

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	Occupancy	x	y	z	B _{eq}
Ni	1.0	0.24397 (14)	0.80566 (7)	0.22878 (5)	2.82 (4)
Cl(1)	1.0	0.7905 (3)	0.65783 (19)	0.18758 (17)	4.80 (11)
Cl(2)	1.0	0.7010 (4)	0.0539 (2)	0.07441 (17)	5.17 (13)
O(1)	1.0	0.7069 (12)	0.7291 (6)	0.2195 (10)	13.8 (10)
O(2)	1.0	0.6908 (13)	0.5845 (6)	0.1929 (10)	12.5 (9)
O(3)	1.0	0.817 (2)	0.6730 (17)	0.1146 (7)	21.7 (17)
O(4)	1.0	0.9302 (11)	0.6422 (7)	0.2275 (7)	9.4 (6)
O(5)	0.85	0.743 (2)	-0.0367 (7)	0.0833 (9)	11.1 (10)
O(5')	0.15	0.692 (6)	0.007 (3)	0.149 (3)	4.1 (9)
O(6)	0.70	0.713 (4)	0.0727 (16)	-0.0015 (9)	14.7 (18)
O(6')	0.30	0.596 (4)	0.028 (3)	0.0201 (19)	9 (2)
O(7)	0.70	0.567 (3)	0.095 (2)	0.0970 (17)	16.0 (19)
O(7')	0.30	0.600 (6)	0.044 (3)	0.137 (3)	8.3 (11)
O(8)	1.0	0.8190 (14)	0.1081 (8)	0.1101 (9)	11.8 (8)
N(1)	1.0	0.2128 (9)	0.8053 (5)	0.3437 (4)	3.7 (3)
N(2)	1.0	0.1431 (9)	0.9221 (5)	0.2227 (5)	3.6 (3)
N(3)	1.0	0.2691 (9)	0.8115 (6)	0.1137 (4)	3.6 (3)
N(4)	1.0	0.3555 (9)	0.6915 (5)	0.2345 (4)	4.3 (3)
C(1)	1.0	0.3018 (19)	0.8563 (10)	0.4795 (6)	7.9 (8)
C(2)	1.0	0.31396 (14)	0.8673 (8)	0.3909 (6)	4.8 (5)
C(3)	1.0	0.4803 (18)	0.8570 (12)	0.3627 (9)	7.8 (9)
C(4)	1.0	0.0387 (12)	0.8128 (8)	0.3627 (6)	4.2 (4)
C(5)	1.0	-0.0364 (12)	0.8984 (8)	0.3370 (7)	4.9 (5)
C(6)	1.0	-0.0256 (12)	0.9166 (7)	0.2476 (6)	4.3 (4)
C(7)	1.0	0.1594 (14)	0.9618 (7)	0.1409 (6)	5.0 (5)
C(8)	1.0	0.1531 (15)	0.8790 (9)	0.0846 (6)	5.6 (6)
C(9)	1.0	0.4282 (15)	0.8428 (10)	0.0963 (7)	5.9 (6)
C(10)	1.0	0.2395 (18)	0.7258 (8)	0.0700 (6)	6.1 (6)
C(11)	1.0	0.3341 (15)	0.6454 (8)	0.0947 (7)	5.8 (6)
C(12)	1.0	0.3070 (13)	0.6176 (7)	0.1765 (8)	5.3 (5)

Table 2. Selected geometric parameters (Å, °)

Ni—N(1)	1.951 (6)	N(3)—C(10)	1.48 (1)
Ni—N(2)	1.915 (7)	N(4)—C(12)	1.52 (1)
Ni—N(3)	1.951 (6)	C(1)—C(2)	1.50 (2)
Ni—N(4)	1.927 (8)	C(2)—C(3)	1.50 (2)
N(1)—C(2)	1.48 (1)	C(4)—C(5)	1.47 (2)
N(1)—C(4)	1.52 (1)	C(5)—C(6)	1.53 (2)
N(2)—C(6)	1.49 (1)	C(7)—C(8)	1.54 (2)
N(2)—C(7)	1.50 (1)	C(10)—C(11)	1.49 (2)
N(3)—C(8)	1.48 (2)	C(11)—C(12)	1.45 (2)
N(3)—C(9)	1.46 (2)		
N(1)—Ni—N(2)	89.7 (3)	C(8)—N(3)—C(9)	109.9 (9)
N(1)—Ni—N(3)	177.2 (3)	C(8)—N(3)—C(10)	107.0 (8)
N(1)—Ni—N(4)	90.8 (3)	C(9)—N(3)—C(10)	109.0 (9)
N(2)—Ni—N(3)	87.5 (3)	Ni—N(4)—C(12)	117.1 (6)
N(2)—Ni—N(4)	177.1 (3)	N(1)—C(2)—C(1)	115 (1)
N(3)—Ni—N(4)	92.0 (3)	N(1)—C(2)—C(3)	108.3 (9)
Ni—N(1)—C(2)	116.8 (6)	C(1)—C(2)—C(3)	112 (1)
Ni—N(1)—C(4)	109.9 (5)	N(1)—C(4)—C(5)	114.9 (8)
C(2)—N(1)—C(4)	114.0 (8)	C(4)—C(5)—C(6)	114.3 (9)
Ni—N(2)—C(6)	111.4 (6)	N(2)—C(6)—C(5)	110.0 (8)
Ni—N(2)—C(7)	110.7 (6)	N(2)—C(7)—C(8)	104.8 (8)
C(6)—N(2)—C(7)	111.5 (7)	N(3)—C(8)—C(7)	107.5 (8)
Ni—N(3)—C(8)	106.6 (6)	N(3)—C(10)—C(11)	116.4 (9)
Ni—N(3)—C(9)	108.3 (6)	C(10)—C(11)—C(12)	113.7 (9)
N—N(3)—C(10)	115.9 (6)	N(4)—C(12)—C(11)	111.4 (8)

The structure was solved by direct and Fourier methods and refined by full-matrix least-squares techniques. H atoms were located by difference Fourier methods and theoretical calculation. *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71637 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1060]

References

- Gabe, E. J., Le Page, Y., White, P. S. & Lee, F. L. (1986). *Acta Cryst.* **A43**, C-294.
 Hinz, F. P. & Margerum, D. W. (1974). *Inorg. Chem.* **13**, 2941–2949.
 Lu, T.-H., Chung, C.-S. & Ashida, T. (1991). *J. Chin. Chem. Soc. (Taipei)*, **38**, 147–153.
 Lu, T.-H., Shan, H.-C., Chao, M.-S. & Chung, C.-S. (1987). *Acta Cryst.* **C43**, 207–209.
 North, A. C. T., Phillips, D. C. & Mathews, F. W. (1968). *Acta Cryst.* **A24**, 351–359.

Acta Cryst. (1994). **C50**, 518–520

[N,N'-Bis(3-aminopropyl)-trans-1,2-cyclohexanediamine-N,N',N'',N''']di(perchlorato-O)copper(II)

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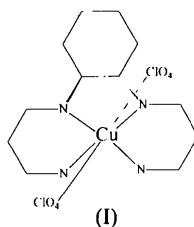
Abstract

The Cu^{II} ion of the title complex, [Cu(ClO₄)₂·(C₁₂H₂₈N₄)], is six-coordinated with four N atoms of the tetradentate ligand on the equatorial plane and two perchlorato O atoms in axial positions. The two asymmetric donor N atoms have the same *R* or *S* configuration. The two terminal six-membered chelate rings are in stable chair forms and the central five-membered chelate ring is in a stable *gauche* form. The cyclohexane ring on the central chelate ring is in a stable chair form. The hydrogen bonds between the NH and NH₂ groups and the perchlorate O atoms help stabilize the crystal structure.

Comment

We have reported previously the crystal structure of [N,N'-bis(3-aminopropyl)-1,2-ethanediamine]per-

chloratocopper(II) perchlorate hemihydrate, $[\text{Cu}(\text{ClO}_4)(\text{C}_8\text{H}_{22}\text{N}_4)]\text{ClO}_4 \cdot 0.5\text{H}_2\text{O}$ (Lee, Lee, Hong, Hsieh, Wu & Chung, 1986). In order to study the steric effect of the cyclohexane ring attached to the central five-membered chelating ring on the structure of the copper(II) complex, we have prepared and studied the structure of the title complex (I).



trans-1,2-Cyclohexanediamine (11.4 g in 100 ml propanol) was added slowly to an ethanol solution of acrylamide (14.2 g in 100 ml ethanol). The resulting solution was refluxed for 3 h and then placed in a refrigerator for one week. White crystals of *N,N'*-bis(β -carbamoyl ethyl)-*trans*-1,2-diaminocyclohexane precipitated slowly from the solution. These white crystals were filtered off and dried in a vacuum. The product was added to an ice-cooled dry tetrahydrofuran solution of excess LiAlH_4 and refluxed for 24 h. Excess water and a solution of NaOH were added to this solution whereupon white solid $\text{Al}(\text{OH})_3$ was rapidly deposited and filtered off. The filtrate was evaporated to dryness, and the ligand, *N,N'*-bis(3-aminopropyl)-*trans*-1,2-cyclohexanediamine, was distilled at *ca* 426 K under vacuum (3.8 mm Hg). A solution of $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (3.7 g, 0.01 mol) in methanol (80 ml) was added dropwise to a solution of the ligand (2.28 g, 0.01 mol) in ethanol (80 ml). The solution changed colour rapidly to reddish blue and was stirred for 3 h until a purple solid formed. The single crystal was obtained by slow evaporation of an aqueous solution at room temperature.

The coordination geometry about the Cu^{II} ion is tetragonally distorted octahedral with four N donor atoms of the tetradentate ligand in equatorial positions and two perchlorate O atoms in axial positions. The four equatorial N atoms are coplanar within 0.02 Å. The Cu—N distances span a narrow range, 2.018 (2) to 2.046 (2) Å, and are comparable to the average Cu—N distance of 2.03 (3) Å for the Cu^{II} macrocyclic complexes (Lu, Chung & Ashida, 1991). The two Cu—O(apical) distances fall within the range [2.520 (2)–2.883 (2) Å] reported by Tasker & Sklar (1975). Comparing the structure of the title complex with that of $[\text{Cu}(\text{ClO}_4)(\text{C}_8\text{H}_{22}\text{N}_4)]\text{ClO}_4 \cdot 0.5\text{H}_2\text{O}$ (Lee *et al.*, 1986), we find that both complexes have the same *R* or *S* configuration of the asymmetric N atoms. In both structures, the central five-membered ring is in a stable *gauche* form and the

two terminal six-membered rings are in stable chair forms. Hydrogen bonds between the NH and NH_2 groups and the perchlorate O atoms help stabilize the crystal structure.

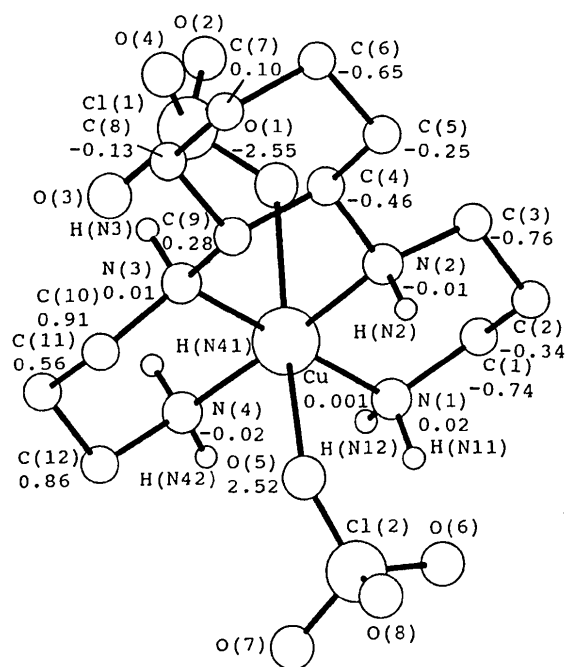


Fig. 1. A perspective view of the title molecule with the atom-numbering scheme, excluding the H atoms attached to the C atoms. Displacements from the best plane formed by the four N atoms coordinated to the Cu^{II} ion are also indicated.

Experimental

Crystal data

$[\text{Cu}(\text{ClO}_4)_2(\text{C}_{12}\text{H}_{28}\text{N}_4)]$

$M_r = 490.828$

Triclinic

$P\bar{1}$

$a = 8.743$ (2) Å

$b = 9.656$ (1) Å

$c = 12.088$ (3) Å

$\alpha = 89.66$ (2)°

$\beta = 77.03$ (2)°

$\gamma = 73.28$ (1)°

$V = 950.5$ (3) Å³

$Z = 2$

$D_x = 1.715$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.7107$ Å

Cell parameters from 25 reflections

$\theta = 5.32$ – 15.35 °

$\mu = 1.48$ mm⁻¹

$T = 298$ (3) K

Parallelepiped

$0.53 \times 0.24 \times 0.19$ mm

Data collection

Nonius CAD-4 diffractometer

$\theta/2\theta$ scans

Absorption correction:

empirical

$T_{\text{min}} = 0.968$, $T_{\text{max}} =$

0.984

5763 measured reflections

5522 independent reflections

4295 observed reflections

$[I \geq 2.5\sigma(I)]$

$R_{\text{int}} = 0.011$

$\theta_{\text{max}} = 29.9$ °

$h = -11 \rightarrow 12$

$k = 0 \rightarrow 13$

$l = -16 \rightarrow 16$

3 standard reflections

frequency: 60 min

intensity variation: $\pm 0.8\%$

Refinement

Refinement on F

R = 0.032

wR = 0.032

S = 0.53

4295 reflections

273 parameters

Only H-atom U's refined

Unit weights applied

(Δ/σ)_{max} = 0.004

$$\Delta\rho_{\max} = 0.48 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$$

Extinction correction:

Zachariasen (1967)

Extinction coefficient:

0.42 (1) (length in mm)

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Cu—O(1)—Cl(1)	124.7 (1)	N(3)—C(9)—C(8)	113.0 (2)
Cu—O(5)—Cl(2)	132.3 (1)	C(4)—C(9)—C(8)	111.4 (2)
Cu—N(1)—C(1)	118.7 (2)	N(3)—C(10)—C(11)	112.2 (2)
Cu—N(2)—C(3)	117.7 (1)	C(10)—C(11)—C(12)	114.5 (2)
Cu—N(2)—C(4)	107.1 (1)	N(4)—C(12)—C(11)	111.8 (2)
C(3)—N(2)—C(4)	112.5 (2)		

The structure was solved by direct and Fourier methods and refined by full-matrix least-squares techniques. H atoms were located by difference Fourier methods. *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j \cdot \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B _{eq}
Cu	0.89377 (3)	0.74711 (3)	0.25802 (2)	1.817 (9)
Cl(1)	0.78292 (7)	0.69847 (7)	-0.00307 (5)	2.65 (2)
Cl(2)	0.96878 (7)	0.77767 (7)	0.54090 (5)	2.89 (2)
O(1)	0.9136 (2)	0.7143 (3)	0.04523 (17)	3.86 (10)
O(2)	0.8445 (3)	0.6551 (3)	-0.12043 (16)	4.64 (12)
O(3)	0.7139 (4)	0.5950 (3)	0.0559 (2)	6.15 (16)
O(4)	0.6601 (3)	0.8353 (3)	0.0107 (2)	5.61 (12)
O(5)	0.8632 (3)	0.8219 (3)	0.46432 (17)	4.15 (10)
O(6)	1.1357 (3)	0.7390 (3)	0.4798 (2)	6.01 (14)
O(7)	0.9337 (4)	0.6560 (3)	0.5967 (3)	6.81 (16)
O(8)	0.9342 (4)	0.8913 (4)	0.6237 (2)	6.89 (16)
N(1)	1.1335 (2)	0.6360 (2)	0.23508 (19)	2.77 (8)
N(2)	0.9379 (2)	0.94103 (19)	0.22683 (15)	1.82 (6)
N(3)	0.6518 (2)	0.86429 (19)	0.28348 (15)	1.79 (6)
N(4)	0.8347 (3)	0.5603 (2)	0.28752 (19)	2.70 (8)
C(1)	1.2550 (3)	0.6799 (3)	0.1471 (2)	2.91 (9)
C(2)	1.2429 (3)	0.8371 (3)	0.1642 (2)	2.93 (10)
C(3)	1.0890 (3)	0.9414 (3)	0.1410 (2)	2.75 (10)
C(4)	0.7869 (3)	1.0412 (2)	0.20154 (17)	1.84 (8)
C(5)	0.7830 (3)	1.2002 (3)	0.2010 (2)	2.67 (10)
C(6)	0.6209 (3)	1.2961 (3)	0.1816 (2)	3.09 (11)
C(7)	0.4764 (3)	1.2768 (3)	0.2695 (3)	3.27 (11)
C(8)	0.4790 (3)	1.1184 (3)	0.2688 (2)	2.91 (10)
C(9)	0.6407 (3)	1.0210 (2)	0.28856 (17)	1.84 (7)
C(10)	0.5369 (3)	0.8264 (3)	0.3808 (2)	2.38 (9)
C(11)	0.5381 (3)	0.6697 (3)	0.3702 (2)	2.82 (10)
C(12)	0.6945 (3)	0.5610 (3)	0.3830 (2)	2.77 (10)

Table 2. Selected geometric parameters (Å, °)

Cu—O(1)	2.555 (2)	N(4)—C(12)	1.481 (3)
Cu—O(5)	2.534 (2)	C(1)—C(2)	1.503 (4)
Cu—N(1)	2.018 (2)	C(2)—C(3)	1.513 (4)
Cu—N(2)	2.035 (2)	C(4)—C(5)	1.526 (3)
Cu—N(3)	2.046 (2)	C(4)—C(9)	1.522 (3)
Cu—N(4)	2.024 (2)	C(5)—C(6)	1.521 (4)
N(1)—C(1)	1.479 (3)	C(6)—C(7)	1.513 (4)
N(2)—C(3)	1.487 (3)	C(7)—C(8)	1.523 (4)
N(2)—C(4)	1.488 (3)	C(8)—C(9)	1.525 (3)
N(3)—C(9)	1.489 (3)	C(10)—C(11)	1.516 (3)
N(3)—C(10)	1.484 (3)	C(11)—C(12)	1.504 (4)
O(1)—Cu—O(5)	170.98 (8)	Cu—N(3)—C(9)	108.2 (1)
O(1)—Cu—N(1)	90.36 (8)	Cu—N(3)—C(10)	116.4 (1)
O(1)—Cu—N(2)	87.40 (7)	C(9)—N(3)—C(10)	112.4 (2)
O(1)—Cu—N(3)	90.56 (7)	Cu—N(4)—C(12)	118.3 (2)
O(1)—Cu—N(4)	91.54 (8)	N(1)—C(1)—C(2)	111.8 (2)
O(5)—Cu—N(1)	93.77 (8)	C(1)—C(2)—C(3)	114.4 (2)
O(5)—Cu—N(2)	84.32 (8)	N(2)—C(3)—C(2)	112.9 (2)
O(5)—Cu—N(3)	85.14 (7)	N(2)—C(4)—C(5)	114.5 (2)
O(5)—Cu—N(4)	96.50 (9)	N(2)—C(4)—C(9)	107.4 (2)
N(1)—Cu—N(2)	94.01 (8)	C(5)—C(4)—C(9)	110.7 (2)
N(1)—Cu—N(3)	178.45 (8)	C(4)—C(5)—C(6)	111.2 (2)
N(1)—Cu—N(4)	89.63 (9)	C(5)—C(6)—C(7)	111.5 (2)
N(2)—Cu—N(3)	84.79 (7)	C(6)—C(7)—C(8)	110.3 (2)
N(2)—Cu—N(4)	176.21 (8)	C(7)—C(8)—C(9)	111.1 (2)
N(3)—Cu—N(4)	91.59 (8)	N(3)—C(9)—C(4)	107.7 (2)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71649 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1069]

References

- Gabe, E. J., Le Page, Y., White, P. S. & Lee, F. L. (1986). *Acta Cryst.* **A43**, C-294.
 Lee, T.-Y., Lee, T.-J., Hong, C.-Y., Hsieh, M.-Y., Wu, D.-T. & Chung, C.-S. (1986). *Acta Cryst.* **C42**, 1316–1319.
 Lu, T.-H., Chung, C.-S. & Ashida, T. (1991). *J. Chin. Chem. Soc. (Taipei)*, **38**, 147–153.
 Tasker, P. A. & Sklar, L. J. (1975). *J. Cryst. Mol. Struct.* **5**, 329–344.
 Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

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Benzenethiolato(triphenylphosphine)gold(I)

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Abstract

The crystal structure of [Au(C₆H₅S)(C₁₈H₁₅P)] contains two Au^I centers; each Au^I is almost linearly coordinated [P—Au—S bond angles of 179.0 (1) and

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