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Supporting Information for:

Well-defined triblock copolymers with photolabile middle block of poly(phenyl vinyl ketone): Facile synthesis, chain-scission mechanism and controllable photocleavability

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Synthesis of PVK

According to a modified method [1], the monomer phenyl vinyl ketone (PVK) was prepared by a a-methylenation reaction between PMK and POM by using the salt complex of DIPA:TFA as catalyzer. Firstly, the catalyzer was prepared by dropwise adding 110 mmol TFA into the DIPA solution in (100 mmol in 100 mL THF) in icewater bath. The reaction mixture is stirred at 0°C for 15 min. Subsequently, PMK (100 mmol) and POM (200 mmol) were added into the above mixture solution and then stirred at reflux for 2h. The mixture will become clear, and then the reaction mixture was cooled down to room temperature. After adding 200 mmol POM again, the reaction mixture was stirred at reflux for an additional 6 h. After cooling down to room temperature, the solvent was removed under reduced pressure and the resulted yellow viscous oil was poured into about 100 mL diethyl ether (Et₂O). After successively washing with 1M HCl, 1M NaOH solution and brine, and drying under vacuum, the crude product was obtained and can be purified by silica gel column chromatography using 5% Et₂O-Hexane as the eluent. The product was characterized by ¹HNMR, as shown in **Figure S1**.

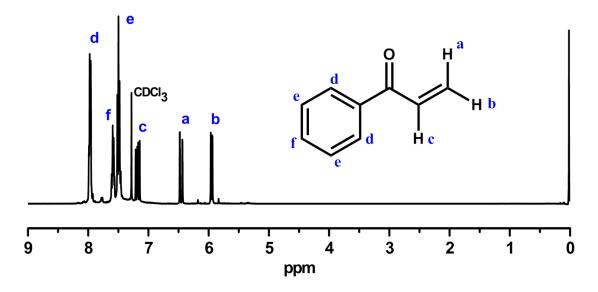


Figure S1. ¹HNMR spectrum of PVK

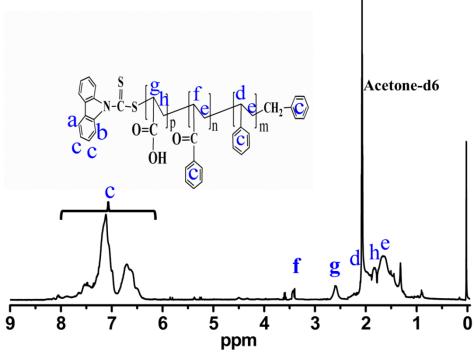


Figure S2. ¹HNMR spectrum of PSt₉₂-PPVK₁₁-PAA₂₆

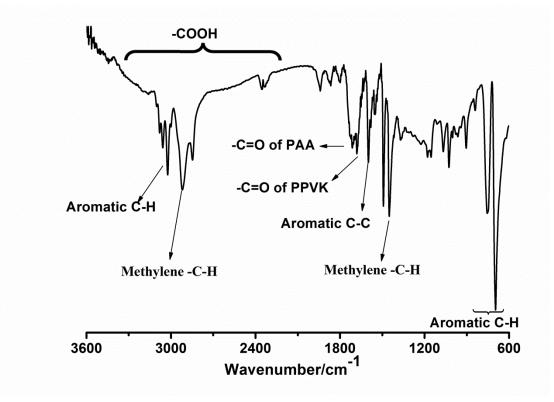


Figure S3. FTIR spectrum of PSt₉₂-PPVK₁₁-PAA₂₆

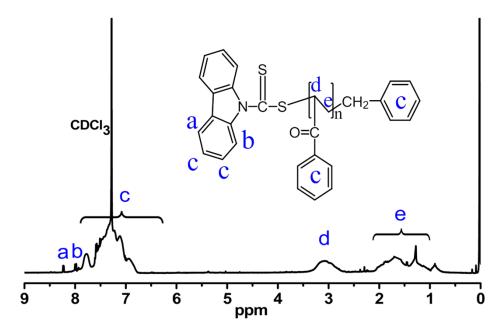


Figure S4. ¹HNMR spectrum of PPVK₄₈

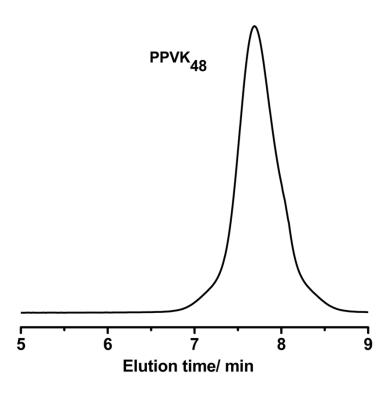


Figure S5. GPC curve of $PPVK_{48}$

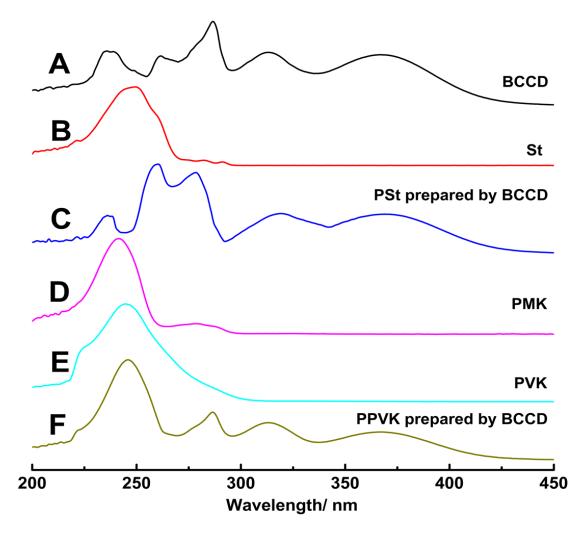
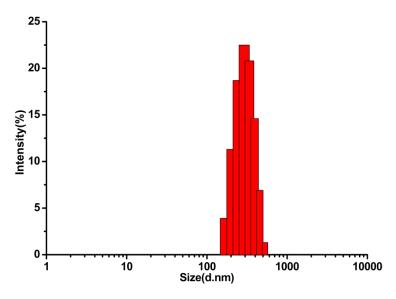


Figure S6. UV curves of BCCD, St, PSt prepared by BCCD, PMK, PVK and PPVK prepared by BCCD



 $\textbf{Figure S7}. \ \ \text{Size and size distribution of } PSt_{92}\text{-}PPVK_{11}\text{-}PAA_{26} \ self\text{-}assembled \ micelles}$

Reference

1 A. Bugarin, K. D. Jones and B. T. Chem. Commun., 2010, 46, 1715–1717.