Supporting Information for

Sandwich-like octacyanometalate-based cadmium assemblies with the 4,4'-dipyridyl sulfide ligand

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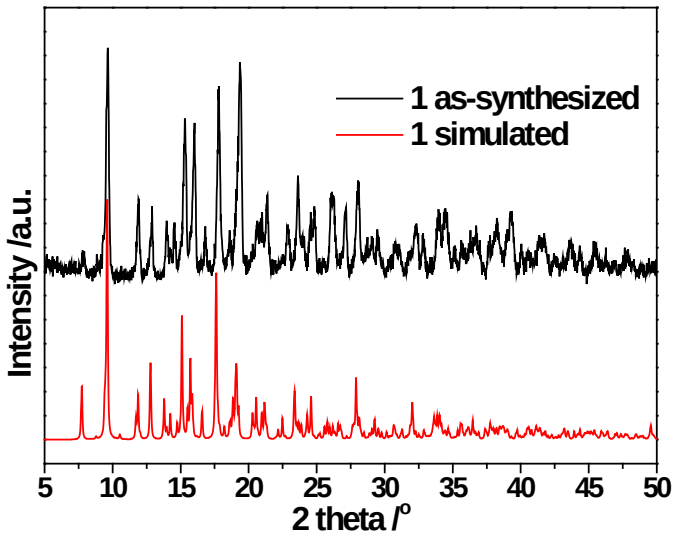
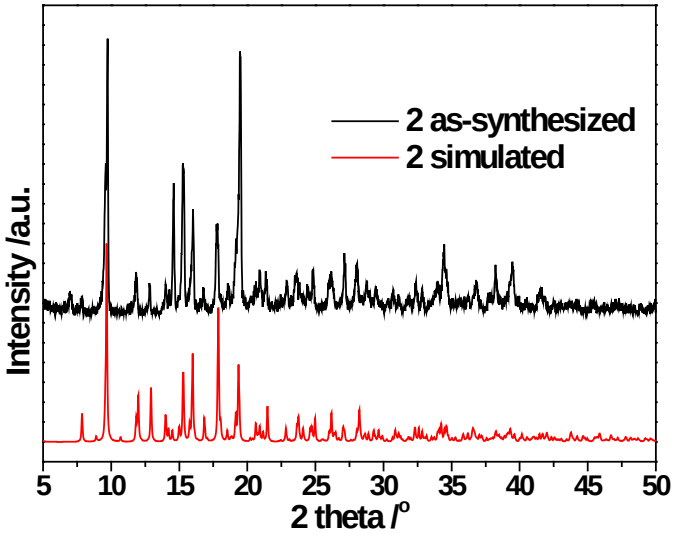
General considerations. Unless otherwise mentioned, all reactants were used as purchased without further purification. All chemicals and solvents were purchase from commercial sources and used as received. $CdSO_4 \cdot 8/3H_2O$ and 4,4'-dipyridyl sulfide (dps) ligand were purchased from commercial sources and used without further purification. The $[HN(n\text{-}C_4H_9)_3]_3W(CN)_8$ precursor was prepared according to the published procedure.[1] IR spectra were measured on a Nicolet FT 1703X spectrophotometer in the form of KBr pellets in the 4000-400 cm^{-1} region. Powder XRD patterns of compound **1**-**3** was collected with Cu-*K*α radiation using a Shimadzu XRD-6000 diffractometer.

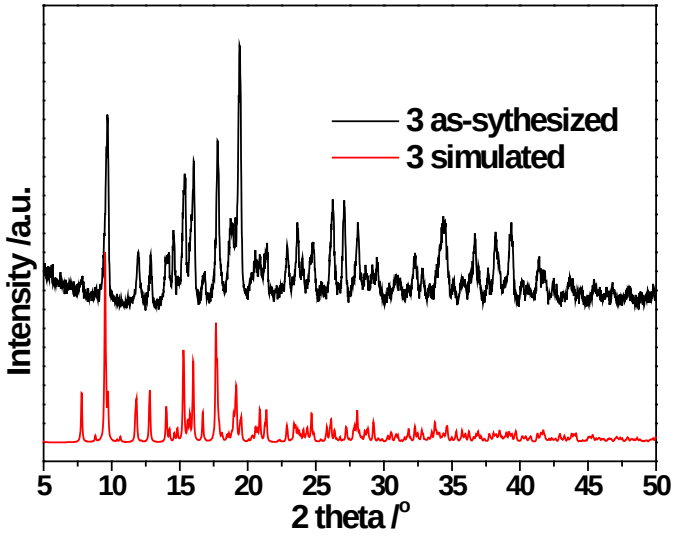
X-ray crystallography. Diffraction data for compounds **1**-**3** and **1'** were collected on a Bruker Smart APEX II diffractometer equipped with Mo-*K*α (λ = 0.71073 Å) radiation. Diffraction data analysis and reduction were performed within *SMART*, *SAINT*, and *XPREP*.[2] Correction for Lorentz, polarization, and absorption effects were performed within *SADABS*.[3] Structures were solved using Patterson method within *SHELXS-97* and refined using *SHELXL-97*.[4-6] All non-hydrogen atoms were refined with anisotropic thermal parameters. The H atoms of dps, acetonitrile, and ethanol were calculated at idealized positions and included in the refinement in a riding mode with U_{iso} for H assigned as 1.2 or 1.5 times U_{eq} of the attached atoms. The H atoms bound to water molecules were located from difference Fourier maps and refined as riding with $U_{iso}(H) = 1.5U_{eq}(O)$. There is a disordered C13 atom in compound **2**, for which an anisotropic refinement was not permissible, and this atom was refined as isotropically.



(*a*)

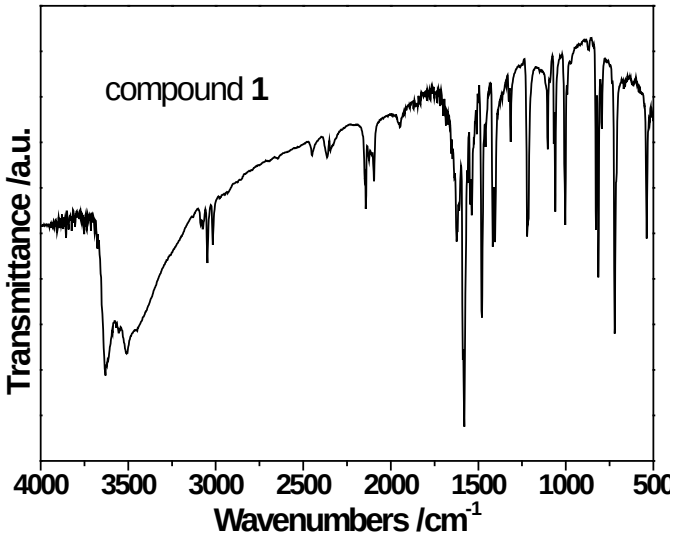


(*b*)

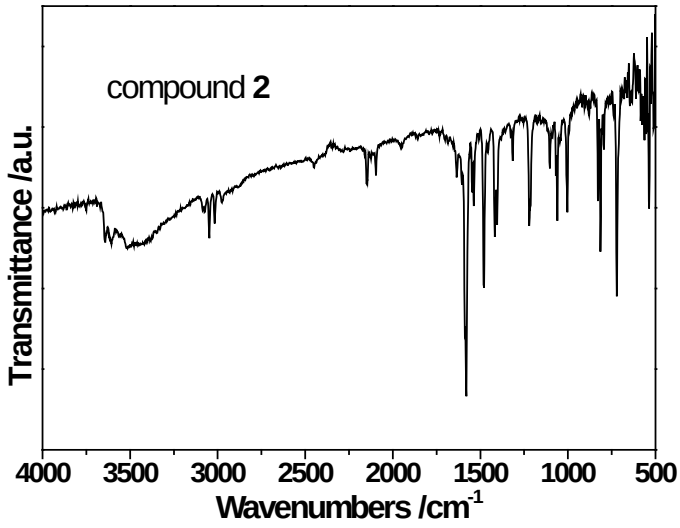


(*c*)

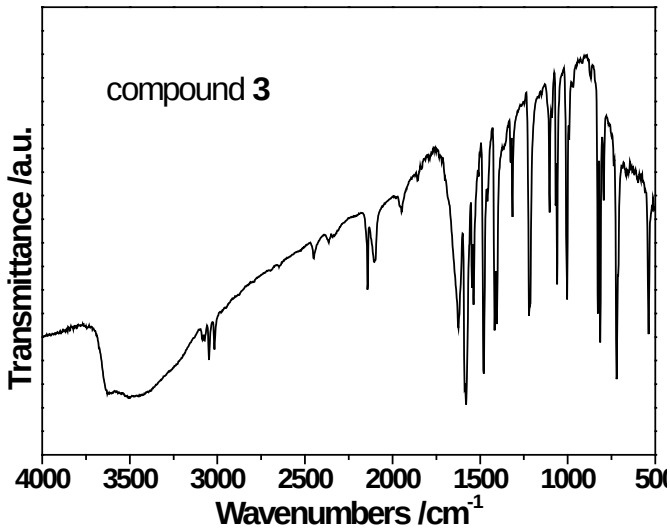
Figure S1 Powder XRD patterns of as-synthesized and simulated from single-crystal data of compounds (*a*) **1**, (*b*) **2**, and (*c*) **3**.



(*a*)



(*b*)



(*c*)

Figure S2 IR spectra of compounds (*a*) **1**, (*b*) **2**, and (c) **3**.

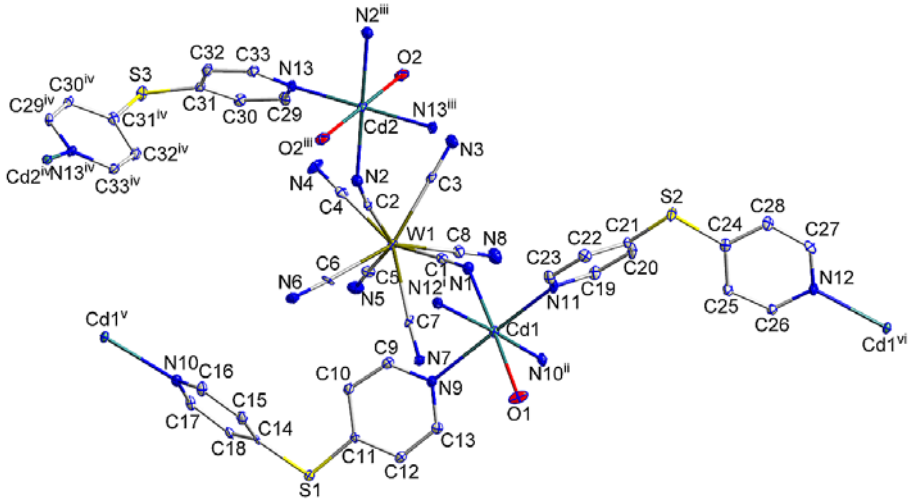


Figure S3 ORTEP diagram of compound **2** with thermal ellipsoids at the 30% probability level. All hydrogen atoms and crystallized water molecules were omitted for clarity. Symmetry codes: (i) $x, -y-1, z-1/2$; (ii) $x, -y, z+1/2$; (iii) $-x, -y, -z$; (iv) $-x, y, -z-1/2$; (v) $x, -y, z-1/2$; (vi) $x, -y-1, z+1/2$. The Cd2 and S3 atoms lie on an inversion centre and a twofold axis, respectively.

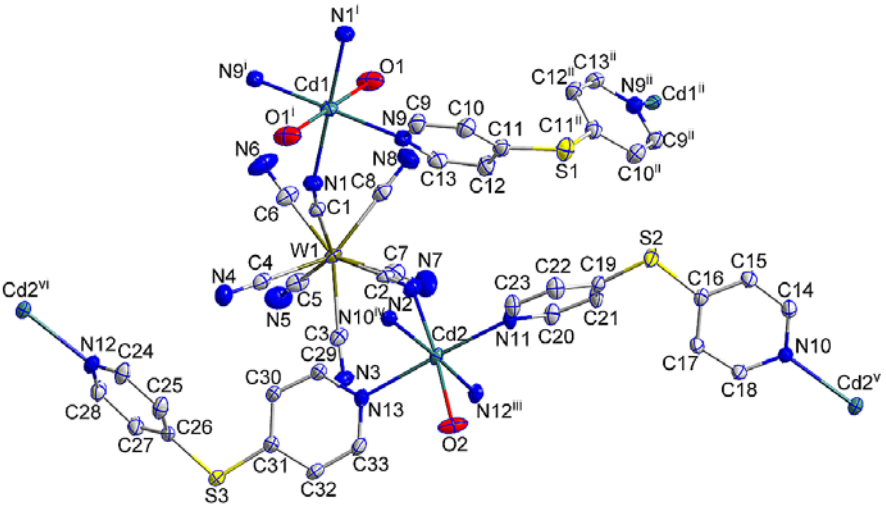


Figure S4 ORTEP diagram of compound **3** with thermal ellipsoids at the 30% probability level. All hydrogen atoms, crystallized ethanol molecules were omitted for clarity. Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, y, -z+1/2$; (iii) $x, -y+1, z+1/2$; (iv) $x, -y, z-1/2$; (v) $x, -y, z+1/2$; (vi) $x, -y+1, z-1/2$. The Cd1 and S1 atoms lie on an inversion centre and a twofold axis, respectively.

References

1. L. D. C. Bok, J. G. Leipoldt and S. S. Basson, *Z. Anorg. Allg. Chem*., 1975, **415**, 81-83.
2. Bruker; *SMART*, *SAINT* and *XPREP*: *Area Detector Control and Data Integration and Reduction Software*, Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA, 1995.

3. G. M. Sheldrick, *SADABS*: *Empirical Absorption and Correction Software*, University of Göttingen, Göttingen, Germany, 1996.
4. G. M. Sheldrick, *SHELXS-97*. *Program for X-ray Crystal Structure Determination*; Göttingen University: Göttingen, Germany, 1997.
5. G. M. Sheldrick, *SHELXL-97*. *Program for X-ray Crystal Structure Determination*; Göttingen University: Göttingen, Germany, 1997.
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