Electronic Supporting Information (ESI) for

**Fast and Effective Decontamination of Aqueous Mercury by a Highly Stable Zeolitic-Like Chalcogenide**

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1. Crystal Structure

1) **Crystal Structure Determination:**

Intensity data collection of GaSnS-1 was performed on a SuperNova Oxford diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) at 100(2) K. The structure of GaSnS-1 was solved by direct methods and refined by full-matrix least-squares on F² using the SHELX–2016 program package.[1] For GaSnS-1, the M site consisting of Ga(1) and Sn(1) atoms with 0.55/0.45 occupancy ratio was determined by ICP-AES, and ultimately refined with common displacement parameters (EADP command). The protonated organic amines and solvent molecules located in the void space of the framework cannot be identified owing to their serious disorder. Therefore, finally, the SQUEEZE[2] option of PLATON was used in refining the structure of GaSnS-1. The empirical formula of GaSnS-1 was calculated by considering the results of TGA, EA and the single-crystal structure, which was further confirmed by the ICP-AES and EDS results. Detailed crystallographic data and structure refinement parameters of GaSnS-1 are summarized in Table S1. Selected bond lengths and bond angles data are listed in Table S2.

**Table S1.** Crystallographic data of GaSnS-1.

| GaSnS-1 |                      |
|---------|----------------------|
| **Empirical formula** | C_{9.6H_{33}}Ga_{2.2}N_{6.4}OS_{8}Sn_{1.8} |
| **Formula weight (g mol⁻¹)** | 877.73 |
| **Temperature (K)** | 100(2) |
| **Wavelength (Å)** | 0.71073 |
| **Crystal system** | cubic |
| **Space group** | I-43m |
| **a (Å)** | 21.2199(3) |
| **V(Å³)** | 9555.0(4) |
| **Z** | 12 |
| Property                        | Value   |
|--------------------------------|---------|
| $D_{\text{calcd}}$ (g cm$^{-3}$) | 1.830   |
| Absorption coefficient (mm$^{-1}$) | 3.776   |
| $R_{\text{int}}$            | 0.0305  |
| $F(000)$                     | 5155    |
| Independent refls.           | 2062    |
| No. of parameters            | 34      |
| Final $R$ indices [$I > 2\sigma(I)$] | $R_1$ = 0.0262, $R_2$ = 0.0553 |
| $R$ indices (all data)       | $R_1$ = 0.0346, $R_2$ = 0.0587 |
| Goodness of fit on $R^2$     | 1.029   |
| CCDC                         | 1585127 |

[a] $R_1 = \sum |F_O| - |F_C|/\sum |F_O|$, [b] $wR_2 = \{\sum w[(F_O)^2 - (F_C)^2]/\sum w(F_O)^2\}^{1/2}$

2) **Selected Bond Lengths and Bond Angles Data:**

**Table S2.** Selected bond lengths (Å) and bond angles (°) data for GaSnS-1.

| Bond                  | Length (Å) | Bond                  | Length (Å) | Bond                  | Length (Å) |
|-----------------------|------------|-----------------------|------------|-----------------------|------------|
| Sn(1)–S(3)            | 2.236(6)   | Ga(1)–S(1)            | 2.250(9)   |
| Sn(1)–S(1)#1          | 2.275(7)   | Ga(1)–S(2)            | 2.231(9)   |
| Sn(1)–S(2)            | 2.379(6)   | Ga(1)–S(3)            | 2.409(8)   |
| Sn(1)–S(1)            | 2.363(7)   | Ga(1)–S(1)#1          | 2.412(9)   |
| S(3)–Sn(1)–S(1)#1     | 104.74(18) | Ga(1)–S(1)–Ga(1)#2   | 98.5(4)    |
| S(3)–Sn(1)–S(2)       | 109.0(3)   | Sn(1)#2–S(1)–Ga(1)#2 | 4.9(3)     |
| S(1)#1–Sn(1)–S(2)     | 111.3(3)   | Sn(1)–S(1)–Ga(1)#2   | 101.74(10) |
| S(3)–Sn(1)–S(1)       | 110.1(3)   | Ga(1)#3–S(2)–Ga(1)   | 100.2(5)   |
| S(1)#1–Sn(1)–S(1)     | 112.6(3)   | Ga(1)#3–S(2)–Sn(1)   | 102.98(10) |
| S(2)–Sn(1)–S(1)       | 108.92(18) | Ga(1)–S(2)–Sn(1)     | 4.8(3)     |
| S(1)–Ga(1)–S(2)       | 118.9(3)   | Ga(1)#3–S(2)–Sn(1)#3 | 4.8(3)     |
| S(1)–Ga(1)–S(3)       | 108.0(4)   | Ga(1)–S(2)–Sn(1)#3   | 102.98(10) |
| S(2)–Ga(1)–S(3)       | 110.2(4)   | Sn(1)#2–S(2)–Sn(1)#3 | 106.0(3)   |
| S(1)–Ga(1)–S(1)#1     | 111.7(4)   | Sn(1)#4–S(3)–Sn(1)   | 109.8(3)   |
| S(2)–Ga(1)–S(1)#1     | 111.7(4)   | Sn(1)#4–S(3)–Ga(1)   | 105.65(8)  |
| S(3)–Ga(1)–S(1)#1     | 95.7(2)    | Sn(1)#3–Ga(1)         | 4.2(4)     |
| Ga(1)–S(1)–Sn(1)#2    | 103.40(13) | Sn(1)#4–S(3)–Ga(1)#4 | 4.2(4)     |
| Ga(1)–S(1)–Sn(1)      | 5.4(3)     | Sn(1)#3–Ga(1)#4      | 105.65(8)  |
| Sn(1)#2–S(1)–Sn(1)    | 106.7(3)   | Ga(1)#3–Ga(1)#4      | 101.5(4)   |

Symmetry transformations used to generate equivalent atoms for GaSnS-1:

#1 $-y+3/2, x+1/2, -z+3/2$. #2 $y-1/2, -x+3/2, -z+3/2$. #3 $-x+1, -y+2, z$. #4 $x, z, y$.

3) **Asymmetric Unit:**
Although GaSnS₄-GeSn₄-Sn, In-SUCR, GaSnS₃ compound 5) days products (e.g. indicated that reaction time may play an important role in the crystallization of the desired 3D anionic skeleton of [Ga₂(Sn₄)₆]₄³⁻ crystallization can be divided into four styles, i.e., sodalite-type, diamond-type, double diamond-type and CrB₂-type. If the same metal ion (e.g., Ga³⁺, Sn⁴⁺) were introduced into the reaction system, different template cations (e.g., TMDP, TAEMA) may lead to similar structures but with different framework composition, as exemplified by GaSnS₃-1 (Ga/Sn = 2.2/1.8) and UCR-20GaSnS-TMDP (Ga/Sn = 1.8/2.2). In addition, although GaSnS₃-1 and UCR-21GaSnS-TAEMA have the similar template cations and secondary building units ([Ga₃Sn₄₋₄S₁₀] T2 cluster), their crystal structures are different. The latter compound features a 3D anionic skeleton of [Ga₂₃Sn₁₆₈S₈]₄²⁻ characteristic of diamond-typed topology, while the GaSnS₃-1 exhibits a sodalite-like framework structure of [Ga₂₂Sn₁₈S₆]₄²⁻. It was indicated that reaction time may play an important role in the crystallization of the desired products (e.g., for UCR-21GaSnS-TAEMA, 190 °C for 5 days; for GaSnS₃-1, 190 °C for 9 days).

5) **A Summary of the Members of Zeolitic-Like Chalcogenide Family:**

**Table S3.** A summary of the members of zeolitic-like chalcogenide family.

| Compound        | Framework Composition | Template | a/Å  | c/Å  | Space Group | Topology  | Temperature |
|-----------------|-----------------------|----------|------|------|-------------|-----------|-------------|
| GaSnS₃-1        | Ga₂₃Sn₁₆₈S₈           | TAEA     | 21.2199(3) | 21.2199(3) | Å3m        | sod       | 190 °C, 9D  |
| UCR-20GaGeS⁶⁶   | Ga₄₋₄Ge₁₃S₈          | AEP      | 20.9884(17) | 20.9884(17) | Å3m        | sod       | 190 °C, 5D  |
| UCR-20GaGeS⁶⁶   | Ga₄₋₄Ge₁₃S₈          | TAEA     | 20.9352(15) | 20.9352(15) | Å3m        | sod       | 190 °C, 5D  |

**Figure S1.** The asymmetric unit of GaSnS-1, which was drawn with 50% probability displacement ellipsoids. The Ga(1) and Sn(1) atoms are disordered with 0.55/0.45 occupancy ratio. Organic parts and lattice water molecules are omitted for clarity.

4) **Structural Discussion:**

Since 2002, the exploratory synthesis of 3D microporous chalcogenide zeolite analogs has attracted considerable attention due to their fascinating structures and great potentials in the fields of fast-ion conduction,[³] photocatalysis,[⁴] ion exchange,[⁵] and so on. In particular, the significant progress has been witnessed by some compounds based on the combination of divalent/trivalent metal ions (e.g., Zn²⁺, Cd²⁺, Ga³⁺, In³⁺) and tetravalent metal ions (e.g., Ge⁴⁺, Sn⁴⁺), such as UCR-20GaSnS-TMDP[⁶] UCR-21GaSnS-TAEMA, UCR-22GaSnS-AEP[⁶] and CPM-120ZnGeS-AEM.[⁷] As shown in Table S3 and Figure S2, their anionic frameworks can be divided into four styles, i.e., sodalite-type, diamond-type, double diamond-type and CrB₂-type.
| Compound                 | Formula      | Capping | m.p. | Ref. |
|-------------------------|--------------|---------|------|------|
| UCR-20GaGeS[6]          | Ga₄Ge₄₆S₈     | BAPP    | 21.293(2) | 190°C, 5D |
| UCR-20GaGeS[6]          | Ga₄Ge₄₆S₈    | AEM     | 21.139(4) | 190°C, 5D |
| UCR-20GaSnS[6]          | Ga₂₈Sn₂₀₆S₈  | TMDP    | 21.5404(17) | 190°C, 5D |
| UCR-20InGeS[6]          | In₃₀Ge₁₀₆S₈  | TMDP    | 21.734(2) | 190°C, 5D |
| UCR-20InSnS[6]          | In₂₀Sn₁₈₆S₈  | TMDP    | 22.1906(18) | 190°C, 5D |
| UCR-20GaGeSe[6]         | Ga₄Ge₄₆Se₈    | TMDP    | 22.157(3) | 190°C, 5D |
| UCR-20GaGeSe[6]         | Ga₄Ge₄₆Se₈   | TMDP    | 21.893(2) | 190°C, 5D |
| CPM-120ZnGeS[7]         | Zn₀₆₅Ge₁₈₆S₈ | AEM     | 20.722(4) | 190°C, 12D |
| CPM-120ZnSnS[7]         | Zn₀₆₅Sn₁₈₆S₈ | BPP     | 22.614(6) | 190°C, 12D |
| CPM-120CdSnS[7]         | Cd₃₄₄Sn₁₈₆S₈ | BPP     | 22.429(6) | 190°C, 12D |
| UCR-21GaGeS[6]          | Ga₄Ge₄₆S₈    | HMI     | 11.5128(13) | 190°C, 5D |
| UCR-21GaGeS[6]          | Ga₄Ge₄₆S₈    | AEP     | 11.280(2) | 190°C, 5D |
| UCR-21GaGeS[6]          | Ga₄Ge₄₆S₈    | APM     | 11.2864(18) | 190°C, 5D |
| UCR-21GaGeS[6]          | Ga₄Ge₄₆S₈    | AEM     | 11.599(4) | 190°C, 5D |
| UCR-21GaSnS[6]          | Ga₄Sn₁₈₆S₈   | AEP     | 11.50(6) | 190°C, 5D |
| UCR-21InGeS[6]          | In₁₄₄Ge₁₈₆S₈ | APP     | 11.5313(15) | 190°C, 5D |
| UCR-21InGeS[6]          | In₁₄₄Ge₁₈₆S₈ | AEM     | 16.354(4) | 190°C, 5D |
| UCR-21InGeS[6]          | In₁₄₄Ge₁₈₆S₈ | APP     | 11.4416(13) | 190°C, 5D |
| UCR-21InSnS[6]          | In₁₄₄Sn₁₈₆S₈ | AEP     | 13.379(2) | 190°C, 5D |
| UCR-21GaSnSe[6]         | Ge₂₈Sn₁₈₆Se₈ | TAEA    | 12.5373(19) | 190°C, 5D |
| CPM-121ZnGeS[7]         | Zn₀₆₅Ge₁₈₆S₈ | AEM/TB  | 11.215(2) | 190°C, 12D |
| CPM-121ZnGeSnS[8]       | Zn₀₆₅Ge₁₈₆Sn₈ | AEM     | NA | NA |
| CPM-121ZnGeSnS[8]       | Zn₀₆₅Ge₁₈₆Sn₈ | AEM     | NA | NA |
| UCR-22GaGeS[6]          | Ga₄₆Ge₄₆S₈   | AEP     | 22.532(2) | 190°C, 5D |
| UCR-22GaSnS[6]          | Ga₂₈Sn₂₀₆S₈  | AEP     | 22.935(2) | 190°C, 5D |
| UCR-22InGeS[6]          | In₂₈Ge₁₈₆S₈  | AEP     | 22.9078(12) | 190°C, 5D |
| UCR-22InGeS[6]          | In₂₈Ge₁₈₆S₈  | TETA    | 22.737(2) | 190°C, 5D |
| UCR-22InGeS[6]          | In₂₈Ge₁₈₆S₈  | TAEA    | 22.813(3) | 190°C, 5D |
Figure S2. The node and topology of the 3D microporous zeolitic-like chalcogenide family, in which the metal atoms and \( S_2^2^-/Se_2^2^- \) atoms are shown as green and purple balls, respectively.

Figure S3. The crystal structures of some TAEA-directed homometallic compound, i.e., (a) UCR-7GaS-TAEA, (b) [TAEAH]_2Sn_3S_7 and (c) [TAEAH_2]_2Sn_2S_6.

2. Characterizations

1) Physical Measurements:

Element analyses (EA) of C, N, and H were performed with a German Elementary Vario EL III instrument. Energy-dispersive spectroscopy (EDS) was recorded on a JEOL
JSM-6700F scanning electron microscope. Thermogravimetry (TG) curves were obtained using a NETZSCH STA449F3 thermogravimetric analyzer at a heating rate of 10 °C/min under a nitrogen atmosphere. Fourier transform infrared (FT−IR) spectra were measured on a Nicolet Magna 750 FT−IR spectrometer in the 4000−500 cm\(^{-1}\) region by using a KBr pellet. X-ray photoelectron spectroscopy (XPS) experiments were carried out on an ESCALAB 250Xi spectrometer with Al K\(\alpha\) radiation. Powder X-ray diffraction (PXRD) patterns were collected at room temperature on a Miniflex II diffractometer using Cu K\(\alpha\) radiation (\(\lambda = 1.5406 \text{ Å}\)) in the 2\(\theta\) range of 3(5)−65°. Elemental ratio of Ga and Sn was determined by inductively coupled plasma−atomic emission spectrometry (ICP−AES, Ultima 2 unit). Raman spectrum was recorded on a microscopic confocal Raman spectrometer using a 633 nm He−Ne laser. The concentrations of metal ions in the solution before and after adsorption process were analyzed with the help of ICP−AES (Ultima 2 unit) and inductively coupled plasma-mass spectroscopy (ICP-MS, XSerise II).

2) ICP-AES:

| Compound | Ga (%) | Sn (%) |
|----------|--------|--------|
| GaSnS-1  | 15.56  | 21.59  |

3) Experimental Section:

In the isotherm experiments, the water solutions of Hg\(^{2+}\) with different concentrations were prepared. The \(V/m\) of all the samples is 1000 mL/g (\(V = 10 \text{ mL}, m = 10 \text{ mg}\)). The adsorption processes lasted 24 h at room temperature (~25 °C), after which the samples were taken out and processed.

For the kinetics studies of GaSnS-1, adsorption experiment of various reaction time (2, 5, 15, 30, 60, 120, 180, 300, 480, 900 and 1440 min) were performed. 30 mg of GaSnS-1 powders were weighed into 30 mL of water solution containing 1841 ppb of Hg\(^{2+}\), and the mixtures were kept under magnetic stirring at room temperature (~25 °C). After the designated reaction time, the suspensions were filtered and analyzed by ICP-MS.

Experiments studying the pH dependence of Hg\(^{2+}\) ion adsorption were also carried out. The mercury contaminated solutions with different pH values (in the range of 1.92−11.84) were prepared. The initial concentrations of Hg\(^{2+}\) ions were in the range of 2.16−7.06 ppm, with \(V/m = 1000 \text{ mL/g (} V = 10 \text{ mL; } m = 0.010 \text{ g}\)). The pH was regulated by NaOH or HCl. The adsorption experiments lasted 24 h at room temperature (~25 °C). Subsequently, the samples were taken out and processed.

The competitive adsorption experiments of Hg\(^{2+}\) ion in the presence of excess alkali/alkaline earth metal ions (Na\(^+\), K\(^+\), Mg\(^{2+}\) and Ca\(^{2+}\)) or other mixed heavy metal ions (Mn\(^{2+}\), Zn\(^{2+}\), Co\(^{2+}\), Ni\(^{2+}\), Cd\(^{2+}\), Pb\(^{2+}\) and Cu\(^{2+}\)) were carried out using a \(V/m\) ratio of 1000 mL/g (\(V = 10 \text{ mL; } m = 0.010 \text{ g}\)), at room temperature (~25 °C) and a 24 h contact time, respectively. The individual heavy metal ions (Cr\(^{3+}\), Co\(^{2+}\), Mn\(^{2+}\), Zn\(^{2+}\), Ni\(^{2+}\), Cu\(^{2+}\), Ag\(^+\), Pb\(^{2+}\), Cd\(^{2+}\) and Hg\(^{2+}\)) uptake experiments were also studied with \(V/m = 1000 \text{ mL/g (} V = 10 \text{ mL; } m = 0.010 \text{ g}\)), at
room temperature (~25 °C) and a 24 h contact time. Then the typical adsorption experiments were performed.

The simulated column adsorption experiment was conducted on an empty glass with inside diameter of 10.2 mm and outside diameter of 13.4 mm. A 100 mg sample of GaSnS-1 polycrystalline powder was weighed into 50 mL of water solution containing 3.31 ppm of mercury (bed volume = 0.21 mL). Subsequently, the Hg$^{2+}$ ion concentration in the outlet solution was tracked and analyzed.

4) **Characterizations**:

![Figure S4](image1.png) **Figure S4.** The photographs of GaSnS-1 and Hg@GaSnS-1.

![Figure S5](image2.png) **Figure S5.** The energy dispersive X-ray (EDX) spectrum of GaSnS-1.

![Figure S6](image3.png) **Figure S6.** PXRD patterns of the pristine GaSnS-1 and Hg$^{2+}$-loaded product of GaSnS-1. The initial concentration of Hg$^{2+}$ ion was about 800 ppm.
Figure S7. TGA curves for GaSnS-1 and Hg@GaSnS-1. The more weight losses of Hg@GaSnS-1 below 100 °C can be attributed to the removal of the more adsorbed water molecules. The initial concentration of Hg$^{2+}$ ion was about 400 ppm.

Figure S8. PXRD patterns of GaSnS-1 in aqueous solution with different pH values.

Figure S9. Raman data of Hg@GaSnS-1.
Figure S10. FT–IR spectra of GaSnS-1 and Hg@GaSnS-1.

Figure S11. The relative amount of Hg$^{2+}$ ion removed (%) vs the time $t$ (min).
Figure S12. PXRD patterns of GaSnS-1 and GaSnS-1 after immersed in aqueous solutions with different individual heavy metal ion.

3. Important Characteristics of Representative Mercury Sorbents

Table S5. The mercury removal efficiency of various sorbents in this work and references.

| Materials       | Time (min) | pH range | Capacity (mg/g) | Removal rate(%) | $K_D$ (mL/g) | Limit (ppb) |
|-----------------|------------|----------|-----------------|-----------------|--------------|-------------|
| GaSnS-1         | <2         | 1.92−11.84 | 213.9           | close 100       | $1.62 \times 10^1$ | <2         |
| LiMoS$_2$[9]    | NA         | US       | NA              | NA              | NA           | NA          |
| KMS-1[10]       | ~5         | 2.5−9.5  | 377             | >98             | $3.9 \times 10^5$ | <2         |
| KMS-2[11]       | ~100       | NA       | 296.8           | >99.9           | NA           | NA          |
| LHMS-1[12]      | 10         | 0−9      | 87              | 99.99           | >10$^6$      | <2         |
| K$_2$MS[13]     | 60         | 3−8      | NA              | NA              | >10$^6$      | NA          |
| Cs$_2$SnSbS$_6$[14] | ~100   | NA       | NA              | 99.9            | $6.8 \times 10^7$ | ~7         |
| InSnOS[15]      | NA         | 2−10     | NA              | ~99             | $1.8 \times 10^4$ | ~100      |
| KTS-3[16]       | <5         | 2−12     | 391             | 99.7            | $2.3 \times 10^4$ | <2         |
| Chalcogel-1[17] | NA         | NA       | NA              | close 100       | NA           | ~40        |
| W-DR-N-MoS$_2$[18] | ~5      | 3−10     | 2506            | 99.83           | $3.53 \times 10^4$ | <2         |
| HKUST-1-SF[19]  | ~120       | NA       | 714.29          | 99.79           | $4.73 \times 10^3$ | ~7.4      |
| PAF-1-SF[20]    | <20        | 1.0−12.8 | 1014            | >99.9          | $5.76 \times 10^7$ | <2         |
| Zr-DMBD[21]     | 720        | NA       | ~118.2          | >99.9          | NA           | ~10        |
| S$_2$–LDH[22]   | NA         | ~3       | 686             | close 100      | ~10$^7$     | <1         |
| MOF-5-TMBP[23]  | 8640       | US       | 107.1           | ~94            | NA          | 5000       |
| TMT[24]         | >300       | 2−7      | 735.3           | 97.6           | NA          | NA          |
| Zeolite<sup>(25)</sup> | ~100 | 3−9 | 1.6 | ~51.73 | NA | NA |
|----------------------|-------|-----|-----|---------|----|----|
| Zeolite-dithizone<sup>(29)</sup> | ~100 | 3−9 | 21.0 | ~99.36 | NA | ~51.2 |
| Activated carbon<sup>(16)</sup> | ~180 | 1−12 | 44.8 | 83.4 | NA | ~33200 |
| MCM-41<sup>[27]</sup> | ~40 | NA | NA | NA | NA | ~800 |
| NaTiO<sub>2</sub><sup>(24)</sup> | ~120 | 2−9 | 454.55 | 98.2 | NA | <15 |
| PPy-Cotton<sup>(29)</sup> | ~100 | 1−6 | 31.7 | 91.0 | 2.4 × 10<sup>4</sup> | ~16 |
| MP-HMS<sup>(30)</sup> | NA | NA | 1436 | NA | NA | ~5 |
| Si<sub>5</sub><sup>(31)</sup> | NA | NA | 2710 | NA | >10<sup>4</sup> | ~230 |
| Mg-MTMS<sup>(31)</sup> | NA | NA | 603 | ~99 | NA | ~200 |
| SOL-AD-JV<sup>(32)</sup> | ~150 | 3−5 | 726 | NA | NA | ~500 |

NA: Not available; 2 ppb: The upper limit of Hg in drinking water; US: Unstable in water.

4. References

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