

Figure S1: <sup>1</sup>H NMR of polystyrene-*b*-polyethylene oxide-alkyne. The integrals specify a ratio of roughly 90:11:1 for PEO:PS: alkyne end groups. Some residual diethyl ether is present (resonances at 1.2 and 3.5 ppm). We are unable to assign the singlet at 2.15 ppm.

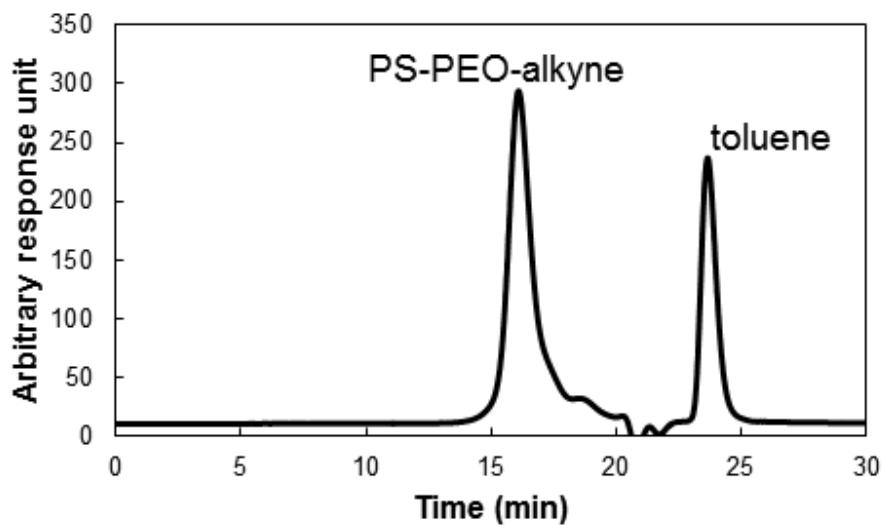


Figure S2: GPC trace of the PS-PEO-alkyne block copolymer. Arbitrary response units reflect the change in refractive index. The GPC was run at 40 °C in DMF + 0.02 M ammonium acetate. Toluene was added as a small molecule standard. The molecular weight was estimated using a  $\text{dn}/\text{dc}$  value of 0.073 corresponding to a polymer composed of 75 wt% PEO ( $\text{dn}/\text{dc} = 0.044$  in DMF) and 25 wt% PS ( $\text{dn}/\text{dc} = 0.16$  in DMF).

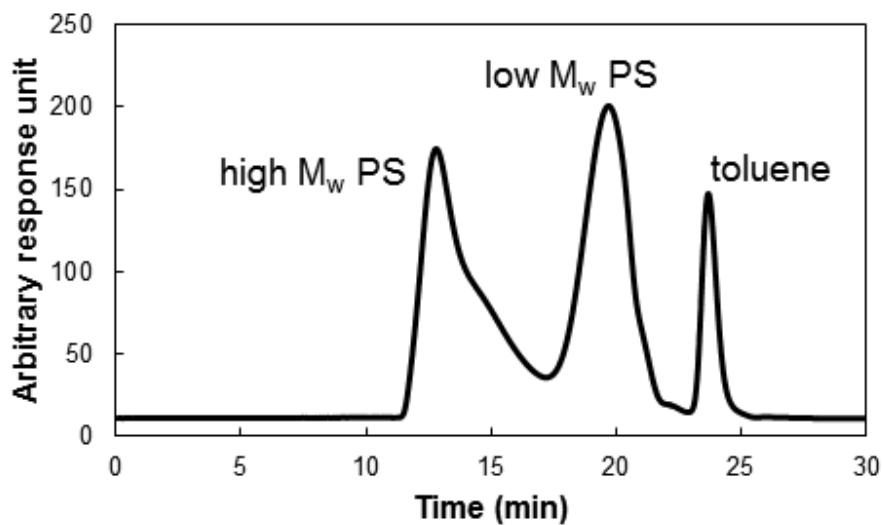


Figure S3: GPC trace of commercial PS used in film crosslinking studies. Conditions for GPC are described in the caption of Figure S2. The sample is comprised of roughly equal masses of high  $M_w$  material ( $M_w = 120,000$ , PDI = 2.0) and low  $M_w$  material ( $M_w = 1900$ , PDI = 1.5).