

Supporting Information for

Synthesis of Repeating Sequence Copolymers of Methylene and Fluorene Monomers

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Detailed experimental procedures for segmers

SF1M10. Using Method A, diiodofluorene **1** (3.00 g, 5.12 mmol) was combined with 180 mL DMF, Pd(PPh₃)₂Cl₂ (0.15 g, 2.5 mol%), K₂CO₃ (2.97 g, 21.5 mmol, 4.2 eq) and 9-hex-5-enyl-9-BBN **9** (2.72 mL, 11.5 mmol, 2.25 eq). The mixture was stirred at room temperature for 8 h. After work up and column chromatography (silica, hexanes), **SF1M10** was recovered as a colorless oil (1.16 g, 45%). ¹H NMR (300 MHz, CDCl₃) δ 0.61 (b, 4 H), 0.75 (t, 6 H), 1.03 (m, 12 H), 1.39 (m, 4 H), 1.63 (quintet, 4 H), 1.88 (q, 4 H), 2.06 (q, 4 H), 2.66 (t, *J* = 7.4 Hz, 4 H), 4.97 (m, 4 H), 5.77 (m, 2 H), 7.07 (m, 4 H), 7.51-7.54 (m, 2 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.1, 22.5, 23.6, 28.4, 29.7, 31.2, 31.4, 33.7, 36.0, 40.4, 54.6, 114.3, 119.0, 122.9, 126.8, 138.9, 141.1, 150.7. MS (EI), *m/z* 498 (M⁺), 429, 343, 273 (base), 204, 55. HRMS calcd for C₃₇H₅₄: 498.4226. Found: 498.4229. Anal. calcd for C₃₇H₅₄: C, 89.09; H, 10.91. Found: C, 89.10, H, 11.02.

SF1M18. Using Method A, diiodofluorene **1** (3.00 g, 5.12 mmol) was combined with 180 mL DMF, Pd(PPh₃)₂Cl₂ (0.15 g, 2.5 mol%), K₂CO₃ (2.97 g, 21.5 mmol, 4.2 eq) and 9-dec-9-enyl-9-BBN **10** (3.48 mL, 11.5 mmol, 2.25 eq). The mixture was stirred at room temperature for 8 h. After work up and column chromatography (silica, hexanes), **SF1M18** was recovered as a colorless oil (1.53 g, 49%). ¹H NMR (300 MHz, CDCl₃) δ 0.66 (b, 4 H), 0.76 (t, 6 H), 1.10 (m, 12 H), 1.32 (b, 20 H), 1.64 (b, 4 H), 1.91-2.02 (m, 8 H), 2.67 (t, *J* = 7.4 Hz, 4 H), 4.95 (m, 4 H), 5.79 (m, 2 H), 7.09 (m, 4 H), 7.53 (m, 2 H).

^{13}C NMR (75 MHz, CDCl_3) δ 14.0, 22.6, 23.7, 29.0, 29.2, 29.5, 29.7, 31.5, 31.8, 33.8, 36.3, 40.5, 54.6, 114.1, 119.0, 122.8, 126.8, 138.9, 141.3, 150.7. HRMS calcd for $\text{C}_{45}\text{H}_{70}$: 610.5478. Found: 610.5487.

SF2M18. Using Method A, diiodobifluorene **2** (1.18 g, 1.29 mmol) was combined with 28 mL DMF, 28 mL toluene, $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (45 mg, 5 mol%), K_2CO_3 (0.80 g, 5.80 mmol, 4.2 eq) and 9-dec-9-enyl-9-BBN **10** (0.97 mL, 3.2 mmol, 2.25 eq). The mixture was stirred at 45° C for 8 h. After work up and column chromatography (silica, hexanes), **SF2M18** was recovered as a viscous oil (0.62 g, 51%). ^1H NMR (300 MHz, CDCl_3) δ 0.70 (b, 20 H), 1.11 (b, 24 H), 1.32 (b, 20 H), 1.66 (b, 4 H) 2.05 (m, 12 H), 2.69 (t, $J = 7.3$ Hz, 4 H), 4.96 (m, 4 H), 5.76 (m, 2 H), 7.14 (m, 4 H), 7.58-7.73 (m, 8 H). ^{13}C NMR (75 MHz, CDCl_3) δ 14.0, 22.5, 23.7, 28.9, 29.1, 29.2, 29.5, 29.7, 31.4, 31.8, 33.8, 36.3, 40.4, 54.9, 114.1, 119.4, 121.3, 123.0, 125.9, 127.0, 138.4, 139.2, 140.0, 140.3, 141.9, 151.1, 151.4. HRMS calcd for $\text{C}_{70}\text{H}_{102}$: 942.7982. Found: 942.8060.

SF3M18. Using Method A, diiodoterfluorene **3** (0.58 g, 0.46 mmol), was combined with 10 mL DMF, 10 mL toluene, $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (13 mg, 2 mol%), K_2CO_3 (0.27 g, 2.0 mmol, 4.2 eq) and 9-dec-9-enyl-9-BBN **10** (0.56 mL, 1.9 mmol, 4 eq). The mixture was stirred at 45° C for 8 h. After work up and column chromatography (silica, hexanes, then 10% CH_2Cl_2 in hexanes), **SF3M18** was recovered as a white powder (0.42 g, 71%). ^1H NMR (300 MHz, CDCl_3) δ 0.77 (b, 30 H), 1.14-1.40 (b, 56 H), 1.67 (b, 4 H) 2.01-2.07 (b, 16 H), 2.70 (t, $J = 7.2$ Hz, 4 H), 4.95 (m, 4H), 5.83 (m, 2 H), 7.16 (m, 4 H), 7.61-7.81 (m, 14 H). ^{13}C NMR (75 MHz, CDCl_3) δ 13.9, 22.5, 23.9, 29.0, 29.2, 29.5, 31.5, 31.7, 33.8, 36.3, 40.4, 55.1, 55.4, 114.1, 119.5, 119.9, 121.6, 123.1, 125.7, 127.1, 138.6, 139.1, 140.1, 140.2, 140.6, 140.8, 142.0, 151.3, 151.5, 151.9.

SF4M10. Using Method A, diiodoquaterfluorene **4** (0.86 g, 0.54 mmol), was combined with 10 mL DMF, 10 mL toluene, $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (15 mg, 2 mol%), K_2CO_3 (0.80 g, 5.80 mmol, 4.5 eq) and 9-hex-5-enyl-9-BBN **9** (0.52 mL, 2.17 mmol, 4.0 eq). The mixture was stirred at 45° C for 8 h. After work up

and column chromatography (silica, hexanes, then 10% CH₂Cl₂ in hexanes), **SF4M10** was recovered as a white powder (0.73 g, 90%). ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 40 H), 1.12 (b, 48 H), 1.42 (quintet, *J* = 7.7 Hz, 4 H), 1.67 (quintet, *J* = 7.6 Hz, 4 H), 2.02-2.13 (m, 20 H), 2.71 (t, *J* = 7.1 Hz, 4 H), 4.97 (m, 4H), 5.83 (m, 2 H), 7.16 (bs, 4 H), 7.36 (q, 4 H), 7.62-7.83 (m, 16 H). ¹³C NMR (75 MHz, CDCl₃) δ. 14.0, 22.5, 23.8, 28.4, 29.7, 31.4, 33.7, 36.1, 40.4, 55.0, 55.3, 114.4, 119.5, 119.9, 121.4, 121.5, 123.0, 126.0, 127.1, 138.5, 138.9, 139.9, 140.3, 140.6, 141.7, 151.2, 151.4, 151.5, 151.8. Anal. calcd for C₁₁₂H₁₅₀: C, 89.90; H, 10.10. Found: C, 89.26, H, 10.03.

SF4M18. Using Method B, diiodoquaterfluorene **4** (1.6 g, 1.0 mmol) was combined with 20 mL toluene, Pd(PPh₃)₂Cl₂ (0.020 g, 3 mol%), K₂CO₃ (10 ml x 2M, 20 mmol, 20 eq) and 9-hex-5-enyl-9-BBN **10** (1.6 g, 6.0 mmol, 6.0 eq). The mixture was stirred at 45° C for 24 h. After work up and column chromatography (silica, hexanes, then 10% CH₂Cl₂ in hexanes), **SF4M18** was recovered as a colorless, viscous oil (1.3 g, 81%). ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 40 H), 1.13-1.33 (b, 68 H), 1.67 (b, 4 H) 2.01-2.09 (b, 20 H), 2.71 (t, *J* = 7.3 Hz, 4 H), 4.97 (m, 4H), 5.83 (m, 2 H), 7.17 (bs, 4 H), 7.36 (q, 4 H), 7.62-7.81 (m, 16 H). ¹³C NMR (75 MHz, CDCl₃) δ 13.9, 22.3, 23.9, 29.0, 29.2, 29.5, 29.7, 29.9, 31.5, 31.7, 33.8, 36.4, 40.4, 55.1, 55.3, 114.1, 119.6, 120.0, 121.7, 123.0, 126.2, 126.3, 126.9, 127.2, 138.6, 139.7, 140.0, 140.2, 140.7, 140.9, 142.0, 151.3, 151.6, 151.9.

SF5M18. Using method B, diiodopentafluorene **5** (1.45 g, 0.76 mmol) was combined with TBABr (0.07 g, 0.23 mmol, 30 mol%), 20 ml toluene, aqueous K₂CO₃ (7.6 mL x 2.0 M, 15.2 mmol), Pd(PPh₃)₂Cl₂ (16 mg, 3 mol%) and 9-dec-9-enyl-9-BBN **10** (0.79 g, 3 mmol, 4 eq). The mixture was heated at 45° C for 18 h. After work up and column chromatography (silica, hexanes, then 15% CH₂Cl₂ in hexanes), **SF5M18** was recovered as a white solid (1.0 g, 68%). ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 58 H), 1.11-1.47 (b, 95 H), 1.63 (b, 5 H), 1.86-2.11 (b, 27 H.), 2.69 (t, *J* = 7.5 Hz, 4 H), 4.95 (m, 4 H), 5.79 (m, 2 H), 7.16 (b, 5 H), 7.59-7.84 (m, 29 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.2, 23.2, 24.5,

29.6, 29.8, 30.1, 30.3, 32.0, 32.4, 34.4, 36.9, 41.0, 55.6, 55.9, 114.7, 120.1, 120.6, 122.1, 123.7, 126.6, 126.8, 127.7, 128.8, 139.0, 139.82, 140.5, 140.6, 141.2, 141.3, 142.6, 151.8, 152.1, 152.4.

SF6M18. Using method B, diiodohexafluorene **6** (0.70 g, 0.31 mmol) was combined with TBABr (0.03 g, 0.093 mmol, 30 mol%), 20 ml toluene, aqueous K₂CO₃ (3.1 mL x 2.0 M, 6.0 mmol), Pd(PPh₃)₂Cl₂ (6 mg, 3 mol%) and 9-dec-9-enyl-9-BBN **10** (0.32 g, 1.3 mmol, 4 eq). The mixture was heated at 45° C for 18 h. After work up and column chromatography (silica, hexanes, then 10% CH₂Cl₂ in hexanes), **SF6M18** was recovered as a white solid (0.3 g, 43%). ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 79 H), 1.11-1.47 (b, 119 H), 1.63 (b, 5 H), 1.86-2.11 (b, 35 H.), 2.69 (t, *J* = 7.5 Hz, 4 H), 4.95 (m, 4 H), 5.79 (m, 2 H), 7.16 (b, 4 H), 7.59-7.84 (m, 419 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.8, 23.9, 28.9, 29.1, 29.2, 29.5, 29.7, 31.4, 31.8, 33.8, 36.3, 40.3, 54.0, 55.3, 114.1, 119.4, 119.5, 119.9, 121.5, 123.0, 125.9, 126.1, 127.0, 138.4, 139.2, 139.8, 139.9, 140.0, 140.4, 140.5, 140.6, 142.0, 151.1, 151.4, 151.8

SF7M18. Using Method B, diiodoheptafluorene **7** (0.46 g, 0.18 mmol) was combined with TBABr (9 mg, 0.05 mmol, 30 mol%), 6 mL toluene, aqueous K₂CO₃ (2.4 mL x 2.0 M, 4.8 mmol), Pd(PPh₃)₂Cl₂ (6 mg, 3 mol%) and 9-dec-9-enyl-9-BBN **10** (0.22 mL, 0.72 mmol, 4 eq). The mixture was heated at 45° C for 8 h. After work up and column chromatography (silica, hexanes, then 15% CH₂Cl₂ in hexanes), **SF7M18** was recovered as a pale green powder (0.36 g, 78%). ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 70 H), 1.11-1.47 (b, 104 H), 1.63 (b, 4 H), 1.86-2.11 (b, 32 H.), 2.69 (t, *J* = 7.5 Hz, 4 H), 4.95 (m, 4 H), 5.79 (m, 2 H), 7.16 (b, 4 H), 7.59-7.84 (m, 38 H). ¹³C NMR (75 MHz, CDCl₃) δ 13.9, 22.5, 23.9, 29.0, 29.2, 29.5, 29.7, 31.5, 31.8, 33.8, 36.3, 40.4, 55.1, 55.4, 114.1, 119.4, 119.5, 120.0, 121.7, 123.1, 126.0, 126.2, 127.1, 138.6, 140.0, 140.1, 140.6, 140.7, 140.9, 142.0, 151.3, 151.5, 151.9.

SF8M18. Using Method B, diiodooctafluorene **8** (0.42 g, 0.15 mmol) was combined with TBABr (7 mg, 0.05 mmol, 30 mol%), 5 mL toluene, aqueous K₂CO₃ (2.0 mL x 2.0 M, 4.0 mmol), Pd(PPh₃)₂Cl₂ (5

mg, 3 mol%) and 9-dec-9-enyl-9-BBN **10** (0.18 mL, 0.58 mmol, 4 eq). The mixture was heated at 45° C for 8 h. After work up and column chromatography (silica, hexanes, then 15% CH₂Cl₂ in hexanes), **SF8M18** was recovered as a pale green powder (0.31 g, 71%). ¹H NMR (300 MHz, CDCl₃) δ 0.62-1.00 (b, 80 H), 1.12-1.41 (b, 116 H), 1.66 (b, 4 H), 1.90-2.25 (b, 36 H), 2.69 (t, *J* = 7.5 Hz, 4 H), 4.96 (m, 4 H), 5.83 (m, 2 H), 7.15 (bs, 4 H), 7.34 (m, 4 H), 7.64-7.84 (m, 40 H). ¹³C NMR (75 MHz, CDCl₃) δ 13.9, 22.5, 23.9, 24.0, 29.0, 29.2, 29.5, 29.7, 31.5, 31.8, 33.8, 40.4, 55.1, 55.4, 114.1, 119.5, 120.0, 121.7, 123.0, 123.1, 126.0, 126.2, 126.8, 127.1, 138.6, 140.0, 140.1, 140.7, 140.9, 142.0, 151.3, 151.6, 151.9.

Detailed experimental procedures for polymers

PF1M10. The general procedure was followed using **SF1M10** (0.61 g, 1.2 mmol), Ph₂O (0.50 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (25 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (1.9 g SiO₂, 10 mL toluene), **PF1M10** was recovered as a white solid (0.39 g, 68%). *M_w* = 30,000; *M_n* = 14,000; PDI = 2.1. ¹H NMR (300 MHz, CDCl₃) δ 0.60-0.90 (b, 10 H), 1.02-1.34 (b, 24 H), 1.61 (b, 4 H), 1.88 (m, 4 H), 2.64 (t, *J* = 7.4 Hz, 4 H), 7.07 (m, 4 H), 7.50 (d, *J* = 8.1 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.7, 29.3, 29.7, 31.4, 31.8, 36.3, 40.4, 54.6, 118.9, 122.9, 126.8, 138.9, 141.4, 150.8.

PF1M18. The general procedure was followed using **SF1M18** (0.57 g, 0.94 mmol), Ph₂O (0.56 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (17 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (1.6 g SiO₂, 8.5 mL toluene), **PF1M18** was recovered as a white solid (0.45 g, 83%). *M_w* = 33,000; *M_n* = 16,000; PDI = 2.1. ¹H NMR (300 MHz, CDCl₃) δ 0.60 (b, 4 H), 0.76 (t, 6 H), 1.02 (m, 12 H), 1.22-1.36 (b, 28 H), 1.61 (t, 4 H), 1.89 (m, 4 H), 2.64 (t, *J* = 7.3 Hz, 4 H), 7.07 (m, 4 H), 7.50 (d, *J* = 8.1 Hz, 2 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.6, 29.3, 29.6, 29.7, 31.4, 31.8, 36.2, 40.4, 54.6, 118.9, 122.9, 126.8, 138.8, 141.3, 150.7.

PF2M10. The general procedure was followed using **SF2M10** (0.47 g, 0.57 mmol), Ph₂O (0.30 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (10 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.97 g SiO₂, 5.5 mL toluene), **PF2M10** was recovered as a white solid (0.27 g, 60%). M_w = 17,000; M_n = 9,200; PDI = 1.9. ¹H NMR (300 MHz, CDCl₃) δ 0.75 (b, 20 H), 1.06 (b, 24 H), 1.31 (b, 12 H), 1.65 (b, 4 H), 1.97 (b, 8 H), 2.67 (t, *J* = 7.4 Hz, 4 H), 7.14 (bs, 4 H), 7.56-7.69 (m, 8 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.7, 29.3, 29.6, 31.4, 31.9, 36.3, 40.4, 54.9, 119.4, 119.5, 121.3, 123.0, 125.9, 127.0, 138.4, 140.0, 140.3, 141.9, 151.1, 151.4. Anal. calcd for C₆₀H₈₄: C, 89.49; H, 10.51. Found: C, 89.24, H, 10.46.

PF2M18. The general procedure was followed using **SF2M18** (0.49 g, 0.54 mmol), Ph₂O (0.50 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (12 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.96 g SiO₂, 5.5 mL toluene), **PF2M18** was recovered as a white solid (0.30 g, 61%). M_w = 21,500; M_n = 13,000; PDI = 1.7. ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 20 H), 1.08 (b, 26 H), 1.26-1.40 (b, 26 H), 1.66 (b, 4 H), 2.02 (b, 8 H), 2.70 (t, *J* = 7.8 Hz, 4 H), 7.15 (bs, 4 H), 7.59-7.70 (m, 8 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.7, 29.3, 29.7, 31.4, 31.8, 36.3, 40.4, 54.9, 119.4, 119.5, 121.3, 123.0, 125.9, 127.0, 138.4, 140.0, 140.4, 141.9, 151.1, 151.4.

PF3M18. The general procedure was followed using **SF3M18** (0.31 g, 0.24 mmol), Ph₂O (0.30 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (5 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.45 g SiO₂, 3 mL toluene), **PF3M18** was recovered as a white solid (0.23 g, 75%). M_w = 20,000; M_n = 13,000; PDI = 1.5. ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 30 H), 1.09 (b, 64 H), 1.66 (b, 4 H), 2.00-2.06 (b, 12 H), 2.69 (t, *J* = 7.3 Hz, 4 H), 7.15 (b, 4 H), 7.60-7.81 (m, 14 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.2, 22.7, 23.9, 29.4, 29.8, 31.6, 32.0, 36.4, 40.5, 55.1, 55.4, 119.5, 119.6, 120.0, 121.5, 123.2, 126.0, 126.2, 127.2, 138.5, 140.0, 140.6, 140.7, 142.2, 151.2, 151.5, 151.8.

PF4M10. The general procedure was followed using **SF4M10** (0.40 g, 0.27 mmol), Ph₂O (0.30 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (5 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.47 g SiO₂, 3.5 mL toluene), **PF4M10** was recovered as a white solid (0.27 g, 69%). M_w = 9,800; M_n = 7,000; PDI = 1.4. ¹H NMR (300 MHz, CDCl₃) δ 0.78 (b, 40 H), 1.12-1.40 (b, 60 H), 1.67 (b, 4 H), 2.03-2.10 (b, 16 H), 2.70 (t, 4 H), 7.15 (b, 4 H), 7.35 (q, 4 H), 7.61-7.81 (m, 16 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.6, 23.8, 29.3, 29.7, 31.4, 31.9, 36.3, 40.4, 55.1, 55.3, 119.4, 119.9, 121.4, 123.0, 125.9, 126.1, 127.0, 138.3, 139.8, 139.9, 140.3, 140.4, 140.6, 142.0, 151.1, 151.4, 151.8.

PF4M18. The general procedure was followed using **SF4M18** (0.50 g, 0.31 mmol), Ph₂O (0.50 g), Ru(=CHPh)(Cl)₂(PPh₃)₂ (5 mg, 2 mol%), and 10 ml toluene for polymerization. Following hydrogenation (0.50 g SiO₂, 10 mL toluene), **PF4M18** was recovered as a white solid (0.30 g, 60%). M_w = 18,800; M_n = 13,100; PDI = 1.4. ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 40 H), 1.09-1.33 (b, 76 H), 1.66 (b, 4 H), 2.03-2.10 (b, 16 H), 2.70 (t, 4 H), 7.15 (b, 4 H), 7.35 (q, 4 H), 7.61-7.81 (m, 16 H). ¹³C NMR (75 MHz, CDCl₃) δ 14.0, 22.5, 23.8, 29.3, 29.6, 31.4, 31.8, 36.3, 40.4, 55.0, 55.3, 119.4, 119.5, 119.9, 121.4, 121.5, 123.0, 125.9, 126.1, 127.0, 138.4, 139.8, 140.0, 140.3, 140.6, 140.8, 142.0, 151.0, 151.1, 151.4, 151.5, 151.8.

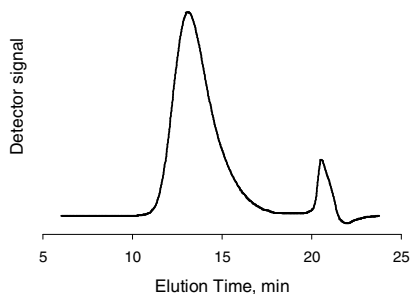
PF5M18. The general procedure was followed using **SF5M18** (0.52 g, 0.26 mmol), Ph₂O (0.50 g), Ru(=CHPh)(Cl)₂(PPh₃)₂ (4 mg, 2 mol), and 10 ml toluene for polymerization. Following hydrogenation (0.22 g SiO₂, 3 mL toluene), **PF5M18** was recovered as a tan powder (0.35 g, 69%). M_w = 57,000; M_n = 36,000; PDI = 1.6. ¹H NMR (300 MHz, CDCl₃) δ 0.76 (b, 64 H), 1.10-1.32 (b, 104 H), 1.52-1.65 (b, 5 H), 2.09 (b, 22 H), 2.68 (b, 4 H), 7.14 (b, 4 H), 7.59-7.83 (b, 24 H). ¹³C NMR (75 MHz, CDCl₃) 14.0, 22.6, 23.9, 29.3, 29.7, 29.8, 30.9, 31.5, 31.9, 36.3, 40.4, 55.0, 55.3, 119.4, 119.5, 120.0, 121.5, 123.1, 126.0, 126.2, 127.1, 138.4, 139.9, 140.0, 140.6, 140.7, 142.1, 151.2, 151.5, 151.8.

PF6M18. The general procedure was followed using **SF6M18** (0.25 g, 0.11 mmol), Ph₂O (0.45 g), Ru(=CHPh)(Cl)₂(PPh₃)₂ (2 mg, 2 mol%), and 10 ml toluene for polymerization. Following hydrogenation (0.50 g SiO₂, 10 mL toluene), **PF6M18** was recovered as a tan powder (0.20 g, 80%). M_w = 45,700; M_n = 24,000; PDI = 1.9. ¹H NMR (300 MHz, CDCl₃) δ 0.76 (b, 76 H), 1.10-1.32 (b, 120 H), 1.52-1.65 (b, 4 H), 2.09 (b, 24 H), 2.68 (b, 4 H), 7.14 (b, 4 H), 7.59-7.83 (b, 336 H). ¹³C NMR (75 MHz, CDCl₃) 14.0, 22.6, 23.9, 29.3, 29.7, 31.5, 31.8, 40.4, 55.0, 55.4, 119.5, 120.0, 121.6, 123.1, 126.2, 127.1, 138.4, 140.0, 140.6, 142.1, 151.2, 151.5, 151.8.

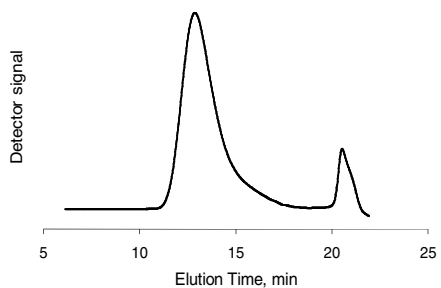
PF7M18. The general procedure was followed using **SF7M18** (0.32 g, 0.13 mmol), Ph₂O (0.15 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (3 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.22 g SiO₂, 3 mL toluene), **PF7M18** was recovered as a tan powder (0.19 g, 59%). M_w = 57,000; M_n = 36,000; PDI = 1.6. ¹H NMR (300 MHz, CDCl₃) δ 0.76 (b, 70 H), 1.10-1.32 (b, 112 H), 1.52-1.65 (b, 4 H), 2.09 (b, 28 H), 2.68 (b, 4 H), 7.14 (b, 4 H), 7.59-7.83 (b, 38 H). ¹³C NMR (75 MHz, CDCl₃) 14.0, 22.5, 23.9, 29.3, 29.7, 31.5, 36.3, 40.4, 55.0, 55.4, 119.5, 120.0, 121.6, 123.0, 126.2, 127.1, 138.5, 140.1, 140.6, 142.1, 151.2, 151.5, 151.9.

PF8M18. The general procedure was followed using **SF8M18** (0.28 g, 0.094 mmol), Ph₂O (0.15 g), and Ru(=CHPh)(Cl)₂(PPh₃)₂ (3 mg, 2 mol%, added as a solution in 2 mL toluene) for polymerization. Following hydrogenation (0.17 g SiO₂, 5 mL toluene), **PF8M18** was recovered as a tan powder (0.11 g, 39%). M_w = 22,000; M_n = 14,000; PDI = 1.6. ¹H NMR (300 MHz, CDCl₃) δ 0.77 (b, 80 H), 1.12-1.32 (b, 124 H), 1.58 (b, 4 H), 2.10 (b, 32 H), 2.69 (t, 4 H), 7.15 (s, 4 H), 7.66-7.84 (m, 44 H). ¹³C NMR (75 MHz, CDCl₃) δ 13.9, 22.5, 23.9, 29.7, 31.5, 40.4, 55.0, 55.4, 118.8, 120.0, 121.6, 123.1, 126.2, 140.1, 140.6, 151.2, 151.5, 151.9.

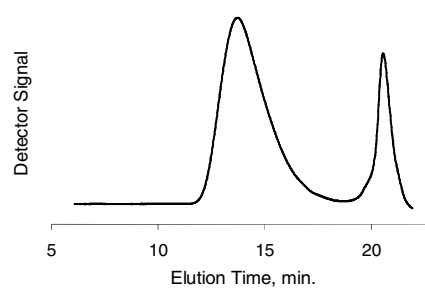
GPC traces for polymers (Note: data were collected on two sets of columns. * denotes data from the Jordi column set)



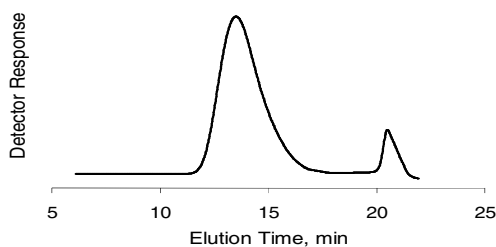
GPC trace of **PF1M10** (THF)



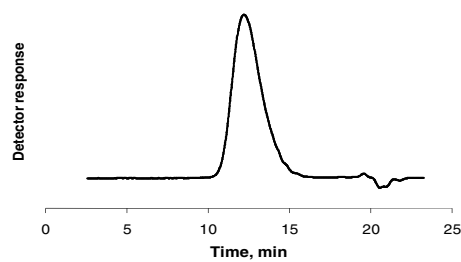
GPC trace of **PF1M18** (THF)



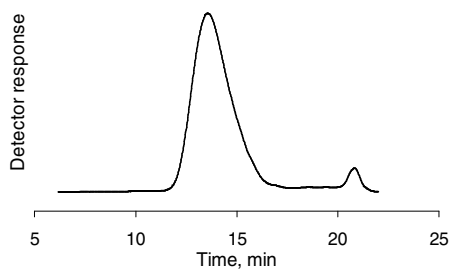
GPC trace of **PF2M10** (THF)



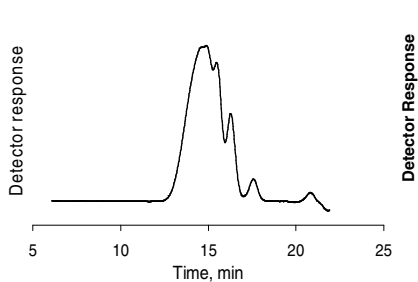
GPC trace of **PF2M18** (THF)



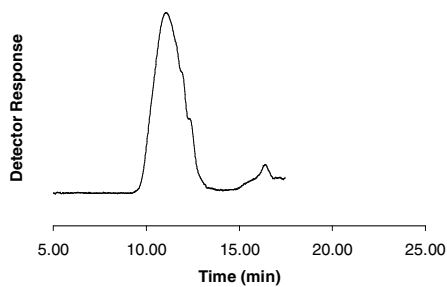
GPC trace of **PF3M10** (THF)



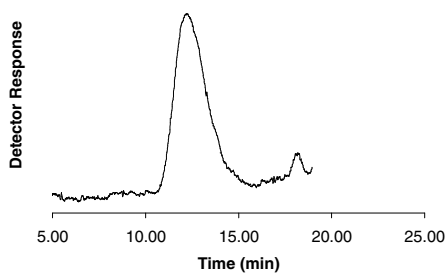
GPC trace of **PF3M18** (THF)



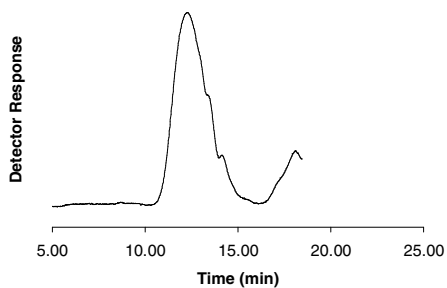
GPC trace of **PF4M10** (THF)



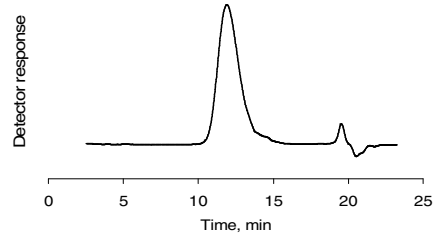
*GPC trace of **PF4M18** (THF)



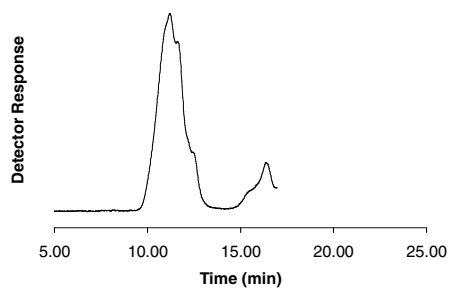
*GPC trace of **PF5M18** (THF)



*GPC trace of **PF6M18** (THF)



GPC trace of **PF7M18** (THF)



*GPC trace of **PF87M18** (THF)