## Redox-responsive core cross-linked micelles based on cypate and cisplatin prodrugs-conjugated block copolymers for synergistic photothermal-chemotherapy of cancer

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Fig. **S1 RP-HPLC** cisplatin, traces recorded for a) b) *c*,*c*,*t*-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>(OH)(O<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H)], c) c,c,t-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>(OH) (O<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CONHCH<sub>2</sub>C≡CH)], and d) c,c,t-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>(OH) (O<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CONHCH<sub>2</sub>C≡CH)] upon treating with 10 mM DTT for 1 h. The mobile phase was 80/20 methanol and water at a flow rate of 1.0 mL/min.



Fig. S2 <sup>1</sup>H NMR spectra recorded for cypate and *alkynyl*-cypate in DMSO-*d*<sub>6</sub>.



**Fig. S3** <sup>1</sup>H NMR spectra recorded for P(MEO<sub>2</sub>MA-*co*-MASI) macroRAFT agent in CDCl<sub>3</sub>, P(MEO<sub>2</sub>MA-*co*-MASI)-*b*-PHPMA block copolymer, P(MEO<sub>2</sub>MA-*co*-MASI-*co*-AzPMA)- *b*-PHPMA, and Pt(IV) complex and cypate-conjugated block copolymer, (Pt-Cy-PMEO<sub>2</sub>MA-*co*-PMASI)-*b*-PHPMA in DMSO-*d*<sub>6</sub>.



**Fig. S4** FT-IR spectra recorded for a) P(MEO<sub>2</sub>MA-*co*-MASI-*co*-AzPMA)-*b*-PHPMA block copolymer and b) (Pt-Cy-PMEO<sub>2</sub>MA-*co*-PMASI)-*b*-PHPMA block copolymer.



**Fig. S5** Cell viability of A549R cells after incubation with  $P(Cy_{21}-MEO_2MA_{67})$ -*b*-PHPMA<sub>148</sub> CCL micelles containing various cypate moiety concentrations.



**Fig. S6** Fluorescence intensity of DPBF in aqueous solution (15  $\mu$ M) in the presence of P(Pt<sub>14</sub>-Cy<sub>7</sub>-MEO<sub>2</sub>MA<sub>67</sub>)-*b*-PHPMA<sub>148</sub> CCL micelles with varying concentrations under an 805 nm NIR laser irradiation at a power density of 1 W/cm<sup>2</sup> for 3 min. The concentrations of cypate moieties are 0, 2.5, 5, 10, and 25  $\mu$ M, respectively.