Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1174). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diperchlorato[(2RS,5SR,9RS,12RS)-2,5,9,12-tetraazatridecane]copper(II)

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Abstract

The Cu^{II} ion in the title compound, [Cu($C_9H_{24}N_4$)(ClO_4)$_2$], is six-coordinate in a distorted square-bipyramidal environment; four tetraamine N atoms are equatorial and two O atoms, one from each perchlorate ion, are axial. The quadridentate ligand is in its stable planar conformation with the central six-membered chelate ring in a chair form and both terminal five-membered rings in *gauche* forms. The four chiral N-atom centres have configurations 2RS, 5SR, 9RS and 12RS.

Comment

The Cu^{II} complexes of aliphatic tetraamines have been studied extensively because of their diverse stereochemistry. We describe here the crystal structure of the title compound, diperchlorato(2,5,9,12-tetraazatridecane)copper(II), [Cu(α,ω-N-Me$_2$-2,3,2-tet)(ClO_4)$_2$], (I) (where tet = tetraazatridecane).

(I)
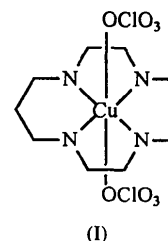
The coordination geometry around the Cu^{II} ion is slightly distorted square bipyramidal with the four tetraamine N atoms equatorial and the two O atoms, one from each perchlorate ion, axial. The quadridentate ligand is in its stable planar conformation with the central six-membered chelate ring in a chair form and both five-membered rings in *gauche* forms. The arrangement of the tetraamine ligand in the title complex appears to be relatively free of strain. The configurations of the four chiral N-atom centres are 2RS, 5SR, 9RS and 12RS. The hydrogen bonds between the tetraamine and the perchlorate ions, O(4)$\cdots$H(N1)—N(1) [3.05(1) Å], O(4)$\cdots$H(N2)—N(2) [3.12(1) Å] and O(8)$\cdots$H(N4)—N(4) [3.17(1) Å], stabilize the crystal structure.
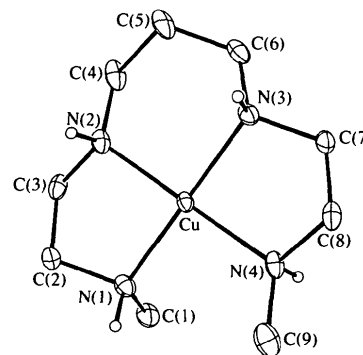

Fig. 1. A perspective view of the molecule with the atom-numbering scheme, excluding the perchlorate ions and the H atoms attached to C atoms. The displacement ellipsoids are drawn at the 50% probability level (Sheldrick, 1987).

The crystal structure of the title complex is almost the same as that of [Cu(2,3,2-tet)(ClO_4)$_2$] (Fawcett *et al.*, 1980). The stability constant of (I) is smaller than that of Cu(2,3,2-tet)$^{2+}$. In addition, [Cu(α,ω-N-Me$_2$-2,2,2-tet)]$^{2+}$ is more stable than [(Cu(2,2,2-tet)]$^{2+}$, but [Cu(α,ω-N-Me$_2$-2,3,2-tet)]$^{2+}$ is less stable than [Cu(2,3,2-tet)]$^{2+}$ (Clay, Corr, Micheloni & Paoletti, 1985). The effects of the two terminal N-Me groups in [Cu(α,ω-N-Me$_2$-2,3,2-tet)]$^{2+}$ and [Cu(α,ω-N-Me$_2$-2,2,2-tet)]$^{2+}$ are completely different from each other.

Experimental

The ligand, 2,5,9,12-tetraazatridecane (α,ω-N-Me$_2$-2,3,2-tet) was prepared according to the procedure of Clay, Corr, Micheloni & Paoletti (1985). The aqueous solution of its tetrahydrochloride salt was passed through an anion-exchange column (Amberlite IR400, OH$^-$ form) directly into a solution of equimolar Cu(ClO$_4$)$_2$.6H$_2$O in ethanol. The resulting solution was evaporated until dry. Crystals were recrystallized from aqueous methanol solution.

Crystal data

[Cu(C$_9$H$_{24}$N$_4$)(ClO$_4$)$_2$]	Mo $K\alpha$ radiation
M_r = 450.76	λ = 0.71073 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 7.949 (1) Å	θ = 10.5–22.5°
b = 9.071 (2) Å	μ = 1.59 mm^{-1}
c = 14.032 (4) Å	T = 298 (3) K
α = 77.58 (2)°	Needle
β = 77.73 (2)°	0.40 × 0.34 × 0.24 mm
γ = 64.18 (1)°	Blue
V = 881.2 (3) Å^3	
Z = 2	
D_x = 1.699 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4	2760 observed reflections
diffractometer	$[I > 2.5\sigma(I)]$
$\theta/2\theta$ scans	R_{int} = 0.012
Absorption correction:	θ_{max} = 25.0°
ψ scans (North, Phillips	$h = 0 \rightarrow 9$
& Mathews, 1968)	$k = -9 \rightarrow 10$
T_{min} = 0.908, T_{max} =	$l = -16 \rightarrow 16$
1.000	3 standard reflections
3449 measured reflections	monitored every 50
3239 independent reflections	reflections
	intensity decay: 1%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.039
R = 0.034	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
wR = 0.036	$\Delta\rho_{min}$ = -0.34 e Å^{-3}
S = 0.98	Extinction correction: none
2760 reflections	Atomic scattering factors
241 parameters	from *International Tables*
Only H-atom U's refined	*for X-ray Crystallography*
Weighting scheme based	(1974, Vol. IV)
on measured e.s.d.'s	

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu	0.23616 (6)	0.79003 (5)	0.24900 (3)	2.389 (19)
Cl(1)	0.35993 (14)	0.78562 (13)	0.47666 (7)	3.62 (5)
Cl(2)	−0.02461 (15)	0.75187 (13)	0.05716 (7)	3.76 (5)
O(1)	0.2338 (7)	0.9510 (5)	0.4767 (3)	9.0 (3)
O(2)	0.2536 (7)	0.6896 (7)	0.5129 (3)	9.4 (4)
O(3)	0.4984 (5)	0.7459 (5)	0.5362 (2)	6.4 (2)
O(4)	0.4424 (4)	0.7592 (4)	0.37692 (19)	4.69 (18)
O(5)	0.0839 (6)	0.7290 (5)	−0.0356 (2)	7.4 (3)
O(6)	−0.2054 (5)	0.8748 (6)	0.0396 (3)	8.8 (3)

O(7)	−0.0447 (10)	0.6096 (6)	0.1074 (3)	12.0 (5)
O(8)	0.0564 (5)	0.8083 (4)	0.1151 (2)	5.00 (18)
N(1)	0.4947 (4)	0.6758 (4)	0.1701 (2)	3.17 (14)
N(2)	0.2620 (4)	0.5594 (3)	0.3105 (2)	2.84 (15)
N(3)	−0.0203 (4)	0.9003 (4)	0.3256 (2)	2.90 (14)
N(4)	0.2101 (4)	1.0240 (4)	0.1922 (2)	2.95 (15)
C(1)	0.4951 (6)	0.6879 (5)	0.0634 (3)	4.2 (2)
C(2)	0.5727 (6)	0.4989 (5)	0.2170 (3)	4.0 (2)
C(3)	0.4131 (6)	0.4440 (4)	0.2461 (3)	3.8 (2)
C(4)	0.0899 (6)	0.5259 (5)	0.3339 (3)	3.8 (2)
C(5)	−0.0676 (6)	0.6452 (5)	0.3961 (3)	4.03 (3)
C(6)	−0.1526 (5)	0.8206 (5)	0.3433 (3)	3.6 (2)
C(7)	−0.1030 (5)	1.0730 (4)	0.2748 (3)	3.44 (18)
C(8)	0.0479 (6)	1.1381 (4)	0.2521 (3)	3.5 (2)
C(9)	0.3750 (7)	1.0642 (6)	0.1800 (4)	4.8 (3)

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

Cu—O(4)	2.573 (3)	N(1)—C(2)	1.498 (5)
Cu—O(8)	2.530 (3)	N(2)—C(3)	1.485 (5)
Cu—N(1)	2.048 (3)	N(2)—C(4)	1.483 (5)
Cu—N(2)	2.022 (3)	N(3)—C(6)	1.471 (5)
Cu—N(3)	2.017 (3)	N(3)—C(7)	1.489 (5)
Cu—N(4)	2.035 (3)	N(4)—C(8)	1.480 (5)
N(1)—C(1)	1.478 (5)	N(4)—C(9)	1.475 (6)
O(4)—Cu—O(8)	175.62 (10)	Cu—N(4)—C(9)	119.2 (3)
O(4)—Cu—N(1)	81.86 (11)	C(8)—N(4)—C(9)	110.4 (3)
O(4)—Cu—N(2)	84.82 (11)	Cu—O(4)—Cl(1)	120.36 (17)
O(4)—Cu—N(3)	98.99 (11)	Cu—O(8)—Cl(2)	157.1 (2)
O(4)—Cu—N(4)	93.41 (11)	Cu—N(1)—C(1)	115.4 (2)
O(8)—Cu—N(1)	93.77 (11)	Cu—N(1)—C(2)	106.5 (2)
O(8)—Cu—N(2)	94.61 (11)	C(1)—N(1)—C(2)	111.1 (3)
O(8)—Cu—N(3)	85.38 (11)	Cu—N(2)—C(3)	106.6 (2)
O(8)—Cu—N(4)	87.25 (12)	Cu—N(2)—C(4)	118.2 (2)
N(1)—Cu—N(2)	85.79 (12)	C(3)—N(2)—C(4)	112.0 (3)
N(1)—Cu—N(3)	178.95 (13)	Cu—N(3)—C(6)	118.4 (2)
N(1)—Cu—N(4)	95.19 (13)	Cu—N(3)—C(7)	106.1 (2)
N(2)—Cu—N(3)	93.66 (12)	C(6)—N(3)—C(7)	111.7 (3)
N(2)—Cu—N(4)	177.84 (12)	Cu—N(4)—C(8)	107.3 (2)
N(3)—Cu—N(4)	85.38 (12)		

The structure was solved by Patterson and Fourier synthesis, and refined by full-matrix least squares. H atoms were located by difference Fourier methods and refined isotropically.

Data collection: *SHELXTL-Plus* (Sheldrick, 1987). Cell refinement: *SHELXTL-Plus*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*. Program(s) used to refine structure: *NRCVAX*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *NRCVAX*.

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Bis(antazoline)dichlorozinc(II)

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Abstract

The structure of the title compound, dichlorobis-[4,5-dihydro-N-phenyl-N-(phenylmethyl)-1H-imidazole-2-methanamine-N^3]zinc(II), [ZnCl$_2$(C$_{17}$H$_{19}$N$_3$)$_2$], is composed of discrete molecules wherein zinc is coordinated to two molecules of antazoline, *via* imidazole N atoms, and two Cl atoms. The geometry around the Zn atom is slightly distorted tetrahedral, with Zn—N bond distances of 2.039 (8) and 2.004 (9) Å, Zn—Cl bond distances of 2.243 (3) and 2.259 (3) Å, and Cl—Zn—Cl and N—Zn—N angles of 116.3 (1) and 108.4 (4)°, respectively.

Comment

Continuing our interest in the studies of anti-allergic drugs effective on H1-type receptor sites, we have synthesized the title compound, (1), which is a transition metal complex of the anti-allergic drug antazoline. In this paper we report the crystal structure of (1), which has been determined in an attempt to understand the influence of transition metals on the conformation of anti-allergic drugs.

(1)
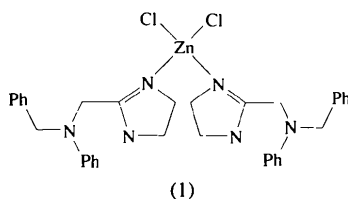
An *ORTEP*II drawing (Johnson, 1976) of the title compound is presented in Fig. 1 together with the atomic numbering scheme. In the crystal structure of (1), the Zn atom has slightly distorted tetrahedral geometry with Zn—Cl bond distances of

2.243 (3) and 2.259 (3) Å, and Zn—N distances of 2.039 (8) and 2.004 (9) Å, which are in accord with the corresponding distances reported for the crystal structures of dichlorobis(xanthine)zinc(II) [Zn—Cl 2.240 (1) and Zn—N 2.029 (3) Å; Dubler, Hanggi & Schmalle, 1992], dichlorobis(imidazole)zinc(II) [Zn—Cl 2.258 (3) and 2.239 (3) Å, and Zn—N 1.995 (11) and 2.020 (11) Å; Lundberg, 1966], dichlorobis{[3-(imidazol-1-yl)-1-oxopropyl]benzene-N}zinc(II) [Zn—Cl 2.226 (1) and 2.242 (1) Å, and Zn—N 2.029 (2) and 2.013 (3) Å; Bremer, Uhlenbrock, Pinkerton & Krebs, 1993] and dichlorobis(1,2-dimethylimidazole)-zinc(II) [Zn—Cl 2.2509 (8) and 2.2468 (8) Å, and Zn—N 2.006 (3) and 2.008 (3) Å; Bharadwaj, Schugar & Potenza, 1991].
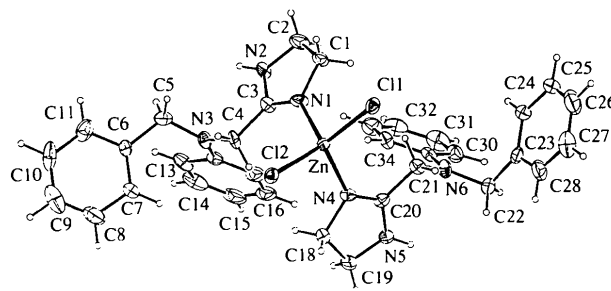

Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the title compound showing the numbering scheme. The non-H atoms are plotted as displacement ellipsoids of 30% probability.

The molecular dimensions in the ligated antazoline fragment are unexceptional [mean bond lengths C=N 1.26 (1), C$_{sp^2}$—N 1.36 (1), C$_{sp^3}$—N 1.46 (1) and C—C(aromatic) 1.37 (2) Å] and agree well with the dimensions of antazoline hydrochloride (Bertolasi, Borea & Gilli, 1982). The imidazole ring in antazoline hydrochloride, however, unlike in (1), has delocalized charge with equal C—N distances, which are between single- and double-bond distances.

The most interesting features of the complex are the angles between the least-squares planes. For example, the N3,C4,C5,C12–C17 moiety and its corresponding moiety in the other half of the complex, both of which are essentially planar [mean deviations 0.04 (1) and 0.03 (1) Å, respectively], are inclined at almost right angles with respect to the imidazole rings [92.4 (9) and 84.9 (9)°, respectively]; the corresponding angle in antazoline hydrochloride is 69.3°. The angles between the C6–C11 and C23–C28 phenyl ring planes and their respective imidazole rings in the two antazoline units of the complex are 47.7 (12) and 89.7 (10)°, compared with 85.0° in antazoline hydrochloride. The angles between the mean planes of the phenyl rings within each ligated antazoline moiety of the complex [84.6 (11) and 99.5 (12)°] are somewhat close to the corresponding angle of 102.5° in antazoline hydrochloride.