Supporting Information for

Three-Component Polycondensation of Bis(diazoketone) with Dicarboxylic Acids and Cyclic

Ethers: Synthesis of New Type of Polyesteretherketones

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Figure S2. ¹³C NMR spectrum for **6**.

¹H NMR (400 MHz, CDCl₃) δ 7.98-7.93 (m, 4H, *o*- or *m*-Ph[-*H*], *o*- or *m*-Ph[-*H*]-*t*Bu), 7.57 (t, *J* = 7.2 Hz, 1H, *p*-Ph[-*H*]), 7.46 (m, 4H, *o*- or *m*-Ph[-*H*], *o*- or *m*-Ph[-*H*]-*t*Bu), 4.75 (s, 2H, -O-CH₂-[C=O]-), 4.34 (t, *J* = 6.0 Hz, 2H, -[C=O]-O-CH₂-), 3.65 (t, *J* = 6.4 Hz, 2H, -CH₂-O-CH₂-[C=O]-), 1.89-1.82 (m, 4H, -O-CH₂-[CH₂]₂-CH₂-O-), 1.33 (s, 9H, -C[CH₃]₃).

¹³C NMR(100MHz, CDCl₃) δ 196.6 (-O-CH₂-[*C*=O]-), 166.6 (-[*C*=O]-O-), 156.5 (Ph), 135.0 (Ph), 133.5 (Ph), 129.4 (Ph), 128.7 (Ph), 127.9 (Ph), 127.6 (Ph), 125.3 (Ph), 73.8 (-O-CH₂-), 71.3 (-O-CH₂-), 64.5 (-O-CH₂-), 35.1 (-CMe₃), 31.1 (-C[CH₃]₃), 26.3(-O-CH₂-CH₂-), 25.5 (-O-CH₂-CH₂-).

Anal Calcd for C₂₃H₂₈O₄·0.4H₂O :C, 73.53; H, 7.51. Found: C, 73.23; H, 7.17.

2. Compound 7.



Figure S4. ¹³C NMR spectrum for **7**.

¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 8.4 Hz, 2H, *o*- or *m*-Ph[-*H*] or *o*- or *m*-Ph[-*H*]-*t*Bu), 7.96 (d, *J* = 7.6 Hz, 2H, *o*- or *m*-Ph[-*H*] or *o*- or *m*-Ph[-*H*]-*t*Bu), 7.61 (t, *J* = 6.4 Hz, 1H, *p*-Ph[-*H*]), 7.52-7.48 (m, 4H, *o*- or *m*-Ph[-*H*], *o*- or *m*-Ph[-*H*]-*t*Bu), 5.56 (s, 2H, -O-CH₂-[C=O]-), 1.35 (s, 9H, -C[CH₃]₃).

¹³C NMR (100MHz, CDCl₃) δ 192.3 (-O-CH₂-[*C*=O]-), 166.1 (-[*C*=O]-O-), 157.1 (Ph), 134.4 (Ph), 133.9 (Ph), 129.9 (Ph), 128.9 (Ph), 127.9 (Ph), 126.6 (Ph), 125.5 (Ph), 66.4 (-O-CH₂-), 35.2 (-*C*Me₃), 31.1 (-C[*C*H₃]₃).

Anal Calcd for C₁₉H₂₀O₃: 77.00; H, 6.80. Found: C, 76.70; H, 7.00.









¹H NMR (400 MHz, CDCl₃) δ 7.98-7.93 (m, 4H, *o*- or *m*-Ph[-*H*], *o*- or *m*-Ph[-*H*]-*t*Bu), 7.57 (t, *J* = 6.8 Hz, 1H, *p*-Ph[-*H*]), 7.46 (t, *J* = 6.8 Hz, 4H, *o*- or *m*-Ph[-*H*], *o*- or *m*-Ph[-*H*]-*t*Bu), 4.73 (s, 2H, -O-CH₂-[C=O]-), 4.31 (t, *J* = 6.4 Hz, 2H, -[C=O]-O-CH₂-), 3.60 (t, *J* = 6.8 Hz, 2H, -CH₂-O-CH₂-[C=O]-), 1.82-1.71 (m, 4H, -O-CH₂-CH₂-CH₂-CH₂-CH₂-O), 1.51 (m, 2H, -O-[CH₂]₂-CH₂-[CH₂]₂-O-), 1.33 (s, 9H, -C[CH₃]₃).

¹³C NMR (100MHz, CDCl₃) δ 196.7 (-O-CH₂-[*C*=O-]-), 166.7 (-[*C*=O]-O-), 156.5 (Ph), 135.0 (Ph), 133.5 (Ph), 129.4 (Ph), 128.7 (Ph), 128.0 (Ph), 127.7 (Ph), 125.3 (Ph), 73.9 (-O- CH_2 -), 71.6 (-O- CH_2 -), 64.7 (-O- CH_2 -), 35.1 (- CMe_3), 31.2 (- $C[CH_3]_3$), 29.3 (-O- CH_2 - CH_2 -), 28.6 (-O- CH_2 - CH_2 -), 22.7 (-O- CH_2 - CH_2 - CH_2 - CH_2 - CH_2 -O-).

Anal Calcd for C₂₄H₃₀O₄: C, 75.36; H, 7.91. Found: C, 74.92; H, 7.93.

4. Polymer 3a.



Figure S7. ¹H NMR spectrum for **3a**.

¹H NMR (400 MHz, CDCl₃) δ 8.07 (s, -O-[C=O]-Ph[-*H*]-), 7.85 (d, *J* = 7.2 Hz, -Ph[-*H*]-SiMe₂-), 7.56 (d, *J* = 7.2 Hz, -Ph[-*H*]-SiMe₂-), 5.57 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.71 (s, -CH₂-O-CH₂-[C=O]- [THF incorporated unit]), 4.38 (t, *J* = 5.6 Hz, -[C=O]-O-CH₂-), 3.64 (t, *J* = 5.6 Hz, -CH₂-O-CH₂-[C=O]-), 1.91 (m, -O-CH₂-CH₂-), 1.81 (m, -O-CH₂-CH₂-), 0.64 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.25 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

5. Polymer 3b.



Figure S8. ¹H NMR spectrum for **3b**.

¹H NMR (400 MHz, CDCl₃) δ 8.80 (m, -naphthalene[-*H*]-), 8.10 (s, -naphthalene[-*H*]-), 7.84 (d, *J* = 7.6 Hz, -Ph[-*H*]-SiMe₂-), 7.60 (m, -naphthalene[-*H*]-), 7.55 (d, *J* = 7.2 Hz, -Ph[-*H*]-SiMe₂-), 5.65 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.70 (s, -CH₂-O-CH₂-[C=O]- [THF incorporated unit]), 4.47 (t, *J* = 6.4 Hz, -[C=O]-O-CH₂-), 3.66 (t, *J* = 5.6 Hz, -CH₂-O-CH₂-[C=O]-), 1.96 (m, -O-CH₂-CH₂-CH₂-), 1.84 (m, -O-CH₂-CH₂-), 0.64(s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.24 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

6. Polymer **3c**.



Figure S9. ¹H NMR spectrum for **3c**.

¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, J = 8.0 Hz, -Ph[-H]-Ph- or -Ph[-H]-SiMe₂-), 7.85 (d, J = 7.6 Hz, -Ph[-H]-Ph- or -Ph[-H]-SiMe₂-), 7.66 (d, J = 8.0 Hz, -Ph[-H]-Ph- or -Ph[-H]-SiMe₂-), 7.55 (d, J = 7.2 Hz, -Ph[-H]-Ph- or -Ph[-H]-SiMe₂-), 5.57 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.71 (s, -CH₂-O-CH₂-[C=O]- [THF incorporated unit]), 4.38 (t, J = 5.2 Hz, -[C=O]-O-CH₂-), 3.65 (t, J = 6.0 Hz, -CH₂-O-CH₂-[C=O]-), 1.91 (m, -[C=O]-O-CH₂-CH₂-CH₂-), 1.83 (m, -[C=O]-O-CH₂-CH₂-CH₂-), 0.64 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.25 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

7. Polymer 3d.



Figure S10. ¹H NMR spectrum for **3d**.

¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, J = 7.6 Hz, -Ph[-H]-SiMe₂-), 7.85 (d, J = 7.6 Hz, -Ph[-H]-SiMe₂-), 7.55 (m, -Ph[-H]-SiMe₂-), 5.53 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.69 (s, -CH₂-O-CH₂-[C=O]- [THF incorporated unit]), 4.35 (t, J = 6.0 Hz, -[C=O]-O-CH₂-), 3.63 (t, J = 6.0 Hz, -CH₂-O-CH₂-[C=O]-), 1.91 (m, -O-CH₂-CH₂-), 1.78 (m, -O-CH₂-CH₂-), 0.65 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.58 (s, -Ph-Si[CH₃]₂-Ph-), 0.25 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

8. Polymer 3e.





¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 6.8 Hz, -Ph[-*H*]-), 7.56 (d, *J* = 7.2 Hz, -Ph[-*H*]-), 5.30 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.69 (s, -CH₂-O-CH₂-[C=O]- [THP incorporated unit]), 4.10 (s, -[C=O]-O-CH₂-), 3.59 (s, -CH₂-O-CH₂-[C=O]-), 2.30 (s, -CH₂-[C=O]-O-), 1.6-1.8 (-CH₂-CH₂-CH₂-CH₂-), 0.65 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.26 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

9. Polymer **3f**.



Figure S12. ¹H NMR spectrum for **3f**.

¹H NMR(400 MHz, CDCl₃) δ 7.85 (d, J = 7.2 Hz, -Ph[-*H*]-), 7.54 (d, J = 7.6 Hz, -Ph[-*H*]-), 5.32 (s, -[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.68 (s, -CH₂-O-CH₂-[C=O]- [THF incorporated unit]), 4.12 (t, J = 5.6 Hz, -[C=O]-O-CH₂-), 3.59 (t, J = 5.2 Hz, -CH₂-O-CH₂-[C=O]-), 2.60 (s, -CH₂-CH₂-[C=O]-O-), 1.73 (s, -O-CH₂-[CH₂]₂-CH₂-O-), 0.65 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.26 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).

10. Polymer 8a.



Figure S13. ¹H NMR spectrum for **8a**.

¹H NMR (400 MHz, CDCl₃) δ 8.25-7.48 (-Ph[-*H*]-), 5.57 (s, -[C=O]-C*H*₂-O-[C=O]- [normal insertion unit]), 4.69 (s, -CH₂-O-C*H*₂-[C=O]- [THP incorporated unit]), 4.35 (t, *J* = 5.2 Hz, -[C=O]-O-C*H*₂-CH₂-), 3.60 (t, *J* = 4.4 Hz, -C*H*₂-O-CH₂-[C=O]-), 1.82 (m, -O-CH₂-C*H*₂-), 1.74 (m, -O-CH₂-C*H*₂-), 1.56 (m, -O-[CH₂]₂-C*H*₂-[CH₂]₂-O-), 0.66 (s, -SiMe₂-[C*H*₂]₂-SiMe₂-), 0.28 (s, -Si[C*H*₃]₂-[CH₂]₂-Si[C*H*₃]₂-).

11. Polymer 8b.



Figure S14. ¹H NMR spectrum for **8b**.

¹H NMR(400 MHz, CDCl₃) δ 8.98-7.48 (-Ph[-*H*]-, naphthalene[-*H*]-), 5.65 (s, -[C=O]-C*H*₂-O-[C=O]- [normal insertion unit]), 4.69 (s, -CH₂-O-C*H*₂-[C=O]- [THP incorporated unit]), 4.43 (t, *J* = 4.4 Hz, -[C=O]-O-C*H*₂-CH₂-), 3.61 (s, -C*H*₂-O-CH₂-[C=O]-), 1.86 (m, -O-CH₂-C*H*₂-), 1.74 (m, -O-CH₂-C*H*₂-), 1.59 (m, -O-[CH₂]₂-C*H*₂-[CH₂]₂-O-), 0.66 (m, -SiMe₂-[C*H*₂]₂-SiMe₂-), 0.29 (m, -Si[C*H*₃]₂-[CH₂]₂-Si[C*H*₃]₂-).

12. Polymer 8d.



Figure S15. ¹H NMR spectrum for 8d.

¹H NMR(400 MHz, CDCl₃) δ 8.2-7.5 (-Ph[-*H*]-), 5.53 (s, -Ph-[C=O]-CH₂-O-[C=O]-Ph- [normal insertion unit]), 4.68 (s, -CH₂-O-CH₂-[C=O]-Ph- [THP incorporated unit]), 4.31 (t, *J* = 5.6 Hz, -Ph-[C=O]-O-CH₂-CH₂-), 3.59 (t, *J* = 6.4 Hz, -CH₂-O-CH₂-[C=O]-Ph-), 1.85-1.77 (-O-CH₂-CH₂-), 1.77-1.69 (-O-CH₂-CH₂-), 1.54 (m, -O-CH₂-CH₂-CH₂-CH₂-CH₂-O), 0.65 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.59 (m, -Ph-Si[CH₂]₂-Ph-), 0.27(s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).



Figure S16. ¹H NMR spectrum for **8e**.

¹H NMR (400 MHz, CDCl₃) δ 7.9-7.5 (-Ph[-*H*]-), 5.31 (s, -Ph-[C=O]-C*H*₂-O-[C=O]- [normal insertion unit]), 4.68 (s, -CH₂-O-C*H*₂-[C=O]- [THP incorporated unit]), 4.07 (t, *J* = 4.8 Hz, -[C=O]-O-C*H*₂-CH₂-), 3.57 (t, *J* = 5.6 Hz, -C*H*₂-O-CH₂-[C=O]-Ph-), 2.51-2.35 (-O-[C=O]-C*H*₂-), 1.8-1.4 (-[C=O]-O-CH₂-C*H*₂-C*H*₂-C*H*₂-C*H*₂-O-, -O-[C=O]-C*H*₂-[C*H*₂]₂-C*H*₂-[C=O]-O-), 0.65 (s, -SiMe₂-[C*H*₂]₂-SiMe₂-), 0.26 (s, -Si[C*H*₃]₂-[C*H*₂]₂-Si[C*H*₃]₂-).



Figure S17. ¹H NMR spectrum for 8f.

¹H NMR (400 MHz, CDCl₃) δ 7.9-7.5 (-Ph-[*H*]-), 5.33 (-[C=O]-CH₂-O-[C=O]- [normal insertion unit]), 4.68 (s, -CH₂-O-CH₂-[C=O]- [THP incorporated unit]), 4.10 (m, -[C=O]-O-CH₂-CH₂-), 3.57 (t, *J* = 6.0 Hz, -CH₂-O-CH₂-[C=O]-), 2.93-2.58(-O-[C=O]-[CH₂]₂-[C=O]-O-), 1.66 (m, -O-CH₂-CH₂-CH₂-CH₂-O-), 1.45 (m, -O-[CH₂]₂-CH₂-[CH₂]₂-O-), 0.64 (s, -SiMe₂-[CH₂]₂-SiMe₂-), 0.26 (s, -Si[CH₃]₂-[CH₂]₂-Si[CH₃]₂-).