

## Supplementary Information

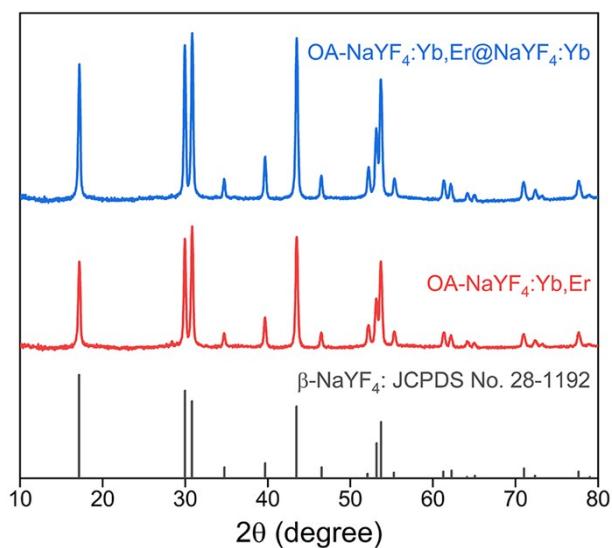
### Fluorescence sensing of cyanide anions based on Au-modified upconversion nanoassemblies

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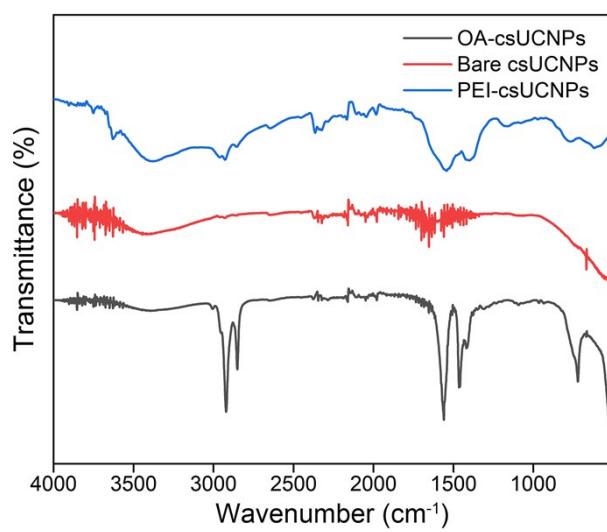
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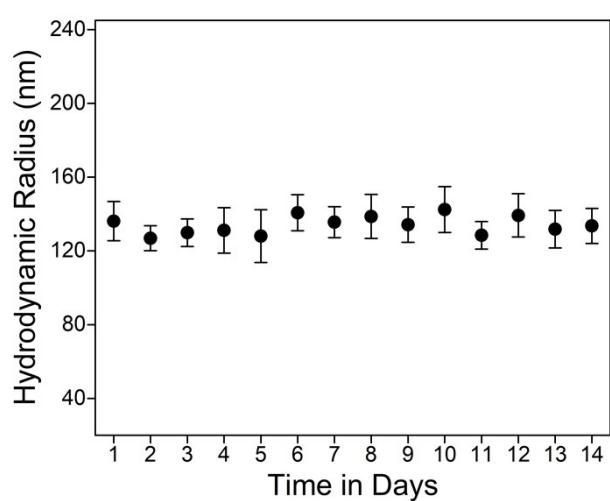
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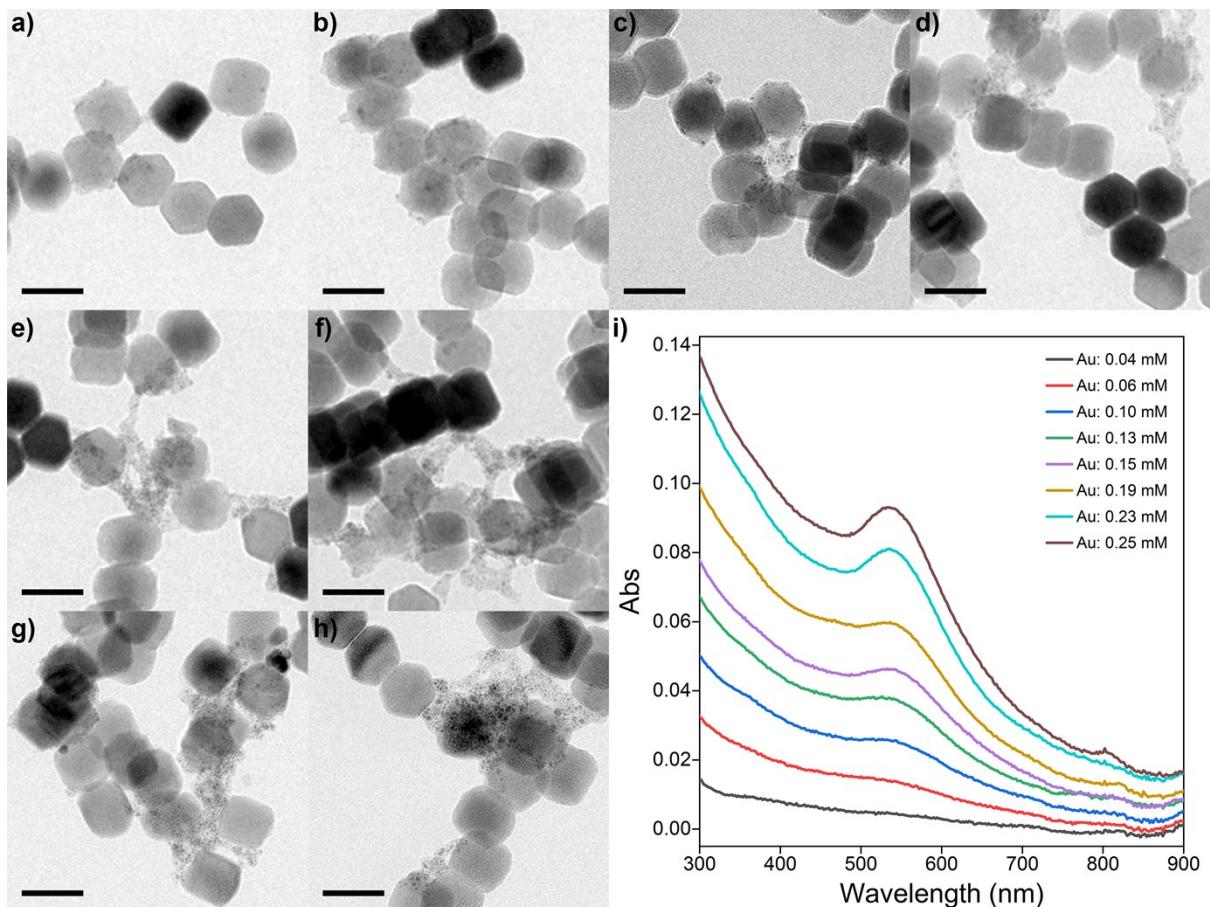
**Fig. S1** XRD patterns of OA-UCNPs, OA-CSUCNPs, and the standard data of hexagonal NaYF<sub>4</sub> (JCPDS No. 28-1192).



**Fig. S2** FT-IR spectra of OA-, ligand-free, and PEI-csUCNPs



**Fig. S3** Hydrodynamic radius of 0.5 mg/mL csUCNPs/Au (0.1 mM Au NPs) nanocomposites with different aging times.



**Fig. S4** TEM images of 0.5 mg/mL csUCNPs/Au nanoassemblies with different contents of Au NPs. (a) 0.04 mM, (b) 0.06 mM, (c) 0.10 mM, (d) 0.13 mM, (e) 0.15 mM, (f) 0.19 mM, (g) 0.23 mM, (h) 0.25 mM. Scale bars: 50 nm. (i) UV-vis absorption spectra of 0.5 mg/mL csUCNPs/Au nanocomposites with different contents of Au NPs.