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Supplementary Information for

## Versatile, Stable, and Air-Tolerant Triplet-Triplet Annihilation Upconversion Block Copolymer Micelles

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## Synthesis and characterization.



Figure S1 Synthesis of the DPA-containing monomer DPAMA.



Figure S2 <sup>1</sup>H NMR spectrum of DPA(CH<sub>2</sub>)<sub>6</sub>OH.



Figure S3 <sup>1</sup>H NMR spectrum of DPAMA.



Figure S4 <sup>13</sup>C NMR spectrum of DPAMA.



Figure S5 <sup>1</sup>H NMR spectrum of PtBA-*b*-PDPAMA diblock copolymer.



Figure S6 TGA trace of the PtBA-b-PDPAMA-diblock copolymer.



Figure S7 DSC trace of PtBA-b-PDPAMA-diblock copolymer.



Figure S8 EM image of the UC micelles after hydrolysis of the PtBA chains and staining with uranyl acetate. Scale bar is 500 nm.



**Figure S9** EM image of the UC micelles co-assembled in (a) *n*-butanol, (b) acetone, (c) 1,4-dioxane, (d) *n*-butyl acetate. Scale bars are 500 nm.



**Figure S10** PL spectra ( $\lambda_{ex} = 532$  nm) of the UC micelles in *n*-butanol (red), and the dissolved diblock copolymer and PtOEP in THF (blue) at 77 K.



Figure S11 Phosphorescence decays of the *n*-butanol solutions of (a) pure PtBA-b-PDPAMA micelles, (b) UC micelles irradiated at 360 nm.

Solution	Diameter from	$D_h$ from	PDI from
	EM image (nm)	DLS (nm)	DLS
<i>n</i> -Butanol	55	61	0.203
Acetone	57	65	0.176
1,4-Dioxane	49	57	0.183
<i>n</i> -Butyl acetate	52	59	0.166

Table S1 Characterization results of PtBA-b-PDPAMA micelles.