Supramolecular Compounds

$\{[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\}_\infty$—An Unusual 3D Porous Coordination Polymer Formed from the Preformed Cluster $[Et_4N]_4[WS_4Cu_4I_6]$**

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In recent years, the assembly of supramolecular structures from preformed metal clusters has been the focus of intense research.[1] So far, several preformed metal clusters have been successfully incorporated into infinite assemblies to produce materials with fascinating properties.[1g,h,2–5] For example, Yaghi and co-workers synthesized frameworks that show promise as hydrogen-storage materials by using tetrazinc carboxylate clusters as 6-connecting nodes.[2] Shriver and co-workers employed hexanuclear clusters that contain the octahedral $[Mo_6(\mu_3\text{-}Cl)_8]^{4+}$ core to generate microporous xerogels for size-selective ion exchange.[3] The importance of Mo(W)/Cu(Ag)/S clusters[6] in biological systems[7a] and materials science[7b-d] as well as their unique structure

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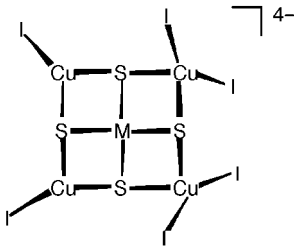
prompted our interest in using such species in the assembly of cluster-containing supramolecular compounds. To date, there have only been a few reports on the use of Mo(W)/Cu(Ag)/S clusters as building blocks for supramolecular assemblies.[8]

After careful consideration of cluster geometry and chemical behavior, we believed the cluster $[Et_4N]_4[WS_4Cu_4I_6]$ (**1**)[9] to be a very good candidate for inclusion into an



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extended network. The tetraanion of **1** has a saddle-shaped cluster framework in which two copper centers are each coordinated by two terminal iodides while each of the other two are coordinated to a single terminal iodide. Given the geometry of **1**, we anticipated that the full or partial substitution of the coordinated iodides with the bridging ligand would give rise to novel structural arrays. Herein, we report the synthesis, structure, and absorption properties of an unusual 3D porous coordination polymer, assembled from **1** and 4,4′-bpy.

Crystals of the compound $\{[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\}\cdot 8\,CH_3CN$ (**2**·8 CH_3CN) were obtained by the reaction of **1** with 2.5 equivalents of 4,4′-bpy in solution in DMF/MeCN. The crystals rapidly lose acetonitrile; however, **2** is stable in air and moisture, and is insoluble in common organic solvents. X-ray crystallographic analysis revealed that the asymmetric unit of **2**·8CH_3CN consists of one quarter of the $[WS_4Cu_4(4,4'\text{-bpy})_4]^{2+}$ dication and one quarter of the $[WS_4Cu_4I_4(4,4'\text{-bpy})_2]^{2-}$ dianion.[10] The oxidation states of the tungsten and copper centers in **1** (+6 and +1, respectively) are retained in **2**. Although the dianion and dication contain a saddle-shaped $[WS_4Cu_4]$ core structure similar to that found in **1**, the Cu centers have different coordination environments. Each Cu atom in the dication of **2** is coordinated by two μ_3-S and two N atoms, whereas in the dianion the Cu atom is tetrahedrally bound by one I, one N, and two μ_3-S atoms. The W···Cu separations in the dication and dianion are 2.673 and 2.719 Å, respectively, which are similar to that found for **1** (av. 2.673 Å).[9]

As indicated in Figure 1, the cationic cluster is coordinated by eight bridging 4,4′-bpy ligands. Four pairs of ligands extend to four crystallographically equivalent clusters that lie at the corners of a distorted tetrahedron. The W atom lies on a site of 222 symmetry. From a topological perspective, each cluster serves as a 4-connecting center in an infinite cationic 3D network. The topology of the net is the same as that found for diamond, with the WS_4Cu_4 clusters serving as tetrahedral nodes. The adamantane-type unit is a characteristic feature of the diamond net, and such a unit within **2** is represented in Figure 2 A. The relationship of the unit represented in

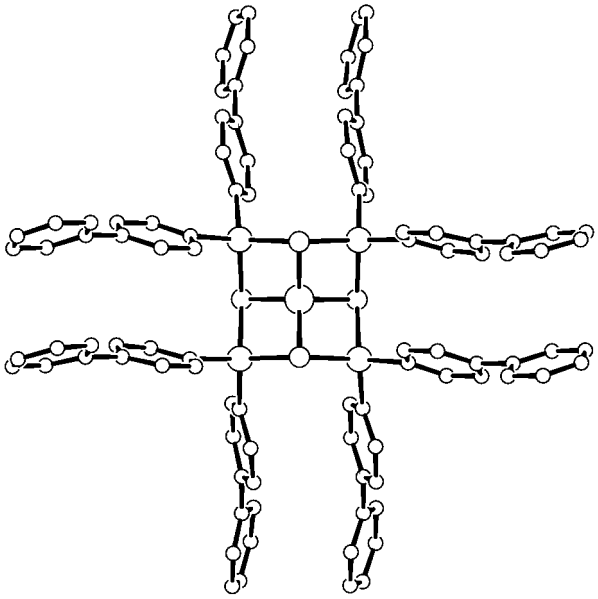

Figure 1. Part of the cationic network within **2** showing the double 4,4′-bpy ligands that extend to equivalent WS_4Cu_4 clusters; hydrogen atoms have been omitted for clarity.

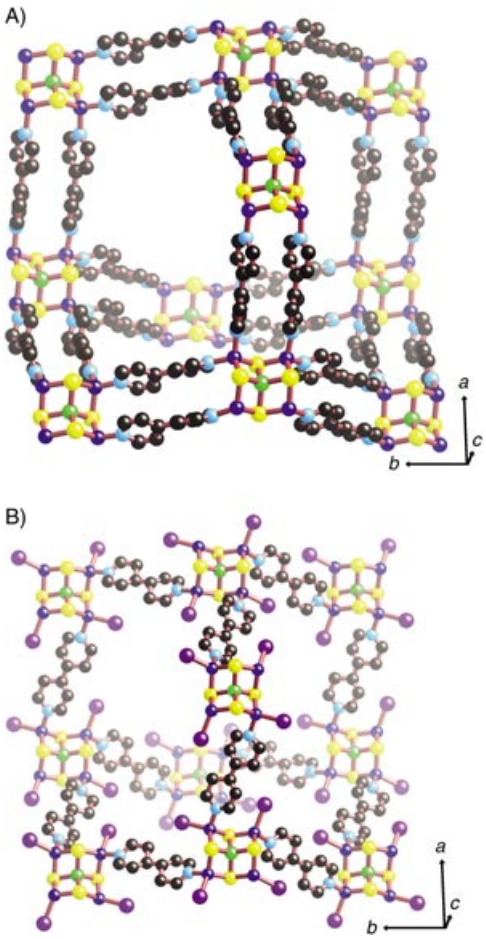

Figure 2. Adamantane-type units within A) the cationic network of **2** and B) the anionic network of **2** viewed from a position just off the *c* axis. Atom color code: W green, Cu royal blue, S yellow, N pale blue, C black; hydrogen atoms have been omitted for clarity. The interpenetrating networks stack along the *c* direction.

Figure 2A with adamantane is highlighted in the Supporting Information. The crystal contains two independent cationic diamond nets that interpenetrate.

In contrast to the cation, the WS_4Cu_4 cluster in the anion retains four iodide ions from the starting material **1** and has its remaining coordination sites occupied by nitrogen atoms from four bridging 4,4′-bpy ligands. The W atom in this case lies on a site of $\bar{4}$ symmetry. The ligands link a cluster to four equivalent clusters that lie at the vertices of a distorted tetrahedron. As with the cation, a diamond-type network results, but now the bridges between clusters are single 4,4′-bpy ligands. An adamantane-related unit is represented in Figure 2B. In the anion, the bridging ligand is inclined to the W···W vector of the linked clusters, whereas in the cation the 4,4′-bpy ligands are parallel to the W···W vector. In the anionic network the 4,4′-bpy makes close contact with a coordinated iodide ion bound to an adjacent Cu center. We consider that this steric clash leads to disorder and slight distortion of the 4,4′-bpy ligands of the anionic network. Within the crystal there are two independent, interpenetrating, anionic diamond networks.

The interpenetration of the four diamond nets (two cationic and two anionic) is remarkable, given that the cationic and anionic nets are so different. Views of the interpenetration of four adamantane units are presented in the Supporting Information. For interpenetration to occur, the spacing of network voids in one network needs to match perfectly the spacing of network nodes in the other and as a consequence, interpenetration is normally observed in crystals that contain identical open networks. Not surprisingly, the occurrence of two different interpenetrating diamondlike nets in the same compound is unusual but not unprecedented.[11]

Although it was not possible to identify solvent molecules in the crystal structure, it was considered likely that large intraframework regions are occupied by solvent molecules. On the basis of the "void" volumes, the formula of the crystal was estimated as **2**·8 CH_3CN. Most of this solvent appears to be lost when the sample is exposed to the atmosphere. Thermogravimetric analysis (TGA) of the compound (see Supporting Information) reveals a weight loss of 2.3% in the region 30–110 °C, consistent with the loss of a small amount of residual acetonitrile from the channels. The compound is stable until ~180 °C; above this temperature a series of decomposition steps commence.

In **2**, the four independent diamondlike networks stack evenly along a direction parallel to the unique *c* axis in such a manner that channels of approximate circular cross-section extending in the *c* direction are produced (Figure 3). Allowing for the van der Waals size of the 4,4′-bpy atoms that line the surface of the channel, it is estimated[12] that each channel has a volume of approximately 880 $Å^3$ over the *c* cell length of the tetragonal unit cell. This corresponds to an average cross-sectional area of ~31 $Å^2$. The channel volume (van der Waals free space) represents 20% of the crystal volume.

When the crystals are dried in vacuo, they fail to diffract as single crystals. However, analysis of the powder diffraction pattern (see Supporting Information) reveals that the framework remains intact upon loss of solvent from the channels.

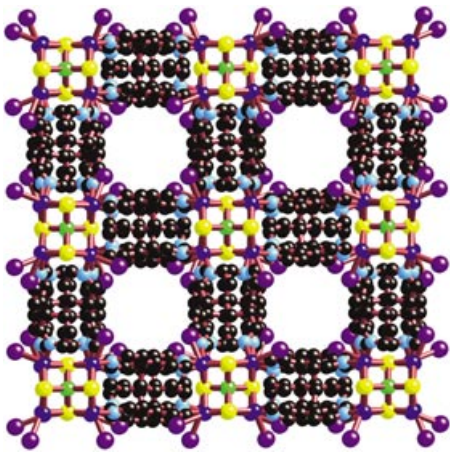

Figure 3. The structure formed from the interpenetration of the four diamond networks (two cationic and two anionic). See Figure 2 for color code; hydrogen atoms have been omitted for clarity. The channels that result from the interpenetration extend in the *c* direction.

The retention of the network structure upon loss of solvent prompted an investigation of whether guest molecules such as I_2 could be incorporated into the channels of the crystal.[13] When crystals are transferred directly from the mother liquor to a CCl_4 solution of I_2, the crystals darken considerably within a matter of minutes to a deep red-brown color, consistent with absorption of iodine. A suitable crystal was transferred directly to protective oil and placed on a diffractometer, where it was cooled to 193 K under a stream of nitrogen. Crystal-structure analysis indicated that the crystal was essentially identical to the parent crystal, except for the fact that I_2 guest molecules were now included in the channels.[10] The crystal formula was found to be **2**·2 I_2. Although the channels are large enough to hold CCl_4, the channels preferentially absorb I_2. The crystal-structure analysis revealed that the iodine is associated with the coordinated iodide. The I_2 molecule slots neatly into a space between coordinated iodide ions of two bridged clusters of an anionic net to form a Cu-I-I-I-I-Cu bridge that runs parallel to the 4,4′-bpy bridge. As indicated in Figure 4, the iodine atoms in this bridge are not evenly spaced and, in fact, may be considered as an I_2 molecule that forms interactions with a pair of coordinated iodide ions. A representation of the channels showing the I_4^{2-} bridges is presented in the Supporting Information. Examples of M-I-I-I-I-M bridges are well known[14,15] (M = a wide variety of metals), and

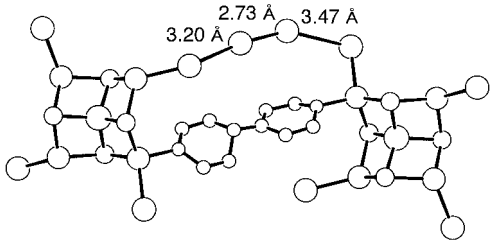

Figure 4. The I_4^{2-} link between two clusters of the anionic network in **2**·2 I_2; hydrogen atoms have been omitted for clarity.

although there is some variation in the geometry of the I_4^{2-} ion, most examples seem to have a short separation between the central iodine atoms, with longer contacts to the terminal iodides. The Raman spectrum of **2**·2 I_2 (see Supporting Information) shows a band at 175 cm^{-1}, which is absent in the spectrum of **2**, and is presumably due to the incorporation of iodine. In the compound $CrPcI_2 \cdot I_2$ (H_2Pc = phthalocyanine), in which iodine molecules are held between coordinated iodides, the vibrational band is at 180 cm^{-1}.[16] Pure I_2 has a vibrational band at 185 cm^{-1}.

As indicated earlier, precautions need to be taken to protect the crystals from iodine loss. If the crystals are exposed to the atmosphere then some iodine is lost relatively easily to form crystals of formula **2**·0.9 I_2 (See Supporting Information). These crystals can withstand washing with solvents (carbon tetrachloride and diethyl ether) without further loss of I_2, however the single-crystal character appears to be affected in the transition from **2**·I_2 to **2**·0.9 I_2. TGA indicates that **2**·0.9 I_2 starts to lose iodine (not the coordinated iodide) at $\approx$60 °C (see Supporting Information), and the mass loss is consistent with the chemical analysis. Following the loss of the iodine, the TGA results are similar to those of the parent compound. Although it may be reasonably argued that the relative ease with which at least half the iodine is lost from **2**·2 I_2 indicates only a weak association, it is clear that the coordinated iodide ions belonging to neighboring clusters of a single net provide a simple and effective receptor site for a guest iodine molecule. Not only are the crystals capable of absorbing iodine from solution, but they can also absorb iodine vapor to form a more deeply colored material (see Supporting Information for photograph). The successful inclusion of the iodine prompted an investigation of whether other guests could be accommodated within the channels. When crystals are placed in neat aniline, the crystals absorb the aniline to form a compound of formula **2**·4.2 aniline. TGA measurements show the loss of $\approx$13 % mass which corresponds to the loss of aniline from the host crystal (see Supporting Information). The incorporation of the aniline despite the absence of a specific receptor site suggests that a range of suitably sized guests may be incorporated into channels.

In summary, we have demonstrated herein a facile synthesis of a novel supramolecular compound through the reaction of a preformed cluster, **1**, with 4,4′-bpy. A particularly novel feature of **2** is its ability to bind guest iodine molecules between a pair of coordinated iodide ions. Although the host properties of a wide range of coordination polymers have been previously investigated, this present work provides strong encouragement that specific receptor sites can be included in coordination polymers. We consider that there is a rich variety of finite and infinite networks that may be constructed by linking copper thiotungstate clusters with a range of ligand bridges. Work is continuing in this area.

Experimental Section

2: A "buffer band" of CH_3CN (3 mL) was carefully added to a zigzag glass tube (60 mm in diameter) containing a red solution of **1** (0.037 g, 0.02 mmol) in DMF (1 mL). This was followed by the slow addition of a solution of 4,4′-bpy (0.009 g, 0.05 mmol) in CH_3CN (2.5 mL). Finally, diethyl ether (2 mL) was carefully layered onto the solution. The glass tube was sealed and left to stand at room temperature; red prisms of **2** contaminated with some red precipitate formed in the glass tube after 5 days. Crystals for single-crystal structure analysis were transferred directly to protective oil. The prisms **2** were mechanically separated from the precipitate under a microscope. Crystals of **2** were washed thoroughly with CH_3CN and Et_2O and dried in vacuo. Yield: 6.2 mg, 24 %. Elemental analysis: calcd for $C_{60}H_{48}Cu_8I_4N_{12}S_8W_2$ (%): C 27.96, H 1.88, N 6.52, I 19.71, Cu 19.73, W 14.27; found: C 27.89, H 1.86, N 6.64, I 19.41, Cu 19.52, W 13.78; IR (KBr pellet): $\tilde{\nu} = \nu$(W-S_{br}), 439 (s) cm^{-1}.

2·2 I_2: Crystals of **2** were transferred from the mother liquor to a solution of iodine in CCl_4 and left immersed for several minutes. A suitable crystal was transferred to protective oil and placed in a stream of nitrogen at 193 K on a single-crystal diffractometer. The crystal analysis showed the formation of $[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\cdot 2\,I_2$.

2·0.9 I_2: Desolvated **2** (45.9 mg) was immersed in a solution of I_2 (80 mg) in CCl_4 (20 mL) in a closed tube for one week and then filtered. The solid was washed thoroughly with carbon tetrachloride and diethyl ether. Yield: 49.2 mg. Thermogravimetric analysis of **2**·0.9 I_2 (Supporting Information) showed a first weight loss of 8.17 %, which corresponds to 0.9 I_2 per formula unit and is consistent with the formulation: $[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\cdot 0.9\,I_2$. Subsequent weight losses follow that found for the desolvated compound. Elemental analysis: calcd for $C_{60}H_{48}Cu_8I_{5.8}N_{12}S_8W_2$ (%) C 25.68, H 1.73, N 5.99, I 26.23; found: C 25.53, H 1.69, N 5.84, I 26.17.

2·4.2 aniline: Desolvated crystals (52 mg) of **2** were added to freshly distilled aniline (20 mL) in a 20-mL Erlenmeyer flask, which was capped. The mixture was stirred for 10 min and allowed to stand at 0 °C for approximately one week. The solid, $[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\cdot 4.2\,NH_2C_6H_5$ was washed with acetonitrile and diethyl ether. Yield: 60.4 mg. Elemental analysis: calcd for $[WS_4Cu_4(4,4'\text{-bpy})_4][WS_4Cu_4I_4(4,4'\text{-bpy})_2]\cdot 4.2$ aniline (**2**·4.2 aniline) (%): C 34.47, H 2.63, N 7.65, I 17.01, Cu 7.13, W 12.39; found: C 34.63, H 2.72, N 7.98, I 16.35, Cu 6.87, W 11.88.

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