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## **Supporting Information**

# H<sub>2</sub>O<sub>2</sub>-responsive polymeric micelle with a benzil moiety for efficient DOX delivery and AIE Imaging

Yan-Dong Dai,<sup>a</sup> Xue-Yi Sun,<sup>b</sup> Wan Sun,<sup>b</sup> Jing-Bo Yang,<sup>b</sup> Rui Liu,<sup>\*b</sup> Yi Luo,<sup>c</sup> Tao Zhang,<sup>c</sup> Yu Tian,<sup>d</sup> Zhong-Lin Lu<sup>\*b</sup> and Lan He<sup>\*a</sup>

<sup>a</sup> National Institutes for Food and Drug Control, Institute of Chemical Drug Control, TianTanXiLi 2, Beijing, 100050, China. E-mail: helan1961@aliyun.com

- <sup>b</sup> Key Laboratory of Radiopharmaceuticals, Ministry of Education, College of Chemistry, Beijing Normal University, Beijing 100875, China. E-mail: rliu@bnu.edu.cn, luzl@bnu.edu.cn
- <sup>c</sup> Guangxi Institute for Food and Drug Control, Yunjing Road 32, Nanning, Guangxi Zhuang Autonomous Region, 530029, China.

<sup>d</sup> Solid Waste and Chemicals Management Center, Ministry of Environmental Protection of the People's Republic of China, Beijing 100029, China

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### 1. AIE Fluorescent Spectra of TPG2



Figure S1. Fluorescent spectra of TPG2 (20  $\mu$ M,  $\lambda_{ex}$  = 320 nm) in different THF/H<sub>2</sub>O ratios.

2. <sup>1</sup>H NMR Spectrum of **TPG1** in water



12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0

Figure S2. <sup>1</sup>H NMR spectrum of TPG1 in D<sub>2</sub>O (600 MHz, 298 K). Inset: the enlarged spectrum of TPG1 in D<sub>2</sub>O.

## 3. MS spectra of TPG1 and TPG2



Figure S3. MALDI-TOF spectrum of TPG1.



Figure S4. ESI-HRMS spectrum of TPG2.

4. NMR Spectra of Synthesized Compounds



Figure S6. <sup>13</sup>C NMR spectrum of compound **1** (151 MHz, CDCl<sub>3</sub>, 298 K)



Figure S8. <sup>13</sup>C NMR spectrum of compound 2 (151 MHz, CDCl<sub>3</sub>, 298 K)



Figure S10. <sup>13</sup>C NMR spectrum of compound **3** (101 MHz, CDCl<sub>3</sub>, 298 K)



Figure S12. <sup>13</sup>C NMR spectrum of compound 4 (101 MHz, CDCl<sub>3</sub>, 298 K)

 10.4430

 8.1353

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Figure S13. <sup>1</sup>H NMR spectrum of compound **5** (400 MHz, CDCl<sub>3</sub>, 298 K)



Figure S14. <sup>13</sup>C NMR spectrum of compound **5** (151 MHz, CDCl<sub>3</sub>, 298 K)



Figure S16..<sup>13</sup>C NMR spectrum of compound 7 (151 MHz, CDCl<sub>3</sub>, 298 K)



Figure S18. <sup>1</sup>H NMR spectrum of PEG<sub>2000</sub>-COOH (400 MHz, CDCl<sub>3</sub>, 298 K)

#### 5. Drug Loading Measurement

5 mg of TPG1 and 2 mg DOX•HCl (the HCl was removed by triethylamine) was taken to prepare the drug loaded micelles named TPG1@DOX, the unloaded DOX was removed via 0.45  $\mu$ m filter membrane, the total volume of micells was 5mL. To determine the mount of loaded drug, the 50 $\mu$ L prepared TPG1@DOX was dissolved in 5 mL methanol and the structure of micelles was destroyed, the fluorescence spectroscopy of DOX was shown in Figure S19.A, the fluorescence intensity at largest emission wavelength (590 nm) was 90. The concentration of DOX encapsulated could be calculated according to the calibration curve of DOX•HCl in methanol in the presence of triethylamine (Figure S19.B). the diluted concentration of DOX was about 2.35  $\mu$ g/mL.

DL (drug loading capability) =  $(2.35 \ \mu g/mL \times 100 \times 5 \ mL)/2 \ mg \times 100\% = 58.8\%$ EE (encapsulation efficiency) =  $(2.35 \ \mu g/mL \times 100 \times 5 \ mL)/5 \ mg \times 100\% = 23.5\%$ 



Figure S19. A) the Fluorescent spectra of TPG1@DOX dissolved in methanol. B) the calibration curve of DOX•HCl in methanol. ( $\lambda_{ex} = 480 \text{ nm}, \lambda_{em} = 590 \text{ nm}, \text{ slit} = 5 \text{ nm}, 5 \text{ nm}$ )