Electronic supplementary information (ESI)

Polymerization with Cu(I)-Catalyzed Doyle-Kirmse Reaction of

Bis(allyl sulfides) and Bis(α-diazoesters)

Zihao Fu,^a Qi Zhou,^a Yiyang Xiao,^a Jianbo Wang^{a,b,*}

 ^aBeijing National Laboratory of Molecular Sciences (BNLMS) and Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, College of Chemistry, Peking University, Beijing 100871, China
^bState Key Laboratory of Organometallic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

Email: <u>wangjb@pku.edu.cn</u>

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1) ¹H and ¹³C NMR spectra of the monomers









 $\begin{matrix} 145 & 135 & 125 & 115 & 105 & 95 & 90 & 85 & 80 & 75 & 70 & 65 & 60 & 55 & 50 & 45 & 40 & 35 & 30 & 25 & 20 & 15 & 10 & 5 & 0 \\ f1 (ppm) \end{matrix}$



l45 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -: f1 (ppm)











¹H and ¹³C NMR spectra of 5d

7.1491 7.7488 7.7399 7.339 7.339 7.135 7.155 7.157	-4.297 -4.281 -4.264	1.751 1.718 1.459 1.450 1.450 1.450 1.432	0.000
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¹H and ¹³C NMR spectra of $\boldsymbol{6}$







2) ¹H and ¹³C NMR spectra of the polymer products



¹H and ¹³C NMR spectra of P2









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## ¹H and ¹³C NMR spectra of P7



¹H and ¹³C NMR spectra of P8

245 245 245 245 245 245 245 245 245 245	63 37 37 10 10 10 10 10 10 10 10 10 10 10 10 10	779 165 151	017 017 017 017 017 017 017 017 017 017	195 180	00
CCCCCCCCC000000	5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	444	0,0,0,0,0,0,0,0,0	445	0.0
		$\checkmark$		arphi	











## 3) MALDI-TOF-MS spectrum of P10



Figure S1. MALDI-TOF-MS spectrum of polymer P10.

## 4) HRMS analysis of hydrolysis of P10



Figure S2. HRMS analysis of 8







h) **P8** 













# 6) TGA data of the polymer products









j) **P10** 





b) **P2** a) P1 -0.2 0.0 *T*_g = 87 ℃ *T*_g = 87 ℃ -0.3 Heat Flow (W/g) Heat Flow (W/g) -0.1 -0.4 -0.2 -0.5 -0.3 <del>|</del> 20 -0.6 ò 20 40 60 80 100 120 140 100 120 140 40 60 80 Temperature (°C) Temperature (°C) c) P3 d) P4 -0.1 0.0 *T*_g = 89 ℃ *T*_g = 80 °C -0.1 Heat Flow (W/g) Heat Flow (W/g) -0.2 -0.2 -0.3 -0.3 -0.4 <del>|</del>-0 -0.4 · 20 40 60 100 120 140 80 0 20 40 60 80 100 120 140 Temperature (°C) Temperature (°C) e) P5 f) **P6** 0.0 0.0 -0.1 *T*_g = 92 ℃ *T*_g = 77 ℃ Heat Flow (W/g) Heat Flow (W/g) -0.1 -0.2 -0.2 -0.3 -0.4 + -0.3 20 40 60 140 80 100 120 60 40 100 120 140 80 Temperature (°C) Temperature (°C)

# 7) DSC data of the polymer products













