

*Supporting information for:*

High Morphological Order in a Nearly Precise Acid-Containing Polymer and Ionomer

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## Experimental Methods

**Materials.** The syntheses of all polymers were reported previously. 11/12SAnh was synthesized via step polymerization of succinic anhydride-alcohol monomers.<sup>1</sup> The precise polymers p9AA and p15AA were synthesized via acyclic diene (ADMET) polymerization of symmetric diene monomers.<sup>2</sup> The pseudorandom copolymer r15AA was synthesized via ring-opening metathesis copolymerization of cyclooctene and 2-(4-cyclooctenyl)acetic acid.<sup>2</sup> The 9 and 15 in p9AA and p15AA refer to the number of backbone carbon atoms in each repeating unit. In 11/12SAnh, the repeating unit can contain either 11 or 12 backbone carbon atoms. In r15AA, the overall chemical composition is identical to p15AA but the architecture is pseudorandom.

Neutralization of 11/12SAnh was accomplished by adding a warm (70–90°C) solution of sodium acetate (345 mg dissolved in 350 mL of 20 v/v% 1,4-dioxane in 1-butanol at 90°C over 3 hours) to a warm (50°C) solution of the polymer (1 gram dissolved in 20 mL of 33 v/v% 1,4-dioxane in 1-butanol) over 20 minutes. The neutralized polymer precipitated out of solution during the addition. After decanting 1,4-dioxane/1-butanol, the precipitated polymer was washed with 2 x 10 mL of 1-butanol, scraped from the flask, and dried overnight under vacuum at ambient temperature. The neutralization procedure for p9AA, p15AA, and r15AA utilized sodium acetate and was reported previously.<sup>3</sup> To determine neutralization level, elemental analysis was performed by Galbraith Laboratories (Knoxville, TN) using elemental analyzers for C, H, O, and Na. The error in the reported neutralization percentage is approximately  $\pm 3\%$ . The neutralization percentage as determined by elemental analysis is included in the naming scheme (43Na means 43% of COOH groups are neutralized with sodium).

**FTIR.** Attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR) was carried out on a Nicolet 6700 (Thermo Fisher) spectrometer using the GATR accessory (Harrick) with a resolution of  $2\text{ cm}^{-1}$ . Curves were normalized by the absorbance of the CH bending mode at  $1466\text{ cm}^{-1}$ . Being a viscous fluid, 11/12SAnh was simply applied onto the Ge crystal with a spatula. 11/12SAnh-43Na was hot-pressed onto a stainless steel AFM disc (Ted Pella, Inc. 16218) at 50°C, and this film was pressed onto the Ge crystal with the AFM disc acting as a support.

**X-ray Scattering.** The un-neutralized 11/12SAnh was applied onto a 20  $\mu\text{m}$  thick sheet of mica using a spatula. The neutralized sample 11/12SAnh-43Na was hot-pressed at 50°C into a film of thickness 400 $\mu\text{m}$  and no substrate was used. X-ray scattering was performed at room temperature at the Multi-Angle X-ray Scattering (MAXS) facility at the University of Pennsylvania, with a Cu K $\alpha$  source and a sample-to-detector distance of 11 cm. Details of the setup can be found in ref 4. Data reduction and analysis was performed using the Datasqueeze software.<sup>5</sup> Low-q peaks were fit with a Lorentzian peak shape and a linear background. The instrumental broadening, estimated from the line widths of silver behenate, was less than 0.04  $\text{\AA}^{-1}$  FWHM.

**DSC.** Samples for DSC were dried for at least 12 hours under vacuum, and were loaded into hermetic pans. DSC was performed using a Q2000 (TA Instruments).  $T_g$  was obtained from the point of inflection upon cooling at a rate of 10°C/min.

## References

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