

## Supporting Information

### **Site-selective Ru-catalyzed C-H bond alkenylation with biologically relevant isoindolinones: a case of catalyst performance controlled by subtle stereo-electronic effects of the weak directing group**

Yu-Chao Yuan, Christian Bruneau, Thierry Roisnel and Rafael Gramage-Doria\*

Univ Rennes, CNRS, ISCR-UMR6226, F-35000 Rennes, France

\**E-mail:* rafael.gramage-doria@univ-rennes1.fr

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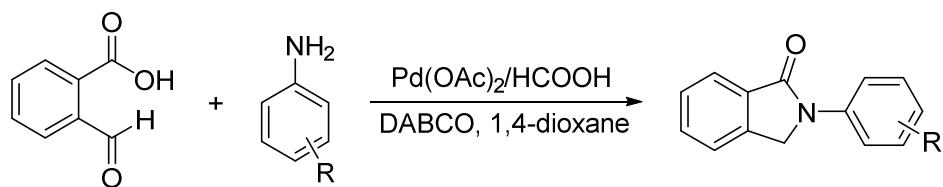
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## 1. General information.

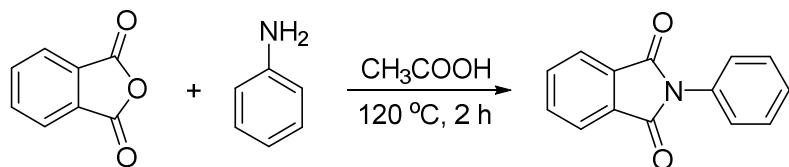
All reagents were obtained from commercial sources and used as supplied. All reactions were carried out in flame-dried glassware under argon atmosphere unless otherwise noted. Catalytic experiments were performed in Schlenk-type flasks under argon atmosphere unless otherwise noted. Organic solutions were concentrated under reduced pressure using a rotary evaporator. Thin-layer chromatography (TLC) were carried out on 0.25 mm Merck silica gel (60-F254). Flash column chromatography was performed using silica gel Silica 60 M, 0.04-0.063 mm. Technical grade petroleum ether (40-60), *n*-heptane and ethyl acetate were used for column chromatography.  $\text{CDCl}_3$  was stored under nitrogen over molecular sieves. NMR spectra were recorded on an AVANCE III 400 spectrometer.  $^1\text{H}$  NMR spectra were referenced to residual protiated solvent ( $\delta = 7.26$  ppm for  $\text{CDCl}_3$ ,  $\delta = 2.50$  ppm for  $\text{DMSO-}d_6$  and  $\delta = 2.05$  ppm for acetone- $d_6$ ) and  $^{13}\text{C}$  chemical shifts are reported relative to deuterated solvents ( $\delta = 77.0$  ppm for  $\text{CDCl}_3$ ,  $\delta = 39.5$  ppm for  $\text{DMSO-}d_6$  and  $\delta = 29.8$  ppm for acetone- $d_6$ ) [Note: acetone- $d_6$  contains traces of water at *ca.* 3 ppm]. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, and br. for broad. GC-MS analyses were performed with a GCMS-QP2010S (Shimadzu) instrument with a GC-2010 equipped with a 30 m capillary column (Supelco, SLBTM-5ms, fused silica capillary column, 30 m x 0.25 mm x 0.25 mm film thickness), which was used with helium as the vector gas. The following GC conditions were used: initial temperature 80 °C for 2 minutes, then rate 20 °C/min until 280 °C and 280 °C for 28 minutes. HRMS were recorded on a Waters Q-Tof 2 mass spectrometer at the corresponding facilities of the CRMPO, Centre Régional de Mesures Physiques de l'Ouest, Université de Rennes 1.

## 2. Synthesis and characterization of substrates 1.

**2.1. Method A:** A mixture of 2-formylbenzoic acid (5.0 mmol, 1 eq.), aniline derivative (6.0 mmol, 1.2 eq.), DABCO (10.0 mmol, 2 eq.), HCOOH (1.25 mL), Pd(OAc)<sub>2</sub> (0.25 mmol, 5 mol%) in 1,4-dioxane (5 mL) was heated to 80 °C for 3 h. After completion of the reaction, the mixture was cooled to room temperature and it was diluted in DCM (50 mL). The solid was removed by filtration, and the filtrate was washed subsequently with water (50 mL) and brine (50 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/acetone = 5/1, *v/v*) to afford the desired product.



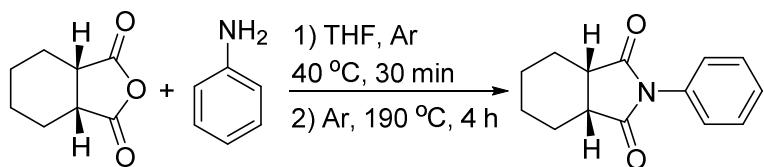
**2.2. Method B:** Phthalic anhydride (5 mmol, 0.74 g, 1 eq.) and the corresponding aniline (5 mmol, 1 eq.) were refluxed in acetic acid (30 mL) for 2-5 h. Once at room temperature, water was added and the solid recovered by filtration. After drying under vacuum the desired phthalimide was obtained.



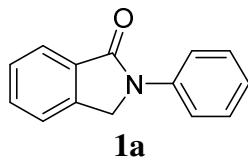
**2.3. Method C:** 1,2-Bis(bromomethyl)benzene (5.0 mmol, 1 eq.), DIPEA (12.5 mmol, 2.5 eq.), and aniline (7.50 mmol, 1.5 eq.) dissolved in toluene (25 mL) were added to a sealed tube before vigorously stirring at 110 °C under a N<sub>2</sub> atmosphere. The resulting mixture was cooled to room temperature and extracted with ethyl acetate (3 x 10 mL). The combined organic phase was washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo. The crude product was purified by column chromatography (petroleum ether) to obtain the desired product as a light yellow solid.



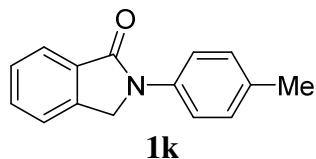
**2.4. Method D:** Hexahydrophthalic anhydride (10 mmol, 1.54 g, 1 eq.) and aniline (10 mmol, 1 eq.) and THF (15 mL) were added to a 100 mL round bottom flask. The solution was stirred for 30 min at 40 °C. Removal of the solvent using a rotary evaporator gave the corresponding carboxylic acid-amide as a white solid. The white solid was then heated at 190 °C under Ar for 4 h. The desired phthalimide was purified by silica gel column chromatography with a mixture of petroleum ether and ethyl acetate as eluent.



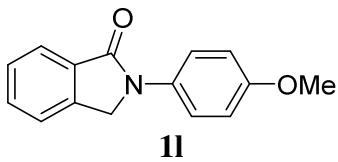
## **2.5. Characterization of substrates 1.**



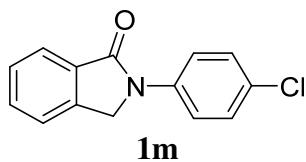
**2-Phenylisoindolin-1-one (1a):** Prepared according to **Method A** starting from aniline in 98% isolated yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.93 (d, *J* = 7.6 Hz, 1H), 7.89-7.86 (m, 2H), 7.62-7.58 (m, 1H), 7.53-7.49 (m, 2H), 7.43 (dd, *J* = 8.4, 7.2 Hz, 2H), 7.18 (dd, *J* = 7.2, 7.2 Hz, 1H), 4.87 (s, 2H) ppm. The spectral data match those previously reported.<sup>1</sup>



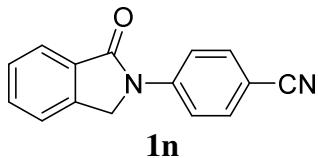
**2-(*p*-tolyl)isoindolin-1-one (1k):** Prepared according to **Method A** starting from *p*-toluidine in 80% isolated yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.92 (d, *J* = 7.2 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.59 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.50 (dd, *J* = 6.8, 6.8 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 4.84 (s, 2H), 2.36 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



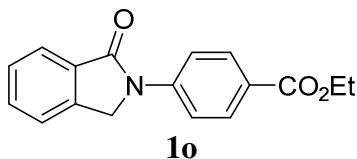
**2-(4-methoxyphenyl)isoindolin-1-one (1l):** Prepared according to **Method A** starting from *p*-anisidine in 62% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.92 (d,  $J$  = 7.6 Hz, 1H), 7.74 (d,  $J$  = 9.2 Hz, 2H), 7.58 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.52-7.48 (m, 2H), 6.97 (d,  $J$  = 9.2 Hz, 2H), 4.83 (s, 2H), 3.83 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



**2-(4-chlorophenyl)isoindolin-1-one (1m):** Prepared according to **Method A** starting from 4-chloroaniline in 61% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.92 (d,  $J$  = 7.2 Hz, 1H), 7.74 (dd,  $J$  = 9.2, 2.4 Hz, 2H), 7.61 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.54-7.50 (m, 2H), 7.39 (dd,  $J$  = 9.2, 2.4 Hz, 2H), 4.84 (s, 2H) ppm. The spectral data match those previously reported.<sup>1</sup>

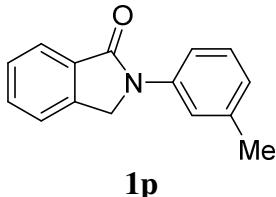


**4-(1-oxoisoindolin-2-yl)benzonitrile (1n):** Prepared according to **Method A** starting from 4-aminobenzonitrile in 64% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.06 (d,  $J$  = 9.2 Hz, 2H), 7.94 (d,  $J$  = 7.2 Hz, 1H), 7.71 (d,  $J$  = 8.8 Hz, 2H), 7.65 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.54 (dd,  $J$  = 7.6, 7.2 Hz, 2H), 4.89 (s, 2H) ppm. The spectral data match those previously reported.<sup>1</sup>

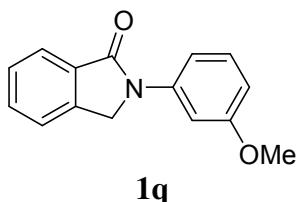


**Ethyl 4-(1-oxoisoindolin-2-yl)benzoate (1o):** Prepared according to **Method A** starting from ethyl 4-aminobenzoate in 96% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  =

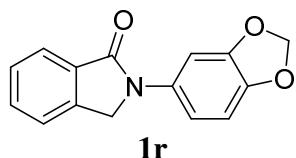
8.11 (d,  $J = 8.4$  Hz, 2H), 7.99 (d,  $J = 8.8$  Hz, 2H), 7.94 (d,  $J = 7.6$  Hz, 1H), 7.63 (dd,  $J = 7.6, 7.6$  Hz, 1H), 7.53 (dd,  $J = 7.6, 7.6$  Hz, 2H), 4.90 (s, 2H), 4.39 (q,  $J = 7.2$  Hz, 2H), 1.41 (t,  $J = 7.2$  Hz, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



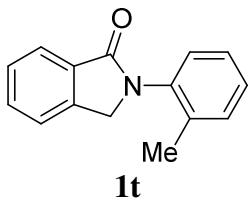
**2-(*m*-tolyl)isoindolin-1-one (1p):** Prepared according to **Method A** starting from *m*-toluidine in 65% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.93$  (d,  $J = 6.8$  Hz, 1H), 7.73 (s, 1H), 7.65-7.58 (m, 2H), 7.51 (dd,  $J = 7.2, 6.8$  Hz, 2H), 7.32 (dd,  $J = 8.0, 8.0$  Hz, 1H), 7.01 (d,  $J = 7.6$  Hz, 1H), 4.86 (s, 2H), 2.41 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



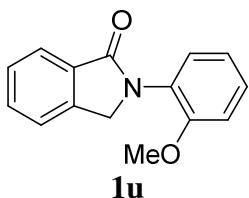
**2-(3-methoxyphenyl)isoindolin-1-one (1q):** Prepared according to **Method A** starting from *m*-anisidine in 80% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.93$  (d,  $J = 7.2$  Hz, 1H), 7.69 (dd,  $J = 2.0, 1.6$  Hz, 1H), 7.62-7.58 (m, 1H), 7.51 (dd,  $J = 7.6, 7.6$  Hz, 2H), 7.34-7.32 (m, 2H), 6.76-6.73 (m, 1H), 4.86 (s, 2H), 3.87 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



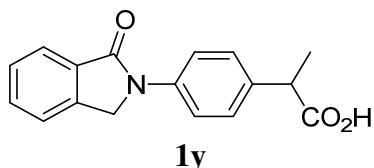
**2-(benzo[d][1,3]dioxol-5-yl)isoindolin-1-one (1r):** Prepared according to **Method A** starting from 3,4-(methylenedioxy)aniline in 58% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.92$  (d,  $J = 8.0$  Hz, 1H), 7.61-7.57 (m, 2H), 7.51 (dd,  $J = 6.4, 5.6$  Hz, 2H), 7.11 (dd,  $J = 8.4, 2.0$  Hz, 1H), 6.85 (d,  $J = 8.4$  Hz, 1H), 5.99 (s, 2H), 4.81 (s, 2H) ppm. The spectral data match those previously reported.<sup>1</sup>



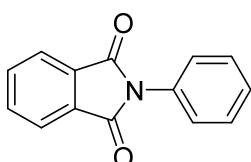
**2-(*o*-tolyl)isoindolin-1-one (1t):** Prepared according to **Method A** starting from *o*-toluidine in 68% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.96 (d,  $J$  = 7.6 Hz, 1H), 7.61 (ddd,  $J$  = 7.2, 7.2, 1.2 Hz, 1H), 7.55-7.50 (m, 2H), 7.35-7.32 (m, 1H), 7.30-7.24 (m, 3H), 4.74 (s, 2H), 2.27 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>



**2-(2-methoxyphenyl)isoindolin-1-one (1u):** Prepared according to **Method A** starting from *o*-anisidine in 61% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94 (d,  $J$  = 7.6 Hz, 1H), 7.58 (ddd,  $J$  = 7.2, 7.2, 1.2 Hz, 1H), 7.49 (dd,  $J$  = 7.6, 7.6 Hz, 2H), 7.43 (dd,  $J$  = 7.6, 2.0 Hz, 1H), 7.32 (ddd,  $J$  = 8.0, 8.0, 1.6 Hz, 1H), 7.06-7.00 (m, 2H), 4.80 (s, 2H), 3.82 (s, 3H) ppm. The spectral data match those previously reported.<sup>1</sup>

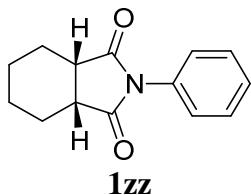


**2-(4-(1-oxoisoindolin-2-yl)phenyl)propanoic acid (also known as indoprofen, 1y):** Prepared according to **Method A** starting from 2-(4-aminophenyl)propanoic acid in 68% yield.  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  = 12.29 (s, 1H), 7.85 (d,  $J$  = 8.8 Hz, 2H), 7.78 (d,  $J$  = 7.6 Hz, 1H), 7.70-7.66 (m, 2H), 7.57-7.53 (m, 1H), 7.35 (d,  $J$  = 8.8 Hz, 2H), 5.02 (s, 2H), 3.69 (q,  $J$  = 7.2 Hz, 1H), 1.38 (d,  $J$  = 7.2 Hz, 3H) ppm. The spectral data match those previously reported.<sup>2</sup>

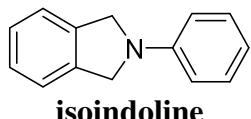


## 1z

**N-Phenylphthalimide (1z):** Prepared according to **Method B** starting from aniline in 80% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.96 (dd,  $J$  = 5.6, 3.2 Hz, 2H), 7.80 (dd,  $J$  = 5.2, 3.2 Hz, 2H), 7.52 (dd,  $J$  = 7.6, 7.6 Hz, 2H), 7.34-7.27 (m, 3H) ppm. The spectral data match those previously reported.<sup>3</sup>



**2-Phenylhexahydro-1*H*-isoindole-1,3(2*H*)-dione (1zz):** Prepared according to **Method D** in 86% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.48-7.44 (m, 2H), 7.39-7.35 (m, 1H), 7.30-7.27 (m, 2H), 3.06-3.00 (m, 2H), 1.95-1.85 (m, 4H), 1.53-1.50 (m, 4H) ppm. The spectral data match those previously reported.<sup>4</sup>

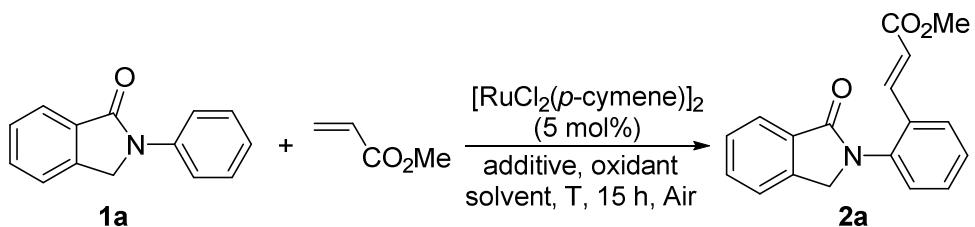


**2-Phenyl-2,3-dihydro-1*H*-isoindole:** Prepared according to **Method C** starting from 1,2-bis(bromomethyl)benzene in 89% isolated yield.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.38-7.31 (m, 6H), 6.78 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 6.71 (d,  $J$  = 7.6 Hz, 2H), 4.68 (s, 4H) ppm. The spectral data match those previously reported.<sup>5</sup>

### 3. Reaction optimization.

**3.1. General procedure:** In an oven dried Schlenk tube, to a solution of isoindolinone **1** (0.1 mmol, 1 eq.) and the corresponding alkene (0.2 mmol, 2 eq.) in 2-MeTHF (0.5 mL) was added the combined solids:  $[\text{RuCl}_2(p\text{-cymene})]_2$  (5 mol%),  $\text{AgSbF}_6$  (20 mol%) and  $\text{Cu}(\text{OAc})_2\text{H}_2\text{O}$  (0.1 mmol, 1 eq.). The Schlenk tube was sealed with a Teflon cap leaving the tap open to air and it was heated to 100 °C for 15 h. The reaction mixture was diluted in EtOAc and filtered using a silica plug eluting with EtOAc. The solvent was removed *in vacuo* and the crude mixture was purified by column chromatography (*n*-heptane/EtOAc, 4/1 to 1/1, *v/v*) to give the pure alkenylated product **2**.

### 3.2. Screening of reaction conditions.



Entry <sup>[a]</sup>	Additive	Oxidant	Solvent	T (°C)	Yield <sup>[b]</sup>
1 <sup>[c]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	120	77
2 <sup>[c]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	96
3 <sup>[c]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	80	50
4 <sup>[d]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	69
<b>5</b>	<b>AgSbF<sub>6</sub></b>	<b>Cu(OAc)<sub>2</sub>·H<sub>2</sub>O</b>	<b>2-MeTHF</b>	<b>100</b>	<b>97(94)</b>
6	AgSbF <sub>6</sub>	—	2-MeTHF	100	trace
7	—	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	29
8 <sup>[e]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	0
9 <sup>[f]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	46
10	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	THF	100	96
11	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	Dioxane	100	90
12	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	DCE	100	90
13	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	H <sub>2</sub> O	100	0
14	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	GVL	100	41
15	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	DEC	100	48
16	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	1-Pentanol	100	39
17 <sup>[g]</sup>	AgSbF <sub>6</sub>	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	2-MeTHF	100	93

<sup>[a]</sup>Reaction conditions: **1** (0.1 mmol), methyl acrylate (0.2 mmol), catalysts (5 mol%), additive (20 mol%), oxidant (100 mol%) and solvent (0.5 mL), 15 h, air. <sup>[b]</sup>Determined by <sup>1</sup>H NMR spectroscopy against an internal standard (dibromomethane). The isolated yield is shown in parentheses. <sup>[c]</sup>3.0 equivalents of methyl acrylate. <sup>[d]</sup>1.5 equivalents of methyl acrylate. <sup>[e]</sup>without [RuCl<sub>2</sub>(*p*-cymene)]<sub>2</sub>. <sup>[f]</sup>1 mol% of [RuCl<sub>2</sub>(*p*-cymene)]<sub>2</sub>. <sup>[g]</sup>under Ar.

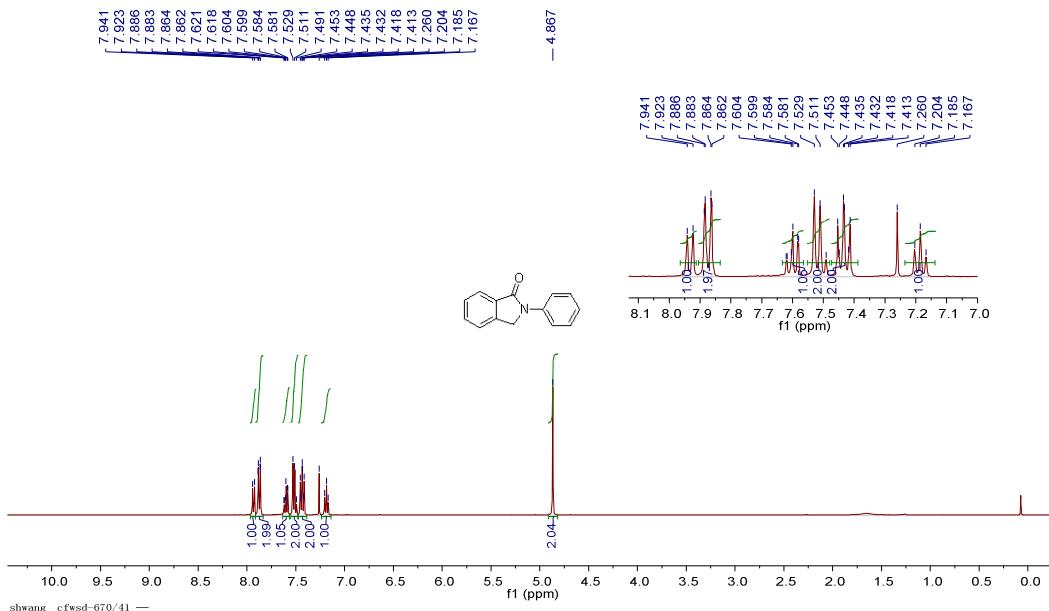
Note: GVL =  $\gamma$ -valerolactone, DEC = diethyl carbonate.

#### 4. Scale-up experiments

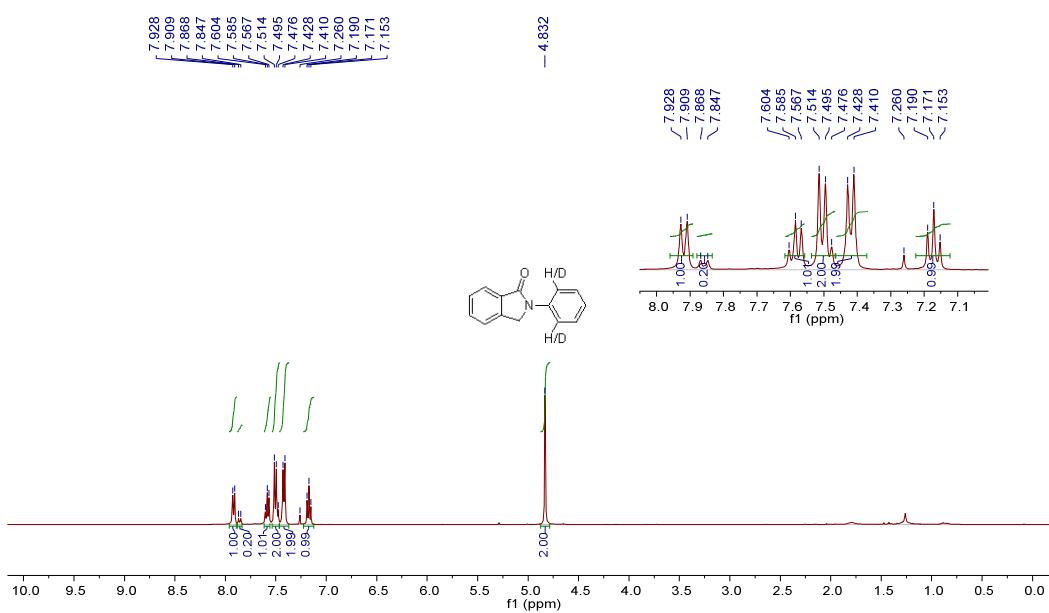
Procedure: In an oven dried Schlenk tube, to a solution of **1k** (3.0 mmol, 1 eq.) and methyl acrylate (6.0 mmol, 2 eq.) in 2-MeTHF (15 mL) was added the combined solids:  $[\text{RuCl}_2(p\text{-cymene})]_2$  (5 mol%),  $\text{AgSbF}_6$  (20 mol%) and  $\text{Cu}(\text{OAc})_2\text{H}_2\text{O}$  (1 eq.). The Schlenk tube was sealed with a Teflon cap leaving the tap open to air and it was heated to 100 °C for 15 h. The reaction mixture was diluted in EtOAc and filtered using a silica plug eluting with EtOAc. The solvent was removed *in vacuo* and the crude mixture was purified by column chromatography (*n*-heptane/EtOAc, 4/1 to 1/1, *v/v*) to give the alkenylated product **2k** as a white solid in 91% yield (839 mg.).

## 5. Deuteration experiments

In an oven dried Schlenk tube, to a solution of isoindolinone **1a** (0.1 mmol, 1 eq.) in 2-MeTHF (0.45 mL) and D<sub>2</sub>O (0.05 mL) was added the combined solids: [RuCl<sub>2</sub>(*p*-cymene)]<sub>2</sub> (5 mol%), AgSbF<sub>6</sub> (20 mol%) and Cu(OAc)<sub>2</sub> (1 eq.). The Schlenk tube was sealed with a Teflon cap leaving the tap open to air and it was heated to 100 °C for 15 h. The reaction mixture was diluted in EtOAc and filtered using a silica plug eluting with EtOAc. The solvent was removed *in vacuo* and the crude mixture was purified by column chromatography (*n*-heptane/EtOAc, 4/1 to 1/1, *v/v*) to give the deuterated product as a white solid in 90% yield (see spectra below).

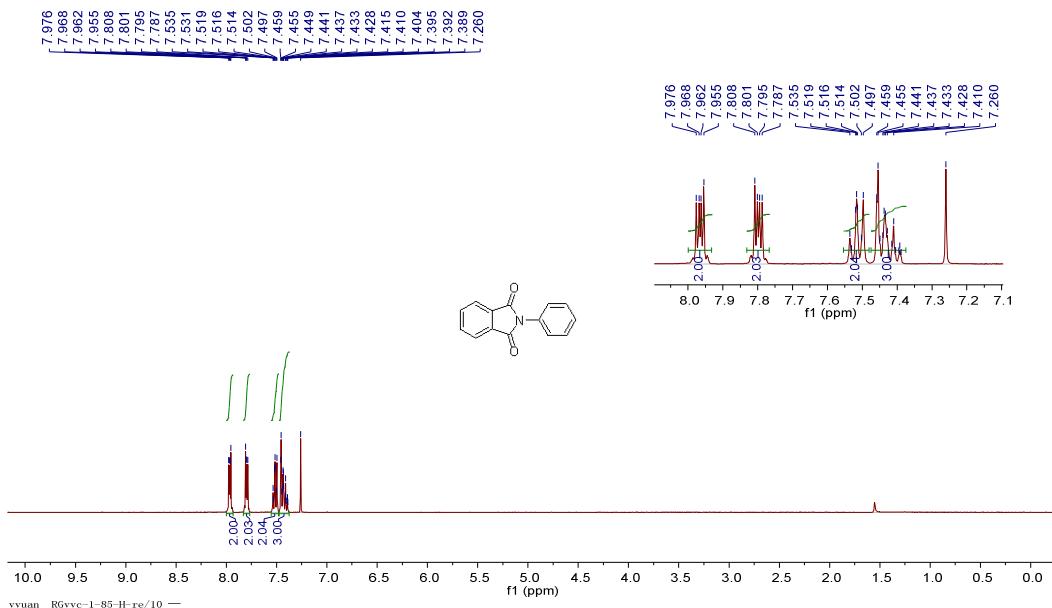


<sup>1</sup>H NMR (CDCl<sub>3</sub>) spectrum of **1a** in a reaction without using D<sub>2</sub>O.

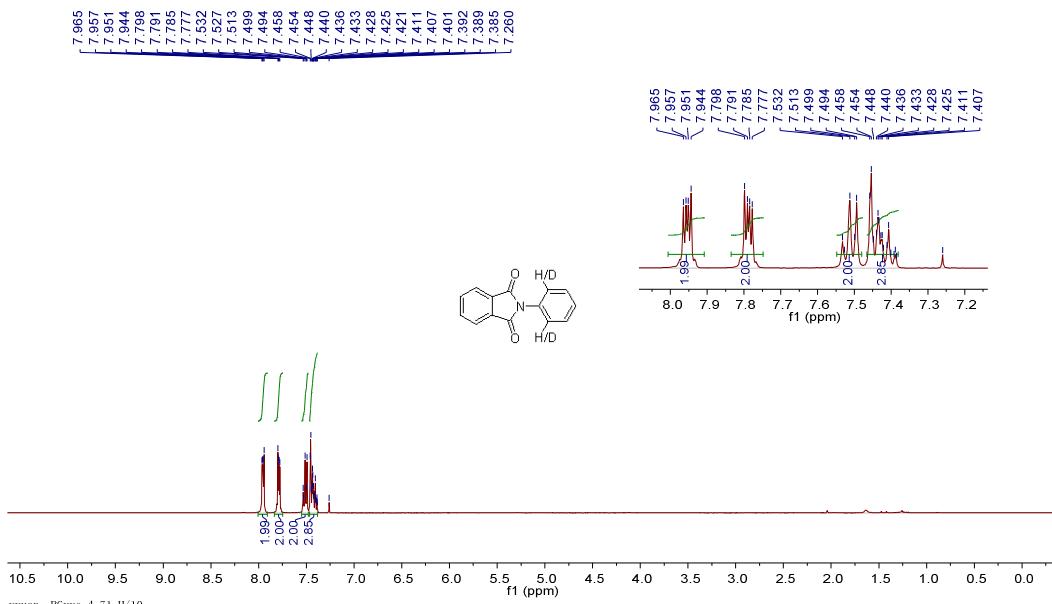


<sup>1</sup>H NMR (CDCl<sub>3</sub>) spectrum of **1a-d** showing low intensity of the peak at 7.86 ppm.

In an oven dried Schlenk tube, to a solution of *N*-phenylphthalimide **1a** (0.1 mmol, 1 eq.) in 2-MeTHF (0.45 mL) and D<sub>2</sub>O (0.05 mL) was added the combined solids: [RuCl<sub>2</sub>(*p*-cymene)]<sub>2</sub> (5 mol%), AgSbF<sub>6</sub> (20 mol%) and Cu(OAc)<sub>2</sub> (1 eq.). The Schlenk tube was sealed with a Teflon cap leaving the tap open to air and it was heated to 100 °C for 15 h. The reaction mixture was diluted in EtOAc and filtered using a silica plug eluting with EtOAc. The solvent was removed *in vacuo* and the crude mixture was purified by column chromatography (*n*-heptane/EtOAc, 4/1 to 1/1, *v/v*) to give the deuterated product as a white solid in 7.5% yield (see spectra below).

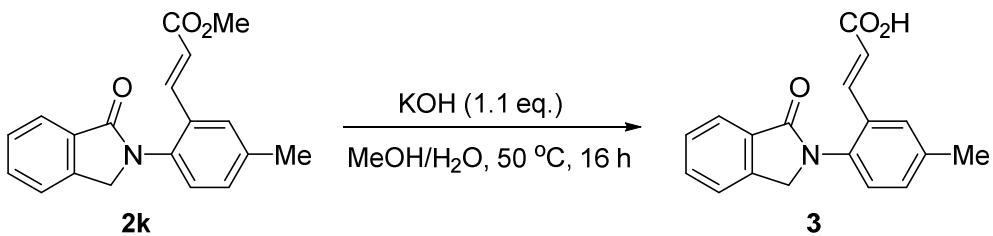


<sup>1</sup>H NMR (CDCl<sub>3</sub>) spectrum of **1z** in a reaction without using D<sub>2</sub>O.

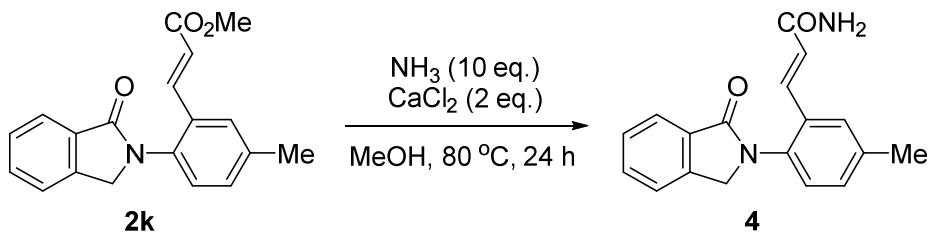


<sup>1</sup>H NMR (CDCl<sub>3</sub>) spectrum of **1z-d** showing the slow decrease of the intensity of the peak at 7.46-7.38 ppm.

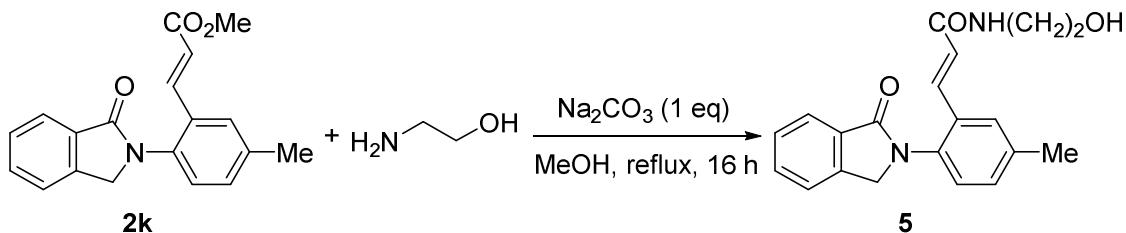
## 6. Post-functionalization reactions.



To a solution of **2k** (0.2 mmol, 1.0 eq.) in H<sub>2</sub>O (0.5 mL) and MeOH (0.5 mL) was added KOH (0.22 mmol, 1.1 eq.) and the reaction mixture was stirred at 50°C overnight. The mixture was brought to pH 1 with HCl 1 M and, then, it was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 10 mL). The organic phase was then washed with brine (30 mL) and it was dried over MgSO<sub>4</sub>. The resulting mixture was concentrated *in vacuo* to give the product **3** (58.7 mg, 99% yield).

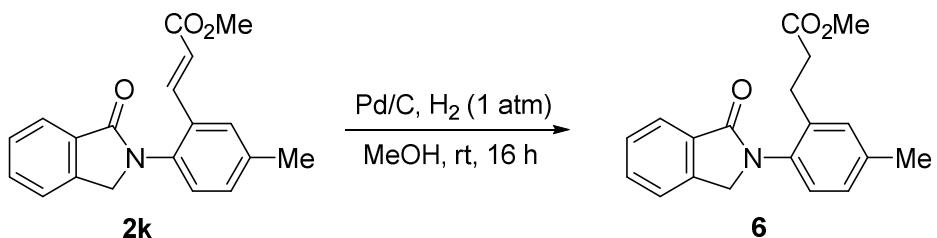


A schlenk tube was charged with **2k** (0.2 mmol, 1.0 eq.) and anhydrous calcium chloride (0.4 mmol, 2.0 eq.). The tube was protected under argon and ammonia in methanol solution (7 N, 2 mmol, 10.0 eq.) was added. The tube was sealed and heated at 80 °C overnight. The mixture was cooled to room temperature and the solvent was evaporated *in vacuo*. The residue was dissolved in water (30 mL) and it was extracted with dichloromethane (2 x 20 mL). The combined organic layer was dried over MgSO<sub>4</sub> and concentrated. The desired product **4** (36.9 mg, 63% yield) was obtained after purification by column chromatography.



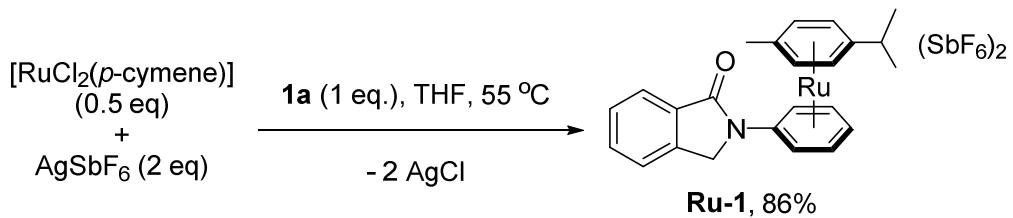
To a mixture of **2k** (0.2 mmol, 1.0 eq.) and Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol, 1.0 eq.) in MeOH (0.5 mL) was added ethanolamine (1 mmol, 5.0 eq.). The reaction mixture was heated to

reflux overnight before being allowed to return to room temperature. The reaction mixture was diluted in MeOH (10 mL) and the remaining solids were filtered. The filtrate was concentrated and the product **5** (54.6 mg, 77% yield) was purified by silica gel column chromatography (acetone/*n*-hexanes, 1/4 to 1/1, *v/v*).

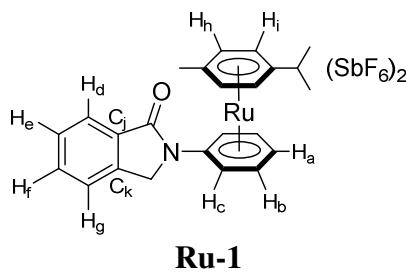


To an oven dried Schlenk tube, **2k** (0.2 mmol, 1.0 eq), palladium 10% on activated carbon (10 mg) and MeOH (1 mL) were added. Hydrogen gas was bubbled through the reaction mixture using a balloon. This was repeated twice before the Schlenk tube was sealed and a balloon of hydrogen gas was placed through the septum. The reaction was left to stir overnight at room temperature. Once TLC indicated completion of the reaction, the mixture was diluted in EtOAc and filtered through a plug of celite eluting with EtOAc. The filtrate was concentrate *in vacuo* to give the product **6** (61.9 mg, 99% yield).

## 7. Synthesis and characterization of Ru-1.

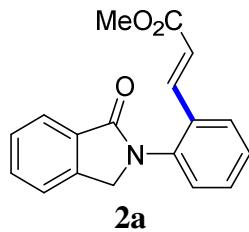


$[\text{RuCl}_2(p\text{-cymene})]_2$  (0.05 mmol, 0.5 eq.), **1a** (0.1 mmol, 1 eq.) and  $\text{AgSbF}_6$  (0.2 mmol, 2 eq.) were mixed in THF (0.5 mL) and stirred at 55 °C over 3 days. Once at room temperature, the reaction mixture was filtered over celite to remove  $\text{AgCl}$ . After solvents evaporation, NMR analysis indicated the presence of **Ru-1** and **1a** in a 86:14 ratio.

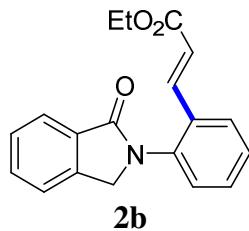


Brown solid, yield = 86%. Mp: >250 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{thf-}d_8$ ):  $\delta$  (assignment by COSY, HSQC and HMBC) = 7.94 (d,  $J$  = 7.7 Hz, 1H, Hd), 7.82-7.85 (m, 3H, Hf and Hc,), 7.77 (d,  $J$  = 7.6 Hz, 1H, Hg), 7.64 (t,  $J$  = 7.4 Hz, 1H, He), 7.06 (t,  $J$  = 6.4 Hz, 2H, Hb), 6.98 (s, 4H, Hh and Hi), 6.89 (t,  $J$  = 5.8 Hz, 1H, Ha), 5.15 (s, 2H), 2.99 (sept,  $J$  = 6.8 Hz, 1H, CH), 2.46 (s, 3H, Me), 1.34 (d,  $J$  = 6.9 Hz, 6H, Me) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{thf-}d_8$ ):  $\delta$  (assignment by COSY, HSQC and HMBC) = 169.22 (C=O), 141.65 (*ipso*-Ck), 135.03 (C-Hf), 129.51 (*ipso*-Cj), 129.09 (C-He), 124.56 (C-Hd), 123.93 (C-Hg), 122.88 (*ipso*-C-N), 121.61 (*ipso*-C of *para*-cymene), 112.24 (*ipso*-C of *para*-cymene), 93.96 (2 x C-Hi or C-Hh), 93.70 (2 x C-Hb), 91.49 (2 x C-Hh or C-Hi), 90.79 (C-Ha), 80.24 (2 x C-Hc), 50.31 ( $\text{CH}_2$ ), 32.02 (CH), 21.65 (2 x  $\text{CH}_3$ ), 18.37 ( $\text{CH}_3$ ) ppm.  $^{19}\text{F}\{\text{H}\}$  NMR (376 MHz,  $\text{thf-}d_8$ ):  $\delta$  = -126 (m) ppm. HRMS (ESI) calcd. for  $[\text{M}]^{2+}$   $\text{C}_{24}\text{H}_{25}\text{NO}^{102}\text{Ru}$  222.5484, found 222.5484 (0 ppm); calcd. for  $[\text{M} + \text{SbF}_6]^+$   $\text{C}_{24}\text{H}_{25}\text{NOF}_6^{102}\text{Ru}^{121}\text{Sb}$  679.9917, found 679.9918 (0 ppm); calcd. for  $[\text{SbF}_6]^- \text{F}_6^{121}\text{Sb}$  234.8948, found 234.8947 (0 ppm).

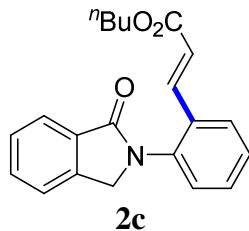
## 8. Characterization data of products 2-6.



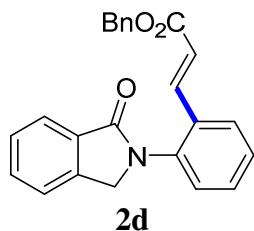
**Methyl (E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2a):** White solid, yield = 94%, 82.7 mg. Mp: 180-183 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.96 (d,  $J$  = 7.6 Hz, 1H), 7.73 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 7.67-7.61 (m, 2H), 7.56-7.46 (m, 3H), 7.40 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.35 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 6.46 (d,  $J$  = 16.0 Hz, 1H), 4.76 (s, 2H), 3.72 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.4, 167.1, 141.6, 140.3, 138.0, 132.7, 132.2, 132.0, 131.1, 128.6, 128.4, 128.2, 127.7, 124.7, 123.0, 120.2, 53.8, 51.8 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{15}\text{NO}_3\text{Na}$  316.0944, found 316.0943 (0 ppm).



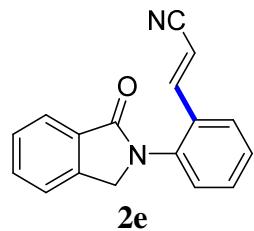
**Ethyl (E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2b):** White solid, yield = 89%, 61.2 mg. Mp: 148-151 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.95 (d,  $J$  = 7.6 Hz, 1H), 7.73 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 7.66-7.59 (m, 2H), 7.55-7.45 (m, 3H), 7.39 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.34 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 6.45 (d,  $J$  = 16.0 Hz, 1H), 4.75 (s, 2H), 4.18 (q,  $J$  = 7.2 Hz, 2H), 1.25 (t,  $J$  = 7.2 Hz, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.4, 166.7, 141.6, 140.0, 138.0, 132.8, 132.1, 132.0, 131.0, 128.5, 128.4, 128.2, 127.6, 124.6, 123.0, 120.6, 60.6, 53.8, 14.3 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_3\text{Na}$  330.1101, found 330.1101 (0 ppm).



**Butyl (E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2c):** White solid, yield = 91%, 61.4 mg. Mp: 104-107 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94 (d,  $J$  = 7.6 Hz, 1H), 7.73 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 7.65-7.58 (m, 2H), 7.54-7.44 (m, 3H), 7.38 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.34 (dd,  $J$  = 7.6, 1.2 Hz, 1H), 6.44 (d,  $J$  = 16.0 Hz, 1H), 4.74 (s, 2H), 4.11 (t,  $J$  = 6.4 Hz, 2H), 1.58 (m, 2H), 1.32 (m, 2H), 0.86 (t,  $J$  = 7.2 Hz, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 166.7, 141.5, 139.9, 137.9, 132.7, 132.1, 131.9, 131.0, 128.5, 128.4, 128.2, 127.5, 124.5, 122.9, 120.5, 64.5, 53.7, 30.7, 19.2, 13.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{21}\text{H}_{21}\text{NO}_3\text{Na}$  358.1414, found 358.1412 (0 ppm).

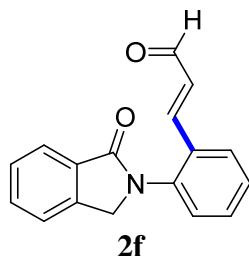


**Benzyl (E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2d):** White solid, yield = 81%, 59.5 mg. Mp < 50 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.97 (d,  $J$  = 7.6 Hz, 1H), 7.75-7.70 (m, 2H), 7.63 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.54 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.51-7.46 (m, 2H), 7.39 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.36-7.28 (m, 6H), 6.51 (d,  $J$  = 16.0 Hz, 1H), 5.18 (s, 2H), 4.75 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 166.4, 141.5, 140.5, 138.0, 136.0, 132.5, 132.1, 131.9, 131.1, 128.6, 128.5, 128.4, 128.17, 128.15, 128.08, 127.5, 124.5, 122.9, 120.0, 66.4, 53.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{24}\text{H}_{19}\text{NO}_3\text{Na}$  392.1257, found 392.1259 (0 ppm).

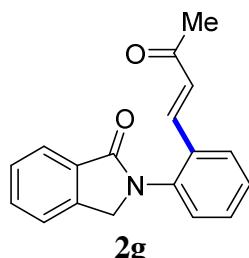


**(E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylonitrile (2e):** White solid, yield = 79%, 41.0 mg. Mp: 140-142 °C.  $^1\text{H}$  NMR (400 MHz, acetone- $d_6$ ):  $\delta$  = 7.90 (d,  $J$  = 8.0 Hz, 1H), 7.82 (d,  $J$  = 7.6 Hz, 1H), 7.71-7.65 (m, 2H), 7.62-7.55 (m, 4H), 7.49-7.45 (m, 1H), 6.28 (d,  $J$  = 16.4 Hz, 1H), 4.98 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz, acetone- $d_6$ ):  $\delta$  = 168.2, 147.6, 143.4, 139.1, 132.81, 132.80, 132.76, 132.4, 129.0, 128.8, 128.7, 127.3,

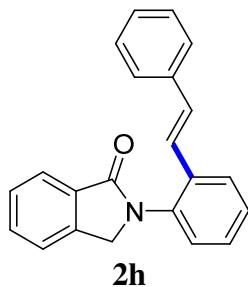
124.4, 124.2, 119.0, 98.6, 53.9 ppm. HRMS (ESI) calcd. for  $[M + Na]^+$   $C_{17}H_{12}N_2O\text{Na}$  283.0842, found 283.0842 (0 ppm).



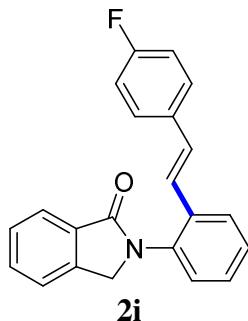
**(E)-3-(2-(1-oxoisoindolin-2-yl)phenyl)acrylaldehyde (2f):** Yellow solid, yield = 80%, 39.2 mg. Mp: 148-150 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 9.58 (d,  $J$  = 7.6 Hz, 1H), 7.96 (d,  $J$  = 7.2 Hz, 1H), 7.78 (d,  $J$  = 8.0 Hz, 1H), 7.65 (dd,  $J$  = 7.2, 7.2 Hz, 1H), 7.58-7.43 (m, 5H), 7.37 (d,  $J$  = 8.0 Hz, 1H), 6.71 (dd,  $J$  = 16.0, 7.6 Hz, 1H), 4.82 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 193.8, 168.2, 147.9, 141.5, 138.1, 132.4, 132.2, 132.0, 131.7, 130.2, 128.7, 128.6, 128.0, 127.9, 124.6, 123.1, 53.7 ppm. HRMS (ESI) calcd. for  $[M + Na]^+$   $C_{17}H_{13}NO_2\text{Na}$  286.0838, found 286.0839 (0 ppm).



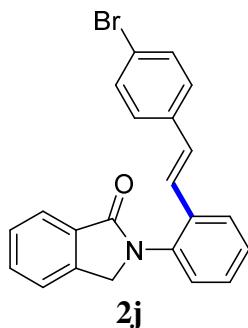
**(E)-2-(2-(3-oxobut-1-en-1-yl)phenyl)isoindolin-1-one (2g):** White solid, yield = 92%, 49.8 mg. Mp: 152-154 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.93 (d,  $J$  = 7.6 Hz, 1H), 7.73 (d,  $J$  = 7.6 Hz, 1H), 7.61 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.57-7.45 (m, 4H), 7.38 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.33 (d,  $J$  = 8.8 Hz, 1H), 6.68 (d,  $J$  = 16.4 Hz, 1H), 4.76 (s, 2H), 2.22 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 198.2, 168.2, 141.4, 138.8, 138.1, 132.6, 132.2, 131.2, 129.8, 128.8, 128.5, 128.3, 127.9, 127.6, 124.4, 123.0, 53.6, 27.7 ppm. HRMS (ESI) calcd. for  $[M + Na]^+$   $C_{18}H_{15}NO_2\text{Na}$  300.0995, found 300.0993 (1 ppm).



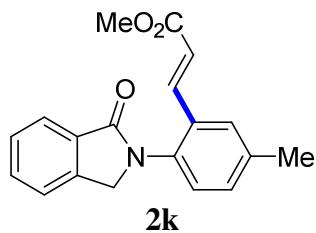
**(E)-2-(2-styrylphenyl)isoindolin-1-one (2h):** Yellow solid, yield = 64%, 39.9 mg. Mp: 169-171 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.03 (d,  $J$  = 7.6 Hz, 1H), 7.80 (d,  $J$  = 7.2 Hz, 1H), 7.64 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.57 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.51 (d,  $J$  = 7.2 Hz, 1H), 7.45-7.34 (m, 5H), 7.31 (dd,  $J$  = 7.6, 7.2 Hz, 2H), 7.24 (dd,  $J$  = 7.2, 7.2 Hz, 1H), 7.14 (d,  $J$  = 16.0 Hz, 1H), 7.09 (d,  $J$  = 16.0 Hz, 1H), 4.75 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 141.7, 137.1, 136.4, 135.6, 132.3, 131.9, 131.5, 128.7, 128.6, 128.5, 128.39, 128.37, 128.0, 126.9, 126.8, 124.4, 124.1, 123.0, 53.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{22}\text{H}_{17}\text{NONa}$  334.1202, found 334.1200 (1 ppm).



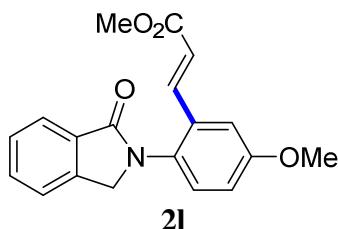
**(E)-2-(2-(4-fluorostyryl)phenyl)isoindolin-1-one (2i):** Yellow solid, yield = 63%, 41.2 mg. Mp: 49-51 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.01 (d,  $J$  = 7.2 Hz, 1H), 7.76 (dd,  $J$  = 7.2, 2.0 Hz, 1H), 7.63 (ddd,  $J$  = 7.2, 7.2, 1.2 Hz, 1H), 7.56 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.51 (d,  $J$  = 7.6 Hz, 1H), 7.43-7.32 (m, 5H), 7.07 (d,  $J$  = 16.4 Hz, 1H), 7.00-6.95 (m, 3H), 4.74 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.4, 162.6 (d,  $J_{\text{C}-\text{F}} = 246.2$  Hz), 141.7, 136.4, 135.5, 133.3 (d,  $J_{\text{C}-\text{F}} = 3.3$  Hz), 132.3, 132.0, 130.2, 128.7, 128.486 (d,  $J_{\text{C}-\text{F}} = 8.0$  Hz), 128.483, 128.4, 126.8, 124.5, 124.0 (d,  $J_{\text{C}-\text{F}} = 2.4$  Hz), 123.0, 115.7 (d,  $J_{\text{C}-\text{F}} = 21.5$  Hz), 53.8 ppm.  $^{19}\text{F}\{\text{H}\}$  NMR (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  = -113.6 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{22}\text{H}_{16}\text{NOFNa}$  352.1108, found 352.1108 (0 ppm).



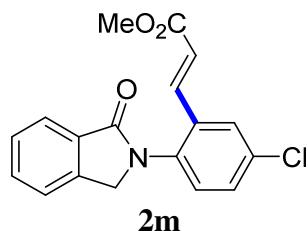
**(E)-2-(2-(4-bromostyryl)phenyl)isoindolin-1-one (2j):** Yellow solid, yield = 55%, 42.8 mg. Mp: 152-154 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.00 (d,  $J$  = 7.6 Hz, 1H), 7.77-7.75 (m, 1H), 7.62 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.56 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.50 (d,  $J$  = 7.6 Hz, 1H), 7.42-7.36 (m, 4H), 7.35-7.32 (m, 1H), 7.25 (d,  $J$  = 8.4 Hz, 2H), 7.06 (d,  $J$  = 16.4 Hz, 1H), 7.02 (d,  $J$  = 16.4 Hz, 1H), 4.74 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 141.6, 136.5, 136.1, 135.3, 132.2, 132.0, 131.8, 130.1, 128.9, 128.51, 128.48, 128.34, 128.31, 126.8, 124.91, 124.89, 124.5, 123.0, 121.8, 53.8 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{22}\text{H}_{16}\text{NOBrNa}$  412.0308, found 412.0309 (0 ppm).



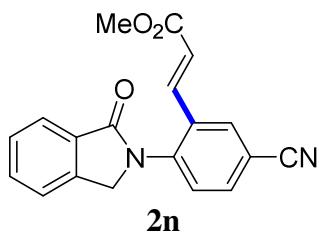
**Methyl (E)-3-(5-methyl-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2k):** White solid, yield = 93%, 85.9 mg. Mp: 173-175 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.95 (d,  $J$  = 7.2 Hz, 1H), 7.64-7.59 (m, 2H), 7.55-7.49 (m, 3H), 7.29 (dd,  $J$  = 8.0, 2.0 Hz, 1H), 7.22 (d,  $J$  = 8.0 Hz, 1H), 6.44 (d,  $J$  = 16.0 Hz, 1H), 4.72 (s, 2H), 3.71 (s, 3H), 2.41 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.5, 167.1, 141.6, 140.3, 138.4, 135.4, 132.3, 132.1, 132.04, 131.98, 128.5, 128.0, 124.5, 122.9, 119.9, 53.8, 51.8, 21.3 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_3\text{Na}$  330.1101, found 330.1103 (1 ppm).



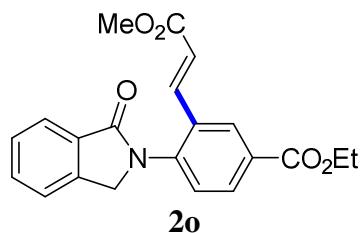
**Methyl (E)-3-(5-methoxy-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2l):** White solid, yield = 93%, 90.1 mg. Mp: 187-189 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94 (d,  $J$  = 7.6 Hz, 1H), 7.63-7.49 (m, 4H), 7.26-7.20 (m, 2H), 7.02 (dd,  $J$  = 8.4, 2.8 Hz, 1H), 6.43 (d,  $J$  = 16.0 Hz, 1H), 4.70 (s, 2H), 3.86 (s, 3H), 3.72 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.7, 167.0, 159.3, 141.6, 140.1, 133.8, 132.0, 130.9, 129.5, 128.5, 124.5, 122.9, 120.4, 117.2, 111.9, 55.7, 54.1, 51.8 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_4\text{Na}$  346.1049, found 346.1048 (0 ppm).



**Methyl (E)-3-(5-chloro-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2m):** White solid, yield = 92%, 90.5 mg. Mp: 201-203 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.95 (d,  $J$  = 7.6 Hz, 1H), 7.70 (s, 1H), 7.65 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.60-7.52 (m, 3H), 7.44 (d,  $J$  = 8.0 Hz, 1H), 7.31 (d,  $J$  = 8.4 Hz, 1H), 6.46 (d,  $J$  = 16.0 Hz, 1H), 4.75 (s, 2H), 3.74 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 166.7, 141.4, 139.0, 136.4, 134.2, 134.1, 132.3, 131.6, 130.9, 129.4, 128.6, 127.5, 124.6, 123.0, 121.2, 53.6, 51.9 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{14}\text{NO}_3\text{ClNa}$  350.0554, found 350.0557 (1 ppm).

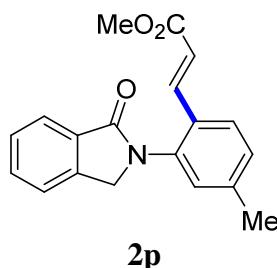


**Methyl (E)-3-(5-cyano-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2n):** White solid, yield = 41%, 38.9 mg. Mp: 214-216 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.97-7.93 (m, 2H), 7.72 (d,  $J$  = 8.4 Hz, 1H), 7.66 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.61-7.49 (m, 4H), 6.48 (d,  $J$  = 16.0 Hz, 1H), 4.80 (s, 2H), 3.75 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.1, 166.5, 141.8, 141.3, 138.6, 133.72, 133.69, 132.8, 131.8, 131.2, 128.9, 128.6, 124.8, 123.1, 122.0, 117.8, 112.0, 53.1, 52.1 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{14}\text{N}_2\text{O}_3\text{Na}$  341.0897, found 341.0898 (0 ppm).

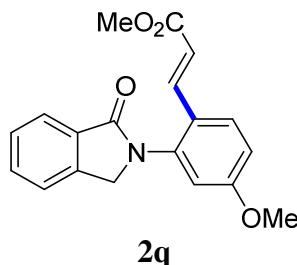


**Ethyl (E)-3-(3-methoxy-3-oxoprop-1-en-1-yl)-4-(1-oxoisindolin-2-yl)benzoate (2o):**

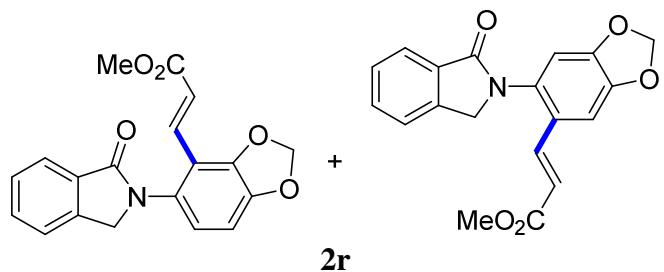
White solid, yield = 87%, 95.6 mg. Mp: 168-170 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.38 (d,  $J$  = 2.0 Hz, 1H), 8.10 (dd,  $J$  = 8.4, 2.0 Hz, 1H), 7.92 (d,  $J$  = 7.2 Hz, 1H), 7.65-7.60 (m, 2H), 7.54-7.49 (m, 2H), 7.42 (d,  $J$  = 8.4 Hz, 1H), 6.54 (d,  $J$  = 16.0 Hz, 1H), 4.79 (s, 2H), 4.40 (q,  $J$  = 7.2 Hz, 2H), 3.72 (s, 3H), 1.40 (t,  $J$  = 7.2 Hz, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.1, 166.8, 165.4, 141.7, 141.4, 139.6, 132.5, 132.4, 131.63, 131.59, 130.1, 129.1, 128.6, 127.7, 124.6, 123.0, 120.9, 61.5, 53.3, 51.8, 14.4 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{21}\text{H}_{19}\text{NO}_5\text{Na}$  388.1155, found 388.1153 (1 ppm).



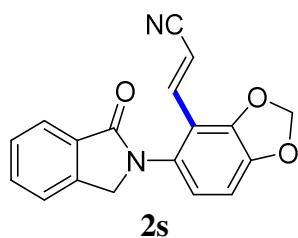
**Methyl (E)-3-(4-methyl-2-(1-oxoisindolin-2-yl)phenyl)acrylate (2p):** White solid, yield = 90%, 83.4 mg. Mp: 174-176 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.93 (d,  $J$  = 7.6 Hz, 1H), 7.63-7.58 (m, 3H), 7.53-7.48 (m, 2H), 7.19 (d,  $J$  = 8.4 Hz, 1H), 7.14 (s, 1H), 6.40 (d,  $J$  = 16.0 Hz, 1H), 4.72 (s, 2H), 3.69 (s, 3H), 2.37 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.4, 167.2, 141.8, 141.5, 140.0, 137.8, 132.0, 131.9, 129.7, 129.3, 128.7, 128.4, 127.4, 124.4, 122.9, 119.0, 53.8, 51.6, 21.3 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_3\text{Na}$  330.1101, found 330.1103 (1 ppm).



**Methyl (E)-3-(4-methoxy-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2q):** White solid, yield = 82%, 78.3 mg. Mp: 196-198 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94 (d,  $J$  = 7.6 Hz, 1H), 7.67 (d,  $J$  = 8.8 Hz, 1H), 7.64-7.49 (m, 4H), 6.95 (dd,  $J$  = 8.8, 2.4 Hz, 1H), 6.85 (d,  $J$  = 2.4 Hz, 1H), 6.33 (d,  $J$  = 16.0 Hz, 1H), 4.74 (s, 2H), 3.82 (s, 3H), 3.69 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.4, 167.4, 161.9, 141.5, 139.8, 139.4, 132.2, 131.9, 128.8, 128.5, 125.1, 124.6, 123.0, 117.6, 115.0, 113.2, 55.7, 53.9, 51.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_4\text{Na}$  346.1050, found 346.1052 (1 ppm).

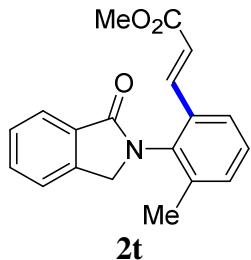


**Methyl (E)-3-(5-(1-oxoisoindolin-2-yl)benzo[d][1,3]dioxol-4-yl)acrylate (major) and methyl (E)-3-(6-(1-oxoisoindolin-2-yl)benzo[d][1,3]dioxol-5-yl)acrylate (minor) (2r, 80:20):** White solid, yield = 89%, 90.1 mg. Mp: 189-191 °C. NMR data corresponding to the major regioisomer:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.91 (d,  $J$  = 7.6 Hz, 1H), 7.59 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.52-7.47 (m, 2H), 7.36 (d,  $J$  = 16.4 Hz, 1H), 6.86-6.75 (m, 3H), 6.10 (s, 2H), 4.69 (s, 2H), 3.68 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.7, 167.5, 147.6, 147.5, 141.4, 134.9, 132.0, 131.8, 131.6, 128.4, 124.5, 123.2, 122.9, 121.5, 116.4, 109.5, 102.3, 54.2, 51.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{15}\text{NO}_5\text{Na}$  360.0842, found 360.0844 (0 ppm).

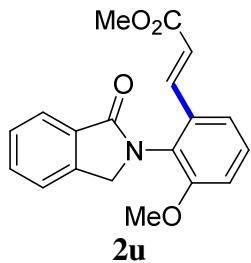


**(E)-3-(5-(1-Oxoisoindolin-2-yl)benzo[d][1,3]dioxol-4-yl)acrylonitrile (2s):** White solid, yield = 90%, 82.0 mg. Mp: 188-190 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94 (d,  $J$  = 7.6 Hz, 1H), 7.64 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.57-7.51 (m, 2H), 7.06 (d,  $J$  = 16.4 Hz, 1H), 6.91 (d,  $J$  = 8.0 Hz, 1H), 6.82 (d,  $J$  = 8.0 Hz, 1H), 6.33 (d,  $J$  = 16.4 Hz, 1H), 6.15

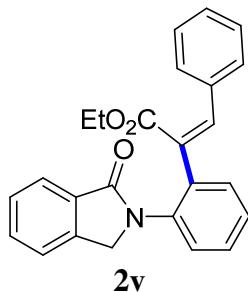
(s, 2H), 4.70 (s, 2H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.7, 147.8, 147.7, 141.3, 141.2, 132.4, 131.5, 131.1, 128.7, 124.6, 123.0, 121.6, 118.4, 115.7, 110.4, 102.7, 101.8, 54.2 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{12}\text{N}_2\text{O}_3\text{Na}$  327.0740, found 327.0745 (2 ppm).



**Methyl (E)-3-(3-methyl-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2t):** White solid, yield = 54%, 38.9 mg. Mp: 137-139 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.97 (d,  $J$  = 7.6 Hz, 1H), 7.64-7.50 (m, 5H), 7.38-7.32 (m, 2H), 6.42 (d,  $J$  = 16.0 Hz, 1H), 4.64 (d,  $J$  = 17.2 Hz, 1H), 4.55 (d,  $J$  = 17.2 Hz, 1H), 3.69 (s, 3H), 2.19 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.3, 167.0, 141.7, 140.1, 137.9, 136.4, 133.7, 132.9, 132.1, 131.9, 128.9, 128.5, 125.2, 124.6, 123.1, 120.7, 52.6, 51.8, 18.1 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_3\text{Na}$  330.1101, found 330.1105 (1 ppm).

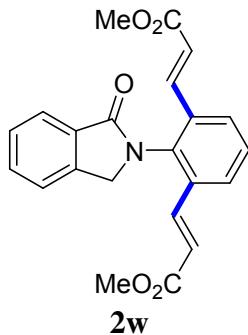


**Methyl (E)-3-(3-methoxy-2-(1-oxoisoindolin-2-yl)phenyl)acrylate (2u):** White solid, yield = 56%, 54.3 mg. Mp: 173-175 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.92 (d,  $J$  = 7.6 Hz, 1H), 7.65-7.57 (m, 2H), 7.49 (dd,  $J$  = 7.2, 7.2 Hz, 2H), 7.38-7.29 (m, 2H), 7.01 (d,  $J$  = 8.0 Hz, 1H), 6.44 (d,  $J$  = 16.0 Hz, 1H), 4.83 (d,  $J$  = 16.8 Hz, 1H), 4.49 (d,  $J$  = 16.8 Hz, 1H), 3.74 (s, 3H), 3.67 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.8, 166.9, 156.3, 142.4, 139.8, 134.9, 132.0, 131.8, 129.4, 128.0, 126.3, 124.4, 122.9, 120.7, 118.8, 113.2, 55.9, 52.0, 51.6 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{19}\text{H}_{17}\text{NO}_4\text{Na}$  346.1050, found 346.1047 (1 ppm).



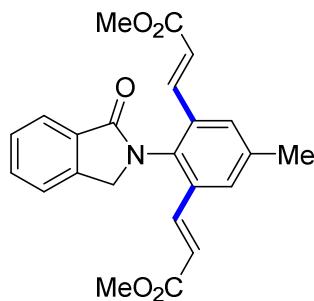
**2v**

**Ethyl (Z)-2-(2-(1-oxoisoindolin-2-yl)phenyl)-3-phenylacrylate (2v):** Brown solid, yield = 26%, 19.9 mg. Mp: 94-96 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.89 (d,  $J$  = 7.2 Hz, 1H), 7.58-7.40 (m, 7H), 7.36 (d,  $J$  = 7.2 Hz, 1H), 7.28-7.25 (m, 4H), 7.13 (s, 1H), 4.72 (s, 2H), 3.62 (q,  $J$  = 7.2 Hz, 2H), 0.68 (t,  $J$  = 7.2 Hz, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.8, 168.0, 142.0, 137.6, 136.9, 136.6, 135.8, 132.8, 132.0, 131.8, 130.9, 129.6, 128.6, 128.4, 128.3, 128.1, 124.2, 122.9, 61.2, 52.7, 13.2 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{25}\text{H}_{21}\text{NO}_3\text{Na}$  406.1414, found 406.1414 (0 ppm).



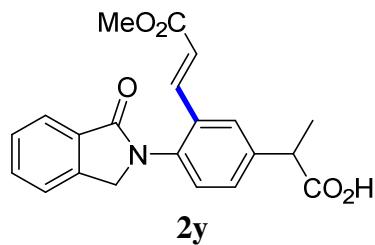
**2w**

**Dimethyl 3,3'-(2-(1-oxoisoindolin-2-yl)-1,3-phenylene)(2E,2'E)-diacrylate (2w):** White solid, yield = 19%, 21.5 mg. Mp: 211-213 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.99 (d,  $J$  = 7.6 Hz, 1H), 7.76 (d,  $J$  = 8.0 Hz, 2H), 7.65 (ddd,  $J$  = 7.6, 7.6, 1.2 Hz, 1H), 7.58 (d,  $J$  = 7.2, 1H), 7.55-7.46 (m, 4H), 6.45 (d,  $J$  = 16.0 Hz, 2H), 4.63 (s, 2H), 3.69 (s, 6H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.8, 166.7, 141.5, 139.2, 136.8, 134.7, 132.4, 131.3, 129.3, 129.1, 128.8, 125.0, 123.2, 121.8, 53.6, 51.9 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{22}\text{H}_{19}\text{NO}_5\text{Na}$  400.1155, found 400.1158 (1 ppm).

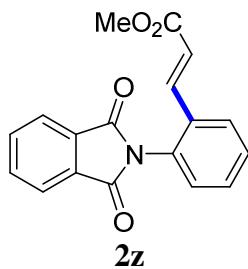


**2x**

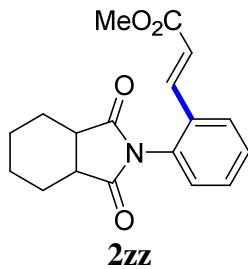
**Dimethyl 3,3'-(5-methyl-2-(1-oxoisoindolin-2-yl)-1,3-phenylene)(2E,2'E)-diacrylate (2x):** White solid, yield = 19%, 14.9 mg. Mp: 241-243 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.96 (d,  $J$  = 7.6 Hz, 1H), 7.62 (dd,  $J$  = 7.6, 7.2 Hz, 1H), 7.55-7.44 (m, 6H), 6.42 (d,  $J$  = 16.0 Hz, 2H), 4.59 (s, 2H), 3.67 (s, 6H), 2.42 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.8, 166.7, 141.5, 139.2, 134.4, 134.2, 132.3, 131.3, 129.8, 128.6, 124.8, 123.2, 121.4, 53.6, 51.8, 21.3 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{23}\text{H}_{21}\text{NO}_5\text{Na}$  414.1312, found 414.1314 (0 ppm).



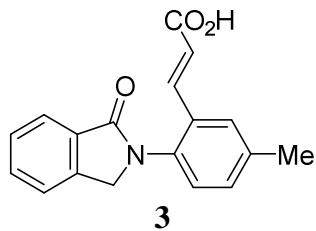
**1-(2-Hydroxyphenyl)-3-methyl-1*H*-pyrrole-2,5-dione (2y):** White solid, yield = 92%, 67.2 mg. Mp: 194-196 °C.  $^1\text{H}$  NMR (400 MHz, acetone- $d_6$ ):  $\delta$  = 7.90 (s, 1H), 7.83 (d,  $J$  = 7.6 Hz, 1H), 7.71-7.50 (m, 6H), 6.56 (d,  $J$  = 16.0 Hz, 1H), 4.94 (s, 2H), 3.92 (q,  $J$  = 7.2 Hz, 1H), 3.66 (s, 3H), 1.54 (d,  $J$  = 7.2 Hz, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz, acetone- $d_6$ ):  $\delta$  = 175.2, 168.2, 167.3, 143.3, 142.2, 141.2, 138.2, 133.4, 132.9, 132.8, 131.0, 129.12, 129.07, 127.4, 124.5, 124.3, 120.3, 54.0, 51.8, 45.5, 19.0 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{21}\text{H}_{19}\text{NO}_5\text{Na}$  388.1155, found 388.1150 (1 ppm).



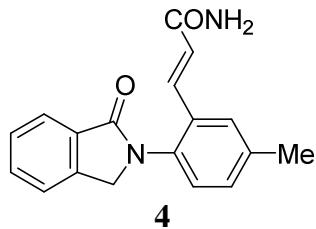
**Methyl (E)-3-(2-(1,3-dioxoisoindolin-2-yl)phenyl)acrylate (2z):** Yellow solid, yield = 20%, 24.6 mg. Mp: 206-208 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.97 (dd,  $J$  = 5.6, 3.2 Hz, 2H), 7.83-7.78 (m, 3H), 7.55-7.48 (m, 3H), 7.29 (dd,  $J$  = 7.2, 1.6 Hz, 1H), 6.47 (d,  $J$  = 16.0 Hz, 1H), 3.72 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 167.4, 166.9, 139.3, 134.7, 133.2, 131.9, 131.2, 131.0, 129.8, 129.5, 127.5, 124.2, 120.7, 51.9 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{13}\text{NO}_4\text{Na}$  330.0737, found 330.0737 (0 ppm).



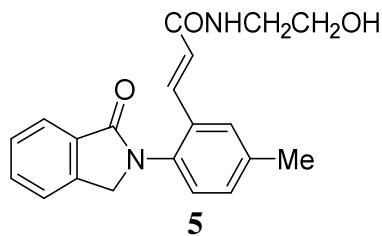
**Methyl (E)-3-(2-(1,3-dioxooctahydro-2H-isoindol-2-yl)phenyl)acrylate (2zz):** White solid, yield = 71%, 88.7 mg. Mp: 108-110 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.72 (d,  $J$  = 7.2 Hz, 1H), 7.50-7.39 (m, 3H), 7.16-7.10 (m, 1H), 6.41 (d,  $J$  = 16.0 Hz, 1H), 3.74 (s, 3H), 3.14-3.05 (m, 2H), 1.99-1.88 (m, 4H), 1.63-1.50 (m, 4H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 178.7, 166.7, 138.9, 132.5, 131.6, 131.0, 129.7, 128.8, 127.2, 120.6, 51.8, 40.4, 24.2, 21.9 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{19}\text{NO}_4\text{Na}$  336.1206, found 336.1206 (0 ppm).



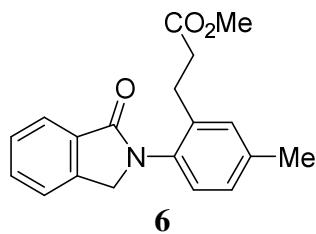
**(E)-3-(5-methyl-2-(1-oxoisoindolin-2-yl)phenyl)acrylic acid (3):** White solid, yield = 99%, 58.7 mg. Mp > 260 °C dec.  $^1\text{H}$  NMR (400 MHz, acetone- $d_6$ ):  $\delta$  = 7.81 (d,  $J$  = 7.6 Hz, 1H), 7.76 (s, 1H), 7.70-7.55 (m, 4H), 7.42-7.35 (m, 2H), 6.51 (d,  $J$  = 16.0 Hz, 1H), 4.90 (s, 2H), 2.44 (s, 3H) ppm.  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  = 7.80-7.77 (m, 2H), 7.73-7.67 (m, 2H), 7.58 (dd,  $J$  = 7.6, 7.6 Hz, 1H), 7.42-7.33 (m, 3H), 6.51 (d,  $J$  = 16.0 Hz, 1H), 4.88 (s, 2H), 2.39 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  = 167.7, 167.1, 142.3, 139.3, 137.7, 135.4, 132.0, 131.7, 131.5, 131.3, 128.2, 127.9, 127.4, 123.6, 123.4, 120.6, 53.2, 20.6 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{15}\text{NO}_3\text{Na}$  316.0944, found 316.0947 (1 ppm).



**(E)-3-(5-methyl-2-(1-oxoisoindolin-2-yl)phenyl)acrylamide (4):** White solid, yield = 63%, 36.9 mg. Mp: 171-173 °C.  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  = 7.79 (d,  $J$  = 7.6 Hz, 1H), 7.72-7.66 (m, 2H), 7.60-7.56 (m, 2H), 7.52 (s, 1H), 7.38 (d,  $J$  = 8.0 Hz, 1H), 7.31 (d,  $J$  = 7.6 Hz, 1H), 7.23 (d,  $J$  = 16.0 Hz, 1H), 7.11 (s, 1H), 6.62 (d,  $J$  = 16.0 Hz, 1H), 4.83 (s, 2H), 2.39 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  = 167.1, 166.5, 142.3, 137.6, 135.2, 134.6, 132.6, 132.0, 131.5, 130.8, 128.3, 128.2, 126.7, 123.8, 123.6, 123.3, 53.3, 20.7 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2\text{Na}$  315.1104, found 315.1107 (1 ppm).



**(E)-N-(2-hydroxyethyl)-3-(5-methyl-2-(1-oxoisoindolin-2-yl)phenyl)acrylamide (5):** White solid, yield = 77%, 54.6 mg. Mp: 245-247 °C.  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  = 8.12 (t,  $J$  = 6.0 Hz, 1H), 7.79 (d,  $J$  = 7.6 Hz, 1H), 7.72-7.66 (m, 2H), 7.60-7.55 (m, 2H), 7.38 (d,  $J$  = 8.0 Hz, 1H), 7.31 (d,  $J$  = 8.0 Hz, 1H), 7.23 (d,  $J$  = 15.6 Hz, 1H), 6.68 (d,  $J$  = 15.6 Hz, 1H), 4.83 (s, 2H), 4.71 (t,  $J$  = 5.2 Hz, 1H), 3.42 (td,  $J$  = 6.0, 5.2 Hz, 2H), 3.19 (td,  $J$  = 5.6, 6.0 Hz, 2H), 2.39 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  = 167.1, 164.8, 142.2, 137.6, 135.1, 134.0, 132.7, 132.0, 131.5, 130.8, 128.24, 128.22, 126.7, 123.8, 123.6, 123.3, 59.8, 53.2, 41.6, 20.8 ppm. HRMS (ESI) calcd. for  $[\text{M} + \text{Na}]^+$   $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_3\text{Na}$  359.1366, found 359.1367 (0 ppm).



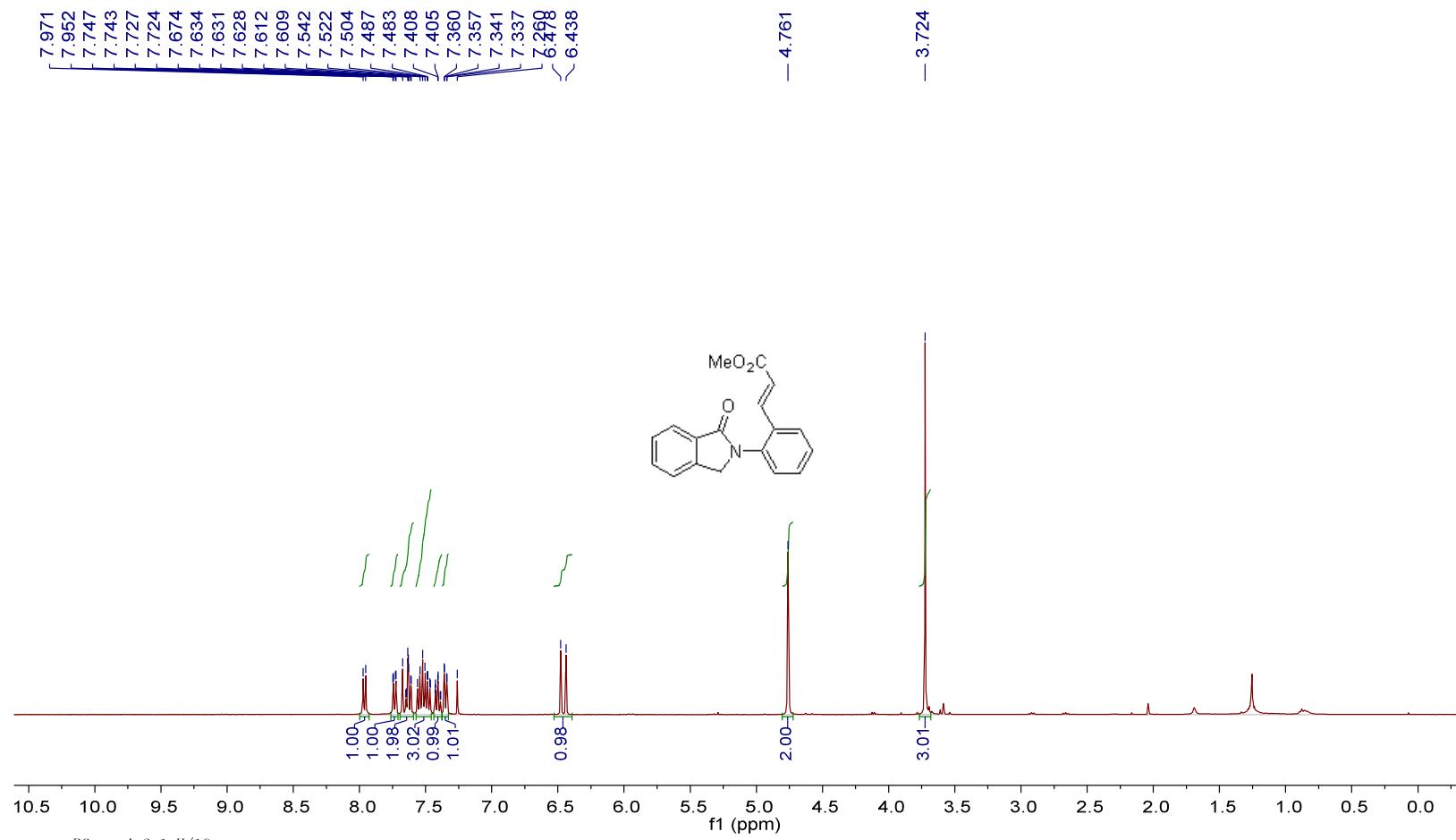
**Methyl 3-(5-methyl-2-(1-oxoisoindolin-2-yl)phenyl)propanoate (6):** Colorless oil, yield = 99%, 61.9 mg.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.93 (d,  $J$  = 7.2 Hz, 1H), 7.59 (ddd,  $J$  = 7.2, 7.2, 1.2 Hz, 1H), 7.51 (dd,  $J$  = 7.6, 7.6 Hz, 2H), 7.15 (s, 1H), 7.11 (d,  $J$  = 1.2 Hz, 2H), 4.73 (s, 2H), 3.58 (s, 3H), 2.87 (t,  $J$  = 8.0 Hz, 2H), 2.65 (t,  $J$  = 8.0 Hz, 2H), 2.36 (s, 3H) ppm.  $^{13}\text{C}\{\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 173.4, 168.3, 141.7, 138.7,

138.5, 134.2, 132.4, 131.7, 130.4, 128.3, 127.9, 124.3, 122.9, 53.8, 51.6, 34.3, 26.4, 21.3 ppm. HRMS (ESI) calcd. for  $[M + Na]^+$   $C_{19}H_{19}NO_3Na$  332.1257, found 332.1257 (0 ppm).

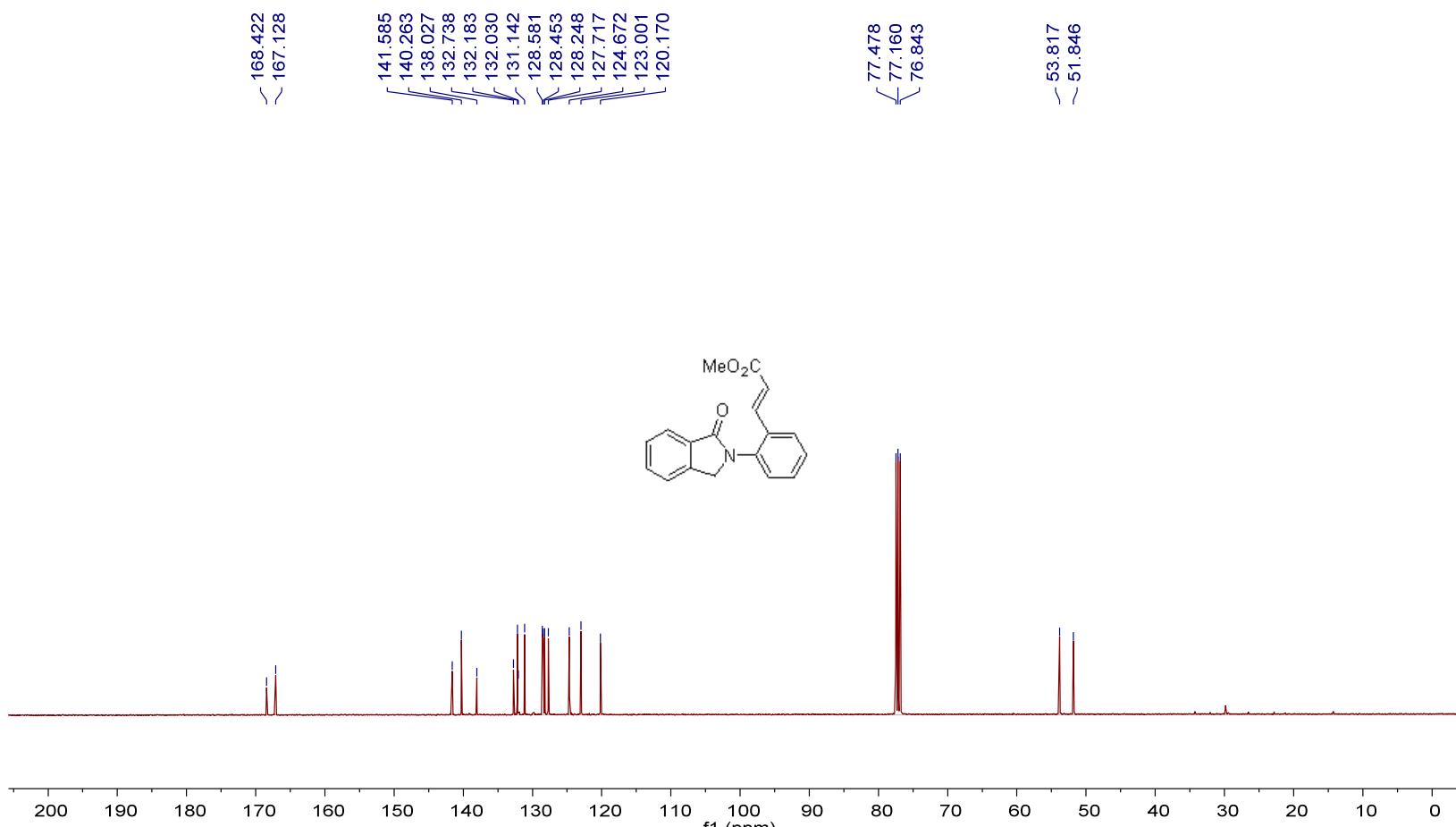
## 9. References.

- [1] Y. Zhou, P. Chen, X. Lv, J. Niu, Y. Wang, M. Lei, L. Hu, *Tetrahedron Letters*, **2017**, *58*, 2232-2235.
- [2] A. Verma, S. Patel, Meenakshi, A. Kumar, A. Yadav, S. Kumar, S. Jana, S. Sharma, Ch. D. Prasad, S. Kumar, *Chem. Commun.*, **2015**, *51*, 1371-1374.
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- [5] C. Lin, L. Zhen, Y. Cheng, H.-J. Du, H. Zhao, X. Wen, L.-Y. Kong, Q.-L. Xu, H. Sun, *Org. Lett.* **2015**, *17*, 2684-2687.

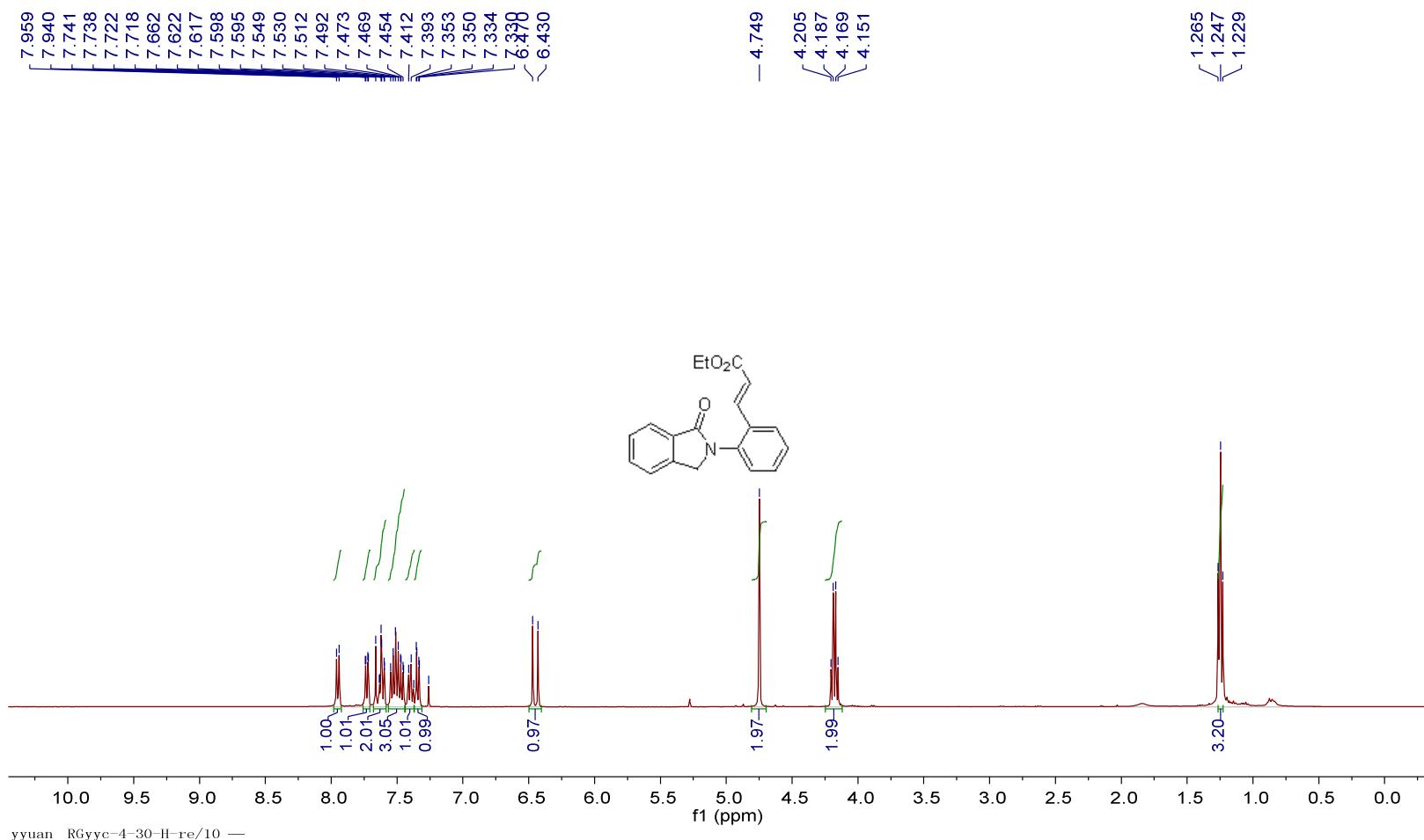
## 10. NMR spectra.



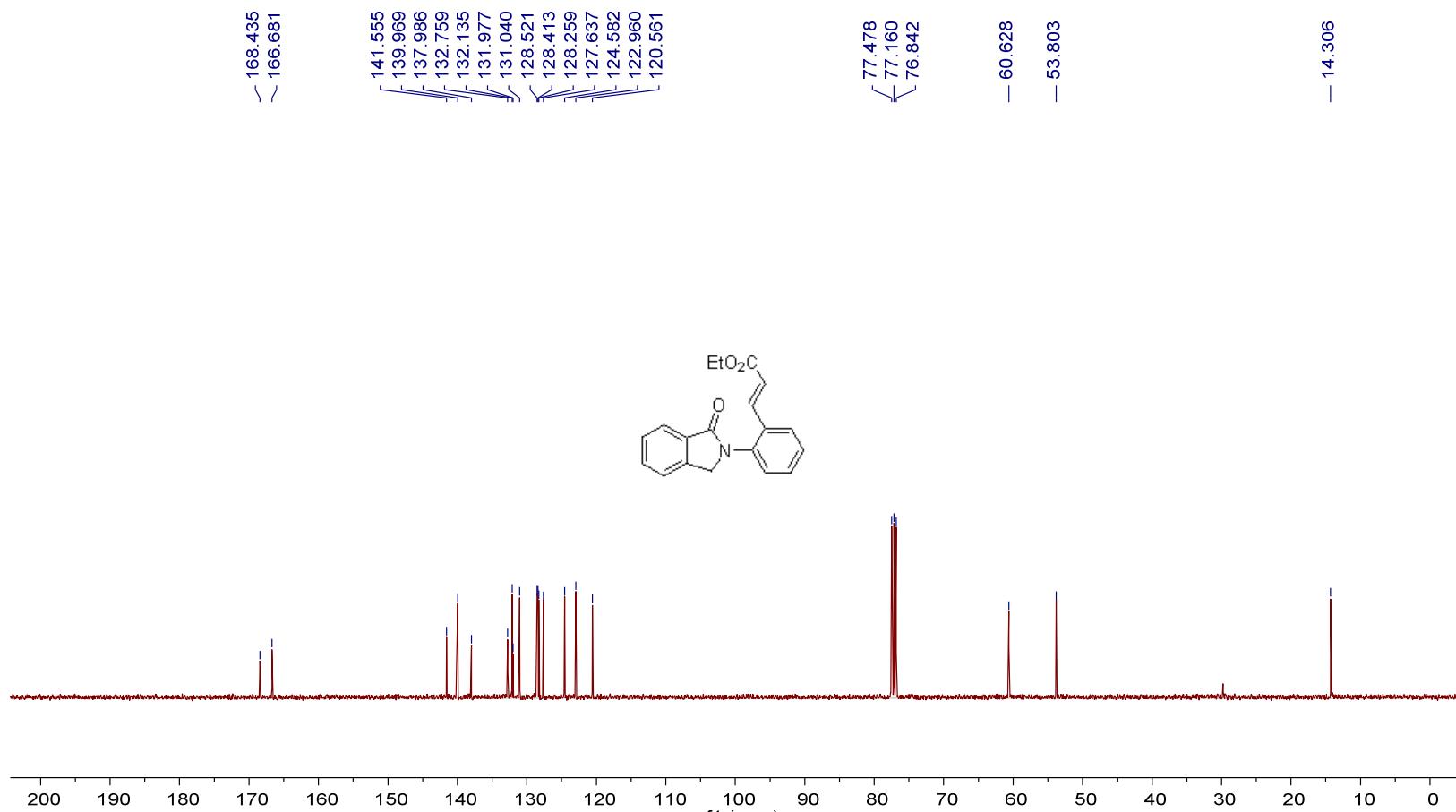
$^1\text{H}$  NMR spectrum of **2a**.



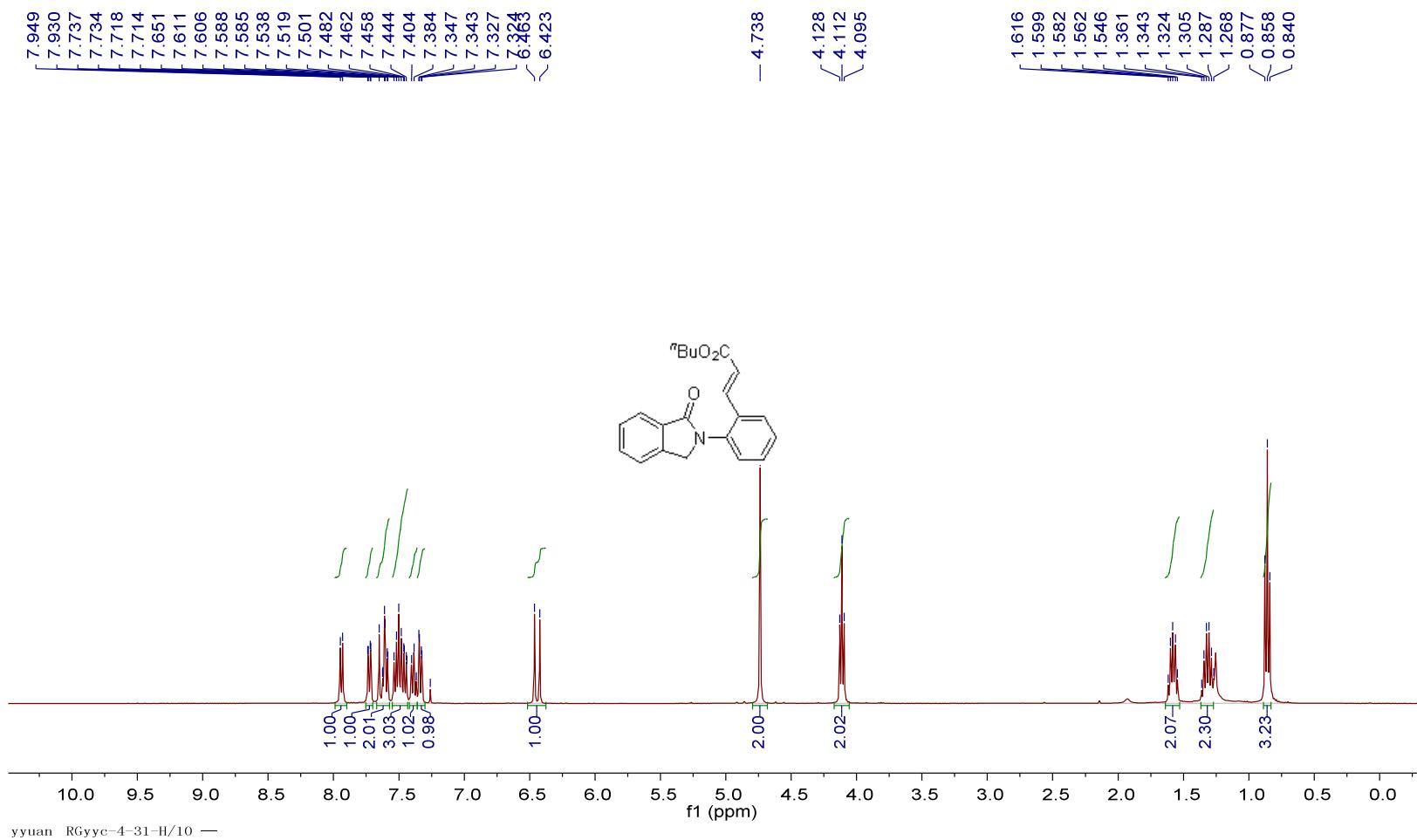
<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2a**.

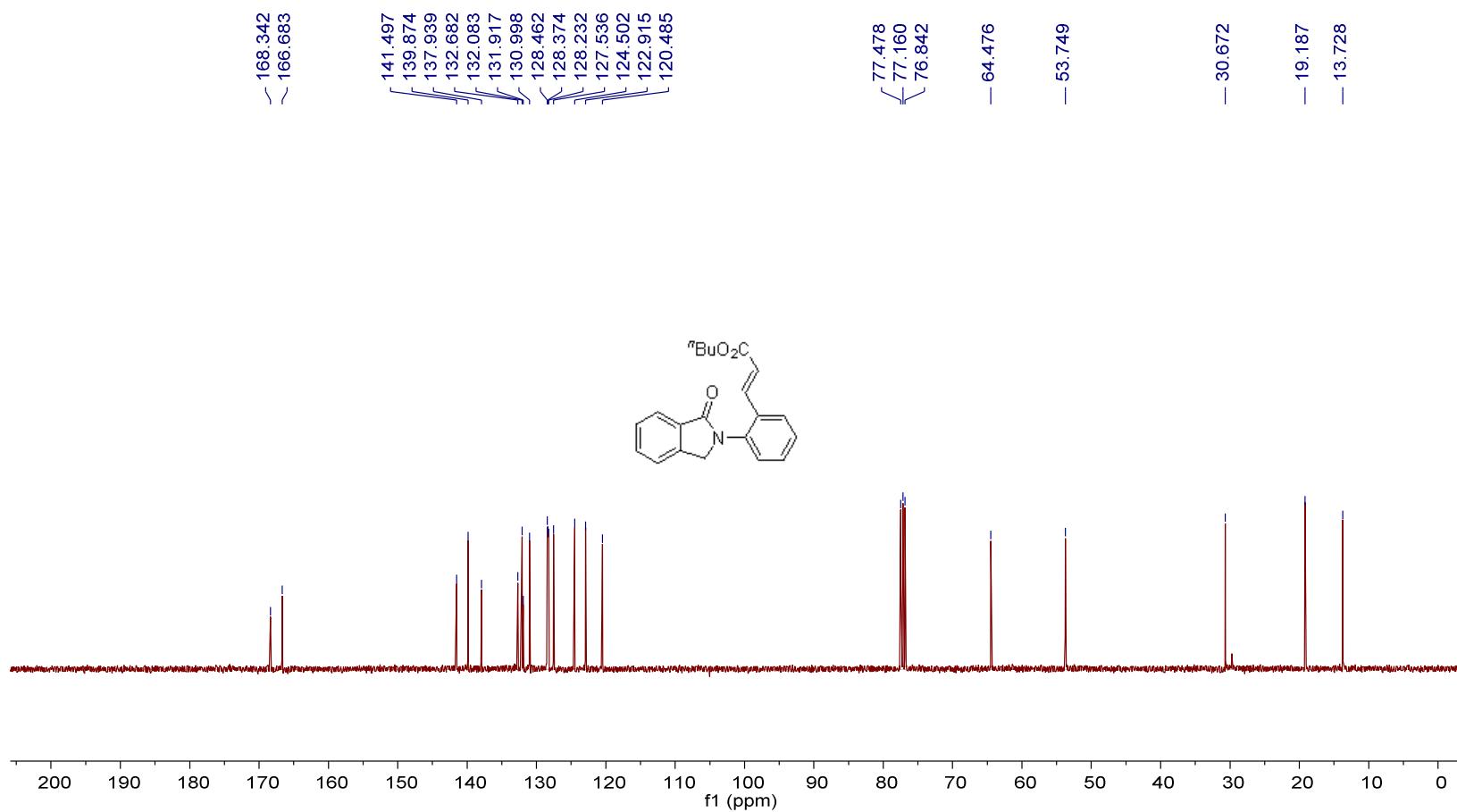


<sup>1</sup>H NMR spectrum of **2b**.

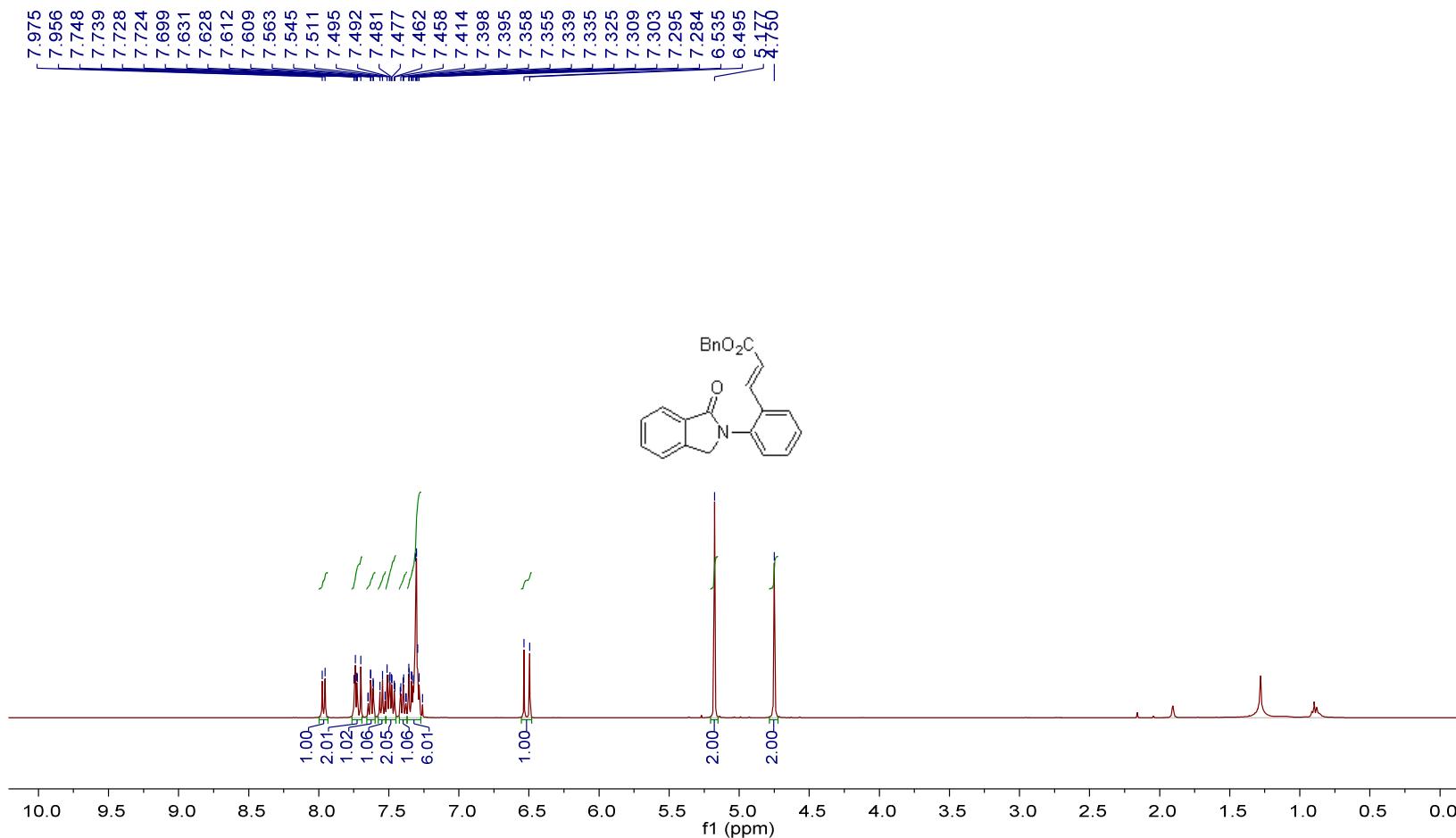


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2b**.

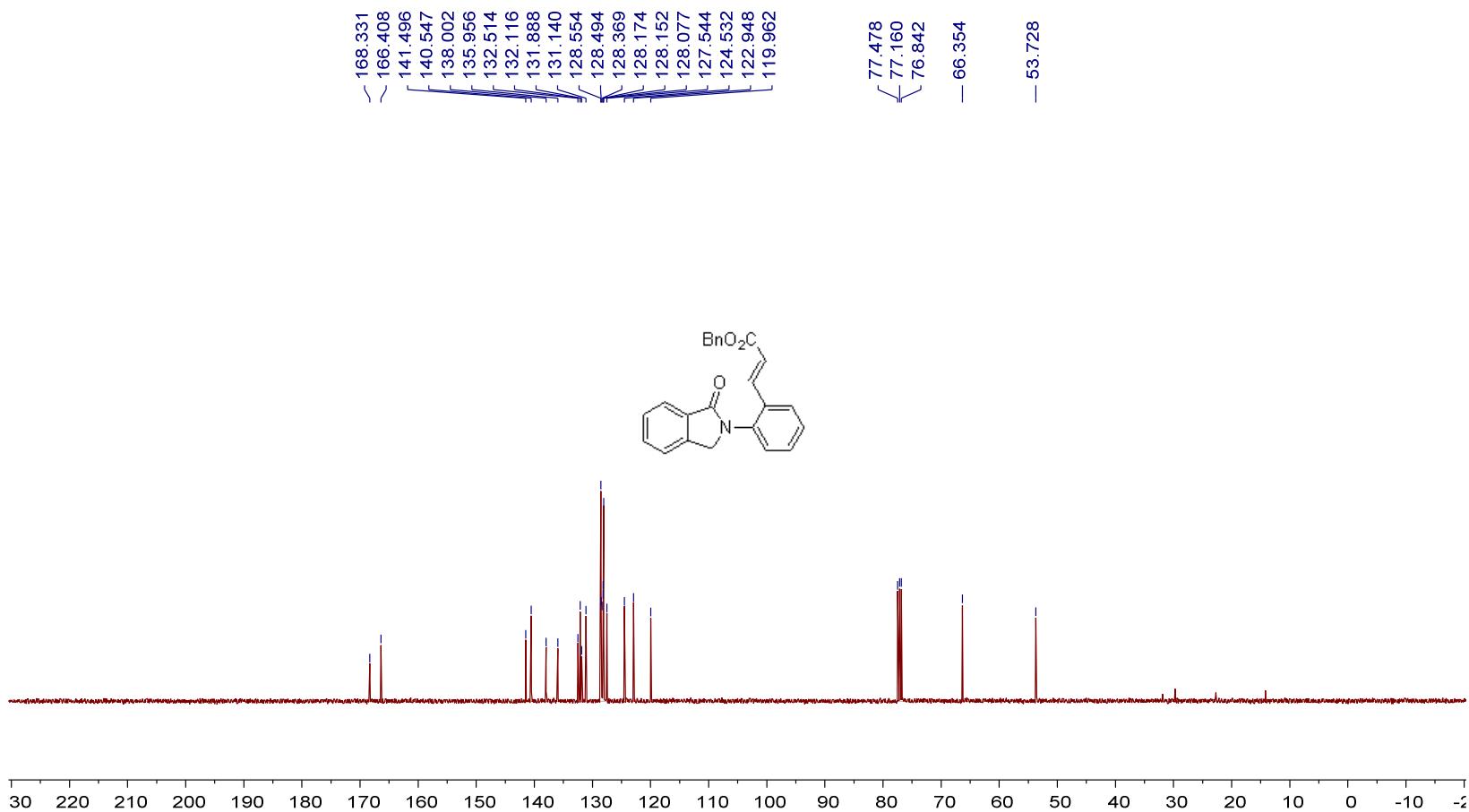




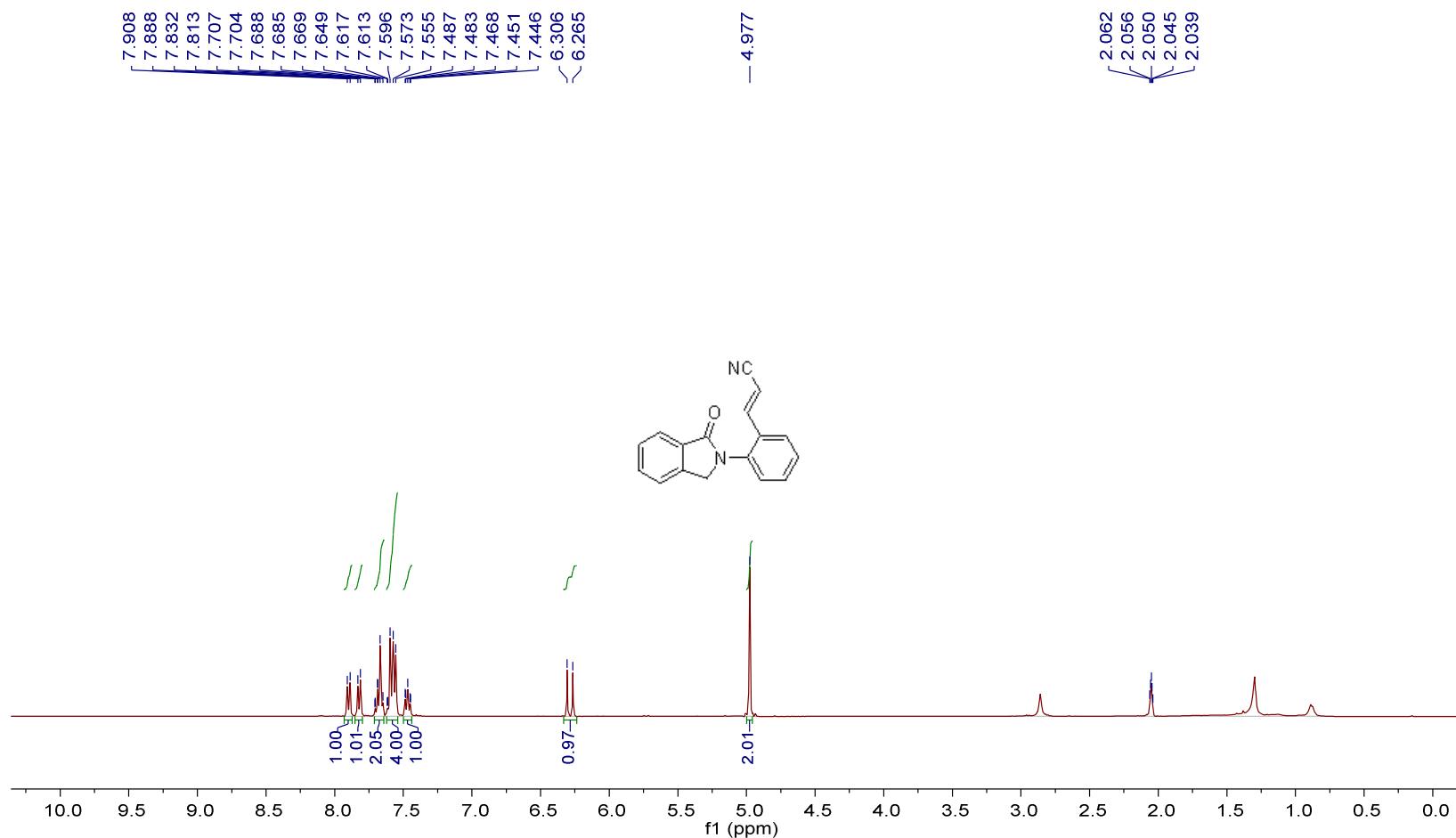
<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2c**.

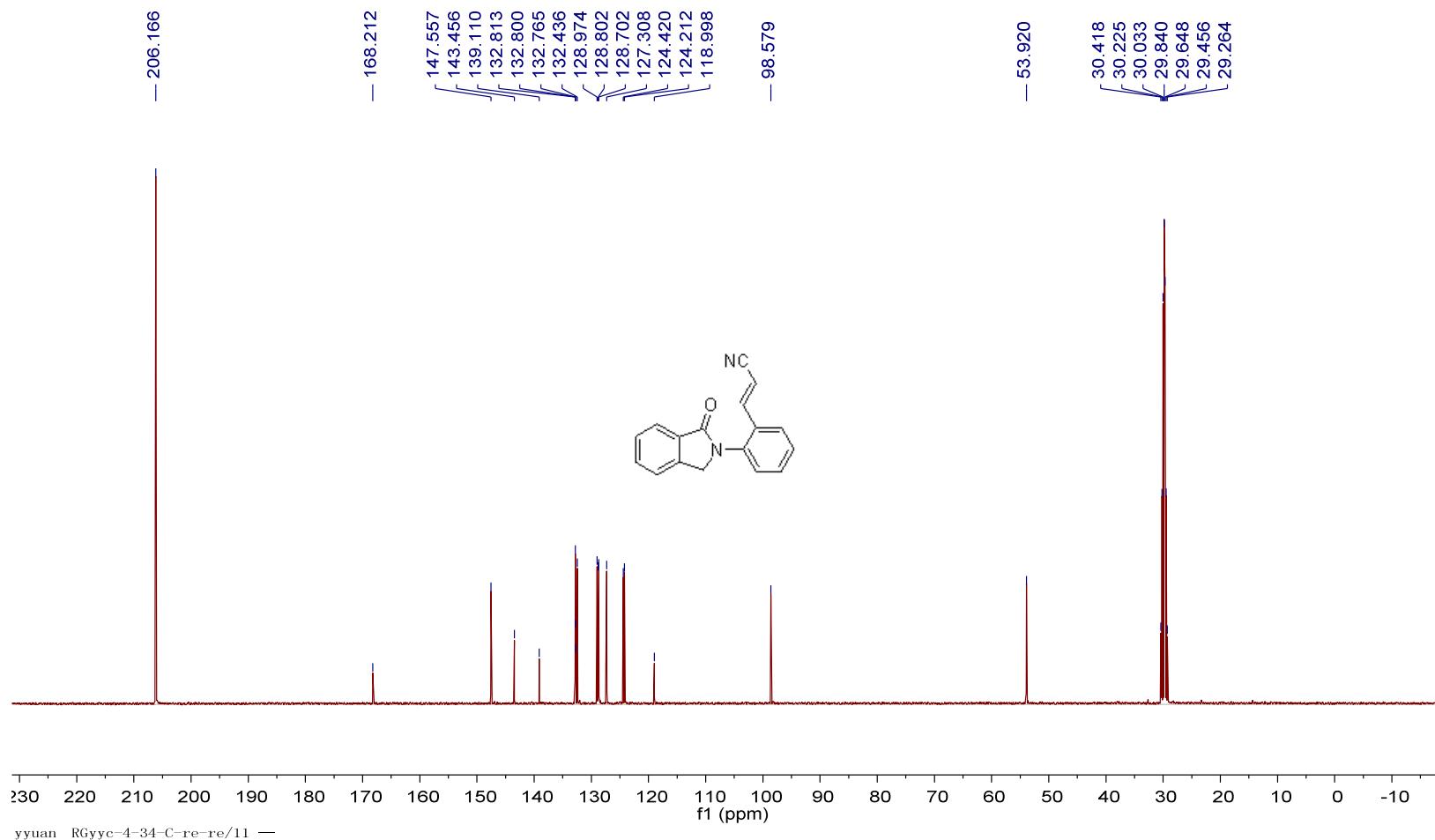


<sup>1</sup>H NMR spectrum of **2d**.

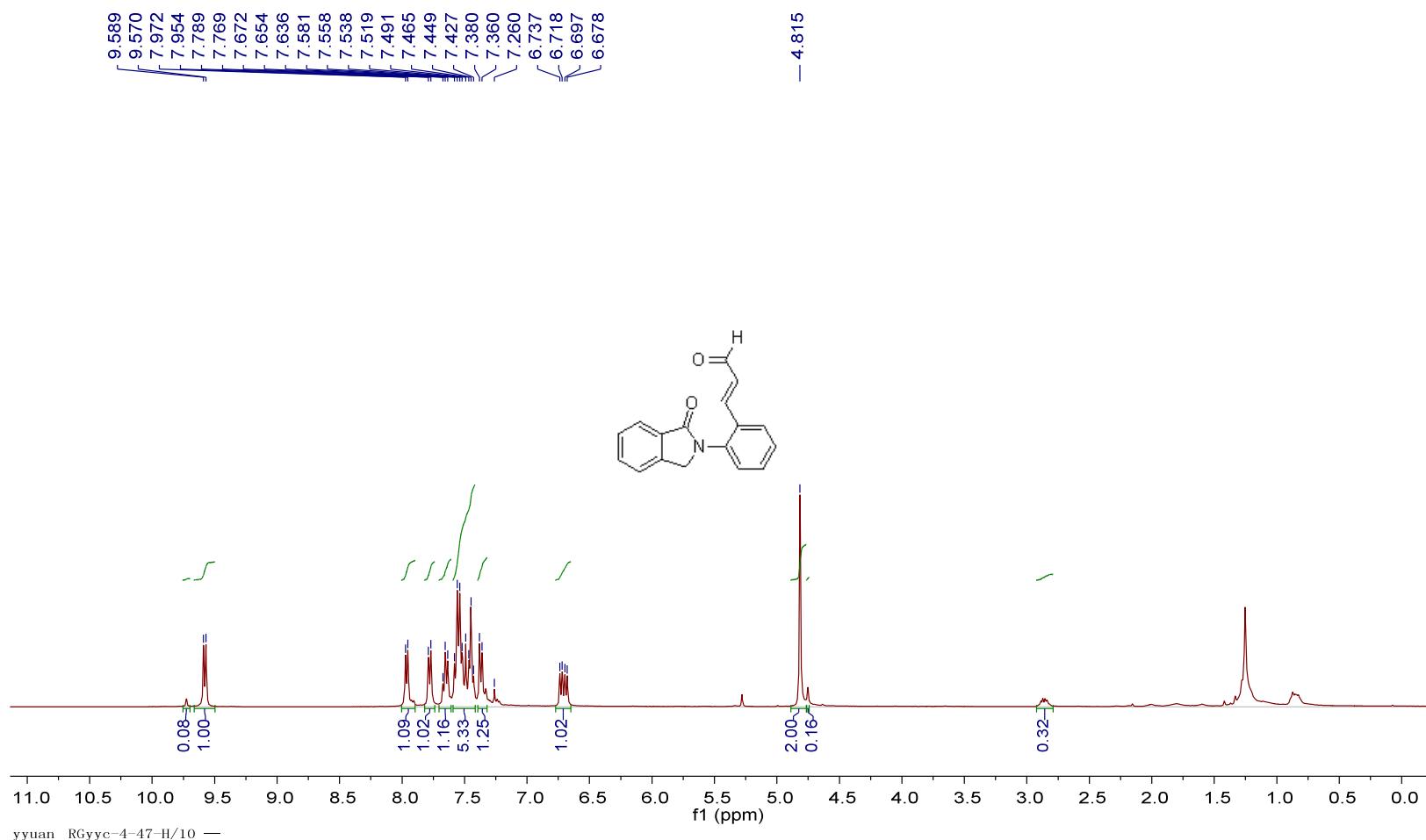


$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2d**.

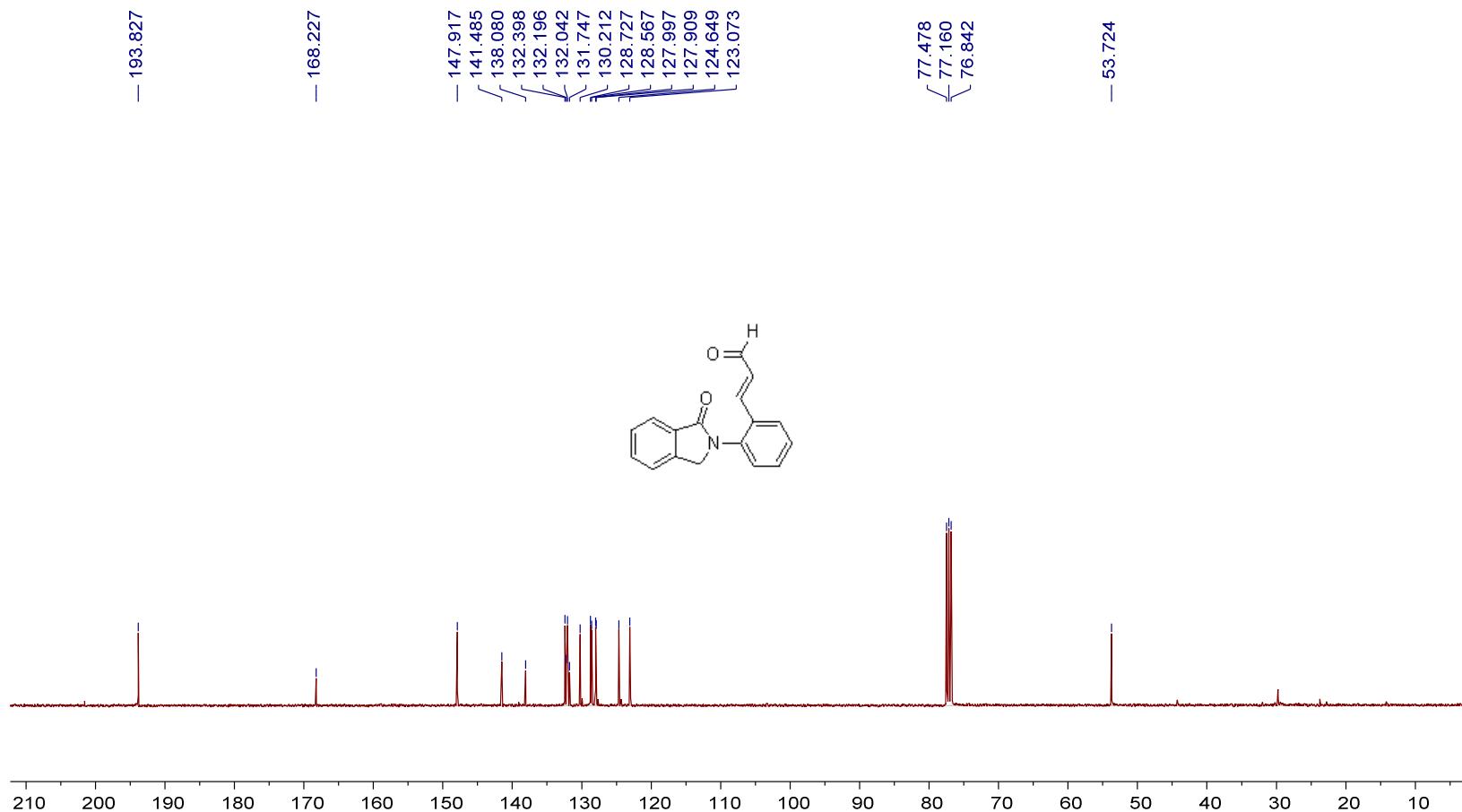




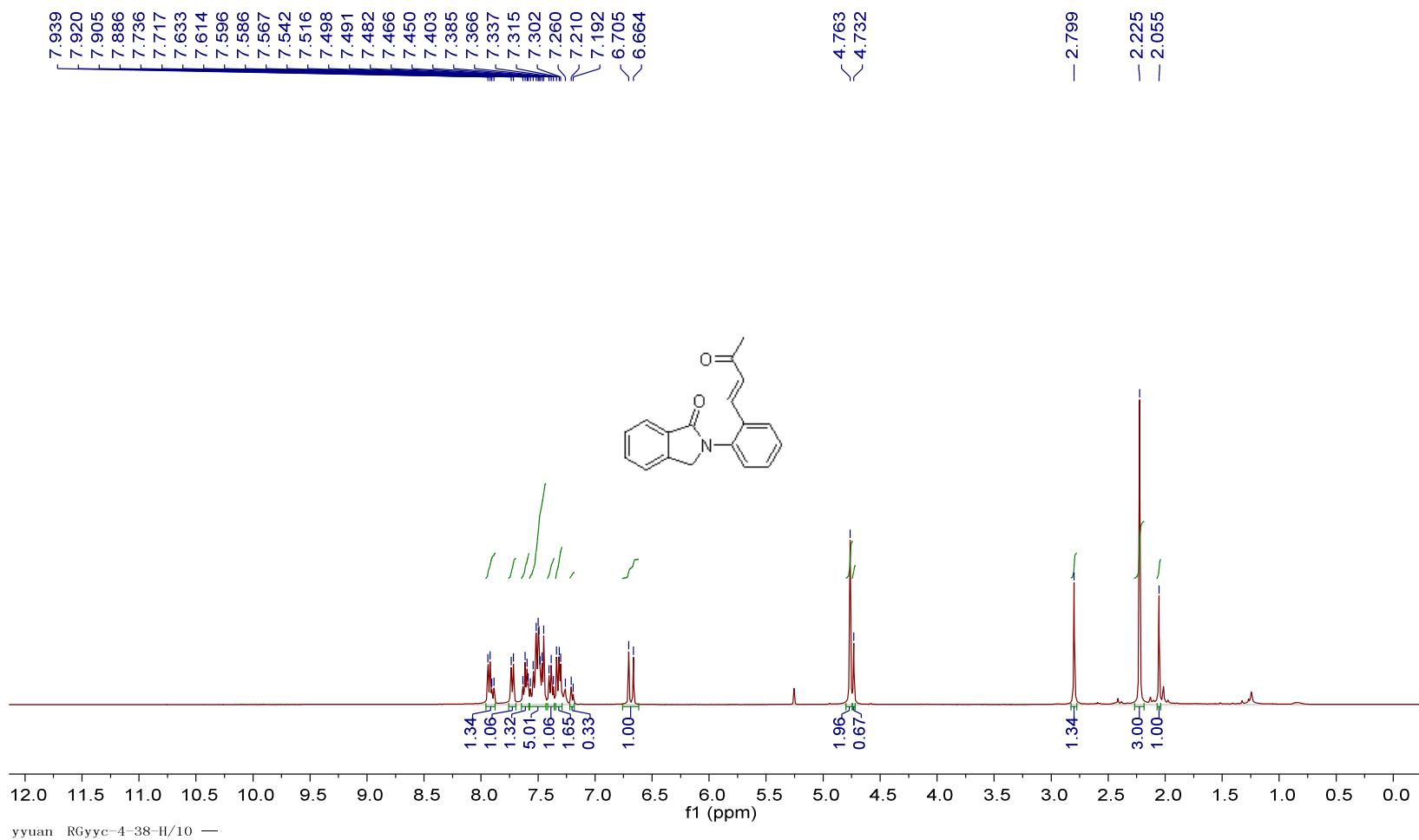
<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2e**.



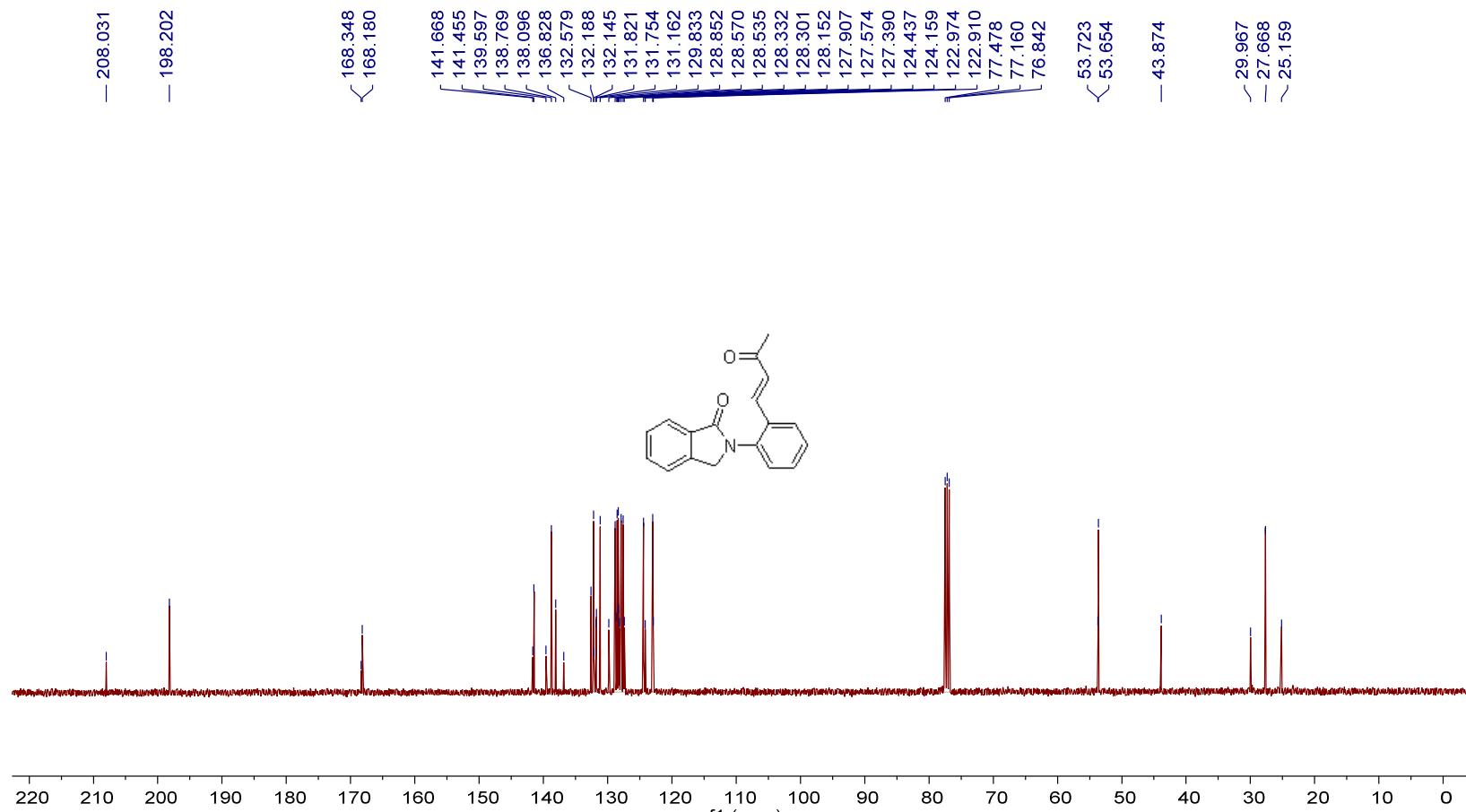
<sup>1</sup>H NMR spectrum of **2f**.



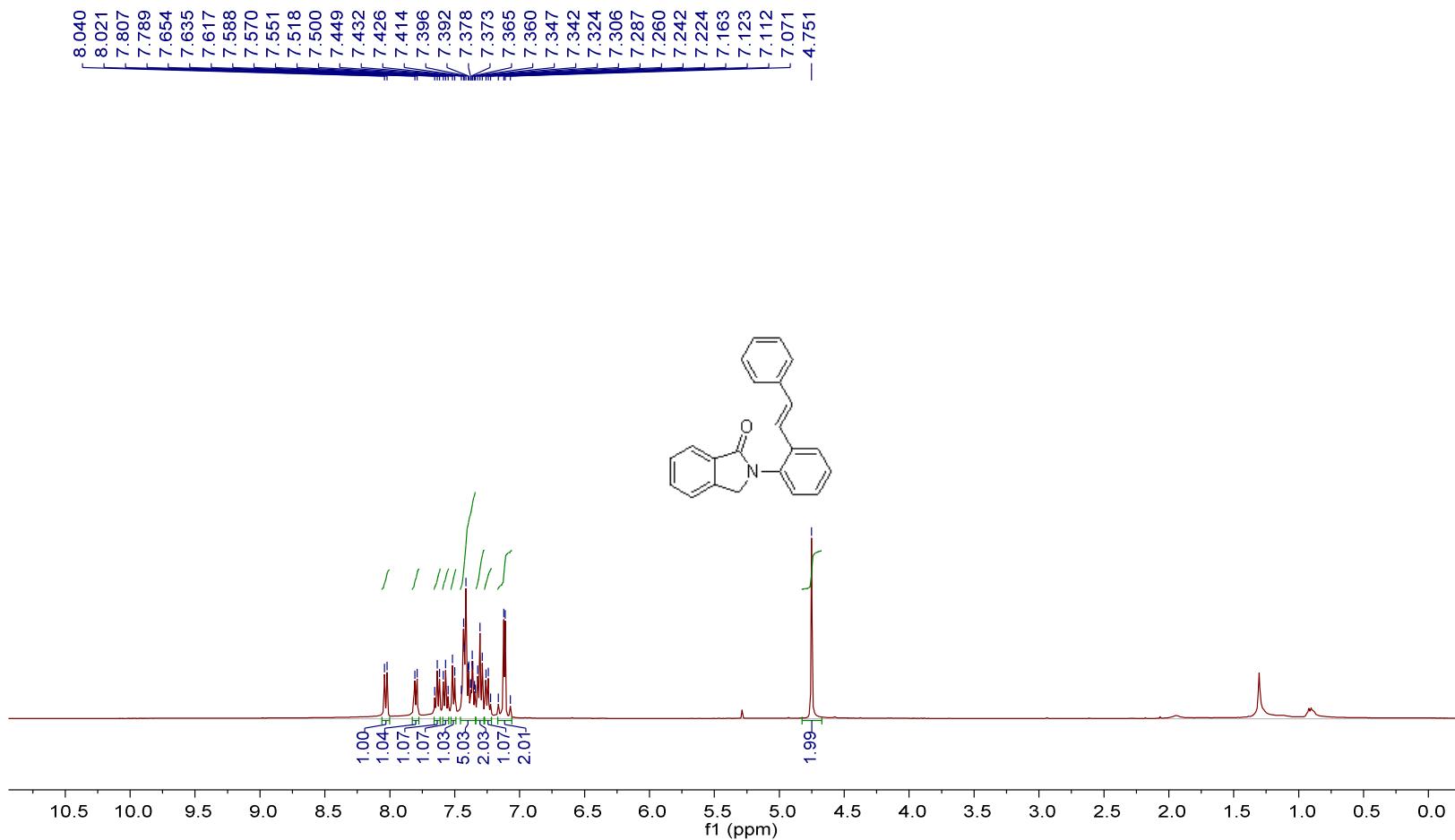
$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2f**.



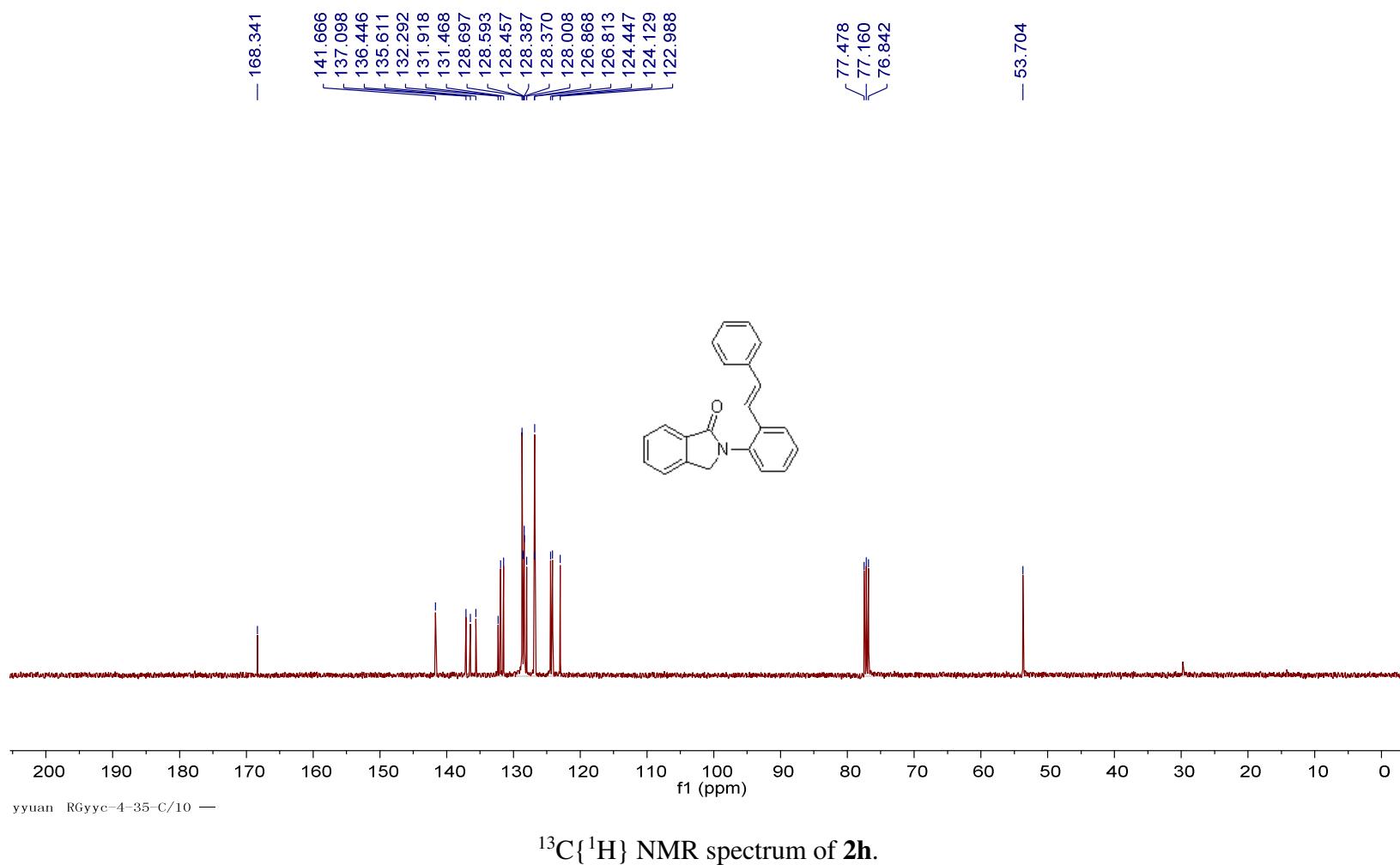
<sup>1</sup>H NMR spectrum of **2g**.

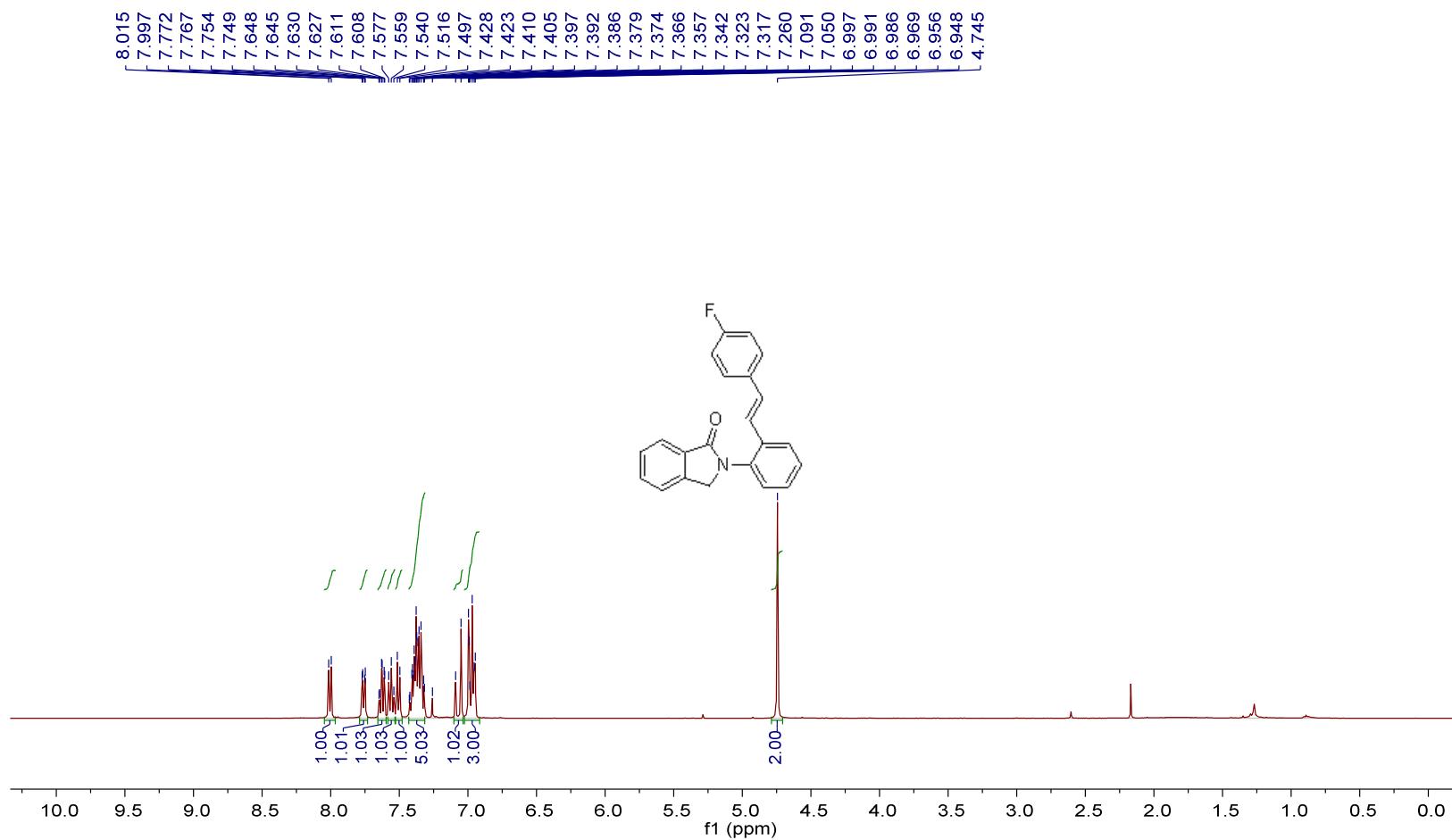


13C{1H} NMR spectrum of **2g**.

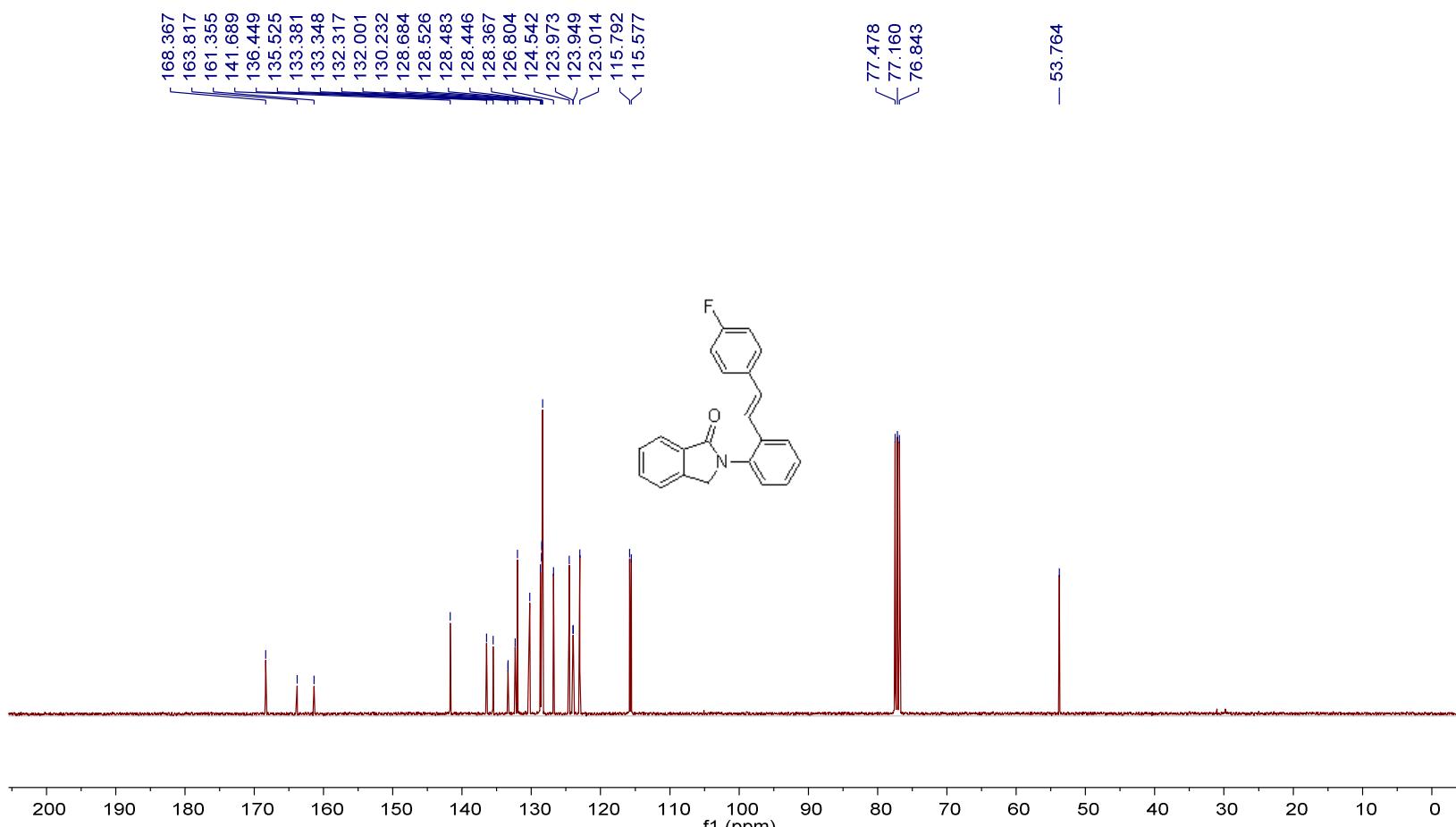


$^1\text{H}$  NMR spectrum of **2h**.



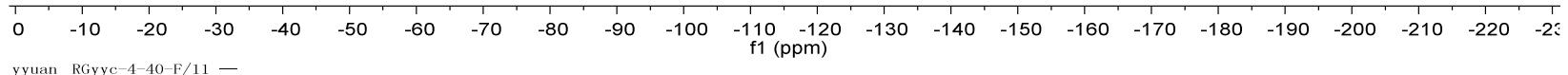
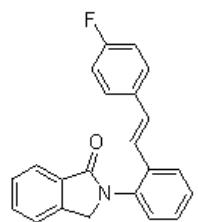


$^1\text{H}$  NMR spectrum of **2i**.

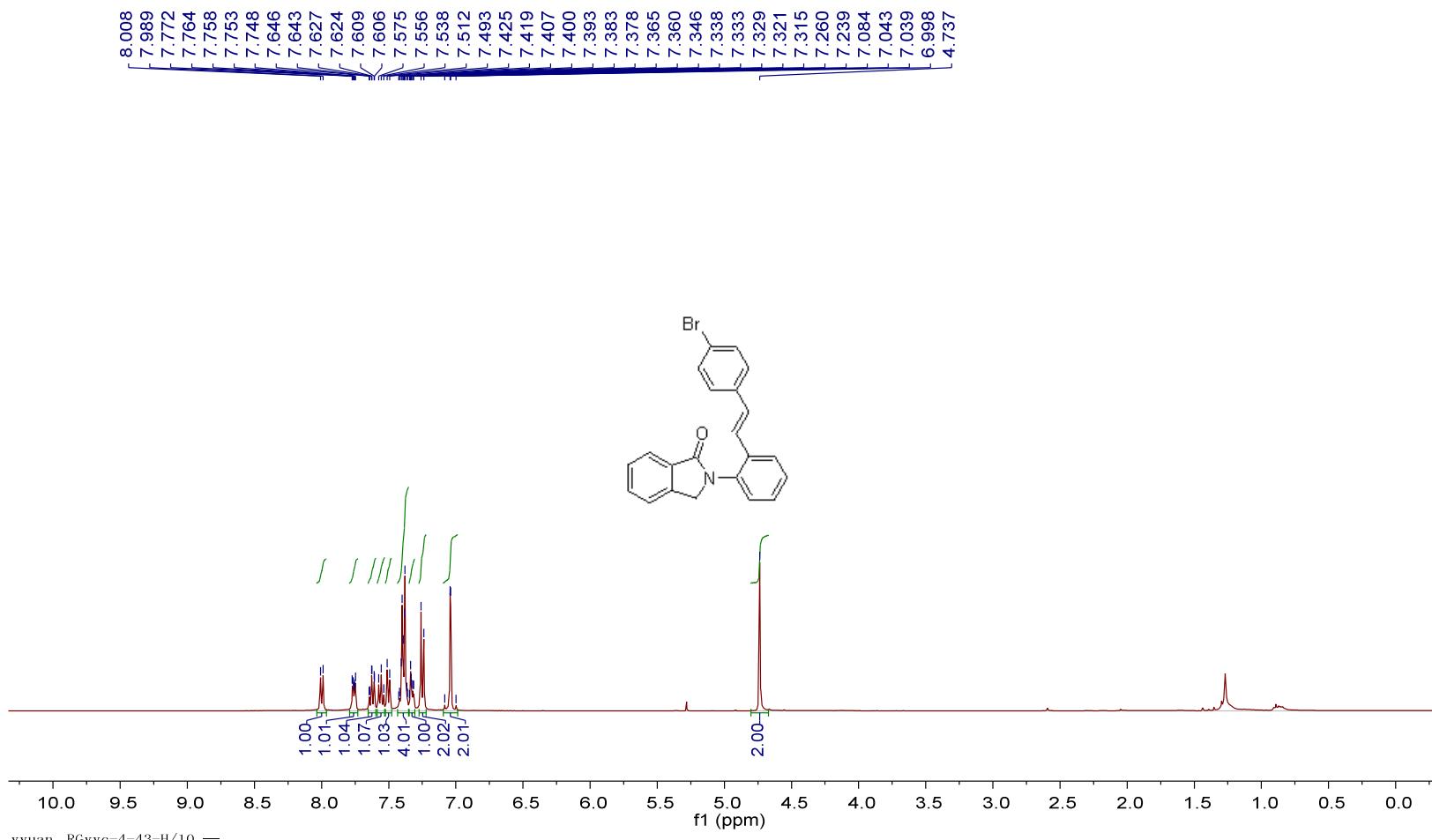


13C{1H} NMR spectrum of **2i**.

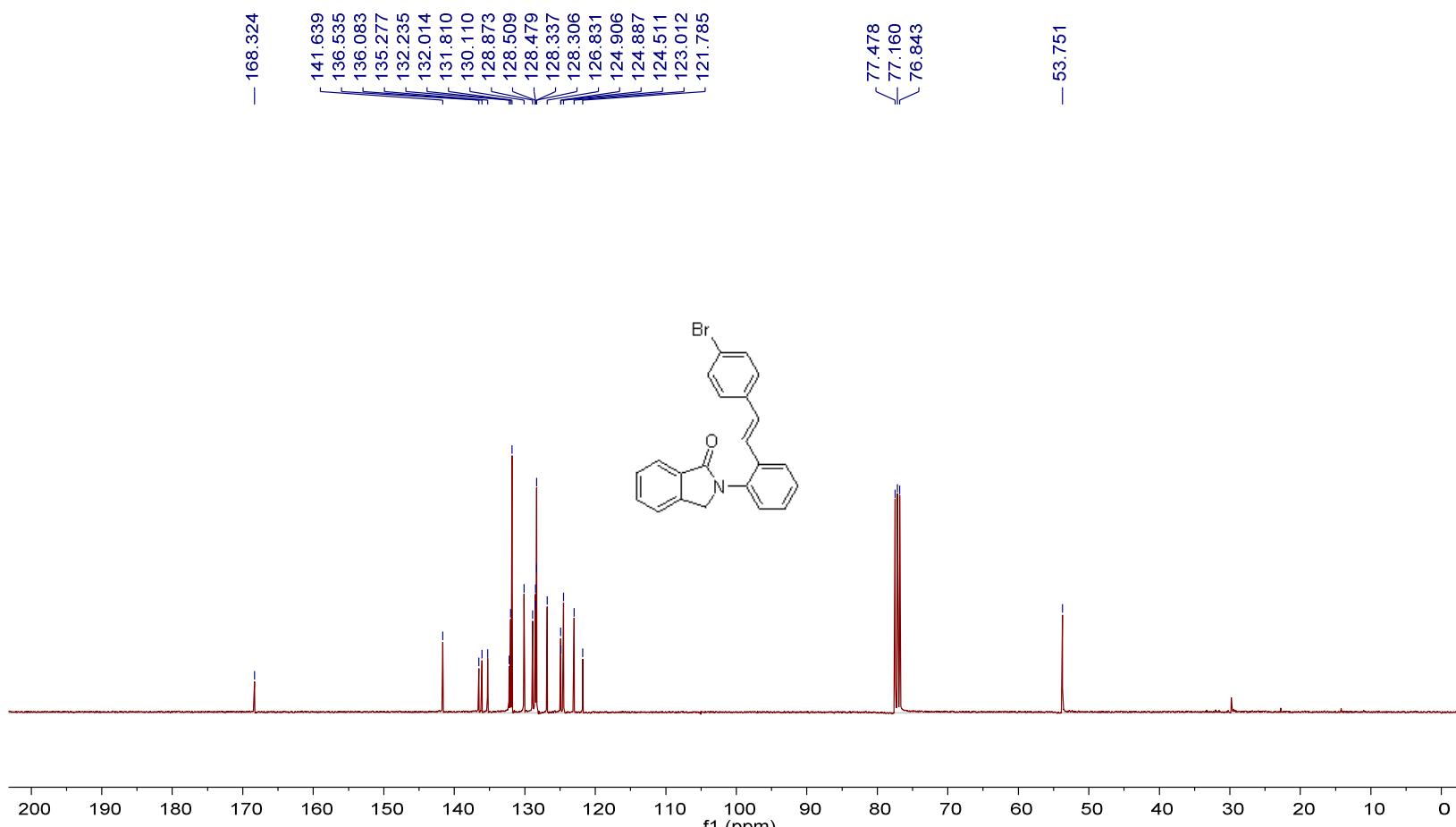
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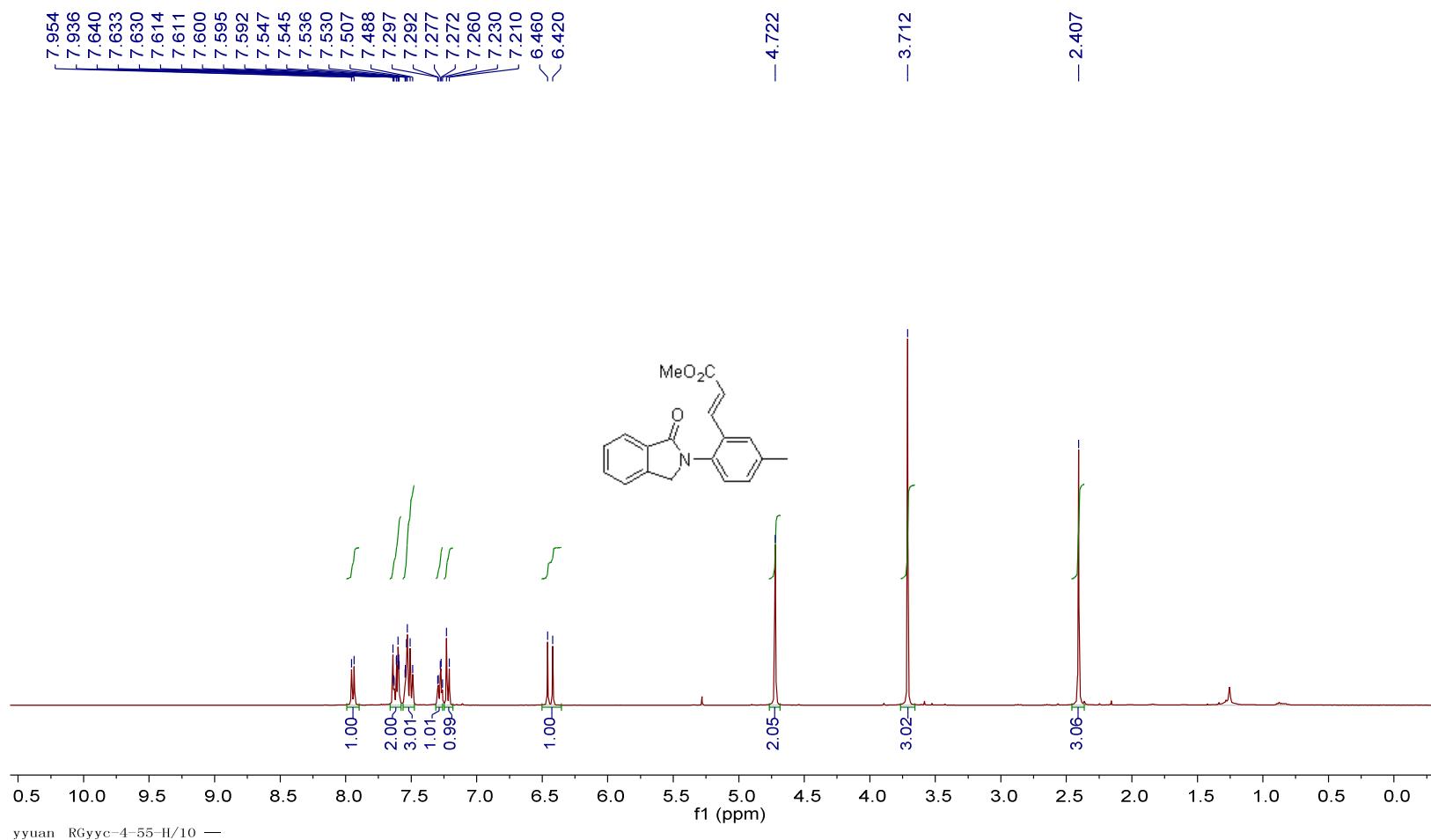


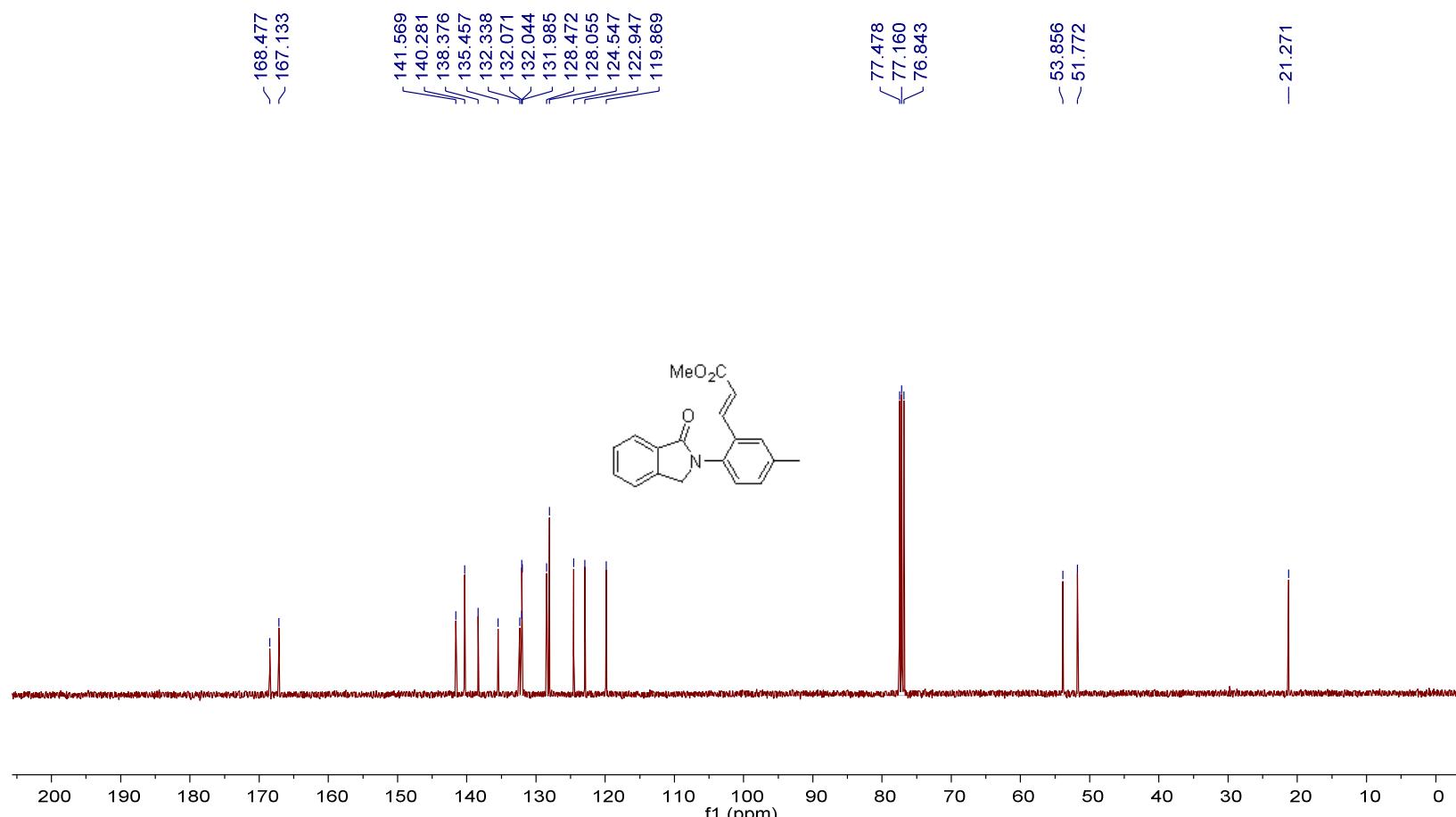
${}^{19}\text{F}\{{}^1\text{H}\}$  NMR spectrum of **2i**.



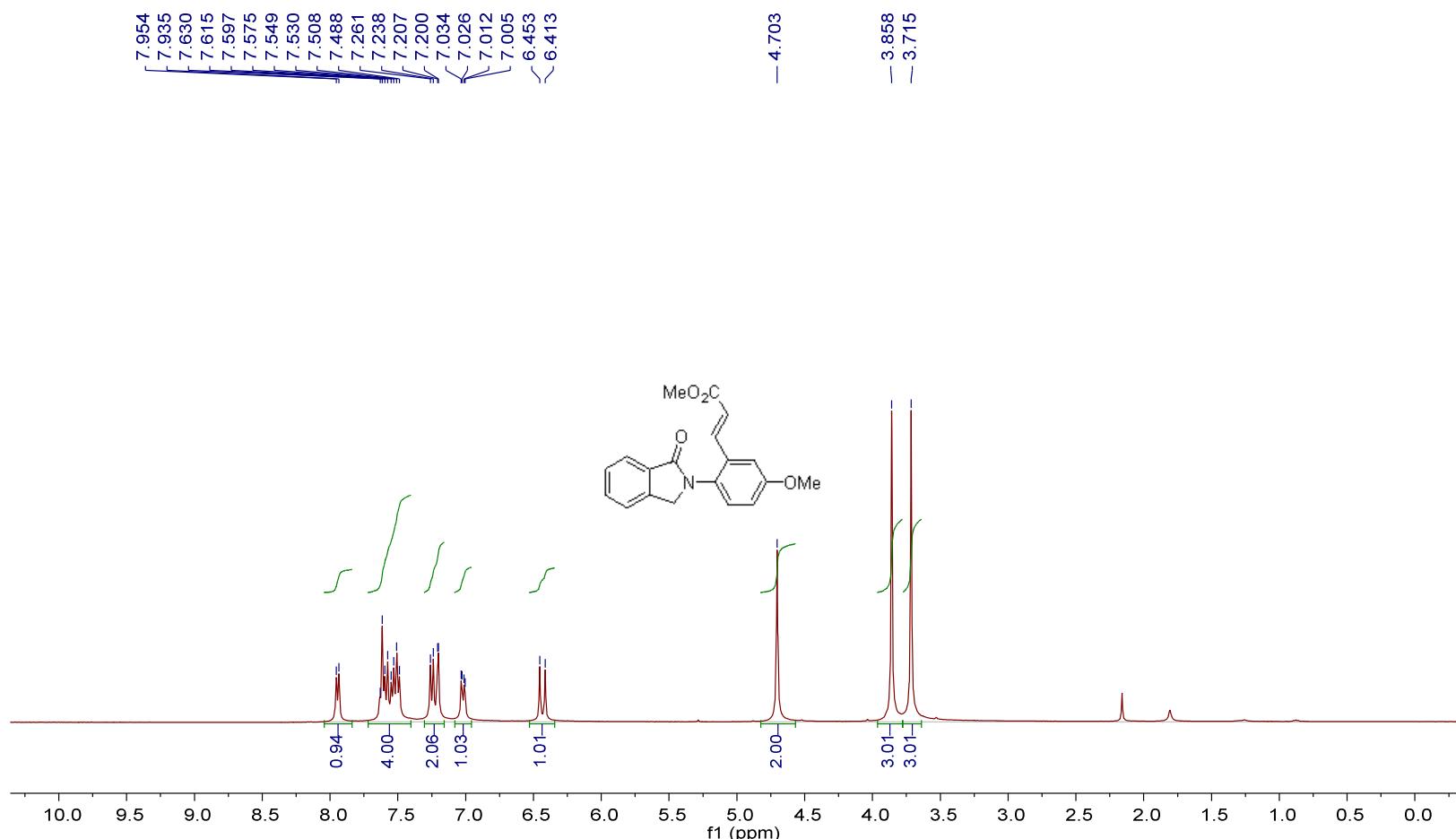
<sup>1</sup>H NMR spectrum of **2j**.



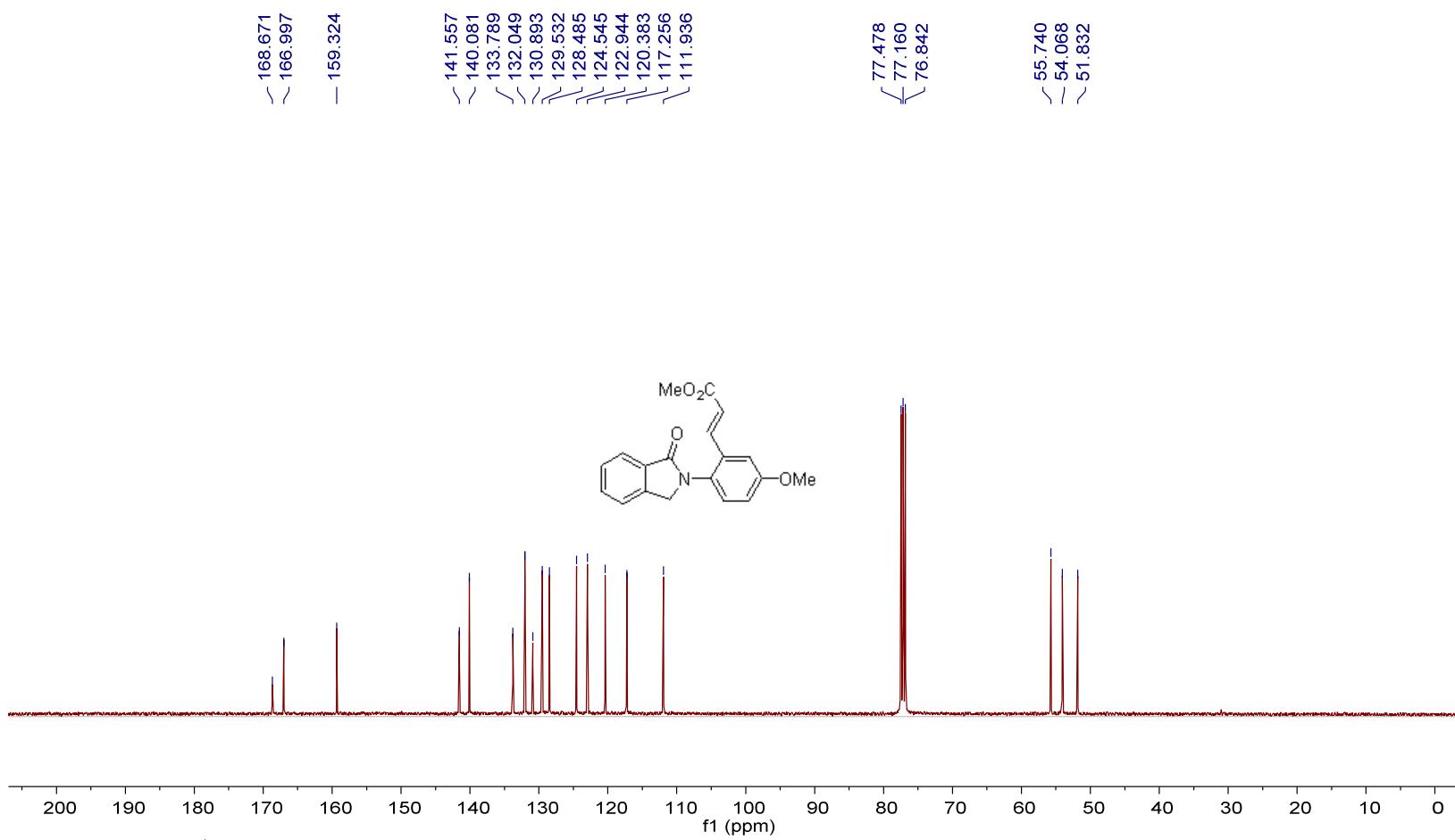


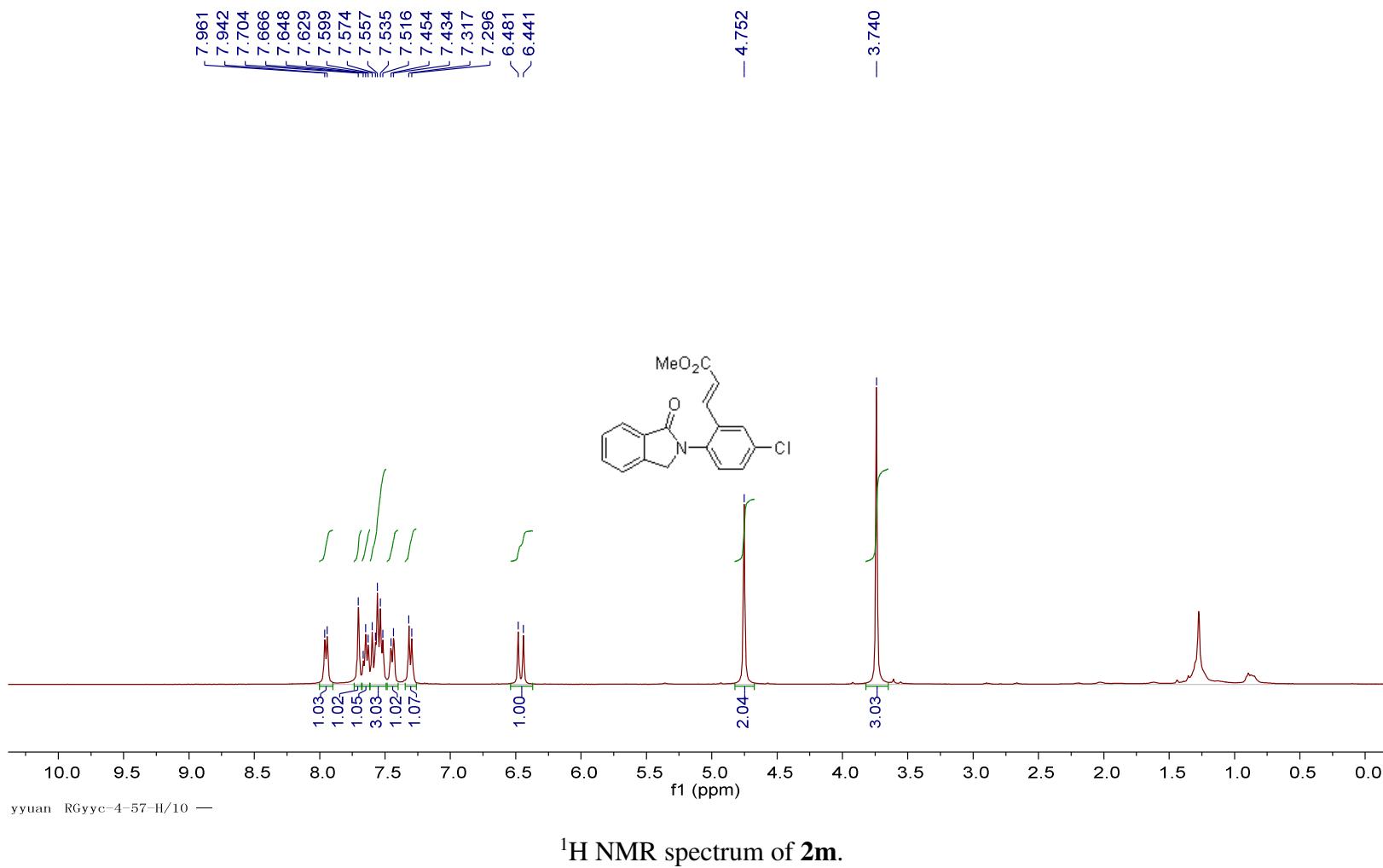


$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2k**.

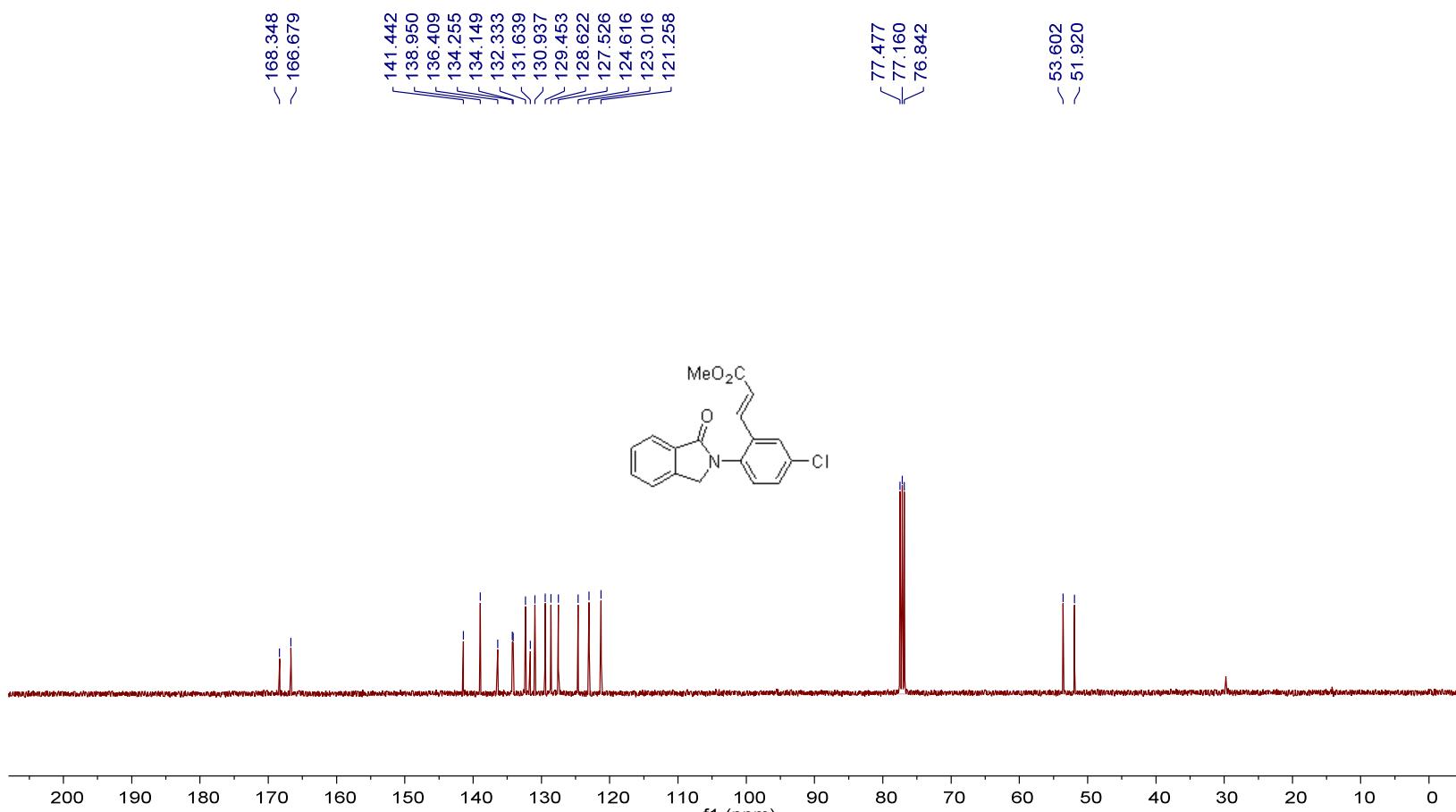


$^1\text{H}$  NMR spectrum of **2l**.

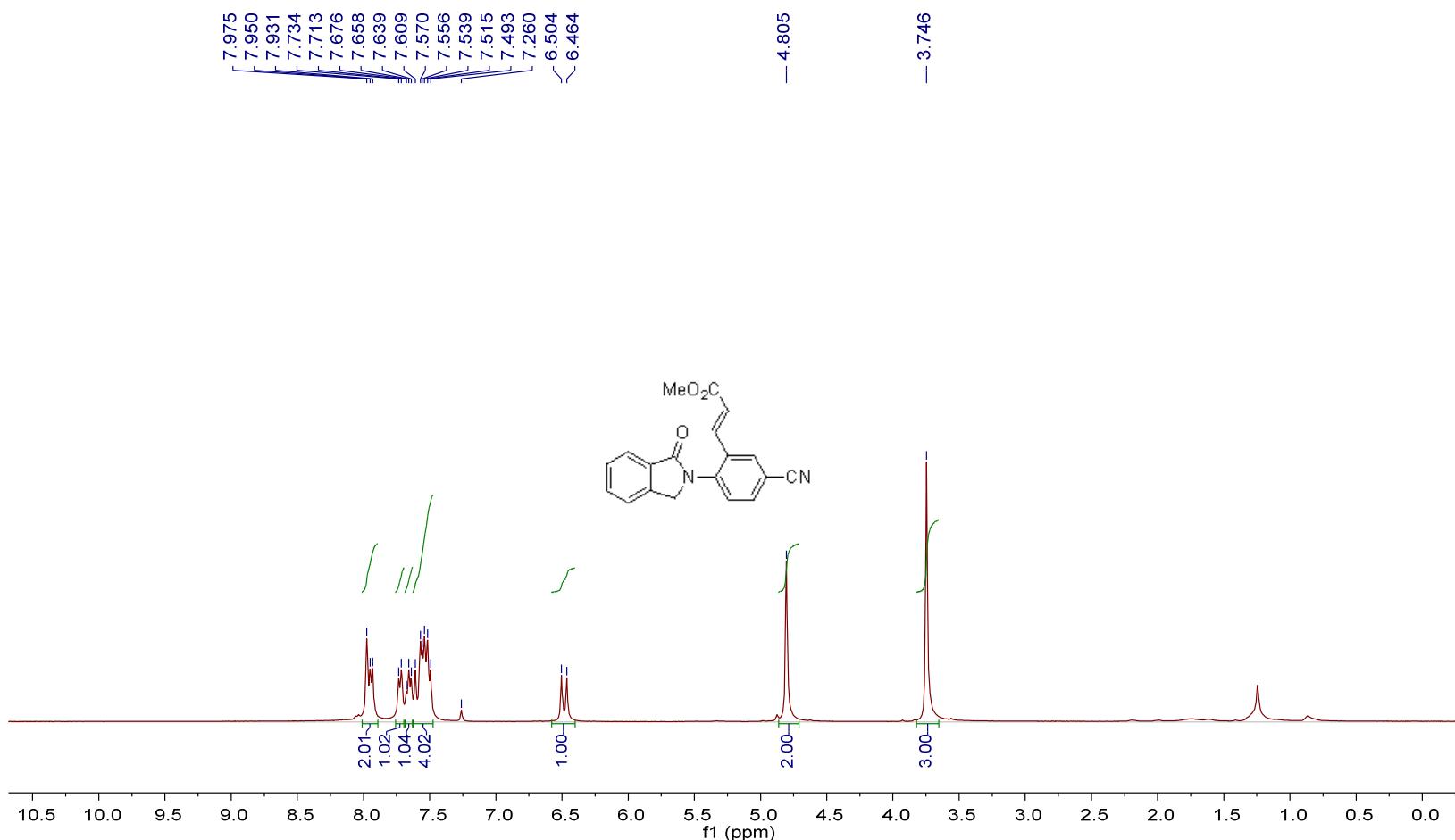




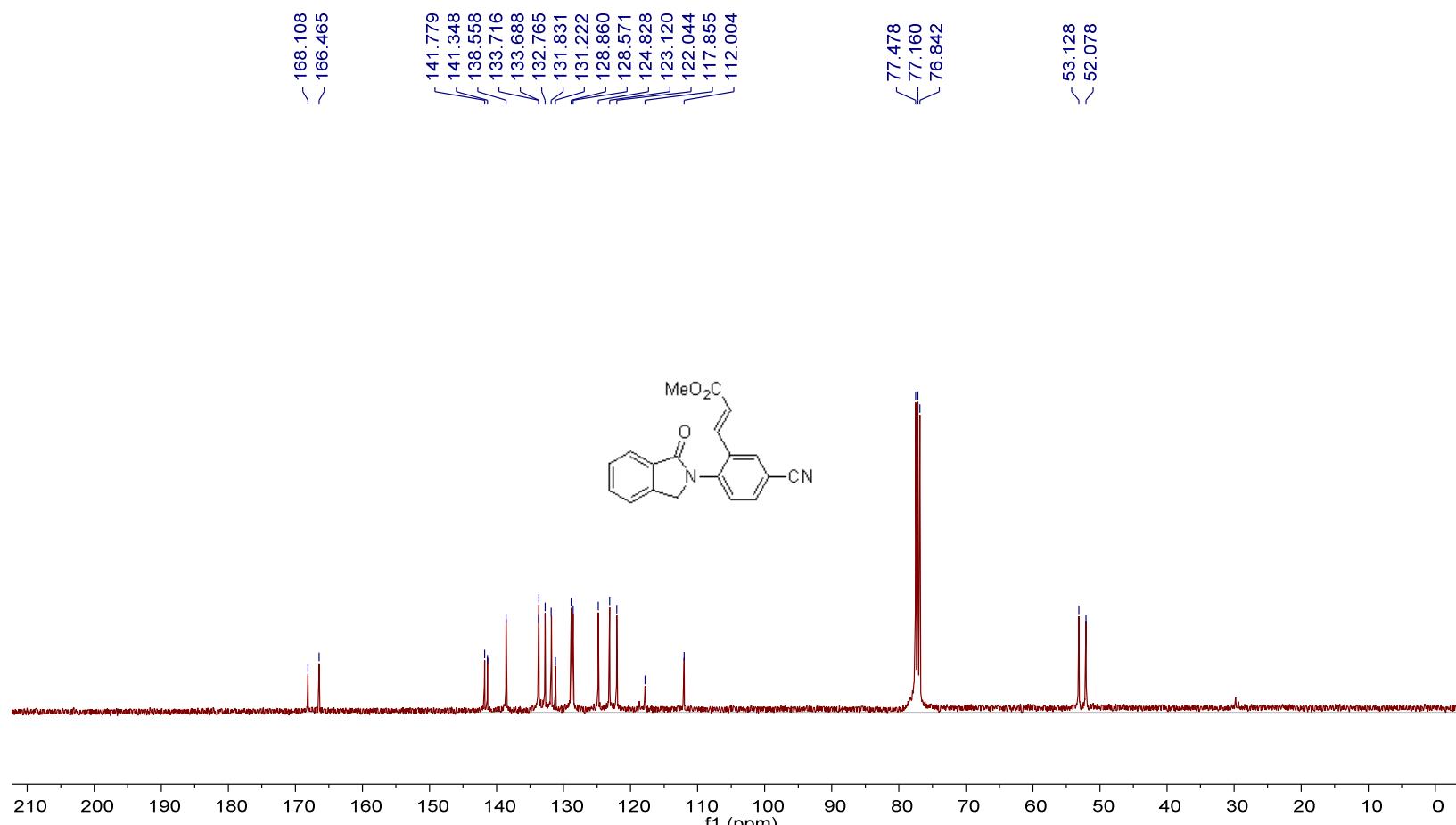
<sup>1</sup>H NMR spectrum of **2m**.

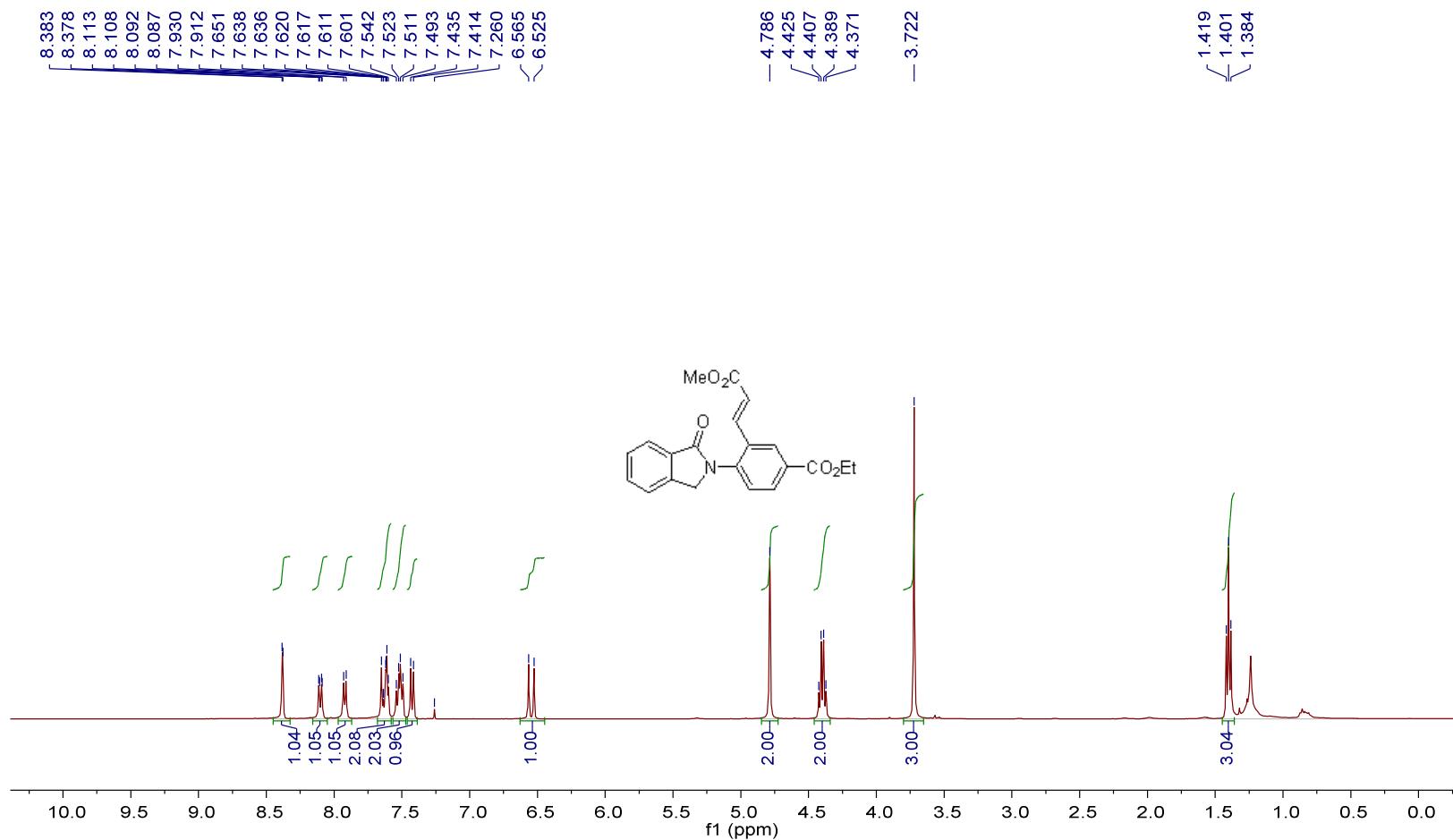


$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2m**.

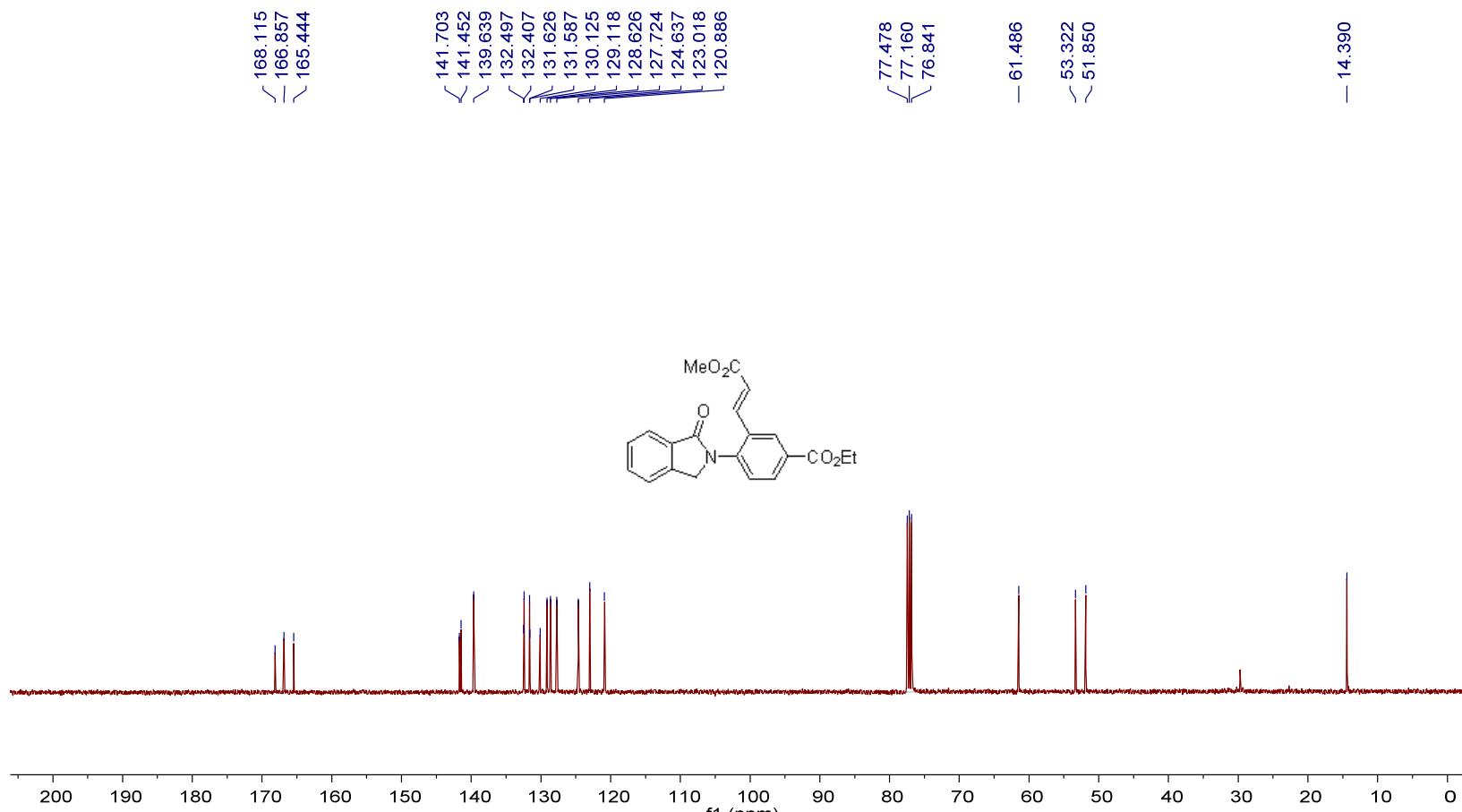


<sup>1</sup>H NMR spectrum of **2n**.

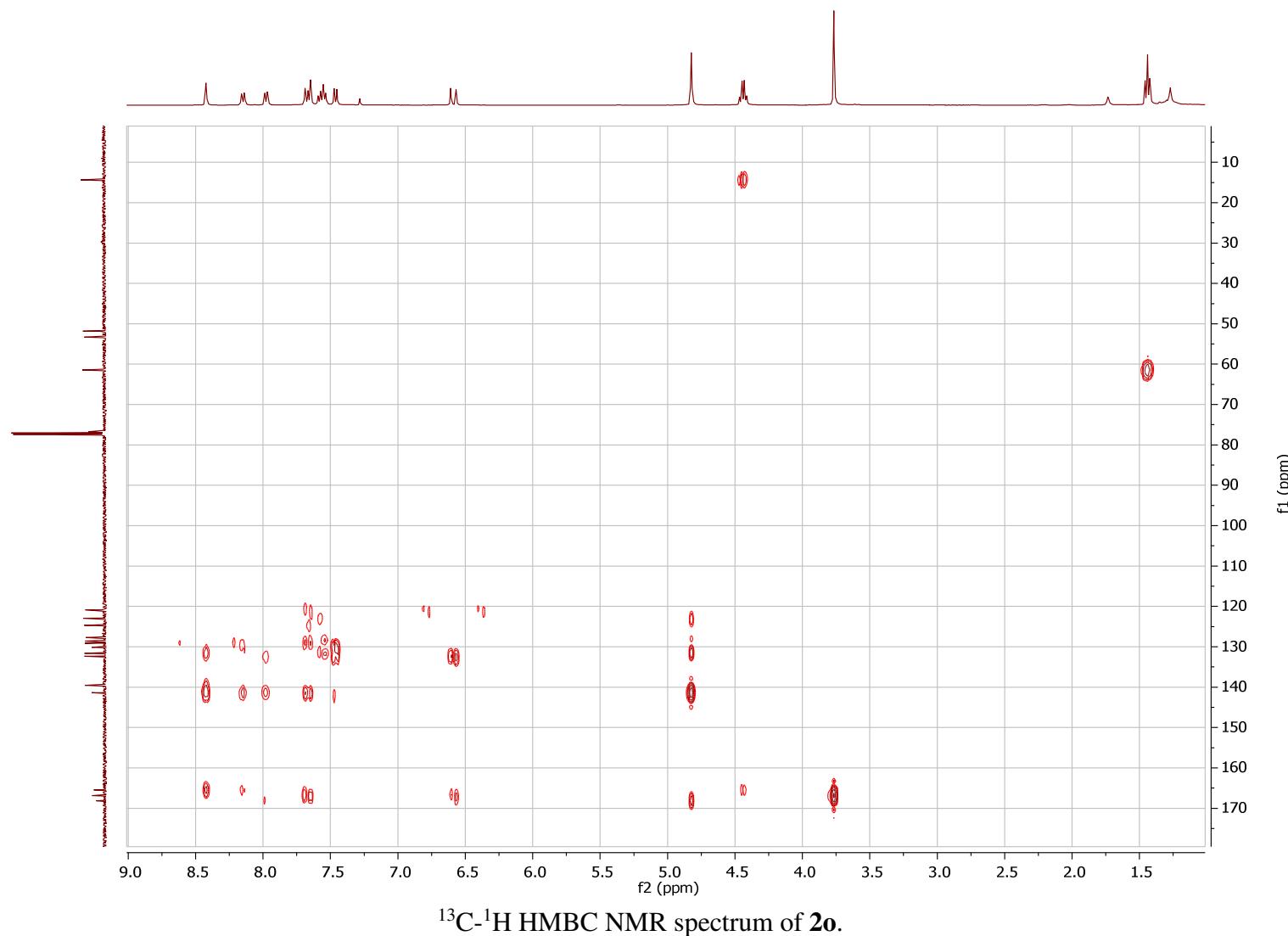


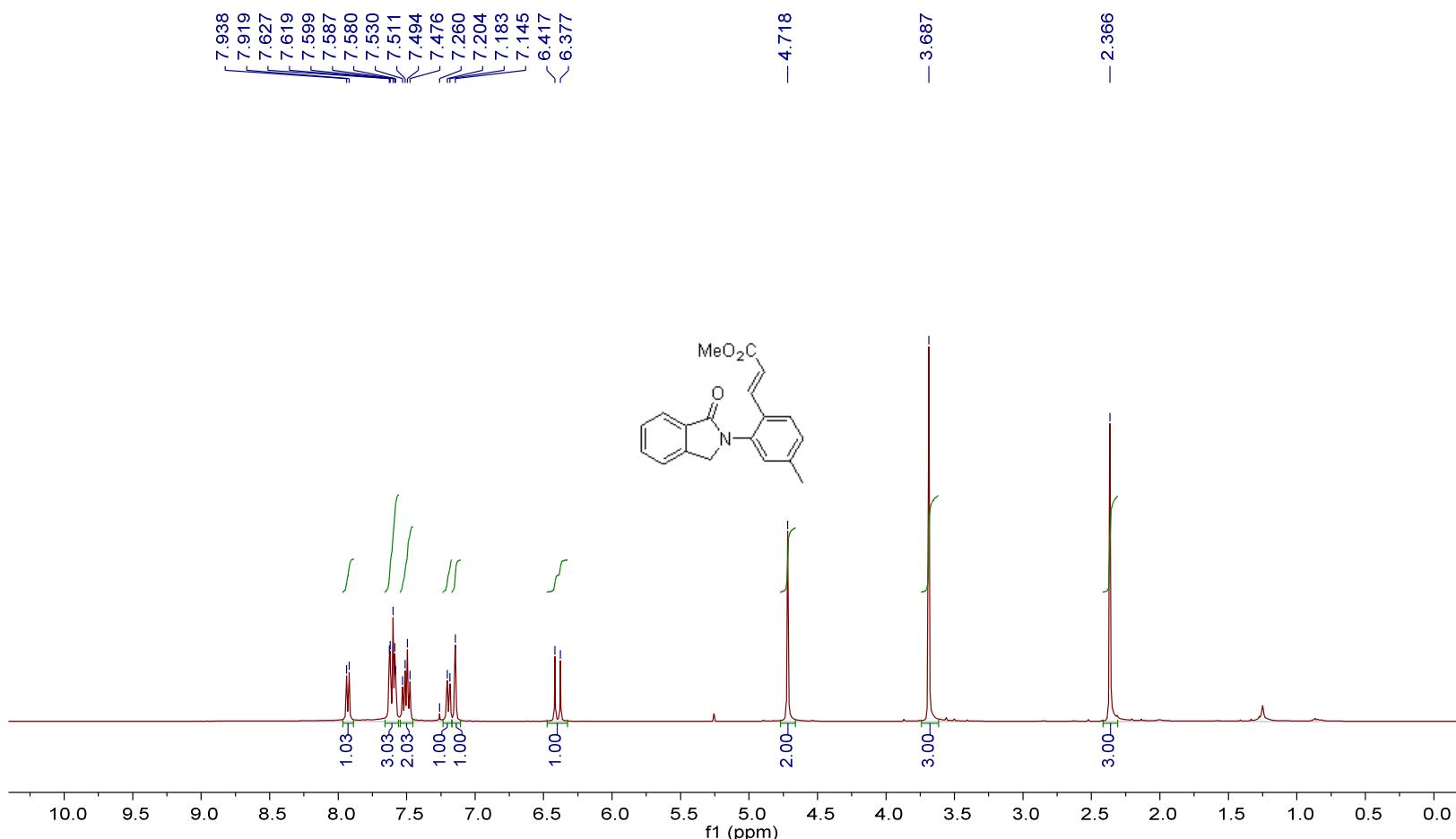


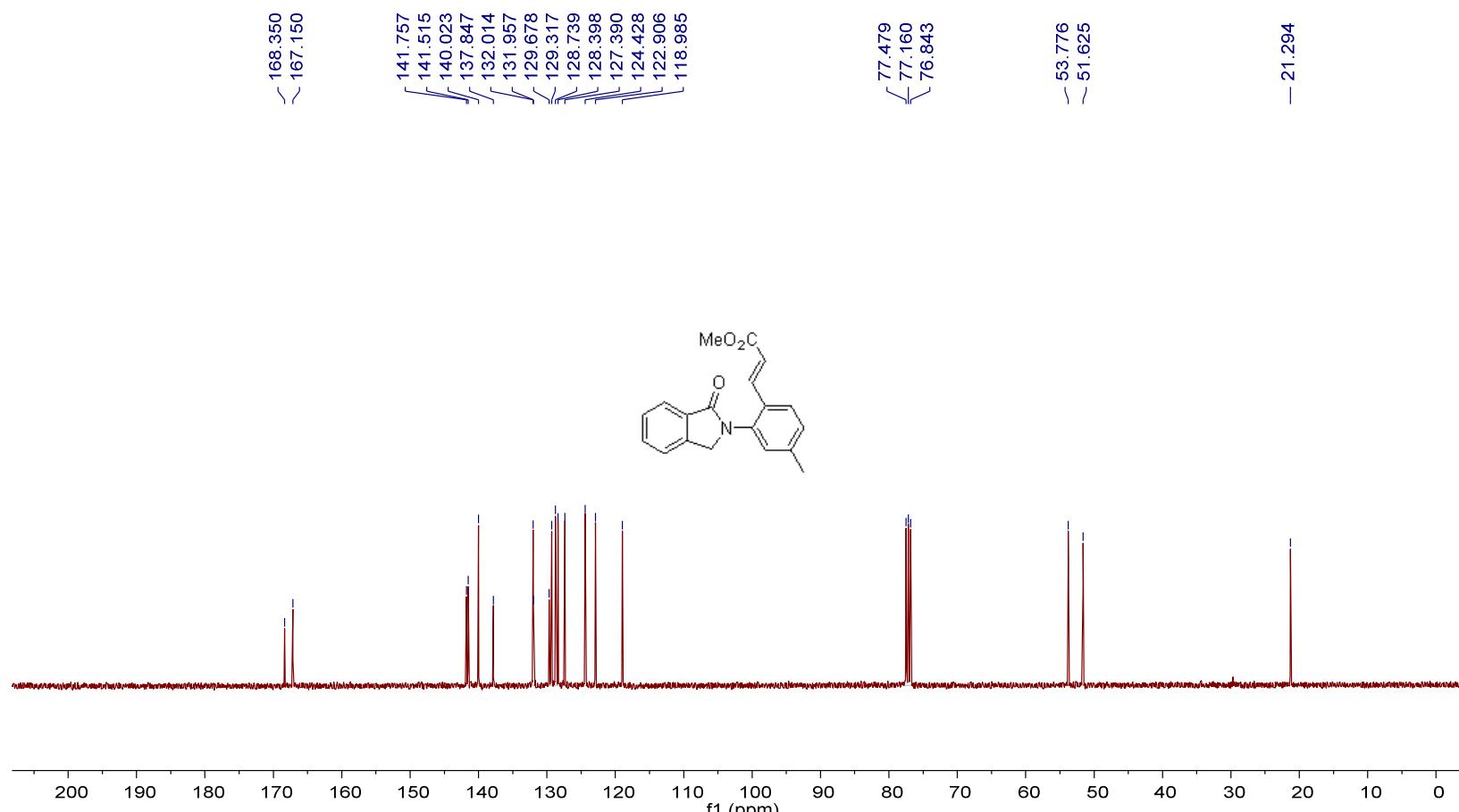
<sup>1</sup>H NMR spectrum of **2o**.



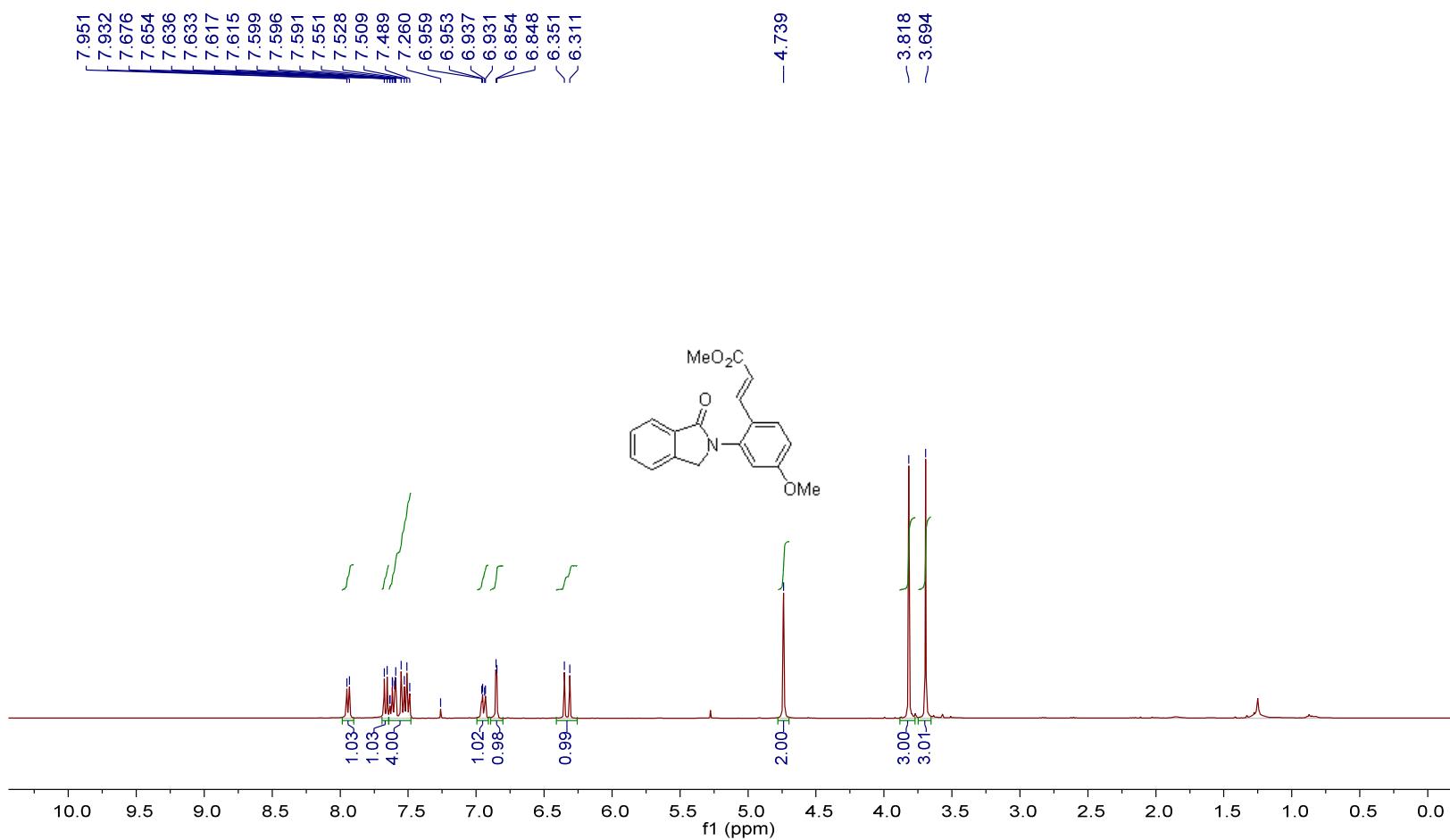
<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2o**.

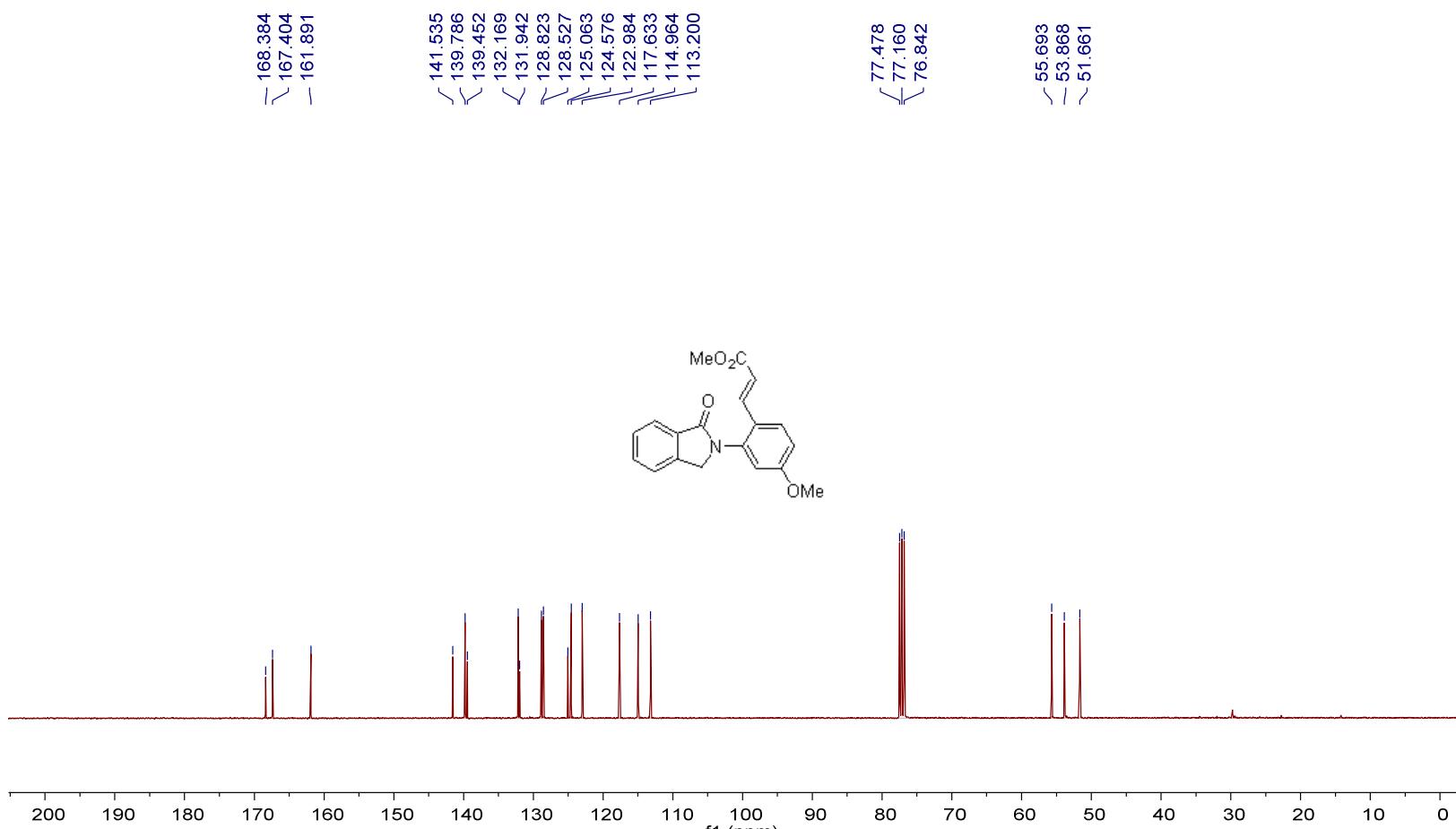




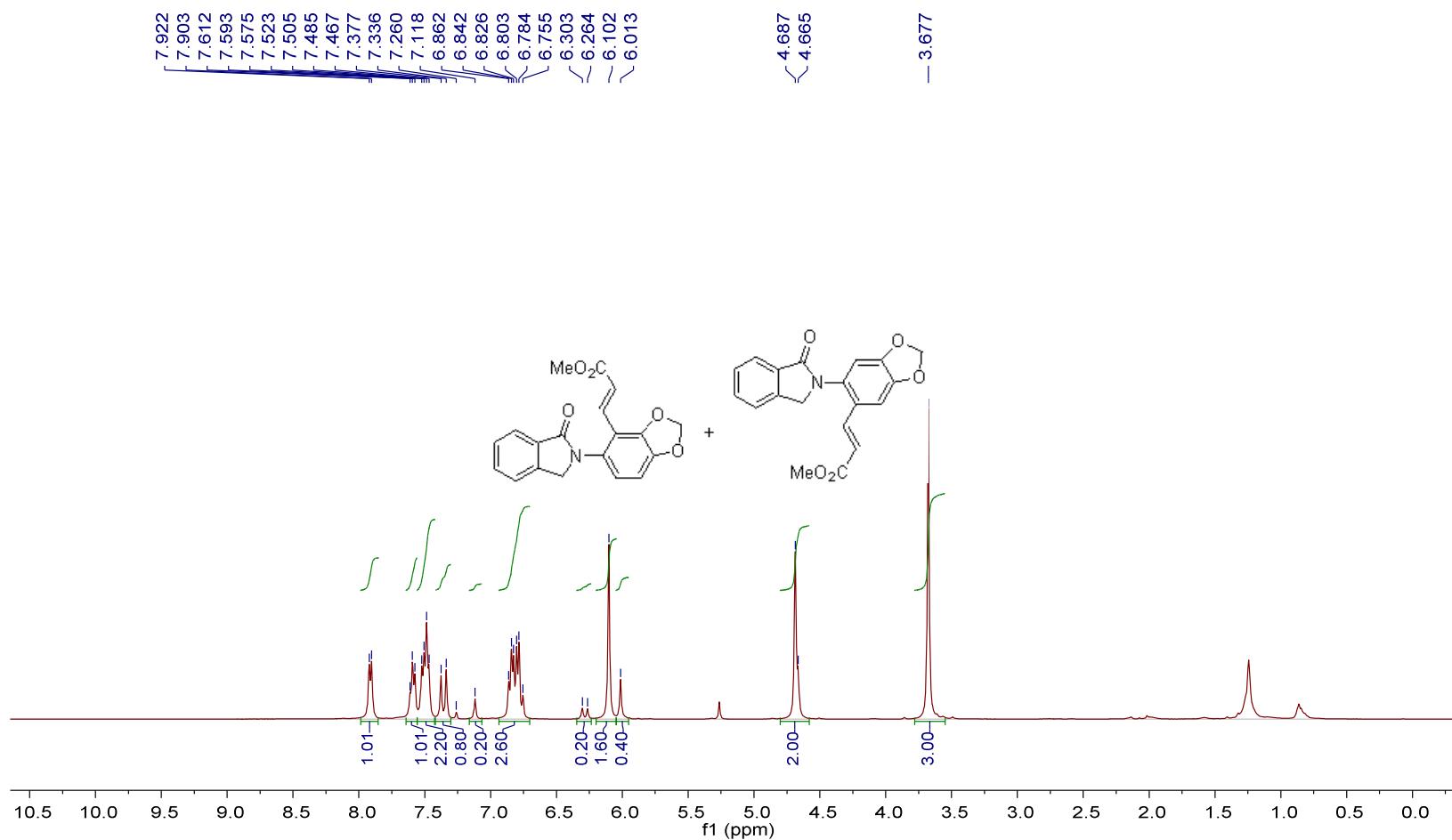


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2p**.

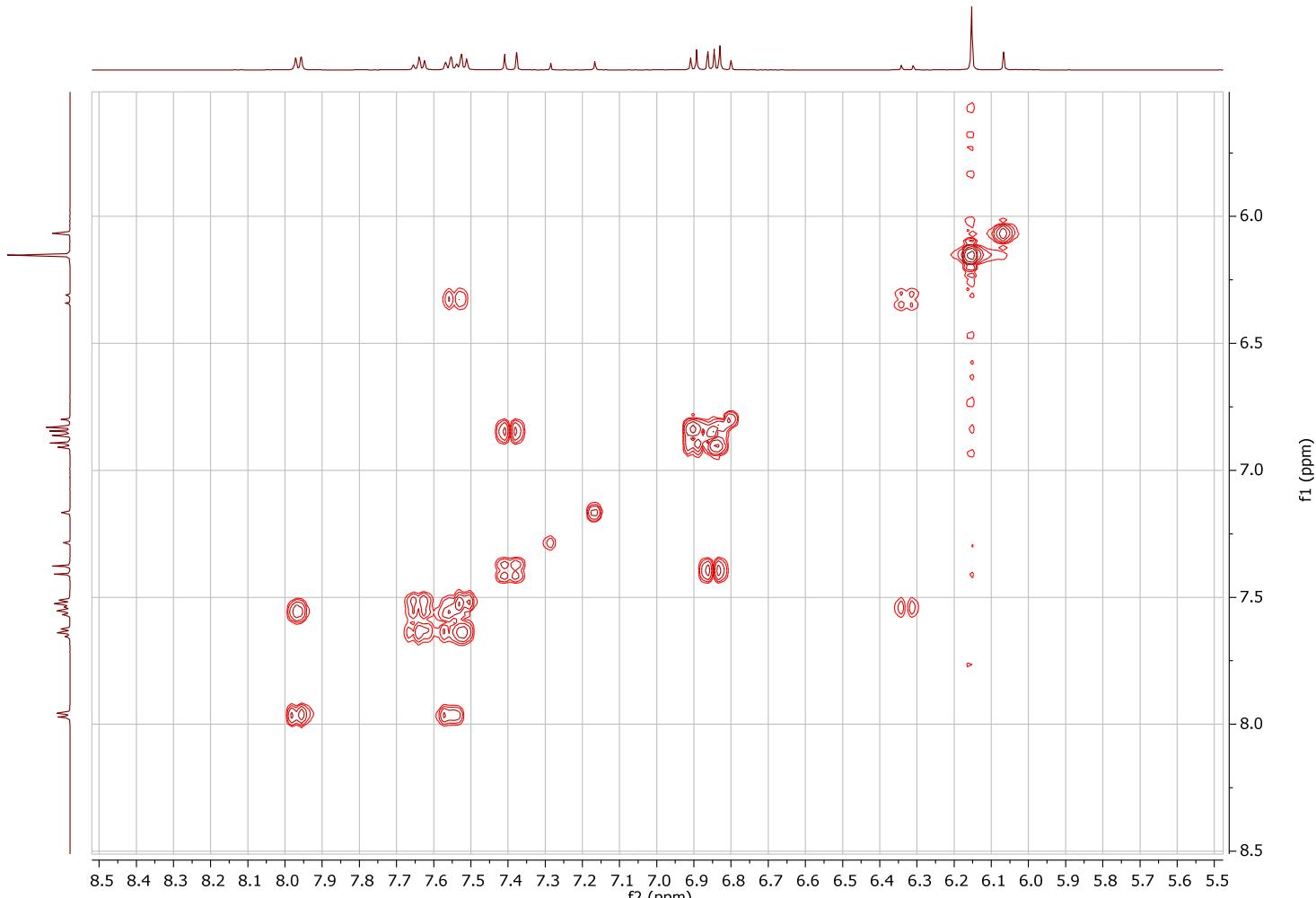


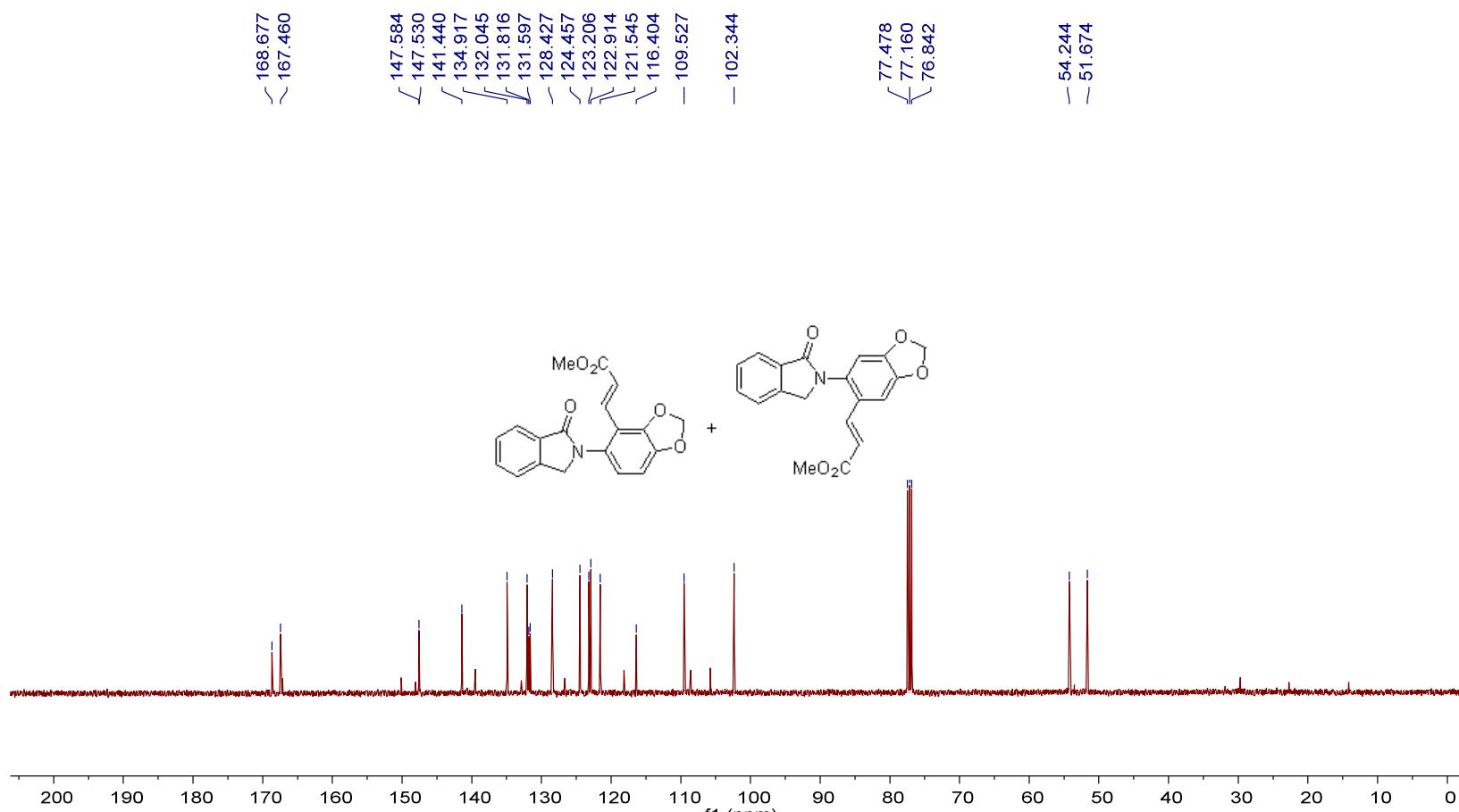


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2q**.

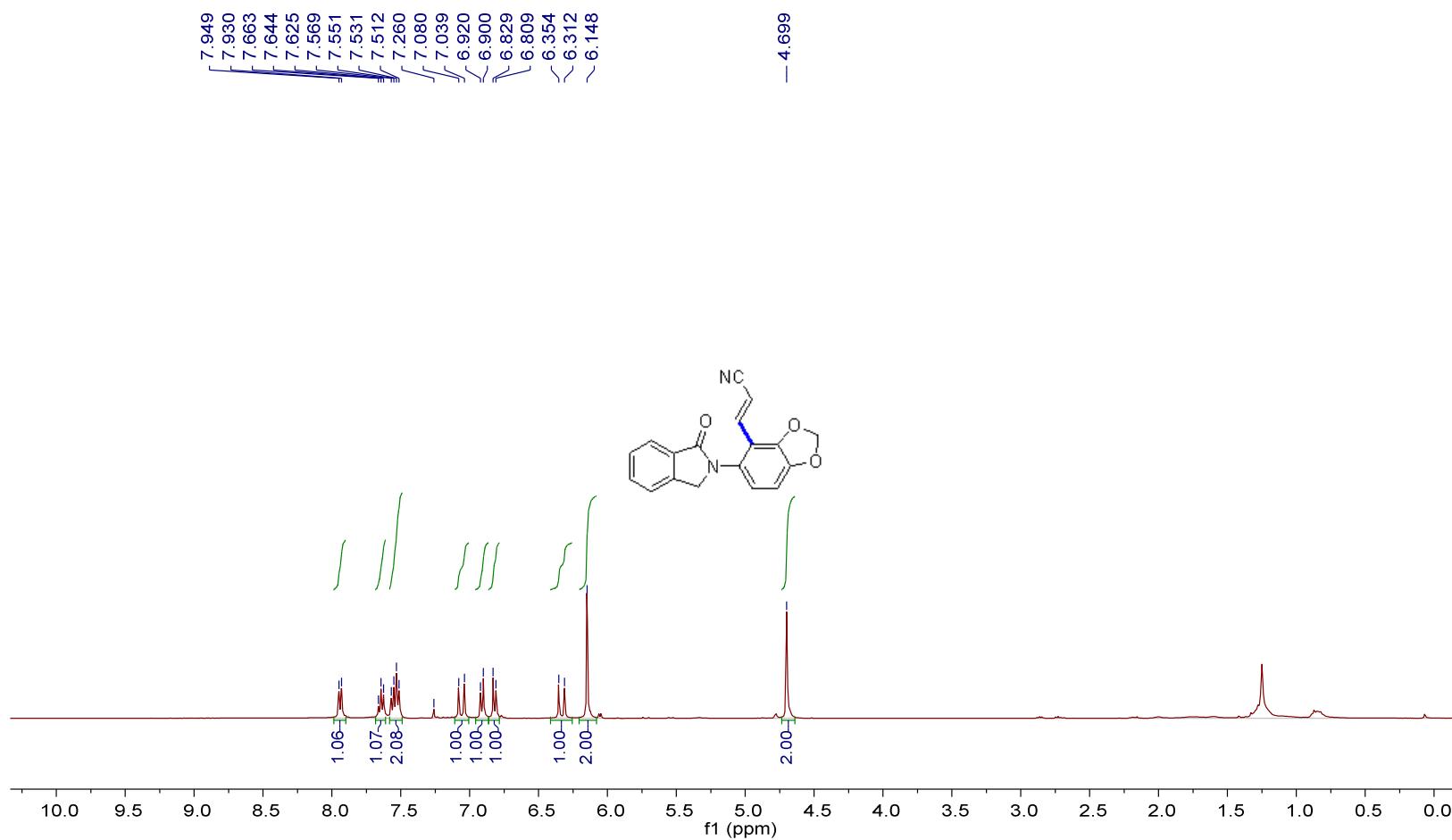


$^1\text{H}$  NMR spectrum of **2r**.

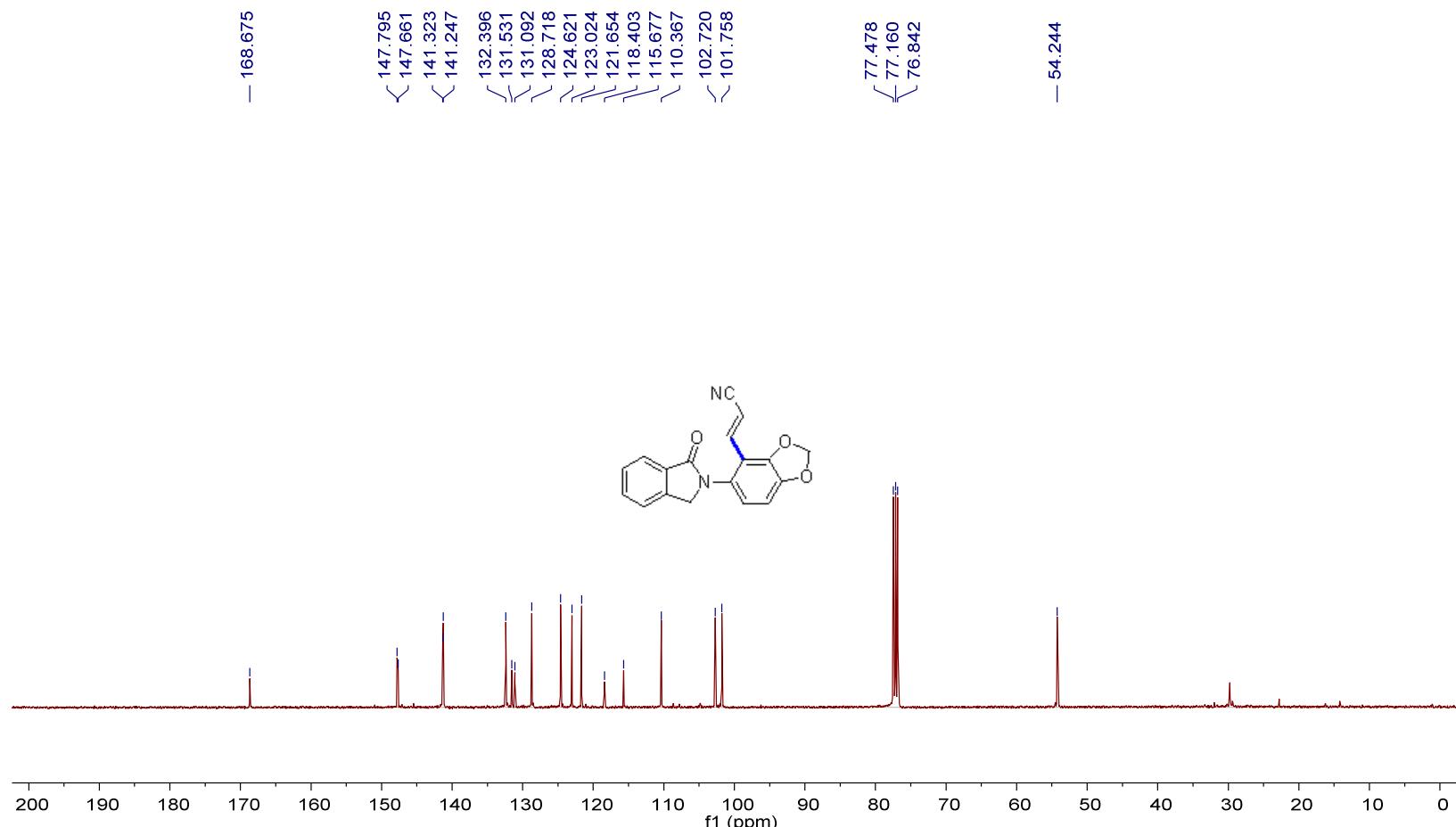




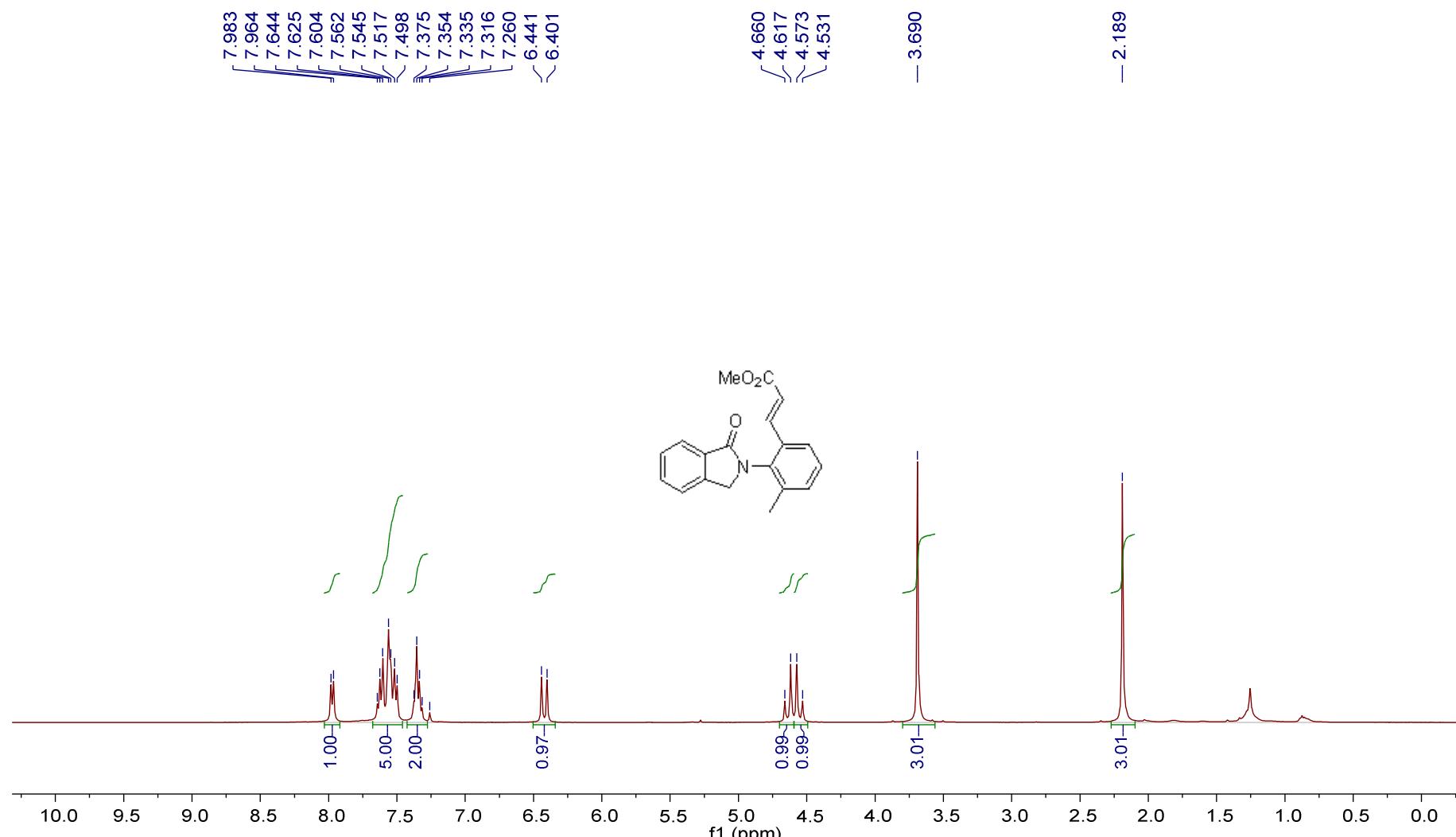
$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2r**.

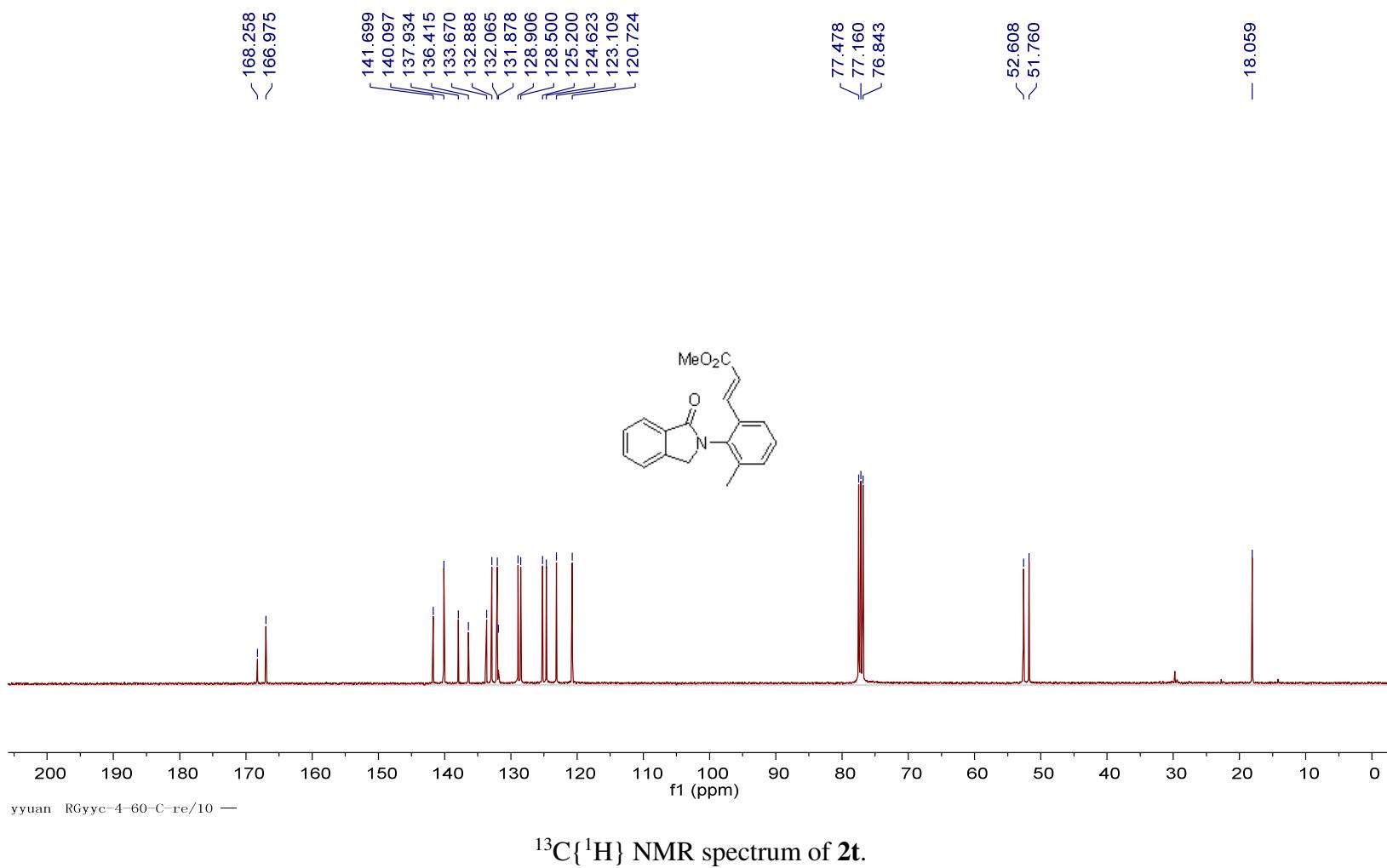


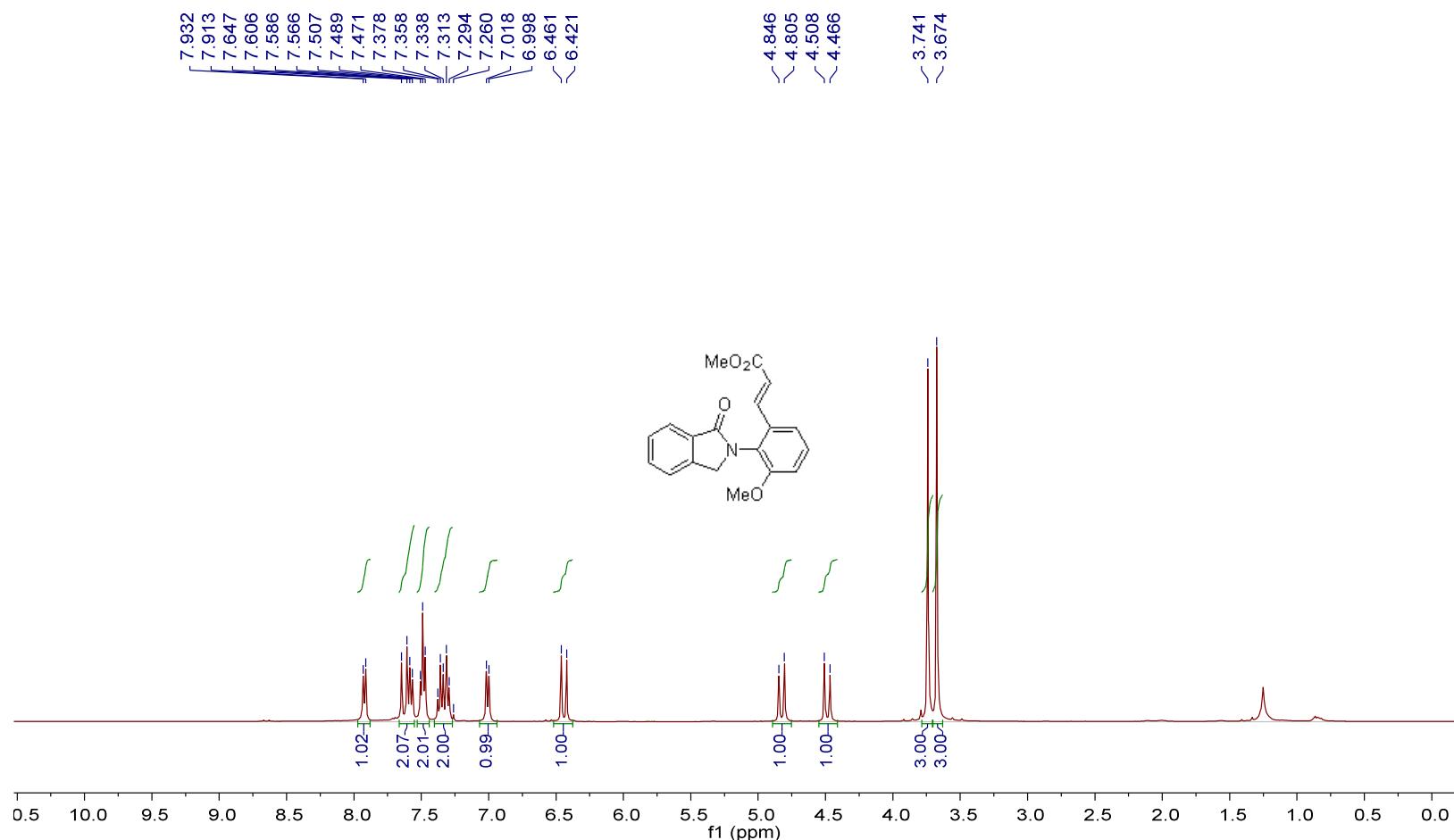
<sup>1</sup>H NMR spectrum of **2s**.

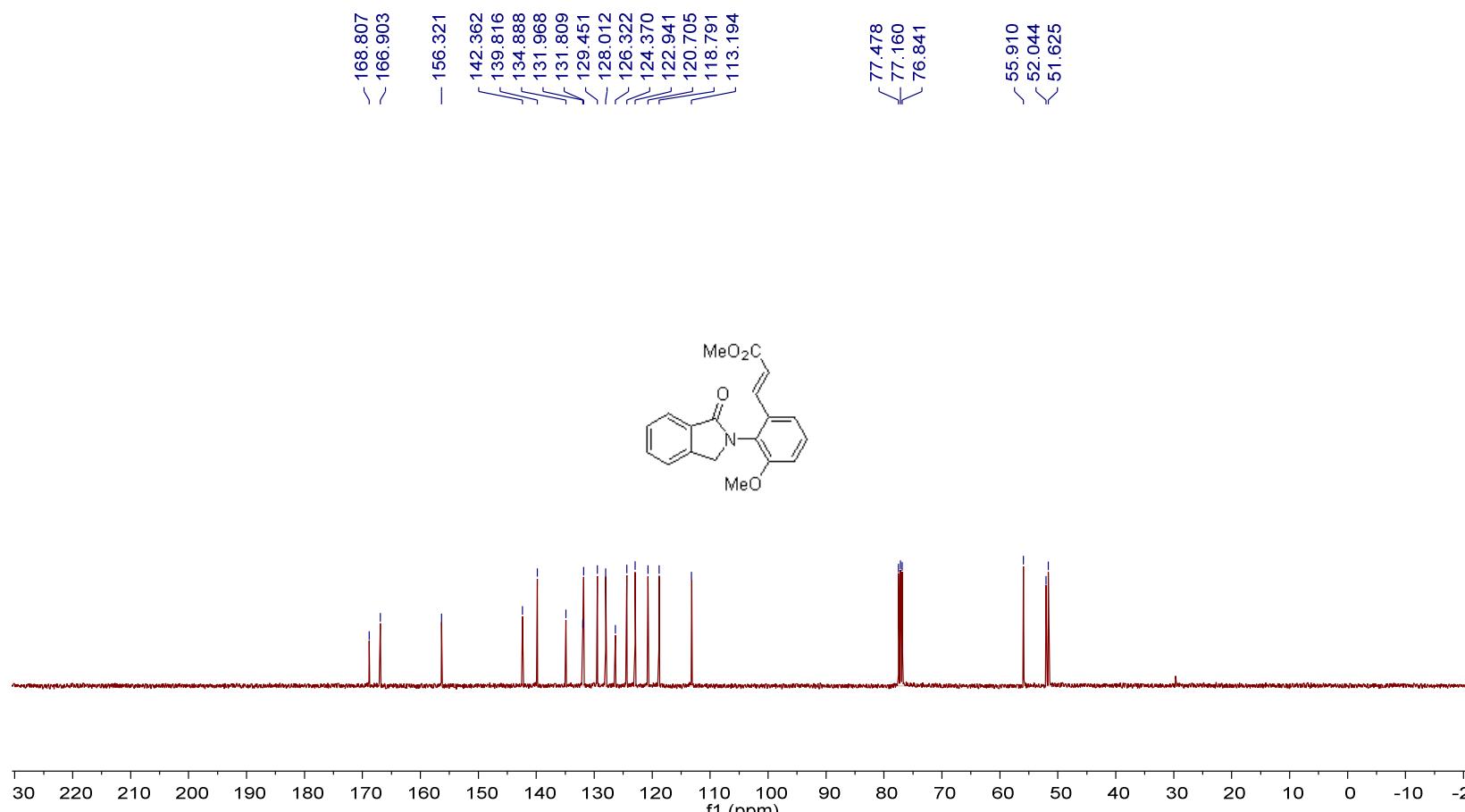


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2s**

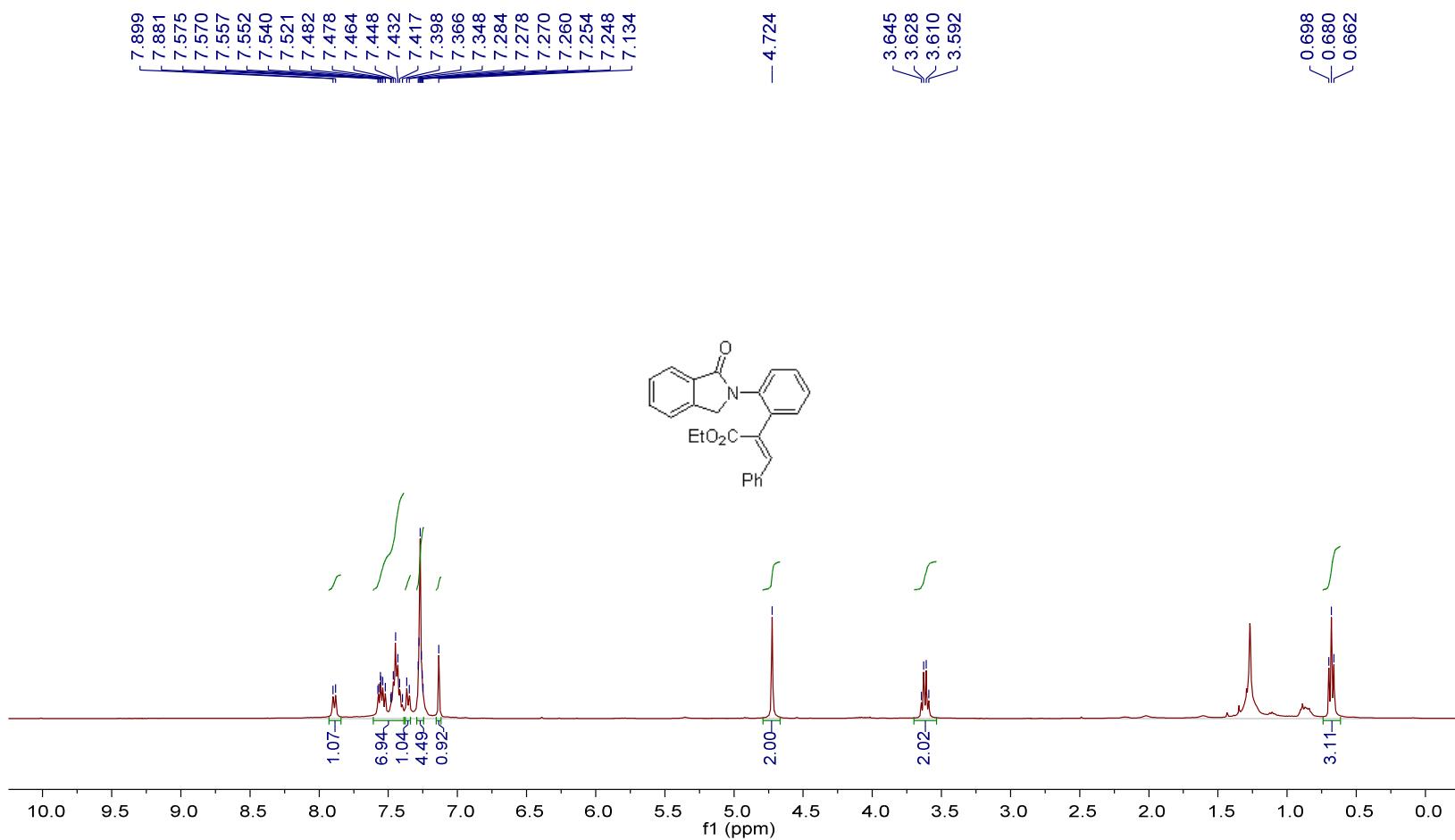




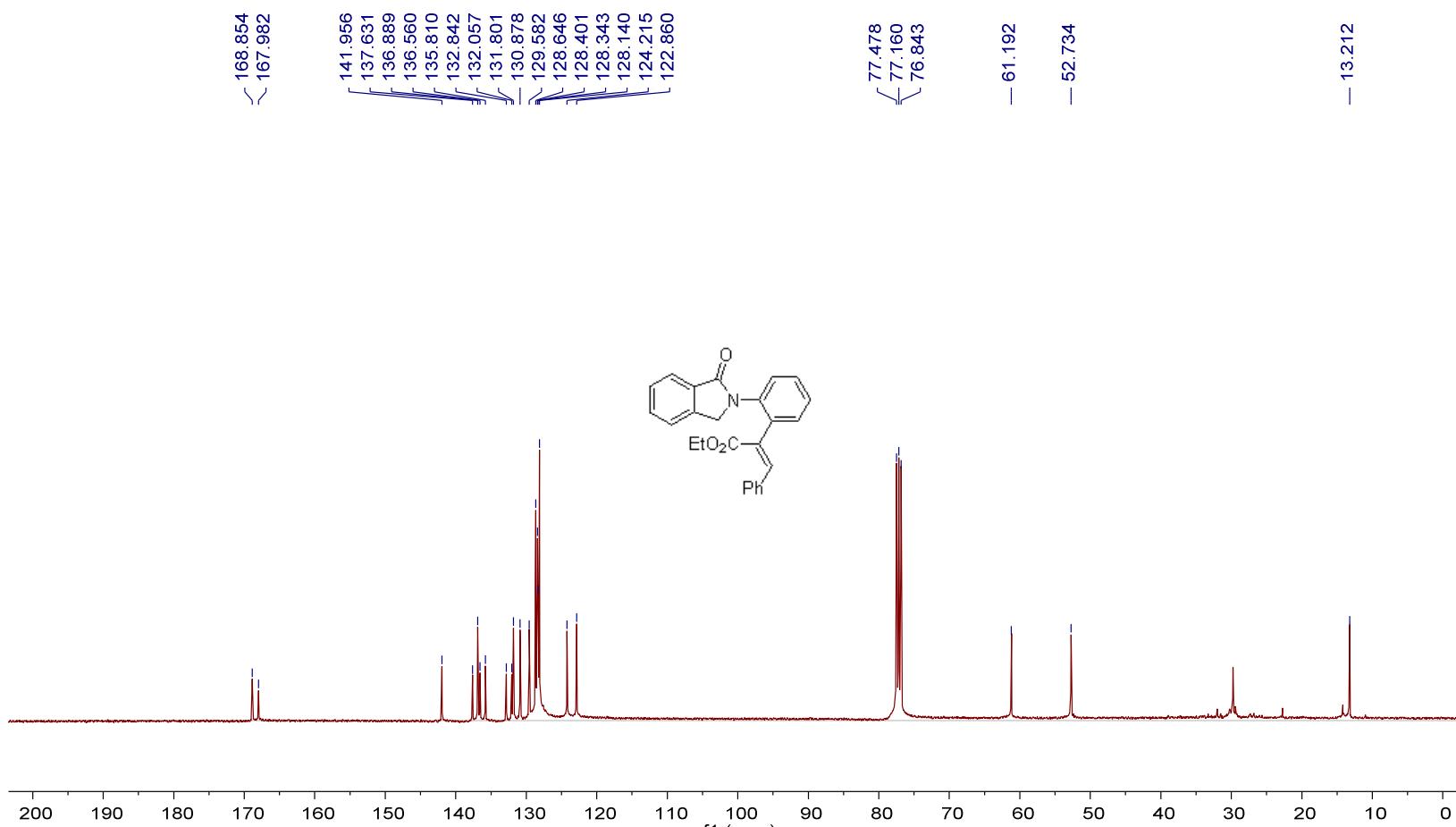


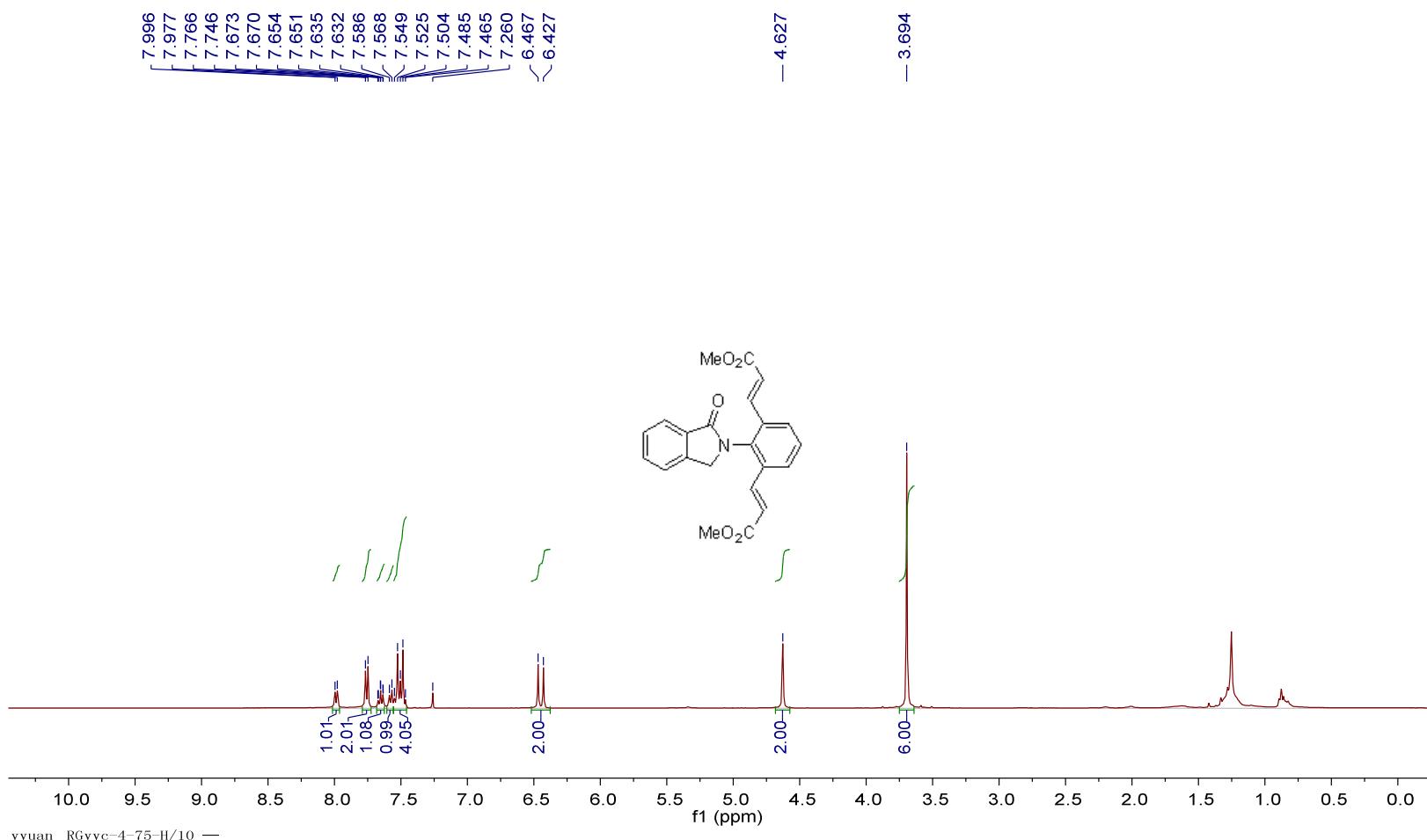


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2u**.

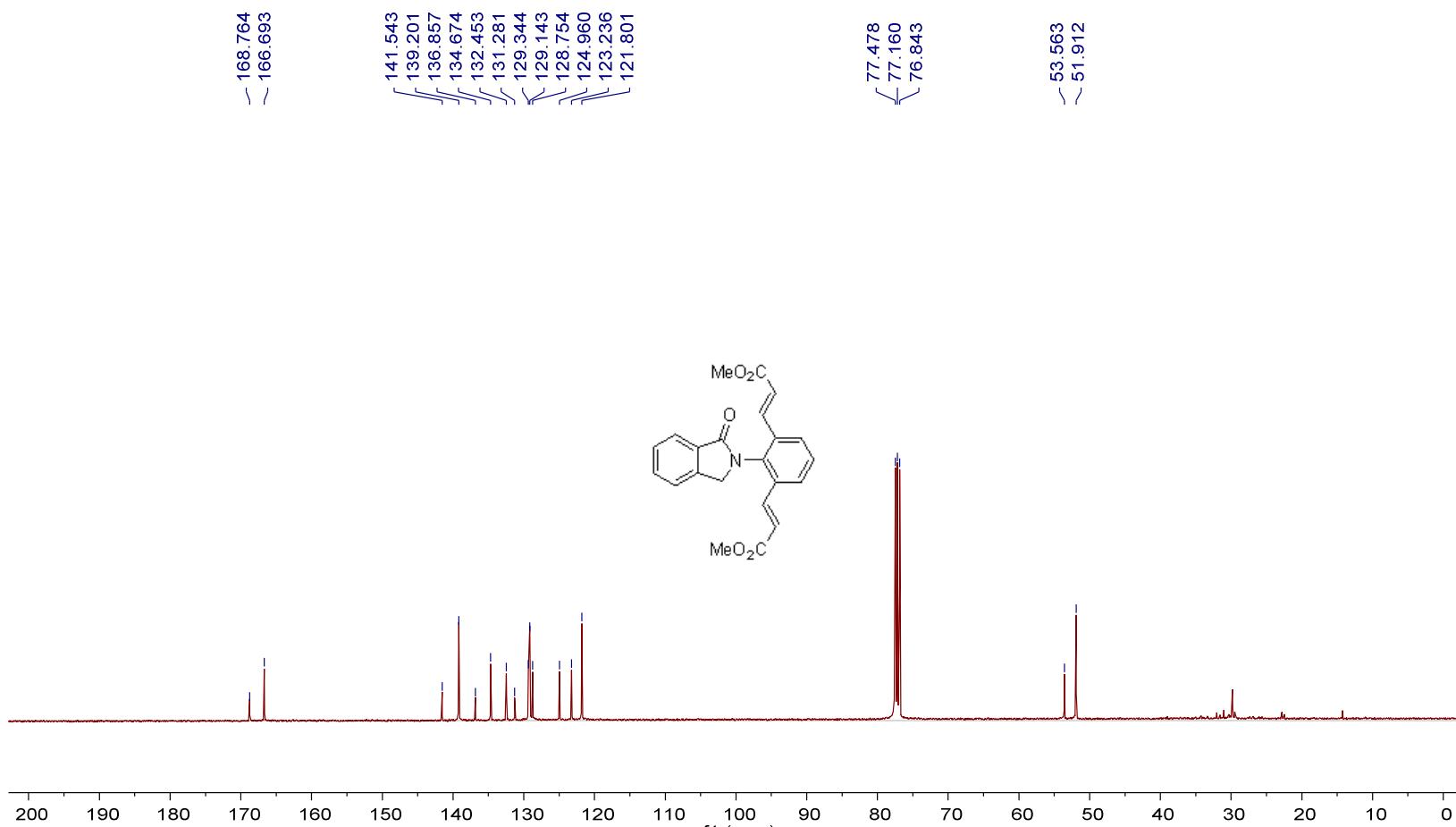


<sup>1</sup>H NMR spectrum of **2v**.

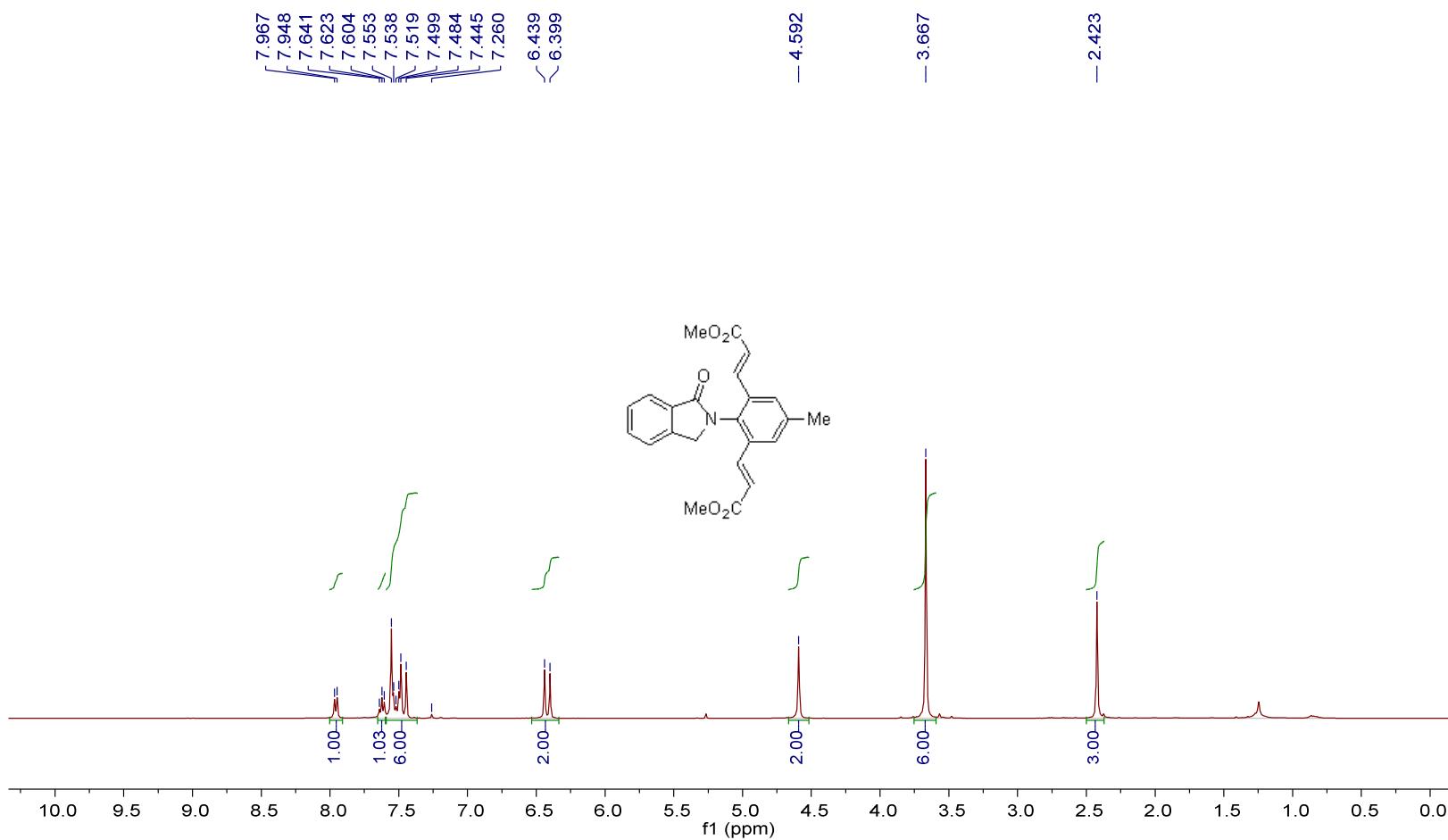




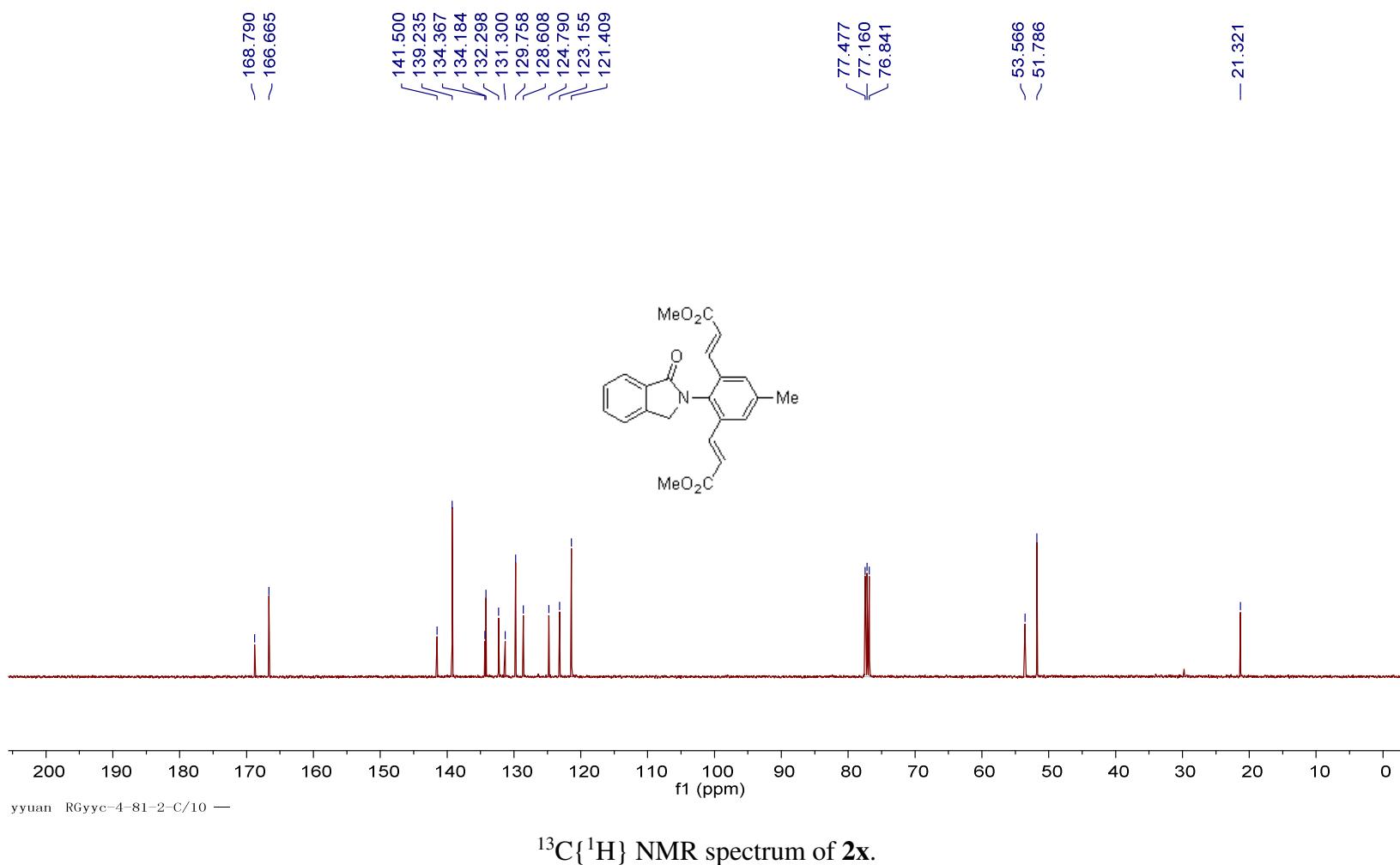
<sup>1</sup>H NMR spectrum of **2w**.

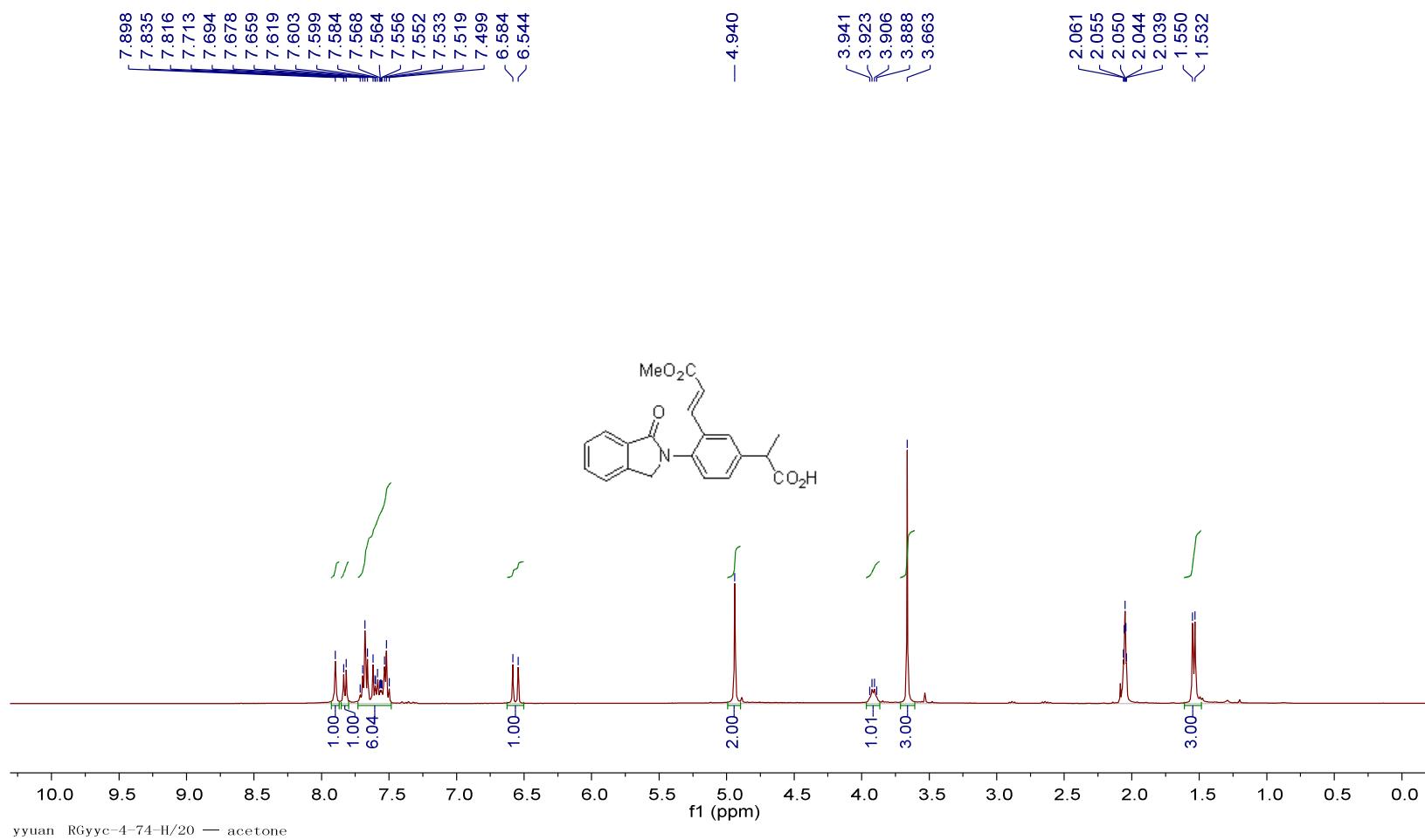


$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2w**.

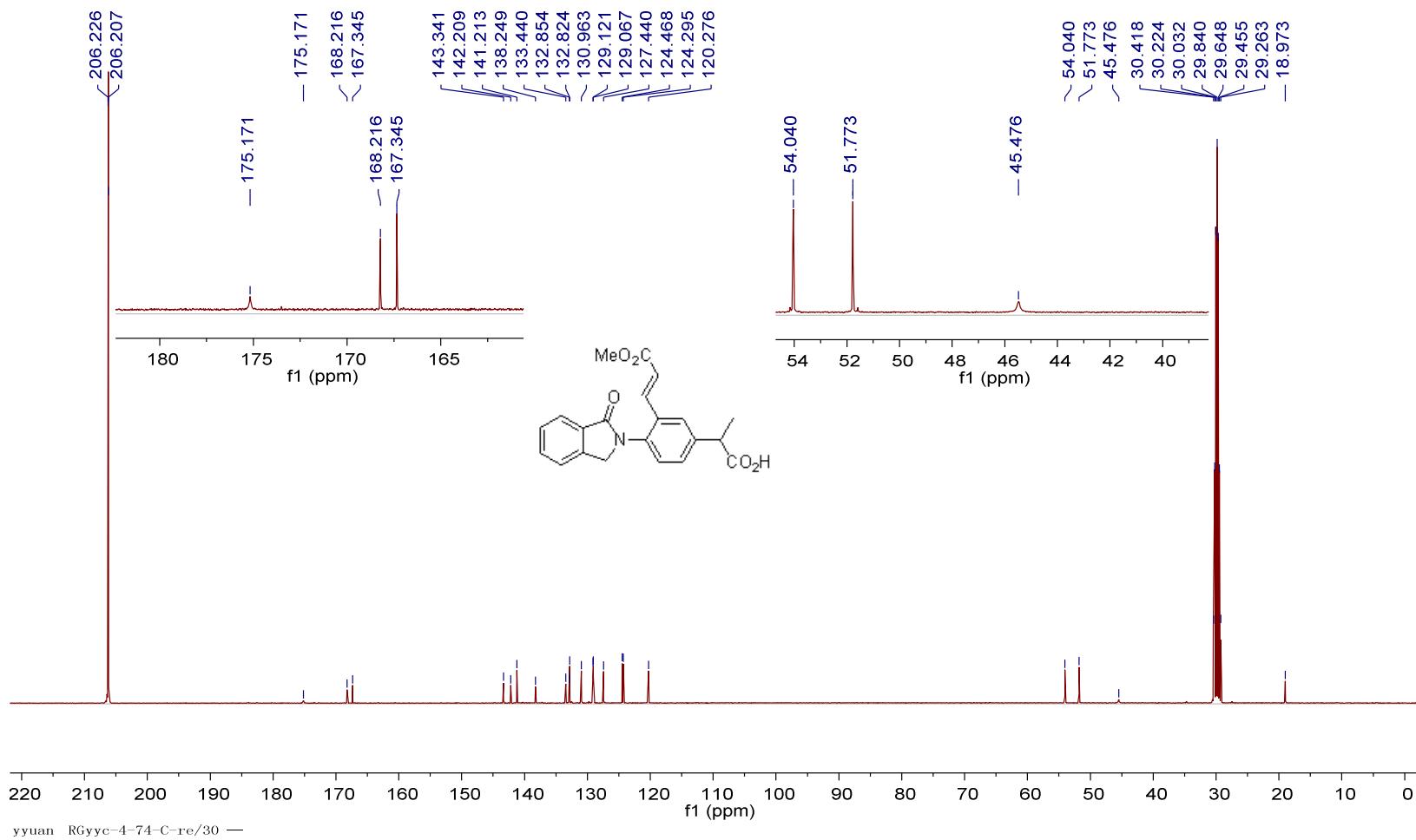


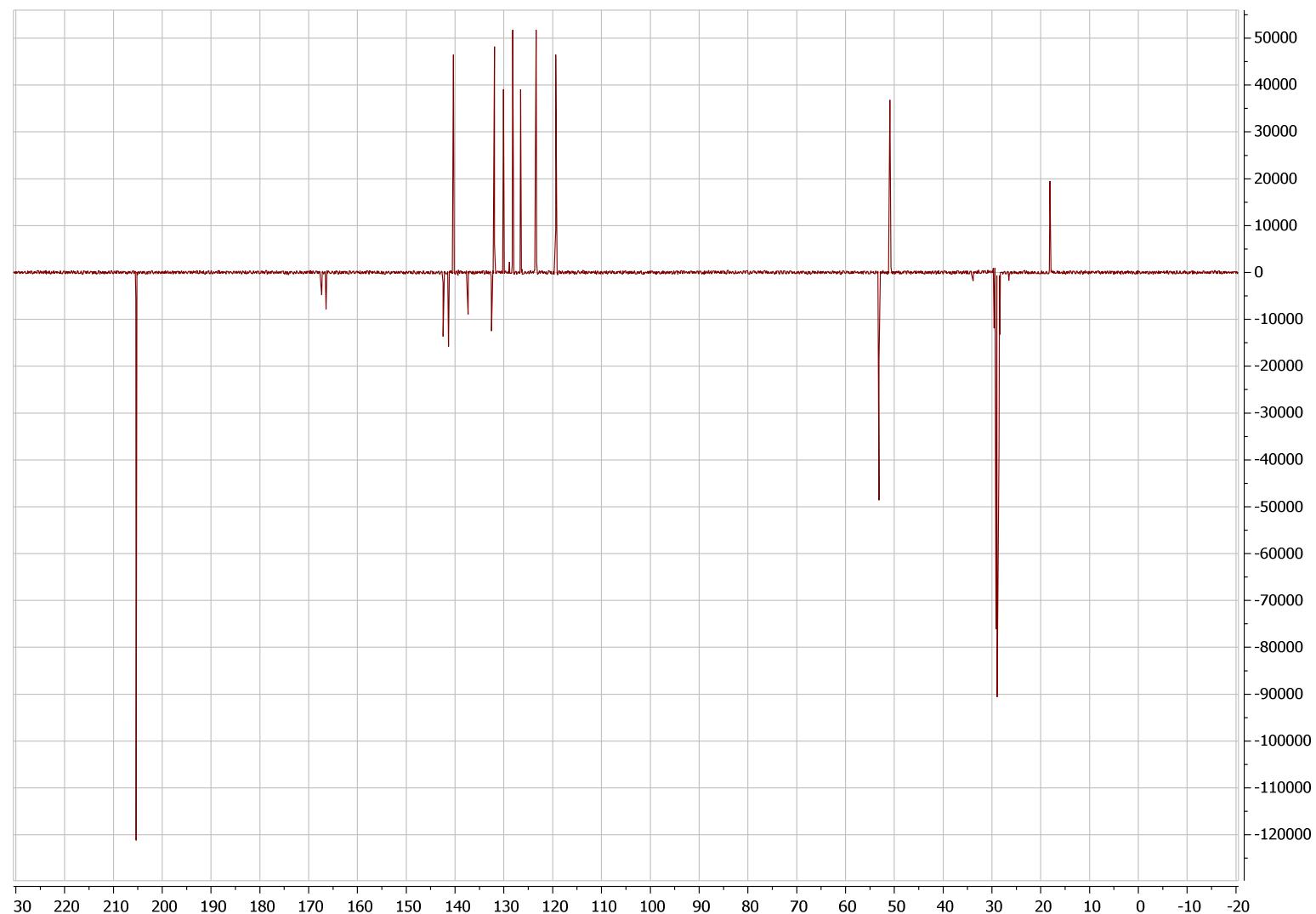
<sup>1</sup>H NMR spectrum of **2x**.



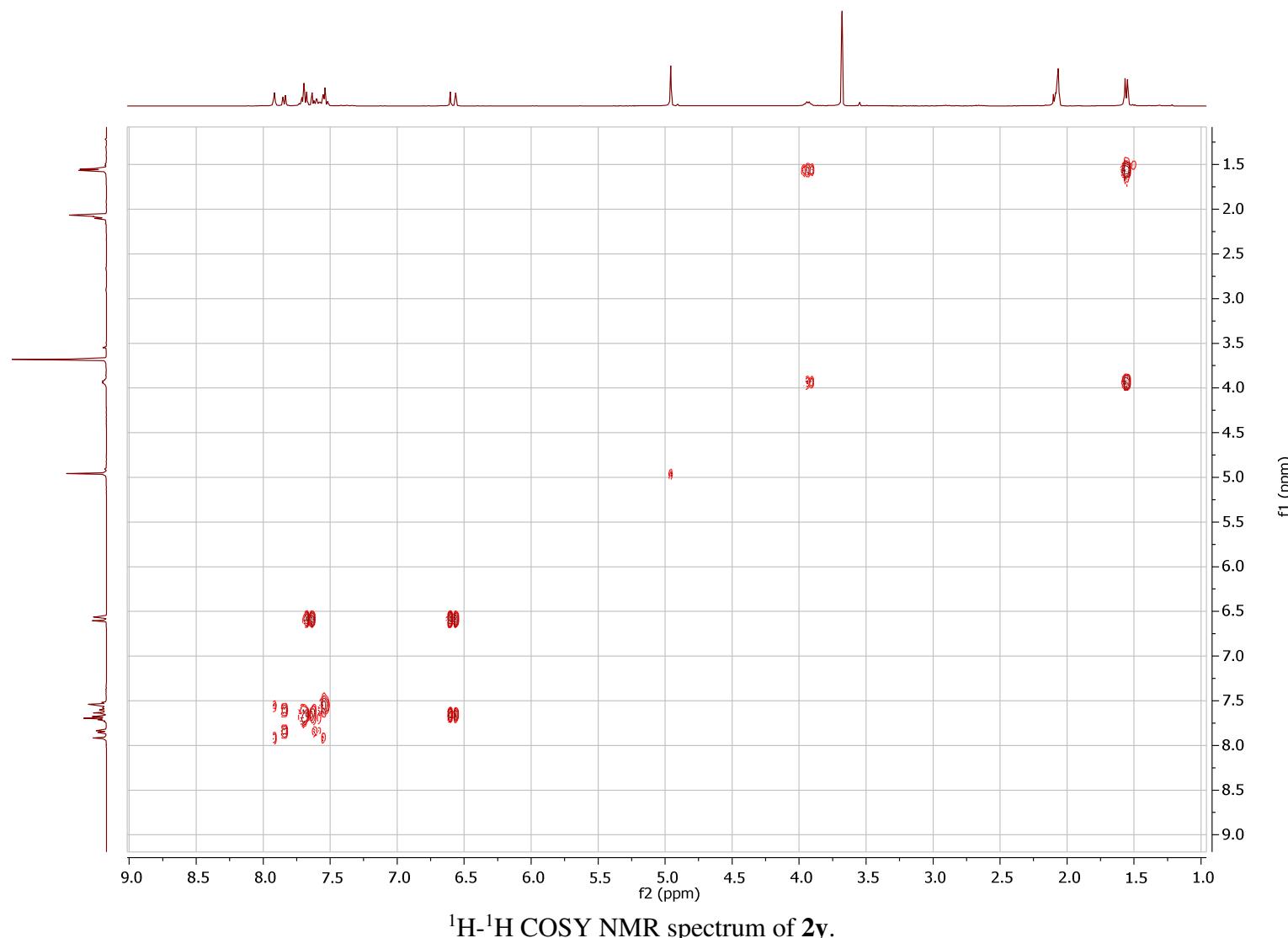


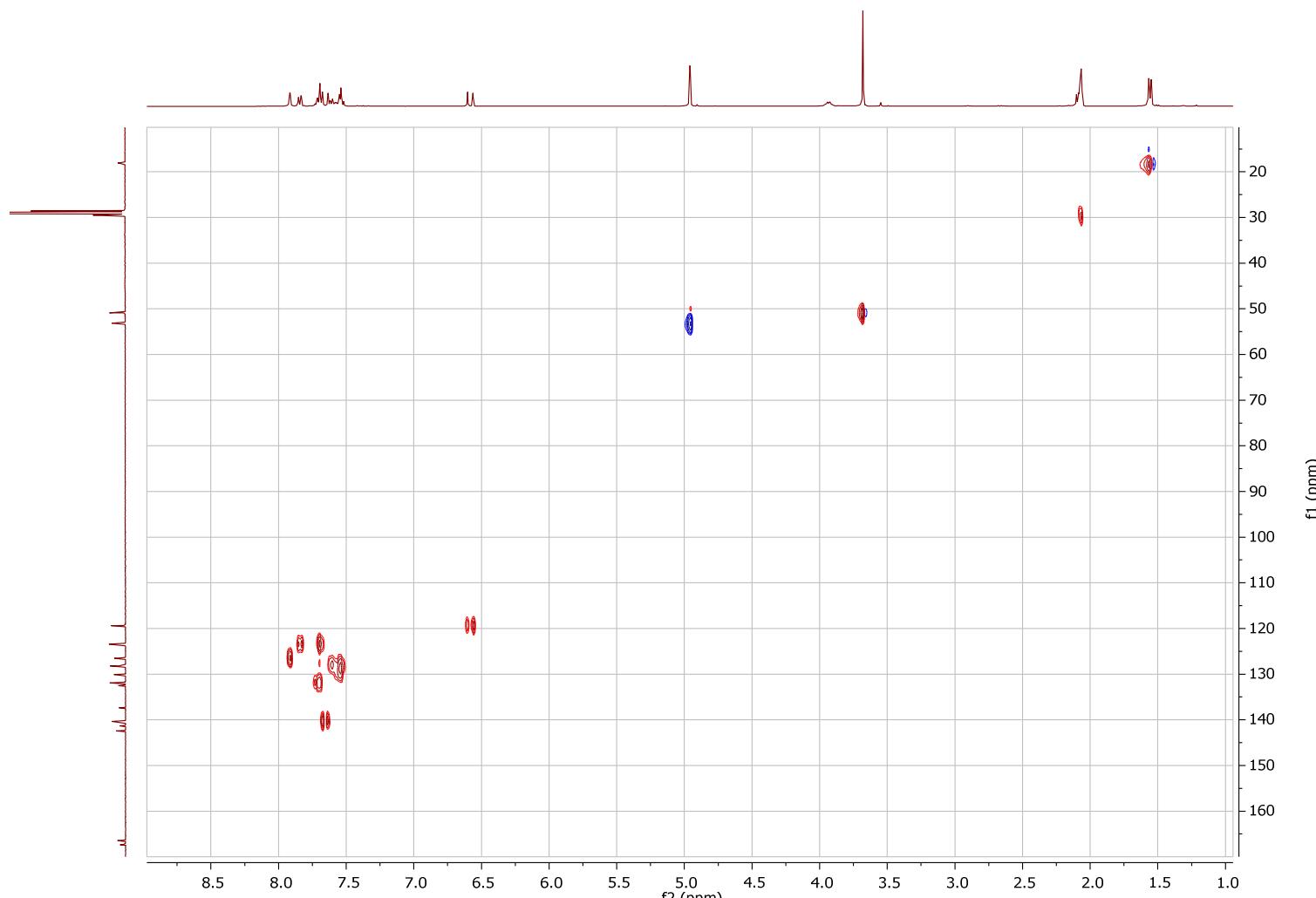
<sup>1</sup>H NMR spectrum of 2y.

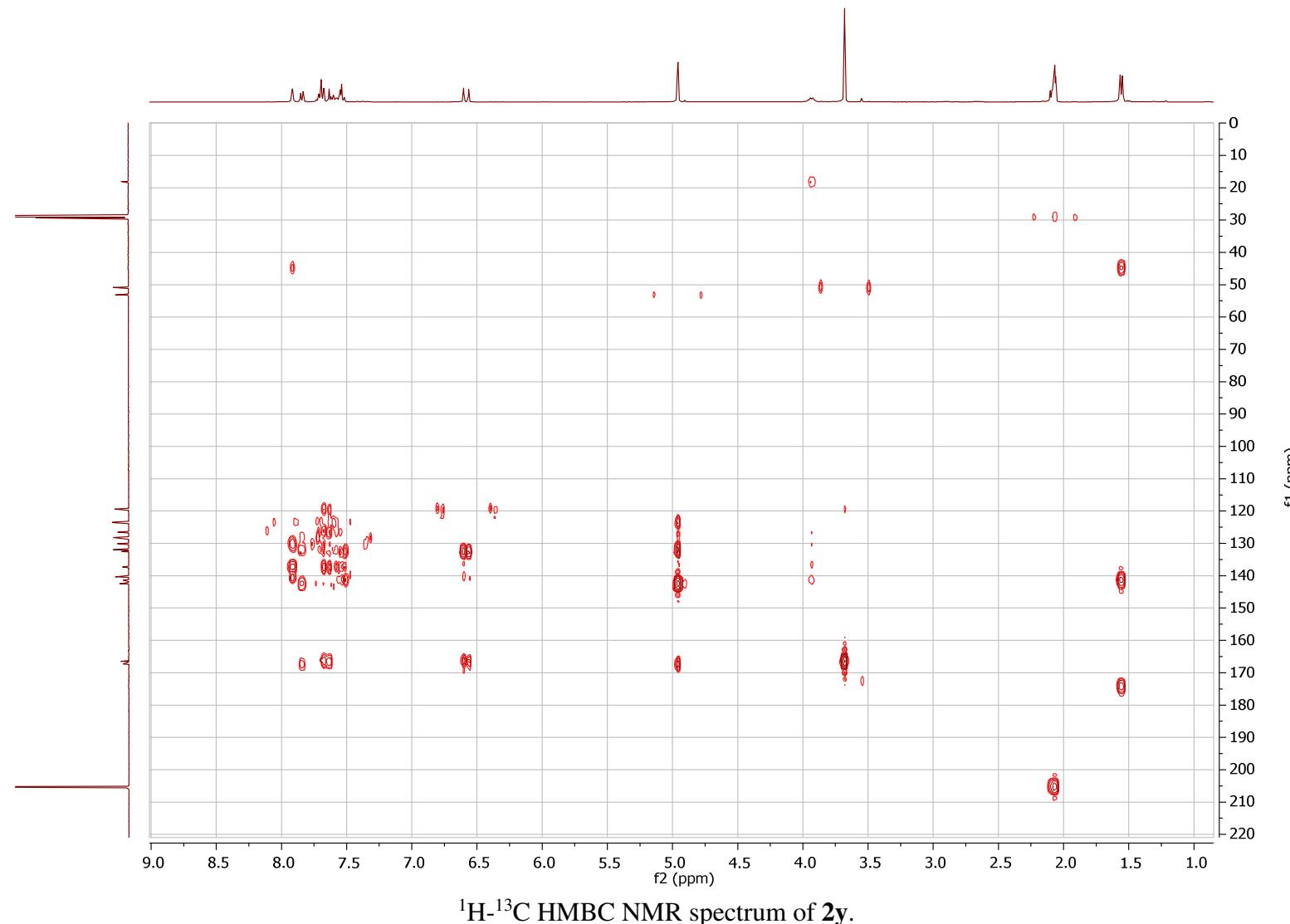


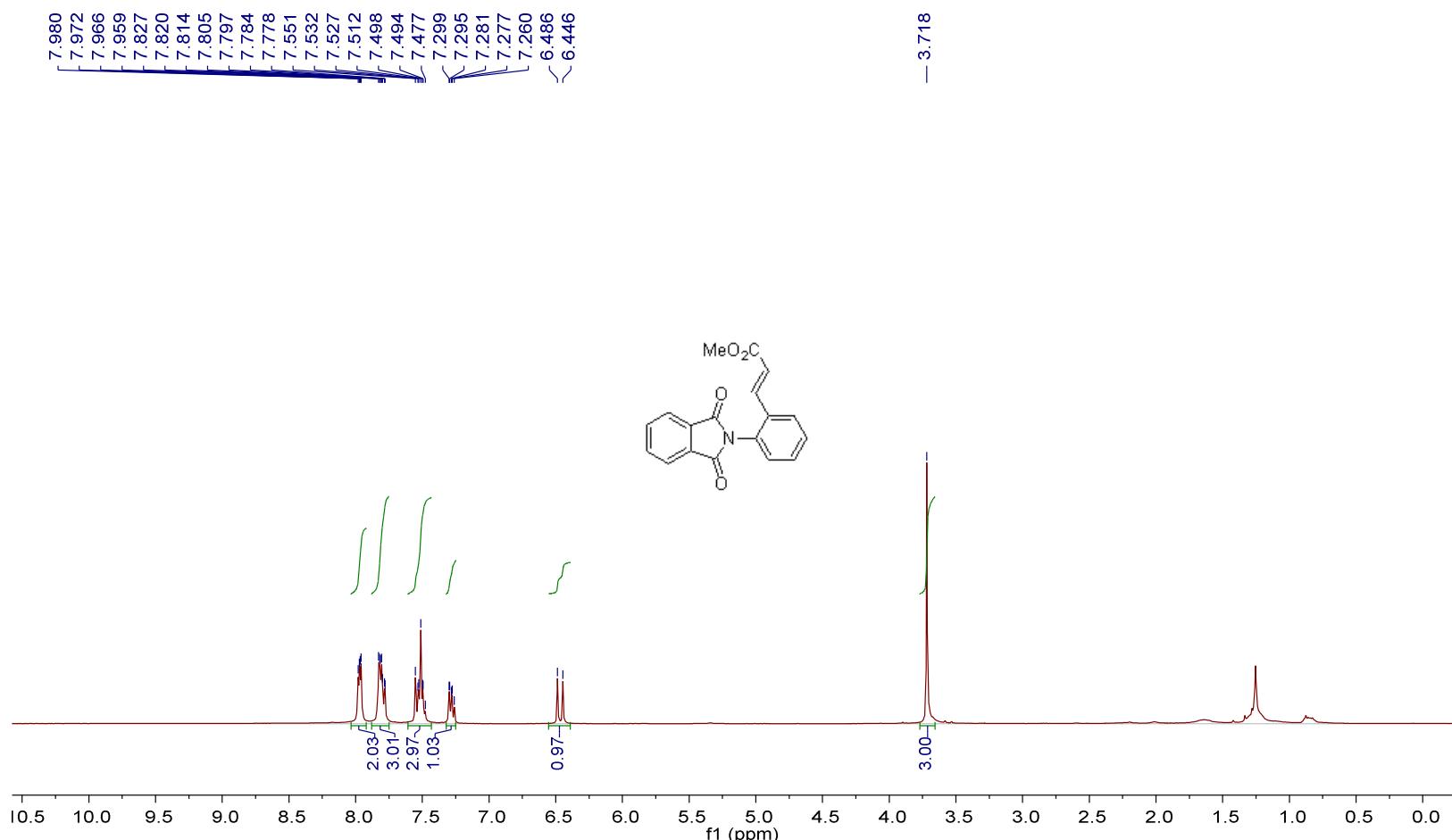


$^{13}\text{C}\{^1\text{H}\}$  JMOD NMR spectrum of **2y**.

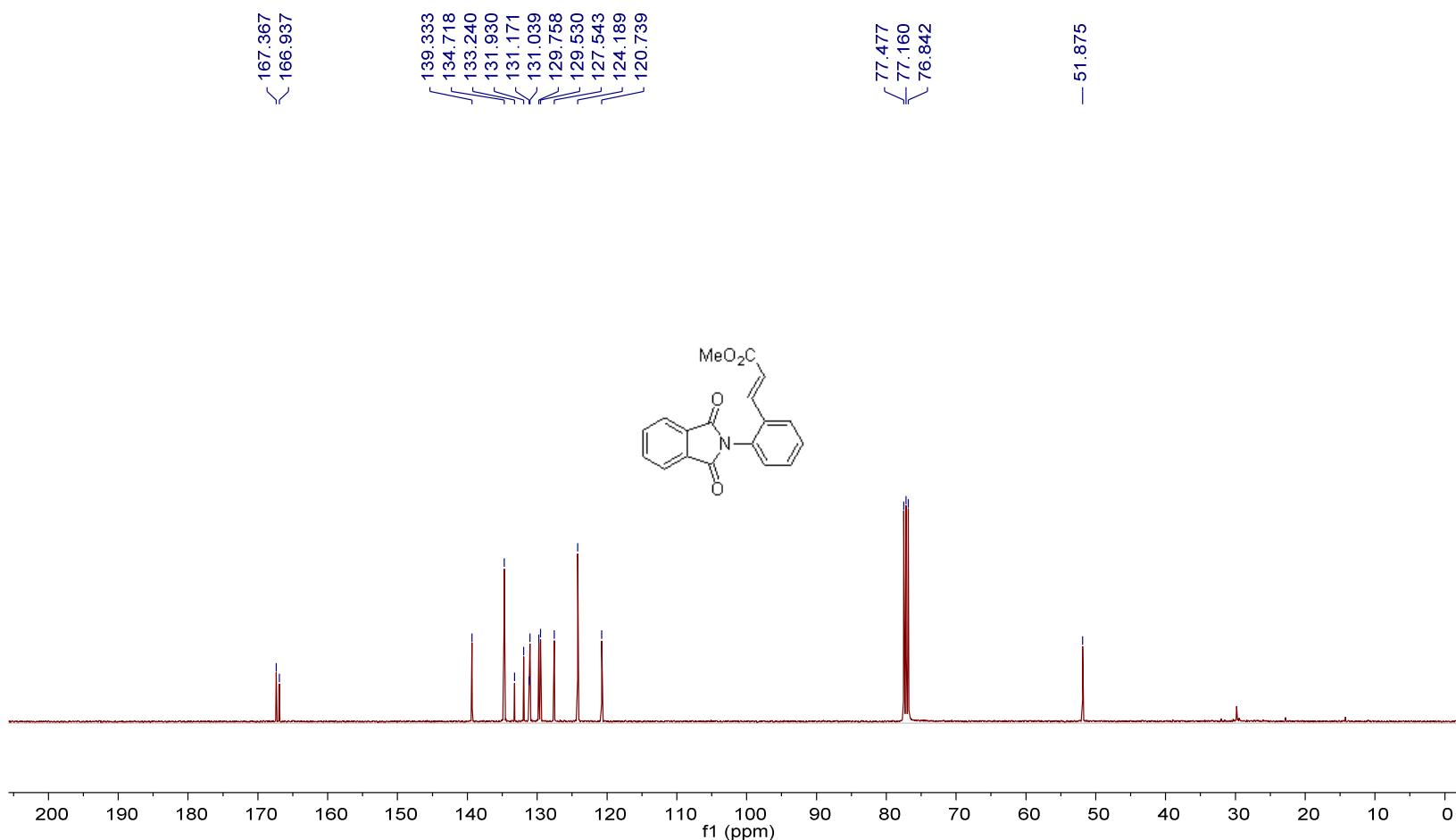




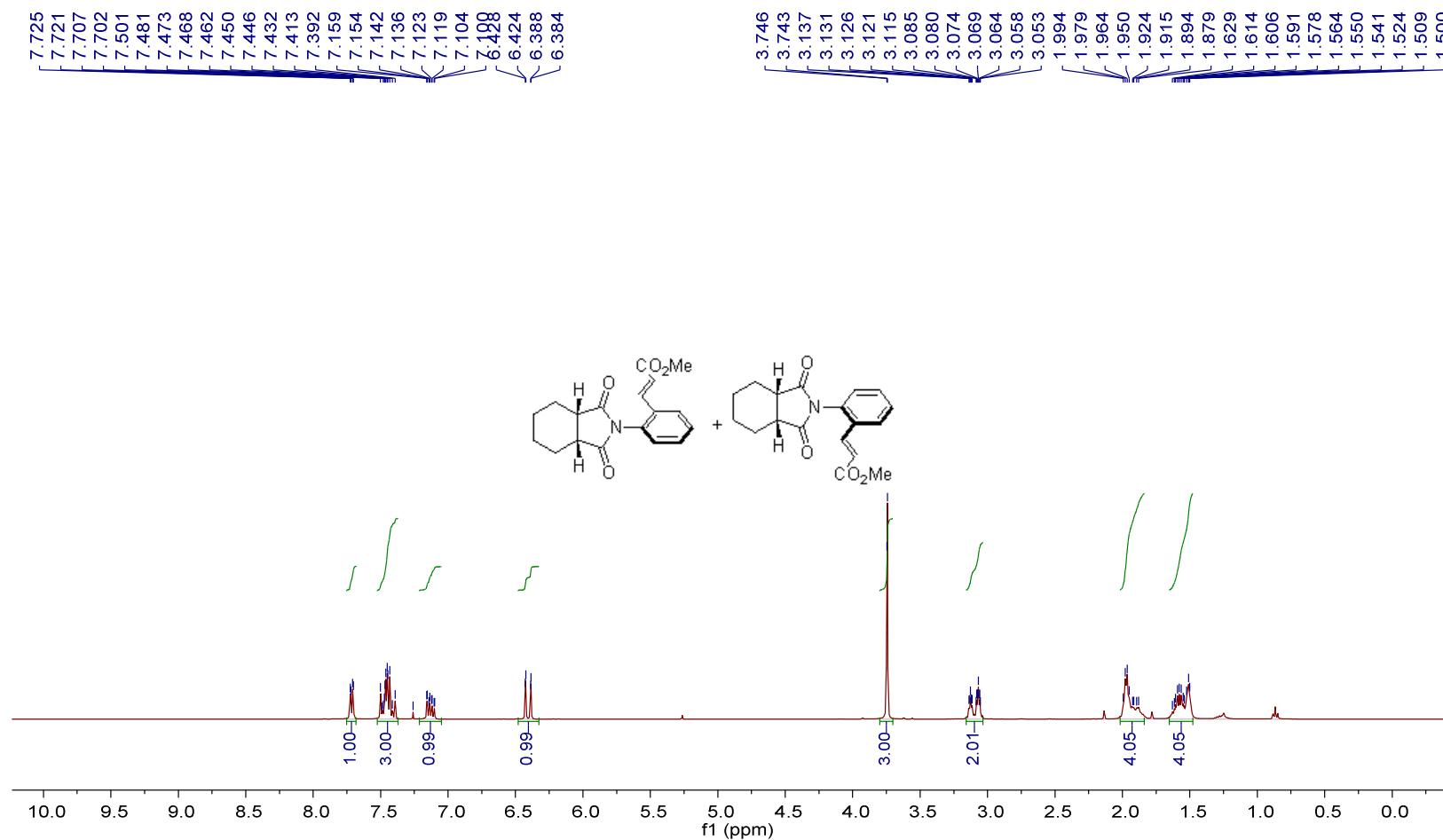


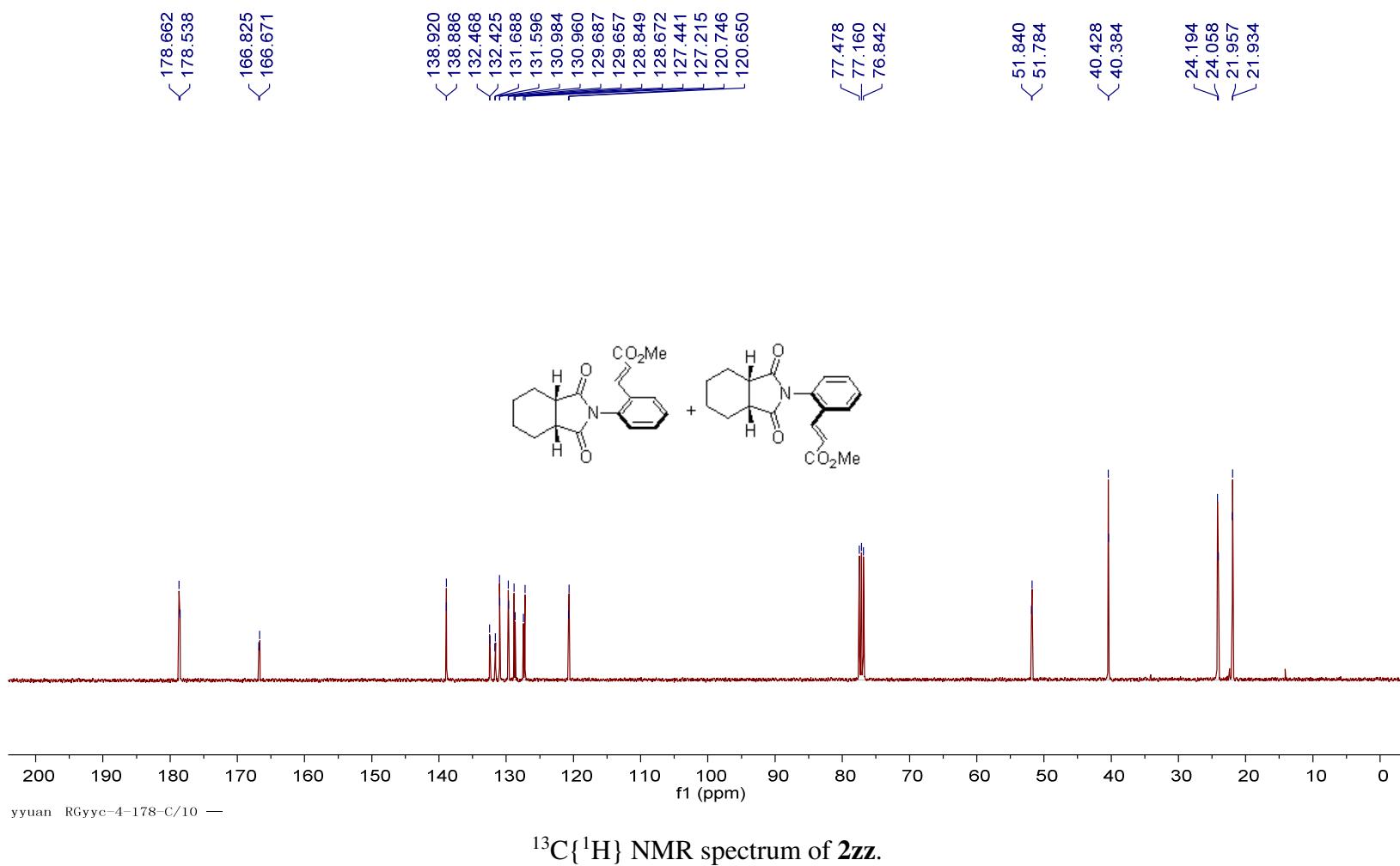


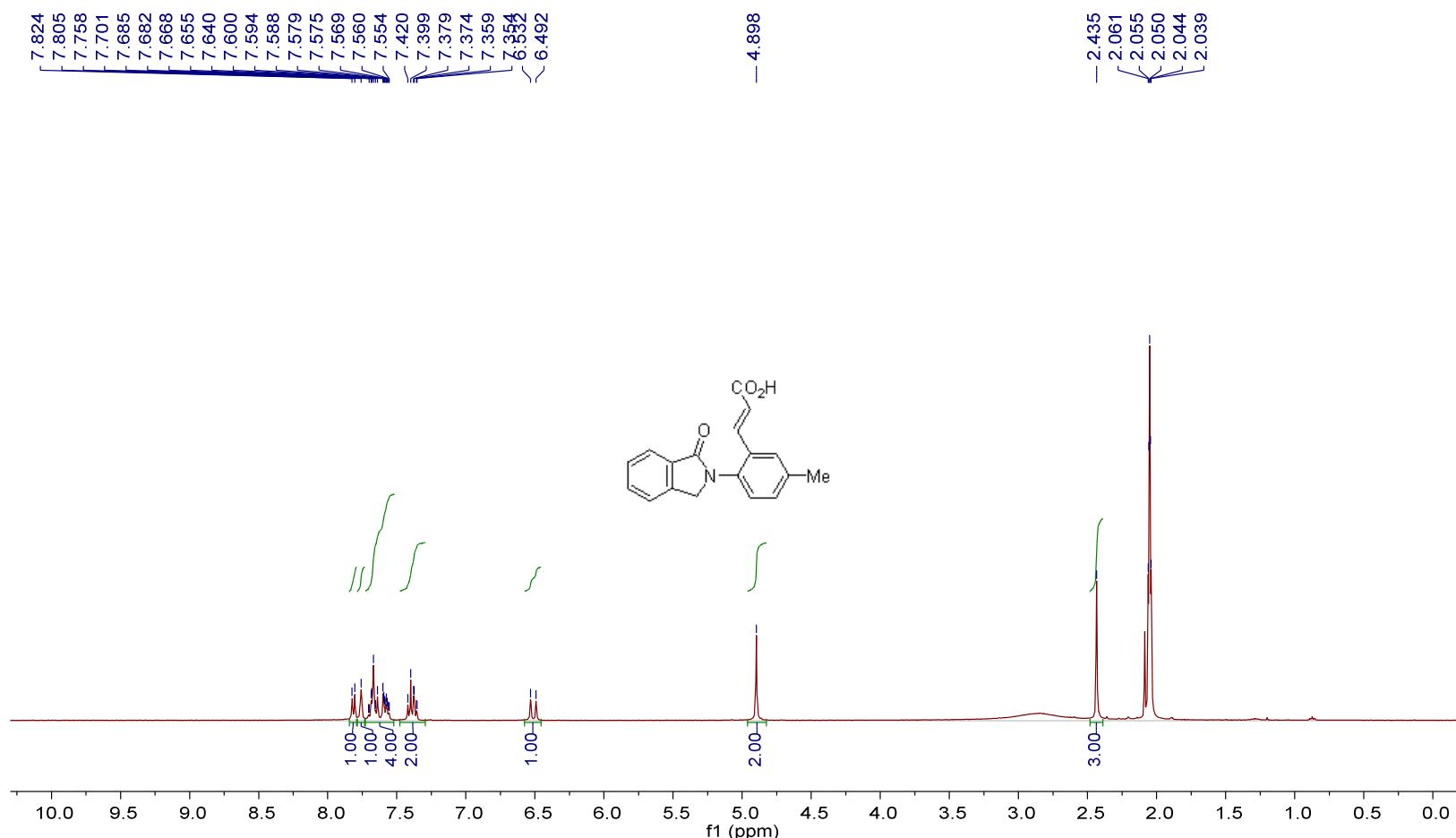
<sup>1</sup>H NMR spectrum of **2z**.

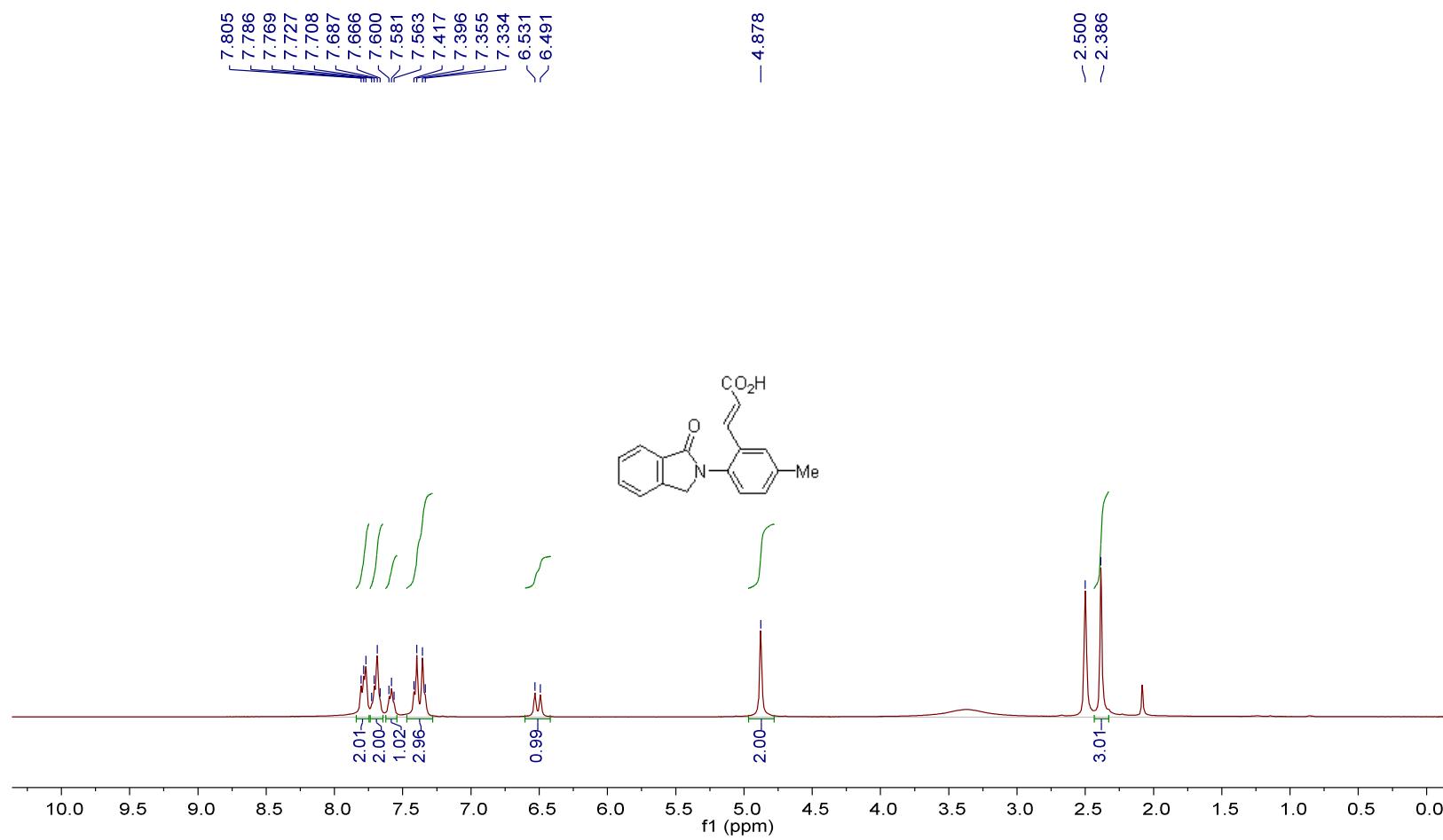


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2z**.

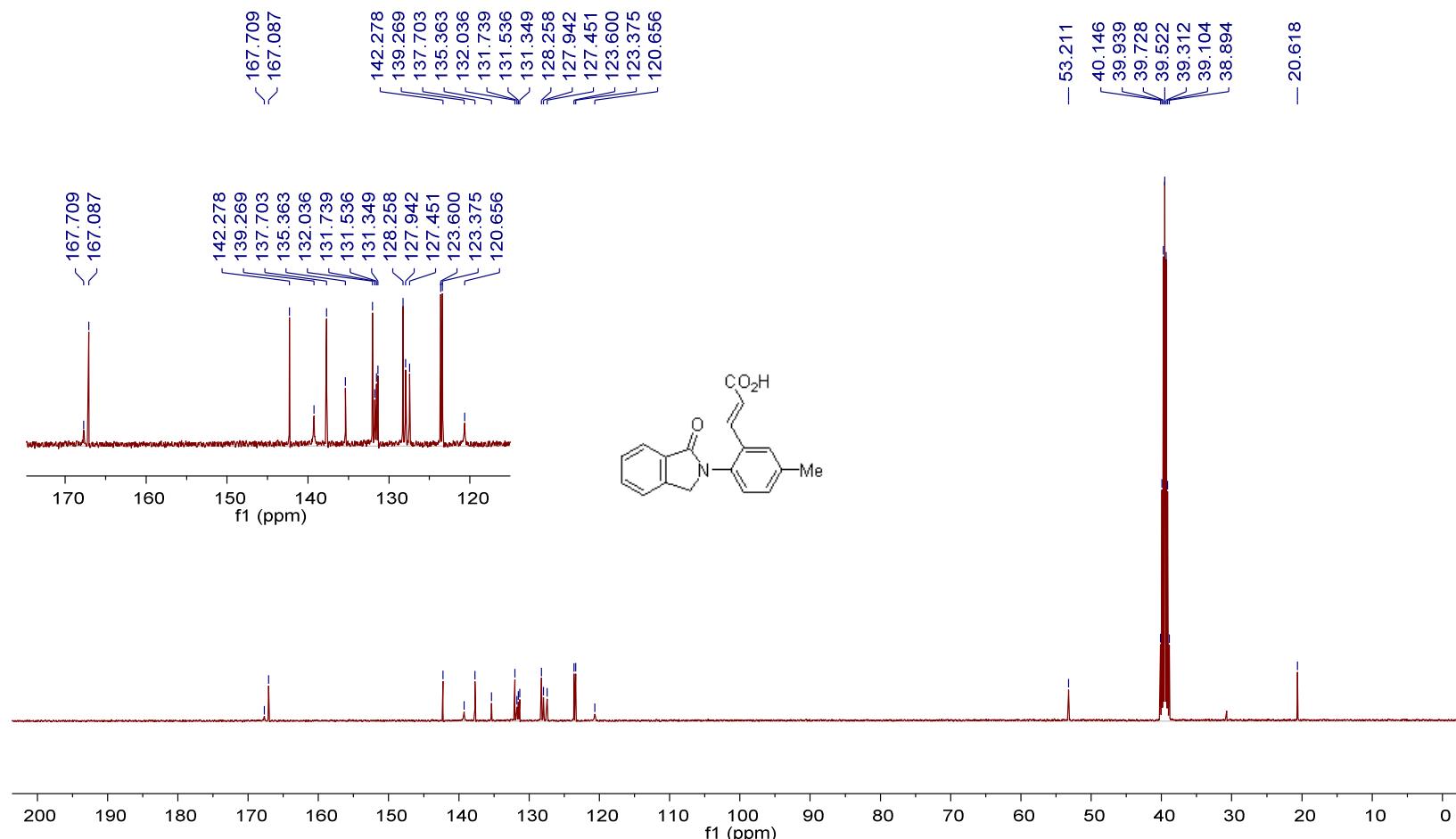




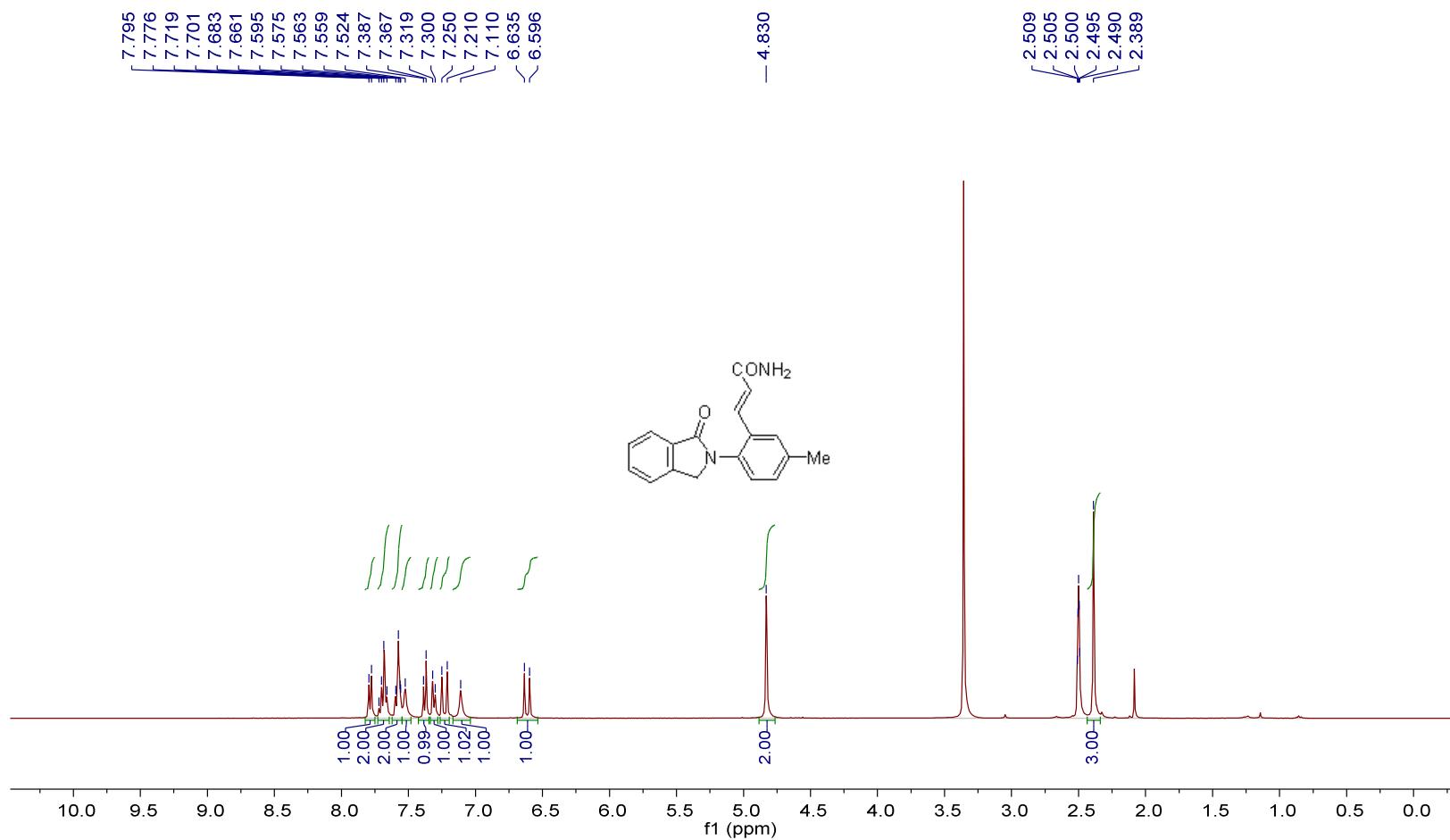


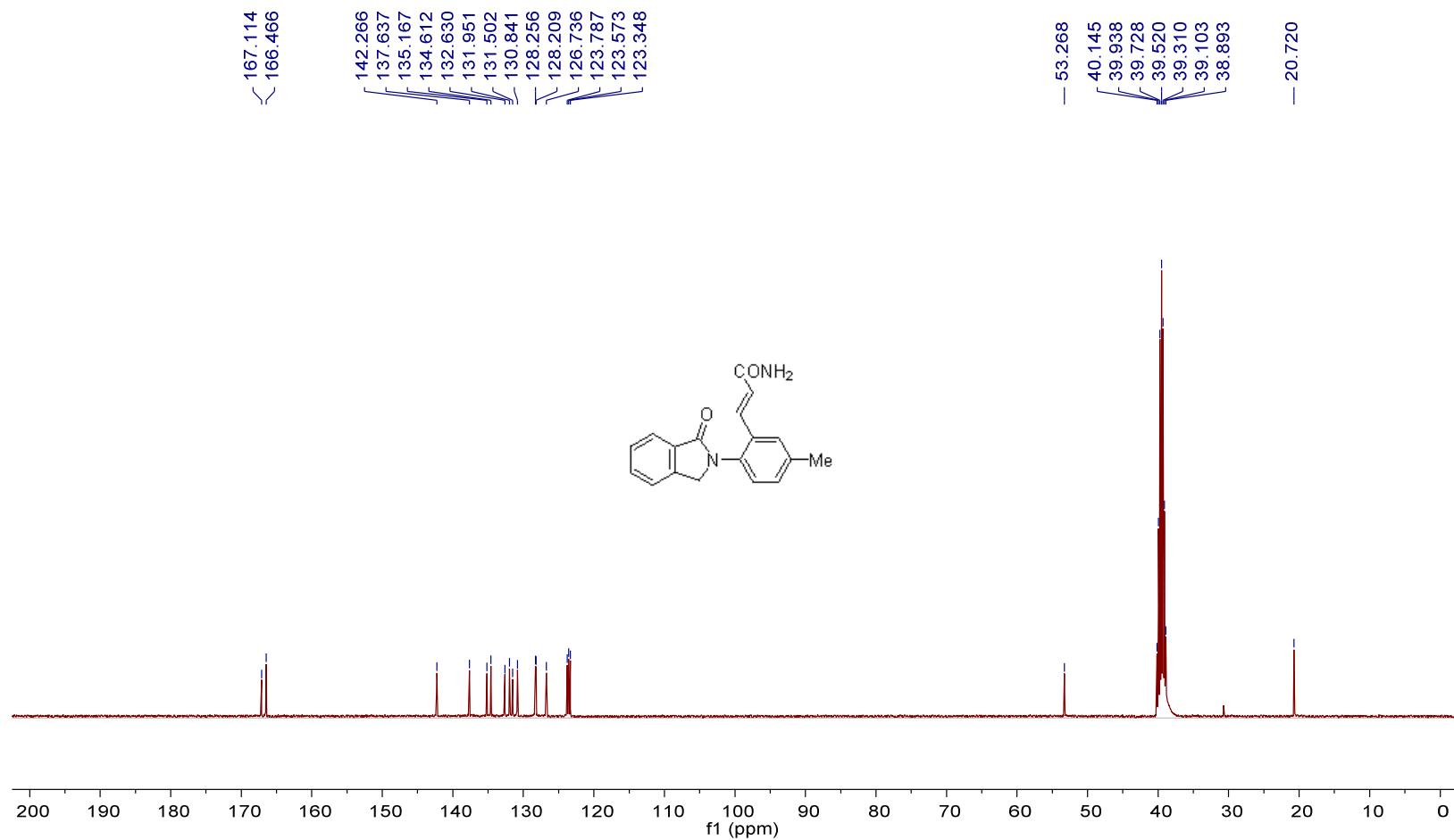


<sup>1</sup>H NMR spectrum of **3**.

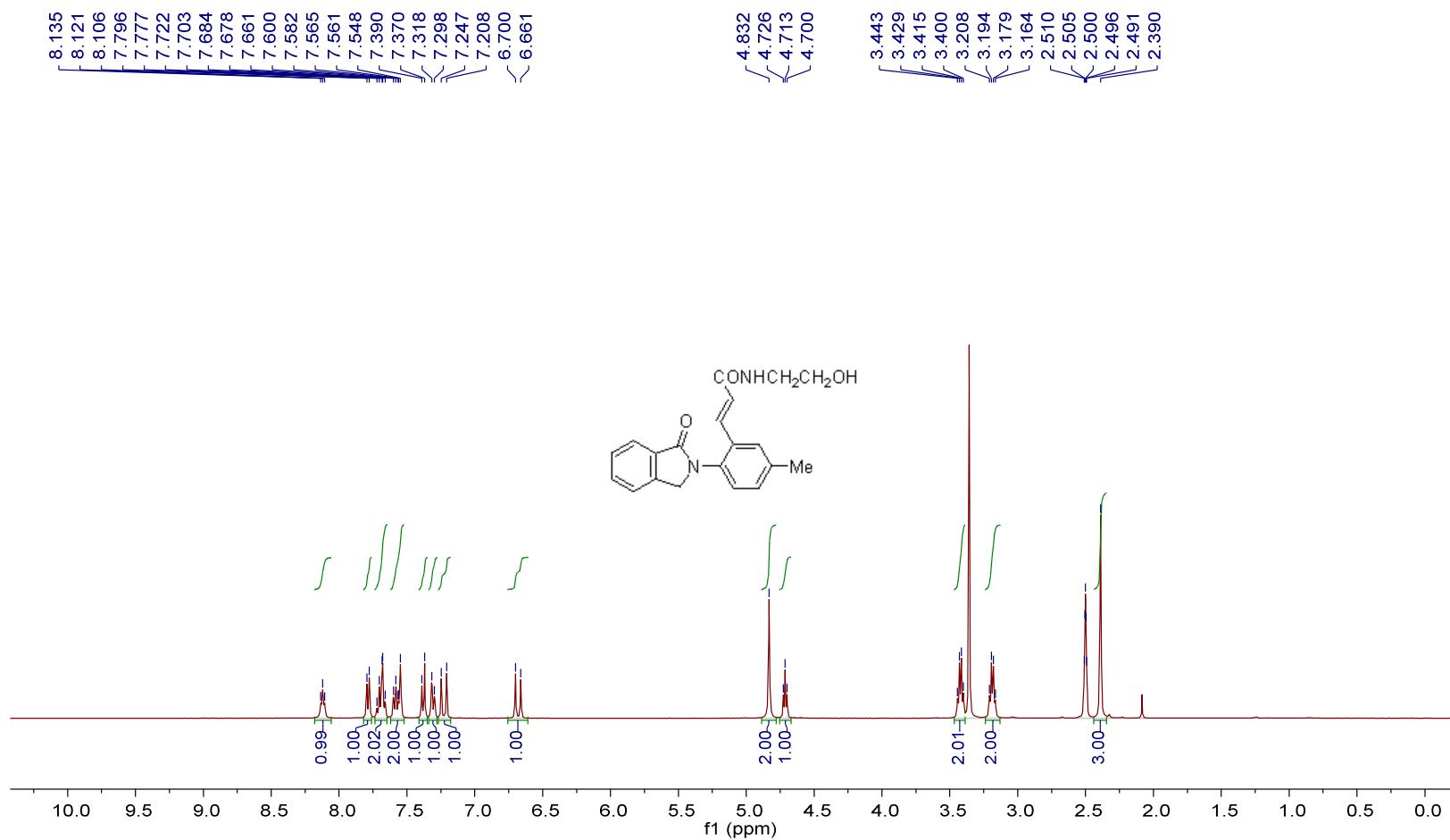


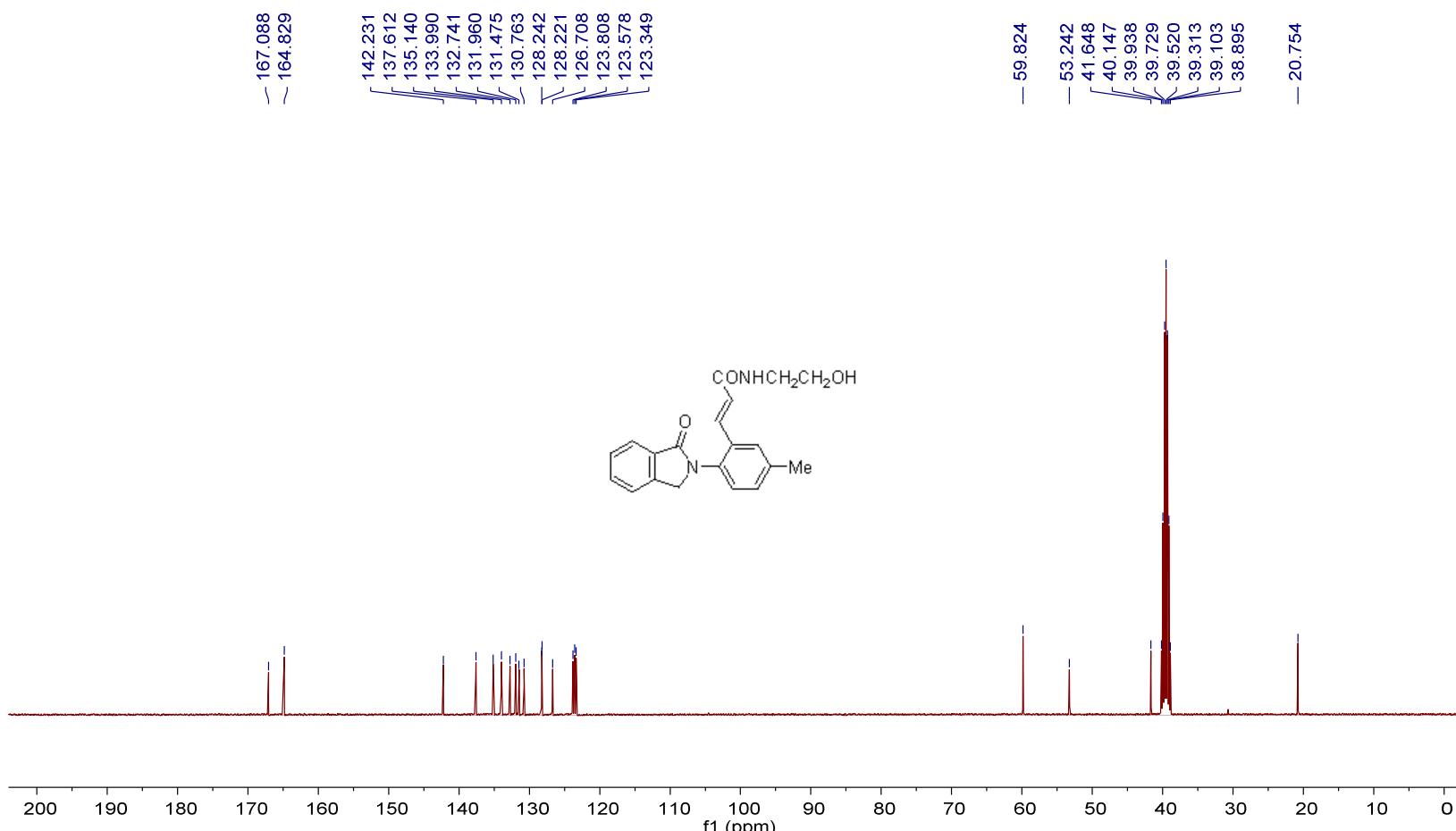
<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **3**.



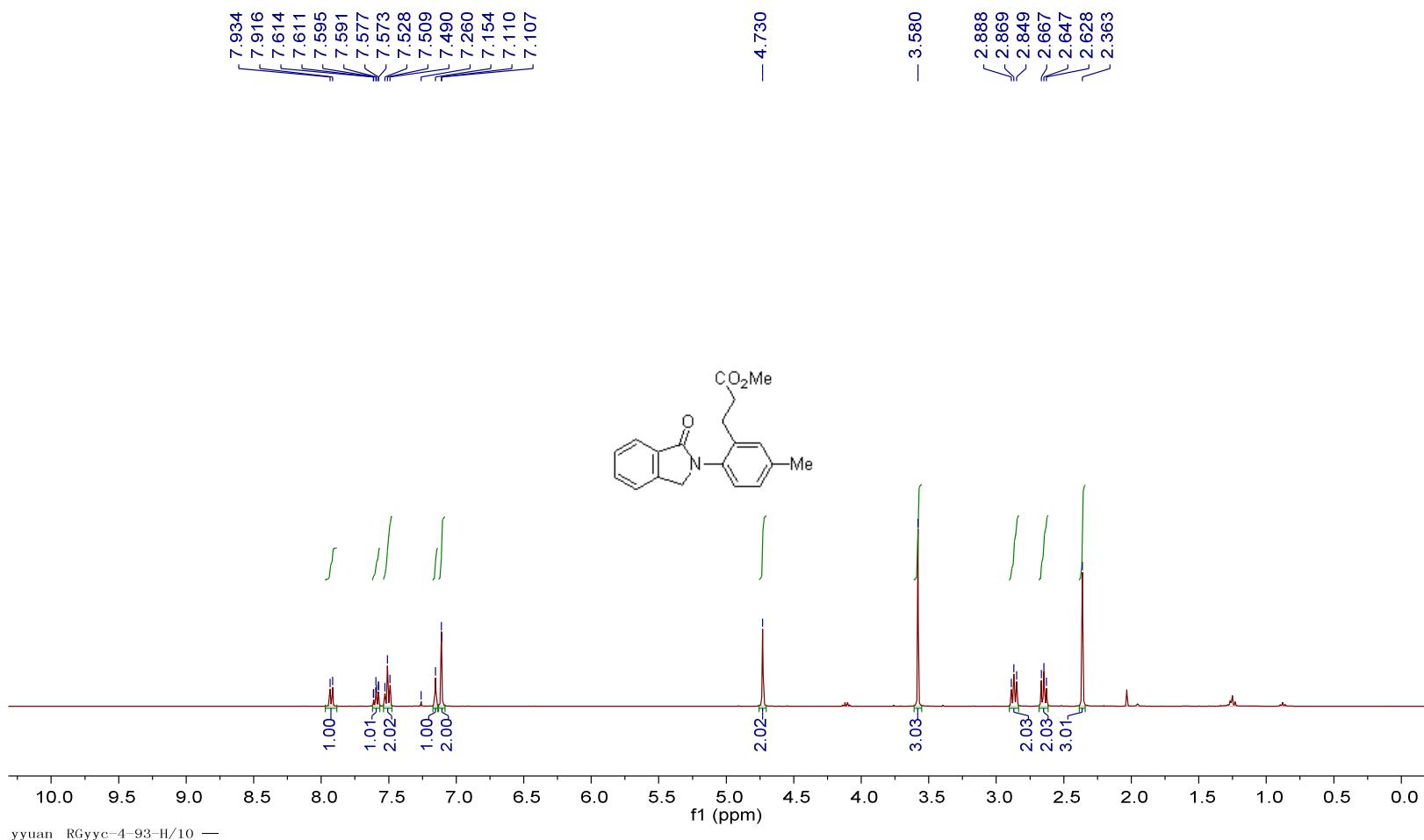


<sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **4**.

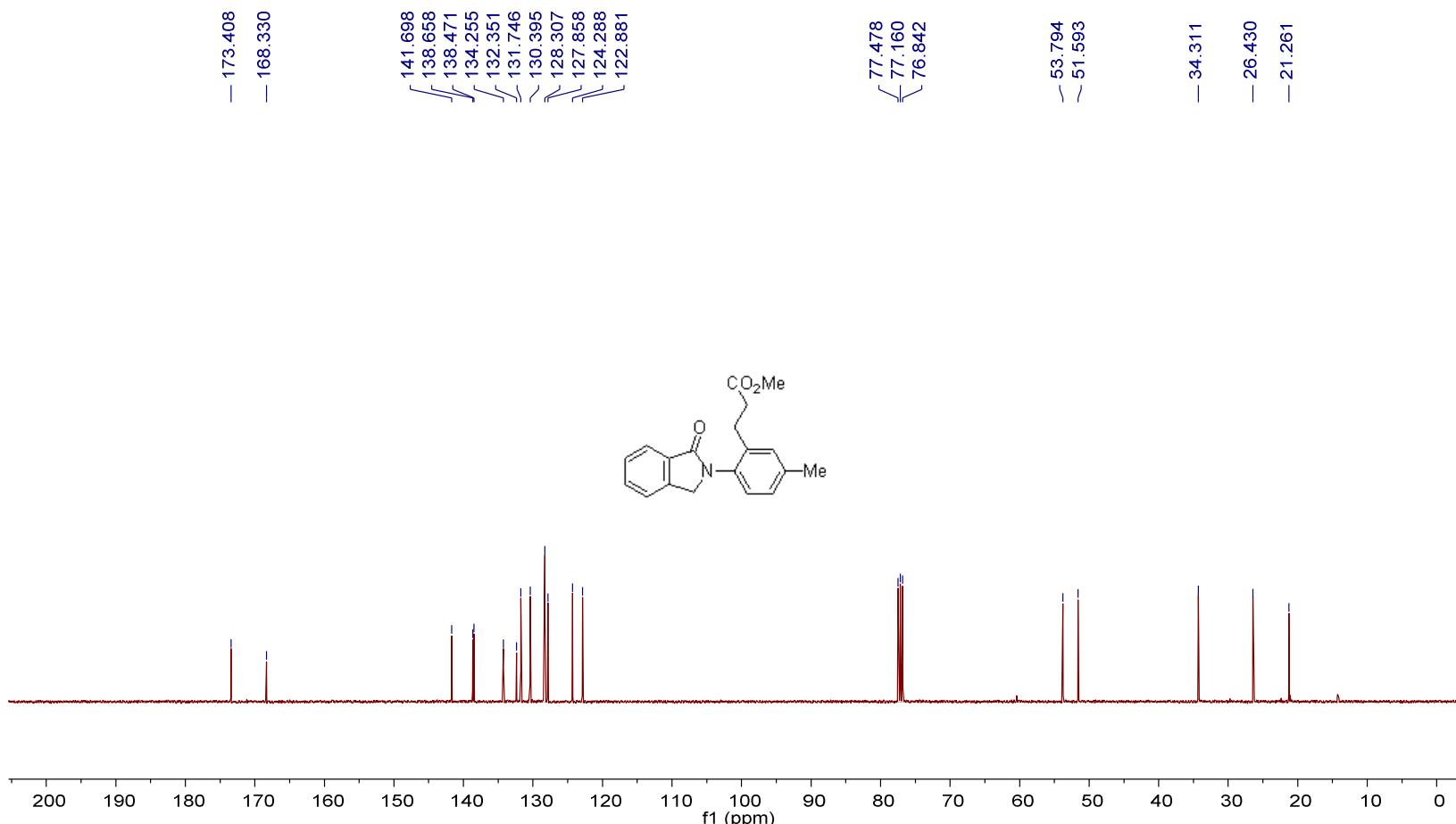


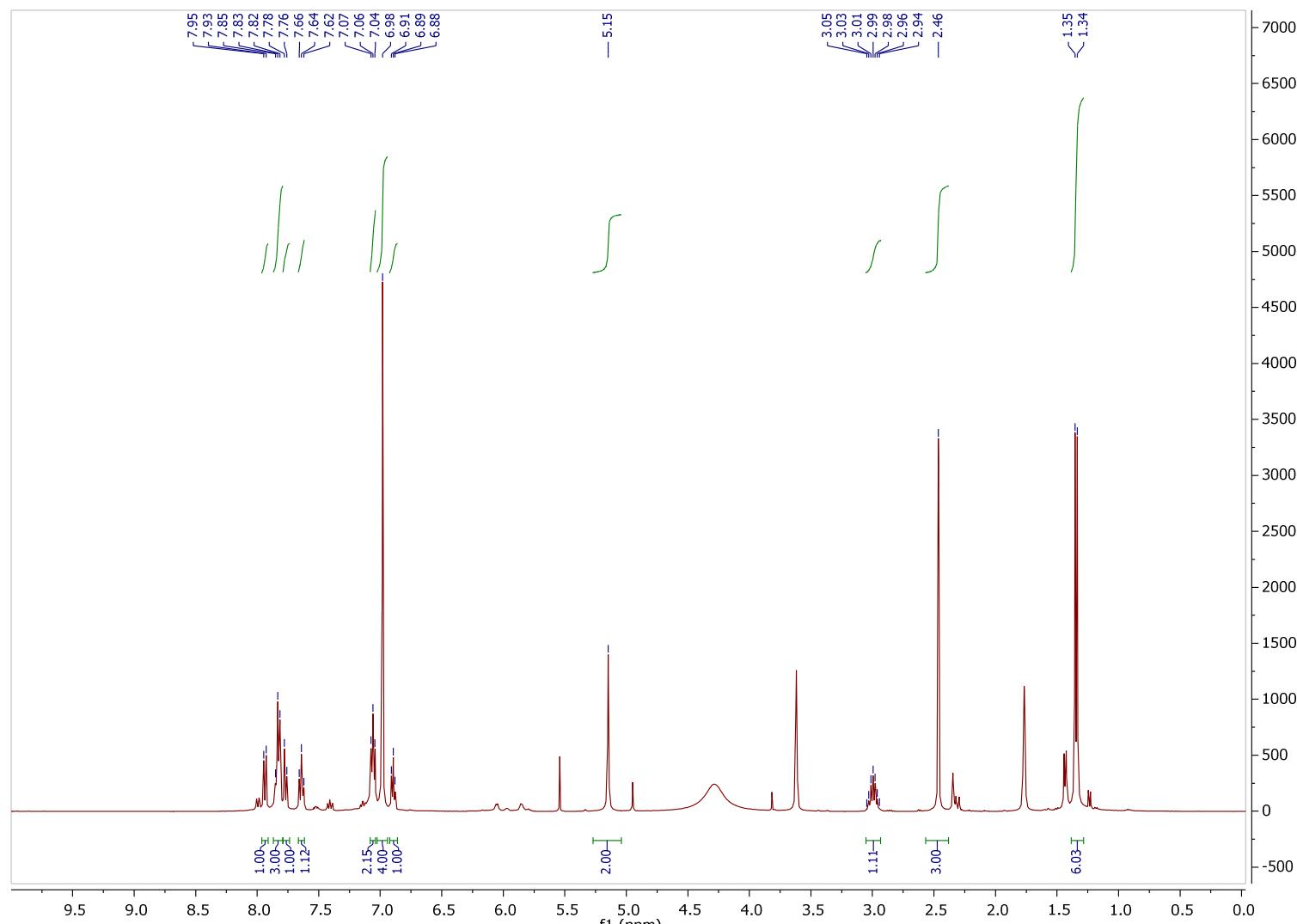


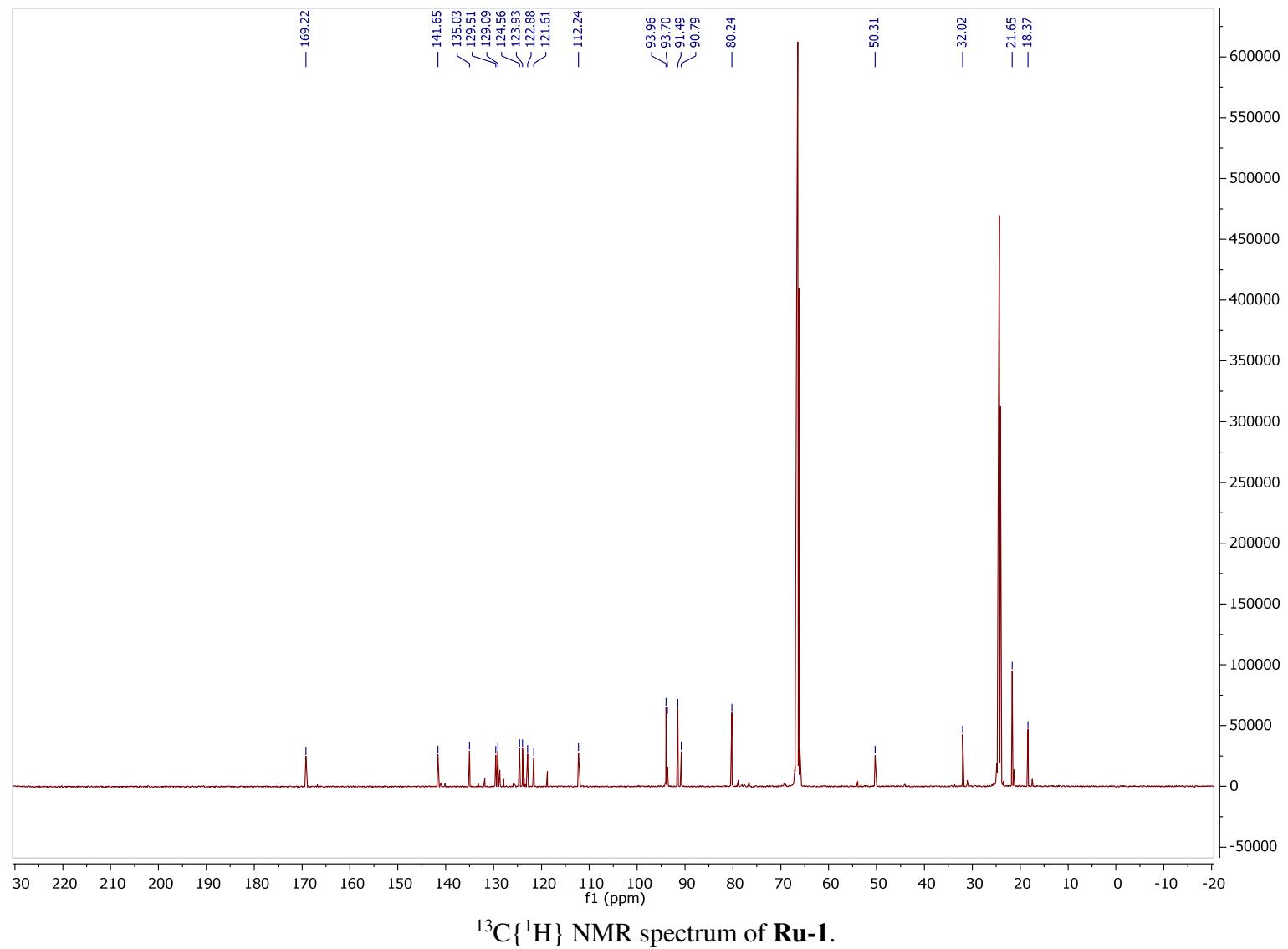
$^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **5**.

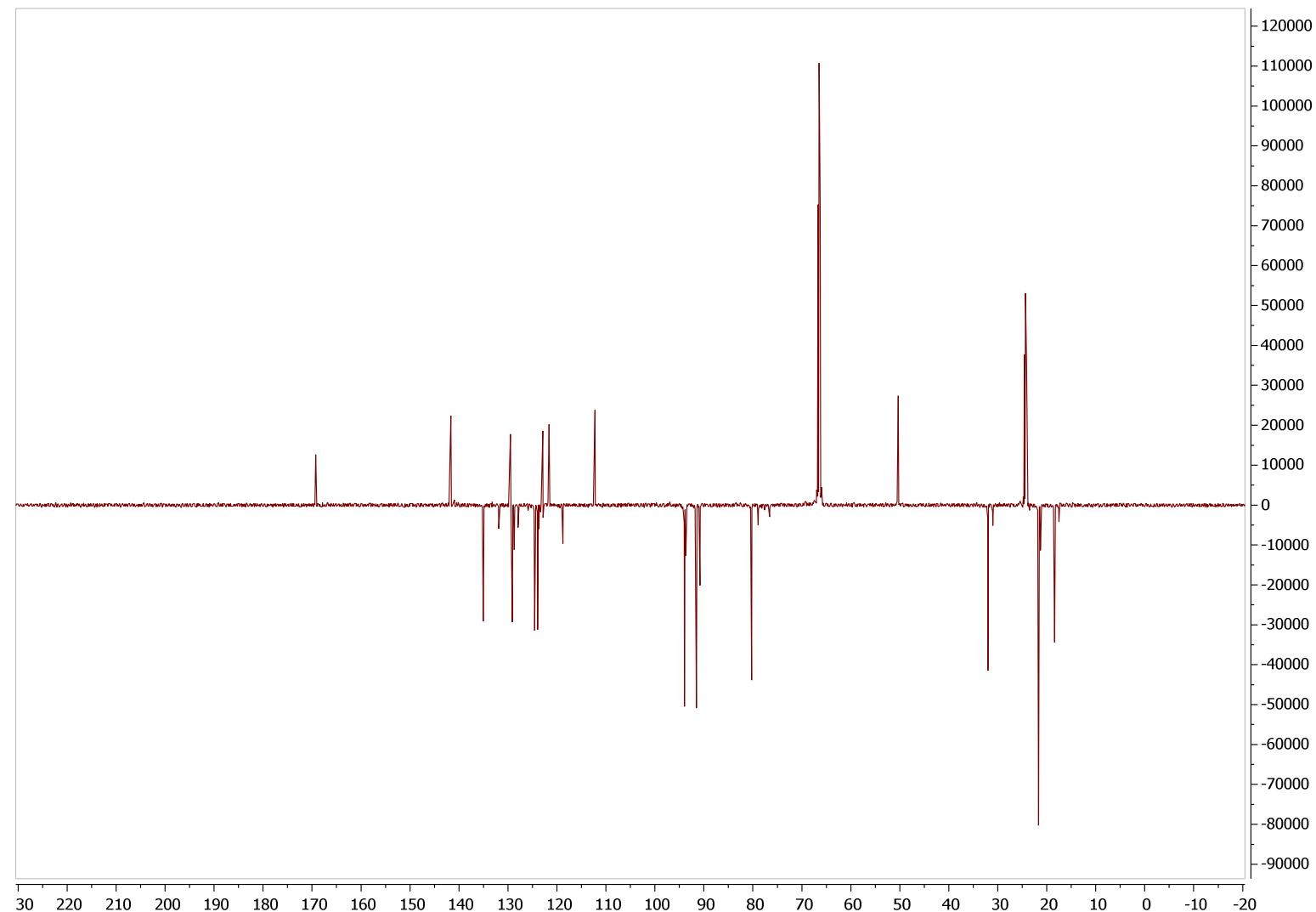


<sup>1</sup>H NMR spectrum of **6**.

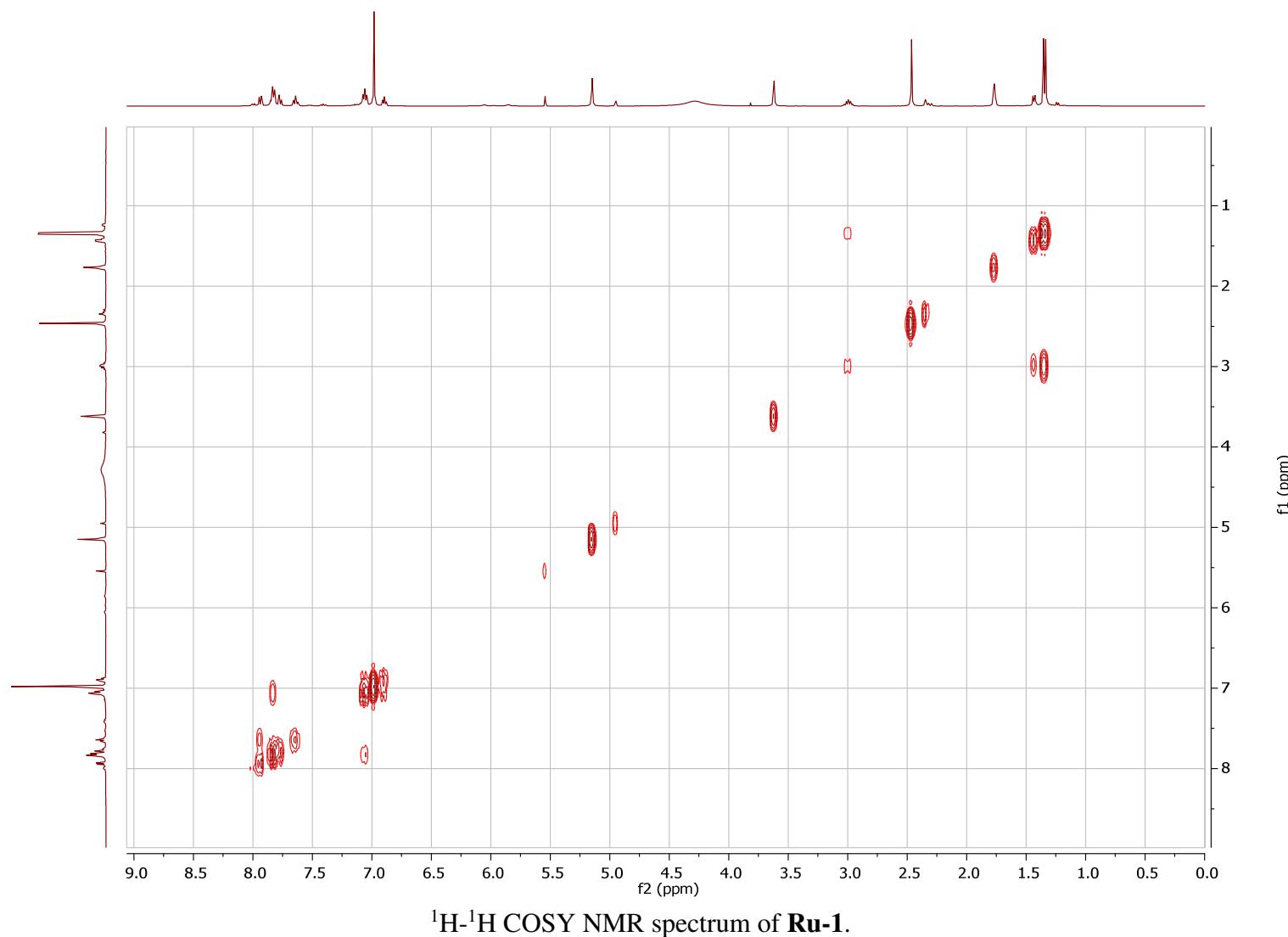


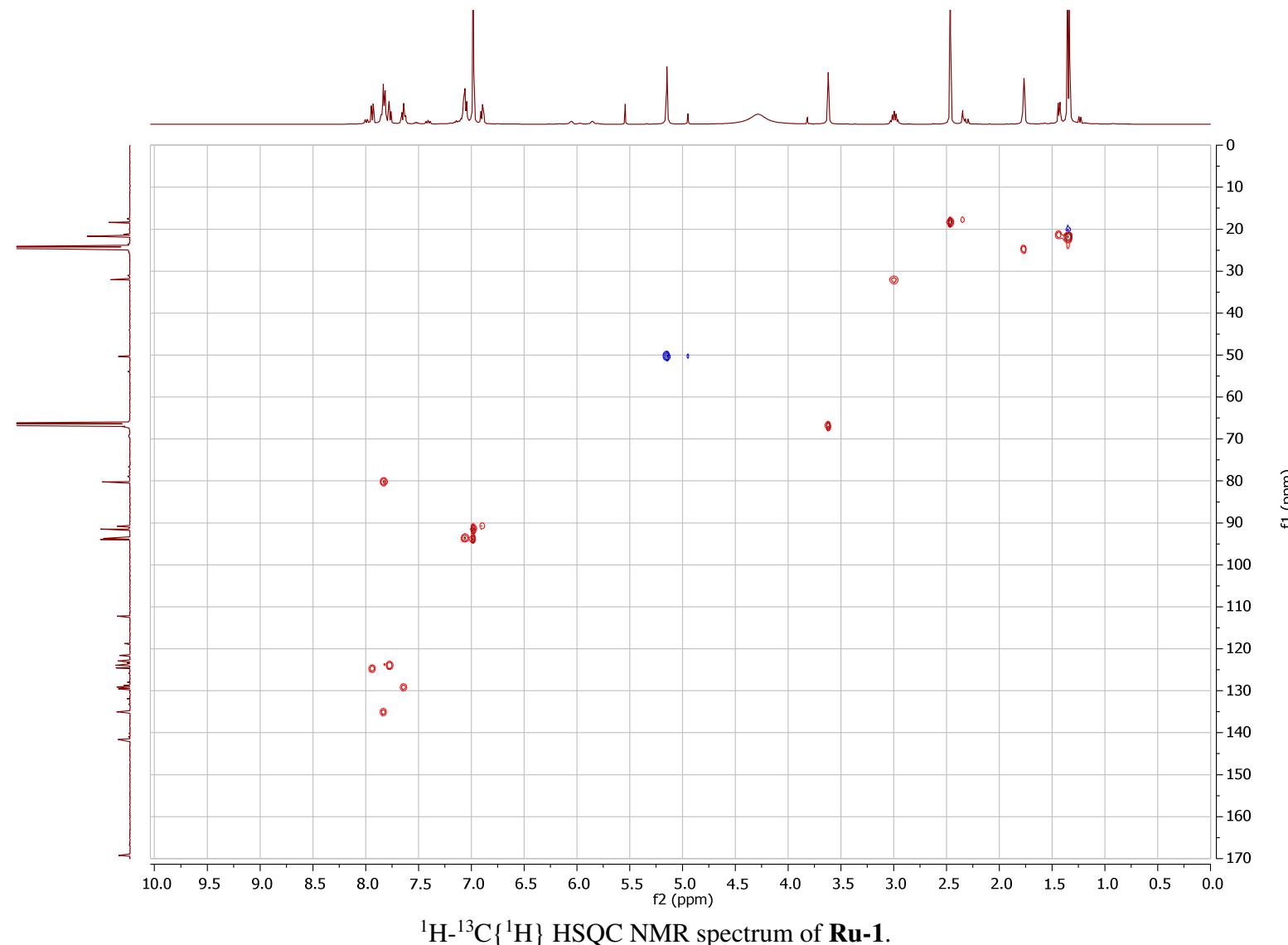


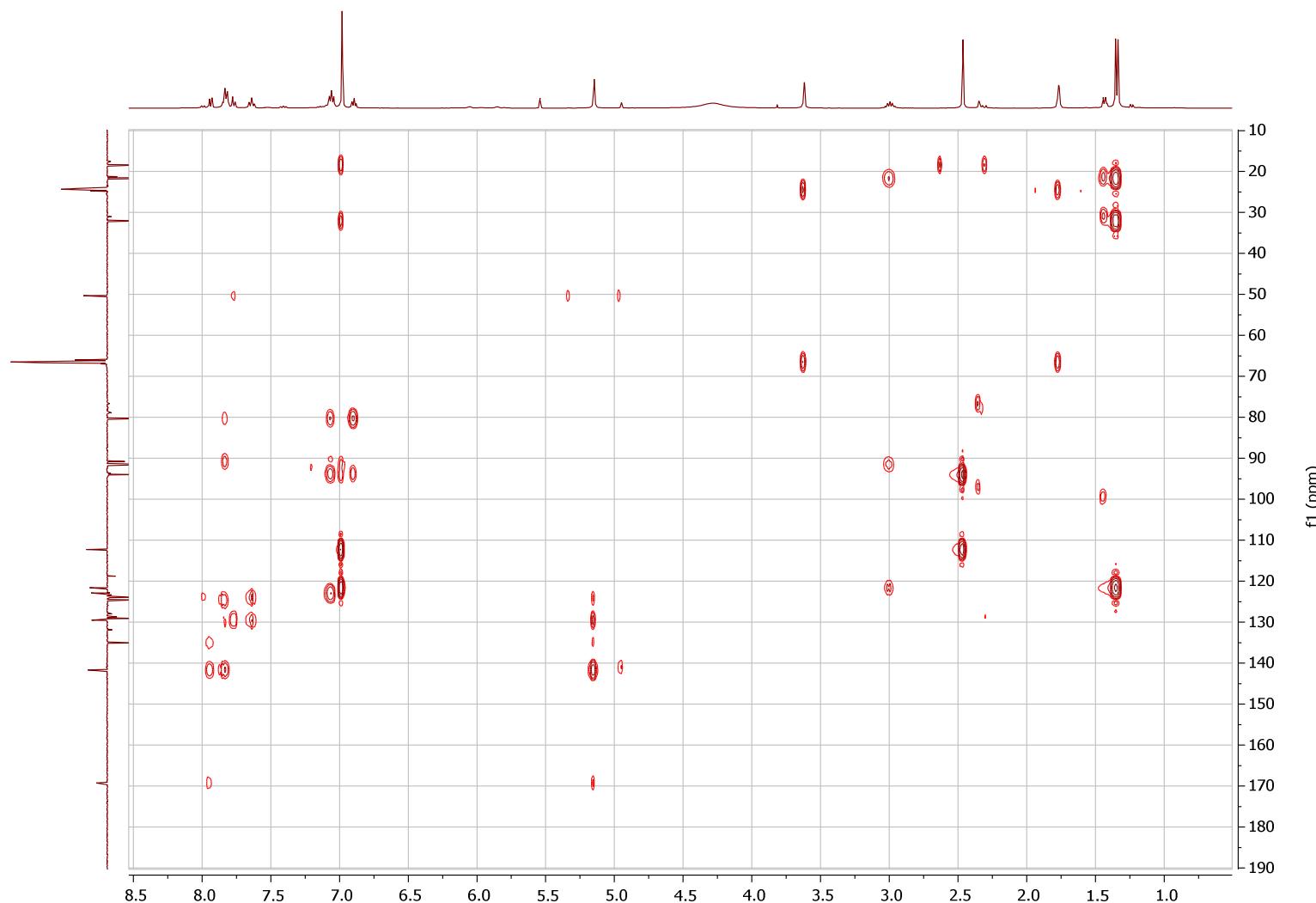




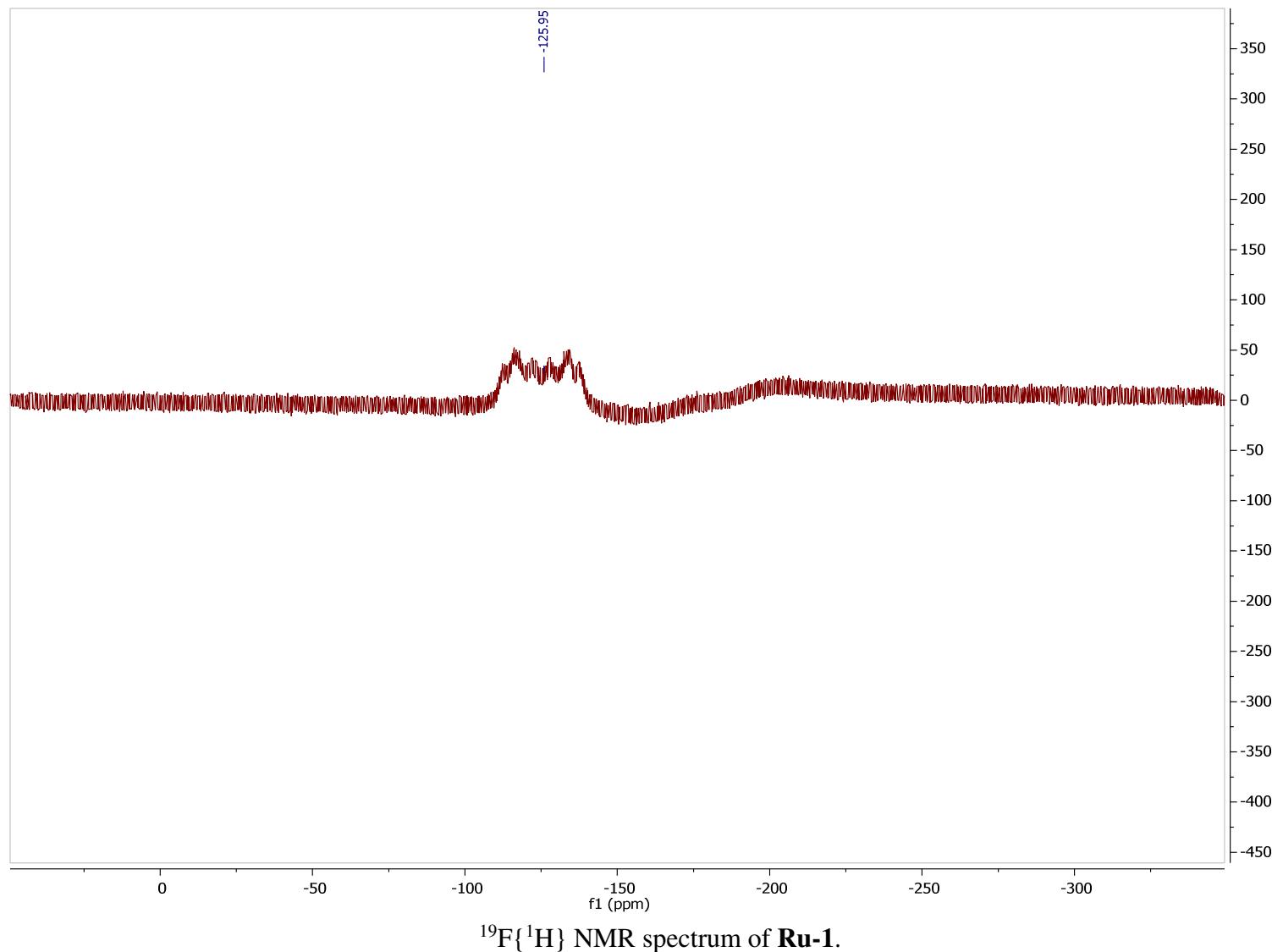
$^{13}\text{C}\{^1\text{H}\}$  JMOD NMR spectrum of **Ru-1**.



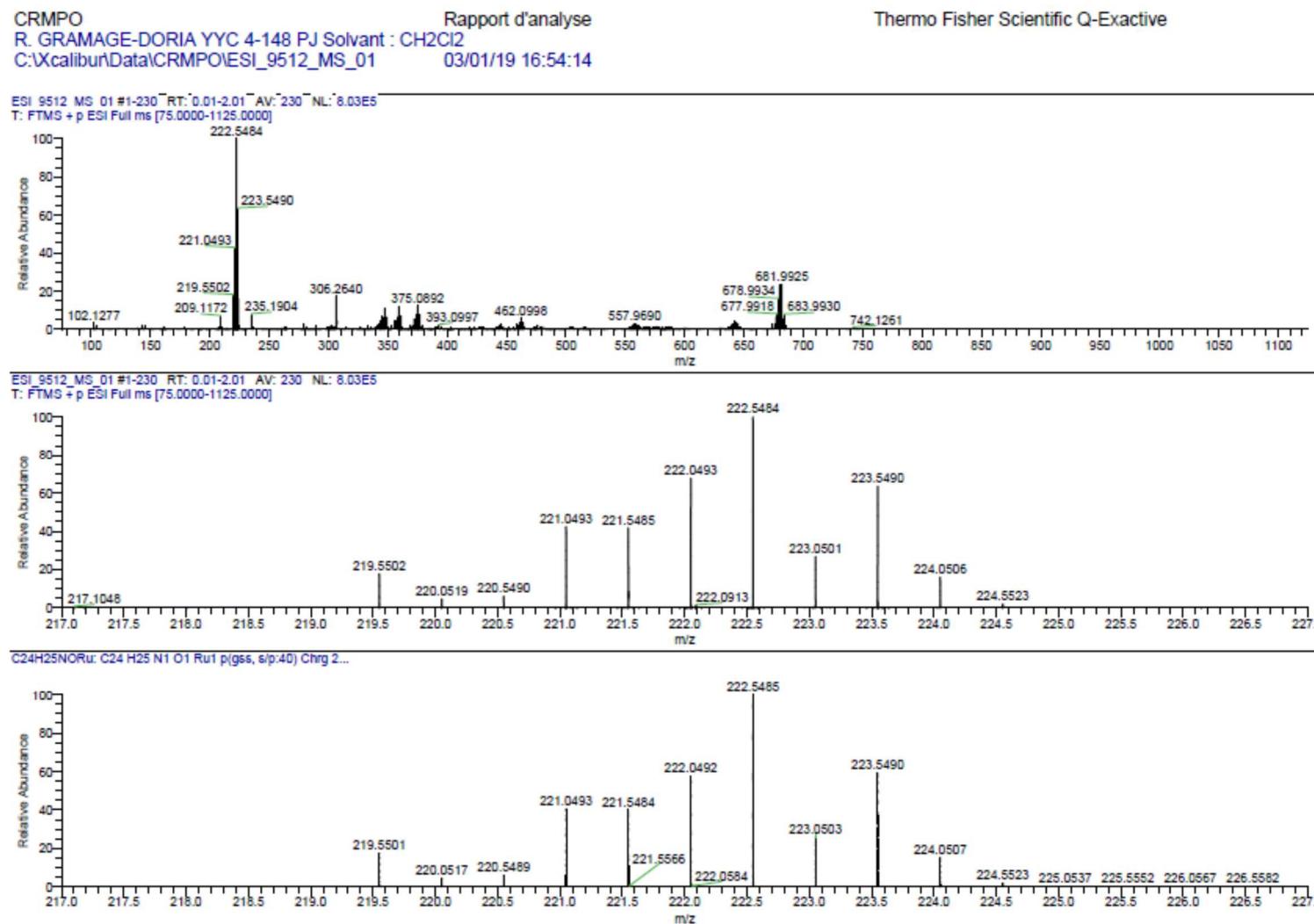




$^1\text{H}$ - $^{13}\text{C}\{^1\text{H}\}$  HMBC NMR spectrum of **Ru-1**.



## 11. HRMS spectra for Ru-1.



CRMPO

Rapport d'analyse

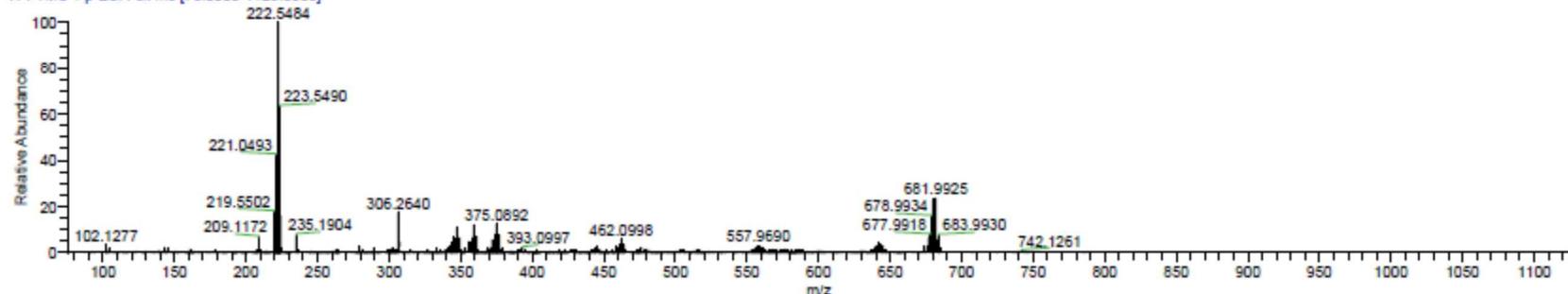
Thermo Fisher Scientific Q-Exactive

R. GRAMAGE-DORIA YYC 4-148 PJ Solvant : CH<sub>2</sub>Cl<sub>2</sub>

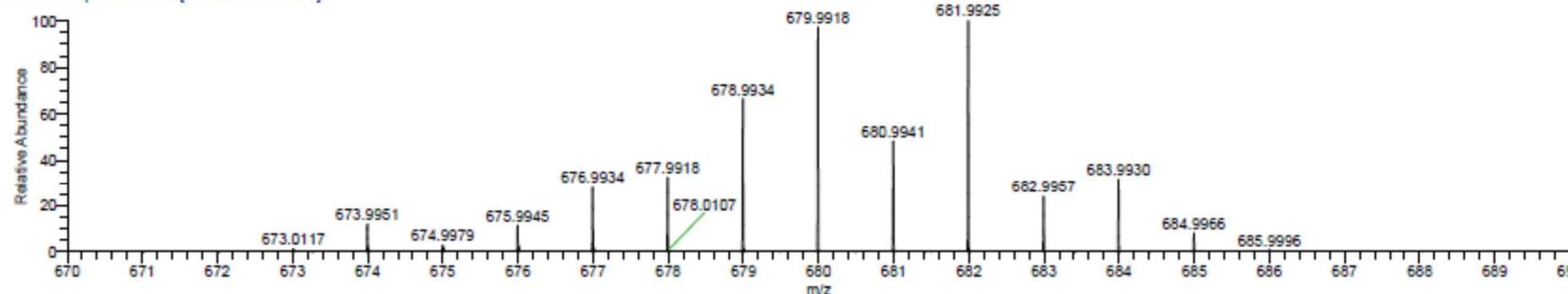
C:\Xcalibur\Data\CRMPO\ESI\_9512\_MS\_01

03/01/19 16:54:14

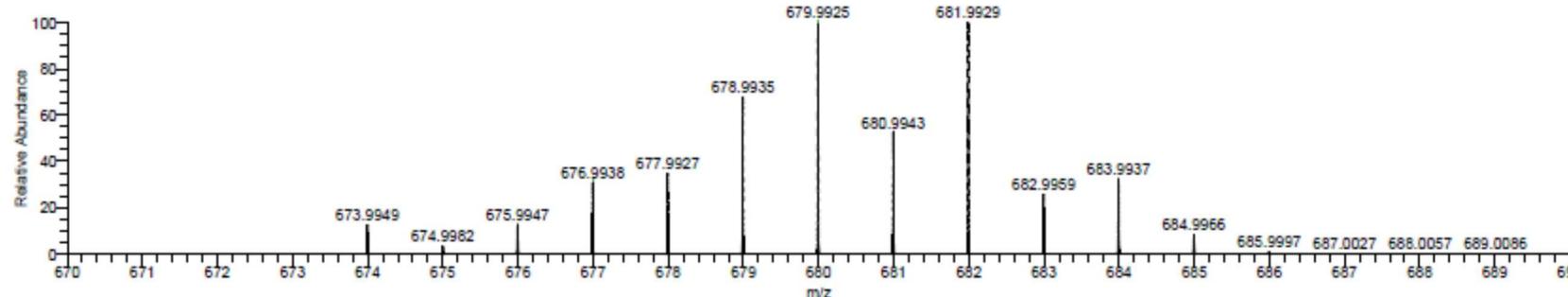
ESI\_9512\_MS\_01 #1-230 RT: 0.01-2.01 AV: 230 NL: 8.03E5  
T: FTMS + p ESI Full ms [75.0000-1125.0000]



ESI\_9512\_MS\_01 #1-230 RT: 0.01-2.01 AV: 230 NL: 1.90E5  
T: FTMS + p ESI Full ms [75.0000-1125.0000]



C24H25NORuSbF6: C24 H25 N1 O1 Ru1 Sb1 F6 p(gss, s/p...)



CRMPO

R. GRAMAGE-DORIA YYC 4-148 PJ Solvant : C4H8O

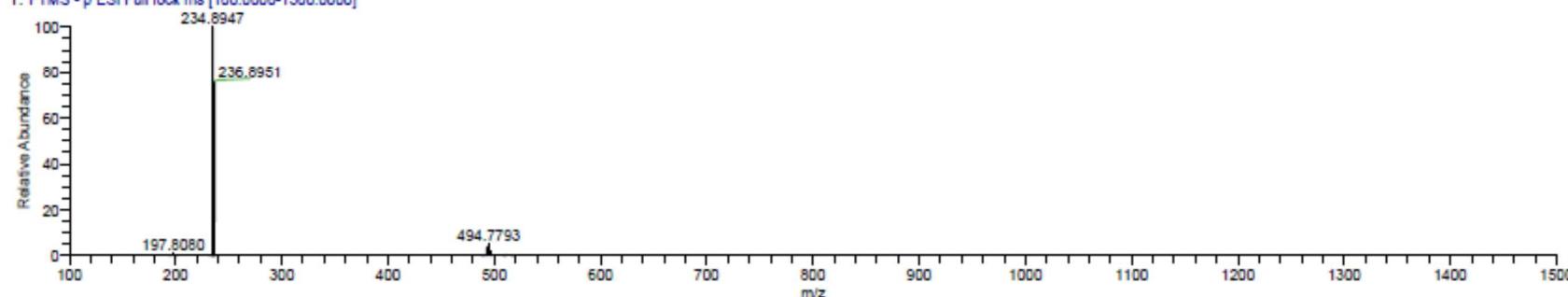
C:\Xcalibur\Data\CRMPO\ESI\_9512\_MS\_02

Rapport d'analyse

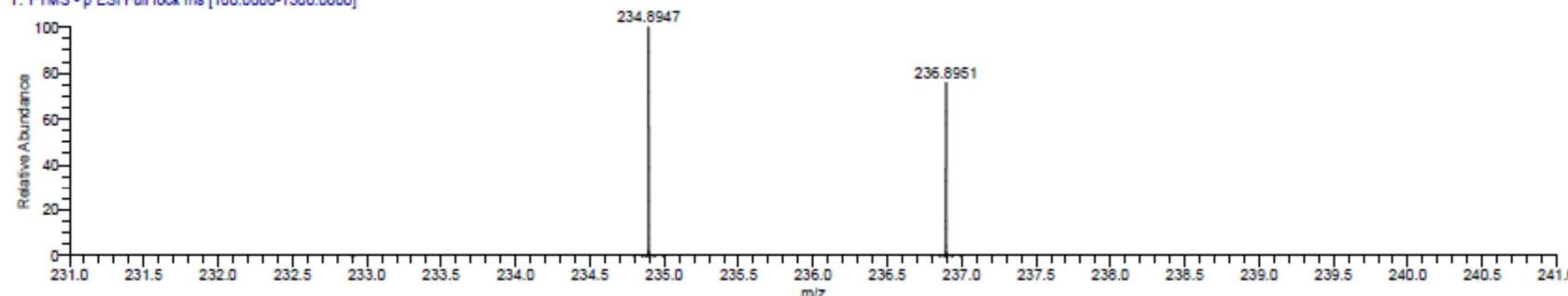
03/01/19 19:26:29

Thermo Fisher Scientific Q-Exactive

ESI\_9512\_MS\_02 #1-230 RT: 0.01-2.01 AV: 230 NL: 1.47E7  
T: FTMS - p ESI Full lock ms [100.0000-1500.0000]



ESI\_9512\_MS\_02 #1-230 RT: 0.01-2.01 AV: 230 NL: 1.47E7  
T: FTMS - p ESI Full lock ms [100.0000-1500.0000]



SbF6. Sb1 F6 p(gss, s/p:40) Chrg 1R: 142400 Res.Pw...

