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## Supporting information

Figure S1 is the general mechanism of tetraphenylethane derivative iniferter mechanism, where X can OH, CN, C<sub>2</sub>H<sub>5</sub>, etc.



Figure S1: Tetraphenylethanes based thermal iniferters

The <sup>1</sup>H NMR spectra of P1 and P2 are shown in Figure S2 and Figure S3. Heteronuclear multiple quantum coherence (HMQC) spectra of P1 and P2 are shown in Figure S5 and Figure S6. The main <sup>1</sup> H peaks were labelled with  $H_c$ ,  $H_f$ , and  $H_d$ ; main <sup>C</sup> peaks were labelled with  $C_M$ ,  $C_N$  and  $C_Q$ .



Figure S2: <sup>1</sup>H NMR spectra of P1 (Mn, GPC=56500 g/mol.)



Figure S3: <sup>1</sup>H NMR spectra of P1P2 (Mn, GPC=91000 g/mol.)



Figure S4: The overlay GPC spectra of macroiniferter, P1 and P2 (sample mixtures after 4 hrs reaction without purification.)



Figure S5: HMQC spectra of P1.



Figure S6: HMQC spectra of P2.



Figure S7: Time VS monomer conversion of macroiniferter:NIPAM=1:500 reaction.



Figure S8: Time VS monomer conversion of macroiniferter:NIPAM=1:3000 reaction

The calibration curve is measured according to six different concentrations: 0.5 mg/mL, 0.125 mg/mL, 0.0625 mg/mL, 0.04167 mg/mL, 0.03125 mg/mL and 0.025 mg/mL. Calibration curve is y=0.6457x-0.0012, R2=0.9998, where x is DOX concentration (mg/mL), y is the absorbance at DOX maximum wavelength, as shown in Figure S9.



Figure S9: The calibration curve of free DOX