Supporting Information for "BisTEGylated poly(*p*-benzamide)s: Combining organo-solubility with shape-persistence"

Synthesis and characterization of polymers Gel permeation chromatography of polymers



Figure S1. GPC elugram of polymer 13b.



Figure S2. GPC elugram of polymerization of monomer 14a in diglyme (*red*) and THF (*blue*).



Figure S3. GPC elugram of protected (*red*) and unprotected (*blue*) polymer 14b.

Bis-TEGylated poly(*p*-benzamide) (13a)



Figure S4. Entire GPC elugram of polymer **13a**, showing no peak at short elution time which would indicate aggregation (toluene at 23.8 min.).



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Figure S6. ¹H NMR spectra (300 MHz) of monomer 9a (green) and polymer 13a (blue) in CDCl₃.



58.90

Figure S8. APT spectrum (100 MHz) of 13a in CDCl₃.



Figure S10. ¹³C NMR spectrum (75 MHz) of 14b in CDCl₃.

N-protected, mono-TEGylated poly(*p*-benzamide) (14b)



Figure S11. MALDI-ToF mass spectrum of 14b.



Figure S12. UV-vis measurement at 25°C of a chloroform solution (10 mg/mL) of polymer **13a** before and after filtration. The solutions were diluted to c = 0.05 mg/mL before the measurement. *Red*: unfiltered, *blue*: filtered through a PTFE filter (0.2 µm).

UV-vis measurements



Figure S13. Hydrodynamic radii of polymer **13a** after 0h (*red*) and 12h (*blue*), calculated by the CONTIN method.





Figure S14. ¹H NMR spectrum (300 MHz) of 3 in DMSO- d_6 .



Figure S16. APT spectrum (75 MHz) of 3 in DMSO-d₆.



Figure S17. High resolution-mass spectrum of 3 (ESI-).



Figure S18. RP-HPLC elugram of 3.

Methyl-2-hydroxy-4-nitrobenzoate (4)





Figure S20. ¹³C NMR spectrum (75 MHz) of 4 in CDCl₃.







Figure S22. High resolution-mass spectrum of 4 (ESI-).



Figure S23. RP-HPLC elugram of 4.



Figure S24. ¹H NMR spectrum (300 MHz) of 5 in CDCl₃.



Figure S25. ¹³C NMR spectrum (75 MHz) of 5 in CDCl₃.





Figure S26. ¹H NMR spectrum (360 MHz) of **6a** in CDCl₃ after purification *via* column chromatography.



Figure S28. DEPT spectrum (90 MHz) of **6a** in CDCl₃ after purification *via* column chromatography.



Figure S29. High resolution-mass spectrum of **6a** (ESI+) after purification *via* column chromatography.



Figure S30. RP-HPLC elugram of **6a** after purification *via* column chromatography.







Figure S34. High resolution-mass spectrum of **6b** (ESI+) after purification *via* column chromatography.



Figure S35. RP-HPLC elugram of **6b** after purification *via* column chromatography.

2,5-Bis(2-(2-(2-methoxy)ethoxy)ethoxy)-4-nitrobenzoic acid (7a)



Figure S36. ¹H NMR spectrum (360 MHz) of 7a in CDCl₃.



Figure S38. DEPT spectrum (360 MHz) of 7a in CDCl₃.



Figure S39. High resolution-mass spectrum of 7a (ESI+).



Figure S40. RP-HPLC elugram of 7a.





Figure S41. ¹H NMR spectrum (300 MHz) of 7b in CDCl₃.



Figure S42. ¹³C NMR spectrum (75 MHz) of 7b in CDCl₃.



Figure S44. High resolution-mass spectrum of 7b (ESI+).



Figure S45. RP-HPLC elugram of 7b.





Figure S46. ¹H NMR spectrum (360 MHz) of 8a in CDCl₃.



Figure S48. APT spectrum (90 MHz) of 8a in CDCl₃.



Figure S49. High resolution-mass spectrum of 8a (ESI+).



Figure S50. RP-HPLC elugram of crude 8a.





Figure S52. ¹³C NMR spectrum (75 MHz) of 8b in CDCl₃.



Figure S53. APT NMR spectrum (75 MHz) of 8b in CDCl₃.



Figure S54. High resolution-mass spectrum of 8a (ESI+).

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Figure S55. RP-HPLC elugram of crude 8b.





Figure S56. ¹H NMR spectrum (300 MHz) of **9a** in CDCl₃ after purification *via* recycling HPLC.



Figure S57. ¹³C NMR spectrum (75 MHz) of **9a** in CDCl₃ after purification *via* recycling HPLC.



Figure S58. DEPT spectrum (75 MHz) of 9a in CDCl₃ after purification via recycling HPLC.



Figure S59. High resolution-mass spectrum of 9a (ESI+) after purification via recycling HPLC.



Figure S60. RP-HPLC elugram of 9a after purification *via* recycling HPLC.









Figure S64. High resolution-mass spectrum of **9b** (ESI+) after purification *via* column chromatography.

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Figure S65. RP-HPLC of 9b after purification via column chromatography.

Phenyl 4-((4-methoxybenzyl)amino)-2,5-bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy) benzoate (10a)



Figure S66. ¹H NMR spectrum (300 MHz) of **10a** in CDCl₃ after purification *via* recycling HPLC.



Figure S67. ¹³C NMR spectrum (75 MHz) of 10a in CDCl₃ after purification *via* recycling HPLC.



Figure S68. APT spectrum (75 MHz) of 10a in CDCl₃ after purification via recycling HPLC.



Figure S69. High resolution-mass spectrum of 10a (ESI+) after purification via recycling HPLC.



Figure S70. RP-HPLC elugram of 10a after purification via recycling HPLC.

Phenyl 4-((4-methoxybenzyl)amino)-2-(2-(2-(2-methoxyethoxy)ethoxy) benzoate (10b)



Figure S71. ¹H NMR spectrum (300 MHz) of 10b in CDCl₃ after purification *via* recycling HPLC.



Figure S72. ¹³C NMR spectrum (75 MHz) of 10b in CDCl₃ after purification *via* recycling HPLC.



Figure S73. APT spectrum (75 MHz) of 10a in CDCl₃ after purification via recycling HPLC.



Figure S74. High resolution-mass spectrum of 10b after purification via recycling HPLC.



Figure S75. RP-HPLC elugram of 10b after purification *via* recycling HPLC.

4-Amino-2,5-bis(2-(2-(2-methoxy)ethoxy)ethoxy)benzoic acid (11)





Figure S75. ¹H NMR spectrum (360 MHz) of 11 in CDCl₃.

Figure S77. DEPT spectrum (90 MHz) of 11 in CDCl₃.



Figure S78. High resolution-mass spectrum of 11 (ESI+).



Figure S79. RP-HPLC elugram of 11.