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Hydrothermal synthesis and structure of a novel 3D framework based on ξ-octamolybdate chains: $[Cu_2(quinoxaline)_2Mo_4O_{13}]_n$

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Abstract

An unprecedented three-dimensional (3D) polymer $[Cu_2(quinoxaline)_2Mo_4O_{13}]_n$, formed from ξ-octamolybdate chains as building units and pairs of 1D $[Cu(quinoxaline)]_n^{n+}$ polymeric chains as linkers, provides the first example of an extended higher dimensional structure based on $[Mo_8O_{26}]_n^{4n-}$ octamolybdate chain. The basic building block of the octamolybdate chain included in the title compound is first reported to be constructed from ξ-isomer of octamolybdate unit.
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1. Introduction

The solid materials of one-dimensional structures with only molybdenum oxide frameworks are now receiving increasing attention not only because of their intriguing architectures but also because of their great potential applications in many fields such as catalysis, sorption, electric conductivity, magnetism and optical materials [1]. Polymeric structures based on infinite chains of oxomolybdenum polyhedra have been often reported as dimolybdate, trimolybdate and tetramolybdate, of which some may be viewed as octamolybdate systems [2–4], although pentamolybdate and heptamolybdate systems have been occasionally reported [5]. Examination of crystal structures of these $Mo_xO_y^{n-}$ chains reveals that although these structures share a common one-dimensional (1D) framework consisting of molybdenum oxide polyhedra, the details of the polyhedral connectivity are different. For example, different connection patterns of oxomolybdenum polyhdedra, adjusted by different combinations of corner-, edge- and face-sharing, lead to three different isomeric forms of $[Mo_3O_{10}]_n^{2n-}$ 1D chain (see Supporting Information, S1), as well as three distinct octamolybdate chains (see Supporting Information, S2). In $[H_2enMe]_2[Mo_8O_{26}]$ [4f], the octamolybdate subunits are connected by sharing double common corners, while in $(NH_4)_6Mo_8O_{27} \cdot 4H_2O$ [6] and $(C_4H_{12}N_2)_3Mo_8O_{27}$ [7], the octamolybdate units share single common corner. The linkage of octamolybdate subunits through common edges, as observed in $(C_5N_2H_7)_4[Mo_8O_{26}]$ [8], again results in an octamolybdate chain.

One-dimensional molybdenum oxide chain could be easily modified because of its high reactivity, and might further be bridged into higher dimensional solids by incorporation of appropriate metal–ligand subunits, since it's a recurring theme in oxide structural chemistry [9]. However, to the best of our knowledge, molybdenum oxide chains that have been reported to be extended to 2D or 3D frameworks are limited to $[Mo_3O_{10}]_n^{2n-}$ [10], except the molybdenum oxide chain in the 3D framework of $[Cu_2(triazolate)_2(H_2O)_2]_n[Mo_4O_{13}]_n$ [11] is of tetramolybdate.

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Although octamolybdate chains have been twice reported to be decorated by coordinated Cu(II) complex fragments [12], $[Mo_8O_{26}]_n^{4n-}$ chain based compound with 2D or 3D framework has never been reported. On the other hand, there are nine isomers of $[Mo_8O_{26}]^{4-}$ cluster, whereas the basic building blocks of the reported $[Mo_8O_{26}]_n^{4n-}$ chain are confined to γ-isomer [12]. Whether the $[Mo_8O_{26}]_n^{4n-}$ infinite chain can be constructed from other isomeric forms of octamolybdate than γ remains a puzzle. Here we report a novel 3D metal oxide framework of $[Cu_2(quinoxaline)_2 Mo_4O_{13}]_{2n}$ (**1**), which features in consisting of 1D ξ-octamolybdate chains linked by $[Cu(quinoxaline)]_n$ chains. Successful isolation of **1** evidences the assumption that other isomeric forms of octamolybdate than γ-isomer can also be functioned as the basic building block of the octamolybdenum oxide chain.

2. Experimental

2.1. General procedures

All reagents for the synthesis were purchased from commercial and used as received. Elemental analyses of C, H and N were performed with an EA1110 CHNS-0 CE elemental analyzer. IR (KBr pellet) spectrum was recorded on a Nicolet Magna 750FT-IR spectrometer. The TG analysis was carried out on a STA449C integration thermal analyzer in flowing air with a heating rate of $15\,°C\,min^{-1}$.

2.2. Synthesis of [Cu₂(quinoxaline)₂Mo₄O₁₃] (1)

A mixture of MoO_3 (0.053 g, 0.37 mmol), $CuSO_4 \cdot 5H_2O$ (0.097 g, 0.39 mmol), quinoxaline (0.048 g, 0.37 mmol) and H_2O (10 mL) was placed in a 18 mL Teflon-lined Parr acid digestion bomb and heated for 3 days at $180\,°C$ under autogenous pressure. Slow cooling of the reaction mixture to room temperature gave dark-red needle-like crystals (yield: 48% based on Mo). Anal. Calc. for $C_{16}H_{12}Cu_2Mo_4$ N_4O_{13}: C 19.62, H 1.24, N 5.72%. Found: C 19.78, H 1.96, N 5.66%. IR (solid KBr pellet, v/cm): 3085(m), 1503 (s), 1358 (m), 1204 (m), 1145 (m), 1056 (m), 945 (s), 902 (s), 843 (s), 763 (s), 713 (s), 597 (s).

2.3. X-ray crystallography

Crystal with dimension of $0.50 \times 0.10 \times 0.05\,mm$ of the title compound was mounted on a glass fiber. X-ray diffraction experiment was performed on a Rigaku Mercury CCD diffractometer with graphite-monochromatic MoKα radiation ($\lambda = 0.71073\,Å$) under liquid dinitrogen atmosphere at $130.15\,K$ by using ω scan. A total of 8458 reflections (4919 unique, Rint = 0.0185) were collected in the range of $3.13 \leqslant \theta \leqslant 27.48$ ($-10 \leqslant h \leqslant 10$, $-14 \leqslant k \leqslant 15$, $-16 \leqslant l \leqslant 12$). The structure was solved by direct methods and refined by full-matrix least-squares calculations with SHELXL-97. All non-hydrogen atoms were located with successive Fourier difference syntheses

Table 1
Crystallographic parameters of compound **1**

Formula	$C_{16}H_{12}Cu_2Mo_4N_4O_{13}$
Formula weight	979.14
Crystal size	$0.50 \times 0.10 \times 0.05$
Crystal color	Dark red
Crystal system	$P\,-1$
Space group	Triclinic
a (Å)	8.2636(8)
b (Å)	11.9370(17)
c (Å)	12.5848(15)
α (°)	114.788(4)
β (°)	95.567(2)
γ (°)	100.159(4)
V (Å^3)	1088.9(2)
Z	2
D_{calc} (Mg m^{-3})	2.986
μ (mm^{-1})	4.234
$F(000)$	932
Goodness-of-fit on F^2	1.053
Max. shift (Δ/σ) in final cycle	0.001
Largest diff. peak and hole(e Å^{-3})	0.747, −0.917
Data/restraints/parameters	4919/0/352
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0257$, w$R_2 = 0.0596$
R indices (all data)	$R_1 = 0.0336$, w$R_2 = 0.0636$

$R_1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$, w$R_2 = \Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

Table 2
Selected bond distances (Å) and angles (°) for **1**

Mo(1)–O(3)	1.726(2)	Mo(3)–O(10)	1.688(3)
Mo(1)–O(1)	1.744(2)	Mo(3)–O(7)	1.728(2)
Mo(1)–O(11)	1.829(2)	Mo(3)–O(8)	1.901(2)
Mo(1)–O(4)	2.098(2)	Mo(3)–O(4)	1.959(2)
Mo(1)–O(2)	2.106(2)	Mo(3)–O(9)	2.186(2)
Mo(1)–O(5)#1	2.247(2)	Mo(4)–O(12)	1.693(2)
Mo(2)–O(6)	1.712(2)	Mo(4)–O(13)	1.715(2)
Mo(2)–O(5)	1.747(2)	Mo(4)–O(8)	1.930(2)
Mo(2)–O(9)	1.891(2)	Mo(4)–O(11)#2	1.980(2)
Mo(2)–O(2)	1.947(2)	Mo(4)–O(9)	2.258(2)
Mo(2)–O(4)	2.124(2)	Mo(4)–O(1)#1	2.263(2)
Mo(2)–O(2)#1	2.421(2)	Cu(1)–O(13)#4	2.419(2)
Cu(1)–N(4)#3	2.005(3)	Cu(2)–N(3)	1.974(3)
Cu(1)–N(1)	2.010(3)	Cu(2)–N(2)#5	1.999(3)
Cu(1)–O(7)	2.111(3)	Cu(2)–O(3)	2.305(2)
O(3)–Mo(1)–O(1)	103.64(11)	O(4)–Mo(1)–O(5)#1	77.33(8)
O(3)–Mo(1)–O(11)	101.63(11)	O(2)–Mo(1)–O(5)#1	76.08(8)
O(1)–Mo(1)–O(11)	103.54(11)	O(10)–Mo(3)–O(7)	105.17(14)
O(3)–Mo(1)–O(4)	89.98(10)	O(10)–Mo(3)–O(8)	102.64(11)
O(1)–Mo(1)–O(4)	153.53(10)	O(7)–Mo(3)–O(8)	97.52(11)
O(11)–Mo(1)–O(4)	95.59(10)	O(10)–Mo(3)–O(4)	99.01(11)
O(3)–Mo(1)–O(2)	96.49(10)	O(7)–Mo(3)–O(4)	104.26(11)
O(1)–Mo(1)–O(2)	84.12(10)	O(8)–Mo(3)–O(4)	143.91(10)
O(11)–Mo(1)–O(2)	157.90(10)	O(10)–Mo(3)–O(9)	103.28(11)
O(4)–Mo(1)–O(2)	71.71(9)	O(7)–Mo(3)–O(9)	151.49(11)
O(3)–Mo(1)–O(5)#1	166.75(10)	O(8)–Mo(3)–O(9)	74.28(9)
O(1)–Mo(1)–O(5)#1	86.70(10)	O(4)–Mo(3)–O(9)	72.85(9)
O(11)–Mo(1)–O(5)#1	83.62(10)	N(1)–Cu(1)–O(13)#4	114.29(11)
N(4)#3–Cu(1)–N(1)	149.67(12)	O(7)–Cu(1)–O(13)#4	80.53(10)
N(4)#3–Cu(1)–O(7)	101.42(11)	N(3)–Cu(2)–N(2)#5	151.84(13)
N(1)–Cu(1)–O(7)	98.59(11)	N(3)–Cu(2)–O(3)	117.89(11)
N(4)#3–Cu(1)–O(13)#4	91.44(10)	N(2)#5–Cu(2)–O(3)	84.88(11)

Symmetry code: #1 $-x+1, -y+1, -z+1$; #2 $x+1, y, z$; #3 $x, y+1, z$; #4 $x-1, y, z$; #5 $x, y, z+1$.

and refined anisotropically. A summary of the crystallographic data and structural determination for compound **1** are listed in Table 1. Selected bond lengths and angles are provided in Tables 2. Atomic coordinates and equivalent isotropical displacement parameters are given in Table S1.

3. Results and discussion

Although it is difficult to control and predict the crystal structures under hydrothermal conditions, hydrothermal synthesis is still an optimum method for the synthesis of the organic–inorganic hybrid materials [13]. The title compound is also hydrothermally prepared by reaction of MoO_3, $CuSO_4$ and quinoxaline in water. It was accepted that Cu(II) ions could be reduced to Cu(I) by 4,4′-bipyridine or 2,2′-dipyridylamine under hydrothermal conditions [14]. A similar reduction process might be performed in this reaction. However, the title compound could also be obtained using the cuprous chloride as the precursor and under same condition. Preliminary studies

demonstrated that the pH value was crucial to the product. For the successful isolation of **1**, the pH value must be controlled to the range of 4–5.

The structure of the three-dimensional polymer, $[Cu_2(quinoxaline)_2Mo_4O_{13}]_n$, is constructed from 1D $[Mo_4O_{13}]_n^{2n-}$ anion chains connected by pairs of $[Cu(quinoxaline)]_n^{n+}$ chains, of which the channels are occupied by the phenyl rings of quinoxaline ligands. As shown in Fig. 1, the asymmetric tetramolybdate subunit in compound **1** consists of three $[MoO_6]$ octahedra and one $[MoO_5]$ square pyramid. These polyhedra are further connected through edge- or corner-sharing to form a 1D tetramolybdate chain running along the *a*-axis (see Fig. 2a).

The $[Mo_4O_{13}]_n^{2n-}$ anion chain in the title compound may also be viewed as being built of octamolybdate subunits connected by sharing pairs of common vertices. Removal of the oxo-groups donated by the adjacent octamolybdate subunits, a ξ-isomer of $[Mo_8O_{26}]^{4-}$ (see Fig. 2b) is noted to be the basic building unit of the molybdenum oxide chain in compound **1**. To the best of our knowledge, such
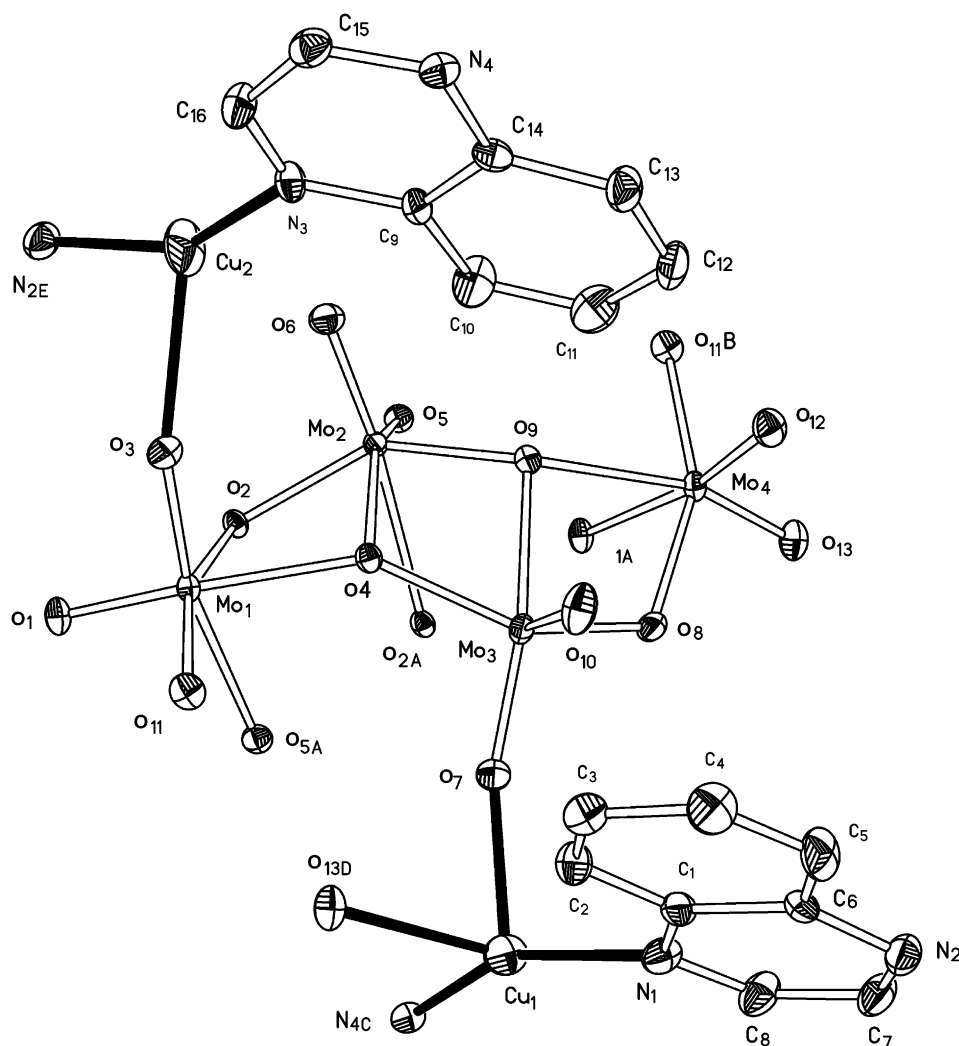

Fig. 1. ORTEP drawing of compound **1** with 50% probability level, showing the coordination environments around Cu and Mo atoms. All the hydrogen atoms are omitted for clarity.
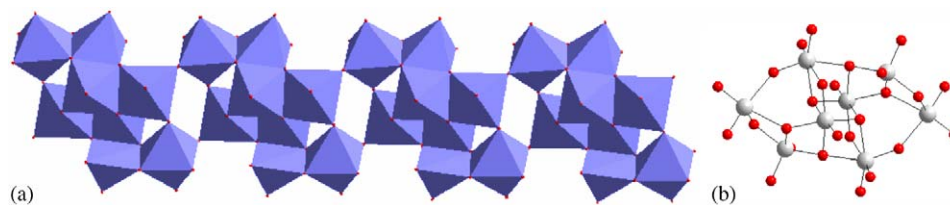
Fig. 2. (a) A polyhedral representation of the one-dimensional $[Mo_8O_{26}]_n^{4n-}$ chain in the compound. (b) A ball and stick representation of the ξ-$[Mo_8O_{26}]^{4-}$ cluster embedded in the molybdate chain of **1**.
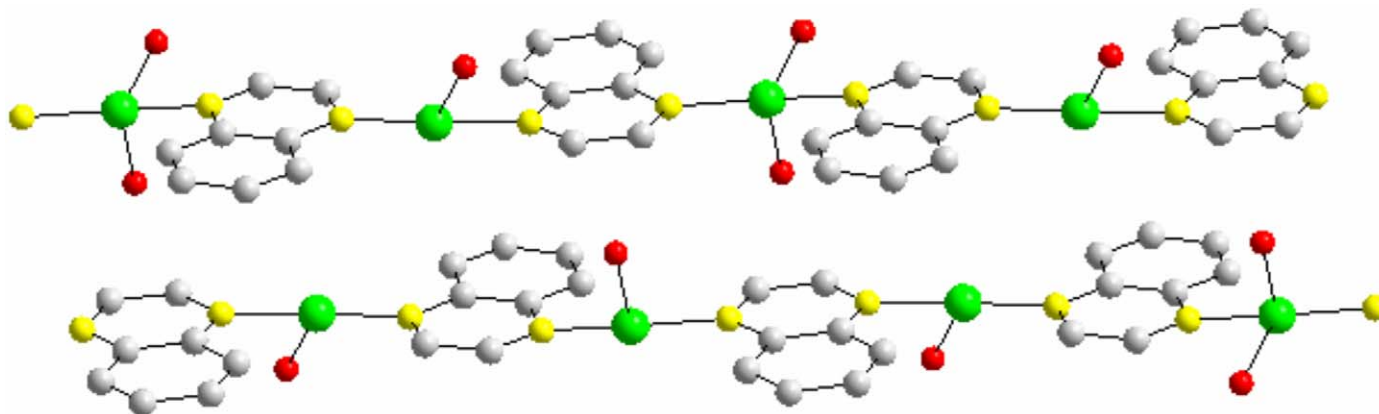

Fig. 3. A view of the [Cu(quinoxaline)] double chains running along the [011] direction in the compound.

molybdenum oxide chain constructed from this kind of octamolybdate cluster has never been described so far in the chemistry of POMs. Such isolated ξ-$[Mo_8O_{26}]^{4-}$ cluster, first described by Xu [15], is composed of a Mo_6O_6 ring containing two octahedral and four square pyramidal molybdenum atoms, and being capped on opposite faces by $[MoO_6]$ octahedra. Otherwise, some previous reports have erroneously equaled this ξ-isomer of the $[Mo_8O_{26}]^{4-}$ to the ζ-isomer [16]. Careful comparison of theses two isomers reveals that, although both of them are built from 4 octahedra and 4 square pyramids, the detailed arrangement of the polyhedra among them are different. In the ζ-isomer [17], all octahedra and two of the four square pyramids are positioned on the Mo_6O_6 ring and the other two square pyramids located on the two opposite cap sites, while in the ξ-isomer of this report, the cap positions are occupied by two octahedra, and the remaining two octahedra and four square pyramids build up the Mo_6O_6 ring (see Supporting Information, Fig. S3).

In **1**, the copper-quinoxaline moiety forms two unique zig-zag chains running approximately along the [011] direction, in which the tetrahedrally and trigonally coordinated Cu(I) sites are connected through quinoxaline ligand (see Fig. 3). The distorted tetrahedron is defined by two nitrogen atoms from the quinoxaline ligands (Cu–N = 2.010(6)–2.005(6) Å) and two terminal oxygen atoms (Cu–O = 2.411(4)–2.420(5) Å) from two adjacent ξ-$[Mo_8O_{26}]^{4-}$ units in the same molybdate chain. The other trigonally coordinated Cu(I) site is coordinated by two nitrogen atoms from quinoxaline ligands (Cu–N =

1.974(7)–1.998(6) Å) and one terminal oxygen group (Cu–O = 2.305(4) Å) from a molybdenum atom in a neighboring molybdate chain (see Fig. S4). These two nearly parallel chains stack in an off-set arrangement via strong π-π interactions with an average distance of 3.34 Å, which may play an important role in the stabilization of the 3D framework. It is quite interesting that within the Cu-ligand chain or even between the two adjacent stacked Cu-ligand chains, the oxygen vertices of the neighboring copper sites point away from each other so that the molybdenum oxide chains are linked alternatively to each side of the Cu-ligand chains (see Fig. 4). The nearest Cu···Cu separation of the neighboring chain is 4.631(7) Å, shorter than that of the intrachain (6.81(2) Å).

Comparing to compound $[Cu_2(C_8H_6N_2)_2(C_7H_6N_2)]$ $[Mo_8O_{26}]$ [18], in which the quinoxaline ligands and its oxidized product benzimidazole coordinate to two copper atoms and form binuclear Cu(I) fragment (see Fig. S5), the quinoxaline ligands in compound **1** are bridged by copper(I) atoms and form double unique isolated 1D chains. In contrast to compound **1**, compound $[Cu_2(C_5H_6 N_2)_2][Mo_4O_{13}]$ [19] also contains two $[Cu(2-methyl prazine)]_n^{n+}$ chains which are further joined up to form one 1D belt via Cu–O–Cu bridge (see Fig. S6).

The most remarkable feature of compound **1** is that the ξ-isomer of the $[Mo_8O_{26}]^{4-}$ anion is utilized as the basic building block, connected by sharing pairs of common vertices to yield an unprecedented 1D chain, and these chains are further connected by double $[Cu(quinoxaline)]_{2n}^{2n+}$ chains to form a 3D framework. To the best of our knowledge, the
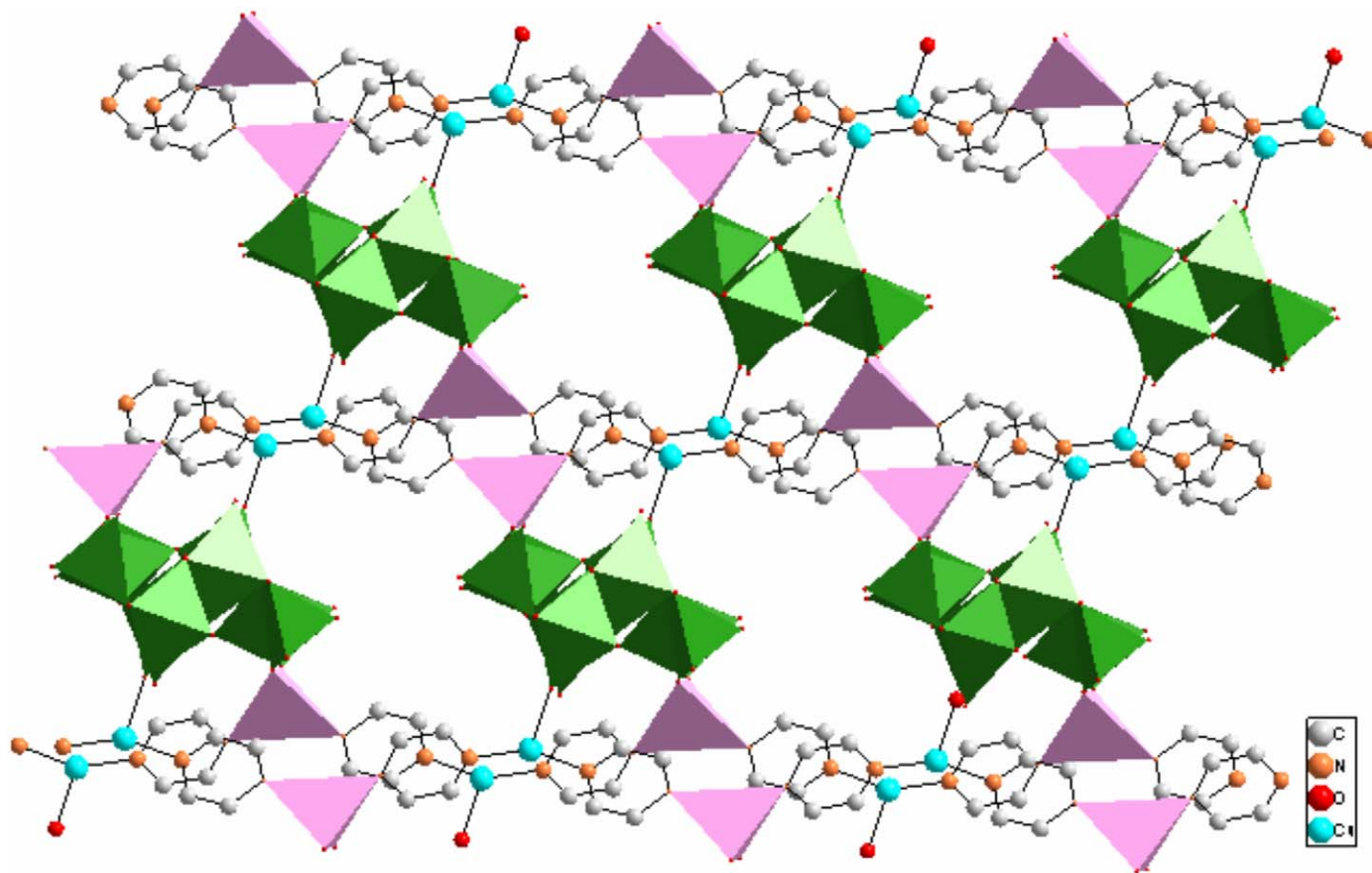
Fig. 4. View of the three-dimensional structure of compound **1** along the crystallographic *a*-axis. The phenyl rings of the quinoxaline and all H atoms are omitted for clarity. The green and magenta polyhedra represent for the $[MoO_x]$ and $[CuO_2N_2]$ polyhedra, respectively.

previous reported $[Mo_8O_{26}]_n^{4n-}$ anion chains have been all described to be built from γ-$[Mo_8O_{26}]^{4-}$ anions connected by sharing one common edge or two common corners. However, no example of analogous extended structure consisting of ξ-isomer of $[Mo_8O_{26}]^{4-}$ cluster has been reported in the literature, and also no examples of 2D sheets or 3D frameworks based on $[Mo_8O_{26}]_n^{4n-}$ anion chain have been reported up to date.

In the infrared spectrum of the title compound (as shown in Fig. S7), the strong bands at 945–816 cm^{-1} are ascribed to Mo$=$O stretching vibrations, while the bands in the range of 763–597 cm^{-1} are vibrations of Mo–O–Mo. The absorption bands in the range of 1502–1055 cm^{-1} can be assigned to characteristic peaks of quinoxaline ligand.

The thermogravimetric curve of the title complex is documented in Fig. 5. It shows that the structure of **1** remains stable up to about 300 °C. The weight loss of **1** between 300 and 430 °C should be assigned to the removal of the quinoxaline ligand (observed, 26.89%; calculated, 26.57%). After that there is a slight weight gain (<2%) in the range 430–500 °C, which might due to the oxidization of Cu(I). It can be observed that the result of the TG analysis basically agrees with that of the structure determination.
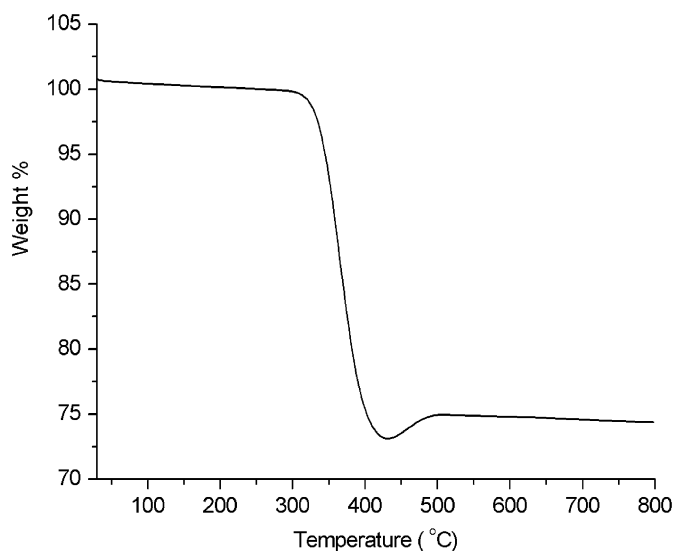

Fig. 5. The TG curve of the compound.

In conclusion, we have successfully synthesized a novel hybrid organic-inorganic material of quinoxaline ligated copper-tetramolybdate $[Cu_2(quinoxaline)_2Mo_4O_{13}]$. The

compound exhibits three-dimensional framework, which contains one dimensional ξ-octamolybdate chains integrated by pairs of copper-ligand chains. It represents the first example of $[Mo_8O_{26}]_n^{4n-}$ based complex containing ξ-octamolybdate chains other than γ-octamolybdate chains, and also the first example that the octamolybdate chain is extended to multi-dimensional framework.

4. Supplementary material

Cryatallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC-290246. Copies of the data can be obtained free of charge on application to CCDC. 12 Union Road Cambridge CB2 1EZ, UK (Fax: +44-1223-336-033; mail to deposit@ccdc.cam.ac.uk).

Acknowledgments

Appendix A. Supporting Information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2006.04.054.

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