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## An oxamidato-bridged binuclear imidazole–copper(II) complex

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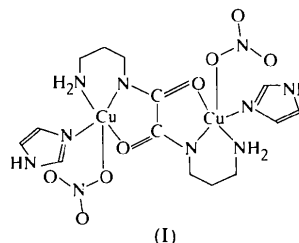
### Abstract

The oxpn ligand [oxpn = *N,N'*-bis(3-aminopropyl)oxamide] in the title compound,  $\mu$ -[*N,N'*-bis(3-aminopropyl)oxamidato(2-)]-*N,N',O':N'',N'''*,*O*-bis[(imidazole-*N*<sup>3</sup>)(nitrate-*O*)copper(II)], [ $\text{Cu}_2(\text{C}_8\text{H}_{16}\text{N}_4\text{O}_2)(\text{NO}_3)_2$ ]( $\text{C}_3\text{H}_4\text{N}_2$ )<sub>2</sub>], is bis-tridentate in a *trans* conformation, bridging two Cu<sup>II</sup> ions to form a centrosymmetric binuclear molecule. The symmetry-equivalent central Cu<sup>II</sup> ions are located in a distorted square-based pyramid, with an O atom from the nitrate ion bonded at the axial site, and a Cu···Cu distance of 5.242 (2) Å.

### Comment

As extending bridging ligands transmitting a strong magnetic exchange interaction, oxamidates have played a key role in the design of polymetallic systems (Ojima & Nonoyama, 1988). One of the most important factors for the versatility of the ligands is their easy *cis*–*trans* conformational change to afford symmetric or asymmetric oxamidato bridges. We report here the

crystal structure of a binuclear copper(II) complex with a bridging *N,N'*-bis(3-aminopropyl)oxamide (oxpn) ligand in the *trans* conformation, (I).



The structure of this complex consists of centrosymmetric binuclear molecules, with oxpn acting as a bis-tridentate ligand, in which the Cu<sup>II</sup> ions are bridged by a *trans*-oxamidato group with an inversion centre in the middle of the C1–C1' bond [symmetry code: (i) 2 – *x*, 1 – *y*, 1 – *z*] (Fig. 1). The Cu<sup>II</sup> ion is in a distorted square-pyramidal geometry, with three atoms from the oxpn ligand (N1, N2 and O1) and one N atom (N3) from the imidazole in the basal plane, while the axial coordination site is occupied by a nitrate O atom (O2). The four atoms of the coordination plane are almost coplanar, with deviations from the least-squares plane of less than 0.055 Å. The Cu<sup>II</sup> ion deviates by 0.043 Å towards the O atom belonging to the semicoordinated nitrate anion [Cu–O2 2.673 (2) Å].

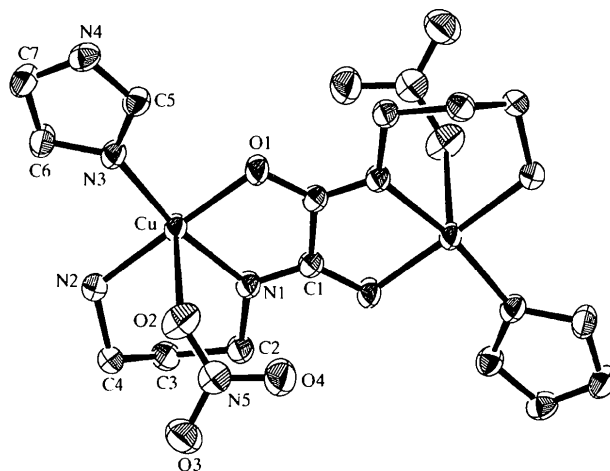


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

The bond distances for Cu–N1, Cu–N2 and Cu–N3 [1.967 (2), 2.003 (2) and 1.982 (2) Å, respectively] are close to those observed in parent oxamidato-bridged copper(II) complexes (Chen, Tang & Yu, 1994; Sanz *et al.*, 1996). The significant shortening of the Cu–N1 bond distance is in agreement with the stronger basicity of the deprotonated amide N atom (Chen, Tang

& Yu, 1994; Chen, Fu, Yu & Tang, 1994). The bis-tridentate character of the ligand produces one five- and one six-membered chelate ring on each Cu atom, the angle subtended at the metal atom by the latter being larger [95.10(8)° for N1—Cu—N2] than that of the former [83.57(8)° for O1—Cu—N1]. The three atoms around N1 lie in a plane, with bond angles of 112.7(2), 129.4(2) and 117.6(2)° for Cu—N1—C1, Cu—N1—C2 and C1—N1—C2, respectively. This fact, together with the values of the bond lengths involved and the planarity of the *N*-oxamidato ligand, reveals that N1 and its symmetry-related N1<sup>i</sup> atom are *sp*<sup>2</sup>-hybridized and that the  $\pi$ -carbonyl electrons are delocalized to form a conjugated system. The length of C1—C1<sup>i</sup> [1.504(4) Å] is very close to that observed in other oxamidato-bridged copper(II) complexes (Chen, Tang & Yu, 1994; Chen *et al.*, 1996).

The nitrate group is planar, as expected. The imidazole ring shows no significant deviation from planarity, with the largest deviation being 0.0025 Å. The Cu...Cu separation through the oxpn bridge is 5.242(2) Å. The binuclear molecules are connected by hydrogen bonds involving the imidazole N4—H9 group and the O4<sup>ii</sup> atom [symmetry code: (ii) 1 - *x*, 1 - *y*, 1 - *z*], with an N...O distance of 2.876(3) Å.

## Experimental

The H<sub>2</sub>oxpn ligand and its mononuclear copper compound, Cu(oxpn)<sub>2</sub>·H<sub>2</sub>O, were obtained according to the literature method of Ojima & Yamada (1968). The binuclear complex was prepared by adding an aqueous solution of copper nitrate (1 mmol, 5 ml) to a heated aqueous solution of Cu(oxpn)<sub>2</sub>·H<sub>2</sub>O (1 mmol, 50 ml) with stirring. An aqueous solution of imidazole (2 mmol, 10 ml) was then added to the resulting blue solution. After standing at room temperature for several days, well formed green crystals of (I) were obtained by slow evaporation.

## Crystal data

[Cu <sub>2</sub> (C <sub>8</sub> H <sub>16</sub> N <sub>4</sub> O <sub>2</sub> )(NO <sub>3</sub> ) <sub>2</sub> ·(C <sub>3</sub> H <sub>4</sub> N <sub>2</sub> ) <sub>2</sub> ]	Mo <i>K</i> α radiation
<i>M<sub>r</sub></i> = 587.50	λ = 0.7107 Å
Triclinic	Cell parameters from 16 reflections
<i>P</i> $\bar{1}$	θ = 18.28–25.18°
<i>a</i> = 7.898(2) Å	μ = 2.0 mm <sup>-1</sup>
<i>b</i> = 10.188(3) Å	<i>T</i> = 293 K
<i>c</i> = 7.064(2) Å	Prismatic
α = 95.00(2)°	0.30 × 0.24 × 0.20 mm
β = 92.92(2)°	Green
γ = 105.17(2)°	
<i>V</i> = 544.9(2) Å <sup>3</sup>	
<i>Z</i> = 1	
<i>D<sub>x</sub></i> = 1.790 Mg m <sup>-3</sup>	
<i>D<sub>m</sub></i> not measured	

## Data collection

Rigaku AFC-7R diffractometer	2328 reflections with <i>I</i> > 3σ( <i>I</i> )
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ω/2θ scans

Absorption correction:

azimuthal scans (TEXSAN; Molecular Structure Corporation, 1985)

*T*<sub>min</sub> = 0.447, *T*<sub>max</sub> = 0.670  
2696 measured reflections  
2513 independent reflections

## Refinement

Refinement on *F*

*R* = 0.027

*wR* = 0.040

*S* = 1.65

2513 reflections

155 parameters

H atoms riding

*w* = 1/[σ<sup>2</sup>(*F*)]

*R*<sub>int</sub> = 0.014

θ<sub>max</sub> = 27.5°

*h* = 0 → 9

*k* = -13 → 13

*l* = -9 → 9

3 standard reflections

every 200 reflections

intensity decay: 0.29%

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.45 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.54 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Cu—O1	1.977(2)	Cu—N3	1.982(2)
Cu—O2	2.673(2)	O1—C1 <sup>i</sup>	1.279(3)
Cu—N1	1.967(2)	N1—C1	1.288(3)
Cu—N2	2.003(2)	C1—C1 <sup>i</sup>	1.504(4)
O1—Cu—O2	95.49(8)	N2—Cu—N3	93.45(9)
O1—Cu—N1	83.57(8)	Cu—O1—C1	111.4(1)
O1—Cu—N2	178.52(7)	Cu—O2—N5	125.4(2)
O1—Cu—N3	87.93(8)	Cu—N1—C1	112.7(2)
O2—Cu—N1	87.68(8)	Cu—N1—C2	129.4(2)
O2—Cu—N2	83.78(8)	C1—N1—C2	117.6(2)
O2—Cu—N3	99.02(8)	Cu—N2—C4	117.7(2)
N1—Cu—N2	95.10(8)	Cu—N3—C5	124.0(2)
N1—Cu—N3	169.66(8)	Cu—N3—C6	130.5(2)

Symmetry code: (i) 2 - *x*, 1 - *y*, 1 - *z*.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1295). Services for accessing these data are described at the back of the journal.

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## (2,2'-Bipyridyl-*N,N'*)di-*n*-butyldichlorotin(IV)

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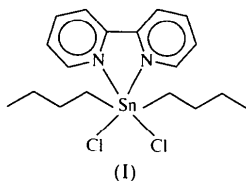
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### Abstract

The title compound, [SnCl<sub>2</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)], is the product of the reaction of 2,2'-bipyridyl with dibutyltin dichloride. The Sn<sup>IV</sup> centre is octahedrally coordinated by a bidentate 2,2'-bipyridyl ligand, two *cis* chlorides and two *trans* butyl groups. The extensively delocalized bipyridyl group is not absolutely planar, with the pyridyl rings twisted by 7.5 (3)°.

### Comment

The title compound is a six-coordinated organotin complex of the type R<sub>2</sub>SnX<sub>2</sub>L<sub>2</sub>, where R is an alkyl or aryl group, X is a halogen and L<sub>2</sub> is a bidentate nitrogen-donor ligand. The importance of this compound comes from its reported antitumour activity (Crowe *et al.*, 1984), which is probably due to the rather long Sn—N bond lengths. The structural investigation of the present compound, (I), is part of an ongoing study of a series of diorganotin dichelate complexes with nitrogenous bidentate ligands.



The Sn, the two Cl and the two N atoms of the bipyridyl group are coplanar, with an r.m.s. deviation of 0.013 Å, and this plane forms angles of 5.9 (2) and 4.1 (3)° with the pyridyl rings (Fig. 1). The two planar butyl groups (C11—C12—C13—C14 and C15—C16—C17—C18) are almost perpendicular to the above-mentioned basal plane of the molecule, with angles of 84.8 (8) and 81.7 (6)°, and form a dihedral angle of 69 (1)°. Each pyridyl ring is planar (r.m.s. deviations of 0.003 and 0.011 Å).

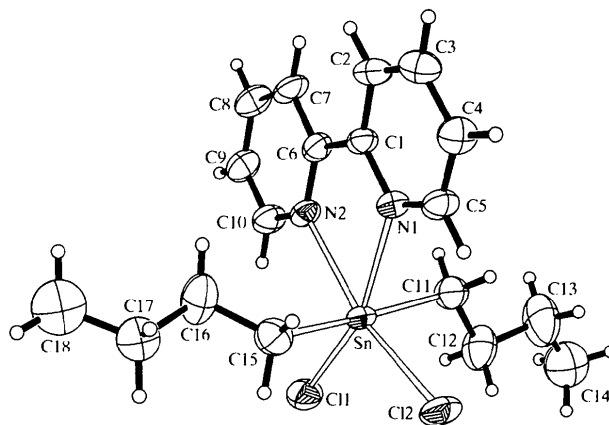


Fig. 1. View of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major disorder component is shown, and one H atom on each of C16 and C18 is wholly obscured by the parent atom.

There are deviations from ideal octahedral geometry about the Sn atom, as was also observed in the case of 2,2'-bipyridyldichlorodiphenyltin (Harrison *et al.*, 1974). The C11—Sn—C15 angle is only 169.3 (4)°, while the bond angles subtended at tin lie in the range 84.5 (3)–96.7 (3)°. The C11—Sn—C12 angle of 104.28 (8)° is the result of the mutual repulsion of the two Cl atoms, while the N1—Sn—N2 angle of 67.8 (2)° is similar to values given in the literature (Harrison *et al.*, 1974; Kabanos *et al.*, 1992).

The difference in the Sn—C11 and Sn—C12 bond lengths is probably due to a specific intermolecular interaction involving the H atom attached to C2 and the C12 atom of a symmetry-related molecule; the C2...C12(−1 + x, y, z) separation is 3.598 (8) Å and the C2—H2...C12 angle has a value of 166°.

### Experimental

The title compound was prepared by addition of a benzene solution of 2,2'-bipyridyl to a benzene solution of dibutyltin dichloride. Crystals suitable for X-ray analysis were obtained from hot benzene (Harrison *et al.*, 1974).