

Title	Exploiting the continuous in situ generation of mesyl azide for use in a telescoped process
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#### SUPPORTING INFORMATION

*Title:* Exploiting the Continuous in situ Generation of Mesyl 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\*

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### 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.

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	

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

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: Genera	1 specifications	for E-Series	continuou	s-flow reactor
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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 <sup>1</sup>H and <sup>13</sup>C NMR Spectra for Compounds 4g and 7a–g

*Figure S2.2:* <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **4g**.



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm Figure S2.4:  $^{13}$ C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7a**.



<sup>210</sup> 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm *Figure S2.6:* <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7b**.



*Figure S2.8:* <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7c**.



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 Figure S2.10: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7d**.









 $\frac{210}{100}$   $\frac{200}{100}$   $\frac{190}{180}$   $\frac{170}{160}$   $\frac{160}{150}$   $\frac{140}{130}$   $\frac{120}{120}$   $\frac{110}{100}$   $\frac{90}{100}$ Figure S2.14:  $^{13}$ C NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **7f**.



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm Figure S2.16: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **7g**.





210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm Figure S2.18: <sup>13</sup>C NMR (100.6 MHz, CDCl<sub>3</sub>) spectrum of **8**.