两个含[MS$_4$Cu$_4$]簇核的一维配位聚合物
{[MS$_4$Cu$_4$(bpe)$_2$(ani)$_2$I$_2$]·3.5ani}$_n$ (M=Mo，W；ani=苯胺)
的组装及其晶体结构

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摘要： 前驱团簇[Et$_4$N]$_4$[MS$_4$Cu$_4$I$_6$](M=Mo (**1a**)；W (**1b**))与双齿桥连配体 1,2-双(4-吡啶基)乙烷(bpe)在苯胺溶液中反应，生成 2 个结构相似的一维[MS$_4$Cu$_4$]团簇基配位聚合物{[MS$_4$Cu$_4$(bpe)$_2$(ani)$_2$I$_2$]·3.5ani}$_n$ (M=Mo (**2**)，W (**3**)；ani=苯胺)。通过元素分析、红外光谱和 X-射线单晶衍射对 **2** 和 **3** 进行了表征。晶体结构分析表明前驱团簇 **1a** 和 **1b** 中五核马鞍形[MS$_4$Cu$_4$]簇核分别在 **2** 和 **3** 中得以保留，2 个桥连配体 bpe 连接相邻的簇核，在[111]方向延伸形成一维"Z"字形链结构。

关键词： Mo(W)-Cu-S 簇；苯胺；配位聚合物；组装；晶体结构
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Two One-Dimensional [MS$_4$Cu$_4$]-Supported Coordination Polymers: Assembly and Crystal Structures of {[MS$_4$Cu$_4$(bpe)$_2$(ani)$_2$I$_2$]·3.5ani}$_n$ (M=Mo, W; ani=aniline)

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Abstract: Reactions of the preformed cluster precursors [Et$_4$N]$_4$[MS$_4$Cu$_4$I$_6$] (M=Mo (**1a**); W (**1b**)) with a ditopic bridging ligand 1,2-bis(4-pyridyl)ethane (bpe) in aniline solutions gave rise to two analogous one-dimensional cluster-supported coordination polymers {[MS$_4$Cu$_4$(bpe)$_2$(ani)$_2$I$_2$]·3.5ani}$_n$ (M=Mo (**2**), W (**3**); ani=aniline). Both **2** and **3** are characterized by elemental analysis, IR spectra, and single crystal X-ray diffraction. X-ray analyses revealed that the pentanuclear saddle-shaped [MS$_4$Cu$_4$] cores in **1a** and **1b** are retained in **2** and **3**. The adjacent [MS$_4$Cu$_4$] cores are interconnected by double bpe bridges to afford one-dimensional zigzag chains extending along the [111] direction. CCDC: 685673, **2**; 685674, **3**.

Key words: Mo(W)-Cu-S cluster; aniline; coordination polymer; assembly; crystal structure

0　Introduction

In view of their intriguing bonding, unique physical and chemical characteristics, the assembly of discrete transition metal clusters derived from [MO$_x$S$_{4-x}$]$^{2-}$ (M= Mo, W; x=0~3) into extended molecular assemblies has been the focus of intense research[1-10]. The succe-ssful combination of the versatile cluster joints and a

wide range of bridging ligands have offered a powerful tool for the construction of a new family of materials with aesthetically appealing structures, tailor-made properties and advanced functions. For example, the reactions of $[WES_3]^{2-}$ (E=S, O) with CuX (X=NCS, CN, I) in the presence of 1,4-bis (imidazole-1-ylmethyl) benzene under solvothermal conditions gave rise to a family of topologically interesting cluster-supported polymeric assemblies[11]. We demonstrated that, by employing the preformed saddle-shaped cluster precursor $[Et_4N]_4[WS_4Cu_4I_6]$ as a starting material, its reaction with a ditopic ligand 4,4′-bipyridine (4,4′-bipy) in MeCN produced a novel 3D porous framework of $\{[WS_4Cu_4(4,4′\text{-bipy})_4][WS_4Cu_4I_4(4,4′\text{-bipy})_2]\}_n$ which showed interesting host behaviors towards elemental I_2[12]. Further attempts using $[Et_4N]_4[WS_4Cu_4I_6]$ and longer spacers such as 1,2-bis(4-pyridyl)ethane, 1,2-bis (4-pyridyl)ethylene and 1,3-bis(4-pyridyl)propane under similar conditions in the pursue of new structural motifs with enlarged pore sizes remained unsuccessful. This is probably due to the extremely fast reaction rates in MeCN or DMF solutions and low solubility of the resultant precipitates. To tackle this problem, we carried out the reactions of $[Et_4N]_4[MS_4Cu_4I_6]$ (M=Mo (**1a**); W (**1b**)) with 1,2-bis(4-pyridyl) ethane (bpe) in aniline solution. Aniline has proven a promising solvent due to its high solubility toward a series of discrete cluster precursors as well as some high dimensional arrays such as $[\{(\eta^5\text{-}C_5Me_5)MoS_3Cu_3\}_2 (NCS)_3(\mu\text{-NCS})(bpe)_3]_n$ (2D network), $[(\eta^5\text{-}C_5Me_5)MoS_3Cu_3 (NCS)(\mu\text{-NCS})(H_2tpyp)_{0.4}(Cu\text{-tpyp})_{0.1}]_n$ (2D network; H_2tpyp=5, 10, 15, 20-tetra(4-pyridyl)-21H,23H-porphyrin) and $[(\eta^5\text{-}C_5Me_5)MoS_3Cu_3(NCS)_2(1,4\text{-pyz})]_n$ (3D net; 1,4-pyz=1,4-pyrazine)[13]. Reactions of **1a** and **1b** with bpe in aniline, however, gave rise to two one-dimensional [MS$_4$Cu$_4$]-based coordination polymers $\{[MS_4Cu_4(bpe)_2 (ani)_2I_2] \cdot 3.5ani\}_n$ (M=Mo (**2**), W (**3**); ani=aniline). We herein report their syntheses and crystal structures.

1 Experimental

1.1 General procedure

The two cluster precursors $[Et_4N]_4[MS_4Cu_4I_6]$ (M= Mo (**1a**)[14] or W (**1b**)[15]) were prepared according to the literature methods. All other chemicals were used as purchased. Aniline was freshly distilled under reduced pressure, while other solvents were pre-dried over activated molecular sieves and refluxed over the appropriate drying agents under nitrogen and collected by distillation. The elemental analyses for C, H and N were performed on a Carlo-Erba CHNO-S microanalyzer. IR spectra were recorded on a Varian 1000 FT-IR spectrometer as KBr disks (4 000~400 cm^{-1}).

1.2 Preparation of $\{[MoS_4Cu_4(bpe)_2(ani)_2I_2] \cdot 3.5ani\}_n$ (2)

A dark red solution of **1a** (35 mg, 0.02 mmol) in aniline (2.0 mL) was placed in a zigzag glass tube (30 cm in length, 6 mm in inner diameter) followed by careful addition of 1.5 mL of aniline serving as a buffer band. A solution containing bpe (8 mg, 0.04 mmol) in aniline (1.5 mL) was slowly added onto the buffer band. Finally, diethyl ether (3 mL) was carefully layered onto the top solution and then the glass tube was capped with a rubber plug, which was further sealed with parafilm. The glass tube was allowed to stand at room temperature for 10 days, forming black prisms of **2**, which were collected by filtration and washed with cold aniline and Et$_2$O (1:5, V/V) and dried in vacuo. Yield: 20.3 mg (63%) based on Mo. Anal. Calcd. for $C_{57}H_{62}Cu_4I_2MoN_{9.5}S_4$ (%): C 42.46, H 3.88, N 8.25; Found(%): C 42.56, H 3.88, N 8.20. IR (KBr disk, cm^{-1}): 2 925 (w), 2 045 (w), 1 937 (s), 1 875 (s), 1 798 (w), 1 744 (s), 1 605 (s), 1 544 (w), 1 497 (s), 1 428 (s), 1 382 (m), 1 281 (w), 1 219 (w), 1 065 (w), 1 019 (w), 825 (m), 756 (m), 695 (m), 648 (w), 594 (w), 548 (w), 509 (w), 448 (m).

1.3 Preparation of $\{[WS_4Cu_4(bpe)_2(ani)_2I_2] \cdot 3.5ani\}_n$ (3)

Compound **3** was prepared in a manner similar to that described for **2**, using **1b** (37 mg, 0.02 mmol) and bpe (8 mg, 0.04 mmol) as starting materials. Yield: 27 mg (78%) based on Mo. Anal. Calcd. for $C_{57}H_{62}Cu_4I_2 N_{9.5}S_4W$ (%): C 40.26, H 3.68, N 7.83; Found (%): C 40.18, H 3.60, N 7.69. IR (KBr disk, cm^{-1}): 2 932 (w), 1 937 (w), 1 613 (vs), 1 540 (w), 1 497 (s), 1 428 (s), 1 382 (m), 1 281 (w), 1 227 (w), 1 173 (w), 1 065 (w), 1 018 (w), 880 (w), 826 (m), 756 (m), 695 (m), 548

(w), 509 (w), 440 (m).

1.4　X-ray crystallography

　　X-ray quality crystals of **2** and **3** were obtained directly from the above preparations. The measurements of **2** and **3** were made on a Rigaku Mercury CCD X-ray diffractometer by using graphite-monochromated Mo $K\alpha$　(λ =0.071 073 nm) radiation. Single crystals were mounted with grease at the top of a glass fiber and cooled at 153(2) K in a liquid N_2 stream. Cell parameters were refined by using the program CrystalClear (Rigaku and MSC, version 1.3, 2001). The collected data were reduced by using the program CrystalStructure (Rigaku and MSC, version 3.60, 2004) while an absorption correction (multi-scan) applied. The reflection data were also corrected for Lorentz and polarization effects.

　　The structures were solved by direct methods and refined with a full-matrix least-squares technique using the SHELXTL-97 program package[16]. For both **2** and **3**, all the non-hydrogen atoms were refined anistropically. Since all the nitrogen atoms in aniline molecules were attached to conjugated systems, it was reasonable to assume that each -NH₂ group was coplanar with the phenyl unit, which enabled the two hydrogen atoms to be placed as an ethylenic hydrogen using a riding model with N-H distances of 0.088 nm and $U_{iso}(H)=1.2U_{iso}(N)$. All other hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms, with d(C-H) =0.095 nm for phenyl and d(C-H)=0.099 nm for meth-ylene and with $U_{iso}(H)=1.2U_{iso}(C)$ for all H atoms. Crystal data as well as details of data collection and refinements for these complexes are summarized in Table 1.

　　CCDC: 685673, **2**; 685674, **3**.

Table 1　Crystallographic data and refinement results for 2 and 3

Compound	2	3
Empirical formula	C₁₁₄H₁₂₄Cu₈I₄Mo₂N₁₉S₈	C₁₁₄H₁₂₄Cu₈I₄N₁₉S₈W₂
Formula Weight	3 224.60	3 400.42
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a / nm	1.028 4(2)	1.028 5(2)
b / nm	1.254 2(3)	1.254 3(3)
c / nm	2.485 9(5)	2.490 4(5)
α / (°)	95.39(3)	95.34(3)
β / (°)	98.22(3)	98.24(3)
γ / (°)	100.57(3)	100.70(3)
V / nm³	3.095 6(11)	3.100 3(11)
Z	1	1
D_c / (g·cm⁻³)	1.730	1.821
F(000)	1 597	1 661
μ / mm⁻¹	2.726	4.373
Collected reflns	30 726	30 152
Unique reflns	11 278	11 280
R_{int}	0.087 3	0.088 9
Observed reflections	7 370	7 572
Parameters	685	667
$R_1{}^a$ ($I>2\sigma(I)$)	0.081 2	0.085 2
$wR_2{}^b$	0.152 3	0.151 5
GOF^c	1.101	1.109

　　^{a} $R_1=\sum ||F_o|-|F_c||/\sum |F_o|$; ^{b} $wR_2=\{w\sum (|F_o|-|F_c|)^2/\sum w|F_o|^2\}^{1/2}$; ^{c} GOF=$\{\sum w(|F_o|-|F_c|)^2/(n-p)\}^{1/2}$, where n is the number of reflections and p is total number of parameters refined.

2　Results and discussion

2.1　Synthetic and spectral aspects

Based on our previous observations, aniline usually serves more as a solvent than a ligand due to its relatively weak coordination ability. And only one example $\{[(\eta^5\text{-}C_5Me_5)MoS_3Cu_3(tpt)(aniline)(NCS)_2]\cdot 0.75aniline\cdot0.5H_2O\}_n$　(tpt =2,4,6-tri (4-pyridyl)-1,3,5-triazine) was isolated wherein one aniline molecule coordinated to one Cu(I) centers[13]. Compounds $\{[MS_4Cu_4(bpe)_2(ani)_2I_2]\cdot3.5ani\}_n$ (M =Mo (**2**), W (**3**); ani = aniline) were isolated in relatively high yields by reactions of $[Et_4N]_4[MS_4Cu_4I_6]$ (M =Mo (**1a**); W (**1b**)) with **2** equivalents of bpe in aniline solution. Analogous reactions using different cluster-to-ligand ratios always afforded the same product, suggesting that **2** and **3** are the most stable products in aniline. Compounds **2** and **3** were relatively stable in air, and soluble in aniline, but insoluble in other solvents such as DMF, MeCN, DMSO, CH_2Cl_2 and $CHCl_3$. However, their aniline solutions became unstable and decomposed to some uncharacterizable precipitates if exposed to air for a long time. In both examples, aniline showed three interesting functions. One is that aniline worked as a good solvent for the formation of less soluble M/Cu/S coordination polymers. The second one, as discussed later in this article, is that aniline served as a ligand to coordinate at each Cu(I) center, which prohibits the formation of higher dimensional coordination frameworks. The third one is that the lattice aniline molecules stabilized the one-dimensional chains by strong hydrogen bonding interactions.

The elemental analyses of **2** and **3** were consistent with their formulae. In their IR spectra, the bands at 1 605 (**2**) and 1 613 (**3**) cm^{-1} indicated the presence of bpe ligands, bands at 448 (**2**) and 440 (**3**) were assigned as the bridging Mo(W)-S stretching vibrations. The identities of **2** and **3** were finally confirmed by X-ray crystallographic studies.

2.2　Crystal structures of $\{[MS_4Cu_4(bpe)_2(ani)_2I_2]\cdot 3.5ani\}_n$ (M=Mo (2), W (3))

Because of the similarities of **2** and **3**, only the crystal structure of **2** is discussed in details.

Compound **2** crystallizes in the triclinic space group $P\bar{1}$ and the asymmetric unit contains one neutral $[MoS_4Cu_4(bpe)_2(ani)_2I_2]$ molecule, three and a half aniline solvent molecules. Fig.1 demonstrates the structure of the $[MoS_4Cu_4]$ cluster core of **2** while Table 2 listed its selected bond lengths and angles.
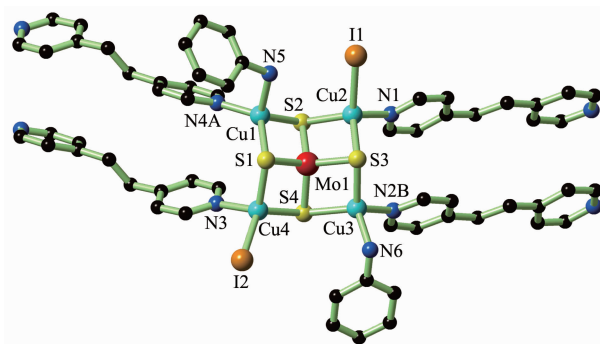
All the lattice solvates and hydrogen atoms are omitted for clarity; Color codes: Mo (red), Cu (cyan), I (orange), S (yellow), N (blue), C (black)

Fig.1　View of the structure of the repeating unit $[MoS_4Cu_4I_2(bpe)_4(ani)_2]$ of **2**

In this core structure, there are four independent Cu(I) centers, all of which adopt a distorted tetrahedral coordination geometry. However, their coordination environments are somewhat different, being coordinated by two μ_3-S atoms from the $[MoS_4]^{2-}$ moiety and two N atoms from one bridging bpe ligand and one aniline ligand (Cu1 and Cu3) or one N atom from one bpe and one terminal iodine atom (Cu2 and Cu4). The mean Mo···Cu contact of 0.267 2 nm is normal and similar to those observed in the precursor compound **1a** (0.266 8 nm)[14], $[MoOS_3Cu_3I(phen)_2]$ (0.263 8 nm, phen=1,10-phenanthroline)[17] and $[MoOS_3Cu_3I(2,2'\text{-}bipy)_2]$ (0.266 4 nm, 2,2'-bipy=2,2'-bipyridine)[18]. The average Mo-S and Cu-S distances (0.223 5 nm vs 0.228 3 nm) are within normal range and comparable to those found in **1a** (0.223 3 nm vs 0.229 3 nm), $[PPh_4]_4[MoOS_3(CuCl)_3][CuCl_2]$ (0.227 2 nm vs 0.224 3 nm)[19] and $[MoOS_3Cu_3(PPh_3)_3Cl]$ (0.225 9 nm vs 0.229 4 nm)[20]. The average Cu-I distance of 0.260 7 nm is slightly shorter than those of **1a** (0.261 8 nm), $[MoS_4Cu_6I_4(py)_4]_n$ (0.267 5 nm, py=pyridine)[21] and $[PPh_4]_4[MoS_4Cu_6I_8]$ (0.261 9 nm)[22]. Additionally, the mean Cu-N(aniline) distance (0.214 4 nm) is slightly longer

Table 2   Selected bond distances (nm) and angles (°) for 2

Mo(1)···Cu(3)	0.264 01(19)	Mo(1)···Cu(1)	0.266 87(19)	Mo(1)···Cu(2)	0.267 40(17)
Mo(1)···Cu(4)	0.270 54(16)	Mo(1)-S(2)	0.223 0(3)	Mo(1)-S(1)	0.223 2(3)
Mo(1)-S(3)	0.223 9(3)	Mo(1)-S(4)	0.224 0(3)	I(1)-Cu(2)	0.263 05(17)
I(2)-Cu(4)	0.258 28(16)	Cu(1)-N(4A)	0.203 3(9)	Cu(1)-N(5)	0.218 4(10)
Cu(1)-S(2)	0.227 3(3)	Cu(1)-S(1)	0.228 6(3)	Cu(2)-N(1)	0.204 4(8)
Cu(2)-S(2)	0.227 0(3)	Cu(2)-S(3)	0.228 4(3)	Cu(3)-N(2B)	0.204 8(8)
Cu(3)-N(6)	0.210 4(9)	Cu(3)-S(4)	0.226 5(3)	Cu(3)-S(3)	0.227 7(3)
Cu(4)-N(3)	0.210 4(9)	Cu(4)-S(4)	0.230 1(3)	Cu(4)-S(1)	0.230 5(3)
S(2)-Mo(1)-S(1)	109.11(11)	S(2)-Mo(1)-S(3)	108.73(11)	S(1)-Mo(1)-S(3)	111.04(10)
S(2)-Mo(1)-S(4)	109.58(11)	S(1)-Mo(1)-S(4)	109.03(11)	S(3)-Mo(1)-S(4)	109.33(11)
S(2)-Cu(1)-S(1)	105.74(11)	S(2)-Cu(2)-S(3)	105.82(11)	S(2)-Cu(2)-I(1)	111.17(9)
S(3)-Cu(2)-I(1)	113.13(9)	S(4)-Cu(3)-S(3)	107.12(11)	S(4)-Cu(4)-S(1)	104.46(11)
S(4)-Cu(4)-I(2)	121.57(9)	S(1)-Cu(4)-I(2)	113.37(9)		

Symmetry transformations used to generate equivalent atoms: A: −x+2, −y+3, −z+1; B: −x, −y+2, −z.

than that of Cu-N (bpe) distance (0.207 4 nm), implying that each aniline molecule is coordinated at Cu(I) center in this cluster core. Interestingly, the two independent bpe ligands show significantly different flexibilities. The dihedral angle between plane N1-C1-C2-C3-C4-C5 and plane C8-C9-C10-N2-C11-C12 is 6.3(3)°, while that between plane N3-C13-C14-C15-C16-C17 and plane C20-C21-C22-N4-C23-C23 is 26.9(7)°. Meanwhile, the torsion angles for C3-C6-C7-C8 and C15-C18-C19-C20 are 177.9(12)° and 175.6(15)°, respectively.

Each [MoS₄Cu₄] cluster core is linked to two equivalent clusters through a pair of bpe ligands to give a one-dimensional (1D) [MoS₄Cu₄]-based zigzag chain extending along the [111] direction (Fig.2). The separation of adjacent chains of *ca.* 1.25 nm is filled with aniline solvents (Fig.3). These 1D chains are interlinked by various types of weak interactions including as π-π, C−H···π, C−H···I, C−H···N, N−H···I interactions among the lattice aniline molecules, the coordinated aniline molecules, bpe and iodine
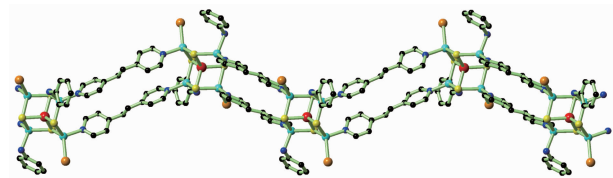
atoms, thereby forming a 2D wave-like network (Fig.3) in the approximately [34$\bar{1}$] direction. Adjacent layers are separated by aniline solvates through a variety of hydrogen bonding interactions and finally formed a three-dimension structure (Fig.4).
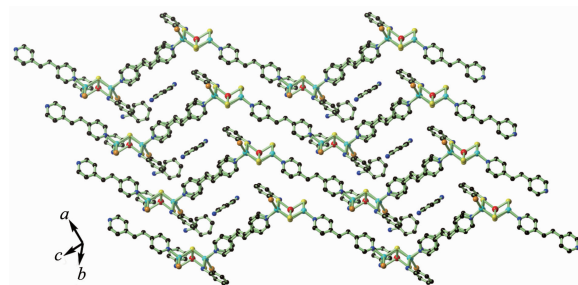

Fig.3　View of the 2D wave-like network formed by weak interactions in **2**, looking down the *a* axis
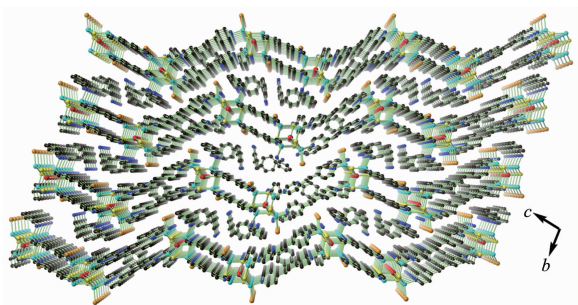

Fig.4　View of the 3D structure formed by hydrogen bonding interactions and other weak interactions in **2**, viewed along the *a* direction

3　Conclusions

In summary, we have synthesized two analogous

Fig.2　View of a section of the 1D zigzag chain of **2** extending along the [111] direction

[MS$_4$Cu$_4$]-supported one-dimensional coordination polymers **2** and **3**. The pentanuclear saddle-shaped [MS$_4$Cu$_4$] cores in **2** and **3** are inherited from **1a** and **1b**, respectively. Pairs of bpe bridges connect adjacent [MS$_4$Cu$_4$] cores and afford a one-dimensional [MS$_4$Cu$_4$]-based zigzag chain. This work also demonstrates that aniline can work as an excellent solvent and ligand in the formation of less soluble M/Cu/S clusters. We are currently exploring other systems using aniline.

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