



## Supplementary Materials

# Degradable Spirocyclic Polyacetal-Based Core-Amphiphilic Assemblies for Encapsulation and Release of Hydrophobic Cargo

Brandon Andrade-Gagnon, Marilyne Bélanger-Bouliga, Phuong Trang Nguyen, Thi Hong Diep Nguyen, Steve Bourgault and Ali Nazemi \*

Department of Chemistry, Université du Québec à Montréal, C.P.8888, Succursale Centre-Ville, Montréal, QC H3C 3P8, Canada; andrade-gagnon.brandon@courrier.uqam.ca (B.A.-G.); belanger-bouliga.marilyne@courrier.uqam.ca (M.B.-B.); phuong.t.ngn@gmail.com (P.T.N.); nguyen.thi\_hong\_diep@courrier.uqam.ca (T.H.D.N.); bourgault.steve@uqam.ca (S.B.)

\* Correspondence: nazemi.ali@uqam.ca

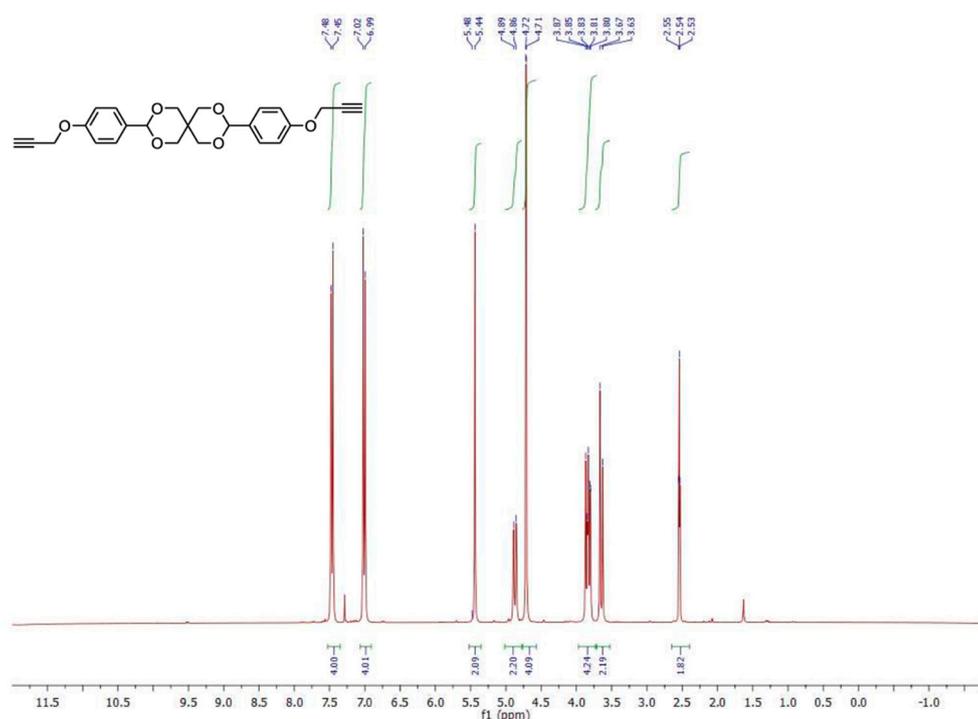
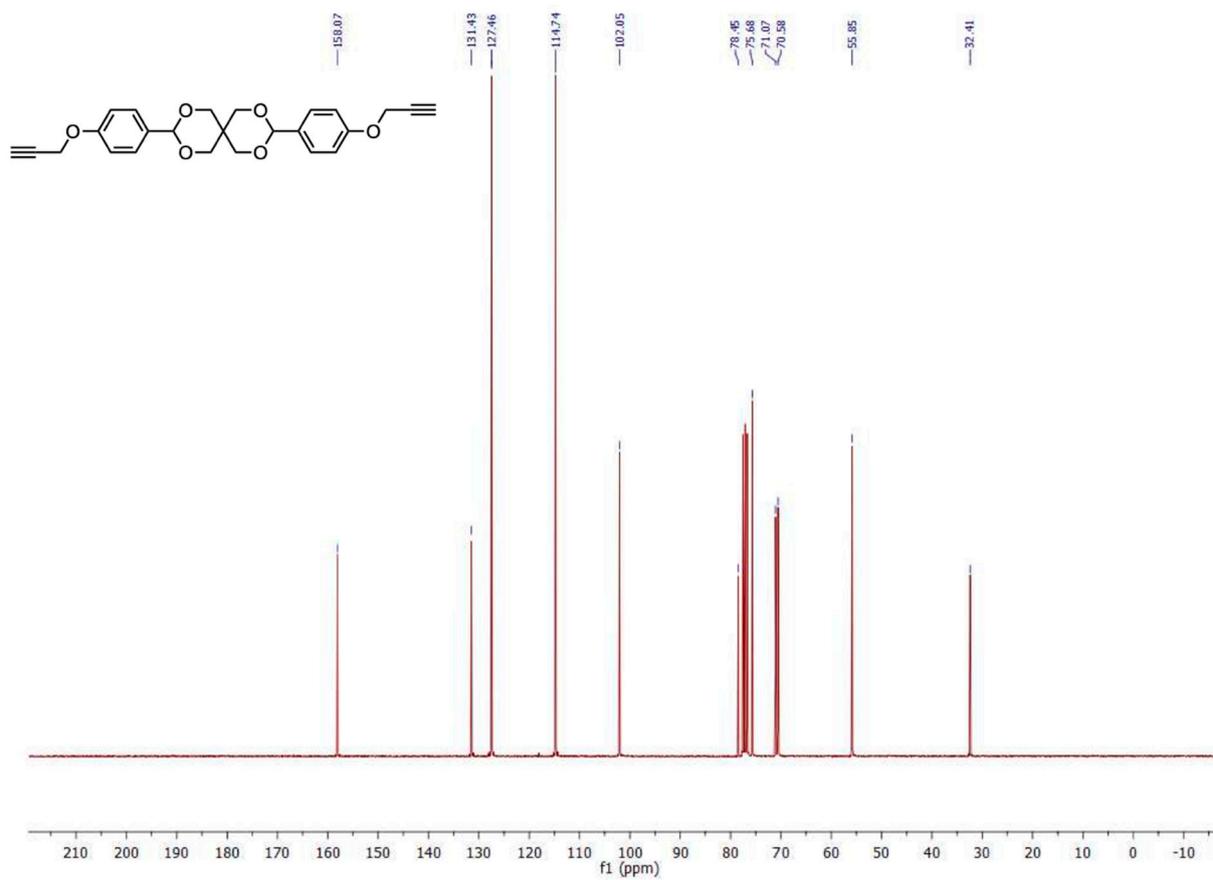
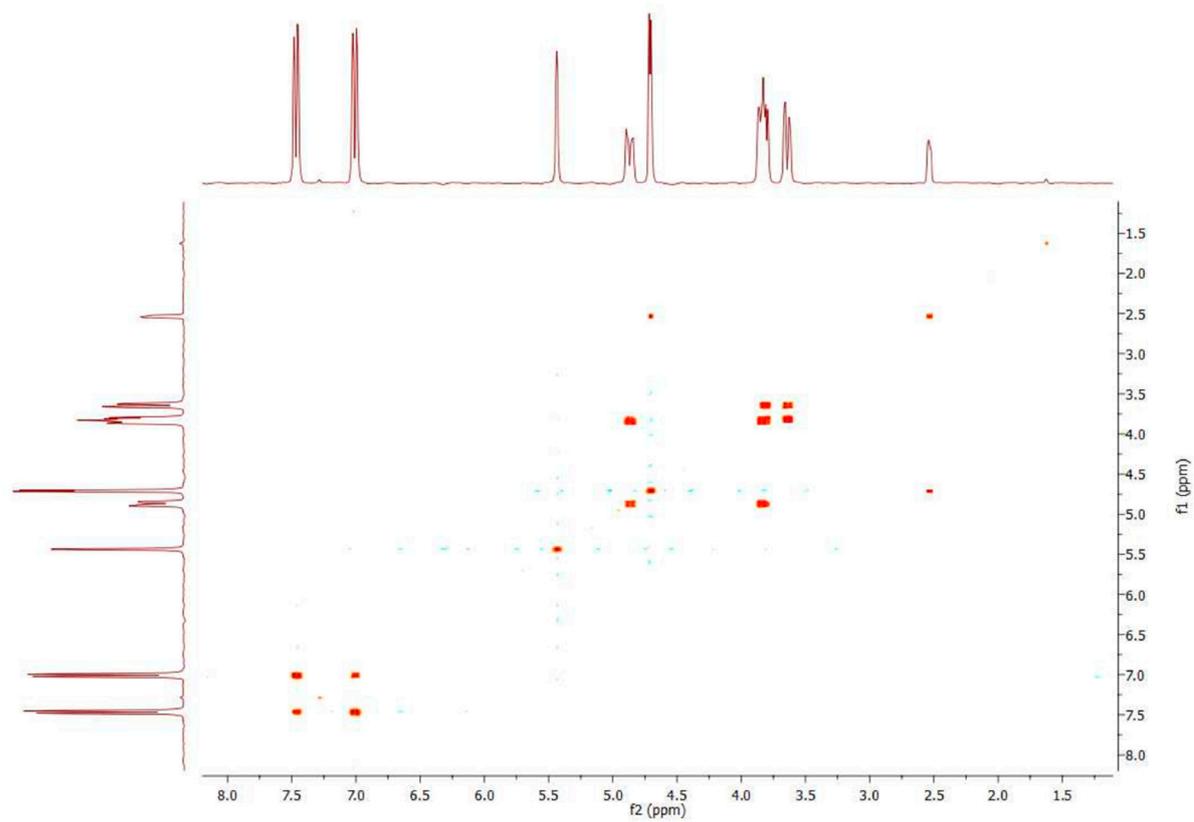


Figure S1. <sup>1</sup>H NMR spectrum of compound 2 (300 MHz, CDCl<sub>3</sub>).



**Figure S2.**  $^{13}\text{C}$  NMR spectrum of compound 2 (75 MHz,  $\text{CDCl}_3$ ).



**Figure S3.** 2D  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound 2 (300 MHz,  $\text{CDCl}_3$ ).

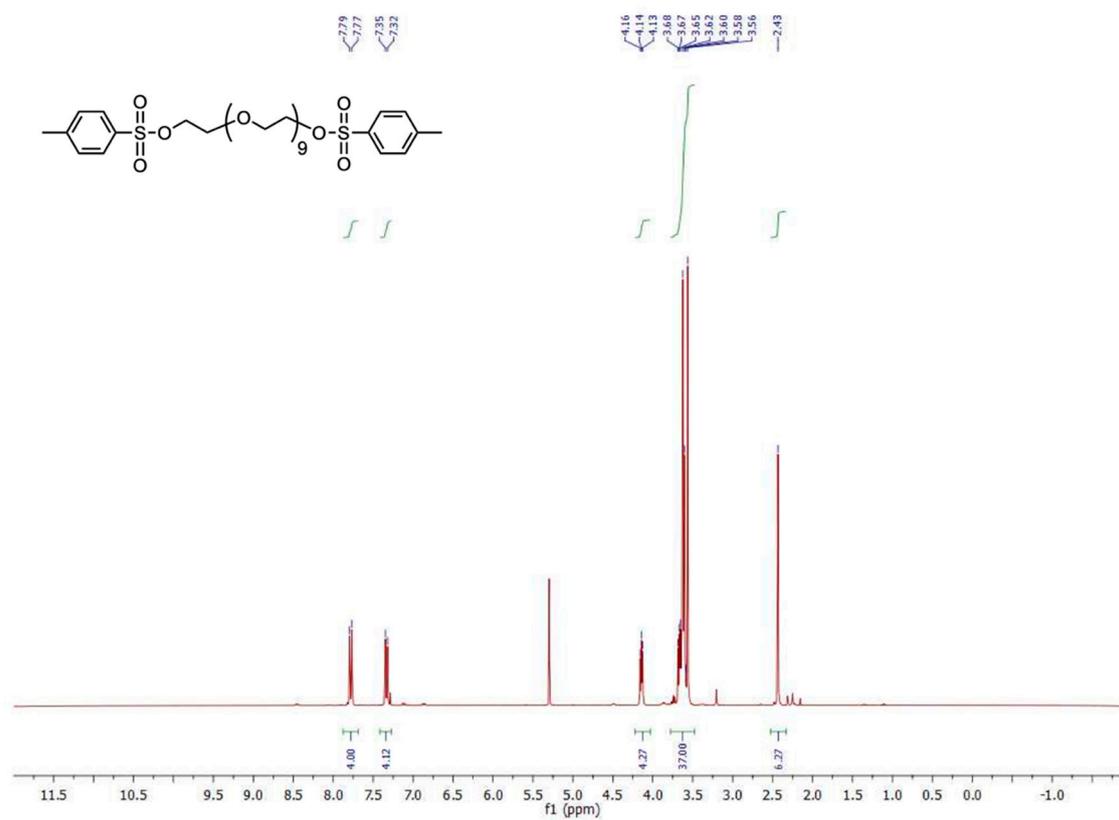


Figure S4. <sup>1</sup>H NMR spectrum of compound 3 (300 MHz, CDCl<sub>3</sub>).

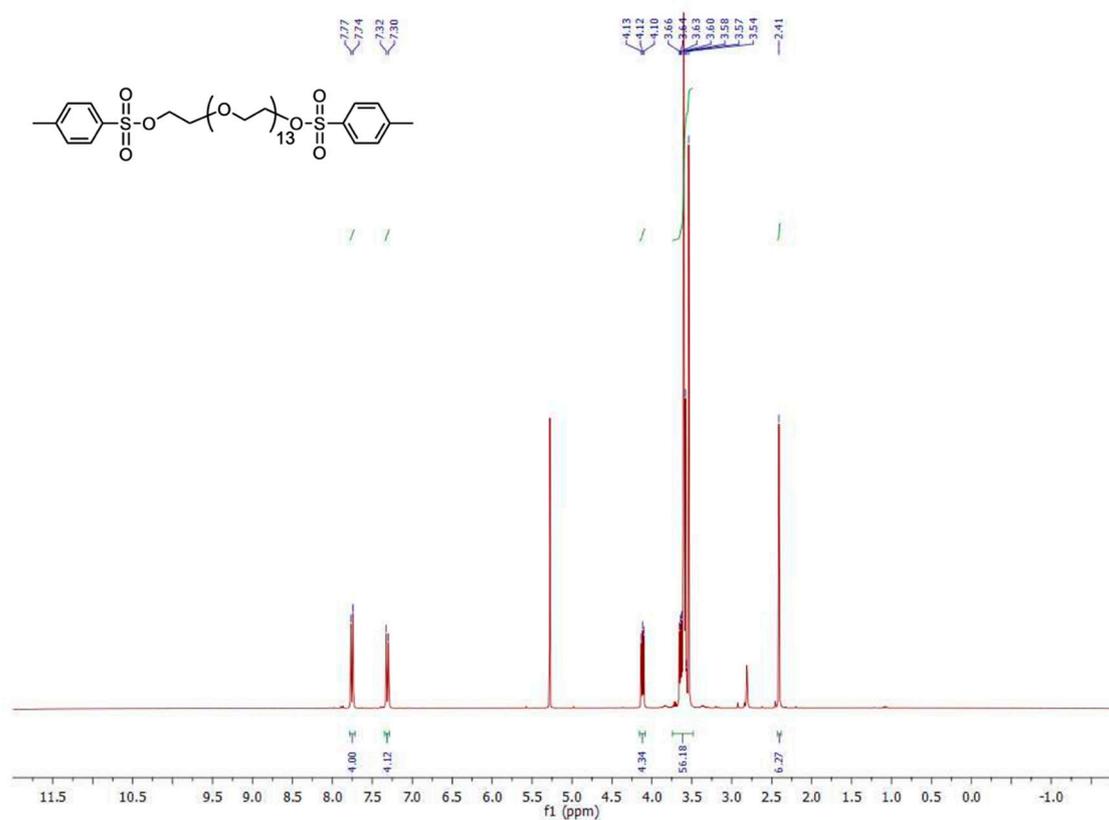
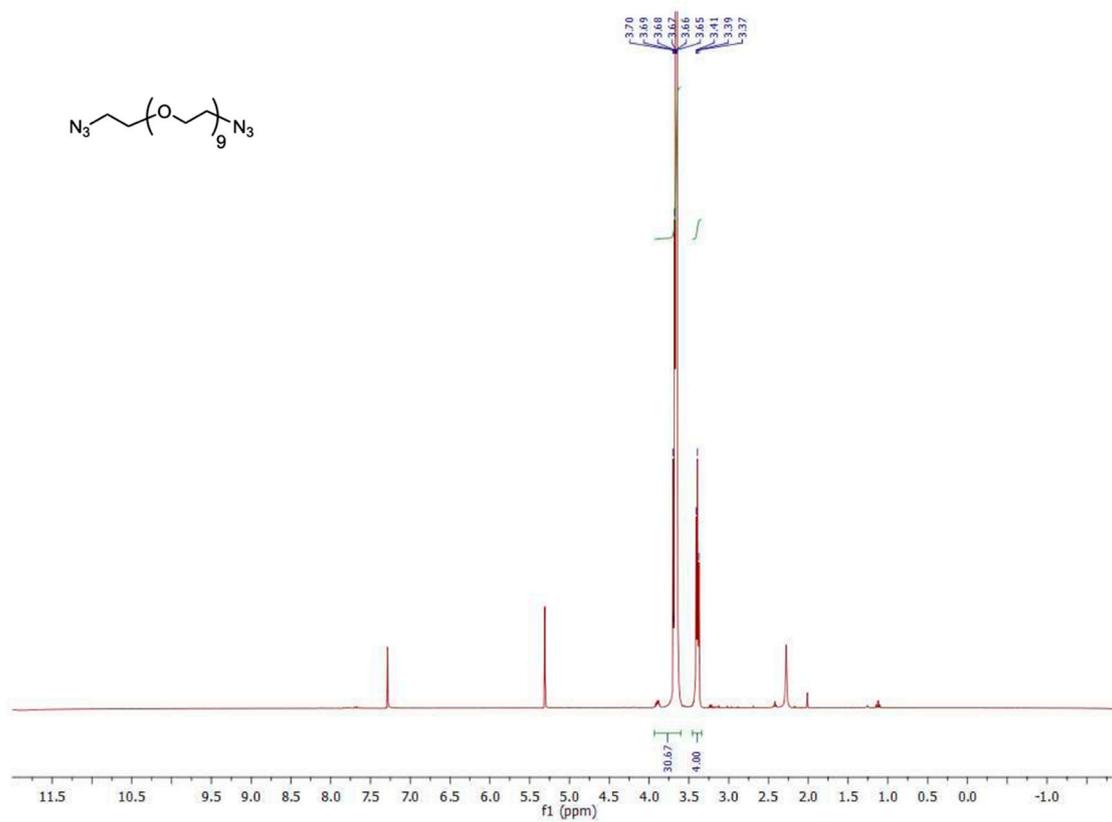


Figure S5. <sup>1</sup>H NMR spectrum of compound 4 (300 MHz, CDCl<sub>3</sub>).



**Figure S6.** <sup>1</sup>H NMR spectrum of compound 5 (300 MHz, CDCl<sub>3</sub>).

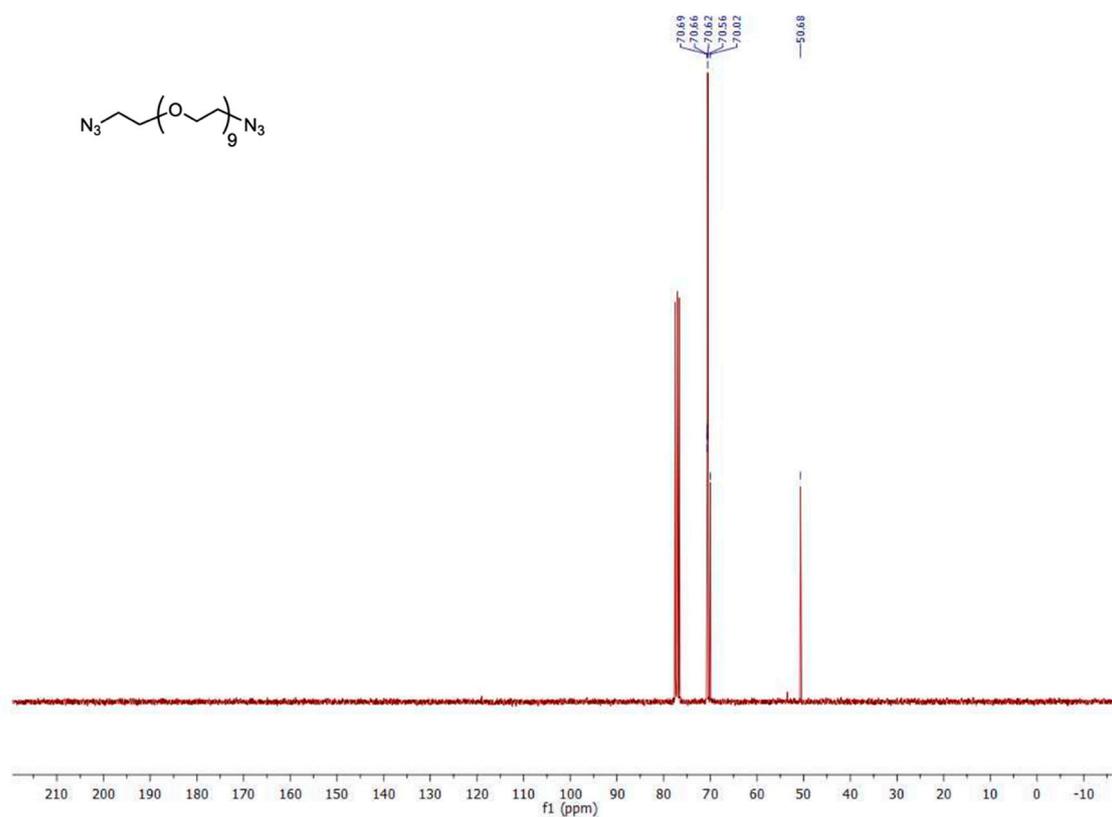


Figure S7.  $^{13}\text{C}$  NMR spectrum of compound 5 (75 MHz,  $\text{CDCl}_3$ ).

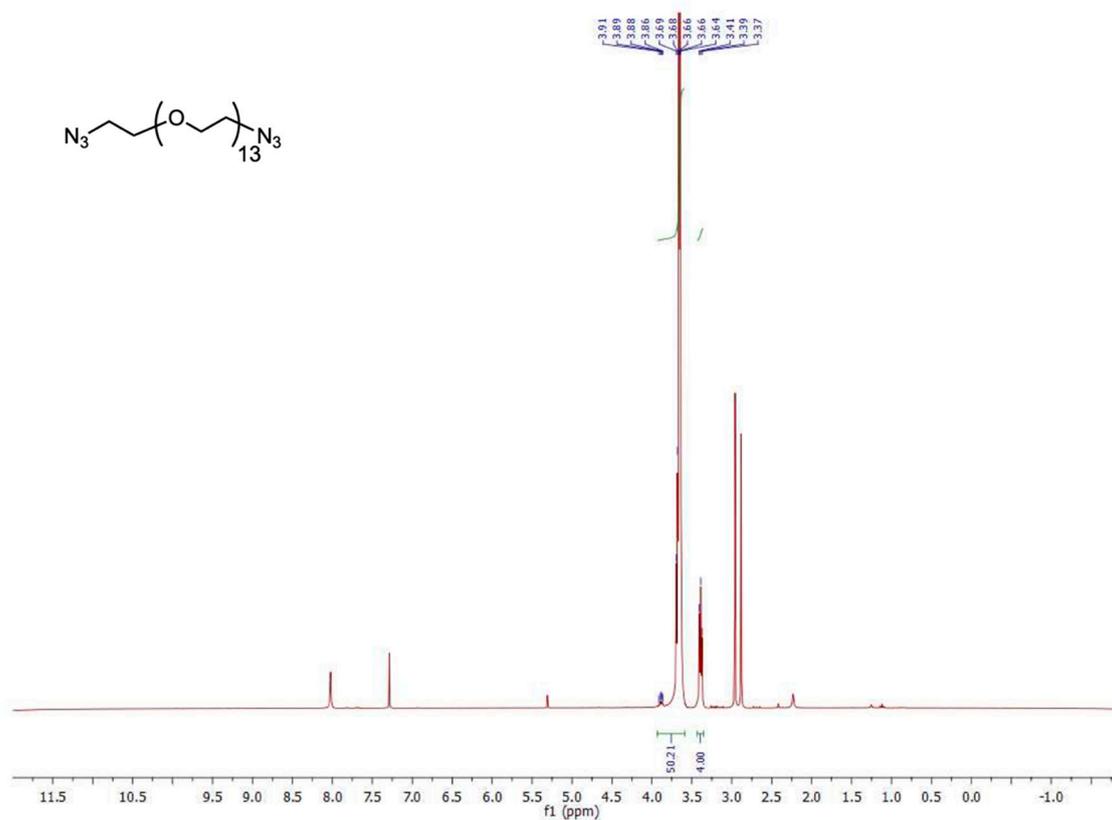
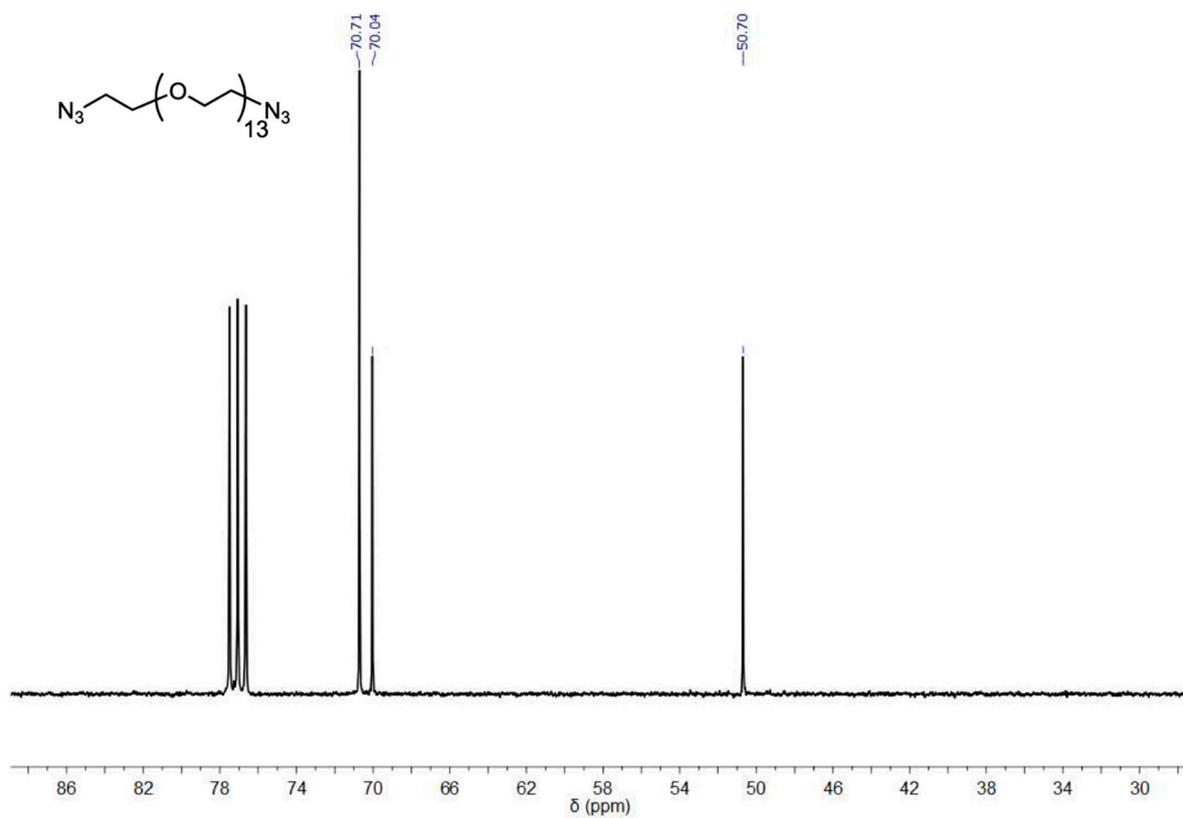
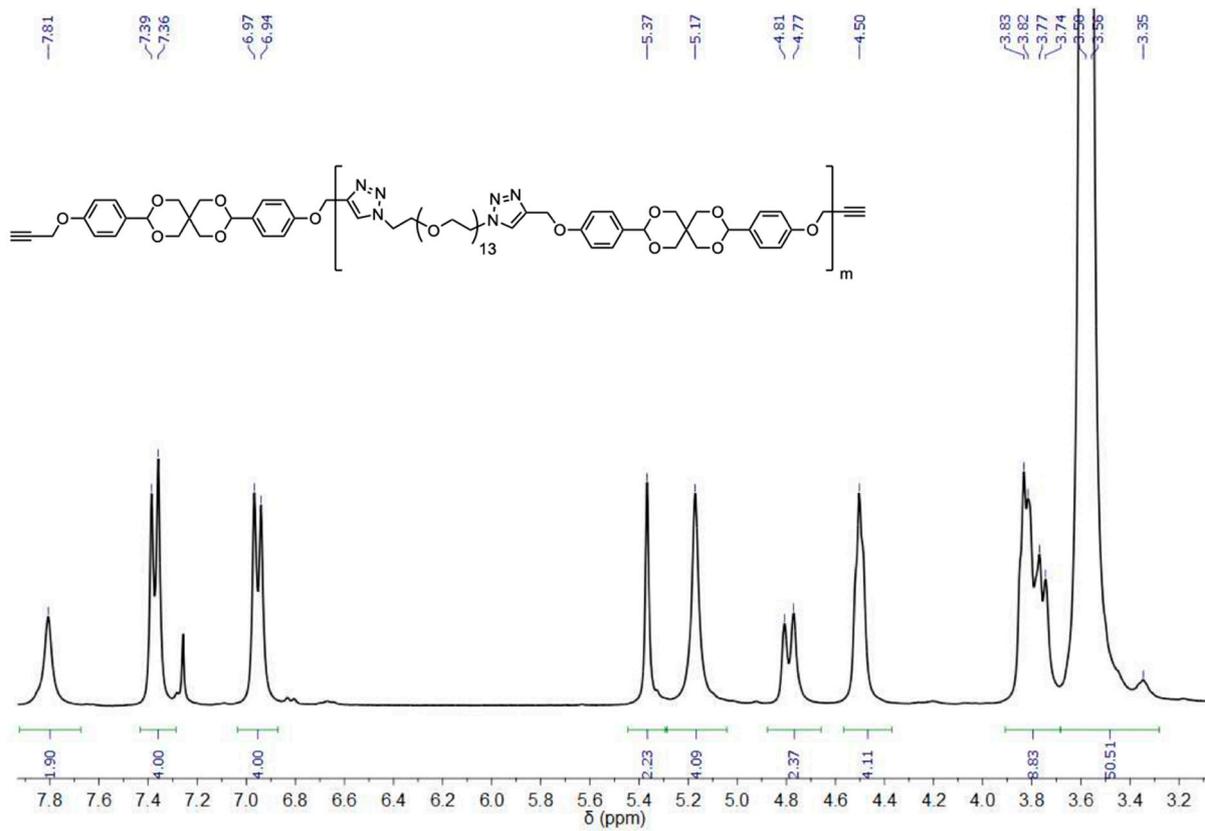


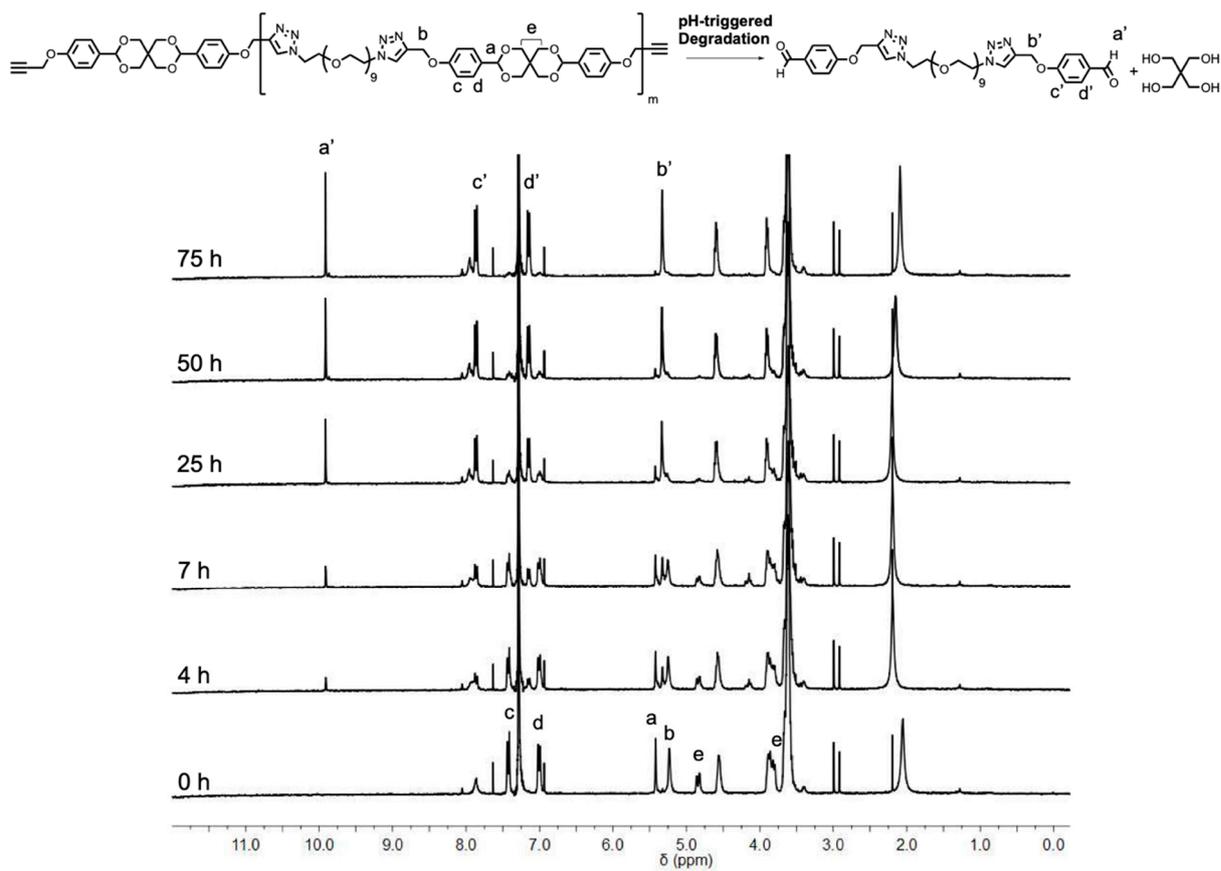
Figure S8.  $^1\text{H}$  NMR spectrum of compound 6 (300 MHz,  $\text{CDCl}_3$ ).



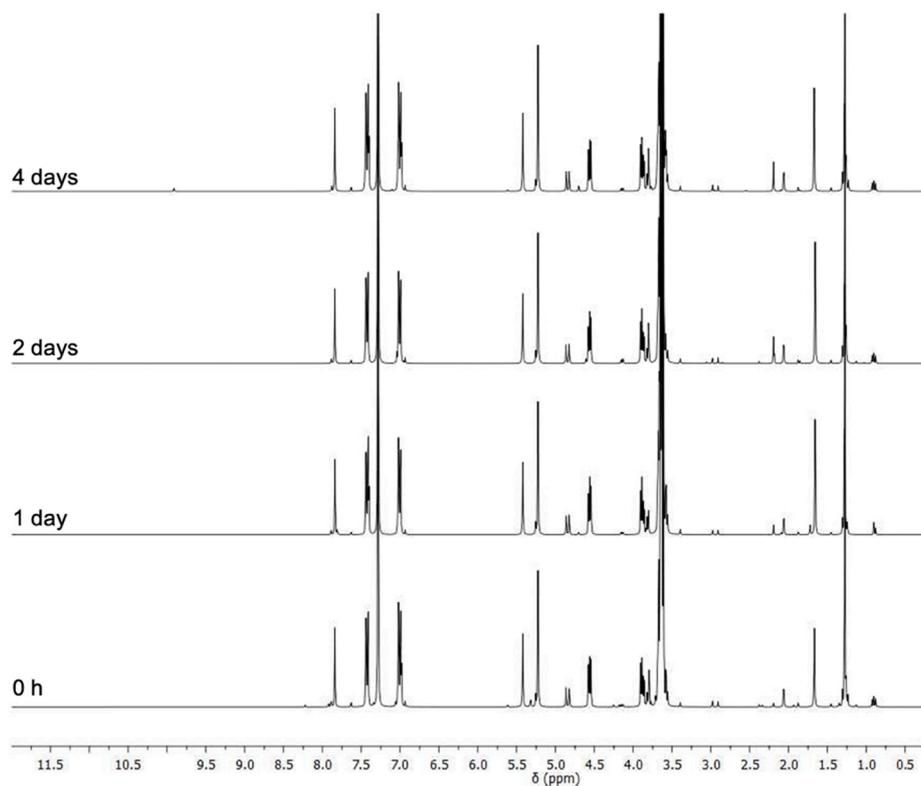
**Figure S9.**  $^{13}\text{C}$  NMR spectrum of compound **6** (75 MHz,  $\text{CDCl}_3$ ).



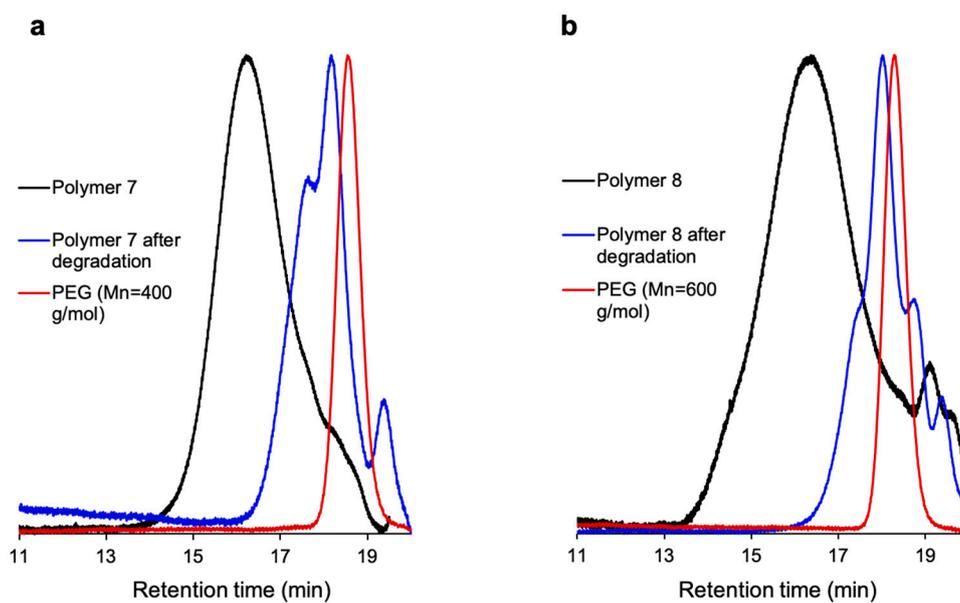
**Figure S10.** <sup>1</sup>H NMR spectrum of polymer 8 (300 MHz, CDCl<sub>3</sub>).



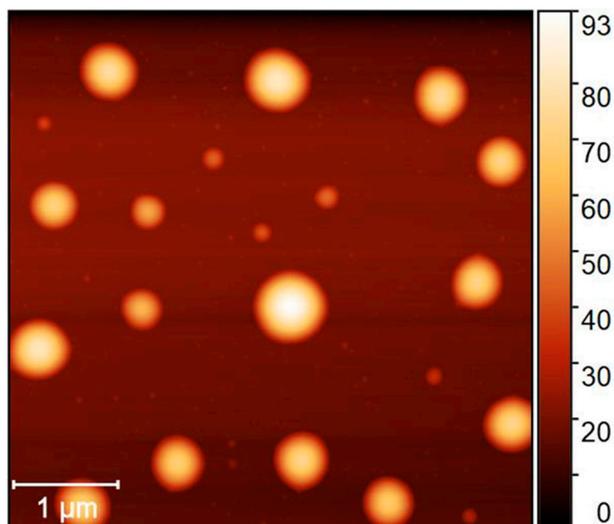
**Figure S11.** pH-triggered degradation of polymer 8 in 80 mM trifluoroacetic acid in  $\text{CDCl}_3$  monitored by  $^1\text{H}$  NMR spectroscopy.



**Figure S12.** Stability of 7 in non-acidic  $\text{CDCl}_3$  monitored by  $^1\text{H}$  NMR spectroscopy.



**Figure S13.** Refractive index (RI) traces in the SEC analysis of (a) polymer 7 before and after degradation as well as the PEG starting material ( $M_n = 400$  g/mol) and (b) polymer 8 before and after degradation as well as the PEG starting material ( $M_n = 600$  g/mol).



**Figure S14.** AFM image of Nile red-loaded particles (**P1**) formed by polymer 7.



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