

研究简报

二个新颖的一维聚合物 $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_\infty$ 和
 $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_\infty$ 的合成和晶体结构张灯青¹ 徐庆锋¹ 陈金香¹ 刘泉¹ 朱炎君¹ 郎建平^{*,1,2}⁽¹⁾ 苏州大学化学化工学院, 苏州 215006)⁽²⁾ 南京大学配位化学国家重点实验室, 南京 210093)关键词: 一维聚合物 铜(I)化合物 晶体结构
分类号: O613.43 O614.121Syntheses and Crystal Structures of Two Novel Bromocuprate (I)
Polymers $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_\infty$ and $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_\infty$ ZHANG Deng-Qing¹ XU Qing-Feng¹ CHEN Jin-Xiang¹ LIU Quan¹ ZHU Yan-Jun¹ LANG Jian-Ping^{*,1,2}⁽¹⁾ School of Chemistry and Chemical Engineering, Suzhou University, Suzhou 215006)⁽²⁾ State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093)

Reactions of CuBr with equimolar PyHBr or $\gamma\text{-MePyHBr}$ afforded two novel one-dimensional bromocuprate (I) polymers $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_\infty$ (**1**) and $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_\infty$ (**2**), respectively. Both **1** and **2** were characterized by IR, elemental analysis, and X-ray crystallography. Crystal data for **1**: monoclinic, space group $P2_1/n$, $a = 0.93262(12)$ nm, $b = 1.3497(2)$ nm, $c = 1.9149(2)$ nm, $\beta = 93.465(7)^\circ$, $V = 2.4060(5)$ nm³, $Z = 4$. Crystal data for **2**: orthorhombic, space group $Pbcm$, $a = 0.8659(4)$ nm, $b = 1.5669(6)$ nm, $c = 1.2849(5)$ nm, $V = 1.7432(2)$ nm³, $Z = 4$. The structure of **1** has a unique helical chain composed of $[\text{Cu}_3\text{Br}_5]$ units and $\mu\text{-bromide}$ anions while that of **2** shows a linear chain consisted of $[\text{Cu}_2(\mu\text{-Br})_2]$ units bridged by pairs of $\mu\text{-Br}$ atoms. CCDC: **1**, 208412; **2**, 208413.

Keywords: one-dimensional polymer copper (I) complex crystal structure

0 Introduction

In the last decades, low-dimensional copper (I) -organic hybrid complexes have attracted much attention due to their own colorful chemistry^[1-9] and their potential applications in fluorescent sensors^[10], olefin

separations^[11], and enantioseparation^[12]. Intriguingly, the structures of some polycopper (I) anionic complexes could be influenced by the size, shape, and charge distribution of the associated organic countercations such as quaternary ammonium and phosphonium cations^[13, 14]. The bromocuprate (I) complexes demon-

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strate lots of examples in which their structures are dependent on the counteranions used in the reactions. For example, reactions of CuBr with different quaternary ammonium and phosphonium bromide produced a series of bromocuprate (I) complexes with different copper nuclearities: $\text{R}[\text{CuBr}_2]$ ($\text{R} = \text{PEt}_4$)^[15], $\text{R}_2[\text{Cu}_2\text{Br}_4]$ ($\text{R} = \text{NEt}_4$)^[16], $\text{R}[\text{Cu}_3\text{Br}_6]$ ($\text{R} = \text{C}_5\text{H}_7\text{N}_2\text{O}$)^[17], $\text{R}_2[\text{Cu}_4\text{Br}_8]$ ($\text{R} = \text{NPr}_4$)^[18], $\text{R}_2[\text{Cu}_5\text{Br}_7]$ ($\text{R} = \text{PyH}$)^[19] and $\text{R}_3[\text{Cu}_6\text{Br}_9]$ ($\text{R} = \text{Et}_3\text{NMe}$)^[14], $\text{R}[\text{Cu}_7\text{Br}_8]$ ($\text{R} = \text{C}_6\text{H}_{12}\text{NH}_3$)^[20], and $\text{R}_6[\text{Cu}_8\text{Br}_{15}]$ ($\text{R} = \text{Ph}_3\text{PMe}$)^[21]. However, very few examples have been reported to explore the effect of the organic cations in the assembly of bromocuprate (I) polymeric complexes. In this context, we report the synthesis and structural characterization of two one-dimensional polymers $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_n$ (**1**) and $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_n$ (**2**).

1 Experimental

1.1 General Procedure

All manipulations were carried out under dinitrogen using standard Schlenk-line techniques. CuBr, pyridine, hydrobromide acid and solvents were of analytical grade, and were used without further purification. IR spectra (KBr disc) were recorded on a Nicolet MagNa-IR 550 spectrophotometer. Elemental analyses were performed on an EA1110-CHNS elemental analyzer.

1.2 Synthesis of $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_n$ (**1**)

To a solution containing PyHBr (1mmol) (prepared in situ from Py and HBr in 15mL of CH_3CN in 1:1 molar ratio) was added CuBr (0.143g, 1mmol). The resulting mixture was allowed to reflux at 85°C for 1h and then filtered. The yellow filtrate was slowly cooled down to room temperature, forming yellow plates of **1**. Yield: 0.27g(89%). Anal. Calcd. for $\text{C}_{15}\text{H}_{18}\text{Br}_6\text{Cu}_3\text{N}_3$ (%): C, 19.79; H, 2.00; N 4.62. Found(%): C, 19.74; H, 1.98; N, 4.70%.

1.3 Synthesis of $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_n$ (**2**)

To a flask containing a solution of $\gamma\text{-MePyHBr}$ (1mmol) (prepared in situ from $\gamma\text{-MePy}$ and HBr in 15mL of CH_3CN in 1:1 molar ratio) was added CuBr (0.143g, 1mmol). Work-up similar to that in the isolation of **1** generated yellow prisms of **2**. Yield: 0.21g (66%). Anal. Calcd. for $\text{C}_{12}\text{H}_{16}\text{Br}_4\text{Cu}_2\text{N}_2$ (%):

C, 22.70; H, 2.54; N, 4.41. Found(%): C, 22.63; H, 2.50; N, 4.58%.

1.4 X-Ray Crystallography

All measurements were made on a Rigaku Mercury CCD X-ray diffractometer (3kV, sealed tube) at 193K by using graphite monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.071070\text{nm}$). A yellow plate crystal of **1** with dimensions $0.25 \times 0.20 \times 0.05\text{mm}^3$ and a yellow prism of **2** with dimensions $0.45 \times 0.38 \times 0.30\text{mm}^3$ were selected for X-ray analysis. Diffraction data were collected at ω mode with a detector distance of 55mm to the crystal. Indexing was performed from 6 images each of which was exposed for 15s. A total of 1080 (**1**) or 720 (**2**) oscillation images were collected in the range $1.99^\circ < 2\theta < 54.96^\circ$ for **1** and $1.95^\circ < 2\theta < 54.96^\circ$ for **2**. The reflection data were reduced by using the program CrystalClear (Rigaku and MSC, Ver. 1.3, 2001), and an empirical absorption correction was applied which resulted in transmission factors ranging from 0.061 to 0.532 for **1** and from 0.007 to 0.031 for **2**. The reflection data were also corrected for Lorentz and polarization effects.

The structures of **1** and **2** were solved by direct methods^[22] and refined by full matrix least-squares on F^2 ^[23]. Anisotropic thermal parameters were used on all non-hydrogen atoms. All hydrogen atoms were introduced at the calculated positions and included in the structure-factor calculations. All calculations were performed on a Dell workstation using the Crystal-Structure crystallographic software package (Rigaku and MSC, Ver. 3.16, 2003). A summary of the key crystallographic information for **1** and **2** is given in Table 1.

CCDC: **1**, 208412; **2**, 208413.

2 Results and Discussion

2.1 Synthesis

The syntheses of **1** and **2** are rather straightforward, carried out by refluxing a mixture containing CuBr and PyHBr or $\gamma\text{-MePyHBr}$ (molar ratio = 1:1) in CH_3CN . After filtration, the filtrate was cooled to produce **1** in 89% yield or **2** in 70% yield. Solids **1** and **2** are moderately stable in air and moisture. However, the yellow solution of either **1** or **2** in CH_3CN turned greenish blue within minutes when exposed to

Table 1 Summary of Crystal Data for $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_n$ (1) and $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_n$ (2)

formula	$\text{C}_{15}\text{H}_{18}\text{Br}_6\text{Cu}_3\text{N}_3$	$\text{C}_{12}\text{H}_{16}\text{Br}_4\text{Cu}_2\text{N}_2$
<i>M</i> _r	910.37	634.98
cryst. system	monoclinic	orthorhombic
space group	$P2_1/n$	<i>Pbcm</i>
<i>a</i> /nm	0.93262(12)	0.8659(4)
<i>b</i> /nm	1.3497(2)	1.5669(6)
<i>c</i> /nm	1.9149(2)	1.2849(5)
β /°	93.465(7)	
<i>V</i> /nm ³	2.4060(5)	1.7432(12)
<i>Z</i>	4	4
<i>D</i> _c /(Mg · m ⁻³)	2.513	2.419
μ /mm ⁻¹	12.60	11.63
<i>F</i> (000)	1704	1200
reflections collected	23826 (<i>R</i> _{int} = 0.076)	13854 (<i>R</i> _{int} = 0.042)
independent reflections	5506	2051
reflections (<i>I</i> > 3.00σ(<i>I</i>))	1844	1007
parameters	262	120
<i>R</i>	0.027	0.031
<i>wR</i>	0.028	0.043
GOF	0.828	1.086
largest residual peaks and hole/(e · nm ⁻³)	760 and -560	830 and -800

open air. The identities of **1** and **2** were finally confirmed by single-crystal X-ray crystallography.

2.2 Structure Description

Compound **1** crystallizes in the monoclinic, space group $P2_1/n$, and the asymmetric unit contains one $[\text{Cu}_3\text{Br}_6]^{3-}$ trianion and three $[\text{PyH}]^+$ cations. As shown in Fig. 1, the Br(3) atom links two $[\text{Cu}_3\text{Br}_5]$ fragments through interactions with Cu(2) of one fragment and Cu(3') of another, forming an intriguing 1D helical array along the *b* axis. Although bromide-bridged copper (I) polymers are ubiquitous, those having helical chain structures are uncommon^[24]. Table 2 lists the selected bond lengths and angles of **1**. The Cu-Br(3)-Cu portion is slightly bent with Cu(2)-Br(3)-Cu(3') angle being 171.86(10)°. This

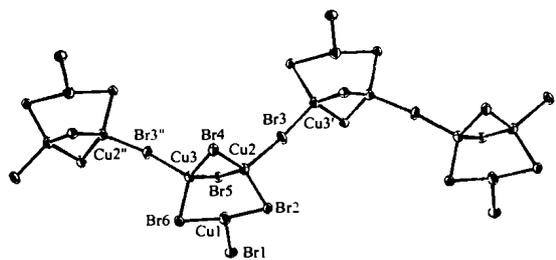


Fig. 1 Perspective view of a part of the $\{[\text{Cu}_3\text{Br}_6]^{3-}\}_n$ polymeric chain in **1** (looking down the *b* axis). The thermal ellipsoids are drawn at the 50% probability level and the counter-cations are omitted for clarity.

$[\text{Cu}_3\text{Br}_5]$ fragment is composed of a Cu_3 triangle with $\text{Cu}(2) \cdots \text{Cu}(1) \cdots \text{Cu}(3) = 58.89(6)^\circ$, $\text{Cu}(1) \cdots \text{Cu}(2) \cdots \text{Cu}(3) = 61.10(8)^\circ$, $\text{Cu}(1) \cdots \text{Cu}(3) \cdots \text{Cu}(2) = 60.01(8)^\circ$, respectively. The three $\text{Cu} \cdots \text{Cu}$ contacts are different with $\text{Cu}(1) \cdots \text{Cu}(2) = 0.2778(3) \text{ nm}$, $\text{Cu}(2) \cdots \text{Cu}(3) = 0.2747(2) \text{ nm}$, and $\text{Cu}(1) \cdots \text{Cu}(3) = 0.2808(3) \text{ nm}$, respectively. The average $\text{Cu} \cdots \text{Cu}$ separation of 0.2778 nm is close to that observed in $[\text{Cu}_4\text{Br}_6]^{2-}$ ($\text{Cu} \cdots \text{Cu} = 0.2734 \text{ nm}$)^[25], but is in-between those found in $[\text{Cu}_6\text{Br}_9]^{3-}$ ($\text{Cu} \cdots \text{Cu} = 0.2650 \text{ nm}$)^[20] and $[\text{Cu}_4\text{Br}_8]^{4-}$ ($\text{Cu} \cdots \text{Cu} = 0.3109 \text{ nm}$)^[26].

Interestingly, the Br(2) and Br(6) atoms bridge the $\text{Cu}(1) \cdots \text{Cu}(2)$ and $\text{Cu}(1) \cdots \text{Cu}(3)$ sides, respectively, while the $\text{Cu}(2) \cdots \text{Cu}(3)$ side is bridged by the Br(4) and Br(5) atoms. Therefore the coordination geometry of Cu(1) is different from Cu(2) and Cu(3), which is coordinated by a terminal Br, and two bridging Br ligands, showing a slightly pyramidalized Y-shape coordination geometry. Such a coordination is uncommon in the copper (I) complexes^[27]. The Cu(1)-Br bond distance ranges from 0.2409(2) nm to 0.2450(2) nm. In addition, we noticed a weak interaction between Cu(1) and Br(5) ($\text{Br}(5) \cdots \text{Cu}(1) = 0.2834(2) \text{ nm}$). On the other hand, the Cu(2) and Cu(3) atoms show slightly distorted tetrahedral geometry with the Br-Cu(2)-Br bond angles of $103.37(8)^\circ \sim 113.40(11)^\circ$ and the Br-Cu(3)-Br bond angles of 104.62

Table 2 Selected Bond Lengths(nm) and Angles($^\circ$) for $\{(\text{PyH})_3[\text{Cu}_3\text{Br}_6]\}_n$ (1)

Br1-Cu1	0.2409(2)	Br2-Cu1	0.2448(2)	Br2-Cu2	0.2467(3)
Br3-Cu2	0.2512(3)	Br4-Cu2	0.2480(3)	Br4-Cu3	0.2491(3)
Br5...Cu1	0.2834(2)	Br5-Cu2	0.2539(3)	Br5-Cu3	0.2568(3)
Br6-Cu1	0.2450(2)	Br6-Cu3	0.2438(3)	Cu1...Cu2	0.2778(3)
Cu1...Cu3	0.2808(3)	Cu2...Cu3	0.2747(2)		
Cu1-Br2-Cu2	68.85(7)	Cu2-Br4-Cu3	67.08(7)	Cu2-Br5-Cu3	65.06(7)
Cu1-Br6-Cu3	70.15(7)	Br1-Cu1-Br2	110.81(9)	Br1-Cu1-Br6	108.85(9)
Br2-Cu1-Br6	128.98(8)	Br2-Cu2-Br3	103.37(8)	Br2-Cu2-Br4	113.40(11)
Br3-Cu2-Br4	109.83(12)	Br2-Cu2-Br5	111.24(11)	Br3-Cu2-Br5	109.67(11)
Br4-Cu2-Br5	109.17(8)	Br4-Cu3-Br5	107.89(8)	Br4-Cu3-Br6	116.53(11)
Br5-Cu3-Br6	108.82(11)	Br4-Cu3-Br3''	104.62(11)	Br5-Cu3-Br3''	107.54(11)
Br6-Cu3-Br3''	111.03(8)	Cu2-Br3-Cu3'	171.86(10)	Cu(2)...Cu(1)...Cu(3)	58.89(6)
Cu(1)...Cu(2)...Cu(3)	61.10(8)	Cu(1)...Cu(3)...Cu(2)	60.01(8)		

Table 3 Selected Bond Lengths(nm) and Angles($^\circ$) for $\{(\gamma\text{-MePyH})_2[\text{Cu}_2\text{Br}_4]\}_n$ (2)

Br1-Cu1	0.2480(2)	Br2-Cu1	0.2504(2)	Br3-Cu1	0.2491(2)
Br3'-Cu1	0.2525(2)	Br2-Cu1'	0.2504(2)	Br1-Cu1'	0.2479(2)
Cu1...Cu1'	0.3207(2)	Cu1...Cu1''	0.3220(2)		
Br1-Cu1-Br2	99.70(5)	Br1-Cu1-Br3	120.79(8)	Br2-Cu1-Br3	111.61(7)
Br1-Cu1-Br3'	111.06(7)	Br2-Cu1-Br3'	114.30(9)	Br3-Cu1-Br3'	100.14(5)
Cu1-Br1-Cu1'	80.63(7)	Cu1-Br2-Cu1'	79.65(7)	Cu1-Br3-Cu1''	79.86(4)
Cu1'...Cu1...Cu1''	177.64(9)				

(11) $^\circ$ ~ 116.53(11) $^\circ$. The mean Cu-Br bond length for Cu(2) and Cu(3) is 0.2499nm, which is not unusual as compared with the structures containing tetrahedrally coordinated Cu (I) such as $[\text{Cu}_4\text{Br}_4(\text{TTT})_2]_n$ (TTT = triallyl-1, 3, 5-triazine-2, 4, 6(1H, 2H, 5H)-trione; Cu-Br = 0.2422 ~ 0.2495nm)^[8] and $[\text{NEt}_4]_2[\text{Cu}_2\text{Br}_4]$ (Cu-Br = 0.2319 ~ 0.2454nm)^[16].

Compound **2** crystallizes in the orthorhombic, space group *Pbcm*, and the asymmetric unit contains one-half of the $[\text{Cu}_2\text{Br}_4]^{2-}$ dianion and two one-half of the $[\gamma\text{-MePyH}]^+$ cations. Table 3 lists the selected bond lengths and angles of **2**. The linear chain structure can be described as extended networks of $[\text{Cu}(\mu\text{-Br})_2\text{Cu}]$ rhombs, which are alternatively parallel and perpendicular with one another along the *c* axis (Fig. 2). Similar structures are observed in $\{[\text{Cu}(\text{NH}_3)_4]_2[\text{Cu}_2\text{Br}_4]\}^{128}$ and $\{(\text{C}_5\text{H}_8\text{N}_3)\text{CuBr}_2\}$ ($\text{C}_5\text{H}_8\text{N}_3 = 2, 6\text{-diaminopyridinium}$)^[29]. In the structure of **2**, each copper atom has a slightly distorted CuBr_4 tetrahedral geometry with Br-Cu(1)-Br bond angles ranging from 99.70(5) $^\circ$ to 120.79(8) $^\circ$. The average Cu...Cu of 0.3214nm is much longer than that of **1**, while the mean Cu(1)- $\mu\text{-Br}$ bond length of 0.2500nm is normal relative to that of **1**.

Fig. 3 and 4 show unit cell packing diagrams of **1**

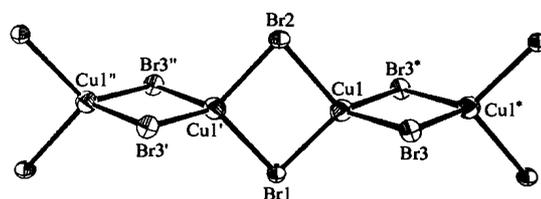


Fig. 2 Perspective view of a part of the $[\text{Cu}_2\text{Br}_4]^{2-}$ polymeric chain in **2** (looking down the *b* axis). The thermal ellipsoids are drawn at the 50% probability level and the counter-cations are omitted for clarity.

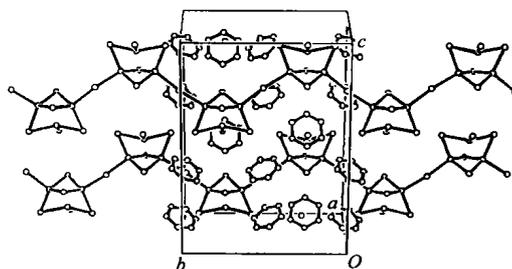


Fig. 3 Cell packing diagram of **1** looking down the *b* axis. Hydrogen atoms are omitted for clarity.

and **2**, respectively. The 1D polymeric array and the packing style of **1** are remarkably different from those of **2**. The reason may be attributed to the larger size of the

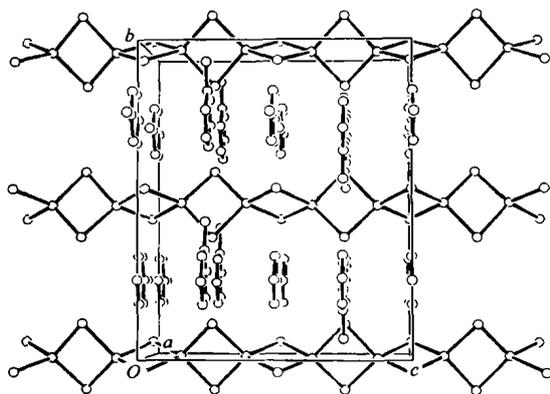


Fig. 4 Cell packing diagram of **2** looking down the *c* axis
Hydrogen atoms are omitted for clarity

$[\gamma\text{-MePyH}]^+$ cations of **2**. In both cases, there are no other interactions but electrostatic forces between the anionic polymeric chains and the associated organic cations ($[\text{PyH}]^+$ or $[\gamma\text{-MePyH}]^+$).

3 Conclusion

Two one-dimensional bromocuprate (I) polymers **1** and **2** were obtained by the facile reactions of CuBr with PyHBr or $\gamma\text{-MePyHBr}$. Both compounds were characterized by X-ray analysis. Compound **1** exhibits an bromide-bridged helical chain structure while **2** has a normal linear chain structure. The remarkable difference in the anionic structures of **1** and **2** may be due to the different sizes of the $[\text{PyH}]^+$ and $[\gamma\text{-MePyH}]^+$ cations. The successful syntheses of **1** and **2** demonstrate that some supramolecular arrays could be regulated by changing the organic counteranions.

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