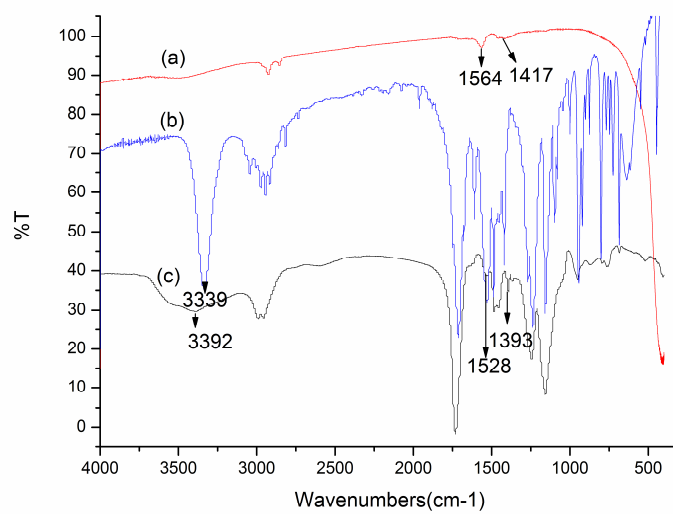
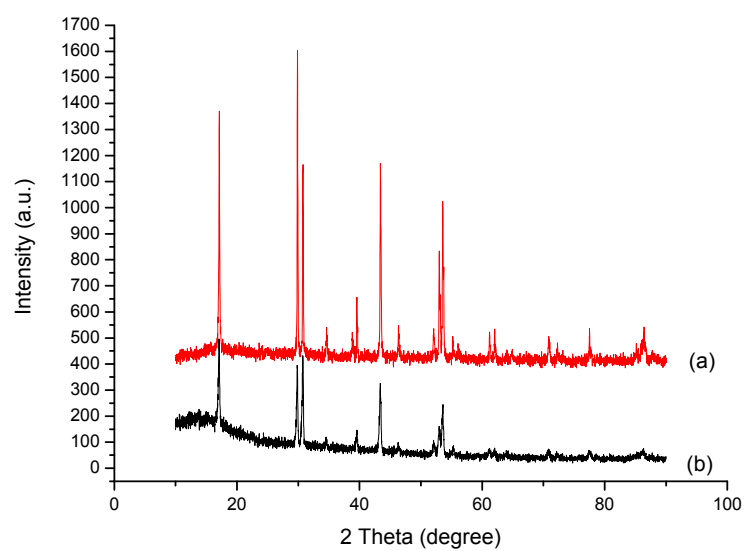


### ***Experimental details***

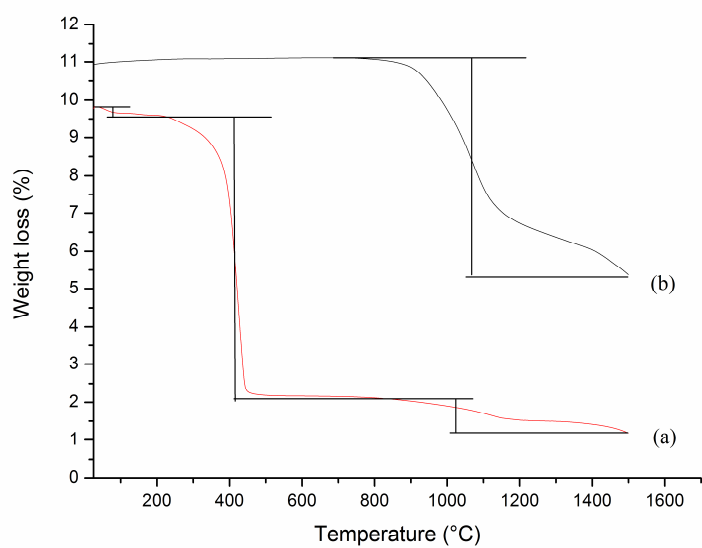
The nanorod structured  $\beta$ -NaYF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup> (2 wt% Er<sup>3+</sup> and 20 wt% Yb<sup>3+</sup> doped NaYF<sub>4</sub>) used in the experiment was prepared by a facile hydrothermal approach. 0.7 g of NaOH, 8.0 mL of oleic acid, and 8.0 mL of ethanol were well mixed at room temperature to form a white viscous solution. Then 5.0 mL H<sub>2</sub>O was poured into the above solution and 8.3 mL (4.80 mmol) of 0.58 mol L<sup>-1</sup> NaF solution was added with vigorous stirring until a translucent solution was obtained. 1.1 mL (0.88 mmol) of 0.80 mol L<sup>-1</sup> Y(NO<sub>3</sub>)<sub>3</sub>, 0.35 mL (0.22 mmol) of 0.63 mol L<sup>-1</sup> Yb(NO<sub>3</sub>)<sub>3</sub> and 0.05 mL (0.02 mmol) of 0.40 mol L<sup>-1</sup> Er(NO<sub>3</sub>)<sub>3</sub> were mixed. After aging for 10 min, the mixture was transferred to a 100 mL Teflon-lined autoclave, the samples were annealed at 230 °C for 12 h to obtain the final hexagonal phase samples. The resulting UCNPs were washed with ethanol, activated with 100 mL Trion X-100/H<sub>2</sub>O (1: 4, v/v), after filtered and dried, the UCNPs were preserved in the desiccator. The synthesis of the UCNPs@MIP was carried out as follows: 0.165 g of metolcarb (1.0 mmol) was dissolved in 2.0 mL tetrahydrofuran (THF) and 2.0 mL toluene, then mixed with 0.340 mL (4.0 mmol) of methacrylic acid (MAA) after 0.250 g UCNPs were added. The mixture was stirred for 60 min before 0.760 mL (4.0 mmol) of cross-linker ethylene glycol dimethacrylate (EGDMA) and 0.02 g of initiator azodiisobutyronitrile (AIBN) were added. After stirring for another 30 min, the mixture was sealed and thermally initiated in a water bath at 60 °C for 20 h. After the polymerization, the template was removed by Soxhlet extraction with 300 mL of methanol/acetic acid (9: 1, v/v) until no analyte was detected using UV spectrometry at 210 nm. For comparison, the UCNPs@NIP was also prepared in the same way in the absence of a template.



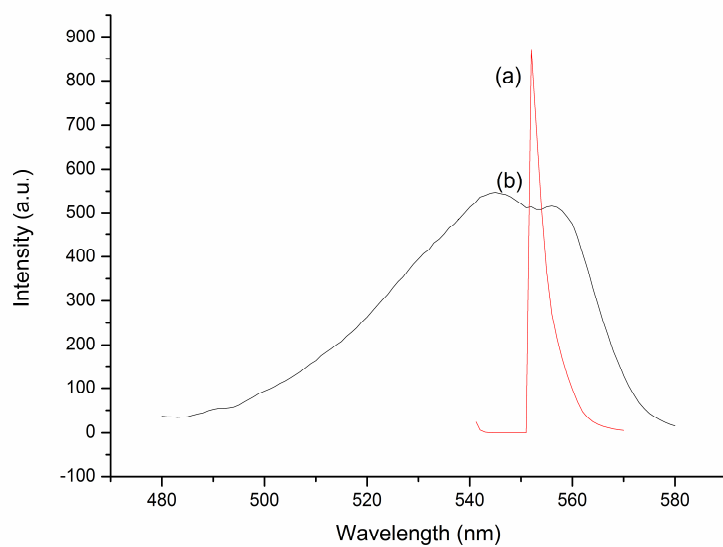
**Fig. S1** FTIR spectrum of (a) UCNPs, (b) metolcarb and (c) UNCPS@MIP (the template not extracted).



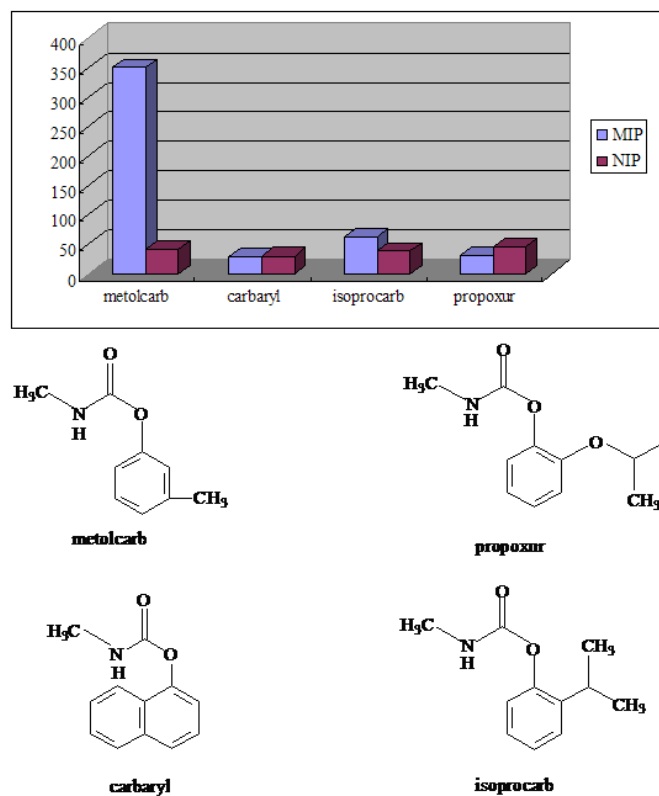
**Fig. S2** Powder X-ray diffraction patterns for (a) UCNPs and (b) UCNPs@MIP.



**Fig. S3** Thermogravimetric analysis for (a) UCNPs and (b) UCNPs@MIP.



**Fig.S4** Spectral overlap: (a) the absorbance spectrum of UCNPs@MIP and (b) the spectrum of metolcarb.



**Fig.S5** Up: Selectivity of UCNPs@MIP and UCNPs@NIP toward metolcarb in individual metolcarb, propoxur, carbaryl and isoprocarb aqueous solution at a concentration of  $1.0 \mu\text{g mL}^{-1}$ . Bottom: The structures of metolcarb, propoxur, carbaryl and isoprocarb.