Acta Chemica Scandinavica A 32 (1978) 501 – 507

The Crystal Structure of (—)$_D$-*u-fac*-Bis[di(2-aminoethyl)-sulfide]cobalt(III) Chloride Dihydrate and the Absolute Configuration of the Cation

A. HAMMERSHØI, ERIK LARSEN and SINE LARSEN

Chemistry Departments I and IV, The H. C. Ørsted Institute, Universitetsparken 5, DK-2100 Copenhagen, Denmark

The crystal structure of (−)$_D$-*u-fac* bis[di(2-aminoethyl)sulfide]cobalt(III) chloride dihydrate has been determined by X-ray diffraction methods. The crystals are orthorhombic, space group $P2_12_12_1$, $Z=4$, with the unit cell dimensions $a=14.665(3)$ Å, $b=15.821(3)$ Å, and $c=7.979(2)$ Å. Cu$K\alpha$ and Mo$K\alpha$ diffraction data were collected on an automatic four circle diffractometer. The structure was determined from Patterson and Fourier methods. The final refinement of 3284 reflections (Mo radiation) led to $R=0.040$ and $R_w=0.046$.

The structure determination confirms an earlier assignment of the structure as unsymmetrical facial. The Cu-data were used to establish the absolute configuration Δ in agreement with an earlier assignment based on the empirical correlation between circular dichroism spectra and absolute configuration. All four five-membered chelate rings have λ conformations. A full description of the cation is therefore (−)$_D$-Δ($\lambda\lambda\lambda\lambda$)-*u-fac*.

The complex bis[di(2-aminoethyl)sulfide]cobalt(III), $[Co(daes)_2]^{3+}$, could in principle exist in three geometric isomers (*symmetrical facial*, *unsymmetrical facial* and *meridional*) each of which would have the possibility of forming several conformers. Recently, an analysis was made of the reaction products from a number of preparative routes leading to $[Co(daes)_2]^{3+}$ including some routes commonly believed to involve equilibrium.[1] This analysis revealed that only one geometric isomer was formed in measurable yield. The result is astonishing in view of the fact that all three geometrical isomers of the corresponding complex bis(diethylenetriamine)-cobalt(III), $[Co(dien)_2]^{3+}$, are formed in approximately equal amounts.[2] Because the bond angle for C−S−C is generally found to be *ca.* 100° the *mer* isomer of $[Co(daes)_2]^{3+}$ would be highly strained and this may well be the reason for the absence of *mer* form. However, from model considerations there seems to be no apparent reason why one of the *fac* forms and not the other should be stable. It was concluded[1] that the found form of $[Co(daes)_2]^{3+}$ was the unsymmetrical *facial* because the complex could be resolved in optically active forms with a pronounced circular dichroism. The *u-fac* form must have a chiral disposition of the chelate rings – a configurational dissymmetry – whereas the *s-fac* isomer can have only a conformational dissymmetry presumably with much less circular dichroism and a low barrier towards racemization. The present structure determination has confirmed that the configuration is *u-fac*.

Since Saito first determined the absolute configuration of a cobalt(III) complex[3] a large body of empirical material has been collected which shows that there is a rather general correlation between the circular dichroism exhibited by a complex with five-membered chelate rings and its absolute configuration[4] expressed for example in terms of Δ and Λ. For cobalt(III) complexes of six nitrogen ligators empirical correlations have been specially successful and extensions to complexes with other ligating atoms than nitrogen have been attempted apparently with good reliability. Thus it seems that cobalt(III) complexes of the absolute configuration Δ have a dominating negative circular dichroism connected to the absorption related to the spin allowed transition having lowest energy in an

octahedral complex. This behaviour was observed for $(-)_D$-$[Co(daes)_2]Cl_3$ and on this basis the absolute configuration was predicted to be Δ.[1] This is substantiated in the present work and with this confirmation of the prediction based on the empirical rule more reliability may be connected to future empirical conclusions also for chelates containing a thioether as a ligator.

During the initial experiments a number of racemic complex salts as well as optically active salts were investigated to search for crystals most suitable for the single crystal X-ray investigation. It was hereby found that the racemic chloride and the optically active chloride were isomorphous. This is a relatively rare situation and accordingly the $(-)_D$-$[Co(daes)_2]Cl_3.2H_2O$ was selected for further work.

EXPERIMENTAL

$(-)_D$-$[Co(daes)_2]Cl_3.2H_2O$ was prepared as published earlier.[1] Crystals suitable for diffraction work were obtained by recrystallization from a 50 % water/ethanol mixture. The water content is not well defined and depending on humidity and temperature water contents were found by thermogravimetry to vary between 2 and 2.5. Found: C 21.5; H 6.5; N 12.6; S 14.6; Cl 23.7. Calc. for $C_8H_{29}N_4O_{2.5}S_2Cl_3Co$: C 21.3; H 6.5; N 12.4; S 14.2; Cl 23.6. Specific rotations for the sample at 25 °C (0.2 % aqueous solution, 2 cm cell length): $[\alpha]_{589}$ −322°; $[\alpha]^{578}$ −446°; $[\alpha]^{546}$ −895°; $[\alpha]_{436}$ +597°.

The compound crystallizes as orange red prisms. X-Ray diffraction photographs showed that the crystals are orthorhombic, the space group being uniquely determined to be $P2_12_12_1$. Crystals from the racemic sample were found to have the same space group and identical unit cell parameters. Powder photographs taken of both the optically active and the racemic samples confirmed this observation. The powder diffraction patterns were obtained using a Hägg-Guinier camera XDC 700 with Cu$K\alpha$ radiation monochromated by a quartz crystal employing silicon as an internal standard.

The density of the crystals was measured by flotation in a mixture of α-bromonaphthalene and 1,2-dibromopropane.

Data collection. A single crystal with dimensions $0.09 \times 0.13 \times 0.32$ mm^3 limited by 20 faces was chosen for the data collection and for the determination of unit cell parameters on a Picker FACS-1 diffractometer. Two sets of data were collected at 22 °C using graphite monochromated Cu$K\alpha$ and Mo$K\alpha$ radiation, respectively.

Both data sets were obtained by operating the diffractometer in a $\theta-2\theta$ scan mode at a rate in 2θ of 2° min^{-1} for the Cu-data and 1° min^{-1} for the Mo-data. The scan was symmetrical and increased with 2θ following the expression $\Delta 2\theta_{SC} = 2A + 2D + \tan\theta$, where with Cu$K\alpha$ radiation $A = 1.6°$, $D = 0.143°$ and for Mo$K\alpha$ radiation $A = 1.5°$ and $D = 0.346°$. Background counts were made at each end of the scan range for 10 s for the Cu-data and for 20 s for the Mo-data. The intensities of standard reflections were measured after every 40 reflections. In both cases these measurements showed that no deterioration or misalignment of the crystal occurred during the data collections. The intensity data were measured for reflections in two octants which were not symmetry related (hkl, $h\bar{k}l$). The Cu-data were obtained for $6° < 2\theta < 127.5°$ and the Mo-data for $6° < 2\theta \leq 54°$. The criterion $I/\sigma(I) > 2.0$ was used to classify a reflection as observed. The data obtained with Cu$K\alpha$ radiation consist of 2867 independent reflections of which 2706 were observed. The data obtained with Mo radiation consist of 4022 independent reflections of which 3284 were observed. The two data sets were corrected for Lorentz and polarization effects and absorption.

For the crystallographic calculations the unit cell parameters used were those resulting from the least-squares refinement of the setting angles of 12 reflections measured with Cu$K\alpha$ radiation.

The following computer programs were used during the computations: The Vanderbilt system [5] for diffractometer operations, a data reduction program of local origin, ORTEP II for the illustrations,[6] the X-ray system [7] for the crystal structure analysis, and Simplex [8] for weight analysis.

The atomic scattering factors employed were those reported by Cromer and Mann [9] taking the values for the uncharged non-hydrogen atoms. For hydrogen the scattering factor calculated by Stewart *et al.*[10] was used. The anomalous dispersion corrections added to the scattering factors for cobalt, sulfur, and chlorine were from Cromer and Liberman.[11]

CRYSTAL DATA

$(-)_D$-Bis[di(2-aminoethyl)sulfide]cobalt(III) chloride dihydrate; $C_8H_{29}N_4O_{2.5}S_2Cl_3Co$; M = 450.8. Orthorhombic, $a = 14.665(3)$ Å, $b = 15.821(3)$ Å, $c = 7.979(2)$ Å; $V = 1851.2$ Å^3; $d_{obs} = 1.609$ g/cm^3; $Z = 4$; $d_{calc} = 1.617$ g/cm^3. μ(Cu$K\alpha$) = 138.2 cm^{-1}, μ(Mo$K\alpha$) = 15.78 cm^{-1}. $F(000) = 920$. Space group $P2_12_12_1$ (No. 19). Developed forms, {110}, {011}, {111} and {120}.

STRUCTURE DETERMINATION AND REFINEMENT

The intensity measurements with Cu$K\alpha$ radiation were performed first and this data set was used to solve the structure. The position of the cobalt atom could be deduced from the three-dimensional Patterson function. The other non-hydrogen atoms were located by Fourier syntheses.

The structure was refined by the method of least-squares minimizing $R=\Sigma w(|F_o|-K|F_c|)^2$. After a full matrix refinement with unit weights of the scale factor, the atomic parameters, anisotropic thermal parameters for Co, S and Cl, and isotropic temperature factors for the other atoms the R-value was 0.078. A difference Fourier calculated after this refinement contained a large peak at a distance of approximately 2.5 Å from one of the oxygen atoms which exhibited a very large temperature factor. From this it was concluded that one of the water molecules in the structure is distributed between at least two sites. The population parameters for the two oxygen atoms placed at these sites were included in the following least-squares refinements. Two cycles of refinement were performed with fixed temperature factors allowing the populations to vary. This was followed by two cycles in which the populations were fixed while the temperature factors were varied.

The sum of population parameters for the two partly occupied sites tended to be bigger than 1.0 and the anisotropic temperature factors for the oxygen atoms took physically unrealistic values. An analysis of the difference Fourier calculated without including the disordered water molecule showed that the water was located in a banana-shaped area that could contain more than one water molecule. A better description of this disorder was obtained by using four different sites each having independent population and isotropic thermal parameters. For the other non-hydrogen atoms anisotropic temperature factors were used.

The 24 hydrogen atoms on the ligands were located in a difference Fourier based on the Mo$K\alpha$ data. The positional parameters of the hydrogen atoms were also included in the least-squares refinement. The thermal parameters for the hydrogen atoms were not refined but were given the refined isotropic temperature factor of the atom to which they are attached.

The weights used were $w=1/(A+B\sigma(F)^2+CF+DF^2)$. The numerical values for A, B, C and D were obtained as described and programmed by Nielsen.[8] During the final cycles of least-squares refinements the coefficients in the weighting function were A, B, C, D (Cu$K\alpha$) = 5.78, 0.75, 0.31, 0.0059 and A, B, C, D (Mo$K\alpha$): 0.0, 1.02, 0.033, 0.0005. This refinement was continued until the maximum change in the population and thermal parameters for the disordered oxygen atom was less than 0.5σ between two successive refinements. The maximum change of the other parameters was 0.05σ. The unit weighted and weighted residuals R and R_w are 0.040 and 0.046 respectively for Mo$K\alpha$ data. For the Cu$K\alpha$ data $R=0.051$ and $R_w=0.059$.

In order to establish the absolute configuration of the complex the mirror image of the structure was refined as well using both data sets. For the Cu-data $R=0.120$ and $R_w=0.154$ and the corresponding values for the Mo-data are $R=0.049$ and $R_w=0.058$. Using Hamilton's R-value test[12] the latter solution can be rejected on a significance level much lower than 0.005 even on the Mo-data alone. By inspecting the list of observed and calculated structure factors of the Cu-data for the two catoptric forms the same conclusion is reached.

The two parameter sets resulting from the refinements using the Cu- and Mo-data sets were compared by performing two full normal probability plots for the positional and thermal parameters, respectively. These plots had the correct slope but did not pass through 0.0 thus indicating the presence of systematic errors. We suspect that insufficient correction for absorption in the Cu-data may have caused the error since the great number of faces made measurements of the crystal inaccurate. The parameters given in Tables 1–3 are thus those obtained from the Mo-data since these are considered the most reliable. Lists of structure factors may be obtained from the authors.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The hexacoordinated cobalt(III) complex has the unsymmetrical *facial* configuration with the sulfur atoms *cis* as shown in Fig. 1. The cation has effectively C_2 symmetry when corresponding bond distances, bond angles, and dihedral angles (see Tables 4 and 5) are compared. The bond angles at the cobalt atom are close to the octahedral values. The bite angles are slightly less than 90° and the other bond angles are opened correspondingly.

Table 1. Final fractional coordinates. The estimated standard deviations are given in parentheses. The labelling corresponds to Fig. 1.

Atom	*x*	*y*	*z*
Co	0.01455(4)	0.11854(3)	0.05597(7)
S1	−0.03216(7)	−0.00633(6)	−0.04623(16)
S2	0.14111(8)	0.05713(7)	0.15821(15)
N1	−0.0942(3)	0.1685(2)	−0.0506(7)
N2	−0.0560(3)	0.0930(2)	0.2597(5)
N3	0.0555(3)	0.2246(2)	0.1601(6)
N4	0.0856(3)	0.1444(3)	−0.1498(6)
C1	−0.1248(3)	0.1223(4)	−0.2017(7)
C2	−0.1325(4)	0.0288(3)	−0.1637(7)
C3	−0.0827(4)	−0.0468(3)	0.1469(7)
C4	−0.1239(3)	0.0241(3)	0.2428(7)
C5	0.1165(4)	0.2101(3)	0.3042(8)
C6	0.1916(4)	0.1500(4)	0.2590(8)
C7	0.2024(3)	0.0529(4)	−0.0379(7)
C8	0.1853(4)	0.1308(4)	−0.1377(8)
Cl1	0.05997(9)	0.01449(9)	0.5576(2)
Cl2	0.18657(8)	0.35138(8)	−0.0433(2)
Cl3	−0.1099(1)	0.2974(1)	0.4061(3)
O1	0.2357(4)	0.3378(3)	0.5665(6)
H1(N1)	−0.085(4)	0.216(4)	−0.075(7)
H2(N1)	−0.141(4)	0.170(3)	0.019(7)
H1(C1)	−0.078(4)	0.135(3)	−0.279(7)
H2(C1)	−0.187(4)	0.152(3)	−0.244(6)
H1(C2)	−0.140(4)	−0.009(3)	−0.255(7)
H2(C2)	−0.194(3)	0.019(3)	−0.100(6)
H1(C3)	−0.028(3)	−0.075(3)	0.197(6)
H2(C3)	−0.127(3)	−0.088(3)	0.108(6)
H1(C4)	−0.144(3)	0.005(3)	0.368(6)
H2(C4)	−0.176(4)	0.046(3)	0.195(6)
H1(N2)	−0.012(3)	0.077(3)	0.338(6)
H2(N2)	−0.077(4)	0.134(3)	0.290(7)
H1(N3)	0.008(4)	0.248(3)	0.180(7)
H2(N3)	0.085(3)	0.258(3)	0.084(6)
H1(C5)	0.076(4)	0.187(3)	0.404(6)
H2(C5)	0.141(4)	0.247(4)	0.332(7)
H1(C6)	0.216(4)	0.129(4)	0.341(8)
H2(C6)	0.238(4)	0.169(3)	0.177(7)
H1(C7)	0.180(3)	0.001(3)	−0.085(6)
H2(C7)	0.260(4)	0.052(3)	−0.010(6)
H1(C8)	0.208(4)	0.124(3)	−0.261(7)
H2(C8)	0.217(4)	0.179(3)	−0.085(7)
H1(N4)	0.069(4)	0.115(3)	−0.244(7)
H2(N4)	0.067(4)	0.194(4)	−0.181(7)

Table 2. Refined parameters for the disordered water molecules. Estimated standard deviations in parentheses.

Atom	Population	*x*	*y*	*z*	$U \times 10^2$ Å^2
O2	0.58(1)	−0.0392(6)	0.3179(6)	−0.203(1)	7.4(2)
O3	0.50(1)	0.0009(8)	0.2950(7)	−0.272(1)	7.6(3)
O4	0.42(3)	−0.102(1)	0.339(1)	0.078(2)	8.2(5)
O5	0.12(2)	−0.109(2)	0.337(3)	0.021(5)	3.7(9)

Table 3. Final thermal parameters, u_{ij}, in units of $Å^2 \times 10^{-2}$. The expression for the temperature factors is $\exp\{-2\pi^2(u_{11}h^2a^{*2}+\cdots+2u_{12}hka^*b^*+\cdots)\}$. The estimated standard deviations are given in parentheses.

Atom	u_{11}	u_{22}	u_{33}	u_{12}	u_{13}	u_{23}
Co	2.00(2)	1.95(2)	2.45(2)	−0.06(2)	0.06(2)	0.10(2)
S1	3.05(5)	2.23(4)	3.27(5)	−0.13(4)	−0.01(5)	−0.43(5)
S2	2.66(6)	2.93(6)	3.49(6)	0.47(5)	−0.38(5)	−0.19(5)
N1	2.9(2)	1.9(2)	4.5(2)	0.1(1)	0.0(2)	0.3(2)
N2	3.3(2)	2.3(2)	2.7(2)	0.0(2)	0.1(2)	0.1(2)
N3	2.9(2)	2.5(2)	4.2(2)	0.0(2)	0.0(2)	−0.4(2)
N4	3.2(2)	4.2(2)	3.4(2)	−0.3(2)	0.8(2)	0.7(2)
C1	3.0(2)	4.3(3)	3.6(3)	0.0(2)	−0.9(2)	0.6(2)
C2	3.5(3)	3.4(3)	3.4(2)	−0.3(2)	−0.9(2)	−0.4(2)
C3	3.6(3)	2.7(2)	4.5(3)	−0.7(2)	−0.2(2)	0.5(2)
C4	2.7(3)	3.4(3)	3.8(3)	−0.8(2)	0.2(2)	0.8(2)
C5	4.7(3)	3.0(3)	4.8(3)	−0.8(2)	−1.0(3)	−1.1(2)
C6	3.6(3)	4.6(3)	4.5(3)	−0.2(2)	−1.4(3)	−1.0(3)
C7	2.3(2)	5.2(3)	4.1(3)	0.8(2)	0.5(2)	−1.3(3)
C8	3.0(3)	4.8(3)	4.6(3)	−0.1(2)	1.5(2)	−0.6(3)
Cl1	4.89(7)	6.77(8)	3.08(6)	0.81(6)	−0.08(7)	−0.36(7)
Cl2	4.12(6)	3.55(5)	5.50(8)	−0.68(5)	0.51(7)	0.47(7)
Cl3	5.00(9)	7.06(11)	10.65(16)	−0.88(8)	0.56(10)	−0.91(11)
O1	10.0(4)	7.5(3)	5.8(3)	3.1(3)	−2.3(3)	−1.4(3)

The Co−S bond lengths of 2.241 and 2.243 Å are identical to those observed [13] in the complex *u-cis*-$[Co(ete)(NO_2)Cl]^+$ (where ete = 3,7-dithianonane-1,9-diamine). The corresponding bond lengths in 6,9-diaza-2,13-dithiatetradecane-5,10-dicarboxylato cobalt(III) monocation is 2.267 Å.[14] In this complex the thioethers are terminal ligators of the hexadentate ligand and therefore not forced closer to the cobalt atom by surrounding chelate rings. All four Co−N distances are substantially equal (mean: 1.973 Å) and in accordance with results obtained for other cobalt(III) chelates with terminal amino groups. The C−S and C−N distances are all found to assume normal values. The C−C distances (mean: 1.493 Å) are somewhat shorter than usually observed. Similar short bonds have been found in a number of free and complexed cyclic ethers and thioethers.

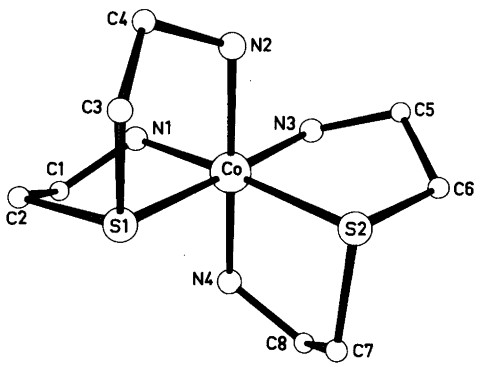

Fig. 1. An ORTEP drawing of the $(-)_D$-$\Delta(\lambda\lambda\lambda\lambda)$-*u-fac*$[Co(daes)_2]^{3+}$ ion illustrating the atomic labelling.

It has been established that the $(-)_D$ catoptric form of the complex has the absolute configuration Δ. This result thus confirms the tentative assignment of the absolute configuration given on the basis of the circular dichroism spectrum of the isomer.[1] The four individual five-membered chelate rings take λ

Table 4. Bond lengths (Å) in $[Co(daes)_2]Cl_3$. Standard deviations in parentheses.

Co−S1	2.244(1)	Co−S2	2.248(1)
Co−N1	1.973(4)	Co−N3	1.965(4)
Co−N2	1.968(4)	Co−N4	1.988(5)
N1−C1	1.480(7)	N3−C5	1.475(8)
C1−C2	1.514(8)	C5−C6	1.500(8)
C2−S1	1.830(6)	C6−S2	1.831(6)
S1−C3	1.826(6)	S2−C7	1.806(6)
C3−C4	1.487(7)	C7−C8	1.489(8)
C4−N2	1.482(7)	C8−N4	1.481(7)

Table 5. Selected bond angles (°).

S1 – Co – S2	90.19(5)	N1 – Co – N3	95.0(2)
S1 – Co – N1	87.1(2)	S2 – Co – N3	87.9(1)
S1 – Co – N2	87.6(1)	S2 – Co – N4	87.5(1)
S1 – Co – N4	92.4(1)	S2 – Co – N2	92.6(1)
N1 – Co – N2	90.8(2)	N3 – Co – N4	90.7(2)
N1 – Co – N4	89.2(2)	N3 – Co – N2	89.2(2)
Co – N1 – C1	113.5(3)	Co – N3 – C5	112.5(3)
N1 – C1 – C2	110.0(4)	N3 – C5 – C6	110.9(5)
C1 – C2 – S1	109.8(4)	C5 – C6 – S2	108.5(4)
C2 – S1 – Co	99.5(2)	C6 – S2 – Co	98.4(2)
C2 – S1 – C3	102.2(2)	C6 – S2 – C7	102.1(3)
S1 – C3 – C4	109.5(3)	S2 – C7 – C8	110.4(4)
C3 – C4 – N2	109.2(4)	C7 – C8 – N4	108.8(4)
C4 – N2 – Co	115.4(3)	C8 – N4 – Co	115.7(4)
Dihedral angles			
N1 – C1 – C2 – S1	– 44.8(5)	N3 – C5 – C6 – S2	– 49.4(6)
N2 – C4 – C3 – S1	– 50.7(5)	N4 – C8 – C7 – S2	– 51.5(5)

conformations. So the isomer studied here can be characterized as $(-)_D$-$\Delta(\lambda\lambda\lambda\lambda)$-*u-fac*-$[Co(daes)_2]^{3+}$. The structure of the closely analogous complex $(-)_D$-*u-fac*-$[Co(dien)_2]^{3+}$ (as a salt of $[Co(CN)_6]^{3-}$) contains the two conformers $\Delta(\lambda\lambda\lambda\lambda)$ and $\Delta(\lambda\delta\lambda\delta)$.[15] This may reflect a smaller degree of conformational rigidity and a smaller energy difference between conformers of the dien complex relative to the daes complex.

The packing in the crystal is determined by hydrogen bonding. In the stereo pair shown in Fig. 2 the hydrogen bonds are illustrated. Two of

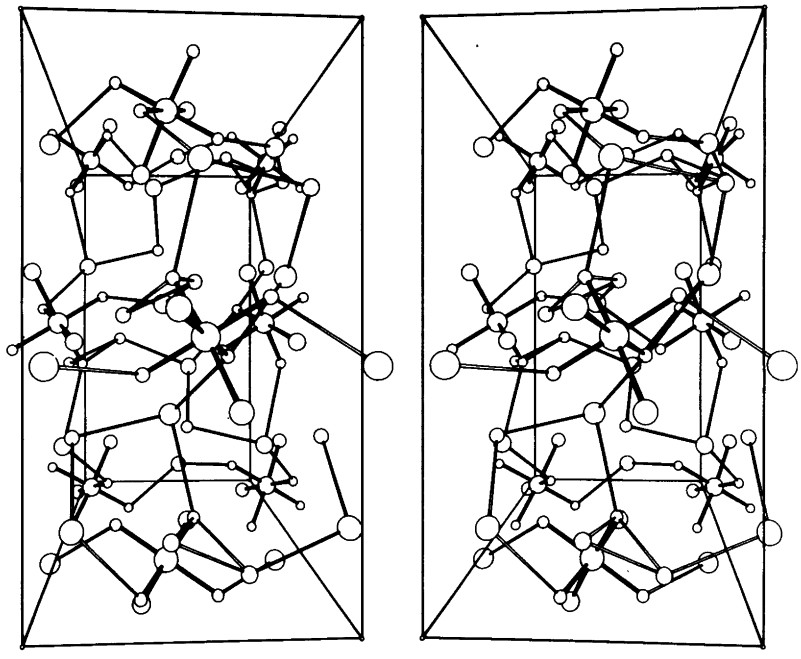

Fig. 2. Stereo pair of the packing viewed along the *c* axis.

the chloride ions Cl1 and Cl2 are hydrogen bonded to the complex ion. Thus Cl1 bridges N2 and N4 of different cations so that chains along the direction of the *c* axis are formed. The distances are Cl1 – N2 3.175 Å and Cl1 – N4 3.133 Å. The two Cl1 – H – N angles are both 162°. Correspondingly Cl2 connects the two nitrogen atoms N1 and N3 (lying in the CoS_2-plane) from different cations making chains along the *a* axis. These interactions are slightly weaker (Cl2 – N1 3.218 Å, Cl2 – N3 3.316 Å). The third chloride is hydrogen bonded to N3, Cl3 – N3 3.326 Å.

The ordered water molecule in the structure has interactions to Cl2 and Cl3 (Cl2 – O1 3.202 Å, Cl3 – O1 3.122 Å). The sites for the disordered water molecules are in an area where it is possible to form hydrogen bonds to Cl1, Cl3, and N1. The distance between the two sites O3 and O4 is 3.25 Å making it possible to fit more than one water molecule into this area.

The efficient hydrogen bonding network described above may account for the spontaneous resolution that occurs when the racemic compound crystallizes. One could imagine that a hypothetical crystal structure which contained both enantiomers in the unit cell would have higher free energy than the crystal structure described here. From solutions of racemic mixtures of the salt at various different temperatures were grown large single crystals (0.01 – 1 cm^3). These crystals were found to have circular dichroism corresponding to the completely resolved samples or at some instances to consist of twinned crystals.

REFERENCES

1. Searle, G. H. and Larsen, E. *Acta Chem. Scand. A 30* (1976) 143.
2. Keene, F. R. and Searle, G. H. *Inorg. Chem. 11* (1972) 148.
3. Saito, Y., Nakatsu, K., Shiro, M. and Kuroya, H. *Acta Crystallogr. 8* (1955) 729.
4. Hawkins, C. J. *Absolute Configuration of Metal Complexes*, Wiley-Interscience, N.Y. 1971.
5. Lehnert, P. G. *J. Appl. Crystallogr. 8* (1975) 568.
6. Johnson, C. K. *ORTEP, A Fortran Ellipsoid Plot Program for Crystal Structure Illustrations*, Report ORNL-3794, Second Rev., Oak Ridge National Laboratory, Oak Ridge 1970.
7. Stewart, J. M. *et al. The X-Ray System 1972*, Technical Report Tr-192, Computer Science Center, University of Maryland, College Park 1972.
8. Nielsen, K. *Acta Crystallogr. A 31* (1977) 1009.
9. Cromer, D. T. and Mann, J. B. *Acta Crystallogr. A 24* (1968) 321.
10. Stewart, R. F., Davidson, E. R. and Simpson, W. T. *J. Chem. Phys. 42* (1965) 3175.
11. Cromer, D. T. and Liberman, D. *J. Chem. Phys. 53* (1970) 1891.
12. Hamilton, W. C. *Acta Crystallogr. 18* (1965) 502.
13. Murray-Rust, J. and Murray-Rust, P. *Acta Crystallogr. B 29* (1973) 2606.
14. Geue, R. and Snow, M. *Acta Crystallogr. B 33* (1977) 70.
15. Konno, M., Marumo, F. and Saito, Y. *Acta Crystallogr. B 29* (1973) 739.

Received February 24, 1978.