

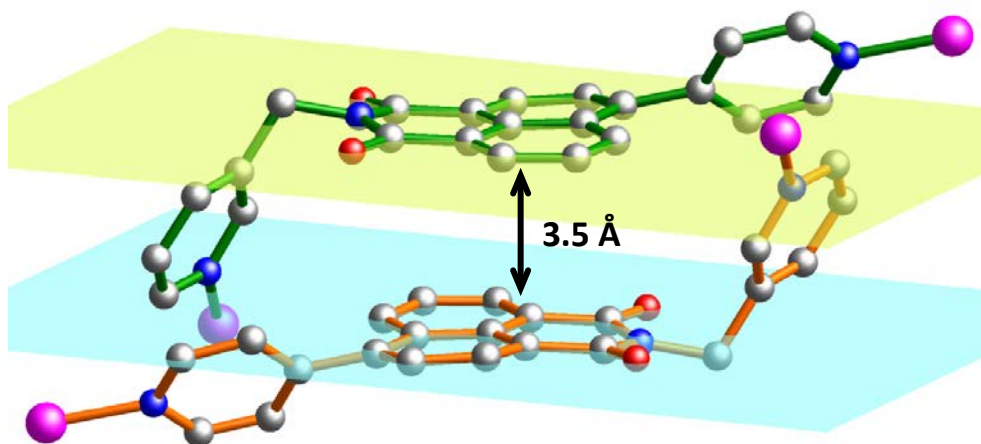
## Supporting Information

### **A Water-Stable 2-Fold Interpenetrating cds Net as a Bifunctional Fluorescence-Responsive Sensor for Selective Detection of Cr(III) and Cr(VI) Ions**

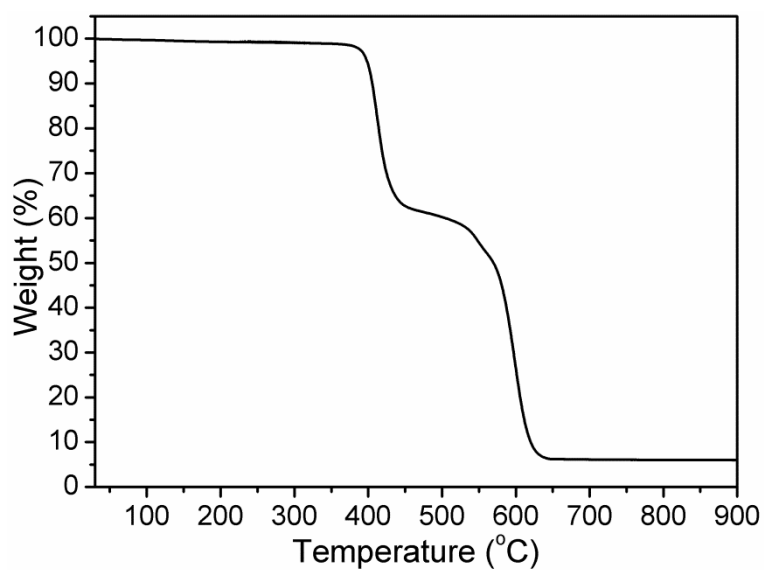
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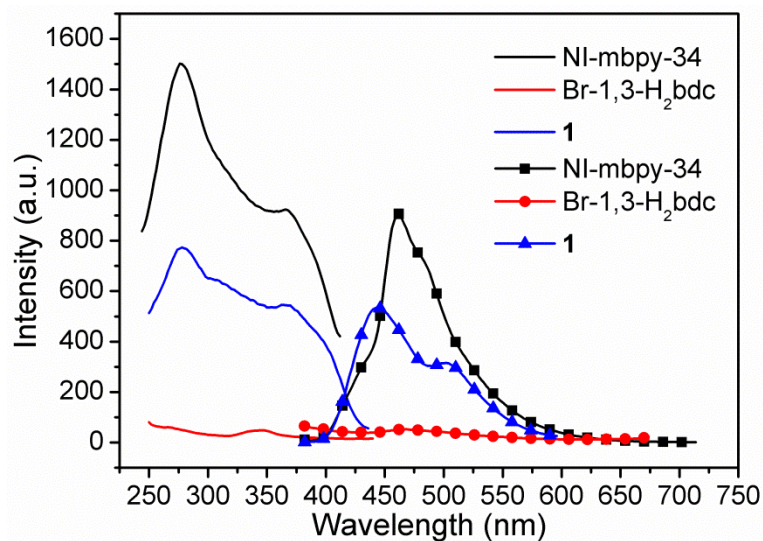
[jyunwu@ncnu.edu.tw](mailto:jyunwu@ncnu.edu.tw)



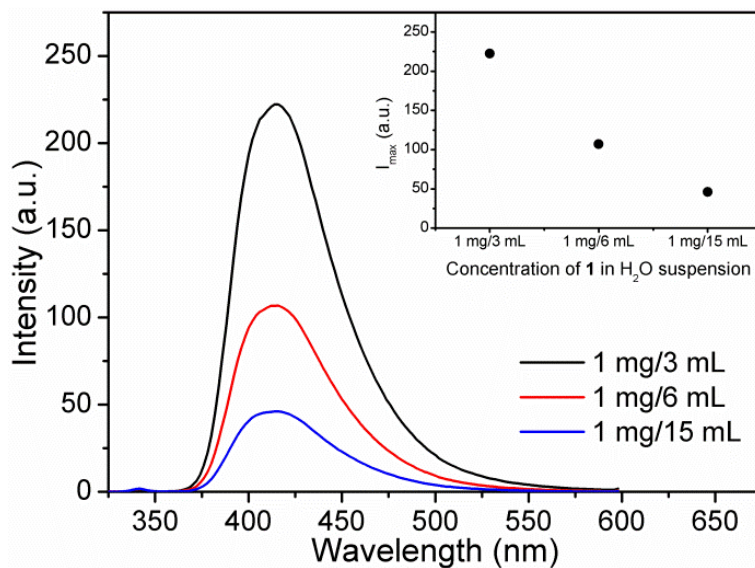
**Figure S1.** Plot of the inter-net  $\pi$ – $\pi$  interactions between two neighboring naphthalimide skeletons in the two independent identical **cds** frameworks in the crystal structure of **1**.



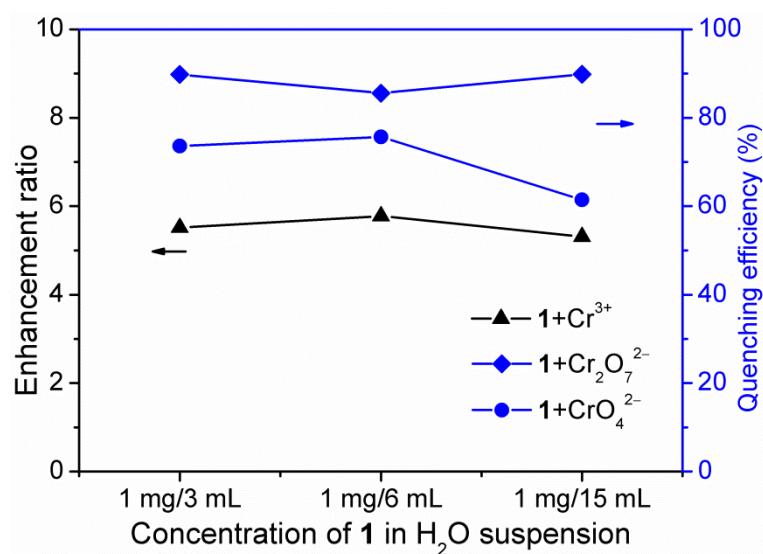
**Figure S2.** TG curve of **1**.



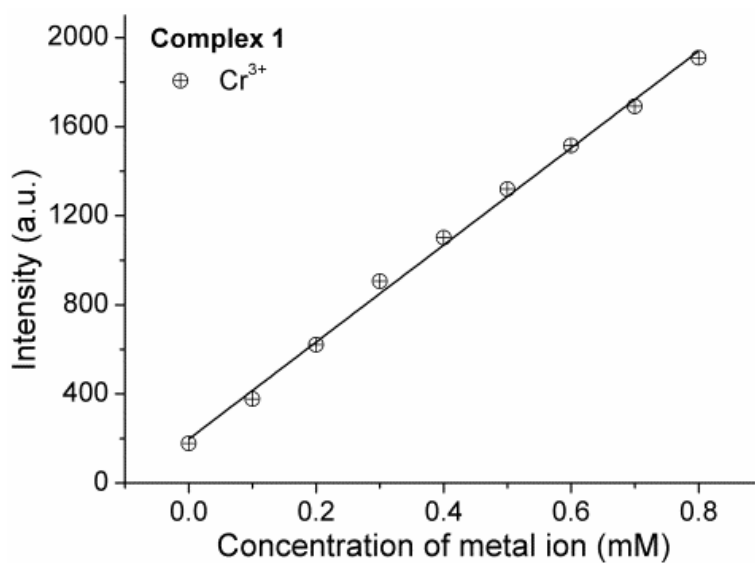
**Figure S3.** Solid-state excitation (solid lines) and emission spectra (solid lines with symbols) of NI-mbpy-34, Br-1,3-H<sub>2</sub>bdc, and **1** at room temperature.



**Figure S4.** Fluorescence emission spectra of **1** in H<sub>2</sub>O suspensions of different concentrations at room temperature upon excitation at 306 nm. Inset: Fluorescence relative ratio responses of **1** in H<sub>2</sub>O suspensions of different concentrations.

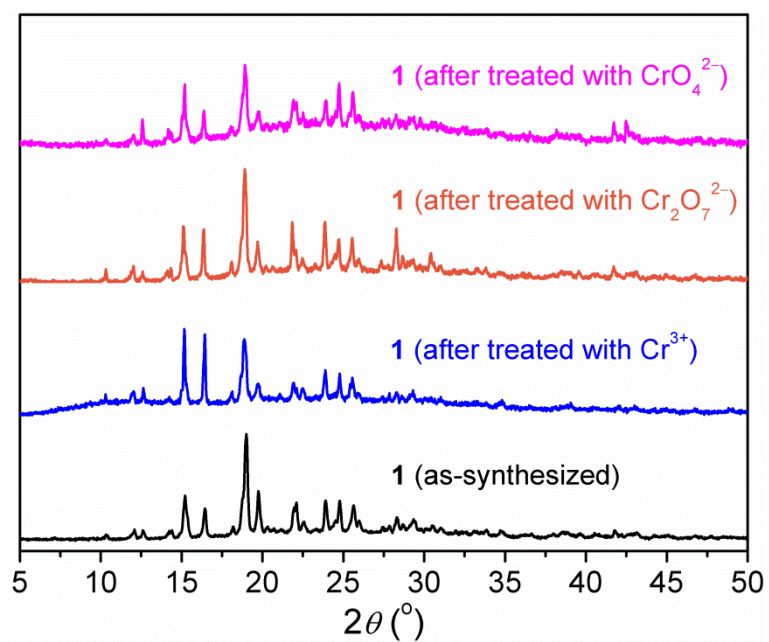


**Figure S5.** Effect of the concentration of **1** in H<sub>2</sub>O suspension on the fluorescence intensity responses upon addition of Cr<sup>3+</sup> ions (enhancement) and Cr(VI) anions (quenching) at 1.0 mM.

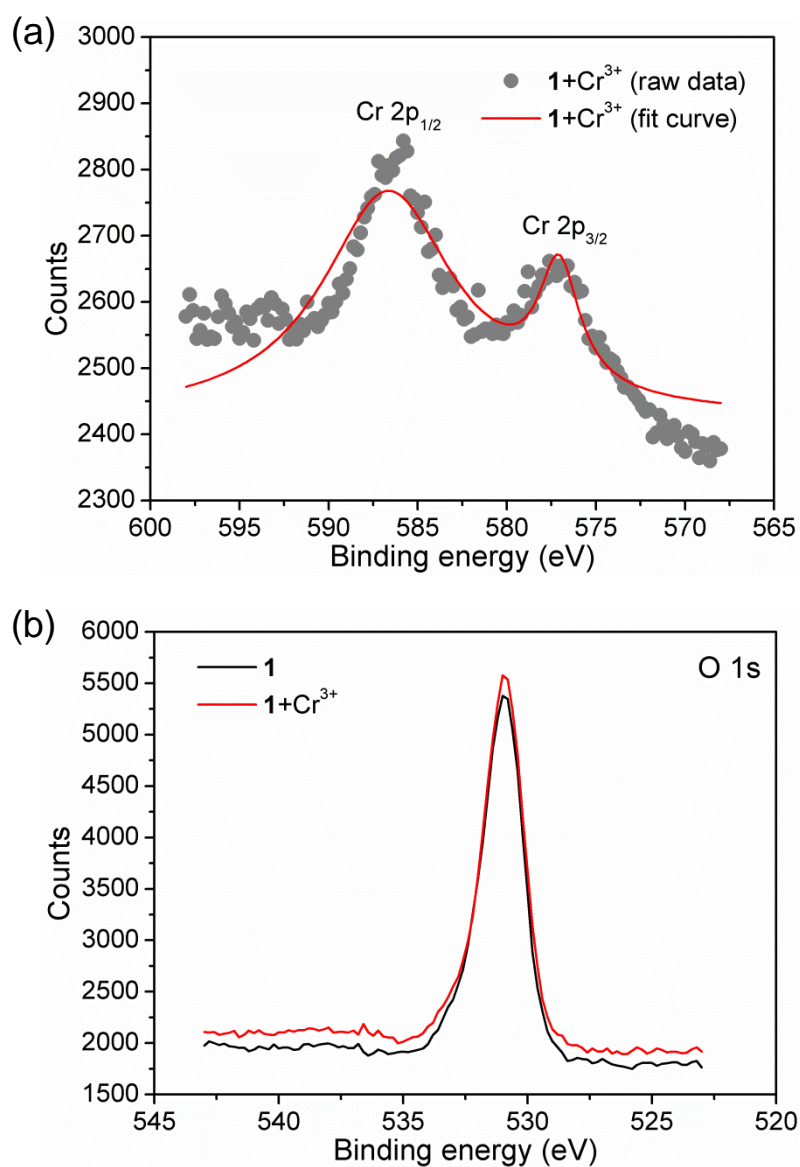


Blank readings	Cr <sup>3+</sup>
#1	177.8
#2	176.9
#3	174.2
#4	174.5
#5	172.1
Standard deviation ( $\sigma$ )	2.27
Slope ( $k$ )	2178.4
$R^2$	0.99631
LOD ( $3\sigma/k$ ), $\mu\text{M}$ (ppb)	3.13 (162.9)

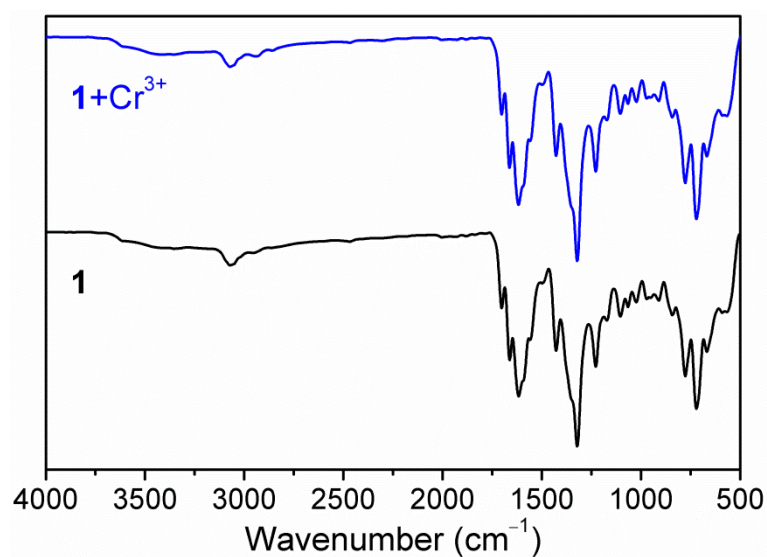
**Figure S6.** Linear region of fluorescence intensity for the H<sub>2</sub>O suspensions of complex **1** upon incremental addition of Cr<sup>3+</sup> ions. The following table lists the relevant parameters of LOD for the H<sub>2</sub>O suspensions of complex **1** toward Cr<sup>3+</sup> ions. Conditions:  $\lambda_{\text{em}} = 414 \text{ nm}$  ( $\lambda_{\text{ex}} = 280 \text{ nm}$ ).



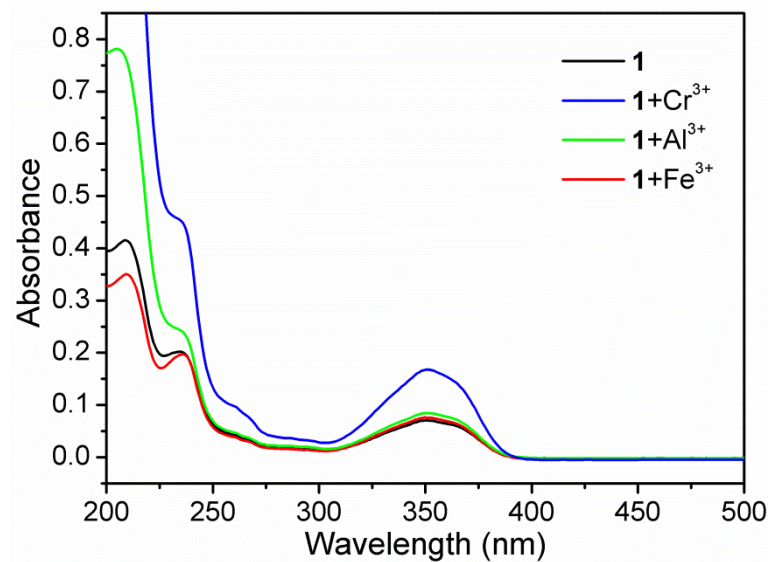
**Figure S7.** XRPD patterns of **1** before and after immersing in  $\text{Cr}^{3+}$ ,  $\text{Cr}_2\text{O}_7^{2-}$ , and  $\text{CrO}_4^{2-}$  aqueous solutions for 24 h.



**Figure S8.** (a) XPS high resolution spectra of Cr 2p for **1** after sensing  $\text{Cr}^{3+}$ . (b) XPS high resolution spectra of O 1s for **1** before and after sensing  $\text{Cr}^{3+}$ .



**Figure S9.** IR spectra of **1** before and after immersing in Cr<sup>3+</sup> aqueous solution for 24 h.

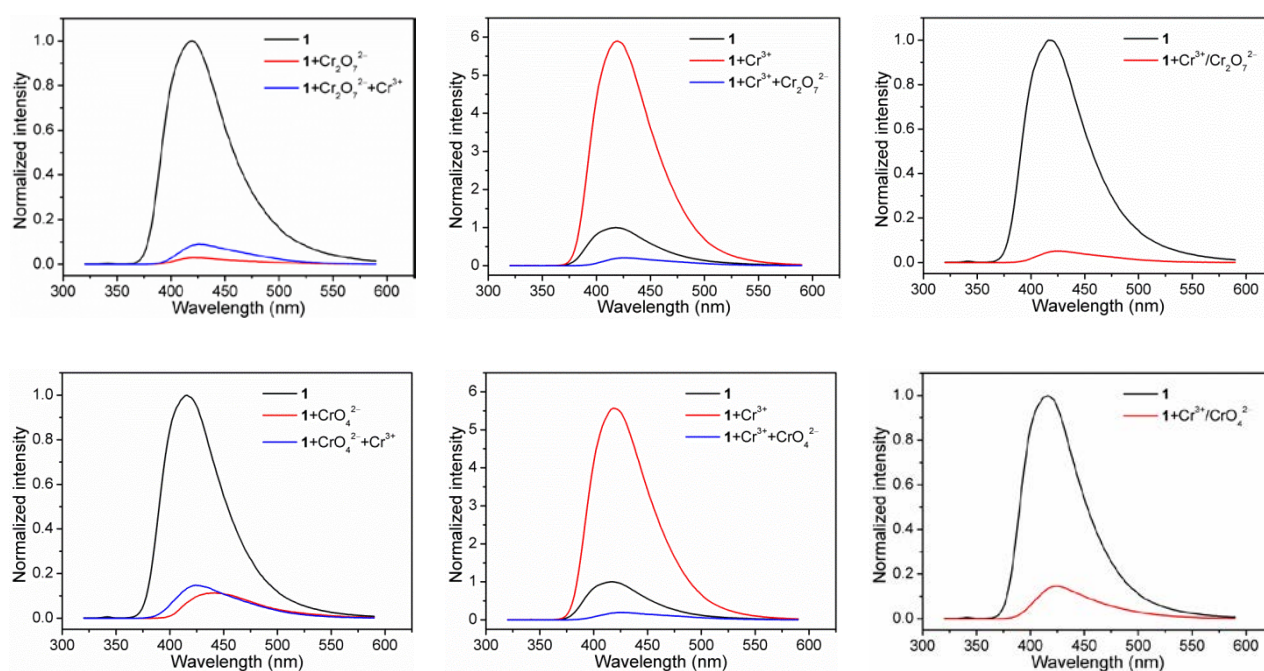


**Figure S10.** UV-vis spectra of **1** before and after immersing in Cr<sup>3+</sup>, Al<sup>3+</sup>, Fe<sup>3+</sup> aqueous solutions for 24 h.

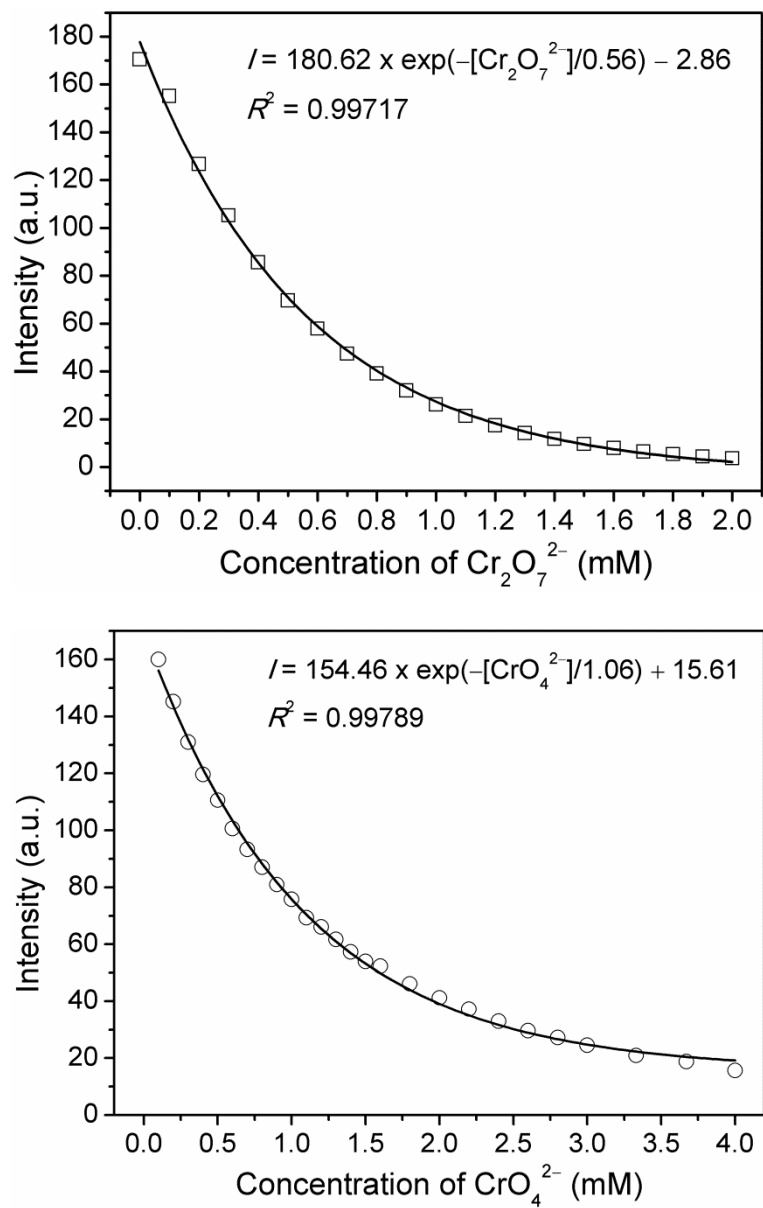


### *Influence of $\text{Cr}^{3+}$ detection in the coexistence of $\text{Cr(VI)}$ anions and vice versa*

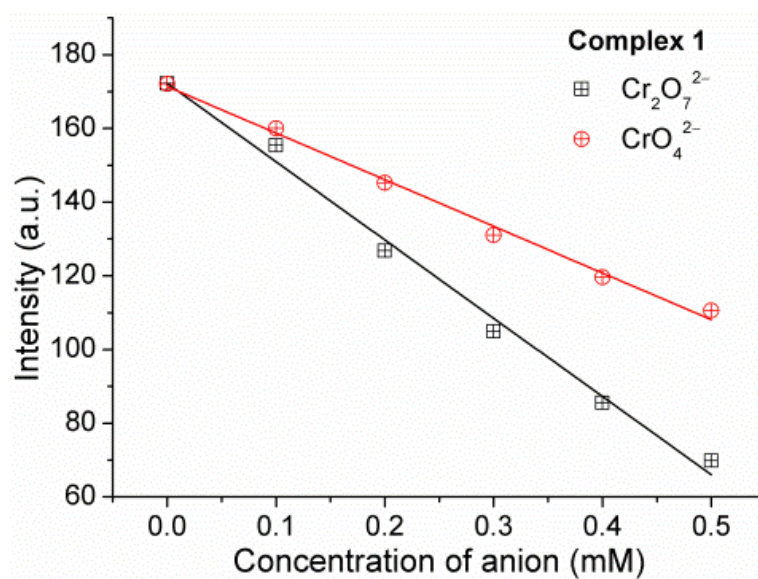
The influence of  $\text{Cr}^{3+}$  detection in the coexistence of  $\text{Cr(VI)}$  anions and vice versa using the well-prepared  $\text{H}_2\text{O}$  suspension of **1** (1 mg/3 mL) as sensor was studied. Interference experiments were carried out in two different ways: (1) adding the interfering ions firstly and then adding the analyte ions; (2) adding the mixture of analyte and interfering ions. The concentrations of analyte and interfering ions both are 1.0 mM. Before and after addition of interfering/analyte ions, the photoluminescence spectra were obtained at an excitation wavelength of 306 nm in each step. As shown in Figure S11, experimental results clearly indicate that  $\text{Cr(VI)}$  anions strongly interfere with  $\text{Cr}^{3+}$  detection while  $\text{Cr}^{3+}$  ions cause no interference on the detection of  $\text{Cr(VI)}$  anions ( $\text{Cr}_2\text{O}_7^{2-}$ ,  $\text{CrO}_4^{2-}$ ).



**Figure S11.** Fluorescence emission spectra of **1** in  $\text{H}_2\text{O}$  suspension (1 mg/3 mL) before and after addition of interfering/analyte ions at room temperature upon excitation at 306 nm.

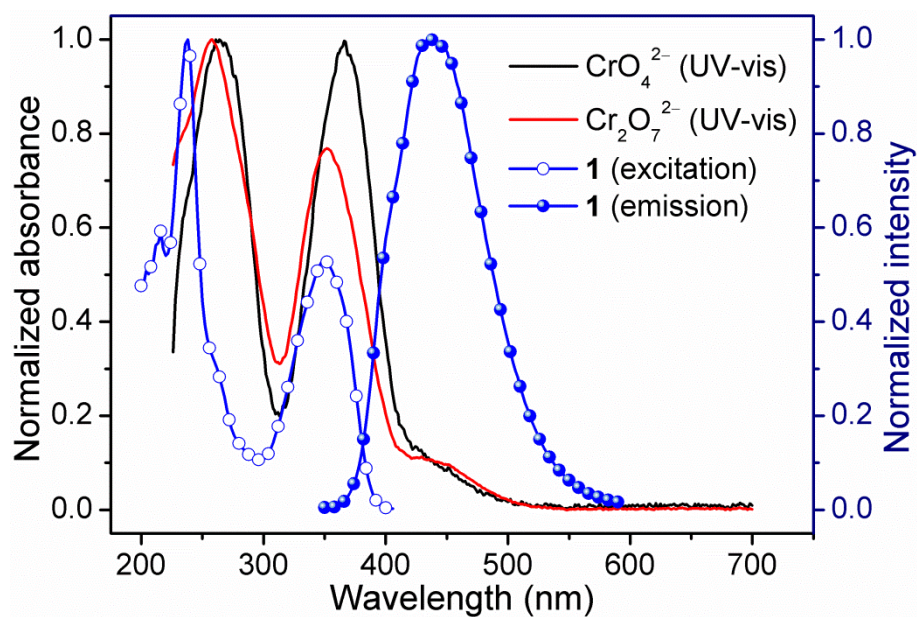


**Figure S12.** Fluorescence intensity traces ( $\lambda_{\text{em}} = 438 \text{ nm}$ ) for the H<sub>2</sub>O suspensions of **1** upon incremental addition of Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup> and CrO<sub>4</sub><sup>2-</sup> ions when excited at 306 nm, following the first-order exponential decay.



Blank readings	Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	CrO <sub>4</sub> <sup>2-</sup>
#1	170.4	172.1
#2	169.8	171.2
#3	176.1	170.0
#4	175.3	172.3
#5	175.6	172.7
Standard deviation ( $\sigma$ )	3.07	1.08
Slope (m)	212.4	126.5
$R^2$	0.99081	0.99277
LOD ( $3\sigma/m$ ), $\mu\text{M}$ (ppm)	43.36 (9.36)	25.57 (2.97)

**Figure S13.** Linear region of fluorescence intensity ( $\lambda_{\text{em}} = 438 \text{ nm}$ ) for the  $\text{H}_2\text{O}$  suspensions of **1** upon incremental addition of  $\text{Cr}_2\text{O}_7^{2-}$  and  $\text{CrO}_4^{2-}$  ions when excited at 306 nm. The following table lists the relevant parameters of LOD for the  $\text{H}_2\text{O}$  suspensions of **1** toward  $\text{Cr}_2\text{O}_7^{2-}$  and  $\text{CrO}_4^{2-}$  ions.



**Figure S14.** Spectral overlap between the normalized emission spectra of **1** in H<sub>2</sub>O suspensions and the normalized absorption spectra of Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup> and CrO<sub>4</sub><sup>2-</sup> in aqueous solutions.