

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 25 μL 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, $\text{Cu}(\text{ClO}_4)_2$, $\text{Hg}(\text{ClO}_4)_2$, AgClO_4 , $\text{Fe}(\text{ClO}_4)_3$, $\text{Zn}(\text{ClO}_4)_2$, $\text{Co}(\text{ClO}_4)_2$, $\text{Pb}(\text{ClO}_4)_2$, $\text{Mn}(\text{ClO}_4)_2$, $\text{Ni}(\text{ClO}_4)_2$, $\text{Ba}(\text{ClO}_4)_2$, $\text{Cd}(\text{ClO}_4)_2$, $\text{Mg}(\text{ClO}_4)_2$, $\text{Al}(\text{ClO}_4)_3$ and $\text{Ca}(\text{ClO}_4)_2$ were chosen.

2. AIE property and application in fluorescence imaging

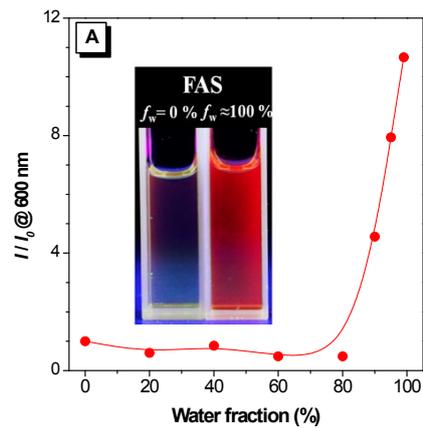


Fig. S1 (A) the plot of I/I_0 versus water fractions of FAS, where I_0 is the PL intensity in pure THF solution (Inset: fluorescence image at $f_w = 0\%$ and $f_w \approx 100\%$)

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

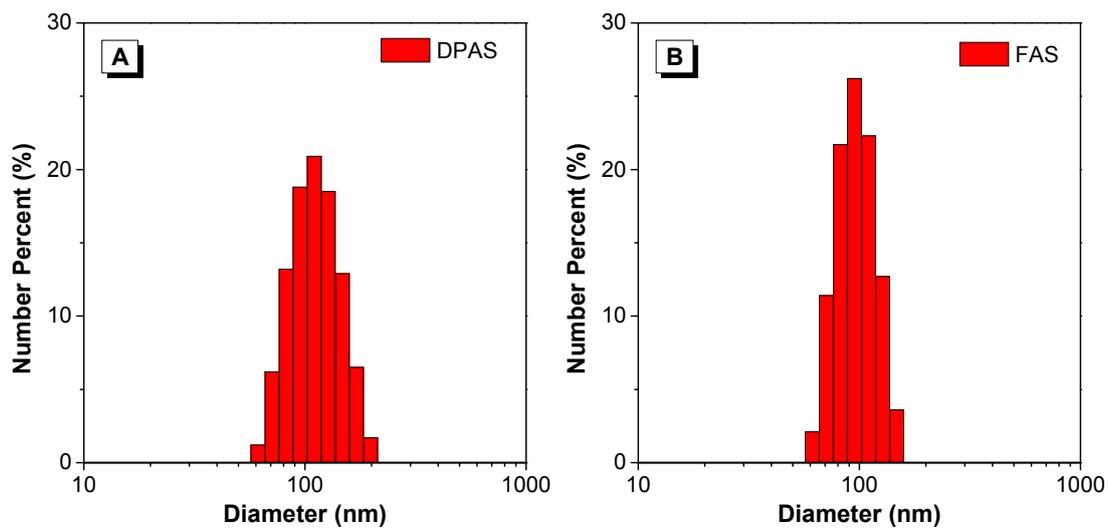


Fig. S2 The diameter distributions of (A) DPAS dots and (B) FAS dots in aqueous solution.

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

Samples	DPAS	FAS
Size (nm)	197.6	204.2
Zeta (mV)	-31.2	-24.9

4. Crystal structures of FAS and DPAS

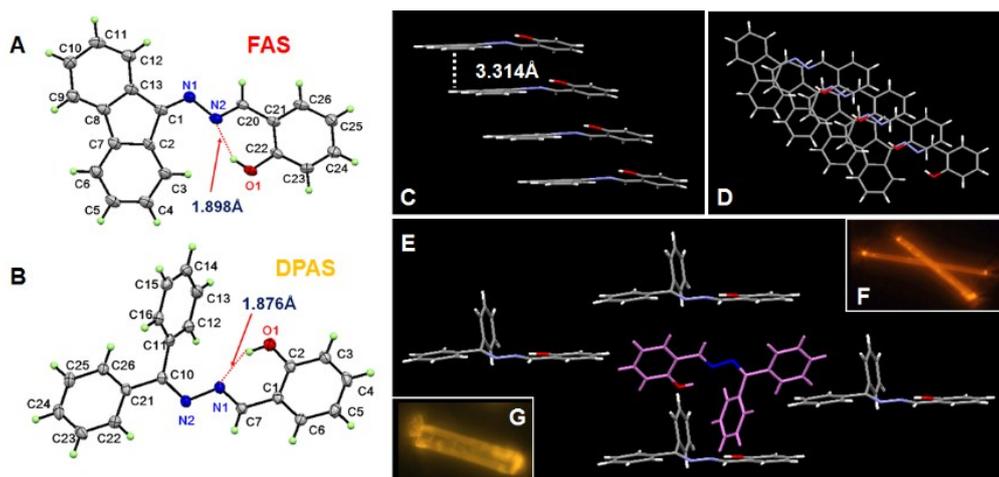


Fig. S3 The ORTEP drawings of single crystal structures of (A) **FAS** (CCDC 1432238) and (B) **DPAS** (CCDC 1432239). Molecular packing patterns of **FAS** (C: side view, D: top view) and **DPAS** (E: side view) in the crystalline state. The fluorescence images of crystals (F) **FAS** and (G) **DPAS**. (Copyright 2016 American Chemical Society)

5. The SEM pictures of DPAS dots and FAS dots

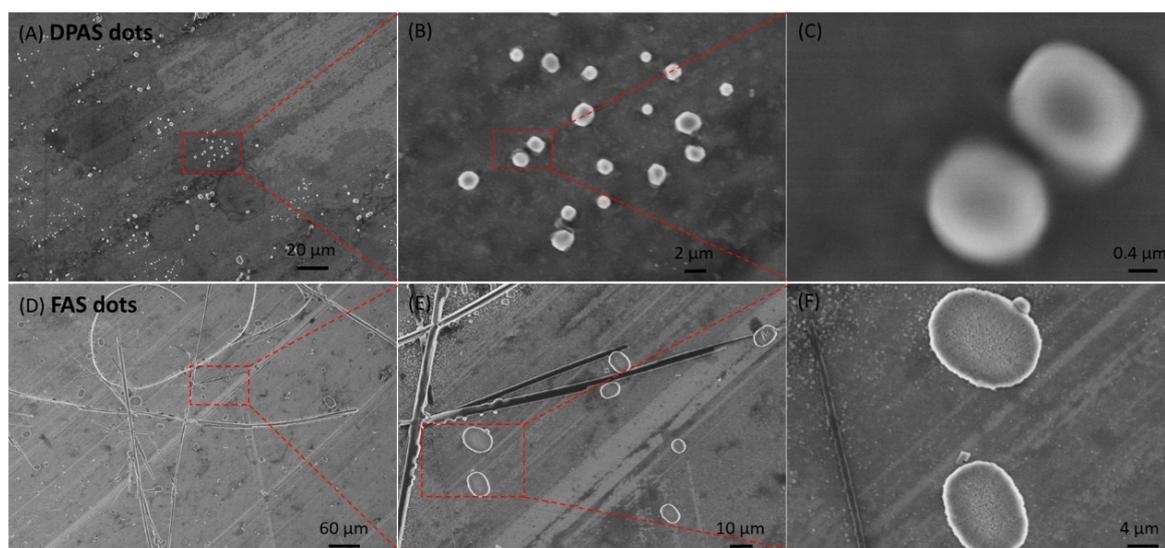


Fig. S4 The SEM pictures of (A) **DPAS** dots and (B, C) the enlarged images; (D) **FAS** dots and (E, F) the enlarged images in aqueous solution.

6. The effects of pH buffered conditions

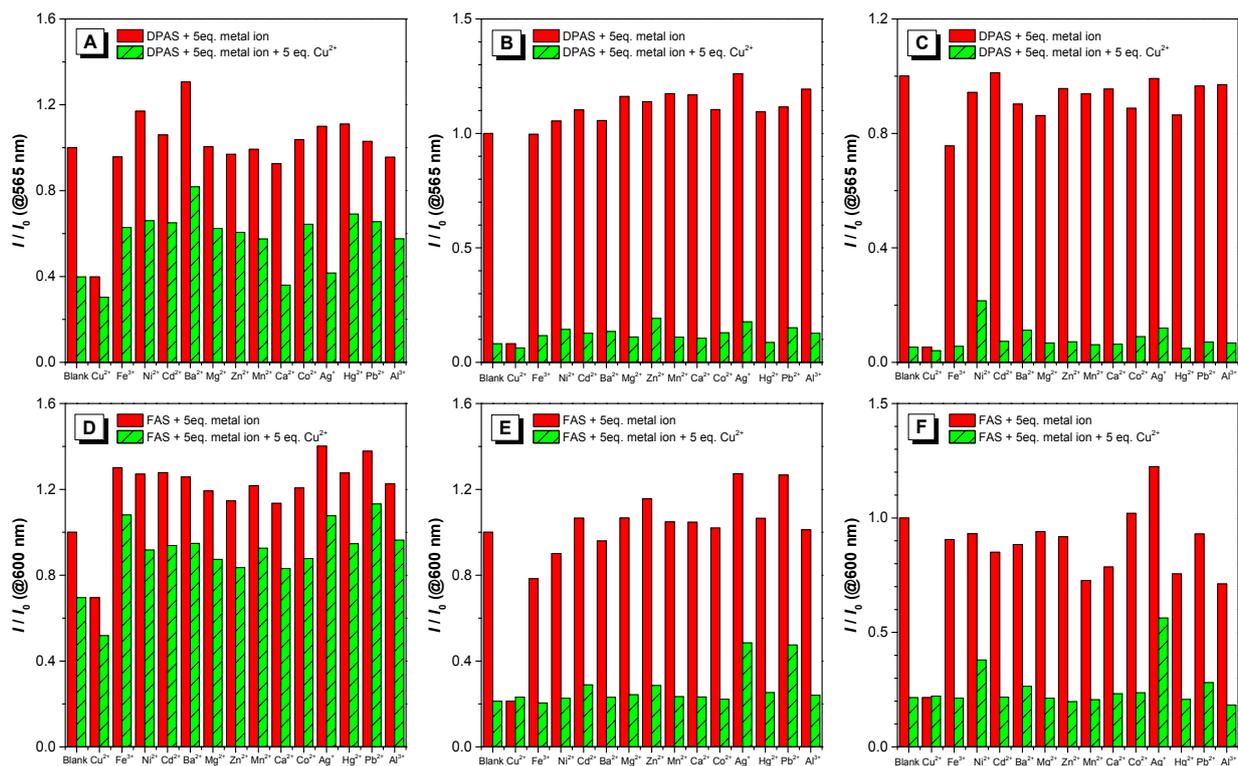


Fig. S5 The histogram analysis of DPAS (A-C) and FAS (D-F) dots upon addition of the single anion or the mixture of Cu^{2+} and other interfering ions (5 eq.) at varied pH values of 4.86 (A, D), 7.4 (B, E) and 9.3 (C, F), where I_0 is the PL intensity of the naked probe at 565 nm for DPAS or 600 nm for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

7. Metal ions sensing properties of FAS

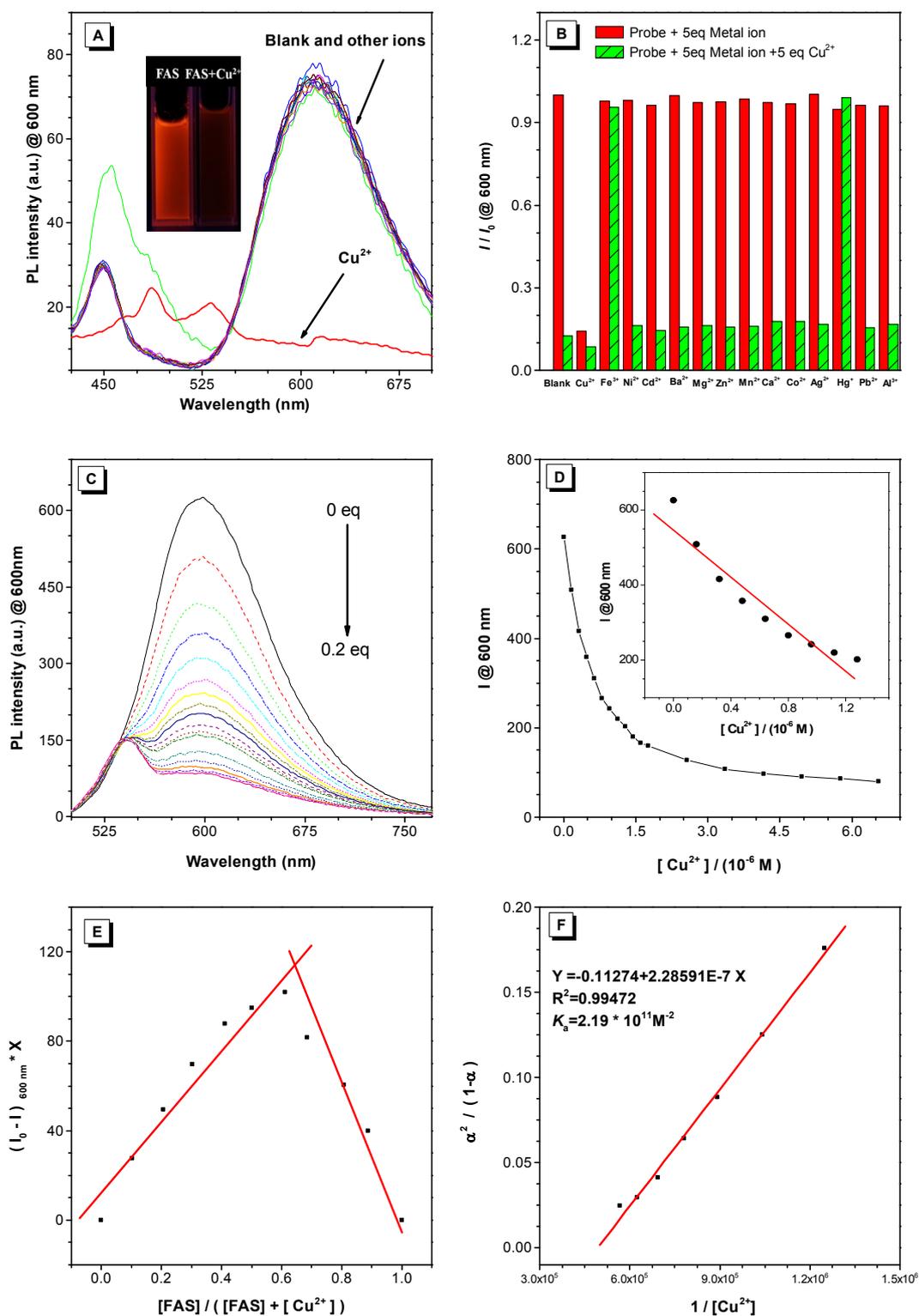


Fig. S6. (A) The changes of PL spectra of **FAS** in the mixture ($f_w \approx 100\%$) upon addition of 5 eq respective ions (as their ClO_4^- salts); (B) The histogram analysis of receptor **FAS** upon addition of the single anion or the mixture of Cu^{2+} and other interfering ions (5 eq), where I_0 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 $f_w \approx 100\%$ 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

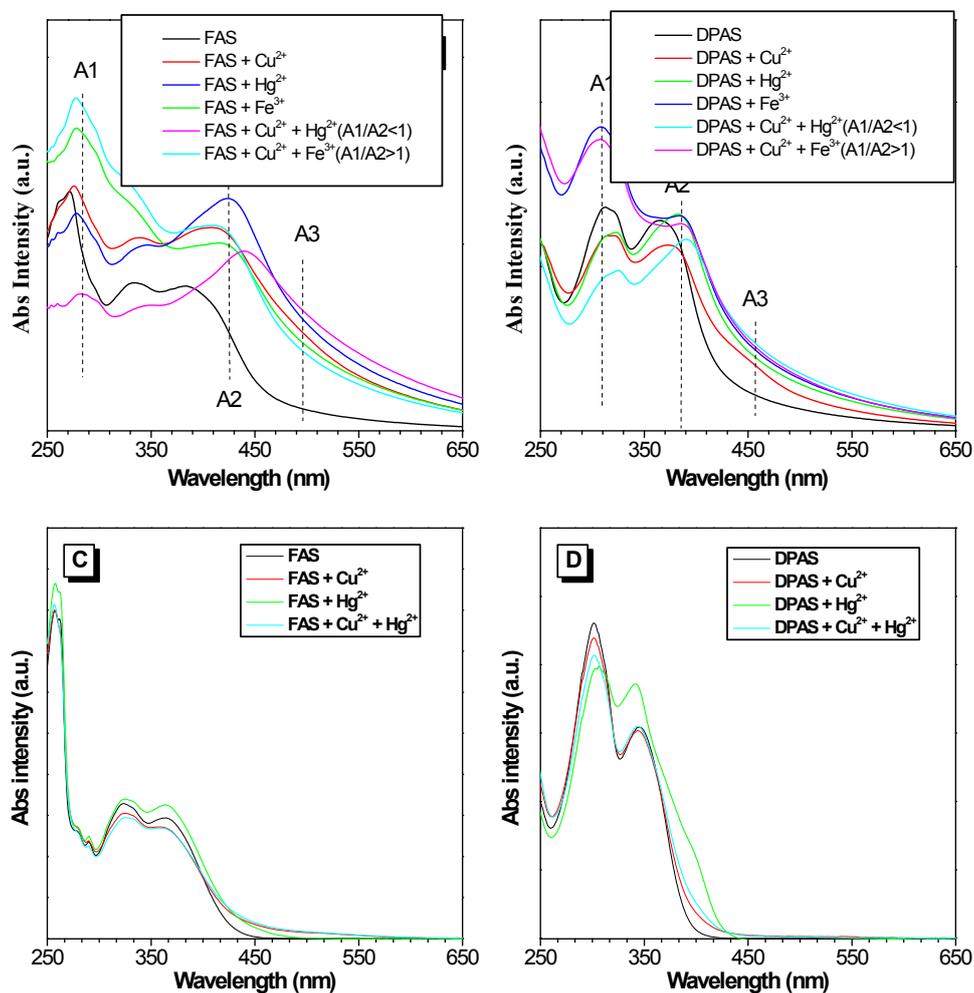


Fig. S7 The absorption spectra of FAS (A) and DPAS (B) dots in aqueous solution, and the absorption spectra of FAS (C) and DPAS (D) dots in CH_3CN solution (upon addition of Cu^{2+} and Hg^{2+}).

9. Hg^{2+} sensing properties of FAS

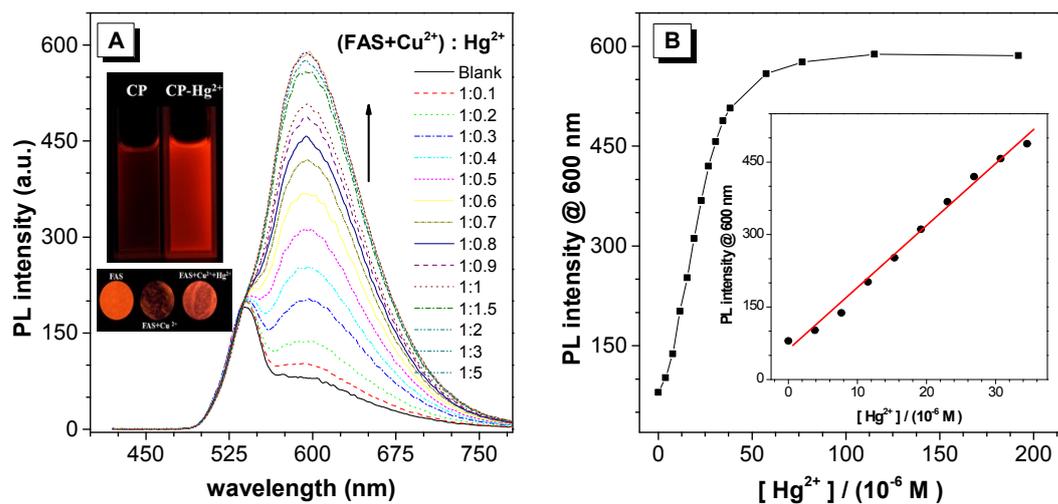


Fig. S8 (A) The changes in PL spectra of **FAS-Cu²⁺** upon addition of various of **Hg²⁺** (from 0.1 eq to 1.5 eq); (B) The curve of PL change as a function of **Hg²⁺** concentrations. (Inset: A, the fluorescence images of **FAS-Cu²⁺** solution with and without excess **Hg²⁺**; solid-state fluorescence response in test paper of **FAS** (1 mM), **FAS-Cu²⁺** (1:5) and **FAS-Cu²⁺-Hg²⁺** (1:5:5) under UV light).

10. Job plots of **FAS-Cu²⁺** and **DPAS-Cu²⁺** to **Hg²⁺**

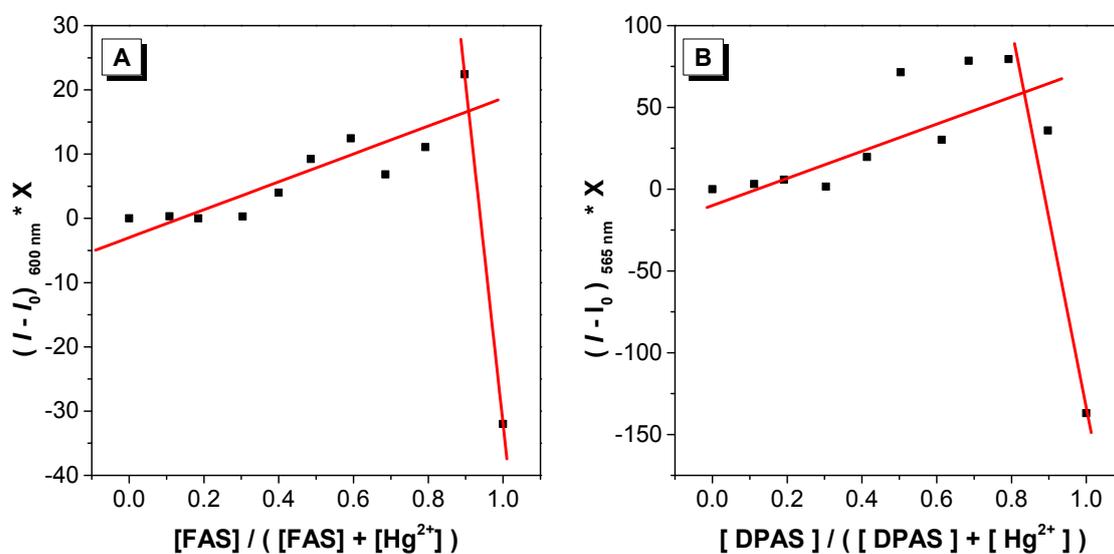


Fig. S9 The *job plots* of **FAS+Cu²⁺/Hg²⁺** (A) and **FAS+Cu²⁺/Hg²⁺** (B) complexes, where the emission intensity at 600 nm or 565 nm is plotted against mole fraction of **FAS** or **DPAS**.

11. NMR spectra of **DPAS** and its complexes

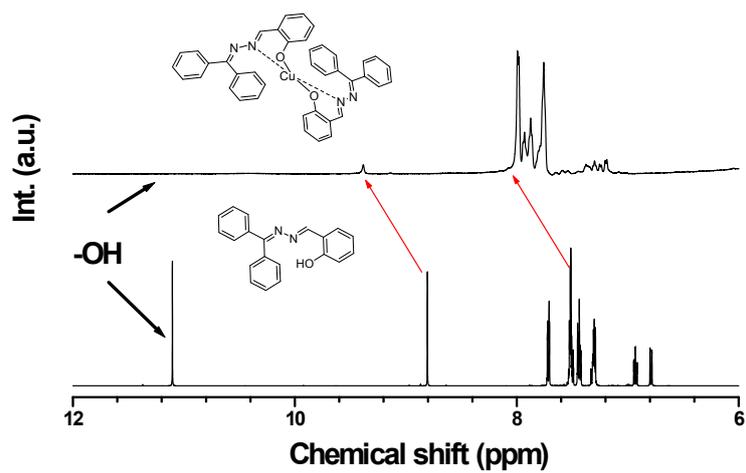


Fig. S10 ¹H NMR spectra of DPAS and its possible structure binding with Cu²⁺ (adding 5 eq. of Cu²⁺ in CD₃CN) in CD₃CN

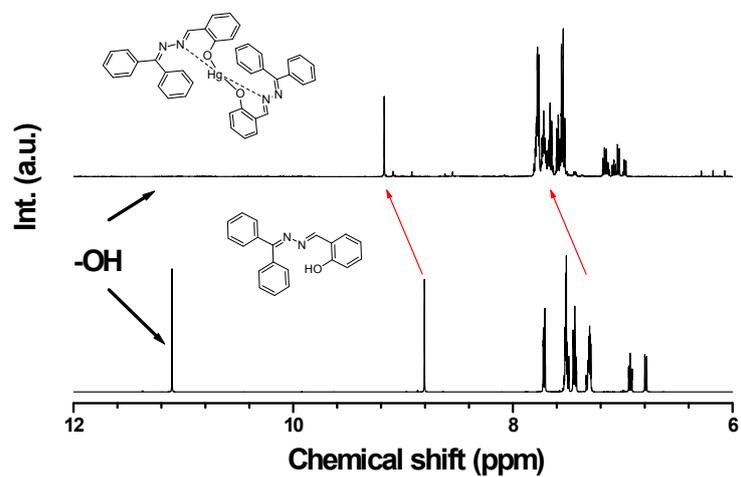


Fig. S11 ¹H NMR spectra of DPAS and its possible structure binding with Hg²⁺ (adding 5 eq. of Hg²⁺ in CD₃CN) in CD₃CN.