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

Ti1—N1	2.241 (4)	Ti1—Cl1	2.3086 (15)
Ti1—Cl3	2.2910 (10)	Ti1—Cl2	2.3485 (10)
N1—Ti1—Cl3	87.30 (8)	Cl3'—Ti1—Cl2	173.17 (6)
$Cl3^i$—Ti1—Cl3	91.23 (5)	Cl3—Ti1—Cl2	90.36 (3)
N1—Ti1—Cl1	177.45 (13)	Cl1—Ti1—Cl2	92.01 (4)
Cl3—Ti1—Cl1	94.48 (5)	Cl2—Ti1—Cl2'	87.29 (5)
N1—Ti1—Cl2	86.15 (8)		

Symmetry code: (i) $-x, y, z$.

The data collection nominally covered over a sphere of reciprocal space by a combination of five sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.95 cm. Coverage of the unique set is over 95% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [$U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$, $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U(\text{H}) = 1.2U_{\text{eq}}(\text{N})$] using a riding model with C—H(methyl) = 0.98, C—H(aromatic) = 0.95 or N—H = 0.88 Å.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*b*). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1120). Services for accessing these data are described at the back of the journal.

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trans-Dichloro(1,4,8,11-tetrazacyclotetradecane)cobalt(III) Chloride Tetrahydrate 0.47-Hydrochloride

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Abstract

The crystal and molecular structures of the title compound, $[\text{CoCl}_2(\text{C}_{10}\text{H}_{24}\text{N}_4)]\text{Cl.4H}_2\text{O.0.47HCl}$, have been determined and establish that the *trans*-$[\text{CoCl}_2(\text{cyclam})]^+$ cations crystallize as the *trans-RSSR* species. The Co atom occupies a crystallographic centre of symmetry and is in a tetragonally elongated octahedral coordinated array, comprising the four N atoms of the macrocycle and two Cl atoms. The chloride counterion exhibits statistical disorder, while one of the solvate water molecules also exhibits orientational disorder as a result of partial protonation. In the crystal packing, all possible hydrogen bonds are formed. The *trans*-$[\text{CoCl}_2(\text{cyclam})]^+$ cations are stacked into columns along the **b** direction forming channels which are occupied by the chloride counterion and the water molecules.

Comment

In studies of the details of the base-catalyzed substitution reactions of amine (aniono) cobalt(III) complexes, a number of systems have been found where the rate constants (k_2) for solvolysis of the amido conjugate base forms are of comparable magnitude with, or even significantly greater than, the rate constants (k_{-1}) for the reprotonation of these forms (Lichtig, Sosa & Tobe, 1984; Ahmed & Tobe, 1974; Marangoni, Panayotou & Tobe, 1973; Poon & Tobe, 1968). The *trans* stable isomer of the dichloro(cyclam)cobalt(III) complex was the first example (Poon & Tobe, 1968) for which it was shown that $k_{-1} << k_2$; in that study, the *RSSR* configuration had been assumed, but no X-ray study had been reported.

In addition to the geometric (*trans–cis*) isomerism in which the ligand is flat or folded, it is necessary to take into account further isomerism, arising from the configuration of the coordinated amine N atoms. Moreover, as far as we know, this is the first example of

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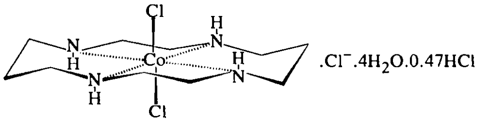
a single-crystal structure determination of a *trans*-Co^{III} complex derived from cyclam.

The study of the X-ray structure of the *cis*-dichloro(cyclam)cobalt(III) chloride complex was published as a footnote (Whimp, Bailey & Curtis, 1970) and the configuration reported was *RRRR* (*SSSS*). The only other complete structure analysis of octahedral cobalt(cyclam) complexes corresponds to *cis*-ethylenediamine(cyclam)cobalt(III) chloride (Lai & Poon, 1976) and *cis*-oxalate(cyclam)cobalt(III) nitrate (Sovilj *et al.*, 1994).

The structure reported here for the *trans*-dichloro(cyclam)cobalt(III) chloride complex, (I) (Fig. 1), confirms the original configuration assumption, *RSSR*, made by Tobe (Bosnich, Poon & Tobe, 1965) by analogy with the structure of the *trans*-$[NiCl_2(cyclam)]$ complex (Bosnich, Tobe & Webb, 1965).



(I)

The *trans*-$[CoCl_2(cyclam)]^+$ cation can be compared with the Ni^{III} (Ito, Sugimoto, Toriumi & Ito, 1981) and the Ru^{III} (Walker & Taube, 1981) analogues, and also with $[Co(cyclam)](ClO_4)_2$ (Endicott *et al.*, 1977).

The Co^{III} ion resides at a crystallographic centre of symmetry and is surrounded by four N atoms of the cyclam in the equatorial plane and two Cl atoms occupying the axial positions of an octahedron. The mean value for the equatorial Co—N bonds is 1.978 (4) Å, whereas the Co—Cl distance is 2.2524 (6) Å. Slight deviations from ideal values for the N—Co—N angles in the five-membered rings (<90°) and six-membered rings (>90°) are observed, causing a distortion from the square-planar coordination, producing a rectangle with sides of length 2.701 (3) and 2.889 (3) Å. Non-bonding interactions within the cation cause a further distortion from a regular octahedral arrangement. The Cl—Co—Cl axis is tilted [1.5 (1)°] as a result of a Cl1···H1 contact (Table 3). As found in other *cis* and *trans* isomers of cyclam complexes (Forsellini, Parasassi, Bombieri, Tobe & Sosa, 1986; Flores-Velez, Sosa-Rivadeneyra, Sosa-Torres, Rosales-Hoz & Toscano, 1991), the corresponding Co—N distances are shorter in the *trans* isomers.

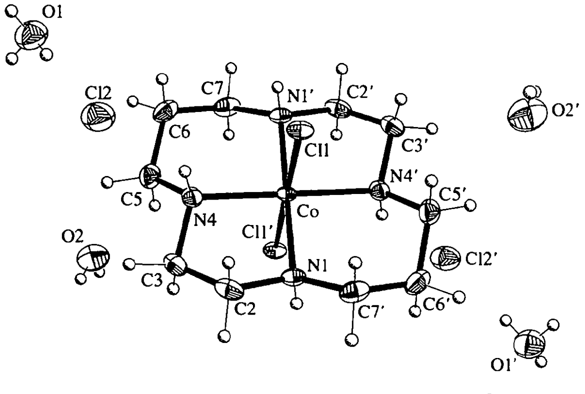


Fig. 1. The title compound showing the labelling of the non-H atoms. Primed atoms are generated by $\bar{1}$ symmetry. Anisotropic displacement ellipsoids are shown at 30% probability levels.

The macrocyclic ring conformation can be described as the *trans*-III isomer of Tobe (Bosnich, Poon & Tobe, 1965) with an *RSSR* configuration. The five-membered chelate rings adopt twist conformations, whereas the six-membered chelate rings are in chair conformations.

The *trans*-$[CoCl_2(cyclam)]^+$ cations are stacked along the **b** direction and held together by N1—H1···Cl1 hydrogen bonds. The channels left by this arrangement are filled by the chloride ion (arising from the counter-ion of the complex and the hydrochloric acid used for crystallization) and the solvate water molecules (two of them appearing as partially protonated moieties) forming a rather complicated hydrogen-bonding scheme (Fig. 2 and Table 3) where all possible hydrogen bonds are formed. The crystal structure is stabilized by these weak interactions and they explain the low stability of the crystals.

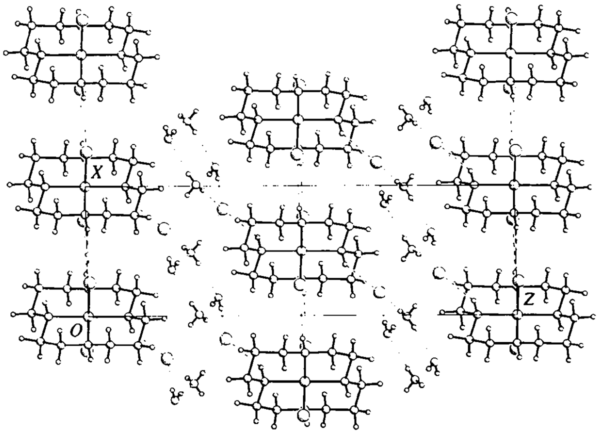


Fig. 2. The crystal packing of $[CoCl_2(cyclam)]Cl$ viewed along the *b* axis.

Experimental

The *trans*-$[CoCl_2(cyclam)]Cl$ complex was synthesized according to the method of Bosnich, Poon & Tobe (1965). (Found: C 32.8, H 6.5, N 15.3%; $C_{10}H_{24}Cl_3CoN_4$ requires C 32.8, H 6.6, N 15.3%.) The nature of the Co^{III} compound was confirmed by electron paramagnetic resonance (EPR) spectroscopy. The EPR spectrum of the compound showed no signal, as expected. Taking into account the high sensitivity of this technique, this can be taken as evidence of the diamagnetic purity of the Co^{III} species. The crystals were grown by slow evaporation of a dilute hydrochloric acid solution of the compound. Intensities were collected on a sample mounted in a Lindeman glass capillary filled with mother liquor to prevent crystal decomposition.

Crystal data

$[CoCl_2(C_{10}H_{24}N_4)]Cl.4H_2O.$-$0.47HCl$
$M_r = 454.81$
Monoclinic
$P2_1/n$
$a = 6.393\,(2)$ Å
$b = 7.433\,(1)$ Å
$c = 21.067\,(3)$ Å
$\beta = 90.97\,(2)^\circ$
$V = 1000.9\,(4)$ Å^3
$Z = 2$
$D_x = 1.509$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 44 reflections
$\theta = 4$–15°
$\mu = 1.340$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.82 × 0.62 × 0.58 mm
Green

Data collection

Siemens *P4/PC* diffractometer
ω scans
Absorption correction: ψ scans (*XEMP*; Siemens, 1990)
$T_{\min} = 0.346$, $T_{\max} = 0.460$
3155 measured reflections
2914 independent reflections

2297 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 30^\circ$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = -29 \rightarrow 29$
3 standard reflections every 97 reflections intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.110$
$S = 1.072$
2912 reflections
159 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.2907P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.040$
$\Delta\rho_{\max} = 0.605$ e Å^{-3}
$\Delta\rho_{\min} = -0.975$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.084 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

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

	x	y	z	$U_{\rm eq}$
Co	1/2	0	1/2	0.0212 (1)
Cl1	0.23878 (7)	0.20309 (7)	0.49486 (3)	0.0321 (2)
Cl2†	0.1763 (2)	0.2511 (2)	0.68061 (6)	0.0666 (5)
N1	0.7152 (3)	0.1904 (3)	0.50221 (10)	0.0296 (4)
C2	0.7181 (4)	0.2728 (4)	0.56615 (14)	0.0403 (6)
C3	0.6882 (4)	0.1251 (4)	0.61340 (13)	0.0400 (6)
N4	0.4982 (3)	0.0222 (3)	0.59369 (9)	0.0307 (4)
C5	0.4729 (4)	−0.1408 (4)	0.63270 (13)	0.0430 (6)
C6	0.2774 (5)	−0.2452 (4)	0.61388 (15)	0.0473 (7)
C7	0.2843 (4)	−0.3275 (3)	0.54865 (14)	0.0405 (6)
O1	−0.0108 (4)	−0.0838 (5)	0.7420 (2)	0.0704 (8)
O2	0.8588 (5)	−0.4502 (5)	0.6962 (2)	0.0836 (9)

† Site occupancy = 0.734 (4).

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

Co—N1	1.974 (2)	Co—Cl1	2.2524 (6)
Co—N4	1.981 (2)		
N1—Co—N4	86.14 (8)	N4—Co—Cl1$^{\rm i}$	91.42 (6)
N1—Co—N4$^{\rm i}$	93.86 (8)	H1*A*—O1—H1*B*	93 (6)
N1—Co—Cl1	92.08 (6)	H1*A*—O1—H1*C*	108 (7)
N4—Co—Cl1	88.58 (6)	H1*B*—O1—H1*C*	111 (8)
N1—Co—Cl1$^{\rm i}$	87.92 (6)	H2*C*—O2—H2*D*	110 (6)
N4—Co—N1—C2	−13.6 (2)	Co—N4—C5—C6	−54.9 (3)
Co—N1—C2—C3	39.0 (2)	N4—C5—C6—C7	66.9 (3)
N1—C2—C3—N4	−52.2 (3)	C5—C6—C7—N1$^{\rm i}$	−66.9 (3)
C2—C3—N4—Co	39.9 (2)	Co—N1$^{\rm i}$—C7—C6	54.6 (3)
N1—Co—N4—C3	−14.7 (2)	N4—Co—N1$^{\rm i}$—C7	−36.8 (2)
N1$^{\rm i}$—Co—N4—C5	37.3 (2)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1$^{\rm i}$	0.84 (3)	2.70 (3)	3.355 (2)	137 (2)
O1—H1*A*···Cl2	0.89 (5)	2.18 (5)	3.058 (4)	169 (6)
O1—H1*B*···O2$^{\rm ii}$	0.69 (5)	2.08 (6)	2.779 (5)	174 (6)
O1—H1*C*···Cl2$^{\rm iii}$	0.70 (5)	2.24 (5)	2.936 (3)	172 (6)
O2—H2*C*···Cl2$^{\rm iv}$	0.78 (4)	2.25 (4)	3.030 (4)	176 (6)
O2—H2*D*···O1$^{\rm i}$	0.73 (4)	2.29 (5)	3.002 (5)	165 (6)
N4—H4···Cl2	0.88 (3)	2.48 (3)	3.257 (2)	148 (2)

Symmetry codes: (i) $1+x, y, z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iv) $1+x, y-1, z$.

The site-occupation factor and the thermal displacement factor of chloride counterion Cl2 were refined by keeping one of them constant and varying the other alternately until they converged to the values indicated. At an advanced anisotropic refining step, three well defined electron-density residues were located surrounding the water O1 atom. Restrained refinement [O—H ~0.85 (5) Å], as for the chloride ion, showed that the occupation factors for those H atoms add up to 2.22 (13). In the final model, a fixed site-occupation factor of 0.7446 and an isotropic displacement parameter $1.2U_{\rm eq}$ of the parent O atom were used for each H atom in the partially protonated water molecule.

Data collection: *XSCANS* (Siemens, 1993). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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(Imidazole-N^3)(N-salicylidene-α-aminoisobutyrato-O,N,O')copper(II) Dimer†

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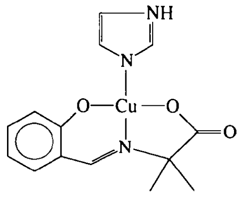
Abstract

The title compound, $[Cu(C_{11}H_{11}NO_3)(C_3H_4N_2)]$, adopts a square-pyramidal Cu^{II} coordination with the tridentate N-salicylidene-α-aminoisobutyrato Schiff base dianion and the imidazole ligand bound in the basal plane. The apex of the pyramid is occupied by a phenolic O atom from the neighbouring chelate at an apical distance of 2.623 (2) Å, building a dimeric structural unit in which the copper(II) ions are magnetically equivalent. The dimeric moieties in the crystal packing are tilted with respect to each other leading to a distorted antiferrodistortive ordering.

Comment

Copper(II) complexes with tridentate Schiff base dianions of the N-salicylideneaminoacidato type (TSB^{2-}) represent a relatively simple model for studies of cooperative bonding effects, which can be investigated by electron paramagnetic resonance (EPR) spectroscopy. In these complexes, which are of the general type $[Cu(TSB)(L)]_n$ (Warda, 1994), three donor atoms (O, N and O) of the Schiff base and a fourth donor atom from the neutral ligand L (N, O or S) normally define the base of a square pyramid. For isolated (monomeric) structures, the copper coordination can be square planar or square pyramidal when a neutral donor ligand is located at the apical site (n = 1; Ueki, Ashida, Sasada & Kakudo, 1969; Warda, Friebel, Sivý, Plesch & Švajlenová, 1996); n = 4 is found in the case of (4-ethylpyridine)(N-salicylideneglycinato)copper(II) (Warda, 1997). Polymeric structures ($n = \infty$) are generally achieved when the apical position is occupied by a carboxylic O atom from an adjacent molecule to make infinite zigzag chains (Warda, Friebel, Sivý, Plesch & Bláhová, 1997). If a phenolic O atom from a neighbouring molecule is apically coordinated, dimeric structures are formed (n = 2; Warda, 1994). In this communication, we report on such a case.



(I)

The molecule is characterized by a square-pyramidal Cu^{II} coordination with the tridentate Schiff base N-salicylidene-α-aminoisobutyrato dianion and a monodentate ligand in the basal plane (imidazole). The involvement of the phenolic O atom of a neighbouring molecule at the apical site, with a Cu—O1^{i} distance of 2.623 (2) Å, leads to a dimeric structure [symmetry code: (i) $2-x$, $1-y$, $-z$]. This distance is comparable with the corresponding bond [2.591 (3) Å] in (imidazole)[N-salicylidene-(R,S)-alaninato]copper(II) (Warda, 1994).

The dimers of the title compound are linked by N3—H31···O3 hydrogen bonds to form a ribbon structure parallel to the x axis (Fig. 2).

It has been found (Warda, 1994) that EPR patterns display a coupled g tensor (non-molecular), indicating a distorted antiferrodistortive ($90 > 2\gamma > 45°$) ordering, with a tilting angle of $2\gamma = 66°$ (EPR result), when the distance between the differently oriented paramagnetic centres is shorter than the critical distance of 8.3 Å; these features are found in the title compound, with a

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† Alternative name: bis-μ-[2-(2-hydroxybenzylideneamino)-2-methylpropionato-O,N,O':O]-bis[(imidazole-N^3)copper(II)].