<sup>1</sup>Supporting Information

## Multifunctional Platinum(IV) and Cyanine Dye-based Polyprodrug for

## **Trimodal Imaging-Guided Chemo-Phototherapy**

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## Synthesis of cis, cis, trans-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>(OH)<sub>2</sub>] (DHP)

According to reports in the literature, cisplatin (1.0 g, 3.33 mmol), ultrapure water (10mL) and 10mL hydrogen peroxide ( $H_2O_2$ , 30%) were added to a 50 mL flask and stirred for 12 h in the dark. After the filtration, the obtained solid (DHP) was washed with ultrapure water and ice-cold acetone for several times, dried in vacuum to obtain a light yellow powder solid.



Scheme S1. Synthetic scheme of the DHP.

## Synthesis of HO-Cy-OH (Cy)

For the preparation of Cy, according to the literature reports, 2,3,3-trimethylindolenine (3 g, 18.9 mmol) and 2-bromoethanol (2.83 g, 22.7 mmol) and 80 mL of anhydrous acetonitrile

were reacted to obtain a pink solid 1-(3-hydroxyethyl)-2,3, 3-trimethyl-3H-indol-1-ium. Then, Cyclohexanone (5 g, 51 mmol) and POCl<sub>3</sub> (20 mL) were reacted to obtain a yellow solid, 1cyclohexene-1-carboxaldehyde. 1-cyclohexene-1-carboxaldehyde (1.73 g, 10 mmol), 1-(3hydroxypropyl) -2,3,3-trimethyl-3H-indol-1-ium (5.735 g, 20 mmol) was dissolved in anhydrous n-butanol (70 mL) and toluene (30 mL), after 12 h of the reaction, the solvent was separated and collected by an oil-water separation device, and after cooling to room temperature, the crude product is separated and purified by a chromatographic column to obtain HO-cy-OH (Cy).



Scheme S2. Synthetic route of Cy.







Figure S2. <sup>1</sup>H NMR spectrum of Cy in CDCl<sub>3</sub>.



Figure S3. FT-IR spectrum of monomer (DHP, Cy, CBDA) and PCPP.



Figure S4. (A) GPC curve of PCPP (DMF as eluent), (B) <sup>1</sup>H NMR spectrum of PCPP in  $D_2O$ .



Figure S5. Zeta potential measurement of PCPP NPs.



Figure S6. Fluorescence spectra of Cy and PCPP NPs.



Figure S7. Absorption spectra of DPBF solution incubated with PCPP NPs in the dark.



**Figure S8.** Flow cytometry (FCM) analyses of HeLa cells incubated with PCPP NPs and Cy for 4 h.



Figure S9. The H22-tumor-bearing BABL/c nude mice after injecting PCPP NPs for 24h.



Figure S10. The isolated tumors of all euthanized H22-tumor-bearing KM mice.



**Figure S11.** Typical H&E-stained slices of major organs tissues collected from different groups. Scale bar: 70 µm.



Figure S12. Blood biochemistry of different groups.