Acta Cryst. (1970). **B26**, 1492

The Crystal Structure of $(-)_{589}$Tris(+*trans*-1,2-diaminocyclohexane)cobalt(III) Chloride Pentahydrate, $(-)_{589}[Co(+chxn)_3]Cl_3.5H_2O$

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(*Received* 12 *December* 1969)

The crystal structure of $(-)_{589}$tris(+*trans*-1,2-diaminocyclohexane)cobalt(III) chloride pentahydrate, $(-)_{589}[Co(+chxn)_3]Cl_3.5H_2O$, has been determined from three-dimensional X-ray data collected by the photographic method. The compound forms hexagonal crystals with $a=12{\cdot}34$, $c=33{\cdot}52$ Å and $Z=6$, in space group $P6_1$. The structure has been refined by least-squares methods with anisotropic temperature factors to a final residual R of 0·110 using 1123 observed reflexions. The complex ion, $(-)_{589}[Co(+chxn)_3]^{3+}$, has approximately the symmetry D_3. The central cobalt atom is bonded nearly octahedrally to six nitrogen atoms of the ligand molecules. All the bond lengths and angles are normal. The complex ion takes the *lel* form. The absolute configuration of the complex ion is designated as $\Lambda\delta\delta\delta$ according to the IUPAC convention.

Introduction

As part of a series of investigations on the absolute configuration of tris(diamine)cobalt(III) complexes, the crystal structure of $(-)_{589}$tris(+*trans*-1,2-diaminocyclohexane)cobalt(III) chloride pentahydrate, $(-)_{589}[Co(+chxn)_3]Cl_3.5H_2O$, was determined (Saito, 1968). Although this complex is laevo-rotatory for the Na D line, its optical rotatory dispersion curve and circular dichroism spectra resemble those of $(+)_{589}[Co(en)_3]^{3+}$ (Woldbye, 1963), but are somewhat different from those of the closely related $(-)_{589}$tris(+*trans*-1,2-diaminocyclopentane)cobalt(III) ion, $(-)_{589}[Co(+cptn)_3]^{3+}$. In order to determine the relation between the geometry and the optical properties of these complexes, the crystal structure and absolute configuration of the complex ion,

$$(-)_{589}[Co(+chxn)_3]^{3+},$$

have been determined.

Experimental

$(-)_{589}$Tris(+*trans*-1,2diaminocyclohexane)cobalt(III) chloride was prepared according to the method described by Jaeger & Bijkerk (1937). The resolution was carried out with (+)-tartaric acid. Orange-red crystals of the pentahydrate were obtained as hexagonal bipyramids by slow evaporation of an aqueous solution. They belong to the hexagonal system, with unit-cell dimensions $a=12{\cdot}342\pm0{\cdot}005$, $c=33{\cdot}517\pm0{\cdot}011$ Å. The observed systematic absences for $00l$, $l\neq6n$, indicate the possible space group $P6_1$ or $P6_5$. There are six formula units $Co(C_6H_{14}N_2)_3Cl_3\ 5H_2O$ in the unit cell ($D_x=1{\cdot}347$ g.cm^{-3}, $D_m=1{\cdot}341$ g.cm^{-3}). The crystals gradually lose the water of crystallization in air. For this reason two crystal specimens were used to collect three-dimensional intensity data. They were shaped into spheres with radii of approximately 0·08 and 0·24 mm respectively. Integrated equi-inclination Weissenberg photographs were taken around the a axis up to the eighth layer with Cu $K\alpha$ radiation. Intensities were measured visually and corrected for Lorentz and polarization factors. The corrections for absorption were applied only to the data obtained from the larger crystal, for which μr was 1·9. Reflexions with indices hkl and $\bar{h}\bar{k}\bar{l}$ were treated as independent because of the anomalous scattering of Cu $K\alpha$ radiation by the cobalt atoms. The photographs taken around the a axis were sufficient for the interlayer scaling, since each layer contains some reflexions equivalent to those on other layers.

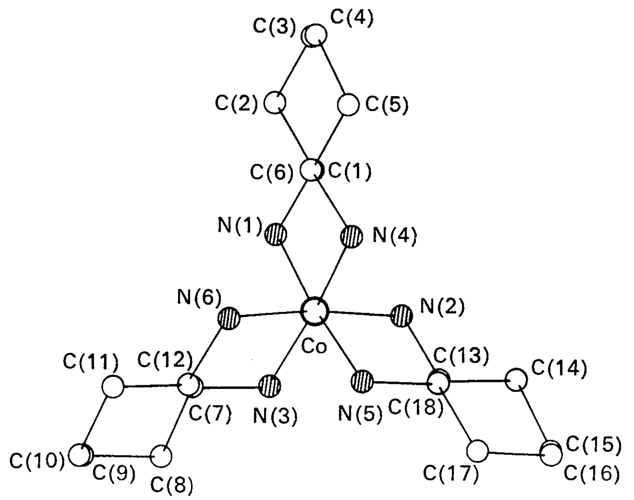

Fig. 1. The complex ion in $(-)_{589}[Co(+chxn)_3]Cl_3\ 5H_2O$ crystals viewed along the normal to the plane N(1)N(2)N(3).

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Determination of the structure

The structure was solved by the heavy atom method. The three-dimensional Patterson function revealed that the arrangement of the complex cations in the crystal is similar to that in $(-)_{589}[Co(cptn)_3]Cl_3.4H_2O$, the structure of which was determined by Ito, Marumo & Saito (1968). This structural similarity is of great help in obtaining the positions of the heavy atoms. In the latter crystal the chemical units, $[Co(+cptn)_3]Cl_3$ have approximately a hexagonal close-packed arrangement, the Co atoms being nearly on the threefold screw axes. If the structure is based on ideal hexagonal close-packing of $[Co(+cptn)_3]Cl_3$ units, the period along the c axis should be one third of the value actually observed, and the threefold screw axes should change to the threefold axis of rotation. This type of subcell was actually observed in the Patterson maps of the crystal under investigation. The mean locations of the cobalt and chloride ions in the idealized structure were easily found from the maps. In order to avoid false symmetry in the electron density maps synthesized with the phases determined from the heavy atoms only, it is essential to find the small deviations in the atomic positions from the average positions in the three subcells. The shifts parallel to the x,y plane were obtained by close examination of the Patterson maps; the values of the z parameters of the chloride ions relative to the cobalt atom found in the structure of $(-)_{589}[Co(+cptn)_3]Cl_3.4H_2O$ were adopted for the z parameters of the starting model of the present structure, since it was impossible to find the z parameters of the chloride ions relative to that of the cobalt atom from the Patterson maps. The positions of the six nitrogen atoms were obtained immediately from the three-dimensional Fourier synthesis, phased by the heavy atoms. All the carbon atoms and the oxygen atoms of the water molecules were found by successive Fourier syntheses. In these Fourier and difference Fourier syntheses, five well-defined peaks appeared, which could be interpreted as the oxygen atoms of the water molecules of crystallization. Although this compound has been reported as the tetrahydrate (Jaeger & Bijkerk, 1937), the five oxygen atoms which appeared to be present in the difference maps were tentatively included in the calculation of the structure factors. This was ratified at a later stage by the refinement of the structure.

The structure was then refined by a block-diagonal three-dimensional least-squares program (Okaya & Ashida, 1967) with isotropic temperature factors. Unit weight was given to all the observed reflexions, and 0·5 to non-observed reflexions. The atomic scattering factors of neutral atoms were used (*International Tables for X-ray Crystallography*, 1962). After five cycles the R value was reduced to 0·18 for all the 1123 observed reflexions. Although a difference Fourier synthesis was calculated at this stage to locate the hydrogen atoms, they could not all be found with certainty. All hydrogen atoms were therefore neglected in subsequent calculations.

Further cycles of refinement with the use of anisotropic temperature factors were carried out on two enantiomorphic structures taking the anomalous dispersion effect into consideration. The R value was reduced to 0·133 for the space group $P6_1$ after two cycles, whereas the value was 0·158 for $P6_5$ indicating $P6_1$ to be the true space group of the crystal. This result for the absolute structure was confirmed by a comparison of the observed relations between the hkl and $hk\bar{l}$ reflexions with the calculated intensity relations (Table 1). The structure was refined in space group $P6_1$ to an R value of 0·110.

Table 1. *Observed and calculated intensity relations between some hkl and hk$\bar{l}$ reflexions of* $(-)_D$-*tris*(+*trans*-1,2-*diaminocyclohexane*)*cobalt*(III) *chloride pentahydrate*

h	k	l	$\lvert F_{calc}(hkl)\rvert^2$	Observed	$\lvert F_{calc}(hk\bar{l})\rvert^2$
0	2	2	100	<	196
0	2	3	121	<	900
0	2	4	784	<	1600
0	3	1	36	<	256
0	4	2	169	<	625
0	5	1	841	>	169

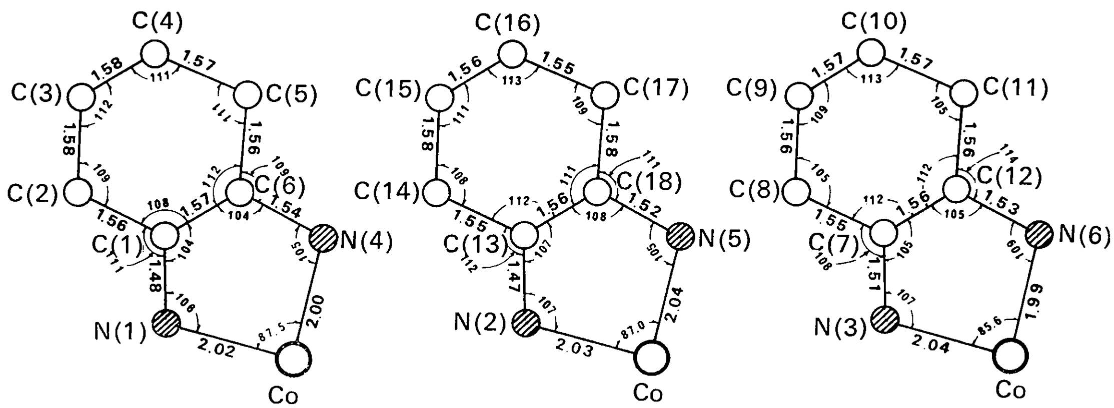

Fig. 2. Bond distances (Å) and angles (degrees) in the complex ion $(-)_{589}[Co(+chxn)_3]^{3+}$.

Table 1 (*cont.*)

0	5	3	1521	>	576
0	6	1	1600	<	2304
0	6	5	361	>	25
0	6	7	2025	<	3364
0	6	8	3249	>	2500
0	7	1	1156	<	1936

Since the temperature factors obtained for the water molecules were fairly large in comparison with those of the other constituent atoms, there remained a possibility that the number of water molecules per chemical unit was four rather than five as described in the literature, and that these four water molecules were distributed statistically over five positions. With the intention of resolving this point, the populations of the oxygen atoms were refined as well as positional and thermal parameters with a version (Sakurai, Nakatsu & Iwasaki, 1967) of the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962). The calculation gave 5·29 for the total population which indicates definitely that there are five water molecules in the chemical unit. The observed crystal densities also indicate five water molecules per chemical unit: the calculated densities for the tetra and pentahydrates are 1·307 and 1·347 g.cm^{-3} respectively, whereas the observed value is 1·341 ± 0·010 g.cm^{-3} at 15°C.

The final positional parameters and temperature factors are given in Tables 2 and 3 respectively with their estimated standard deviations. The calculated and observed structure amplitudes are listed in Table 4.

Table 2. *Final positional parameters and their standard deviations* (*in parentheses*)

	x	y	z
Co	0·7166 (4)	0·3690 (4)	0·0798 (2)
Cl(1)	0·4334 (9)	0·4815 (10)	0·0661 (5)
Cl(2)	0·1109 (8)	0·6535 (9)	0·0715 (5)
Cl(3)	0·6050 (9)	0·9716 (8)	0·1005 (3)
N(1)	0·6000 (17)	0·2119 (20)	0·0494 (7)
N(2)	0·6911 (18)	0·4749 (20)	0·0386 (8)
N(3)	0·8662 (19)	0·3881 (19)	0·0483 (7)
N(4)	0·5699 (19)	0·3378 (22)	0·1133 (8)
N(5)	0·8184 (20)	0·5295 (17)	0·1115 (6)
N(6)	0·7609 (19)	0·2775 (19)	0·1196 (7)
C(1)	0·4722 (22)	0·1872 (26)	0·0584 (8)
C(2)	0·3719 (25)	0·0503 (27)	0·0468 (9)
C(3)	0·2387 (28)	0·0237 (28)	0·0613 (10)
C(4)	0·2342 (33)	0·0430 (34)	0·1076 (10)
C(5)	0·3413 (25)	0·1756 (27)	0·1208 (8)
C(6)	0·4707 (25)	0·2002 (23)	0·1051 (8)
C(7)	0·8970 (25)	0·2923 (23)	0·0645 (9)
C(8)	0·0290 (24)	0·3277 (25)	0·0493 (8)
C(9)	0·0582 (31)	0·2301 (36)	0·0687 (11)
C(10)	0·0573 (31)	0·2433 (37)	0·1153 (13)
C(11)	0·9257 (27)	0·2104 (28)	0·1325 (8)
C(12)	0·8926 (23)	0·3023 (23)	0·1107 (7)
C(13)	0·7867 (27)	0·6061 (24)	0·0464 (9)
C(14)	0·7585 (32)	0·6983 (28)	0·0236 (10)
C(15)	0·8632 (32)	0·8353 (28)	0·0348 (8)
C(16)	0·8675 (32)	0·8562 (28)	0·0808 (13)
C(17)	0·8914 (29)	0·7622 (25)	0·1047 (10)
C(18)	0·7905 (24)	0·6246 (22)	0·0925 (8)
O(1)	0·5610 (32)	0·8921 (33)	0·0063 (9)
O(2)	0·3383 (25)	0·6484 (26)	0·0279 (6)
O(3)	0·5617 (31)	0·7205 (28)	0·1431 (9)
O(4)	0·3334 (26)	0·8051 (30)	0·1322 (8)
O(5)	0·3083 (57)	0·5542 (39)	0·1342 (13)

Table 3. *Final thermal parameters and their standard deviations* (*in parantheses*)

The values have been multiplied by 10^4 and refer to the expression: $\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\right]$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Co	46 (4)	55 (5)	7 (1)	60 (8)	0 (3)	−3 (2)
Cl(1)	89 (11)	138 (13)	53 (4)	186 (21)	8 (10)	17 (11)
Cl(2)	60 (9)	79 (11)	46 (3)	22 (15)	16 (9)	−31 (9)
Cl(3)	126 (11)	78 (9)	17 (1)	100 (17)	−19 (6)	−6 (5)
N(1)	8 (18)	80 (25)	12 (3)	21 (34)	−6 (11)	−28 (13)
N(2)	11 (20)	43 (24)	12 (3)	−19 (36)	−1 (13)	7 (14)
N(3)	32 (21)	37 (23)	12 (3)	11 (36)	21 (13)	19 (14)
N(4)	34 (22)	74 (26)	12 (3)	69 (43)	17 (14)	2 (16)
N(5)	104 (25)	17 (19)	3 (2)	37 (37)	−30 (12)	0 (10)
N(6)	61 (24)	55 (23)	5 (2)	79 (40)	12 (12)	7 (12)
C(1)	16 (25)	72 (31)	7 (3)	−12 (44)	−1 (14)	7 (16)
C(2)	33 (28)	93 (34)	7 (4)	28 (52)	−9 (16)	−23 (17)
C(3)	74 (34)	65 (34)	13 (4)	34 (56)	5 (19)	−11 (19)

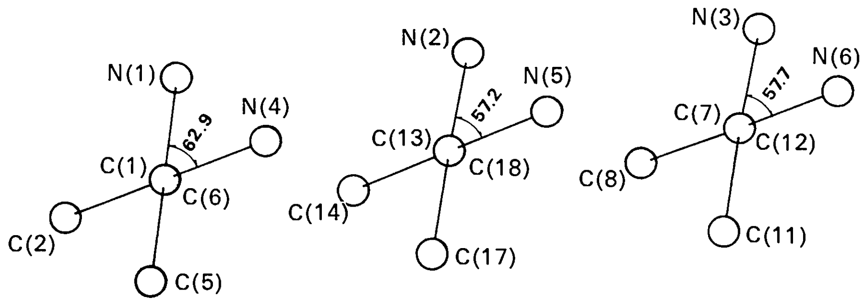

Fig. 3. Newman projections along the bonds C(6)–C(1), C(12)–C(7) and C(18)–C(13). The numbers indicate the values of the torsional angles.

Table 3 (*cont.*)

C(4)	138 (43)	136 (44)	7 (4)	149 (76)	30 (22)	21 (22)
C(5)	56 (29)	94 (33)	7 (3)	108 (53)	8 (15)	7 (16)
C(6)	62 (29)	34 (26)	10 (4)	54 (47)	12 (16)	−6 (15)
C(7)	65 (29)	36 (26)	12 (4)	85 (49)	16 (16)	25 (16)
C(8)	53 (28)	73 (30)	5 (3)	91 (49)	24 (15)	12 (15)
C(9)	87 (38)	163 (50)	17 (5)	169 (73)	34 (21)	70 (26)
C(10)	64 (36)	162 (51)	21 (6)	136 (72)	6 (24)	24 (30)
C(11)	104 (35)	138 (39)	3 (3)	203 (65)	7 (16)	18 (17)
C(12)	70 (27)	60 (26)	2 (2)	120 (47)	15 (13)	6 (14)
C(13)	85 (33)	26 (26)	10 (4)	69 (52)	−19 (18)	−14 (16)
C(14)	154 (44)	58 (34)	10 (4)	79 (65)	−32 (21)	24 (19)
C(15)	158 (44)	76 (35)	3 (3)	95 (65)	−24 (19)	6 (17)
C(16)	137 (43)	64 (34)	16 (5)	86 (66)	−13 (27)	27 (23)
C(17)	106 (37)	21 (27)	13 (4)	25 (53)	−21 (21)	−7 (17)
C(18)	108 (28)	21 (24)	13 (4)	43 (42)	−34 (16)	13 (15)
O(1)	245 (51)	208 (48)	24 (4)	190 (88)	17 (23)	−41 (22)
O(2)	142 (35)	169 (35)	13 (2)	91 (61)	14 (15)	1 (16)
O(3)	374 (50)	315 (41)	38 (4)	466 (80)	138 (24)	91 (21)
O(4)	205 (37)	349 (51)	18 (4)	348 (74)	23 (20)	61 (24)
O(5)	804 (131)	325 (68)	22 (7)	662 (173)	−19 (55)	32 (38)

Table 4. *Observed and calculated structure amplitudes*

[illegible]

Table 4 (*cont.*)

[illegible]

Discussion

The complex cation has approximate symmetry D_3, as shown in Fig. 1. In the complex each ligand molecule is coordinated to the central cobalt atom by its nitrogen atoms, which acts as a bidentate ligand. All the C–C bonds in the chelate rings are nearly parallel to the threefold axis of rotation, namely the complex ion has *lel* conformation. The interatomic distances and bond angles within the complex ion are given in Table 5 with their standard deviations and illustrated in Fig. 2. The cobalt atom has a distorted octahedral coordination of nitrogen atoms with distances ranging from 1·99 to 2·04 Å. These values are quite normal in tris(diamine) cobalt(III) complexes, *e.g.* $(+)_{589}[Co(en)_3]Cl_3.H_2O$ (Iwata, Nakatsu & Saito, 1969), $(-)_{589}[Co(-pn)_3]Br_3$ (Iwasaki & Saito, 1966) and $(-)_{589}[Co(tn)_3]Br_3.H_2O$ (Nomura, Marumo & Saito, 1969). The C–C and C–N distances are in agreement with the single-bond distances within experimental error. The N–Co–N angles in the five-membered chelate rings are all less than 90° owing to the formation of the chelate rings. Consequently, the triangle N(1)N(2)N(3) is rotated about the threefold axis by about 5·5° with respect to the triangle N(4)N(5)N(6) from the position expected for a regular octahedron. This is a common feature of tris(diamine) cobalt(III) complexes with five-membered chelate rings. Newman projections along the bonds C(6)–C(1), C(12)–C(7) and C(18)–C(13) are shown in Fig. 3. The mean value of the dihedral angle between the planes N(1)C(1)C(6) and N(4)C(6)C(1) and the corresponding angles in the other two chelate rings is about 59·3°, which is almost identical with the value expected in a free *trans*-1,2-diaminocyclohexane molecule. Thus the molecule seems to be only a little strained by the formation of the chelate ring. The cyclohexane ring has a chair conformation. All the bond distances and angles within the six-membered ring are quite normal and agree well with those observed for other related compounds (Shimada, Okaya & Nakamura, 1955).

Table 5. *Interatomic distances and bond angles in the complex with their standard deviations*

	Distance
Co—N(1)	2·021 (20) Å
Co—N(2)	2·031 (28)
Co—N(3)	2·035 (25)
Co—N(4)	1·998 (26)
Co—N(5)	2·036 (19)
Co—N(6)	1·994 (27)
N(1)—C(1)	1·480 (37)
N(2)—C(13)	1·473 (29)
N(3)—C(7)	1·513 (44)
N(4)—C(6)	1·543 (30)
N(5)—C(18)	1·518 (41)
N(6)—C(12)	1·525 (38)
C(1)—C(2)	1·563 (37)
C(2)—C(3)	1·583 (48)
C(3)—C(4)	1·576 (48)
C(4)—C(5)	1·568 (39)
C(5)—C(6)	1·562 (44)
C(6)—C(1)	1·574 (40)
C(7)—C(8)	1·546 (42)
C(8)—C(9)	1·562 (60)
C(9)—C(10)	1·572 (57)
C(10)–C(11)	1·574 (52)
C(11)–C(12)	1·564 (50)
C(12)–C(7)	1·559 (38)
C(13)–C(14)	1·548 (53)
C(14)–C(15)	1·575 (37)
C(15)–C(16)	1·562 (51)
C(16)–C(17)	1·554 (56)
C(17)–C(18)	1·577 (32)
C(18)–C(13)	1·560 (41)

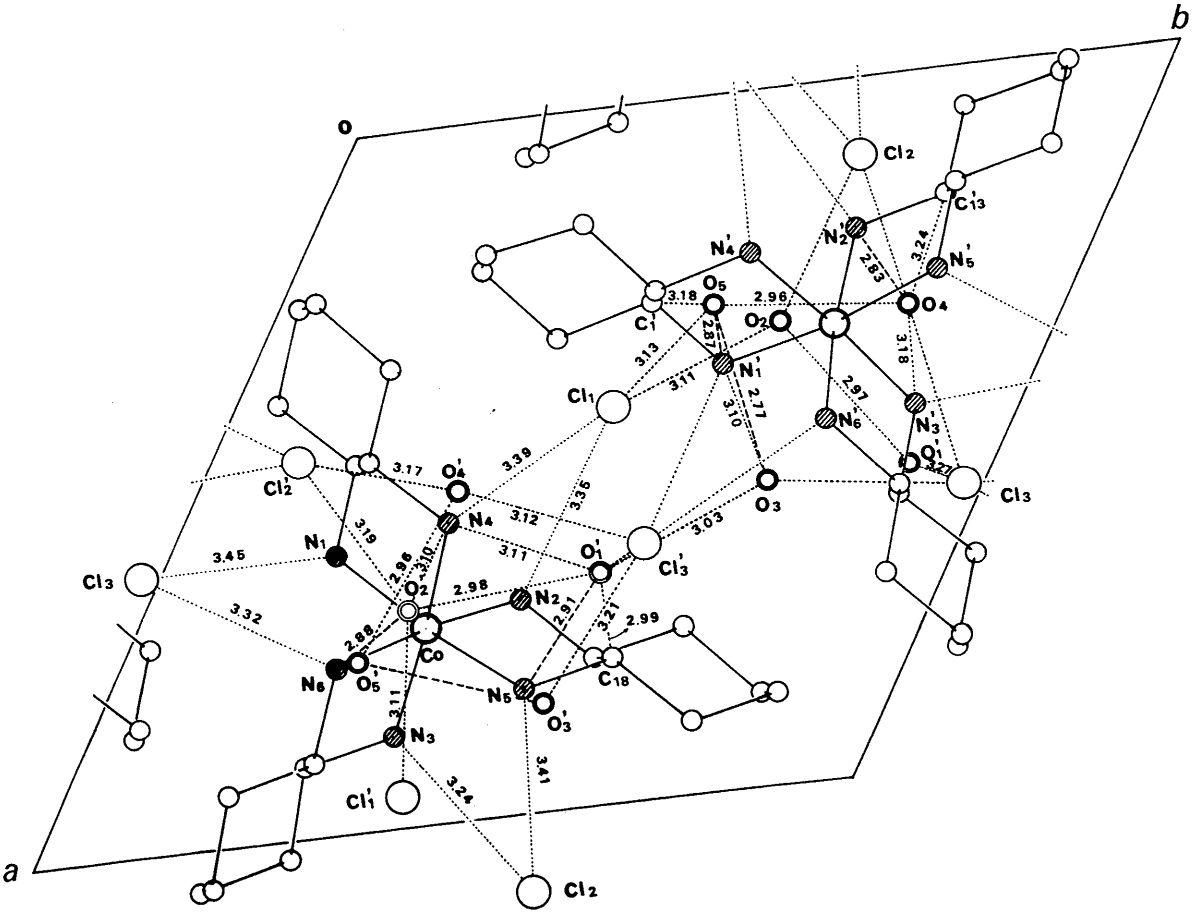

Fig. 4. Projection of the structure along the *c* axis. Only one-third of the repeating unit along the *c* axis is drawn. Dashed lines indicate probable hydrogen bonds.

Table 5 (*cont.*)

	Angle
N(1)—Co—N(4)	87·5 (0·9)°
N(2)—Co—N(5)	87·0 (0·9)
N(3)—Co—N(6)	85·6 (1·0)
N(1)—Co—N(5)	174·0 (0·8)
N(2)—Co—N(6)	174·0 (1·1)
N(3)—Co—N(4)	174·9 (1·1)
Co—N(1)—C(1)	105·6 (1·8)
Co—N(2)—C(13)	106·8 (1·6)
Co—N(3)—C(7)	106·9 (1·7)
Co—N(4)—C(6)	104·8 (1·7)
Co—N(5)—C(18)	105·4 (1·7)
Co—N(6)—C(12)	108·7 (1·6)
N(1)—C(1)—C(2)	111·0 (2·5)
N(1)—C(1)—C(6)	104·3 (2·1)
C(2)—C(1)—C(6)	108·3 (2·0)
C(1)—C(2)—C(3)	109·1 (2·7)
C(2)—C(3)—C(4)	112·5 (2·6)
C(3)—C(4)—C(5)	111·2 (2·4)
C(4)—C(5)—C(6)	110·7 (2·7)
N(4)—C(6)—C(1)	104·3 (1·9)
N(4)—C(6)—C(5)	108·9 (2·4)
C(5)—C(6)—C(1)	112·3 (2·4)
N(2)—C(13)-C(14)	111·7 (2·6)
N(2)—C(13)-C(18)	106·6 (2·0)
C(14)-C(13)-C(18)	112·5 (2·8)
C(13)-C(14)-C(15)	107·9 (2·7)
C(14)-C(15)-C(16)	110·7 (2·4)
C(15)-C(16)-C(17)	112·9 (3·1)
C(16)-C(17)-C(18)	109·2 (2·6)
N(5)—C(18)-C(13)	107·7 (2·4)
N(5)—C(18)-C(17)	111·2 (2·2)
C(17)-C(18)-C(13)	111·3 (2·0)
N(3)—C(7)—C(8)	107·6 (2·1)
N(3)—C(7)—C(12)	105·2 (2·4)
C(8)—C(7)—C(12)	112·3 (2·3)
C(7)—C(8)—C(9)	105·1 (2·6)
C(8)—C(9)—C(10)	108·5 (3·5)
C(9)—C(10)-C(11)	113·3 (3·0)
C(10)-C(11)-C(12)	104·7 (2·4)
N(6)—C(12)-C(7)	104·8 (2·1)
N(6)—C(12)-C(11)	113·7 (2·2)
C(11)-C(12)-C(7)	111·9 (2·6)

The crystal structure projected along the *c* and *a* axes is shown in Figs. 4 and 5 respectively. In Fig. 4 only one-third of the unit-cell content is illustrated. The stacking of the complex cations is related to hexagonal closest packing. The central metal atom is a little off the threefold screw axis. The pseudo threefold axis of the complex ion is inclined at about 8° to the *c* axis. Intermolecular contacts less than 3·5 Å are given in Table 6. Each chloride ion is surrounded by two nitrogen atoms on one side and by two or three water molecules on the other. There may be weak hydrogen bonds between Cl and N atoms similar to those found in the structure of $(+)_{589}[Co(en)_3]Cl_3.H_2O$ (Iwata, Nakatsu & Saito, 1969), because the relative positions of the Cl^- ions around to the complex ions are very similar to those found in the ethylenediamine analogue. All the oxygen atoms of the water molecules possess irregular coordinations. In particular, O(5) is surrounded only by carbon atoms at fairly large distances on one side. This explains O(5) having the largest temperature factor of the five oxygen atoms. Hydrogen bonds seem to exist between the water molecules and nitrogen atoms and also between the water molecules, which are illustrated in Figs. 4 and 5. Fairly short C···O distances are observed as in the case of the $(-)_{546}$*cis*-*β*-dinitro-(L-3,8-dimethyltriethylenetetramine)cobalt(III) perchlorate structure (Ito, Marumo & Saito, 1970): O(1)···C(18), 2·99; O(4)···C(13), 3·24; O(5)···C(1), 3·18 Å, while the sum of the van der Waals radii is 3·40 Å. These short contacts might indicate the existence of the weak C–H...O bonds suggested by Sutor (1963).

Table 6. *Intermolecular distances less than* 3·5 Å *with their standard deviations in parantheses*

	Distance	Symmetry operation applied to second atom
Cl(1)-N(2)	3·35 (3) Å	1
-N(4)	3·39 (3)	1
-O(2)	3·11 (4)	1
-O(5)	3·13 (4)	1
Cl(2)-N(3)	3·24 (2)	2
-N(5)	3·41 (3)	2
-O(2)	3·19 (4)	1
-O(4)	3·17 (3)	1
Cl(3)-N(1)	3·45 (3)	3
-N(6)	3·32 (2)	3
-O(1)	3·27 (3)	1
-O(3)	3·21 (4)	1
-O(4)	3·12 (3)	1
O(1)-Cl(3)	3·27 (3)	1
-O(2)	2·98 (4)	1
-O(3)	3·03 (6)	4
-N(4)	3·11 (4)	4
-N(5)	2·91 (4)	4
-C(17)	3·48 (4)	4
-C(18)	2·99 (4)	4
O(2)-Cl(1)	3·11 (4)	1
-Cl(2)	3·19 (4)	1
-O(1)	2·98 (4)	1
-N(4)	3·10 (3)	4
-N(5)	3·47 (3)	4
-N(6)	2·88 (3)	4
O(3)-Cl(3)	3·21 (4)	1
-O(1)	3·03 (6)	5
-O(4)	3·48 (6)	1
-O(5)	2·77 (4)	1
-N(1)	3·10 (4)	6
-N(3)	3·46 (3)	6
O(4)-Cl(2)	3·17 (3)	1
-Cl(3)	3·12 (3)	1
-O(3)	3·48 (6)	1
-O(5)	2·96 (6)	1
-N(2)	2·83 (4)	6
-N(3)	3·18 (4)	6
-C(13)	3·24 (4)	6
O(5)-Cl(1)	3·13 (4)	1
-O(3)	2·77 (4)	1
-O(4)	2·96 (6)	1
-N(1)	2·87 (4)	6
-N(2)	3·43 (3)	6
-C(1)	3·18 (4)	6

Key to symmetry operations

1	x, y, z
2	$x-1, y, z$
3	$x, y+1, z$
4	$y, -x+y+1, z-\frac{1}{6}$
5	$x-y+1, x, z+\frac{1}{6}$
6	$x-y, x, z+\frac{1}{6}$

The absolute configuration of the complex ion, $(-)_{589}$–$[Co(+chxn)_3]^{3+}$, can be designated as $\Lambda\delta\delta\delta$ according to the IUPAC convention (1968). The similarity of the optical rototory dispersion and circular dichroism of this complex cation and those of $(+)_{589}[Co(en)_3]^{3+}$ corresponds to the close similarity in the arrangements of the three five-membered chelate rings around the central cobalt atom.

The Fourier synthesis program used was *ANSFR*-I written by H. Iwasaki. The entire calculations were carried out on a HITAC 5020 E computer at the Computer Centre of this University. Part of the cost of this investigation was met by a Scientific Research Grant of the Ministry of Education, to which the authors' thanks are due.

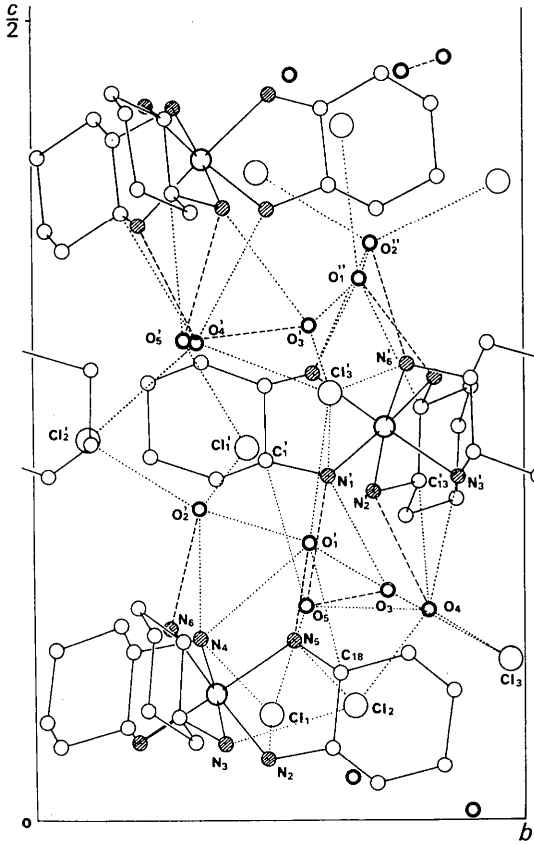

Fig. 5. Projection of the structure along the *a* axis. Dashed lines indicate probable hydrogen bonds.

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