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Syntheses and Crystal Structures of Four Supramolecular Halides/Pseudohalides: $[(ZnCl_4)(BPX)]$, $[(CdCl_4)(BPX)]$, $[(HgCl_4)(BPX)]$, and $[Cu_4(SCN)_6(BPX)]_n$ Directed by 1, 4-Bis(pyridinium)xylol Cations

Wen-Zhen Fu [a], Han-Qi Wang [a], Bei Wang [a], Hao-Jie Duan [a], Xin-Xin Cui [a], Wei-Juan Wang [a], Zhe-Guang Wang [b], Zhen-Hua Zhang [a], Xiu-Cun Liu [a] & Yun-Yin Niu [a]

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Syntheses and Crystal Structures of Four Supramolecular Halides/Pseudohalides: $[(ZnCl_4)(BPX)]$, $[(CdCl_4)(BPX)]$, $[(HgCl_4)(BPX)]$, and $[Cu_4(SCN)_6(BPX)]_n$ Directed by 1, 4-Bis(pyridinium)xylol Cations

Wen-Zhen Fu,[1] Han-Qi Wang,[1] Bei Wang,[1] Hao-Jie Duan,[1] Xin-Xin Cui,[1] Wei-Juan Wang,[1] Zhe-Guang Wang,[2] Zhen-Hua Zhang,[1] Xiu-Cun Liu,[1] and Yun-Yin Niu[1]

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Four supramolecular compounds, $[(ZnCl_4)(BPX)]$ 1, $[(CdCl_4)(BPX)]$ 2, $[(HgCl_4)(BPX)]$ 3 and $[Cu_4(SCN)_6(BPX)]_n$4 (BPX = 1, 4-bis(pyridinium)xylol), were synthesized and characterized by IR spectrum and X-ray crystallography. The crystal structures of 1-3 show that they all crystallize in monoclinic systems with mononuclear tetrachlorometallate structure. Compound 4 crystallizes in triclinic system, space group P-1 in which four SCN^-ions serve as bridging ligands to link four Cu(I) ions, giving rise to a 16-numbered ring. Many 16-membered rings are bridged by $SCN^-$to form a one-dimensional chain structure.Furthermore, Cu-Cu bonds bridge two chain structures to form a one-dimensional complex chain structure.

Keywords 1, 4-bis(pyridinium)xylol, crystal structure, coordination polymer

INTRODUCTION

Recently, more attention has been paid to design and synthesis of new supramolecular compounds because of their novel structures and potential applications in magnetism, electrical conductivity, luminescence, biology, catalysis, etc.[1−7] The supramolecular compounds with certain structures and functions can be obtained by self-assembly, that is forming spontaneously by synergistic actions of hydrogen bonding, van der Waals force, electrostatic interaction, C—H/π interaction, etc.[8−11]

With the rapid development of supramolecular compounds, a large number of coordination polymers have been synthesized with different crystal structures since discovered. And the supramolecular polymers directed by organic cations have become a new research focus owning to their unique features, such as good catalytic effect, powerful magnetic performance, good nonlinear optical property, and so on.[12−13] Organic cations connect with polyanions by weak interactions to form stacked structures of infinite space. Bridging N-donor ligands had generally been employed for the construction of M(IB, IIB)-X-containing polymers, and thiocyanate (SCN^-) coordinating to metal centers as an end-to-end bridging ligand between two metal centers, though both sulfur and nitrogen atoms were infrequently applied in the synthesis successfully.[14−18]

In this article, the authors select the azotic heterocyclic organic cation BPX to carry on a self-assembly and four novel M(IB, IIB) compounds, $[(ZnCl_4)(BPX)]$ **1**, $[(CdCl_4)(BPX)]$ **2**, $[(HgCl_4)(BPX)]$ **3** and $[Cu_4(SCN)_6(BPX)]_n$ **4**, were obtained and structurally characterized. We had previously reported several coordination compounds, $[Cu_2(SCN)_4(BPX)]_n$ **5**[19], $[(Bph_4)_2(BPX)]$[20] **6**, $[(HgCl_{2.75}I_{1.25})(BPX)]$[21]**7**, $[Fe(CN)_6(BPX).4H_2O]$[22] **8**, directed by the same organic cation BPX. Interestingly, the structural unit of the polymer **5** consisted of two Cu(I) centers, four SCN^-ions, and one BPX. It formed an infinite one-dimensional ladder-shaped structure. However, the structural unit of the title polymer $[Cu_4(SCN)_6(BPX)]_n$ **4** consists of four Cu(I) centers, six SCN^-ions, and one BPX. The structure units connect to each other by SCN^-ions to form an infinite one-dimensional chain structure. Moreover, two such one-dimensional chains connect to each other through Cu(I)—Cu(I) bonds to form an interesting one-dimensional complex chain structure. From the conclusion

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TABLE 1
Crystallographic data and structural refinement for compounds **1–4**

Compound	**1**	**2**	**3**	**4**
Empirical formula	C_{18} H_{18} Cl_4 N_2 Zn	C_{18} H_{18} Cd Cl_4 N_2	C_{18} H_{18} Cl_4 Hg N_2	C_{12} H_9 Cu_2 N_4 S_3
Formula weight	469.51	516.54	604.73	432.49
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	P2 (1)/c	P2 (1)/c	P2 (1)/c	P-1
Crystal size(mm)	0.45 × 0.36 × 0.32	0.26 × 0.13 × 0.09	0.19 × 0.13 × 0.10	0.25 × 0.15 × 0.10
a/°A	13.5795 (11)	13.8251 (17)	13.7838 (17)	7.3672 (10)
b/°A	9.1468 (7)	9.2389 (12)	9.2356 (11)	10.3530 (13)
c/°A	16.4189 (13)	16.395 (2)	16.324 (2)	10.8252 (14)
α/°	90	90	90	106.7770 (10)
β/°	98.6170 (10)	98.9000 (10)	98.840 (2)	103.080 (2)
γ/°	90	90	90	10.8252 (14)
V/ °A3	2016.4 (3)	2068.9 (4)	2053.4 (4)	768.12 (17)
Dc/g· Ecm−3	1.547	1.658	1.956	1.870
Z	4	2	4	2
μ	1.752 mm^{-1}	1.576 mm^{-1}	8.020 mm^{-1}	3.171 mm^{-1}
F (000)	952	1024	1152	430
R (int)	0.0192	0.0280	0.0424	0.0148
Theta range for data collection (·)	2.56 to 27.50	2.51 to 27.50	2.54 to 25.50	2.42 to 25.50
Data / restraints / parameters	4624 / 0 / 226	4730 / 0 / 226	3701 / 0 / 226	2851 / 0 / 190
Goodness-of-fit on Fˆ2	1.015	1.013	1.001	1.038
Reflections collected/unique	17317 / 4624	15653 / 4730	11749 / 3701	5902 / 2851
Max. and min. transmission	0.6058 and 0.5075	0.8712 and 0.6811	0.5101 and 0.3140	0.7360 and 0.5033
Final R indices[I>2sigma(I)]	R1 = 0.0269, wR2 = 0.0640	R1 = 0.0296, wR2 = 0.0608	R1 = 0.0334, wR2 = 0.0583	R1 = 0.0250, wR2 = 0.0537
R indices(all data)	R1 = 0.0365, wR2 = 0.0683	R1 = 0.0480, wR2 = 0.0678	R1 = 0.0627, wR2 = 0.0691	R1 = 0.0327, wR2 = 0.0570
Largest diff. peak and hole	0.302 and −0.224 e.Aˆ-3	0.235 and −0.390 e.Aˆ −3	0.519 and −0.669 e.Aˆ −3	0.326 and −0.333 e.Aˆ −3

that we had reported, the compounds were easy to generate 1D or 2D polymers when we used CuSCN.

EXPERIMENTAL

Material and Measurement

Reagents and solvents all were analytical pure grade, and $\alpha\ \alpha'$-Dichlor-p-xylol was purchased from Sigma-Aldrich, used without further purification. The organic cation BPX was prepared as the reported method.[19]The IR spectra were measured with a Bruker Tensor 27 spectrophotometer in the range 4000–400 cm^{-1}using KBr pellets.

Synthesis of [($ZnCl_4$)(BPX)] (1)

To a solution of $ZnCl_2$ (0.05 mmol, 6.8 mg) in DMF (5 ml) was added dropwise a solution of BPX·Cl_2(0.05 mmol, 8.8 mg) in CH_3OH (5 ml), and then added dropwise additional DMF until white precipitate completely disappeared. The reaction mixture was allowed to stand at room temperature for about a week in the dark. The white crystals suitable for X-ray single crystal diffraction analysis were collected, with a yield of 52%. IR (KBr, cm^{-1}): 3051 m, 1630 s, 1480 s, 1200 m, 1151 s, 783 m, 750 s, 676 s, 606 m, 486 m.

Synthesis of [($CdCl_4$)(BPX)] (2)

Compound (**2**) was obtained via a similar procedure as that of (**1**). The white crystals suitable for X-ray single crystal

TABLE 2
Selected bonds lengths (A°) and angles (·) for compounds **1–3**

Compound **1**		Compound **2**		Compound **3**	
Zn(1)-Cl(3)	2.2527(6)	Cd(1)-Cl(2)	2.4317(8)	Hg(1)-Cl(1)	2.4411(18)
Zn(1)-Cl(4)	2.2603(6)	Cd(1)-Cl(3)	2.4373(8)	Hg(1)-Cl(2)	2.4440(18)
Zn(1)-Cl(2)	2.2871(5)	Cd(1)-Cl(1)	2.4746(7)	Hg(1)-Cl(4)	2.5151(15)
Zn(1)-Cl(1)	2.2932(5)	Cd(1)-Cl(4)	2.4788(8)	Hg(1)-Cl(3)	2.5217(17)
Cl(3)-Zn(1)-Cl(4)	111.63(2)	N(1)-C(5)	1.321(3)	N(1)-C(5)	1.341(7)
Cl(3)-Zn(1)-Cl(2)	110.52(2)	N(1)-C(1)	1.330(3)	N(1)-C(1)	1.342(7)
Cl(4)-Zn(1)-Cl(2)	110.45(2)	N(1)-C(6)	1.497(3)	N(1)-C(6)	1.504(7)
Cl(3)-Zn(1)-Cl(1)	110.93(2)	N(2)-C(10)	1.339(3)	N(2)-C(14)	1.305(7)
Cl(4)-Zn(1)-Cl(1)	110.52(2)	N(2)-C(14)	1.343(3)	N(2)-C(10)	1.344(8)
Cl(2)-Zn(1)-Cl(1)	102.43(2)	N(2)-C(15)	1.500(3)	N(2)-C(15)	1.490(7)
		Cl(2)-Cd(1)-Cl(3)	111.65(3)	Cl(1)-Hg(1)-Cl(2)	113.97(6)
		Cl(2)-Cd(1)-Cl(1)	109.81(3)	Cl(1)-Hg(1)-Cl(4)	109.62(6)
		Cl(3)-Cd(1)-Cl(1)	111.18(3)	Cl(2)-Hg(1)-Cl(4)	111.38(7)
		Cl(2)-Cd(1)-Cl(4)	112.88(3)	Cl(1)-Hg(1)-Cl(3)	112.45(6)
		Cl(3)-Cd(1)-Cl(4)	111.46(3)	Cl(2)-Hg(1)-Cl(3)	111.16(6)
		Cl(1)-Cd(1)-Cl(4)	99.25(2)	Cl(4)-Hg(1)-Cl(3)	97.03(5)

diffraction analysis were collected, with a yield of 46%. IR (KBr, cm^{-1}): 3049 m, 1627 s, 1483 s, 1207 m, 1154 s, 780 m, 746 s, 676 s, 606 m, 486 m.

Synthesis of [($HgCl_4$)(BPX)] (3)

Compound (**3**) was obtained via a similar procedure as that of (**1**). The white crystals suitable for X-ray single crystal diffraction analysis were collected, with a yield of 63%. IR (KBr, cm^{-1}): 3047 m, 1627 s, 1483 s, 1205 m, 1153 s, 780 m, 746 s, 676 s, 607 m, 460 m.

Synthesis of $[Cu_4(SCN)_6(BPX)]_n$(4)

To a mixing solution of $CuCl_2$(0.05 mmol, 6.75 mg) and KSCN (0.01 mmol, 9.7 mg) in DMF/H_2O was added dropwise a solution of BPX·Cl_2(0.05 mmol, 8.8 mg) in CH_3OH (5 ml), and then added dropwise additional DMF to light yellow precipitate

TABLE 3
Selected bonds lengths (A°) and angles (·) for compound **4**

Cu(1)-N(2)#1	1.899(2)	Cu(1)-N(1)	1.903(2)
Cu(1)-S(3)#2	2.3206(8)	Cu(1)-Cu(1)#1	3.0575(8)
Cu(2)-N(3)	1.946(2)	Cu(2)-S(1)	2.2875(7)
Cu(2)-S(2)	2.4192(8)	Cu(2)-S(2)#2	2.4525(8)
Cu(2)-Cu(2)#2	2.8798(8)	S(2)-Cu(2)#2	2.4525(8)
S(3)-Cu(1)#2	2.3206(8)	N(2)-Cu(1)#1	1.898(2)
N(2)#1-Cu(1)-N(1)	131.61(9)	N(2)#1-Cu(1)-S(3)#2	111.75(7)
N(1)-Cu(1)-S(3)#2	116.37(7)	N(2)#1-Cu(1)-Cu(1)#1	91.83(7)
N(1)-Cu(1)-Cu(1)#1	87.09(7)	S(3)#2-Cu(1)-Cu(1)#1	97.36(3)
N(3)-Cu(2)-S(1)	120.61(7)	N(3)-Cu(2)-S(2)	101.97(7)
S(1)-Cu(2)-S(2)	114.66(3)	N(3)-Cu(2)-S(2)#2	106.10(7)
S(1)-Cu(2)-S(2)#2	105.19(3)	S(2)-Cu(2)-S(2)#2	107.53(2)
N(3)-Cu(2)-Cu(2)#2	114.23(7)	S(1)-Cu(2)-Cu(2)#2	125.00(2)
S(2)-Cu(2)-Cu(2)#2	54.30(2)	S(2)#2-Cu(2)-Cu(2)#2	53.23(2)
C(1)-S(1)-Cu(2)	102.58(9)	C(2)-S(2)-Cu(2)	103.90(9)
C(2)-S(2)-Cu(2)#2	100.91(8)	Cu(2)-S(2)-Cu(2)#2	72.47(2)
C(3)-S(3)-Cu(1)#2	99.15(9)	C(1)-N(1)-Cu(1)	165.7(2)
C(2)-N(2)-Cu(1)#1	170.9(2)	C(3)-N(3)-Cu(2)	162.1(2)
C(3)-N(3)-Cu(2)	162.1(2)	C(3)-N(3)-Cu(2)	162.1(2)
C(3)-N(3)-Cu(2)	162.1(2)		

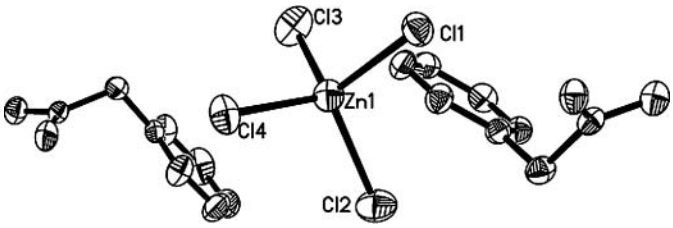

FIG. 1. The basic structural unit of compound **1**.

until completely disappeared. The reaction mixture was allowed to stand at room temperature for several days. The light yellow transparent crystals suitable for X-ray single crystal diffraction analysis were collected, with a yield of 43%. IR (KBr, cm^{-1}): 3053 m, 2091 s, 1626 m, 1484s, 1208 m, 1160 m, 797 m, 769 s, 739 m, 674 m, 600 m, 448 m.

X-ray Structural Determination

Crystallographic data for the title compounds **1–4** were collected at 291(2) K on a Bruker APEX-II area-detector diffractometer equipped with graphite-monochromatized Mo-Ka radiation ($\lambda = 0.71073A^{\circ}$). The structures were solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were assigned with common isotropic displacement factors and included in the final refinement by using geometrical constraints. The structures were refined with full-matrix least-squares techniques on F2 using the SHELXTL-97 program package. Crystal data are summarized in detail in Table 1. Selected bond lengths and angles of the title compounds **1–4** are respectively listed in Tables 2–3.

RESULTS AND DISCUSSION

Crystal Structure of [($ZnCl_4$)(BPX)] (1)

Single-crystal structure analysis revealed that compound **1** crystallized in monoclinic system with space group P2(1)/c. The crystal structural unit of compound **1** is shown in Figure 1.

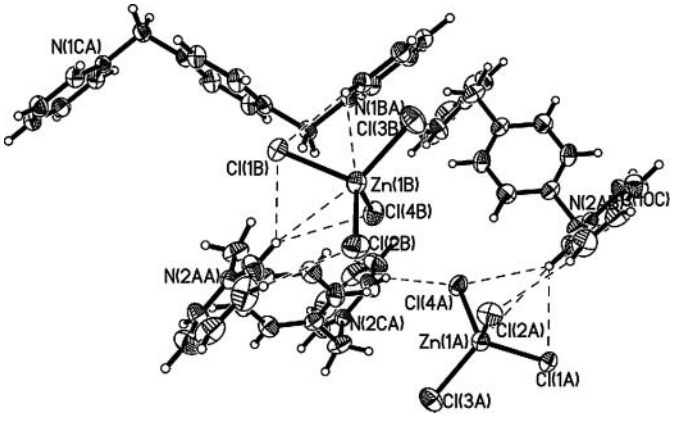

FIG. 2. Weak hydrogen bonds between Cl and BPX^{2+} in **1**.

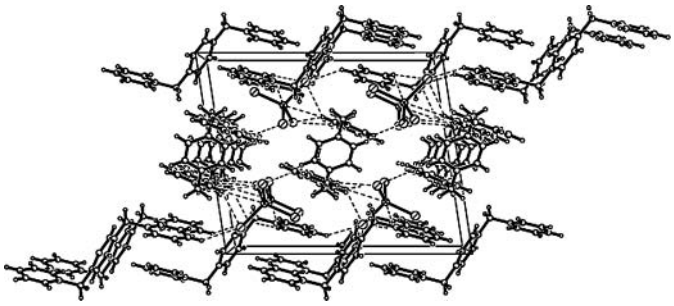

FIG. 3. 3D architecture of compound **1**.

The structural unit consists of one Zn(II), four Cl^- ions and one BPX. The Zn atom is four-coordinated with four Cl atoms to form deformed tetrahedron geometry, the bond lengths of Zn-Cl arrange from 2.2513(7) Å to 2.2903(7) Å, bond angles vary from 110.39(3)° to 111.45(3)°

Within each unit, Cl atom connects with BPX^{2+} through hydrogen bond (shown in Figure 2), and the units form a 3D architecture through this weak interactions (shown in Figure 3).

Crystal Structure of [($CdCl_4$)(BPX)] (2)

The structure of compound [($CdCl_4$)(BPX)] is similar with compound **1**.

The structural unit consists of one Cd(II), four Cl^- ions, and one BPX. The Cd atom is four-coordinated with four Cl atoms to form deformed tetrahedron geometry, the bond lengths arrange from 2.4317(8) Å to 2.4788(8) Å , the bond angles vary from 109.81(3)° to 112.88(3)°. Within each unit Cl atom connects with BPX^{2+} through hydrogen bond (shown in Figure 4), and the units form a huge 3D architecture through this weak interaction (shown in Figure 5).

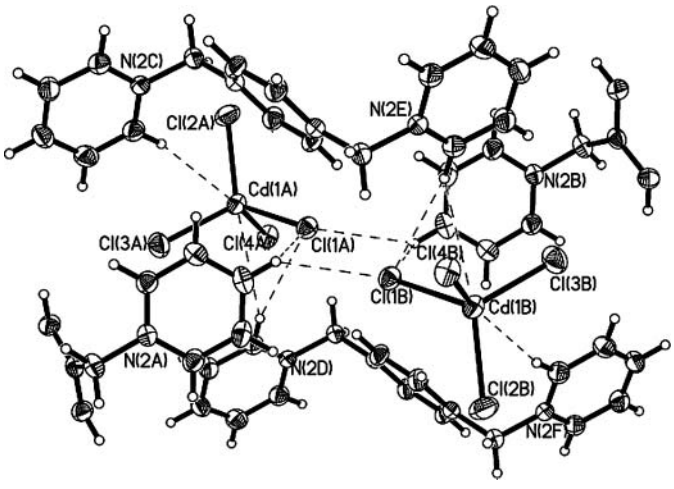

FIG. 4. Weak hydrogen bonds between Cl and BPX^{2+} in **2**.

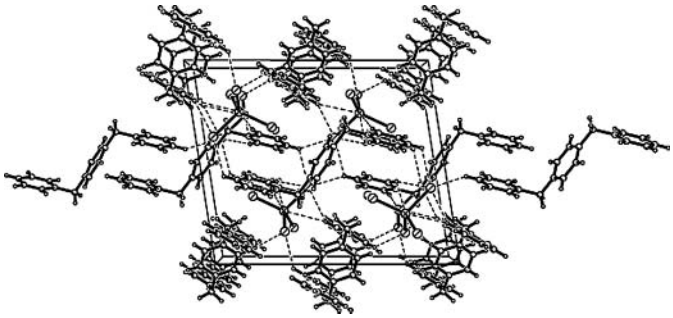

FIG. 5. Stereo architecture of compound **2**.

Crystal Structure of [($HgCl_4$)(BPX)] (3)

The structural unit consists of one Hg(II), four Cl^- ions, and one BPX (shown in Figure 6). The Hg atom is tetra-coordinated with four Cl atoms to form tetrahedron geometry, the bond lengths arrange from 2.4427(11) Å to 2.5226(10) Å, the bond angles vary from 97.10(3)° to 113.82(4)° . Within each unit Cl atom and Hg atom connect with BPX^{2+} through hydrogen bonds (shown in Figure 6), and the units form a 3D architecture through this weak fact (shown in Figure 7).

Crystal Structure of [$Cu_4(SCN)_6(BPX)]_n$(4)

Single-crystal X-ray diffraction analysis reveals that $[Cu_4(SCN)_6(BPX)]_n$**4** crystallizes in triclinic system with space group P-1. Selected bond lengths and angles of compound **4** are list in Table 3.

The structure unit consists of four Cu centers, six SCN^- ions, and one BPX. Four SCN^- ions serve as bridging ligands to link four Cu ions, giving rise to a sixteen-membered ring, among which Cu(1) and Cu(1A) form one Cu^I—Cu^I bond between them (shown in Figure 8).

There are two kinds of coordinate environments in the solid state. One Cu ion is tri-coordinated and each atom is surrounded by three NCS^- ions to form triangle planar geometry; and the other Cu ion is tetra-coordinated with four NCS^- ions to form

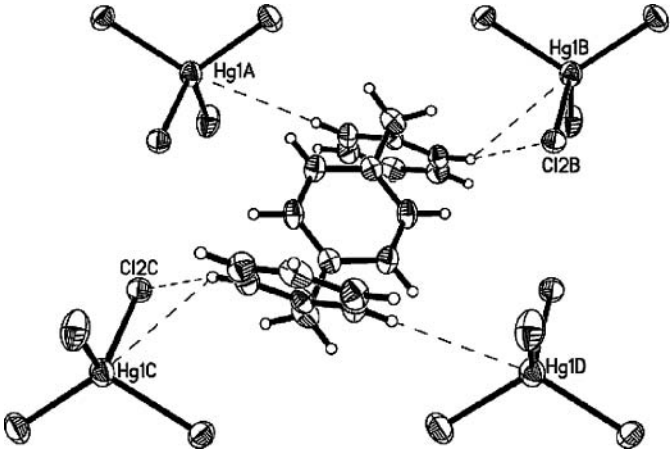

FIG. 6. Weak hydrogen bonds within Cl and BPX^{2+}, Hg and BPX^{2+} in **3**.

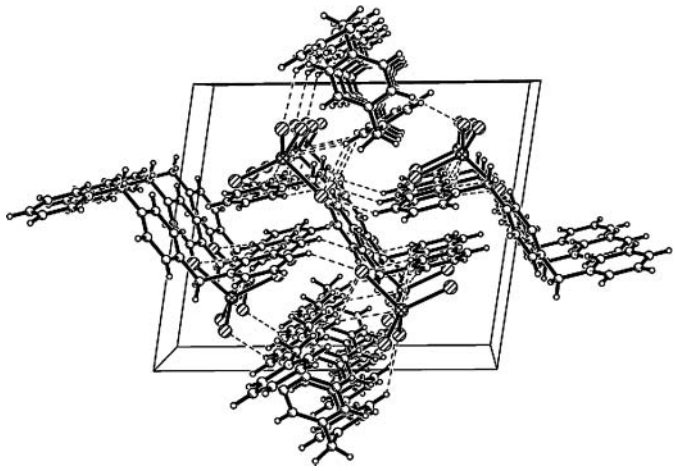

FIG. 7. 3D architecture of compound **3**.

tetrahedron geometry (shown in Figure 9). Furthermore, SCN^- ions serve as bridging ligands to link each sixteen-membered tetra center to form an infinite one-dimensional chain structure, two such one-dimensional chains combine together through Cu(I)-Cu(I) bonds to form a one-dimensional composite chain structure. Two kinds of Cu· · ·Cu bond lengths are 5.584Å and 5.358Å in each sixteen-membered ring. The Cu(1)-Cu(1)# and Cu(2) and Cu(2)# bond lengths are 3.0575(8) Å and 2.8798(8) Å, Cu(1)-N bond lengths arrange from 1.899(2) Å to 1.946(2) Å, Cu(1)-S bond lengths arrange from 2.2875(7) Å to 2.4525(8) Å. The N(2)#1-Cu(1)-N(1) bond angle is 131.61(9) °, N-Cu(1)-S bond angles vary form 111.75(7)° to 116.37(7)°, N-Cu(2)-S bond angles vary from 101.97(7)° to 106.10(7)°, S-Cu(1)-S bond angles vary from 105.19(3)° to 114.66(3)°.

The sulfur atom, nitrogen atom and copper atom of each structural unit form weak H· · ·S bond, H· · ·N bond with hydrogen atoms from BPX^{2+}cation as is shown in Figure 10. Thus, these crystal structure units are connected each other to form an infinite space packing structure, as is shown in Figure 11.

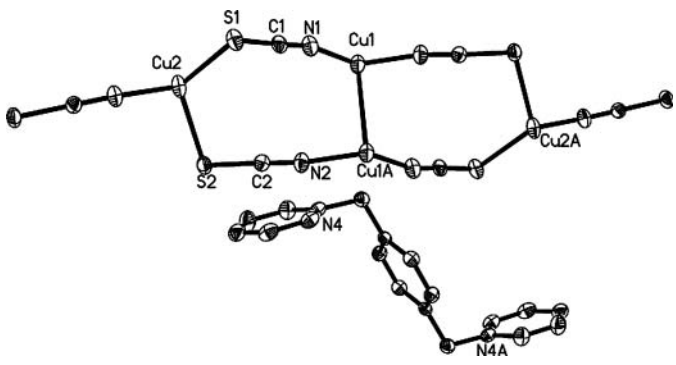

FIG. 8. The basic structural unit of compound **4**.

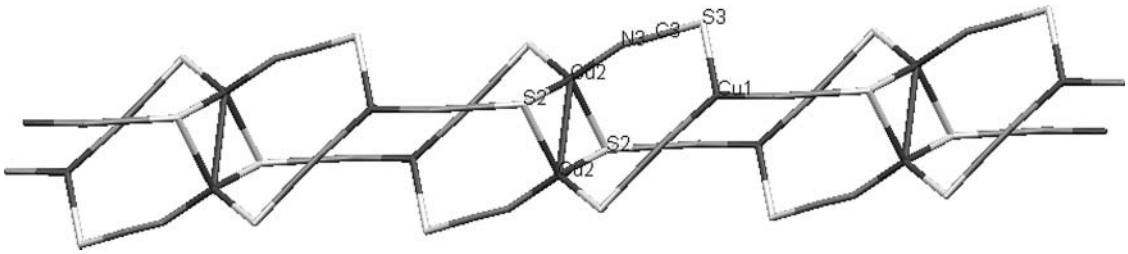

FIG. 9. The one-dimensional complex chain structure of compound **4**.

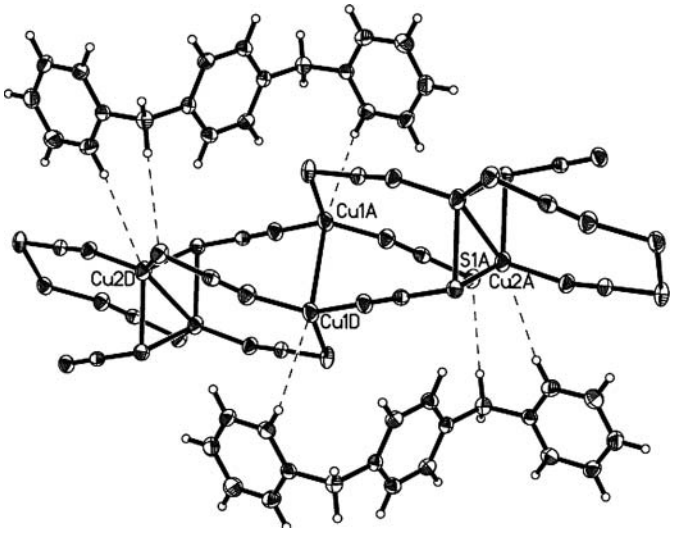

FIG. 10. Weak H· · ·S bond interactions of compound **4**.

CONCLUSION

In summary, we select the azotic heterocyclic organic cation BPX to carry on a self-assembly and four novel M(IB, IIB) compounds, $[(ZnCl_4)(BPX)]$**1**, $[(CdCl_4)(BPX)]$**2**, $[(HgCl_4)(BPX)]$**3** and $[Cu_4(SCN)_6(BPX)]_n$**4**, were obtained and structurally characterized.

More intriguing, the structural unit of the polymer $[Cu_4(SCN)_6(BPX)]_n$**4** consists of four Cu(I) centers, six SCN^- ions, and one BPX. The structure units connect to each other by SCN^- ions to form an infinite one-dimensional chain structure. Moreover, two such one-dimensional chains connect to each other through Cu(I)-Cu(I) bond to form a one-dimensional composite chain structure. By contrast, the conformations of the BPX cations in **1**-**8** differ slightly. Extensive hydrogen bonding is found in the crystal packing structure of all these compounds.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center,

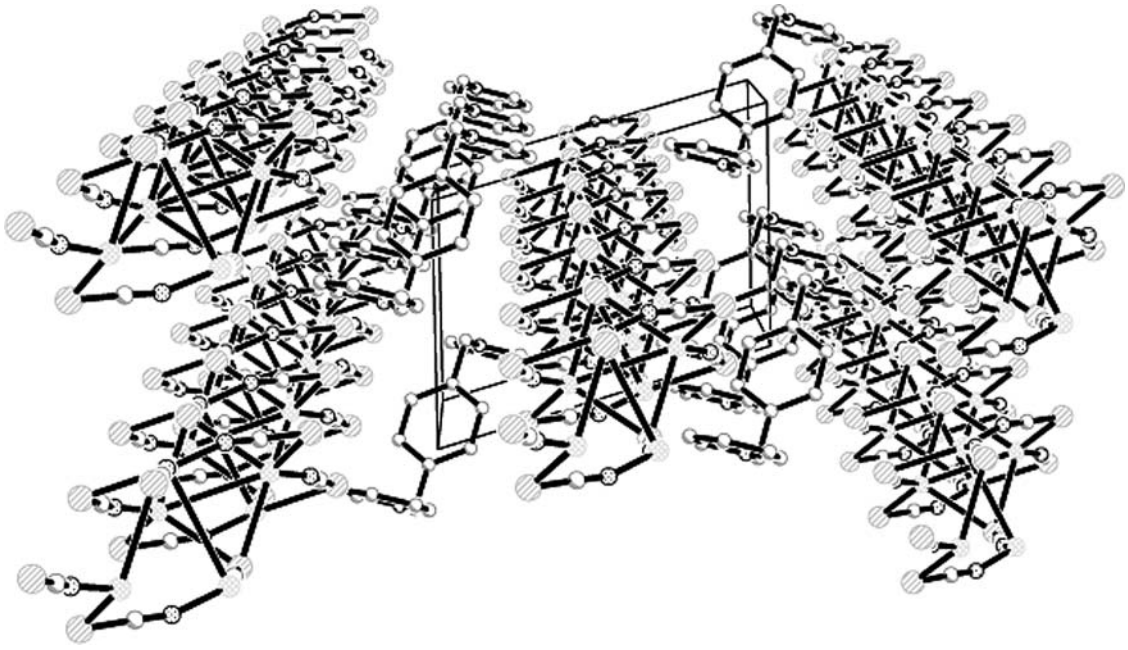

FIG. 11. Packing structure of compound **4**.

CCDC Nos. 623346, 623353, 623345, 623645 for compounds **1**-**4.** Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: 44-1223-336–033; email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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