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trans*-[Theophyllinatochlorobis(ethylenediamine)cobalt(III)] Chloride Dihydrate

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[Co(C₂N₂H₈)₂Cl(C₇N₄O₂H₇)]Cl·2H₂O is triclinic, space group $P\bar{1}$, with $a = 10.034$ (3), $b = 10.711$ (4), $c = 9.499$ (4) Å, $\alpha = 109.49$ (3), $\beta = 93.17$ (3), $\gamma = 75.87$ (2)°, $V = 932.8$ Å³, $Z = 2$, $D_m = 1.63$ (1), $D_c = 1.65$ g cm⁻³. Intensities for 4315 independent reflections (4202 above zero) were collected by counter methods. The structure was solved by standard heavy-atom methods, and full-matrix least-squares refinement has led to final R values of 0.043 (excluding zeros) and 0.045 (including zeros). The final weighted R value and goodness-of-fit are 0.052 and 2.1, respectively. The coordination about the cobalt is approximately octahedral with the two bidentate ethylenediamine ligands in *trans* positions. The chloride ligand and N(7) of the theophylline moiety complete the coordination sphere. The complex exhibits interligand hydrogen bonds from the ethylenediamine ligands to the carbonyl group, C(6)–O(6), on theophylline.

Introduction

Crystals of [Co(C₂N₂H₈)₂Cl(C₇N₄O₂H₇)]Cl were kindly supplied by Professor L. G. Marzilli and Mr C. H. Chang (1973, private communication). Preliminary photographs indicated a triclinic lattice. The unit-cell dimensions for the Friedel-reduced cell were derived from the 2θ , ω and χ values for 15 reflections. A density measurement, using flotation methods, suggested one formula unit and two waters of crystallization per asymmetric volume.

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Intensity measurements were made on a Syntex $P\bar{1}$ computer-controlled diffractometer; the incident beam was monochromatized by a graphite crystal. The crystal used in data collection was a cut cube about 0.25 mm on an edge. Intensity data were collected with Mo $K\alpha$ radiation by the θ – 2θ scan technique; individual scan speeds were determined by a rapid scan at the calculated Bragg peak, and the rate of scanning varied from 2 to 24° min⁻¹. Three standards were measured after every 100 reflections, and their intensities showed no unusual fluctuations or decay with time. A total of 4315 independent reflections in the $+h$ hemisphere to $2\theta = 55^\circ$ were surveyed; 4202 of these had intensities above zero and were assigned observational variances based on the equation: $\sigma^2(I) = S + (B_1 + B_2) (T_S/2T_B)^2 +$

$(pI)^2$, where S , B_1 and B_2 are the scan and background counts, T_S and T_B are the scan and individual background counting times ($T_B = \frac{1}{4}T_S$ for all reflections) and p was taken to be 0.03 and represents the error proportional to the diffracted beam intensity (Busing & Levy, 1957). Reflections with negative intensities were assigned F 's and $w(F)$'s equal to zero. The intensities and their standard deviations were corrected for Lorentz and polarization effects, but no correction for absorption was applied ($\mu = 12.8 \text{ cm}^{-1}$). The structure factor amplitudes were placed on an approximately absolute scale by the method of Wilson (1942).

A detailed examination of a three-dimensional Patterson synthesis allowed the positioning of the cobalt atom and the six atoms in its primary coordination sphere. A subsequent Fourier map indicated the positions of the remaining 19 heavy atoms in the asymmetric unit (including all nonhydrogen atoms, $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.27$). Six cycles of isotropic least-squares refinement, minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F_o)$, reduced the R value to 0.11. At this point, it was noted that one of the ethylenediamine rings, N(10)–C(10)–C(11)–N(11), had refined to a geometry which was essentially planar and contained unusual bond lengths and angles. A difference Fourier map was computed with the above four atoms removed. Examination of this map allowed the positioning of two ethylenediamine groups each approximately one-half occupied. Four further cycles were computed with the inclusion of anisotropic temperature parameters for

the cobalt and the two chlorine atoms ($R = 0.085$). A second difference Fourier map was then calculated in order to position the hydrogen atoms. Acceptable positions were obtained in all instances except for the following: (1) the 16 hydrogens associated with the disordered ethylenediamine group were placed in idealized positions with temperature parameters of 4.0 \AA^2 ; (2) one of the water hydrogen atoms off O(7) could not be located owing to the relatively high thermal motion of this water molecule.

Four cycles were then computed with the following parameters refined: (1) a scale factor; (2) positional parameters of all heavy atoms; (3) anisotropic thermal parameters for all heavy atoms except the six atoms of the disordered ethylenediamine group which were allowed only isotropic temperature parameters; (4) positional and isotropic temperature factors on the 18 non-idealized hydrogen atoms. The final R indices are 0.043 (excluding zeros) and 0.045 (including zeros). The final weighted R value $\{[\sum w(F_o - F_c)^2 / \sum w F_o^2]^{1/2}\}$ and goodness-of-fit $\{[\sum w(F_o - F_c)^2 / (n - p)]^{1/2}\}$ for $n = 4202$ observations of non-zero weight and $p = 295$ parameters are 0.052 and 2.1, respectively. The scattering factors for all heavy atoms were taken from the compilation of Hanson, Herman, Lea & Skillman (1964). The scattering curve for H was that of Stewart, Davidson & Simpson (1965). The real parts of the scattering curves for Co and Cl were corrected for anomalous dispersion effects (Cromer, 1965). In the final cycle of refinement, no parameter shifted by as much as 0.6

Table 1. Final heavy-atom parameters and their estimated standard deviations ($\times 10^5$)

The form of the anisotropic ellipsoid is $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + 2B_{12}hk + 2B_{13}hl + 2B_{23}kl)]$. Atoms C(10A) through C(11B) have been assigned population factors of 0.5.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Co	22473 (3)	17299 (3)	31997 (3)	637 (3)	374 (3)	582 (4)	-161 (2)	-167 (2)	172 (2)
Cl(1)	23314 (7)	37630 (5)	49008 (6)	1162 (8)	489 (5)	858 (7)	-290 (5)	-272 (6)	74 (5)
Cl(2)	8351 (8)	23324 (7)	84976 (8)	1170 (9)	1015 (8)	1176 (9)	-226 (6)	12 (7)	491 (7)
O(2)	38083 (18)	-54584 (16)	7452 (21)	924 (20)	494 (16)	1418 (27)	-134 (14)	-73 (19)	289 (17)
O(6)	33379 (19)	-12503 (16)	42661 (18)	1203 (22)	626 (17)	726 (20)	-145 (15)	-254 (17)	270 (15)
O(7)	39612 (29)	19583 (31)	80371 (57)	1035 (53)	1428 (38)	6596 (129)	-103 (29)	582 (53)	-565 (56)
O(8)	7438 (22)	-52706 (21)	17145 (24)	1263 (26)	1143 (24)	1435 (30)	-68 (20)	-63 (22)	731 (23)
N(1)	34801 (19)	-33329 (18)	24939 (21)	710 (20)	495 (18)	928 (25)	-161 (15)	-122 (18)	323 (17)
N(3)	28330 (18)	-36361 (17)	129 (21)	638 (20)	429 (17)	859 (24)	-108 (14)	-95 (17)	66 (16)
N(7)	21914 (18)	-462 (16)	17428 (19)	765 (19)	438 (17)	603 (20)	-175 (14)	-136 (15)	175 (15)
N(9)	19029 (19)	-15529 (18)	-5387 (20)	710 (20)	541 (18)	696 (22)	-133 (15)	-128 (17)	159 (16)
N(10)	30442 (20)	22533 (20)	16920 (22)	2.78 (3)					
N(11)	41589 (21)	10053 (21)	36379 (23)	3.04 (3)					
N(12)	3511 (19)	25439 (17)	28117 (20)	725 (20)	482 (18)	716 (22)	-133 (15)	-120 (17)	220 (16)
N(13)	14381 (21)	12875 (18)	47307 (20)	964 (23)	531 (19)	664 (22)	-94 (16)	-50 (18)	232 (17)
C(1)	39332 (28)	-39571 (26)	36383 (30)	1060 (31)	724 (26)	1172 (36)	-102 (22)	-59 (27)	539 (26)
C(2)	33975 (21)	-42182 (21)	10464 (26)	513 (21)	460 (20)	1058 (31)	-150 (16)	-18 (20)	204 (20)
C(3)	26940 (28)	-45049 (24)	-15164 (29)	1052 (31)	533 (23)	992 (33)	-67 (21)	-191 (25)	-90 (22)
C(4)	24578 (20)	-22397 (20)	3852 (23)	472 (20)	476 (20)	732 (26)	-134 (16)	-52 (18)	147 (18)
C(5)	26484 (20)	-13788 (19)	17887 (22)	540 (20)	397 (19)	691 (25)	-143 (15)	-107 (18)	188 (18)
C(6)	31599 (21)	-19067 (21)	29437 (23)	598 (22)	508 (20)	734 (26)	-179 (17)	-90 (19)	246 (19)
C(8)	17668 (22)	-2494 (21)	3321 (23)	737 (23)	521 (21)	636 (25)	-154 (18)	-160 (19)	245 (19)
C(10A)	44663 (50)	15082 (51)	12770 (56)	2.97 (8)					
C(10B)	45738 (51)	21622 (51)	21610 (56)	2.95 (8)					
C(11A)	50989 (52)	15350 (60)	27575 (58)	3.05 (9)					
C(11B)	50804 (56)	9116 (63)	26128 (62)	3.34 (9)					
C(12)	-5718 (25)	26461 (25)	40239 (27)	742 (26)	781 (26)	935 (31)	-60 (20)	57 (22)	320 (23)
C(13)	-368 (27)	13905 (26)	44679 (29)	961 (29)	777 (26)	1038 (33)	-231 (22)	113 (25)	396 (25)

e.s.d. Final atomic parameters for the heavy atoms are collected in Table 1.*

The structure factor and Fourier calculations were done with the X-RAY 67 series of programs (Stewart, 1967); the least-squares refinements were performed with an extensively modified version of *ORFLS* (Busing, Martin & Levy, 1962); best planes were computed with the program of Pippy & Ahmed (1968); the illustrations were prepared with the aid of the computer program *ORTEP* (Johnson, 1965).

* The lists of structure factors and hydrogen atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30605 (30 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

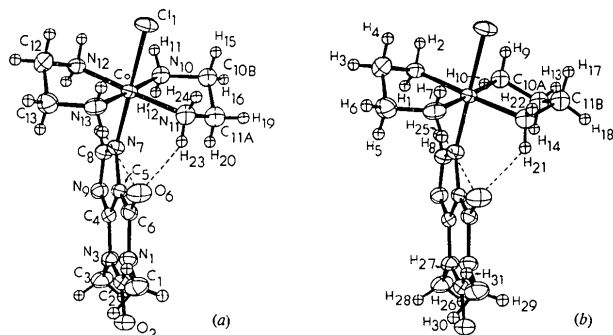


Fig. 1. A perspective view of the *trans*-[theophyllinatochlorobis(ethylenediamine)cobalt(III)]⁺ cation. (a) the $\delta\lambda$ -conformer, (b) the $\delta\delta$ -conformer. The dashed lines indicate the interligand hydrogen bonds. The thermal ellipsoids are drawn at the 50% probability level.

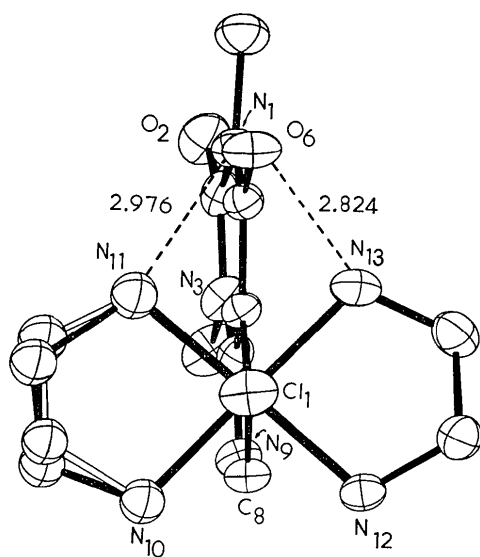


Fig. 2. A projection view of the complex cation down the Cl(1)–Co bond. The thermal ellipsoids are drawn at the 50% probability level.

Discussion

We have been particularly intrigued by the possibility that interligand hydrogen bonds may in some cases impart selectivity to the reaction of metal complexes with purine or pyrimidine bases and their nucleosides and nucleotides (Kistenmacher, Marzilli & Chang, 1973; Marzilli, Kistenmacher & Chang, 1973; Kistenmacher, 1974; Marzilli, Kistenmacher, Darcy, Szalda & Beer, 1974), selectivity of reaction being of particular importance to studies involved in the possible sequencing of polynucleotides by electron microscope techniques (Wiggins & Beer, 1972). In this paper, we present detailed structural data on crystals containing the *trans* isomer of the [theophyllinatochlorobis(ethylenediamine)cobalt(III)]⁺ ion, where theophylline is the substituted purine 1,3-dimethyl-2,6-dioxopurine. Coordination of the metal ion takes place through N(7) on the purine, a site which is accessible in polynucleotides. Furthermore, the presence of interligand hydrogen bonds in both the *trans*- and *cis*-theophylline complexes (Kistenmacher & Szalda, 1975) and in several other compounds we have studied (Szalda, Kistenmacher & Marzilli, 1974; Kistenmacher, Marzilli, Sorrell & Epps, 1974) serves to indicate the range of systems in which such interactions are found, and these studies offer the hope that such considerations will be of importance in polynucleotide sequencing efforts.

The molecular geometry of the *trans*-[theophyllinatochlorobis(ethylenediamine)cobalt(III)]⁺ cation is illustrated in Fig. 1(a) and (b). The ethylenediamine ring N(12)–C(12)–C(13)–N(13) has a conformation designated δ ,* while the chelate ring N(10)–C(10)–C(11)–N(11) is disordered and assumes both the δ and λ conformations in the crystal. Fig. 1(a) shows the chelate rings such that the overall molecular geometry has approximately mirror symmetry (chelate rings $\delta\lambda$; the $\lambda\delta$ conformation also exists in the crystal owing to the centrosymmetric space group); Fig. 1(b) illustrates the more asymmetric conformer–ring conformations $\delta\delta$ ($\lambda\lambda$). In each case the interligand hydrogen bonds are indicated by dashed lines. The hydrogen-bond distances range from 2.04 to 2.38 Å [H...O(6) distance]; the latter can only be considered a weak hydrogen bond. The ethylenediamine rings are somewhat swept away from the site occupied by the theophylline anion, probably owing to the steric requirements involved in the formation of the interligand hydrogen bonds. Furthermore, while the chloride ligand and N(7) are almost exactly *trans* [Cl(1)–Co–N(7) angle, 179.2 (1)°], the Co–N(7)–C(5) angle, 132.1 (2)°, and Co–N(7)–C(8) angle, 124.9 (2)°, are quite dissymmetric; again probably in response to the formation of the interligand hydrogen bonds. Fig. 2 is a conformational view of the complex cation down the Cl(1)–Co bond. The theophylline ring system has positioned itself nearly sym-

* For details of the nomenclature for the conformation of the ethylenediamine rings, see *Inorg. Chem.* (1970), **9**, 1–5.

metrically between the Co–N(10)···Co–N(12) and the Co–N(11)···Co–N(13) bonds. There is a slight tilt of the pyrimidine end of the theophylline moiety towards the disordered ethylenediamine ligand. There would seem to be no intrinsic reason why the hydrogen bonds should be different with the ethylenediamine rings in *trans* positions. This tilt, then, is presumably due to packing forces and causes the slight dissymmetry in the N(ethylenediamine)···O(6) distances.

Complete heavy-atom distances and angles are given in Table 2. The Co–Cl(1) distance of 2.255 (1) Å is in good agreement with the Co–Cl distance, 2.259 (1) Å, found in *cis*-[adeninatochlorobis(ethylenediamine)cobalt(III)] bromide (Kistenmacher, 1974). The average Co–N(ethylenediamine) distance at 1.958 (2) Å agrees very well with the average distance of 1.959 (3) Å in the *cis*-adenine complex. The Co–N(7) distance to the coordinated theophylline anion, 1.956 (2) Å, is comparable to the Co–N(9) distance to the coordinated adenine anion, 1.949 (3) Å. The geometry of the ordered ethylenediamine chelate ring, N(12)–C(12)–C(13)–N(13), is nearly identical with that of the ethylenediamine groups in the *cis*-adenine complex. The disordered ethylenediamine group is less well defined, and the bond lengths and angles have considerable scatter.

The parameters in the coordinated theophylline anion are of interest since metal binding has been shown to produce significant effects on distances, and particularly, on bond angles in coordinated purines (Sundaralingam & Carrabine, 1971; Kistenmacher, 1974). Theophylline is one of a few purines which exist as the N(7) tautomer (Sutor, 1958; Shefter, 1969). Qualitatively, then, the coordination complex can be viewed as arising from the substitution of a [chlorobis(ethylenediamine)cobalt(III)]²⁺ cation for the proton at N(7). Table 2 gives a complete list of dimensions for the theophylline moiety. A comparison with the values Shefter (1969) found in a theophylline:5-chlorosalicylic acid hydrogen-bond dimer reveals the following significant differences in bond lengths: N(7)–C(5) 1.404 (3) Å in the complex, 1.373 (7) Å in theophylline; C(6)–O(6) 1.245 (3) Å in the complex, 1.217 (7) Å in theophylline. The lengthening of the N(7)–C(5) bond in the complex is surely a result of the formation of the N(7)–Co coordination bond. The increase in the C(6)–O(6) bond length can be attributed to the formation of the interligand hydrogen-bond system. The differences in bond angles are somewhat more numerous, and essentially confined to the endocyclic and exocyclic bond angles in the imidazole ring and to that portion of the pyrimidine ring directly involved in the interligand hydrogen bonds.

The hydrogen atoms refined to geometrically acceptable values. The average (11 values) C–H distance is 0.93 Å, while the average N–H (4 values) and O–H (3 values) distances are 0.86 and 0.75 Å, respectively. All of these distances are consistent with values obtained from other X-ray diffraction studies (Churchill, 1973).

The idealized hydrogen atoms were positioned to give acceptable bond angles and C–H and N–H distances of 0.95 and 0.87 Å, respectively.

There are significant deviations from planarity in the nine-atom framework of the theophylline moiety. The imidazole portion of the molecule is quite planar, while the pyrimidine fragment retains a measureable non-

Table 2. *Heavy-atom interatomic distances and angles*

Estimated standard deviations: Co–Cl 0.001, Co–N 0.002, N(O)–C 0.004 Å, angles 0.2–0.3°.

(a) Coordination sphere about the cobalt atom

Co–Cl(1)	2.255 Å	Co–N(11)	1.964 Å
Co–N(7)	1.956	Co–N(12)	1.960
Co–N(10)	1.969	Co–N(13)	1.944
Cl(1)–Co–N(7)	179.2°	N(7)–Co–N(12)	92.2°
Cl(1)–Co–N(10)	88.9	N(7)–Co–N(13)	90.5
Cl(1)–Co–N(11)	88.8	N(10)–Co–N(11)	85.6
Cl(1)–Co–N(12)	88.5	N(10)–Co–N(12)	93.6
Cl(1)–Co–N(13)	89.1	N(10)–Co–N(13)	177.8
N(7)–Co–N(10)	91.5	N(11)–Co–N(12)	177.2
N(7)–Co–N(11)	90.5	N(11)–Co–N(13)	95.2
		N(12)–Co–N(13)	85.5

(b) Ethylenediamine chelate rings

Ordered

N(12)–C(12)	1.476 Å	Co—N(12)–C(12)	109.5°
C(12)–C(13)	1.508	Co—N(13)–C(13)	109.4
C(13)–N(13)	1.471	N(12)–C(12)–C(13)	106.9
		N(13)–C(13)–C(12)	106.6

Disordered

N(10)–C(10A)	1.458 Å	N(10)–C(10B)	1.563 Å
C(10A)–C(11B)	1.505	C(10B)–C(11A)	1.504
N(11)–C(11B)	1.606	N(11)–C(11A)	1.342

Co—N(10)–C(10A)	113.1°
N(10)–C(10A)–C(11B)	103.0
C(10A)–C(11B)–N(11)	108.6
C(11B)–N(11)–Co	106.2

Co—N(10)–C(10B)	103.2
N(10)–C(10B)–C(11A)	108.6
C(10B)–C(11A)–N(11)	105.7
C(11A)–N(11)–Co	113.9

(c) Theophylline anion

N(1)–C(1)	1.455 Å	N(7)–C(8)	1.346 Å
N(1)–C(2)	1.398	N(9)–C(4)	1.341
N(1)–C(6)	1.400	N(9)–C(8)	1.342
N(3)–C(2)	1.359	C(2)–O(2)	1.228
N(3)–C(3)	1.461	C(4)–C(5)	1.379
N(3)–C(4)	1.375	C(5)–C(6)	1.413
N(7)–C(5)	1.404	C(6)–O(6)	1.245
C(1)–N(1)–C(2)	116.7°	N(1)–C(2)–O(2)	120.8°
C(1)–N(1)–C(6)	117.2	N(3)–C(2)–O(2)	122.5
C(2)–N(1)–C(6)	126.1	N(3)–C(4)–N(9)	125.6
C(2)–N(3)–C(3)	119.4	N(3)–C(4)–C(5)	122.3
C(2)–N(3)–C(4)	120.3	N(9)–C(4)–C(5)	112.1
C(3)–N(3)–C(4)	120.2	N(7)–C(5)–C(4)	106.4
Co—N(7)–C(5)	132.1	N(7)–C(5)–C(6)	132.9
Co—N(7)–C(8)	124.9	C(4)–C(5)–C(6)	120.6
C(5)–N(7)–C(8)	102.8	N(1)–C(6)–C(5)	113.7
C(4)–N(9)–C(8)	102.4	N(1)–C(6)–O(6)	118.9
N(1)–C(2)–N(3)	116.7	C(5)–C(6)–O(6)	127.4
		N(7)–C(8)–N(9)	116.2

Table 3. *Hydrogen bonds and close contacts of the type D-H...A*

D	H	A	D...A	H...A	$\angle D-H...A(\angle H-D...A)$
(a) Interligand hydrogen bonds					
N(13)	H(8)	O(6) ^a	2.824 Å	2.04 Å	161 (14) ^o
N(11)	H(23)	O(6) ^a	2.976	2.19	150 (21)
N(11)	H(21)	O(6) ^a	2.976	2.38	126 (40)*
(b) Intermolecular hydrogen bonds					
N(10)	H(11)	O(8) ^a	3.057	2.25	153
N(12)	H(2)	O(8) ^e	2.974	2.15	148
O(7)	H(32)	Cl(2) ^a	3.100	2.43	164
O(8)	H(34)	Cl(2) ^c	3.259	2.53	173
O(8)	H(35)	Cl(2) ^b	3.227	2.40	167
(c) Possible weak hydrogen bonds and other close contacts					
N(13)	H(7)	Cl(2) ^a	3.415	2.77	137
N(10)	H(10)	Cl(2) ^d	3.671	2.82	165
N(12)	H(1)	N(9) ^f	3.163	2.38	145
C(8)	H(25)	Cl(2) ^d	3.650	2.80	166
	(a)	x, y, z	(d)	x, y, -1+z	
	(b)	-x, -y, 1-z	(e)	x, 1+y, z	
	(c)	x, -1+y, -1+z	(f)	-x, -y, -z	

* A weak hydrogen bond which is shown in this subsection for completeness.

planarity much of which is probably due to the interactions between the exocyclic carbonyl oxygen and methyl groups. As is typical for the purine fused-ring system (Sletten & Jensen, 1969; Voet & Rich, 1970), the imidazole and pyrimidine rings are tilted about the C(4)-C(5) bond with a dihedral angle of 2.3 (4)^o.

A packing diagram of the unit cell, viewed down the *c** axis is given in Fig. 3. Hydrogen bonds (Table 3) and electrostatic forces dominate the crystal packing.

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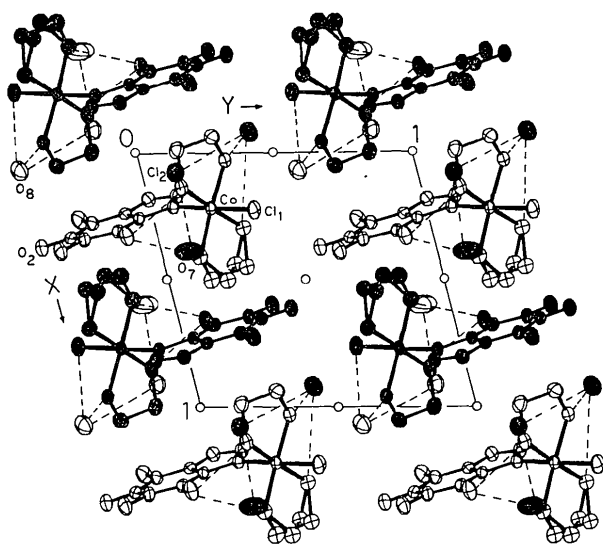


Fig. 3. A view down the *c** axis of the crystal packing. The dashed lines indicate hydrogen bonds. The labeled atoms have the coordinates given in Table 2.

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