## Supporting Information for

# Polycondensation of $bis(\alpha$ -diazo-1,3-dicarbonyl) compounds with dicarboxylic

### acids: an efficient access to functionalized alternating polyesters

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#### **EXPERIMENTAL SECTION**

#### Materials

1,4-Cyclohexanedicarboxylic acid (*cis*- and *trans*- mixture) (99%), Adipic acid (AR,  $\geq$ 98%), 2,2-bis(4caoboxyphenyl)hexafluoropropane (98%), itaconic acid (AR, >99.0%), acetylenedicarboxylic acid (98%), 3hydroxyglutaric Acid ( $\geq$ 95.0%), boc-*L*-glutamic acid (98%) were purchased from Tokyo Chemical Industry Co., Ltd. (Shanghai, China). 1,4-Butanediol (AR, 98%), ethyl benzoylacetate (95%), triethylamine (AR, 99.0%), acetonitrile (AR, 99.0%), sodium azide (99%), *p*-toluenesulfonyl chloride(AR, 99%), diketene, ethyl acetoacetate (AR, 98%) were obtained from J&K Scientific (Beijing, China). All the other chemicals were purchased from Xiya-Reagent (Chengdu, China). Compound **5** and **6** were prepared according to the literature methods.<sup>1</sup>

#### Measurements

<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker-400 spectrometer using tetramethysilane as an internal standard, and chloroform-*d* (CDCl<sub>3</sub>) or dimethyl sulfoxide-*d*<sub>6</sub> (DMSO-*d*<sub>6</sub>) as the solvents at 25 °C. Molecular weights and polydispersity were measured using a Waters 515 gel permeation chromatography (GPC) equipped with a Waters 2414 differential refractive index detector and a series of Styragel columns (HR6, HR4, and HR2). Tetrahydrofuran was used as the eluent at a flow rate of 1.0 mL min<sup>-1</sup> at 30 °C, and PMMA standards were used for the calibration. Glass transition temperatures were determined using an EXSTAR DSC6000 DSC (Seiko Instruments Inc.) from -50 to 200 °C at a heating rate of 10 °C min<sup>-1</sup>. Thermal stability was assessed using a TGA Q50 (TA Instruments) system at a heating rate of 10 °C min<sup>-1</sup> under nitrogen atmosphere.

#### **Monomer synthesis**



Scheme S1 Preparation of monomer compounds of 1a and 1b.

Synthesis of monomer compound **1a**. Sodium azide (0.95 g, 30 mmol), 4-toluene sulfonyl chloride (5.72 g, 30 mmol), acetonitrile (40 mL) were placed into a 100 mL round-bottom flask with a magnetic stirrer. The mixture was stirred at room temperature for 1 h. Then the compound **5** (2.58 g, 10 mmol) was added to the mixture. After triethylamine (8.32 mL, 60mmol) was added dropwise, the mixture was stirred at room temperature for 10 h. After the solvent was removed under reduced pressure, the product was purified by chromatography on silica gel with ethyl acetate as eluent to give **1a** as a light yellow soild (2.58 g, yield: 83%). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  4.38–4.23 (m, 2H), 2.48 (s, 3H), 1.80 (p, *J* = 3.3 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  190.0, 161.3, 77.2, 64.6, 28.3, 25.3.

Synthesis of monomer compound **1b**. Sodium azide (0.95 g, 30 mmol), 4-toluene sulfonyl chloride (5.72 g, 30 mmol), acetonitrile (40 mL) were placed into a 100 mL round-bottom flask with a magnetic stirrer. The mixture was stirred at room temperature for 1 h. Then the compound **6** (4.34 g, 10 mmol) was added to the mixture. After triethylamine (8.32 mL, 60mmol) was added dropwise, the mixture was stirred at room temperature for 10 h. After the solvent was removed under reduced pressure, the product was purified by chromatography on silica gel with ethyl acetate as eluent to give **1b** as a light yellow soild (3.48 g, yield: 80%). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.65–7.59 (m, 2H), 7.56–7.49 (m, 1H), 7.42 (dd, *J* = 8.4, 7.0 Hz, 2H), 4.15 (td, *J* = 4.3, 3.6, 1.8 Hz, 2H), 1.56 (h, *J* = 3.1 Hz, 2H).<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  186.7, 161.1, 137.1, 132.3, 128.3, 127.9, 77.2, 64.8, 25.1.

**Model reaction** 



Scheme S2 Preparation of model compounds of 3a'a and 3a'c.

Synthesis of compound **1a'**. Sodium azide (NaN<sub>3</sub>) (1.05 g, 33 mmol), 4-toluene sulfonyl chloride (PTSC) (6.29 g, 33 mmol), acetonitrile (40 mL) were placed into a 100 mL round-bottom flask with a magnetic stirrer. The mixture was stirred at room temperature for 1 h. Then compound **4** (3.90 g, 30 mmol) was added to the mixture. After triethylamine (8.32 mL, 60mmol) was added dropwise, the mixture was stirred at room temperature for 10 h. After the solvent was removed under reduced pressure, the product was purified by chromatography on silica gel with ethyl acetate as eluent to give **1a'** as yellow oil (4.26 g, yield: 91%). <sup>1</sup>H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  4.24 (q, *J* = 7.1 Hz, 2H), 2.38 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  189.7, 161.4, 76.0, 61.6, 28.3, 14.5.

Synthesis of model compound **3a'a**. Compound **1a'** (0.34 g, 2.2 mmol), compound **2a** (cis- and trans- mixture) (0.17 g, 1 mmol), and Rh<sub>2</sub>(OAc)<sub>4</sub> (0.12 g, 0.02 mmol) were placed into a 10 mL Schlenk flask under nitrogen. CH<sub>2</sub>Cl<sub>2</sub> (5 mL) was added into the reaction using a syringe. After refluxed for 4 h at 40 °C, the reaction solution was concentrated by rotary evaporation. The crude product was further purified by silica-gel column chromatography using ethyl acetate as the eluent, affording **3a'a** as white oil (0.36 g, yield: 84%). <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  5.50 (s, 2H), 4.37–4.16 (m, 4H), 2.71 (dp, *J* = 7.3, 3.6 Hz, 2H), 2.35 (s, 6H), 2.04 (dt, *J* = 13.7, 5.5 Hz, 4H), 1.89–1.72 (m, 4H), 1.31 (t, *J* = 7.2 Hz, 6H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 173.4, 164.5, 77.7, 62.5, 40.1, 27.3, 25.7, 14.0.

Synthesis of model compound **3a'c**. Compound **1a'** (0.34 g, 2.2 mmol), compound **2c** (0.13 g, 1 mmol), and Rh<sub>2</sub>(OAc)<sub>4</sub> (0.12 g, 0.02 mmol) were placed into a 10 mL Schlenk flask under nitrogen. CH<sub>2</sub>Cl<sub>2</sub> (5 mL) was added into the reaction using a syringe. After refluxed for 4 h at 40 °C, the reaction solution was concentrated by rotary evaporation. The crude product was further purified by silica-gel column chromatography using ethyl acetate as the eluent, affording **3a'c** as yellow oil (0.32 g, yield: 87%). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  6.46 (s, 1H), 6.12 (d, *J* = 1.1 Hz, 1H), 5.71 (d, *J* = 1.6 Hz, 1H), 5.66 (d, *J* = 2.0 Hz, 1H), 4.19 (qt, *J* = 7.0, 2.2 Hz, 4H), 3.62 (s, 2H), 2.30 (s, 3H), 2.27 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 6H). <sup>13</sup>C NMR (101 MHz, DMSO)  $\delta$  198.5, 169.4, 164.8, 164.5, 132.3, 132.1, 78.6, 62.3, 36.6, 27. 6, 14.2.

### **Polymer synthesis**

As a typical procedure, polymerization of **1a** with **2a** was described as follows. Under nitrogen atmosphere, **1a** (306.1 mg, 1 mmol), **2a** (172.2 mg, 1 mmol), and Rh<sub>2</sub>(OAc)<sub>4</sub> (56 mg, 0.01 mmol) were placed in a 5 mL Schlenk tube with a magnetic stirrer. After CH<sub>2</sub>Cl<sub>2</sub> (1.9 mL) was injected, the mixture was stirred at 40 °C for 6 h. The reaction mixture was added dropwise into 15 mL petroleum ether via a cotton filter to give a yellow solid **3aa** (440.0 mg, yield: 92%) at room temperature.  $M_n$  = 18.4 kg mol<sup>-1</sup>;  $M_w/M_n$  = 1.74. <sup>1</sup>H NMR (500 MHz, Chloroform-*d*)  $\delta$  5.48 (m, 2H, CH), 4.25 (m, 4H, CH<sub>2</sub>), 2.70 (m, 2H, CH), 2.34 (m, 6H, CH<sub>3</sub>), 2.02 (m, 4H, CH<sub>2</sub>), 1.81 (m, 4H, CH<sub>2</sub>), 1.75 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  197.7, 173.4, 164.6, 77.6, 65.6, 40.1, 27.3, 25.7, 24.9.

All other polymers were prepared through the same method.

Polymer **3ab**. Yellow solid; yield 93% (Table 2, entry 2).  $M_n = 16.1 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 2.36$ . <sup>1</sup>H NMR (400 MHz, DMSO $d_6$ )  $\delta$  5.83–5.55 (m, 2H, CH), 4.33–3.92 (m, 4H, CH<sub>2</sub>), 2.50 (m, 4H, CH<sub>2</sub>), 2.28 (m, 6H, CH<sub>3</sub>), 1.89–1.42 (m, 8H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  198.8, 172.0, 165.1, 78.1, 65.6, 33.0, 27.7, 24.9, 24.0.

Polymer **3ac**. Yellow solid; yield 87% (Table 2, entry 3).  $M_n$  = 12.9 kg mol<sup>-1</sup>;  $M_w/M_n$  = 2.53. <sup>1</sup>H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  6.45 (m, 1H, CH), 6.11 (m, 1H, CH), 5.74–5.56 (m, 2H, CH), 4.16 (m, 4H, CH<sub>2</sub>), 3.61 (m, 2H, CH<sub>2</sub>), 2.27 (m, 6H, CH<sub>3</sub>), 1.64 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  198.4, 169.2, 164.8, 164.5, 132.3, 132.0, 78.6, 65.6, 36.5, 27.5, 24.8.

Polymer **3ad**. Yellow solid; yield 91% (Table 2, entry 4).  $M_n$  = 8.8 kg mol<sup>-1</sup>;  $M_w/M_n$  = 1.35. <sup>1</sup>H NMR (500 MHz, Chloroform-*d*)  $\delta$  5.61 (m, 2H, CH), 4.29 (m, 4H, CH<sub>2</sub>), 2.40 (m, 6H, CH<sub>3</sub>), 1.79 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  195.3, 163.0, 149.7, 79.0, 75.2, 66.2, 27.2, 24.9.

Polymer **3ae**. Yellow solid; yield 90% (Table 2, entry 5).  $M_n = 16.4$  kg mol<sup>-1</sup>;  $M_w/M_n = 1.95$ . <sup>1</sup>H NMR (500 MHz, Chloroform-*d*)  $\delta$  8.14 (m, 4H, CH), 7.51 (m, 4H, CH), 5.72 (m, 2H, CH), 4.30 (m, 4H, CH<sub>2</sub>), 2.42 (m, 6H, CH<sub>3</sub>), 1.79 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  197.2, 164.3, 164.1, 138.5, 130.5, 130.0, 129.4, 127.1, 124.8, 122.5, 120.2, 78.3, 65.8, 64.8, 27.4, 25.0.

Polymer **3af**. Yellow solid; yield 94% (Table 2, entry 6).  $M_n = 7.1 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 2.01$ . <sup>1</sup>H NMR (500 MHz, Chloroform-*d*)  $\delta$  5.67–5.41 (m, 2H, CH), 4.61 (m, 1H, CH), 4.26 (m, 4H, CH<sub>2</sub>), 2.80 (m, 2H, CH<sub>2</sub>), 2.45–2.29 (m, 6H, CH<sub>3</sub>), 1.94–1.65 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  197.6, 169.9, 164.6, 77.9, 65.9, 64.8, 40.6, 27.3, 24.9.

Polymer **3ag**. Yellow solid; yield 90% (Table 2, entry 7).  $M_n = 7.0 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 1.35$ . <sup>1</sup>H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  7.48 (m, 1H, NH), 5.84–5.55 (m, 2H, CH), 4.16 (m, 6H, , CH/CH<sub>2</sub>), 2.62 (m, 2H, CH<sub>2</sub>), 2.34–2.19 (m, 6H, CH<sub>3</sub>), 2.17–1.98 (m, 1H, CH), 1.94–1.75 (m, 1H, CH), 1.76–1.53 (m, 4H, CH<sub>2</sub>), 1.37 (m, 9H, CH<sub>3</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  198.2, 171.1, 164.6, 164.4, 155.5, 78.6, 77.7, 65.2, 52.4, 29.3, 28.1, 27.2, 25.6, 24.4.

Polymer **3ba**. Yellow solid; yield 94% (Table 2, entry 8).  $M_n = 26.7$  kg mol<sup>-1</sup>;  $M_w/M_n = 1.86.^{1}$ H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  7.96 (m, 4H, CH), 7.64 (m, 2H, CH), 7.50 (m, 4H, CH), 6.58 (m, 2H, CH), 3.96 (m, 4H, CH<sub>2</sub>), 2.62 (m, 2H, CH), 1.87–1.68 (m, 4H, CH<sub>2</sub>), 1.71–1.52 (m, 4H, CH<sub>2</sub>), 1.28 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  191.2, 173.5, 165.3, 134.8, 134.6, 129.5, 129.3, 74.6, 65.4, 39.6, 25.6, 24.6.

Polymer **3bb**. Yellow solid; yield 92% (Table 2, entry 9).  $M_n = 26.2 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 2.87.^{1}\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  7.95 (m, 4H, CH), 7.64 (m, 2H, CH), 7.50 (m, 4H, CH), 6.57 (m, 2H, CH), 3.96 (m, 4H, CH<sub>2</sub>), 2.41 (m, 4H, CH<sub>2</sub>), 1.53 (m, 4H, CH<sub>2</sub>), 1.28 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  191.1, 172.0, 165.3, 134.8, 134.6, 129.5, 129.3, 74.7, 65.5, 33.0, 24.6, 24.0.

Polymer **3bc**. Yellow solid; yield 89% (Table 2, entry 10).  $M_n$  = 15.7 kg mol<sup>-1</sup>;  $M_w/M_n$  = 2.60. <sup>1</sup>H NMR (400 MHz, DMSOd<sub>6</sub>)  $\delta$  8.14–7.90 (m, 4H, CH), 7.76–7.60 (m, 2H, CH), 7.52 (m, 4H, CH), 6.66 (m, 1H, CH), 6.56 (m, 1H, CH), 6.36 (m, 1H, CH), 6.08 (m, 1H, CH), 4.06 – 3.87 (m, 4H, CH<sub>2</sub>), 3.59 (m, 2H, CH<sub>2</sub>), 1.28 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  189.5, 168.2, 163.8, 163.3, 133.7, 133.4, 131.1, 130.9, 128.4, 128.2, 73.9, 73.7, 64.4, 35.3, 23.4.

Polymer **3bd**. Yellow solid; yield 82% (Table 2, entry 11).  $M_n = 15.2 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 1.50$ . <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (m, 4H, CH), 7.63 (m, 2H, CH), 7.51 (m, 4H, CH), 6.38 (m, 2H, CH), 4.10 (m, 7H, CH<sub>2</sub>), 1.45 (m, 5H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  187.5, 163.5, 149.8, 134.8, 133.6, 129.2, 129.0, 75.7, 75.3, 66.0, 24.6.

Polymer **3be**. Yellow solid; yield 95% (Table 2, entry 12).  $M_n = 14.2 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 2.45$ . <sup>1</sup>H NMR (400 MHz, DMSO $d_6$ )  $\delta$  8.25–7.98 (m, 8H, CH), 7.69 (m, 2H, CH), 7.56 (m, 8H, CH), 6.94 (m, 2H, CH), 3.99 (m, 4H, CH<sub>2</sub>), 1.59–1.21 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, DMSO)  $\delta$  190.3, 164.6, 163.6, 137.3, 134.4, 134.1, 130.5, 129.9, 129.1, 128.9, 128.3, 124.6, 124.2, 122.3, 120.1, 75.1, 65.2, 64.0, 24.2.

Polymer **3bf**. Yellow solid; yield 79% (Table 2, entry 13).  $M_n = 5.4$  kg mol<sup>-1</sup>;  $M_w/M_n = 2.35$ . <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.00 (m, 4H, CH), 7.59 (m, 2H, CH), 7.47 (m, 5H, CH), 6.34 (m, 1H, CH), 4.59 (m, 1H, CH), 4.07 (m, 5H, CH<sub>2</sub>), 2.89 (m, J = 105.1 Hz, 2H, CH<sub>2</sub>), 1.68–1.30 (m, 5H, CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  188.4, 168.8, 164.1, 133.4, 133.0, 128.2, 128.0, 73.5, 64.7, 63.8, 39.7, 23.7.

Polymer **3bg**. Yellow solid; yield 73% (Table 2, entry 14).  $M_n = 6.0 \text{ kg mol}^{-1}$ ;  $M_w/M_n = 1.87.^{1}\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  8.00 (m, 4H, CH), 7.67 (m, 2H, CH), 7.60–7.42 (m, 4H, CH), 7.08 (m, NH) , 6.78–6.59 (m, 2H, CH), 4.21 (m, CH), 3.98 (m, 4H, CH<sub>2</sub>), 2.80–2.55 (m, 2H, CH<sub>2</sub>), 2.05 (m, 1H, CH), 1.95–1.76 (m, 1H, CH), 1.52–1.10 (m, 13H, CH<sub>3</sub>/CH<sub>2</sub>). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  188.0, 170.3, 164.0, 163.4, 154.3, 133.3, 133.0, 128.2, 127.8, 79.2, 73.3, 64.6, 51.7, 28.7, 27.2, 26.0, 23.6.

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Entry	Cat. (mol%)	Cont. (wt%)	Temp. (°C)	Yield (%) <sup>a</sup>	M <sub>n</sub> <sup>b</sup>	PDI <sup>b</sup>
1	1	10	40	84	11.2	1.65
2	0.5	10	40	57	12.1	1.41
3	2	10	40	81	10.9	1.67
4	1	5	40	78	12.4	1.39
5	1	20	40	92	18.4	1.74
6	1	30	40	77	10.5	1.52
7	1	20	25	54	4.6	1.57
8	1	20	60	89	16.4	1.82
9 <sup>c</sup>	1	20	40	82	9.4	1.69
10 <sup>d</sup>	1	20	40	87	14.4	1.98

TableS1Optimizationofpolymerizationconditions.

<sup>*a*</sup> Isolated yield. <sup>*b*</sup> *M<sub>n</sub>*: number average molecular weights (kg mol<sup>-1</sup>); PDI: polydispersity; determined by GPC. <sup>*c*</sup> In toluene. <sup>*d*</sup> In 1,2-dichloroethane.

#### **THERMAL PROPERTIES**





Fig. S2 Thermogravimetric analysis (TGA) of (A) 3aa-3ag and (B) 3ba-3bg.



Fig. S3 Gel permeation chromatography(GPC) of respective polymers of (A) 3aa-3ad and (B) 3ba-3bd.

<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra



Fig. S4 <sup>1</sup>H NMR spectrum for 1b.





Fig. S6 <sup>1</sup>H NMR spectrum for 1a'.





Fig. S8 <sup>1</sup>H NMR spectrum for **3ab**.



**Fig. S10** <sup>1</sup>H NMR spectrum for **3ad**.





8.0

7.5

7.0

6.5

6.0

5.5

5.0 4.5 f1 (ppm)

4.0

3.5

3.0

2.5

2.0

1.5



Fig. S14 <sup>1</sup>H NMR spectrum for 3af.



**Fig. S16** <sup>1</sup>H NMR spectrum for **3ag**.

















**Fig. S24** <sup>1</sup>H NMR spectrum for **3bd**.













Fig. S28 <sup>1</sup>H NMR spectrum for 3bf.



Fig. S30 <sup>1</sup>H NMR spectrum for 3bg.



#### Reference

1. (a) H. Xue, Y. Zhao, H. Wu, Z. Wang, B. Yang, Y. Wei, Z. Wang and L. Tao, *J. Am. Chem. Soc.*, 2016, *138*, 8690-8693; (b) N. S. Nandurkar, M. J. Bhanushali, D. S. Patil and B. M. Bhanage, *Synthetic Commun.*, 2007, *37*, 4111-4115.