Table 2. *Hydrogen-bond parameters* (Å *and* deg)

Donor (D)	Acceptor (A)	D–A	H–A	D–H–A
N(1)	N(3^{i})	2·959 (3)	2·12 (3)	170 (3)
N(1)	O(W3^{ii})	2·802 (3)	1·95 (3)	169 (3)
N(4)	O(W2^{iii})	2·870 (3)	2·13 (3)	140 (2)
O(W1)	O(13^{ii})	2·909 (3)	2·12 (4)	155 (3)
O(W1)	O(12^{iv})	2·935 (2)	2·11 (4)	165 (4)
O(W2)	O(2^{v})	2·882 (2)	2·06 (4)	170 (4)
O(W2)	O(W1)	2·786 (3)	1·89 (5)	172 (3)
O(W3)	O(11^{vi})	2·846 (3)	2·04 (3)	171 (3)
O(W3)	O(2^{iii})	2·958 (3)	2·24 (4)	150 (4)

Symmetry code for superscripts: none x,y,z; (i) $3-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $2-x, 1-y, 1-z$; (iv) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (v) $-1+x, y, z$; (vi) $-1+x, y, 1+z$.

Table 3. *Sodium-ion coordination* (Å)

Na–O(2)	2·471 (2)	Na–O(W1^{iii})	2·475 (2)
Na–O(12^{i})	2·391 (2)	Na–O(W2)	2·344 (2)
Na–O(13^{ii})	2·323 (2)	Na–O(W3^{iv})	2·652 (2)

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

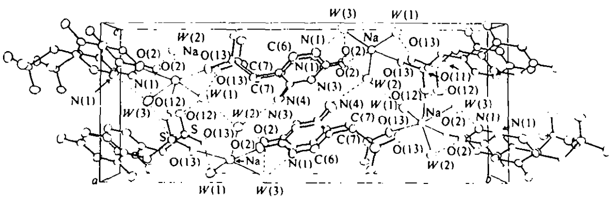

Fig. 2. Packing interactions. Two layers along the **a** direction are shown in perspective. The dashed lines represent hydrogen bonds. The fine solid lines represent sodium coordination. A hydrogen bond from N(4) to N(3) is not included in the drawing (Table 2).

Fig. 2 is a view of the packing interactions. The anions are packed with the aromatic rings stacked, forming columns along the **a** direction. The closest contacts are N(3)···C(6) 3·466 (3), N(4)···C(7) 3·431 (3), and O(11)···O(12) 3·573 (2) Å. The columns form pairs linked by hydrogen bonds from N(4) to N(3) and are further bridged by hydrogen bonds to water molecules (Table 2) and by coordination to the Na^+ ion (Table 3).

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Structure of *lel lel lel* Tris[(±)-2,3-butanediamine]cobalt(III) Chloride

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Abstract. $[Co(C_4H_{12}N_2)_3]Cl_3$, trigonal, space group $P\bar{3}1c$, $a = 12{\cdot}275\ (4)$, $c = 7{\cdot}874\ (1)$ Å, $Z = 2$. The structure was refined anisotropically to $R = 0{\cdot}039$, $R_w = 0{\cdot}045$ for 988 observed reflections. The cationic complexes have D_3 symmetry and are hydrogen bonded to chloride anions with N···Cl contacts of two

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each at 3·234 (2) and 3·404 (2) Å. The chelate ring N—C—C—N dihedral angle is 55·1 (3)°.

Introduction. Although there has been much interest in the conformations of chelate rings formed by 2,3-butanediamine (bn) (DeHayes & Busch, 1973; Niketić & Rasmussen, 1978; Hald & Rasmussen, 1978), to date only one structure of a 2,3-butanediamine complex, that of *cis*-dichloro(*meso*-2,3-butanediamine)-palladium(II) (Ito, Marumo & Saito, 1971), has been reported. Here we report the first structure determination on a tris complex of 2,3-butanediamine and the first determination on any complex containing the racemic form of this ligand.

Owing to the presence of multiple chiral sites, tris complexes of 2,3-butanediamine can exist in a number of diastereomeric forms (Tapscott & Marcovich, 1978; Woldbye & Borch, 1967). The *lel lel lel* diastereomer, consisting of a racemic mixture of Δ-[Co(R,R-bn)$_3$]$^{3+}$ and Λ-[Co(S,S-bn)$_3$]$^{3+}$, was isolated as described elsewhere (Hilleary, Them & Tapscott, 1980) and the chloride salt was obtained by ion exchange. Hexagonal orange crystals were obtained upon evaporation of an aqueous solution and a single crystal of dimensions 0·45 × 0·45 × 0·40 mm was selected and mounted in a quartz capillary. Diffraction data were collected at room temperature on a Syntex *P*3/*F* diffractometer system equipped with a graphite monochromator using Mo $K\alpha$ radiation (λ = 0·71069 Å). The system correctly assigned the crystal class as trigonal. Cell dimensions were determined from a least-squares fit to automatically centered settings for 25 reflections. The space group was determined by the systematic absences $hh\bar{2h}l$, $l = 2n + 1$, which correspond to $P\bar{3}1c$ (No. 163) and its non-centrosymmetric counterpart $P31c$ (No. 159). Subsequent satisfactory refinement showed the correctness of the former assignment.

The intensities were measured in the 2θ–θ scan mode with a scan range of 0·9° below $2\theta(K\alpha_1)$ to 1·1° above $2\theta(K\alpha_2)$ using a background counting time/total scan time ratio of 0·5. A total of 6708 reflections ($\pm h,k,\pm l$) were collected with $1\cdot0° \leq 2\theta \leq 60\cdot0°$. Equivalent reflections were averaged to give 1017 unique reflections of which 988 were considered observed with $I \geq 1\cdot5\sigma(I)$. The intensities of two standard reflections, which were monitored every 94 reflections, indicated no significant decay during the data collection. The data were corrected for Lorentz and polarization effects, extinction using $F = F_o[1 - (xF^2 \times 10^{-4}/\sin\theta)]$, x = 0·0126 (16), and absorption using an empirical correction based on ψ scans [μ(Mo $K\alpha$) = 12·3 cm^{-1})].

The Nicolet *SHELXTL* direct-methods program (Sheldrick, 1979) produced the starting positions for all non-H atoms except Cl. A difference map after two cycles of isotropic least-squares refinement provided the correct Cl position. Two additional cycles of isotropic least-squares refinement with the Cl atom correctly placed decreased R $(=\sum||F_o| - |F_c||/\sum|F_o|)$ from 0·51 to 0·13. Anisotropic refinement on the non-H atoms further reduced R to 0·06. A difference map provided six highest density peaks (0·68–0·92 e Å^{-3}) which gave reasonable H-atom positions. These were included and the H atoms were refined with isotropic thermal parameters and the non-H atoms with anisotropic thermal parameters until convergence was reached at R = 0·038 and R_w = 0·044, where $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w(F_o)^2]^{1/2}$. A difference map showed the two highest peaks to be 0·75 Å from Cl (~0·75 e Å^{-3}) and 0·90 Å from Co (~0·63 e Å^{-3}). Since neutral-atom scattering factors and anomalous-dispersion corrections had been applied to all atoms up to this point, a change to scattering factors and anomalous-dispersion corrections for Co^{3+} and Cl^- was made. Sixteen cycles of least-squares refinement converged to R = 0·039, R_w = 0·045, and goodness of fit (g.o.f.) = 1·43, where g.o.f. = $[\sum w(|F_o| - |F_c|)^2/(m - n)]^{1/2}$, m = unique data used (988), n = number of variables refined (59). The final difference map showed each of the top six peaks (0·82–0·46 e Å^{-3}) to be within 0·75 Å from Co or within 1·2 Å from Cl. All other difference-map peaks were less than or equal to 0·4 e Å^{-3}. The use of Co^{3+} and Cl^- scattering factors instead of neutral-atom factors resulted in slightly higher standard deviations, essentially no changes in bond lengths and bond angles involving non-H atoms ($\leq 1\cdot3\sigma$), and very slight changes in those parameters involving H atoms ($<1\cdot8\sigma$). The final atomic parameters are given in Table 1.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36560 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates and isotropic thermal parameters*

	x	y	z	U_{eq}* or U_{iso} (Å^2)
Co	0·33333	0·66667	0·25000	0·0189 (2)*
Cl	0·54260 (4)	−0·54260 (4)	0·25000	0·0431 (3)*
N	0·2060 (2)	0·5309 (2)	0·3902 (2)	0·0289 (6)*
C(1)	0·2019 (2)	0·4111 (2)	0·3467 (3)	0·0296 (7)*
C(2)	0·0859 (2)	0·2984 (2)	0·4208 (4)	0·0435 (9)*
H(1)	0·220 (3)	0·545 (3)	0·500 (4)	0·047 (8)
H(2)	0·132 (3)	0·524 (3)	0·366 (4)	0·036 (7)
H(11)	0·280 (3)	0·415 (2)	0·390 (4)	0·032 (6)
H(21)	0·088 (3)	0·309 (3)	0·554 (4)	0·059 (9)
H(22)	0·008 (3)	0·292 (3)	0·372 (4)	0·051 (8)
H(23)	0·074 (3)	0·215 (3)	0·404 (4)	0·056 (9)

* $U_{eq} = \langle U^2 \rangle$ (Willis & Pryor, 1975).

Discussion. The complex cations lie on sites of D_3 crystallographic symmetry with both enantiomers present in the unit cell. Table 2 gives interatomic distances and angles for the complex, which is shown as the Λ-[Co(S,S-bn)$_3$]$^{3+}$ enantiomer in Fig. 1. The conformation is *lel lel lel* with all methyl groups equatorial in the five-membered chelate rings, as expected (Niketić & Rasmussen, 1978). Of particular interest is the detailed chelate-ring conformation as reflected in the torsional angles given in Table 3. The N—C(1)—C(1)′—N′ dihedral angle of 55·1 (3)° and the N···N and C—C vector crossing angle of 30·1° are slightly larger than the average values of 51 and 28° found for the analogous angles in recently published structures of *lel lel lel* tris(ethylenediamine)cobalt(III) (Brouty, Spinat & Whuler, 1980; Templeton, Zalkin, Ruben & Templeton, 1979) though a survey of other structures indicates that these values are rather variable. Surprisingly, the N—C—C—N torsional angle of 53·3° found in the only *meso*-2,3-butanediamine chelate structure determined to date (Ito *et al.*, 1971) is only slightly less than the value that we find for chelated (±)-2,3-butanediamine. Molecular-mechanics calculations predict a ring flattening corresponding to an N—C—C—N torsion-angle decrease of about 6° for chelated *meso*-2,3-butanediamine compared with the racemic ligand (Niketić & Rasmussen, 1978). We hasten to point out, however, that a comparison of our structure with that of the *meso*-2,3-butanediamine complex, where a monokis chelate of palladium(II) is present, may be misleading.

Comparison of bonding parameters other than dihedral angles for the (±)-2,3-butanediamine chelate ring in this structure with average values for corresponding parameters as determined in recent cobalt(III) ethylenediamine structure determinations (Niketić & Rasmussen, 1978) shows a very small decrease (0·015 Å) in the Co—N bond length and a decrease (2·5°) in the N—C—C ring angle as the only significant changes upon going from the ethylenediamine chelate ring to the (±)-2,3-butanediamine chelate ring.

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Co—N	1·958 (2)	N—H(2)	0·89 (4)
N—C(1)	1·486 (3)	C(1)—H(11)	1·00 (3)
C(1)—C(2)	1·521 (3)	C(2)—H(21)	1·06 (4)
C(1)—C(1)′	1·525 (4)	C(2)—H(22)	1·00 (4)
N—H(1)	0·88 (3)	C(2)—H(23)	0·97 (4)
N—Co—N′	85·0 (1)	H(1)—N—H(2)	109 (3)
Co—N—C(1)	109·3 (1)	C(1)—C(2)—H(21)	108 (2)
N—C(1)—C(1)′	104·9 (2)	C(1)—C(2)—H(22)	111 (2)
N—C(1)—C(2)	111·4 (2)	C(1)—C(2)—H(23)	119 (2)

Primed atom positions are generated by $y-x, y, \frac{1}{2}-z$.

Table 3. *Dihedral angles* (°)

N—C(1)—C(1)′—N′	55·1 (3)	C(2)—C(1)—C(1)′—N′	177·3 (2)
C(2)—C(1)—C(1)′—C(2)′	−60·5 (2)	Co—N—C(1)—C(1)′	−42·8 (2)

Primed atoms are generated by $y-x, y, \frac{1}{2}-z$.

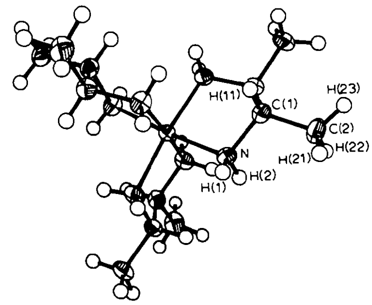

Fig. 1. The *lel lel lel* Λ-[Co(S,S-bn)$_3$]$^{3+}$ ion. The thermal ellipsoids are shown at the 50% probability level.

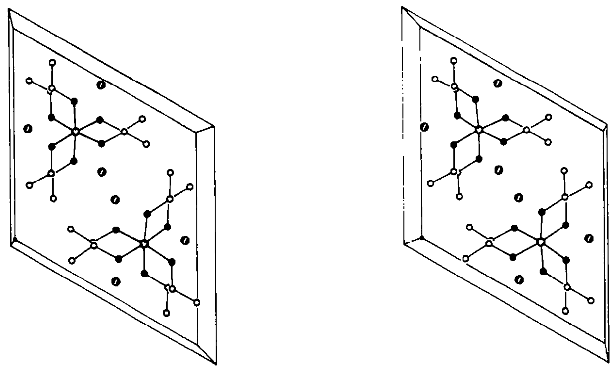

Fig. 2. The unit-cell contents for *lel lel lel* [Co{(±)-bn}$_3$]Cl$_3$ as viewed down the c axis.

The unit-cell contents are shown in Fig. 2. Each chloride ion is at a site of C_2 symmetry with four hydrogen-bonding contacts with amine groups. There are two N···Cl distances of 3·234 (2) Å [H(1)···Cl, 2·56 (4) Å; N—H(1)···Cl, 133 (3)°] and two N···Cl distances of 3·404 (2) Å [H(2)···Cl, 2·54 (4) Å; N—H(2)···Cl, 165 (3)°]. These contacts are similar to those observed in related structures (see *e.g.* Iwata, Nakatzu & Saito, 1969).

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Refinement of Diaquabis(glycinato-*O*,*N*)nickel(II)

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Abstract. $[Ni(C_2H_4NO_2)_2(H_2O)_2]$, $C_4H_{12}N_2NiO_6$, $M_r = 242\cdot86$, monoclinic, $P2_1/n$, $a = 7\cdot616\,(1)$, $b = 6\cdot601\,(1)$, $c = 9\cdot247\,(1)$ Å, $\beta = 110\cdot95\,(1)°$, $V = 434\cdot1\,(1)$ Å^3, $Z = 2$, $D_c = 1\cdot858$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1\cdot54184$ Å, μ(Cu $K\alpha$) = $3\cdot10$ mm^{-1}, $F(000) = 250$. The structure was refined with 823 independent reflections by full-matrix least squares with anisotropic temperature factors for all non-hydrogen atoms. Hydrogens were treated isotropically with their bond lengths constrained to fixed values. Final R factors are $R = 0\cdot036$, $R_w = 0\cdot035$. The crystal, which shows an antiferromagnetic transition at 0·88 K, is strongly stabilized by a net of hydrogen bonds which are potentially the most likely paths for superexchange interactions between Ni ions.

Introduction. Recent magnetic susceptibility measurements on diaquabis(glycinato-*O*,*N*)nickel(II) (NiDB) have shown an antiferromagnetic transition at 0·88 K (Calvo & Nascimento, 1981). The crystal structure of this compound was established by two-dimensional Fourier analysis several years ago (Stosick, 1945) but the limitations inherent in this technique and the lack of computing facilities to perform least-squares refinements severely limited the accuracy of the results. The structure was reinvestigated from visually estimated three-dimensional photographic data and refined to an R factor of 0·095 but the positions of the hydrogen atoms could not be established with certainty and were therefore not reported (Freeman & Guss, 1968). The crystal structure is known to be stabilized by a net of hydrogen bonds. Since these hydrogen bonds are potentially the most likely paths for superexchange interactions between the Ni ions of the structure (see, for instance, Watanabe, 1962) a model for the transition mechanism would require accurate knowledge of the proton sites.

To obtain this information a diffractometric three-dimensional Fourier analysis and least-squares refinement of the NiDB complex was undertaken.

NiDB was synthetized as outlined by Stosick (1945) and detailed by Sen, Mizushima, Curran & Quagliano (1955). The material was purified by recrystallization and suitable crystals for X-ray analysis were obtained by slow evaporation from water solution at room temperature.

A fragment of irregular shape with maximum and minimum linear dimensions of about 0·20–0·25 mm

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