

catena-Poly[[2,2'-diamino-4,4'-bithiazole] $\{\mu_3$ -*cis*-*N*-(2-carboxylatophenyl)-*N'*-[3-(dimethylamino)propyl]-oxamidato(3-)]dicopper(II)] nitrate 0.6-hydrate]

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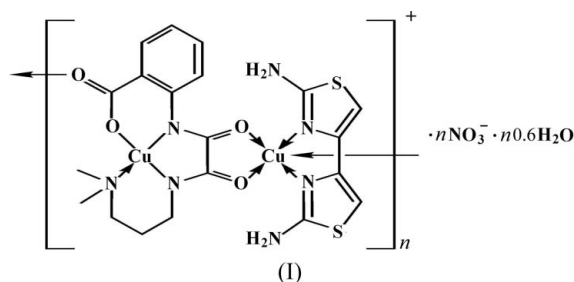
The title complex, $\{[\text{Cu}_2(\text{C}_{14}\text{H}_{16}\text{N}_3\text{O}_4)(\text{C}_6\text{H}_6\text{N}_4\text{S}_2)]\text{NO}_3 \cdot 0.6\text{H}_2\text{O}\}_n$, is a one-dimensional copper(II) coordination polymer bridged by *cis*-oxamide and carboxylate groups. The asymmetric unit is composed of a dinuclear copper(II) cation, $[\text{Cu}_2(\text{dmapob})(\text{dabt})]^+$ {dmapob is *N*-(2-carboxylatophenyl)-*N'*-[3-(dimethylamino)propyl]oxamidate and dabt is 2,2'-diamino-4,4'-bithiazole}, one nitrate anion and one partially occupied site for a solvent water molecule. The two Cu^{II} ions are located in square-planar and square-pyramidal coordination environments, respectively. The separations of the Cu atoms bridged by oxamide and carboxylate groups are 5.2053 (3) and 5.0971 (4) Å, respectively. The complex chains are linked by classical hydrogen bonds to form a layer and then assembled by π - π stacking interactions into a three-dimensional network. The influence of the terminal ligand on the structure of the complex is discussed.

Comment

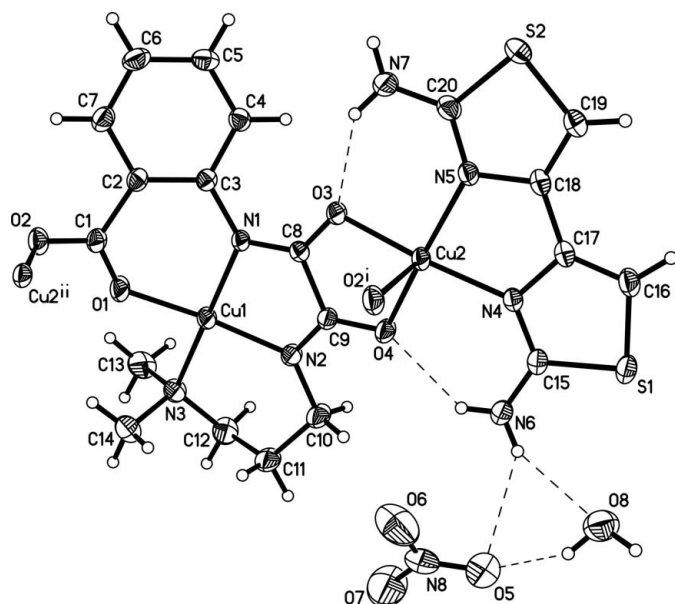
Polymeric metal complexes, due to their novel structures, special properties and potential applications as metallo-enzymes and catalysts, are of current interest, attracting increasing research effort (Miller & Epstein, 1994; Zhan *et al.*, 2007). A successful strategy to construct polymeric metal complexes is self-assembly of metal ions and ligands with versatile coordination modes (Chen *et al.*, 1998). *N,N'*-Disubstituted oxamides have played a key role in the design and synthesis of polymetallic systems, because their coordination abilities towards transition metal ions can be modified by changing the nature of the amide substituents (Ojima & Nonoyama, 1988). One of the outstanding characteristics of these ligands is the easy transformation of *cis*-*trans* confor-

mations, which makes it practical to design tunable molecular materials with extended structures and desired properties (Ruiz *et al.*, 1999). To date, many polymeric structures based on symmetrical *N,N'*-disubstituted oxamides have been reported (Costes *et al.*, 1999; Gulbrandsen *et al.*, 1993; Jiang *et al.*, 2009; Li *et al.*, 2008; Lloret *et al.*, 1993; Nakatani *et al.*, 1991; Rangmathan *et al.*, 1995; Pei *et al.*, 1988; Zhang *et al.*, 2001). In contrast, due to the difficulty of synthesis, reports of complexes based on disymmetrical *N,N'*-disubstituted oxamides are few (Pei *et al.*, 1989; Zang *et al.*, 2003). To the best of our knowledge, only a two-dimensional copper(II)-manganese(II) polymer, *viz.* $\{[\text{Cu}(\text{oxbe})]\text{Mn}(\text{H}_2\text{O})[\text{Cu}(\text{oxbe})\text{-(DMF)}]\cdot\text{DMF}\cdot\text{H}_2\text{O}\}_n$ [oxbe is *N'*-(2-aminoethyl)-*N*-benzoatoxamide and DMF is dimethylformamide], (II), bridged simultaneously by disymmetrical *cis*-oxamide and carboxylate groups on the benzoate, has been reported (Zang *et al.*, 2003), and no one-dimensional copper(II) complexes bridged by disymmetrical *N,N'*-disubstituted oxamides with the same coordination mode are known to date. However, complexes bridged by disymmetrical *N,N'*-disubstituted oxamides have shown magnetic properties (Larionova *et al.*, 1997; Matović *et al.*, 2005; Pei *et al.*, 1989, 1991; Zang *et al.*, 2003) and this prompted us to design and synthesize new polynuclear complexes.

Recently, we have synthesized a series of disymmetrical *N,N'*-disubstituted-oxamide-bridged polynuclear metal complexes (Gu *et al.*, 2009; Li *et al.*, 2003, 2004; Liu *et al.*, 2008). As an extension of our earlier research work and in order to investigate further the effect of terminal ligands on forming coordination polymers, we used *N*-(2-carboxylatophenyl)-*N'*-[3-(dimethylamino)propyl]oxamidate (dmapob) as a bridging ligand and 2,2'-diamino-4,4'-bithiazole (dabt) as a terminal ligand to synthesize the title novel one-dimensional copper(II) complex, (I).



Compound (I) exists as a one-dimensional coordination polymer extending along the *b* axis, constructed by the *cis*-oxamide and carboxylate groups connecting the Cu^{II} ions, and this structural unit can be considered as a *cis*-oxamide-bridged dinuclear copper(II) fragment with dabt as the terminal ligand. An uncoordinated nitrate anion and a (depleted) solvent water molecule link to the chain through N—H···O and O—H···O hydrogen bonds (Fig. 1 and Table 2). In the dinuclear fragment, the *cis*-oxamide group bridges the Cu^{II} ions in the usual chelating mode, with a bite angle of 84.68 (8)° for Cu1 and 83.74 (7)° for Cu2. Atoms Cu1 and Cu2 are 5.2053 (4) Å apart through the oxamide bridge. The dinuclear

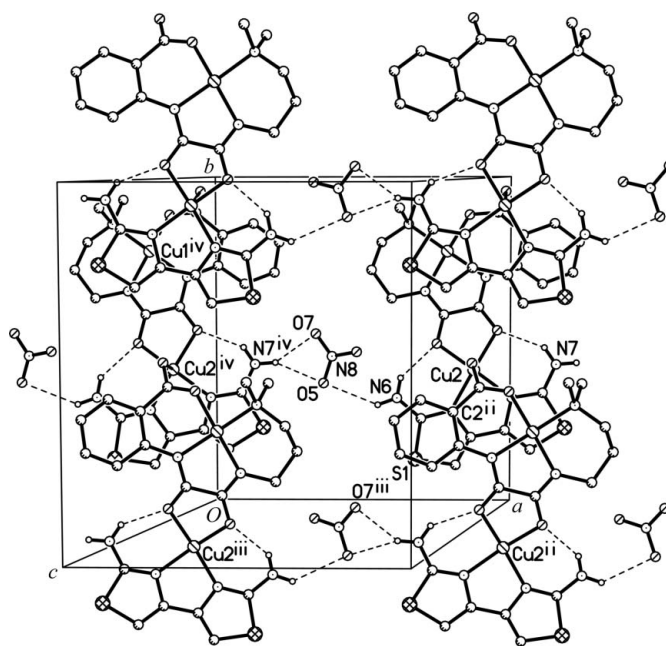

Figure 1

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$]

fragments are bridged by the carboxylate groups in a nonplanar skew-skew fashion. The torsion angles Cu1—O1—C1—O2 [$-148.44(19)^\circ$] and Cu2ⁱⁱ—O2—C1—O1 [$95.1(2)^\circ$] [symmetry code: (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; Table 1] are similar to those found in other complexes with such carboxylate groups (Duan *et al.*, 2006; Tong *et al.*, 1997). The Cu...Cu separation through the carboxylate bridge is 5.0970 (4) Å.

The coordination environments of the two Cu^{II} ions are different. Atom Cu1, at the inner site of the *cis*-dmapob ligand, is located in a distorted [CuN₃O] square-planar environment. The maximum displacement of the coordination atoms from their least-squares plane is 0.1990 (13) Å for atom N1, and the displacement of atom Cu1 from this plane is 0.1067 (11) Å. Atom Cu2 is in a slightly distorted [CuN₃O₂] square-pyramidal coordination geometry with a τ value of 0.11 (Addison *et al.*, 1984). The basal plane consists of two *exo* O atoms from the oxamide group and two N atoms from the dabt ligand, with a maximum deviation from the least-squares plane of 0.0588 (9) Å (atom N5). The apical position is occupied by a carboxylate O atom [O2ⁱ; symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$]. In this disposition, atom Cu2 is displaced 0.1881 (10) Å out of the basal plane towards the apical site.

The oxamide ligand coordinates to atom Cu1 in a tetradentate manner, forming one five- and two six-membered chelate rings (Fig. 1). The five-membered ring is planar. Regarding the two six-membered rings, the one formed by the propylenediamine fragment adopts a half-chair conformation, with puckering parameters (Cremer & Pople, 1975) of $Q = 0.539(3)$ Å, $\theta = 127.3(3)^\circ$ and $\varphi = 30.8(3)^\circ$. The second six-membered ring has a boat conformation, with puckering parameters $Q = 0.332(2)$ Å, $\theta = 89.0(3)^\circ$ and $\varphi = 119.7(4)^\circ$. The Cu1—N1 and Cu1—N2 bonds are shorter than Cu1—N3,


Figure 2

The two-dimensional hydrogen-bonded structure of (I), parallel to (001), viewed perpendicular to the plane of the thiazole ring containing atom S1 of the dabt ligand. Hydrogen bonds are shown as dashed lines and H atoms not involved in hydrogen bonding have been omitted for clarity. Note that solvent water molecule O8 is depleted, with an occupancy factor of 0.60. [Symmetry codes: (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $x - 1, y, z$]

which is consistent with the stronger donor abilities of the Nsp^2 atoms over the Nsp^3 atoms (Jubert *et al.*, 2002).

In the crystal structure, the (depleted) solvent water molecules and nitrate ions are assembled into a one-dimensional hydrogen-bonded chain parallel to the complex polymers (Fig. 2 and Table 2). The latter are linked to the chains through hydrogen bonds involving the amino groups of the dabt ligands, giving rise to a two-dimensional classical hydrogen-bonding structure parallel to the (010) plane. Besides the hydrogen bonds, the layer contains an aromatic π - π stacking interaction between the thiazole ring containing atom S1 and the benzene ring of the dmapob ligand at $(-x + 2, y - \frac{1}{2}, -z + \frac{1}{2})$, denoted (i). The smallest separation is 3.325 (3) Å (C2ⁱ, Table 3). Moreover, there is another kind of π - π interaction between the layers. It also involves the same S1-thiazole and benzene rings, but now the benzene ring is at $(-x + 2, -y + 1, -z)$, denoted (v). The minimum distance is 3.215 (3) Å (Table 3, C5^v of the thiazole ring).

On comparing complex (I) with the copper(II)-manganese(II) heterotrimeric polymer, (II), reported by Zang *et al.* (2003), it can be seen that the two complexes have a similar skeleton, *viz.* an oxamidate bridging structure. Both of them are simultaneously bridged by disymmetrical *cis*-oxamide and carboxylate groups on benzoates, and the main difference resides in the terminal ligand (dabt) in complex (I), which generates two distinct results. Firstly, the chelating terminal ligand reduces the number of coordination sites of the metal ions used for coordinating to the bridging groups, and this has an influence on the dimensionality of the complex polymer. As

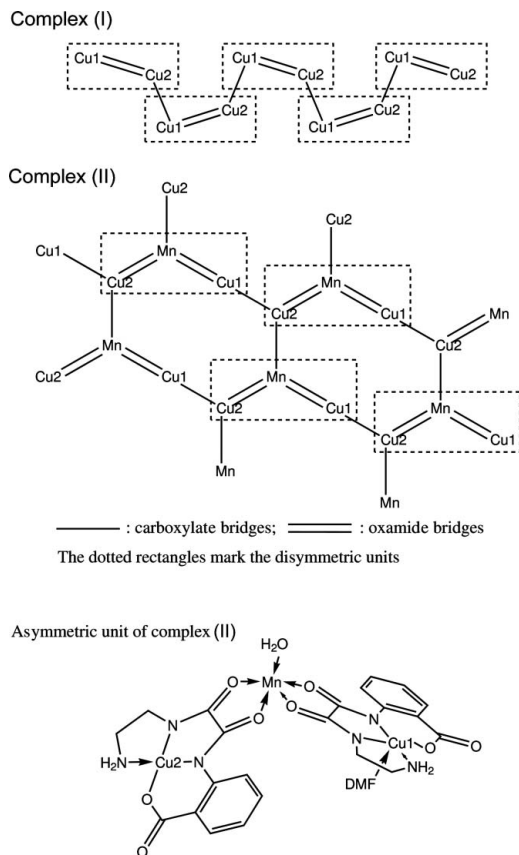


Figure 3
A schematic drawing of the bridging skeletons in complex polymers (I) and (II).

shown in Fig. 3, in complex (II) the structural unit is a heterotrimeric $\text{Cu}^{\text{II}}\text{-Mn}^{\text{II}}\text{-Cu}^{\text{II}}$ fragment, which leaves two uncoordinated O atoms of the coordinated carboxylate groups on atoms Cu1 and Cu2 and two spare coordination sites on atoms Cu2 and Mn. Consequently, complex (II) exists as a two-dimensional polymer, while in the unit of (I) only one donor O atom and one spare coordination site remain, thus giving rise to a one-dimensional complex chain. Secondly, the aromatic terminal ligands also affect the supramolecular structure due to $\pi\text{-}\pi$ stacking interactions. In complex (II), the two-dimensional complex polymers are assembled only by hydrogen bonds to form a three-dimensional network. However, in complex (I), as depicted above, the aromatic stacking interactions make an important contribution to the three-dimensional supramolecular structure. This is a clear example of how terminal ligands may strongly affect the construction of metal complex polymers by influencing the coordination sites and intermolecular interactions, and further investigations on the subject are proceeding in our laboratory.

Experimental

All chemicals were of analytical reagent grade. H_3dmapob (0.0147 g, 0.05 mmol) was dissolved in methanol (5 ml). Piperidine (0.75 ml, 0.15 mmol) and a solution of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (0.0242 g, 0.10 mmol) in methanol (5 ml) were added with continuous stirring. After 0.5 h, a solution of *dabt* (0.01 g, 0.05 mmol) in methanol (5 ml) was added

Table 1
Selected bond lengths (\AA).

| | | | |
|--------|-------------|---------------------|-------------|
| Cu1—N1 | 1.9737 (18) | Cu1—O1 | 1.9072 (18) |
| Cu1—N2 | 1.950 (2) | Cu2—O2 ⁱ | 2.3192 (18) |
| Cu1—N3 | 2.040 (2) | Cu2—O4 | 1.9547 (17) |
| Cu2—N4 | 1.9658 (19) | Cu2—O3 | 1.9629 (16) |
| Cu2—N5 | 1.9853 (19) | | |

Symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$.

Table 2
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O8—H8A...O5 | 0.82 | 1.97 | 2.717 (5) | 152 |
| O8—H8B...O7 ⁱⁱ | 0.82 | 2.16 | 2.838 (6) | 140 |
| N6—H6A...O4 | 0.80 (3) | 2.22 (3) | 2.889 (3) | 140 (3) |
| N6—H6B...O5 | 0.82 (3) | 2.56 (3) | 3.206 (4) | 136 (3) |
| N6—H6B...O8 | 0.82 (3) | 2.19 (3) | 2.953 (5) | 154 (3) |
| N7—H7A...O3 | 0.83 (2) | 2.15 (2) | 2.863 (3) | 144 (3) |
| N7—H7B...O5 ⁱⁱⁱ | 0.84 (2) | 2.34 (2) | 3.101 (4) | 151 (2) |
| N7—H7B...O7 ⁱⁱⁱ | 0.84 (2) | 2.22 (2) | 2.986 (4) | 151 (2) |

Symmetry codes: (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x + 1, y, z$.

Table 3
Geometric parameters of the $\pi\text{-}\pi$ stacking interactions in (I).

R1 denotes the N4/C15/S1/C16/C17 thiazole ring and *R2* the C2—C7 benzene ring. C—C, α and β denote the inter-centroid separation, the dihedral angle between the ring planes and the offset angle, respectively. The separations are the distances between individual atoms of the second ring and the first ring plane, these atoms being projected perpendicularly on to the first ring.

| First ring | Second ring | C—C (\AA) | α ($^\circ$) | β ($^\circ$) | Separations (\AA) |
|------------|------------------------|----------------------|-----------------------|----------------------|---|
| <i>R1</i> | <i>R2</i> ⁱ | 3.6717 (14) | 12.18 (12) | 14.63 | C2 ⁱ , 3.325 (3) C3 ⁱ , 3.585 (2) C4 ⁱ , 3.827 (3) |
| <i>R1</i> | <i>R2</i> ^v | 3.9438 (14) | 22.46 (12) | 24.47 | C5 ^v , 3.215 (3) C6 ^v , 3.717 (3) |

Symmetry codes: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (v) $-x + 2, -y + 1, -z$.

dropwise. The reaction mixture was stirred at 333 K for 6 h and filtered. Green block-shaped crystals of (I) of suitable size for X-ray diffraction analysis were obtained by slow evaporation of the filtrate at room temperature for three weeks (yield 63%). Elemental analysis calculated for $\text{C}_{20}\text{H}_{23.2}\text{Cu}_2\text{N}_8\text{O}_{7.6}\text{S}_2$: C 23.18, H 2.25, N 10.8%; found: C 25.02, H 2.46, N 10.03%.

Crystal data

| | |
|---|---|
| $[\text{Cu}_2(\text{C}_{14}\text{H}_{16}\text{N}_3\text{O}_4)(\text{C}_6\text{H}_6\text{N}_4\text{S}_2)]\cdot\text{NO}_3 \cdot 0.6\text{H}_2\text{O}$ | $\beta = 107.103 (2)^\circ$ |
| $M_r = 688.51$ | $V = 2558.77 (10) \text{\AA}^3$ |
| Monoclinic, $P2_1/c$ | $Z = 4$ |
| $a = 12.6991 (3) \text{\AA}$ | Mo $K\alpha$ radiation |
| $b = 14.0263 (3) \text{\AA}$ | $\mu = 1.89 \text{ mm}^{-1}$ |
| $c = 15.0300 (3) \text{\AA}$ | $T = 296 \text{ K}$ |
| | $0.42 \times 0.15 \times 0.06 \text{ mm}$ |

Data collection

| | |
|--|--|
| Bruker APEX CCD area-detector diffractometer | 11743 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2002) | 5918 independent reflections |
| $T_{\min} = 0.505, T_{\max} = 0.895$ | 4589 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.020$ |

Refinement

$$R[F^2 > 2\sigma(F^2)] = 0.034$$

$$wR(F^2) = 0.094$$

$$S = 1.04$$

5918 reflections

380 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

$$\Delta\rho_{\max} = 0.44 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.31 \text{ e } \text{Å}^{-3}$$

The occupancy of the depleted solvent water molecule (O8) was initially refined, and then fixed in the final stages of refinement. Water and amino H atoms were found in an electron-density difference map. Water H atoms were further idealized, with O—H = 0.82 Å, and then treated as riding, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. Amino H atoms were refined with N7—H restrained to 0.84 (2) Å and free isotropic displacement parameters. The remaining H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 Å (methylene), and refined in riding mode, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic and methylene groups or $1.5U_{\text{eq}}(\text{C})$ for methyl groups.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BG3129). Services for accessing these data are described at the back of the journal.

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