DAHL, L. F., COSTELLO, W. R. & KING, R. B. (1968). *J. Amer. Chem. Soc.* **90**, 5422.
DAHL, L. F., GIL, E. R. & FELTHAN, R. D. (1969). *J. Amer. Chem. Soc.* **91**, 1653.
DAHL, L. F. & WEI, C-H. (1963). *Inorg. Chem.* **2**, 328.
DAVIES, G. R. & KILBOURN, B. T. (1971). *J. Chem. Soc.* (*A*), p. 87.
DIAS, A. R. & GREEN, M. L. H. (1969). *Chem. Commun.* p. 962.
EDWARDS, R. R., JOHNSON, C. E. & WILLIAMS, R. J. P. (1967). *J. Chem. Phys.* **47**, 2074.
FERGUSON, G., HANNAWAY, C. & ISLAM, K. M. S. (1968). *Chem. Commun.* p. 1165.
KNOX, J. R. & PROUT, C. K. (1969*a*). *Acta Cryst.* B**25**, 2013.
KNOX, J. R. & PROUT, C. K. (1969*b*). *Acta Cryst.* B**25**, 2482.
LAWTON, D. & MASON, R. (1965). *J. Amer. Chem. Soc.* **87**, 921.
MAIS, R. H. B., OWSTON, P. G. & THOMPSON, D. T. (1967). *J. Chem. Soc.* (*A*), 1735.
PROUT, C. K. & REES, G. V. (1971). To be published.
ROLLETT, J. S. (1970). *Crystallographic Computing*, p. 167. Copenhagen: Munksgaard.
SPENCE, J. T. (1969). *Co-ord. chem. Rev.* **4**, 475.
STEVENSON, D. L. & DAHL, L. F. (1967). *J. Amer. Chem. Soc.* **89**, 3721.
WASER, J. (1963). *Acta Cryst.* **16**, 1091.
WEBER, H. P. & BRYAN, R. F. (1967). *J. Chem. Soc.* (*A*), p. 182.

Acta Cryst. (1972). B**28**, 457

The Crystal Structure of $(-)_{589}$-*cis*-α-Dinitro-(L-3,8-dimethyltriethylenetetramine)-cobalt(III) Perchlorate, *cis*-α-[$Co(NO_2)_2$(L-3,8-dimetrien)]ClO_4

BY M. ITO, F. MARUMO AND Y. SAITO

The Institute for Solid State Physics, The University of Tokyo, Roppongi-7, Minato-ku, Tokyo 106, *Japan*

(*Received* 26 *April* 1971)

The structure of $(-)_{589}$-*cis*-α-[$Co(NO_2)_2$(L-3,8-dimetrien)]ClO_4 has been determined from the three-dimensional X-ray data collected by the diffractometer method. The compound forms orthorhombic crystals with $a=12{\cdot}395$, $b=16{\cdot}829$, $c=7{\cdot}864$ Å and $Z=4$, in space group $P2_12_12_1$. The structure has been refined by least-squares methods with anisotropic temperature factors to an R value of 0·056 for 2268 observed reflexions. The perchlorate anion is disordered over two positions. Six nitrogen atoms of the dimetrien ligand and nitro groups are bonded nearly octahedrally to the central cobalt atom. The complex cation has approximately the symmetry 2 (C_2). The average Co–N(dimetrien) distance is 1·958 (7) Å, and the average NCoN angle of terminal chelate rings is 86·1 (3)°, whereas that of the central ring is 87·6 (3)°. The complex cation has the absolute configuration *A*, and the conformations of the three chelate rings are δ, λ and δ, the two methyl groups being in equatorial positions with respect to the chelate rings. The absolute configurations of the two asymmetric nitrogen atoms are both *S*.

Introduction

Three possible isomers of disubstituted triethylenetetraminecobalt(III) complexes (*cis*-α, *cis*-β and *trans*) were recently prepared and isolated as perchlorates by using the stereoselective quadridentate ligand, L-3,8-dimethyltriethylenetetramine: $H_2NCH_2CH(CH_3)NHCH_2CH_2NHCH(CH_3)CH_2NH_2$, 3,8-dimetrien (Yoshikawa, Saburi, Sawai & Goto, 1969). The structure of the *cis*-β isomer has already been determined (Ito, Marumo & Saito, 1970). The authors' interest lay in the comparison of the strain energies of the three isomers. Dwyer & Maxwell (1970) have determined the structure of racemic *cis*-α-(amminechlorotriethylenetetramine)cobalt(III) nitrate. From the result large non-bonded interactions between the chelate rings are also expected for L-3,8-dimetrien coordinated in *cis*-α configuration. The crystal structure of $(-)_{589}$-*cis*-α-[$Co(NO_2)_2$(L-3,8-dimetrien)]ClO_4 has been determined in order to establish the stereochemical configuration of the complex cation. The calculation of the strain energy will be reported in the next paper together with that of the *cis*-β and *trans* isomers (Ito, Marumo & Saito, 1972).

Experimental

The specimens were kindly supplied by Professor S. Yoshikawa of this University. They are orange prismatic crystals elongated along the *c* axis, and belong to the orthorhombic system. The cell dimensions, $a=12{\cdot}395\pm0{\cdot}002$, $b=16{\cdot}829\pm0{\cdot}002$ and $c=7{\cdot}864\pm0{\cdot}002$ Å, were determined by using a single-crystal diffractometer with Mo $K\alpha$ radiation ($\alpha_1=0{\cdot}70926$, $\alpha_2=0{\cdot}71354$ Å). The systematic absences indicate that the space group is $P2_12_12_1$. There are four formula units of $C_8H_{22}ClCoN_6O_8$ in the unit cell ($D_x=1{\cdot}72$ g.cm^{-3}, $D_m=1{\cdot}72$ g.cm^{-3}). The crystals were reformed into a sphere with a diameter of about 0·22 mm. The intensity data were collected on a Rigaku automatic four-

circle diffractometer. The specimen was mounted with the *c* axis parallel to the φ axis of the diffractometer. Mo *Kα* radiation monochromated by LiF crystal was used. The ω–2θ scan technique was employed at a rate of 0·5°(ω) per minute, and scan times were varied as calculated from the tangent relation of Alexander &

Table 1. *Observed and calculated structure factors* (×10)

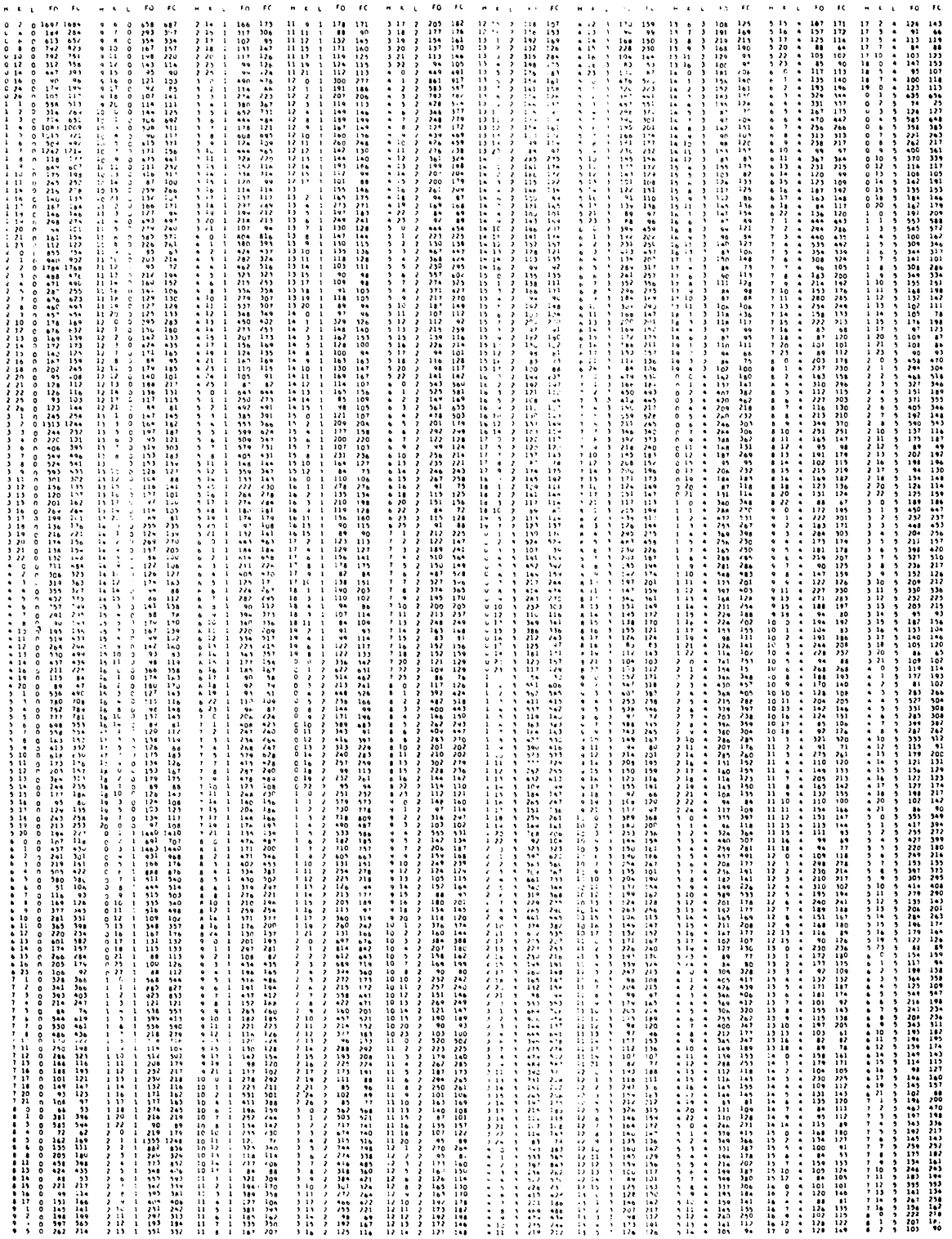
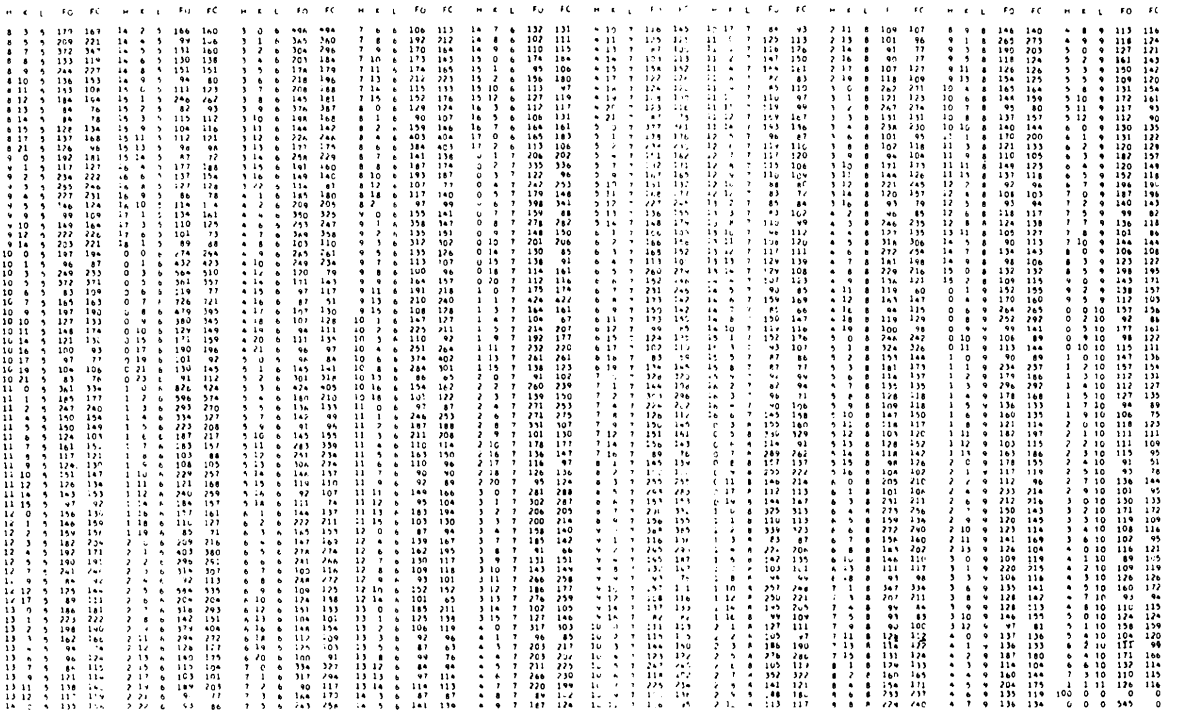


Table 1 (*cont.*)



Smith (1964). Backgrounds were measured for 15 sec at both limits of the scan. A total of 3150 intensities were measured in the range of $30° < 2\theta < 70°$, and the lower angle data, $2\theta < 30°$, were supplied by a Rigaku manual four-circle diffractometer. 2268 of these had significantly non-zero intensities. The intensities were corrected for Lorentz and polarization factors, but corrections for absorption ($\mu r = 0{\cdot}3$) and extinction were not made.

Determination and refinement of the structure

The cobalt and chlorine atoms could easily be located from a three-dimensional sharpened Patterson function synthesized only with the reflexions in the range $2\theta < 40°$. The positions of the lighter atoms were determined by successive Fourier syntheses except for the oxygen atoms of the perchlorate ion. The Fourier maps revealed that the perchlorate group was disordered over two sites. Six cycles of block-diagonal least-squares refinement with isotropic temperature factors reduced the R value (defined as $\sum||F_o|-|F_c||/\sum|F_o|$) to 0·108. Refinement yielded an unsatisfactory geometry for the perchlorate group. Three of the Cl–O distances were too long (from 1·5 to 1·8 Å) and the fourth was too short (1·1 Å). Further refinement using anisotropic temperature factors for all the atoms converged with $R = 0{\cdot}064$. During the refinements the molecular geometry of the perchlorate was re-examined every second cycle and was corrected by treating the group as with the normal Cl–O distance of 1·43 Å and OClO angle of 109°. The populations for two orientations of the perchlorate group were estimated from the Fourier synthesis. At this stage a difference Fourier synthesis was carried out, which revealed all the hydro-

Table 2. *Final atomic parameters*

(*a*) Final positional parameters and their standard deviations (in parentheses)

The values have been multiplied by 10^4. m^*: Population.

	x	y	z	m^*
Co	2208 (1)	2893 (1)	5365 (1)	
Cl	252 (2)	801 (1)	1357 (3)	
N(1)	2786 (6)	3953 (4)	5652 (8)	
N(2)	1056 (5)	3240 (4)	6908 (8)	
N(3)	2940 (5)	2454 (4)	7362 (8)	
N(4)	1603 (5)	1832 (4)	5072 (9)	
N(5)	3397 (6)	2611 (4)	3959 (9)	
N(6)	1426 (5)	3241 (4)	3454 (9)	
C(1)	2147 (7)	4453 (4)	6827 (11)	
C(2)	994 (6)	4141 (5)	6828 (11)	
C(3)	1229 (6)	2913 (5)	8611 (9)	
C(4)	2396 (6)	2770 (5)	8921 (10)	
C(5)	2996 (6)	1562 (5)	7170 (11)	
C(6)	1904 (7)	1296 (5)	6494 (12)	
C(7)	310 (8)	4537 (6)	8145 (13)	
C(8)	3325 (9)	1140 (6)	8757 (14)	
O(1)	4243 (4)	3024 (4)	3984 (9)	
O(2)	3410 (6)	2022 (5)	3031 (10)	
O(3)	1828 (5)	3723 (4)	2392 (8)	
O(4)	485 (5)	3029 (4)	3206 (9)	
O(5)	63 (11)	661 (8)	3091 (17)	0·6
O(6)	795 (15)	140 (9)	665 (21)	0·6
O(7)	−629 (20)	923 (15)	392 (36)	0·6
O(8)	1049 (14)	1394 (12)	1294 (25)	0·6
O(9)	−230 (12)	1420 (14)	2319 (39)	0·4
O(10)	−312 (15)	113 (14)	1782 (31)	0·4
O(11)	1334 (17)	875 (10)	1586 (28)	0·4
O(12)	−74 (15)	1009 (13)	−229 (20)	0·4

Table 2 (*cont.*)

(*b*) Final thermal parameters and their standard deviations (in parentheses).
The values have been multiplied by 10^4 and refer to the expression:

$$\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Co	21 (0)	17 (0)	61 (1)	0 (0)	− 1 (1)	2 (1)
Cl	62 (2)	28 (1)	145 (4)	1 (1)	21 (2)	7 (2)
N(1)	35 (4)	30 (2)	84 (12)	4 (3)	2 (7)	10 (4)
N(2)	28 (4)	22 (2)	65 (10)	2 (3)	− 6 (5)	−13 (4)
N(3)	30 (4)	25 (2)	88 (11)	6 (3)	7 (6)	5 (4)
N(4)	25 (3)	26 (2)	122 (15)	−1 (2)	−10 (6)	1 (5)
N(5)	43 (4)	25 (3)	77 (11)	−2 (3)	−12 (6)	3 (5)
N(6)	36 (4)	20 (2)	91 (11)	1 (3)	6 (6)	7 (4)
C(1)	46 (5)	17 (2)	139 (15)	6 (4)	1 (9)	16 (5)
C(2)	36 (5)	21 (3)	103 (14)	−6 (3)	14 (7)	6 (6)
C(3)	52 (5)	25 (3)	51 (11)	−6 (4)	17 (6)	−11 (6)
C(4)	46 (5)	27 (3)	74 (11)	2 (3)	− 9 (6)	4 (5)
C(5)	33 (5)	24 (3)	121 (15)	−11 (3)	5 (7)	−13 (6)
C(6)	45 (6)	25 (3)	125 (15)	2 (3)	4 (8)	−17 (6)
C(7)	67 (7)	32 (4)	156 (19)	−12 (5)	17 (10)	19 (7)
C(8)	74 (8)	35 (4)	181 (21)	−14 (5)	− 5 (12)	−44 (8)
O(1)	22 (3)	47 (3)	217 (14)	1 (3)	25 (6)	9 (6)
O(2)	79 (6)	48 (3)	237 (16)	−4 (4)	66 (8)	53 (7)
O(3)	74 (5)	45 (3)	97 (11)	−4 (4)	4 (7)	−32 (5)
O(4)	37 (4)	40 (3)	184 (13)	−1 (3)	−44 (6)	− 6 (6)
O(5)	109 (13)	52 (6)	198 (29)	2 (8)	88 (18)	33 (12)
O(6)	178 (20)	54 (7)	277 (39)	−40 (10)	33 (24)	57 (15)
O(7)	207 (29)	157 (18)	650 (81)	−112 (20)	−136 (45)	55 (36)
O(8)	144 (18)	109 (12)	361 (50)	72 (13)	73 (26)	12 (22)
O(9)	56 (18)	77 (15)	915 (138)	−16 (15)	15 (45)	215 (40)
O(10)	177 (34)	57 (14)	433 (94)	60 (19)	81 (50)	1 (32)
O(11)	52 (17)	147 (27)	697 (129)	−61 (19)	14 (43)	35 (56)
O(12)	257 (47)	103 (19)	289 (73)	42 (25)	−164 (53)	−51 (35)

Table 2 (*cont.*)

(*c*) Positional and isotropic thermal parameters for the hydrogen atoms.
The values for the positional parameters have been multiplied by 10^3.

	x	*y*	*z*	*B* (Å^2)
H(N11)	356 (8)	380 (6)	602 (14)	3·0 (2·7)
H(N12)	264 (8)	420 (6)	438 (13)	2·1 (2·3)
H(N21)	40 (7)	300 (5)	651 (11)	0·9 (2·0)
H(N31)	367 (8)	261 (6)	738 (14)	3·3 (2·8)
H(N41)	78 (9)	192 (6)	482 (15)	5·5 (3·1)
H(N42)	181 (8)	163 (6)	382 (13)	2·4 (2·6)
H(C11)	243 (9)	511 (6)	660 (13)	3·1 (2·6)
H(C12)	247 (8)	425 (5)	812 (13)	2·3 (2·6)
H(C21)	80 (7)	429 (5)	549 (13)	1·1 (2·1)
H(C31)	73 (8)	248 (6)	845 (14)	2·9 (2·7)
H(C32)	85 (8)	325 (6)	957 (14)	2·5 (2·6)
H(C41)	248 (8)	257 (6)	1021 (15)	2·7 (2·7)
H(C42)	255 (9)	312 (6)	922 (14)	3·6 (3·0)
H(C51)	338 (8)	146 (6)	610 (14)	3·0 (2·8)
H(C61)	137 (9)	136 (6)	727 (15)	4·0 (2·8)
H(C62)	205 (9)	69 (6)	616 (15)	5·2 (3·6)
H(C71)	33 (10)	512 (7)	755 (17)	6·0 (3·4)
H(C72)	−32 (9)	420 (6)	842 (15)	4·7 (3·2)
H(C73)	48 (9)	451 (6)	948 (15)	3·4 (2·7)
H(C81)	274 (10)	112 (6)	941 (16)	5·8 (3·2)
H(C82)	391 (9)	127 (7)	926 (15)	3·6 (3·0)
H(C83)	348 (8)	49 (6)	861 (13)	1·8 (2·6)

gen atoms. The final set of least-squares calculations was carried out including the contributions of the hydrogen atoms. Temperature factors for the hydrogen atoms were assumed to be isotropic. After one cycle of the refinement the *R* value was reduced to 0·056. Unit weight was given for all $|F_o|$s larger than 20·0 and 0·2 for the remainders.

The absolute configuration of the whole complex ion can be determined as *A* from knowledge of the absolute configuration of the L-3,8-dimetrien ligand.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The observed and calculated structure factors are listed in Table 1. Final atomic parameters are given in Table 2, with their estimated standard deviations.

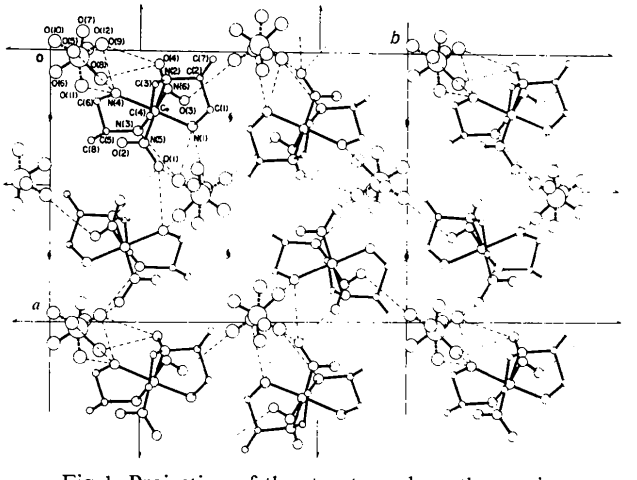


Fig. 1. Projection of the structure along the *c* axis.

Results and discussion

The crystal structure can be described in terms of successive layers of cations and anions parallel to the plane (100). Fig. 1 represents the arrangement of the complex cations and perchlorate anions in the unit cell, viewed down the *c* axis. Oxygen atoms of the perchlorate groups are disordered over two sites. Those shown by broken lines have a smaller population of 0·4 and the other oxygen atoms have a slightly larger value of 0·6. Dashed lines indicate some close contacts. The packing relation in this crystal is similar to those observed in the *cis-β* isomer (Ito, Marumo & Saito, 1970) and in the *trans* isomer (Ito, Marumo & Saito, 1972). Fig. 2 is a representation of the anisotropic thermal motion of the complex cation. It represents correctly the absolute configuration of the complex ion, which can be described as a skew chelate pair, *Λ*. The bond lengths and angles are listed in Table 3 with their estimated standard deviations.

The combination of the conformations for three chelate rings is *δ*, *λ* and *δ*, and the two methyl groups lie in equatorial positions with respect to the planes of the chelate rings. Consequently, the absolute configurations about both the asymmetric nitrogen atoms are *S*. The above mentioned stereochemical features, which had been expected from the circular dichroism and proton magnetic resonance measurements (Yoshikawa & Saburi, 1971), were indeed confirmed by the present study.

The Co–N(dimetrien) distances are almost equal within twice the standard deviations. Their mean value, 1·958 (7) Å is comparable with 1·955 (9) Å in *cis-α*-[Co(trien)NH_3Cl]$(NO_3)_2$ (Dwyer & Maxwell, 1970) and with 1·970 (15) Å in *cis-β*-[Co$(NO_2)_2$(L-3,8-dimetrien)]ClO_4 (Ito, Marumo & Saito, 1970). The average C–N distance of 1·493 Å and C–C distance of 1·504 Å are in agreement with those observed in other trien structures (Freeman & Maxwell, 1969). The two nitrogen atoms of the nitro groups are coordinated at a distance of 1·895 (8) Å which is shorter than those of the *cis-β* and the *trans* isomers.

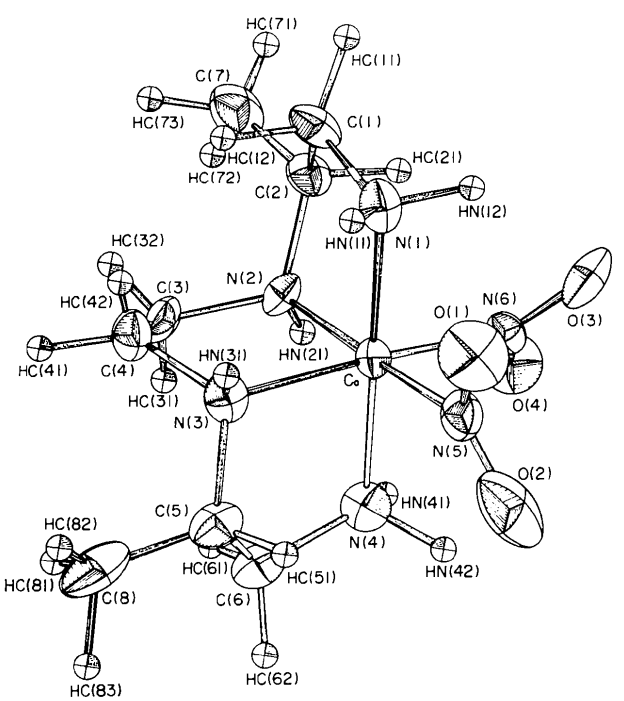


Fig. 2. A perspective drawing of the complex cation showing ellipsoids of thermal motion.

Table 3. *Interatomic distances and bond angles in the complex ion and the perchlorate ion*

The estimated standard deviations given in parentheses are for least significant figures.

The complex ion

Co···N(1)	1·935 (7) Å	C(1)···N(1)	1·480 (12) Å
Co···N(2)	1·964 (7)	C(2)···N(2)	1·520 (11)
Co···N(3)	1·959 (7)	C(3)···N(2)	1·464 (11)
Co···N(4)	1·951 (7)	C(4)···N(3)	1·497 (11)
Co···N(5)	1·902 (7)	C(5)···N(3)	1·510 (11)
Co···N(6)	1·883 (7)	C(6)···N(4)	1·485 (11)
C(1)···C(2)	1·522 (13)	N(5)···O(1)	1·258 (10)
C(3)···C(4)	1·487 (12)	N(5)···O(2)	1·232 (10)
C(5)···C(8)	1·493 (14)	N(6)···O(3)	1·266 (10)
C(2)···C(7)	1·495 (13)	N(6)···O(4)	1·235 (10)
C(5)···C(6)	1·522 (13)		
N(1)CoN(2)	85·7 (3)°	C(1)C(2)C(7)	113·5 (8)
N(2)CoN(3)	87·6 (3)	C(7)C(2)N(2)	116·4 (7)
N(3)CoN(4)	86·5 (3)	N(2)C(3)C(4)	110·7 (7)
CoN(1)C(1)	113·6 (6)	C(3)C(4)N(3)	111·2 (7)
CoN(2)C(2)	107·9 (5)	N(3)C(5)C(6)	106·7 (7)
CoN(2)C(3)	110·3 (5)	C(5)C(6)N(4)	108·0 (7)
CoN(3)C(4)	108·3 (5)	N(3)C(5)C(8)	113·7 (8)
CoN(3)C(5)	108·4 (5)	C(8)C(5)C(6)	113·7 (9)
CoN(4)C(6)	111·7 (5)	CoN(6)O(3)	121·5 (6)
CoN(5)O(1)	120·0 (6)	CoN(6)O(4)	121·5 (6)
CoN(5)O(2)	123·7 (6)	O(3)N(6)O(4)	117·8 (7)
O(1)N(5)O(2)	117·3 (7)	C(2)N(2)C(3)	114·5(4)
N(1)C(1)C(2)	107·8 (7)°	C(4)N(3)C(5)	117·1(4)
C(1)C(2)N(2)	107·3 (7)		

The perchlorate ion

Cl···O(5)	1·404 (14) Å	Cl···O(9)	1·419 (19) Å
Cl···O(6)	1·409 (16)	Cl···O(10)	1·393 (23)
Cl···O(7)	1·346 (26)	Cl···O(11)	1·359 (27)
Cl···O(8)	1·405 (19)	Cl···O(12)	1·357 (22)
O(5)ClO(6)	108·8 (9)°	O(9)ClO(10)	105·7 (12)°
O(5)ClO(7)	116·0 (13)	O(9)ClO(11)	106·1 (14)
O(5)ClO(8)	105·7 (10)	O(9)ClO(12)	100·1 (12)
O(6)ClO(7)	106·9 (13)	O(10)ClO(11)	122·7 (15)
O(6)ClO(8)	102·2 (10)	O(10)ClO(12)	106·6 (13)
O(7)ClO(8)	116·3 (14)	O(11)ClO(12)	113·1 (15)

The angles subtended at the cobalt atom by the outer two chelate rings [85·8 (3), 86·5 (3)°] are equal within twice the standard deviations, whereas the angle subtended by the inner chelate ring is slightly larger at 87·6 (3)°. The two outer chelate rings have unsymmetrical skew conformations. The ring carbon atoms C(1) and C(2) lie at −0·6 and 0·0 Å, respectively, from the N(1)–Co–N(2) plane. The corresponding deviations of C(5) and C(6) from the N(3)–Co–N(4) plane are −0·1 and 0·5 Å. In the central chelate ring, on the other hand, C(3) and C(4) are found to lie 0·24 Å above

and 0·27 Å below the plane of N(2)–Co–N(3), respectively. In a number of ethylenediamine complexes of Co(III), the dihedral angles (N–C–C–N) formed by the plane of N–C–C and the plane of C–C–N are about 50°. However, the angles for the two outer chelate rings in the present compound are 40 and 42°, and the central chelate ring has a much smaller value of 35°. These deformations of the chelate rings may be attributed to the non-bonded interactions between hydrogen atoms of the three chelate rings, since the ligand is turning from one ring to the next. Table 4 shows the short distances between the hydrogen atoms belonging to the different chelate rings. This point will be discussed in detail in the next paper (Ito, Marumo & Saito, 1972). The conformations of the three chelate rings are such that the *cis*-α dimetrien skeleton possesses an approximate twofold rotational axis through the cobalt atom and bisecting the C(3)–C(4) bond.

Table 4. *The short distances between the hydrogen atoms belonging to the different chelate rings within the same molecule*

H(C12)···H(C42)	2·09 Å
H(C31)···H(C61)	2·25
H(N11)···H(N31)	2·27
H(N21)···H(N41)	2·30

Table 5. *Intermolecular distances less than* 3·5 Å

Key to symmetry operations

(1)	x,	y,	z
(2)	$-x$,	$0{\cdot}5+y$,	$0{\cdot}5-z$
(3)	$0{\cdot}5-x$,	$-y$,	$0{\cdot}5+z$
(4)	$0{\cdot}5+x$,	$0{\cdot}5-y$,	$1{\cdot}0-z$
(5)	$0{\cdot}5-x$,	$1{\cdot}0-y$,	$0{\cdot}5+z$

	Distances	Symmetry operation applied to second atom
N(4)···O(5)	3·16 Å	(1)
N(4)···O(8)	3·14	(1)
N(4)···O(9)	3·21	(1)
N(4)···O(11)	3·20	(1)
O(2)···O(8)	3·40	(1)
O(2)···O(11)	3·41	(1)
O(4)···O(8)	3·21	(1)
O(4)···O(9)	2·93	(1)
N(6)···O(10)	3·45	(2)
C(2)···O(6)	3·40	(2)
C(2)···O(10)	3·38	(2)
C(7)···O(6)	3·45	(2)
C(7)···O(12)	3·39	(2)
O(3)···O(10)	3·07	(2)
C(5)···O(6)	3·44	(3)
C(8)···O(6)	3·43	(3)
N(1)···O(5)	3·06	(4)
N(1)···O(9)	3·00	(4)
N(1)···O(10)	3·48	(4)
N(3)···O(4)	3·29	(4)
N(3)···O(9)	2·97	(4)
C(1)···O(10)	3·41	(4)
C(4)···O(7)	3·34	(4)
C(4)···O(9)	3·39	(4)
C(5)···O(4)	3·18	(4)
C(8)···O(4)	3·39	(4)
O(1)···N(2)	3·17	(4)
O(1)···N(4)	3·03	(4)
O(1)···O(4)	3·22	(4)
O(1)···O(5)	3·35	(4)
O(1)···O(9)	3·12	(4)
O(2)···N(2)	3·31	(4)
C(1)···O(3)	3·35	(5)

Close contacts between the coordinated nitrogen atoms and perchlorate oxygen atoms, and between the oxygen atoms of the nitro groups and the perchlorate oxygen atoms of less than 3·5 Å are listed in Table 5. All these distances agree with those observed in the *cis*-β isomer (Ito, Marumo & Saito, 1970).

Calculation of the Fourier synthesis and the least-squares were carried out on the HITAC 5020E computer at the Computer Centre of this University with the programs *ANSFR*-1, *RSFR*-5, *HBLS*-4 and *ORTEP* of the *UNICS* system written by Dr Iwasaki, Dr Sakurai, Dr Ashida and Dr Johnson respectively. All other calculations were computed on the FACOM 270-30 computer at this Institute.

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. The authors are grateful to Professor S. Yoshikawa and Dr M. Saburi for the kind supply of the specimens.

References

ALEXANDER, L. E. & SMITH, G. S. (1964). *Acta Cryst.* **17**, 1195.

DWYER, M. & MAXWELL, I. E. (1969). *Inorg. Chem.* **9**, 1459.

FREEMAN, H. C. & MAXWELL, I. E. (1969). *Inorg. Chem.* **8**, 1293.

International Tables for X-ray Crystallography (1962). Vol. II. Birmingham: Kynoch Press.

ITO, M., MARUMO, F. & SAITO, Y. (1970). *Acta Cryst.* B**26**, 1408.

ITO, M., MARUMO, F. & SAITO, Y. (1972). *Acta Cryst.* B **28**, 463.

YOSHIKAWA, S. & SABURI, M. (1971). Private communication.

YOSHIKAWA, S., SABURI, M., SAWAI, T. & GOTO, M. (1969). *Proc.* XII *ICCC*, p. 155. Sydney.