mL) and extracted with EtOAc (3 x 1.5 mL). Combined organic phase was separated, dried over anh. Na<sub>2</sub>SO<sub>4</sub>, filtered, evaporated. The residue was dissolved in CDCl<sub>3</sub> and analyzed by <sup>1</sup>H-NMR spectroscopy.



**Table S3.** Solvent optimization experiments

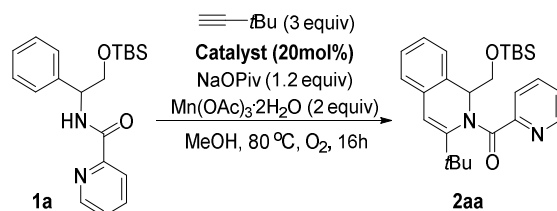
Entry	Solvent	Substrate : Product ratio	NMR yield, % <sup>a</sup>
1	MeOH	2.5:1	28
2	EtOH	98:1	1
3	TFE	6.7:1	13
4	DCE	32:1	3
5	PhCl	32:1	3
6	Toluene	17.6:1	5

<sup>a</sup>NMR yield using triphenylmethane as an internal standard.

### 3.4. Catalyst

*General procedure for catalyst optimization experiments.*

A 4 mL vial with a screw cap (PTFE/Liner) was charged with *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)picolinamide (35.6 mg, 0.1 mmol), Mn(OAc)<sub>3</sub> · 2 H<sub>2</sub>O (53.6 mg, 2 equiv), NaOPiv (14.9 mg, 0.12 mmol, 1.2 equiv), catalyst (0.01 - 0.02 mmol, 10 - 20 mol%), 3,3-dimethyl-1-butyne (37 μL, 0.3 mmol, 3 equiv), and MeOH (1.0 mL). Vial was purged with O<sub>2</sub> for 1 minute and the resulting mixture was heated at 80 °C for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1). To reaction mixture Ph<sub>3</sub>CH (24.4 mg, 0.1 mmol, 1 equiv) was added, mixture was diluted with dist. H<sub>2</sub>O (1.5 mL) and extracted with EtOAc (3 x 1.5 mL). Combined organic phase was separated, dried over anh. Na<sub>2</sub>SO<sub>4</sub>, filtered, evaporated. The residue was dissolved in CDCl<sub>3</sub> and analyzed by <sup>1</sup>H-NMR spectroscopy.



**Table S4.** Catalyst optimization experiments

Entry	Catalyst (mol%)	Substrate : Product ratio	NMR yield, % <sup>a</sup>
1	Co(OAc) <sub>2</sub> (20)	2.5:1	28
2	Co(acac) <sub>2</sub> (20)	2.3:1	30
3	CoCl <sub>2</sub> (20)	>10:1	ND
4	Co(acac) <sub>3</sub> (20)	5:1	17
5	Co(dpm) <sub>2</sub> (20)	1:13.7	82
6 <sup>b</sup>	Co(dpm) <sub>2</sub> (20)	1:16.8	84
7 <sup>c</sup>	Co(dpm) <sub>2</sub> (20)	1:1	49
8	Co(dpm) <sub>2</sub> (10)	1:1.4	57
9	Cu(OAc) <sub>2</sub> (20)	1:0	ND
10	w/o catalyst	1:0	ND

<sup>a</sup> NMR yield using triphenylmethane as an internal standard. <sup>b</sup> Time:24 h. <sup>c</sup> Without purging with O<sub>2</sub>.

*Note! Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)cobalt(II) (Co(dpm)<sub>2</sub>) can be used commercial (Alfa Aesar, CAS: 13986-53-3) or self-made (by procedure written below).*

### **Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)cobalt(II)**

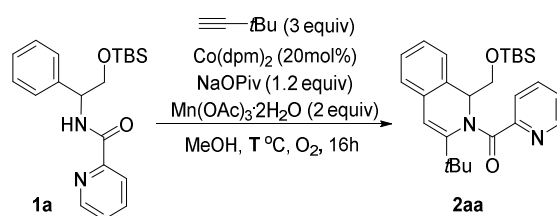
Co(NO<sub>3</sub>)<sub>2</sub> · 6 H<sub>2</sub>O (1.61 g, 5.53 mmol, 1 equiv) and 2,2,6,6-tetramethyl-3,5-heptanedione (2.36 mL, 11.29 mmol, 2.04 equiv) were dissolved in MeOH (15 mL), then 2 M NaOH<sub>aq</sub> (443 mg, 11.07 mmol, 15 mL dist. H<sub>2</sub>O) was added dropwise (formation of pink precipitate) and the resulting mixture was heated at 80 °C for 2 h. Mixture was cooled to room temperature, the pink precipitate was filtered off. Then precipitate was dissolved in Et<sub>2</sub>O (10 mL), filtered, filtrate was evaporated and dried under reduced pressure to give 1.5 g (64%) of bis(2,2,6,6-tetramethyl-3,5-heptanedionato)cobalt(II) as a pink powder, mp 142-144 °C (Et<sub>2</sub>O).

### **3.5. Temperature**

*General procedure for temperature optimization experiments.*

A 4 mL vial with a screw cap (PTFE/Liner) was charged with *N*-(2-((*tert*-butyldimethylsilyloxy)-1-phenylethyl)picolinamide (35.6 mg, 0.1 mmol), Mn(OAc)<sub>3</sub> · 2 H<sub>2</sub>O (53.6 mg, 2 equiv), NaOPiv (14.9 mg, 0.12 mmol, 1.2 equiv), Co(dpm)<sub>2</sub> (8.5 mg, 0.02 mmol, 20 mol%), 3,3-dimethyl-1-butyne (37 μL, 0.3 mmol, 3 equiv), and MeOH (1.0 mL). Vial was purged with O<sub>2</sub> for 1 minute and the

resulting mixture was heated at corresponding temperature for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1). To reaction mixture Ph<sub>3</sub>CH (24.4 mg, 0.1 mmol, 1 equiv) was added, mixture was diluted with dist. H<sub>2</sub>O (1.5 mL) and extracted with EtOAc (3 x 1.5 mL). Combined organic phase was separated, dried over anh. Na<sub>2</sub>SO<sub>4</sub>, filtered, evaporated. The residue was dissolved in CDCl<sub>3</sub> and analyzed by <sup>1</sup>H-NMR spectroscopy.



**Table S5.** Temperature optimization experiments

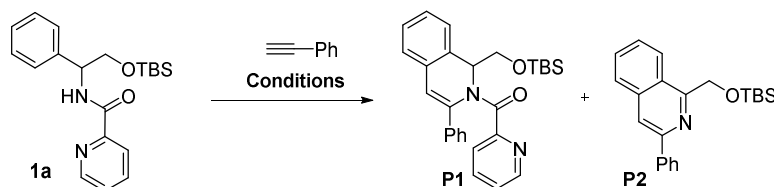
Entry	Temperature, °C	Substrate : Product ratio	NMR yield, % <sup>a</sup>
1	80	1:13.7	82
2	60	2.3:1	29

<sup>a</sup>NMR yield using triphenylmethane as an internal standard.

## 2. Cobalt-catalyzed $sp^2$ C-H alkenylation using literature procedure for benzamides

We tested the reported reaction conditions for cobalt catalyzed benzamide  $sp^2$  C-H alkenylation with alkynes using our model substrate – *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)-picolinamide.

*Note! This substrate was not tested by Carretero, Cui and Daugulis groups.*



**Table S6.** Control reactions using literature procedures

Entry	Conditions	NMR yield, % <sup>a</sup> 1a:P1:P2	Reference
1	Picolinamide <b>1a</b> (0.1 mmol, 1 equiv), phenylacetylene (1.5 equiv), Co(OAc) <sub>2</sub> (0.015 mmol, 15 mol%), NaOAc (0.15 mmol, 1.5 equiv), EtOH (1 mL), O <sub>2</sub> , 100 °C, 16h	NR	1
2	Picolinamide <b>1a</b> (0.1 mmol, 1 equiv), phenylacetylene (1.5 equiv), Co(OAc) <sub>2</sub> (0.015 mmol, 15 mol%), NaOAc (0.15 mmol, 1.5 equiv), 1,4-Dioxane (1 mL), O <sub>2</sub> , 100 °C, 2.5h	NR	
3	Picolinamide <b>1a</b> (0.1 mmol, 1 equiv), phenylacetylene (1.5 equiv), Co(OAc) <sub>2</sub> ·4 H <sub>2</sub> O (0.05 mmol, 50 mol%), KPF <sub>6</sub> (0.05 equiv, 50 mol%), PEG-400, O <sub>2</sub> , 140 °C, 16h	33:13:2	2
4	Picolinamide <b>1a</b> (0.1 mmol, 1 equiv), phenylacetylene (1.5 equiv), Co(OAc) <sub>2</sub> ·4 H <sub>2</sub> O (0.1 mmol, 1 equiv), NaOPiv (0.2 mmol, 2 equiv), Mn(OAc) <sub>2</sub> (0.2 mmol, 2 equiv), air, TFE (1 mL), 100 °C, 16h	98:2:0	3

<sup>a</sup>NMR yield using triphenylmethane as an internal standard.

<sup>1</sup> Martínez, M.; Rodríguez, N.; Gjmez-Arrayás, R.; Carretero, J. C. Cobalt-Catalyzed *ortho*-C–H Functionalization/Alkyne Annulation of Benzylamine Derivatives: Access to Dihydroisoquinolines. *Chem. Eur. J.* **2017**, 23, 11669.

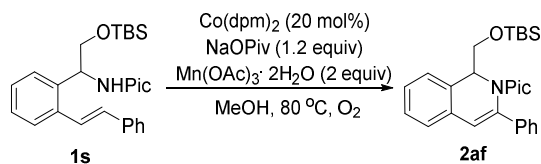
<sup>2</sup> Kuai, C.; Wang, L.; Li, B.; Yang, Z.; Cui, X. Cobalt-Catalyzed Selective Synthesis of Isoquinolines Using Picolinamide as a Traceless Directing Group. *Org. Lett.* **2017**, 19, 2102.

<sup>3</sup> Grigorjeva, L.; Daugulis, O. Cobalt-Catalyzed, Aminoquinoline-Directed C( $sp^2$ )-H Bond Alkenylation by Alkynes. *Angew. Chem. Int. Ed.* **2014**, 53, 10209.

## Mechanistic considerations

### Cyclization of picolinamide **1s**

Reactions were performed using general procedure for cobalt-catalyzed  $sp^2$  C-H alkenylation/cyclization.



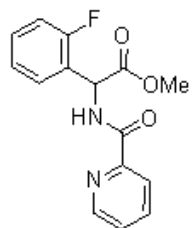
**Table S7.** Cyclization of picolinamide **1d**

Entry	Conditions	NMR yield <b>2af</b> , % <sup>a</sup>	Comments
1	w/o $\text{Co(dpm)}_2$	0	No reaction
2	Standard	0	Formation of byproducts

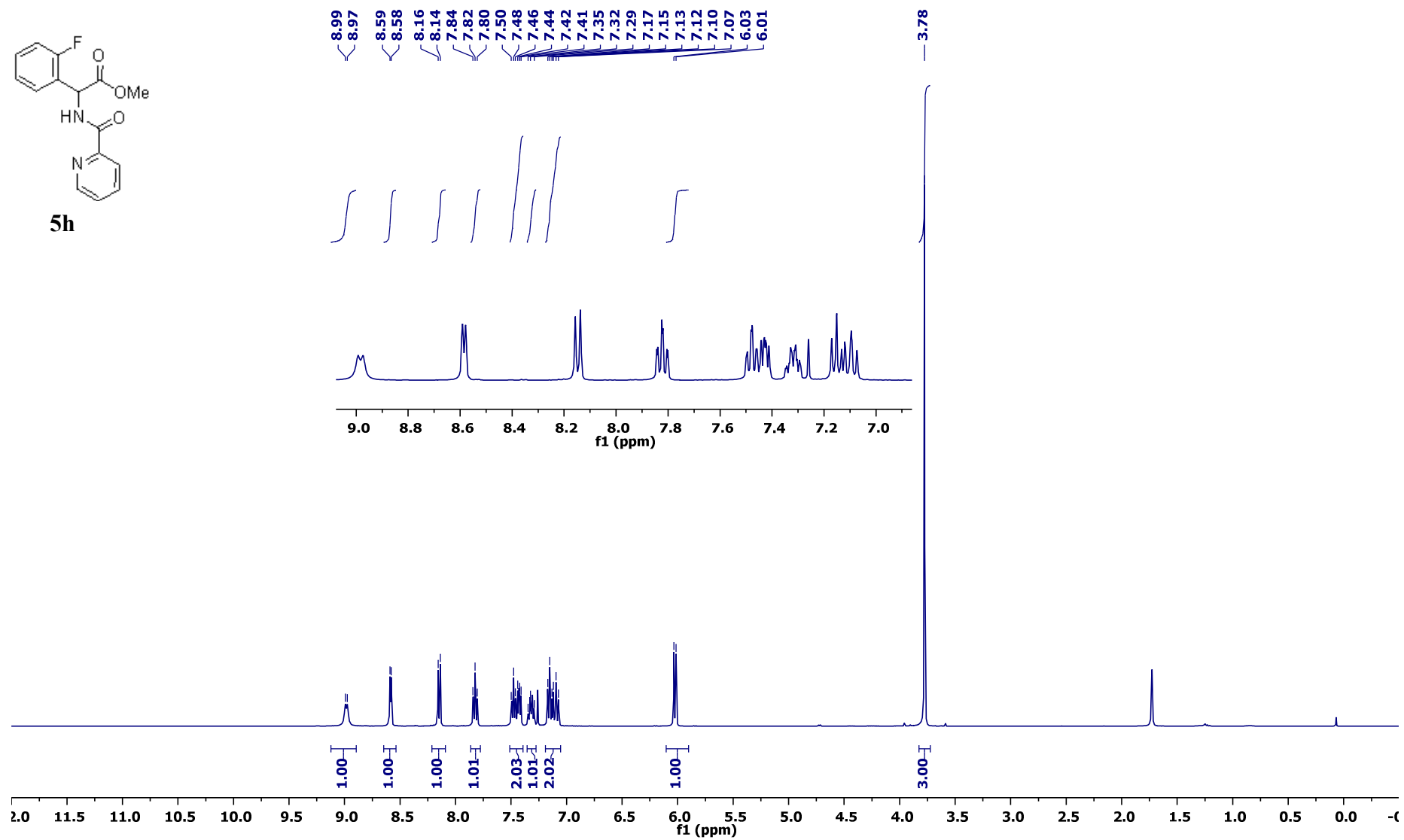
<sup>a</sup> NMR yield using triphenylmethane as an internal standard.

# NMR spectra

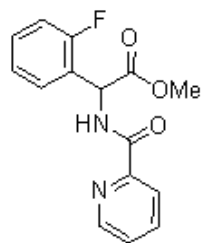
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



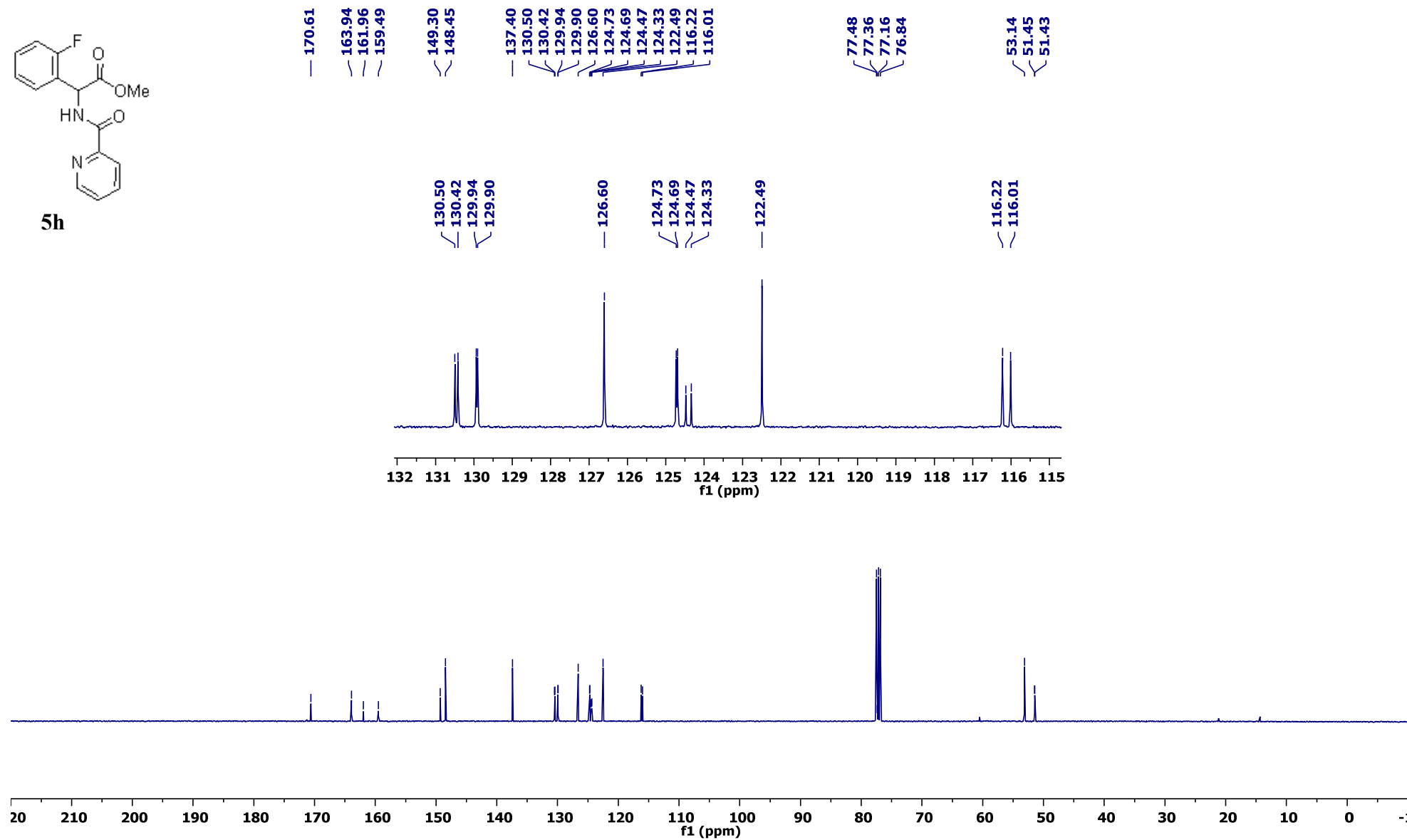
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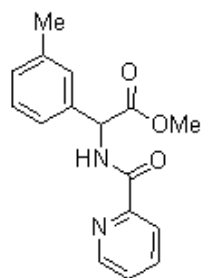
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



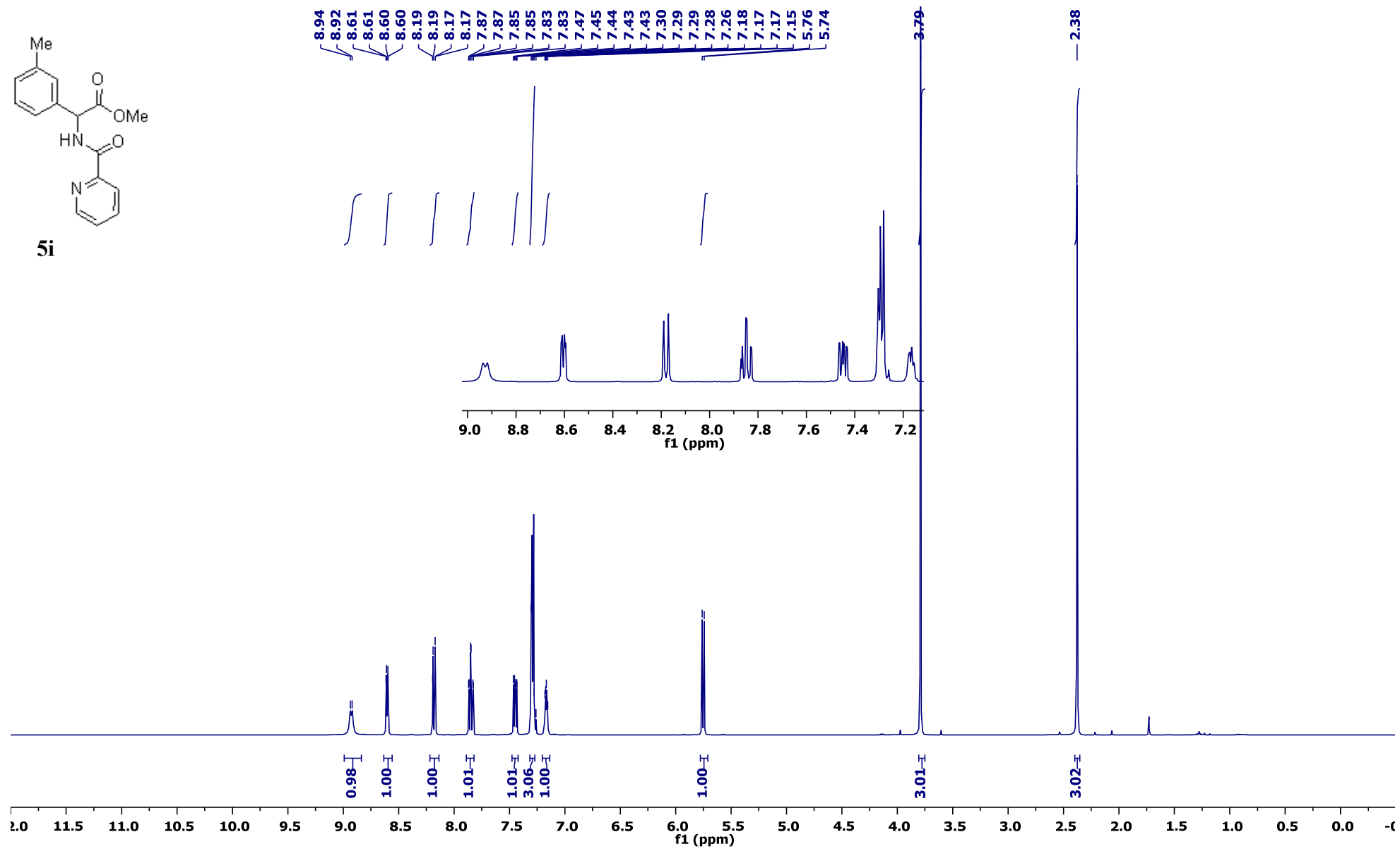
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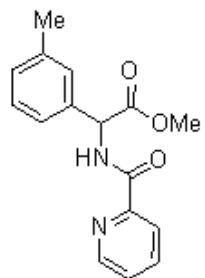
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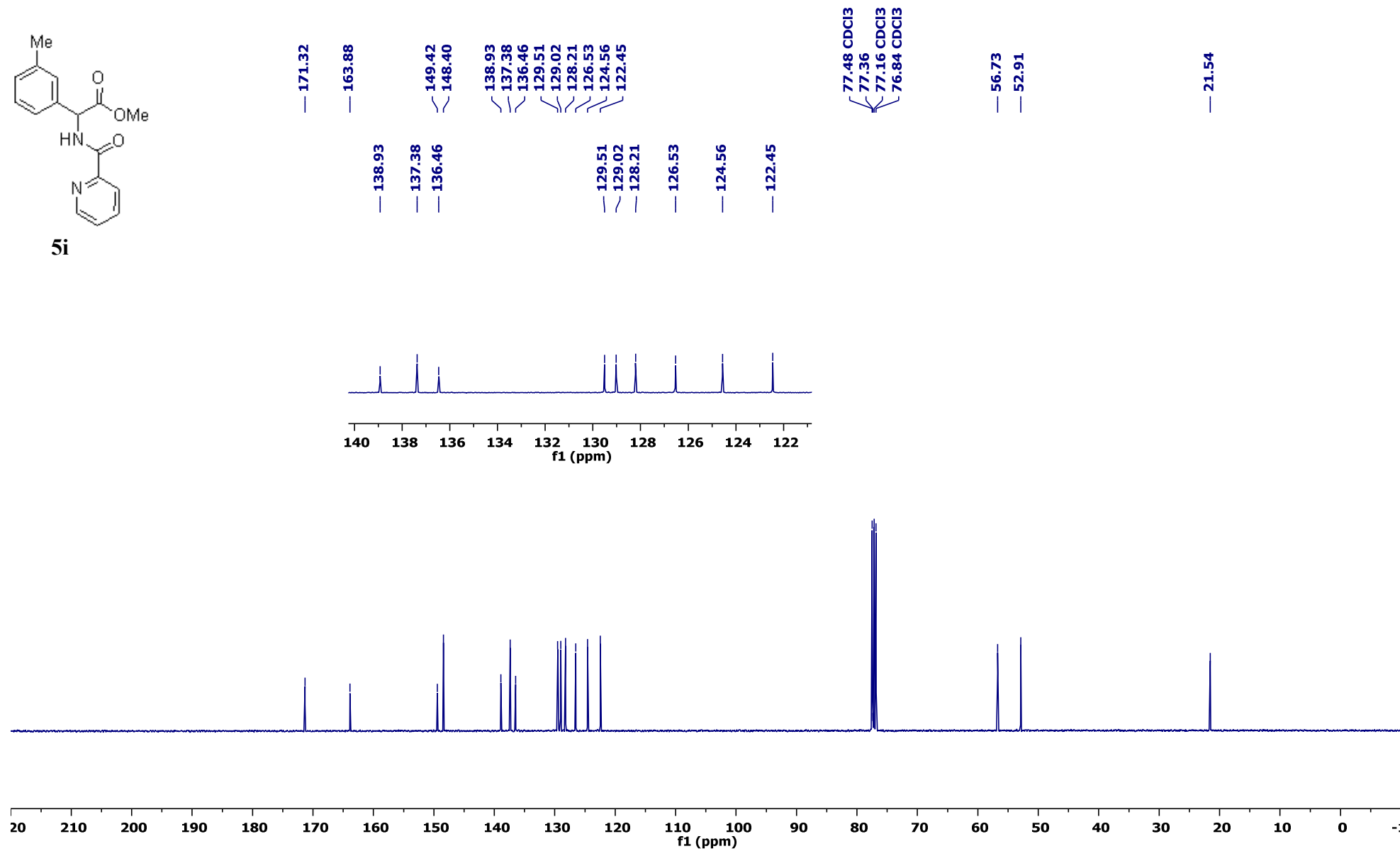
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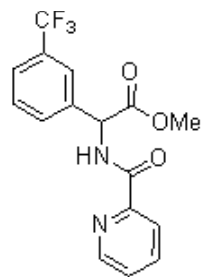
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



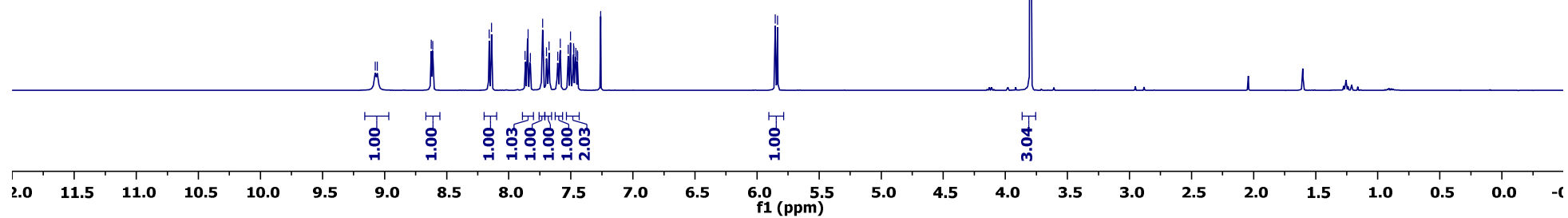
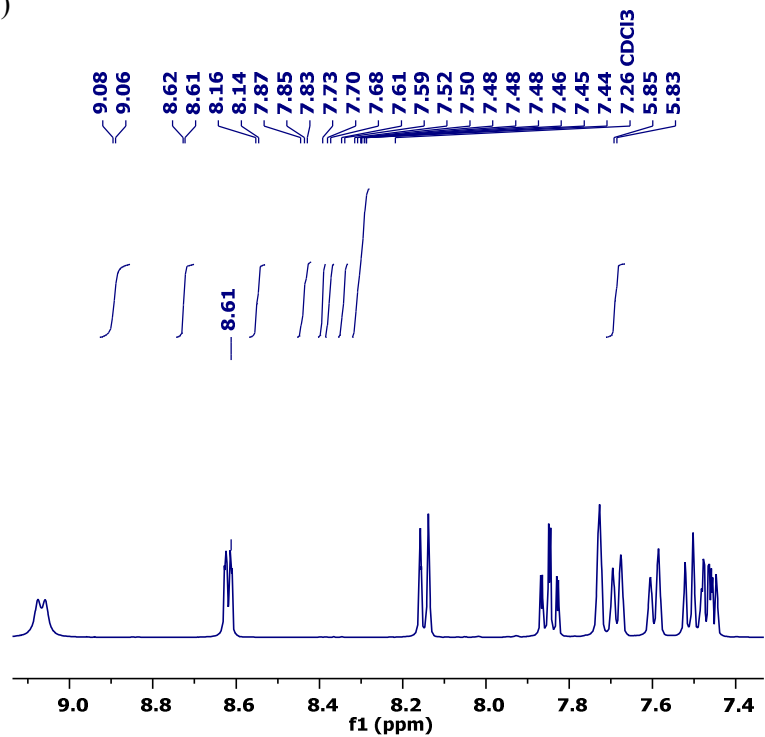
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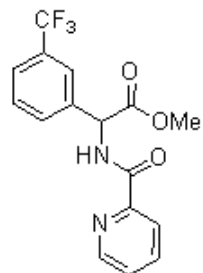
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



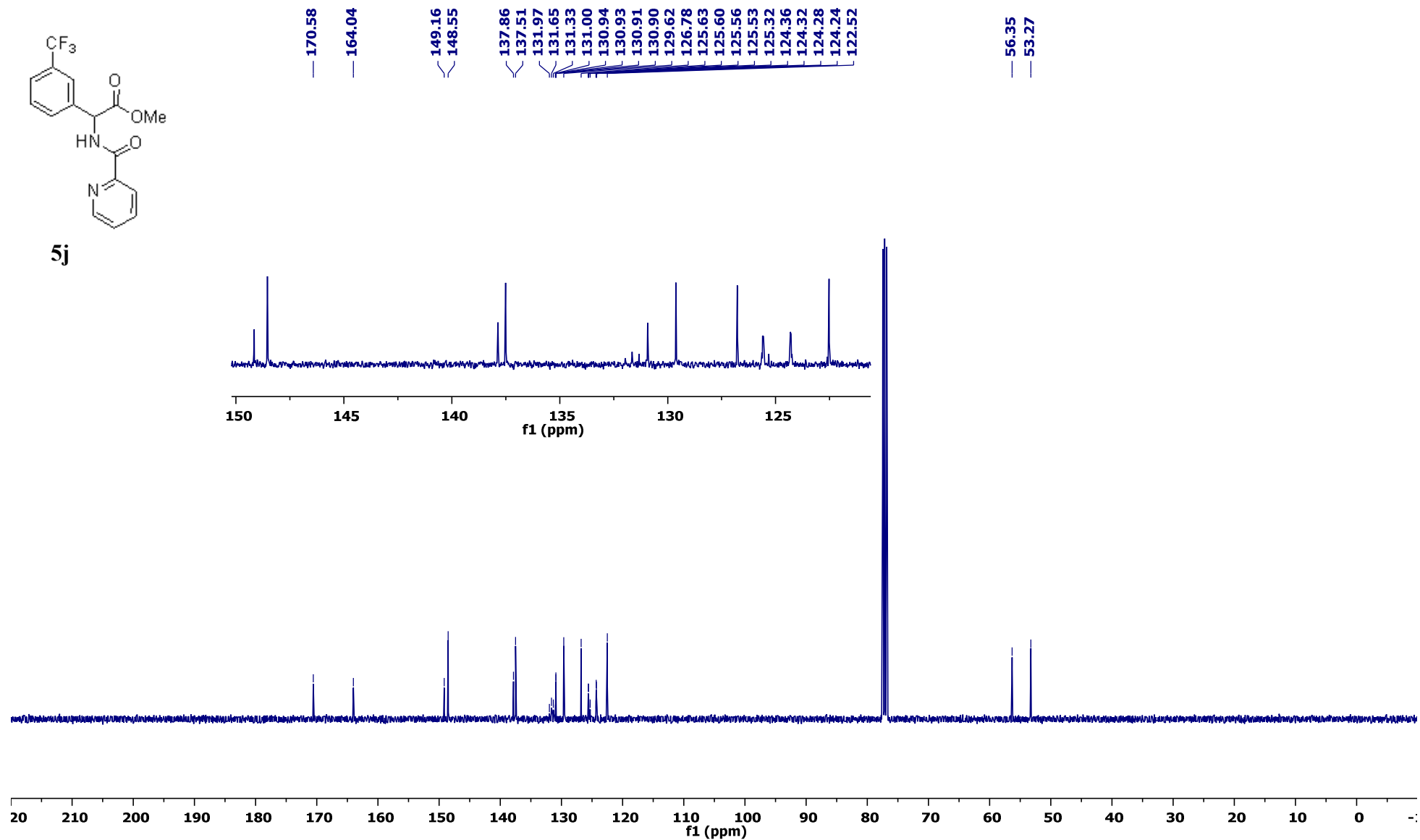
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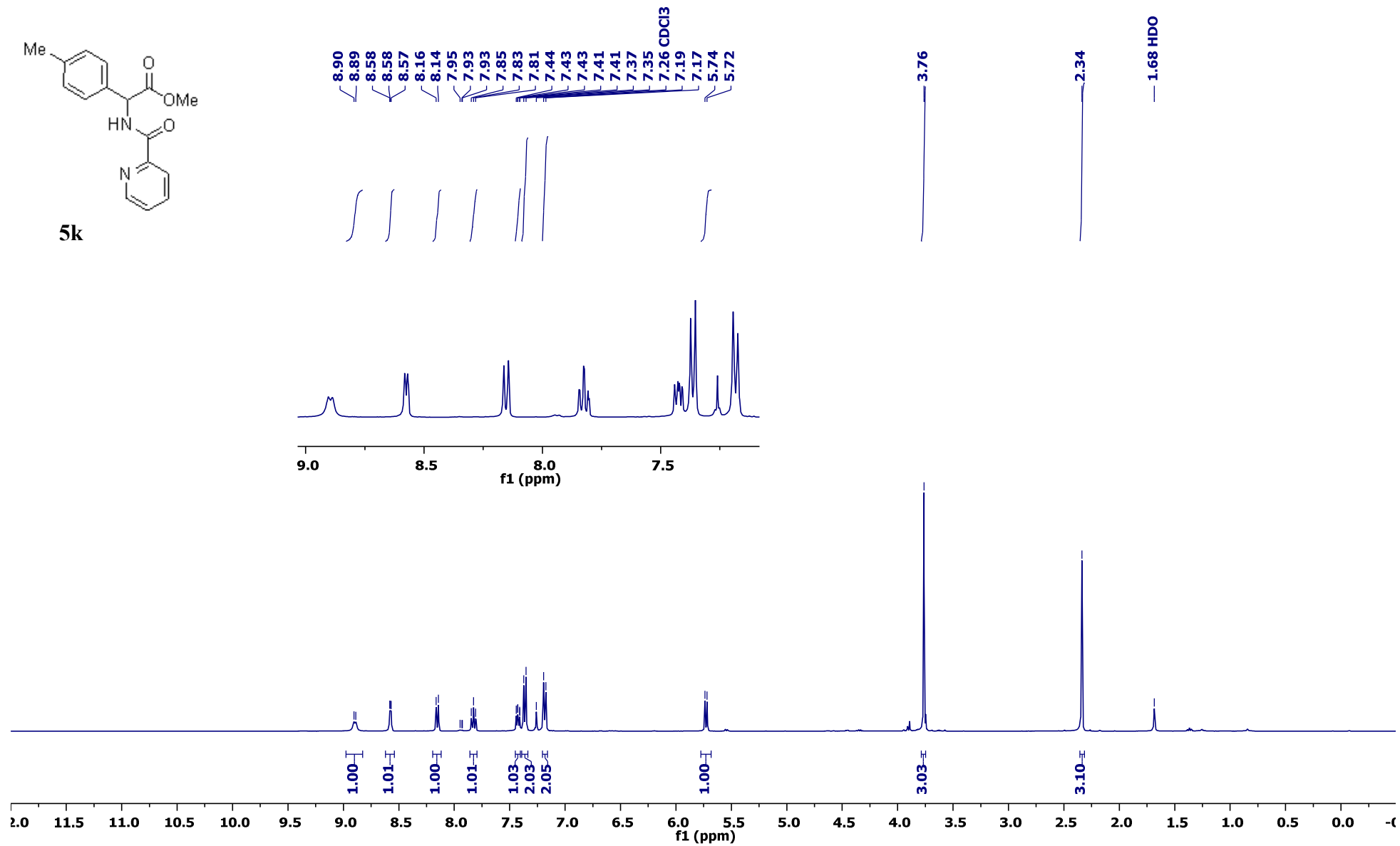
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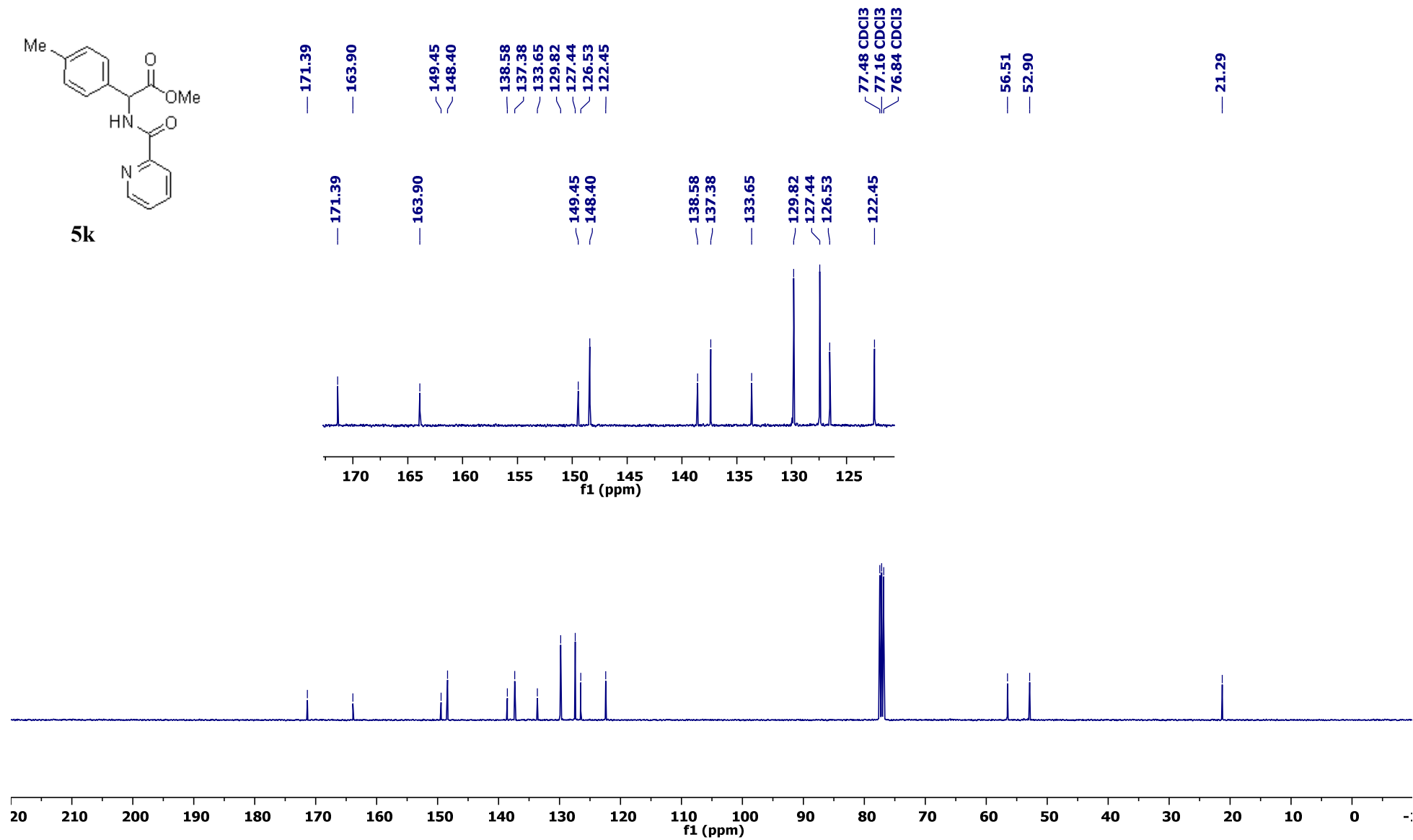
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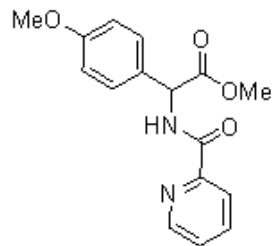
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



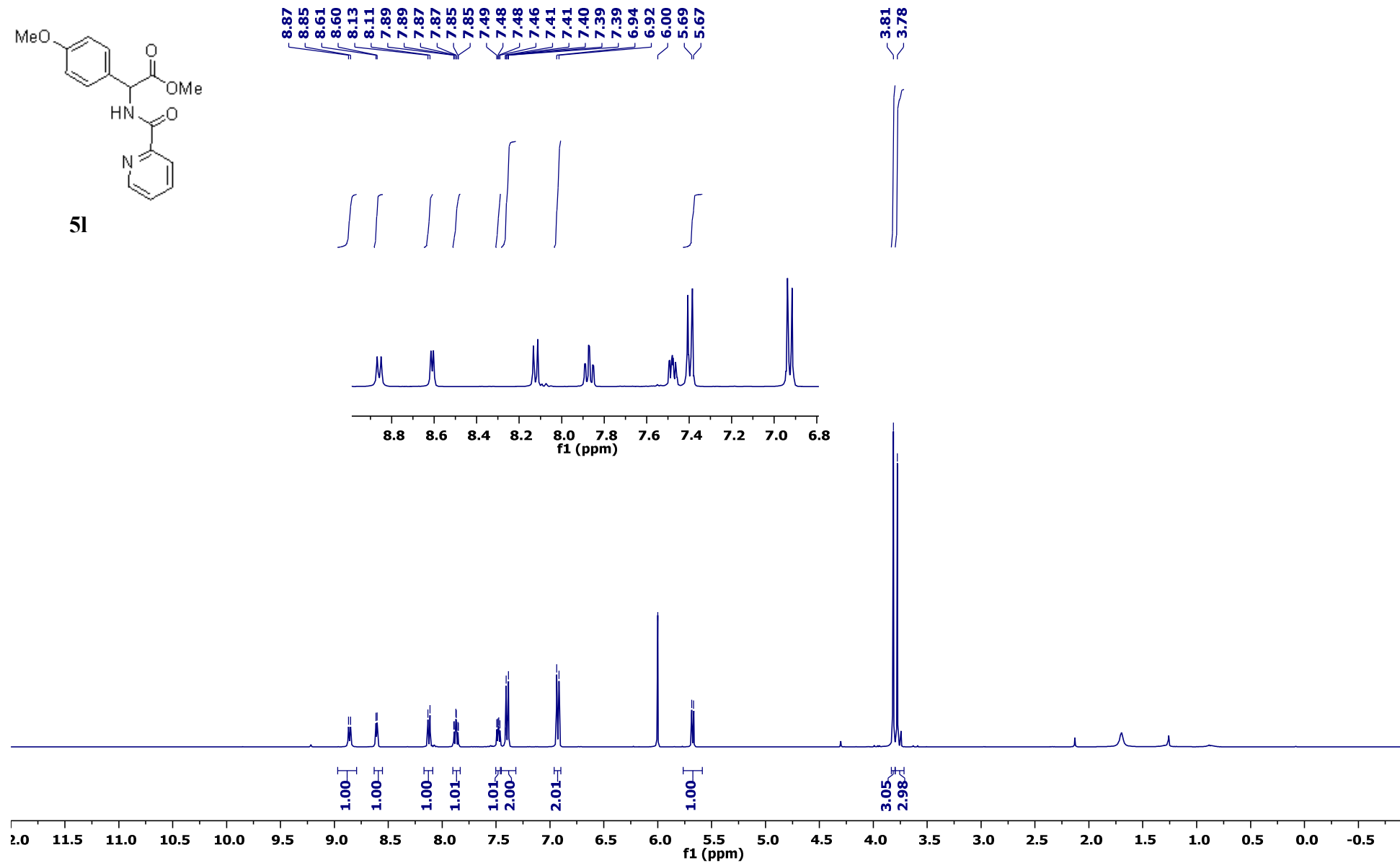
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



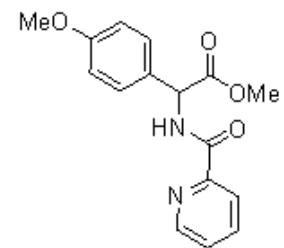
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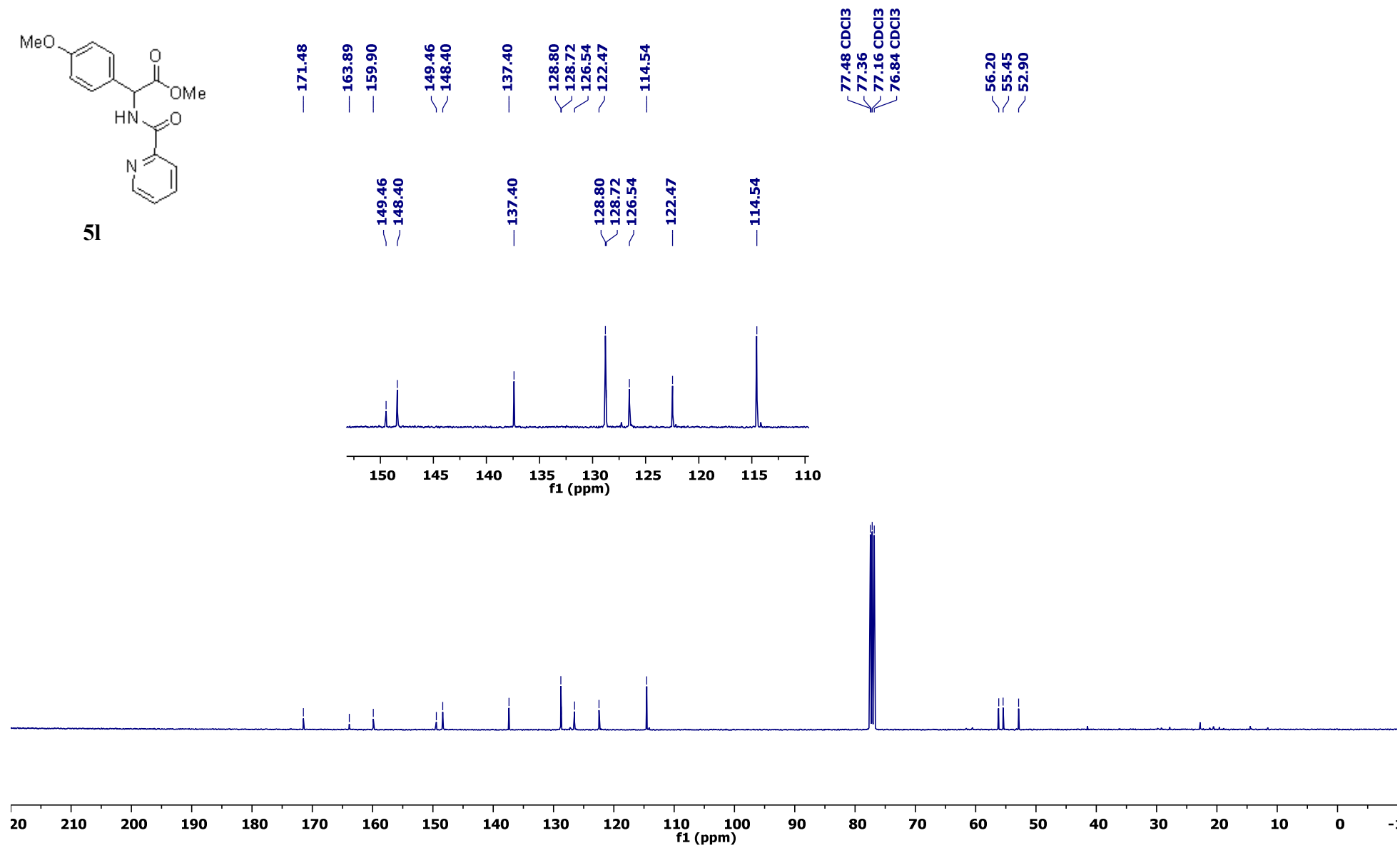
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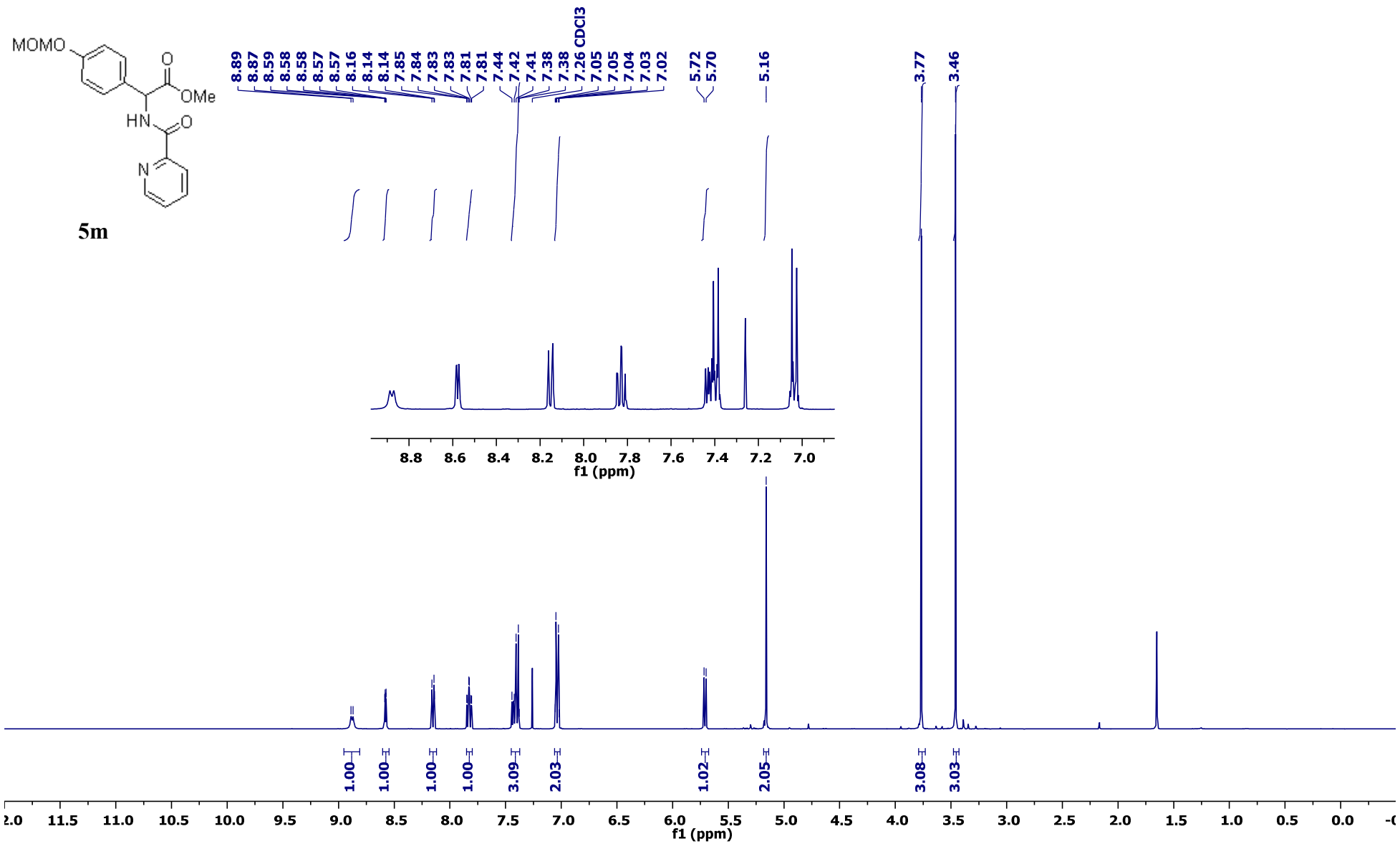
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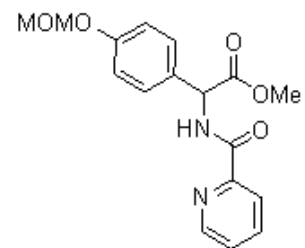
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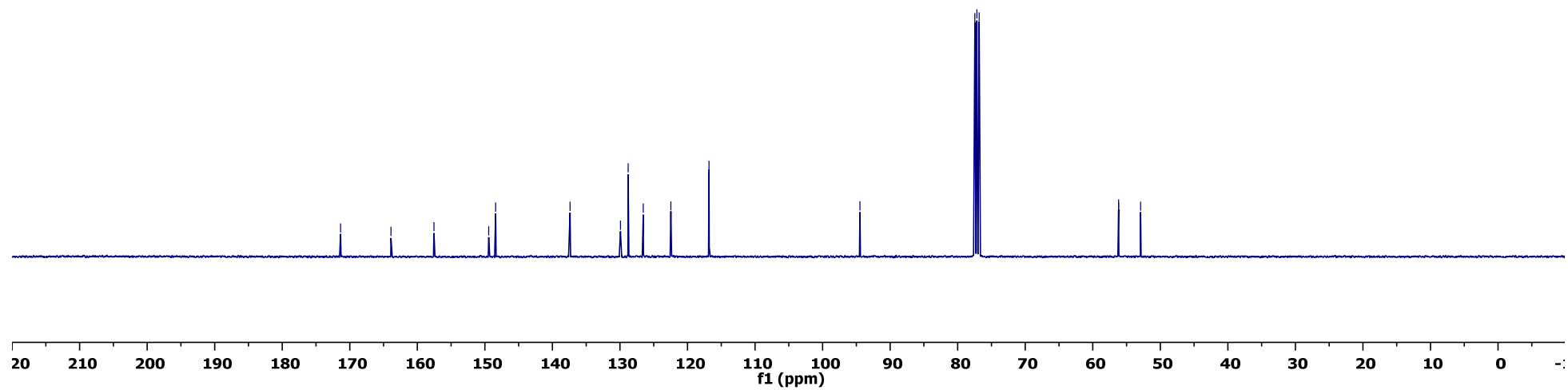
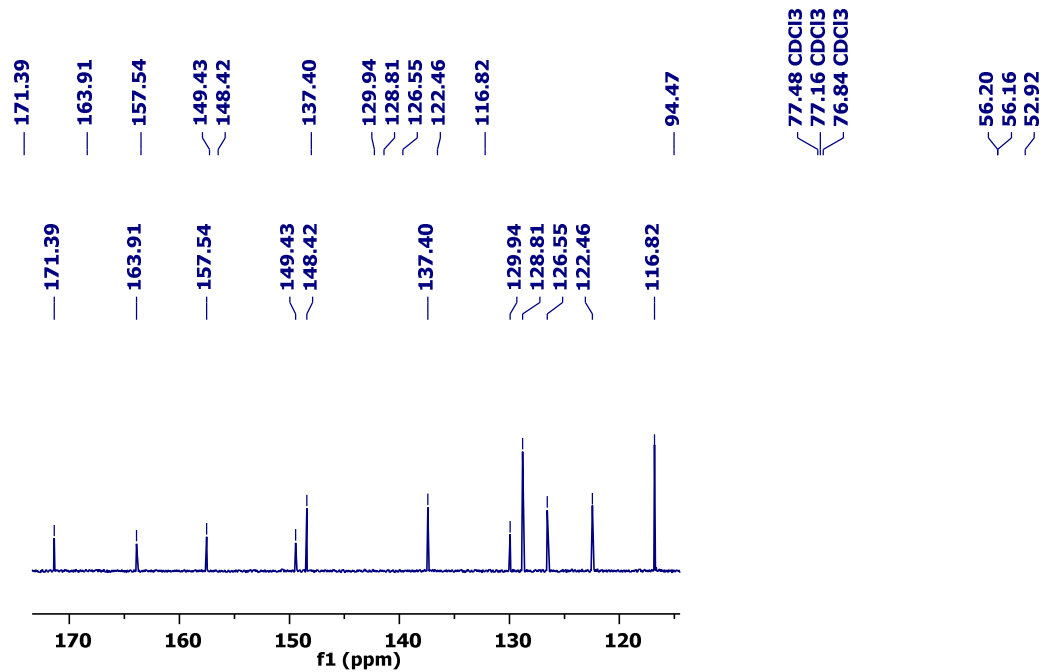
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



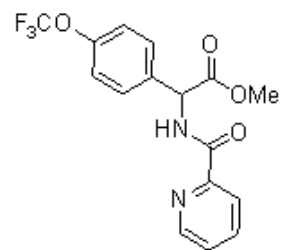
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



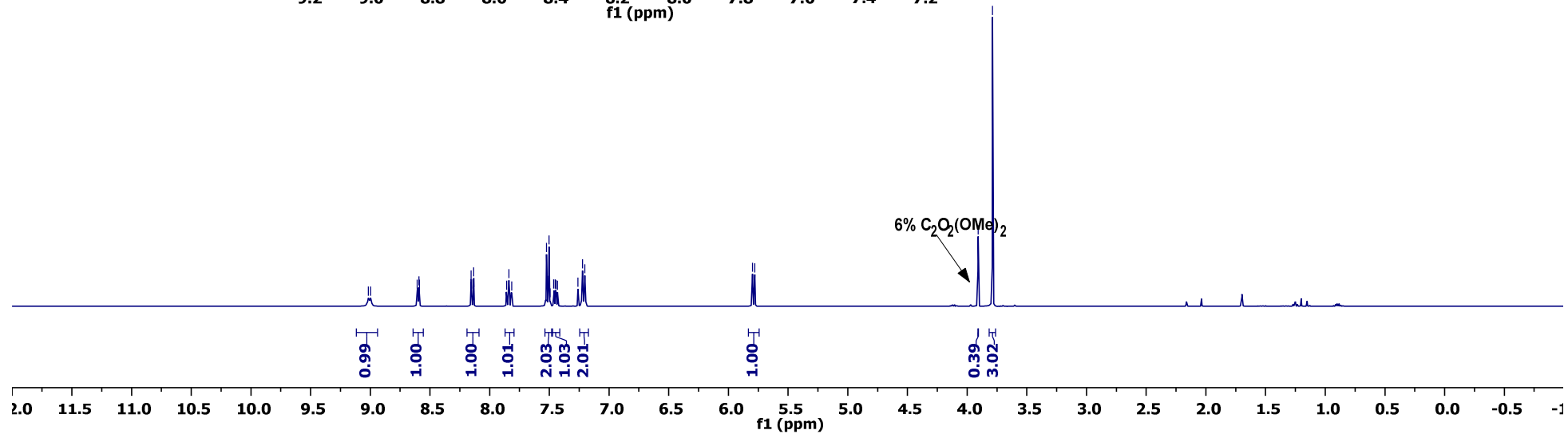
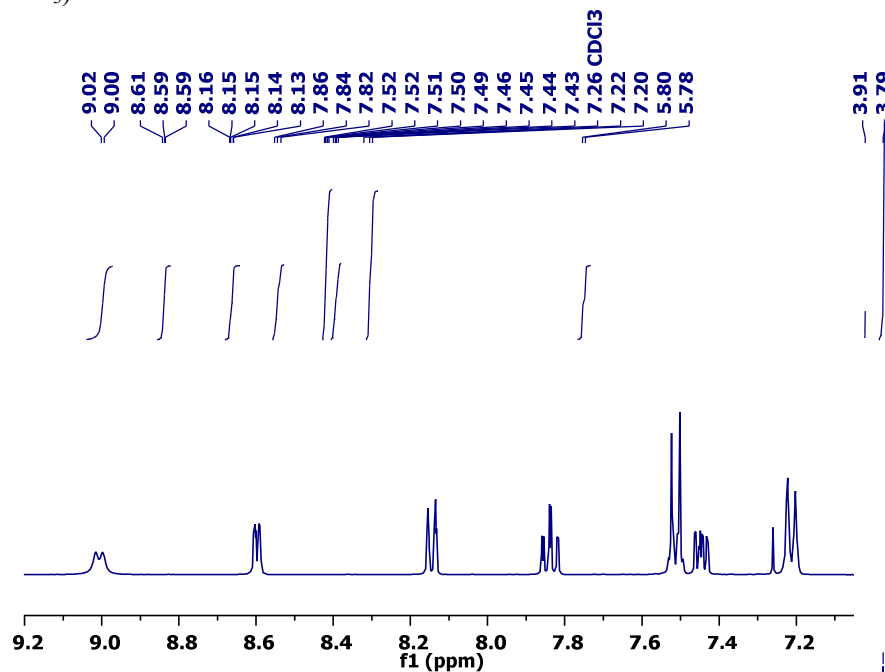
5m



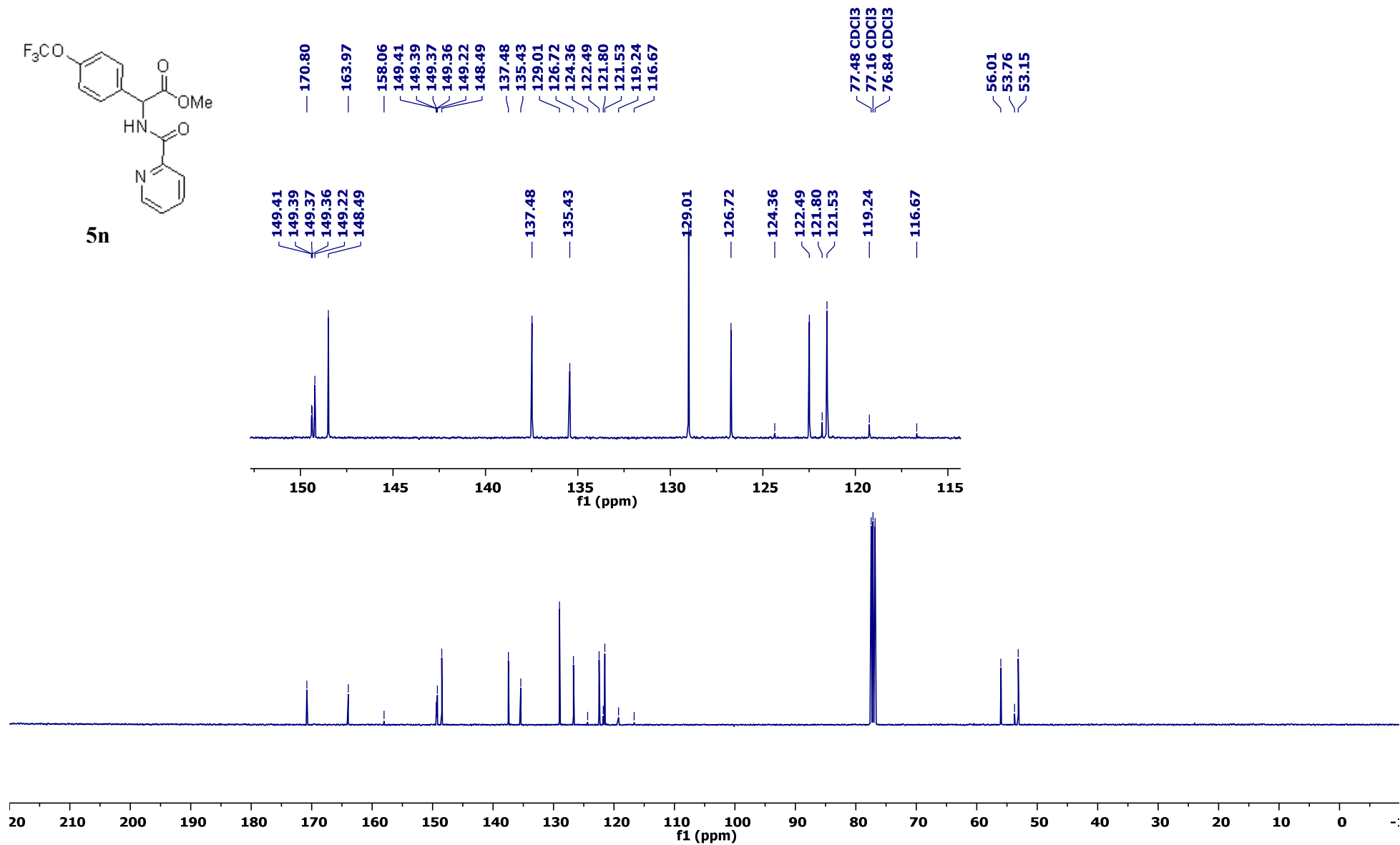
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



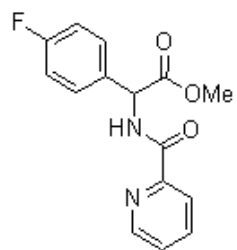
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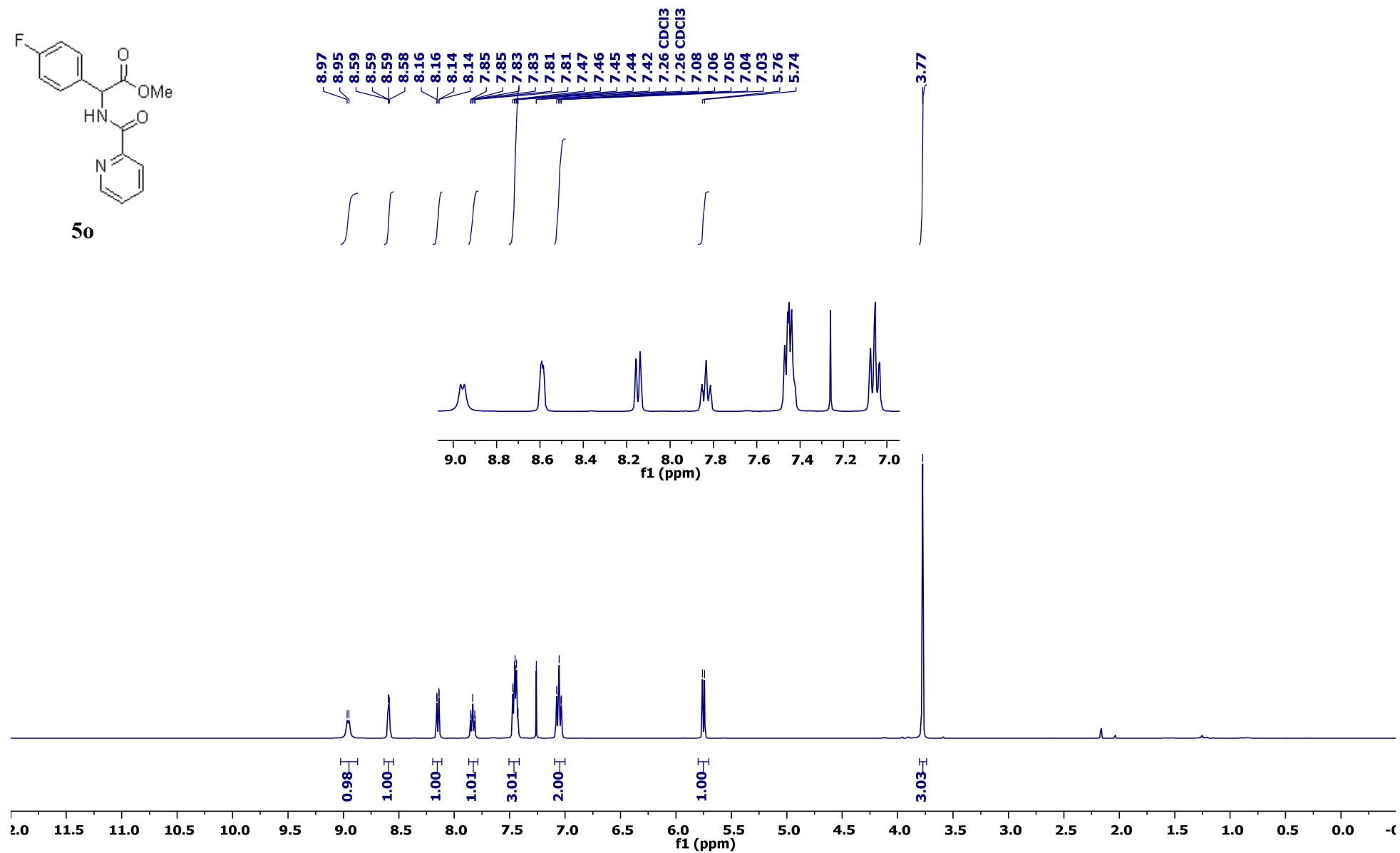
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



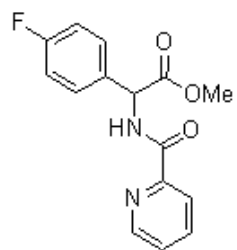
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



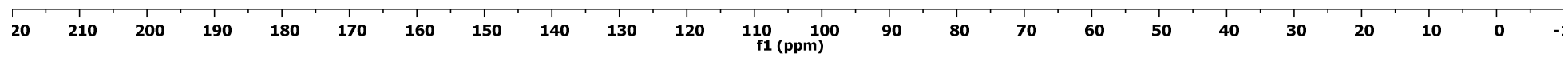
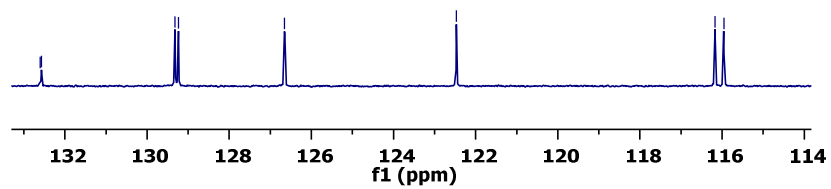
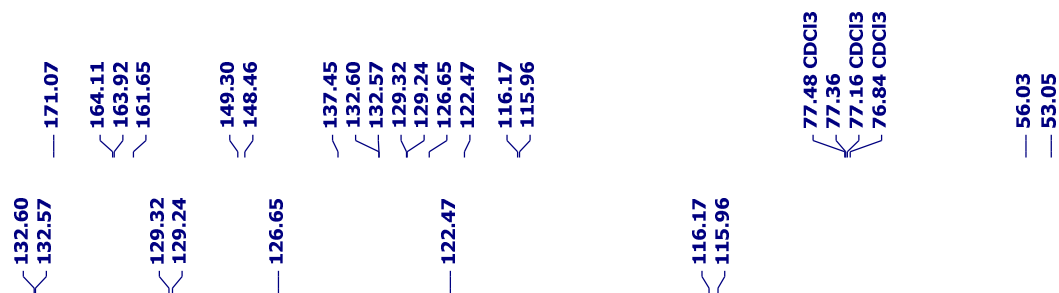
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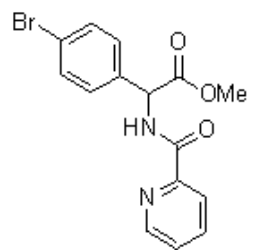
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



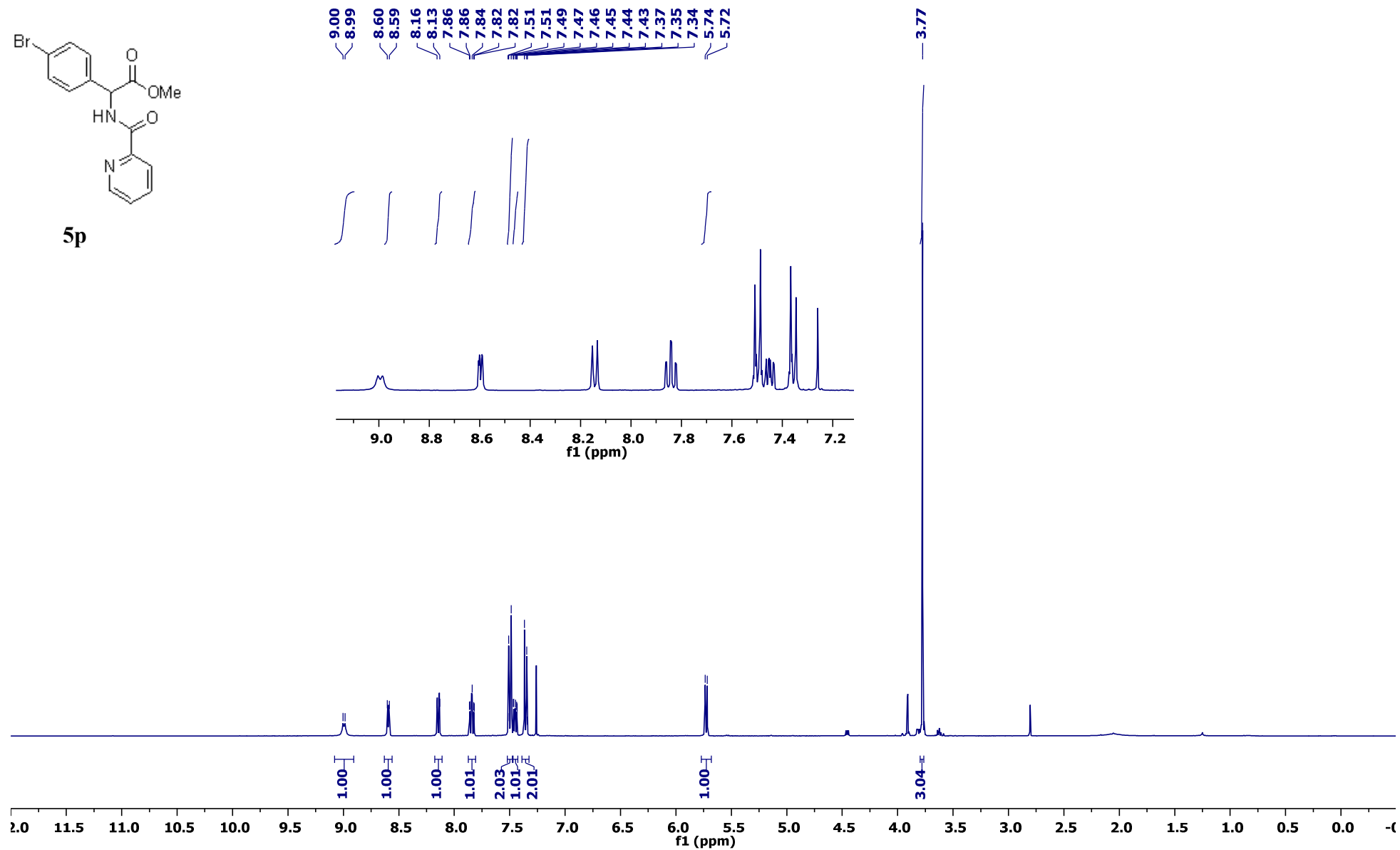
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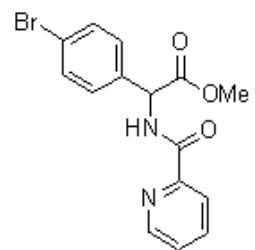
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



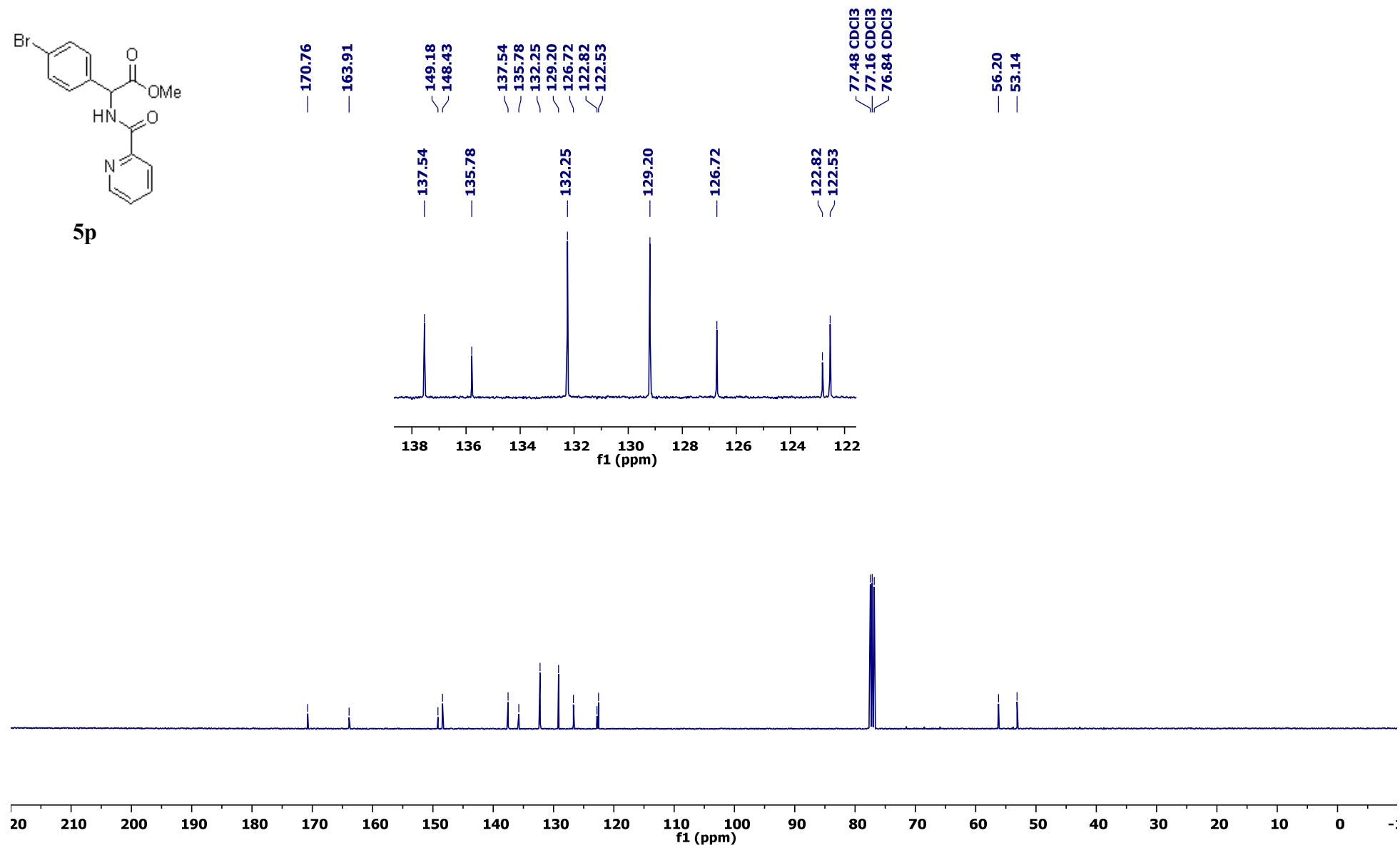
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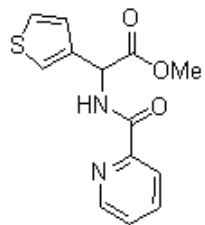
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



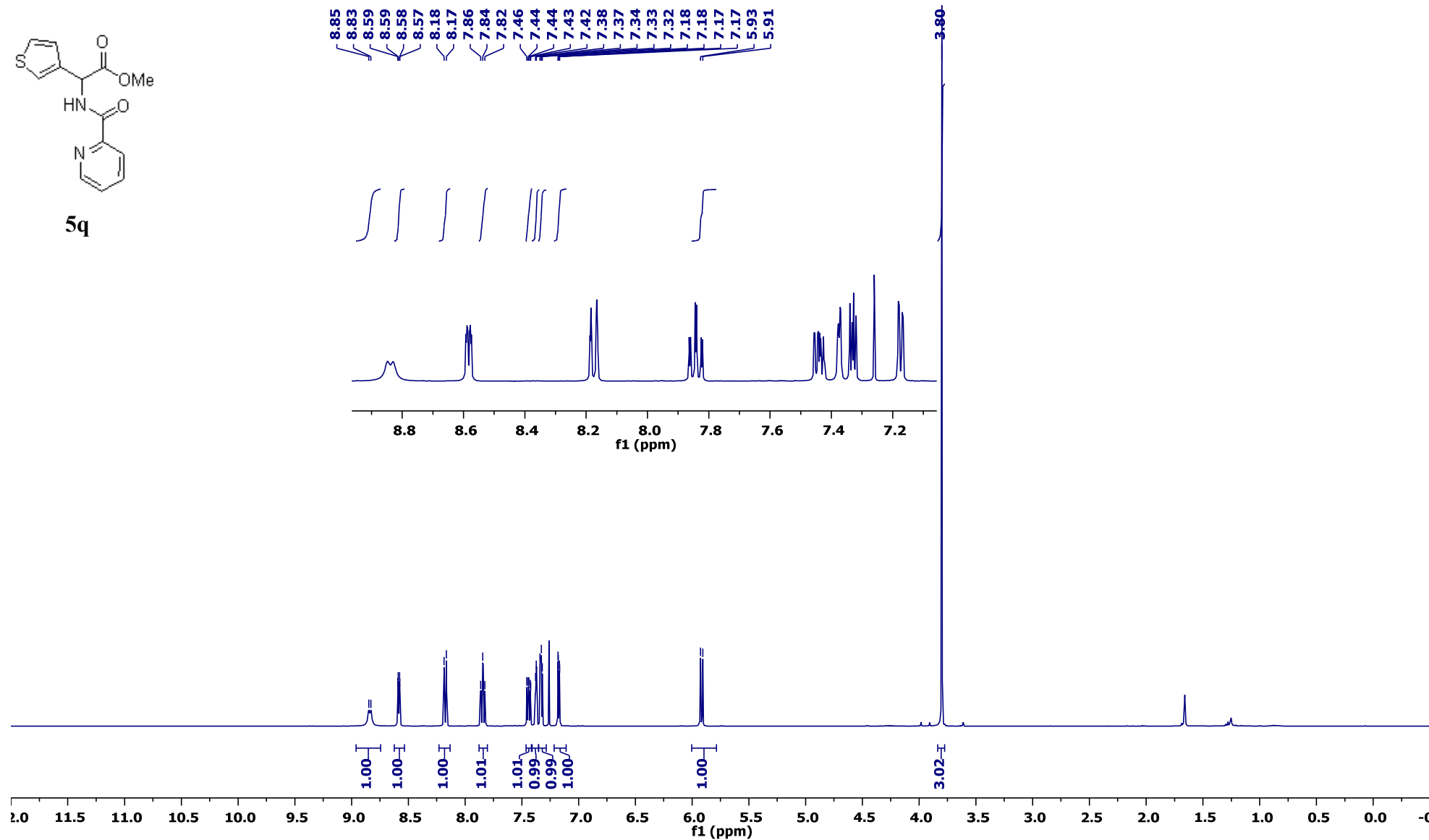
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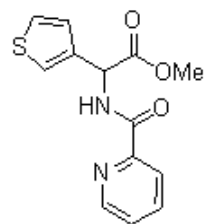
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



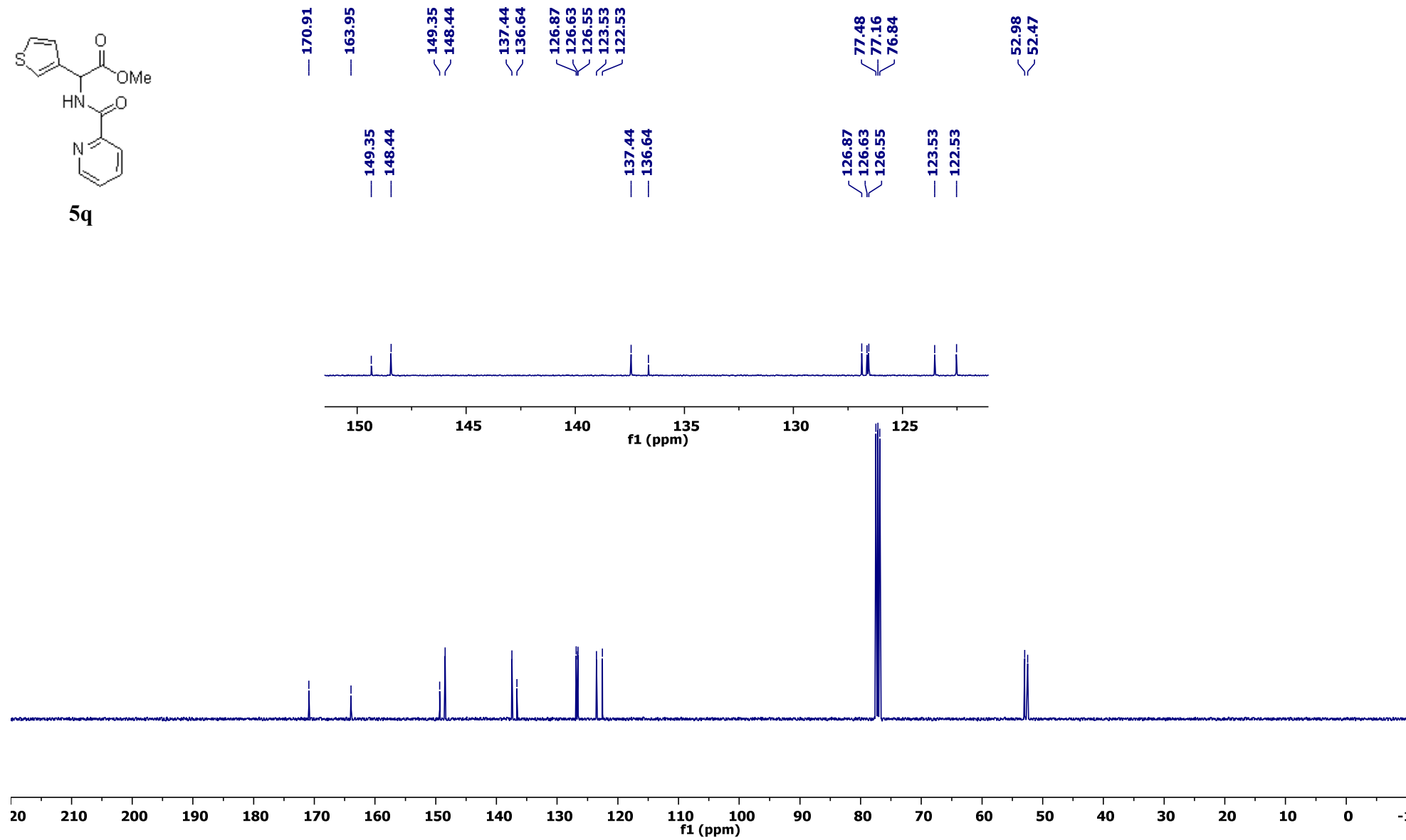
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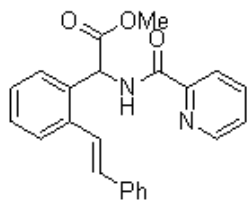
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



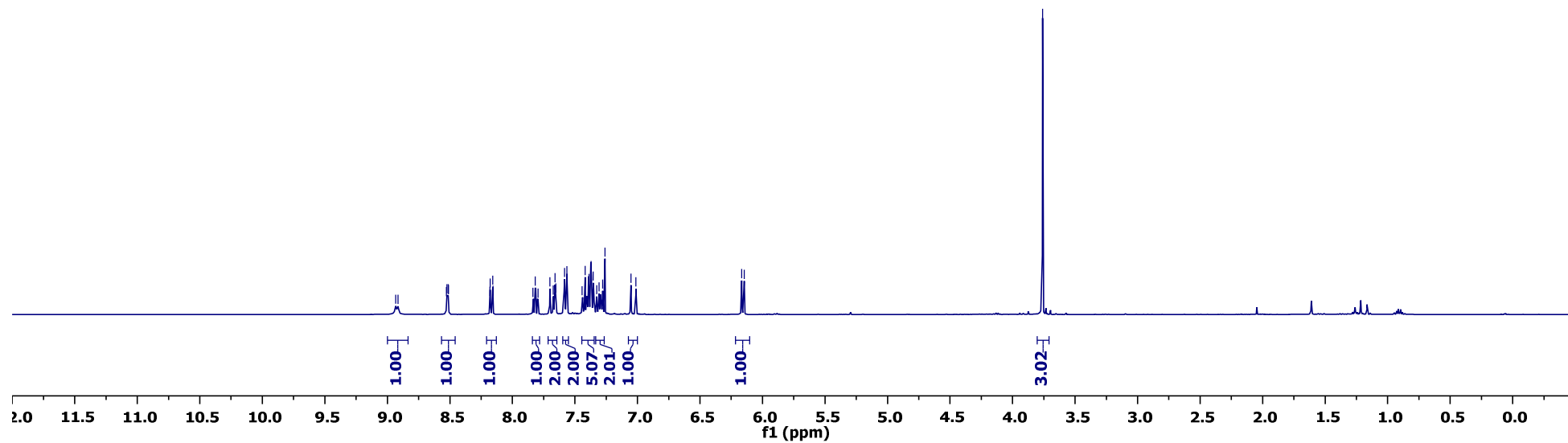
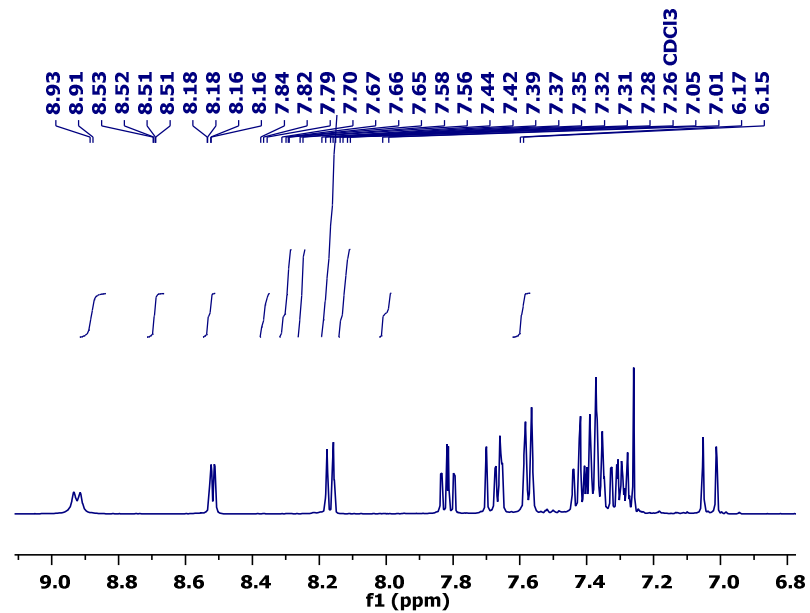
**5q**



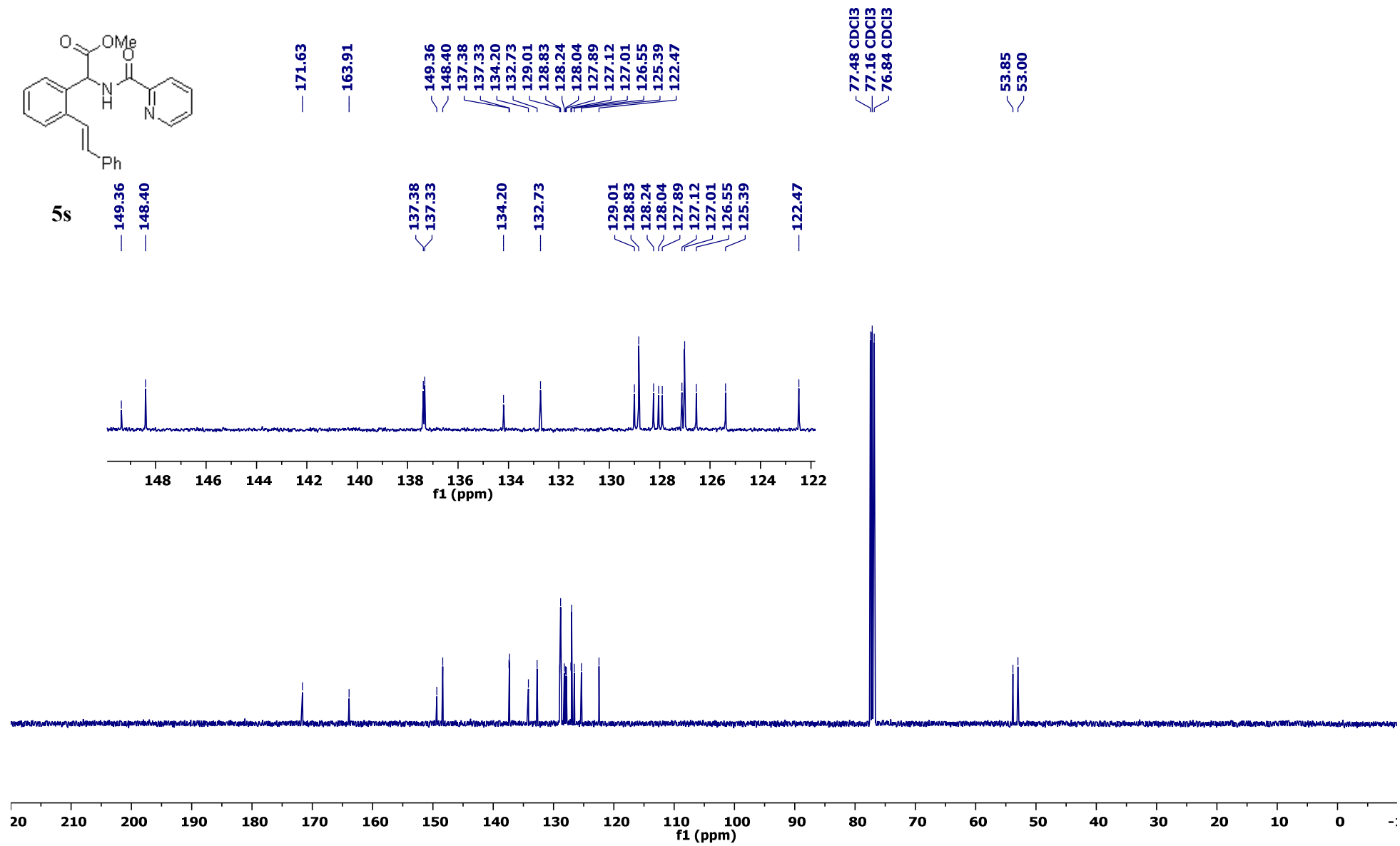
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



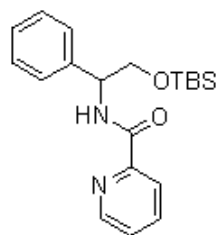
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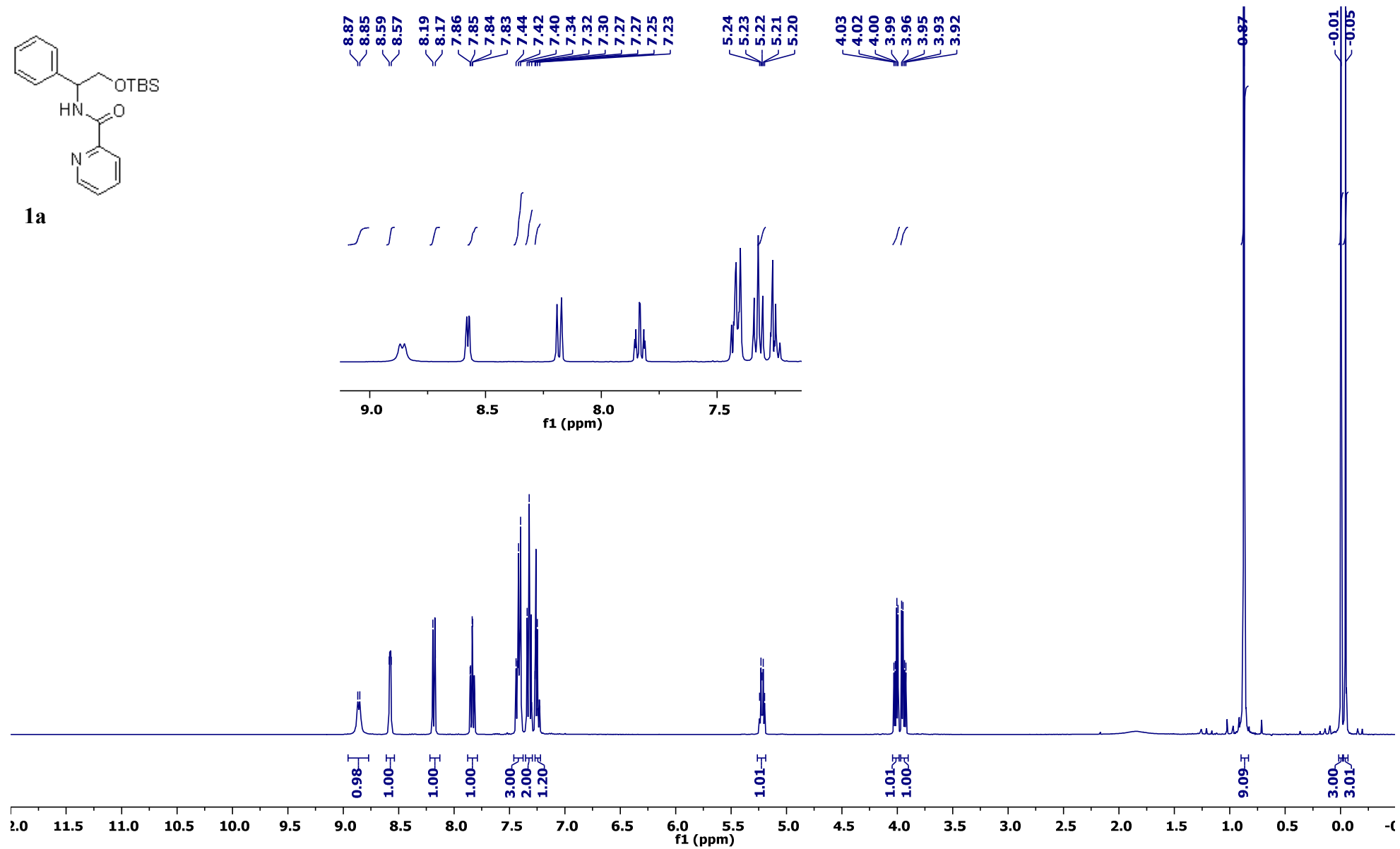
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )

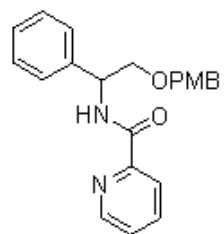


**1a**

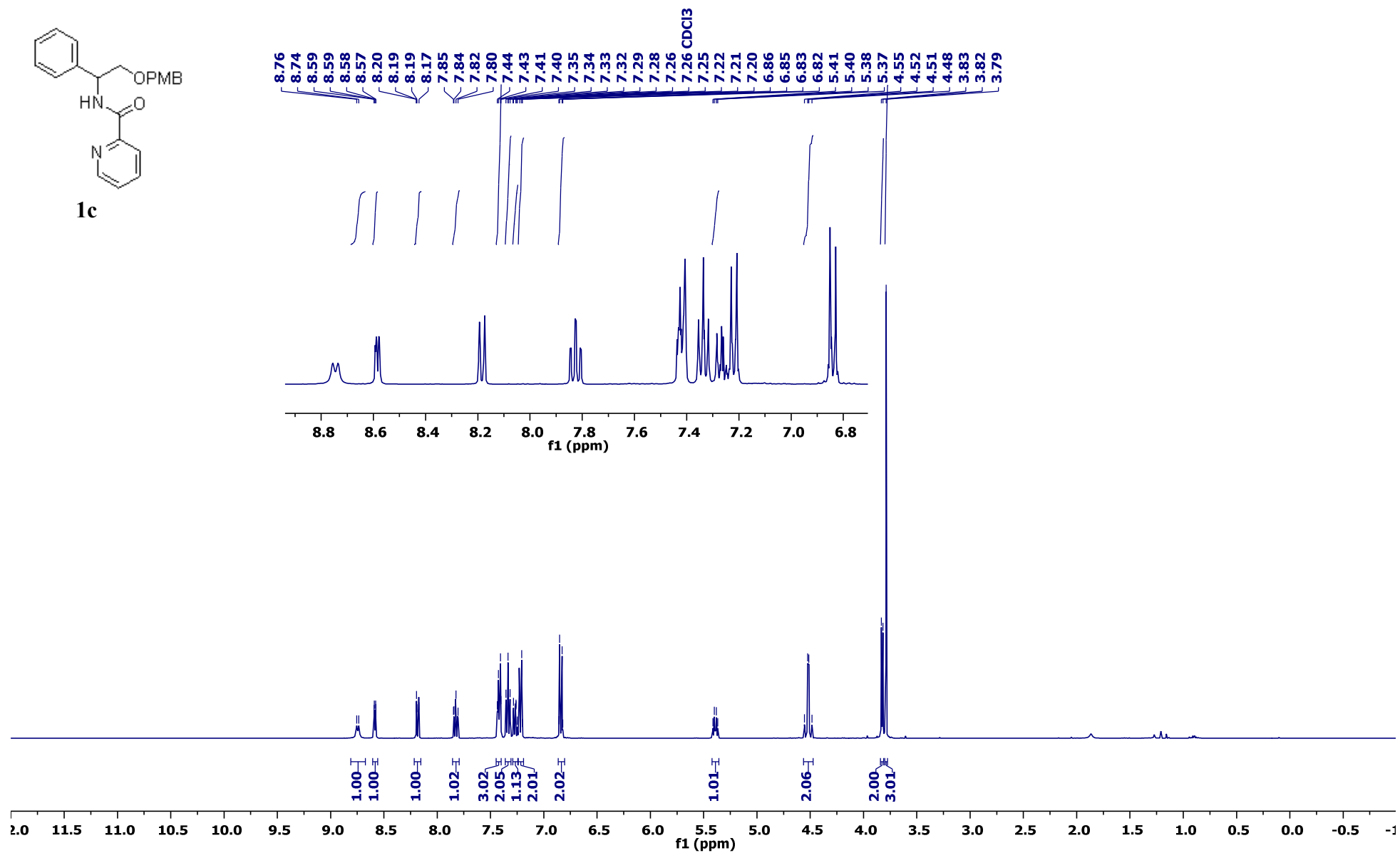




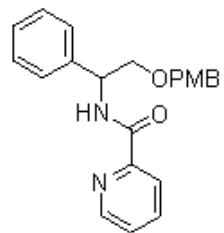
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



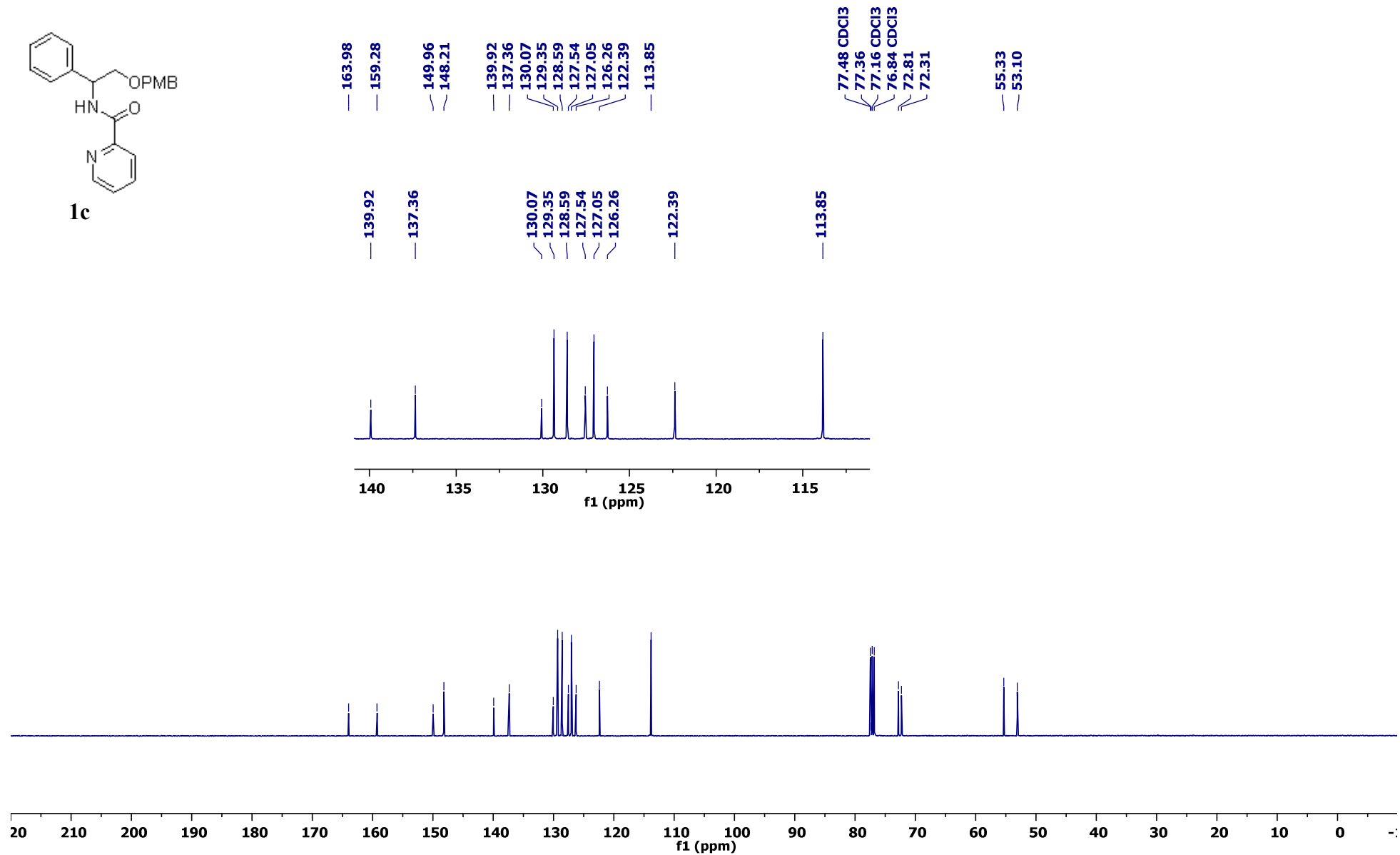
**1c**



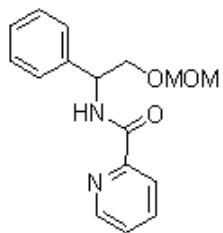
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



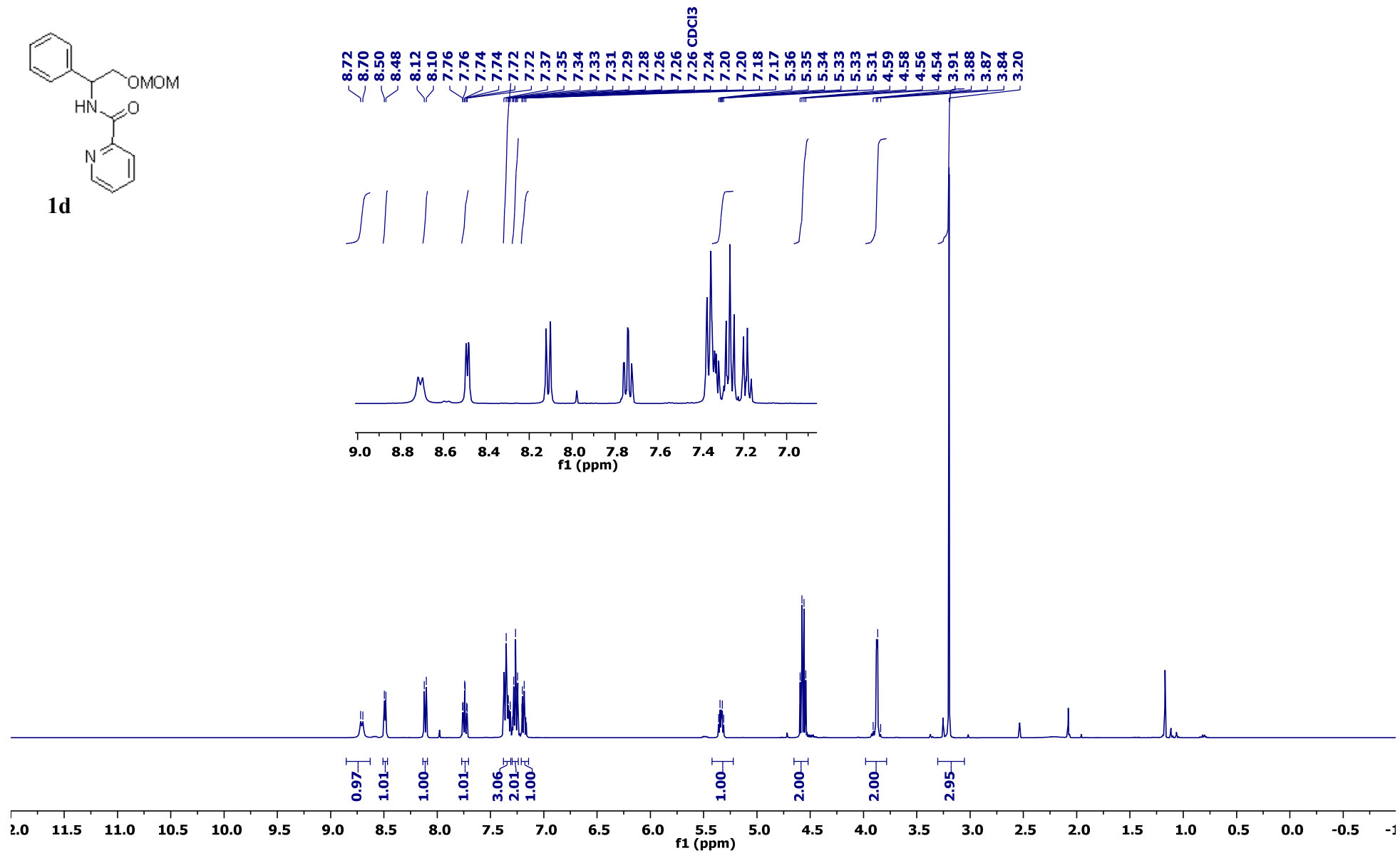
**1c**



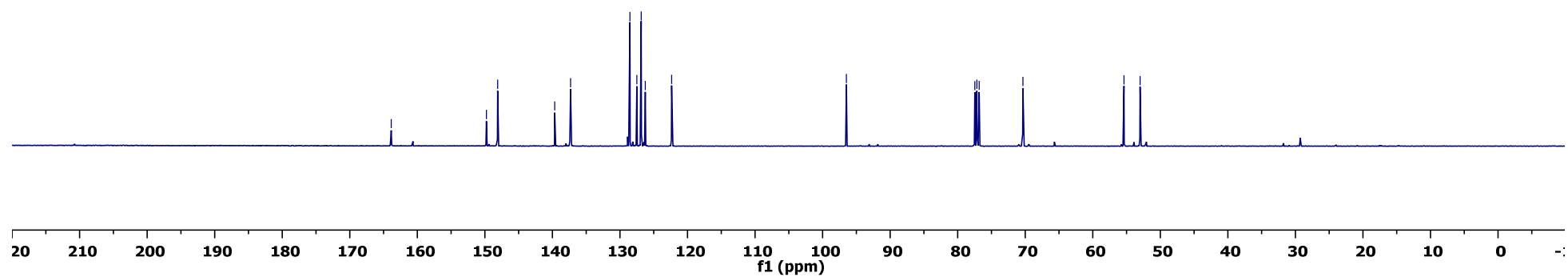
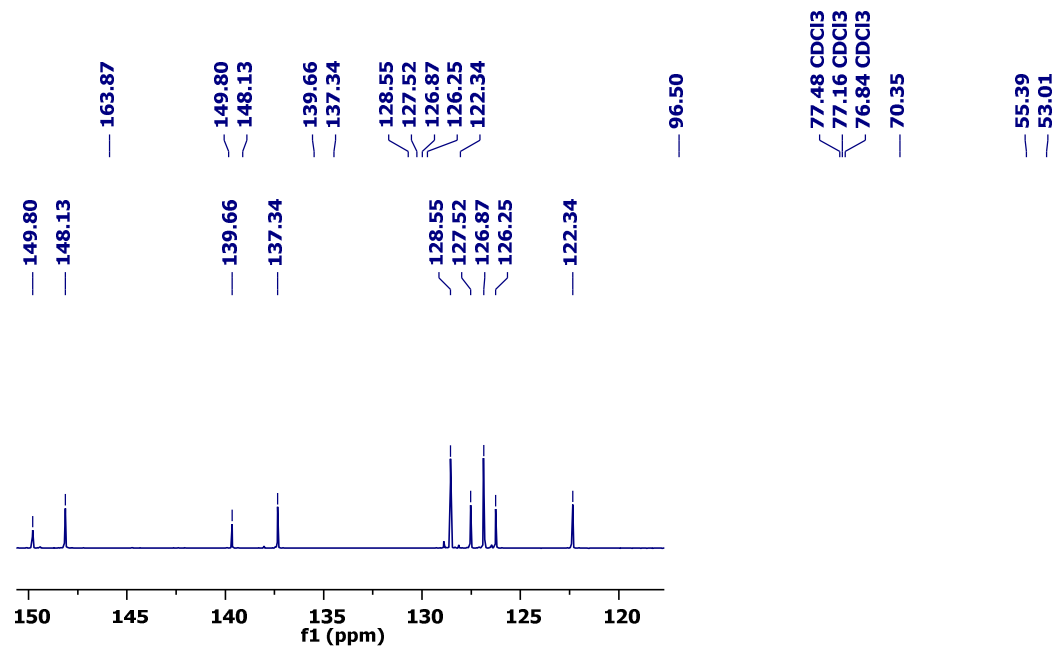
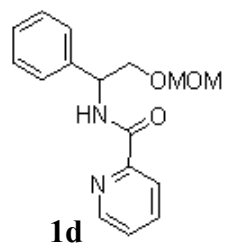
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



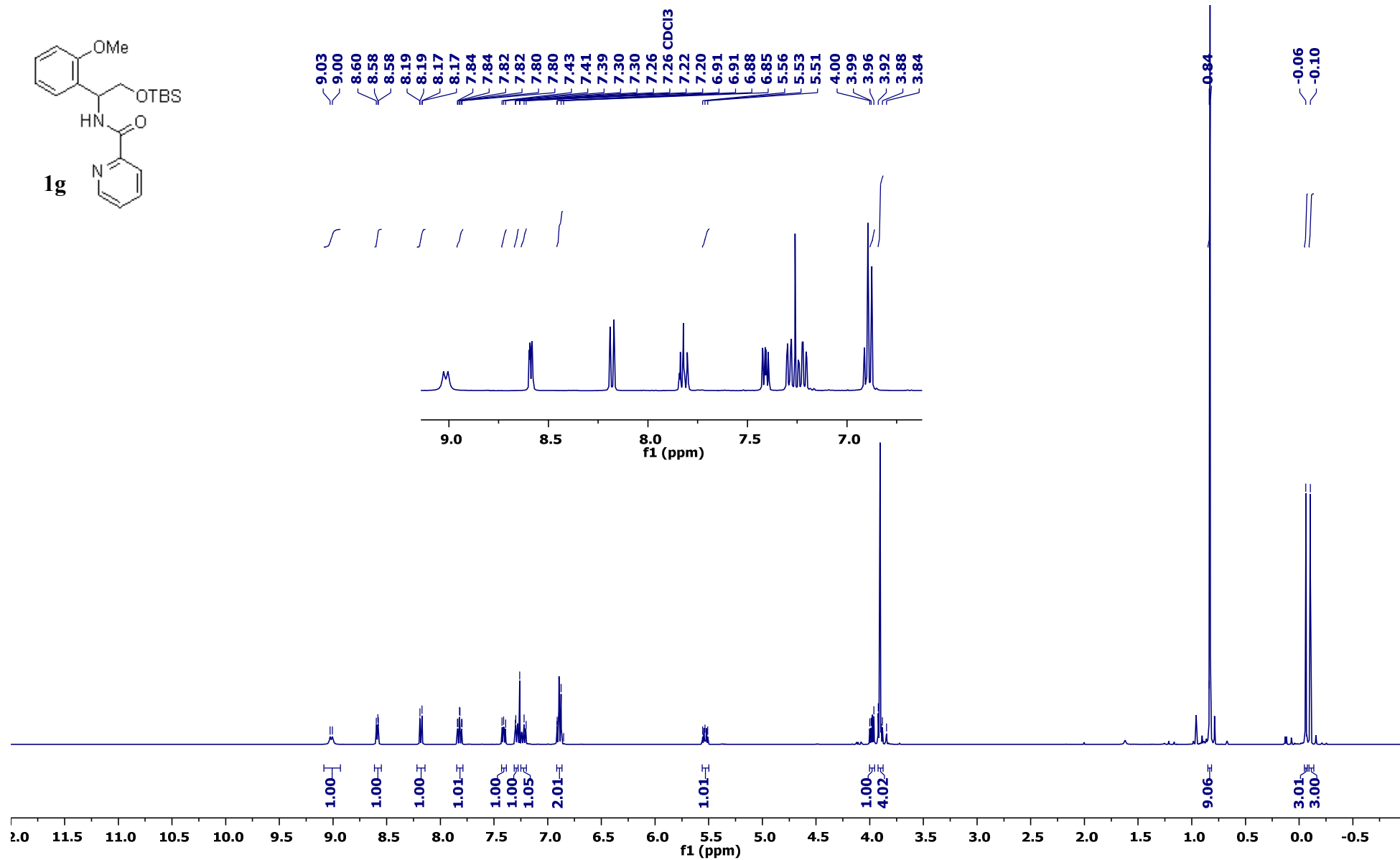
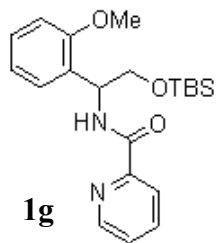
**1d**



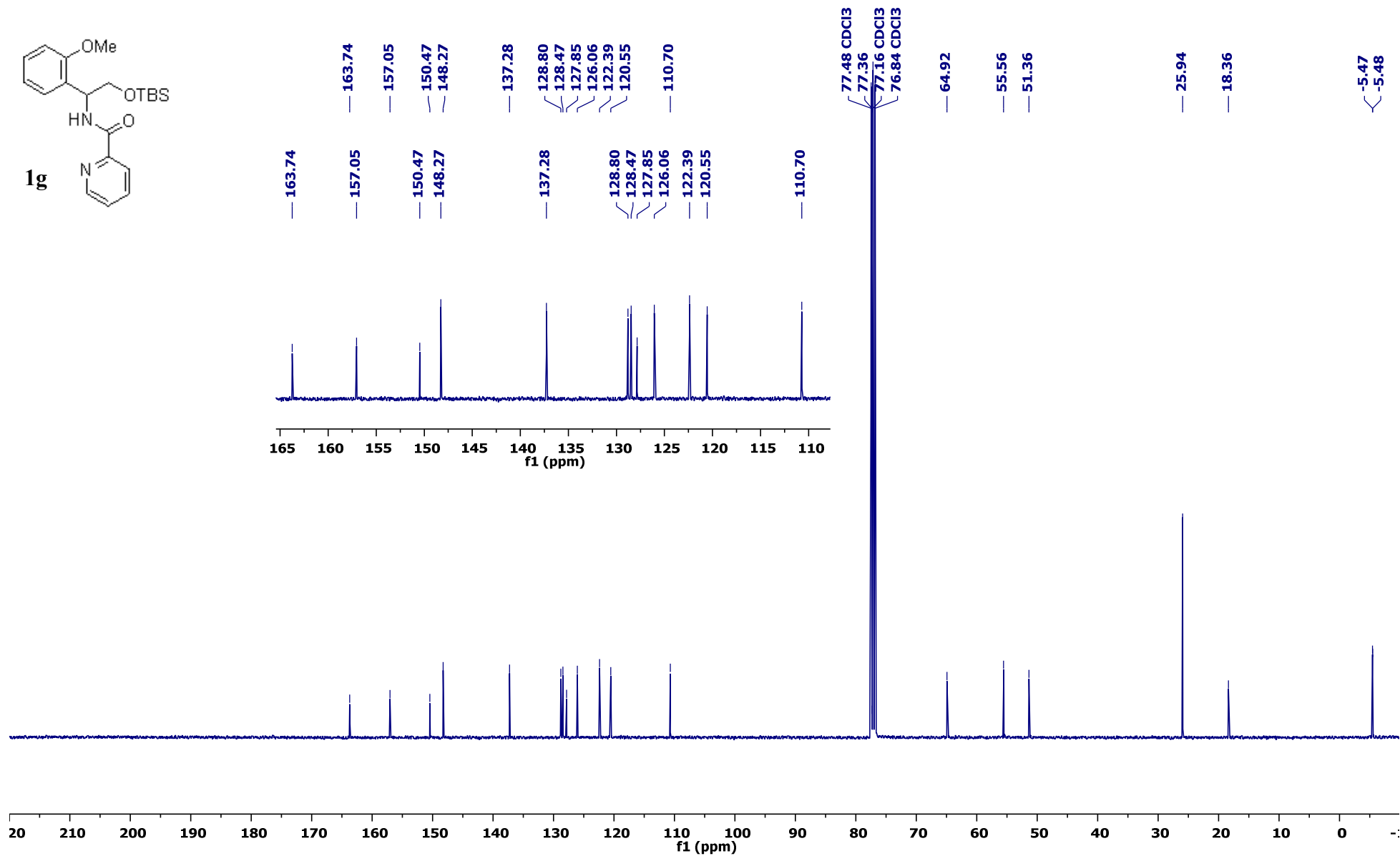
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



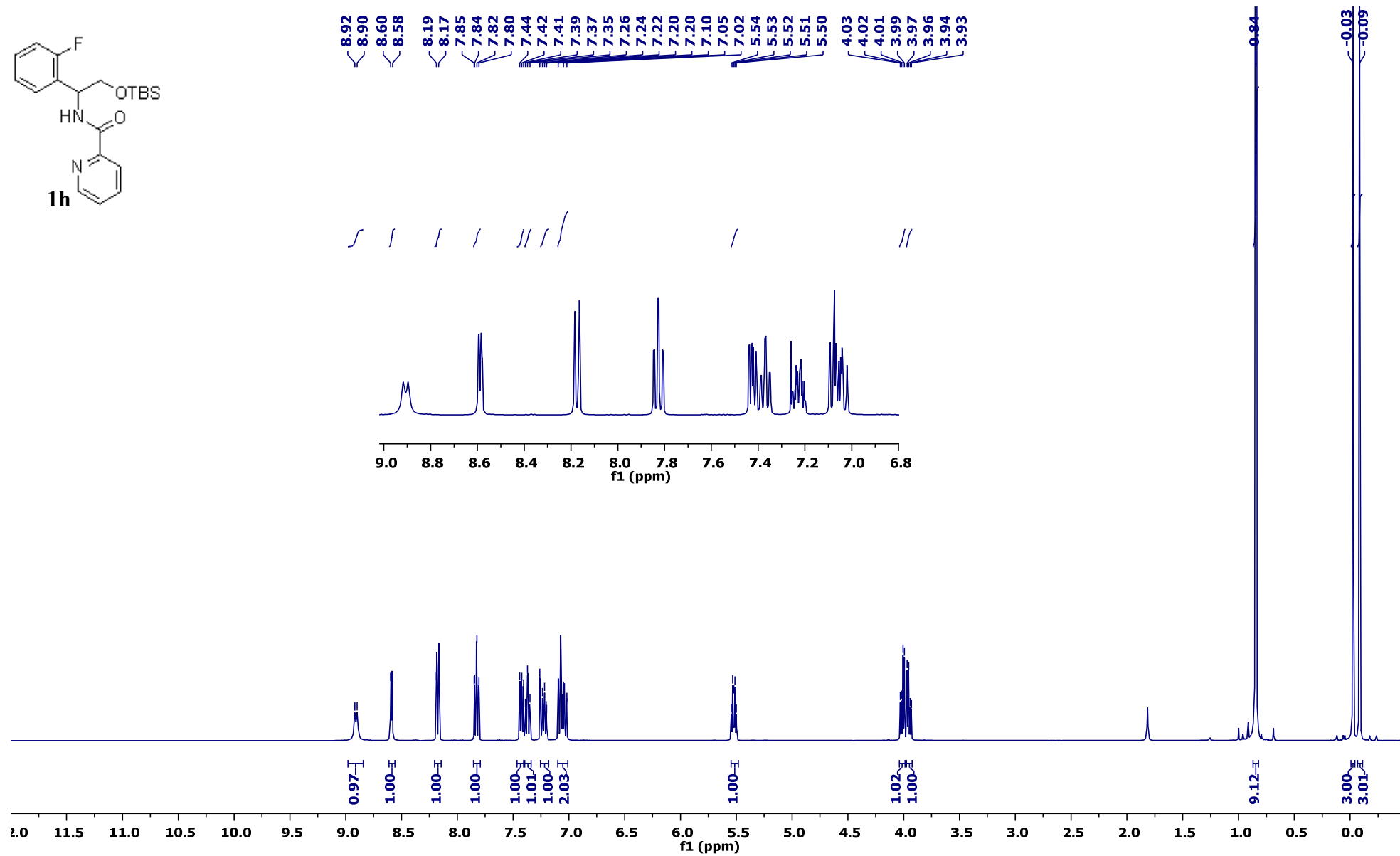
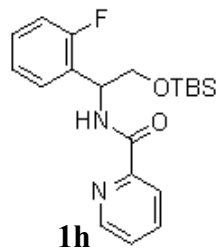
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



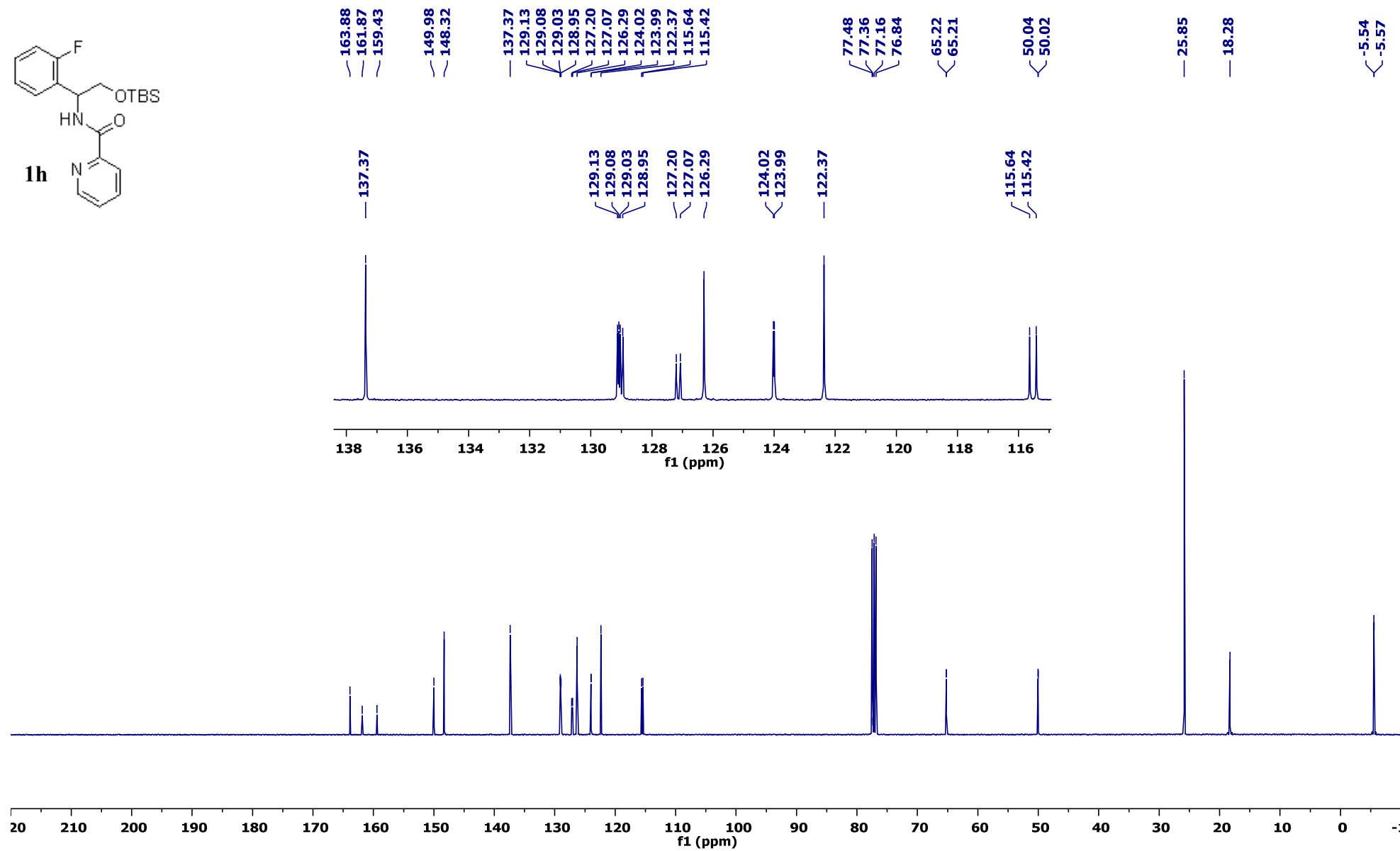
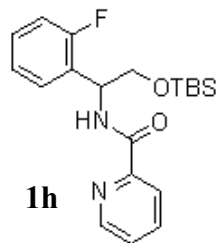
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



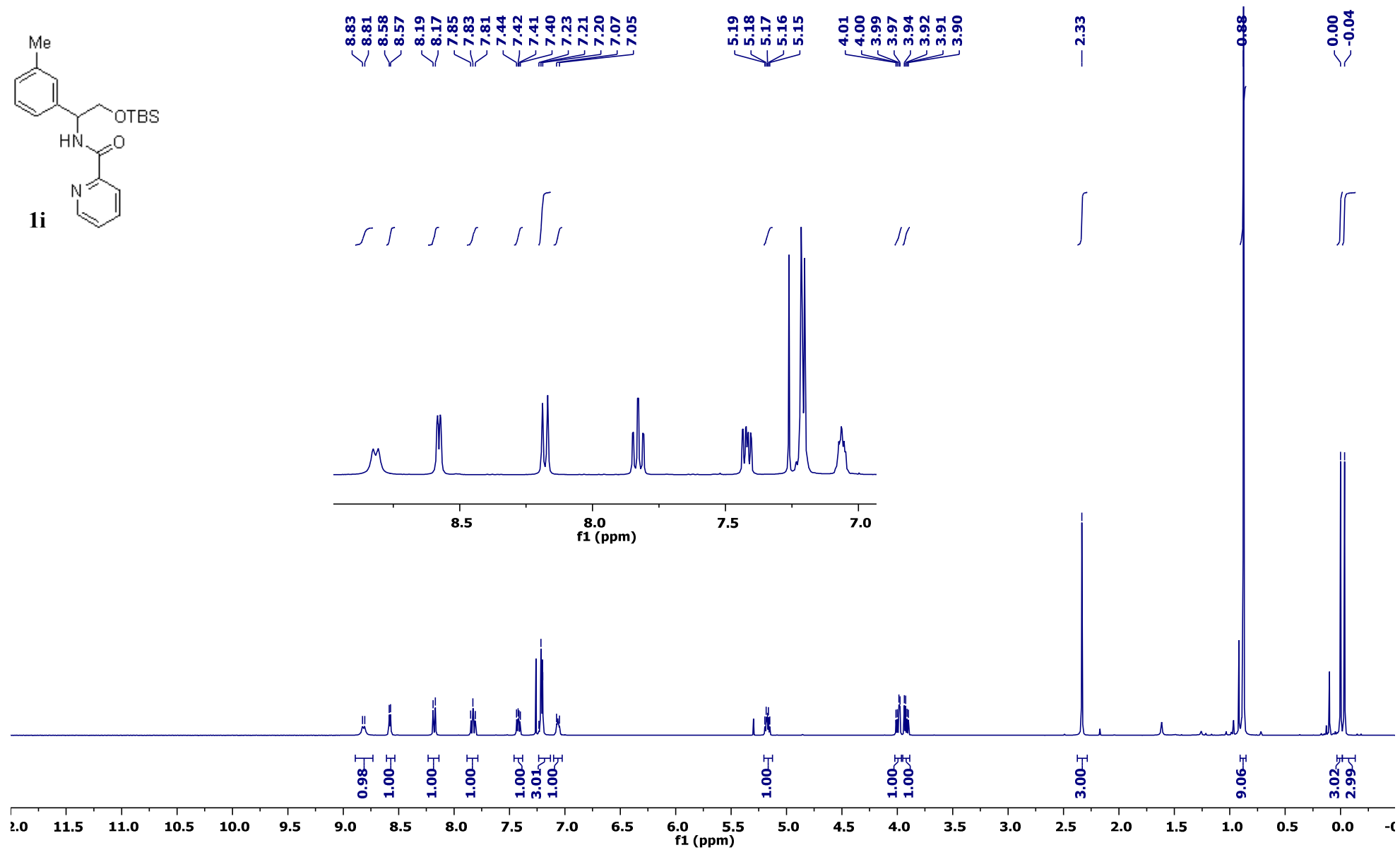
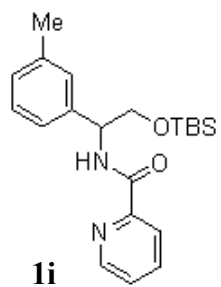
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



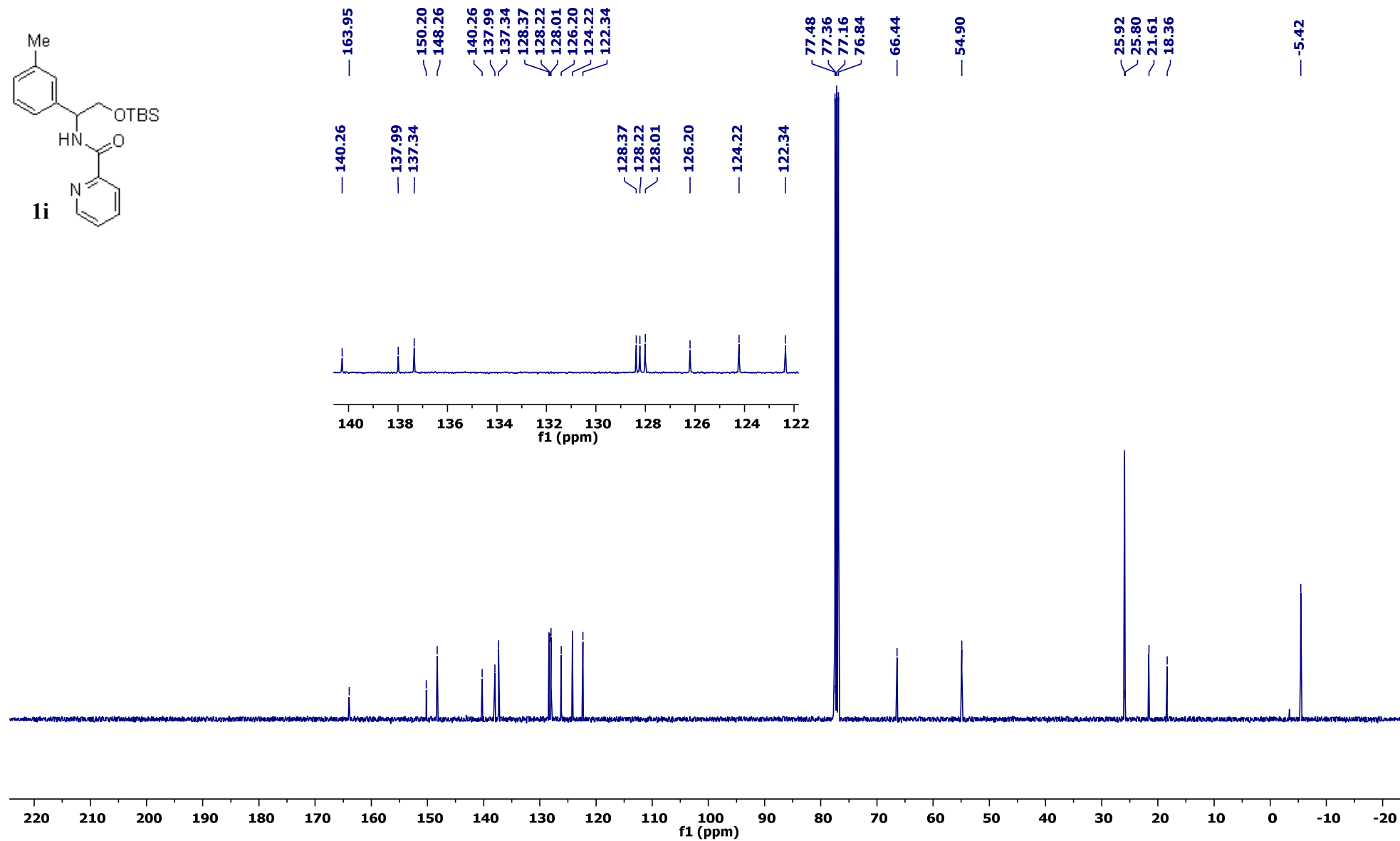
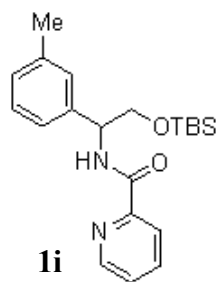
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



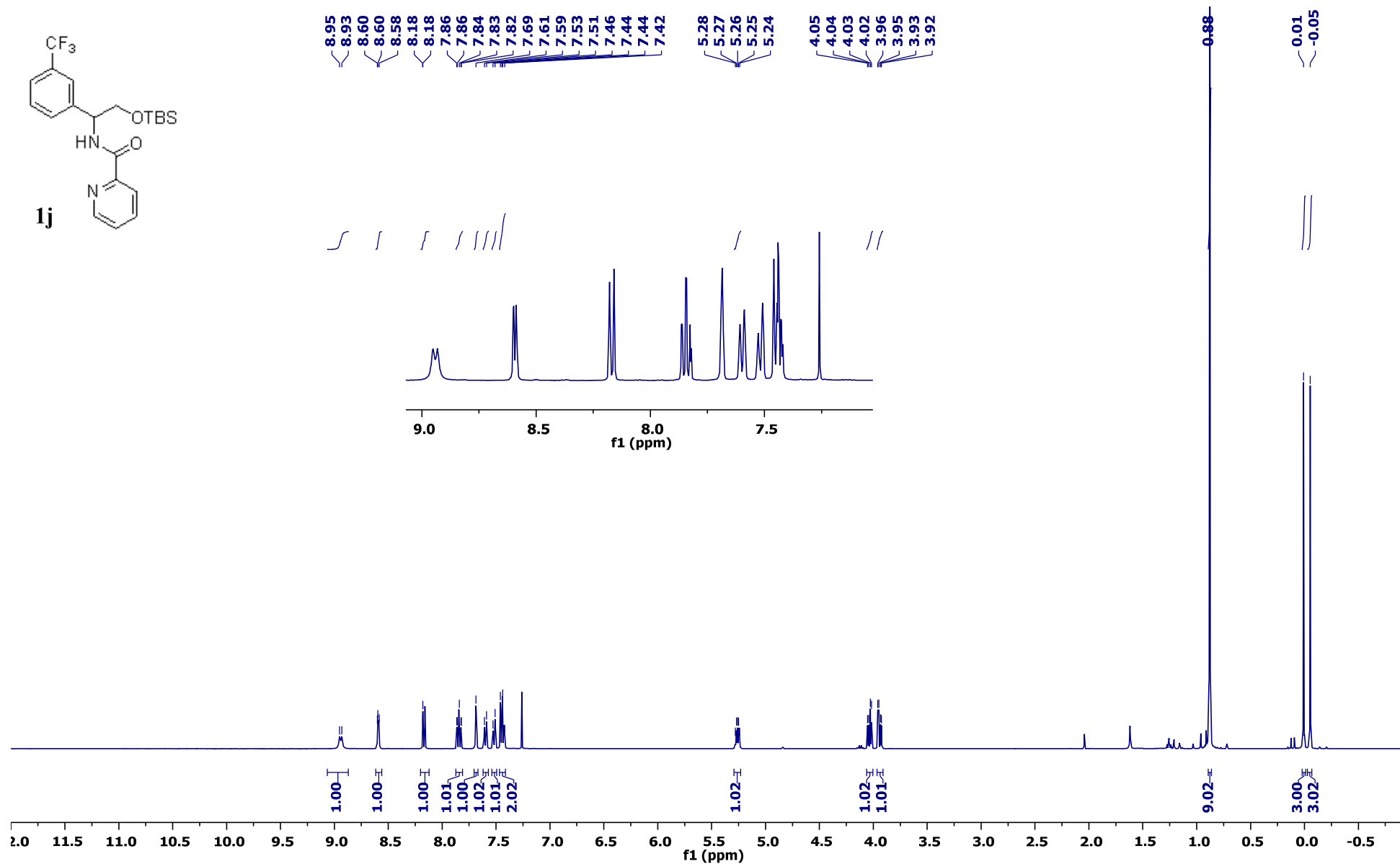
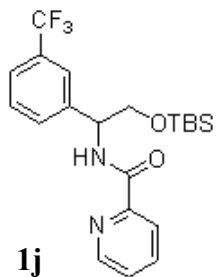
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



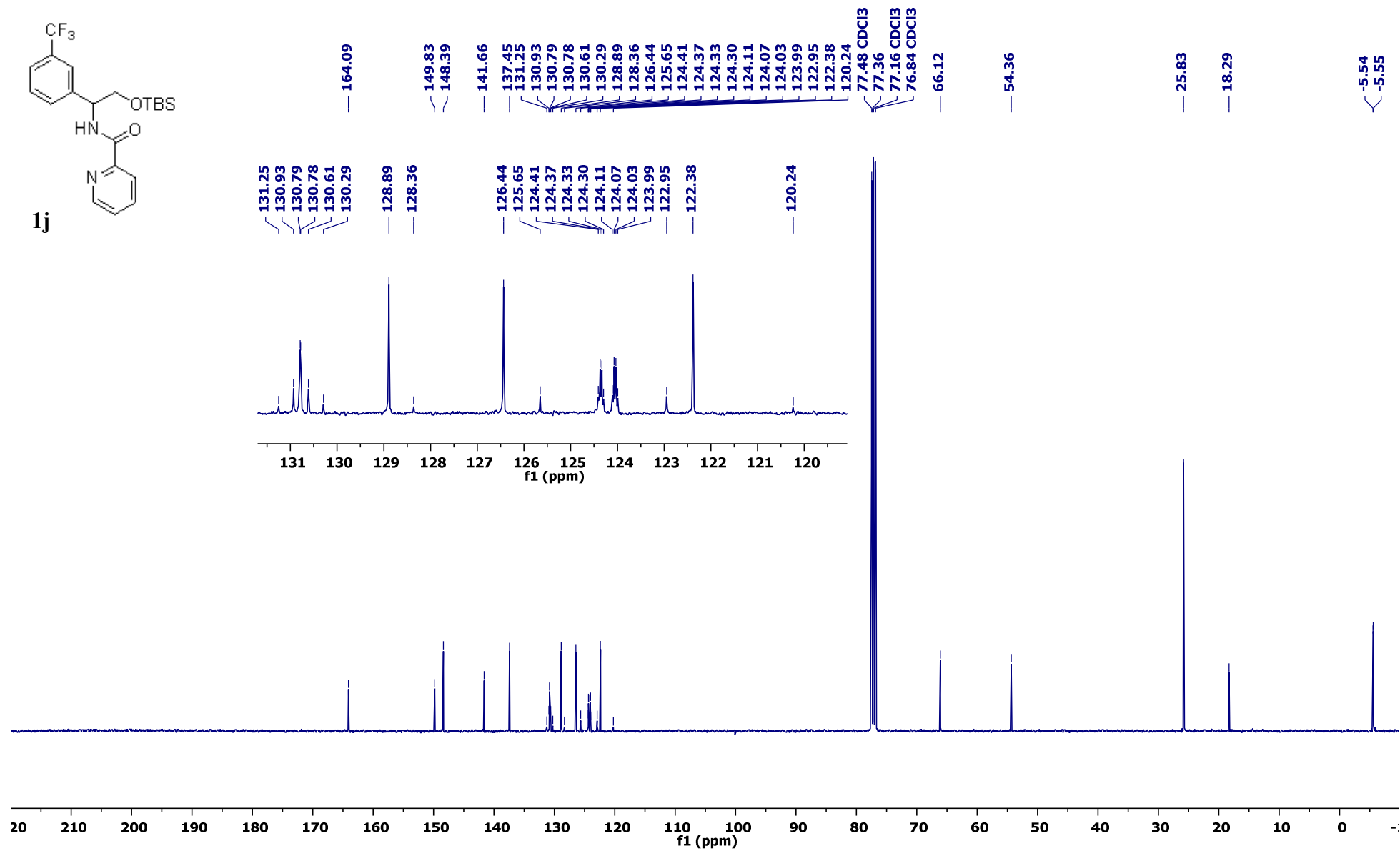
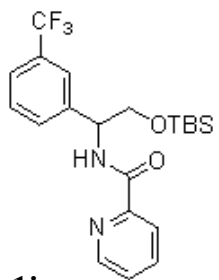
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



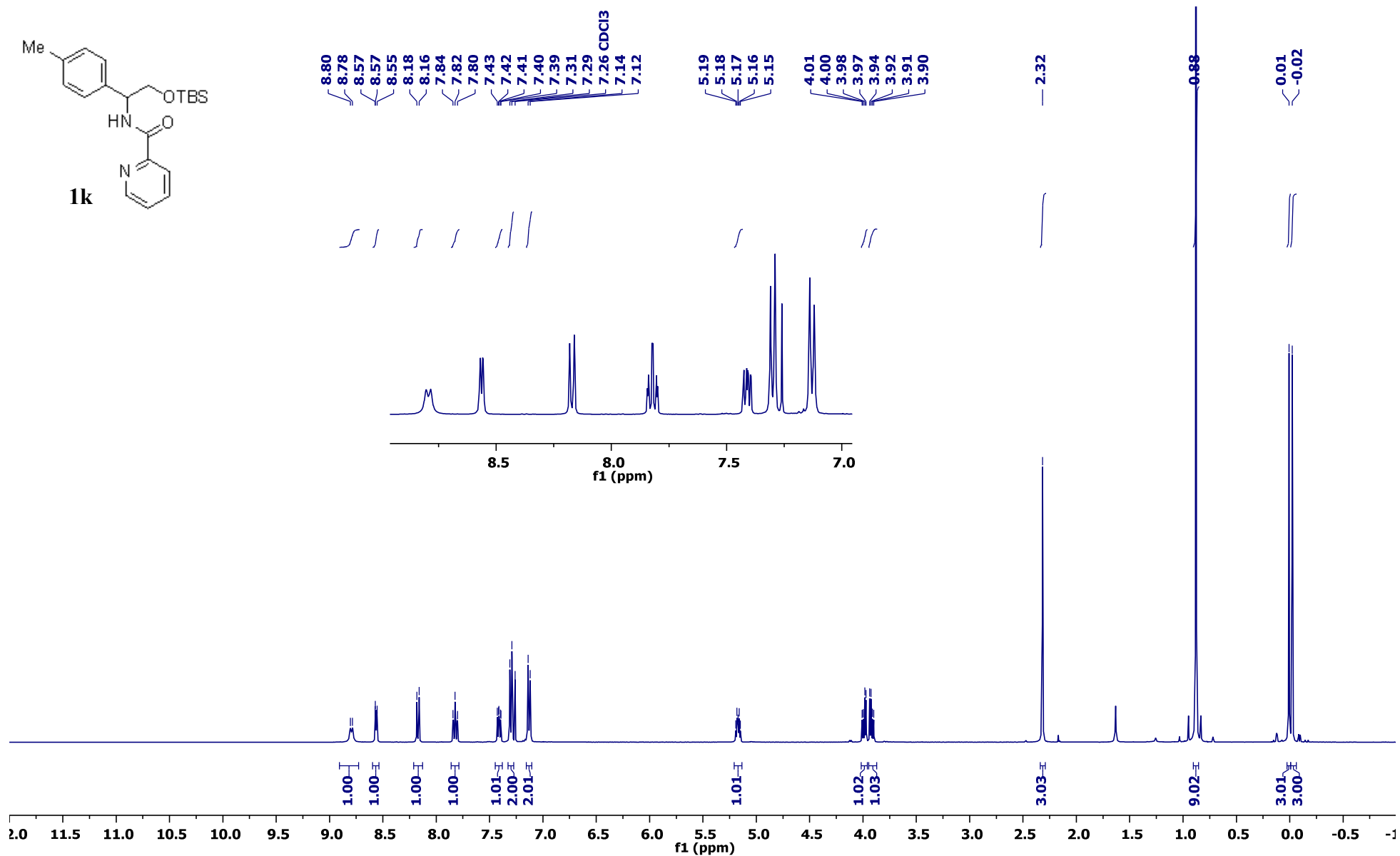
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



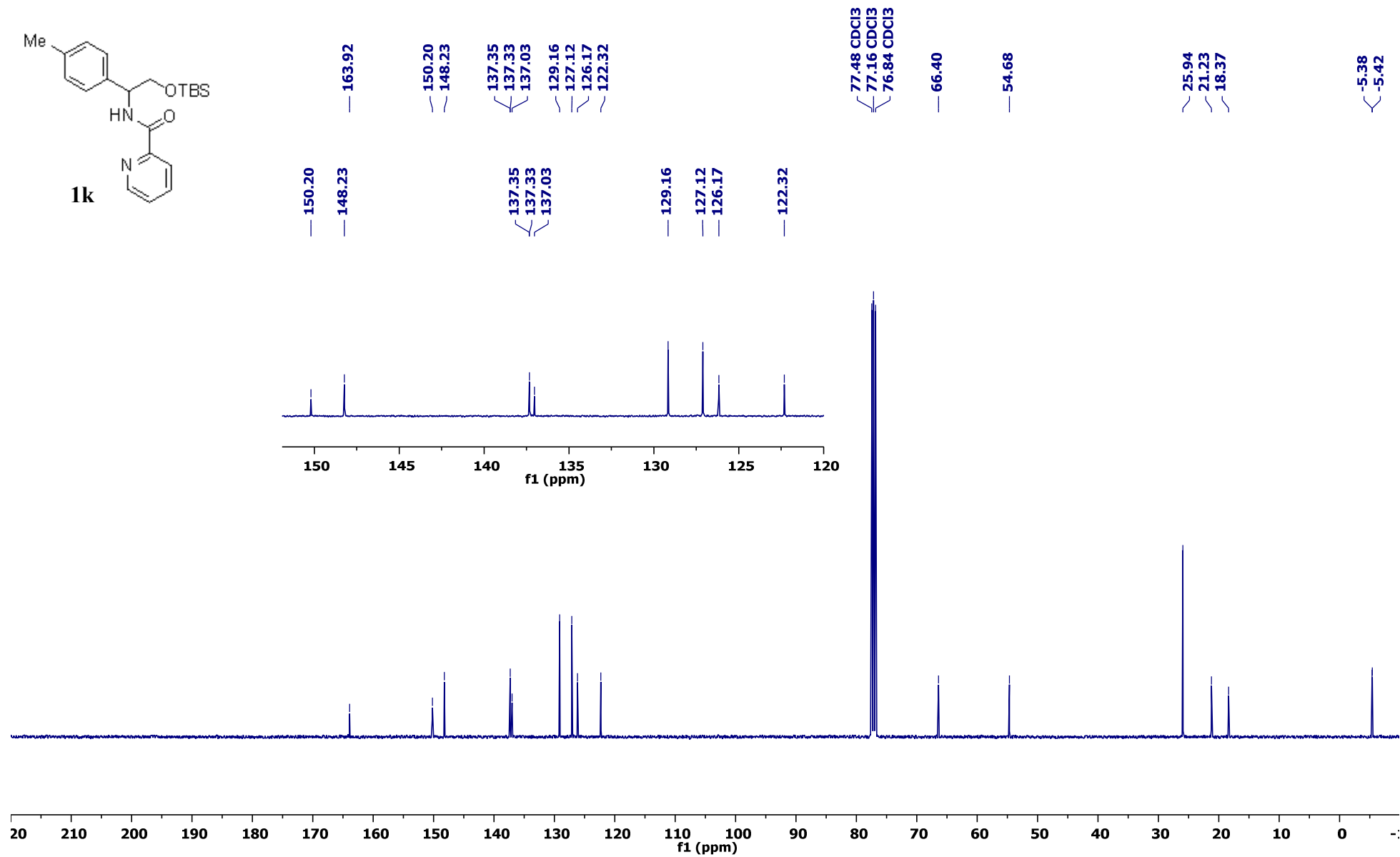
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



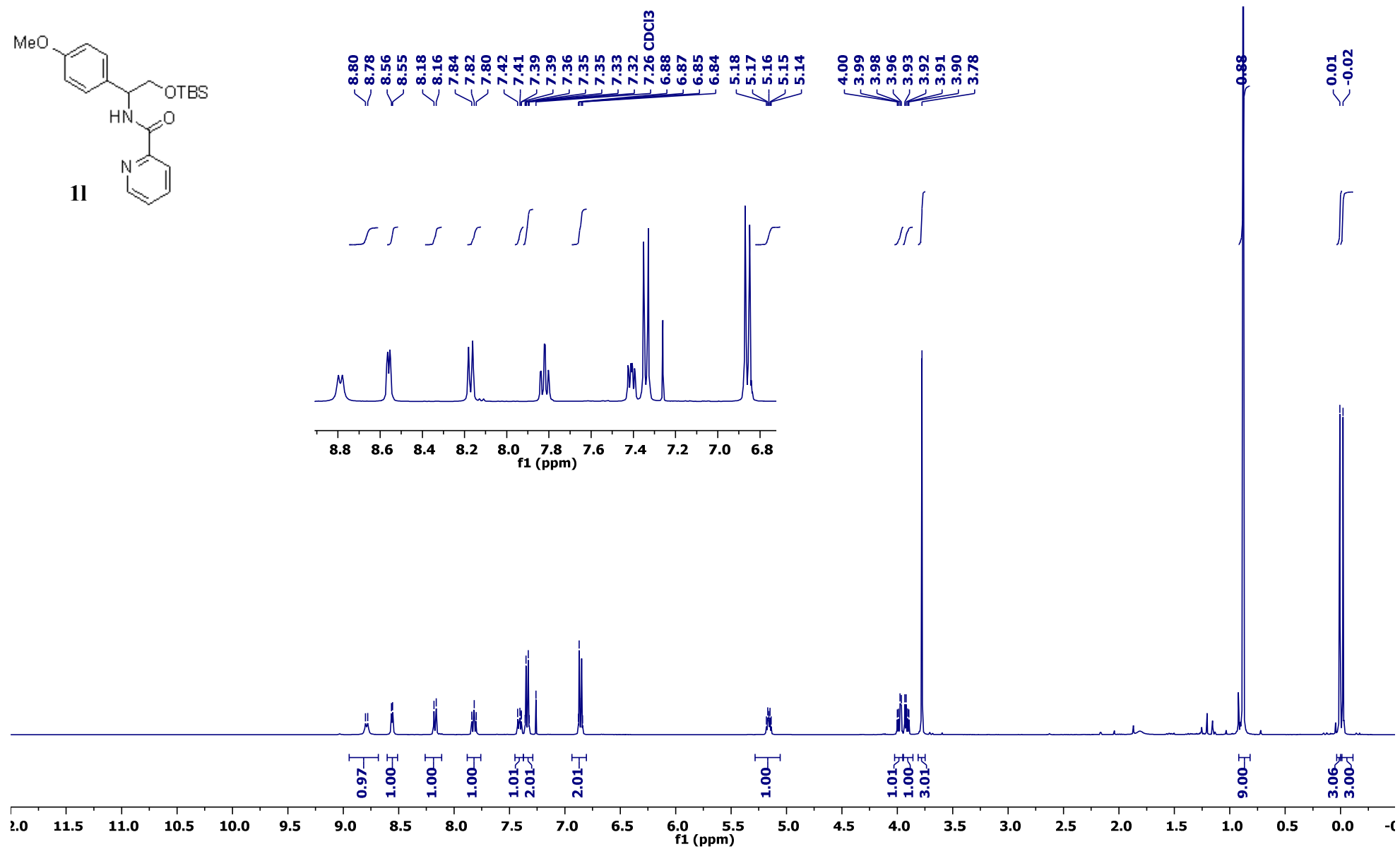
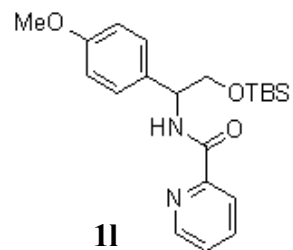
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



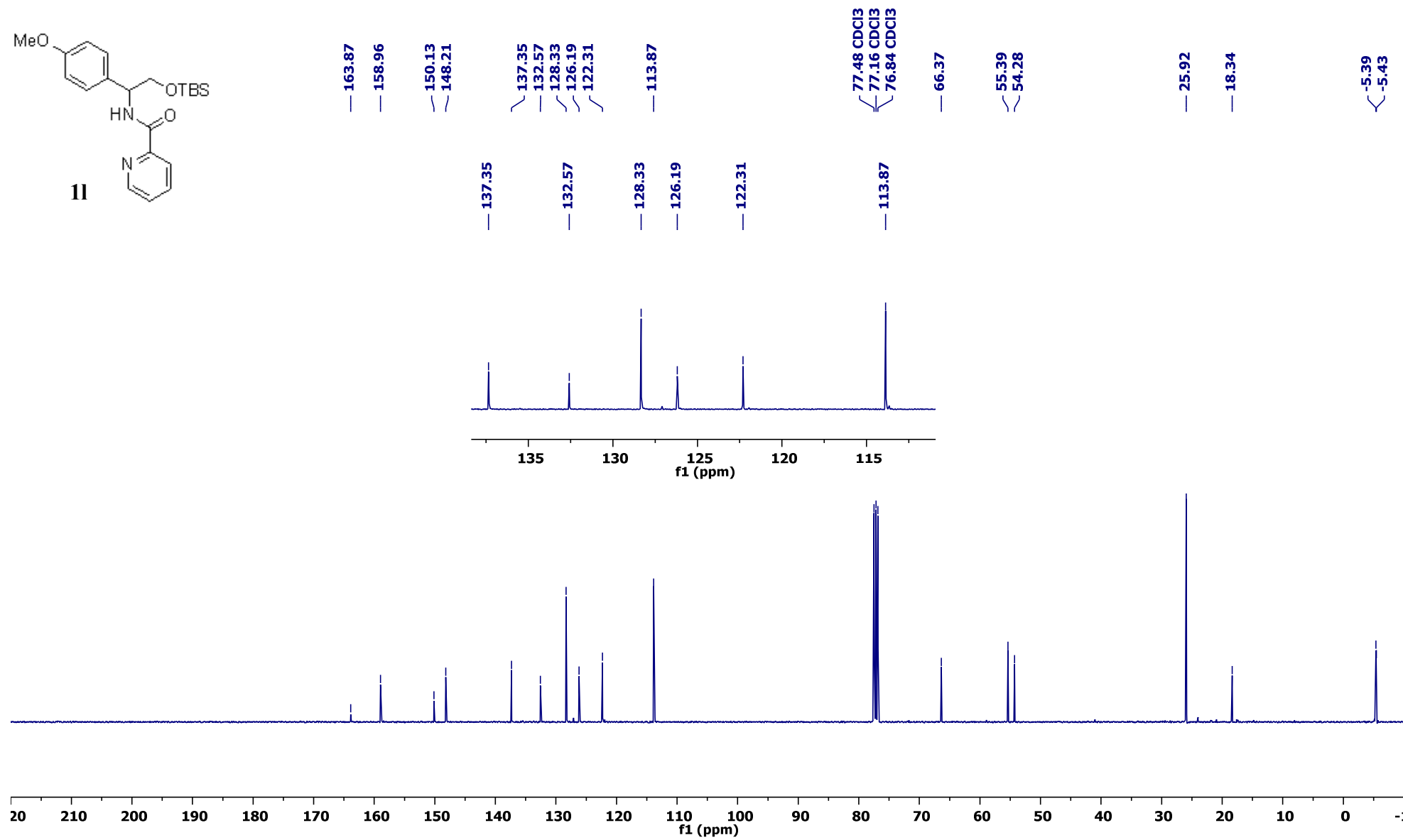
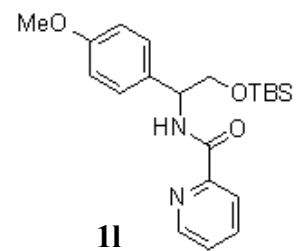
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

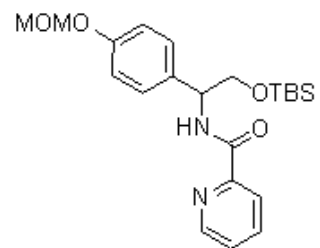


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

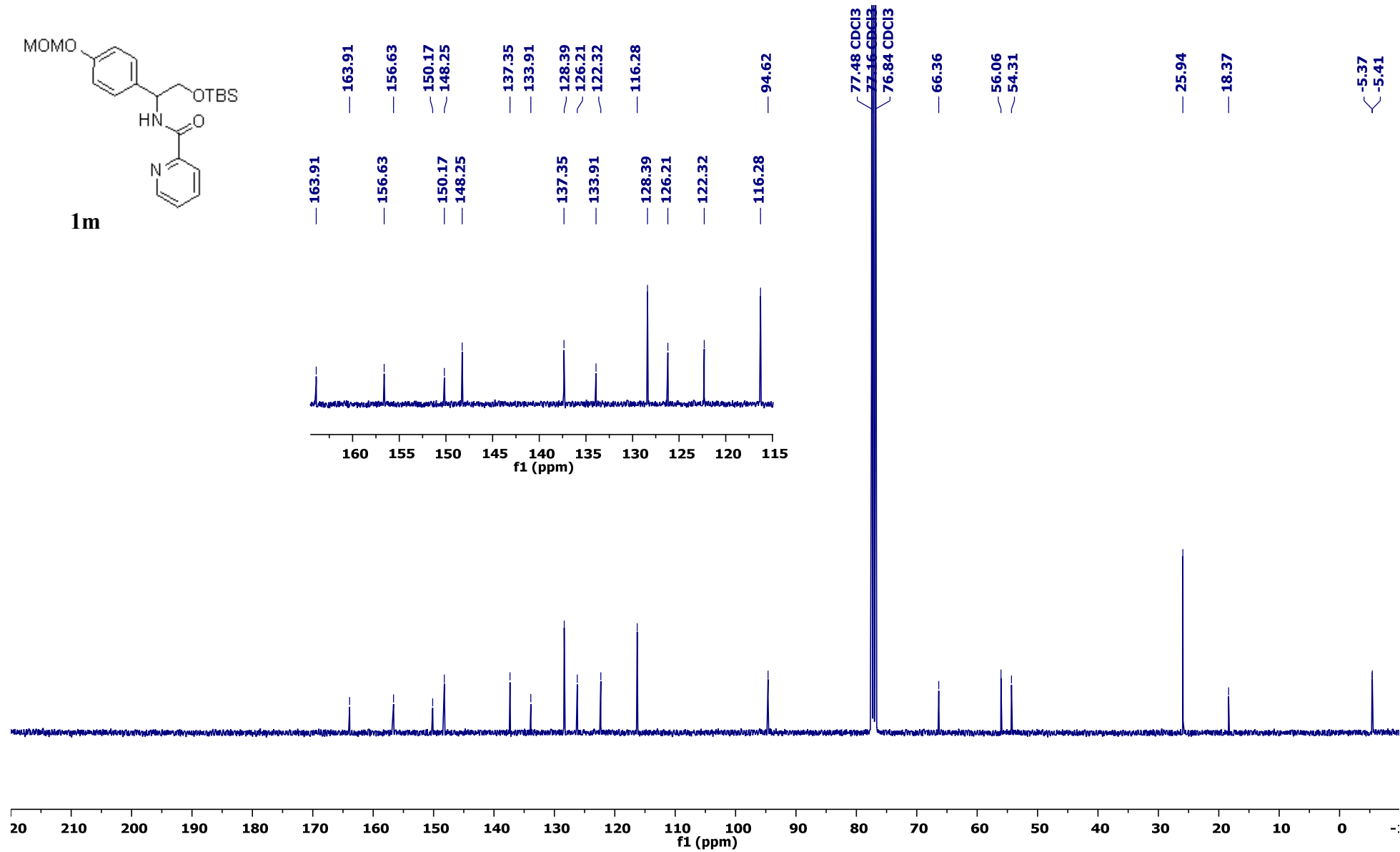




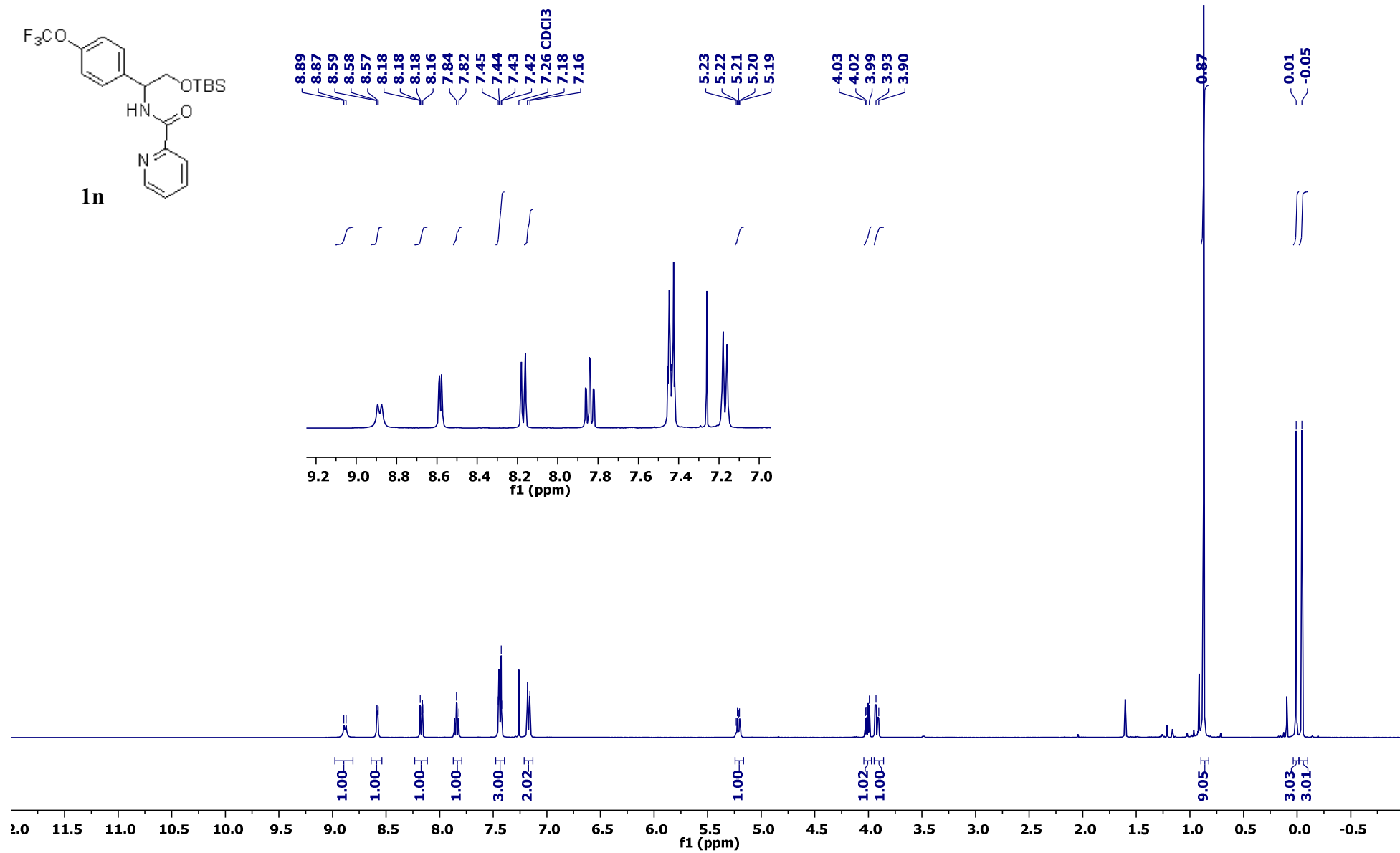
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



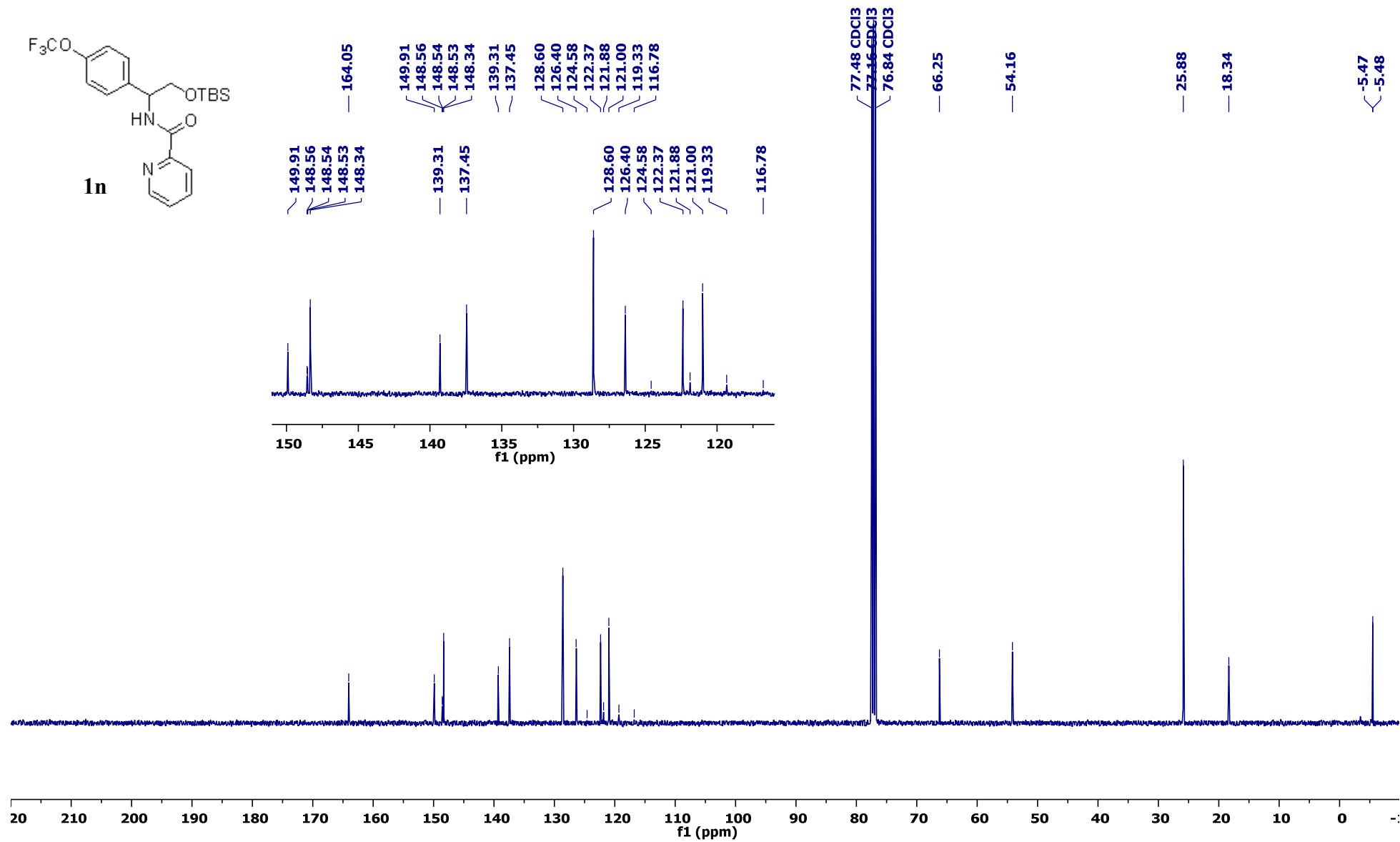
**1m**



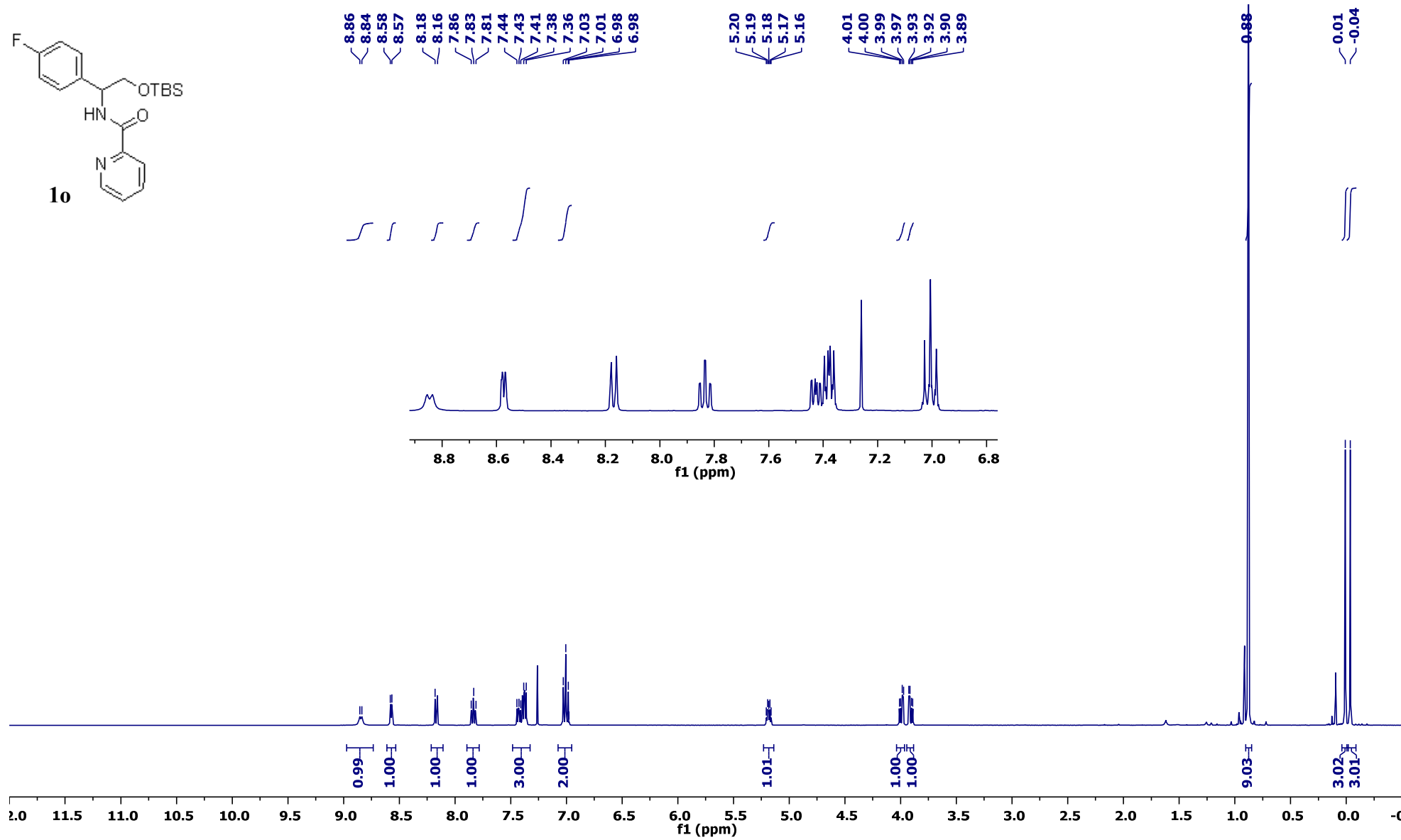
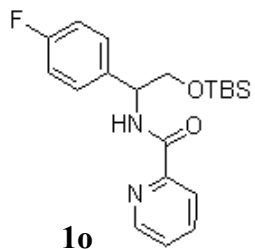
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



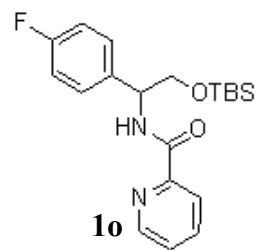
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



163.97  
163.45  
161.02  
150.01  
148.31  
137.41  
136.30  
136.27  
128.84  
128.76  
126.32  
122.35  
115.37  
115.16

163.97  
163.45  
161.02

150.01  
148.31

137.41  
136.30  
136.27

128.84  
128.76  
126.32

122.35

115.37  
115.16

77.48  
77.16  
76.84

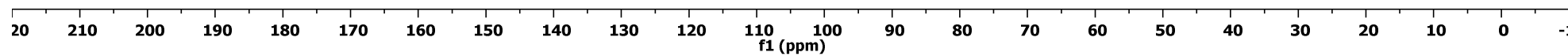
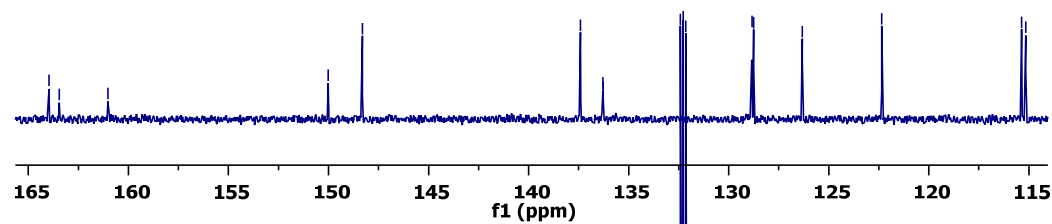
66.32

54.15

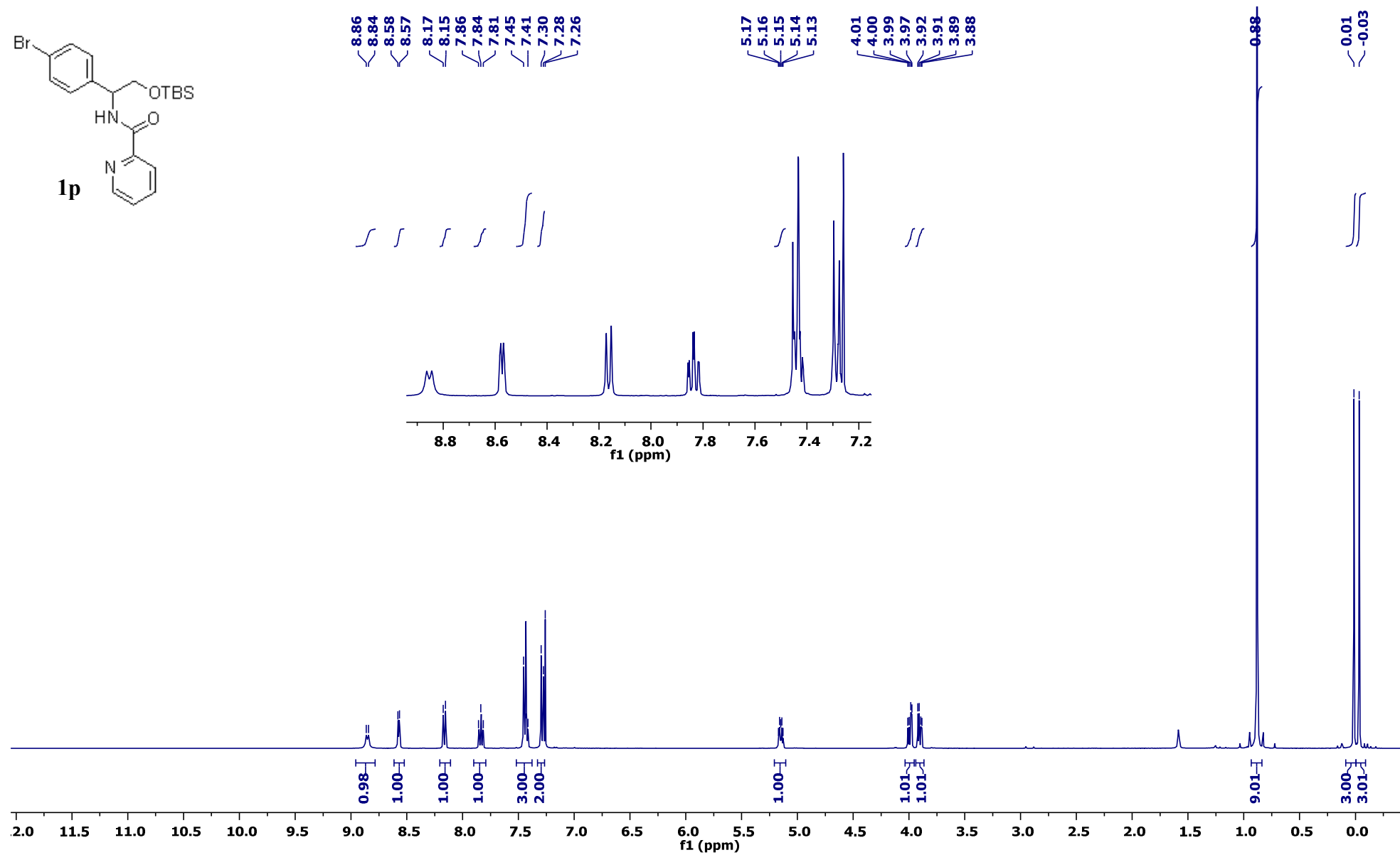
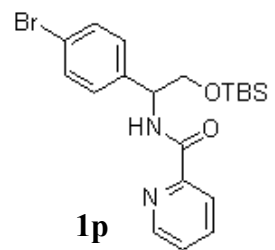
25.90  
25.80

18.35

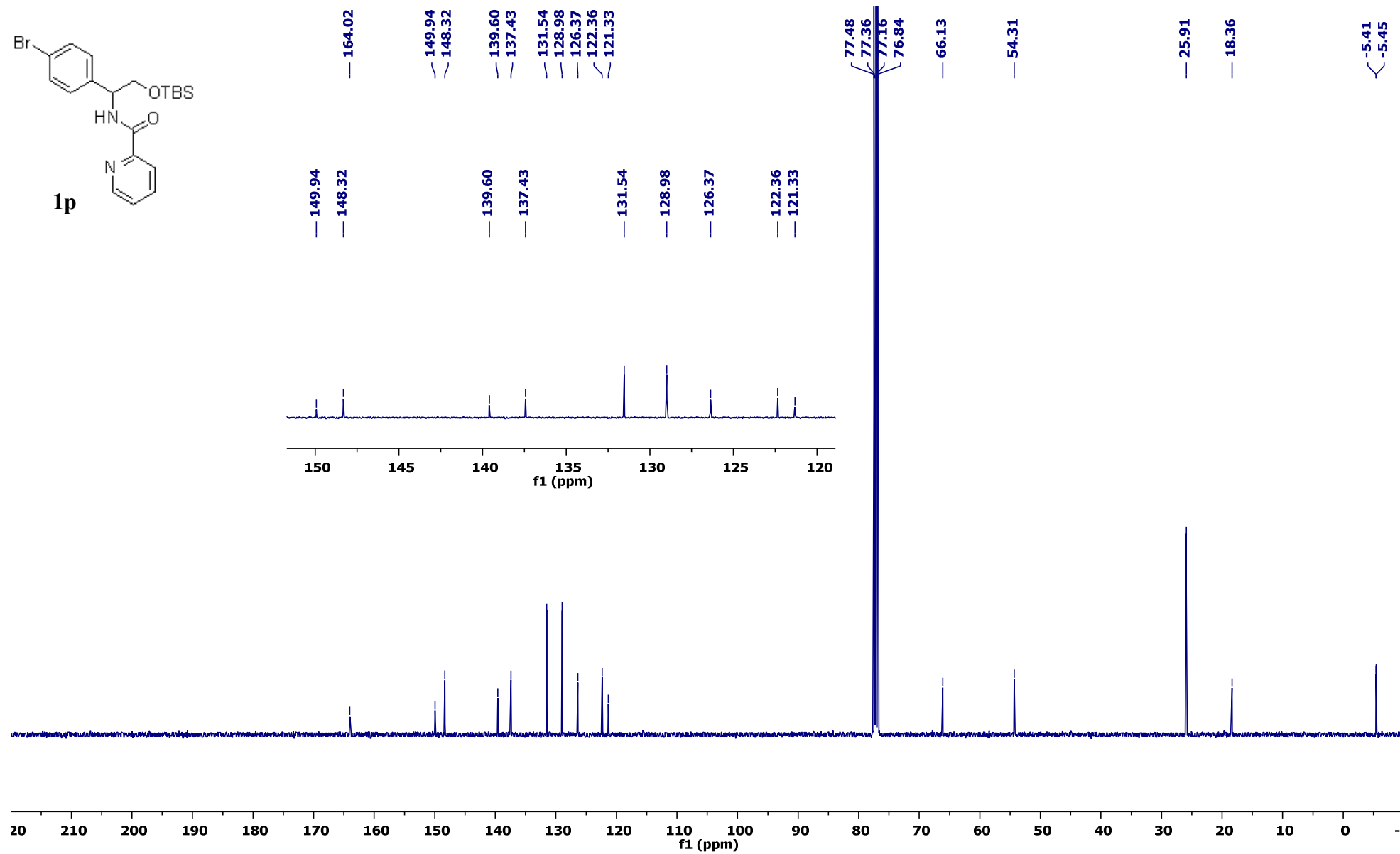
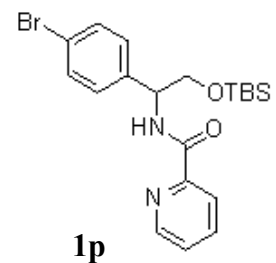
-5.44  
-5.45



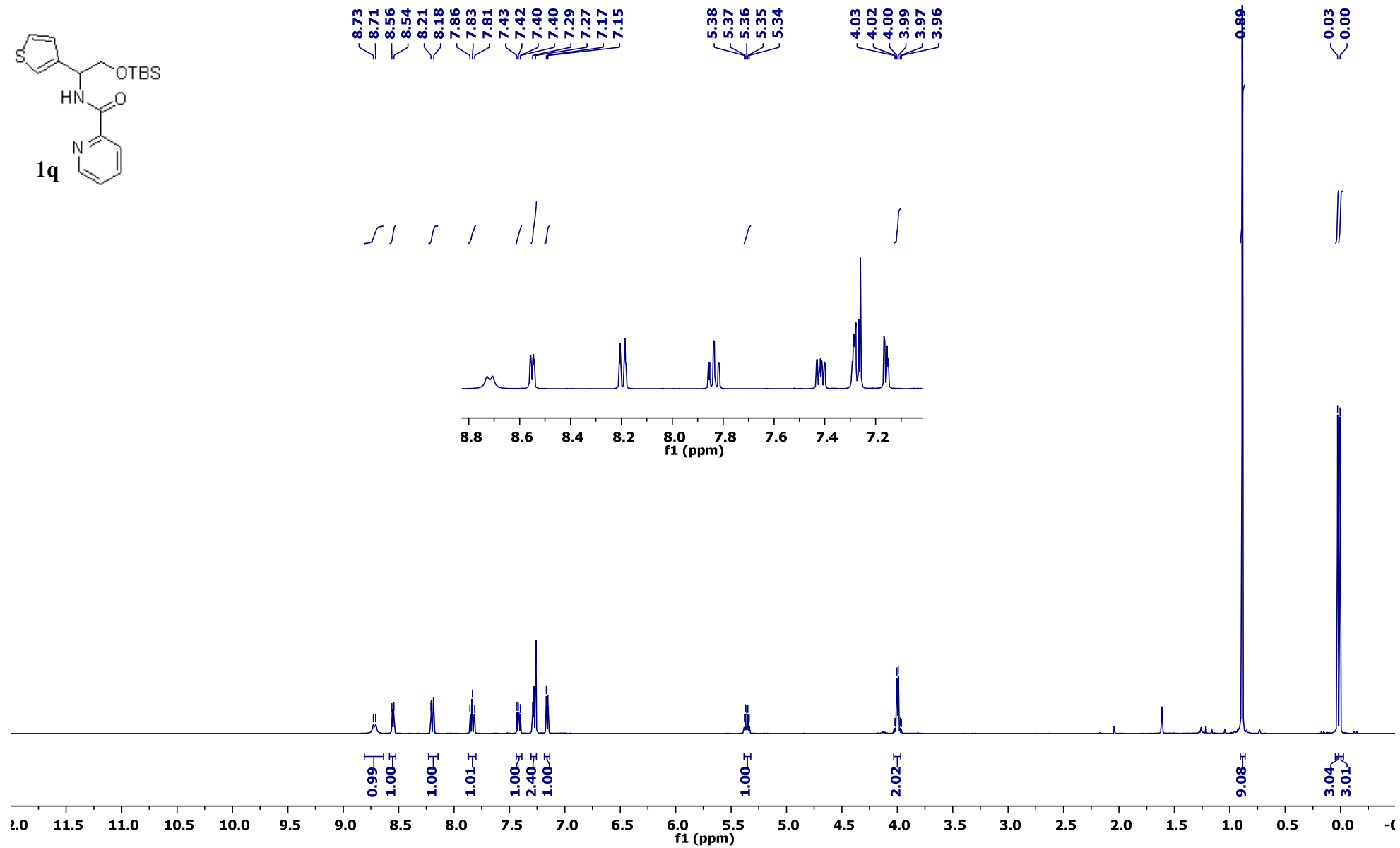
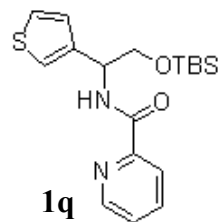
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



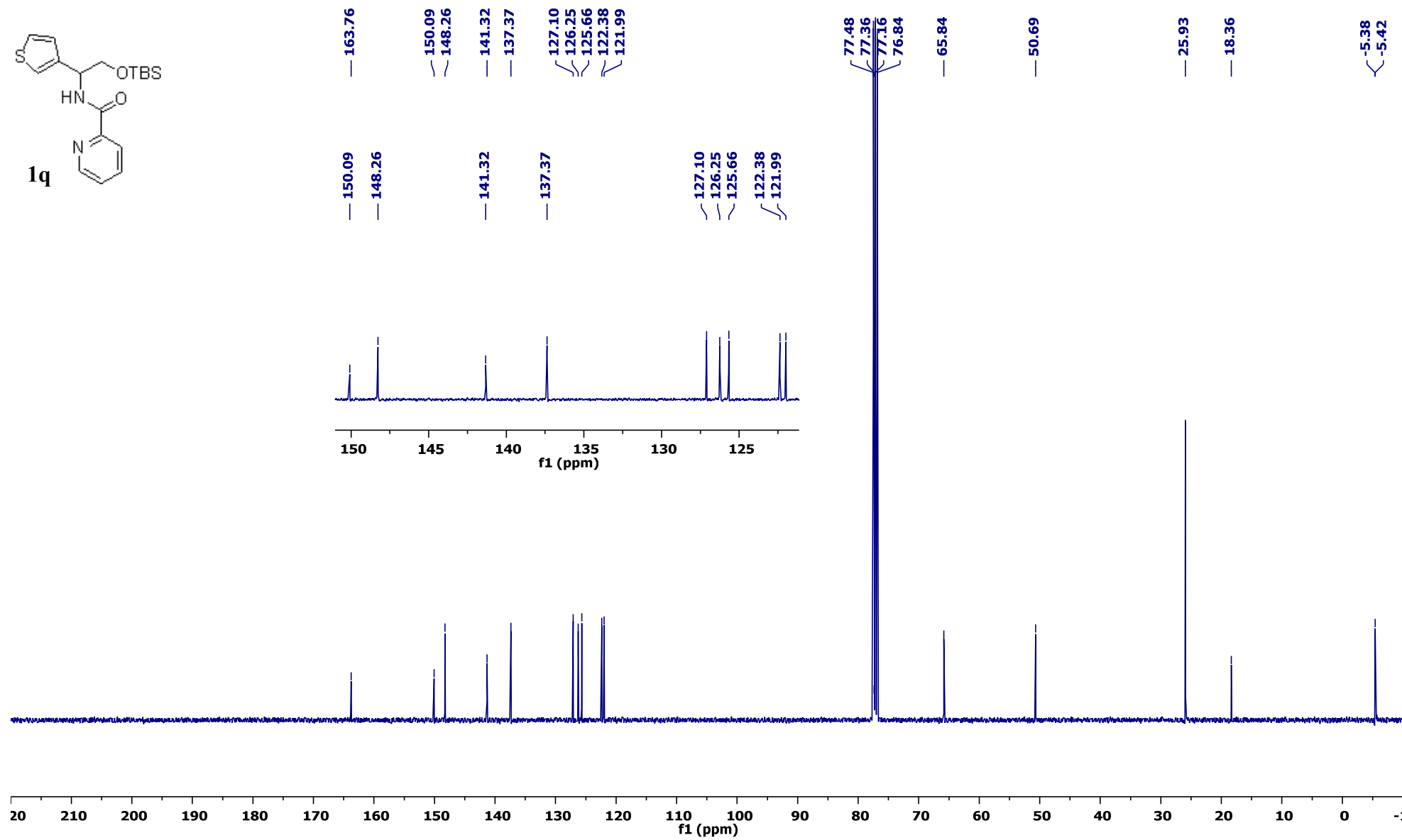
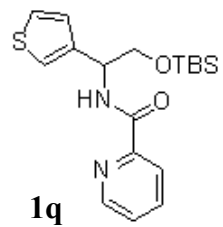
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



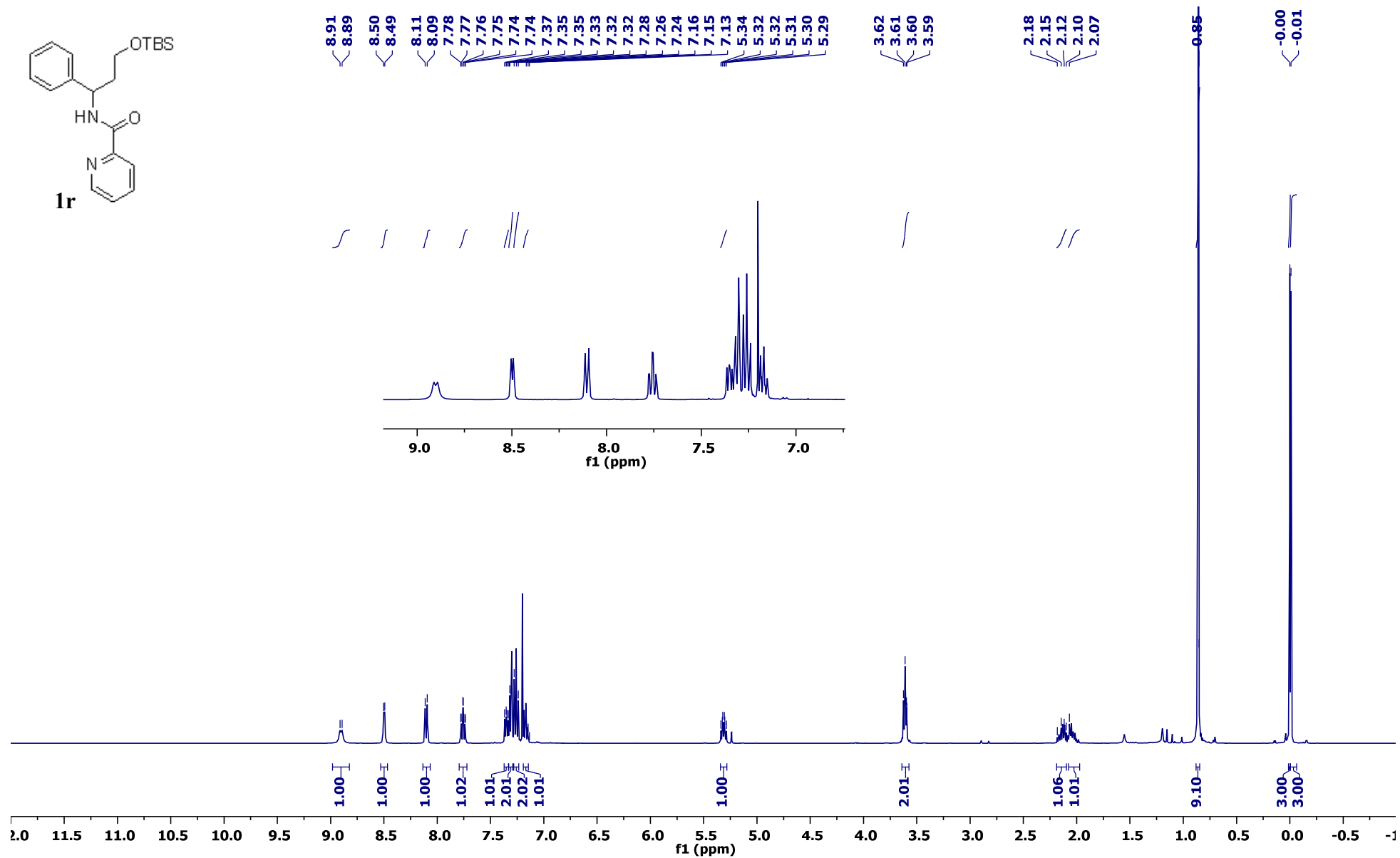
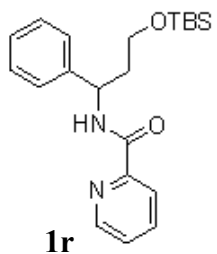
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



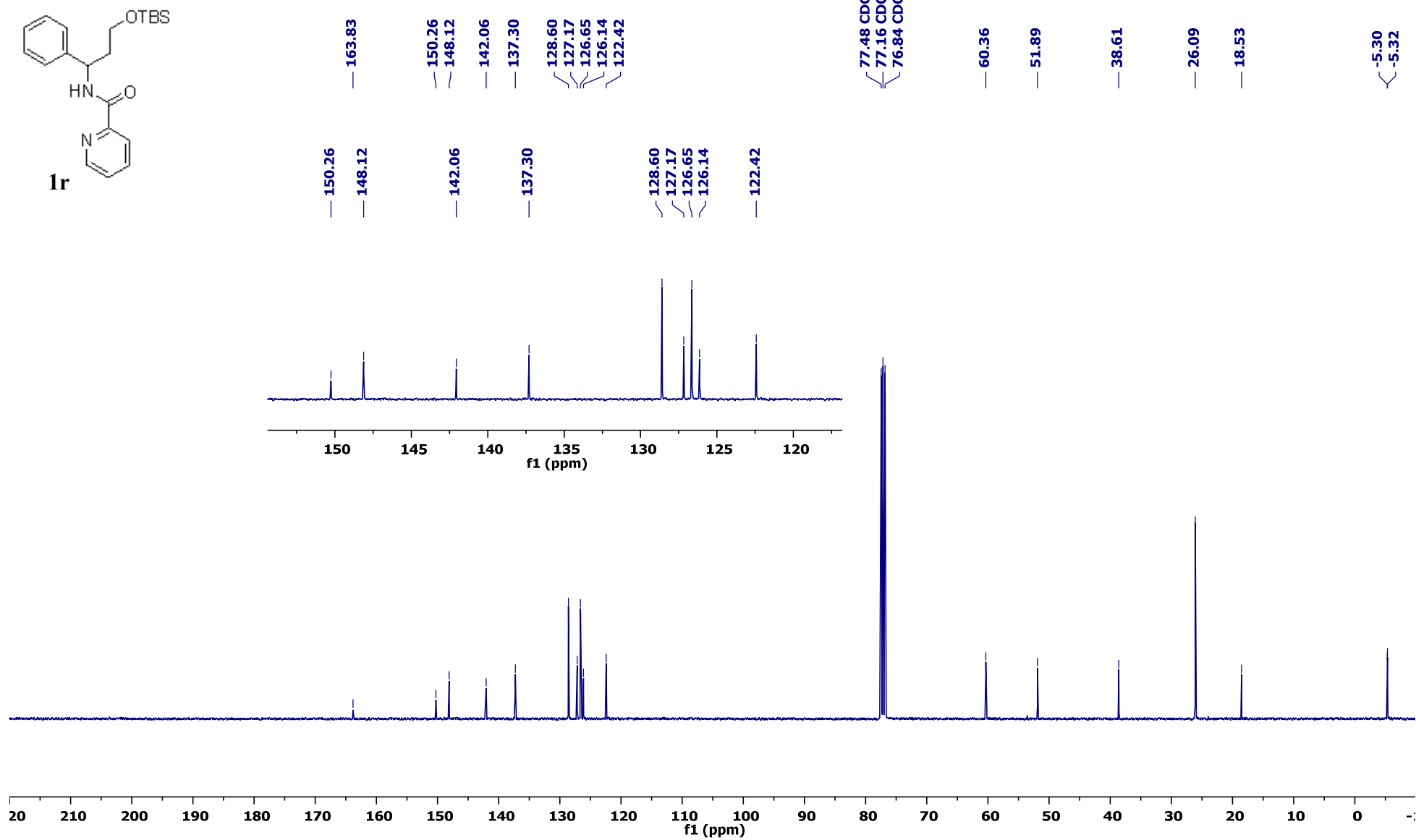
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



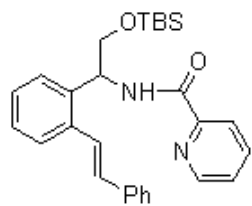
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )



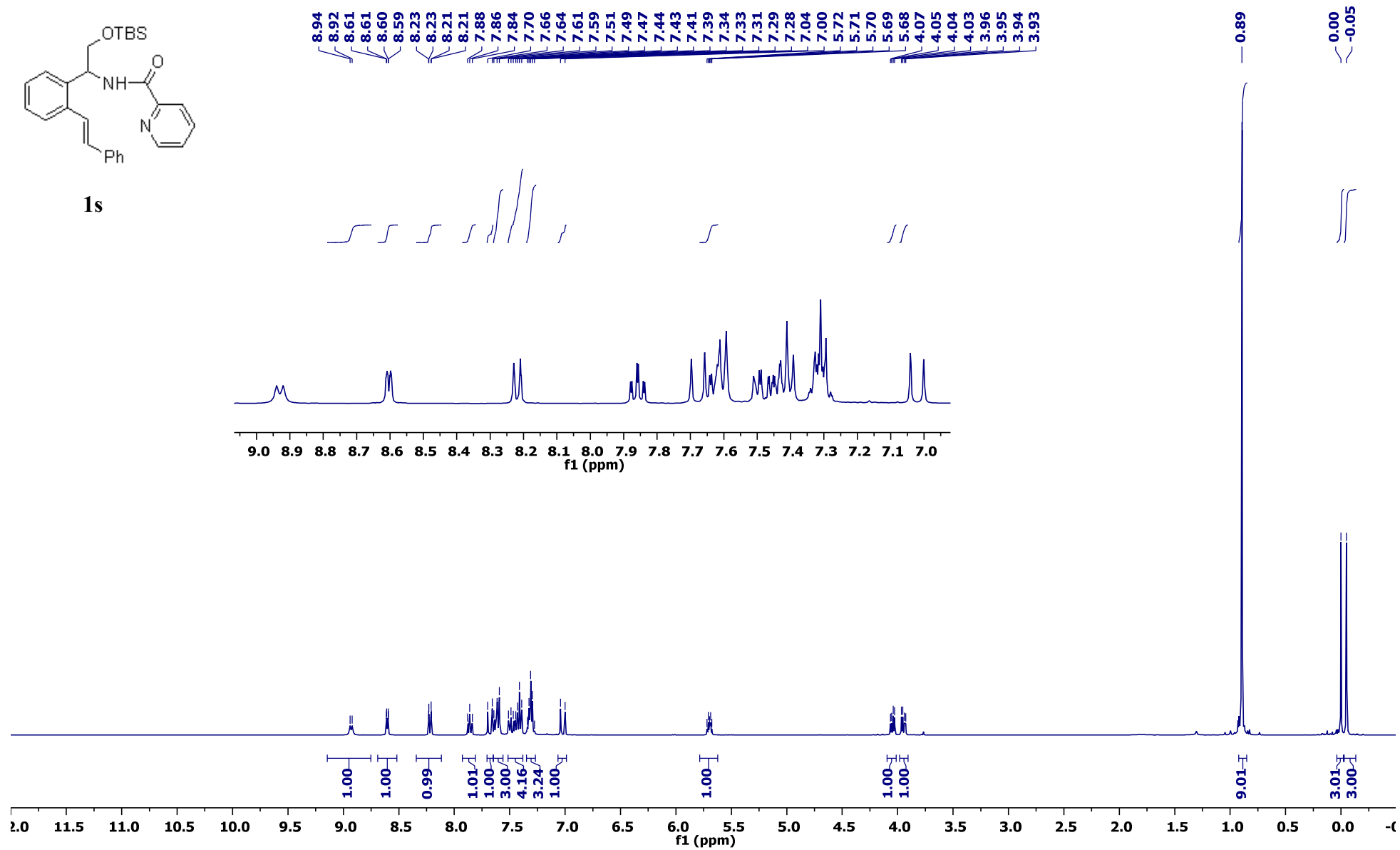
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



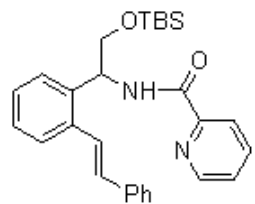
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



**1s**



$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



**1s**

137.72  
137.58  
137.36  
136.42

163.86

150.08  
148.26  
137.72  
137.58  
137.36  
136.42  
131.70  
128.76  
127.79  
127.66  
127.64  
126.87  
126.85  
126.71  
126.23  
126.21  
122.31

131.70

128.76  
127.79  
127.66  
127.64  
126.87  
126.85  
126.71  
126.23  
126.21

122.31

77.48  $\text{CDCl}_3$   
77.16  $\text{CDCl}_3$   
76.84  $\text{CDCl}_3$

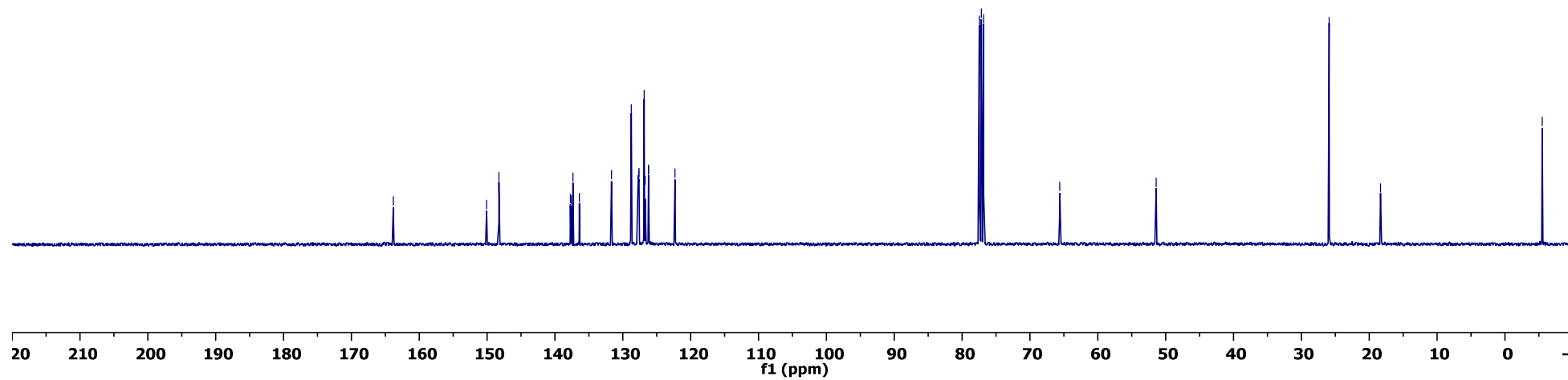
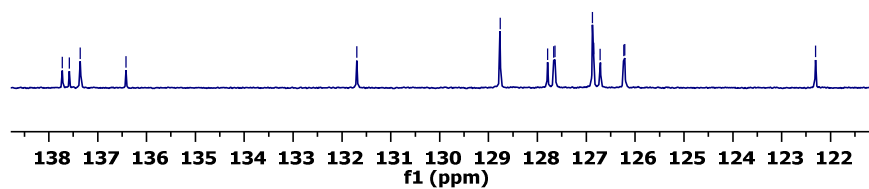
65.61

51.40

25.92

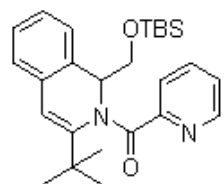
18.32

-5.47

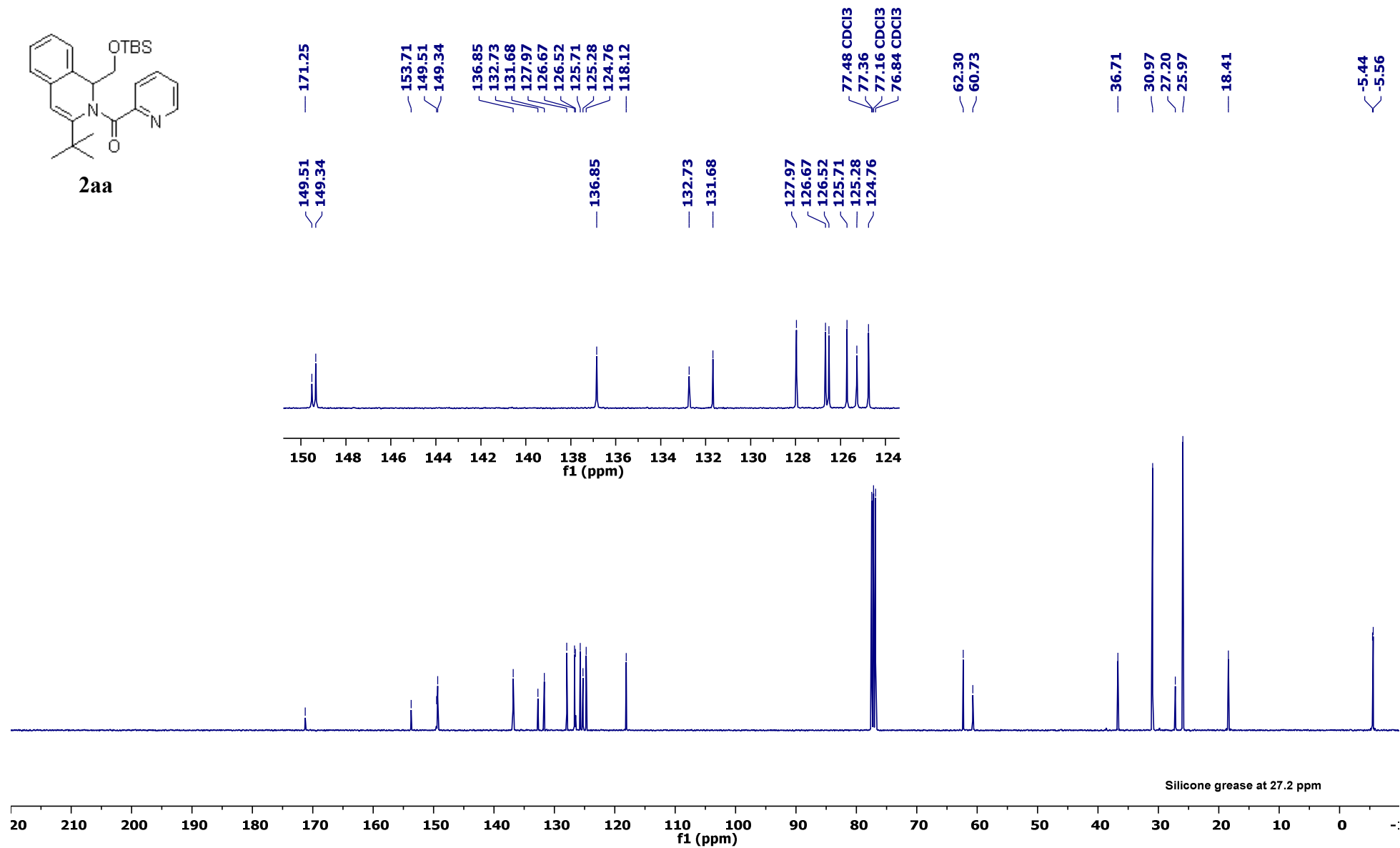


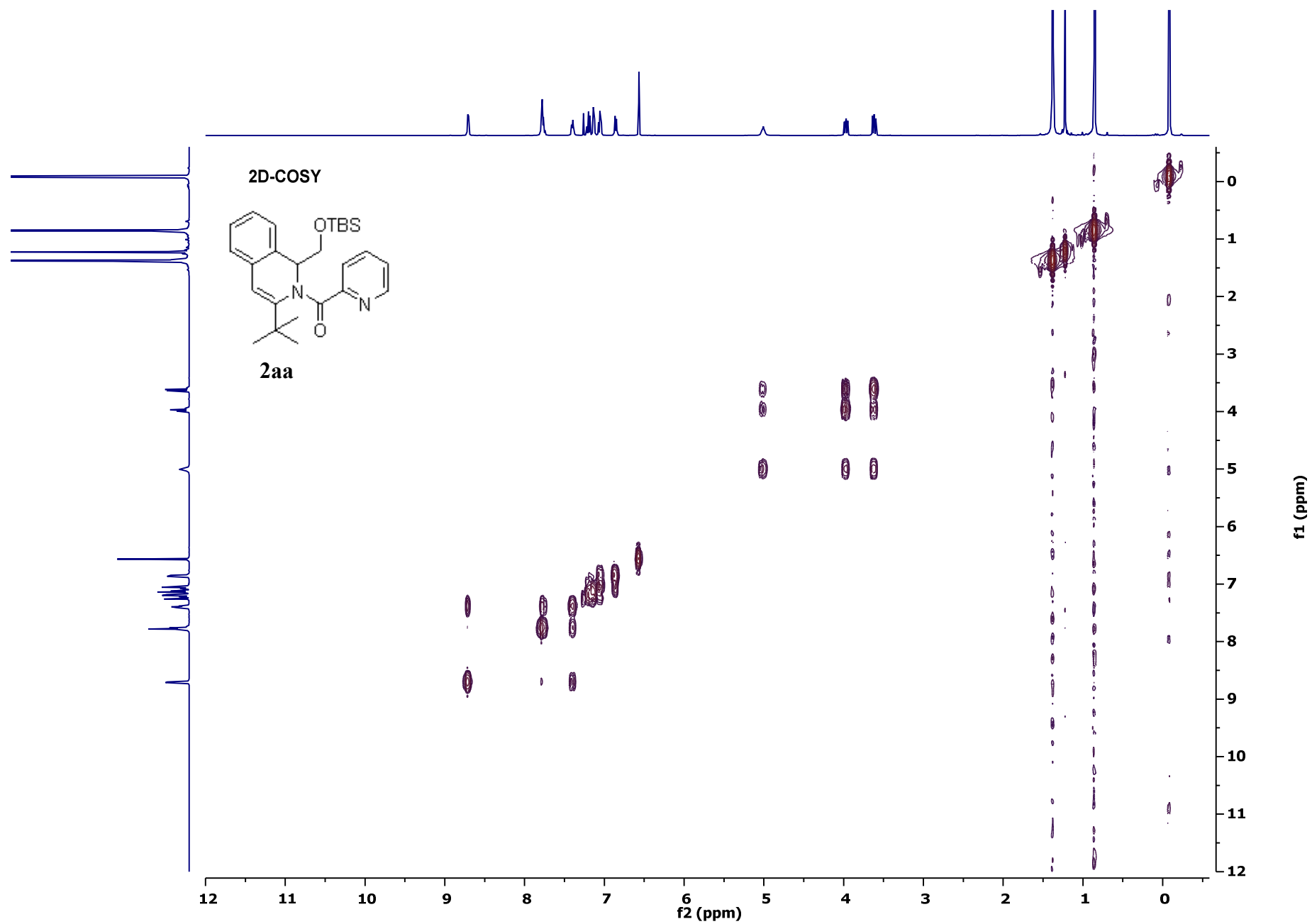


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

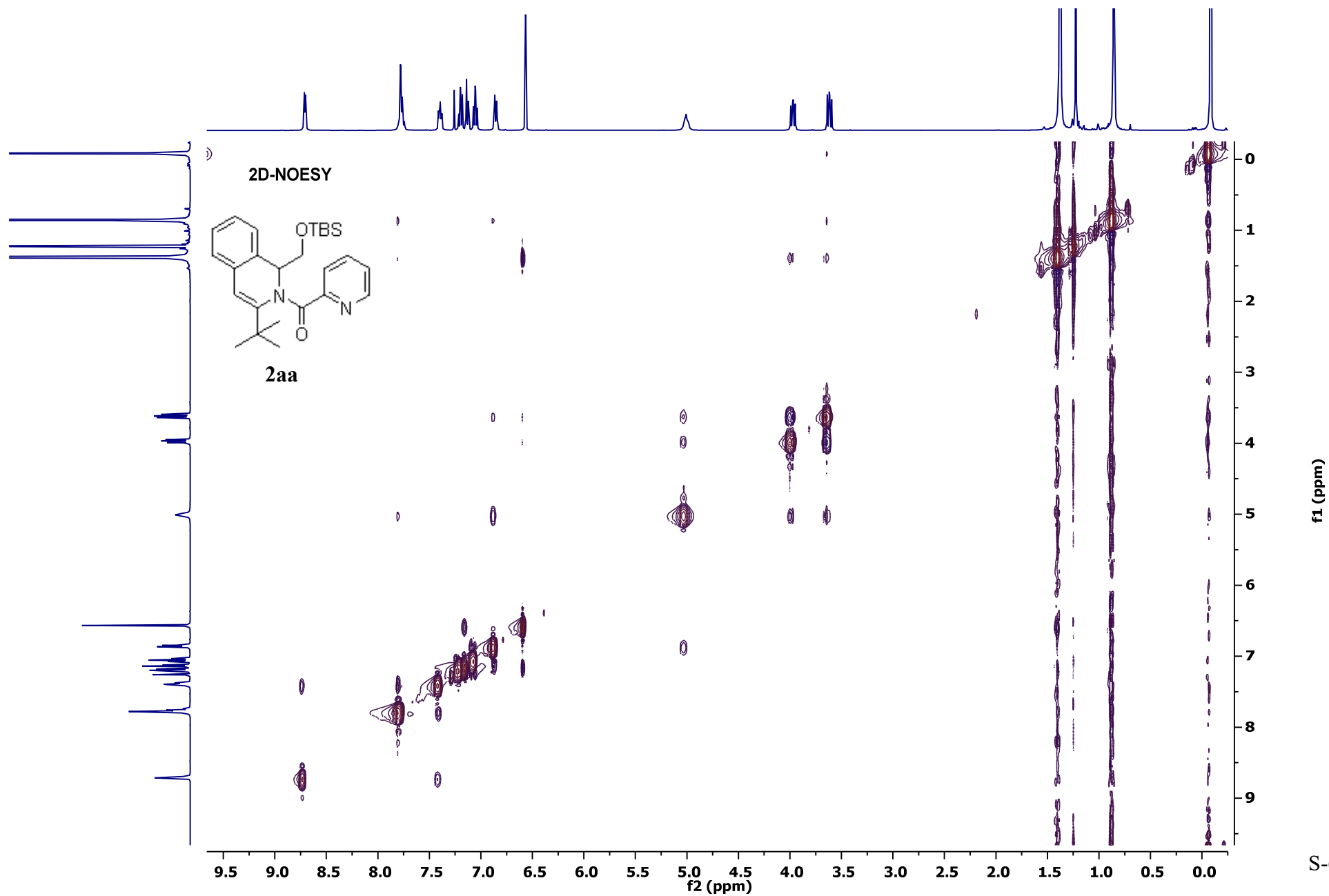


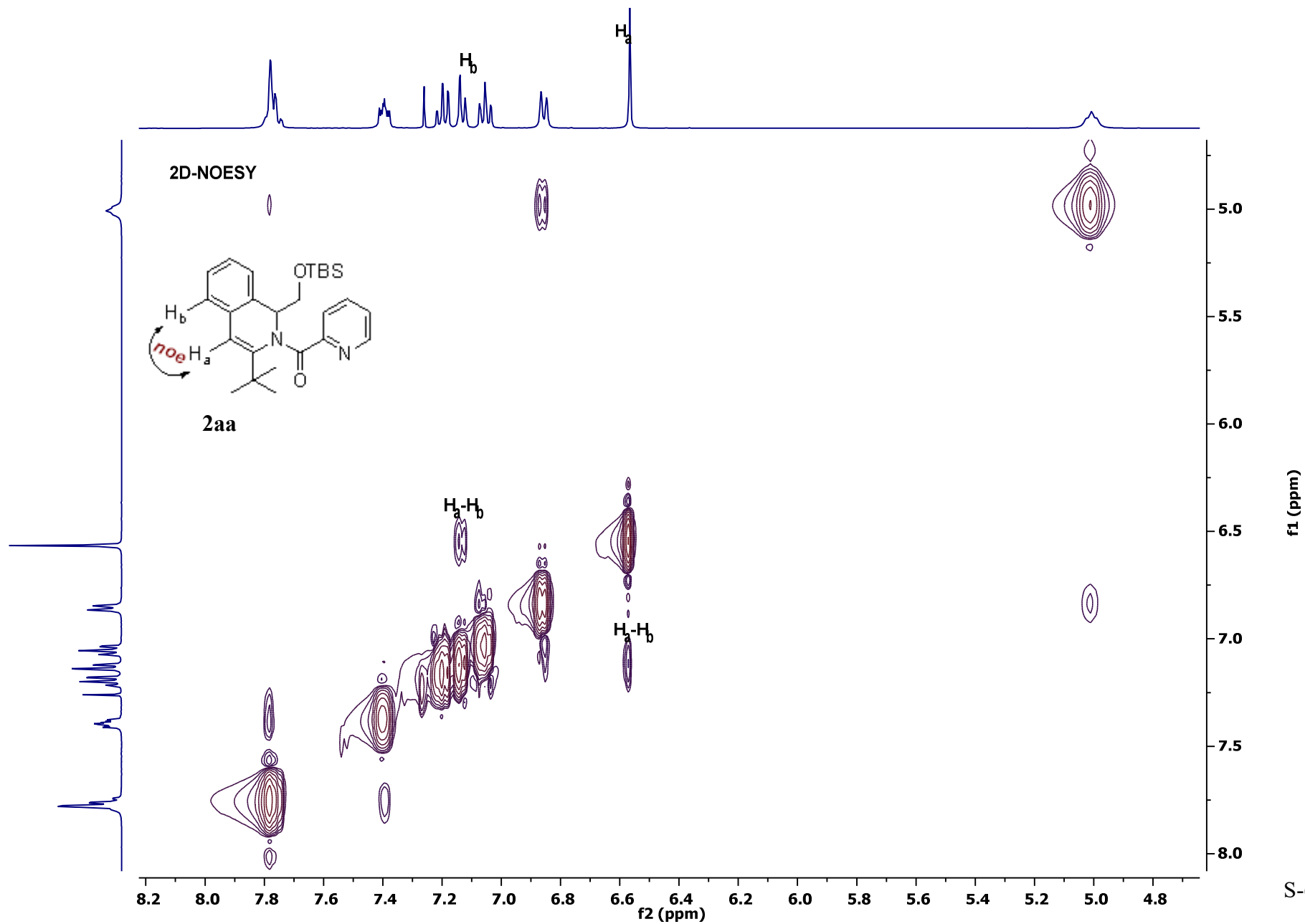
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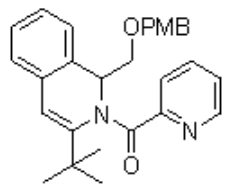


2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)

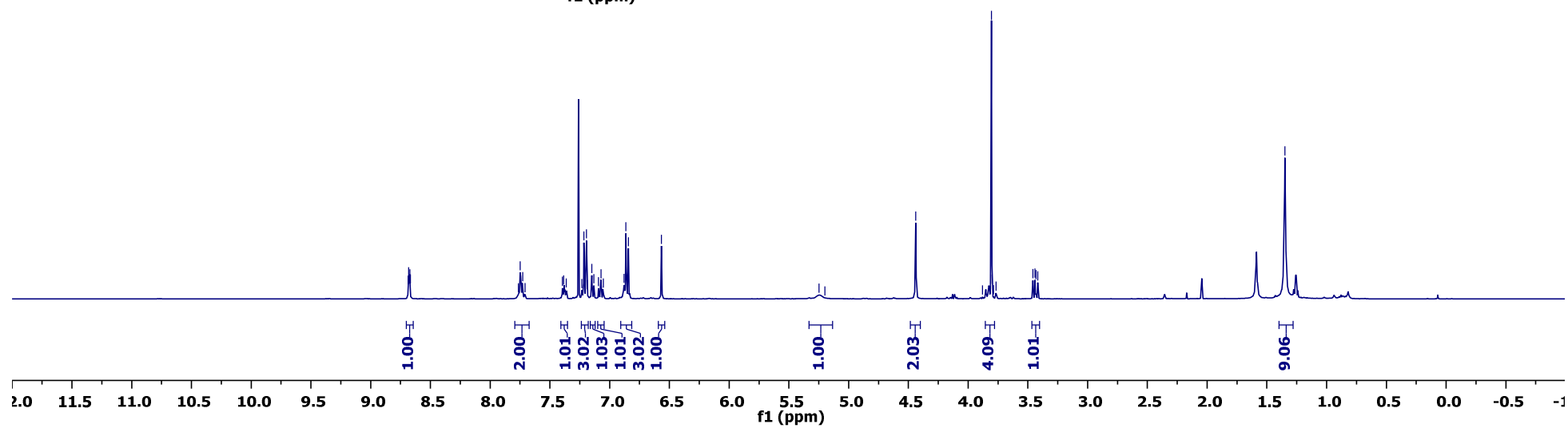
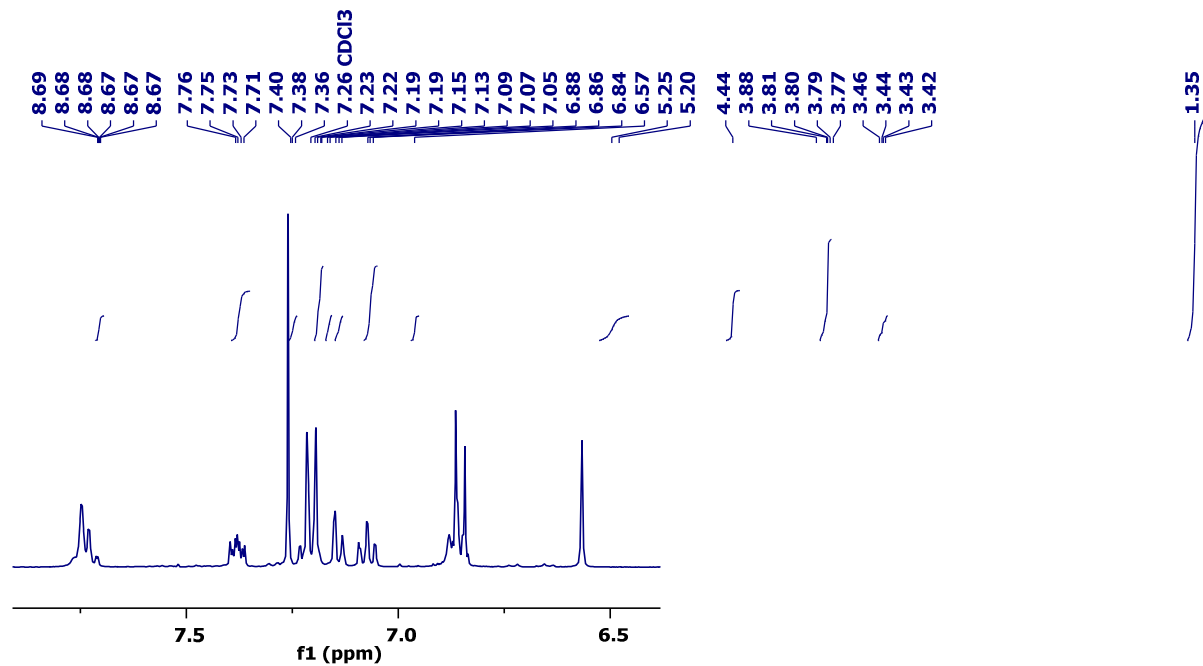




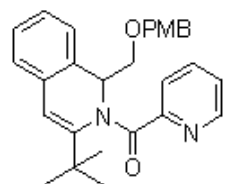
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



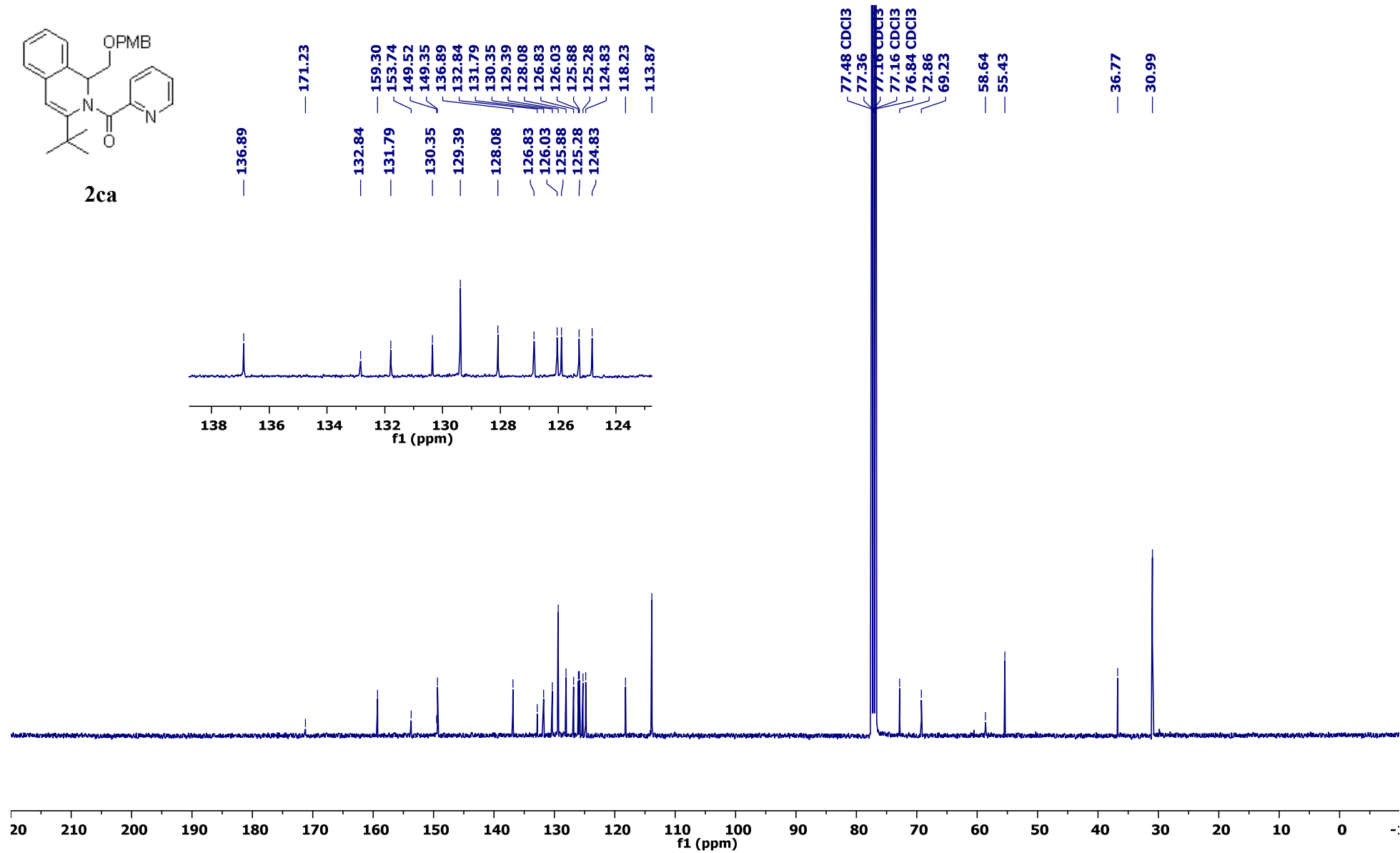
**2ca**



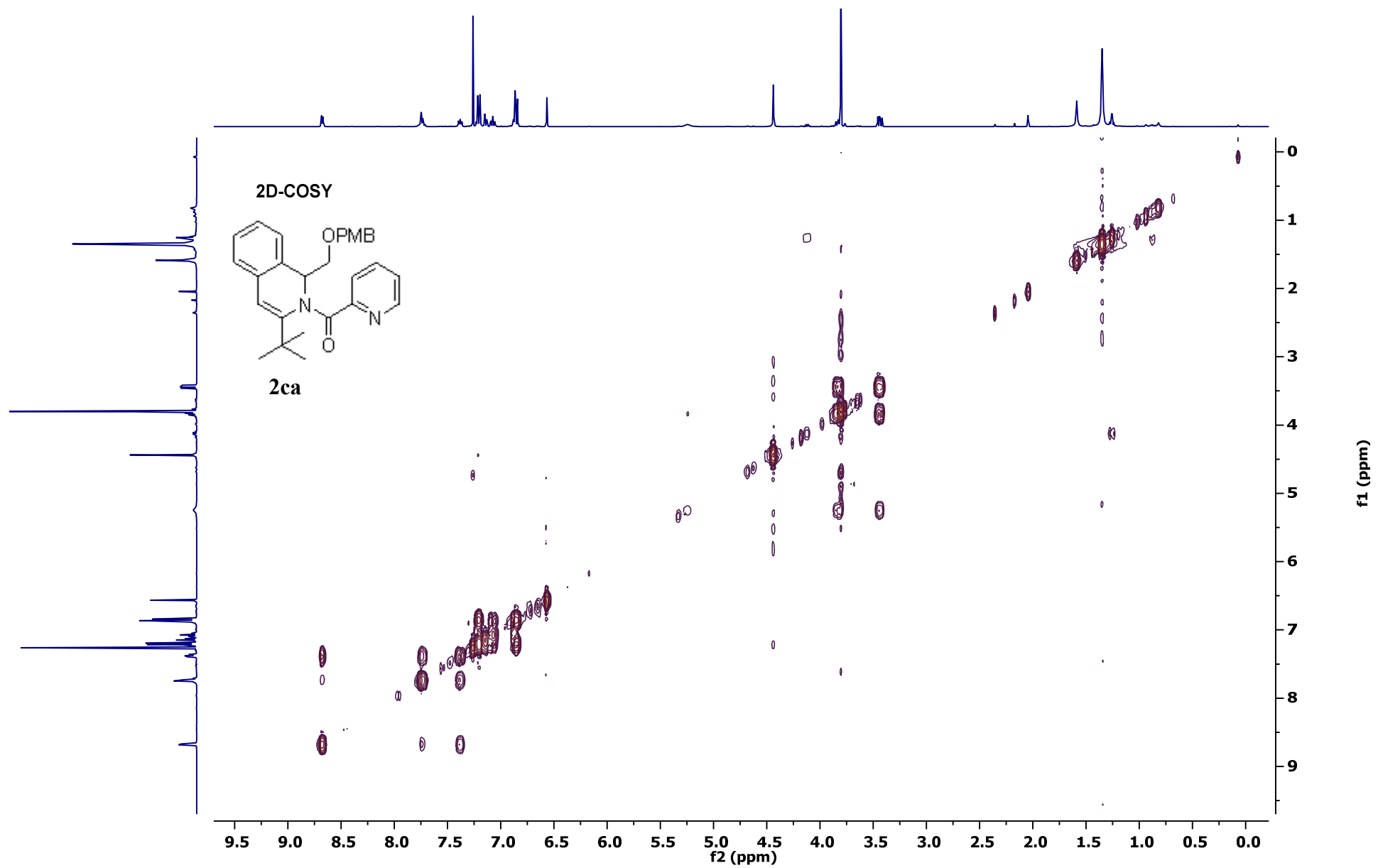
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



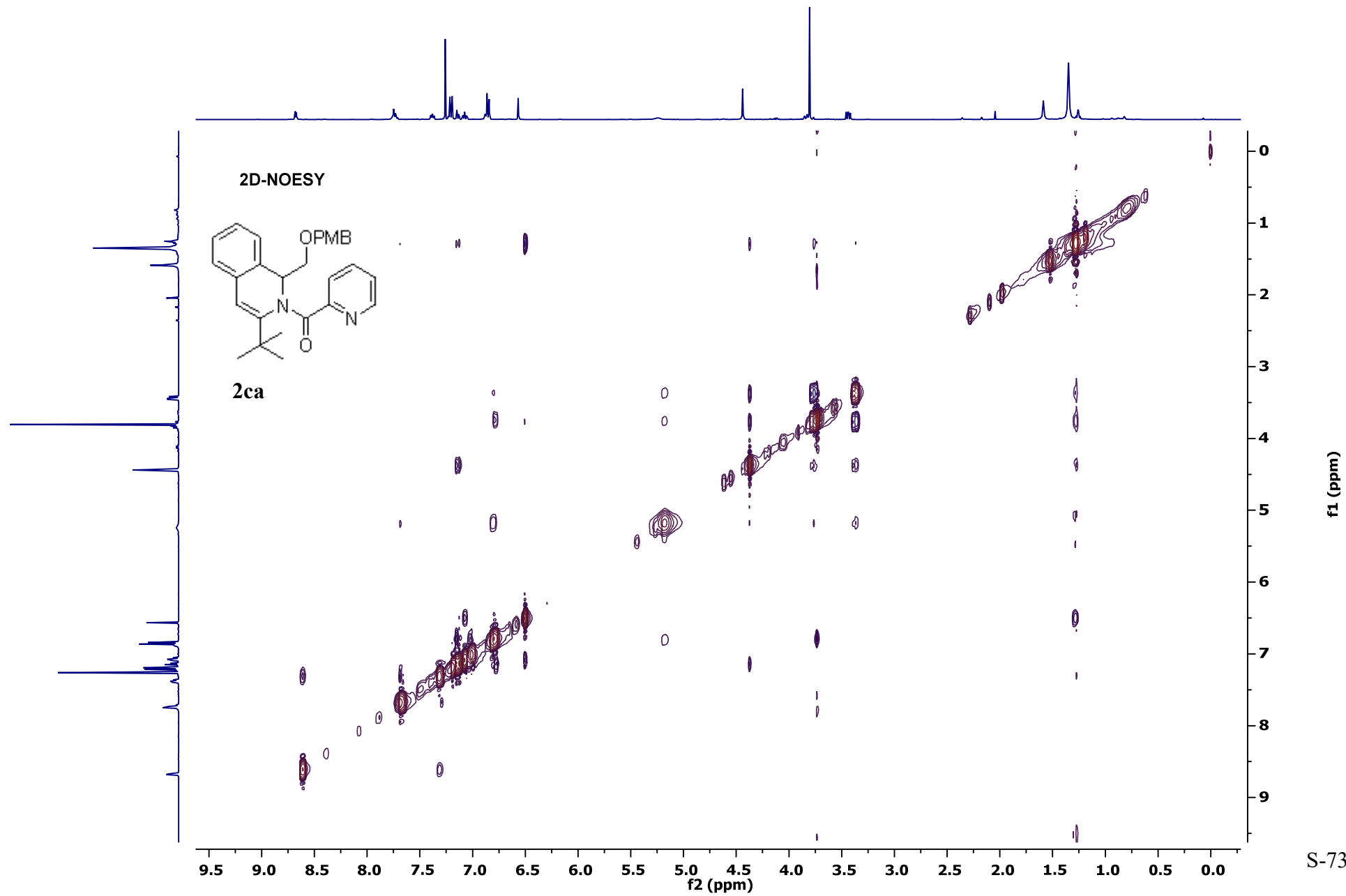
2ca



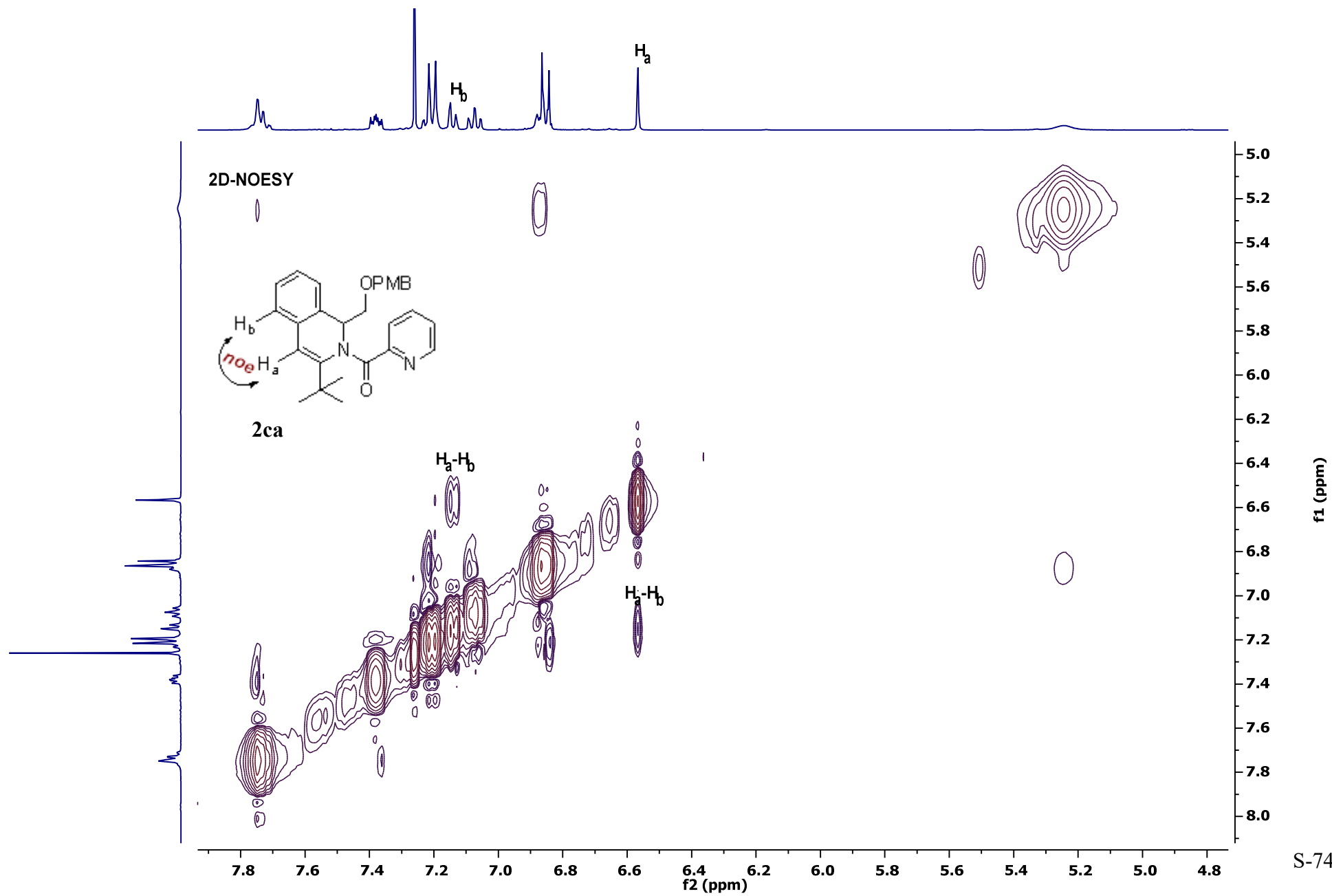
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



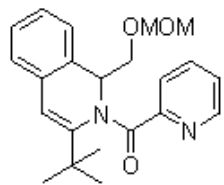
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



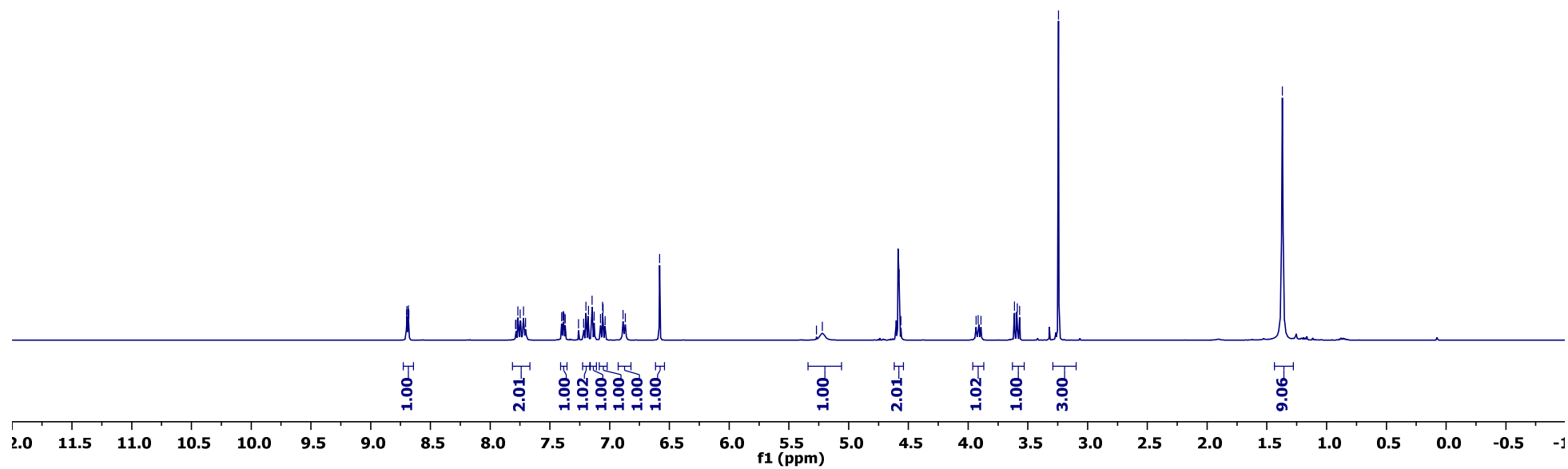
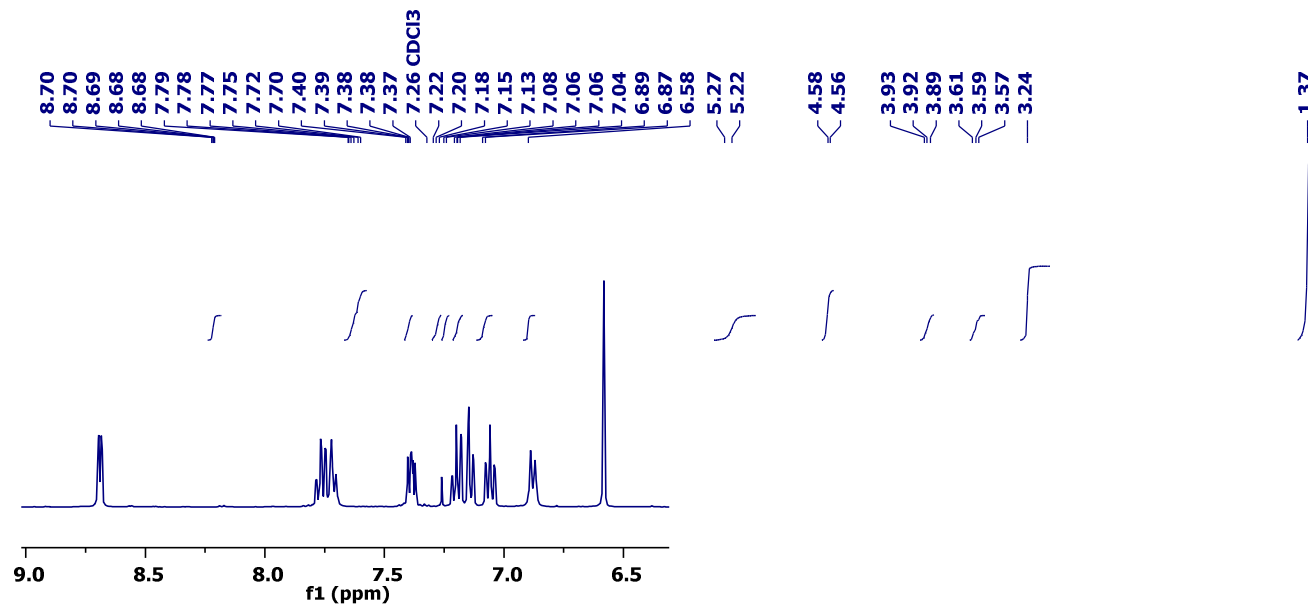
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



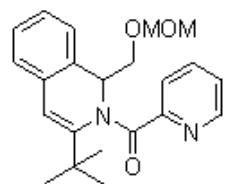
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



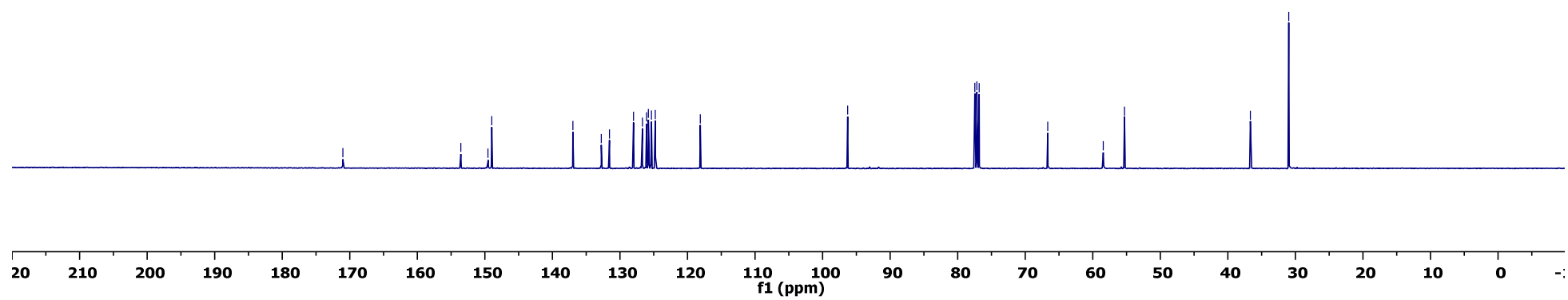
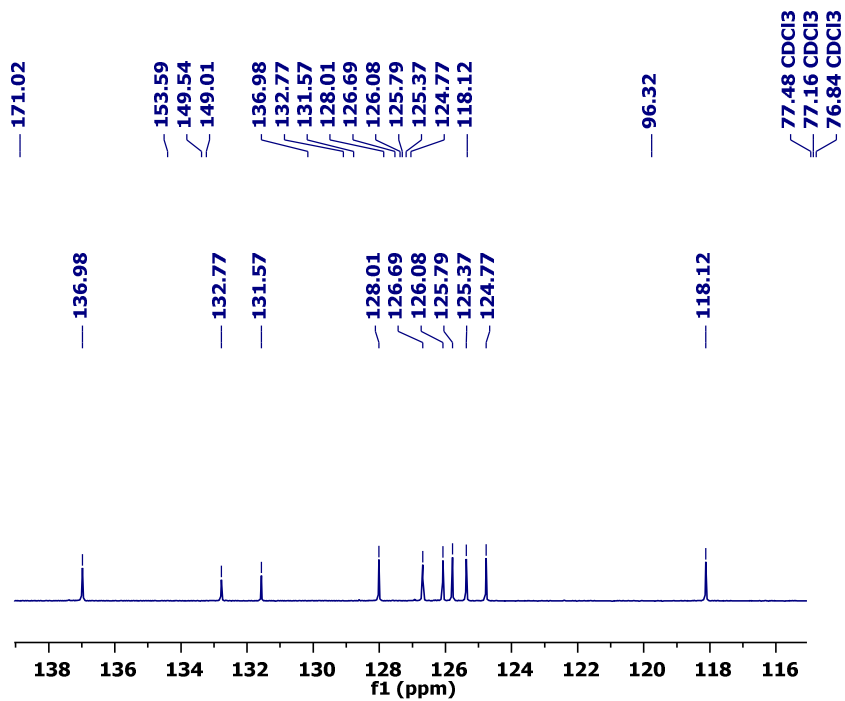
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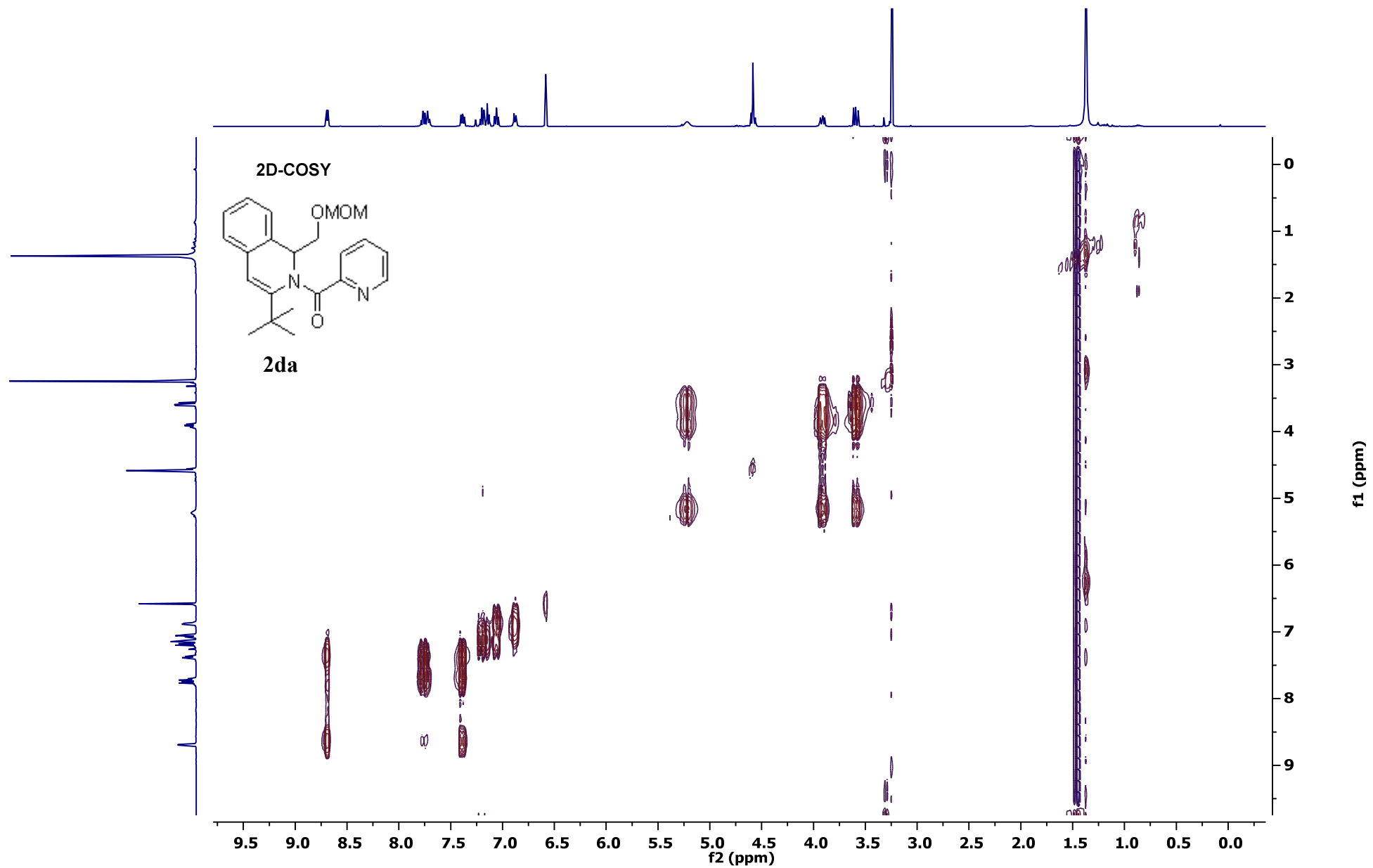
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



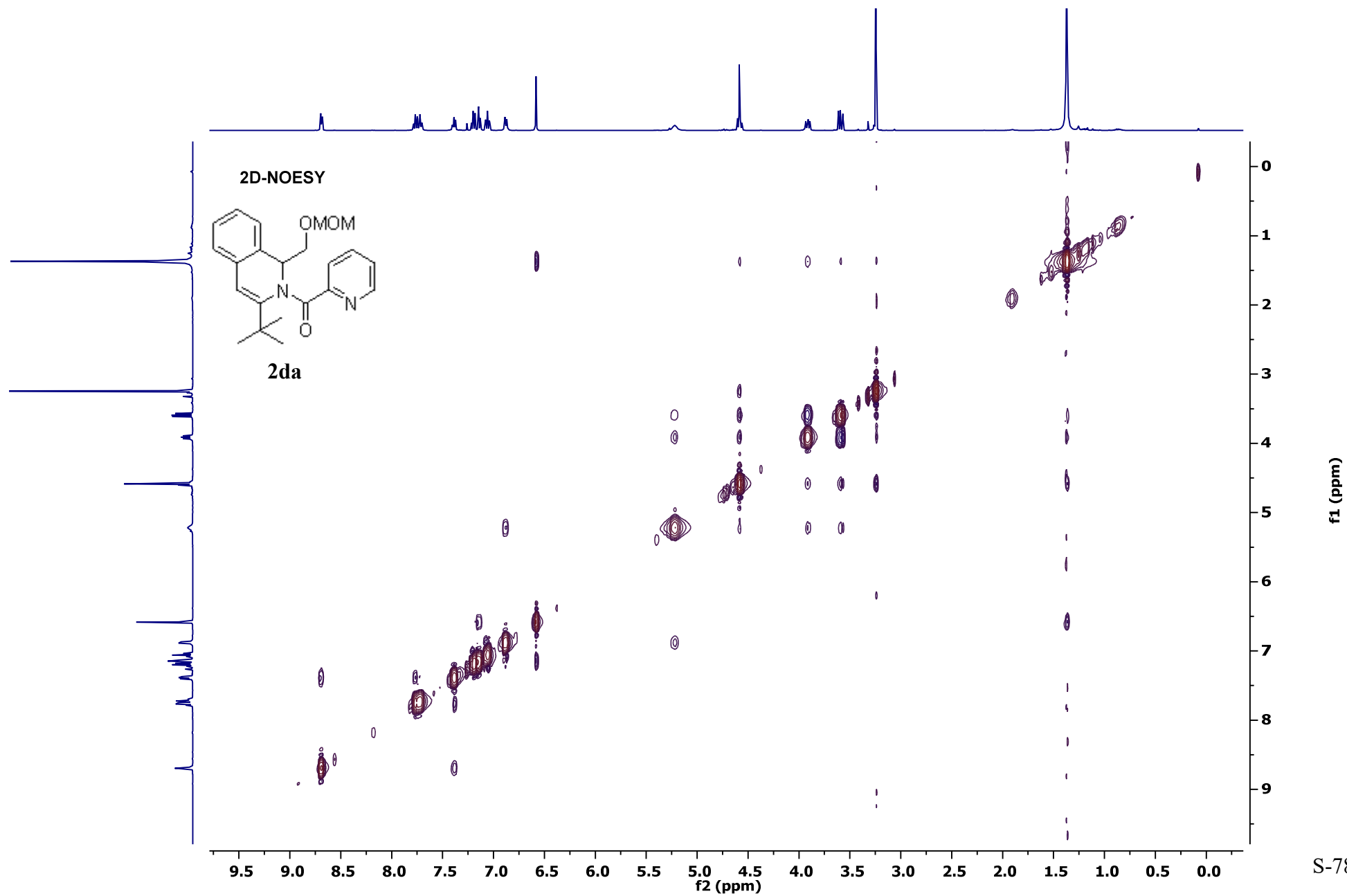
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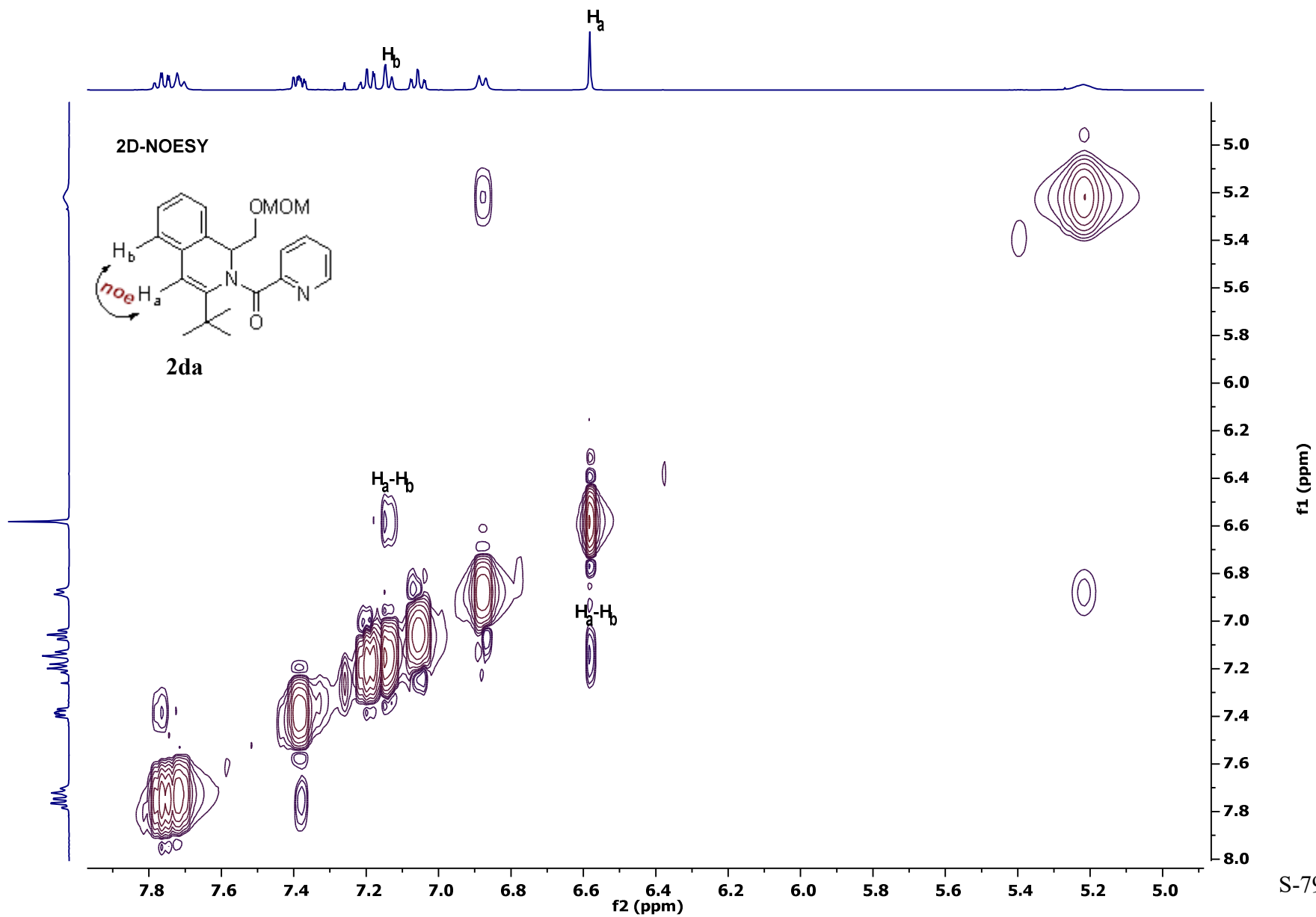


2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)

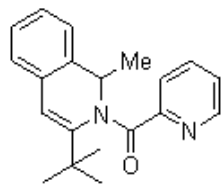


2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)

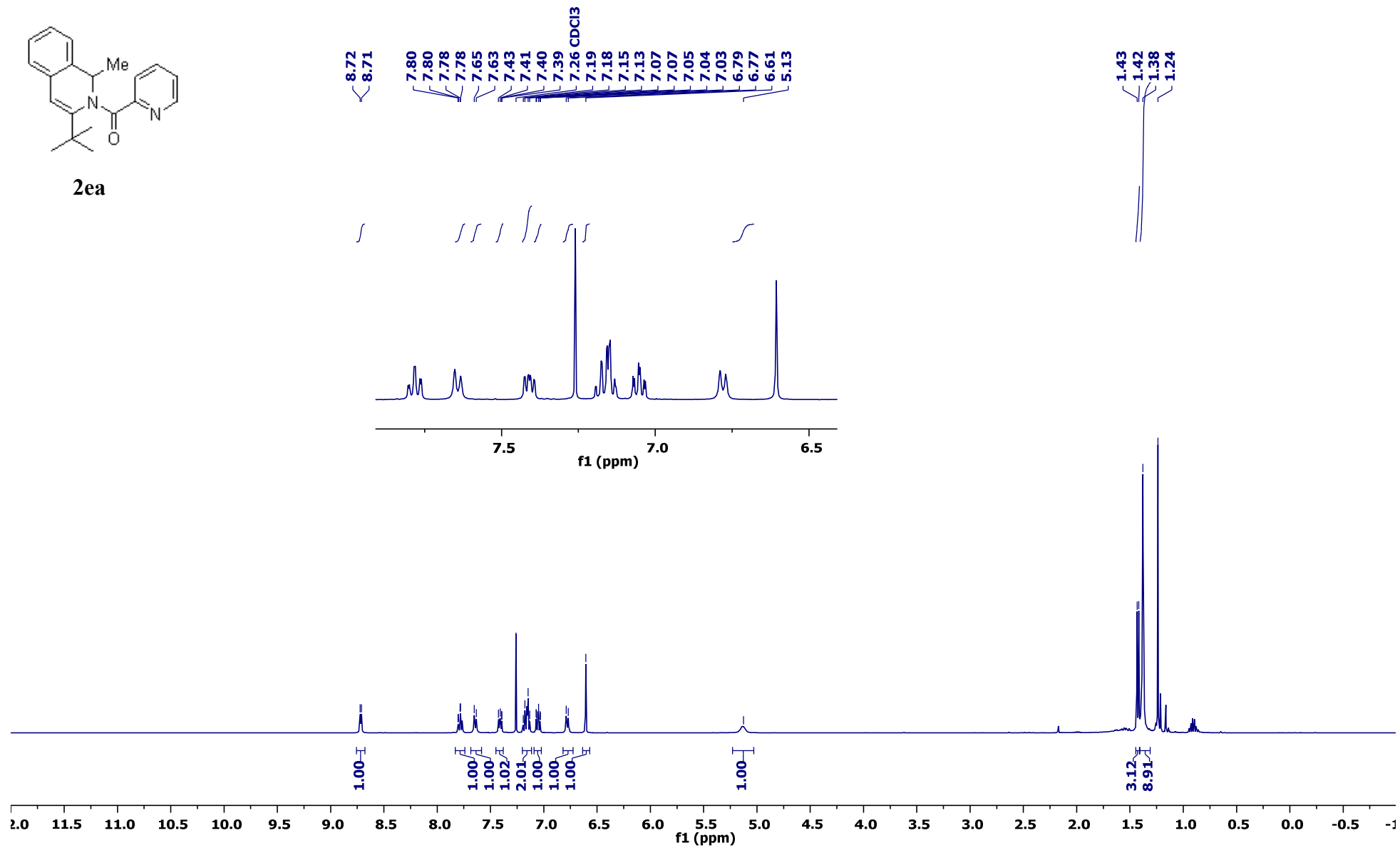




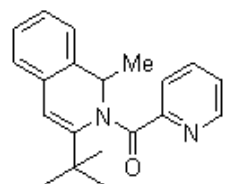
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



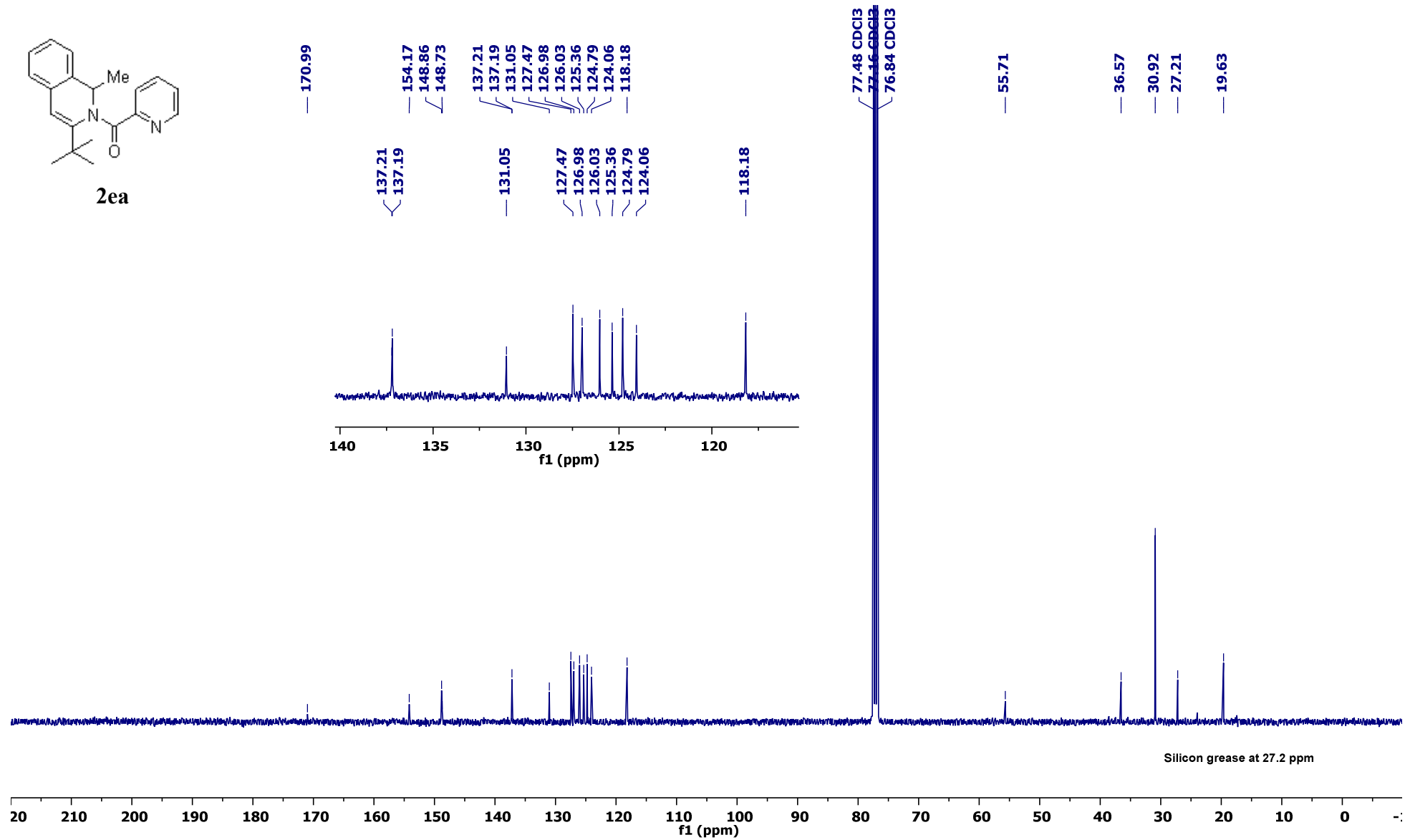
**2ea**



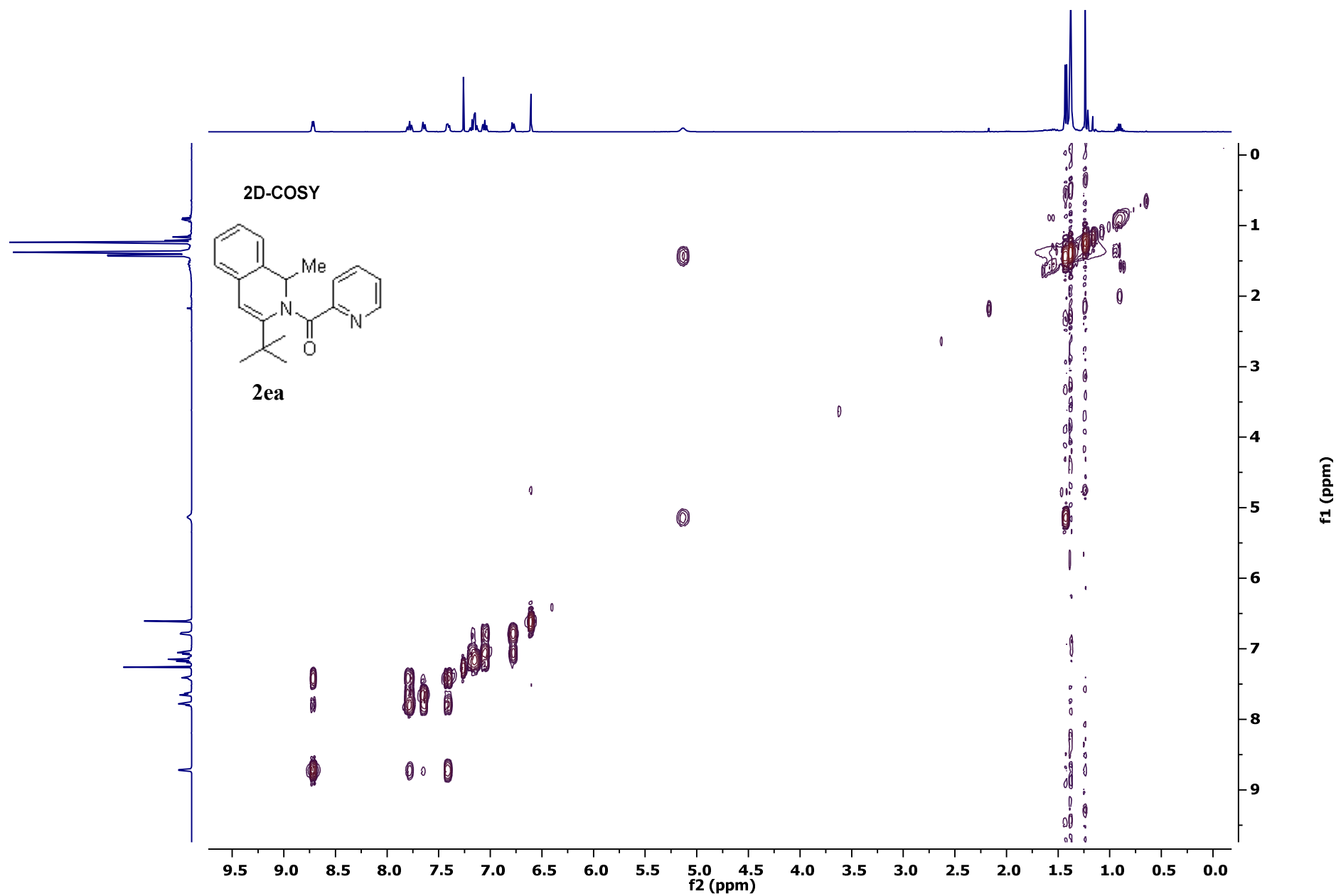
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

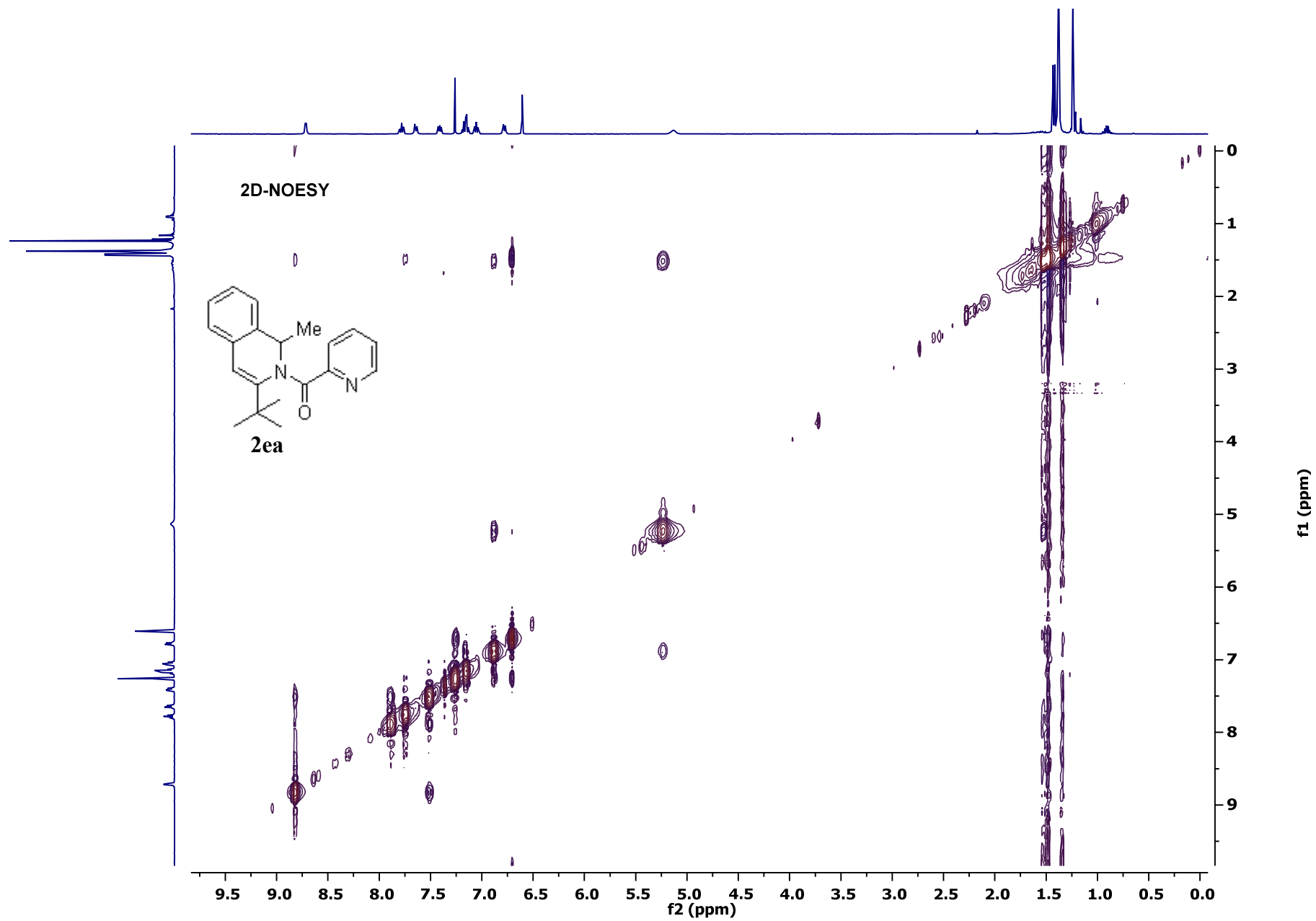


2ea

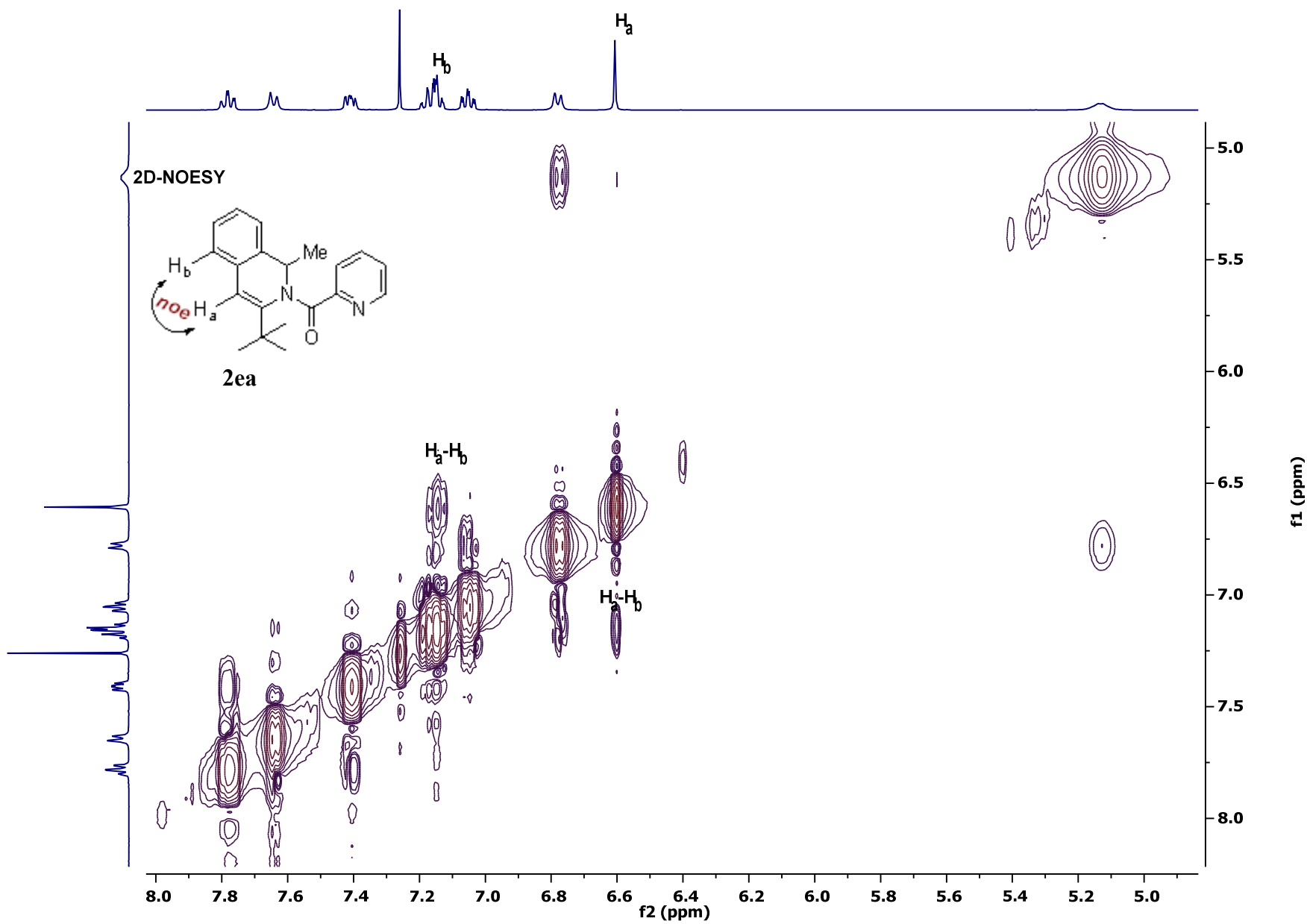


2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)

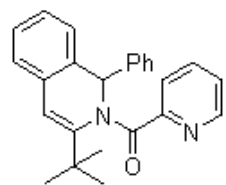




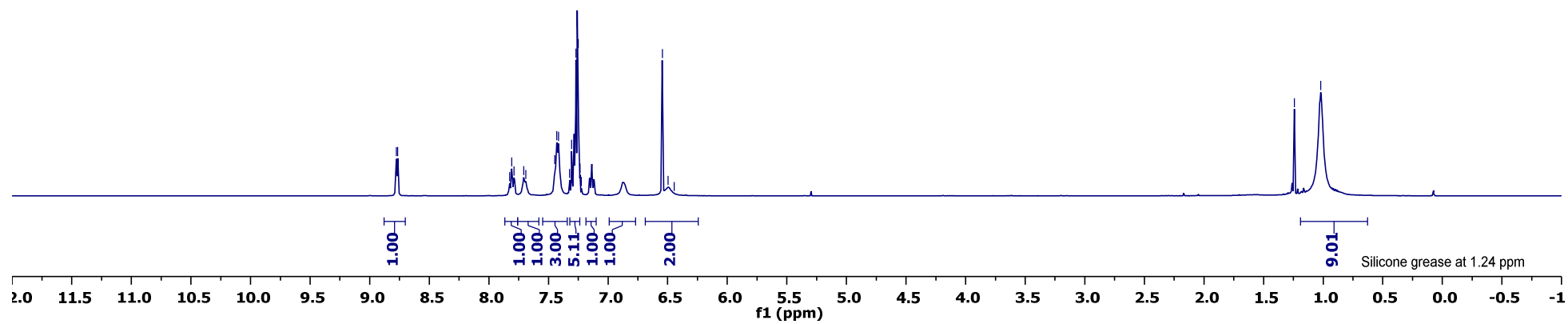
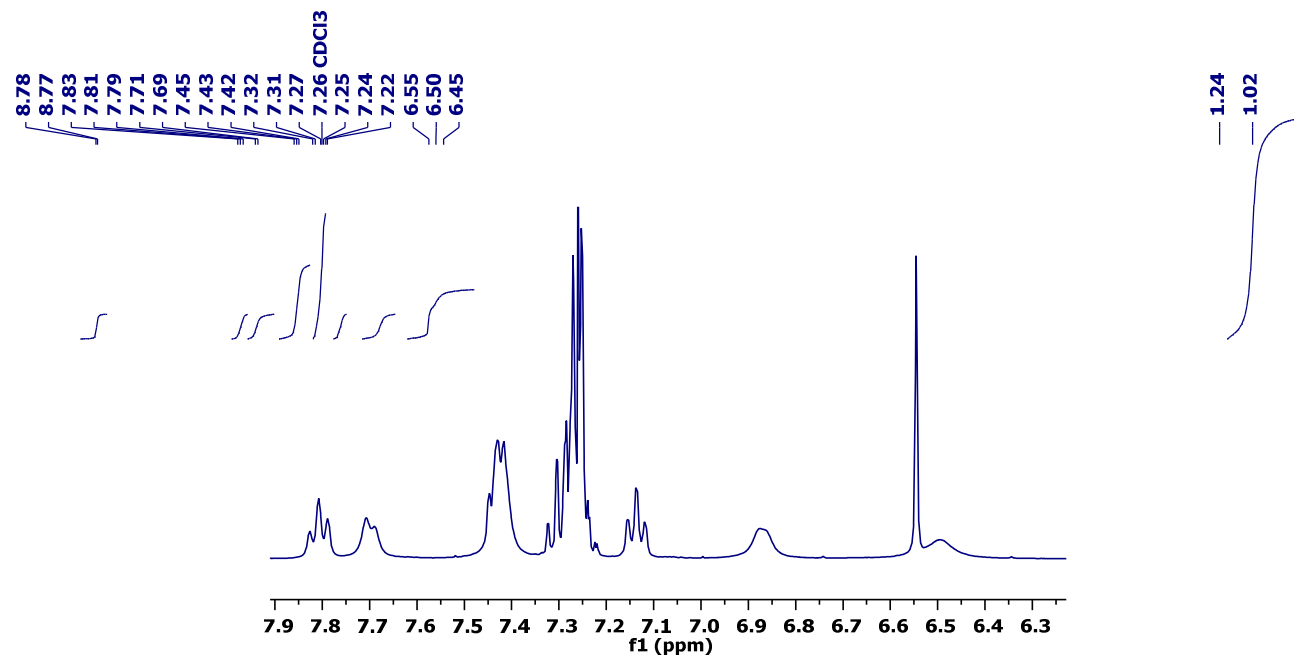
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



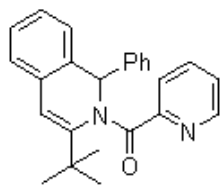
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



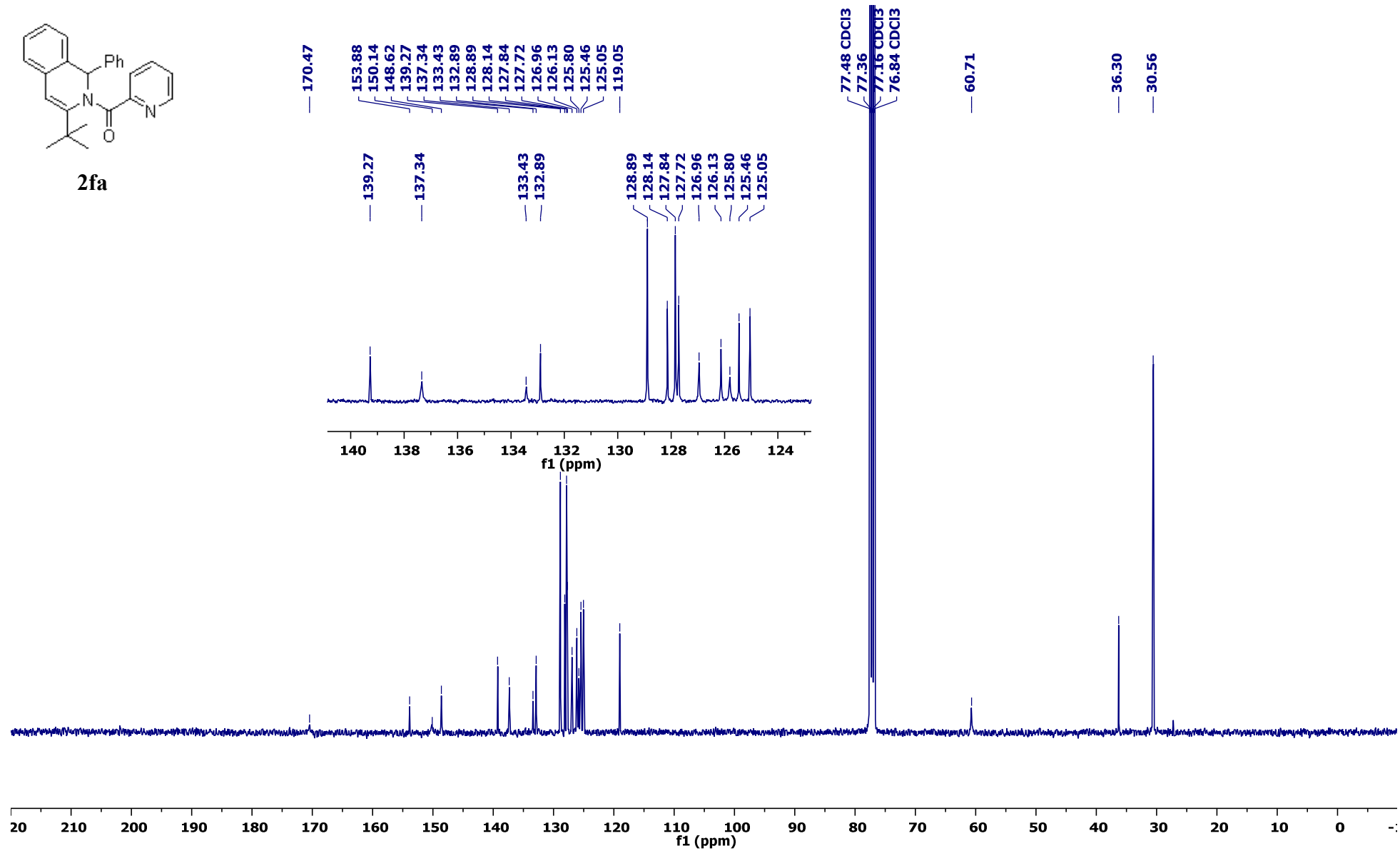
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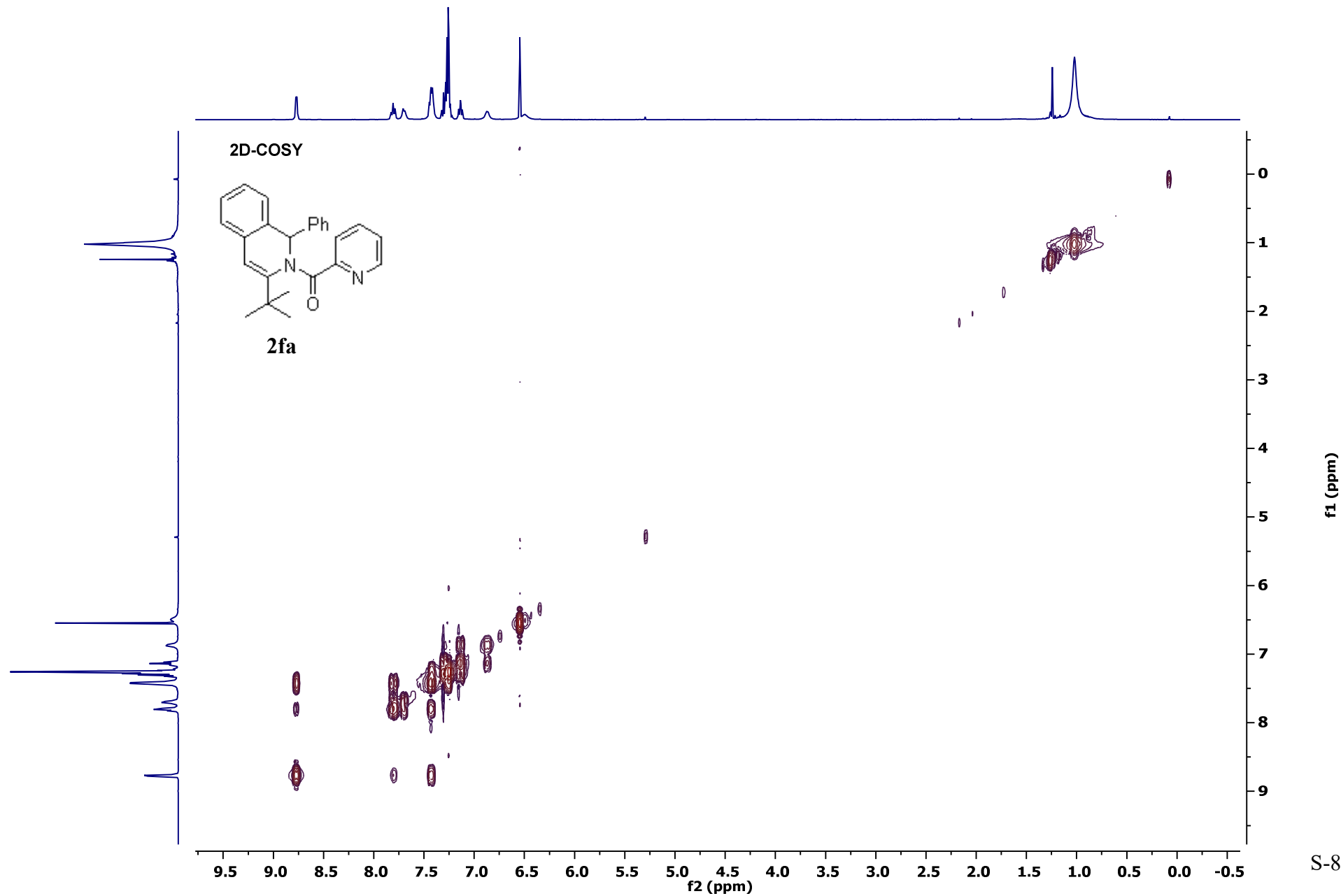


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

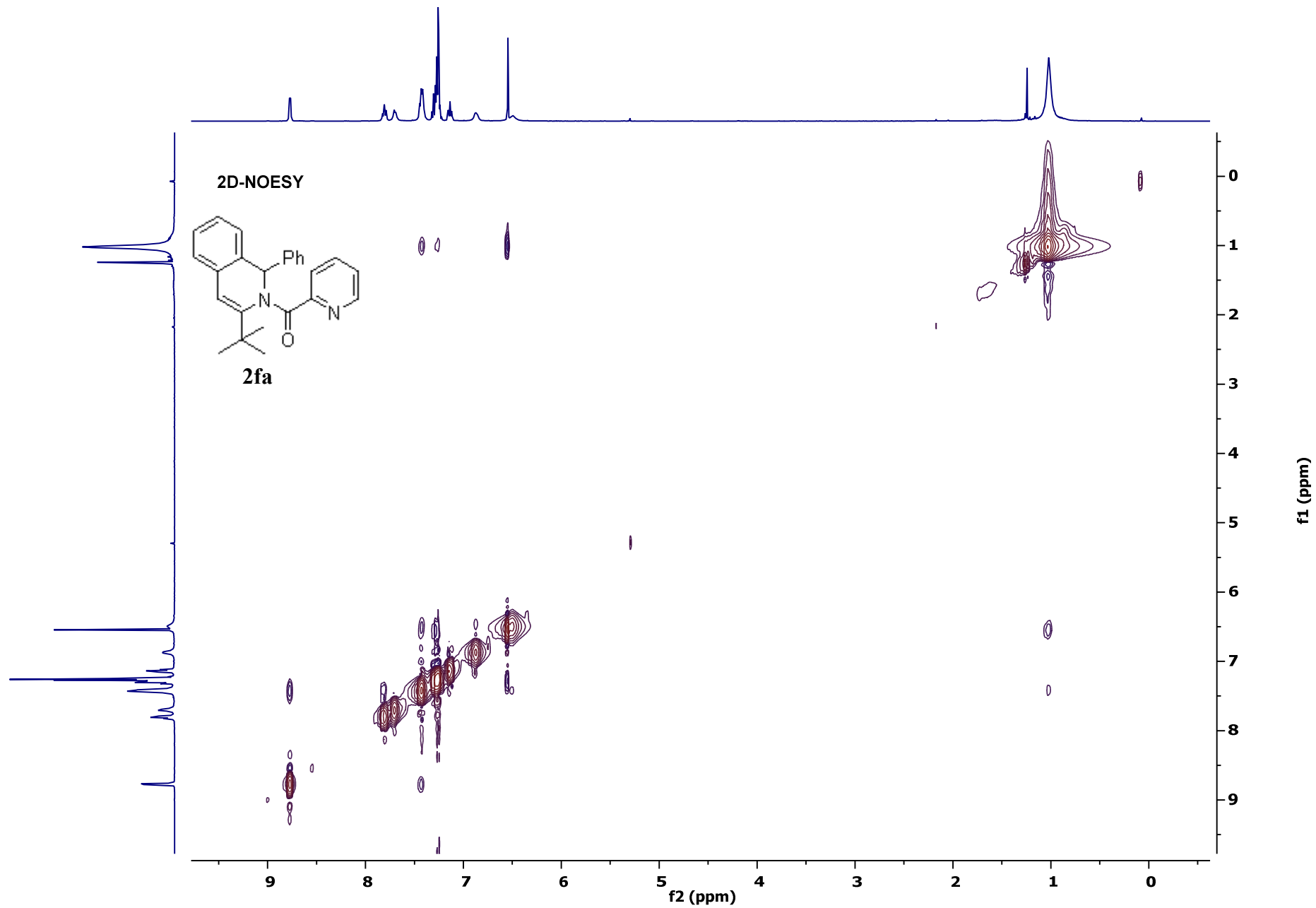


**2fa**

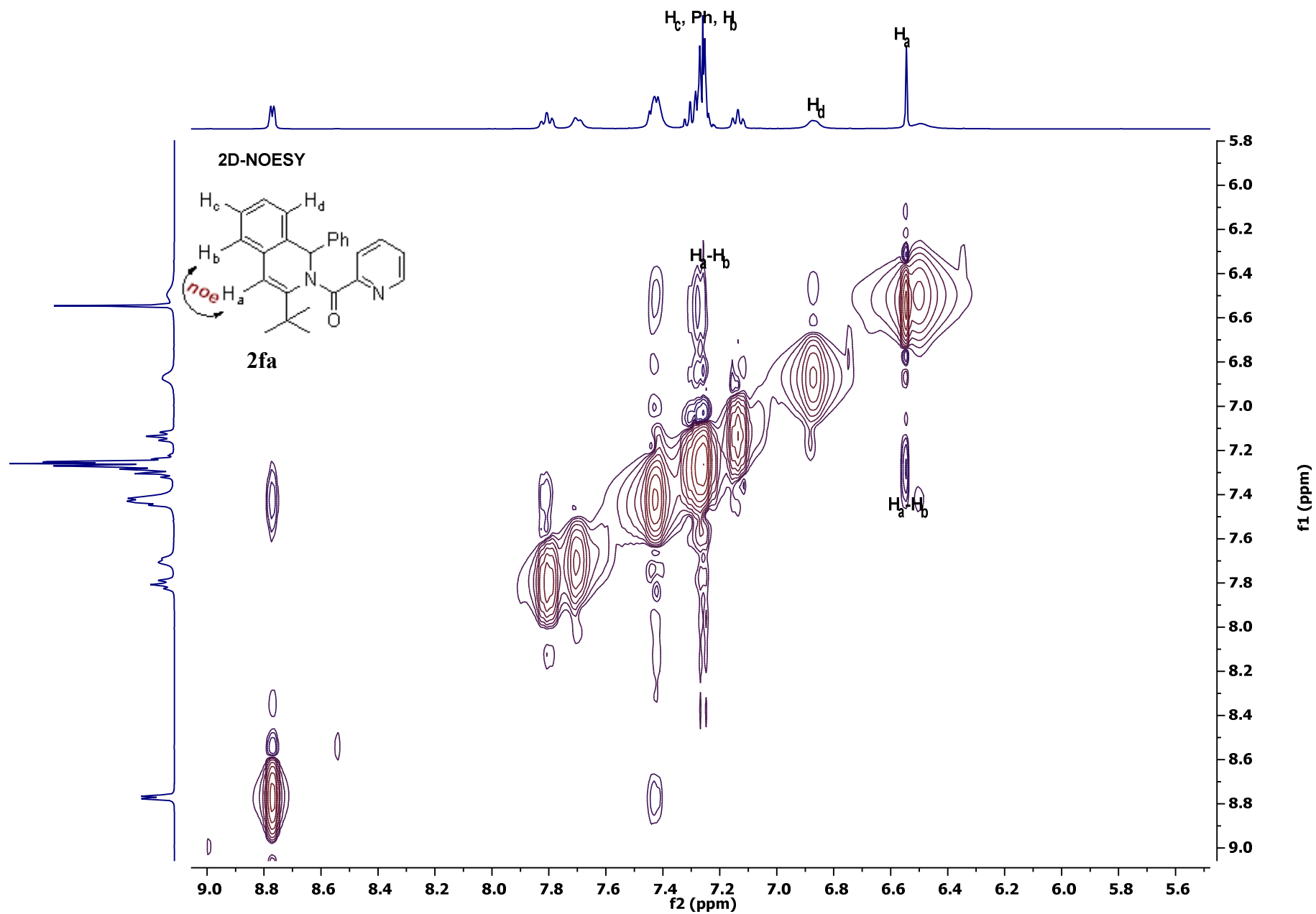




2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)

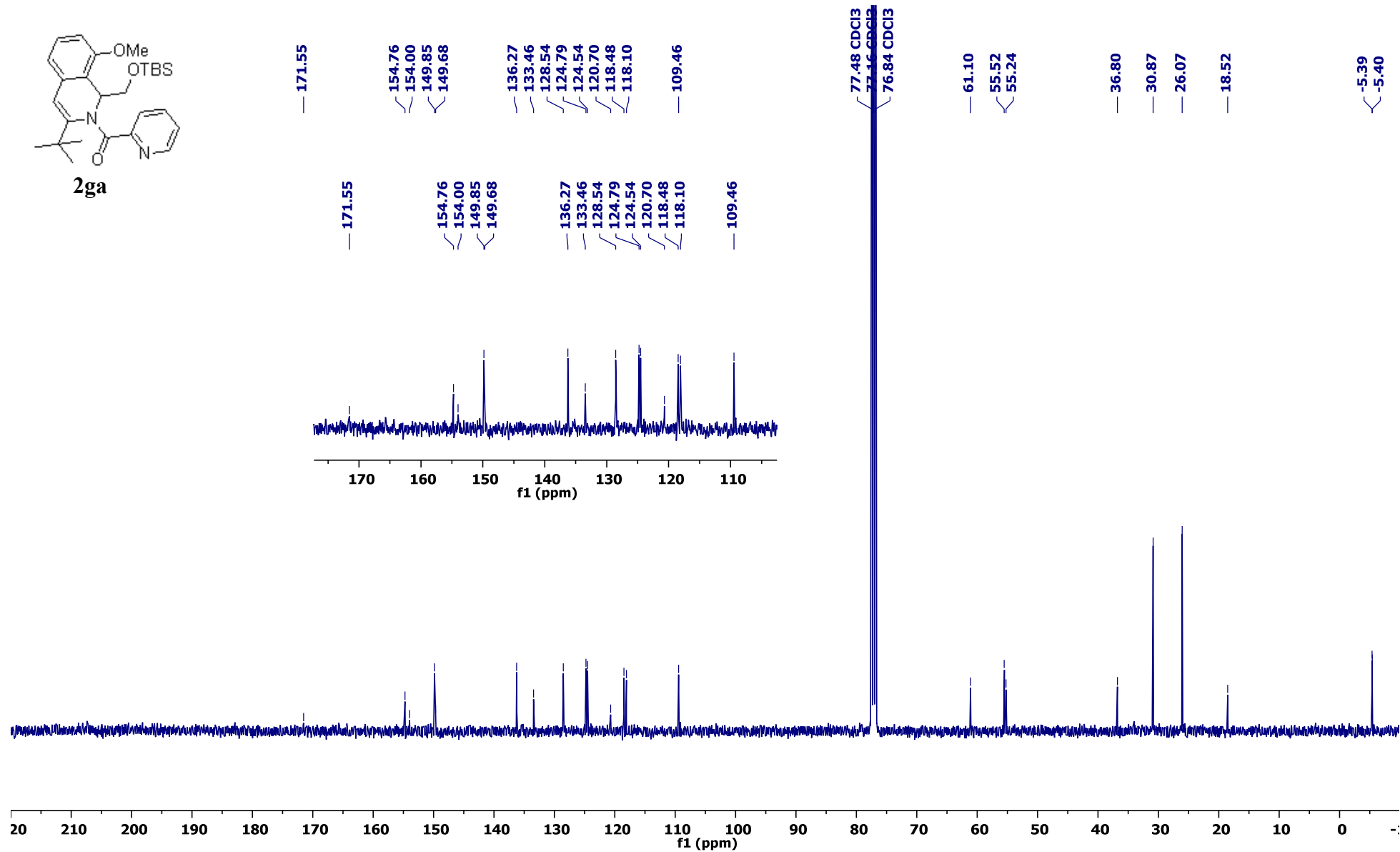
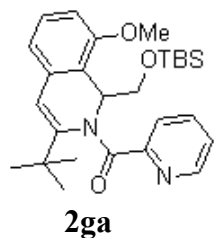


2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)

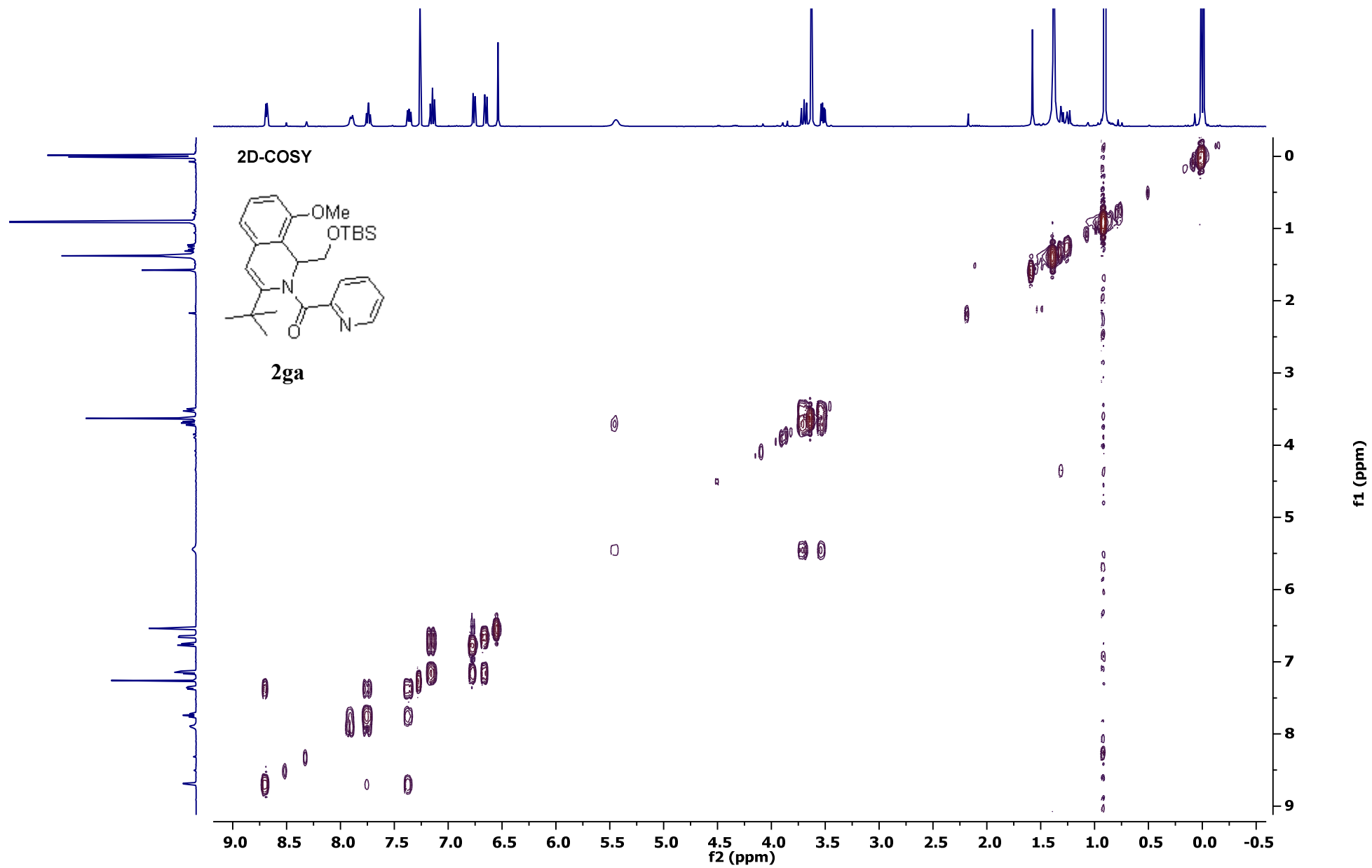




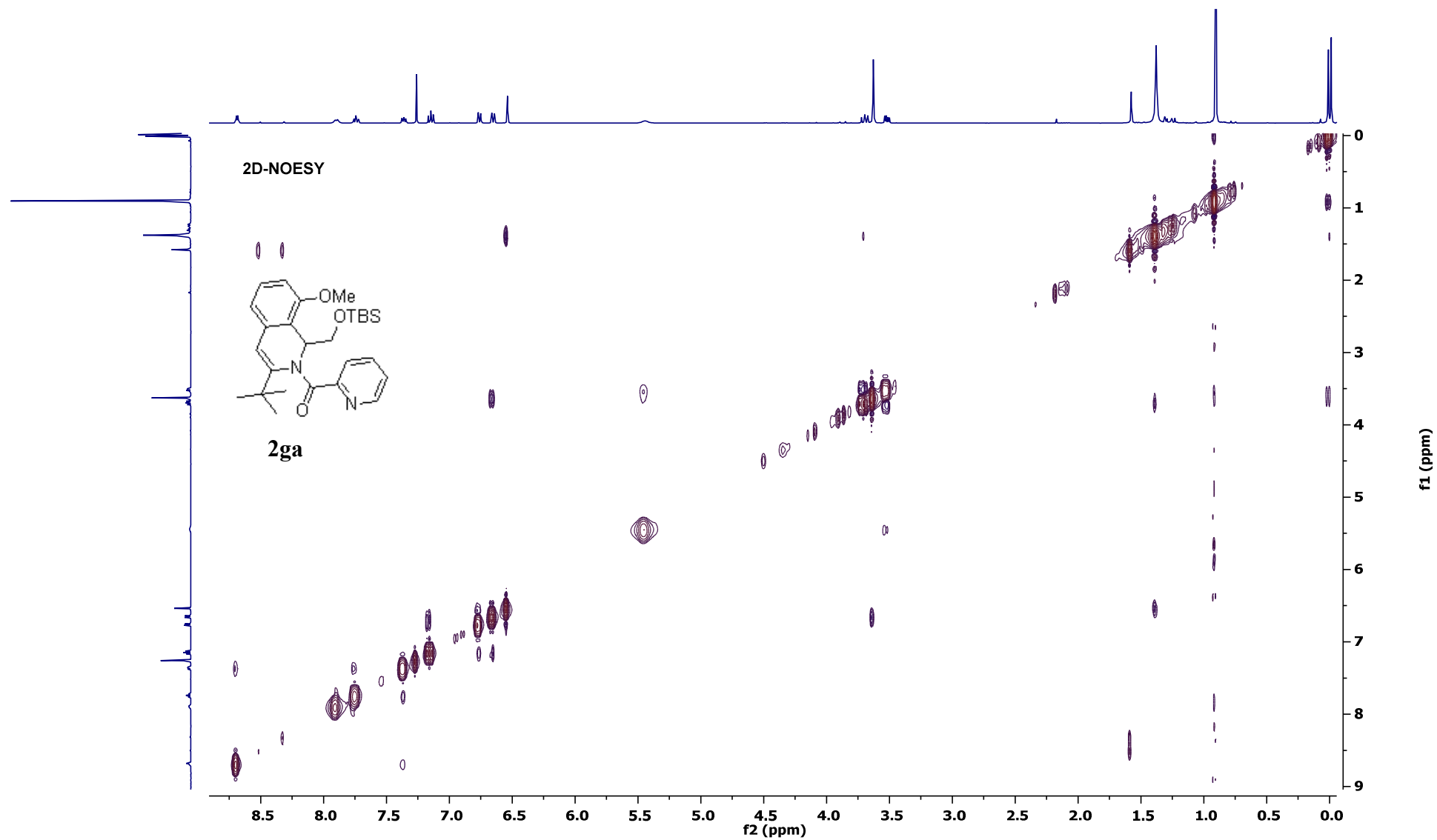
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



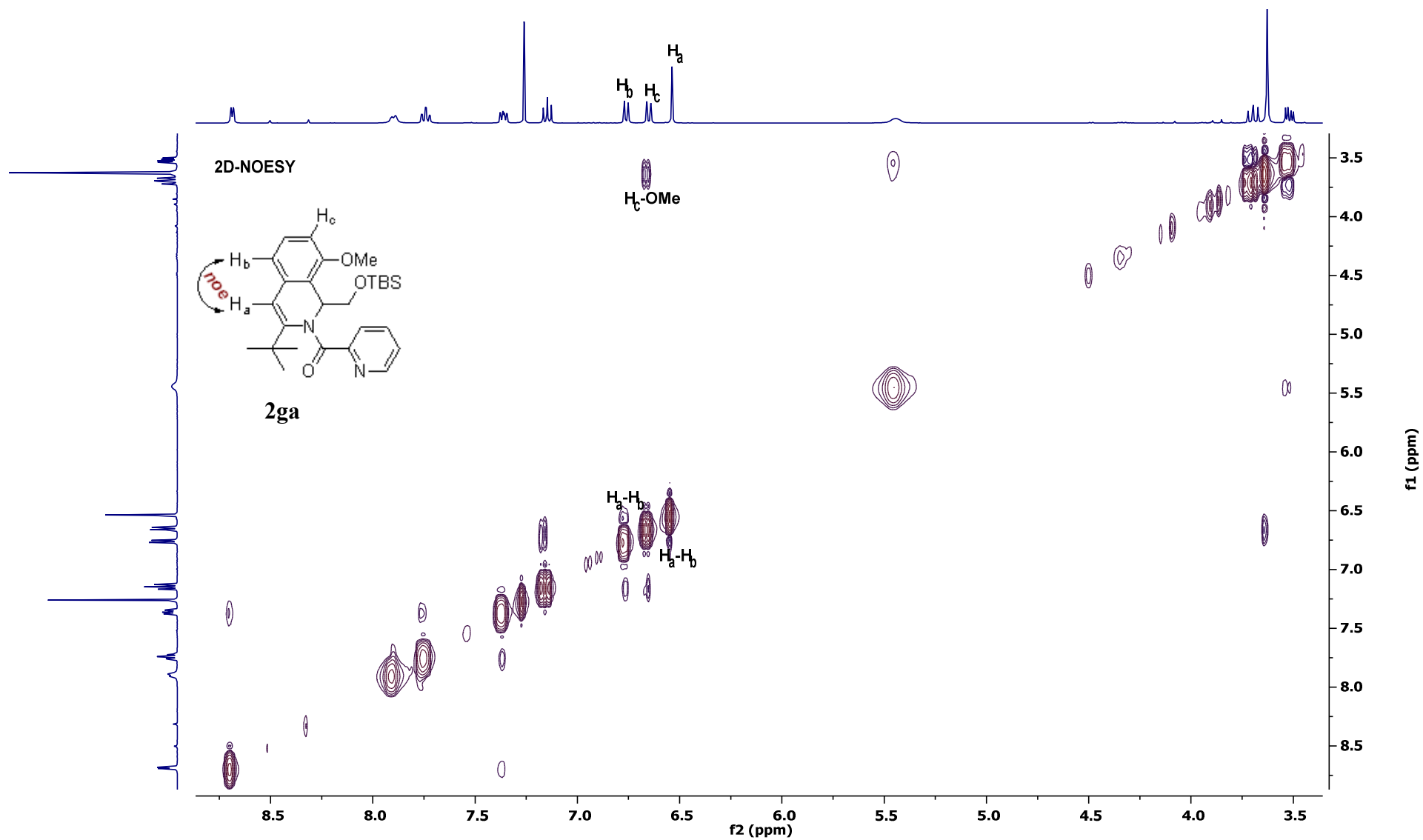
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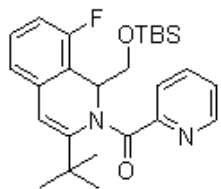
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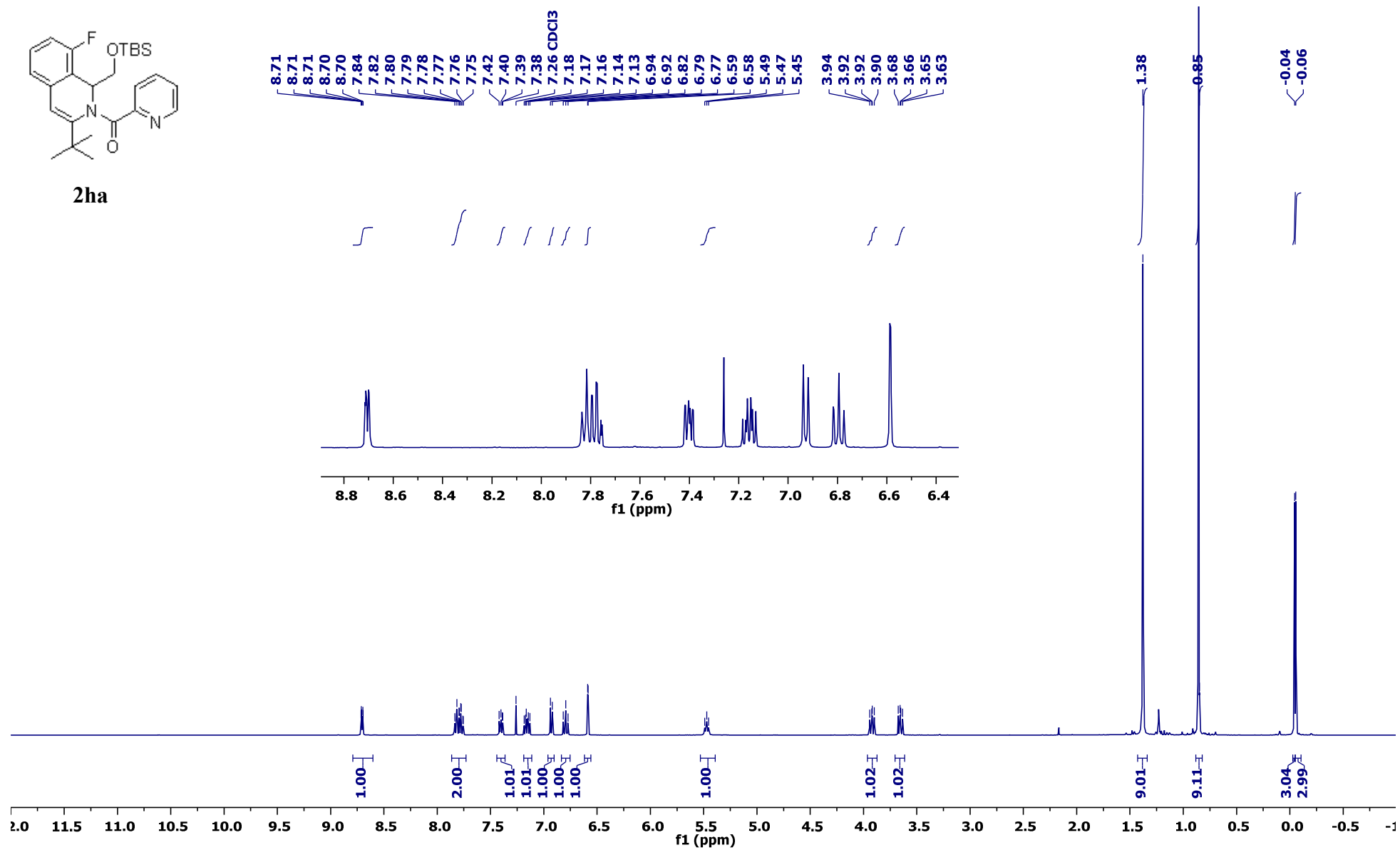
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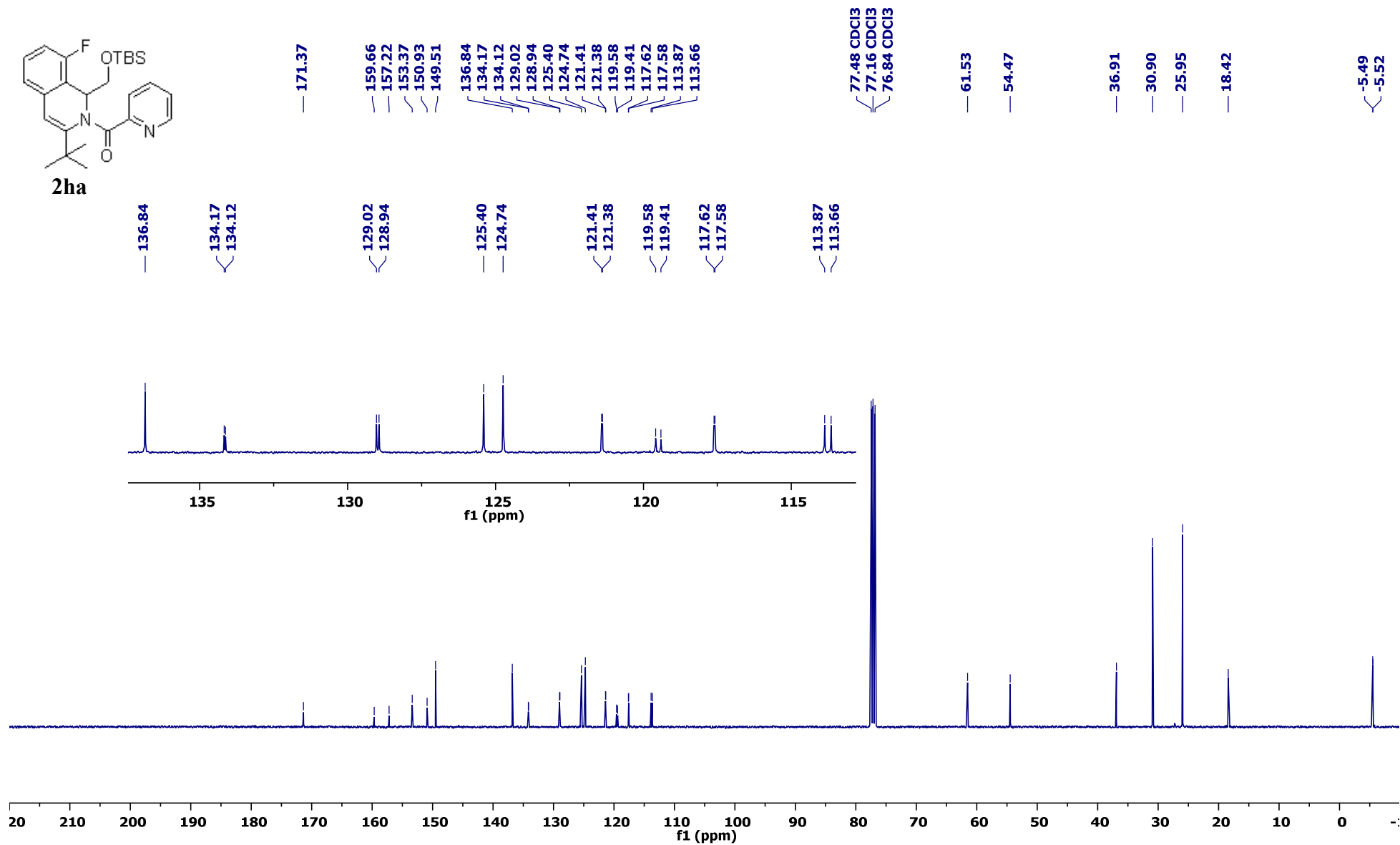
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



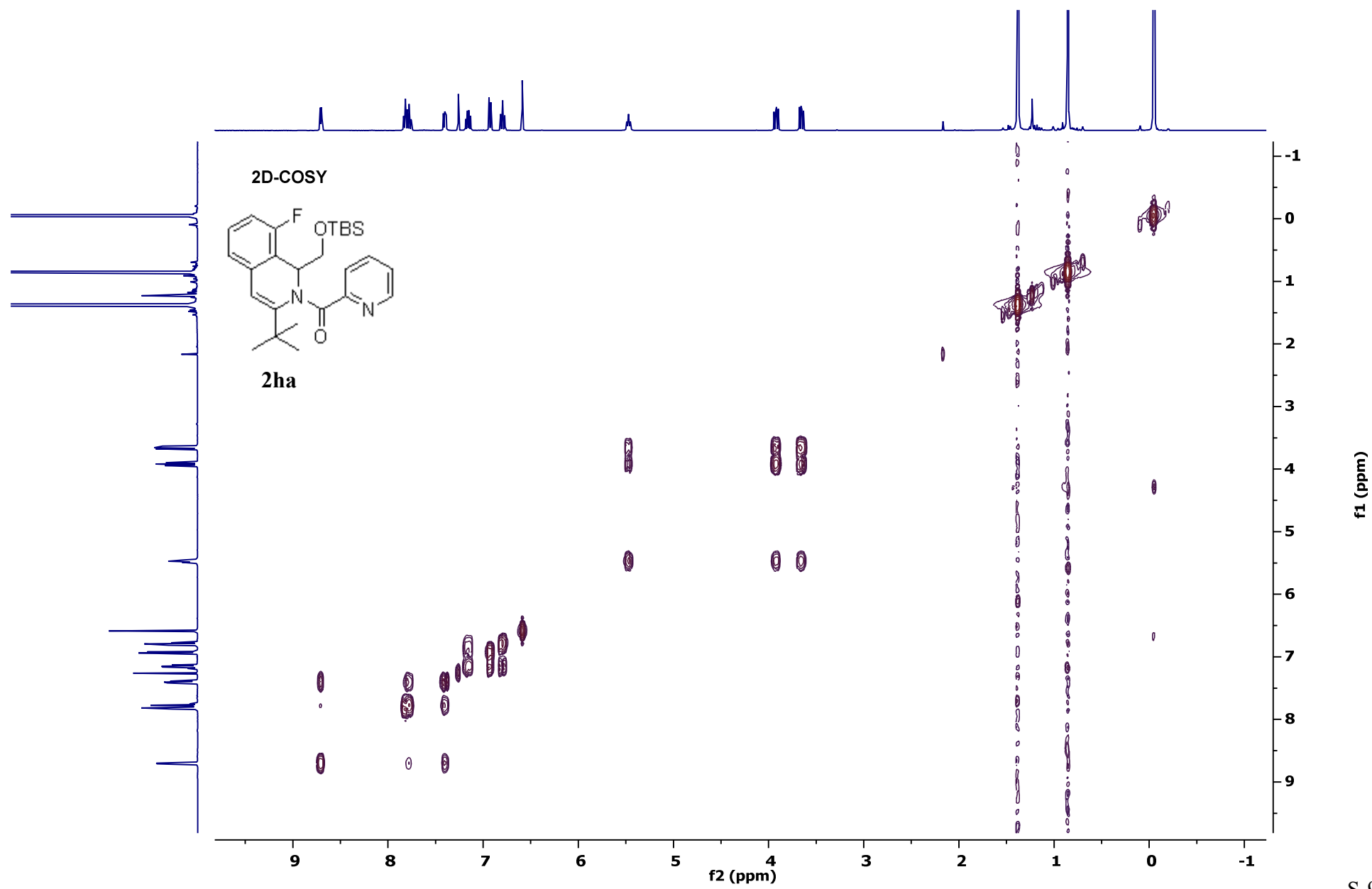
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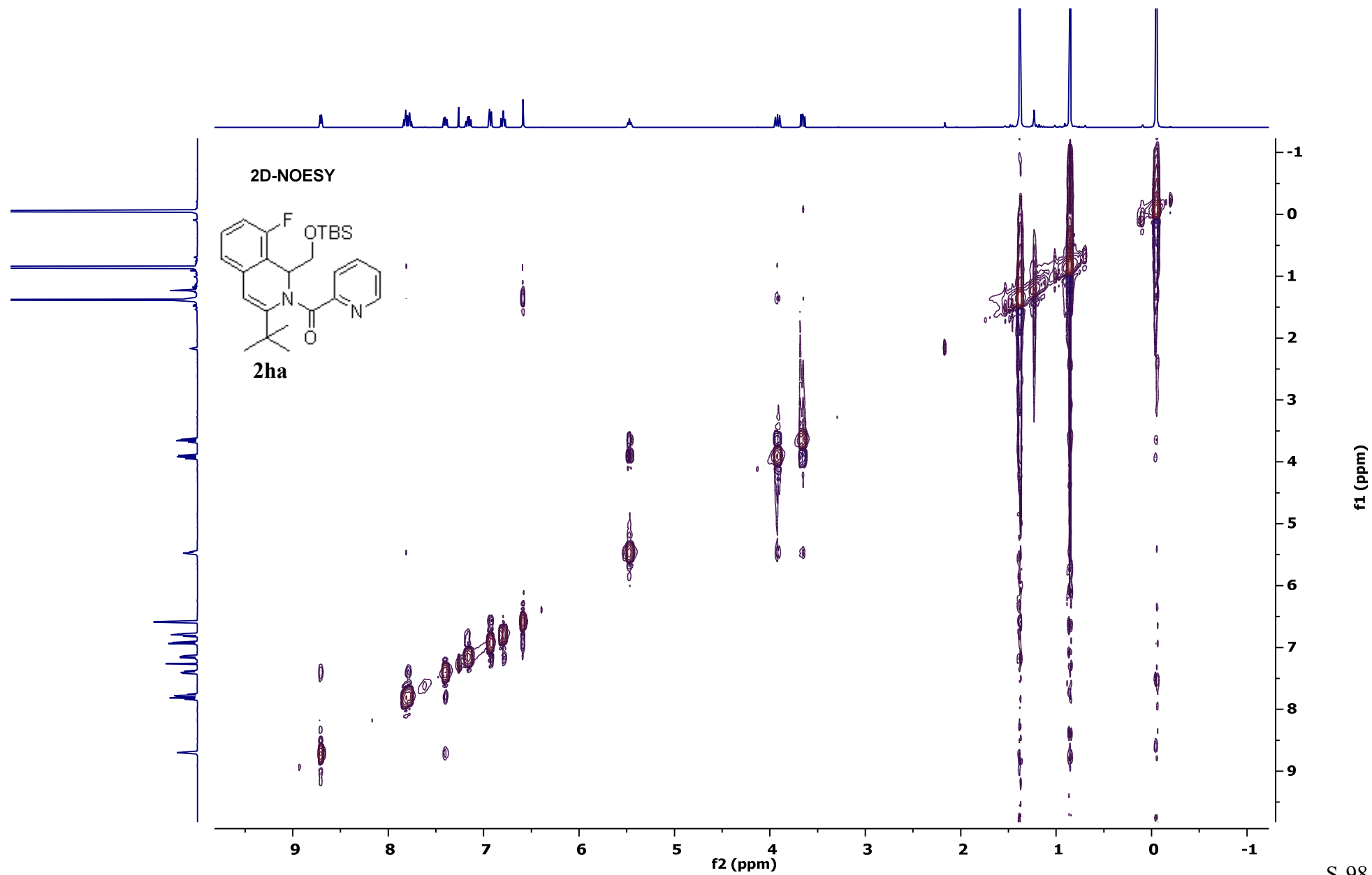
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



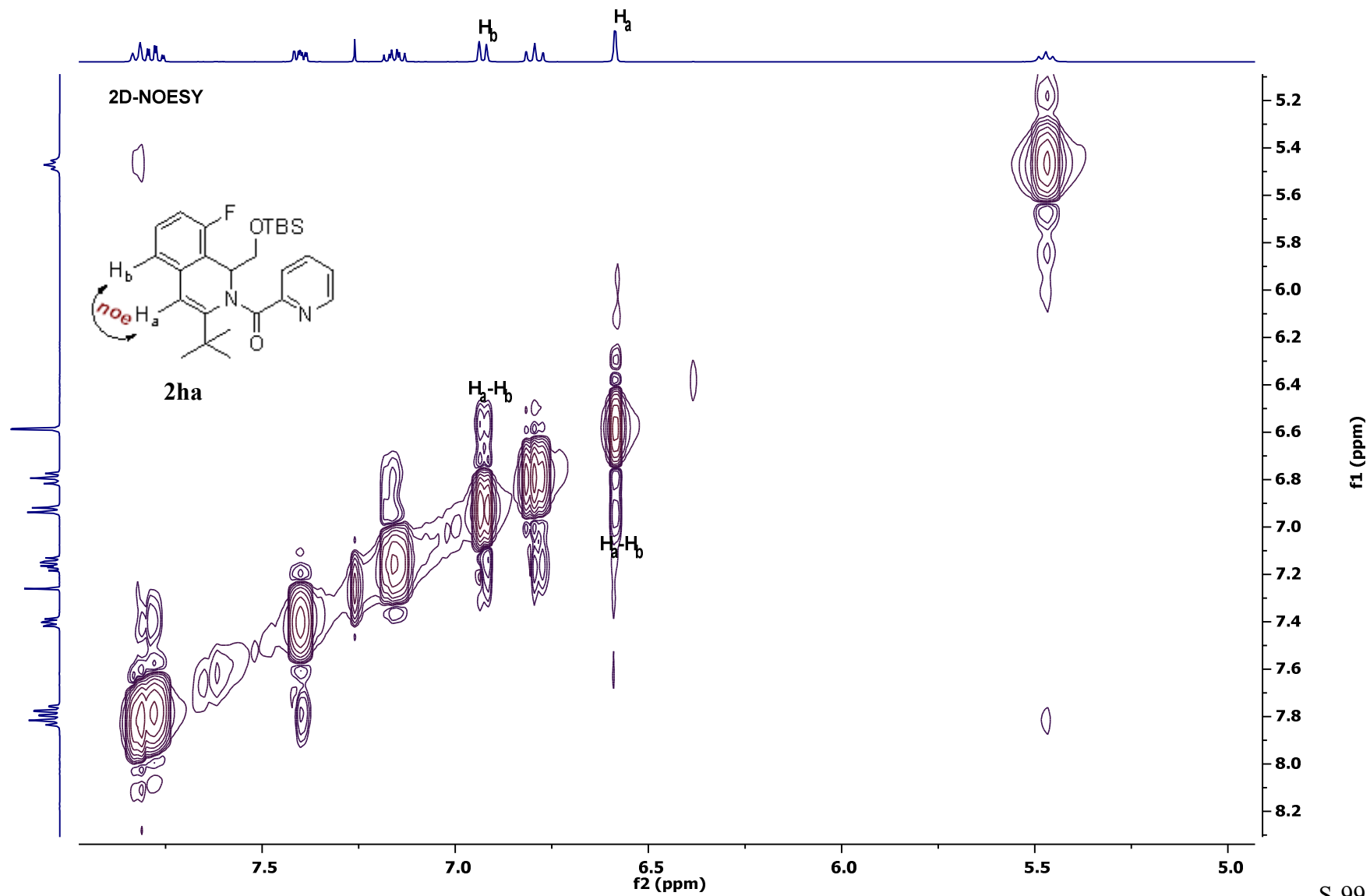
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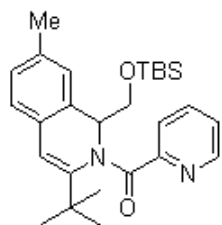
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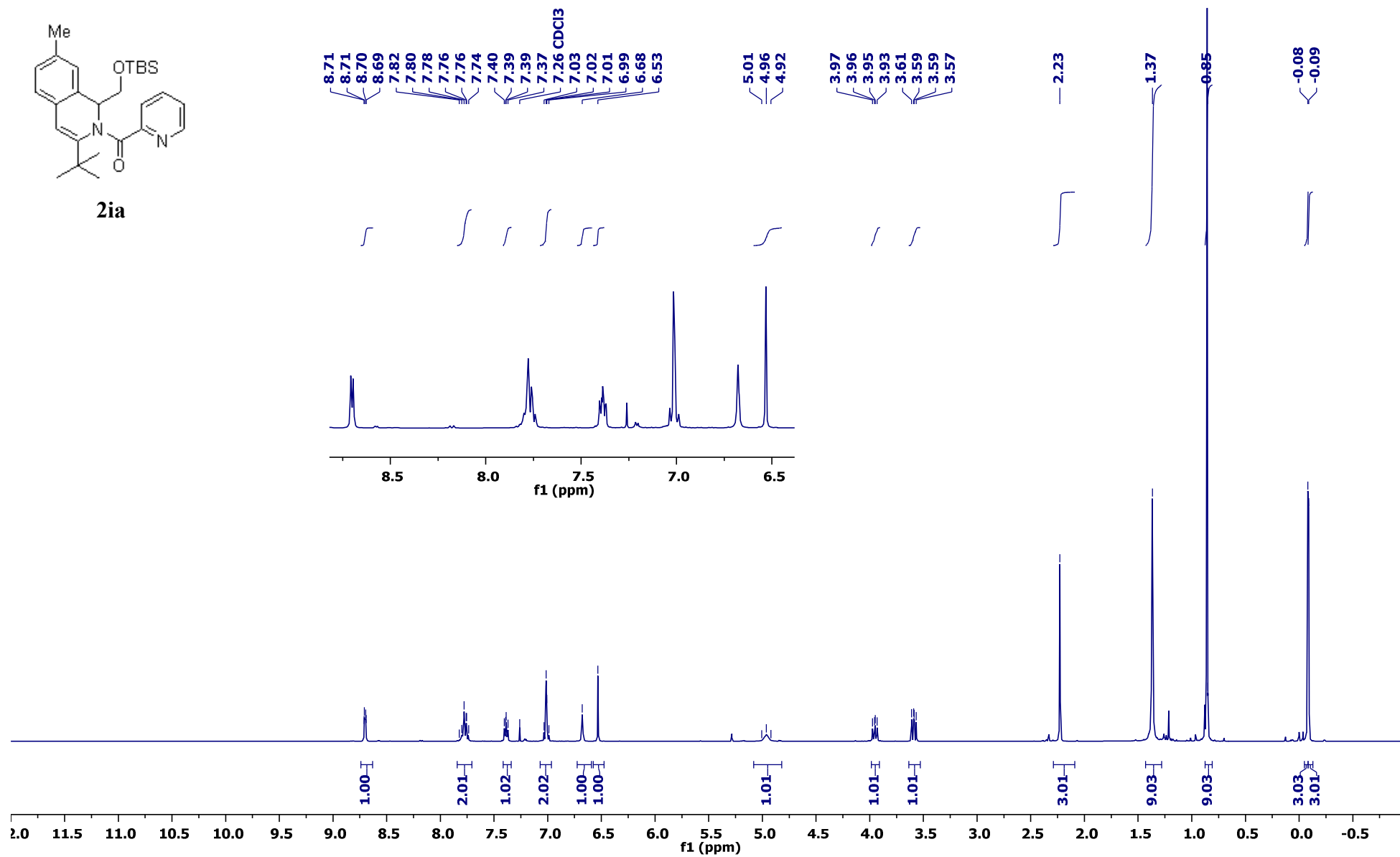
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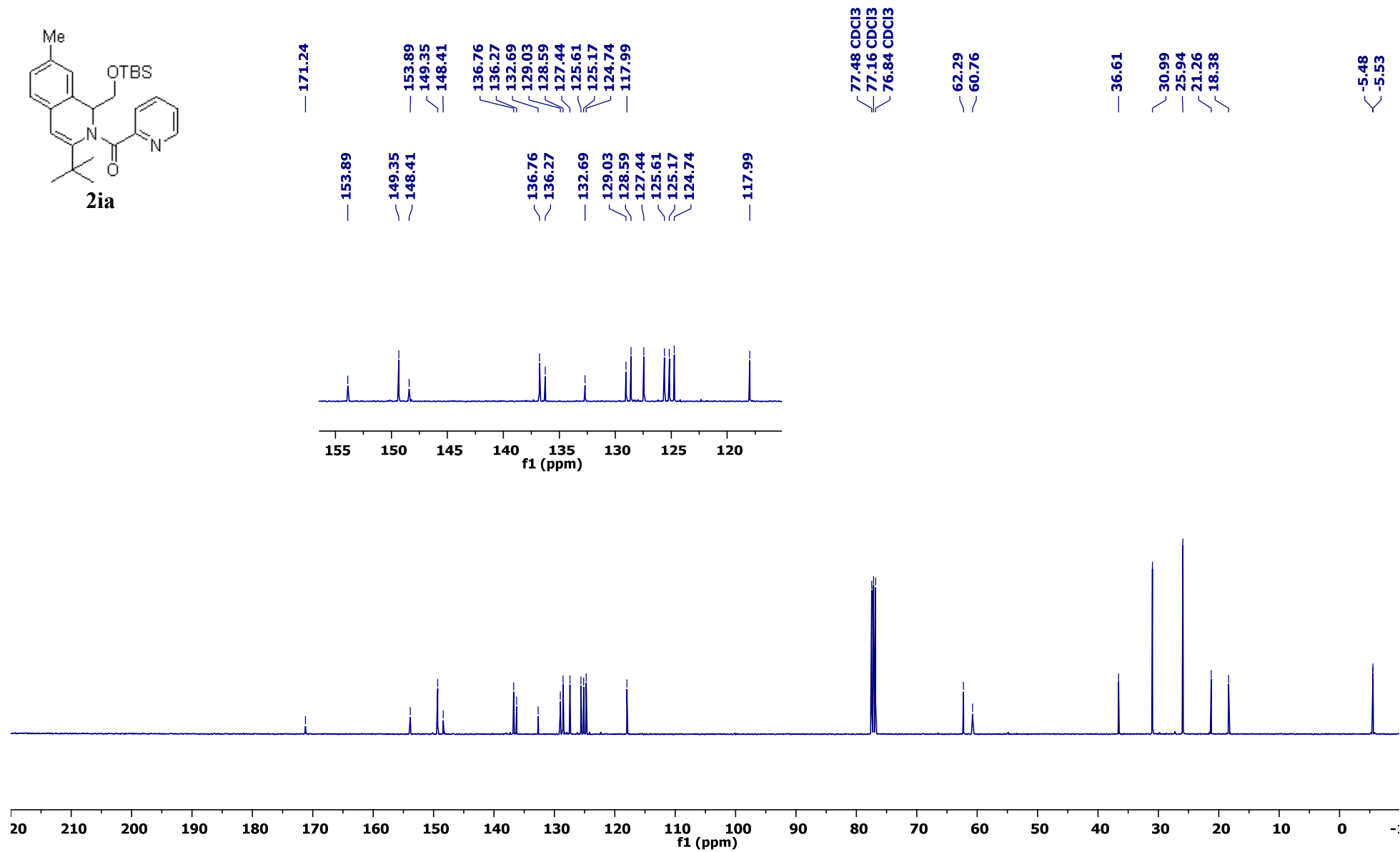
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

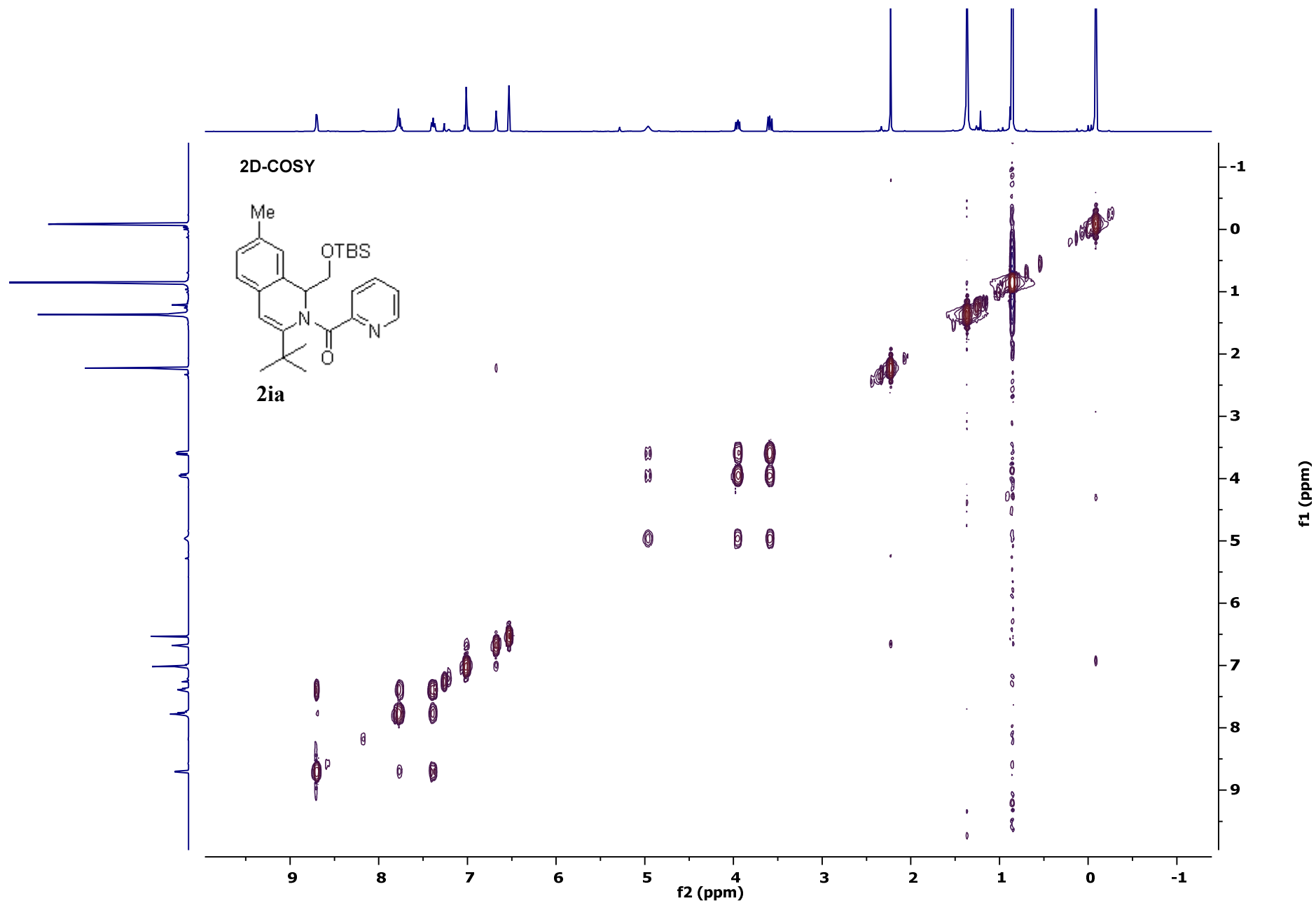


**2ia**

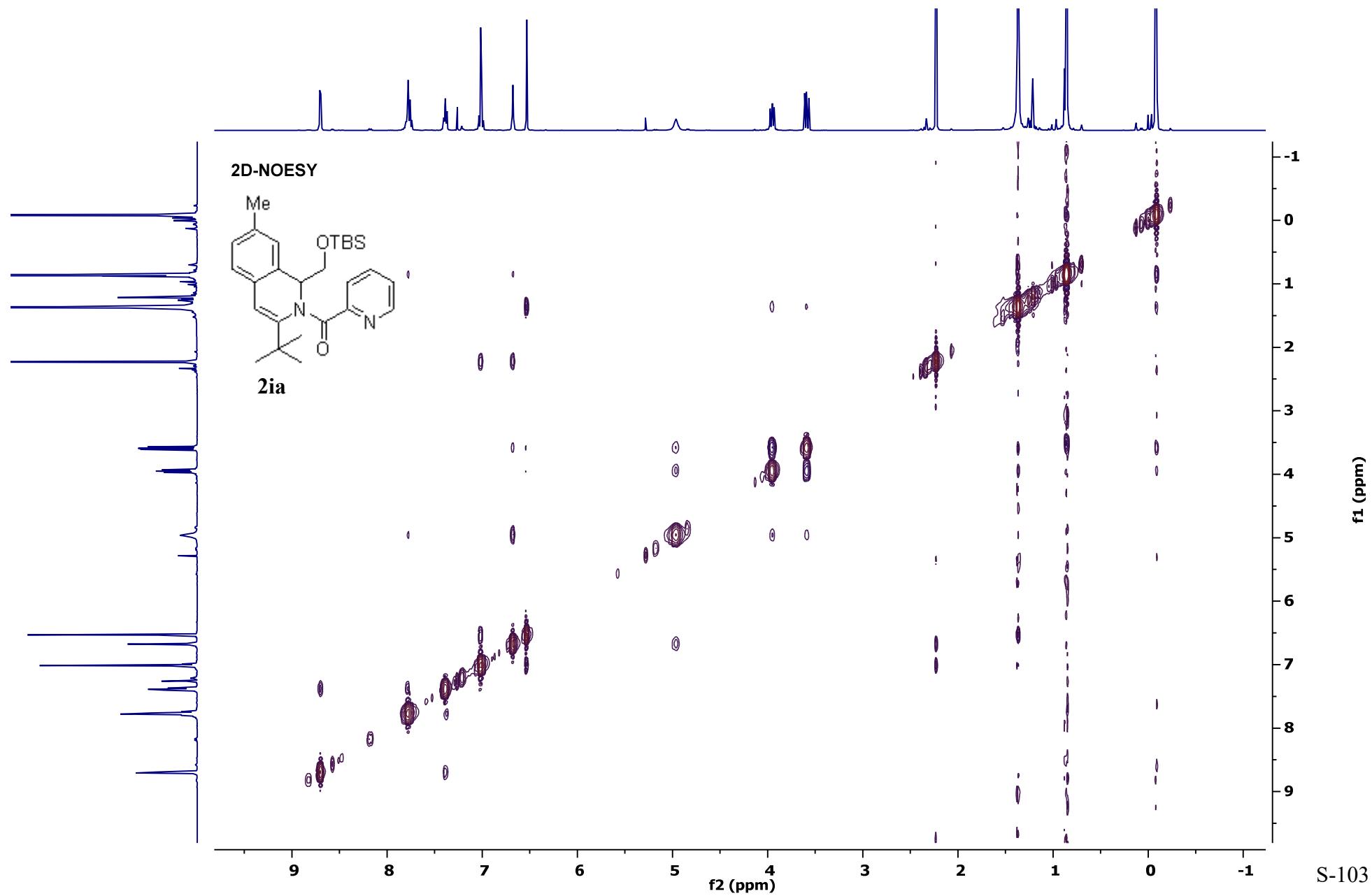


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

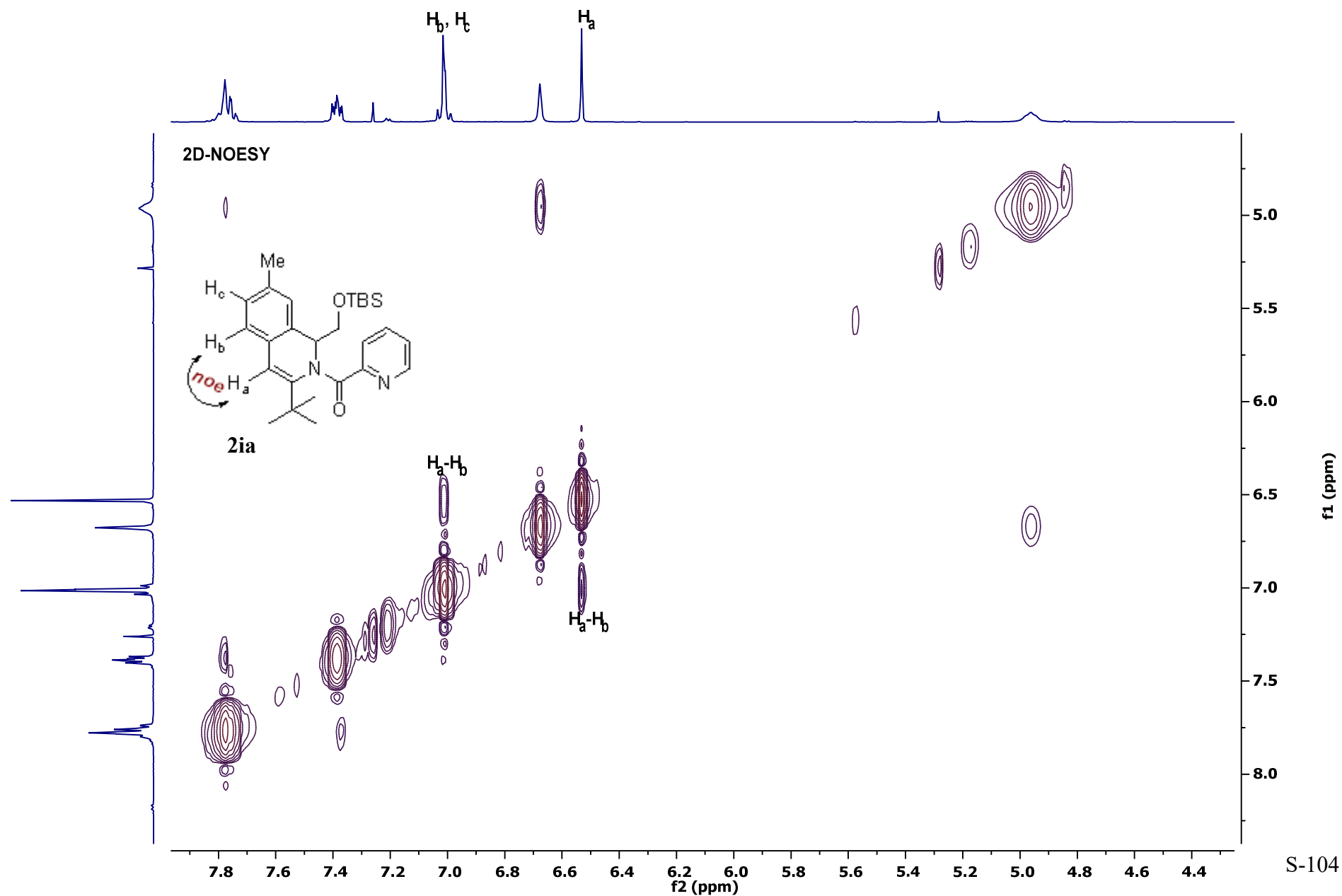




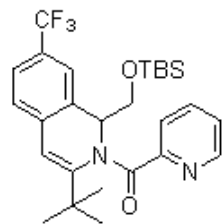
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



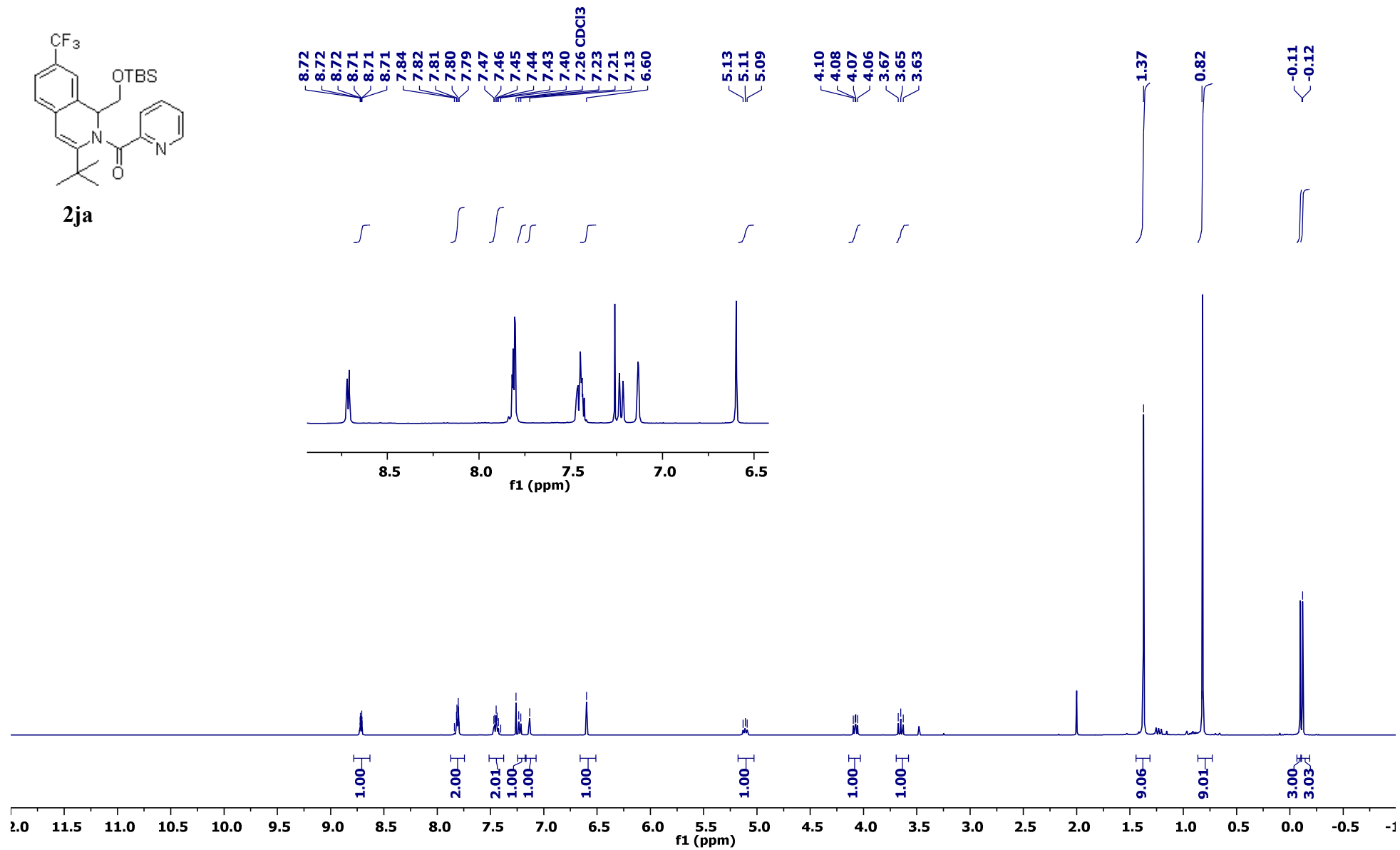
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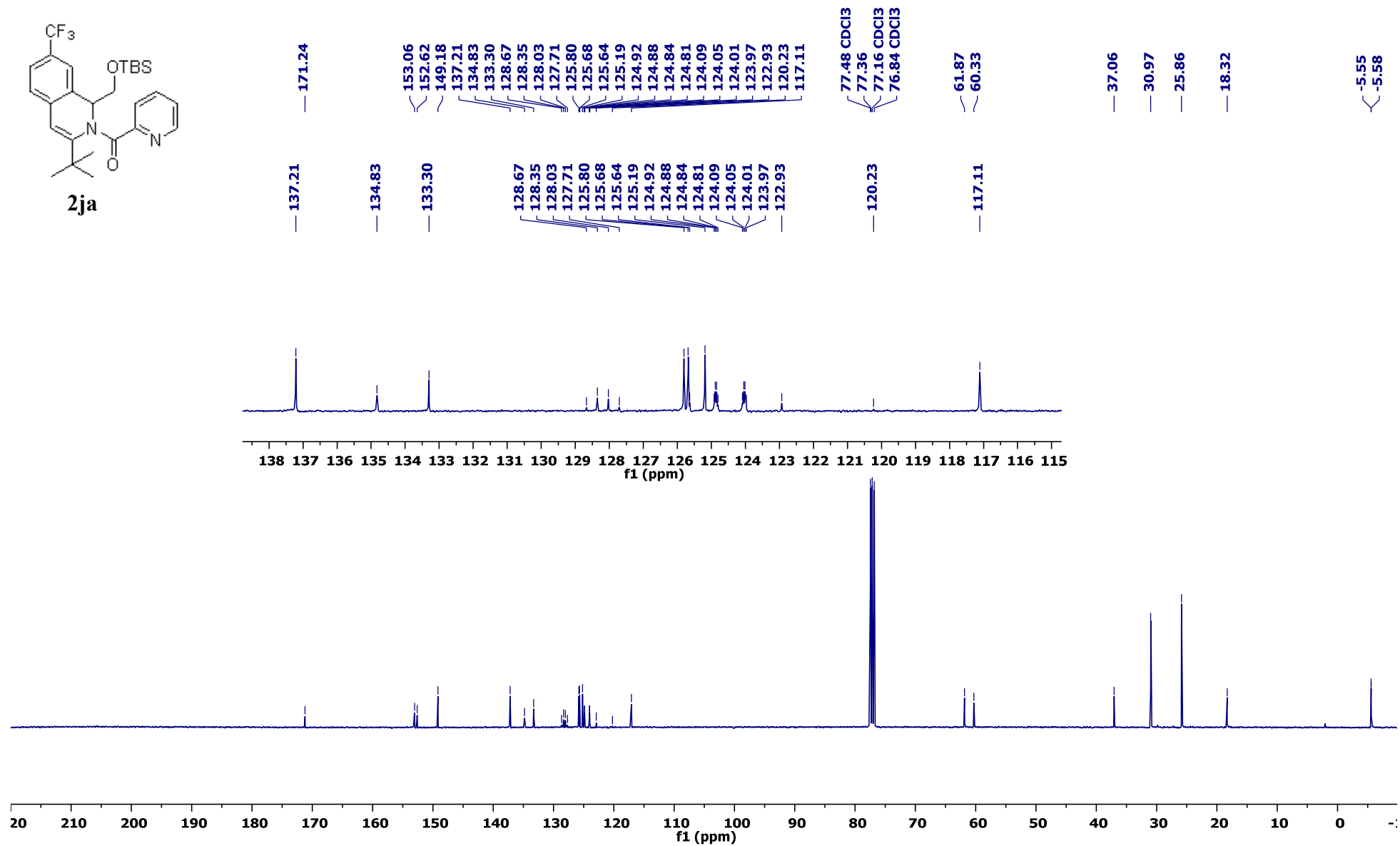
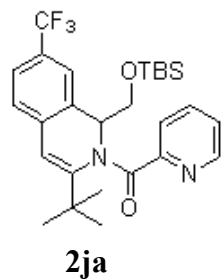
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



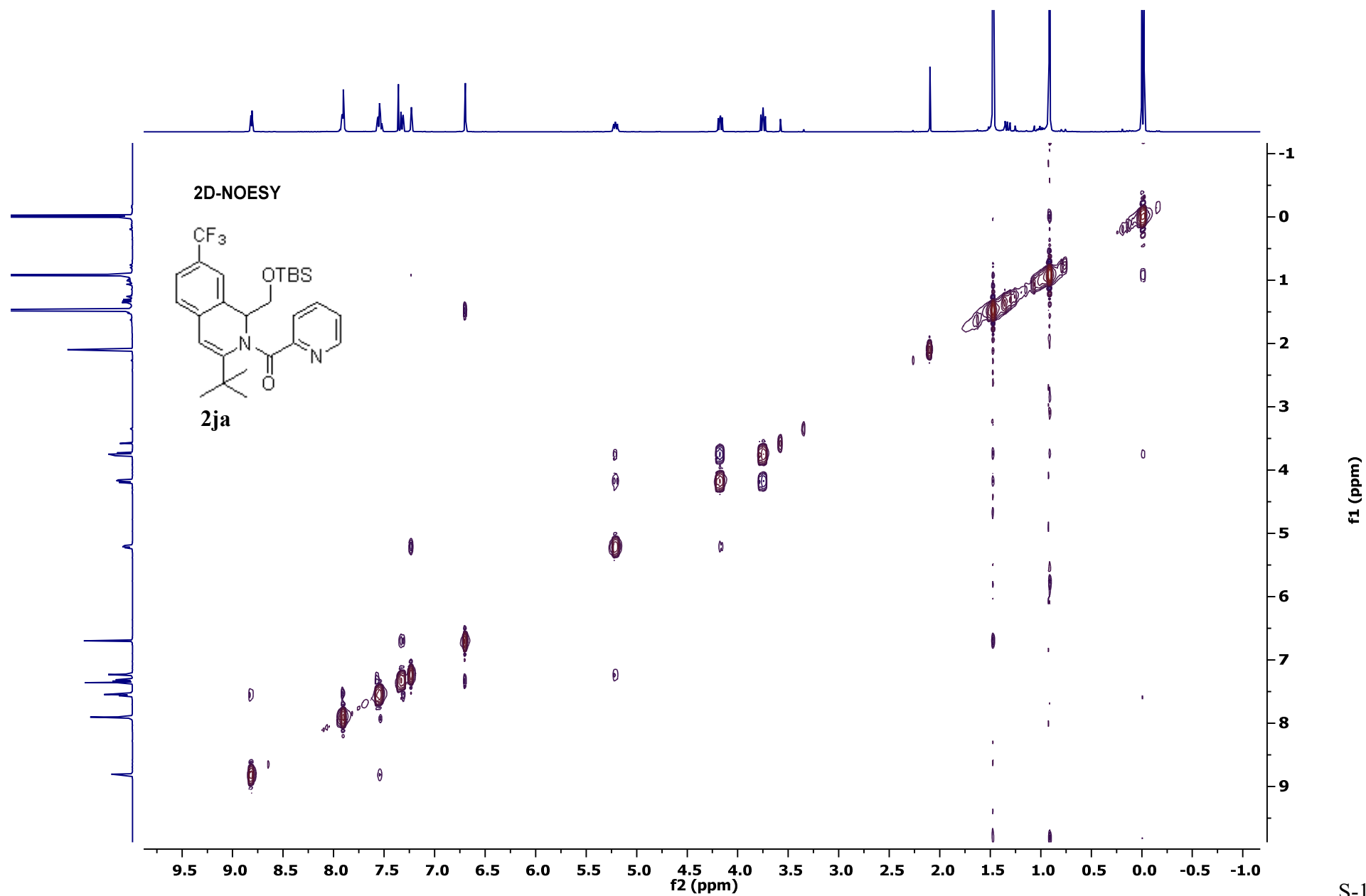
**2ja**



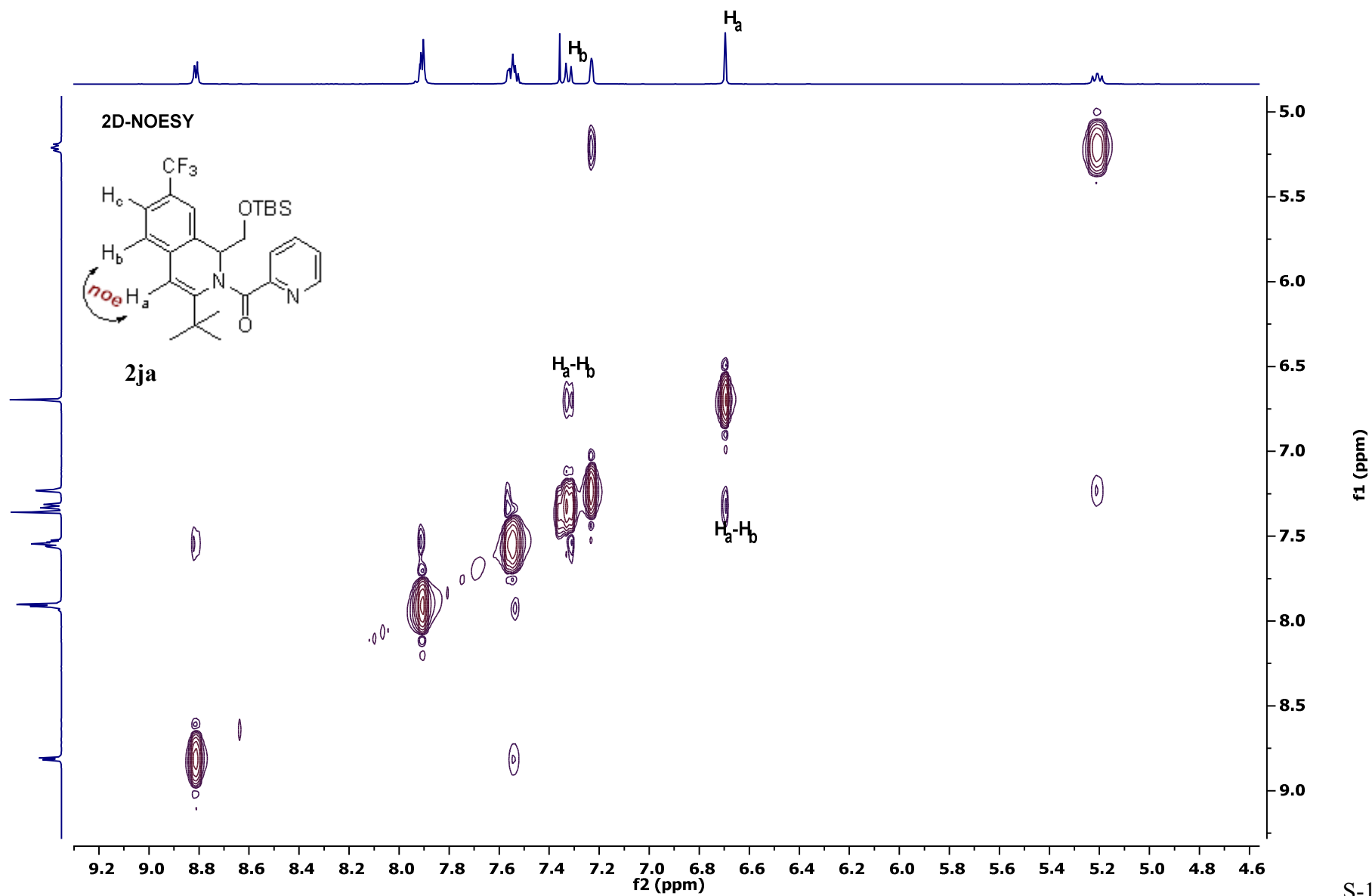
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



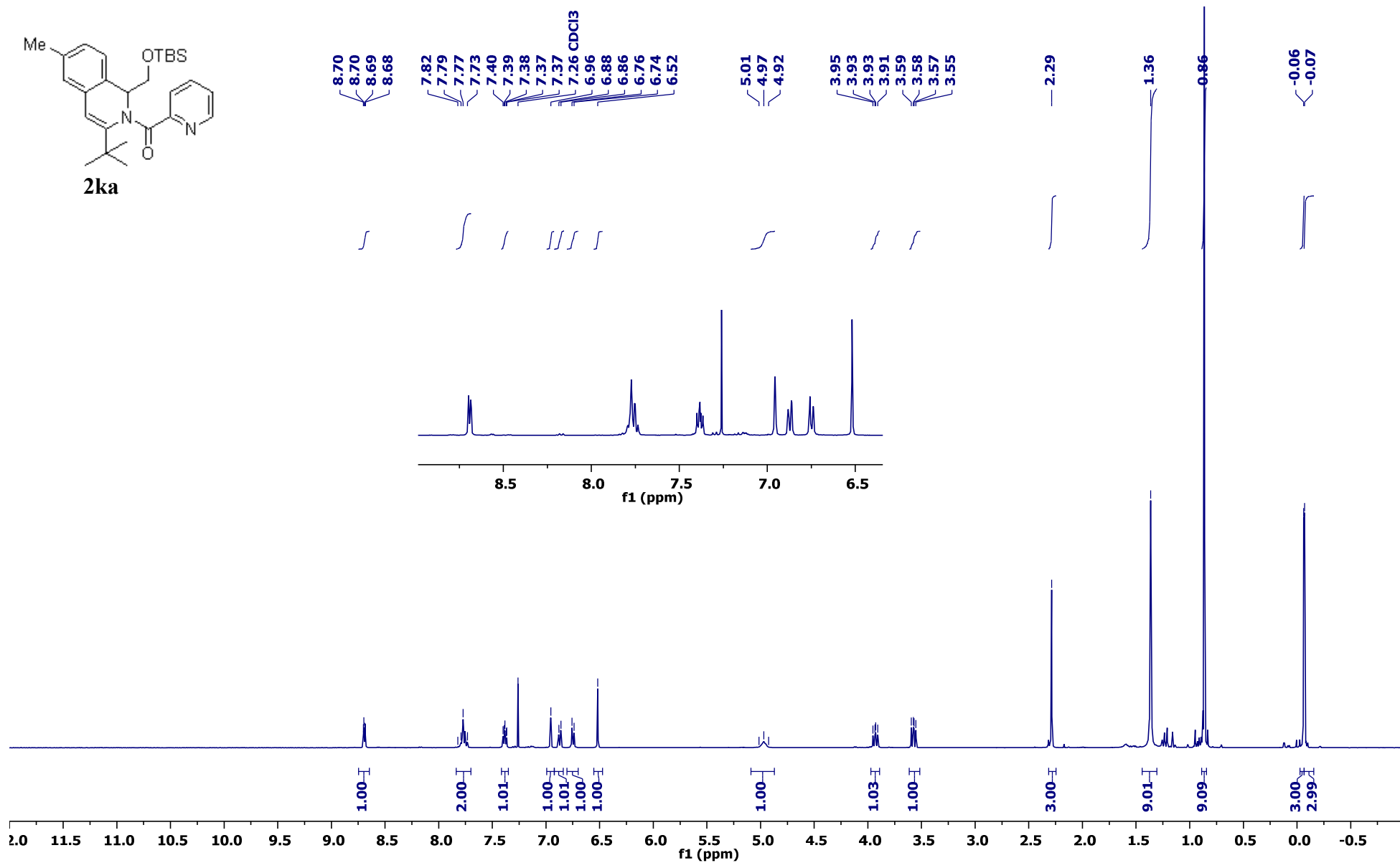




2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)

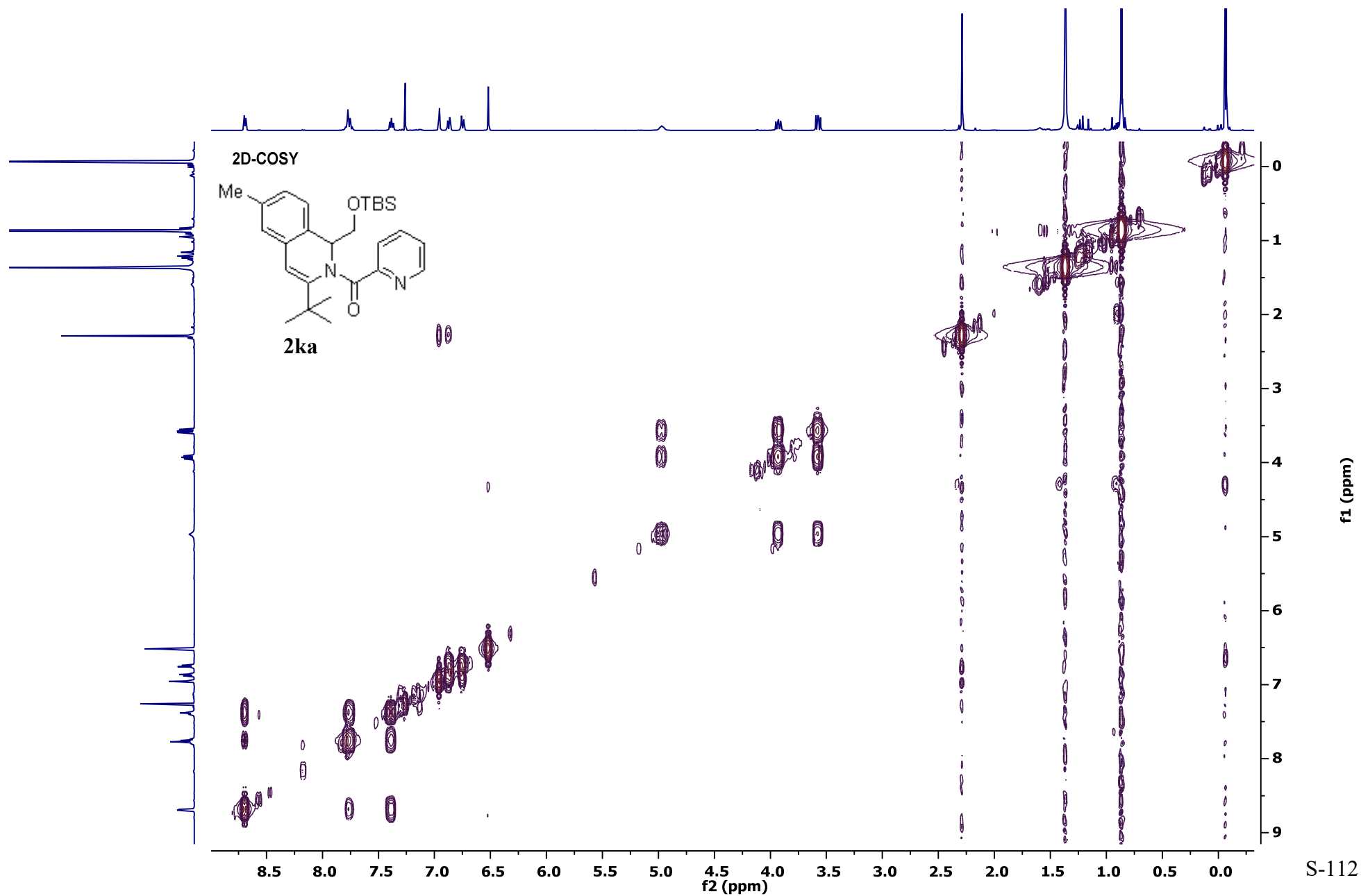


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )

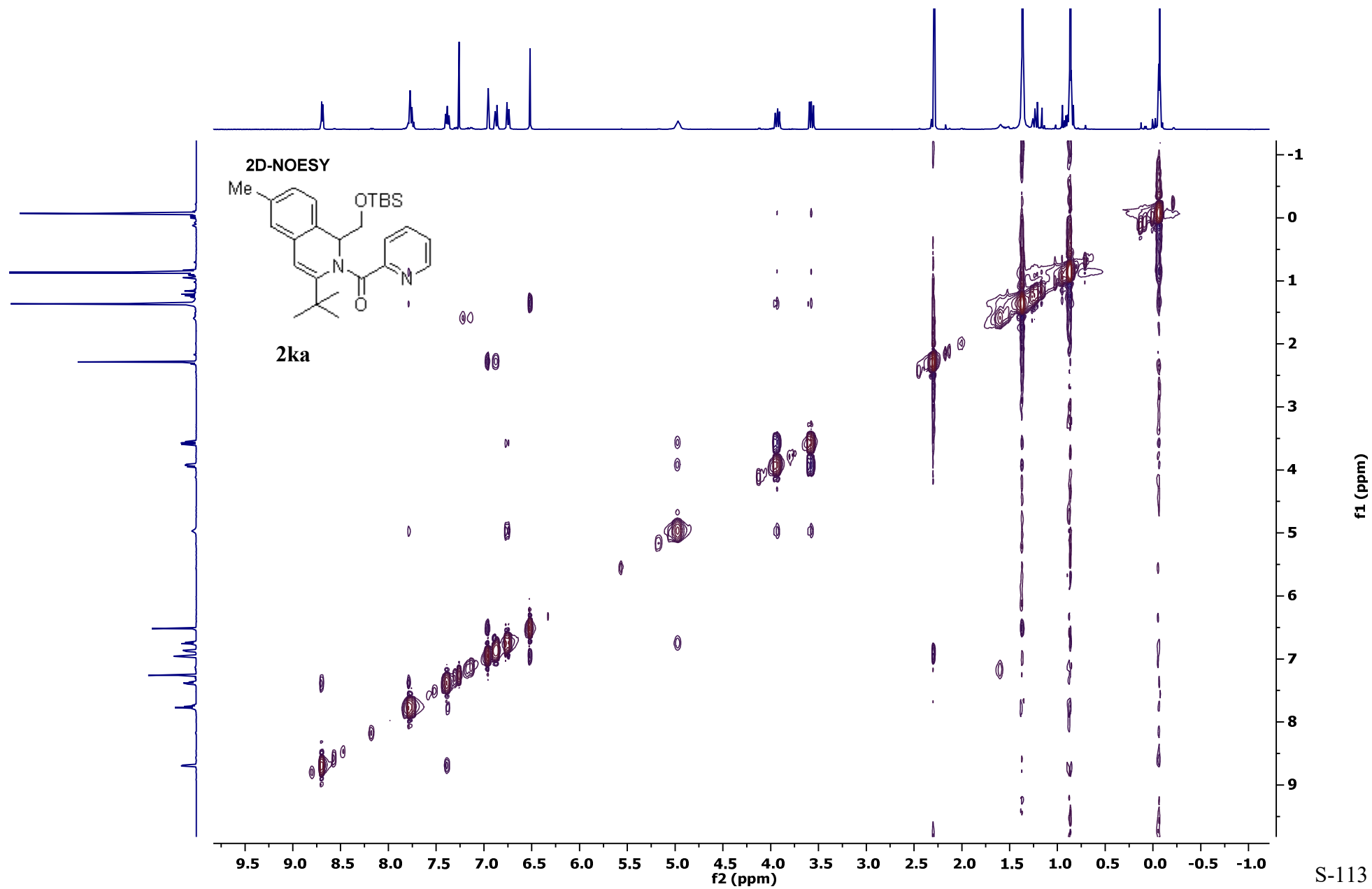




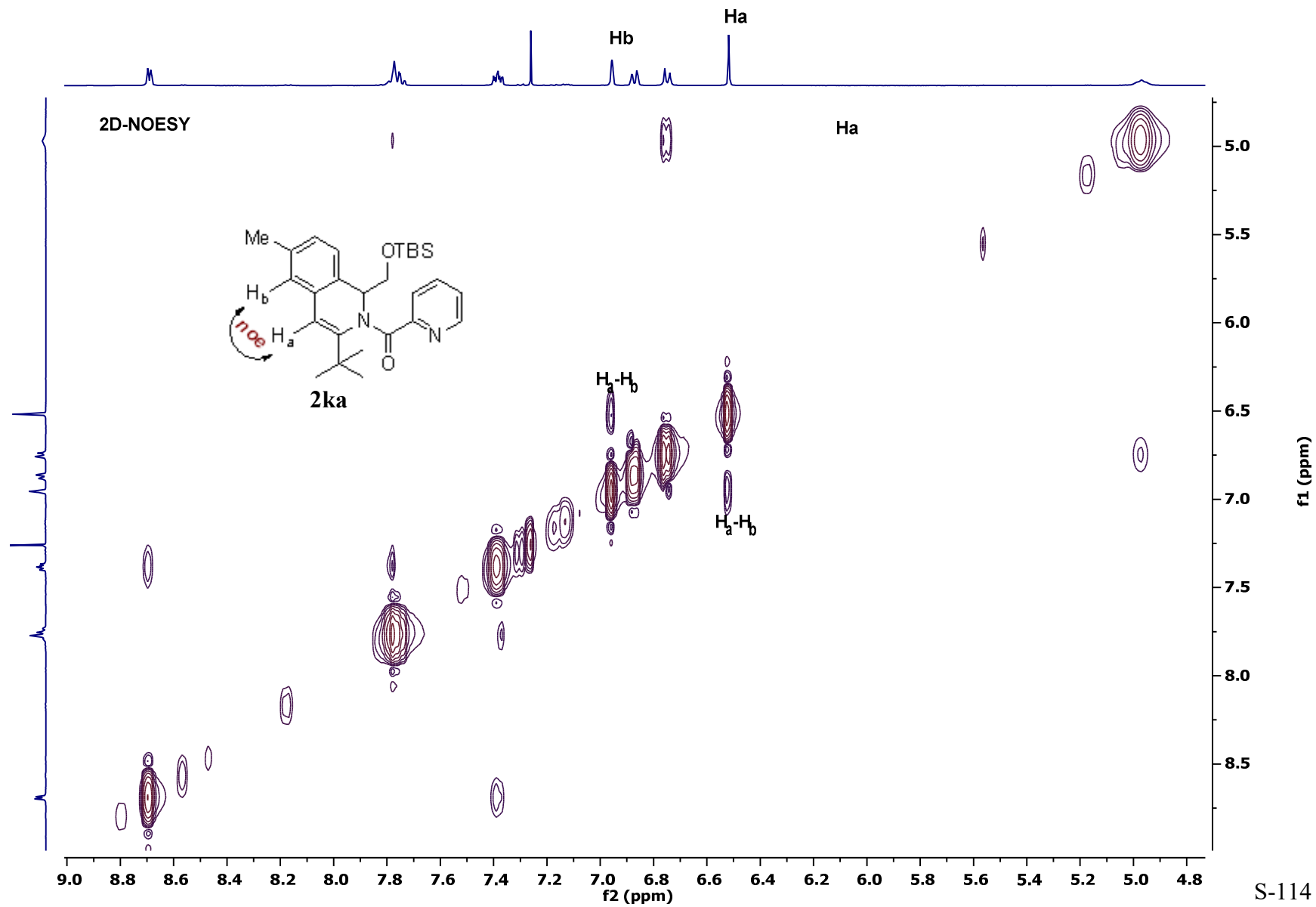
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



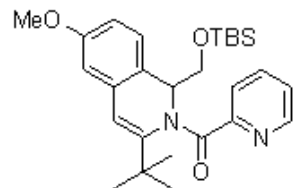
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



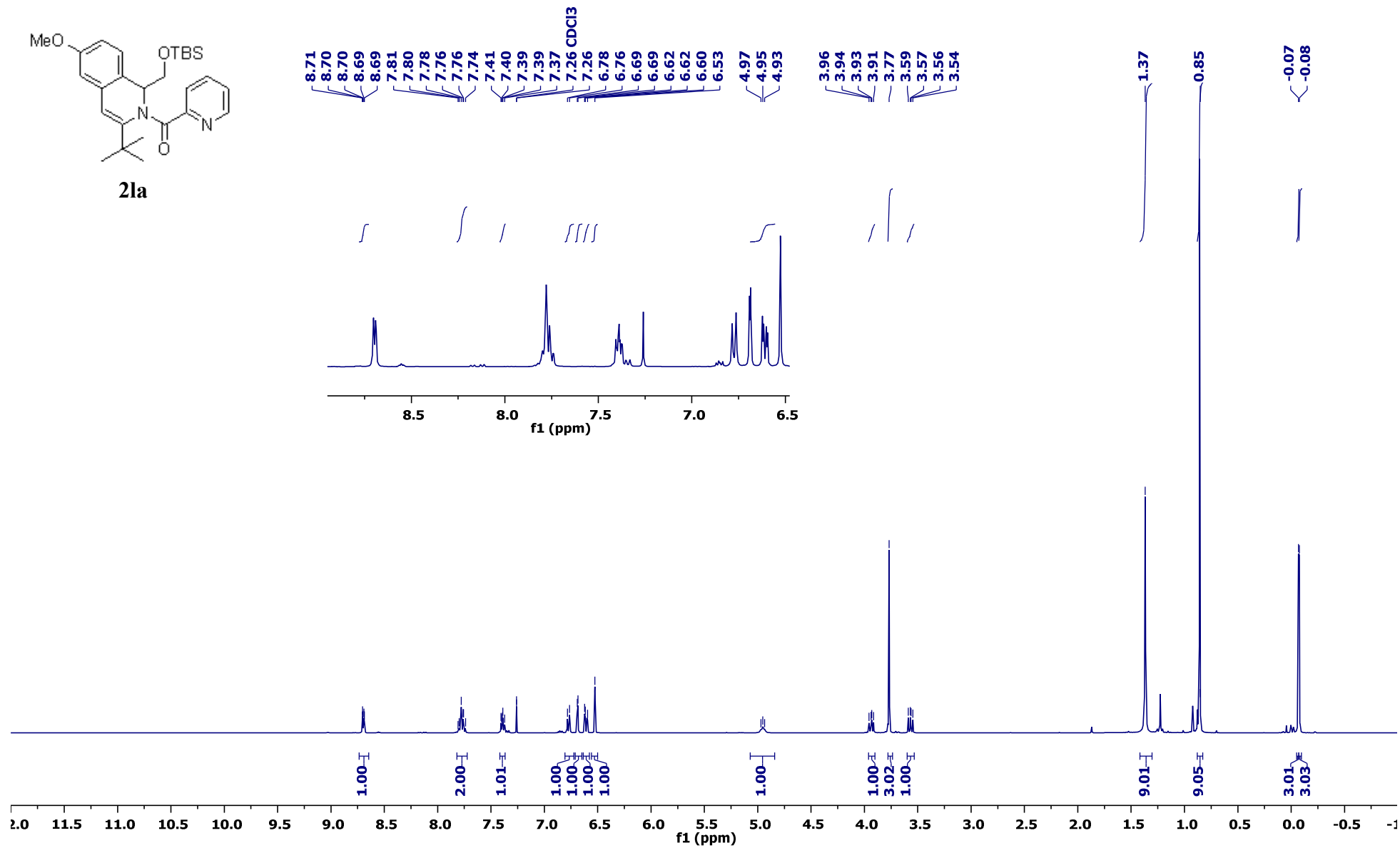
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

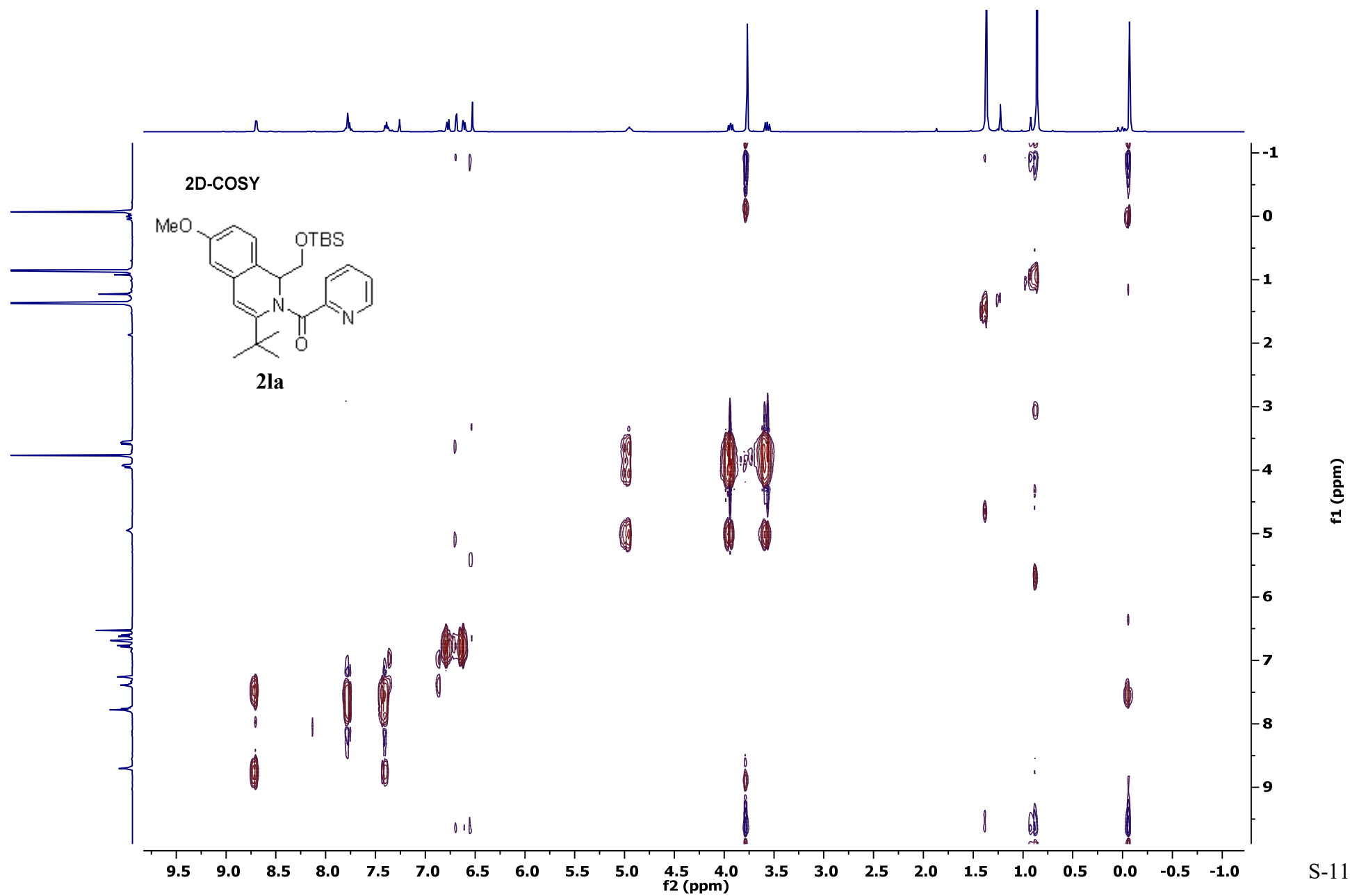


**21a**

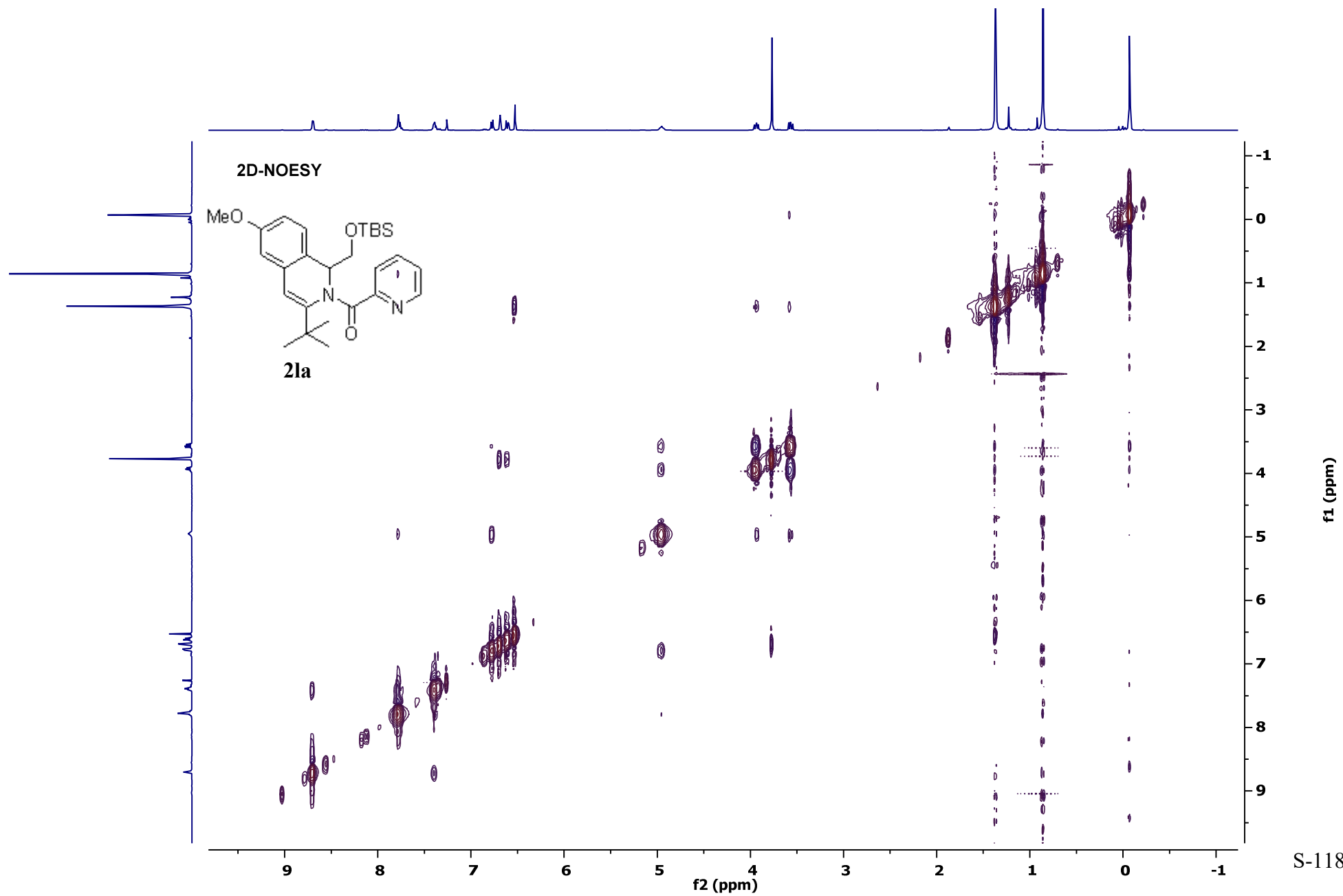




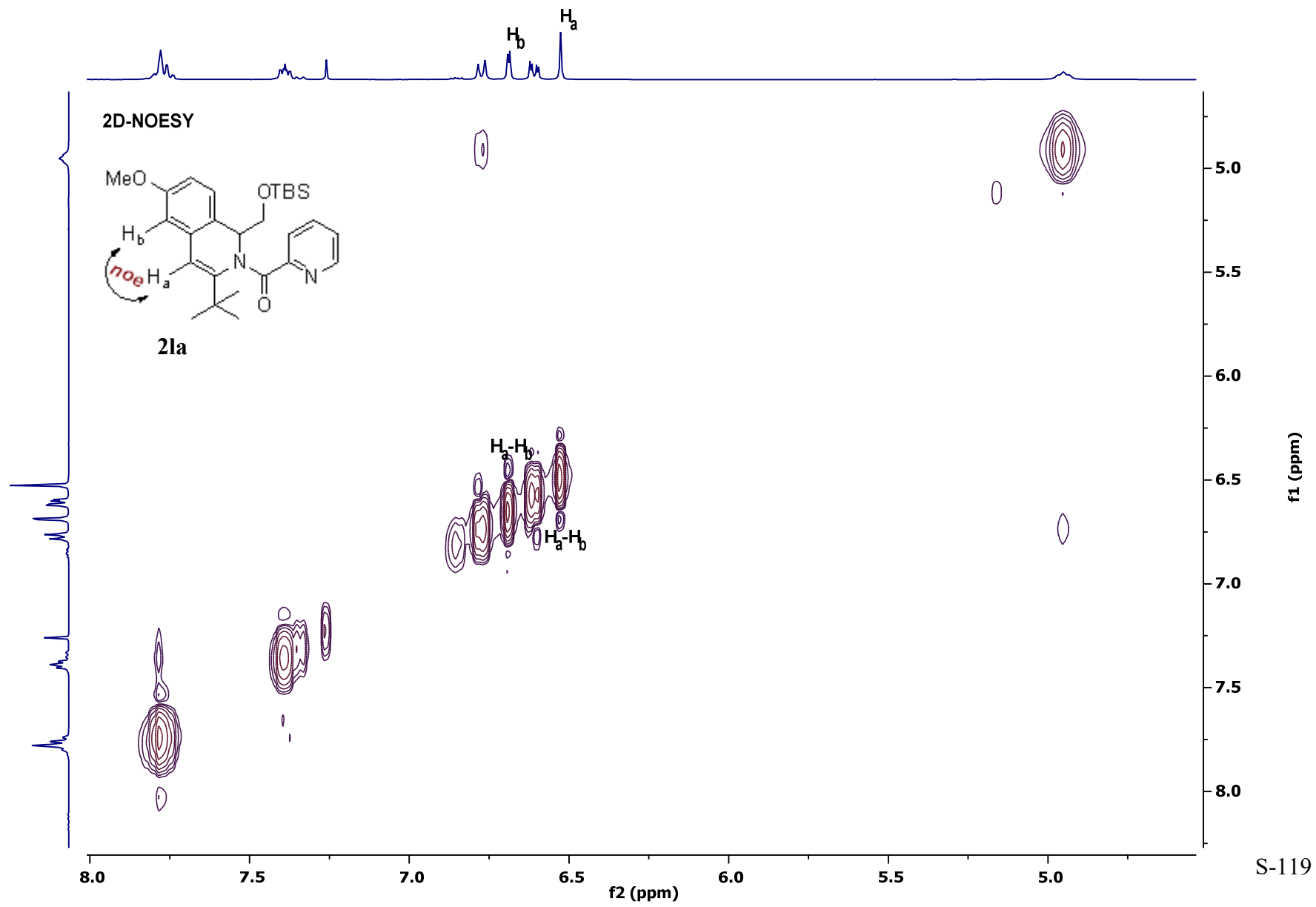
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



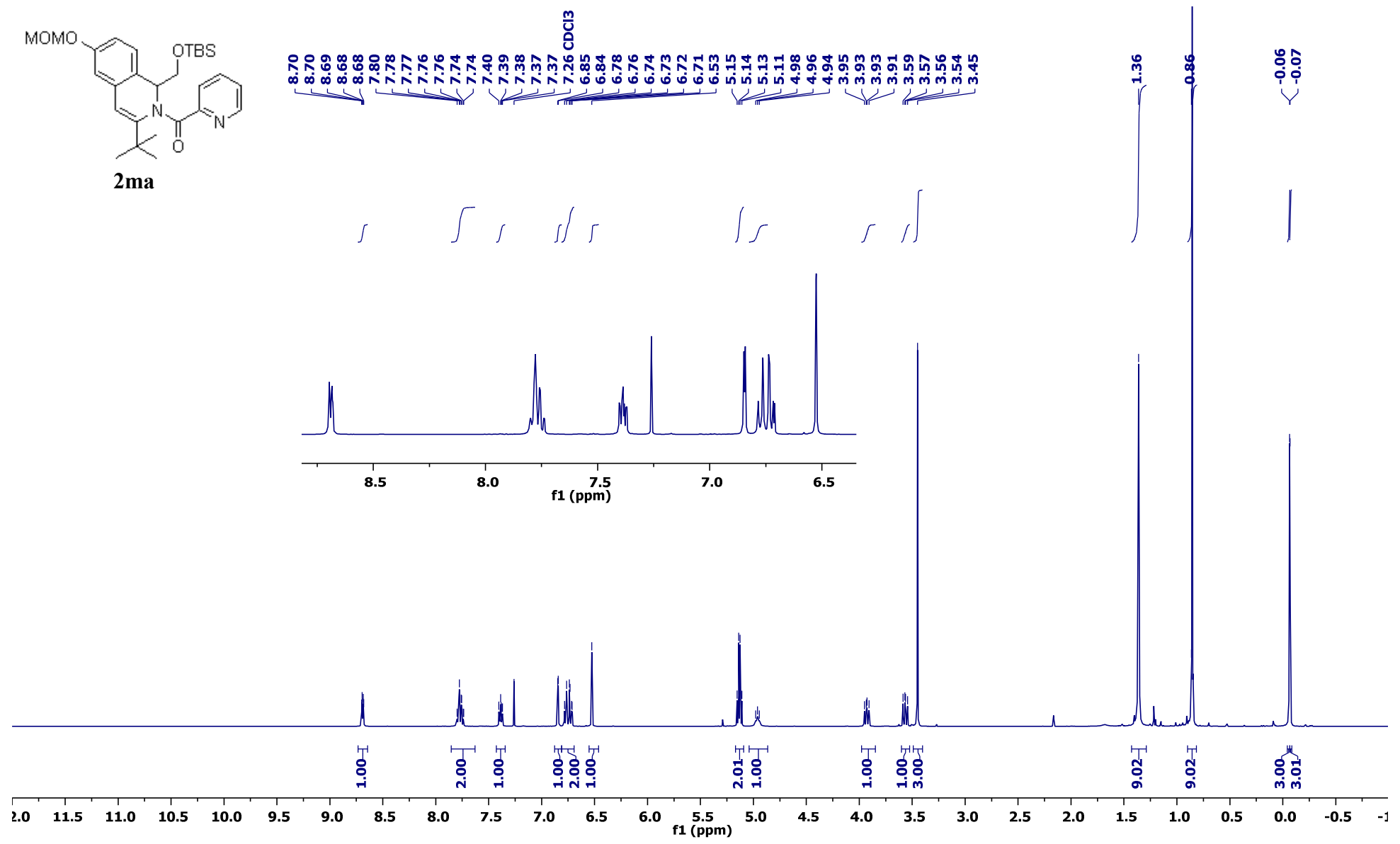
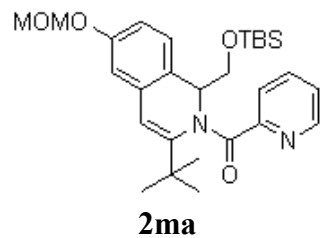
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



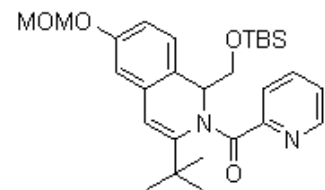
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



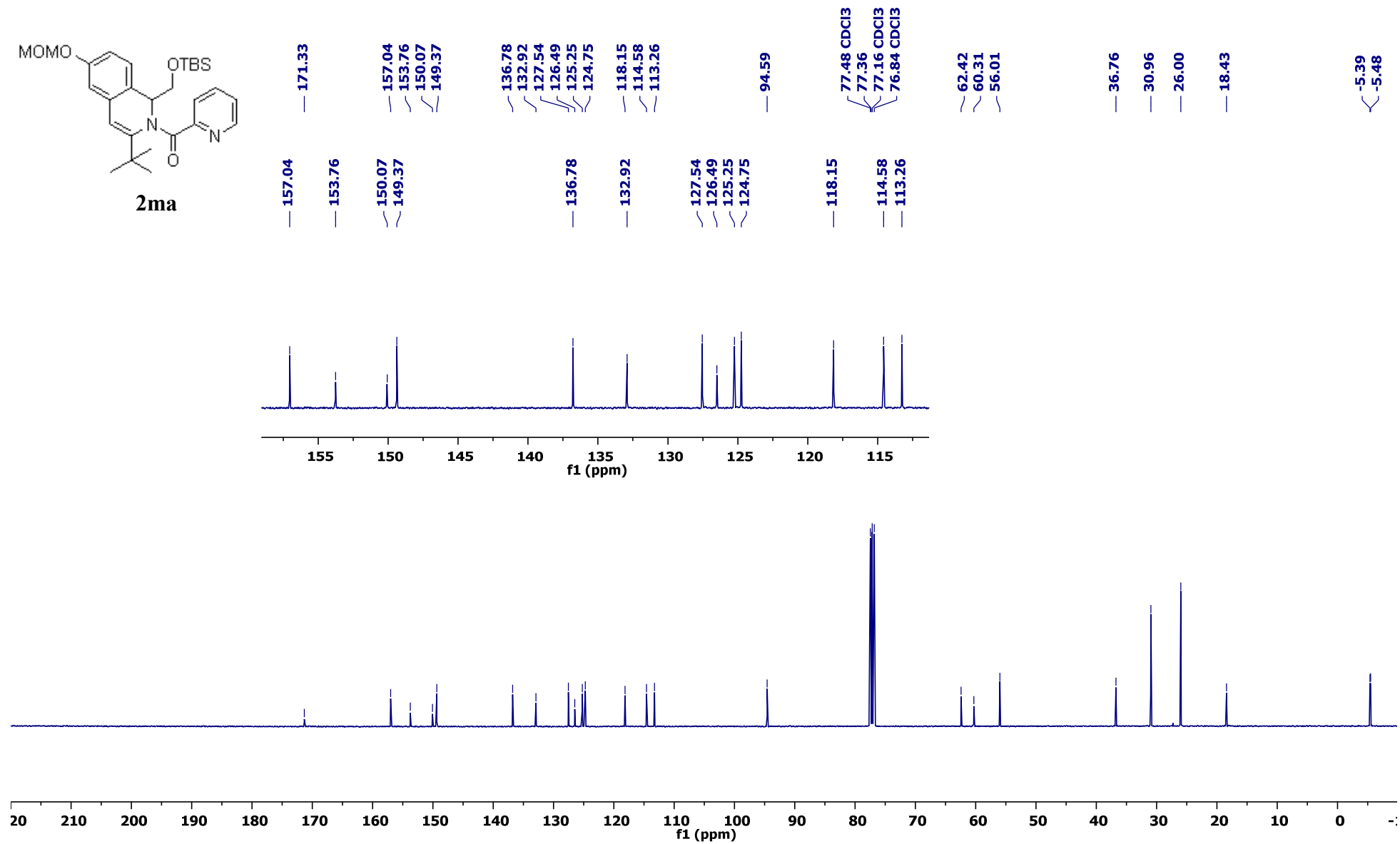
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



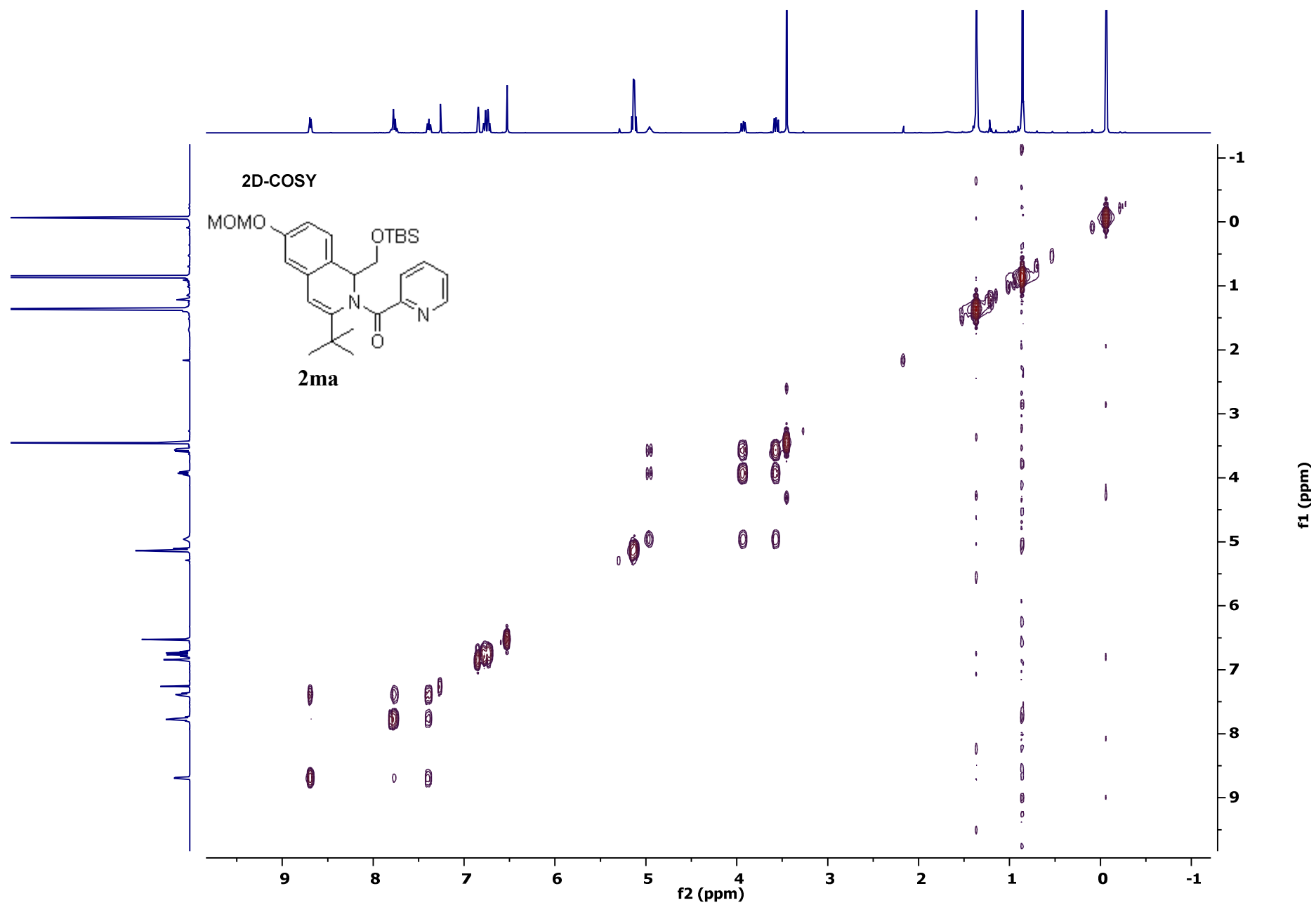
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



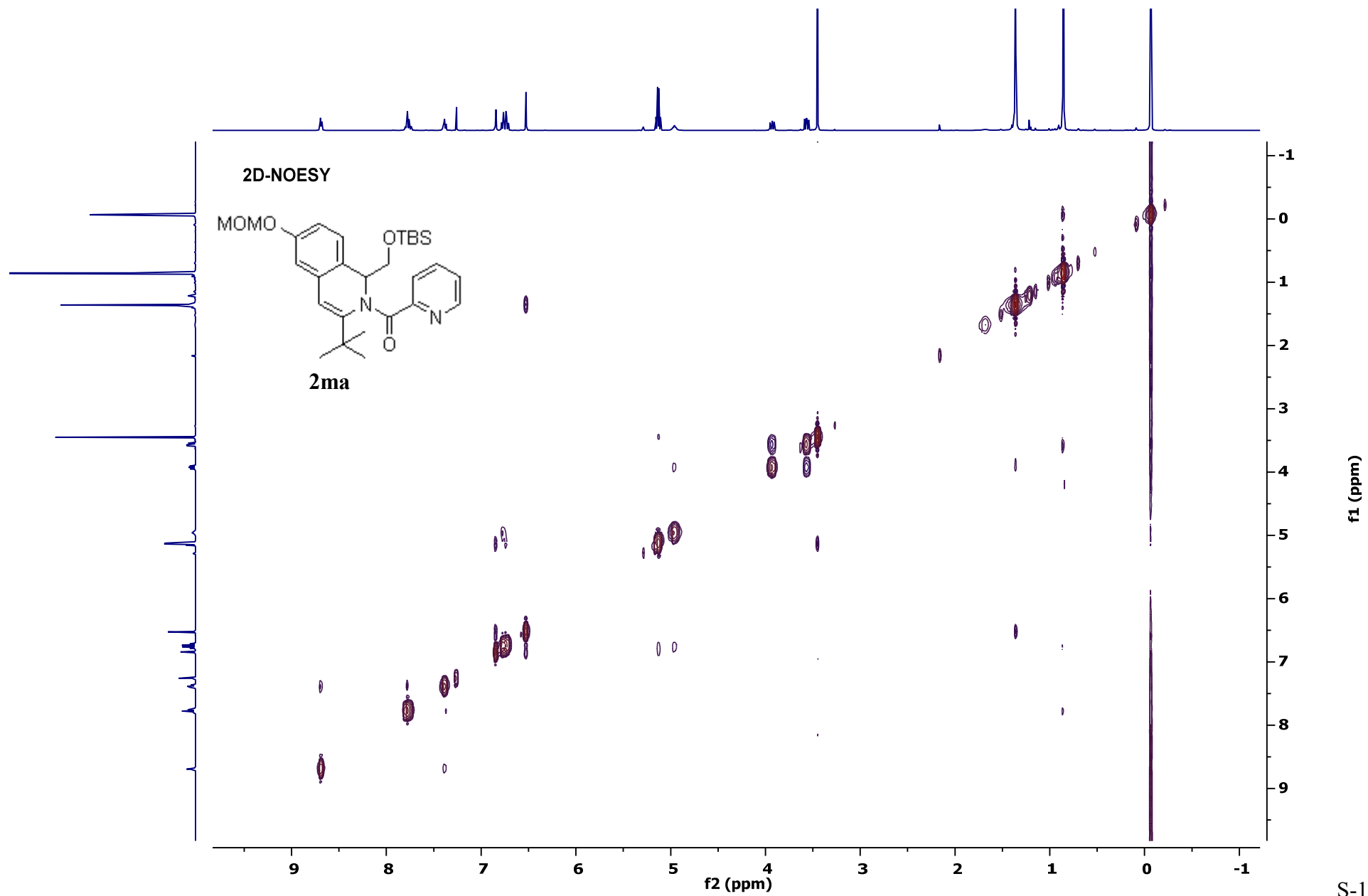
**2ma**



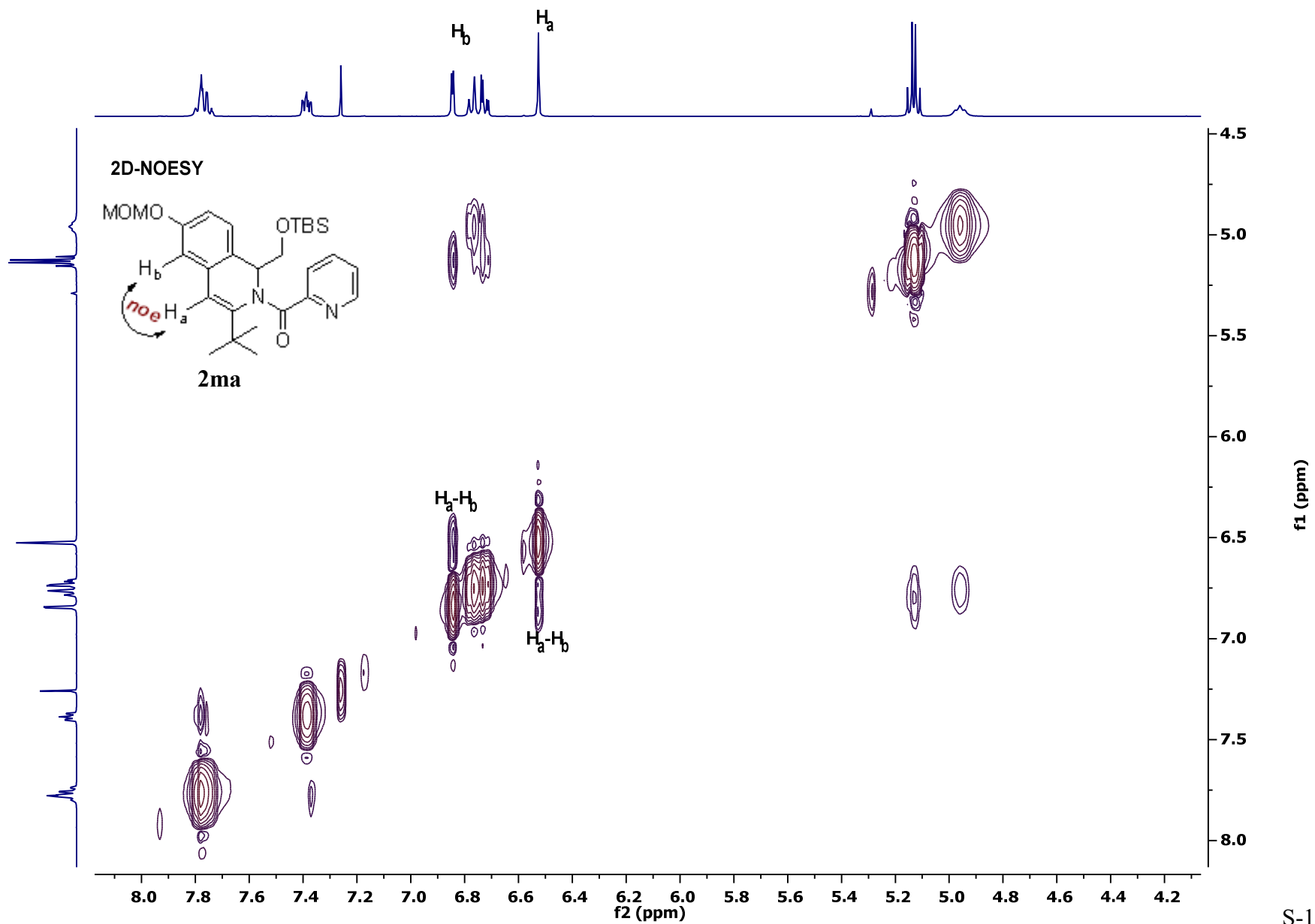
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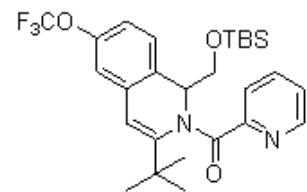
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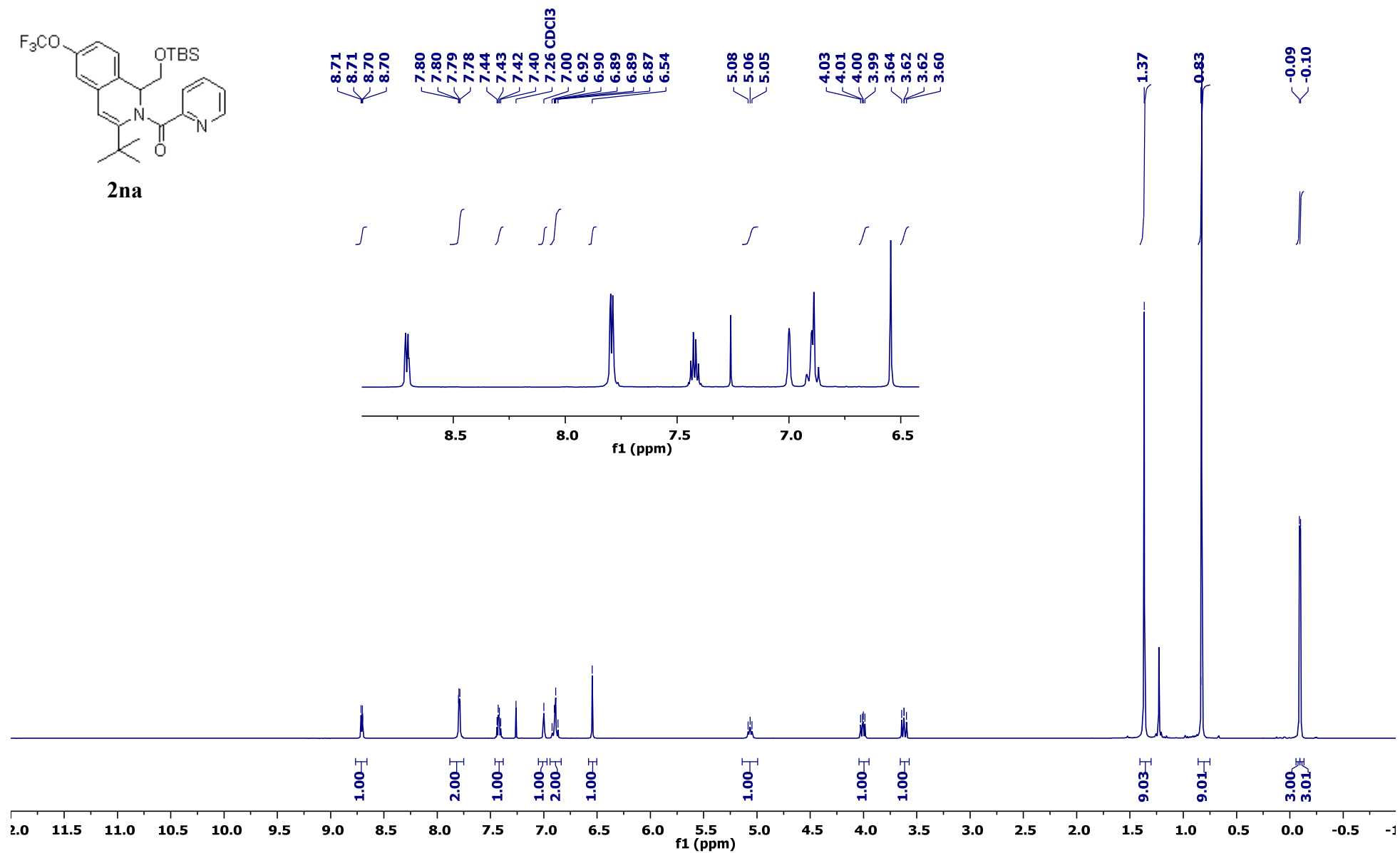
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



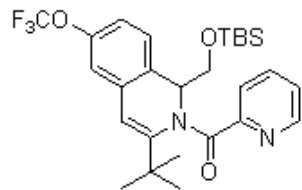
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



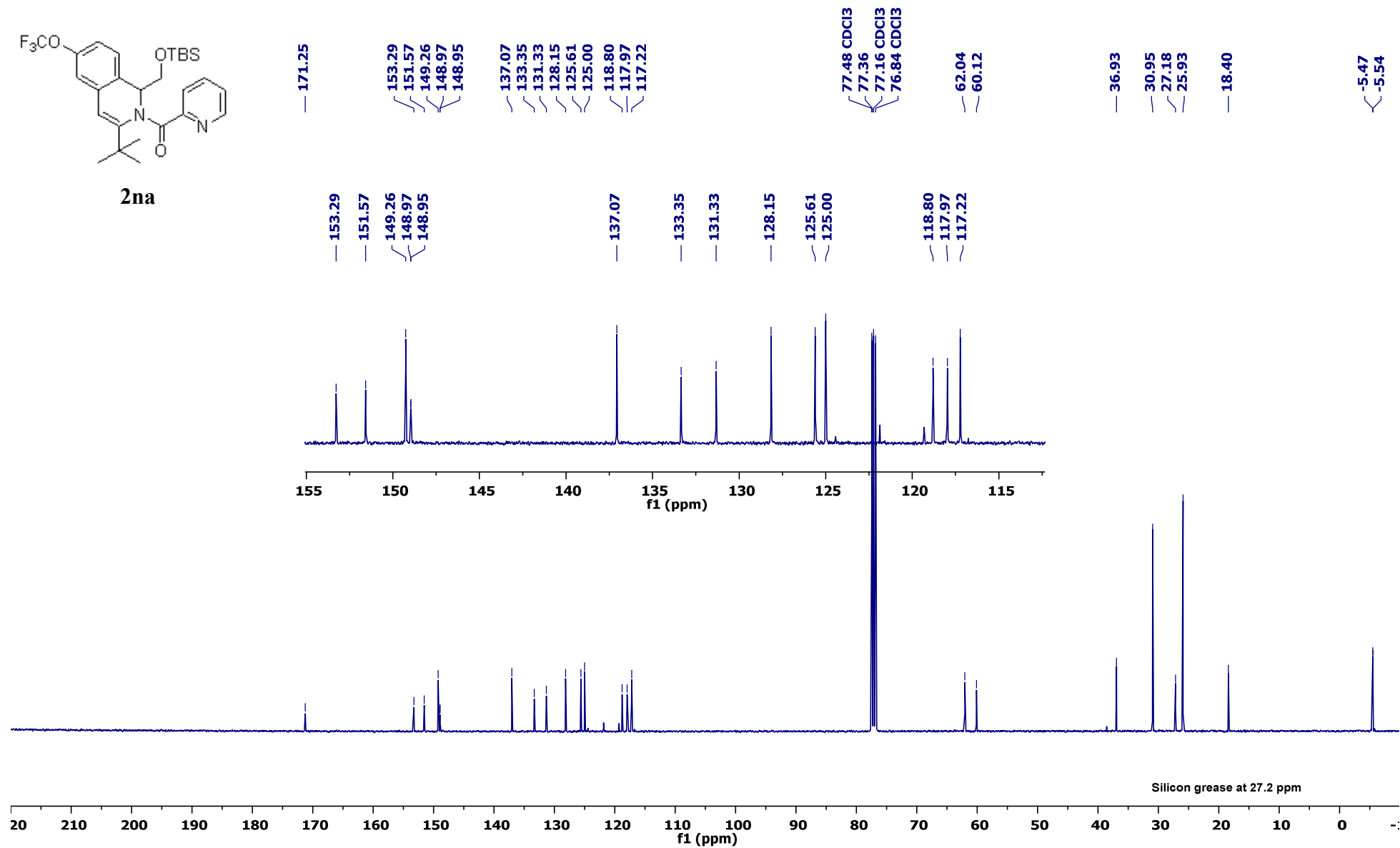
**2na**



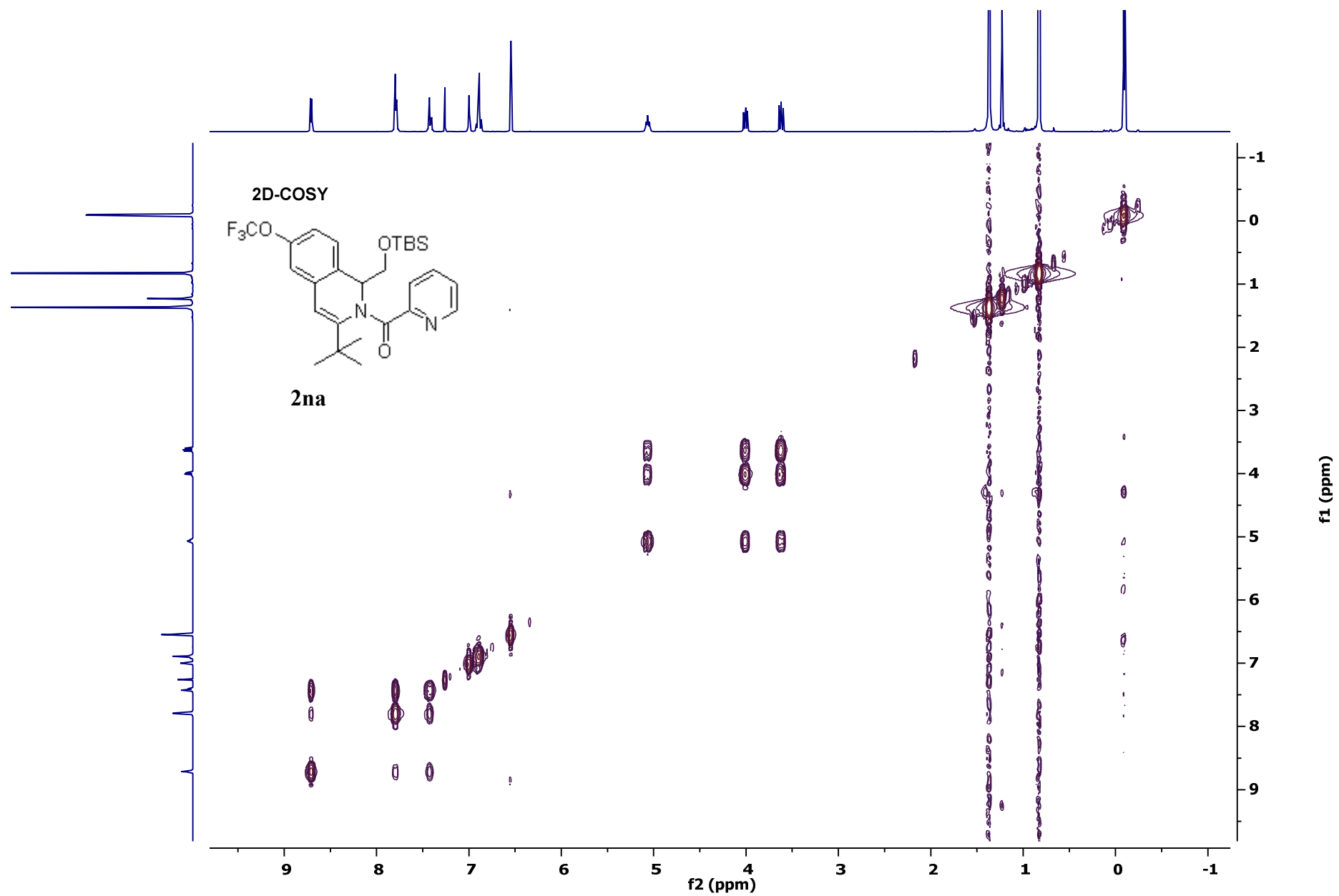
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



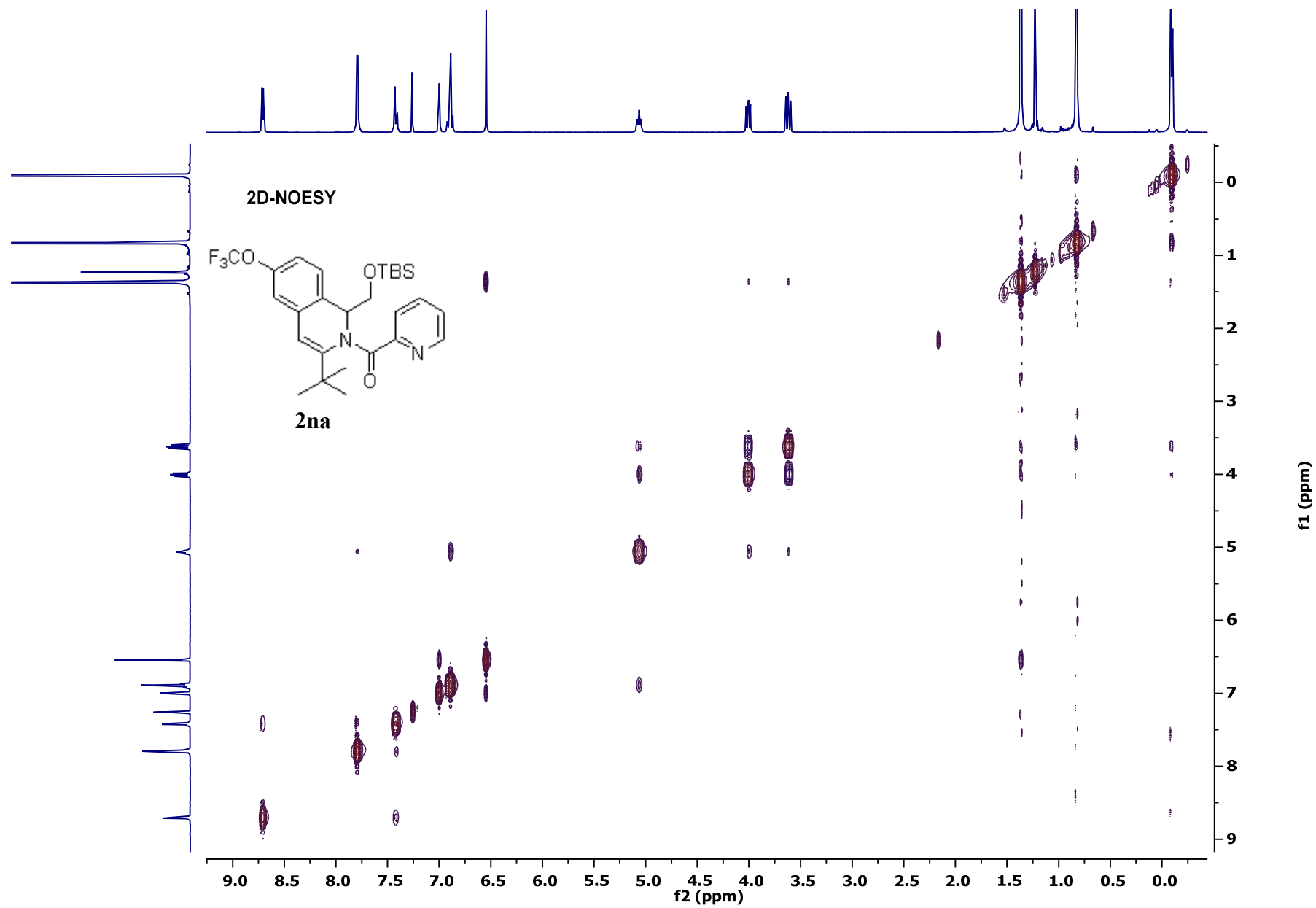
**2na**



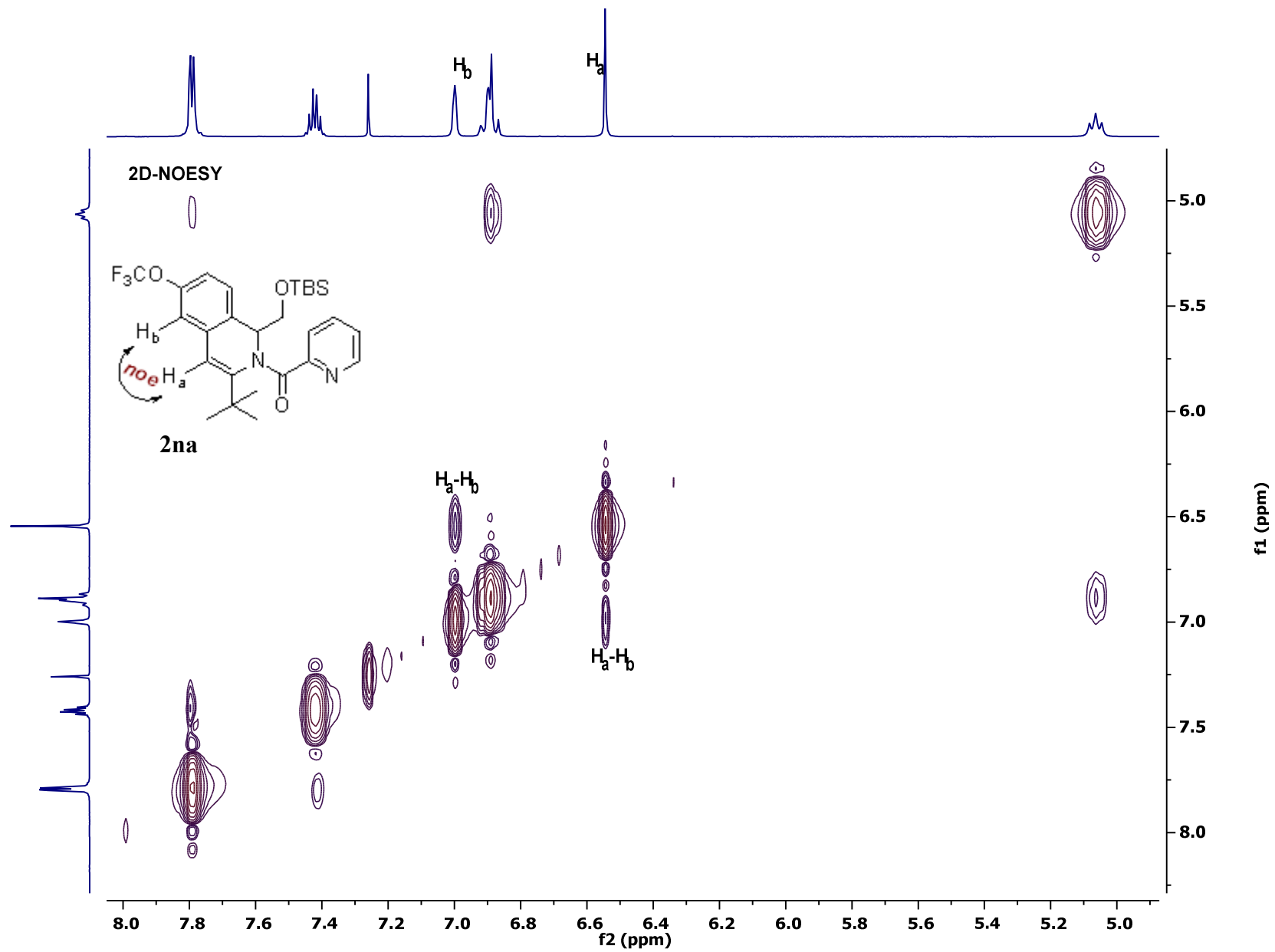
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



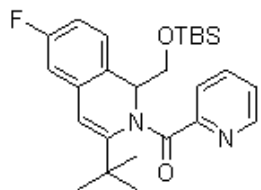
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



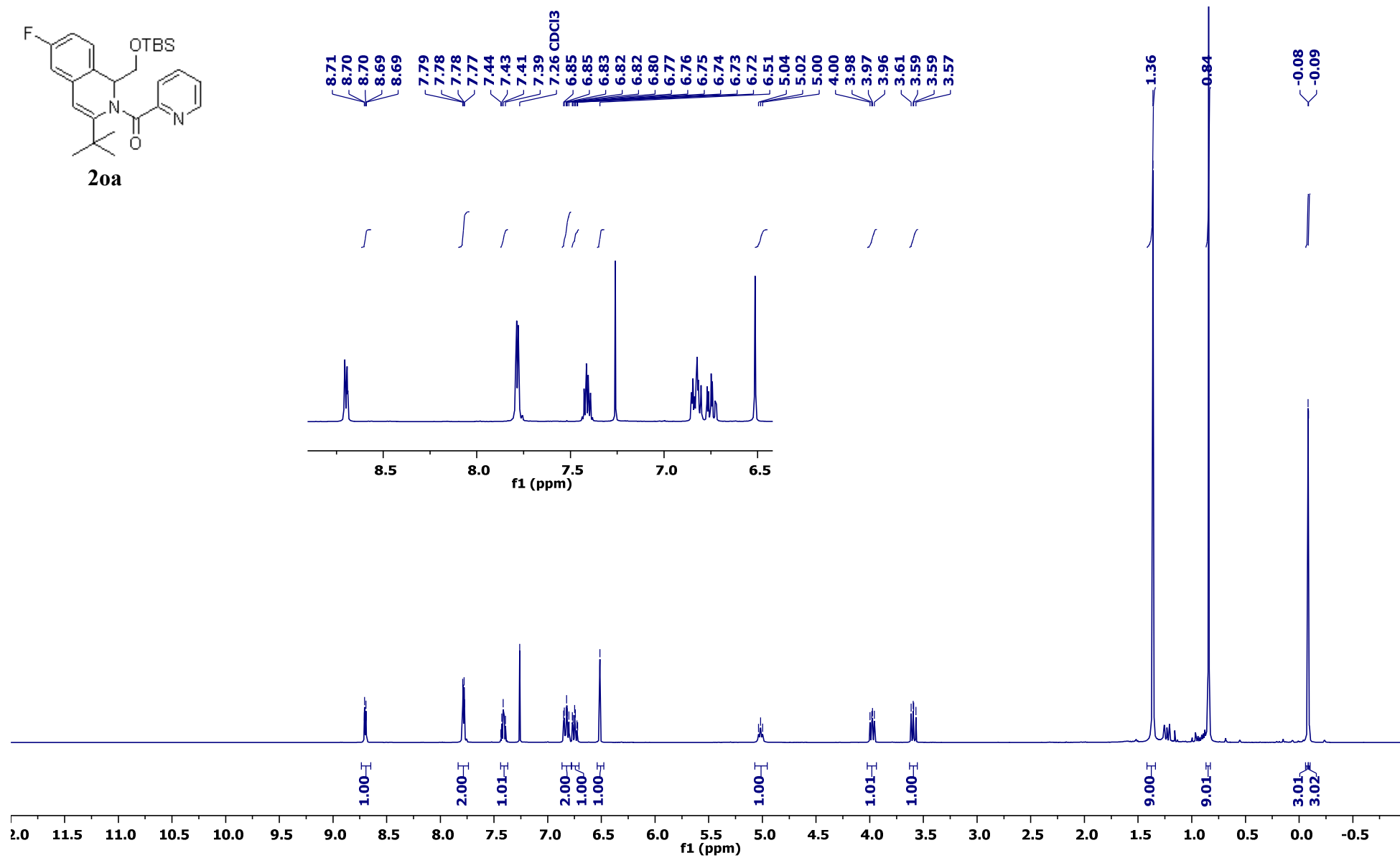
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

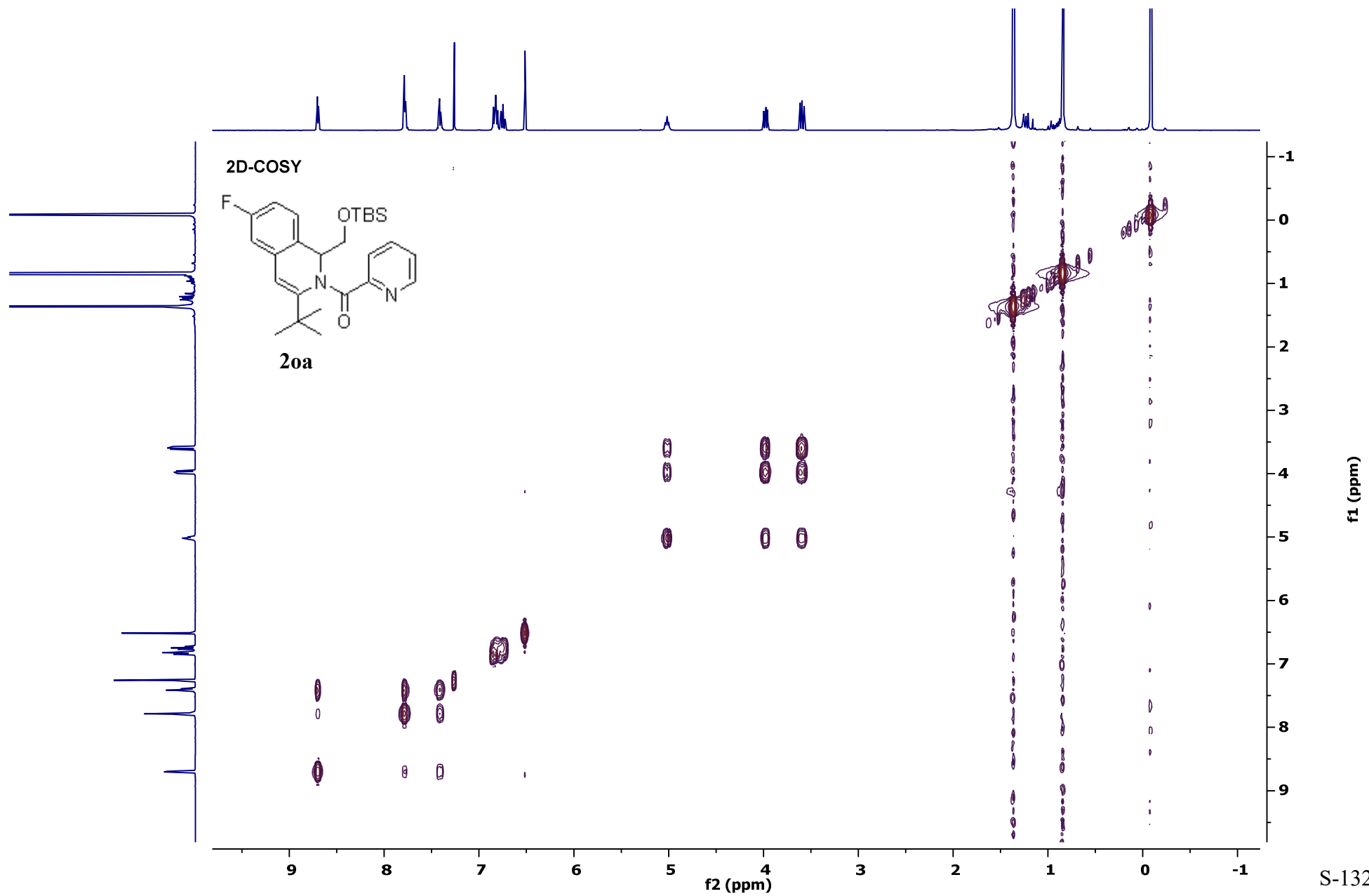


**2oa**

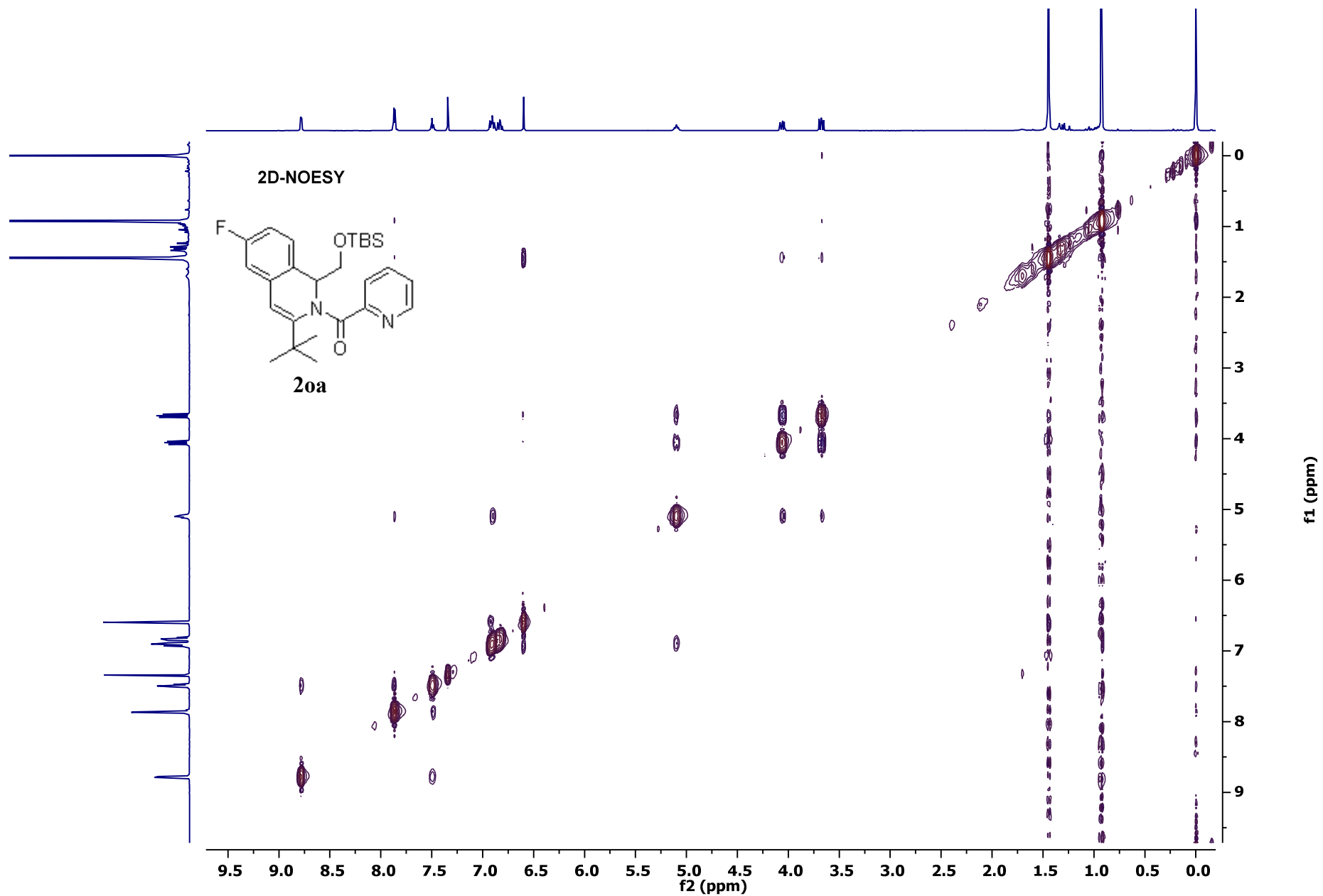




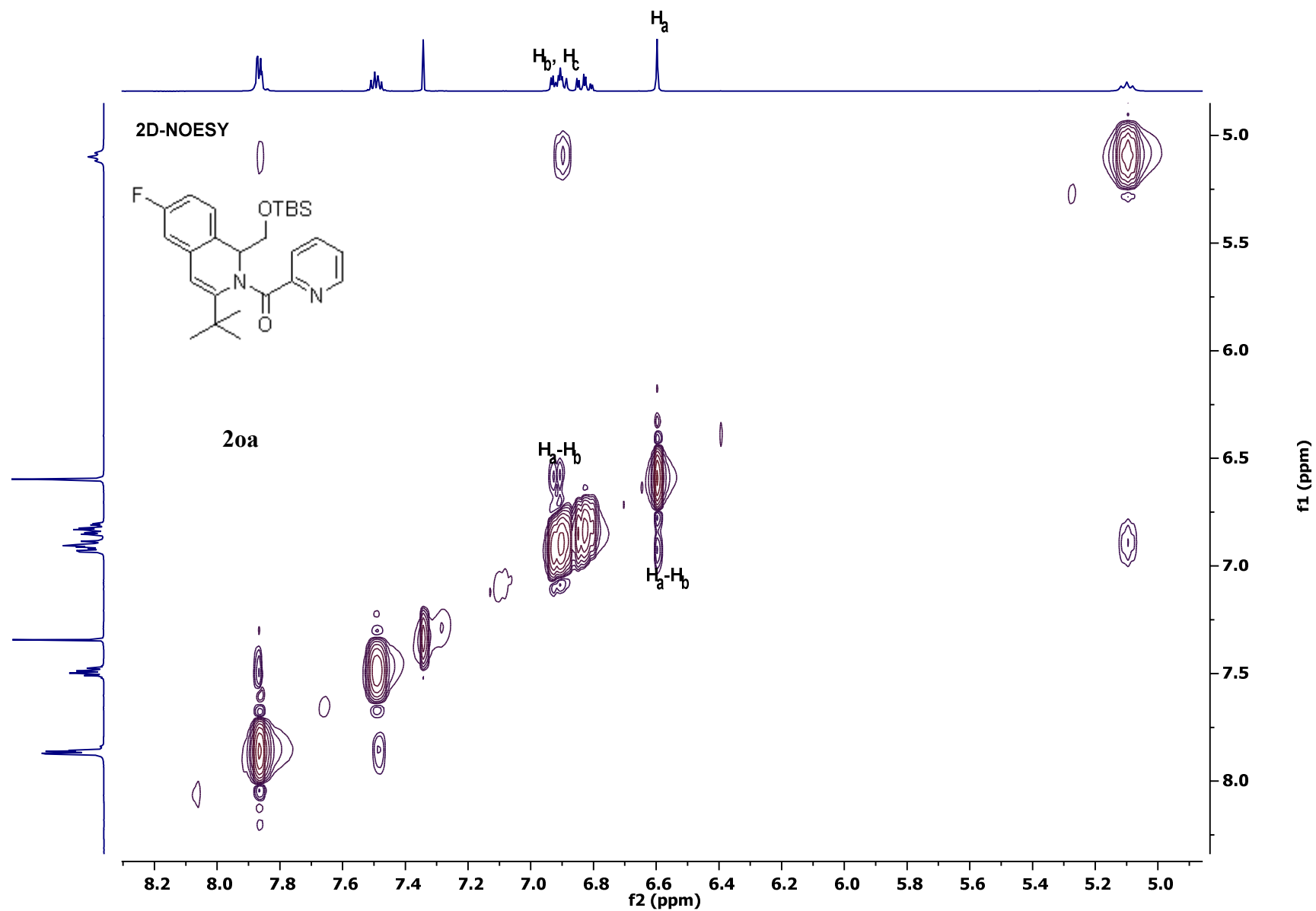
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



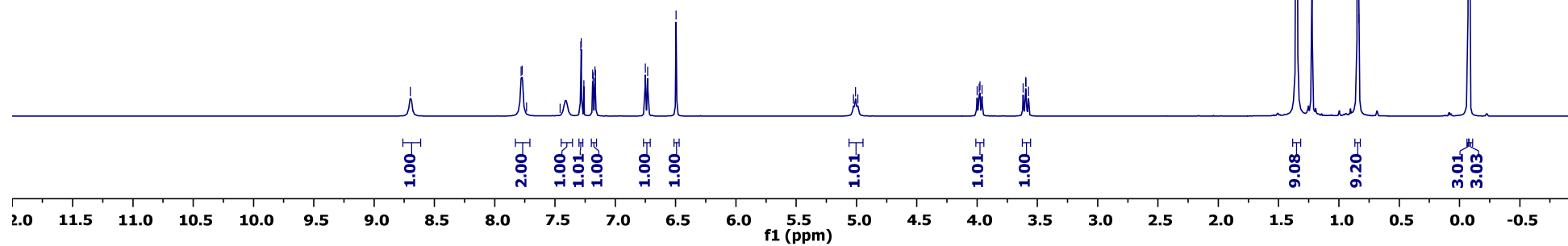
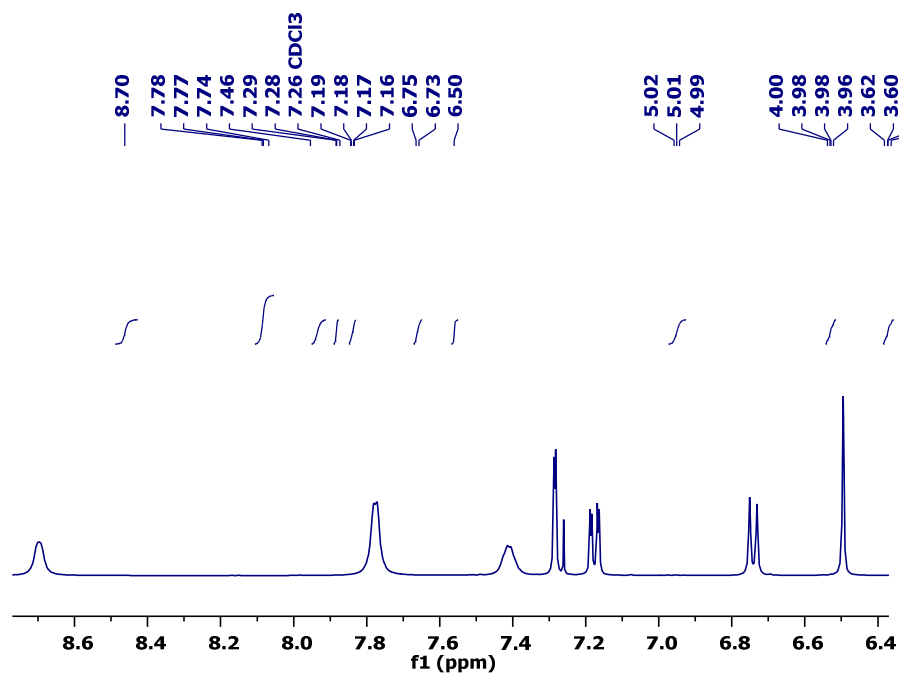
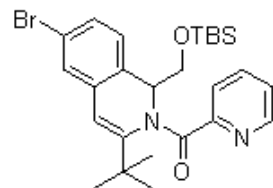
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



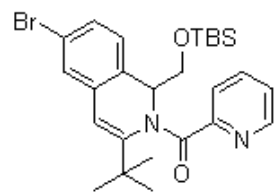
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



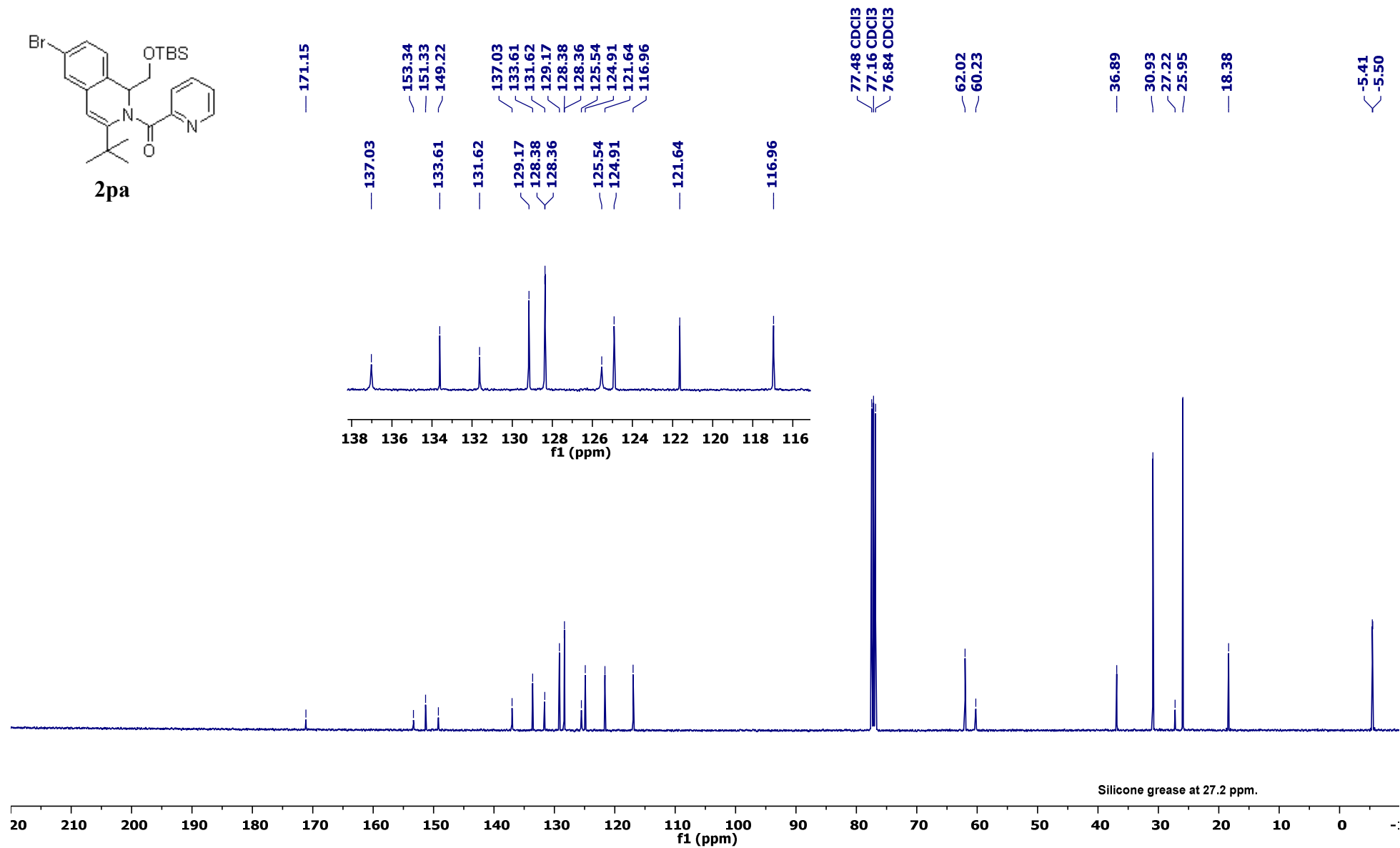
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

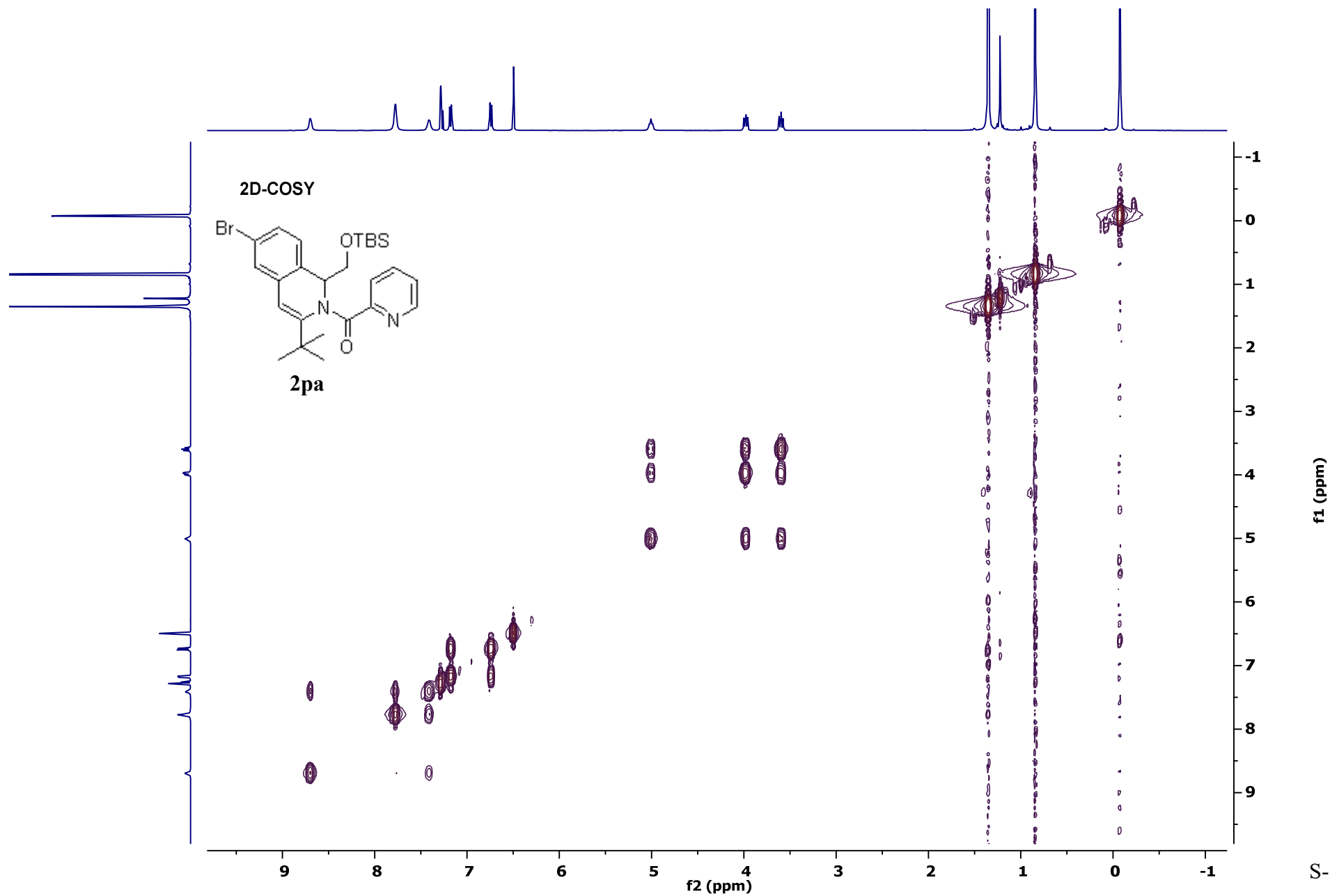


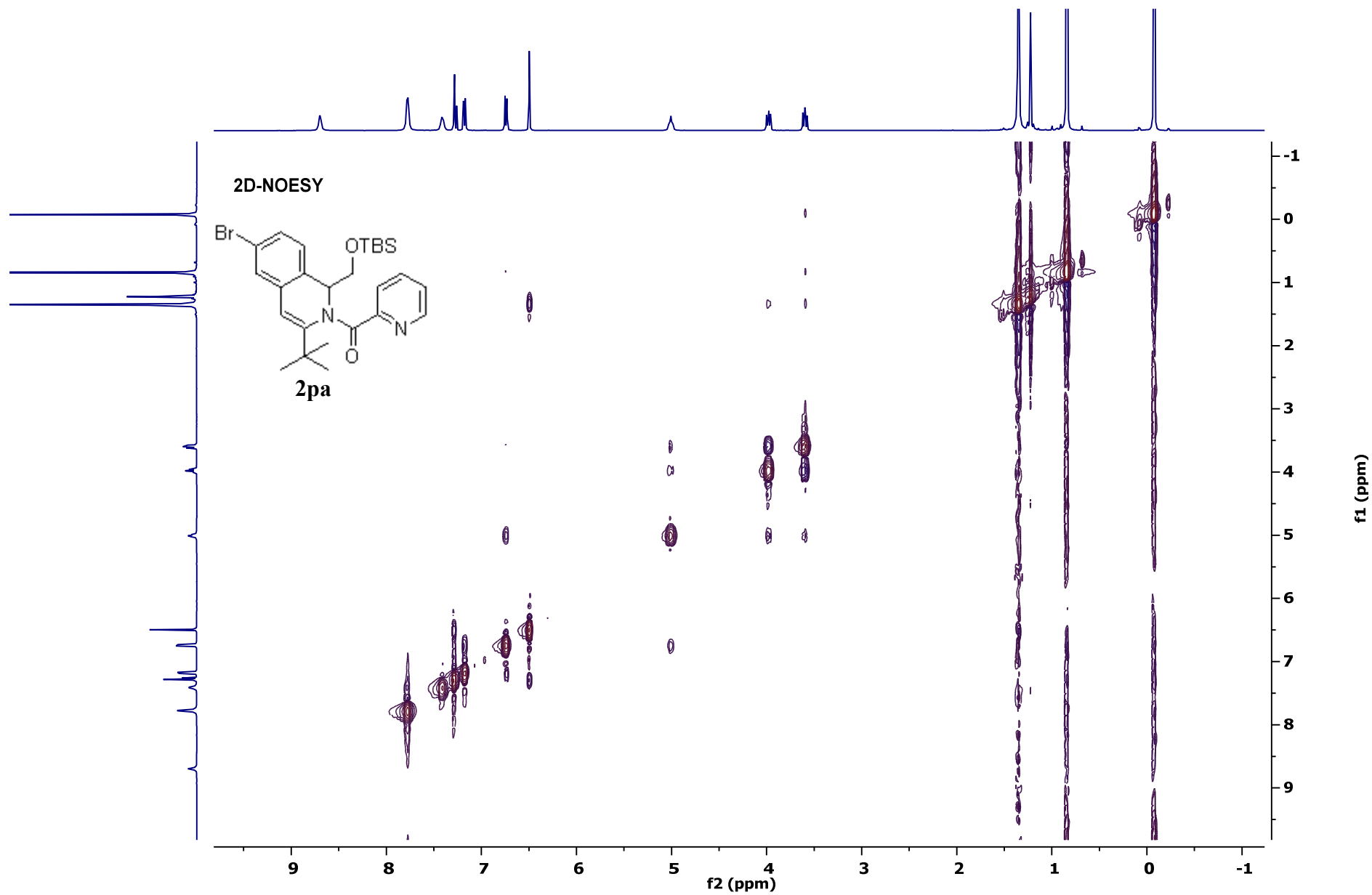
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



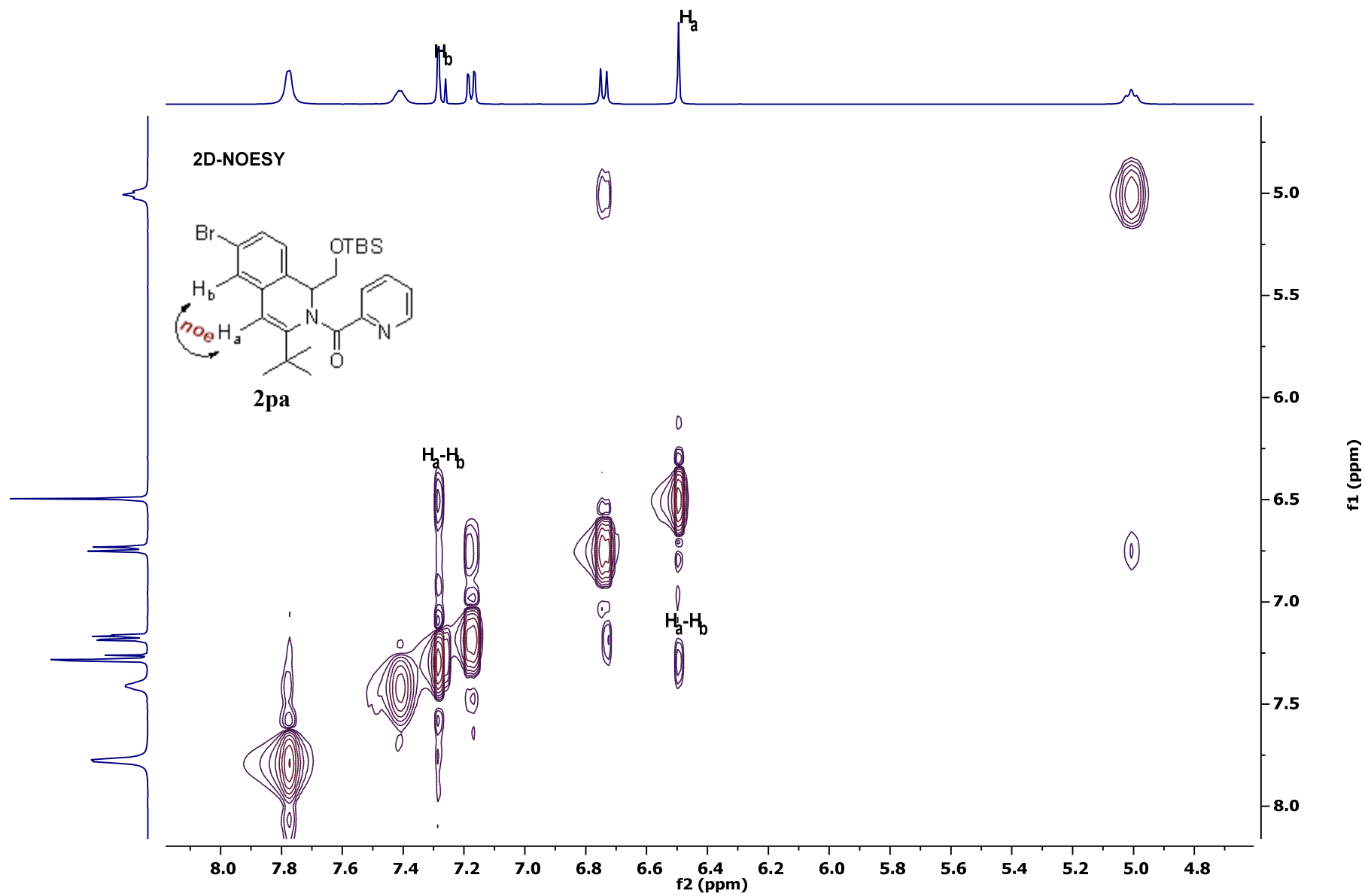
2pa



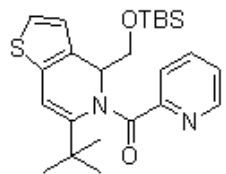




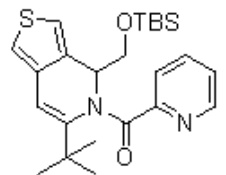
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



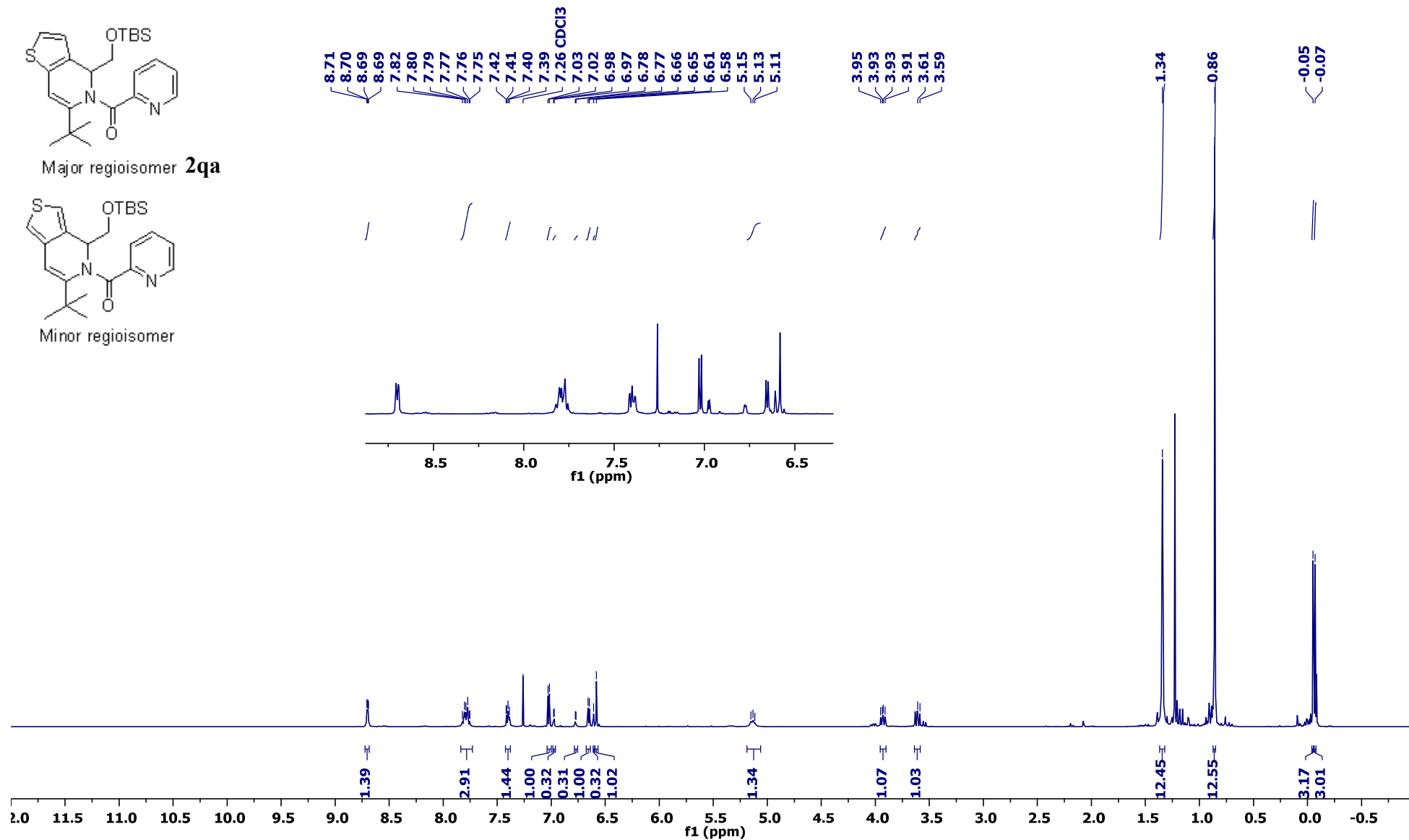
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



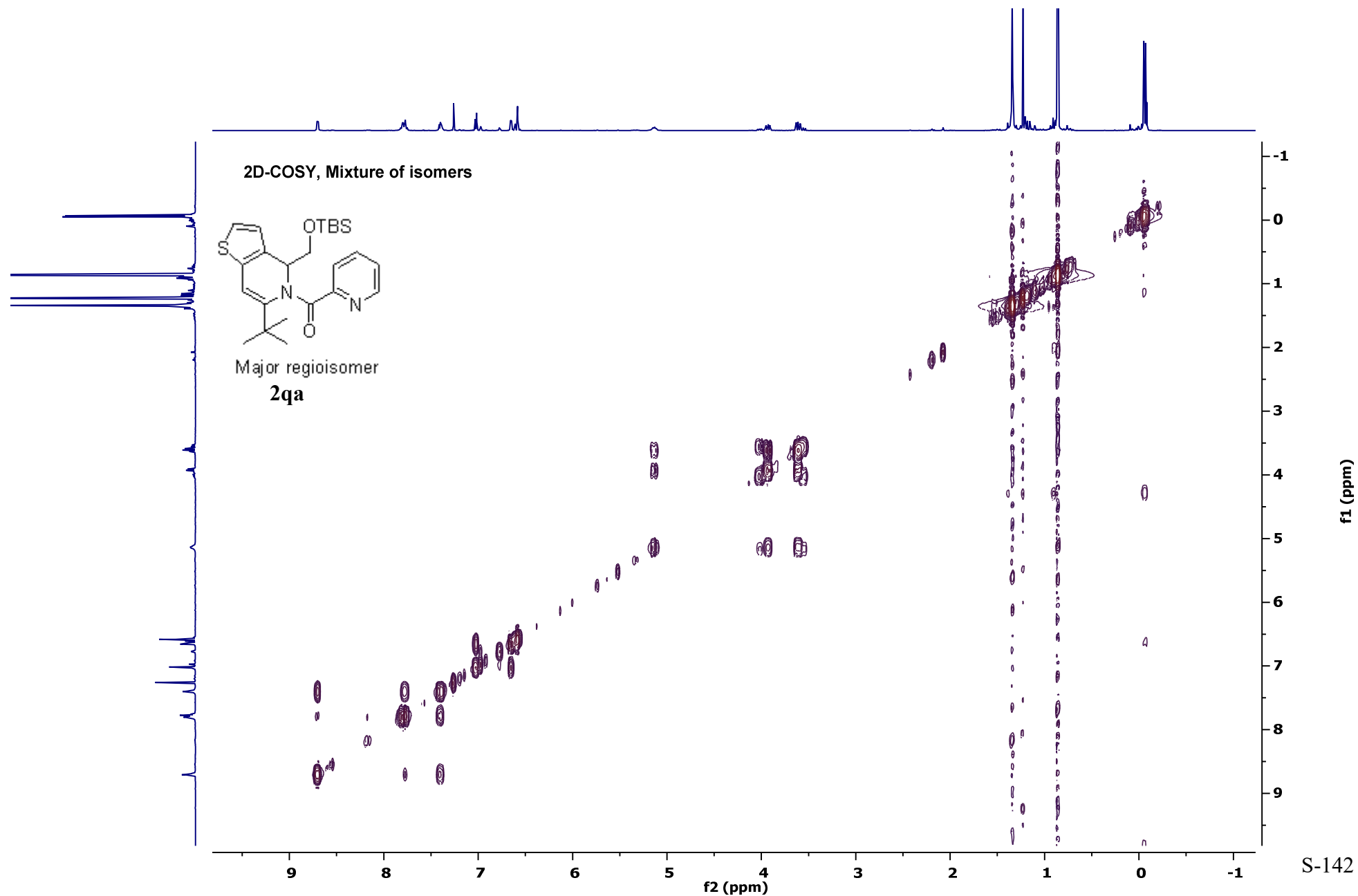
Major regioisomer **2qa**

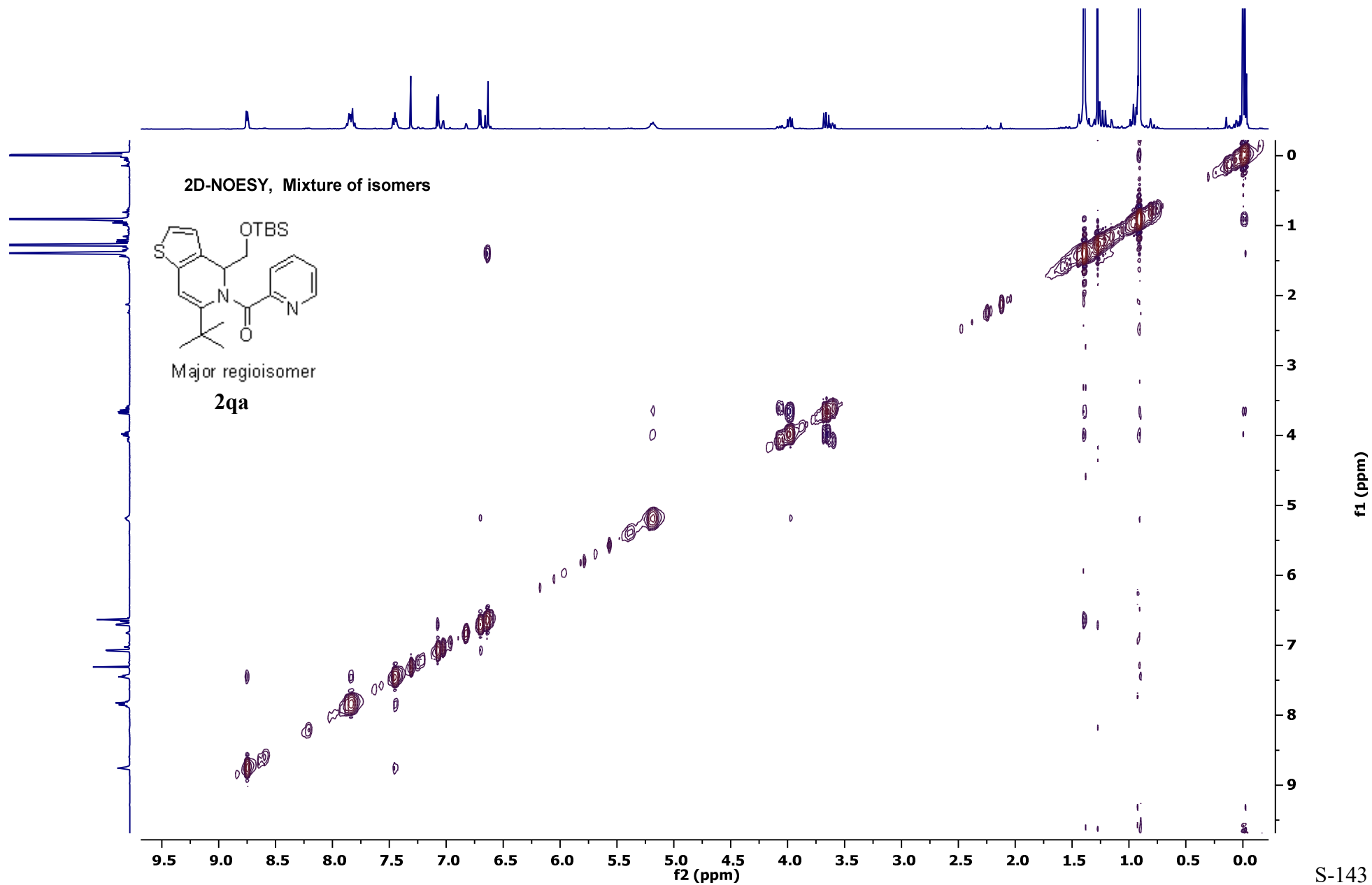


Minor regioisomer

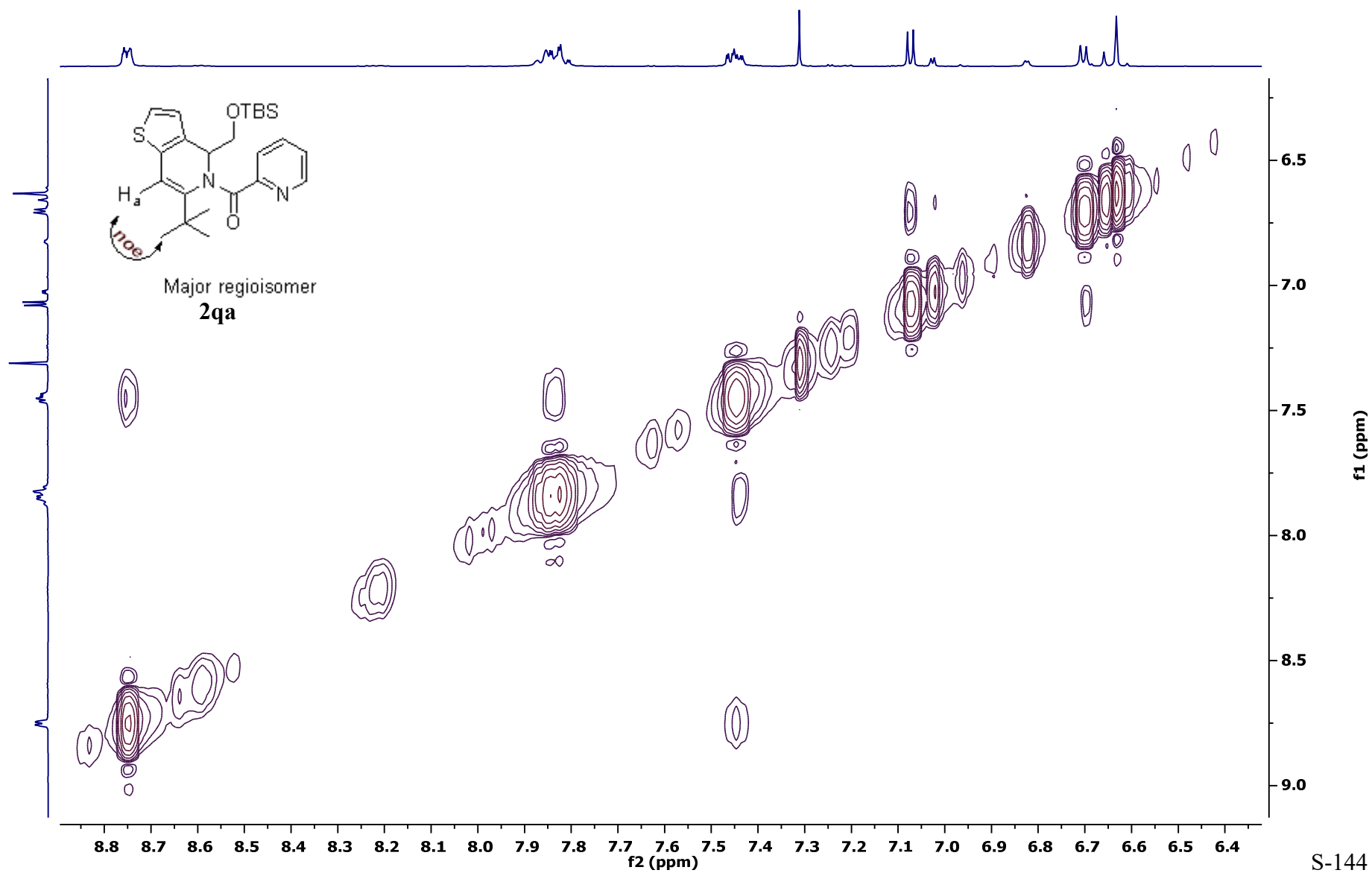






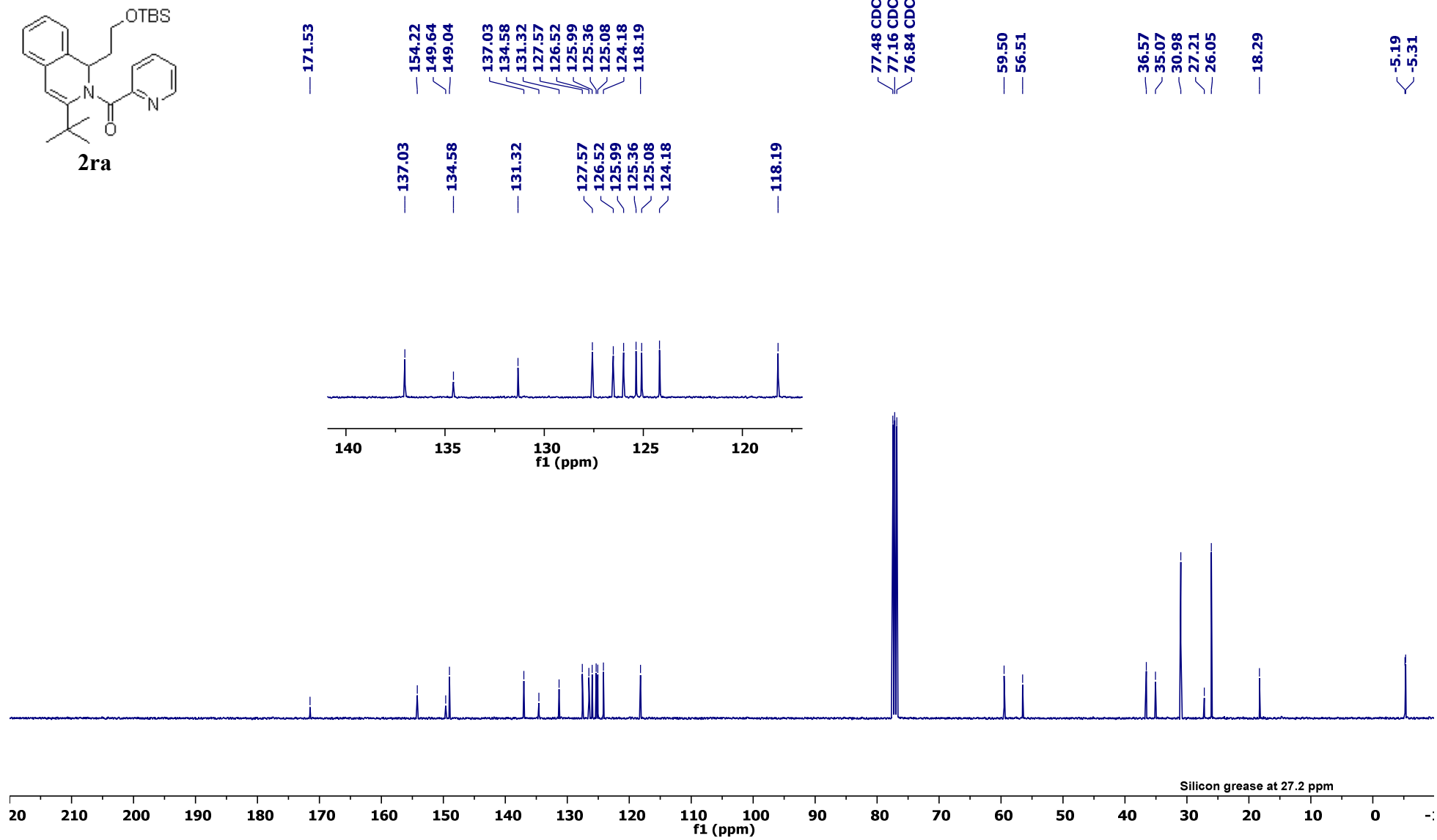


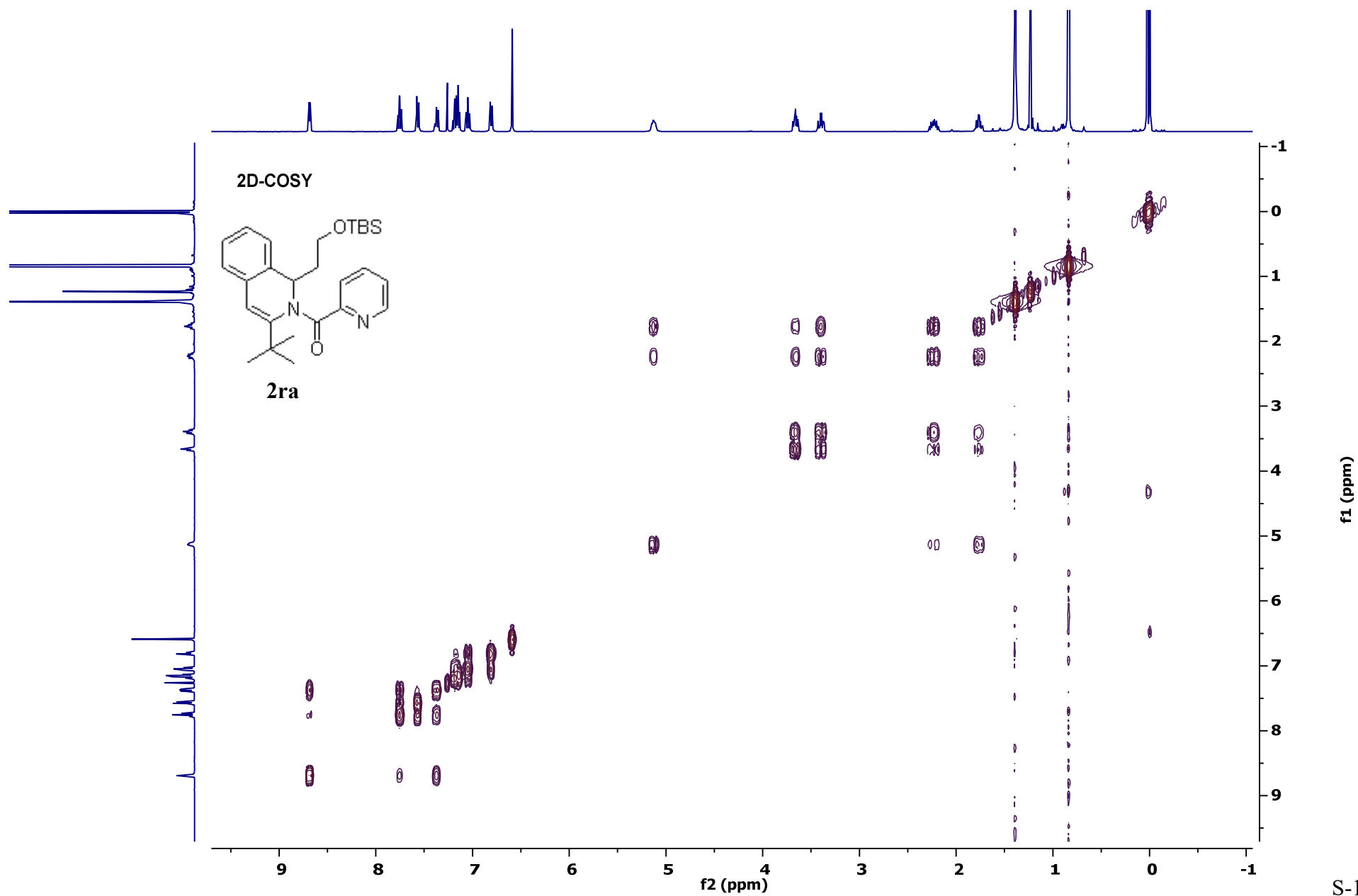
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



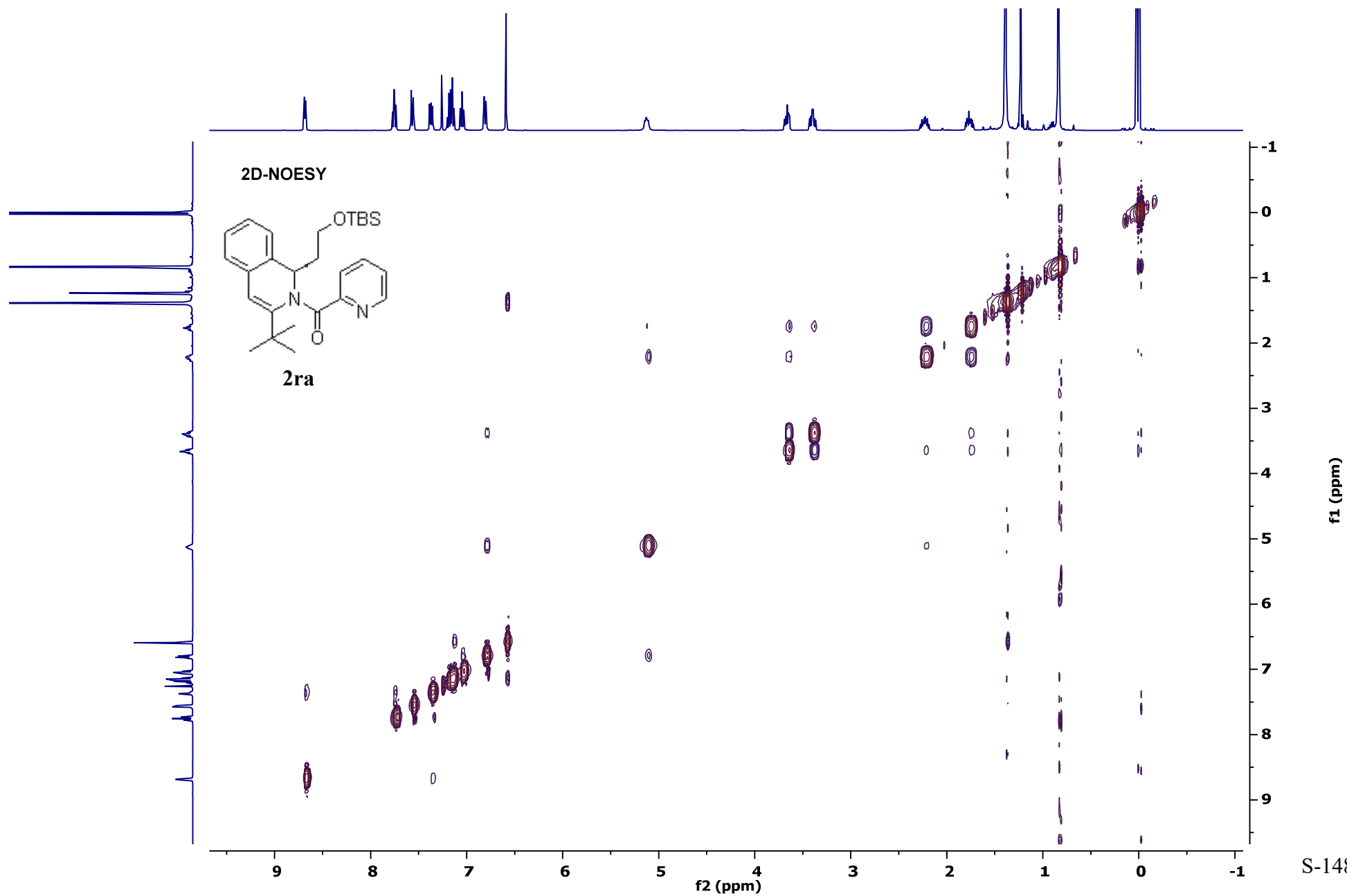


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

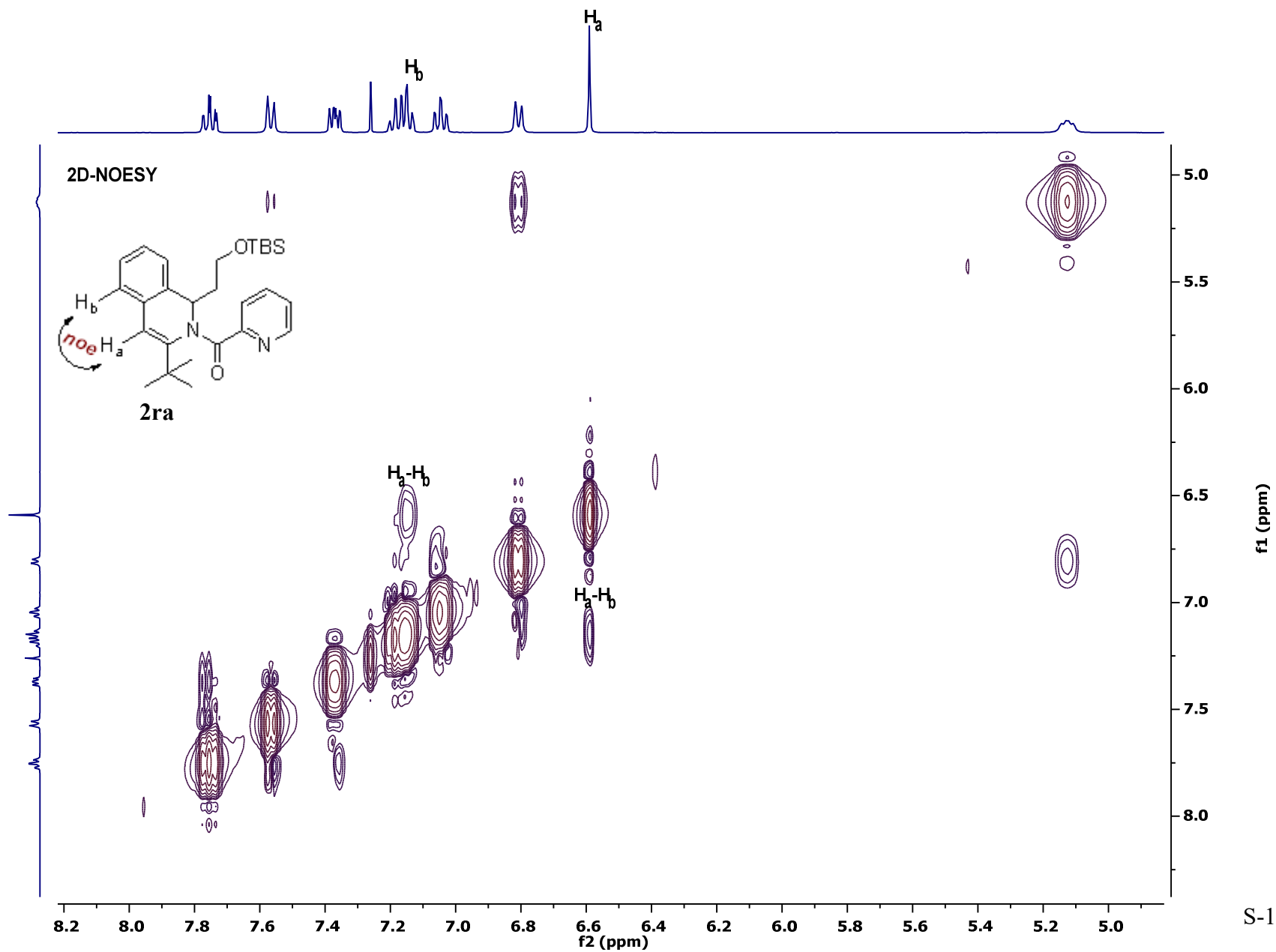




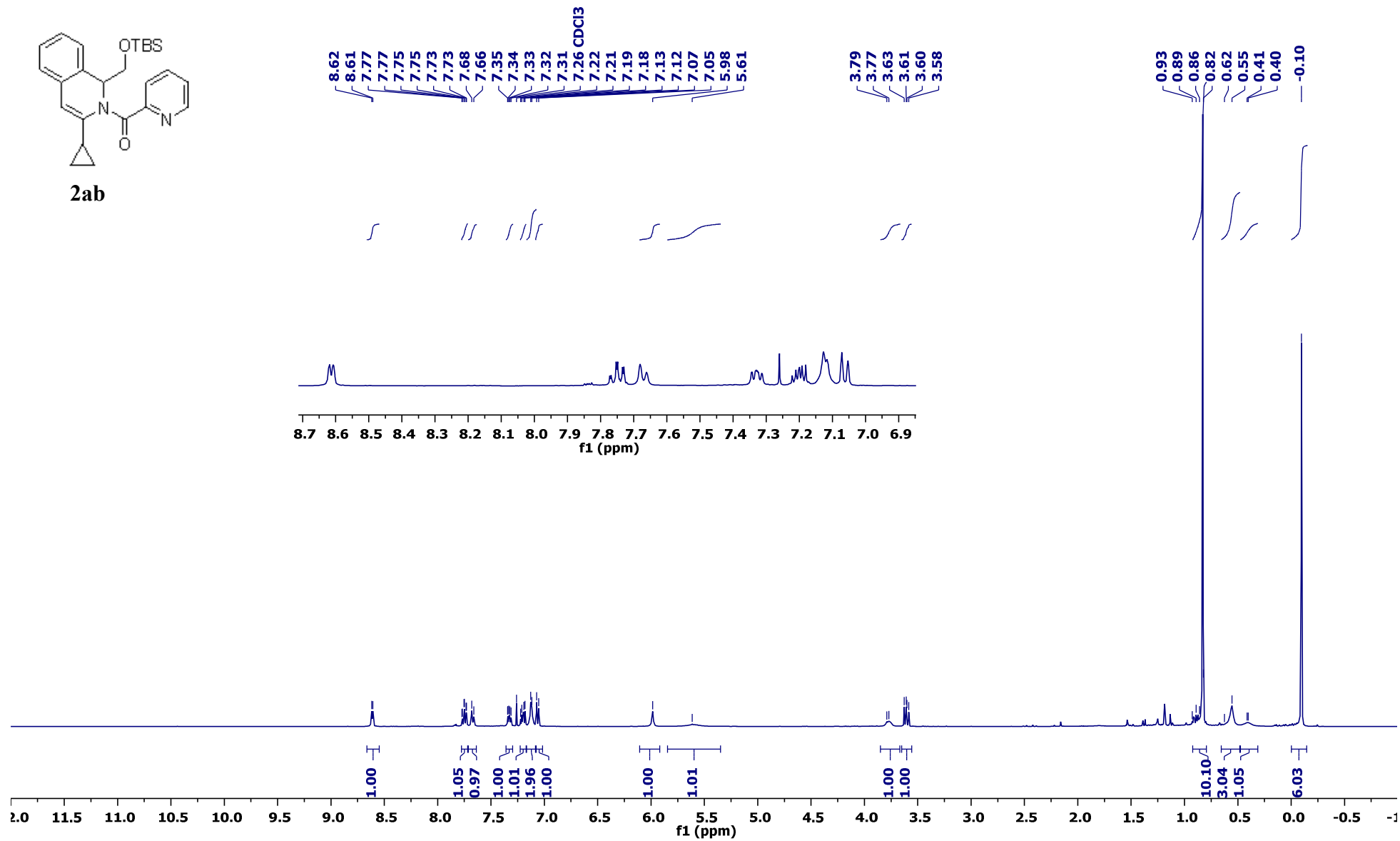
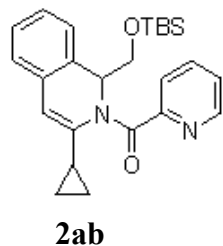
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



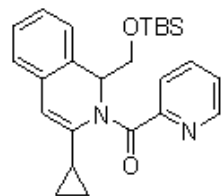
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



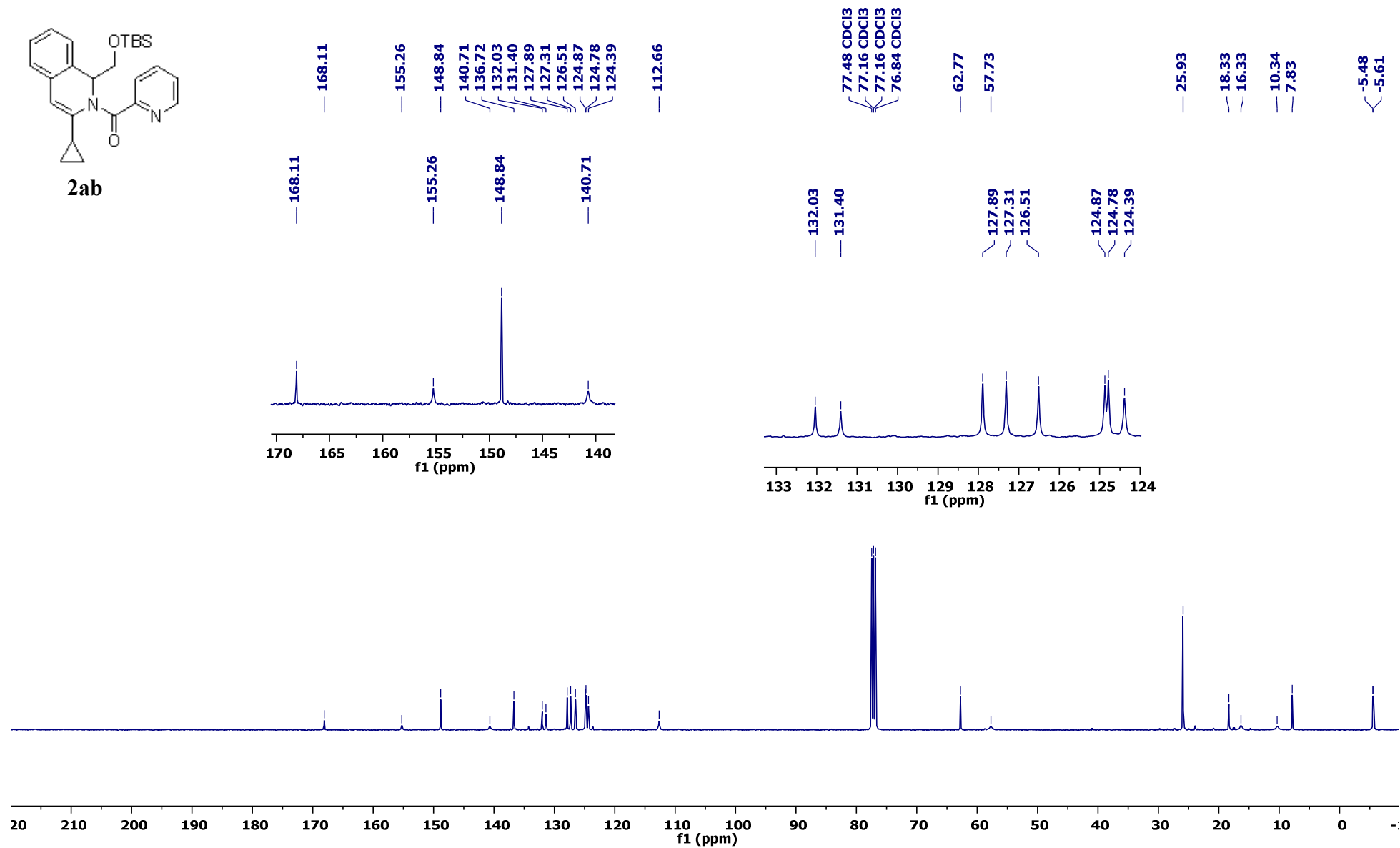
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



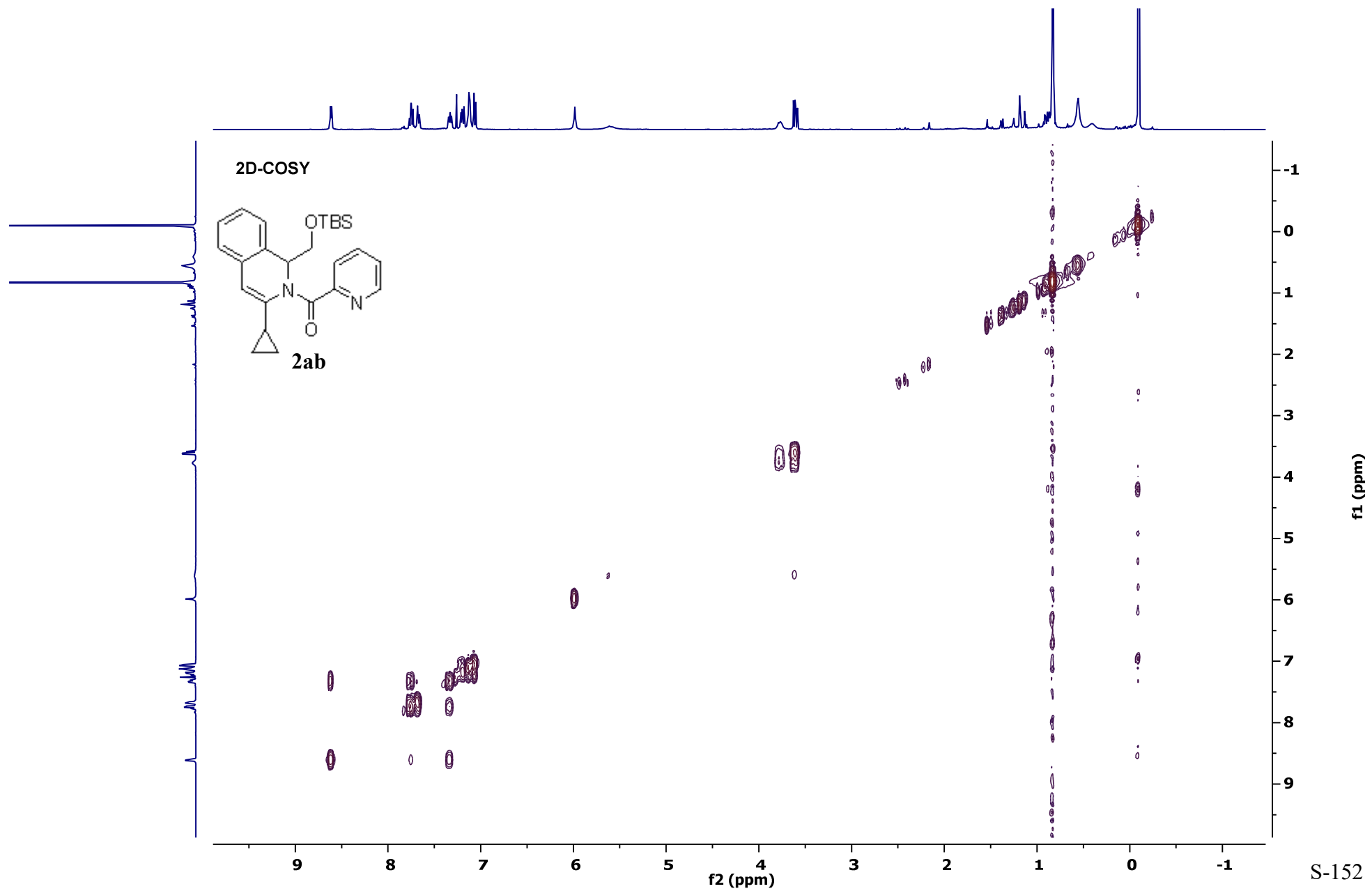
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



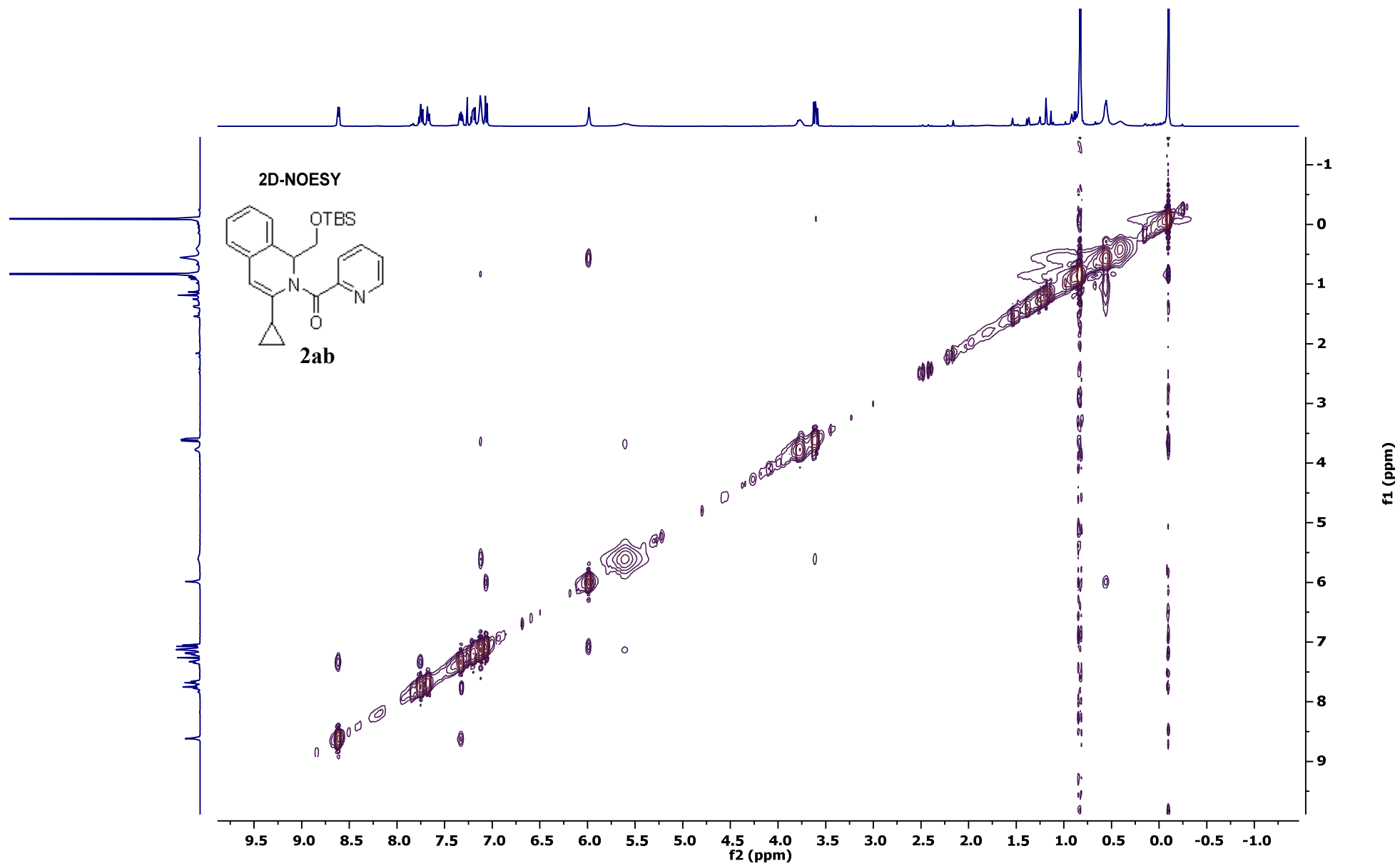
**2ab**



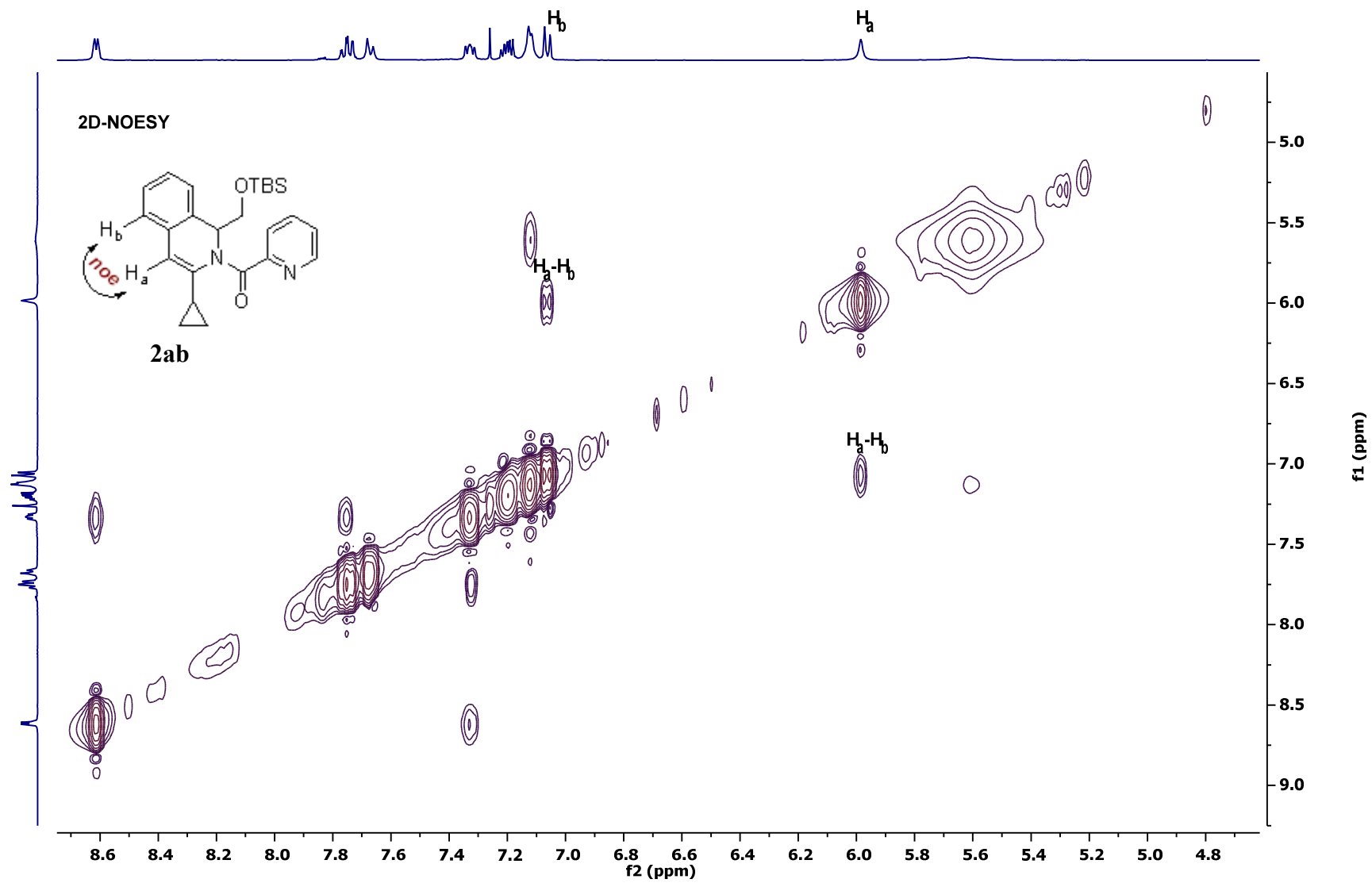
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



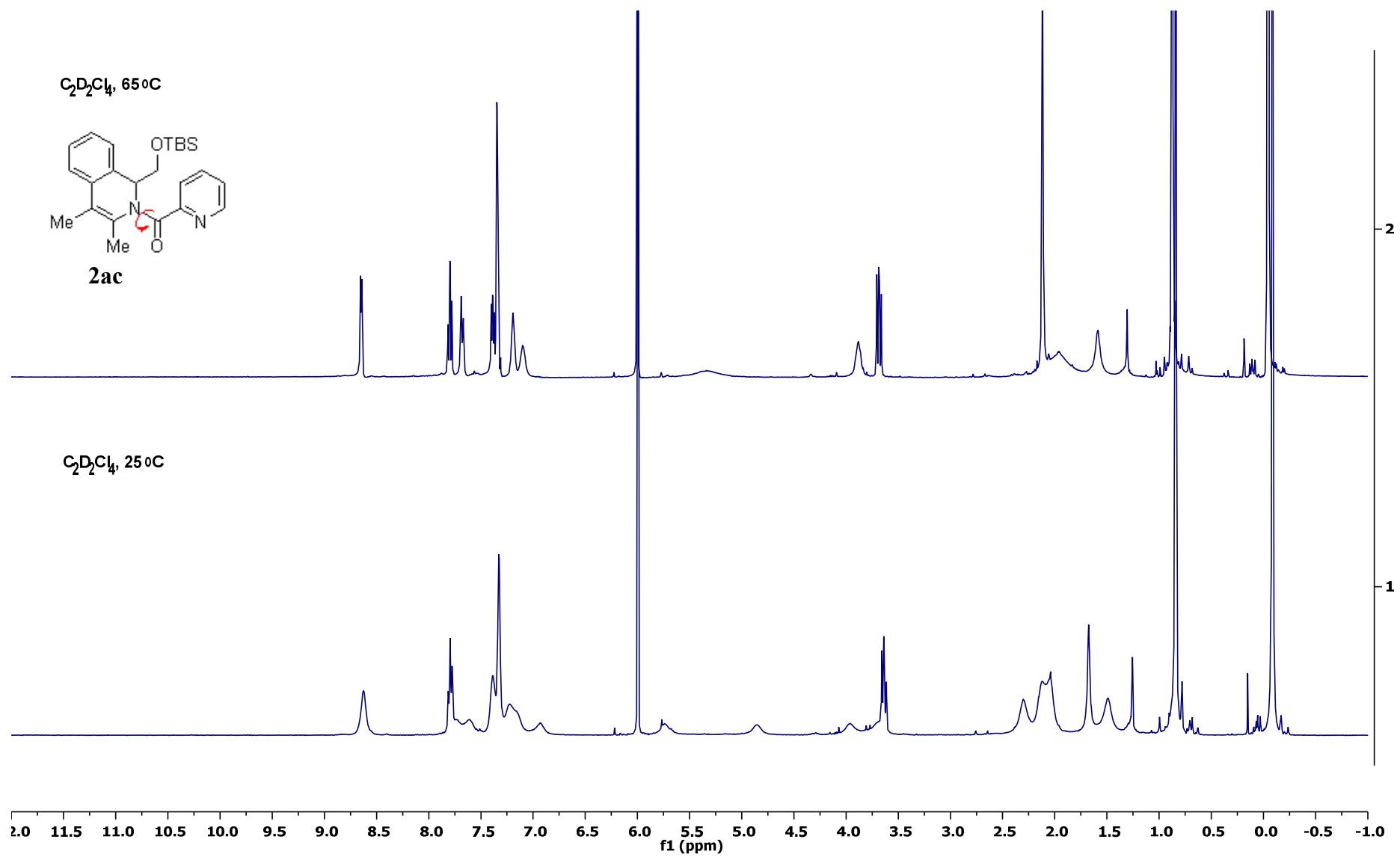
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



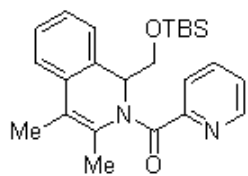
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

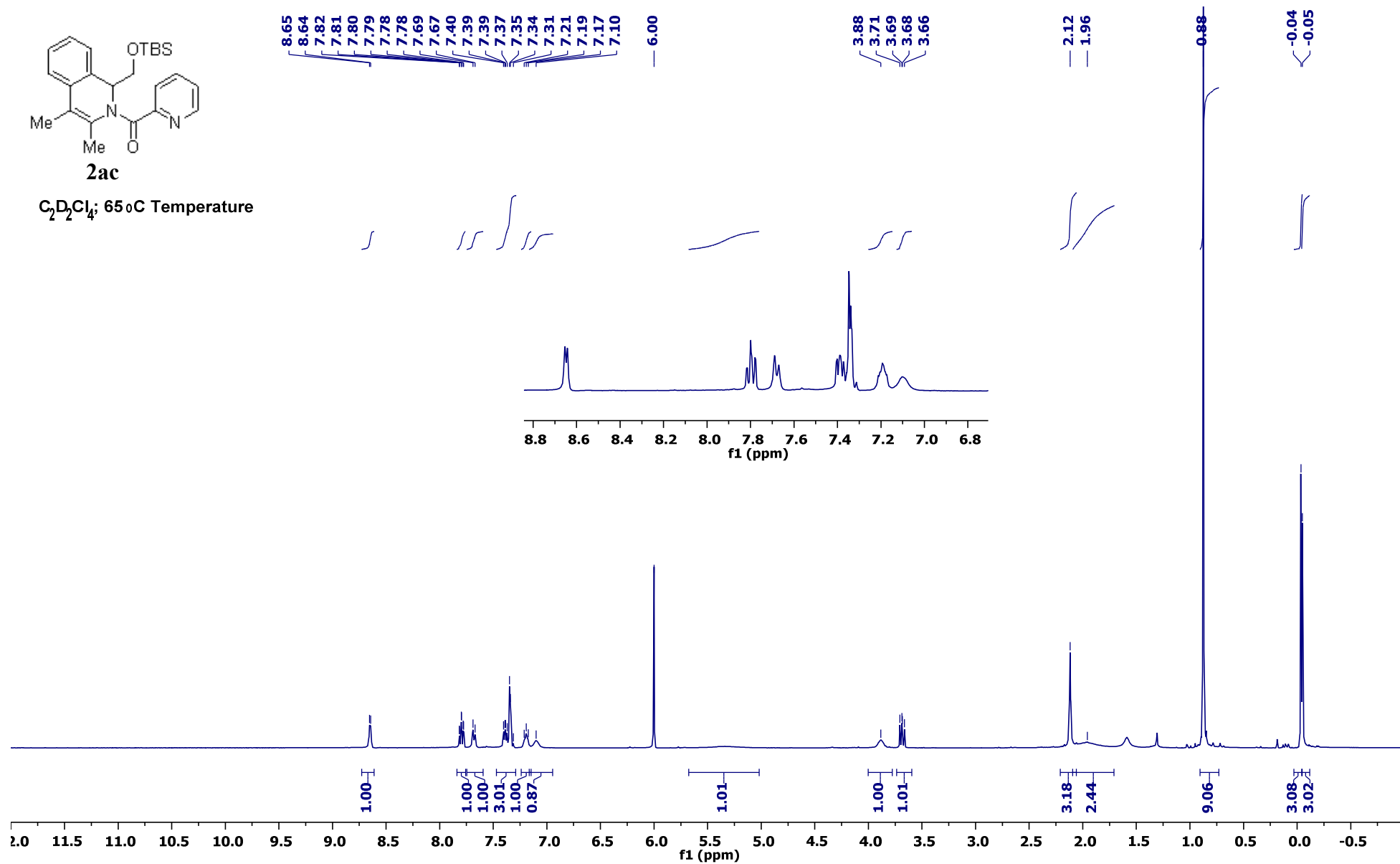


$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

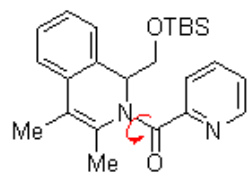


**2ac**

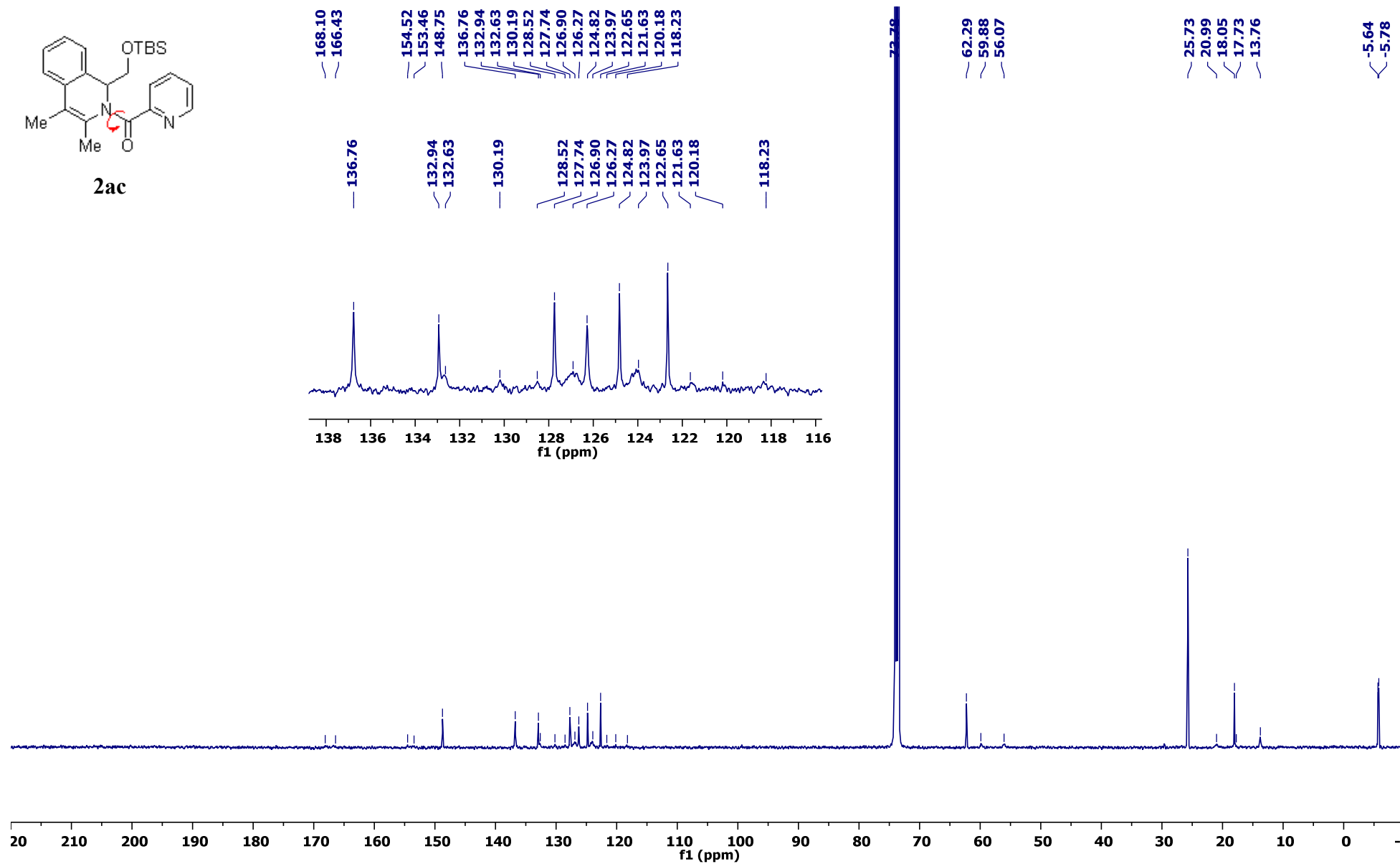
$\text{C}_2\text{D}_2\text{Cl}_4$ ; 65°C Temperature



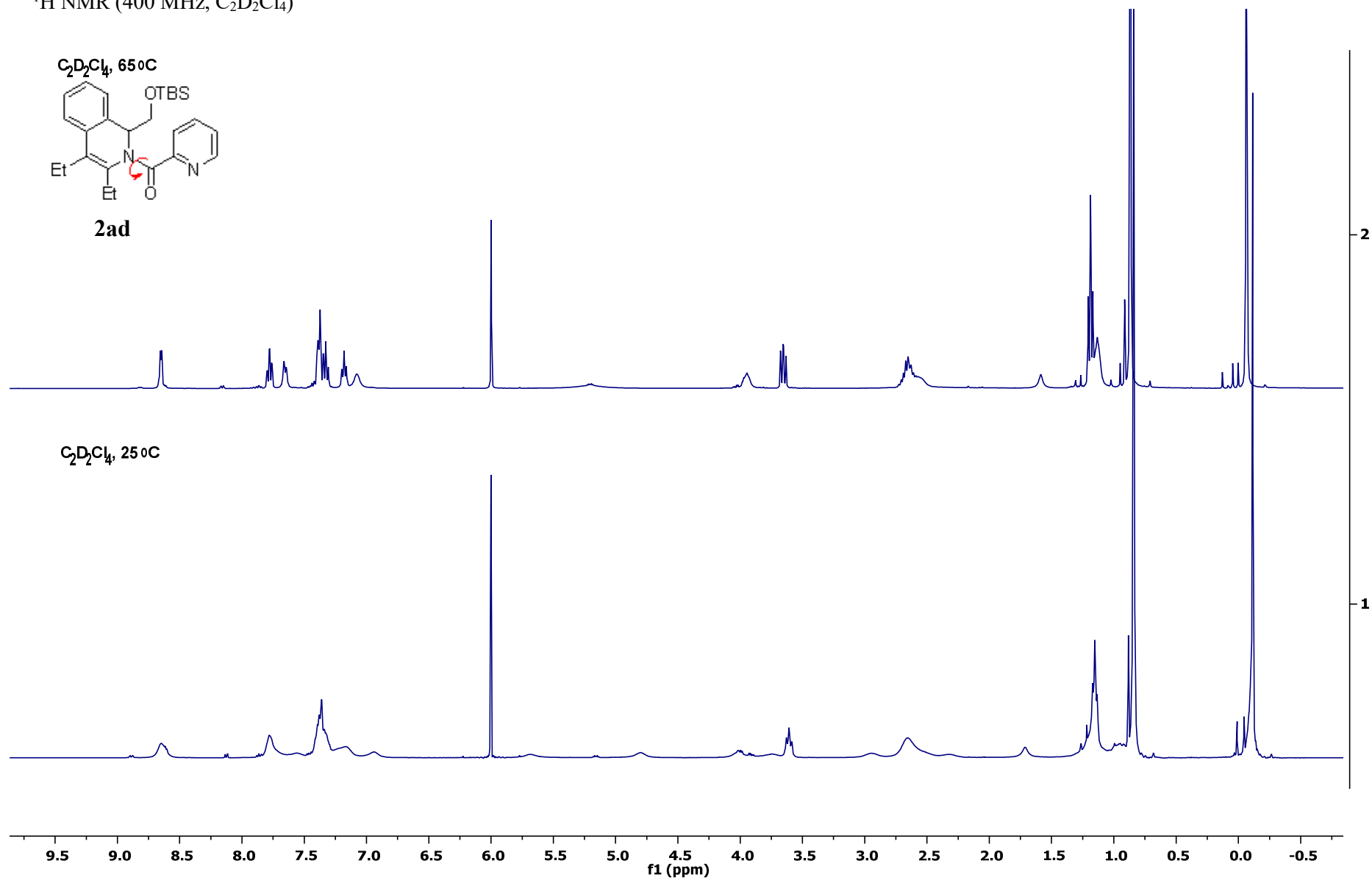
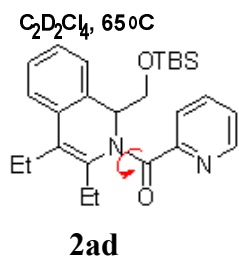
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



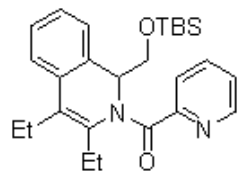
**2ac**



$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

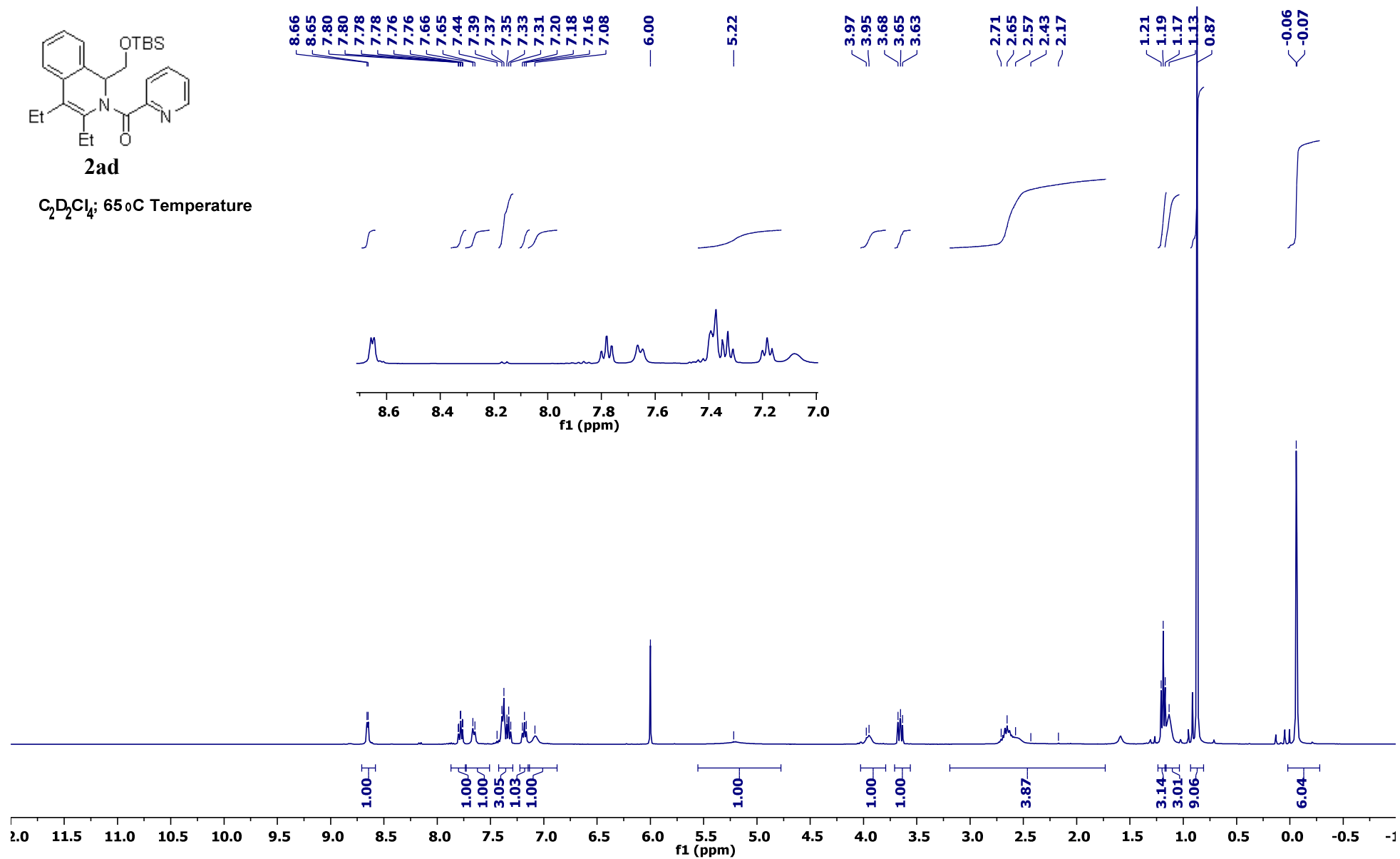


$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

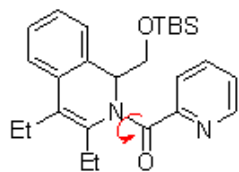


**2ad**

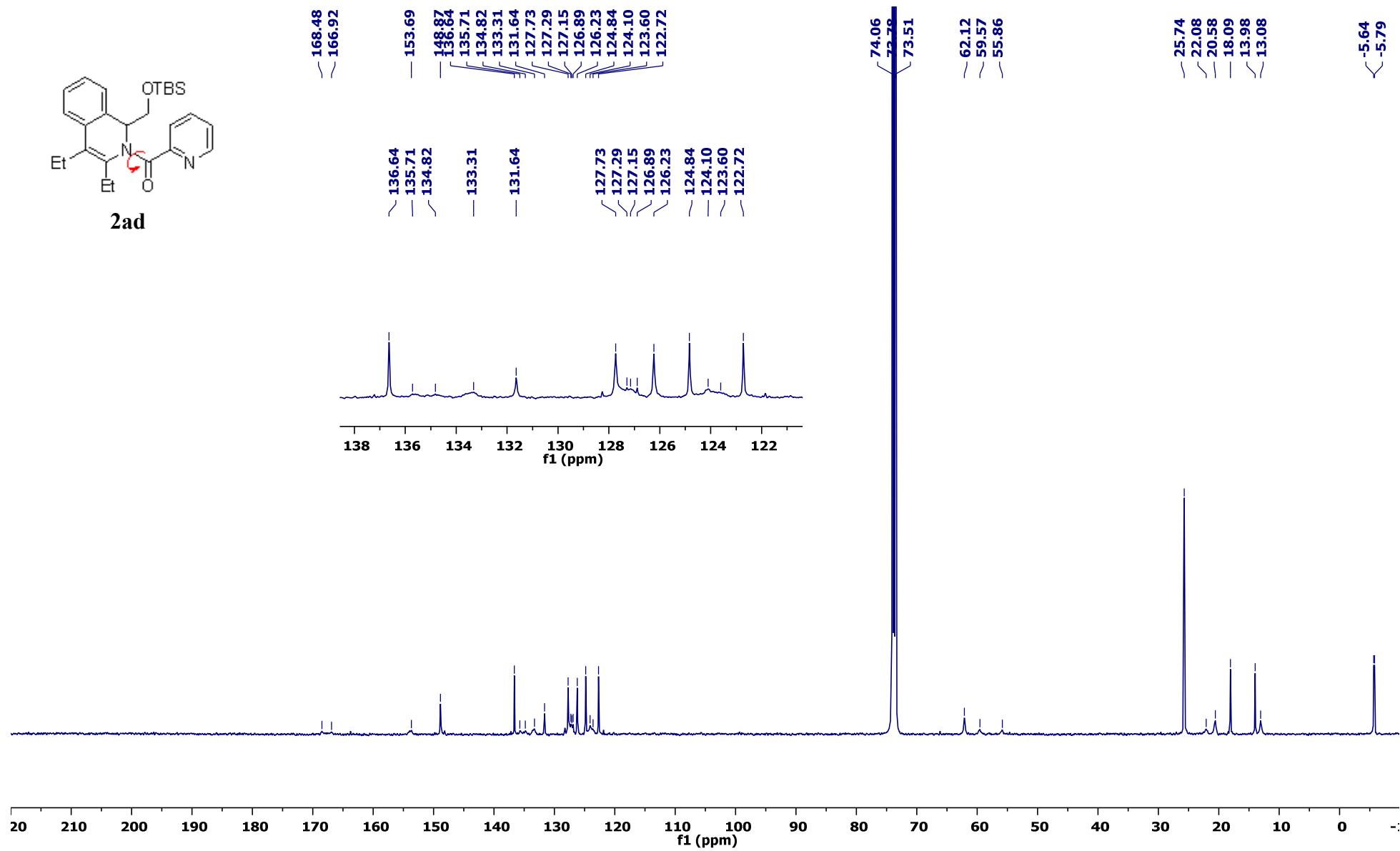
$\text{C}_2\text{D}_2\text{Cl}_4$ ; 65°C Temperature



$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

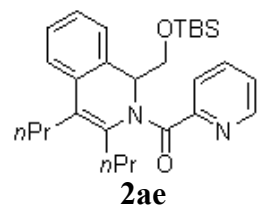


**2ad**

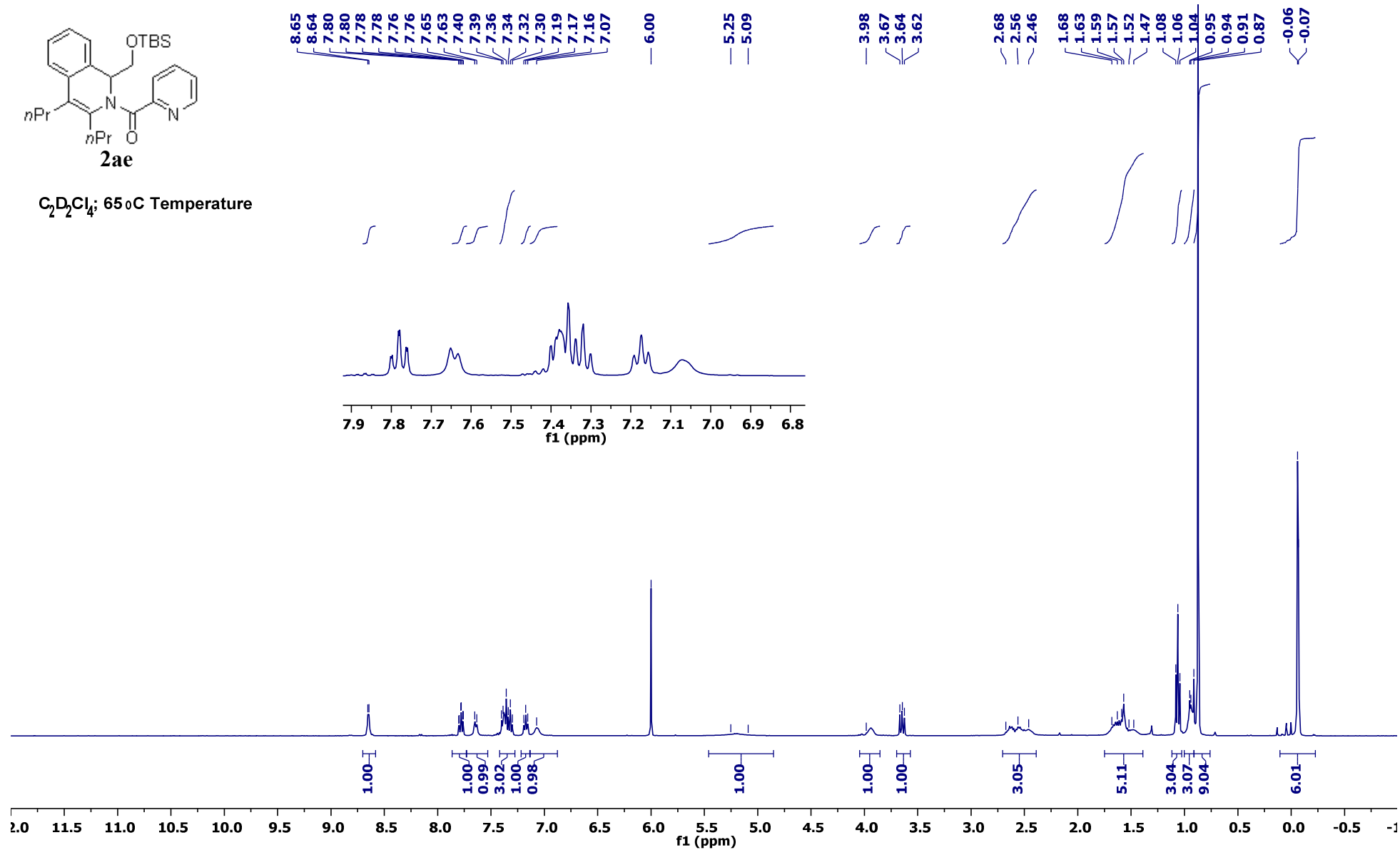




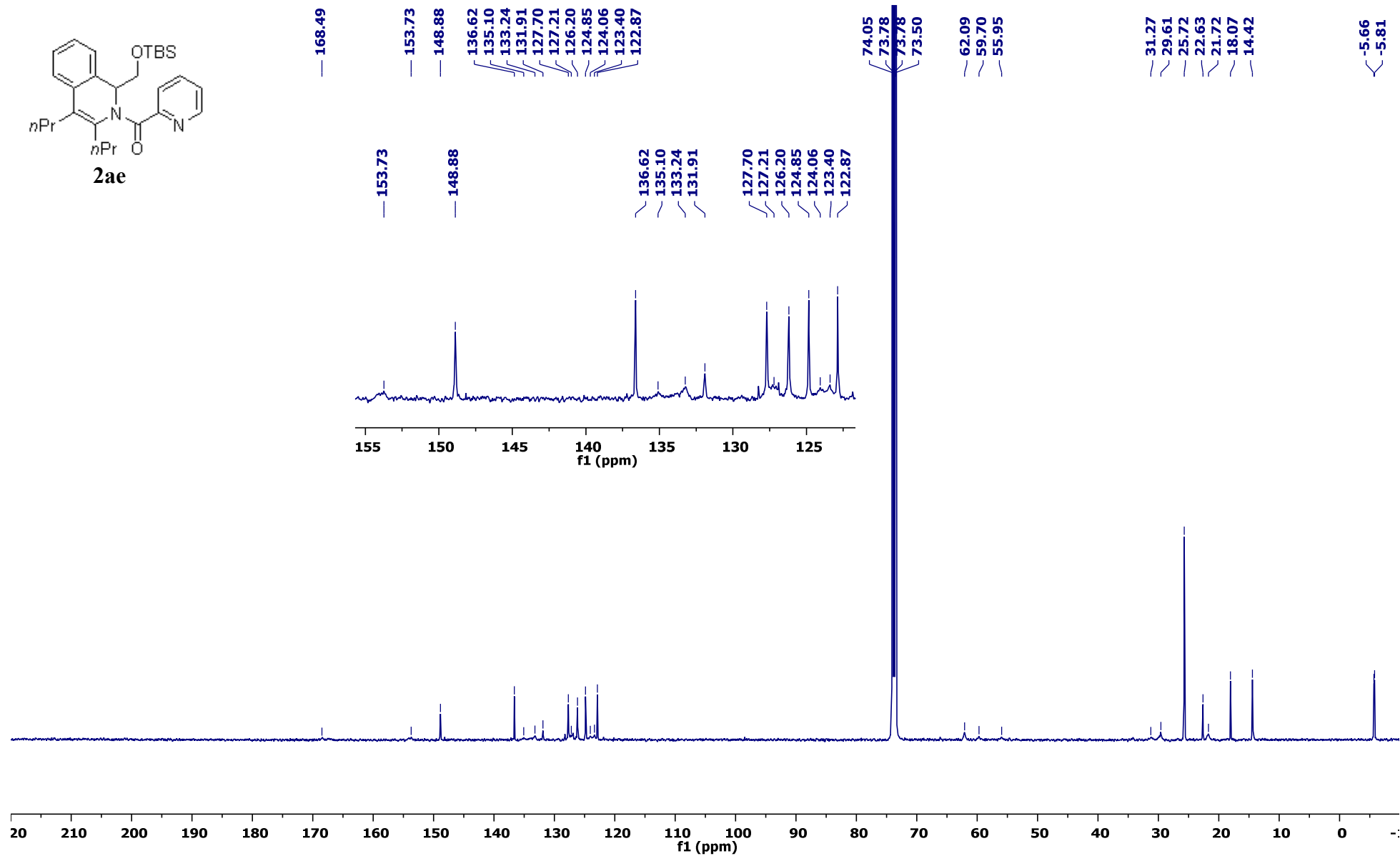
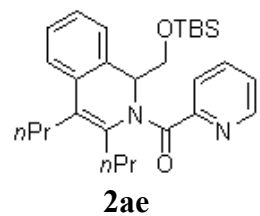
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



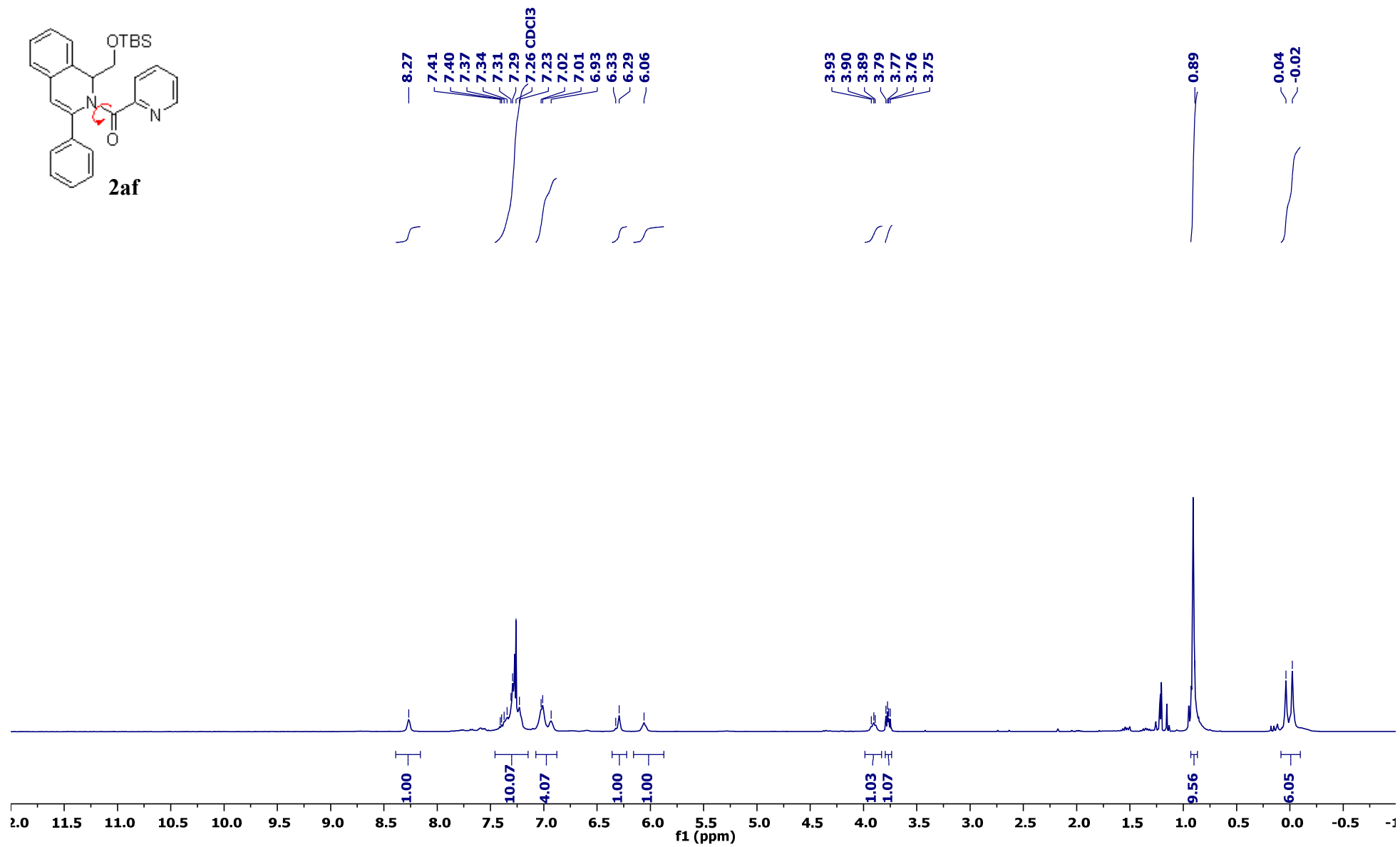
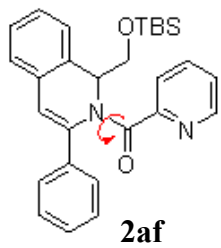
$\text{C}_2\text{D}_2\text{Cl}_4$ ; 65°C Temperature



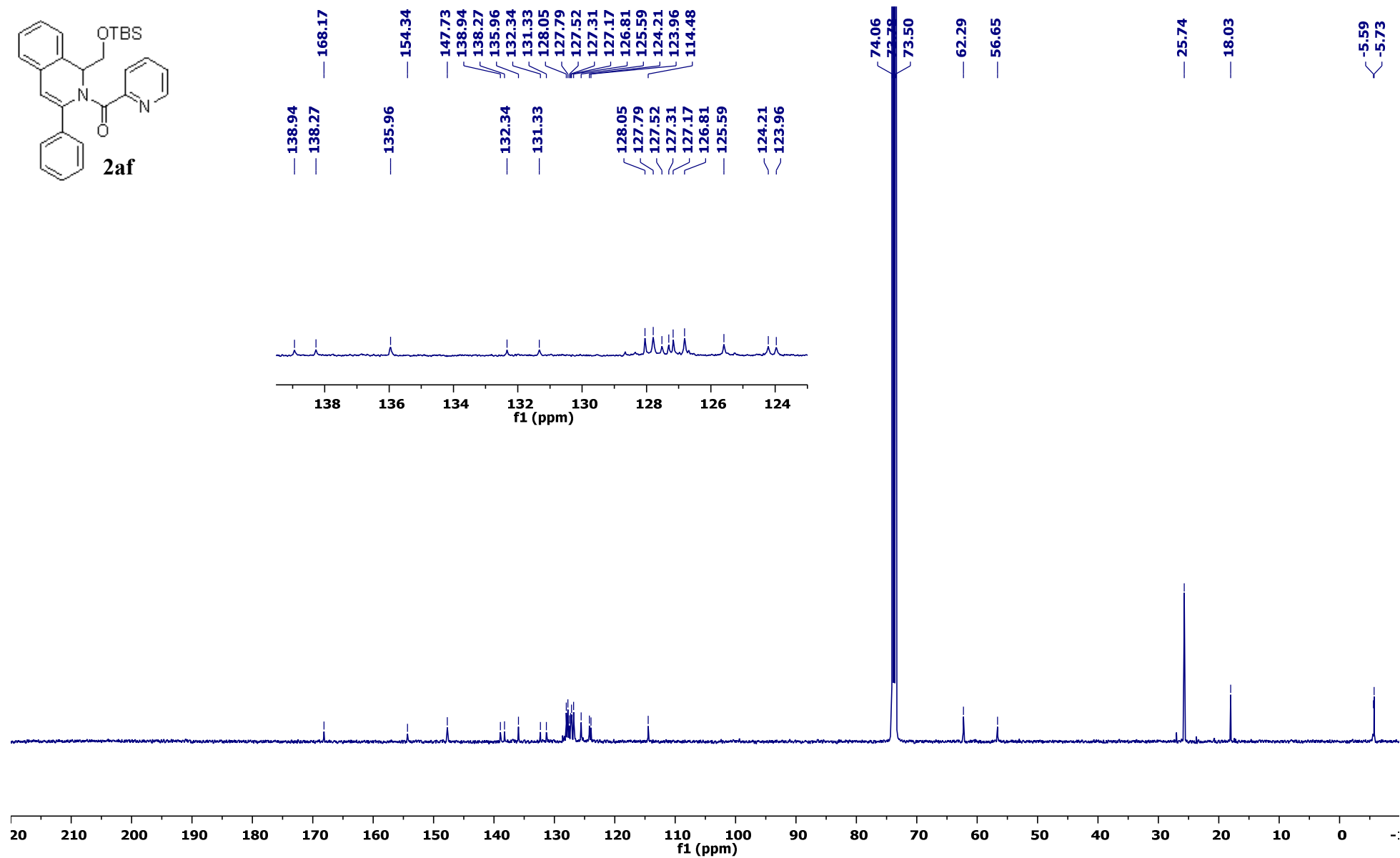
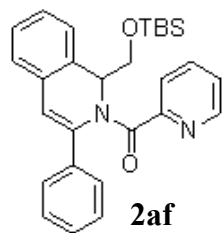
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

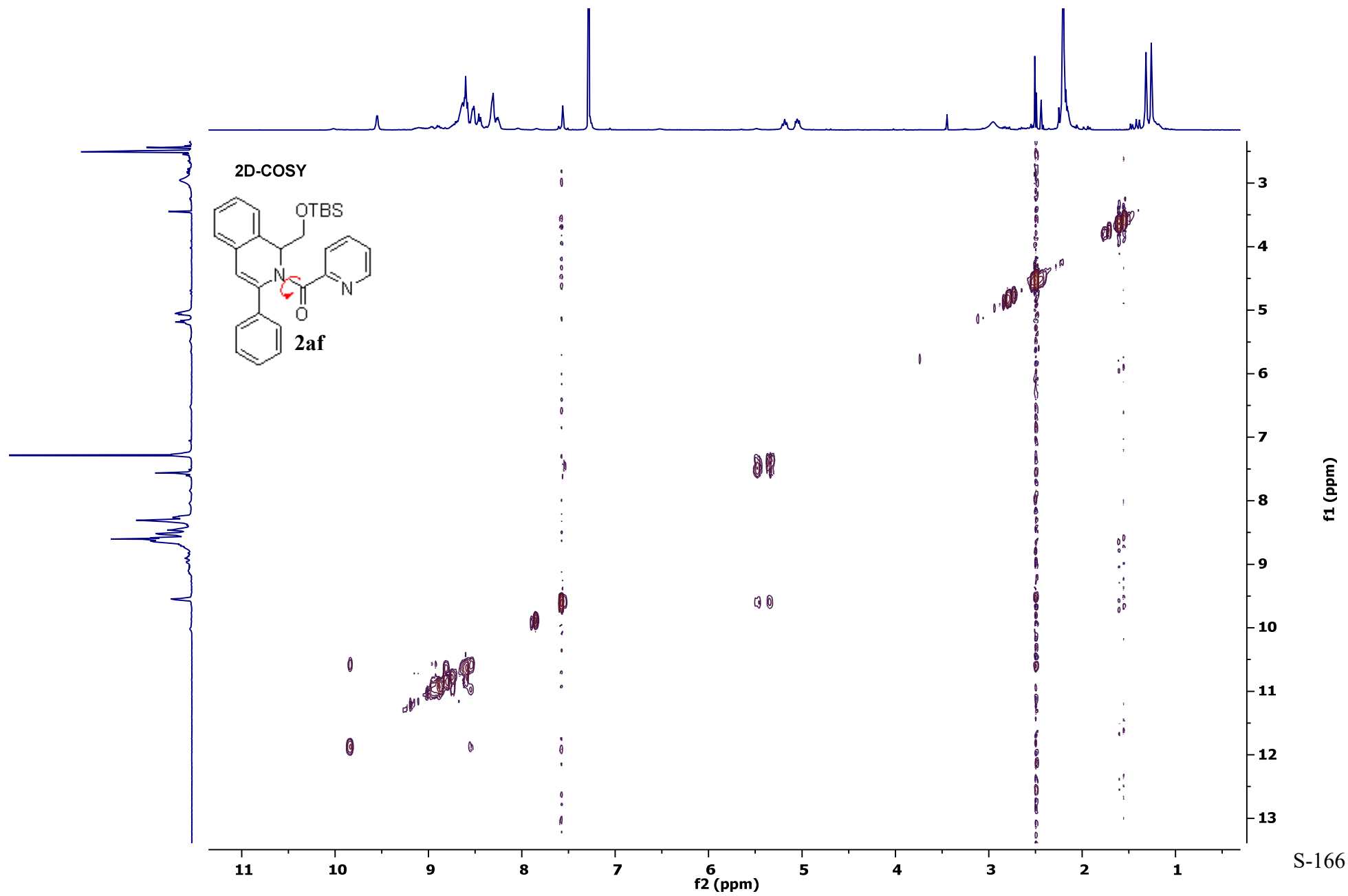


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

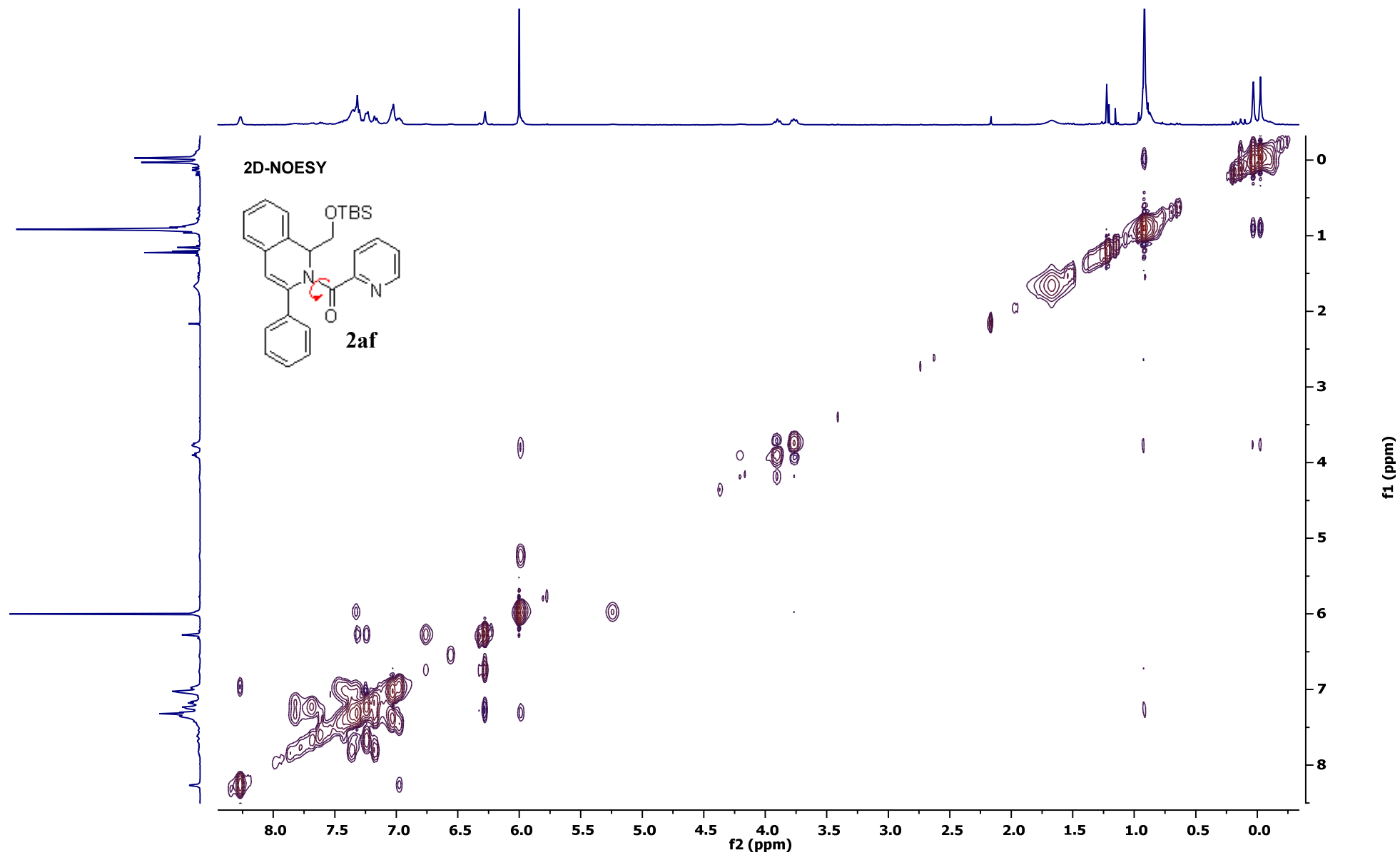


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

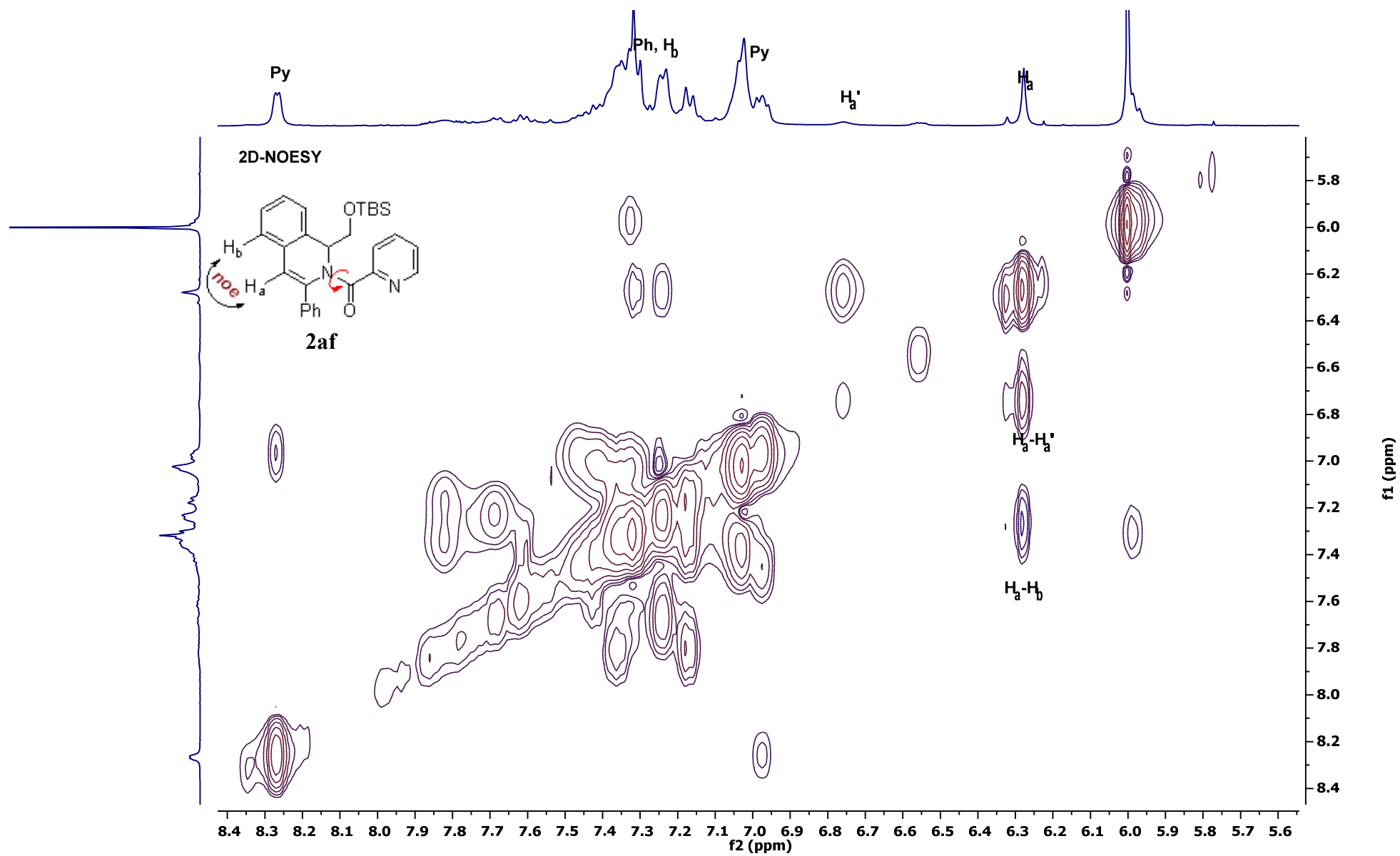




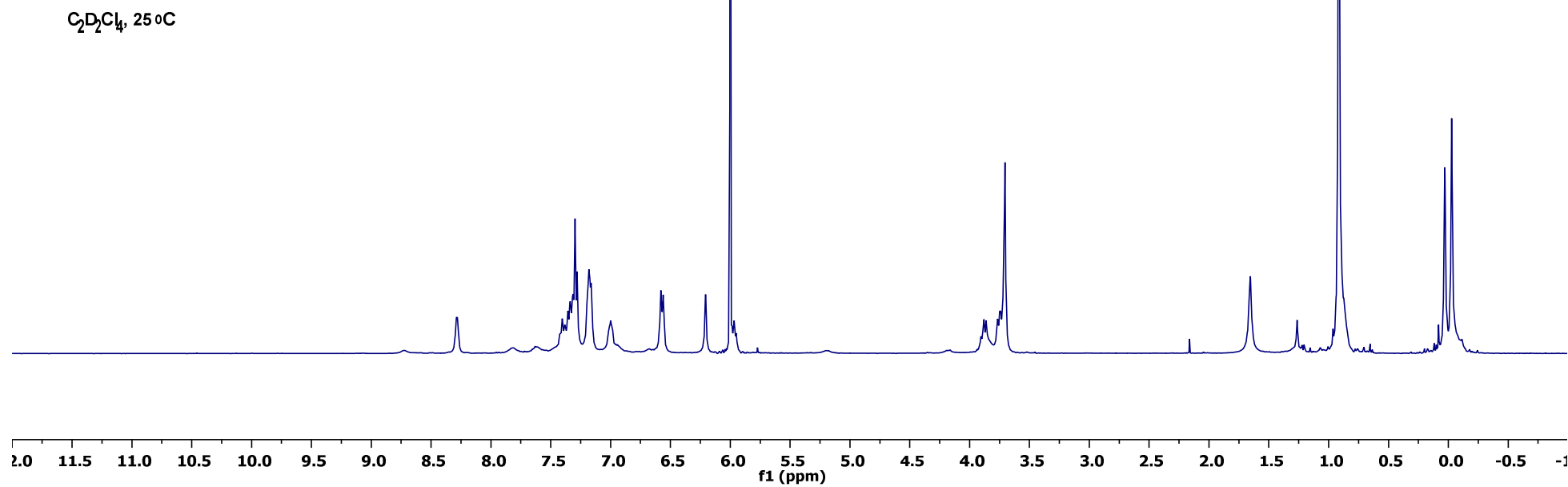
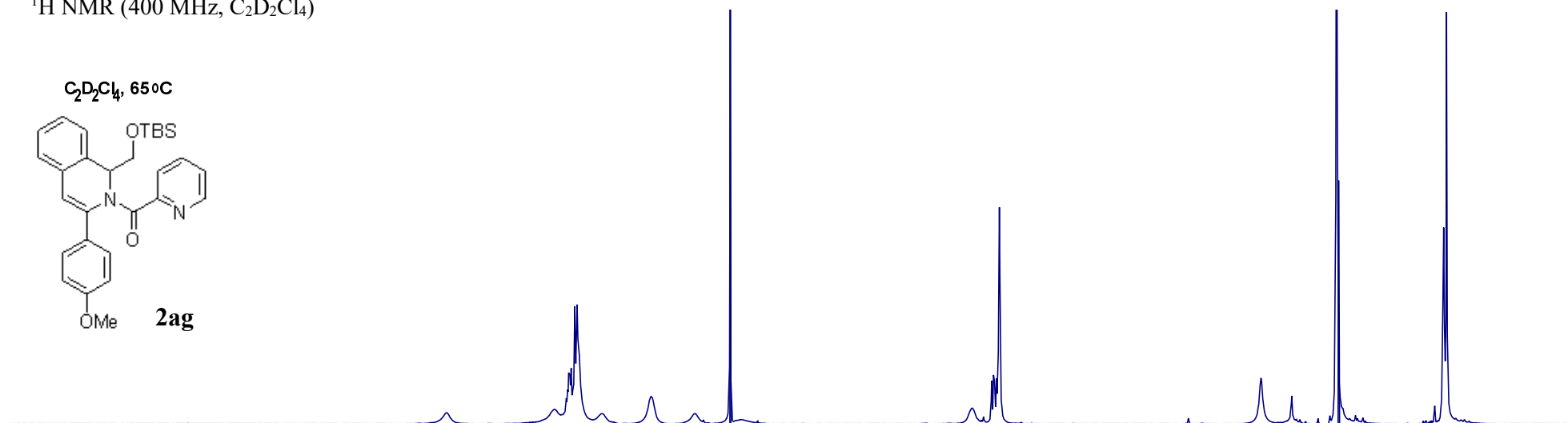
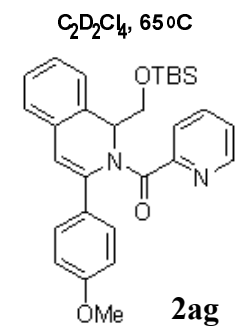
2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)



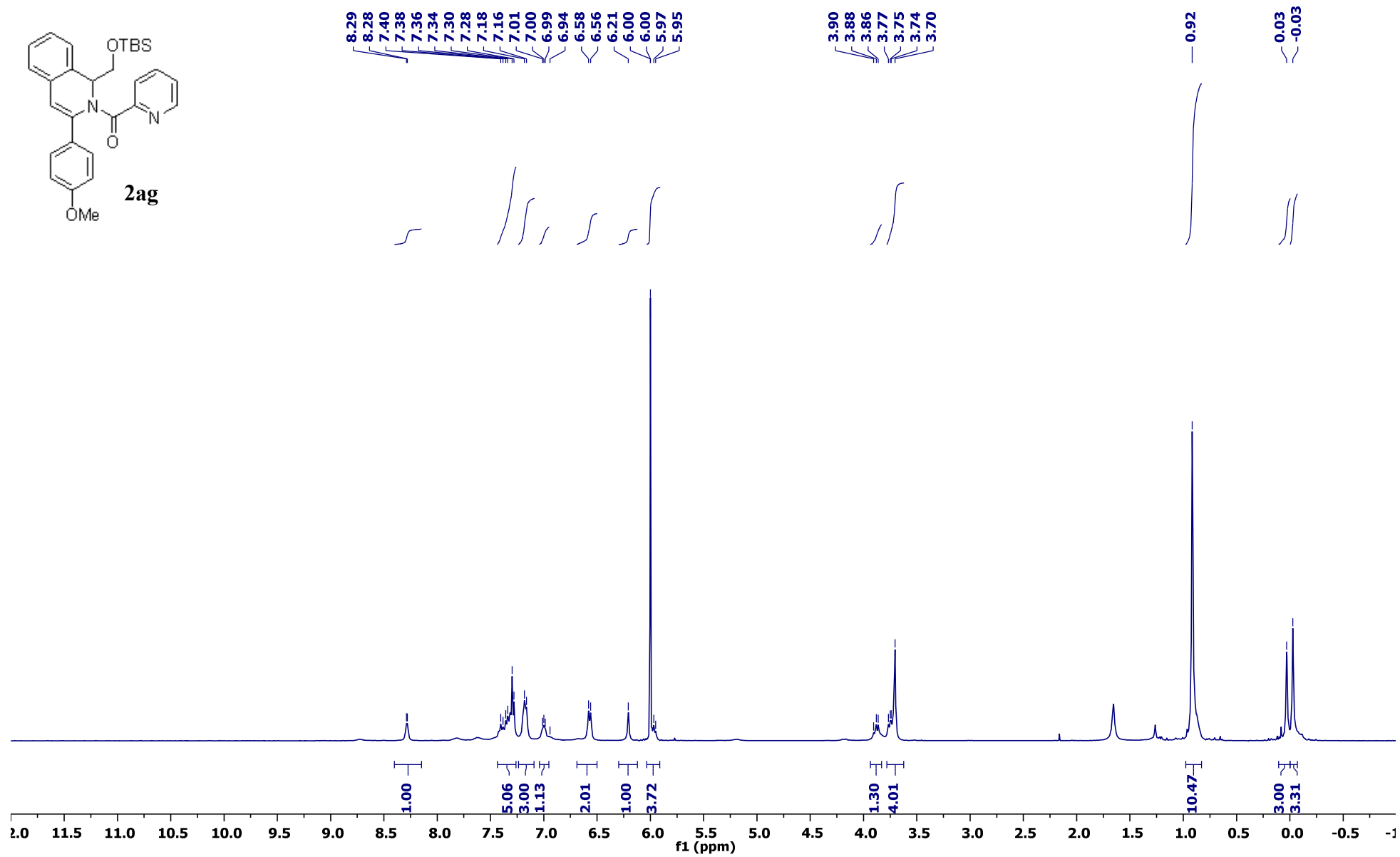
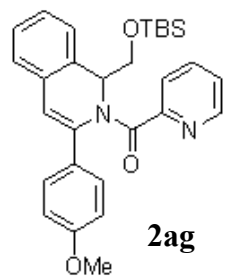
2D (COSY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)



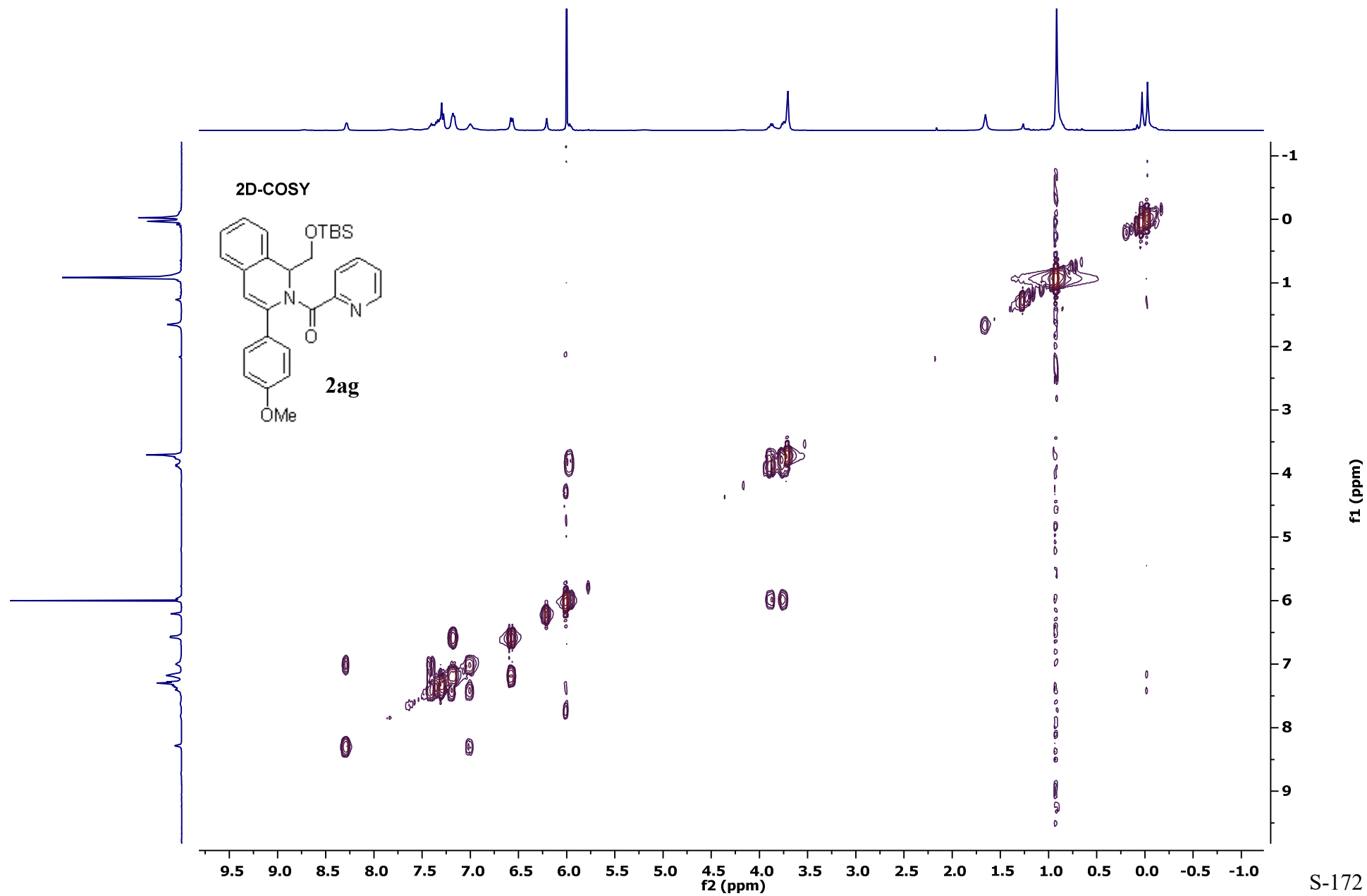
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



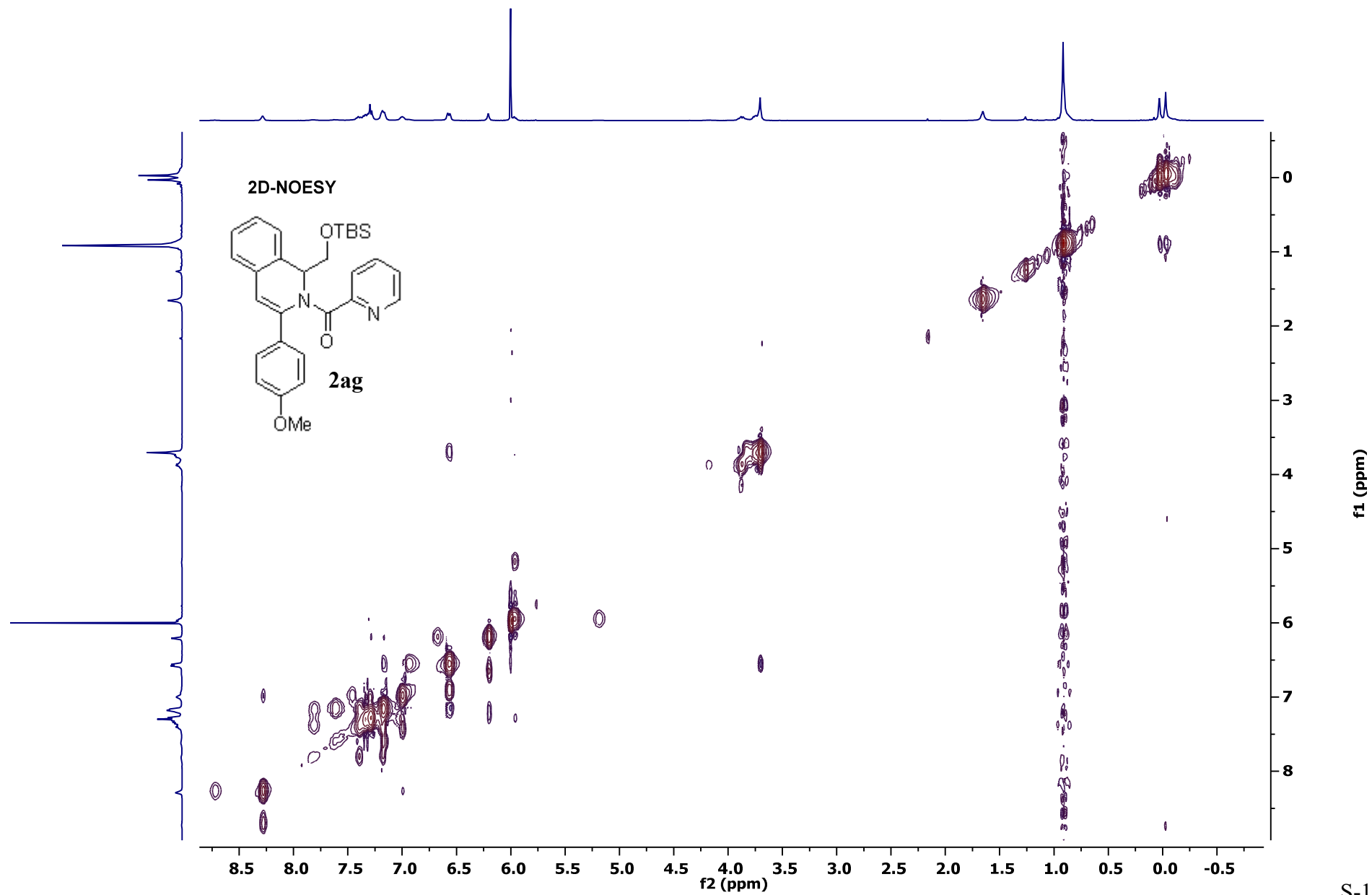
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



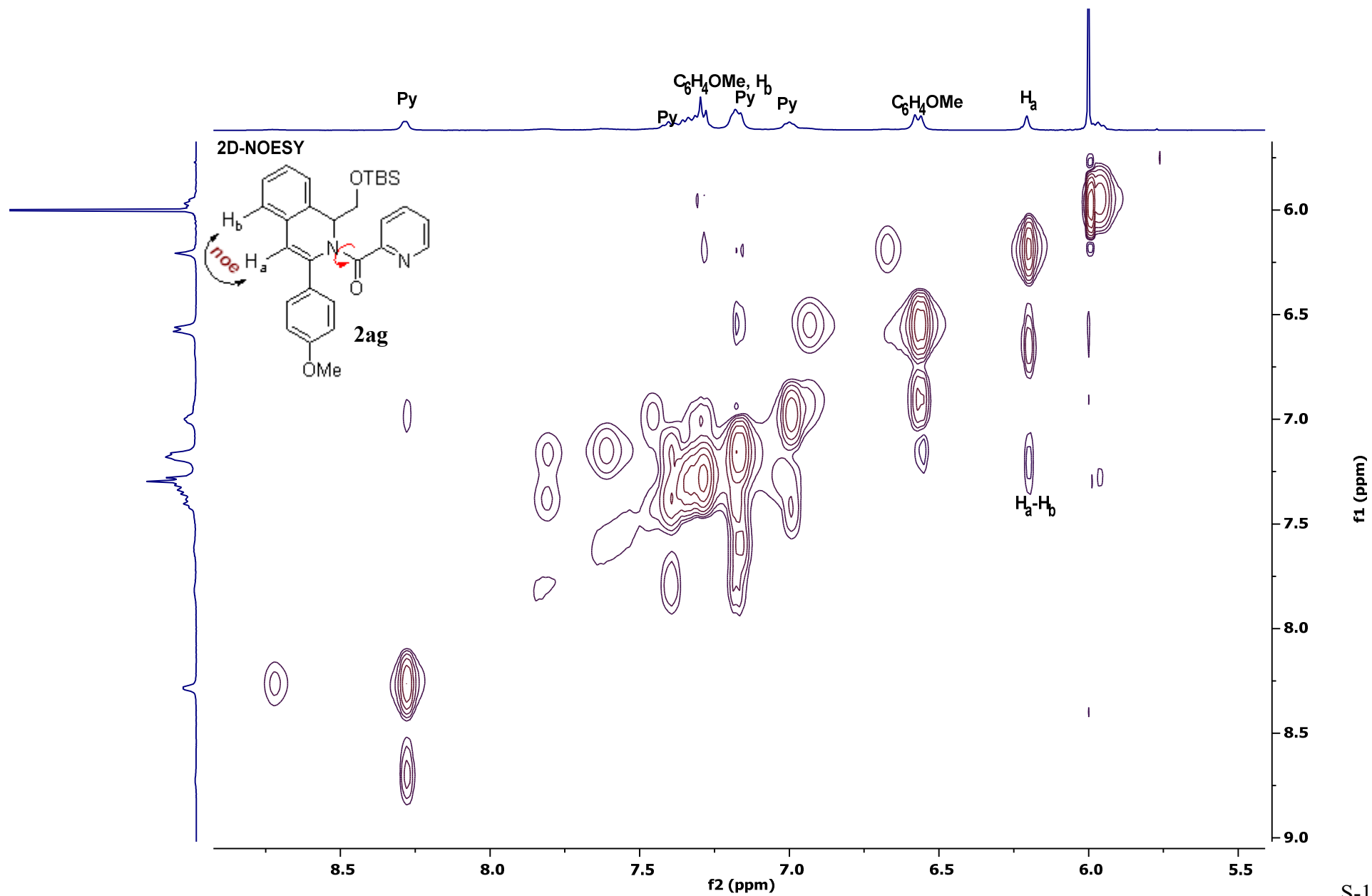




2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

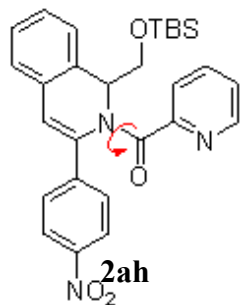


2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

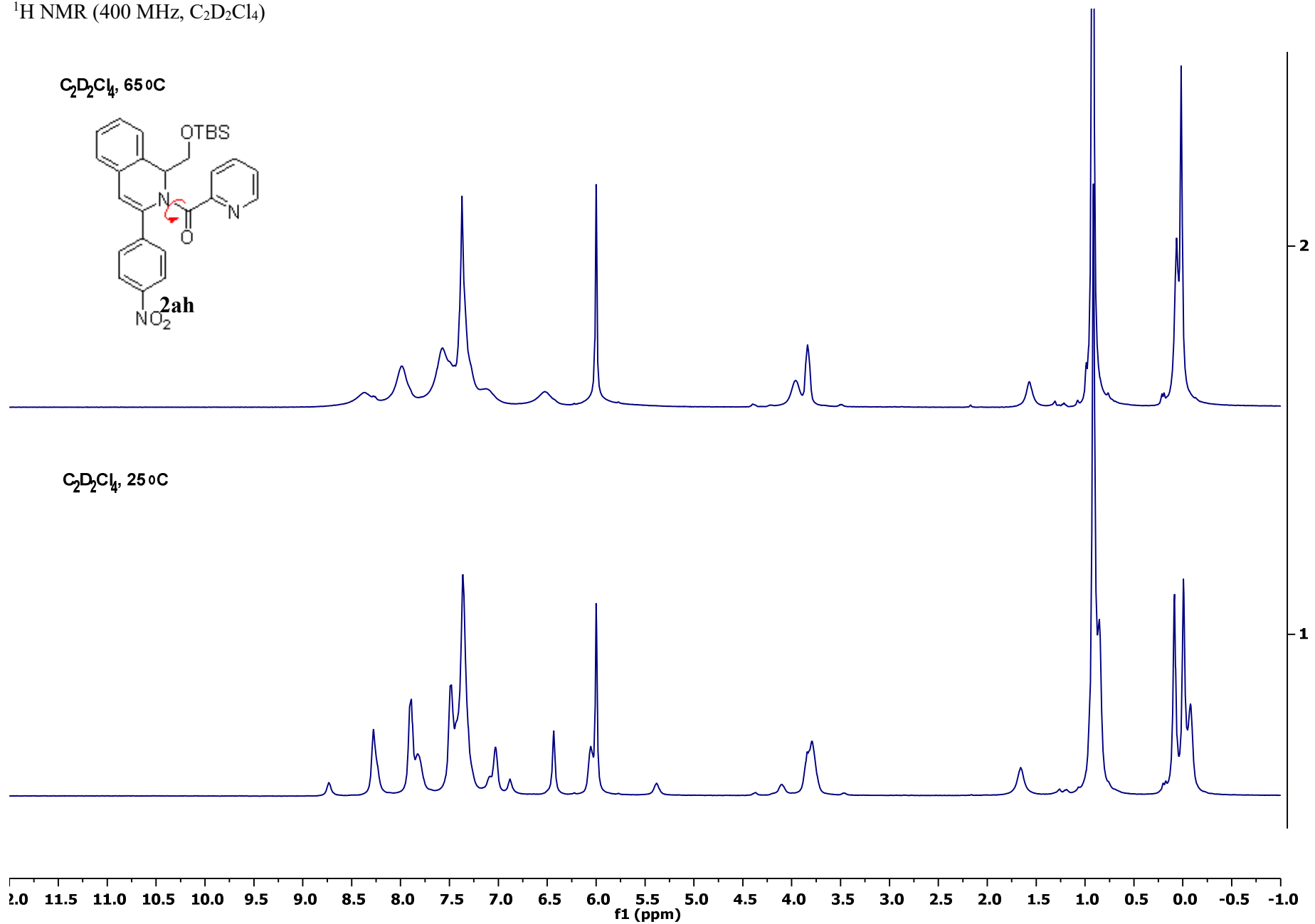


$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

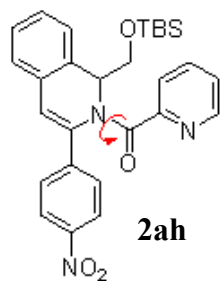
$\text{C}_2\text{D}_2\text{Cl}_4$ , 65°C



**2ah**



$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



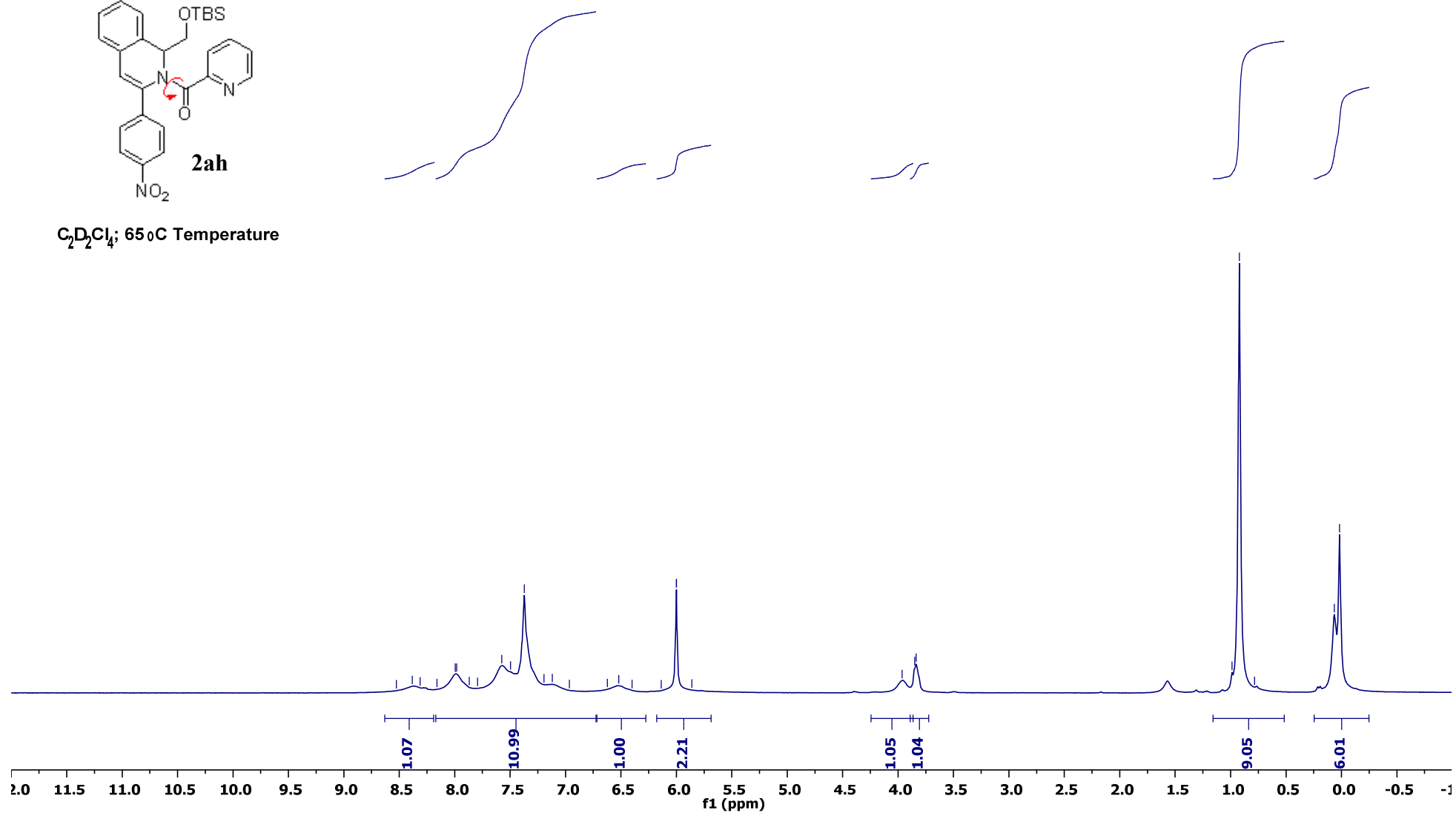
$\text{C}_2\text{D}_2\text{Cl}_4$ ; 65°C Temperature

8.53  
8.38  
8.31  
8.16  
7.99  
7.98  
7.87  
7.79  
7.58  
7.50  
7.37  
7.19  
7.12  
6.97  
6.62  
6.52  
6.40  
6.14  
6.00  
6.00  
5.86

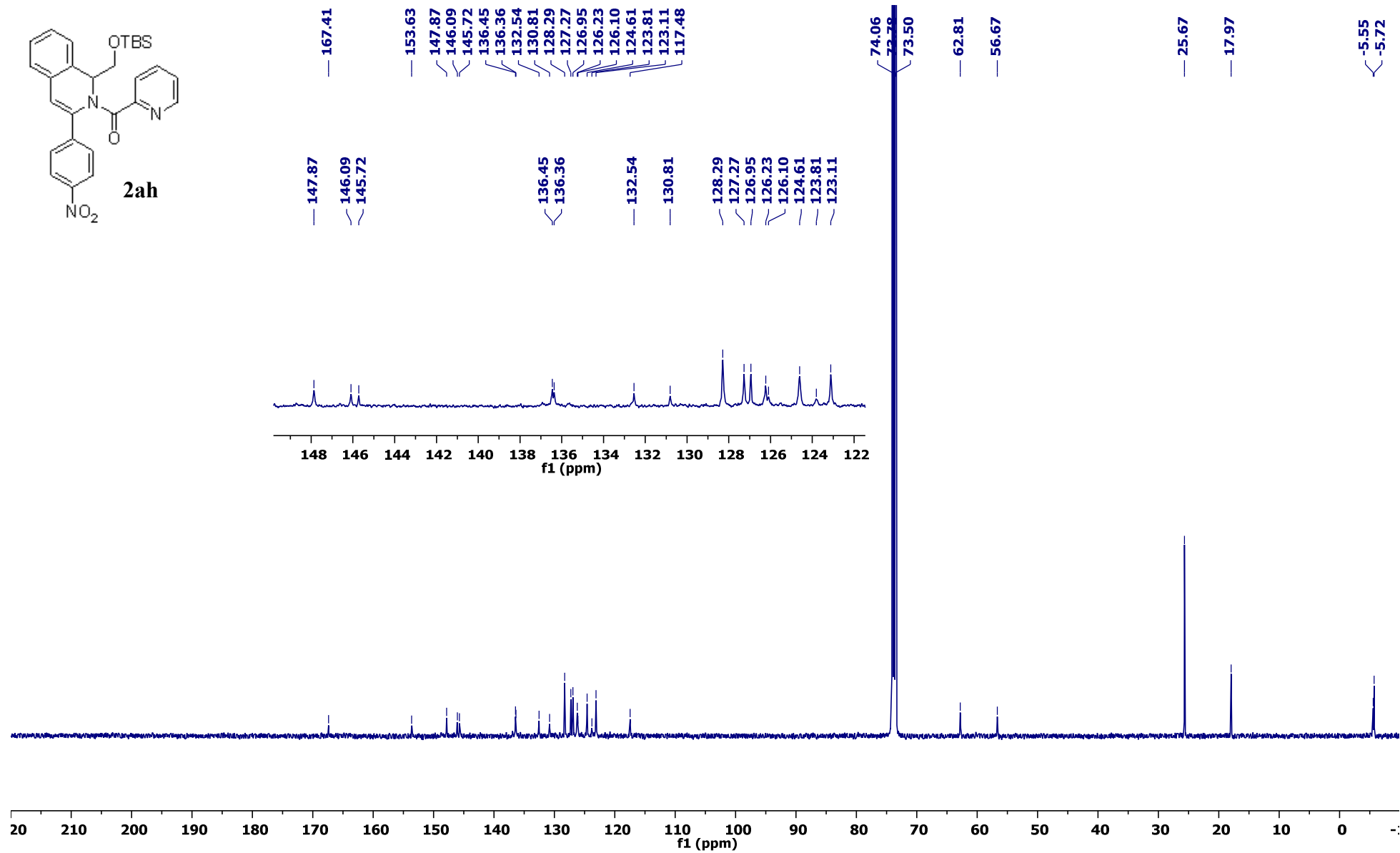
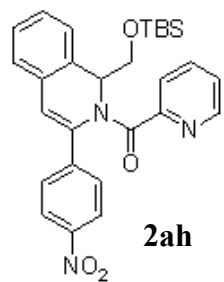
3.96  
3.85  
3.84

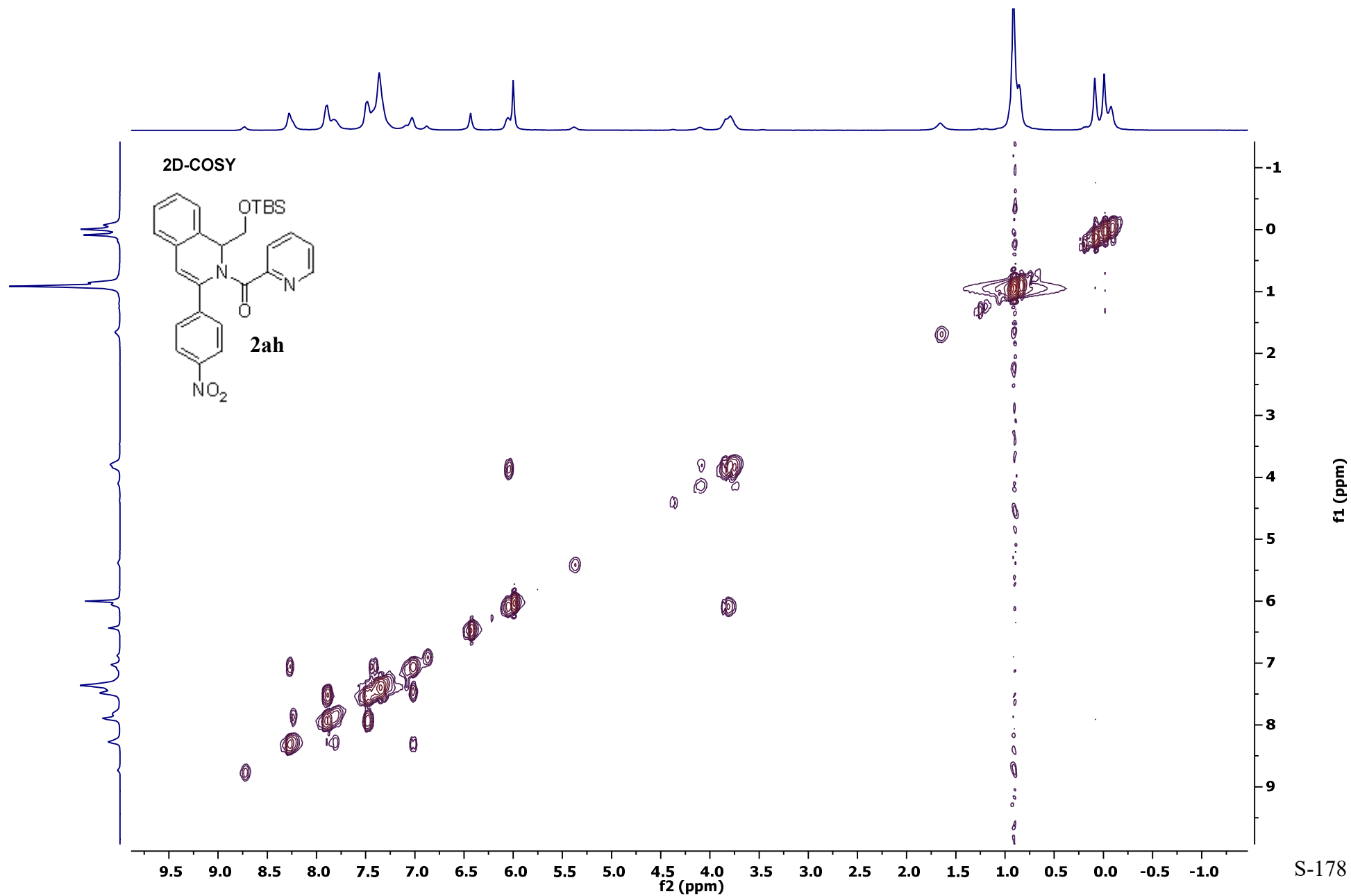
0.99  
0.92  
0.78

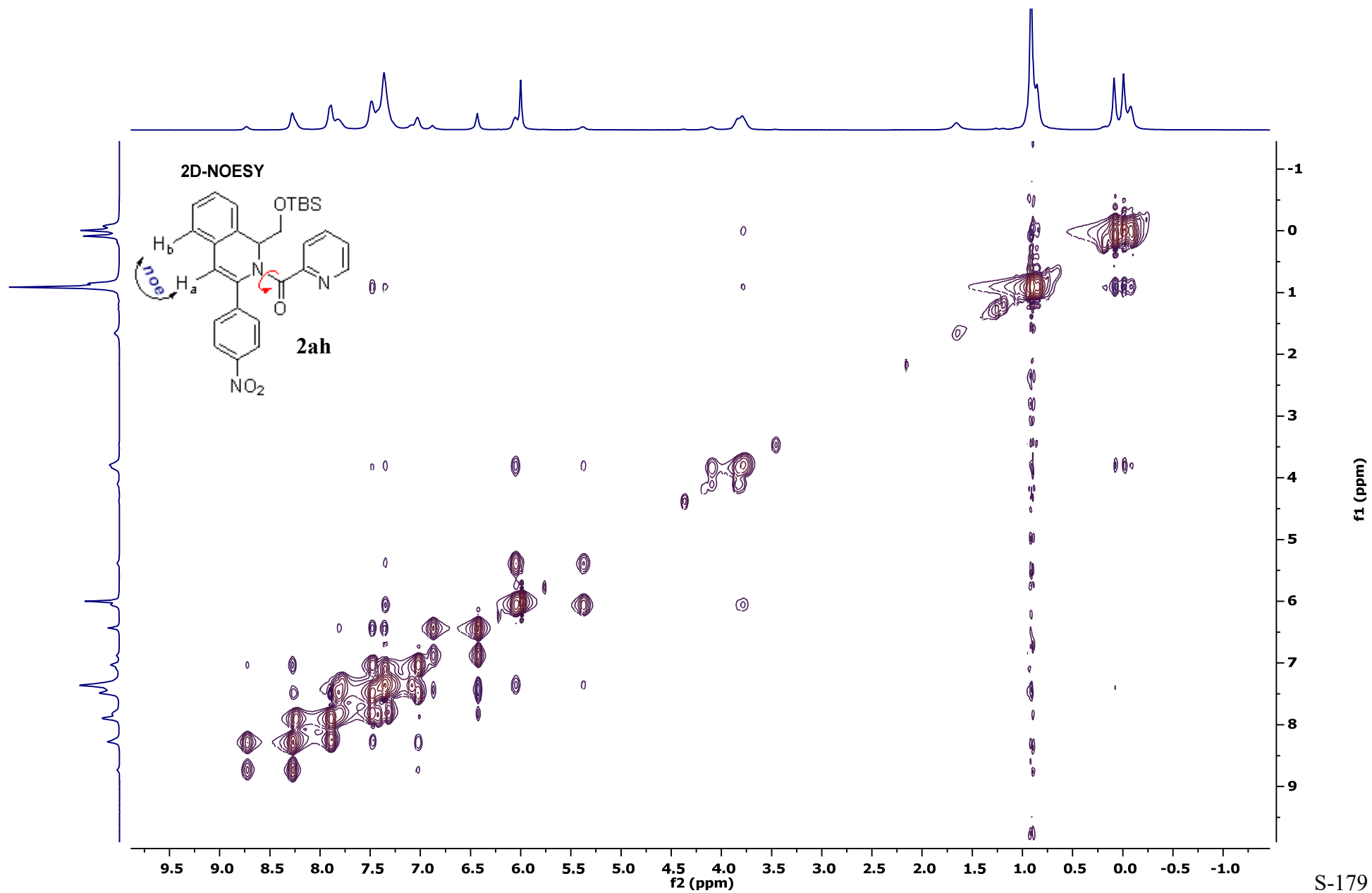
0.06  
0.02



$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

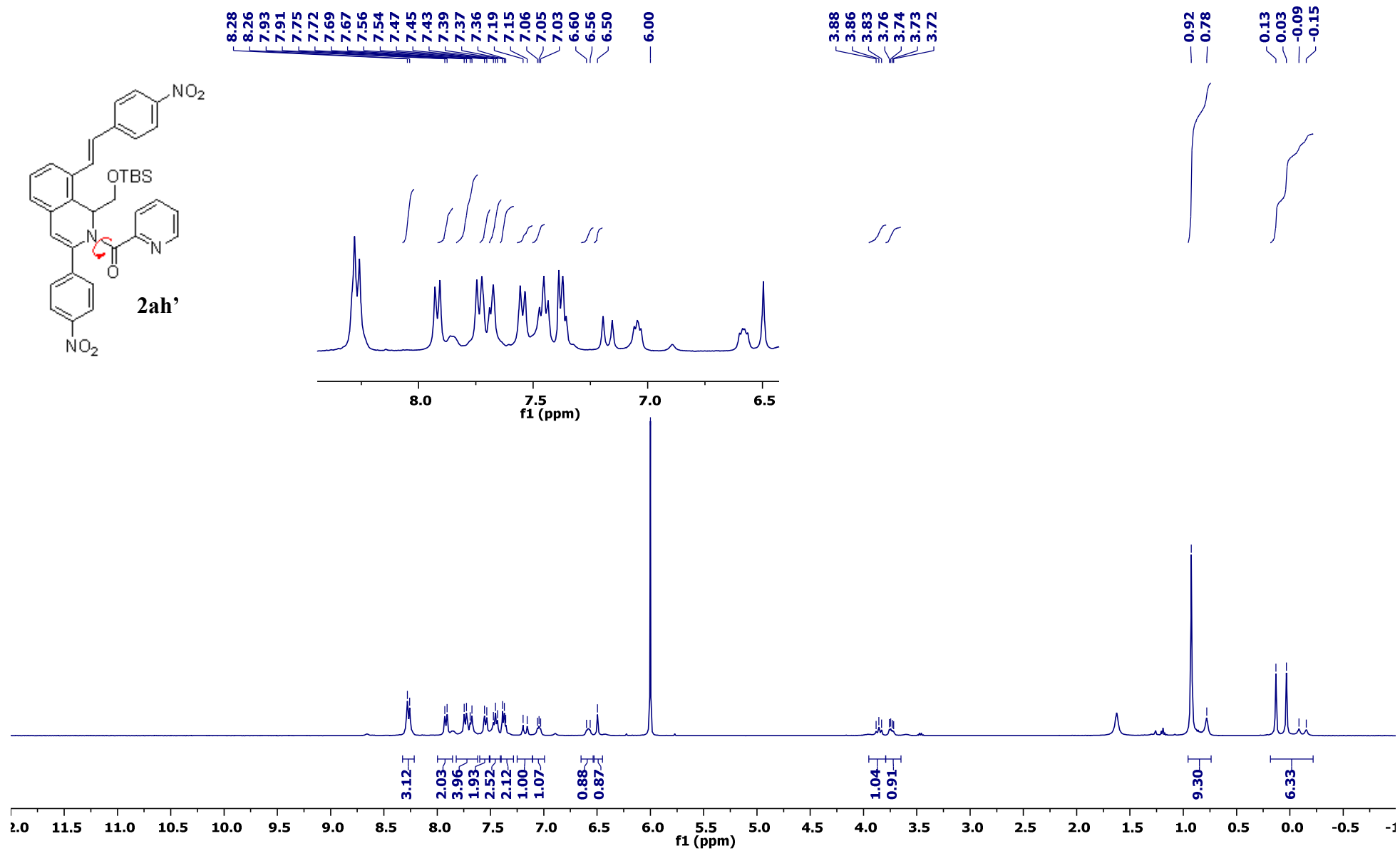




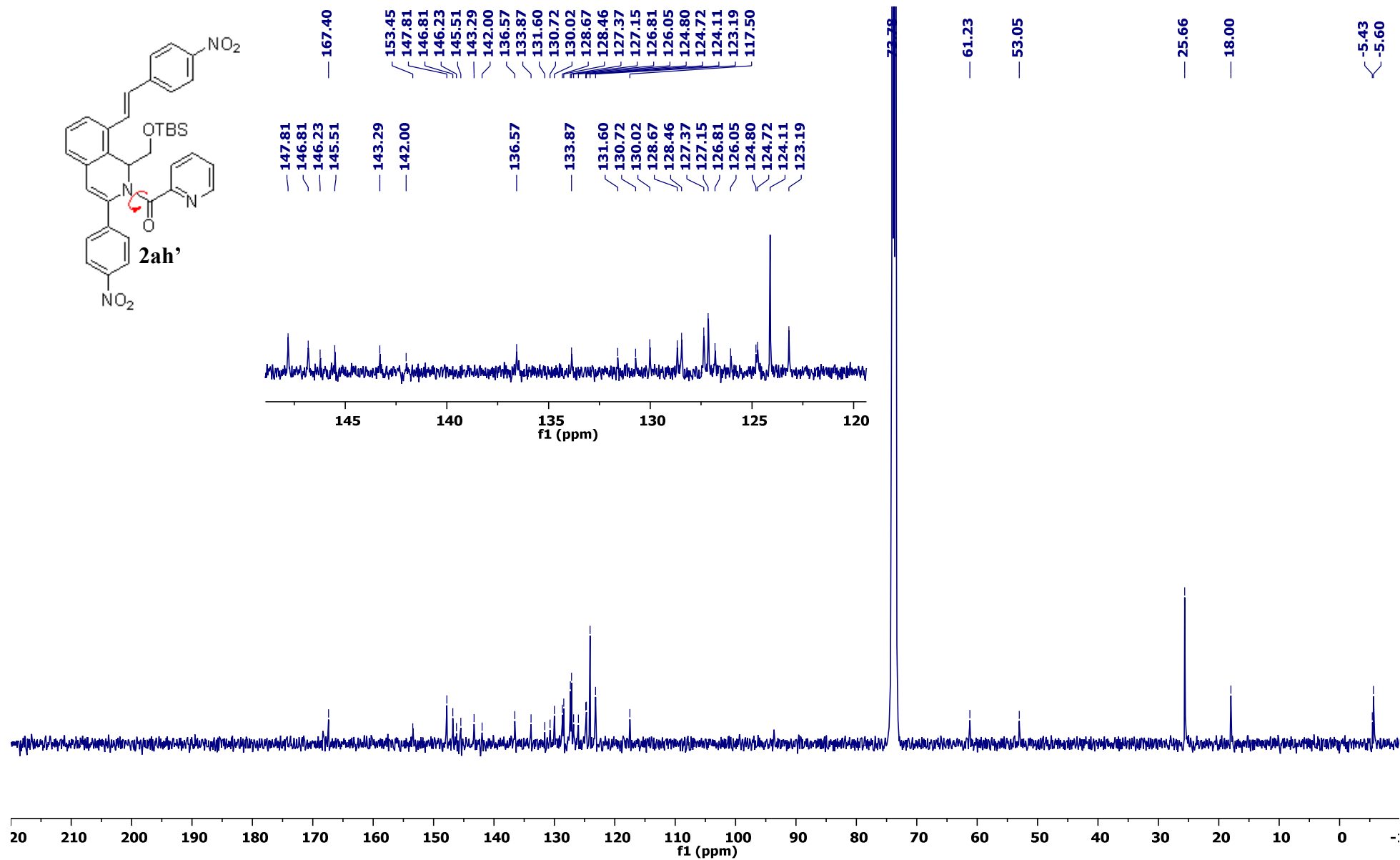


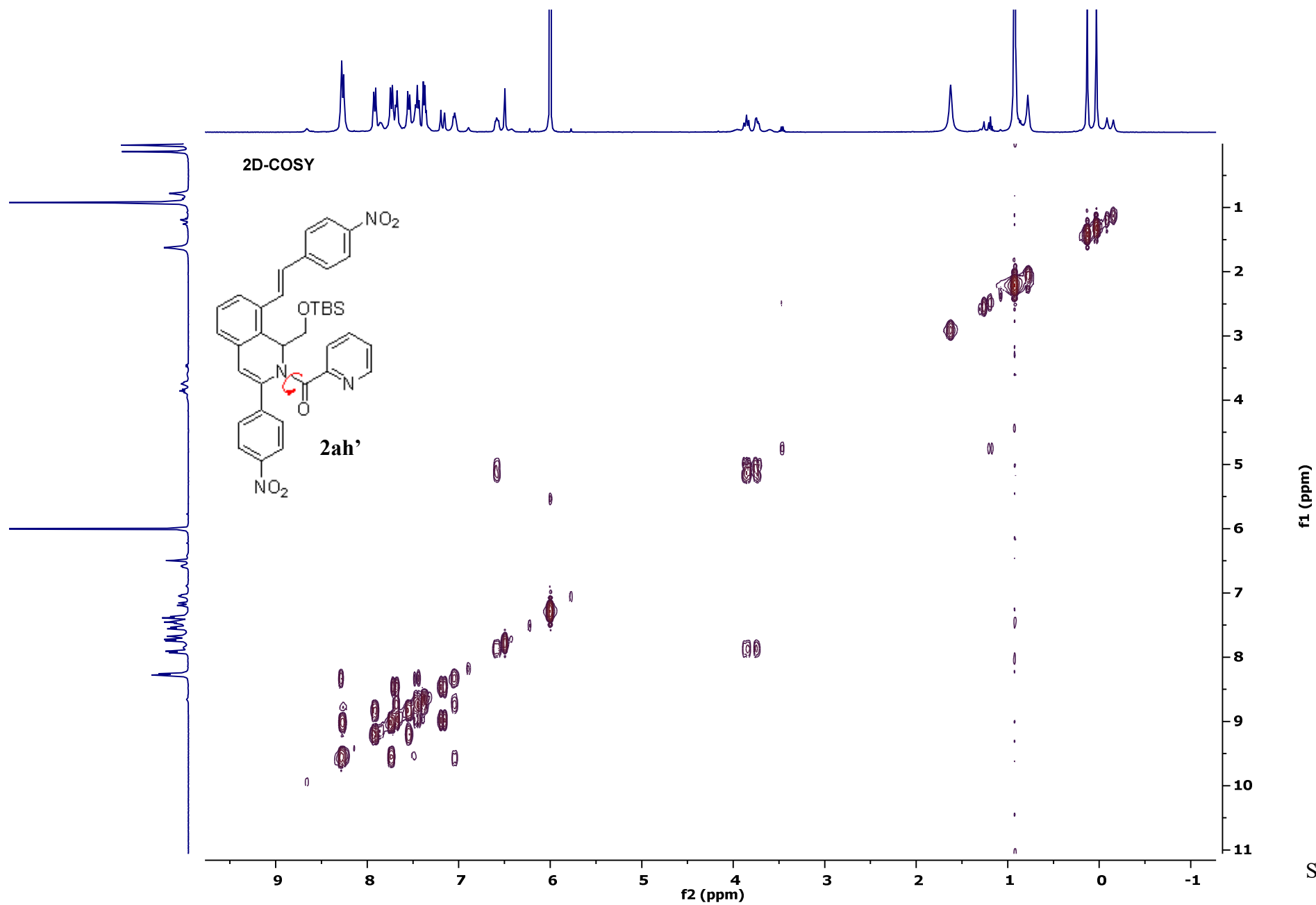


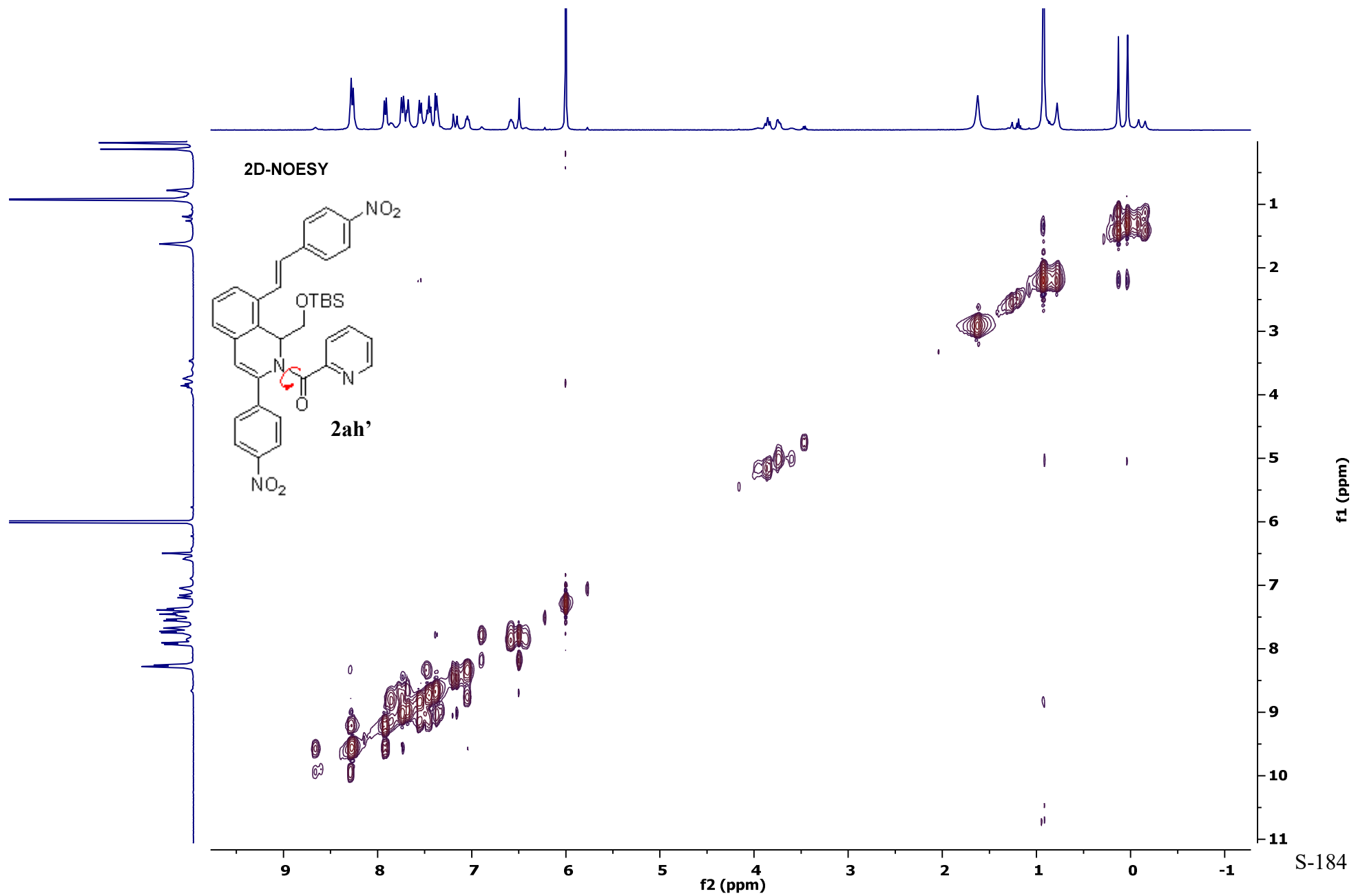
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

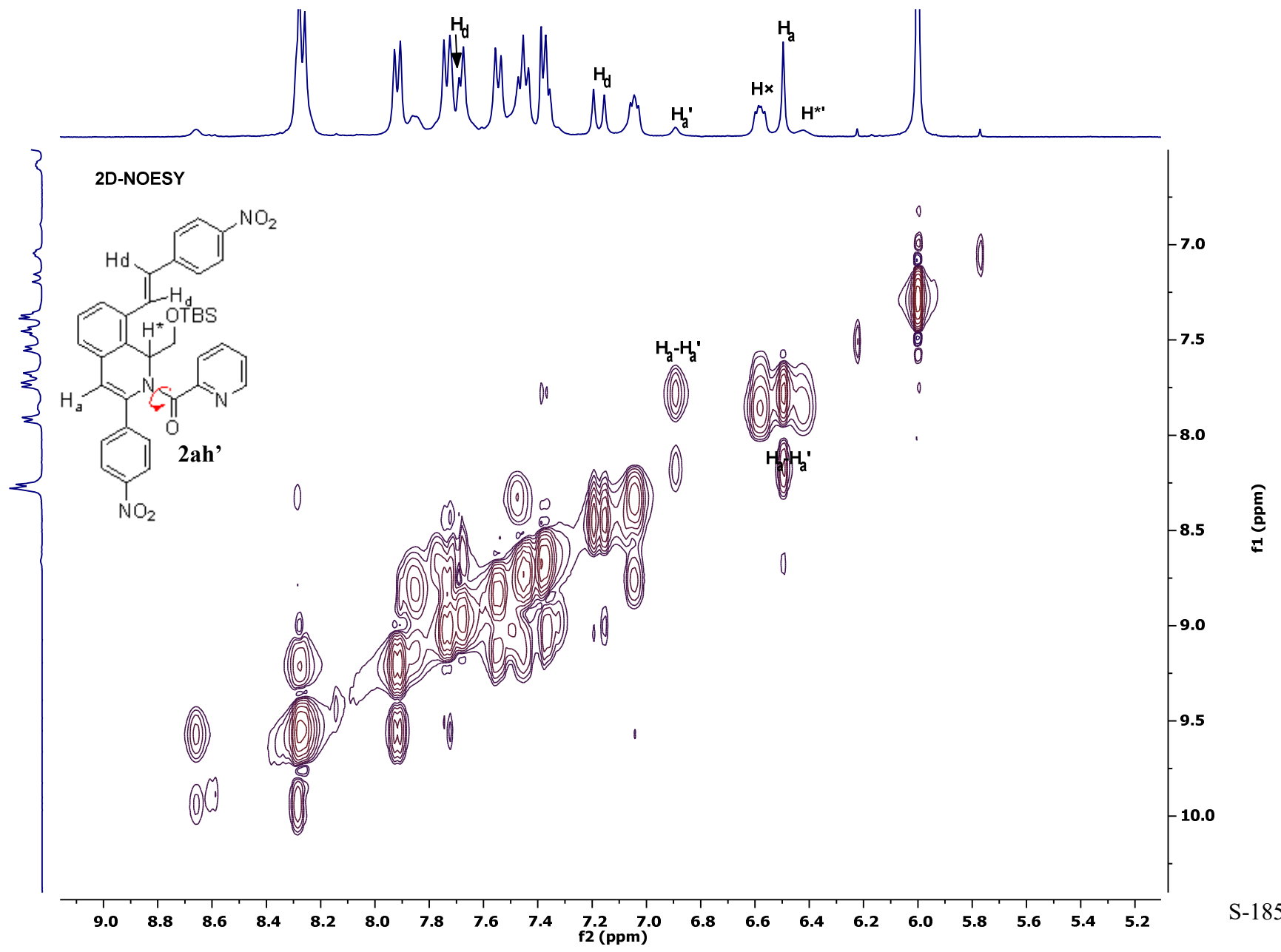


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

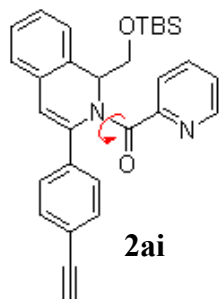




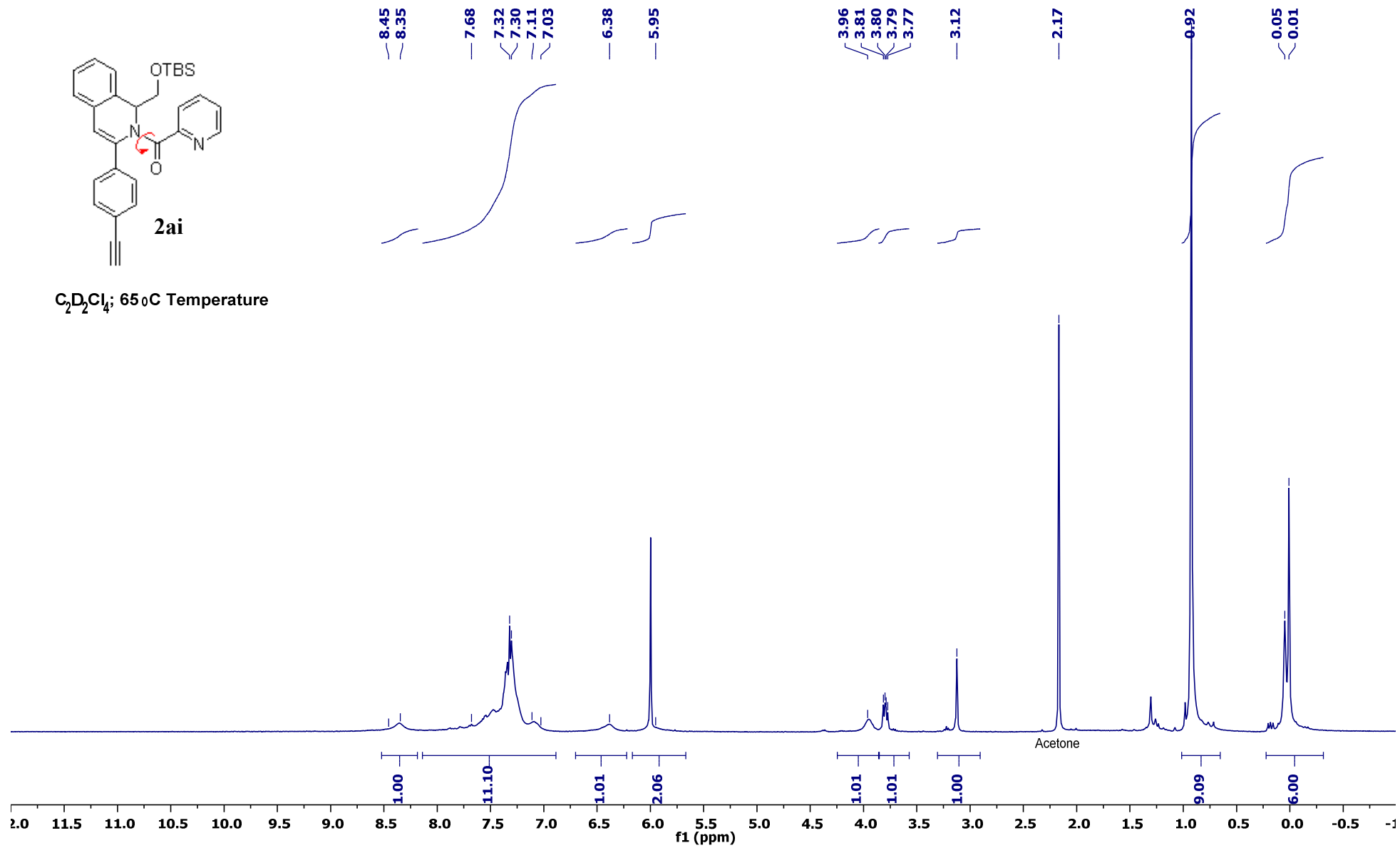




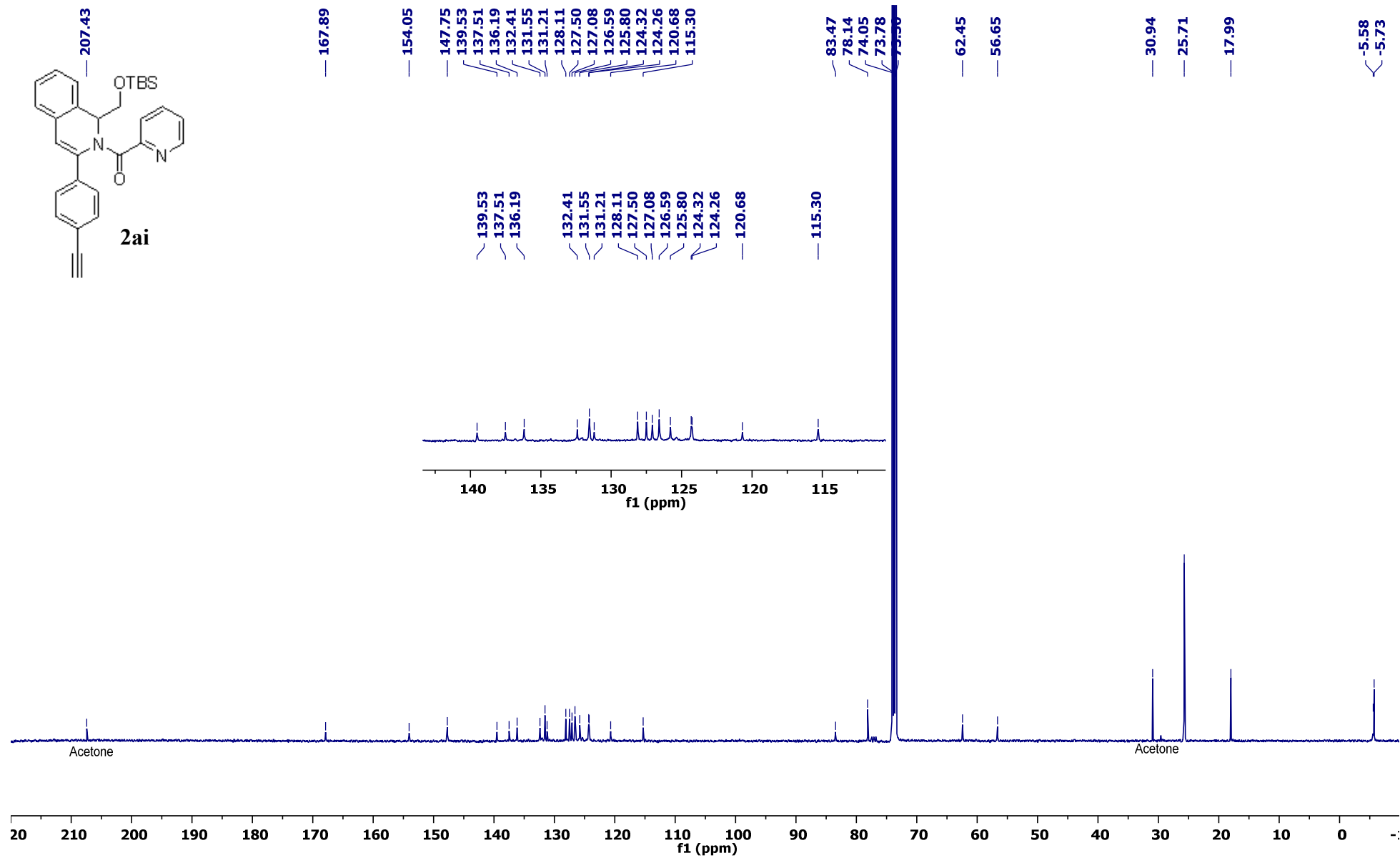
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



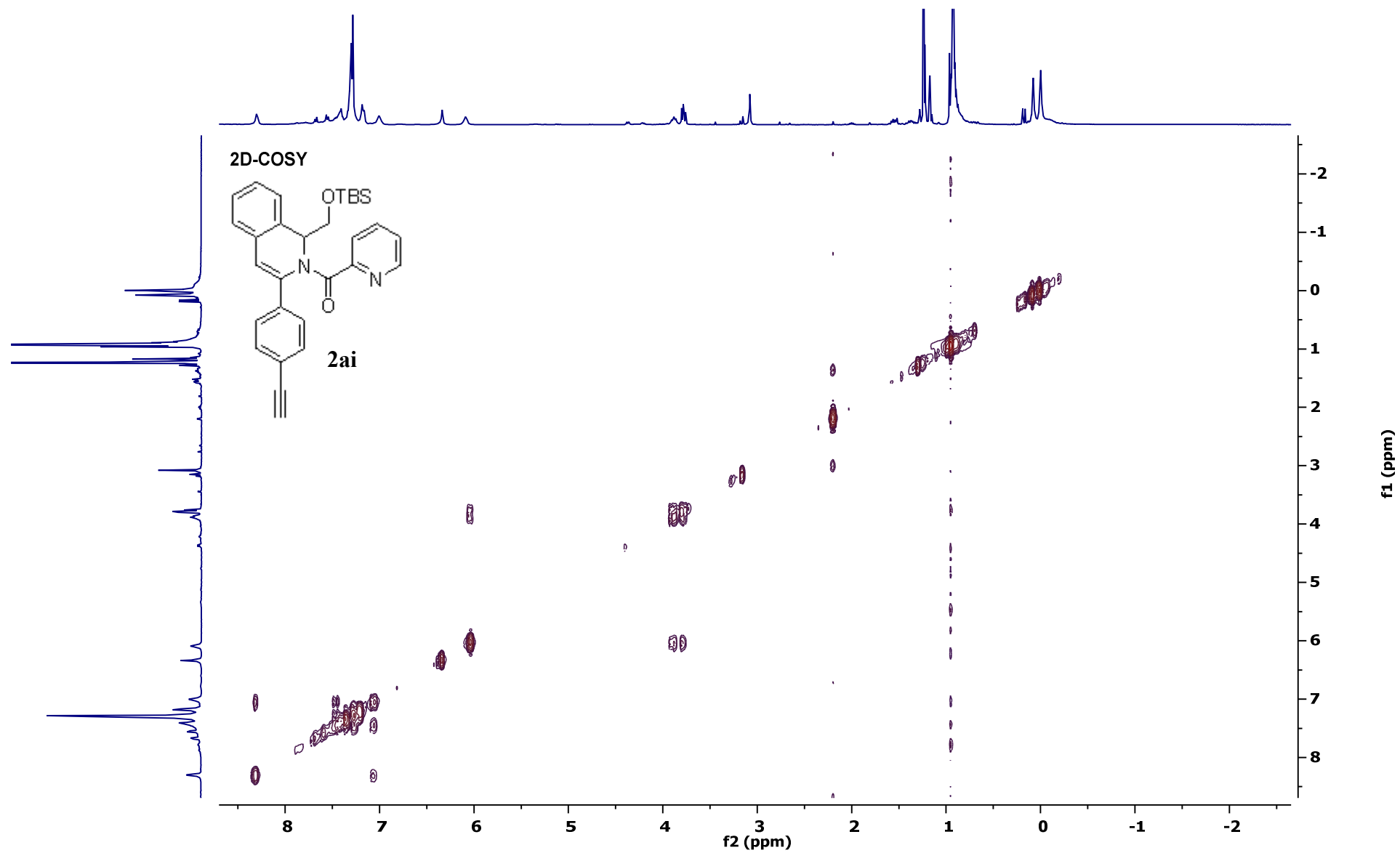
$\text{C}_2\text{D}_2\text{Cl}_4$ ; 65 °C Temperature



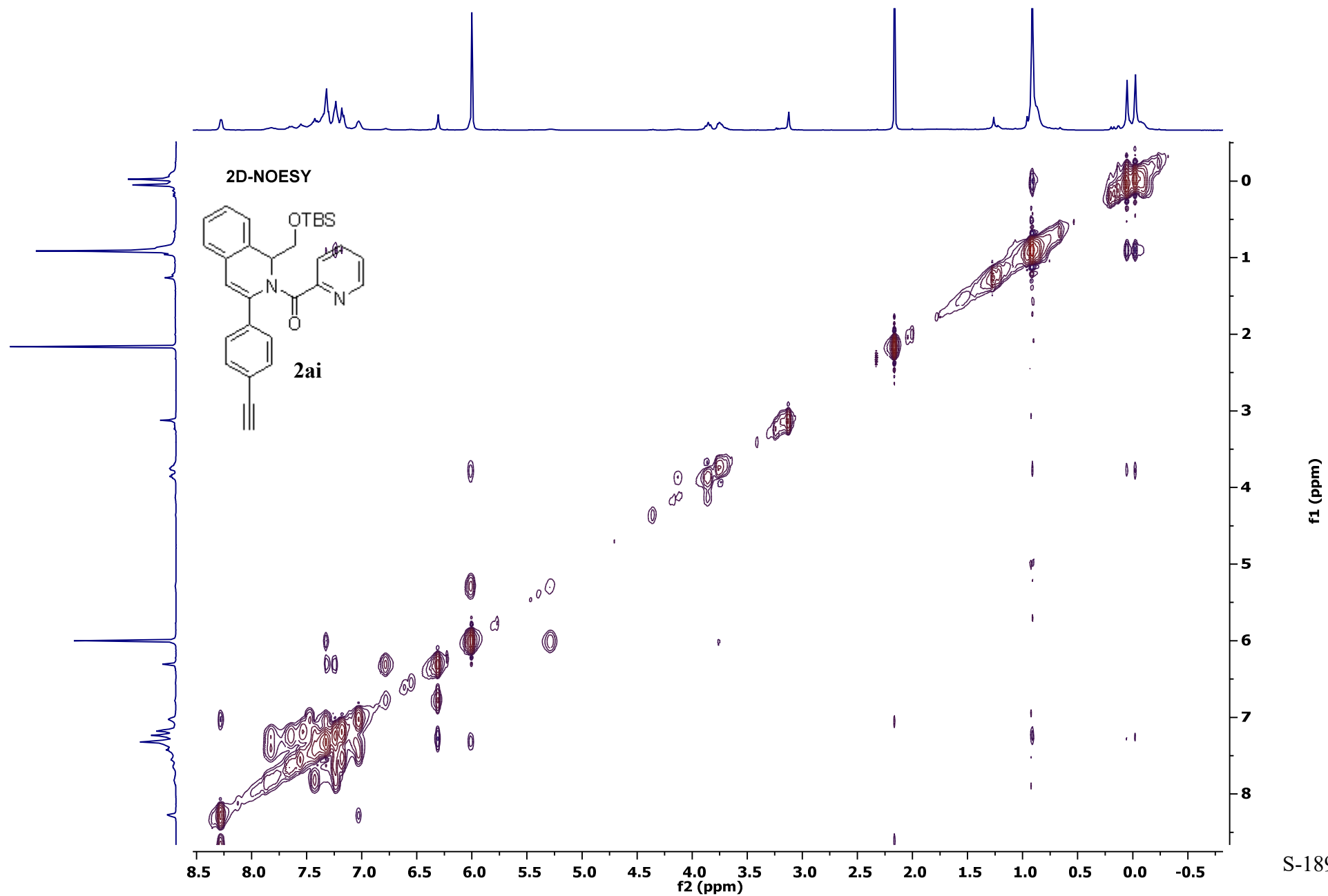
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

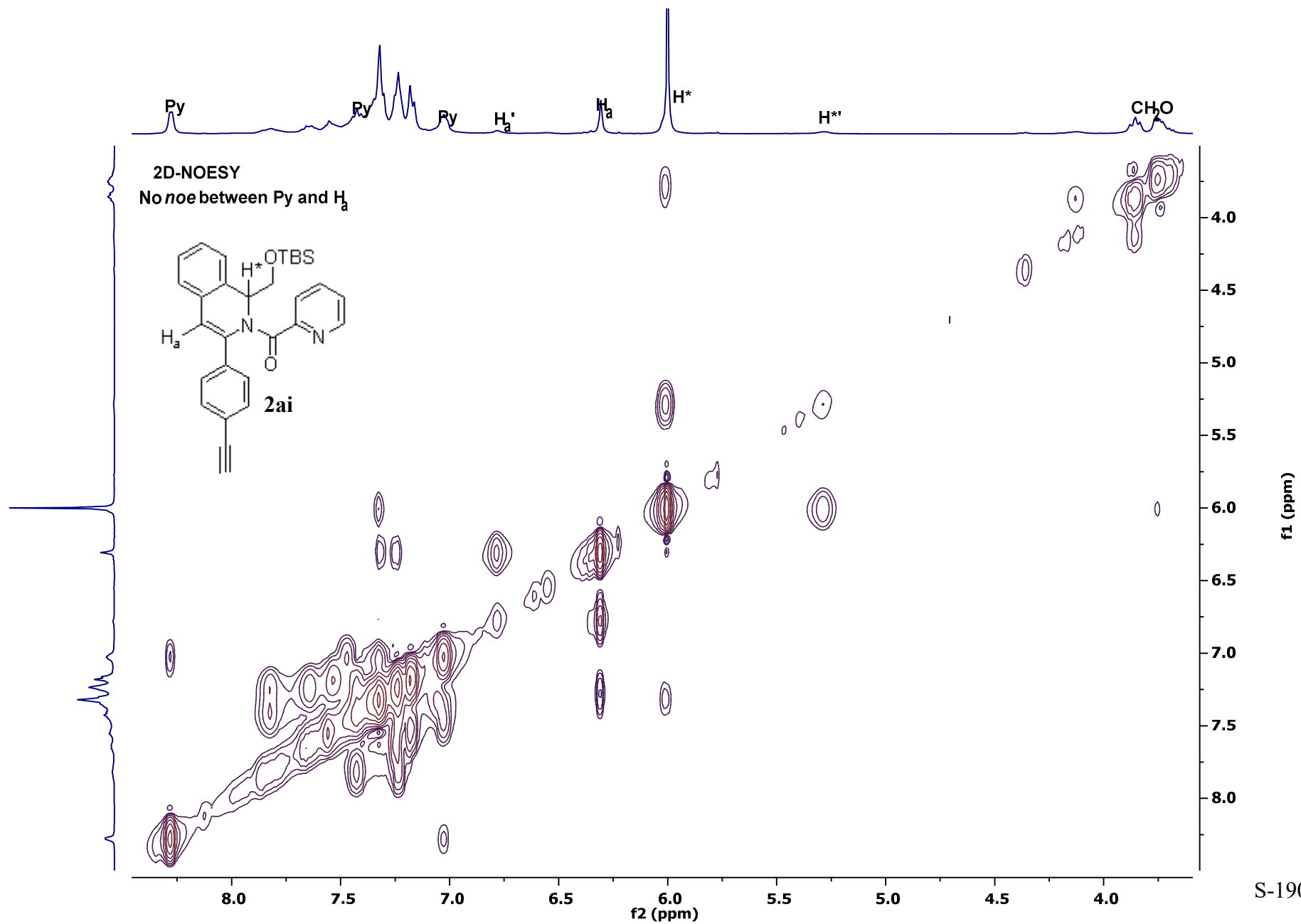


2D (COSY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

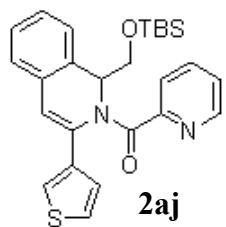


2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

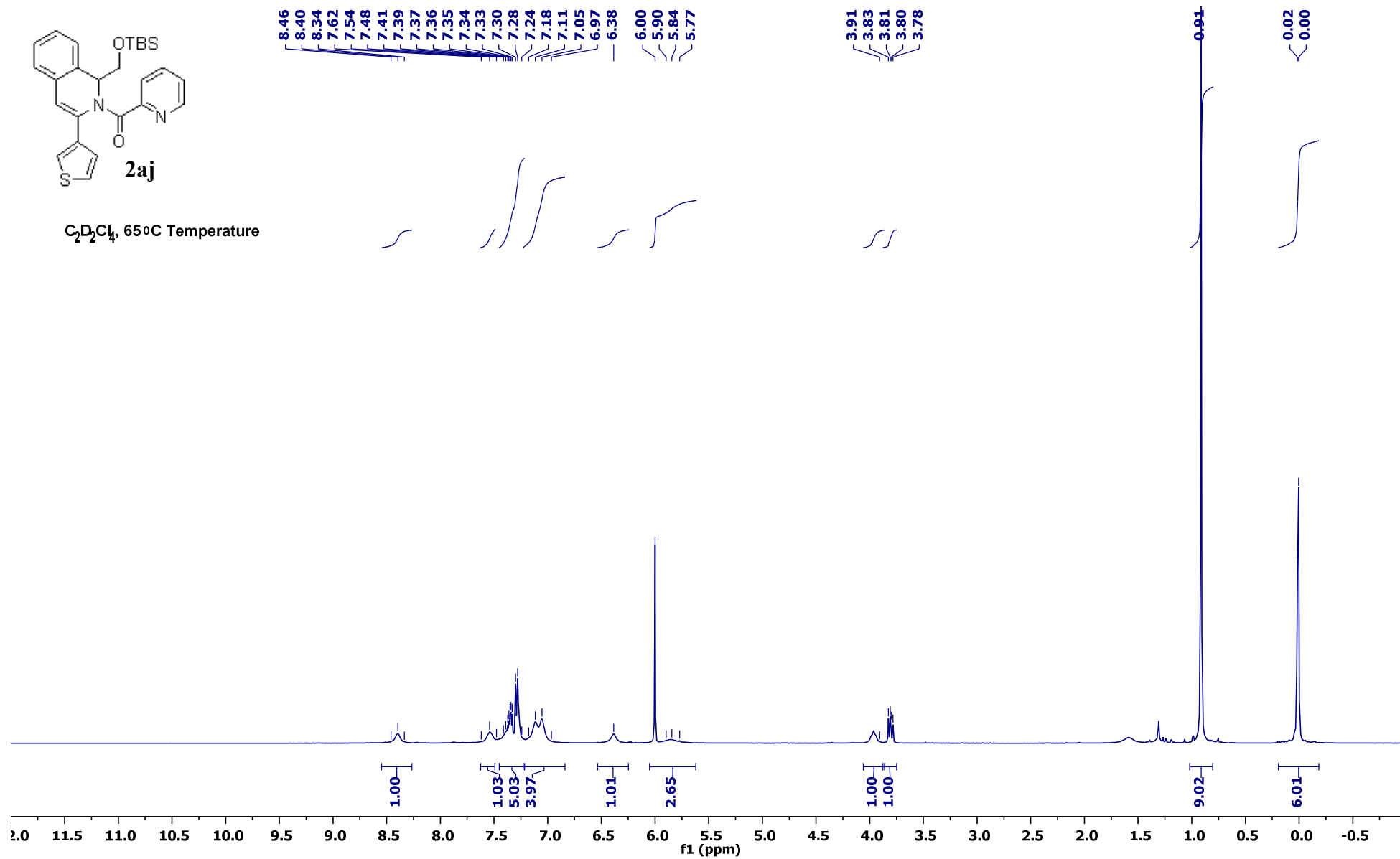




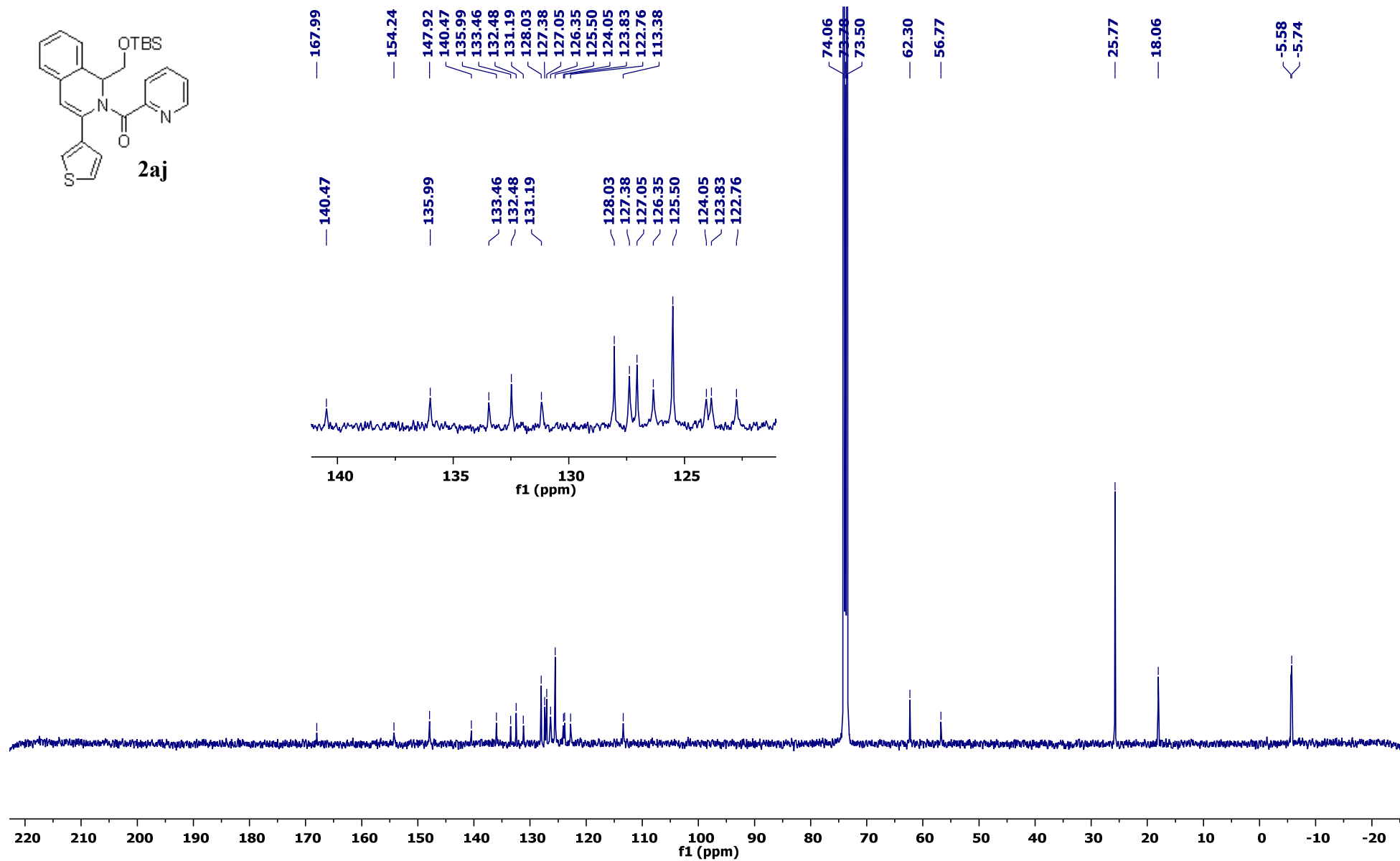
$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



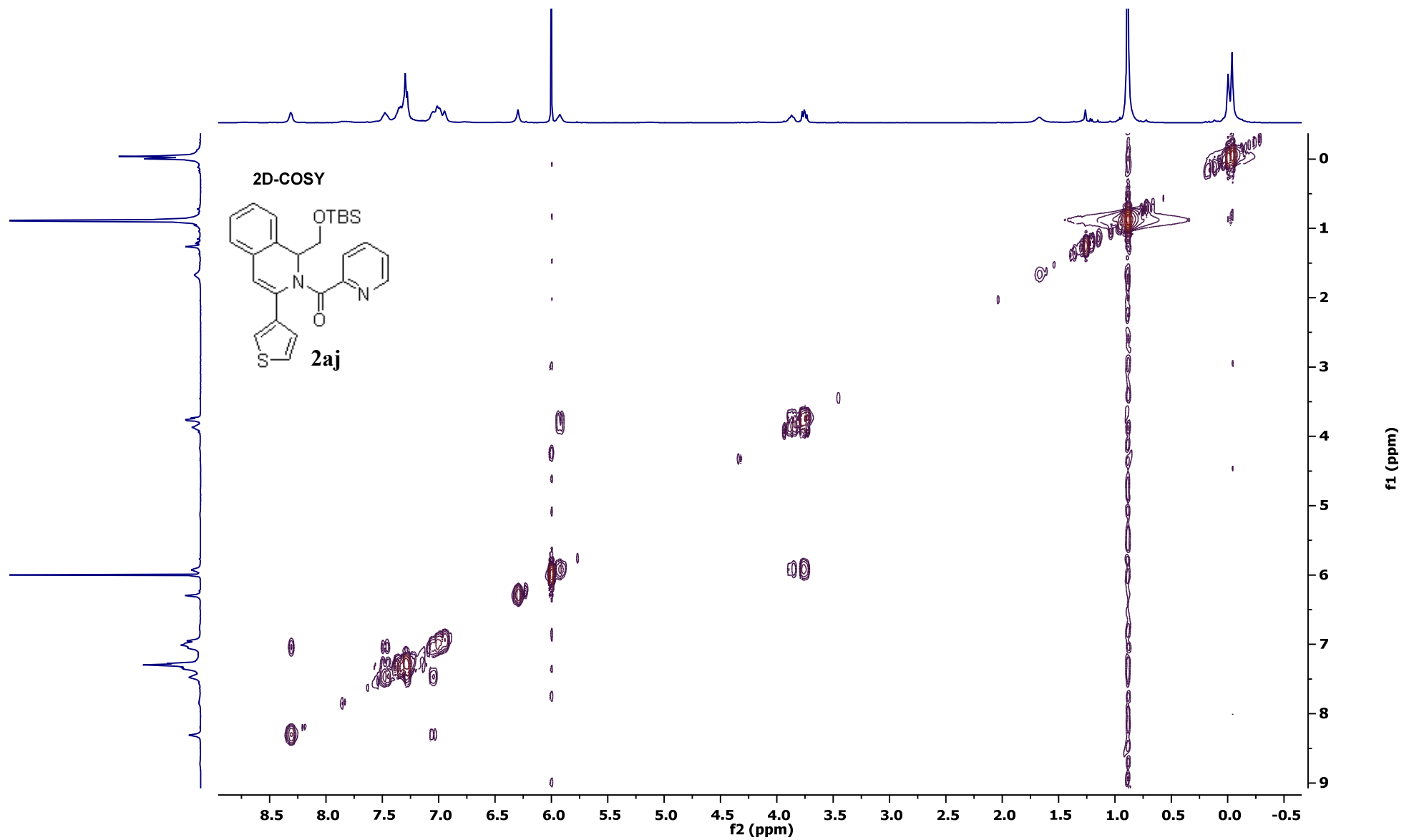
$\text{C}_2\text{D}_2\text{Cl}_4$ , 65°C Temperature



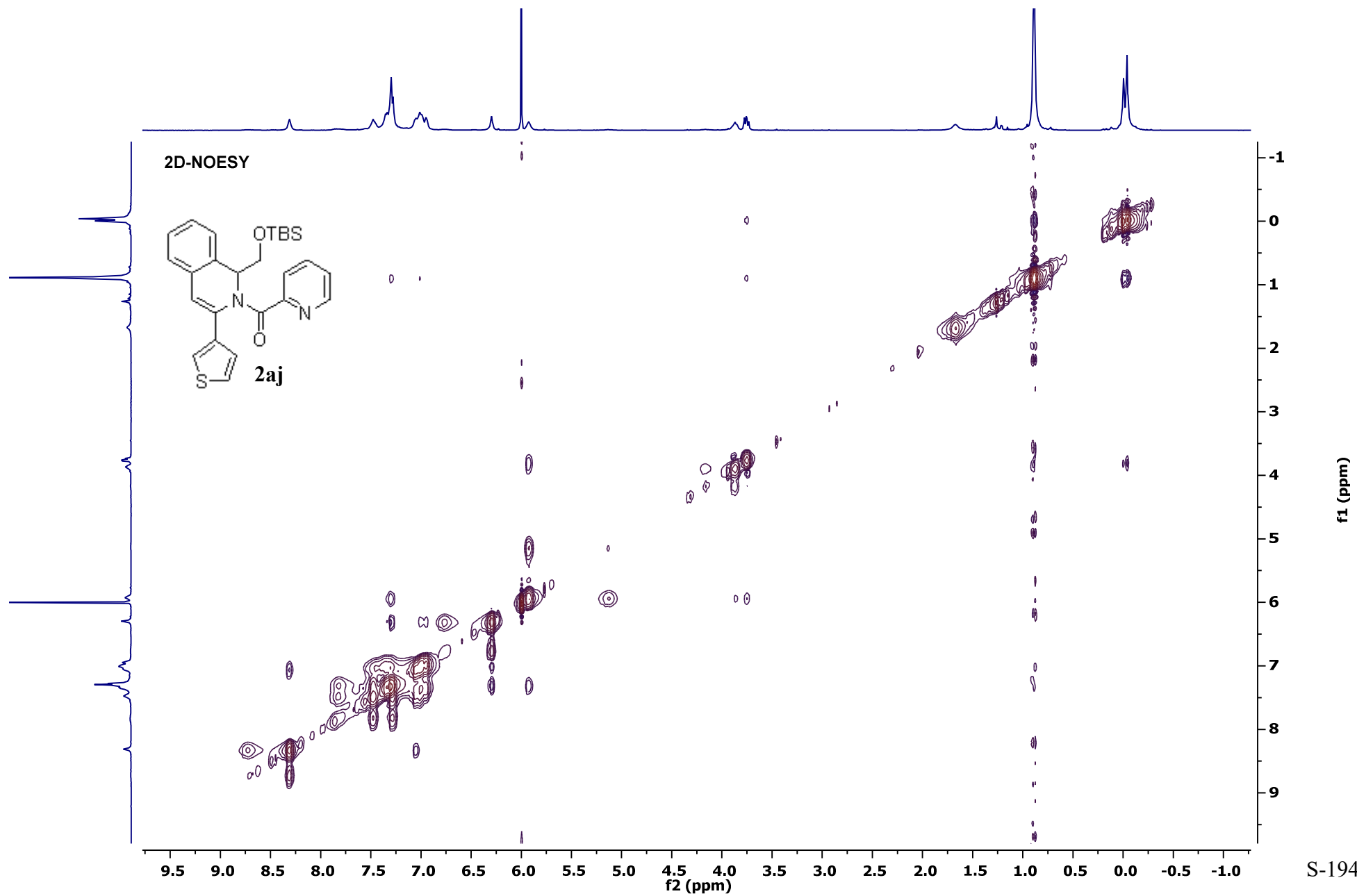
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

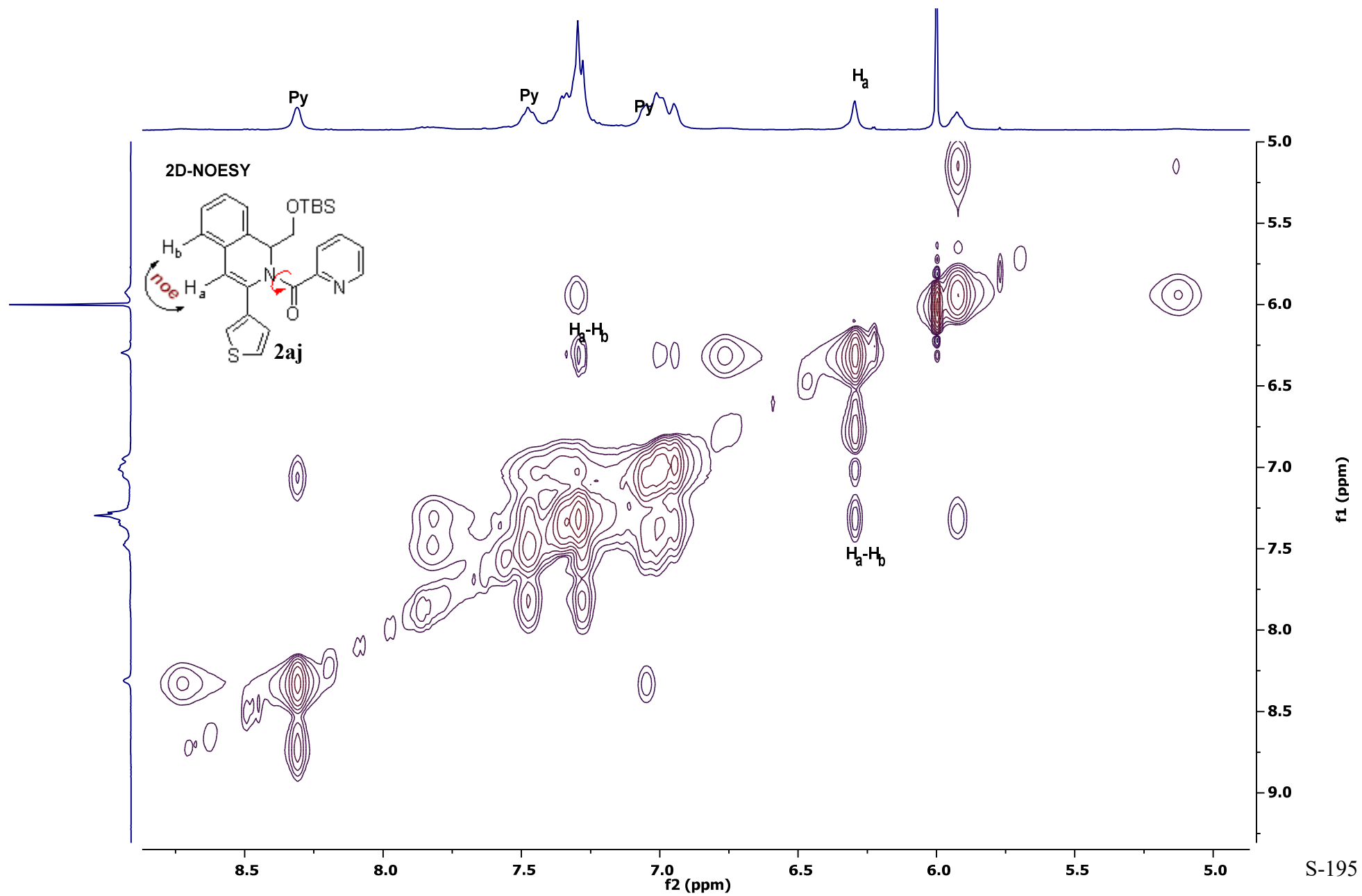


2D (COSY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)



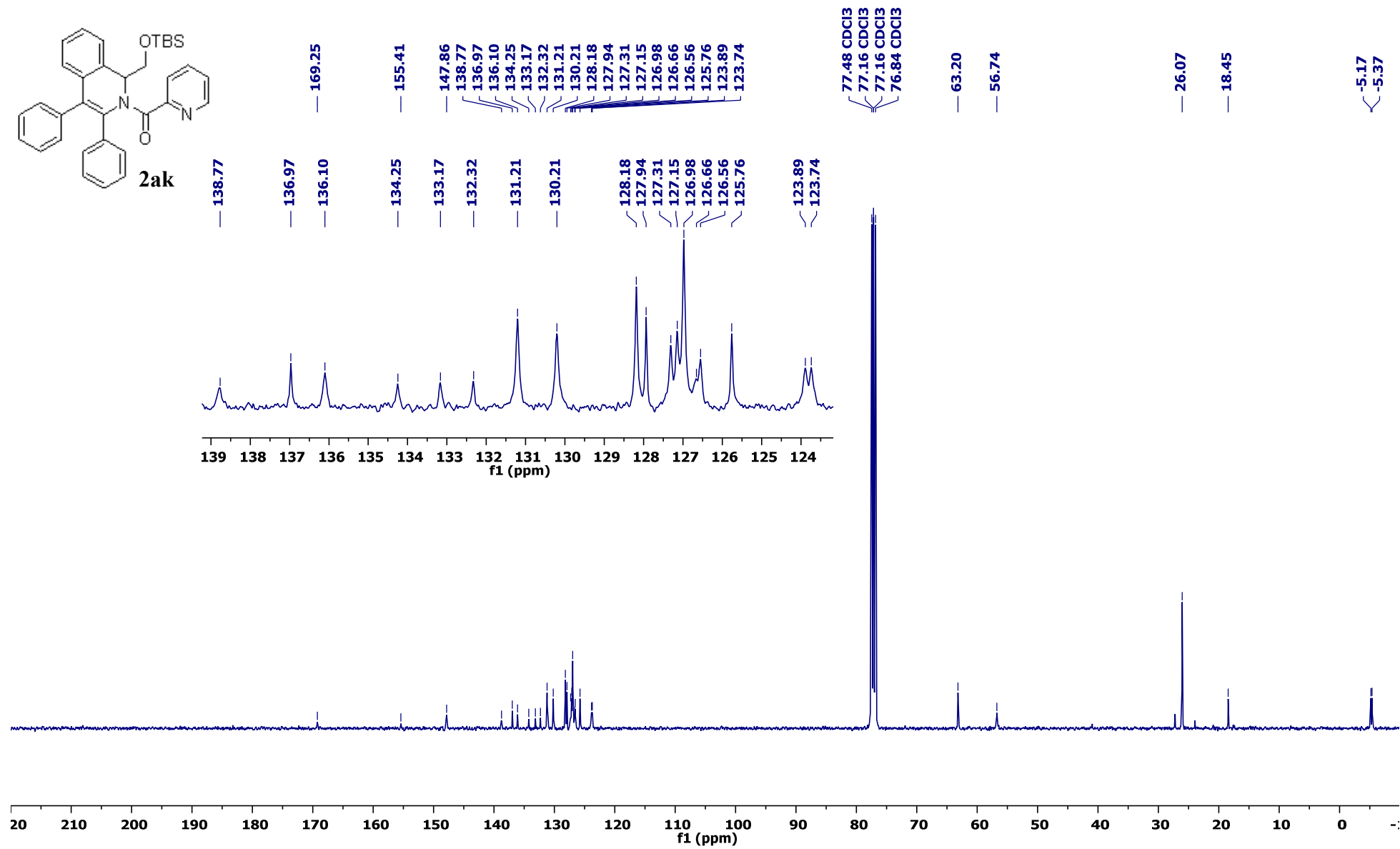
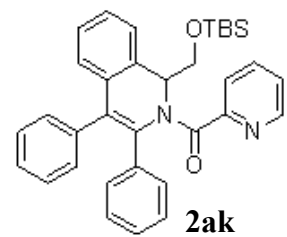
2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

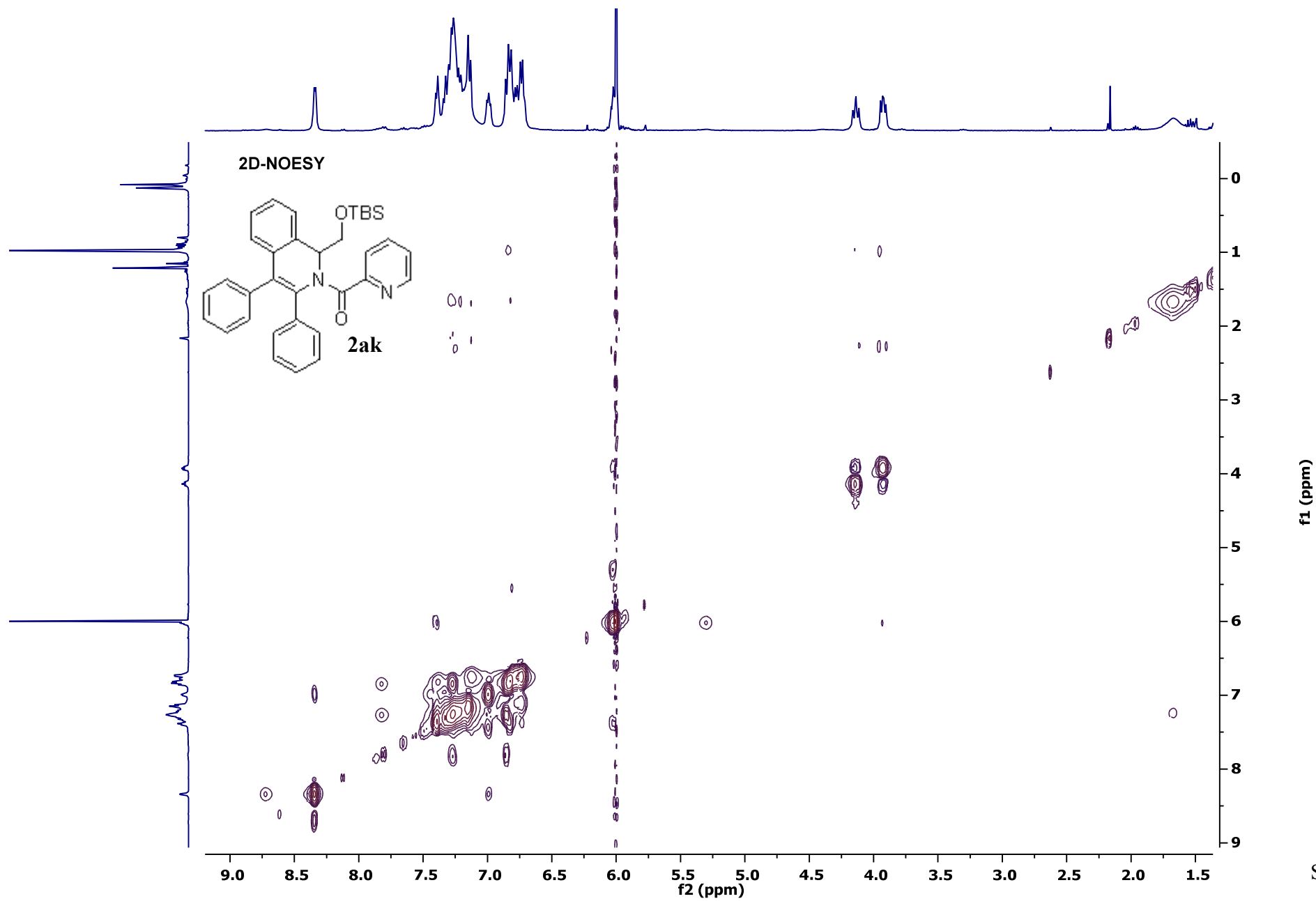




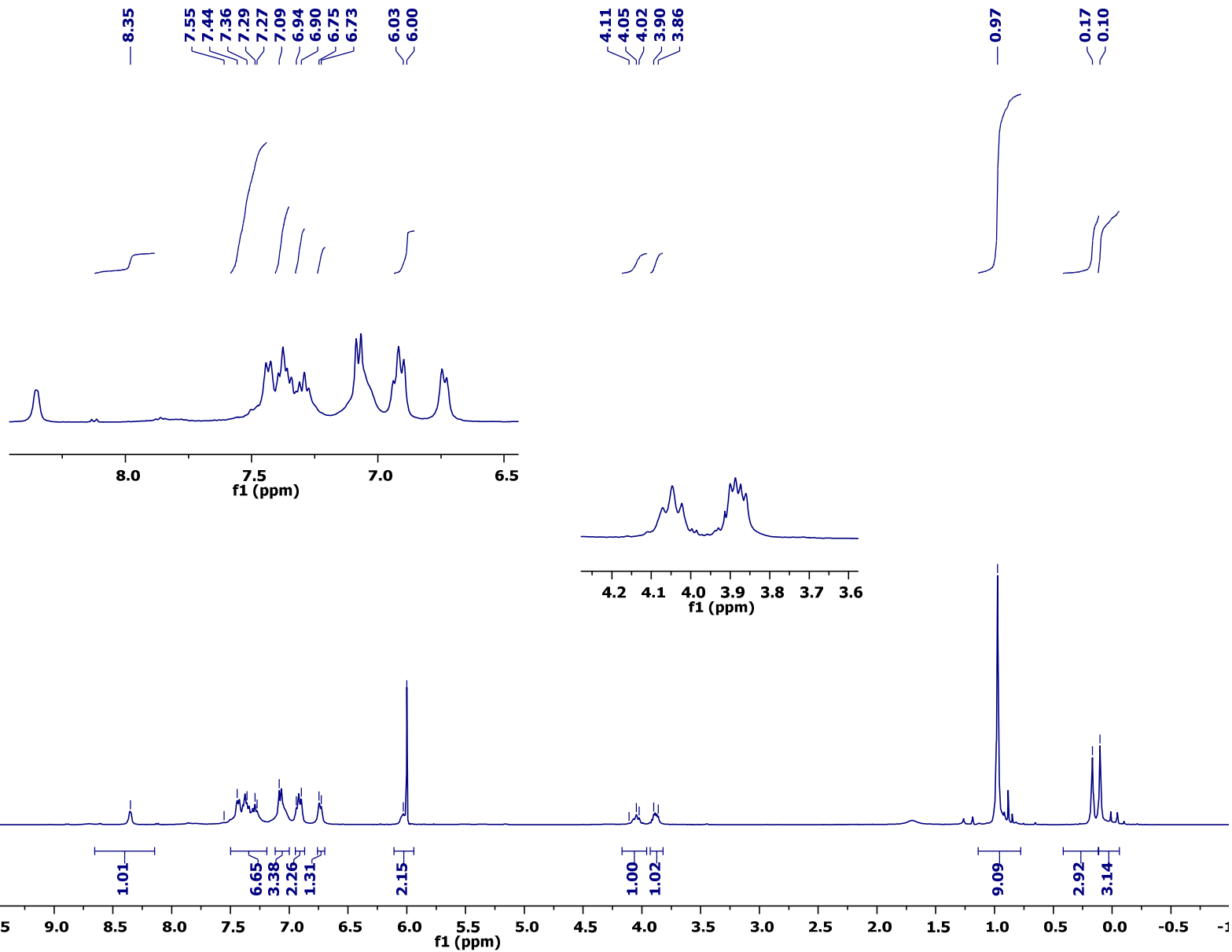
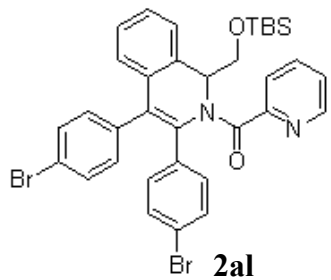


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

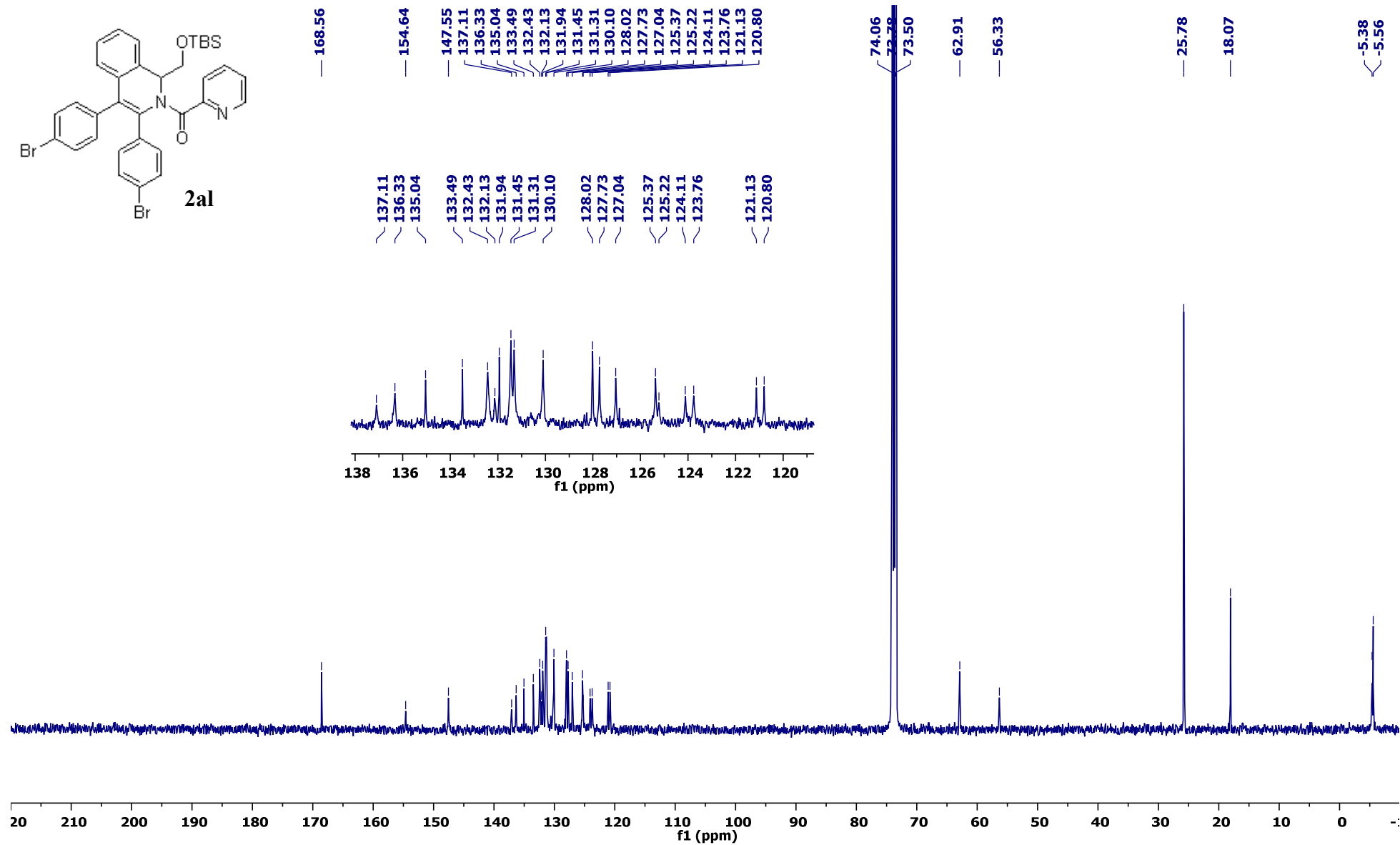
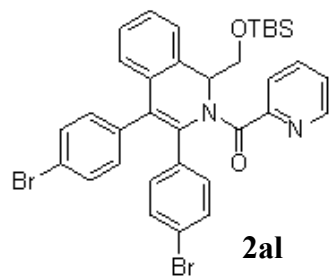




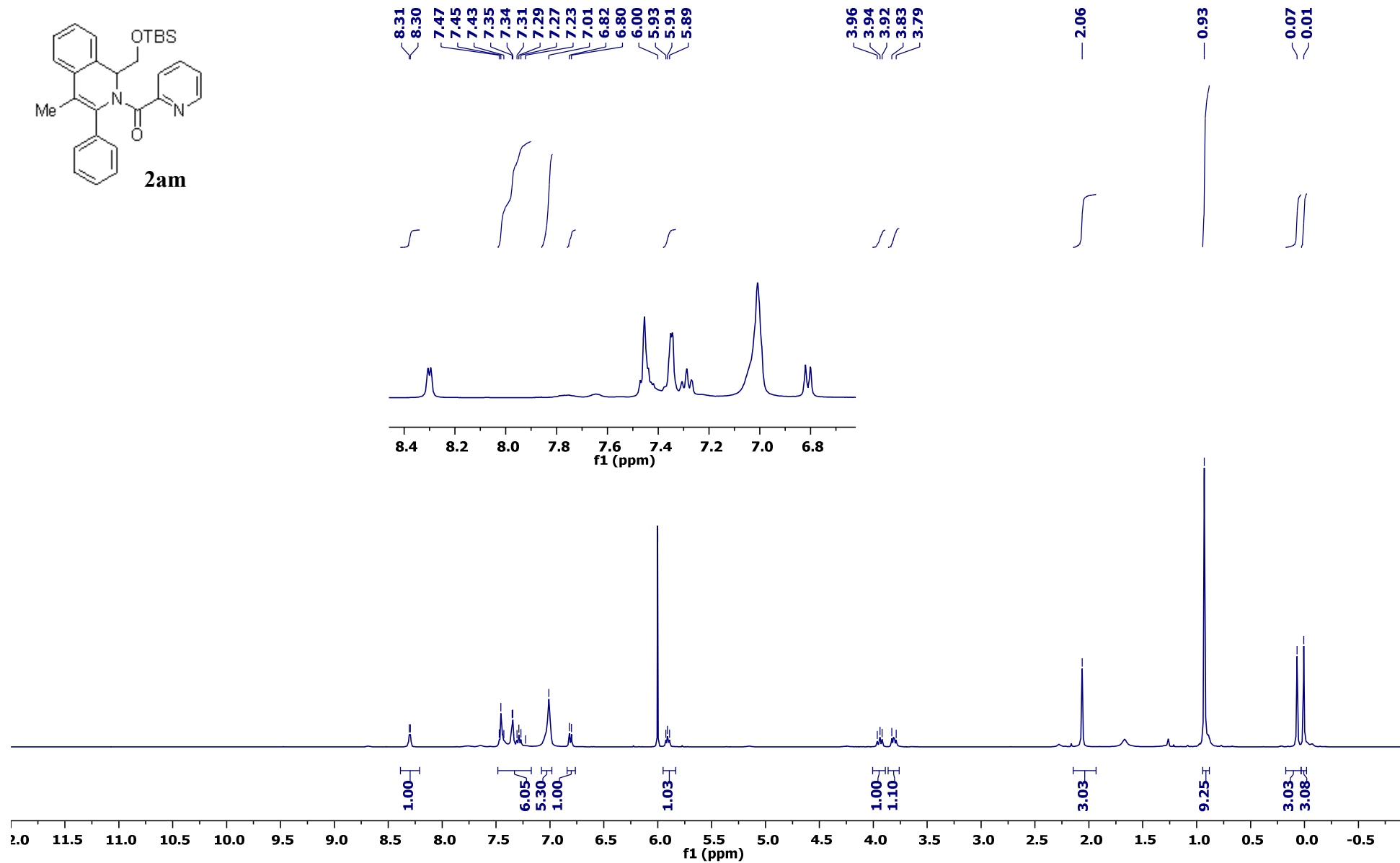
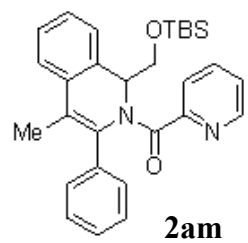
<sup>1</sup>H NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)



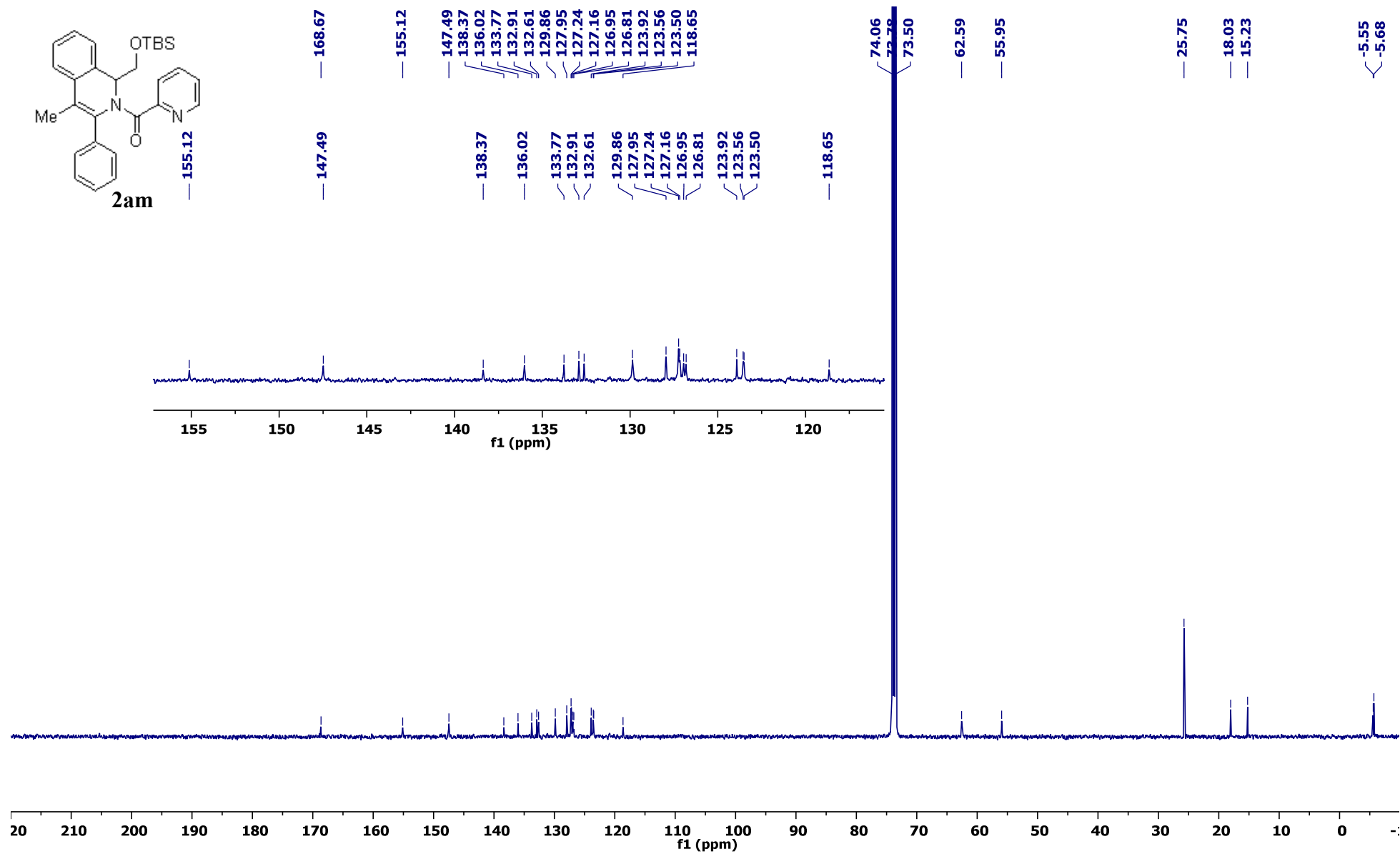
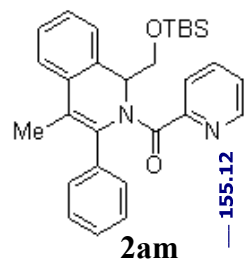
$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )



$^1\text{H}$  NMR (400 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

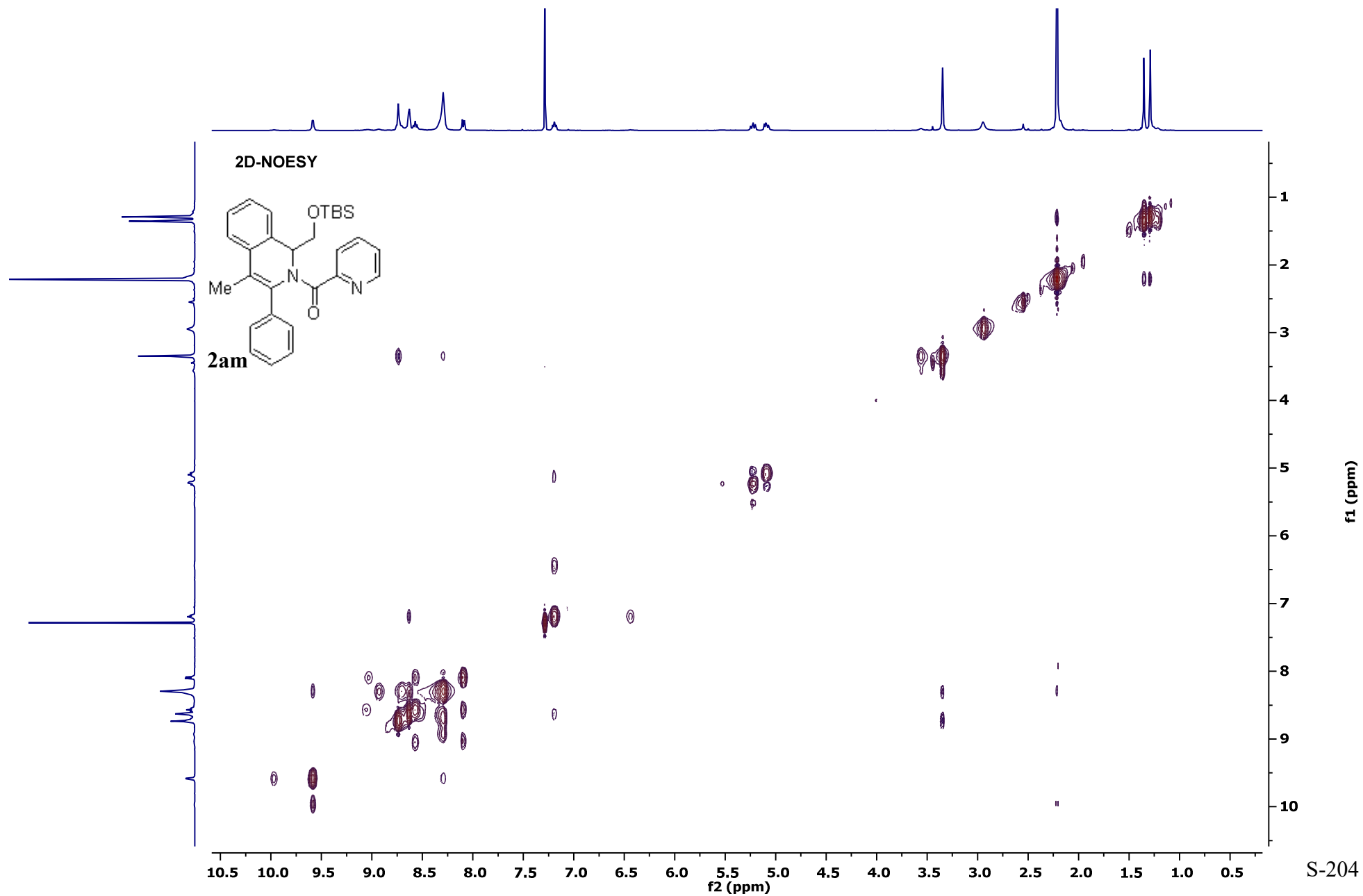


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{C}_2\text{D}_2\text{Cl}_4$ )

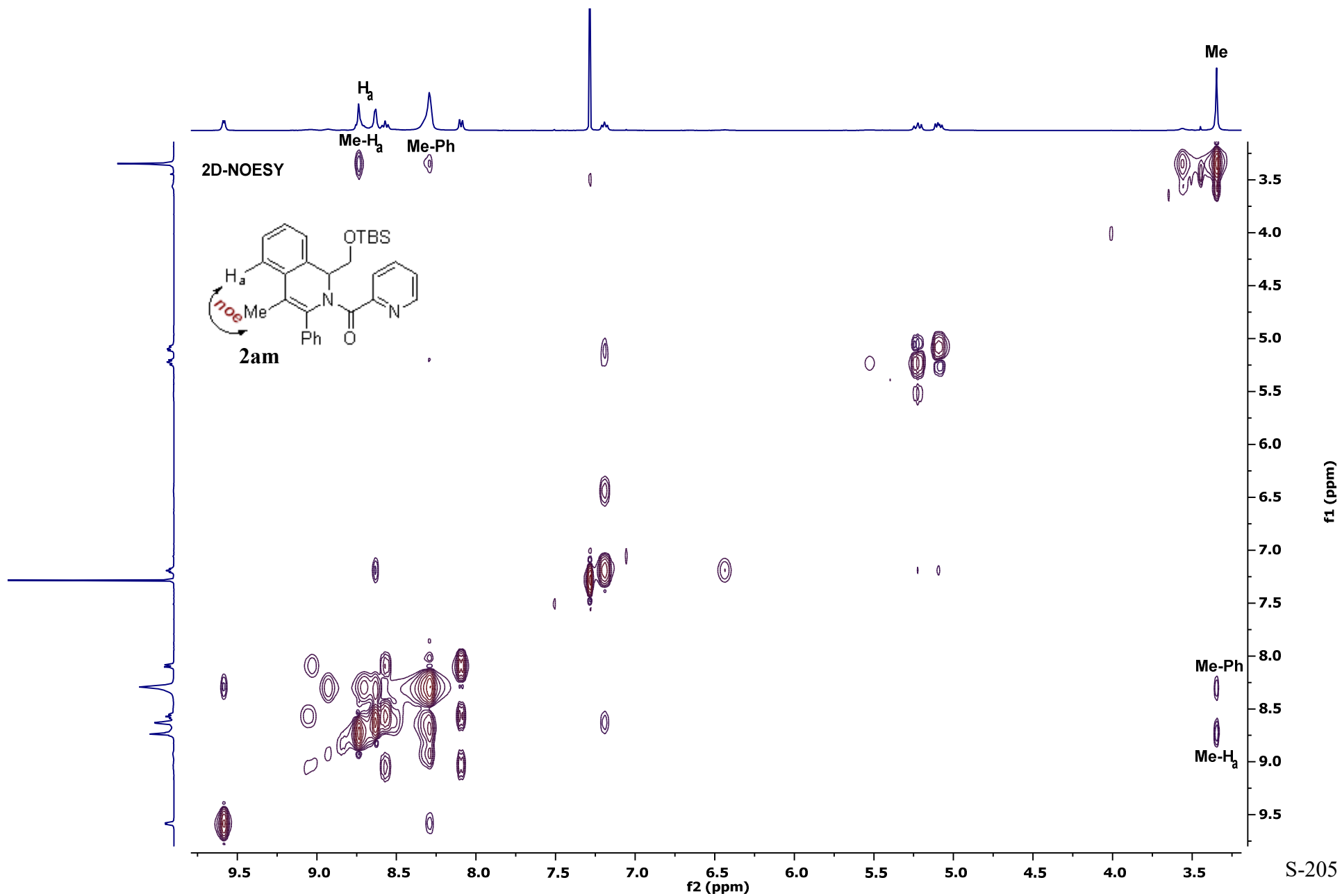




2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

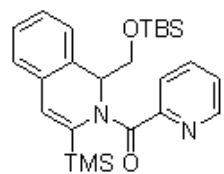


2D (NOESY) NMR (400 MHz, C<sub>2</sub>D<sub>2</sub>Cl<sub>4</sub>)

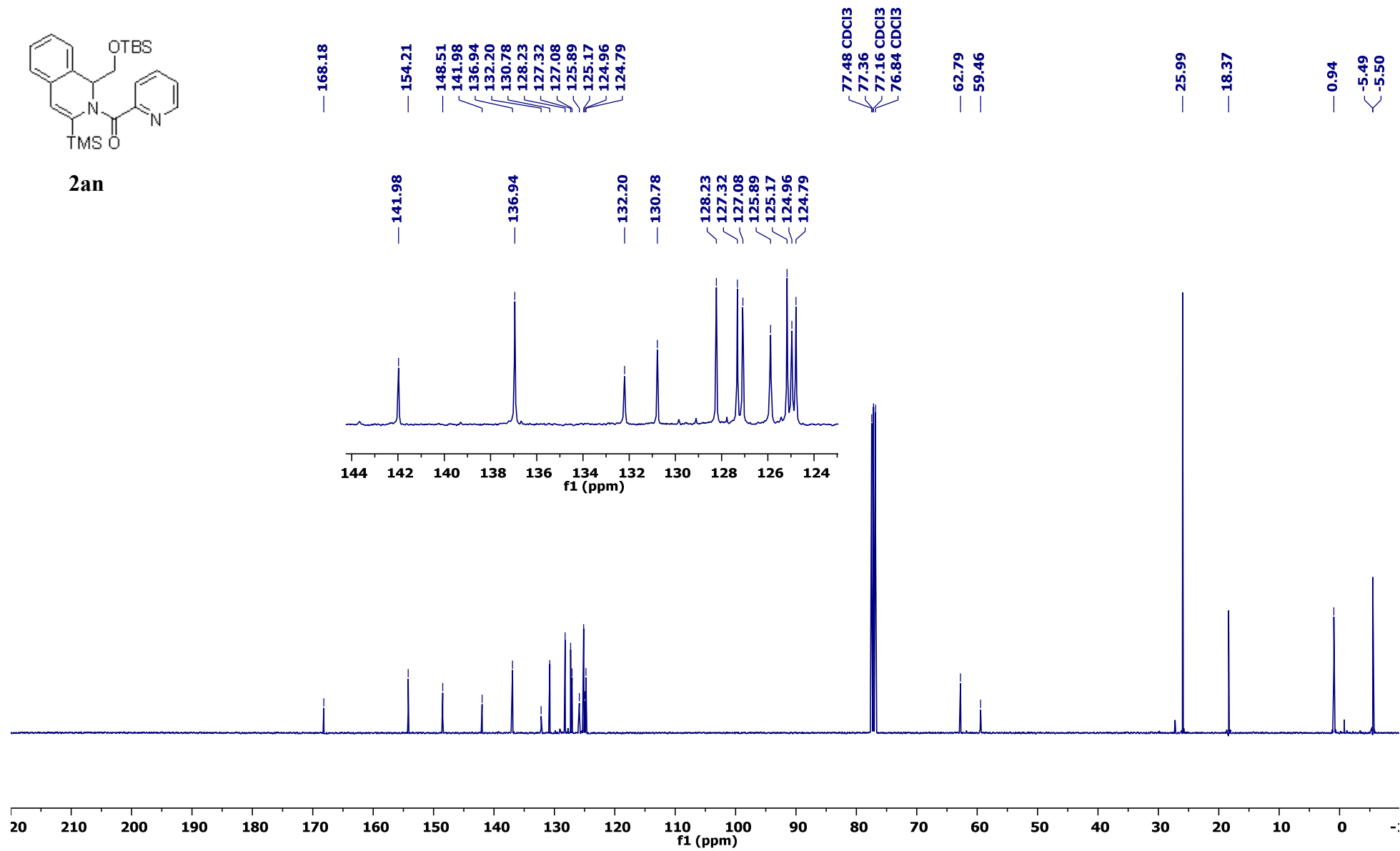




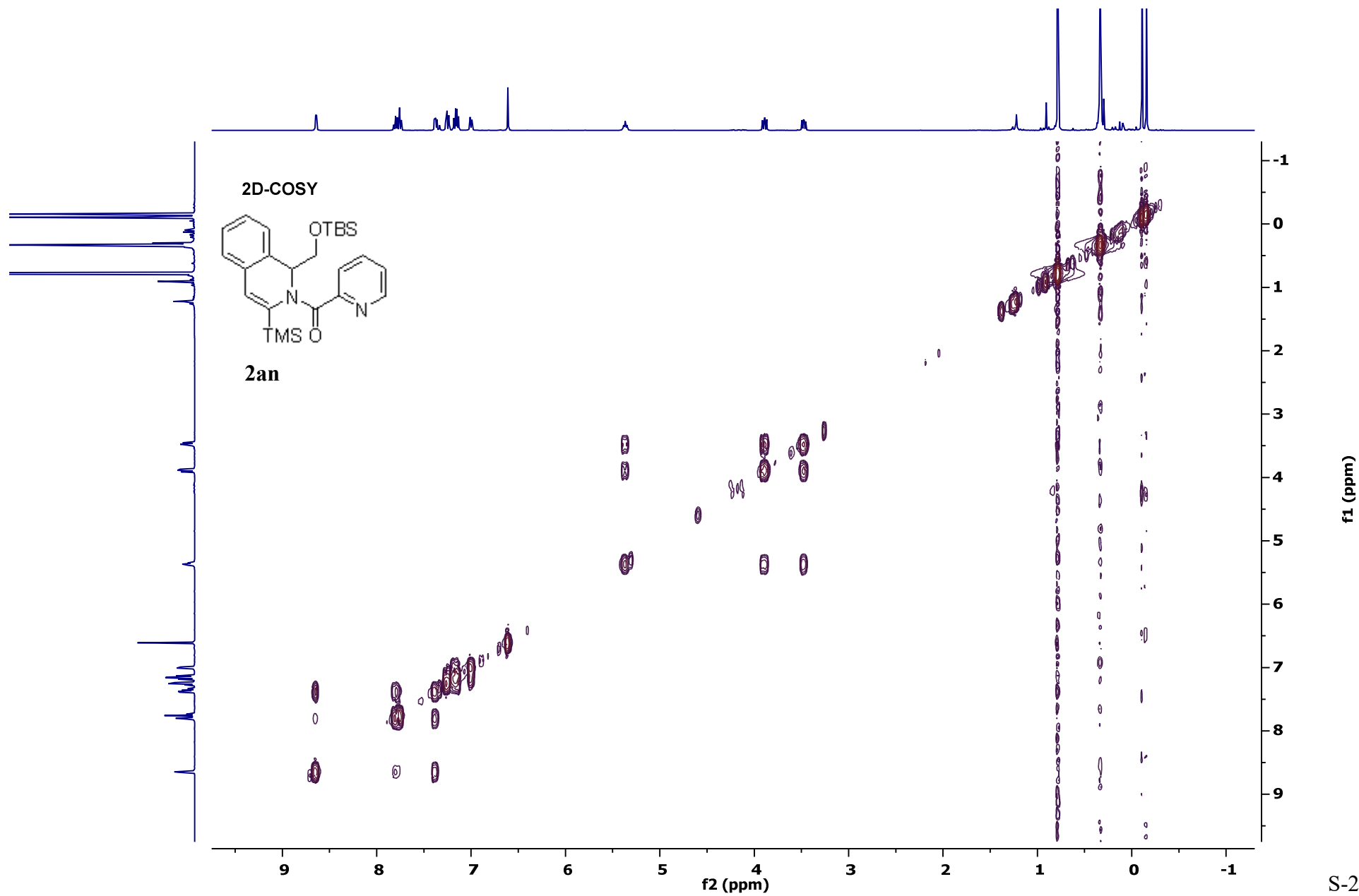
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



**2an**



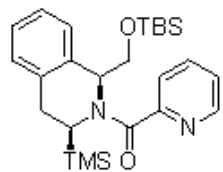
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



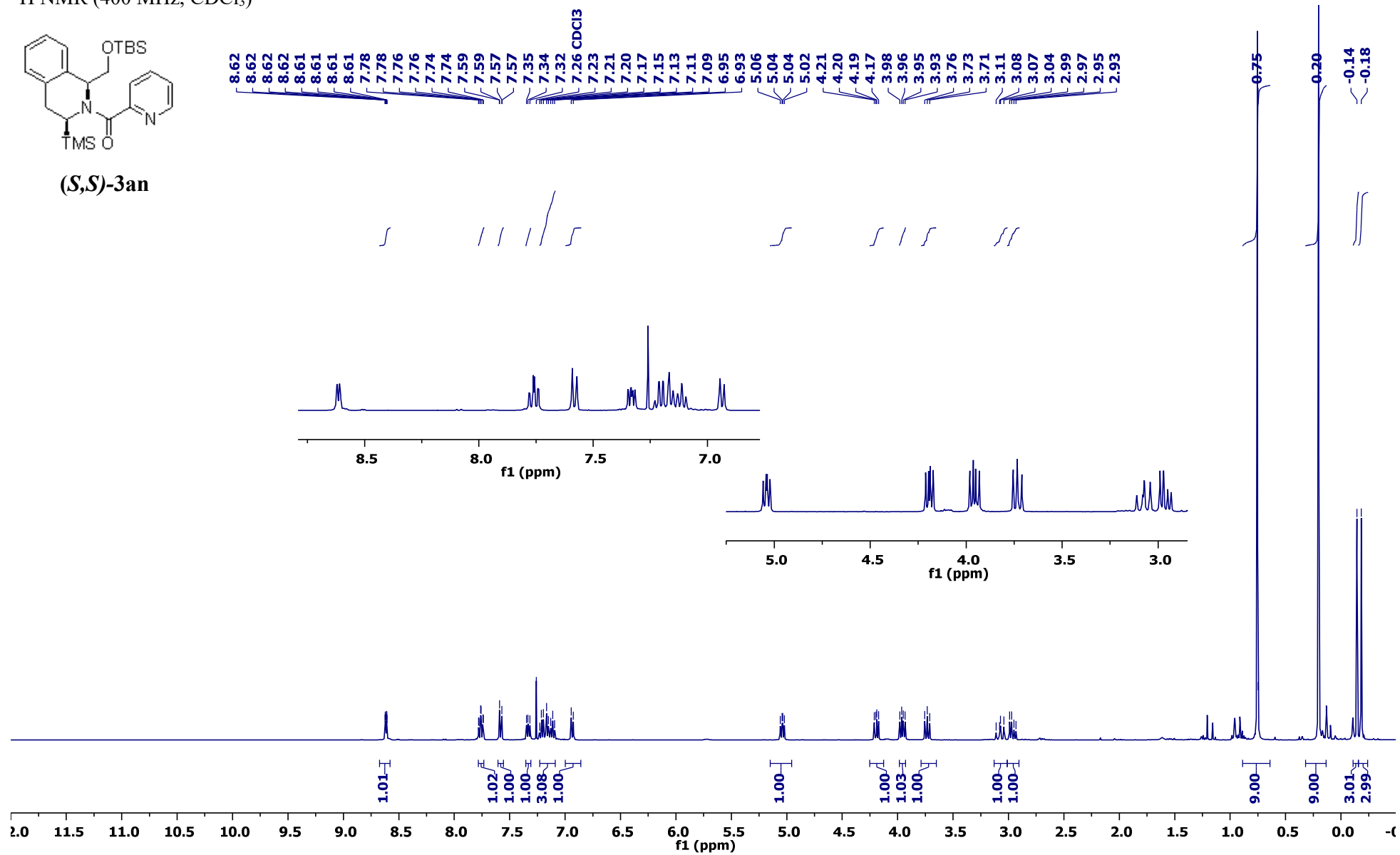




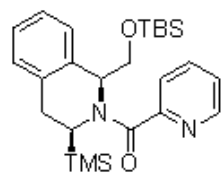
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



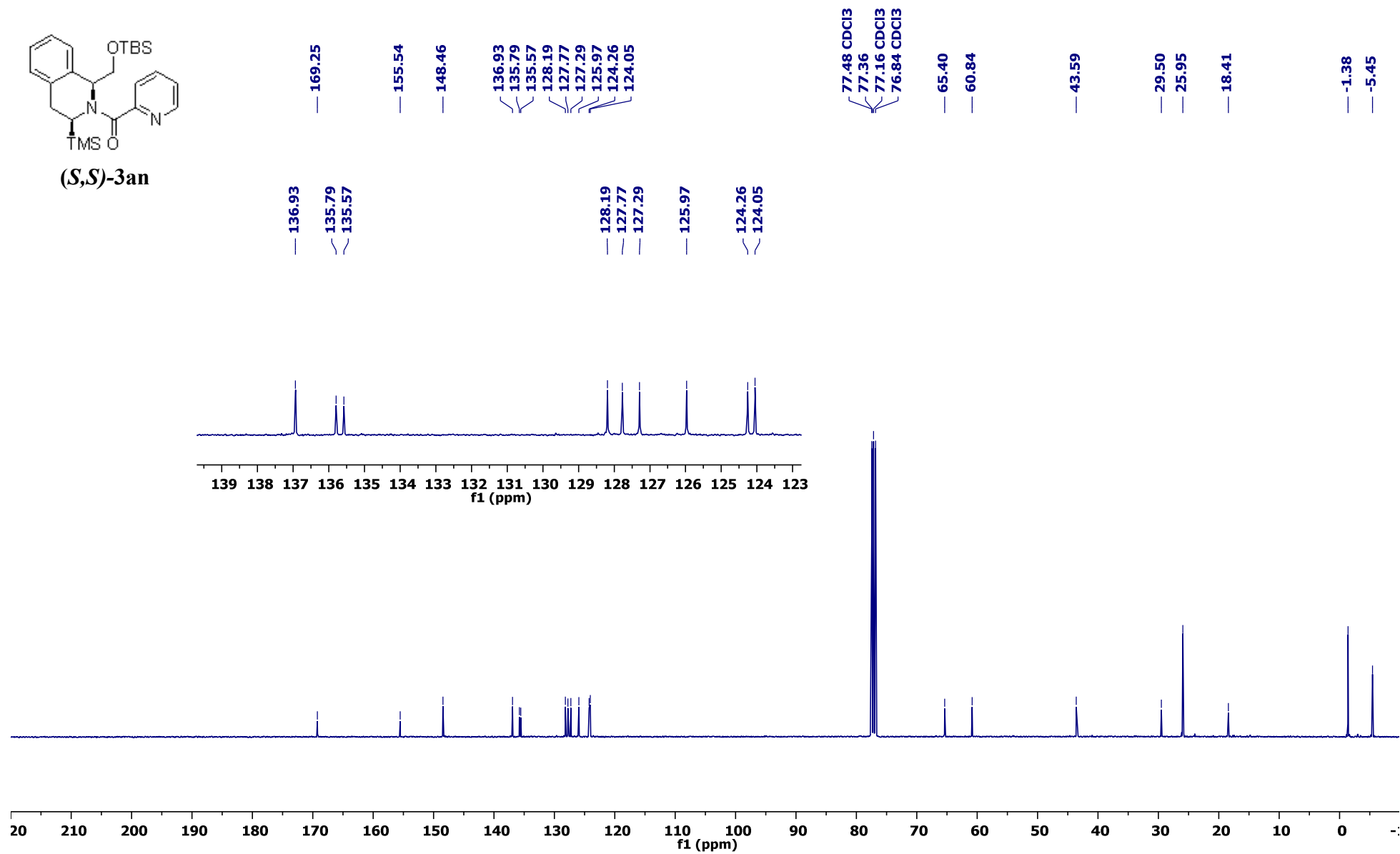
(*S,S*)-3an

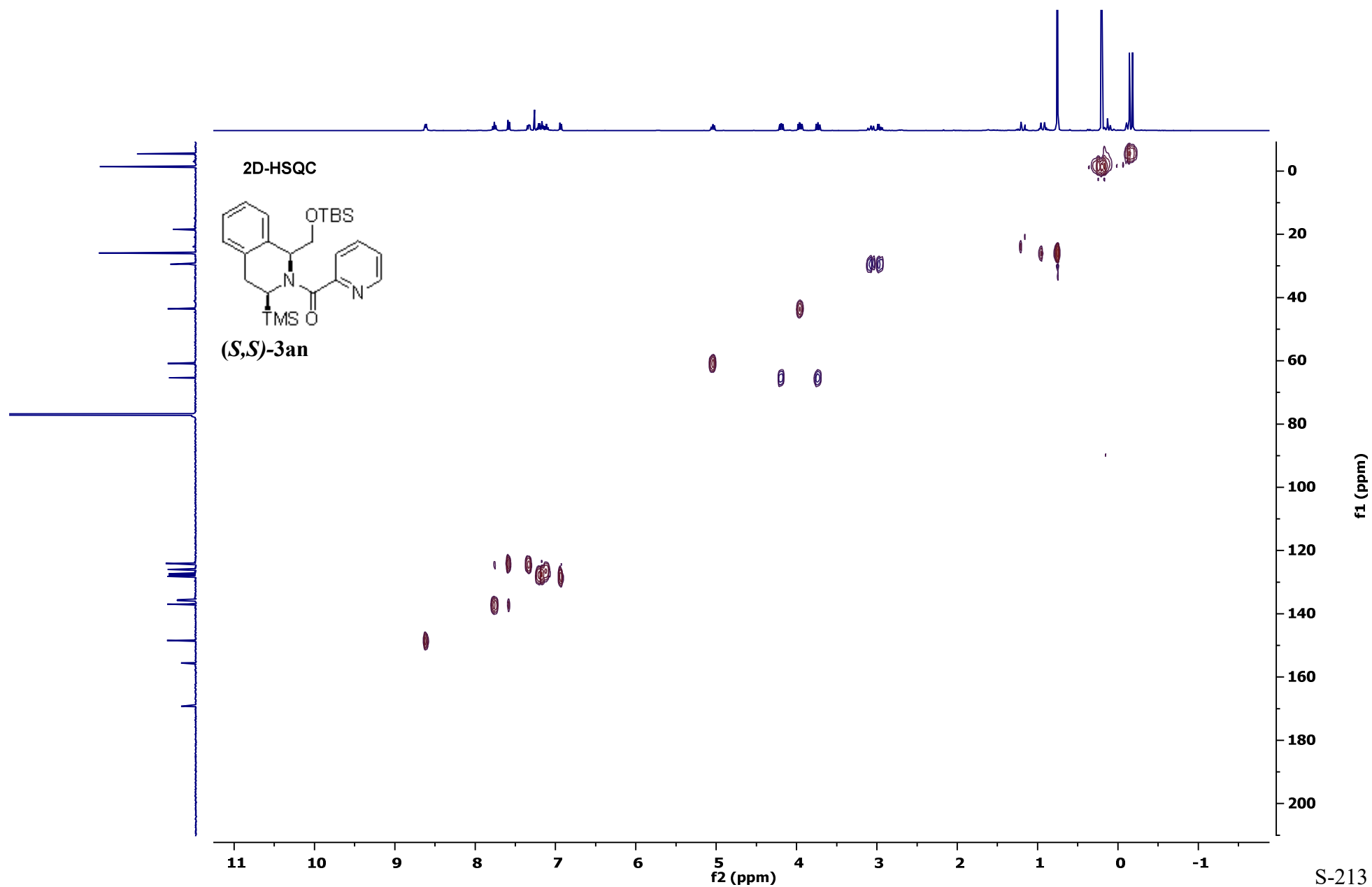


$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )

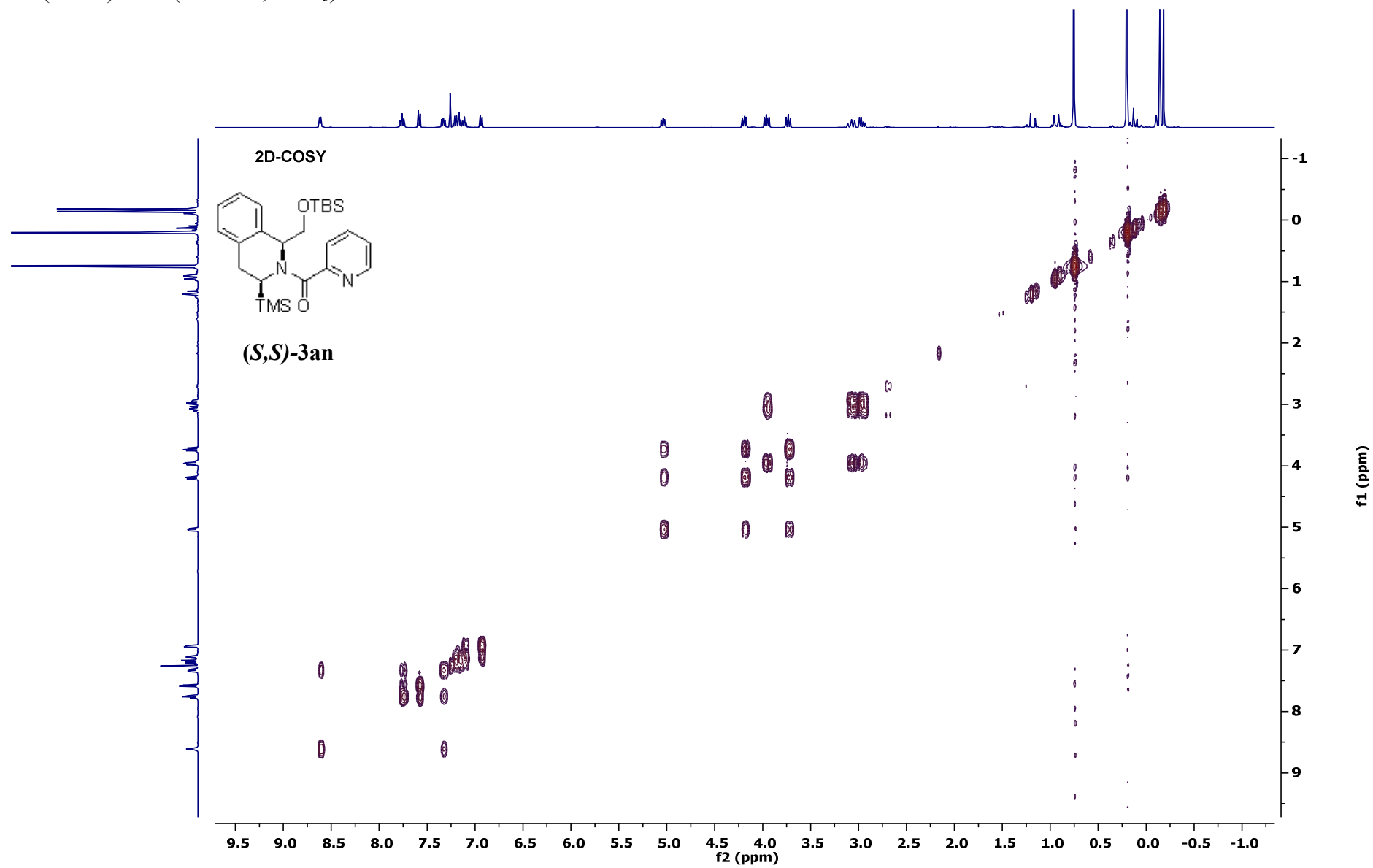


(*S,S*)-3an

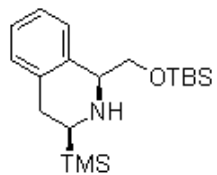




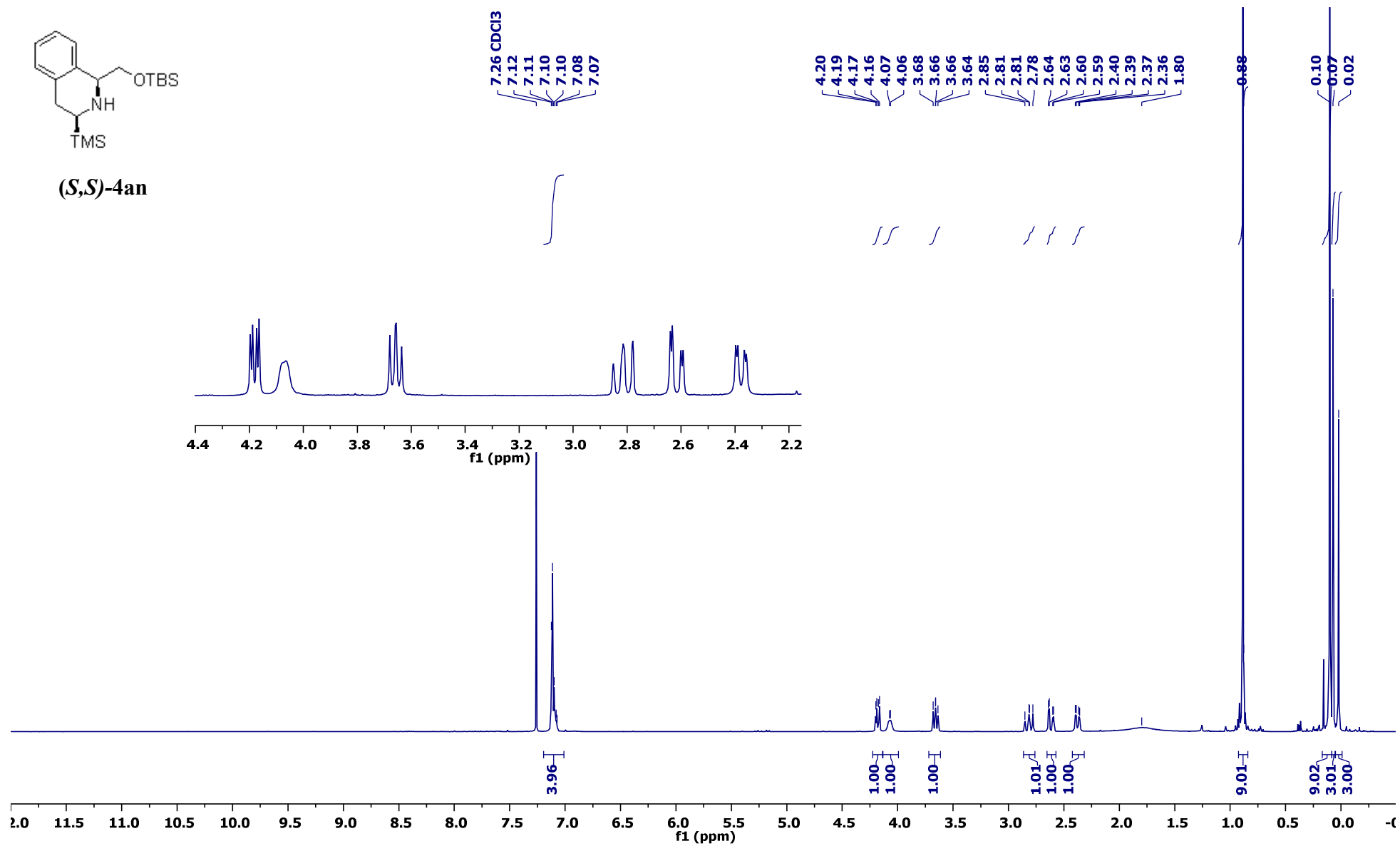
2D (COSY) NMR (400 MHz, CDCl<sub>3</sub>)



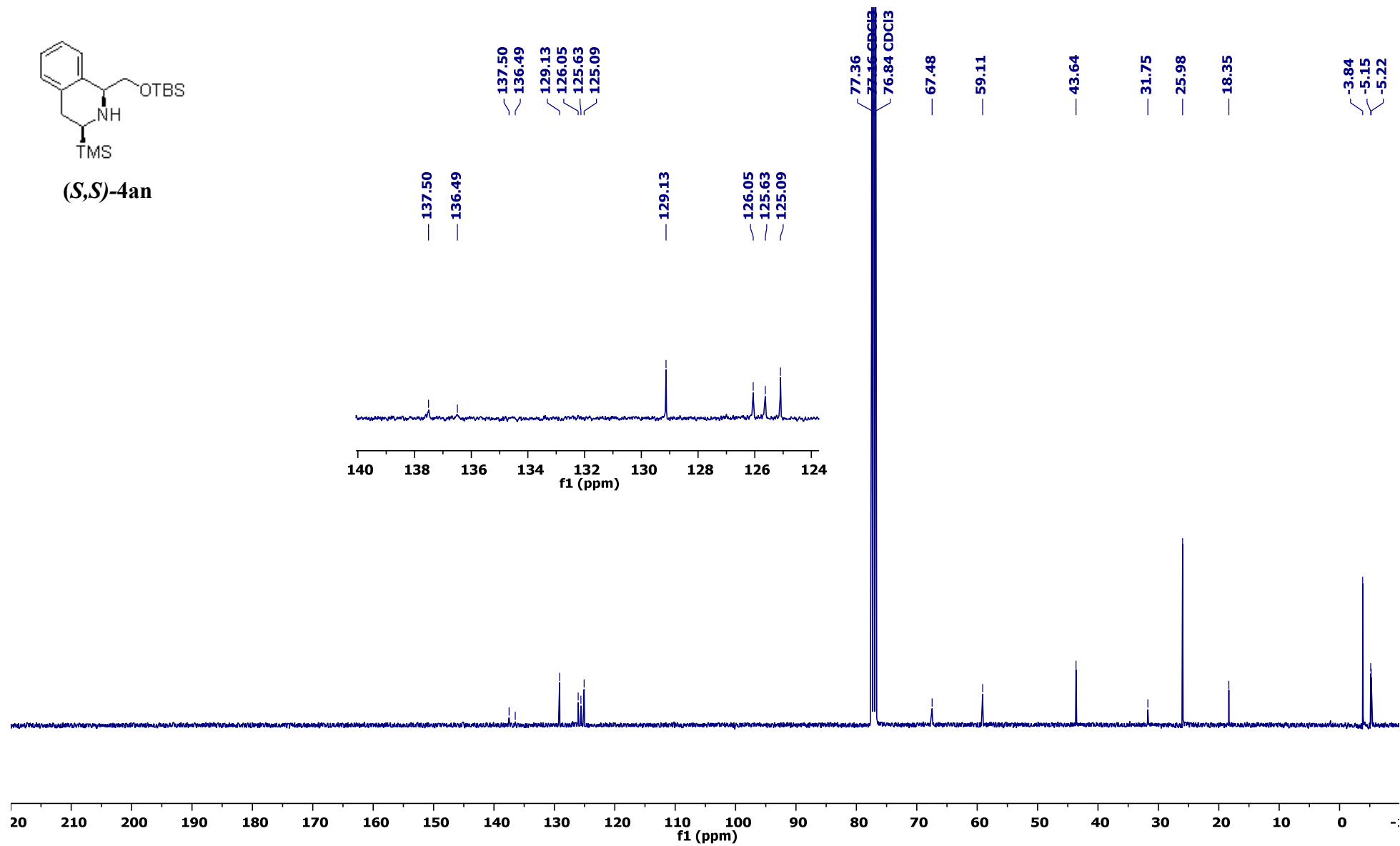
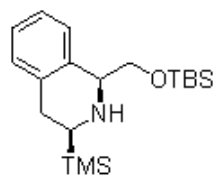
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



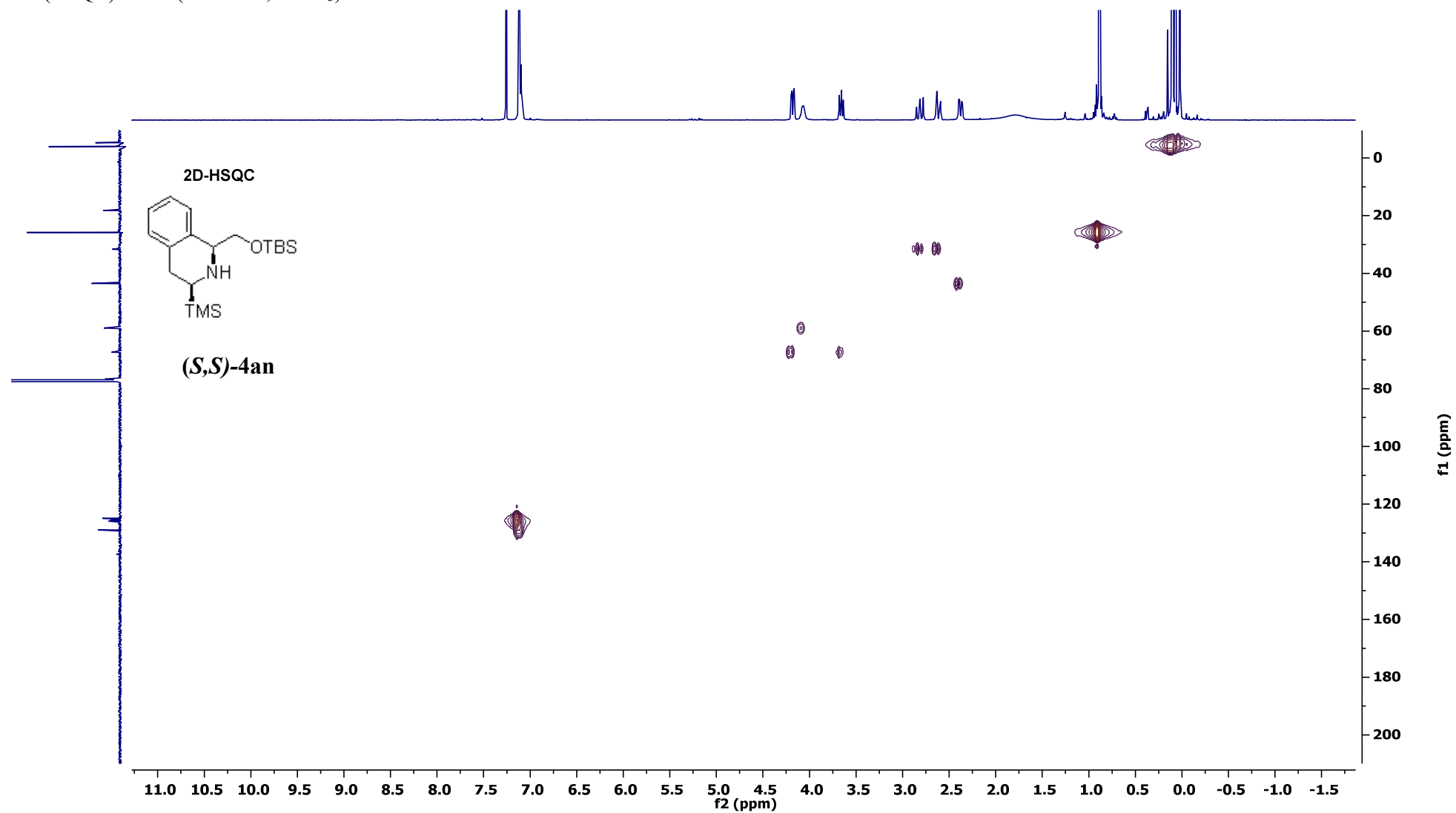
(S,S)-4an



$^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ )



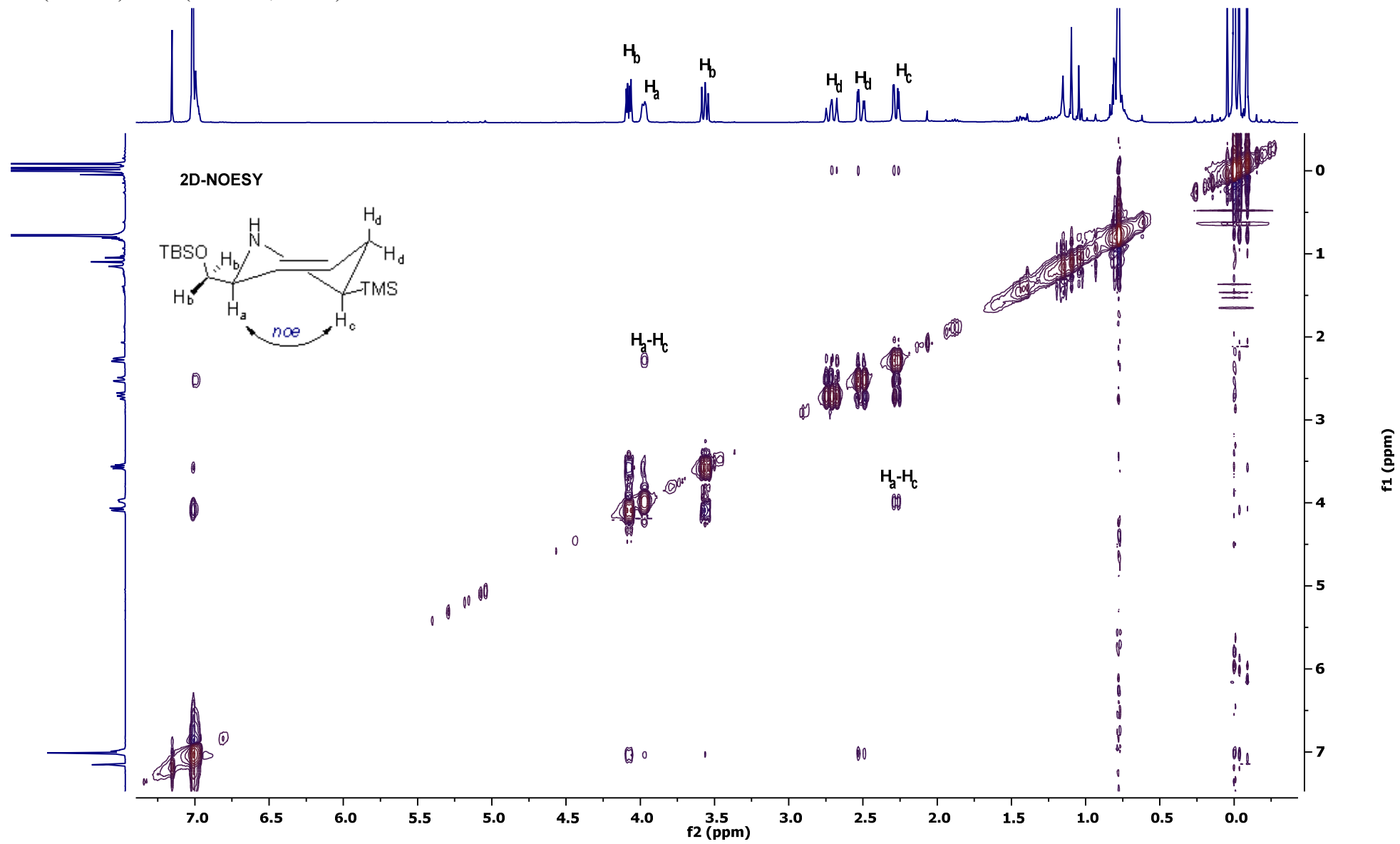
2D (HSQC) NMR (100 MHz, CDCl<sub>3</sub>)







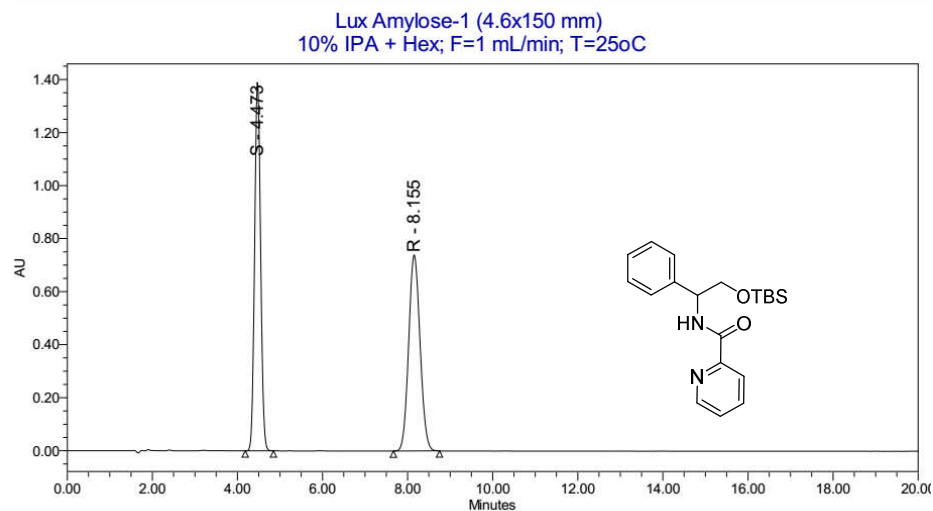
2D (NOESY) NMR (400 MHz, CDCl<sub>3</sub>)



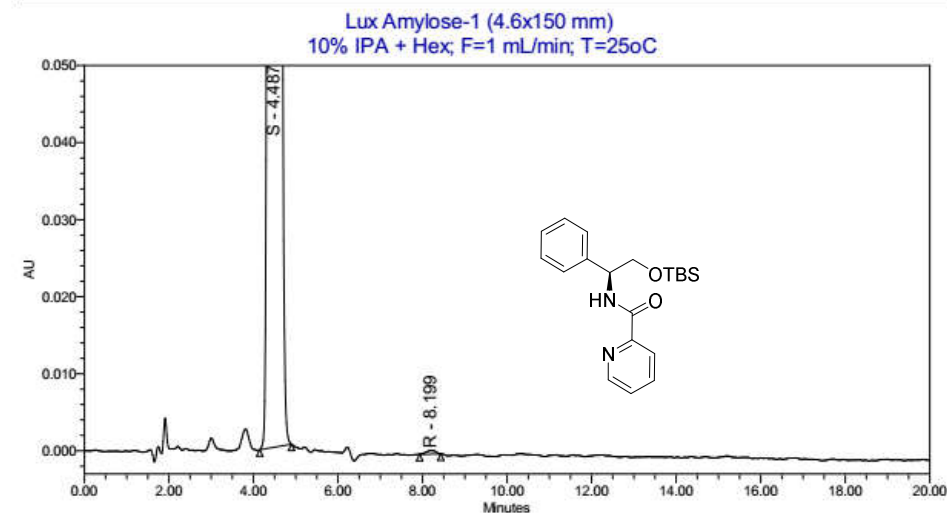
# HPLC data

SAMPLE INFORMATION			
Sample Name:	#876-Ch-276-JB-656 rac	Sample Set Name:	260819
Sample Type:	Unknown	Acq. Method Set:	Iz_210_254_F1_90%A_10%B
Vial:	38	Processing Method:	Ch_276 LA1
Injection #:	1	Channel Name:	W2489 ChA
Injection Volume:	10.00 ul	Proc. Chnl. Descr.:	W2489 ChA 210nm
Run Time:	20.0 Minutes		
Date Acquired:	8/26/2019 4:38:42 PM EEST	Acquired By:	System
Date Processed:	8/27/2019 9:06:34 AM EEST		

SAMPLE INFORMATION			
Sample Name:	#876-Ch-276-JB-663	Sample Set Name:	260819
Sample Type:	Unknown	Acq. Method Set:	Iz_210_254_F1_90%A_10%B
Vial:	39	Processing Method:	Ch_276 LA1
Injection #:	1	Channel Name:	W2489 ChA
Injection Volume:	10.00 ul	Proc. Chnl. Descr.:	W2489 ChA 210nm
Run Time:	20.0 Minutes		
Date Acquired:	8/26/2019 4:59:32 PM EEST	Acquired By:	System
Date Processed:	8/27/2019 9:06:58 AM EEST		



Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 S	4.473	13651802	49.77	1393305	4726			0.153	1.678
2 R	8.155	13777447	50.23	739801	4383	9.807	2.314	0.290	3.883

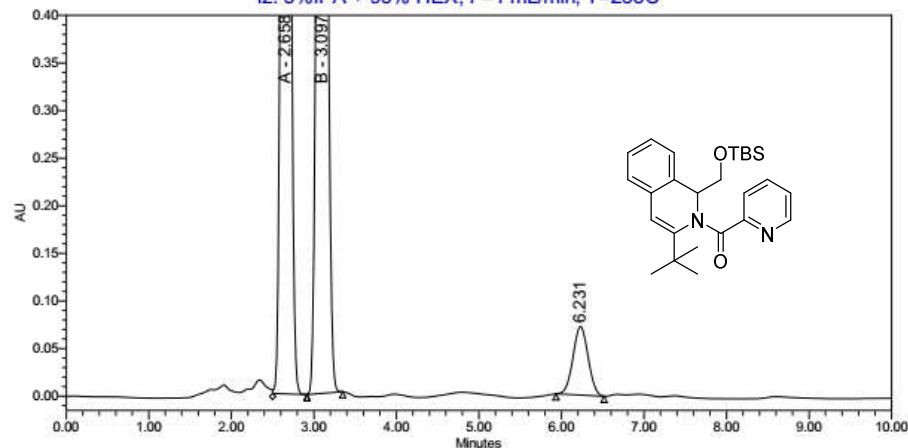


Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 S	4.487	32120072	99.98	2974539	3778			0.172	1.687
2 R	8.199	7615	0.02	472	4846	9.756	2.318	0.277	3.910

### SAMPLE INFORMATION

Sample Name:	892_Ch-278_LG-1-70-3	Sample Set Name:	29082019_Ch277
Sample Type:	Unknown	Acq. Method Set:	Iz_210_254_F1_95A_5B
Vial:	58	Processing Method:	Ch_278
Injection #:	1	Channel Name:	W2489 ChA
Injection Volume:	10.00 ul	Proc. Chnl. Descr.:	W2489 ChA 210nm
Run Time:	10.0 Minutes		
Date Acquired:	8/29/2019 11:10:17 AM EEST	Acquired By:	System
Date Processed:	8/29/2019 1:22:35 PM EEST		

Lux Amylose-1(2) (4.6x150 mm)  
Iz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



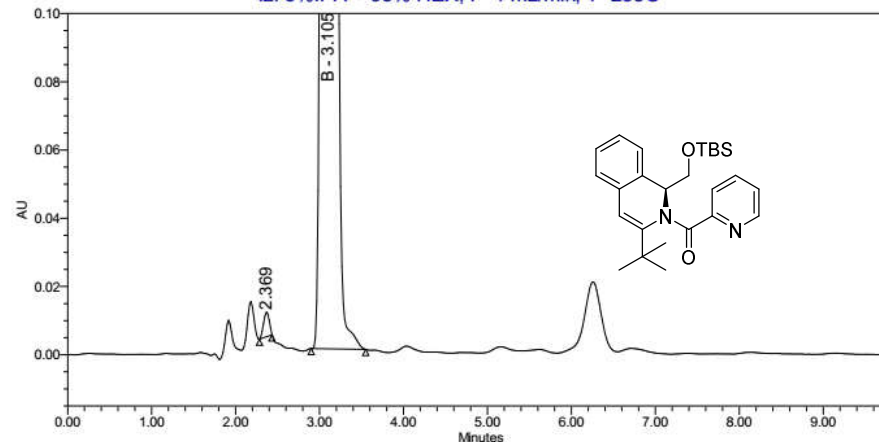
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 A	2.658	12236636	48.12	1970454	4079			0.098	0.641
2 B	3.097	12273656	48.27	1758255	4459	2.502	1.423	0.109	0.912
3	6.231	917256	3.61	72215	5455	12.016	3.121	0.199	2.846

### SAMPLE INFORMATION

Sample Name:	892_Ch-278_JB-4-53-2	Sample Set Name:	29082019_Ch277
Sample Type:	Unknown	Acq. Method Set:	Iz_210_254_F1_95A_5B
Vial:	64	Processing Method:	Ch_278
Injection #:	1	Channel Name:	W2489 ChA
Injection Volume:	25.00 ul	Proc. Chnl. Descr.:	W2489 ChA 210nm
Run Time:	20.0 Minutes		
Date Acquired:	8/29/2019 1:18:04 PM EEST	Acquired By:	System
Date Processed:	8/29/2019 1:26:36 PM EEST		

Lux Amylose-1(2) (4.6x150 mm)  
Iz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



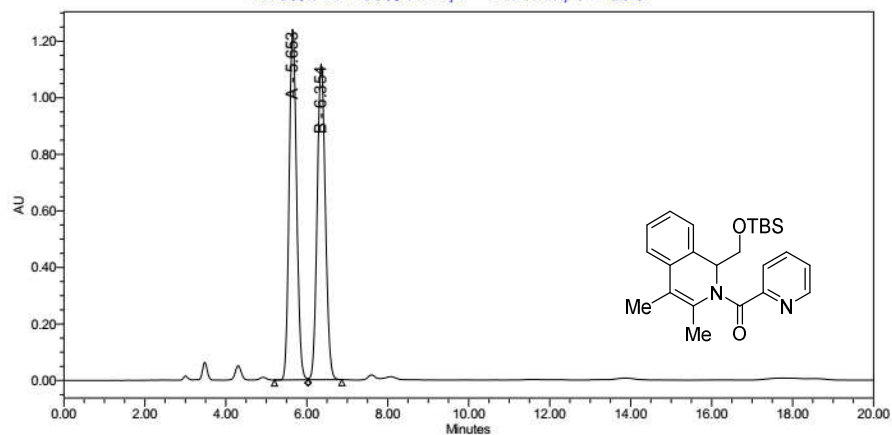
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1	2.369	33696	0.24	7181	5057			0.078	0.462
2 B	3.105	14086439	99.76	1931174	4156	4.528	1.982	0.113	0.917

### SAMPLE INFORMATION

Sample Name: 848-Ch-277-rac48-1	Sample Set Name: 21082019_Ch277
Sample Type: Unknown	Acq. Method Set: lz_210_254_F1_100%D
Vial: 36	Processing Method: Ch_277 LA1
Injection #: 1	Channel Name: W2489 ChA
Injection Volume: 10.00 ul	Proc. Chnl. Descr.: W2489 ChA 210nm
Run Time: 20.0 Minutes	
Date Acquired: 8/21/2019 4:16:31 PM EEST	Acquired By: System
Date Processed: 8/22/2019 9:23:42 AM EEST	

Lux Amylose-1(2) (4.6x150 mm)  
Iz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



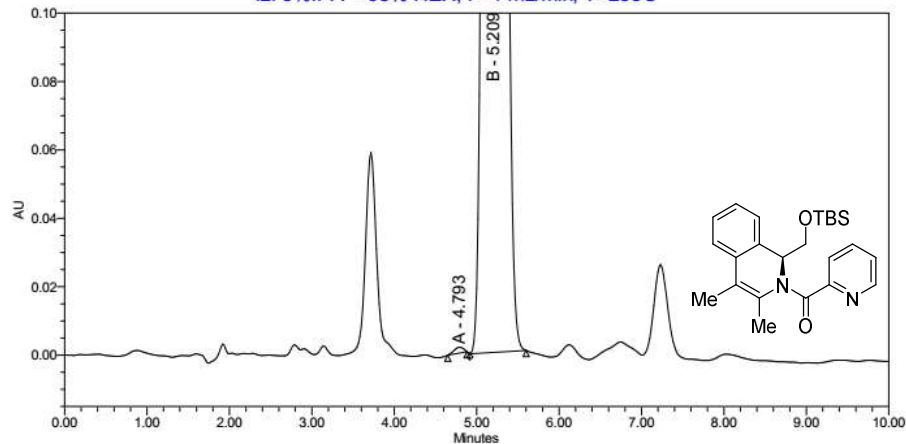
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 A	5.653	15739274	50.03	1239999	4526			0.198	2.385
2 B	6.354	15718707	49.97	1116981	4645	1.984	1.176	0.219	2.805

### SAMPLE INFORMATION

Sample Name: 891_Ch-277_JB-4-53-4	Sample Set Name: 29082019_Ch277
Sample Type: Unknown	Acq. Method Set: lz_210_254_F1_95A_5B
Vial: 57	Processing Method: Ch_277
Injection #: 1	Channel Name: W2489 ChA
Injection Volume: 10.00 ul	Proc. Chnl. Descr.: W2489 ChA 210nm
Run Time: 10.0 Minutes	
Date Acquired: 8/29/2019 10:49:16 AM EEST	Acquired By: System
Date Processed: 8/29/2019 3:39:08 PM EEST	

Lux Amylose-1(2) (4.6x150 mm)  
Iz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



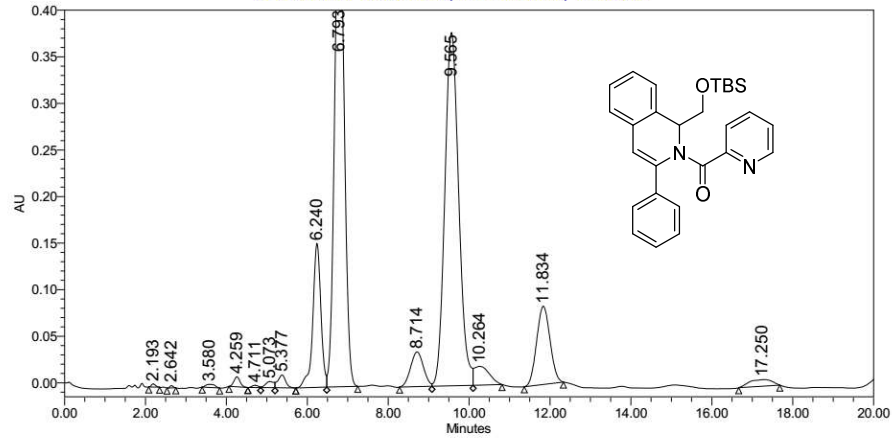
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 A	4.793	13728	0.07	1704	6761			0.137	1.959
2 B	5.209	19738196	99.93	1736085	4739	1.558	1.131	0.178	2.216

### SAMPLE INFORMATION

Sample Name: 894_Ch-281_LG-1-70-2	Sample Set Name: 29082019_Ch277
Sample Type: Unknown	Acq. Method Set: lz_210_254_F1_95A_5B
Vial: 60	Processing Method: Ch_281
Injection #: 1	Channel Name: W2489 ChA
Injection Volume: 10.00 ul	Proc. Chnl. Descr.: W2489 ChA 210nm
Run Time: 20.0 Minutes	
Date Acquired: 8/29/2019 12:39:12 PM EEST	Acquired By: System
Date Processed: 8/29/2019 1:45:22 PM EEST	

Lux Amylose-1(2) (4.6x150 mm)  
Iz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



C~1mg/mL (5%IPA + HEX)

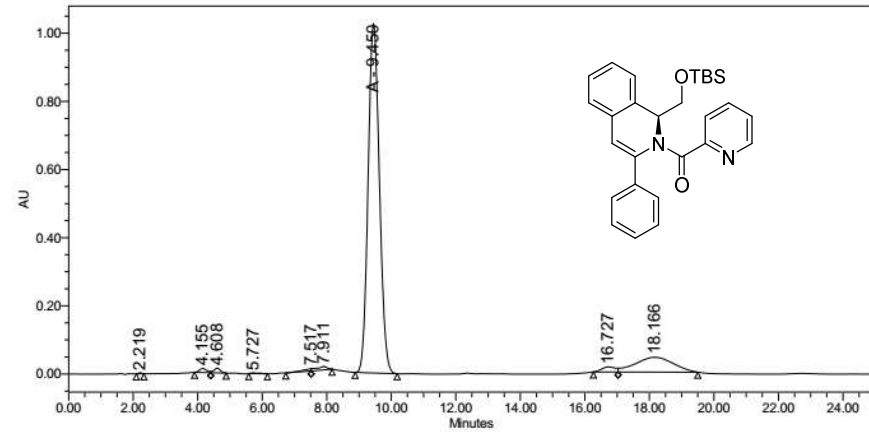
RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1	2.193	25300	0.10	3435			0.118	0.354
2	2.642	14530	0.06	2453	4141	2.468	1.783	0.097

RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
9	6.793	8927968	36.88	554714	4042	1.433	1.120	0.251
10	8.714	820985	3.39	37330	3457	3.776	1.371	0.349
11	9.565	9068986	37.47	379260	3723	1.400	1.120	0.369
12	10.264	521909	2.16	20071			1.088	5.336
13	11.834	1922022	7.94	83947	5922		1.182	0.362
14	17.250	282624	1.17	7016	3515		6.106	1.530

### SAMPLE INFORMATION

Sample Name: 938_Ch-281_JB-4-53-2hr	Sample Set Name: 12092019_Ch281
Sample Type: Unknown	Acq. Method Set: lz_210_254_F1_100%D
Vial: 40	Processing Method: Ch_281
Injection #: 1	Channel Name: W2489 ChA
Injection Volume: 10.00 ul	Proc. Chnl. Descr.: W2489 ChA 210nm
Run Time: 25.0 Minutes	
Date Acquired: 9/12/2019 11:59:46 AM EEST	Acquired By: System
Date Processed: 9/12/2019 12:29:46 PM EEST	

Lux Amylose-1(2) (4.6x150 mm)  
5% IPA + Hex; F=1 mL/min; T=25oC



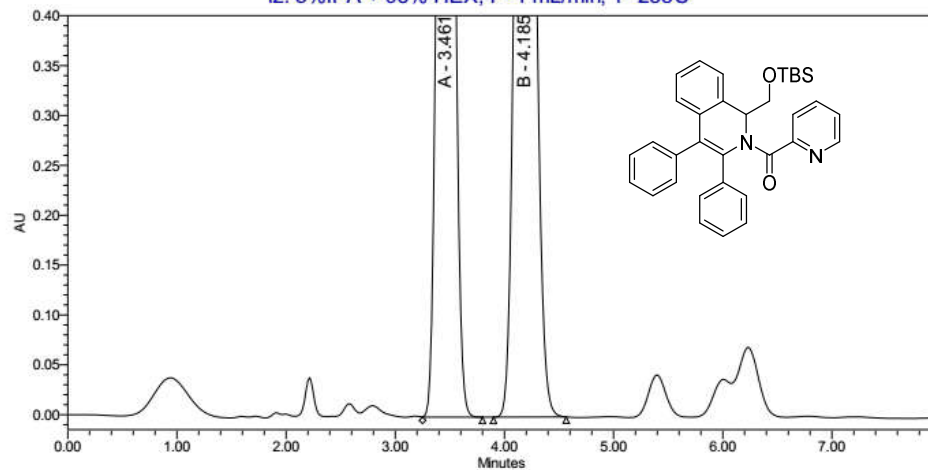
c=1 mg/ml KF

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1	2.219	19191	0.06	3134	2783			0.099	0.370
2	4.155	183508	0.62	11737	1629	6.691	4.229	0.242	1.565
3	4.608	191345	0.65	13676	2493	1.163	1.179	0.217	1.844
4	5.727	38758	0.13	2111	1469	2.322	1.375	0.352	2.535
5	7.517	133472	0.45	6519			1.436		3.640
6	7.911	244326	0.82	9986			1.067		3.883
7 A	9.450	24837247	83.82	1025026	3442		1.245	0.379	4.833
8	16.727	440624	1.49	14743			1.929		9.326

### SAMPLE INFORMATION

Sample Name: 893\_Ch-280\_LG-1-70-1  
 Sample Type: Unknown  
 Vial: 59  
 Injection #: 1  
 Injection Volume: 10.00 ul  
 Run Time: 10.0 Minutes  
 Date Acquired: 8/29/2019 11:21:04 AM EEST  
 Date Processed: 8/29/2019 1:32:04 PM EEST  
 Sample Set Name: 29082019\_Ch277  
 Acq. Method Set: lz\_210\_254\_F1\_95A\_5B  
 Processing Method: Ch\_280  
 Channel Name: W2489 ChA  
 Proc. Chnl. Descr.: W2489 ChA 210nm  
 Acquired By: System

Lux Amylose-1(2) (4.6x150 mm)  
 lz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



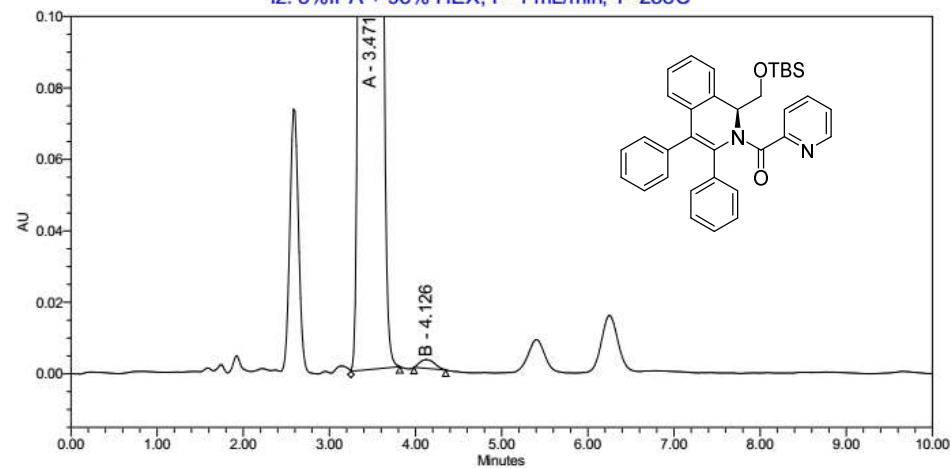
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 A	3.461	14669771	49.74	1750696	3844			0.131	1.136
2 B	4.185	14823296	50.26	1433536	3729	2.920	1.394	0.161	1.583

### SAMPLE INFORMATION

Sample Name: 893\_Ch-280\_JB-4-53-1  
 Sample Type: Unknown  
 Vial: 62  
 Injection #: 1  
 Injection Volume: 10.00 ul  
 Run Time: 10.0 Minutes  
 Date Acquired: 8/29/2019 12:03:51 PM EEST  
 Date Processed: 8/29/2019 1:37:18 PM EEST  
 Sample Set Name: 29082019\_Ch277  
 Acq. Method Set: lz\_210\_254\_F1\_95A\_5B  
 Processing Method: Ch\_280  
 Channel Name: W2489 ChA  
 Proc. Chnl. Descr.: W2489 ChA 210nm  
 Acquired By: System

Lux Amylose-1(2) (4.6x150 mm)  
 lz. 5%IPA + 95% HEX; F=1 mL/min; T=25oC



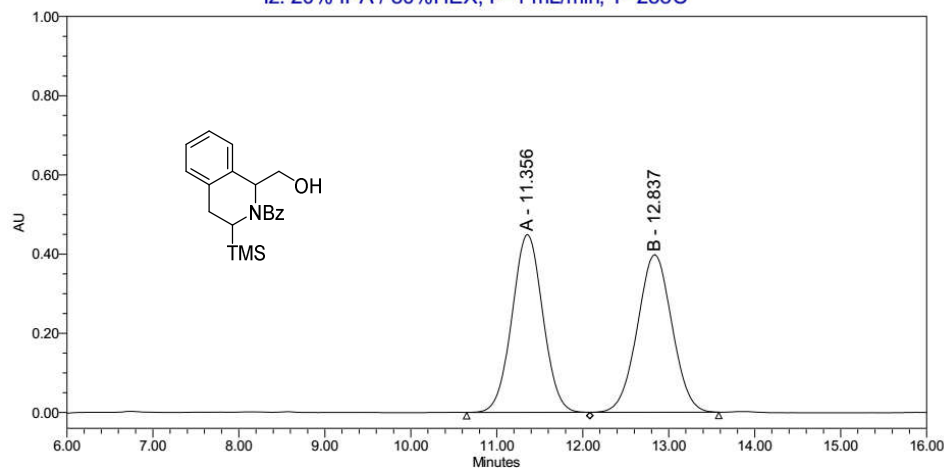
C~1mg/mL (5%IPA + HEX)

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime
1 A	3.471	30255822	99.90	3215236	2927			0.151	1.142
2 B	4.126	29569	0.10	2451	2291	2.186	1.354	0.203	1.547

### SAMPLE INFORMATION

Sample Name:	Ch-294-1335_JB-758r-IC-1 20%IPA		
Sample Type:	Unknown	Sample Set Name:	04122019_Ch294_IC1
Vial:	59	Acq. Method Set:	Iz_210_254_F1_80%A_20%B
Injection #:	1	Processing Method:	Chiralpak_IC1_t0
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	20.0 Minutes	Proc. Chnl. Descr.:	W2489 ChA 210nm
Date Acquired:	12/4/2019 9:20:31 AM EET	Acquired By:	System
Date Processed:	12/4/2019 9:45:06 AM EET		

Chiralpak IC-1(4.6x250 mm)  
Iz. 20% IPA / 80%HEX; F=1 mL/min; T=25oC



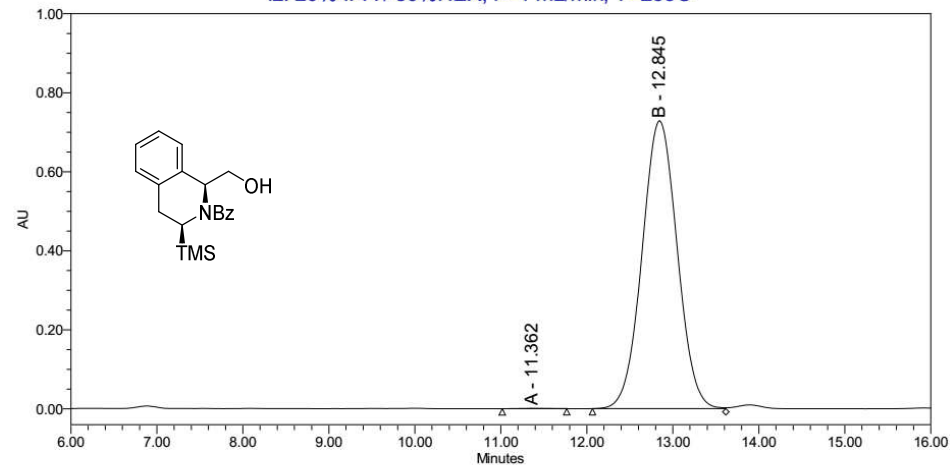
c=1 mg/ml KF

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime	RRT
1 A	11.356	11201424	50.10	449092	4783			0.386	2.594	
2 B	12.837	11157381	49.90	397634	4802	2.125	1.181	0.436	3.062	1.130

### SAMPLE INFORMATION

Sample Name:	Ch-294-1335_JB-759-IC-1 20%IPA		
Sample Type:	Unknown	Sample Set Name:	04122019_Ch294_IC1
Vial:	60	Acq. Method Set:	Iz_210_254_F1_80%A_20%B
Injection #:	1	Processing Method:	Chiralpak_IC1_t0
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	20.0 Minutes	Proc. Chnl. Descr.:	W2489 ChA 210nm
Date Acquired:	12/4/2019 9:42:37 AM EET	Acquired By:	System
Date Processed:	12/4/2019 10:11:36 AM EET		

Chiralpak IC-1(4.6x250 mm)  
Iz. 20% IPA / 80%HEX; F=1 mL/min; T=25oC



c=1 mg/ml KF

Peak Name	RT	Area	% Area	Height	EP Plate Count	Resolution	Selectivity	Width @ 50%	K Prime	RRT
1 A	11.362	19420	0.09	837	5287			0.368	2.595	
2 B	12.845	20664402	99.91	728623	4684	2.163	1.181	0.442	3.065	1.131