

## Supporting Information

### **Biocompatible, pH-sensitive AB<sub>2</sub> Miktoarm Polymer-Based Polymersomes: Preparation, Characterization, and Acidic pH-Activated Nanostructural Transformation**

Haiqing Yin<sup>a#</sup>, Han Chang Kang<sup>b#</sup>, Kang Moo Huh<sup>c</sup>, You Han Bae<sup>\*ad</sup>

<sup>a</sup> Department of Pharmaceutics and Pharmaceutical Chemistry, The University of Utah, 421 Wakara Way, Suite 318, Salt Lake City, Utah 84108, USA

<sup>b</sup> Department of Pharmacy and Integrated Research Institute of Pharmaceutical Sciences, College of Pharmacy, The Catholic University of Korea, 43 Jibong-ro, Wonmi-gu, Bucheon-si, Gyeonggi-do 420-743, Republic of Korea

<sup>c</sup> Department of Polymer Science and Engineering, Chungnam National University, 220 Gungdong, Yuseong-gu, Daejeon 305-764, Republic of Korea

<sup>d</sup> Utah-Inha Drug Delivery Systems (DDS) and Advanced Therapeutics Research Center, 7-50 Songdo-dong, Yeonsu-gu, Incheon, 406-840, Republic of Korea

# YH and HCK equally contributed to this work.

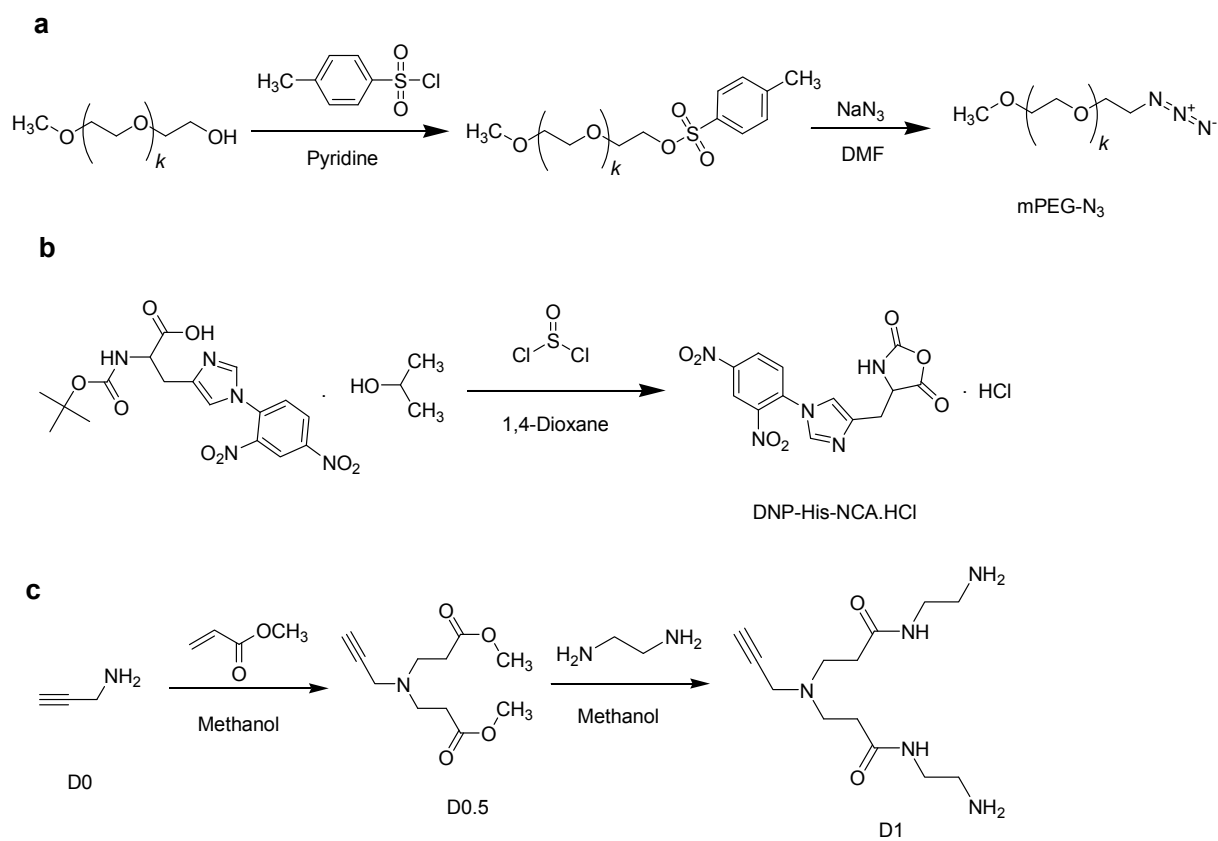
\* Correspondence to:

Professor You Han Bae, Department of Pharmaceutics and Pharmaceutical Chemistry, The University of Utah, 421 Wakara way, Suite 318, Salt Lake City, Utah 84108, USA

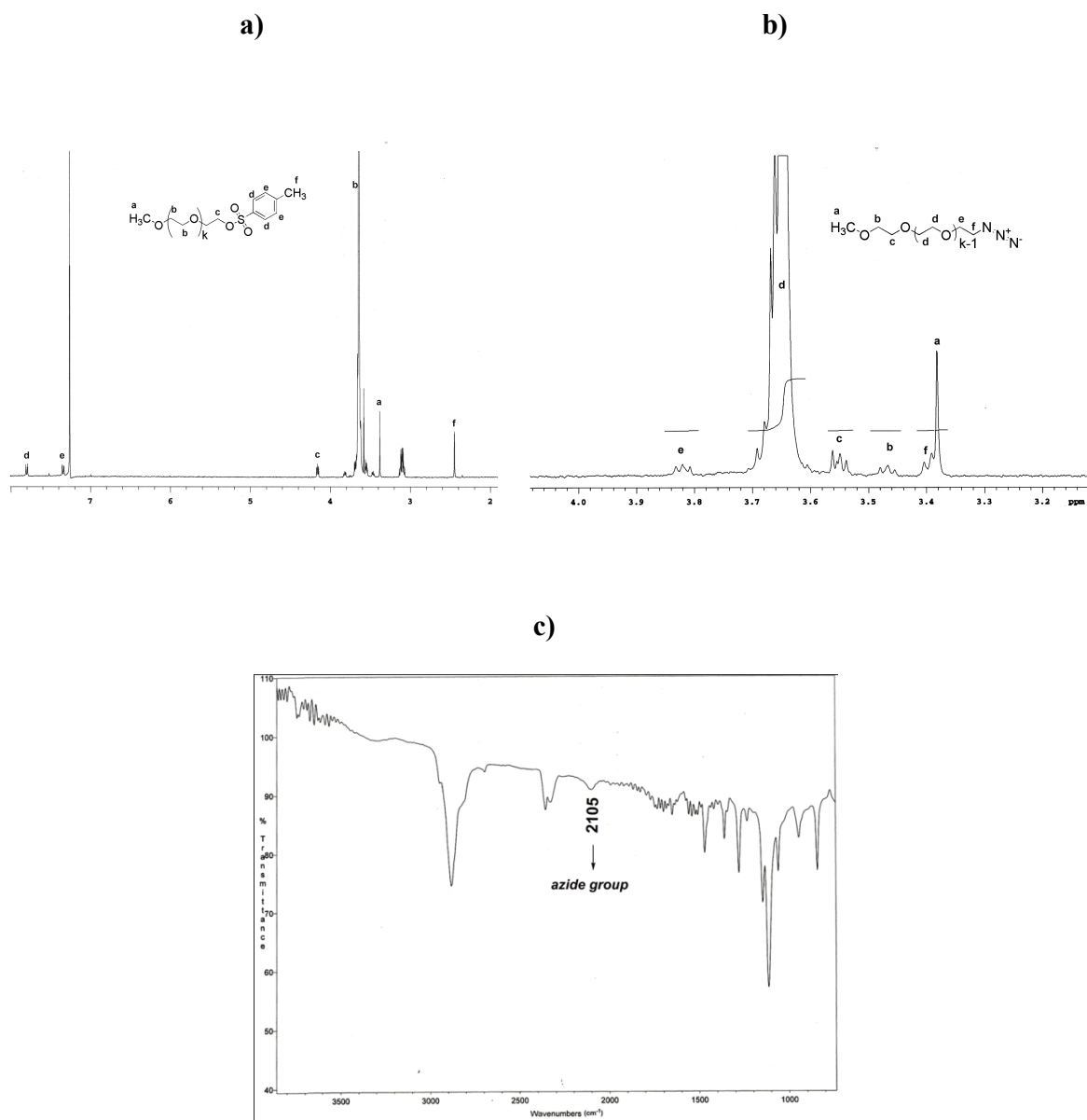
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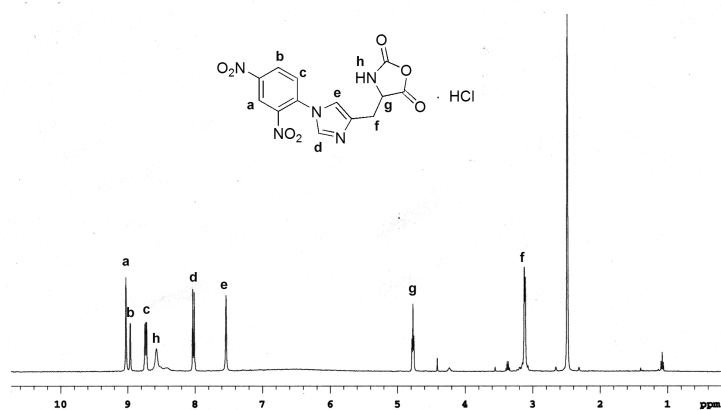
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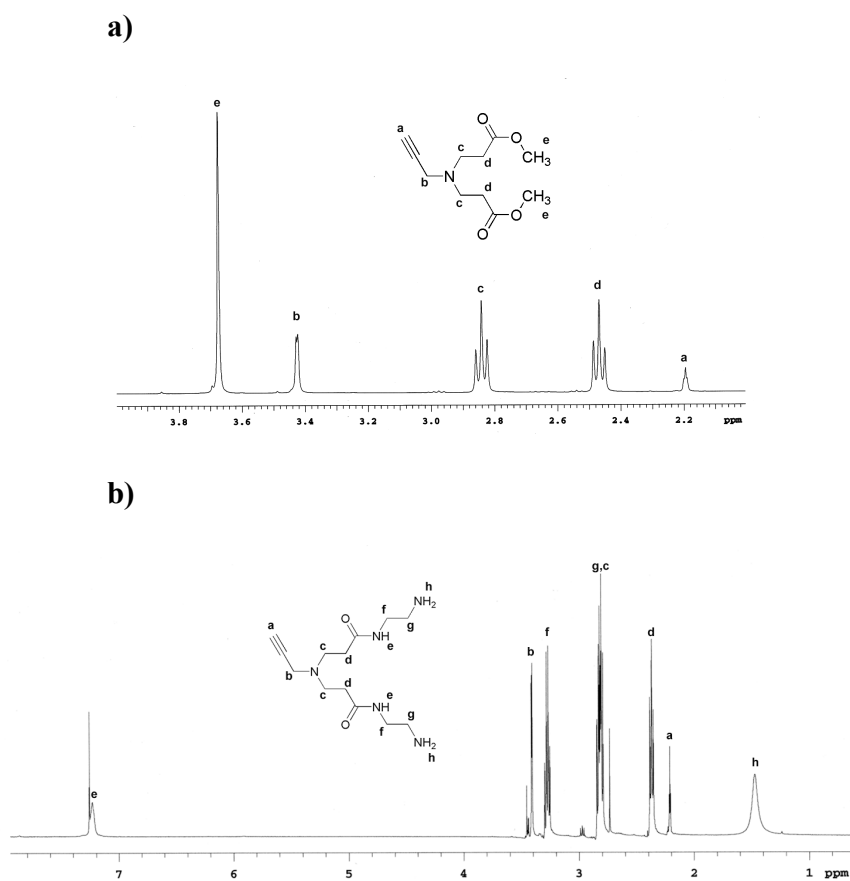
**Fig. S1.** Synthesis of block and core components for the 3-miktoarm block copolymers.



**Fig. S2.**  $^1\text{H-NMR}$  spectra of a) mPEG-TOS and b) mPEG- $\text{N}_3$  in  $\text{CDCl}_3$   
and c) FT-IR spectrum of mPEG- $\text{N}_3$ .

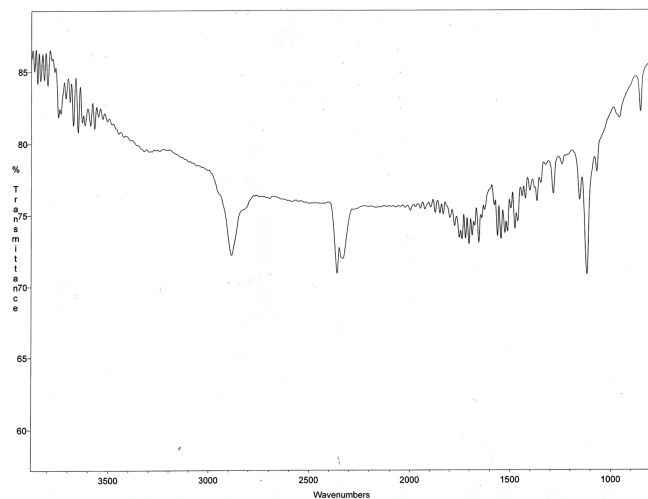


**Fig. S3.** <sup>1</sup>H-NMR spectrum of DNP-His-NCA·HCl in d<sub>6</sub>-DMSO.



**Fig. S4.** <sup>1</sup>H-NMR spectra of a) D0.5 and b) D1 in CDCl<sub>3</sub>.

a)



b)

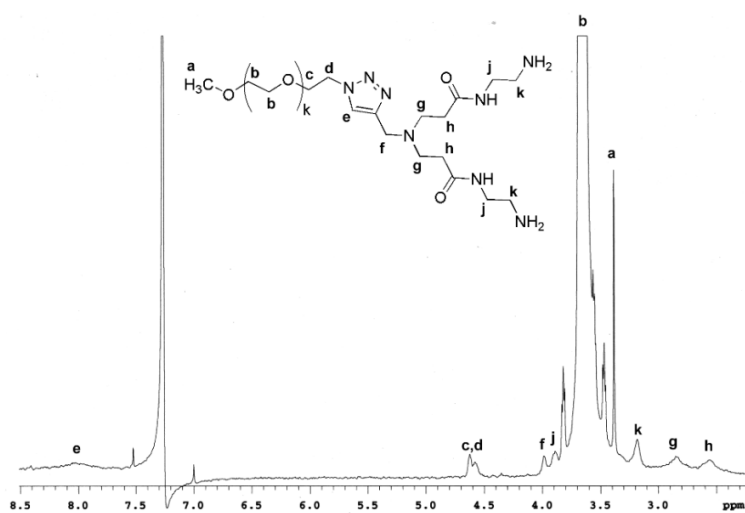
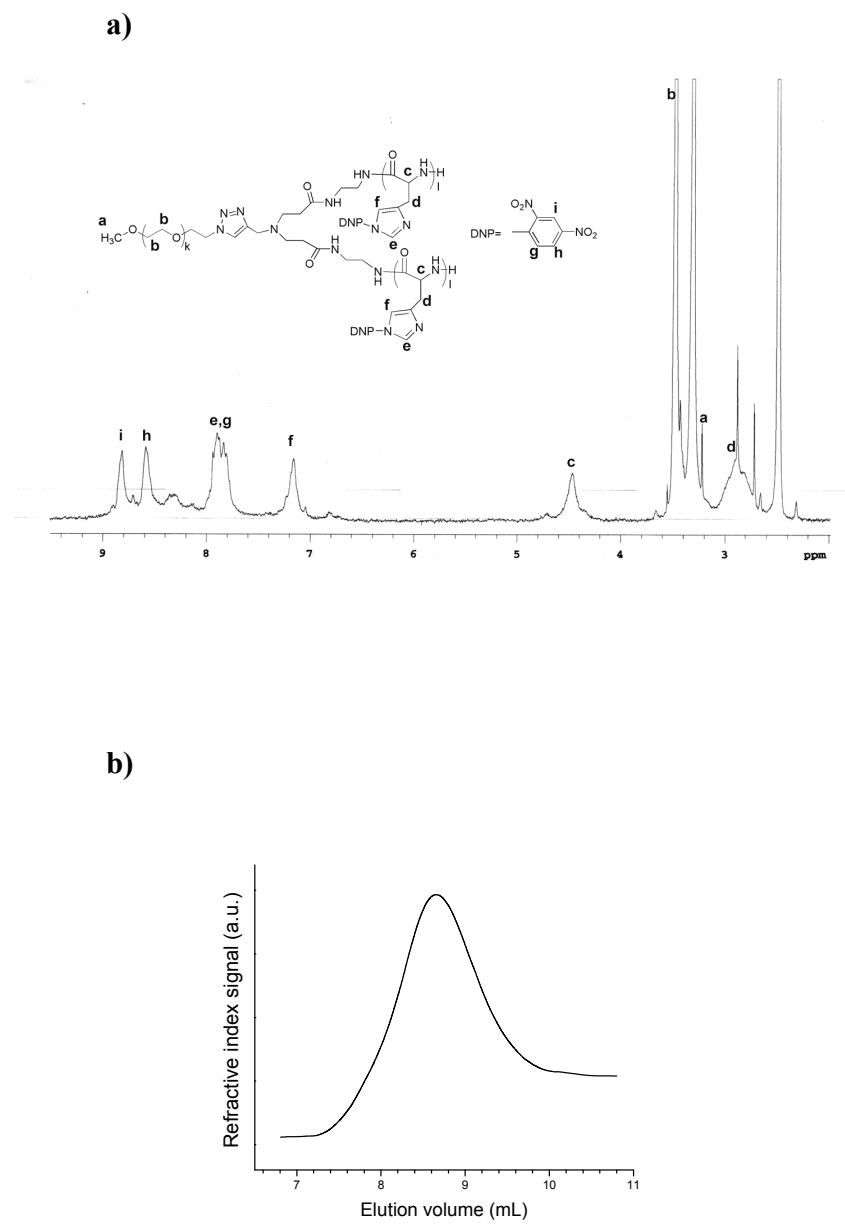
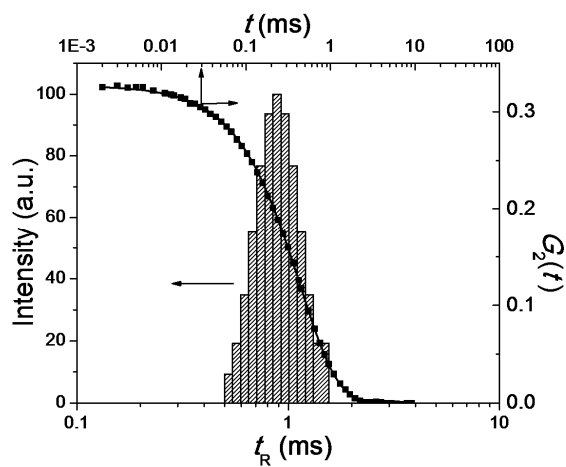


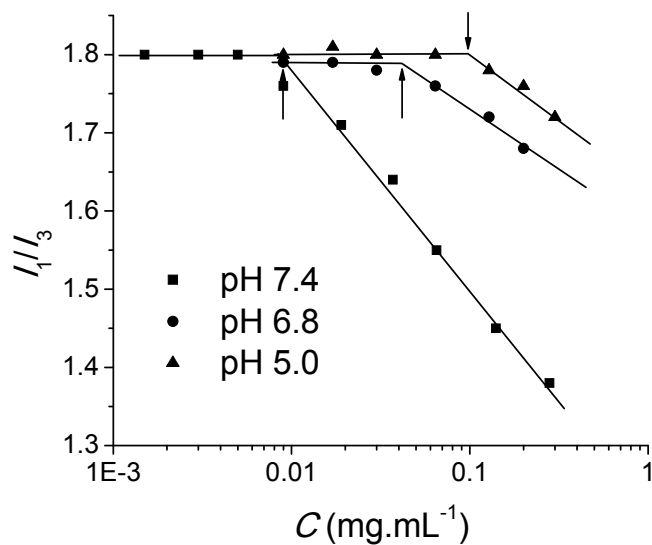
Fig. S5. a) FT-IR spectrum of mPEG-D1 and b) <sup>1</sup>H-NMR spectrum of mPEG-D1 in CDCl<sub>3</sub>.



**Fig. S6.** a)  $^1\text{H}$ -NMR spectrum ( $d_6$ -DMSO) and b) GPC trace of  $m\text{PEG-}b\text{-(poly(DNP-His))}_2$ .



**Fig. S7.** DLS autocorrelation function and normalized time relaxation distribution at  $90^\circ$  for the polymer solution (pH 9.0, 0.3 mg/mL).



**Fig. S8.** The  $I_1/I_3$  value of pyrene fluorescence as a function of polymer concentration at different pHs. Solid lines are guides for the eye. The CAC value was determined as the concentration at the intercept between the two straight lines within the low concentration range indicated by the solid arrows.