Acta Cryst. (1975). B**31**, 1247

The Crystal Structure of $(+)_{589}$-*cis*-β-Carbonato-(3*S*,8*S*-dimethyltriethylenetetramine)cobalt(III) Perchlorate

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(*Received* 21 *November* 1974; *accepted* 7 *December* 1974)

$(+)_{589}$-*cis*-β-Carbonato-(3*S*,8*S*-dimethyltriethylenetetramine)cobalt(III) perchlorate, $(+)_{589}$-*cis*-β-[Co(CO$_3$) (3,8-dimetrien)]ClO$_4$ crystallizes in the space group $P2_12_12_1$, with four formula units in a cell of dimensions $a=12\cdot284$ (2), $b=17\cdot913$ (8) and $c=7\cdot179$ (3) Å. The structure was solved by the heavy-atom method and the positional and thermal parameters were refined by the method of least squares, using anisotropic thermal factors for the non-hydrogen atoms. The final R value for the 1894 observed reflexions collected by diffractometry is 0·041. The quadridentate ligand is linked to the central metal atom in *cis*-β coordination. One of the substituted methyl groups lies in an equatorial position with respect to the chelate ring, while the other is in an axial position. All the bond lengths are normal. The Co–N distances range from 1·935 (5) to 1·958 (5) Å. The absolute configuration of the complex ion can be designated as $\Lambda(\delta\lambda\lambda)$. The absolute configurations about the two secondary nitrogen atoms are both *R*.

Introduction

The investigation of the structure and absolute configuration of the present compound was undertaken as part of a series of structure determinations of cobalt(III) complexes containing (3*S*,8*S*)-dimethyltriethylenetetramine (Ito, Marumo & Saito, 1970, 1972*a*, *b*). There are four possible ways of coordinating a triethylenetetramine molecule, H$_2$NCH$_2$CH$_2$NHCH$_2$CH$_2$NHCH$_2$CH$_2$-NH$_2$: trien, in the *cis*-β configuration. Fig. 1 illustrates two of them which form a diastereoisomeric pair of *cis*-β-[Co(trien)X$_2$]$^{n+}$. The other two are enantiomers to those shown in the Figure. In the case of the unsubstituted trien molecule, the β-*R*,*S* isomer, in which the configurations about the two secondary nitrogen atoms are antimeric, was shown to be less stable than the β-*R*,*R* isomer in which the configurations about the two nitrogen atoms are the same (Buckingham, Marzilli & Sargeson, 1967; Sargeson & Searle, 1967). Crystal-structure analyses of three unsubstituted trien complexes showed that the absolute configurations of the two nitrogen atoms are the same (Freeman & Maxwell, 1969, 1970; Freeman, Marzilli & Maxwell, 1970). Buckingham, Marzilli & Sargeson (1967) suggested that the *cis*-β(*R*,*S*) form exists as a product of the Hg^{2+}-promoted acid hydrolysis reaction of the optically active *trans*-[CoCl$_2$(trien)]$^+$ ion. Saburi, Sawai & Yoshikawa (1972) used a substituted trien, (3*S*,8*S*)-dimethyltriethylenetetramine, and obtained crystals of Δ-*cis*-β-(*R*,*S*)-[Co(NO$_2$)$_2$(3,8-dimetrien)]ClO$_4$, of which the absolute configuration was verified by X-ray crystal-structure analysis (Ito *et al.*, 1970). This is because of the equatorial preference of substituted methyl groups which stabilized the δ-conformation of the two end chelate rings. Recently, crystals of carbonato-(3*S*,8*S*-dimethyltriethylenetetramine)cobalt(III) perchlorate were isolated and assigned to the Λ-*cis*-β-(*R*,*R*) isomer on the basis of circular dichroism and n.m.r. spectra (private communication from Professor Yoshikawa, to be published). The crystal was subjected to crystal structure analysis in order to establish its absolute configuration and conformational details.

Experimental

Orange-red crystals of $(+)_{589}$-*cis*-β-[Co(CO$_3$) (3,8-dimetrien)]ClO$_4$ were kindly supplied by Professor Yoshikawa of this University. Oscillation and Weissenberg photographs were taken with Cu $K\alpha$ radiation to determine the cell dimensions and the space group. The cell dimensions were refined by least-squares calculations on the basis of higher-order reflexions measured on a diffractometer (Mo $K\alpha$ radiation, $\lambda=0\cdot7107$ Å). They belong to the orthorhombic system, with unit-cell dimensions: $a=12\cdot284$ (2), $b=17\cdot913$ (8) and $c=7\cdot179$ (3) Å; $U=1579\cdot9$ Å^3. The space group is $P2_12_12_1$. There are four formula units in the unit cell ($D_x=1\cdot65$, $D_m=1\cdot65$ g cm^{-3} by flotation). The linear absorption coefficient, $\mu_{\text{Mo }K\alpha}=13\cdot4$ cm^{-1}.

Intensities were measured on a Rigaku automated four-circle diffractometer, using Mo $K\alpha$ radiation monochromated by a graphite plate. A crystal shaped into a sphere of 0·25 mm diameter was mounted with the c axis approximately parallel to the φ axis of the goniostat. Reflexions with 2θ less than 55° were collected with the ω–2θ scan technique at a scan rate of 4° min^{-1}. The scan range was calculated according to the formula $1\cdot5°+0\cdot5°\times\tan\theta$. Background counts were taken at each end of the scan. To check the electronic circuit and crystal stability during the period of data collection the intensities of three standard reflexions were measured every 50 reflexions. They showed 3·0% statistical fluctuation from the mean. In total, 2110 reflexions were measured. Reflexions with inten-

Table 1. *Atomic parameters*

(*a*) Positional and anisotropic thermal parameters for non-hydrogen atoms with their estimated standard deviations ($\times 10^4$). Thermal parameters refer to the expression $h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23}$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Co	6005 (1)	743 (0)	3715 (1)	28 (0)	15 (0)	53 (1)	3 (0)	−4 (1)	−2 (1)
N(1)	7146 (4)	925 (3)	1890 (7)	29 (3)	22 (2)	83 (9)	0 (2)	4 (5)	−7 (3)
N(2)	5875 (4)	1830 (3)	3848 (7)	34 (3)	18 (1)	101 (9)	4 (2)	−4 (6)	3 (4)
N(3)	4774 (4)	763 (3)	2034 (7)	32 (3)	22 (2)	93 (9)	4 (2)	−15 (5)	−11 (4)
N(4)	6010 (5)	−338 (3)	3306 (7)	54 (4)	18 (2)	85 (10)	2 (2)	−2 (6)	4 (3)
C(1)	7158 (6)	1725 (4)	1335 (11)	55 (5)	20 (2)	120 (13)	−4 (3)	21 (8)	5 (5)
C(2)	6867 (6)	2190 (4)	3037 (11)	46 (5)	19 (2)	155 (14)	−2 (3)	12 (7)	−2 (5)
C(3)	4865 (6)	2051 (4)	2780 (12)	50 (5)	20 (2)	176 (16)	14 (3)	−10 (8)	11 (5)
C(4)	4088 (6)	1404 (4)	2649 (11)	41 (5)	24 (2)	188 (16)	16 (3)	−30 (8)	−16 (5)
C(5)	4226 (6)	21 (4)	2188 (11)	40 (5)	28 (2)	169 (16)	−3 (3)	−8 (7)	−20 (6)
C(6)	5132 (6)	−541 (4)	1971 (11)	61 (6)	23 (2)	119 (13)	−3 (3)	−1 (8)	−10 (5)
C(7)	7754 (6)	2232 (5)	4482 (13)	48 (5)	32 (3)	218 (19)	−10 (3)	−3 (8)	−38 (7)
C(8)	3310 (8)	−82 (6)	786 (17)	70 (7)	45 (4)	418 (38)	3 (4)	−99 (14)	−72 (10)
O(*c*1)	6934 (4)	655 (3)	5870 (6)	43 (3)	27 (2)	90 (9)	3 (2)	−18 (4)	2 (3)
O(*c*2)	5177 (4)	625 (3)	5933 (6)	48 (3)	24 (2)	58 (8)	1 (2)	0 (4)	−0 (3)
O(*c*3)	6100 (5)	500 (3)	8621 (7)	95 (5)	40 (2)	60 (8)	2 (3)	−11 (7)	1 (4)
C(*c*4)	6061 (6)	589 (3)	6915 (8)	54 (4)	18 (2)	72 (9)	1 (3)	−18 (7)	−1 (4)
Cl	5557 (2)	3260 (1)	8012 (3)	56 (1)	30 (1)	132 (3)	4 (1)	−1 (2)	−2 (1)
O(*p*1)	4659 (6)	3719 (5)	8314 (16)	68 (5)	82 (4)	712 (43)	19 (4)	−18 (13)	−172 (12)
O(*p*2)	5269 (7)	2490 (4)	8050 (12)	151 (8)	41 (3)	332 (22)	−17 (4)	−62 (12)	12 (7)
O(*p*3)	5914 (10)	3400 (5)	6233 (12)	268 (14)	72 (4)	261 (19)	40 (7)	134 (18)	43 (9)
O(*p*4)	6393 (6)	3404 (5)	9325 (13)	94 (6)	66 (4)	458 (29)	−19 (4)	−99 (12)	14 (9)

sities greater than three times their standard deviations $\sigma(I)$ were regarded as 'observed'; thus 1894 reflexions were collected. The data were corrected for Lorentz and polarization effects, but no corrections were made for extinction or absorption.

Solution and refinement of the structure

The structure was solved by the heavy-atom method. The positions of the cobalt atoms were deduced from three-dimensional Patterson maps. The positions of the chlorine atoms and those of the other lighter atoms, apart from hydrogen, were determined by successive Fourier syntheses of electron density. Block-diagonal least-squares refinement using anisotropic temperature factors reduced the conventional R index to 0·053. At this stage a difference synthesis revealed all the hydrogen atoms in plausible positions. Further refinement was carried out including hydrogen atoms, where the thermal parameters of the hydrogen atoms were assumed to be isotropic. The final R index became 0·041 for the 1894 observed reflexions. At this stage the positional shifts were well within one half of the standard deviations of the positional parameters. Unit weight was given to all the reflexions. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The final set of atomic parameters is listed in Table 1, together with the estimated standard deviations. The observed and calculated structure amplitudes are listed in Table 2.*

Table 1 (*cont.*)

(*b*) Positional parameters for hydrogen atoms with their estimated standard deviations ($\times 10^3$).

Mean isotropic temperature factor of the hydrogen atoms is 3·5 Å^2.

	x	y	z
H(N11)	777 (7)	79 (5)	236 (13)
H(N12)	695 (7)	68 (5)	112 (15)
H(N21)	572 (11)	201 (7)	512 (18)
H(N31)	510 (7)	82 (5)	53 (13)
H(N41)	590 (7)	−51 (5)	428 (12)
H(N42)	659 (8)	−54 (6)	303 (15)
H(C11)	804 (9)	189 (6)	65 (17)
H(C12)	666 (7)	177 (5)	20 (13)
H(C21)	679 (7)	275 (5)	265 (13)
H(C31)	463 (7)	254 (5)	307 (13)
H(C32)	509 (7)	220 (5)	127 (14)
H(C41)	379(7)	134 (5)	411 (13)
H(C42)	358 (7)	144 (5)	192 (13)
H(C51)	394 (7)	−2 (5)	366 (13)
H(C61)	529 (7)	−63 (5)	88 (13)
H(C62)	488 (7)	−110 (5)	244 (15)
H(C71)	787 (7)	184 (5)	484 (13)
H(C72)	836 (7)	249 (5)	390 (14)
H(C73)	762 (8)	252 (6)	566 (14)
H(C81)	284 (9)	27 (6)	80 (17)
H(C82)	364 (7)	0 (5)	−59 (13)
H(C83)	306 (9)	−59 (6)	70 (17)

Determination of the absolute configuration

The absolute configuration of the complex ion was determined with reference to the known absolute configuration of the ligand. In order to check this result, equi-inclination Weissenberg photographs were taken with Cu $K\alpha$ radiation. Table 3 lists some of the intensities of hkl and $h\bar{k}l$ with their calculated values. Comparison of observed and calculated differences indicates that the complex ion $(+)_{589}$-*cis*-β-[Co(CO$_3$) (3,8-di-

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* Table 2 has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30818 (15 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

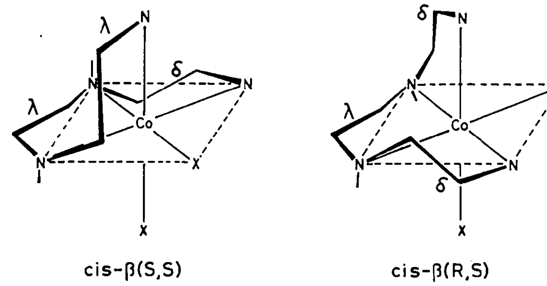

Fig. 1. A diastereoisomeric pair of *cis*-β-$[Co(trien)X_2]^{n+}$ ions.

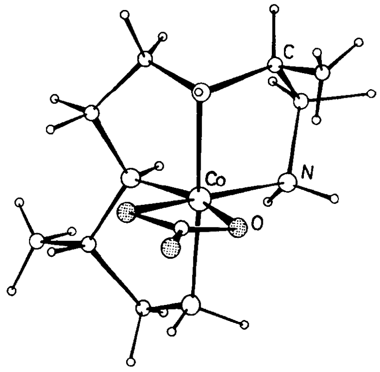

Fig. 2. A perspective drawing of the complex ion, $(+)_{589}$-*cis*-β-$[Co(CO_3)$ (3,8-dimetrien)$]^+$.

Table 3. *The relationship between F(hkl) and F($\bar{h}\bar{k}\bar{l}$) in their calculated and observed values*

h	k	l	$F_c(hkl)$	Observed	$F_c(\bar{h}\bar{k}\bar{l})$
1	4	1	32	>	21
2	1	1	28	<	36
3	5	1	28	<	39
3	8	1	32	>	21
4	2	1	18	>	3
4	4	1	38	>	25
4	6	1	27	>	16
6	3	1	24	<	37
6	5	1	39	>	27
8	2	1	14	<	23
8	6	1	24	>	15

metrien)$]^+$ has the absolute configuration Λ illustrated in Fig. 2 (*IUPAC Information Bulletin*, 1968).

Description of the structure and discussion

A perspective drawing of the complex ion $(+)_{589}$-*cis*-β-$[Co(CO_3)$(3,8-dimetrien)$]^+$ is presented in Fig. 2. It correctly represents the absolute configuration. The interatomic distances and bond angles within the complex ion are given in Table 4. Projections of the structure along the *c* and *b* axes are shown in Figs. 3 and 4, respectively. A molecule of 3,8-dimetrien is linked to the central cobalt atom in *cis*-β coordination. The cobalt atom is surrounded by four nitrogen atoms and two oxygen atoms at the apices of a slightly distorted octahedron. As discerned from Table 4, one of the

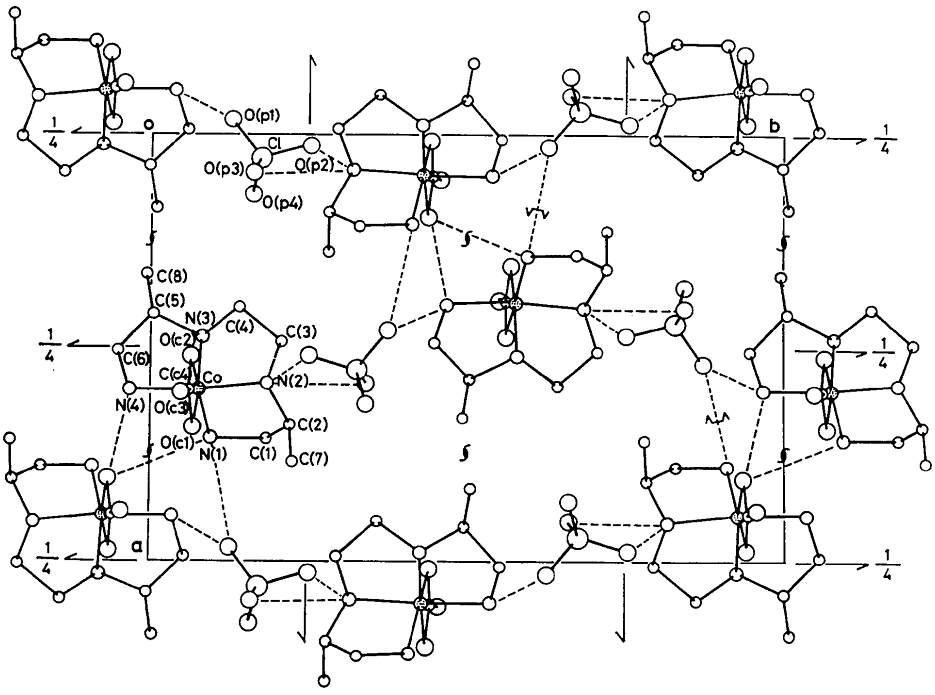

Fig. 3. A projection of the structure along the *c* axis.

two secondary nitrogen atoms N(3), which connects the two chelate rings in the same coordination plane, is bonded to the cobalt atom with a significantly shorter distance than other Co–N distances. Such shortening is also observed for the two secondary nitrogen atoms in *trans*-[Co(NO$_2$)$_2$(3,8-dimetrien)]$^+$ (Ito *et al.*, 1972*b*). The Co–N distances may be compared with those in other related compounds: (−)$_{589}$-*cis*-*β*-[Co(NO$_2$)$_2$(3,8-dimetrien)]ClO$_4$; (+)$_{546}$-*trans*-[Co(NO$_2$)$_2$(3,2,3-tet)]Br$_3$ and (−)$_{589}$-[CoCl$_2$(3,2,3-tet)](NO$_3$) (Payne, 1972, 1973).* The average angle subtended at the cobalt atom by the chelate rings is compressed to 86·1 (2)°. But this angle by the central chelate ring is slightly larger at 86·7 (2)° as in the case of the *cis*-*α*-dinitro analogue. The three five-membered chelate rings adopt the *gauche* conformation, and have absolute configurations *λ*, *λ* and *δ*, respectively, which are shown in Fig. 5. These absolute configurations of the chelate rings are the same as in the triethylenetetramine ligand of (+)$_{589}$-*cis*-*β*-[Co(S-pro)(trien)]-ZnCl$_4$ (Freeman & Maxwell 1970). One of the two substituted methyl groups, C(7), is bonded axially with respect to the chelate ring, whereas the other, C(8), is in an equatorial position. If both methyl groups were in equatorial positions, the absolute configurations of the chelate rings would become *δ*, *λ* and *δ*, respectively. The absolute con-

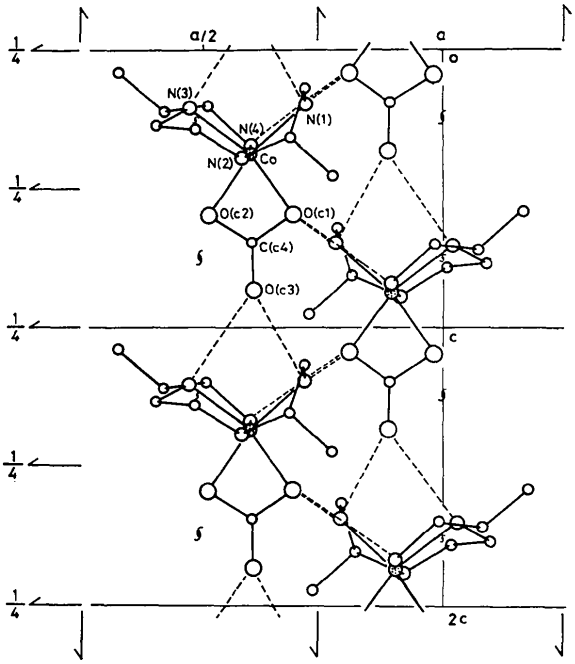

Fig. 4. A projection of the structure along the *b* axis.

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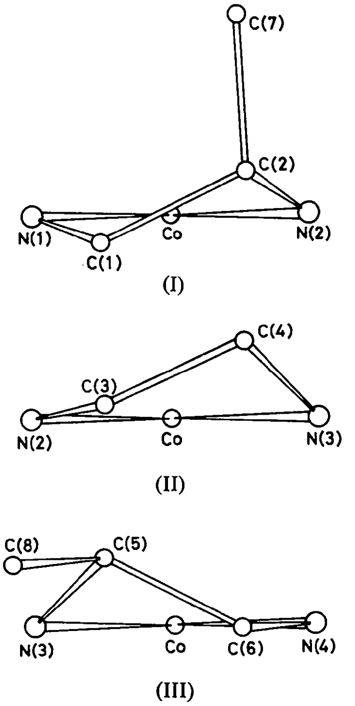
* 3,2,3-tet: 1,10-diamino-4,7-diazadecane, H$_2$N(CH$_2$)$_3$NH(CH$_2$)$_2$NH-(CH$_2$)$_3$NH$_2$.

Table 4. *Bond lengths* (Å) *and angles* (°) *with their estimated standard deviations*

Co—N(1)	1·946 (5)	C(1)—C(2)	1·522 (10)
Co—N(2)	1·957 (5)	C(3)—C(4)	1·504 (10)
Co—N(3)	1·935 (5)	C(5)—C(6)	1·509 (10)
Co—N(4)	1·958 (5)	C(2)—C(7)	1·507 (11)
Co—O(*c*1)	1·929 (4)	C(5)—C(8)	1·520 (13)
Co—O(*c*2)	1·901 (4)	O(*c*1)–C(*c*4)	1·314 (8)
N(1)–C(1)	1·488 (9)	O(*c*2)–C(*c*4)	1·296 (8)
N(2)–C(2)	1·496 (9)	O(*c*3)–C(*c*4)	1·236 (8)
N(2)–C(3)	1·511 (9)	Cl——O(*p*1)	1·392 (8)
N(3)–C(4)	1·492 (9)	Cl——O(*p*2)	1·423 (8)
N(3)–C(5)	1·495 (9)	Cl——O(*p*3)	1·373 (9)
N(4)–C(6)	1·487 (10)	Cl——O(*p*4)	1·418 (9)
N(1)–Co—N(2)	85·7 (2)	O(*c*1)–Co—O(*c*2)	68·6 (2)
N(2)–Co—N(3)	86·7 (2)	Co——O(*c*1)–C(*c*4)	89·0 (4)
N(3)–Co—N(4)	85·9 (2)	Co——O(*c*2)–C(*c*4)	90·8 (4)
Co—N(1)–C(1)	110·4 (3)	O(*c*1)–C(*c*4)–O(*c*2)	111·6 (5)
Co—N(2)–C(2)	110·1 (3)	O(*c*1)–C(*c*4)–O(*c*3)	123·1 (6)
Co—N(2)–C(3)	107·6 (3)	O(*c*2)–C(*c*4)–O(*c*3)	125·3 (6)
Co—N(3)–C(4)	105·8 (3)	O(*p*1)–Cl——O(*p*2)	111·9 (3)
Co—N(3)–C(5)	106·8 (3)	O(*p*1)–Cl——O(*p*3)	106·9 (5)
Co—N(4)–C(6)	109·6 (4)	O(*p*1)–Cl——O(*p*4)	111·3 (4)
N(1)–C(1)–C(2)	108·1 (5)	O(*p*2)–Cl——O(*p*3)	105·9 (4)
N(2)–C(2)–C(1)	105·6 (4)	O(*p*2)–Cl——O(*p*4)	110·4 (4)
N(2)–C(3)–C(4)	110·6 (3)	O(*p*3)–Cl——O(*p*4)	110·7 (5)
N(3)–C(4)–C(3)	104·6 (5)		
N(3)–C(5)–C(6)	104·6 (5)		
N(4)–C(6)–C(5)	107·8 (4)		
N(2)–C(2)–C(7)	110·1 (6)		
N(3)–C(5)–C(8)	113·1 (6)		
C(2)–N(2)–C(3)	111·0 (5)		
C(4)–N(3)–C(5)	114·1 (4)		
C(1)–C(2)–C(7)	114·2 (6)		
C(6)–C(5)–C(8)	113·4 (6)		

figurations about the two asymmetric nitrogen atoms are both *R*. The average C–N distance of 1·495 (9) Å and C–C distance of 1·512 (10) Å are comparable with those observed in other trien structures. The bond angles including N(3), C(4)–N(3)–C(5), N(3)–C(4)–C(3) and N(3)–C(5)–C(6) are largely distorted from the strain-free angle. The carbonatocobalt group is shown in Fig. 6. In the carbonatocobalt ring the Co–O(*c*1) bond is significantly longer than the Co–O(*c*2) bond. This difference may be owing to hydrogen-bond formation between O(*c*1) and N(1) and between O(*c*1) and N(4) (see Table 5). The carbonato group is planar but makes an angle of 4·3° with the plane formed by Co, N(1) and N(3). The dimensions of the coordinated carbonato group closely resemble those of other cobalt–carbonato systems reported (Kaas & Sørensen, 1973; Geue & Snow, 1971; Barclay & Hoskins, 1962).



(I)

(II)

(III)

Fig. 5. Conformation of the chelate rings.

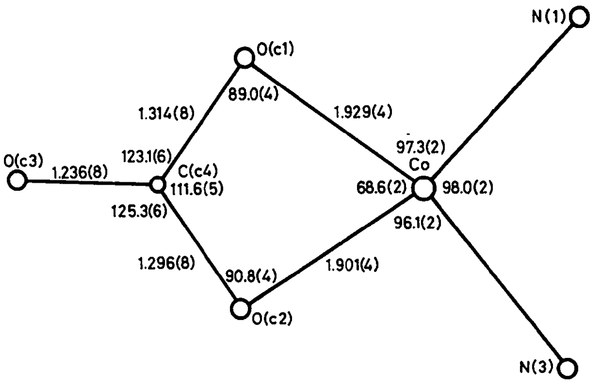


Fig. 6. Bond lengths and angles of the carbonatocobalt(III) system.

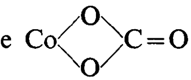
Table 5. *Hydrogen-bond distances* (Å) *and angles* (°) *with their estimated standard deviations*

			Symmetry operations applied to first atoms
N(1)–H···O(*c*3)	2·782 (7)	152 (10)	i′
N(3)–H···O(*c*3)	2·980 (7)	149 (5)	i′
N(1)–H···O(*c*1)	3·134 (7)	113 (5), 103 (4)	iv
N(4)–H···O(*c*1)	3·124 (7)	148 (9)	iv
N(1)–H···O(*p*1)	3·156 (9)	130 (6)	iii
N(4)–H···O(*p*1)	3·069 (12)	164 (6)	ii
N(2)–H···O(*p*2)	3·324 (10)	176 (10)	i
N(2)–H···O(*p*3)	3·294 (10)	125 (2)	i

Key to symmetry operations

i	*x*	*y*	*z*
ii	1·0−*x*	0·5+*y*	1·5−*z*
iii	−0·5+*x*	0·5−*y*	1·0−*z*
iv	1·5−*x*	−*y*	0·5+*z*
i′	*x*	*y*	1·0+*z*

The shape and the size of the perchlorate ion are normal, the average Cl–O distance being 1·402 (8) Å. No orientational disorder of the anion was observed as in many other related perchlorates.

The crystal is composed of discrete ions, linked by weak hydrogen bonds, which form a three-dimensional network. Selected hydrogen-bond distances and angles are listed in Table 5. A complex cation is linked to the adjacent one, related to the first by a unit translation along the *c* axis, by strong hydrogen bonds between the oxygen atom O(*c*3) of a carbonato group and the amino nitrogen atoms N(1) and N(3). The complex ions form an infinite chain along the *c* axis. Two such chains are laterally held together by N(1)···O(*c*1) and N(4)···O(*c*1) hydrogen bonds. Thus the complex cations form a double chain structure around the two-fold screw axis parallel to the *c* axis. Such an arrangement suggests dipole–dipole interactions between the complex cations of which the dipole moment might be parallel to the  direction. In fact, the Co···Co distance in the chain is 5·784 Å. The Co···Co distances range from 6·20 to 7·0 Å in other related *cis*-dinitro complexes.

The authors are grateful to Professor Yoshikawa and Dr Saburi of this University for providing crystals of $(+)_{589}$-*cis*-*β*-[Co(CO_3) (3,8-dimetrien)]ClO_4. The calculations were performed on the FACOM 270–30 at this Institute with a local version of the Universal Crystallographic Computation Program System (Crystallographic Society of Japan). This research was aided by a Scientific Research Grant from the Ministry of Education, to which the authors' thanks are due.

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Structure Cristalline du Monoséléniure d'Indium InSe

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(*Reçu le* 11 *octobre* 1974, *accepté le* 29 *novembre* 1974)

Indium monoselenide (InSe) is rhombohedral with space group $R3m$. The hexagonal unit cell contains six formula units with $a_h = 4{\cdot}00$ Å, $c_h = 25{\cdot}32$ Å. A reliability index (R) of 0·077 was obtained after a least-squares refinement of the atomic positions. This structure can be considered as being formed of double layers of selenium, parallel to the (001) plane, between which occur pairs of indium atoms.

Le monoséléniure d'indium InSe a déjà fait l'objet de deux études. Schubert, Dörre & Grünzel (1954) proposent un réseau rhomboédrique avec $a_h = 4{,}02$ Å, $C_h = 25{,}00$ Å, mais n'indiquent pas le groupe spatial ni la structure. Semiletov (1958) décrit la structure cristalline par diffraction d'électrons à partir d'une couche mince, obtenue par vaporisation. Il conclut à une maille hexagonale: groupe spatial $P6_3/mmc$ avec $a = 4{,}05$ Å, $c = 16{,}93$ Å, $Z = 4$. La distance In–In obtenue par Semiletov (3,16 Å) est nettement supérieure à celles qui sont généralement observées dans les structures des autres séléniures d'indium déjà décrits. Dans In_6Se_7 Hogg & Duffin (1971) obtiennent une distance égale à 2,76 Å et dans In_4Se_3 Likforman & Etienne (1972) trouvent des valeurs de 2,75 et 2,77 Å, confirmées par Hogg, Sutherland & Williams (1973). De même la distance In–Se égale à 2,51 Å nous paraît nettement trop courte.

Nous avons fait cette étude par diffraction des rayons X sur des monocristaux obtenus par chauffage des éléments à 800°C, en ampoule de silice scellée sous vide, pendant trois jours puis refroidissement jusqu'à la température ordinaire. Les paramètres de la maille hexagonale triple sont $a = 4{,}00 \pm 0{,}01$, $c = 25{,}32 \pm 0{,}03$ Å. Ceux de la maille rhomboédrique $a = 8{,}76$ Å, $\alpha = 26{,}40°$.

La densité expérimentale est de 5,53; la masse volumique calculée avec six masses formulaires par maille hexagonale triple est de 5,51 g cm^{-3}. La structure feuilletée des cristaux les rend très fragiles, au point d'être brisés du seul fait d'être mis en contact avec de la colle. Pour pallier cette difficulté nous avons choisi un gros cristal parallélépipédique dont les dimensions sont $60 \times 160 \times 400$ μm. Les clichés de Weissenberg nous ont montré que ce parallélépipède contenait en fait deux cristaux maclés de tailles inégales, dont le plus petit n'était cependant pas négligeable devant le plus gros. Dans l'impossibilité d'obtenir un cristal unique en bon état, nous avons décidé de poursuivre notre étude sur cette macle. La Fig. 1 représente la coupe du réseau direct par le plan xOy. La rangée [130] est un axe de symétrie supplémentaire d'ordre 2 qui n'existe pas dans le cristal. Les rangées [100] et [001] déterminent le plan de macle. La rangée [130] étant exactement perpendiculaire à ce plan réticulaire, l'obliquité de la macle est nulle. Il s'agit donc d'une macle par mériédrie réticulaire.

Les réflexions des deux individus sont exactement