

C(44)–N(4) bond distance of 1.54 (3) Å, and this could be the consequence of some error due to the observed disorder.

We thank Professor P. Domiano for help in preparing Fig. 1.

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Structure of {4-[2-(2-Aminoethylamino)ethylimino]pentan-2-onato-N,N',N'',O}copper(II) Perchlorate Monohydrate

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Abstract. [Cu(C₉H₁₈N₃O)]ClO₄·H₂O, *M_r* = 365.3, triclinic, *P*1̄, *a* = 8.603 (2), *b* = 9.666 (2), *c* = 9.993 (3) Å, α = 77.41 (2), β = 67.59 (2), γ = 80.94 (2)°, *V* = 747.2 (3) Å³, *Z* = 2, *D_m* = 1.64 (2), *D_x* = 1.623 (1) Mg m⁻³, λ(Mo *K*α) = 0.70926 Å, μ = 1.72 mm⁻¹, *F*(000) = 378, *T* = 293 K, *R* = 0.040, 4375 unique reflections. The structure consists of [CuC₉H₁₈N₃O]⁺ complex cations and perchlorate anions. The coordination polyhedron around Cu is a distorted square with the C₉H₁₈N₃O⁻ ligand tetradentate to the metal through one O and three N atoms.

Introduction. The study of the title compound was undertaken as part of an investigation of complexes with unsymmetrical tetradentate Schiff bases (Podlahová, Knížek, Loub & Hašek, 1988).

Experimental. The complex was prepared from copper(II) perchlorate, diethylenetriamine and acetylacetone (2,5-pentanedione) in methanolic solution in stoichiometric ratio. The solution was allowed to stand in air for a few days. Dark violet crystals were obtained, stable in air and to X-rays. Density determined by flotation method (mixture of diiodomethane and tetrachloromethane). A spherical crystal of *r* = 0.13 mm was used for the measurements. Syntex *P2*₁ diffractometer, graphite monochromator, 15 reflections with 2θ from 8 to 26° for measuring lattice parameters; ω–2θ scan; absorption ignored; max. (sinθ)/λ = 0.69 Å⁻¹; *h* 0→12, *k* –13→13, *l* –13→13; three standard reflections after 97 reflections with variation <2%; 4645 measured reflections, 4375 unique reflections, 3838 reflections used with *I* ≥ 1.96σ(*I*); heavy-atom method; *F* magnitudes in full-matrix least-squares refinement; H atoms [except H₂O(6)] localized in theoretical positions; all positional parameters were refined with the

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) with e.s.d.'s in parentheses

$$U_{eq} = [U_{11} + U_{22} + U_{33} + 2(U_{12}\cos\gamma + U_{13}\cos\beta + U_{23}\cos\alpha)]/3.$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Cu	163.8 (3)	2179.0 (2)	3108.3 (3)	35.1 (1)
Cl	3584 (1)	7798 (1)	8107 (1)	53.8 (2)
O(1)	-1748 (2)	2011 (2)	4915 (2)	42.0 (5)
O(2)	4790 (4)	7181 (5)	6961 (4)	128 (2)
O(3)	4159 (3)	7640 (4)	9282 (3)	99 (1)
O(4)	1993 (3)	7256 (3)	8597 (3)	80 (1)
O(5)	3381 (4)	9279 (4)	7555 (5)	106 (2)
O(6)	2397 (3)	850 (2)	4256 (2)	57 (1)
N(1)	139 (3)	202 (2)	2748 (2)	43 (1)
N(2)	2134 (2)	2346 (2)	1200 (2)	41 (1)
N(3)	221 (3)	4206 (2)	2905 (2)	42 (1)
C(1)	1563 (4)	-74 (3)	1422 (3)	56 (1)
C(2)	2013 (4)	1319 (3)	368 (3)	53 (1)
C(3)	2121 (4)	3847 (3)	493 (3)	49 (1)
C(4)	1709 (4)	4751 (3)	1672 (3)	52 (1)
C(5)	-949 (3)	5028 (2)	3690 (3)	42 (1)
C(6)	-2410 (3)	4501 (3)	4872 (3)	46 (1)
C(7)	-2738 (3)	3109 (3)	5384 (2)	42 (1)
C(8)	-862 (5)	6631 (3)	3399 (4)	58 (1)
C(9)	-4377 (4)	2753 (4)	6635 (3)	64 (1)

exception of H atoms; anisotropic thermal parameters of Cu, Cl, O, N, C atoms and isotropic thermal parameters of H atoms (one for methylene and amine, one for methyl) were refined; $R = 0.040$, $wR = 0.045$, $w = 1/[\sigma^2(F_o) + 0.0009F_o^2]$; the ratio of maximum least-squares shift to e.s.d. in the last refinement cycle -0.092 ; maximum and minimum height in final $\Delta\rho$ map 0.56 and $-0.57 e \text{\AA}^{-3}$; atomic scattering factors and corrections for anomalous dispersion from *International Tables for X-ray Crystallography* (1974); ICL 4-72 computer; programs *SHELXS86* (Sheldrick, 1986) and *SHELX76* (Sheldrick, 1976).

Discussion. The final atomic parameters are given in Table 1.* Selected intramolecular bond distances and angles are given in Table 2. The complex cation drawn with program *SCHAKAL86* (Keller, 1986) is depicted in Fig. 1.

In the cation, the coordination polyhedron around Cu approximates to a square formed by three N and one O atoms of the ligand. Two next-neighbouring O atoms [O(6), O(4ⁱ), (i) = x, 1-y, 1-z] are located at distances of $2.630(2)$ and $2.886(2) \text{\AA}$ respectively and therefore are considered as non-bonded. The deviations of the CuN₃O core from the best plane are in the range $-0.094(2)$ to $0.109(3) \text{\AA}$. The deviations of the C atoms in the five-membered chelate rings from the

Table 2. Bond distances (\AA) and angles ($^\circ$)

Cu—O(1)	1.919 (2)	C(2)—N(2)	1.465 (4)
Cu—N(1)	2.025 (2)	N(2)—C(3)	1.469 (3)
Cu—N(2)	2.006 (2)	C(3)—C(4)	1.520 (4)
Cu—N(3)	1.932 (2)	C(4)—N(3)	1.470 (3)
Cl—O(2)	1.394 (3)	N(3)—C(5)	1.295 (3)
Cl—O(3)	1.410 (3)	C(5)—C(6)	1.429 (4)
Cl—O(4)	1.410 (2)	C(6)—C(7)	1.364 (3)
Cl—O(5)	1.426 (3)	C(7)—O(1)	1.304 (3)
N(1)—C(1)	1.463 (3)	C(5)—C(8)	1.522 (3)
C(1)—C(2)	1.518 (4)	C(7)—C(9)	1.514 (3)
O(1)—Cu—N(1)	95.0 (1)	N(1)—C(1)—C(2)	109.4 (2)
O(1)—Cu—N(3)	95.2 (1)	C(1)—C(2)—N(2)	107.9 (2)
N(1)—Cu—N(2)	84.4 (1)	C(2)—N(2)—C(3)	116.3 (2)
N(2)—Cu—N(3)	85.2 (1)	N(2)—C(3)—C(4)	107.9 (2)
O(2)—Cl—O(3)	110.2 (2)	C(3)—C(4)—N(3)	107.1 (2)
O(2)—Cl—O(4)	113.0 (2)	C(4)—N(3)—C(5)	122.7 (2)
O(2)—Cl—O(5)	107.0 (3)	N(3)—C(5)—C(6)	122.7 (2)
O(3)—Cl—O(4)	110.6 (2)	C(5)—C(6)—C(7)	126.5 (2)
O(3)—Cl—O(5)	108.4 (2)	C(6)—C(7)—O(1)	126.2 (2)
O(4)—Cl—O(5)	107.5 (2)		

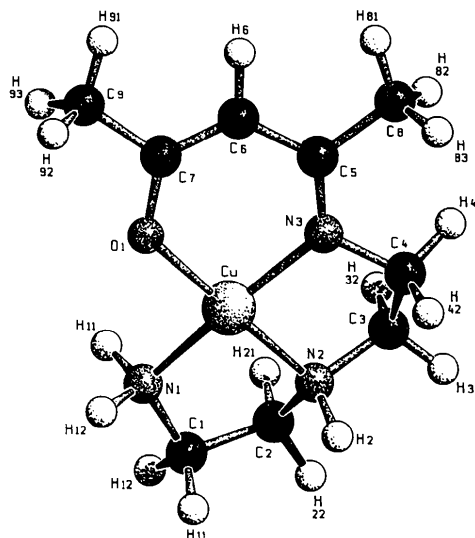


Fig. 1. A view of the [CuC₉H₁₈N₃O]⁺ complex cation.

planes Cu—N(1)—N(2) and Cu—N(2)—N(3) are $0.093(3)$ and $-0.540(3) \text{\AA}$ for C(1) and C(2) and $-0.470(3)$ and $0.184(3) \text{\AA}$ for C(3) and C(4). The conformation of the first ring is an unsymmetric envelope and of the second a half-chair. The deviations of the atoms in the chelate six-membered ring Cu—N(3)—C(5)—C(6)—C(7)—O(1) from the best plane are in the range of $-0.090(3)$ (Cu) to $0.103(2)$ [O(1)] \AA . The torsion angles N(1)—C(1)—C(2)—N(2) and N(2)—C(3)—C(4)—N(3) are 49 and -49° . The largest dihedral angle between the planes considered above is 21° , between the Cu—N(1)—C(1)—C(2)—N(2) and Cu—N(3)—C(5)—C(6)—C(7)—O(1) planes.

Bond lengths and angles in the perchlorate anion are in the usual ranges. An inspection of the crystal packing shows that the bonding between the complex cations and the perchlorate anions is predominantly ionic. The

* Lists of atomic coordinates and isotropic thermal parameters for H atoms, of anisotropic thermal parameters for non-H atoms and of the structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51153 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

shortest intermolecular contact is $O(1)\cdots O(6^{ii}) = 2.789(3) \text{ \AA}$, $(ii) = \bar{x}, \bar{y}, 1 - z$.

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Structure of Isopropylammonium *cyclo*-Triphosphate

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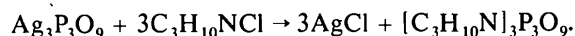
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Abstract. $[\text{NH}_3(\text{C}_3\text{H}_7)]_3\text{P}_3\text{O}_9$, $M_r = 417.27$, monoclinic, $C2$, $a = 25.22(2)$, $b = 12.22(2)$, $c = 15.45(2) \text{ \AA}$, $\beta = 123.90(5)^\circ$, $V = 3953(17) \text{ \AA}^3$, $Z = 8$, $D_x = 1.402 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.350 \text{ mm}^{-1}$, $F(000) = 1776$, $T = 293 \text{ K}$, final $R = 0.039$ for 5327 independent reflexions. The P_3O_9 ring anions are aligned in rows parallel to the c axis. These rows arranged in an almost hexagonal way form large hexagonal channels lined by the isopropylammonium groups.

Introduction. A systematic investigation of isopropylammonium phosphates, condensed or not, has shown the existence of a monophosphate: $[\text{C}_3\text{H}_{10}\text{N}][\text{H}_2\text{PO}_4]$ (Averbuch-Pouchot, Durif & Guitel, 1988*a*) and of a *cyclo*-tetrakisphosphate: $[\text{C}_3\text{H}_{10}\text{N}]_4\text{P}_4\text{O}_{12}\cdot\frac{3}{2}\text{H}_2\text{O}$ (Averbuch-Pouchot, Durif & Guitel, 1988*b*). In the present work we describe the chemical preparation and crystal structure of the *cyclo*-triphosphate.

Experimental. Crystalline samples of isopropylammonium *cyclo*-triphosphates have been prepared by using the metathesis reaction first described by Boullé (1938) and extensively used in organic chemistry for the preparation of water-soluble *cyclo*-triphosphates. To a slurry of silver *cyclo*-triphosphate and water, the stoichiometric amount of an aqueous solution of isopropylamine hydrochloride is slowly added:



After removing the silver chloride by filtration, the resulting solution is kept at room temperature until formation of the crystals, which appear as pseudo-hexagonal thick plates. Several recrystallizations are necessary to obtain good quality crystals.

Density not measured. Plate fragment $0.25 \times 0.30 \times 0.30 \text{ mm}$. Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Systematic absences: hkl , $h + k = 2n$. 19 reflexions ($12 < \theta < 18^\circ$) for refining unit-cell dimensions. ω scan. 7386 reflexions measured ($3 < \theta < 32.5^\circ$), $\pm h, k, l$, $h_{\max} = 39$, $k_{\max} = 18$, $l_{\max} = 21$. Scan width: 1.20° , scan speed between 0.02 and $0.04^\circ \text{ s}^{-1}$, total background measuring time: between 17 and 30 s.

Two orientation (426 and $4\bar{2}6$) and two intensity ($10, 2, 0$ and $\bar{1}0, \bar{2}, 0$) reference reflexions, no variation. Lorentz and polarization corrections, no absorption correction. Crystal structure solved by direct methods (*MULTAN*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977). Anisotropic full-matrix least-squares refinement (on F) for non-hydrogen atoms, isotropic for H atoms. Unit weights. Final refinement with 5327 reflexions ($I > 3\sigma$). Final $R = 0.039$ ($wR = 0.039$). For the complete set of unique reflexions (6473) $R = 0.049$. Extinction not refined. Max. $\Delta/\sigma = 0.26$ [β_{33} of $C(22)$]. Maximum peak height in final difference Fourier map: 0.267 e \AA^{-3} . $S = 0.702$. Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* used for all calculations. Computer used: MicroVAX II.

Discussion. Final atomic coordinates are given in Table 1.* Two crystallographically independent P_3O_9 ring anions and six independent isopropylammonium groups

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51213 (53 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.