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<b>Author(s)</b>	O'Mahony, Rosella M.; Lynch, Denis; Hayes, Hannah L. D.; Ní Thuama, Eilís; Donnellan, Philip; Jones, Roderick C.; Glennon, Brian; Collins, Stuart G.; Maguire, Anita R.
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**SUPPORTING INFORMATION**

**Title:** Exploiting the Continuous in situ Generation of Mesityl Azide for Use in a Telescoped Process

**Author(s):** Rosella M. O'Mahony, Denis Lynch, Hannah L. D. Hayes, Eilís Ní Thuama, Philip Donnellan, Roderick C. Jones, Brian Glennon, Stuart G. Collins,\* Anita R. Maguire\*

## Contents

1. Experimental Continuous-flow Setup.....	3
2. Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra for Compounds <b>4g</b> , <b>7a-g</b> and <b>8</b> .....	5

## 1. Experimental Continuous-flow Setup

All continuous processes were performed using either a Vapourtec R-Series flow reactor or a Vapourtec E-Series flow reactor.

The R-Series flow reactor consists of four piston pumps and up to four temperature controlled tubular reactors. To prepare the reactor for operation pumps were purged with the solvent to be used in the reaction prior to use. All reaction tubing, coils, inlets and connections were also purged thoroughly in a similar manner.

**Table S1:** General specifications for R-Series continuous-flow reactor

General Specifications	
Material of tubing	PFA
Diameter of tubing	1 mm
Working flow rates	0.05 mL/min – 9.99 mL/min
Tubular reactor working volume	10 mL
Temperature range	-70 °C to 250 °C

The Vapourtec E-Series flow reactor was used for the final telescoped reactions alongside the R-series. The E-Series flow reactor consists of three peristaltic pumps and up to two temperature controlled tubular reactors. To prepare the reactor for operation pumps were, again, purged with the solvent to be used in the reaction prior to use. All reaction tubing, coils, inlets and connections were also purged thoroughly in a similar manner.

**Table S2:** General specifications for E-Series continuous-flow reactor

General Specifications	
Material of tubing	PFA
Diameter of tubing	1 mm
Working flow rates	0.02 mL/min – 10.0 mL/min
Tubular reactor working volume	10 mL
Temperature range	-70 °C to 250 °C

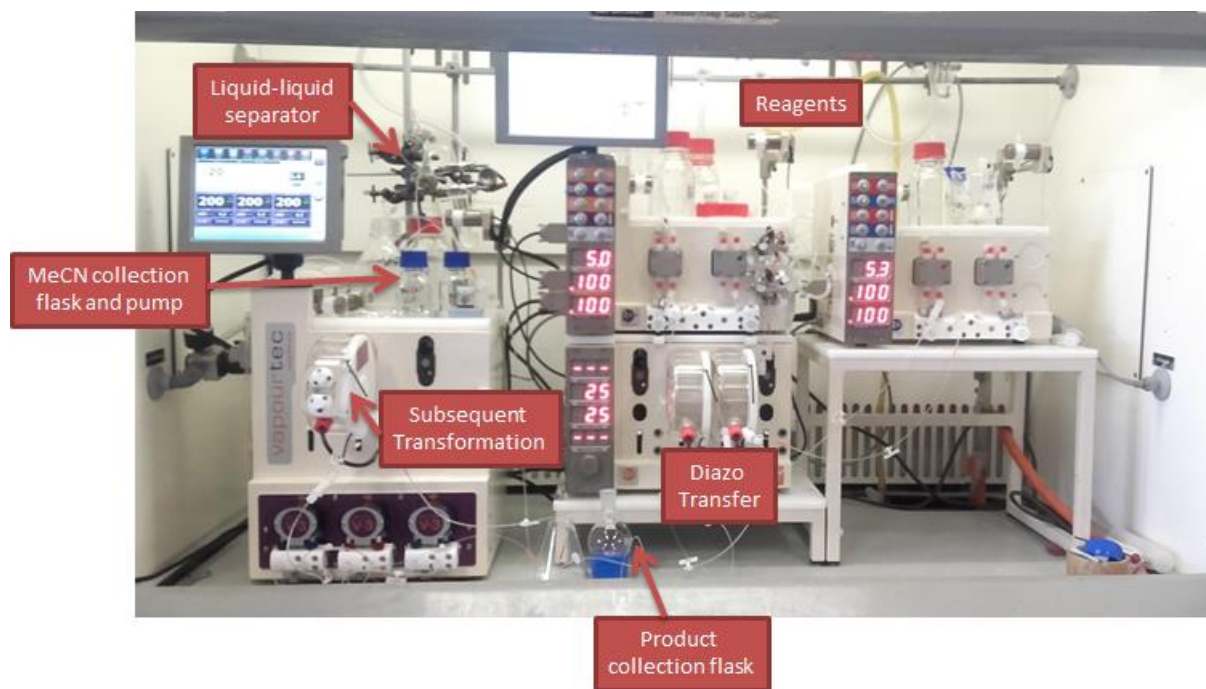


Figure S1: Experimental Continuous Flow Setup.

## 2. Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra for Compounds 4g and 7a–g

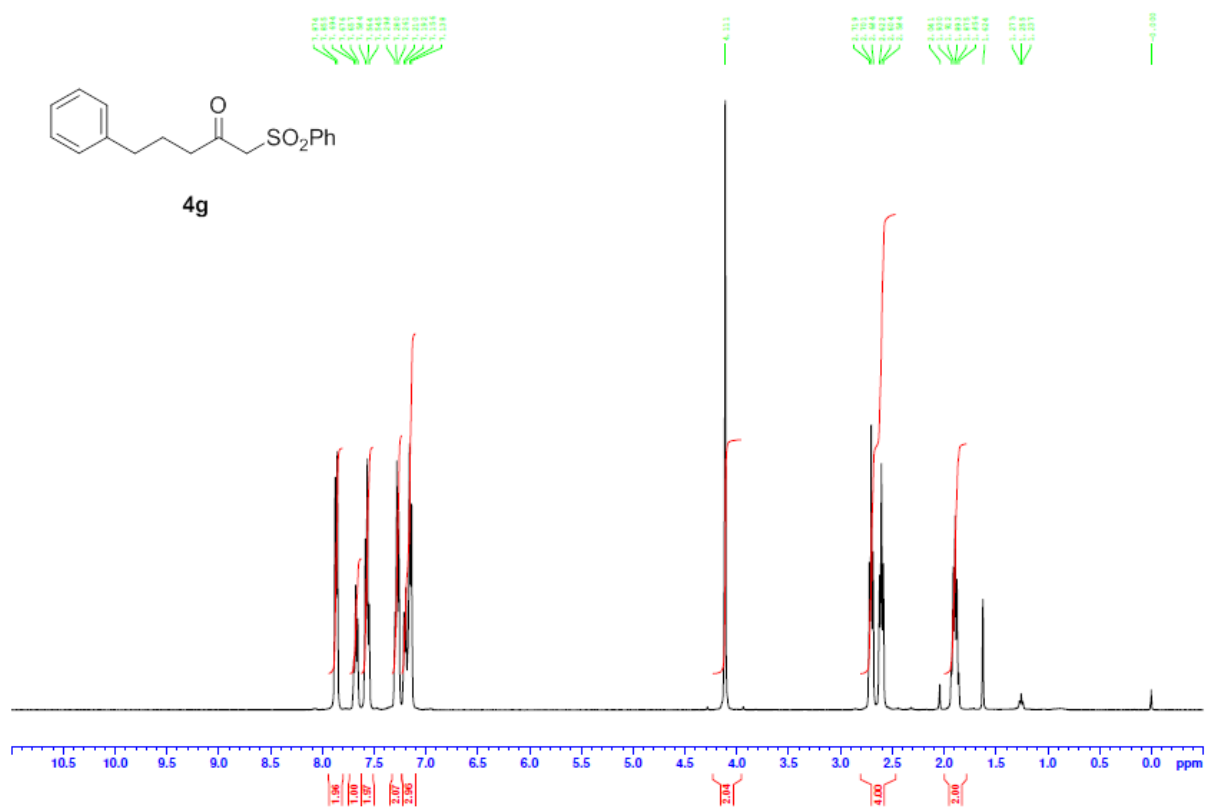


Figure S2.1:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of 4g

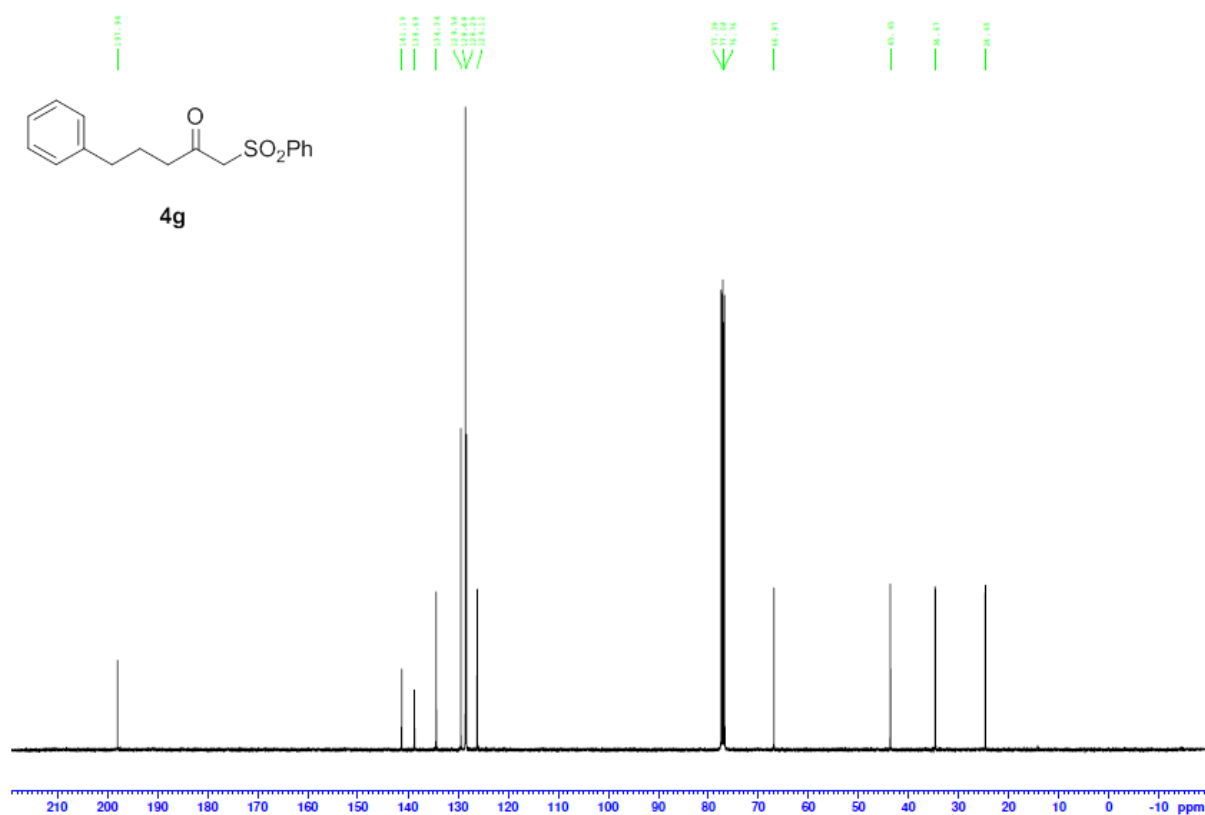


Figure S2.2:  $^{13}\text{C}$  NMR (100.6 MHz,  $\text{CDCl}_3$ ) spectrum of 4g.

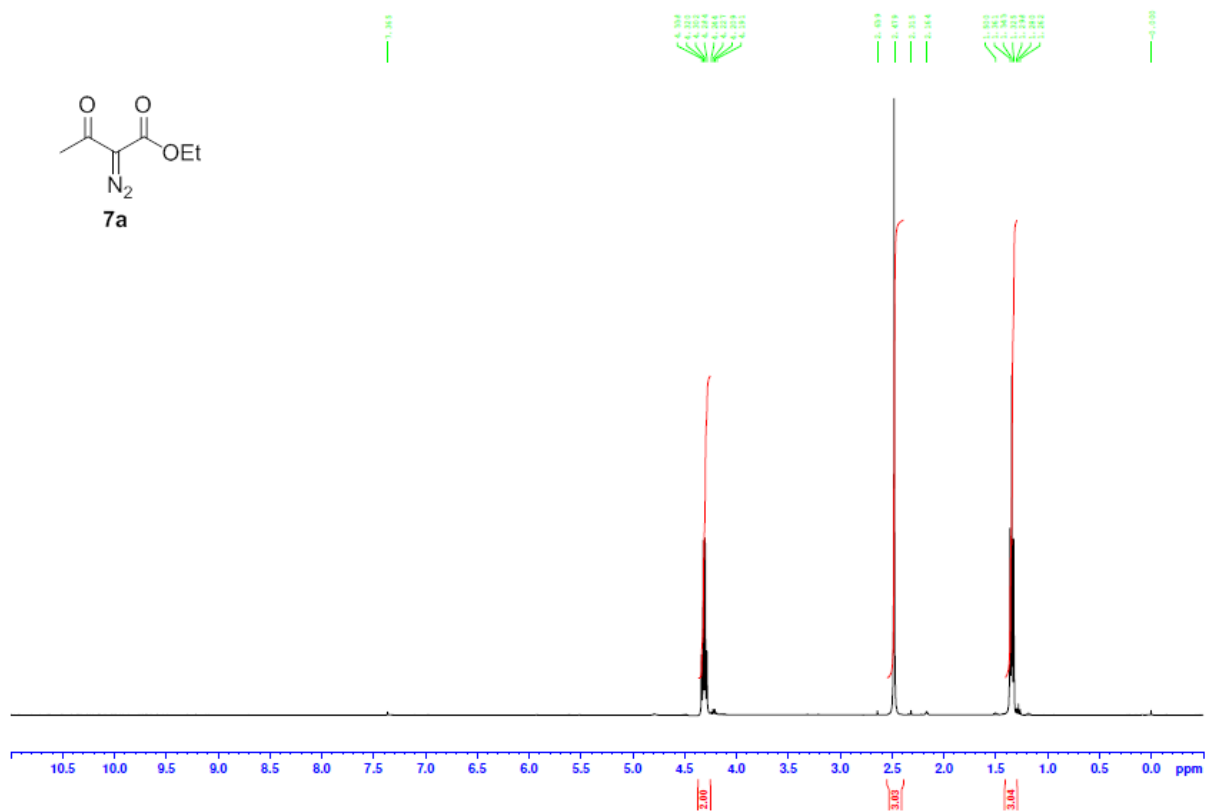


Figure S2.3:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of **7a**.

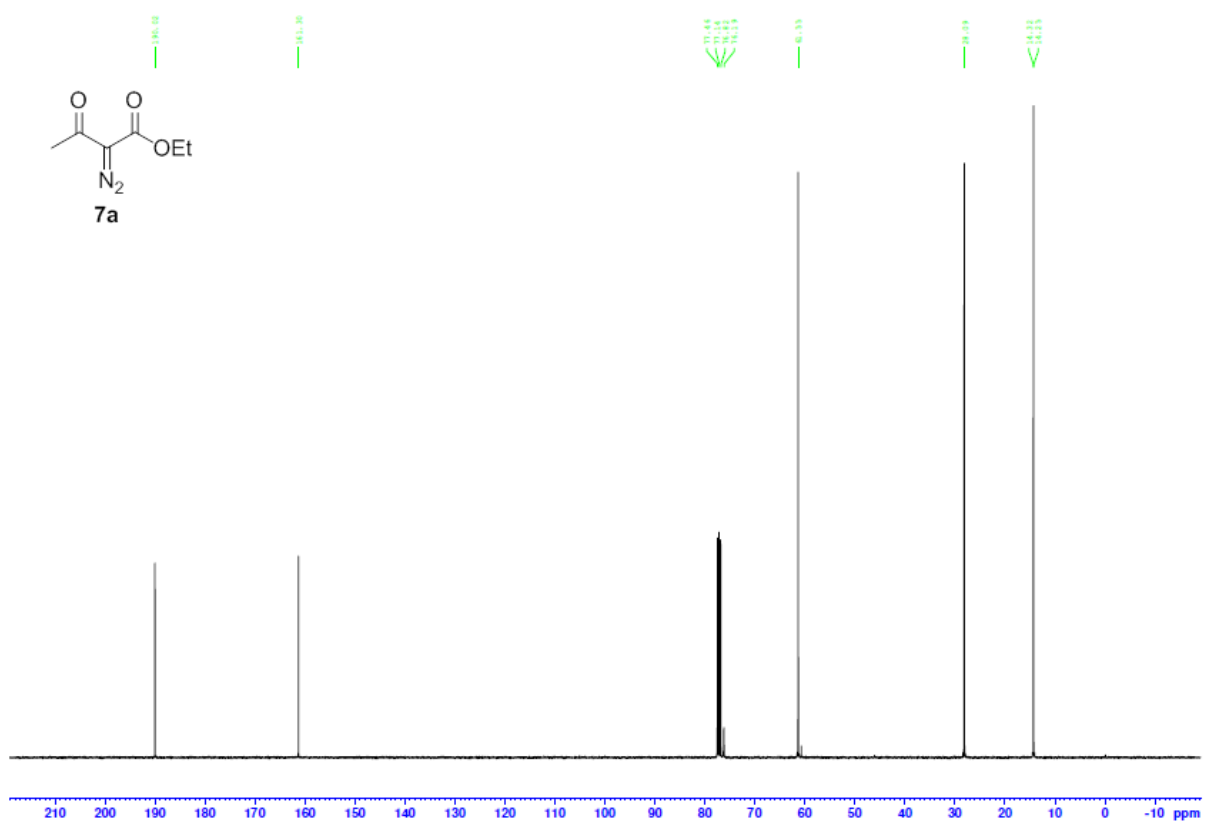


Figure S2.4:  $^{13}\text{C}$  NMR (100.6 MHz,  $\text{CDCl}_3$ ) spectrum of **7a**.

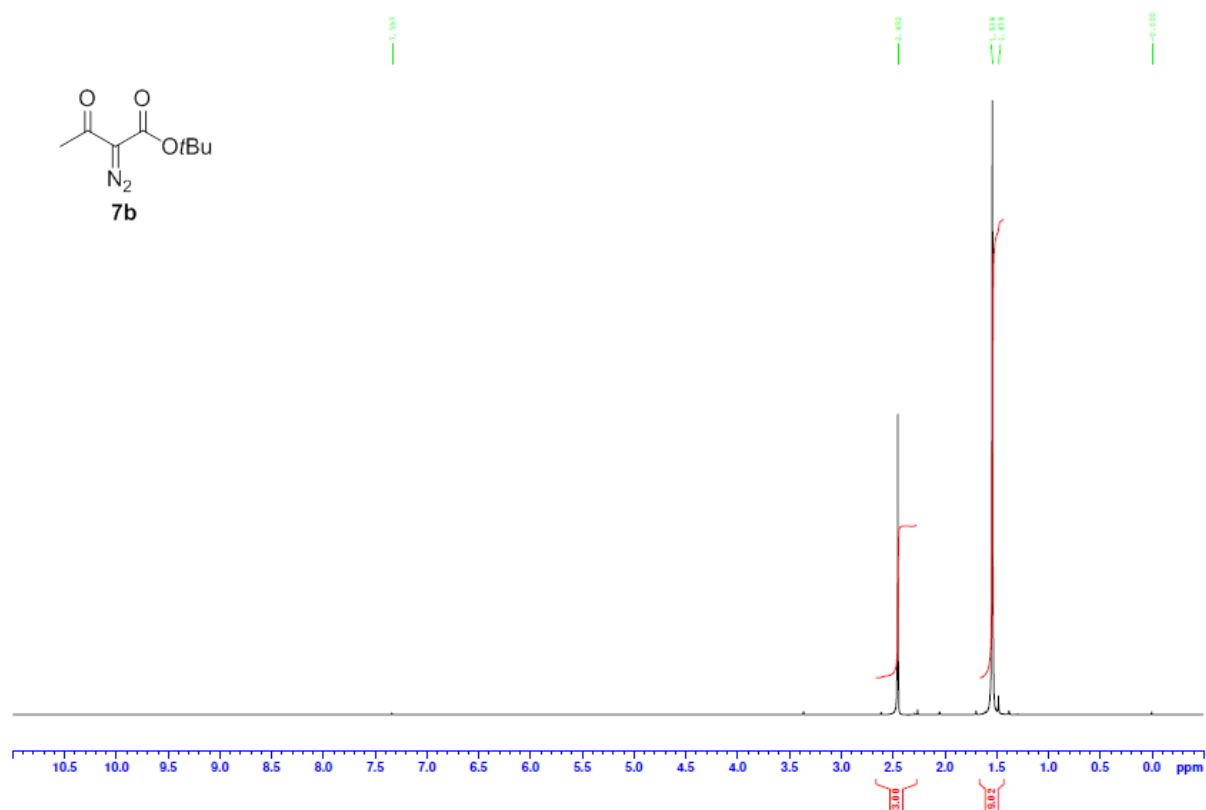


Figure S2.5: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectrum of **7b**.

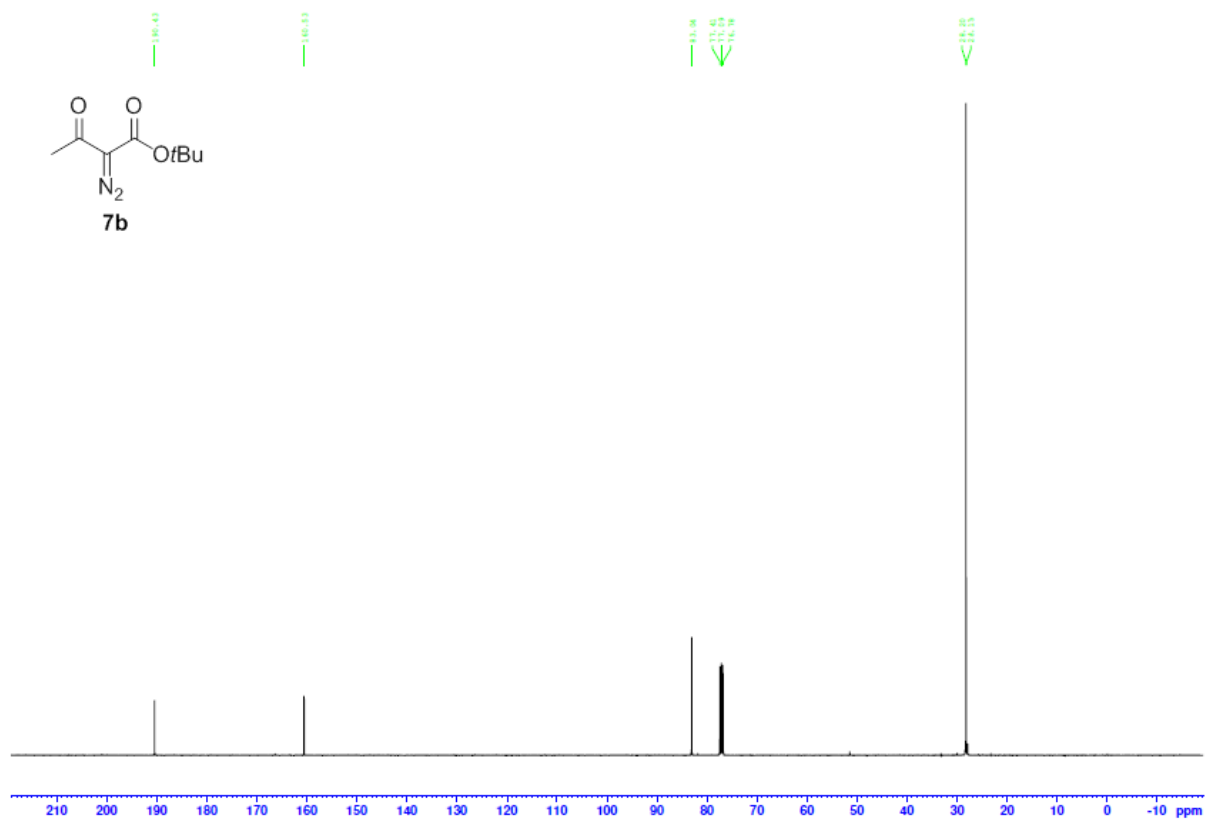


Figure S2.6: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7b**.



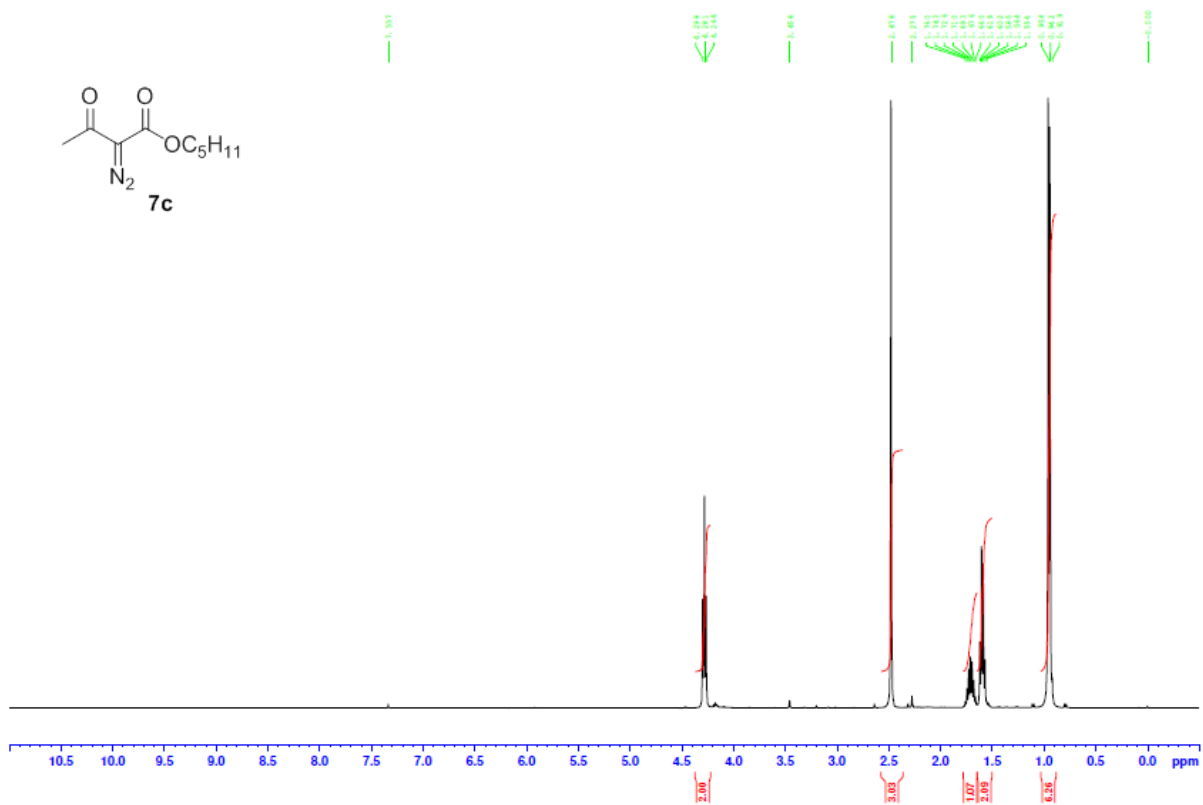


Figure S2.7:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of **7c**.

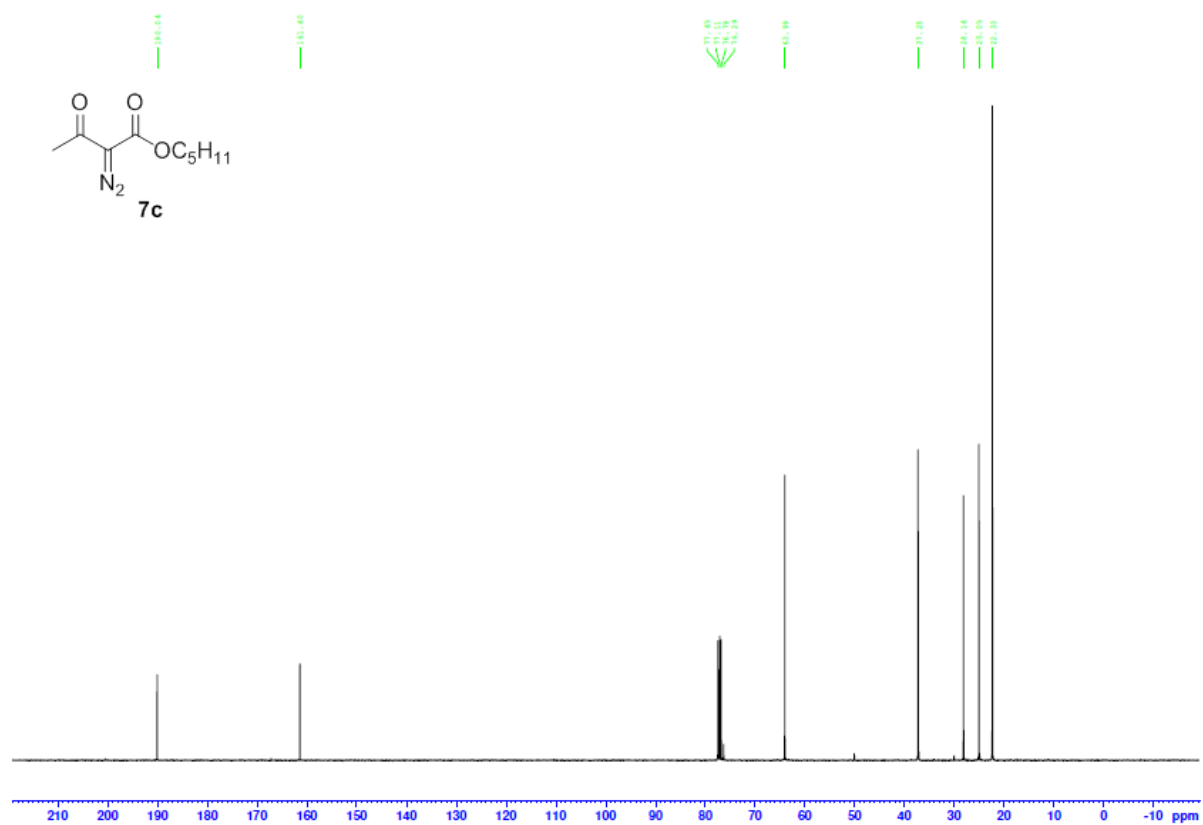


Figure S2.8:  $^{13}\text{C}$  NMR (100.6 MHz,  $\text{CDCl}_3$ ) spectrum of **7c**.

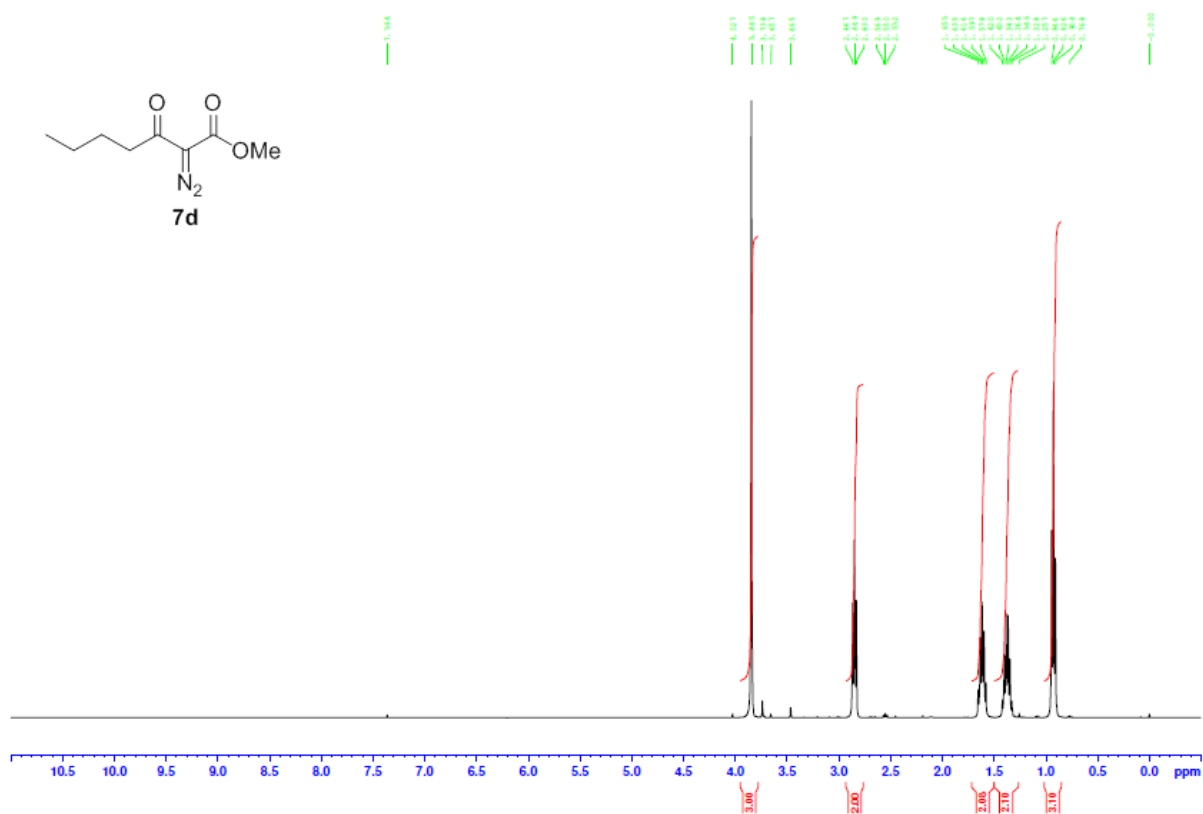


Figure S2.9: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectrum of **7d**.

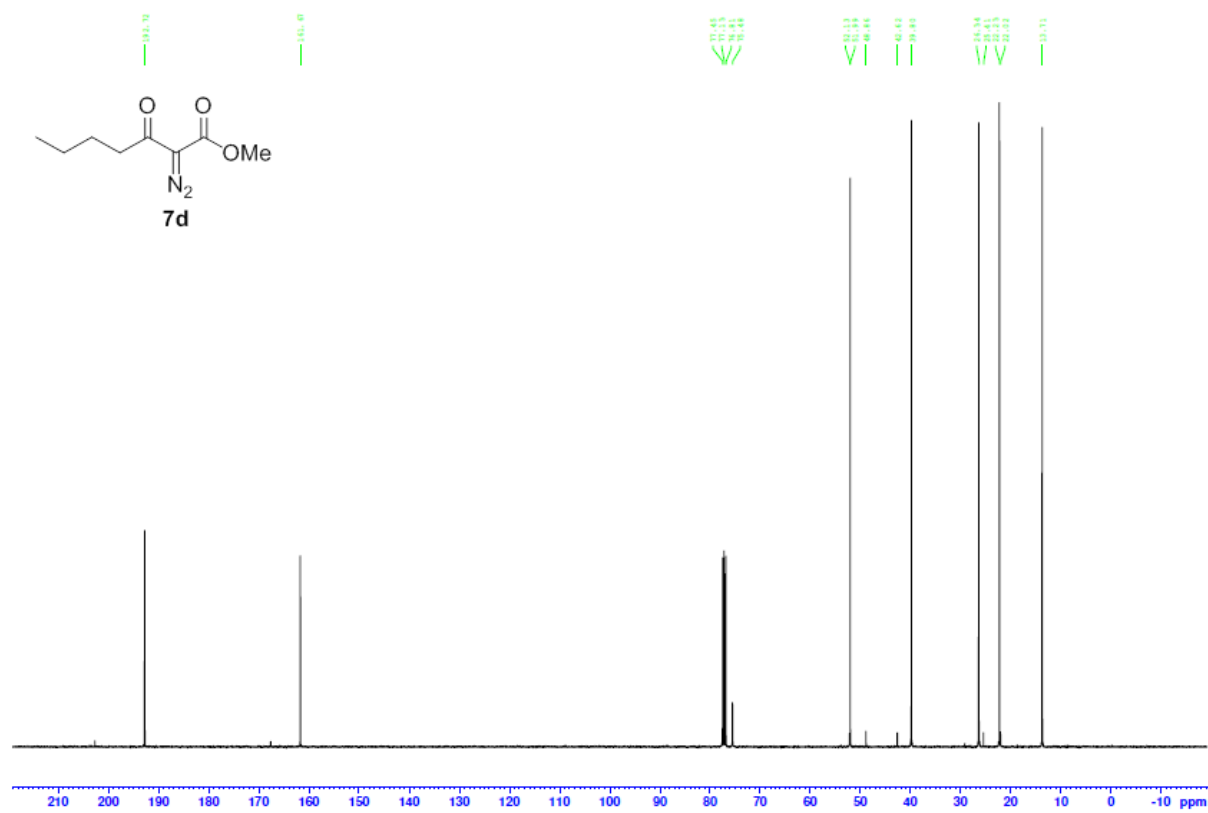
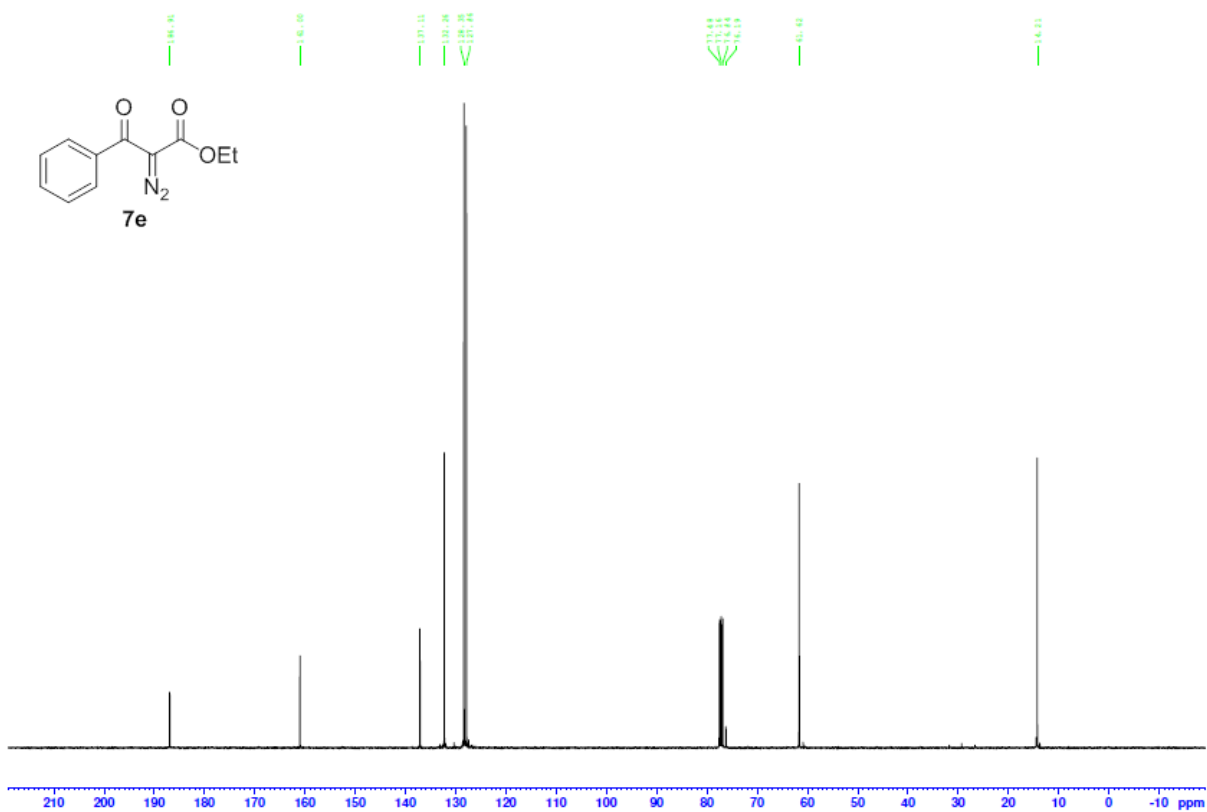
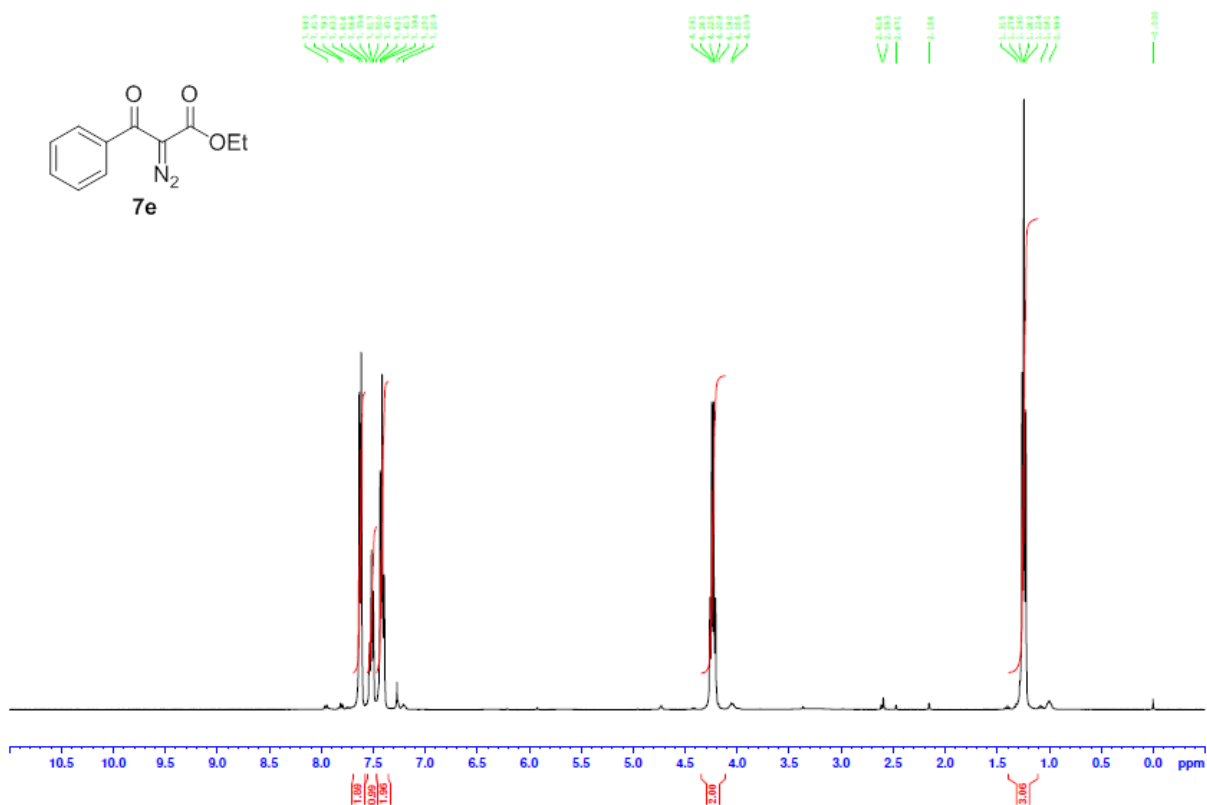


Figure S2.10: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7d**.



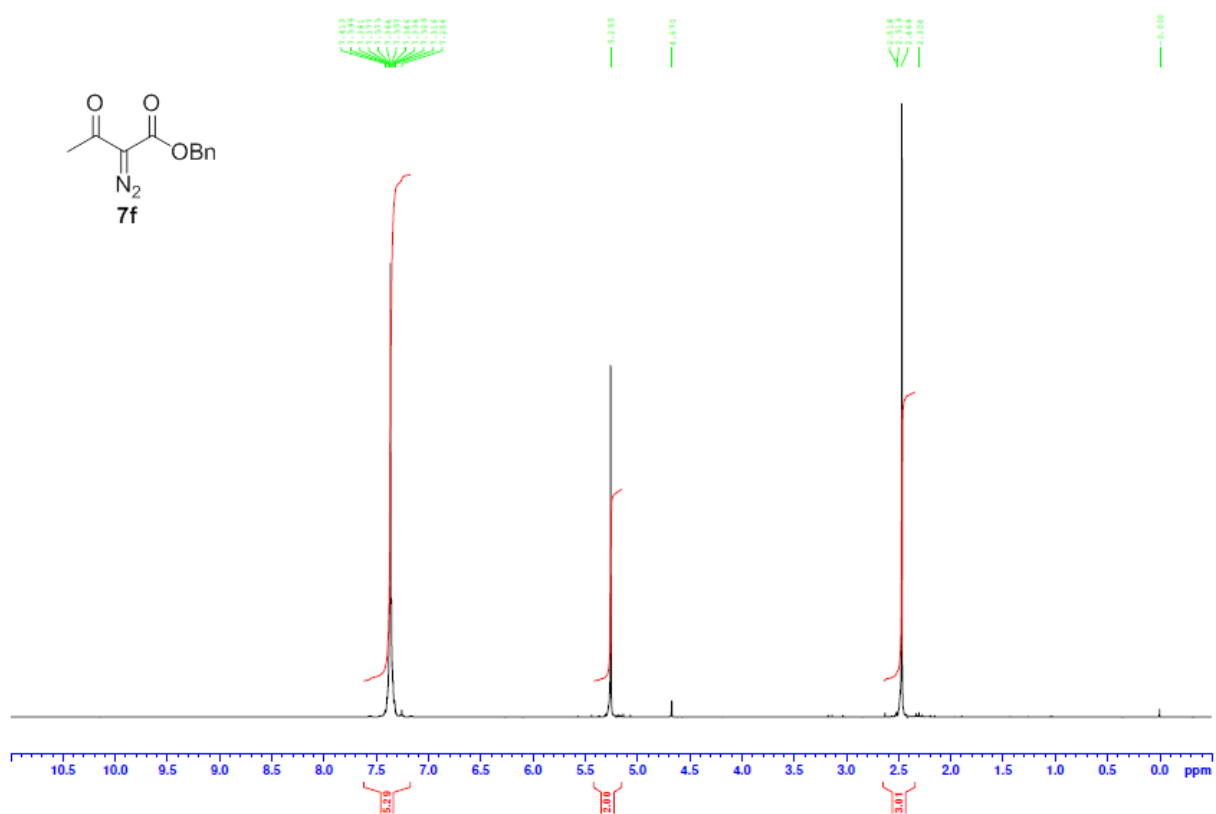


Figure S2.13: <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of **7f**.

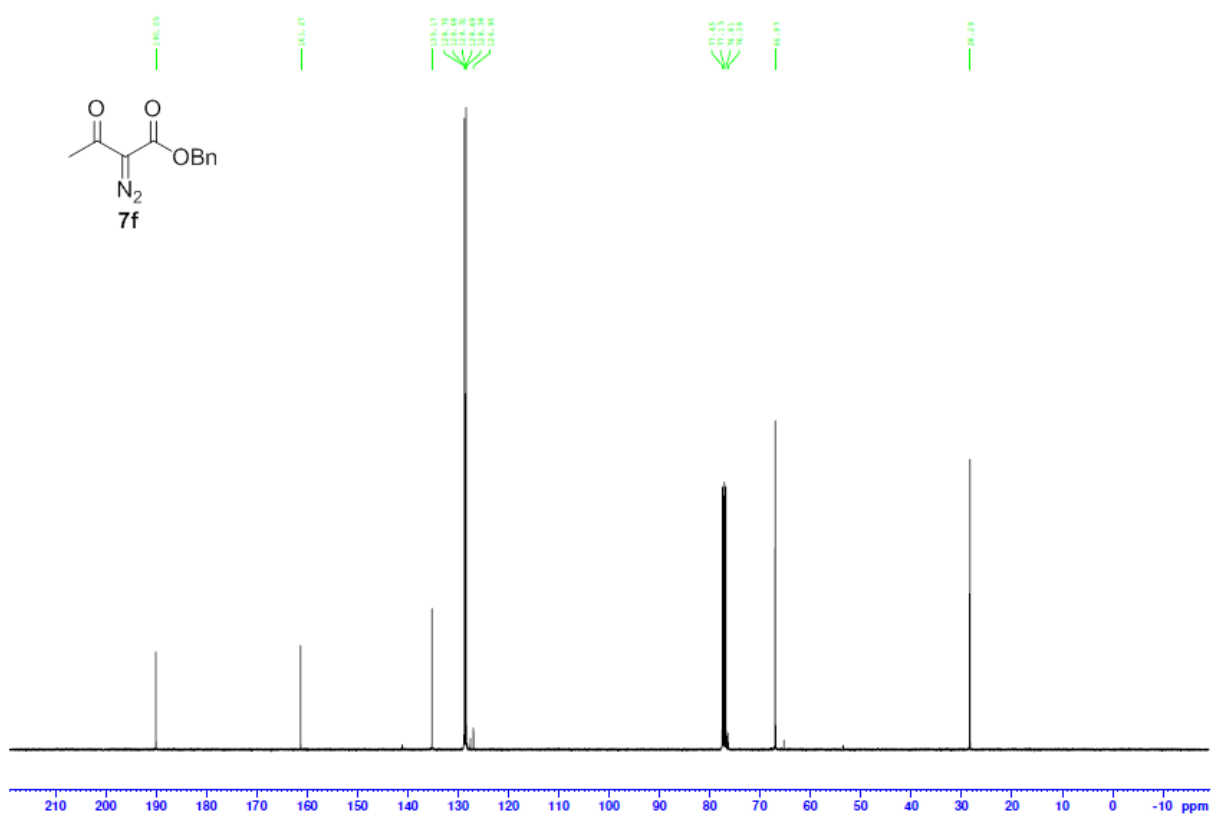


Figure S2.14: <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **7f**.

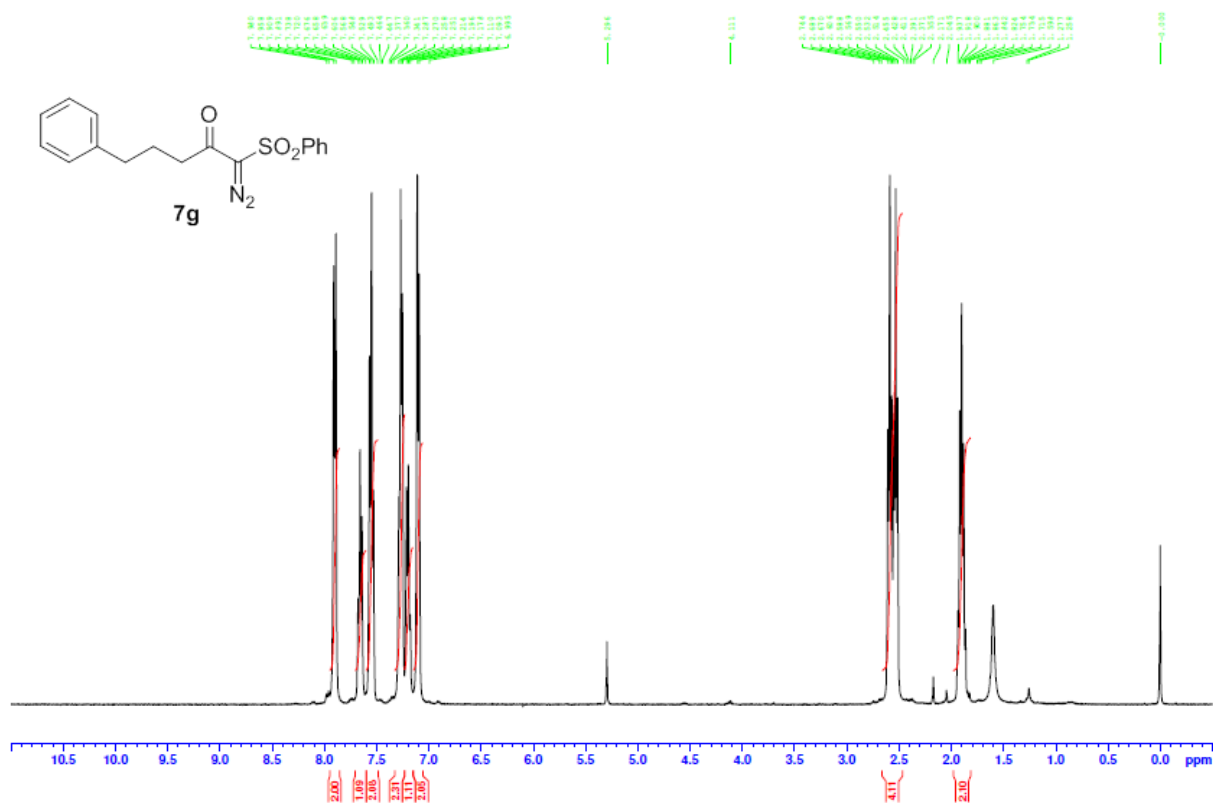


Figure S2.15: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectrum of **7g**.

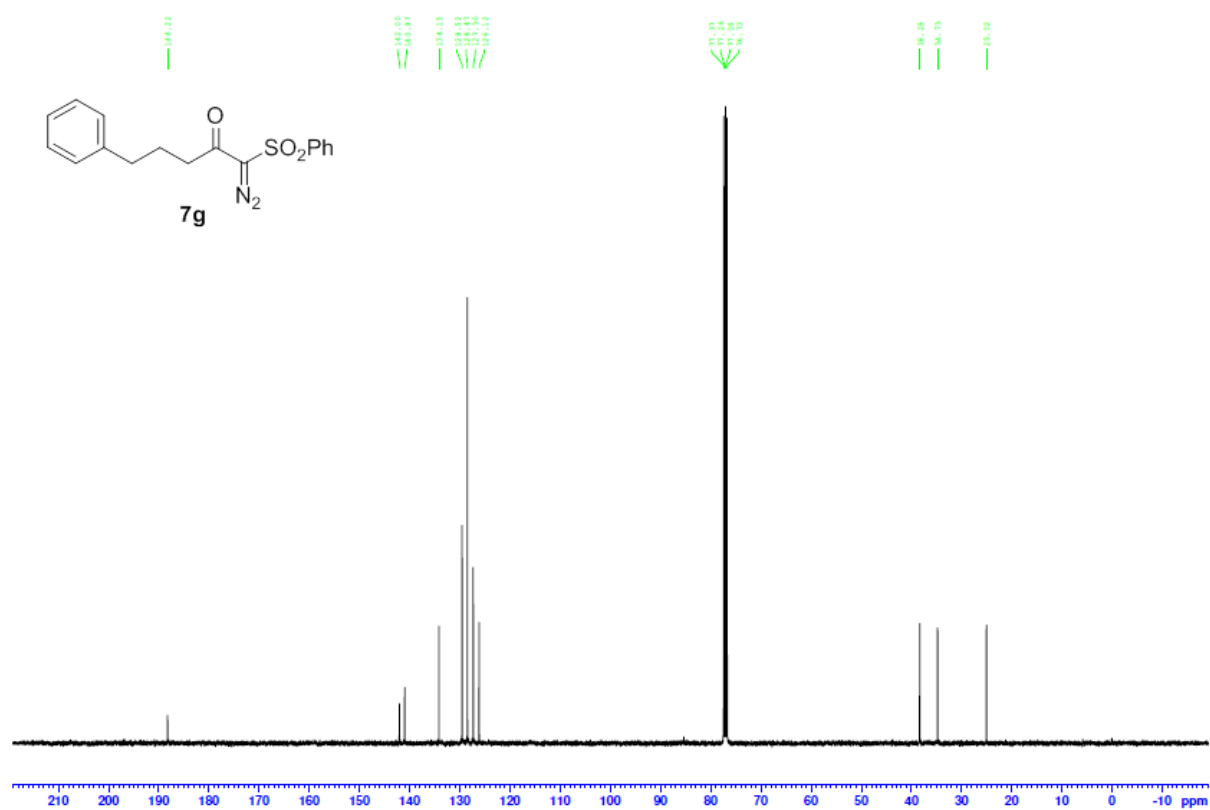


Figure S2.16: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7g**.

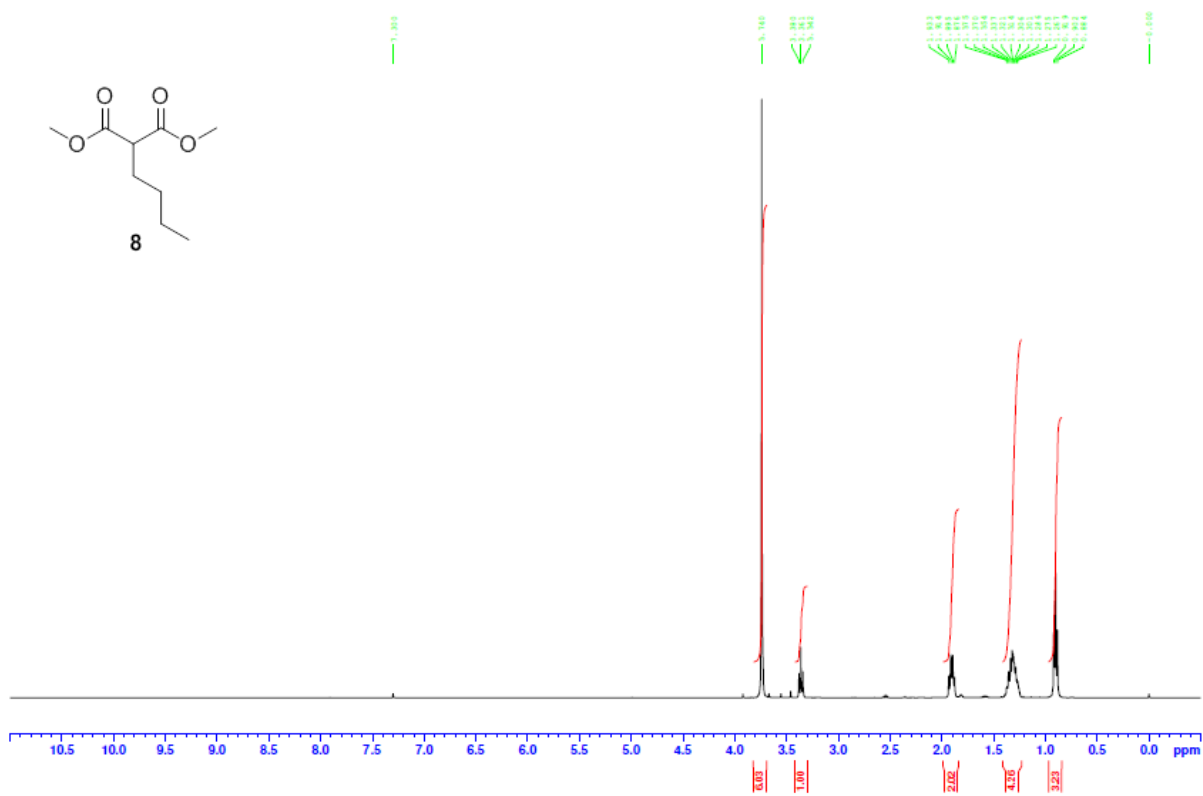


Figure S2.17: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectrum of **8**.

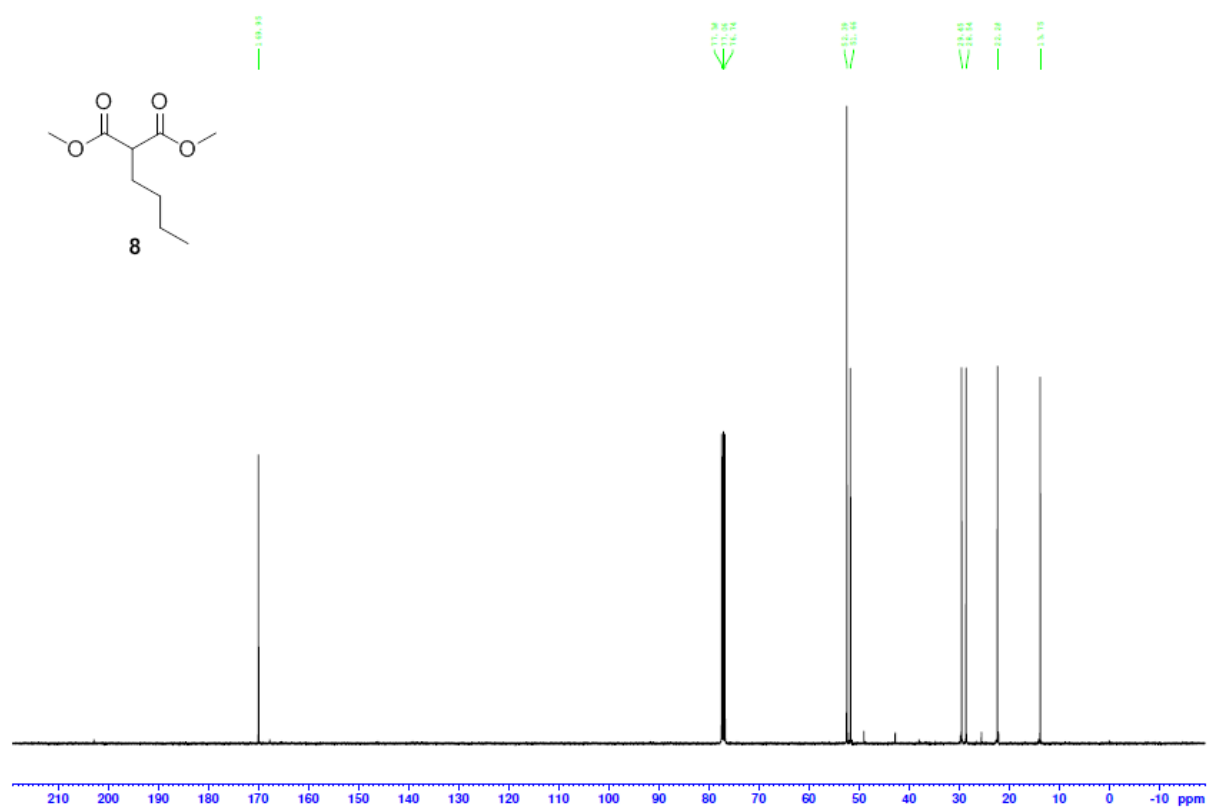


Figure S2.18: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **8**.