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Zhi-Hong Lei^a & Xia Li^a

^a Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China

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Two cadmium(II) addition compounds with 3-hydroxy-2,7-naphthalenedisulfonate containing aromatic diamines and their 3-D net

ZHI-HONG LEI and XIA LI*

Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China

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{[CdCl(2,2'-bipy)₂(H₂O)]⁺[Cd(3-O⁻-2,7-NDS)(2,2'-bipy)₂]⁻·3H₂O} (1) and {[Cd(phen)₃]²⁺·2[Cd(3-O⁻-2,7-NDS)(phen)₂]⁻·8.5H₂O} (2) (3-OH-2,7-NDS = 3-hydroxy-2,7-naphthalenedisulfonate, phen = 1,10-phenanthroline, and 2,2'-bipy = 2,2'-bipyridine) were prepared and characterized by X-ray single-crystal diffraction. Compound 1 contains a discrete coordination cation [CdCl(2,2'-bipy)₂(H₂O)]⁺ and a coordination anion [Cd(3-O⁻-2,7-NDS)(2,2'-bipy)₂]⁻; 2 contains a discrete coordination cation [Cd(phen)₃]²⁺ and two coordination anions [Cd(3-O⁻-2,7-NDS)(phen)₂]⁻. There are numerous weak interactions among the coordination cation, coordination anion, and free water molecules, such as O–H···O hydrogen bonds, π···π stacking, and Cl⁻···π interactions in 1 and π···π stacking and C–H···π interactions in 2. The cations and anions as building blocks are connected to construct different 3-D supramolecular architectures *via* weak intermolecular interactions. Particularly, the capsule structure of 1 was observed.

Keywords: Cadmium addition compound; 3-Hydroxy-2,7-naphthalenedisulfonate; Aromatic diamine; Crystal structure

1. Introduction

The crystal engineering of coordination polymers and supramolecules have attracted attention due to their fascinating architectures and potential applications in catalysis, magnetism, non-linear optics, fluorescent sensing, etc., [1–7]. Numerous metal complexes with polycarboxylates [8–14], phosphonates [15, 16], and sulfonates [4–6, 17–32] have been synthesized. The sulfonic group is regarded as weakly coordinating. Aromatic diamines 1,10-phenanthroline (phen) and 2,2'-bipyridine (2,2'-bipy) are frequently used in constructing new compounds that display different structural motifs [23–30]. Furthermore, they facilitate supramolecular self-assembly *via* π-stacking and hydrogen-bonding interactions [28–32]. 3-Hydroxy-2,7-naphthalenedisulfonate (3-OH-2,7-NDS) has three potential coordinating groups, one –OH and two –SO₃H groups. The –SO₃H group can coordinate in a variety of modes; crystal structures containing 3-OH-2,7-NDS are not found in the literature. Using 3-OH-2,7-NDS and 2,2'-bipy/phen as ligands, we synthesized [CdCl(2,2'-bipy)₂(H₂O)]⁺[Cd(3-O⁻-2,

*Corresponding author. Email: xiali@mail.cnu.edu.cn

7-NDS)(2,2'-bipy)₂]⁻·3H₂O (**1**) and [[Cd(phen)₃]²⁺·2[Cd(3-O⁻-2,7-NDS)(phen)₂]⁻·8.5H₂O (**2**) compounds consisting of different coordination cations and anions. The different 3-D architectures of **1** and **2** are constructed by cations and anions with weak intermolecular interactions.

2. Experimental

2.1. Synthesis of the complexes

2.1.1. Synthesis of 1. CdCl₂ (0.25 mmol) and 3-OH-2,7-NDSNa₂ (0.25 mmol) were dissolved in 15 mL deionized water. The pH of the solution was adjusted to 5–6 with 2 mol L⁻¹ NaOH aqueous solution and 5 mL ethanol solution of 2,2'-bipy (0.25 mmol) was dripped into the solution. The mixture was heated at 60°C with stirring for 2 h and then filtered. Yellow block crystals were collected after 1 month. Yield: 26% (based on Cd). Anal. Calcd (Found) for C₅₀H₄₅Cd₂ClN₈S₂O₁₁ (%): C, 47.68 (47.73); H, 3.58 (3.57); N, 8.90 (8.75). Selected IR (KBr pellet, cm⁻¹): 3455(s, br), 1594(s), 1475(m), 1440(s), 1351(m), 1319(m), 1221(s), 1297(m), 1156(s), 1097(m), 1032(s), 1016(m), 852(w), 769(s), 695(m), 625(w), 526(w), 412(w).

2.1.2. Synthesis of 2. A mixture of CdCl₂ (0.20 mmol), 3-OH-2,7-NDSNa₂ (0.20 mmol), phen (0.4 mmol), 1 mol L⁻¹ NaOH (0.4 mL) aqueous solution, and 7 mL deionized water in a 25 mL Teflon-lined stainless steel autoclave was heated at 160°C for 3 days, and then cooled slowly to room temperature. Yellow flake crystals were obtained. Yield: 23% (based on Cd). Anal. Calcd (Found) for C₁₀₄H₈₃Cd₃N₁₄O_{22.5}S₄ (%): C, 53.06 (53.18); H, 3.55 (3.74); N, 8.32 (8.05). Selected IR (KBr pellet, cm⁻¹): 3443(s, br), 1617(m), 1584(m), 1515(m), 1446(s), 1426(s), 1344(w), 1320(w), 1184(s), 1099(s), 1031(s), 847(s), 729(s), 669(m), 627(m), 615(w), 525(w), 419(w).

2.2. Materials and physical measurements

All chemicals used for the synthesis are of analytical grade and commercially available. Elemental analyses (C, H, N) were performed on an Elementar Vario EL analyzer. IR spectra (4000–400 cm⁻¹) were recorded on a Bruker EQUINOX-55 using KBr pellets. Thermogravimetric analyses (TGA) were carried out on a WCT-1A Thermal Analyzer at a heating rate of 10°C min⁻¹ from room temperature to 1000°C in air.

2.3. X-ray crystal structure determination

The X-ray single-crystal data collections for **1** and **2** were performed on a Bruker SMART APEX II CCD diffractometer equipped with graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) at 296(2) K. Semi-empirical absorption corrections were applied using SADABS [33]. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 using the SHELXL program package [34]. Non-hydrogen atoms in each structure were refined with anisotropic

displacement parameters; hydrogens were placed in idealized positions and allowed to ride on the relevant carbons. All non-hydrogen atoms in the complexes were refined anisotropically. Positions of hydrogens of water were obtained from Fourier-difference maps. In **2**, hydrogens of water cannot be found even in high refinement, and their hydrogens were thus not added in the final refinement. A summary of the crystallographic data and details of the structure refinement is listed in table 1. Selected bond distances and angles are listed in tables S1 and S2.

3. Results and discussion

3.1. Crystal structure of **1**

Complex **1** is constructed from a discrete coordination cation $[\text{CdCl}(\text{2,2}'\text{-bipy})_2(\text{H}_2\text{O})]^+$, coordination anion $[\text{Cd}(\text{3-O}^- \text{-2,7-NDS})(\text{2,2}'\text{-bipy})_2]^-$, and three free water molecules (figure 1). Cd1 and Cd2 are octahedral with different coordination environments. In the anion, Cd1 is chelated by two 2,2'-bipys and one 3-O⁻-2,7-NDS anion with its hydroxyl and sulfonate oxygens. In the cation, Cd2 is coordinated by two 2,2'-bipy molecules, one chloride, and one water molecule. The average bond distances of Cd1–N and Cd1–O are 2.342 and 2.277 Å, respectively. The average bond distances of Cd2–N, Cd2–O(water), and Cd2–Cl are 2.360, 2.374, and 2.4730 Å, respectively.

Table 1. Crystallographic data and structure refinement for **1** and **2**.

Complex	1	2
Empirical formula	C ₅₀ H ₄₅ Cd ₂ ClN ₈ S ₂ O ₁₁	C ₂₀₈ H ₁₃₂ Cd ₆ N ₂₈ O ₄₅ S ₈
Formula weight	1258.35	4674.44
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$ (No. 2)	<i>P</i> 2 ₁ / <i>c</i> (No. 14)
Unit cell dimensions (Å, °)		
<i>a</i>	14.0576(3)	29.6310(9)
<i>b</i>	14.1926(3)	15.5368(4)
<i>c</i>	15.9382(4)	23.9776(6)
α	91.352(1)	90.00
β	113.928(1)	112.239(2)
γ	115.281(1)	90.00
Volume (Å ³), <i>Z</i>	2552.62(11), 2	102175(5), 2
Calculated density (Mg m ⁻³)	2.016	1.519
Absorption coefficient (mm ⁻¹)	1.036	0.779
<i>F</i> (000)	1268	4704
Crystal size (mm ³)	0.30 × 0.20 × 0.10	0.40 × 0.30 × 0.12
Temperature (K)	296(2)	296(2)
Radiation (Å)	(Mo-K α) 0.71073	(Mo-K α) 0.71073
θ range for data collection (°)	1.4–27.96	1.51–23.15
Limiting indices	–18 ≤ <i>h</i> ≤ 18; –18 ≤ <i>k</i> ≤ 18; –20 ≤ <i>l</i> ≤ 18	–29 ≤ <i>h</i> ≤ 32; –17 ≤ <i>k</i> ≤ 17; –26 ≤ <i>l</i> ≤ 26
Reflections collected/unique	39,690/12286	108,410/14487
Reflections observed [<i>I</i> > 2 σ (<i>I</i>)]	9943	9612
Data/restraints/parameters	11,992/9/667	14,487/1/1354
Goodness-of-fit on <i>F</i> ²	1.106	1.026
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0293, <i>wR</i> ₂ = 0.0705	<i>R</i> ₁ = 0.0660, <i>wR</i> ₂ = 0.1814
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0387, <i>wR</i> ₂ = 0.0760	<i>R</i> ₁ = 0.0990, <i>wR</i> ₂ = 0.2079
Largest difference peak and hole (e Å ⁻³)	–0.686 and 0.744	–0.620 and 0.940

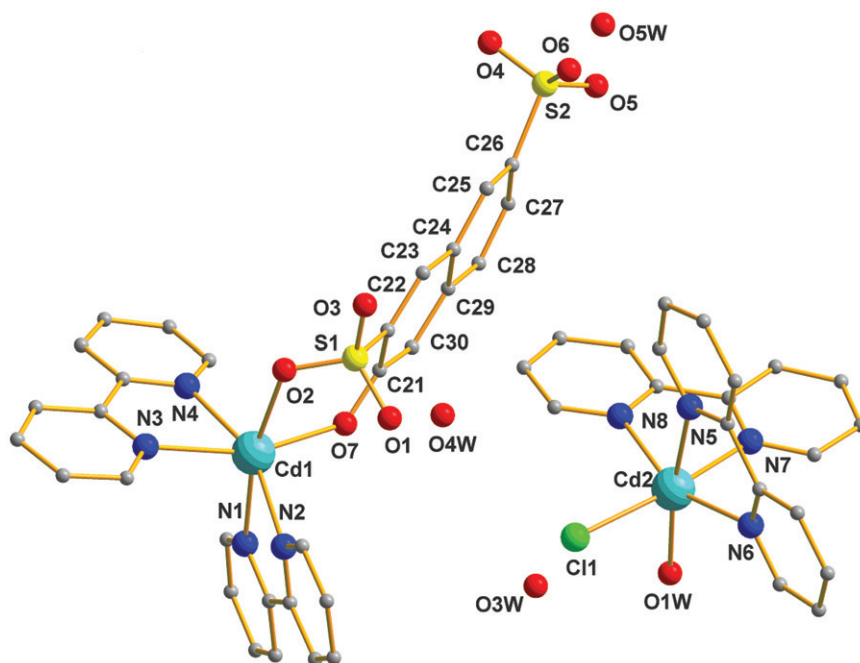


Figure 1. Molecular structure of **1** (30% probability ellipsoids). Hydrogens are omitted for clarity.

There are numerous weak interactions among the coordination cation, coordination anion, and free water molecules (figure S1). O–H \cdots O hydrogen bonds exist among cations, anions, and water molecules. The O \cdots O distances range from 2.738(4) to 2.984(4) Å (table S3). $\pi\cdots\pi$ stacking interactions are observed between pyridine rings from adjacent coordination anions and adjacent coordination cations with centroid–centroid distances of 3.585 and 3.800 Å, respectively. Interaction exists between chloride of the coordination cation and pyridine of the coordination anion at 3.433(2) Å. Cl $^-\cdots\pi$ charge-assisted interaction is unusual and different from other reported anion– π interactions [35, 36]. Coordination anions bind to free water *via* intermolecular interactions, resulting in anionic cavities occupied by the coordination cations. Thus, a guest–host 3-D supramolecular structure is formed (figure 2).

3.2. Crystal structure of **2**

Complex **2** is constructed from [Cd(phen) $_3$] $^{2+}$, two coordination anions, [Cd(3-O $^-$ -2,7-NDS)(phen) $_2$] $^-$, and free water molecules (figure 3). There are three cadmium ions, Cd1, Cd2, and Cd3, with octahedral coordination environments. Cd1 and Cd2 have similar coordination, chelated by two phen and 3-O $^-$ -2,7-NDS $^{3-}$ anion with its sulfonate and hydroxyl oxygens. The coordination sphere about Cd3 is chelated by three phens, comparable to the previously reported [Co(phen) $_3$] $^{2+}$ [29]. The bond distances of Cd1–N, Cd1–O, Cd2–N, Cd2–O, and Cd3–N are 2.359, 2.276, 2.344, 2.265, and 2.334 Å, respectively.

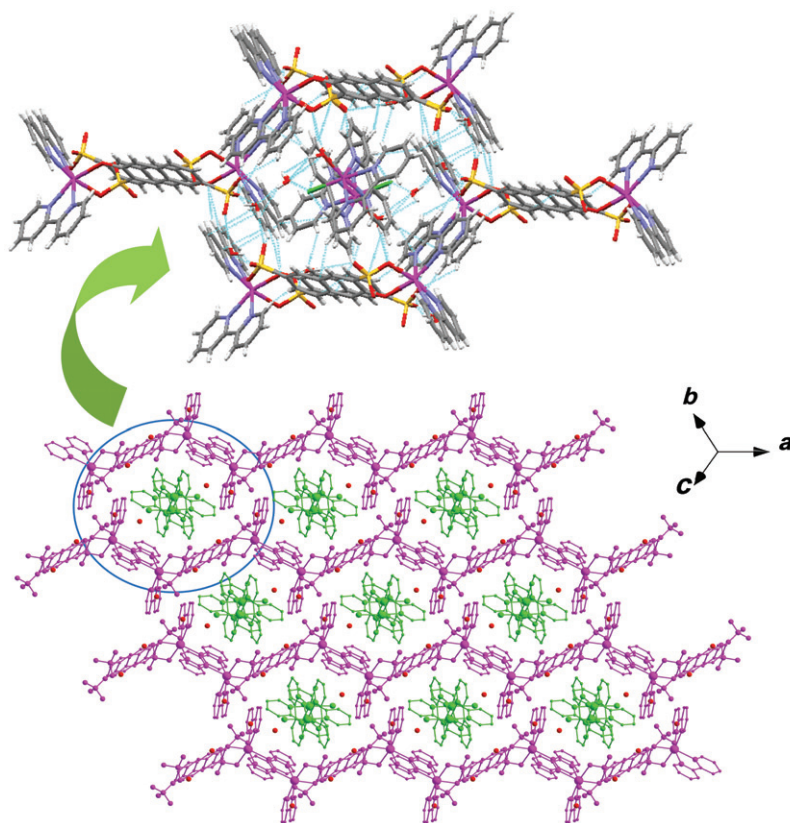


Figure 2. 3-D clathrate structure of **1** (color code: $[\text{Cd}(3\text{-O}^- \text{-}2,7\text{-NDS})(2,2'\text{-bipy})_2]^-$, pink; $[\text{CdCl}(2,2'\text{-bipy})_2(\text{H}_2\text{O})]^+$, green; free water molecules, red).

$[\text{Cd}(\text{phen})_3]^{2+}$ and $[\text{Cd}(3\text{-O}^- \text{-}2,7\text{-NDS})(\text{phen})_2]^-$ interdigitate with each other, stacking along the *b* crystal direction to establish the pseudo-3-D structure (figure 4). The $\pi \cdots \pi$ stacking interactions are observed between phen rings from adjacent coordination anions or cations (figure S2). The centroid–centroid separations range from 3.559 to 3.969 Å. C–H $\cdots\pi$ hydrogen bonds form between C–H(phen) of the anion and phen of the cation (figure S2). The C $\cdots\pi$ distances range from 3.689(16) to 3.812(15) Å. Some water molecules are disordered in the crystal lattice. The pseudo-3-D supramolecular structure is strongly stabilized by π -stacking interactions and hydrogen bonds.

3.3. Thermogravimetric analysis

TGA-DTA analyses of **1** and **2** were studied from room temperature to 1000°C (figure S3). The first weight losses of 5.98% for **1** and 9.80% for **2** occurred at 53–110°C and 50–110°C, respectively, from the removal of lattice water molecule (Calcd 5.72% for **1** and 11.00% for **2**). At 210°C for **1** and 280°C for **2**, there is no other weight loss, which

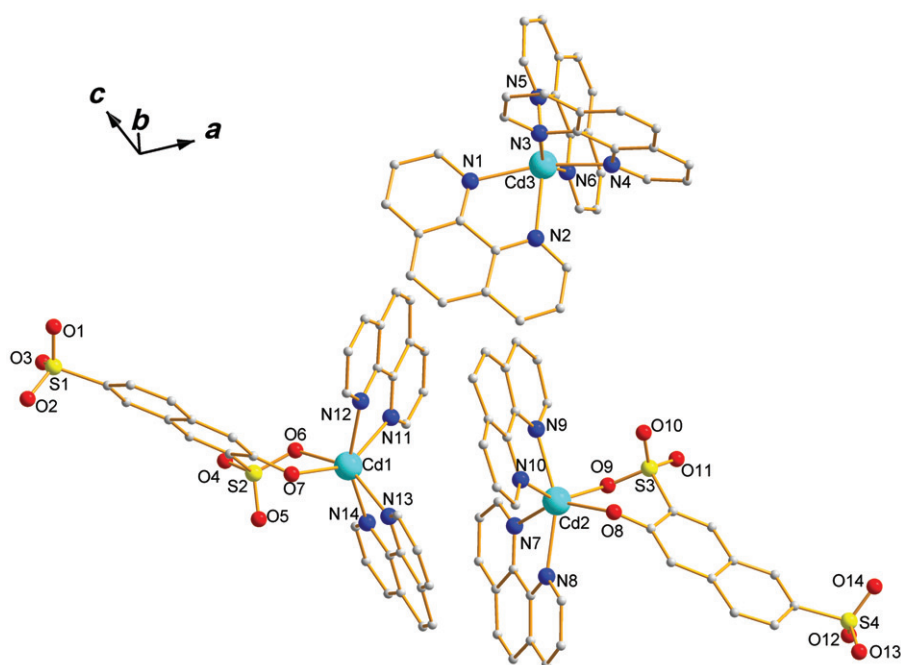


Figure 3. Molecular structure of **2** (30% probability ellipsoids). Hydrogens and free water molecules are omitted.

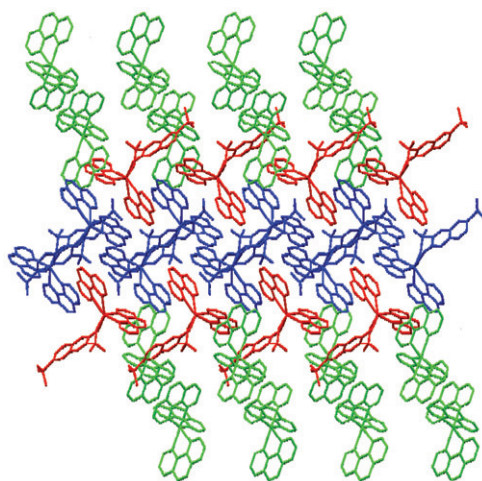


Figure 4. 3-D supramolecular structure of **2** down the *b*-axis; hydrogens and free water molecules are omitted for clarity (color code: [Cd1(3-O⁻-2,7-NDS)(phen)₂]⁻, blue; [Cd2(3-O⁻-2,7-NDS)(phen)₂]⁻, red; [Cd3(phen)₃]²⁺, green).

indicates that the dehydrated frameworks are thermally stable. Above 674°C for **1** and 679°C for **2**, the complexes were completely degraded and the final residue of CdO was obtained [37, 38]. The total weight losses are 82.95% for **1** and 86.76% for **2**, respectively (Calcd 79.60% for **1** and 84.31% for **2**).

4. Conclusion

Reactions of CdCl_2 with 3-OH-2,7-NDS and 2,2'-bipy/phen gave **1** and **2**. The two complexes are different from other metal sulfonate complexes containing phen/2,2'-bipy [23–30], such as $[\text{Zn}(\text{phen})_2(\text{H}_2\text{O})(2,6\text{-NDS})]\cdot 2\text{H}_2\text{O}$, $[\text{Zn}(\text{phen})_2(2,6\text{-NDS})]\cdot \text{H}_2\text{O}$, and $[\text{Zn}(2,2'\text{-bipy})_2(\text{H}_2\text{O})(2,6\text{-NDS})]\cdot 3\text{H}_2\text{O}$ (2,6-NDS = 2,6-naphthalenedisulfonate) [23]. The three zinc complexes are all mononuclear with a crystallographic-independent zinc and monodentate 2,6-NDS. The complexes show unusual structures that contain an intriguing unit cell having two or three crystallographic independent metal ions. The coordination anions result from weak coordination of 3-O⁻-2,7-NDS³⁻ and chelating 2,2'-bipy/phen; 3-O⁻-2,7-NDS³⁻ is a bidentate terminal ligand using sulfonate and hydroxyl oxygens to chelate Cd^{2+} . The cation, anion, and lattice water molecules are interconnected into 3-D architectures *via* intermolecular interactions. We have shown that 2,2'-bipy/phen can be used with weakly coordinating sulfonates to facilitate supramolecular self-assembly through aromatic–aromatic or C–H \cdots aromatic interactions. In addition, the presence of different N-ligands resulted in significant differences in compositions and packing structures of the two compounds.

Supplementary material

Crystallographic data for the compounds have been deposited with the Cambridge Crystallographic Data Centre. CCDC reference numbers are 773223 for **1** and 758594 for **2**. Copies of the information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Tel: (44) 01223 762910; Fax: (44) 01223 336033; E-mail: deposit@ccdc.cam.ac.uk or visit website: <http://www.ccdc.cam.ac.uk/deposit>).

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