

# Synthesis of $\alpha$ -Nitro Carbonyls via Nitrations in Flow

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## Corresponding Author

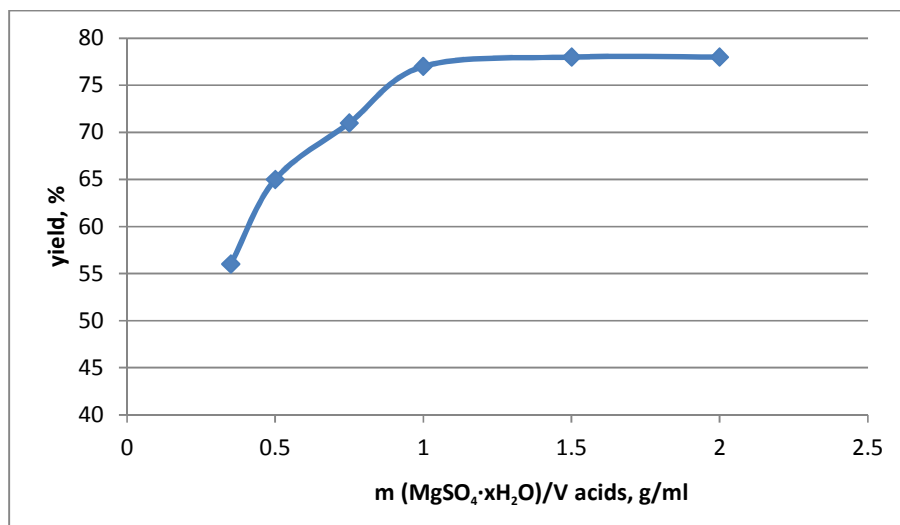
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§ These authors contributed equally to this work

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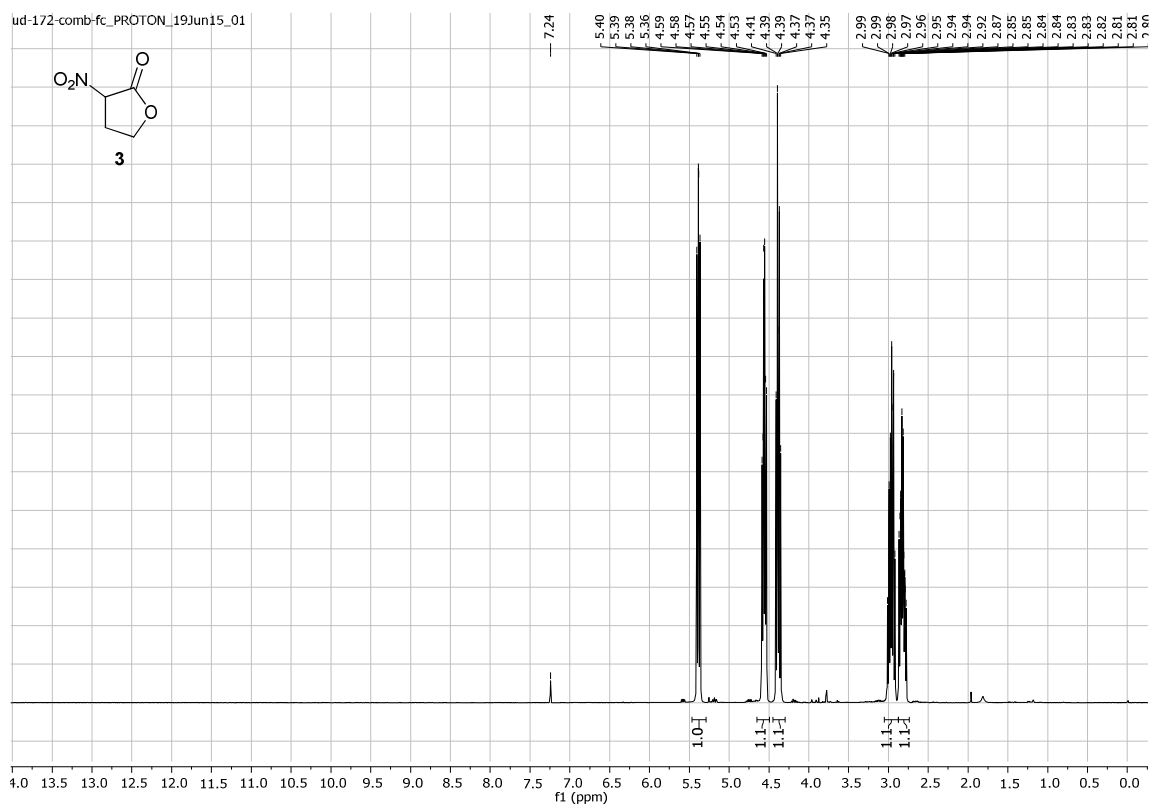
Optimization experiments to determine minimum amount of desiccant needed for the quenching procedure:



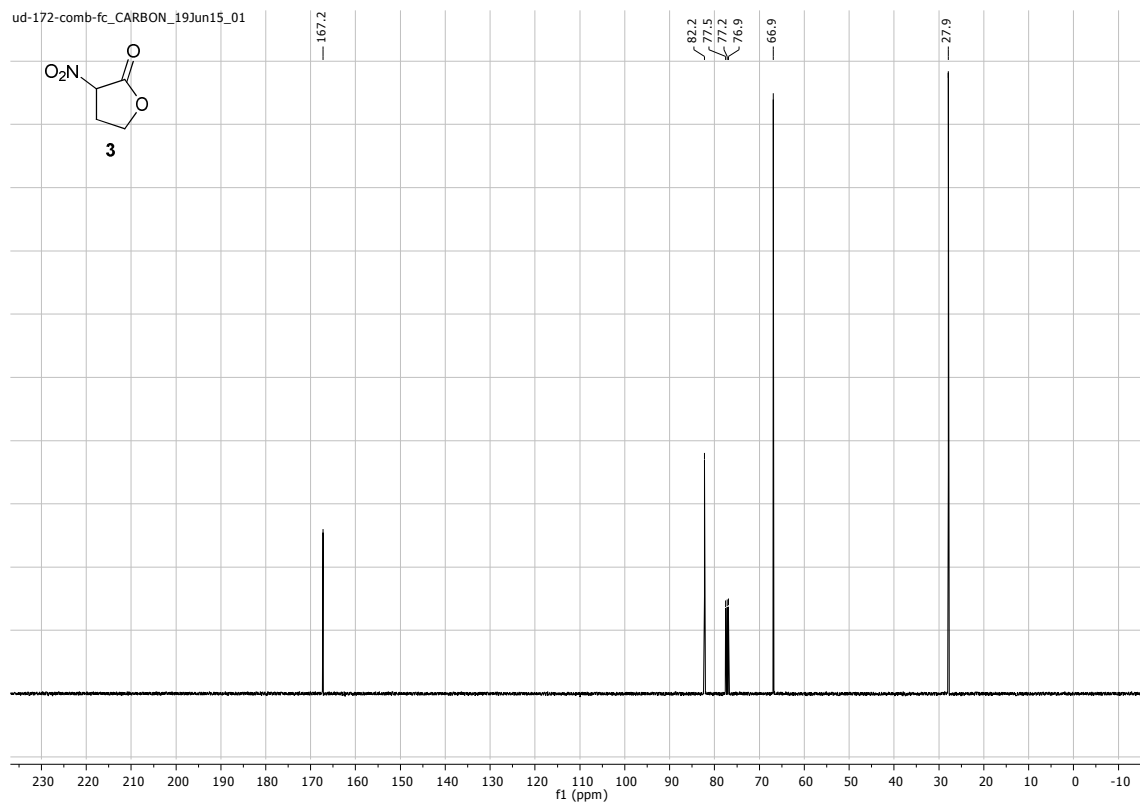
**Figure 1:** Dependence of product yield on amount of desiccant<sup>[a]</sup>

[a] Condition: Nitric acid (90+%, 16  $\mu$ L/min, 0.343 mmol/min) was mixed with sulfuric acid (96%, 103  $\mu$ L/min, 1.85 mmol/min) at 5 °C (cooling bath was used) using a T-mixer. The resulting flow stream was passed through 0.03 mL PTFE-tubing and mixed with the  $\alpha$ -acetylbutyrolactone solution (0.36 M in CH<sub>2</sub>Cl<sub>2</sub>, 0.663 mL/min, 0.239 mmol/min) using second T-mixer at the same temperature. The biphasic mixture was passed through 0.6 mL PTFE-tubing and collected in an Erlenmeyer flask containing a stirred suspension of MgSO<sub>4</sub> in CH<sub>2</sub>Cl<sub>2</sub> (20 mL) at room temperature. For 11 mL of the collected solution different amount of MgSO<sub>4</sub> was used, the suspension was stirred on average 14 min starting from the moment of collection. The reaction mixture was filtered, washed with CH<sub>2</sub>Cl<sub>2</sub>, treated with MeOH (2 mL) and concentrated under reduced pressure (30 °C). The yield was determined by <sup>1</sup>H NMR using mesitylene as an internal standard.

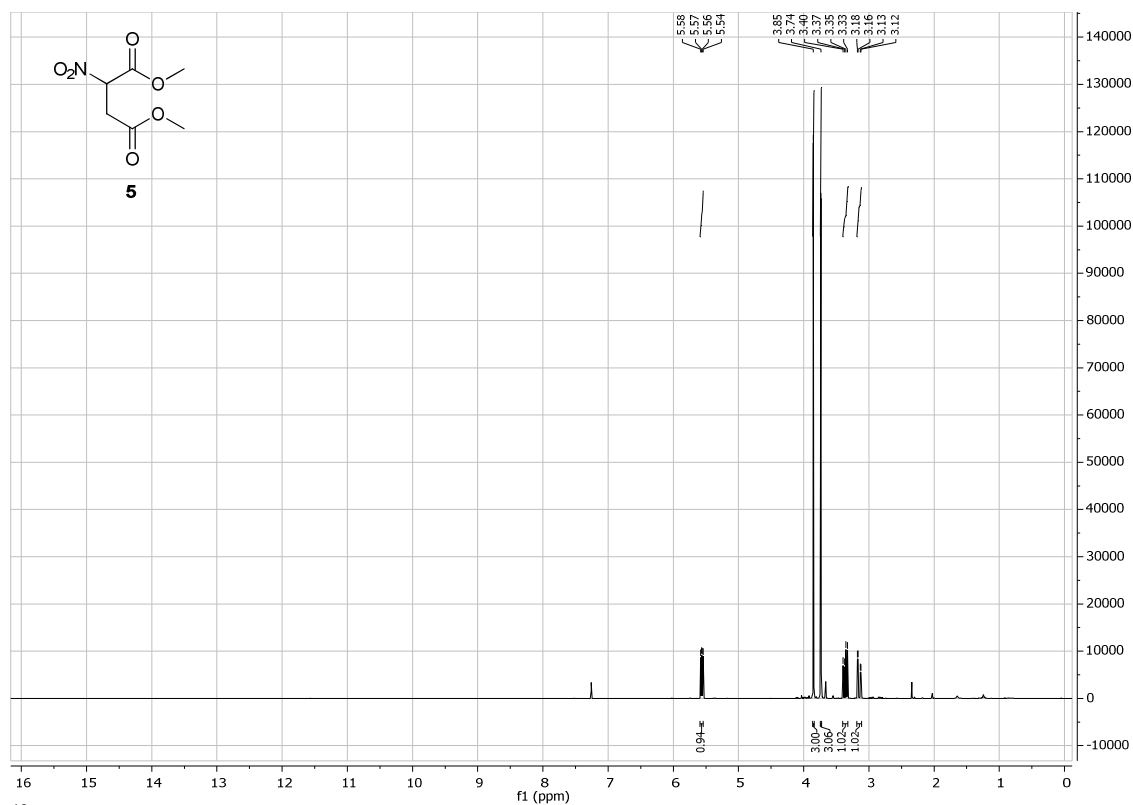
**NMR spectra:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



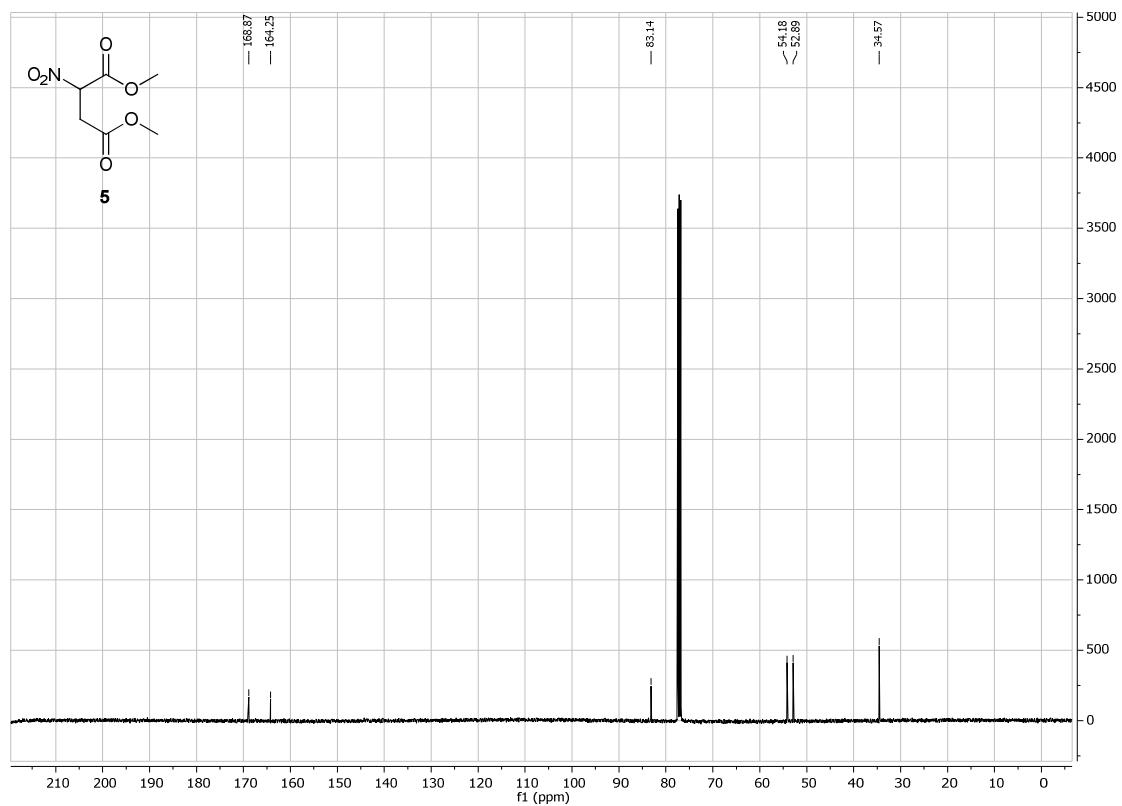
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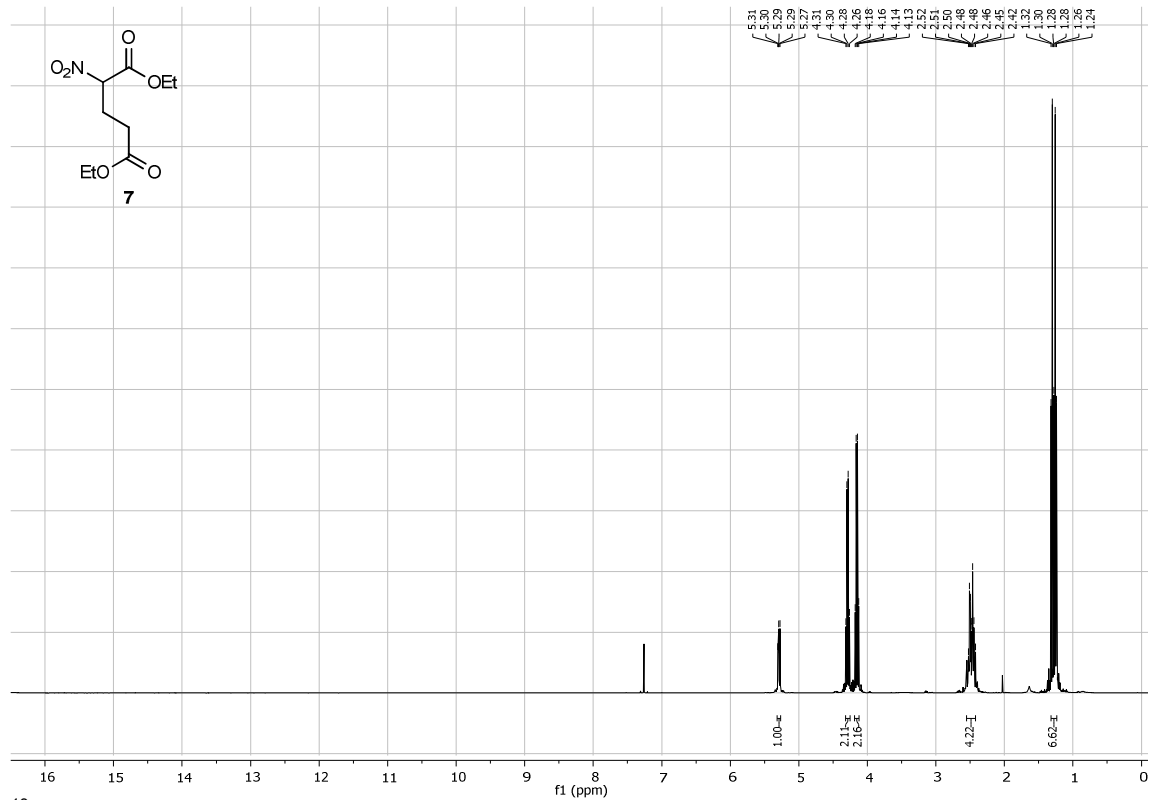
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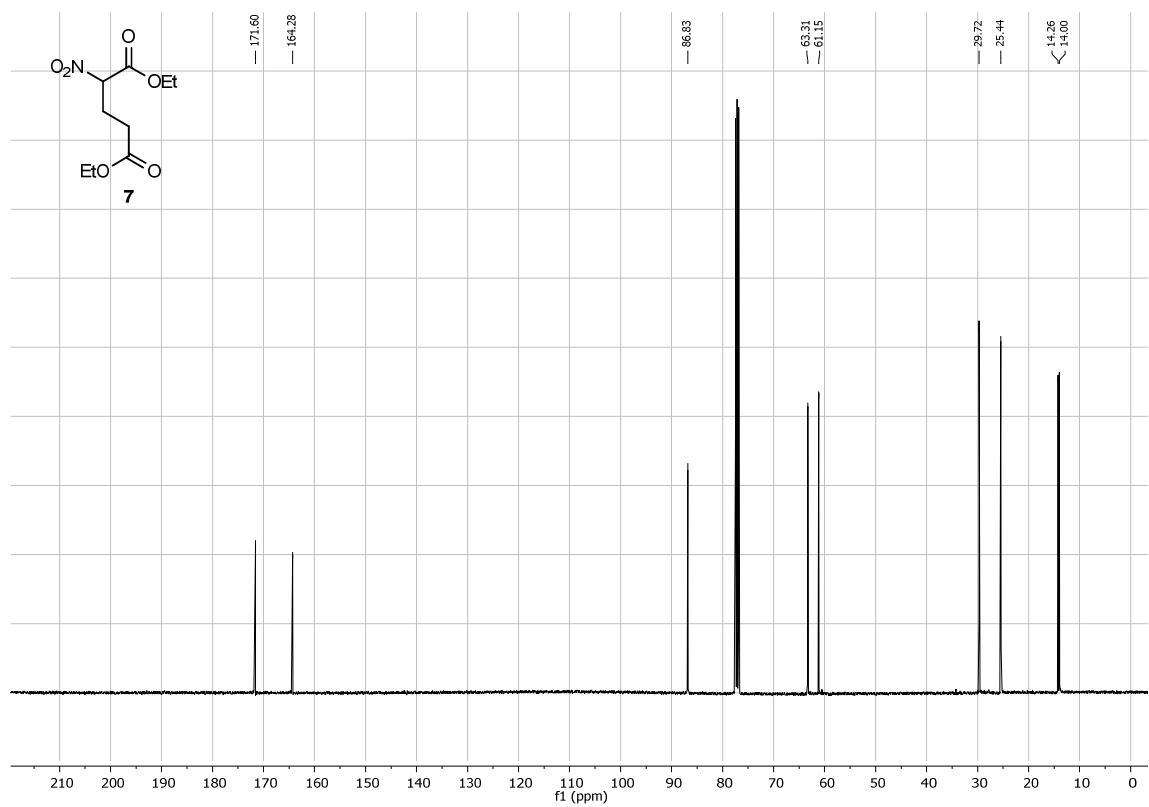
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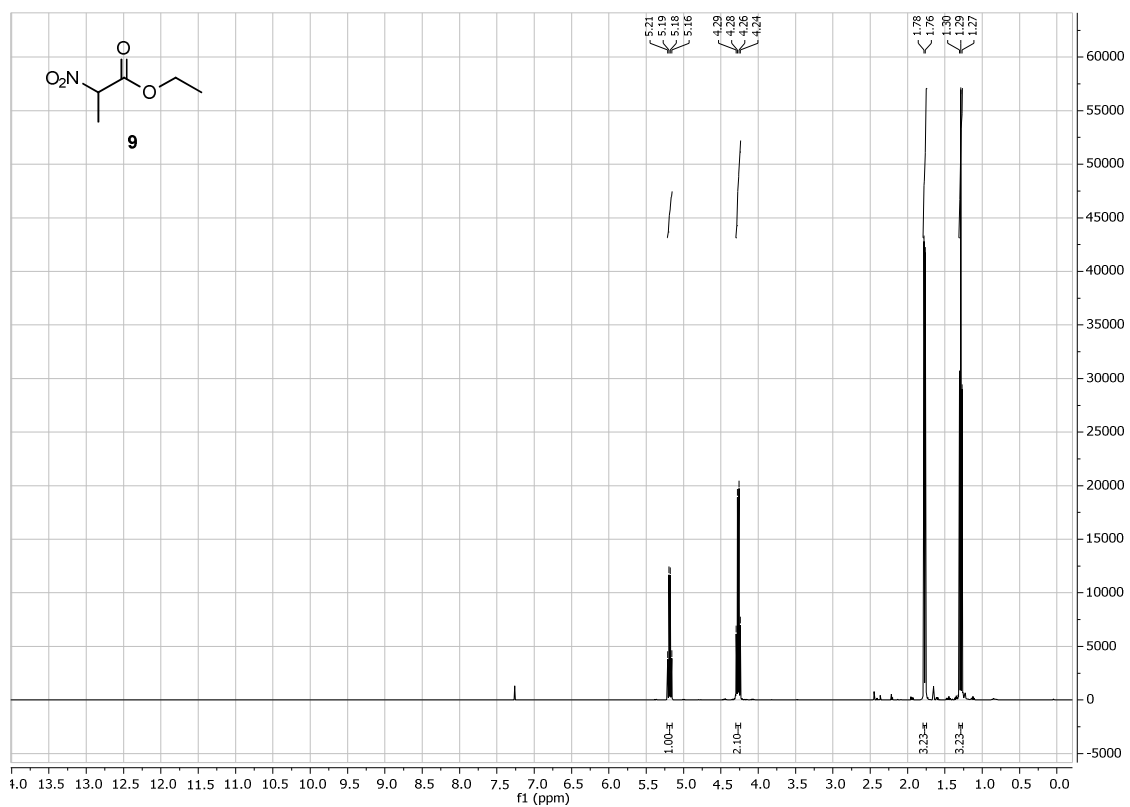
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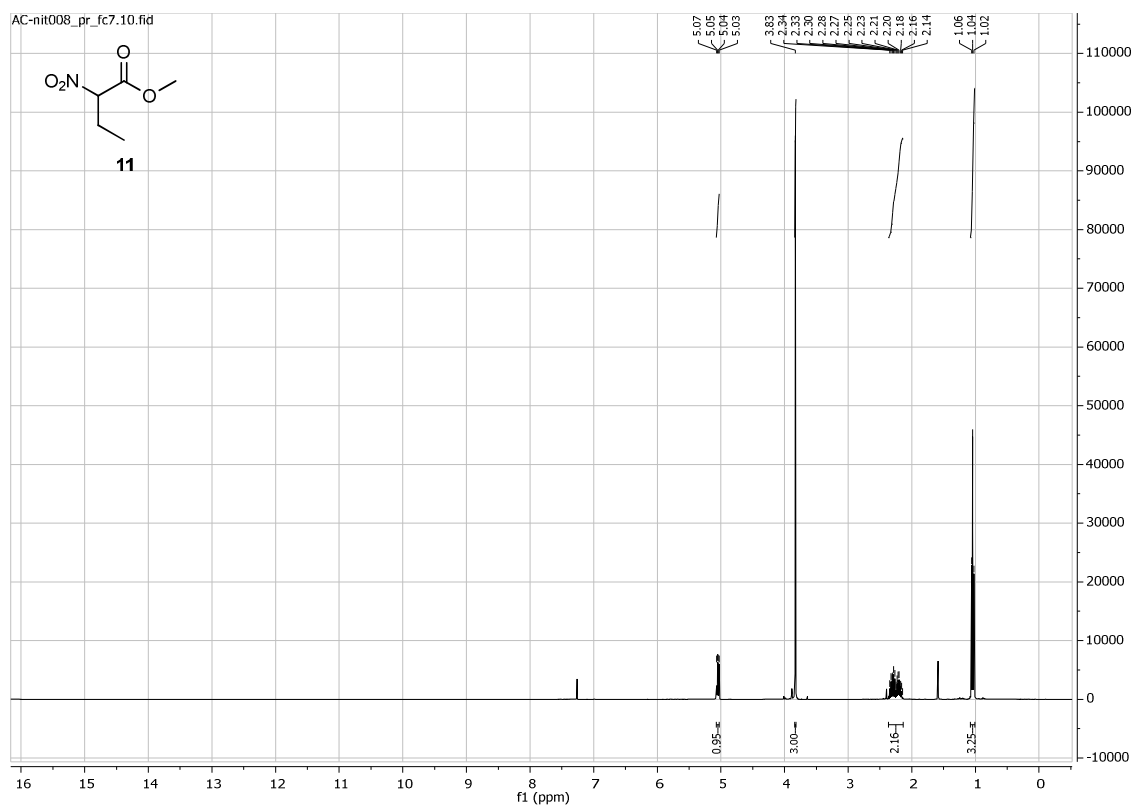
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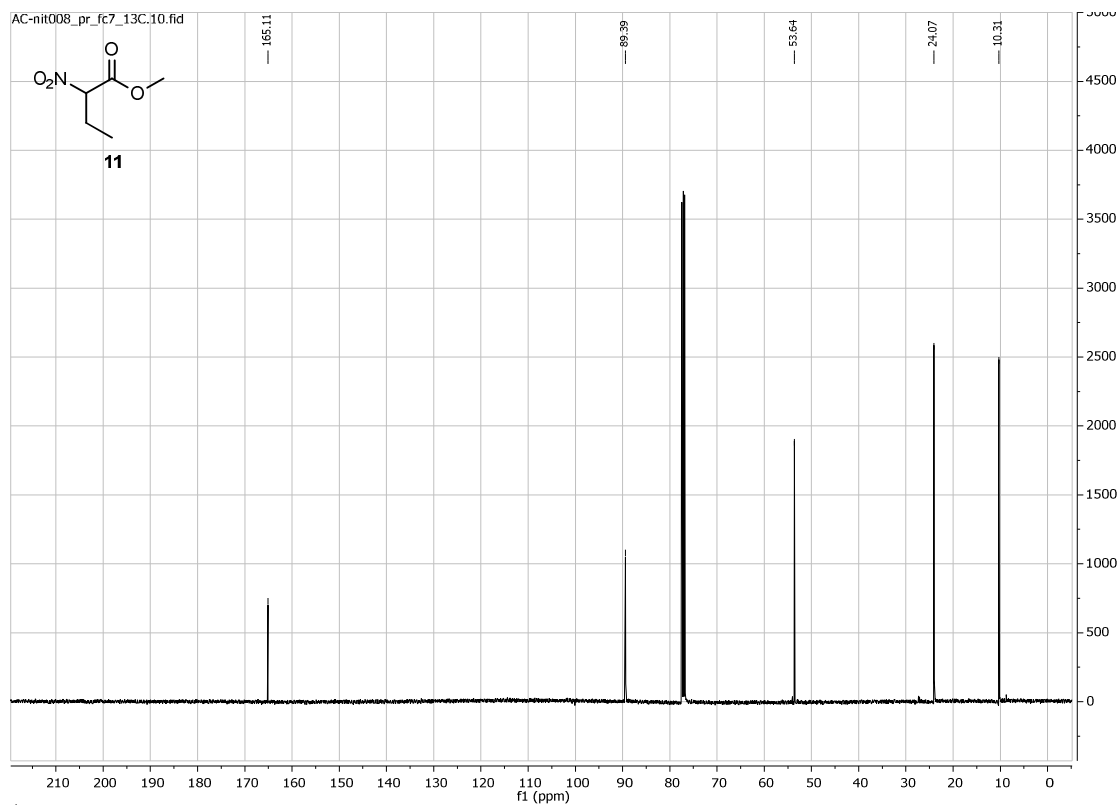
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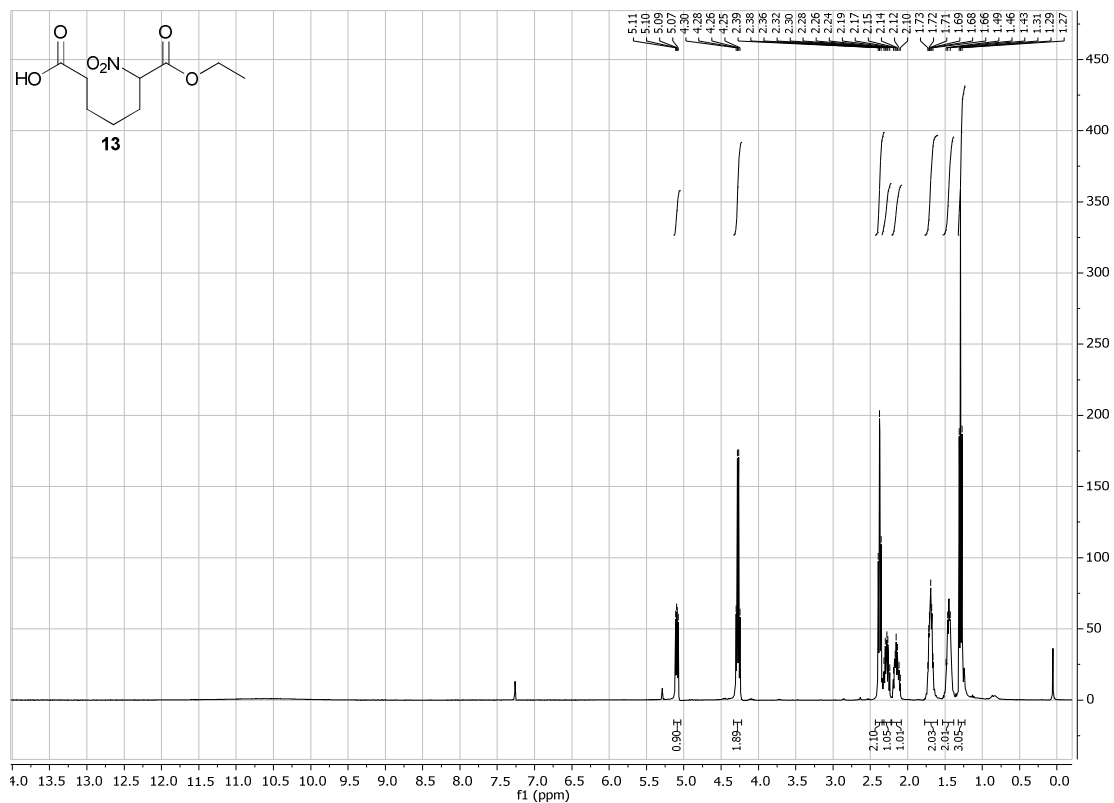
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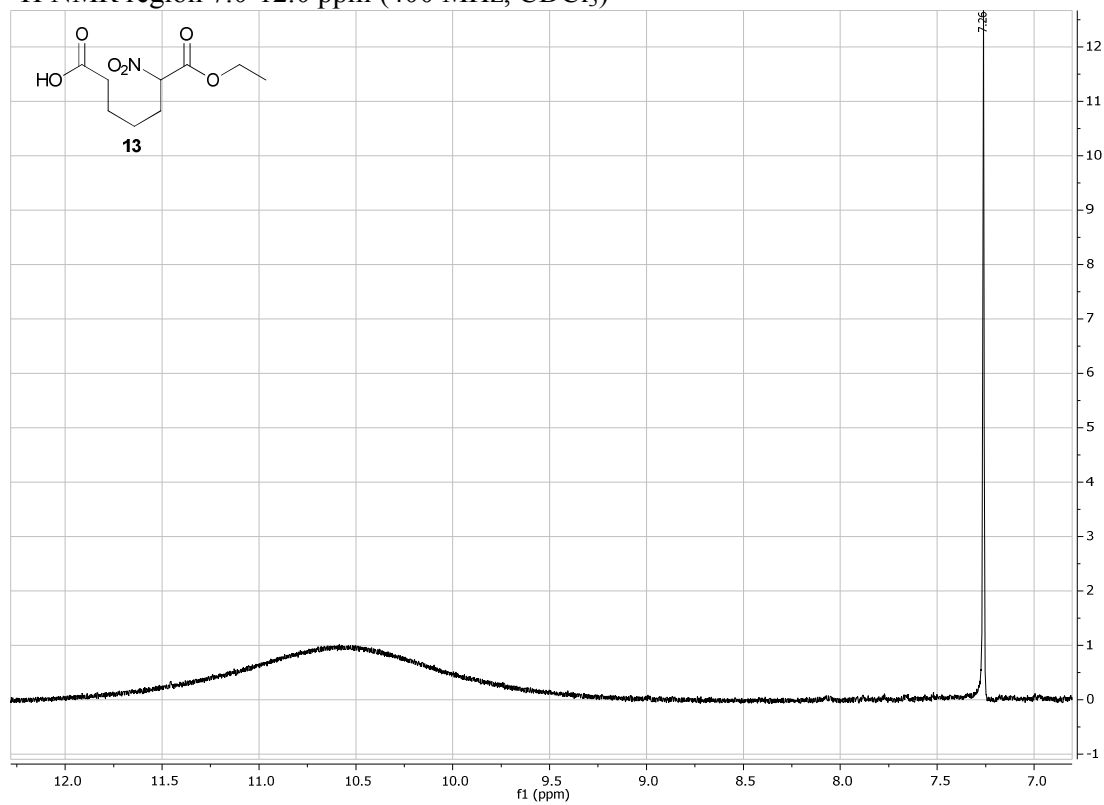
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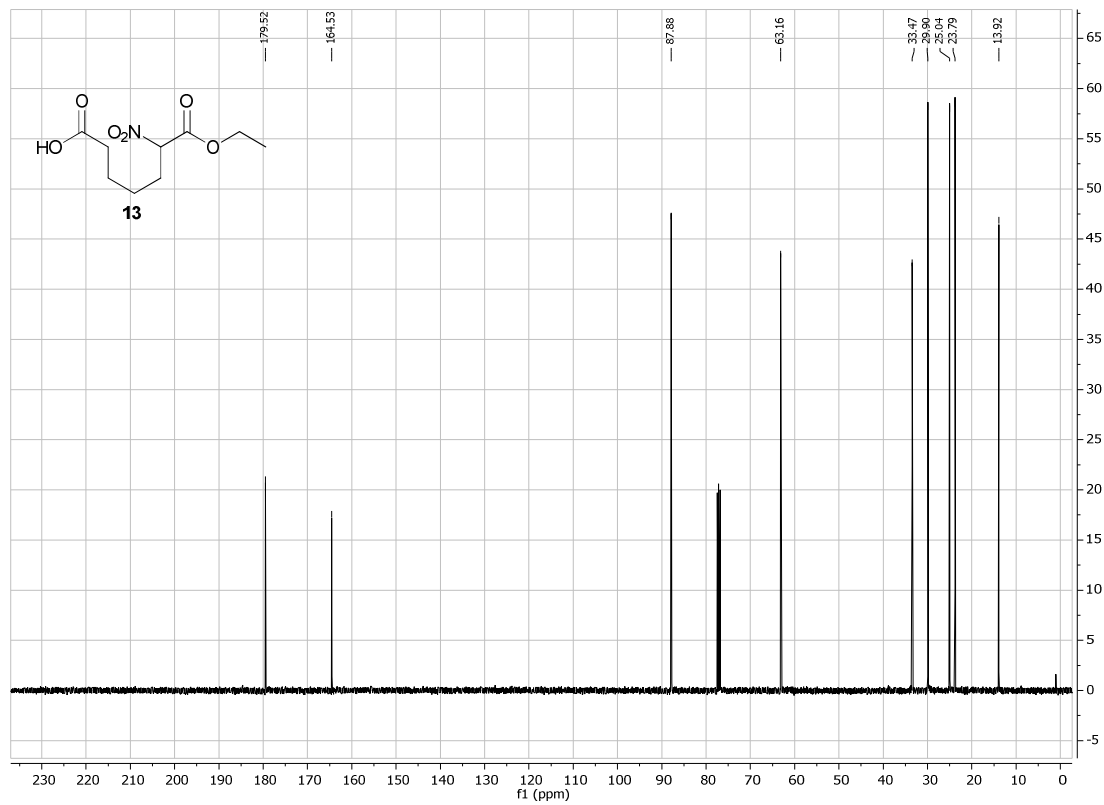
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



$^1\text{H}$  NMR region 7.0-12.0 ppm (400 MHz,  $\text{CDCl}_3$ )

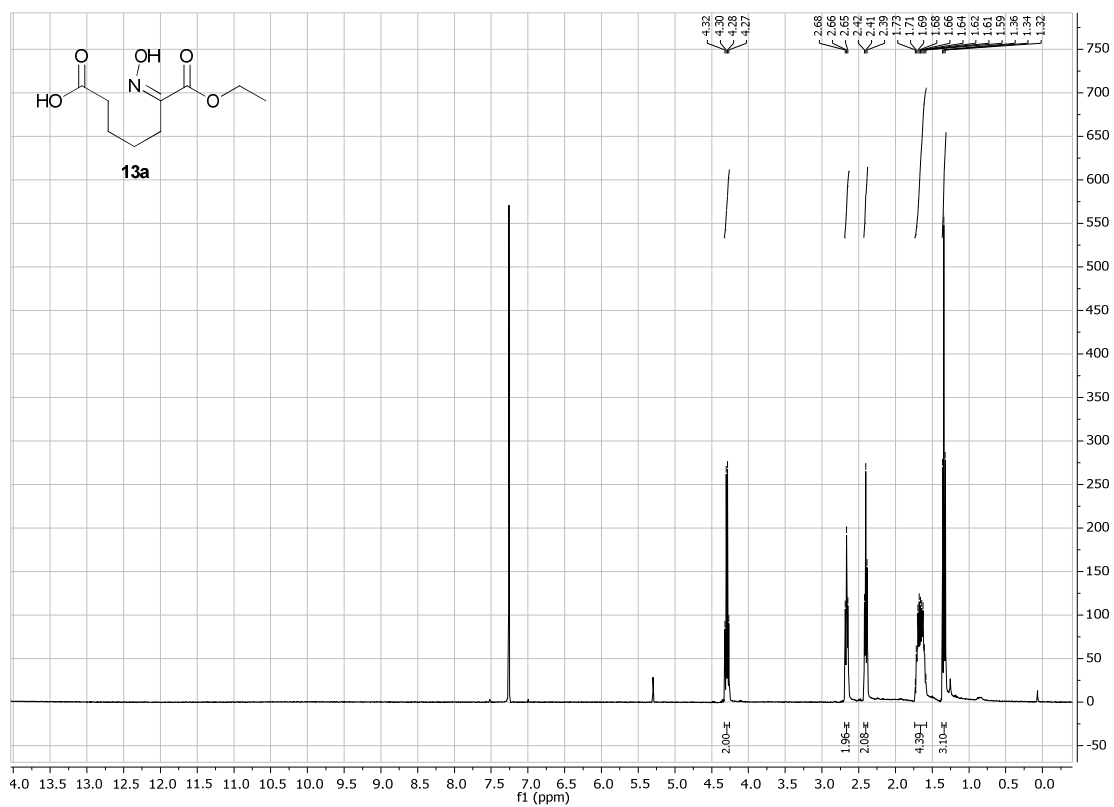


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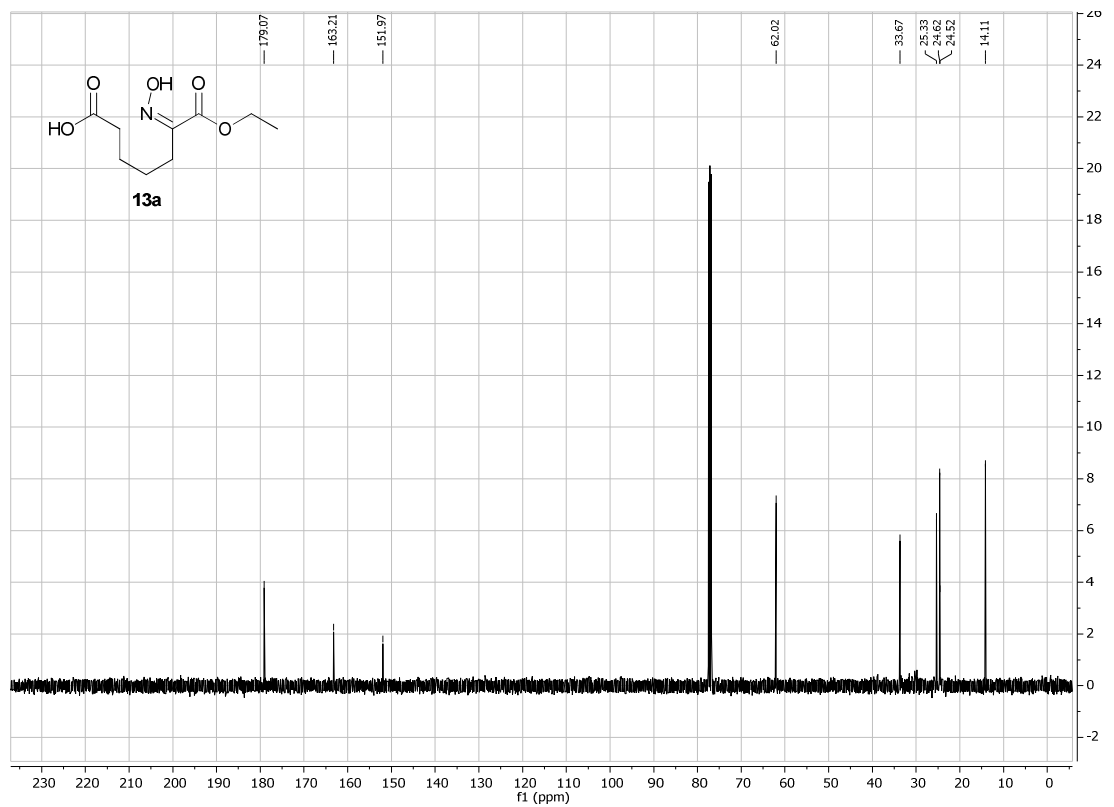




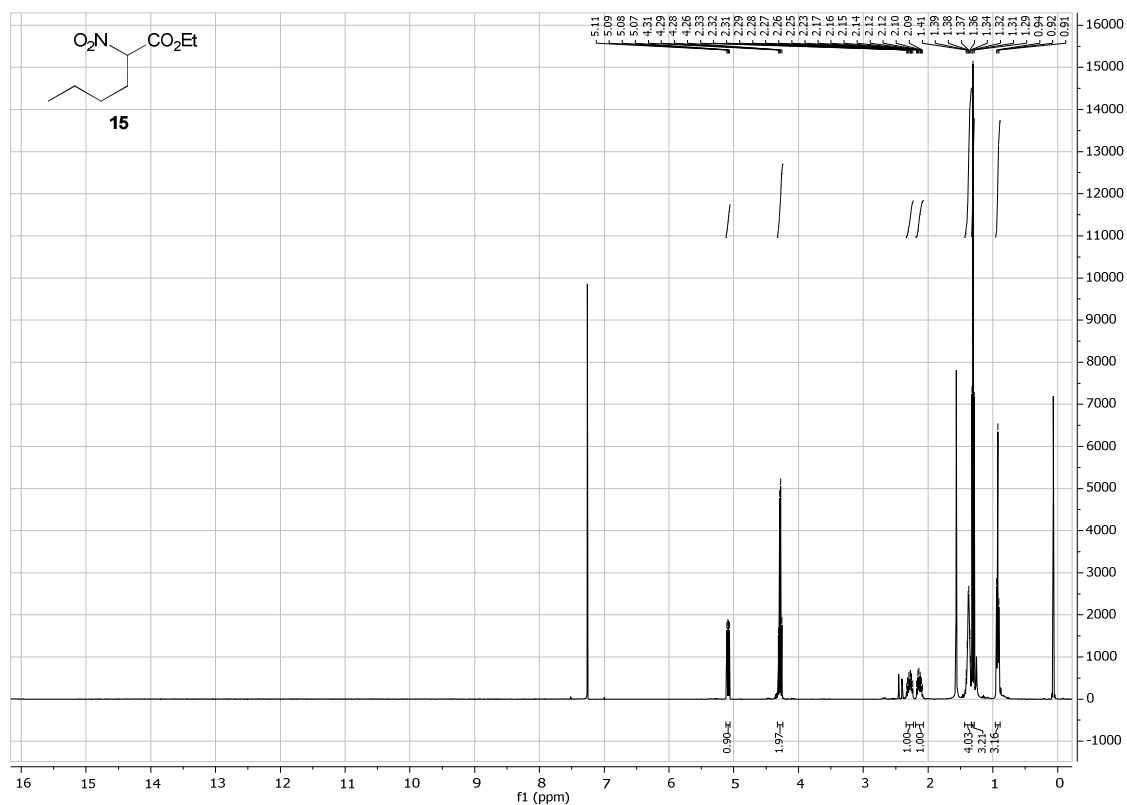
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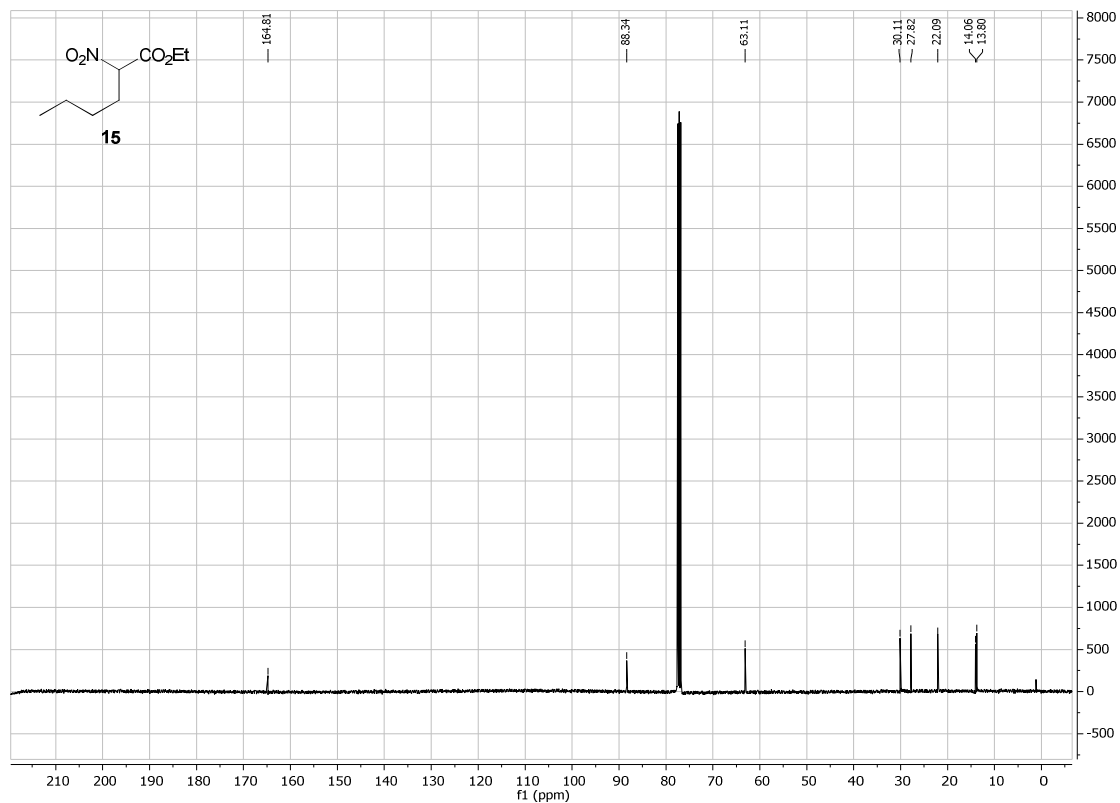
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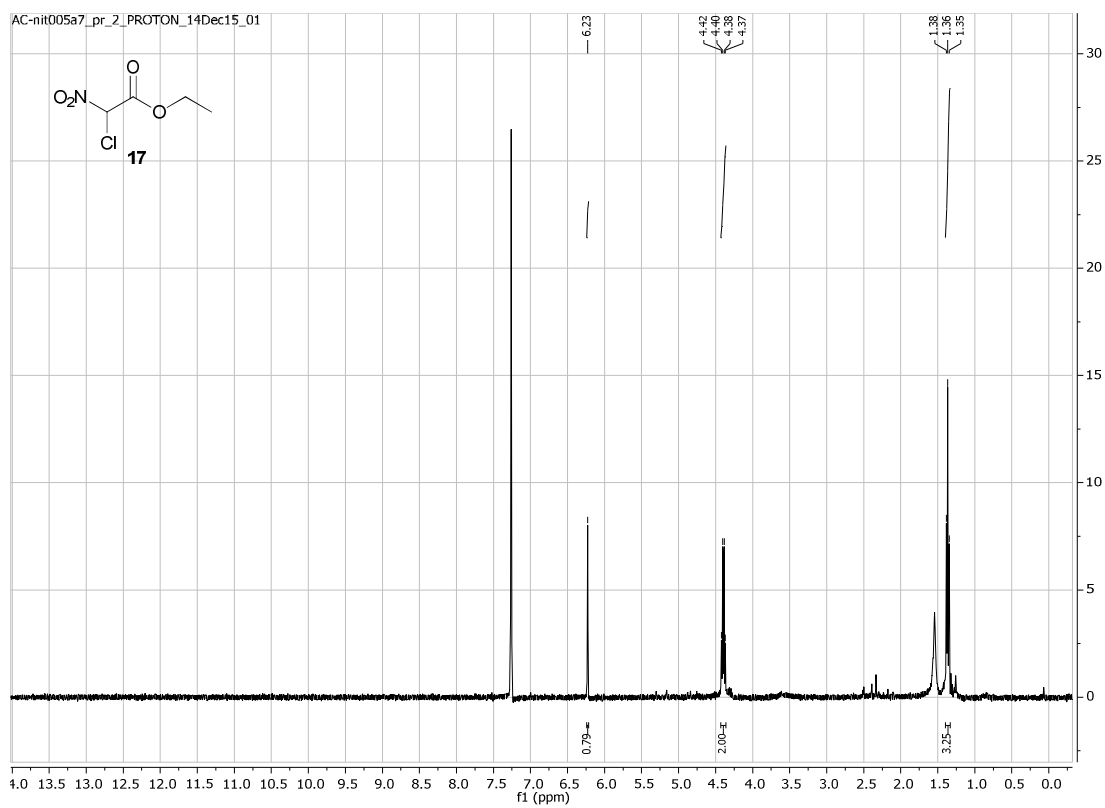
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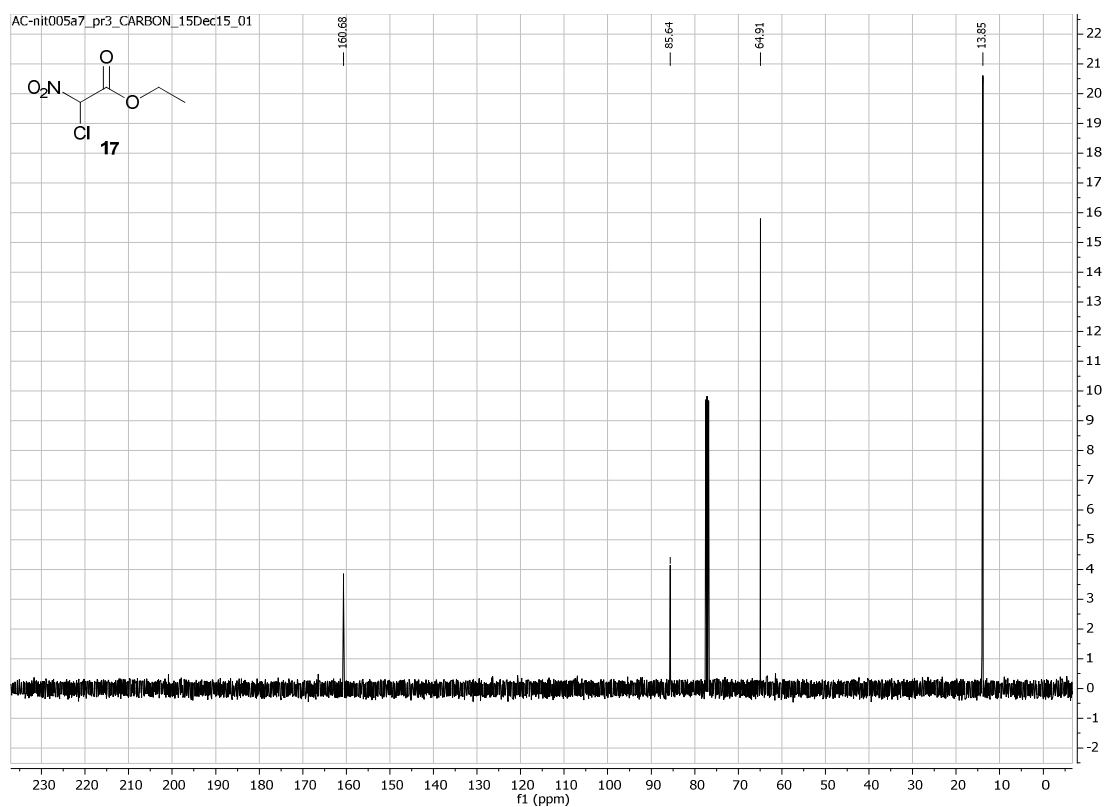
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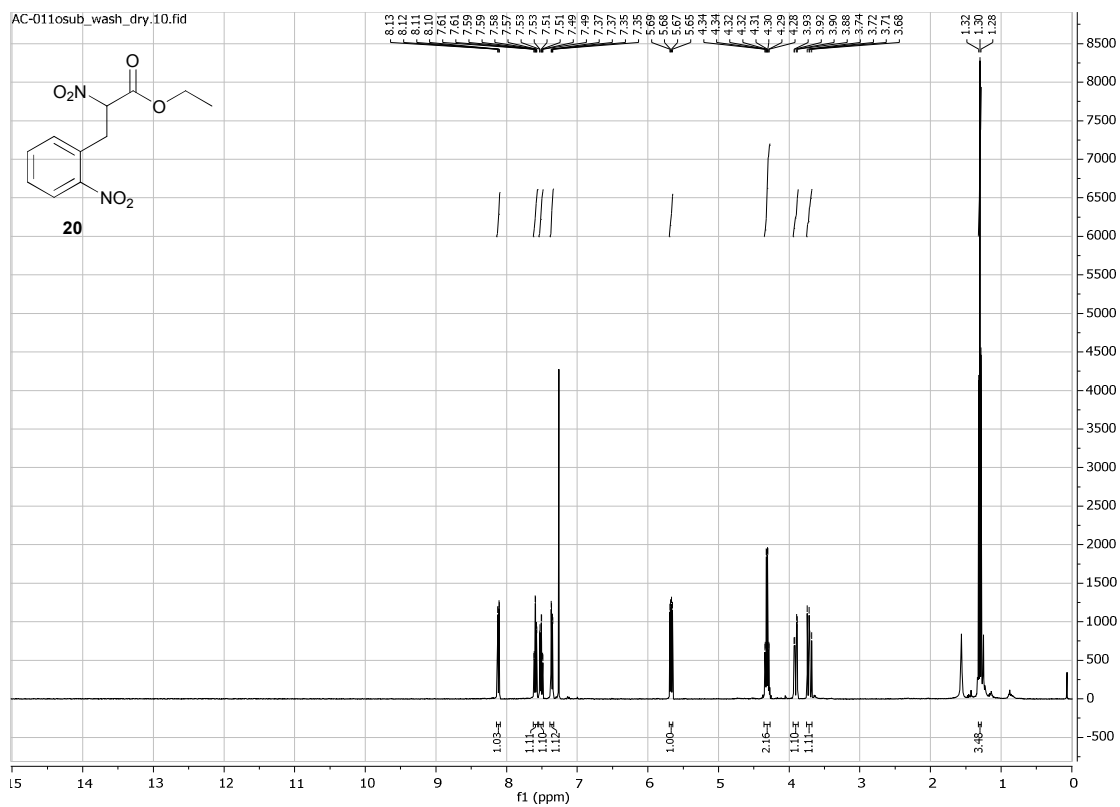
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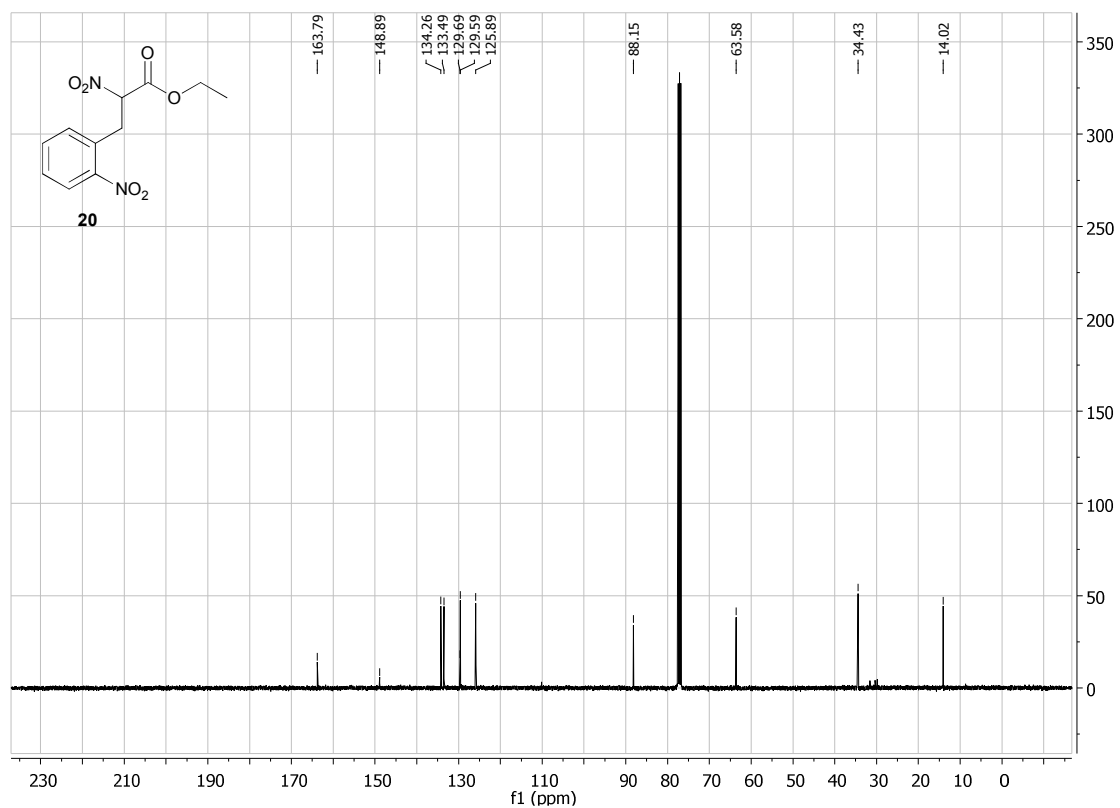
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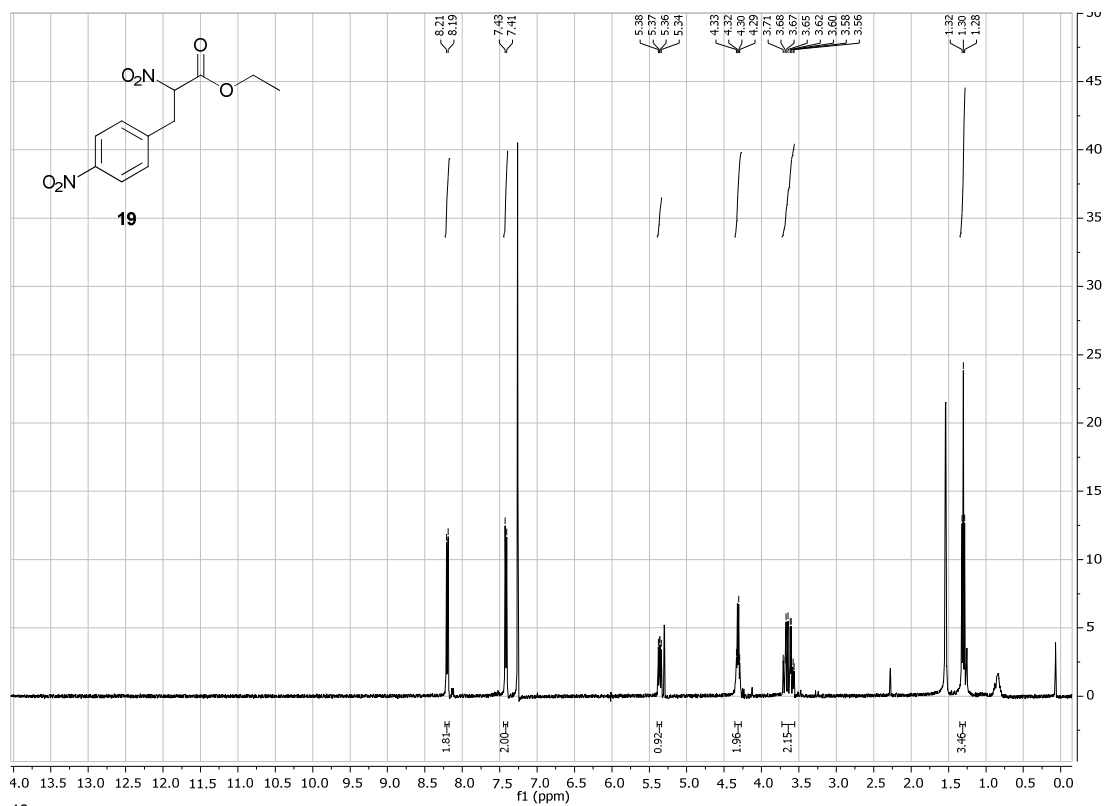
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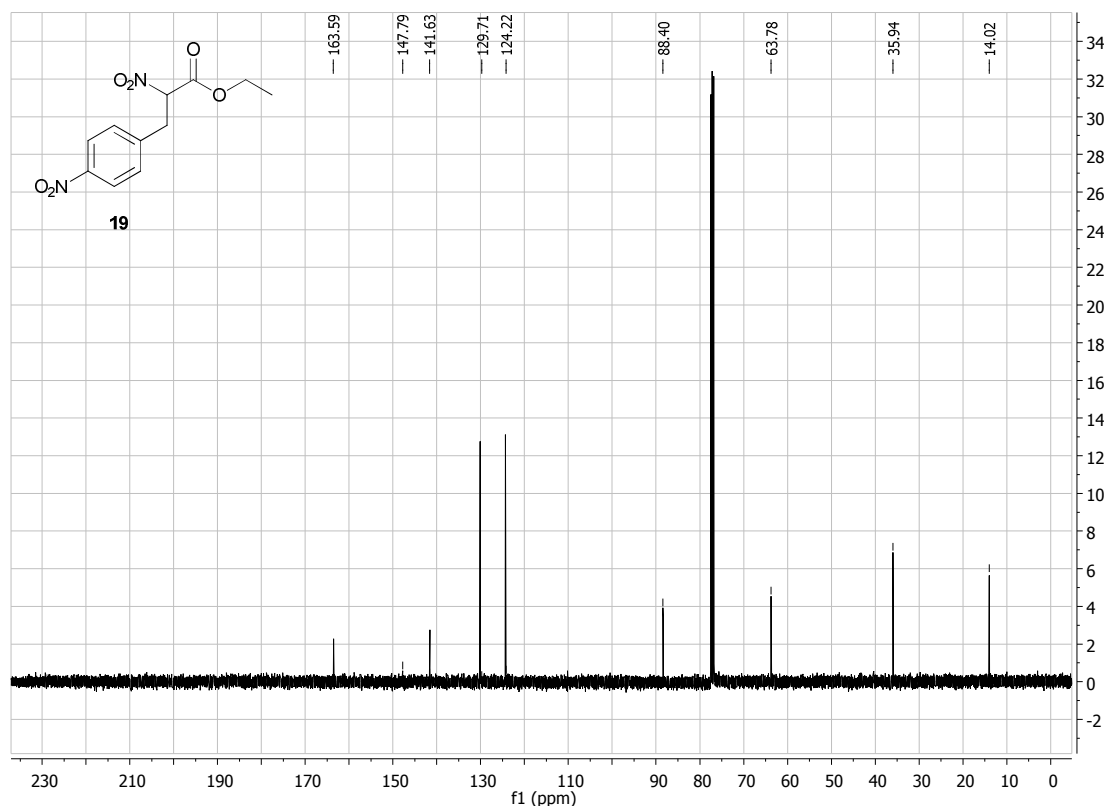
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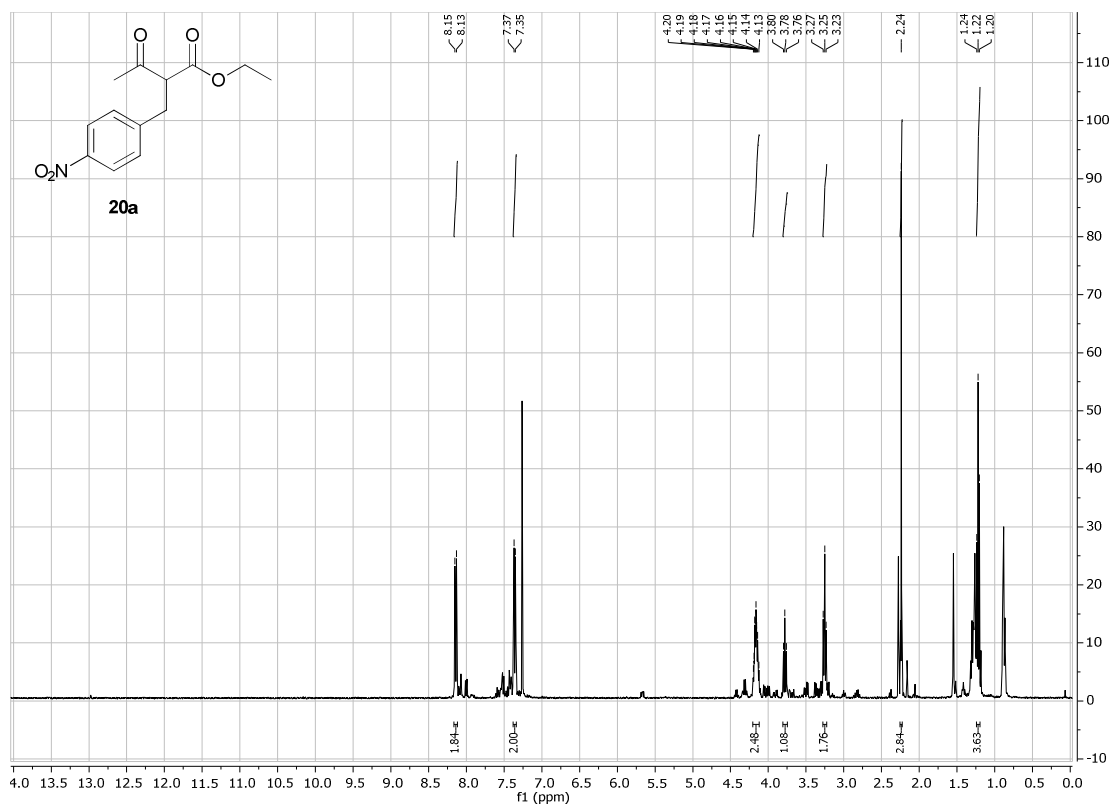
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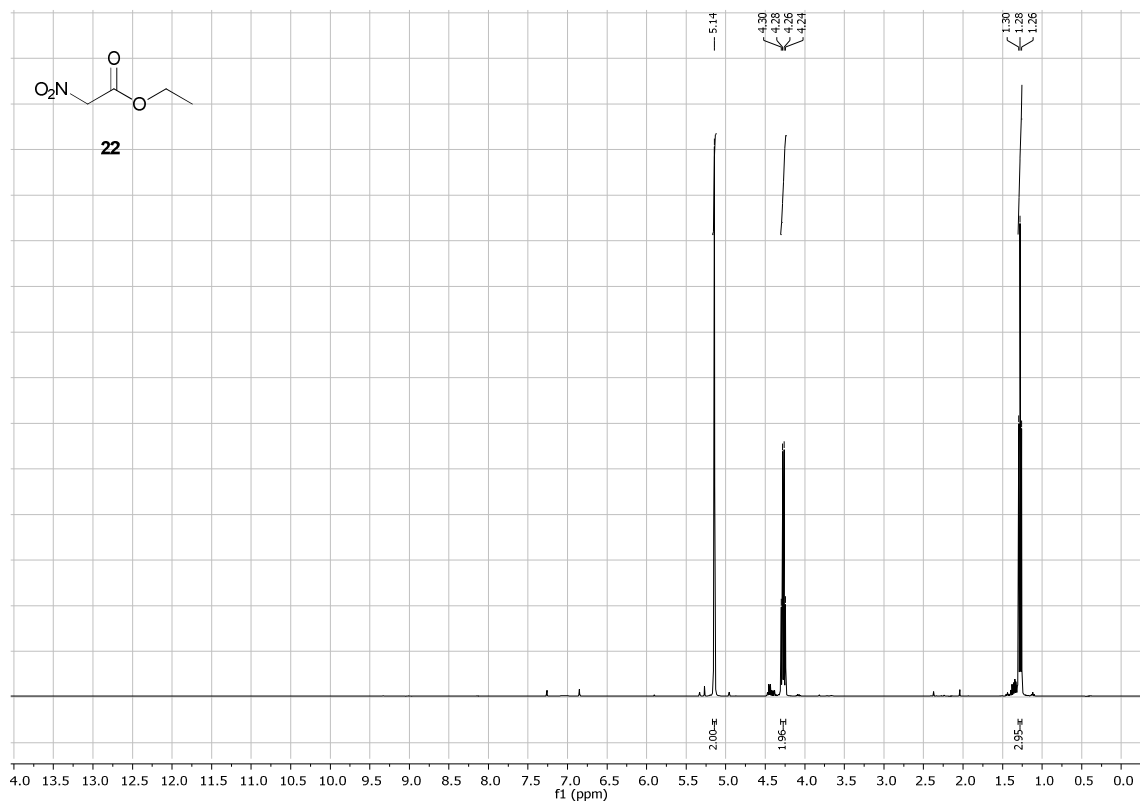
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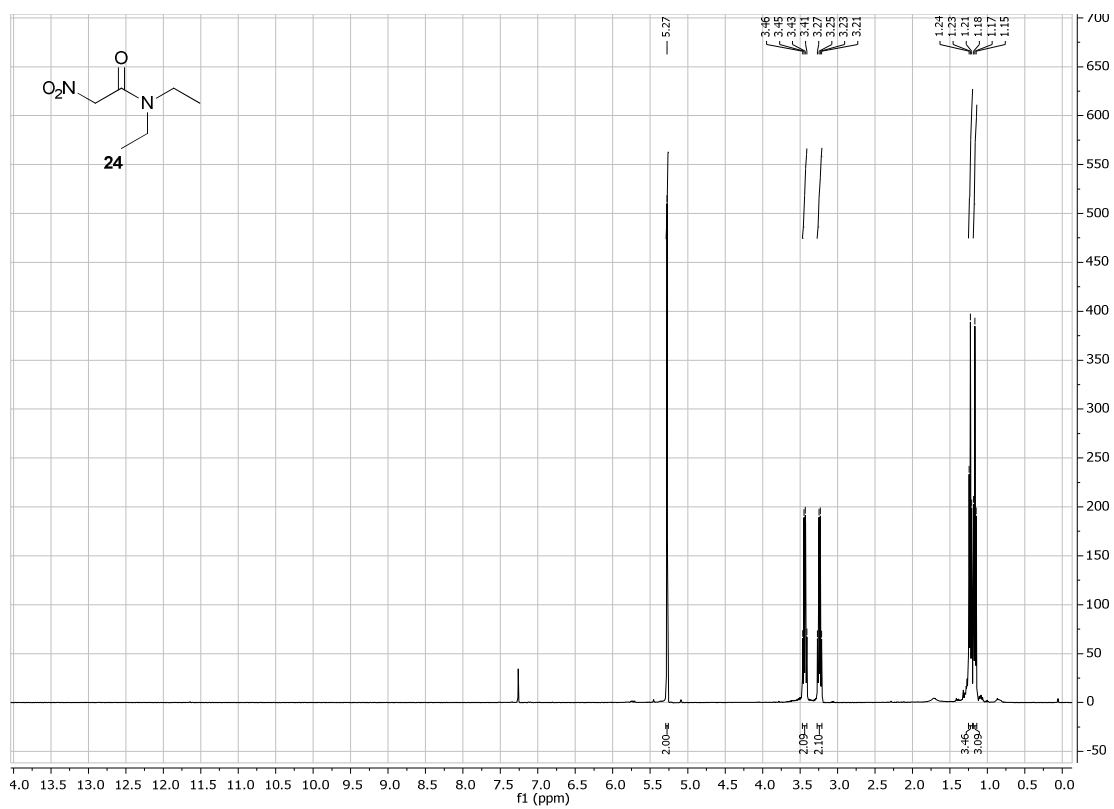
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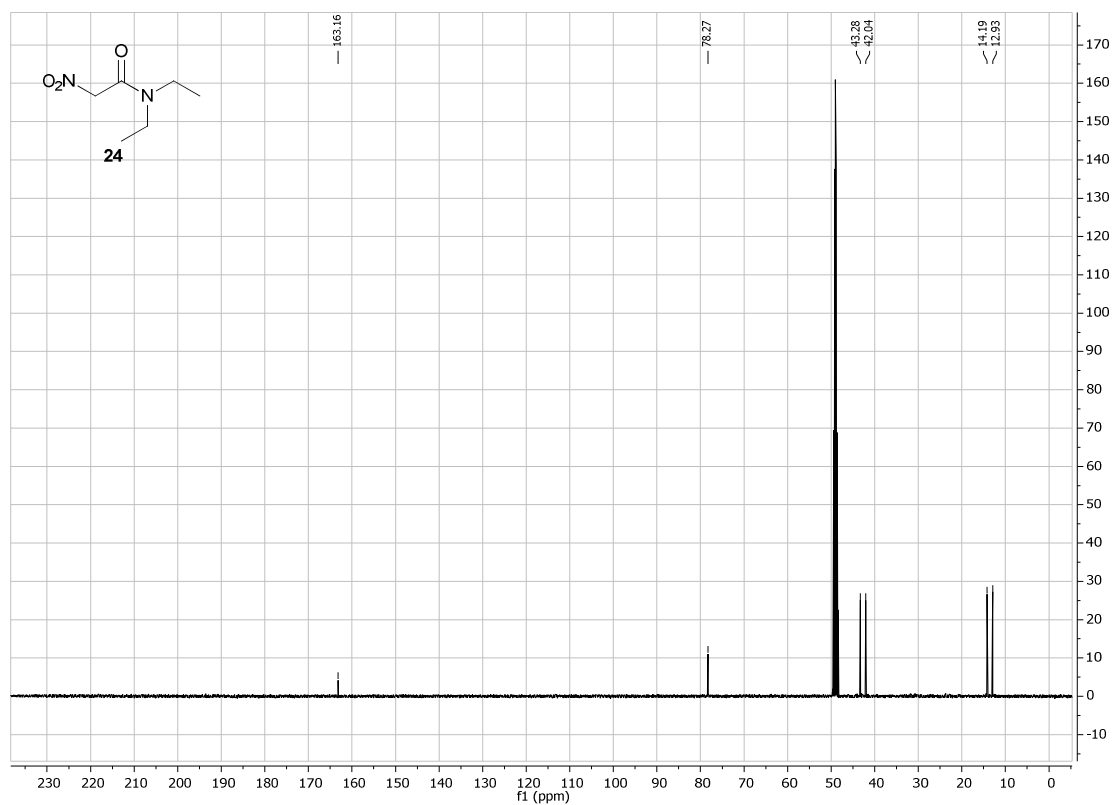
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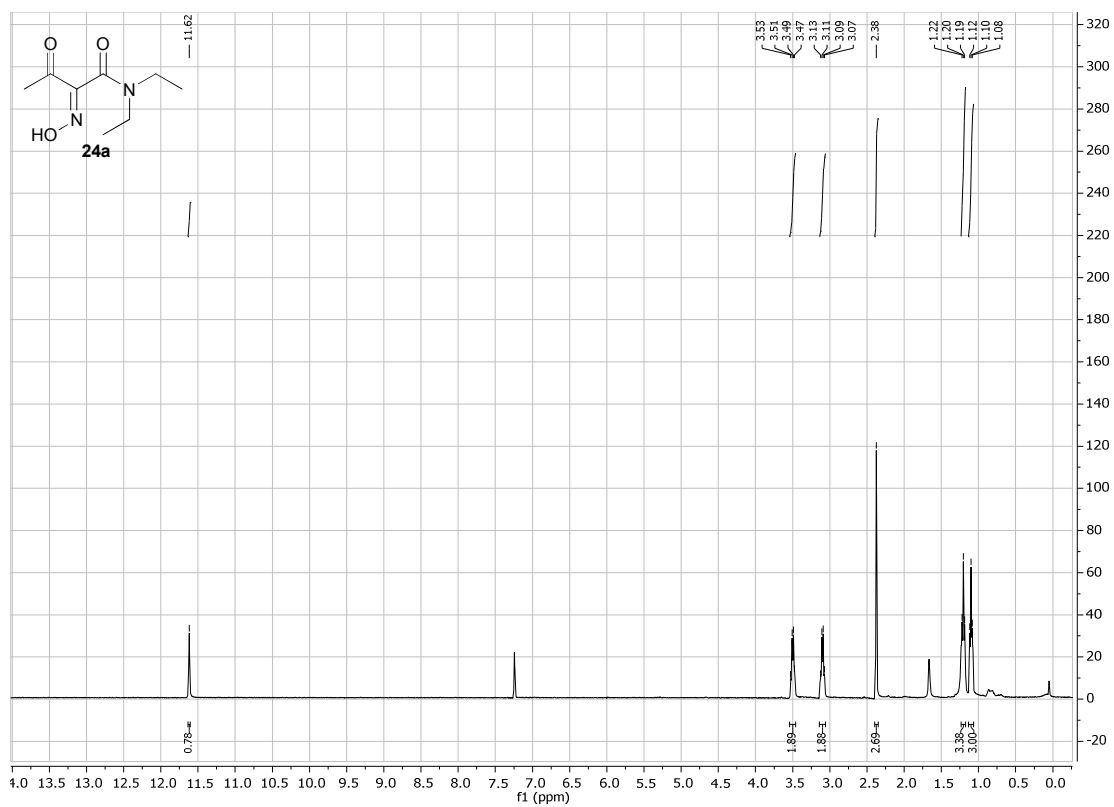
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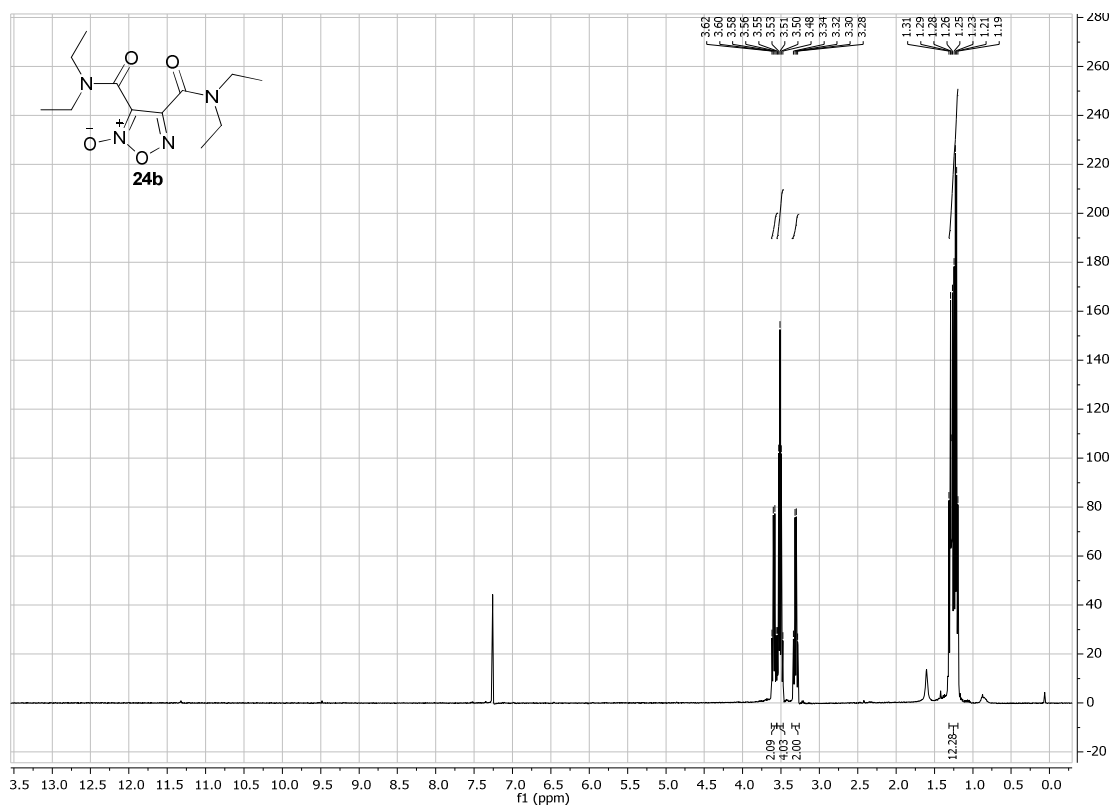
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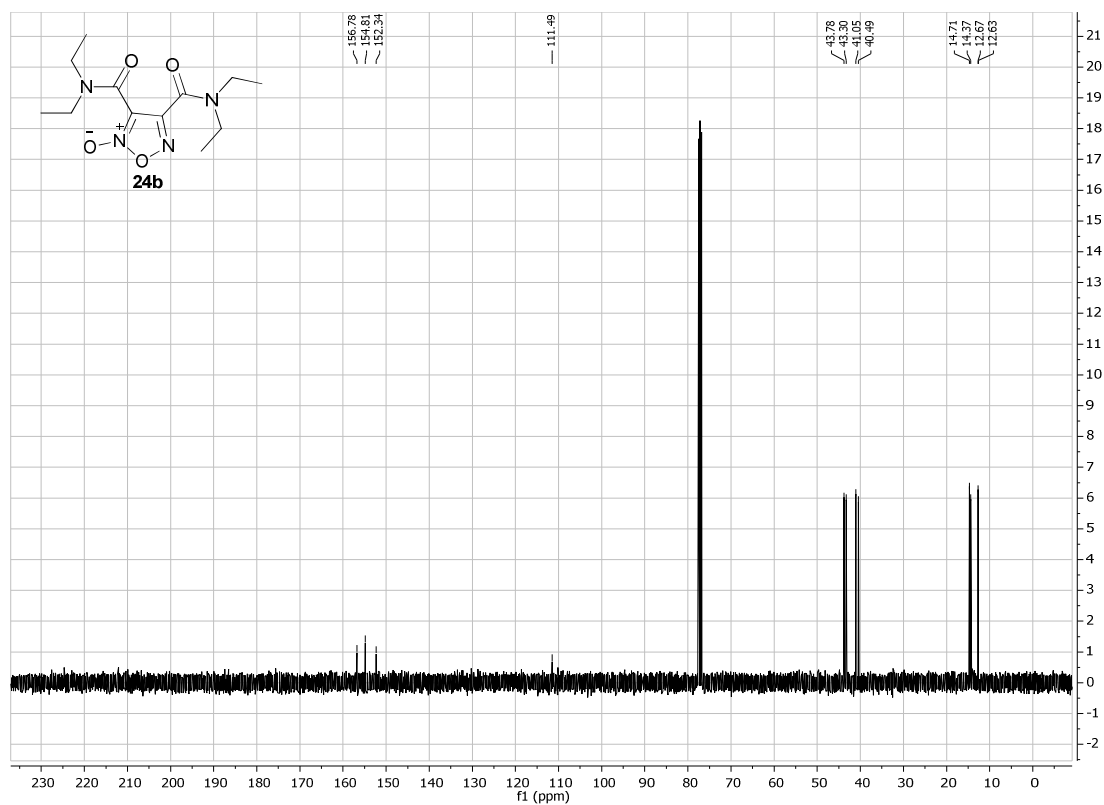


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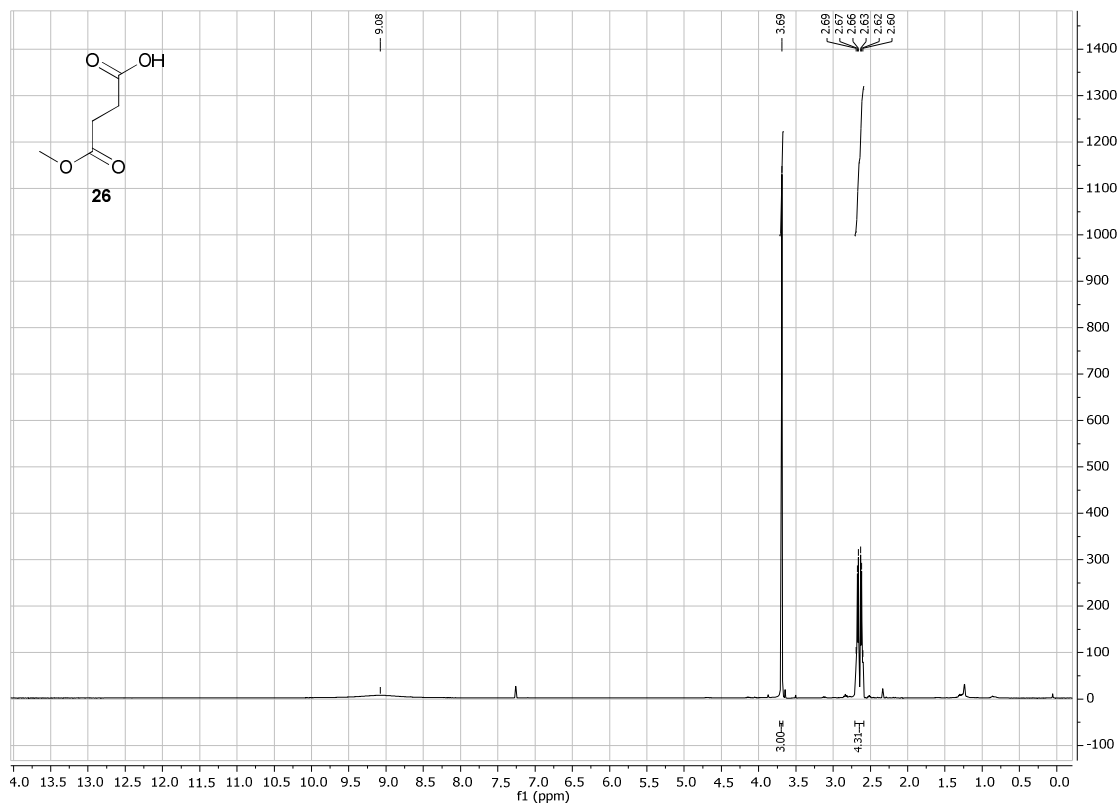




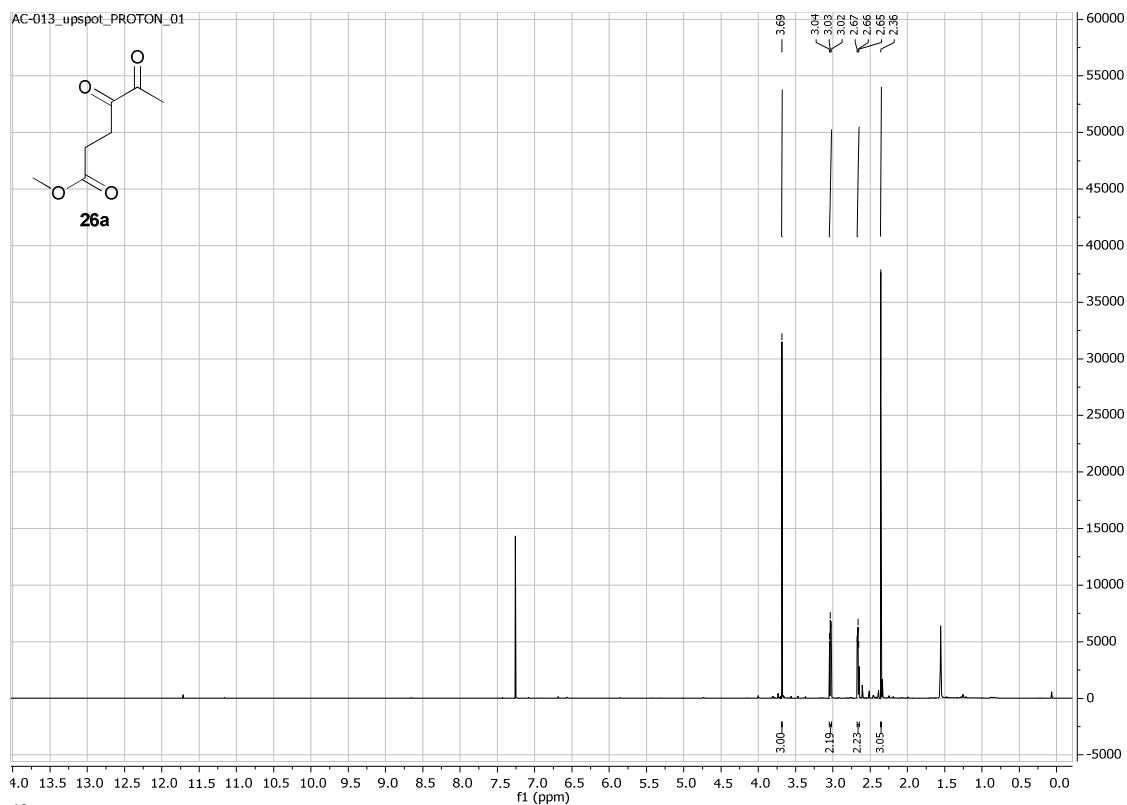
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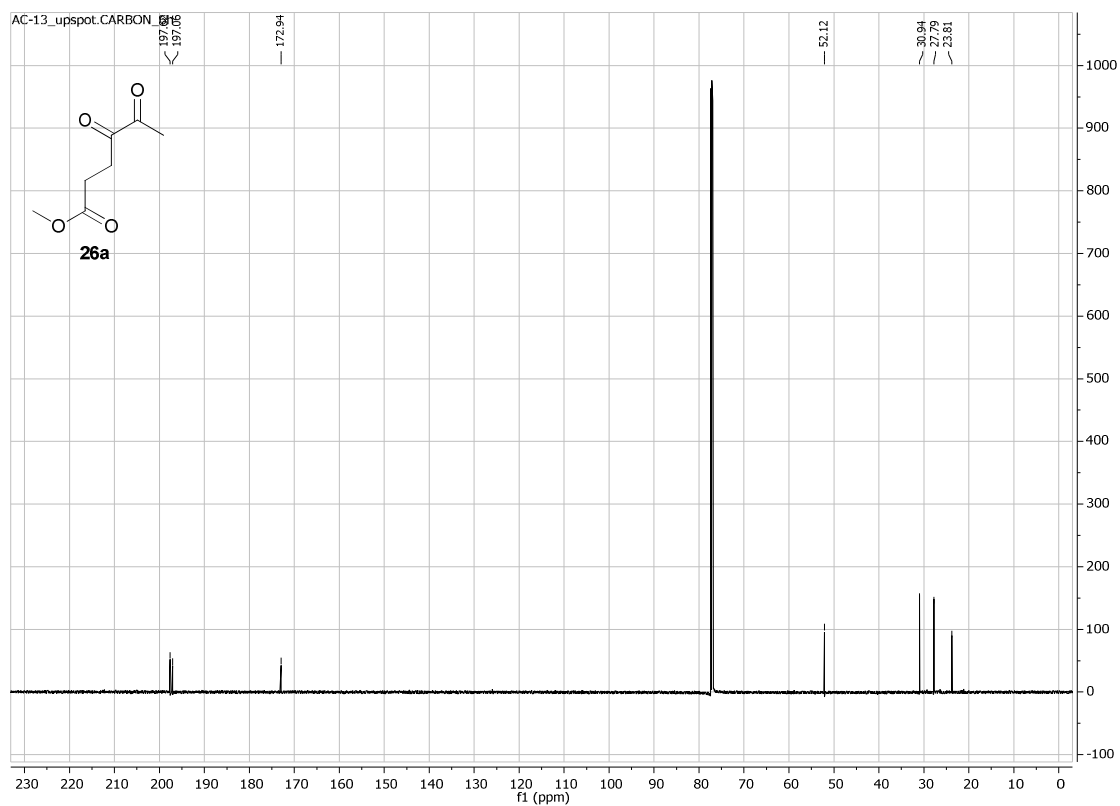
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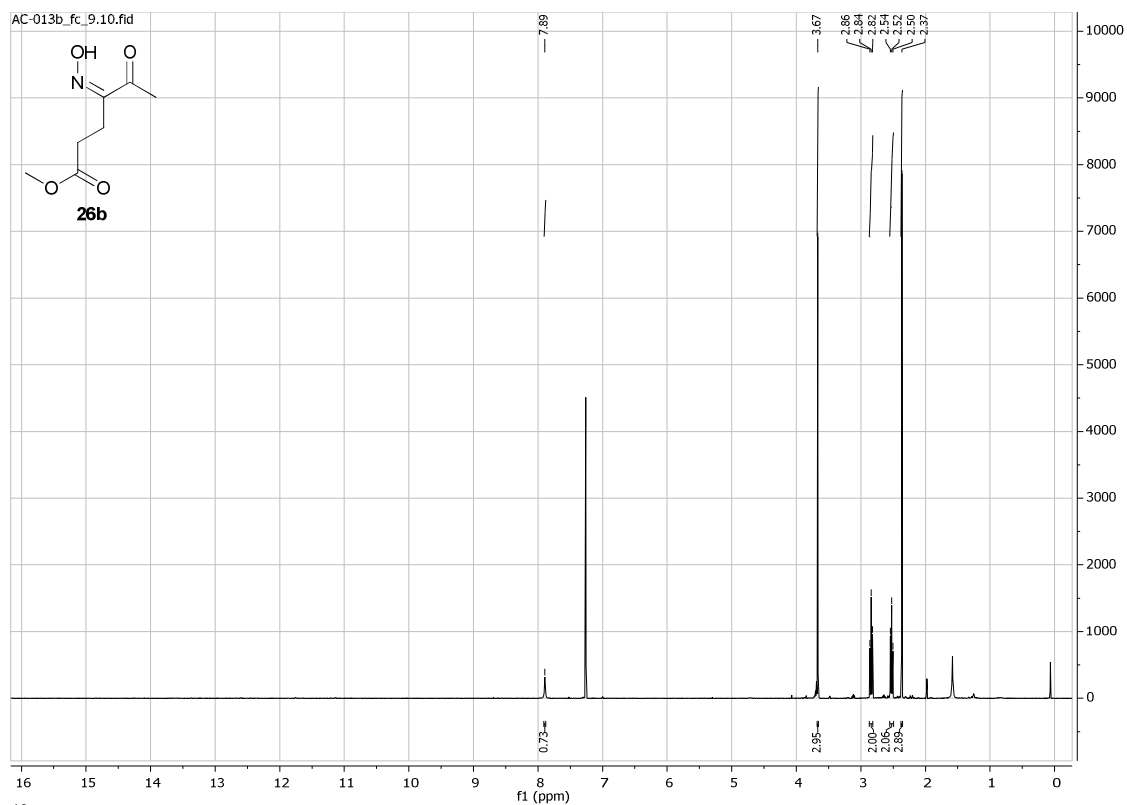
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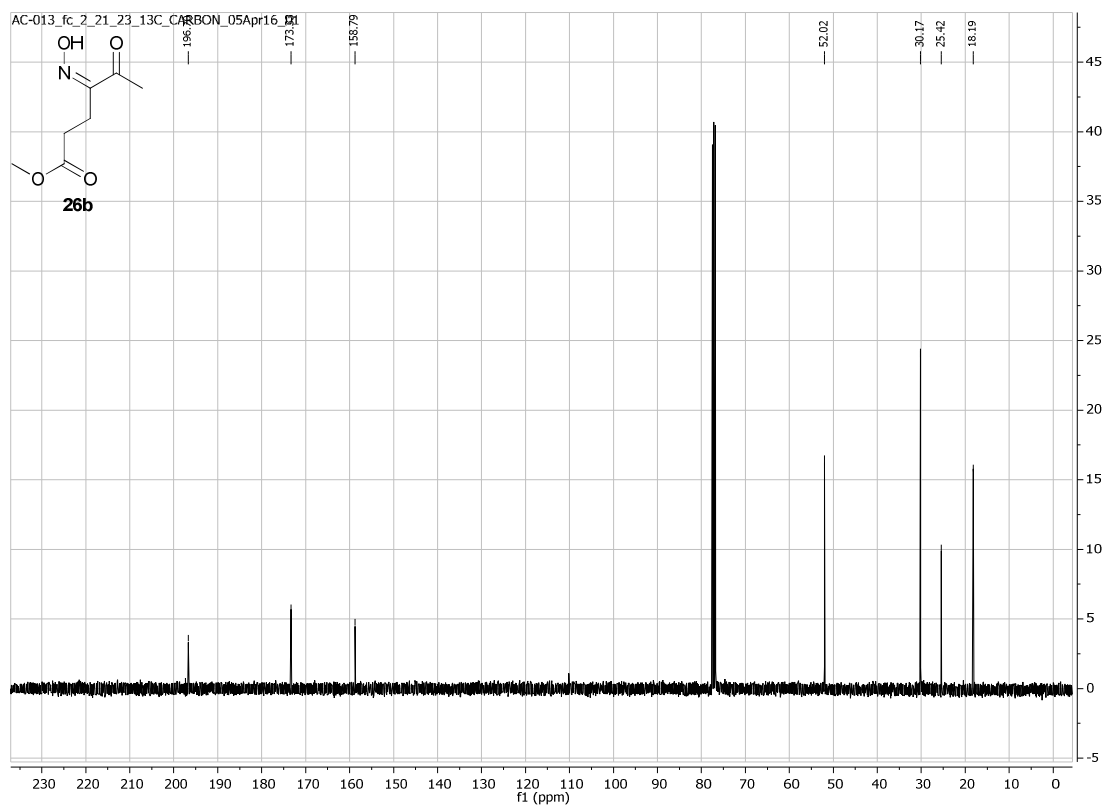
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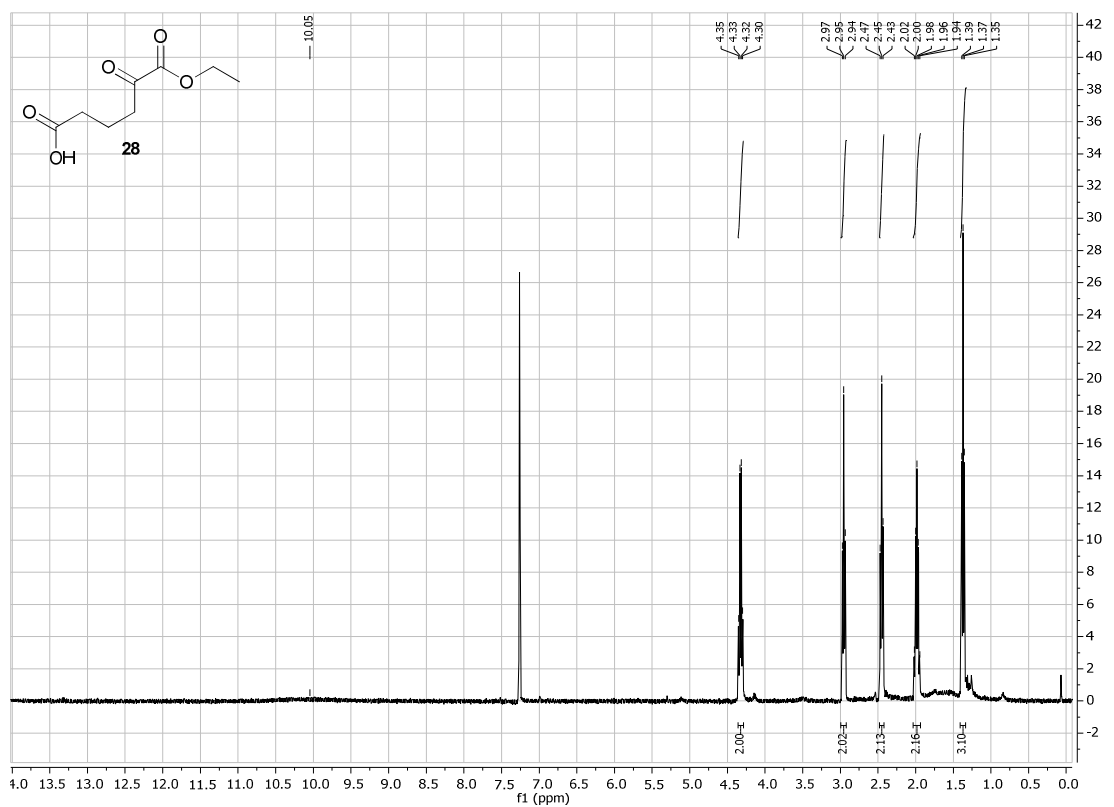
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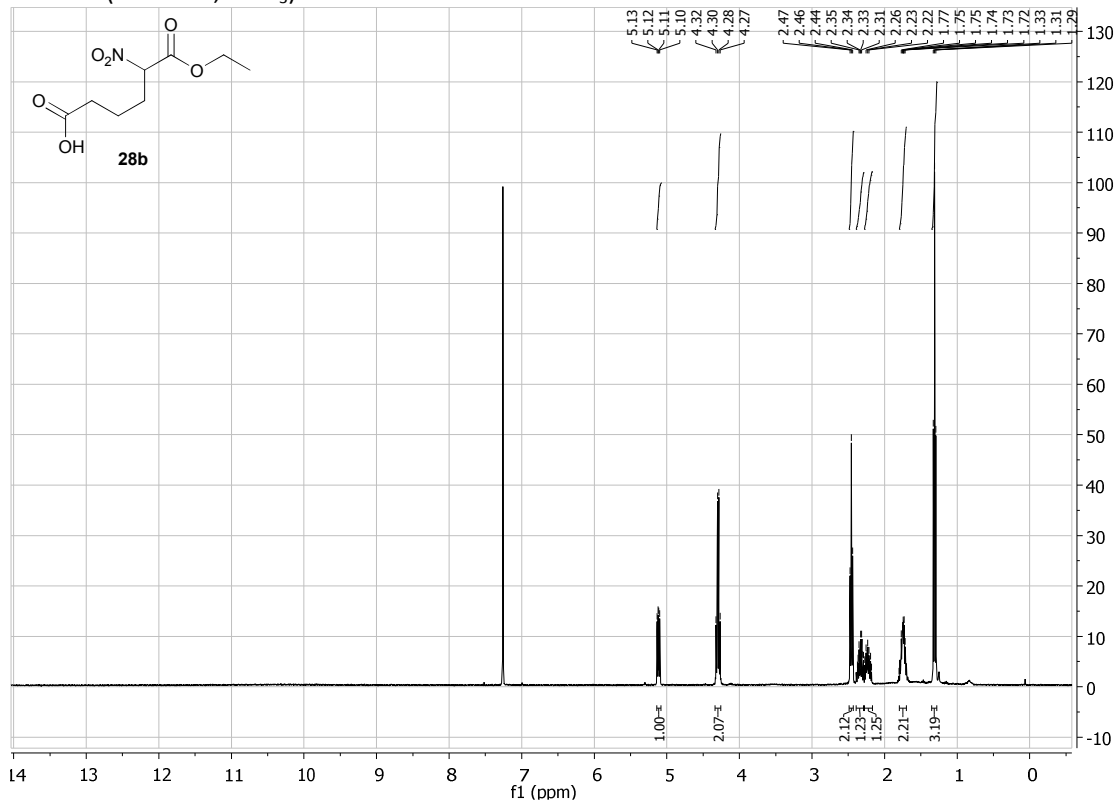
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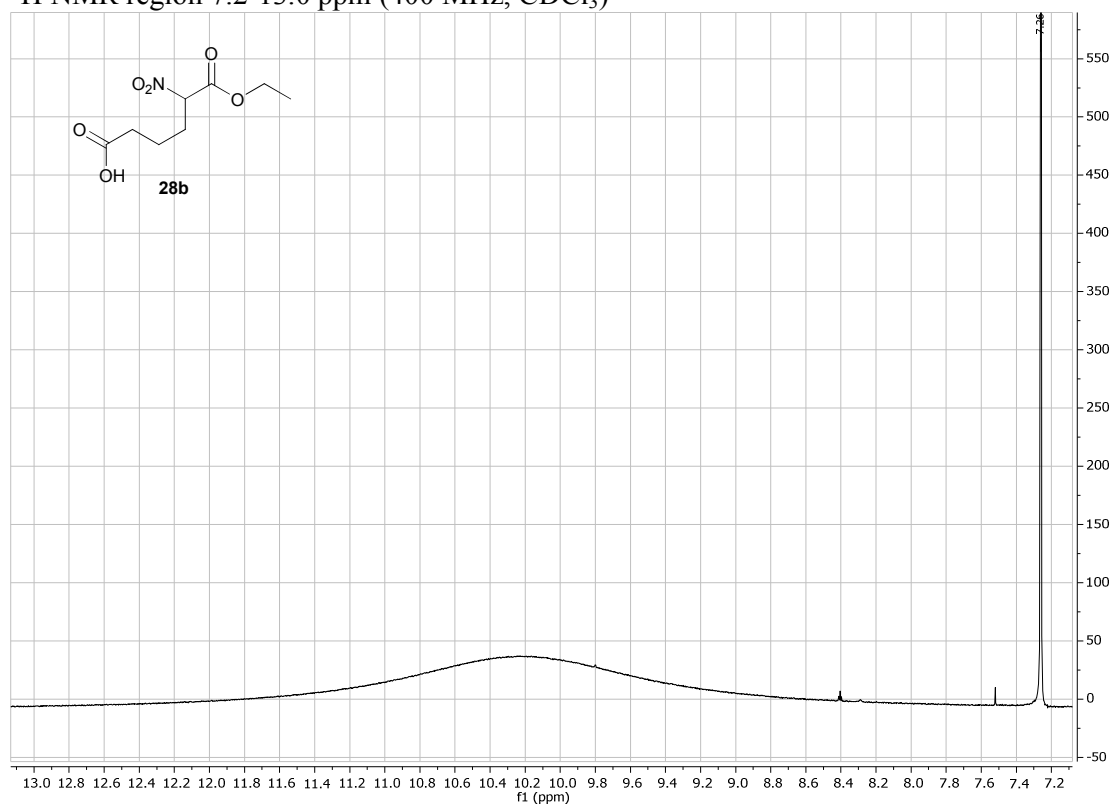
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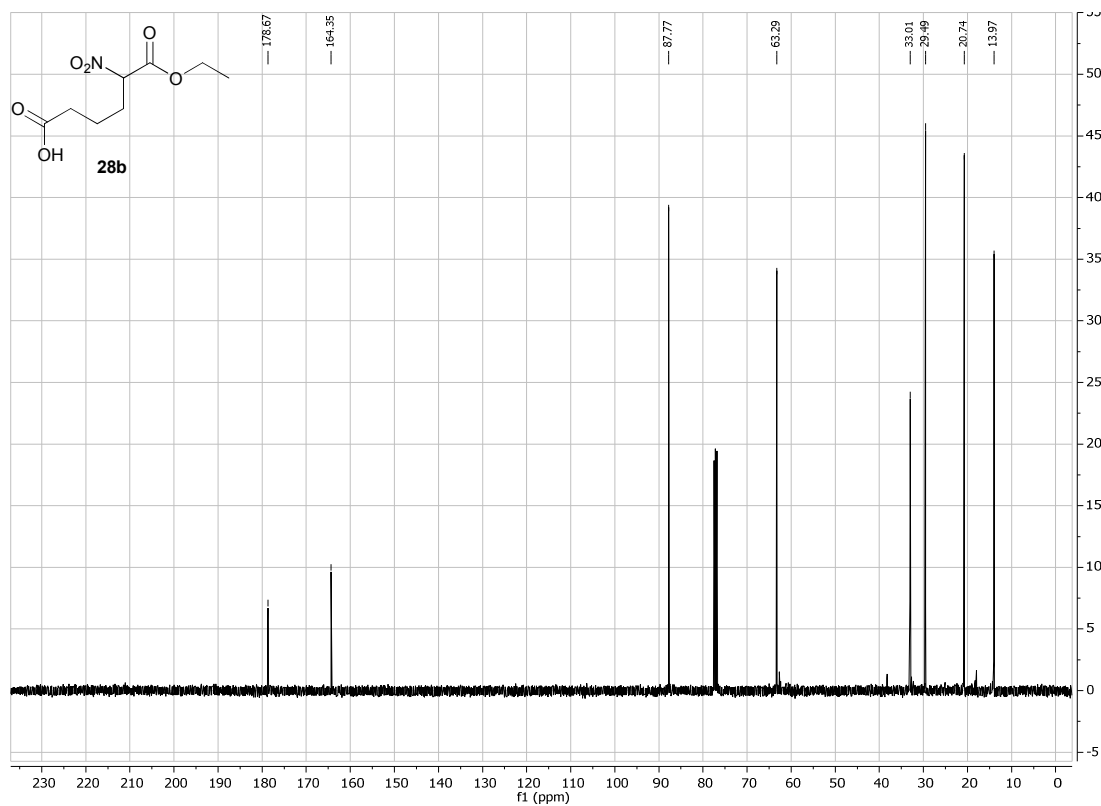
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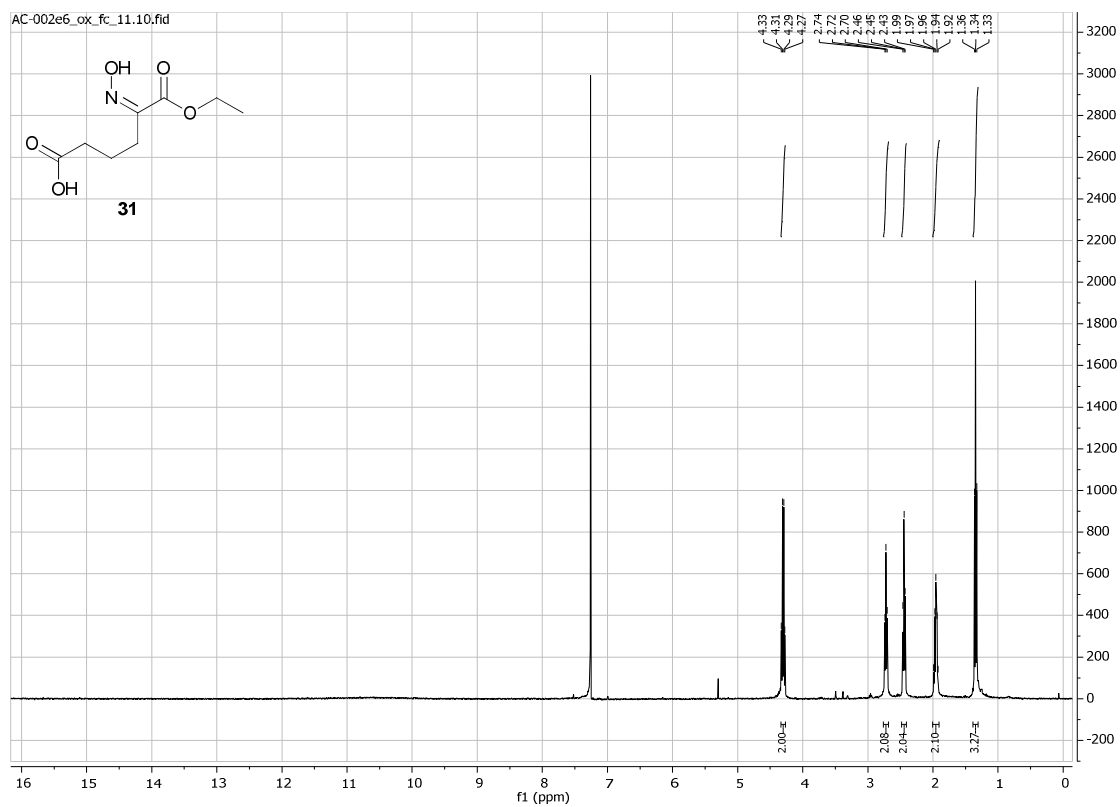
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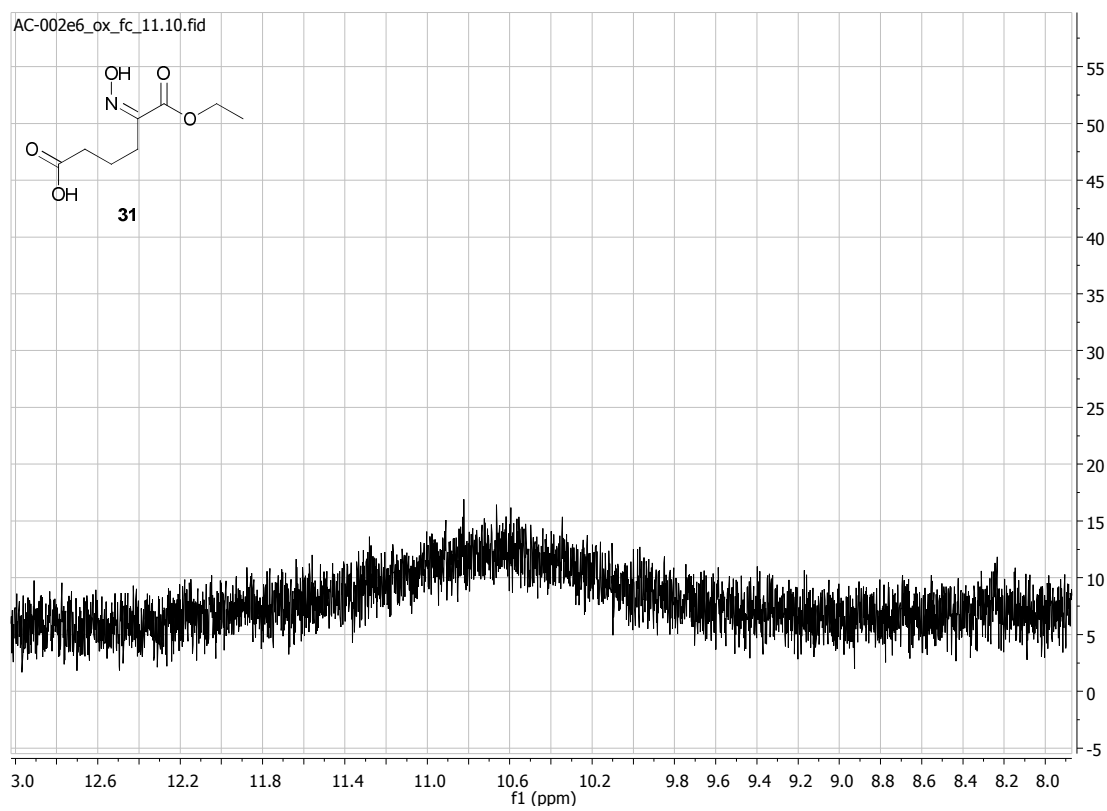
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$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



$^1\text{H}$  NMR region 8-13.0 ppm (400 MHz,  $\text{CDCl}_3$ )



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

