Supporting Information for:

Co-Crystallization Phase Transformations in All-Conjugated Block Copolymers with Different Main-Chain Moieties

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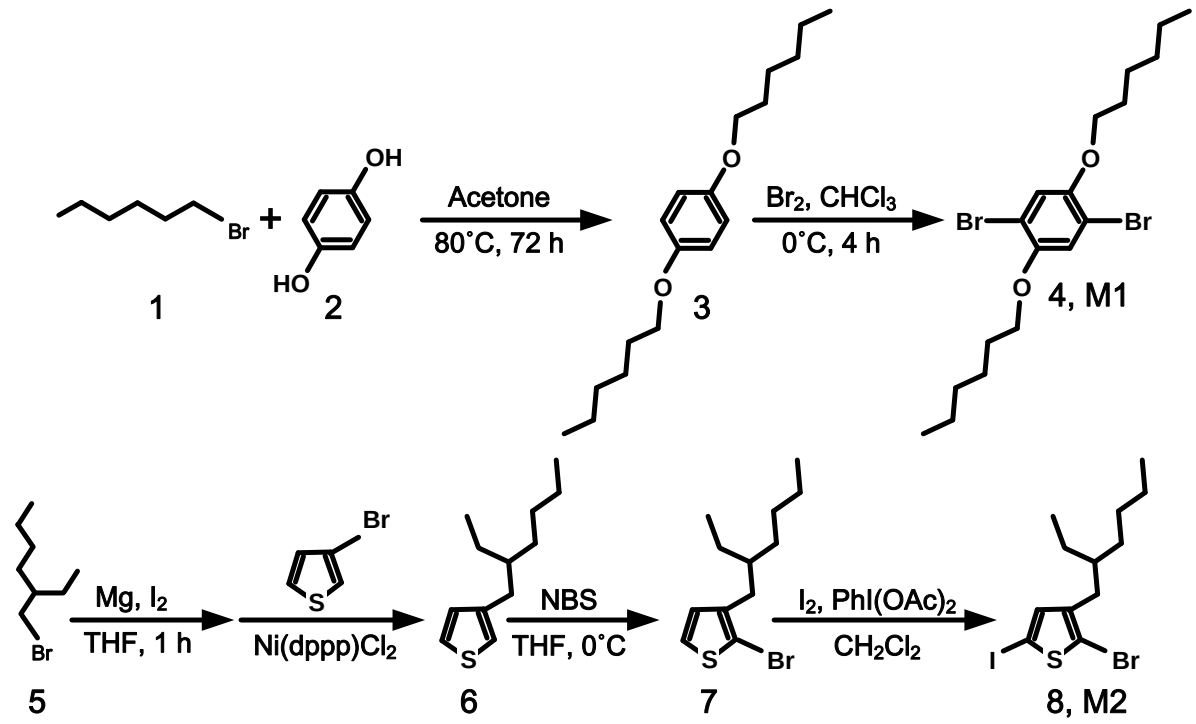
Materials.

Acetone (Acros, 99.8%), 1-bromohexane (Acros, 99%), hydroquinone (Acros, 99.5%), iodobenzene diacetate (Acros, 98%), 1-bromo-2-ethylhexane (Acros 95%), [1,3-bis(diphenylphosphino)propane]dichloronickel(II) ($Ni(dppp)Cl_2$, Acros, 99%), iodine (Mallinckrodt, 99.8%), bromine (Br_2, Acros, 99%), magnesium turnings (Acros, 99.9%), potassium carbonate (K_2CO_3, Acros, 99%), and *N*-bromosuccinimide (NBS, Acros, 99%) were used as received without purification. Column chromatography was performed on silica gel (230-400 mesh ASTM, Merck silica gel).



Scheme S1 Synthetic routes of **M1** and **M2** monomers.

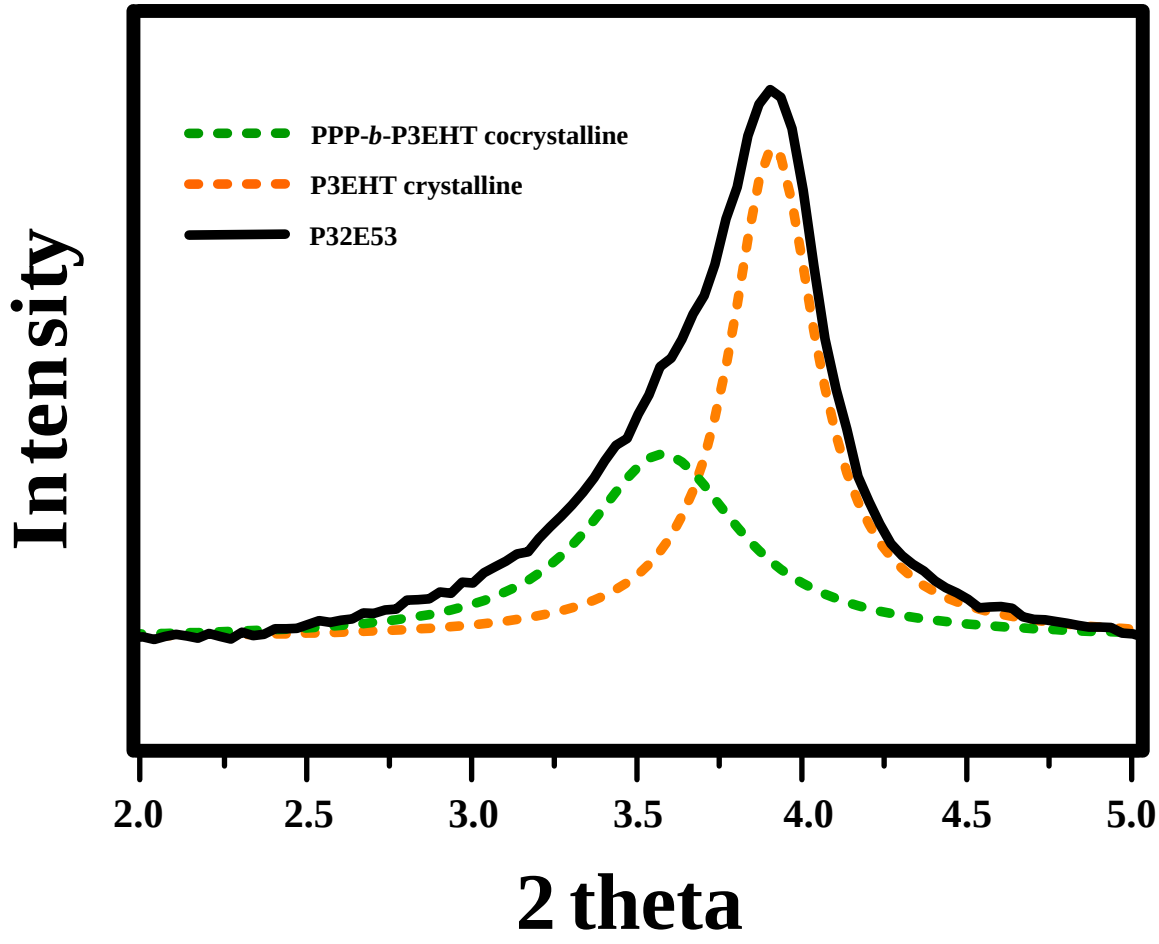


Fig. S1 The deconvolution of the (100) peak of **P32E53** is determined by a procedure based on the best fitting of a Lorentzian function. The orange and green dashed lines indicate the corresponding (100) peak of P3EHT crystalline and PPP-P3EHT cocrystalline domains, respectively.

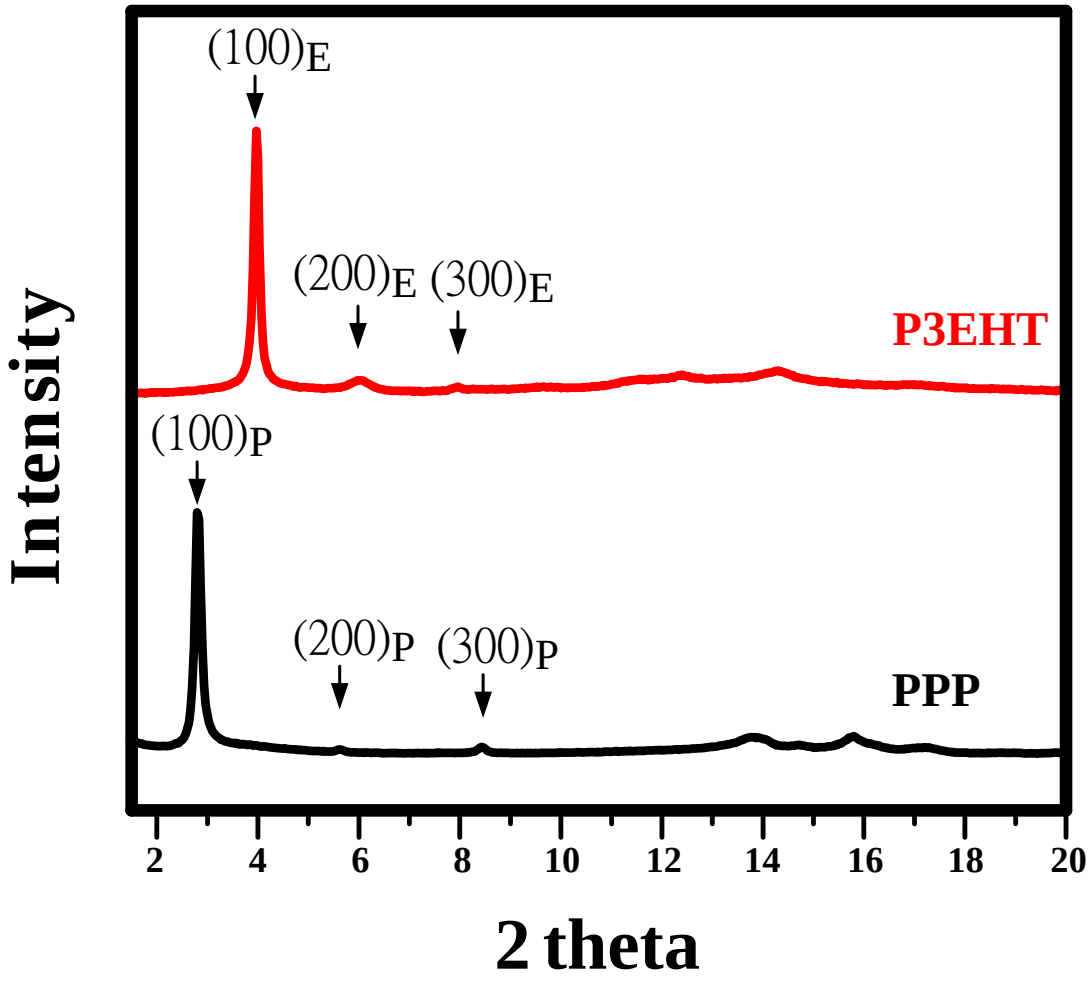


Fig. S2. X-ray diffraction (XRD) scans of PPP and P3EHT homopolymers.

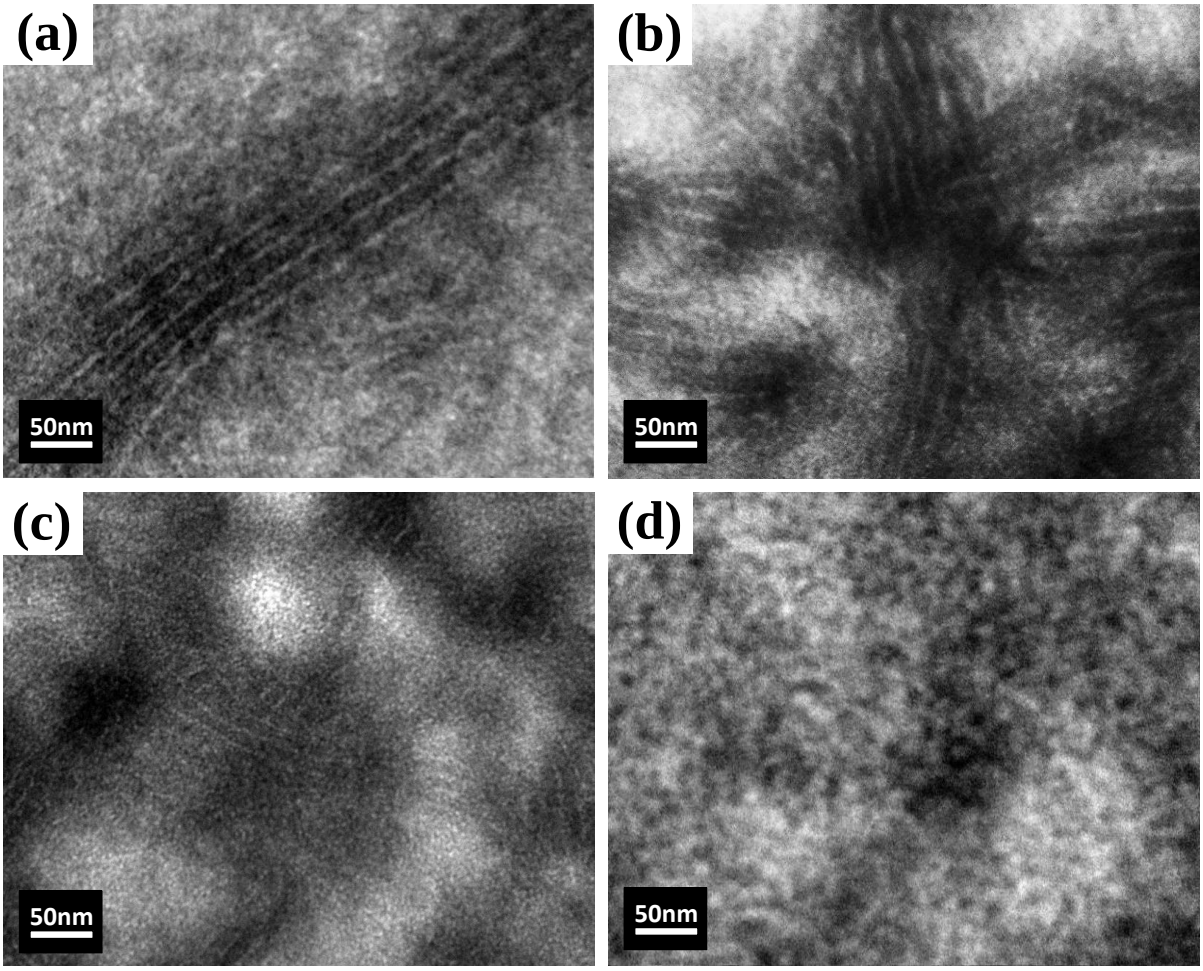


Fig. S3. In order to investigate the staining time on the TEM contrast, P34E64 samples were used by using RuO_4 as the staining agent for P3EHT domains. (a) 3 mins, (b) 5 mins, (c) 10 mins, and (d) 30mins. The RuO_4 staining for 5 mins giving the best contrast is used for all block copolymers.

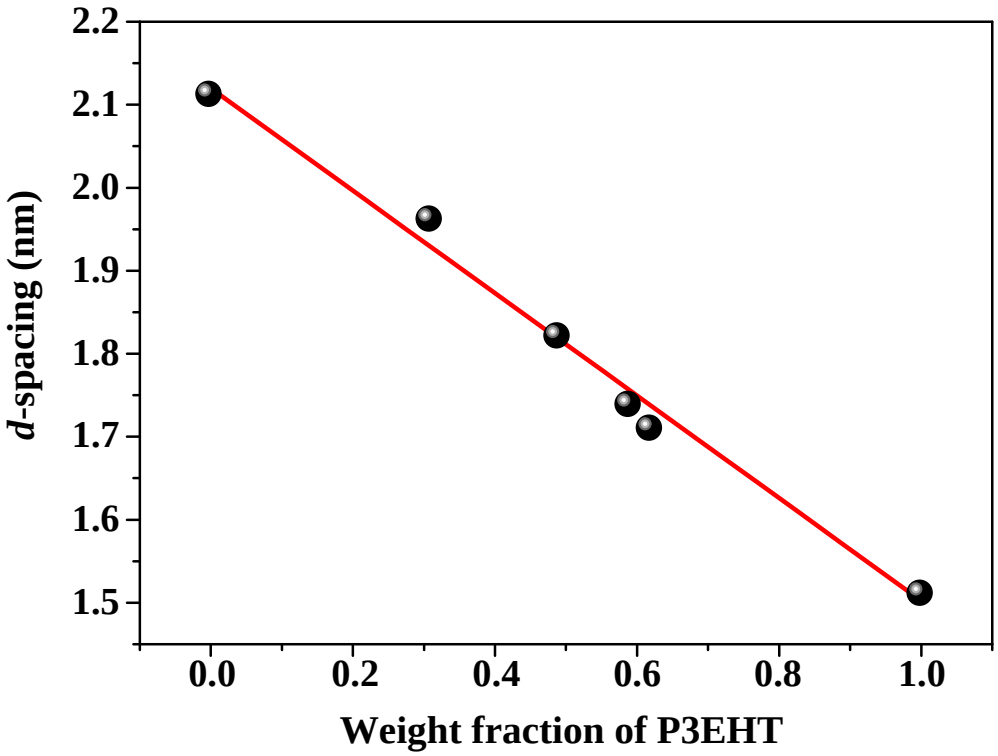


Fig. S4. Dependence of the d-spacing of $(100)_{P+E}$ plane on the weight fraction of P3EHT in the cocrystals.