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## **Supporting Information**

A smart two-photon fluorescent platform based on desulfurization-cyclization: phthalimide-rhodamine chemodosimeter for Hg<sup>2+</sup>, NIR emission at 746 nm and through-bond energy transfer

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Fig. S1. <sup>1</sup>H-NMR spectrum of CyRSN.

Fig. S2. <sup>13</sup>C-NMR spectrum of CyRSN.

Fig. S3. MS spectrum of CyRSN.

Fig. S4. <sup>1</sup>H-NMR spectrum of M<sub>2</sub>.

Fig. S5. <sup>13</sup>C-NMR spectrum of M<sub>2</sub>.

Fig. S6. MS spectrum of M<sub>2</sub>.

Fig. S7. <sup>1</sup>H-NMR spectrum of  $M_3(CyR)$ .

Fig. S8. <sup>13</sup>C-NMR spectrum of M<sub>3</sub> (CyR).

**Fig. S9.** <sup>1</sup>H-NMR spectrum of **NG-ML**.

Fig. S10. The linear relationship between the fluorescence intensity of probe at 746 nm and Hg<sup>2+</sup> in concentration within the range 0-10  $\mu$ M and 30-50  $\mu$ M.

**Fig. S11.** (a) Fluorescence ratio response (F746 nm/F540 nm) of free CyRSN (10  $\mu$ M) and after addition of Hg<sup>2+</sup> (30  $\mu$ M) in a PBS buffer-MeOH (v/v = 50/50, 50 mM PBS) solution as a function of different pH values. The excitation wavelength was 390 nm; (b) time-dependence on the fluorescence intensity at 746 nm of CyRSN (10  $\mu$  M) upon addition of 2.0 equiv. of Hg2+ at room temperature; in EtOH/PBS buffer solutions (pH 7.4,v/v, 1 : 1). Excitation wavelength was 670 nm.





Fig. S2. <sup>13</sup>C-NMR spectrum of CyRSN.





Fig. S4. <sup>1</sup>H-NMR spectrum of M<sub>2</sub>.

![](_page_2_Figure_2.jpeg)

Fig. S5. <sup>13</sup>C-NMR spectrum of  $M_2$ .

![](_page_3_Figure_0.jpeg)

![](_page_3_Figure_1.jpeg)

Fig. S7. <sup>1</sup>H-NMR spectrum of M<sub>3</sub> (CyR).

![](_page_4_Figure_0.jpeg)

Fig. S9. <sup>1</sup>H-NMR spectrum of NG-ML.

![](_page_5_Figure_0.jpeg)

Fig. S10. The linear relationship between the fluorescence intensity of probe at 746 nm and Hg<sup>2+</sup>

in concentration within the range (a) 0-10  $\mu$ M and (b) 30-50  $\mu$ M.

![](_page_5_Figure_3.jpeg)

**Fig. S11.** (a) Fluorescence ratio response ( $F_{746 \text{ nm}}/F_{540 \text{ nm}}$ ) of free CyRSN (10 µM) and after addition of Hg<sup>2+</sup> (30 µM) in a PBS buffer-MeOH (v/v = 50/50, 50 mM PBS) solution as a function of different pH values. The excitation wavelength was 390 nm; (b) time-dependence on the fluorescence intensity at 746 nm of CyRSN (10 µ M) upon addition of 2.0 equiv. of Hg<sup>2+</sup> at room temperature; in EtOH/PBS buffer solutions (pH 7.4,v/v, 1 : 1). Excitation wavelength was 670 nm.