

# *Supporting Information*

## **Synthesis of $\alpha$ -iodoketals from methyl ketones via sustainable and orthogonal tandem catalysis**

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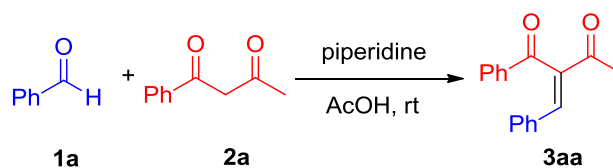
### **Appendix**

Spectral copies of <sup>1</sup>H NMR and <sup>13</sup>C NMR of compounds obtained in this study

## 1 General methods:

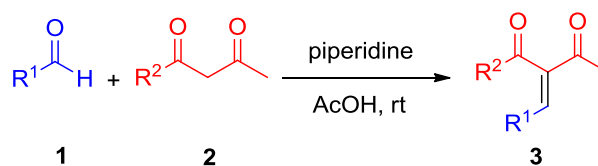
All reagents were purchased from commercial suppliers and used without further purification. IR spectra were recorded on an infrared spectrometer as KBr pellets with absorption in  $\text{cm}^{-1}$ .  $^1\text{H}$  spectra were recorded in  $\text{CDCl}_3$  on 400/600 MHz NMR spectrometers and resonances ( $\delta$ ) are given in ppm relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, brs = broad singlet), coupling constants (Hz) and integration.  $^{13}\text{C}$  spectra were recorded in  $\text{CDCl}_3$  on 100/150 MHz spectrometers and resonances ( $\delta$ ) are given in ppm relative to the center line of a triplet at 77.0 ppm of chloroform-*d*. HRMS were obtained on an Apex-Ultra MS equipped with APCI or ESI source. Melting points were determined using XT-4 apparatus and not corrected. The X-ray crystal-structure determination of **3ba**, **3ia** and **4aa** was obtained on a Bruker SMART APEX CCD system. Column chromatography was performed on silica gel (200–300 mesh).

## 2 General procedure for the preparation of (Z)-2-arylidene-1-arylbutane-1,3-dione (3aa as an example):



Piperidine (20 mL) was added dropwise to the stirred solution of acetic acid (25 mL) in ice bath. Benzaldehyde **1a** (1.06 g, 10.0 mmol) and 1-phenylbutane-1,3-dione **2a** (1.62 g, 10.0 mmol) were then added into the mixture solution and stirred at room temperature for overnight. After the reaction completed, the mixture was diluted with water and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 200 mL), the combined organic extracts were washed with NaOH (5% w/w, aq.) and brine successively, then dried with Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 10:1) to afford a white solid **3aa** (2.15 g, 86%).

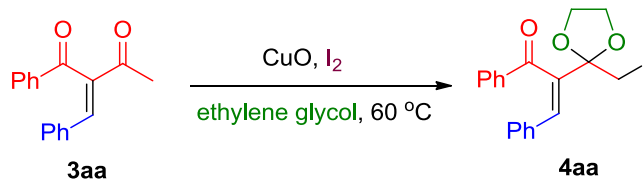
**Table 1.** Scope of substrates.<sup>a</sup>



Entry	R <sup>1</sup>	R <sup>2</sup>	<b>3</b>	Yield (%) <sup>b</sup>
1	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3aa</b>	86
2	<b>1b</b> (4-MeC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ba</b>	82
3	<b>1c</b> (4-MeOC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ca</b>	80
4	<b>1d</b> (4-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3da</b>	88
5	<b>1e</b> (1-naphthyl)	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ea</b>	77
6	<b>1f</b> (2-naphthyl)	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3fa</b>	78
7	<b>1g</b> (4-ClC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ga</b>	83
8	<b>1h</b> (4-BrC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ha</b>	84
9	<b>1i</b> (4-FC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ia</b>	80
10	<b>1j</b> (2-thienyl)	<b>2a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>3ja</b>	84
11	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2b</b> (4-MeC <sub>6</sub> H <sub>4</sub> )	<b>3ab</b>	83
12	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2c</b> (2-naphthyl)	<b>3ac</b>	75
13	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2d</b> (4-ClC <sub>6</sub> H <sub>4</sub> )	<b>3ad</b>	83
14	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2e</b> (4-BrC <sub>6</sub> H <sub>4</sub> )	<b>3ae</b>	84
15	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2f</b> (2-furyl)	<b>3af</b>	60
16	<b>1a</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2g</b> (2-thienyl)	<b>3ag</b>	70

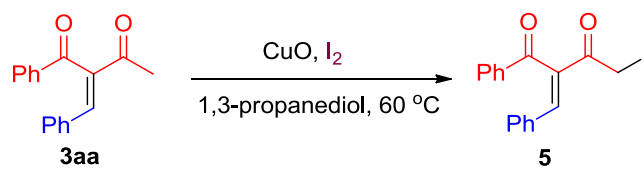
<sup>a</sup> Reaction was performed with aldehyde **1** (10.0 mmol), 1-arylbutane-1,3-dione **2** (10.0 mmol), and piperidine (20 mL) in acetic acid (25 mL) at rt overnight. <sup>b</sup> Isolated yield.

**General procedure for the preparation of  $\alpha$ -iodoketals of (Z)-2-arylidene-1-arylbutane-1,3-dione (4aa as an example):**



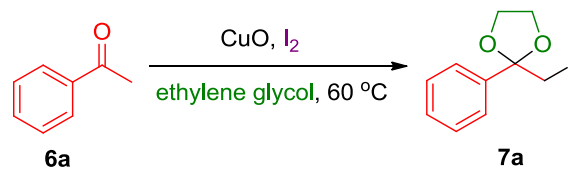
(Z)-2-benzylidene-1-phenylbutane-1,3-dione **3aa** (250 mg, 1.0 mmol),  $\text{CuO}$  (120 mg, 1.5 mmol), and iodine (305 mg, 1.2 mmol) were placed in a sealed tube. After addition of anhydrous ethylene glycol (5 mL), the mixture was stirred at  $60\text{ }^\circ\text{C}$  for 5 h. After the reaction completed, the mixture was diluted with water and treated with  $\text{Na}_2\text{S}_2\text{O}_3$  (5% w/w, aq.). The mixture was then extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 20\text{ mL}$ ), the combined organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 20:1) to afford a white solid **4aa** (357 mg, 85%).

**General procedure for the preparation of 5:**



(Z)-2-benzylidene-1-phenylbutane-1,3-dione **3aa** (250 mg, 1.0 mmol),  $\text{CuO}$  (120 mg, 1.5 mmol), and iodine (305 mg, 1.2 mmol) were placed in a sealed tube. After addition of anhydrous 1,3-propanediol (5 mL), the mixture was stirred at  $60\text{ }^\circ\text{C}$  for 5 h. After the reaction completed, the mixture was diluted with water and treated with  $\text{Na}_2\text{S}_2\text{O}_3$  (5% w/w, aq.). The mixture was then extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 20\text{ mL}$ ), the combined organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 15:1) to afford a yellow solid **5** (339 mg, 90%).

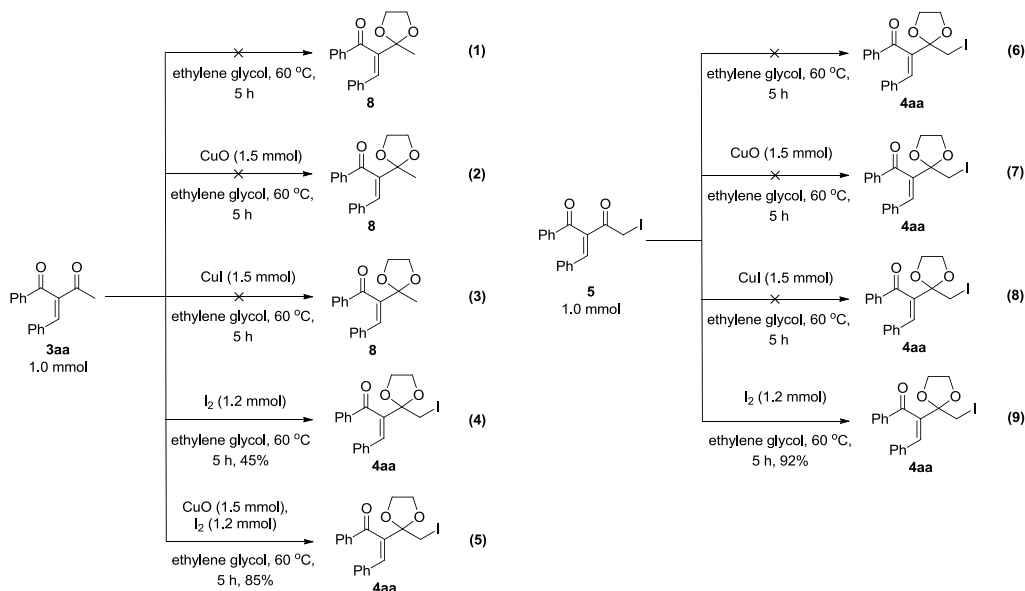
**General procedure for the preparation of  $\alpha$ -iodoketals of (Z)-2-arylidene-1-arylbutane-1,3-dione (7a as an example):**



Acetophenone **6a** (120 mg, 1.0 mmol),  $\text{CuO}$  (120 mg, 1.5 mmol), and iodine (305 mg, 1.2 mmol) were placed in a sealed tube. After addition of anhydrous 1,3-propanediol (5 mL), the mixture was stirred at  $60\text{ }^\circ\text{C}$  for 5 h. After the reaction completed, the mixture was diluted with water and treated with  $\text{Na}_2\text{S}_2\text{O}_3$  (5% w/w, aq.). The mixture was then extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 20\text{ mL}$ ), the combined organic extracts were

dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 80:1) to afford a yellow solid **7a** (104 mg, 36%).

### 3 Control experiments to provide insight into the mechanism:

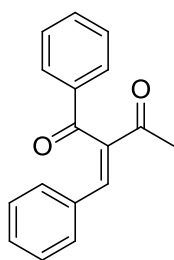


#### (10) Results and discussion of the control experiments:

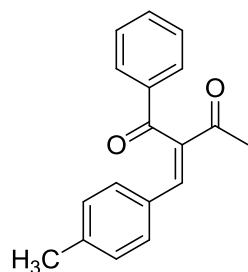
When **3aa** was used as substrate, no ketolization product **8** was obtained without  $\text{I}_2$  (Eq. 1, 2, 3); when **5** was used as substrate, no desired product **4aa** was obtained without  $\text{I}_2$  (Eq. 6, 7, 8); when **3aa** was used as substrate, the desired product **4aa** was obtained in 45% yield with  $\text{I}_2$  (Eq. 4) and 85% yield with CuO and  $\text{I}_2$  (Eq. 5); when **5** was used as substrate, the desired product **4aa** was obtained in 92% yield in the presence of  $\text{I}_2$  (Eq. 9).

Based on our previous studies and the control experiments, it's clearly confirmed that copper oxide could promote the iodination reaction and iodine could promote the ketalization reaction with ethylene glycol.

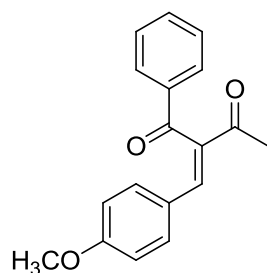
### 4 Spectroscopic Data:



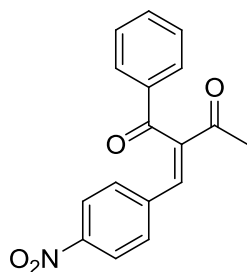
**(Z)-2-Benzylidene-1-phenylbutane-1,3-dione (3aa).** Yield 86%; White solid; m.p. 92.8–93.5 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 7.92 (d, *J* = 7.6 Hz, 2H), 7.79 (s, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.41–7.38 (m, 2H), 7.35–7.33 (m, 2H), 7.28–7.20 (m, 3H), 2.39 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): δ 198.0, 195.8, 141.1, 139.6, 135.9, 134.0, 132.8, 130.4, 130.2, 129.1, 128.9, 128.8, 27.2; IR (KBr): 1678, 1651, 1619, 1233, 1207 cm<sup>-1</sup>; HRMS (APCI): *m/z* [M + H]<sup>+</sup> calcd for C<sub>17</sub>H<sub>15</sub>O<sub>2</sub>: 251.1067; found: 251.1068.



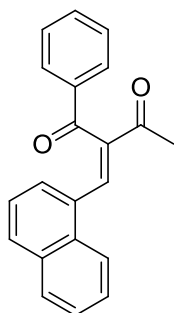
**(Z)-2-(4-Methylbenzylidene)-1-phenylbutane-1,3-dione (3ba).** Yield 82%; White solid; m.p. 72.5–74.2 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 7.94–7.91 (m, 2H), 7.76 (s, 1H), 7.55–7.51 (m, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 2.38 (s, 3H), 2.25 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): δ 198.3, 195.9, 141.3, 141.1, 138.5, 136.0, 134.0, 130.4, 129.9, 129.5, 129.1, 128.9, 27.0, 21.3; IR (KBr): 1669, 1646, 1601, 1246, 1234, 1210 cm<sup>-1</sup>; HRMS (APCI): *m/z* [M + H]<sup>+</sup> calcd for C<sub>18</sub>H<sub>17</sub>O<sub>2</sub>: 265.1223; found: 265.1224.



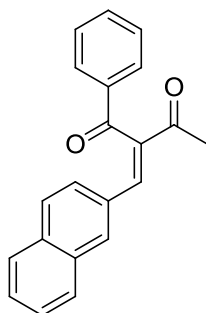
**(Z)-2-(4-Methoxybenzylidene)-1-phenylbutane-1,3-dione (3ca).** Yield 80%; White solid; m.p. 70.6–72.1 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.93 (d,  $J = 7.2$  Hz, 2H), 7.74 (s, 1H), 7.53 (t,  $J = 7.2$  Hz, 1H), 7.40 (t,  $J = 7.2$  Hz, 2H), 7.31–7.27 (m, 2H), 6.73 (d,  $J = 8.8$  Hz, 2H), 3.72 (s, 3H), 2.37 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  198.5, 195.8, 161.4, 141.0, 137.2, 136.0, 133.9, 132.4, 129.0, 128.8, 125.2, 114.3, 55.2, 26.9; IR (KBr): 1670, 1645, 1601, 1513, 1178  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$  [ $\text{M} + \text{H}$ ] $^+$  calcd for  $\text{C}_{18}\text{H}_{17}\text{O}_3$ : 281.1172; found: 281.1173.



**(Z)-2-(4-Nitrobenzylidene)-1-phenylbutane-1,3-dione (3da).** Yield 88%; Light yellow solid; m.p. 152.1–153.6 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.08–8.05 (m, 2H), 7.89 (d,  $J = 8.0$  Hz, 2H), 7.82 (s, 1H), 7.60–7.56 (m, 1H), 7.50 (d,  $J = 8.8$  Hz, 2H), 7.45–7.41 (m, 2H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.9, 195.1, 148.2, 142.7, 139.2, 137.7, 135.4, 134.7, 130.6, 129.2, 129.1, 123.9, 27.5; IR (KBr): 1678, 1657, 1595, 1519, 1347, 1231  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$  [ $\text{M} + \text{H}$ ] $^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{NO}_4$ : 296.0917; found: 296.0917.



**(Z)-2-(Naphthalen-1-ylmethylene)-1-phenylbutane-1,3-dione (3ea).** Yield 77%;  
Yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.60 (s, 1H), 8.10 (d,  $J = 8.4$  Hz, 1H),  
7.80–7.78 (m, 3H), 7.71 (d,  $J = 8.4$  Hz, 1H), 7.63–7.59 (m, 1H), 7.54–7.50 (m, 1H),  
7.40–7.35 (m, 2H), 7.24–7.19 (m, 3H), 2.48 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$   
197.4, 195.5, 141.4, 139.2, 136.1, 133.7, 133.3, 131.4, 130.6, 130.4, 128.9, 128.8,  
128.6, 127.9, 127.0, 126.4, 125.2, 123.7, 27.8; IR (KBr): 1679, 1658, 1595, 1231  $\text{cm}^{-1}$ ;  
HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{21}\text{H}_{17}\text{O}_2$ : 301.1223; found: 301.1224.

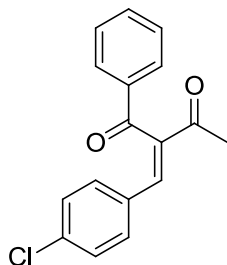


**(Z)-2-(Naphthalen-2-ylmethylene)-1-phenylbutane-1,3-dione (3fa).** Yield 78%;  
Light yellow solid; m.p. 98.7–99.4  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.97–7.95 (m,  
3H), 7.89 (s, 1H), 7.73–7.68 (m, 2H), 7.61 (d,  $J = 8.4$  Hz, 1H), 7.51–7.35 (m, 6H),  
2.43 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  198.2, 195.8, 141.2, 139.5, 136.1, 134.1,  
133.8, 132.8, 131.9, 130.3, 129.1, 128.9, 128.6, 128.5, 127.7, 127.5, 126.7, 125.9,



27.2; IR (KBr): 1675, 1648, 1610, 1362, 1262, 1231, 1174  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$

$[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{21}\text{H}_{17}\text{O}_2$ : 301.1223; found: 301.1224.



**(Z)-2-(4-Chlorobenzylidene)-1-phenylbutane-1,3-dione (3ga).** Yield 83%; White

solid; m.p. 90.2–91.7 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.90 (d,  $J = 7.6$  Hz, 2H),

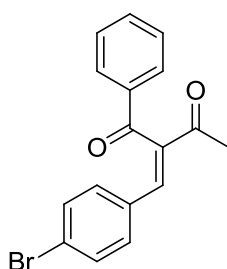
7.74 (s, 1H), 7.58–7.54 (m, 1H), 7.42 (t,  $J = 7.2$  Hz, 2H), 7.29–7.27 (m, 2H), 7.20 (d,

$J = 8.0$  Hz, 2H), 2.39 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.7, 195.5, 139.9,

139.5, 136.5, 135.7, 134.3, 131.4, 131.3, 129.1, 129.0, 27.2; IR (KBr): 1679, 1655,

1618, 1230, 1210, 1089  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{ClO}_2$ :

285.0677; found: 285.0679.



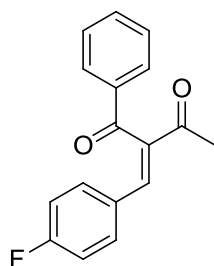
**(Z)-2-(4-Bromobenzylidene)-1-phenylbutane-1,3-dione (3ha).** Yield 84%; White

solid; m.p. 88.0–89.4 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.90 (d,  $J = 8.0$  Hz, 2H),

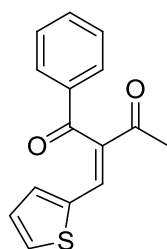
7.71 (s, 1H), 7.54 (t,  $J = 7.6$  Hz, 1H), 7.41 (t,  $J = 7.6$  Hz, 2H), 7.35–7.33 (m, 2H),

7.20 (d,  $J = 8.4$  Hz, 2H), 2.38 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.6, 195.5,

140.0, 139.5, 135.6, 134.2, 131.9, 131.6, 131.4, 129.0, 128.9, 124.9, 27.1; IR (KBr): 1673, 1653, 1616, 1583, 1238  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{BrO}_2$ : 329.0172; found: 329.0173.

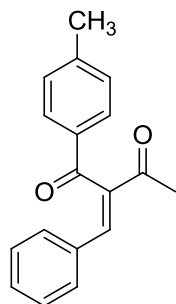


**(Z)-2-(4-Fluorobenzylidene)-1-phenylbutane-1,3-dione (3ia).** Yield 80%; Light yellow solid; m.p. 110.6–111.8 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.92–7.90 (m, 2H), 7.76 (s, 1H), 7.56 (t,  $J = 7.6$  Hz, 1H), 7.42 (t,  $J = 7.6$  Hz, 2H), 7.36–7.33 (m, 2H), 6.92 (t,  $J = 8.4$  Hz, 2H), 2.38 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  198.0, 195.6, 165.0, 162.5, 139.8, 139.27, 139.25, 135.8, 134.3, 132.4, 132.3, 129.1, 129.0, 116.2, 116.0, 27.3; IR (KBr): 1681, 1650, 1620, 1596, 1508, 1228, 1164  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{FO}_2$ : 269.0972; found: 269.0974

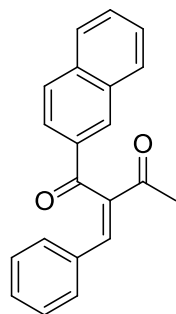


**(Z)-1-Phenyl-2-(thiophen-2-ylmethylene)butane-1,3-dione (3ja).** Yield 84%; White solid; m.p. 123.6–124.9 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.99–7.97 (m, 2H), 7.90 (s, 1H), 7.59 (t,  $J = 7.6$  Hz, 1H), 7.45 (t,  $J = 7.6$  Hz, 2H), 7.38 (d,  $J = 4.8$  Hz, 1H),

7.27–7.26 (m, 1H), 6.98–6.96 (m, 1H), 2.36 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.7, 195.2, 136.5, 136.2, 135.9, 134.2, 133.2, 132.0, 129.2, 128.9, 128.0, 27.0; IR (KBr): 1670, 1647, 1601, 1267, 1238, 1219, 1202  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{13}\text{O}_2\text{S}$ : 257.0631; found: 257.0632.

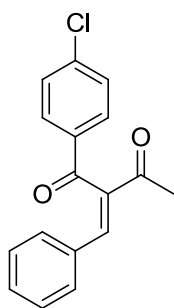


**(Z)-2-Benzylidene-1-(p-tolyl)butane-1,3-dione (3ab).** Yield 83%; White solid; m.p. 106.0–107.2  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.82 (d,  $J = 8.0$  Hz, 2H), 7.77 (s, 1H), 7.35 (d,  $J = 7.6$  Hz, 2H), 7.26–7.19 (m, 5H), 2.37 (s, 3H), 2.35 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.5, 195.8, 145.1, 140.8, 139.7, 133.6, 132.8, 130.3, 130.2, 129.6, 129.2, 128.7, 27.2, 21.6; IR (KBr): 1675, 1649, 1620, 1605, 1237, 1208, 1182  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{18}\text{H}_{17}\text{O}_2$ : 265.1223; found: 265.1224.

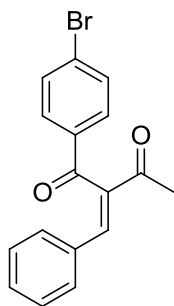


**(Z)-2-Benzylidene-1-(naphthalen-2-yl)butane-1,3-dione (3ac).** Yield 75%; Yellow solid; m.p. 86.5–87.5  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.37 (s, 1H), 8.07 (d,  $J = 8.4$

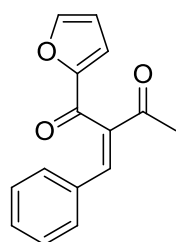
Hz, 1H), 7.88–7.81 (m, 4H), 7.56 (t,  $J = 6.8$  Hz, 1H), 7.48 (t,  $J = 6.8$  Hz, 1H), 7.40–7.38 (m, 2H), 7.20–7.16 (m, 3H), 2.42 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.9, 195.8, 141.2, 139.6, 136.0, 133.4, 132.8, 132.5, 131.8, 130.5, 130.3, 129.7, 129.0, 128.9, 128.8, 127.7, 126.8, 123.8, 27.3; IR (KBr): 1668, 1653, 1620, 1243, 1182  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{21}\text{H}_{17}\text{O}_2$ : 301.1223; found: 301.1224.



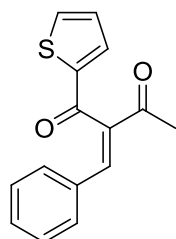
**(Z)-2-Benzylidene-1-(4-chlorophenyl)butane-1,3-dione (3ad).** Yield 83%; White solid; m.p. 108.4–111.0 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.86–7.82 (m, 2H), 7.79 (s, 1H), 7.39–7.35 (m, 2H), 7.33–7.22 (m, 5H), 2.42 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.7, 195.9, 141.6, 140.6, 139.1, 134.3, 132.6, 130.7, 130.4, 130.2, 129.3, 128.9, 27.1; IR (KBr): 1681, 1648, 1621, 1586, 1230, 1208  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{ClO}_2$ : 285.0677; found: 285.0679.



**(Z)-2-Benzylidene-1-(4-bromophenyl)butane-1,3-dione (3ae).** Yield 84%; White solid; m.p. 125.4–126.5 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.79–7.75 (m, 3H), 7.54 (d,  $J = 8.4$  Hz, 2H), 7.33–7.23 (m, 5H), 2.42 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.9, 195.9, 141.6, 139.1, 134.7, 132.5, 132.2, 130.7, 130.5, 130.2, 129.4, 128.9, 27.0; IR (KBr): 1680, 1647, 1622, 1583, 1230, 1209  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{BrO}_2$ : 329.0172; found: 329.0173.

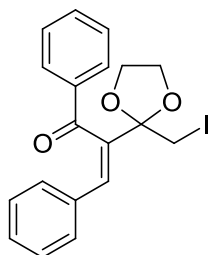


**(Z)-2-Benzylidene-1-(furan-2-yl)butane-1,3-dione (3af).** Yield 60%; White solid; m.p. 118.5–119.6 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.79 (s, 1H), 7.56 (brs, 1H), 7.39–7.37 (m, 2H), 7.33–7.25 (m, 3H), 7.06 (d,  $J = 3.6$  Hz, 1H), 6.45–6.43 (m, 1H), 2.42 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  195.3, 184.6, 152.2, 147.9, 142.1, 138.5, 132.8, 130.5, 130.1, 128.8, 120.4, 112.6, 27.2; IR (KBr): 1665, 1646, 1617, 1242  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{13}\text{O}_3$ : 241.0859; found: 241.0860.



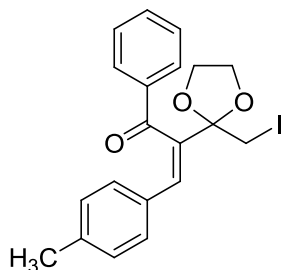
**(Z)-2-Benzylidene-1-(thiophen-2-yl)butane-1,3-dione (3ag).** Yield 70%; White solid; m.p. 96.3–97.5 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.79 (s, 1H), 7.66 (d,  $J = 4.0$  Hz, 1H), 7.51 (d,  $J = 2.8$  Hz, 1H), 7.43–7.41 (m, 2H), 7.32–7.24 (m, 3H), 7.01 (t,  $J =$

4.4 Hz, 1H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  195.1, 189.8, 143.6, 141.2, 139.2, 135.7, 134.7, 132.8, 130.6, 130.3, 128.8, 128.5, 27.3; IR (KBr): 1660, 1638, 1615, 1413, 1246  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{15}\text{H}_{13}\text{O}_2\text{S}$ : 257.0631; found: 257.0633.



**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-1,3-diphenylprop-2-en-1-one (4aa).**

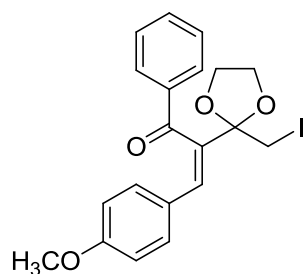
Yield 85%; White solid; m.p. 135.5–136.4 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.84–7.82 (m, 2H), 7.43 (t,  $J = 7.6$  Hz, 1H), 7.30 (t,  $J = 7.6$  Hz, 2H), 7.21 (s, 1H), 7.19–7.12 (m, 5H), 4.19–4.16 (m, 2H), 4.04–4.01 (m, 2H), 3.88 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.7, 137.6, 135.9, 134.1, 133.4, 131.4, 129.2, 129.1, 128.6, 128.5, 128.4, 107.2, 66.1, 13.3; IR (KBr): 1650, 1238, 1036  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{19}\text{H}_{18}\text{IO}_3$ : 421.0295; found: 421.0295.



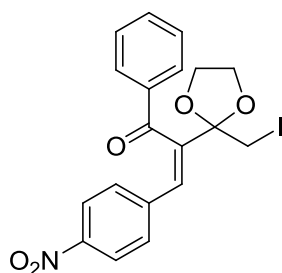
**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-1-phenyl-3-(p-tolyl)prop-2-en-1-one**

**(4ba).** Yield 83%; White solid; m.p. 145.1–146.9 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$

7.86–7.83 (m, 2H), 7.44 (t,  $J = 7.2$  Hz, 1H), 7.30 (t,  $J = 7.2$  Hz, 2H), 7.16 (s, 1H), 7.07 (d,  $J = 8.0$  Hz, 2H), 6.92 (d,  $J = 8.0$  Hz, 2H), 4.17–4.14 (m, 2H), 4.02–3.99 (m, 2H), 3.87 (s, 2H), 2.19 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.9, 138.7, 136.4, 136.0, 133.3, 131.3, 131.1, 129.1, 128.5, 107.2, 66.0, 21.1, 13.4; IR (KBr): 1648, 1237, 1038  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{20}\text{H}_{20}\text{IO}_3$ : 435.0452; found: 435.0452.



**(*E*)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (4ca).** Yield 82%; White solid; m.p. 127.2–128.7 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.86–7.84 (m, 2H), 7.45 (t,  $J = 7.2$  Hz, 1H), 7.31 (d,  $J = 7.2$  Hz, 2H), 7.14–7.10 (m, 3H), 6.65 (d,  $J = 8.8$  Hz, 2H), 4.17–4.14 (m, 2H), 4.02–3.99 (m, 2H), 3.87 (s, 2H), 3.69 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  198.1, 159.8, 136.0, 135.2, 133.4, 131.0, 130.7, 129.2, 128.5, 126.6, 113.8, 107.2, 66.0, 55.1, 13.5; IR (KBr): 1643, 1608, 1511, 1261, 1178, 1032  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{20}\text{H}_{20}\text{IO}_4$ : 451.0401; found: 451.0401.



**(E)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-3-(4-nitrophenyl)-1-phenylprop-2-en-1-o**

**ne (4da).** Yield 84%; White solid; m.p. 163.7–164.5 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):

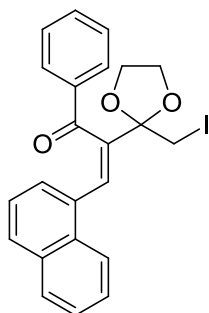
δ 7.99 (d, *J* = 8.8 Hz, 2H), 7.81–7.79 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.35–7.31 (m,

4H), 7.25 (s, 1H), 4.23–4.19 (m, 2H), 4.07–4.03 (m, 2H), 3.85 (s, 2H); <sup>13</sup>C NMR

(CDCl<sub>3</sub>, 100 MHz): δ 196.8, 147.3, 142.0, 140.6, 135.5, 134.0, 129.6, 129.1, 128.82,

128.76, 123.7, 107.2, 66.3, 12.4; IR (KBr): 1656, 1594, 1515, 1342, 1234, 1034 cm<sup>-1</sup>;

HRMS (APCI): *m/z* [M + H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>17</sub>INO<sub>5</sub>: 466.0146; found: 466.0146.



**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-3-(naphthalen-1-yl)-1-phenylprop-2-en-**

**1-one (4ea).** Yield 60%; White solid; m.p. 131.3–132.4 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400

MHz): δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.95 (s, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.67–7.65 (m,

2H), 7.60–7.56 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.25–7.16 (m, 2H), 7.11 (t, *J* = 7.2

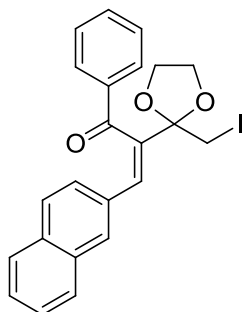
Hz, 1H), 7.01 (t, *J* = 7.6 Hz, 2H), 4.29–4.23 (m, 2H), 4.22–4.16 (m, 2H), 4.01 (s, 2H);

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): δ 197.2, 139.5, 136.3, 133.2, 132.9, 131.6, 131.2, 130.6,

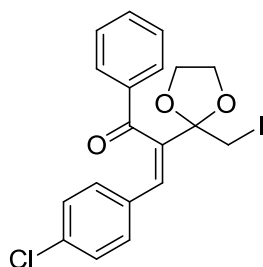
129.0, 128.63, 128.58, 128.0, 127.6, 126.5, 126.0, 125.1, 123.8, 107.5, 66.2, 13.1; IR



(KBr): 1658, 1230, 1176, 1036  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{23}\text{H}_{20}\text{IO}_3$ : 471.0452; found: 471.0452.

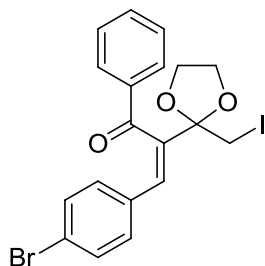


**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-3-(naphthalen-2-yl)-1-phenylprop-2-en-1-one (4fa).** Yield 72%; White solid; m.p. 120.1–122.0 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.88–7.86 (m, 2H), 7.69–7.65 (m, 3H), 7.55(d,  $J = 8.4$  Hz, 1H), 7.41–7.35 (m, 4H), 7.27–7.23 (m, 3H), 4.21–4.18 (m, 2H), 4.07–4.03 (m, 2H), 3.93 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.8, 137.7, 136.0, 133.4, 132.91, 132.87, 131.6, 131.4, 129.4, 129.0, 128.5, 128.1, 128.0, 127.4, 126.6, 126.3, 126.0, 107.3, 66.1, 13.3; IR (KBr): 1645, 1233, 1038  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{23}\text{H}_{20}\text{IO}_3$ : 471.0452; found: 471.0453.

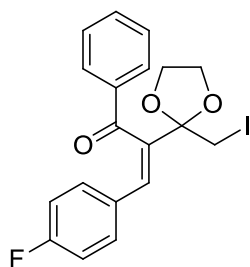


**(E)-3-(4-Chlorophenyl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-1-phenylprop-2-en-1-one (4ga).** Yield 85%; White solid; m.p. 123.7–124.6 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.83–7.81 (m, 2H), 7.47 (t,  $J = 7.2$  Hz, 1H), 7.32 (t,  $J = 7.2$  Hz, 2H), 7.15 (s,

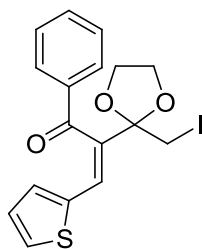
1H), 7.10 (brs, 4H), 4.19–4.16 (m, 2H), 4.03–4.00 (m, 2H), 3.85 (s, 2H);  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  197.5, 138.3, 135.7, 134.5, 133.7, 132.6, 130.3, 130.0, 129.1, 128.64, 128.60, 107.2, 66.1, 13.0; IR (KBr): 1649, 1237, 1038 cm<sup>-1</sup>; HRMS (APCI):  $m/z$  [M + H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>17</sub>ClIO<sub>3</sub>: 454.9905; found: 454.9906.



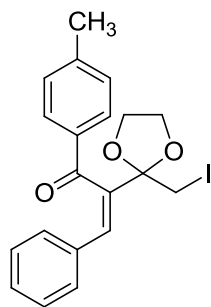
**(E)-3-(4-Bromophenyl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-1-phenylprop-2-en-1-one (4ha).** Yield 78%; White solid; m.p. 126.0–127.0 °C;  $^1\text{H}$  NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.83–7.81 (m, 2H), 7.47 (t,  $J$  = 7.6 Hz, 1H), 7.32 (t,  $J$  = 7.6 Hz, 2H), 7.25 (d,  $J$  = 8.4 Hz, 2H), 7.12 (s, 1H), 7.04 (d,  $J$  = 8.4 Hz, 2H), 4.19–4.15 (m, 2H), 4.03–4.00 (m, 2H), 3.85 (s, 2H);  $^{13}\text{C}$  NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  197.4, 138.4, 135.7, 133.7, 133.0, 131.6, 130.5, 129.9, 129.1, 128.6, 122.8, 107.1, 66.1, 13.0; IR (KBr): 1648, 1236, 1038 cm<sup>-1</sup>; HRMS (APCI):  $m/z$  [M + H]<sup>+</sup> calcd for C<sub>19</sub>H<sub>17</sub>BrIO<sub>3</sub>: 498.9400; found: 498.9402.



**(E)-3-(4-Fluorophenyl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-1-phenylprop-2-en-1-one (4ia).** Yield 73%; White solid; m.p. 113.7–115.1 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.83–7.81 (m, 2H), 7.46 (t,  $J = 7.6$  Hz, 1H), 7.31 (t,  $J = 7.6$  Hz, 2H), 7.17–7.14 (m, 3H), 6.81 (t,  $J = 8.8$  Hz, 2H), 4.20–4.16 (m, 2H), 4.04–4.00 (m, 2H), 3.87 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.6, 163.8, 161.3, 137.5, 135.8, 133.6, 130.9, 130.8, 130.3, 130.25, 130.2, 129.1, 128.5, 115.6, 115.4, 107.2, 66.1, 13.1; IR (KBr): 1657, 1597, 1505, 1234, 1035  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{19}\text{H}_{17}\text{FIO}_3$ : 439.0201; found: 439.0201.

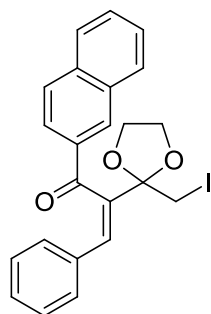


**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-1-phenyl-3-(thiophen-2-yl)prop-2-en-1-one (4ja).** Yield 89%; White solid; m.p. 144.4–146.0 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.95–7.93 (m, 2H), 7.52 (d,  $J = 7.6$  Hz, 1H), 7.39 (t,  $J = 7.6$  Hz, 2H), 7.23 (s, 1H), 7.15 (d,  $J = 5.2$  Hz, 1H), 6.94 (d,  $J = 3.6$  Hz, 1H), 6.84–6.82 (m, 1H), 4.18–4.14 (m, 2H), 4.04–4.01 (m, 2H), 3.81 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.2, 137.0, 136.0, 135.7, 133.7, 129.9, 129.2, 128.7, 128.1, 127.3, 123.3, 106.9, 66.1, 13.1; IR (KBr): 1653, 1232, 1209, 1170, 1035  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{16}\text{IO}_3\text{S}$ : 426.9859; found: 426.9860.



**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-3-phenyl-1-(p-tolyl)prop-2-en-1-one**

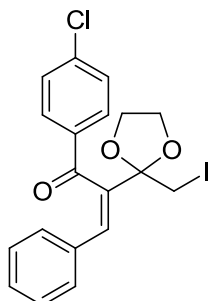
**(4ab).** Yield 82%; White solid; m.p. 92.6–94.2 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.74 (d,  $J = 8.0$  Hz, 2H), 7.20–7.17 (m, 3H), 7.13–7.08 (m, 5H), 4.18–4.14 (m, 2H), 4.03–3.99 (m, 2H), 3.87 (s, 2H), 2.31 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.3, 144.3, 137.7, 134.1, 133.5, 131.0, 129.3, 129.2, 129.1, 128.5, 128.4, 107.2, 66.0, 21.6, 13.4; IR (KBr): 1651, 1604, 1240, 1211, 1179, 1039  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$  [ $\text{M} + \text{H}$ ] $^+$  calcd for  $\text{C}_{20}\text{H}_{20}\text{IO}_3$ : 435.0452; found: 435.0452.



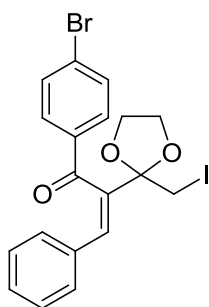
**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one**

**(4ac).** Yield 43%; White solid; m.p. 112.1–113.6 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.34 (s, 1H), 7.95–7.93 (m, 1H), 7.82–7.76 (m, 3H), 7.56–7.52 (m, 1H), 7.49–7.45 (m, 1H), 7.27–7.23 (m, 3H), 7.12–7.06 (m, 3H), 4.20–4.16 (m, 2H), 4.07–4.04 (m, 2H), 3.91 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  197.7, 137.8, 135.7, 134.1, 133.4, 132.4, 131.53, 131.49, 129.7, 129.1, 128.64, 128.60, 128.5, 127.7, 126.6,

124.3, 107.3, 66.2, 13.3; IR (KBr): 1653, 1626, 1218, 1184, 1033  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{23}\text{H}_{20}\text{IO}_3$ : 471.0452; found: 471.0452.

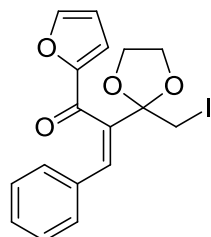


**(E)-1-(4-Chlorophenyl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-3-phenylprop-2-en-1-one (4ad).** Yield 78%; White solid; m.p. 98.7–100.1  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.77–7.75 (m, 2H), 7.27–7.25 (m, 2H), 7.23 (s, 1H), 7.15 (brs, 5H), 4.20–4.17 (m, 2H), 4.02–3.99 (m, 2H), 3.86 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.5, 139.8, 137.1, 134.3, 133.9, 131.7, 130.5, 129.1, 128.84, 128.80, 128.5, 107.3, 66.1, 13.0; IR (KBr): 1658, 1228, 1040  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{19}\text{H}_{17}\text{ClIO}_3$ : 454.9905; found: 454.9906.



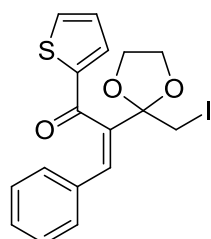
**(E)-1-(4-Bromophenyl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-3-phenylprop-2-en-1-one (4ae).** Yield 73%; White solid; m.p. 111.5–112.6  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.68 (d,  $J = 8.4$  Hz, 2H), 7.43 (d,  $J = 8.4$  Hz, 2H), 7.22 (s, 1H), 7.15 (brs,

5H), 4.20–4.17 (m, 2H), 4.02–3.99 (m, 2H), 3.86 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.7, 137.1, 134.7, 133.8, 131.8, 131.7, 130.5, 129.0, 128.8, 128.7, 128.5, 107.2, 66.1, 13.0; IR (KBr): 1658, 1221, 1040  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{19}\text{H}_{17}\text{BrIO}_3$ : 498.9400; found: 498.9402.



**(E)-1-(Furan-2-yl)-2-(2-(iodomethyl)-1,3-dioxolan-2-yl)-3-phenylprop-2-en-1-one**

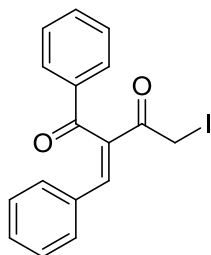
**(4af)**. Yield 85%; White solid; m.p. 137.6–139.2 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.434–7.428 (m, 1H), 7.21–7.17 (m, 6H), 6.93 (d,  $J = 3.6$  Hz, 1H), 6.34–6.33 (m, 1H), 4.21–4.17 (m, 2H), 4.07–4.04 (m, 2H), 3.88 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  184.6, 152.4, 147.1, 137.0, 134.2, 132.7, 128.8, 128.6, 128.4, 119.7, 112.3, 106.8, 66.0, 12.8; IR (KBr): 1641, 1624, 1040  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{16}\text{IO}_4$ : 411.0088; found: 411.0087



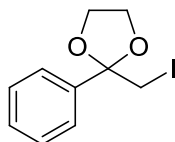
**(E)-2-(2-(Iodomethyl)-1,3-dioxolan-2-yl)-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-**

**one (4ag)**. Yield 88%; White solid; m.p. 136.4–137.3 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.53–7.51 (m, 1H), 7.45–7.44 (m, 1H), 7.26–7.23 (m, 2H), 7.19–7.16 (m,

4H), 6.91–6.89 (m, 1H), 4.20–4.19 (m, 2H), 4.07–4.04 (m, 2H), 3.89 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  189.5, 143.8, 138.0, 134.7, 134.2, 133.9, 131.8, 129.1, 128.7, 128.4, 128.1, 107.0, 66.1, 13.0; IR (KBr): 1642, 1625, 1411, 1245, 1054  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{16}\text{IO}_3\text{S}$ : 426.9859; found: 426.9860.

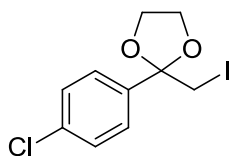


**(E)-2-Benzylidene-4-iodo-1-phenylbutane-1,3-dione (5).** Yield 90%; Yellow solid; m.p. 112.5–113.6  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.98 (s, 1H), 7.95 (d,  $J = 7.2$  Hz, 2H), 7.52 (t,  $J = 7.8$  Hz, 1H), 7.39 (t,  $J = 7.8$  Hz, 2H), 7.33 (d,  $J = 7.2$  Hz, 2H), 7.29–7.26 (m, 1H), 7.22 (t,  $J = 7.2$  Hz, 2H), 4.20 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  196.8, 190.6, 143.6, 135.9, 135.8, 134.1, 132.6, 130.8, 130.5, 129.4, 128.8, 128.7, 2.3; IR (KBr): 1668, 1639, 1616, 1258, 1233, 1211  $\text{cm}^{-1}$ ; HRMS (APCI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{IO}_2$ : 377.0033; found: 377.0034.

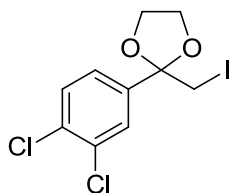


**2-(Iodomethyl)-2-phenyl-1,3-dioxolane (7a).** Yield 36%; Yellow solid; m.p. 65.5–67.0  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.51–7.49 (m, 2H), 7.38–7.33 (m, 3H), 4.20–4.17 (m, 2H), 3.88–3.84 (m, 2H), 3.58 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$

139.1, 128.6, 128.3, 125.8, 106.7, 65.6, 14.0; IR (KBr): 1208, 1159, 1035, 960, 703  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{12}\text{IO}_2$ : 290.9876; found: 290.9877.

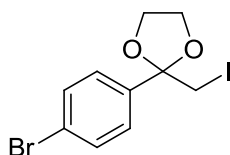


**2-(4-Chlorophenyl)-2-(iodomethyl)-1,3-dioxolane (7b).** Yield 43%; Yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.44 (d,  $J = 8.4$  Hz, 2H), 7.33 (d,  $J = 8.4$  Hz, 2H), 4.20–4.18 (m, 2H), 3.87–3.84 (m, 2H), 3.54 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150 MHz):  $\delta$  137.7, 134.6, 128.5, 127.4, 106.4, 65.7, 13.5; IR (KBr): 1487, 1208, 1091, 1040, 978, 832  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{ClIO}_2$ : 324.9487; found: 424.9488.

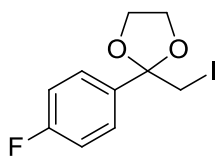


**2-(3,4-Dichlorophenyl)-2-(iodomethyl)-1,3-dioxolane (7c).** Yield 46%; Yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.60 (d,  $J = 2.4$  Hz, 1H), 7.33 (d,  $J = 8.4$  Hz, 1H), 7.35–7.32 (m, 1H), 4.21–4.18 (m, 2H), 3.89–3.85 (m, 2H), 3.51 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  139.7, 132.6, 130.4, 128.1, 125.4, 106.0, 65.9, 12.8; IR (KBr): 1467, 1378, 1208, 1167, 1033, 977  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{10}\text{Cl}_2\text{IO}_2$ : 358.9097; found: 358.9099.

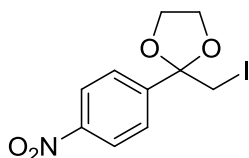




**2-(4-Bromophenyl)-2-(iodomethyl)-1,3-dioxolane (7d).** Yield 52%; Yellow solid; m.p. 56.2–58.3 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.49 (d,  $J = 8.4$  Hz, 2H), 7.38 (d,  $J = 8.4$  Hz, 2H), 4.20–4.17 (m, 2H), 3.87–3.84 (m, 2H), 3.53 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  138.3, 131.5, 127.7, 122.9, 106.5, 65.7, 13.3; IR (KBr): 1584, 1479, 1405, 1208, 1039, 976, 956, 940, 830  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{BrIO}_2$ : 368.8982; found: 368.8982.

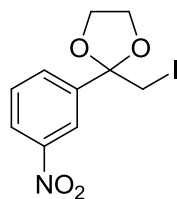


**2-(4-Fluorophenyl)-2-(iodomethyl)-1,3-dioxolane (7e).** Yield 44%; Yellow oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.49–7.47 (m, 2H), 7.05–7.02 (m, 2H), 4.20–4.19 (m, 2H), 3.87–3.85 (m, 2H), 3.55 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150 MHz):  $\delta$  163.6, 162.0, 135.0, 127.8, 127.7, 115.2, 115.1, 106.4, 65.7, 13.8; IR (KBr): 1602, 1504, 1225, 1158, 1040, 978, 840, 566  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{FIO}_2$ : 308.9782; found: 308.9784.

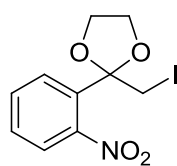


**2-(Iodomethyl)-2-(4-nitrophenyl)-1,3-dioxolane (7f).** Yield 98%; White solid; m.p. 108.1–111.4 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.22 (d,  $J = 8.8$  Hz, 2H), 7.70 (d,  $J =$

8.8 Hz, 2H), 4.26–4.23 (m, 2H), 3.91–3.88 (m, 2H), 3.55 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  148.1, 146.4, 127.1, 123.5, 106.3, 66.0, 12.3; IR (KBr): 1518, 1345, 1214, 1037, 855, 700  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{INO}_4$ : 335.9727; found: 335.9723.



**2-(Iodomethyl)-2-(3-nitrophenyl)-1,3-dioxolane (7g).** Yield 84%; White solid; m.p. 83.0–84.1 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.39–8.38 (m, 1H), 8.22–8.20 (m, 1H), 7.87–7.85 (m, 1H), 7.57 (t,  $J = 8.0$  Hz, 1H), 4.27–4.23 (m, 2H), 3.92–3.89 (m, 2H), 3.56 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  148.2, 141.8, 132.1, 129.4, 123.7, 121.2, 106.2, 66.0, 12.5; IR (KBr): 1524, 1347, 1213, 1045, 979, 690  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{INO}_4$ : 335.9727; found: 335.9726.



**2-(Iodomethyl)-2-(2-nitrophenyl)-1,3-dioxolane (7h).** Yield 72%; White solid; m.p. 108.3–110.5 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.72 (d,  $J = 7.6$  Hz, 1H), 7.57–7.47 (m, 3H), 4.18–4.15 (m, 2H), 3.92 (s, 2H), 3.79–3.76 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  149.6, 132.0, 131.2, 129.9, 129.0, 123.5, 105.7, 65.9, 12.5; IR (KBr): 1539, 1361, 1205, 1030, 976, 786  $\text{cm}^{-1}$ ; HRMS (ESI):  $m/z$   $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{10}\text{H}_{11}\text{INO}_4$ : 335.9727; found: 335.9727.

## 5. X-ray structure of compound **3ba**, **3ia**, **4aa**

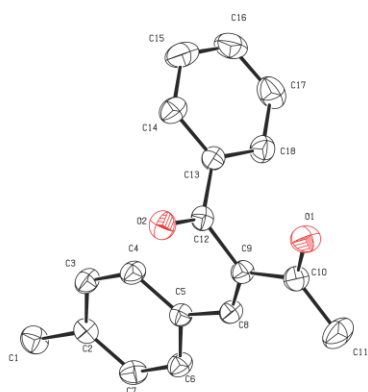


Figure S1. X-ray structure of compound **3ba** (all Hydrogen atoms are omitted for clarity, CCDC 884836).

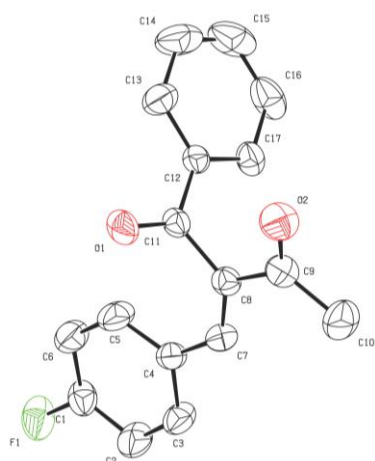


Figure S2. X-ray structure of compound **3ia** (all Hydrogen atoms are omitted for clarity, CCDC 884837).

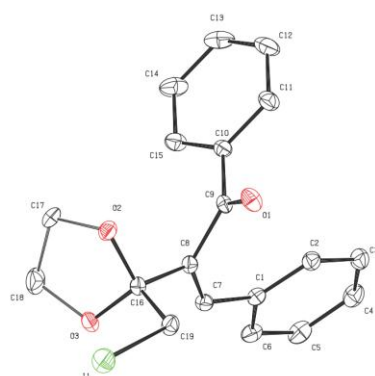


Figure S3. X-ray structure of compound **4aa** (all Hydrogen atoms are omitted for clarity, CCDC 885165).

## *Appendix*

### **Spectral Copies of $^1\text{H}$ NMR and $^{13}\text{C}$ NMR of Compounds Obtained in This Study**



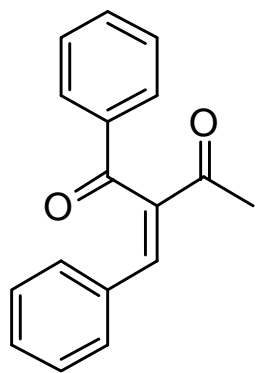
197.952  
195.767

141.140  
139.561  
135.933  
134.024  
132.778  
130.445  
130.243  
129.102  
128.877  
128.754

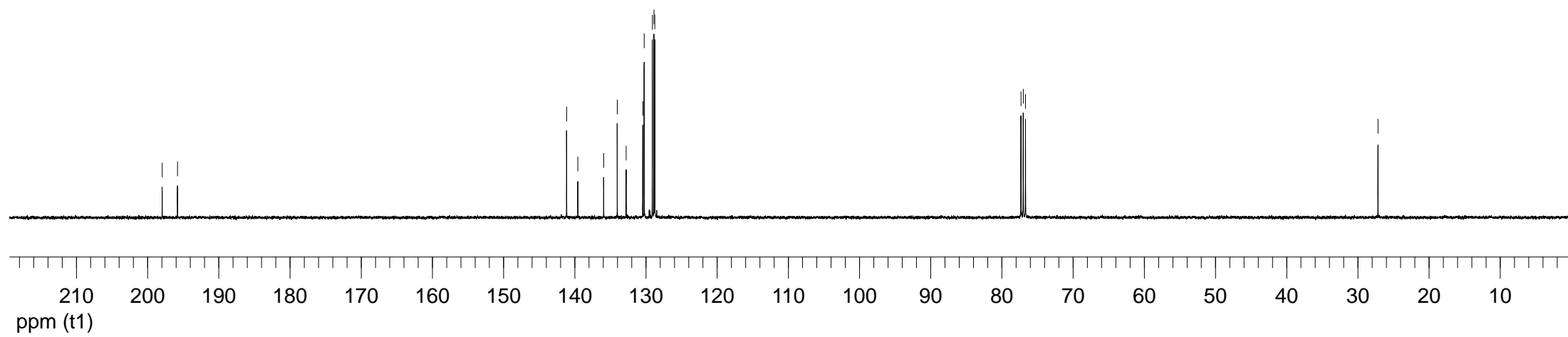
77.318  
77.000  
76.682

27.168

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



3aa

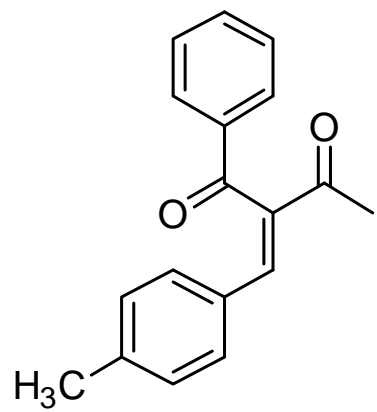


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

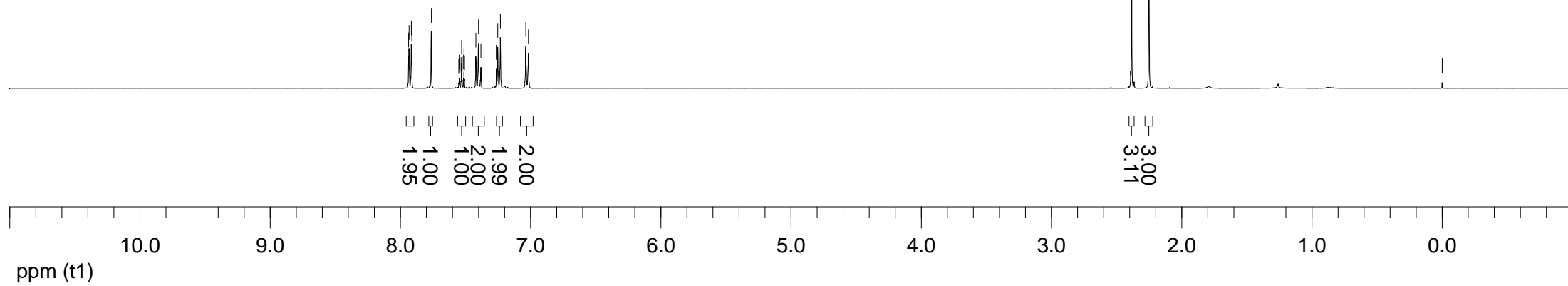
7.936  
7.934  
7.916  
7.912  
7.762  
7.551  
7.548  
7.545  
7.534  
7.530  
7.525  
7.514  
7.511  
7.508  
7.420  
7.400  
7.381  
7.263  
7.252  
7.232  
7.037  
7.016

2.384  
2.251

-0.000000



3ba



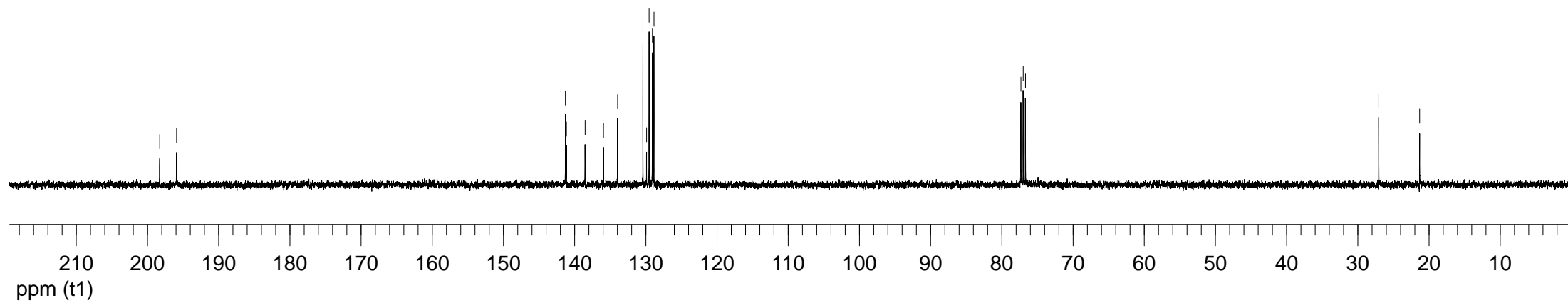
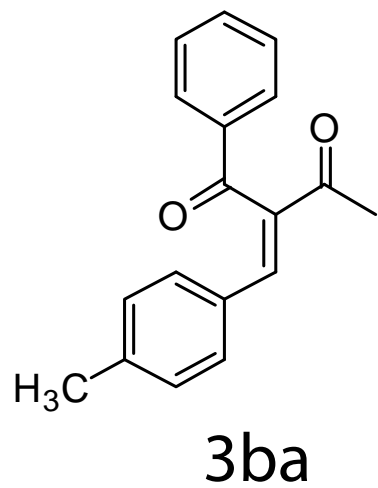
198.271  
195.908

141.288  
141.149  
138.532  
135.950  
133.959  
130.396  
129.894  
129.535  
129.076  
128.856

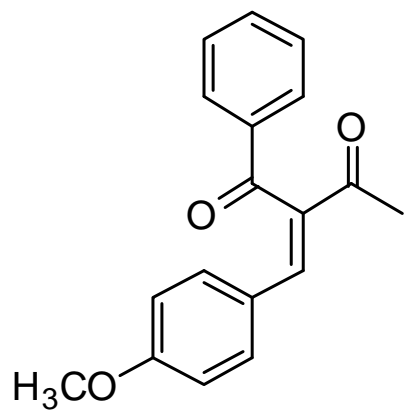
77.318  
77.000  
76.682

27.048  
21.312

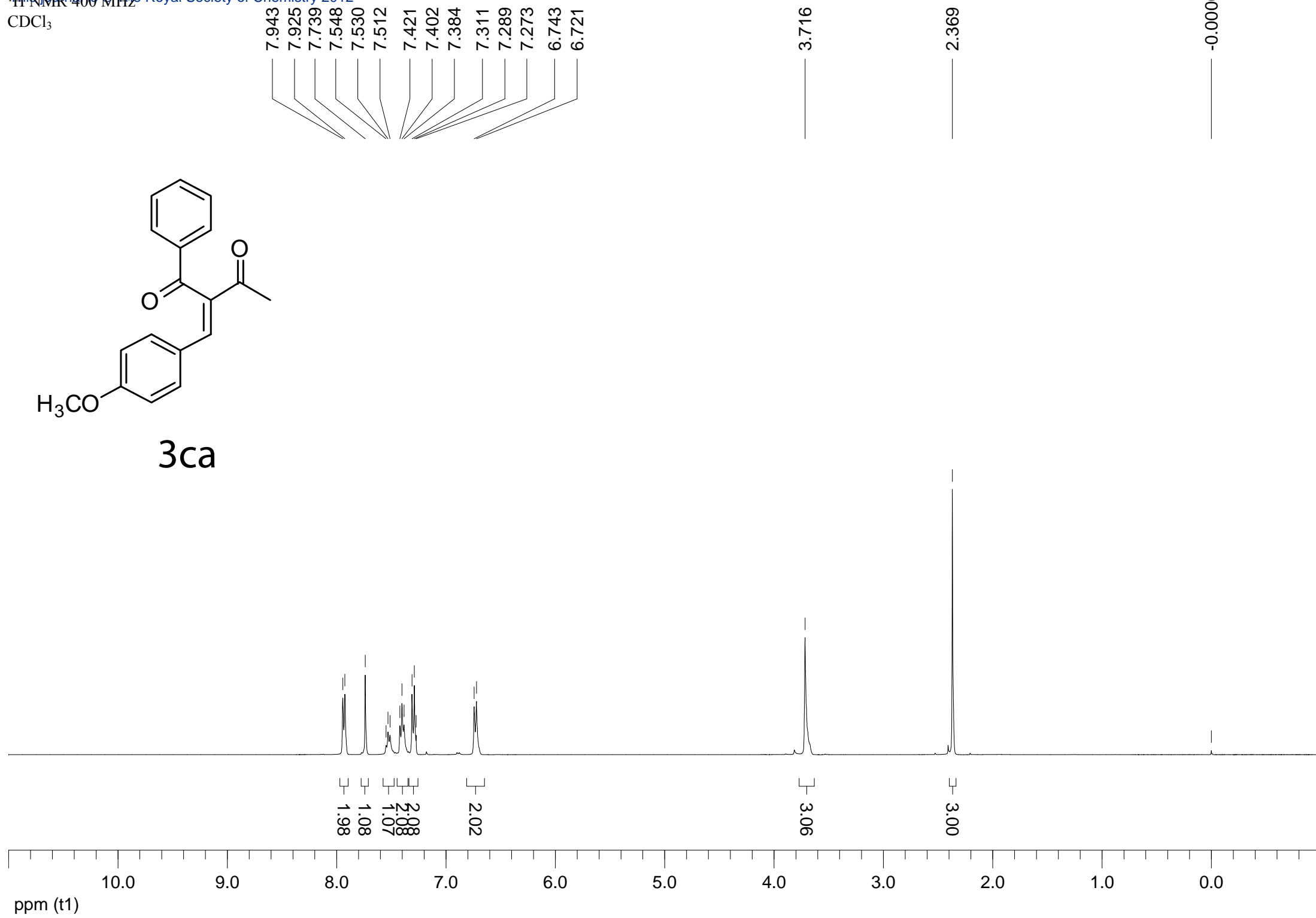
$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$

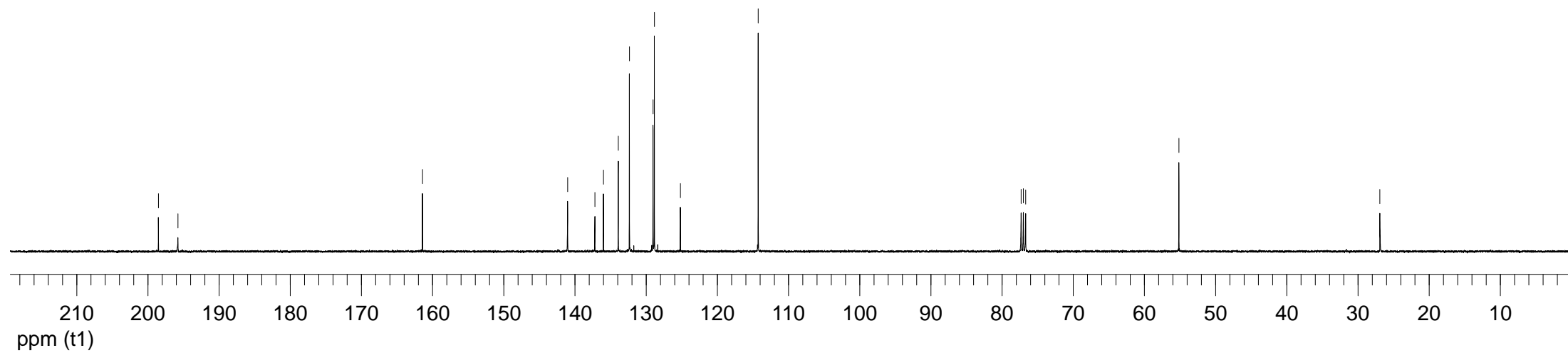
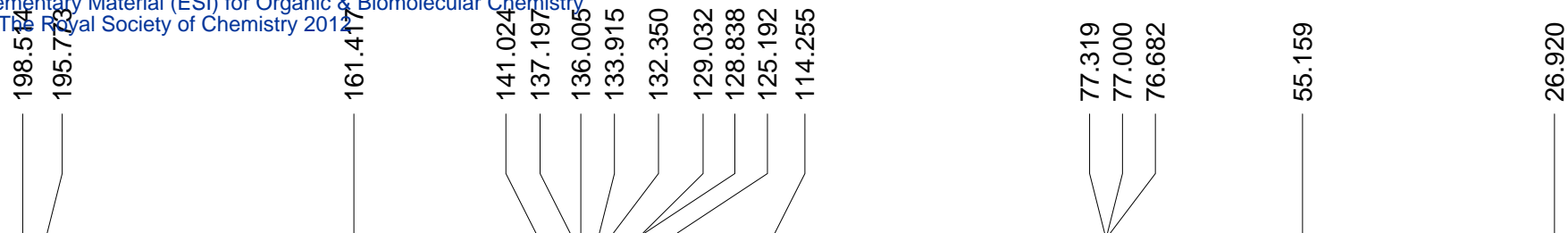
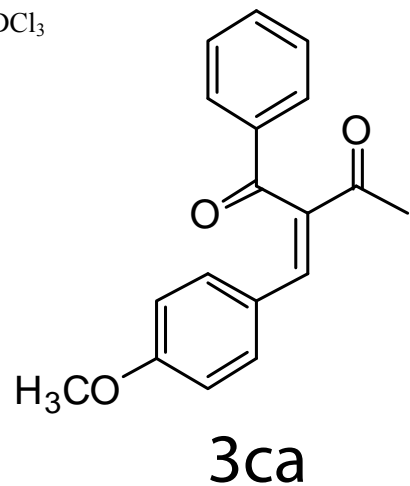






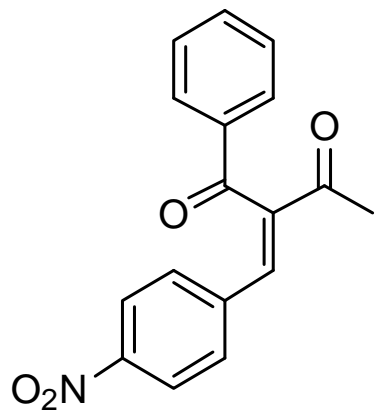
**3ca**



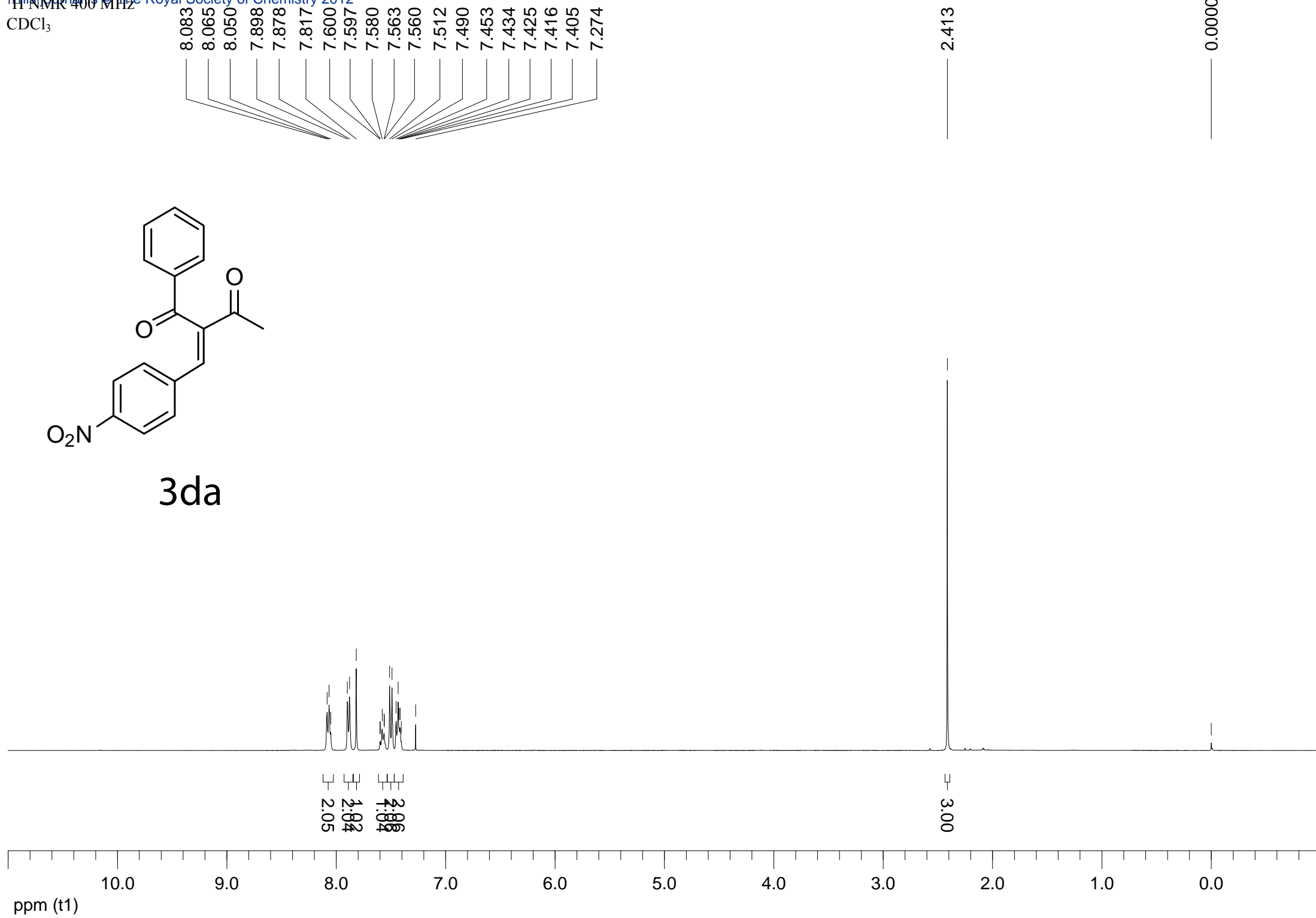


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

8.083  
8.065  
8.050  
7.898  
7.878  
7.817  
7.600  
7.597  
7.580  
7.563  
7.560  
7.512  
7.490  
7.453  
7.434  
7.425  
7.416  
7.405  
7.274



3da



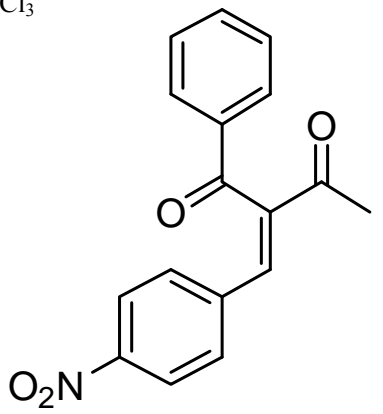
196.899  
195.061

148.1511  
142.6955  
139.1777  
137.6722  
135.432  
134.663  
130.577  
129.151  
129.087  
123.852

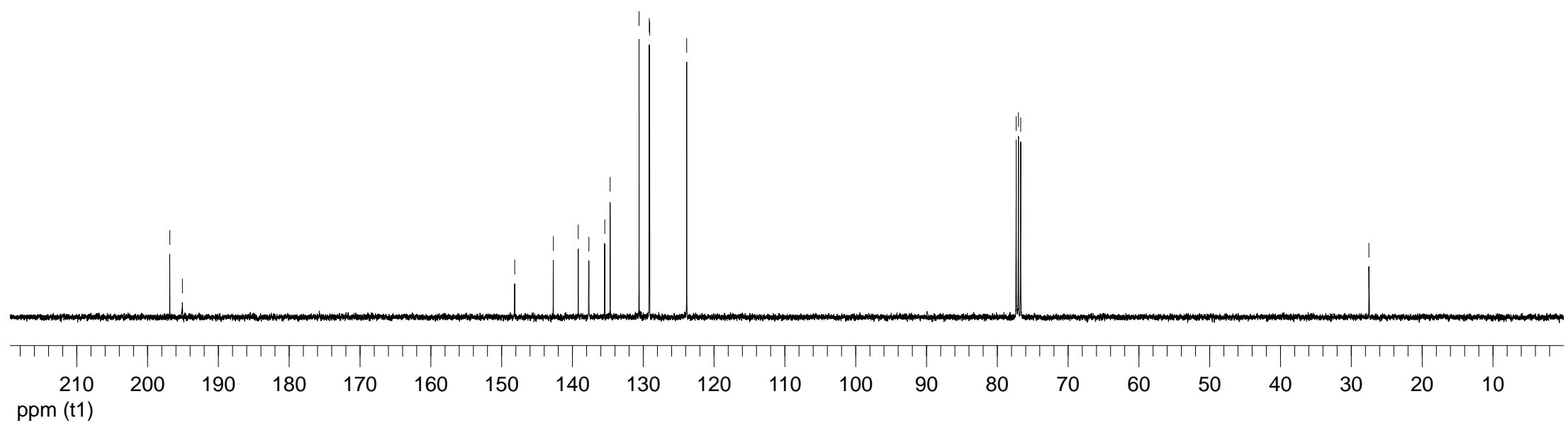
77.318  
77.000  
76.682

27.504

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

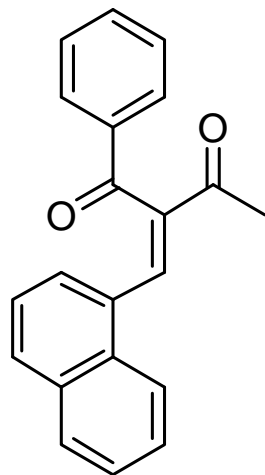


3da



8.604  
8.115  
8.094  
7.803  
7.786  
7.782  
7.724  
7.703  
7.630  
7.627  
7.613  
7.609  
7.606  
7.592  
7.588  
7.540  
7.538  
7.520  
7.517  
7.503  
7.500  
7.398  
7.391  
7.388  
7.380  
7.369  
7.365  
7.354  
7.351  
7.347  
7.257  
7.241  
7.227  
7.221  
7.207  
7.203  
7.189

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



3ea

1.00  
1.04  
2.99  
1.23  
1.02  
4.04  
3.07

3.00

0.000000

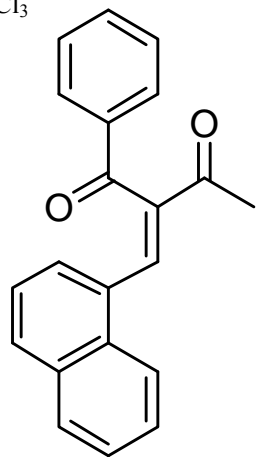
ppm (t1)

11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

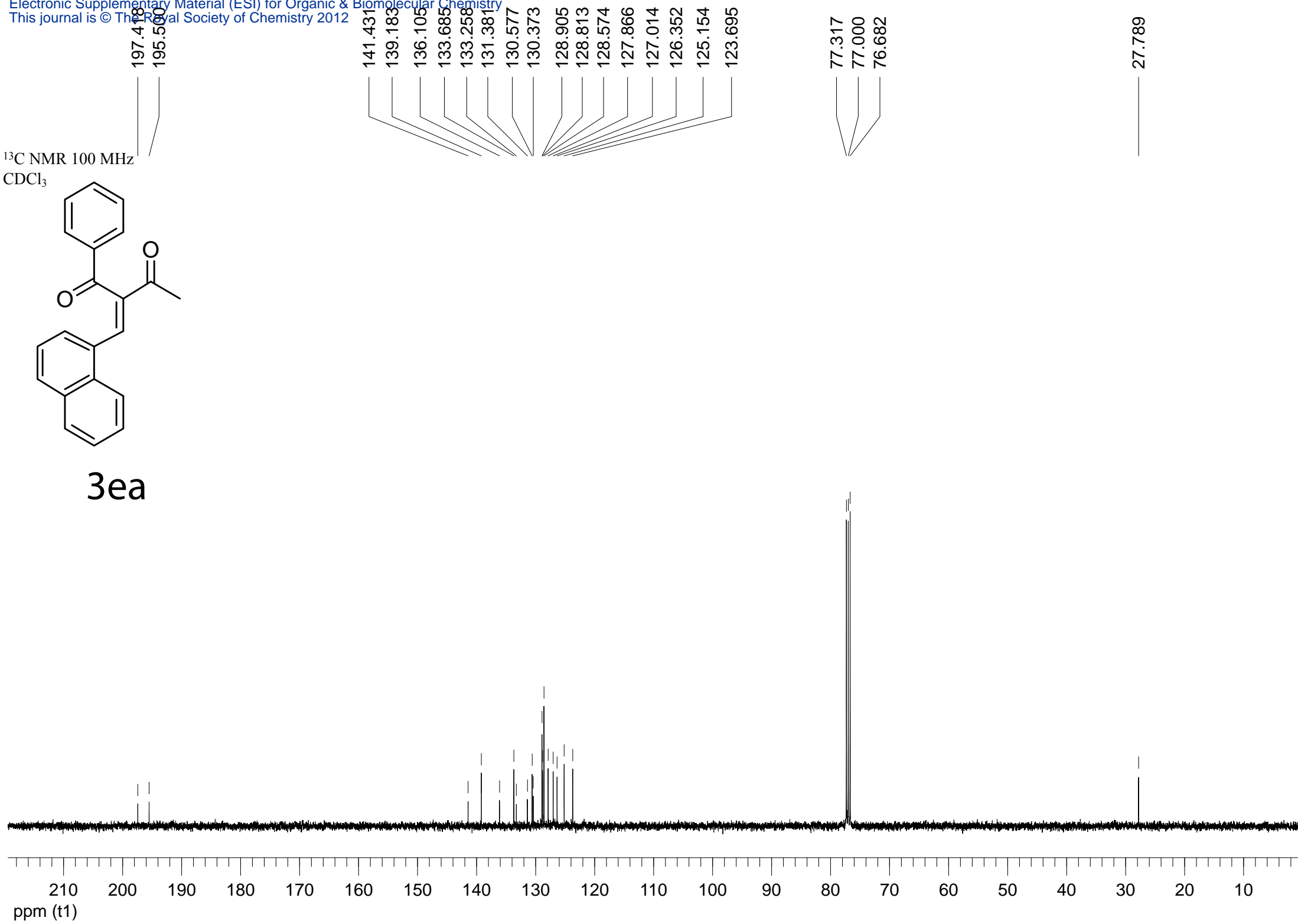
2.475

0.000000

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$

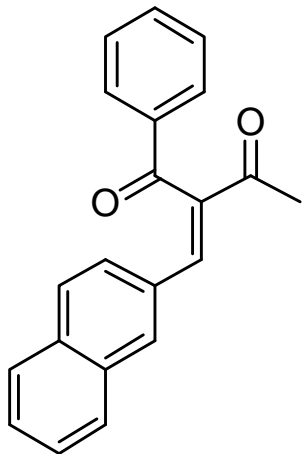


3ea

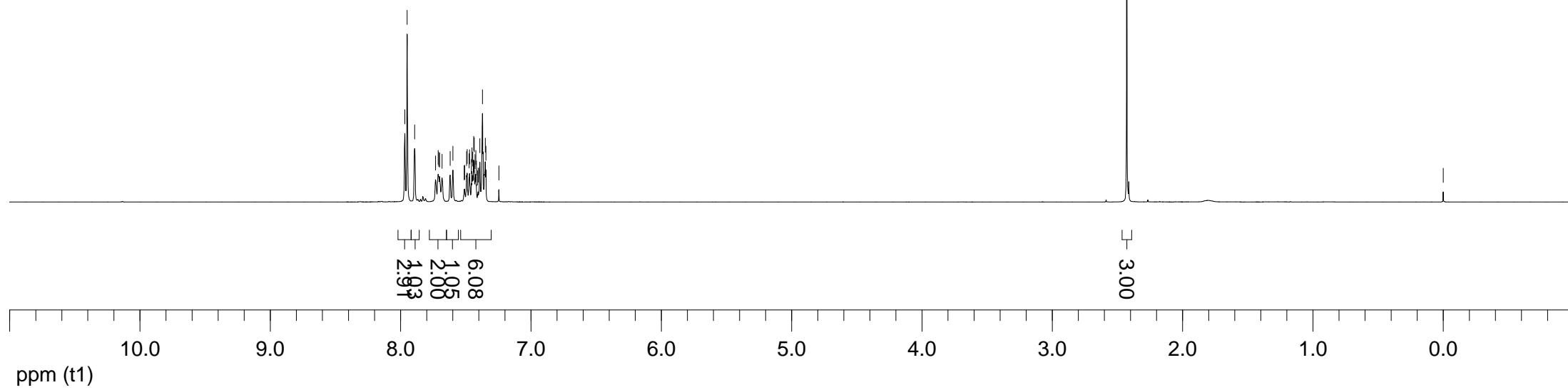


7.968  
7.950  
7.892  
7.732  
7.712  
7.708  
7.700  
7.682  
7.620  
7.599  
7.512  
7.508  
7.494  
7.489  
7.475  
7.471  
7.457  
7.453  
7.446  
7.440  
7.436  
7.429  
7.423  
7.419  
7.409  
7.405  
7.391  
7.372  
7.350  
7.345  
7.246

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

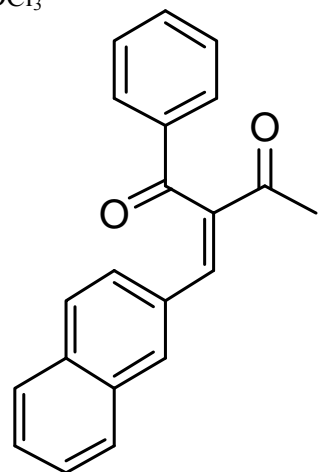


3fa



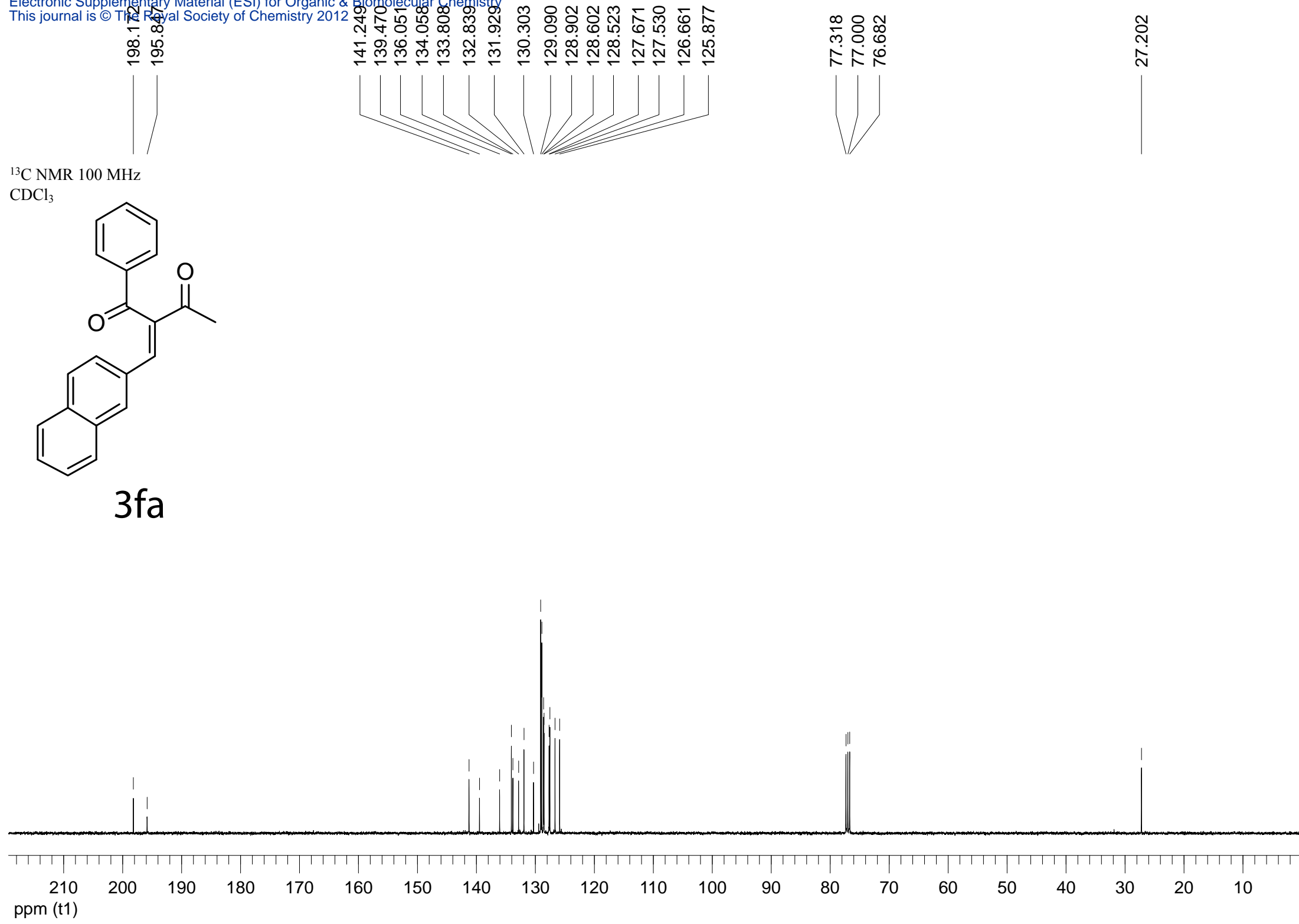
2.428

0.000000



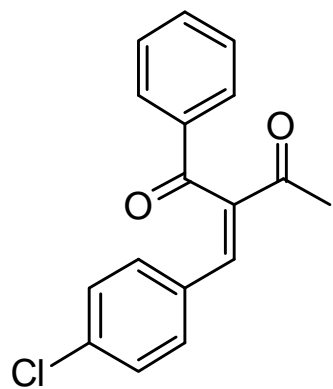
3fa

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$





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

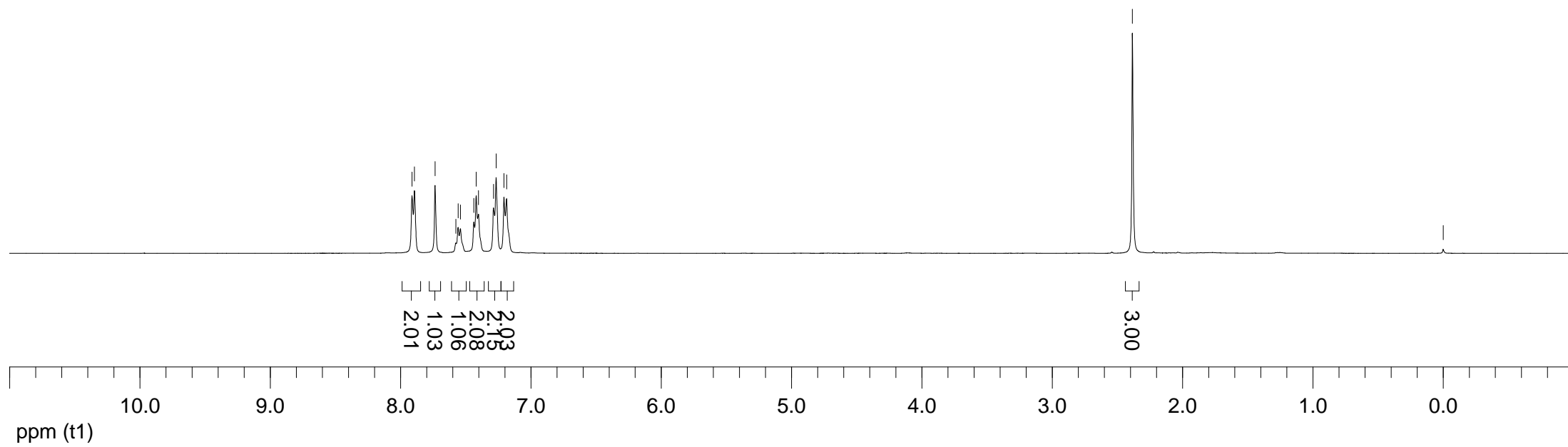


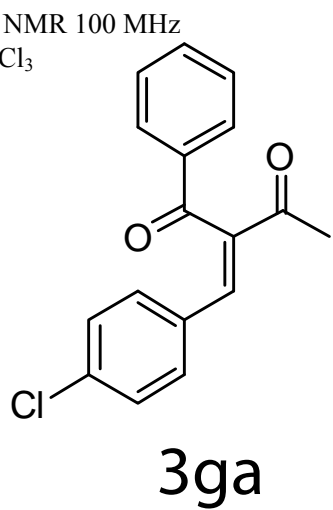
3ga

7.912  
7.893  
7.736  
7.576  
7.559  
7.541  
7.438  
7.420  
7.402  
7.287  
7.267  
7.206  
7.186

2.385

0.0000000



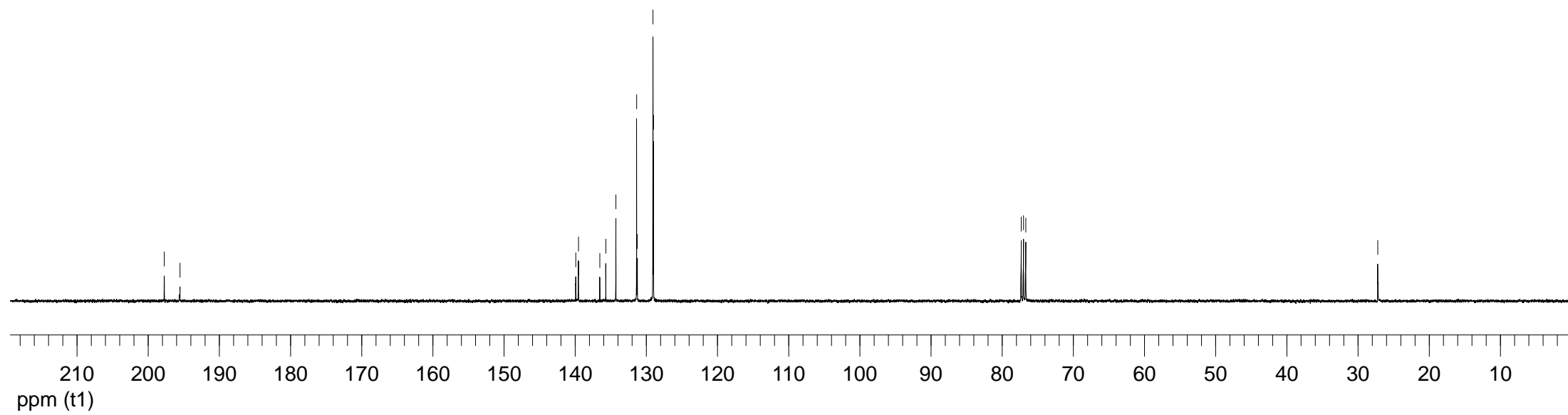


197.731  
195.534

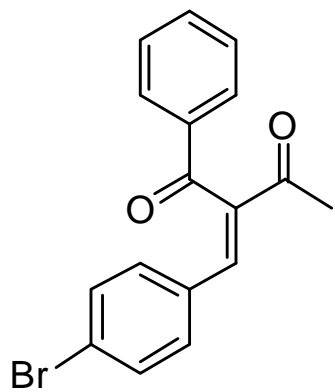
139.908  
139.526  
136.540  
135.689  
134.279  
131.363  
131.259  
129.069  
128.990

77.318  
77.000  
76.682

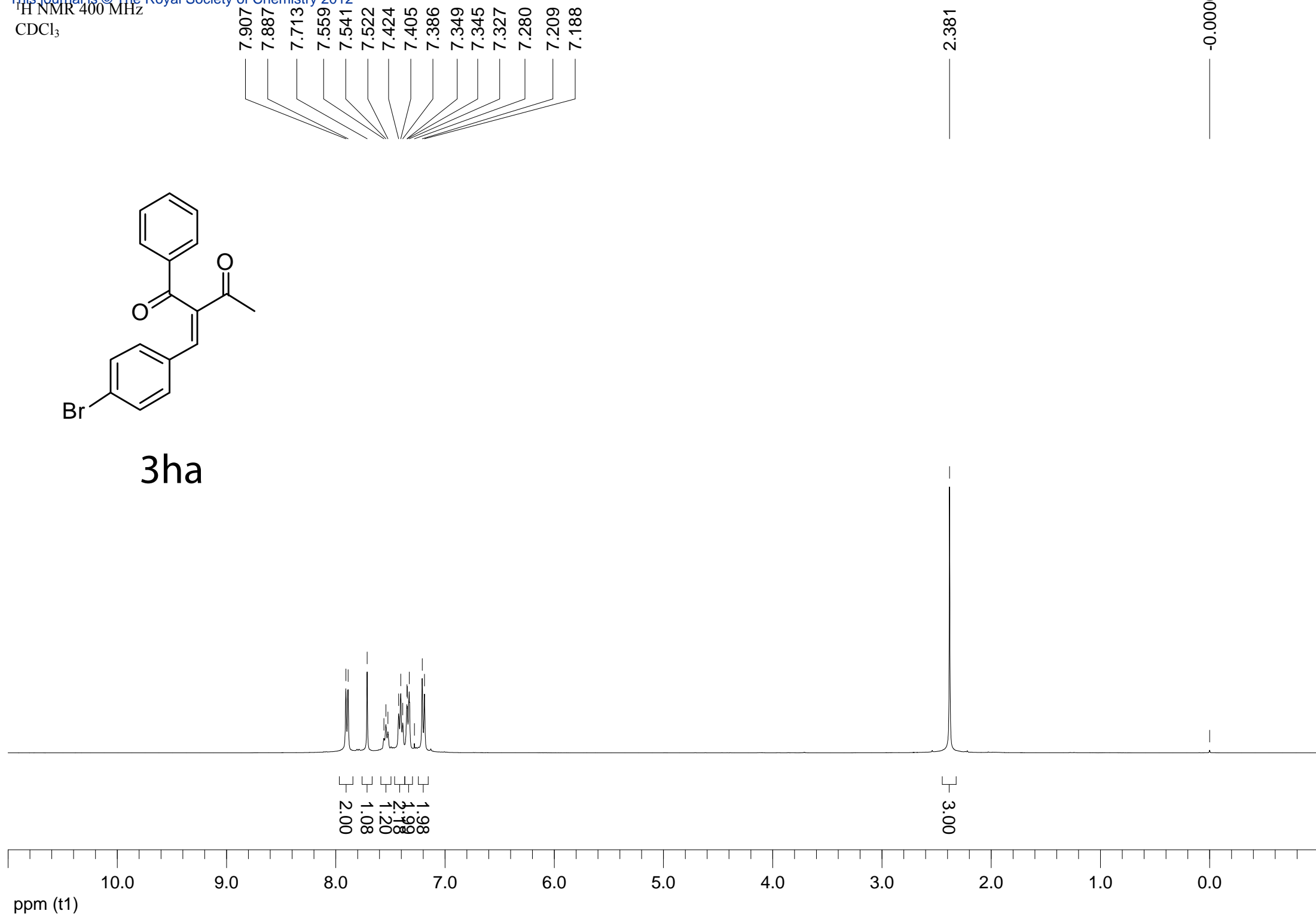
27.229

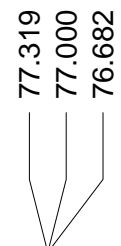
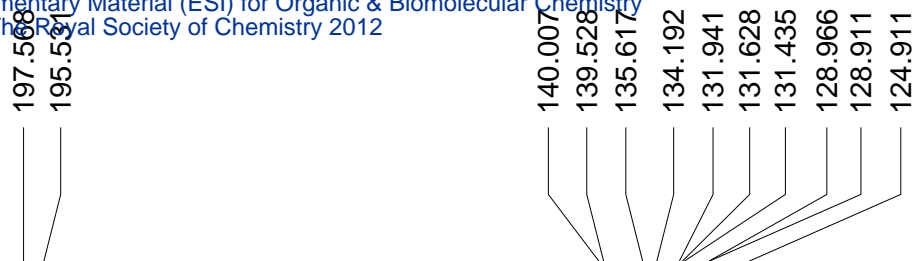


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

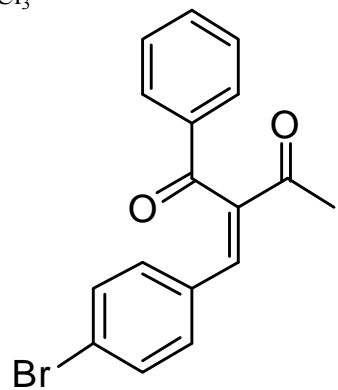


3ha

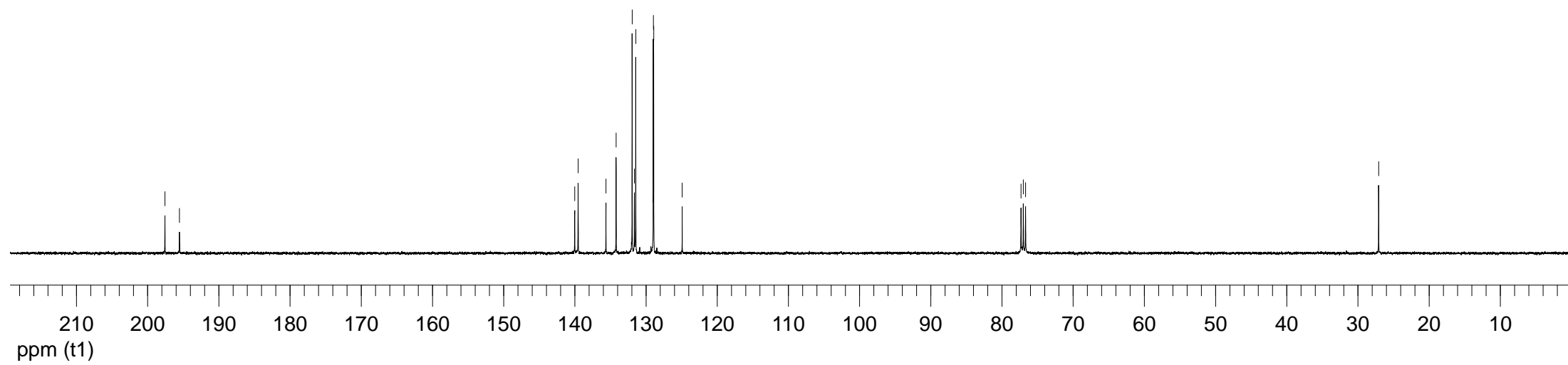


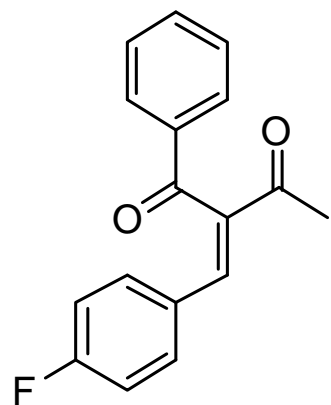


<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



3ha



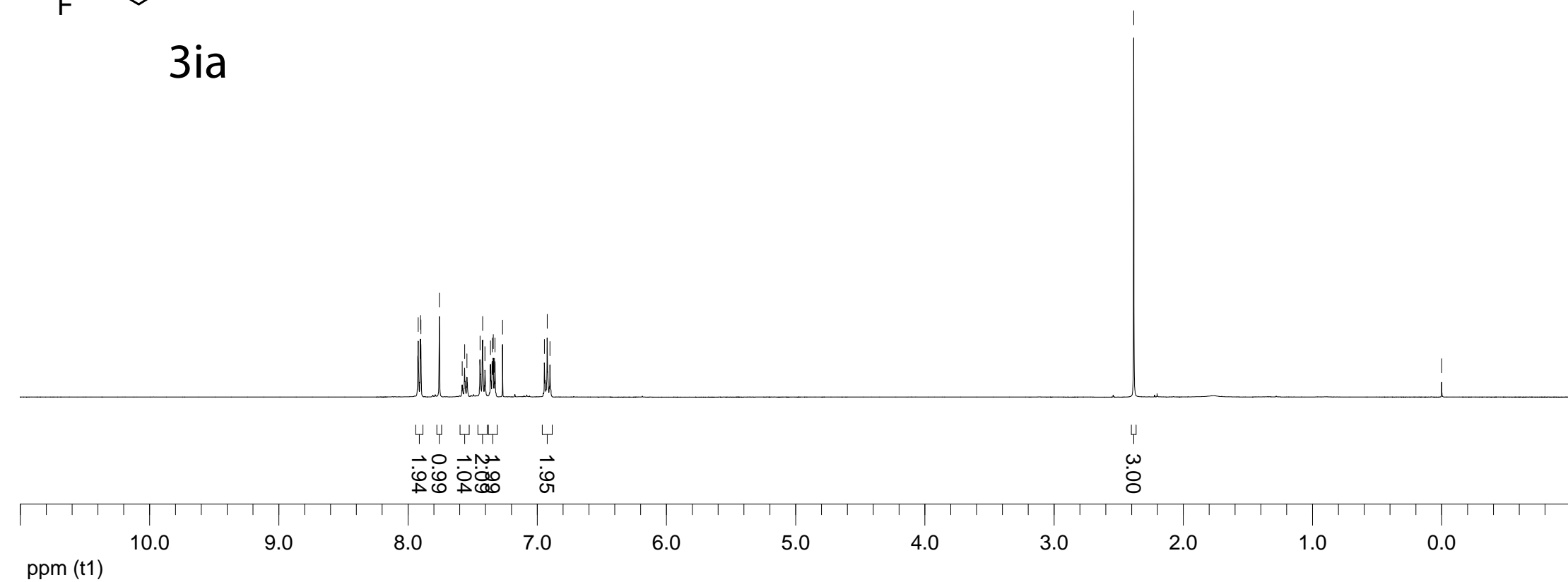


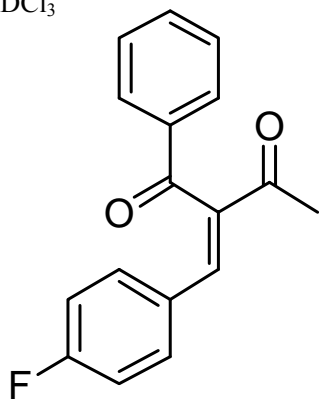
3ia

7.921  
7.903  
7.900  
7.757  
7.580  
7.562  
7.543  
7.442  
7.422  
7.404  
7.362  
7.348  
7.340  
7.332  
7.327  
7.268  
6.944  
6.922  
6.901

2.384

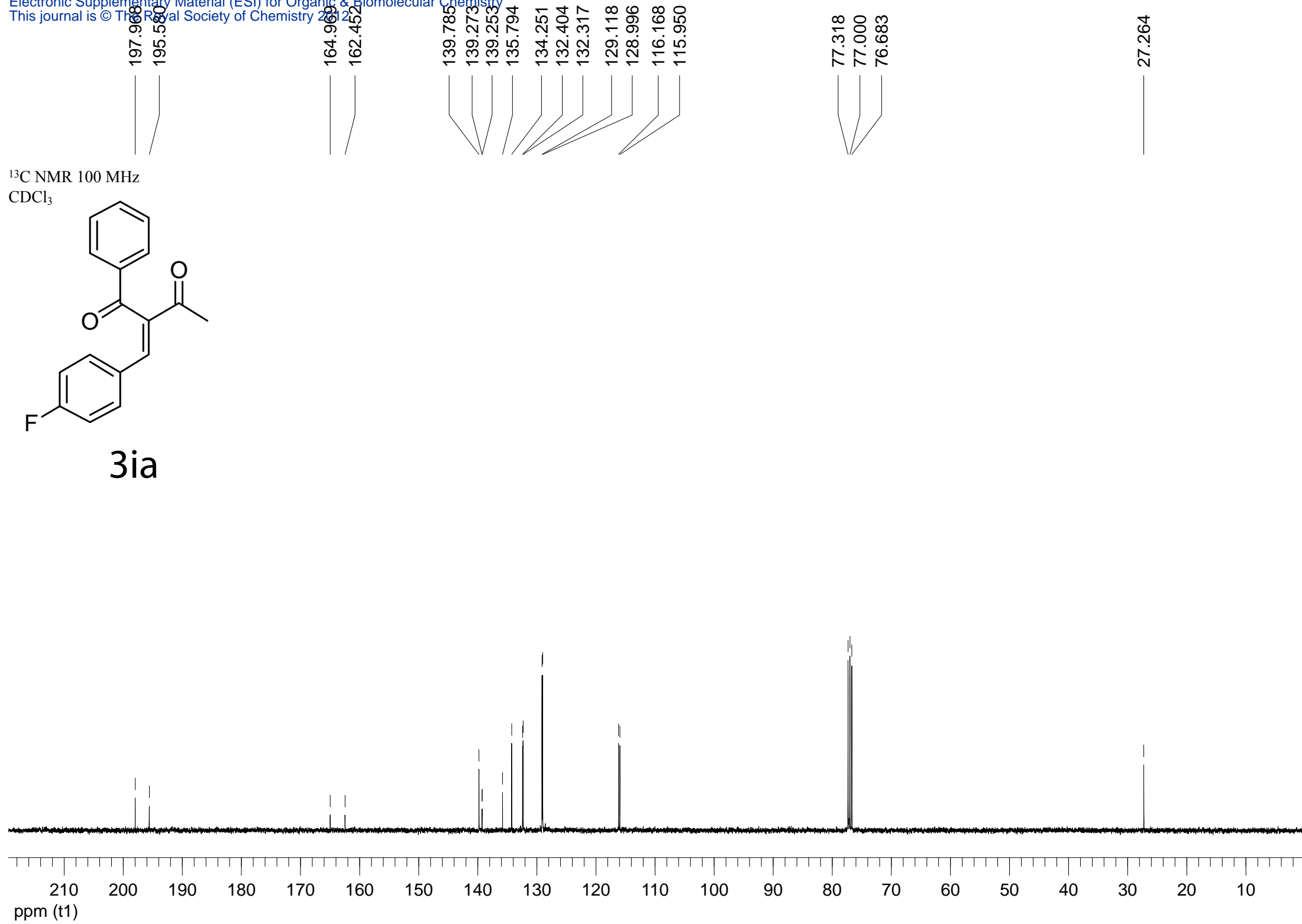
-0.000000



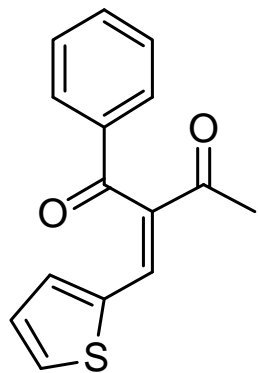


**3ia**

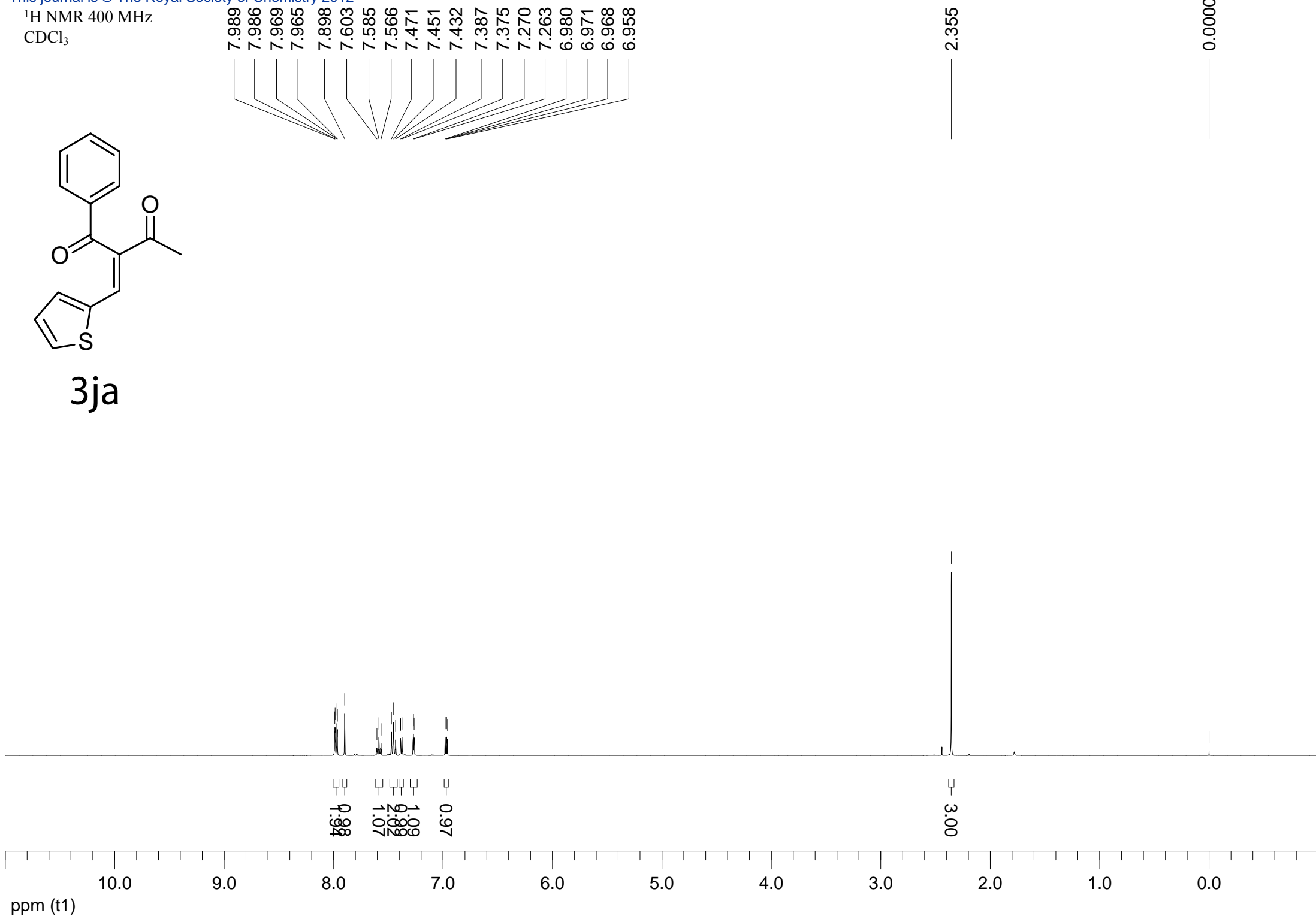
$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



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



3ja



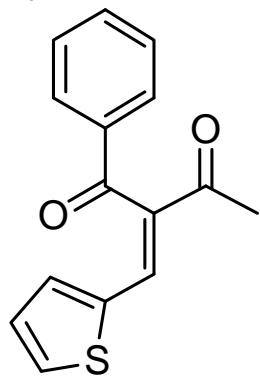
197.694  
195.233

136.492  
136.184  
135.877  
134.211  
133.213  
131.962  
129.164  
128.938  
127.986

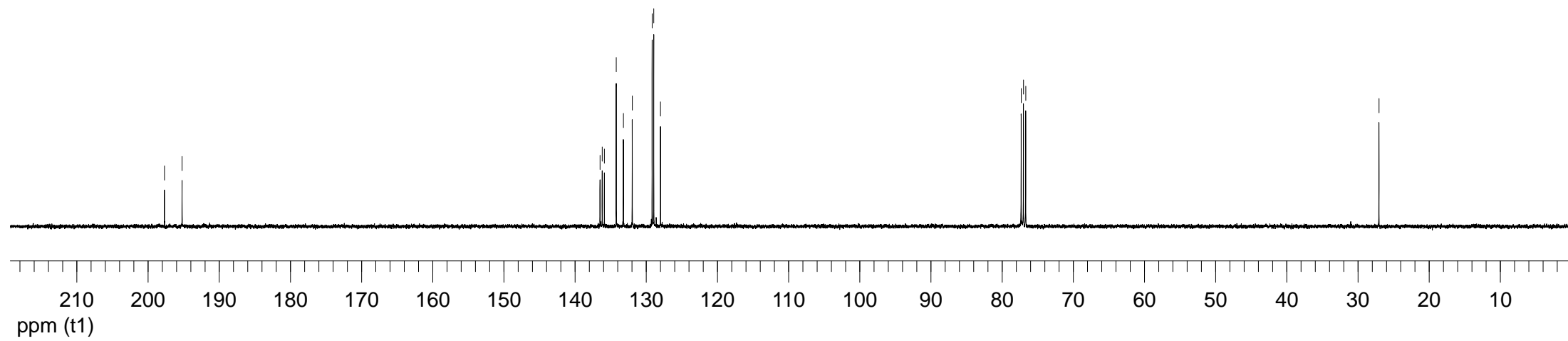
77.318  
77.000  
76.682

27.047

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

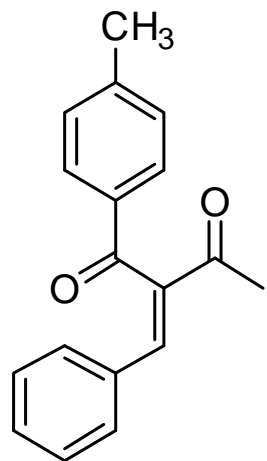


3ja





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

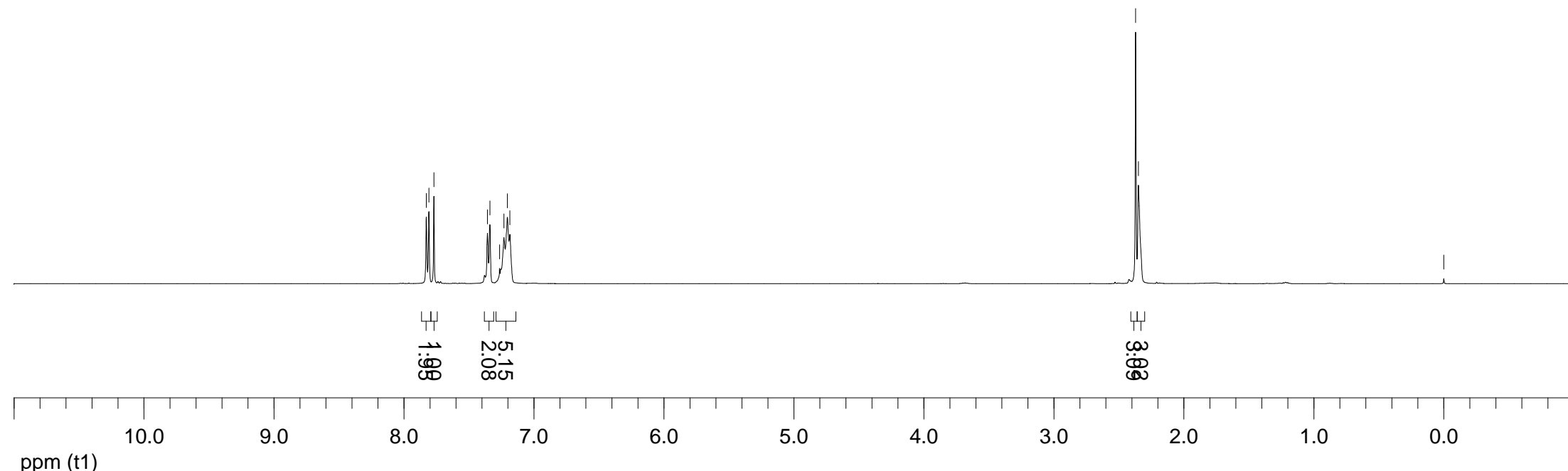


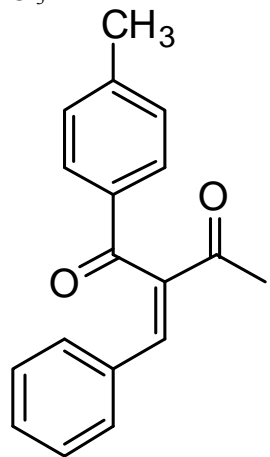
3ab

7.828  
7.808  
7.769  
7.357  
7.338  
7.264  
7.230  
7.204  
7.185

2.371  
2.349

0.00000





3ab

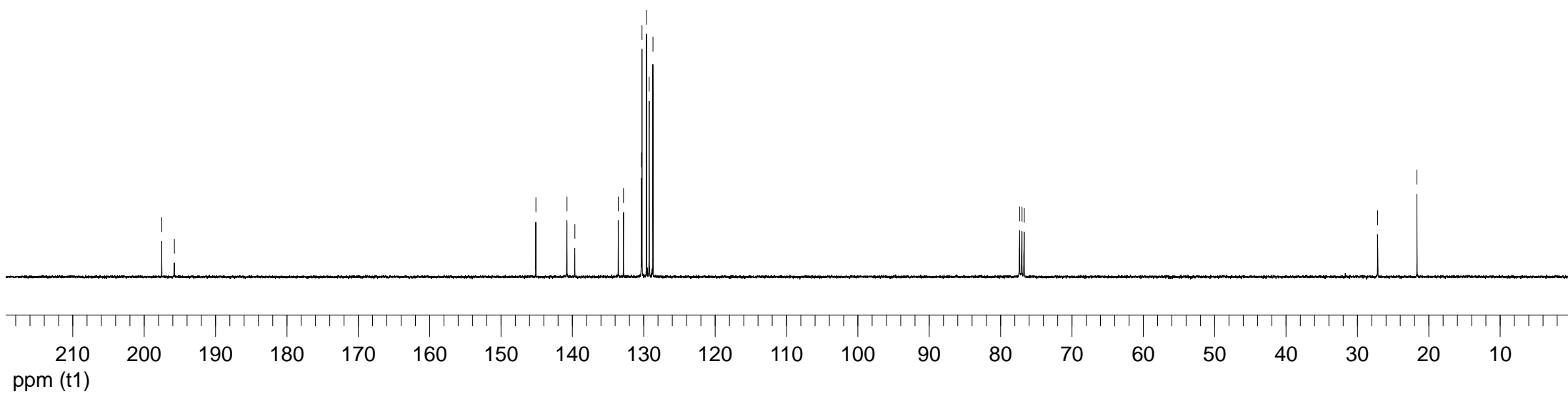
<sup>1</sup>H NMR 100 MHz  
CDCl<sub>3</sub>

197.522  
195.761

145.110  
140.756  
139.651  
133.560  
132.838  
130.331  
130.234  
129.607  
129.228  
128.700

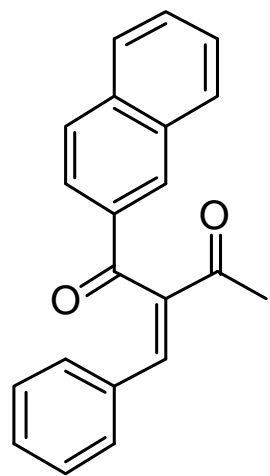
77.319  
77.000  
76.683

27.165  
21.644

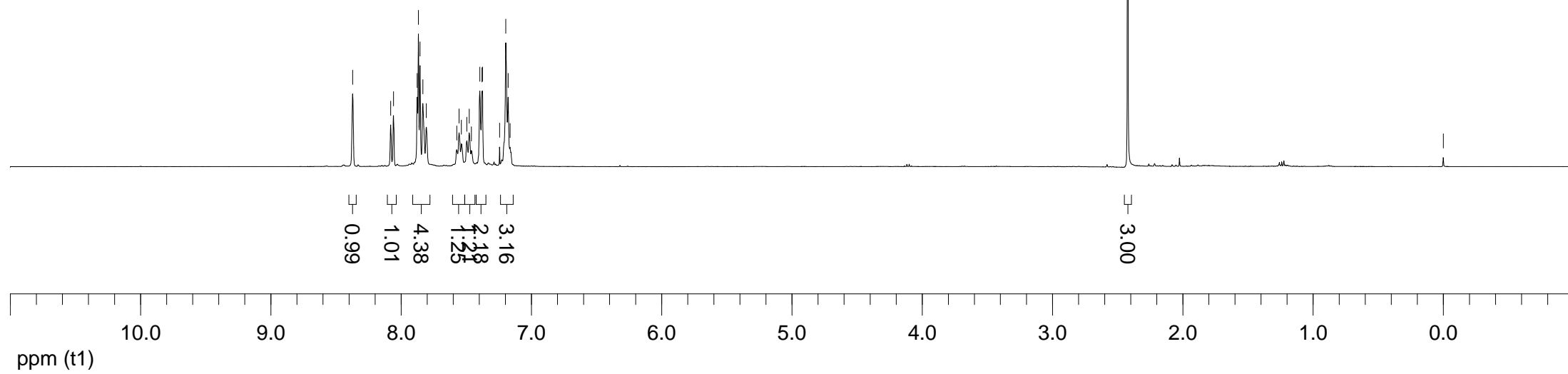


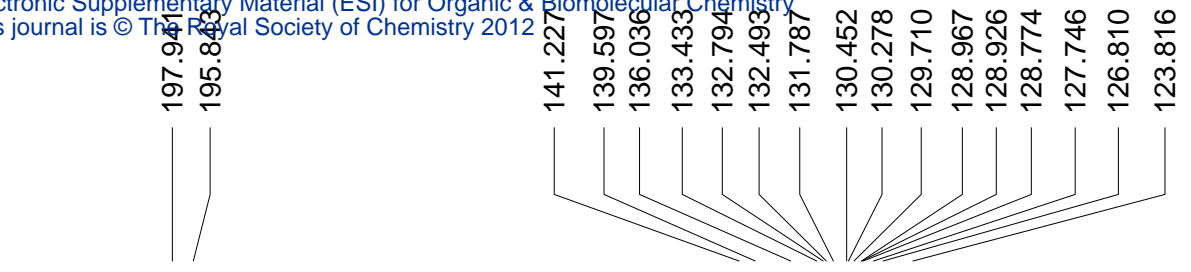
<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

8.372  
8.080  
8.059  
7.876  
7.867  
7.855  
7.833  
7.806  
7.573  
7.555  
7.538  
7.495  
7.477  
7.460  
7.395  
7.379  
7.376  
7.244  
7.196  
7.178  
7.164

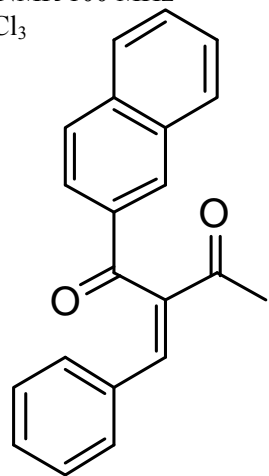


3ac

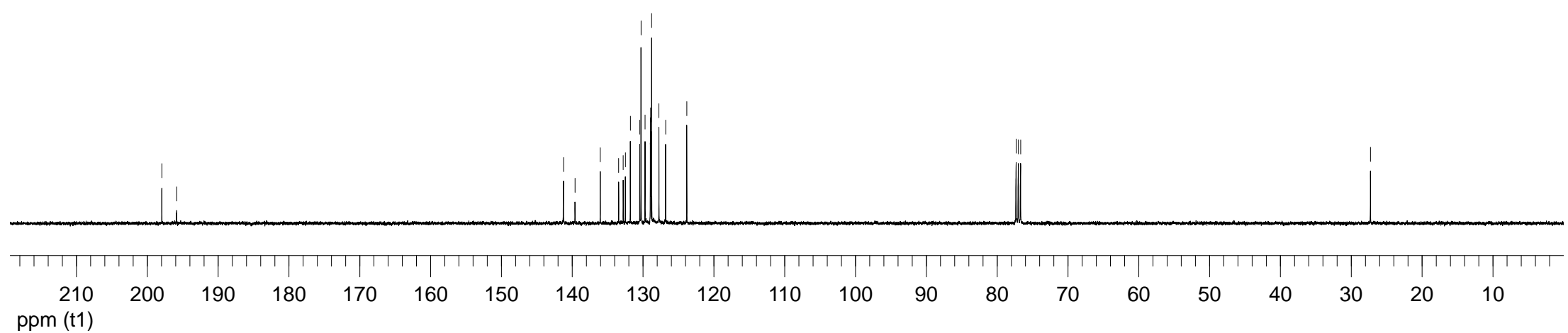


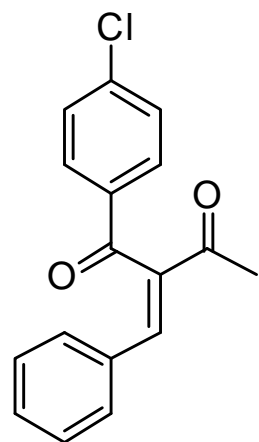


<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

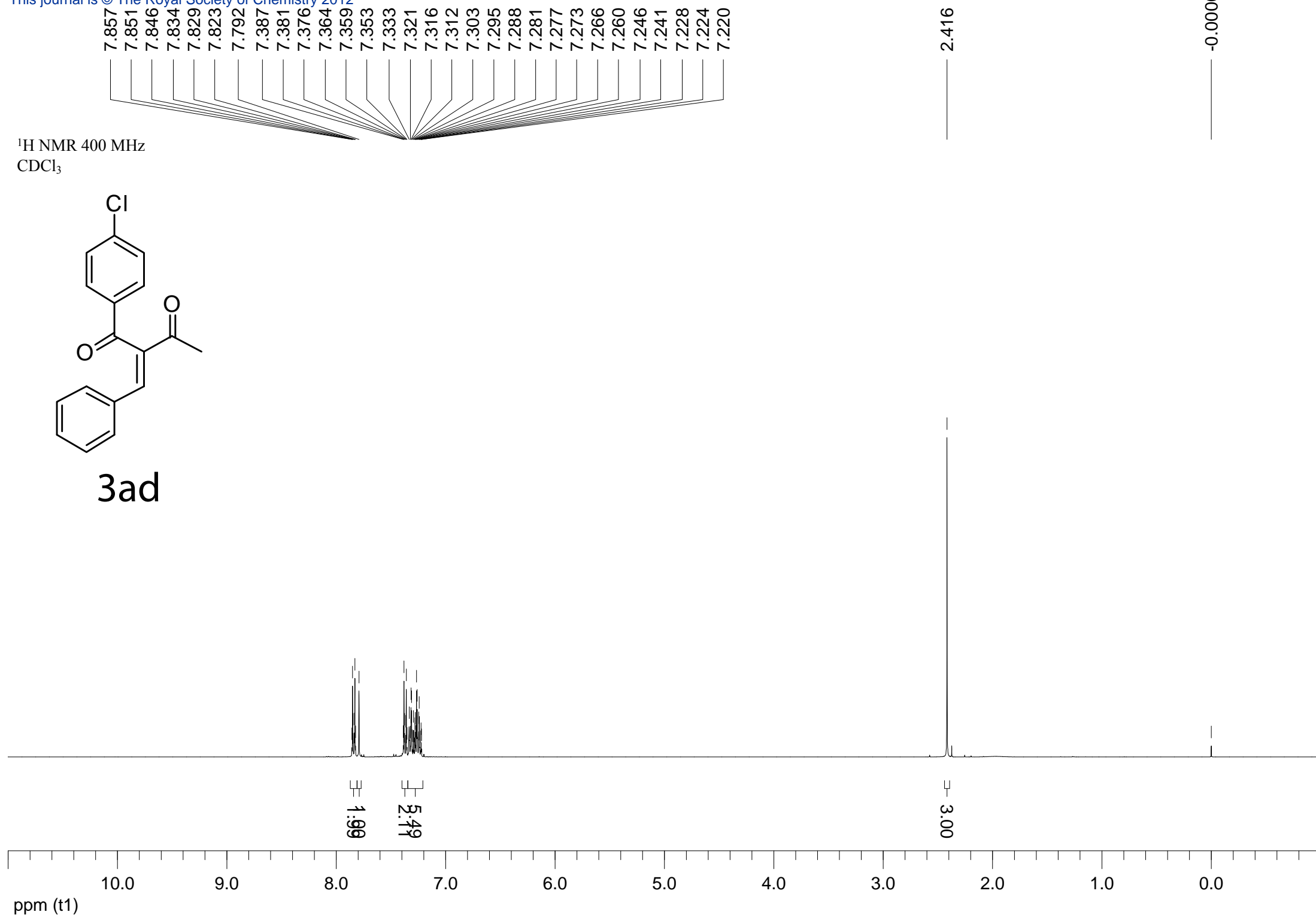


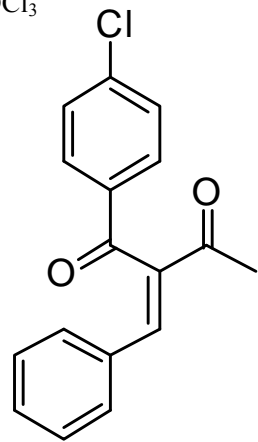
3ac





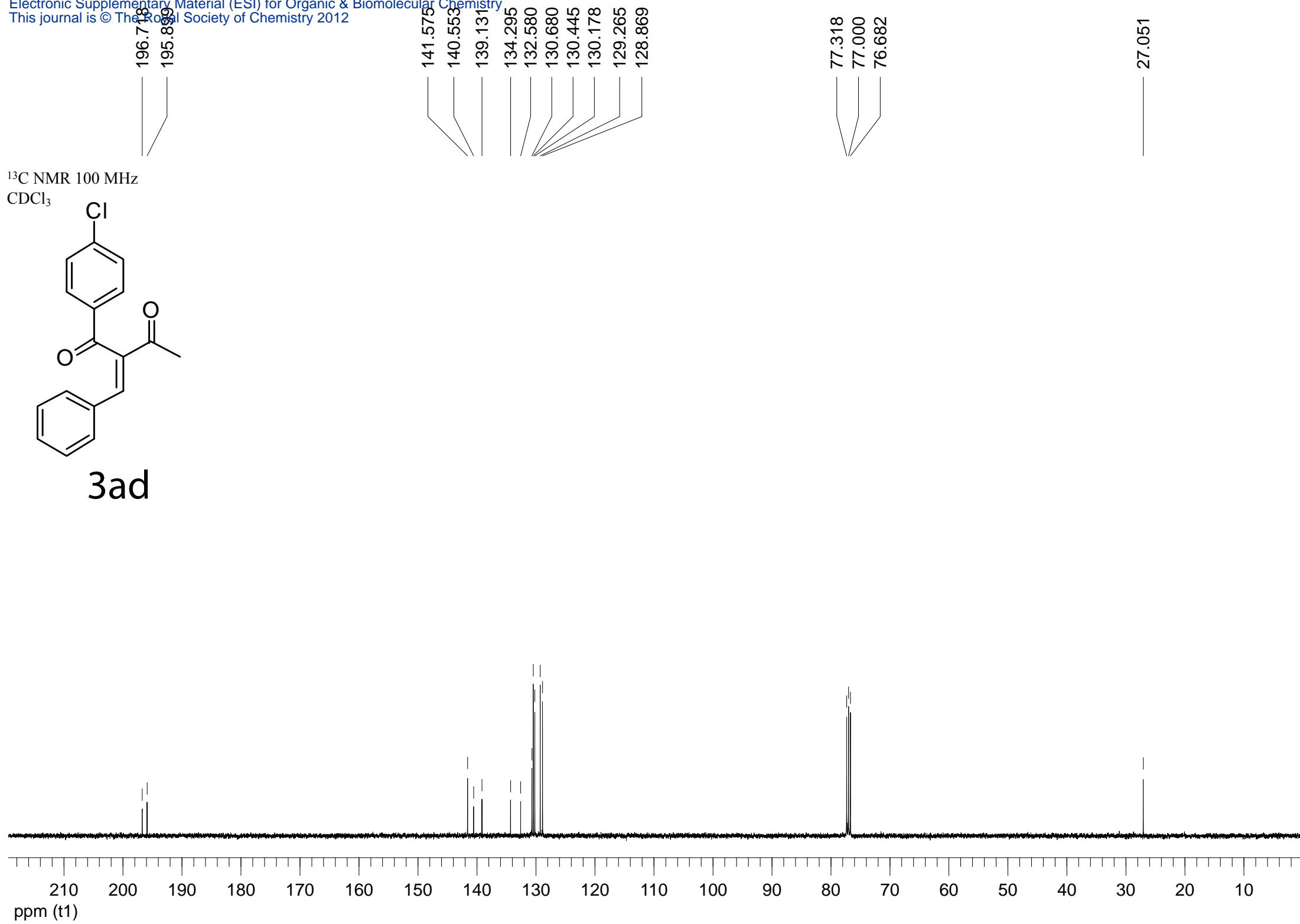
3ad



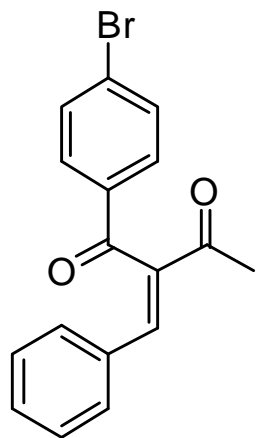


3ad

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



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

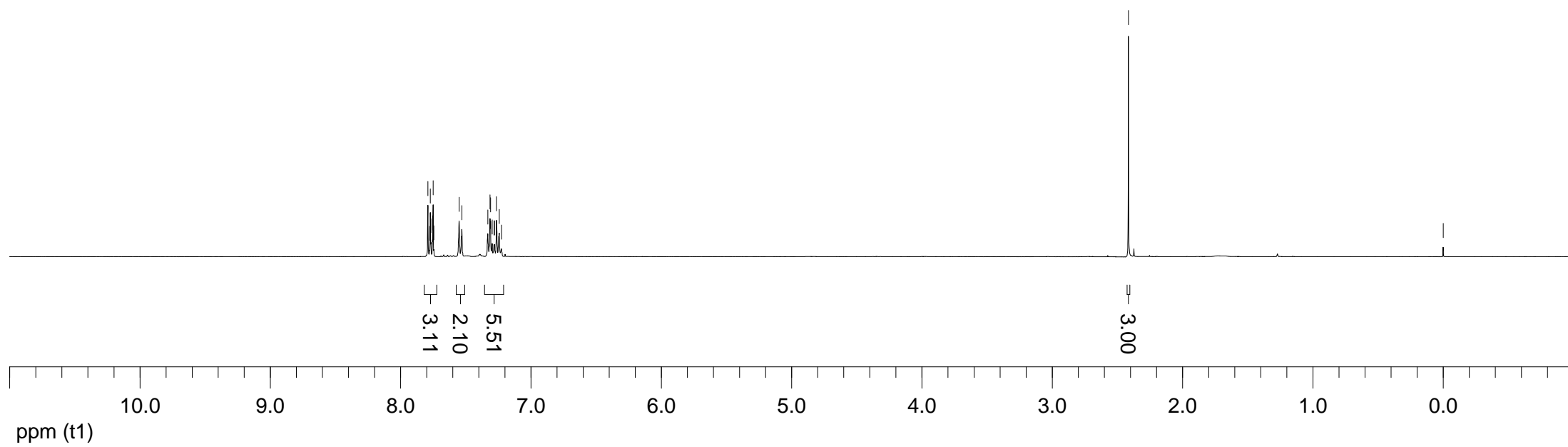


3ae

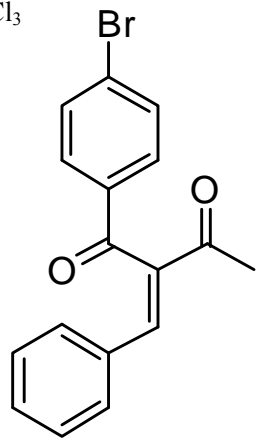
7.791  
7.778  
7.772  
7.767  
7.755  
7.750  
7.745  
7.551  
7.530  
7.331  
7.314  
7.310  
7.297  
7.283  
7.280  
7.265  
7.243  
7.226

2.415

-0.000000



$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



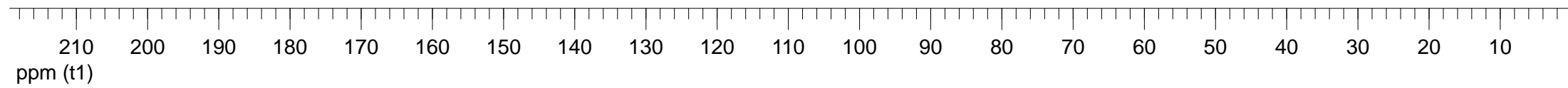
3ae

196.929  
195.898

141.613  
139.077  
134.670  
132.547  
132.247  
130.694  
130.497  
130.170  
129.422  
128.873

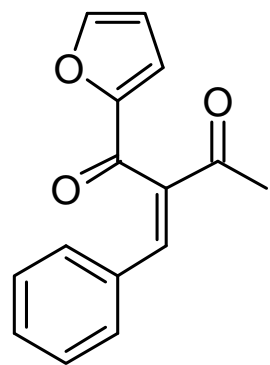
77.317  
77.000  
76.682

27.036

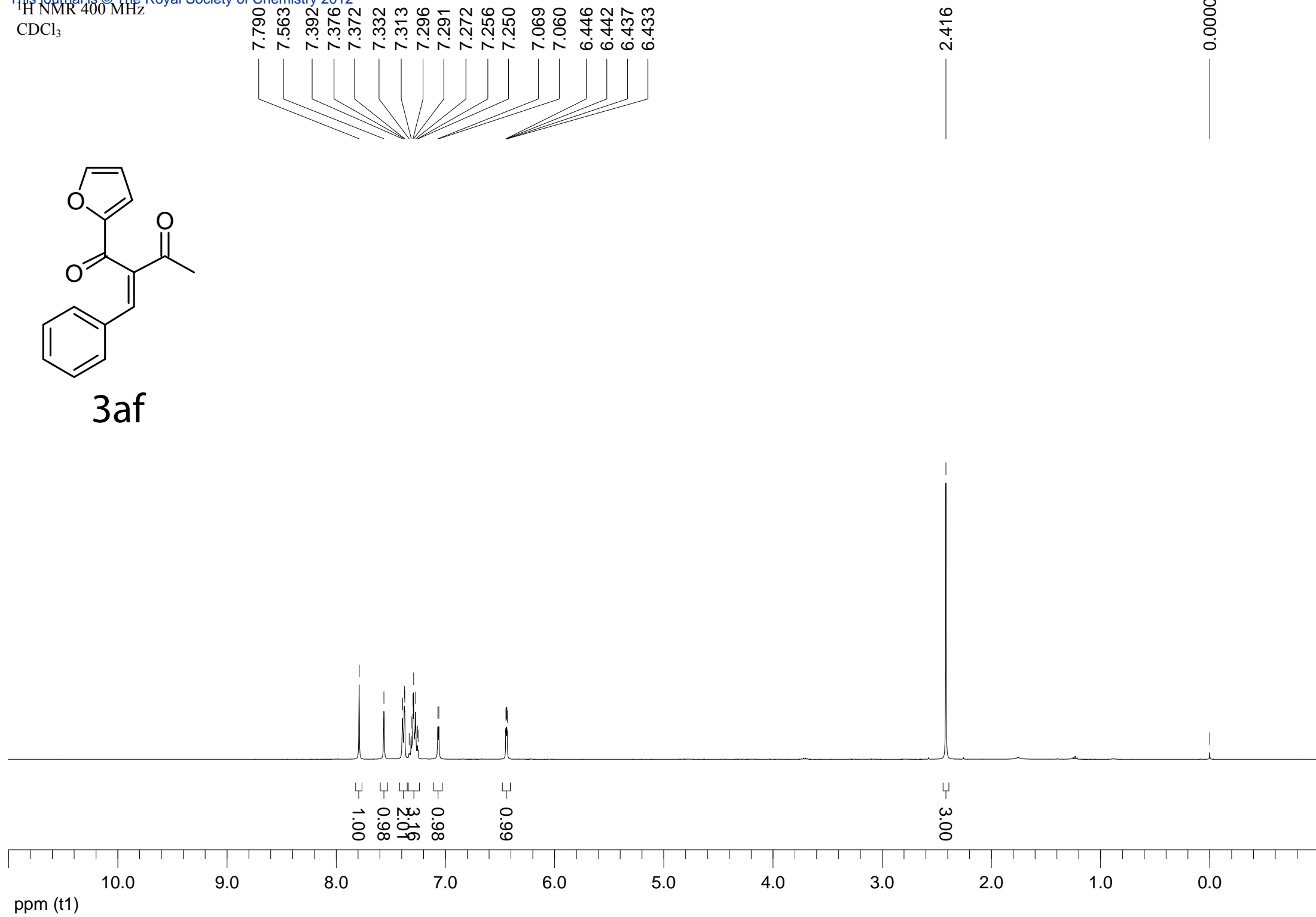


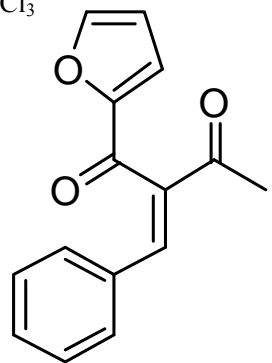


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>



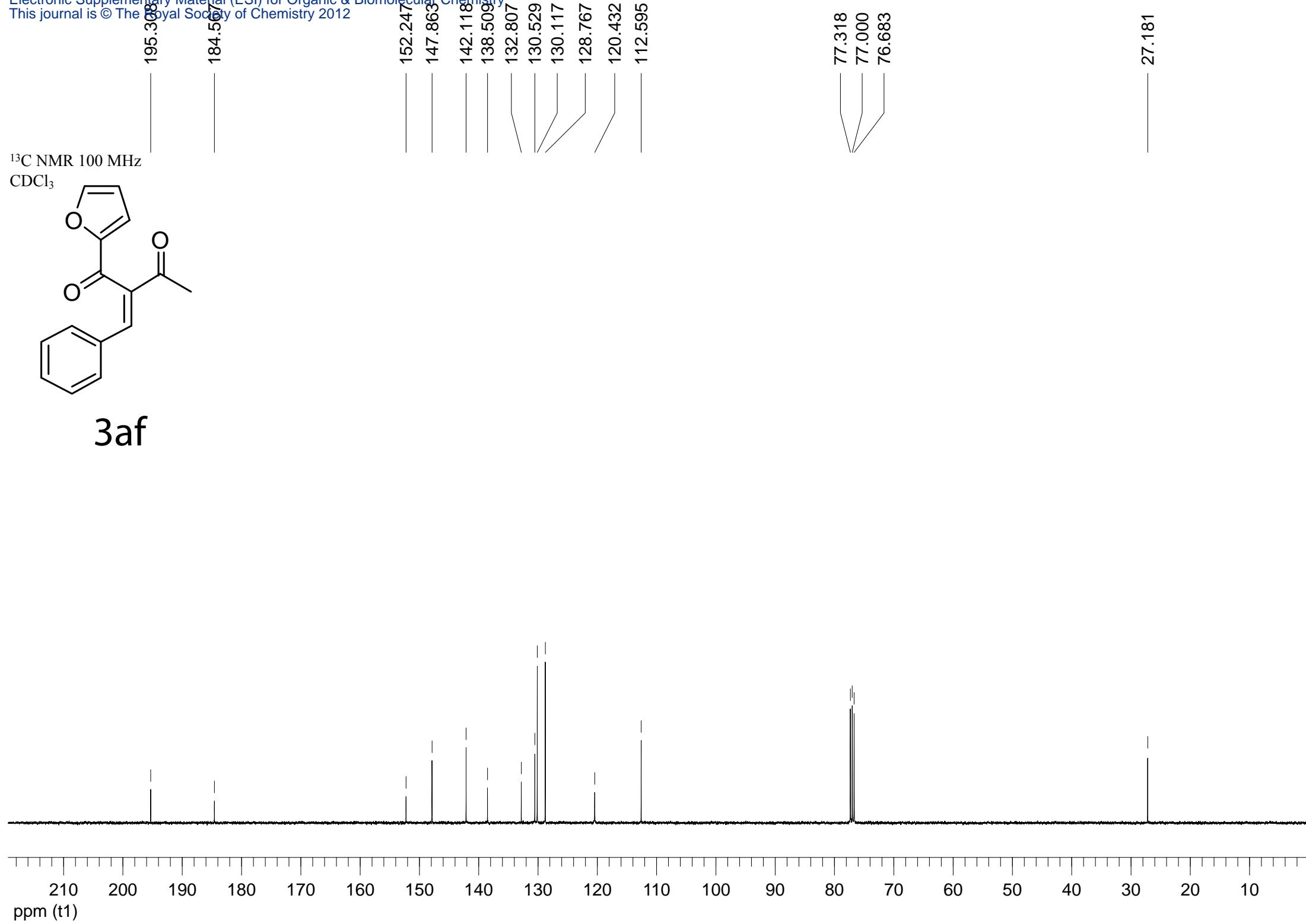
3af



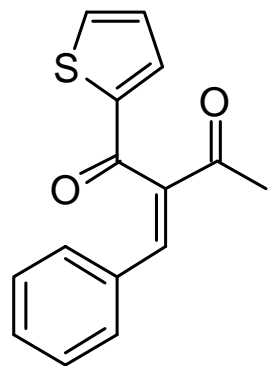


3af

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



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

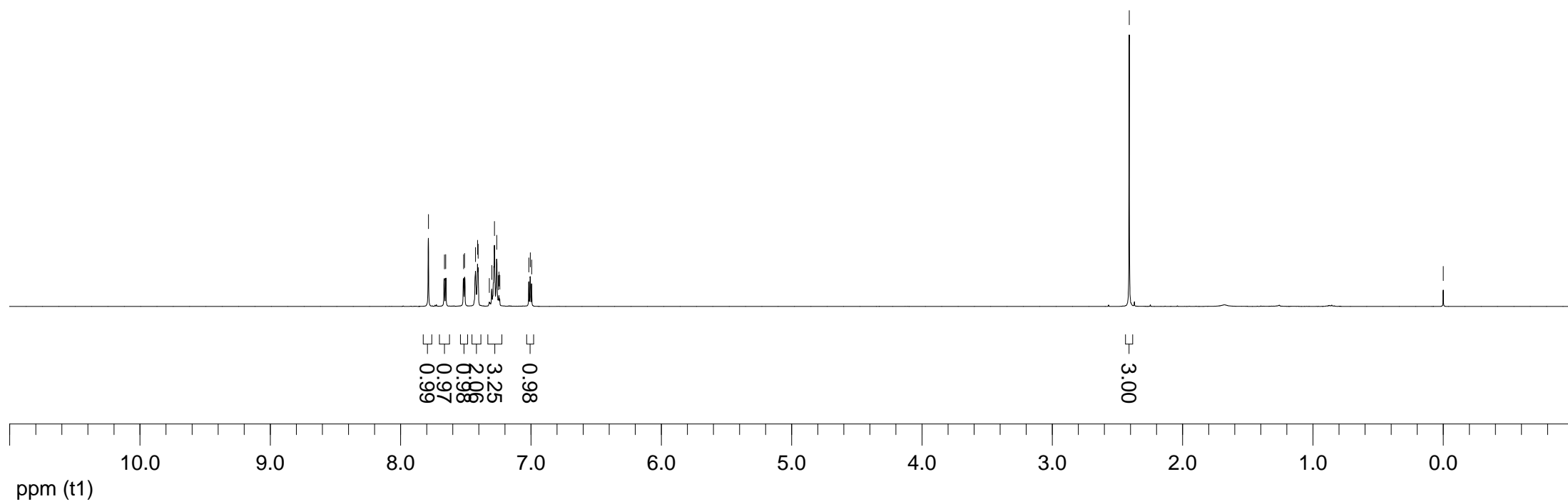


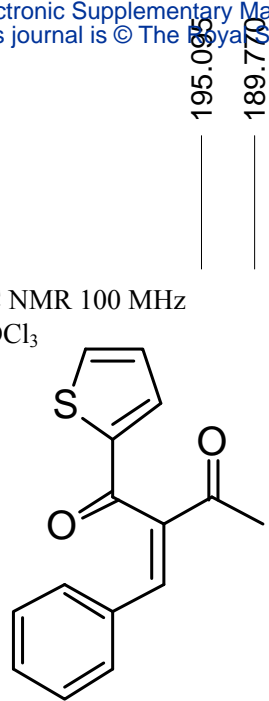
3ag

7.787  
7.664  
7.654  
7.516  
7.509  
7.425  
7.409  
7.405  
7.319  
7.300  
7.280  
7.263  
7.250  
7.245  
7.239  
7.016  
7.005  
6.994

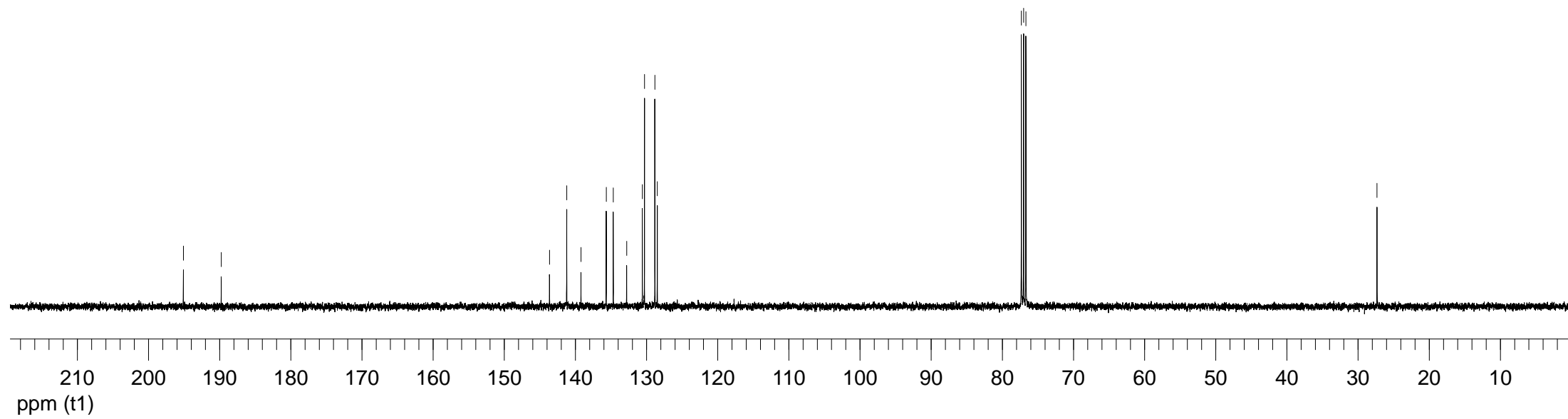
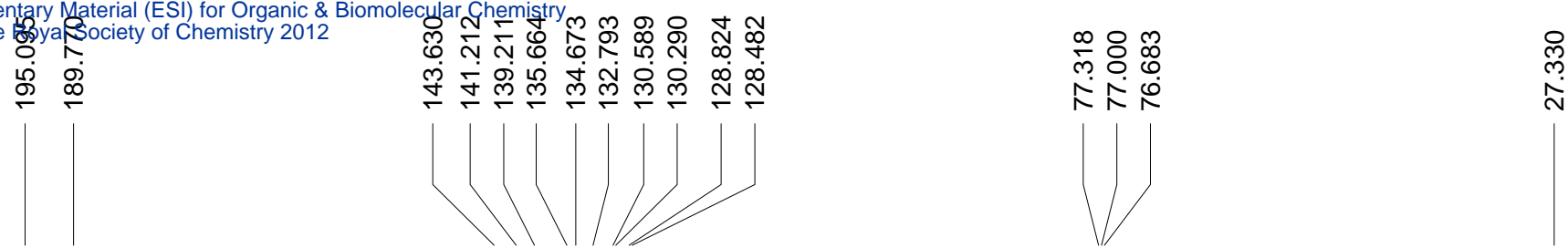
2.409

0.000000

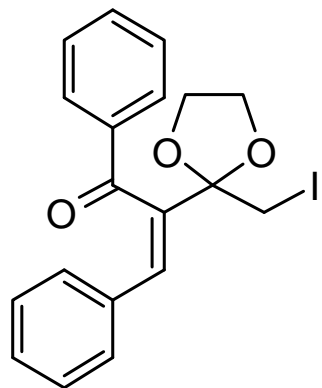




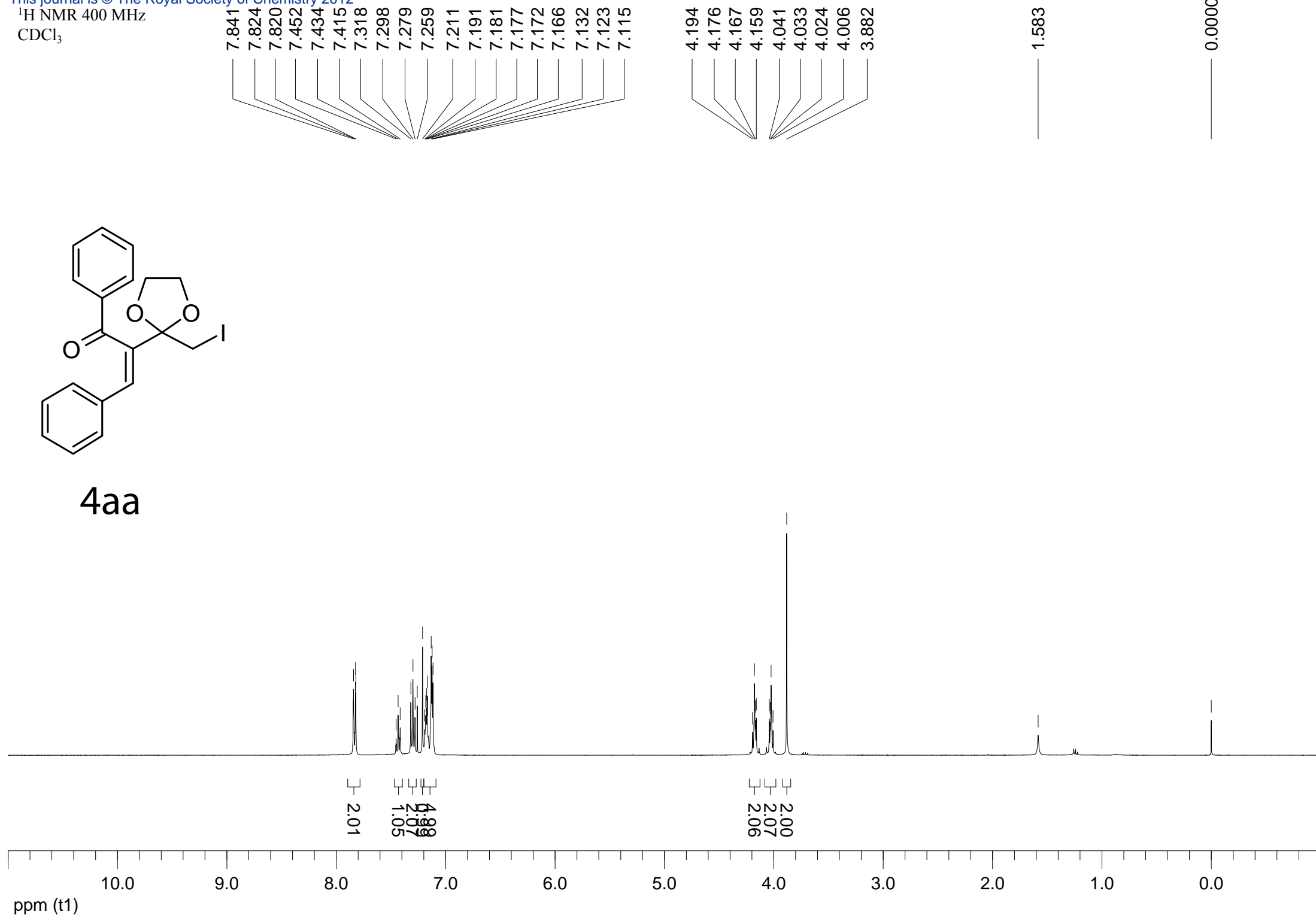
<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

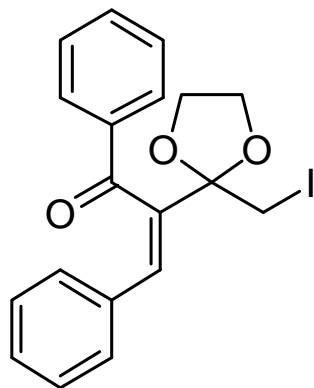


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>



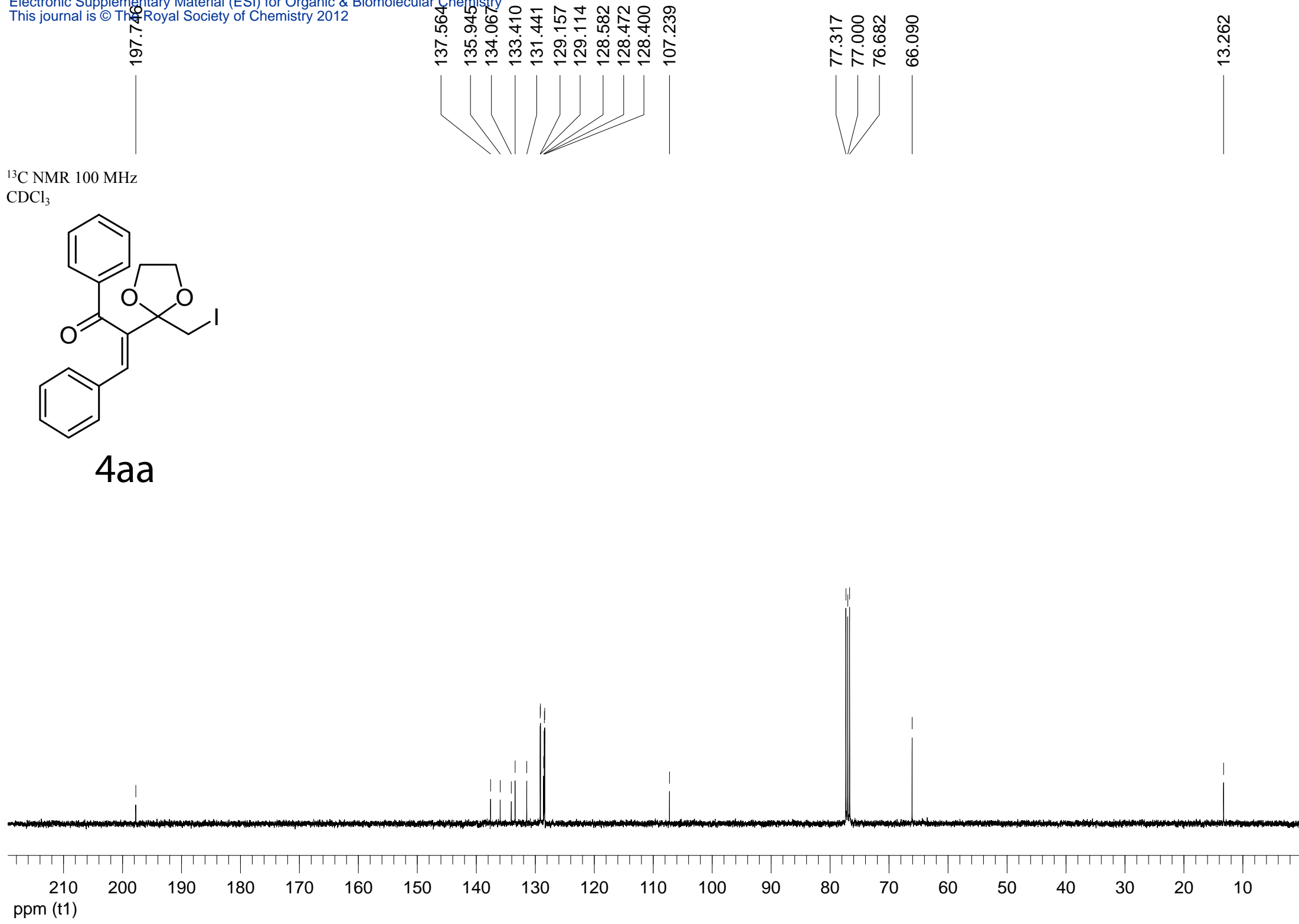
4aa





4aa

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$

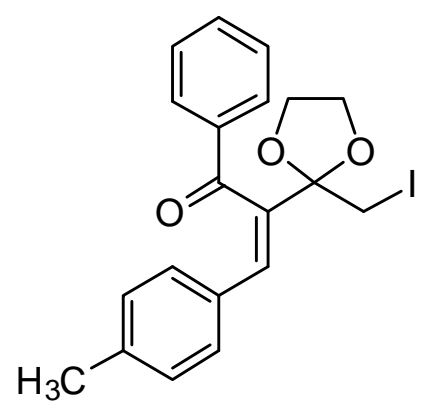


7.855  
7.837  
7.834  
7.455  
7.436  
7.418  
7.323  
7.303  
7.285  
7.253  
7.161  
7.075  
7.055  
6.934  
6.914

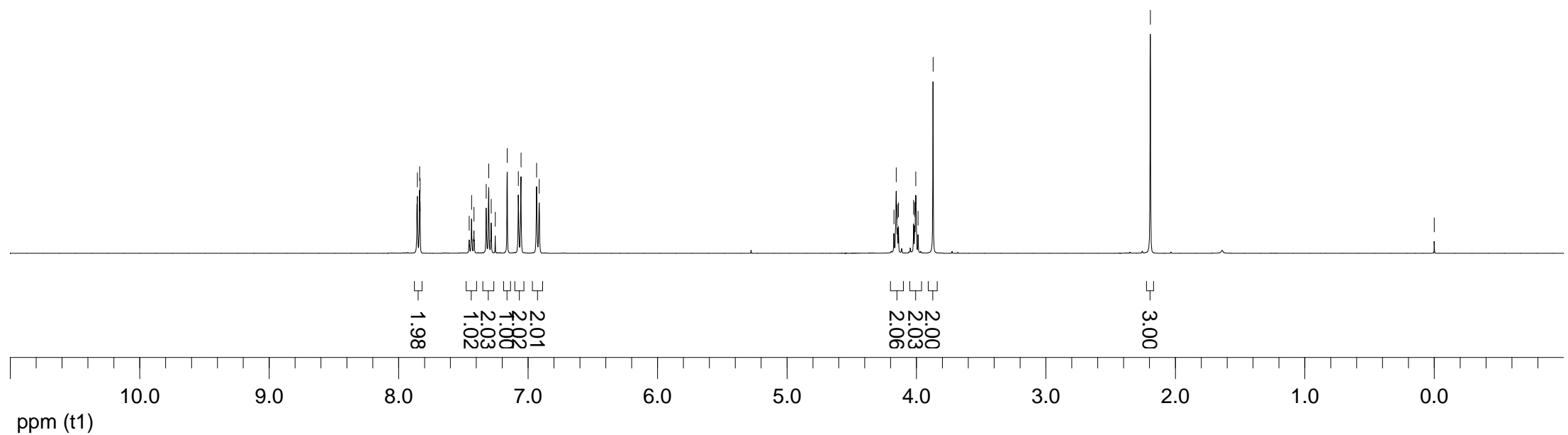
4.173  
4.156  
4.147  
4.139  
4.022  
4.013  
4.004  
3.987  
3.871

2.193

-0.000



4ba



197.928

138.655

136.423

135.988

133.326

131.276

131.145

129.119

128.462

107.208

77.318

77.000

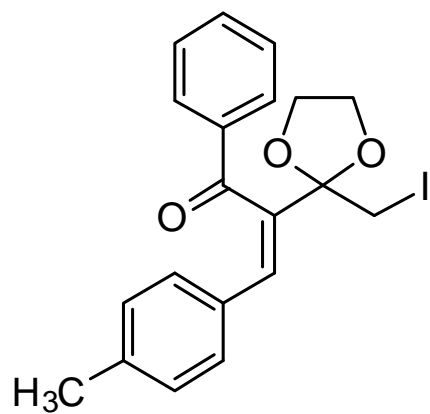
76.682

66.026

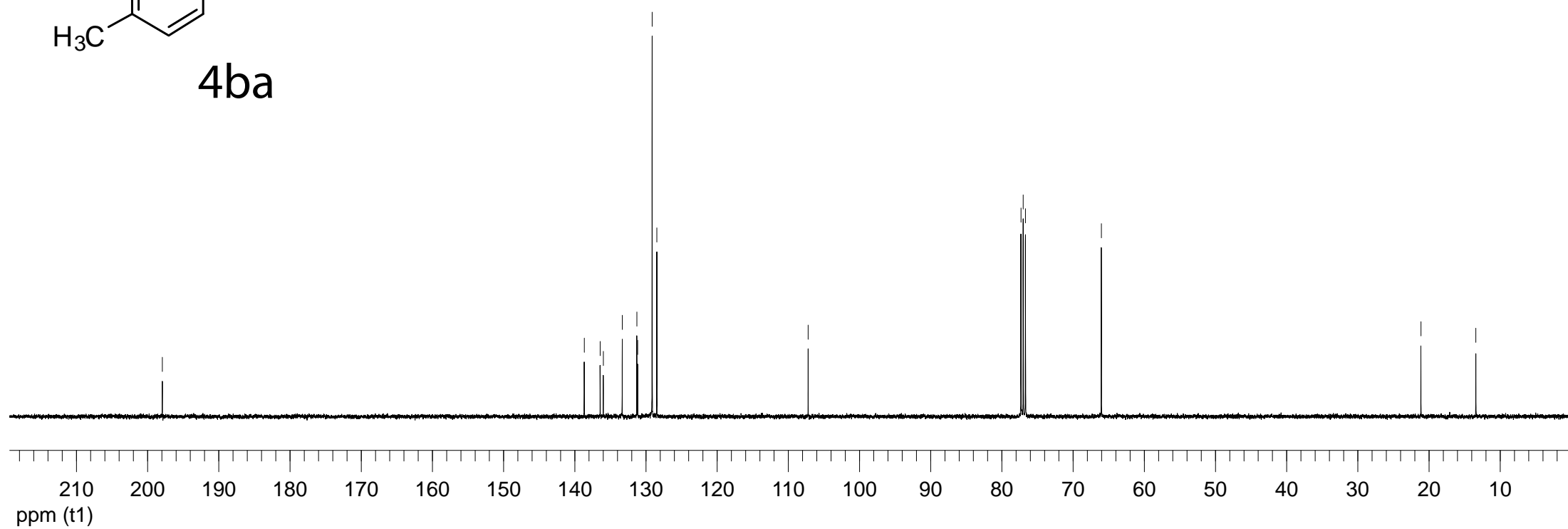
21.128

13.417

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



4ba



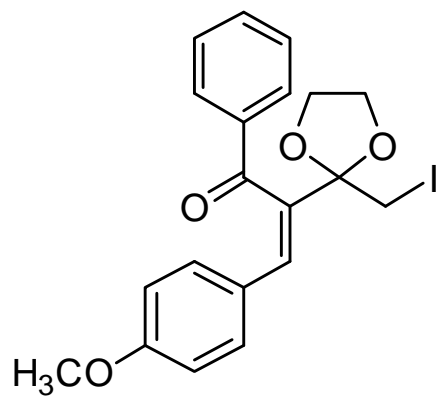


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

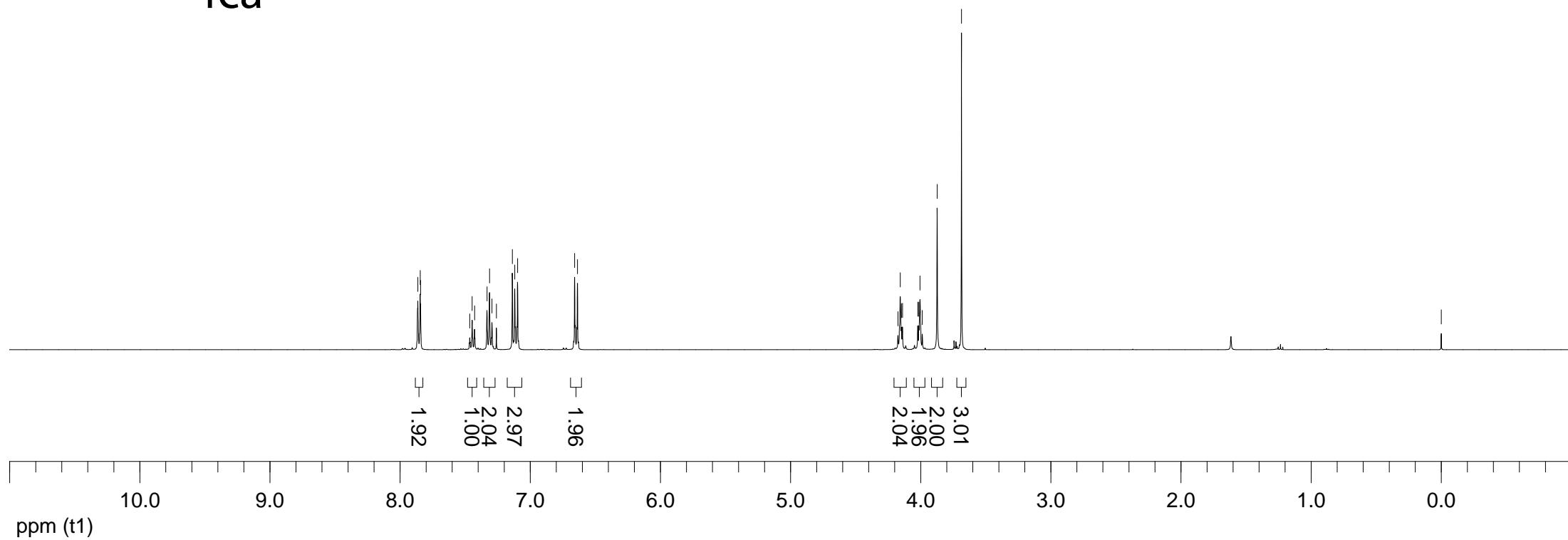
7.863  
7.845  
7.842  
7.465  
7.446  
7.428  
7.332  
7.312  
7.294  
7.260  
7.137  
7.119  
7.097  
6.959  
6.637

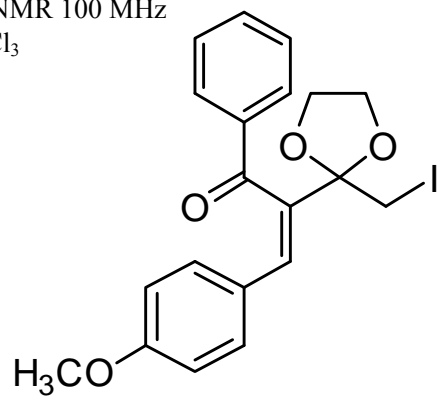
4.174  
4.157  
4.148  
4.140  
4.022  
4.014  
4.005  
3.987  
3.873  
3.686

0.0000000



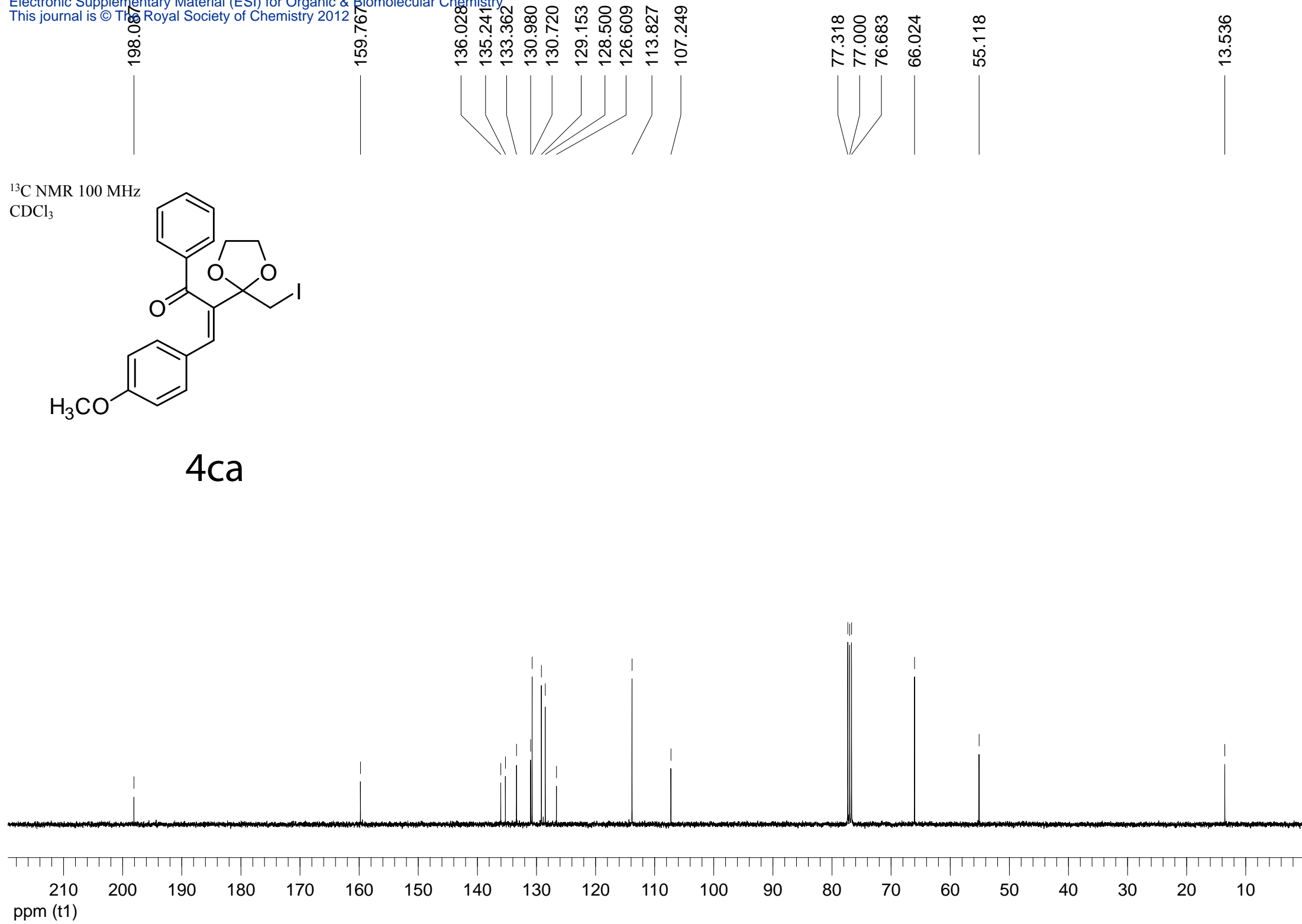
4ca



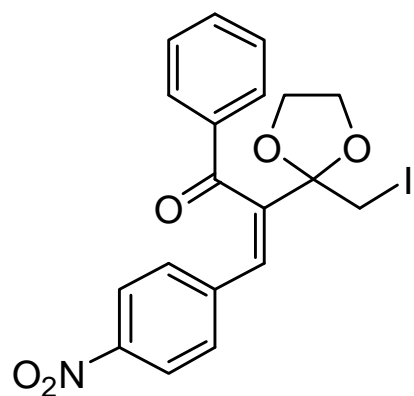


4ca

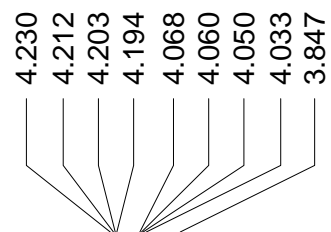
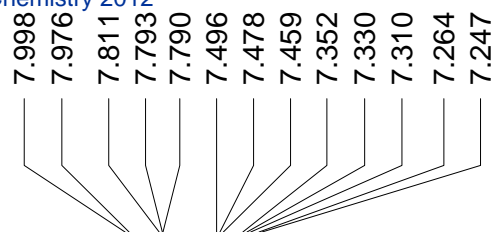
<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



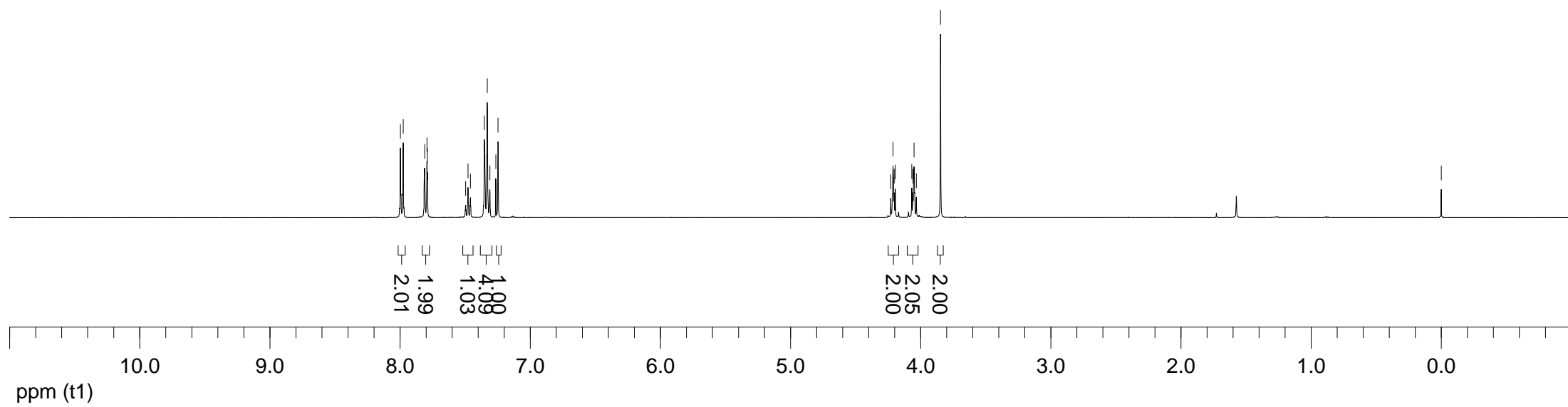
<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

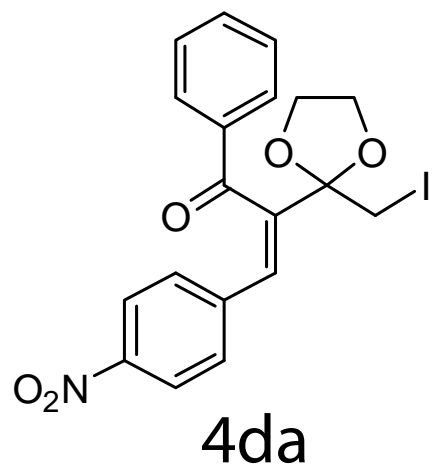


4da



-0.000000





$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$

196.755

147.283

141.968

140.594

135.500

134.042

129.640

129.070

128.817

128.756

123.659

107.173

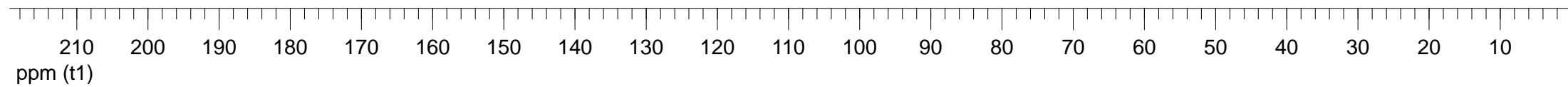
77.317

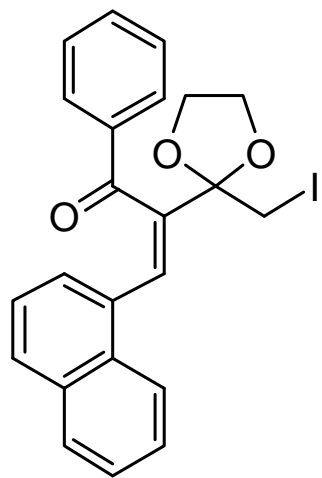
77.000

76.682

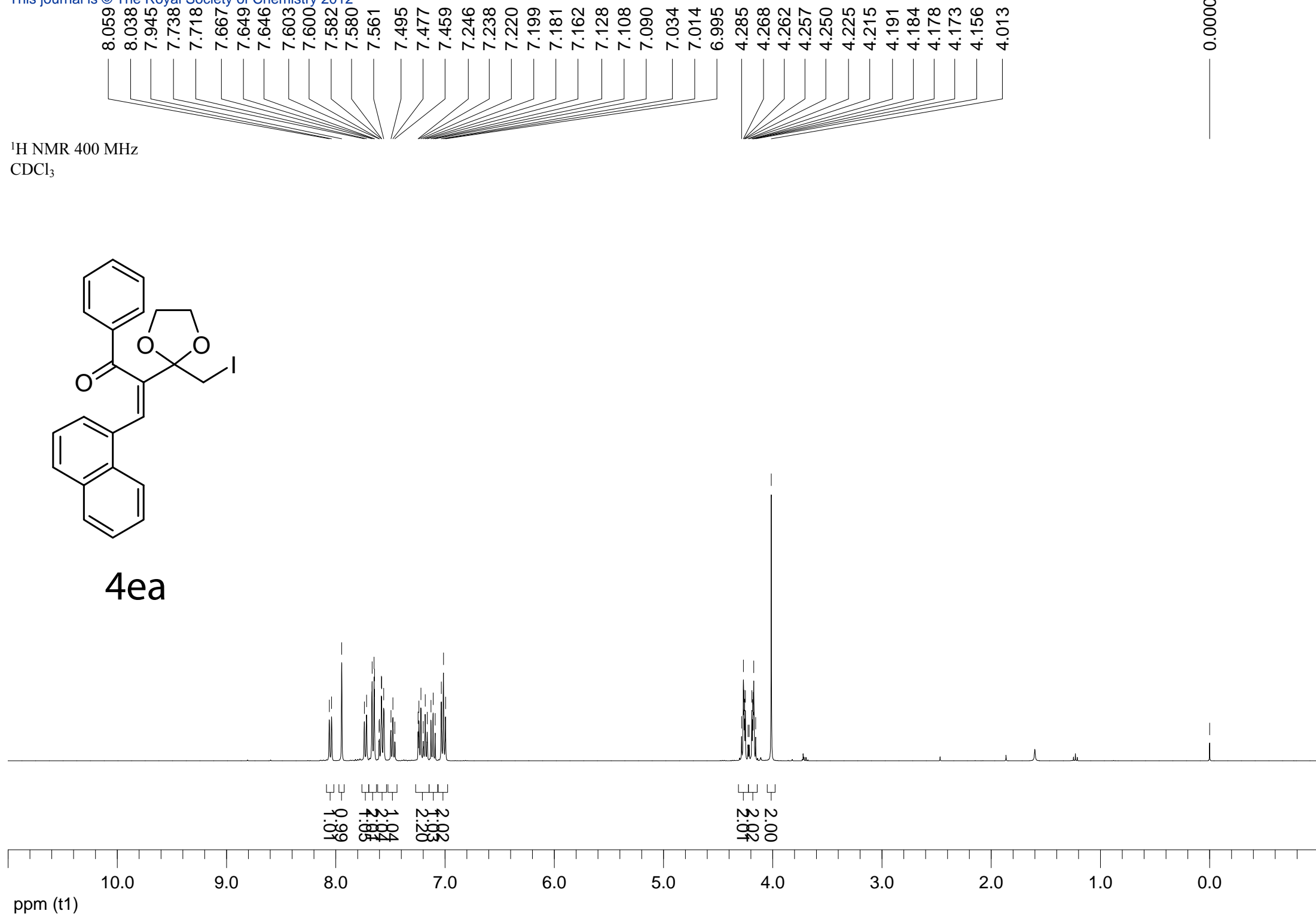
66.270

12.372





4ea



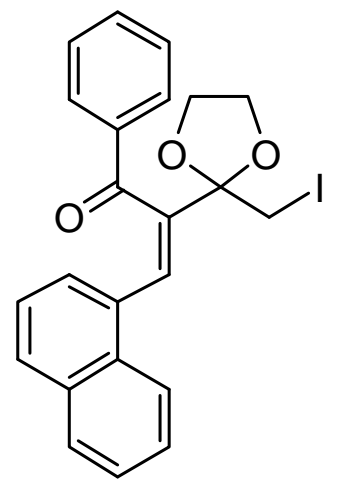
197.185

139.538  
136.284  
133.169  
132.870  
131.556  
131.180  
130.595  
129.042  
128.625  
128.581  
127.967  
127.636  
126.512  
125.972  
125.117  
123.834  
107.479

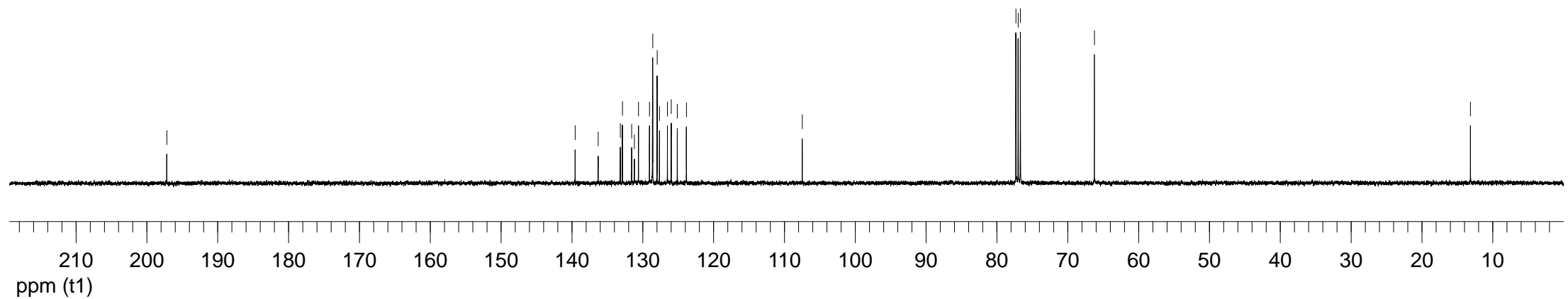
77.317  
77.000  
76.682  
66.241

13.146

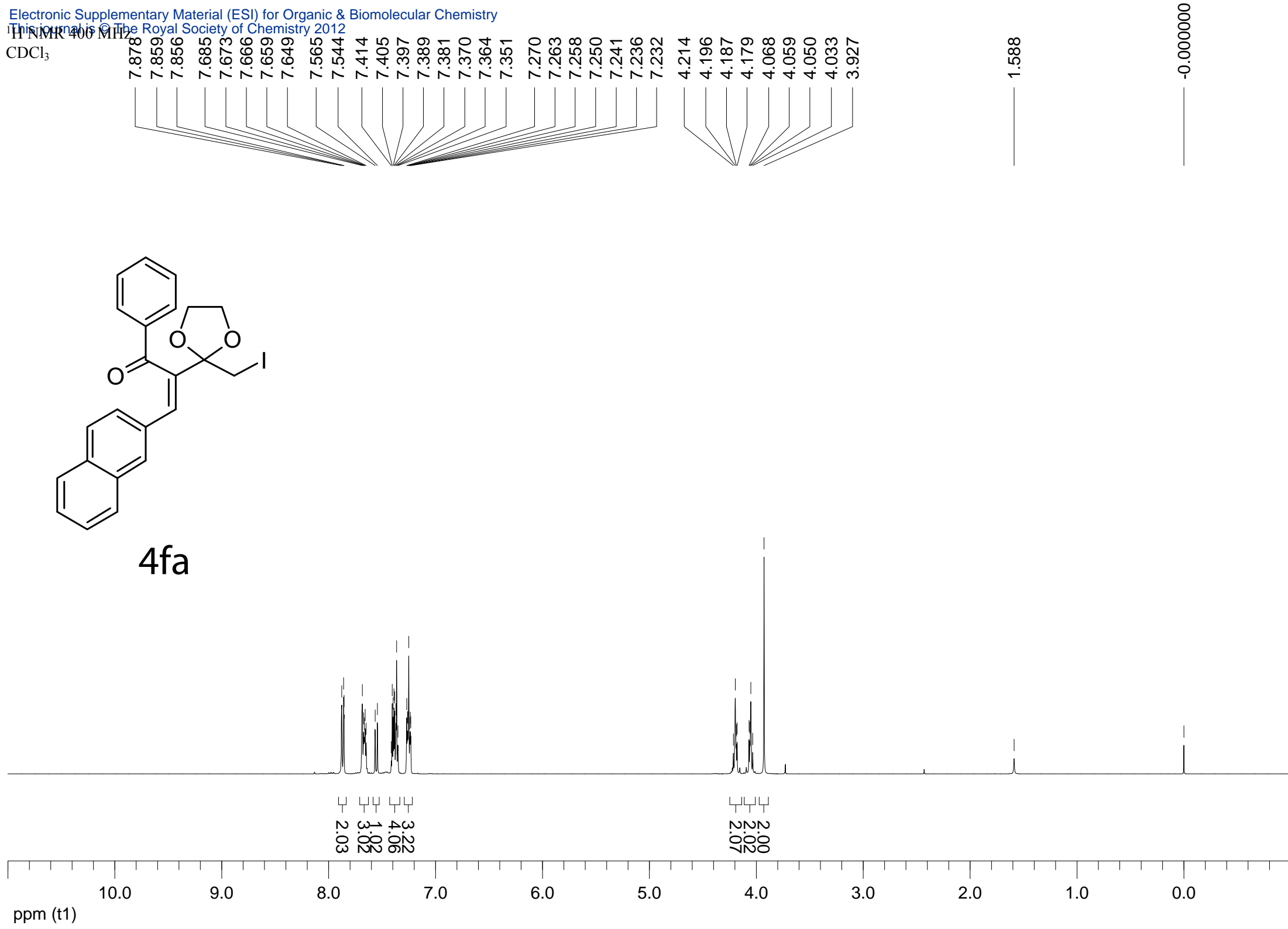
<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

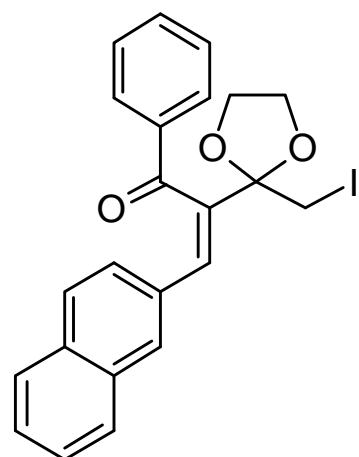


4ea

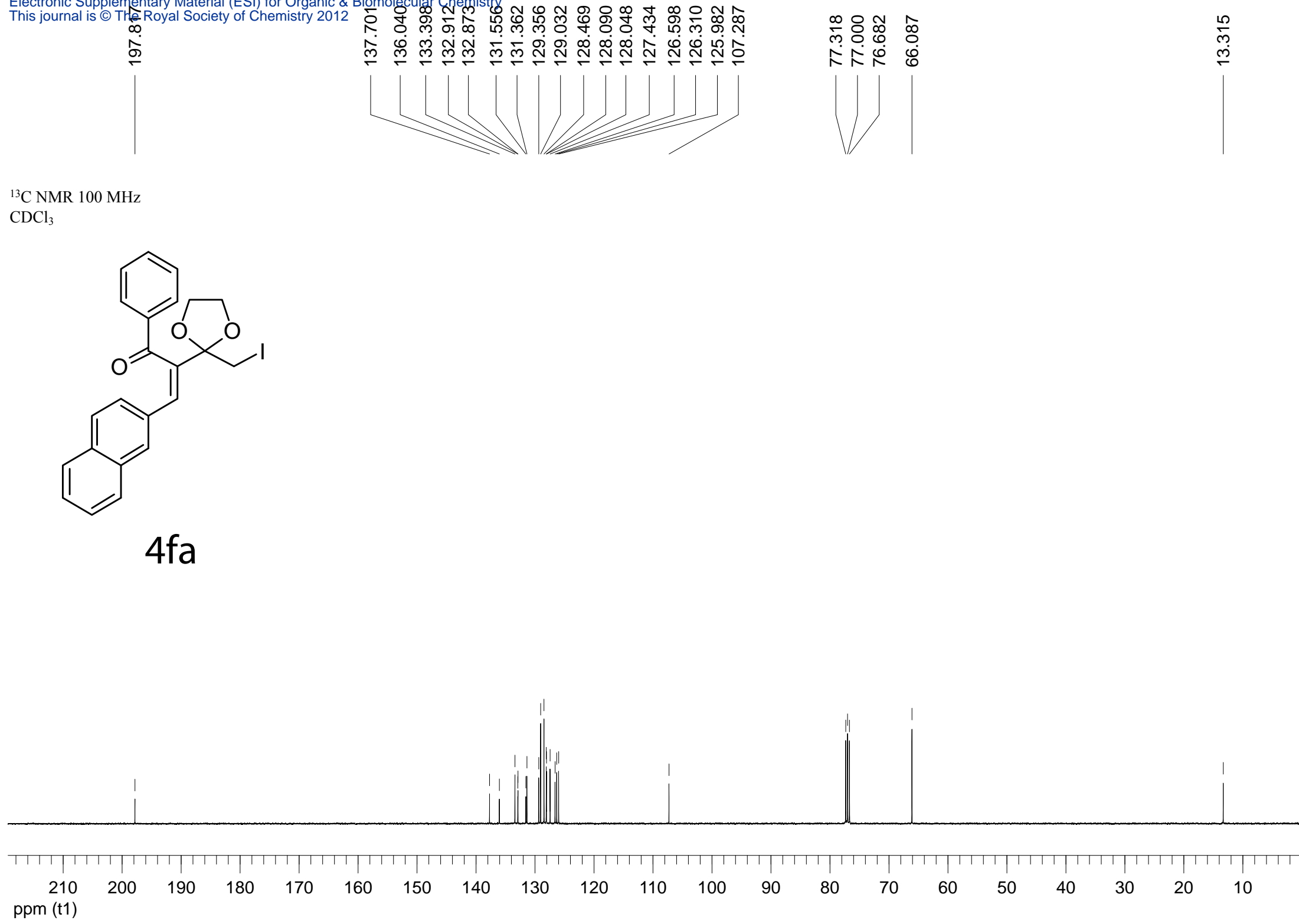


CDCl<sub>3</sub>

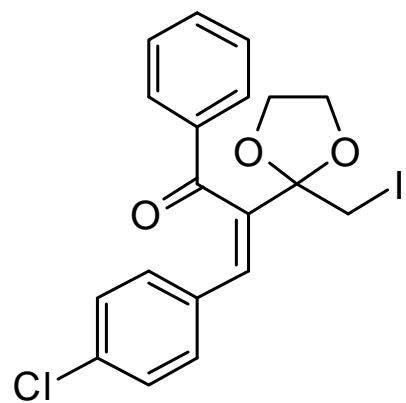




4fa





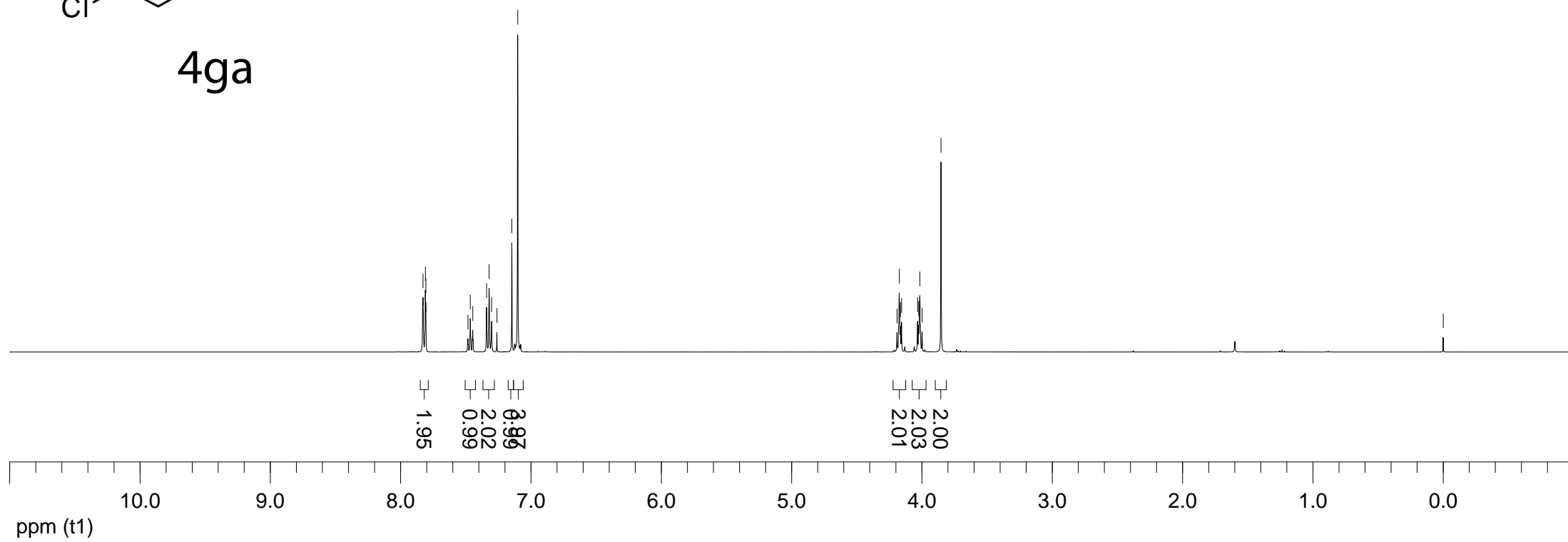
<sup>1</sup>H NMR 400 MHzCDCl<sub>3</sub>

4ga

7.828  
7.810  
7.807  
7.484  
7.465  
7.447  
7.340  
7.320  
7.302  
7.261  
7.146  
7.101

4.191  
4.174  
4.165  
4.156  
4.033  
4.025  
4.016  
3.998  
3.853

-0.000000

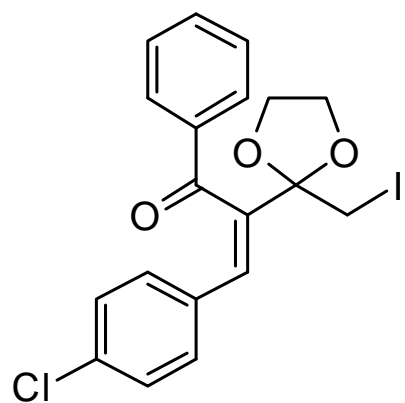


197.459  
138.333  
135.731  
134.475  
133.654  
132.557  
130.276  
129.961  
129.094  
128.643  
128.596  
107.174

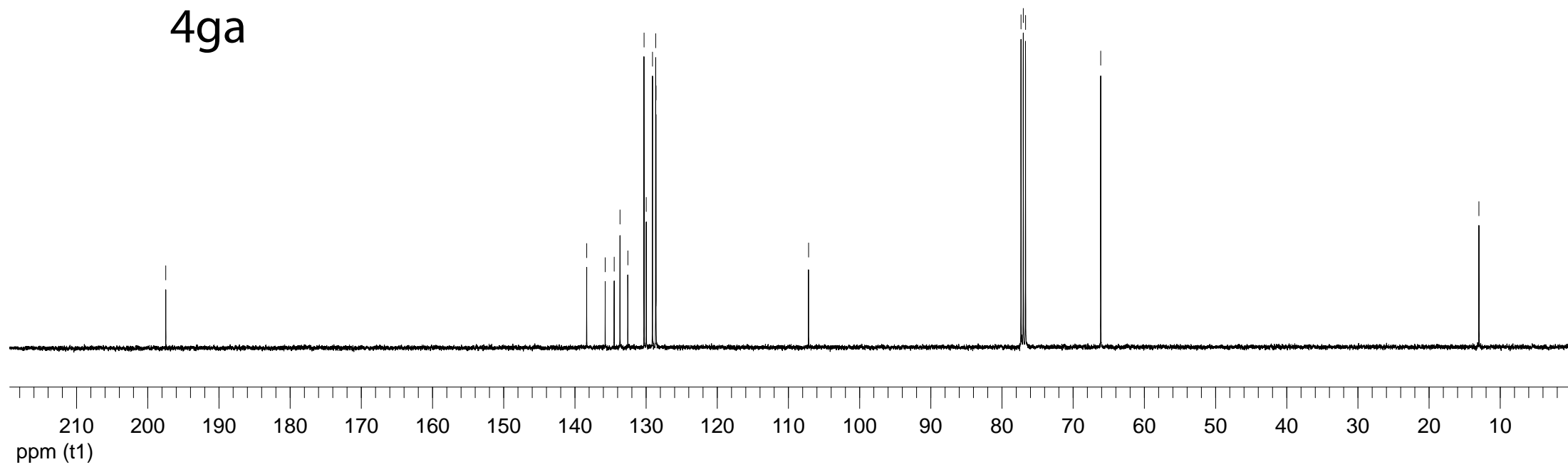
77.318  
77.000  
76.683  
66.119

12.988

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



4ga

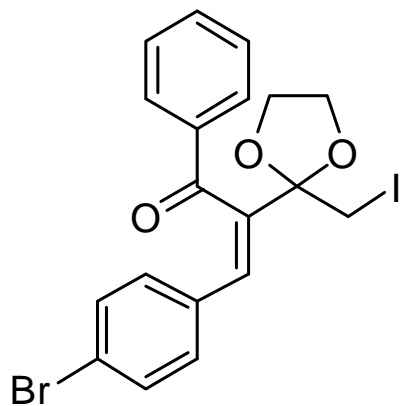


<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

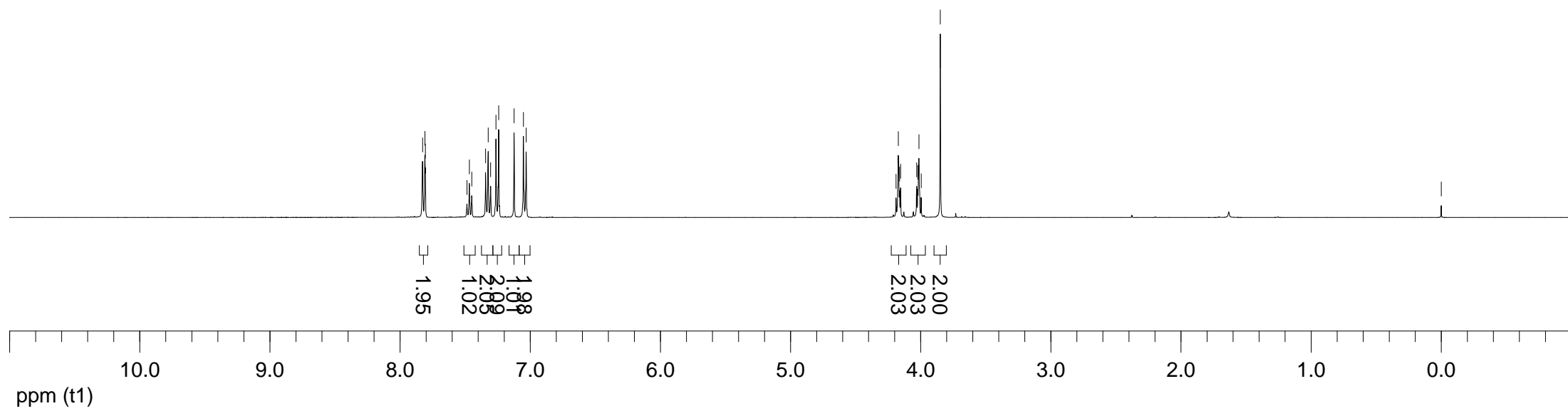
7.827  
7.809  
7.806  
7.486  
7.468  
7.449  
7.342  
7.323  
7.304  
7.263  
7.242  
7.124  
7.052  
7.031

4.189  
4.171  
4.162  
4.154  
4.030  
4.022  
4.013  
3.996  
3.849

-0.000000



4ha



197.408

138.413

135.666

133.656

132.953

131.573

130.496

129.947

129.056

128.587

122.760

107.143

77.318

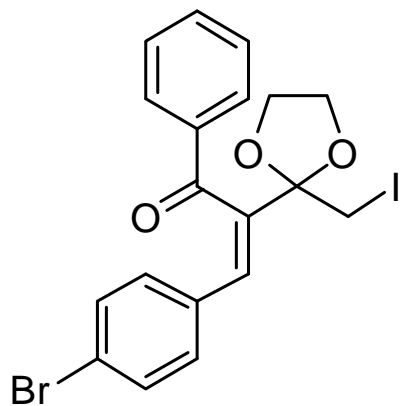
77.000

76.682

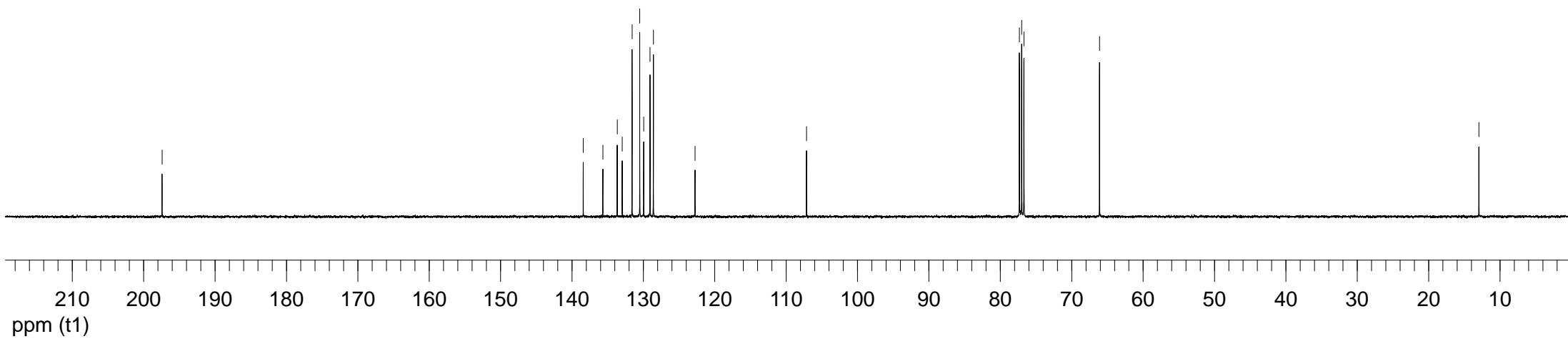
66.094

12.951

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



4ha



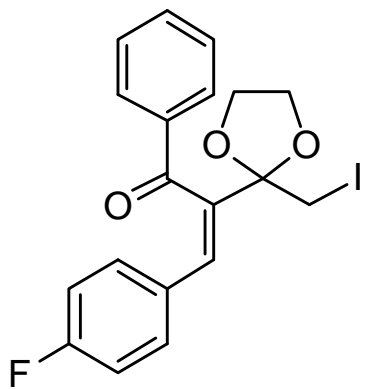
<sup>1</sup>H NMR 400 MHzCDCl<sub>3</sub>

7.833  
7.830  
7.813  
7.809  
7.475  
7.457  
7.438  
7.334  
7.314  
7.295  
7.262  
7.171  
7.158  
7.150  
7.137  
6.836  
6.814  
6.792

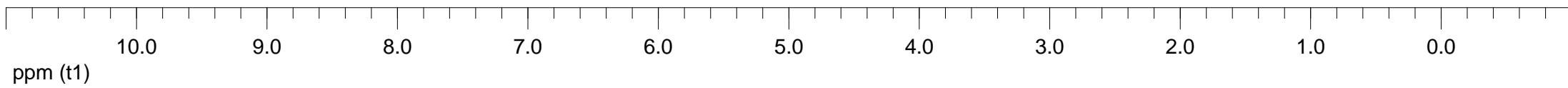
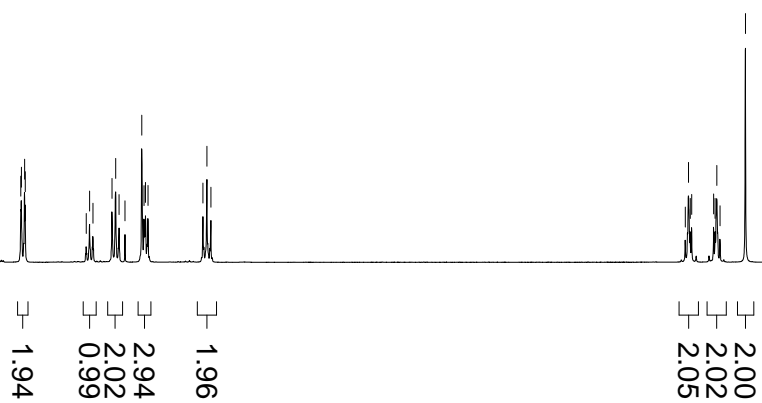
4.195  
4.177  
4.168  
4.160  
4.039  
4.031  
4.022  
4.004  
3.865

1.611

0.000000



4ia



197.637

163.802

161.321

137.502

135.799

133.576

130.927

130.845

130.283

130.249

130.203

129.116

128.548

115.594

115.379

107.199

77.318

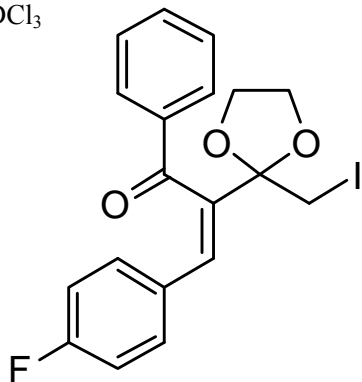
77.000

76.683

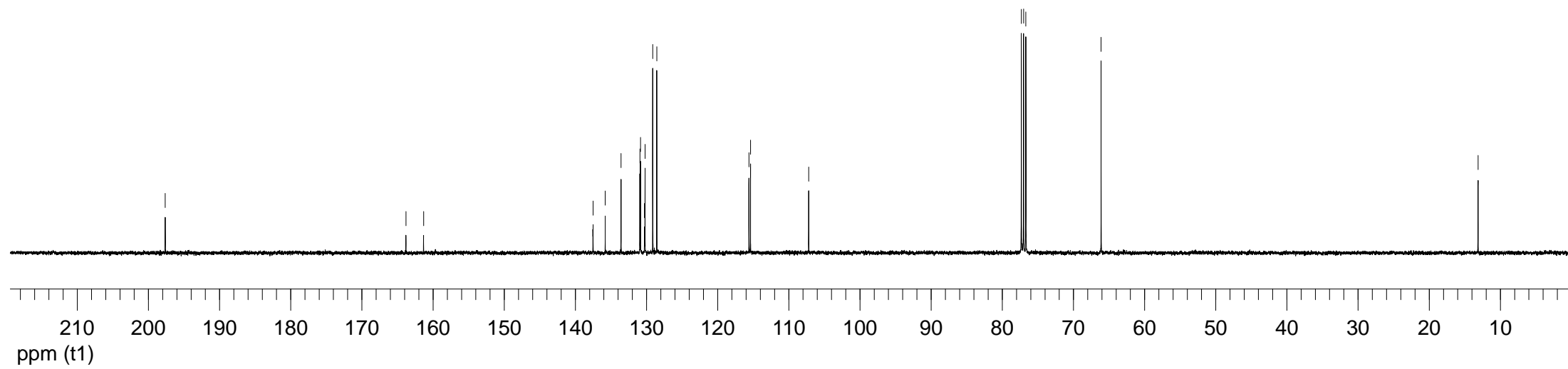
66.105

13.130

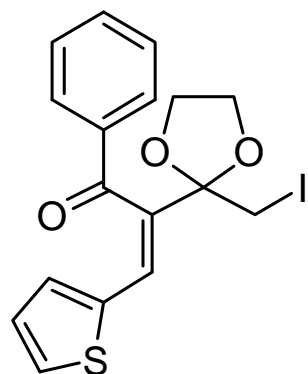
<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



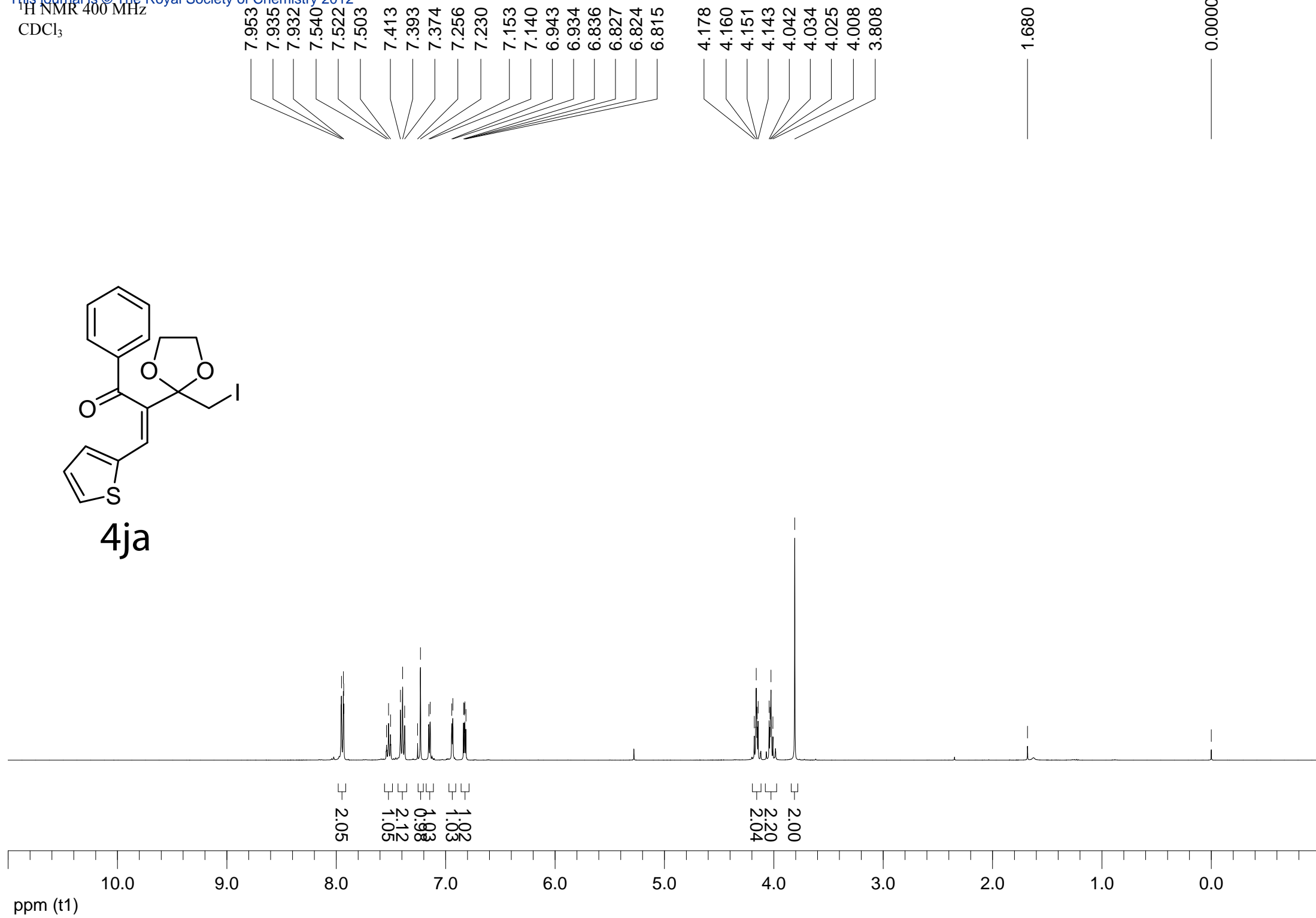
4ia



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



4ja



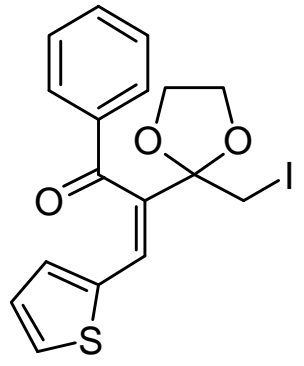
197.235

137.002  
136.029  
135.674  
133.679  
129.905  
129.213  
128.652  
128.084  
127.339  
123.264  
106.908

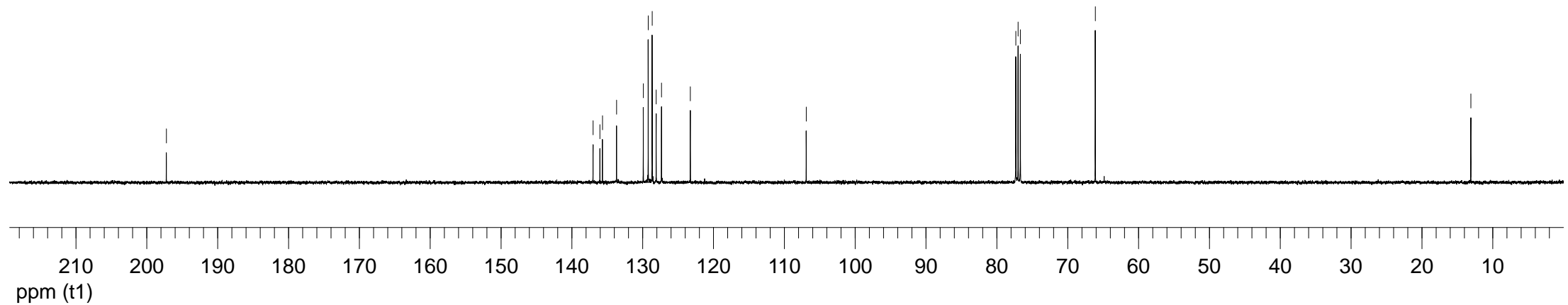
77.318  
77.000  
76.682  
66.100

13.086

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



4ja





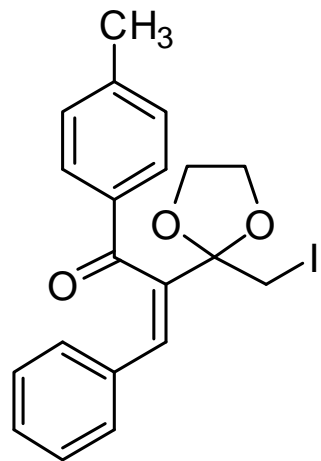
<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>

7.746  
7.726  
7.255  
7.199  
7.190  
7.180  
7.174  
7.168  
7.133  
7.123  
7.116  
7.104  
7.083

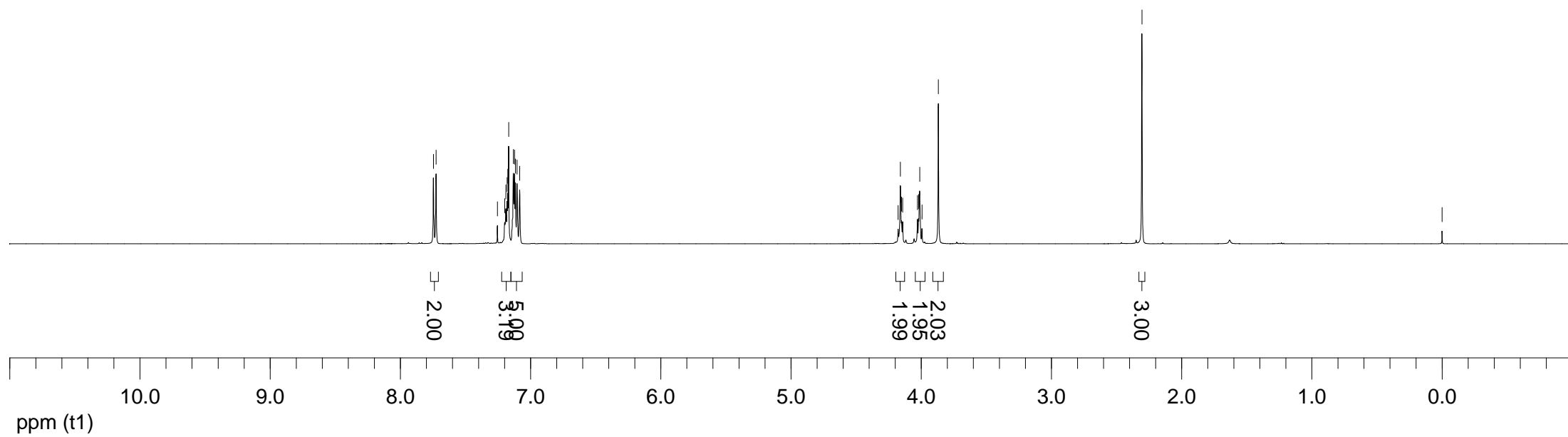
4.177  
4.160  
4.151  
4.142  
4.029  
4.021  
4.012  
3.994  
3.868

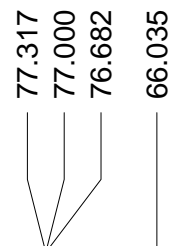
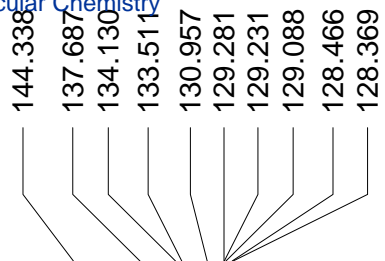
2.305

-0.000000

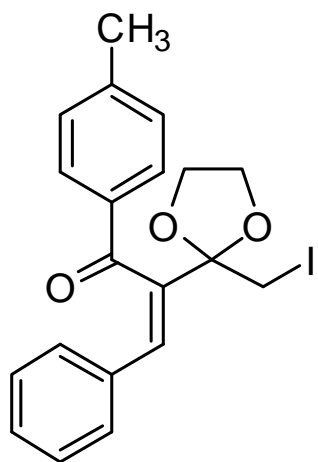


4ab

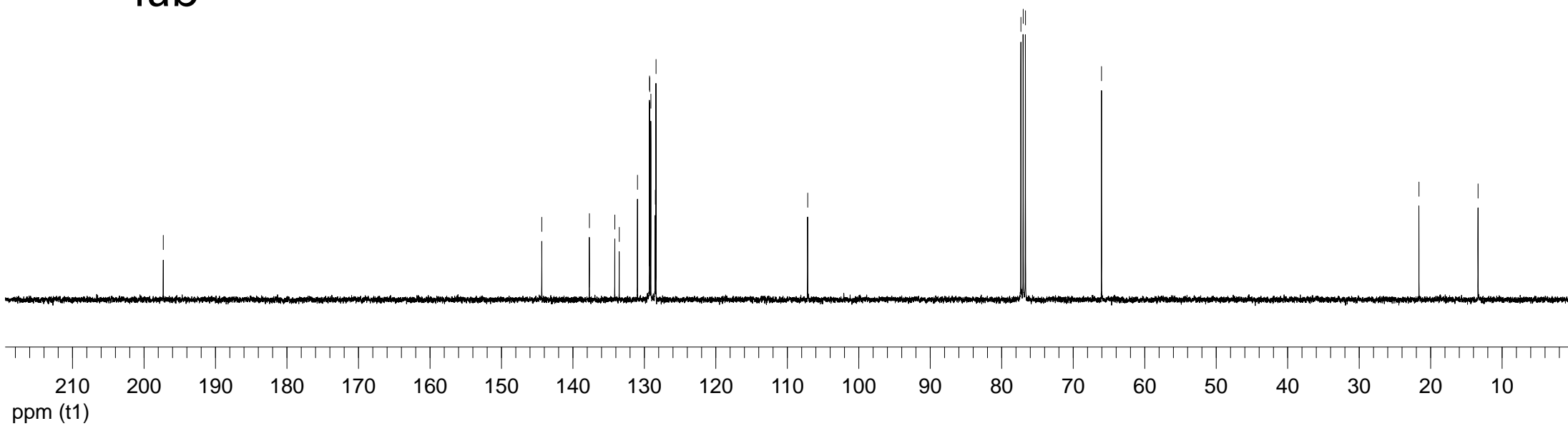


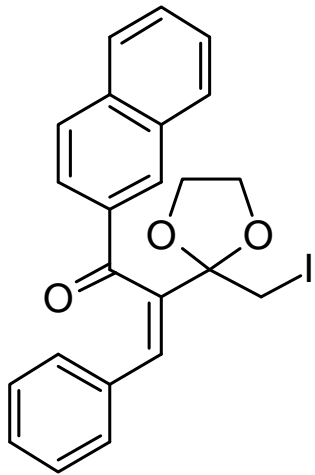


<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>

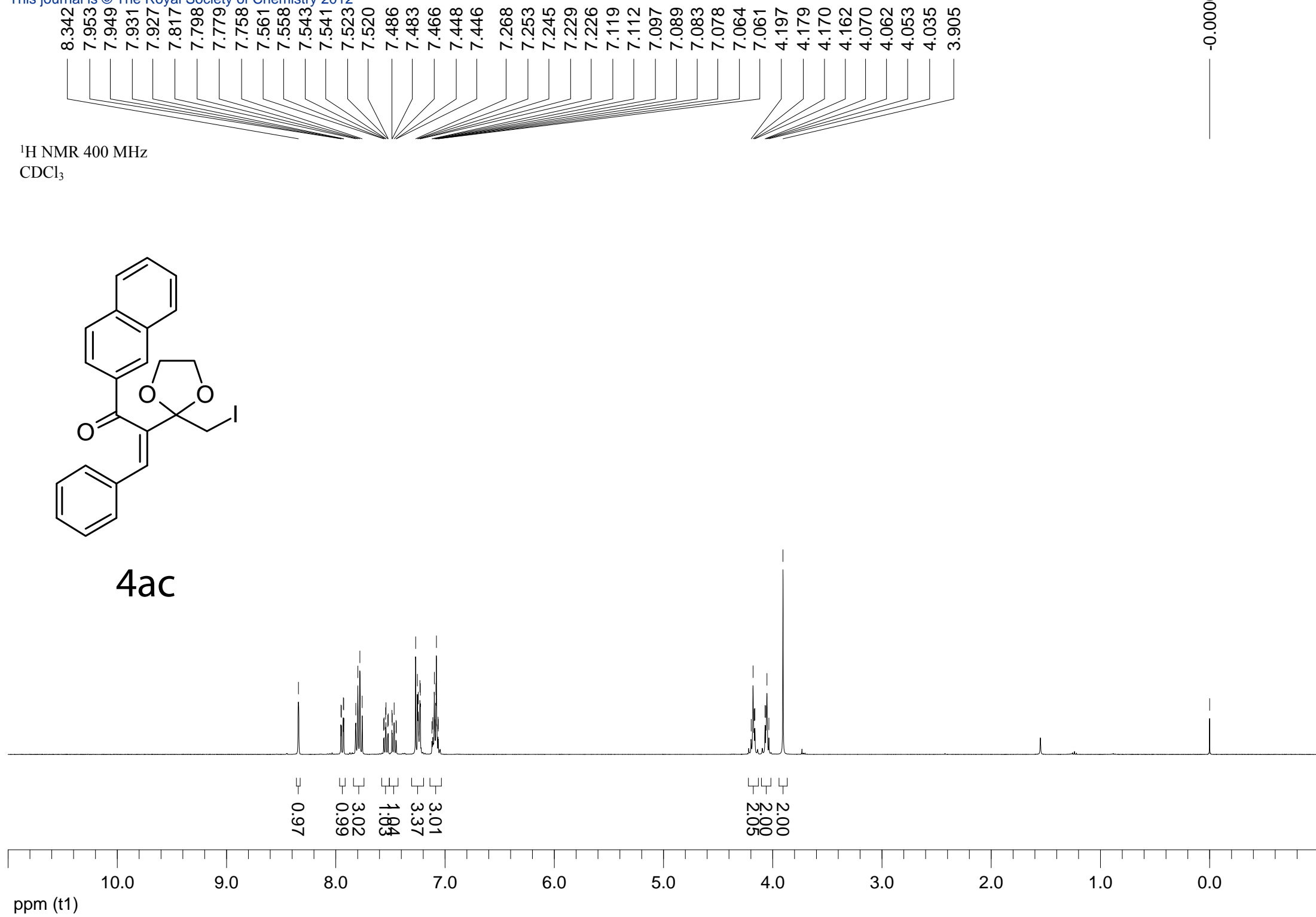


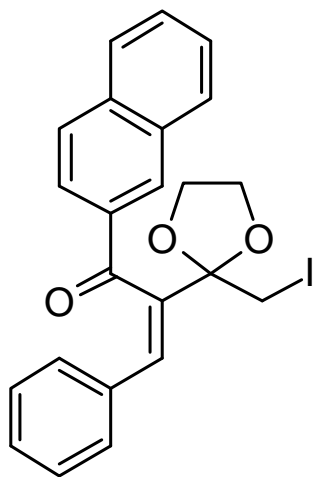
4ab





4ac





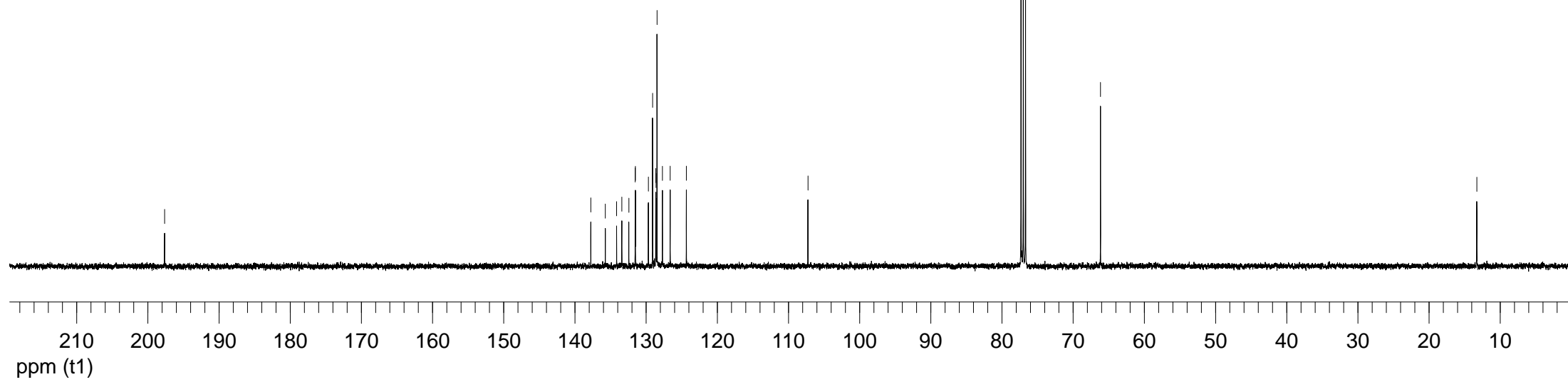
4ac

$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$

197.650  
137.764  
135.719  
134.132  
133.398  
132.420  
131.531  
131.489  
129.675  
129.083  
128.637  
128.596  
128.469  
127.697  
126.610  
124.335  
107.259

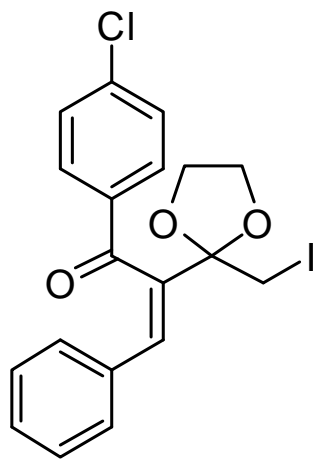
77.318  
77.000  
76.682  
66.150

13.280



<sup>1</sup>H NMR 400 MHz

CDCl<sub>3</sub>

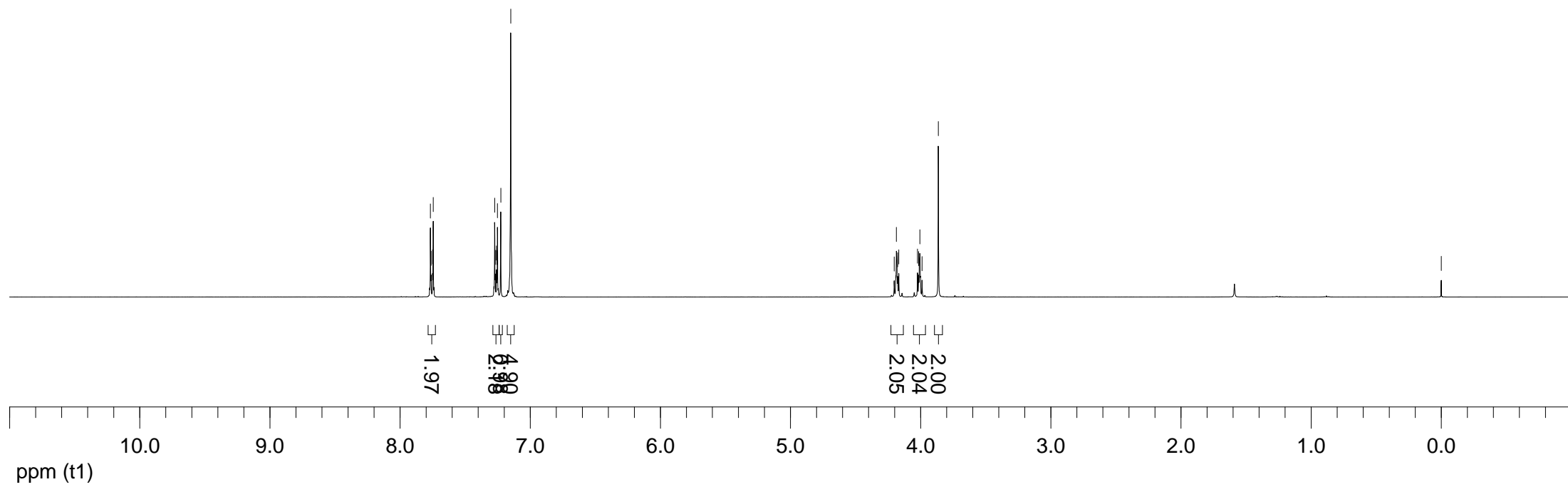


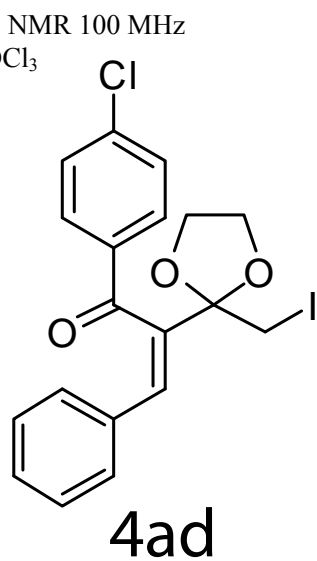
4ad

7.766  
7.762  
7.750  
7.745  
7.273  
7.268  
7.259  
7.256  
7.251  
7.226  
7.149

4.203  
4.186  
4.176  
4.168  
4.024  
4.015  
4.006  
3.989  
3.864

0.000000





196.468

139.835

137.140

134.338

133.878

131.748

130.487

129.050

128.841

128.804

128.500

107.274

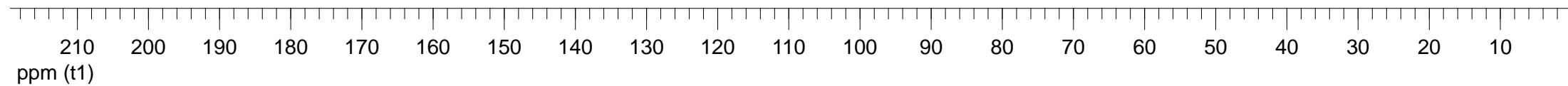
77.317

77.000

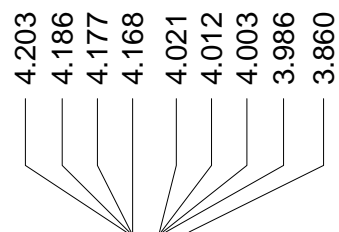
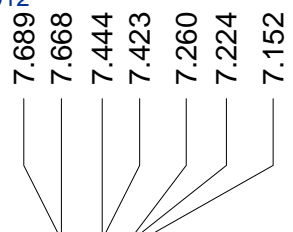
76.682

66.135

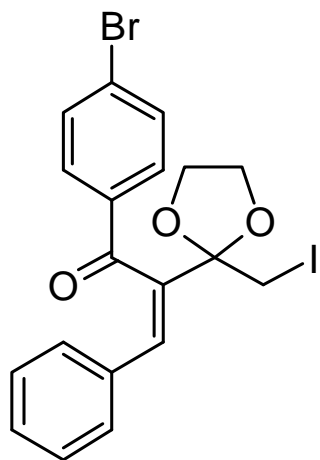
12.977



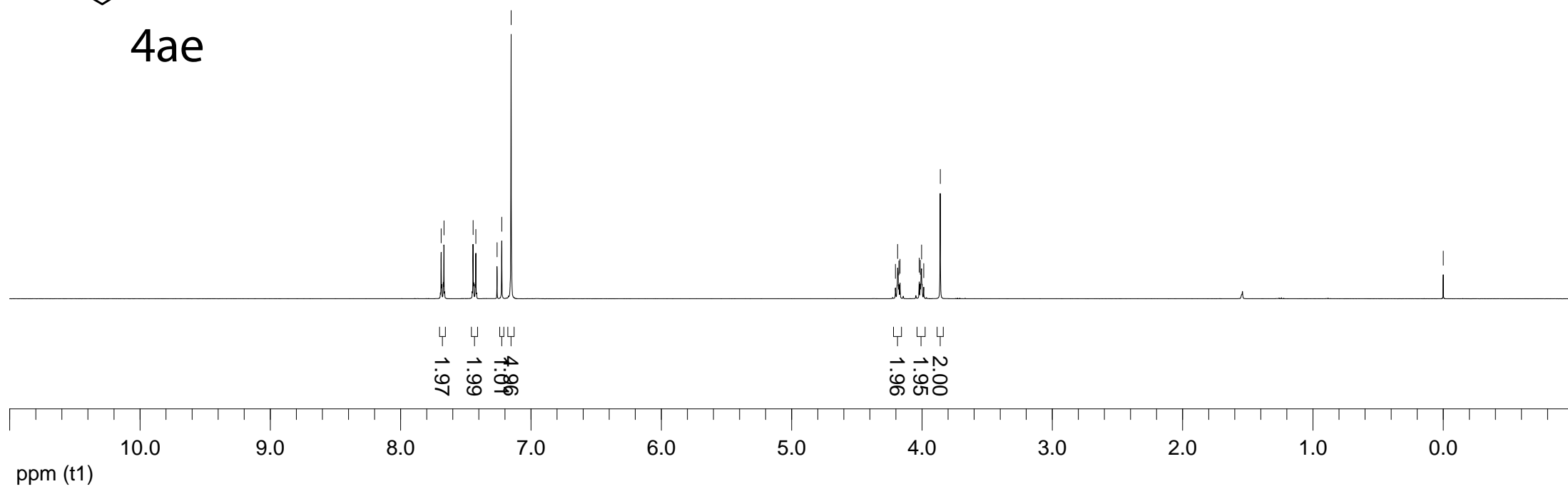
<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>



-0.000000-



4ae



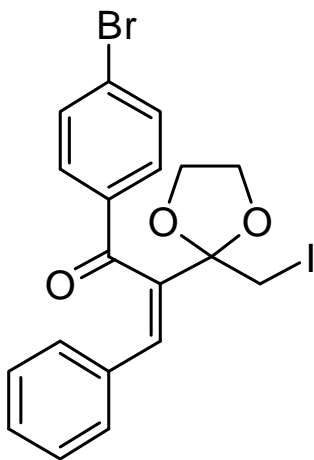
196.658

137.062  
134.696  
133.824  
131.803  
131.727  
130.544  
129.017  
128.795  
128.675  
128.490  
107.230

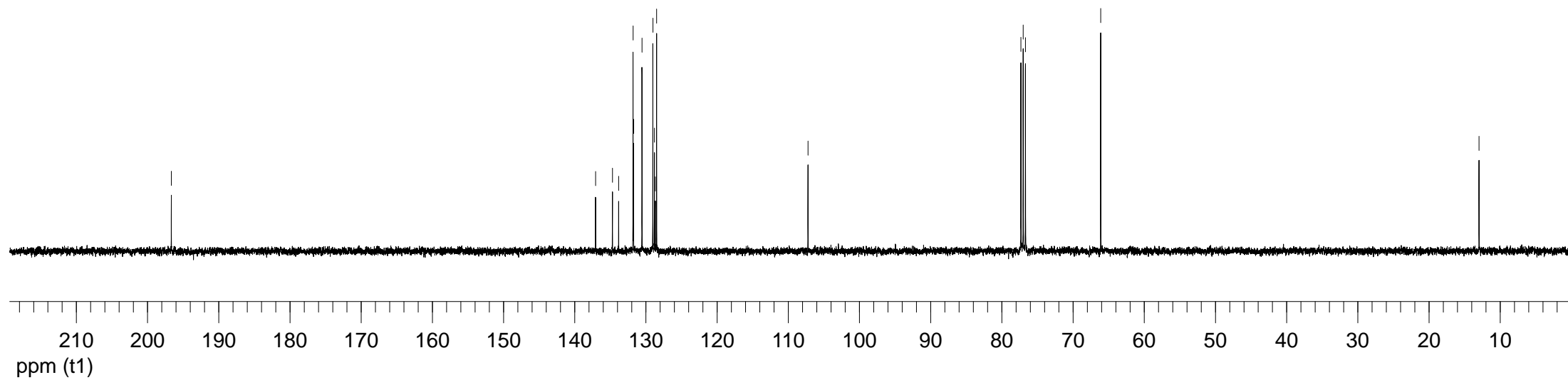
77.318  
77.000  
76.682  
66.108

12.967

<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



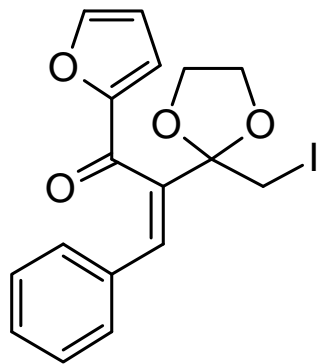
4ae





<sup>1</sup>H NMR 400 MHz

CDCl<sub>3</sub>

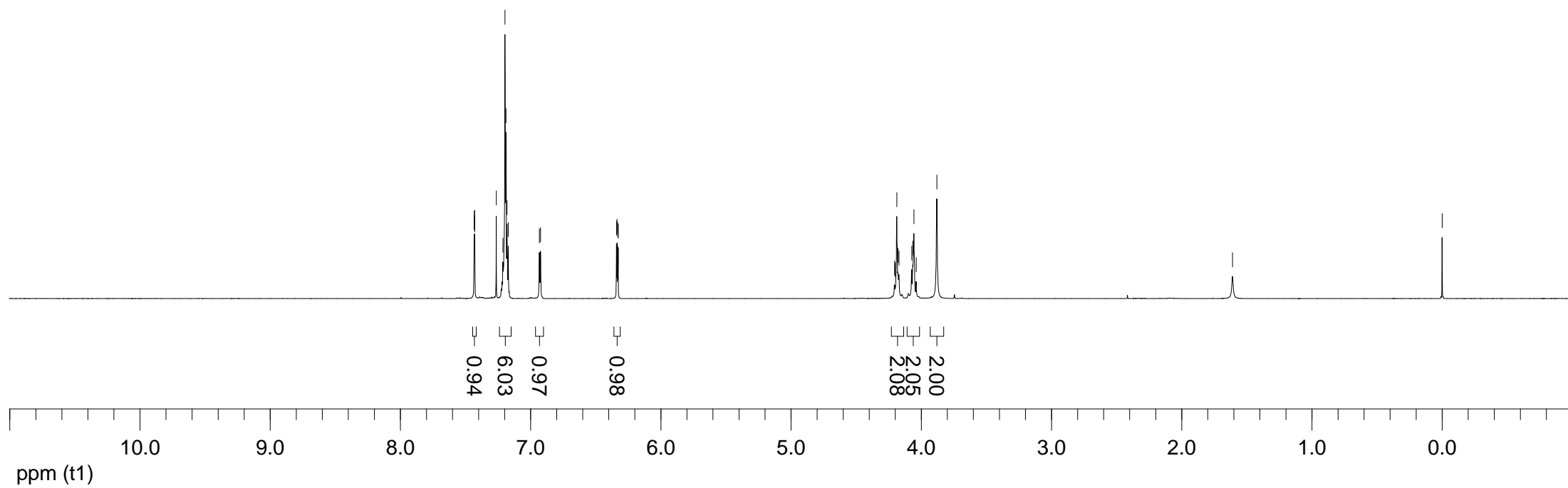


4af

7.434  
7.432  
7.430  
7.428  
7.263  
7.213  
7.196  
7.190  
7.181  
7.171  
6.933  
6.924  
6.340  
6.336  
6.331  
6.327  
4.205  
4.199  
4.188  
4.179  
4.171  
4.074  
4.065  
4.057  
4.039  
3.880

1.610

0.000000



184.610

152.377

147.083

136.990

134.209

132.745

128.841

128.575

128.390

119.666

112.307

106.821

77.317

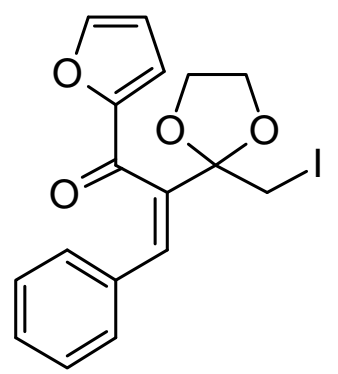
77.000

76.682

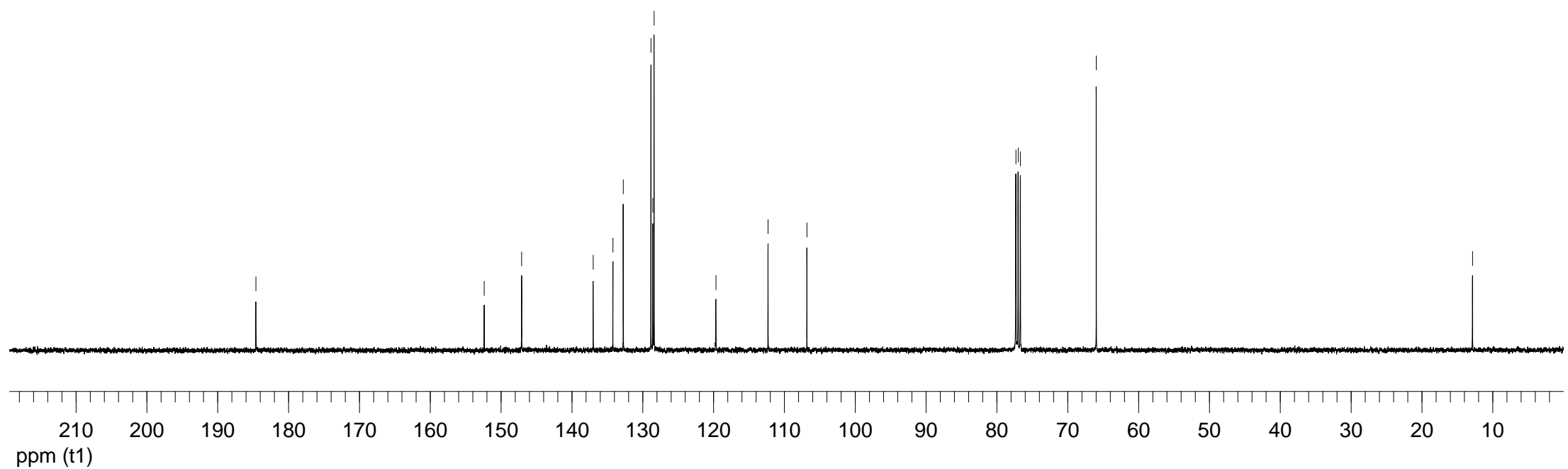
65.967

12.849

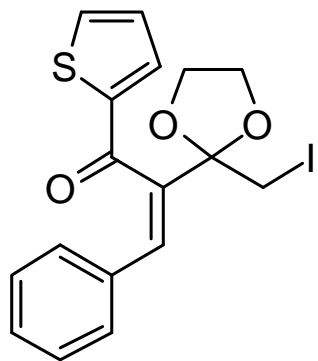
<sup>13</sup>C NMR 100 MHz  
CDCl<sub>3</sub>



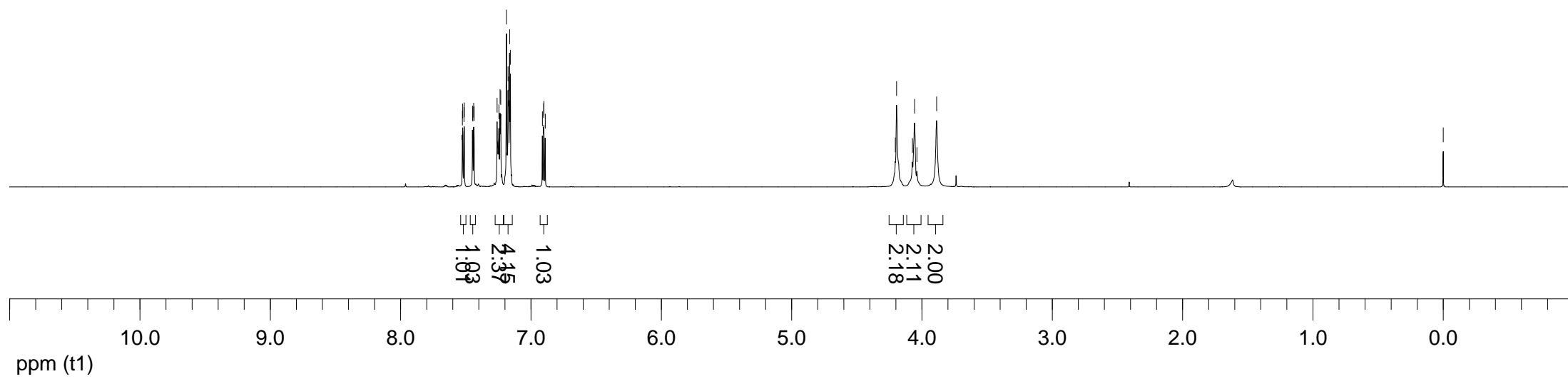
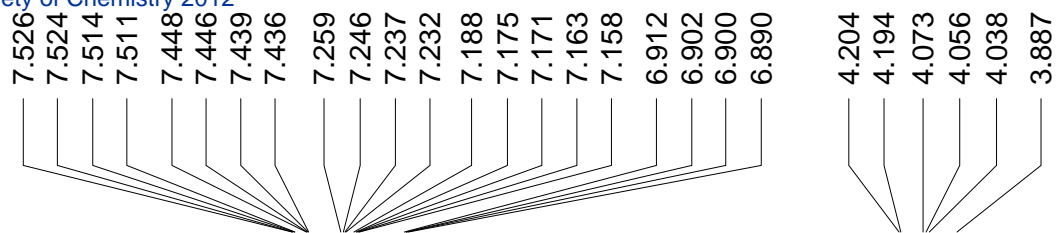
4af



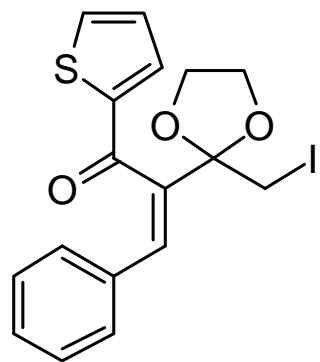
<sup>1</sup>H NMR 400 MHz  
CDCl<sub>3</sub>



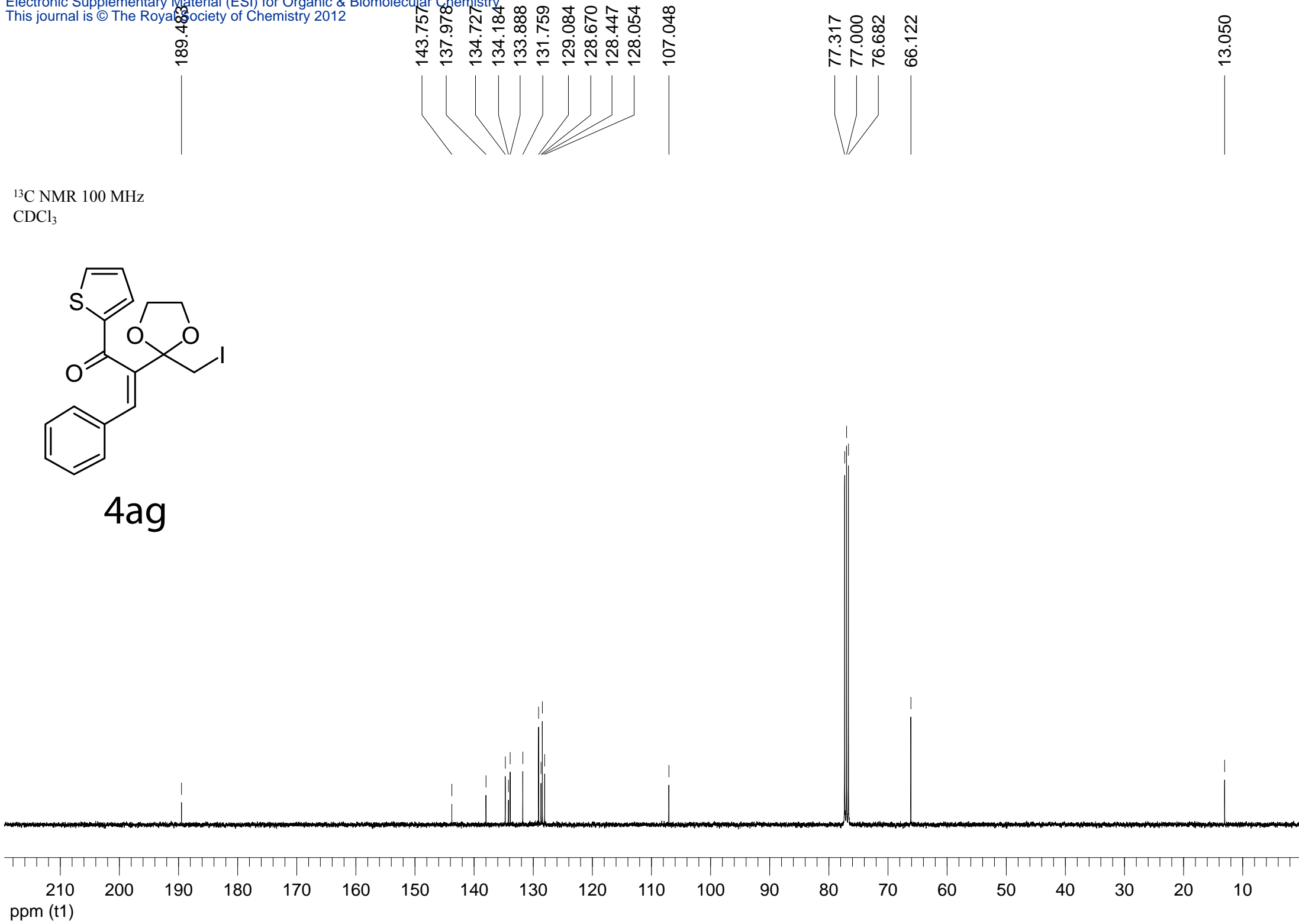
4ag



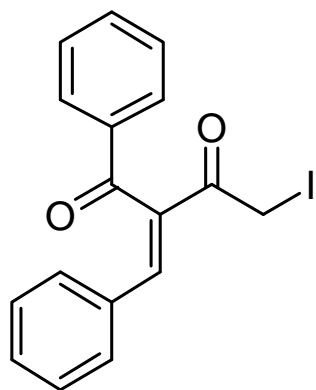
-0.000000



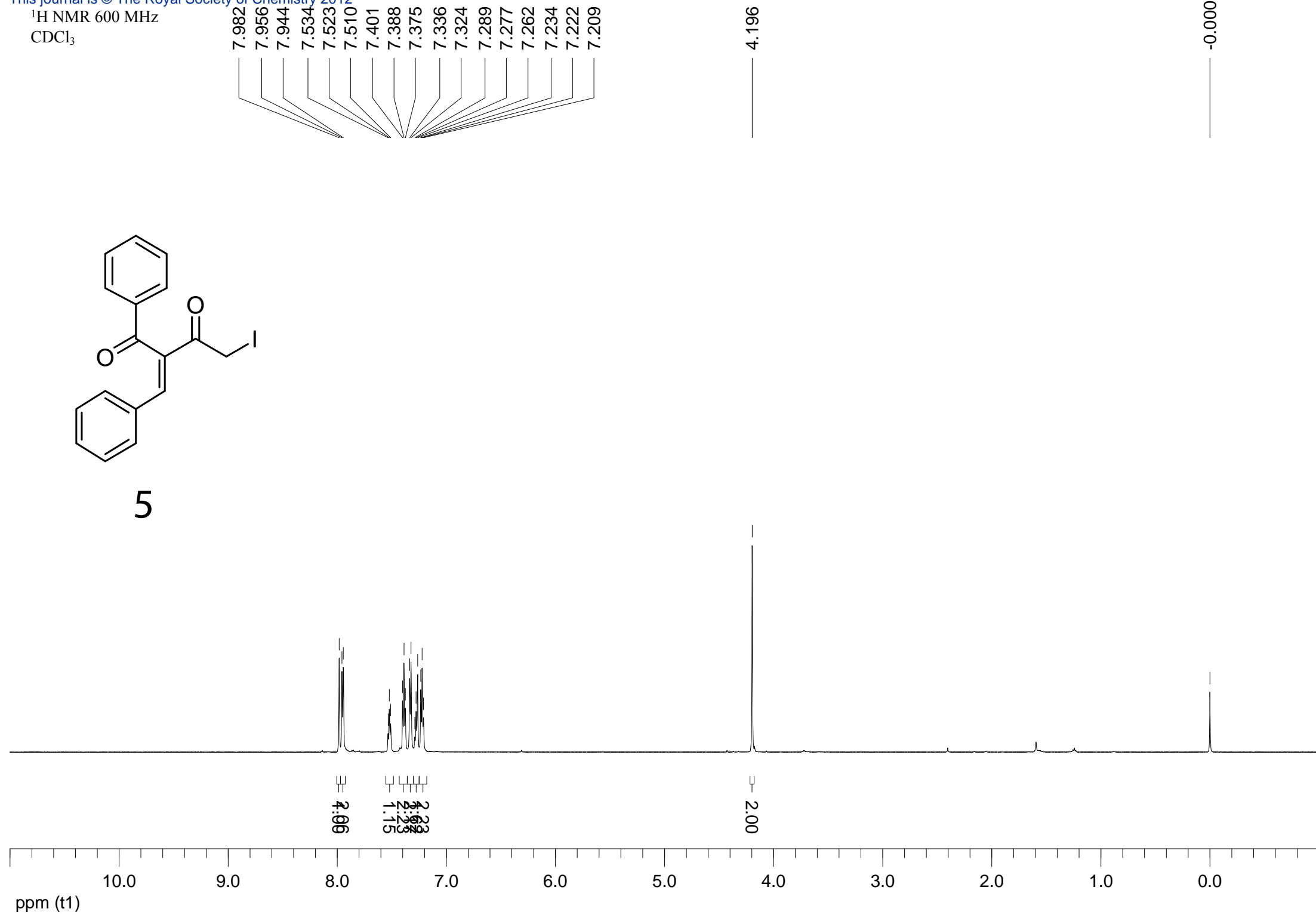
4ag



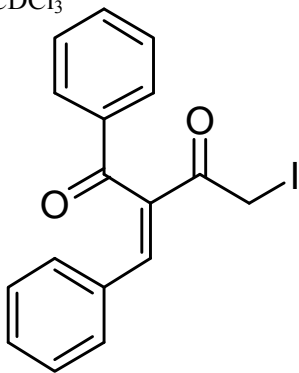
<sup>1</sup>H NMR 600 MHz  
CDCl<sub>3</sub>



5



$^{13}\text{C}$  NMR 100 MHz  
 $\text{CDCl}_3$



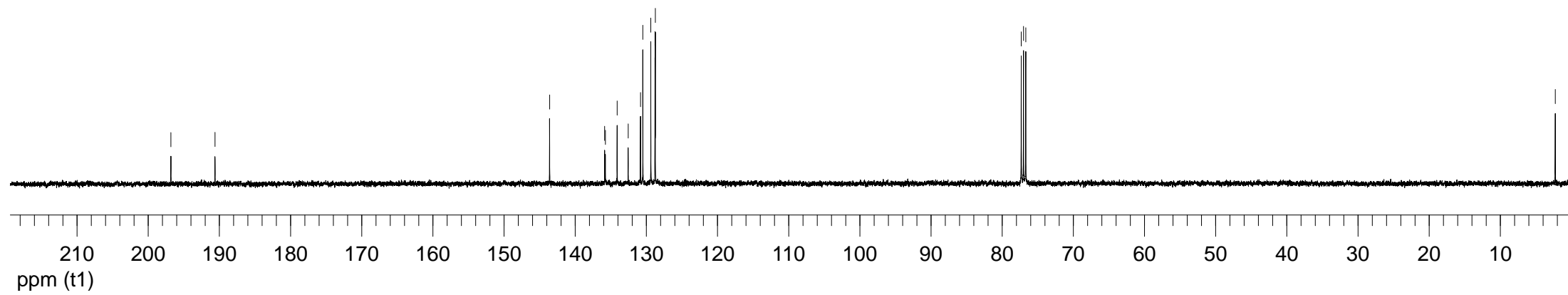
5

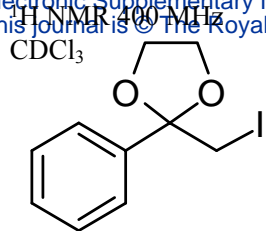
196.832  
190.622

143.593  
135.862  
135.757  
134.105  
132.554  
130.825  
130.489  
129.367  
128.780  
128.741

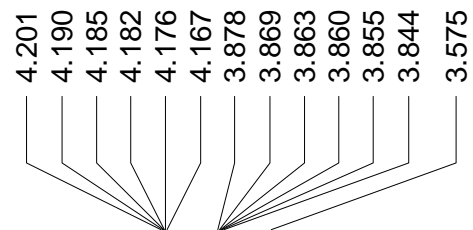
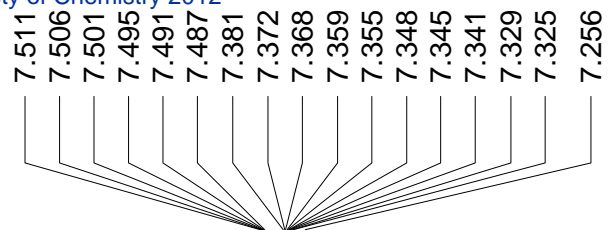
77.317  
77.000  
76.682

2.279

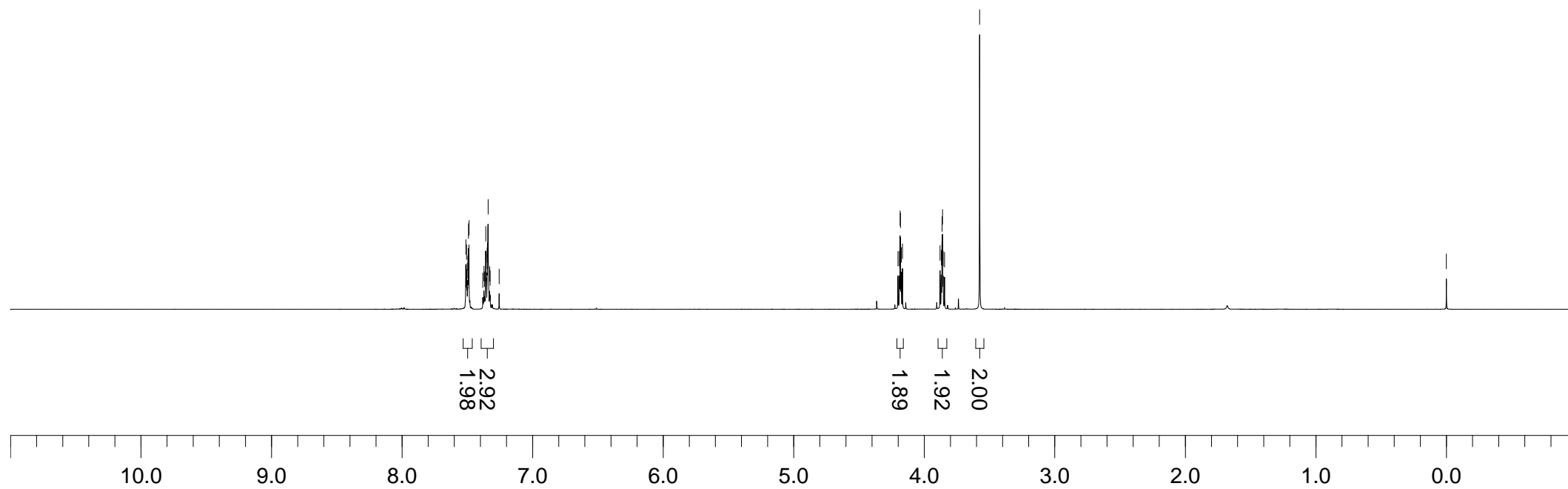




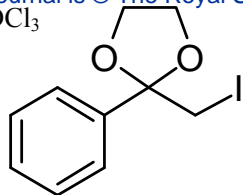
7a



0.000000



<sup>1</sup>H NMR (100 MHz, CDCl<sub>3</sub>)



7a

139.080

128.632

128.311

125.795

106.679

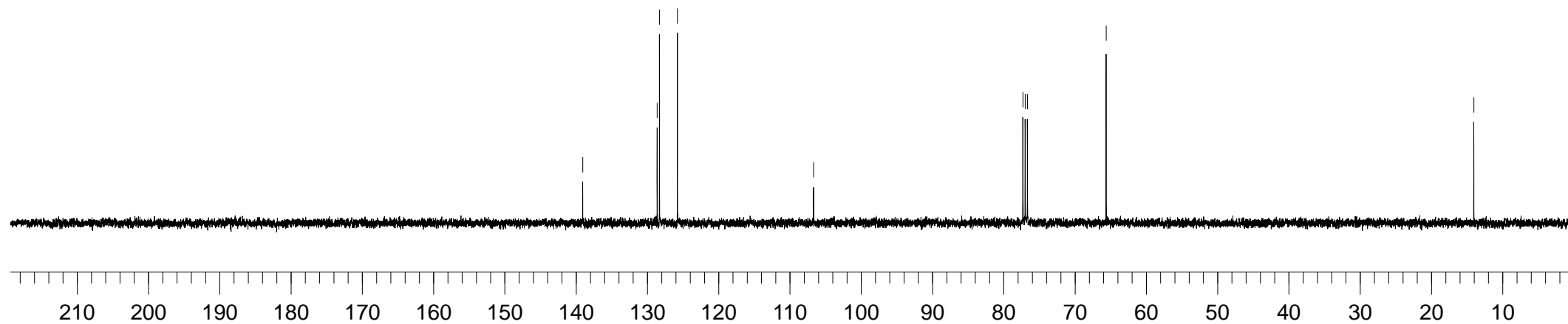
77.317

77.000

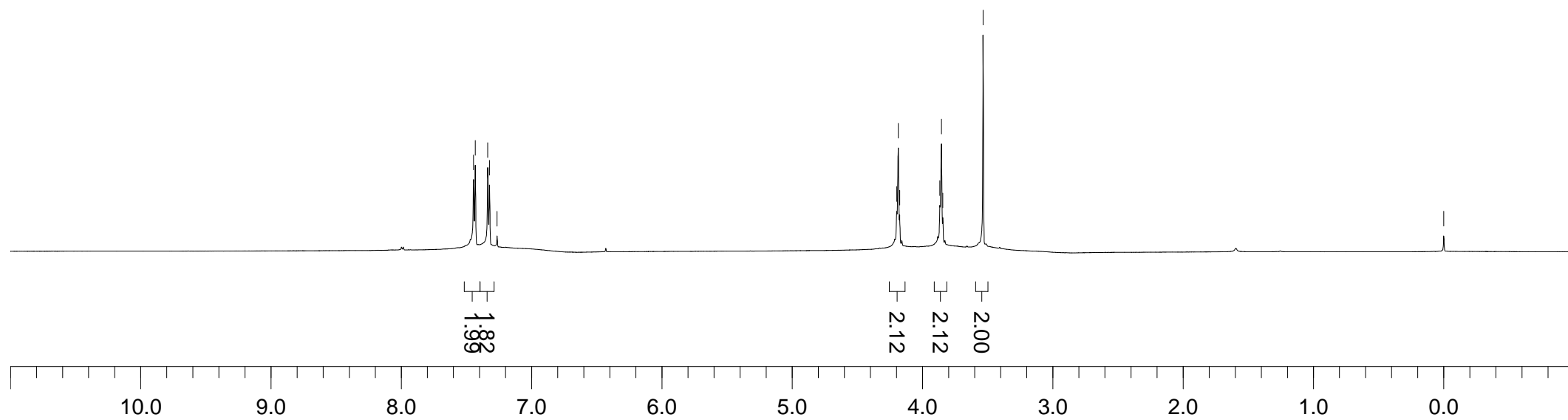
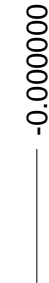
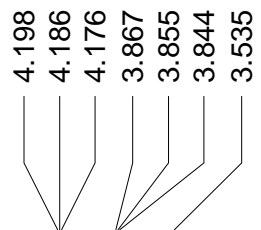
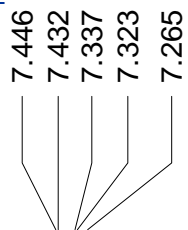
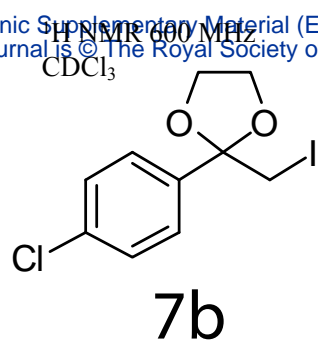
76.682

65.637

14.049

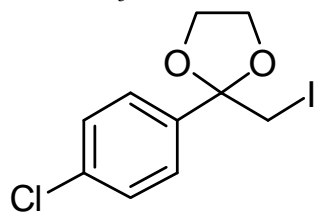






<sup>13</sup>C-NMR (101 MHz,

CDCl<sub>3</sub>)



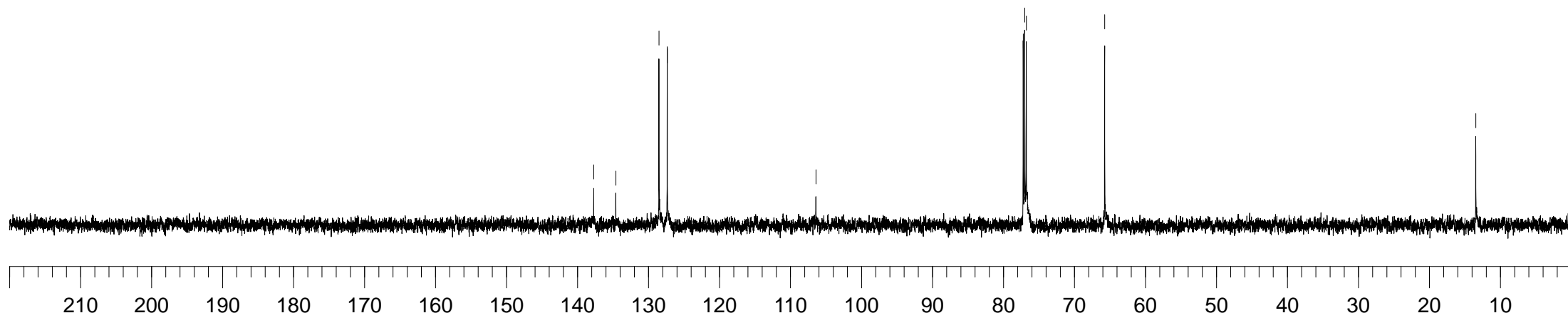
**7b**

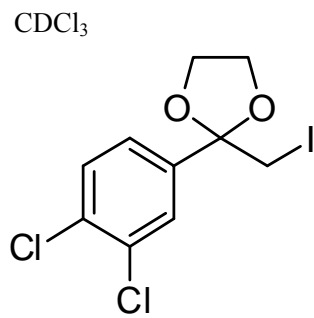
137.714  
134.604  
128.525  
127.359

106.417

77.210  
76.998  
76.786  
65.739

13.469





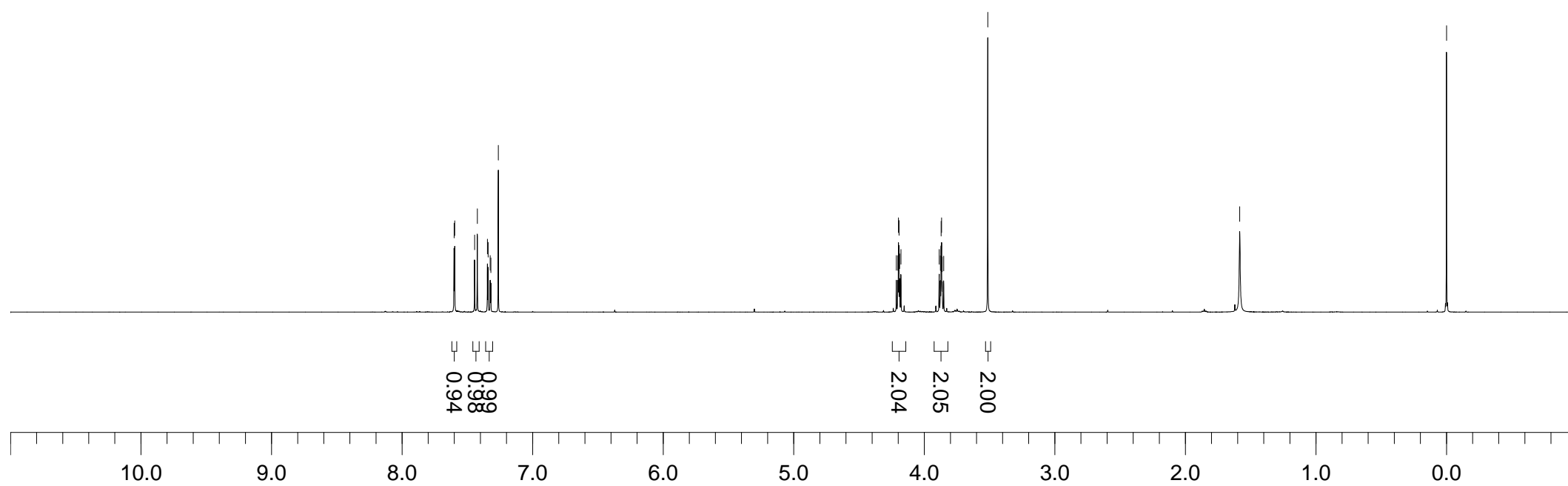
7c

7.602  
7.596  
7.444  
7.423  
7.346  
7.341  
7.325  
7.320  
7.263

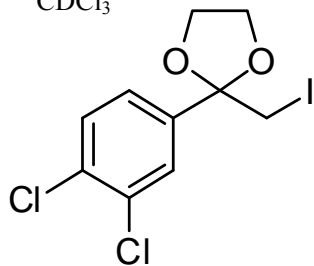
4.214  
4.202  
4.198  
4.194  
4.188  
4.179  
3.886  
3.876  
3.871  
3.867  
3.862  
3.851  
3.514

1.583

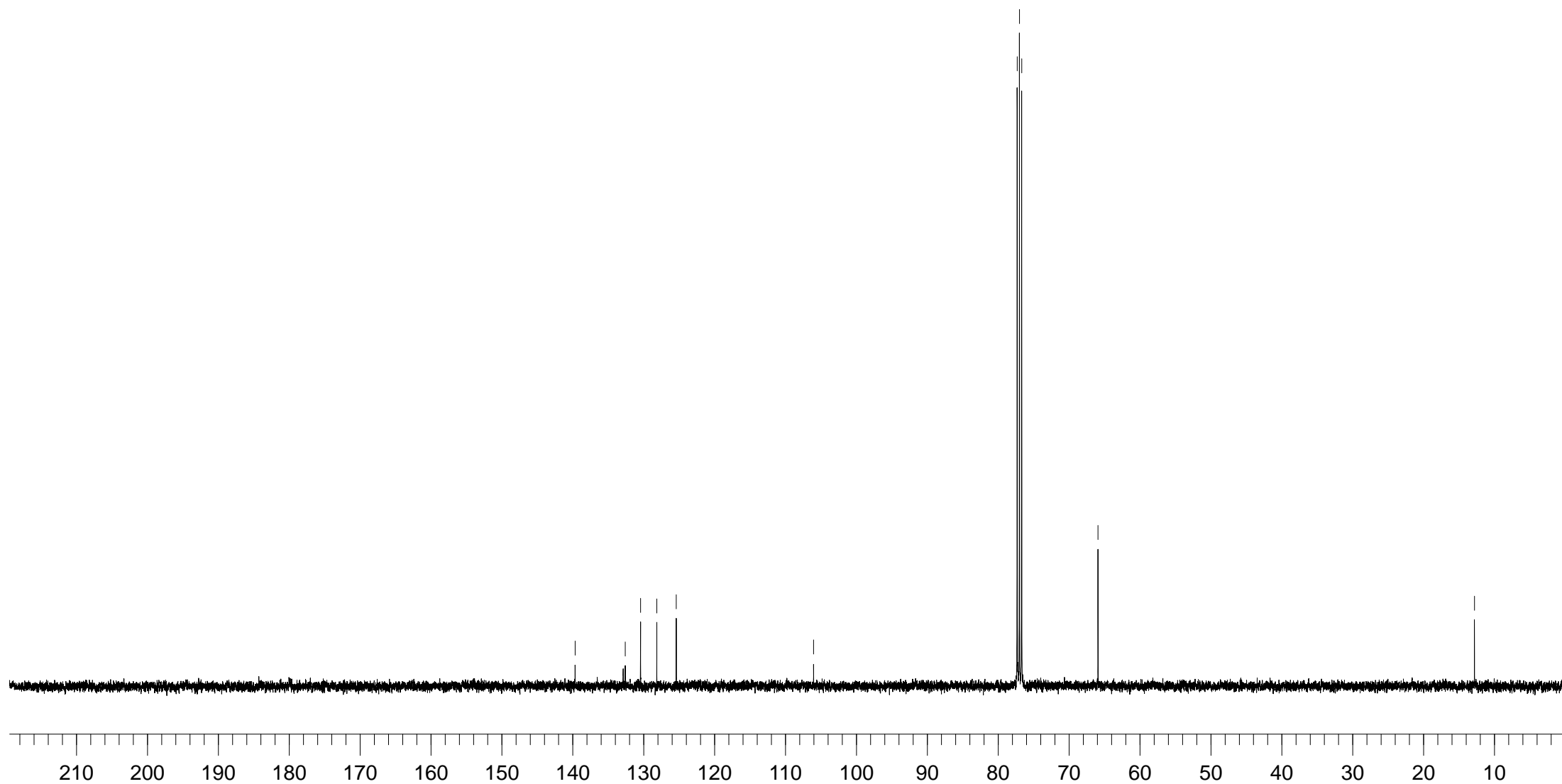
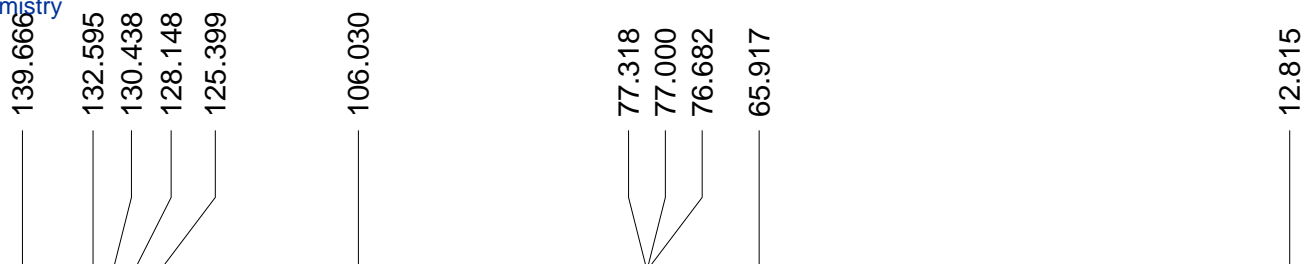
-0.000

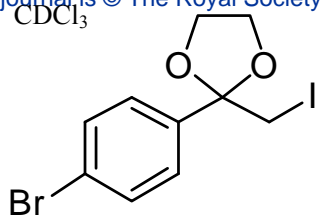


<sup>1</sup>H NMR (100 MHz)  
CDCl<sub>3</sub>

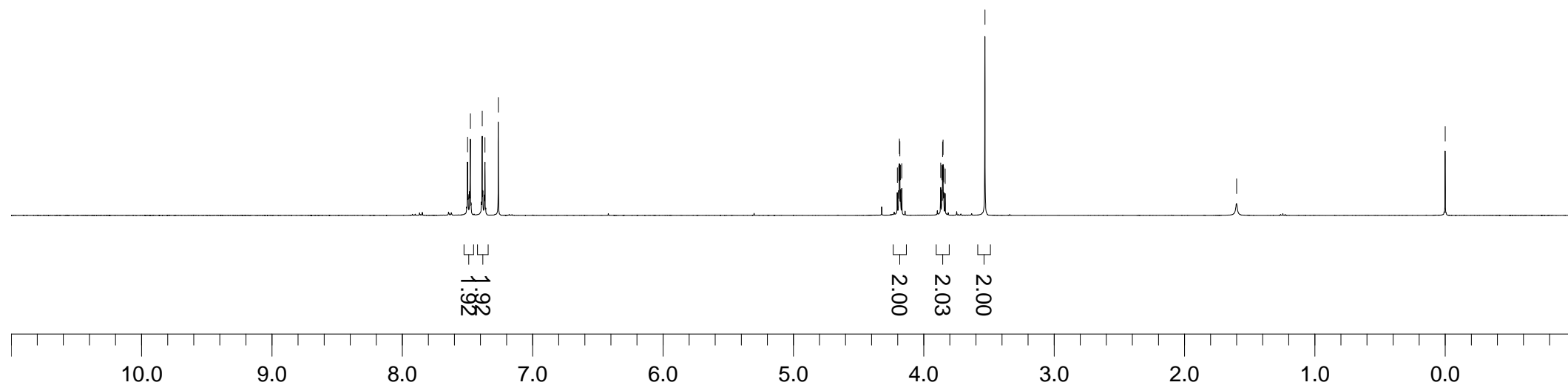
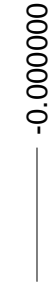
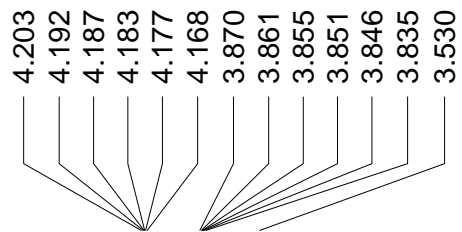
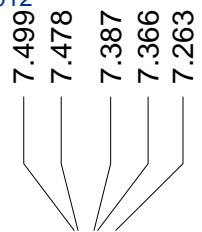


**7c**

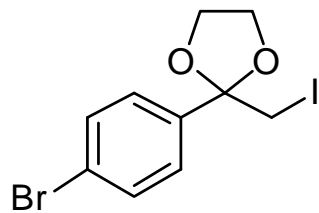




7d



<sup>1</sup>H NMR 100 MHz  
CDCl<sub>3</sub>



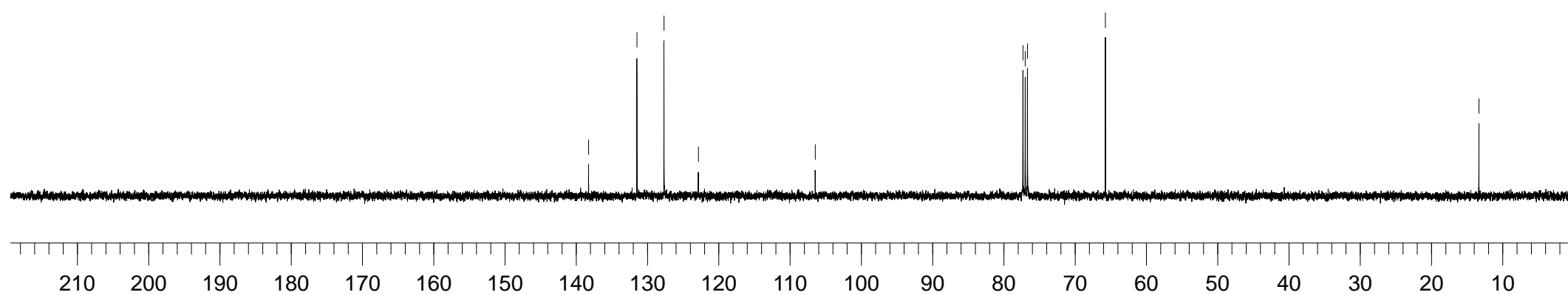
7d

138.271  
131.489  
127.684  
122.862

106.463

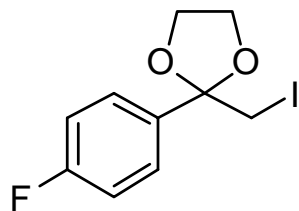
77.317  
77.000  
76.682  
65.745

13.337



<sup>1</sup>H NMR 600 MHz

CDCl<sub>3</sub>

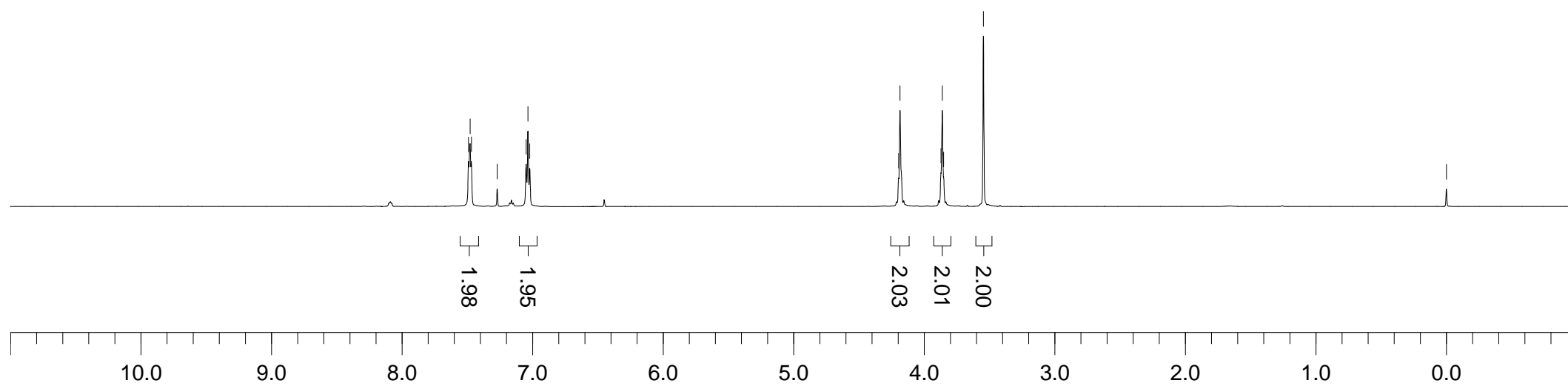


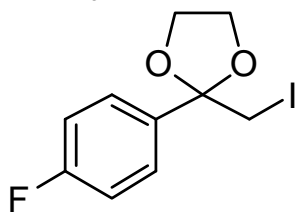
7e

7.491  
7.479  
7.469  
7.271  
7.049  
7.035  
7.022

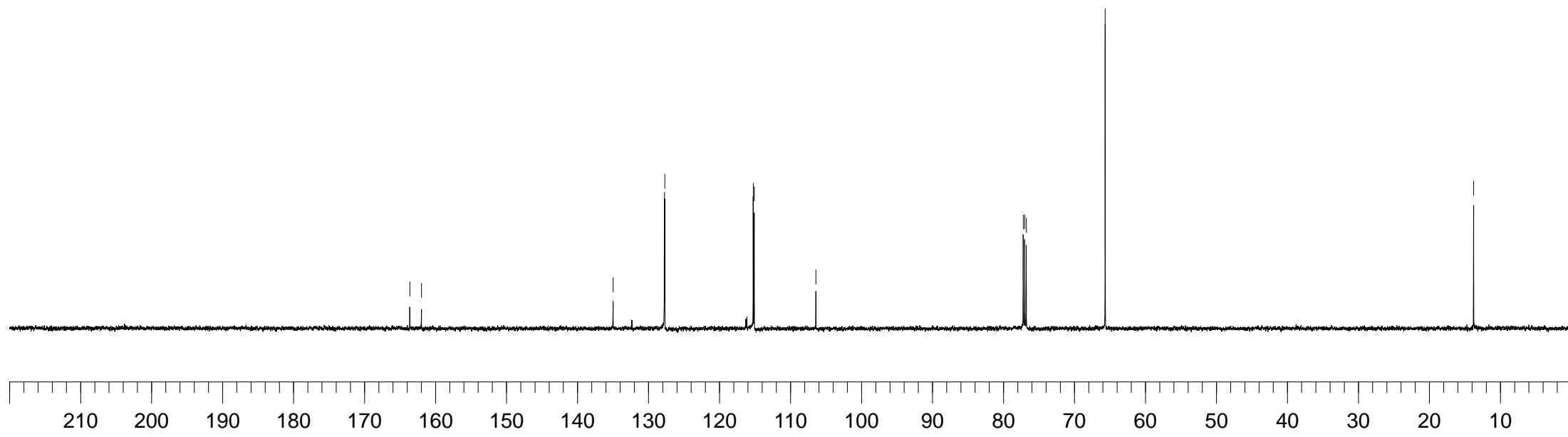
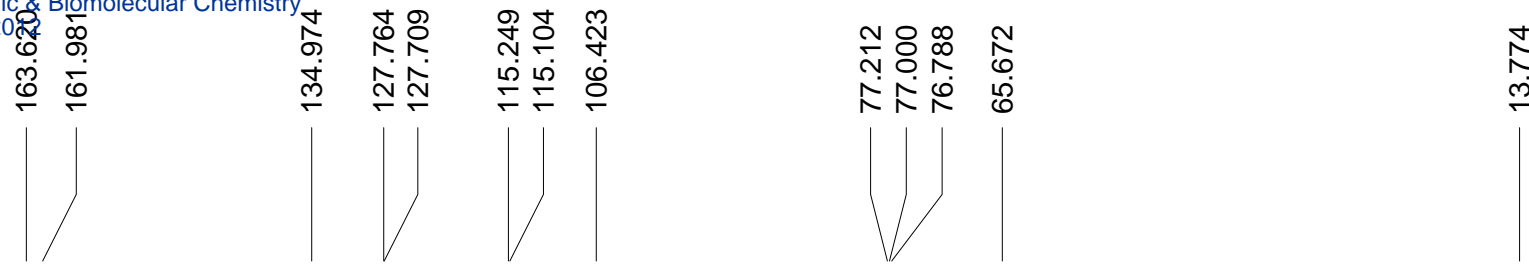
4.196  
4.186  
3.872  
3.862  
3.852  
3.547

0.000000

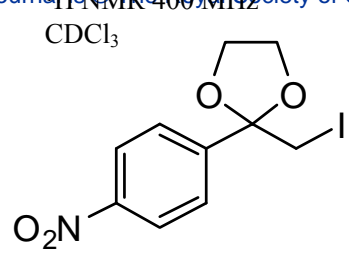




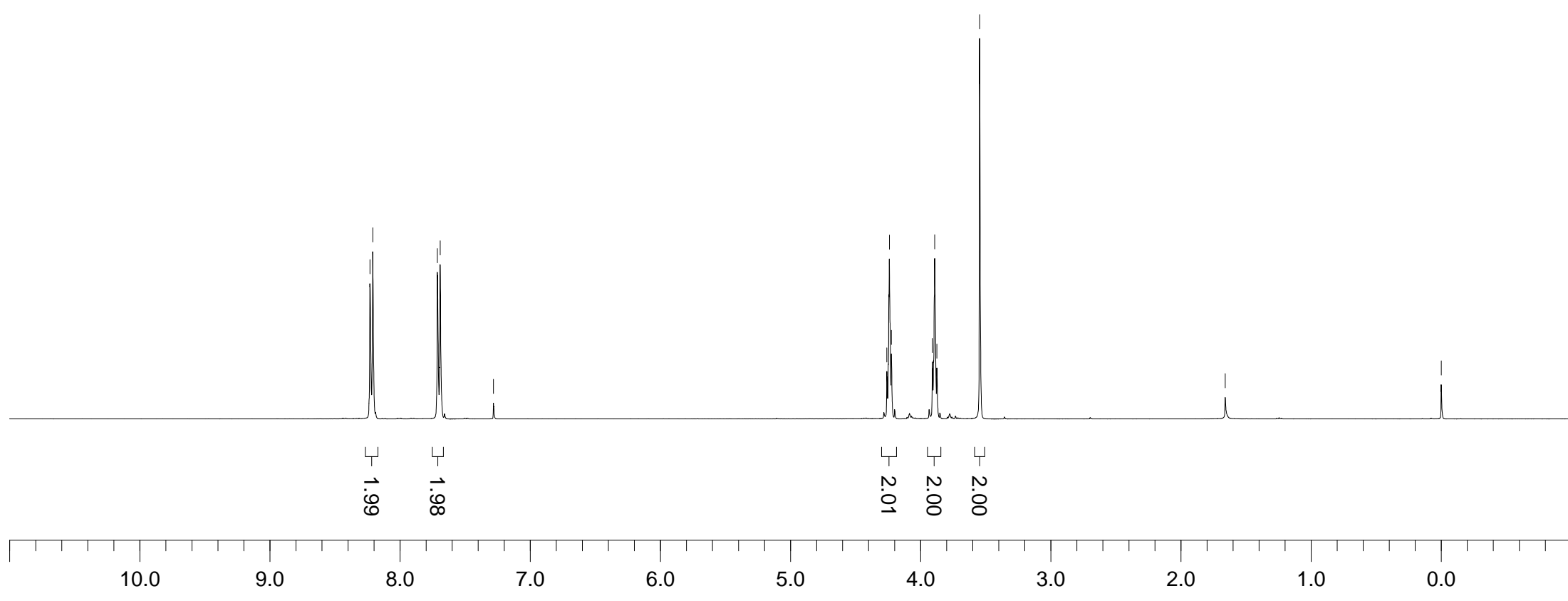
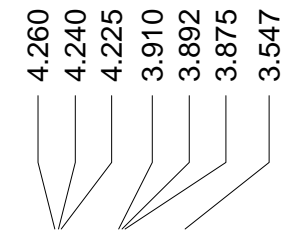
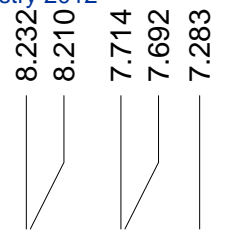
7e

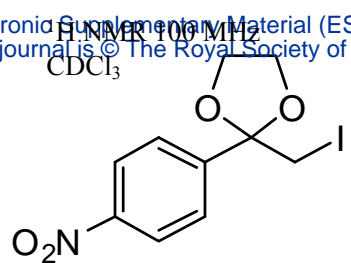






7f





7f

148.075

146.378

127.064

123.529

106.327

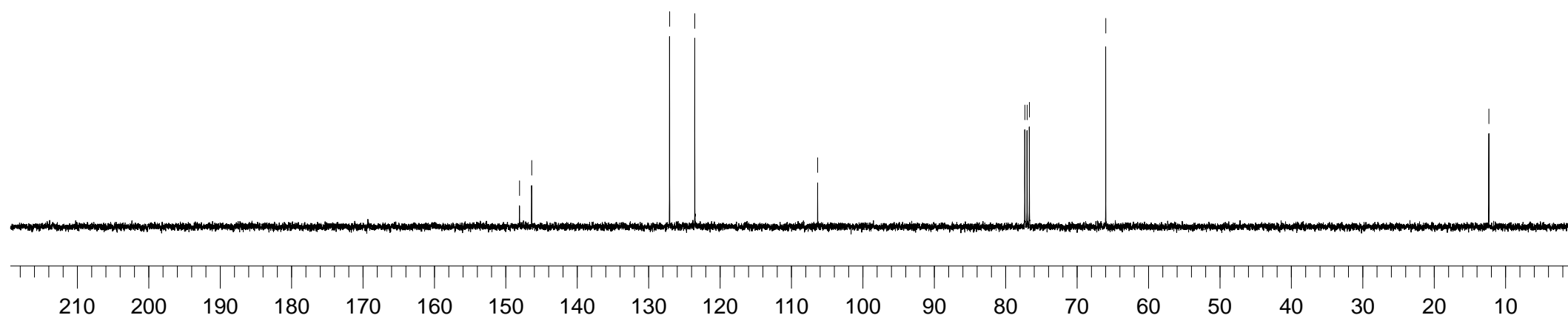
77.317

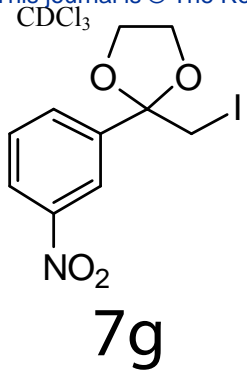
77.000

76.682

65.981

12.344



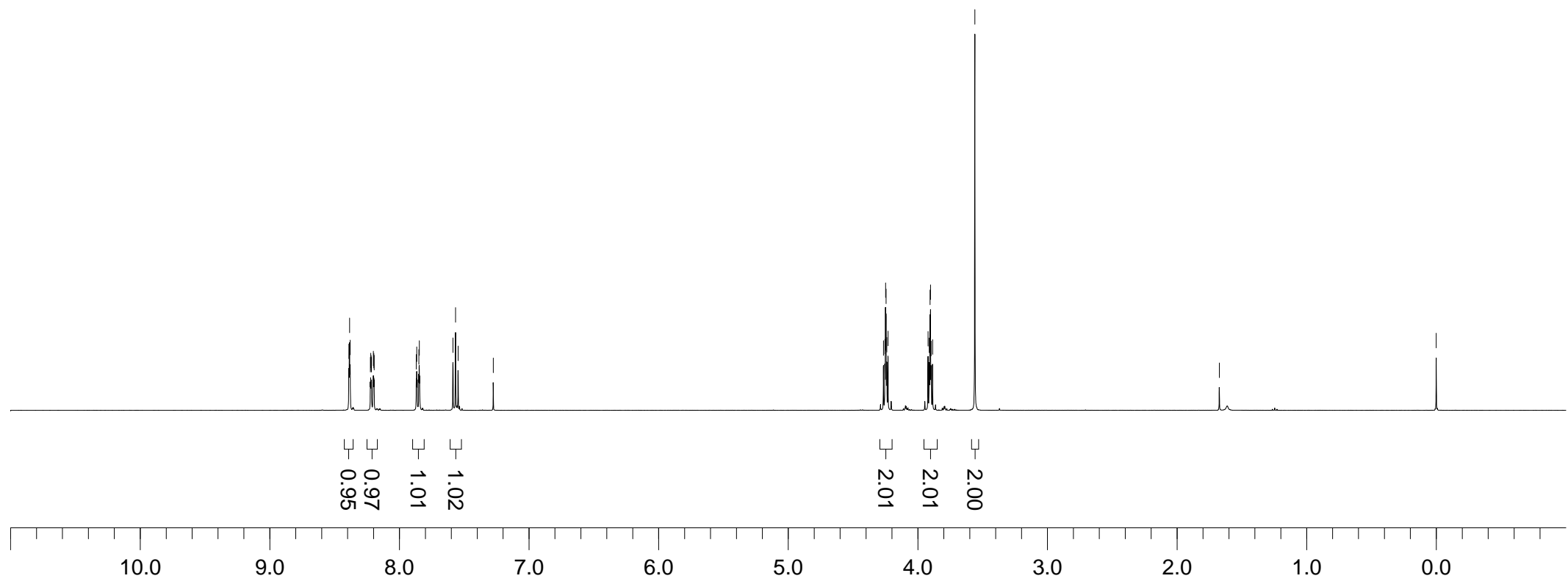


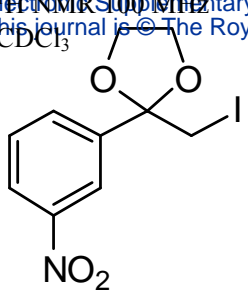
8.390  
8.385  
8.380  
8.224  
8.221  
8.218  
8.216  
8.201  
8.198  
8.195  
7.869  
7.866  
7.850  
7.846  
7.587  
7.567  
7.547  
7.275

4.265  
4.254  
4.249  
4.245  
4.239  
4.230  
3.921  
3.912  
3.906  
3.902  
3.897  
3.886  
3.560

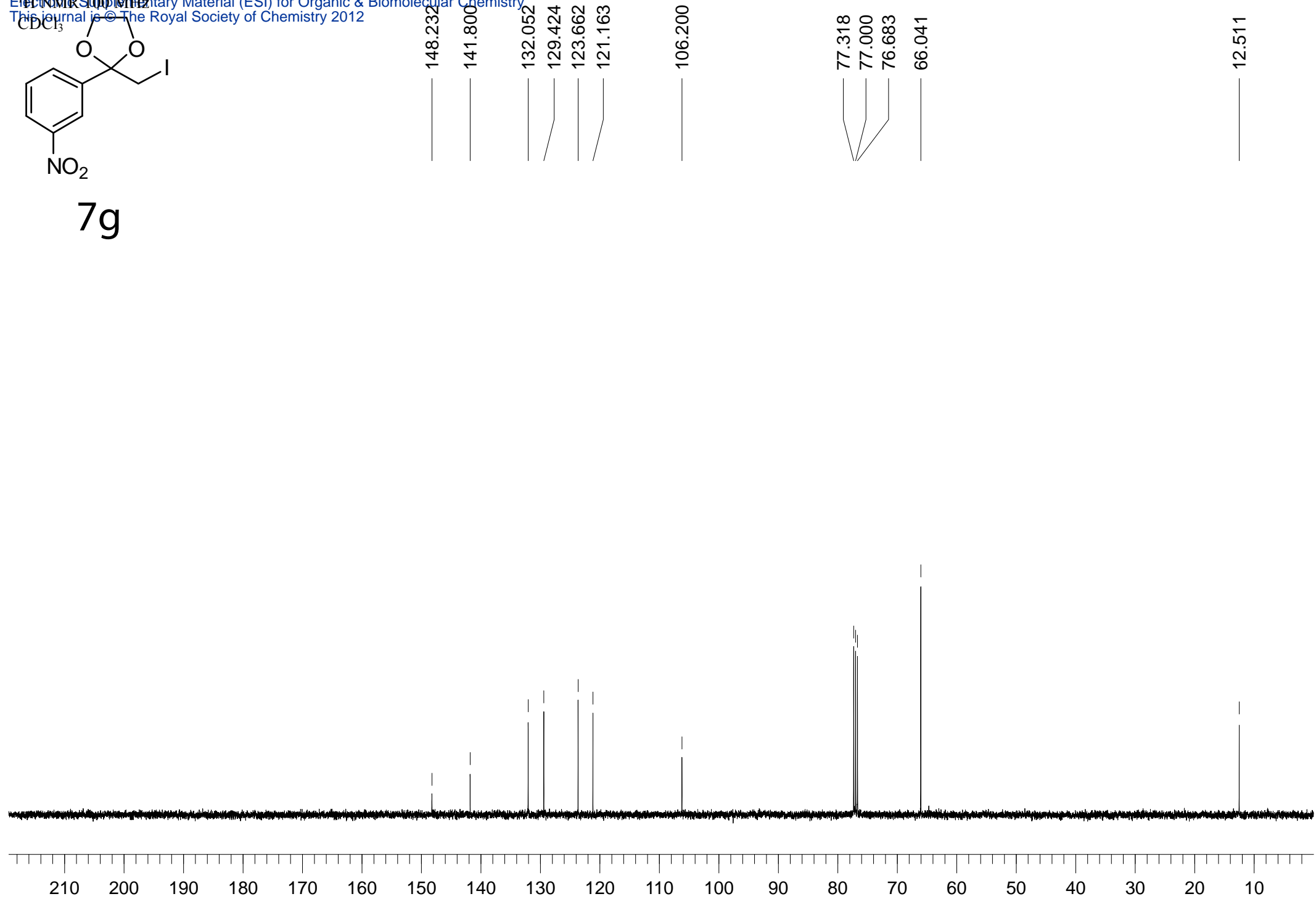
1.674

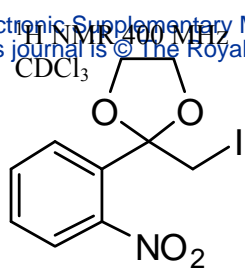
-0.000000



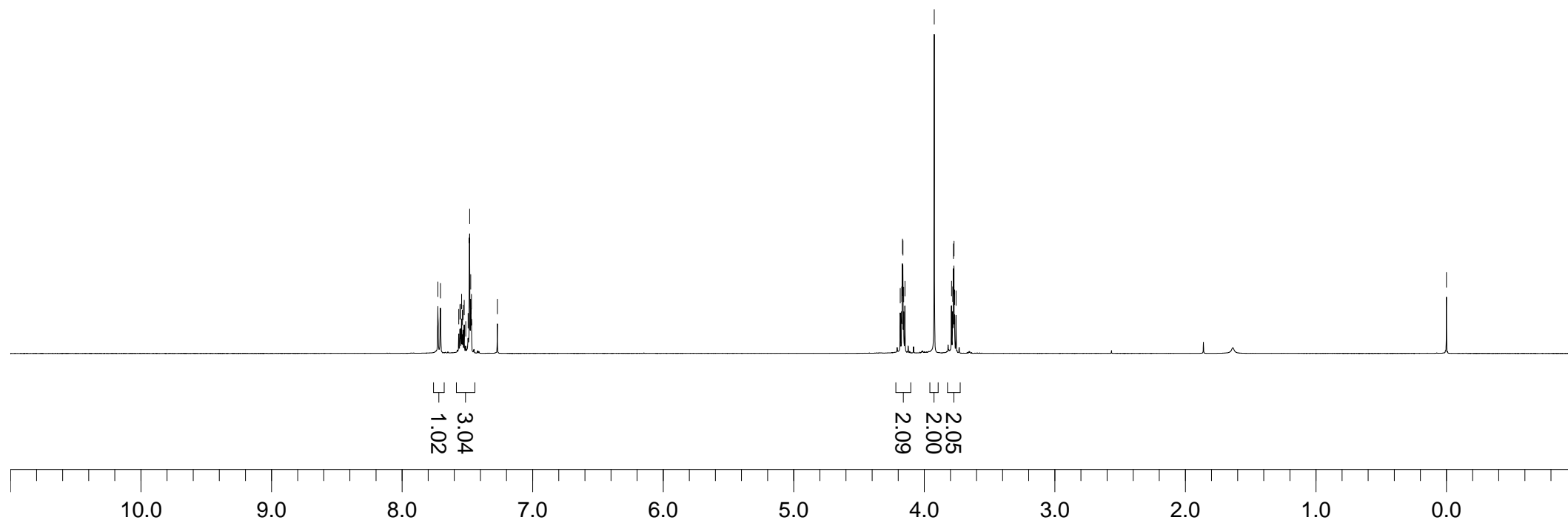
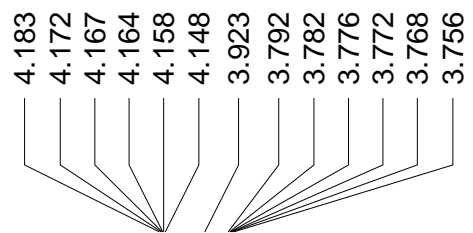
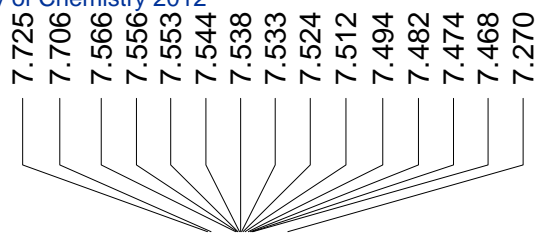


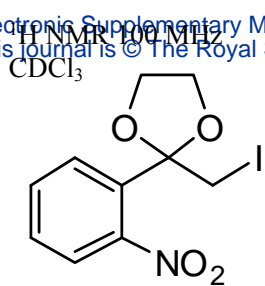
7g





7h





7h

