N—H 0.70 (4)–0.95 (4) and O—H 0.55 (5)–1.11 (4) Å. For compound (II), some H atoms attached to N and O atoms could not be located. Those which could be positioned were included as fixed isotropic scatterers. No absorption correction was applied for (I) because ψ scans of three reflections indicated a transmission range of less than 5%. As a result of the generally weak reflection intensities for (II), the θ range for the unit cell is unusually low which may cause systematic errors in the cell parameters.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structures: *MITHRIL* (Gilmore, 1983); program(s) used to refine structures: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: BK1220). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aqua[bis(3-aminopropyl)amine-*N*,*N'*,*N''*](glutarato-*O*)copper(II) Tetrahydrate

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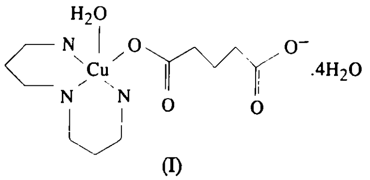
Abstract

The title compound, $[Cu(C_5H_6O_4)(C_6H_{17}N_3)(H_2O)]$.$4H_2O$, consists of monomeric complex molecules in which the central Cu atom is coordinated by three N atoms from the amine ligand and by one glutarate and one water O atom. The coordination polyhedron is square pyramidal.

Comment

Only a few structures containing copper(II) and glutarate ions have been reported. The coordination of the glutarate anion in the title compound, (I), is of particular interest because glutarate can form both polymeric (Pajunen & Pajunen, 1977) and dimeric (Pajunen & Pajunen, 1979) complexes.



(I)

The coordination polyhedron of the Cu atom is a square pyramid whose base is formed by three N atoms from the amine ligand and the O1 atom of the glutarate ion (Fig. 1). The O5 atom of a coordinated water molecule occupies the apex of the pyramid at a distance of 2.482 (2) Å. The Cu atom also has two long contacts to the water O6 and glutarate O2 atoms. Even though these distances are long [3.061 (3) and 3.102 (2) Å, respectively], the atoms must have some interaction with the Cu atom since the Cu atom lies 0.013 (1) Å from the mean plane defined by the amine N and O1 atoms towards the O6 atom. This is in contrast to the situation found in most square-pyramidal structures, where the Cu atom is lifted *ca* 0.2 Å towards the apex of the pyramid (Hathaway & Billing, 1970). An interesting feature of the structure is the dissimilar conformations of the two six-membered chelate rings. The ring including the N2 and N3 atoms adopts a chair conformation, while that including the N1 and N2 atoms is best described as having a half-boat conformation. The terminal Cu—N1 and Cu—N3 distances are shorter than the Cu—N2 distance, a common feature of bis(3-aminopropyl)amine complexes (Einstein & Willis, 1981). An extensive hydrogen-bonding network is present in the crystal and involves all the amine and water H atoms (Fig. 2 and Table 3).

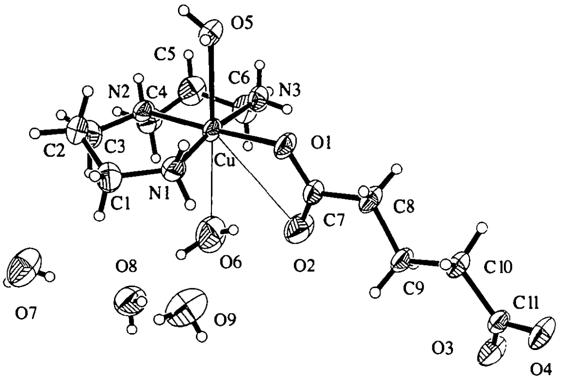


Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids. Thin lines indicate weak Cu···O hydrogen bonds.

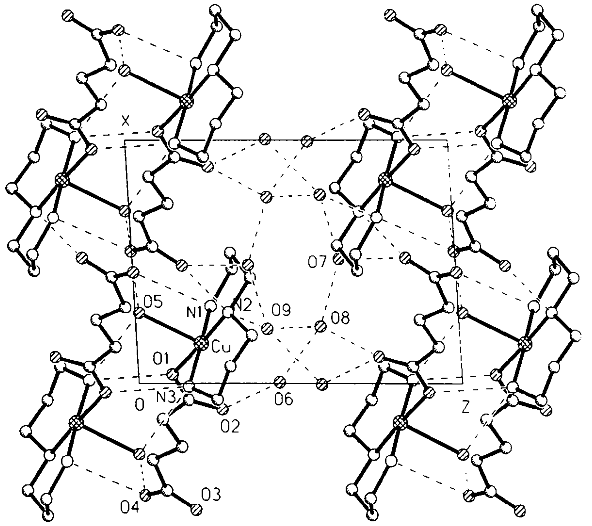


Fig. 2. Packing diagram viewed down the *b* axis showing the hydrogen bonds.

Experimental

Blue crystals of the title compound were prepared from $CuCO_3$, glutaric acid and bis(3-aminopropyl)amine according to Pajunen & Näsäkkälä (1977), and recrystallized from ethanol. The density D_m was measured by flotation in a mixture of toluene and methyl iodide.

Crystal data

$[Cu(C_5H_6O_4)(C_6H_{17}N_3)(H_2O)].4H_2O$
$M_r = 414.94$
Triclinic
$P\overline{1}$
$a = 8.3934\ (8)$ Å
$b = 10.0093\ (13)$ Å
$c = 11.9537\ (11)$ Å
$\alpha = 71.137\ (8)^\circ$
$\beta = 94.378\ (8)^\circ$
$\gamma = 93.974\ (9)^\circ$
$V = 946.5\ (2)$ Å^3
$Z = 2$
$D_x = 1.456$ Mg m^{-3}
$D_m = 1.46\ (2)$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 6.73\text{–}10.37^\circ$
$\mu = 1.200$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.30 × 0.25 × 0.20 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4842 measured reflections
4842 independent reflections
3718 observed reflections $[I > 2\sigma(I)]$
$\theta_{\max} = 29.97^\circ$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = 0 \rightarrow 16$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0335$
$wR(F^2) = 0.0889$
$S = 1.038$
4842 reflections
219 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.2109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.32$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu	0.16323 (3)	0.38586 (3)	0.19975 (2)	0.02948 (8)
O1	0.0367 (2)	0.27593 (15)	0.10467 (13)	0.0348 (3)
O2	−0.1087 (3)	0.1517 (2)	0.2524 (2)	0.0620 (6)
O3	−0.5159 (2)	−0.2351 (2)	0.15267 (15)	0.0473 (4)
O4	−0.4560 (2)	−0.2923 (2)	−0.00175 (15)	0.0416 (4)
O5	0.2896 (2)	0.5200 (2)	0.01253 (14)	0.0381 (3)
O6	0.0048 (3)	0.2493 (2)	0.4348 (2)	0.0789 (7)
O7	0.5124 (3)	0.1761 (3)	0.6409 (2)	0.0759 (7)
O8	0.2326 (3)	0.0711 (2)	0.5715 (2)	0.0654 (6)
O9	0.2229 (3)	−0.0652 (2)	0.4060 (2)	0.0784 (7)
N1	0.3170 (2)	0.2308 (2)	0.2347 (2)	0.0352 (4)
N2	0.2919 (2)	0.5052 (2)	0.28758 (15)	0.0333 (4)
N3	−0.0132 (2)	0.5203 (2)	0.1533 (2)	0.0365 (4)
C1	0.4700 (3)	0.2236 (3)	0.3065 (2)	0.0450 (5)
C2	0.5366 (3)	0.3680 (3)	0.3043 (2)	0.0474 (6)
C3	0.4338 (3)	0.4411 (3)	0.3621 (2)	0.0471 (6)
C4	0.1962 (3)	0.5662 (3)	0.3574 (2)	0.0435 (5)
C5	0.0687 (3)	0.6611 (3)	0.2844 (2)	0.0505 (6)

C6	−0.0677 (3)	0.5829 (3)	0.2388 (2)	0.0460 (5)
C7	−0.0690 (2)	0.1813 (2)	0.1497 (2)	0.0315 (4)
C8	−0.1495 (3)	0.1076 (2)	0.0663 (2)	0.0360 (5)
C9	−0.2596 (2)	−0.0213 (2)	0.1202 (2)	0.0332 (4)
C10	−0.3127 (2)	−0.0943 (2)	0.0292 (2)	0.0323 (4)
C11	−0.4368 (2)	−0.2166 (2)	0.0652 (2)	0.0293 (4)

Table 2. *Selected geometric parameters (Å, °)*

Cu—N3	1.995 (2)	N2—C4	1.488 (3)
Cu—N1	2.006 (2)	N3—C6	1.474 (3)
Cu—O1	2.023 (1)	C1—C2	1.506 (3)
Cu—N2	2.038 (2)	C2—C3	1.504 (4)
Cu—O5	2.482 (2)	C4—C5	1.507 (4)
O1—C7	1.265 (2)	C5—C6	1.509 (3)
O2—C7	1.233 (3)	C7—C8	1.516 (3)
O3—C11	1.237 (3)	C8—C9	1.524 (3)
O4—C11	1.264 (2)	C9—C10	1.521 (3)
N1—C1	1.481 (3)	C10—C11	1.523 (3)
N2—C3	1.482 (3)		
N3—Cu—O1	86.61 (7)	C3—C2—C1	113.4 (2)
N1—Cu—O1	84.15 (7)	N2—C3—C2	114.3 (2)
N3—Cu—N2	92.06 (7)	N2—C4—C5	113.9 (2)
N1—Cu—N2	97.22 (7)	C4—C5—C6	113.5 (2)
N3—Cu—O5	88.90 (7)	N3—C6—C5	111.0 (2)
N1—Cu—O5	91.89 (6)	O2—C7—O1	123.8 (2)
O1—Cu—O5	86.19 (5)	O2—C7—C8	121.3 (2)
N2—Cu—O5	91.12 (6)	O1—C7—C8	115.0 (2)
C7—O1—Cu	122.04 (13)	C7—C8—C9	117.1 (2)
C1—N1—Cu	125.06 (14)	C10—C9—C8	110.6 (2)
C3—N2—C4	108.7 (2)	C9—C10—C11	117.1 (2)
C3—N2—Cu	116.80 (14)	O3—C11—O4	123.5 (2)
C4—N2—Cu	115.09 (13)	O3—C11—C10	120.8 (2)
C6—N3—Cu	120.38 (15)	O4—C11—C10	115.7 (2)
N1—C1—C2	111.9 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	2.12	2.964 (2)	164
N1—H2···O9	2.29	3.098 (3)	152
N2—H3···O3^{ii}	2.25	3.011 (2)	159
N3—H4···O5^{iii}	2.34	3.026 (2)	148
N3—H5···O1^{iii}	2.33	3.099 (2)	149
O5—H51···O4^{ii}	1.91	2.723 (2)	173
O5—H52···O4^{i}	2.10	2.796 (2)	173
O6—H61···O2	2.00	2.747 (3)	173
O6—H62···O8	1.80	2.760 (3)	172
O7—H71···O3^{iv}	2.01	2.714 (3)	157
O7—H72···O8	2.03	2.689 (3)	176
O8—H81···O2^{iv}	2.00	2.732 (3)	165
O8—H82···O9	1.92	2.736 (3)	167
O9—H91···O6^{iv}	2.17	2.910 (3)	163
O9—H92···O7^{v}	1.97	2.724 (3)	148

Symmetry codes: (i) $-x, -y, -z$; (ii) $1 + x, 1 + y, z$; (iii) $-x, 1 - y, -z$; (iv) $-x, -y, 1 - z$; (v) $1 - x, -y, 1 - z$.

The H atoms involved in the hydrogen bonds were located from a difference synthesis; all other H atoms were included in calculated positions with fixed displacement parameters (1.2 times the displacement parameter of the host atom).

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: MU1258). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Bimetallic Complex of Ethylenediaminetetraacetic Acid: $Ag[Cu(C_{10}H_{13}N_2O_8)(H_2O)].H_2O$

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Abstract

The structure of silver aqua(hydrogen ethylenediaminetetraacetato)cuprate(II) monohydrate, $Ag[Cu(C_{10}H_{13}N_2O_8)(H_2O)].H_2O$, is described. This molecular compound is only the second complex of ethylenediaminetetraacetic acid (edta) with silver to be reported. It crystallizes as a three-dimensional network which can be described as stacked layers connected by Cu^{2+} ions.

Comment

This work is a continuation of the study of complexes used as molecular precursors of oxide and sulfide materials (Brouca-Cabarrecq, Galy & Trombe, 1991). Carboxylate ligands are known to be good complexing