

bc plane of DL isoleucine in the same way as found in the *ac* plane of DL-valine. The *R* factor for this first trial model was 0.24. A difference Fourier synthesis soon indicated the presence in the same site of two diastereoisomeric molecules.

The structure was refined by least-squares methods with anisotropic thermal parameters for all atoms except those [C(4), C(5), C(6-1), C(6-2)] involved in the statistical substitution. No attempt was made to locate the hydrogen atoms. The final conventional *R* value is 0.15, which should be considered an acceptable value on the basis of the poor quality and twinning of the crystals, and the partially statistical nature of the structure.

The structural feature common to the four crystals is the presence of layers containing hydrogen bonded molecules having the same configuration (in the particular case of our sample of DL-isoleucine, we refer to the configuration of the α -carbon atom only). The relationship between such layers in DL-isoleucine and DL-valine is of quasi-identity [Fig. 1(a) and (b)]. This is an indication of the predominant role played by the hydrogen bonding compared with the side-chain interactions. Significant, but still small, differences distinguish the packing of iso-configurational molecules in DL and L crystals [Fig. 2, *cf.* Fig. (a) and (b)].

A second crystallographically independent molecule appears in the L crystals with a different conformation on the side chain. In both racemic and optically active

forms, molecular layers are tightly associated in pairs *via* hydrogen bonding. The coupled layers are crystallographically related by inversion centres in the racemic forms and by binary screw axes in the optically active ones.

The crystals are built up by a sequence of such pairs of molecular layers related by an unitary lattice translation (Fig. 3) or, as in DL-valine, by a 2_1 symmetry operation.

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The Crystal Structure of (+)₅₈₉-*cis*- β -Dinitro-(*R*-5-methyltriethylenetetramine)cobalt(III) Chloride, (+)₅₈₉-*cis*- β -[Co(NO₂)₂(*R*-5-metrien)]Cl

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The structure of (+)₅₈₉-*cis*- β -[Co(NO₂)₂(*R*-5-metrien)]Cl has been determined from three-dimensional X-ray data collected by diffractometer. The crystals are orthorhombic, space group $P2_12_12_1$, with unit cell dimensions: $a = 12.696$, $b = 14.364$ and $c = 7.542$ Å. There are four formula units in the unit cell. The structure was solved by the heavy-atom method and refined by the anisotropic block-diagonal least-squares method to an *R* value of 0.069 for 1567 observed reflexions. The quadridentate ligand is linked to the central cobalt atom with its four nitrogen atoms in *cis*- β coordination. A substituted methyl group lies in an equatorial position relative to the plane of the chelate ring. It is attached to the carbon atom next to the secondary nitrogen atom common to the two chelate rings both in the equatorial plane of the octahedron. The absolute configuration of the complex ion can be described as a skew chelate pair, *A*. The conformation of the chelate ring with the methyl group is λ and those of the other two are δ and λ , respectively. The absolute configurations of the two secondary nitrogen atoms are both *S*.

Introduction

This study of (+)₅₈₉-*cis*- β -dinitro(*R*-5-methyltriethylenetetramine)cobalt(III) chloride,

(+)₅₈₉-*cis*- β -[Co(NO₂)₂(*R*-5-metrien)]Cl,
R-5-metrien:

$\text{H}_2\text{NCH}_2\text{CH}_2\text{NHCH}(\text{CH}_3)\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2$,
 is part of the crystal structure analysis of substituted triethylenetetraminecobalt(III) complexes (Ito, Marumo & Saito, 1970, 1972*a*, *b*). The authors were interested in comparing the geometries of trien ligands.

Experimental

The specimens were kindly supplied by Professor S. Yoshikawa of this University. They are red tabular crystals. Oscillation and Weissenberg photographs were taken to determine the cell dimensions and the space group, using Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The

crystal data are: $\text{C}_7\text{H}_{20}\text{ClCoN}_6\text{O}_4$, M.W. 346.7, orthorhombic, $a = 12.696(2)$, $b = 14.364(2)$, $c = 7.542(2) \text{ \AA}$, $U = 1375 \text{ \AA}^3$, $D_m = 1.66$, $D_x = 1.67 \text{ g cm}^{-3}$, $Z = 4$, linear absorption coefficient for Mo $K\alpha$ radiation $\mu = 15 \text{ cm}^{-1}$, space group $P2_12_12_1$ (No. 19).

The intensity data were collected on a Rigaku automated four-circle diffractometer. A crystal shaped into

Table 1. Observed and calculated structure factors

h	k	l	F _o	F _c	F _c /F _o	h	k	l	F _o	F _c	F _c /F _o	h	k	l	F _o	F _c	F _c /F _o
0	0	0	1000	1000	1.000	0	0	0	1000	1000	1.000	0	0	0	1000	1000	1.000
0	0	1	114	114	1.000	0	0	1	114	114	1.000	0	0	1	114	114	1.000
0	0	2	114	114	1.000	0	0	2	114	114	1.000	0	0	2	114	114	1.000
0	0	3	114	114	1.000	0	0	3	114	114	1.000	0	0	3	114	114	1.000
0	0	4	114	114	1.000	0	0	4	114	114	1.000	0	0	4	114	114	1.000
0	0	5	114	114	1.000	0	0	5	114	114	1.000	0	0	5	114	114	1.000
0	0	6	114	114	1.000	0	0	6	114	114	1.000	0	0	6	114	114	1.000
0	0	7	114	114	1.000	0	0	7	114	114	1.000	0	0	7	114	114	1.000
0	0	8	114	114	1.000	0	0	8	114	114	1.000	0	0	8	114	114	1.000
0	0	9	114	114	1.000	0	0	9	114	114	1.000	0	0	9	114	114	1.000
0	0	10	114	114	1.000	0	0	10	114	114	1.000	0	0	10	114	114	1.000
0	0	11	114	114	1.000	0	0	11	114	114	1.000	0	0	11	114	114	1.000
0	0	12	114	114	1.000	0	0	12	114	114	1.000	0	0	12	114	114	1.000
0	0	13	114	114	1.000	0	0	13	114	114	1.000	0	0	13	114	114	1.000
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0	0	15	114	114	1.000	0	0	15	114	114	1.000	0	0	15	114	114	1.000
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0	0	21	114	114	1.000	0	0	21	114	114	1.000	0	0	21	114	114	1.000
0	0	22	114	114	1.000	0	0	22	114	114	1.000	0	0	22	114	114	1.000
0	0	23	114	114	1.000	0	0	23	114	114	1.000	0	0	23	114	114	1.000
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0	0	82	114	114	1.000	0	0	82	114	114	1.000						

Table 3. Fractional atomic coordinates of hydrogen atoms ($\times 10^3$)(Average value of the isotropic temperature factors of the hydrogen atoms is 2.63 \AA^2).

	x	y	z	Bonded to
H(1)	682 (9)	424 (9)	-236 (17)	N(1)
H(2)	585 (10)	412 (10)	-325 (20)	N(1)
H(3)	639 (10)	559 (9)	-79 (17)	C(1)
H(4)	579 (8)	566 (7)	-279 (14)	C(1)
H(5)	457 (9)	586 (7)	-35 (15)	C(2)
H(6)	418 (8)	517 (7)	-174 (15)	C(2)
H(7)	526 (9)	480 (8)	145 (16)	N(2)
H(8)	326 (11)	404 (10)	26 (21)	C(3)
H(9)	438 (11)	359 (9)	357 (18)	C(4)
H(10)	340 (11)	298 (10)	261 (21)	C(4)
H(11)	455 (12)	210 (10)	110 (19)	N(3)
H(12)	537 (11)	225 (9)	401 (17)	C(5)
H(13)	600 (9)	174 (9)	223 (17)	C(5)
H(14)	622 (8)	377 (7)	359 (15)	C(6)
H(15)	720 (9)	289 (8)	336 (17)	C(6)
H(16)	684 (8)	423 (7)	129 (14)	N(4)
H(17)	728 (11)	328 (10)	78 (20)	N(4)
H(18)	314 (11)	544 (11)	178 (21)	C(7)
H(19)	277 (12)	455 (11)	295 (21)	C(7)
H(20)	386 (11)	512 (10)	350 (22)	C(7)

the intensities of hkl and $h\bar{k}l$ with their calculated values. Comparison of observed and calculated differences showed that the complex ion, $(+)_{589}\text{-[Co(NO}_2)_2(\text{R-5-metrien})]^+$, has the absolute configuration *A* shown in Fig. 1 (*IUPAC Information Bulletin*, 1968). Calculations were carried out on the FACOM 230-30 computer at this Institute.

Table 4. The determination of the absolute configuration

<i>h</i>	<i>k</i>	<i>l</i>	$F_c(hkl)$	Obs.	$F_c(h\bar{k}l)$
4	1	1	655.4	<	745.3
6	1	1	590.5	>	488.4
7	1	1	610.1	<	772.8
9	1	1	1225.0	<	1376.4
11	2	1	1156.0	<	1239.0
1	3	1	2724.8	<	3237.6

Description and discussion of the structure

Fig. 1 shows a perspective drawing of the complex ion, which also represents the absolute configuration. The bond lengths and angles are listed in Table 5 with their e.s.d.'s. A cobalt atom is surrounded octahedrally by four nitrogen atoms from the trien ligand and two nitrogen atoms of the nitro groups. The nitro groups are in *cis* positions. The two nitrogen atoms, N(5) and N(6), are coordinated at distances of 1.92 and 1.94 Å respectively. The metrien molecule acts as a tetradentate ligand and is in the *cis-β* configuration. There are three five-membered chelate rings and the absolute configuration of the complex ion can be described as a skew-chelate pair *A*. The N-Co-N bond angles in the chelate rings I, II and III are compressed to 85.0, 86.4 and 85.8°, respectively. The bond angles around the two secondary nitrogen atoms and asymmetric car-

bon atom show significant deviation from the normal tetrahedral angle:

$$\text{C}(2)\text{-N}(2)\text{-C}(3) = 117.0 (7)^\circ$$

$$\text{C}(4)\text{-N}(3)\text{-C}(5) = 111.7 (8)$$

$$\text{N}(2)\text{-C}(3)\text{-C}(4) = 105.1 (7).$$

These deviations are very similar to those observed in crystals of $(-)_{546}\text{-cis-}\beta\text{-[Co(NO}_2)_2(\text{S-3,8-dimetrien})]\text{-ClO}_4$ (Ito, Marumo & Saito, 1970). The C-C distances in the chelate rings are in the range, 1.50~1.51 Å, in

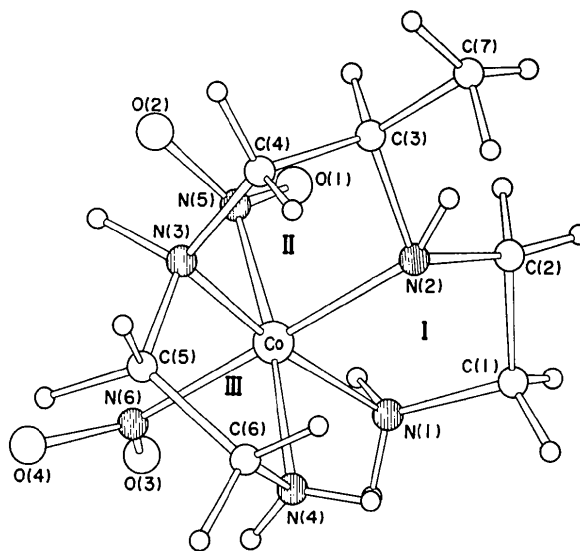
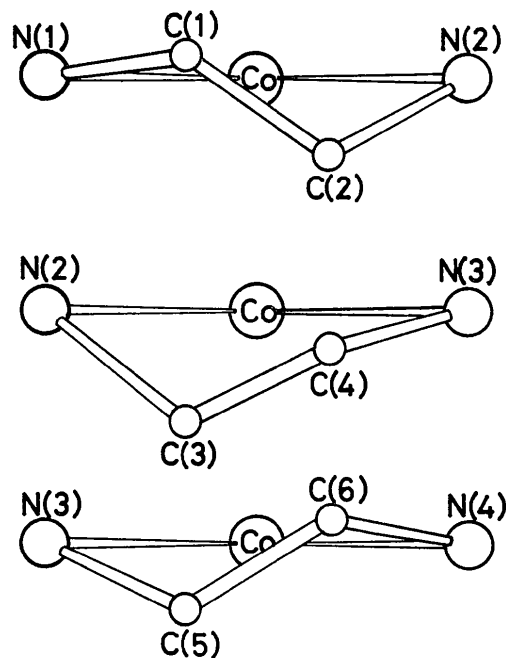
Fig. 1. A perspective drawing of the complex ion, $(+)_{589}\text{-cis-}\beta\text{-[Co(NO}_2)_2(\text{R-5-metrien})]^3+$.

Fig. 2. Conformations of the three chelate rings.

agreement with those in cobalt(III) complexes of linear aliphatic polyamines, e.g. *cis-β*-[Co-(trien)ClO₄]₂ (ClO₄)₂ (Freeman & Maxwell, 1969). The absolute configurations about the two asymmetric nitrogen atoms are both *S*. Fig. 2 represents conformations of the three chelate rings, I, II, and III.

Table 5. Bond lengths and angles with their *e.s.d.*'s

	Bond length (Å)		Bond angle (°)
Co-N(1)	1.945 (10)	N(1)-Co-N(2)	85.0 (3)
Co-N(2)	1.938 (9)	Co-N(1)-C(1)	111.0 (6)
Co-N(3)	1.960 (9)	N(1)-C(1)-C(2)	107.1 (6)
Co-N(4)	1.967 (9)	C(1)-C(2)-N(2)	105.2 (8)
Co-N(5)	1.922 (10)	C(2)-N(2)-Co	109.4 (6)
Co-N(6)	1.938 (10)		
N(1)-C(1)	1.481 (15)	N(2)-Co-N(3)	86.4 (3)
N(2)-C(2)	1.492 (14)	Co-N(2)-C(3)	107.5 (4)
N(2)-C(3)	1.500 (14)	N(2)-C(3)-C(4)	105.1 (7)
N(3)-C(4)	1.503 (16)	C(3)-C(4)-N(3)	113.6 (8)
N(3)-C(5)	1.484 (15)	C(4)-N(3)-Co	108.1 (4)
N(4)-C(6)	1.469 (15)	N(2)-C(3)-C(7)	113.4 (6)
		C(4)-C(3)-C(7)	112.9 (10)
C(1)-C(2)	1.506 (16)	N(3)-Co-N(4)	85.8 (3)
C(3)-C(4)	1.498 (19)	Co-N(3)-C(5)	108.3 (6)
C(5)-C(6)	1.514 (17)	N(3)-C(5)-C(6)	109.4 (6)
C(3)-C(7)	1.521 (19)	C(5)-C(6)-N(4)	107.4 (8)
		C(6)-N(4)-Co	111.7 (6)
N(5)-O(1)	1.220 (13)	O(1)-N(5)-O(2)	117.7 (9)
N(5)-O(2)	1.237 (13)	Co-N(5)-O(1)	123.2 (6)
N(6)-O(3)	1.205 (15)	Co-N(5)-O(2)	119.1 (7)
N(6)-O(4)	1.205 (14)	O(3)-N(6)-O(4)	119.1 (8)
		Co-N(6)-O(3)	119.0 (6)
		Co-N(6)-O(4)	121.9 (7)

Both terminal chelate rings, I and III, take asymmetrical skew conformations. The carbon atoms C(1) and

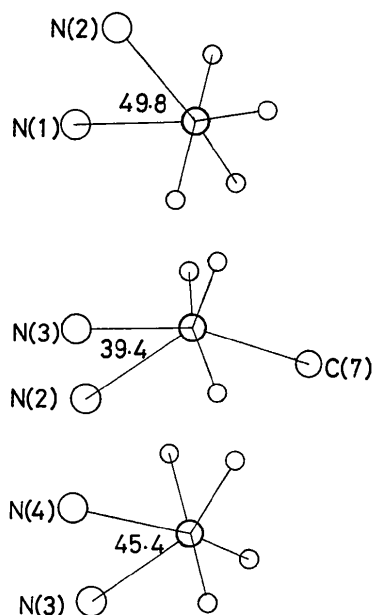


Fig. 3. The Newman projection of the chelate rings along the central C-C bond.

C(2) lie at -0.18 and 0.51 Å respectively from the plane through N(1), Co and N(2). C(5) and C(6) are off the plane formed by N(3), Co and N(4) at distances of 0.44 and -0.17 Å respectively. Similar asymmetrical skew conformations were observed in crystals of *cis-β*-[Co(trien)ClO₄]₂(ClO₄)₂. On the other hand the central ring, II, is different and the two carbon atoms lie on the same side of the plane through N(2), Co and N(3), the deviations being 0.78 and 0.26 Å respectively. The dihedral angles between the planes of the chelate rings are: I-II: 2.0 , II-III: 87.6 , I-III: 87.7° .

The conformation of the central chelate ring with methyl group is λ . The two outer chelate rings, I and III, have δ and λ conformations respectively. The methyl group lies in an equatorial position relative to the plane of the chelate rings. The Newman projections of the chelate rings along the central C-C bond are presented in Fig. 3. The dihedral angle NCCN in ring II with the substituted methyl group is significantly smaller than those of rings I and III without the methyl group. The same feature was observed in crystals of $(-)$ ₅₄₆-*cis-β*-[Co(NO₂)₂(*S*-3,8-dimetrien)]ClO₄.

There are two possible ways of coordinating an *R*-5-metrien ligand to a cobalt atom in *Λ-cis-β* coordination: namely the ligand can coordinate with the asymmetric carbon atom at C(3) or C(4) in Fig. 1. From the X-ray evidence, the methyl group is attached to C(3). This is certainly due to the repulsion of the equatorial hydrogen atom attached to N(3). H(10)-H(11) is as short as 2.2 Å. Thus it is very unlikely that the methyl group is attached to C(4) in place of a hydrogen atom in an equatorial position.

N-O distances and O-N-O angles in the nitro groups average 1.22 Å and 118.5° respectively, in agreement with those observed in other nitro complexes

Table 6. Interatomic distances less than 3.5 Å

	Distance	Symmetry operations applied to second atoms
Cl...N(1)	3.268 (10) Å	1
Cl...N(4)	3.430 (10)	1
Cl...N(3)	3.349 (9)	3
Cl...N(2)	3.283 (10)	5
Cl...N(4)	3.418 (10)	5
C(4)...O(1)	3.238 (15)	2
C(5)...O(3)	3.281 (16)	2
C(6)...O(3)	3.187 (16)	2
C(6)...O(2)	3.225 (16)	3
N(4)...O(2)	3.065 (13)	3
O(4)...C(3)	3.318 (16)	3
C(1)...O(2)	3.474 (15)	4

Key to symmetry operations

1	$x,$	$y,$	z
2	$x,$	$y,$	$1.0+z$
3	$0.5+x,$	$0.5-y,$	$-z$
4	$1.0-x,$	$0.5+y,$	$-0.5-z$
5	$1.5-x,$	$1.0-y,$	$-0.5+z$

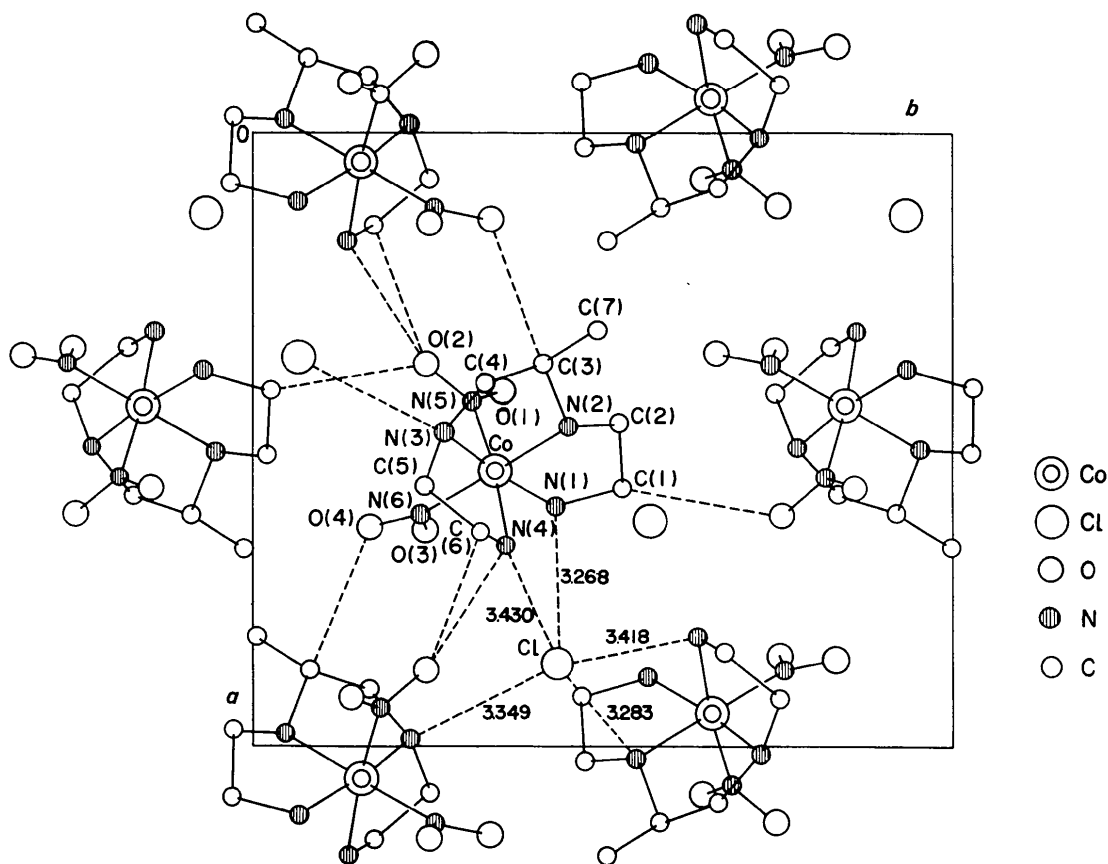


Fig. 4. A projection of the structure along the c axis. Broken lines indicate distances in the range of $3.2\sim 3.4$ Å.

(e.g. Ito, Marumo & Saito, 1970). The two nitro groups are coordinated to the cobalt atom with their O–O directions oriented nearly parallel to N(1)–Co–N(3). Such coplanarity of the nitro groups with one of the N–Co–N axes of the coordination octahedron was found in $(+)_589\text{-cis-dinitrobis}[(\text{-})_{589}\text{-1,2-propylenediamine}]\text{-Co(III)}$ chloride (Barclay, Goldschmied & Stephenson, 1970).

A projection of the structure is shown in Fig. 4. Intermolecular distances less than 3.5 Å are listed in Table 6. Short contacts occur between chloride ions and nitrogen atoms of the complex cation. The distances are in the range of $3.2\sim 3.4$ Å and they are indicated by broken lines in Fig. 4. A chloride ion is surrounded by five nitrogen atoms.

The complex ion shows a positive Cotton effect in the longer wavelength circular dichroism band in the region of the octahedral (T_{1g}) absorption and the net chirality is A , in conformity with the empirical rule relating circular dichroism spectra and absolute configuration of the complex ion (Hawkins & Larsen, 1965; Legg & Douglas, 1966).

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