

atom is irregular and may be derived from a distorted octahedral complex with a vacant coordination site. The nearest neighbour of the Tl atom on the side distant from the N and two O atoms is the pyrrole N atom of an adjacent dimer ( $\text{Tl} \cdots \text{N} = 3.43 \text{ \AA}$ ). The Tl atom has no other contacts shorter than  $4.0 \text{ \AA}$  with atoms outside its own dimer. The  $(\text{CH}_3)_2\text{Tl}$  unit is nearly linear with  $\text{C-Tl-C} = 165^\circ$ . With no significant close contacts to the Tl atom in the crystal, the large  $\text{C-Tl-C}$  angle cannot be explained by intermolecular interactions. The vacant coordination site and the observation that all the identifiable bonds lie on one side of the Tl atom suggest that the large  $\text{C-Tl-C}$  angles are due to internal bonding factors. Similar characteristics were found for both the dimeric  $[(\text{CH}_3)_2\text{TLX}]_2$  derivatives (where X is a good bridging group leading to dimer formation) (Burke, Gray, Hayward, Matthews, McPartlin & Gillies, 1977) and the polymeric  $[(\text{alkyl})_2\text{TLX}]_n$  derivatives (Chow & Britton, 1975a,b,c; Milburn & Truter, 1967).

The Tl-C lengths are in the range found for Tl-C bonds in similar  $(\text{CH}_3)_2\text{Tl}^+$  dimer species,  $2.11$  to  $2.20 \text{ \AA}$  (Chow & Britton, 1975a,b,c; Burke *et al.*, 1977), but the Tl-O lengths are significantly longer than the  $2.36$  to  $2.40 \text{ \AA}$  found for  $\text{Tl}^{\text{III}}$  phenoxide derivatives (Burke *et al.*, 1977). The Tl-N length of  $2.66$  ( $1$ )  $\text{ \AA}$  is also longer than the Tl-N distances of  $2.46$  and  $2.57 \text{ \AA}$  from pyridyl N atoms (Deacon, Faulks, Gatehouse & Jozsa, 1977). The ligand dimensions in the [DL-TRP] residue in  $(\text{CH}_3)_2\text{Tl}[\text{DL-TRP}]$  do not differ significantly from those found for TRPH.HBr (Takigawa, Ashida, Sasada & Kakudo, 1966) or for TRPH formate (Bye, Mostad & Rømming, 1973).

There are three hydrogen bonds involving the  $\text{H}_2\text{O}$  molecule (Table 2) which serve to link the dimer units into infinite chains (Fig. 2) and to tie adjacent dimer chains together.

The  $^1\text{H}$  NMR spectrum of  $(\text{CH}_3)_2\text{Tl}(\text{DL-TRP})$  in aqueous solution (pH 7) shows a value of  $420 \pm 2\text{ Hz}$

for  $^2J(^{205}\text{Tl}-^1\text{H})$ . This is close to the values of  $^2J(^{205}\text{Tl}-^1\text{H})$  found for other  $(\text{CH}_3)_2\text{TLX}$  derivatives in aqueous solution (Burke, Matthews & Gillies, 1976).

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### *mer-cis(N)-cis(O)-Bis(glycinato)aminoethanolatocobalt(III) Hydrate*

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**Abstract.** Racemic *mer-cis(N)-cis(O)-[Co(NH<sub>2</sub>CH<sub>2</sub>-COO)<sub>2</sub>(NH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O)].H<sub>2</sub>O*, FW 285.1, monoclinic,  $P2_1/n$ ,  $Z = 4$ ,  $a = 9.687$  (3),  $b = 11.957$  (3),  $c = 9.629$  (3)  $\text{ \AA}$ ,  $\beta = 103.75$  (2) $^\circ$ ,  $U = 1083 \text{ \AA}^3$ ;  $\mu(\text{Mo } K\alpha)$

( $\lambda = 0.71069 \text{ \AA}$ ) =  $16.7 \text{ cm}^{-1}$ ;  $D_m = 1.746$ ,  $D_x = 1.748 \text{ g cm}^{-3}$ . Block-diagonal least-squares calculations based on 2544 independent counter intensities reduced the  $R$  factor to 0.041. The average Co-O-

(carboxyl) distance is 1.920 (4) Å, whereas an alkoxide O atom is coordinated to Co at 1.895 (5) Å.

**Introduction.** The structure of *mer-cis(N)-cis(O)-[Co(gly)<sub>2</sub>(eta)].H<sub>2</sub>O* (gly = glycinato, eta = aminoethanolato) has been determined as part of a series of studies on aminoethanolatocobalt(III) complexes (Miyamae, Nishide, Ogino, Sato & Saito, 1976). Another aim was to determine the orientation of the complex molecule in the unit cell. This is necessary for the interpretation of single-crystal absorption spectra by plane-polarized light (Shibata, to be published).

Blue platy crystals with a metallic lustre were kindly supplied by Professor M. Shibata of Kanazawa University. They are stable in air and X-radiation. Intensities were collected on a Rigaku automated four-circle diffractometer with a crystal of dimensions 0.3 × 0.3 × 0.2 mm with Mo K $\alpha$  radiation monochromated by a graphite plate; the  $\omega$ -2 $\theta$  scan technique was employed. 3685 reflexions were measured up to 2 $\theta$  = 60°, of which 2544 with  $|F| > 3\sigma(|F|)$  were utilized for structure determination. Corrections were made for Lorentz and polarization factors but not for absorption. The positions of Co and its ligating atoms were deduced from a Patterson synthesis. The remaining lighter atoms could be located by the routine application of Fourier methods. By assuming anisotropic thermal parameters for non-hydrogen atoms and isotropic ones for H atoms, the atomic parameters were refined to give a final *R* value of 0.041 for 2544 independent reflexions. The atomic scattering factors for all atoms were those listed in *International Tables for X-ray Crystallography* (1974). Table 1 lists the final atomic coordinates.\*

**Discussion.** Fig. 1 shows a partial projection of the structure along *c* and the labelling of the atoms. The complex and water molecules are linked by N—H...O [N(1)...O(3<sup>ii</sup>)† 2.888 (5), N(2<sup>iii</sup>)...O(1) 2.914 (5), N(3<sup>iii</sup>)...O(4) 2.996 (6) and N(3)...OW 2.978 (7) Å] and O—H...O [OW...O(3<sup>i</sup>) 2.752 Å] hydrogen bonds to form a three-dimensional network.

In the neutral complex molecule, the Co atom is surrounded nearly octahedrally by three N and three O atoms. Fig. 2 gives interatomic distances and bond angles within the molecule. The Co-eta ring is not planar and the central C—C bond in the chelate ring is nearly parallel to the pseudo threefold axis of the

Table 1. *Final positional parameters* ( $\times 10^4$ , for H  $\times 10^3$ ) *with estimated standard deviations in parentheses*

|        | <i>x</i> | <i>y</i>  | <i>z</i> |
|--------|----------|-----------|----------|
| Co     | 2445 (1) | 948 (1)   | 1627 (1) |
| N(1)   | 1771 (4) | 924 (4)   | -454 (4) |
| N(2)   | 3146 (4) | -568 (3)  | 1715 (4) |
| N(3)   | 2916 (4) | 1075 (4)  | 3716 (4) |
| O(1)   | 1898 (4) | 2487 (3)  | 1472 (3) |
| O(2)   | 4309 (3) | 1385 (3)  | 1438 (4) |
| O(3)   | 615 (3)  | 475 (3)   | 1765 (4) |
| O(4)   | 1182 (4) | 3852 (3)  | -67 (4)  |
| O(5)   | 6455 (4) | 671 (4)   | 1496 (5) |
| C(1)   | 1583 (7) | 2079 (4)  | -996 (5) |
| C(2)   | 1535 (5) | 2888 (4)  | 199 (5)  |
| C(3)   | 4716 (5) | -552 (4)  | 1972 (6) |
| C(4)   | 5215 (5) | 562 (4)   | 1611 (5) |
| C(5)   | 1547 (6) | 1095 (6)  | 4161 (6) |
| C(6)   | 581 (6)  | 247 (5)   | 3198 (6) |
| OW     | 3529 (5) | 2956 (4)  | 5799 (5) |
| H1(N1) | 248 (7)  | 26 (6)    | -87 (7)  |
| H2(N1) | 98 (7)   | 31 (6)    | -73 (7)  |
| H1(N2) | 294 (14) | -161 (12) | 91 (14)  |
| H2(N2) | 302 (14) | -183 (11) | 248 (14) |
| H1(N3) | 339 (7)  | 23 (6)    | 412 (7)  |
| H2(N3) | 343 (6)  | 179 (5)   | 407 (6)  |
| H1(C1) | 239 (8)  | 226 (7)   | -146 (8) |
| H2(C1) | 83 (7)   | 211 (6)   | -169 (7) |
| H1(C3) | 510 (8)  | -33 (6)   | 292 (8)  |
| H2(C3) | 509 (9)  | -168 (8)  | 154 (9)  |
| H1(C5) | 169 (13) | 78 (11)   | 519 (13) |
| H2(C5) | 110 (6)  | 192 (5)   | 405 (6)  |
| H1(C6) | -42 (6)  | 13 (5)    | 337 (6)  |
| H2(C6) | 85 (6)   | -22 (5)   | 347 (6)  |
| H1(OW) | 294 (7)  | 309 (5)   | 625 (7)  |
| H2(OW) | 411 (7)  | 312 (6)   | 616 (7)  |

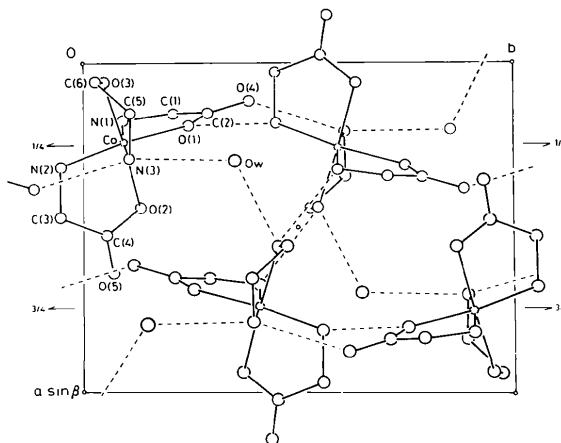


Fig. 1. Partial projection of the crystal structure along *c*.

\* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33178 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

† Symmetry code: (i)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (ii)  $-x, -y, -z$ ; (iii)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ .

complex (7.6°). Thus the chelate ring takes the *lel* conformation. The mean Co—N bond length of 1.948 (4) Å is slightly shorter than the normal Co—N(primary amino) distance of 1.978 Å.

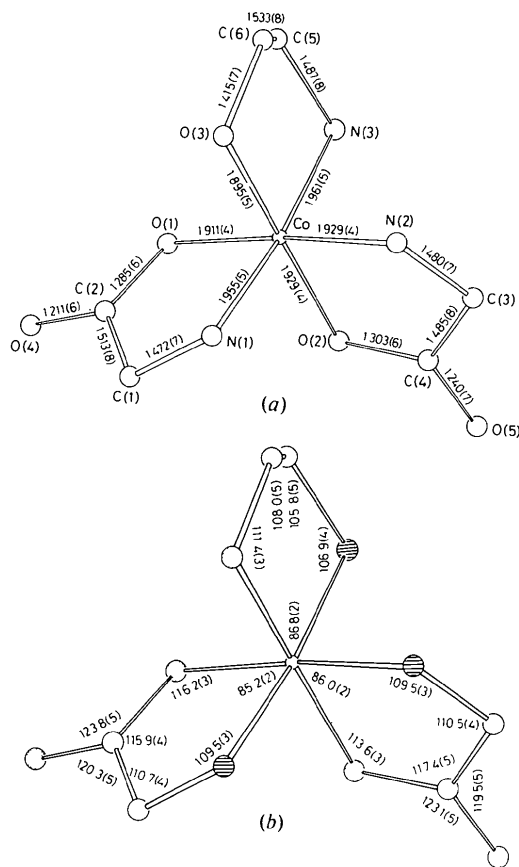


Fig. 2. Projection of the complex molecule on the N(1)–N(2)–O(3) plane with (a) interatomic distances (Å) and (b) bond angles (°).

Fig. 3 shows projections of the three five-membered chelate rings along the central C–C bonds. The shapes and sizes of the two glycinate moieties are normal. The glycinate rings are nearly planar. N–C–C–O (ligating O atom) torsion angles are 11.4 and 14.1°.

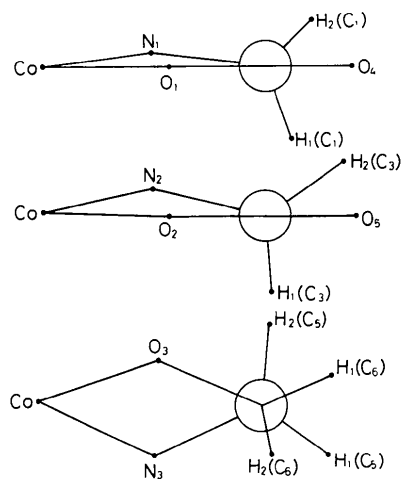


Fig. 3. Projections of the five-membered chelate rings along the central C–C bonds.

In contrast, the dihedral angle N–C–C–O in eta is 49.7°, indicating a strain-free conformation of the chelate ring. The Co–O(eta) distance of 1.895 (5) Å is significantly shorter than the average Co–O(gly) distance of 1.920 (5) Å. This shorter Co–O(eta) distance may be ascribed to a greater charge localization on this O atom than on the carboxyl O atom.

Calculations were carried out on the FACOM 230/48 computer of this Institute.

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### Polymeric (2,2'-trimethylenedipyridine 1,1'-dioxide)bis[dichloromercury(II)]

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**Abstract.** C<sub>13</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>Cl<sub>4</sub>Hg<sub>2</sub>, M<sub>r</sub> = 773.0, orthorhombic, *Fdd*2, *a* = 46.465 (21), *b* = 18.903 (12), *c* = 4.238 (5) Å, *U* = 3722.4 Å<sup>3</sup>, *Z* = 8, *d<sub>c</sub>* = 2.76, *d<sub>m</sub>* =

2.72 (5) g cm<sup>-3</sup>, Mo K $\alpha$  radiation,  $\lambda$  = 0.7107 Å,  $\mu$  = 177.0 cm<sup>-1</sup>. Each 2,2'-trimethylenedipyridine 1,1'-dioxide ligand is bonded through O to four HgCl<sub>2</sub>