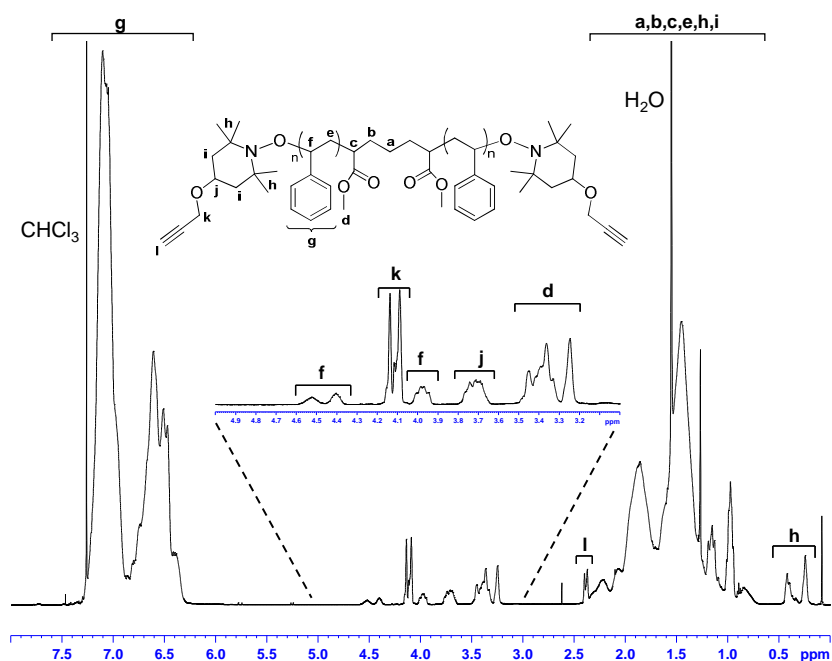


**Accessory Publication**

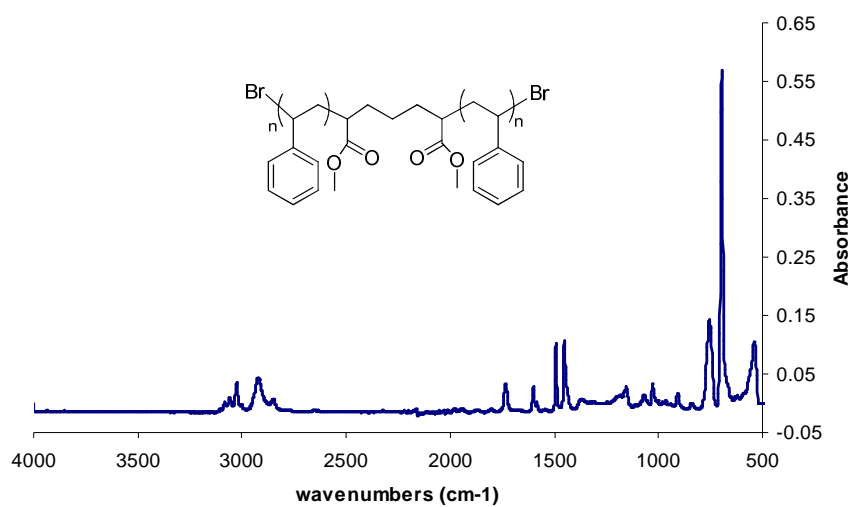
**Ultrafast and Reversible Multiblock Formation  
via an SET-Nitroxide Radical Coupling  
Reaction.**

*Jakov Kulis, Craig A. Bell, Aaron S. Micallef and Michael J. Monteiro\**

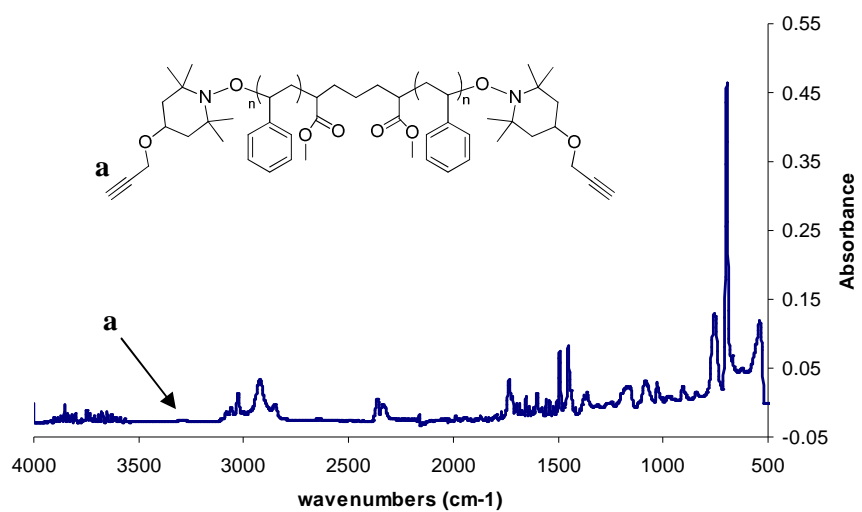
1. Australian Institute for Bioengineering and Nanotechnology, University of  
Queensland, Brisbane QLD 4072, Australia



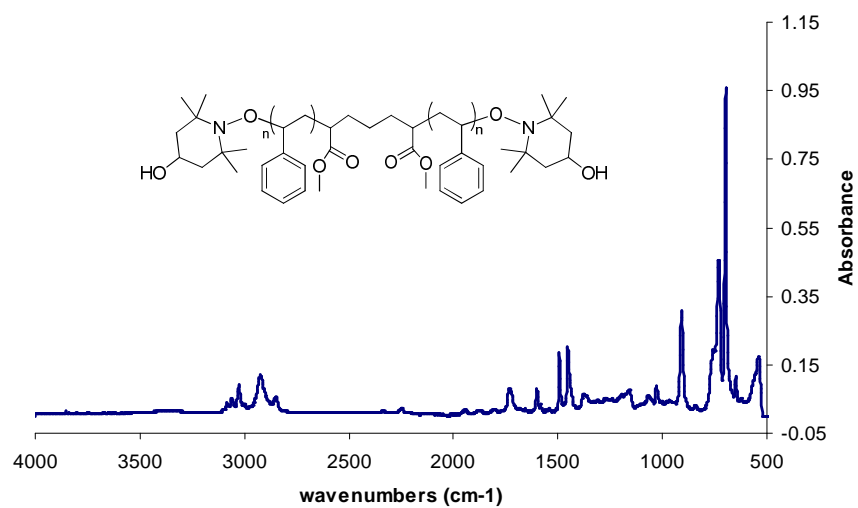
**Figure S1:** <sup>1</sup>H NMR spectra of ≡-T-PSTY-T-≡ formed from SET-NRC of Br-PSTY-Br and Tempo-≡, with expanded view of region between 3-5 ppm. The sample was obtained on a Bruker DRX 400 MHz spectrometer and the solvent used was CDCl<sub>3</sub>.



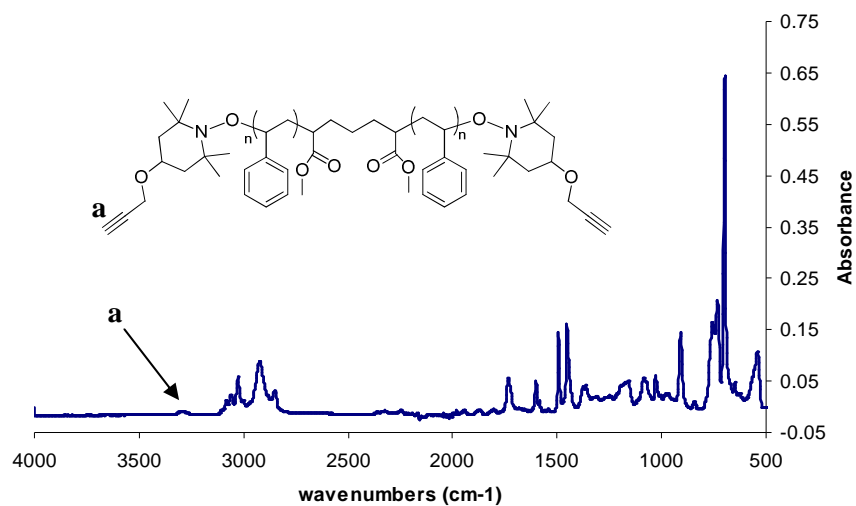
**Figure S2:** ATR-FTIR spectrum of Br-PSTY-Br



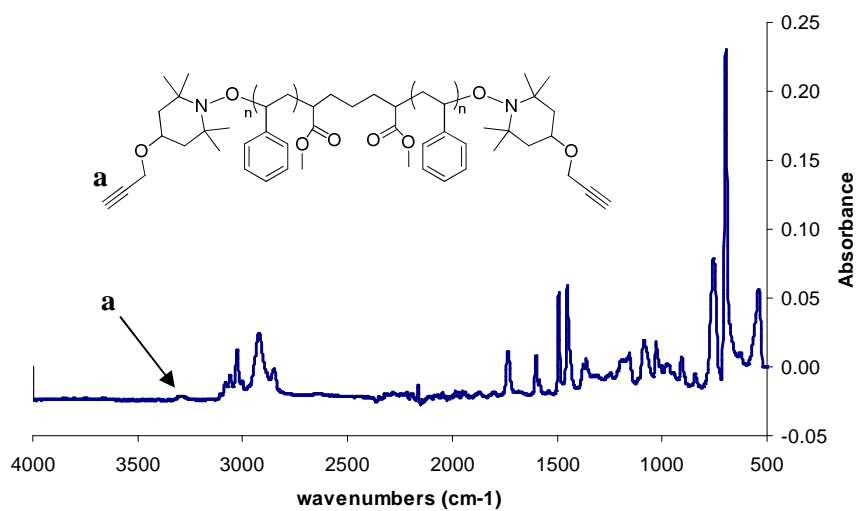
**Figure S3:** ATR-FTIR spectrum of  $\equiv$ -T-PSTY-T- $\equiv$  formed from exchange reaction with multiblocks and TEMPO- $\equiv$



**Figure S4:** ATR-FTIR spectrum of OH-T-PSTY-T-OH formed from exchange reaction with multiblocks and TEMPO-OH



**Figure S5:** ATR-FTIR spectrum of  $\equiv$ -T-PSTY-T- $\equiv$  formed from nitroxide exchange of OH-T-PSTY-T-OH with TEMPO- $\equiv$



**Figure S6:** ATR-FTIR spectrum of  $\equiv$ -T-PSTY-T- $\equiv$  formed from the NRC reaction of Br-PSTY-Br and TEMPO- $\equiv$