

especially between the 1,2- and 2,3-naphthaldehyde derivatives [1.904 (4) and 1.937 (5) Å, respectively]. Although similar differences have been described for Cu–N bond lengths in several copper–salicylaldimine complexes (Jain & Syal, 1973), those may be partly caused by the variation in the group bonded to the N atoms, while in (II) and (III), the same N-substituent is used.

The observed variation could then be ascribed in this case to the different positions of the coordinating groups within the naphthalene rings (1,2- or 2,3-). The 1,2-bond in naphthalene has been ascribed (Badger, 1969) a larger double-bond character than the 2,3-bond and this might favour a larger delocalization of the electron density in the 1,2-derivative which would then make compound (II) more stable than compound (III).

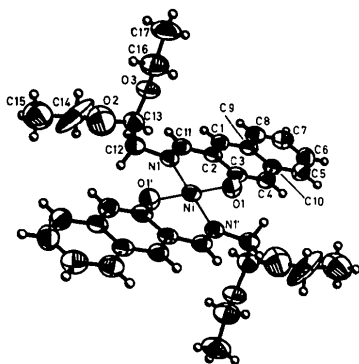


Fig. 4. The molecular structure of (III) showing the atom-numbering scheme adopted. The thermal ellipsoids show 50% probability.

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## Structure of [*N,N'*-Bis(3-aminopropyl)-1,2-ethanediamine]perchloratocopper(II) Perchlorate Hemihydrate

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**Abstract.** [Cu(ClO<sub>4</sub>)(C<sub>8</sub>H<sub>22</sub>N<sub>4</sub>)]ClO<sub>4</sub>·½H<sub>2</sub>O, *M<sub>r</sub>* = 445.74, orthorhombic, *Pbca*, *a* = 13.939 (3), *b* = 16.169 (6), *c* = 15.325 (5) Å, *V* = 3454 (6) Å<sup>3</sup>, *Z* = 8, *D<sub>m</sub>* = 1.68 (8) (by flotation in CCl<sub>4</sub>/hexane mixture), *D<sub>x</sub>* = 1.714 Mg m<sup>-3</sup>, Mo *K*α, λ = 0.71069 Å, μ =

The large vibrational amplitudes of the ethoxy groups in all of the compounds limited the precision of the structure determinations. In order to achieve a better understanding of the system, further studies on related compounds were undertaken and the results will be published elsewhere (Fernández, Rosales, Rubio, Salcedo, Toscano & Vela, in preparation).

We thank Mr Abelardo Cuellar for technical assistance.

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**Introduction.** Copper(II) exhibits a diversity of stereochemistry in its complexes with aliphatic tetraamines: trigonal bipyramidal (Sheu, Lee, Lu, Liang & Chung, 1983), square pyramidal (Lu, Wu & Chung, 1986), elongated tetragonal octahedral (Clay, Murray-Rust & Murray-Rust, 1979), or a distorted stereochemistry involving out-of-the-plane ligands positioned both along and off the *z* axis (Davey & Stephens, 1971). As part of our continuing studies on the structures of copper(II)-tetraamine complexes (Lu, Wu & Chung, 1986; Lee, Lee, Hong, Wu & Chung, 1986), the structure of the title compound has been determined and is reported here along with a comparison of its structural features with those reported for other Cu<sup>II</sup> complexes of linear aliphatic tetraamines of the type H<sub>2</sub>N(CH<sub>2</sub>)<sub>*l*</sub>NH-(CH<sub>2</sub>)<sub>*m*</sub>NH(CH<sub>2</sub>)<sub>*n*</sub>NH<sub>2</sub> denoted by the symbol *l,m,n*-tet, where the values of *l*, *m*, and *n* are 2 or 3 (Marongiu, Lingafelter & Paoletti, 1969; Fawcett, Rudich, Toby, Lalancette, Potenza & Schugar, 1980).

**Experimental.** Copper perchlorate hexahydrate (2.0 g in 25 ml water) was added to ethanol solution of *N,N'*-bis(3-aminopropyl)-1,2-ethanediamine (1.0 g in 40 ml ethanol). Blue crystals of [*N,N'*-bis(3-aminopropyl)-1,2-ethanediamine]copper(II) perchlorate formed immediately (Hedwig, Love & Powell, 1970). Washed with 2-propanol, then ether. The single crystal used in X-ray diffraction analysis was obtained by recrystallization from 25% aqueous solution of 2-propanol-methanol, after slow evaporation of the solvent at room temperature.

Experimental data and structure-solution parameters together with the standard refinement procedures are listed in Table 1. The hydrogen atoms are located on a difference Fourier map and their temperature factors were initially assigned according to  $B_{\text{H}} = B_{\text{N}} + 1$ , where *n* is the atom to which the H atom is bonded (Lee, Lee, Juang & Chung, 1986).

**Discussion.** The atomic coordinates and temperature factors are listed in Table 2.\* A perspective view of the molecule with the numbering scheme and the deviations of the atoms from the least-squares plane through the basal atoms, N(1), N(2), N(3), N(4), are indicated in Fig. 1.

The coordination geometry about the copper ion is square pyramidal. The four donor N atoms of the tetraamine form a very slightly distorted plane and a perchlorate O atom occupies the axial position. The CuN<sub>4</sub> chromophore is planar to  $\pm 0.09 \text{ \AA}$  (Fig. 1). The equatorial Cu-N distances span a rather narrow range,

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43022 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Experimental data and structure-refinement parameters*

Crystal size (mm)	0.3 × 0.3 × 0.35
Diffractometer and data-collection technique used	$\omega$ -2 $\theta$ scan, four-circle diffractometer (Nonius CAD-4) with graphite monochromator
Scan width	2(0.7 + 0.35 tan $\theta$ )°
Number and $\theta$ range used for measuring lattice parameters	25 reflections with $11 < 2\theta < 20^\circ$
Absorption correction applied	Experimental absorption correction based on $\phi$ scan (North, Phillips & Mathews, 1968)
Max. and min. transmission factor	0.705, 0.606
Max. (sin $\theta$ )/ $\lambda$ in intensity measurement	0.6501 $\text{\AA}^{-1}$
Range of <i>hkl</i>	0, 0, 1 to 18, 20, 19
Standard reflections and intensity variation	082, 443, 506; < 3%
Number of reflections measured	3541
Number of unique reflections	2105 [ $I > 3\sigma(I)$ ]
Method used to solve structure	Patterson and Fourier methods
$\rho_{\text{max}}$ and $\rho_{\text{min}}$ heights in final difference Fourier synthesis	1.034 and 0.37 e $\text{\AA}^{-3}$
Parameters refined, nonhydrogen atoms	Coordinates, and anisotropic temperature factors (216 parameters)
hydrogen atoms	Isotropic temperature factors (108 parameters)
Atomic scattering factors, $f'$ and $f''$	<i>International Tables for X-ray Crystallography</i> (1968)
Number of reflections per parameter	10
$R(F)$ and $wR$ , $w = 1/\sigma_f^2$	0.069 and 0.068
<i>S</i>	3.80
$R_{\text{int}}$	0.032
Max., av. $\Delta/\sigma$	0.37, 0.005
Programs used	THUCP (Hsieh & Lee, 1985); XTAL83 (Stewart, Hall, Alden, Olthof-Hazekamp, Doherty, Pogoaga & Norden, 1983); ORTEPII (Johnson, 1976).
Computer	IBM PC-XT and CDC Cyber-840

Table 2. *Atomic positional and thermal parameters of non-hydrogen atoms*

$B_{\text{eq}} = \frac{4}{3} \sum_i \sum_j a_i \cdot a_j B_{ij}$ , where the  $a_i$ 's are the length vectors in direct space.

	Population	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}} (\text{\AA}^2)$
Cu	1.0	0.13146 (7)	0.05881 (6)	0.22397 (6)	2.9
Cl(1)	1.0	-0.1208 (2)	0.0267 (2)	0.2200 (2)	4.5 (1)
Cl(2)	1.0	0.4148 (3)	0.3121 (3)	0.4457 (2)	7.9 (2)
O(11)	1.0	-0.0415 (5)	0.0541 (6)	0.1722 (5)	6.5 (4)
O(12)	1.0	-0.0935 (8)	-0.0215 (9)	0.2885 (7)	13.1 (8)
O(13)	1.0	-0.1763 (10)	0.0876 (7)	0.2442 (10)	14.4 (9)
O(14)	1.0	-0.1758 (10)	-0.0282 (12)	0.1717 (8)	17.7 (12)
O(21)	1.0	0.4098 (1)	0.3307 (13)	0.5322 (8)	18.9 (13)
O(22)	1.0	0.3263 (8)	0.3068 (9)	0.4096 (7)	11.0 (7)
O(23)	1.0	0.4844 (8)	0.3523 (10)	0.4035 (8)	14.4 (10)
O(24)	1.0	0.4527 (14)	0.2282 (13)	0.4521 (12)	20.3 (16)
O(W)	0.5	0.3707 (12)	-0.0039 (11)	0.0212 (9)	6.5 (8)
N(1)	1.0	0.0971 (7)	0.0880 (7)	0.3490 (5)	5.7 (5)
N(2)	1.0	0.1238 (5)	0.1791 (5)	0.1909 (5)	3.9 (3)
N(3)	1.0	0.1766 (6)	0.0411 (6)	0.0981 (5)	4.4 (4)
N(4)	1.0	0.1320 (7)	-0.0621 (5)	0.2511 (6)	5.2 (4)
C(1)	1.0	0.1379 (9)	0.1644 (9)	0.3864 (7)	6.6 (6)
C(2)	1.0	0.1069 (10)	0.2377 (9)	0.3372 (8)	7.2 (7)
C(3)	1.0	0.1554 (8)	0.2445 (7)	0.2506 (8)	5.7 (6)
C(4)	1.0	0.1723 (8)	0.1899 (7)	0.1047 (7)	5.1 (5)
C(5)	1.0	0.1484 (8)	0.1159 (7)	0.0502 (6)	5.2 (5)
C(6)	1.0	0.1480 (11)	-0.0383 (8)	0.0542 (7)	7.2 (7)
C(7)	1.0	0.1736 (10)	-0.1100 (7)	0.1044 (9)	7.3 (7)
C(8)	1.0	0.1126 (9)	-0.1240 (7)	0.1853 (10)	6.9 (7)

2.012 (8)–2.031 (8) Å, which are typical for copper(II)–tetraamine complexes (Fawcett, Rudich, Toby, Lalancette, Potenza & Schugar, 1980). The Cu–O(apical) distance, 2.539 (7) Å, is within the range of 2.52 (2)–2.883 (2) Å reported (Tasker & Sklar, 1975). No other donor atoms are located within reasonable bonding distance of the Cu<sup>II</sup> atom. The two asymmetric nitrogens, N(2) and N(3), are of the same *R* or *S* configuration. The Cu-3,2,3-tet moiety has a pseudo-twofold axis through Cu and the center of the C(4)–C(5) bond in the central five-membered chelate ring. The conformation of the quadridentate ligand, 3,2,3-tet, is in its most stable planar form, with both six-membered chelate rings having a chair form and the five-membered ring having a *gauche* form. The chelate angles around Cu<sup>II</sup> are those expected for copper(II)–tetraamine complexes with an alternating sequence of five- and six-membered rings (Marongiu, Lingafelter & Paoletti, 1969).

In view of the above considerations, the arrangement of the 3,2,3-tet ligand in the title complex appears to be relatively strain-free. These structural features suggest that the N<sub>4</sub> ligand donor sets nearly match the symmetry properties of the Cu<sup>II</sup> orbitals and are consistent with the relatively low heat of formation and large stability constant of this Cu<sup>II</sup> complex (Bianchini, Fabbri, Paoletti & Lever, 1975).

A water molecule with half occupancy is situated among the perchlorate ions and [Cu(3,2,3-tet)]<sup>2+</sup>. The possible intra- and intermolecular hydrogen bonds among this water molecule, perchlorate ions, –NH and NH<sub>2</sub> groups are listed in Table 3 and indicated in Fig. 2. These hydrogen bonds stabilize the crystal lattice to a

large extent. Hence the crystal deteriorates as it is exposed to air and collapses after several days. The perchlorate ions suffer orientational disorder. This is manifested in the residual peaks around the perchlorate ions. The rather high *R* value is probably due to this effect.

Table 3. Bond lengths (Å) and angles (°)

Cu–N(1)	2.031 (8)	N(3)–C(6)	1.502 (16)
Cu–N(2)	2.012 (8)	C(6)–C(7)	1.437 (18)
Cu–N(3)	2.049 (7)	C(7)–C(8)	1.520 (20)
Cu–N(4)	1.998 (8)	C(8)–N(4)	1.447 (16)
Cu–O(11)	2.539 (7)	Cl(1)–O(11)	1.398 (8)
N(1)–C(1)	1.476 (17)	Cl(1)–O(12)	1.361 (12)
C(1)–C(2)	1.469 (19)	Cl(1)–O(13)	1.305 (13)
C(2)–C(3)	1.493 (18)	Cl(1)–O(14)	1.386 (16)
C(3)–N(2)	1.466 (14)	Cl(2)–O(21)	1.361 (13)
N(2)–C(4)	1.495 (13)	Cl(2)–O(22)	1.355 (11)
C(4)–C(5)	1.497 (16)	Cl(2)–O(23)	1.335 (14)
C(5)–N(3)	1.469 (14)	Cl(2)–O(24)	1.460 (21)
N(1)–Cu–N(2)	90.1 (4)	C(7)–C(8)–N(4)	111.2 (10)
N(2)–Cu–N(3)	85.1 (3)	C(8)–N(4)–Cu	122.1 (7)
N(3)–Cu–N(4)	93.3 (4)	O(11)–Cu–N(1)	94.5 (3)
N(4)–Cu–N(1)	91.8 (4)	O(11)–Cu–N(2)	84.3 (3)
N(1)–Cu–N(3)	173.3 (4)	O(11)–Cu–N(3)	89.6 (3)
N(2)–Cu–N(4)	176.2 (3)	O(11)–Cu–N(4)	92.2 (3)
Cu–N(1)–C(1)	118.0 (7)	O(11)–Cl(1)–O(12)	111.4 (6)
N(1)–C(1)–C(2)	111.3 (10)	O(11)–Cl(1)–O(13)	112.2 (7)
C(1)–C(2)–C(3)	112.5 (11)	O(11)–Cl(1)–O(14)	111.1 (6)
C(2)–C(3)–N(2)	111.4 (9)	O(12)–Cl(1)–O(13)	112.2 (8)
C(3)–N(2)–Cu	121.6 (6)	O(12)–Cl(1)–O(14)	101.5 (9)
Cu–N(2)–C(4)	108.2 (6)	O(13)–Cl(1)–O(14)	107.8 (9)
N(2)–C(4)–C(5)	107.4 (8)	O(21)–Cl(2)–O(22)	111.4 (8)
C(4)–C(5)–N(3)	108.7 (8)	O(21)–Cl(2)–O(23)	113.6 (10)
C(5)–N(3)–Cu	105.9 (6)	O(21)–Cl(2)–O(24)	99.1 (11)
Cu–N(3)–C(6)	117.4 (7)	O(22)–Cl(2)–O(23)	119.7 (8)
N(3)–C(6)–C(7)	112.5 (10)	O(22)–Cl(2)–O(24)	107.3 (10)
C(6)–C(7)–C(8)	114.7 (11)	O(23)–Cl(2)–O(24)	102.8 (10)

Hydrogen bonds	Lengths	Angles
N(1)–H(17)···O(W.4)*	3.00 (2) Å	130.9 (7)°
N(1)–H(18)···O(12)	3.33 (2)	132.0 (8)
N(2)–H(19)···O(11)	3.08 (1)	126.7 (6)
N(3)–H(20)···O(W)	3.04 (2)	152.9 (6)
N(4)–H(21)···O(14.8)*	2.98 (2)	150.4 (7)

\* The numbers 4 and 8 after the decimal point in the parentheses indicate the corresponding equivalent atoms respectively [symmetry codes: 4 for ( $\frac{1}{2}-x, -y, \frac{1}{2}+z$ ); 8 for ( $\frac{1}{2}+x, y, \frac{1}{2}-z$ ); *International Tables for Crystallography* (1983), Vol. A, p. 156].

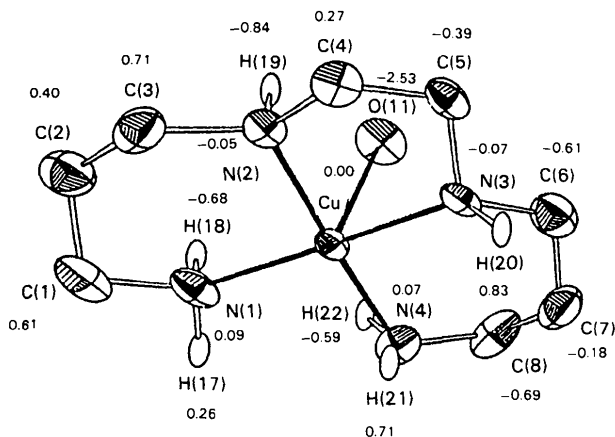


Fig. 1. Perspective view of the [Cu(3,2,3-tet)]<sup>2+</sup>, showing the atomic numbering scheme and displacements of atoms from least-squares plane of N(1), N(2), N(3) and N(4) (in Å). E.s.d.'s are around 0.01 Å. Thermal ellipsoids are depicted at the 50% probability level. H atoms on carbon atoms are not plotted, but the amide hydrogens, H(17)–H(22), are. These latter are plotted as plane ellipses. O(11) is the coordinated O atom of the perchlorate ion.

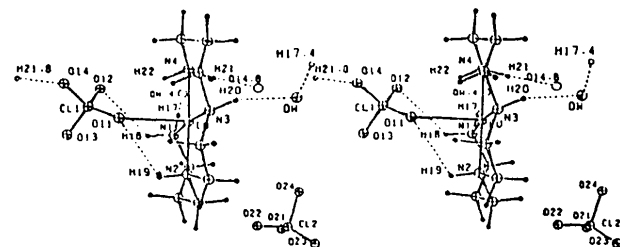


Fig. 2. Stereoview of [Cu(3,2,3-tet)(ClO<sub>4</sub>)]ClO<sub>4</sub>·0.5H<sub>2</sub>O, showing the coordination geometry around the copper(II) atom and the possible hydrogen bonds. The numbers 4 and 8 after the decimal points indicate the corresponding equivalent atoms respectively [symmetry codes: 4 for ( $\frac{1}{2}-x, -y, \frac{1}{2}+z$ ); 8 for ( $\frac{1}{2}+x, y, \frac{1}{2}-z$ )] *International Tables for Crystallography* (1983), Vol. A, p. 156].

The structures of some Cu<sup>II</sup> complexes of linear aliphatic tetraamines have been reported. A comparison of the selected structural data of these complexes with those of the title compound is given in Table 4. These structural data are expected to influence the kinetic properties of these complexes. This aspect is being taken up shortly.

Table 4. Selected structural data for Cu<sup>II</sup>-tetraamine complexes

Linear aliphatic tetraamines of the type H<sub>2</sub>N(CH<sub>2</sub>)<sub>l</sub>NH(CH<sub>2</sub>)<sub>m</sub>NH(CH<sub>2</sub>)<sub>n</sub>NH<sub>2</sub> are denoted by the symbol *l,m,n*-tet.

	Cu(2,2,2-tet)(SCN) <sub>2</sub>	Cu(2,3,2-tet)(ClO <sub>4</sub> ) <sub>2</sub>	Cu(3,2,3-tet)(ClO <sub>4</sub> ) <sub>2</sub>	Cu(3,3,3-tet)(ClO <sub>4</sub> ) <sub>2</sub>
Cu-N (Å)	2.008 (7)	2.016 (6)	2.012 (8)	2.02 (2)
distance	2.030 (5)	2.032 (6)	2.031 (8)	2.04 (2)
Cu-O (Å)	—*	2.667 (5)	2.539 (7)	
distance		2.527 (5)		
Chelate angle for 5-membered ring (°)	84.6 (3)	85.3 (2)	85.1 (3)	
Chelate angle for 6-membered ring (°)	84.3 (2)	85.3 (3)		
<i>trans</i> N-Cu-N (°)	154.0	176.4 (3)	173.3 (4)	159.4 (7)
Coordination geometry about Cu <sup>II</sup>	distorted square pyramidal	tetragonally distorted octahedral	square pyramidal	tetragonally distorted octahedral
Conformation of chelate rings	<i>gauche, gauche, gauche</i>	<i>gauche, chair, gauche</i>	<i>chair, gauche, chair</i>	<i>chair, chair, chair</i>
References	Marongiu, Lingafelter & Paoletti (1969)	Fawcett, Rudich, Toby, Lalancette, Potenza & Schugar (1980)	Present work	Fawcett, Rudich, Toby, Lalancette, Potenza & Schugar (1980)

\* The Cu-S bond distance for this complex is 2.607 (2) Å.

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## The Chain Polymeric Structure of Lead(II) Crotonate

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**Abstract.** *catena-μ<sub>3</sub>-(Crotonato-μ-O,μ-O')-(crotonato-O,O')*-lead(II), [Pb(C<sub>4</sub>H<sub>5</sub>O<sub>2</sub>)<sub>2</sub>], *M<sub>r</sub>* = 377.4, triclinic, *P* $\bar{1}$ , *a* = 5.1930 (4), *b* = 7.3386 (6), *c* = 13.1528 (12) Å, *α* = 98.797 (8), *β* = 91.668 (8), *γ* = 94.059 (8)°, *V* = 493.70 Å<sup>3</sup>, *Z* = 2 monomeric units, *D<sub>x</sub>* = 2.538 Mg m<sup>-3</sup>, *λ*(Mo *Kα*) = 0.71073 Å, *μ* = 17.2 mm<sup>-1</sup>, *F*(000) = 344, *T* = 295 K, *R* = 0.036 for

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