# **Electronic Supplementary Information (ESI)**

## 1. Reagents and Chemicals

All chemicals used in this work are of AR or HPLC grade. 18 M $\Omega$ -cm deionized water (DIW) was produced with a water purification system (PCWJ-10, Pure Technology Co. Ltd, Chengdu, China). N, N-dimethylformamide (DMF), 1, 4-benzenedicarboxylate (BDC), FeCl<sub>3</sub>•6H<sub>2</sub>O, CCl<sub>4</sub> and ethanol were purchased from Kelong Chemical Reagent Co. Ltd. (Chengdu, China). Standard solutions of MeHg<sup>+</sup>, EtHg<sup>+</sup>, PhHg<sup>+</sup> and Hg<sup>2+</sup> in methanol (76.6 ppm) were obtained from National Institute of Metrology China (Beijing, China). Methanol was purchased from Amethyst Chemicals (J&K Scientific, China). All standards and stock solutions were stored at 4 °C in a refrigerator until use.

### 2. Instruments

A Uwave-1000 microwave reactor was purchased from Sineo Microwave Chemistry Technology Co. Ltd. (Shanghai, China). The fluorescence data were collected with an F-7000 fluorescence spectrometer (Hitachi, Japan) using a 390 nm optical filter. The PXRD patterns were obtained with an X'Pert Pro MPD (Philips, Netherlands) X-ray diffraction spectrometer using  $Cu_{ka}$  radiation. The SEM images were recorded on a JEOL JSM-7500F scanning electron microscope at 30.0 kV.

#### 3. Figures and Captions



Fig. S1 FL intensities of the  $CCl_4$  suspension prepared with four different MIL MOFs containing MgHg<sup>+</sup> with the same concentration.



Fig. S2 FL intensity of MIL-53(Fe) suspension prepared with the dispersant of water, ethanol, or tetrachloromethane, with 500 ppb of Hg species added into each suspension.



Fig. S3 FL spectra obtained from the MIL-53(Fe) suspension prepared with the dispersant of  $CCl_4$  or  $H_2O$ .



Fig. S4 The lengths/angles of C-H bonds and the lengths of C-Hg bonds in MeHg<sup>+</sup> and EtHg<sup>+</sup>. The figures were made using the Material Studio software (version 6.0, Accelrys).

Table S1 Pore size of different MIL MOF

MIL MOF	MIL-53(Al) <sup>1</sup>	MIL-53(Fe) <sup>2</sup>	$MIL-68(In)^3$	MIL-88B(Fe) <sup>4</sup>
Pore size (Å)	6.57	8	6	9.5

#### References

1 T. Loiseau, C. Serre, C. Huguenard, G. Fink, F. Taulelle, M. Henry, T. Bataille and G. Férey, *Chem. Eur. J.*, 2004, **10**, 1373.

2 P. Horcajada, C. Serre, G. Maurin, N. A. Ramsahye, F. Balas, M. Vallet-Regi, M. Sebban, F. Taulelle and G. Ferey, *J. Am. Chem. Soc.*, 2008, **130**, 6774.

3 J. Liu, F. Zhang, X. Zou, S. Zhou, L. Li, F. Sun and S. Qiu, Eur. J. Inorg. Chem., 2012, 35, 5784.

4 P. Horcajada, F. Salles, S. Wuttke, T. Devic, D. Heurtaux, G. Maurin, A. Vimont, M. Daturi, O.

David, E. Magnier, N. Stock, Y. Filinchuk, D. Popov, C. Riekel, G. Férey and C. Serre, J. Am. Chem. Soc., 2011, 133, 17839.