

## Supporting Information

# Mg(OMe)<sub>2</sub> Promoted Allylic Isomerization of $\gamma$ -Hydroxy- $\alpha$ , $\beta$ -Alkenoic Esters to The Synthesis of $\gamma$ -Ketone Esters

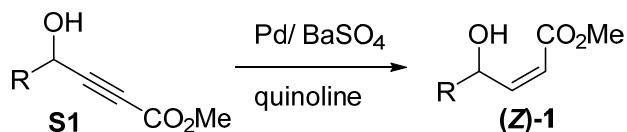
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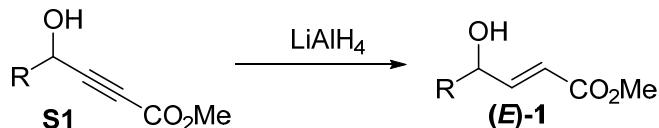
**General Methods.** Unless otherwise noted, all reactions were carried out with dry, freshly distilled solvents under anhydrous conditions, in flame-dried, round bottom flasks with magnetic stirring. Solvent were dried according to established procedures. Yields refer to chromatographically and spectroscopically ( $^1\text{H}$  NMR) homogenous materials. All reactions were monitored by TLC unless otherwise stated. Column chromatography purifications were carried out using silica gel.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral were recorded on 300 MHz and 75 MHz instruments. Data for  $^1\text{H}$  NMR are recorded as follows: chemical shift ( $\delta$ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, coupling constant(s) in Hz, integration). Data for  $^{13}\text{C}$  NMR are reported in terms of chemical shift ( $\delta$ , ppm).

**General procedure for the synthesis of (Z)- $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -alkenoic Esters (Z)-1:**



Lindlar Pd catalyst (5 wt% on BaSO<sub>4</sub>, 10 mg) in combination with quinoline (10  $\mu\text{l}$ ) was added to the solution of  $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -acetylenic ester (2 mmol) in hexane or Et<sub>2</sub>O (5 mL). The suspension was vigorously stirred under H<sub>2</sub> (in balloon) for 2h until the reaction completed (monitored by  $^1\text{H}$  NMR). The catalyst was then removed by filtration through a small portion of Celite®, and washed with hexane. The hexane solution was concentrated in vacuo and used for the next step without purification. It was found that the residual quinoline did not affect on the subsequent allylic isomerization.

**General procedure for the synthesis of (E)- $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -alkenoic Esters (E)-1o:**



To a suspension of LiAlH<sub>4</sub> (1 equiv) in dry THF or diethyl ether was added the  $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -acetylenic ester in THF (or ether) dropwise at 0°C, which was then allowed to stir at room temperature. After the starting material had disappeared (TLC, 2 h), the reaction was quenched with saturated ammonium chloride solution, diluted with ether, filtered over Celite® and the organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>. The residue obtained after removal of the solvent was purified by column chromatography (PE / EA = 9:1 to 5:1) to furnish the desired (E)- $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -alkenoic esters (E)-1o as a colorless liquid.  $^1\text{H}$  NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.44 – 7.27 (m, 5H), 7.05 (dd,  $J$  = 15.6, 4.8 Hz, 1H), 6.18 (dd,  $J$  = 15.6, 1.6 Hz, 1H), 5.36 (s, 1H), 3.73 (s, 3H), 2.26 (d,  $J$  = 3.0 Hz, 1H).  $^{13}\text{C}$  NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  166.9, 148.8, 140.9, 128.9, 128.4, 126.6, 119.8, 73.5, 51.76. <sup>[1]</sup>

**General procedure for the allylic isomerization of 1 to  $\gamma$ -keto esters 2.** To a round-bottom flask was added  $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -alkenoic esters 1 (0.2 mmol), TMEDA (15  $\mu$ L, 0.1 mmol, 50 mol %) and Mg(OMe)<sub>2</sub> (0.134 mmol, 67 mol %) in 2 mL MeOH. After stirring at room temperature for 2 h, the resulting solution was concentrated under vacuum. The residue was purified by chromatography on silica gel (PE / EA = 9:1 to 7:1) to afford the  $\gamma$ -keto esters 2.<sup>[2]</sup>

**Methyl 4-oxoundecanoate (2a):**<sup>[2a]</sup> 91% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  3.68 (s, 3H), 2.72 (t,  $J$  = 6.2 Hz, 2H), 2.58 (t,  $J$  = 9.6, 2H), 2.44 (t,  $J$  = 7.5 Hz, 2H), 1.70 – 1.51 (m, 2H), 1.36 – 1.18 (m, 8H), 0.88 (t,  $J$  = 6.7 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  209.1, 173.3, 51.7, 42.8, 37.0, 31.6, 29.1, 29.0, 27.7, 23.8, 22.6, 14.0. EI-MS (*m/z*): 214 (M<sup>+</sup>), 183 (M<sup>+</sup>-31), 143 (M<sup>+</sup>-71), 127(M<sup>+</sup>-87), 115 (M<sup>+</sup>-99), 57 (M<sup>+</sup>-157).

**Methyl 4-oxo-4-(*m*-tolyl)butanoate (2b):**<sup>[2c]</sup> 88% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (dd,  $J$  = 6.6, 1.2 Hz, 2H), 7.41 – 7.30 (m, 2H), 3.71 (s, 3H), 3.31 (t,  $J$  = 6.7 Hz, 2H), 2.77 (t,  $J$  = 6.7 Hz, 2H), 2.41 (s, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  198.2, 173.4, 138.3, 136.5, 133.9, 128.5, 128.4, 125.2, 51.8, 33.4, 28.0, 21.3. EI-MS (*m/z*): 206 (M<sup>+</sup>), 175 (M<sup>+</sup>-31), 119 (M<sup>+</sup>-87), 91 (M<sup>+</sup>-115).

**Methyl 4-oxo-4-(*p*-tolyl)butanoate (2c):**<sup>[2b]</sup> 92% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.88 (d,  $J$  = 8.2 Hz, 1H), 7.26 (d,  $J$  = 7.9 Hz, 1H), 3.70 (s, 1H), 3.30 (t,  $J$  = 6.7 Hz, 1H), 2.76 (t,  $J$  = 6.7 Hz, 1H), 2.41 (s, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 173.4, 144.0, 134.0, 129.2, 128.1, 51.7, 33.2, 28.0, 21.6. EI-MS (*m/z*): 206 (M<sup>+</sup>), 175 (M<sup>+</sup>-31), 119 (M<sup>+</sup>-87), 91 (M<sup>+</sup>-115).

**Methyl 4-(3-chlorophenyl)-4-oxobutanoate (2d):** 84% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (s, 1H), 7.86 (d,  $J$  = 7.6 Hz, 1H), 7.55 (d,  $J$  = 7.9 Hz, 1H), 7.43 (q,  $J$  = 7.4 Hz, 1H), 3.71 (s, 3H), 3.29 (t,  $J$  = 6.6 Hz, 2H), 2.77 (t,  $J$  = 6.6 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  196.8, 173.1, 138.1, 135.0, 133.1, 130.0, 128.2, 126.1, 51.9, 33.5, 27.9. HRMS (ESI) *m/z* calcd for C<sub>11</sub>H<sub>11</sub>ClO<sub>3</sub> [M + H]<sup>+</sup>: 227.0469, found *m/z* 227.0472.

**Methyl 4-(4-chlorophenyl)-4-oxobutanoate (2e):**<sup>[2c]</sup> 94% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.98 – 7.87 (m, 2H), 7.48 – 7.39 (m, 2H), 3.71 (s, 3H), 3.29 (t,  $J$  = 6.6 Hz, 2H), 2.77 (t,  $J$  = 6.6 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 173.4, 144.0, 134.0, 129.2, 128.1, 51.8, 33.2, 28.0, 21.6. EI-MS (*m/z*): 228 (M<sup>+</sup>), 226 (M<sup>+</sup>), 197 (M<sup>+</sup>-31), 195 (M<sup>+</sup>-31), 141 (M<sup>+</sup>-87), 139 (M<sup>+</sup>-87), 113 (M<sup>+</sup>-115), 111 (M<sup>+</sup>-115).

**Methyl 4-(2-chlorophenyl)-4-oxobutanoate (2f):**<sup>[2c]</sup> 79% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.59 – 7.53 (m, 1H), 7.46 – 7.38 (m, 2H), 7.38 – 7.30 (m, 1H), 3.71 (s, 3H), 3.27 (t,  $J$  = 6.6 Hz,

2H), 2.78 (t,  $J$  = 6.6 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  201.1, 173.0, 138.8, 131.9, 130.1, 130.6, 129.2, 126.9, 51.8, 37.6, 28.3. EI-MS ( $m/z$ ): 228 ( $\text{M}^+$ ), 226 ( $\text{M}^+$ ), 197 ( $\text{M}^+-31$ ), 195 ( $\text{M}^+-31$ ), 141 ( $\text{M}^+-87$ ), 139 ( $\text{M}^+-87$ ), 113 ( $\text{M}^+-115$ ), 111 ( $\text{M}^+-115$ ).

**Methyl 4-(naphthalen-2-yl)-4-oxobutanoate (2g):** <sup>[2c]</sup> 94% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (s, 1H), 8.04 (dd,  $J$  = 8.6, 1.7 Hz, 1H), 7.97 (d,  $J$  = 7.8 Hz, 1H), 7.93 – 7.82 (m, 2H), 7.64 – 7.50 (m, 2H), 3.73 (s, 3H), 3.47 (t,  $J$  = 6.7 Hz, 2H), 2.83 (t,  $J$  = 6.7 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  198.0, 173.4, 135.7, 133.9, 132.5, 129.8, 129.6, 128.5, 128.4, 127.8, 126.8, 123.7, 51.8, 33.5, 28.1. EI-MS ( $m/z$ ): 242 ( $\text{M}^+$ ), 211 ( $\text{M}^+-31$ ), 155 ( $\text{M}^+-87$ ), 127 ( $\text{M}^+-115$ ).

**Methyl 4-(4-methoxyphenyl)-4-oxobutanoate (2h):** <sup>[2c]</sup> 96% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 – 7.91 (m, 2H), 6.98 – 6.88 (m, 2H), 3.87 (s, 3H), 3.71 (s, 3H), 3.28 (t,  $J$  = 6.7 Hz, 2H), 2.75 (t,  $J$  = 6.7 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  196.5, 173.5, 163.5, 130.3, 129.6, 113.7, 55.4, 51.8, 33.0, 28.1. EI-MS ( $m/z$ ): 222 ( $\text{M}^+$ ), 191 ( $\text{M}^+-31$ ), 135 ( $\text{M}^+-87$ ).

**Methyl 4-(2-methoxyphenyl)-4-oxobutanoate (2i):** <sup>[2c]</sup> 75% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (dd,  $J$  = 7.7, 1.7 Hz, 1H), 7.54 – 7.39 (m, 1H), 7.06 – 6.91 (m, 2H), 3.92 (s, 3H), 3.70 (s, 3H), 3.33 (t,  $J$  = 6.7 Hz, 2H), 2.71 (t,  $J$  = 6.7 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  199.8, 173.6, 158.9, 133.7, 130.5, 127.4, 120.6, 111.5, 55.5, 51.7, 38.7, 28.5. EI-MS ( $m/z$ ): 222 ( $\text{M}^+$ ), 191 ( $\text{M}^+-31$ ), 135 ( $\text{M}^+-87$ ).

**Methyl 4-(furan-2-yl)-4-oxobutanoate (2j):** <sup>[2d]</sup> 92% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59 (d,  $J$  = 1.0 Hz, 1H), 7.23 (d,  $J$  = 3.5 Hz, 1H), 6.55 (dd,  $J$  = 3.5, 1.7 Hz, 1H), 3.70 (s, 3H), 3.18 (t,  $J$  = 6.8 Hz, 2H), 2.75 (t,  $J$  = 6.8 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  187.2, 173.1, 152.4, 146.3, 117.0, 112.2, 51.8, 33.1, 27.6. EI-MS ( $m/z$ ): 182 ( $\text{M}^+$ ), 167 ( $\text{M}^+-15$ ), 151 ( $\text{M}^+-31$ ), 123 ( $\text{M}^+-59$ ), 95 ( $\text{M}^+-87$ ).

**(E)-Methyl 4-oxo-6-phenylhex-5-enoate (2k):** <sup>[2e]</sup> 95% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.59 (d,  $J$  = 16.4 Hz, 1H), 7.56 – 7.52 (m, 2H), 7.39 (dd,  $J$  = 4.0, 2.4 Hz, 3H), 6.76 (d,  $J$  = 16.2 Hz, 1H), 3.70 (s, 3H), 3.02 (t,  $J$  = 6.7 Hz, 2H), 2.70 (t,  $J$  = 6.7 Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  197.9, 173.2, 142.8, 134.3, 130.5, 128.9, 128.2, 125.7, 51.7, 35.1, 27.8. EI-MS ( $m/z$ ): 218 ( $\text{M}^+$ ), 187 ( $\text{M}^+-31$ ), 159 ( $\text{M}^+-59$ ), 131 ( $\text{M}^+-87$ ), 103 ( $\text{M}^+-115$ ), 77 ( $\text{M}^+-141$ ).

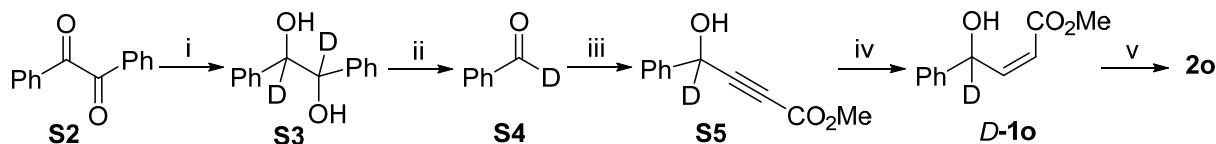
**Methyl 4-cyclohexyl-4-oxobutanoate (2l):** <sup>[2f]</sup> 80% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.67 (s, 3H), 2.76 (t,  $J$  = 6.6 Hz, 2H), 2.58 (t,  $J$  = 6.4 Hz, 2H), 2.38 (tt,  $J$  = 11.3, 3.4 Hz, 1H), 1.94 – 1.60 (m, 6H), 1.45 – 1.11 (m, 6H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.0, 173.4, 51.7, 50.7, 34.9, 28.4, 27.6, 25.8, 25.6. EI-MS ( $m/z$ ): 198 ( $\text{M}^+$ ), 167 ( $\text{M}^+-31$ ), 115 ( $\text{M}^+-83$ ), 111 ( $\text{M}^+-87$ ), 83 ( $\text{M}^+-115$ ).

**Methyl 4-oxodecanoate (2m):** [2f] 78% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.68 (s, 3H), 2.73 (t,  $J = 6.4$  Hz, 2H), 2.59 (t,  $J = 6.2$  Hz, 2H), 2.45 (t,  $J = 7.5$  Hz, 2H), 1.57 (q,  $J = 6.9$  Hz, 2H), 1.35 – 1.22 (m, 6H), 0.88 (t,  $J = 6.6$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  209.1, 173.3, 51.6, 42.8, 37.0, 31.6, 28.9, 27.7, 23.8, 22.5, 14.0. EI-MS ( $m/z$ ): 200 ( $\text{M}^+$ ), 169 ( $\text{M}^+ - 31$ ), 143 ( $\text{M}^+ - 57$ ), 115 ( $\text{M}^+ - 85$ ), 43 ( $\text{M}^+ - 157$ ).

**Methyl 4-oxooctanoate (2n):** [2g] 66% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.68 (s, 3H), 2.73 (t,  $J = 6.4$  Hz, 2H), 2.58 (t,  $J = 9.6, 3.6$  Hz, 2H), 2.45 (t,  $J = 7.5$  Hz, 2H), 1.58 (dt,  $J = 15.3, 7.4$  Hz, 2H), 1.32 (td,  $J = 14.8, 7.3$  Hz, 2H), 0.91 (t,  $J = 7.3$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  209.1, 173.3, 51.7, 42.4, 36.9, 27.7, 25.8, 22.2, 13.8. EI-MS ( $m/z$ ): 172 ( $\text{M}^+$ ), 141 ( $\text{M}^+ - 31$ ), 115 ( $\text{M}^+ - 57$ ), 85 ( $\text{M}^+ - 87$ ), 57 ( $\text{M}^+ - 115$ ).

**Methyl 4-oxo-4-phenylbutanoate (2o):** [2a] 91% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 – 7.92 (m, 2H), 7.62 – 7.53 (m, 1H), 7.53 – 7.40 (m, 2H), 3.71 (s, 3H), 3.33 (t,  $J = 6.6$  Hz, 2H), 2.78 (t,  $J = 6.6$  Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  198.0, 173.3, 136.5, 133.2, 128.6, 128.0, 51.8, 33.4, 28.0. EI-MS ( $m/z$ ): 192 ( $\text{M}^+$ ), 161 ( $\text{M}^+ - 31$ ), 105 ( $\text{M}^+ - 87$ ), 77 ( $\text{M}^+ - 115$ ).

### Deuteration Experiment:



**Reduction of Benzil (**S2**):** Following the similar procedure in literature with minor modification:<sup>[3a-b]</sup> firstly the Benzil (**S2**, 1.05g, 5 mmol) was dissolved in a mixture solvent, which containing dried THF (10 mL) and D<sub>2</sub>O (0.5 mL). At 0°C, NaBD<sub>4</sub> (250 mg, 6.2 mmol, >98% D isotopic purity) was added in portion-wise over one hour. The mixture was stirred until No **S2** left, monitored via TLC. After general workup, the reaction mixture was extracted with EtOAc, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated to give the product **S3** as a white solid in nearly quantity yield, which was pure enough for the next step. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.31 – 7.26 (m, 6H), 7.24 – 7.19 (m, 4H), 2.95 (s, 1H, *dl*), 2.30 (s, 1H, *Meso*). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 139.6, 128.2, 128.1, 128.0, 127.9, 127.0, 126.9.

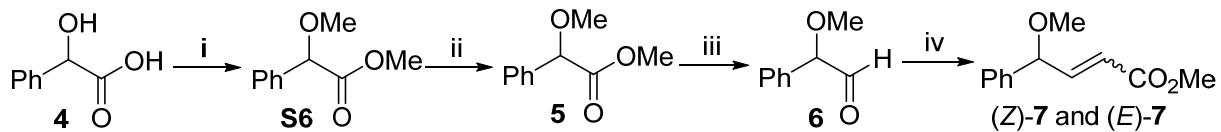
**Synthesis of Deuteriobenzaldehyde **S4**:**<sup>[3b]</sup> The diol **S3** was oxidized with NaIO<sub>4</sub> in ethanol overnight at room temperature. After the reaction was complete (monitored by TLC), the mixture was treated with general workup. Deuteriobenzaldehyde **S4** was purified over silica gel.

**Synthesis of  $\gamma$ -hydroxy- $\alpha$ , $\beta$ -acetylenic ester **S5**:** Alkynylation of deuteriobenzaldehyde **S4** via treatment with n-BuLi in THF at -78°C afforded **S5** in 70% yield. 91% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.50 – 7.46 (m, 2H), 7.41 – 7.32 (m, 3H), 3.76 (s, 3H), 3.62 (s, 1H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 153.8, 138.4, 128.7, 128.6, 126.5, 86.8, 77.2, 63.9, 63.6, 63.3, 52.8.

Lindlar reduction of **S5** was carried following the general procedure for the synthesis of (*Z*)- $\gamma$ -hydroxy- $\alpha$ , $\beta$ -alkenoic Esters (*Z*)-**1**. Without using quinolone, Pd catalyst was filtrated off until the hydrogenation complete (monitored via <sup>1</sup>H NMR). The organic solvent was evaporated under vacuum to afford *D*-**1o** which was used directly in the isomerization step. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.49 – 7.45 (m, 2H), 7.39 – 7.26 (m, 3H), 6.45 (d, *J* = 12 Hz, 1H), 5.89 (d, *J* = 12 Hz, 1H), 3.77 (s, 3H).

Under standard condition as in the general procedure for the allylic isomerization of **1** to  $\gamma$ -keto esters **2**, the allylic isomerization was carried out, and only afforded compound **2o**. No deuterated product was observed.

### Synthesis of Methyl $\gamma$ -Methoxy- $\gamma$ -phenylbutenate 7:



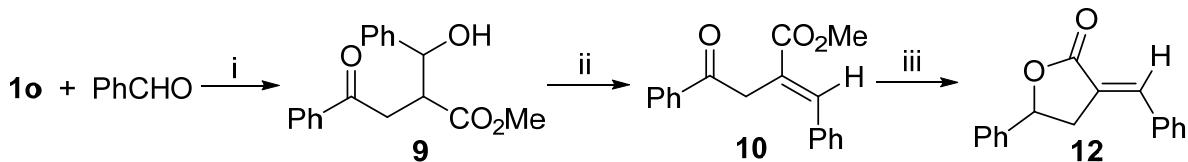
The suspension of (3.04g, 20 mmol) of the Mandelic acid (**4**) in 25mL of methanol was cooled down to 0 °C, 6mL of thionyl chloride was slowly dropped in. It was refluxed for 2h and then stirred for 8h at room temperature. The solvent was concentrated in vacuo and dried on air to give **S6** as a white solid (quantitative).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33-7.44 (m, 5H), 5.19 (s, 3H), 3.77 (s, 3H). <sup>[4a]</sup>

Sodium hydride (240 mg, 6mmol) was suspended in THF (5 mL) and cooled to 0 C, to which the solution of methyl mandelate **S6** (0.5 mg, 5mol) in THF (2 mL) was added dropwise. After stirring for 0.5h, methyl iodide (0.8 g, 6 mmol) was added and the reaction mixture was warmed to room temperature. To the reaction mixture was quenched by  $\text{NH}_4\text{Cl}$  solution (10mL) after 4h. The resulting mixture was extracted with diethyl ether (50mL) twice. The combined organic layer was washed with brine (5mL), dried over  $\text{Mg}_2\text{SO}_4$ , and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to give **5** colorless oil (51% yield).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26-7.42 (m, 5H), 4.78 (s, 1H), 3.73 (s, 3H), 3.41 (s, 3H). <sup>[4b]</sup>

To the solution of **5** (0.4 g, 2mmol) in  $\text{Et}_2\text{O}$  (10mL) at -78 °C, 1M DIBAL-H solution in hexane (2 mL) was added dropwise and the reaction mixture was allowed to stir at -78 °C for 2h. The reaction was quenched with MeOH (0.6 mL) and allowed to warm to room temperature. The mixture was treated with brine (10 mL) and extracted with  $\text{Et}_2\text{O}$  (50 mL X 3). The combined organic phase was washed with brine (10 mL) and dried over  $\text{Na}_2\text{SO}_4$ . The crude aldehyde was obtained after filtration and concentration in vacuo and was used for the next step without further purification.

Solution of methyl triphenylphosphine acetate (0.67 g, 2mmol) in MeOH (5 mL) was added to the above obtained aldehyde and stirred at 0°C overnight. After the addition of water (10mL), the product was extracted with  $\text{Et}_2\text{O}$ , dried over  $\text{Na}_2\text{SO}_4$  and purified by flash chromatography on gel (PE / EA = 25:1) to give (*Z*)-7 and (*E*)-7 as colorless oil. (*Z*)-7: 58% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 – 7.40 (m, 2H), 7.40 – 7.19 (m, 3H), 6.30 (dd,  $J$  = 11.6, 8.8 Hz, 1H), 5.98 (d,  $J$  = 8.9 Hz, 1H), 5.85 (dd,  $J$  = 11.6, 1.0 Hz, 1H), 3.72 (s, 3H), 3.32 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  166.1, 149.0, 140.1, 128.4, 127.8, 126.7, 119.3, 76.6, 56.2, 51.2. (*E*)-7: 29% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44 – 7.18 (m, 5H), 6.97 (dd,  $J$  = 15.7, 5.4 Hz, 1H), 6.10 (dd,  $J$  = 15.7, 1.5 Hz, 1H), 4.78 (dd,  $J$  = 5.4, 1.2 Hz, 1H), 3.71 (s, 3H), 3.32 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 147.5, 138.8, 128.6, 128.2, 128.3, 120.3, 82.3, 56.6, 51.5. <sup>[4c]</sup>

**Synthesis of (*E*)- $\alpha$ -alkenyl- $\gamma$ -butyrolactone.**



**Tandem allylic isomerization-Aldol reaction to 9.** To a round-bottom flask was added Mg(OMe)<sub>2</sub> (5 mL, 1.34 mmol, 67 mol %), TMEDA (150  $\mu$ L, 1 mmol, 50 mol %), benzaldehyde (2 mmol) and  $\gamma$ -hydroxy- $\alpha$ ,  $\beta$ -alkenoic esters **1o** (2 mmol). After stirring at room temperature overnight, the mixture was quenched by addition of 1N HCl solution, extracted with ethyl acetate, and washed with brine. The combined organic phase was dried over sodium sulfate and concentrated under vacuum. The crude product was purified by flash chromatography on silica gel (PE / EA = 12:1) to give **9** as a white solid, 89 % yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 – 7.81 (m, 2H), 7.59 – 7.50 (m, 1H), 7.47 – 7.38 (m, 3H), 7.38 – 7.32 (m, 5H), 7.32 – 7.26 (m, 1H), 5.01 (d, *J* = 7.1 Hz, 1H), 3.69 (s, 3H), 3.49 – 3.40 (m, 1H), 3.40 – 3.23 (m, 2H), 3.11 (dd, *J* = 17.7, 4.5 Hz, 1H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 174.7, 141.2, 136.3, 133.4, 128.64, 128.58, 128.2, 128.0, 126.3, 74.5, 52.2, 47.9, 37.8. ESI-MS (M-OH)<sup>+</sup>: 281. [<sup>5a</sup>]

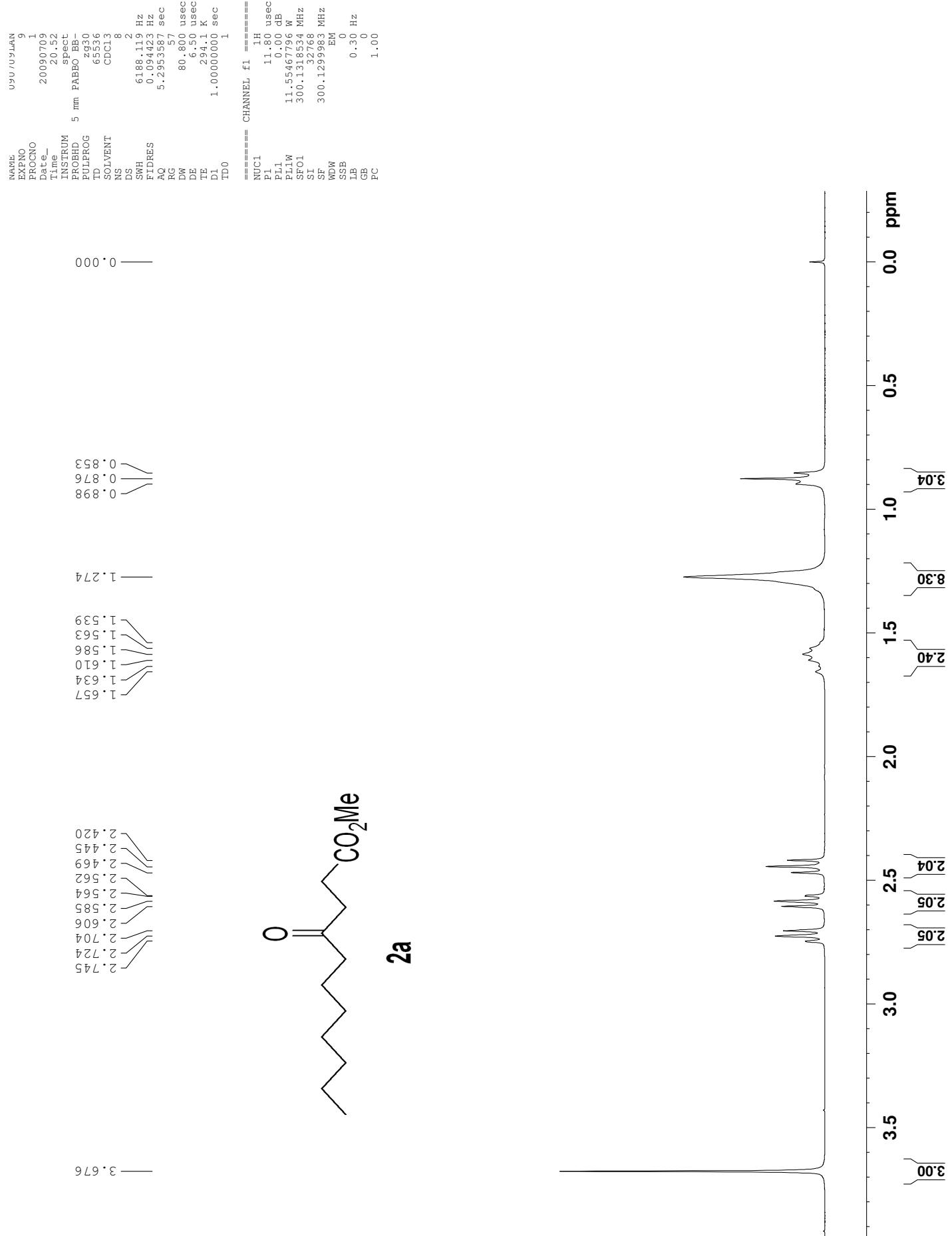
**Dehydration of Reformatsky-like adduct 9 to 10.** To a solution of **9** (107 mg, 0.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (5 mL) at room temperature (1.1 ml, 30 equ.) was added TFA in dropwise. The reaction mixture was stirred overnight and then quenched with NaHCO<sub>3</sub> (sat. solution), extracted with ethyl acetate, washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The crude product was purified by flash chromatography on silica gel (PE / EA = 30:1) to give **10** as pale yellow oil, 98 % yield based on the recovered starting material (32%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.09 – 7.94 (m, 3H), 7.64 – 7.54 (m, 1H), 7.54 – 7.43 (m, 2H), 7.36 – 7.27 (m, 5H), 4.21 (s, 2H), 3.79 (s, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  197.3, 167.9, 142.3, 136.6, 135.2, 133.3, 128.7, 128.64, 128.60, 128.3, 127.0, 52.2, 38.1, 29.7. EI-MS (*m/z*): 280 (M<sup>+</sup>), 249 (M<sup>+</sup>-31), 105 (M<sup>+</sup>-175), 77 (M<sup>+</sup>-203). [<sup>5b</sup>]

**Synthesis of (*E*)- $\alpha$ -benzylidene- $\gamma$ -phenyl butyrolactone 12.** [<sup>6</sup>] After dissolving **10** (64 mg, 0.3 mmol) in 5 ml of MeOH, NaBH<sub>4</sub> (34.2 mg, 3 equiv) was added to the stirred solution in small portions at 0 °C. While the reduction completed (monitored by TLC, 0.5 h), 1 ml 1N HCl was then added to quench the reaction. The mixture was stirred for another half hour, extracted with EtOAc (5 ml  $\times$  3) and washed with brine, then dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The crude product was purified by flash chromatography on silica gel (PE / EA = 30:1) to give afford **7** as a white solid, 70% yield. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.64 (t, *J* = 2.9 Hz,

1H), 7.52 – 7.46 (m, 2H), 7.46 – 7.38 (m, 4H), 7.38 – 7.35 (m, 3H), 7.35 – 7.33 (m, 1H), 5.61 (dd,  $J$  = 8.3, 6.0 Hz, 1H), 3.70 (ddd,  $J$  = 17.5, 8.3, 2.7 Hz, 1H), 3.16 (ddd,  $J$  = 17.5, 5.9, 3.0 Hz, 1H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.9, 140.2, 136.9, 134.5, 130.0, 129.9, 128.9, 128.8, 128.5, 125.3, 124.0, 78.1, 36.5. HRMS (ESI) m/z calcd for  $\text{C}_{17}\text{H}_{14}\text{O}_2$  [M + H] $^+$ : 251.1067, found m/z 251.1071.  $^{[5\text{c}]}$

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- [1] K. Burgess, J. Cassidy and I. Henderson, *J. Org. Chem.* 1991, **56**, 2050.
- [2] (a) O. G. Kulinkovich and V. L. Sorokin, *Synthesis* 1994, **4**, 361. (b) E. A. Jo and C. H. Jun, *Eur. J. Org. Chem.* 2006, 2504. (c) W. G. Dauben and H. Tilles, *J. Org. Chem.* 1950, **15**, 785. (d) Y. J. Liu and Y. M. Zhang, *Tetrahedron* 2003, **59**, 8429. (e) J. C. del Amo, M. J. Mancheno, M. Gomez-Gallego and M. A. Sierra, *Organometallics* 2004, **23**, 5021. (f) I. Ryu, K. Kusano, H. Yamazaki and N. Sonoda, *J. Org. Chem.* 1991, **56**, 5003. (g) H. Cherkaoui, M. Soufiaoui and R. Grée, *Tetrahedron* 2001, **57**, 2379. (h) K. Miura, N. Fujisawa, H. Saito, D. Wang and A. Hosomi, *Org. Lett.* 2001, **3**, 2591.
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- [6] Un-optimized conditions.



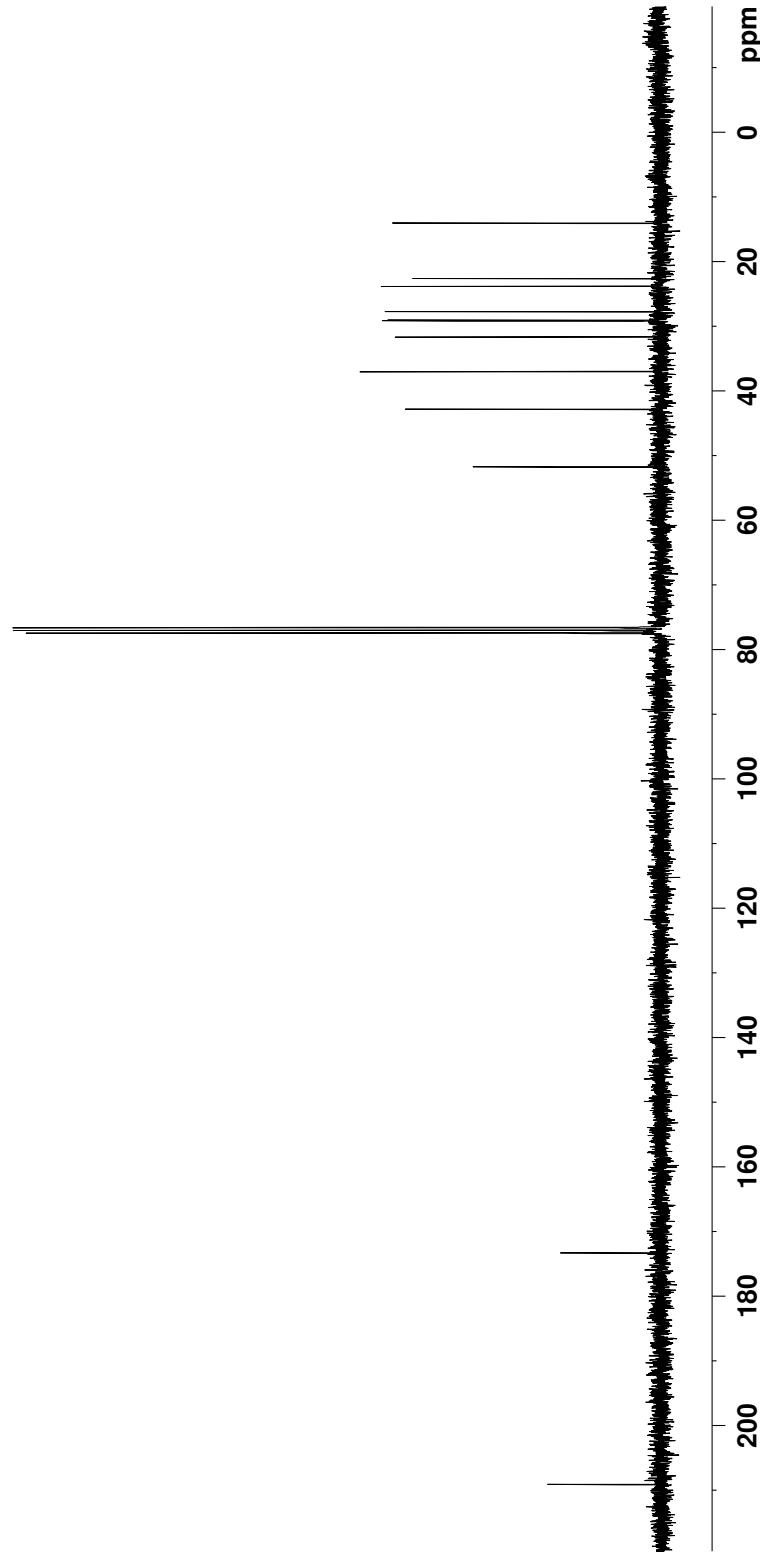
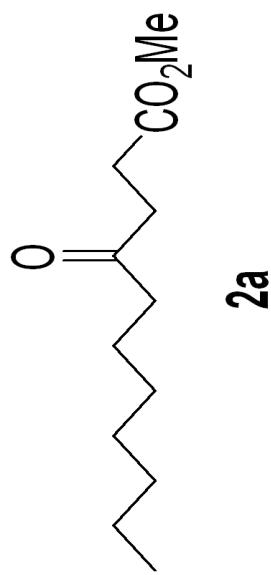
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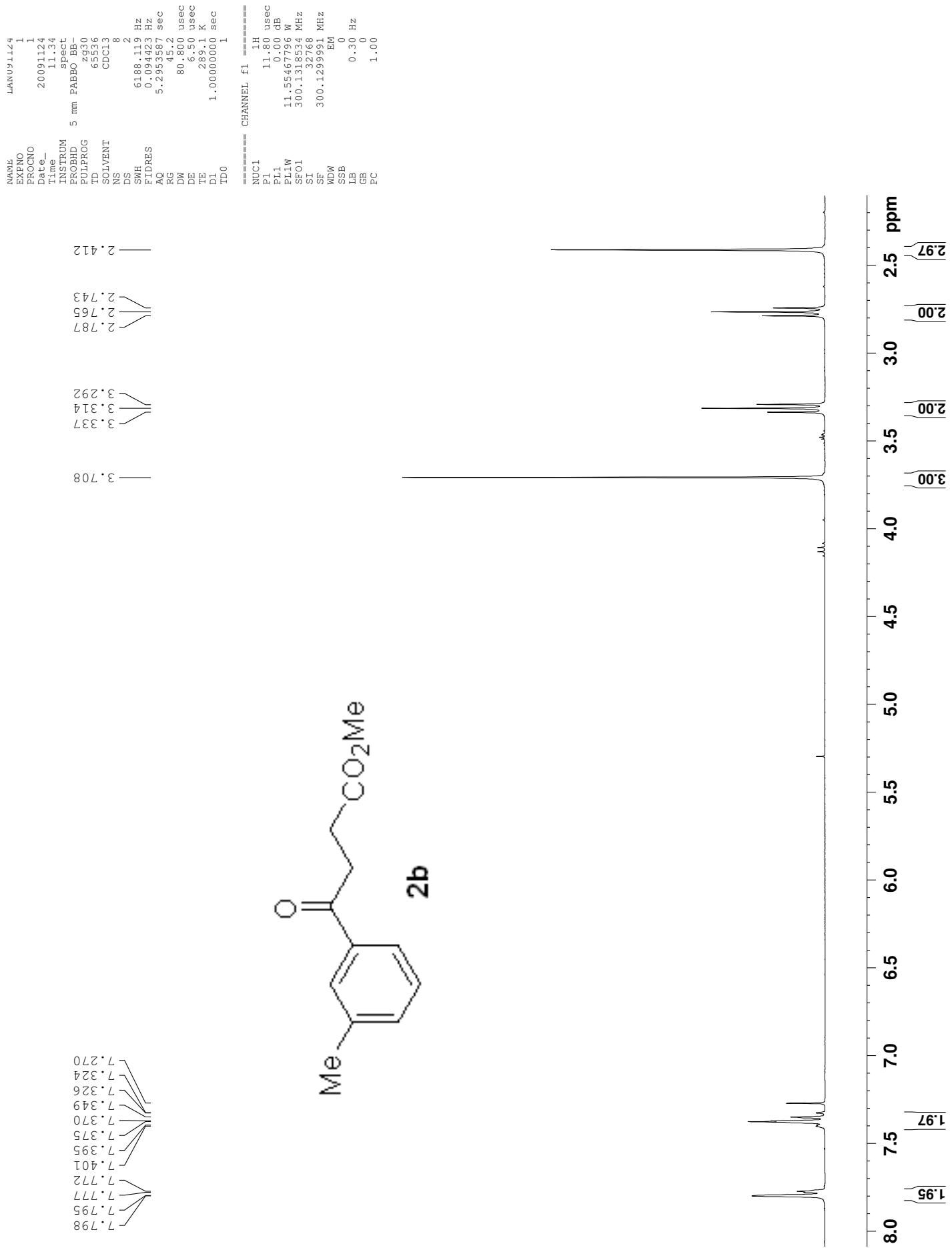
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p13

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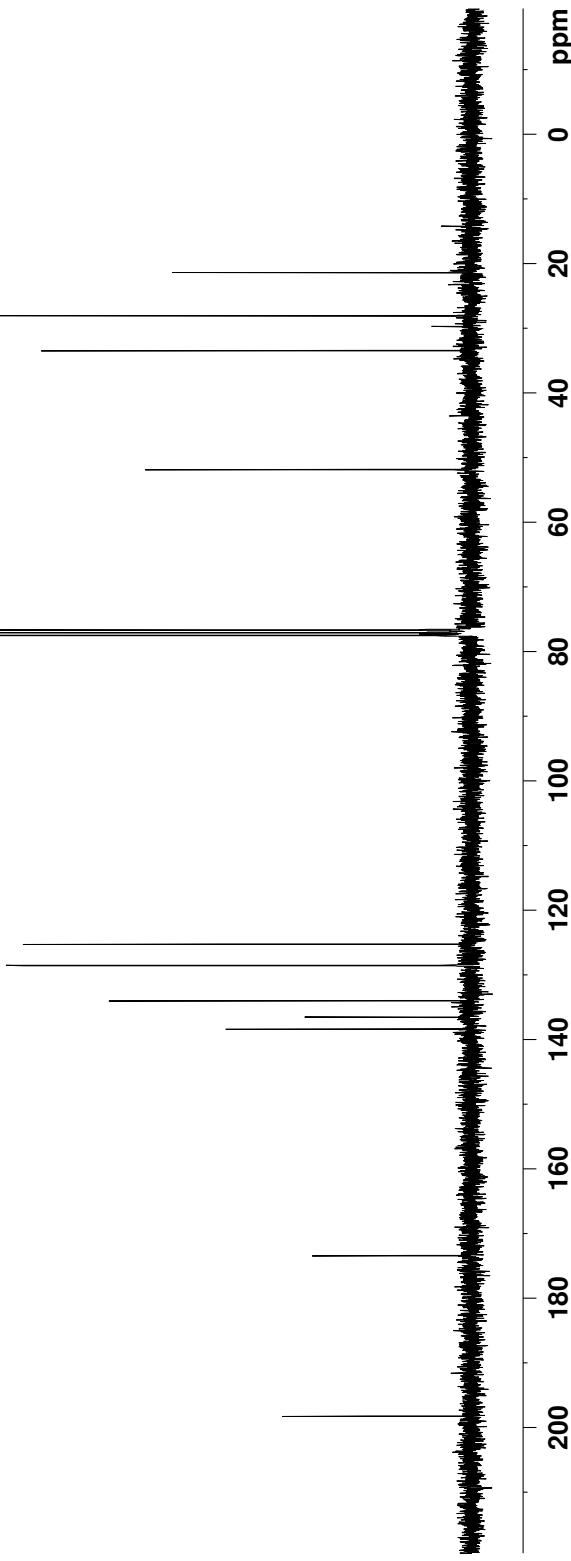
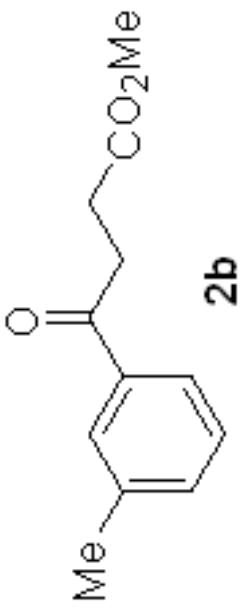
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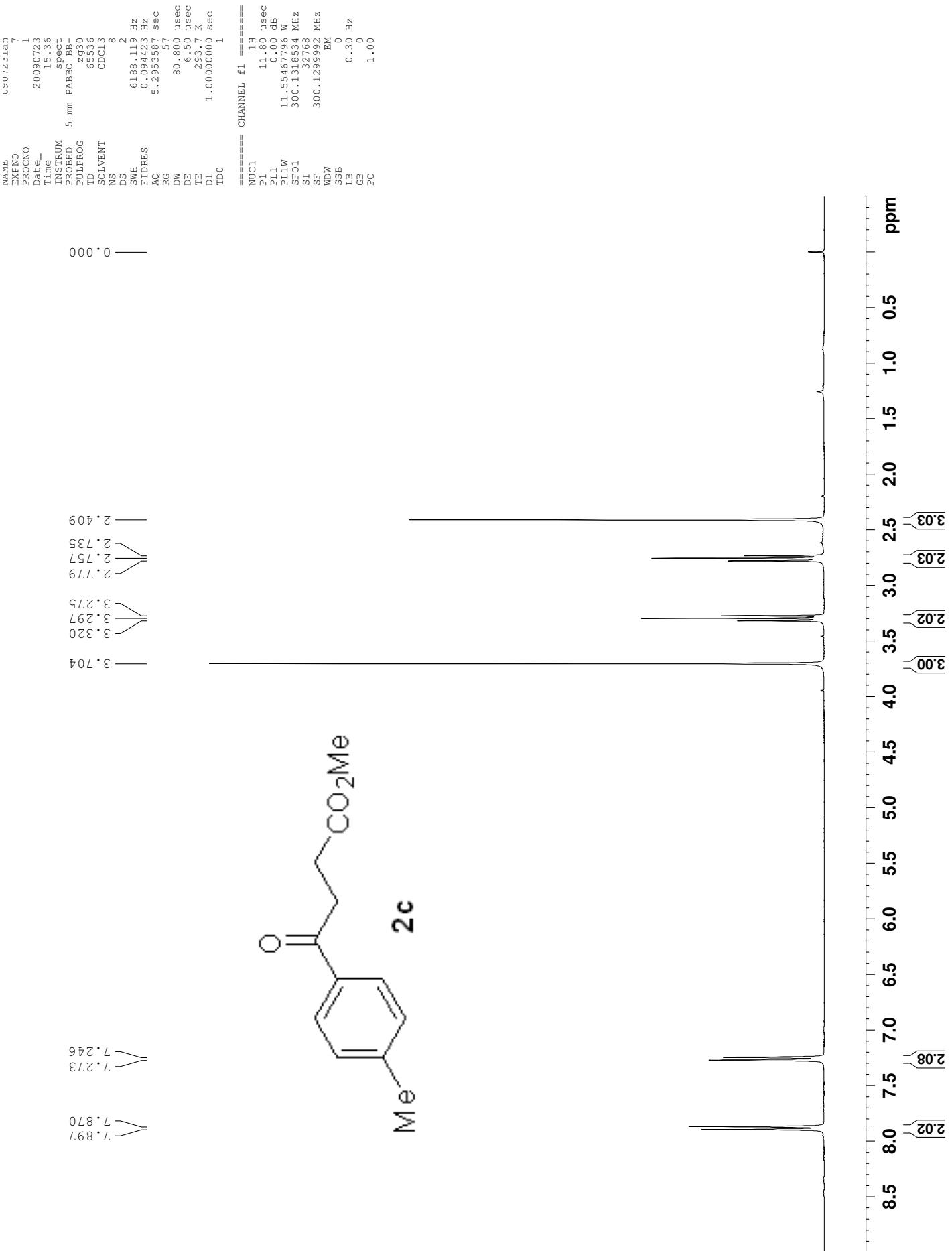
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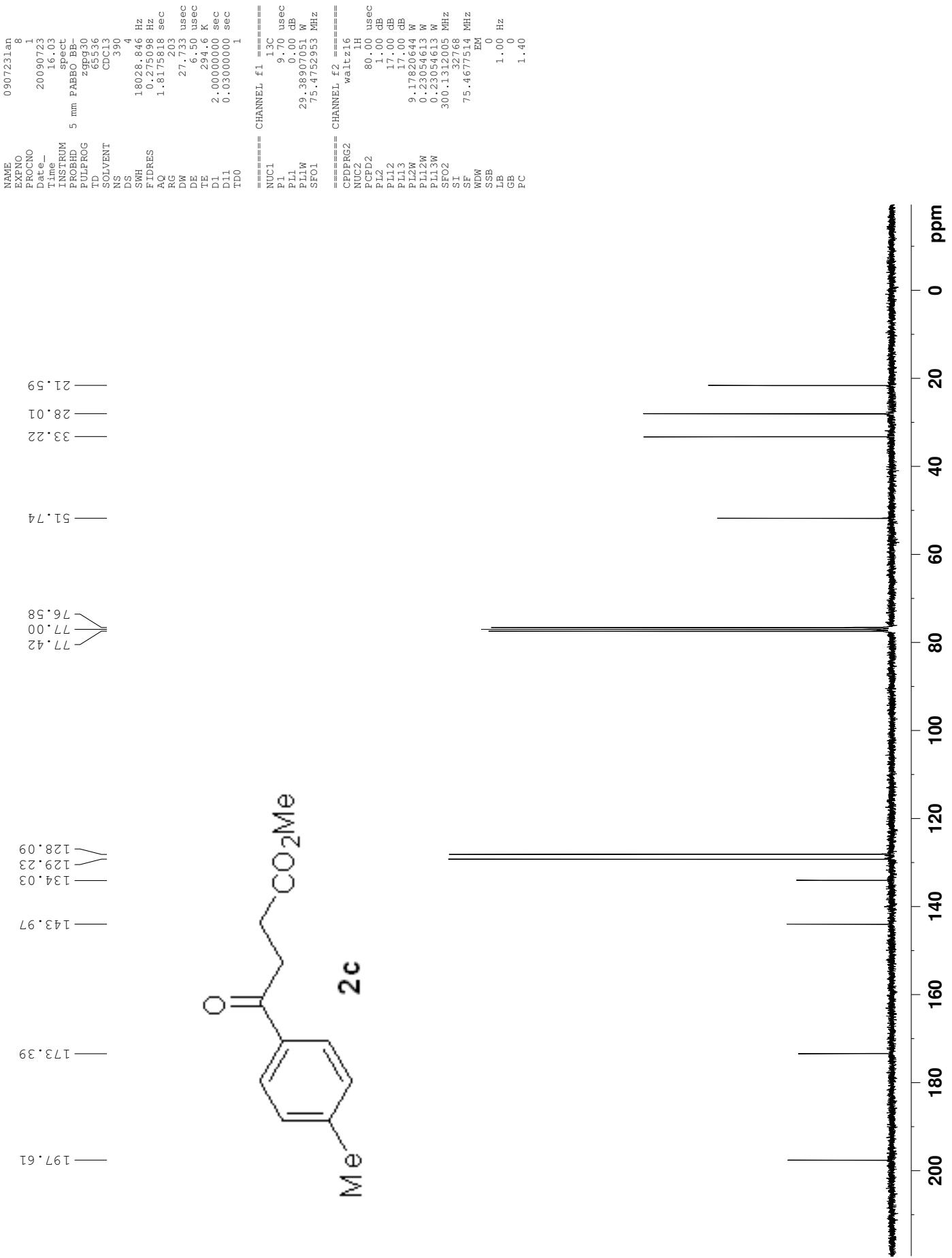
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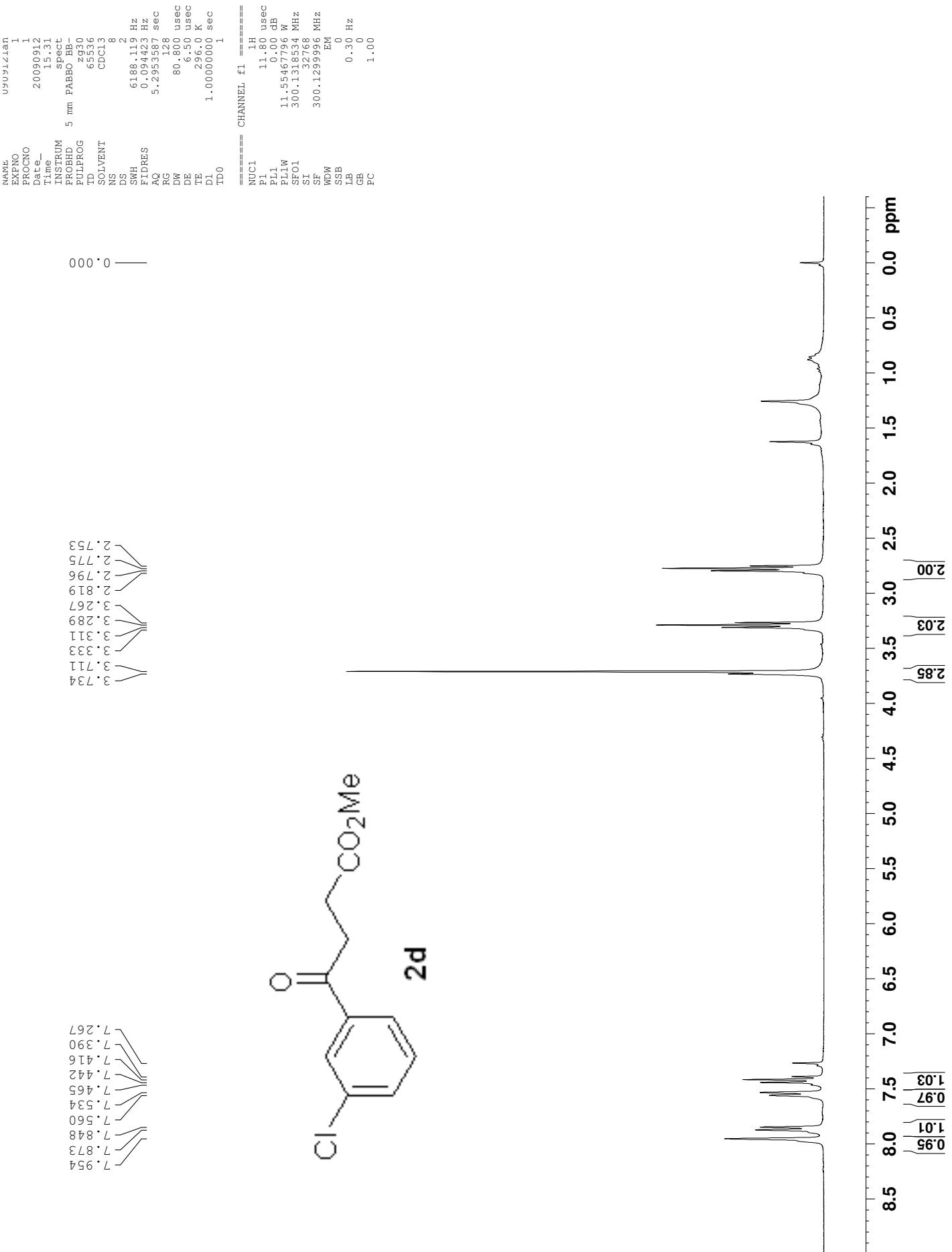
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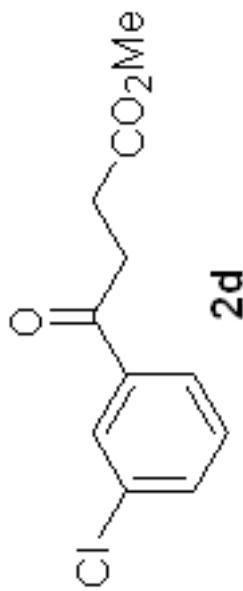








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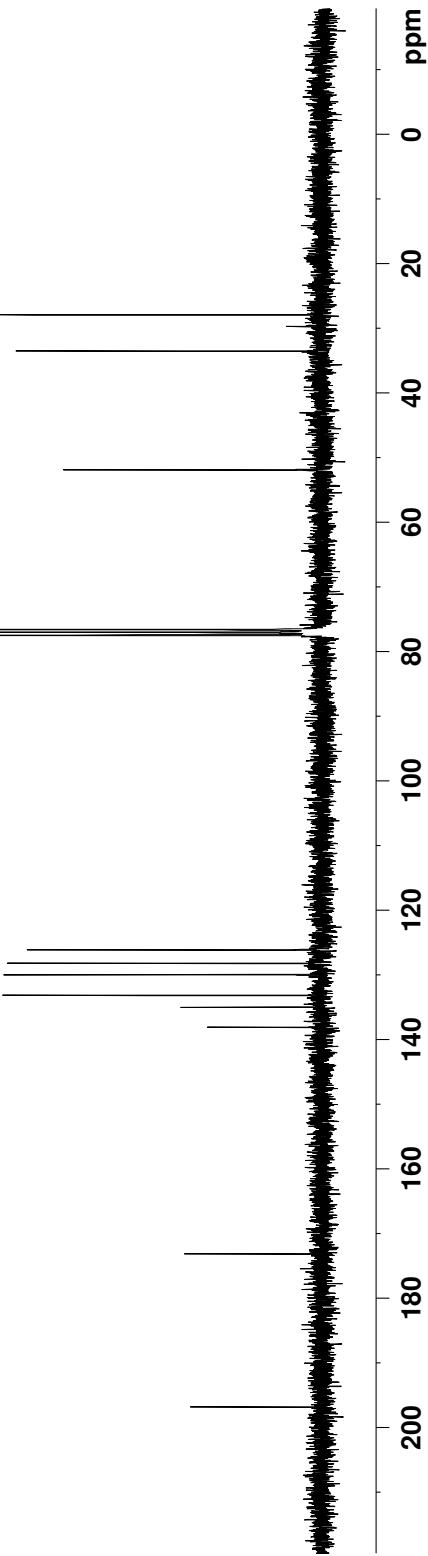
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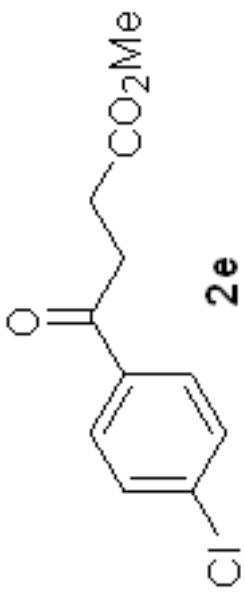
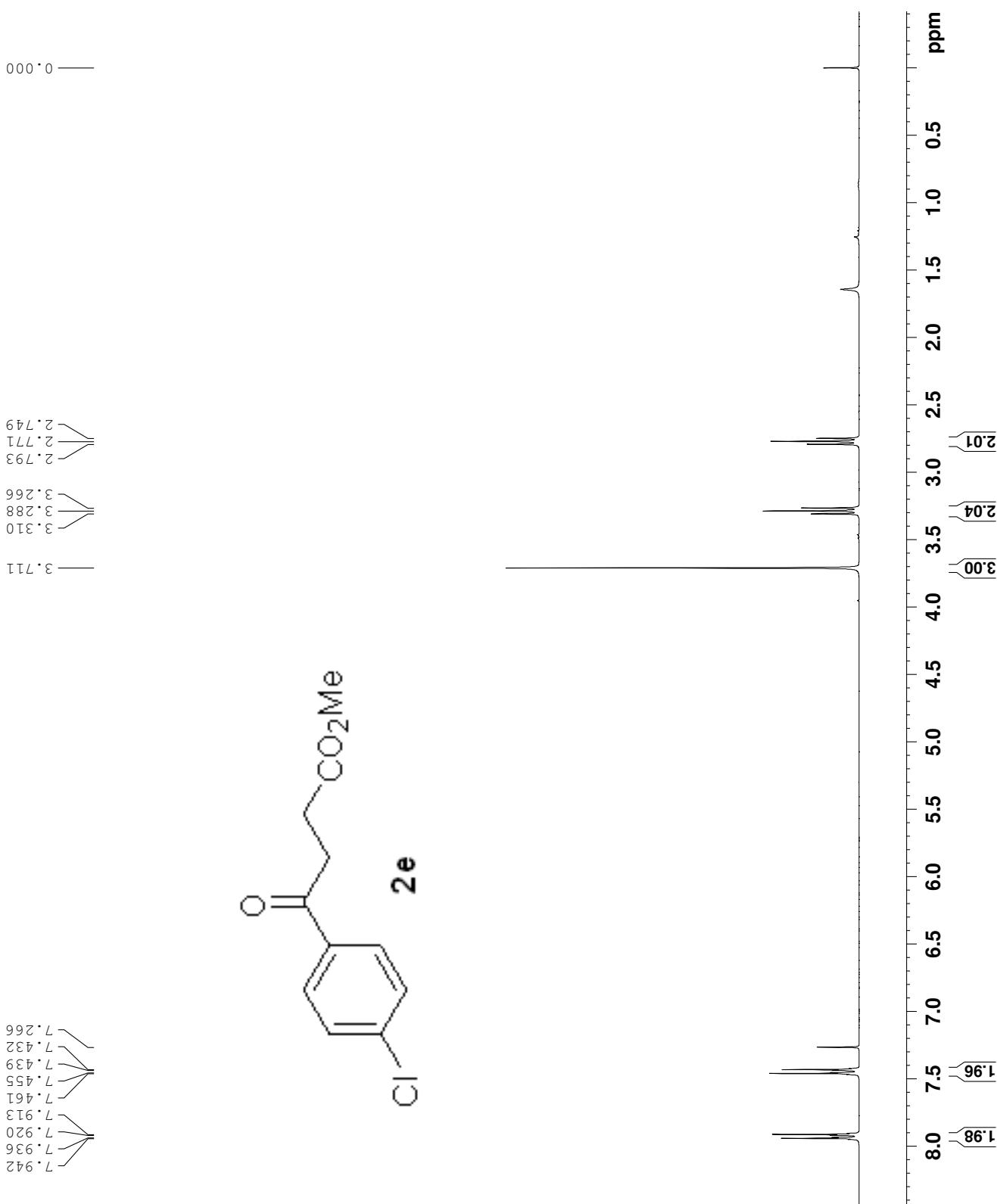
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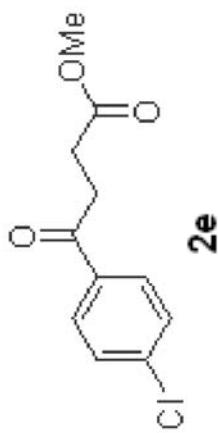
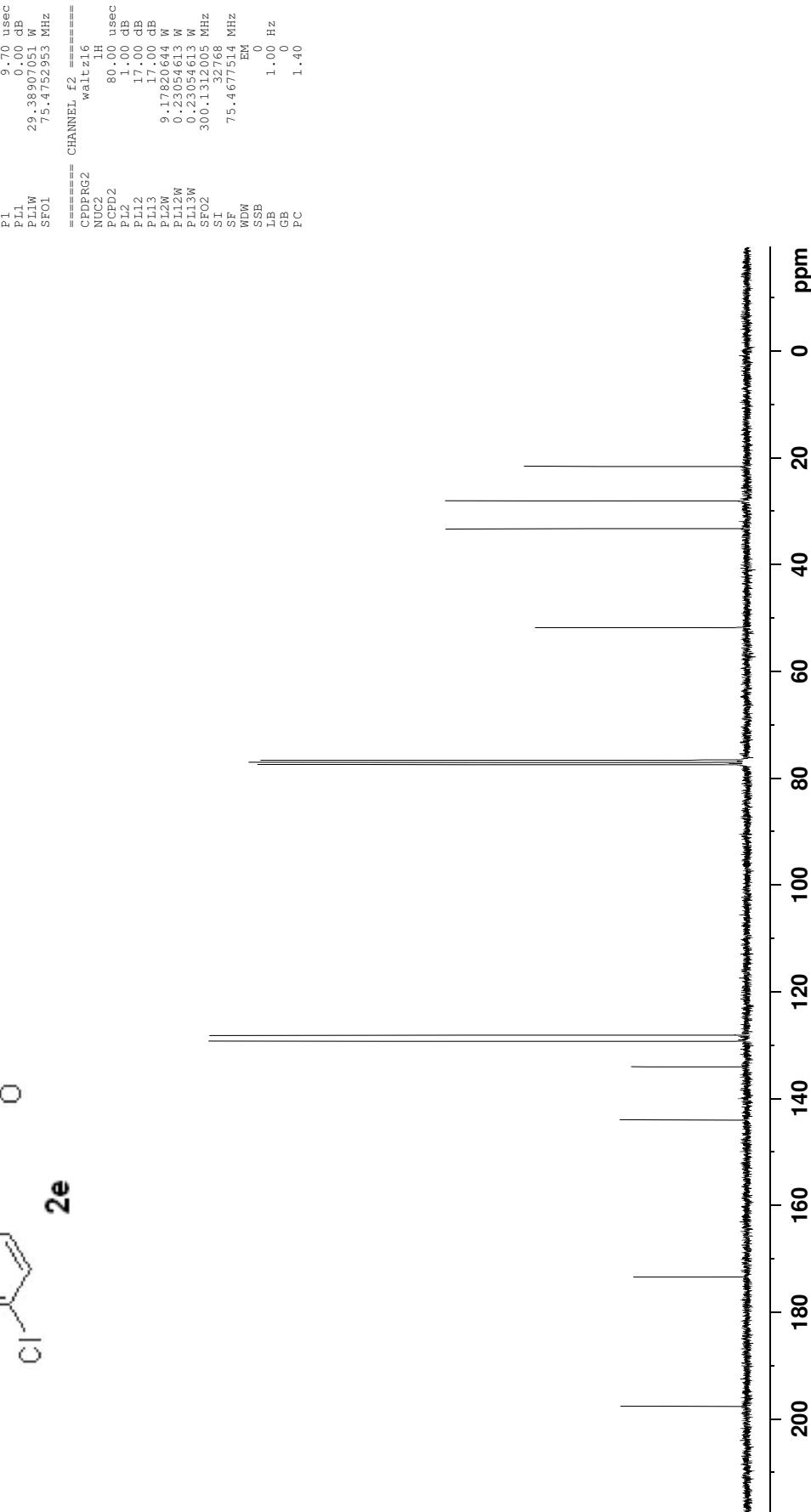


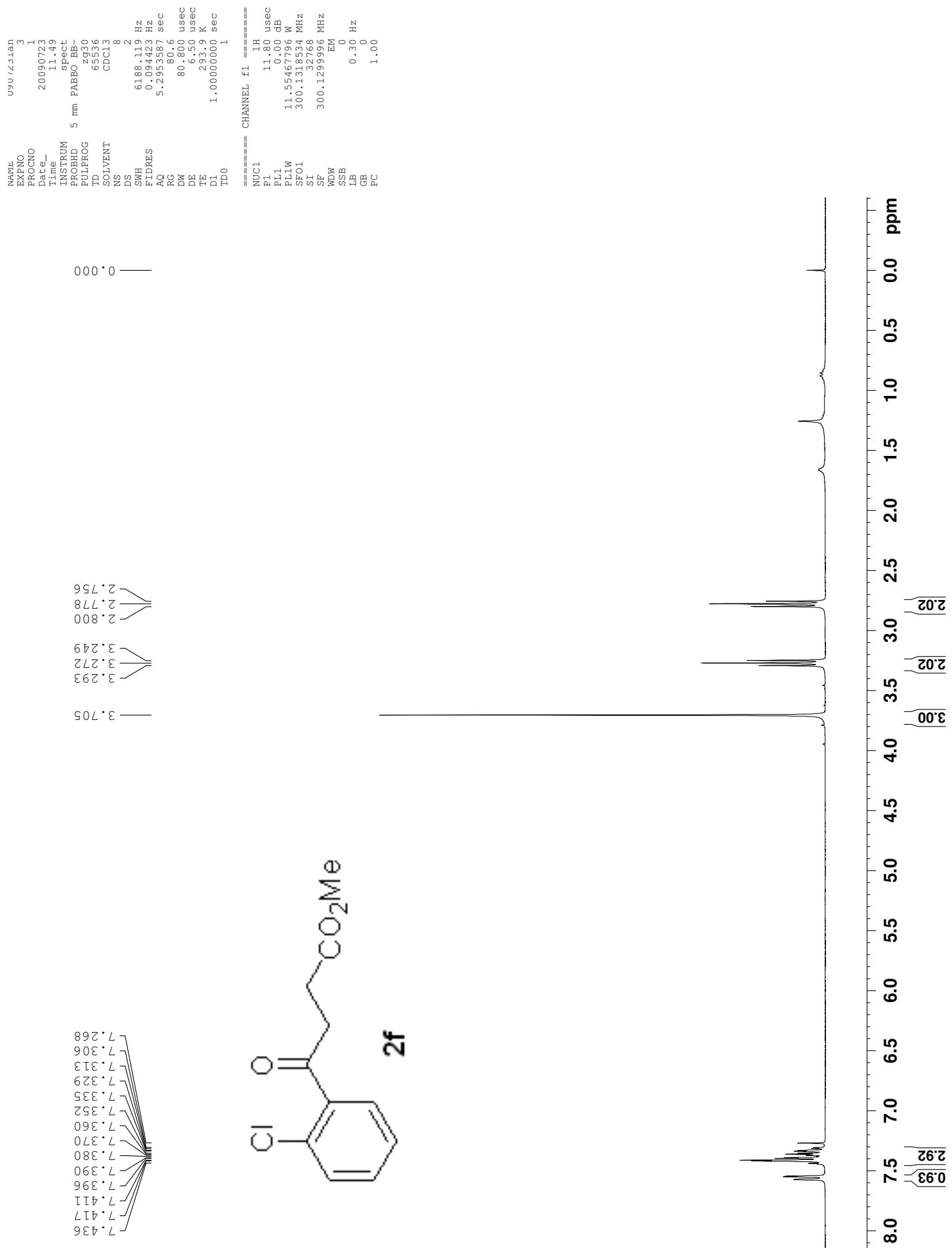


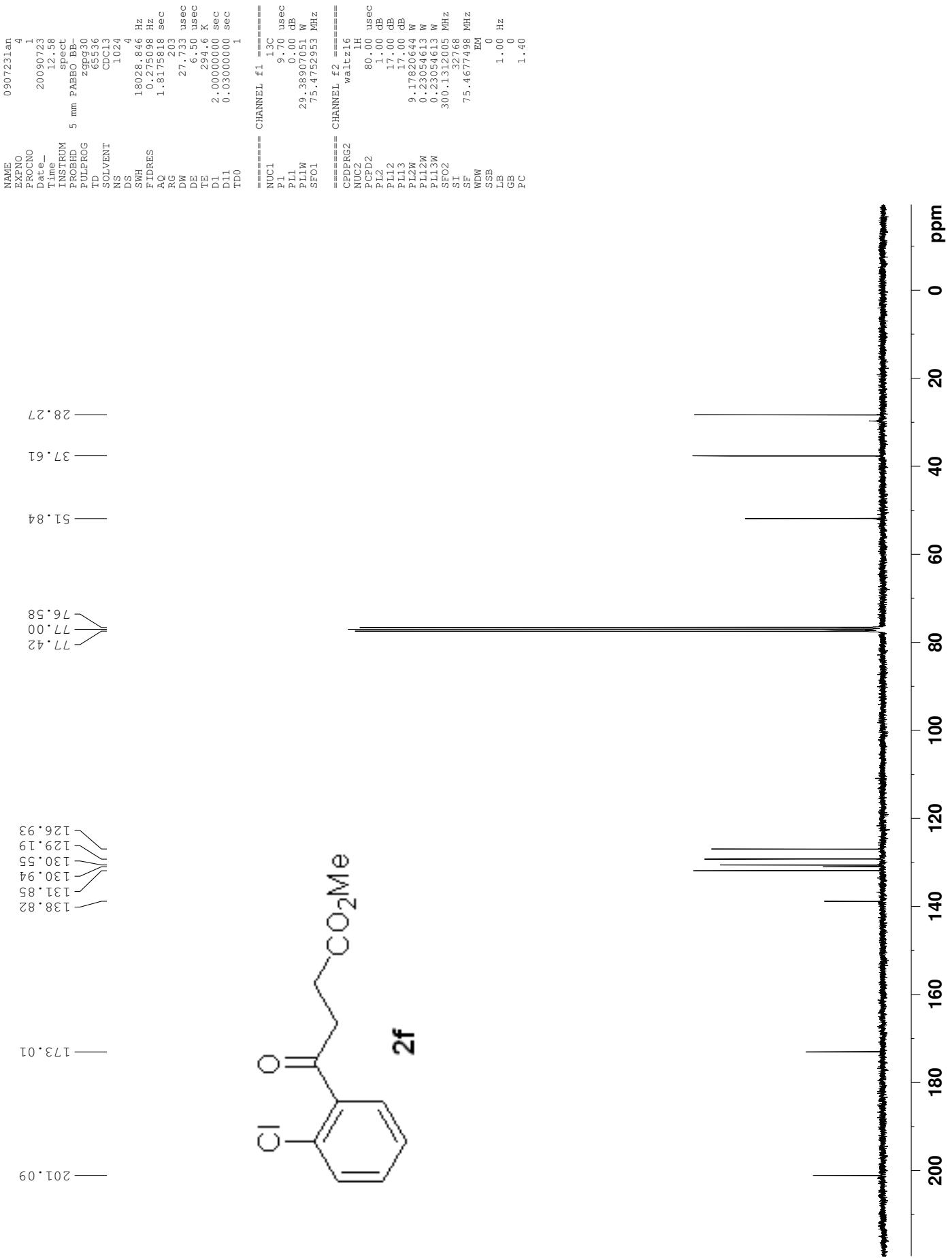
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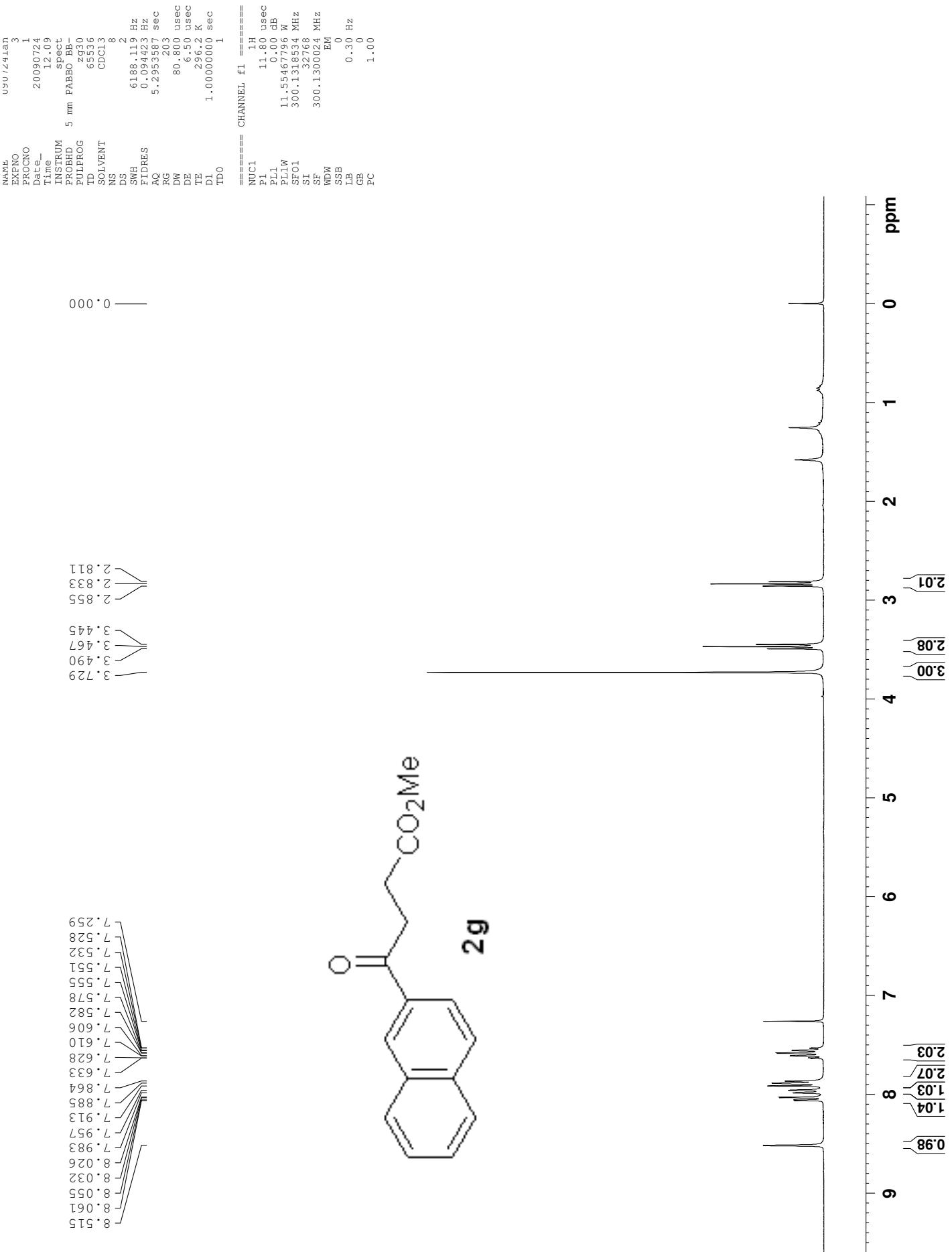
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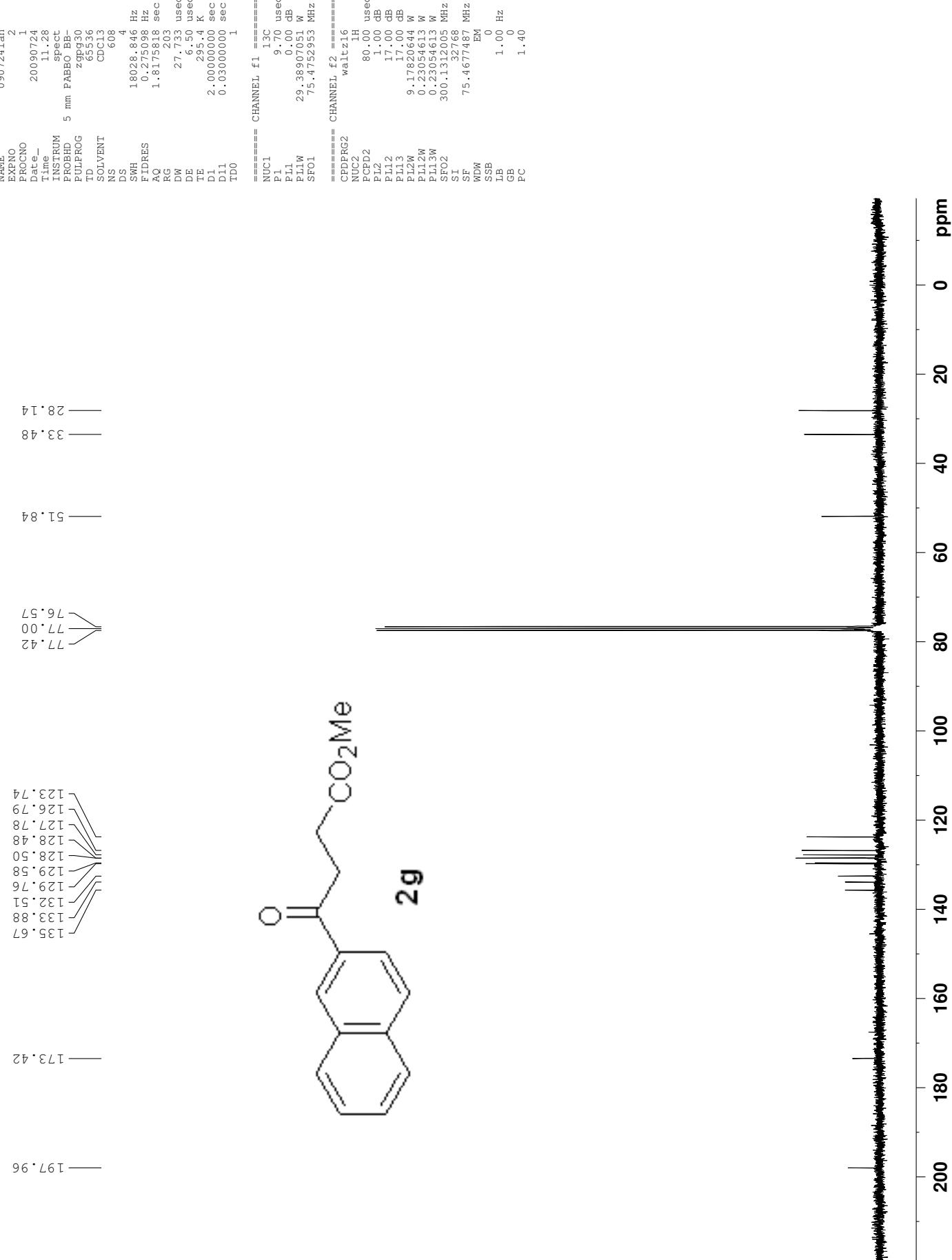
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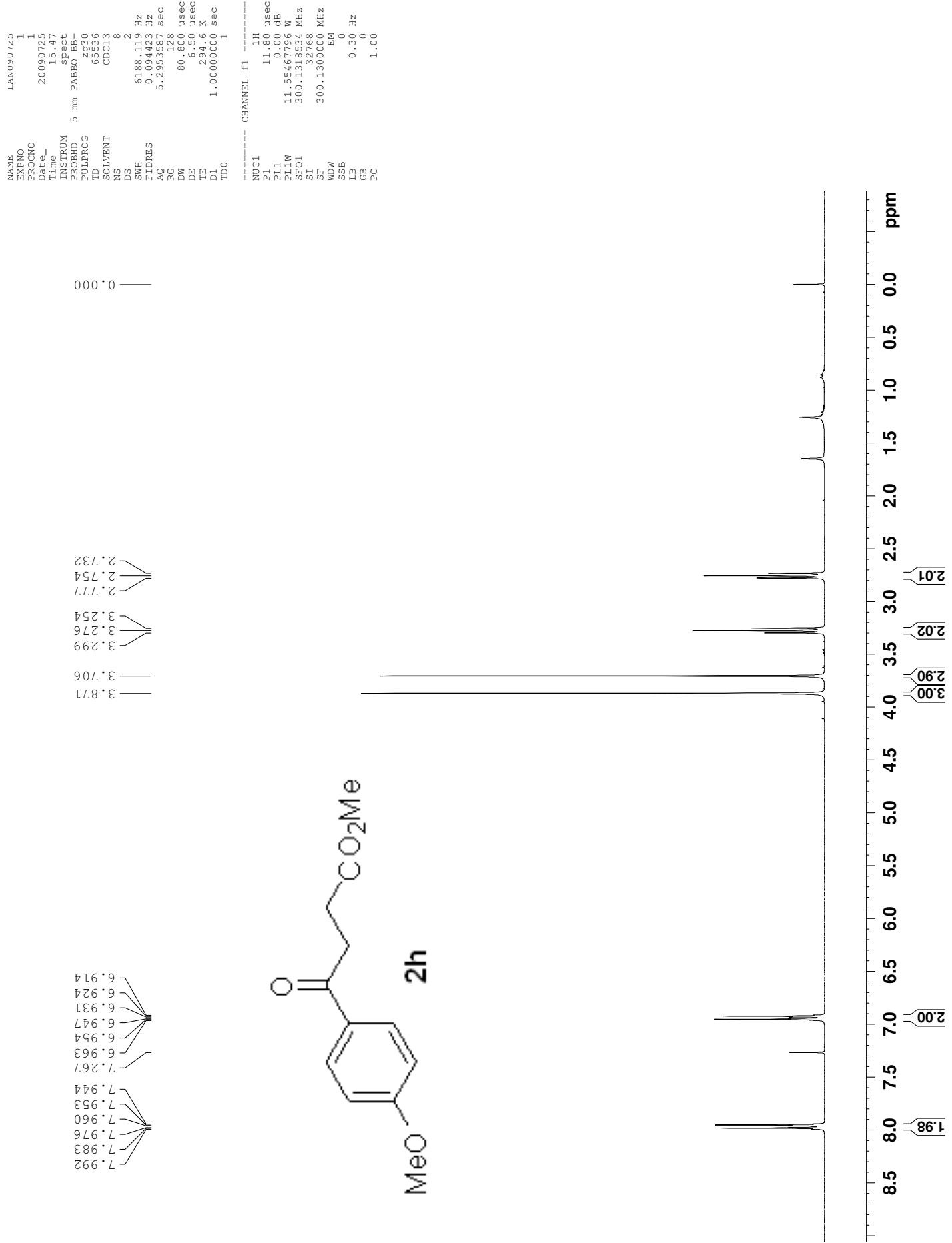


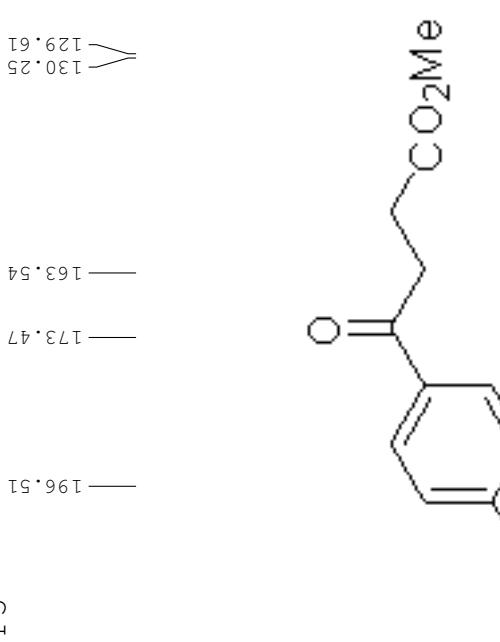












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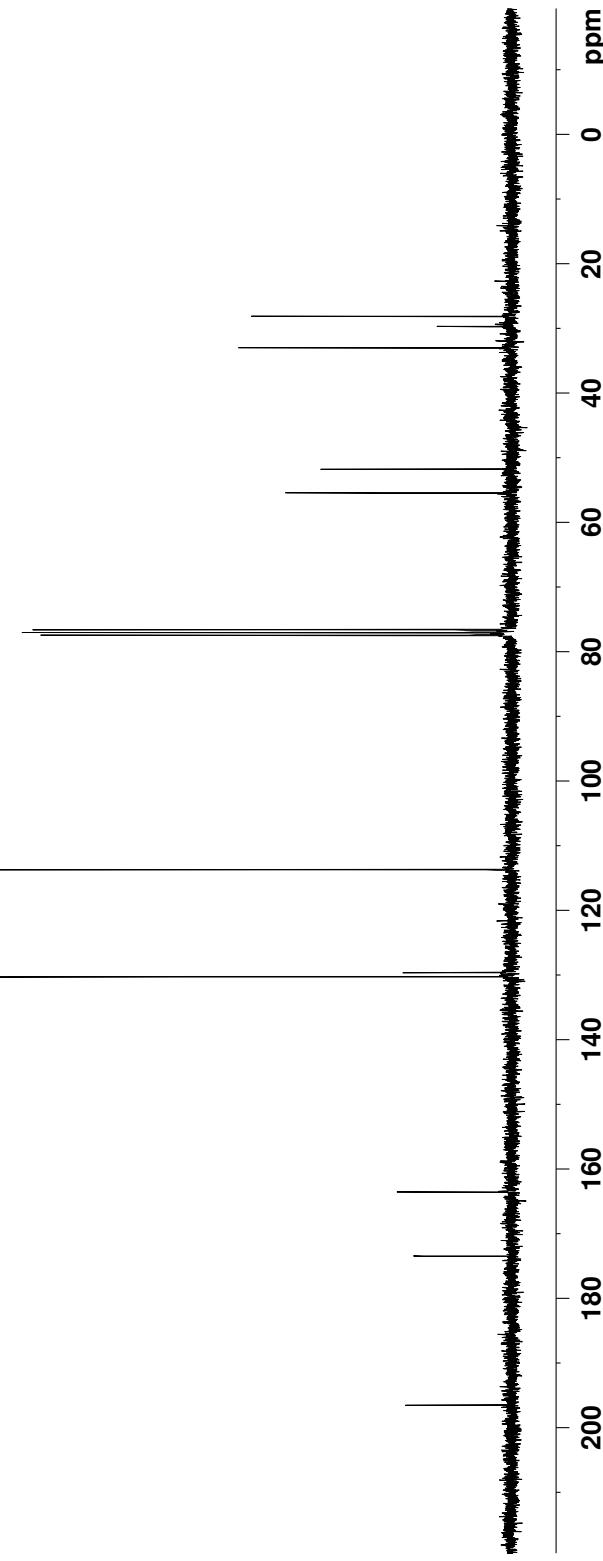
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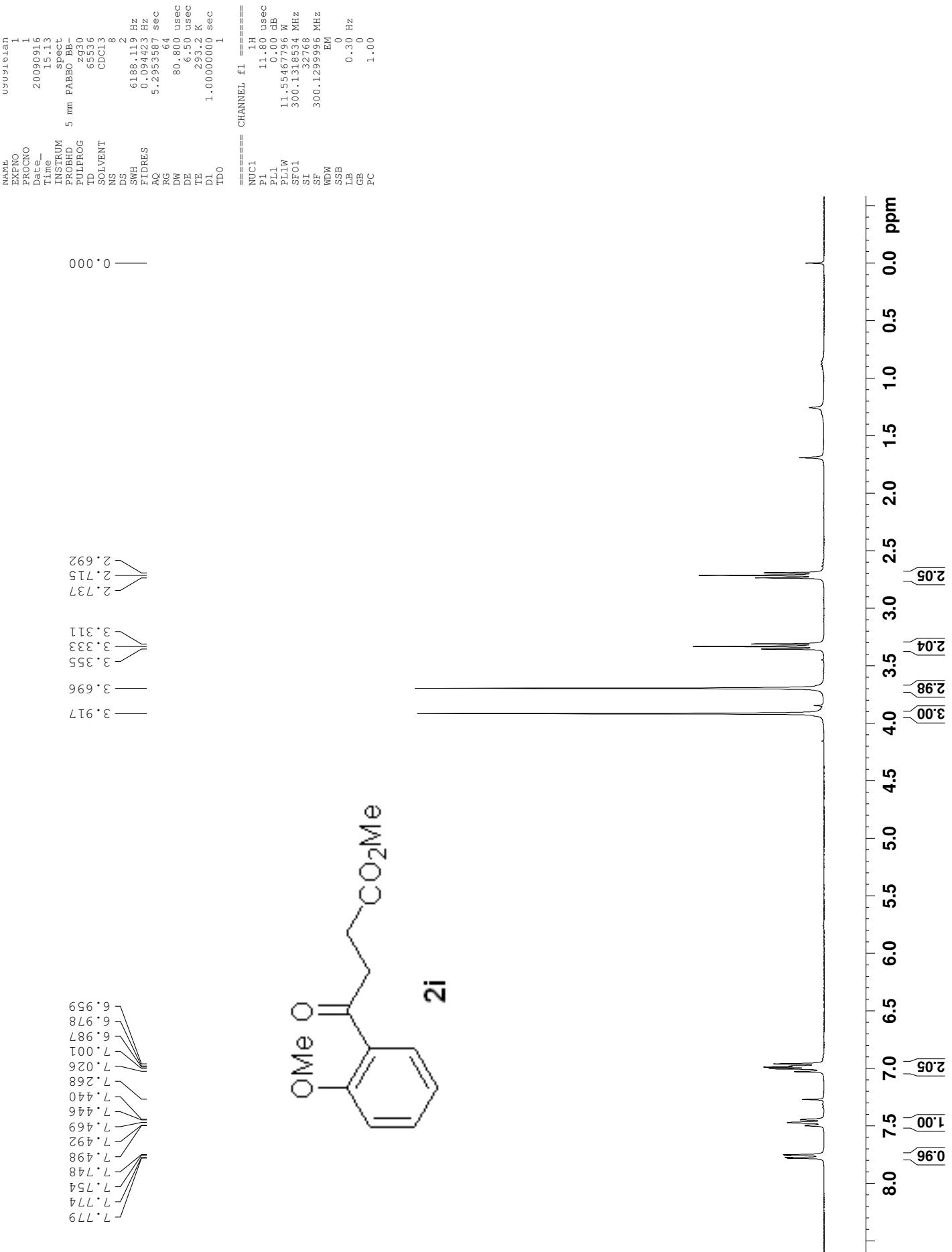
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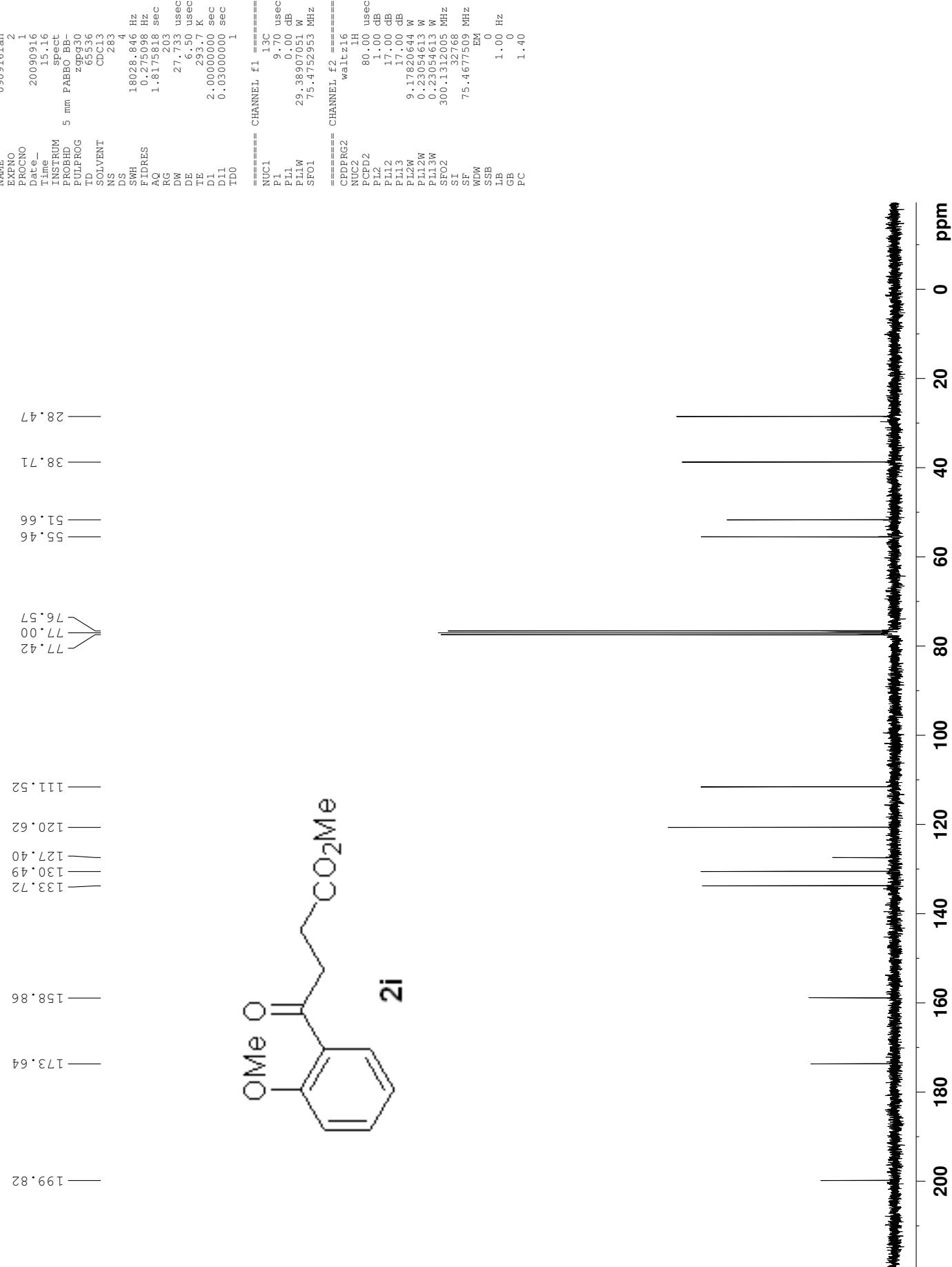
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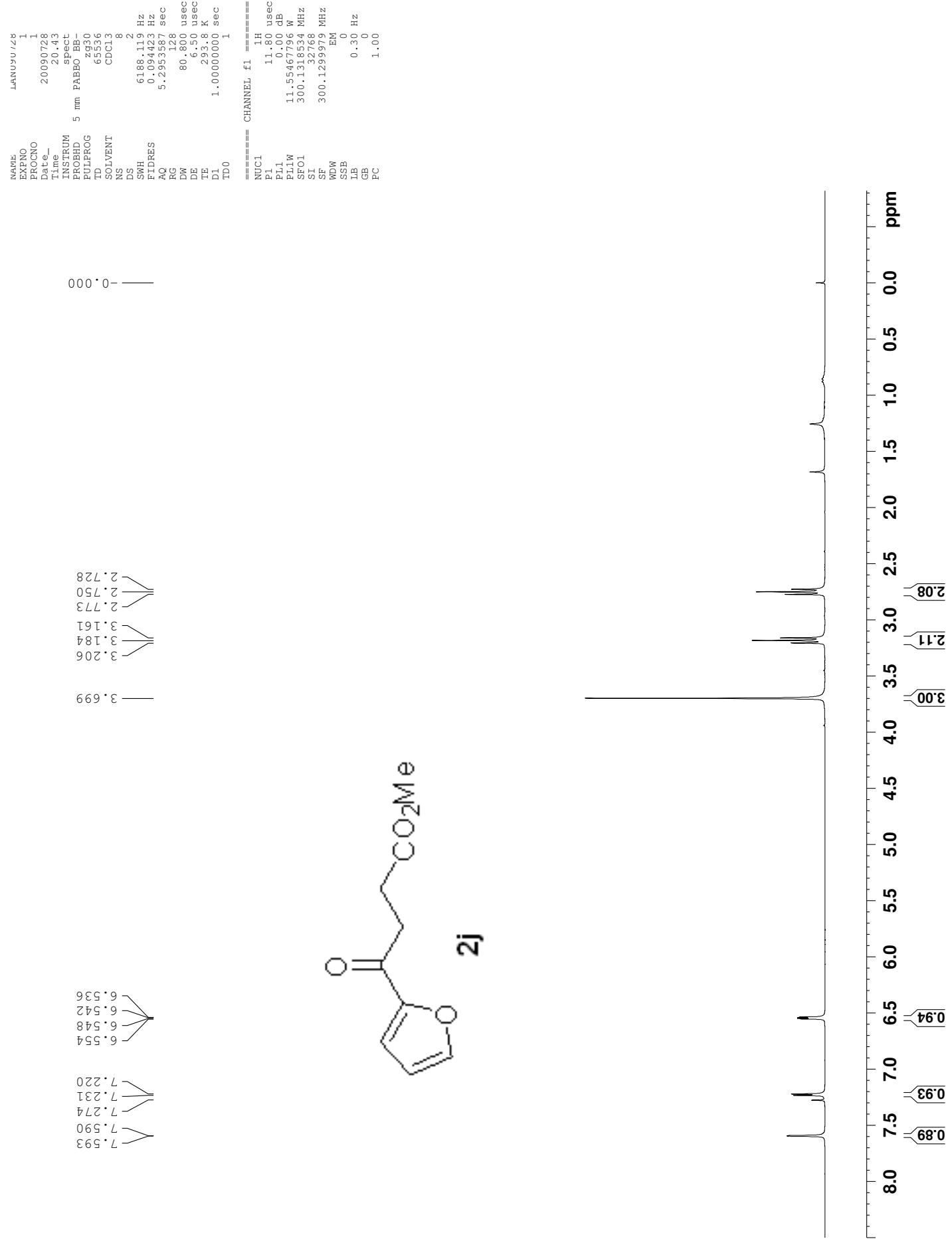
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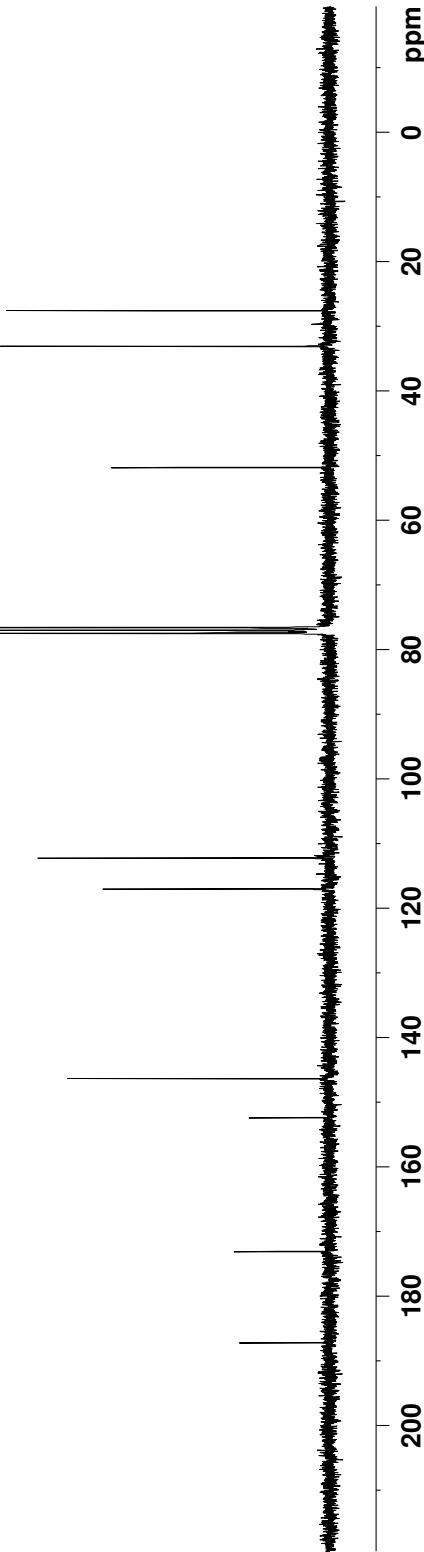
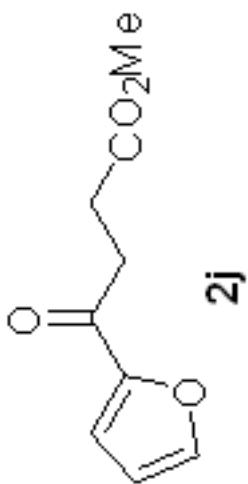
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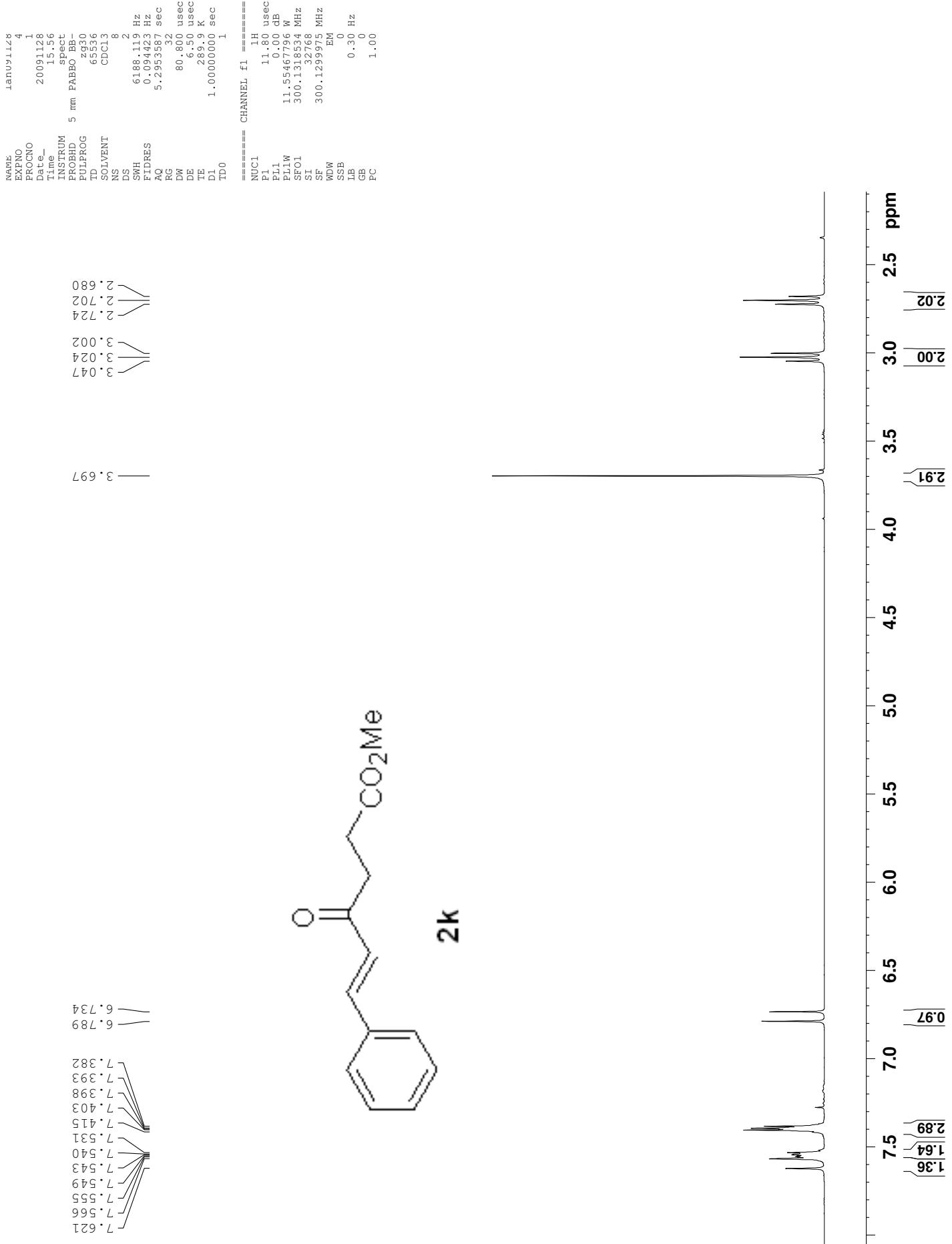
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PL13W         0.23034613 W
SF02         300.1312005 MHz
SI            322.768
SF           75.4677430 MHz
WDW          EN
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

```



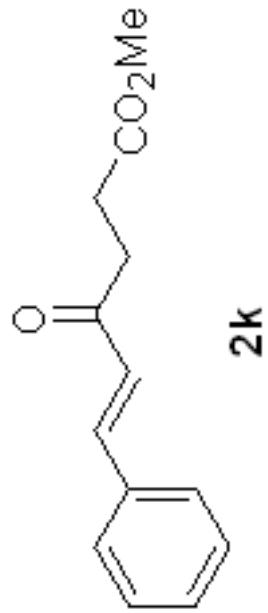


P15

142.82  
134.26  
130.45  
128.85  
128.21  
125.69

77.42  
77.00  
76.57

51.71  
51.12  
27.80



173.24

197.86

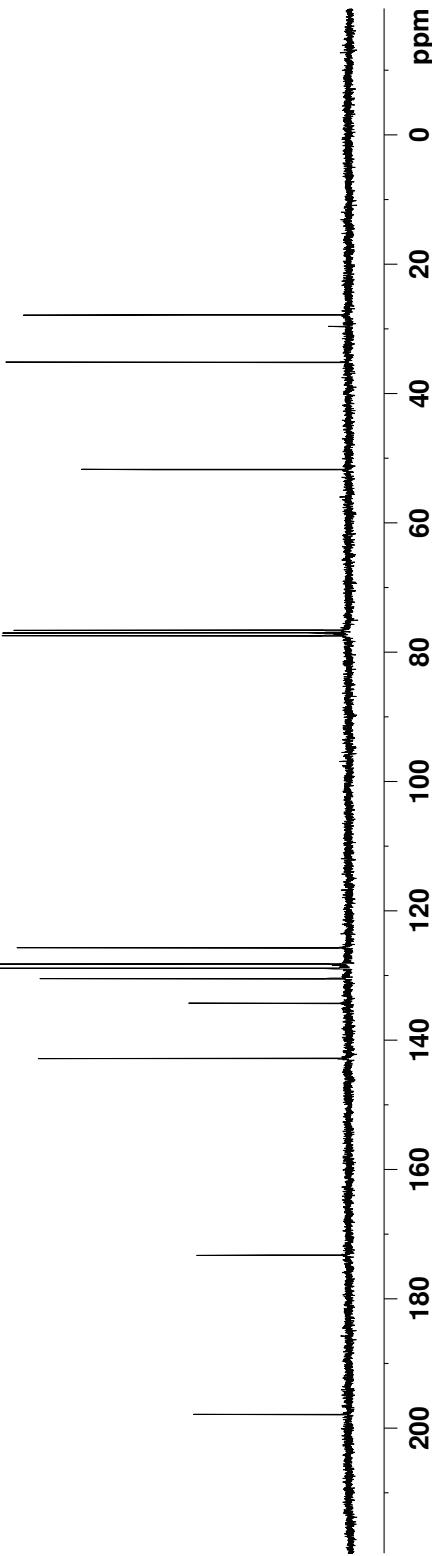
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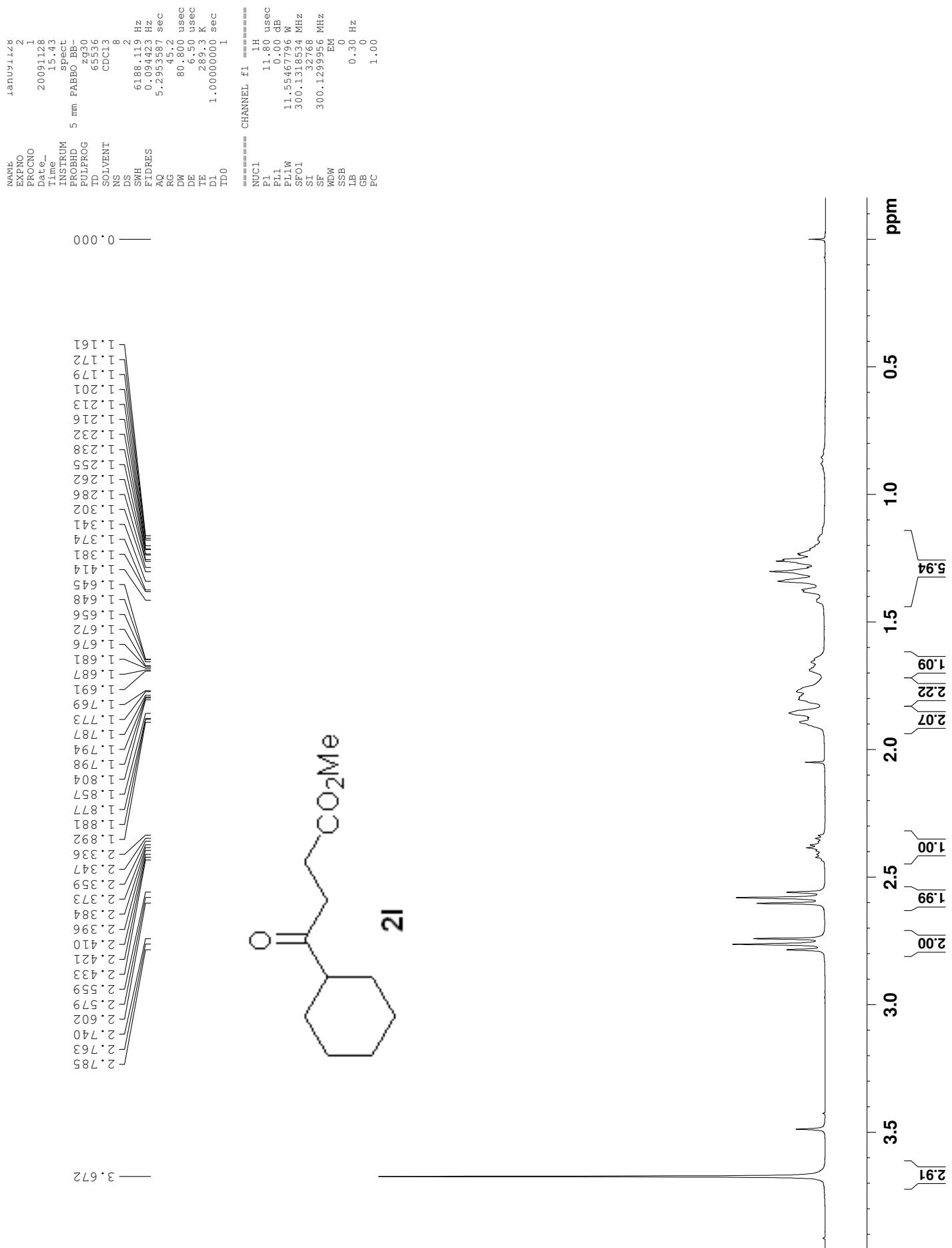
NAME      lan091128
EXENO     5
PROCNO   1
Date_    20091128
Time_    16.03
INSTRUM spect
PROBHD  5 mm PABBO BB-
PULPROG zppg36
TD       65536
SOLVENT  CDCl3
NS      154
DS       4
SWH     18028.816 Hz
FIDRES  0.225098 Hz
AQ      1.8175818 sec
RG      203
DW      27.733 usec
DE      6.50 usec
TE      290.5 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1    13C
P1      9.70 usec
PL1    0.00 dB
PL1W   29.38907051 W
SF01   75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2    1H
PCPD2   80.00 usec
PL2      1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W   0.23034613 W
PL12W  0.23034613 W
PL13W  300.1312005 MHz
SF02    75.4677564 MHz
SI      EM
WDW    0
SSB    1.00 Hz
LB      0
GB      1.40
PC

```





```

NAME      lan091128
EXENO     3
PROCNO   1
Date_    20091128
Time_    15.49
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG zppg30
TD      65536
SOLVENT  CDC13
NS      96
DS      4
SWH     18028.816 Hz
FIDRES  0.225098 Hz
AQ      1.8175818 sec
RG      203
DW      27.733 usec
DE      6.50 usec
TE      290.6 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0     1

===== CHANNEL f1 =====
NUC1    13C
P1      9.70 usec
PL1     0.00 dB
PL1W    29.38907051 W
SF01    75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2    1H
PCPD2   80.00 usec
PL2     1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W    0.23034613 W
PL12W   0.23034613 W
PL13W   0.23034613 W
SF02    300.1312005 MHz
SI      322.768
SF      75.467754 MHz
WDW    EM
SSB    0
LB      1.00 Hz
GB      0
PC      1.40

```

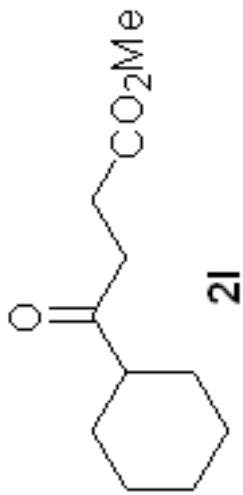
25.57  
25.77  
26.44  
27.64  
28.41  
34.94  
50.65  
51.70  
52.57

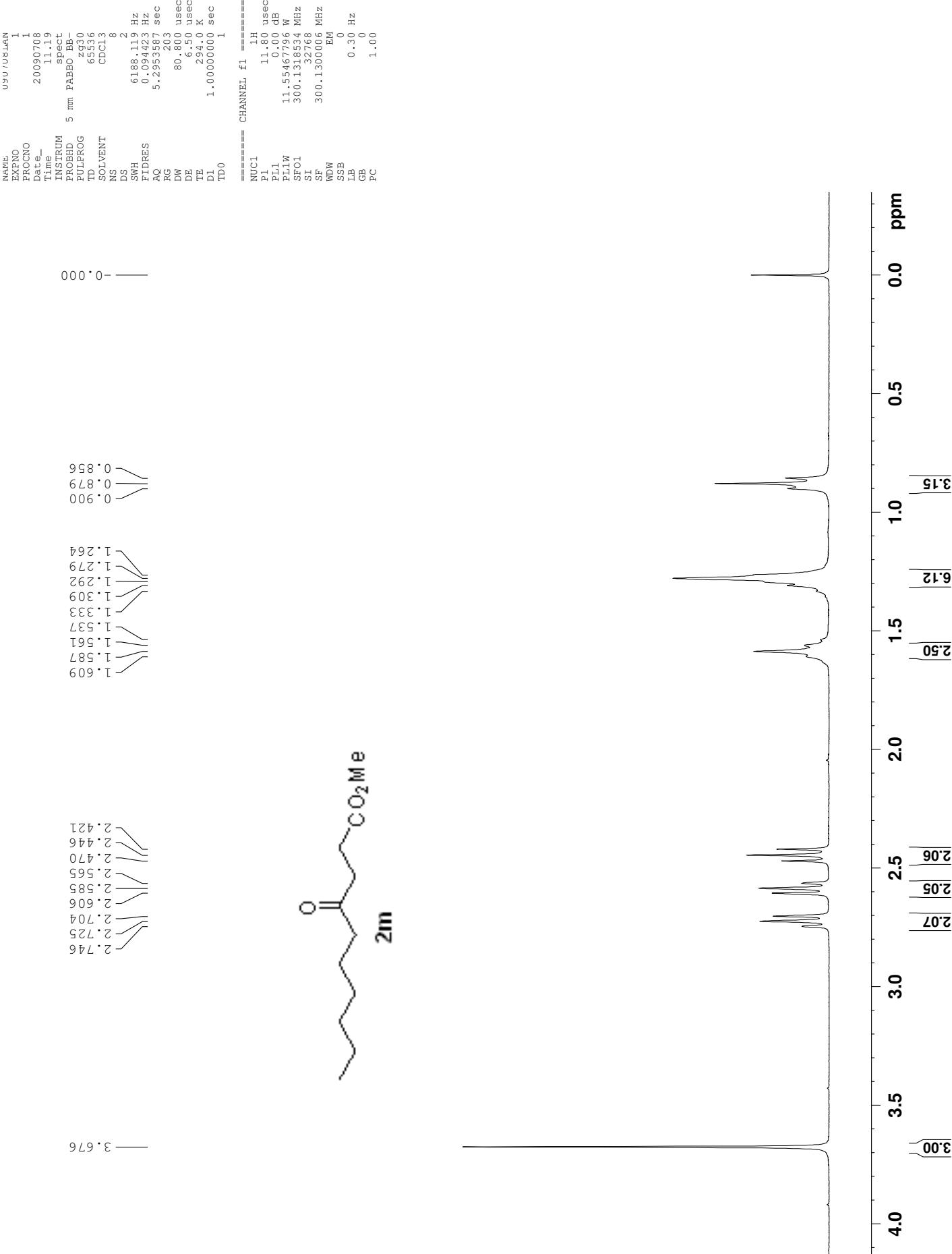
76.57  
77.00  
77.42

173.38

P14

212.038





genq quan

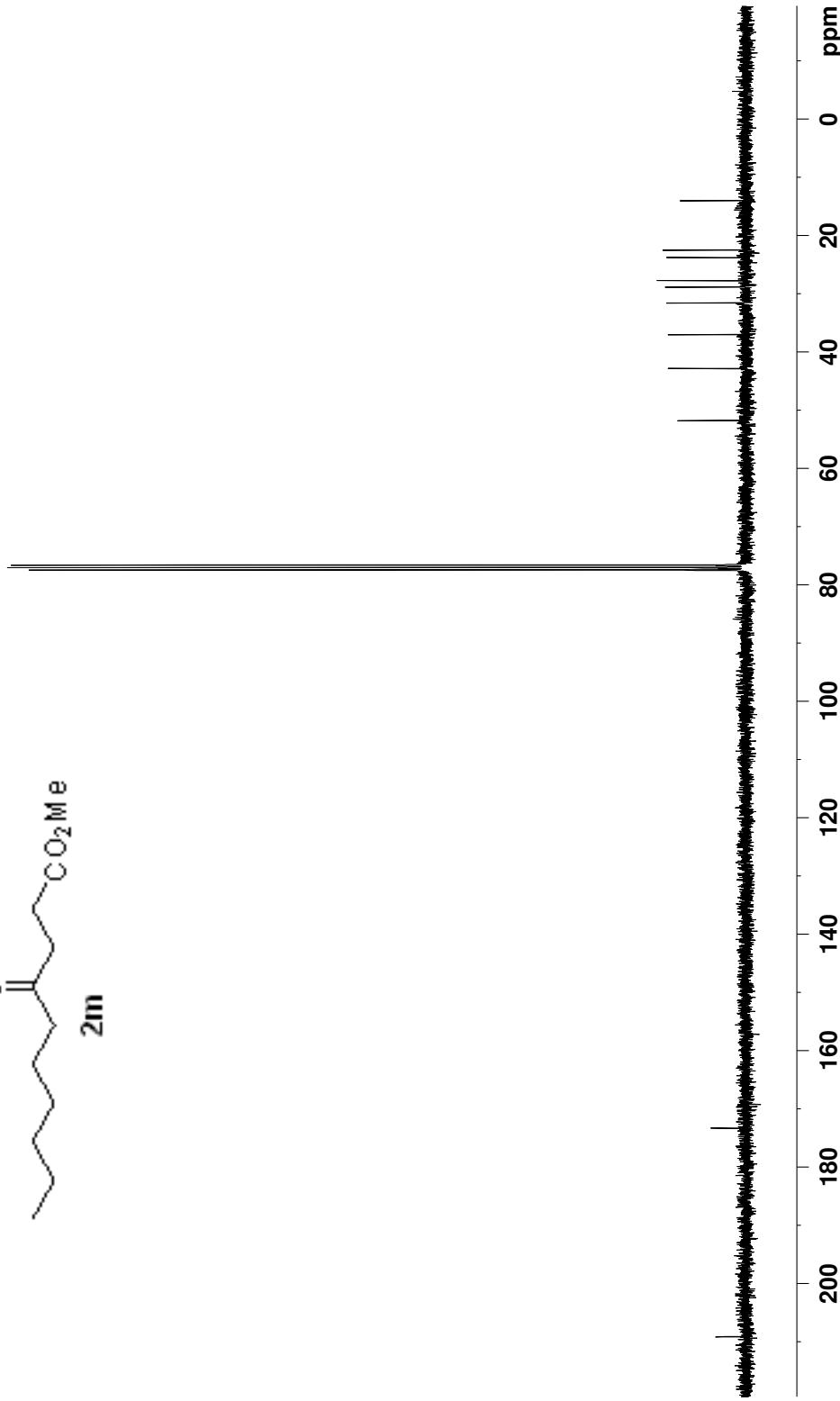
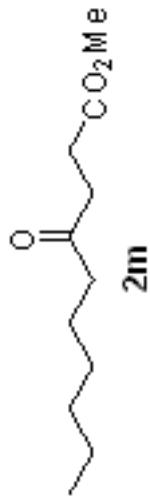
173.32

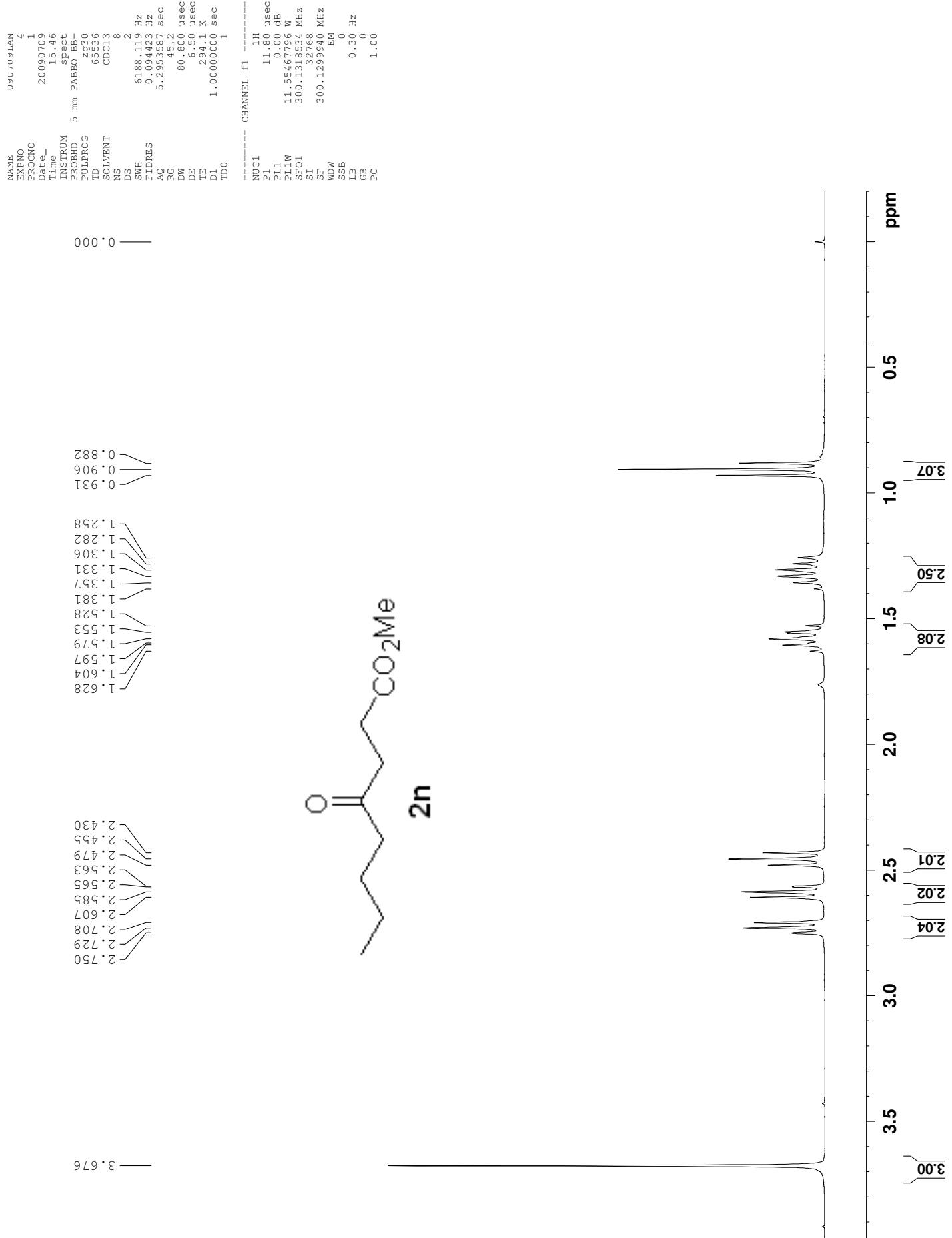
209.14

77.42  
77.00  
76.58

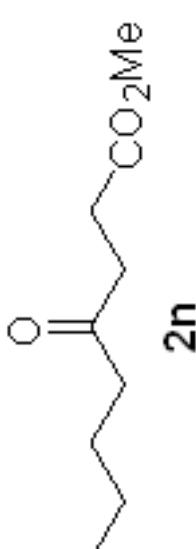
51.75  
42.81  
37.00  
31.56  
28.56  
27.71  
22.46  
23.76  
27.76  
28.56  
31.56  
42.00

NAME 090709LAN  
EXENO 7  
PROCNO 1  
Date\_ 20090709  
Time 16.02  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zppg36  
TD 65536  
SOLVENT CDC13  
NS 293  
DS 4  
SWH 18028.816 Hz  
FIDRES 0.25098 Hz  
AQ 1.8175818 sec  
RG 203  
DW 27.733 usec  
DE 6.50 usec  
TE 294.7 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.70 usec  
PL1 0.00 dB  
PL1W 29.38907051 W  
SF01 75.4732933 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 1.00 dB  
PL12 17.00 dB  
PL13 9.17820644 W  
PL2W 0.23034613 W  
PL12W 0.23034613 W  
PL13W 300.1312005 MHz  
SF02 75.4677484 MHz  
SI EM  
SF 0  
WDW 1.00 Hz  
SSB LB  
LB 1.00 Hz  
GB 0  
PC 1.40





173.26



**2n**

173.26  
13.76  
22.24  
25.84  
25.66  
36.94  
42.44  
51.69

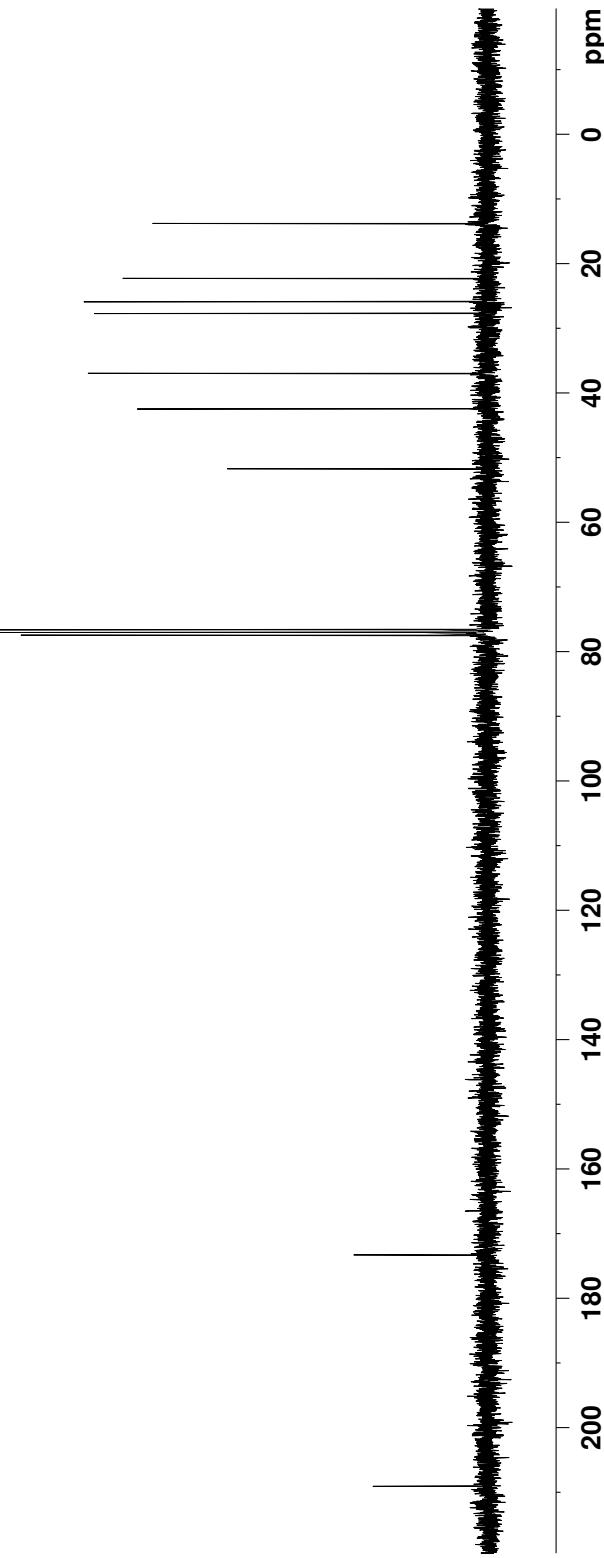
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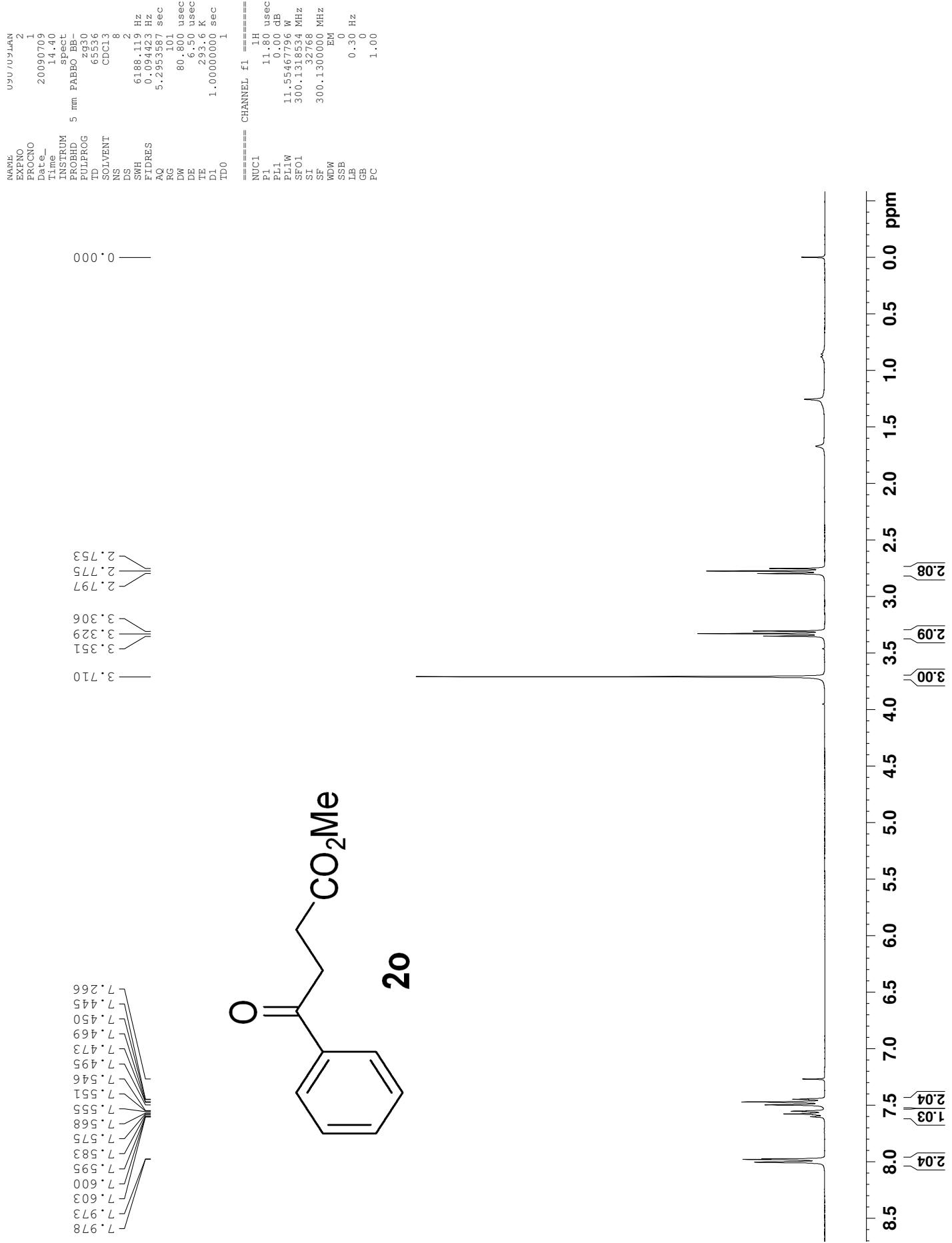
NAME          090709LAN
EXENO         5
PROCNO        1
Date_         20090709
Time          15.51
INSTRUM       spect
PROBHD       5 mm PABBO BB-
PULPROG      ZPP936
TD           65536
SOLVENT       CDC13
NS            70
DS            4
SWH          18028.816 Hz
FIDRES       0.225098 Hz
AQ            1.8175818 sec
RG            203
DW           27.733 usec
DE            6.50 usec
TE            294.8 K
D1           2.0000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1            9.70 usec
PL1           0.00 dB
PL1W          29.38907051 W
SF01          75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           1.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W          9.17820644 W
PL12W         0.23034613 W
PL13W         0.23034613 W
SF02          300.1312005 MHz
SI            322768
SF            75.467753 MHz
WDW           EM
SSB            0
LB            1.00 Hz
GB            0
PC            1.40

```



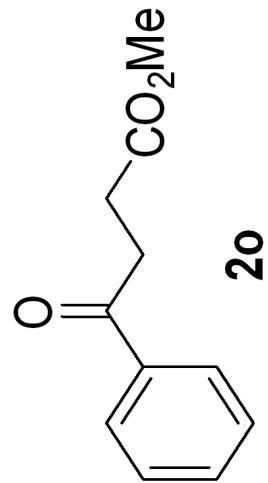


ben\_jia\_quan

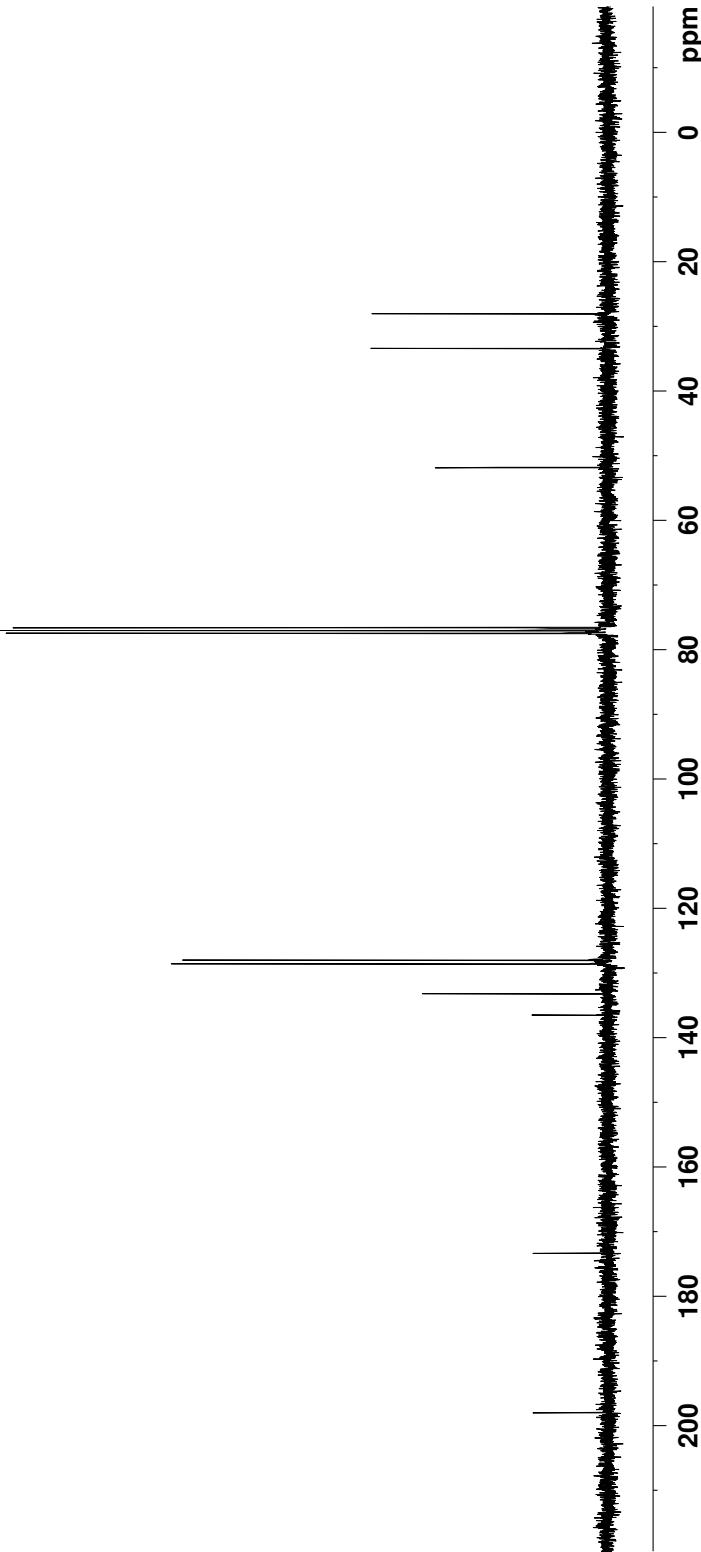
136.50  
133.21  
128.59  
128.00

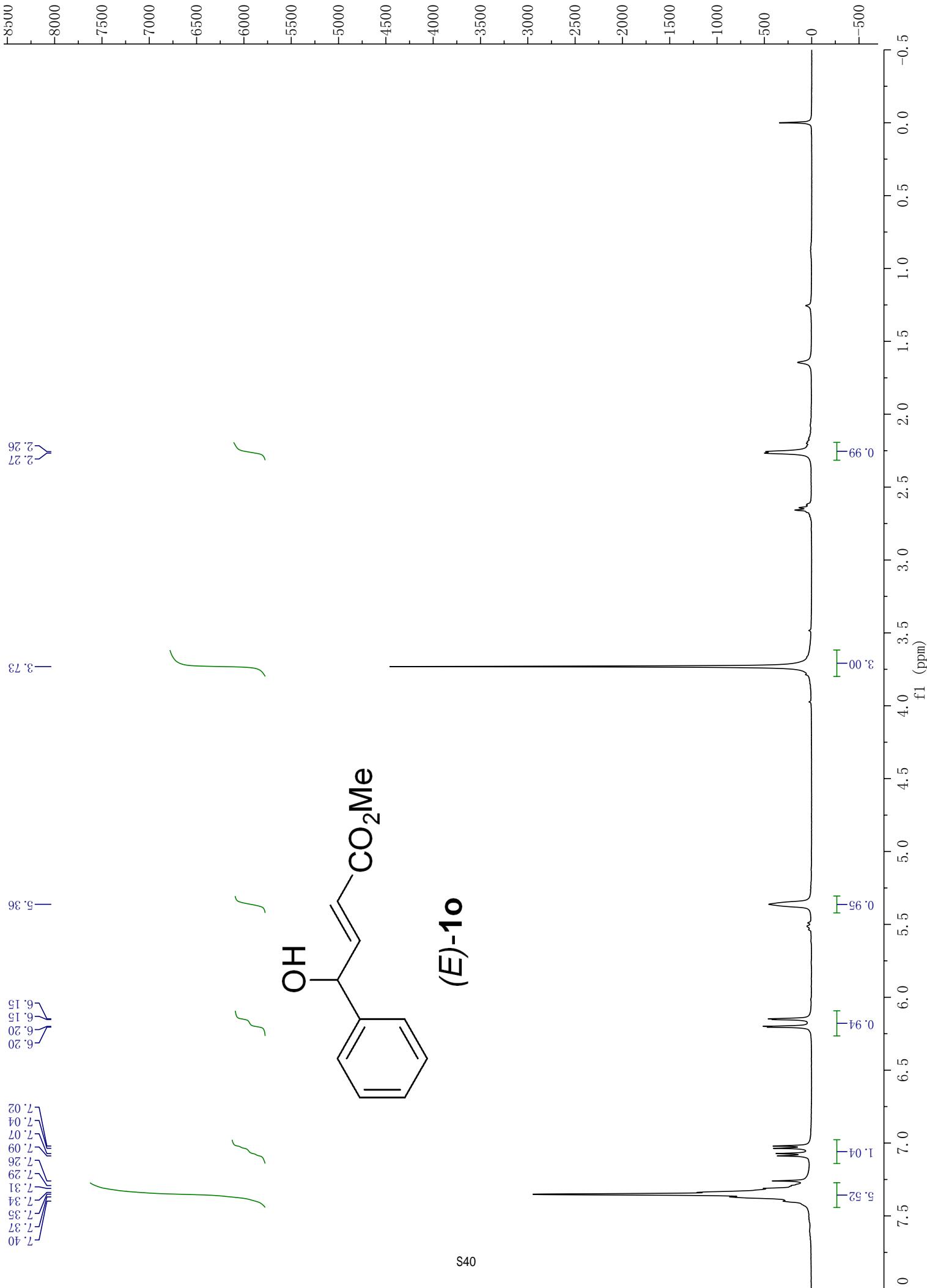
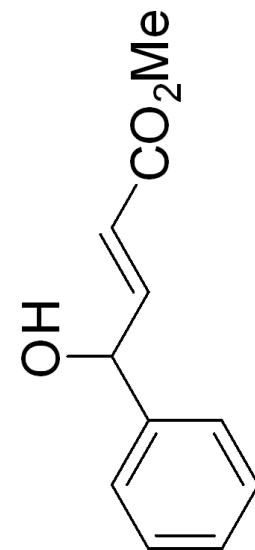
51.80  
33.37  
27.99

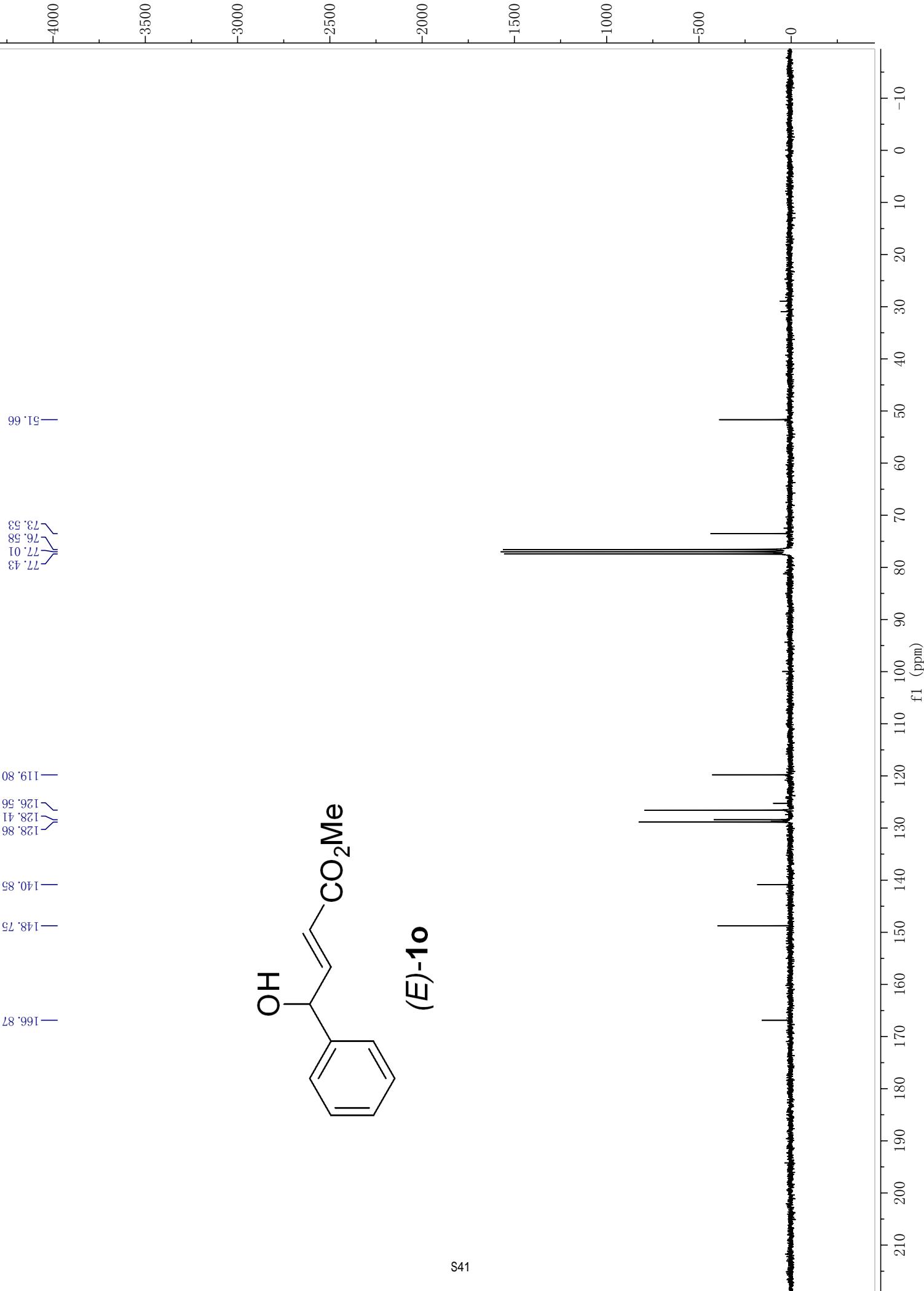
77.42  
77.00  
76.58



NAME 090709LAN  
EXENO 3  
PROCNO 1  
Date\_ 20090709  
Time 14.49  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zppg36  
TD 65536  
SOLVENT CDCl3  
NS 127  
DS 4  
SWH 18028.816 Hz  
FIDRES 0.25098 Hz  
AQ 1.8175818 sec  
RG 203  
DW 27.733 usec  
DE 6.50 usec  
TE 294.4 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.70 usec  
PL1 0.00 dB  
PL1W 29.38907051 W  
SF01 75.4732933 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waitz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 1.00 dB  
PL12 17.00 dB  
PL13 9.17820644 W  
PL2W 0.23034613 W  
PL12W 0.23034613 W  
PL13W 0.23034613 W  
SF02 300.1312005 MHz  
SI 322768  
SF 75.467753 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



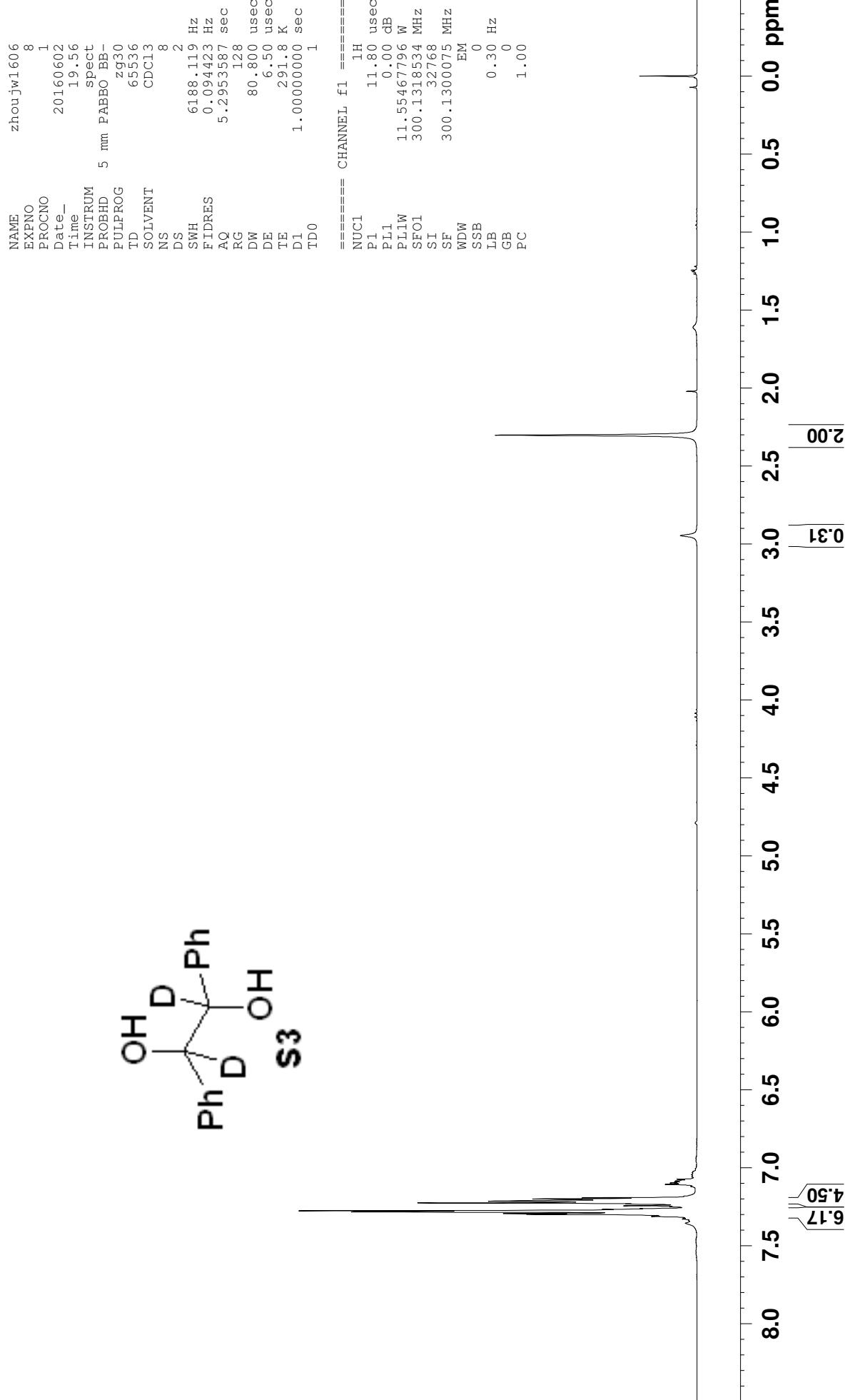
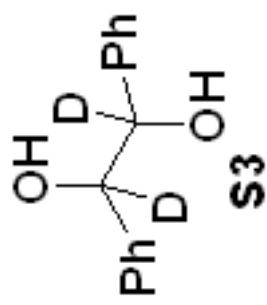


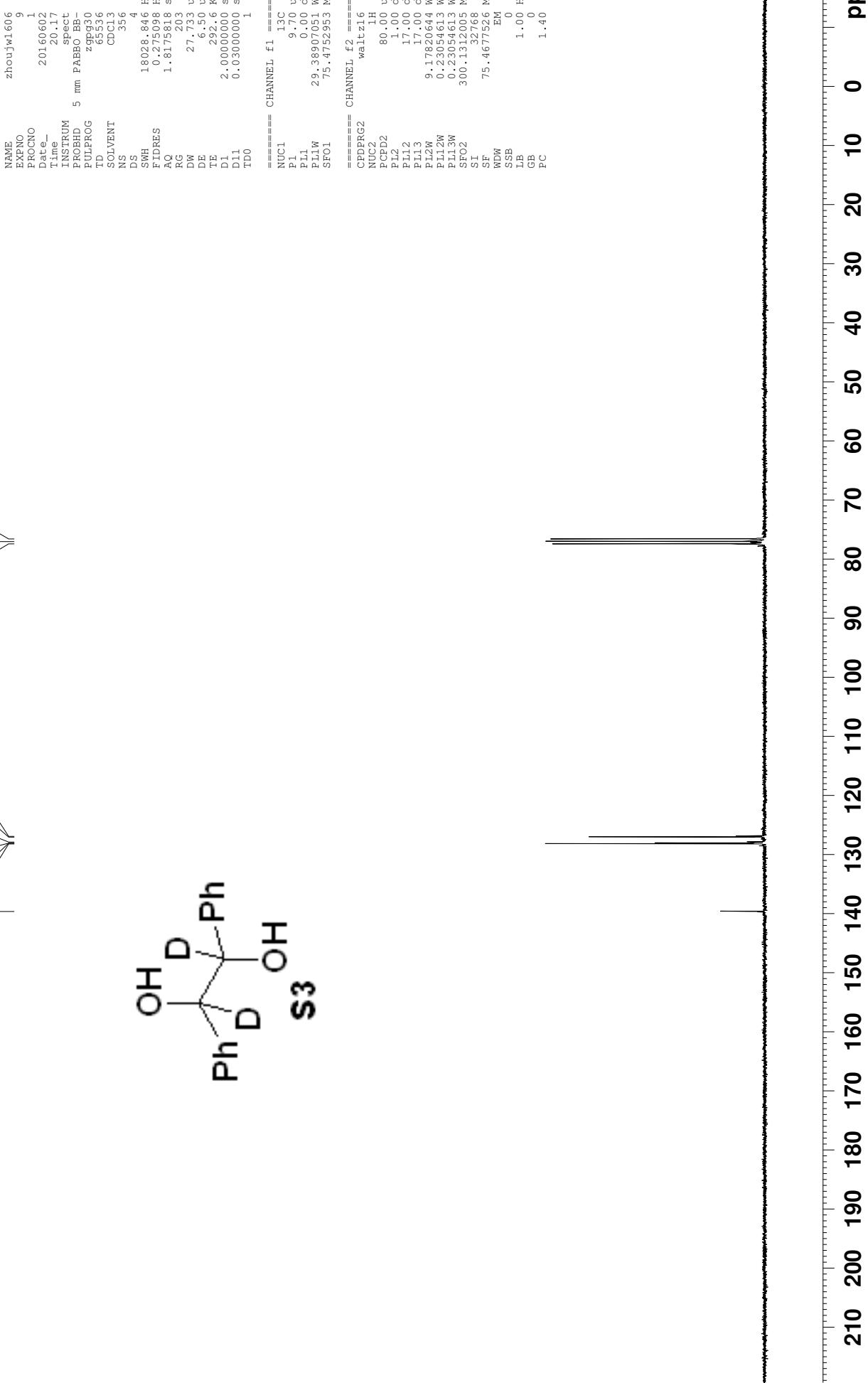


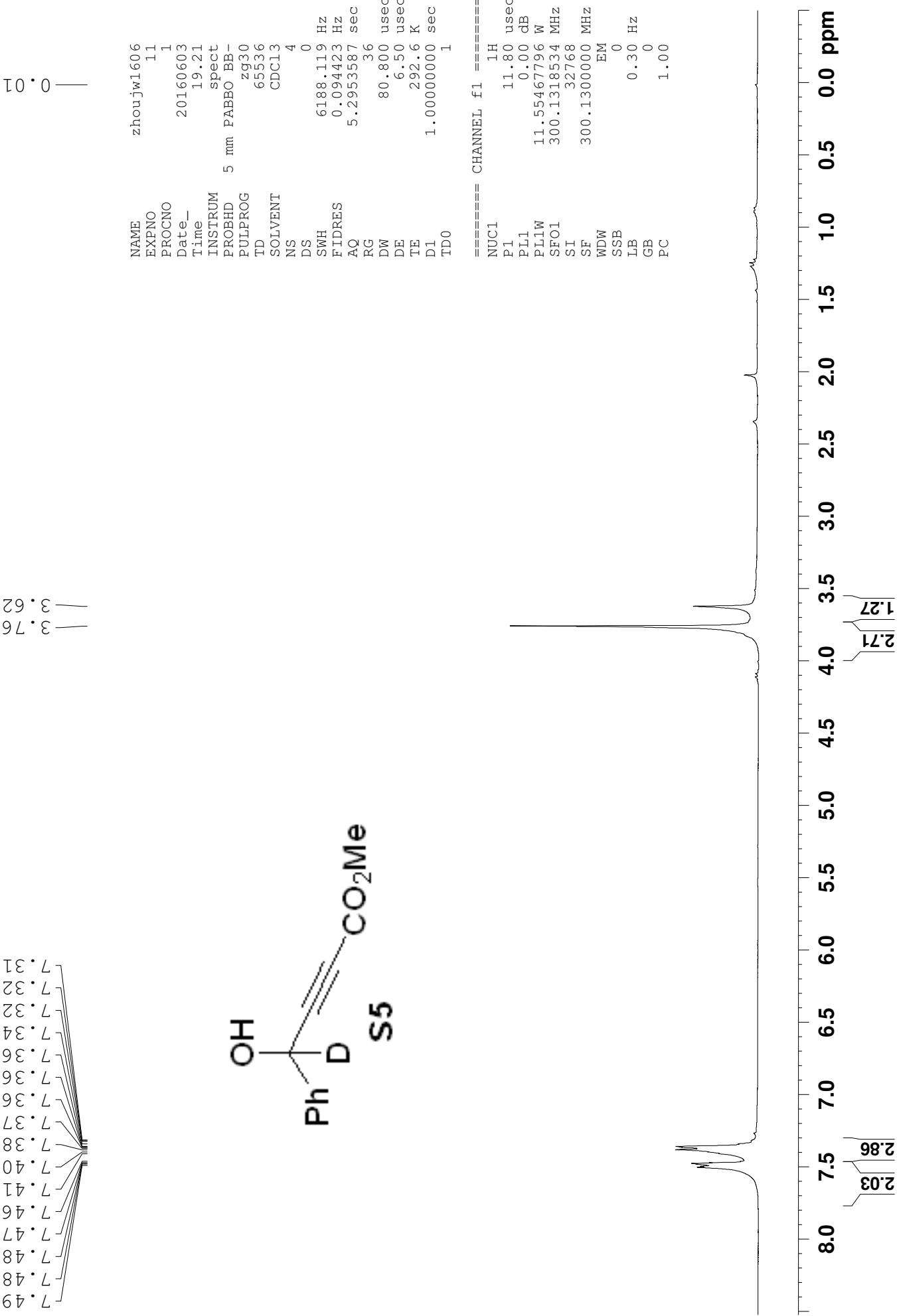
7.19  
7.20  
7.21  
7.22  
7.23  
7.24  
7.26  
7.27  
7.28  
7.29  
7.30  
7.31

2.30  
2.95

0.00









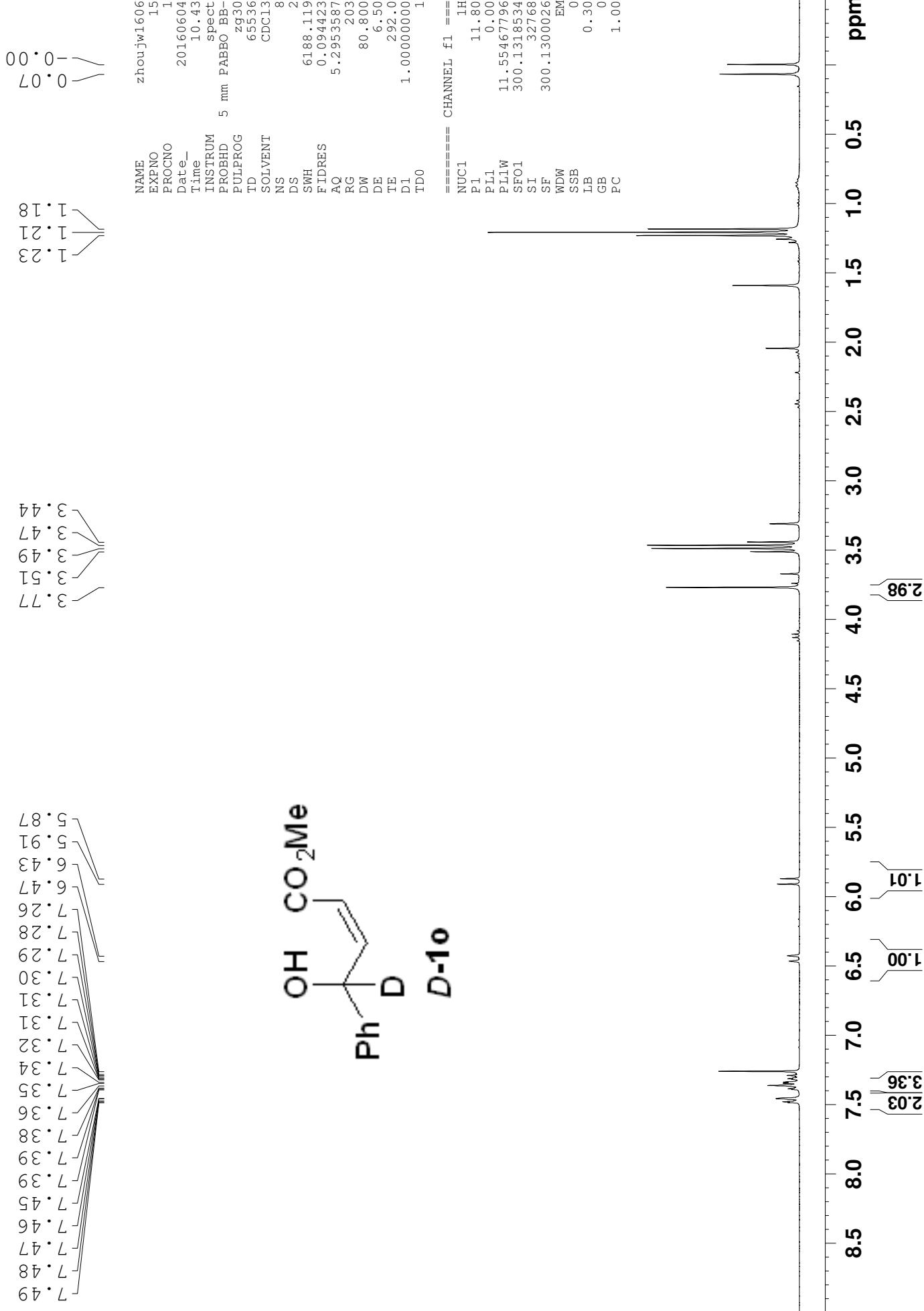
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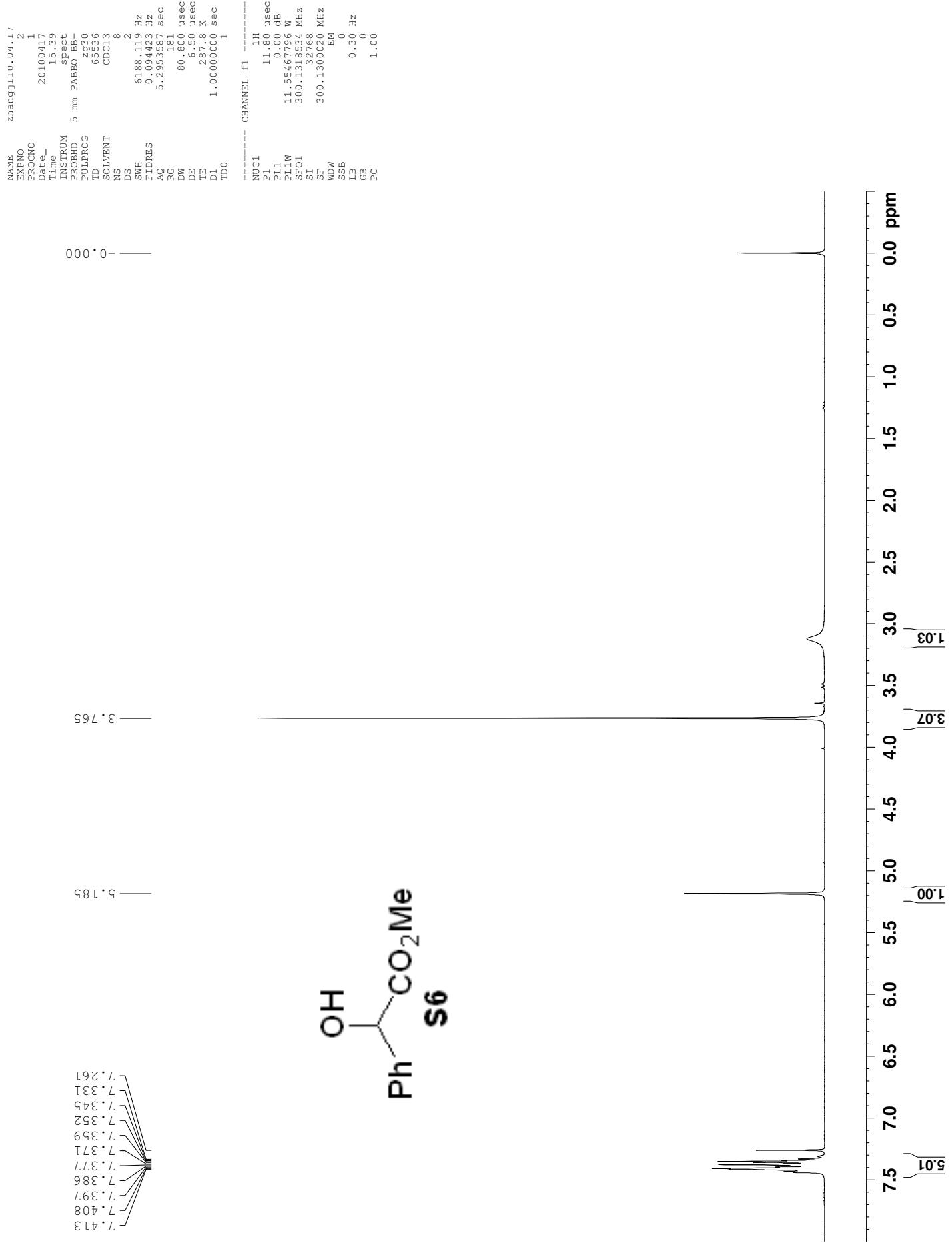
zhoujw1.606          52.84
EXPNO                12
PROCNO               1
Date_--              20160603
Time                 19.27
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zgpg30
TD                   65536
SOLVENT               CDC13
NS                  197
DS                    0
SWH                 18028.846 Hz
FIDRES              0.275098 Hz
AQ                   1.8175818 sec
RG                   203
DW                   27.733 usec
DE                   6.50 usec
TE                   292.6 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0                   1

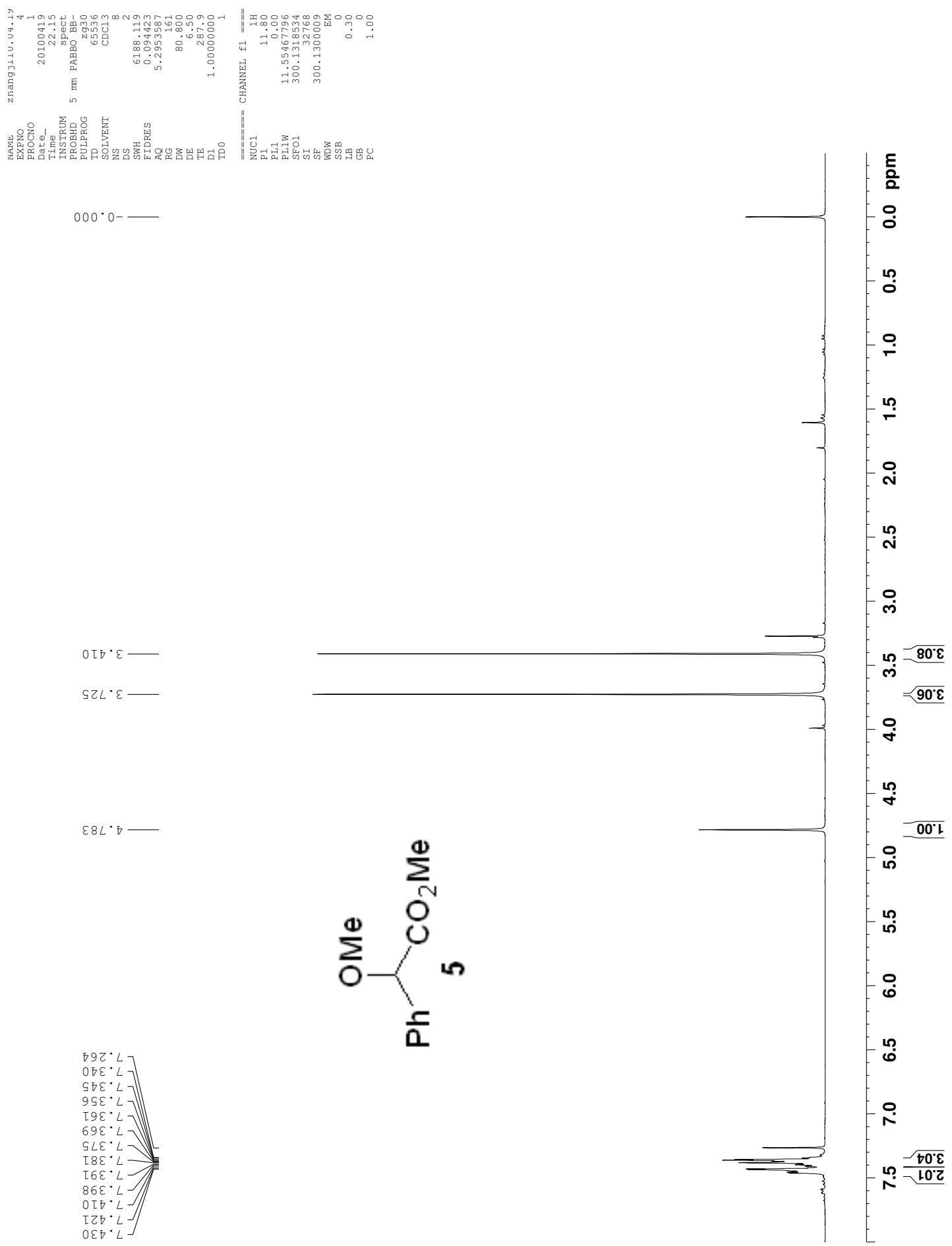
===== CHANNEL f1 =====
NUC1            13C
P1             9.70 usec
PL1            0.00 dB
PL1W           29.38907051 W
SFO1           75.472953 MHz

===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2            1H
PCPD2           80.00 usec
PL2             1.00 dB
PL12            17.00 dB
PL13            17.00 dB
PL2W           9.17820644 W
PL12W          0.23054613 W
PL13W          0.23054613 W
SFO2            300.1312005 MHz
SI              32768
SF              75.4677597 MHz
WDW             EM
SSB              0
LB              1.00 Hz
GB              0
PC              1.40

```





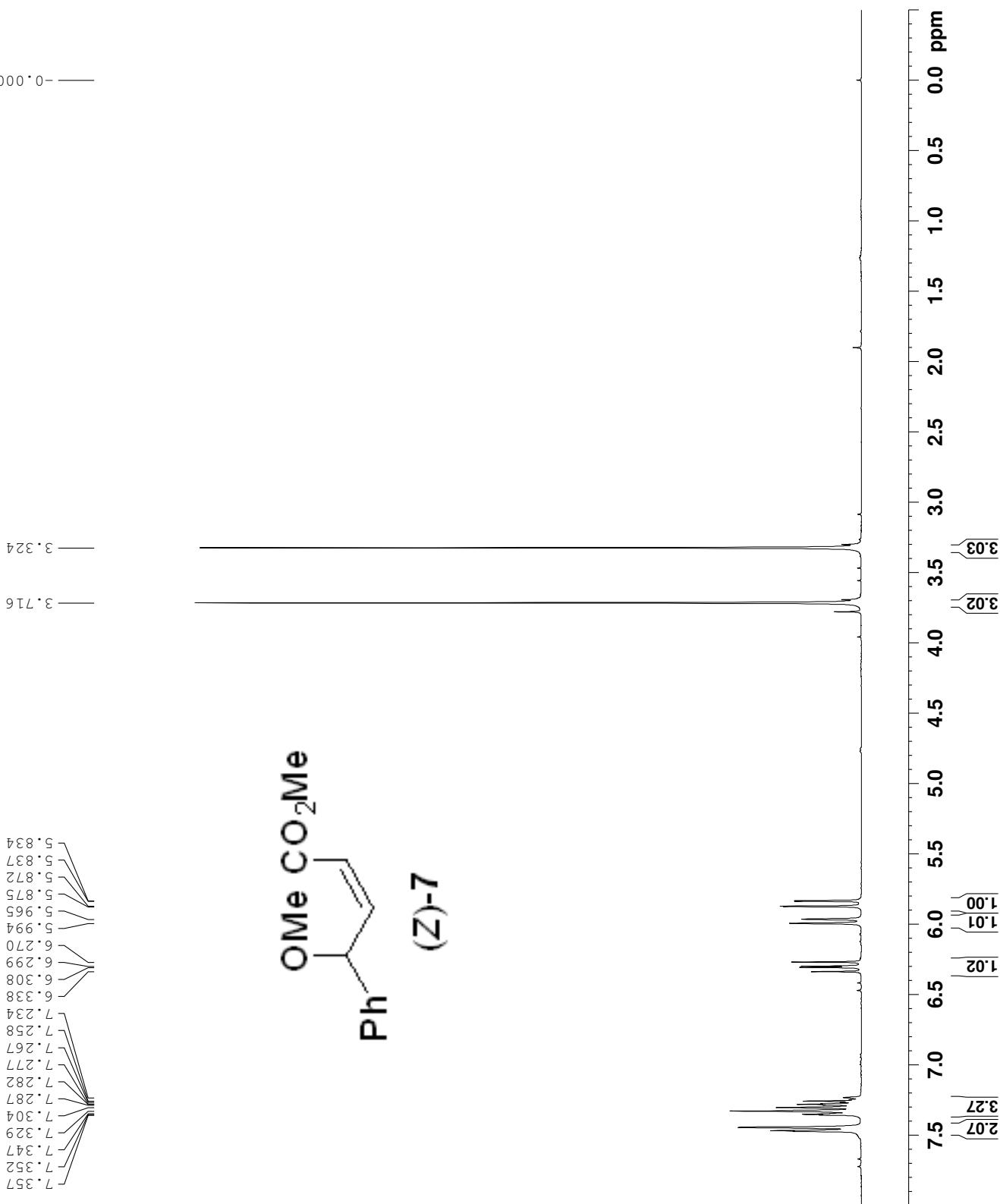


```

=====
      CHANNEL f1 =====
      zhangjiu.u,v,u /
      NAME          6
      EXPRO        1
      PROCG       20100507
      Date_       22.07
      Time_        22.07
      INSTRUM     spect
      PROBHD       5 mm PABBO BB-
      PULPROG      z930
      TD           6536
      SOLVENT      CDC13
      NS            8
      DS           2
      SWH          61.88-119 Hz
      FIDRES      0.054423 Hz
     AQ           5.293557 sec
      RG           14.2
      DW           80.800 usec
      DE           6.000 usec
      TE           28.9 K
      D1          1.0000000 sec
      TDO          1

=====
      CHANNEL f1 =====
      NUCCL        1H
      P1          11.80 usec
      PLL         0.00 dB
      PLW         11.5546776 W
      SFLOW      300.318534 MHz
      SI          300.1300101 MHz
      SF          322768
      WDW         EM
      SSB         0
      LB          0.30 Hz
      GB          0
      PC          1.00

```



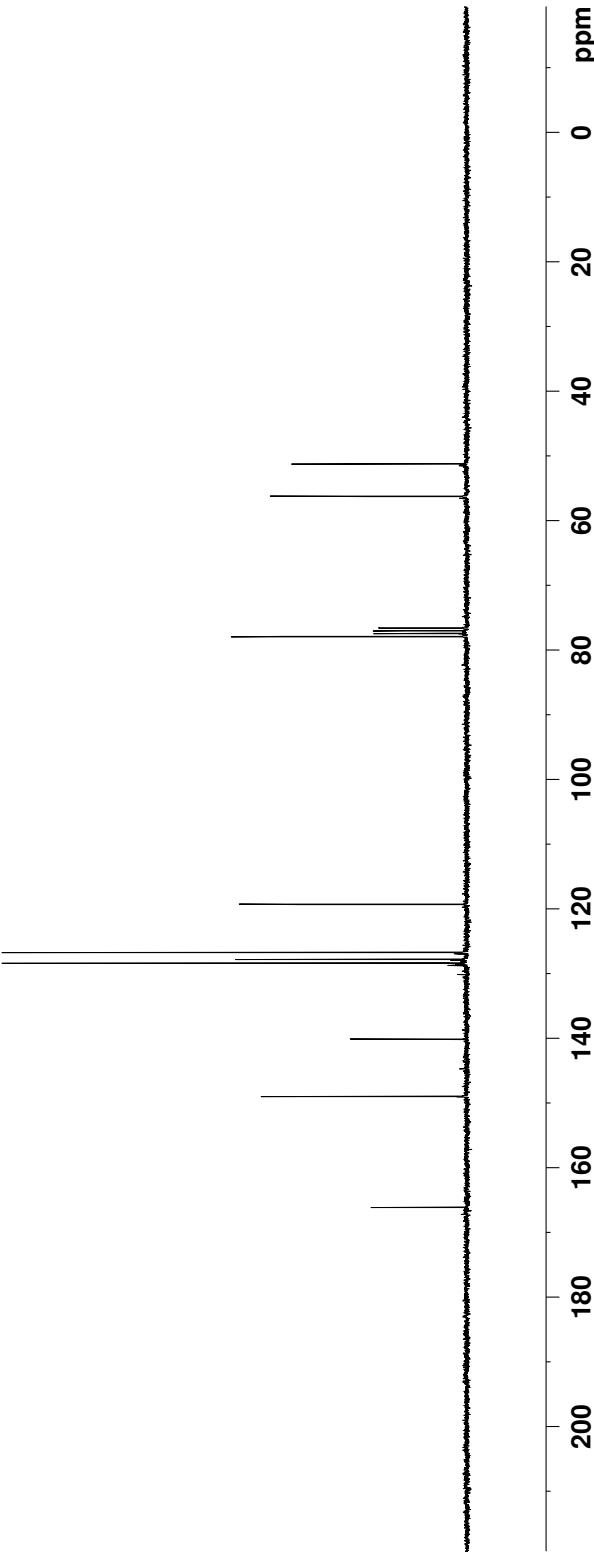
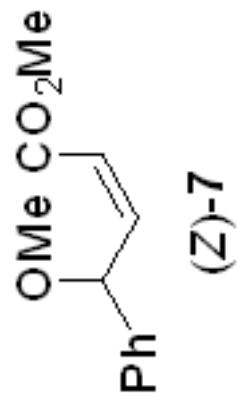
```

NAME          zhangj110.05.07
EXENO         7
PROCNO       1
Date_        20100507
Time_        22.12
INSTRUM      spect
PROBHD      5 mm PABBO BB-
PULPROG     ZGP936
TD           65536
SOLVENT      CDC13
NS            40
DS            4
SWH          18028.816 Hz
FIDRES      0.225098 Hz
AQ           1.8175818 sec
RG           203
DW           27.733 usec
DE           6.50 usec
TE           290.4 K
D1           2.0000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1         13C
P1           9.70 usec
PL1          0.00 dB
PL1W         29.38907051 W
SF01         75.4732933 MHz

===== CHANNEL f2 =====
CPDPGR22   waltz16
NUC2         1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          17.00 dB
PL13          17.00 dB
PL2W         9.17820644 W
PL12W        0.23034613 W
PL13W        0.23034613 W
SF02         300.1312005 MHz
SI           322.768
SF           75.4677619 MHz
WDW          0
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

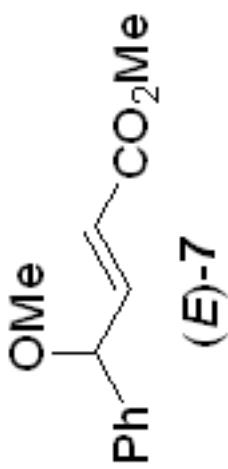
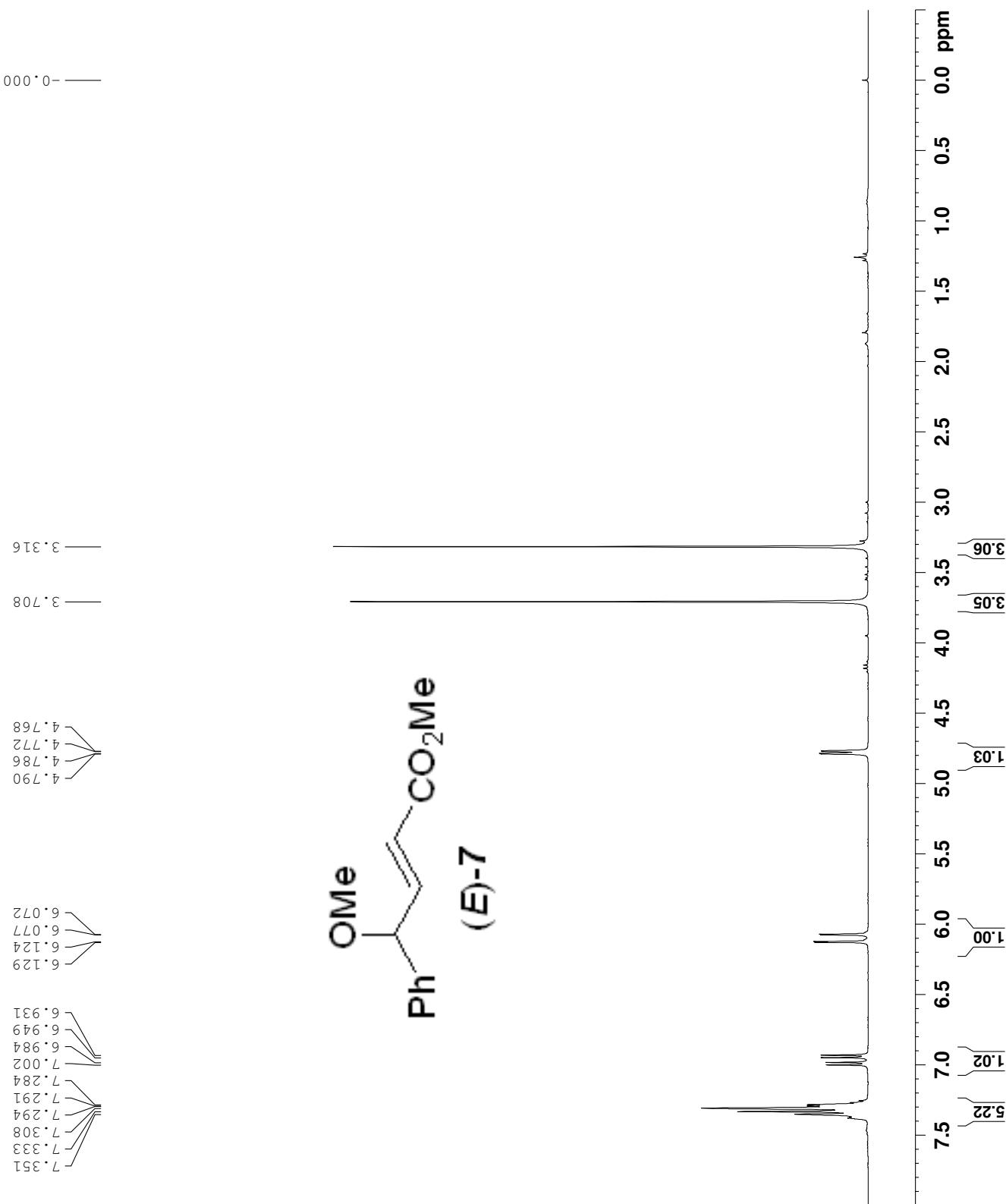
```



```

HAWES      zhangjiu.u.v.u/v
EXPNO      11
PROCNO     1
Date-      20100507
Time-      22.33
INSTRUM   MM
PROBOD    5 mm
TD        8192
SOLVENT   US
NS        128
SWH      2
WIDRES   2
AQ        0.004423 Hz
RG        5.293587 sec
RGXG      20.2
DW        80.800 usec
DE        6.50  usec
TE        28.98 K
TEDE     1.0000000 sec
D1        1
DDFO     1
=====
CHANNEL f1 =====
NUC1      11H
P1        11.80 usec
P1L      0.00 dB
P1LW     11.55467796 W
F01      300.1318534 MHz
SI        300.132768 MHz
NDW      EM
NDW      300.1300035 MHz
SSB      0.30 Hz
SSB      0.00
SSB      1.00
PC

```



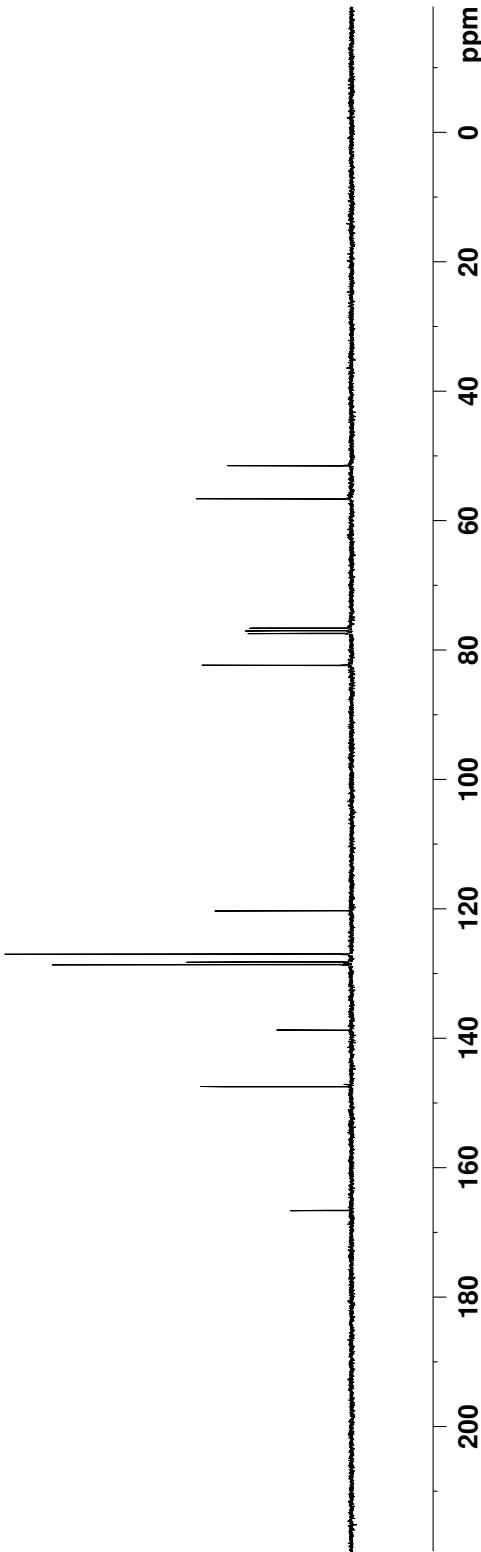
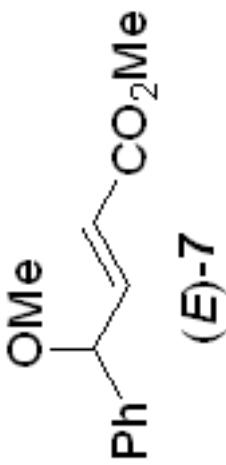
```

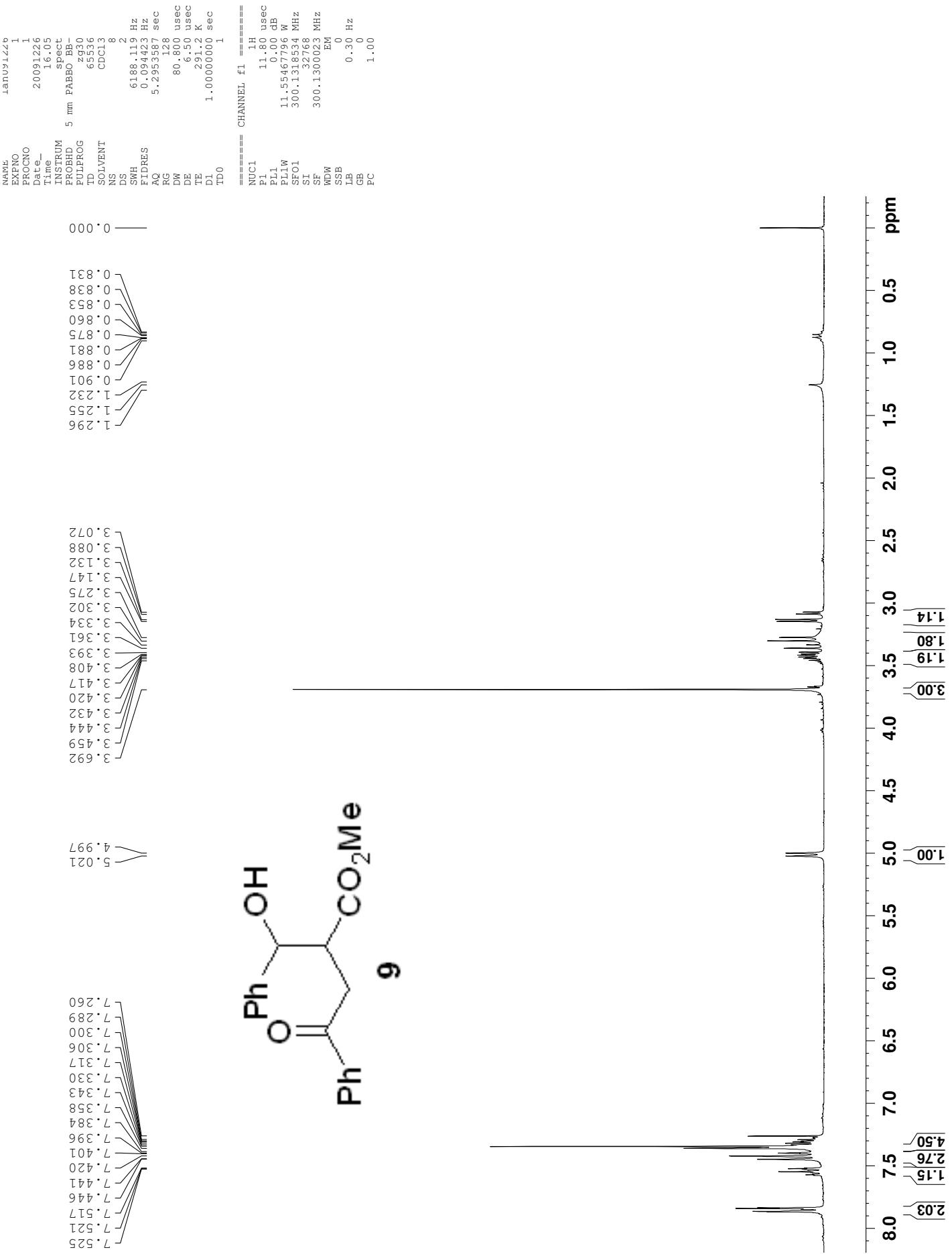
NAME      zhangj110.05.07
EXENO     1
PROCNO   1
Date_    20100507
Time_    22.38
INSTRUM spect
PROBHD  5 mm PABBO BB-
PULPROG zppg30
TD       65536
SOLVENT  CDC13
NS      53
DS       4
SWH     18028.816 Hz
FIDRES  0.225098 Hz
AQ      1.8175818 sec
RG      203
DW      27.733 usec
DE      6.50 usec
TE      290.6 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1    13C
P1      9.70 usec
PL1     0.00 dB
PL1W    29.38907051 W
SF01    75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2    1H
PCPD2   80.00 usec
PL2     1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W    0.23034613 W
PL12W   0.23034613 W
PL13W   300.1312005 MHz
SF02    75.467759 MHz
SI      EM
SF      0
WDW    1.00 Hz
SSB    0
LB      1.00 Hz
GB      0
PC      1.40

```





```

NAME      lan091226
EXENO     2
PROCNO   1
Date_    20091226
Time_    16.33
INSTRUM spect
PROBHD  5 mm PABBO BB-
PULPROG zppg36
TD       65536
SOLVENT  CDC13
NS      321
DS       4
SWH     18028.846 Hz
FIDRES  0.225098 Hz
AQ      1.8175818 sec
RG      27.733 usec
DW      6.500 usec
DE      292.2 K
TE      2.0000000 sec
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1    13C
P1      9.70 usec
PL1     0.00 dB
PL1W    29.38907051 W
SF01    75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2    1H
PCPD2   80.00 usec
PL2     1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W    0.23034613 W
PL12W   0.23034613 W
PL13W   300.1312005 MHz
SF02    75.4677430 MHz
SI      EM
SF      0
WDW    1.00 Hz
SSB    0
LB      1.00 Hz
GB      0
PC      1.40

```

37.78

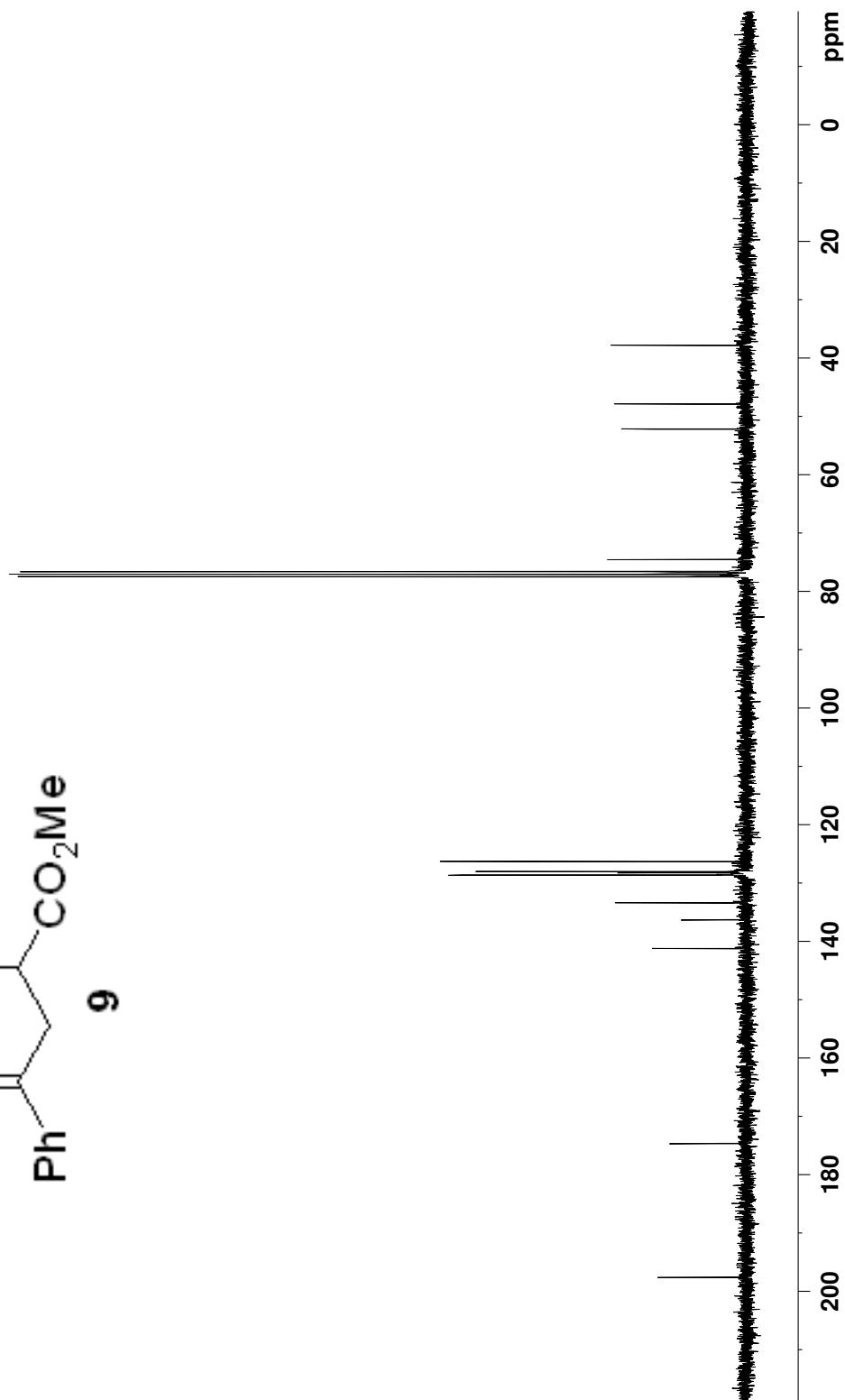
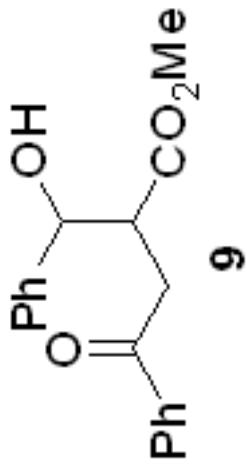
47.84

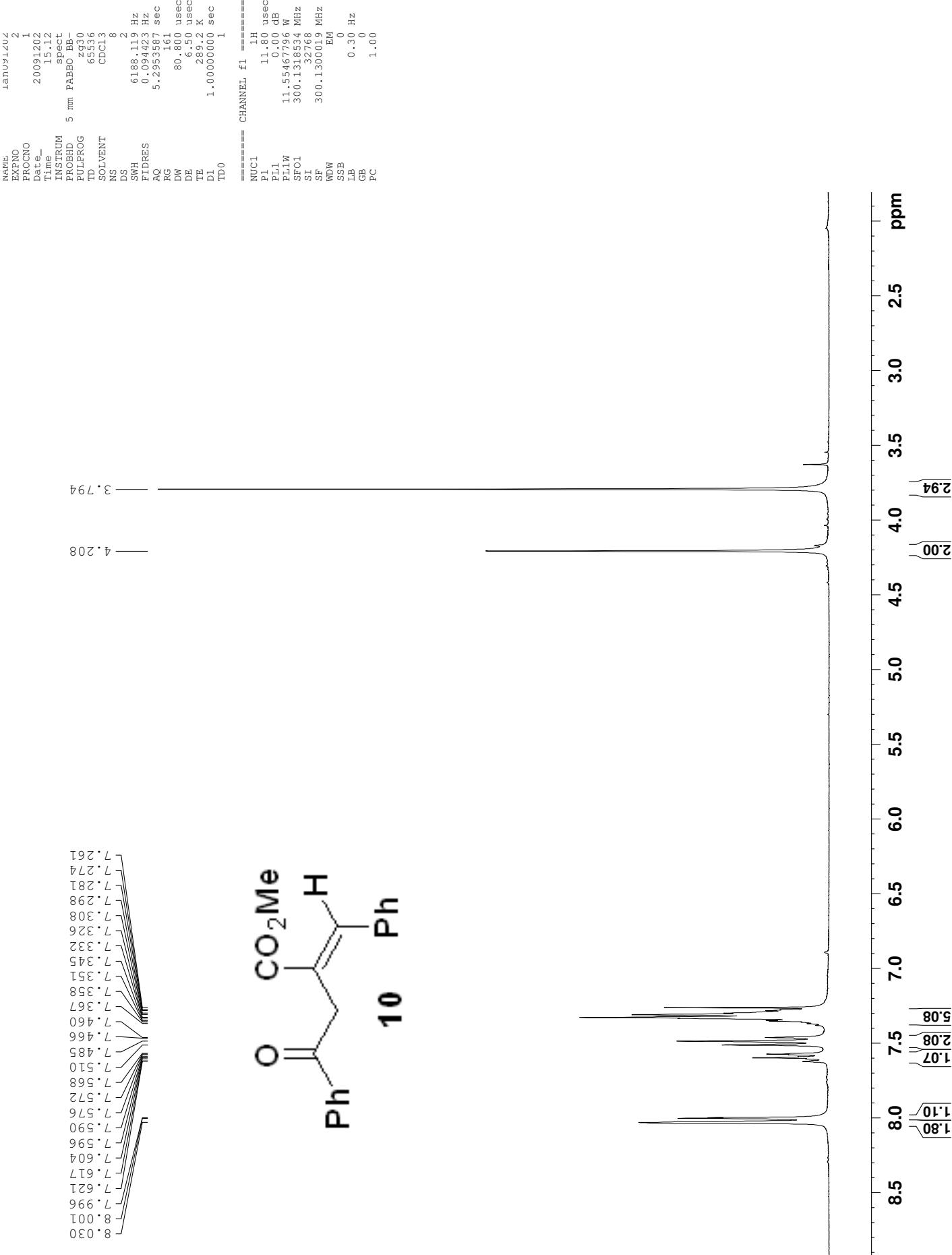
74.52  
76.60  
77.02  
77.45

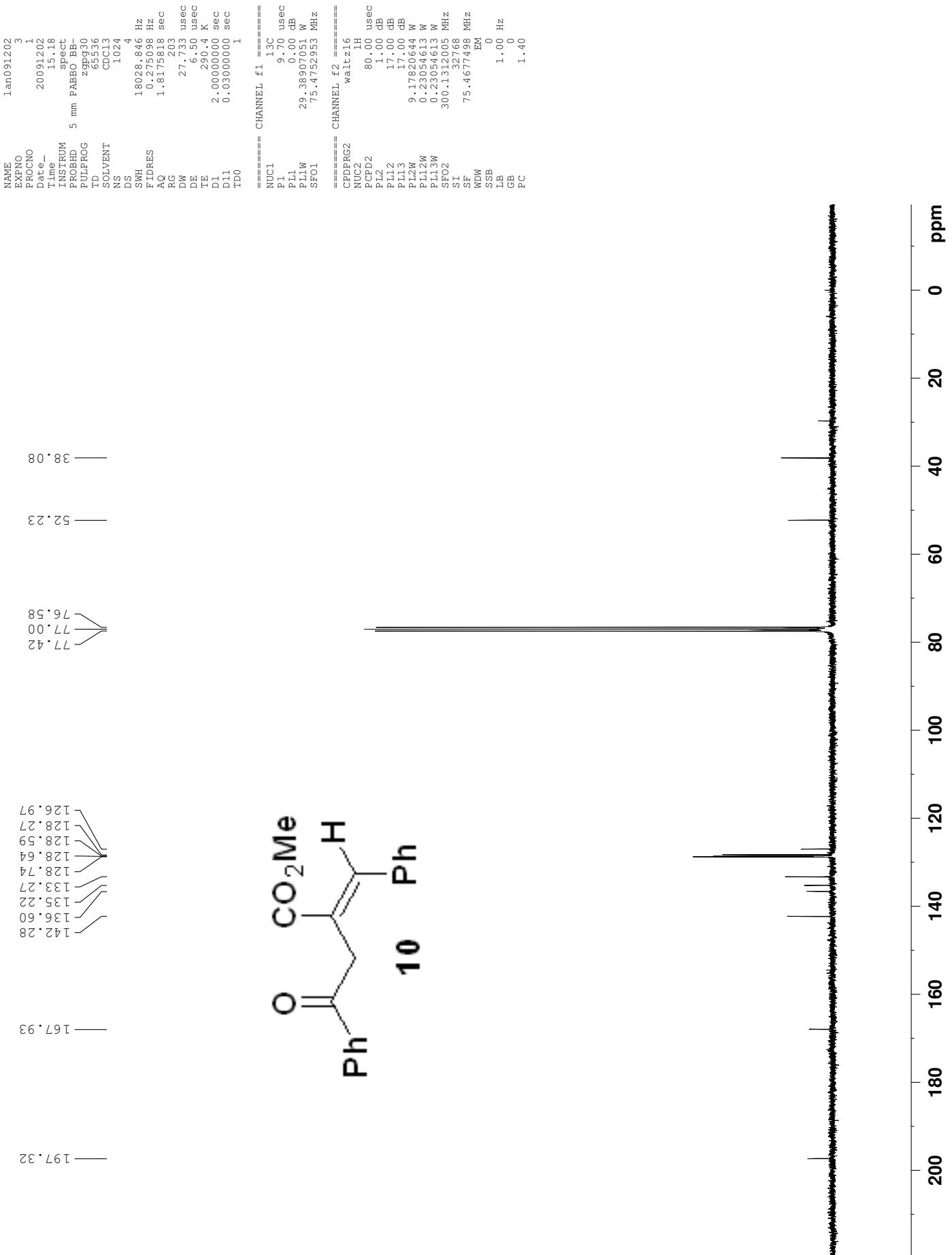
128.00  
128.22  
128.58  
128.63  
128.65  
131.22  
133.35  
136.30  
141.22

174.66

197.60





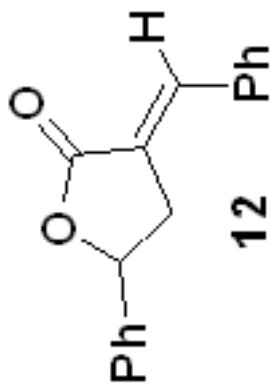
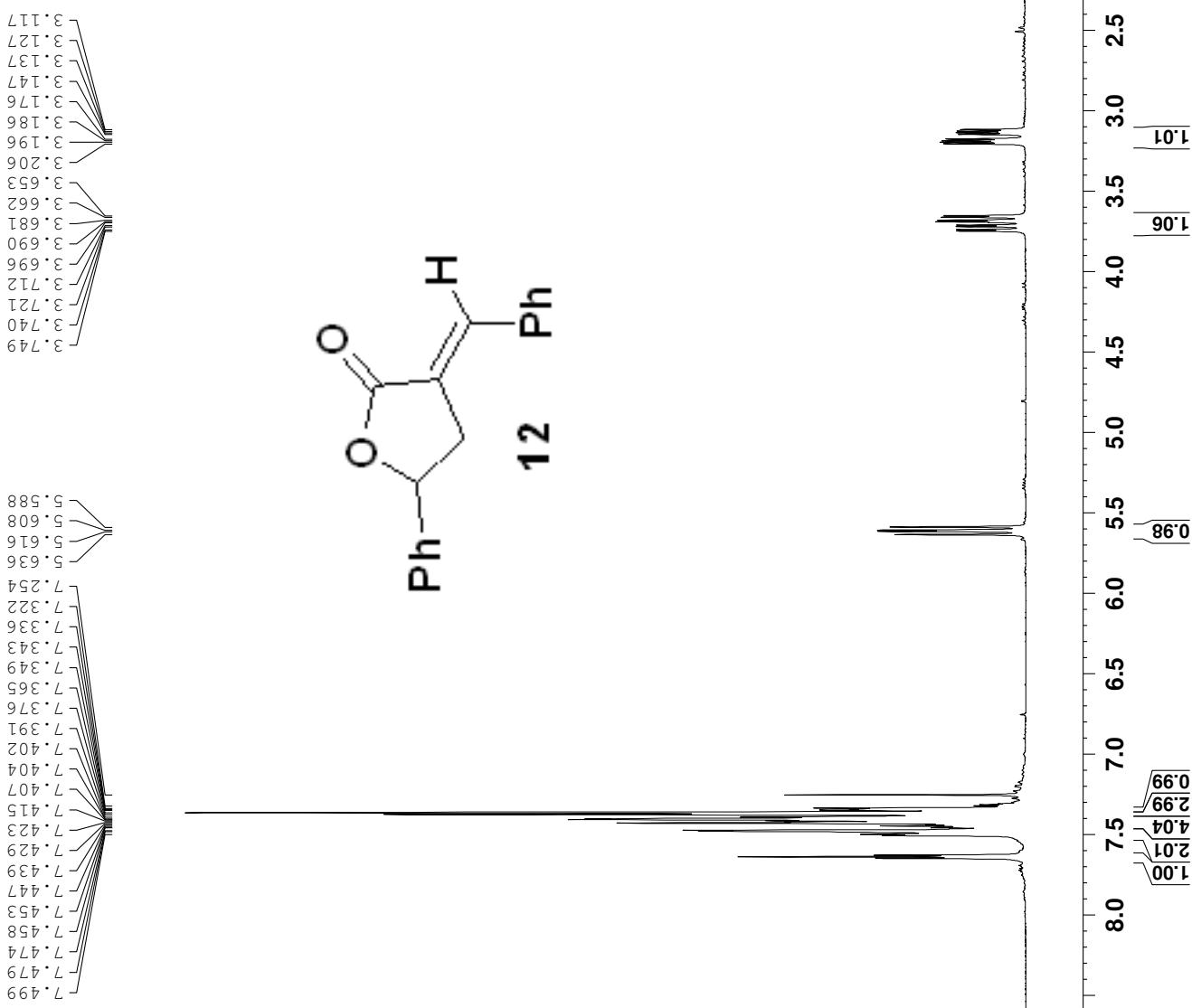


```

L I N E U P D A T E S U B J E C T
N A M E : F E X N O
P R O C D O : 1
D a t e _ : 20100507
T i m e _ : 17.59
I N S T R U M : P R O B H
S P E C T R U M : P A B O , B R -
P U L P R O G : 65536
T D : 65536
S O L V E N T : C D C L 3
N S : 8
D S : 2
S W H E : 61.88,119 Hz
F E T T E R S : 0.054422 Hz
A Q : 5.2933587 sec
R G : 557
D E : 80..800 usec
D E : 6.50 usec
T E : 289.4 K
D I : 1.0000000 sec
T D O : 1

===== CHANNEL f1 =====
NUC1 : 1 H
P1 : 1.00 usec
PL1 : 0.00 dB
P1W : 11.5546776 W
SF01 : 300.1318534 MHz
SI : EM
WDW : 0.0 Hz
SSB : 0.0 MHz
LB : 0..30 Hz
GBC : 0.0
PC : 1.00

```



```

NAME      LinLi20100520
EXENO     2
PROCNO    1
Date_     20100520
Time      19.05
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpp36
TD       65536
SOLVENT   CDCl3
NS        994
DS        4
SWH      18028.816 Hz
FIDRES   0.25098 Hz
AQ        1.8175818 sec
RG        203
DW       27.733 usec
DE        6.50 usec
TE       290.6 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1        9.70 usec
PL1      0.00 dB
PL1W    29.38907051 W
SF01    75.4732933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      1.00 dB
PL12    17.00 dB
PL13    9.17820644 W
PL2W    0.23034613 W
PL12W   0.23034613 W
PL13W   300.1312005 MHz
SF02    75.4677530 MHz
SI        EM
SF        0
WDW     1.00 Hz
SSB      0
LB        1.00 Hz
GB        0
PC        1.40

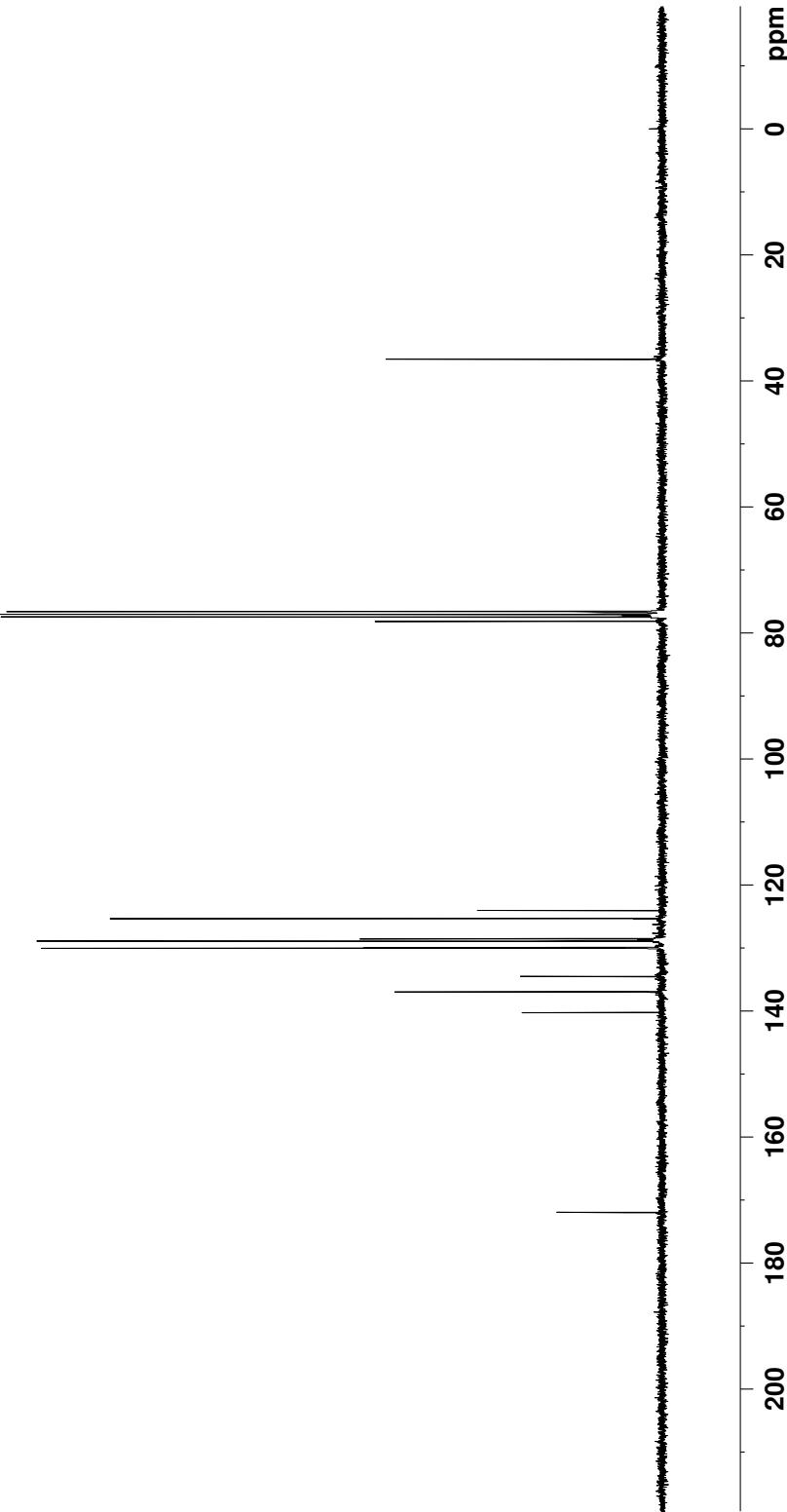
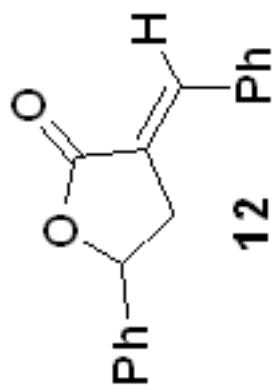
```

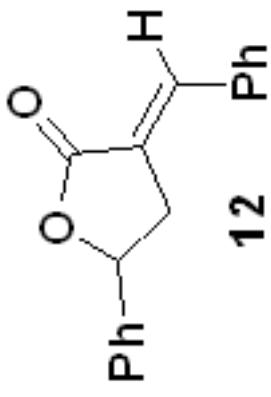
— 36.50 —

78.12  
77.42  
77.00  
76.58

140.23  
136.95  
134.48  
130.00  
129.92  
128.89  
128.84  
125.32  
124.02

— 171.95 —





136.95  
130.00  
129.92  
128.92  
128.89  
128.84  
128.84  
130.00  
134.48  
136.23  
140.23  
124.02  
125.32  
128.51  
128.84  
128.89  
128.92  
130.00  
134.48  
136.23  
140.23

36.50  
—  
78.12  
—

```

NAME: LiniLi20100520
EXPTNO: 1
PROCNO: 3
Date: 20100520
Time: 19.09
INSTRUM: 5 mm PABBBB
PROBODIM: 5 mm
PULPROG: TD
TD: 65536
SOLVENT: CDCl3
NS: 66
SWH: 18028.84 Hz
SF: 0.271088 Hz
AQ: 1.817818 sec
RG: 203
DW: 27.733 usec
DE: 6.500 usec
TE: 0.000000 K
CPSI: 145.000000 sec
D1: 2.0000000 sec
D2: 0.00044828 sec
D12: 0.00002000 sec
TDD: 1

===== CHANNEL f1 =====
NUCL: 13C
NUCL: 1H
P1: 9.70 usec
P2: 19.40 usec
PL1: 0.00 dB
PL1W: 29.38807051 W
SF01: 75.4752953 MHz

===== CHANNEL f2 =====
NUCL: 1H
P1: 13.00 usec
PL1: 0.00 dB
PL1W: 1.00 dB
P2: 17.00 dB
PL2: 9.17826164 W
PL2W: 0.39356163 W
SF02: 300.1327058 MHz
ST: 327058
SF: 75.4977556 MHz
NOW: EM
SSB: 0
LB: 1.00 Hz
GB: 1.0
PC: 1.40

```

