

**Table S1** Comparison of the recoveries of spiked Hg<sup>2+</sup> standard solution in drinking water and Zhujiang River water

|                         | Drinking water |        |        | Zhujiang River water |        |        |
|-------------------------|----------------|--------|--------|----------------------|--------|--------|
| Added/ $\mu\text{M}$    | 0.5            | 1.0    | 2.0    | 0.5                  | 1.0    | 2.0    |
| Detected/ $\mu\text{M}$ | 0.4271         | 1.0154 | 1.8439 | 0.4086               | 0.9609 | 1.8344 |
| Recoveries/%            | 85.42          | 101.54 | 92.20  | 81.72                | 96.09  | 91.97  |
| RSD/%                   | 3.563          | 2.891  | 2.932  | 1.409                | 0.8040 | 3.303  |

**Table S2** Crystal data and structure refinement for **Hg-MOF1**

|                                                                                                                                       |                                                                               |
|---------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------|
| Empirical formula                                                                                                                     | C <sub>14</sub> H <sub>12</sub> N <sub>4</sub> O <sub>6</sub> Hg <sub>2</sub> |
| Formula weight                                                                                                                        | 733.46                                                                        |
| Crystal system                                                                                                                        | Monoclinic                                                                    |
| Space group                                                                                                                           | P2 <sub>1</sub> /c                                                            |
| <i>a</i> (Å)                                                                                                                          | 10.117(3)                                                                     |
| <i>b</i> (Å)                                                                                                                          | 20.587(6)                                                                     |
| <i>c</i> (Å)                                                                                                                          | 8.167(3)                                                                      |
| $\alpha$ (°)                                                                                                                          | 90                                                                            |
| $\beta$ (°)                                                                                                                           | 95.673(6)                                                                     |
| $\gamma$ (°)                                                                                                                          | 90                                                                            |
| <i>V</i> (Å <sup>3</sup> )                                                                                                            | 1692.7(9)                                                                     |
| <i>Z</i>                                                                                                                              | 4                                                                             |
| $\rho_{\text{calc}}$ (g/cm <sup>3</sup> )                                                                                             | 2.886                                                                         |
| <i>F</i> (000)                                                                                                                        | 1336.0                                                                        |
| $\mu$ (mm <sup>-1</sup> )                                                                                                             | 18.159                                                                        |
| Reflections collected                                                                                                                 | 3050                                                                          |
| GOF                                                                                                                                   | 1.035                                                                         |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> indices [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]                                          | 0.037, 0.163                                                                  |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> indices (all data)                                                                     | 0.122, 0.264                                                                  |
| <sup>a</sup> $R_1 = \sum   F_o  -  F_c   / \sum  F_o $ . <sup>b</sup> $wR_2 = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum (F_o^2)^2 \}^{1/2}$ |                                                                               |

**Table S3** Selected bond lengths (Å) and bond angles (°) for **Hg-MOF1**

| Hg-MOF     |            |             |           |
|------------|------------|-------------|-----------|
| Hg1-N2     | 2.263(13)  | Hg1-Hg2-O6  | 127.4(3)  |
| Hg1-N1     | 2.369(13)  | N4-O6-Hg2   | 110.9(10) |
| Hg1-Hg2    | 2.5205(10) | C13-N2-C10  | 120.2(14) |
| Hg2-O3     | 2.239(12)  | C13-N2-Hg1  | 124.0(11) |
| Hg2-O6     | 2.546(12)  | C10-N2-Hg1  | 115.8(10) |
| O1-N3      | 1.21(2)    | N2-C13-C12  | 121.1(16) |
| O6-N4      | 1.273(19)  | N2-C13-C14  | 118.9(16) |
| O4-N4      | 1.25(2)    | C12-C13-C14 | 119.9(16) |
| O5-N4      | 1.235(19)  | C6-N1-C2    | 118.5(14) |
| N2-C13     | 1.33(2)    | C6-N1-Hg1   | 113.8(10) |
| N2-C10     | 1.37(2)    | C2-N1-Hg1   | 127.6(11) |
| C13-C12    | 1.42(2)    | N3-O3-Hg2   | 112.6(11) |
| C13-C14    | 1.49(2)    | O4-N4-O5    | 119.6(15) |
| N1-C6      | 1.37(2)    | O4-N4-O6    | 118.2(15) |
| N1-C2      | 1.37(2)    | O5-N4-O6    | 122.2(16) |
| O3-N3      | 1.303(19)  | C1-C12-C13  | 119.7(16) |
| O2-N3      | 1.22(2)    | C12-C11-C9  | 120.3(16) |
| C12-C11    | 1.35(3)    | C10-C9-C11  | 117.5(16) |
| C11-C9     | 1.42(2)    | C10-C9-C8   | 121.2(16) |
| C9-C10     | 1.41(2)    | C11-C9-C8   | 121.3(16) |
| C9-C8      | 1.44(3)    | C7-C8-C9    | 119.4(16) |
| C8-C7      | 1.39(3)    | C8-C7-C5    | 120.6(16) |
| C7-C5      | 1.46(3)    | C4-C5-C6    | 118.6(17) |
| C5-C4      | 1.39(3)    | C4-C5-C7    | 121.9(17) |
| C5-C6      | 1.39(2)    | C6-C5-C7    | 119.5(16) |
| C6-C10     | 1.45(2)    | N1-C6-C5    | 122.5(16) |
| C2-C3      | 1.40(3)    | N1-C6-C10   | 116.8(15) |
| C2-C1      | 1.49(2)    | C5-C6-C10   | 120.4(15) |
| C3-C4      | 1.38(3)    | N1-C2-C3    | 120.3(16) |
| N2-Hg1-N1  | 73.1(5)    | N1-C2-C1    | 117.6(15) |
| N2-Hg1-Hg2 | 157.3(3)   | C3-C2-C1    | 122.1(16) |
| N1-Hg1-Hg2 | 128.9(3)   | C4-C3-C2    | 120.8(17) |
| O3-Hg2-Hg1 | 158.8(3)   | C3-C4-C5    | 119.1(17) |
| O3-Hg2-O6  | 73.7(4)    | N2-C10-C9   | 121.2(14) |

**Table S4** Crystal data and structure refinement for **Hg-MOF2**

|                                                                                                                                       |                                                                                |
|---------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------|
| Empirical formula                                                                                                                     | C <sub>28</sub> H <sub>24</sub> Cl <sub>4</sub> N <sub>4</sub> Hg <sub>3</sub> |
| Formula weight                                                                                                                        | 1160.08                                                                        |
| Crystal system                                                                                                                        | Monoclinic                                                                     |
| Space group                                                                                                                           | C 2/c                                                                          |
| <i>a</i> (Å)                                                                                                                          | 10.435(2)                                                                      |
| <i>b</i> (Å)                                                                                                                          | 16.024(3)                                                                      |
| <i>c</i> (Å)                                                                                                                          | 17.348(3)                                                                      |
| $\alpha$ (°)                                                                                                                          | 90                                                                             |
| $\beta$ (°)                                                                                                                           | 94.861(3)                                                                      |
| $\gamma$ (°)                                                                                                                          | 90                                                                             |
| <i>V</i> (Å <sup>3</sup> )                                                                                                            | 2890.3(9)                                                                      |
| <i>Z</i>                                                                                                                              | 4                                                                              |
| $\rho_{\text{cald}}$ (g/cm <sup>3</sup> )                                                                                             | 2.666                                                                          |
| <i>F</i> (000)                                                                                                                        | 2112.0                                                                         |
| $\mu$ (mm <sup>-1</sup> )                                                                                                             | 16.293                                                                         |
| Reflections collected                                                                                                                 | 2205                                                                           |
| GOF                                                                                                                                   | 1.014                                                                          |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> indices [ <i>I</i> > 2σ ( <i>I</i> ) ]                                                 | 0.054,0.196                                                                    |
| <i>R</i> <sub>1</sub> , <i>wR</i> <sub>2</sub> indices (all data)                                                                     | 0.139,0.293                                                                    |
| <sup>a</sup> $R_1 = \sum   F_o  -  F_c   / \sum  F_o $ . <sup>b</sup> $wR_2 = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum (F_o^2)^2 \}^{1/2}$ |                                                                                |

**Table S5** Selected bond lengths (Å) and bond angles (°) for **Hg-MOF2**

| Hg-MOF   |           |            |           |
|----------|-----------|------------|-----------|
| Hg1-Cl2  | 2.391(3)  | C1-N1-Hg2  | 125.0(6)  |
| Hg1-Cl2  | 2.391(3)  | C12-N1-Hg2 | 116.1(6)  |
| Hg1-Cl1  | 2.619(3)  | C10-N2-C11 | 119.0(8)  |
| Hg1-Cl1  | 2.619(3)  | C10-N2-Hg2 | 125.2(6)  |
| Hg2-N1   | 2.348(7)  | C11-N2-Hg2 | 115.8(6)  |
| Hg2-N2   | 2.351(7)  | N1-C1-C2   | 123.0(9)  |
| Hg2-Hg2  | 2.5523(8) | N1-C1-C14  | 117.4(8)  |
| Hg2-Cl1  | 2.868(3)  | C2-C1-C14  | 119.6(9)  |
| N1-C1    | 1.322(12) | C3-C2-C1   | 118.0(10) |
| N1-C12   | 1.359(11) | C3-C2-H2   | 121.0     |
| N2-C10   | 1.308(12) | C1-C2-H2   | 121.0     |
| N2-C11   | 1.346(11) | C2-C3-C4   | 120.9(9)  |
| C1-C2    | 1.435(14) | C2-C3-H3   | 119.5     |
| C1-C14   | 1.510(13) | C4-C3-H3   | 119.5     |
| C2-C3    | 1.373(14) | C3-C4-C5   | 125.2(9)  |
| C2-H2    | 0.9300    | C3-C4-C12  | 117.0(9)  |
| C3-C4    | 1.399(14) | C5-C4-C12  | 117.8(10) |
| C3-H3    | 0.9300    | C6-C5-C4   | 122.7(9)  |
| C4-C5    | 1.424(13) | C6-C5-H5   | 118.6     |
| C4-C12   | 1.441(12) | C4-C5-H5   | 118.6     |
| C5-C6    | 1.328(15) | C5-C6-C7   | 121.6(9)  |
| C5-H5    | 0.9300    | C5-C6-H6   | 119.2     |
| C6-C7    | 1.429(14) | C7-C6-H6   | 119.2     |
| C6-H6    | 0.9300    | C8-C7-C6   | 124.4(9)  |
| C7-C8    | 1.390(14) | C8-C7-C11  | 116.4(9)  |
| C7-C11   | 1.430(12) | C6-C7-C11  | 119.2(10) |
| C8-C9    | 1.341(14) | C9-C8-C7   | 120.7(9)  |
| C8-H8    | 0.9300    | C9-C8-H8   | 119.7     |
| C9-C10   | 1.406(13) | C7-C8-H8   | 119.7     |
| C9-H9    | 0.9300    | C8-C9-C10  | 119.4(10) |
| C10-C13  | 1.528(14) | C8-C9-H9   | 120.3     |
| C11-C12  | 1.439(14) | C10-C9-H9  | 120.3     |
| C13-H13A | 0.9600    | N2-C10-C9  | 122.4(9)  |
| C13-H13B | 0.9600    | N2-C10-C13 | 117.6(9)  |
| C13-H13C | 0.9600    | C9-C10-C13 | 120.0(9)  |

|           |            |               |          |
|-----------|------------|---------------|----------|
| C14-H14A  | 0.9600     | N2-C11-C7     | 122.1(9) |
| C14-H14B  | 0.9600     | N2-C11-C12    | 119.1(8) |
| C14-H14C  | 0.9600     | C7-C11-C12    | 118.7(9) |
| Cl2Hg1Cl2 | 139.81(14) | N1-C12-C11    | 117.9(8) |
| Cl2Hg1Cl1 | 101.29(9)  | N1-C12-C4     | 122.2(9) |
| Cl2Hg1Cl1 | 101.97(9)  | C11-C12-C4    | 119.9(8) |
| Cl2Hg1Cl1 | 101.97(9)  | C10-C13-H13A  | 109.5    |
| Cl2Hg1Cl1 | 101.29(9)  | C10-C13-H13B  | 109.5    |
| Cl1Hg1Cl1 | 108.16(12) | H13A-C13-H13B | 109.5    |
| N1Hg2N2   | 71.0(3)    | C10-C13-H13C  | 109.5    |
| N1Hg2Hg2  | 135.49(17) | H13A-C13-H13C | 109.5    |
| N2Hg2Hg2  | 146.30(19) | H13B-C13-H13C | 109.5    |
| N1Hg2Cl1  | 87.9(2)    | C1-C14-H14A   | 109.5    |
| N2Hg2Cl1  | 100.77(19) | C1-C14-H14B   | 109.5    |
| Hg2Hg2Cl1 | 100.60(6)  | H14A-C14-H14B | 109.5    |
| Hg1Cl1Hg2 | 112.85(10) | H14A-C14-H14C | 109.5    |
| C1N1C12   | 118.9(8)   | H14B-C14-H14C | 109.5    |