

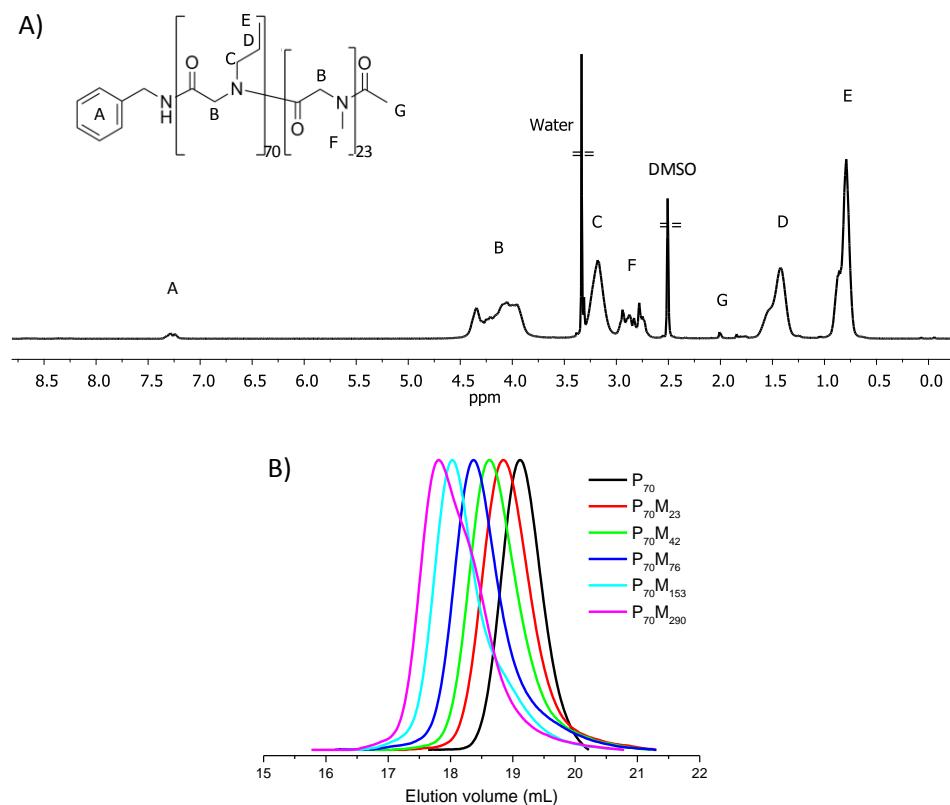
Supporting Information to

# Thermo-Induced Aggregation and Crystallization of Block Copolypeptoids in Water

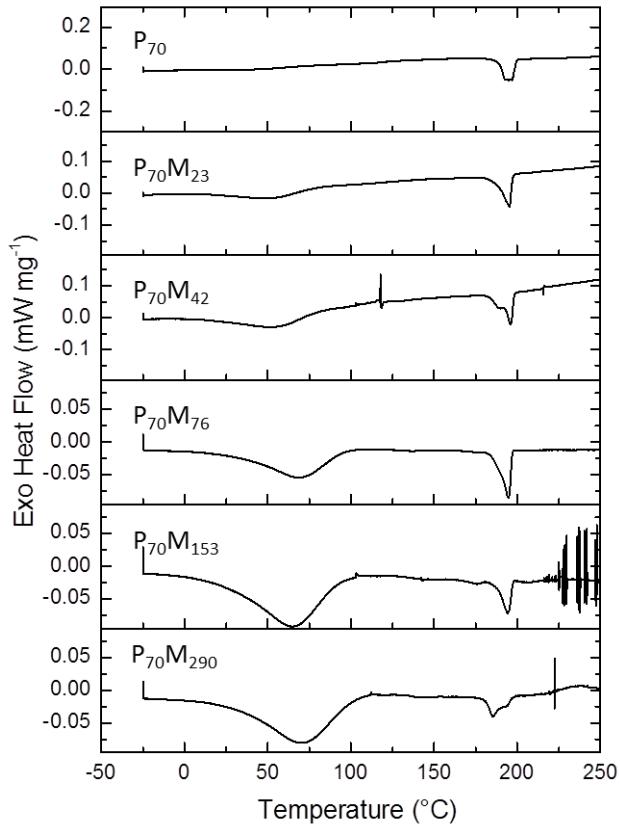
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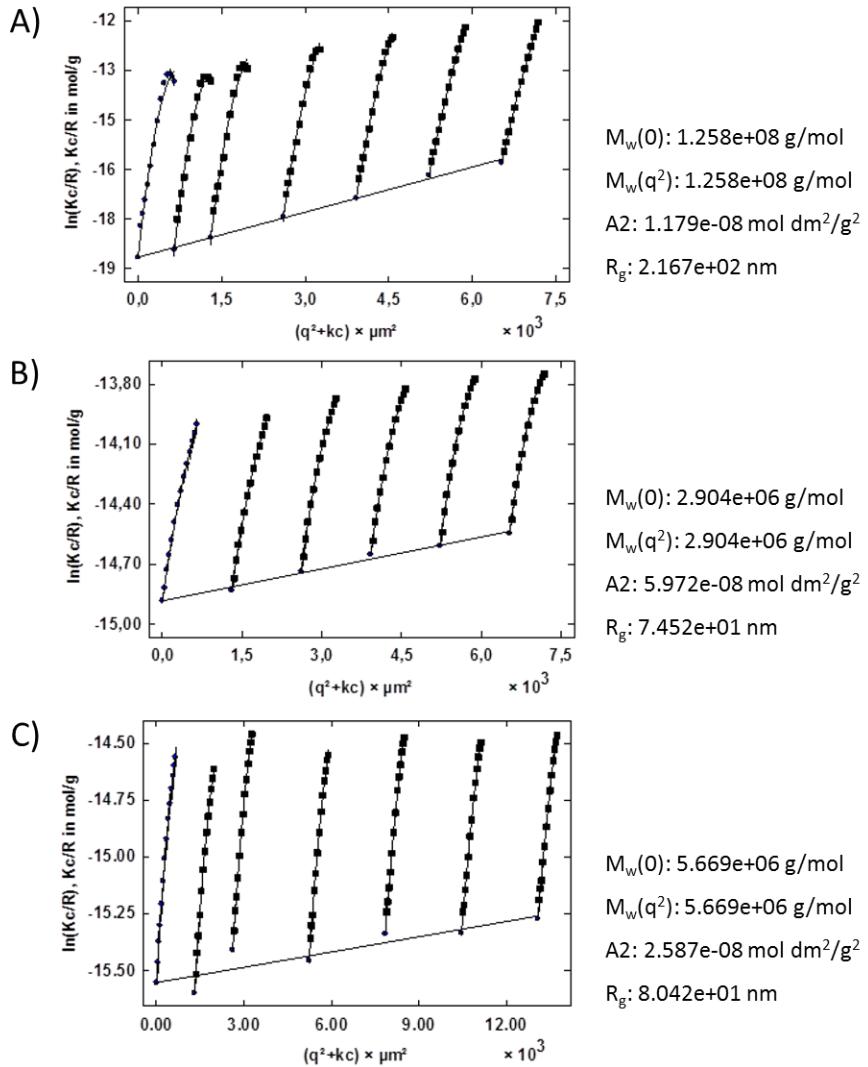
**Figure S1.** A) Exemplary  $^1\text{H}$ -NMR spectrum (600 MHz) of  $P_{70}M_{23}$  in  $\text{DMSO-d}_6$  at room temperature. B) SEC RI traces (eluent: NMP) of the  $P_{70}M_m$  series.



**Figure S2.** DSC first heating scans (heating rate: 1 K min<sup>-1</sup>) of the thermally annealed (in water) and freeze-dried block copolypeptoids P<sub>70</sub>M<sub>m</sub>.

**Table S1.** Glass transition ( $T_g$ ) and melting temperatures ( $T_m$ ) of thermally annealed (in water) and freeze-dried block copolypeptoids P<sub>70</sub>M<sub>m</sub>, as determined by DSC (Figure S2).

Sample	$T_g$ (°C)	$T_m$ (°C)
P <sub>70</sub>	58	192, 194, 197
P <sub>70</sub> M <sub>23</sub>	65	193, 195
P <sub>70</sub> M <sub>42</sub>	68	187, 194
P <sub>70</sub> M <sub>76</sub>	84	188, 194
P <sub>70</sub> M <sub>153</sub>	82	194
P <sub>70</sub> M <sub>290</sub>	89	185, 194



**Figure S3.** SLS Guinier plots for 0.01-0.1 wt % aqueous dispersions of thermally annealed block copolypeptoids A) P<sub>70</sub>M<sub>23</sub>, B) P<sub>70</sub>M<sub>42</sub>, and C) P<sub>70</sub>M<sub>76</sub>.