

### Electronic Supplementary Information

#### PEGylated poly(diselenide-phosphate) nanogel as efficient self-delivery nanomedicine for cancer therapy

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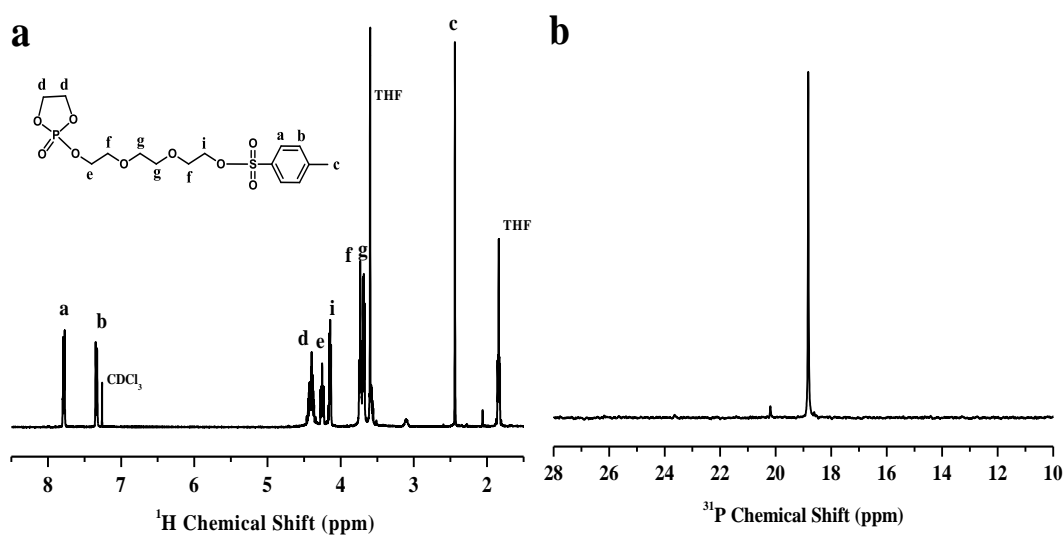


Fig. S1 (a) <sup>1</sup>H NMR and (b) <sup>31</sup>P NMR spectrum of MBS-EEEP in CDCl<sub>3</sub>

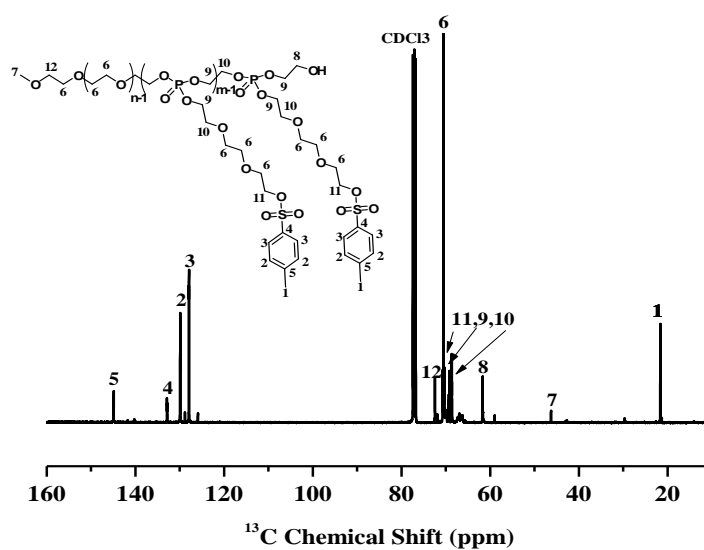


Fig. S2 <sup>13</sup>C NMR spectra of mPEG-*b*-PPMBS

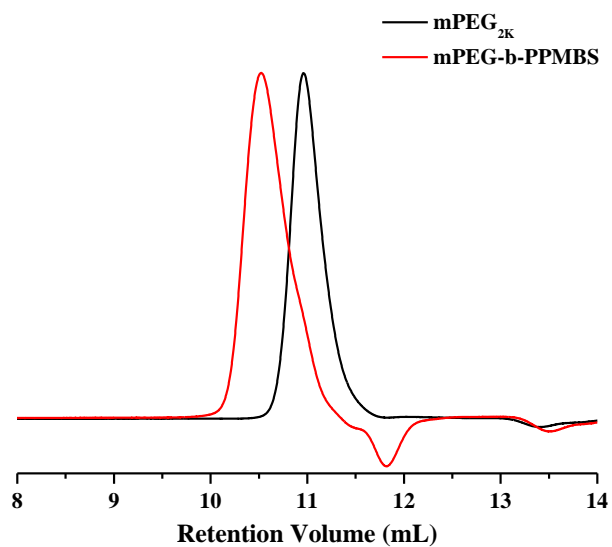


Fig. S3 GPC curves of (a)  $m\text{PEG}_{2k}$  and (b)  $m\text{PEG-b-PPMBS}$

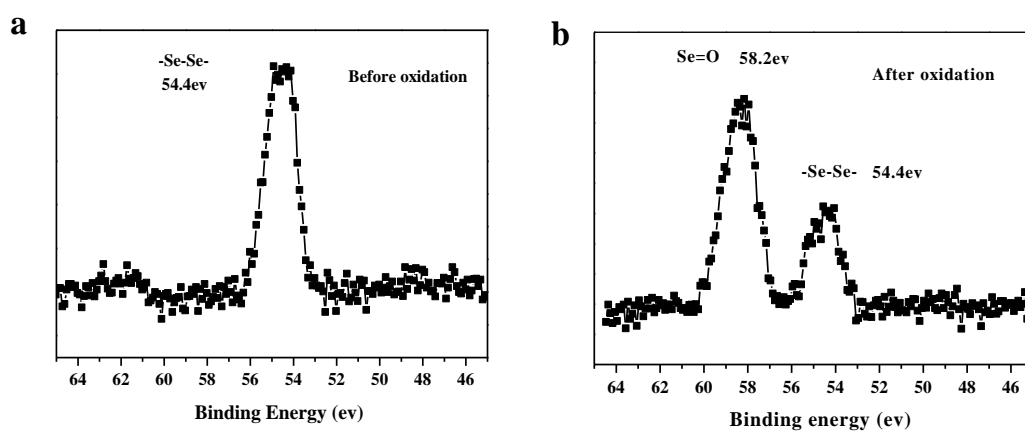
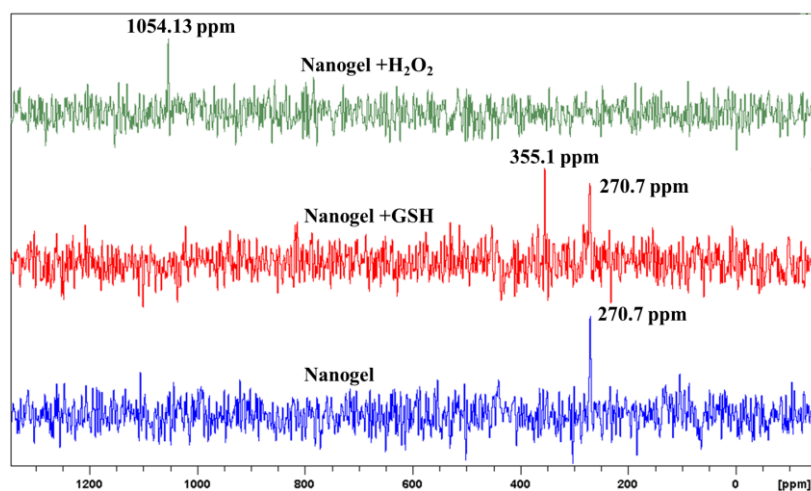


Fig. S4 XPS analysis of PEGylated polyphosphate-diselenide nanogels (a) before and (b) after treatment with 0.1wt%  $\text{H}_2\text{O}_2$



**Fig. S5** <sup>77</sup>Se NMR spectra of the PEGylated poly(diselenide-phosphate) nanogel and that treated with H<sub>2</sub>O<sub>2</sub> and GSH, respectively

As shown in Fig. S5, the signal assigned to diselenide groups at 270.7 ppm was disappeared completely and simultaneously a new signal was found at 1054.3 ppm when the PEGylated poly(diselenide-phosphate) nanogel was incubated with 1wt% H<sub>2</sub>O<sub>2</sub> for 24 h. This indicated that all diselenide bonds in nanogels were broken entirely at such an oxidation environment and changed into the seleninic acid.<sup>1</sup> Meanwhile, when the nanogel was incubated with 100 mM GSH for 24 h, a new signal emerged at 355.1 ppm, which indicated part of diselenide bonds in nanogels were split under the reduction environment and transformed into certain selenol groups. However, due to the intrinsic property of diselenide bond which is more sensitive to the oxidative stimuli than reduction, the signal of diselenide groups at 270.7 ppm was not disappeared completely even with increase of GSH concentration or prolonging the incubation time.

1 N. Ma, H. Xu and X. Zhang, *J. Am. Chem. Soc.*, 2010, **132**, 442-443.