

## SUPPORTING INFORMATION

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Transition Metal-Free N-Arylation of Isatins with Diaryliodonium Salts

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## S-I. General Information

**Materials.** All commercially available chemicals and solvents of high purity were used without purification unless otherwise stated. Toluene was distilled from sodium/benzophenone and kept under argon over MS 4Å.

**Chromatography.** Flash-column chromatography was performed using silica gel (0.040–0.063 mm). Analytical thin-layer chromatography (TLC) was carried out on silica gel 60 F254 plates that were visualized by exposure to UV light.

**Instrumentation.**  $^1\text{H}$ ,  $^{13}\text{C}\{\text{H}\}$  and  $^{19}\text{F}$  NMR were recorded at 400, 100 and 376 MHz, respectively, for solutions in  $\text{CDCl}_3$ . Chemical shifts are referenced relative to the signal of  $\text{CDCl}_3$  and quoted in  $\delta$  (ppm). Coupling constants ( $J$ ) are reported in Hz. Multiplicities are abbreviated as follows: singlet (s), doublet (d), triplet (t), quartet (q), doublet – doublet (dd), doublet – triplet (dt), triplet – doublet (td) and multiplet (m). High resolution mass spectra (HRMS) were measured using electrospray ionization (ESI) on an Orbitrap spectrometer. Compound **8** was analyzed using GC-MS Thermo ISQ instrument (capillary column Varian VF-5ms, length – 30 m, I.D. – 0.25 mm, df – 0.25  $\mu\text{m}$ , carrier gas – helium, injector temperature – 270  $^\circ\text{C}$ , the initial chromatograph oven temperature – 40  $^\circ\text{C}$ , heating rate 15  $^\circ\text{C}/\text{min}$  to 300  $^\circ\text{C}$ , followed by isotherm for 10 minutes; MS parameters: electron ionization (EI) with 70 eV ionization energy, source temperature – 230  $^\circ\text{C}$ , mass range 10-800 Da, scan rate 2 scan/s). The sample was injected into the system as a chloroform solution.

**Melting points** were determined in open capillaries and are uncorrected.

**Yields** refer to isolated and analytically pure compounds.

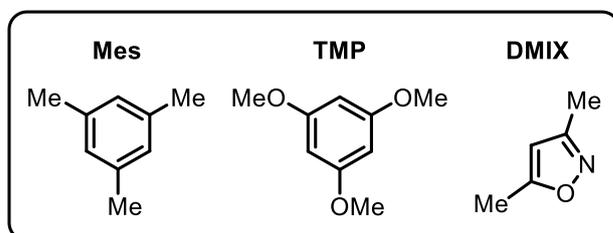
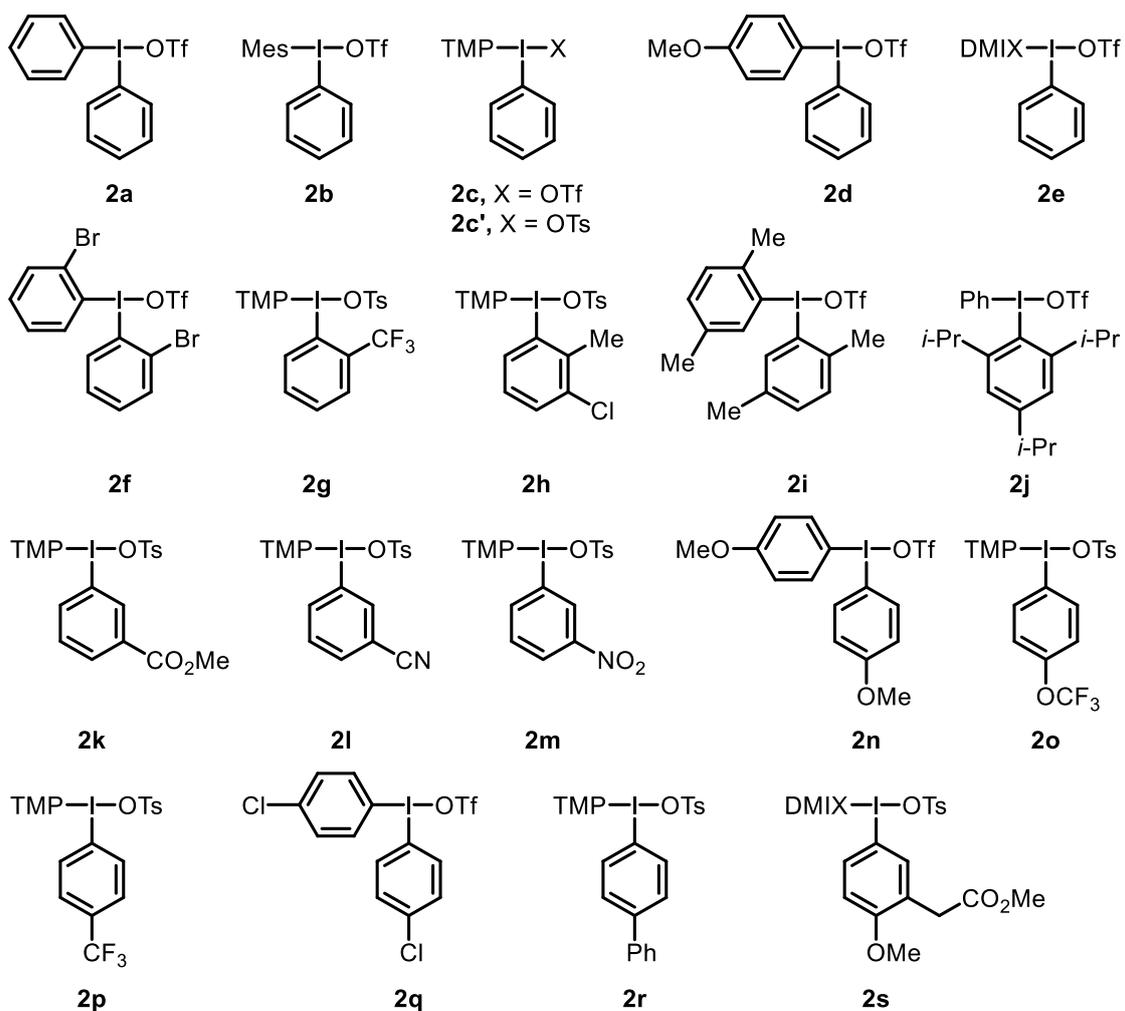
## S-II. Preparation of Diaryliodonium Salts

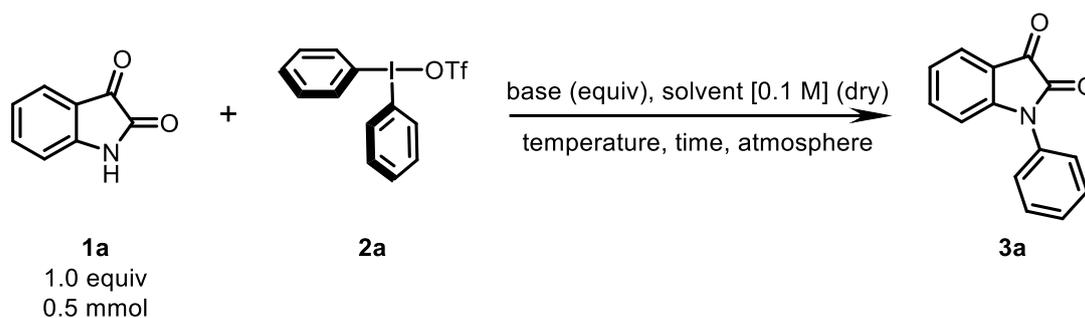
Symmetrical diaryliodonium salts **2a**<sup>1</sup>, **2f**<sup>2</sup>, **2i**<sup>1</sup>, **2n**<sup>3</sup>, **2q**<sup>4</sup> were synthesized according to previously reported procedures. Unsymmetrical diaryliodonium salts **2b**<sup>1</sup>, **2c**<sup>5</sup>, **2d**<sup>1</sup>, **2e**<sup>6</sup>, **2g-2h**<sup>7</sup>, **2j**<sup>8</sup>, **2k**<sup>7</sup>, **2l**<sup>5</sup>, **2m**<sup>9</sup>, **2o**<sup>7</sup>, **2p**<sup>5</sup>, **2r**<sup>7</sup>, **2s**<sup>10</sup> were synthesized according to previously reported procedures. **2c**<sup>5</sup> was synthesized according to a previously reported procedure followed by anion exchange with NaOTf.

Characterization data obtained for these compounds matched those previously reported in the literature:

diphenyliodonium trifluoromethanesulfonate (**2a**)<sup>3</sup>, mesityl(phenyl)iodonium trifluoromethanesulfonate (**2b**)<sup>3</sup>, phenyl(2,4,6-trimethoxyphenyl)iodonium trifluoromethanesulfonate (**2c**)<sup>11</sup>, phenyl(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2c'**)<sup>5</sup>, (4-methoxyphenyl)(phenyl)iodonium trifluoromethanesulfonate (**2d**)<sup>3</sup>, (3,5-dimethylisoxazol-4-yl)(phenyl)iodonium trifluoromethanesulfonate (**2e**)<sup>6</sup>, bis(2-bromophenyl)iodonium trifluoromethanesulfonate (**2f**)<sup>12</sup>, (2-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2g**)<sup>13</sup>, (3-chloro-2-methylphenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2h**)<sup>7</sup>, bis(2,5-dimethylphenyl)iodonium trifluoromethanesulfonate (**2i**)<sup>1</sup>, phenyl(2,4,6-triisopropylphenyl)iodonium trifluoromethanesulfonate (**2j**)<sup>8</sup>, (3-(methoxycarbonyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2k**)<sup>14</sup>, (3-cyanophenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2l**)<sup>5</sup>, (3-nitrophenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2m**)<sup>9</sup>, bis(4-methoxyphenyl)iodonium trifluoromethanesulfonate (**2n**)<sup>3</sup>, (4-(trifluoromethoxy)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2o**)<sup>15</sup>, (4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2p**)<sup>5</sup>, bis(4-chlorophenyl)iodonium trifluoromethanesulfonate (**2q**)<sup>1</sup>, [1,1'-biphenyl]-4-yl(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2r**)<sup>5</sup>, (3,5-dimethylisoxazol-4-yl)(4-methoxy-3-(2-methoxy-2-oxoethyl)phenyl)iodonium *p*-toluenesulfonate (**2s**)<sup>3</sup>.

Diaryliodonium salts used in this work:



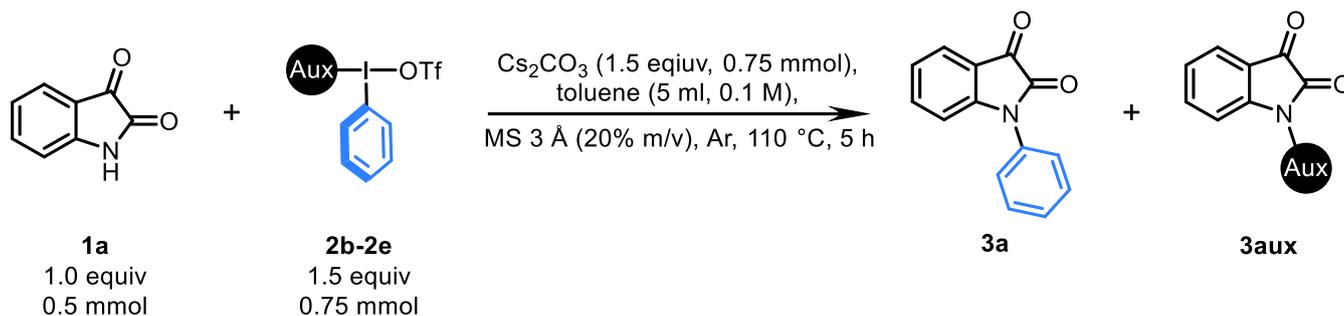
S-III. Optimization of the Reaction Conditions for the Preparation of 1-Arylindoline-2,3-diones **3**

entry	base, equiv	solvent	Ph <sub>2</sub> IOTf, equiv	additive	temp., °C	atmosphere	yield of <b>3a</b> , % <sup>a</sup>
1	NaH, 1.0	DMF	1.0	-	40	argon	-
2	NaH, 1.5	toluene	1.5	-	rt	argon	-
3	<i>t</i> -BuOK, 3.0	THF	1.2	-	0 °C - rt	-	-
4	<i>t</i> -BuOK, 1.5	DMF	1.5	-	55	-	16
5	<i>t</i> -BuOK, 1.5	toluene	1.5	-	55	-	12
6	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	DMF	1.5	-	55	-	trace
7	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	-	55	-	29
8	K <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	-	55	-	9
9	Na <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	-	55	-	trace
10	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	-	90	-	45
11	Cs <sub>2</sub> CO <sub>3</sub> , 1.0	toluene	1.5	-	90	-	39
12	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.0	-	90	-	15
13	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	-	90	-	29 <sup>b</sup>
14	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	90	-	52
15	<i>t</i> -BuOK, 1.5	toluene	1.5	MS 3 Å (20% m/v)	90	argon	34
16	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	-	63
17	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	CCl <sub>4</sub>	1.5	MS 3 Å (20% m/v)	77	-	24
18	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	THF	1.5	MS 3 Å (20% m/v)	66	-	25
19	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	DMSO	1.5	MS 3 Å (20% m/v)	90	-	trace
20	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	PhCl	1.5	MS 3 Å (20% m/v)	110	-	28
21	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	argon	81 (73 <sup>c</sup> )
22	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	argon	72 <sup>d</sup>
23	K <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	argon	27
24	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	<i>o</i> -xylene	1.5	MS 3 Å (20% m/v)	140	argon	69
25	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	argon	74 <sup>e</sup>
26	Cs <sub>2</sub> CO <sub>3</sub> , 1.5	toluene	1.5	MS 3 Å (20% m/v)	110	argon	80 <sup>f</sup>

<sup>a</sup>Reactions were performed on 0.5 mmol (74 mg, 1.0 equiv) of isatin **1a** in 5.0 mL of solvent [0.1 M], reaction time 5 h. <sup>1</sup>H NMR yields were determined by using 1,3,5-trimethoxybenzene as an internal standard.

<sup>b</sup>Commercially available toluene was used without purification. <sup>c</sup>Isolated yield. <sup>d</sup>Bu<sub>4</sub>NBF<sub>4</sub> (10 mol. %) was added. <sup>e</sup>Reaction time 4 h. <sup>f</sup>Reaction time 6 h.

### Auxiliary group optimization



Entry	Aux	Total yield, % <sup>a</sup>	3a : 3aux
1		80	0 : 1
2		13 <sup>b</sup> (14 <sup>c</sup> )	1 : 0
3		trace <sup>b</sup>	- <sup>d</sup>
4		trace <sup>b</sup>	- <sup>d</sup>

<sup>a</sup>NMR yields were determined by using 1,3,5-trimethoxybenzene (Entries 1, 3, and 4) and 1,4-dibromo-2,5-dimethylbenzene (Entry 2) as internal standards. <sup>b</sup>Diaryliodonium salt decomposed under the reaction conditions. <sup>c</sup>TsO<sup>-</sup> instead of TfO<sup>-</sup>. <sup>d</sup>The ratios of the reaction products could not be determined due to the presence of a large amount of by-products in the reaction mixtures.

## S-IV Decomposition of the Diaryliodonium Salts under the Reaction Conditions

### Control experiments without addition of isatin

A flame-dried reaction tube was charged with a stir bar. To the tube were added toluene (5 ml, 0.15 M), anhydrous  $\text{Cs}_2\text{CO}_3$  (244 mg, 0.75 mmol, 1.0 equiv) and activated powdered 3 Å molecular sieves (1 g, 20% m/v). The resulting solution was stirred for 20 min at room temperature. Diaryliodonium salt **2** (0.75 mmol, 1.0 equiv) was added, the tube was sealed with a septum cap, evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was stirred for 5 h at 110 °C. After cooling down to room temperature, molecular sieves were filtered through a paper filter, filter was washed with DCM and filtrate was removed under reduced pressure.

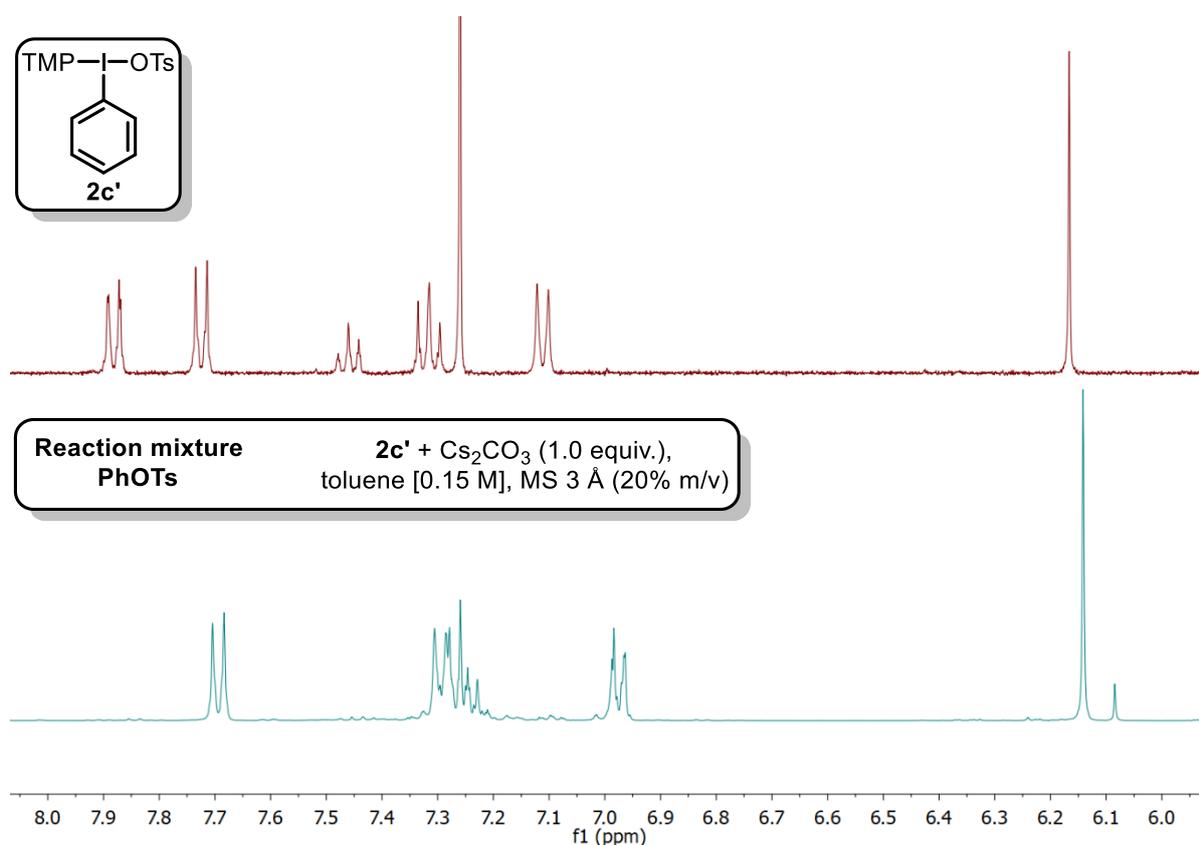
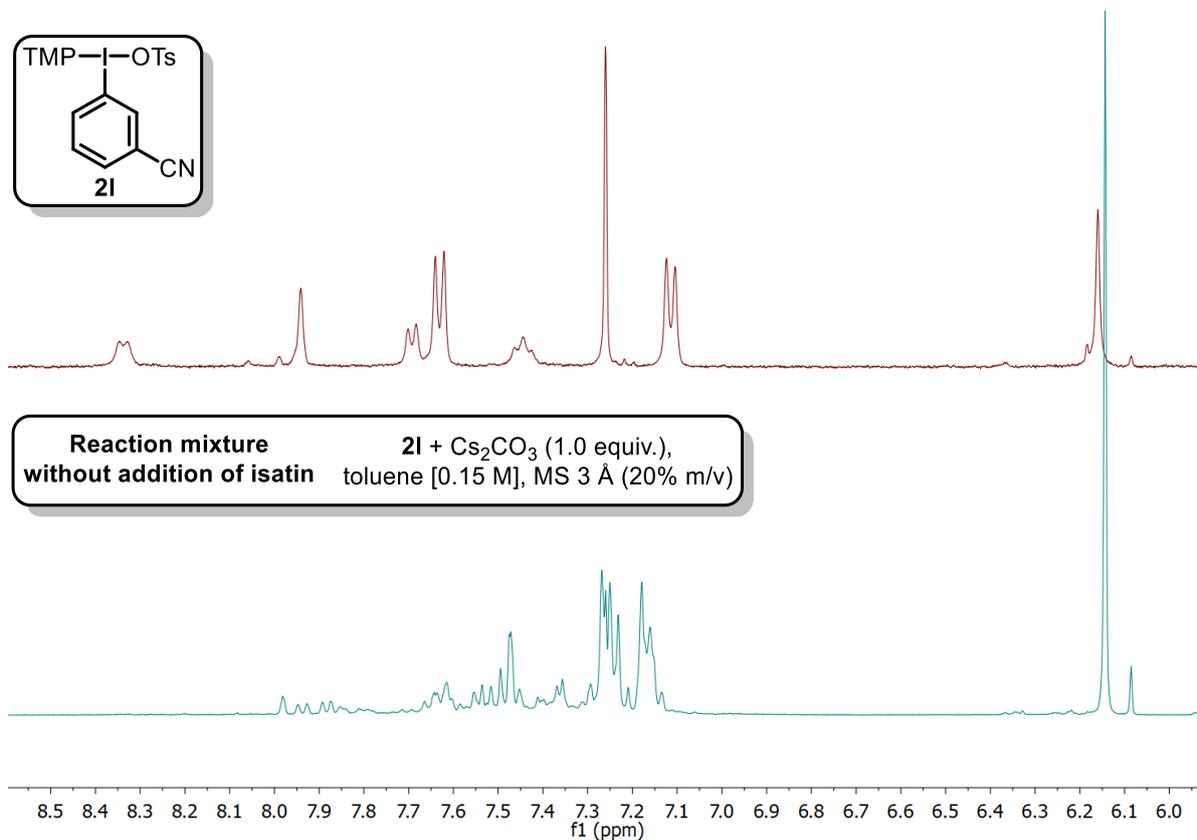
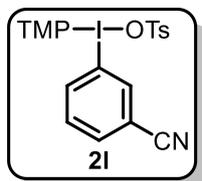


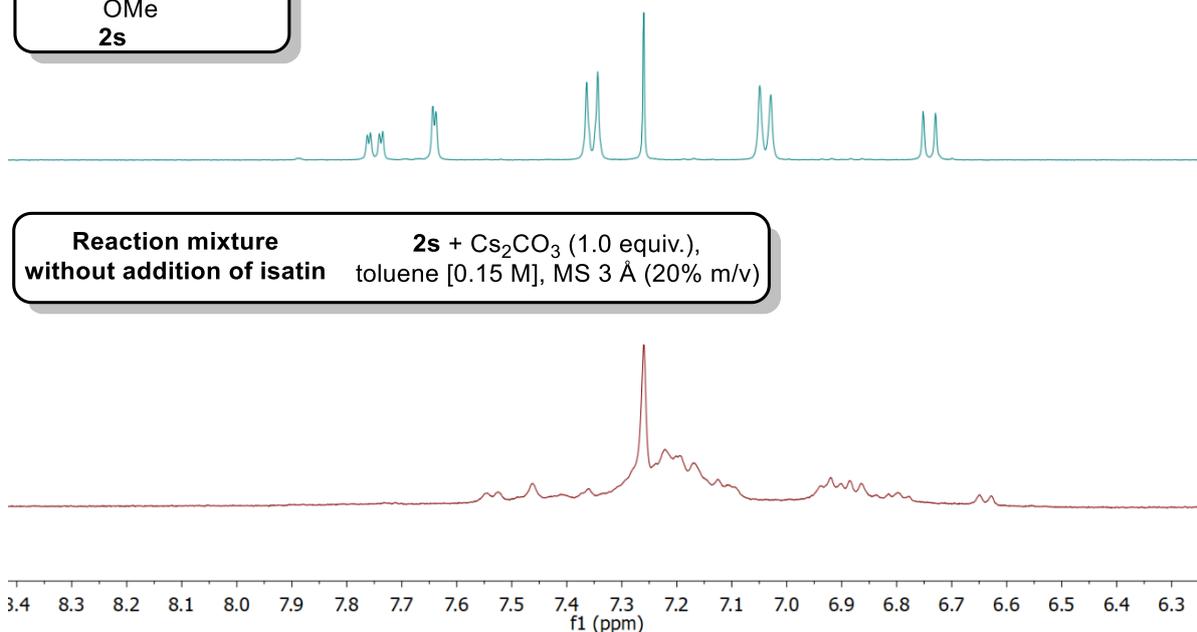
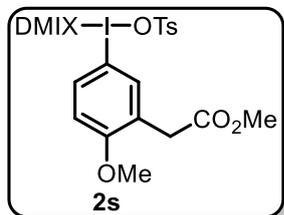
Figure S1.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of phenyl(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2c'**) and PhOTs after reaction

Reaction with phenyl(2,4,6-trimethoxyphenyl)iodonium trifluoromethanesulfonate (**2c**) under the same conditions led to the decomposition of the diaryliodonium salt.



**Reaction mixture**      **2I + Cs<sub>2</sub>CO<sub>3</sub> (1.0 equiv.),**  
**without addition of isatin**    toluene [0.15 M], MS 3 Å (20% m/v)

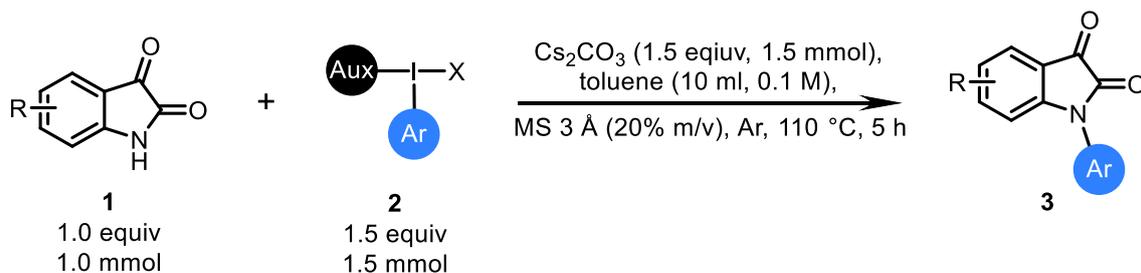
Figure S2. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of (3-cyanophenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (**2I**) and the reaction mixture



**Reaction mixture**      **2S + Cs<sub>2</sub>CO<sub>3</sub> (1.0 equiv.),**  
**without addition of isatin**    toluene [0.15 M], MS 3 Å (20% m/v)

Figure S3. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of (3,5-dimethylisoxazol-4-yl)(4-methoxy-3-(2-methoxy-2-oxoethyl)phenyl)iodonium *p*-toluenesulfonate (**2S**) and the reaction mixture

#### S-IV. General Procedure for the Synthesis of 1-Arylindoline-2,3-diones **3**

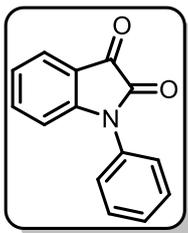


A flame-dried reaction tube was charged with a stir bar. To the tube were added toluene (10 ml, 0.1 M), anhydrous Cs<sub>2</sub>CO<sub>3</sub> (489 mg, 1.5 mmol, 1.5 equiv) and activated powdered 3 Å molecular sieves (2 g, 20% m/v). The resulting solution was stirred for 20 min at room temperature. Isatin **1** (1.0 mmol, 1.0 equiv) and diaryliodonium salt **2** (1.5 mmol, 1.5 equiv) were added, the tube was sealed with a septum cap, evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was stirred for 5 h at 110 °C. After cooling down to room temperature, the solvent was removed under reduced pressure. Then the mixture quenched with 1.5 M HCl (10 ml) and diluted with water (10 ml), transferred to a separatory funnel and extracted with ethyl acetate (3 x 15 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel to afford the desired product.

#### S-VI. General Procedure for the Synthesis of 1-Arylindoline-2,3-diones **3** (Gram-Scale)

A flame-dried round-bottom flask was charged with a stir bar. To the flask were added toluene (0.2 M), anhydrous Cs<sub>2</sub>CO<sub>3</sub> (1.5 equiv) and activated powdered 3 Å molecular sieves (20% m/v). The resulting solution was stirred for 30 min at room temperature. Isatin **1** (0.5 g, 3.4 mmol or 1.0 g, 6.8 mmol, 1 equiv) and diaryliodonium salt **2** (1.5 equiv) were added, the flask was sealed with a septum cap, evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was stirred for 12 h at 110 °C in an oil bath. After cooling down to room temperature, the solvent was removed under reduced pressure. Then the mixture quenched with 1.5 M HCl (20 ml) and diluted with water (20 ml), transferred to a separatory funnel and extracted with ethyl acetate (3 x 30 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel to afford the desired product.

S-VII. Characterization Data for 1-Arylindoline-2,3-diones **3**



**1-Phenylindoline-2,3-dione (3a)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 163 mg (73%).

**Physical state:** orange solid.

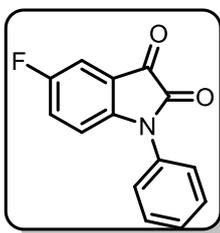
**Melting point (EtOAc – *n*-hexane):** 121 – 123 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.70 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.51 – 7.59 (m, 3H), 7.40 – 7.49 (m, 3H), 7.18 (t, *J* = 7.4, 0.8 Hz, 1H), 6.90 (d, *J* = 8.0 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 183.0, 157.4, 151.8, 138.5, 132.9, 130.1, 128.9, 126.1, 125.7, 124.4, 117.6, 111.4.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.50.

Characterization data obtained for this compound matched those previously reported in the literature<sup>16</sup>.



**5-Fluoro-1-phenylindoline-2,3-dione (3b)** was obtained according to the General Procedure from **5-fluoroindoline-2,3-dione (1b)** (165 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 135 mg (56%).

**Physical state:** orange solid.

**Melting point (EtOAc – *n*-hexane):** 166 – 168 °C.

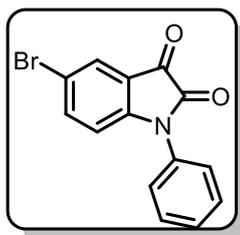
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.53 – 7.60 (m, 2H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.36 – 7.44 (m, 3H), 7.22 – 7.29 (m, 1H), 6.88 (dd, *J* = 8.7, 3.7 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.49 (d, *J* = 1.5 Hz), 159.63 (d, *J* = 246 Hz), 147.9 (d, *J* = 2.0 Hz), 157.1, 132.8, 130.2, 129.1, 126.0, 124.9 (d, *J* = 24.3 Hz), 118.2 (d, *J* = 7.4 Hz), 112.7 (d, *J* = 7.2 Hz), 112.5 (d, *J* = 24.3 Hz).

**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):** δ -117.4.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.41.

Characterization data obtained for this compound matched those previously reported in the literature<sup>17</sup>.



**5-Bromo-1-phenylindoline-2,3-dione (3c)** was obtained according to the General Procedure from **5-bromoindoline-2,3-dione (1c)** (226 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 154 mg (51%).

**Physical state:** red solid.

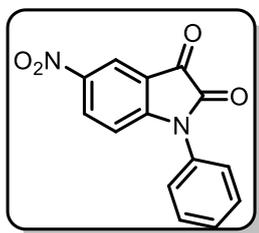
**Melting point (EtOAc – *n*-hexane):** 195 – 197 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.80 (d, *J* = 2.1 Hz, 1H), 7.65 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.54 – 7.60 (m, 2H), 7.44 – 7.51 (m, 1H), 7.37 – 7.42 (m, 2H), 6.81 (d, *J* = 8.5 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 181.9, 156.7, 150.5, 140.7, 132.6, 130.2, 129.2, 128.4, 126.0, 118.8, 117.2, 113.1.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.52.

Characterization data obtained for this compound matched those previously reported in the literature<sup>17</sup>.



**5-Nitro-1-phenylindoline-2,3-dione (3d)** was obtained according to the General Procedure from **5-nitroindoline-2,3-dione (1d)** (192 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (5 : 1) as an eluent.

**Yield:** 48 mg (18%).

**Physical state:** orange solid.

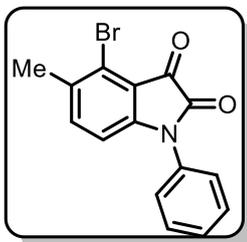
**Melting point (EtOAc – *n*-hexane):** 178 – 180 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 8.56 (d, *J* = 2.3 Hz, 1H), 8.46 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 2H), 7.03 (d, *J* = 8.8 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 181.0, 157.1, 155.6, 144.6, 133.6, 132.0, 130.5, 129.9, 126.2, 121.3, 117.2, 111.7.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.26.

Characterization data obtained for this compound matched those previously reported in the literature<sup>18</sup>.



**4-Bromo-5-methyl-1-phenylindoline-2,3-dione (3e)** was obtained according to the General Procedure from **4-bromo-5-methylindoline-2,3-dione (1e)** (240 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 164 mg (52%).

**Physical state:** orange solid.

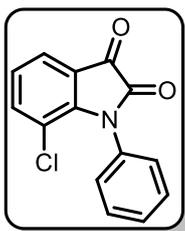
**Melting point (EtOAc – *n*-hexane):** > 200 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.52 – 7.59 (m, 2H), 7.42 – 7.49 (m, 1H), 7.32 – 7.32 (m, 3H), 6.74 (d, *J* = 8.0 Hz, 1H), 2.40 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 180.7, 156.6, 151.4, 138.9, 135.2, 132.8, 130.2, 129.1, 126.3, 124.1, 116.7, 109.9, 21.6.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>15</sub>H<sub>11</sub>BrNO<sub>2</sub><sup>+</sup> 315.9968; Found 315.9972 (1.3 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.54.



**7-Chloro-1-phenylindoline-2,3-dione (3f)** was obtained according to the General Procedure from **7-chloroindoline-2,3-dione (1f)** (182 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 72 mg (28%).

**Physical state:** orange solid.

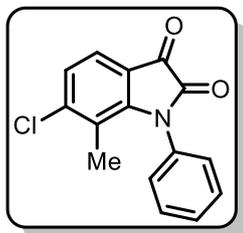
**Melting point (EtOAc – *n*-hexane):** 122 – 124 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.65 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.44 – 7.55 (m, 4H), 7.32 – 7.38 (m, 2H), 7.09 – 7.15 (m, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.5, 158.4, 146.6, 140.9, 134.1, 129.5, 129.4, 128.2, 125.1, 124.3, 120.3, 118.4.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>14</sub>H<sub>9</sub>ClNO<sub>2</sub><sup>+</sup> 258.0316; Found 258.0317 (0.4 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.44.



**6-Chloro-7-methyl-1-phenylindoline-2,3-dione (3g)** was obtained according to the General Procedure from **6-chloro-7-methylindoline-2,3-dione (1g)** (195 mg, 1.0 mmol) and **diphenyliodonium trifluoromethanesulfonate (2a)** (645 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 54 mg (20%).

**Physical state:** orange solid.

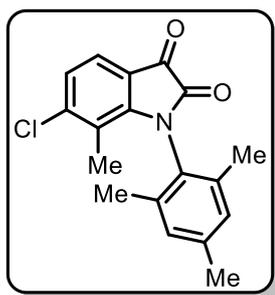
**Melting point (EtOAc – *n*-hexane):** 187 – 189 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.45 – 7.58 (m, 4H), 7.30 – 7.38 (m, 2H), 7.23 (d, *J* = 8.0 Hz, 1H), 1.77 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.5, 159.3, 150.2, 147.0, 135.6, 130.0, 129.4, 127.7, 125.6, 123.8, 122.1, 117.4, 15.4.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>15</sub>H<sub>11</sub>ClNO<sub>2</sub><sup>+</sup> 272.0473; Found 272.0479 (2.2 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.52.



**6-Chloro-1-mesityl-7-methylindoline-2,3-dione (3h)** was obtained according to the General Procedure from **6-chloro-7-methylindoline-2,3-dione (1g)** (196 mg, 1.0 mmol) and **mesityl(phenyl)iodonium trifluoromethanesulfonate (2b)** (708 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (10 : 1) as an eluent.

**Yield:** 31 mg (10%).

**Physical state:** orange solid.

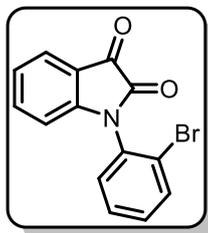
**Melting point (EtOAc – *n*-hexane):** 125 – 127 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.00 (s, 2H), 2.35 (s, 3H), 2.14 (s, 6H), 1.70 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.4, 158.6, 149.7, 146.9, 140.0, 136.4, 130.8, 129.6, 125.3, 124.0, 121.8, 117.2, 21.3, 18.2, 12.8.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>18</sub>H<sub>17</sub>ClNO<sub>2</sub><sup>+</sup> 314.0942; Found 314.0946 (1.3 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.62.



**1-(2-Bromophenyl)indoline-2,3-dione (3i)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **bis(2-bromophenyl)iodonium trifluoromethanesulfonate (2f)** (882 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (5 : 1) as an eluent.

**Yield:** 145 mg (48%).

**Physical state:** orange solid.

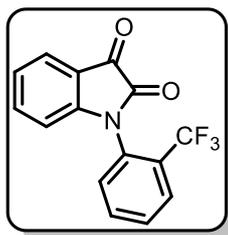
**Melting point (EtOAc – *n*-hexane):** 138 – 140 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  7.77 – 7.82 (m, 1H), 7.72 (dd,  $J = 7.6, 1.3$  Hz, 1H), 7.49 – 7.57 (m, 2H), 7.38 – 7.45 (m, 2H), 7.18 (td,  $J = 7.6, 0.8$  Hz, 1H), 6.54 (dt,  $J = 8.1, 0.7$  Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):**  $\delta$  182.4, 157.2, 151.4, 138.6, 134.4, 132.5, 131.4, 130.3, 129.3, 125.7, 124.5, 122.8, 117.5, 111.7.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.29.

Characterization data obtained for this compound matched those previously reported in the literature<sup>16</sup>.



**1-(2-(Trifluoromethyl)phenyl)indoline-2,3-dione (3j)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(2-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2g)** (916 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 146 mg (50%).

**Physical state:** orange solid.

**Melting point (EtOAc – *n*-hexane):** 168 – 170 °C.

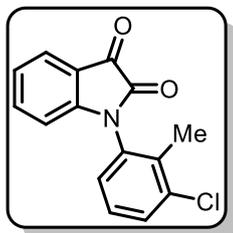
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  7.89 – 7.94 (m, 1H), 7.76 – 7.82 (m, 1H), 7.66 – 7.74 (m, 2H), 7.52 (td,  $J = 7.8, 1.4$  Hz, 1H), 7.43 (d,  $J = 7.8$  Hz, 1H), 7.18 (td,  $J = 7.5, 0.8$  Hz, 1H), 6.44 (d,  $J = 8.0$  Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):**  $\delta$  182.3, 157.9, 152.5, 138.7, 134.2, 131.2 (q,  $J = 1.8$  Hz), 131.0, 130.6, 129.9 (q,  $J = 31.3$  Hz), 128.3 (q,  $J = 4.6$  Hz), 125.5, 124.5, 122.9 (q,  $J = 274$  Hz), 117.5, 111.6.

**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):**  $\delta$  -61.25.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>15</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>2</sub><sup>+</sup> 292.0580; Found 292.0584 (1.4 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 2 : 1)** = 0.48.



**1-(3-Chloro-2-methylphenyl)indoline-2,3-dione (3k)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(3-chloro-2-methylphenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2h)** (886 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 144 mg (53%).

**Physical state:** orange solid.

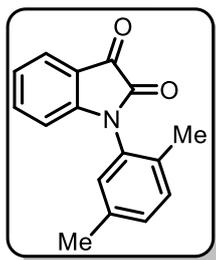
**Melting point (EtOAc – *n*-hexane):** 168 – 170 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.72 (d, *J* = 7.5 Hz, 1H), 7.50 – 7.56 (m, 2H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.16 – 7.23 (m, 2H), 6.55 (d, *J* = 7.9 Hz, 1H), 2.26 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.7, 157.3, 151.7, 138.8, 136.6, 135.2, 133.1, 130.8, 128.1, 126.4, 125.8, 124.6, 117.6, 111.4, 15.7.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for <sup>+</sup> C<sub>15</sub>H<sub>11</sub>ClNO<sub>2</sub> 272.0473; Found 272.0480 (2.6 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.48.



**1-(2,5-Dimethylphenyl)indoline-2,3-dione (3l)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **bis(2,5-dimethylphenyl)iodonium trifluoromethanesulfonate (2i)** (729 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 168 mg (67%).

**Physical state:** orange solid.

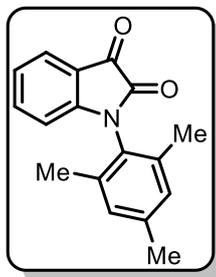
**Melting point (EtOAc – *n*-hexane):** 129 – 131 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.69 (d, *J* = 7.5 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 7.9 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.06 (s, 1H), 6.55 (d, *J* = 7.9 Hz, 1H), 2.36 (s, 3H), 2.17 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 183.2, 157.4, 152.1, 138.6, 137.6, 133.0, 131.7, 131.4, 130.6, 128.0, 125.5, 124.2, 117.5, 111.4, 20.9, 17.5.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>16</sub>H<sub>14</sub>NO<sub>2</sub><sup>+</sup> 252.1019; Found 252.1020 (0.4 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.45.



**1-Mesitylindoline-2,3-dione (3m)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **mesityl(phenyl)iodonium trifluoromethanesulfonate (2b)** (708 mg, 1.5 mmol) and to the General Procedure (Gram-Scale) from **indoline-2,3-dione (1a)** (1.0 g, 6.8 mmol) and **mesityl(phenyl)iodonium trifluoromethanesulfonate (2b)** (4.81 g, 10.2 mmol) using a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 207 mg (78%) and 992 mg (55%) respectively.

**Physical state:** orange solid.

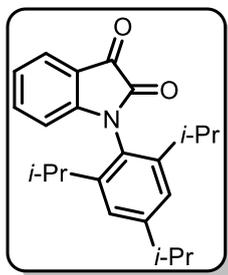
**Melting point (EtOAc – *n*-hexane):** 169 – 171 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.70 (d, *J* = 7.4 Hz, 1H), 7.50 (td, *J* = 7.8, 1.2 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.03 (s, 2H), 6.43 (d, *J* = 7.9 Hz, 1H), 2.34 (s, 3H), 2.13 (s, 6H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 183.2, 157.5, 151.7, 139.8, 138.8, 136.3, 129.9, 127.9, 125.8, 124.2, 117.7, 111.1, 21.2, 18.0.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.37.

Characterization data obtained for this compound matched those previously reported in the literature<sup>19</sup>.



**1-(2,4,6-Triisopropylphenyl)indoline-2,3-dione (3n)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **phenyl(2,4,6-triisopropylphenyl)iodonium trifluoromethanesulfonate (2j)** (835 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (10 : 1) as an eluent.

**Yield:** 185 mg (53 %).

**Physical state:** orange solid.

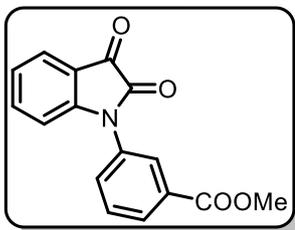
**Melting point (EtOAc – *n*-hexane):** 154 – 156 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.70 (d, *J* = 7.5 Hz, 1H), 7.45 – 7.54 (m, 1H), 7.10 – 7.20 (m, 3H), 6.45 (d, *J* = 8.0 Hz, 1H), 2.90 – 3.02 (m, 1H), 2.74 – 2.86 (m, 2H), 1.29 (d, *J* = 6.9 Hz, 6H), 1.17 (d, *J* = 6.8 Hz, 6H), 1.11 (d, *J* = 6.8 Hz, 6H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 183.3, 158.5, 153.4, 151.0, 146.8, 138.7, 125.6, 125.4, 124.2, 122.8, 117.5, 111.4, 34.5, 29.3, 24.6, 24.1, 23.9.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>23</sub>H<sub>28</sub>NO<sub>2</sub><sup>+</sup> 350.2115; Found 350.2121 (1.7 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.68.



**Methyl 3-(2,3-dioxindolin-1-yl)benzoate (3o)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(3-(methoxycarbonyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium p-toluenesulfonate (2k)** (600 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (5 : 1) as an eluent.

**Yield:** 143 mg (51%).

**Physical state:** orange solid.

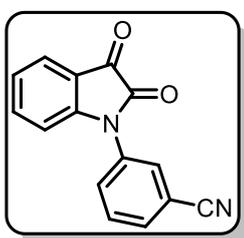
**Melting point (EtOAc – *n*-hexane):** 178 – 180 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 8.08 – 8.18 (m, 2H), 7.70 – 7.75 (m, 1H), 7.62 – 7.69 (m, 2H), 7.57 (td, *J* = 7.8, 1.4 Hz, 1H), 7.21 (td, *J* = 7.6, 0.8 Hz, 1H), 6.90 (d, *J* = 8.1 Hz, 1H), 3.95 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.5, 166.0, 157.3, 151.2, 138.6, 133.3, 132.2, 130.7, 130.3, 129.9, 127.0, 125.9, 124.7, 117.6, 111.3, 52.6.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.20.

Characterization data obtained for this compound matched those previously reported in the literature<sup>16</sup>.



**3-(2,3-Dioxindolin-1-yl)benzonitrile (3p)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(3-cyanophenyl)(2,4,6-trimethoxyphenyl)iodonium p-toluenesulfonate (2l)** (851 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (4 : 1) as an eluent.

**Yield:** 79 mg (32%).

**Physical state:** orange solid.

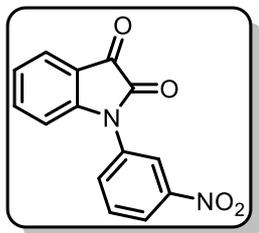
**Melting point (EtOAc – *n*-hexane):** > 200 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.68 – 7.80 (m, 5H), 7.61 (td, *J* = 7.8, 1.4 Hz, 1H), 7.22 – 7.28 (m, 1H), 6.94 (d, *J* = 8.0 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 181.8, 157.1, 150.5, 138.7, 134.1, 132.3, 131.2, 130.5, 129.3, 126.3, 125.2, 117.8, 117.6, 114.4, 111.1.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>15</sub>H<sub>9</sub>N<sub>2</sub>O<sub>2</sub><sup>+</sup> 249.0659; Found 249.0658 (0.4 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 1 : 2)** = 0.41.



**1-(3-Nitrophenyl)indoline-2,3-dione (3q)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(3-nitrophenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2m)** (881 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 110 mg (41%).

**Physical state:** orange solid.

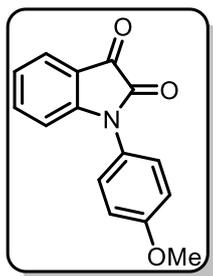
**Melting point (EtOAc – *n*-hexane):** 178 – 180 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 8.28 – 8.41 (m, 2H), 7.83 – 7.87 (m, 1H), 7.74 – 7.81 (m, 2H), 7.60 – 7.66 (m, 1H), 7.27 (t, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 8.1 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 181.8, 157.1, 150.4, 149.2, 138.8, 134.2, 132.1, 131.1, 126.3, 125.2, 123.5, 121.0, 117.8, 111.1.

**R<sub>f</sub> (*n*-hexane : EtOAc = 2 : 1)** = 0.50.

Characterization data obtained for this compound matched those previously reported in the literature<sup>20</sup>.



**1-(4-Methoxyphenyl)indoline-2,3-dione (3r)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **bis(4-methoxyphenyl)iodonium trifluoromethanesulfonate (2n)** (735 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (4 : 1) as an eluent.

**Yield:** 18 mg (7%).

**Physical state:** orange solid.

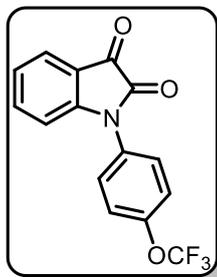
**Melting point (EtOAc – *n*-hexane):** 152 – 154 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.68 (dd, *J* = 7.4, 1.4 Hz, 1H), 7.53 (td, *J* = 7.8, 1.4 Hz, 1H), 7.29 – 7.35 (m, 2H), 7.15 (td, *J* = 7.5, 0.9 Hz, 1H), 7.03 – 7.08 (m, 2H), 6.83 (d, *J* = 8.0 Hz, 1H), 3.87 (s, 3H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 183.2, 159.8, 157.8, 152.2, 138.5, 127.6, 125.6, 125.5, 124.3, 117.5, 115.3, 111.3, 55.7.

**R<sub>f</sub> (*n*-hexane : EtOAc = 2 : 1)** = 0.39.

Characterization data obtained for this compound matched those previously reported in the literature<sup>16</sup>.



**1-(4-(Trifluoromethoxy)phenyl)indoline-2,3-dione (3s)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(4-(trifluoromethoxy)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2o)** (940 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (8 : 1) as an eluent.

**Yield:** 92 mg (30%).

**Physical state:** orange solid.

**Melting point (EtOAc – *n*-hexane):** 130 – 132 °C.

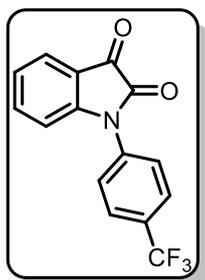
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.71 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.57 (td, *J* = 7.8, 1.3 Hz, 1H), 7.46 – 7.52 (m, 2H), 7.41 (d, *J* = 8.6 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.4, 157.4, 151.2, 149.0, 138.6, 131.5, 127.7, 126.0, 124.8, 122.6, 120.51 (q, *J* = 258 Hz), 117.7, 111.2.

**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):** δ -57.9.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>15</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>3</sub><sup>+</sup> 308.0529; Found 308.0533 (1.3 ppm).

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.57.



**1-(4-(Trifluoromethyl)phenyl)indoline-2,3-dione (3t)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **(4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2p)** (916 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 210 mg (72%).

**Physical state:** red solid.

**Melting point (EtOAc – *n*-hexane):** 179 – 181 °C.

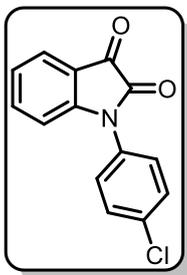
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.75 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.54 – 7.64 (m, 3H), 7.23 (td, *J* = 7.6, 0.8 Hz, 1H), 6.97 (d, *J* = 8.1 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.2, 157.1, 150.8, 138.6, 136.2, 130.8 (q, *J* = 33.1 Hz), 127.2 (q, *J* = 3.7 Hz), 126.2, 126.1, 124.9, 123.7 (q, *J* = 272 Hz), 117.7, 111.3.

**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):** δ -62.7.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.36.

Characterization data obtained for this compound matched those previously reported in the literature<sup>21</sup>.



**1-(4-Chlorophenyl)indoline-2,3-dione (3u)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **bis(4-chlorophenyl)iodonium trifluoromethanesulfonate (2q)** (749 mg, 1.5 mmol) using a mixture of *n*-hexane and ethyl acetate (6 : 1) as an eluent.

**Yield:** 70 mg (27%).

**Physical state:** orange solid.

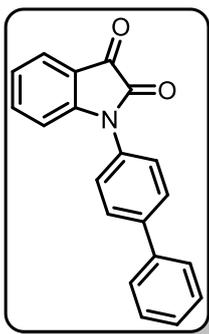
**Melting point (EtOAc – *n*-hexane):** 198 – 200 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.68 – 7.74 (m, 1H), 7.50 – 7.60 (m, 3H), 7.34 – 7.42 (m, 2H), 7.20 (t, *J* = 7.5 Hz, 0.8 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.5, 157.3, 151.3, 138.6, 134.7, 131.4, 130.3, 127.4, 125.9, 124.7, 117.6, 111.2.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.48.

Characterization data obtained for this compound matched those previously reported in the literature<sup>22</sup>.



**1-([1,1'-Biphenyl]-4-yl)indoline-2,3-dione (3v)** was obtained according to the General Procedure from **indoline-2,3-dione (1a)** (147 mg, 1.0 mmol) and **[1,1'-biphenyl]-4-yl(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2r)** (928 mg, 1.5 mmol) and to the General Procedure (Gram-Scale) from **indoline-2,3-dione (1a)** (0.5 g, 3.4 mmol) and **[1,1'-biphenyl]-4-yl(2,4,6-trimethoxyphenyl)iodonium *p*-toluenesulfonate (2r)** (3.15 g, 5.1 mmol) using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 123 mg (41%) and 397 mg (39%) respectively.

**Physical state:** orange solid.

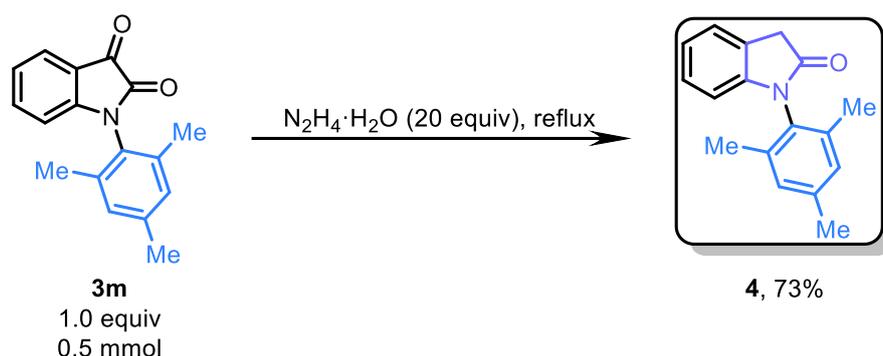
**Melting point (EtOAc – *n*-hexane):** 198 – 202 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.74 – 7.79 (m, 2H), 7.72 (d, *J* = 7.4 Hz, 1H), 7.60 – 7.64 (m, 2H), 7.54 – 7.60 (m, 1H), 7.46 – 7.53 (m, 4H), 7.38 – 7.43 (m, 1H), 7.17 – 7.22 (m, 1H), 6.98 (d, *J* = 8.0 Hz, 1H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 182.9, 157.5, 151.7, 142.0, 140.0, 138.5, 132.0, 129.1, 128.7, 128.0, 127.3, 126.4, 125.8, 124.5, 117.7, 111.5.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.50.

Characterization data obtained for this compound matched those previously reported in the literature<sup>17</sup>.

**1-Mesitylindolin-2-one (4)**

According to the previously reported procedure<sup>23</sup>, to the round-bottom flask charged with a stir bar were added **1-mesitylindoline-2,3-dione (3m)** (133 mg, 0.5 mmol, 1.0 equiv) and hydrazine hydrate (500 mg, 10.0 mmol, 20.0 equiv). The reaction mixture was refluxed for 12 h. Then the mixture was diluted with water (10 ml), transferred to a separatory funnel and extracted with ethyl acetate (2 x 10 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using a mixture of *n*-hexane and ethyl acetate (7 : 1) as an eluent.

**Yield:** 92 mg (73%).

**Physical state:** white solid.

**Melting point (EtOAc – *n*-hexane):** 99 – 101°C.

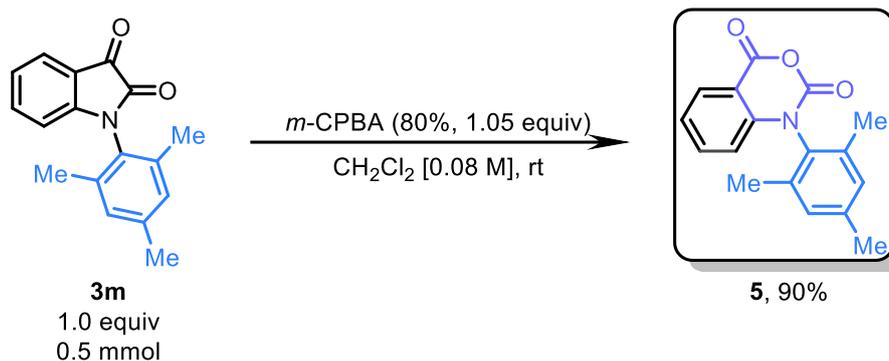
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.32 (d, *J* = 7.3 Hz, 1H), 7.14 – 7.19 (m, 1H), 7.05 (td, *J* = 7.5, 1.1 Hz, 1H), 7.01 (s, 2H), 6.35 (d, *J* = 7.7 Hz, 1H), 3.74 (s, 2H), 2.34 (s, 3H), 2.08 (s, 6H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 174.5, 144.9, 139.0, 136.5, 129.6, 129.5, 128.1, 124.74, 124.68, 122.7, 109.2, 36.0, 21.2, 17.9.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1) = 0.45.**

Characterization data obtained for this compound matched those previously reported in the literature<sup>24</sup>.

## 1-Mesityl-2*H*-benzo[*d*][1,3]oxazine-2,4(1*H*)-dione (5)



According to the previously reported procedure<sup>25</sup>, to the round-bottom flask charged with a stir bar were added **1-mesitylindoline-2,3-dione (3m)** (133 mg, 0.5 mmol, 1.0 equiv) and DCM (6.25 ml, 0.08 M), followed by *m*-CPBA (80%, 113 mg, 0.525 mmol, 1.05 equiv). The reaction mixture was stirred for 12 h at room temperature. Then the mixture diluted with DCM (5 ml), transferred to a separatory funnel and washed with saturated NaHCO<sub>3</sub> solution (2 x 10 mL). The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using a mixture of *n*-hexane and ethyl acetate (10 : 1) as an eluent.

**Yield:** 127 mg (90%).

**Physical state:** white solid.

**Melting point (EtOAc – *n*-hexane):** 178 – 181°C.

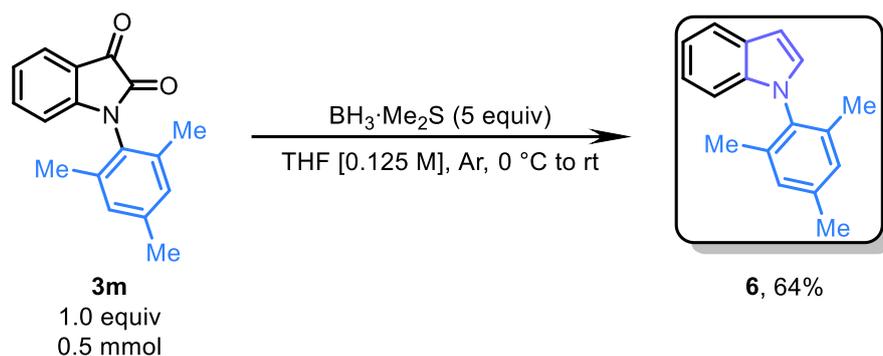
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 8.21 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.53 – 7.59 (m, 1H), 7.27 – 7.32 (m, 1H), 7.07 (s, 2H), 6.45 (d, *J* = 8.4 Hz, 1H), 2.37 (s, 3H), 2.07 (s, 6H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 158.8, 146.6, 142.1, 140.1, 137.5, 135.9, 130.8, 130.3, 129.9, 124.5, 114.6, 111.2, 21.2, 17.5.

**R<sub>f</sub> (*n*-hexane : EtOAc = 10 : 1)** = 0.28.

Characterization data obtained for this compound matched those previously reported in the literature<sup>26</sup>.

## 1-Mesityl-1H-indole (6)



According to the previously reported procedure<sup>27</sup>, to the round-bottom flask charged with a stir bar were added **1-mesitylindoline-2,3-dione (3m)** (133 mg, 0.5 mmol, 1.0 equiv) and dry THF (4 ml, 0.125 M). The flask was sealed with a septum cap, evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was cooled to 0 °C and borane dimethyl sulfide complex (190 mg, 2.5 mmol, 5.0 equiv) was added dropwise. The reaction mixture was allowed to warm up to room temperature and stirred for 3 h, then it was poured into saturated  $\text{NH}_4\text{Cl}$  solution (15 ml), transferred to a separatory funnel and extracted with ethyl acetate (2 x 15 mL). The combined organic layers were dried over anhydrous  $\text{Na}_2\text{SO}_4$ , the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using *n*-hexane as an eluent.

**Yield:** 75 mg (64%).

**Physical state:** viscous colorless oil, slowly turning brown over time.

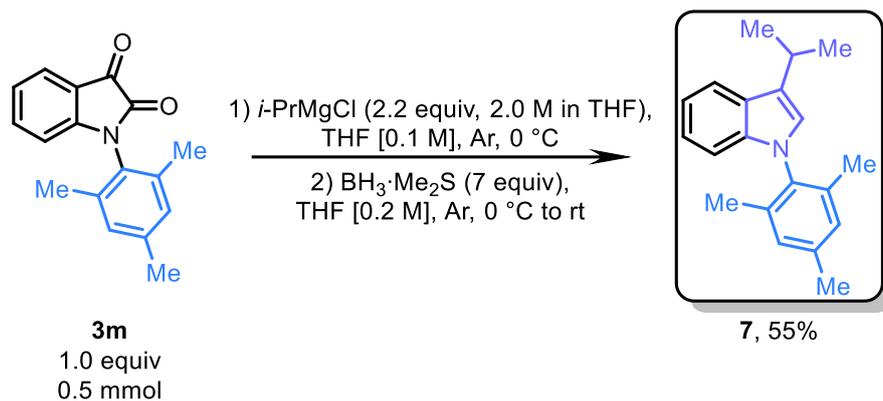
**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  7.67 – 7.72 (m, 1H), 7.11 – 7.16 (m, 2H), 7.05 (d,  $J = 3.1$  Hz, 1H), 7.00 – 7.02 (m, 2H), 6.87 – 6.91 (m, 1H), 6.68 (dd,  $J = 3.1, 0.9$  Hz, 1H), 2.38 (s, 3H), 1.89 (s, 6H).

**$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  138.3, 137.0, 136.6, 134.6, 129.1, 128.3, 128.2, 122.1, 120.9, 119.8, 110.3, 102.4, 21.2, 17.5.

**HRMS (ESI)  $m/z$ :**  $[\text{M}+\text{H}^+]$  Calcd for  $\text{C}_{17}\text{H}_{18}\text{N}^+$  236.1434; Found 236.1435 (0.4 ppm).

**$R_f$  (*n*-hexane : EtOAc = 40 : 1) = 0.7.**

### 3-Isopropyl-1-mesityl-1*H*-indole (7)



According to the previously reported procedure<sup>28</sup>, to the round-bottom flask charged with a stir bar were added **1-mesitylindoline-2,3-dione (3m)** (133 mg, 0.5 mmol, 1.0 equiv) and dry THF (5 ml, 0.1 M). The flask was sealed with a septum cap, the flask was evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was cooled to 0 °C and isopropylmagnesium chloride (2.0 M solution in THF, 0.55 ml, 1.1 mmol, 2.2 equiv) was added dropwise. The reaction mixture was stirred for 2 h at 0 °C, then quenched with 1.5 M HCl and diluted with water (15 ml). Then the mixture was transferred to a separatory funnel and extracted with ethyl acetate (2 x 15 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. Crude intermediate was dissolved in dry THF (2.5 ml), the flask was sealed with a septum cap, evacuated and refilled with argon five times, then argon-filled balloon was attached. The reaction mixture was cooled to 0 °C and borane dimethyl sulfide complex (266 mg, 3.5 mmol, 7 equiv) was added dropwise. The reaction mixture was allowed to warm up to room temperature and stirred for 3 h, then it was poured into saturated NH<sub>4</sub>Cl solution (15 ml), transferred to a separatory funnel and extracted with ethyl acetate (2 x 15 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using *n*-hexane as an eluent.

**Yield:** 76 mg (55%).

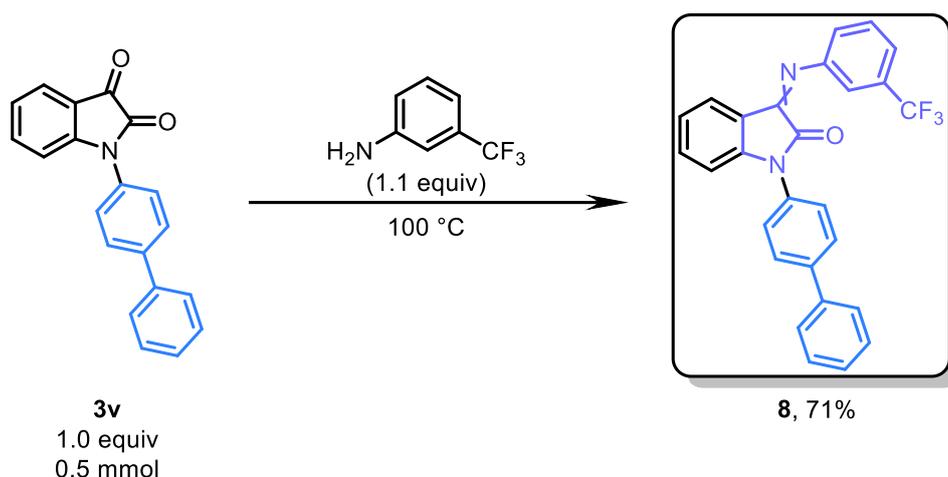
**Physical state:** viscous colorless oil, slowly turning brown over time.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ 7.70 – 7.75 (m, 1H), 7.10 – 7.16 (m, 2H), 7.00 (s, 2H), 6.83 – 6.88 (m, 1H), 6.81 (s, 1H), 3.24 – 3.36 (m, 1H), 2.38 (s, 3H), 1.90 (s, 6H), 1.41 (d, *J* = 6.9 Hz, 6H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):** δ 138.0, 137.2, 134.9, 129.1, 129.0, 127.1, 124.0, 123.6, 121.8, 119.5, 118.8, 110.3, 25.7, 23.7, 21.2, 17.6.

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>20</sub>H<sub>24</sub>N<sup>+</sup> 278.1903; Found 278.1904 (0.4 ppm).

**R<sub>f</sub> (*n*-hexane)** = 0.47.

**1-([1,1'-Biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**)**

According to the previously reported procedure<sup>29</sup>, to the round-bottom flask charged with a stir bar were added **1-([1,1'-biphenyl]-4-yl)indoline-2,3-dione (**3v**)** (150 mg, 0.5 mmol, 1.0 equiv) and 3-(trifluoromethyl)aniline (89 mg, 0.55 mmol, 1.1 equiv). The reaction mixture was heated neat at 100 °C for 3 h. After cooling to room temperature, diethyl ether was added and the resulting solid was filtered and dried under vacuum. **1-([1,1'-Biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one** was isolated as a mixture of *E* : *Z* isomers.

**Yield:** 157 mg (71%).

**Physical state:** yellow solid.

**Melting point (Et<sub>2</sub>O):** 140 – 142 °C.

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  7.28 – 7.84 (m, 13.3H), 7.15 – 7.27 (m, 1H), 6.88 – 6.96 (m, 1H), 6.82 (t, *J* = 7.7 Hz, 0.85H), 6.62 (d, *J* = 7.7 Hz, 0.85H).

**<sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>):**  $\delta$  162.3, 156.6, 155.1, 152.7, 150.7, 149.5, 148.4, 146.9, 141.8, 141.5, 140.12, 140.10, 134.8, 134.4, 132.45, 132.4, 132.1 (q, *J* = 32.5 Hz) 131.0, 130.3, 129.13, 129.05, 129.0, 128.7, 128.4, 127.93, 127.87, 127.3, 127.2, 126.8, 126.4, 126.3, 124.1, 123.9 (q, *J* = 273 Hz) 123.7, 123.5, 122.1 (q, *J* = 4.1 Hz), 121.7 (q, *J* = 3.6 Hz), 121.3, 121.1, 116.2 (q, *J* = 3.9 Hz), 115.5, 115.0 (q, *J* = 3.9 Hz), 111.1, 110.4.

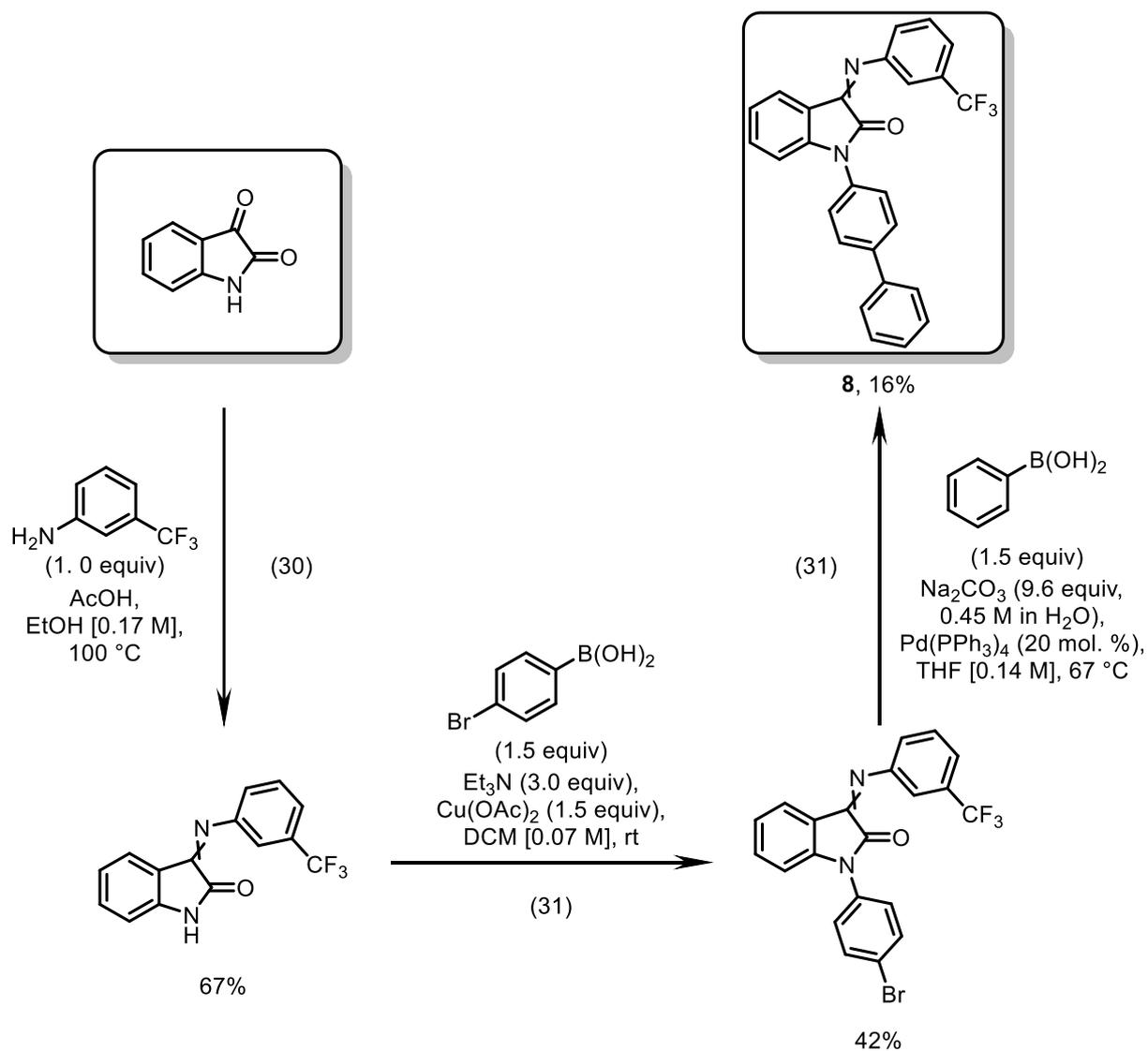
**<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):**  $\delta$  -62.6, -62.7.

**R<sub>f</sub> (*n*-hexane : EtOAc = 4 : 1)** = 0.52.

**MS, *m/z* (I, %):** 442 (M<sup>+</sup>, 50), 414 (100).

**HRMS (ESI) *m/z*:** [M+H<sup>+</sup>] Calcd for C<sub>27</sub>H<sub>18</sub>F<sub>3</sub>N<sub>2</sub>O<sup>+</sup> 443.1366; Found 443.1369 (0.7 ppm).

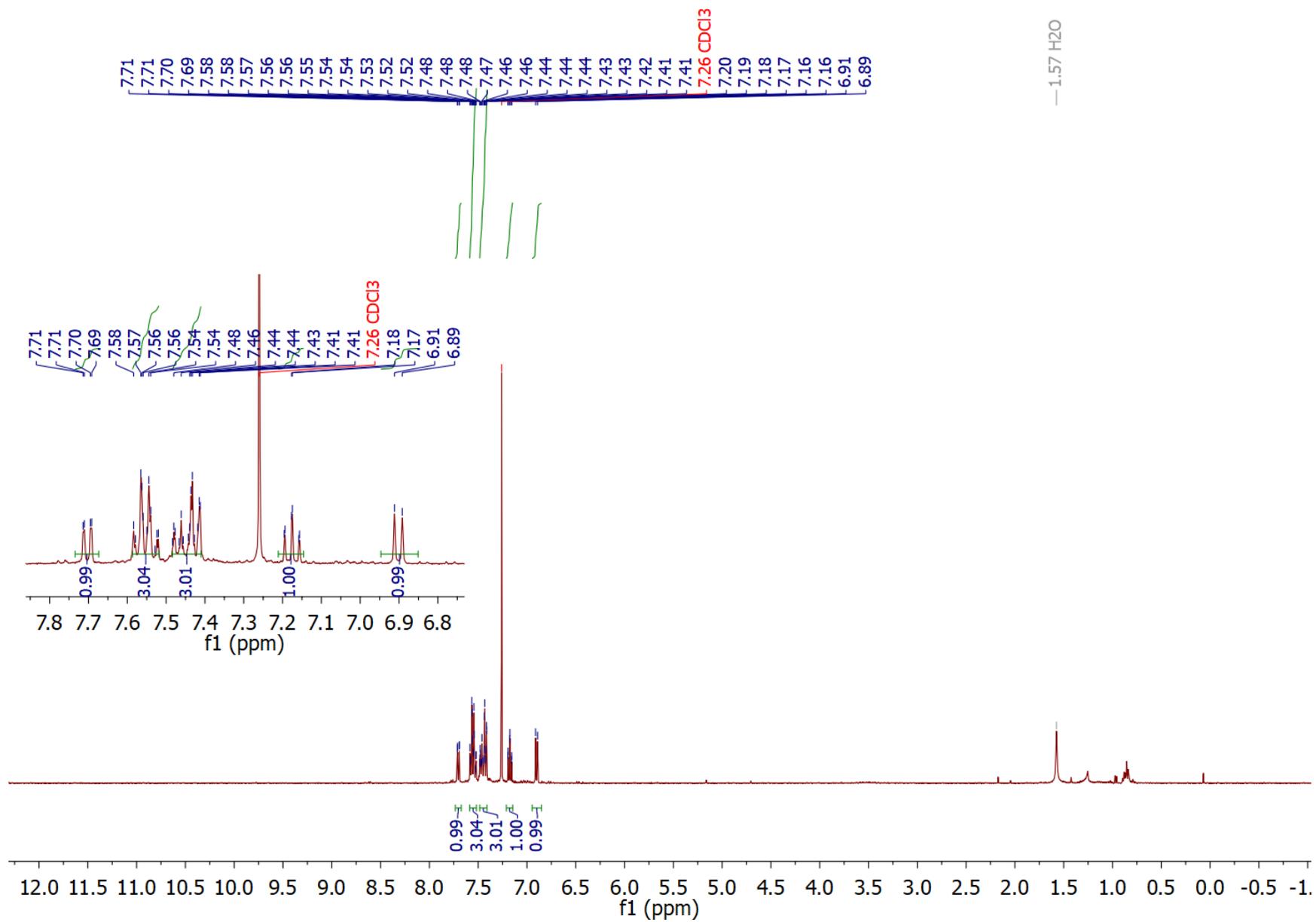
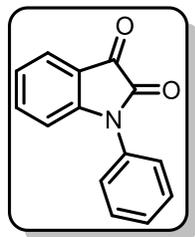
Previously reported synthetic approach to the compound **8**<sup>30,31</sup>



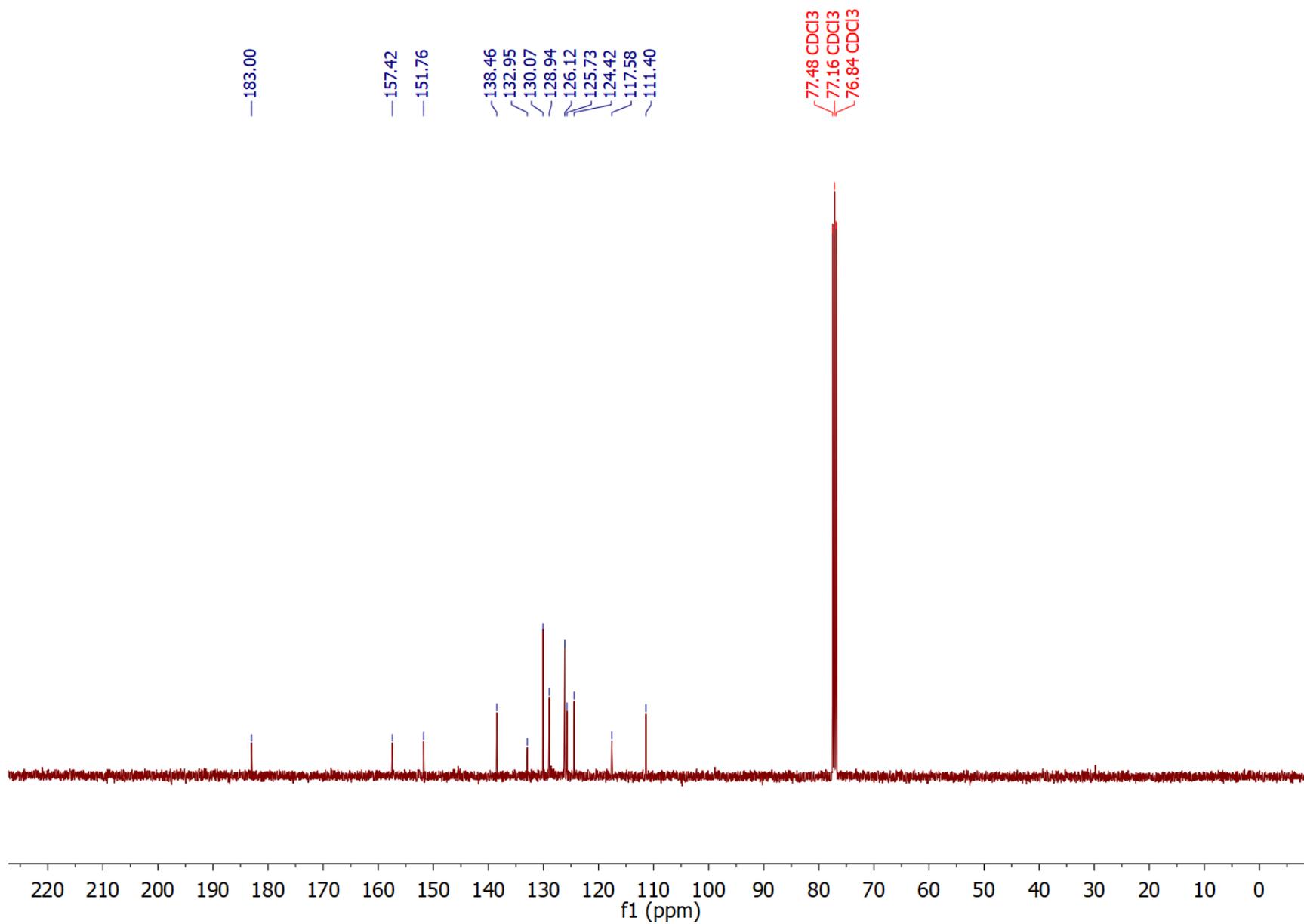
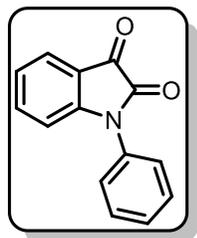
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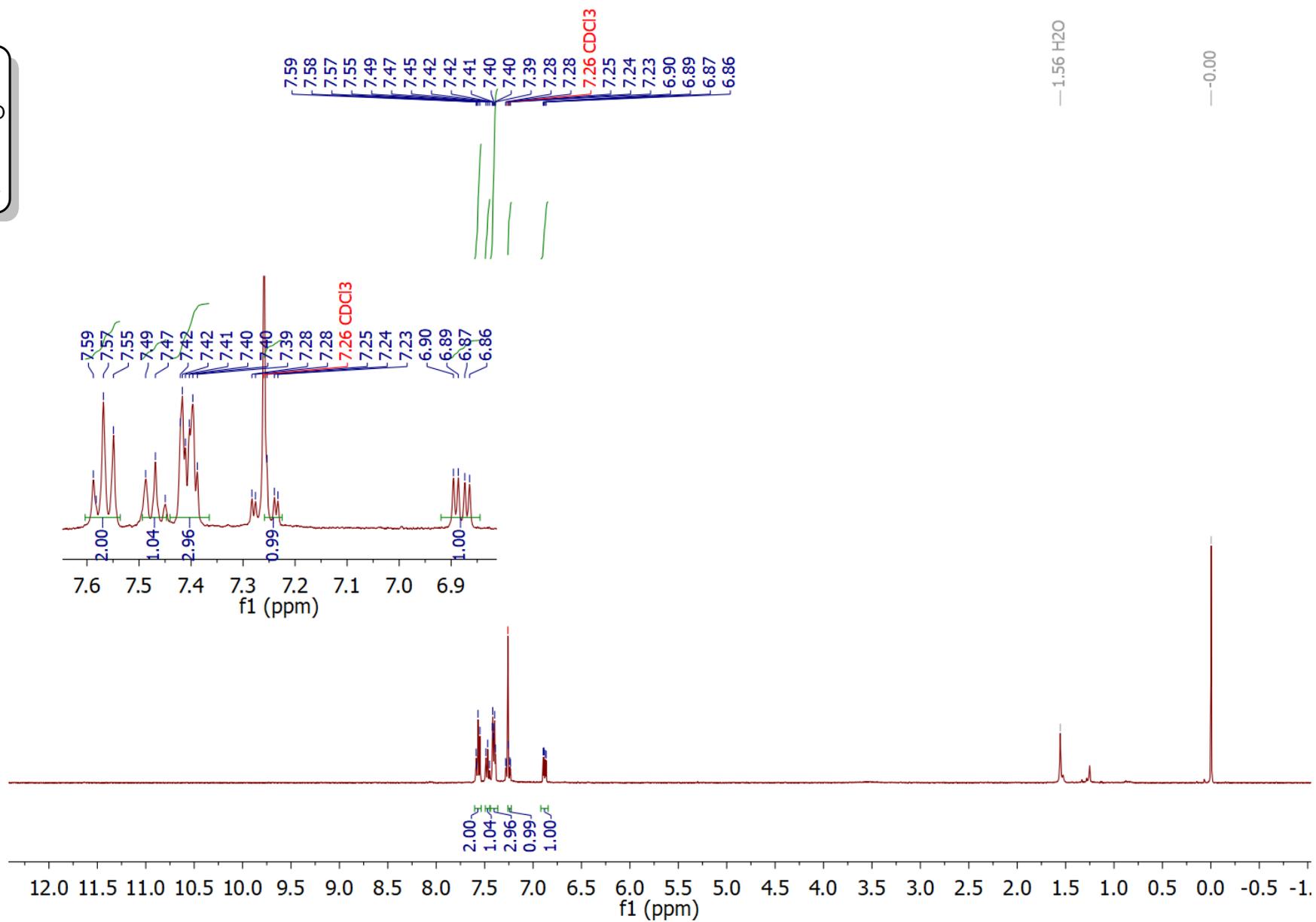
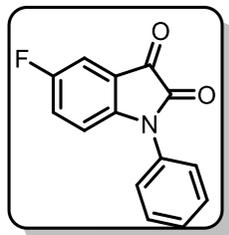
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- 31 WO2004/014376A1.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of 1-phenylindoline-2,3-dione (**3a**)

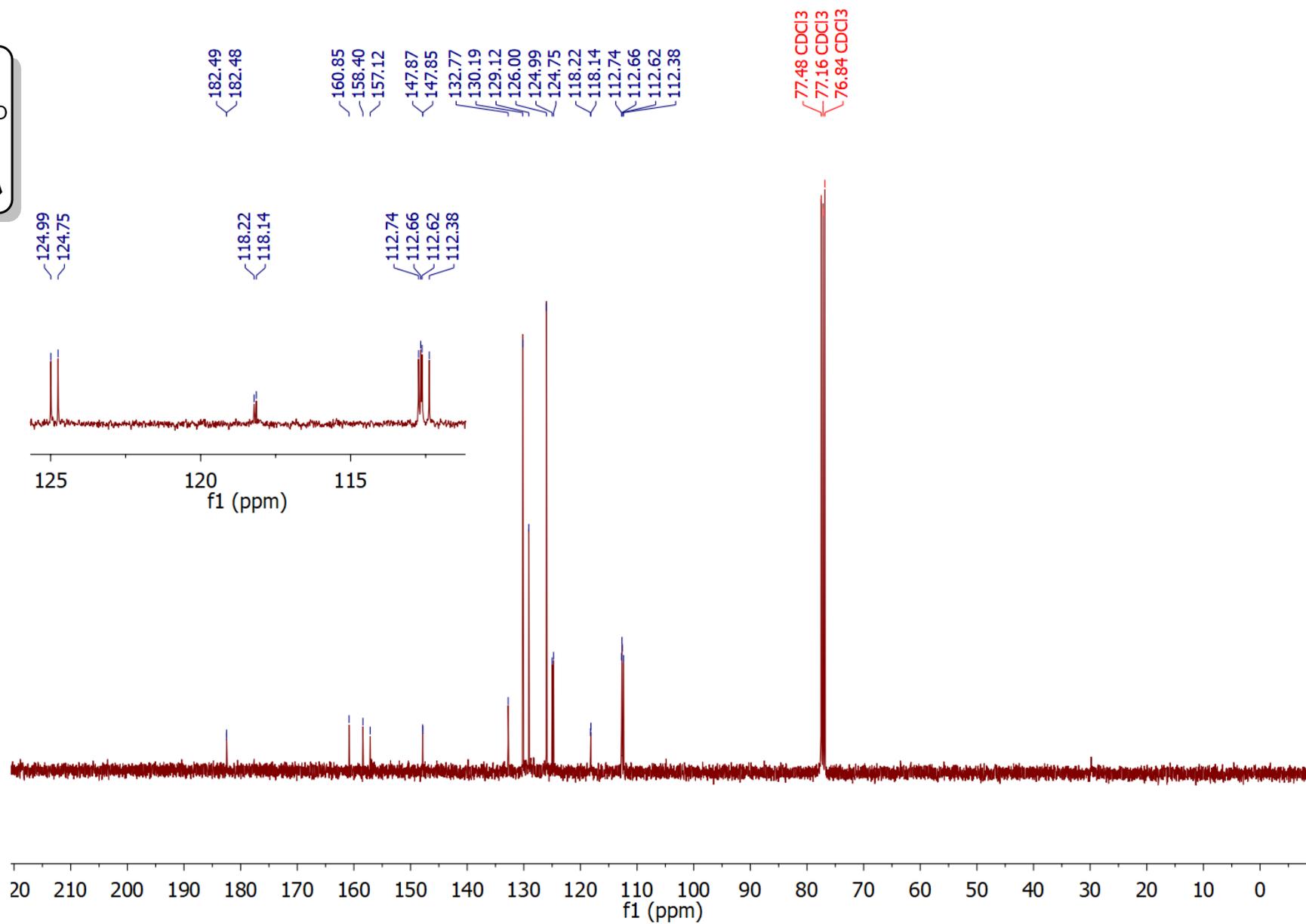
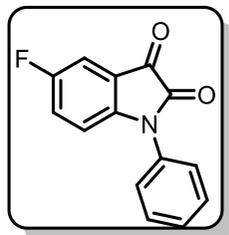
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-phenylindoline-2,3-dione (**3a**)



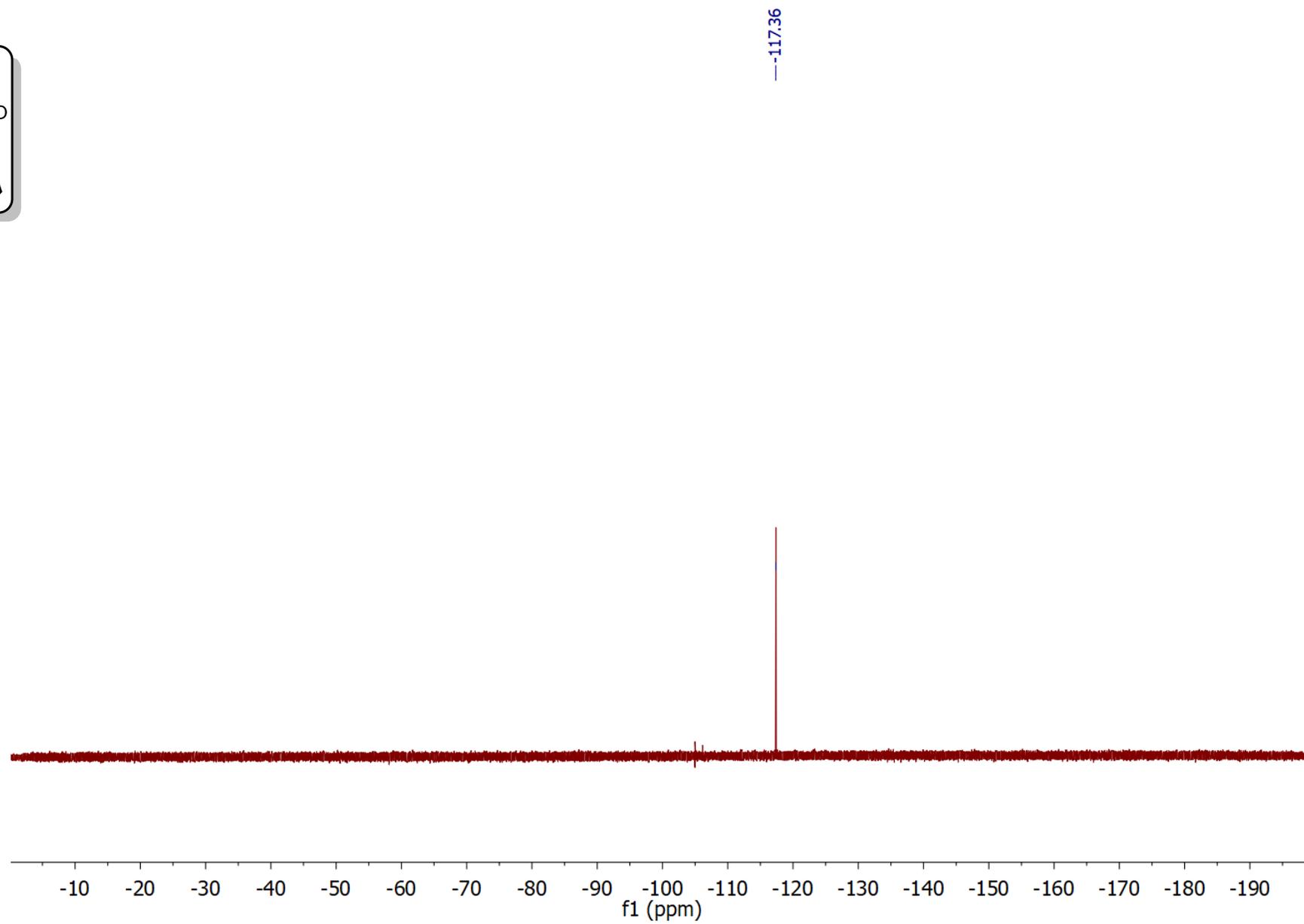
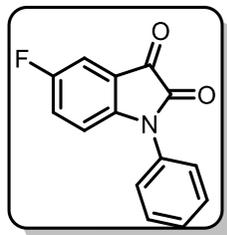
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 5-fluoro-1-phenylindoline-2,3-dione (**3b**)



$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 5-fluoro-1-phenylindoline-2,3-dione (**3b**)

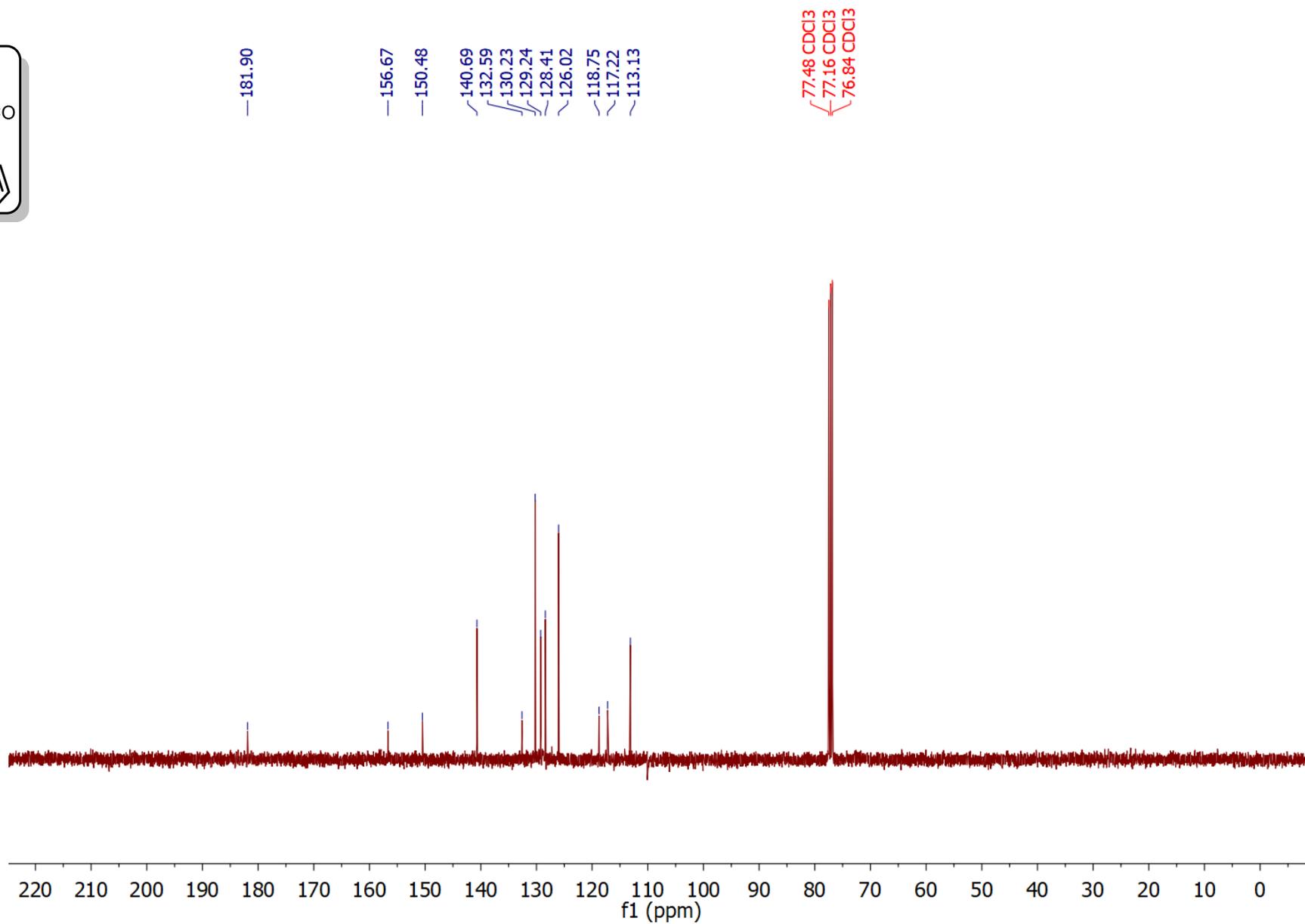
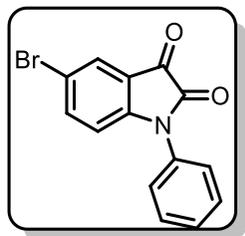


$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) of 5-fluoro-1-phenylindoline-2,3-dione (**3b**)

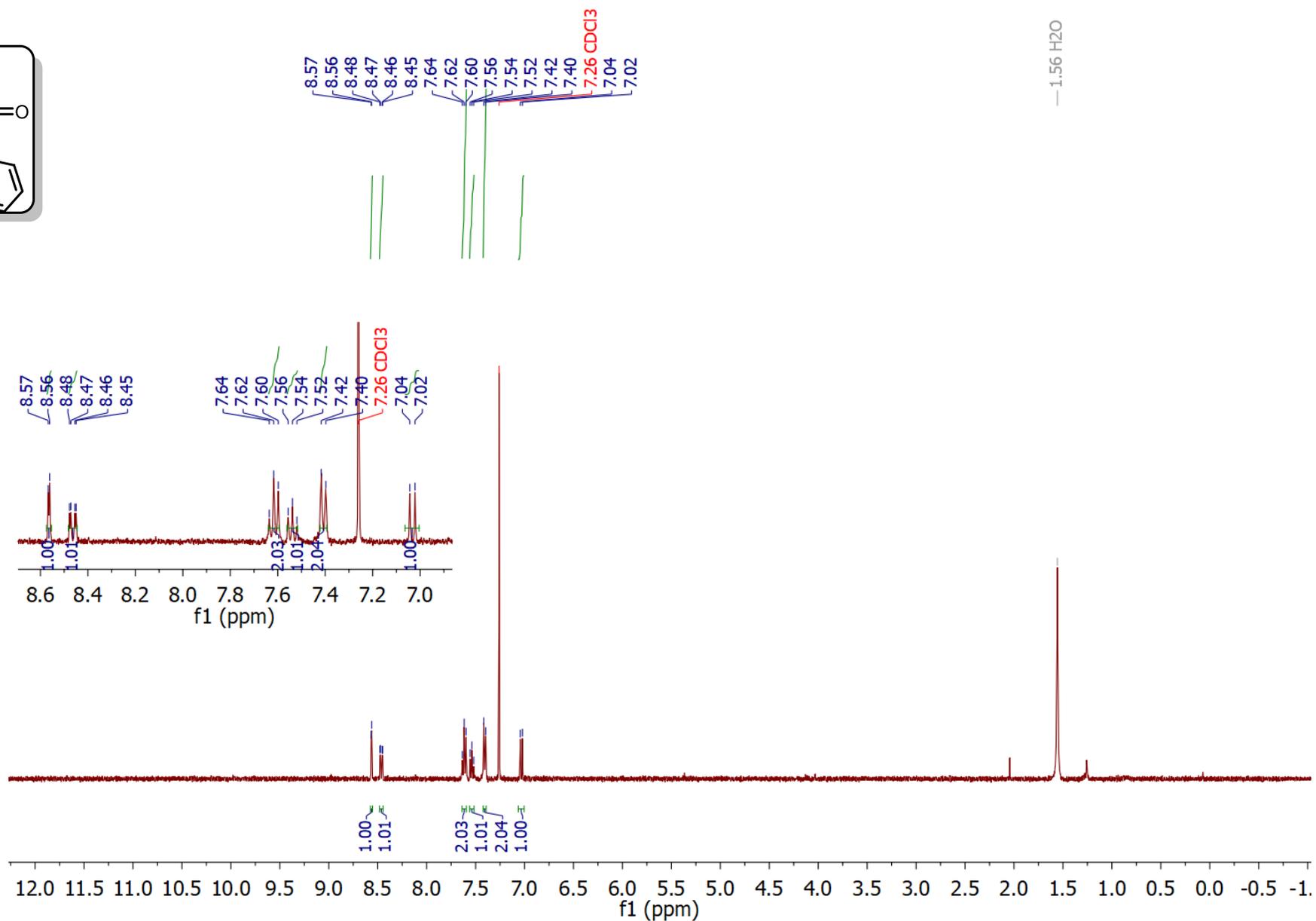
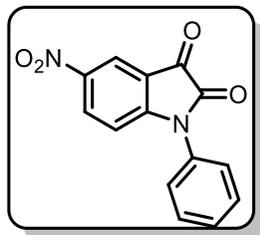




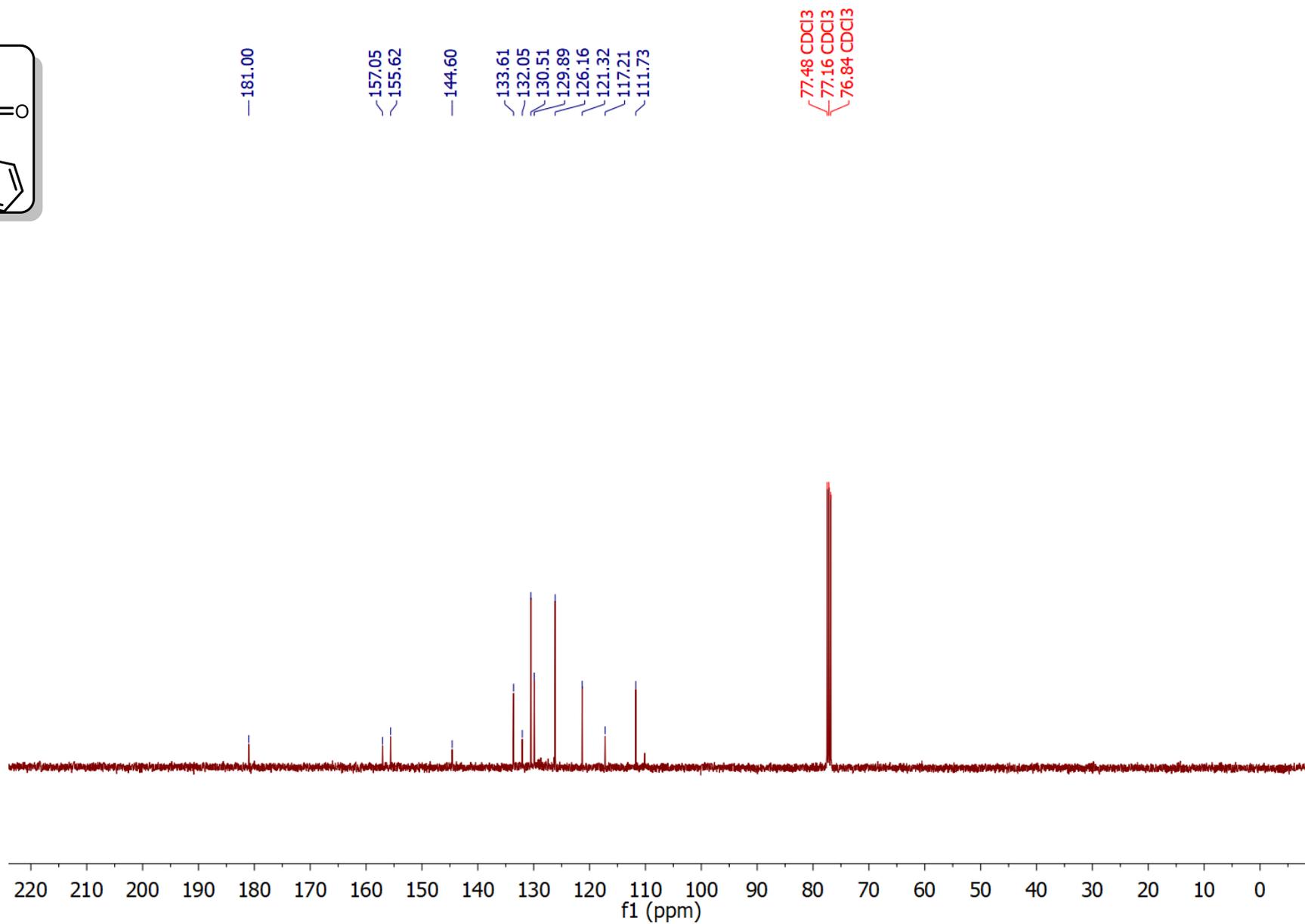
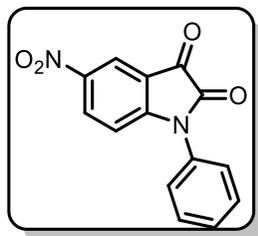
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 5-bromo-1-phenylindoline-2,3-dione (**3c**)



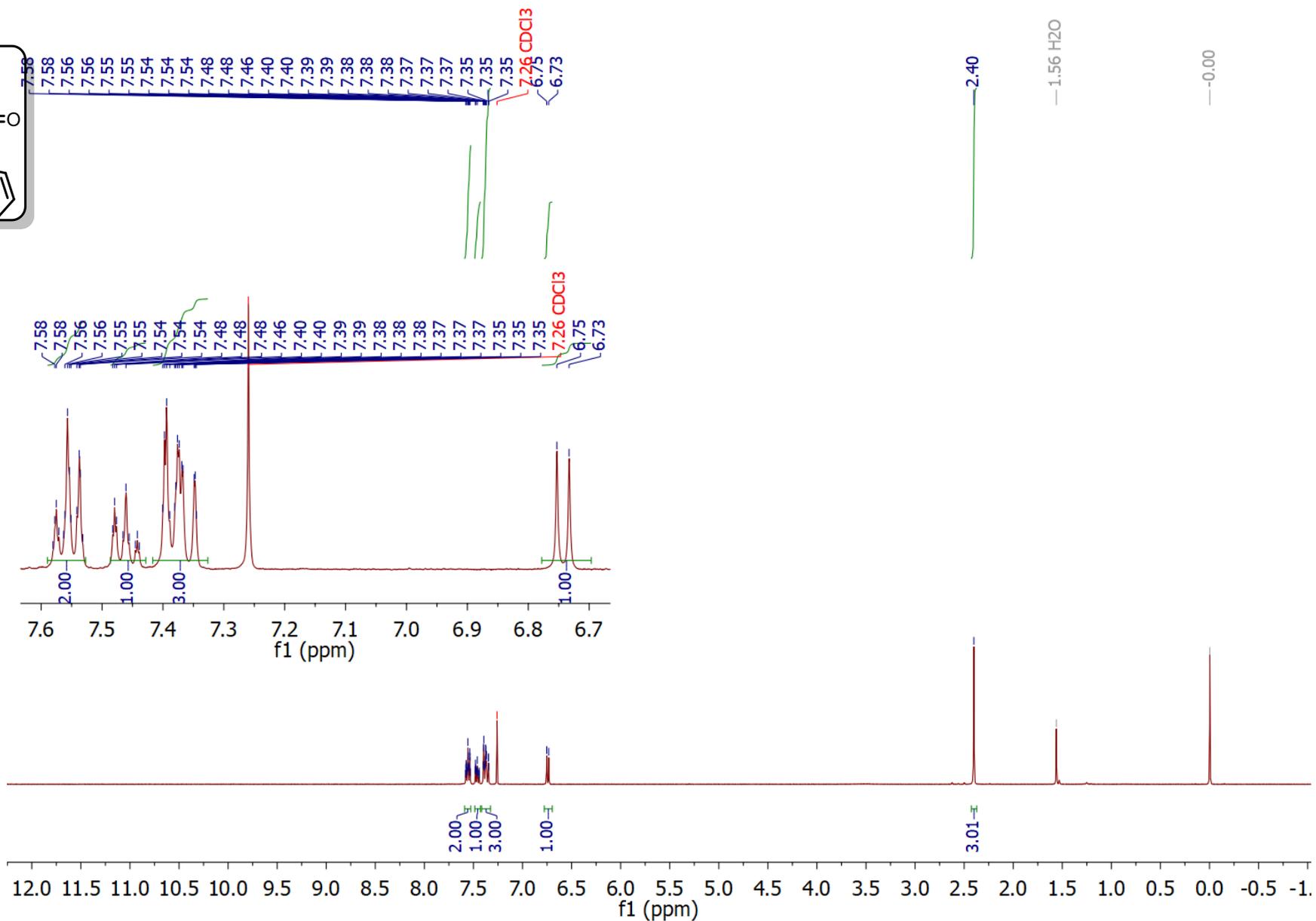
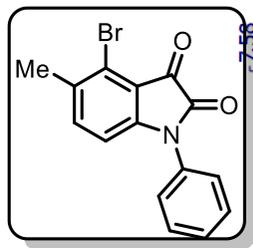
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 5-nitro-1-phenylindoline-2,3-dione (**3d**)



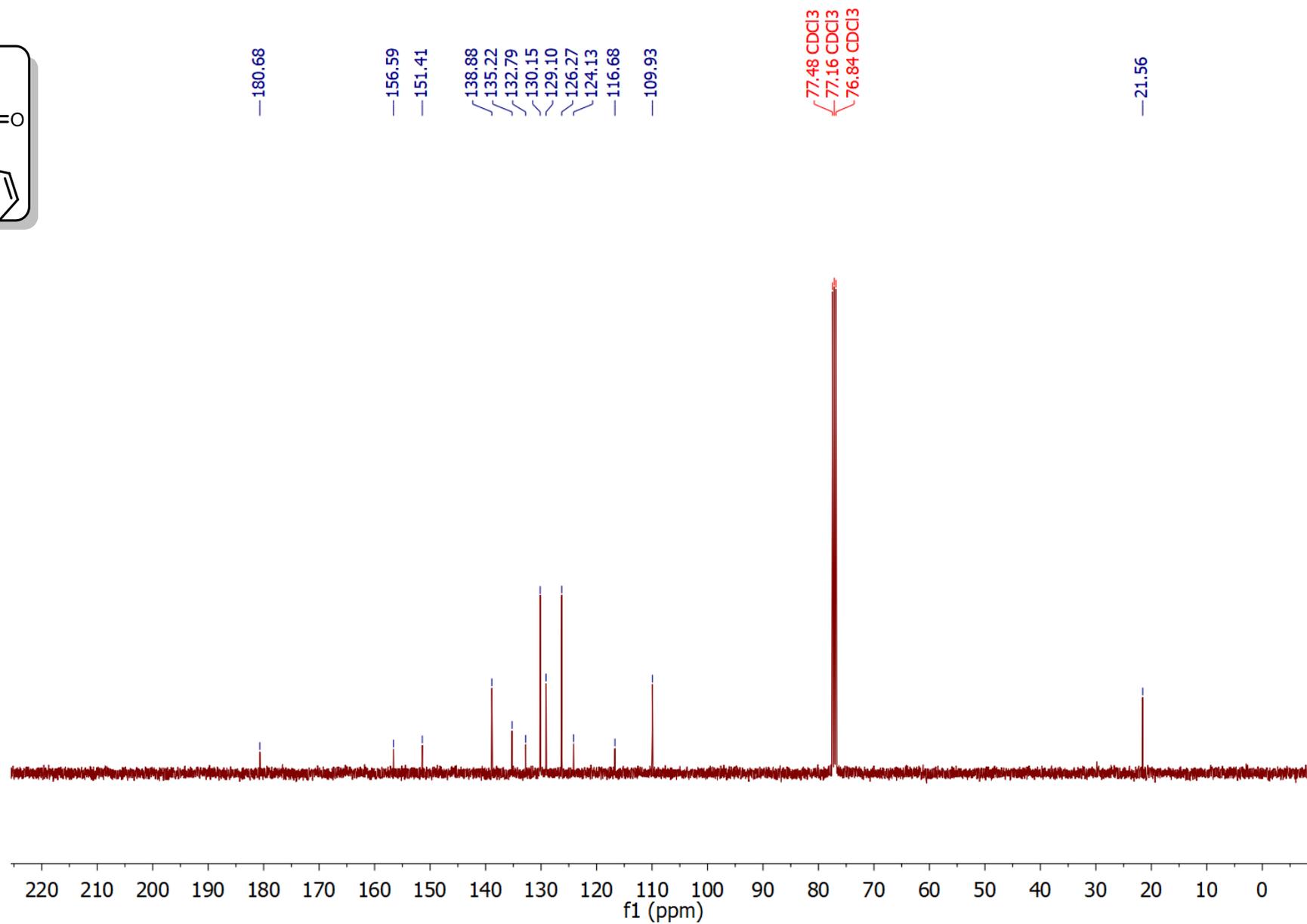
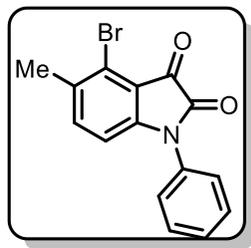
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 5-nitro-1-phenylindoline-2,3-dione (**3d**)



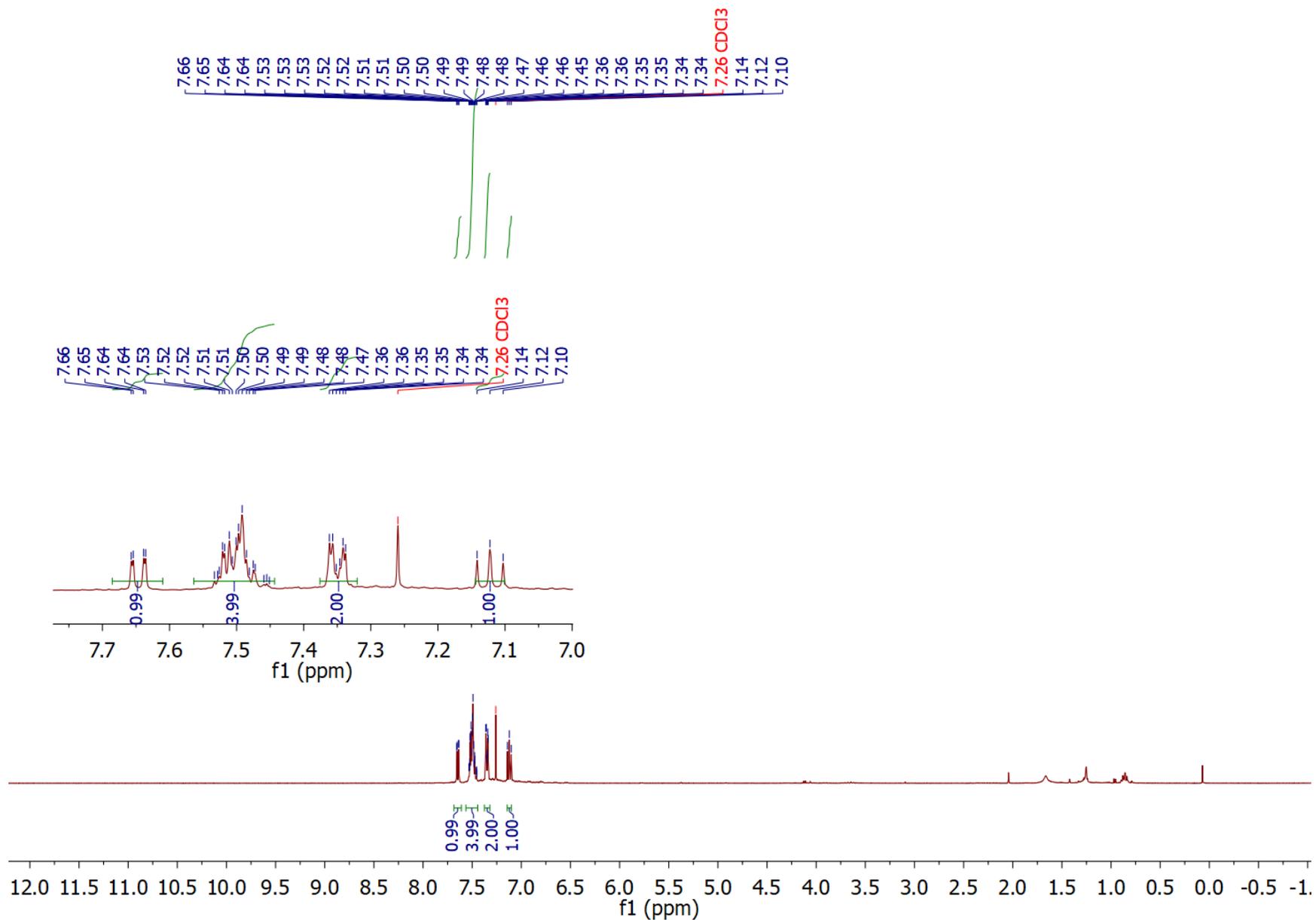
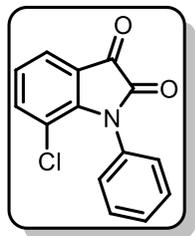
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 4-bromo-5-methyl-1-phenylindoline-2,3-dione (**3e**)



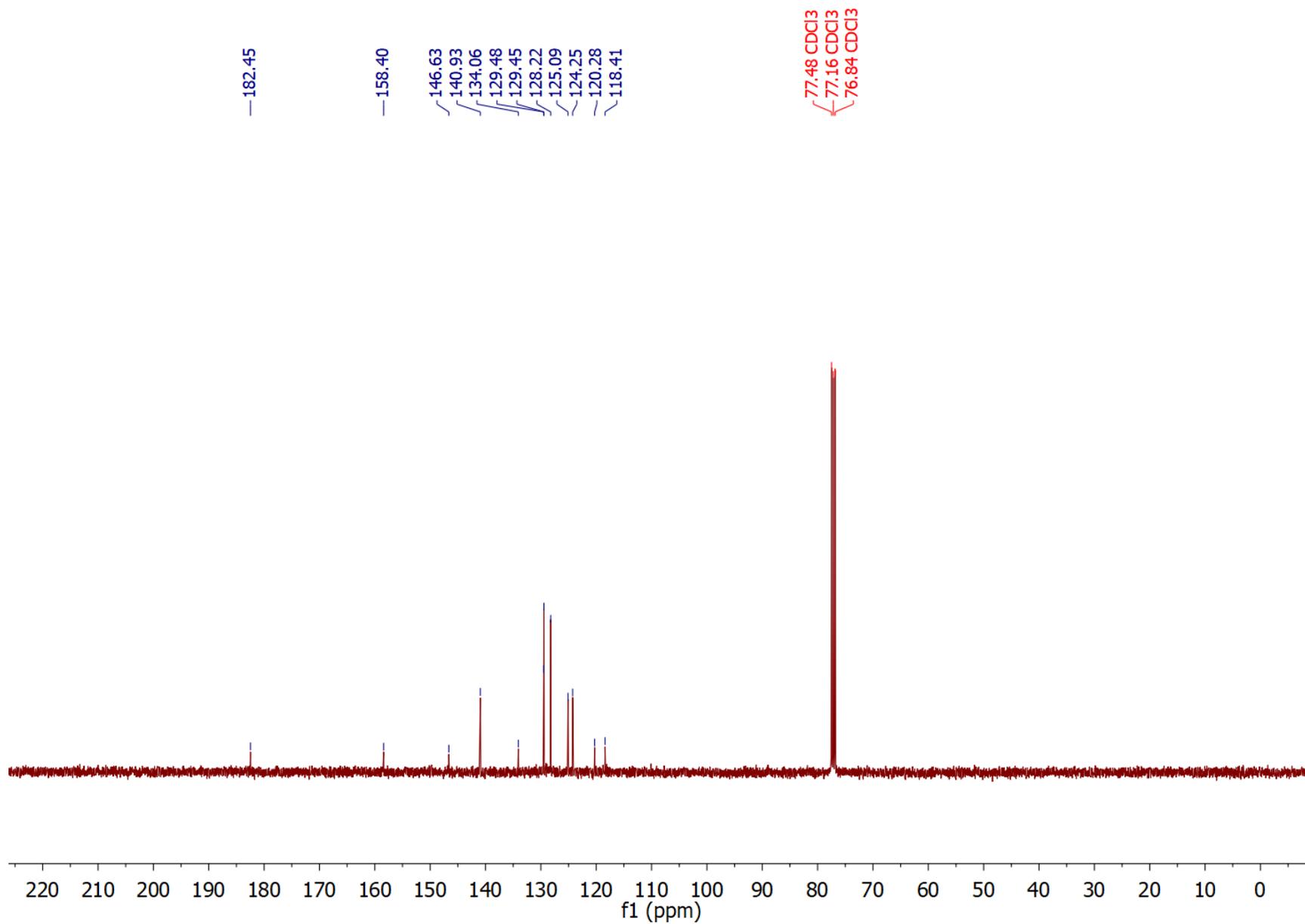
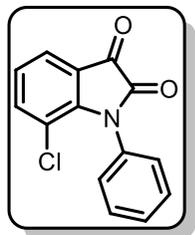
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 4-bromo-5-methyl-1-phenylindoline-2,3-dione (**3e**)



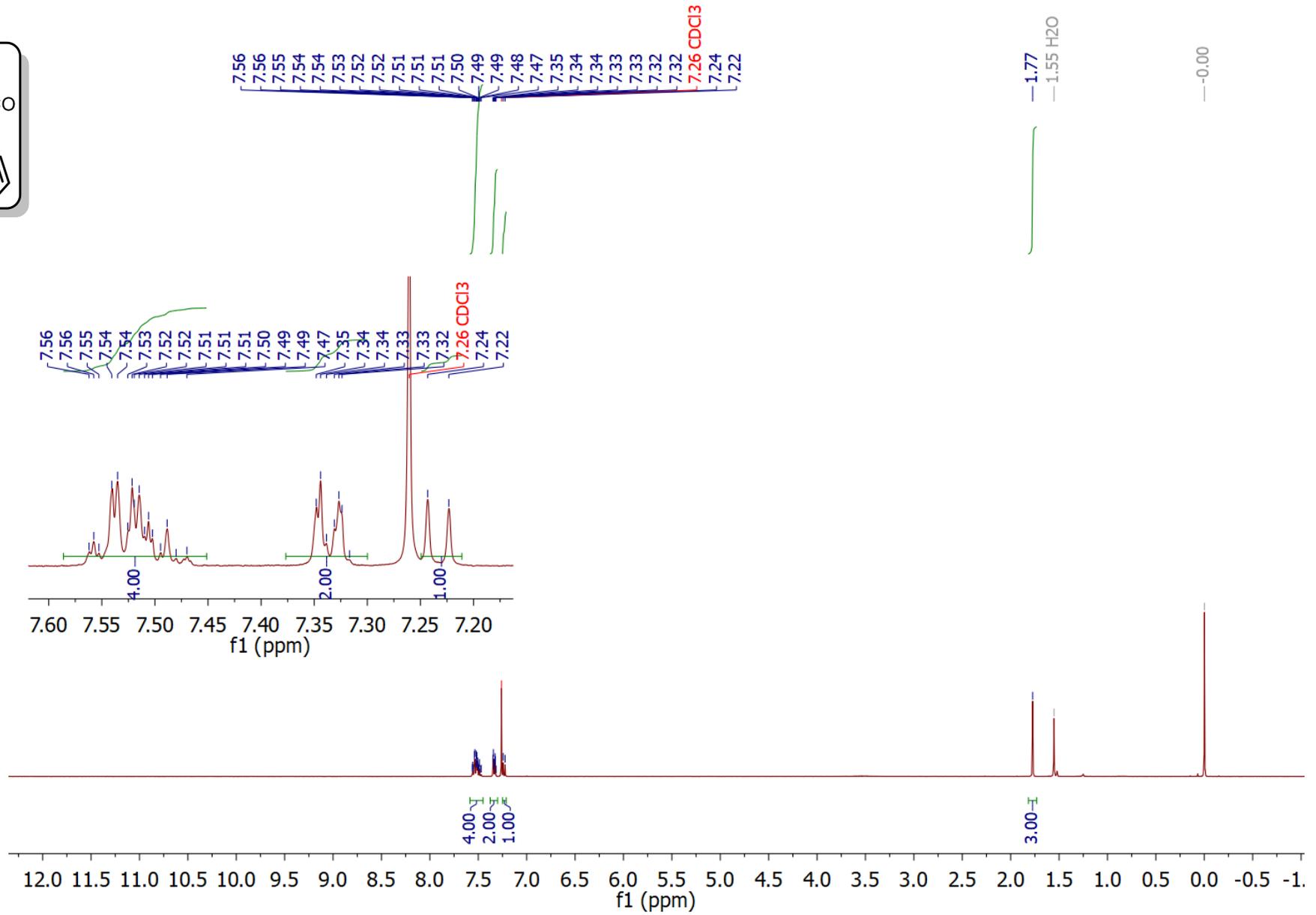
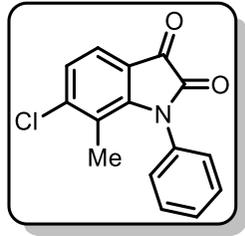
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 7-chloro-1-phenylindoline-2,3-dione (**3f**)



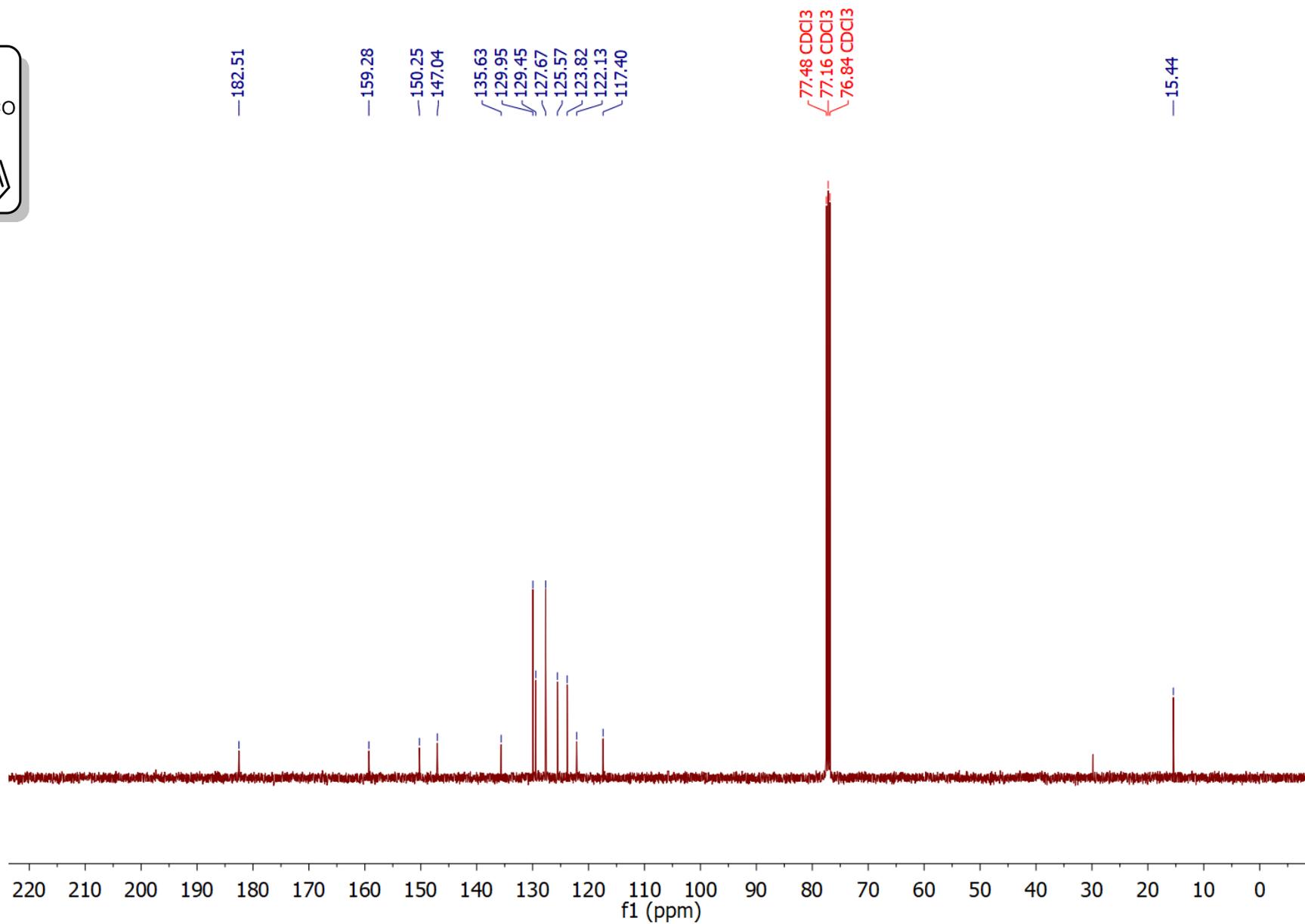
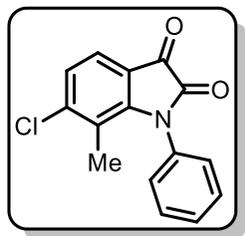
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 7-chloro-1-phenylindoline-2,3-dione (**3f**)



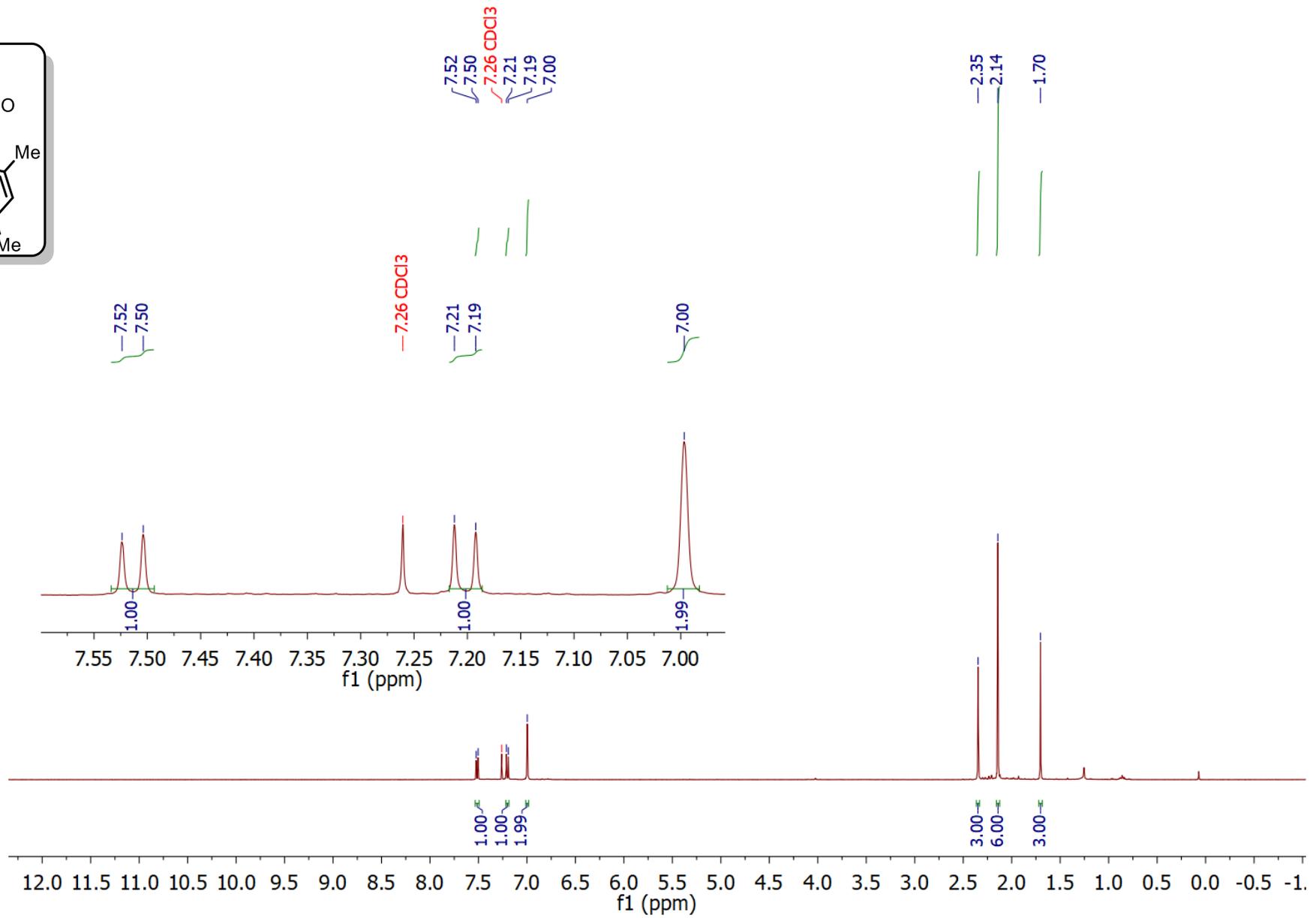
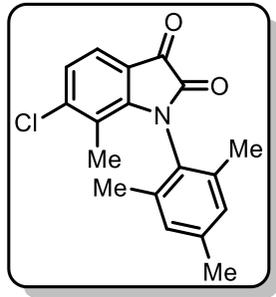
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 6-chloro-7-methyl-1-phenylindoline-2,3-dione (**3g**)



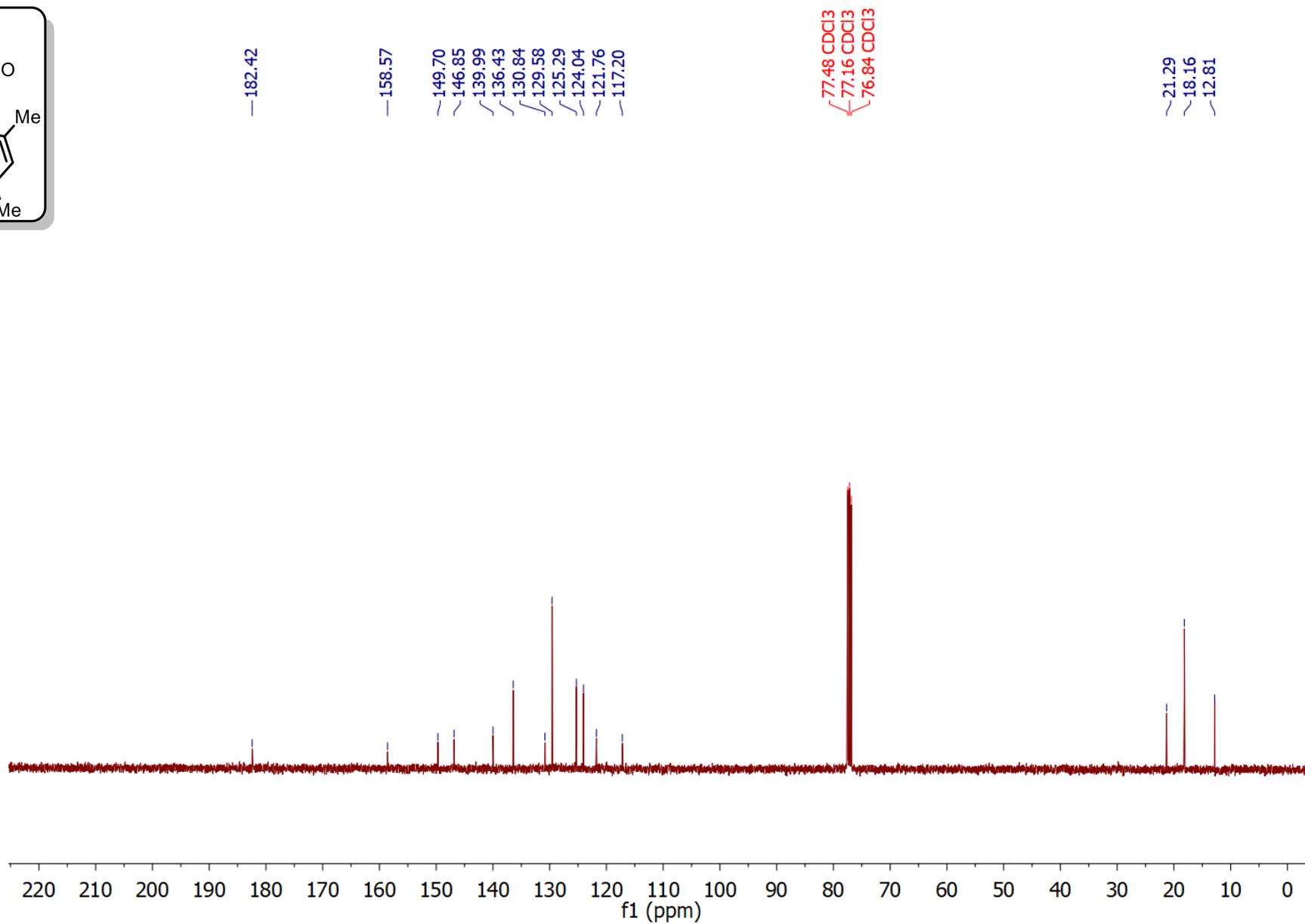
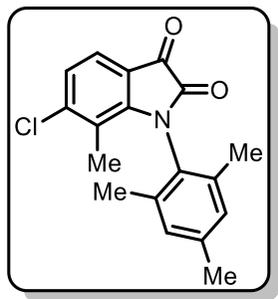
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 6-chloro-7-methyl-1-phenylindoline-2,3-dione (**3g**)



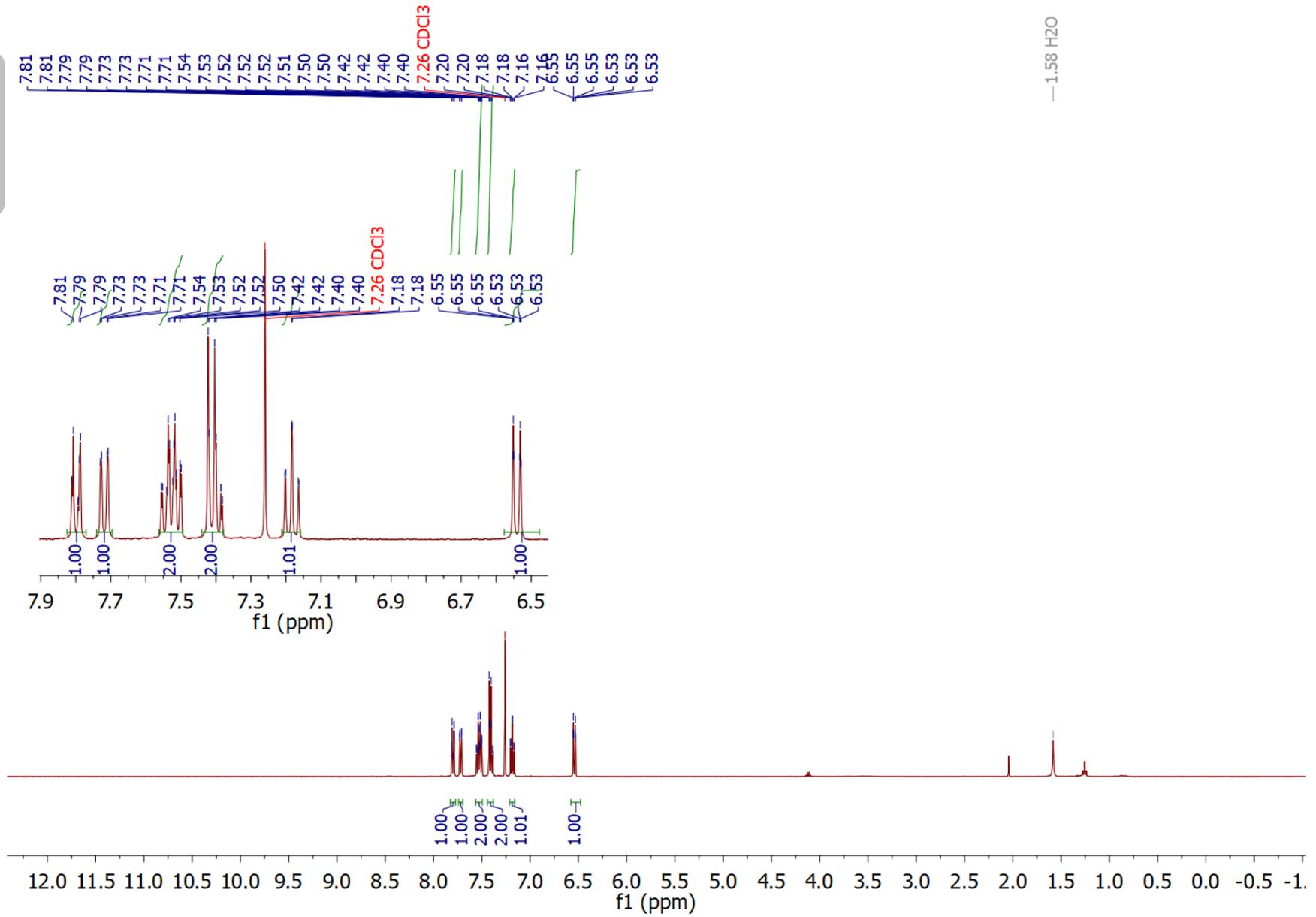
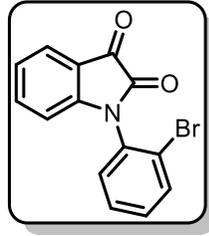
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 6-chloro-1-mesityl-7-methylindoline-2,3-dione (**3h**)



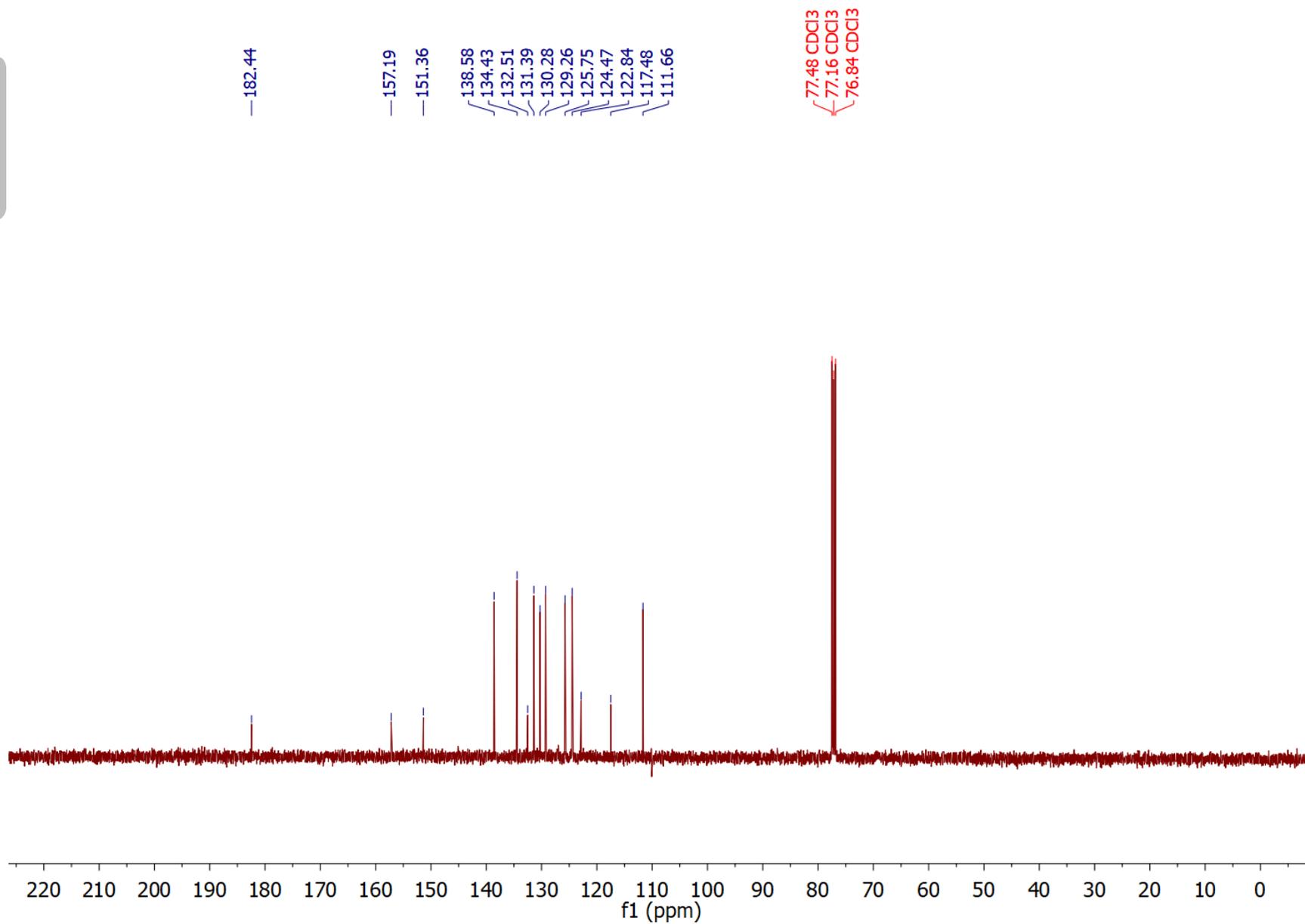
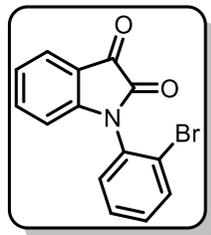
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 6-chloro-1-mesityl-7-methylindoline-2,3-dione (**3h**)



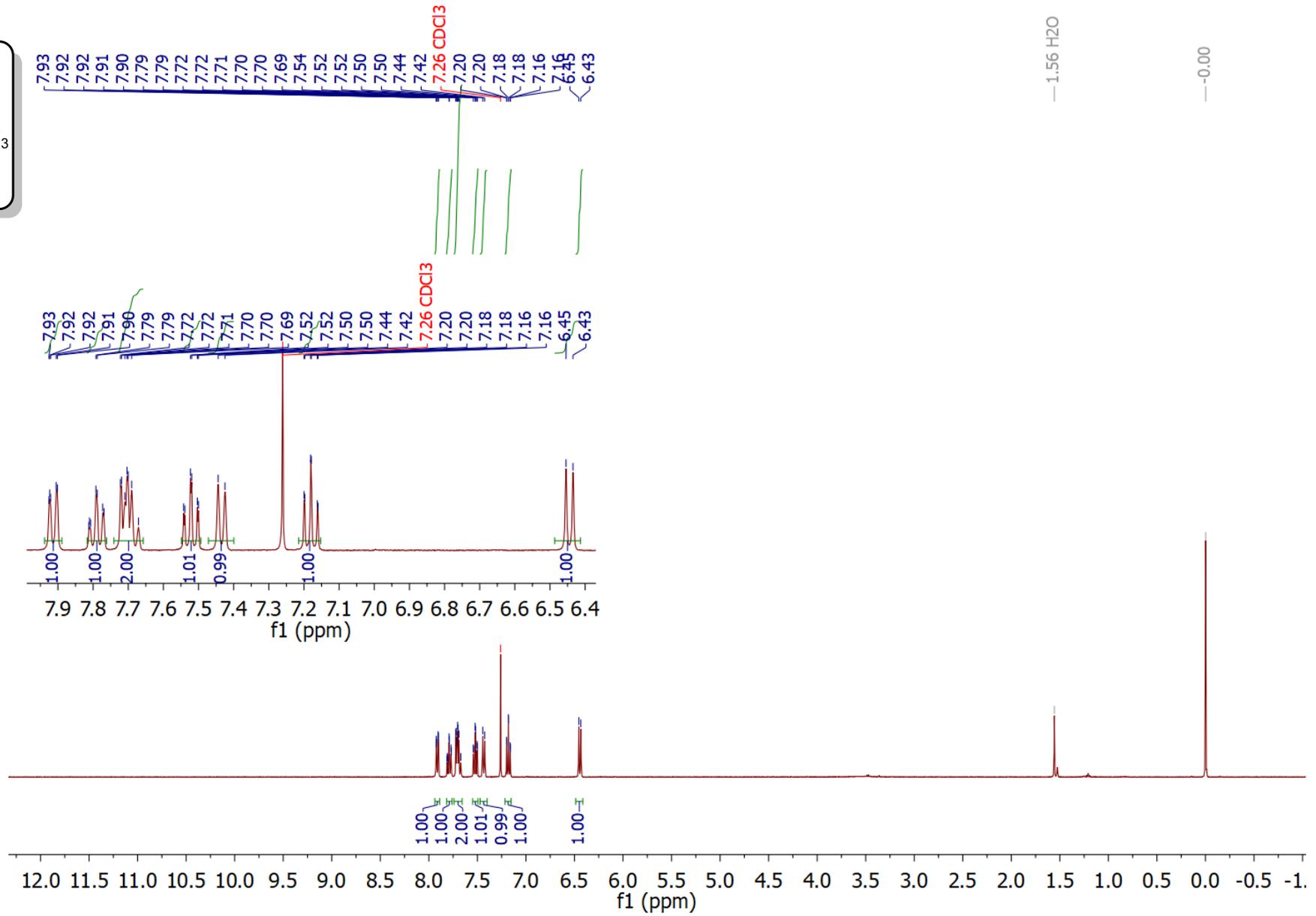
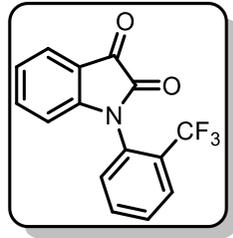
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(2-bromophenyl)indoline-2,3-dione (**3i**)



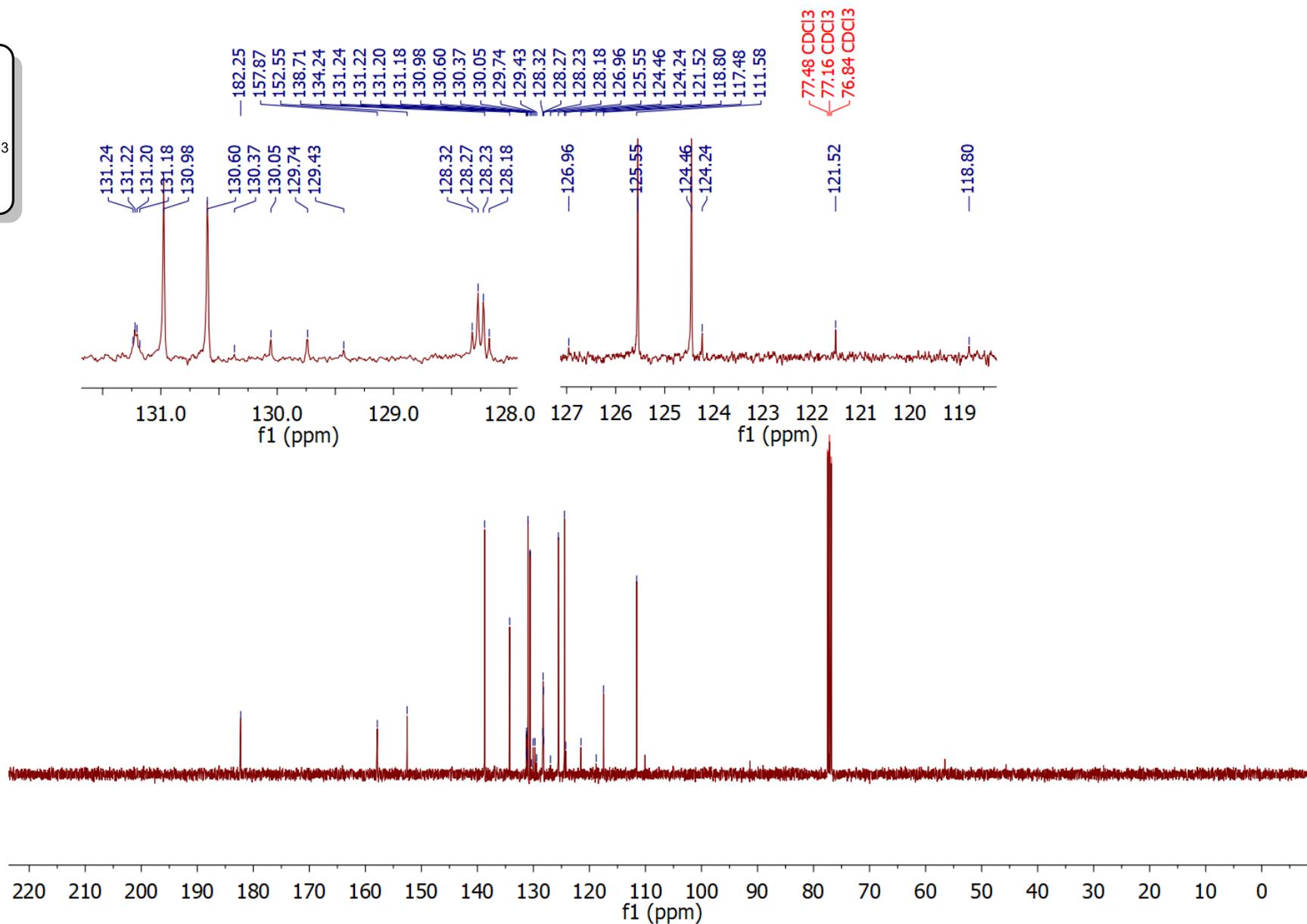
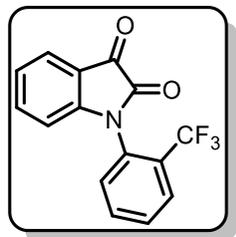
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(2-bromophenyl)indoline-2,3-dione (**3i**)



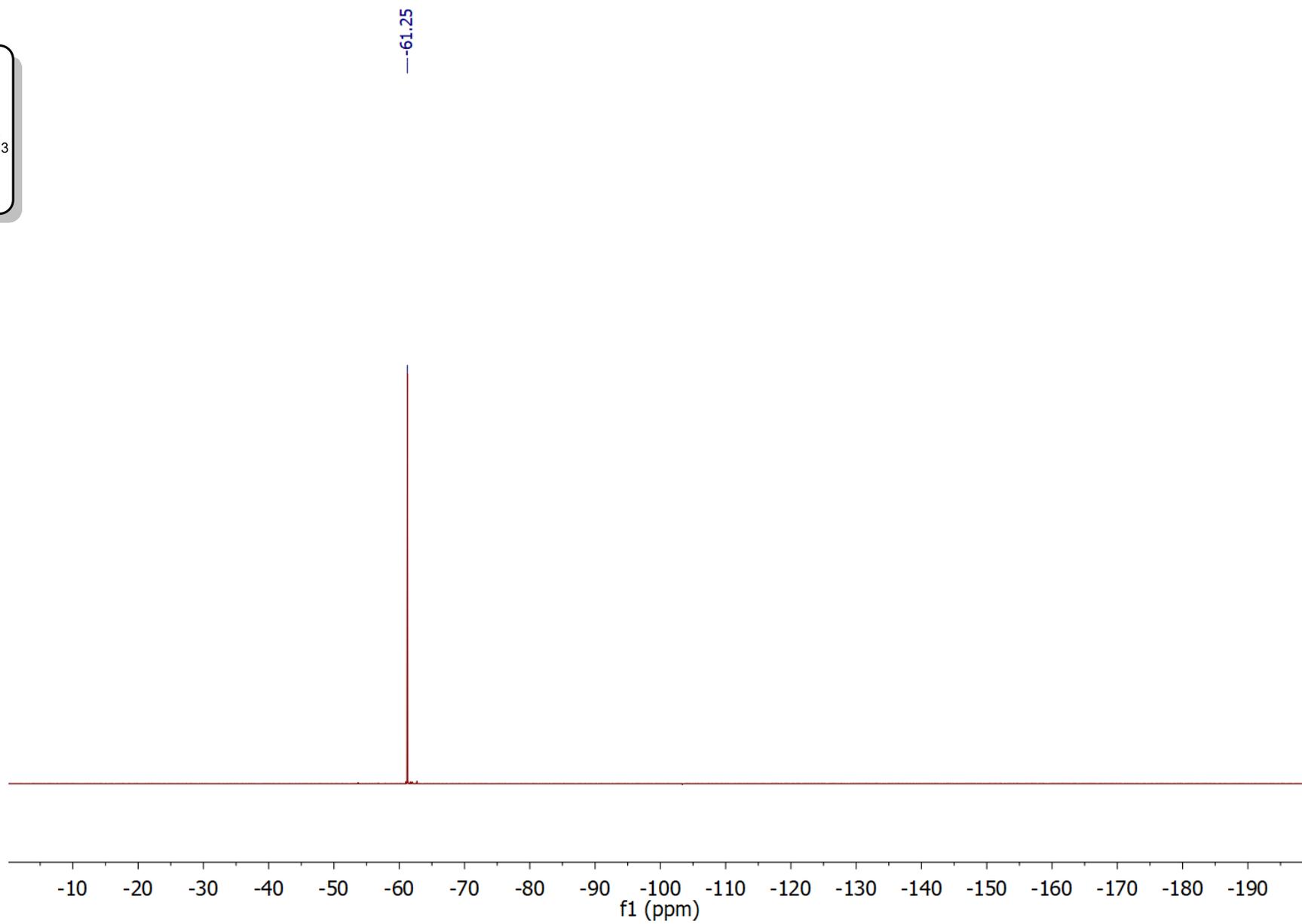
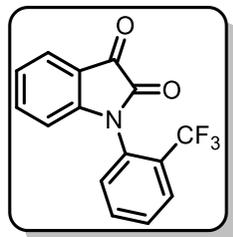
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(2-(trifluoromethyl)phenyl)indoline-2,3-dione (**3j**)



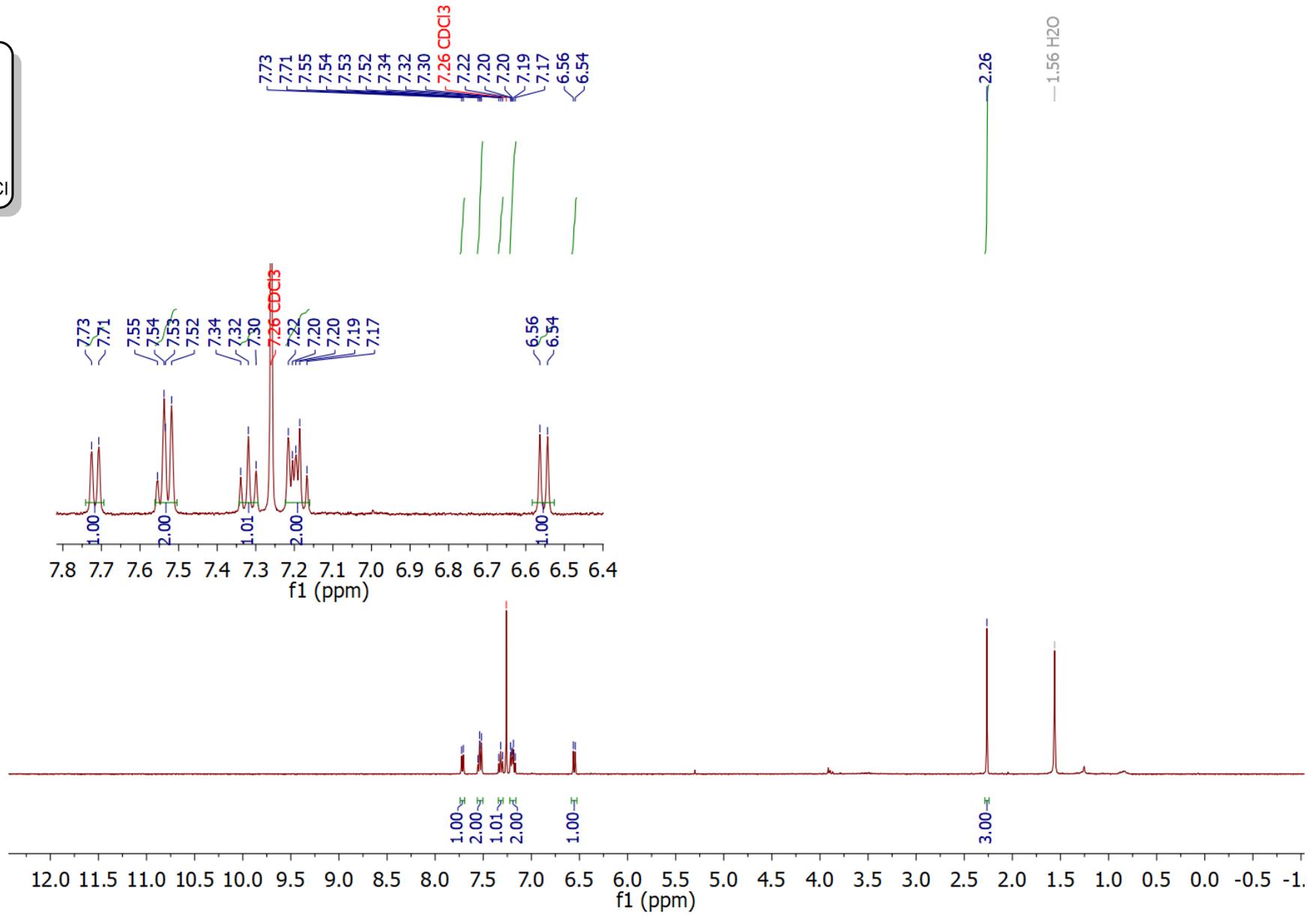
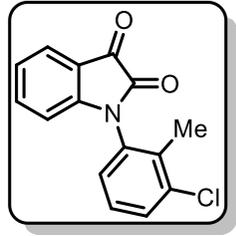
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(2-(trifluoromethyl)phenyl)indoline-2,3-dione (**3j**)



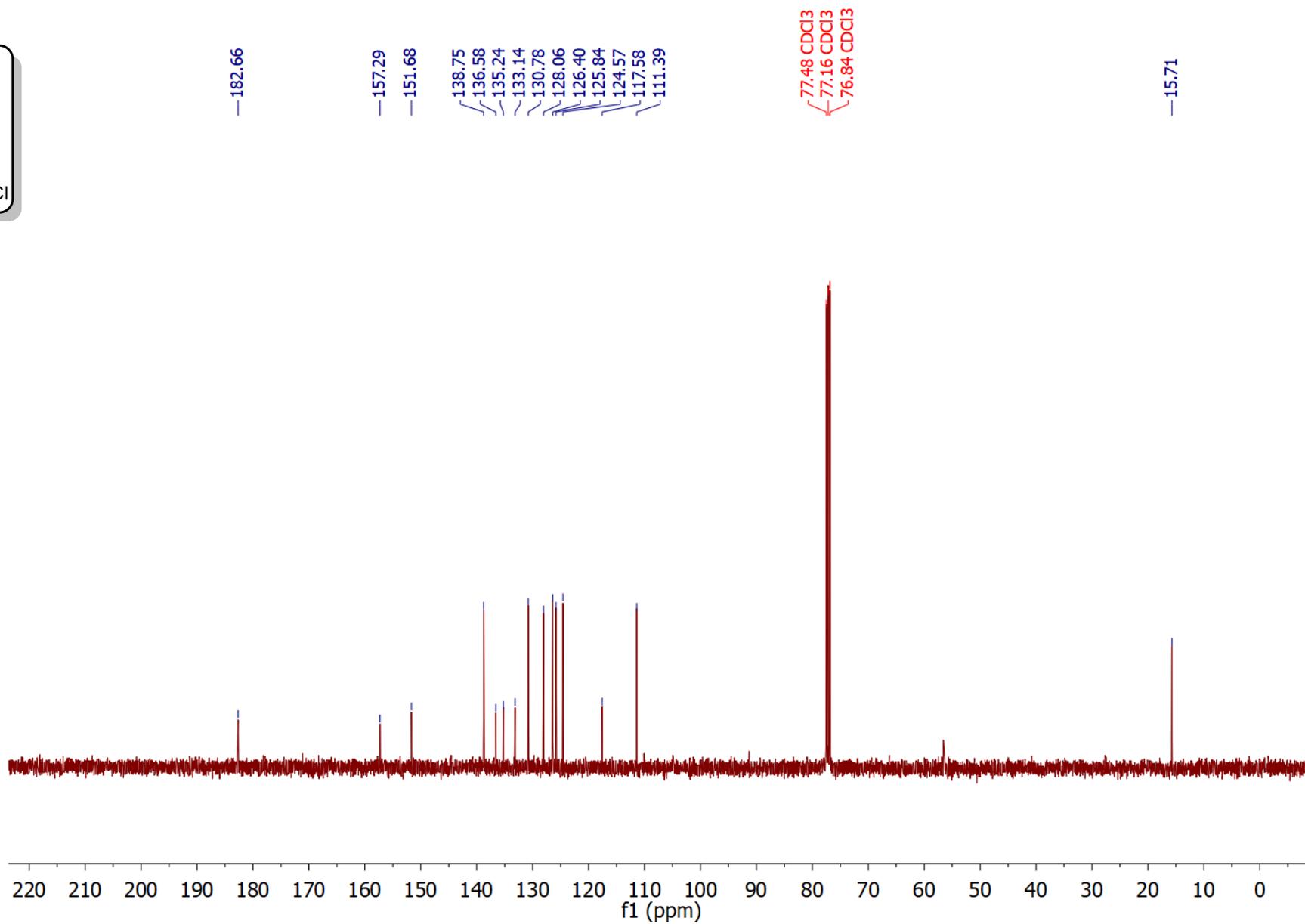
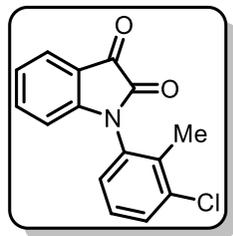
$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) of 1-(2-(trifluoromethyl)phenyl)indoline-2,3-dione (**3j**)



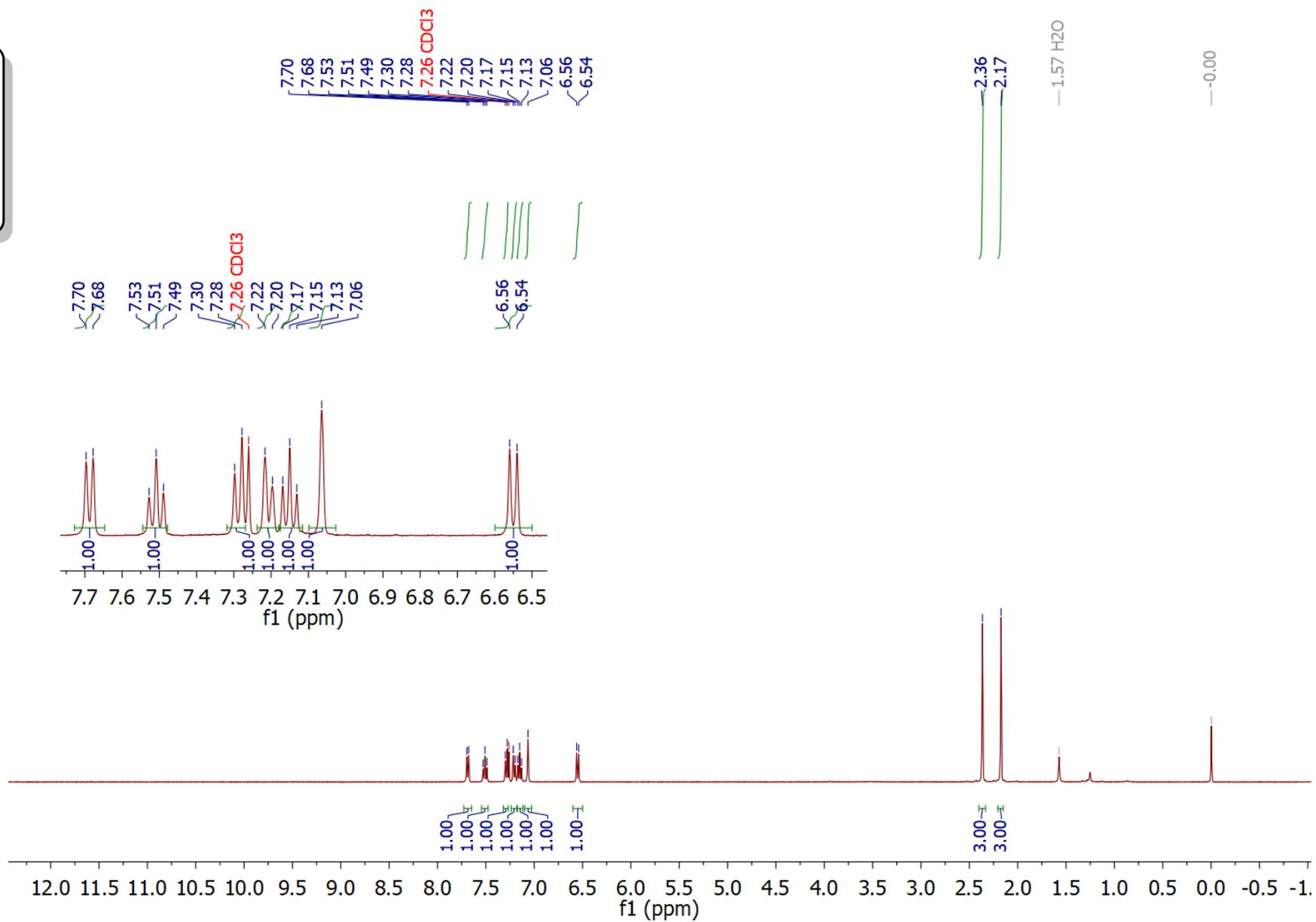
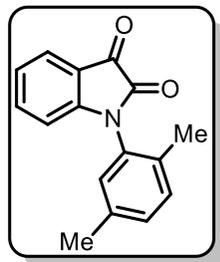
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(3-chloro-2-methylphenyl)indoline-2,3-dione (**3k**)



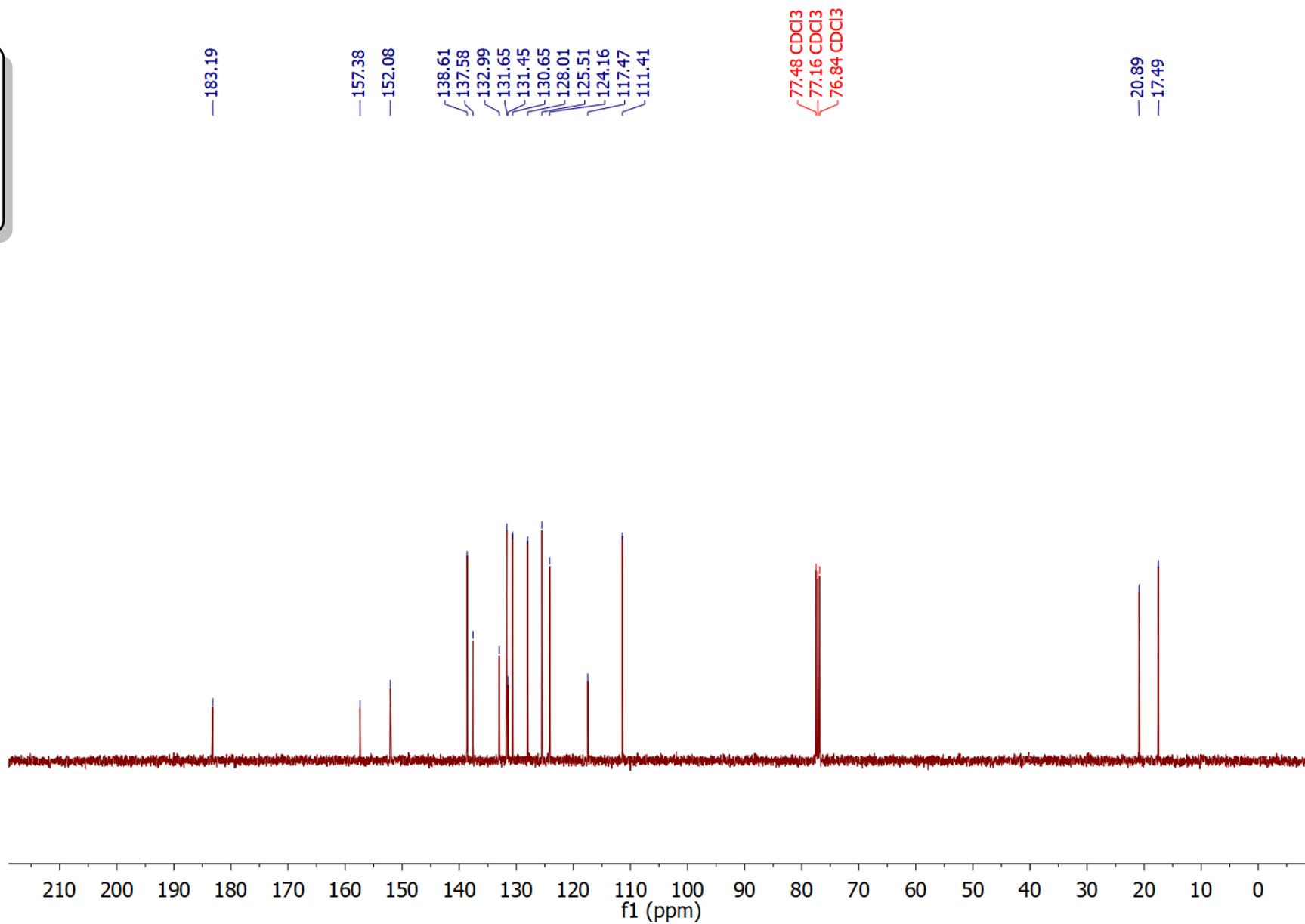
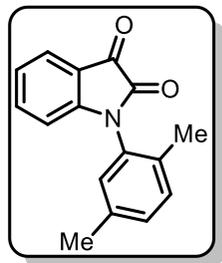
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(3-chloro-2-methylphenyl)indoline-2,3-dione (**3k**)



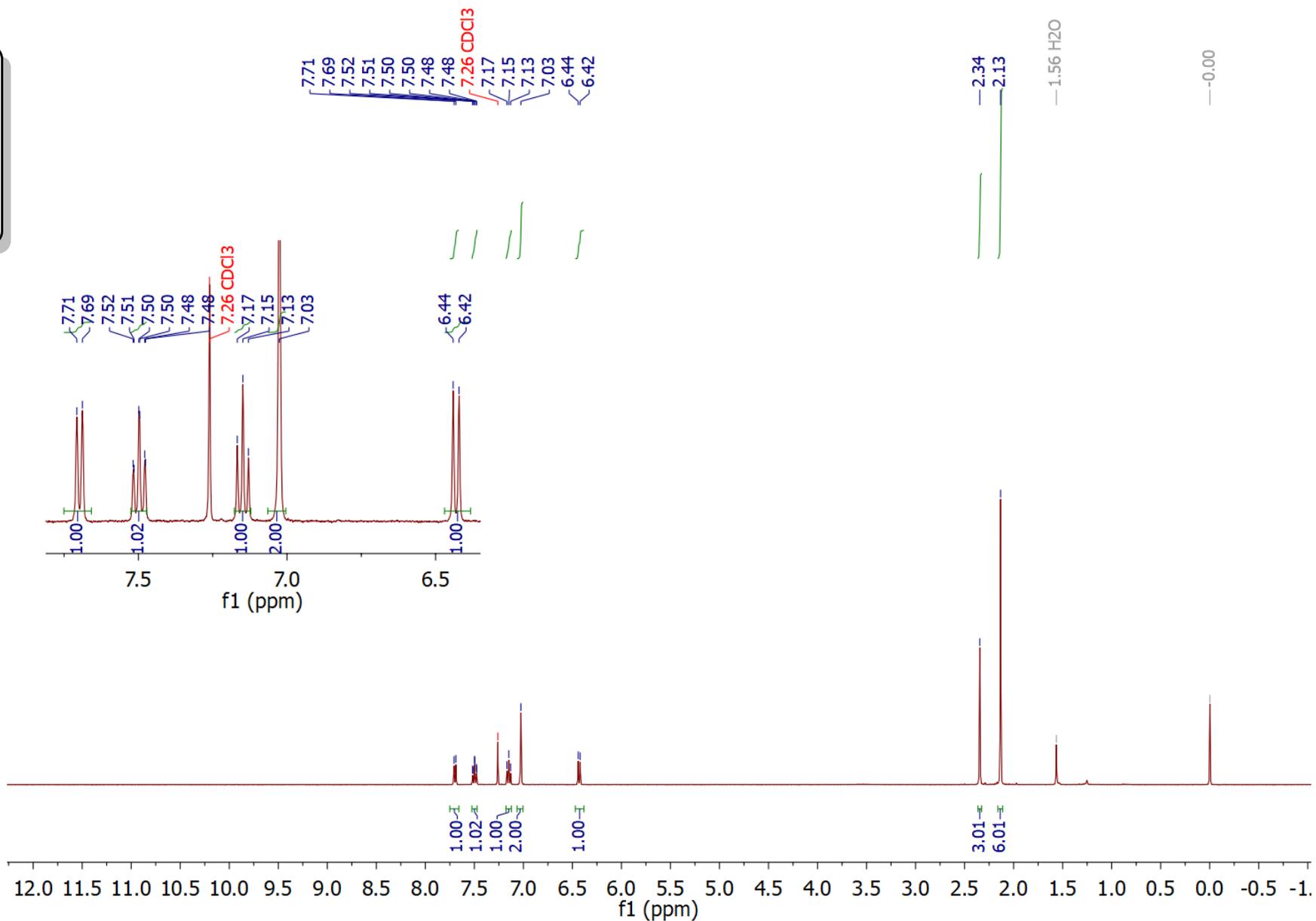
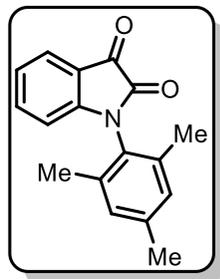
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(2,5-dimethylphenyl)indoline-2,3-dione (**31**)



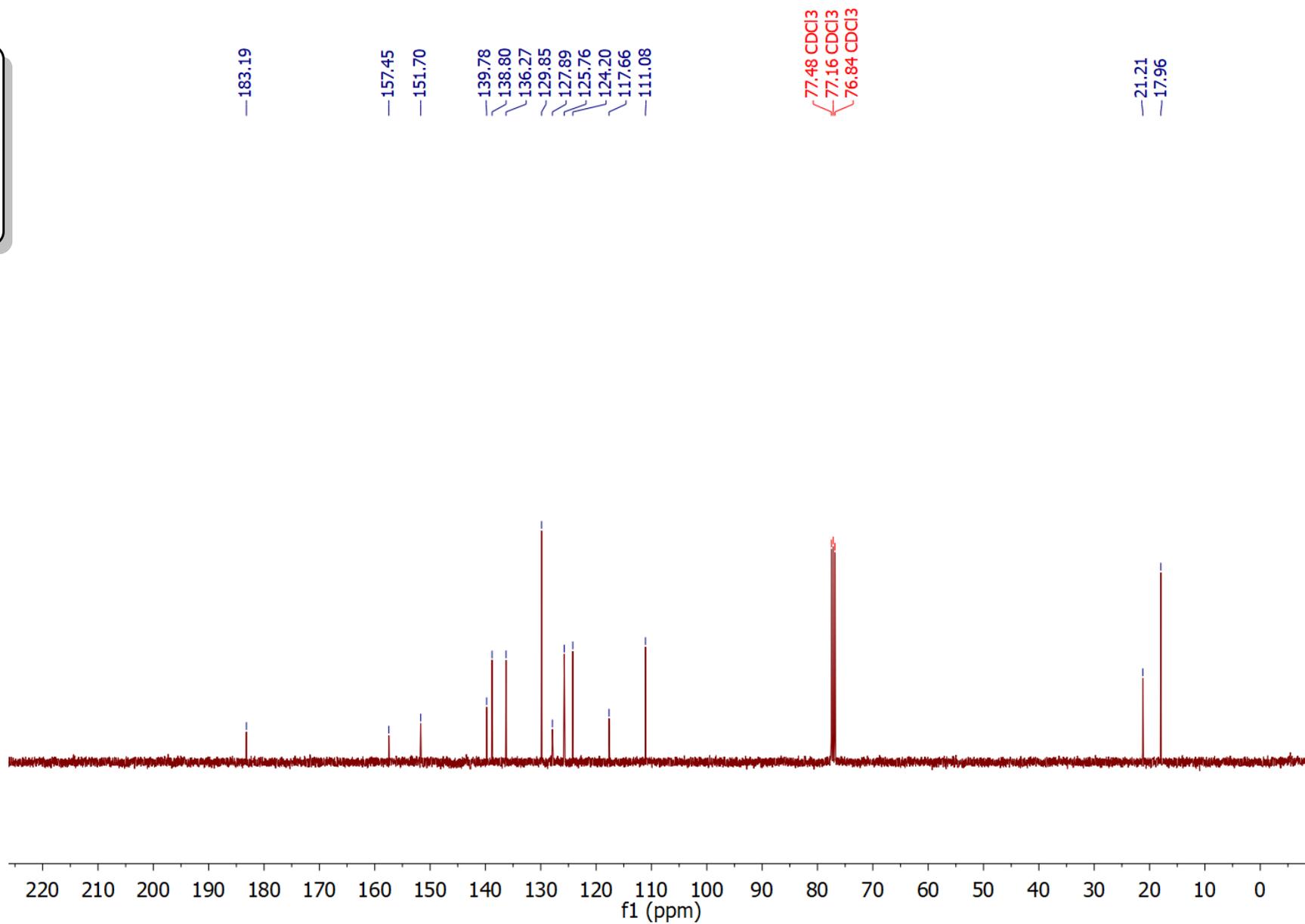
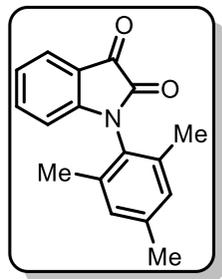
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(2,5-dimethylphenyl)indoline-2,3-dione (**3**)



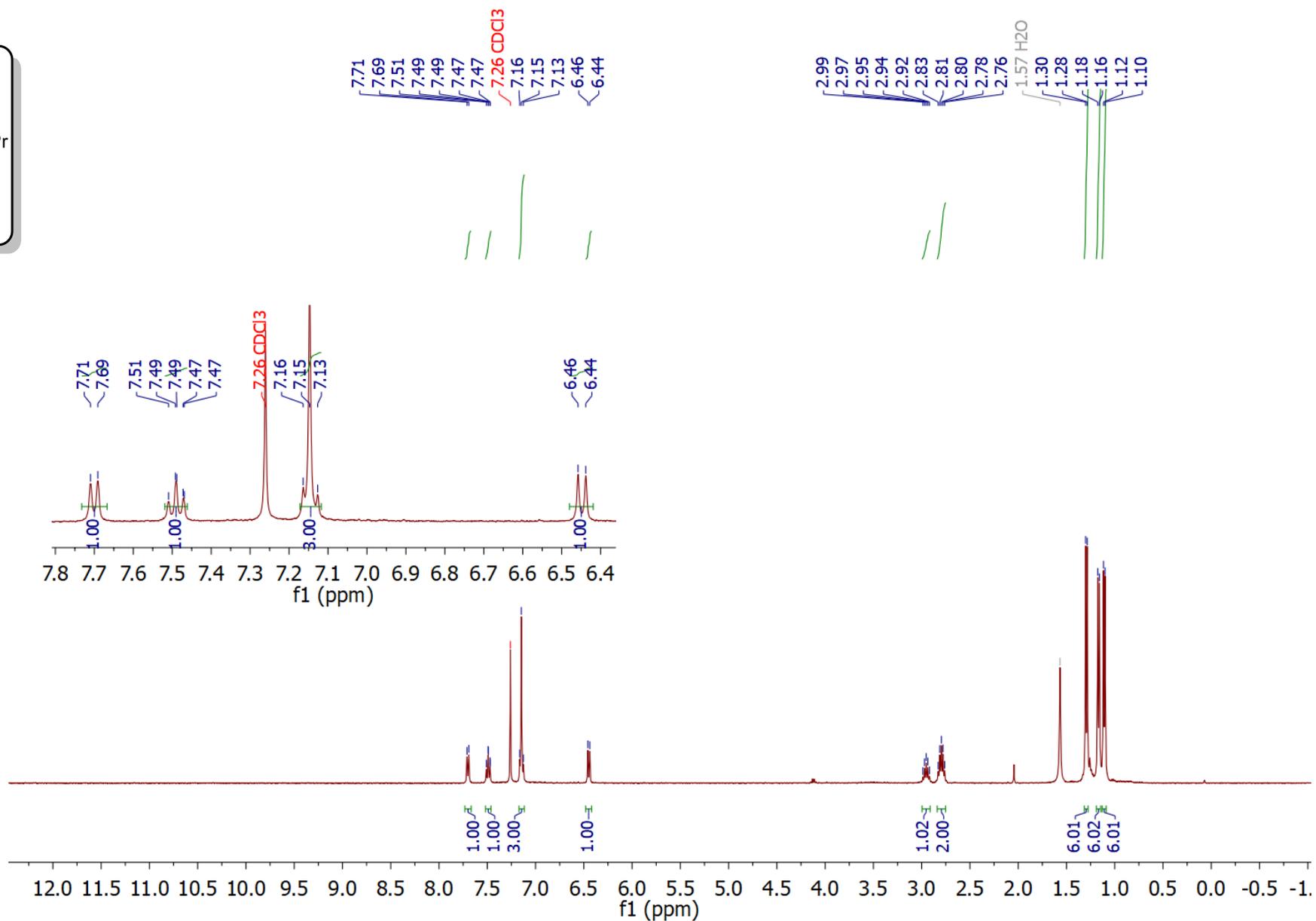
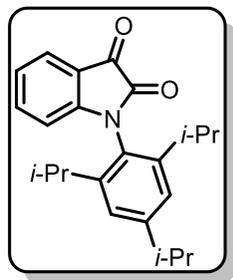
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-mesitylyndoline-2,3-dione (**3m**)



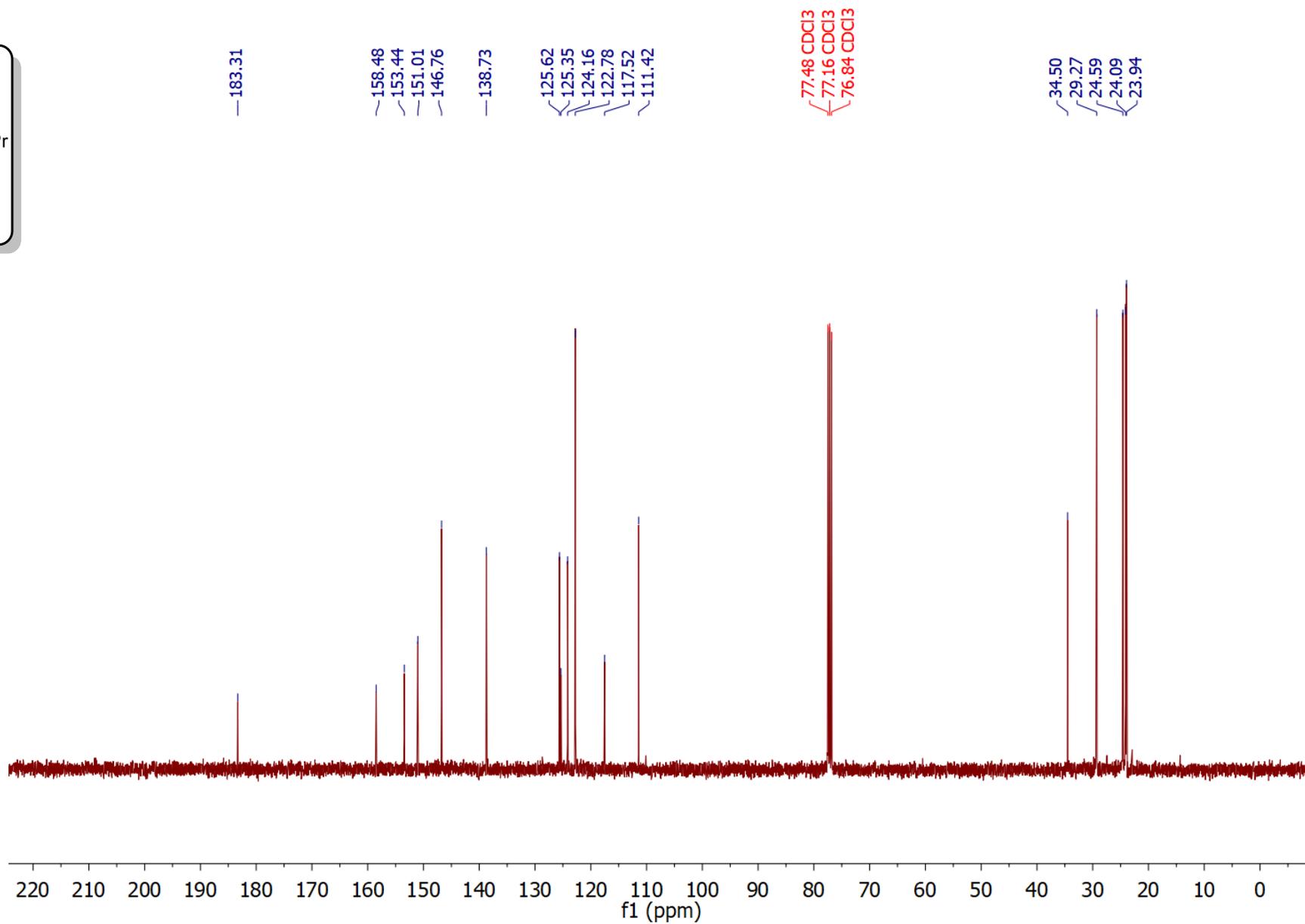
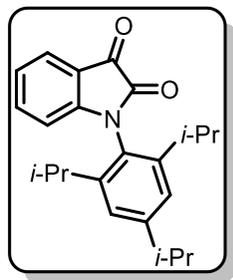
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-mesitylyndoline-2,3-dione (**3m**)



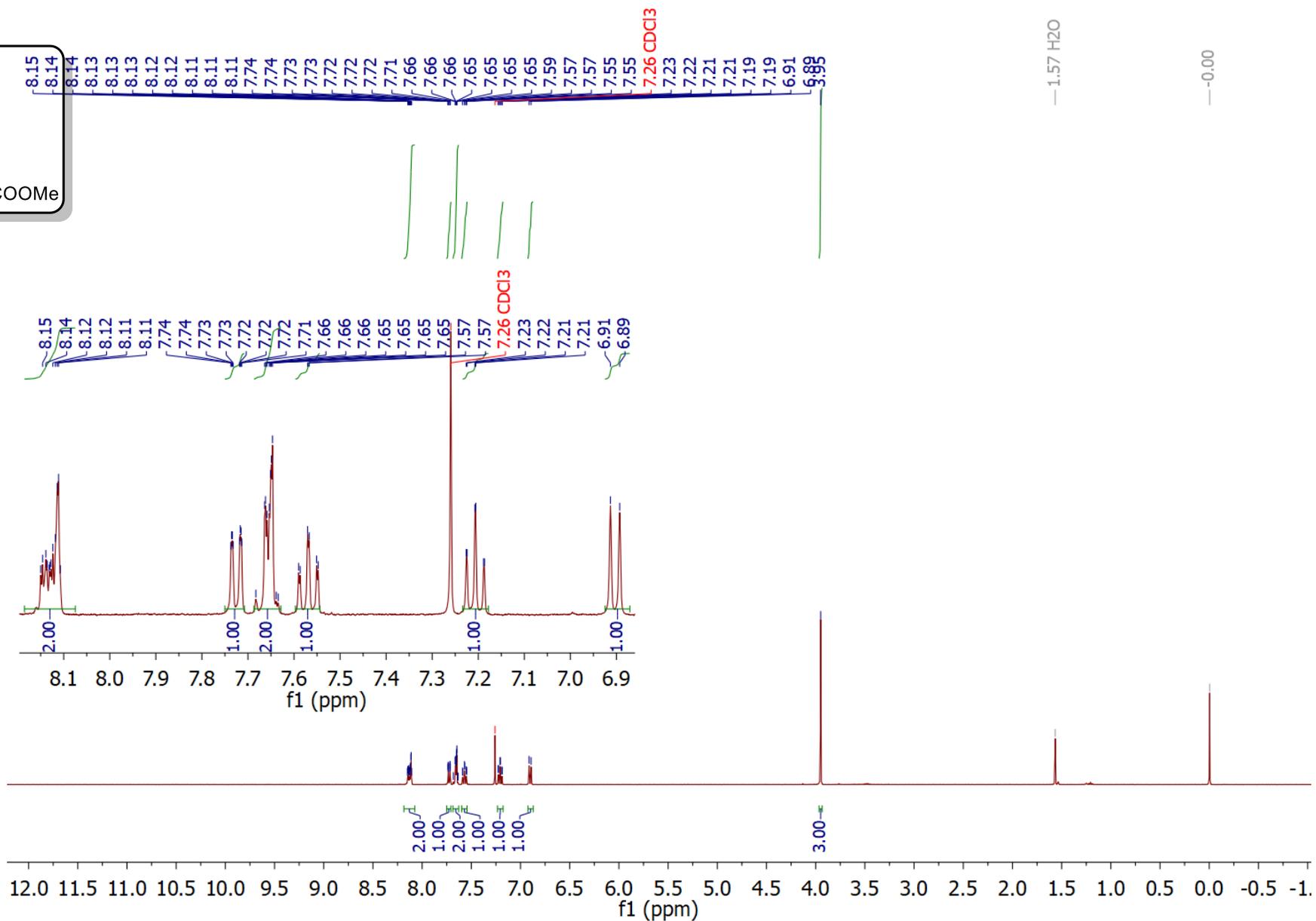
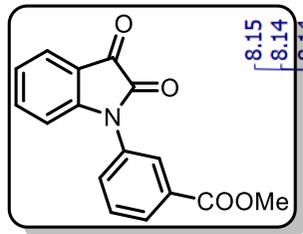
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(2,4,6-triisopropylphenyl)indoline-2,3-dione (**3n**)



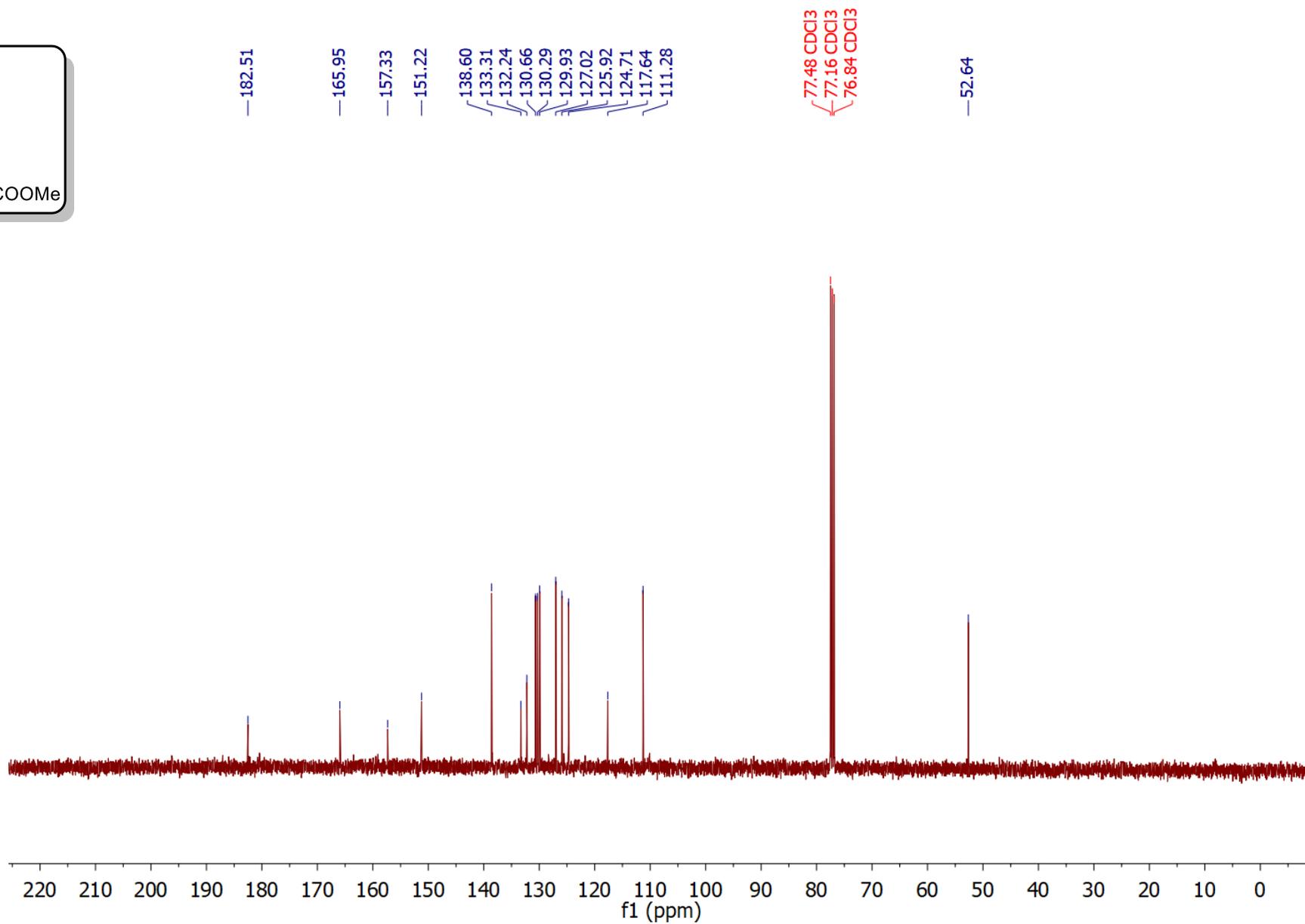
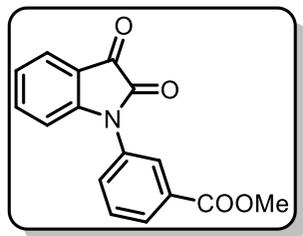
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(2,4,6-triisopropylphenyl)indoline-2,3-dione (**3n**)



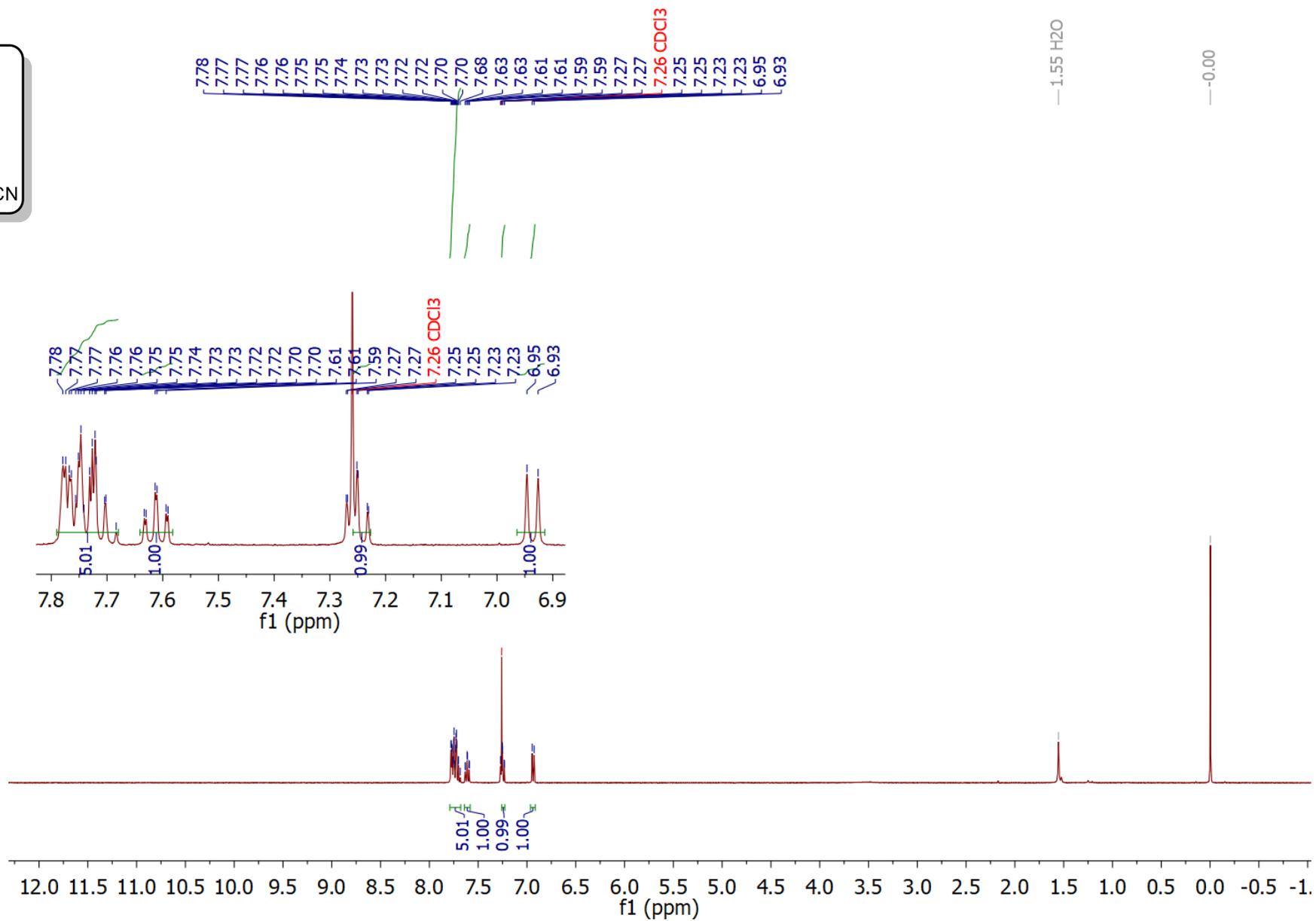
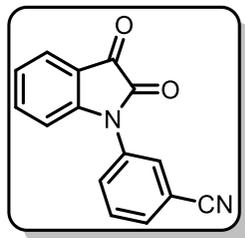
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of methyl 3-(2,3-dioxindolin-1-yl)benzoate (**3o**)



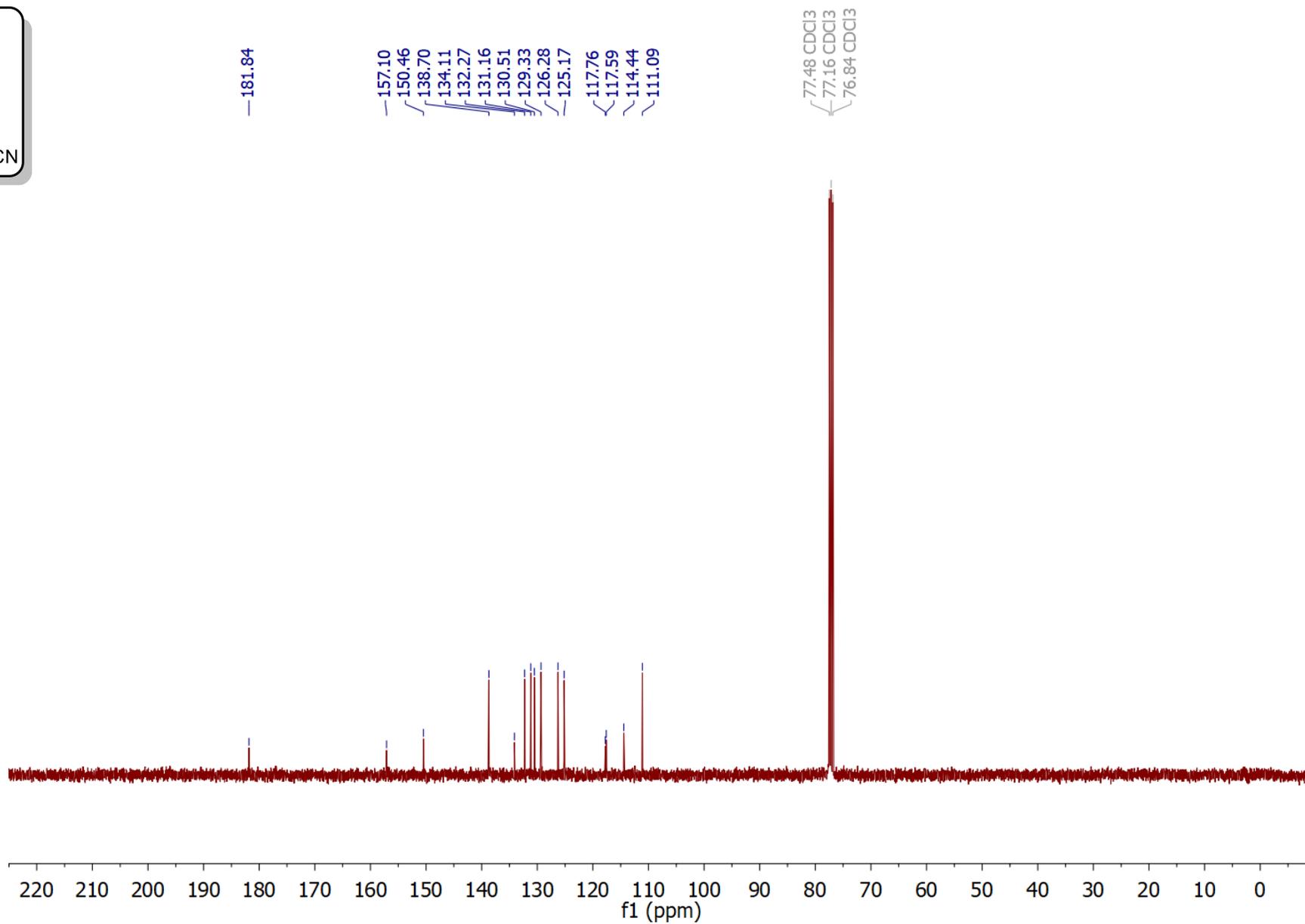
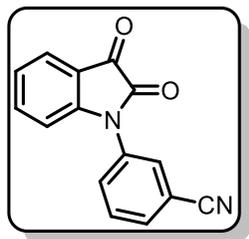
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of methyl 3-(2,3-dioxindolin-1-yl)benzoate (**3o**)



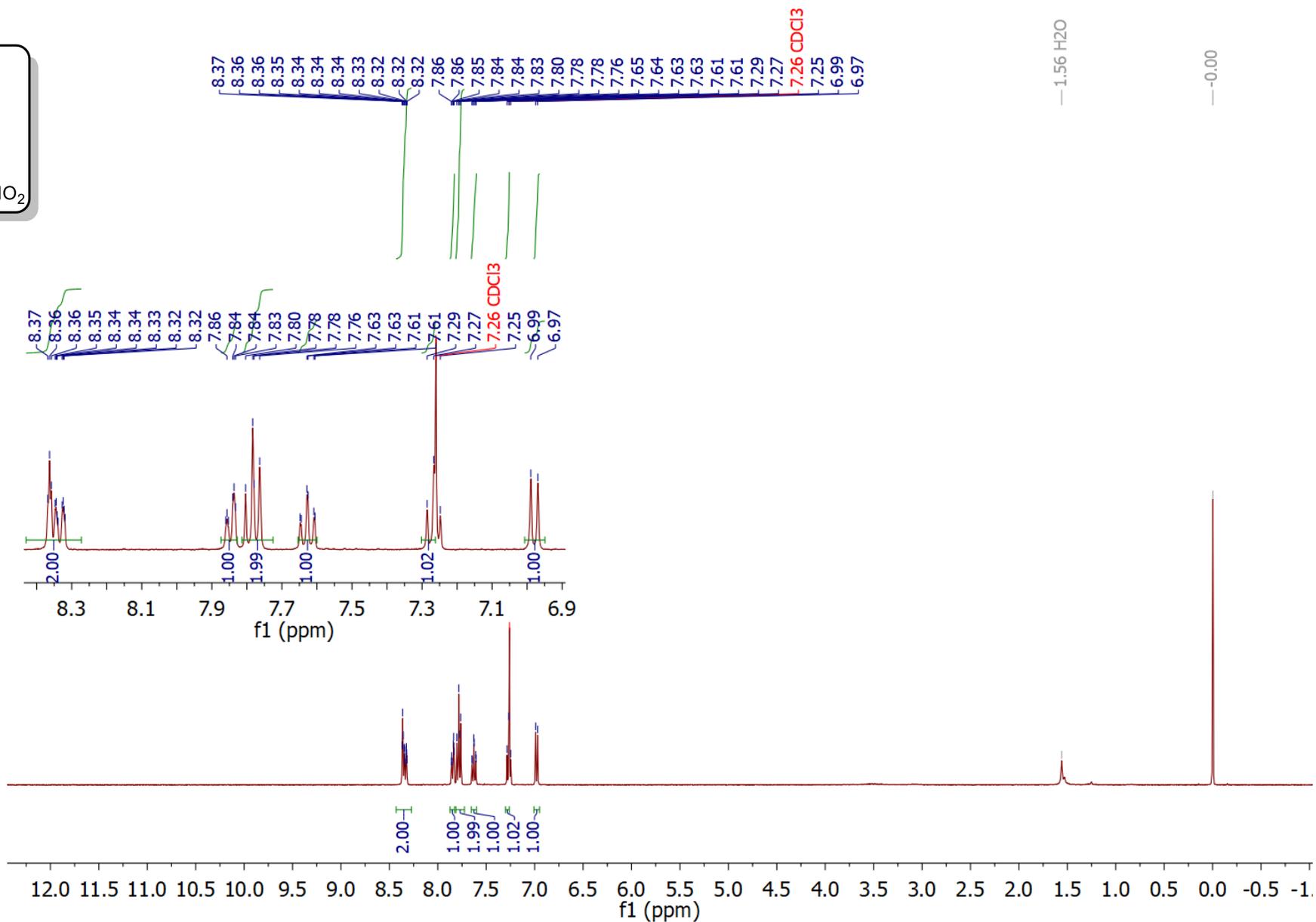
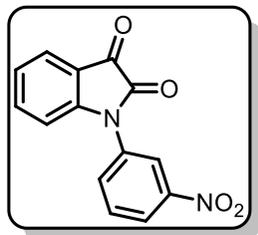
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3-(2,3-dioxindolin-1-yl)benzonitrile (**3p**)



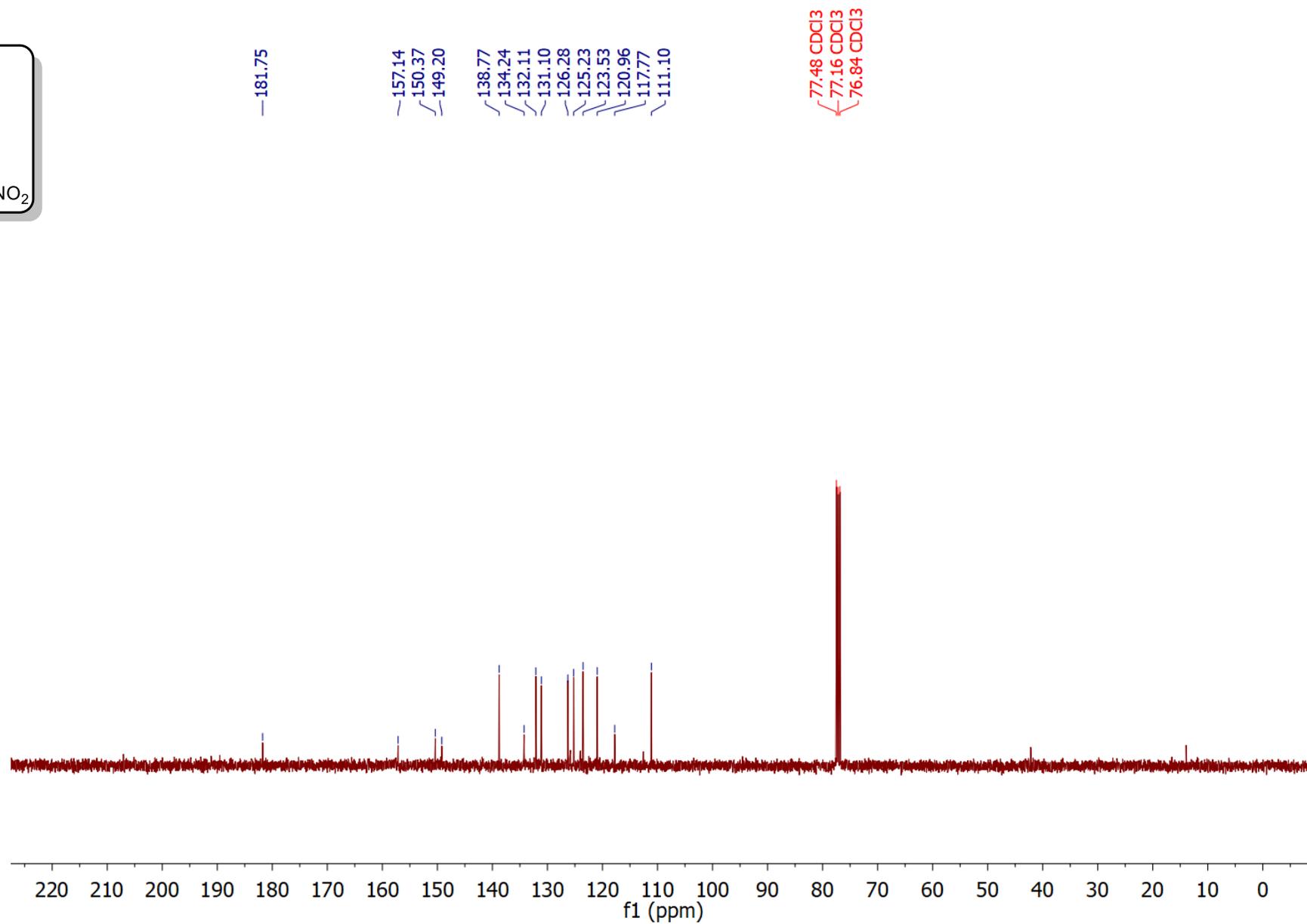
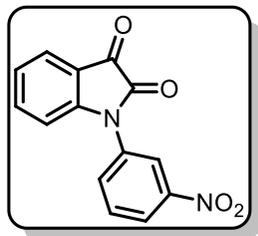
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 3-(2,3-dioxindolin-1-yl)benzonitrile (**3p**)



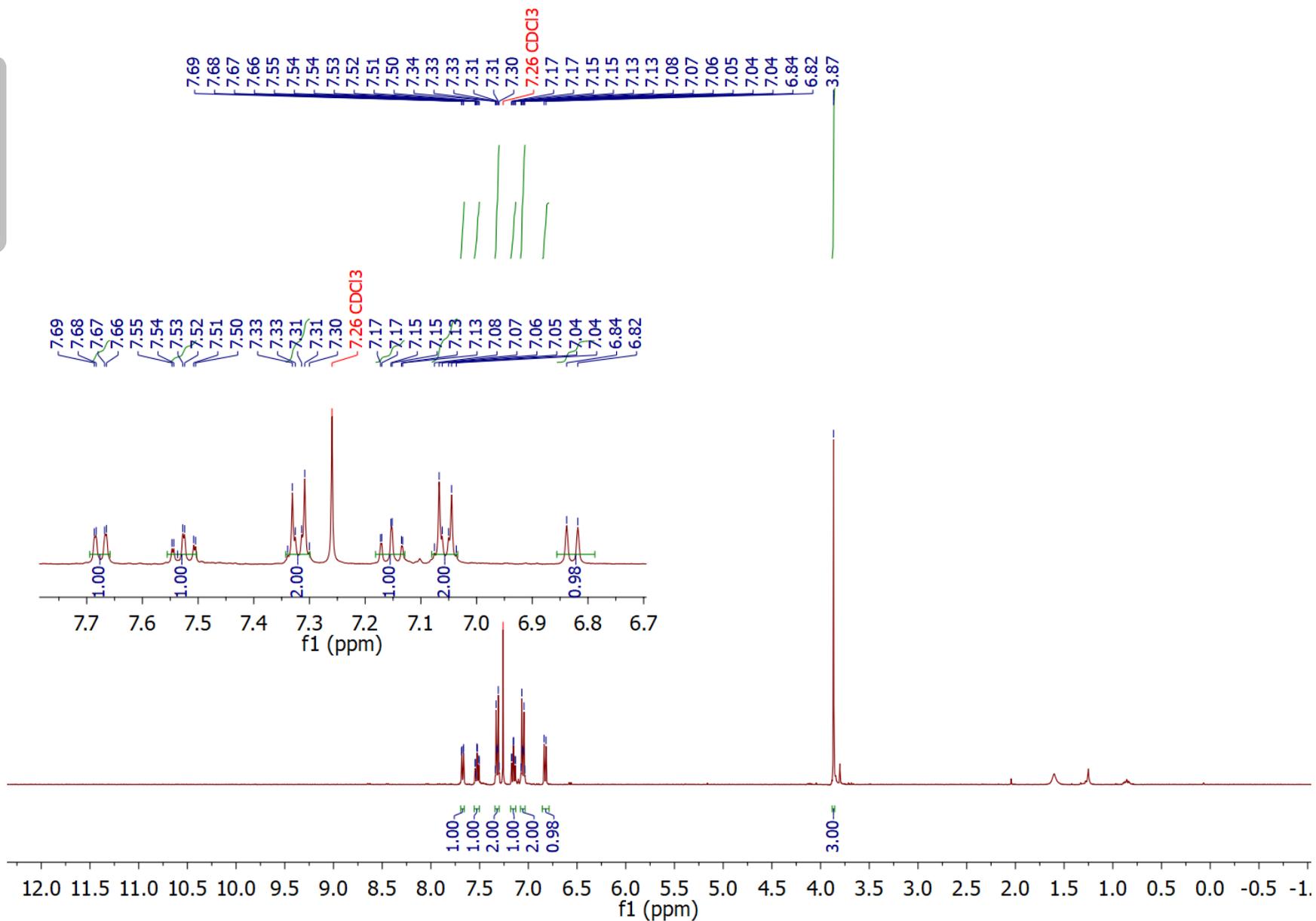
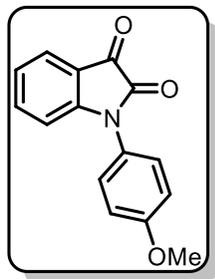
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(3-nitrophenyl)indoline-2,3-dione (**3q**)



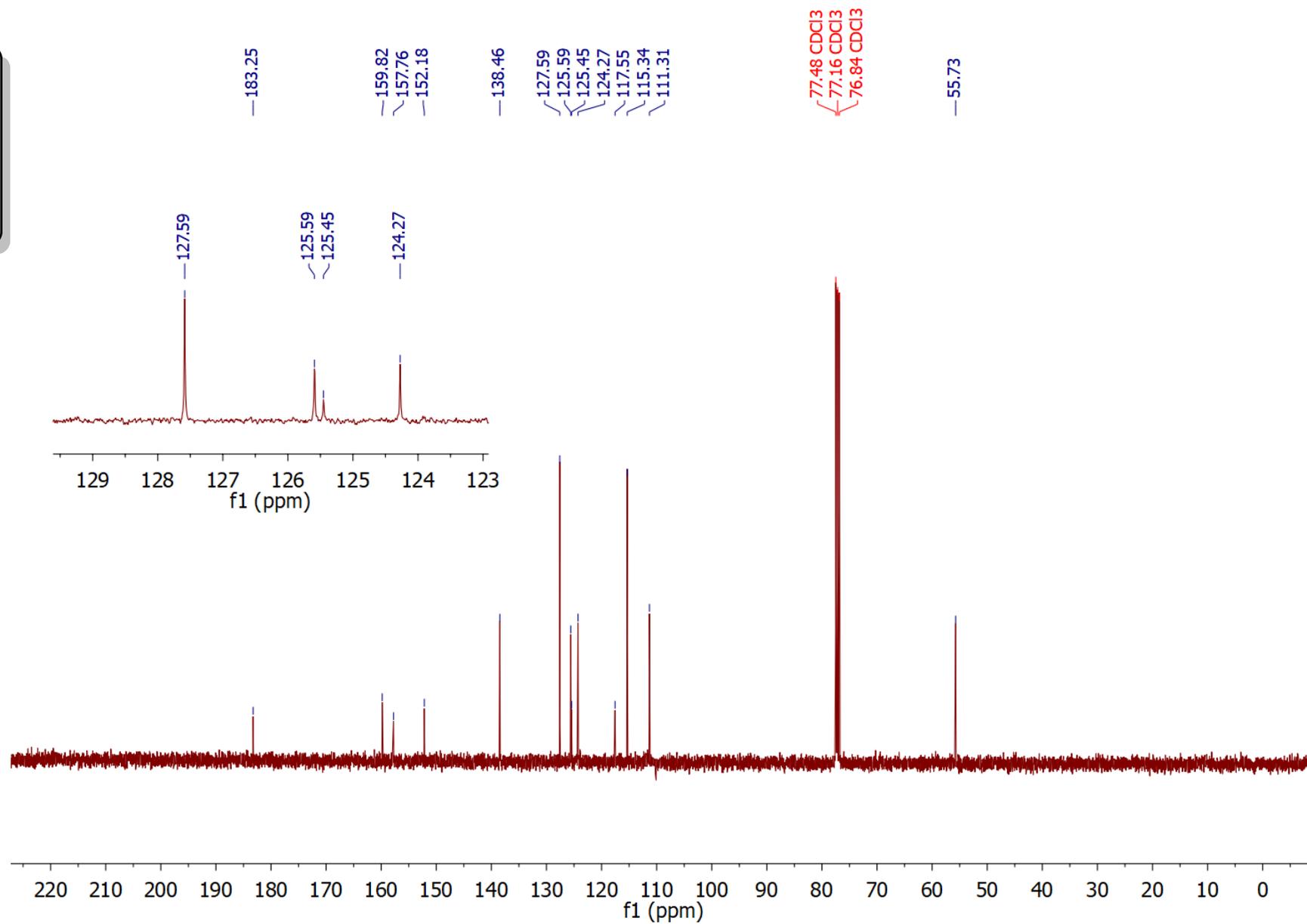
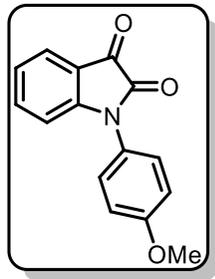
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(3-nitrophenyl)indoline-2,3-dione (**3q**)



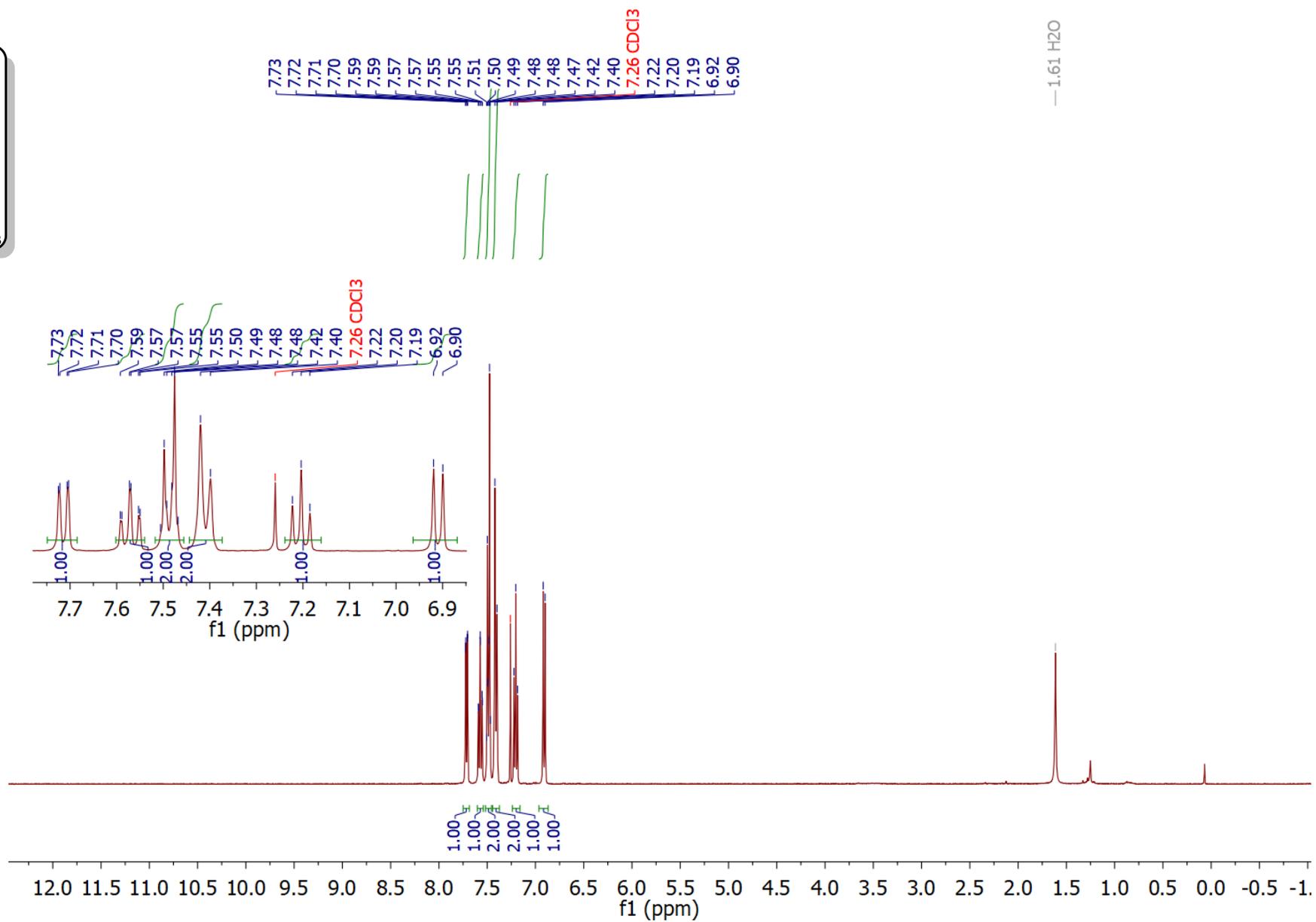
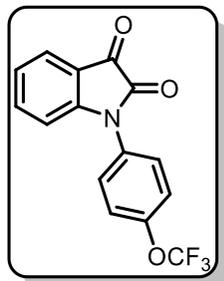
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(4-methoxyphenyl)indoline-2,3-dione (**3r**)



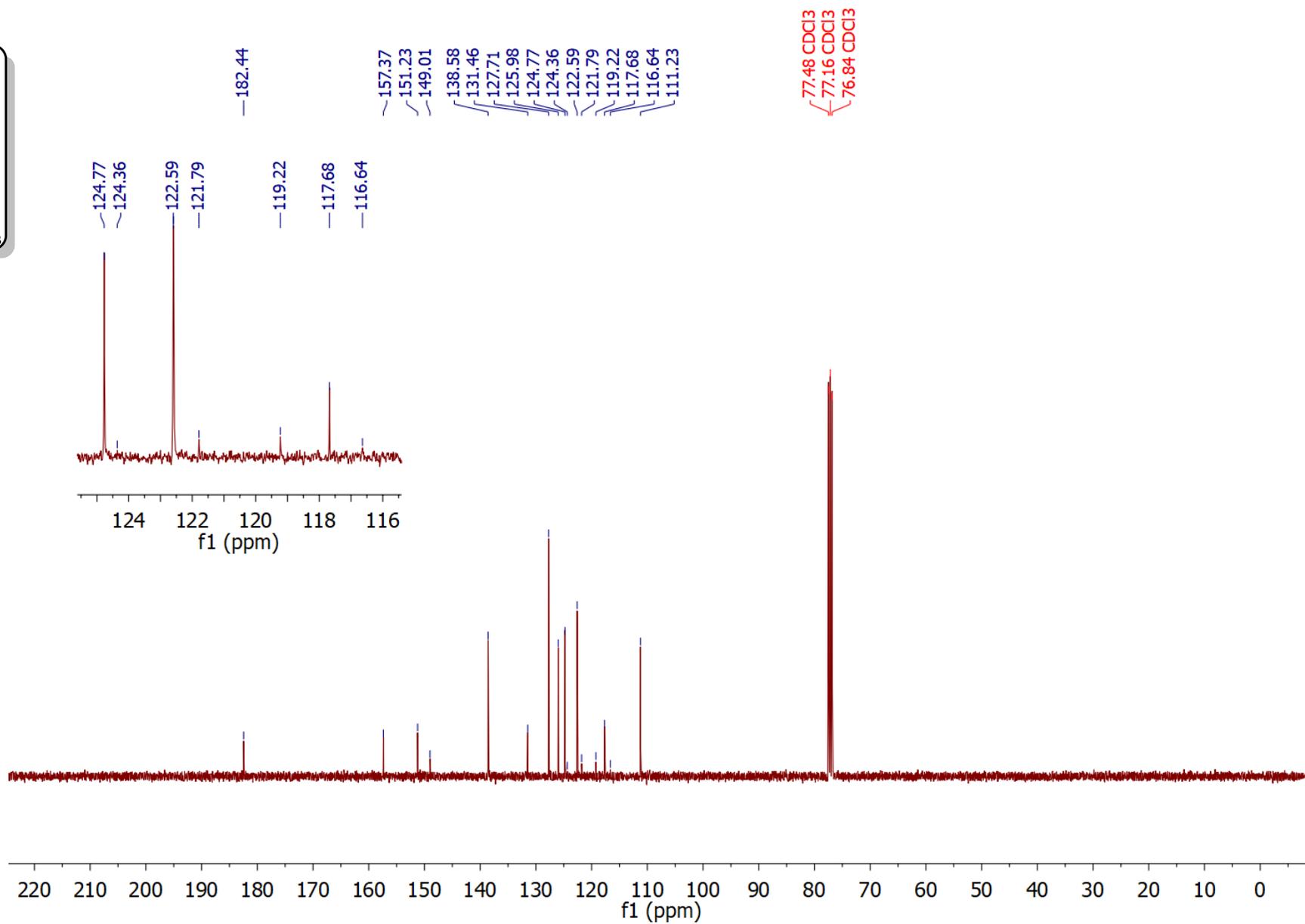
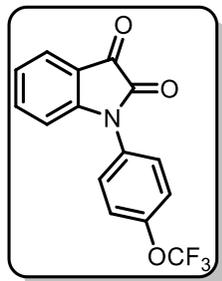
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(4-methoxyphenyl)indoline-2,3-dione (**3r**)



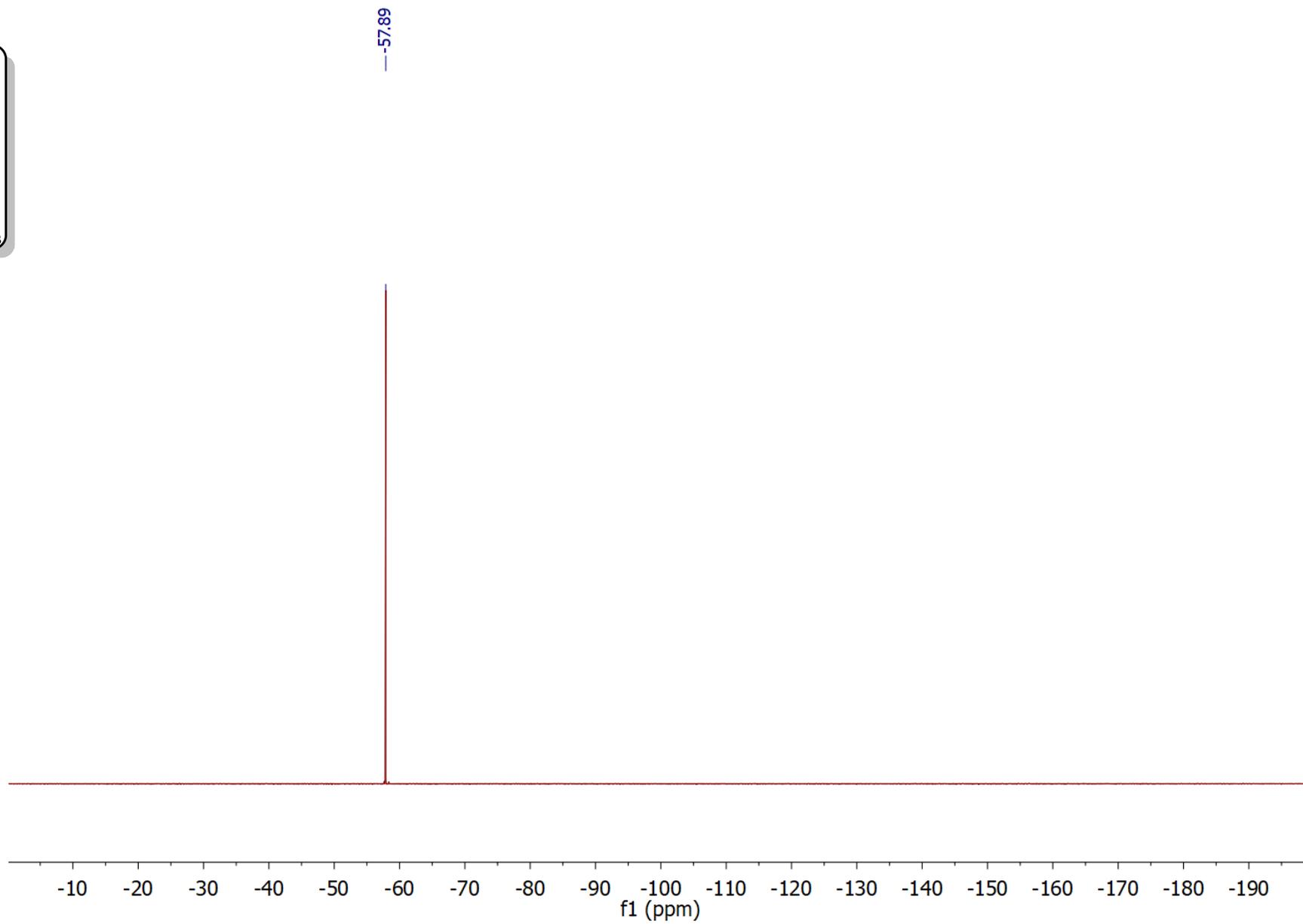
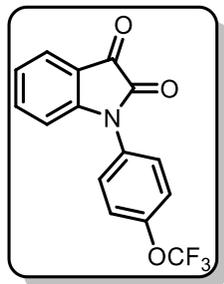
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(4-(trifluoromethoxy)phenyl)indoline-2,3-dione (**3s**)



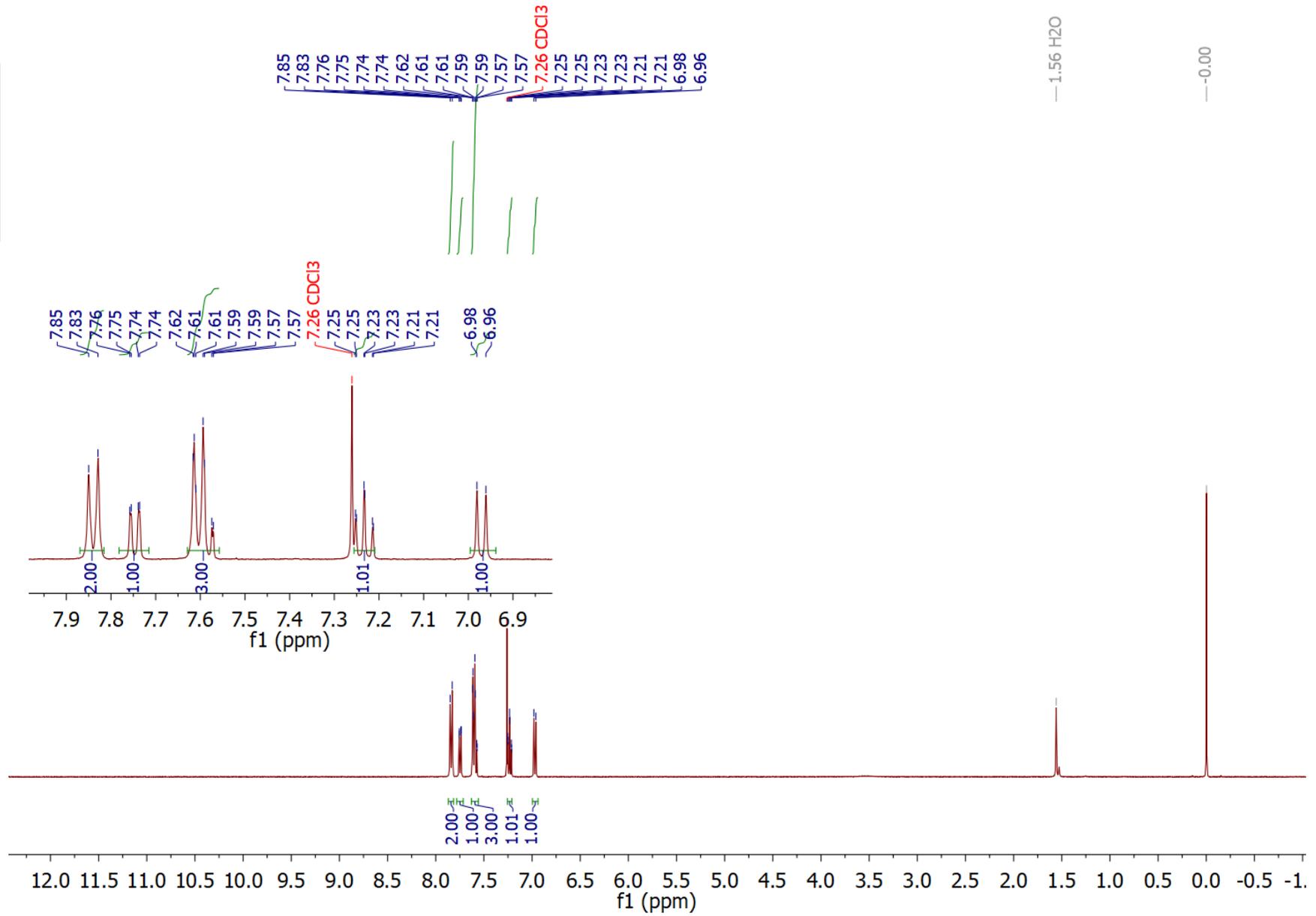
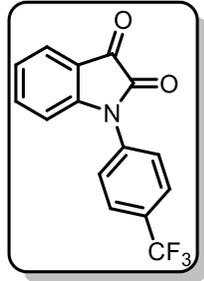
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(4-(trifluoromethoxy)phenyl)indoline-2,3-dione (**3s**)



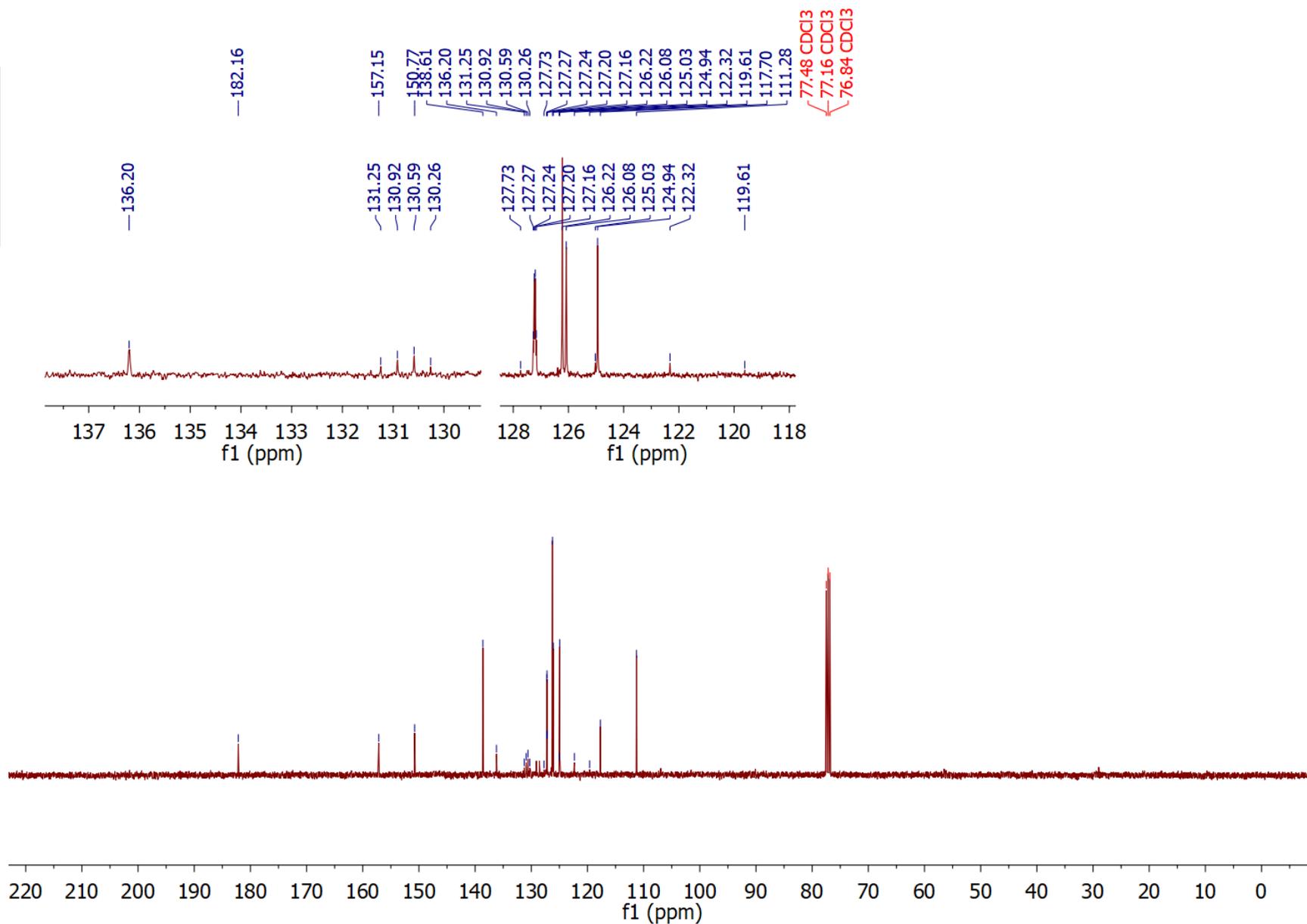
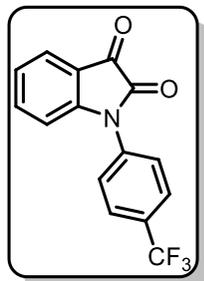
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) of 1-(4-(trifluoromethoxy)phenyl)indoline-2,3-dione (**3s**)



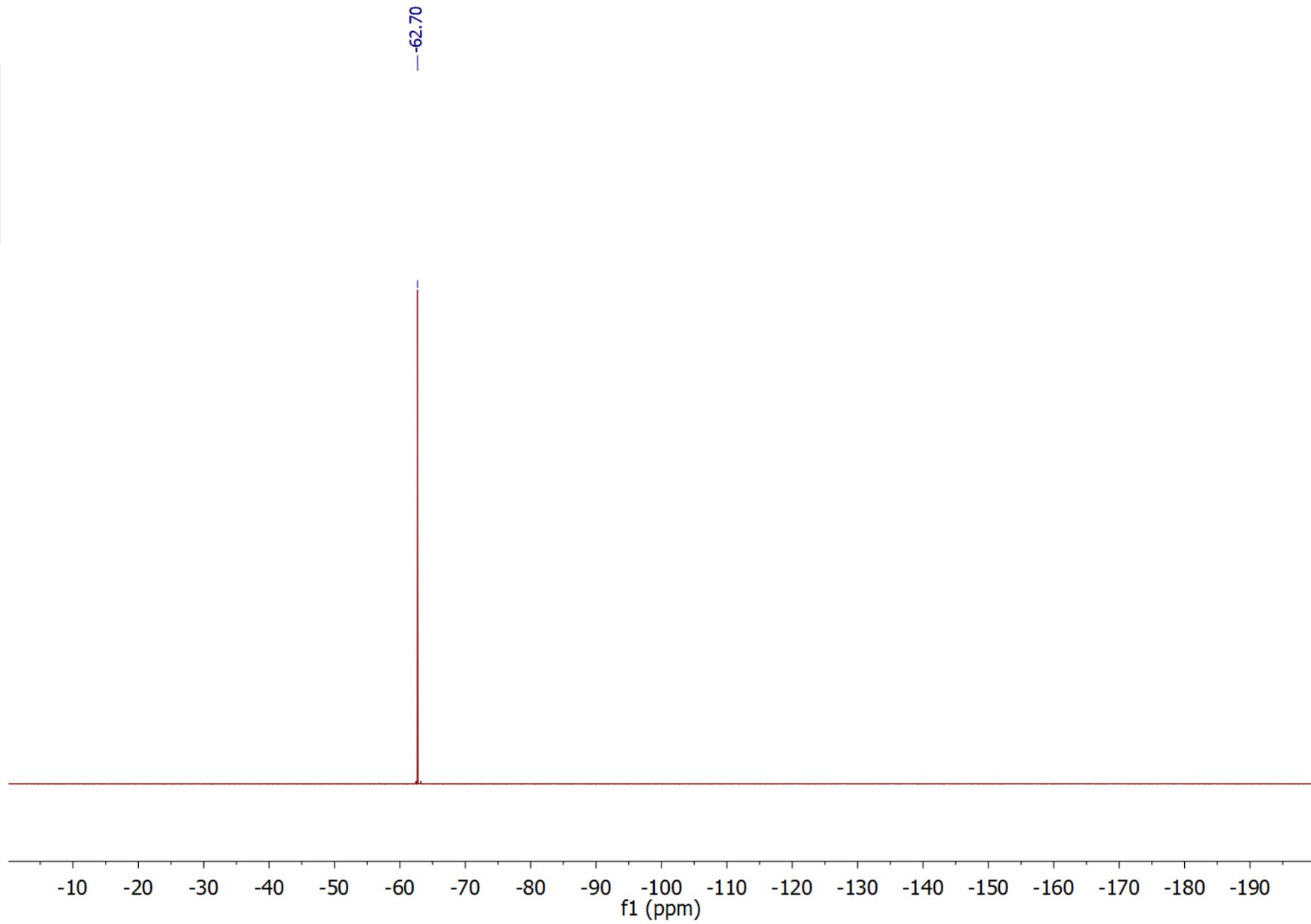
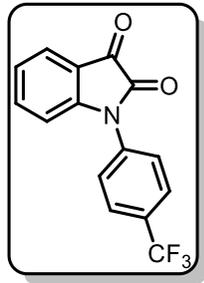
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(4-(trifluoromethyl)phenyl)indoline-2,3-dione (**3t**)



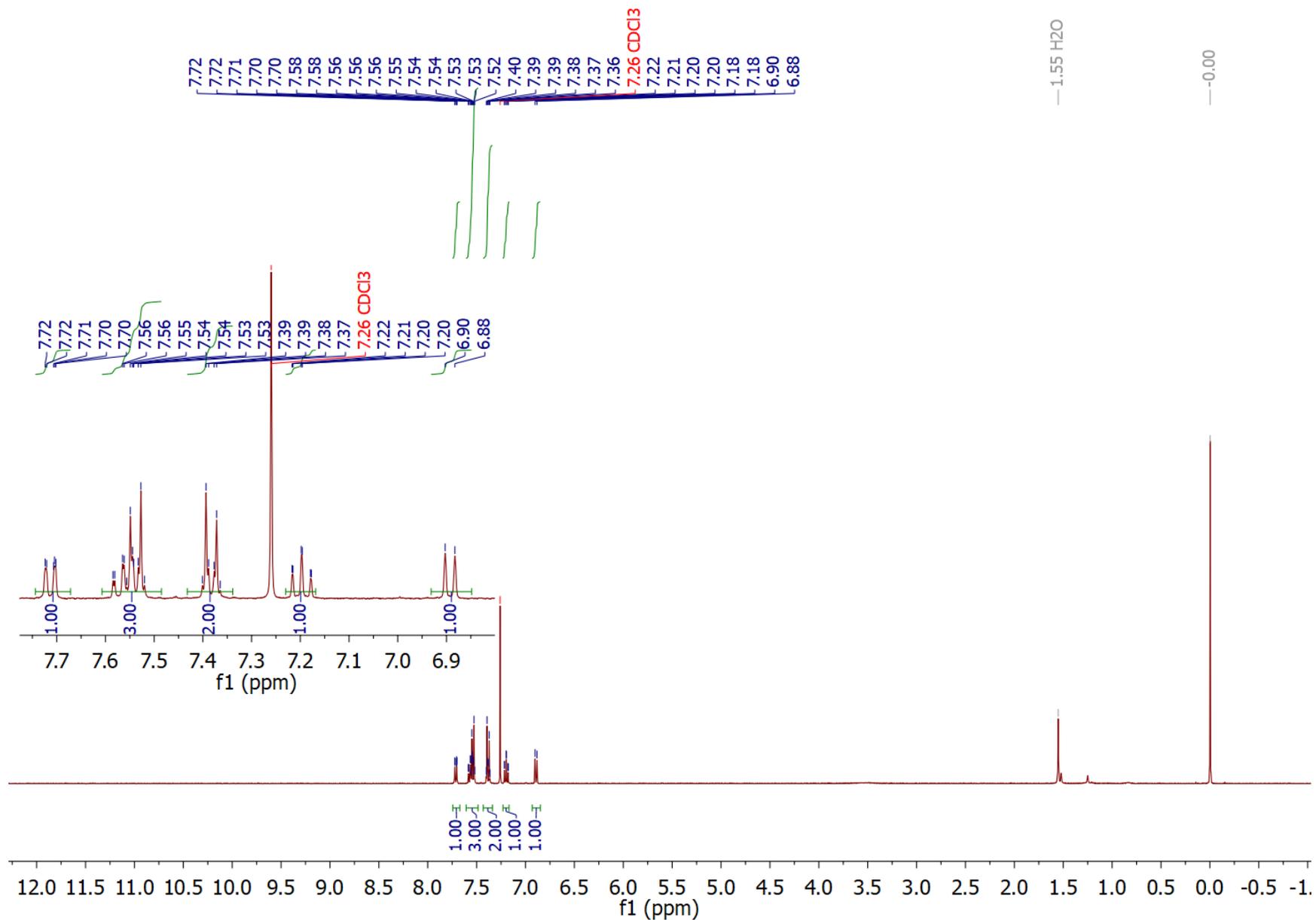
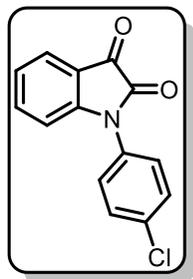
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(4-(trifluoromethyl)phenyl)indoline-2,3-dione (**3t**)



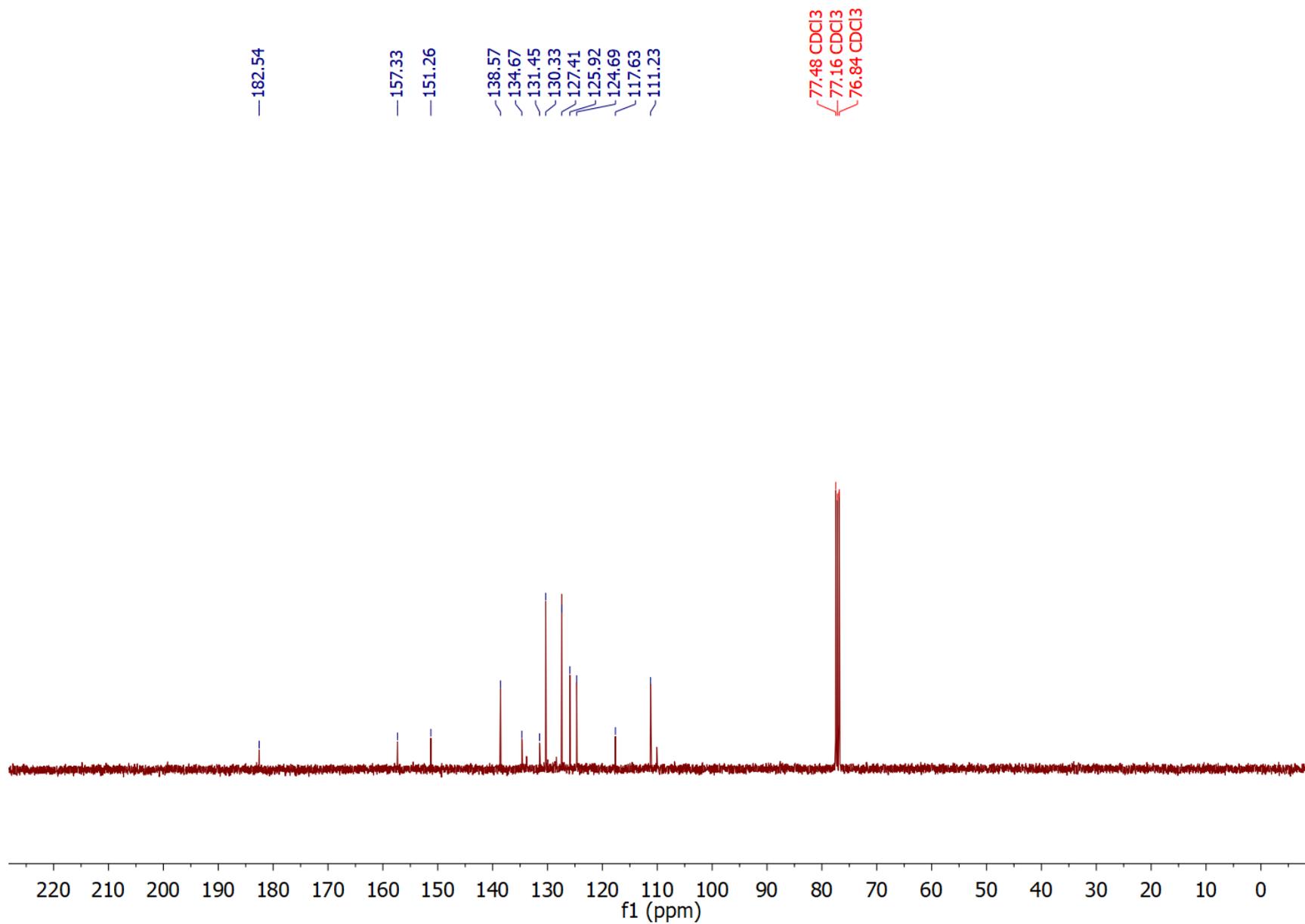
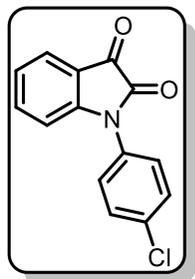
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) of 1-(4-(trifluoromethyl)phenyl)indoline-2,3-dione (**3t**)



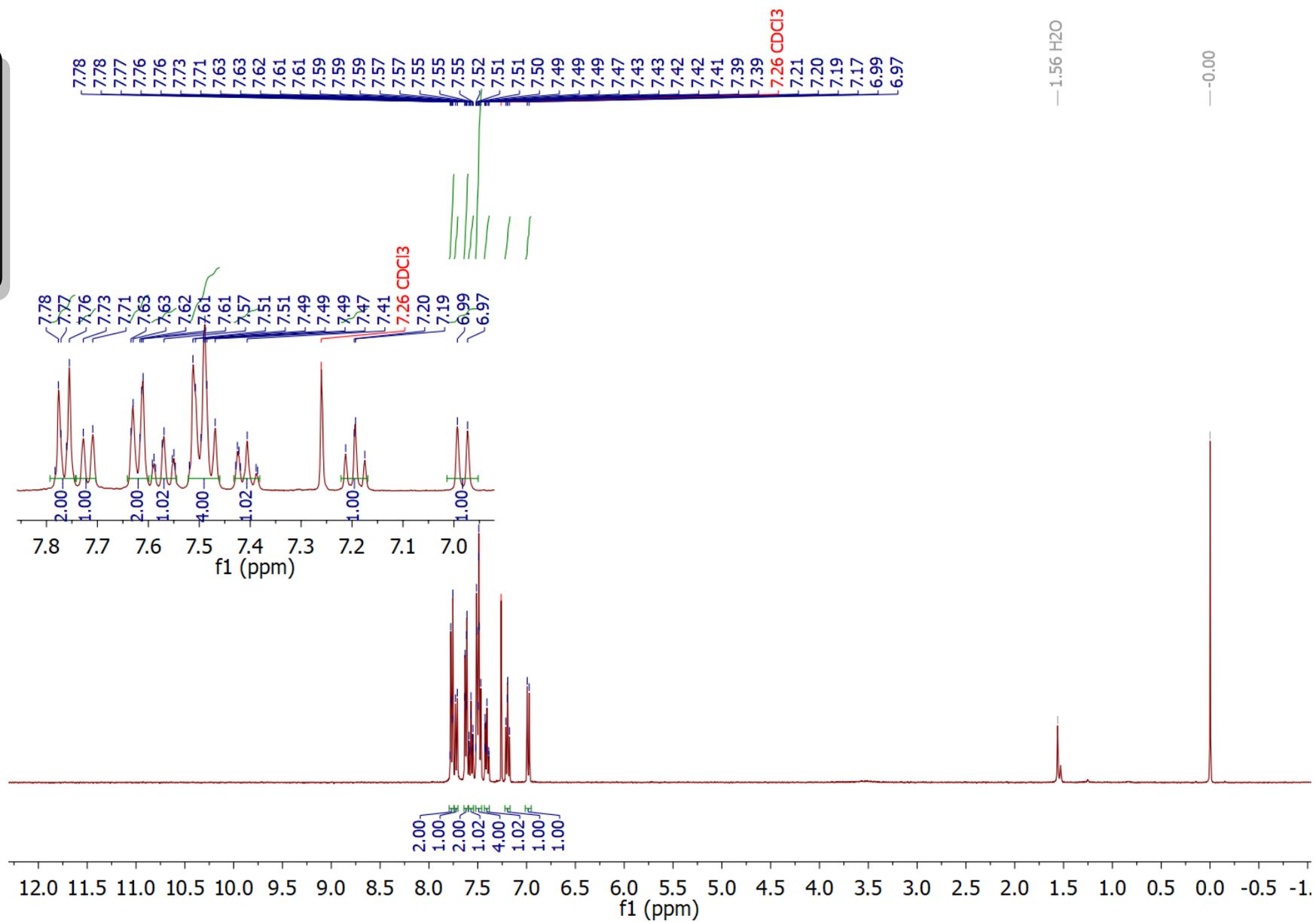
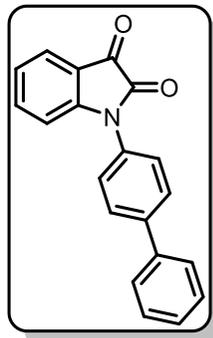
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-(4-chlorophenyl)indoline-2,3-dione (**3u**)



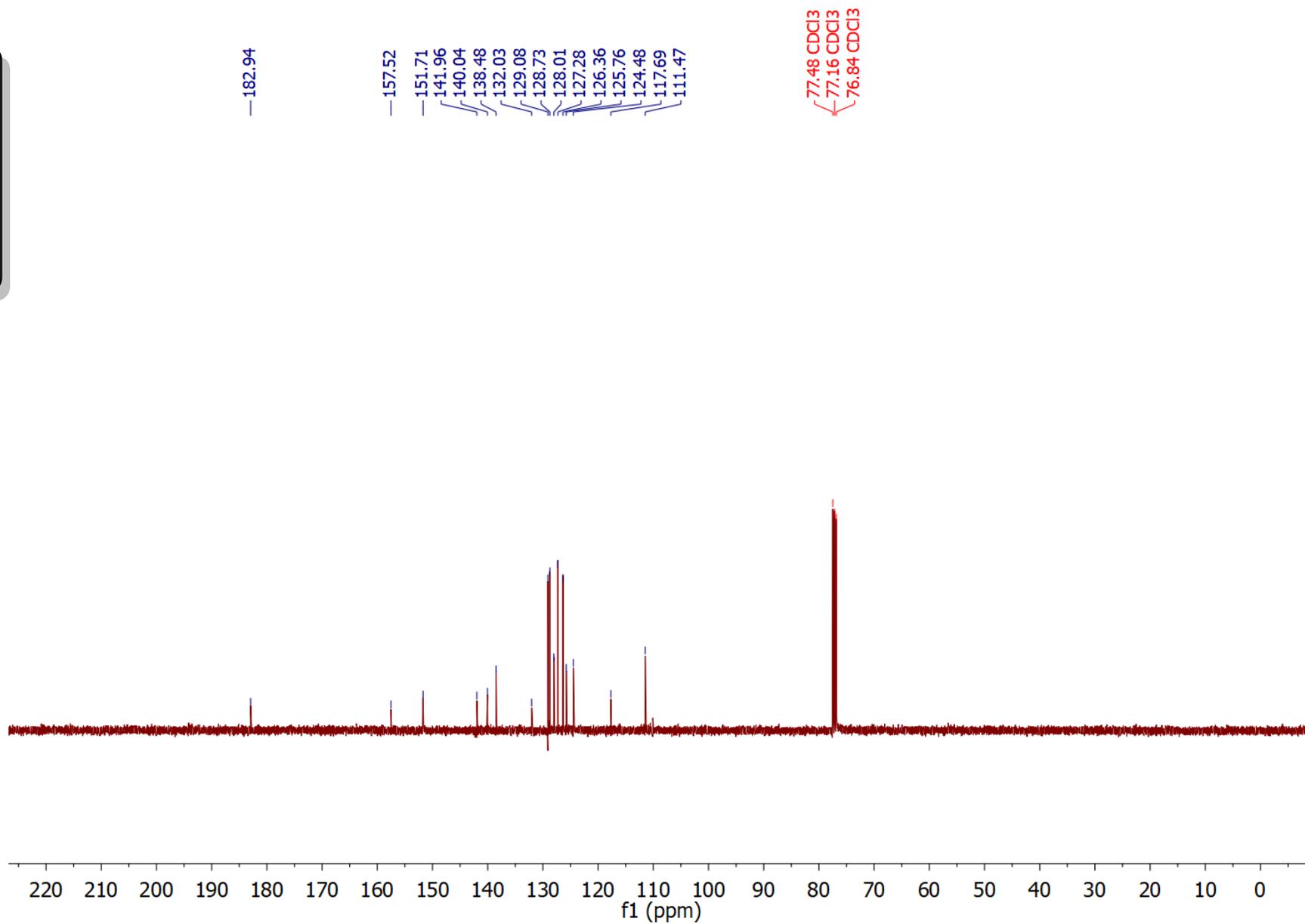
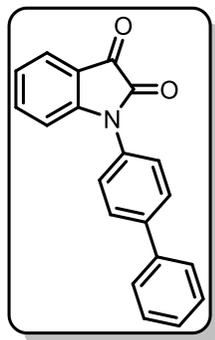
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-(4-chlorophenyl)indoline-2,3-dione (**3u**)



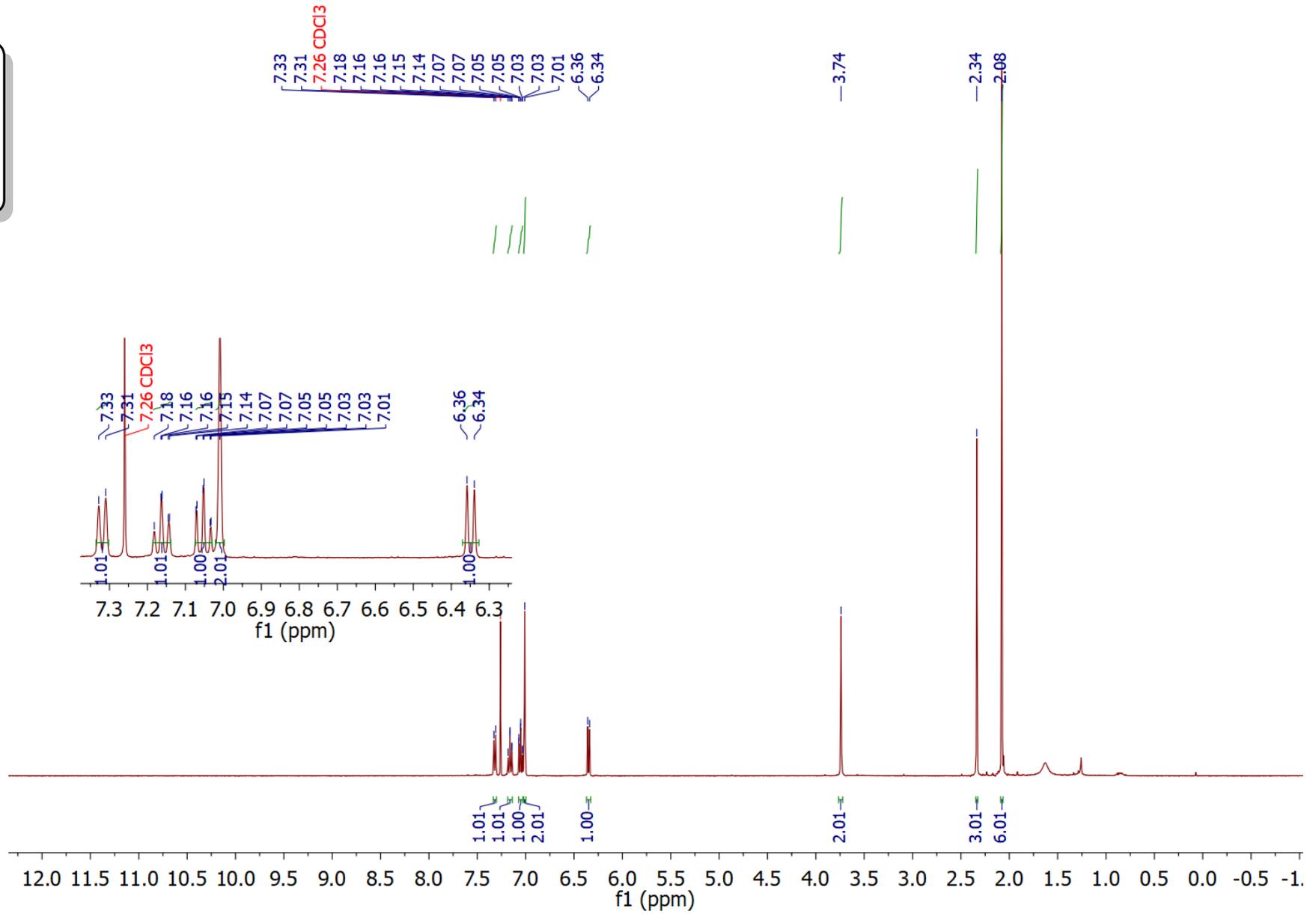
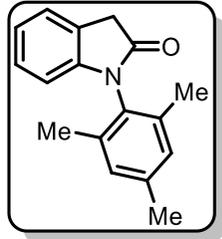
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-([1,1'-biphenyl]-4-yl)indoline-2,3-dione (**3v**)



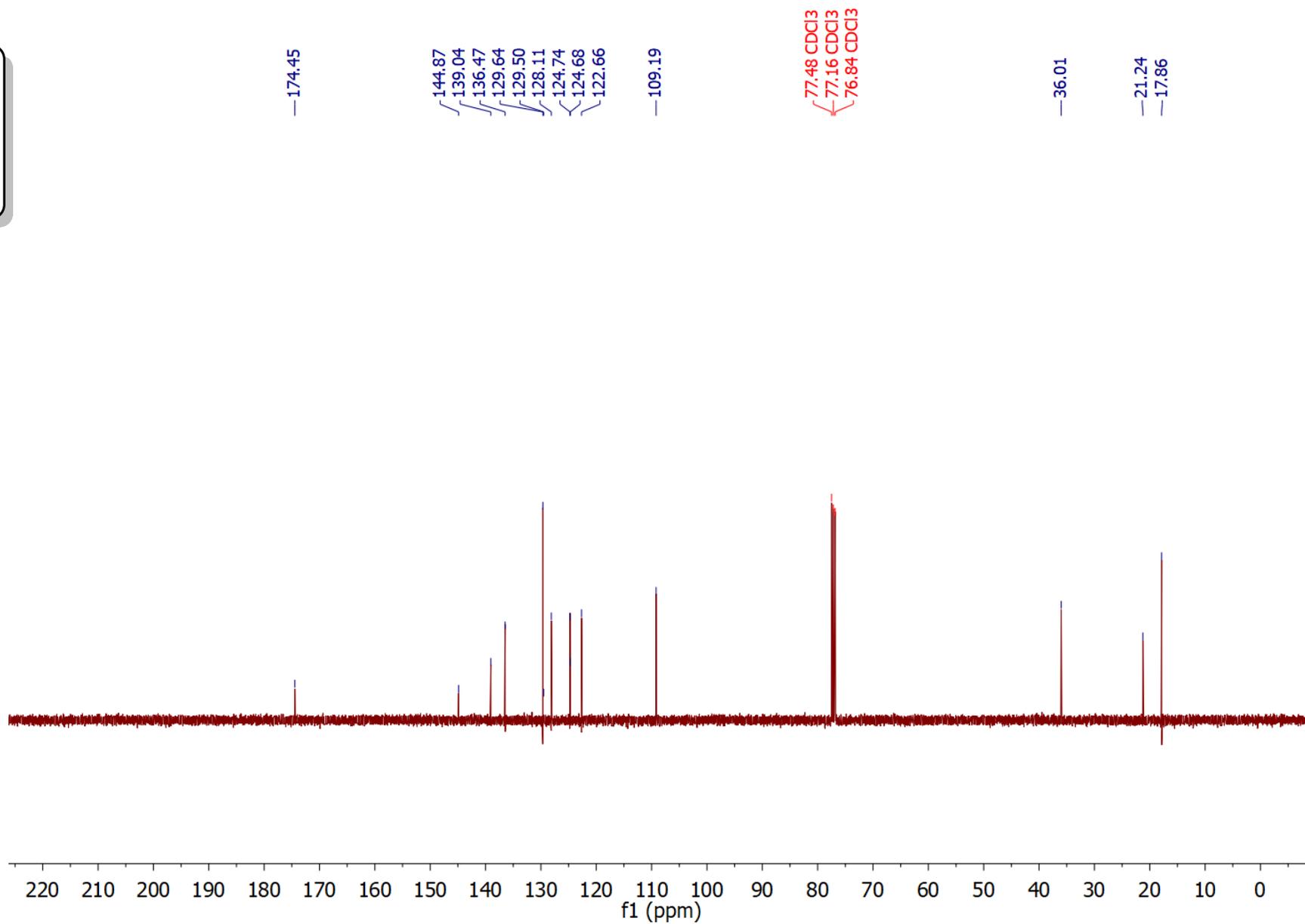
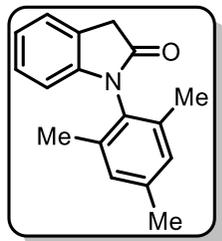
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-([1,1'-biphenyl]-4-yl)indoline-2,3-dione (**3v**)



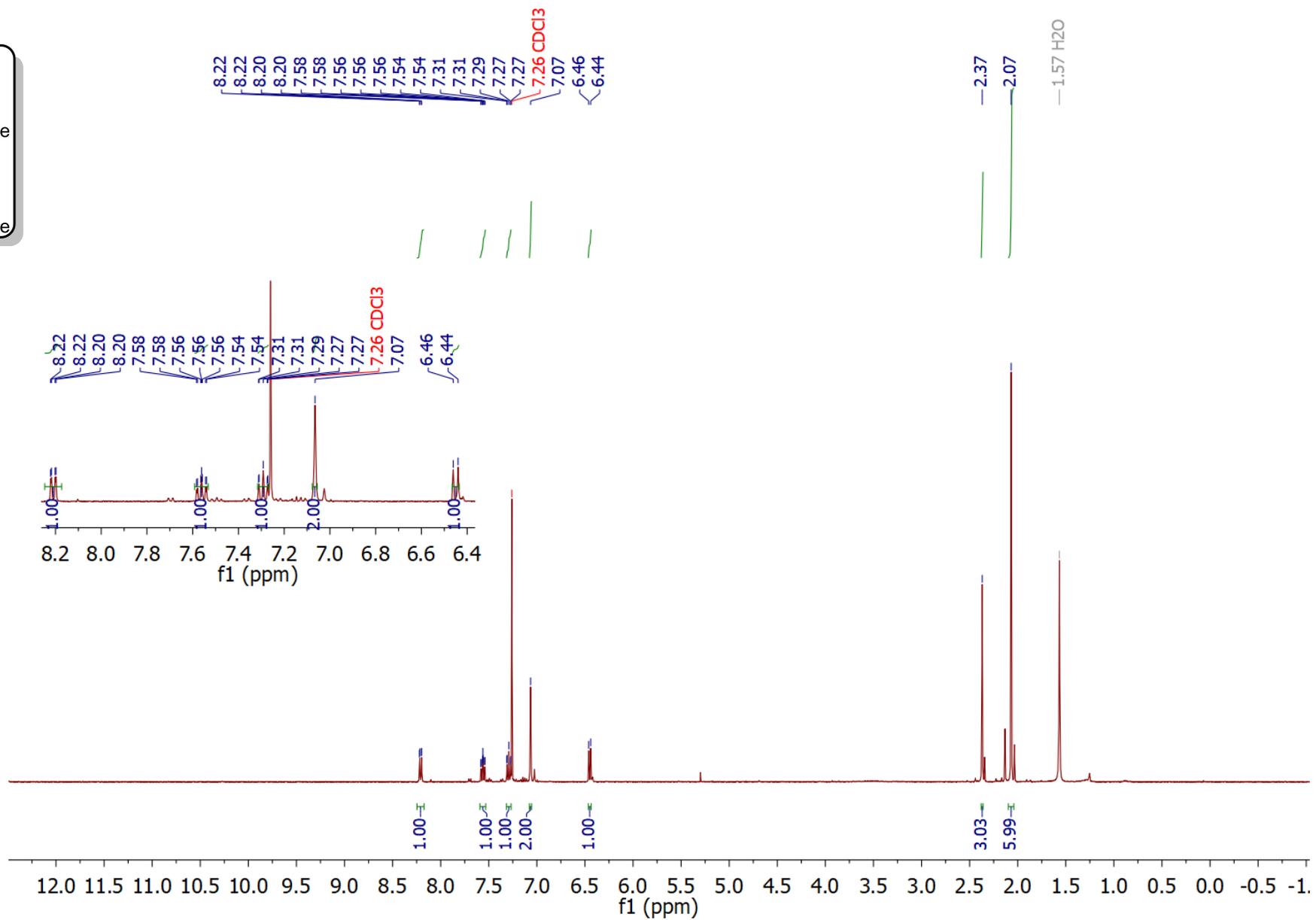
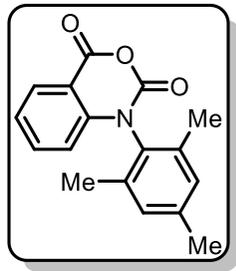
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-mesitylindolin-2-one (**4**)



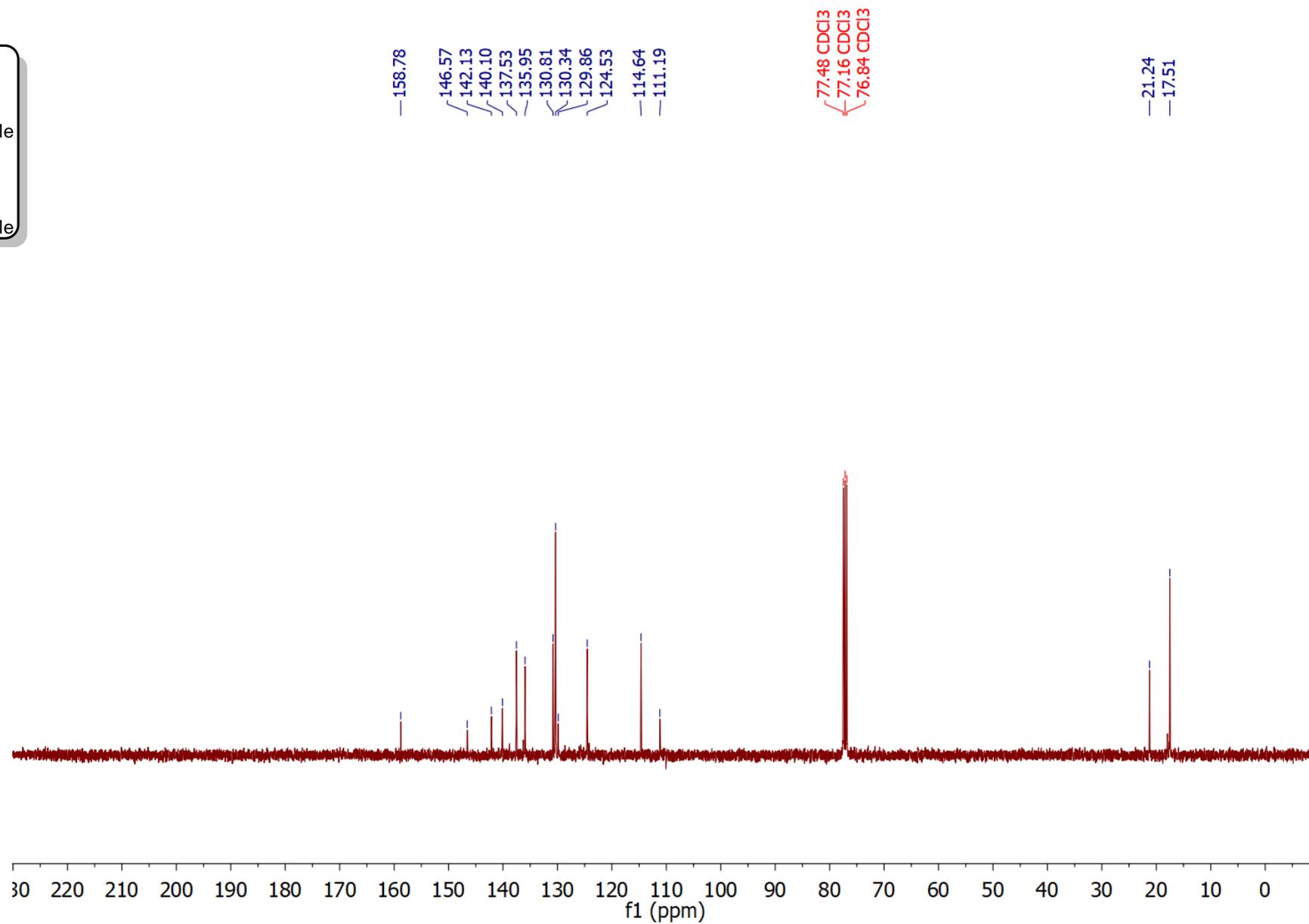
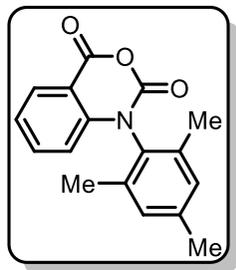
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-mesitylindolin-2-one (**4**)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-mesityl-2H-benzo[d][1,3]oxazine-2,4(1H)-dione (**5**)

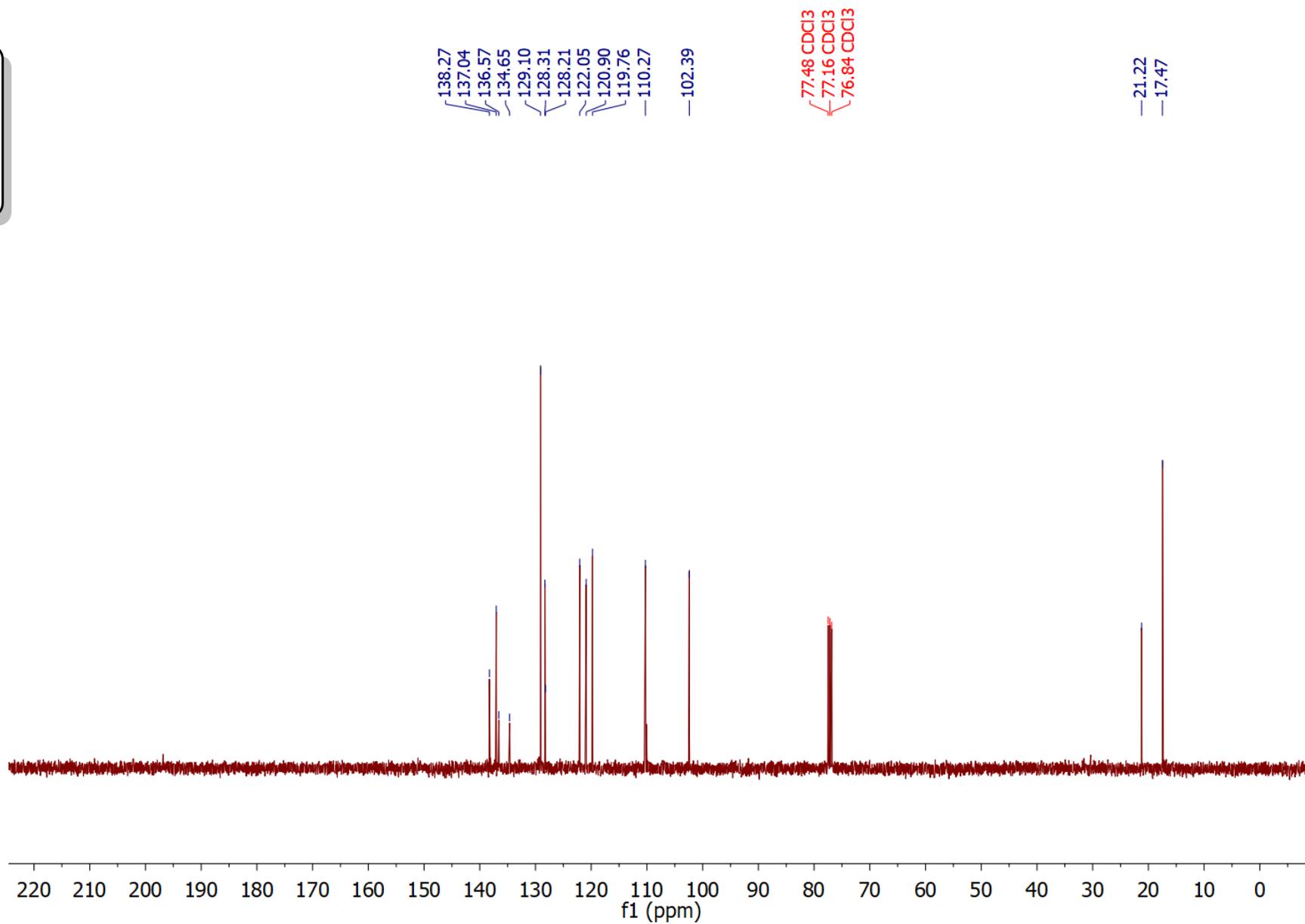
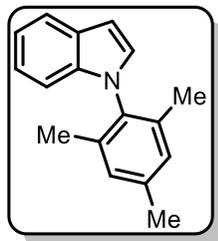


$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-mesityl-2*H*-benzo[*d*][1,3]oxazine-2,4(1*H*)-dione (**5**)

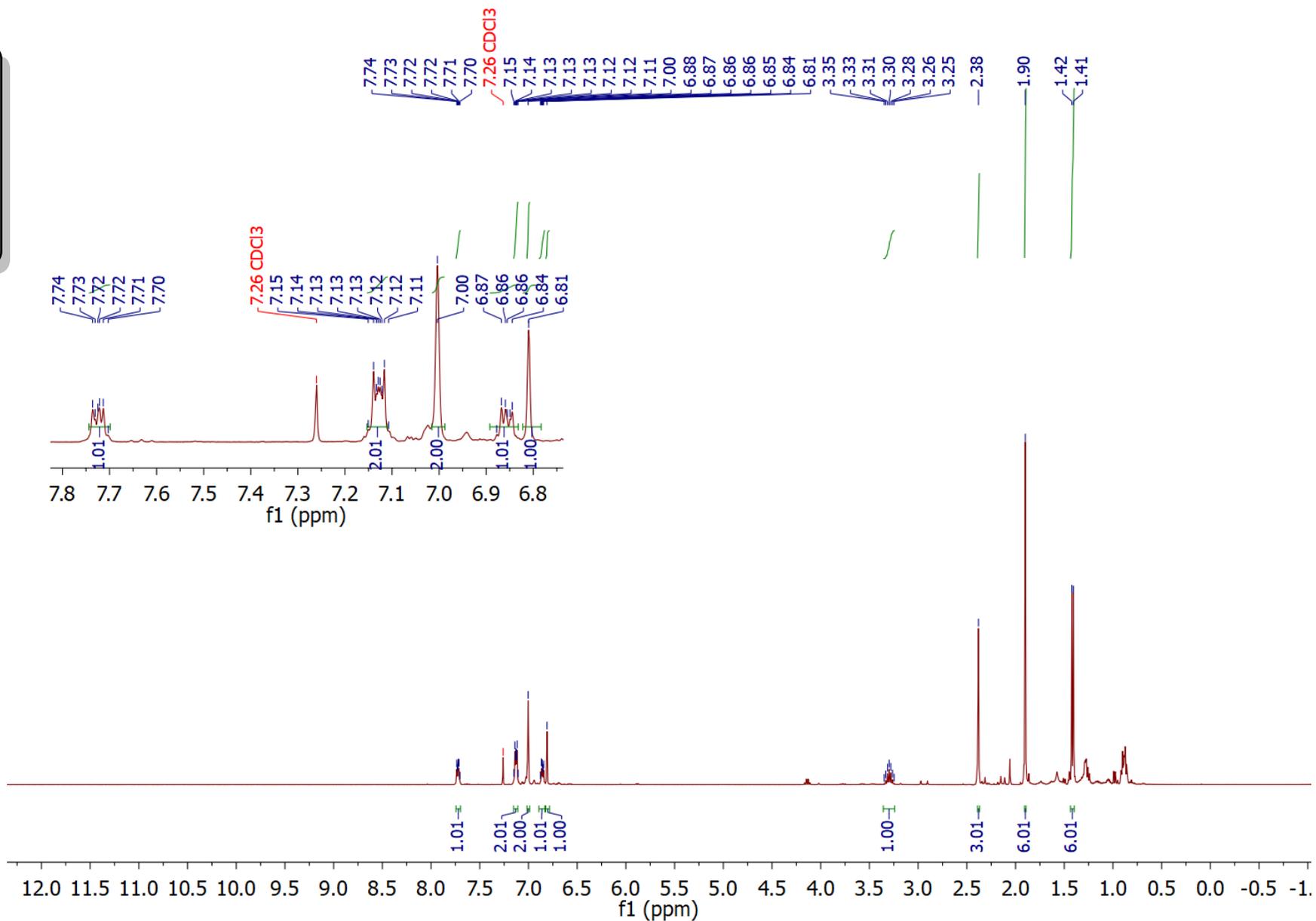
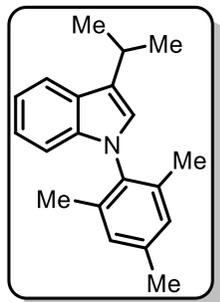




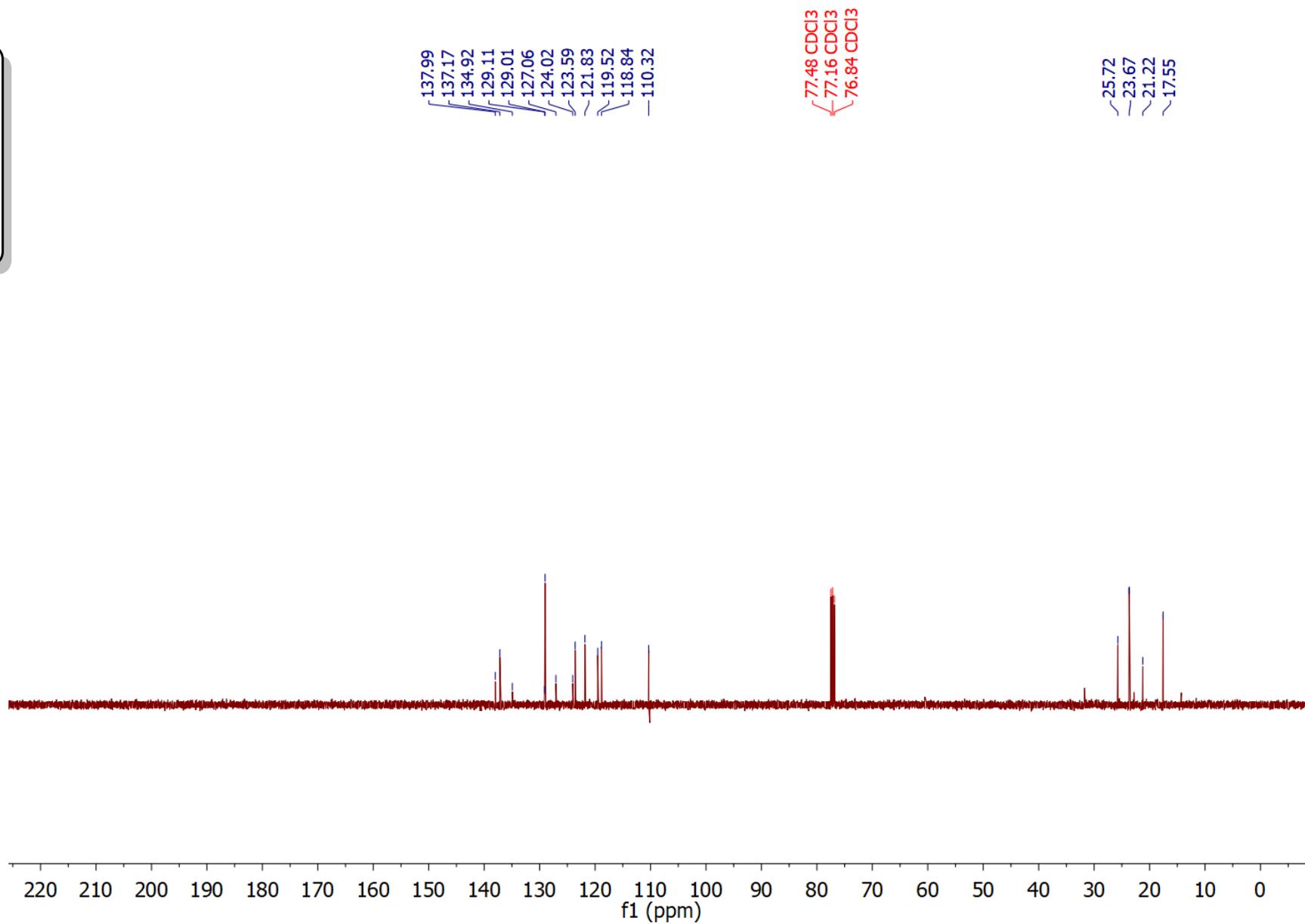
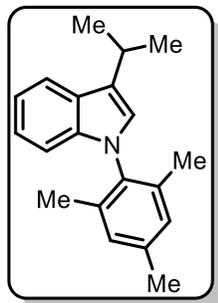
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-mesityl-1H-indole (**6**)



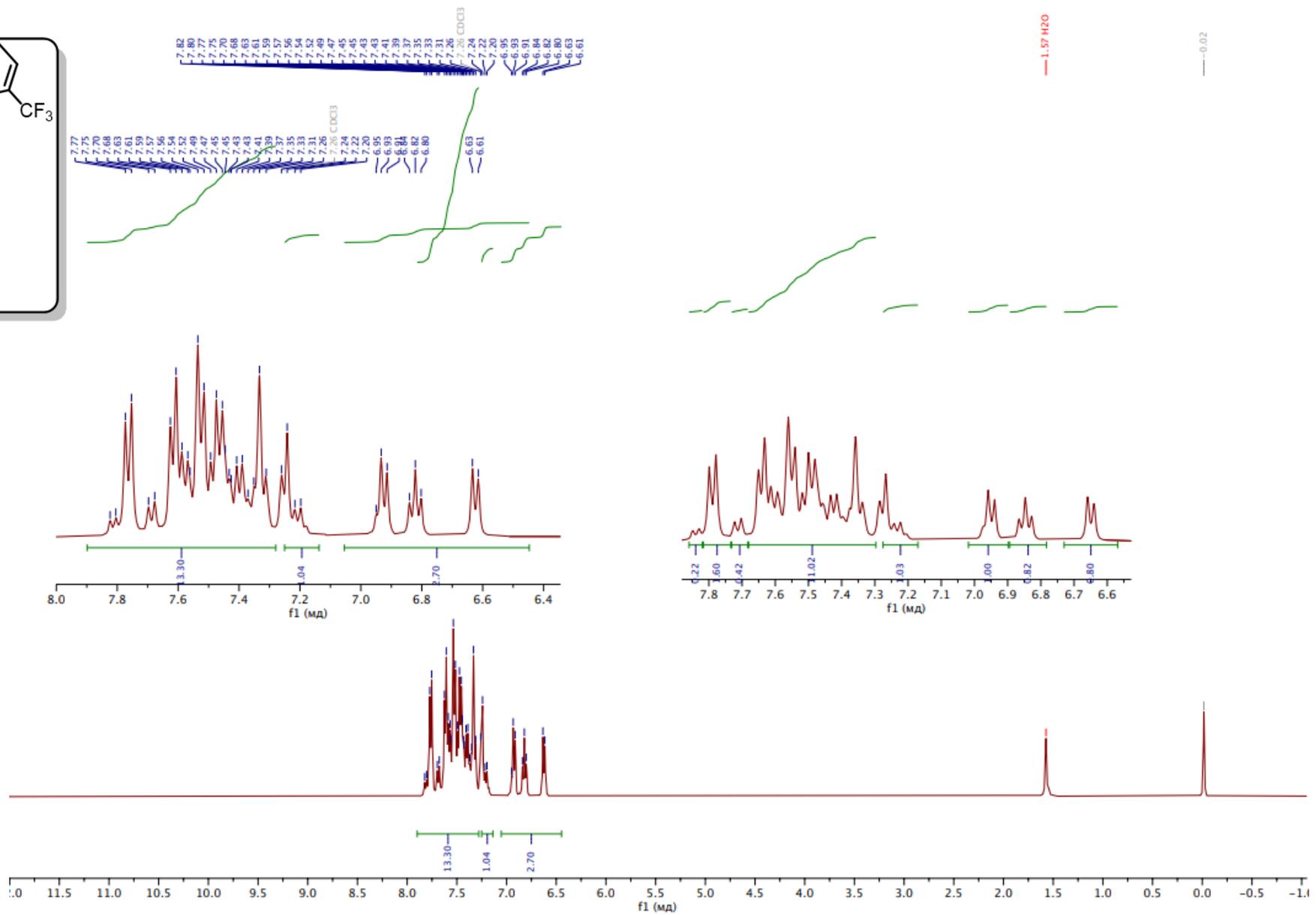
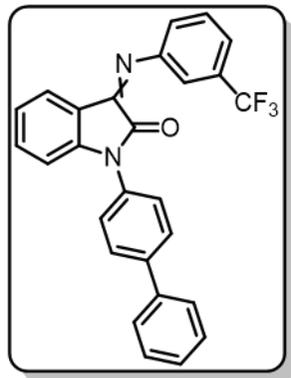
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3-isopropyl-1-mesityl-1*H*-indole (**7**)



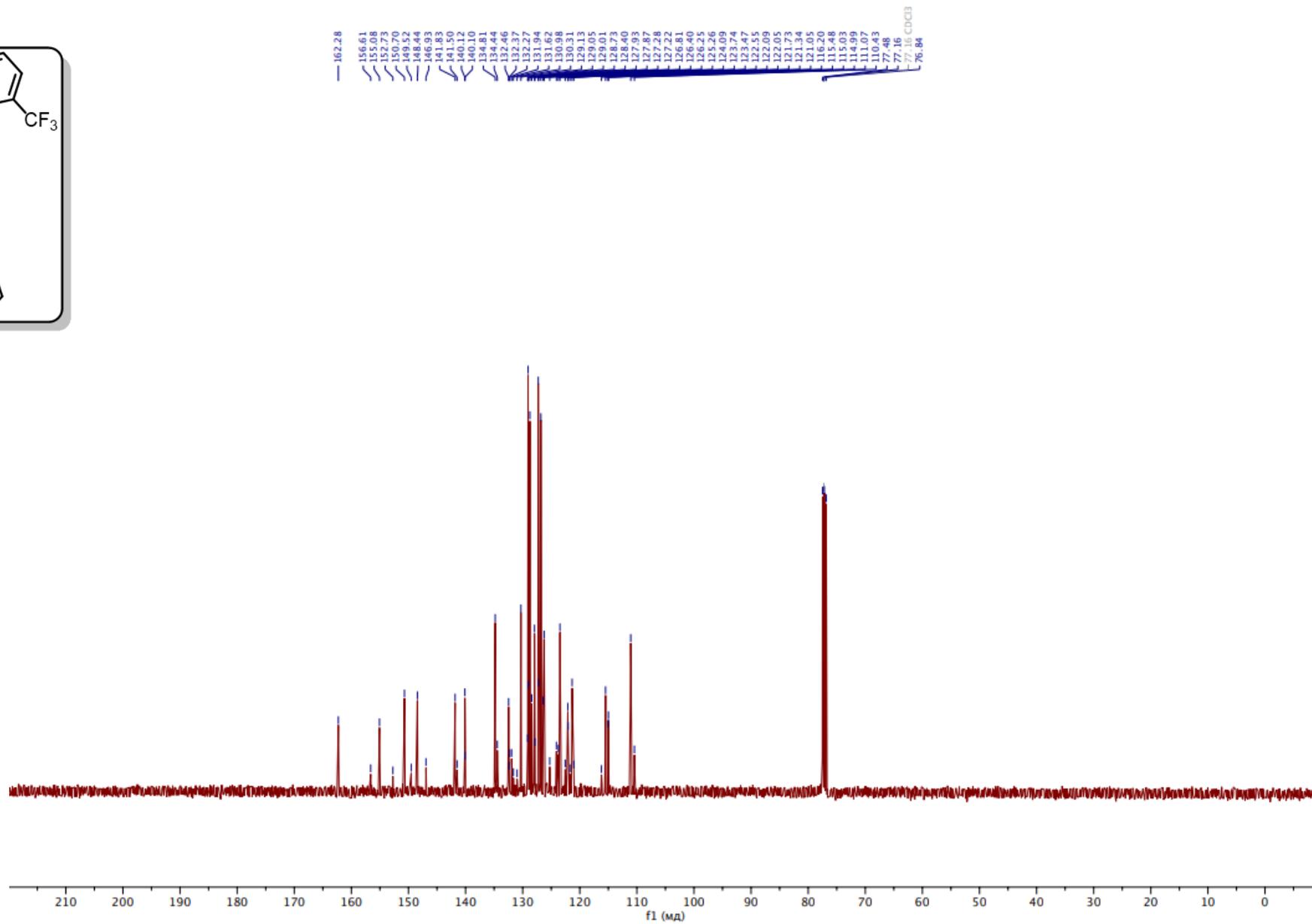
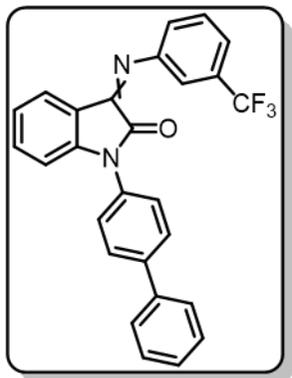
$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 3-isopropyl-1-mesityl-1*H*-indole (**7**)



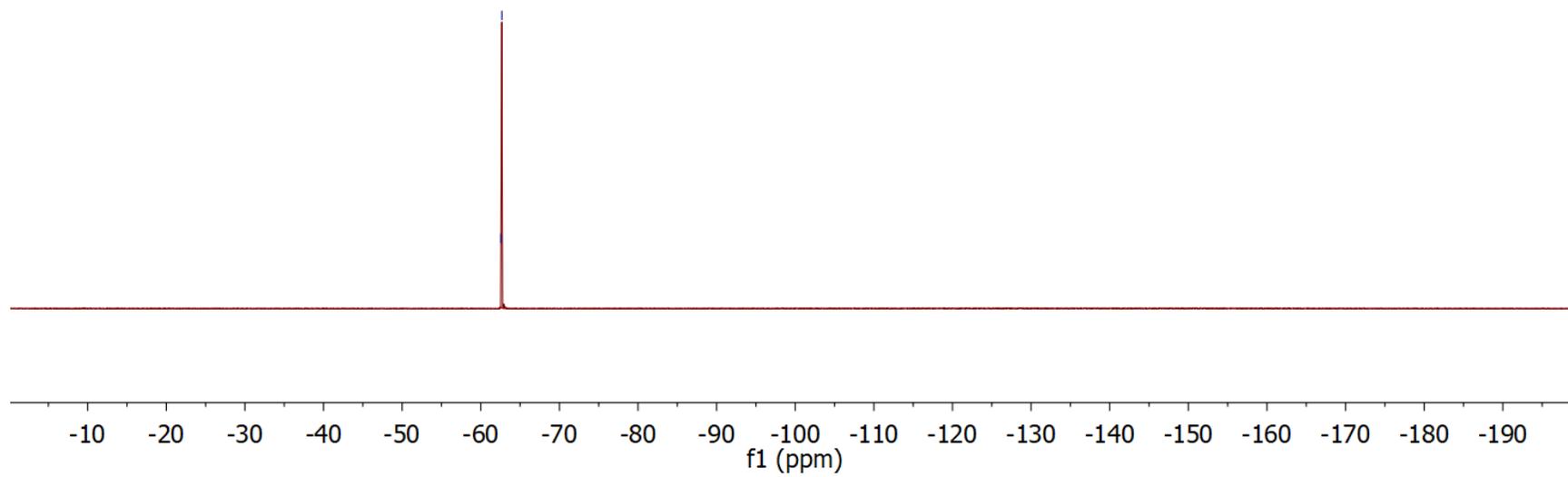
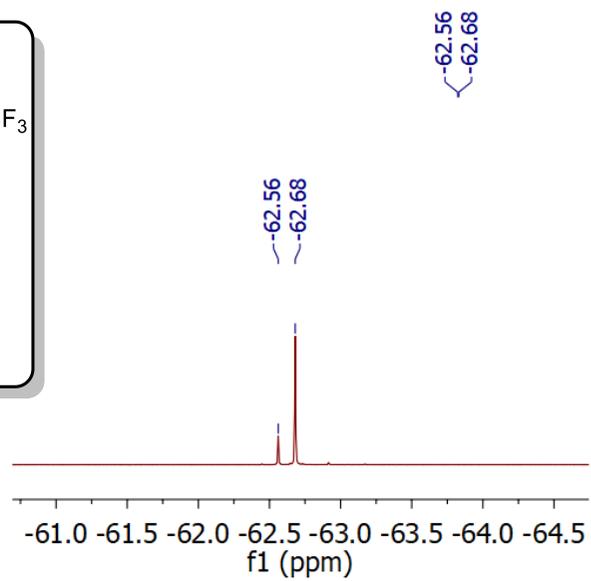
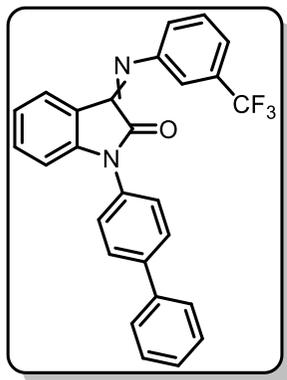
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 1-([1,1'-biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**)



$^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of 1-([1,1'-biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**)



$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) of 1-([1,1'-biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**)



S-XII. GC-MS for **8**

Chromatogram of 1-([1,1'-biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**) (obtained by GC/MS) (top) and electron ionization mass spectrum of 1-([1,1'-biphenyl]-4-yl)-3-((3-(trifluoromethyl)phenyl)imino)indolin-2-one (**8**) (below)

