

## *Supporting Information*

### Acyldrazone functionalized naphthalene-based self-assembled supramolecular gels for efficient fluorescence detection of Fe<sup>3+</sup>

Jutao Liu<sup>a‡</sup>, Shang Wu<sup>a‡\*</sup>, Shuaishuai Fu<sup>a</sup>, Jiajia Wang<sup>a</sup>, Penghui Zhang<sup>a</sup>, Yanbin Wang<sup>a\*</sup>, Chen Chen<sup>a</sup>, Xiangfei Zhao<sup>a</sup>, Zhenhua Li<sup>a</sup>, Quanlu Yang<sup>b\*</sup>

<sup>a</sup> Key Laboratory for Utility of Environmental Friendly Composite Materials and Biomass in University of Gansu Province, Key Laboratory of Environment-Friendly Composites of the State Ethnic Affairs Commission, Gansu Provincial Biomass Function Composites Engineering Research Center, College of Chemical Engineering, Northwest Minzu University, Lanzhou, Gansu 730030, P. R. China.

<sup>b</sup> College of Chemical Engineering, Lanzhou University of Arts and Science, Beimiantan 400, Lanzhou, Gansu 730000, P. R. China.

<sup>‡</sup>These authors contributed equally to this work. They should thus be considered co-first authors.

\*Corresponding authors. E-mail: chwush84@163.com (S. Wu), 1278616076@qq.com (Y. Wang), yangquanlu2002@163.com (Q. Yang).

## Table of contents

<b>1. Materials and methods.....</b>	<b>3</b>
<b>2. General procedure.....</b>	<b>3-4</b>
<b>3. Experimental Section.....</b>	<b>5-11</b>

## 1. Materials and methods

All reagents and raw materials were purchased from commercial suppliers and used without any handling, unless otherwise stated. All metal ions were prepared from the perchlorate salts. NMR spectra of all compounds was tested on Varian 400 MHz and Varian 600 MHz instruments. Electrospray ionisation mass spectrometry (ESI-MS) was measured on a Bruker Esquire 6000 mass spectrometer. Powder X-ray diffractometer (XRD) was performed on an EMPYREAN diffractometer (copper target;  $\lambda = 1.540598 \text{ \AA}$ ). **N**, **ON** and **ON-Fe** morphologies (SEM) was carried out on Gemini Sigma 300. The fluorescence emission spectra was recorded on a Shimadzu RF-5301PC fluorescence spectrometer. Infrared spectroscopy (FT-IR) was obtained by a JASCO FT/IR-660 Plus infrared spectrometer. The element diagram and band diagram of **ON** and **ON-Fe** were acquired by a ESCALAB 250Xi X-ray photoelectron spectrometer (XPS).

## 2. General procedure

### 2.1 The gelation test

2 mg of compound **N** was added to a 2 mL (13 mm  $\times$  35 mm) glass vial, and then 200  $\mu$ L of solvent was added and warmed over a heater ( $> 80 \text{ }^\circ\text{C}$ ) until the solid was completely dissolved. The vial was then cooled down naturally to room temperature ( $25 \text{ }^\circ\text{C}$ ). The gel **ON** was formed if there is no significant flow, when the vial is inverted (counter-flow method).

### 2.2 Fluorescence spectral properties

Gel **ON** fluorescence detection test was recorded on a Shimadzu RF-5301PC fluorescence spectrometer test. The solution of  $\text{Zn}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Pd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cr}^{3+}\text{Cu}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Bi}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Pb}^{2+}$ , and  $\text{La}^{2+}$  ( $c = 1 \text{ M}$ , in aqueous medium, 0.7 equivalent) was infused into **ON** at room temperature ( $25 \text{ }^\circ\text{C}$ ). The mixed was heated until it was completely dissolved, followed by cooling to obtain a stable metal gel. The data of fluorescence emission intensity was recorded at  $\lambda_{\text{ex}}$

= 400 nm.

### **2.3 Research of infrared spectroscopy**

Infrared spectral data of the desired test substance was recorded on a JASCO FT/IR-660 Plus FT-IR spectrometer. The samples are dried in an oven. Testing by KBr compression method.

### **2.4 Study of field emission scanning electron microscope (FE-SEM)**

The microscopic morphology of **N**, **ON** and **ON-Fe** was tested in a Gemini Sigma 300 FE-SEM microscope. The samples were laid flat on conductive plastic and sprayed with gold powder. The system was evacuated. FE-SEM images were obtained at an accelerating voltage of 3.0 kV.

### 3. Experimental Section

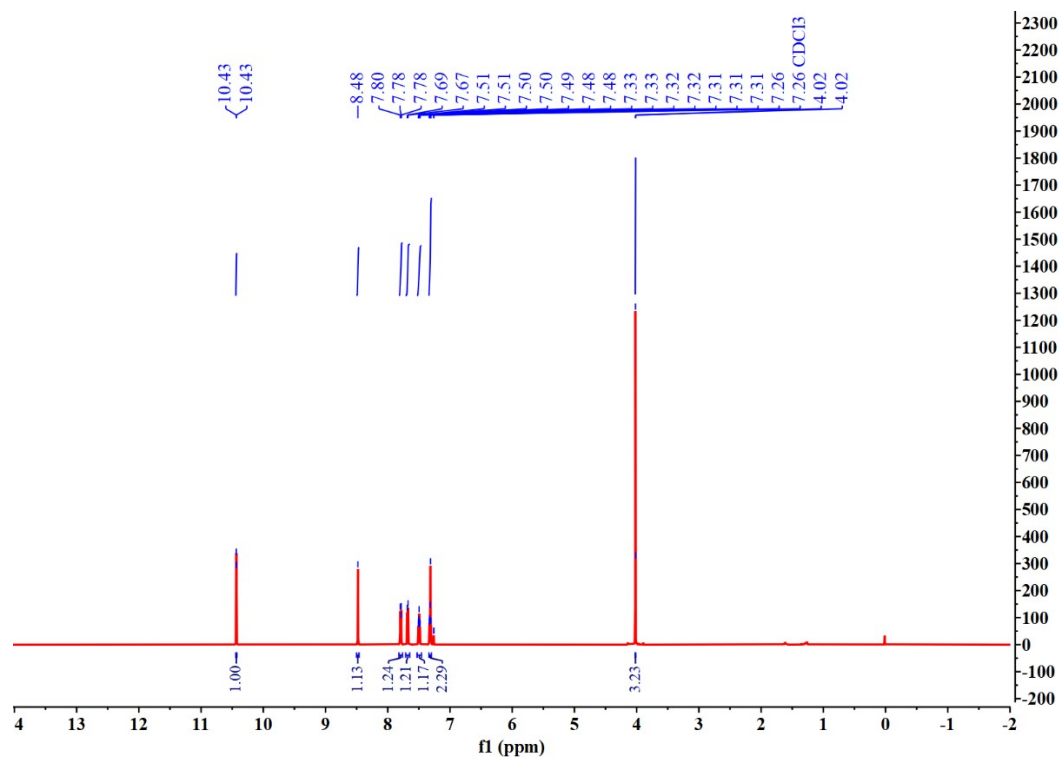


Fig. S1 <sup>1</sup>H NMR spectra of A in Chloroform-*d* (600 MHz, 298K).

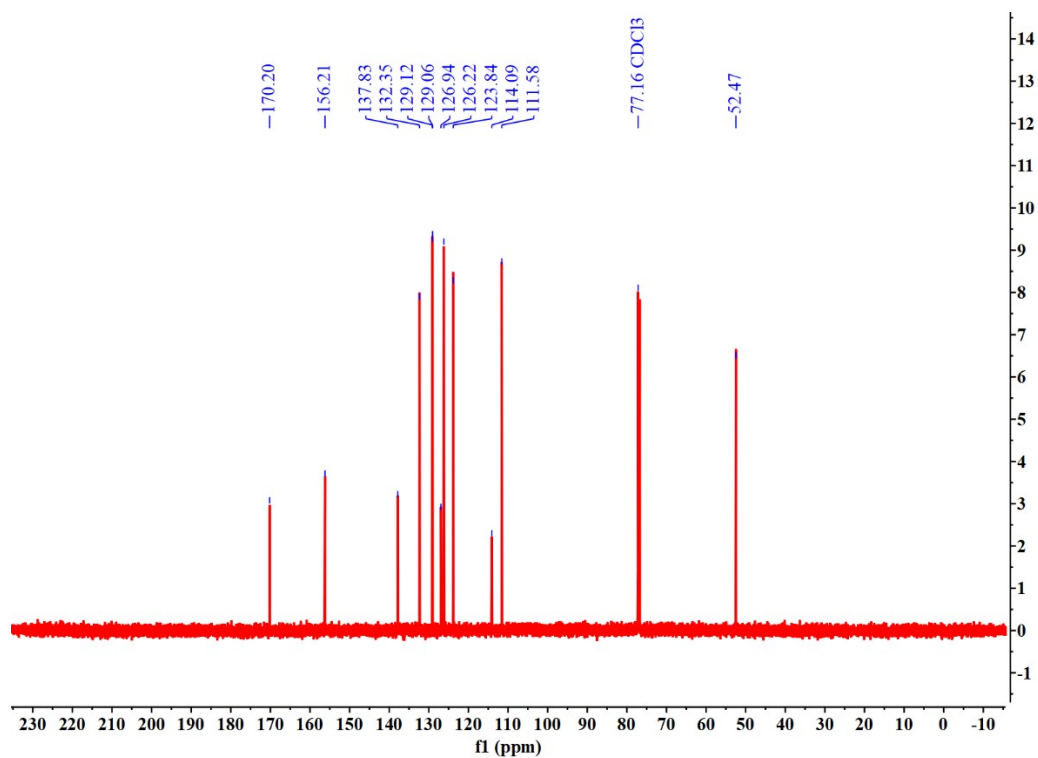


Fig. S2 <sup>13</sup>C NMR spectra of A in Chloroform-*d* (151 MHz, 298K).

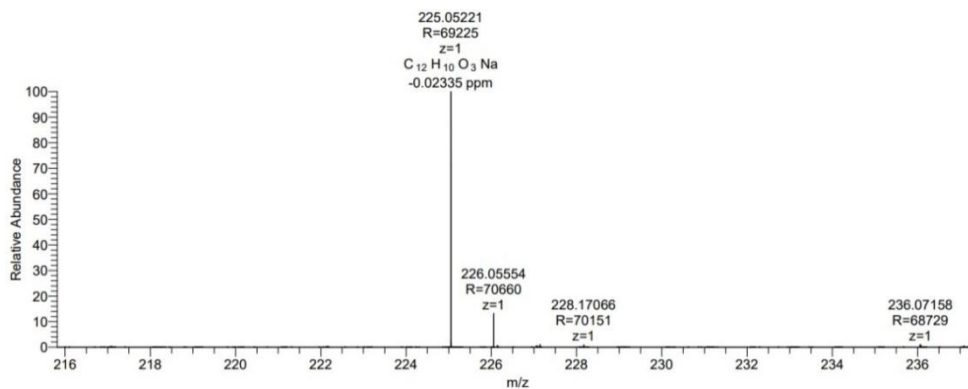


Fig. S3 ESI-MS of A.

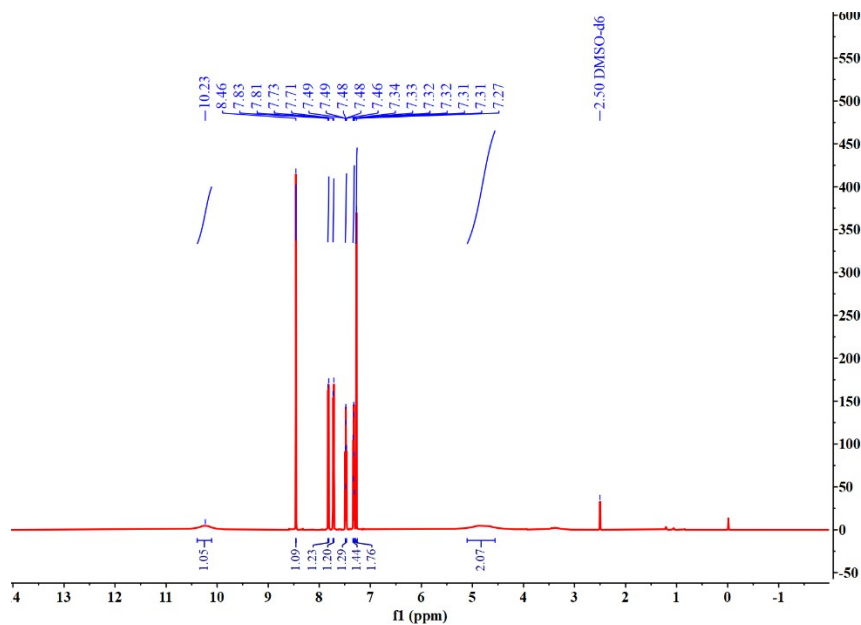


Fig. S4 <sup>1</sup>H NMR spectra of B in DMSO-*d*<sub>6</sub> (600 MHz, 298K).

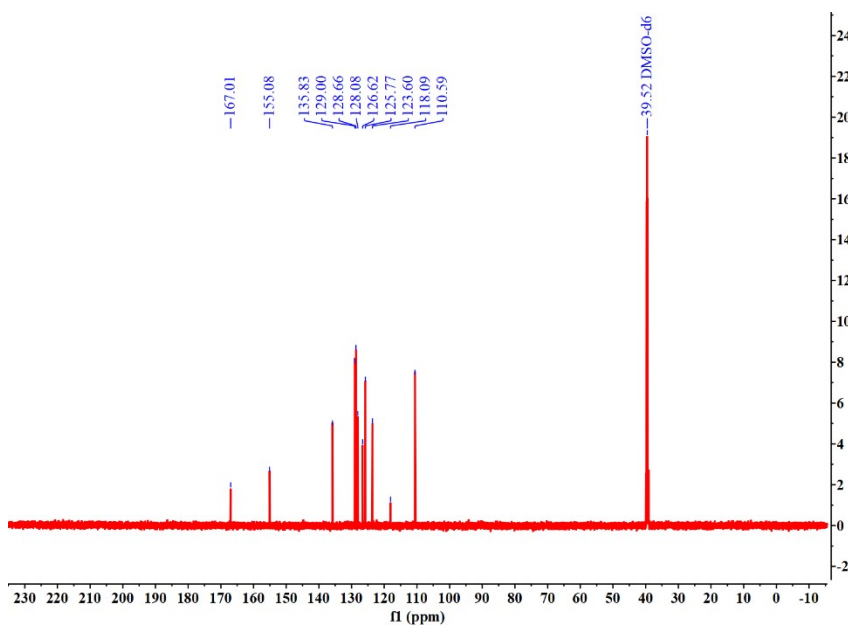


Fig. S5 <sup>13</sup>C NMR spectra of B in DMSO-*d*<sub>6</sub> (151 MHz, 298K).

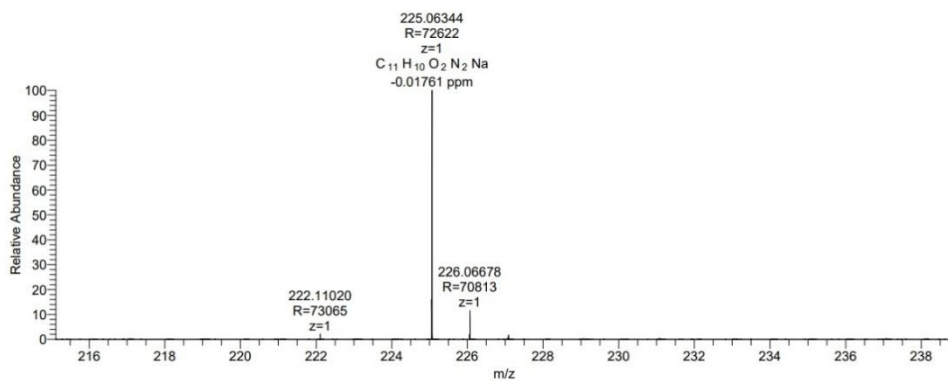


Fig. S6 ESI-MS of B.

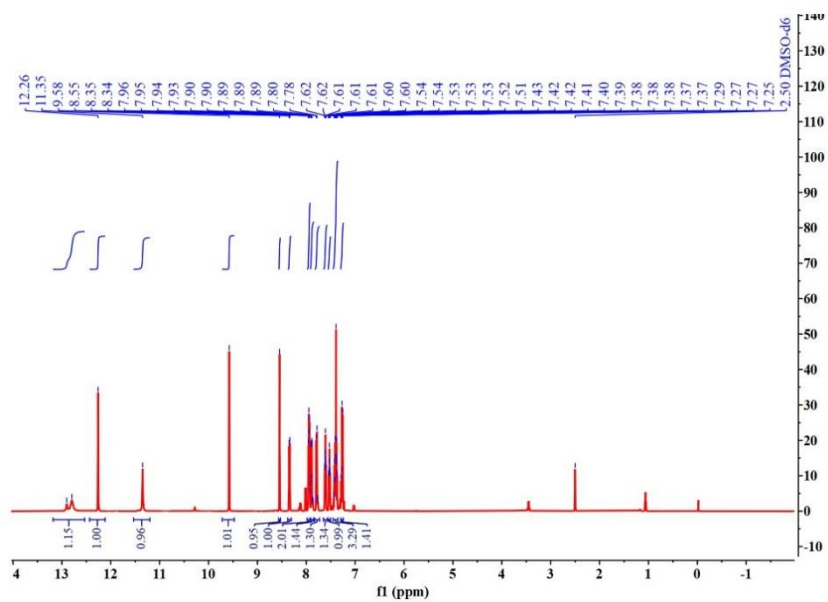


Fig. S7 <sup>1</sup>H NMR spectra of N in DMSO-*d*<sub>6</sub> (600 MHz, 298K).

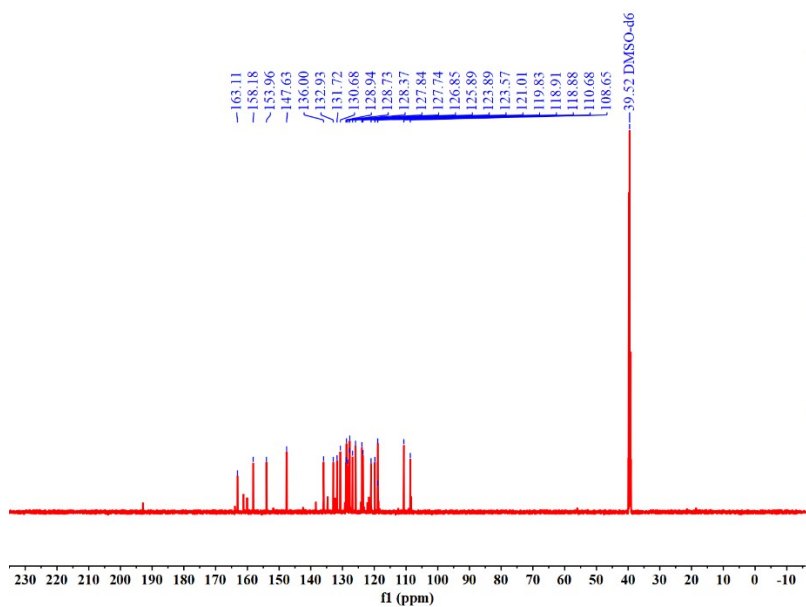
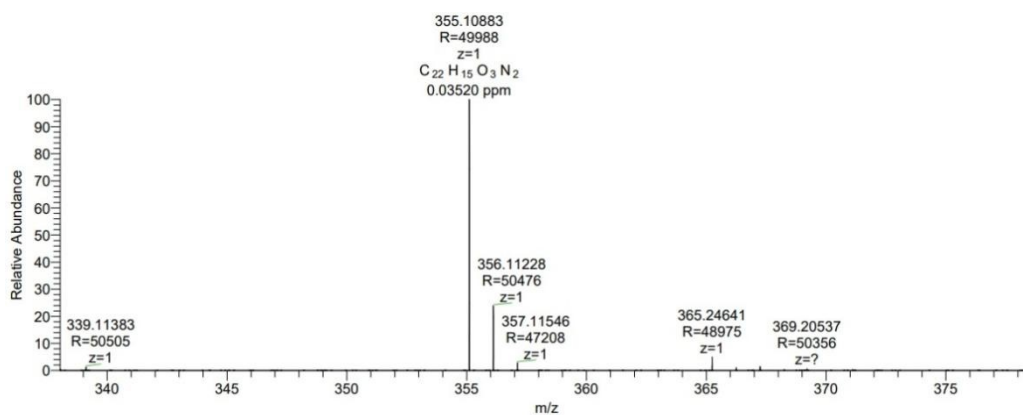


Fig. S8 <sup>13</sup>C NMR spectra of N in DMSO-*d*<sub>6</sub> (151 MHz, 298K).



**Fig. S9** ESI-MS of **N**.

**Table S1.** Gelation properties of **N**

	Solvent	State <sup>a</sup>	CGC <sup>b</sup> (%)	T <sub>gel</sub> <sup>c</sup> (°C)
1	Acetonitrile	SP	\	\
2	EtOH	SP	\	\
3	i-PrOH	SP	\	\
4	n-BuOH	S	\	\
5	DMF	S	\	\
6	CH <sub>2</sub> Cl <sub>2</sub>	SP	\	\
7	1,2-Propanediol	SP	\	\
8	CHCl <sub>3</sub>	SP	\	\
9	Acetone	SP	\	\
10	DMSO	S	\	\
11	DMSO: H <sub>2</sub> O (V: V = 4:1)	G	1	55
12	CCl <sub>4</sub>	SP	\	\
13	MeOH	SP	\	\

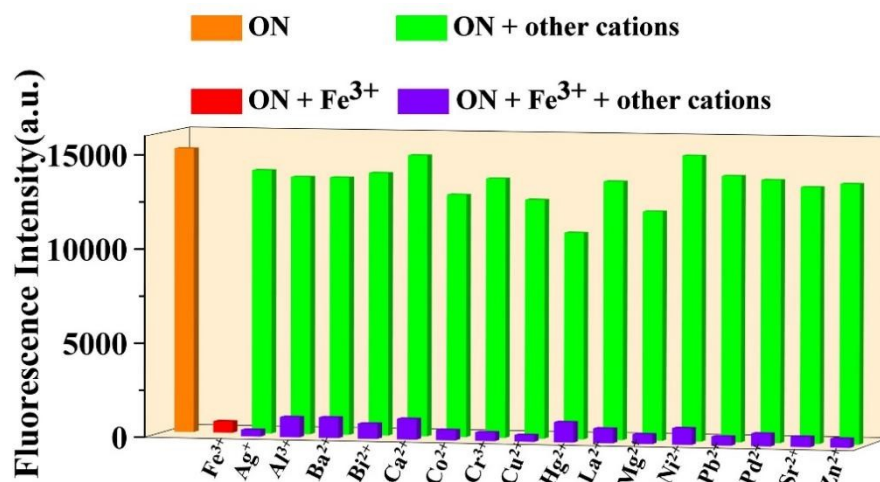
a G, SP and S denote gelation, solution precipitate and solution, respectively.

b The critical gelation concentration (wt %, 10 mg/ml = 1.0 %).

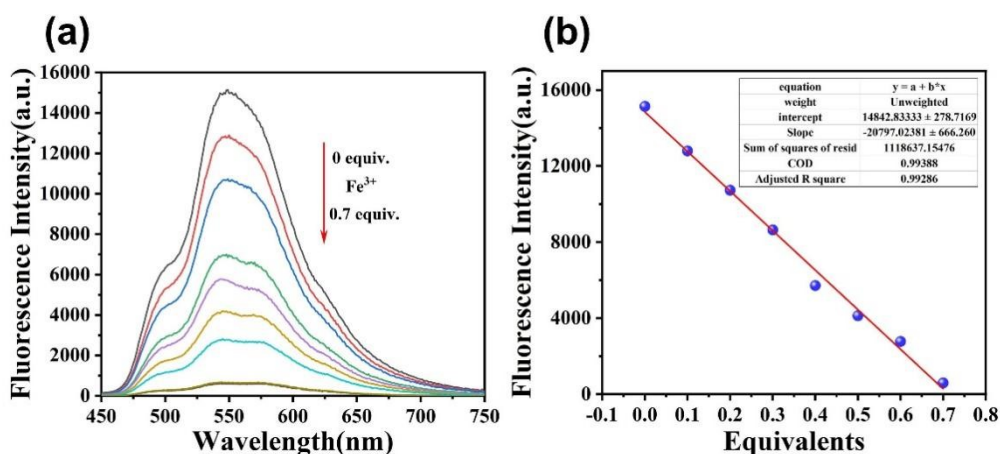
c The gelation temperature (°C).

/ The compound **N** form solution precipitate or solution without critical gel concentration (CGC) or gel-sol transition temperature (T<sub>gel</sub>).





**Fig. S10** Fluorescence of the supramolecular gel **ON** at 549 nm with addition of 0.7 equiv. of  $\text{Fe}^{3+}$  in the presence of 0.7 equiv. of other cations in dimethyl sulfoxide (DMSO) and water (V: V = 4: 1) mixed solvents.



**Fig. S11** (a) Fluorescence spectra of **ON** ( $1 \times 10^{-4}$  M) in the presence of different concentrations of  $\text{Fe}^{3+}$  (0-0.7 equiv.) in dimethyl sulfoxide (DMSO) and water (V: V = 4: 1) mixed solvents, ( $\lambda_{\text{ex}} = 400$  nm). (b) Fluorescence intensity versus concentration plot of **ON** ( $1 \times 10^{-4}$  M) upon the addition of various concentrations of  $\text{Fe}^{3+}$  to **ON** in dimethyl sulfoxide (DMSO) and water (V: V = 4: 1) mixed solvents.

Calculation formula of LOD

$$\text{Linear Equation: } Y = -20797.02381X + 14842.83333$$

$$S = a \times 10^6 = 20797.02381 \times 10^6$$

$$\delta = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \quad (n=20) = 904.42$$

$$K = 3$$

$$\text{LOD} = K \times \frac{\delta}{S} = 1.30 \times 10^{-7} \text{ M}$$

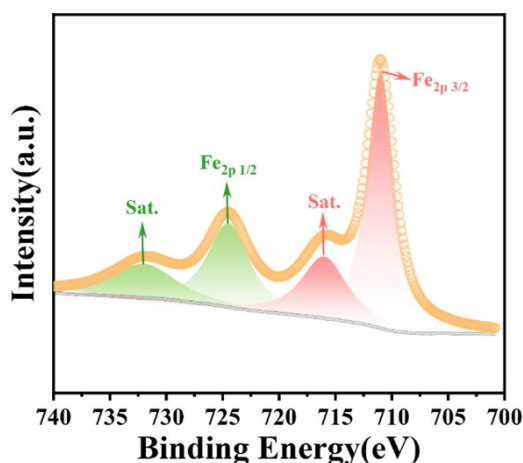


Fig. S12 XPS spectra of Fe<sub>2p</sub> (Fe-ON).

**Table S2.** Comparison of the detection limits of different fluorescent sensors for ions.

Selectivity	Solvent	LOD (M)	References
Fe <sup>3+</sup>	EtOH	$4.0 \times 10^{-7}$	[S1]
Fe <sup>3+</sup>	H <sub>2</sub> O	$9.00 \times 10^{-7}$	[S2]
Fe <sup>3+</sup>	Tris-HCl	$2.0 \times 10^{-7}$	[S3]
Fe <sup>3+</sup>	DMSO: H <sub>2</sub> O	$1.81 \times 10^{-7}$	[S4]
Fe <sup>3+</sup>	EtOH: H <sub>2</sub> O	$2.5 \times 10^{-7}$	[S5]
Fe <sup>3+</sup>	H <sub>2</sub> O	$5.20 \times 10^{-7}$	[S6]
Fe <sup>3+</sup>	THF: H <sub>2</sub> O	$5.15 \times 10^{-7}$	[S7]
Fe <sup>3+</sup>	H <sub>2</sub> O	$2.00 \times 10^{-7}$	[S8]
Fe <sup>3+</sup>	THF: H <sub>2</sub> O	$8.25 \times 10^{-6}$	[S9]
Fe <sup>3+</sup>	H <sub>2</sub> O	$1.32 \times 10^{-6}$	[S10]
Fe <sup>3+</sup>	H <sub>2</sub> O	$3.40 \times 10^{-7}$	[S11]
Fe <sup>3+</sup>	DMSO: H <sub>2</sub> O	$1.81 \times 10^{-6}$	[S12]
<b>Fe<sup>3+</sup></b>	<b>DMSO: H<sub>2</sub>O</b>	<b><math>1.30 \times 10^{-7}</math></b>	<b>This work</b>

### Reference

- [S1] P. Miao, Y. Tang, K. Han, B. Wang, *J. Mater. Chem. A.*, 2015, **3**, 15068-15073.
- [S2] C. Yang, H. Ren, X. Yan, *Anal. Chem.*, 2013, **85**, 7441-7446.
- [S3] R. Guo, S. Zhou, Y. Li, X. Li, L. Fan, N. Voelcker, *ACS Appl. Mater. Interfaces*, 2015, **7**, 23958-23966.
- [S4] X. Wu, S. Zhang, Q. Niu, T. Li, *Tetrahedron Lett.*, 2016, **30**, 3407-3411.
- [S5] T. Zhou, X. Chen, Q. Hua, W. Lei, Q. Hao, B. Zhou, C. Su, X. Bao, *Sens. Actuators B*, 2017, **253**, 292-301.
- [S6] Y. Song, C. Zhu, J. Song, H. Li, D. Du, Y. Lin, *ACS Appl. Mater. Interfaces*, 2017, **9**, 7399-7405.
- [S7] B. Shen, Y. Qian, *J. Mater. Chem. B*, 2016, **4**, 7549-7559.
- [S8] K. Rajar, E. Alveroglu, *Inorg. Mater. Res. Bull.*, 2020, **124**, 110748.

- [S9] C. Guo, Y. Zhou, X. Chen, Y. Wang, J. Shan, J. Hou, *Colloid Surf. A-Physicochem. Eng. Asp.*, 2023, **663**, 131045.
- [S10] X. Cui, Z. Si, Y. Li, Q. Duan, *Dyes Pigment.*, 2020, **173**, 107873.
- [S11] X. Wang, J. Tang, X. Ma, D. Wu, J. Yang, *Crystengcomm*, 2021, **23**, 1243-1250.
- [S12] P. Rasin, M. Mathew, V. Manakkadan, V. Palakkeezhillam, A. Sreekanth, *J. Fluoresc.*, 2022, **32**, 1229-1238.