Electronic Supplementary Information (ESI)

1. Experimental

All reagents are purchased from commercial suppliers and used as received without further purification. The absorption spectra are recorded using a UV-2600 spectrophotometer (Shimadzu) with quartz cuvettes of 1 cm pathlength. Fluorescence spectra are obtained using a LS-55 Fluorescence spectrophotometer (PerkinElmer) at room temperature. The hydrodynamic size distribution and zeta potentials are tested using zetasizer nano series (Malvern) at room temperature. The BET surface areas of DPAS and FAS are measured by Micromeritics (ASAP 2020 HD88) under nitrogen. The SEM pictures of DPAS dots and FAS dots are taken by SU-70 scanning electron microscope (HITACHI).

In AIE property measurement, aliquot 50 μL stock solution (1mM) was added to a 5.0 mL flask with different amount of water and THF following our previous report. The aggregates of FAS and DPAS (AIE dots) were prepared by adding 25μL stock solution to 2.5 mL water in stirring at 1000 r/s for 30 seconds, when their fluorescence gave maximum emission intensity for measurement.

In metal ion sensor measurement, all the ClO$_4^-$ salts of respective ions were dissolved in 5 mL aqueous solution (1 mmol). The spectra of AIE dots without any ions were defined as “Blank” in 2.5 mL water containing 25 μL stock solution, and the different metal ion (about 50 μL) was added to the “Blank” for investigating the selectivity and anti-interference ability. Herein, the concentration of anion is 5 times of that of probe. In fluorescence titration experiment, the concentration of ion was diluted to a specified concentration and added to the mixture gradually in proportion. To the composite probe, the excessive of Cu$^{2+}$ (5 eq) was added to AIE dots above, and the specified concentration of Hg$^{2+}$ injected to the mixture gradually in proportion. Herein, Cu(ClO$_4$)$_2$, Hg(ClO$_4$)$_2$, AgClO$_4$, Fe(ClO$_4$)$_3$, Zn(ClO$_4$)$_2$, Co(ClO$_4$)$_2$, Pb(ClO$_4$)$_2$, Mn(ClO$_4$)$_2$, Ni(ClO$_4$)$_2$, Ba(ClO$_4$)$_2$, Cd(ClO$_4$)$_2$, Mg(ClO$_4$)$_2$, Al(ClO$_4$)$_3$ and Ca(ClO$_4$)$_2$ were chosen.

2. AIE property and application in fluorescence imaging
Fig. S1 (A) the plot of $I/I_0$ versus water fractions of FAS, where $I_0$ is the PL intensity in pure THF solution (Inset: fluorescence image at $f_w = 0\%$ and $f_w \approx 100\%$)

3. The hydrodynamic size distribution and zeta potentials of two AIE dots and them with metal ions

Fig. S2 The diameter distributions of (A) DPAS dots and (B) FAS dots in aqueous solution.

Table S1. The diameters and zeta potentials of two AIE dots

<table>
<thead>
<tr>
<th>Samples</th>
<th>DPAS</th>
<th>FAS</th>
</tr>
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<tbody>
<tr>
<td>Size (nm)</td>
<td>197.6</td>
<td>204.2</td>
</tr>
<tr>
<td>Zeta (mV)</td>
<td>-31.2</td>
<td>-24.9</td>
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4. Crystal structures of FAS and DPAS
Fig. S3 The ORTEP drawings of single crystal structures of (A) FAS (CCDC 1432238) and (B) DPAS (CCDC 1432239). Molecular packing patterns of FAS (C: side view, D: top view) and DPAS (E: side view) in the crystalline state. The fluorescence images of crystals (F) FAS and (G) DPAS. (Copyright 2016 American Chemical Society)

5. The SEM pictures of DPAS dots and FAS dots

Fig. S4 The SEM pictures of (A) DPAS dots and (B, C) the enlarged images; (D) FAS dots and (E, F) the enlarged images in aqueous solution.

6. The effects of pH buffered conditions
Fig. S5 The histogram analysis of DPAS (A-C) and FAS (D-F) dots upon addition of the single anion or the mixture of Cu$^{2+}$ and other interfering ions (5 eq.) at varied pH values of 4.86 (A, D), 7.4 (B, E) and 9.3 (C, F), where $I_0$ is the PL intensity of the naked probe at 565 nm for DPAS or 600 nm for FAS (defined as “Blank”).

7. Metal ions sensing properties of FAS
Fig. S6. (A) The changes of PL spectra of FAS in the mixture ($I_0=100\%$) upon addition of 5 eq respective ions (as their ClO$_4^-$ salts); (B) The histogram analysis of receptor FAS upon addition of the single anion or the mixture of Cu$^{2+}$ and other interfering ions (5 eq), where $I_0$ is the PL intensity of the naked probe at 600 nm (defined as “Blank”); (C) The changes in PL spectra of FAS upon addition of various of Cu$^{2+}$ (from 0 eq to 0.2 eq); (D) The curve of PL change as a function of Cu$^{2+}$ concentrations; (E) The job plot of FAS-Cu$^{2+}$ complexes, where the emission intensity at 600 nm is plotted against mole fraction of FAS; (F) The plot of FAS with Cu$^{2+}$ in $I_0=100\%$ mixture solution. (Inset: A, the fluorescence images of FAS solutions with and without excess Cu$^{2+}$; D, the linear range of PL change).
8. Absorption of fluorescent probes and their complexes

![Absorption spectra graphs](image)

**Fig. S7** The absorption spectra of FAS (A) and DPAS (B) dots in aqueous solution, and the absorption spectra of FAS (C) and DPAS (D) dots in CH$_3$CN solution (upon addition of Cu$^{2+}$ and Hg$^{2+}$).

9. Hg$^{2+}$ sensing properties of FAS
**Fig. S8** (A) The changes in PL spectra of FAS-Cu$^{2+}$ upon addition of various of Hg$^{2+}$ (from 0.1 eq to 1.5 eq); (B) The curve of PL change as a function of Hg$^{2+}$ concentrations. (Inset: A, the fluorescence images of FAS-Cu$^{2+}$ solution with and without excess Hg$^{2+}$; solid-state fluorescence response in test paper of FAS (1 mM), FAS-Cu$^{2+}$ (1:5) and FAS-Cu$^{2+}$-Hg$^{2+}$ (1:5:5) under UV light).

10. **Job plots of FAS-Cu$^{2+}$ and DPAS-Cu$^{2+}$ to Hg$^{2+}$**

**Fig. S9** The job plots of FAS+Cu$^{2+}$/Hg$^{2+}$ (A) and FAS+Cu$^{2+}$/Hg$^{2+}$ (B) complexes, where the emission intensity at 600 nm or 565 nm is plotted against mole fraction of FAS or DPAS.

11. **NMR spectra of DPAS and its complexes**
Fig. S10 $^1$H NMR spectra of DPAS and its possible structure binding with Cu$^{2+}$ (adding 5 eq. of Cu$^{2+}$ in CD$_3$CN) in CD$_3$CN.

Fig. S11 $^1$H NMR spectra of DPAS and its possible structure binding with Hg$^{2+}$ (adding 5 eq. of Hg$^{2+}$ in CD$_3$CN) in CD$_3$CN.